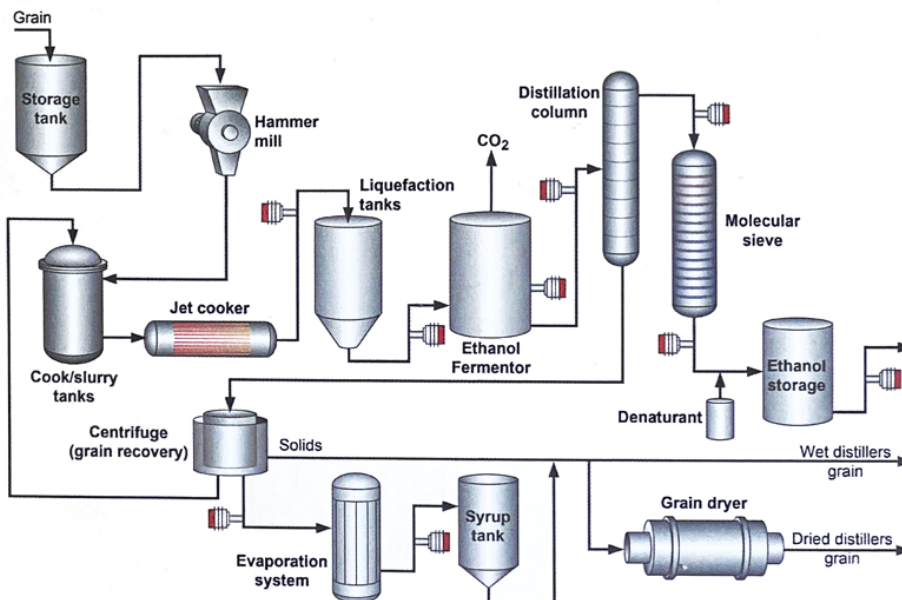
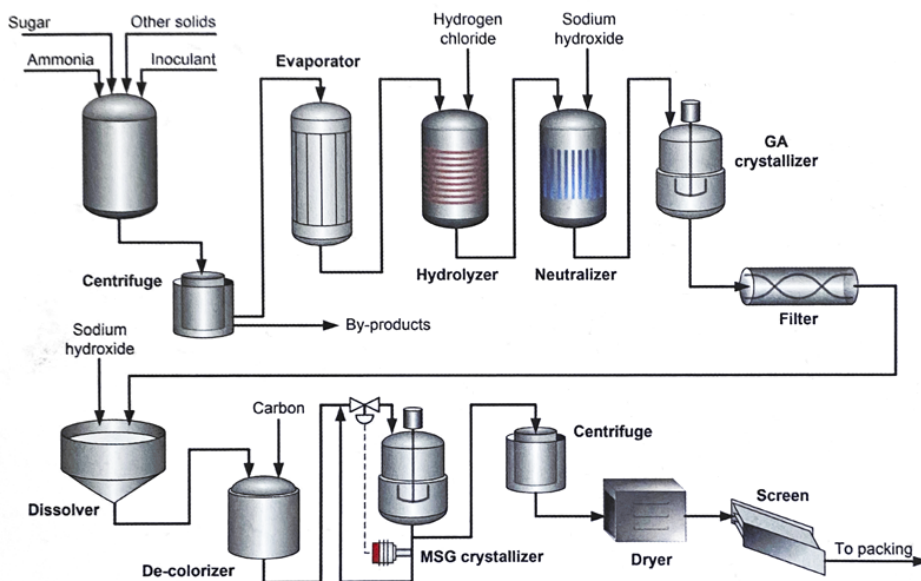
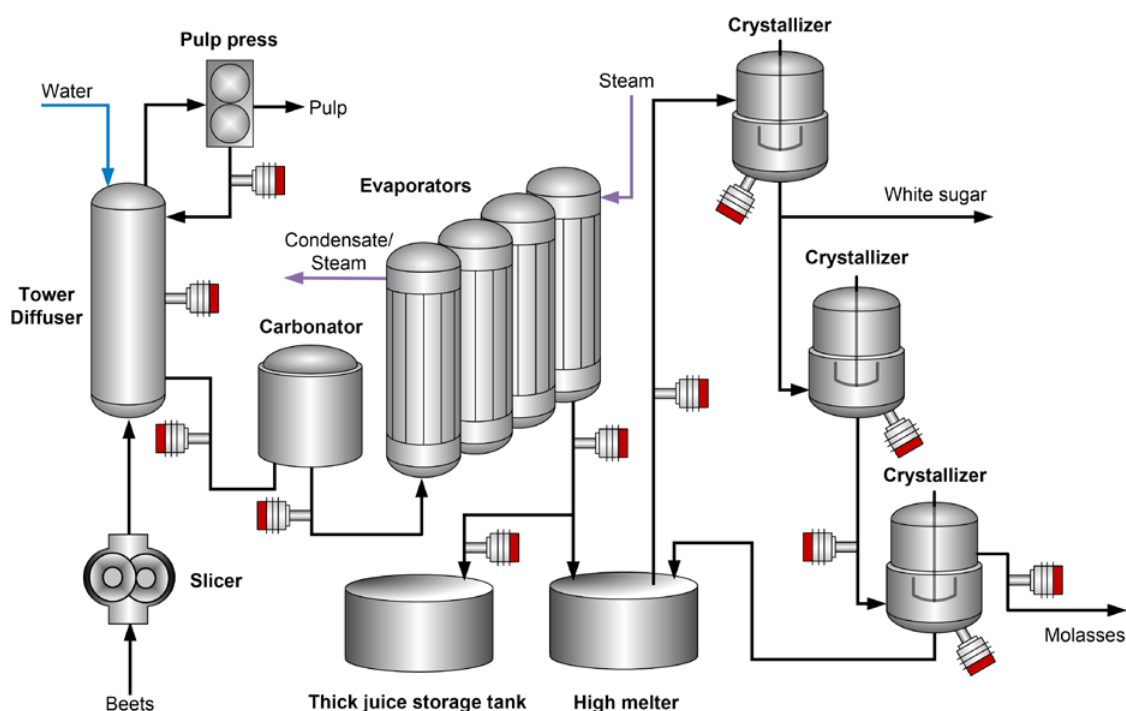


# PROCESS REFRACTOMETER APPLICATION NOTES



**SECOND  
EDITION**

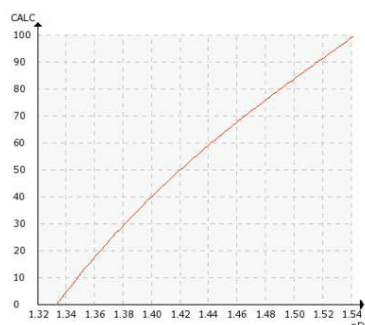


## BEET SUGAR

### Typical end products

Sugar for sweetening soft drinks, beer brewing, pastries, preserves, sausages, beverages, sweets, confectionery, ice cream, liqueurs, pharmaceuticals, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Sugar beet is a form of beet which grows mainly in temperate climate zones. Because its juice contains high amount of sucrose, sugar beet is used to produce sugar and is second only to sugarcane as the major source of the world's sugar.

The agricultural cost of producing beet sugar is significantly higher than the cost of cane sugar, so optimal control and efficiency of production is a high priority.

### Application

#### Diffusion

After reception at the processing plant, the beet roots are washed, mechanically sliced into thin strips called *cosettes*, and passed on to the diffuser, a processor used for the extraction of sugar content into a water solution. The diffuser process is slow, so real-time and continuous measurement of the concentration is important to monitor its progress.

#### Juice Purification

The diffusion juice contains, in addition to sucrose, some non-sugar impurities. The process of liming and carbonation is in two stages. The first stage is primarily for the removal of sludge. The second stage involves further addition of carbon dioxide to remove the lime, which remains in the solution. The resulting solution is called *thin juice*.

#### Evaporation

The thin juice is heated and pumped into the multiple-effect evaporators to create a *thick juice*. The dissolved solids concentration is raised from its initial concentration to 50-75 %.



## Crystallization

The thick juice moves on to crystallization. Crystallization takes place in boiling pans or in vacuum pans.

In the crystallizer, the liquor is concentrated further by boiling and then *seeded* with fine sugar crystals. The crystals grow as sugar from the *mother liquor* forms around them, thus decreasing the sugar content of the liquor. The resulting sugar crystal and syrup mix is called a *massecuite*.

Crystallization stops when the crystals have reached the desired size. The massecuite is then passed on to a centrifuge to separate the crystals from the liquid before they are dried in a granulator with warm air.

The remaining syrup stills contains some sugar which is recovered by further crystallization. The resulting sugar is of lower quality and used to make the mother liquor. The separated syrup is molasses, which still contains sugar, but has too many impurities to undergo further processing economically.

## Final molasses

*Final molasses*, in beet sugar manufacturing, is the final syrup, which is centrifuged from the sugar crystals after repeated crystallization. The final molasses still contains a high amount of sucrose and it can be sold as beet pulp pellets for animal feed, or it can be further processed to recover more sugar. Tight control of molasses processing is a must to ensure the economic viability of the process.




## Instrumentation and installation

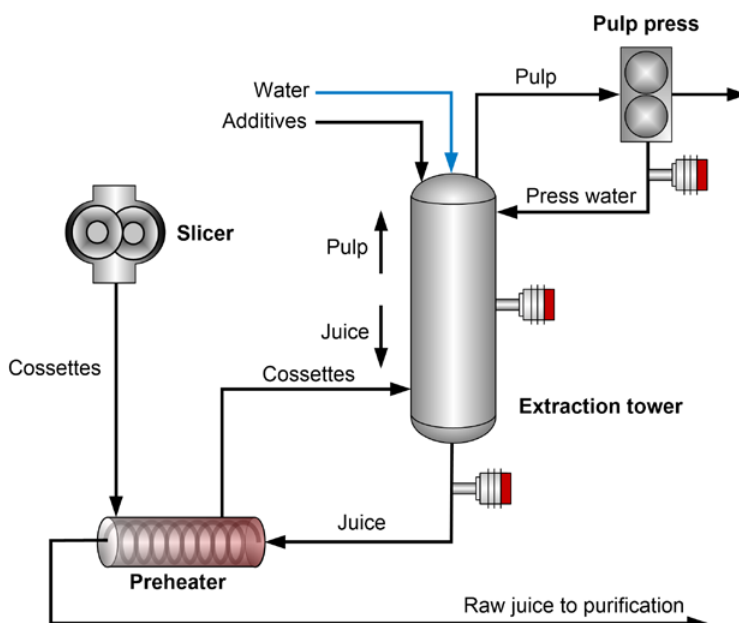
Vaisala K-PATENTS® Process Refractometer PR-43 provides accurate in-line Brix and concentration measurements for the beet sugar refining process.

The performance of all basic steps in this process can be optimized. The refractometer provides rapid detection of process disturbances during diffusion. Control of the concentration levels saves energy by minimizing the quantity of water, which requires evaporation later in the process. In addition, calculations of the diffuser plant output can be made utilizing the process refractometer.

At the evaporators, the PR-43 refractometer assists in regulating the product flow to best suit the evaporator's capacity, thus saving energy. The refractometer's output can also be used to control the steam flow and optimize the evaporation process.

Refractive index technology is the best option for control and monitoring of crystallization. The controlling refractometer monitors selectively the concentration of the mother liquor to maintain the supersaturation state required for the formation of crystals. Due to our unique technology, the refractometer measurement is not influenced by bubbles or the crystals formed.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

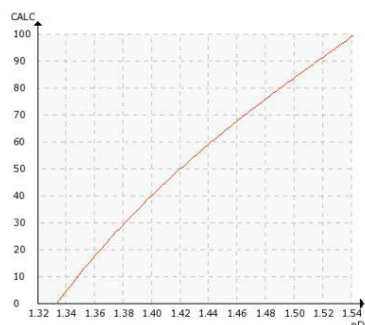


## BEET SUGAR JUICE

### Typical end products

Sugar for sweetening soft drinks, beer brewing, pastries, preserves, sausages, beverages, sweets, confectionery, ice cream, liqueurs, pharmaceuticals, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

In the manufacture of sugar from beet, the extraction step aims at extracting the sucrose from the beet cells with hot water by a countercurrent principle. This process is known as *diffusion*.

The products from extraction are beet pulp and raw juice. The beet pulp can be used for cattle feed or can be modified to obtain fibers for human consumption. The raw juice is further processed to finally make the sugar crystals.

### Application

After the beets are thoroughly washed, they are passed through the slicers, where they are cut into long, thin strips or *cossettes*. The cossettes, upon entering the continuous diffuser, are elevated by means of a perforated flight scroll or similar device. Countercurrent water is introduced at the upper end of the diffuser. The diffuser is steam-heated by means of external jackets. The extracted sugar leaves the diffuser at a concentration of 10 to 15 Brix, which accounts for about 98 % extraction of the beets.

Control of the whole extraction process is complex. Precise real-time data from different areas of the diffuser tower is required. Accurate control of the concentration levels saves energy by minimizing the amount of water, which has to be evaporated later in the process.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 allows precise control of the extraction process and fast detection of process disturbances.




The refractometer is installed in the press water line, where it measures the sugar concentration of the water separated from the pulp. As this water is recycled to the upper part of the diffuser, it is important to obtain accurate concentration readings. The typical range is 0-5 Brix and the temperature is 60 °C (140

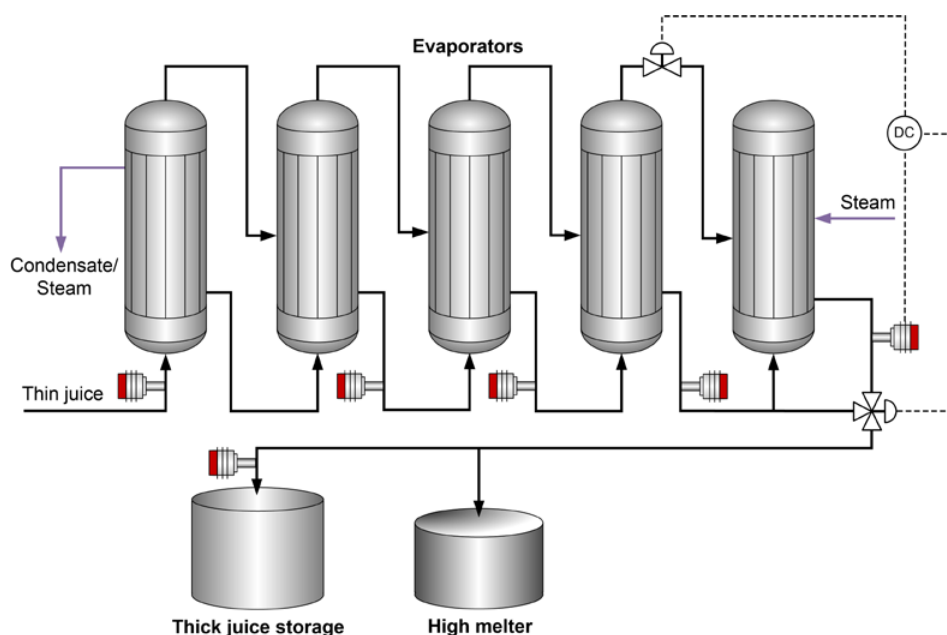
°F). The pipe diameter should be reduced just before the refractometer, so that a flow rate of 1.5 m/s (5 feet/s) is achieved. Automatic prism cleaning with steam is recommended at this point of the process.

Another refractometer at the midpoint of the extraction tower measures the Brix of the liquid, so that the fluctuation of the sugar content in the cossettes leaving the tower can be detected at the earliest possible stage. Then, by regulating the flow of raw juice, optimum efficiency, reduced energy costs and minimized sugar loss can be achieved.

Concentration measurement at the diffuser tower outlet also provides valuable data for balancing the process. The refractometer should be installed after a stone trap. Due to the impurities present in raw juice, prism cleaning with steam is recommended.

The in-line and real-time measurements improve the extraction control significantly. The optimum compromise balance between juice flow and water evaporation can be achieved, and costly and time-consuming manual sampling avoided.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

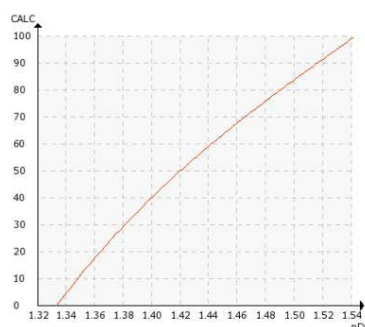


## BEET SUGAR JUICE

### Typical end products

Sugar for sweetening soft drinks, beer brewing, pastries, preserves, sausages, beverages, sweets, confectionery, ice-cream, liqueurs, pharmaceuticals, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



## Introduction

The evaporator station forms an important link between the juice production and crystallization steps of the sugar production plant. Usually, the evaporator station comprises 4 to 5 evaporator stages, of which the first ones consist of multiple evaporators. As the pressure in the evaporators is regulated, so that it decreases from stage to stage, the juice passes sequentially through all the stages.

Once the required concentration is reached, the thick juice is discharged from the last stage of the evaporator station.

A general requirement is that the concentration of thick juice in the evaporator outlet should be as high as possible, reaching 68-75 % dry substances in modern factories. It is then possible to apply crystallization technologies, which ensure a high sugar quality and facilitate low energy consumption.

## Application

### Evaporator Plant Feed

Appropriate feed concentration control for an evaporation plant is essential for optimal results. Normally Vaisala K-PATENTS® Process Refractometer signal is used to adapt the feed of the thin juice to the capacity of the evaporation plant (using feed-forward control).

The refractometer signal can also be used for keeping the feed concentration to the evaporation plant constant by the controlled addition of thick juice to the incoming thin juice. This measurement is made after mixing to maintain precise control of the mix.

### Evaporator Plant Outlet and Intermediate Effects Control

Evaporation in the multiple-effect evaporation plant is more cost effective than in the crystallizer, which economically justifies an evaporation control system. The Process Refractometer is not only used after the final evaporation stage, but also after the intermediate stages. The refractometer is also used in a control loop, which keeps the thick juice concentration constant by

regulating the steam flow or the quantity of the thick juice recirculated to the evaporators. As a result, the specific energy consumption can be minimized.

#### Thick Juice to Storage Tank

In this application, the process refractometer helps to maximize the storage capacity by keeping the thick juice at as high a concentration level as possible.


#### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 is an ideal instrument for measuring Brix in the demanding beet sugar evaporation process. The refractometer is installed via a flange or a clamp connection.

Lime residuals and other additives may coat the prism and an automatic steam wash is recommended in thin juice applications with typical range of 0-25 Brix.

In some plants, the thick juice also contains supersaturated impurities (e.g. oxalic acid), which may crystallize on the prism. Therefore, an automatic prism wash with hot high-pressure water may be necessary. In extreme cases, the refractometer can be installed in a narrower by-pass line flowing from a cooling heat-exchanger, where the impurities crystallize.

Because of its digital sensing technology, the refractometer can measure dissolved sugar uninfluenced by the presence of undissolved particles and crystals.

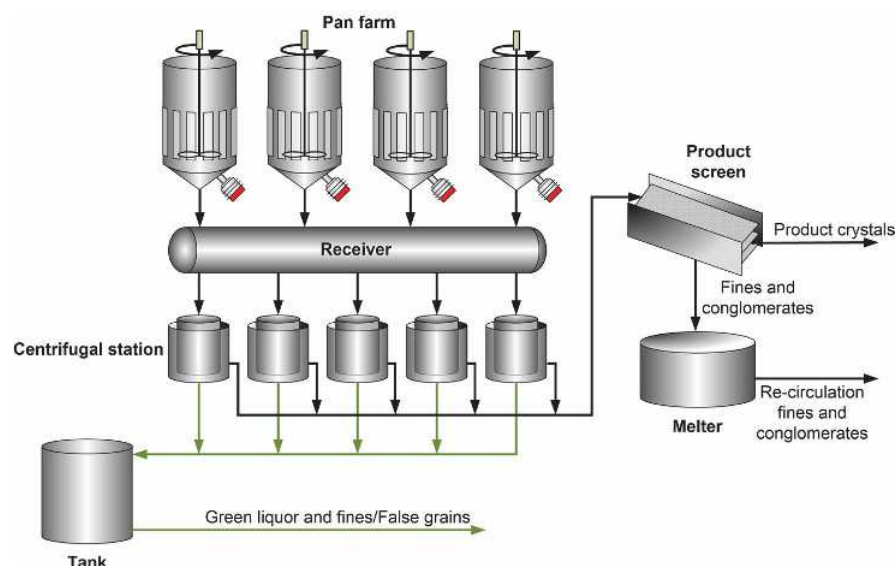
Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Prism wash systems	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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## Beet sugar juice

Beet sugar crystallization | 1.01.03



### Measured medium

- Sugar and sweeteners

### Typical end products

- Crystallized sugar

## Control of crystallization

Crystallization can only take place if the solution is supersaturated. Supersaturation is a multivariable function of several parameters of the liquid phase only (syrup or mother liquor). Crystals can grow only if supersaturation is higher than 1.0.

No single instrument can provide on-line data on supersaturation. Conventional sensors used to monitor crystallization provide data of a single massecuite parameter only.

However, two sensors can provide information on the important massecuite parameters. These sensors are not influenced by other process parameters:

1. Vaisala's process refractometer. The refractive index technology is successfully used for selective concentration measurement of the liquid phase over the complete crystallization strike.
2. A sensor for measurement of the total solids content (brix of the massecuite).

## Introduction

The crystallization process takes place in vacuum pans, which boil the thick juice. When the juice reaches the correct concentration, it is seeded with sugar crystals, which provide the nucleus for larger crystals to grow. When the crystals reach the required size, the process is stopped and the resultant mixture of crystal sugar and syrup, known as *massecuite*, is spun in centrifuges to separate the sugar from the mother liquor. The sugar crystals are washed and, after drying and cooling, conveyed to storage silos.

## Application

Crystallization has a major effect on product quality and production costs. Supersaturation is the driving force of crystallization and crystal growth, and the speed of crystallization depends on this parameter. Too low

supersaturation increases the strike time. Too high supersaturation results in poor crystal quality (fines and conglomerates). These crystals are melted, concentrated, recirculated and crystallized again, which wastes time and energy and decreases the yield of sugar per strike, while increasing water usage and production costs. To decrease the amount of recycled sugar the particle size and particle size distribution must be as close to the target values as possible.

Besides supersaturation, the other important parameters are: crystal content, mother liquor purity and massecuite solids content. Instead of using a single probe as the main instrument, a measurement of the liquid phase (syrup and mother liquor concentration) as well as a measurement of the syrup and massecuite solids content, are required for successful control of the supersaturation.

## Instrumentation and installation

Zutora SeedMaster-4 is a unique, fourth generation crystallization transmitter and seeding device to be used with Vaisala's process refractometer. The SeedMaster-4 allows for accurate in-line and real-time monitoring of supersaturation and crystal content over the complete process of crystallization, and implementation and control of automatic or manual seeding. The SeedMaster-4 can be connected to one or two process refractometers and to one or two crystallizers. The SeedMaster-4 provides the following:

1. Electronic data capture on massecuite parameters.
2. On-line calculation and transmission of massecuite parameters for the advanced control of sugarcrySTALLIZATION with control system.

3. Organization and storage of strike history data archive.
4. Advanced communication with the control system.
5. Automatic seeding of the vacuum pans.
6. Serves as user interface for the pan and controlsystem operators.

The mounting location of the refractometer sensor should be carefully evaluated. Despite the use of stirrers in crystallizers, circulation of the massecuite becomes sluggish when the crystal content increases. This means that the syrup/mother liquor concentration and temperature, which have a considerable influence on supersaturation, will not be the same in the full massecuite volume. This is a limitation for all types of sensors.

In general, Vaisala K-PATENTS® PR-23-GP refractometer should be installed in a location where the measured sample is representative for the largest volume of the syrup or massecuite (in terms of sugar content and temperature). The preferred installation position is under the calandria using a counter flange adapter for process connection to minimize dead space around the flange. This is critical for the product quality as the leftovers may grow from batch to batch resulting in a negative effect on the product quality and in controlling the strike (see Figure 1).

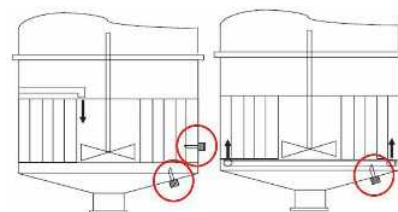


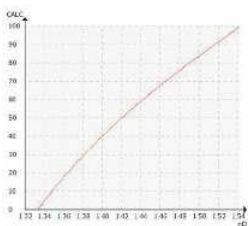


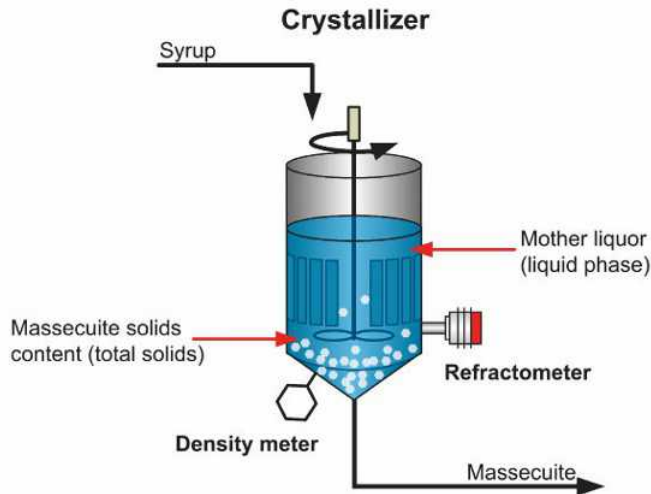
Figure 1. Recommended sensor locations.

Instrumentation	Description
	SeedMaster-4 for multiparameter sugar crystallization monitoring and automatic seeding.  Crystallization transmitter and seeding device is used with the Process Refractometer PR-23-GP. It allows for accurate in-line and real-time monitoring of supersaturation and crystal content over the complete strike, and implementation and control of automatic or manual seeding.
	Process Refractometer PR-23-GP is an industrial refractometer for crystallizers. Installation through a flange connection and Counter flange adapter -AP for vacuum pan installations.
<b>Prism wash system with warm water</b>	The integral prism wash system helps to avoid crystals deposit or scaling on the prism surface. Prism wash system components are a refractometer with integral water wash nozzle mounted at the refractometer probe, a warm feed water source (hot condensate), and an Indicating transmitter with build-in relays for driving the water valve and controlling the wash.
<b>Measurement range</b>	Refractive Index (nD) 1.3200–1.5300, corresponding to 0–100 Brix.
<b>Chemical curve</b>	R.I. per BRIX at Ref. Temp. of 20 °C



# Beet sugar, cane sugar

## Sugar crystallization: massecuite solids content and mother liquor concentration



### Particularities of the crystallization process

Varying process conditions present a challenge to measuring massecuite solids content accurately. Process medium changes during different production phases from liquid to massecuite and consists of both liquid and crystals. Generally, calibrating the massecuite solids content meter can be quite easy either for the liquid phase or for massecuite phase, but not for both. This means that the massecuite solids content sensors cannot solely produce reliable results as they do not cover the whole processing range.

For accurate results, the calibration must cover the full range from pure liquid to the point where Vol. 55 % of the massecuite is crystals. However, if the vacuum pan is also equipped with a refractometer, the calibration difficulties can be mostly avoided.

In the beginning of the strike the process medium is pure liquid. At this point the refractometer and the massecuite solids content meter should give the same measurement value (Figure 1).

The crystals are introduced only after the pan has been seeded. After seeding the massecuite solids content increases as the crystal content increases, whereas refractometric concentration stays rather constant ( $\pm 3$  Brix). Improved accuracy in massecuite solids measurement can be achieved when a refractometer is combined with a microwave sensor. Moreover, a refractometer can offer calibration reference value for the

### Introduction

This note explains the methods and calibration procedure for measuring successfully the massecuite solids content and mother liquor concentration in sugar vacuum pans over the entire crystallization process. Both parameters need to be measured and controlled, as they influence the quality of the sugar crystals.

Massecuite solids content, or total sugar content, is typically determined using e.g. microwave measuring technology whereas mother liquor concentration (dissolved sugar) is measured with a refractometer. The common measurement scale is Brix.

### Refractometer

Vaisala K-PATENTS® Process Refractometer is successfully used for selective measurement of liquid phase over the complete crystallization strike. Due to the unique digital principle, the refractometer measures the true concentration of the mother liquor,

without being influenced by the sugar crystals or bubbles in the pan. Moreover, the refractometer does not require re-calibration.

### Massecuite solids content meter

A microwave sensor can measure only the total solids (liquid and undissolved solids phase). Microwave probes are based on the measurement of attenuation and phase shift of microwave radiation. Both are related to the length travelled by the radiated signal, and the density and dielectric characteristics of the medium.

Phase shift is the result of decreasing speed of propagation. Due to the fact that water has a high dielectric constant compared to sugar and the accompanying non-sugars, the water content (and, consequently, solids content) is the major parameter, which determines the dielectric properties of the medium. As an output the microwave probes provide density or solids content of the massecuite.

massecuite solid content meter at the seeding point.

The recommended control practice is to use a refractometer to measure the concentration from the beginning of the strike until the seeding point. The refractometer provides accurate and repeatable seeding.

## Calibration procedure

The process refractometer is factory calibrated according to International Commission for Uniform Methods of Sugar Analysis (ICUMSA) Brix table. The factory calibration should be verified against the production laboratory when commissioning the instrument. The laboratory reference values should be taken in the beginning of the strike when there is only liquid present in the vacuum pan. Only a small BIAS adjustment might be needed to match the refractometer and the production laboratory.

Typically, the massecuite solids content meter needs regular calibration and calibration checks. The best calibration result is typically achieved when the microwave density meter is calibrated on a narrow range for either liquid sugar or massecuite. In vacuum pans the recommended

calibration practice is to calibrate the microwave density meter from seeding point to the end of the strike, which means that the reference samples should be taken at the seeding point, end of the strike and one sample in between (minimum three samples).

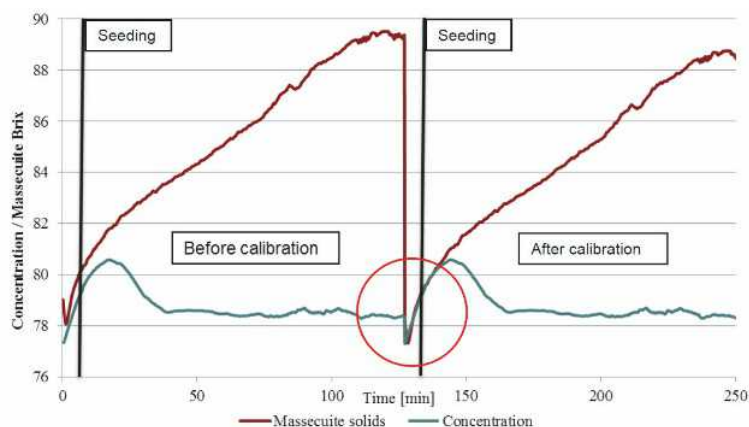


Figure 1. Refractometer and massecuite solids content measurement trends before and after calibration. The liquid concentration and massecuite solids content should be the same before seeding. After seeding they separate as the crystals start to grow.

Table 1. Example of calibration table.

Sample	LAB (Mass.Sol.Cont)	Refractometer (Concentration)	Microwave (Mass.Sol.Cont)	Difference refractometer -LAB	Difference microwave/nuclear -LAB
1	77.6	77.5	78.5	-0.1	+0.9
2	79.5 (seeding)	79.4	80.9	-0.1	+1.4
3	85.6	78.5	86.3	-	+0.7
4	90.5	77.9	91.3	-	+0.8

Table 2. Example of calibration table after calibration procedure. Refractometer offset adjustment +0.1 Massecuite solids meter offset adjustment -0.9.

Sample	LAB (Mass.Sol.Cont)	Refractometer (Concentration)	Microwave (Mass.Sol.Cont)	Difference refractometer -LAB	Difference microwave/nuclear -LAB
1	77.6	77.6	77.6	0.0	0.0
2	79.5 (seeding)	79.5	80	0.0	+0.5
3	85.6	78.5	85.4	-	-0.2
4	90.5	77.9	90.4	-	-0.1

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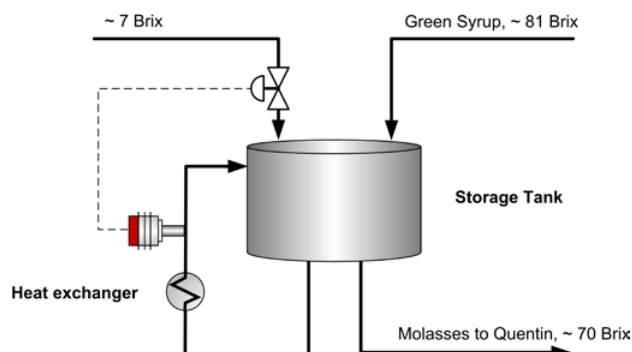


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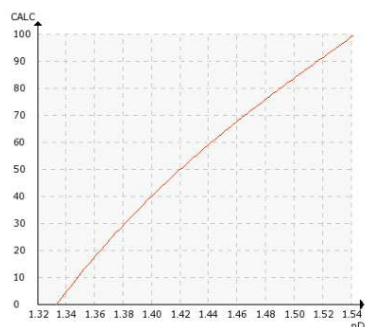


## BEET SUGAR AND GREEN SYRUP

### Typical end products

Animal food, food additives, chelating agent, rum, industrial ethanol, alternative fuels, yeast, organic chemicals, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

After each crystallization step, the massecuite is centrifuged to separate the sugar crystals from the liquid phase (mother liquor or syrup). The syrup left from the final crystallization stage is called *molasses* and intermediate syrup is referred to as *green syrup*.

Molasses is a viscous product which still contains a high amount of sucrose, up to 60 % on dry basis. This sugar is still of considerable value and it needs to be extracted and recycled within the crystallization plant to maximize production.

### Application

A method occasionally used in Europe for molasses desugarization is the *Quentin process*, in which the potassium and sodium in beet juice are replaced by magnesium through ion exchange. Quentin is an ion exchange column. Alkaline ions (potassium/sodium) are exchanged with magnesium to reduce the solubility of sugar in water. This allows a higher proportion of sucrose to be crystallized but obviously produces molasses with lower sugar content (about

5% less), increased magnesium level, and reduced potassium and sodium levels.

The performance of the ion exchange column is improved by a stable syrup concentration.




### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 is used to control the feed to the Quentin columns, giving a constant load. This allows the ion-exchange resins to function longer, and results in higher efficiency of the desugarization process. Typical concentration range of molasses to Quentin is 60 to 80 Brix and typical temperature 85 °C (185 °F). A minimum flow velocity of 1.5 m/s (5 feet/s) is recommended.

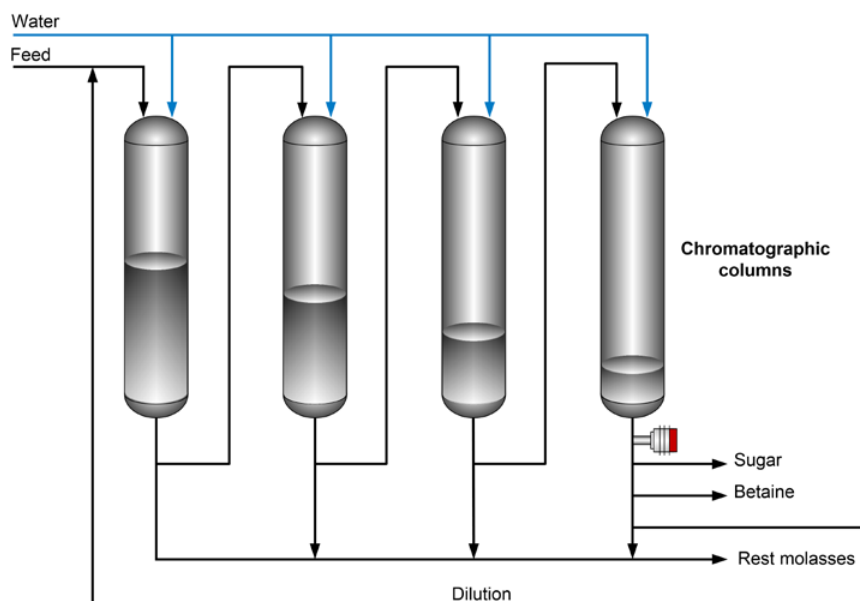
The PR-43 refractometer is also used to check the concentration of the final molasses to ensure that it is above the lowest concentration limit specified by the buyer. The refractometer provides Ethernet and 4-20 mA output signals for real-time control of dilution to avoid uneconomically high concentrations.

Typical concentration range is 70-85 Brix and typical process temperature is 80 °C (176 °F).



Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
<b>User Interface</b>	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
<b>Prism wash systems</b>	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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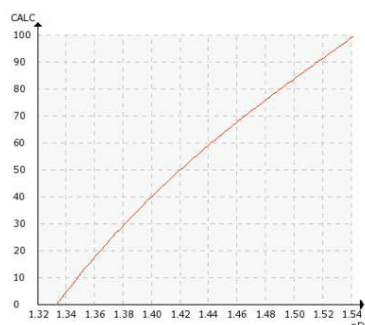


## SUGAR AND BETAINE

### Typical end products

Animal feed, cosmetics, fertilizers, etc.

Chemical curve: R.I. per BRX at Ref. Temp. of 20°C



### Introduction

Molasses desugarization by ion-exclusion and chromatographic separation is a process for extracting sugar from molasses.

Another method is betaine separation from beet molasses, using chromatographic separation and having at least three chromatographic columns connected in series. Betaine is used in the pharmaceutical, cosmetic, feed, food and chemical industries.

Diluted molasses is supplied to a chromatographic water feed column. Different fractions leave the bottom of the column. First is a non-sugar waste fraction, a second fraction contains a substantial proportion of the sugars in the feed and a third fraction of betaine,

which is eluted at the tail end after the sugar fraction. The third fraction contains a high proportion of the betaine in the feed, as much as 80 % or more, on dry substance. From this betaine fraction, betaine can be recovered by crystallization or may be recovered as hydrochloride, if desired. Use of these processes has enabled a 95 % recovery of the sugar in molasses.

### Application

#### Desugarization process

During the chromatographic separation, a wide spectrum of adsorption and exclusion of various ionic and non-ionic components takes place. These processes divide the feed into several fractions with different compositions (Figure 1).

CONC.

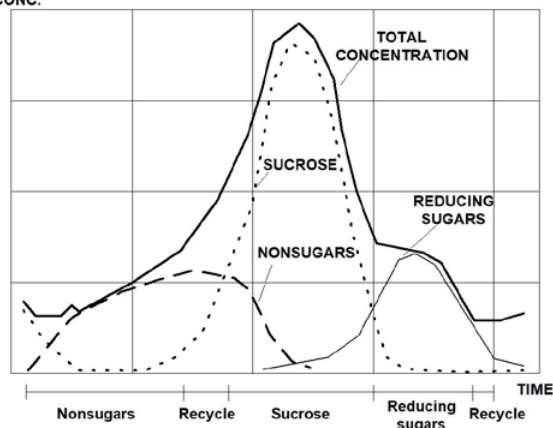



Figure 1. Chromatographic separation of molasses.

The sugar fraction leaves the separation column at about 30 Brix. After that, it can be evaporated to higher concentrations for further use. The salts and non-sugar fraction with 4 to 8 Brix can be evaporated and used as animal feed.

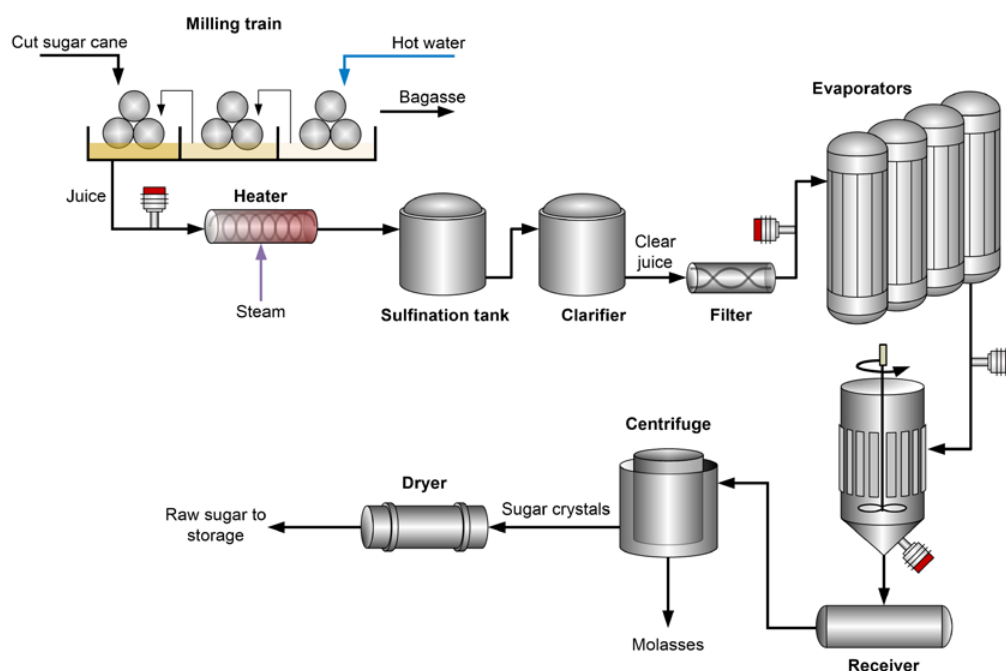
### Instrumentation and installation

Vaisala K-PATENTS® Refractometer PR-43 provides in-line and real-time Brix measurement to improve the chromatographic separation and successfully recover betaine.

The PR-43 provides Ethernet and 4-20 mA output signals that can be connected to the process control system for real-time process control. The information by the refractometer is used for the continuous calculation of sucrose purity and concentration. Because of its fast response time, the PR-43 is also used to determine exact cut-off points for the recovered fractions to increase productivity, and ensure high quality of the product.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Prism wash systems	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, water (or solvent) and high-pressure water.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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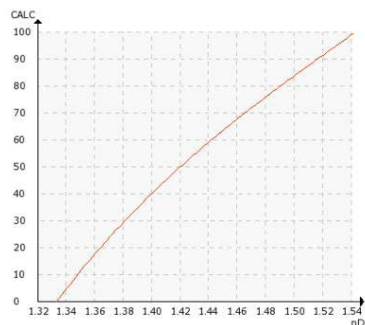


## MILLED CANE SUGAR

### Typical end products

Sugar and syrup for sweetening soft drinks, beer brewing, preserves, beverage, sweets, liqueurs, ethanol, etc.

### Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



## Introduction

After sugar cane has been harvested, it must be processed in under 24 hours to avoid sugar loss by inversion of sucrose to glucose and fructose. Traditionally, sugar cane processing requires two stages:

1. Milling extracts raw sugar from freshly harvested cane and sometimes bleaches it to make *mill white sugar* for local consumption.
2. Refineries are often located close to consumers in North America, Europe and Japan to produce refined white sugar. These two stages are slowly merging into combined milling and refining.

## Application

### Preparation and extraction

The mill washes, chops and shreds the cane mechanically. Shredded cane is repeatedly mixed with water and crushed between rollers. The collected juices contain 10–15 Brix and the remaining fibrous solids, called *bagasse*, are burned for fuel. About 93% of the juice is extracted. Water and weak juice from the last mill are added to help soften the cane and to aid in the extraction. The surplus bagasse can be used in animal feed, paper manufacturing or as a fuel for commercial electricity generation.

### Heating

The juice is sent to multiple heaters, where the sugar content is increased to 16–17 Brix.

### Sulfitation and clarification

Sulfur dioxide is added to the juice to remove impurities and to decolorize it. After that, lime is added to precipitate impurities and to help remove coloring matter, organic acids and other suspended materials. The limed juice is sent to clarification to settle. The clear juice goes to the evaporation plant.

Rotary filters are generally used to recover the sugar from the settled-out mud.

#### Evaporation

The clarified juice is concentrated in a multiple-effect evaporator to make syrup at about 60 Brix.

#### Crystallization

The thick juice syrup is further concentrated under vacuum until it becomes supersaturated and is then seeded with crystalline sugar. On cooling, more sugar crystallizes from the syrup.




#### Centrifuging and drying

A centrifuge separates the sugar from the molasses. Additional crystallizations extract more sugar and the final residue is called *blackstrap*. After drying the crystals, the color of the raw sugar varies from

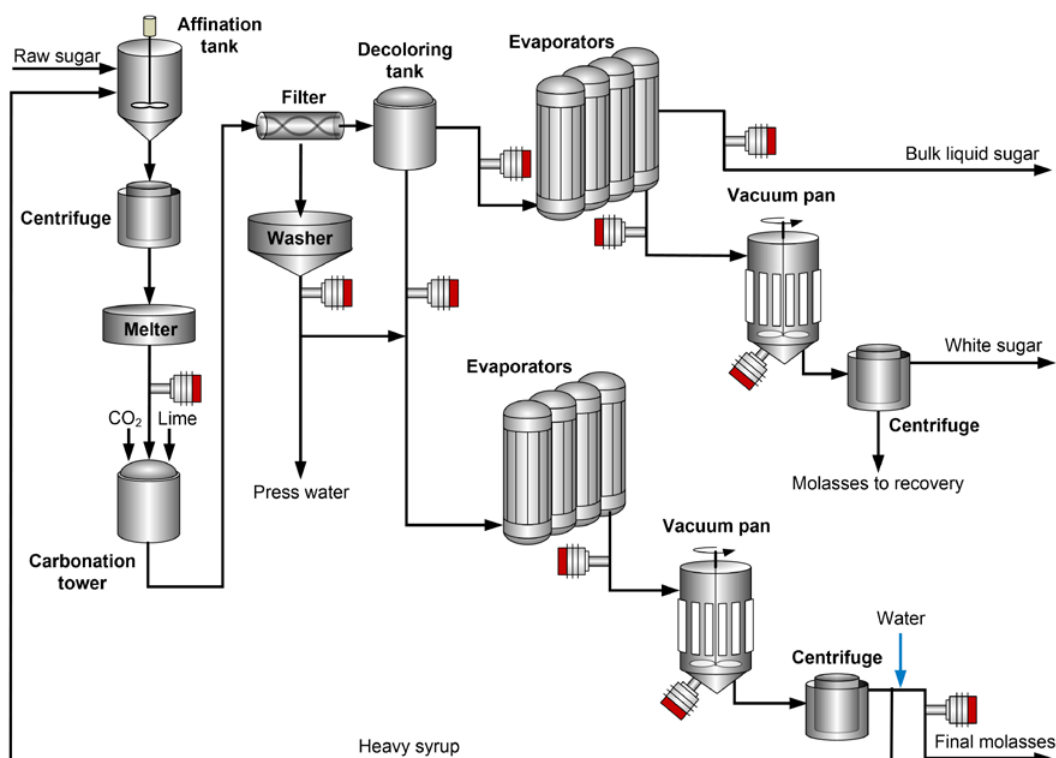
yellow to brown. Bubbling sulfur dioxide, through the cane juice before evaporation, bleaches most color-forming impurities into colorless ones. This sulfitation produces sugar known as *mill white*, *plantation white* and *crystal sugar*. These are the most commonly consumed sugars in sugar cane producing countries.

#### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 is used for real-time control at various stages in the cane sugar process. Our unique digital technology combined with refractometer's sturdy design results in highly accurate and reliable measurements that give improved control over the complete process.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.



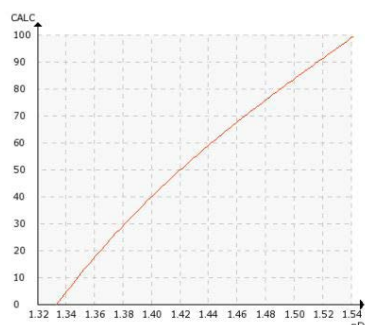


## REFINED CANE SUGAR

### Typical end products

Sugar and syrup for sweetening soft drinks, beer brewing, preserves, beverage, sweets, liqueurs, ethanol, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

The raw sugar received by a refinery contains 96.5 to 98.5 % sucrose and 1.5 to 3.5 % impurities which comprise organic matter, inorganic compounds, water and micro-organisms. These impurities make the raw sugar highly colored.

The sugar needs to go through a series of steps in a refinery to lighten it in color and make it suitable for human consumption.

### Application

The first step in the sugar refining process is called *affination*, wherein the raw sugar crystals are treated with heavy syrup (typically 60-80 Brix) to remove the film of adhering molasses. This strong syrup dissolves little or none of the sugar but softens or dissolves the coating impurities. The mixture, called *magma*, is spun in centrifuges and washed with hot water to remove the adhering molasses film.

The washed raw sugar crystals are then dissolved in water and diluted to about 70 Brix.

During carbonation the syrup is mixed with milk of lime and reacted with carbon dioxide to produce a precipitate of calcium carbonate (chalk). The chalk precipitate entraps organic non-sucrose and inorganic impurities.

Pressure filters are used to remove the chalk precipitates and to produce clear, light brown syrup.

The brown syrup is then passed over a series of acrylic and styrene resin columns and granular activated carbon columns. The resulting low colored syrup (fine liquor) is used for crystallization of white sugar or to produce bulk liquid sugar.

The water content of the fine liquor is reduced by multiple-effect evaporation, before it is fed to vacuum boiling pans. Crystallization is initiated by seeding the concentrated liquor with slurry. The process is continued until the crystals reach the desired size. The resultant mixture of crystals and mother liquor is fed in centrifuges and the sugar crystals are washed with hot water to remove any adhering syrup.




## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 is used at several stages in the refining process. The measurements taken are unaffected by entrapped air bubbles, undissolved components or color variations in the product, which are typical sources of error in other measurement instruments.

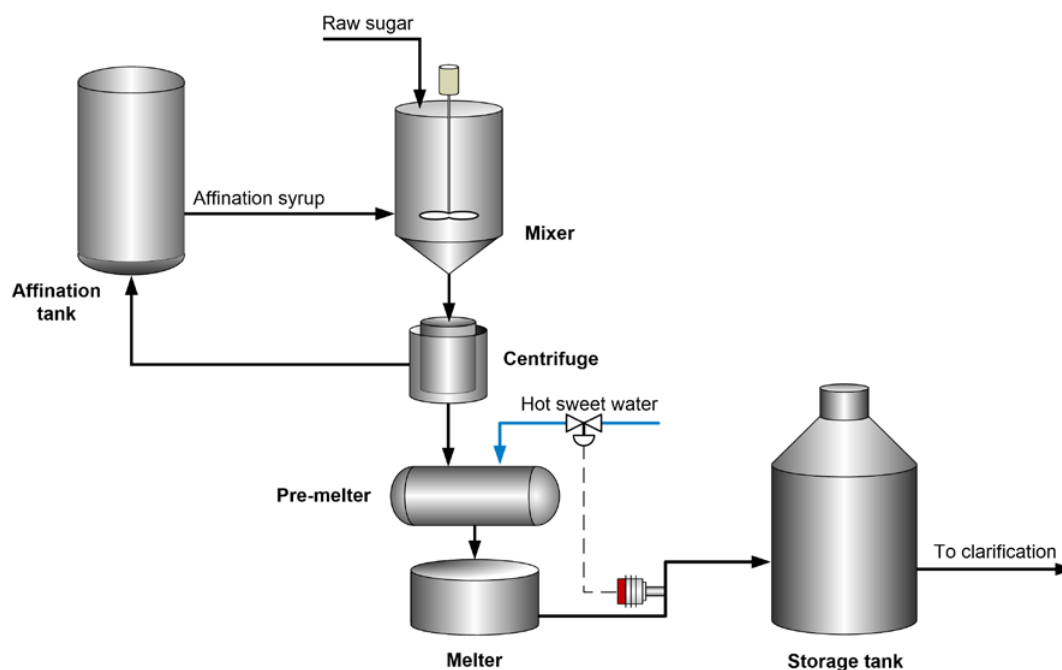
Accurate and real-time concentration information facilitates process control, increases productivity and improves product quality.

With the refractometer the affination, decolorization, evaporation, crystallization and recovery operations can be closely monitored and optimized.

Reliable monitoring is particularly important for the control of the crystallization pans. Good control over the crystallization process ensures consistency in crystal size and increases the yield. This eliminates the need for recycling and reprocessing which can considerably increase operation costs. Furthermore, the growth of false grain can be avoided which reduces the need for product screening.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
<b>User Interface</b>	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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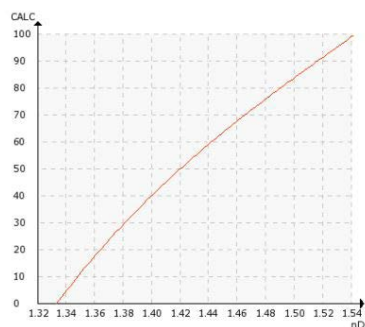


## CANE SUGAR JUICE

### Typical end products

Sugar and syrup for sweetening soft drinks, beer brewing, preserves, beverage, sweets, liqueurs, ethanol, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Sugar refining is the production of a higher quality sugar from re-melted raw cane sugar. The first step in refining is affination, where the raw sugar crystals are mixed with a heavy syrup (typically 60 to 80 Brix) to remove the residues of molasses. The syrup is used as a washing solution for the sugar crystals.

### Application

Raw sugar and affination syrup are mixed together. During this mixing, the molasses film on the crystal surface is softened and partly dissolved. The crystals are then separated from the syrup in centrifuges


and washed by a water spray. After centrifuging, the sugar crystals are dissolved in hot sweet water in the melter and diluted to a 54 Brix solution. The liquor is screened to remove coarse impurities and it moves on in the process for clarification and decolorizing.

### Instrumentation and installation

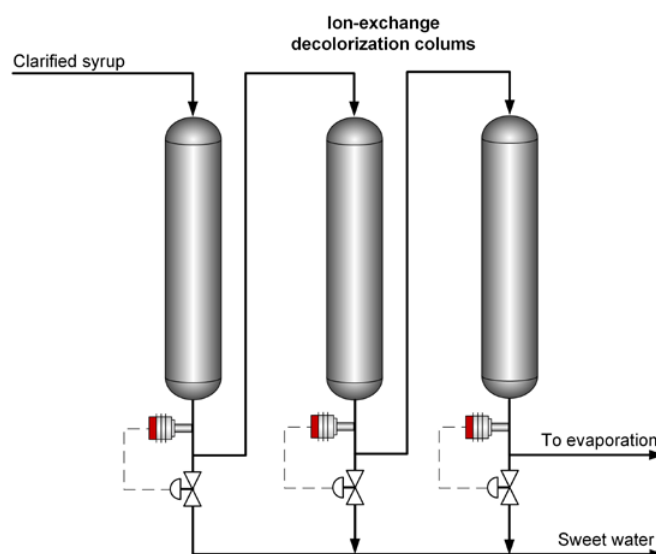
Vaisala K-PATENTS® Process Refractometer PR-43 can be installed directly in the pipeline after the melter, or in a recirculating line to monitor in real-time the concentration of the liquor to clarification.

The refractometer provides Ethernet and 4-20 mA output signals that can be used for automatic control of the dilution operation to ensure the target concentration is achieved. Typical measurement range in this application is 40-80 Brix, and typical process temperature is about 60 °C (140 °F).

The refractometer facilitates the monitoring and control of the affination process ensuring the optimal operation of the subsequent processes.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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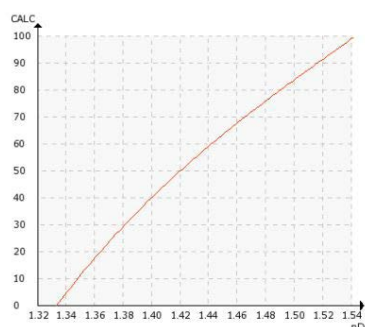


## CANE SUGAR SYRUP, SWEET WATER

### Typical end products

Sugar and syrup for sweetening soft drinks, beer brewing, preserves, beverage, sweets, liqueurs, ethanol, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Color is an important parameter in the sugar refineries. It is an important specification for raw sugar as well as for the refined products.

The decolorization process removes organic impurities, which impart color to the sugar liquor. These colorants are soluble impurities which are removed by adsorption processes. Typical adsorbents are granular activated carbon, powdered carbon and ion-exchange resins.

Ion-exchange resins are preferred as they can be regenerated chemically and wet, which reduces fuel consumption considerably, thus providing a significant economic benefit.

### Application

The clear brown syrup passes through a series of columns, known as *ion-exchange columns*. The adsorbents can be for example, acrylic and styrene resin and granular activated carbon, but other resins and/or resin combinations can be used depending on the liquor quality.

The resulting light-colored syrup or fine liquor is used for white sugar crystallization or to produce bulk liquid sugar once it has been further purified.


The resins in the columns require periodical *backwashing* for their regeneration. After decolorization, hot water is pumped into the column and the discharge flow is directed to the sweet water line. Once regeneration is completed, the sweet water line is closed and decolorization continues.

### Instrumentation and installation

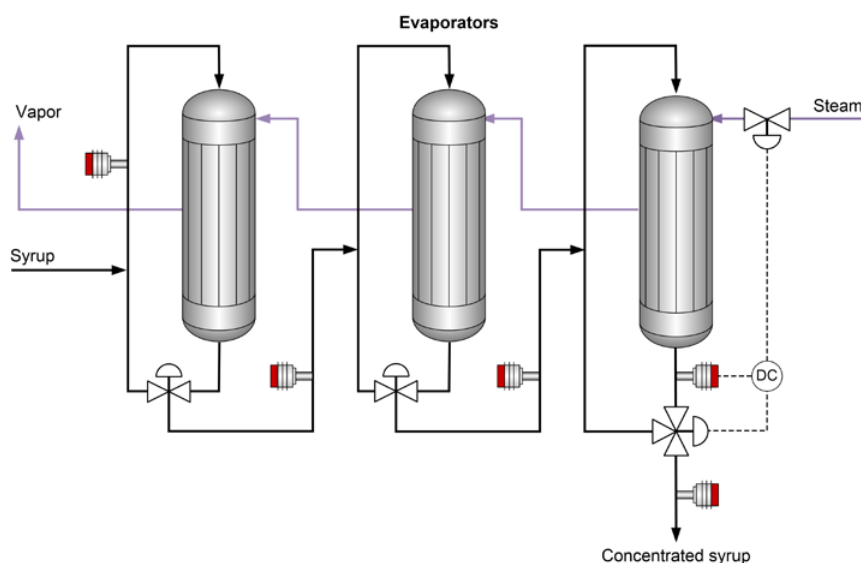
Vaisala K-PATENTS® Process Refractometer PR-43 is installed in the sweet water line for washing control. The refractometer accurately measures in real-time the concentration of the liquid to detect instantly the interface between the syrup and the washing water. Typical measurement range is 0-20 Brix and typical process temperature is about 60 °C (140 °F).

The refractometer's output signal can be used to control the operation of the discharge valve in response to the process. This allows automated control of the regeneration process, increasing yield and avoiding production losses.



Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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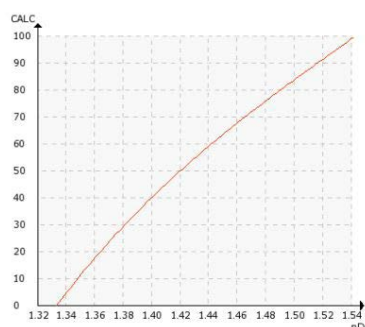


## CANE SUGAR SYRUP

### Typical end products

Sugar and syrup for sweetening soft drinks, beer brewing, preserves, beverage, sweets, liqueurs, ethanol, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



## Introduction

Evaporation is a crucial step in the sugar making process. The main purpose of the evaporation is to raise the sugar content in the juice to reach the saturated condition required for crystallization.

Evaporation usually takes place in a multiple effect evaporator heated with steam. Evaporators are the largest heat users and major contributors to losses in sugar cane factories. These factors make effective evaporator control crucial to overall factory efficiency.

## Application

The *thin syrup* (light-colored syrup) from the extraction plant is evaporated in multiple effect evaporators to concentrate it and to obtain the syrup which is fed to the pan station (crystallization). The dissolved solids concentration is raised from an initial concentration of

10-15 Brix to 50-75 Brix. The concentrated solution is known as *heavy syrup*.

Control of evaporation is crucial for the sugar factories. The main objectives for an evaporator control system are control of Brix and a smooth operation.

An optimally concentrated syrup improves the operation downstream and ensures a high product quality. If the syrup fed to the crystallizers exceed its limit, spontaneous nucleation can happen producing crystals out of specification which require re-processing and increase the operating costs.

Good evaporation control also allows the evaporators to smooth out process fluctuations and to adjust automatically and immediately to the changes in the syrup flowrate.

## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 is the ideal instrument for controlling sugar factory operations. The refractometer can be installed directly in the process line for continuous monitoring the Brix of the juice and syrup throughout the entire process. The Brix measurement by the refractometer is accurate and reliable and it is not affected by the presence of undissolved particles, crystals or bubbles.


The refractometer is installed in the inlet and outlet of evaporation as well as intermediate evaporation stages. The information given by the refractometer helps to optimize the process and ensure high-quality product for the downstream factory.

The refractometer provides Ethernet and 4-20 mA output signals that can be used for real-time process control. For example, the refractometer's signal can be used to adjust the feed of syrup to the capacity of the evaporation plant by using a feed-forward control. It is also possible to use the signal to regulate the quantity of heavy syrup added to the feed syrup, thus keeping the feed concentration to the evaporation plant constant.

In the intermediate stages, the refractometer is used in a control loop that keeps the syrup concentration constant by regulating the steam flow, or by regulating the amount of syrup returned to the evaporator.

The refractometer also helps to maximize the storage capacity by maintaining automatically the concentrated syrup at a high-level of solids.

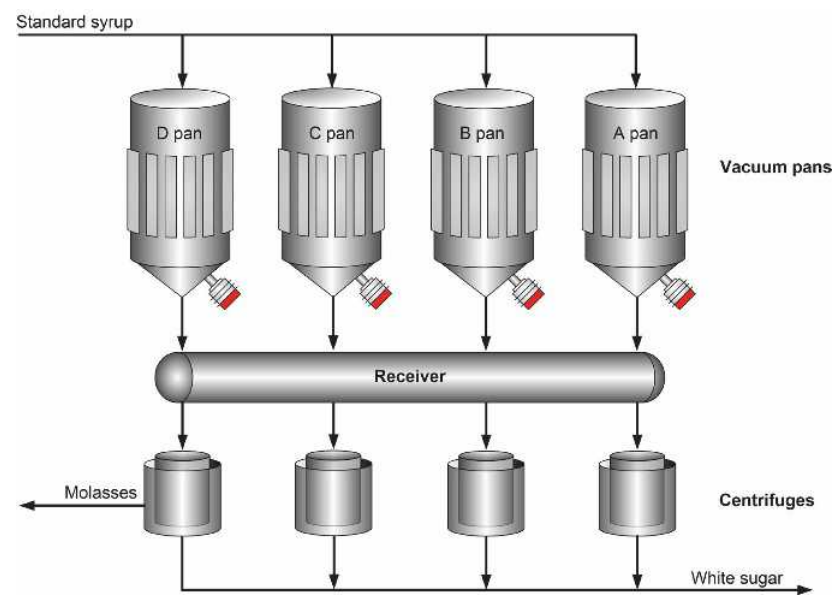
In some plants, the heavy syrup contains additives and supersaturated impurities (e.g. oxalic acid), which may crystallize on the prism. Therefore, an automatic steam wash is required. In some extreme cases, it is preferable to mount the refractometer in a narrow by-pass line downstream of a small cooling heat-exchanger, where the impurities crystallize. Typical measurement range in this application is 50-80 Brix.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Prism wash systems	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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## Cane sugar syrup

Cane sugar crystallization | 1.02.04



### Measured medium

- Sugar and sweeteners

### Typical end products

- Sugar and syrup for sweetening soft drinks, beer brewing, preserves, beverage, sweets, liqueurs, ethanol, etc.

However, two sensors can provide information on the important massecuite parameters. These sensors are not influenced by other process parameters:

1. Vaisala's process refractometer. The refractive index technology is successfully used for selective concentration measurement of the liquid phase over the complete crystallization strike.
2. A sensor for measurement of the total solids content (brix of the massecuite).

## Introduction

Crystallization is the final stage in a cane sugar mill and it refers to the formation of the sugar crystals from the concentrated syrup.

Crystallization takes place in vacuum boiling pans. These vacuum pans are the heart of the sugar manufacturing plants. The efficiency of the factory, the quality of the sugar product, the capacity of the plant and the thermal balance, all depend on the operation and control of the vacuum pans to produce a high quality massecuite.

## Application

The *thick juice or syrup* is fed to the vacuum pans and evaporated until saturated. Crystallization is initiated by adding (or seeding) fine sugar crystals to the pan, or *strike*. The crystals start to grow, and the process is continued until

the crystals reach the specified size.

The resultant mixture is known as *massecuite* (raw sugar crystals and molasses). The sugar crystals are separated from the molasses by centrifugation and are then washed with hot water to remove any adhering syrup.

## Control of crystallization

Crystallization can only take place if the solution is *supersaturated*. Supersaturation is a multivariable function of several parameters of the liquid phase only (syrup or mother liquor). Crystals can grow only if supersaturation is higher than 1.0.

No single instrument can provide on-line data on supersaturation. Conventional sensors used to monitor crystallization provide data of a single massecuite parameter only.

Control of supersaturation is critical to the final outcome of the strike. The number of crystals should remain constant from the seeding until the end of the strike. If the supersaturation drops outside its limit, the crystals will stop growing and might even melt. If the supersaturation level rises too high, new crystals will form spontaneously. Spontaneous nucleation results in poor quality crystals of irregular shape and size (fines and conglomerates) which require reprocessing.

## Instrumentation and installation



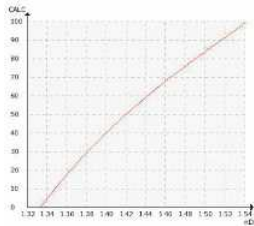
Zutora SeedMaster-4 is a unique, fourth generation crystallization transmitter and seeding device to be used with Vaisala's process refractometer. The SeedMaster-4 allows for accurate in-line and real-time monitoring of supersaturation and crystal content over the complete process of crystallization, and implementation and control of automatic or manual seeding. The SeedMaster-4 can be connected to one or two process refractometers and to one or two crystallizers.

The SeedMaster-4 provides the following:

1. Electronic data capture on massecuite parameters.
2. On-line calculation and transmission of massecuite parameters for the advanced control of sugar crystallization with control system.
3. Organization and storage of strike history data archive.
4. Advanced communication with the control system.
5. Automatic seeding of the vacuum pans.
6. Serves as user interface for the pan and control system operators.

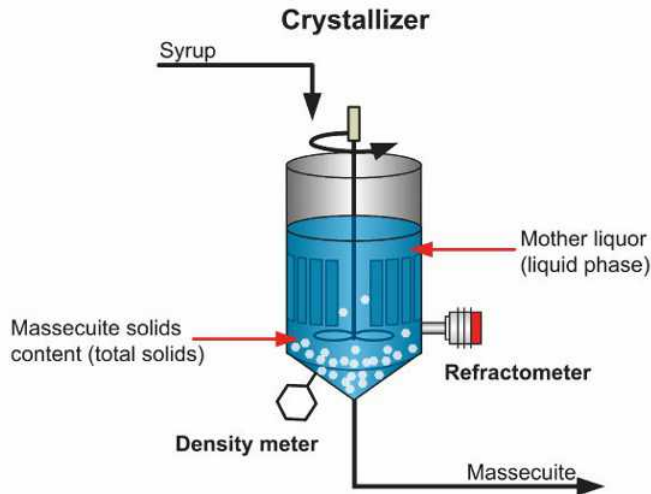
The refractometer is installed directly into the crystallizer. The measurement is accurate and reliable and the prism remains clean because of crystal friction. Typical measurement range in this application is 65-90 Brix.

Control of crystallization with the refractometer increases productivity, reduces the need for crystal re-processing and guarantees a high-product quality.

Instrumentation	Description
	SeedMaster-4 for multiparameter sugar crystallization monitoring and automatic seeding.  Crystallization transmitter and seeding device is used with the Process Refractometer PR-23-GP. It allows for accurate in-line and real-time monitoring of supersaturation and crystal content over the complete strike, and implementation and control of automatic or manual seeding.
	Process Refractometer PR-23-GP is an industrial refractometer for crystallizers. Installation through a flange connection and Counter flange adapter -AP for vacuum pan installations.
<b>Prism wash system with warm water</b>	The integral prism wash system helps to avoid crystals' deposit or scaling on the prism surface. Prism wash system components are a refractometer with integral water wash nozzle mounted at the refractometer probe, a warm feed water source (hot condensate), and an Indicating transmitter with build-in relays for driving the water valve and controlling the wash.
<b>Measurement range</b>	Refractive Index (nD) 1.3200–1.5300, corresponding to 0-100 Brix.
<b>Chemical curve</b> 	R.I. per BRUX at Ref. Temp. of 20 °C

# Beet sugar, cane sugar

## Sugar crystallization: massecuite solids content and mother liquor concentration



### Particularities of the crystallization process

Varying process conditions present a challenge to measuring massecuite solids content accurately. Process medium changes during different production phases from liquid to massecuite and consists of both liquid and crystals. Generally, calibrating the massecuite solids content meter can be quite easy either for the liquid phase or for massecuite phase, but not for both. This means that the massecuite solids content sensors cannot solely produce reliable results as they do not cover the whole processing range.

For accurate results, the calibration must cover the full range from pure liquid to the point where Vol. 55 % of the massecuite is crystals. However, if the vacuum pan is also equipped with a refractometer, the calibration difficulties can be mostly avoided.

In the beginning of the strike the process medium is pure liquid. At this point the refractometer and the massecuite solids content meter should give the same measurement value (Figure 1).

The crystals are introduced only after the pan has been seeded. After seeding the massecuite solids content increases as the crystal content increases, whereas refractometric concentration stays rather constant ( $\pm 3$  Brix). Improved accuracy in massecuite solids measurement can be achieved when a refractometer is combined with a microwave sensor. Moreover, a refractometer can offer calibration reference value for the

### Introduction

This note explains the methods and calibration procedure for measuring successfully the massecuite solids content and mother liquor concentration in sugar vacuum pans over the entire crystallization process. Both parameters need to be measured and controlled, as they influence the quality of the sugar crystals.

Massecuite solids content, or total sugar content, is typically determined using e.g. microwave measuring technology whereas mother liquor concentration (dissolved sugar) is measured with a refractometer. The common measurement scale is Brix.

### Refractometer

Vaisala K-PATENTS® Process Refractometer is successfully used for selective measurement of liquid phase over the complete crystallization strike. Due to the unique digital principle, the refractometer measures the true concentration of the mother liquor,

without being influenced by the sugar crystals or bubbles in the pan. Moreover, the refractometer does not require re-calibration.

### Massecuite solids content meter

A microwave sensor can measure only the total solids (liquid and undissolved solids phase). Microwave probes are based on the measurement of attenuation and phase shift of microwave radiation. Both are related to the length travelled by the radiated signal, and the density and dielectric characteristics of the medium.

Phase shift is the result of decreasing speed of propagation. Due to the fact that water has a high dielectric constant compared to sugar and the accompanying non-sugars, the water content (and, consequently, solids content) is the major parameter, which determines the dielectric properties of the medium. As an output the microwave probes provide density or solids content of the massecuite.

massecuite solid content meter at the seeding point.

The recommended control practice is to use a refractometer to measure the concentration from the beginning of the strike until the seeding point. The refractometer provides accurate and repeatable seeding.

## Calibration procedure

The process refractometer is factory calibrated according to International Commission for Uniform Methods of Sugar Analysis (ICUMSA) Brix table. The factory calibration should be verified against the production laboratory when commissioning the instrument. The laboratory reference values should be taken in the beginning of the strike when there is only liquid present in the vacuum pan. Only a small BIAS adjustment might be needed to match the refractometer and the production laboratory.

Typically, the massecuite solids content meter needs regular calibration and calibration checks. The best calibration result is typically achieved when the microwave density meter is calibrated on a narrow range for either liquid sugar or massecuite. In vacuum pans the recommended

calibration practice is to calibrate the microwave density meter from seeding point to the end of the strike, which means that the reference samples should be taken at the seeding point, end of the strike and one sample in between (minimum three samples).

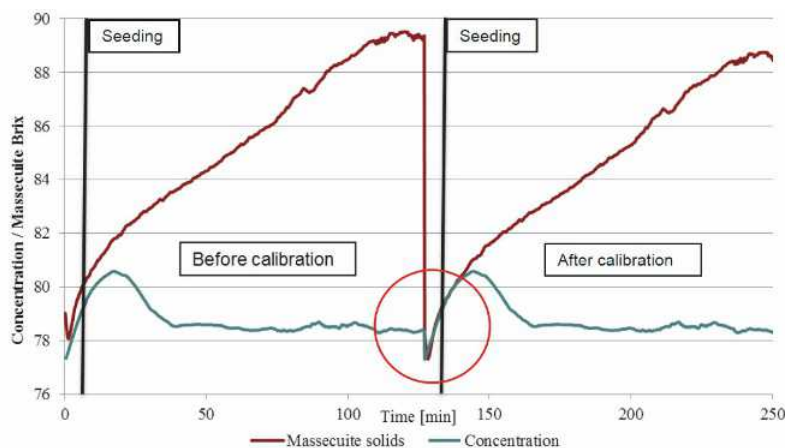


Figure 1. Refractometer and massecuite solids content measurement trends before and after calibration. The liquid concentration and massecuite solids content should be the same before seeding. After seeding they separate as the crystals start to grow.

Table 1. Example of calibration table.

Sample	LAB (Mass.Sol.Cont)	Refractometer (Concentration)	Microwave (Mass.Sol.Cont)	Difference refractometer -LAB	Difference microwave/nuclear -LAB
1	77.6	77.5	78.5	-0.1	+0.9
2	79.5 (seeding)	79.4	80.9	-0.1	+1.4
3	85.6	78.5	86.3	-	+0.7
4	90.5	77.9	91.3	-	+0.8

Table 2. Example of calibration table after calibration procedure. Refractometer offset adjustment +0.1 Massecuite solids meter offset adjustment -0.9.

Sample	LAB (Mass.Sol.Cont)	Refractometer (Concentration)	Microwave (Mass.Sol.Cont)	Difference refractometer -LAB	Difference microwave/nuclear -LAB
1	77.6	77.6	77.6	0.0	0.0
2	79.5 (seeding)	79.5	80	0.0	+0.5
3	85.6	78.5	85.4	-	-0.2
4	90.5	77.9	90.4	-	-0.1

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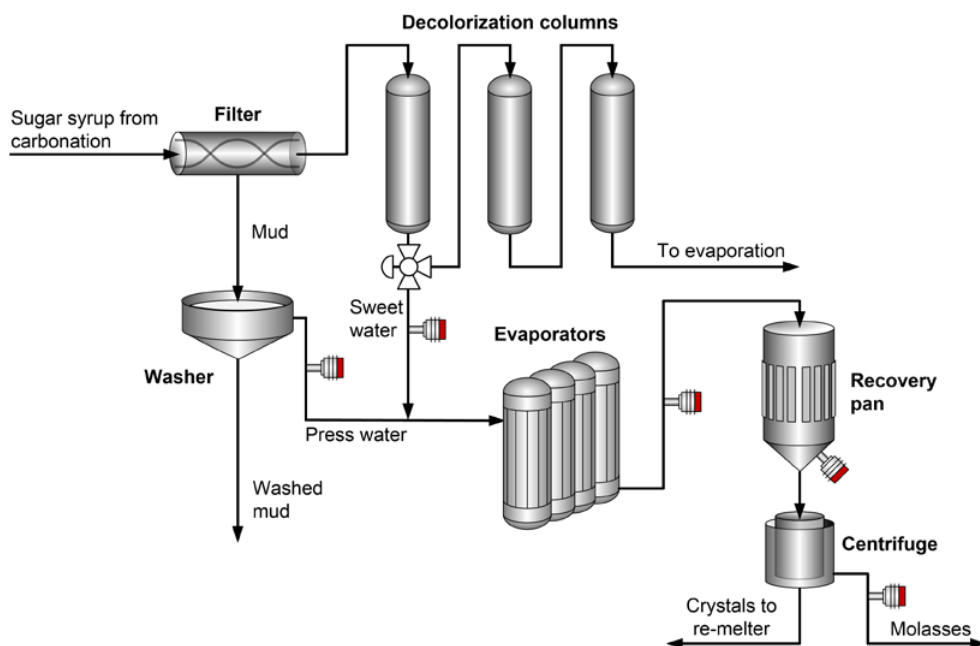


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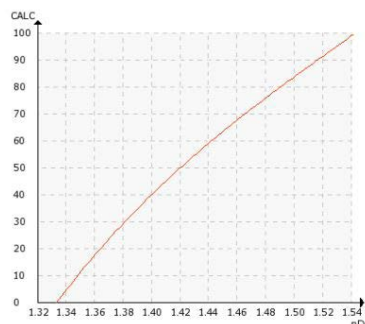


## CANE SUGAR SYRUP, MOLASSES

### Typical end products

Sugar and syrup for sweetening soft drinks, beer brewing, preserves, beverage, sweets, liqueurs, ethanol, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Sugar is recovered from the syrup by repeated crystallization in vacuum boiling pans. The resulting mixture of crystal and liquor is known as the *massecuite*. The white sugar crystals are separated from the liquor in centrifuges and then dried in drum dryers. The residual liquor or syrup moves on to the next crystallization step.

At the last crystallization step, the low-quality sugar is circulated back for remelting and reuse, and the final syrup known as the *final molasses* is sent to recovery.

This residual product still contains a high amount of soluble sucrose. Sugar factories aim to maximize productivity by recovering the sucrose from the

molasses. Control of this process is of outmost importance as it is crucial factor in the economic viability of the factory.

### Application

The final molasses is a heavy, viscous liquid with a high content of sucrose, but from which sugar can no longer be crystallized.

Some refineries sell the molasses as a valuable by-product for cattle feed or fermentation. However, because of its sucrose content, molasses can also be further treated with different techniques to recover more sugar, e.g. by ion exclusion or liquid chromatography.

### Instrumentation and installation


Vaisala K-PATENTS® Process Refractometer PR-43 is installed at various stages for monitoring and controlling the recovery operations. The refractometer measures sugar content in the entire range from 0-100 Brix, making it suitable for different applications such as:

1. Together with the SeedMaster SM-3, determining the seeding point and monitoring the drop of mother liquor concentration after seeding in the recovery pan.
2. Monitoring the final molasses to ensure that the concentration complies with the buyers' specification. Typical measurement range is 60-90 Brix.

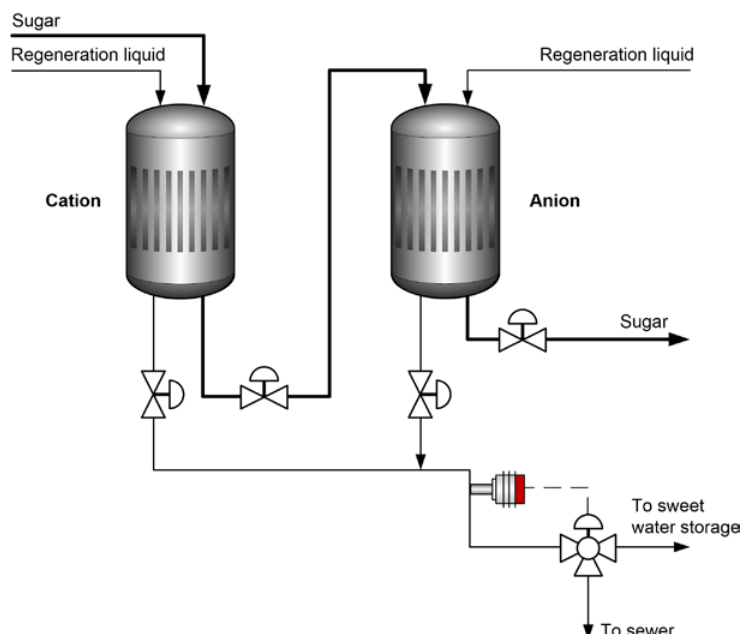
3. Dilution control to maintain the concentration at a constant level and the control of separation for the molasses.

4. Monitoring the waste water line of a sugar cane refinery to detect inadvertent leakage of sugar into the waste water stream. Typical measurement range is 0-5 Brix.

The refractometer provides Ethernet and 4-20 mA output signals that can be integrated to the factory's control system for real-time process control. Accurate and reliable measurements help to maximize the recovery of sucrose and to maintain the economic viability of the factory.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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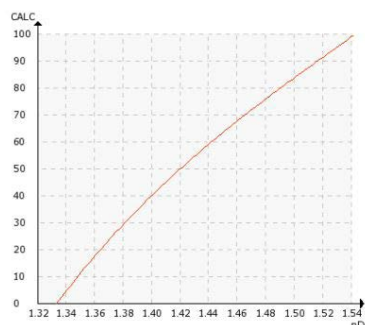


## SWEETENERS: DEXTROSE, FRUCTOSE, GLUCOSE SYRUP, SORBITOL

### Typical end products

Sweeteners for beverages, beer brewing, jams, preserves, sweets, confectionery, ice cream, liqueurs, pharmaceuticals, etc.

Chemical curve: R.I. per BRUX at Ref. Temp. of 20 °C



### Introduction

Sweeteners are produced by converting the starch components (i.e. glucose) into compounds with the same chemical form (i.e. fructose) by isomerization.

The isomerization reaction requires some divalent cations from salts for catalytic activity. On the other hand, some cations may inhibit the reaction.

Proper demineralization of the liquor prior and after isomerization is essential. Demineralization before the reaction enhances the reaction rate. After isomerization it removes the trace components picked up during isomerization to ensure high product quality.

### Application

Demineralization is usually performed through anion and cation exchange resins. The decolorized syrups are treated in ion exchangers to remove the salts and ionic content that increased, when chemicals such as acids, bases and calcium were added during the process.

Six-bed ion-exchangers are the most common ones. They have three beds of cation and three beds of anion columns. One bed is always at the regeneration phase. The cation resin is used in the hydrogen form ( $H^+$ ), thus, it must be regenerated with an acid, in this case, with a strong Hydrochloric acid  $HCl$ .  $HCl$  controls the pH of process water streams providing water purity, demineralizes water and rinses cations from the resins. The cation resin removes hardness from water while it is also alkaline.

The anion resin is used in hydroxyl form ( $OH^-$ ), thus, it must be regenerated with a strong alkali, such as sodium hydroxide ( $NaOH$ ). Alkaline environment softens the solution by removing strong acids.

Before regeneration can take place, the columns must be rinsed with water to remove any sugar residue. This operation is usually referred to as *sweetening-off*.

## Instrumentation and installation


Vaisala K-PATENTS® Process Refractometer PR-43 monitors the interface between the product and the rinse water, which is pumped through the ion exchange columns prior to the regeneration process. Typically, the refractometer sensor is installed in the waste line, before the diversion valve. Because of its fast response, the refractometer instantly detects the liquid in the pipe (due to its unique refractive index value) and ensures it is directed to the right line.

The refractometer's output signal can be used for process control. If the dissolved solids concentration in the process stream increases above a set point (typically from 0.5 % to 2.0 %), the stream is

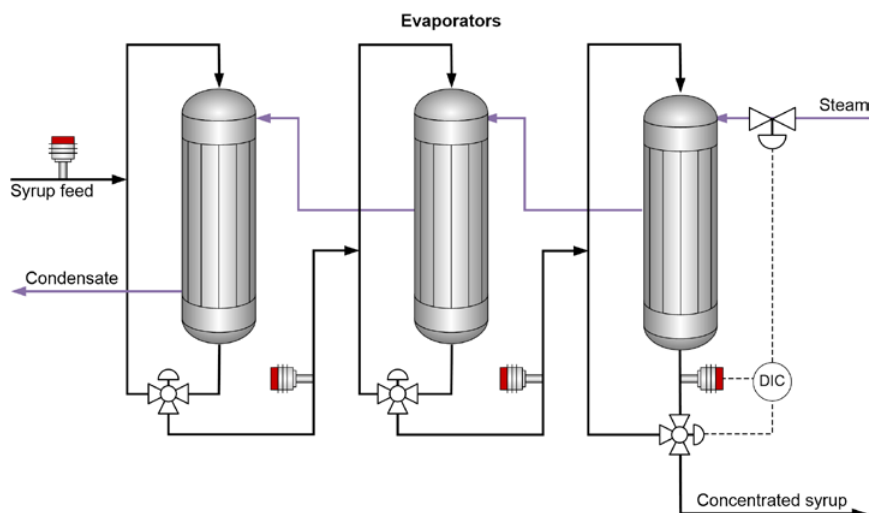
automatically diverted to the sweet water storage. At below the set point, it is released into the sewer.

Optical analyzers typically have an extremely short response time to fluctuations in product concentration. The refractometer detects the cutting point rapidly ensuring faster operation and increased productivity.

The typical measurement range after the ion exchange is 0-30 Brix and the process temperature is about 45 °C (113 °F).

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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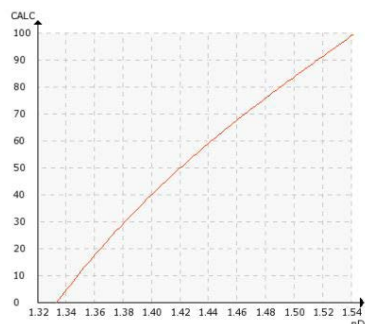


## SWEETENERS: DEXTROSE, FRUCTOSE, GLUCOSE SYRUP, SORBITOL

### Typical end products

Sweeteners for beverage, beer brewing, jams, preserves, sweets, confectionery, ice cream, liqueurs, pharmaceuticals, etc.

### Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Evaporation is used at different stages of the process, for example, for concentration and recovery of sweetener from sweet water, or for concentrating the syrup to the final product specifications.

Evaporation is one of the largest heat consumption operations in the starch industry. This explains why one of the major activities in the industry is the development of methods for optimizing evaporation and reducing energy consumption.

### Application

Falling film evaporators, with two or more effects, are frequently used by the liquid sweeteners industry. The evaporators with up to seven effects have been employed since the mid-seventies to offset rising energy costs. Evaporators are used to remove excess water from syrups and to raise the concentration to a predetermined level.

There are generally four variables involved in the equation used for evaporation control: inlet concentration, outlet concentration, steam flow and product flow. For the maximum efficiency and minimum steam consumption, all these variables must be coordinated.




### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 suits perfectly for evaporator control. Usually, one refractometer is used to adjust the syrup feed (according to concentration) to the capacity of the evaporation plant (feed-forward control). Typical measurement range for the evaporator feed control is 0-35 %.

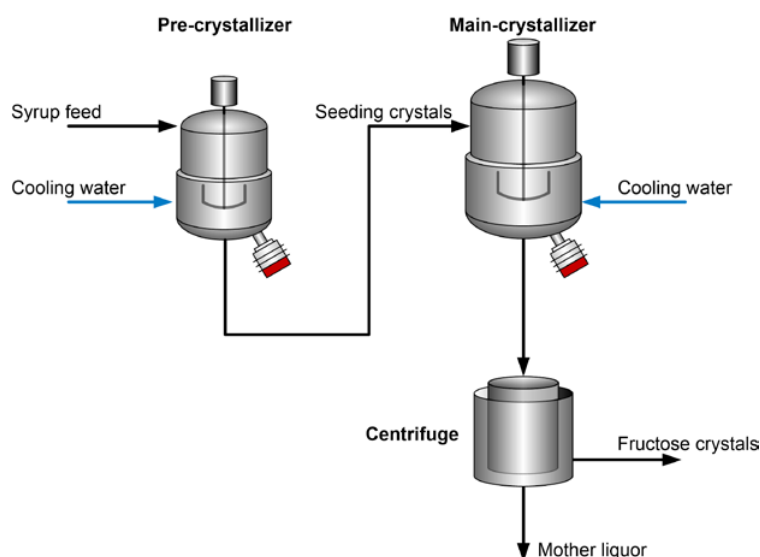
Another refractometer is used in the final effect outflow. It is typically installed in a control loop to keep the syrup concentration constant. This is done by adjusting the steam flow or the quantity of syrup returned to evaporation. Typical measurement range for the evaporator outlet control is 70-85%.

For optimized evaporator control, other refractometers can also be installed at the outflow for each single effect.

Precise concentration control at the evaporator outlet is very important for optimizing downstream processes, such as crystallization and adsorption separation.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
<b>User Interface</b>	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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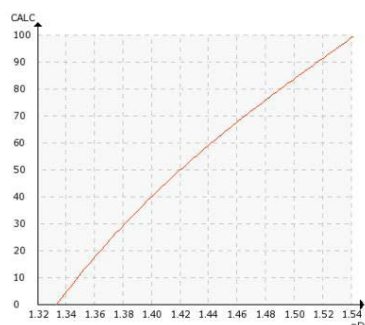


## SWEETENERS: DEXTROSE, FRUCTOSE, GLUCOSE SYRUP, SORBITOL

### Typical end products

Sweeteners for beverage, beer brewing, jams, preserves, sweets, confectionery, ice cream, liqueurs, pharmaceuticals, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

The starch industry can produce sweeteners as a syrup or as a crystalline product.

Crystalline starch sweeteners are used in the pharmaceutical as well as food and beverage industries because of their unique sweetness and physical and functional properties.

### Application

Cooling crystallizers are mainly used for the glucose and fructose syrups crystallization.

The solid level and temperature of crystallization depends on the type of carbohydrate. For example, fructose will only crystallize at 90 % dissolved solids.

The saturated syrup reaches supersaturation as the temperature slowly decreases. At this stage, the crystals start to form and grow. As the crystals grow bigger, the concentration of the mother liquid decreases because of the transfer of sweetener to the crystals. For optimal crystallization, the supersaturation condition must be maintained by addition of fresh syrup into the crystallizer.



### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 can be installed directly in the crystallizing pan for real-time control and monitoring of the liquid concentration.

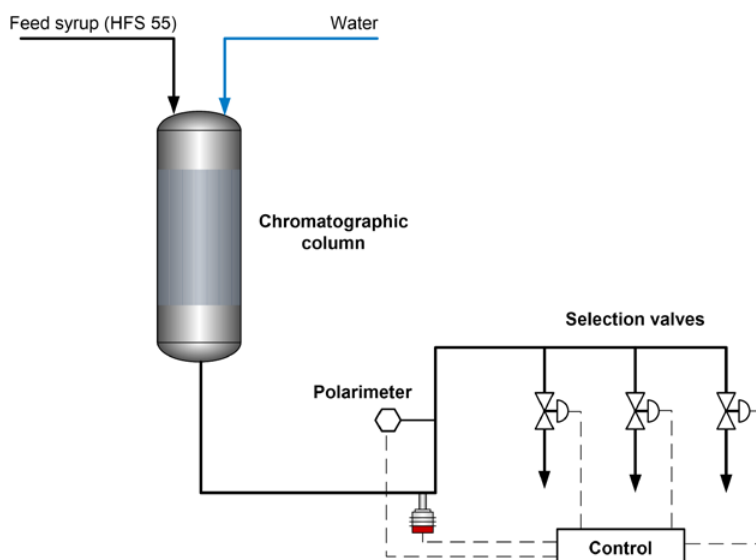
The refractometer provides important information for keeping the supersaturation constant at its optimal level. If the supersaturation drops below its limits, crystallization will stop, if the supersaturation rises at a high-level, crystals outside the specifications will form. The refractometer ensures high quality crystals for improved productivity and decreased operation costs.

The refractometer is ideal for controlling and optimizing sweeteners crystallization as the measurement is not affected by the crystals or bubbles and is selective to the liquid phase.



Instrumentation	Description
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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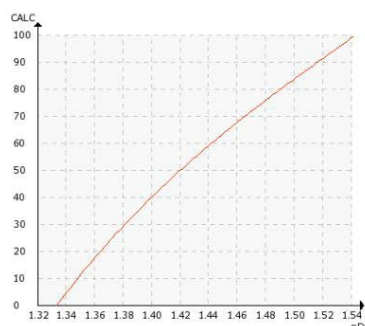


## SWEETENERS, FRUCTOSE, GLUCOSE (DEXTROSE)

### Typical end products

Sweeteners for beverages, beer brewing, jams, preserves, sweets, confectionery, ice cream, liqueurs, pharmaceuticals, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Conversion of glucose (dextrose) to fructose is economically limited to 42-46 % fructose on a dry basis. This concentration of fructose is useful for many applications, however, in some applications, 42 % high fructose syrup is not sweet enough and a higher level of fructose is necessary.

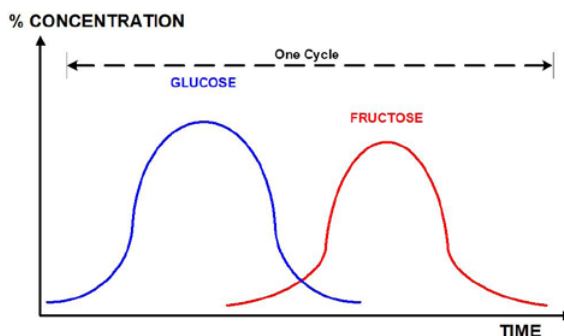
Using chromatographic separation, fructose concentration can easily be increased to over 90 %. Blending the high fructose cut with additional 42 % syrup results in a 55 % HFS product suitable as a sweetener for soft drink bottlers.

### Application

Fructose and glucose separation can be done with column-chromatographic technology.

First, the high fructose syrup (HFS) is fed to the fixed-bed separation columns filled with special absorbents, which absorb the fructose and glucose to different degrees (Figure 1). The glucose is the product, which leaves the columns first and can be separated from the fructose fraction.

With this technology high-purity (>99 %) glucose and high purity fructose (>90 %) can be produced.



**Figure 1.** Fractionation of glucose and fructose by chromatographic separation.




### Instrumentation and installation

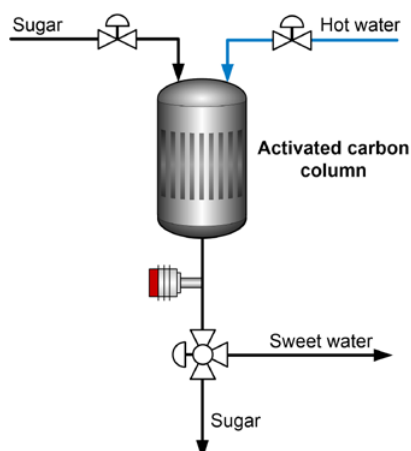
Vaisala K-PATENTS® Process Refractometer PR-43 provides the basis for accurate and reliable control of the chromatographic separation.

The refractometer is installed after the chromatographic column. The refractometer's signal is connected to the process control system together with the measurement of a polarimeter. The refractometer measures in real-time the total concentration of the

liquid and the polarimeter measures the fructose and glucose purity.

These two signals are sent to a computer, which calculates the fructose and glucose concentrations separately. The computer utilizes this information to control the selection valves, ensuring that the fructose and the glucose are fed into separate storage tanks.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

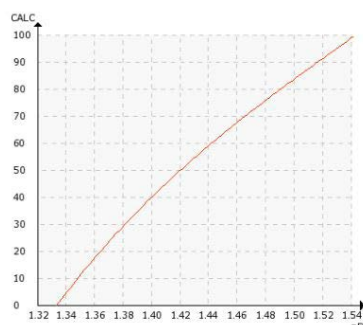


## SWEETENERS: PURIFIED FRUCTOSE, DEXTROSE (GLUCOSE)

### Typical end products

Sweeteners for beverages, beer brewing, jams, preserves, sweets, confectionery, ice cream, liqueurs, pharmaceuticals, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

After the saccharification and isomerization reactions the sweetener syrup needs to be clarified and purified. This is usually done by passing it through activated carbon beds.

The carbon treatment of the sweeteners is important as it will remove a major portion of the colored bodies, odoriferous, flavoring materials and other impurities that will affect their shelf life.

### Application

Carbon columns are used for the elimination of syrup impurities. Usually these columns contain packed granular carbon, although powdered carbon may also be used.

After the treatment the activated carbon beds become saturated and need to be reactivated.

For reactivation of the beds, hot water is pumped into these columns to remove the fine liquor. This operation is known as *sweetening off*.




After the hot water starts to break through the carbon bed, the discharge is directed to the sweet water line. When the concentration drops to 0.5 %, the column is ready for carbon transfer and reactivation.

### Instrumentation and installation

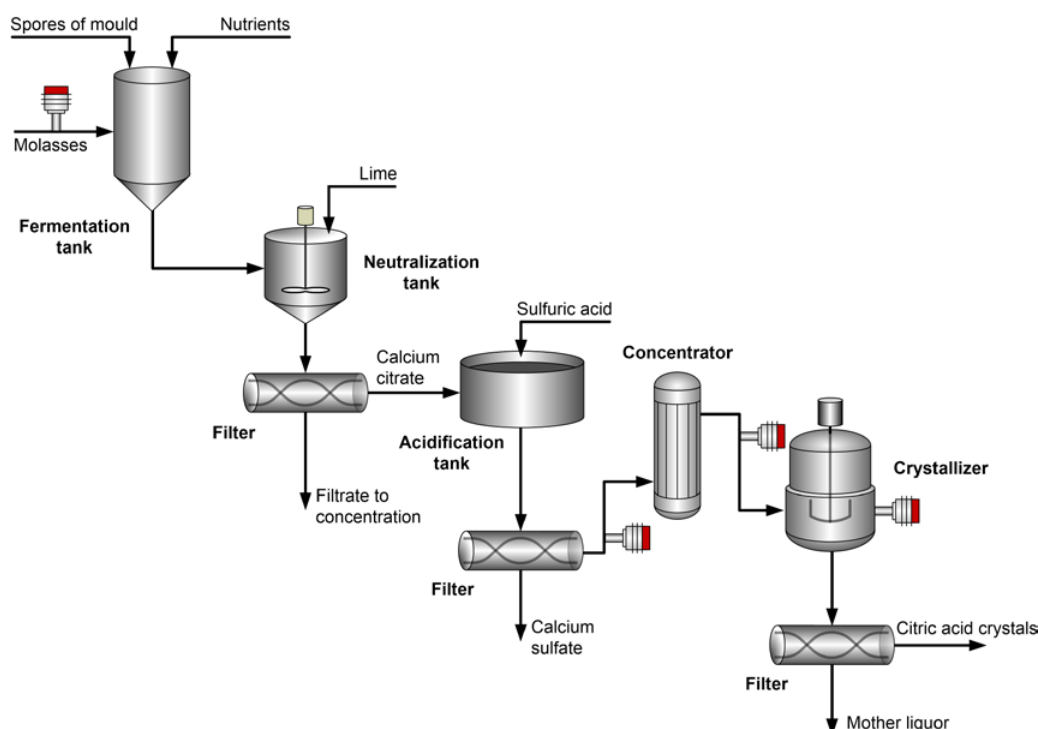
Vaisala K-PATENTS® Process Refractometer PR-43 is installed in the sweet water discharge line of the carbon column. The purpose of the in-line measurement is to provide a real-time signal to the process controller, enabling it to control and direct the flow as required.

A similar measurement is required when the column is placed on-line and used for draining.

Typical measurement range for carbon column control is 0-30 %. The automatic steam wash system is always required for this application.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Prism wash systems	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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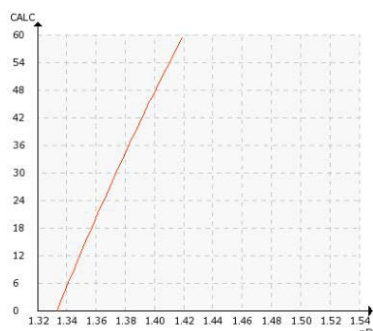


## CITRIC ACID (C<sub>6</sub>H<sub>8</sub>O<sub>7</sub>)

### Typical end products

Pharmaceutical, food, industrial applications, beverages, jams, laundry detergents, cosmetics, etc.

Chemical curve: Citric acid per Conc. % b.w. at Ref. Temp. of 20 °C



### Introduction

Citric acid is a naturally occurring fruit acid. Citric acid is produced commercially through the carbohydrates fermentation (e.g. glucose) which are often found in starches and sugars. Citric acid is primarily used as an organic acidulant and pH-control agent for food, beverages, pharmaceuticals and technical applications.

Citric acid is available as a colorless crystal, white crystalline powder or as a liquid solution.

### Application

Fermentation is the most economical and widely used method for the production of citric acid. In fermentation, the sugar or molasses is converted to citric acid by microbial activity with the addition of nutrients.

After fermentation, the citric acid needs to be recovered and purified from the broth. Lime is added in a neutralization tank and the resultant calcium citrate is filtered off and acidified with sulfuric acid. The decomposition of calcium citrate with sulfuric acid results in soluble citric acid and insoluble calcium sulfate.

The aqueous citric acid is separated by filtration and concentrated using evaporators before crystallization. The product citric acid is filtered from the mother liquor and dried for a crystalline or powder product. Citric acid can be also supplied as a 50 % liquid solution.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 is used for accurate and reliable concentration measurements at various stages of the citric acid production process. The refractometer is installed in-line for real-time and continuous information on the process and quality of the product.



The refractometer is used at the feed to the fermenter to ensure that the sugar or molasses Brix concentration is within the desired limit, usually between 17 to 22 Brix. This maximizes the use of molasses and minimizes the quantity of unfermented sugar in the broth.

The refractometer also measures the concentration of citric acid in the evaporator feed and outlet to ensure the target concentration is achieved. The refractometer provides Ethernet and 4-20 mA signals that can be used for real-time control of the steam flow for optimizing the energy consumption.

Another refractometer monitors the crystallization process for a high-quality crystal product. Our unique

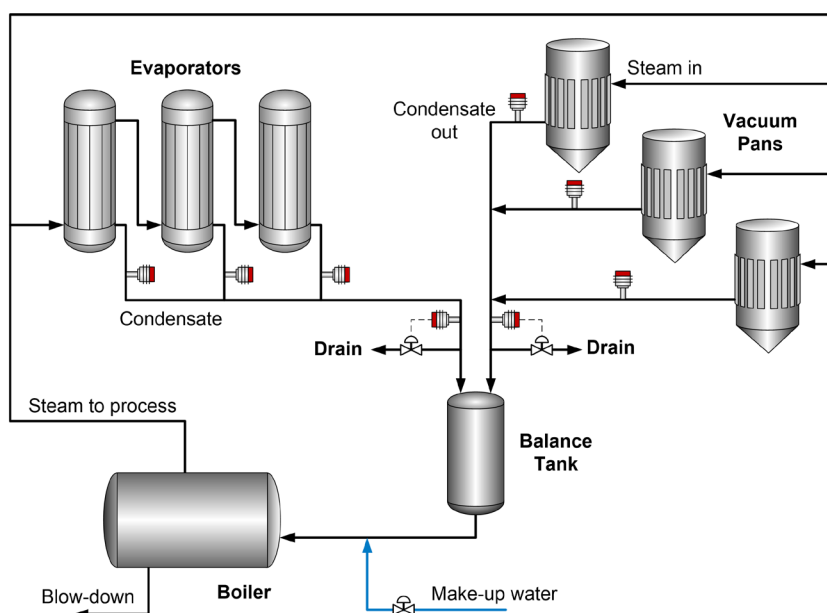
digital refractive index technology is not influenced by bubbles or particles, thus making the refractometer ideal for monitoring liquid-solid operations. A typical measurement range for the crystallizer is 60-95 % and the process temperature is between 40 and 60 °C (104-140 °F).

If the citric acid is dissolved for a final liquid product, the refractometer can be used for automated and reliable dilution of the acid, thus enhancing productivity and guaranteeing the product is within specifications. The end-product ranges from 45 to 65 % and temperature varies from 20 to 40 °C (68-104 °F).

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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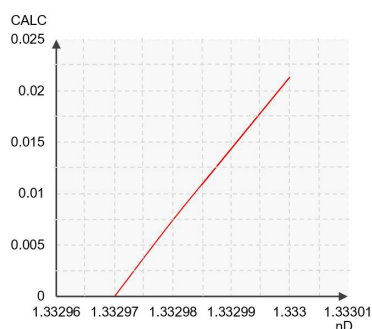


## SUGAR IN CONDENSED WATER

### Typical end products

Pure water for the boiler system.

Chemical curve: R.I. per Brix at Ref. Temp. of 20°C



### Introduction

Sugar mills often use the condensed water from the process as a distilled water feed which is safe for the boiler. However, condensates might get contaminated if juice leaks or overflows into the equipment making it unfit for use.

Detection of sugar shots in the boiler feed water is important for operating and maintaining the boiler system. Accurate and reliable measurements of overly high sugar levels permits acting promptly to avoid damage to the boiler system and extra incurred production costs.

### Application

Steam from the boiler is used as a source of energy, for example, in the evaporators. It is common practice in the mills to utilize the vapor from one or several effects of the evaporators to the clarifiers or settling tanks. This leads to the condensation of the vapors resulting in a distilled water that can be used as feed to the boilers.

Feed water with excessive amount of sugar (above 200 ppm) can be extremely damaging for boilers causing a variety of problems including foaming, fouling, and corrosion.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 provides an accurate and real-time Total Dissolved Solids (TDS) measurement for early detection of sugar traces and leakages in the boiler system. The in-line measurement allows to take corrective actions before the contaminated condensates reach the boiler, thus preventing process upsets and damage to the equipment.



The refractometer is installed through a sample conditioning line before the boiler's balance tank by a flow cell. Other refractometers can be installed in all condensate flows, for example from the evaporators, vacuum pans, clarifiers and settling tanks, allowing the earliest leakage point detection.

The refractometer provides Ethernet and 4-20 mA signals for real-time process control. This allows the creation of alarms and control of the drain's shutter valve to avoid feeding contaminated condensates to the boiler and to ensure a safe operation.

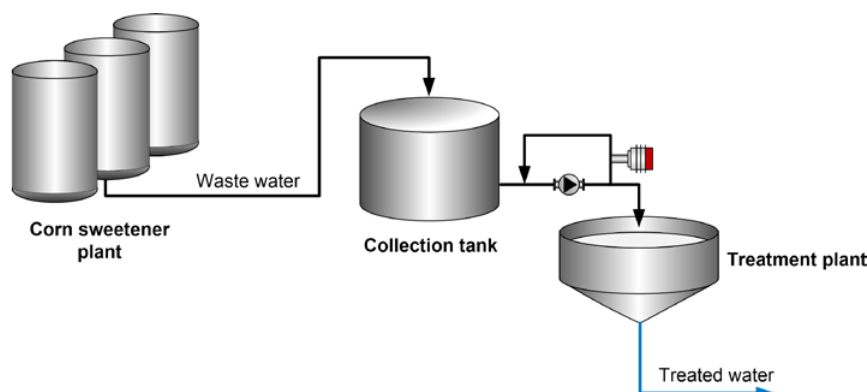
Moreover, the refractometer can be specially calibrated and mounted on a conditioning line to detect sugar contents down to a level of 0.02 Brix (200 pm). This way, lower levels of TDS caused by inorganics salts and other components that do not cause damages to

the equipment are neglected, and only truly harmful sugar content is considered. This way false alarms can be eliminated.

Our solution for sugar leakages detection in boiler feedwater provides instant, accurate and reliable information of TDS allowing for sufficient time to react and eliminate any contaminated water from the system. This increases equipment life, reduces maintenance and operating costs and prevents production losses.

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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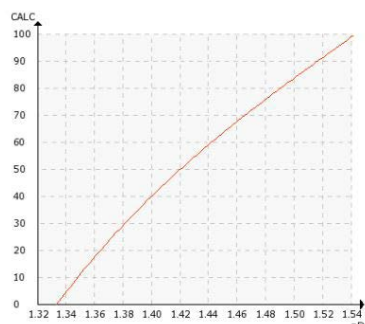


## WATER, DISSOLVED ORGANIC MATERIAL

### Typical end products

Alcohols, proteins, sugars, fats, etc. in various effluent streams in the corn sweetener and beer brewing industries.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

The sugar and sweetener plants are required to monitor and control the organic load in effluent streams. Excess organics, e.g. alcohols and sugars, in various wastewater streams can incur fines and penalties. In severe cases the plant can even be closed.

Before treatment and recycling, the quality of the water must be defined. Traditional methods are Total Organic Carbon (TOC) and Chemical Oxygen Demand (COD) tests.

Refractive index has proven to be a very successful measurement method for organics monitoring in these effluent streams, as the TOC level is typically high (up to 10000 PPM).

### Application

In sugar and sweeteners processing plants, effluent streams containing sugar, or *sweet water* streams, are generated in different plant processes, such as ion exchange columns regeneration. The environmental

load from the mills is reduced with water treatment and by recycling the used water whenever possible.

Wastewater treatment plants need to know the incoming TOC to keep the treatment process in proper operation and compliant. TOC monitoring is particularly important to warn the treatment plant about excess organic concentration.

TOC has traditionally been measured both on- and off-line. Conductivity and non-dispersive infrared (NDIR) are the two common detection methods used in on-line TOC analyzers. However, these analyzers are expensive to maintain and calibrate and are susceptible to frequent breakdowns in their operation.

Laboratory off-line sampling and testing is used to confirm the TOC analyzer readings. Although these tests are accurate, potential TOC load shocks may get missed if sampling and analysis are not performed often enough.

To control the effluent effectively and provide the wastewater treatment plant with warning of exceptional loads, the measuring instrument used must be reliable and in operation at all times.

### Instrumentation and installation



Vaisala K-PATENTS® refractometer PR-43 provides in-line and real-time indication of TOC content in wastewater by means of dissolved solids measurement. The refractometer is installed before the sewage proceeds to the treatment plant to inform the incoming TOC level and keep the treatment process in proper operation and compliance.

The process refractometer acts as a watchdog to provide reliable alarms for high-levels of incoming dissolved solids, which is an indication of high organics concentration. The refractometer's output signal can

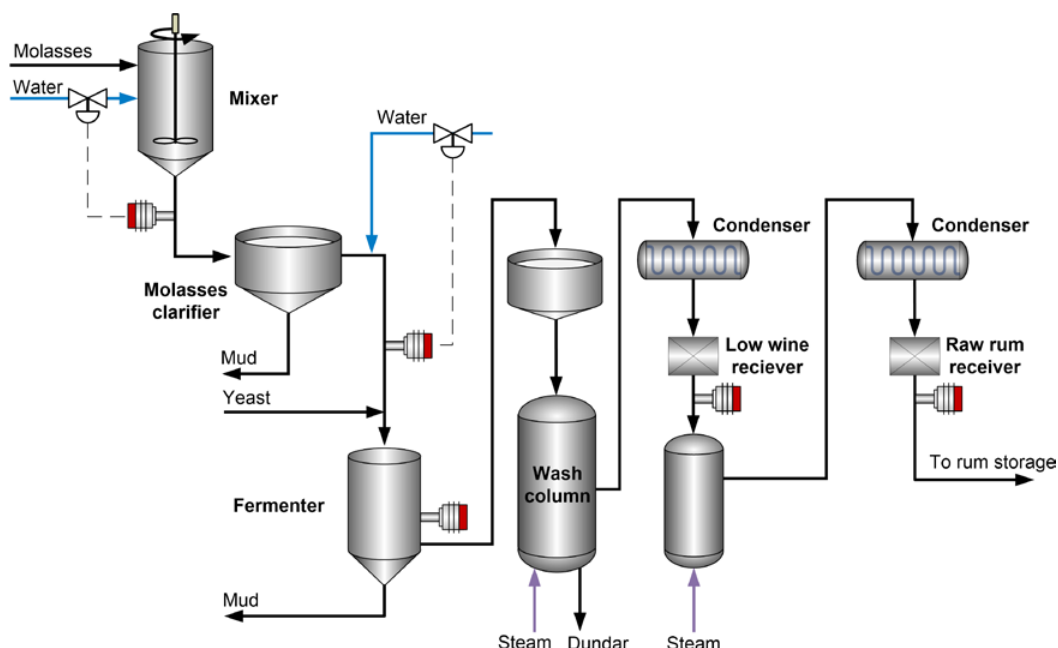
be used to create alarm and control strategies to improve the operation of the wastewater plant. For instance, alarms can be set at 6500 PPM and the pump shut-off point at 7500 PPM.

The output signal from the refractometer can also be used to detect water of sufficient quality for re-use in the process. Recycling water will reduce the volume of waste and the environmental impact of the mill to a great extent.

The refractometer PR-43 provides in-line measurement of the effluent sewer at its dispersing outlet to the treatment plant. The refractometer can read 0-1 % total dissolved solids that correlate with 0-10000 ppm in TOC scale.

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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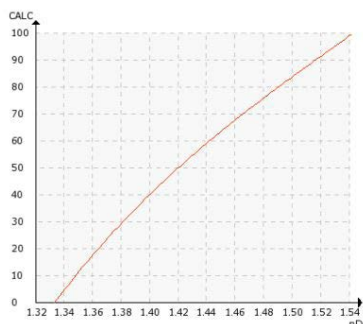


## RUM

### Typical end products

Rum

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Rum is a distilled spirit made by fermenting and distilling sugar cane by-products, such as molasses and sugar cane juice. The distillate, a clear liquid, is usually aged in oak barrels or in similar containers until it becomes mellow and palatable.

### Application

#### Clarification of Molasses

The first step in rum manufacture is the conversion of the molasses to alcohol, and the many other components produced in fermentation which give the rum its distinctive flavor. Before this, however, molasses must be pre-treated.

Molasses is diluted with water and *dunder* (waste from the wash column) and the mixture is heated. A flocculant is added to help unwanted inorganic impurities settle as sludge in a clarifying tank. Clarification of the molasses prevents scaling which is harmful for the still, distillation efficiency and product quality.

The clarified molasses is sterilized in a heating process to ensure minimum bacterial growth during fermentation.

The last pretreatment stage is the addition of water. This lowers the viscosity and sugar content of the molasses to a concentration at which fermentation can take place.

#### Fermentation

A yeast solution is added and mixed to the molasses in the fermentation tanks. Enzymes from the yeast convert the sugar into ethanol and carbon dioxide in nearly equal proportions. Initially the solution contains about 16-18 % sucrose. The alcohol content at the end of fermentation is about 6.5-9 %.

Once fermentation is completed, the tanks are allowed to rest to settle the dead yeast and other solids as mud. The fermented mixture is known as *the wash*.

## Stillhouse

The fermented mixture is pumped to the top of tall wash columns, which are special steam-heated distillation columns. Alcohol vapour is distilled out and removed at the top at about 50 % by volume.

The condensed alcohol mixture is diluted with water in the low wine charger tank and fed to pot stills in batches. The pot stills are like large kettles where the liquid is heated. The fractions, from the start and the end of the batch distillation process, are rejected to waste streams for reprocessing. The strength of the raw rum produced is of about 78 % alcohol.

Modern distilleries use continuous distillation for separation of the fermenting juice. This increases productivity in order to meet the increasing demand.

## Maturation Vats

From the raw rum receiver, the water-clear liquid is transferred to maturing vats, where it gradually converts from raw alcohol to smooth rum. During maturing, the color is adjusted by carefully adding controlled amounts of special alcohol caramel. After maturing, the rum is diluted from storage strength to different bottling strengths.



The refractometer is used for measuring and controlling the dilution operations in the pretreatment of molasses. The refractometer's signal can be used to monitor and automatically adjust the concentration of molasses when mixed with dunder and water to a concentration of 47 Brix or 28 Brix before fermentation. The refractometer ensures that the exact sugar level of the molasses is fed to the fermenter for a smooth fermenting process and a high-quality product.

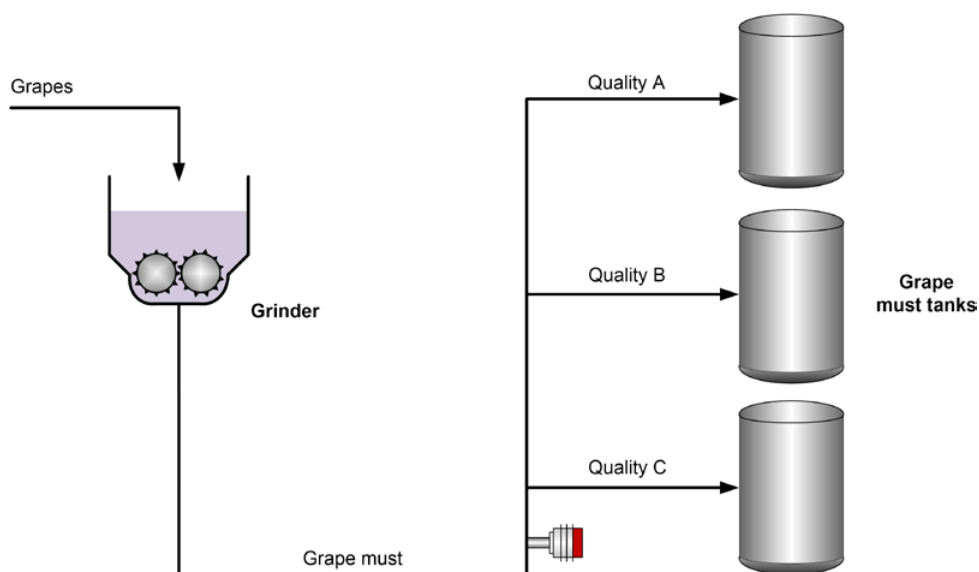
During fermentation the density of the liquid decreases due to the conversion of sugars to alcohol. The refractometer can be used to follow the changes of refractive index in the fermenting juice and to determine the end point (see also application note *Alcoholic Fermentation*).

The refractometer also monitors the concentration of low wine and raw rum to ensure only high quality rum is sent to the maturation vats. If continuous distillation is used, the refractometer provides valuable information on the concentration of overhead product for automatic control of the distillation columns. All this optimizes the energy consumption and increases productivity.

## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A provides accurate Brix measurements for the rum distillation process.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

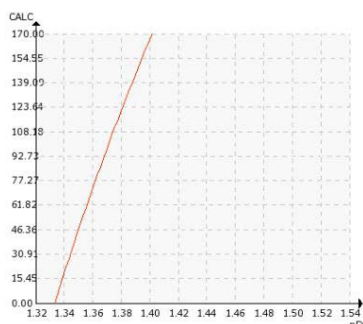


## GRAPE MASH, WINE

### Typical end products

Wine.

Chemical curve: R.I. per Oechsle (°Oe) at Ref. Temp. of 20°C



### Introduction

There are two main steps in wine production: the growing of the grapes and the processing of the grapes into wine.

There are growers who make no wine, and there are wine producers who have no vineyards and consequently buy grapes from growers.

If the wine producer receives grapes from various vineyards, each crop supplied is processed separately. The price paid for each separate crop depends on the quantity and the quality of the grapes. The main quality factor is sweetness of the grape, as the final alcohol in the wine is given by the sugar content in the grape.

### Application

The grapes are received, sorted and ground to produce the sugar rich *must*. The quality, and therefore the value, of the must depends on the sugar content and is measured in terms of °Oechsle, °Brix or °Baumé.


The sugar concentration varies within the batch. It is very difficult to determine a value for the whole crop, and consequently the payment to be given to the producer without continuous measurement.

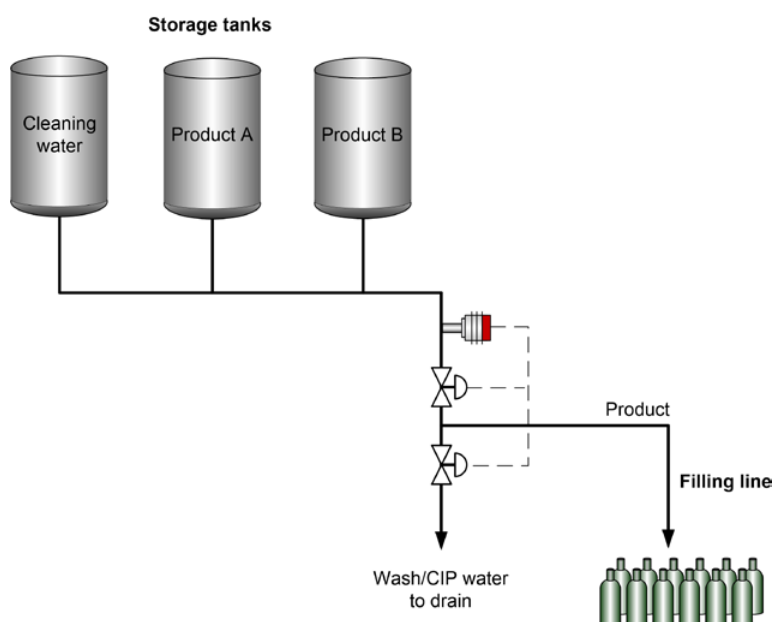
### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-AC is used to measure the sugar concentration continuously in each batch of mashed grape. The calculated average value of sweetness is used to determine a fair payment for the producer.

The refractometer is installed directly in the must pipe before the must is distributed between the various tanks. The must, which is pumped directly from the grinder contains a high quantity of suspended solids such as seeds and peel. The refractometer has a digital sensing technology, which is uninfluenced by the presence of grape solids. Typical measurement range is 0-140 °Oechsle and the temperature is 15-25 °C (59-77 °F).



Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

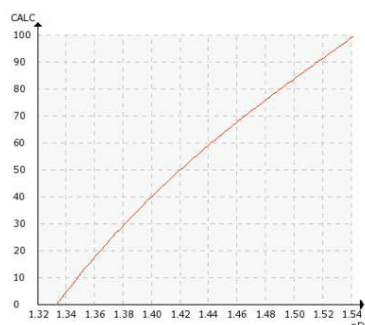


## WINE, CIP WATER

### Typical end products

Wine.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Many wine processing plants use the same filling station for a range of different products. For example, wine factories bottle wines from different barrels in the same station.

High-speed filling machine operations can be improved by utilizing real-time refractive index measurement technology. Automated monitoring and control of the Clean-In-Place (CIP) cleaning process allows wines to be switched without the need for a shutdown. This way, a higher productivity is achieved without compromising the end product.

### Application

After the first wine batch is run through a pipeline to packaging, the pipes are flushed with CIP cleaning chemicals and water.

The cleaning water goes to the sewer and the drain valve is closed. After this the filling line is ready to pass the next product.

In order to save valuable production time, the second wine batch is pumped through the pipeline right after the wash cycle.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-AC instantly detects the product-to-product and product-to-CIP cleaning interfaces.


The Sanitary Refractometer is installed at the end of the filling line to monitor the concentration level of the medium. When the concentration reaches a pre-set limit and there is no water present, the refractometer's 4-20 mA or Ethernet signal activates the end product filling with no delay.

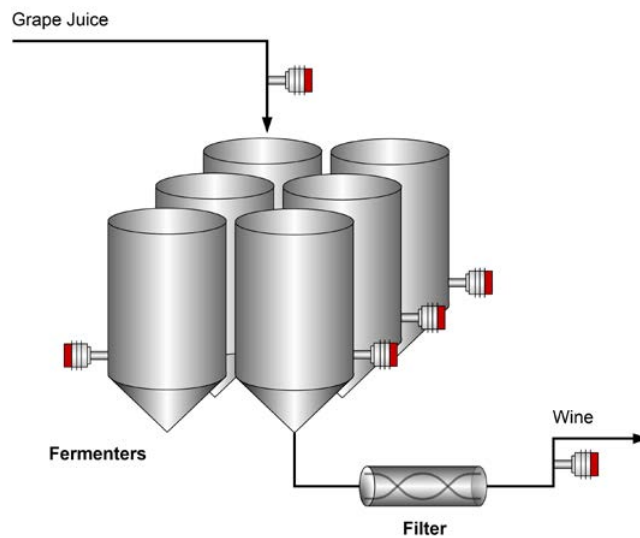
When there are separate lines for the product and for the CIP cleaning media, the water flows to the sewer while the pipe fills with the product. The refractometer gives an instant alarm when the concentration reaches its top limit, and this signal can be used to switch the valve direction. During filling, the valve is open to the filling line and closed to the sewer. During

CIP cleaning and at the start of a new wine batch, the valve closes the filling line and the stream is then diverted to the sewer.

The refractometer is available with 3-A Sanitary and EHEDG certifications to meet the highest hygiene requirements for beverage production. Brix monitoring with the refractometer allows for instantaneous and real-time filling station quality control.

The refractometer's output signal can also be utilized for quality control monitoring, ensuring a correct product and bottle combinations, and that the end product complies with specifications. Colorimeters, which are commonly used in wine production, have been proven to be unreliable in detecting product-to-product wine interfaces

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

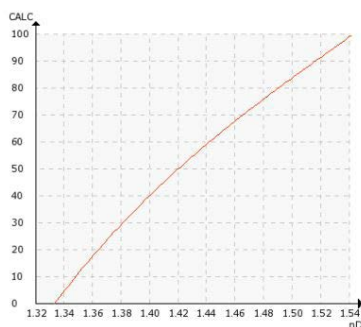


## FERMENTING JUICE, BEER WORT

### Typical end products

Alcoholic beverages made from fermented juices such as wines, beers and ciders.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Fermentation is a key process in the making of alcoholic beverages such as wines, beers and ciders. In this process, yeast species metabolize carbohydrates, mainly sucrose or glucose, and amino acids under anaerobic conditions into ethanol and carbon dioxide.

Alcoholic fermentation determines the strength and quality of the final product. Thus, monitoring and controlling fermentation is critical for ensuring a consistent and high-quality beverage.

### Application

For alcoholic fermentation, grape juice (or other fruit or cereal juice containing sugars) is taken to large fermentation tanks. The sugar content in the juice is important as it determines the final alcohol content. For example, for a red wine the typical starting value

is 25 Brix, corresponding to a refractive index of about 1.37.

As the fermentation progresses alcohol and carbon dioxide are produced and the Brix value decreases. This happens because the alcohol has a lower density than the water. Fermentation is stopped when the ideal sugar and alcohol level is reached.

After the fermenters, the product is filtered, and its quality is analyzed before it is finally bottled.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-A monitors the Brix content, which indicates the conversion rate, the degree of fermentation and the alcohol volume (%) in the production of alcoholic beverages.

The Sanitary Compact Refractometer PR-43-AC is installed in the feed to the fermenters to monitor the Brix value of the juice before fermentation. This ensures the target alcohol level is achievable and prevents upsets in the fermentation process.

The Sanitary Probe Refractometer PR-43-AP is installed directly in the fermenters to monitor and control in real-time the fermentation process. Alternatively, the Sanitary Compact Refractometer PR-43-AC can be installed on a bypass via a sanitary coupling or Varinline® connection.

Changes in the refractive index value are used to continuously follow the fermentation process and to determine the degree of fermentation as alcohol is produced. This allows a better understanding



of fermentation process, provides an indirect measurement of alcohol content, and helps to determine when the batch is ready.

Final sugar content and alcohol level of the beverage is conventionally measured by periodical sampling after fermentation and analyses in the lab. The refractometer can also be installed after the fermenters to monitor in real-time the quality of the final product.

The refractometer is the ideal tool for process optimization and for increasing productivity as it has a

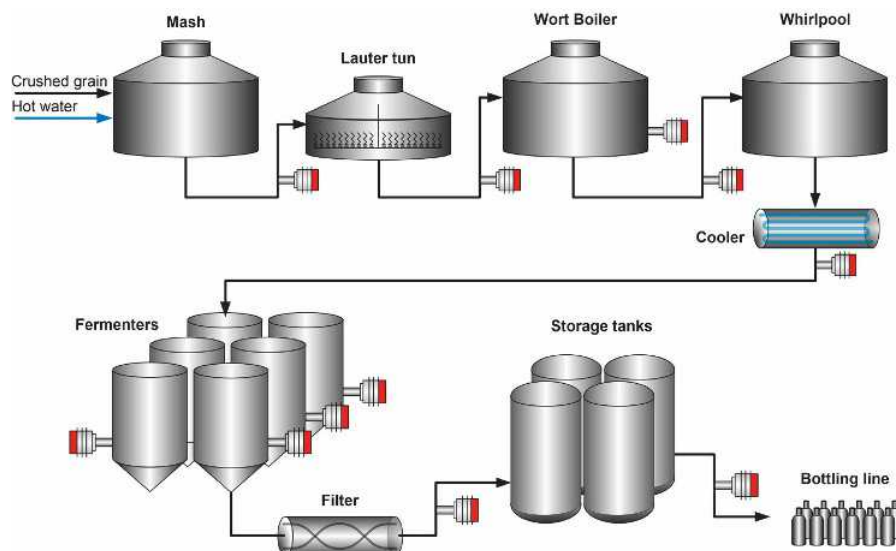
fast response. The refractometer reduces the need for manual sampling and the risk of contamination.

3-A Sanitary and EHEDG certifications are available. Moreover, our technology is accurate and reliable as it is not affected by suspended particles, bubbles or color of the medium, which are a common sources of error in other instruments in this application.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

## Beer Brewing Process

Food and Beverage | Beer Brewing Process | 2.03.00



### Typical end products

■ Beer

### Introduction

The first step in the beer brewing process is the preparation of malt grains. The extract received from the processing of this raw material is called *wort*. The second step is fermentation by yeast. The last steps are conditioning and final filtration. After filtration, the beer is ready for bottling.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A is used at many stages of the brewery process to determine in real-time and accurately the concentration of dissolved solids. The refractometer takes an optical measurement of the refractive index of a solution and its output can be calibrated in Plato, Brix, Balling, gravity or density,

depending on the preference of the brewery.

The Sanitary Refractometer is available with 3-A Sanitary and EHEDG certifications and it withstands CIP/SIP cleaning and rinsing of the facilities.

### 1. Mash tank

Mashing is the process of mixing the crushed malt with very hot water. In this process, the malted grain breaks down to create enzymes, which become active when exposed to water at a specific temperature. These active enzymes convert the starches into sugars. The resultant sugary liquid is known as the *wort*. Mashing is a crucial step as it determines the final structure of the beer.

In the mashing stage, the refractometer is used to measure

the concentration of the mash in water at the outlet pipe to maintain a consistent concentration. Automatic prism wash with steam or high-pressure hot water is recommended for this application.

### 2. Lauter Tun

Lauter tun is a vessel used to separate the extracted wort from the spent grain. The solids in the lautur tun are rinsed with water to separate the clear liquid wort from the solids. The liquid concentration gradually decreases during the rinsing.

At the lautur tun the refractometer's output signal is used to detect the shut-off point for rinsing, thus preventing excessive use of water. Automatic prism wash with steam or high-pressure water is recommended for this application.

### 3. Wort Boiler (Brew Kettle)

In the wort boiler the wort is pasteurized, and its flavor is adjusted by the addition of hops (or other flavors such as ginger or molasses). The brew is boiled until a certain strength or gravity is achieved. This step is essential for the quality of the final beer.

The refractometer is installed directly on the wort boiler to measure the wort strength/gravity. It provides an instant feedback when the wort has reached its required strength. No by-pass arrangements are required. The purpose is to eliminate sampling, optimize the boiling time, and to improve beer consistency and quality. Automatic prism wash with steam or high-pressure water is recommended for this application.

### 4. Hot Wort from Boiler to Whirlpool

The refractometer is installed in the wort boiler outlet to monitor the quality of the wort. Before the wort goes on to the next stage, solids are removed from the liquid by using a whirlpool. All hops and other solids are forced to the center of the whirlpool. When the whirlpool is stopped, the solids settle at the bottom, forming a fairly solid central cone. The liquid can then be drained off.

### 5. Chilled Wort from Heat Exchanger

The wort is cooled down to the correct temperature for the yeast. The refractometer is used for quality control, by a way of measuring the cold wort before it enters the fermentation process. This is an alternative measurement to point 4.

### 6. Fermentation

Fermentation starts when the yeasts are added to the wort. The yeast converts the sugars and amino acids into alcohol and carbon dioxide. The fermenting reaction is slow, and its progress must be monitored to determine the end point.

The spent yeast settles at the bottom of the tank and is frequently removed. This process helps to clarify the beer.

In fermentation, The refractometer detects continuously the changes in refractive index because of the conversion of sugars into alcohol. This allows monitoring the conversion rate, the degree of fermentation and provides an indirect measurement of the alcohol volume (%) for determining the end point (see also application note *Alcoholic Fermentation*).

### 7. Filtering

After fermentation, the beer is let to rest, so that the suspended dead yeast settles at the bottom. This conditioning process helps the maturing of the beer. The refractometer provides a quality control measurement for the wort filtering output.

### 8. Packing and Interface Detection



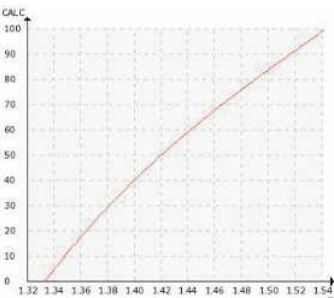
Packing is typically done by filling the beer into bottles or aluminum cans via a filling machine. Some brewers may use the same filling line for different products. In this case a Clean-In-Place (CIP) operation is required between the filling of each product.

At the filling line, the refractometer instantly detects the product-

to-product and product to-CIP cleaning interfaces in bottling. The refractometer output signal can be utilized for quality control monitoring, and to ensure correct product-to-bottle selection and a product quality within specification.

If the same filling station is used for different products, the refractometer can be used for automated monitoring and controlling of the CIP cleaning process, allowing products to be switched freely. This results in increased productivity, without compromising the end product's quality and safety.



Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
<b>User Interface</b>	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
<b>Prism wash systems</b>	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.
<b>Chemical curve</b> 	R.I. per BRUX at Ref. Temp. of 20°C

**VAISALA**

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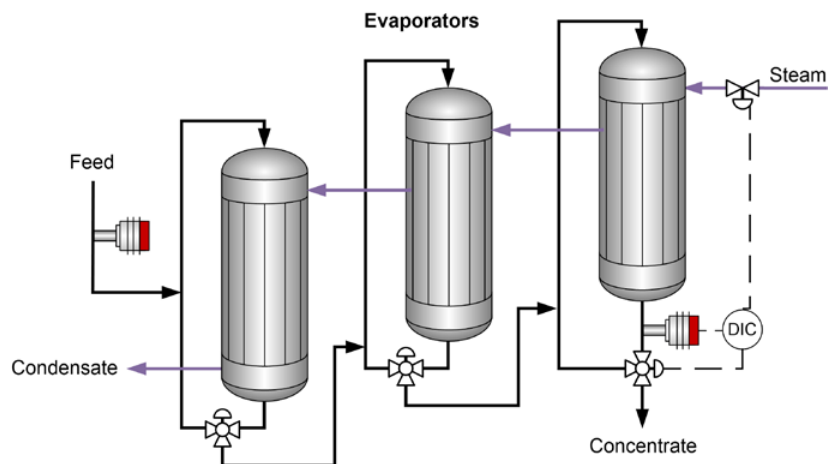
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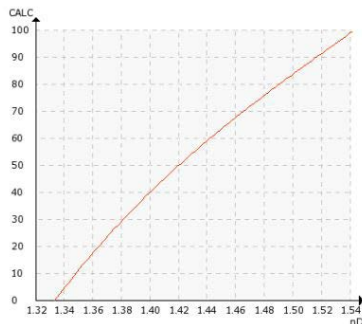


## JUICE CONCENTRATE

### Typical end products

Fruit and vegetable juice concentrate from apple, orange, grapefruit, pineapple, tomato, passion fruit, mango, carrot, grape, cherry, cranberry, guava, pomegranate, etc.

Chemical curve: R.I. per Brix at Ref. Temp. of 20°C



## Introduction

Fruit juice concentration by evaporation is one of the oldest and well-known methods for preserving fruit and vegetables and their derived products. This operation consists in removing part of the water content of the juice, so that all the solid components such as fruit sugars, minerals and vitamins are left in a more concentrated solution.

Juice evaporation is performed to lengthen the shelf life, minimize packing and storage, facilitate transportation and simplify the handling of the concentrated product.

## Application

After the juice is extracted from the fruit, it is screened and purified, for example by centrifugation. The concentration of the juice at this stage varies from 9 to 12 Brix. The inconsistency is due to various factors such as the fruit quality, origin and annual rainfall. To

balance the differences in concentration, the juice is sent to a primary tank, before it is evaporated.



For fruit juice concentration, a three stage falling film evaporation plant is commonly used. The evaporators usually operate at a constant boiling rate. In the evaporation process, the concentration value is typically increased from 10 to 65 Brix.

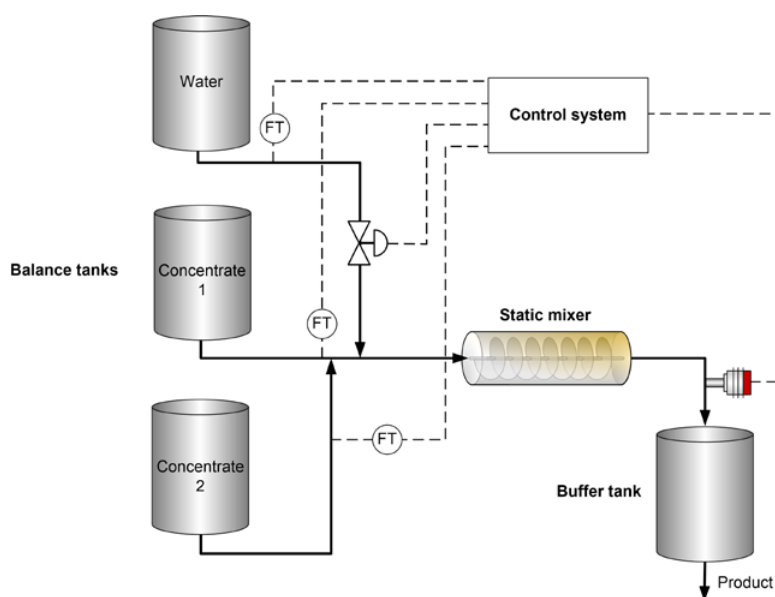
## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-A is the ideal instrument for concentration monitoring in food and beverage processing. The refractometer meets the strict requirements for hygienic processing and is available with 3-A Sanitary and EHEDG certifications.

The refractometer is mounted directly on the evaporator outlet to ensure the target concentration is achieved and to guarantee a high-quality product. The refractometer provides Ethernet and 4-20 mA output signals that can be connected to the process controller to regulate the final Brix concentration by automatically adjusting the evaporator inlet flow or steam flow.

For instance, if the Brix value increases, the valve allows higher product flow rate through the evaporators. This brings the Brix value back to the set-point. Typical measurement range is 30-80 Brix.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
<b>User Interface</b>	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
<b>Prism wash systems</b>	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

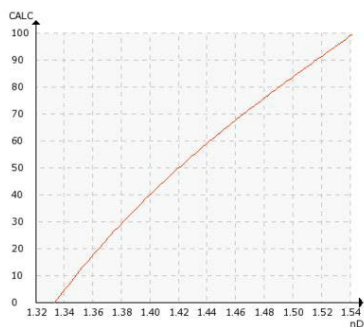


## JUICE, NECTAR AND STILL DRINKS

### Typical end products

Fruit and vegetable juice concentrate from apple, orange, grapefruit, pineapple, tomato, passion fruit, mango, carrot, grape, cherry, cranberry, guava, pomegranate, etc.

Chemical curve: R.I. per Brix at Ref. Temp. of 20°C



### Introduction

Juice is obtained by mechanically squeezing fresh fruits or vegetables. The extracted juice has 100 % fruit content and can be used for the preparation of drinks such as nectars and still drinks (non carbonated).

The juice is mixed with water, sugar and other ingredients to a certain concentration. Nectars have a juice content of 25-99 %. Still drinks have a concentration of 0-24 %, which can come from fruit, vegetable or flavors.

### Application

Nectars and other still drinks are often prepared by in-line blending of juice.

In this process, water and juice concentrates enter the system from balance tanks. The concentrate is fed into the water stream to form a pre-blended juice. The ratio of the two streams is controlled by flow meters and a process controller. Precise dosing of the juice ingredients is essential for achieving the desired concentration and ensuring a consistent product quality.

The concentration is adjusted by adding small quantities of water via a separate line to achieve a pre-set Brix value.

The juice passes through a static mixer before it moves on to further processing and packing.

A highly automated process is essential for achieving precise in-line juice blending. Instabilities in the juice concentrate and water flows, and variations in tank contents and pumping rates, lead to fluctuations in the concentrate/water ratio. These fluctuations are difficult to control when using traditional blending methods.


### Instrumentation and installation

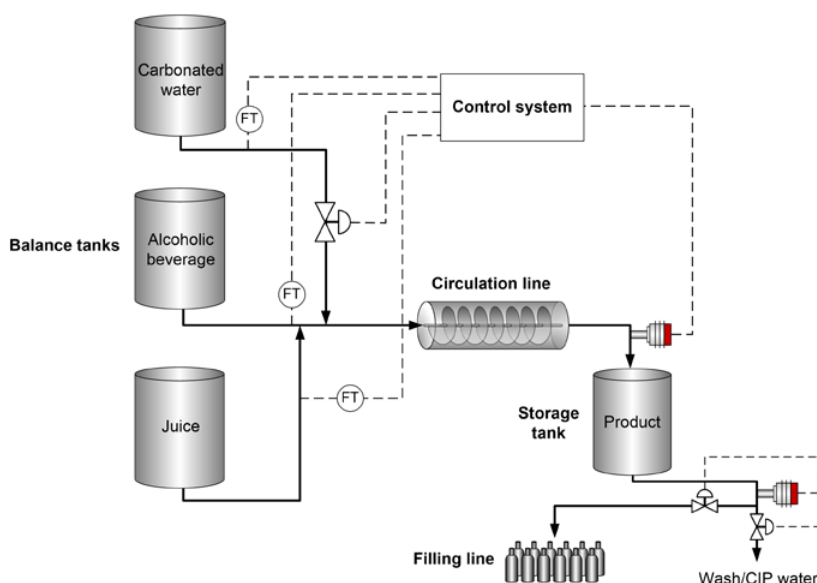
Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A is installed immediately after the static mixer to measure continuously the final Brix concentration of the product. The refractometer output signal is connected to the controller to provide the required information to adjust the ingredients dose. If the Brix value after blending is below the pre-set value, the controller opens the concentrate feed valve to increase the Brix content. Similarly, the controller can

command the addition of water if the concentration of the product exceeds the desired value. The typical measurement range is 10-15 Brix at a temperature of 10-20°C (50-68°F).

The Sanitary Process Refractometer is designed to meet the highest standards for safe food processing. Moreover, the refractometer's measurement is not influenced by fruit pulp, color, fibers, solid particles or air bubbles.

In-line blending control with the refractometer eliminates the need for reblending or penalties due to a too low Brix level. The refractometer's accurate concentration measurement also minimizes concentrate loss and ensures a consistent product quality.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

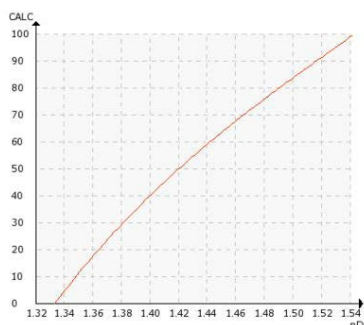


## CARBONATED SOFT AND ALCOHOLIC DRINK MIX (CHUHAI)

### Typical end products

Carbonated soft drink mix with whiskey, vodka or other alcoholic beverage, water and juice (grapefruit, lime, apple, orange, pineapple, grape, kiwi, peach, strawberry cream, cream soda), Chuhai.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Alcoholic and soft drink mix, e.g. Chuhai (an alcoholic drink originating from Japan) is prepared by mixing carbonated water flavored for example with grapefruit juice, and alcoholic beverage, such as whiskey, vodka or shōchū (a Japanese distilled beverage).

Quality assurance using in-line process refractometer for fast and reliable product identification and set-point detection is important. Combining a refractometer with automatic controls can minimize transmix of products, reduce waste, reduce the filling times, decrease safety risks, reduce sampling and minimize operator errors. A highly automated process is essential for achieving precise in-line alcoholic soft drink mix.

### Application

Water, juice and alcoholic beverage, such as whiskey, enter the system via balance tanks. The feed ratio of the three streams is controlled by flow meters and a process controller. Immediately after this, final blending to a pre-set Brix value is achieved by adding a small amount of water by way of a separate line. Then, the juice is passed through a circulation line, before it is ready for packing or bottling.

After the first batch is run through the pipeline to the filling machine, the pipes are flushed with Clean-In-Place (CIP) chemicals and water to avoid mixing between products. In order to save valuable production time, the second batch is pumped through the pipeline right after the wash cycle. At this point, it is important to detect instantly the product-to-product and product-to-CIP cleaning interfaces.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-AC provides accurate concentration measurement for precise control in the blending of carbonated alcoholic drinks. Because of its fast response, the refractometer instantly detects the product-to-product and product-to-CIP liquid interfaces.

The refractometers' output signal is also used for quality control monitoring, ensuring a correct product

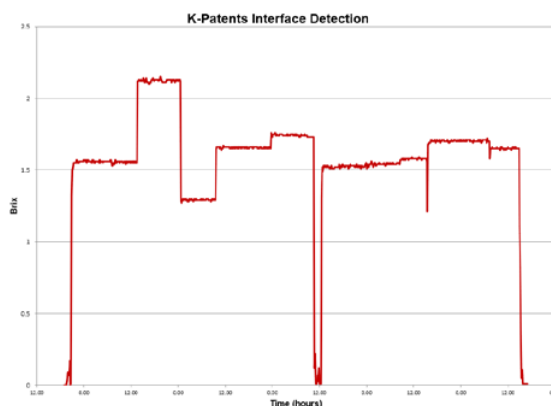
and can or bottle combinations, and that the end product complies with specifications.

The PR-43-AC refractometer can be installed in two process points:

1. Concentration control in the blending unit. Here an accurate measurement of total concentration after adding all ingredients is important. Continuous in-line measurement provides important information that the final product is within specifications.

2. Product interface and product identification measurement in the pasteurizer to eliminate accidental mixing of the liquids. The product identification is based on Brix measurement. Refractive index and Brix measurement is a reliable method for identifying products since each liquid has a different and distinct refractive index value. The refractive index is a property inherent to the liquid and can be used as a fingerprint for product identification (Figure 1).


The refractometer is installed in the circulation line. It is angle mounted on the outer radius of a pipe bend, either directly or through a flow cell and a Sanitary clamp or a Varinline® connection. The typical measurement range is 0-15 Brix at a temperature of 2-20°C (35 - 68°F).



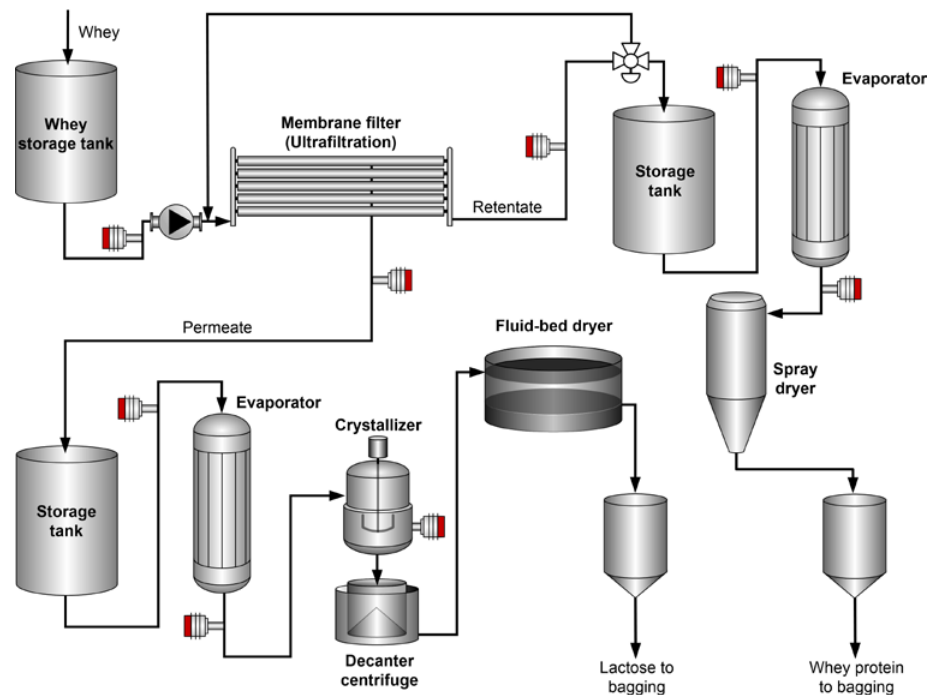
**Figure 1.** Example of interface detection between products. Each segment of the chart represents a different product.

With the refractometer, a highly accurate concentration measurement is achieved. Thus, re-blending or penalties due to lower than specified Brix levels are avoided and loss of concentrate due to an overly high Brix level are minimized.

The PR-43-AC is available with 3-A Sanitary and EHEDG certifications. Monitoring of product Brix with the refractometer allows for instantaneous and real-time filling station quality control.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.



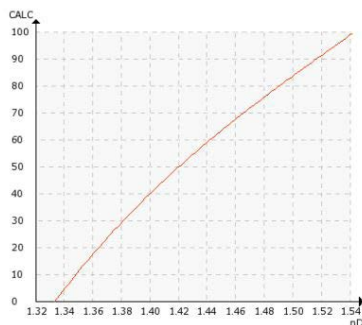


## WHEY PROTEIN AND LACTOSE

### Typical end products

Whey protein concentrate (WPC), whey powder, lactose, casein.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Whey is a liquid residue of cheese and casein production, which contains a large amount of food protein. Whey comprises 80-90 % of the total volume of milk entering the process. It also contains about 50 % of the nutrients of the milk: protein, lactose, vitamins and minerals. Whey Protein Concentrate (WPC) is processed by ultrafiltration (UF). Reverse osmosis (RO) or diafiltration are also used. UF is the most widely used process of membrane filtration (fractionation) in the dairy industry. The process works on the principle that a membrane restricts the passage of particles over a certain size. For example, in UF large particles like fats and proteins are retained

(retentate), while small particles, like salts and sugars, pass through the membrane (permeate).

### Application

Whey is processed as soon as it is collected to reduce bacterial activity. The process starts with the separation of fines and fat, followed by Ultrafiltration (UF). In UF, the whey is passed through a membrane filter to separate the whey protein as a retentate and lactose as the permeate. After separation both products are concentrated by evaporation. For a powdered product, the whey protein is fed to a spray dryer. The lactose is crystallized and separated from the mother liquor by centrifugation and dried in a fluid bed dryer. The final powdered products are then bagged.

Whey Protein Concentrate (WPC) is a powder, which contains 35-85 % protein in dry matter. To obtain more than 80 % protein concentration, it is necessary to diafilter, add water to the UF feed to wash out low molecular components, to remove more lactose and to raise the protein concentration in relation to total dry matter.

Lactose is the main constituent of whey and there are two basic methods of lactose recovery. The crystallization of untreated but concentrated whey, and the crystallization of whey, from which the protein has been removed by UF. After UF, the whey is first

concentrated to 60-62 % dry matter by evaporation, and then it is transferred to crystallization tanks, where seed crystals are added to it. After crystallization, the crystals are separated by centrifuges and dried into powder in fluid bed driers. Before packing the lactose is also ground and sifted.

## Instrumentation and installation

In both, the whey and lactose processing applications, Vaisala K-PATENTS® Sanitary Refractometer PR-43-AC is used to control the correct feed product concentration for the following process step. Typically, the refractometer is installed on a pipe bend. It is angle mounted on the outer radius of the pipe bend directly or by using a flow cell. This way the best flow conditions and self-cleaning effect can be achieved.


The refractometer helps to control and adjust concentration levels after the ultrafiltration, and at the evaporator inlet. The concentration measurement from the evaporator outlet helps to optimize energy consumption. It also ensures the correct feed product concentration to the spray dryer or crystallizer.

The refractometer is installed directly in the crystallizer to monitor the supersaturation of the lactose solution, and to determine the exact seeding point.

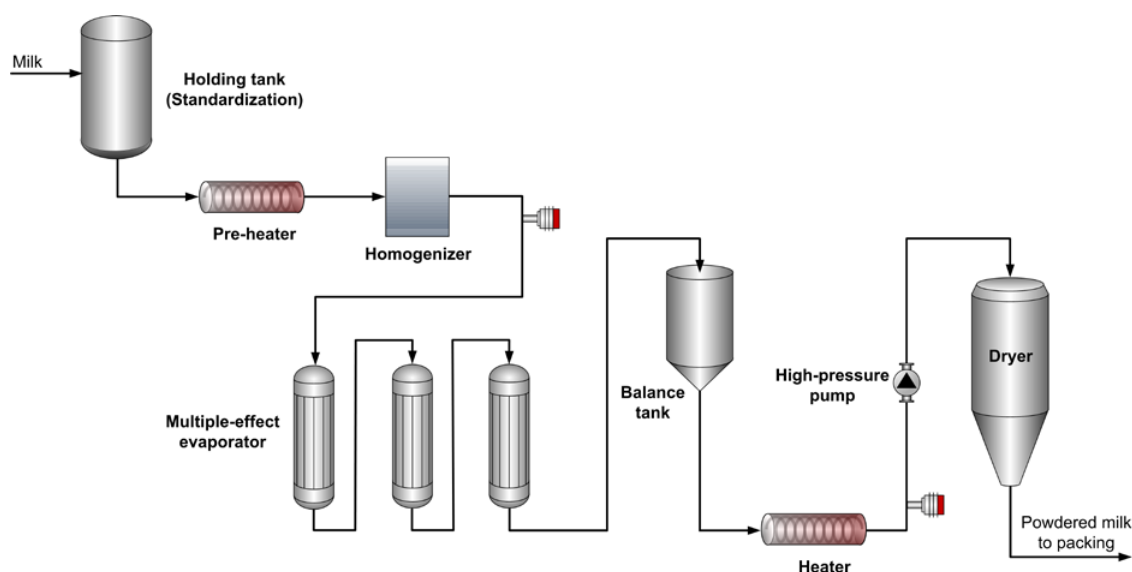
If an instrument is used in the RO process, it has to be mounted outside the loop, due to the high process pressure of about 40 bar.

Typically, prism wash is not required for any of these applications, since the plants are CIP cleaned every 10-20 hours. Typical process temperature for whey application is 10-70 °C (50-158 °F) and the measurements vary between 0 and 85 Brix.

The high accuracy control achieved with our precise in-line concentration measurement helps to improve the quality of the final product and to reduce operating costs. Additionally, the refractometer has a built-in web server that can be accessed via Ethernet, which significantly improves its ease-of-use. Obtaining real-time measurement data displays and diagnostics, altering instrument configuration settings or updating program versions can all be done remotely from the control room.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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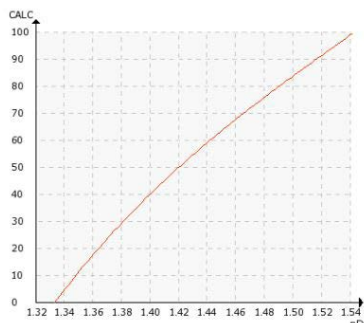


## EVAPORATED OR CONDENSED MILK

### Typical end products

Unsweetened condensed milk (evaporated milk), sweetened condensed milk and powdered milk.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Evaporation is one of the oldest methods for preserving milk. In this operation, water is removed from the milk to obtain a concentrated dairy product.

Depending on the process, the evaporated milk may be the desired end product, or evaporation may just be a prior step to further processing, for example, to produce sweetened condensed milk or powdered milk.

### Application

The first step in the production of unsweetened condensed milk (or evaporated milk) and powdered milk is the standardization of the fat and dry matter content of the raw milk to the level required in the final product. This is followed by a heat treatment

to destroy microorganisms and to stabilize the milk. Preheating the raw milk before evaporation has a significant effect on the shelf life of the final product.

The milk is then evaporated to a specific dry solid concentration. For the production of unsweetened and sweetened condensed milk, the milk is evaporated to obtain a concentration of 30-40 % dry solids. For the production of powdered milk, the milk is concentrated to about 40-50 % dry solids for a spray dryer, and about 18 % dry solids for a roller dryer. The total dissolved solids concentration achieved in evaporation is critical as it affects the performance of subsequent operations and the quality of the final product.

After evaporation, the milk is homogenized. Homogenization reduces the mean size of the fat globules so that they are distributed uniformly in the milk and do not rise to the top creating a creamy layer. This is not a required step in the production of powdered milk, but it is applied to facilitate milk reconstitution.

The evaporated, homogenized milk then moves on to cooling, sterilization and packing for a canned evaporated milk, or to drying for a powdered milk product.

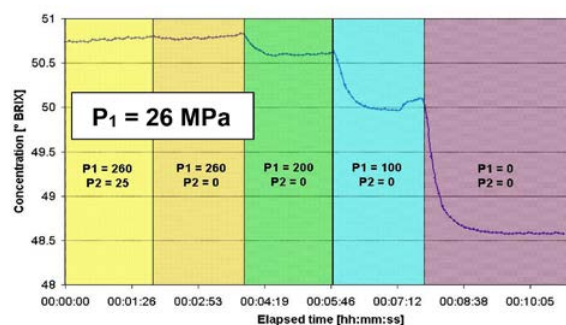
### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A provides real-time and accurate Total Dissolved Solids (TDS) measurements for better control and monitoring of the milk evaporation process. The refractometer can be calibrated to read

TDS or another scale preferred by the manufacturer, e.g. Brix.

The refractometer is used for standardization after the holding tank to achieve the precise milk solids content as indicated by legal standards.

If a homogenization step is performed, a refractometer is installed after the homogenizer to include the fat content in the measurement. The refractometer detects fat globules as long as they are smaller than 6 µm. This globule size can be achieved by adjusting the pressure of the homogenizer. The recommended homogenizer's primary pressure is  $P_1 = 26$  MPa (260 bar).





An additional refractometer between the evaporator and the high-pressure pump to the dryer allows for continuous control of the evaporation performance and dry solids concentration levels.

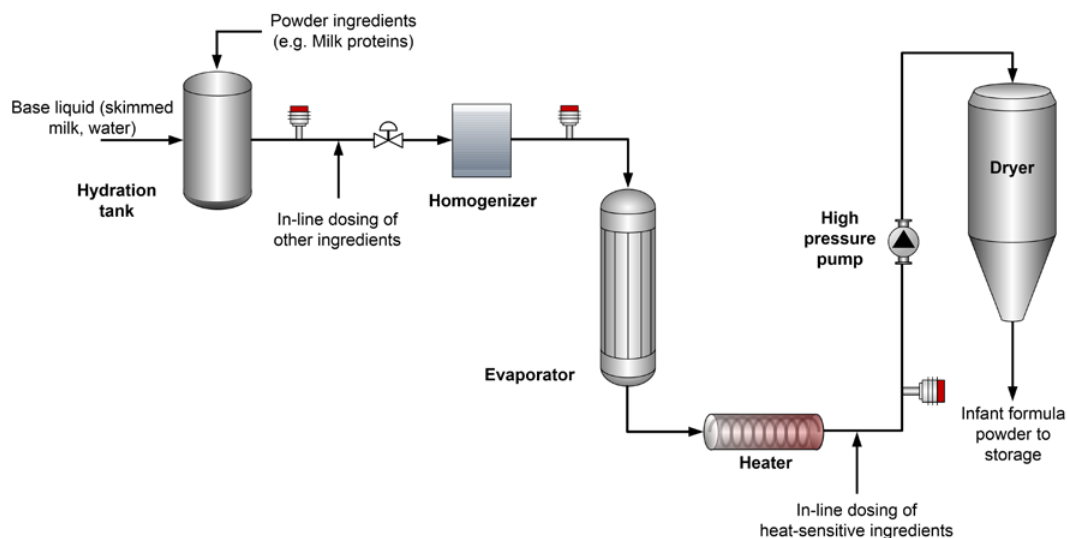
Accurate TDS measurement after evaporation is important in order to achieve the desired quality of the evaporated milk product and to enhance the drying operation. If the dry solids content exceeds the targeted level, the viscosity of the milk increases and creates problems with atomization during drying. Low solids content increases the energy consumption at the drying stage.

Thanks to the refractometer's self-cleaning design, a prism wash system is usually not required. However, for fluids with a dry solids content above 40 % or a flow velocity below 1.5 m/s, a steam prism wash solution consisting of the Sanitary Probe Refractometer PR-43-AP-L42 with an aseptic steam valve ASV and side flow cell is required.

The refractometer provides Ethernet and 4-20 mA output signals for real-time process control. The refractometer is available with 3-A Sanitary and EHEDG certifications.

The control achieved with the precise and accurate in-line concentration measurements helps to improve end-product quality and to reduce operating costs.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash is required in applications where flow velocity is below 1.5 m/s (5 ft/s) or where dry solids exceed 40 %. The wash media is aseptic steam. The components of a steam wash system are refractometer PR-43-AP-L42 with insertion length of 42 mm, Side flow cell SFC-HHSS-H10/15/20/25, Aseptic steam valve ASV-H/ESS-H05, and Multichannel user interface MI for automatic prism wash diagnostics and control.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

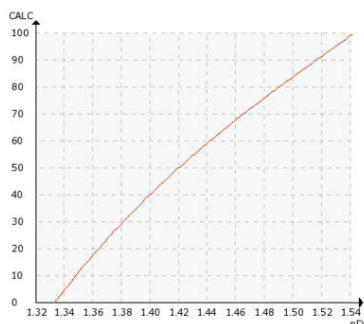


## INFANT FORMULA

### Typical end products

Powdered infant formula, powdered baby milk.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Baby milk, or infant formula, is a substitute for breast milk. It is available as either a dried powder, or as a ready-to-use canned or bottled liquid. Regardless of the type, all formulas contain a protein source, a fat source, a carbohydrate source and added supplements.

Infant formula can be manufactured by a wet-mixing process where all ingredients are handled in liquid form, a dry-blend process where ingredients are blended as powders, or a combination of these processes.

Wet-mixing is the most used technology as it ensures control over the milk's composition and microbiological safety. The formula's composition should be carefully monitored to achieve a high-quality product that

matches as close as possible to breast milk, and which is safe for infant consumption.

### Application

The wet-mixing process involves three stages: preparation of the mix, evaporation and drying. For the preparation of the mix, the base liquid can be water or skimmed milk. Other water-soluble powder ingredients are added to the base liquid (e.g. milk proteins) and the resulting mixture is stored in a large vessel for complete hydration.

A homogenization step follows to increase the uniformity and stability of the emulsion by reducing the size of the fat and oil particles in the formula.

Evaporation of the milk mixture is an essential step that enhances the spray-drying operation and increases the final product shelf life. Other ingredients, which are heat-sensitive, are added after evaporation.

Finally, the concentrated milk is typically dried in a spray-dryer system. The temperature as well as the solids content of the milk mixture are kept as high as possible for maximum efficiency. Low solid content results in small particle size, as well as poor wettability and short self life of the final product. Excessively high dry solids content increases the milk's viscosity which results in larger particles and affects the spray dryer capacity.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-A is the ideal in-line measurement instrument for safe,

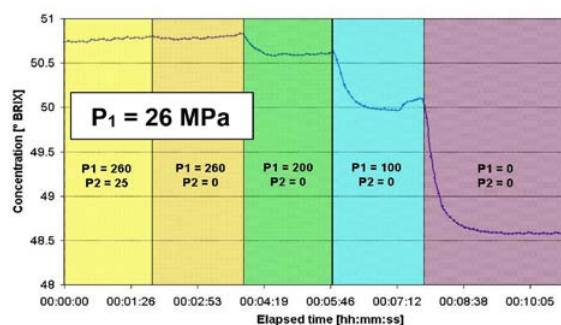
hygiene and accurate manufacturing of infant formula. The refractometer is available with 3-A Sanitary and EHEDG certifications. It is designed to withstand CIP and high process temperatures. Moreover, the refractometer can be calibrated to measure either Brix, dry solids, or Total Dissolved Solids (TDS).

A refractometer is installed after the hydration tank to measure TDS content. This ensures the right in-line dosing of other ingredients to meet the recipe requirements.

A second refractometer after the heater monitors the dissolved solids content. This helps to determine the right dose of heat-sensitive ingredients and ensures that the right concentration is fed to the dryer. Solid content prior drying is critical to optimize the spray dryer performance, energy consumption and final product quality.

A third refractometer can be installed before the evaporator for maximum evaporation performance control. When the products contain fat or oil, the refractometer should be installed after the homogenizer where the fat globules break down to a smaller size. The refractometer detects fat globules as long as they are smaller than 6 µm.



The pressure in the homogenizer can be adjusted to obtain this globule size. The higher the pressure the smaller the globule. The recommended homogenizer's primary pressure is  $P_1 = 26$  MPa (260 bar).



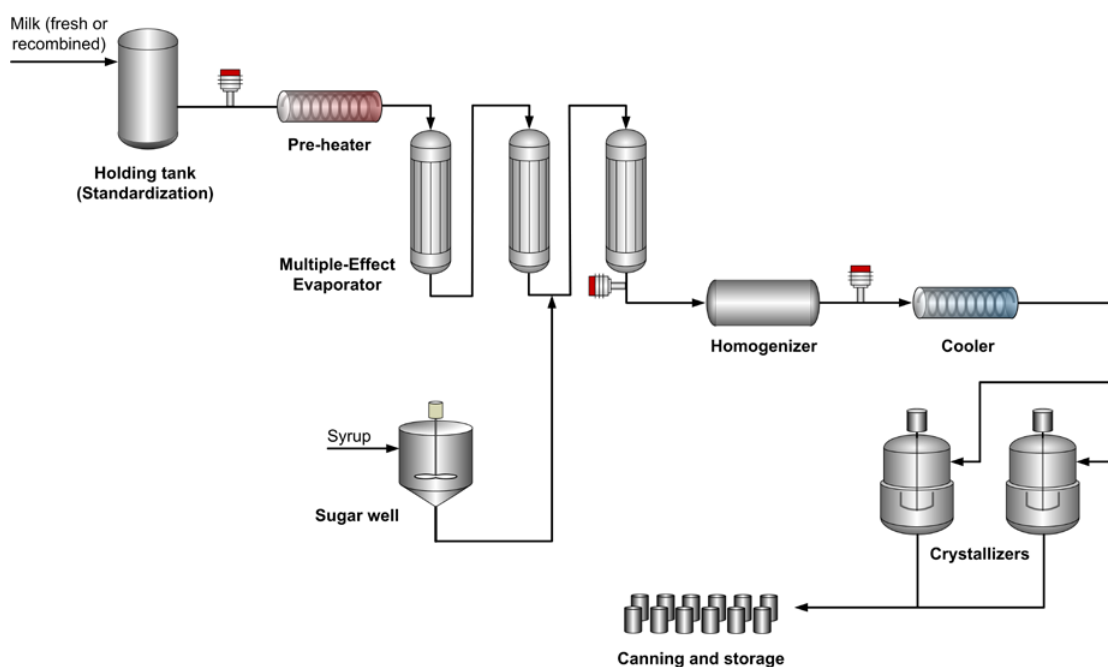
Typically, prism wash is not required for any of these applications. However, a steam prism wash solution using Process Refractometer PR-43-AP-L42, aseptic steam ASV valve, and a side flow cell is required for low velocities or for fluids with a dry solid content above 40 %.

The refractometer is a stand-alone device capable of operating independently or with one of several different user interface options. It provides an Ethernet or 4-20 mA output signal for real-time process control.

The accurate control achieved with the precise in-line concentration measurements ensures a high quality and safe end product, as well as optimized operating costs.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash is required in applications where flow velocity is below 1.5 m/s (5 ft/s) or where dry solids exceed 40 %. The wash media is aseptic steam. The components of a steam wash system are refractometer PR-43-AP-L42 with insertion length of 42 mm, Side flow cell SFC-HHSS-H10/15/20/25, Aseptic steam valve ASV-H/ESS-H05, and Multichannel user interface MI for automatic prism wash diagnostics and control.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.



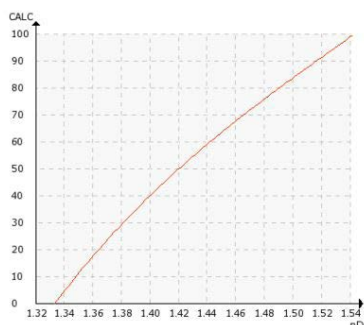


## SWEETENED CONDENSED MILK

### Typical end products

Sweetened condensed milk.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Sweetened condensed milk is a product obtained simply by evaporating milk to decrease the water content and adding a sweetener. The sweetener can be sucrose, dextrose or any other natural sugar.

This product was invented as a way to preserve milk. The sugar content in the sweetened condensed milk increases the osmotic pressure to a level where most of the microorganisms are destroyed.

Sweetened condensed milk is typically used in the making of many sweets and desserts.

### Application

Sweetened condensed milk can be produced from whole or skim milk. The first step in this process is the standardization of fat and solids-not-fat to a level that meets legal standards.

The following preheating step stabilizes the milk and destroys microorganisms. The preheated milk is pumped to the evaporator where the solids content is increased by the removal of water.

A multiple-effect evaporator that operates under vacuum conditions is typically used to raise the solids concentration to 30-40 % (see also application note *Milk Evaporation and Drying*).

Sugar is added as a dry solid either before evaporation or as a syrup during the process. It is important to accurately monitor the quantity of sugar added as the shelf life of the milk depends on the osmotic pressure being sufficiently high. The sugar in the milk is generally between 62.5-64.5 %.

The amount of sugar required can be predicted from the total dissolved solids content in the fresh and the evaporated milk.

After evaporation, some manufacturers homogenize the milk to regulate the viscosity of the end product. The sweetened condensed milk then moves on to



cooling and crystallization. After this, the milk is ready for canning or packing.

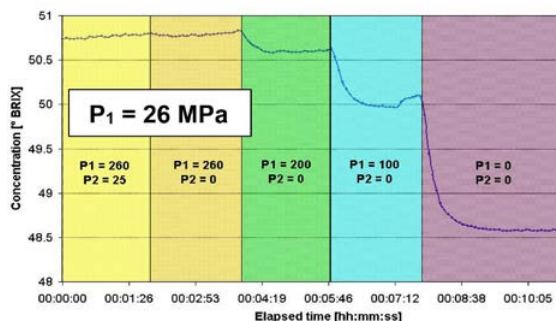
## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A accurately controls the Total Dissolved Solids (TDS) content of the milk in real-time to obtain a high quality product with a long shelf life.

The refractometer is used for standardization after the holding tank to achieve the precise solids content required to meet legal standards. Accurate TDS measurement in the fresh milk also helps to determine the exact amount of sugar required for addition before or during the evaporation step.

A refractometer after the evaporation step monitors the performance of the evaporators and helps to achieve the desired concentration of the milk.



If the manufacturing process involves a homogenization step, a third refractometer can be installed after that stage. The refractometer detects fat globules as long as they are smaller than 6 µm. This globule size can be achieved by adjusting the pressure of the homogenizer. The recommended homogenizer's primary pressure is  $P_1 = 26 \text{ MPa}$  (260 bar).

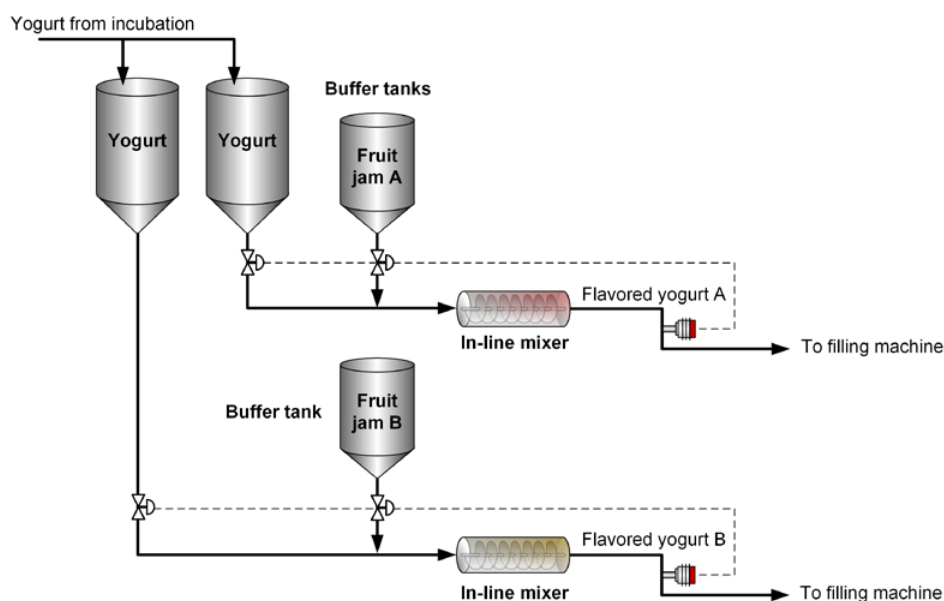


The refractometer can be calibrated to read the factory's preferred scale, either Brix or Total Dissolved Solids. Moreover, the refractometer provides Ethernet and 4-20 mA output signals that can be used for real-time process control.

Usually a prism wash system is not required except when the dry solids content exceeds 40 % or the flow velocity is below 1.5 m/s. For these conditions, the Sanitary Probe Refractometer PR-43-AP-L42 with steam prism wash, aseptic steam valve ASV, and side flow cell is recommended.

The Process Sanitary Refractometer PR-43-A complies with the standards required for safe food processing and for achieving a high-quality product. The refractometer is available with 3-A Sanitary and EHEDG certifications, and it withstands both CIP cleaning and high process temperatures.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash is required in applications where flow velocity is below 1.5 m/s (5 ft/s) or where dry solids exceed 40 %. The wash media is aseptic steam. The components of a steam wash system are refractometer PR-43-AP-L42 with insertion length of 42 mm, Side flow cell SFC-HHSS-H10/15/20/25, Aseptic steam valve ASV-H/ESS-H05, and Multichannel user interface MI for automatic prism wash diagnostics and control.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

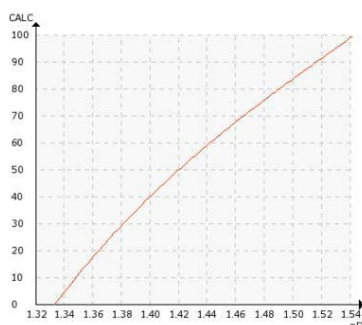


## FRUIT YOGURT

### Typical end products

Flavored chilled dairy products, such as fruit yogurts and desserts.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



it is packaged for sale. In a continuous industrial process, yogurt flavoring is done in-line when it is transferred from the buffer tanks to the filling lines. The amount of ingredients is commonly measured by a variable-speed metering pump which feeds them into the yogurt in a fruit blending unit. The fruit additives can be sweet with a sugar content between 50-55 %, or natural (unsweetened). The ratio of the ingredients is carefully measured to a determined final product Brix level.

After mixing, the flavored yogurt is analyzed for quality control, before it moves on to the filling machine and cool storage.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-AC is mounted on a pipe bend after the in-line mixer. The refractometer measures the Brix concentration of the flavored yogurt for final quality control purposes, before the product passes to the filling machines and is packed.

The measurement of the refractometer is not influenced by particles, bubbles or the color of the medium. The measurement is selective to the liquid phase and fruit chunks, seeds or berries mixed in the yogurt do not influence the measurement.

### Introduction

Yogurt is a very popular chilled dairy product and numerous brands and flavors can be found in the supermarkets' chiller cabinets. Yogurt is made by the lactic fermentation of whole, standardized and skimmed milks.

The consistency, flavor and aroma vary from one district to another. In some areas, yogurt is produced in the form of a highly viscous liquid, while in some countries it is in the form of a softer gel. Yogurt is also produced in frozen form as a dessert, or as a drink.

### Application


Fresh fruit, jam, juice, honey, syrup and other sweeteners may be added to flavor the yogurt before

The refractometer provides Ethernet and 4-20 mA output signals that can be used as real-time feedback for automatic control of the flavoring process. The precise and continuous measurement by the

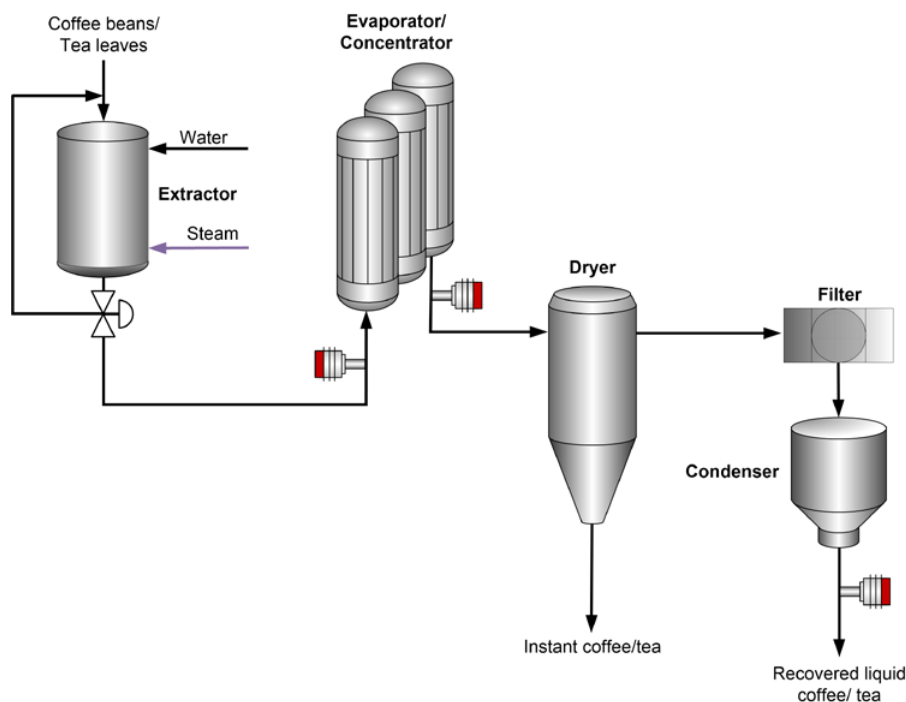
refractometer ensures a correct ratio of yogurt to fruit and a consistent product quality.

If many products are packed through the same filling line, a second refractometer can be used to detect the product-to-product, as well as the product-to-CIP liquid interfaces (see application note *Food and Beverage Interface Detection*).

The PR-43-AC is designed to meet the strict aseptic conditions in dairy processing. The refractometer is available with 3-A Sanitary and EHEDG certifications.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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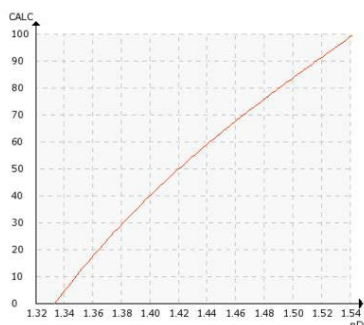


## SOLUBLE COFFEE AND TEA SOLIDS

### Typical end products

Instant coffee, tea extract, instant tea.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Coffee and tea are the most consumed beverages worldwide after water. Coffee and tea are traditionally prepared by brewing coffee beans or tea leaves, respectively. Today, coffee and tea are available as instant powder for easier and quicker preparation.

### Application

Instant coffee is manufactured by using a coffee extraction process. Coffee beans are boiled in water to extract the soluble solids. The coffee extract is evaporated and dried either by spray drying or freeze drying.

A spray dryer is sometimes preferred as it produces fine, rounded particles. In the spray dryer, the concentrated coffee is atomized by a nozzle and warm air is fed to the tower to dehydrate the droplets into particles. Some of the coffee exits the tower with the air. This *coffee dust* passes through a filter to recover the solids. The coffee is dissolved in steam, and the recovered liquid coffee extract is returned to the process.

Tea leaves go through the same process as coffee beans: extraction, concentration and spray drying. The most difficult part in the instant tea process is aroma preservation.

### Instrumentation and installation



Vaisala K-PATENTS® Sanitary Refractometer PR-43-A is used for accurate concentration measurement of soluble solids at different stages of the process.

The refractometer is used as a reliable and real-time indicator of extraction efficiency. Typical measurement range is 0-30 Brix and the normal process temperature is 5 °C (41 °F). Prism wash with high-pressure hot water is recommended in this point.

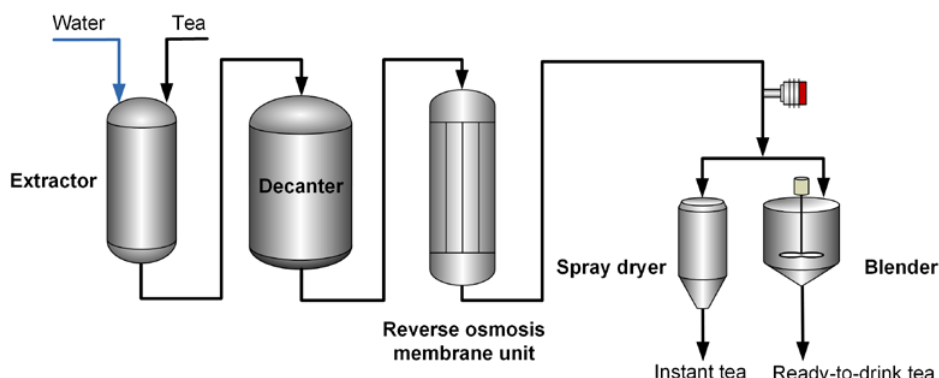
At the evaporators, the refractometer is used to ensure that the desired concentration is fed to the dryer, thus optimizing the energy consumption. The evaporation is a triple-stage process, with stage

concentrations consisting of 10-20 % dry solids, 15-30 % dry solids and 35-65 % dry solids. Under normal circumstances the process temperature is 52-82 °C (126-180 °F). Prism cleaning with steam is recommended at the evaporator outlet.

The refractometer is also used to measure the concentration of recovered instant coffee dust after filtration. Typical measurement range is 8-20 Brix and the normal process temperature is 75-85 °C (167-185 °F).

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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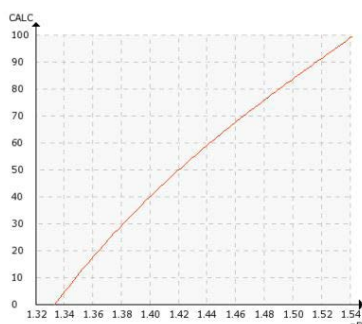


## TEA EXTRACT

### Typical end products

Instant tea, ready-to-drink tea.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Instant tea is a powder that water is added to in order to be reconstituted into a cup of tea. Instant teas are becoming more and more popular due to their convenience in use and variety of tastes.

### Application

In order to produce instant tea, active substances of tea leaves must be extracted from the tea leaf cells by means of aqueous extraction. It is important to preserve the soluble components since they are responsible for the taste, aroma and effect. There are also undesired insoluble components responsible for turbidity, thus they have to be removed from the tea extract by means of separators or decanters. After extraction and separation stages the extract of 2-3 Brix proceeds to a concentration stage.

Tea extracts are heat-sensitive. When exposed to high temperatures, extracts may lose color, flavor and active components. Reverse Osmosis (RO) membrane filtration has been introduced as an

alternative to the traditional thermal concentration. Reverse osmosis is used to concentrate tea extracts at a temperature of 25-30 °C (77-86 °F), causing no changes in the physical structure of water. Moreover, RO produces high product quality and maintains the aroma, flavor, and nutritional characteristics of the extract.


Osmosis involves the movement of pure water through a semipermeable membrane from a low to a high concentration solution. Reverse osmosis occurs when pressure greater than the osmotic pressure is applied to the concentrated solution. Water is then forced to flow from the concentrated to the diluted side, and solutes are retained by the membrane.

In RO membrane filtration the tea extract is concentrated to 15-18 Brix. The Sanitary Refractometer PR-43-AC monitors and controls the concentration of extract in order to comply with the stringent product requirements. If in compliance, the tea extract proceeds to spray drying after which it is refined with ingredients and sent to packaging as instant tea. Alternatively, the extract can be blended to ready-to-drink tea.

### Instrumentation and installation

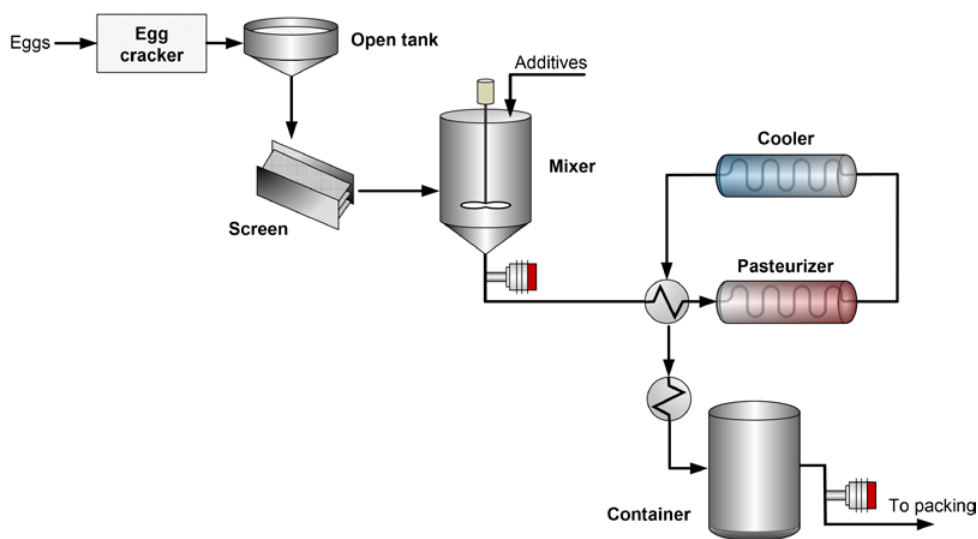
Vaisala K-PATENTS® Sanitary Refractometer PR-43-AC is used to measure extraction efficiency. Typical measurement range is 0-20 Brix and the normal process temperature is 25-30 °C (77-86 °F). The process pressure ranges between 20 and 30 bar. The refractometer is installed on the pipe bend after the RO membrane unit, and before the tea extract is sent to the final stage of production.

A prism cleaning system, using high pressure water is recommended in this application.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash with high pressure water. The components of a high pressure water system are a sensor with integral water nozzle mounted at the sensor head, a high pressure pump together with a power relay unit and an indicating transmitter equipped with relays.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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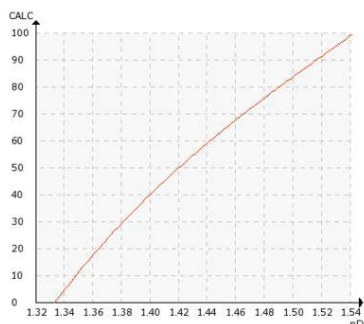


## EGG

### Typical end products

Egg white, egg yolk, whole egg, egg powder.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



## Introduction

The egg processing operation separates eggs into different egg products: egg white, egg yolk, whole egg and several mixes, e.g. by adding sugar or salt. The pasteurized liquid egg is either packed as a final product or, in case of powdered eggs production, it goes via pipelines into a spray dryer plant.

It is very important to verify the concentration of the final egg product. This enables the supplier to assure customers that the products coming out of the processing plant meet the specifications.

Strict hygiene control is essential throughout the production process to ensure there is no contamination of the products. During the egg refining process the mass is tested several times to insure that all the products are clean. When the product is ready, it must

be stored at a temperature of 4 °C (39 °F). The storage temperature required for egg yolk is 12 °C (54 °F).

## Application

The eggs are fed in to a cracker/separator, where each egg has its own cup. Then, the egg shells are cut and the egg white flows off the cup while the yolk is retained. Usually there is a separate pipe for each product, leading to separate open tanks.

The egg white, the yolk and the whole egg go through a screen before they are floated into their own containers or mixing tanks. They are screened to remove all unwanted particles. When the product is a mixture, the additives are introduced into the mixing tank.

The next phase is pasteurization. The mass is preheated through a heat exchanger before pasteurization. The yolk is pasteurized at 60 °C (140 °F) and the egg white pasteurized at 57 °C (135 °F). When the mass leaves the pasteurizer, it is cooled by utilizing the same heat exchanger, which it passed through before entering the pasteurization tank.

The pasteurized mass is stored in product tanks before it moves on for packaging. If the level of dry solids for the whole egg mass is not high enough, egg yolk can be added to increase the dry solids to the specified level before packaging.


## Instrumentation and installation

Refractive index measurement is needed at different stages in this process. The most common measurement points are in-line after the mixing tank, or optionally directly in the final product mixing tank prior to packaging.

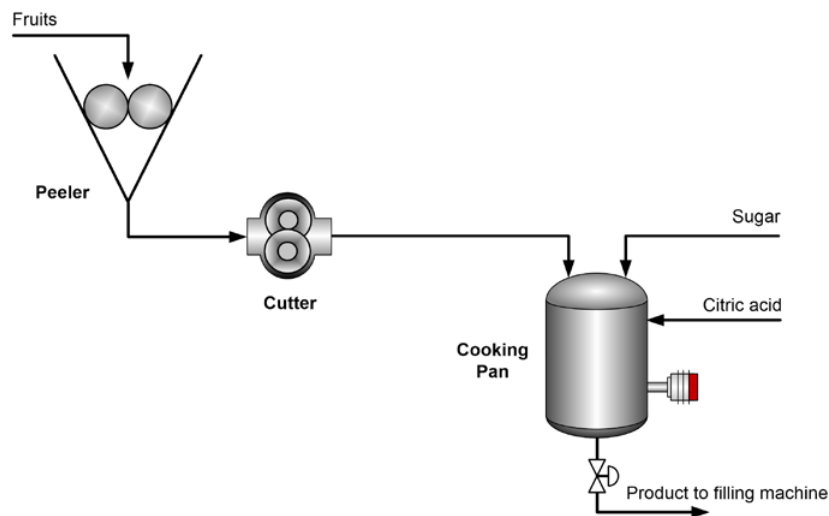
The dry solids content of the yolk, egg white and whole egg are measured before and after pasteurization. Typical dry solids content of a whole egg is 26.2 % before pasteurization and 26.0-26.5 % after. With egg yolk the values are 45.0 % and 43.0-44.5 %, respectively. For egg white 14.5-15.5 % dry solids is a typical value after pasteurization.

Vaisala K-PATENTS® Sanitary Refractometer PR-43-A is available with 3-A Sanitary and EHEDG certifications. Therefore, it meets the stringent hygiene requirements for egg refining processes.

The measurement by the refractometer has two important functions in egg processing. First, it replaces periodical sampling of the water to dry solids interface; and second, it provides real-time quality control measurement. Both of these, speed up production and reduce wastage.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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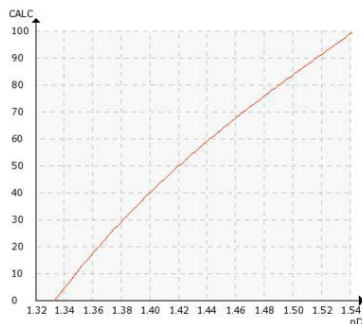


## JAM, JELLY, MARMALADE

### Typical end products

Jams, jellies, marmalades, and similar fruit preserves.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Jam-making is an industry that converts fruits into a spread mixture. Fruits in the form of puree, pulp or juice are dissolved in water in the presence of a syrup, concentrated to a high dissolved material content by cooking, and cooled rapidly to obtain a mass with the appropriate gel consistency.

### Application

Jam cooking is usually performed in batches of 500 to 3000 kg (1100 to 6600 lbs). Each batch takes 1-3 hours and consists of four main phases:

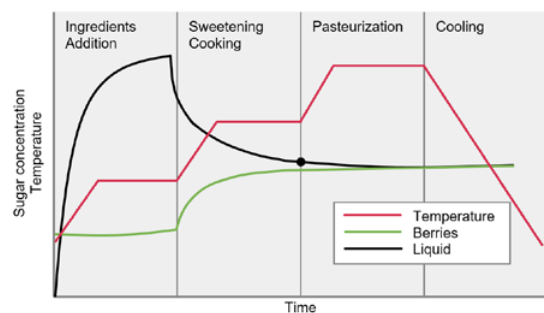
- Ingredients Addition:** Berries, fruits, pectin and sugar are mixed with water. Frozen berries thaw as temperature slowly increases.
- Sweetening Cooking:** In this phase, berries and fruits absorb sugar from the liquid until equilibrium is

achieved. The temperature of this process is below 90 °C (194 °F). This phase is not required if there are no solid substances present in the medium.

**3. Pasteurization:** Cooking temperature is raised up to 100-150 °C (212-302 °F) for 10-20 minutes to destroy any bacteria.

**4. Cooling:** The jam is cooled down to 20-40 °C (68-104 °F) before the vessel is discharged. Aromas are often added during this phase.

Changes in the sugar content, temperature and liquid at different cooking phases are illustrated below:



### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Probe Refractometer PR-43-AP-L or the Flush Mounted Refractometer PR-43-AP-T provide accurate and in-line Brix measurement. The refractometer allows for continuous monitoring of the cooking process to ensure consistent product quality and to optimize the process.

Traditionally, it has been possible to check Brix levels off-line through batch sampling at the end of the process. The refractometer can be installed directly in any type of cooker to monitor the batch progress and final product quality. The refractometer has a measurement range of 0-100 Brix and provides Ethernet or 4-20 mA output signals for real-time control. The refractometer's signal can be used to set quality control functions to take immediate corrective actions if the batch reaches unusually low or high Brix levels.

The refractometer eliminates the need for batch sampling and off-line analyzing, improves product consistency and quality, and optimizes sugar usage. Typical measurement range is 10-70 Brix (jam cooking) and 50-85 Brix (marmalade cooking), and the process temperature is about 70-90 °C (158-194 °F).

## Open boiling pan

The open boiling pan is a traditional cooker type in which the jam is steam heated from the bottom. A scraper or agitator prevents the jam burning from the contact with the hot pan.

The Sanitary Probe Refractometer PR-43-AP-L is installed in a pipeline or vessel using a 2.5 inch or 4 inch Sanitary clamp. The Flush Mounted Sanitary Refractometer PR-43-AP-T is designed for vessels containing scrapers and mixers. These refractometers are installed through an APV Tank Bottom Flange and can also be installed through steam jackets.

## Vertical vacuum cooker

The vertical vacuum cooker is more efficient than the open pan type. During heating and pasteurization, the high pressure prevents overcooking and the berries remain unbroken.

The probe refractometer is installed in the vessel bottom, where it is in continuous contact with the jam and where the cooling effect from the vessel is at its best.

## Horizontal vacuum cooker

An Ala vacuum cooker is horizontally mounted and has double the capacity of vertical pans. These cookers are fitted with a heat exchanging agitator/scraper. They also contain a spiral coil heater, which enables faster heating and cooling either under overpressure or under vacuum.



A probe or flush mounted refractometer can be installed either via the steam jacket or at the end of the pan (avoiding having to cut an opening through the steam jacket).

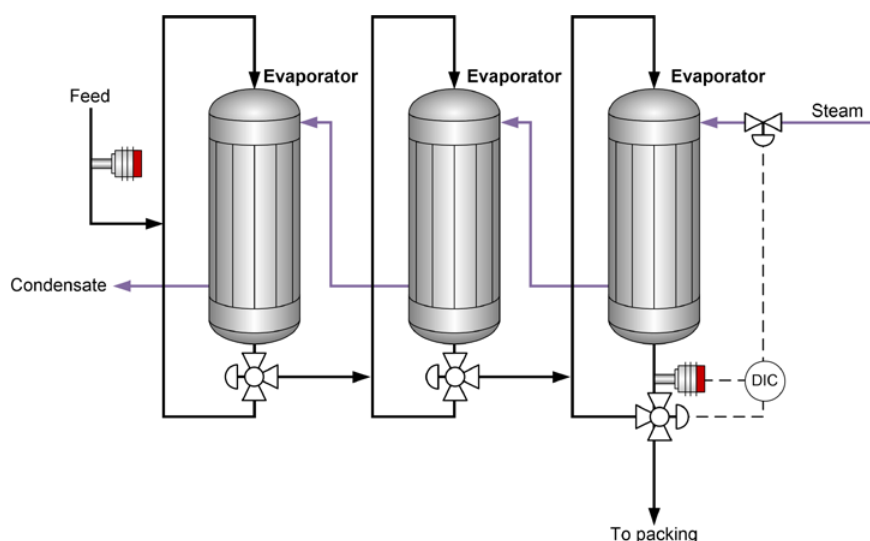
## Pipe cooker

The pipe cooker is a continuous flow cooker, where the whole cooking process takes place. Berries, fruit, sugar pectin and other ingredients are mixed and preheated, and then pumped through the cooking tubes for further heating. After cooking, the product is passed through cooling pipes before packaging. The flow velocity is 0.1 m/s for the whole process.

The probe refractometer is installed after the cooking phase to measure the concentration of the end product, since the sweetening of the berries occurs during heating. At this point, the temperature is at its highest and the risk for prism coating due to low product flow rate is avoided. The refractometer can also be installed in the feeder tank to estimate the product concentration and to determine the additives quantities to be introduced during cooking, thus optimizing cooking time.

Prism coating is rarely an issue because the batch processing times and CIP cleaning intervals are short.

Instrumentation	Description
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Sanitary Flush Mounted Refractometer PR-43-AP-T for hygienic flush mounting installations in cookers, cooling crystallizers and other vessels that have scrapers or mixers. Installation through an APV Tank bottom flange.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

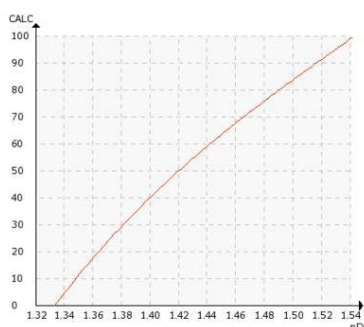


## TOMATO CONCENTRATE

### Typical end products

Tomato concentrate, tomato paste, tomato puree.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

It is vital that the tomato concentrate processing line operates constantly at a full capacity. It is not possible to work at reduced capacity, intermittently or every other day. Every time the tomato processing line is shut down, all the machinery must be cleaned. This involves several working hours, the consumption of a great amount of water and wastage of the product contained in the evaporator.

Furthermore, fresh and ripe tomatoes cannot be held in storage for processing at ambient temperatures over 30 °C (86 °F) for more than 24-48 hours. This will result in an inferior, low quality final product with a low Brix level.

The entire production cycle is carried out according to extremely stringent sanitary and hygiene standards.

### Application

Tomato juice concentration involves the reduction of the water content, leaving all the original solids as a concentrated solution.


Typically, single or multiple-stage (also called effect) evaporation plants are used for the tomato concentrate production. The juice inside the evaporator flows through different stages, where its concentration level will gradually increase until the required density is obtained at the final stage. Here, the tomato paste is automatically extracted via a pump controlled by the Process Refractometer.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-AC provides continuous concentration information to optimize the evaporators performance while reducing energy consumption.

The refractometer is installed on the evaporator outlet. The signal from the refractometer adjusts the evaporator inlet flow or the steam flow to regulate the final Brix value. Typical measurement range is 5-35 Brix and typical process temperature about 95 °C (203 °F).

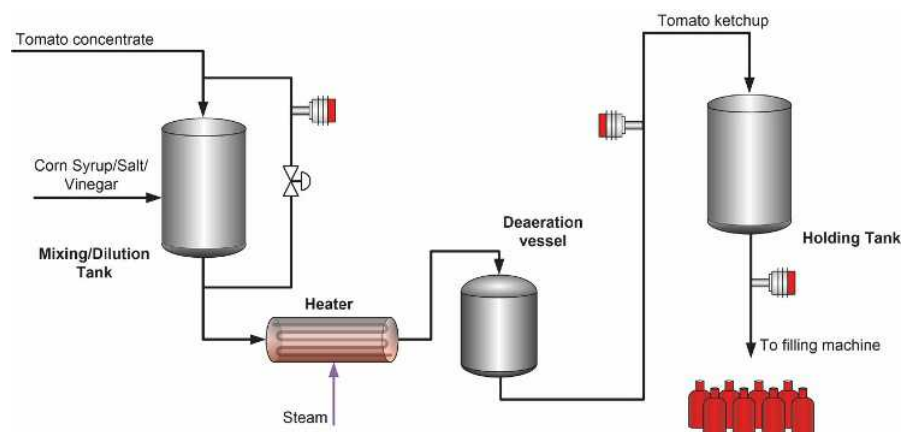
The refractometer is also used for tomato pulp standardization control. Typical measurement range is 4-7 Brix and typical process temperature about 20 °C (68 °F).

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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## Tomato ketchup and sauces from tomato concentrate plant

Food and Beverage | Fruits and Vegetables Processing | 2.08.03



### Measured medium

- Tomato ketchup and sauces

### Typical end products

- Tomato ketchup, tomato sauce.

### Introduction

Tomato ketchup/sauce is one of the most common used condiments. The recipe, viscosity and solids content of sauces and ketchups vary widely. Typically, they are made from tomato concentrate, but the recipe can be based on whole peeled tomatoes.

Ketchup and tomato sauce also contain water, sugar, vinegar, salt and seasoning. A starch-based thickener is also added to achieve the desired product viscosity

### Application

The ketchup manufacturer utilizes tomato paste or puree from a tomato concentrator. In the mixing tank the product is then diluted with water to the desired Brix. After that, some sugar or sweetener, salt and preservatives

are added to the mixture according to the recipe.

After the tomato paste has been diluted and mixed with other ingredients it proceeds to sterilization and de-aeration. The ketchup must be de-aerated to prevent discoloration and growth of bacteria. Once mixed, the product may be passed through a highpressure homogenizer or colloid mill to obtain the required consistency.

Ketchup is then kept in the holding tank to be further packed in containers, glass bottles or pouch packs.

During the ketchup preparation process it is very important to continuously monitor and control the concentration of the product, as it affects the consistency of the ketchup.

### Instrumentation and installation


Vaisala K-PATENTS® Sanitary Refractometer PR-43-A is installed at three locations:

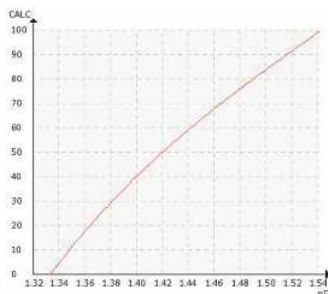
1. In the mixing/dilution tank. The refractometer ensures a constant Brix-value of tomato paste, as the paste from different suppliers may differ in concentration.
2. In-line to final product holding tank. After the sterilization and de-aeration processes the concentration of the ketchup may vary. The refractometer provides the final quality control measurement before storing the ketchup in a holding tank.
3. In the filling line. The refractometer measures the concentration of the end product before bottling.



The refractometer provides constant quality control over the entire process, and assures high quality of the end product. The refractometer is available with 3-A Sanitary and EHEDG certifications. The instrument design withstands CIP- and SIP-cleaning.

The PR-43-A refractometer meets the demands of National Regulations aiming at Safety of Food Contact Materials. Moreover, all our products fulfil the material traceability requirements of the food industry.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.
Chemical curve	R.I. per BRIX at Ref. Temp. of 20°C



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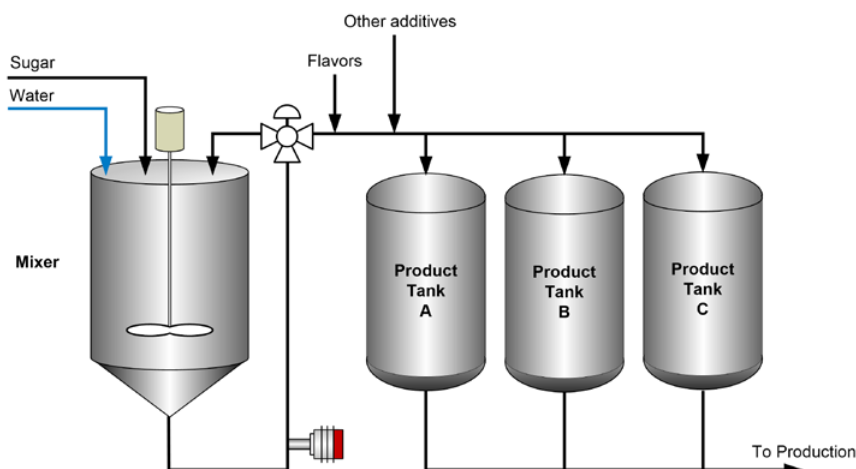
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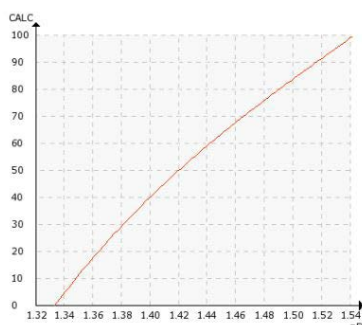


## STANDARDIZED SUGAR SYRUP

### Typical end products

Sugar syrup for the preparation of various products such as soft drinks, confectionery and canned products.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



## Introduction

In the beverage, confectionery and canned product industries, sugar is mostly processed as an aqueous solution or syrup. Therefore, the first stage of the process is the preparation of the *standardized sugar syrup*.

Syrup is prepared simply by mixing sugar with water to a concentration of 60-67 °Brix. The dissolving process can be discontinuous or continuous and the system can be hot or cold.

## Application

The crystal sugar is typically dissolved in water, using one of a variety of mixing techniques. The most efficient way is continuous dissolving using a *jet mixer*, which ensures extremely rapid dissolving.


In order to obtain a uniform product quality, the Brix concentration of the sugar solution must be carefully controlled. For example, too much or too little sugar affects the composition of toffee, and too much sugar in soft drinks means uneven quality and excessive use of the other ingredients.

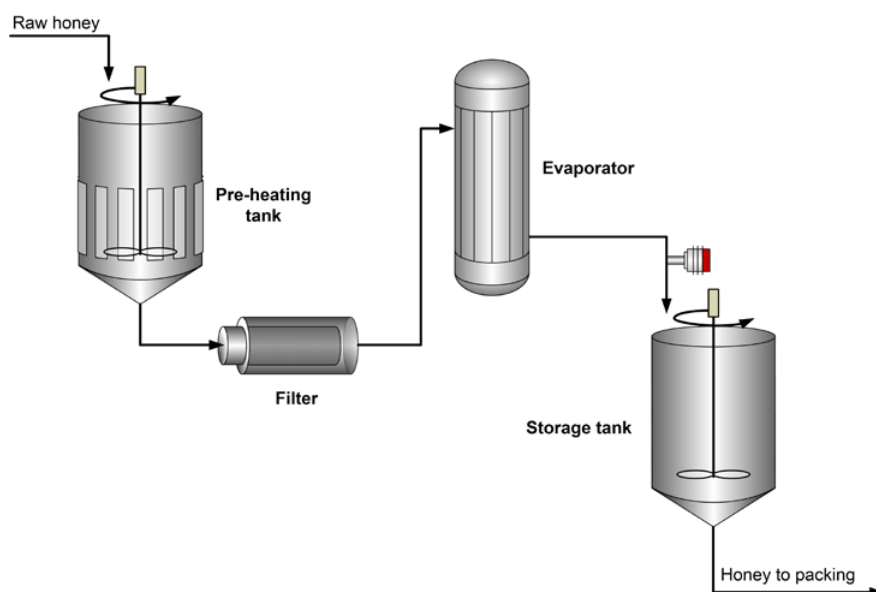
## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-AC is installed in the mixer outlet to measure the concentration of dissolved sugar.

The mixed sugar solution contains a large quantity of air bubbles. These bubbles have an influence on traditional density measuring devices. Due to its unique digital sensing technology, the refractometer only measures the liquid concentration, and the measurement is unaffected by the presence of air bubbles.

The precise and rapid in-line measurement obtained by the refractometer, helps to optimize processing time and to save raw materials.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

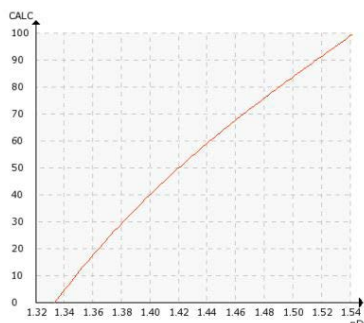


## HONEY

### Typical end products

Different honey products such as liquid, cut comb and creamed honey.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Honey is a syrupy biological product produced by honeybees from the nectar of flowers. It contains glucose, fructose and water, in addition to small quantities of proteins, minerals, organic acids and vitamins. However, its exact composition and color depends on the type of flower that supplies the nectar.

Honey is consumed in every country in the world in some form. It is mostly used as a sweetener for drinks or in the preparation of foods, but also as a medicine and health product.

### Application

After the raw honey is extracted from the honeycombs, it is poured into drums and taken to a commercial distributor for processing.

The main steps in honey processing are filtration and heating. Honey is firstly poured into a tank and pre-heated to 49 °C (120 °F) to melt out possible crystals and to reduce its viscosity for easier processing. A filtration step follows to separate the pollens, beeswax and all other visible impurities.

After all suspended particles are separated, the honey is heated, usually in an evaporator. Thermal processing eliminates the microorganisms responsible for spoilage, and reduces the moisture content to a level that retards the fermentation process. The final moisture content is usually between 17-18.5 % (81.5-83 % honey). However, the chances of crystallization will increase as the moisture content is reduced, so it is important to monitor carefully the concentration of the treated honey.

After thermal treatment, the honey is cooled and stored until final packaging.

### Instrumentation and installation


Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-AC is installed after the evaporator or heater to measure in real-time the concentration of honey.

Honey contains a sugar-tolerant yeast which will ferment if the moisture content is too high (above 20 %), giving the honey a foul taste. On the other hand, if the moisture content is too low the honey may start to crystallize. The refractometer is installed in-line to provide continuous and accurate concentration or Brix measurement, thus ensuring the final moisture content in the honey is within the safe level.

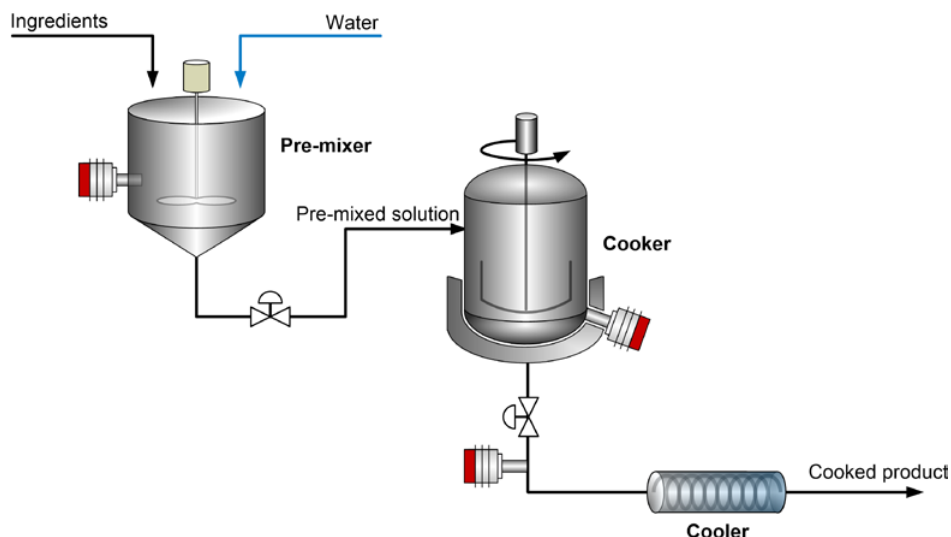
Some distributors may blend the processed honey with unprocessed honey to adjust the moisture content to the required level. In these cases, another refractometer can be installed to control the ratio of the components to achieve the right concentration.

The PR-43-AC is designed to comply with all the strict regulations for safe food and beverage production. The refractometer is available with 3-A Sanitary and EHEDG certifications.

Concentration measurement in honey processing with the refractometer guarantees a high product quality, which is safe for long-term storage.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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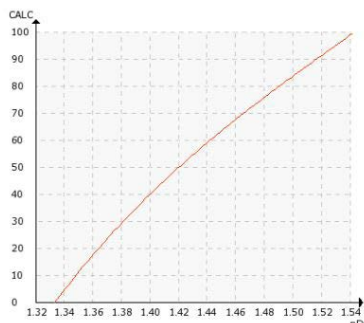


## CARAMEL, CHOCOLATE FILLING, MARSHMALLOW

### Typical end products

Cooked sugar confections such as caramel, chocolate filling, candies, jelly beans, marshmallows and dulce de leche.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Sugar confections are food products rich in sugar and carbohydrates. Sugar confections include candies, chocolate fillings, chewing gum, marshmallows and other sweet items which are made mainly from sugar.

Sugar confectionery is a large and ever-changing industry. In order to meet the quality and demand, manufacturers need sophisticated technology and equipment to process the ingredients.

### Application

Sugar confections and chocolate fillings are produced by preparing a diluted mixture of the ingredients, and then concentrating the sugar content to the desired level by cooking and evaporating the water.

The ingredients are mixed either in a pre-mixing vessel and then transferred for cooking, or added directly in the cooker. The cooker is a heated vessel, usually a jacketed vessel equipped with scrapers, where water is evaporated from the liquid for raising its sugar content to a set level. The final Brix of the mixture determines the flavor, consistency, and overall quality of the final product.

The ingredients used, as well as the cooking conditions are defined by the type of final product. For example, to produce caramel, sugar is dissolved in water to prepare a liquid syrup which is thickened in a cooker. The cooking of the syrup also gives the final product, caramel, its distinctive color. The final sugar concentration ranges between 85 and 95 Brix, and it should be monitored reliably to ensure high quality and to avoid re-processing. If the moisture content is higher than desired, it will affect the consistency and increase the risk of mold formation. If the moisture content is too low, the caramel may dry faster than is desired, making upstream processing difficult.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A measures continuously the Brix level of the cooking solution. This provides real-time information to determine the end-point of cooking, and to ensure a consistent product quality. Typical concentration in this application ranges between 85-97 Brix, at a temperature of 100-130 °C (210-266 °F).




The refractometer can be installed directly in the cooker or in the cooker's outlet pipe. In a batch process, the flush mounted refractometer PR-43-AP-T is installed at the bottom of the vessel through an APV Tank Bottom Flange. The refractometer is specially designed for vessels with scrapers and mixers and can also be installed through steam jackets.

For continuous cooking, the refractometer is installed directly at the outlet of the cooker. The refractometer provides Ethernet and 4-20 mA output signals that can be used for real-time process control. If the Brix level after cooking is different than the specified value, the refractometer sends a signal to the process controller to control the valves as needed. For example, the controller opens the valve to recycle the product for re-processing, and sends information to adjust

the ingredients ratio. Another refractometer can be installed at the pre-mixer to ensure the right feed concentration to the cooker.

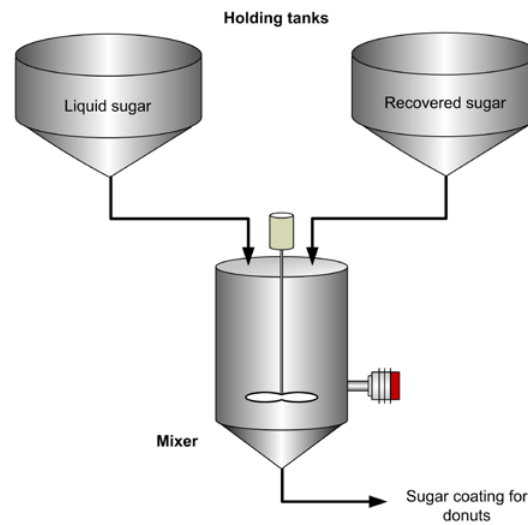
The measurement by the refractometer is continuous and unaffected by suspended particles or bubbles. The real-time information provided by the refractometer eliminates the need for sampling and reduces processing time. Continuous measurement is particularly important in cookers that work under vacuum. The refractometer prevents disrupting the process and saves valuable processing time.

In direct cooker installations the Teflon scrapers keep the prism clean during the cooking process. Other installations may require a prism wash.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Sanitary Flush Mounted Refractometer PR-43-AP-T for hygienic flush mounting installations in cookers, cooling crystallizers and other vessels that have scrapers or mixers. Installation through an APV Tank bottom flange.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash is required in applications where flow velocity is below 1.5 m/s (5 ft/s) or where dry solids exceed 40 %. The wash media is aseptic steam. The components of a steam wash system are refractometer PR-43-AP-L42 with insertion length of 42 mm, Side flow cell SFC-HHSS-H10/15/20/25, Aseptic steam valve ASV-H/ESS-H05, and Multichannel user interface MI for automatic prism wash diagnostics and control.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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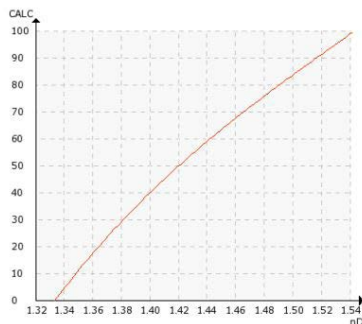


## LIQUID SUGAR

### Typical end products

Sugar coated donuts and other bakery products.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



## Introduction

Sugar coating of donuts and other specialty bakery products is very common. Coating is done by topping the product with a sugar mass prepared with granulated or a powdered sugar. The sugar coating is usually flavored to meet different preferences by the consumers.

Certain sugar preparations are specifically formulated to increase the shelf life of the donuts. Fine and uniform sugar coating also plays a key role in the bakery products' final appearance, flavor and quality.

## Application

Sugar flows are applied over the surface of baked donuts for an even all-round coating. It is the powdered sugar quality that affects the final product coating.




Liquid sugar is prepared from powdered or granulated sugar. This *fresh liquid sugar* is mixed with *recovered sugar* to prepare a coating mix according to the recipe specifications. The finer the sugar mix, the better it adheres to the donut surface.

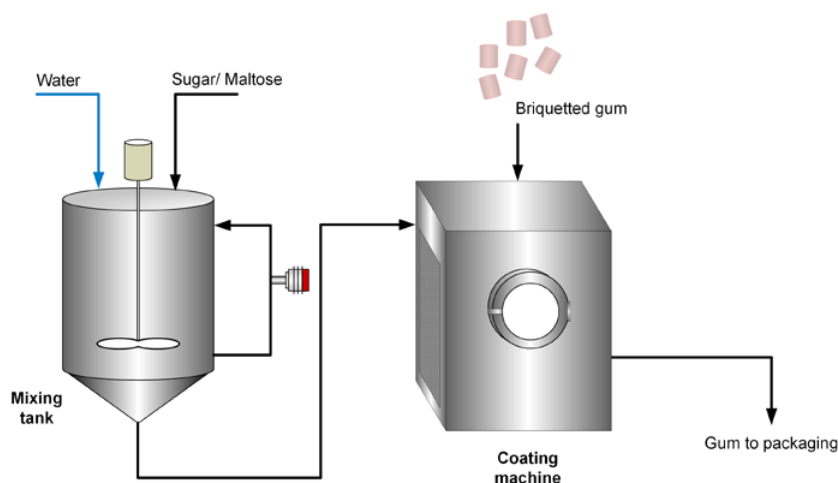
Vaisala K-PATENTS® refractometer is used for quality control of the sugar mix before the final stage of the donuts preparation process, i.e. before the sugar coating is applied to the baked donuts. The excess of the sugar mix is shaken off and collected for reuse.

## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Probe Refractometer PR-43-AP is installed directly at the lower part of the mixer. The refractometer measures the Brix of the sugar coating before it is spread over the surface of the baked donuts. A Flush Mounted Refractometer PR-43-AP-T is available for vessels with a scraper or mixer.

The PR-43-AP refractometer is designed to meet the strict sanitary requirements for food and beverage processing. The refractometer withstands Clean-In-Place (CIP) conditions and is available with 3-A Sanitary and EHEDG certification. Moreover, the refractometer provides a full measurement range corresponding to 0-100 Brix and is automatically temperature compensated.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Sanitary Flush Mounted Refractometer PR-43-AP-T for hygienic flush mounting installations in cookers, cooling crystallizers and other vessels that have scrapers or mixers. Installation through an APV Tank bottom flange.
<b>User Interface</b>	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

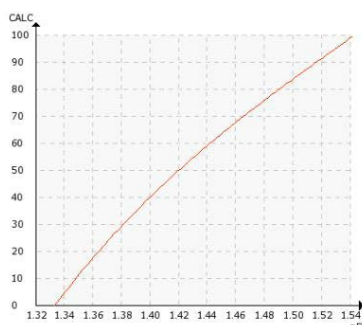


## SUGAR SYRUP

### Typical end products

Pillow-shaped chewing gum, other sugar coated confectionery products.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Confectionery coating plays a decisive role in the final product's appearance and functionality. Fine coating has an effect on the flavor and the quality of confectionery products, such as chewing gum. Moreover, if applied unevenly the coating can result in inconsistencies in the chewing gum pad sizes, thus, causing packaging problems.

### Application

Preparation of syrup for chewing gum coating starts with adding the syrup components, in particular, sugar or its substitutes, e.g. maltose, and water to the mixing tank. The exact syrup concentration is critical for securing the correct flavor and to prevent the gum pads sticking together in the coating machine (drum).

Chewing gum is pillow-shaped when fed to the coating machine. The briquetted gum is then sprayed with

the sugar syrup. The mixing process takes about 5 hours. During this time, up to 40 microthin sweetener layers are applied step-by-step to the chewing gum pads to produce a crunchy coating. For instance, over 100 000 pieces of coated briquetted gum can be produced from a 200 kg batch.

The concentration of the sugar syrup must be carefully monitored. If the syrup is out-of-specification, it may result in the gums getting sticky and the whole batch being spoiled. This may also cause drum damage.


After the chewing gum has been coated, it proceeds for packing.

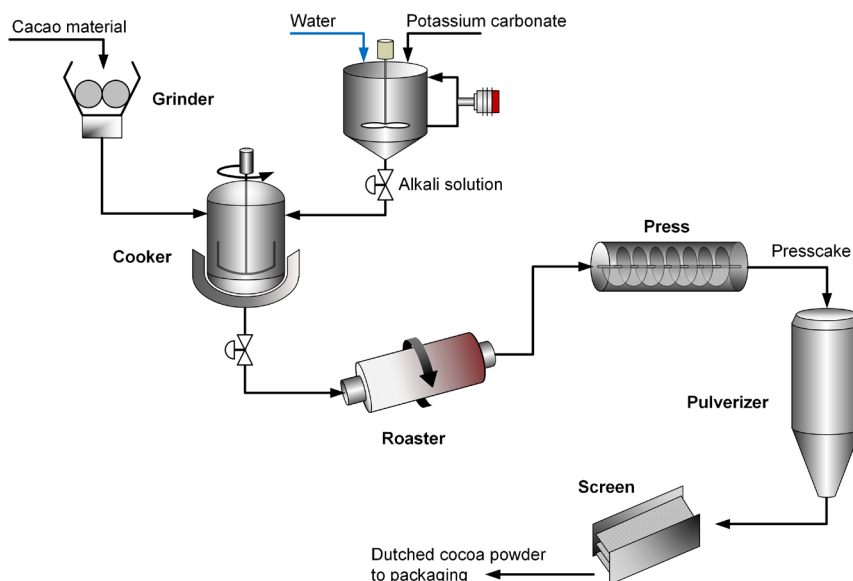
### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-AC is used to measure the concentration of the sugar syrup upstream before it is sprayed to the gum for coating. The installation of the refractometer assures an efficient chewing gum manufacturing process and prevents the risk of defective batches.

Typical installation is the elbow flow cell installation on the recycling pipe.

The refractometer is available with 3-A Sanitary and EHEDG certifications. Brix monitoring with the refractometer allows for continuous and real-time quality control of the manufacturing process.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

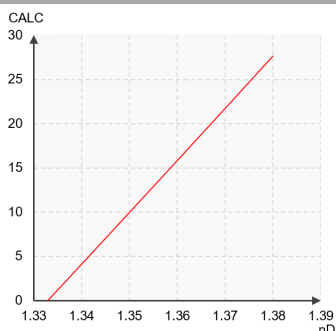


## POTASSIUM CARBONATE $K_2CO_3$ , SODIUM CARBONATE $Na_2CO_3$ , AMMONIUM BICARBONATE $NH_4HCO_3$

### Typical end products

Dutched cocoa powder for making chocolate and other chocolate-flavored foodstuffs such as confectionery, desserts and drinks.

Chemical curve: Potassium Carbonate R.I. per % b.w. at Ref. Temp. of 20°C



### Introduction

Cocoa powder is the most important ingredient in the preparation of chocolate and other chocolate-flavored sweet and confectionery, bakery products, ice creams and drinks. Cocoa powder is produced by processing the beans from cacao plants.

There are two types of cocoa powder. *Natural cocoa powder* is brown in color, and naturally acidic (with a pH between 5 and 6). *Dutch process cocoa powder* or *Dutched cocoa powder* is made from cacao beans that have been washed with an alkaline solution to neutralize their acidity to a pH of 7. *Dutching* produces a variety of cocoa powders for specific applications. Dutched cocoa powders have a softer flavor, deeper

brown or red color, and even improved solubility in liquids compared to the natural ones.

Dutched cocoa powders are more commonly used in Europe. In America, most cocoa powder brands available in the supermarket are of natural type. However, Dutched cocoa powders are widely used in the making of american style chocolate sandwich cookies, since cookies prepared with these acquire an attractive, rich chocolate color.

### Application

*The Dutching process*, also known as *Alkalizing* or *Alkalization*, simply consists of reacting cacao material with an alkalizing agent, typically in the form of an aqueous solution.

Dutching can be done to cocoa bean kernels, or nibs, cocoa liquor or even directly to cocoa powder. Cocoa beans are harvested and fermented for some days to change the flavor, aroma and color of the bean to brown. After fermentation, the cacao beans are cleaned and usually broken in pieces known as *nibs* before the manufacture of chocolate or cacao products. The nibs can also be treated with steam or roasted before alkalization.

For Dutching, the particle size of the cacao material is further reduced, for example in a grinder. The ground cacao material is then mixed with an aqueous alkali solution at a temperature around 90 °C (200 °F).

The alkali agent used can be potassium hydroxide, potassium carbonate, sodium hydroxide, sodium carbonate, sodium bicarbonate, ammonium bicarbonate, ammonium hydroxide, or mixtures of these. The alkali and its concentration are specific of the desired properties and end use for the cocoa powder being produced. For instance, potassium compounds are used to obtain a dark red product. Some countries may have regulations on the level of alkali used for dutching, so it is of outmost importance to monitor and control the preparation of the alkalizing solution. The total amount of alkali concentration ranges from 1-20 % based on the weight of the cacao material.

The reaction happens in a closed jacketed vessel. After addition of the alkali solution, the temperature is raised to about 120 °C (250 °F). The total cooking time depends on many factors including the alkali concentration, the cacao material being treated, and the temperature employed, but usually it is about 10-30 hours. The process conditions differ for each final product. For example, for reddish colored cocoas, air is usually introduced into the mixer during reaction, for black cocoas, the introduction of air is minimized. The cooking can be done under atmospheric conditions, under pressure or under vacuum to facilitate the following drying step.




Once cooking is completed, the dutched cacao material is removed from the cooker. Then the nibs are roasted, if not previously done so, cooled to room temperature and milled to form a chocolate liquor and pressed to remove fat and create a presscake. Some producers remove the fat already before dutching to minimize the amount of soap produced from the reaction of fat (or fatty acids) in the cacao material and the alkali.

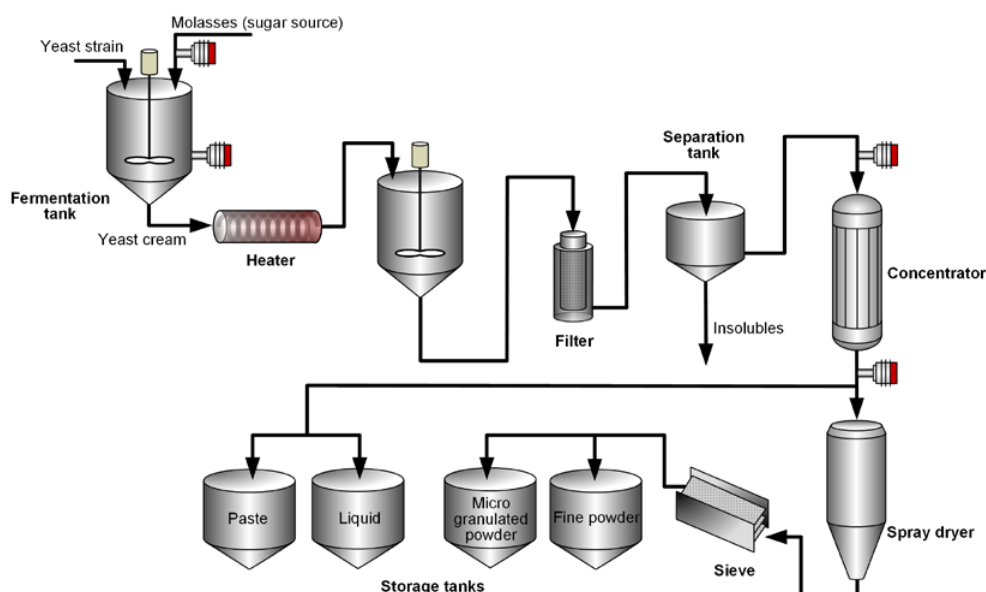
The cocoa presscake is pulverized, dried and screened to obtain a final dutched cocoa powder product.

## Instrumentation and installation

The type and concentration of the alkali used for dutching are critical parameters to obtain the desired dutched cocoa powder. The Vaisala K-PATENTS® Process Refractometer can be installed directly in the alkali preparation tank to measure in real time the concentration as the aqueous alkali solution is prepared. The refractometer can be installed in a recirculating line, or directly in the vessel. A refractometer flush mounted option is available for vessels with stirrers.

The accurate measurement by the refractometer ensures that dutching takes place at the correct alkalizing levels, in order to comply with regulations and to obtain the correct dutched cocoa powder product which is suitable for the desired end application.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Sanitary Flush Mounted Refractometer PR-43-AP-T for hygienic flush mounting installations in cookers, cooling crystallizers and other vessels that have scrapers or mixers. Installation through an APV Tank bottom flange.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % b.w.

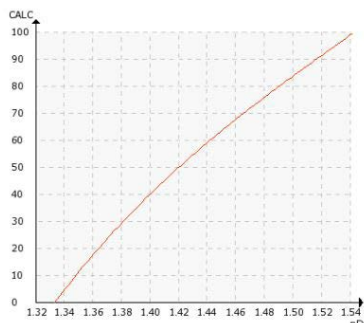


## YEAST EXTRACT SOLUTION

### Typical end products

Yeast, food ingredients, flavorings and seasoning, nutrients for bacterial culture media and skincare products.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Yeast extract is a processed product, which comprises the water-soluble components of yeast cells.

Yeast extract is used in a broad range of applications. In the food industry, it is used as food flavoring, e.g. in soups, sauces and ready meals. Yeast extract is also used as an additive, vitamin supplement, as well as nutritional source for bacterial culture media.

### Application

Yeast extract is prepared by extracting the cell contents from *baker's or brewer's yeast (Saccharomyces cerevisiae)*. The process starts with the selection of the right yeast strain that meets the customer's needs. This yeast strain is the inoculum fed into a tank where it grows at sterile conditions before it is transferred

to a larger fermenter. The strain is cultivated in progressively larger vessels until it reaches the capacity of the production fermenters. During cultivation air and a sugar source, for example molasses, is added to nourish the yeast. The fermentation allows yeast to grow in an aerobic environment and at a controlled temperature (around 30 °C or 86 °F). After fermentation the residual sugar is removed, and the yeast is washed and concentrated. The product from fermentation, known as *the yeast cream*, undergoes a heat treatment at a moderate temperature that allows opening the cells while conserving the enzymes.

The next step is *autolysis*. Autolysis is the process by which a cell will digest itself through the action of its own enzymes, releasing the intracellular content. Autolysis involves the freeing of enzymes within the yeasts to break down the proteins. This step of the process is performed under mild pH and temperature. Its duration depends on the type of yeast and the extent the proteins must be broken down.

After autolysis, the insoluble part in the solution, such as yeast cell walls, is separated from the water-soluble components. Separation is done by filtration. The water-soluble part will constitute the *yeast extract*.

Before proceeding to packaging or spray drying, the yeast extract must be concentrated and pasteurized. In this step, water is removed from the solution to achieve a desired concentration. The final concentration must be measured continuously to ensure a high-quality product and optimal operation of the downstream equipment. The concentration for



a liquid product must be 50-65 % dry solids, and for a paste yeast 70-80 % dry solids. The yeast extract can be dried to a fine powder or granulated particles by spray drying.

The final yeast extract is then packed.

## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-A allows yeast extract plants to continuously monitor different stages of their process. The refractometer can be used at the initial step to control dilution of molasses and to monitor that the sugar source for cell cultivation is at the correct Brix level. This ensures that enough nutrients are supplied for cell growth and enhances the yeast cultivation process.



The refractometer can also be installed directly in the fermentation tanks to follow reaction progress and to monitor yeast content inside the vessel. The measurement by the refractometer is selective to

the dissolved material in the liquid phase and is not affected by suspended solids or bubbles.

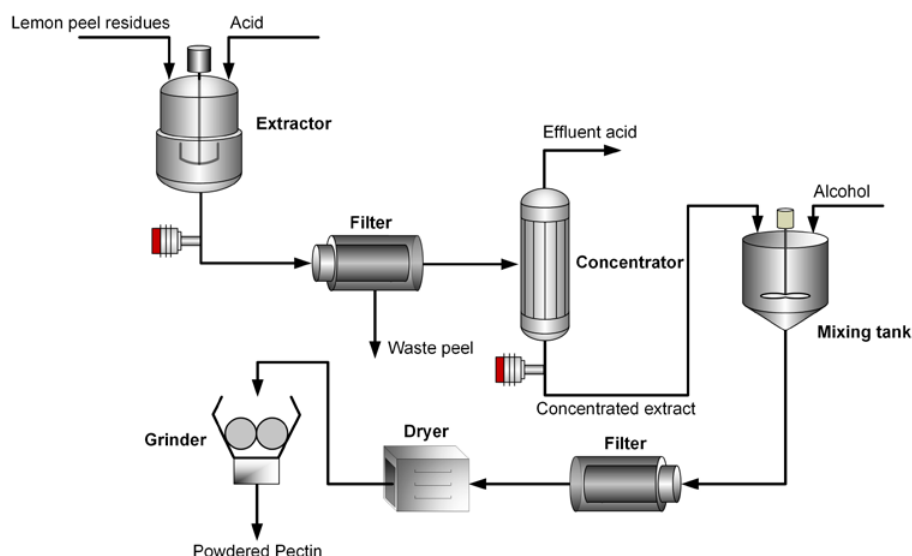
At the concentration step, the refractometer is mounted directly on the pipe bend after the concentrator or pasteurizer. The refractometer measures in-line the concentration of the yeast extract to ensure the target dry solids level is achieved. In addition, a refractometer can be installed before the evaporator or concentrator. The refractometers' mA or Ethernet output signals can then be used not only for quality control, but also for controlling automatically the process and for optimizing energy consumption.

Typical temperature in this application is 30-60 °C (86-140 °F), and the measurement range is 0-100 Brix. Automatic prism wash might be required in this application.

The PR-43-A is designed to meet the highest hygiene requirements. The refractometer is available with 3-A and EHEDG certifications.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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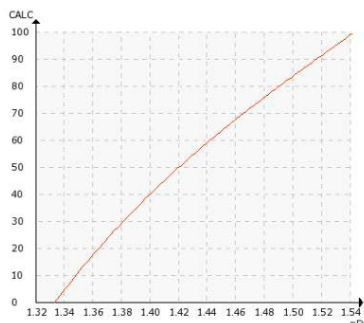


## PECTIN SLURRY, PECTIN EXTRACT

### Typical end products

Powdered Pectin.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Pectin is an acidic polysaccharide that occurs in the cell walls of a fruit. Pectin has excellent water binding and gel forming properties even at low concentrations. For this reason, the main use for pectin is as a food additive for gelling, thickening and stabilizing in the food industry. Pectin is also used in the cosmetics industry in the manufacture of oils and creams, paints, toothpaste and shampoos, and in the manufacture of wound healing preparations and gel caps for the pharmaceutical industry.

The major sources of pectin are citrus peels, the waste from extraction of lemon and orange juices, and apple pomace, the dry residue from the extraction of apple juice. Therefore, pectin is a by-product of either cider or juice production.

The largest pectin plants today are in Europe and Latin America.

### Application

Although there are various alternative processes, most pectin is produced by the extraction from the raw material with hot aqueous mineral acid. The process is known as *acid hydrolysis* and the most commonly used acid is hydrochloric acid (HCl).

The process conditions are developed by the manufacturer to suit the major type of raw material processed in the plant, and to promote the production of the desired pectin type. The acid hydrolysis results in a slurry containing a solid residue which is easily separated by filtration or centrifugation.

After a concentrating step, the concentrated extract is treated to isolate the pectin. The most commonly used method is to mix the extract with an organic solvent in which pectin is insoluble, but which will allow many of the impurities to remain in the solution. International food standards permit the use of only methanol, ethanol or isopropanol as the organic solvent. At this stage, the pectin is a firm precipitate that can be easily separated by a physical separation technology.

The isolated pectin precipitate may be washed successively with alcohol of increasing strength, finishing with pure alcohol. This results in a fibrous pectin that is dried and ground to a fine powder product.

## Instrumentation and installation



Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A measures continuously the concentration of the liquid pectin extract to ensure optimum control and the highest product quality.

The refractometer can be installed directly in the reaction vessel or at the outlet pipe to provide real-time information on the amount of extracted pectin, thus eliminating the need for time-consuming sampling and laboratory tests. The measurement by the refractometer is not influenced by suspended solids or bubbles in the slurry and is selective to the liquid phase. The continuous measurement by the refractometer helps to determine extraction efficiency

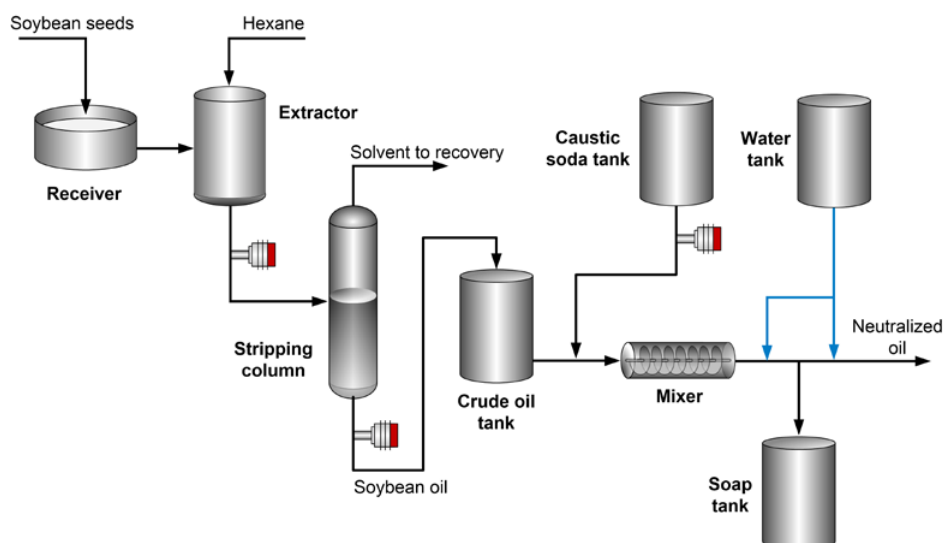
and to adjust process parameters. This ensures a liquid phase from the reactor with the highest possible pectin concentration.

A refractometer after the concentration step monitors the target concentration of the pectin extract to ensure its consistency. The refractometer's output signal can be used for automated control of the concentration step, and consequently for optimizing energy consumption and alcohol usage in the following precipitation step.

The PR-43-A refractometer complies with the highest standards for safe food and beverage processing. The refractometer is also available with 3-A and EHEDG certifications.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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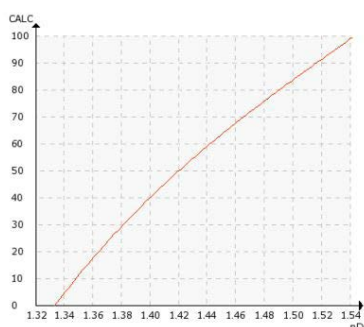


## SOYBEAN OIL, VEGETABLE OIL

### Typical end products

Soybean oil for food and biodiesel.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

Soybean seed contains about 19 % oil. Soybean oils, both liquid and partially hydrogenated, are sold as *vegetable oil* or are used in a wide variety of processed foods.

Soybean oil is also used as a feedstock for biodiesel production.

### Application

#### Solvent extraction

To extract soybean oil from the seeds, the soybeans are cracked and commercial hexane is used for solvent extraction. Hexane dissolves the oil out of the seeds. Oilseeds are usually flaked to increase the exposure to hexane. This type of extractor drips the

hexane down onto the flaked soybeans to dissolve the oil in a manner similar to a coffee percolator.

Ninety percent of the solvent remaining in the extracted oil simply evaporates and as it does, it is collected for reuse. The rest is separated with a stripping column. The oil is boiled with steam, the lighter hexane floats upward and the resultant condensate is collected.

The bottom product of the column is the crude oil, which is refined and blended for different applications, e.g. by hydrogenation.

#### Refining

The refining stage is designed to remove the phospholipids and free fatty acids from the crude oil.

Refining can be done in two stages. The first is degumming.

The second stage of refining is neutralization or caustic refining. This process removes the free fatty acids present in the crude oil. An alkali solution, usually sodium hydroxide, is added and it reacts with the free fatty acids to produce *soap*. This soap is insoluble in the oil, and is easily separated by washing with water. The alkali solution also neutralizes any acid remaining from the degumming stage.

The alkali will also react with the triglycerides in the oil, so the neutralization parameters (type of alkali, solution strength, temperature, agitation and time) must be optimized to minimize the yield loss. There may be additional losses from emulsification and oil droplets suspension in the soap solution.

A by-product of the caustic refining is a mixture of soap, water and oil known as *soapstock*. This has been considered as a low-cost feedstock for biodiesel but its high water content and conversion of the soaps to methyl esters are significant obstacles for cost-effective utilization.




## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A is used to monitor and optimize the extraction process. The refractometer measures continuously the amount of extracted oil in hexane, providing real-time information on extraction efficiency. Typically, the concentration of the mixture after extraction is 30 % oil and 70 % hexane. The process temperature is 60°C (140° F).

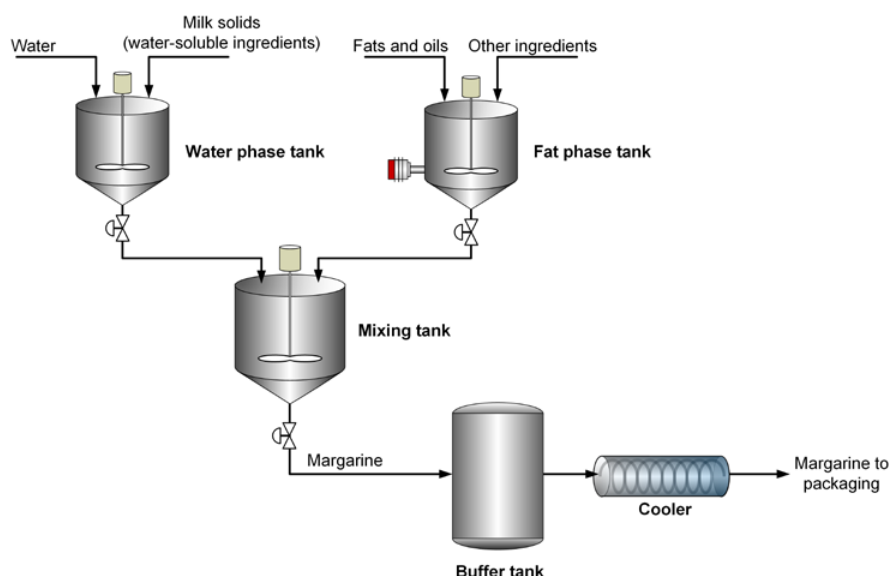
After the extraction, the solvent is recovered in the separation column. The refractive index value of the oil after solvent removal can be monitored for quality control purpose. In-line measurement by the refractometer allows for effective control of the extraction process and optimized extraction efficiency.

The concentration of caustic soda is also monitored using a refractometer so that the neutralization process proceeds efficiently.

Appropriate equipment hazardous and intrinsic safety approvals are available for hazardous area installations.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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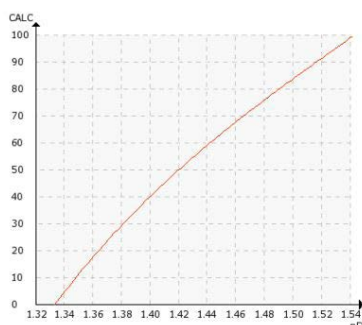


## FATS AND OILS

### Typical end products

Margarine and spreads.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Margarine is a stabilized water-in-oil emulsion which was developed as a substitute for butter. Margarine is made from one or more vegetable oils or animal fats, mixed with an aqueous portion containing milk products, salts, and other ingredients such as flavoring agents, emulsifiers, vitamins, preservatives and butter.

Margarine is used principally as a spread but is used also for flavoring and cooking.

### Application

The main components in the production of margarine are a fat phase, a water phase and minor ingredients including salt and emulsifiers.

The fat phase contains the fat-soluble ingredients. These are usually fat-soluble flavors, vitamins as well as emulsifiers and carotenes. The aqueous phase holds the water-soluble ingredients, which are generally water-soluble flavors, salt, milk or milk solids, and in special cases, preservatives.

The first stage in margarine production is usually done in batches and involves the preparation of the water and fat phase. The water phase is prepared by mixing in water milk proteins, salt, preservatives and other water-soluble ingredients. The fat phase is a blend of different fats and oils. The ratio between the fats and oils is decisive for the performance of the final product and it can be controlled, for instance, by refractive index measurement.



Next, the phases are mixed together thoroughly and pumped into a buffer tank. From there on the process is continuous. The final margarine product is cooled and then moves on to packaging.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-AP-T is installed directly in the fat phase tank through an APV Tank Bottom Flange. The refractometer measures accurately and continuously the refractive index of the blend during the preparation of the fat phase. This helps to determine the correct fat and oil proportion, and it is an important quality measurement before continuing to emulsification.

The in-line measurement by the refractometer provides real-time QA monitoring of the blend and eliminates the need for sampling and laboratory tests, thus increasing productivity and reducing batch time.

In addition, the real-time measurement by the refractometer provides instant information for ingredients dosing, and helps to produce margarine with the desired characteristics, functionalities and consistent quality.

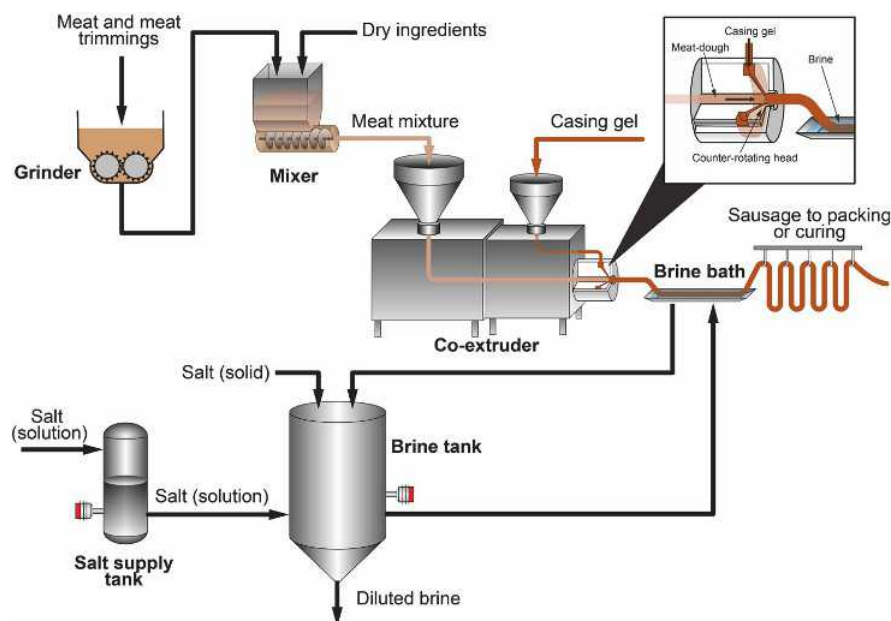
Instrumentation	Description
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Sanitary Flush Mounted Refractometer PR-43-AP-T for hygienic flush mounting installations in cookers, cooling crystallizers and other vessels that have scrapers or mixers. Installation through an APV Tank bottom flange.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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## Sausage processing: brining of co-extruded synthetic sausage casings

Food and Beverage | Fish and Meat products | 2.14.02



### Measured medium

- Brine, sodium chloride NaCl, DI potassium phosphate  $K_2HPO_4$ , calcium chloride  $CaCl_2$

### Typical end products

- Co-extruded alginate, collagen or hybrid gel sausage casings and fresh sausages, cooked sausages, dry or semidry sausages, and emulsion meat sausages such as hot dogs.

is put into contact with the brine solution for removal of water by osmosis. This is done by either passing the sausages through a brine bath or by spraying brine on them.

The brine bath is a salt solution, and as the sausage passes by, the amount of water increases and the brine becomes diluted. To ensure optimal product quality, fresh salt is added to the brine either in solid form or as a solution to keep the brine at the desired concentration level.

After the brine bath, the sausage is ready to move on for packing or curing. Curing can be done, either by smoking, cooking or drying. The concentration of the salt should be carefully monitored and controlled as the quality of the casing affects the consistency and quality.

The concentration of brine defines the color and texture of

### Introduction

Sausages are manufactured by mincing or grinding meat and mixing the ground protein with various ingredients and water. The mixture is then stuffed into a casing to give it its distinctive shape and appearance and the sausage is finally treated or cured to meet the requirements of the desired final product.

Sausage casings can be natural or synthetic. Synthetic casings made of synthetic material such as alginate, collagen or hybrid gel have better processing characteristics (better strength and uniform quality) and make it possible to produce sausages continuously in large volumes with less costs.

### Application

Sausages with synthetic casings are often produced by co-extrusion. Instead of filling a casing, this method creates the casing as the sausage is produced. The meat and the casing gel are passed simultaneously through an extruder. The meat mixture or emulsion is pumped into the inner orifice while the casing suspension is applied to the outside surface of the meat mixture.

The gel usually has a high-water content, and needs to be coagulated by dehydration with a brine solution such as sodium chloride, dipotassium phosphate or calcium chloride to increase its mechanical properties. Therefore, after the co-extruder, the sausage

the final sausage. Too low brine concentration results in a less firm and opaque product, while too high concentration causes the product to over dry dry leaving the sausage with a rubbery texture.

## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-A monitors and controls continuously the concentration of salt in the brine, such as sodium chloride, dipotassium phosphate or Calcium chloride, for a higher consistency and quality of the final product. This provides real-time information to the operators on when the



addition of fresh salt is required to maintain the optimal brine concentration.

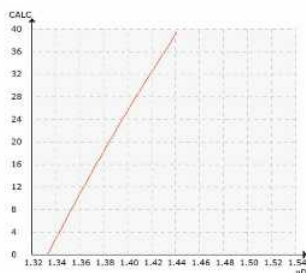
The refractometer is installed directly in the brine tank to monitor continuously the concentration of the brine as water gets transferred from the casing to the solution. A refractometer can also be installed in the liquid salt supply tank to ensure the right concentration and dosing.

The refractometer provides Ethernet or 4-20 mA output signal that can be used for real-time process control. The refractometer in the brine tank, sends measurement data to a controller connected to the salt

feed valve and discharge valve. If the concentration falls below the target level, the controller opens the feed valve and adds salt solution in the brine tank until the concentration of the brine is set back to the optimal level. As the tank volume is limited, the controller also opens the discharge valve to discharge part of the diluted brine.

Our refractive index technology is accurate and reliable for in-line concentration measurements. The Sanitary refractometer PR-43-AC is available with Sanitary 3-A and EHEDG certifications to meet the highest hygiene requirements of food production.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.
Chemical Curve	Salt R.I. per Conc % b.w. at Ref. T of 20 °C



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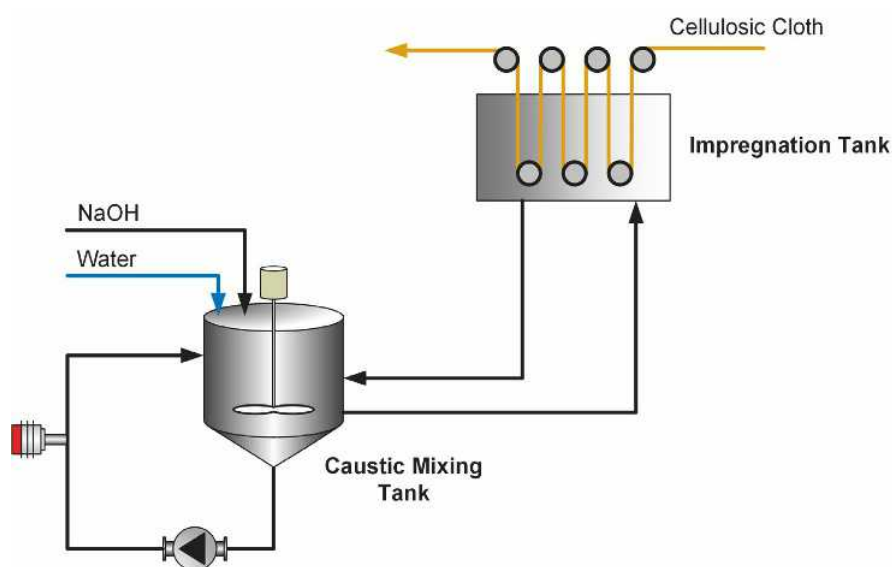
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## Sausage processing: preparation of synthetic sausage skins

Food and Beverage | Fish and Meat Products | 2.14.02



### Measured medium

- Caustic soda, sodium hydroxide, NaOH

### Typical end products

- Synthetic sausage casings made from cellulosic fibers.

### Introduction

Cellulose is widely used in the production of synthetic sausage casings. The most common method for processing cellulose into sausage skins is to mix the cellulosic material (e.g. wood) with an alkaline solution to produce a dissolved cellulosic feedstock. This dope is further processed to regenerate the cellulose and extrude it in a tubular form. The tubular skins are then dried and rolled or coiled for easier filling with the sausage mix by automated machinery.

The cloth is desulfurized by passing it through a Sodium Hydroxide (NaOH) bath by means of conveying rollers. The caustic solution is supplied from a tank, where it is prepared by mixing water and a concentrated solution of NaOH (above 50 %). A typical concentration of the caustic solution is 200-250 g/L.

The excess of NaOH returns to the mixing tank, where the concentration is replenished because caustic is lost in the cloth impregnation process. The cloth moves on for further processing.

and real-time information provided by the refractometer helps to maintain the target concentration of the solution and eliminates the need for manual sampling and testing.



The refractometer can operate as a stand-alone device or with a user interface selected by the user from a range of options. The refractometer gives mA and Ethernet output signals that allow automatic operation of the process. Moreover, the refractometers can be calibrated to read the concentration of NaOH in g/L, wt-% or any other engineering unit preferred by the factory. Automatic prism wash may be needed in this application.

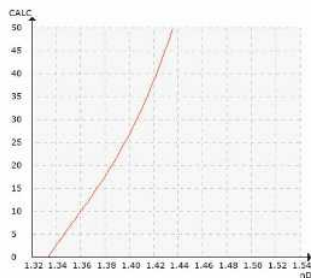
### Application

A cloth made of cellulose fibers is produced for creating the sausage casing. This cloth may be treated with different baths for the precipitation of the viscose or further purification.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP monitors continuously the sodium hydroxide content in the caustic solution preparation stage. The continuous

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.
Chemical curve	NaOH R.I. per Conc% b.w. at Ref. Temp. of 20°C



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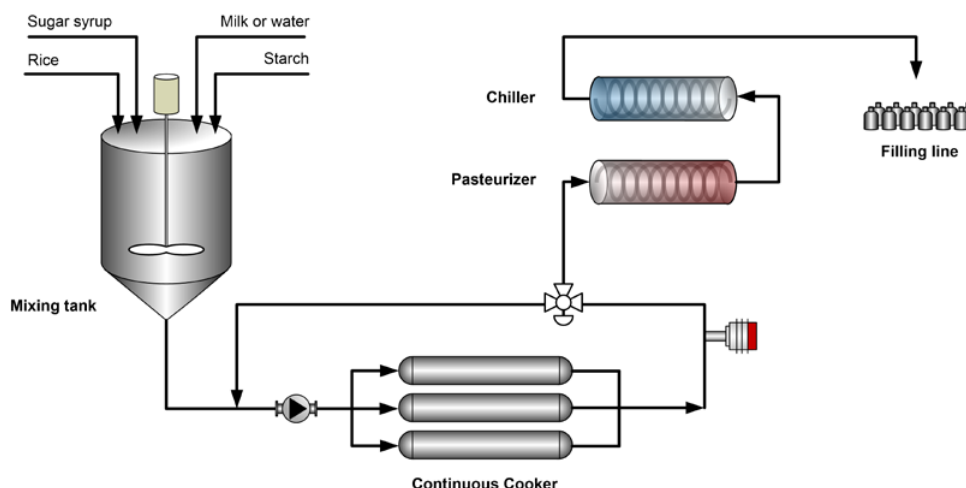
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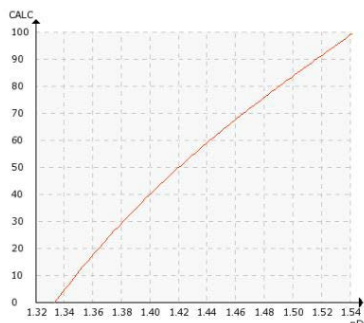


## PUDDING SLURRY

### Typical end products

Rice pudding and other dairy desserts.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



## Introduction

The production of dairy desserts, for example rice pudding, has high product quality and taste requirements. In order to ensure end product homogeneity and consistency, it is of utmost importance to continuously control the quality of the pudding slurry.

## Application

Rice pudding is prepared from rice mixed with hot water or milk and sugar, and possibly other ingredients depending on the recipe, such as cinnamon and raisins.

Rice pudding in hot water or milk is prepared by blending pre-cooked rice with a sugar syrup to form a first coating on the rice grains. The sugar-coated rice

is then blended with an aqueous starch slurry to form a second coating on the rice grains. The amount of water used to prepare the coating is limited so that no subsequent drying step is necessary.

The sugar syrup for the first coating on the rice is prepared by dry blending sugar, salt, flavors and colors, and then adding a limited quantity of water. The mixture is then heated for a sufficient period of time at a temperature high enough to dissolve all of the water-soluble ingredients. The amount of water added is the minimum required to create a solution at elevated temperatures. This means that when the sugar syrup is cooled it will solidify to a dry mass.

The starch slurry is prepared by mixing starch and sufficient (but limited) amount of water to form a uniform dispersion.

Sugar syrup is then blended with a predetermined quantity of pre-cooked rice to evenly coat the rice with the syrup. The resulting mixture is blended with the starch slurry and mixed until the rice is dry and there are no clumps.

The mixed mass is pumped with a rotary lobe pump to a continuous cooker to achieve integrity of the grains. To ensure a desirable product, the solids content recommended to be 25-40 %.

At this stage product bacteriological safety is ensured through pasteurization. Pasteurization is also the reason for the dairy products' extended shelf life.

Then the pudding slurry is cooled and filled into single use packages.



## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-A is installed in the pipe after the cooking stage. The refractometer measures the total dissolved solids of the liquid phase before the pudding slurry moves on to further treatment. If the pudding slurry complies with the recipe's texture, it continues to the pasteurization

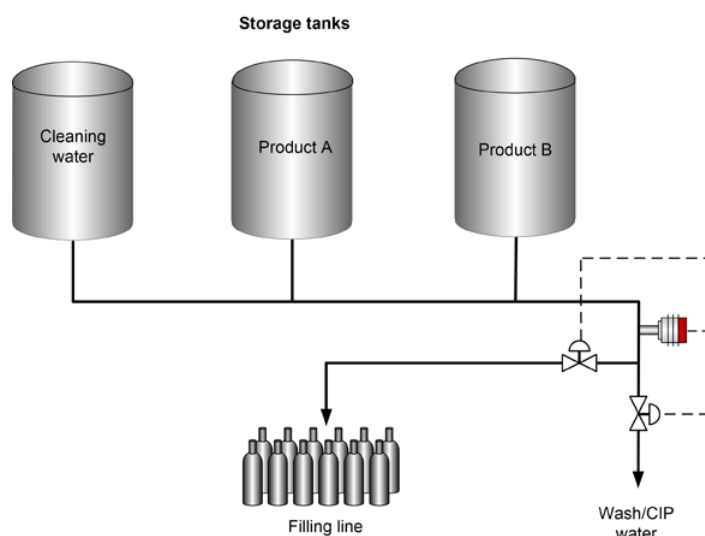
stage. If the product is out-of-specification, it is returned to the mixing stage.

The PR-43-A is designed to meet the highest hygiene requirements for aseptic dairy production. The refractometer is available with 3-A Sanitary and EHEDG certifications.

The refractometer also provides Ethernet and 4-20 mA output signals for real-time process control. Moreover, The refractometer measurement is not influenced by the solid components such as rice grains in the slurry.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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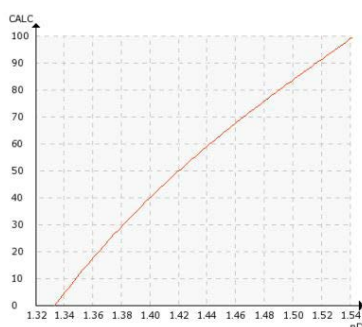


## PRODUCT AND CIP LIQUID INTERFACE

### Typical end products

Different liquid food and beverages such as egg, ice-cream, beer, juice, soft drinks, flavored water, wine, etc.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Many processing plants use the same filling station for a range of different products. For example, breweries run beer and soft drinks in the same station. The operation of high-speed in-line filling machines can be improved by utilizing real-time Refractive Index measurement technology.

Automated monitoring and control of the Clean-In-Place (CIP) process allows products to be switched, without the need for a shutdown. Therefore, productivity can be increased without compromising the end product.

### Application

CIP is performed to remove traces of products in the filling line, and for food safety and quality assurance.

After a first product is run through the pipeline to packaging, the pipes are flushed with CIP cleaning chemicals and water. When CIP is completed, the filling line is ready to pass a fresh product.

In order to save valuable production time, the second product is pumped through the pipeline right after the wash cycle. This can only be done if the interface between product-to-CIP liquid and product to-product is instantly detected.

### Instrumentation and installation

Vaisala K-PATENTS® Sanitary Refractometer PR-43-AC is installed at the end of the filling line to monitor concentration level of the medium. When the concentration reaches a pre-set limit and there is no water present, the refractometer 4-20 mA or Ethernet output signal activates the end-product filling with no delay.

When there are separate lines for the product and for the CIP cleaning media, the water flows to a sewer while the pipe is full of product. The refractometer gives an instant alarm when the concentration reaches its top limit. This signal can be used to switch valve direction. During filling, the valve is open to the filling line and closed to the sewer. During CIP cleaning and at the initiation of the fresh product release, the valve closes the filling line and the stream is then diverted to the sewer.

The refractometer instantly detects the product-to-product and product-to-CIP cleaning interfaces. The refractometer's output signal can also be utilized for quality control monitoring. This ensures a correct

product and container combinations, and that the end product complies with the specifications.


## High accuracy -HAC version for low concentration beverages

The high accuracy version Sanitary Process Refractometer PR-43-A-HAC is capable of measuring low concentration beverages, diet soft drinks and flavored waters directly and on a continuous basis, rather than depending on periodic samples or indirect methods, such as density measurement. This refractometer version is intended to be used in

applications where the accuracy and repeatability requirement are high, but the process variations are low and operating range limits of 0–30 Brix and 4–30°C (40–85 °F).

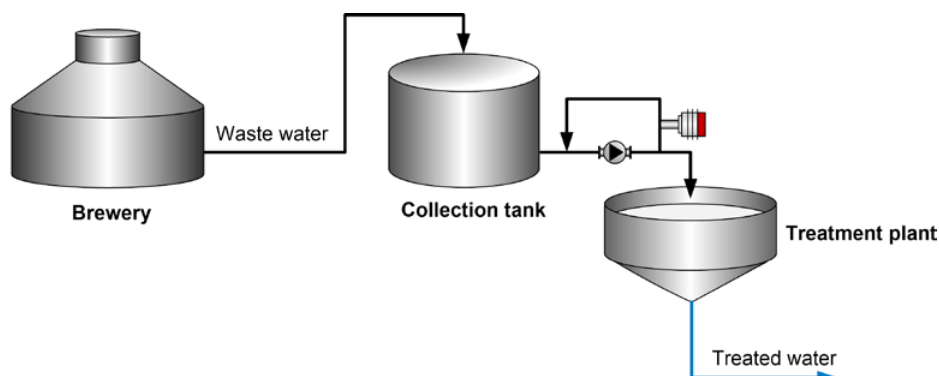
The PR-43-AC is available with 3-A sanitary and EHEDG certification

The refractometer monitoring of product Brix allows for instantaneous and real-time filling station quality control.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0–100 Brix.

Ref. B212104EN-A © Vaisala 2020



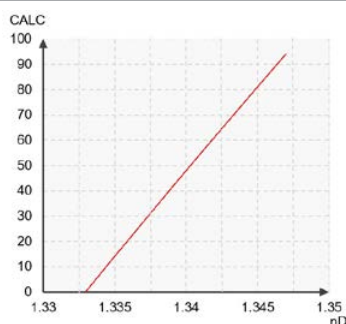


## WATER, DISSOLVED ORGANIC MATERIAL

### Typical end products

Alcohols, proteins, sugars, fats, beer and other food and beverage products.

Chemical curve: R.I. per g/L COD at Ref. Temp. of 20° C



### Introduction

The Food, Beverage and Brewing industries use high quantities of water and generate high-organic strength wastewater. Organic pollutants are problematic to the environment as their decomposition process consumes the oxygen contained in the receiving water. The oxygen in the water can be consumed at a greater rate than it can be replenished, causing oxygen depletion and severe consequences to the stream ecology.

Organic pollutants from the food and beverage industry include oils and fats, alcohol, proteins and carbohydrates.

Wastewater generated from the production needs to be monitored to ensure compliance with environmental regulations. Major pollutants are often defined by Chemical Oxygen Demand (COD) or Total Organic Carbon (TOC). Organics monitoring helps food and beverage facilities to detect quickly and accurately the carbon content in their wastewater streams. Excess organics can incur fines and penalties. In severe cases the plant can even be closed.

### Application

Food and beverage facilities, e.g. breweries, progressively try to minimize their environmental load by monitoring and treating their effluent before discharge. TOC and COD are the most common parameters used for water quality.

It is difficult to achieve an efficient operation of the wastewater plant because of the constant changes in the influent properties. To avoid these load shocks to the treatment plant, the effluents from the process are commonly stored in collection tanks.

At that point, the water is analyzed and treated according to its organic content. For example, water with a low COD content is sent to the treatment plant, while the water with a high COD is treated in a reactor first.

Traditional COD and TOC testing methods are off-line and time consuming, rendering them unsuitable for real-time control. While TOC can be measured on-line by conductivity and non-dispersive infrared (NDIR) based methods, these analyzers are expensive to maintain and calibrate, resulting in frequent breakdowns in their operation.

Food and beverage facilities require a reliable and in-line measurement of organics content in the effluents. Real-time control and monitoring of the effluent provide the wastewater treatment plant with instant warning of exceptional loads and help to optimize its operation.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 provides reliable and continuous measurement to estimate the pollutant content in the water from the production facilities. The refractometer's measurement is accurate and requires no recalibration.

The refractometer measures the refractive index of the effluents providing instant information on the amount of dissolved solids. The refractive index technique has proven to be a successful method for effluent monitoring as it has good correlation with COD and TOC values.



Typical measurement range is 0-10000 ppm (COD or TOC). The refractometer provides Ethernet and 4-20 mA output signals that can be connected to the process controller for real-time control. For instance, alarms can be set at 6500 ppm and the pump shut-off at 7500 ppm. The alarms are a reliable method for indicating a large amount of dissolved solids (high organics) to the treatment plant.

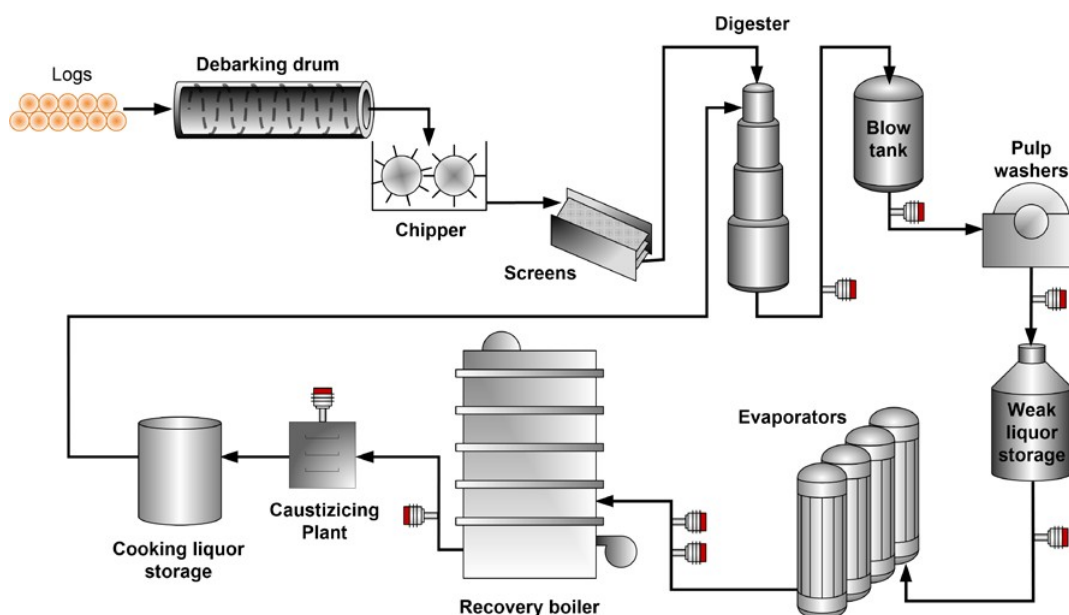
Similarly, the refractometer can be installed in the discharge lines within the production facilities. Water of suitable quality can be recycled to reduce costs

and the demand of water supply. Moreover, the integration of continuous water quality monitoring in the production areas provides real-time alarm of product losses allowing quick corrective actions.

The refractometer is installed directly at the dispersing pipe's outlet of the effluent sewer. The refractometer can read 0-1 % total dissolved solids that correlates with 0-10000 ppm TOC scale.

With the refractometer's reliable and real-time measurement, the wastewater plant can adjust their operation for the incoming load, reduce the treatment cost, and increase their efficiency to meet the environmental regulations.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

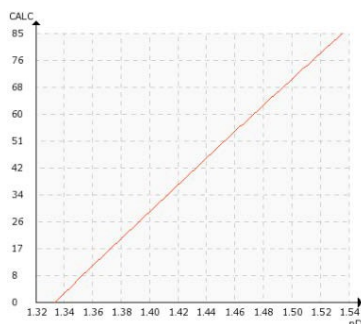


## KRAFT BLACK LIQUOR, GREEN LIQUOR, BROWN STOCK

### Typical end products

Wood pulp, paper and board.

Chemical curve: Black liquor R.I. per Conc% by weight at Ref. Temp. of 20°C



### Introduction

Wood pulp for paper and board manufacturing can be produced with several different methods. These are chemical pulp, semi-chemical or chemi-mechanical (NSSC and CTMP), and thermo-mechanical pulp (TMP). Refractometer applications are in the chemical and semi-chemical pulp production processes.

The Kraft process converts wood into wood pulp, which consists of almost pure cellulose fibers. The process entails treating wood chips in alkaline cooking conditions, which break the bonds that link lignin to cellulose.

### Application

#### Pulping process

Wood chips are fed into vessels called *digesters* that withstand high pressures. Some digesters operate in a batch manner and some in a continuous process, such as the Kamyr digester.

Wood chips are impregnated with cooking liquors. The cooking liquors consist of warm *black liquor* and *white liquor*. The warm black liquor is the spent cooking liquor from the blow tank. White liquor is a mixture of sodium hydroxide and sodium sulfide, which are produced in the recovery process.

In a continuous digester the materials are fed at a rate, which allows the pulping reaction to be completed by the time the materials exit the reactor. Typically, delignification requires several hours at a temperature of 130-180 °C (266-356 °F). Under these conditions lignin and some hemicellulose degrade, creating fragments, which are soluble in the basic liquid.

The solid pulp (about 50 % by weight based on the dry wood chips) is collected and washed. At this point, the pulp is in a suspension form of deep brown color and is known as *brown stock*. The combined liquids, known as *black liquor*, contain lignin fragments, carbohydrates from the breakdown of hemicellulose, sodium carbonate, sodium sulfate and other inorganic salts.

## Fiber line and brown stock washing (BSW)

After cooking, the sulfate pulp passes through the blow line to the blow tank and then on to the washing stage.

During brown stock washing, the spent cooking liquors are separated from the cellulose fibers. Normally a pulp mill has 3-5 washing stages in a series. The pulp passes through further washing stages following oxygen delignification and after each bleaching stage. Several processes are involved: thickening/dilution, displacement and diffusion. The dilution factor is a measure of the actual amount of water used in the washing, compared with the theoretical amount required to displace the liquor from the thickened pulp. A lower dilution factor reduces energy consumption, while a higher dilution factor normally gives cleaner pulp. Thorough washing reduces the organic material in the pulp suspension, and consequently, the Chemical Oxygen Demand (COD).

In a modern mill, *brown stock* (cellulose fibers containing approximately 5 % residual lignin), produced by the pulping, is first washed to remove some of the dissolved organic material and then further delignified through the bleaching stages.

## Recovery process

The excess black liquor comprises about 15 % solids and is concentrated in a multiple-effect evaporator. After the first effect, the black liquor's concentration is raised to 20-30 % solids.

The weak black liquor is further evaporated to 60 % or even 80 % solids (*heavy black liquor*). This is then burned in the recovery boiler to recover the

inorganic chemicals for reuse in the pulping process. The high solid content in the concentrated black liquor increases the energy and chemical efficiency of the recovery cycle. However, a higher viscosity may cause precipitation of solids, and may lead to plugging and fouling of the equipment.



The molten salts (*smelt*) from the recovery boiler are dissolved in process water, known as *weak wash* or *weak white liquor*. This process water is composed of all liquors used to wash lime mud and green liquor precipitates. It is kept in a weak wash storage tank. The resulting solution of sodium carbonate and sodium sulfide is known as *green liquor*. This liquid is mixed with calcium hydroxide to produce the white liquor used in the pulping process.

The recovery boiler also generates high pressure steam, which passes through steam turbine generators to produce electricity and to reduce the steam pressure for mill use. Therefore, a modern Kraft mill is self-sufficient for its electrical energy supply.

## Instrumentation and installation

Vaisala K-PATENTS® SAFE-DRIVE Process Refractometer PR-23-SD is used to measure in real-time the concentration of the liquors at various stages of the pulping process. The refractometer is mounted directly in the pipeline for continuous measurement of dry solids content of the liquors and the pulp suspension, thus reducing the need for sampling and laborious laboratory tests.

The SAFE-DRIVE refractometer helps to optimize the pulping process to obtain a high-quality pulp, reduce energy consumption and environmental load and improve the economics of the mill.

Instrumentation	Description
	SAFE-DRIVE Process Refractometer PR-23-SD for measuring black liquor dry solids and green liquor density or TTA in kraft chemical recovery process. SAFE-DRIVE design allows for safe and easy insertion and retraction of the sensor under full operating pressure without having to shut down the process.
	Digital Divert Control System DD-23 for safe operation of kraft chemical recovery boiler. The DD-23 system complies strictly with all recommendations of BLRBAC. The DD-23 system includes two SAFE-DRIVE Refractometer PR-23-SD sensors in the main black liquor line, and two Indicating transmitters and a Divert control unit in an integrated panel. Remote monitoring and event data logging via Ethernet.

Measurement range	<p>Refractive Index (nD) 1.3200 - 1.5300 (-73 prism) or Refractive Index (nD) 1.3600 - 1.5700 (-72 prism)</p> <p>The typical -73 prism suits the full measurement range for majority of measurement points in the process. Prism -72 is needed for measuring over 80 % solids in acacia and eucalyptus hardwood based black liquor.</p>
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Ref. B211970EN-B © Vaisala 2021

## Recommended refractometer prism option for high solids black liquor applications



### Introduction

In the kraft pulp recovery process, the by-product black liquor is concentrated in evaporators and burned in a recovery boiler to recover the inorganic chemicals for reuse in the pulping process, while producing energy for the pulp mill's use. The weak black liquor is evaporated to 60 % or even up to 90 % dry solids, also known as heavy black liquor.

A higher dry solids content in the black liquor increases the energy and chemical efficiency of the recovery cycle. However, high dry solids may also lead to plugging and fouling of the equipment due to high viscosity.

typical -73 prism with refractive index (nD) range 1.33-1.53, suits majority of applications in the pulp mills. However, black liquor from hardwood pulp has slightly higher refractive index than black liquor from softwood.

### Black liquor and prism measurement range

The Vaisala K-PATENTS® Process Refractometer PR-23-SD with

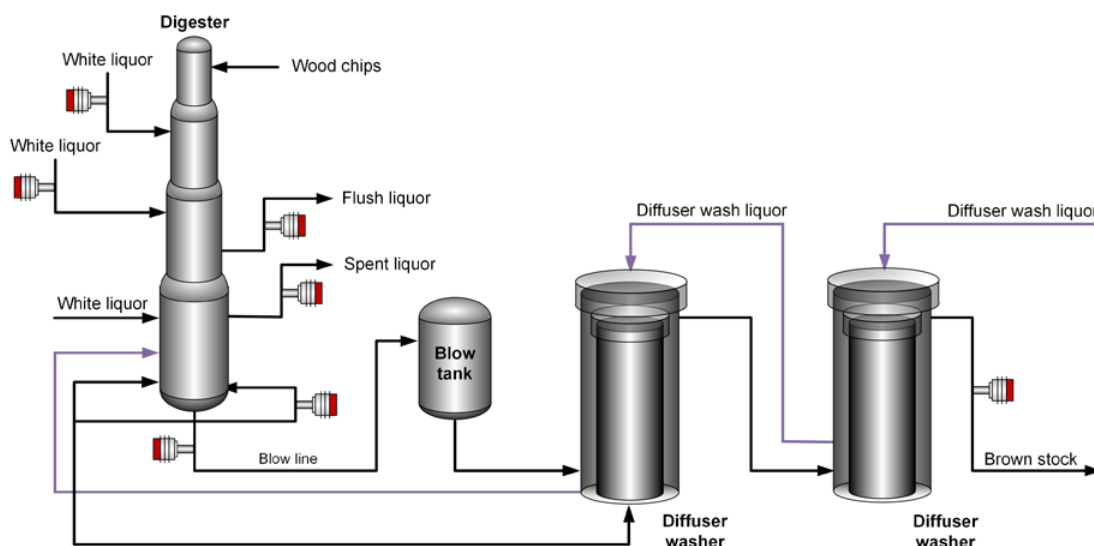
We have identified that in countries with acacia and eucalyptus hardwood pulp, the black liquor refractive index may exceed the maximum range of the -73 prism.

### Vaisala recommendation

Vaisala recommends using PR-23-SD model with the -72-prism option (nD range 1.36-1.57) for high solids, above 80 % dry solids black liquor applications in countries with acacia and eucalyptus hardwood. Such high solids measurement points are after high dry solids or super concentration, and in recovery boiler.

It should be noted that the refractometers with the -72-prism option do not suit low dry solids applications such as Brown Stock Washing.



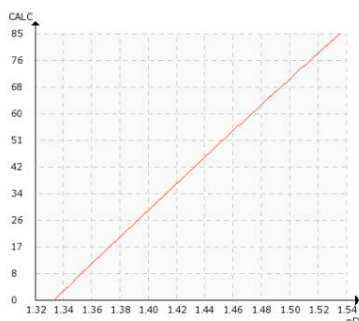


## KRAFT PULP, BLACK LIQUOR, WHITE LIQUOR, BROWN STOCK

### Typical end products

Unbleached Kraft pulp, bleached Kraft pulp.

Chemical curve: R.I. per black liquor conc% at ref. temp. of 20°C



### Introduction

The first operation in the Kraft pulping process involves the extraction of cellulose from wood by dissolving the lignin that binds the fibers together. This is done in a strongly alkaline solution.

This process is known as *cooking*. After the wood pulp is obtained, it is washed and bleached to obtain the fibrous product.

To optimize the pulp chemical consumption and water usage, the black liquor concentrations have to be measured before and after washing.

### Application

Incoming wood is debarked and chipped to an optimal size to minimize fibre damage, and to maximize the impregnation with the cooking liquor. The chips and the cooking liquor are fed into a large vessel known as *digester*. The pulping reaction takes place under pressure and at a high temperature.

After cooking, the pulp passes through a blow line to the blow tank and then to a washing section. The diffuser washers separate the black liquor from the fibers by washing them with a washing liquor or water. The products from the fiber line are a clean pulp, and a diluted black liquor known as *weak liquor*.

The washed pulp is then screened before it is sent to the bleaching plant, and the weak black liquor passes from the washing section to the chemical recovery process.

### Instrumentation and installation

Vaisala K-PATENTS® SAFE-DRIVE Refractometer PR-23-SD measures in real-time the Total Dissolved Solids (TDS) content in black liquor.


The refractometer is installed in-line in different points after the digester. TDS measurement in the blow pulp suspension after the digester enables monitoring of the diffuser operation. Together with other measurements (e.g. filtrate and flush liquor) this provides the mill with the ability to control the performance of the washing zone in the digester. In addition, TDS measurement



in the blow line allows the performance of the digester to be monitored, ensuring that it yields the correct concentration. The combination of these measurements facilitates continuous calculation of the mass balances of the digester.

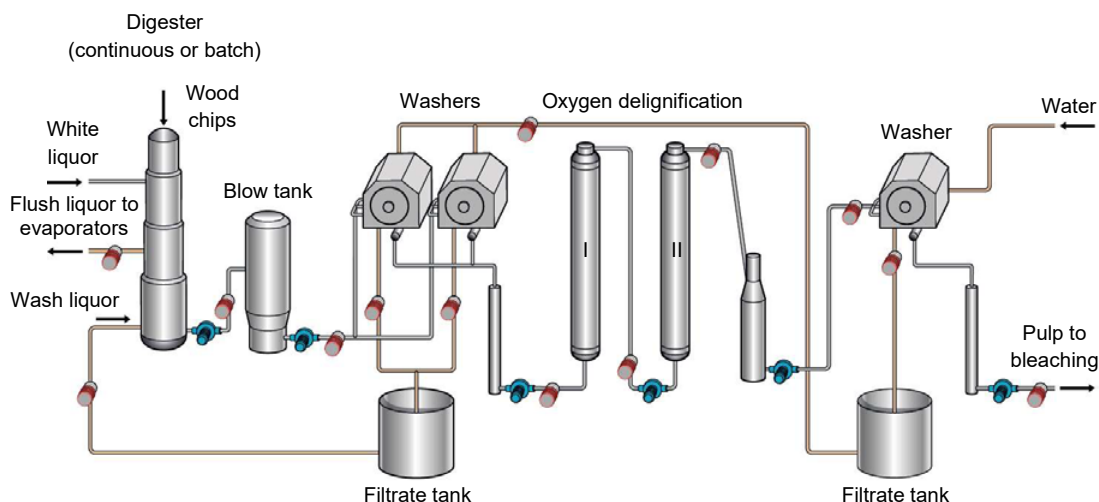
The refractometer's measurement is unaffected by bubbles, particles, consistency, flow, ion changes, pH, temperature, pressure, color or turbulent flow. The measurement surface is periodically cleaned using an integrated and automatic prism cleaning system.

Black liquor concentration measurement with the SAFE-DRIVE process refractometer helps to increase washing efficiency, obtain a consistent pulp quality, reduce bleaching chemical consumption and environmental load, and increase evaporation efficiency.

Instrumentation	Description
	SAFE-DRIVE Process Refractometer PR-23-SD for measuring black liquor dry solids and green liquor density or TTA in kraft chemical recovery process. K-Patents SAFE-DRIVE design allows for safe and easy insertion and retraction of the sensor under full operating pressure without having to shut down the process.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

Ref. B211971EN-A © Vaisala 2020



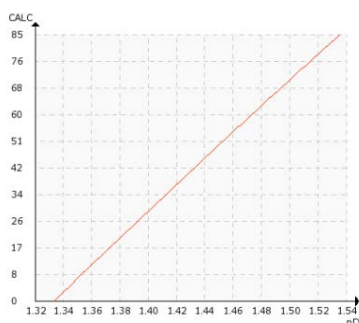


## WEAK BLACK LIQUOR, BROWN STOCK WASHING FILTRATES

### Typical end products

Unbleached Kraft pulp, bleached Kraft pulp.

Chemical curve: R.I. per black liquor conc% at ref. temp. of 20 °C



### Introduction

Brown stock washing (BSW) can be considered the key operation influencing the economics of the pulping process, as well as the environmental load of the mill. The purpose of brown stock washing is to remove both organic and inorganic soluble compounds from the pulp suspension (brown stock), while using the lowest possible amount of wash liquor or water.

Through optimization of the brown stock washing process and raising the black liquor solids content, mills gain immediate profits, cleaner and better quality pulp for bleaching, as well as optimized use of water, chemicals and energy.

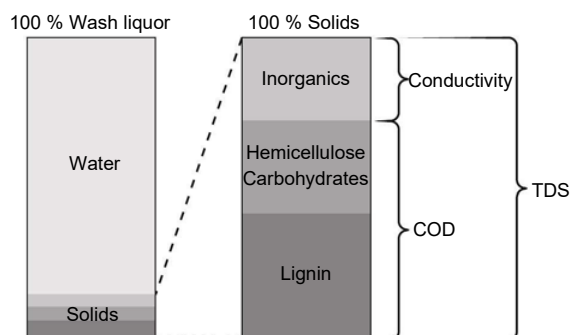
The performance of the washing process has traditionally been controlled in two main ways: using *wash loss* and *dilution factor* (DF). Wash loss refers to the quantity of washable compounds in the pulp suspension that could have been removed by washing.

The DF indicates the amount of water per ton of pulp added during washing, and which ultimately dilutes the black liquor.

Operators have found the washing process difficult to manage efficiently due to a lack of robust instruments that reliably measure wash loss in-line. For instance, conductivity is widely used for this purpose, even though it is based on the measurement of the ionic sodium species in the liquor (inorganic phase) and does not directly measure the organic phase, especially lignin and hemicellulose.

Other conventional methods also fail to measure the wash loss satisfactorily. Means of monitoring these variables, such as Chemical Oxygen Demand (COD) tests performed on the filtrate, only indicate organic compounds. Furthermore, off-line laboratory methods, such as standard dry solids analysis or COD analysis, have considerable time requirements, rendering them unsuitable for advance process control.

Total Dissolved Solids (TDS) has proven to be a reliable parameter for measuring washing efficiency, because it takes account of both the inorganic and organic fractions, thus all washable solids (real wash loss) are quantified (Figure 1). TDS can be measured in-line with a process refractometer that provides real-time wash performance information, while enabling a quick response to potential changes or disturbances in the process. The refractometer's output can also be calibrated to read COD.



**Figure 1.** Comparison of measurement methods for dissolved solids in wash liquor.

## Application

When examining the overall economic relationship between operating cost and efficiency, the first considerations are maximizing the solids yield due to its heat value in the recovery boiler and minimizing the dilution factor to save steam in the evaporators. The next key factors are the cost of make-up chemicals for replacing sodium losses and the cost of effluent treatment. Other important considerations include evaporator efficiency limitations in many mills and bleach chemical consumption in bleachable grade mills.

The development of Vaisala K-PATENTS® SAFE-DRIVE Process Refractometer PR-23-SD for the continuous measurement of dissolved solids content in a fiber suspension has made it possible to follow the performance of individual process steps continuously. TDS changes in the feed and outlet stock lines, as well as in incoming and outgoing filtrate lines, can be detected immediately.

Once a mill has the appropriate number of in-line total dissolved solids measurements, it can calculate and optimize its brown stock washing variables, such as the optimum Dilution Factor (DF), Displacement Ratio (DR), relative washing loss (1-Y), and the entire plant Efficiency Factor (E). This enables the implementation of upper level control in order to achieve the benefits of the full optimization of brown stock washing operations.

### Digester washing zone and blow line

TDS measurement in the blow pulp suspension after the digester enables monitoring of the diffuser operation. Together with other measurements (e.g. filtrate and flush liquor) this provides the mill with the ability to control the performance of the washing zone in the digester. In addition, TDS measurement in the blow line allows the performance of the digester

to be monitored, ensuring that it yields the correct concentration.

We recommend three TDS measurement points around a continuous digester: wash liquor feed to the washing zone, flush liquor outlet and blow line. The combination of these measurements facilitates continuous calculation of the mass balances and the creation of operating models of the digester.

In a batch digester, three measurement points are recommended: weak liquor inlet, liquor circulation flow and pulp out. Monitoring TDS in the liquor circulation flow of the batch digester helps to determine when the batch is ready.

### Pulp feed to washers

Accurate and real-time TDS measurement within the feed pulp allows a quick response to process changes and prevents disturbances from being carried over into subsequent washing stages. Process variables, such as the dilution factor, can be controlled in accordance with the properties of the inlet pulp, and mass balances can be continuously monitored.

### Washing stage

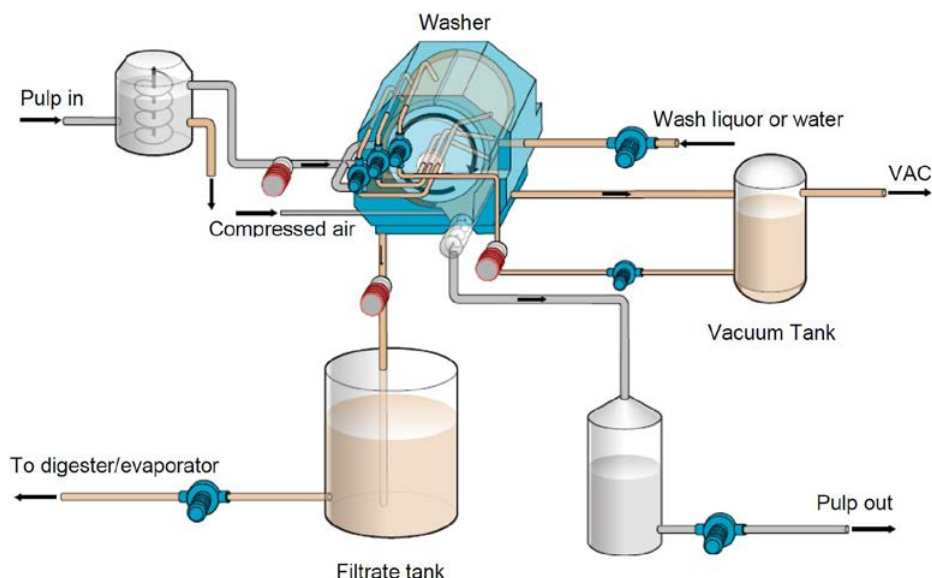
At this stage, in-line TDS measurements provide better control and help to determine the optimum dilution factor, right operation consistency and optimum concentration of solids in the stream to the recovery boiler. This prevents excess consumption of water in the washers, thus reducing the energy requirements of the evaporator and the need for make-up chemicals. Important TDS measurement points in all types of washers are the pulp inlet, pulp outlet, wash liquid feed and wash filtrate (Figure 2).

### Pulp to oxygen delignification (OD)

The cost and performance of the OD process can be optimized using in-line TDS measurements. Wash loss reduction in the inlet pulp to the reactors decreases the amount of alkali. Alkali is consumed in neutralization reactions of the organic acids. In addition, the temperature of the reactor can be optimized and the amount of oxygen decreased.

### Pulp discharge to bleaching

Reliable continuous measurements of wash loss in the discharge pulp make it possible to control the success of the washing process. By minimizing the wash loss, the subsequent process steps can be optimized. Lower wash loss in the outlet pulp will improve its quality while also decreasing the amount of chemicals required in the bleaching stage. Reliable



**Figure 2.** DD-washer control with in-line TDS measurement.

measurements improve environmental performance and can reduce effluent treatment costs.


## Instrumentation and installation

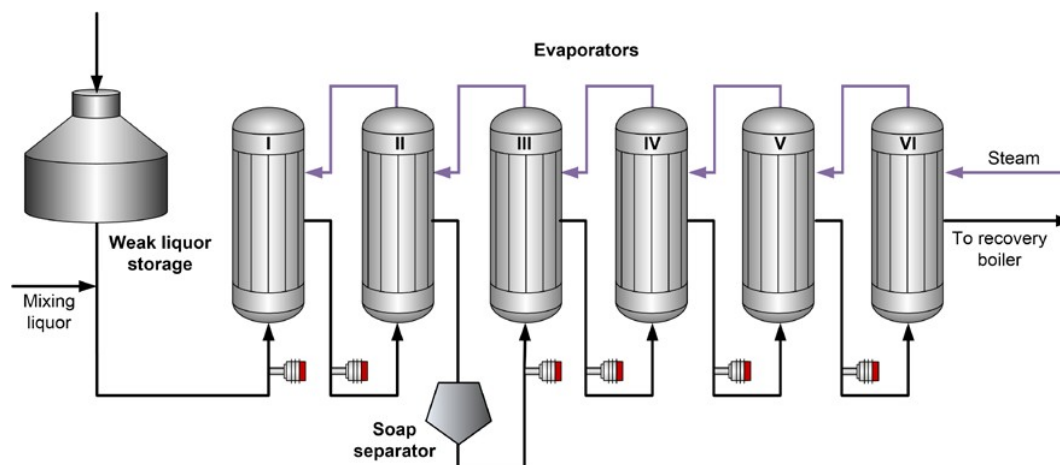
Vaisala K-PATENTS® SAFE-DRIVE Process Refractometer PR-23-SD is installed at different points in the fiber line for real-time BSW control and optimization. There are two types of SAFE-DRIVE Process Refractometer systems specifically designed for reliability and easy installation directly in the process pipe line:

1. SAFE-DRIVE Process Refractometer PR-23-SD with Isolation valve SDI2-23-SN2-XS and steam wash for installations in the wash filtrates and weak black liquor.

2. SAFE-DRIVE Process Refractometer PR-23-SD with Isolation valve SD12-23-PL-SS and pulp line installation plate without wash for installations in the blow line and pulp stock line.

We can also provide a complete consultation service that begins with a pre-study and evaluation of the existing process, including potential bottlenecks. The service also offers recommendations on implementing total dissolved solids measurements for individual washing stages, and it provides guidance on connecting the technology to the existing process control system. The ultimate purpose of in-line measurements is to reveal the optimum changes required to achieve the greatest efficiency at each stage of the process.

Instrumentation	Description
	SAFE-DRIVE Process Refractometer PR-23-SD for measuring black liquor dry solids and green liquor density or TTA in kraft chemical recovery process. SAFE-DRIVE design allows for safe and easy insertion and retraction of the sensor under full operating pressure without having to shut down the process.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash with steam, only for installations in filtrates or weak black liquor lines. The components of a steam wash system are a sensor with integral steam nozzle mounted at the SAFE-DRIVE valve, a shut-off valve for steam line and an indicating transmitter equipped with relays to drive the wash valves.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

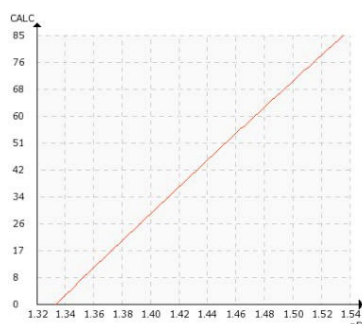


## BLACK LIQUOR

### Typical end products

Wood pulp, paper, board.

Chemical curve: R.I. per black liquor conc% at ref. temp. of 20 °C



## Introduction

A by-product from the Kraft process is *black liquor*. It is a combination of removed lignin, water and chemicals used in the extraction process. The black liquor, which originates from the washing process, is concentrated through evaporation and then used to fire a boiler in order to generate high-pressure steam for the mill's operations.

When softwood is used in pulping (e.g. conifers), a soap-like substance is collected after the process. This soap is then acidified and used to produce *tall oil*. Tall oil is a source of fatty acids, resin acids and various other chemicals.

## Application

The feed to the evaporation stage should be as consistent as possible to maximize the capacity and stabilize the output concentration. Feed concentration

is kept constant by regulating the mixing liquor. This is possible using Vaisala K-PATENTS®SAFE DRIVE Process Refractometer PR-23-SD for concentration measurement.


The concentration after each evaporation step can be controlled by regulating the steam flow through the evaporators. This control is achieved with the continuous measurement by the refractometer. The final concentration can also be measured. Typical operating temperature range in this application is 120-130 °C (248-266 °F).

It is recommended to measure the liquor concentrations at intermediate evaporation stages to minimize total steam consumption. This is particularly important in mills where the cost of steam is high.

## Instrumentation and installation

Typical concentration at the evaporation feed is 10 to 30 % dry solids and after the concentrator phase 60 to 80 % dry solids.

Automatic prism wash with steam is recommended for evaporation installations. When using high dry solids or super concentrator, the refractive index of above 80 % dry solids in acacia and eucalyptus hardwood based black liquor may exceed 1.5300, and therefore the prism -72 is recommended.

Instrumentation	Description
	SAFE-DRIVE Process Refractometer PR-23-SD for measuring black liquor dry solids and green liquor density or TTA in kraft chemical recovery process. SAFE-DRIVE design allows for safe and easy insertion and retraction of the sensor under full operating pressure without having to shut down the process.
<b>Automatic prism wash</b>	Prism wash with steam. The components of a steam wash system are a sensor with integral steam nozzle mounted at the SAFE-DRIVE valve, a shut-off valve for steam line and an indicating transmitter equipped with relays to drive the wash valves.
<b>Measurement range</b>	<p>Refractive Index (nD) 1.3200 - 1.5300 (-73 prism) or Refractive Index (nD) 1.3600 - 1.5700 (-72 prism)</p> <p>The typical -73 prism suits the full measurement range for majority of measurement points in the process. Prism -72 is used for measuring solids over 80 % in acacia and eucalyptus hardwood based black liquor.</p>

Ref. B211973EN-B © Vaisala 2021

## Recommended refractometer prism option for high solids black liquor applications



### Introduction

In the kraft pulp recovery process, the by-product black liquor is concentrated in evaporators and burned in a recovery boiler to recover the inorganic chemicals for reuse in the pulping process, while producing energy for the pulp mill's use. The weak black liquor is evaporated to 60 % or even up to 90 % dry solids, also known as heavy black liquor.

A higher dry solids content in the black liquor increases the energy and chemical efficiency of the recovery cycle. However, high dry solids may also lead to plugging and fouling of the equipment due to high viscosity.

typical -73 prism with refractive index (nD) range 1.33-1.53, suits majority of applications in the pulp mills. However, black liquor from hardwood pulp has slightly higher refractive index than black liquor from softwood.

### Black liquor and prism measurement range

The Vaisala K-PATENTS® Process Refractometer PR-23-SD with

We have identified that in countries with acacia and eucalyptus hardwood pulp, the black liquor refractive index may exceed the maximum range of the -73 prism.

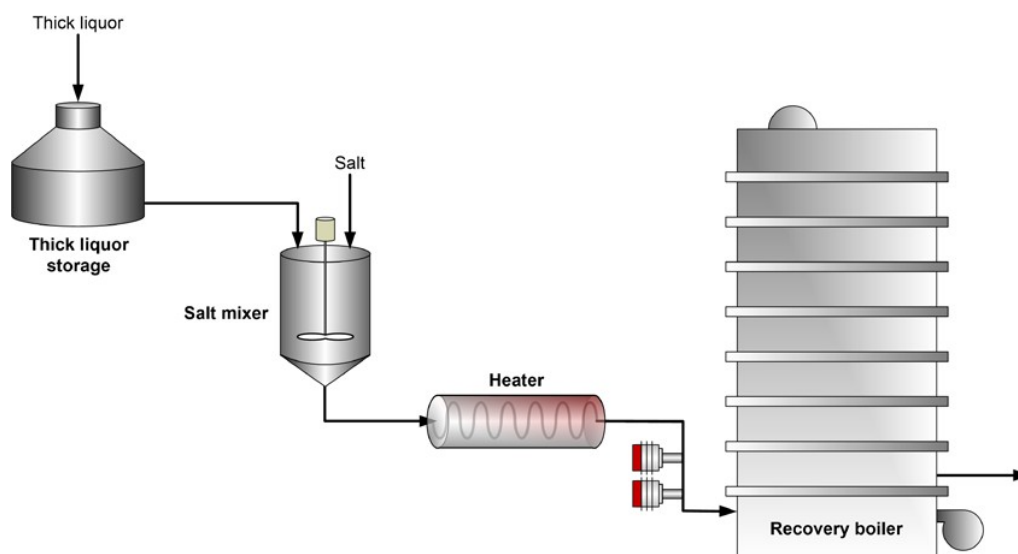
### Vaisala recommendation

Vaisala recommends using PR-23-SD model with the -72-prism option (nD range 1.36-1.57) for high solids, above 80 % dry solids black liquor applications in countries with acacia and eucalyptus hardwood. Such high solids measurement points are after high dry solids or super concentration, and in recovery boiler.

It should be noted that the refractometers with the -72-prism option do not suit low dry solids applications such as Brown Stock Washing.





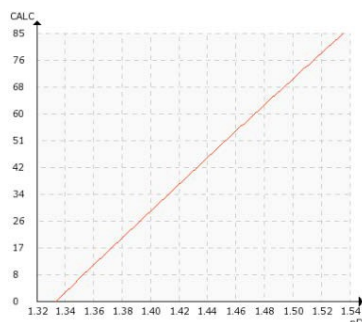


## BLACK LIQUOR

### Typical end products

Wood pulp, paper, board.

Chemical curve: R.I. per Black liquor Conc% at Ref. Temp. of 20°C



### Introduction

The recovery boiler plays a central role in the chemical cycle of a modern pulp mill. The recovery boiler is a chemical reactor, which is used for recovering chemicals from spent kraft liquor and generating energy at the same time.

In the recovery boiler, the organic matter is burned. The dry solids liquor content required for firing is at least 60 %, but preferably more than 65 %. Black liquor is concentrated by evaporating water from the liquor. When the concentration of black liquor is maximized, so is the energy production. Before entering the burners, sodium sulfate decahydrate, or *glauber salt*, is added to cover chemical losses.

### Application

The liquor should have a high content of combustible dry solids in order to minimize flue gas emissions and maximize boiler efficiency.

Too low concentration of dry solids fed to the burners may result in a steam explosion with consequent damage or destruction to the boiler. Therefore, it is essential to utilize a refractometer to monitor the black liquor feed to the recovery boiler to ensure a safe operation.

### Instrumentation and installation


Vaisala K-PATENTS® Digital Divert Control System DD-23 complies strictly with all recommendations of the Black Liquor Recovery Boiler Advisory Committee (BLRBAC).

The DD-23 system includes two SAFE-DRIVE Process Refractometer sensors in the main black liquor line, two indicating transmitters and a divert control unit in an integrated panel.

The sensors are installed using our patented SAFE-DRIVE Isolation valve. This allows for safe and easy insertion and retraction of the refractometers under full operating pressure, without having to valve off the liquor piping or having to shut down the process. The SAFE-DRIVE Isolation valve contains a steam wash system for automatic prism cleaning. The system contains a SAFE-DRIVE Retractor Tool SDR-23 for safe sensor insertion and retraction.

Each refractometer is a completely independent measurement system. Each system sends a separate mA output signal, which can be used by a control system (PLC or DCS). The DD-23 system is entirely microprocessor controlled. The digital signal

transmission and microprocessor implemented diagnostics ensure error free operation.

Instrumentation	Description
	<p>Digital Divert Control System DD-23 for safe operation of kraft chemical recovery boiler. The DD-23 system complies strictly with all recommendations of BLRBAC. The DD-23 system includes two SAFE-DRIVE Refractometer PR-23-SD sensors in the main black liquor line, and two Indicating transmitters and a Divert control unit in an integrated panel. Remote monitoring and event data logging via Ethernet.</p>
Measurement range	<p>Refractive Index (nD) 1.3200 - 1.5300 (-73 prism) or Refractive Index (nD) 1.3600 - 1.5700 (-72 prism)</p> <p>The typical -73 prism suits the full measurement range for majority of measurement points in the process. Prism -72 is used for measuring solids over 80 % in acacia and eucalyptus hardwood based black liquor.</p>

Ref. B211974EN-B © Vaisala 2021



## Recommended refractometer prism option for high solids black liquor applications



### Introduction

In the kraft pulp recovery process, the by-product black liquor is concentrated in evaporators and burned in a recovery boiler to recover the inorganic chemicals for reuse in the pulping process, while producing energy for the pulp mill's use. The weak black liquor is evaporated to 60 % or even up to 90 % dry solids, also known as heavy black liquor.

A higher dry solids content in the black liquor increases the energy and chemical efficiency of the recovery cycle. However, high dry solids may also lead to plugging and fouling of the equipment due to high viscosity.

typical -73 prism with refractive index (nD) range 1.33-1.53, suits majority of applications in the pulp mills. However, black liquor from hardwood pulp has slightly higher refractive index than black liquor from softwood.

### Black liquor and prism measurement range

The Vaisala K-PATENTS® Process Refractometer PR-23-SD with

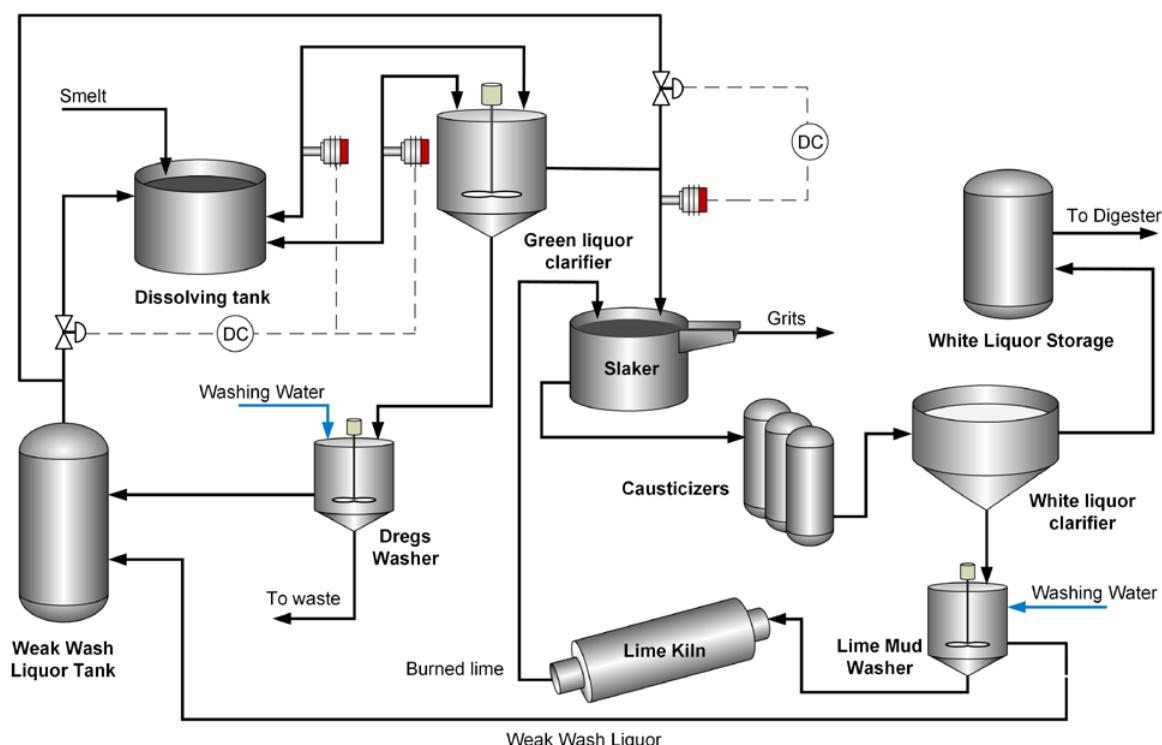
We have identified that in countries with acacia and eucalyptus hardwood pulp, the black liquor refractive index may exceed the maximum range of the -73 prism.

### Vaisala recommendation

Vaisala recommends using PR-23-SD model with the -72-prism option (nD range 1.36-1.57) for high solids, above 80 % dry solids black liquor applications in countries with acacia and eucalyptus hardwood. Such high solids measurement points are after high dry solids or super concentration, and in recovery boiler.

It should be noted that the refractometers with the -72-prism option do not suit low dry solids applications such as Brown Stock Washing.



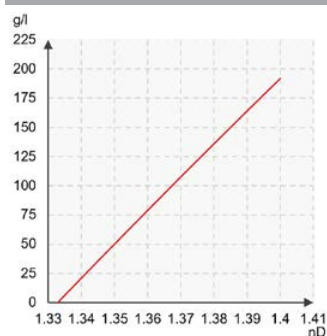


## GREEN LIQUOR

### Typical end products

Unbleached kraft pulp, bleached kraft pulp.

Chemical curve: R.I. per Green liquor density g/l at ref. temp. of 20°C



### Introduction

The chemical recovery process ensures the operation and economic viability of the mill. In this process, the pulping chemicals are regenerated from the inorganics dissolved in the spent liquor, and the organic material is burned to generate steam. The process consists of an evaporation plant, a recovery boiler and a causticizing plant.

In the causticizing plant sodium carbonate ( $\text{Na}_2\text{CO}_3$ ) is converted into the active cooking chemical, sodium hydroxide ( $\text{NaOH}$ ). The process can be divided in three main parts: slaking, causticizing and liquor

preparation. The result is the white liquor which is used in the digester. High quality white liquor with consistent and high strength improves the productivity of the whole mill.

### Application

The *smelt* from the recovery boiler is dissolved in weak wash in the dissolving tank to produce *green liquor*. The raw green liquor, consisting mostly of sodium carbonate and sodium sulfide, is commonly pumped into a stabilization tank to even out fluctuations in density and temperature, and to ensure a more constant liquor composition to the causticizing process.

The green liquor clarifier aims to obtain a good clarified green liquor for the slaker. This also reduces dregs carryover which causes problems in downstream equipment. In the slaker, the clarified green liquor is brought into contact with reburned lime. This slaking reaction converts the green liquor into *white liquor* by converting sodium carbonate into sodium hydroxide, an active pulping chemical. Impurities known as *grits* are also separated at this stage. The mixture moves on to the causticizers to provide enough resident time for increasing the causticizing degree to 80-82 %.

The white liquor is produced by the separation of the lime mud,  $\text{CaCO}_3$ , a by-product of causticizing, which is converted back into lime using a lime kiln.

The causticizing process is controlled by controlling the slaker operation, which in turn depends on the concentration of the raw green liquor's Total Titratable Alkali (TTA). The goal is to stabilize the density or TTA concentration in the green liquor feed to the slaker to avoid overliming and ensure a safe operation. TTA measurements in the main green liquor lines (from dissolving tank and clarifier) are required for control purposes.

## Instrumentation and installation

Vaisala K-PATENTS® SAFE-DRIVE Process Refractometer PR-23-SD measures the density or TTA concentration of green liquor at two stages of the process: after the green liquor dissolving tank, and after the green liquor clarifier at the slaker feed.

The refractometer's sensor is mounted directly in the pipelines for in-line measurements allowing real-time and active control to meet the target TTA.


Pirssonite formation in the pipe walls is a frequent problem for instrumentation in green liquor application. This may be as much as an inch per week. Traditional

methods such as density meters and  $dP$  are not reliable due to constant scaling inside the instrument's tubes and drifting of the measurement.

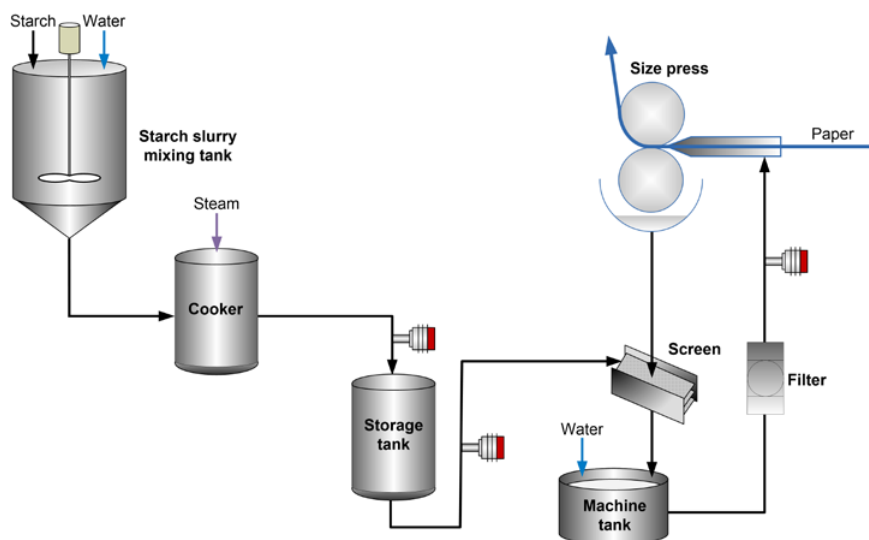
The SAFE-DRIVE refractometer has been designed for accurate measurement in these difficult scaling conditions. The digital measurement is unaffected by bubbles, suspended particles or color changes to the green liquor.

Automatic prism is required to keep the prism clean, securing representative samples and continuous information for real-time control. We recommend using blow out water or intermediate feed water to the boiler as the water source for the prism wash system (around 30-50 bars and above 90 °C). Typical measurement range is 100-150 g/l (6.0-8.5 lb/ft³) and the process temperature is 85 °C (185 °F).

Effective causticizing control improves the quality and stability of white liquor, decreases operating costs and increases pulping efficiency. Well performed lime dosage control reduces the recirculation flow of lime in the process, leading to less lime reburning in the lime kiln and decreased energy consumption.

Instrumentation	Description
	SAFE-DRIVE Process Refractometer PR-23-SD for measuring black liquor dry solids and green liquor density or TTA in kraft chemical recovery process. SAFE-DRIVE design allows for safe and easy insertion and retraction of the sensor under full operating pressure without having to shut down the process.
<b>Automatic prism wash</b>	<p><b>For dissolving tank installations:</b> prism wash with hot water. The components of a wash water system are a sensor with integral water nozzle mounted at the sensor head, a HPV wash system, a power relay unit and an indicating transmitter equipped with relays. We recommend using blow out water or intermediate feed water to the boiler as the water source for the wash system (around 30-50 bars and above 90 °C). If none of these are available on site, please contact your local representative or your Area Manager for advice.</p> <p><b>For slaker installations:</b> prism wash with steam. The components of a steam wash system are a sensor with integral steam nozzle mounted at the SAFE-DRIVE valve, a shut-off valve for steam line and an indicating transmitter equipped with relays to drive the wash valves.</p>
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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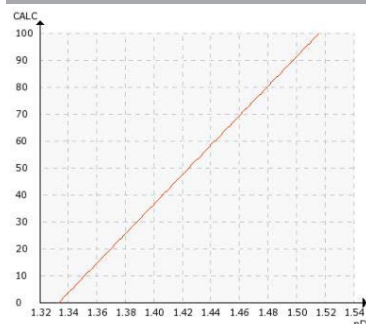


## SIZING LIQUID, PVA, CMC, STARCH

### Typical end products

Bond, ledger, writing and other types of paper.

Chemical curve: CMC R.I. per Conc. % by weight at Ref. Temp. of 20°C



### Introduction

Sizing is used in papermaking to reduce the paper's tendency to absorb liquids when dry, so that inks and paints remain dry on the surface of the paper, rather than absorbing into the paper. The various treatments can be divided into internal sizing, surface sizing and coating. Refractometer applications are designed for the use and preparation of these sizing liquids.

The most common material used in sizing solutions is starch. Other additives such as Polyvinyl Alcohol (PVA) and Carboxymethyl Cellulose (CMC) can be used to provide specific strength and optical improvements to the paper. Normally, a relatively low-viscosity concentrated starch solution (6-14 %) is used at the size press in order to achieve a starch pickup of about 45 kg (99 lb) per 1000 kg (2200 lb) of product.

### Application

#### Sizing Liquid Cooking

The liquid is usually cooked in continuous cookers. The cooked starch is diluted with water to the desired concentration before storage. The temperature in the storage tanks is regulated with a steam heating system and a mixer.

#### Size Press


The size liquid is commonly added to the paper with a *size press* or *blade coater*. This liquid is re-circulated at the size press. As the paper runs through the size press, it absorbs a portion of the solution, while any surplus is collected for re-circulation to the press.

### Instrumentation and installation

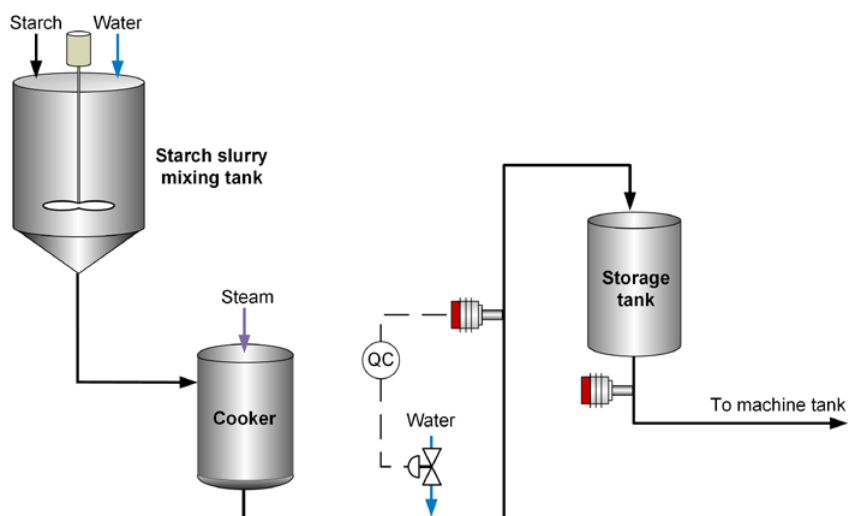
Vaisala K-PATENTS® Process Refractometer PR-43 measures dissolved components such as starch, latex, CMC and PVA.

Kaolin and pigments cannot be measured as they have no effect on the Refractive Index.

Typical measurement range of starch is 0-20 %. High pressure water prism cleaning is recommended.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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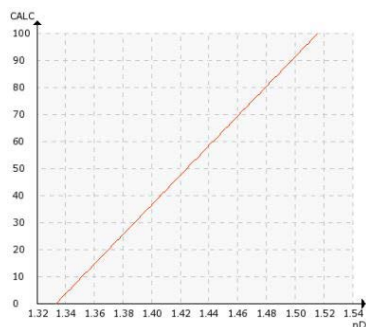


## SIZING LIQUID, PVA, CMC, STARCH

### Typical end products

Bond, ledger, writing and other types of paper.

Chemical curve: CMC R.I. per Conc. % by weight at Ref. Temp. of 20°C



diluted to the desired concentration by adding water to it before it enters the storage tank.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to control the concentration of the cooked starch. The refractometer verifies that the liquid has been cooked properly. Precise measurement of the concentration provides consistency for the different paper grades production.

Typical measurement range is 0-15 % starch. Automatic prism wash using high-pressure warm water is recommended in this application.


### Introduction

Cooked starch is used in paper sizing to reduce the paper's tendency to absorb liquid when dried. The goal is to allow inks and paints to remain on the surface of the paper and to dry there.

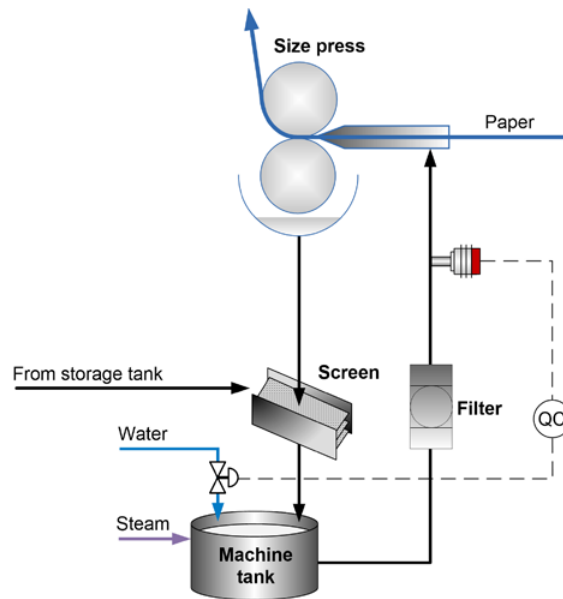
In addition, sizing affects abrasiveness, creasability, finish, printability, smoothness, and surface bond strength and decreases surface porosity and fuzzing.

### Application

The liquid is usually cooked in continuous cookers. The starch is cooked by introducing steam directly into the starch slurry. The cooked starch is cooled and

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

Ref. B211978EN-A © Vaisala 2020

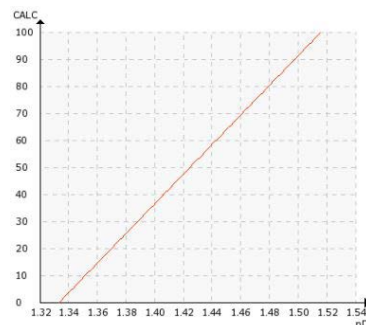


## SIZING LIQUID, PVA, CMC, STARCH

### Typical end products

Bond, ledger, writing and other types of paper.

Chemical curve: CMC R.I. per Conc. % by weight at Ref. Temp. of 20°C



### Introduction

The size liquid is commonly added to the paper with a *size press* or a *blade coater*. From the storage tank, the sizing liquid is pumped into the size press circulation system.

There is a constant flow from the machine tank to the size press. At the size press, the sizing liquid is sprayed onto the paper. The paper absorbs a portion of the sizing liquid with the surplus being recirculated through a screen to the machine tank.

At the size press, the paper absorbs more water than sizing material. This changes the concentration of the liquid to the recirculation line. Therefore, it is

extremely important to measure precisely the size's concentration levels in the size press circulation system. Water must be added into the circulation to maintain the dilution levels.


### Application

Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to control the sizing liquid concentration in order to optimize the paper sizing process. The refractometer stabilizes sizing conditions and keep paper quality constant.

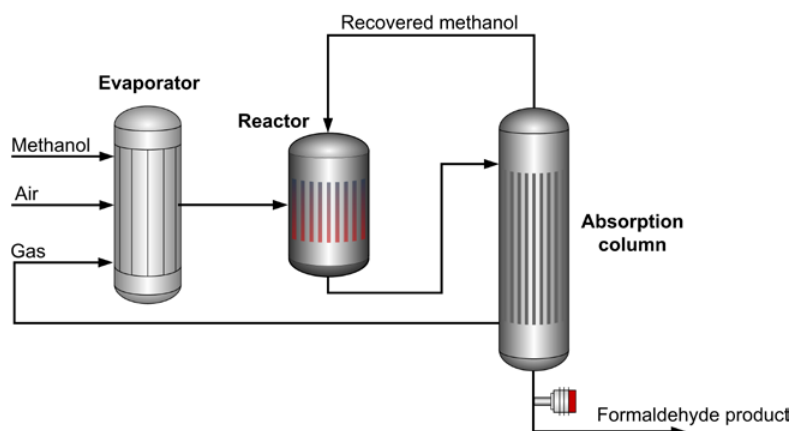
### Instrumentation and installation

Typical measurement range is 0-15 % starch. Usually the bottom layer concentration is lower than the top layer concentration. Automatic prism wash with high-pressure warm water is recommended in this application.



Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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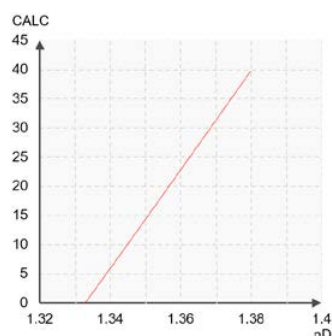


## FORMALDEHYDE, CH<sub>2</sub>O (METHANAL)

### Typical end products

Urea (30 %), phenol (20 %), melamine-, acetal (10 %), polyformaldehyde resins, ethylene glycol, pentaerythritol, hexamethylenetetramine, sterilising mediums, resins.

Chemical curve: Formaldehyde R.I. per Conc. % b.w. at Ref. Temp. of 20 °C



### Introduction

Formaldehyde, CH<sub>2</sub>O (also known as *methanal*), is a colorless gas with a characteristic pungent odor. It is a powerful germicide used for sterilizing purposes. It is also the simplest aldehyde.

Formaldehyde can be obtained from its cyclic trimer trioxane and the polymer paraformaldehyde. It exists in water as hydrate H<sub>2</sub>C(OH)<sub>2</sub>. Aqueous solutions of formaldehyde are referred to as formalin. Its pure form, 100 % formalin, consists of saturated solution of formaldehyde (this is about 40 % by volume or 37 % by mass) in water, with a small amount of stabilizer, usually methanol, to limit oxidation and polymerization.

### Application

Formaldehyde production is based on a direct oxidation technique and also, to a certain extent, on a silver catalyst method.

The oxidation technique employs an oxide catalyst for a direct oxidation route to formaldehyde. In this process, a mixture of liquid methanol, air and recycled stack gases are fed into an evaporator. The gas mixture from the evaporator is then fed into a fixed bed tube reactor, and charged with a molybdenum-based catalyst. The methanol content is maintained at 6-10 % of volume.



When the reaction has reached about 350 °C (662 °F), the gases are cooled in the evaporator, where the heat is used for methanol evaporation. The formaldehyde is then absorbed in an absorption tower by using the condensation as the absorption liquid.

### Instrumentation and installation

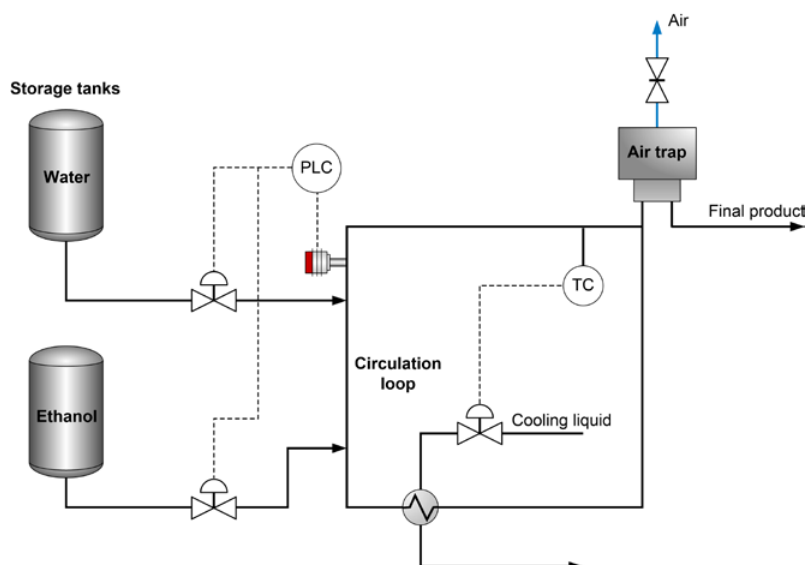
Vaisala K-PATENTS® Process Refractometer PR-43-G is mounted after the formaldehyde pump. With the refractometer the formaldehyde concentration can be measured and maintained within the specified limits. Typical product concentrations vary between 37 % and 45 %.

We recommend a product flow rate of at least 1.5 m/s (5 ft/s).

Substantial savings can be achieved due to consistent product quality. Precise product concentrations will also minimize the need for further product treatment.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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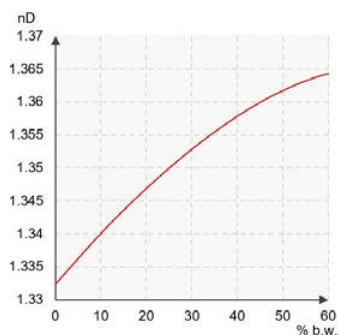
## ETHANOL C<sub>2</sub>H<sub>5</sub>OH, ETHYL ALCOHOL

### Typical end products

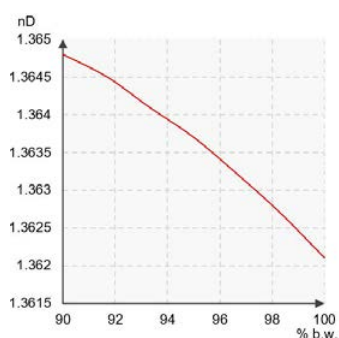
Antiseptics, antidotes, solvent, synthesis of chemicals, spirits, fuel (see also Application Note *Bioethanol Stillage Processing*).

Chemical curve: Ethanol R.I. per Conc% b.w. at Ref. Temp. of 20 °C

### Range 0-60 %



### Range 90-100 %



### Introduction

Ethanol (Ethyl Alcohol) is a colorless liquid, which is miscible with water when heat is applied to it. Alcohol has traditionally been manufactured by the fermentation of biomass containing starch and sugars.

At present, most of the alcohol is produced by the catalytic hydration of ethene. Ethanol, an alcohol suitable for human consumption, is widely used as a solvent and for the synthesis of other chemical products. Ethanol is also used as a fuel.

### Application



Ethanol is used for many applications in different industries. For instance, in the pharmaceutical industry a mixture of alcohol and water is used as the *mobile phase* in HPLC (High Performance Liquid Chromatography).

It is common practice to receive and store ethanol in a concentrated form, and dilute it to the required concentration just prior its use. The required concentration is obtained by mixing a full-strength alcohol solution with water.

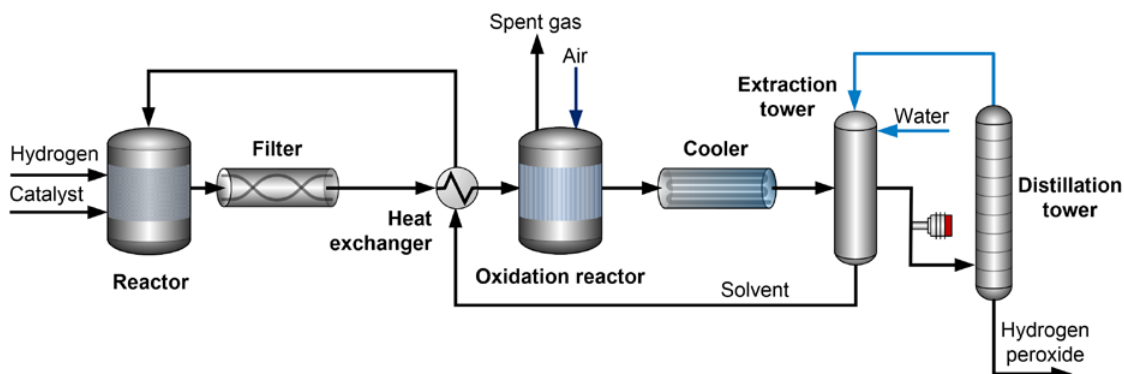
### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-G is used to control and measure the concentration of alcohol in water. The refractometer provides Ethernet and 4-20 mA output signals that can be used as feedback to control the valves in order to achieve always the desired concentration.

For use as a mobile phase in the pharmaceutical industry, a typical measurement range is 0-50 % and the normal process temperature is about 15 °C (59 °F). Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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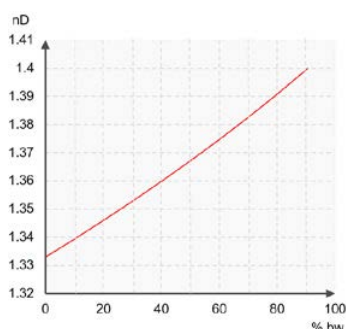


## HYDROGEN PEROXIDE, H<sub>2</sub>O<sub>2</sub>

### Typical end products

Chemical synthesis, metals, alloys, printed circuit board cleaning, etching, brightening, textile bleaching, paper pulp bleaching, aseptic packaging, water filtration, ink removal from waste paper.

Chemical curve: Hydrogen peroxide R.I. per Conc. % b.w. at Ref. Temp. of 20°C



### Introduction

Hydrogen peroxide is a clear, colorless and slightly viscous liquid.

### Application

Hydrogen peroxide is manufactured almost exclusively by the *autoxidation (AO) process*. The process is based on a reduction of anthraquinone, followed by oxidation resulting in the formation of H<sub>2</sub>O<sub>2</sub>.



Hydrogen peroxide is separated from water by extraction and is concentrated to produce grades at standard commercial strengths of 35-65 %. The main purpose of concentration is to decrease storage and the transportation costs of hydrogen peroxide. Moreover, the concentration removes some amounts of impurities, such as organic solvents in hydrogen peroxide.

### Instrumentation and installation

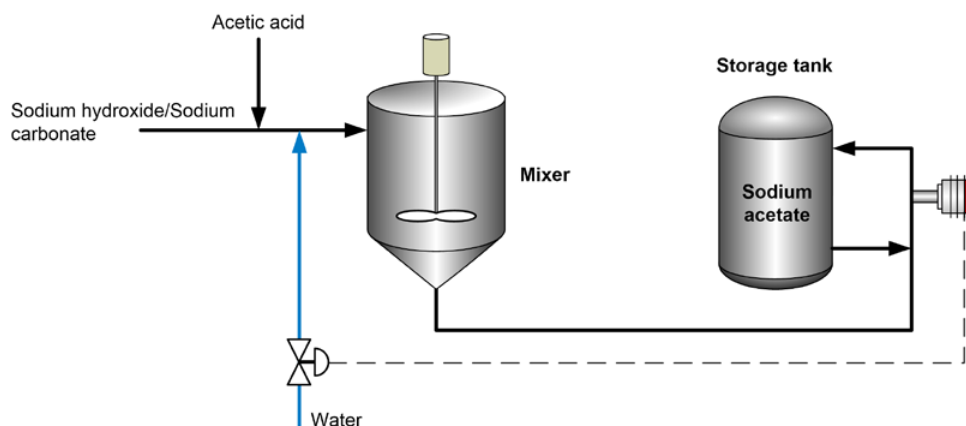
Vaisala K-PATENTS® Process Refractometer PR-43-G can be installed at different points in a H<sub>2</sub>O<sub>2</sub> plant. The refractometer measures the concentration after peroxide extraction to ensure maximum efficiency. At this point the concentration is 30-40 %.

After extraction, H<sub>2</sub>O<sub>2</sub> is purified and concentrated by distilling it to the specified concentration. The refractometer is ideally suited for distillation control. The refractometer provides Ethernet and 4-20 mA output signals that can be used for real-time control of the distillation column. The instant measurement of the refractometer can be used to adjust reflux and boil-up in distillation in order to enhance separation and reduce energy costs.

Hydrogen Peroxide often contains large quantities of hydrogen gas bubbles, which can cause measurement errors in density meters. Due to its digital sensing technology, the refractometer is not subject to measurement errors caused by bubbles, color or particles, and the measurement is selective to the liquid phase. Moreover, the refractometer can be mounted directly in the main line, thus eliminating the need for sampling and off-line tests.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

Ref. B211983EN-A © Vaisala 2020



## SODIUM ACETATE, SODIUM ETHANOATE $C_2H_3NaO_2$

### Typical end products

Pharmaceuticals, photography, dyeing, soaps, pH control, foodstuffs, electroplating.

### Introduction

Sodium acetate ( $NaO_2C_2H_3$ ) is a colorless crystalline compound, which is known as *anhydrous salt* or *trihydrate*.

### Application


Sodium hydroxide, or sodium carbonate, reacts in a static mixer with acetic acid to form sodium acetate.

In the manufacturing process, adding water to the final concentration is very important. The measurement of the final product's concentration has formerly been carried out using titration in a laboratory. This technique often gives inaccurate results due to many possible external factors. Laboratory measurement is also unable to give a continuous indication of true concentration levels.

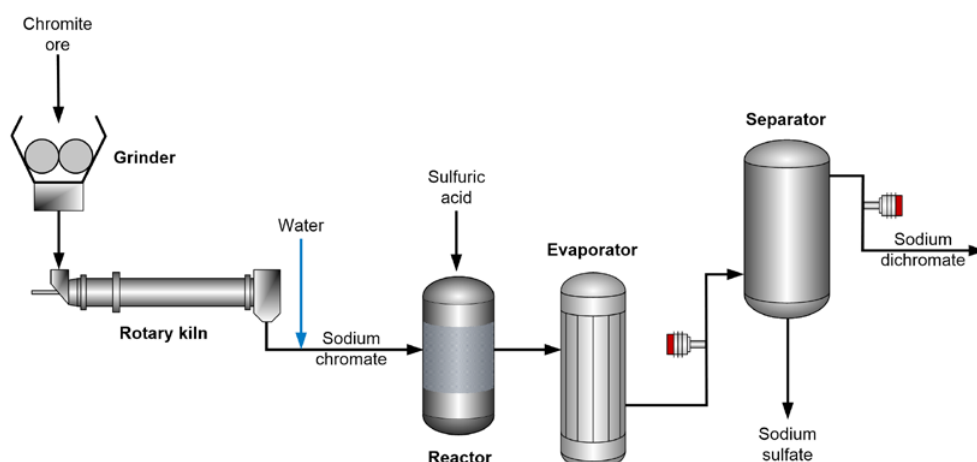
### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GC is mounted in the circulation loop. Output signals from the refractometer are used to control the addition of water to the solution prior to the static mixer, maintaining the final sodium acetate concentration at approximately 7 % b.w. The temperature is between 40 and 50 °C (104 and 122 °F).

Automatic prism wash system with steam is recommended in this application.

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash with steam. The components of a steam wash system are a sensor with integral steam nozzle mounted at the flow cell, a shut-off valve for steam line and an indicating transmitter equipped with relays to drive the wash valves.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.



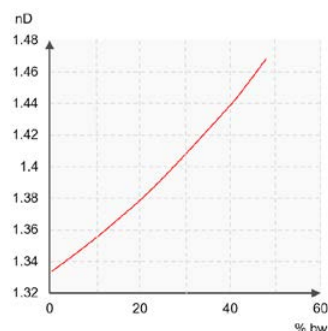


## SODIUM DICHROMATE, $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$

### Typical end products

Chromium metal, magnetic tapes, tanning of leather, preservation compound for timber, metal finishing, pigments for plastic and ceramic industry.

Chemical curve: Sodium dichromate R.I. per Conc b.w. at Ref. Temp. of 20 °C



### Introduction

Sodium dichromate ( $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$ ) is used in the manufacturing of chromium metal, magnetic tapes, leather tanning, timber preservation compounds and metal finishing, as well as pigments for the plastic and ceramic industry. Other applications are used as catalysts and corrosion inhibitors, as well as in the oil and detergent industry.

### Application


Ground chromite ore is roasted with soda ash in large rotary kilns at high temperatures and in an oxidizing atmosphere. This renders the chromium into an extractable form.

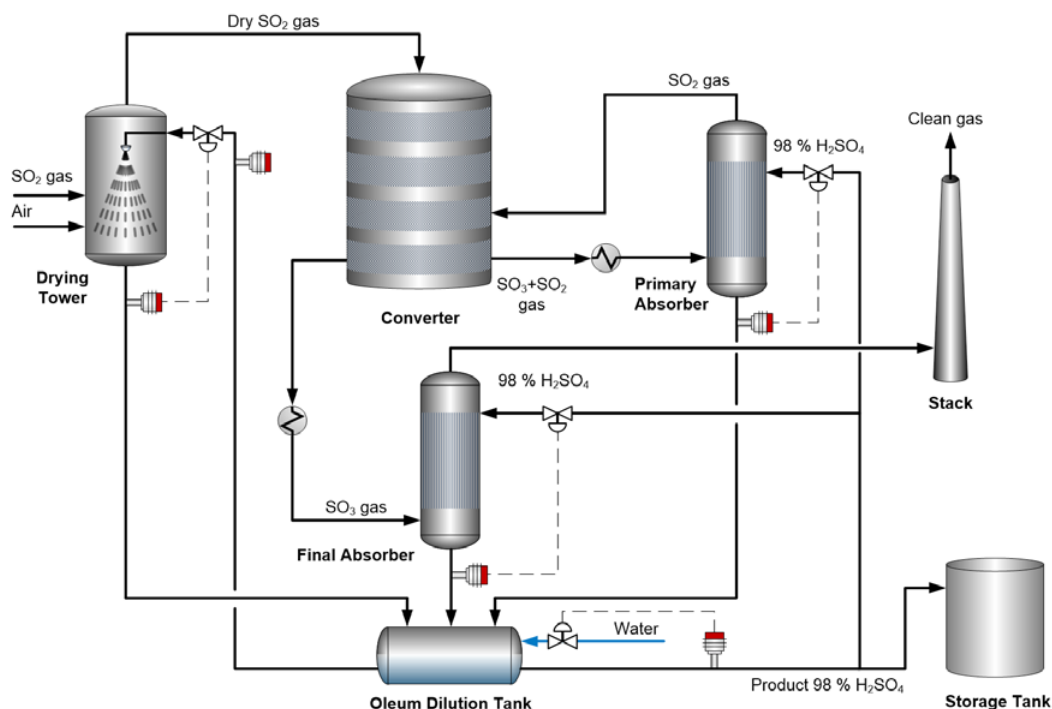
The product is quenched with water, and the sodium chromate is extracted and reacted with sulfuric acid to produce a commercially pure solution of sodium dichromate.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is installed before and after the evaporator to measure in-line the concentration of sodium dichromate. The output signal of the refractometer can be used for real-time monitoring of evaporation efficiency, and for controlling the heat-flow and feed valves to keep a constant concentration.

Typical measurement range of sodium dichromate is 25-55 % and the process temperature is about 85 °C (185°F).

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.



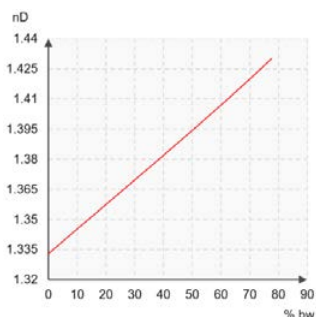
## SULFURIC ACID, $\text{H}_2\text{SO}_4$ , AND OLEUM

### Typical end products

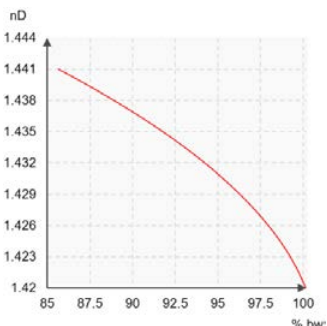
Sulfuric acid and oleum for different uses, e.g. for the production of fertilizers, explosives, dyes, and petroleum products.

Chemical curve: Sulfuric acid R.I. per wt-% at  $T_{\text{REF}}$  of 20 °C

Range 0-80 %



Range 85-100 %



### Introduction

Sulfuric acid ( $\text{H}_2\text{SO}_4$ ) is the most produced chemical in the world. It is widely used in all industries for the manufacture of different substances.

Sulfuric acid is sold in the form of various solutions of  $\text{H}_2\text{SO}_4$  in water or of sulfur trioxide ( $\text{SO}_3$ ) in  $\text{H}_2\text{SO}_4$ . The latter mixture is called *fuming sulfuric acid* or *oleum*, and its marketing is based on the percentage of dissolved free  $\text{SO}_3$ .

Sulfuric acid and oleum are produced industrially in Contact Plants from sulfur-containing gases resulting from, for example, sulfur burning, acid regeneration, or metallurgical operations. The process consists of the catalytic oxidation of sulfur dioxide ( $\text{SO}_2$ ) to  $\text{SO}_3$ , and the hydration of  $\text{SO}_3$  to  $\text{H}_2\text{SO}_4$  by absorption in concentrated acid. Depending on the number of absorption steps, the Contact Plants are classified as *Single* or *Double Contact Process*. The Double Contact Process is the most used technology.

### Application

In the Double Contact Process, dry  $\text{SO}_2$ -containing gas is fed into a large fixed bed reactor consisting of 4 catalytic beds known as *the converter*. The gases are removed from the third catalytic bed, cooled, and passed through a *primary absorber* where  $\text{SO}_3$  is

absorbed in concentrated sulfuric acid (~98 %). The remaining gases (mostly SO<sub>2</sub>) return to the fourth bed of the converter.

After the converter, the gases flow into a *final absorber* where once again SO<sub>3</sub> is absorbed in concentrated acid. The result is oleum and is stored in an oleum dilution tank, where by addition of water the desired acid concentration is achieved. The concentration of the liquid is usually kept at 98 % to avoid acid scaping from the liquid.

The operation of the absorbers is different depending on the desired product. For a sulfuric acid product, 93-98 % acid is used for the absorption, and for oleum or liquid SO<sub>3</sub> the tower is irrigated with 22 % or 35 %oleum.

## Instrumentation and installation



Vaisala K-PATENTS® Process Refractometer PR-43-G is used in various stages of the Contact Process. The refractometer monitors and controls the concentration of acid during drying, absorption and dilution steps to keep the concentration of H<sub>2</sub>SO<sub>4</sub> constant at 93, 98 or 104 % by weight.

The refractometer provides Ethernet and 4-20 mA output signals for real-time process control. The refractometers can be installed in a control loop

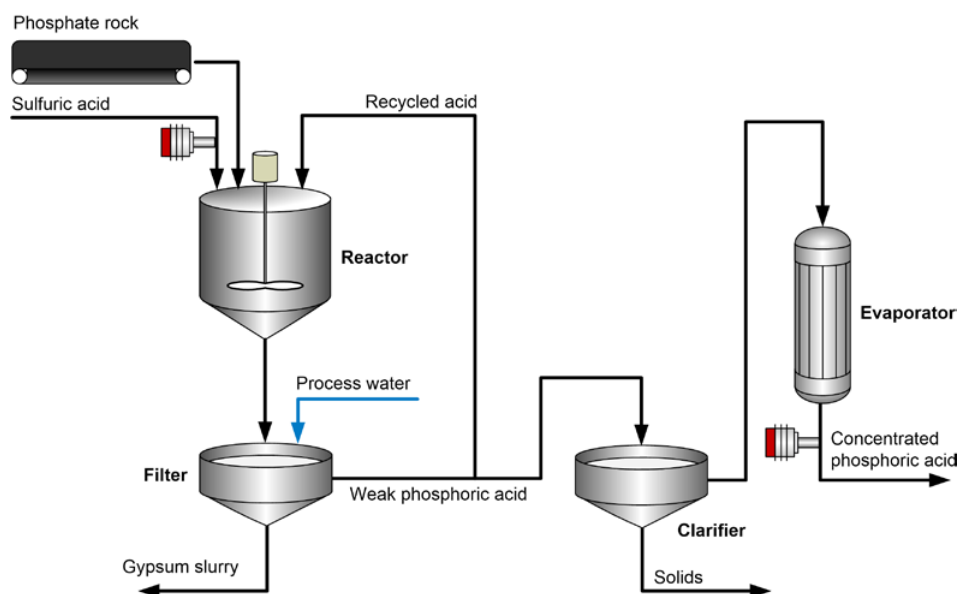
measuring the concentration of acid as it gets blended or concentrated. The refractometer also controls the acid circulation to the towers to ensure operation within the optimal concentration range, and to maximize the absorption.

In the production of oleum, the final product is viscous with a temperature of 80 °C (176 °F) and containing small air bubbles. This is a source of errors in density and ultrasonic meters. The measurement by the refractometer is not affected by bubbles, color or changes in flow. Typical concentrations in oleum applications are 22-35 % at a temperature range of 60-80 °C (140-176 °F).

The refractometer is designed to withstand harsh, corrosive environments and are available with special wetted parts materials and intrinsically safe and hazardous area certification. Both storage tanks and process piping in a sulfuric acid plant can be stainless steel, providing that the sulfuric acid concentration never drops below 95 %. For less concentrated acid, which is very aggressive, we offer a chemically resistant Teflon material option (PR-23-M or PR-23-W) to ensure a viable service life.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Low: 0-80 % H <sub>2</sub> SO <sub>4</sub> . High: 85-100 % H <sub>2</sub> SO <sub>4</sub> . 0-30 % Oleum.

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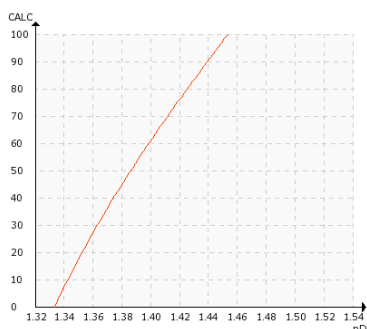


## PHOSPHORIC ACID $\text{H}_3\text{PO}_4$ , SULFURIC ACID $\text{H}_2\text{SO}_4$

### Typical end products

Phosphoric acid for different industrial applications such as the manufacture of agricultural phosphate fertilizers, detergents, pesticides, metal coating, etc.

Chemical curve: R.I. vs. Phosphoric Acid % b.w. at Ref. Temp. of 20°C



### Introduction

Phosphoric acid ( $\text{H}_3\text{PO}_4$ ) is the second most produced inorganic acid in volume, after sulfuric acid ( $\text{H}_2\text{SO}_4$ ). It is an important acid used industrially mainly in the production of phosphate agricultural fertilizers. Due to its non-toxic and mildly acidic nature, phosphoric acid is also used in other applications including food flavoring, soft drinks, pharmaceuticals, dental products, cosmetics, and skin care products.

Phosphoric acid is produced by mining the naturally occurring phosphate rock and processing it via a *wet process* or a *thermal process*. The wet process is by far the most common method, as the resulting phosphoric acid is of adequate quality to produce

fertilizers. About 80 % of the world's phosphoric acid is obtained by the wet process.

The concentration of phosphoric acid is normally expressed as percent of phosphoric anhydride (%  $\text{P}_2\text{O}_5$ ) rather than %  $\text{H}_3\text{PO}_4$ . Phosphoric acid is available as a commercial or merchant grade acid (40-54 %  $\text{P}_2\text{O}_5$ ), and high grade acid (75-85 %  $\text{P}_2\text{O}_5$ ).

### Application

The wet process consists of three main steps: reaction, filtration and concentration. The phosphate rock is first ground and fed to a reactor vessel for an acidulation reaction with sulfuric acid. In the reaction, the tricalcium phosphate in the phosphate rock is converted to phosphoric acid and to the insoluble salt calcium sulfate ( $\text{CaSO}_4$ ), also known as *gypsum*.

The concentration of sulfuric acid is carefully monitored and maintained to 93-98 %. Keeping the concentration at the specified level is important as it affects the acidulation reaction rate and crystallization of gypsum. In addition, control of the sulfuric acid concentration ensures the production of the strongest possible acid which reduces the energy requirement at the evaporators.

The next step is filtration. The thick slurry from the reactor contains approximately 30 % of solids particles, mainly gypsum and unreacted phosphate rock. The solids are separated and washed from the phosphoric acid by filtration, and the result is a phosphoric acid of 32 %  $\text{P}_2\text{O}_5$  (about 50 %  $\text{H}_3\text{PO}_4$ ).

Finally, the acid concentration is increased by evaporation to obtain a commercial grade acid of 54 %  $P_2O_5$  (70 %  $H_3PO_4$ ). Some facilities may perform further purification to obtain a higher-grade phosphoric acid which is suitable for other applications, for example in the food, pharmaceutical and cosmetics industries.

## Instrumentation and installation

The concentration of the acids during the wet process can be measured in-line with Vaisala K-PATENTS® Process Refractometer PR-43-GP. The refractometer is installed directly in the process line to provide real-time measurement of the sulfuric and phosphoric acid concentrations, eliminating the need for sampling and laboratory titration tests.


At the reactor step, the refractometer is used to monitor the concentration and quality of the sulfuric acid feed. By using the correct sulfuric acid concentration (93-98 %), the factories ensure that only phosphoric acid of high strength is produced and less energy at the evaporators is consumed. Moreover, control of sulfuric acid concentration is necessary to regulate the heat load in the reactor vessel, as well as to control gypsum crystallization. Good crystal shape and size

distribution optimize the efficiency of the filtration step, which has impact to the overall productivity of the process.

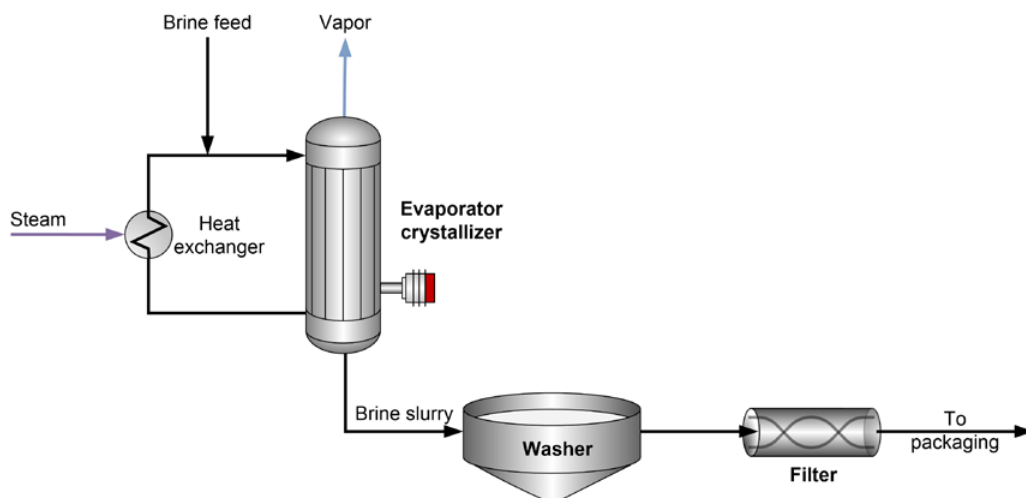
Another refractometer at the evaporation step helps to reduce operation costs by monitoring the final phosphoric acid concentration. The refractometer's Ethernet and mA output signals can be used for automatic control of the evaporator, ensuring the specified concentration is always obtained, while optimum energy consumption is maintained.

The output of the refractometer can be configured to indicate the preferred concentration unit by the operators, for example percent of sulfuric acid, phosphoric acid, phosphoric anhydride or simply the refractive index value.

Due to its unique digital sensing technology, the measurement of the refractometer is not affected by bubbles or suspended solids, and the measurement is selective to the liquid phase. This makes the refractometer ideal for measuring acid concentrations also at other points of the process if required, for instance in mid-points between reaction and filtration, or filtration and concentration.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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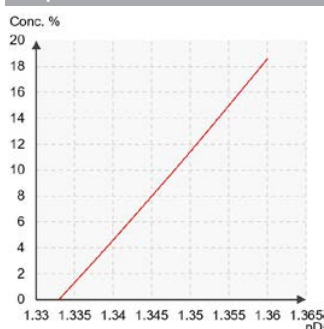


## SODIUM SULFATE, Na<sub>2</sub>SO<sub>4</sub>

### Typical end products

Table salt, flavoring agents, preservatives.

Chemical curve: Sodium sulfate R.I. per Conc% b.w. at Ref. Temp. of 20°C



For special grades of brine to be used in foods or chemicals, pretreatment of the brine solution may be required to remove calciums. The main impurity left in the saturated brine feed is dissolved sodium sulfate (Na<sub>2</sub>SO<sub>4</sub>).

The saturated brine solution is recycled through a heat exchanger in a crystallizing evaporator. By adding heat, the saturated brine solution starts to crystallize and evaporate.

If the brine solution is over heated, a large quantity of sodium sulfate will also crystallize. This makes the salt impure. It is essential to know the amount of sulfate present in the brine to achieve both rapid production and high salt quality.

The salt-brine slurry is then washed, dried and filtered, before packaging.

### Introduction

Sodium chloride (NaCl), or common table salt, is a water soluble and colorless crystalline solid. The solution of dissolved salt in water is called *brine*.

Sodium chloride occurs as rock salt in nature, in natural brines, such as sea water. Rock salt deposits are mainly located in the USA.

The largest use of salt (in the form of brine) is in the electrolytic production of chlorine. In the food industry, salt is used as a food flavoring agent, preservative and color developer.




### Application

Salt is obtained in three different ways: solar evaporation of seawater, mining of rock salt, and from brine wells (solution mining).

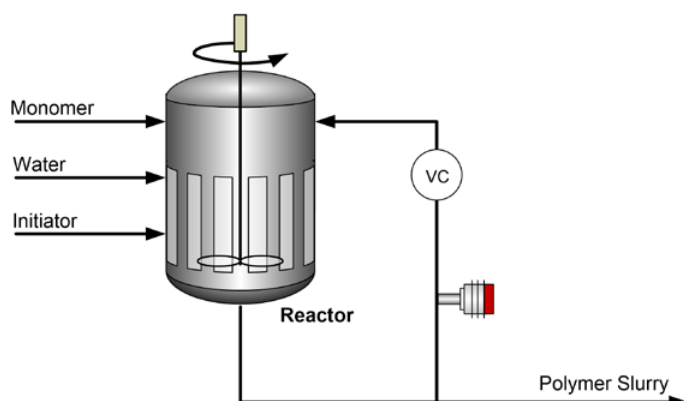
### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer measures the sulfate levels of the brine in the crystallizing evaporator. By measuring the amount of impurities, the crystallization process can be controlled better, thus giving high purity salt with reduced production times.

Typical measurement ranges are 0-30 g/l (0-0.25 lb/gal) sulfate in brine. Common temperatures are 40-60 °C (104-140 °F).

Instrumentation	Description
	Teflon Body Refractometer PR-23-M. A compact refractometer for chemically aggressive solutions and ultra-pure fine chemical processes. Connected to the process by a G1/2" female or a 1/2" NPT process connection. It has a built-in flow cell designed to keep all metal and other easily corroding parts from coming into contact with the process liquid.
	Saunders Body Refractometer PR-23-W. A heavy-duty refractometer for chemically aggressive liquids in large-scale production and in large pipe sizes (diameter 50, 80 or 100mm/2", 3" or 4"). The Saunders body material is graphite cast iron, which provides a solid mechanical base. A PFA-lining ensures the chemical resistance.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
<b>User Interface</b>	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.



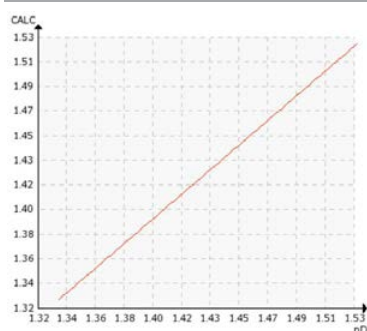


## POLYMER SLURRY

### Typical end products

Polymers and copolymers such as nitrile butadiene rubber (NTB), polyvinyl alcohol and polyacrylates.

Chemical curve: R.I. at Ref. Temp. of 20 °C



### Introduction

Polymers are macromolecules built up by a large number of small molecules called *monomers*. Polymers are found everywhere in our everyday life and are used for many purposes, for example, for packaging, paints, fabrics, rubber, plastic bags, electric components and medical supplies.

Polymers are produced through chemical reactions that combine monomers into large polymer molecules. This process is known as *polymerization*. If the resulting polymer involves two or more types of monomers, the process is known as *copolymerization*.

Polymerization on an industrial scale is conducted using five basic methods: bulk, solution, suspension, emulsion, and gas-phase. In all polymerization processes, the reaction should be closely controlled as the polymer properties are very sensitive to the operational conditions. Control of the polymerization results in high quality specialty polymers that meet specific applications and market requirements.

### Application

Polymerization is commonly done as a solution in stirred reactors. A charge normally consists of monomer, initiator, and a liquid reaction medium (e.g. a solvent or water). Different monomers can be combined to improve the final properties and produce advanced technical polymers.

The reaction starts once the initiator is added. During polymerization, the initially non-viscous liquid monomer is converted into a polymer solution with increasing solution viscosity. As the reaction proceeds, the amount of monomer decreases, and the polymer concentration increases, changing the refractive index value of the polymer slurry.

The reaction continues until the target conversion or polymerization degree is reached. At that point, other ingredients are added, or the polymerization is simply stopped. After polymerization, the polymer slurry is removed from the vessel and is pumped to further treatment and purification, for example, through distillation, condensation, filtration or monomer recovery.

The polymerization reaction can be carried out at low or elevated temperatures and it can take several hours. The amount of reactants and reaction end-point typically depends on the polymer specific recipe. Manufacturers that produce more than one polymer or copolymer, usually have different recipes for each product based on the polymer's refractive index or viscosity value.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP provides accurate and reliable refractive index measurement of the polymer slurry. The



refractometer is installed in-line in the bypass line of the reactor or directly at the bottom of the vessel, through the steam jacket. The continuous measurement of the refractometer reduces the need for sampling and laboratory analyses, promoting safe processing, and increased productivity.

Changes in the refractive index can be monitored in real-time to follow closely the polymerization reaction and the degree of polymerization. The PR-43-GP indicates when the target conversion and reaction end-point are achieved. This increases monomer conversion and polymer productivity.

The measurement by the refractometer also provides valuable information for better understanding of the reaction. For example, the residence times can be optimized, and the concentration of residual monomer and side-products can be minimized. The presence of excessive monomer or other components in the polymer slurry might have a negative effect in post-polymerization treatments and require more costly operations.

Conventional methods to monitor polymerization, such as Gas Chromatography (GC) or laboratory tests, require time-consuming sampling and analyses. In addition, solvent evaporation, variations in the sampling line versus the reactor, and the operator's own assessment are sources of error.


In-line viscosity is also widely use for monitoring polymerization. However, some polymerization reactions are very sensitive to the amount or concentration of charged initiator. For instance, an

excess of initiator will result in a solution that appears to not polymerize (polymer chains stay so short that gelation does not take place and the polymer stays in the solution). The PR-43-GP provides a reliable measurement of all the dissolved material in the polymer slurry in order to identify problems and take corrective actions. The refractometer's output signal can also be used for automatic dilution of the polymer product with water, when necessary.

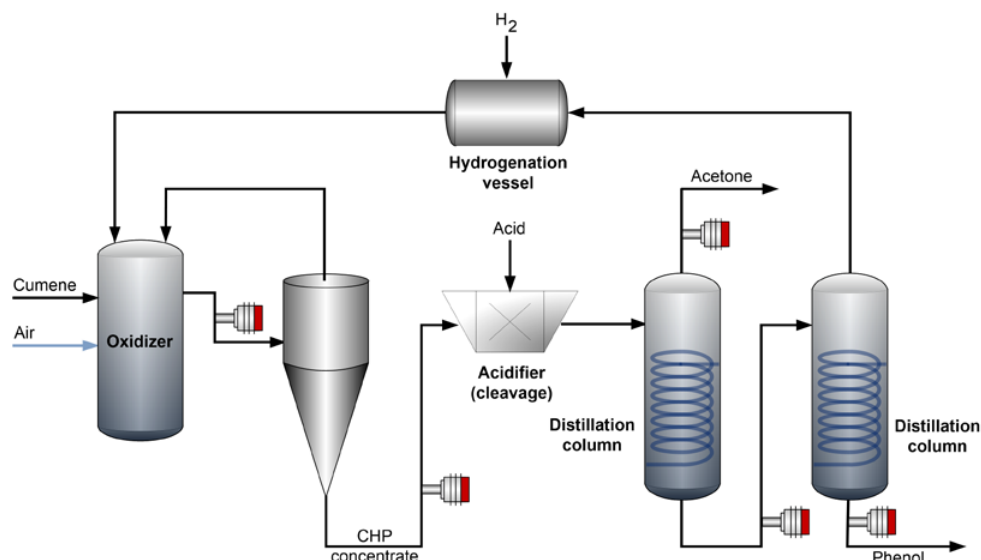
The refractometer is factory calibrated for the full refractive index and temperature range. The refractometer converts the measured refractive index and temperature values directly to concentration units for the given process medium. The conversion of the refractometer matches exactly the standard chemical curves. Alternatively, a refractive index based curve can be used for a refractive index set-point. Temperature changes are automatically compensated in the readings. Because of its unique 3-layer calibration method, the refractometer offers free interchangeability between applications or recipes without any mechanical adjustment. The refractometer is maintenance-free and does not require recalibration.

The precise, in-line measurement of refractive index by the refractometer provides an important tool for controlling product quality, and optimizing the process.

Automatic prism wash may be required in this application. Hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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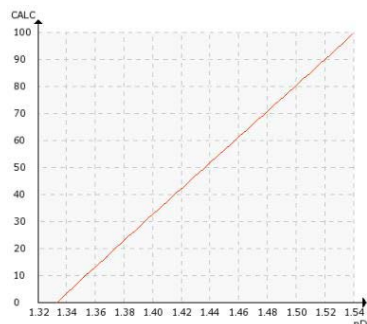


## PHENOL $C_6H_5OH$ , CUMENE HYDROPEROXIDE $C_6H_5C(CH_3)_2O_2H$ , ACETONE $CH_3COCH_3$

### Typical end products

Phenolic resins, bisphenol A, caprolactam, alkylphenols, nylon, dyes, pharmaceuticals, perfumes.

Chemical curve: Phenol R.I. per Conc. % b.w. at Ref. Temp. of 20°C



### Introduction

Phenol ( $C_6H_5OH$ ) is a white, crystalline mass. It has a distinctive sweet, tarry odor and a burning taste.

### Application

The cumene oxidation route is the leading commercial process of synthetic phenol production, accounting for more than 95 % of phenol produced in the world. The remaining 5 % is manufactured with a toluenebenzoic acid process.

### Cumene Process

A typical phenol plant can be divided into two main areas: a reaction area and a recovery area. In the reaction area, cumene is oxidized to produce cumene hydroperoxide (CHP). The cumene hydroperoxide is concentrated to about 80 % and fed to a reactor in which the cumene peroxide is cleaved to phenol and acetone. The cleavage reaction is carried out in the presence of an acid catalyst (e.g. sulfuric acid). The by-products of the cleavage reactions, as well as the catalyst, must be neutralized and extracted to avoid corrosion problems downstream.

In the recovery area, the phenol and acetone products are recovered and purified by distillation. Alpha-methylstyrene (AMS) is also recovered in this section. The AMS may be hydrogenated back to cumene or retained as a product. The recovered cumene (hydrogenated AMS) is recycled as feedstock to the reaction area.

### Instrumentation and installation

The K-PATENTS® Process Refractometer measures in-line the concentration of CHP after the reactor and at the evaporation stage. The measurement from the refractometer ensures process safety and provides the basis for continuous process optimization.


Typical measurement range after the reactor is 10-50 % at a temperature of 100 °C (212 °F). After

evaporation the concentration is about 90 % at a temperature of 90 °C (194 °F).

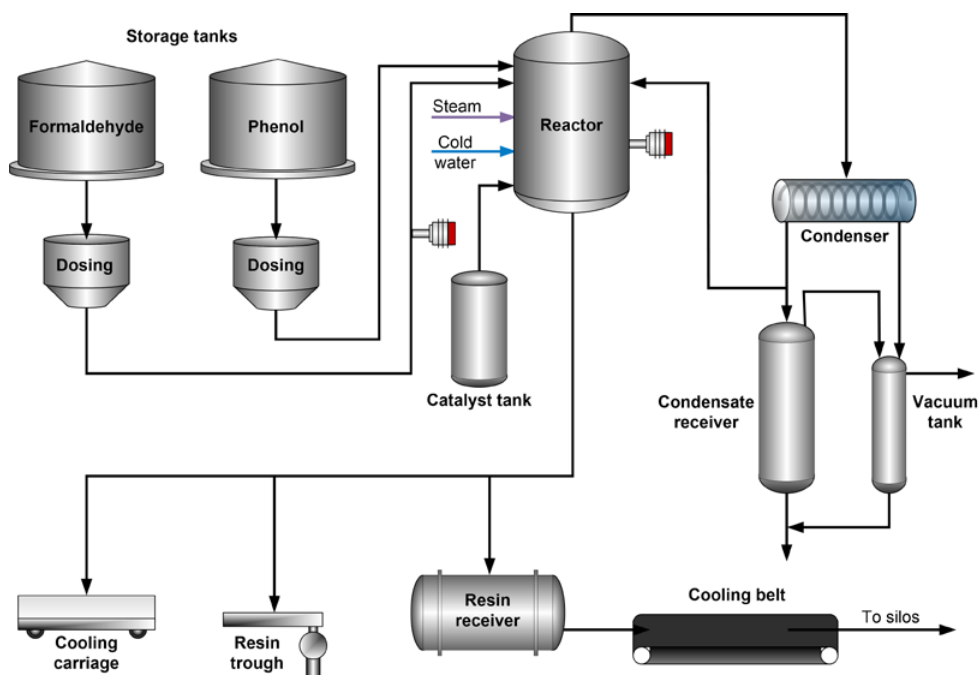
The refractometer is also installed at the top and bottom of the distillation towers to measure phenol and acetone concentrations.

The refractometer's output signal can be used as real-time feedback to adjust reflux and boil-up rates of the distillation tower for maximized efficiency.

Hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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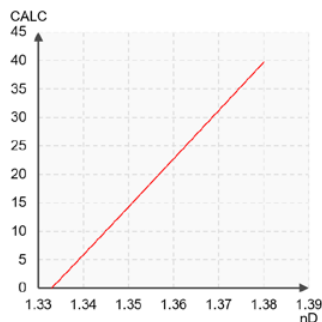


## PHENOL $C_6H_5OH$ , FORMALDEHYDE $CH_2O$ (HCHO)

### Typical end products

Moulded products, coatings, bonding, Bakelite.

Chemical curve: Phenol R.I. per Conc. % b.w. at Ref. Temp. of 20°C



### Introduction

Phenolic resins are formed by reacting phenol and formaldehyde. In the basic process, where a high ratio of formaldehyde to phenol is used, the result is a resol phenolic resin (base catalyst). When using an acid catalyst combined with a predominance of phenol, the result is a novolak phenolic resin. The production is either a batch or a continuous process.

### Application

#### Novolak resins

In a conventional novolak process, molten phenol is placed into the reactor, followed by a precise amount of acid catalyst. The formaldehyde solution is added at a temperature of around 90 °C (194 °F), and a formaldehyde-to-phenol molar ratio of 0.75:1 to 0.85:1 should be achieved. For safety reasons, it is preferred to add the formaldehyde gradually instead of adding the entire charge at once. The reaction is completed after 6-8 hours at 95 °C (203 °F). After the reaction, volatiles, water, and some free phenol are removed. The final phenol content in the resin is monitored carefully, as the free phenol content determines the resin properties.

#### Resol Resins

Phenol and formaldehyde solutions are added simultaneously to the reactor at a molar ratio of formaldehyde to phenol of 1, 2-3,0:1. In the reaction phase, the temperature is held at 80-90 °C (176-194 °F) and the reaction lasts 1-3 hours. When the desired end-point is reached, the content of the reaction vessel is cooled. In cases when liquid resin is recovered as a 40-50 wt-% water solution, the resin is refrigerated for storage.

The reaction steps are typically condensation, followed by distillation. Process temperature and the pH are closely monitored during the reaction. The Refractive Index gives an in-line indication of the reaction progress. In the distillation phase, the degree and completion of the reaction is determined by the refractive index.


## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP measures the formalin concentration before the reactor.

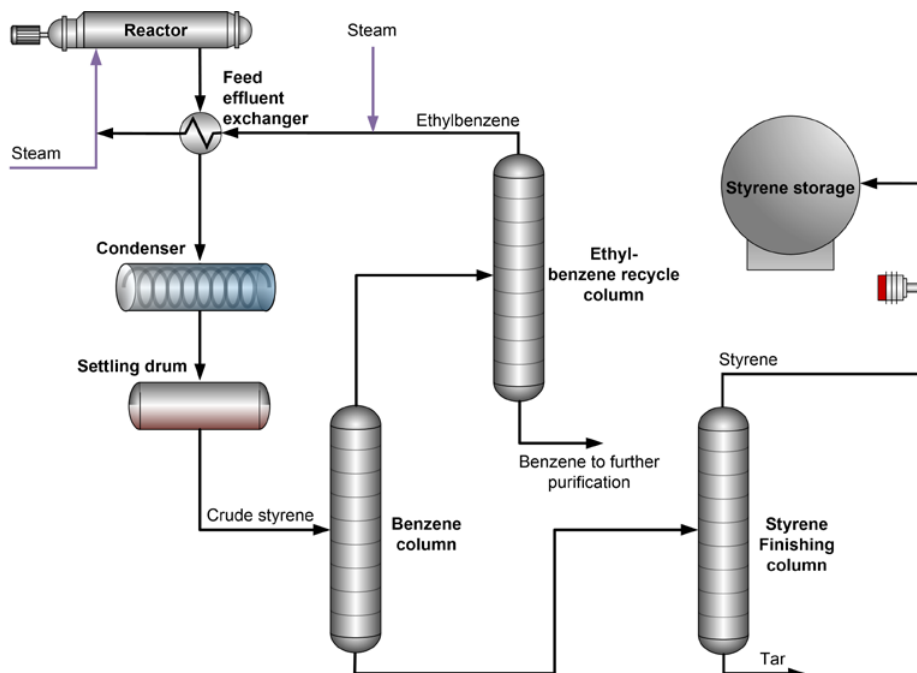
In a batch or continuous reactor, the refractometer is used in the distillation phase to monitor the degree of reaction, to follow the progress of the reaction and to determine the end-point of the reaction.

The refractometer offers precise and reliable measurement. By using a special YAG prism, refractive indexes as high as 1.63 can be verified.

In both applications, the continuous measurement offers valuable real-time information for process control. Automatic prism wash with steam or high-pressure water is recommended. Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic Prism Wash	<p><b>Prism wash with steam:</b> The components of a steam wash system are a sensor with integral steam nozzle mounted at the sensor head, a shut-off valve for steam line and an indicating transmitter equipped with relays to drive the wash valves.</p> <p><b>Prism wash with high pressure water:</b> The components of a high-pressure water system are a sensor with integral water nozzle mounted at the sensor head, a high pressure pump together with a power relay unit and an indicating transmitter equipped with relays.</p>
Measurement range	Refractive Index (nD) 1.3700 – 1.6300, corresponding to 0-100 % by weight.

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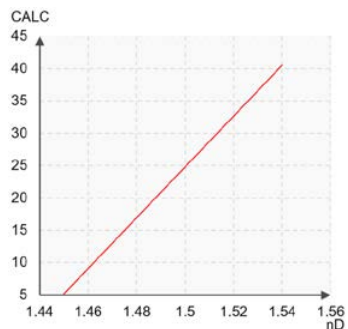


## STYRENE C<sub>8</sub>H<sub>8</sub>

### Typical end products

Polystyrene, ABS plastics, styrene butadiene, rubber.

Chemical curve: Ethylbenzene in Styrene R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Styrene is a colorless, aromatic liquid. The main use for commercial styrene is as a feedstock for polymerization and copolymerization.

The two process routes that are used for styrene manufacturing are dehydrogenation and coproduction with propylene oxide. Nearly 90 % of styrene production utilizes dehydrogenation, mainly because of its simplicity and cost-effectiveness.

### Application

#### Dehydrogenation of Ethylbenzene

In the dehydrogenation process, fresh and recycled ethylbenzene is mixed with steam and heated to the required reaction temperature in a heat exchanger. The mixture is then fed into the reactors. The reactor's hot effluent passes through heat exchangers to preheat the ethylbenzene-steam mixture. It is also utilized to generate steam for the distillation train. Then, the condensed reactor effluent is separated into vent gas, condensate and crude styrene, in a settling drum.


#### Purification of Styrene

Typically, a distillation train is used for styrene purification. Crude styrene is brought to a benzene-toluene column, where benzene and toluene by-products are recovered. The rest is distilled in the ethylbenzene recycle column, where ethylbenzene and styrene are separated. Following this, the ethylbenzene is recycled to the dehydrogenation section and the residue is pumped into the styrene finishing column, where the final styrene purification is achieved.

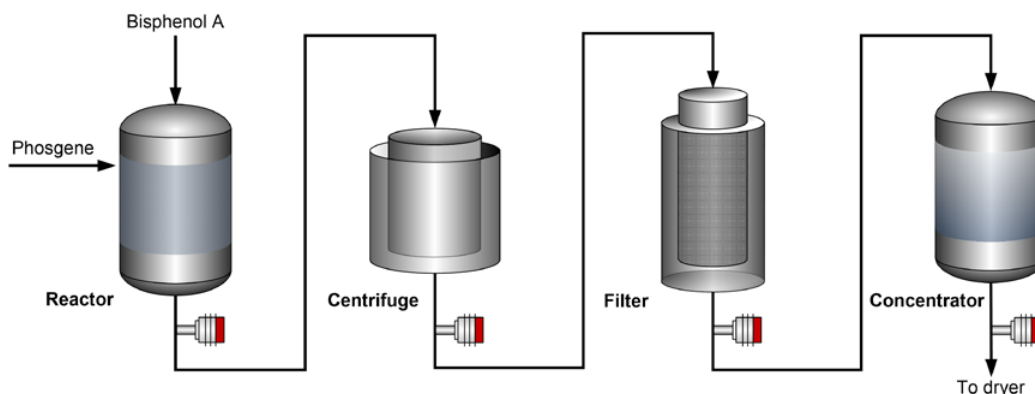
### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to measure the final product concentration. It is installed in the long pipe, after the styrene finishing column. The process temperature directly after the

column is about 80-90 °C (176-194 °F). It decreases to ambient temperatures of 5-35 °C (41-95 °F), according to seasonal fluctuations, when it is ready for storage. The measured concentration range is 95-96 %. Appropriate equipment hazardous and intrinsic safety approvals are available for hazardous area installations.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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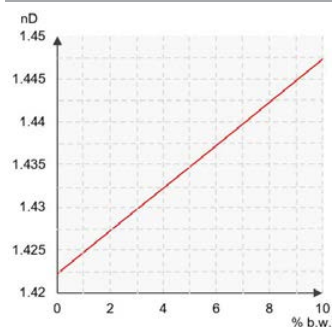


## POLYCARBONATE

### Typical end products

Bottles, food and drink containers, windows, computer and TV housings, furnishing etc.

Chemical curve: Lexan R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Polycarbonates are a group of thermoplastics, which are characterized by their toughness, high softening point and clarity.

The most common type of polycarbonate plastic is made by synthesizing bisphenol A (BPA) and phosgene (carbonyl dichloride,  $\text{COCl}_2$ ). This polycarbonate is a very durable material. Typical end products are found in bottles, windows and in the electronics industry.

The top 5 polycarbonate producing countries are USA, Germany, China, South Korea and Japan.


### Application

LEXAN is the tradename used by GE Plastics for their polycarbonate. The basis of the manufacturing process is the reaction of bisphenol A and phosgene in a methyl chloride solvent. After reaction, the polymer solution is centrifuged and filtered. Finally, the polymer solution is concentrated and dried.

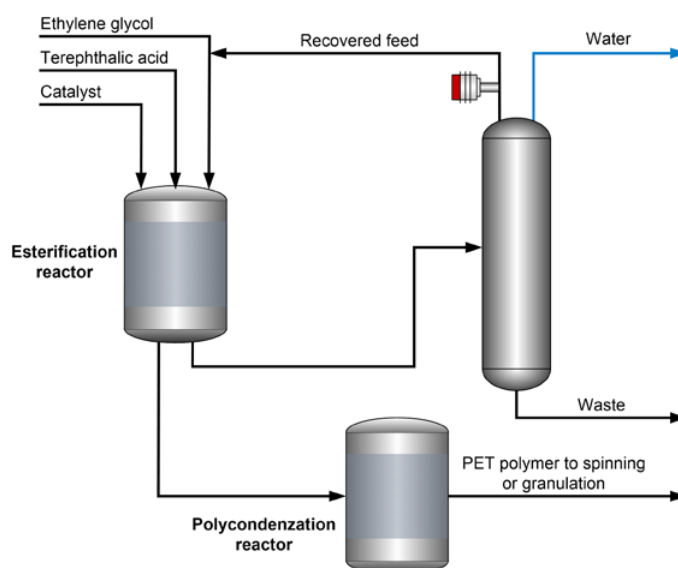
### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to monitor the concentration of resin in solvent to maintain precise control of the manufacturing process.

Typical measurement range is 5-30 % polymer in solvent, and the normal process temperature is about 40 °C (104 °F). Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.



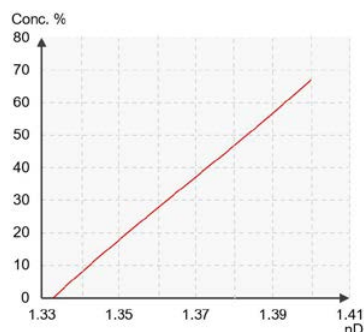


## ETHYLENE GLYCOL (CH<sub>2</sub>OH)<sub>2</sub>

### Typical end products

Synthetic fibres, thermoforming applications, engineering resins, PET bottles.

Chemical curve: Ethylene Glycol R.I. per Conc% b.w. at Ref. Temp. of 20 °C



### Introduction

Polyethylene terephthalate (PET) plastic is used to produce fibers and yarn, engineering plastics, photo and packing film, beverage and food containers. The majority of the world's PET production is for synthetic fibers (more than 60 %) with bottle production accounting for around 30 % of global demand.

In textile applications, PET is generally referred to as simply *polyester*, while *PET* is most often used to refer to packaging applications.

### Application

The monomers Terephthalic Acid (TPA) and Ethylene Glycol (EG) are reacted in the presence of a catalyst to form PET plastic.

However, all the EG does not react. The excess EG, along with other minor reaction products, such as Propylene Glycol (PG) and Triethylene Glycol (TEG), are later recovered and recycled to the fresh reactor feed.


It is important to measure reliably the concentration of EG in the recycling line in order to maintain the reactor feed at the desired level. The concentration of the EG feed has an important effect on the quality and opacity of the polymer product.

### Instrumentation and installation

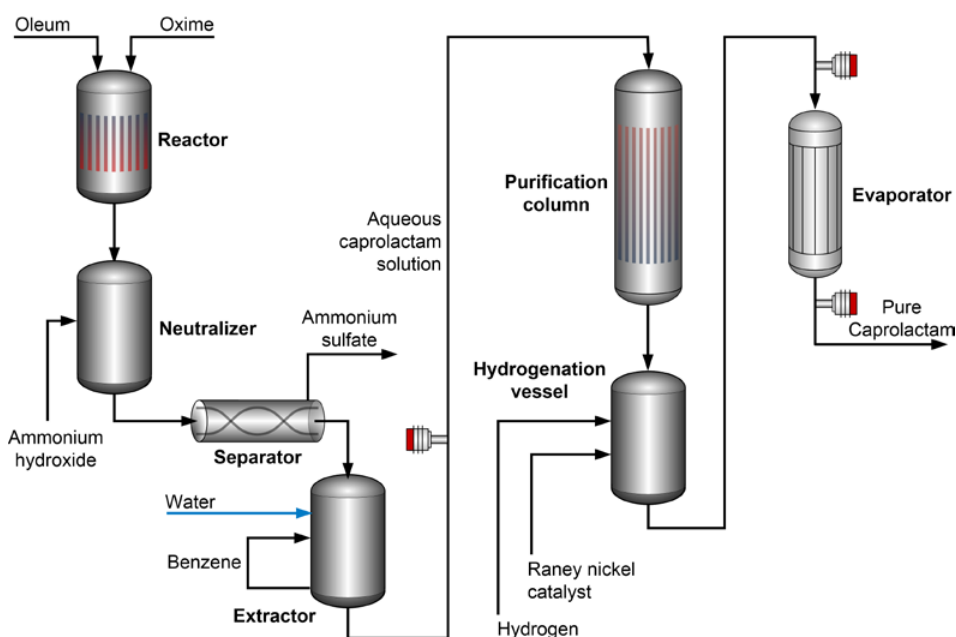
Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to measure the concentration of ethylene glycol in the recycle stream. The measurement taken by the refractometer helps adjust the feed ratio of fresh EG to ensure the correct quantity is fed to the reactor.

Typical measurement range is from 90 % to 100 %. The process temperature in the recycle stream ranges from 20 °C (68 °F) to 30 °C (86 °F).

Automatic prism wash with steam is recommended in this application. Hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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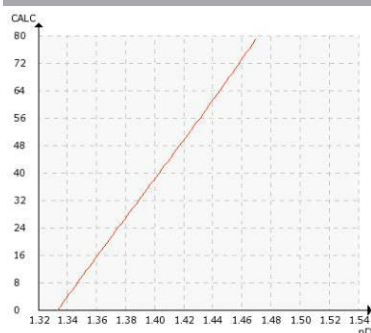


## CAPROLACTAM $C_6H_{11}NO$ , CYCLOHEXANONE $(CH_2)_5CO$

### Typical end products

Nylon-6, Cyclohexanone.

Chemical curve: Caprolactam R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Caprolactam ( $C_6H_{11}NO$ ) is the raw material for Nylon-6 plastics and fiber engineering.

Caprolactam is a chemical compound consisting of carbon, nitrogen, oxygen and hydrogen. It is made by using either cyclohexane or phenol. When caprolactam is at temperatures above its melting point, it becomes a colorless liquid.

Cyclohexanone  $(CH_2)_5CO$ , an intermediate of caprolactam, is an organic ketone and has the appearance of clear water.

### Application


Conventional caprolactam technology is based on the key intermediate cyclohexanone, which is usually produced by the oxidation of cyclohexane, but can also be made from phenol or toluene.

Separately, hydroxylamine sulfate is manufactured by the oxidation of ammonia to nitrous oxide. This is followed by hydrogenation in the presence of sulfuric acid. Then, the hydroxylamine sulfate is reacted with the cyclohexanone to produce cyclohexanone oxime. This is followed by a *Beckmann rearrangement*, using oleum to yield caprolactam.

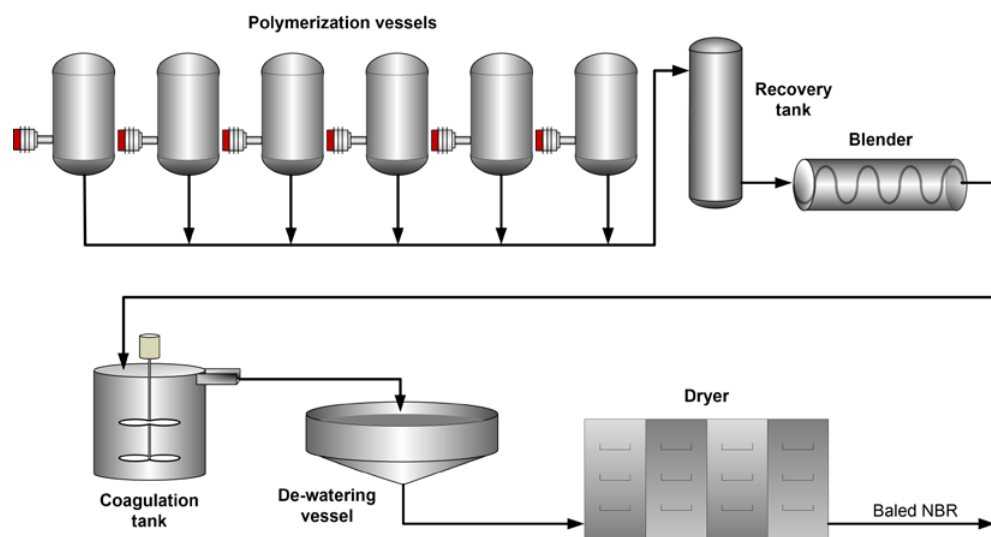
### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to measure the concentration of aqueous caprolactam solution after the initial extraction to control and maintain high extraction efficiency.

The refractometer is also used for evaporation process control. The refractometer is mounted in the outlet of the evaporator. It provides a signal to a controller to regulate the concentration value by varying the inlet flow through the evaporators. If the concentration value increases, the inlet regulating valve increases the product flow rate through the evaporators, thereby bringing the concentration back to the required value.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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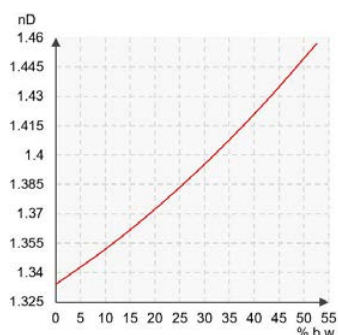


## NITRILE BUTADIENE RUBBER (NBR), SYNTHETIC LATEX

### Typical end products

Synthetic latex, synthetic rubber and automotive products, roll covers, hydraulic hoses, conveyor belts, oil field packers, seals.

Chemical curve: Nitrile butadiene R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Nitrile Butadiene Rubber (NBR) is considered to be the keystone for industrial and automotive rubber products, such as synthetic latex. Actually, NBR is a complex family of unsaturated acrylonitrile and butadiene copolymers. By selecting an elastomer with the appropriate acrylonitrile content in balance with other properties, the rubber compounder can use NBR for a wide variety of applications requiring oil, fuel and chemical resistance. The uses for NBR in the automotive industry include fuel and oil hoses, seals and grommets, and water handling applications.

### Application

NBR is produced in an emulsion polymerization system. The water, emulsifier/soap, monomers (butadiene and acrylonitrile), radical generating


activator, and other ingredients are introduced into the polymerization vessels. The emulsion process yields a polymer latex, which is coagulated using various materials (e.g. calcium chloride and aluminum sulfate) to form crumb rubber, which is dried and compressed into bales. Some specialty products are packaged in the crumb form.

NBR producers vary polymerization temperatures to make *hot* and *cold* polymers. Acrylonitrile (ACN) and butadiene (BD) ratios are varied for specific oil and fuel resistance and low temperature requirements. Specialty NBR polymers containing a third monomer (e.g. divinyl benzene and methacrylic acid) are also offered. Some NBR elastomers are hydrogenated to reduce the chemical reactivity of the polymer chain, significantly improving heat resistance. Each modification contributes to uniquely different properties.

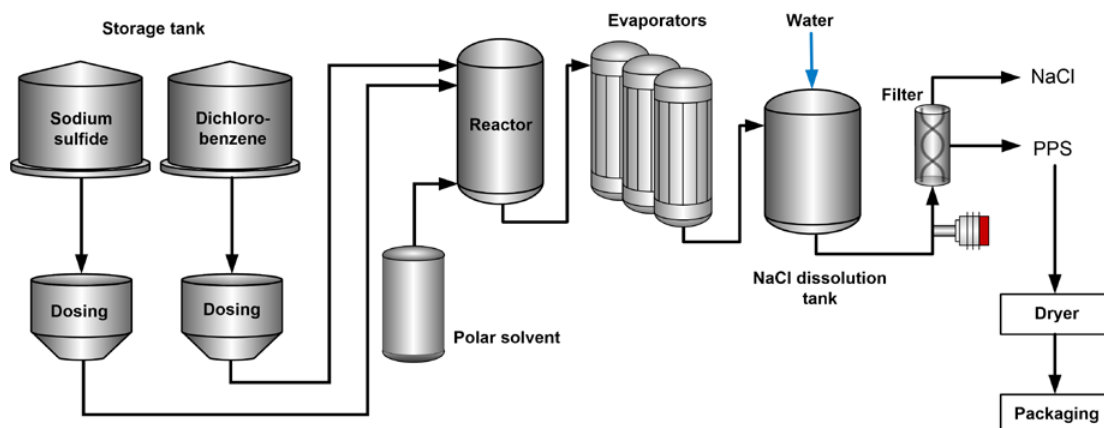
### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is installed in the polymerization vessel to accurately and reliably determine the degree, and the end-point of the polymerization reaction. The refractometer output signal indicates the degree of polymerization (see also Application Note *Control of Polymerization*). The information is based on the conversion rate from monomer to polymer. This is determined by the relationship between conversion and the solids content (%) of the components involved. Each individual polymerization vessel requires the installation of a refractometer to accurately monitor the conversion rate.

Appropriate equipment with hazardous and intrinsic safety approvals are available when required. Automatic prism wash with high pressure water is recommended in this application.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	<b>Prism wash with high pressure water.</b> The components of a high-pressure water system are a sensor with integral water nozzle mounted at the sensor head, a high-pressure pump together with a power relay unit and an indicating transmitter equipped with relays.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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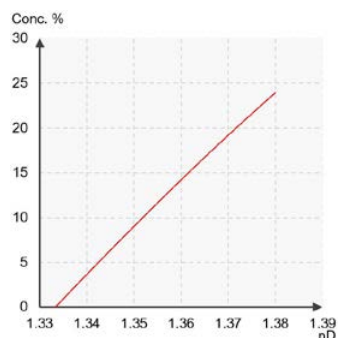


## SODIUM CHLORIDE NaCl

### Typical end products

Electrical appliance components, automotive and industrial applications, corrosion preventive coatings, filter and filter bags, etc.

Chemical curve: Sodium Chloride, NaCl. per Conc% b.w. at Ref. Temp. of 20 °C



### Introduction

Polyphenylene sulfide (PPS) resin, a form of engineering plastic, is comprised of about 30 % sulfur and about 70 % benzene by weight. PPS resin has captured wide attention as a material for automobile and electronic parts due to its outstanding chemical resistance, high-temperature stability, good dimensional stability, inherent flame retardance and good electrical properties.

### Application

Polyphenylene sulfide (PPS) resin is made from sodium sulfide and dichlorobenzene in a polar solvent. The process medium is a suspension containing PPS and sodium chloride. The suspension goes through evaporation to separate the volatile substances.

The ionic content interferes in electrical applications and must be removed by repeated washing. If chloride and sodium from raw materials remain in the resin,

they can lead to the corrosion of molds and functional deterioration of metal parts. Moreover, chlorine has a potentially harmful environmental impact if burned. That is why it is important to remove NaCl in order to produce chlorine- and sodium-free PPS resin.

The powder is then dried and packed for further processing and used in various electrical, automotive and other industrial applications. Production is either by batch or continuous process.


### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP measures the NaCl concentration at the outlet line of the dissolution tank. NaCl is dissolved in the solution and the PPS resin remains undissolved in the mixture. The refractometer can monitor and control the NaCl dissolved solids concentration without being influenced by the undissolved PPS resin.

Concentration measurement range for this application is 16-25 %, and the temperature is 25-30 °C.

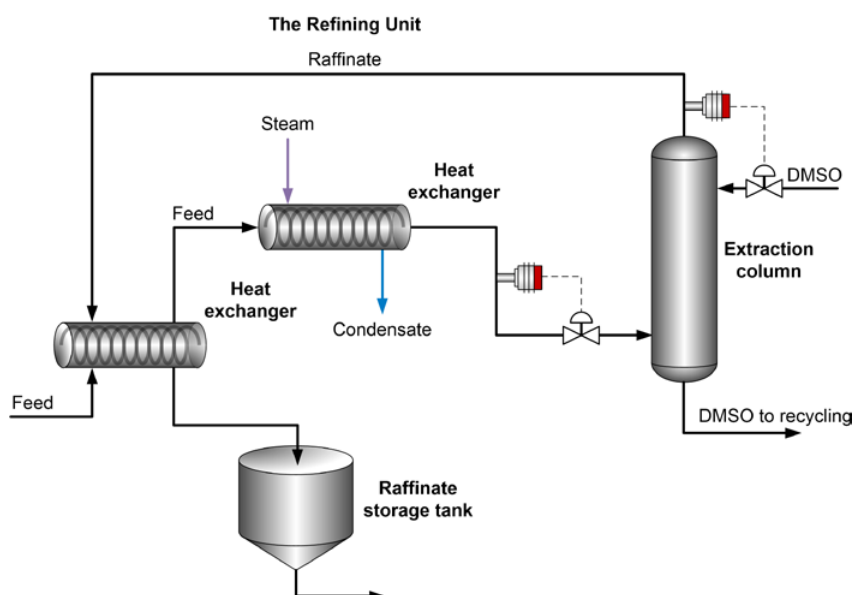
The refractometer provides precise, accurate and reliable measurement. Continuous measurement assures valuable real-time information for process control, as well as high quality chlorine- and sodium-free PPS compounds.

Automatic prism wash with steam or high-pressure water is recommended. Appropriate equipment with hazardous and intrinsic safety approvals is available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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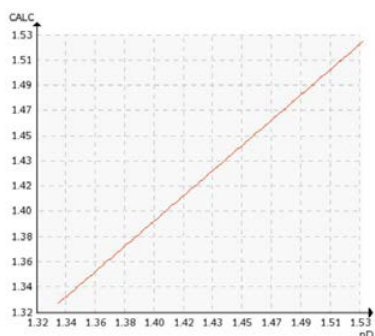


## EXTENDER OIL

### Typical end products

Green automotive tires, synthetic rubbers, rubber products.

Chemical curve: R.I. at Ref. Temp. of 20 °C



### Introduction

Extender oil is used to improve the mechanical properties and other important properties of natural and synthetic rubbers, as well as in automotive tire production. The raw material is the Residual Aromatic Extract (RAE) and Distillate Aromatic Extract (DAE) of heavy oil fractions, which are waste products from lube oils refining (see also Application Note *Lube Oil Refining Process*). Extender oils have high-levels of Polycyclic Aromatic Hydrocarbons (PAHs), which have a carcinogenic effect on human health. Automotive tires release extender oil with PAHs to the environment.

REGULATION (EC) No 1907/2006 of the European Parliament has imposed a prohibition on the use of

carcinogenic extender oils. Green automotive tire production uses carcinogens treatment technology aimed at producing environmentally safer tires.

### Application

The feedstock (comprised of RAE and DAE) enters the refining unit. The molecules of potentially dangerous carcinogens are removed in an extraction column through selective solvent treatment with dimethylsulfoxide (DMSO). PAHs are highly soluble in DMSO, so they are easily separated from the oil and discharged at the column bottom. The raffinate (purified oil) rises to the column top due to its lower density and is further pumped to storage.

It is essential to monitor changes in the refractive index at the intake and outlet to maintain high extraction efficiency.

### Instrumentation and installation


Vaisala K-PATENTS® Process Refractometer PR-43-GP provides real-time indication of the aromatic content of the oil stream, which strongly correlates with PAH concentration. Typical refractive index measurement range is between 1.5000 and 1.5600 at a temperature of 20 °C (68 °F).

In addition to the main process, DMSO recycling process can be monitored with the Process Refractometer PR-43-GP.

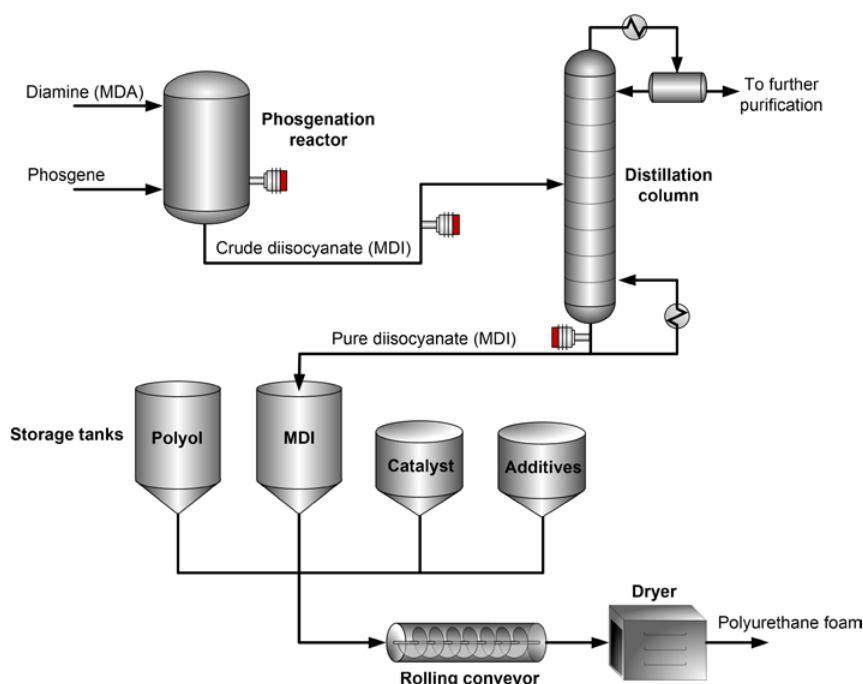
The recommended installation points are in the feed stream before extraction, and in the column's outlet, where the process temperatures are the highest (typically 90-110 °C (194-230 °F)).

The refractometer provides 4-20 mA and Ethernet output signals that can be used for real-time control of the extraction process. For instance, the signal is used to adjust either the extraction temperature, or the input streams to the column (DMSO or oil) in order to achieve the best performance.

Appropriate equipment with hazardous and intrinsic safety approvals are available when required. Stainless steel 316L SS can be used for the refractometer's wetted parts. Non-standard YAG prism is used due to a specific measurement range. Automatic prism wash with steam is necessary especially at the final product line. The wash interval is typically up to 1 minute.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	<b>Prism wash with steam.</b> The components of a steam wash system are a sensor with integral steam nozzle mounted at the sensor head, a shut-off valve for steam line and an indicating transmitter equipped with relays to drive the wash valves. The wash interval can typically be up to 1 minute.
Measurement range	Refractive Index (nD) 1.3700 – 1.6300, corresponding to 0-100 % by weight.

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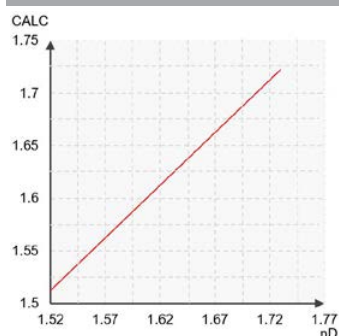


## METHYLENE DIPHENYL DIISOCYANATE (MDI), 1,6 HEXAMETHYLENE DIISOCYANATE (HDI)

### Typical end products

Polyurethane foam for different applications, for example, bedding, furniture, packaging, coatings and elastomers.

### Chemical curve: R.I. for MDI at Ref. Temp. of 25°C



### Introduction

Polyurethanes (PUs) are a class of versatile materials with great potential for use in different applications. They are used in the manufacture of many different items, such as paints, liquid coatings, elastomers, insulators, elastics, foams and integral skins.

Polyurethanes are produced by polymerization of a diisocyanate with a polyol. Because a variety of diisocyanates and a wide range of polyols can be used to produce polyurethane, a broad spectrum

of materials can be produced to meet the needs for specific applications.

### Application

The most commonly used isocyanates for the production of PU are methylene diphenyl diisocyanate (MDI) and toluene diisocyanate (TDI). If color and transparency are important, an aliphatic diisocyanate such as hexamethylene diisocyanate (HDI) is used.

The first step in the production of polyurethane is the synthesis of the raw materials. The diisocyanate is typically produced from basic raw materials via nitration, hydrogenation and phosgenation. The feedstock and initial processing steps depend on the desired isocyanate, but all go through a phosgenation step.

For instance, for the production of MDI, benzene and nitric acid are reacted to produce nitrobenzene. After a hydrogenation step, the nitrobenzene is converted to aniline. It is then condensed with formaldehyde to produce methylene diphenyl diamine (MDA), the precursor for MDI. The MDA is fed to a phosgenation reactor where it reacts with phosgene to produce the end-product MDI. MDI is further purified by several distillation steps to obtain pure and polymeric MDI (PM).

The products can be used to produce commercial liquid MDI and MDI prepolymers, or can be reacted with a polyol in the presence of a catalyst to produce the polyurethane foam.

## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP provides accurate and reliable refractive index measurement for different applications in the production of isocyanates and PU.


The refractometer can be installed directly in the polymerization reactor to monitor continuously the progress of the reaction. This helps to determine the degree of polymerization and end-point of the reaction (see also Application Note *Control of Polymerization*).

The refractometer also measures in-line the concentration of isocyanate MDI, TDI, HDI after the reaction and after the purification steps. This ensures the right concentration is always fed to the

polyurethane process. This is a crucial measurement point as a slight change in the raw materials can change the properties of the final PU foam.

The refractometer can also be used to monitor and control the concentration at other points of the process, for example, in feed catalyst lines or solvent recovery.

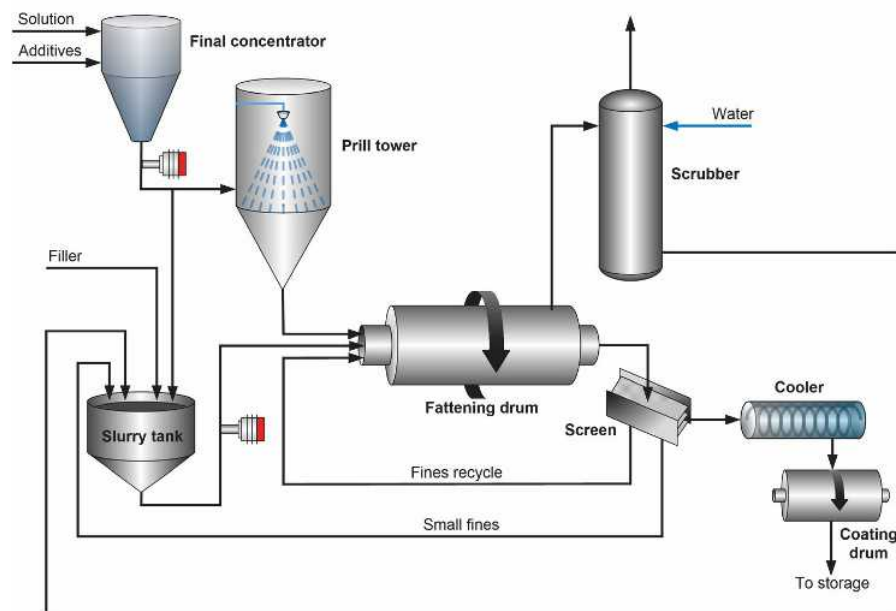
Typical measurement ranges in this application are 1.3200-1.5300 for HDI and ADI applications, and 1.5200-1.7300 MDI and PM measurements in polymerization. The temperature can be up to 200 °C (392 °F). The refractometer can be equipped to safely withstand high temperatures. Automatic prism wash may be required in this application. Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.5200 – 1.7300, corresponding to 0-100 % by weight.

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## Ammonium nitrate production process

Chemicals and Allied | Fertilizers and Explosives | 4.04.01



### Measured medium

- Ammonium nitrate,  $\text{NH}_4\text{NO}_3$

### Typical end products

- Fertilizers, explosives.

### Introduction

Ammonium nitrate is a salt, which consists of two ions: a cation, the ammonium ion ( $\text{NH}_4^+$ ) and an anion, the nitrate ion ( $\text{NO}_3^-$ ). The primary industrial use for ammonium nitrate ( $\text{NH}_4\text{NO}_3$ ) is in the explosives and fertilizers industries. Ammonium nitrate is also used for the treatment of titanium ores.

### Application

Ammonium nitrate is produced by reacting nitric acid with ammonia. The resulting solution is concentrated to 97.5-98 % in a final concentrator. From there, the concentrated ammonium nitrate

solution and quality additives are fed into a prilling tower. Some of the concentrated solution is gravity fed into a slurry tank. Small pellets of ammonium nitrate are formed in the prilling tower.

In the slurry tank, the filler is dispersed, the oversize and small fines are melted, the 97.5 % ammonium nitrate solution from the concentrator is discharged, and the moisture content of the mixture is adjusted by the addition of scrubbing liquor.

The slurry is conveyed from the tank through a hot air atomizing system to form tiny droplets. These droplets are sprayed onto

a curtain of falling granules, made up from the prilling tower product and fines separated by screening.

After passing through the fattening drum, the material is discharged to screens, where it is classified into four fractions. The graded product is then fed into the fluidized bed cooler.


The cooled ammonium nitrate is treated to prevent caking during storage. The treatment is performed in a rotary drum by spraying a liquid coating agent derivative, made of oil and amine.

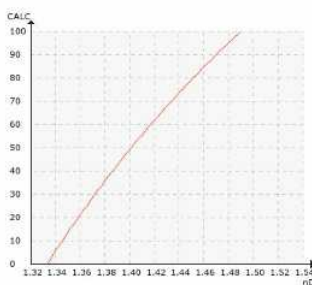
## Instrumentation and installation

Vaisala K-PATENTS<sup>®</sup> refractometer provides continuous monitoring of the ammonium nitrate process to achieve a product of high quality. The Process Refractometer PR-43-GP is installed on the concentrator and slurry tank outflows. This measurement is critical to create a uniform *prill* and to prevent the need for reprocessing. Traditional methods to calculate the concentration based on nuclear density and paper charts are confusing and time-consuming. The refractometer provides a direct measurement of ammonium nitrate concentration which can be sent to the control room through Ethernet or 4-20 mA output signals. The refractometer's signal warns the operators of changes in the process and allows for real-time adjustments.

The concentration of the outflow  $\text{NH}_4\text{NO}_3$  solution is 90-98 %, at a process temperature of 160-180 °C (320-356 °F). From the slurry tank, the  $\text{NH}_4\text{NO}_3$  solution is 90-98 % and the process temperature is 150-160 °C (302-320°F).

Typical measurement range is 60-100 %  $\text{NH}_4\text{NO}_3$  and typical accuracy is 0.2 %  $\text{NH}_4\text{NO}_3$ . Automatic prism wash with steam may be required in this application. Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.
Chemical curve	Ammonium nitrate R.I. per Conc% b.w. at Ref. Temp. of 20°C



# VAISALA

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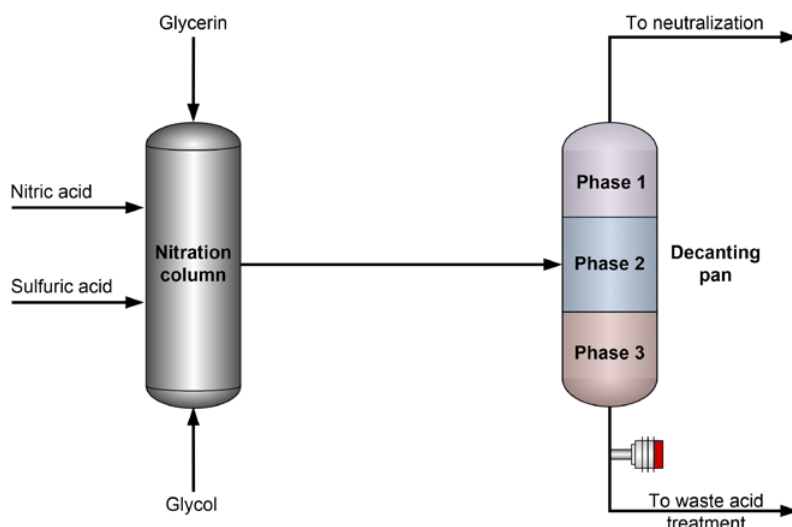
Please contact us at  
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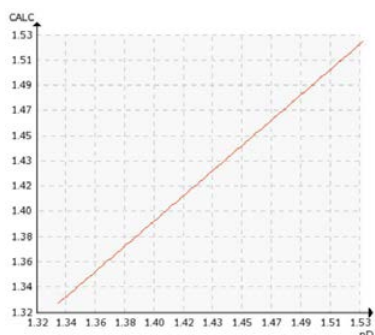


## NITROGLYCERINE, $C_3H_5N_3O_9$

### Typical end products

Explosives (dynamite and propellants), medicines.

Chemical curve: R.I. at Ref. Temp. of 20°C



## Introduction

Nitroglycerine is an oily liquid, which is prepared by treating glycerine with a mixture of nitric acid and sulfuric acids. The pure nitroglycerine is colorless, odorless and insoluble in water. It is a very powerful and dangerous explosive and should never be used in its pure form due to its sensitivity.

## Application

Nitroglycerine is manufactured by nitration of natural or synthetic glycerine. Nitration is achieved by slowly adding high purity glycol and glycerol to the mixture, having the approximate composition of 59.5 %  $H_2SO_4$ , 40 %  $HNO_3$  and 0.5 %  $H_2O$ .

After nitration the mixture of nitroglycerin and nitroglycol, with acid in the solution (phase 1) is sent to neutralization. The waste acid and a small amount


of nitroglycerine and nitroglycol (phase 3) go to waste acid treatment.

## Instrumentation and installation

Sometimes phase two (2) becomes thick and results in a *phase inversion*. This is extremely dangerous, as the probability for an explosive reaction in the waste acid treatment is very high.

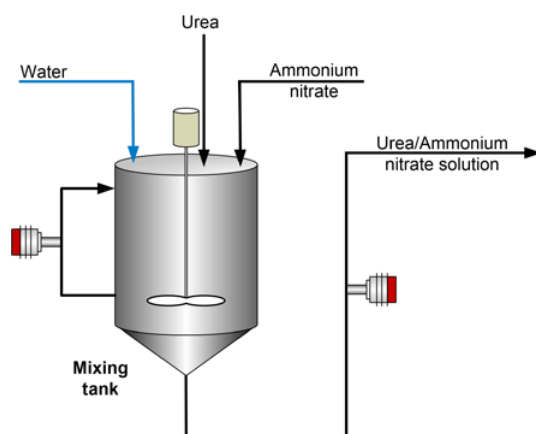
Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to ensure a safe operation by continuously monitoring the nitroglycerine and nitroglycol concentration in the waste.

Typical refractive index measurement range for nitroglycerine is 1.43 to 1.45, at a process temperature of 20-30 °C (68-86 °F). Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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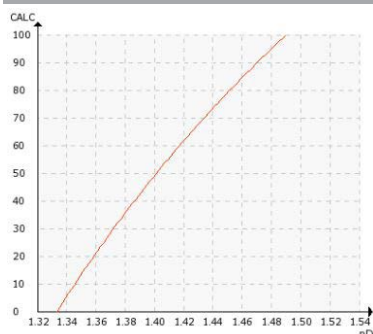


## UREA-AMMONIUM NITRATE (UAN)

### Typical end products

UAN liquid fertilizer.

Chemical curve: Ammonium nitrate R.I. per Conc% b.w. at Ref. Temp. of 20 °C



### Introduction

Ammonium nitrate (AN) and urea are used as feedstocks in the urea-ammonium nitrate (UAN) liquid fertilizers production. Typically, most UAN solutions contain 28, 30 or 32 % nitrogen but other customized concentrations (including additional nutrients) are also produced. Most of the large scale UAN production units are integrated into complexes, where either urea, ammonium nitrate or both are produced.

The concentrated UAN solution has higher nitrogen content than the standard urea. Liquid UAN is easy to transport and to distribute through pipelines. UAN solutions are manufactured in normal fertilizer plants.

### Application

Urea, ammonium nitrate and water are mixed in the final tank. A typical end product consists of 40 % ammonium nitrate and 30 % urea with water making up the remainder.

Continuous and batch type processes are used. In both processes, concentrated urea and ammonium nitrate solutions are measured, mixed and then cooled. The objective of the mixing process is to achieve the final required value of total nitrogen content (28, 30 or 32 %).

In the continuous process, the ingredients of the UAN solution are continuously fed to and mixed in a series of appropriately sized static mixers. Raw material flow, as well as finished product flow, pH and concentration are continuously measured and adjusted. The finished product is cooled and transferred to a storage tank for distribution.


In the batch process, the raw materials are sequentially fed to a mixing vessel fitted with an agitator. The dissolving rate of the solid raw materials is measured and can be enhanced by recirculation and heat exchange, if required.

### Instrumentation and installation

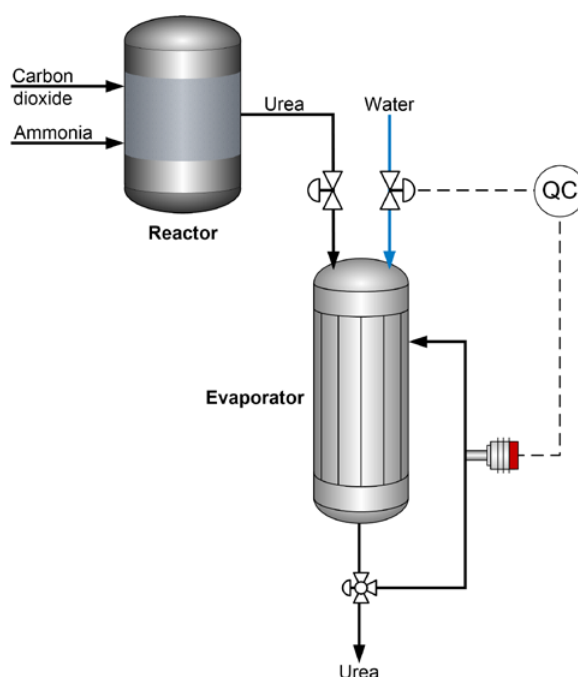
Vaisala K-PATENTS® Process Refractometer PR-43-GP is installed to monitor the mixing of urea and ammonium nitrate.

A second installation point is for the finished product during transfer to the end storage tank. The refractive index (R.I.) value is used to monitor the addition of urea and ammonium nitrate. R.I. is an essential quality parameter for the finished product concentration. Typical R.I. range is 1.425-1.455 at the temperature of 20 °C (68 °F).

It is possible to have an output of total nitrogen content of the solution by combining the measurements from the refractometer and density device.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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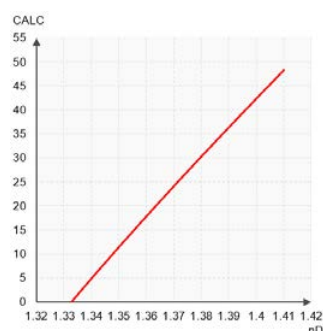


## UREA, $\text{NH}_2\text{CONH}_2$

### Typical end products

Fertilizers, non-protein feed supplement for ruminants, urea-formaldehyde resins, plastics, adhesives, coatings, textile agents, ion-exchange resins.

Chemical curve: Urea R.I. per Conc% b.w. at Ref. Temp. of 20 °C



### Introduction

Urea,  $\text{NH}_2\text{CONH}_2$ , is a colorless crystal, which dissolves in water. It is a weak alkaline, which forms salts in combination with strong acids. Urea is largely used as a fertilizer and as a non-protein feed supplement for ruminants. It is also used in the manufacture of urea-formaldehyde resins, plastics, adhesives, coatings, textile agents and ion-exchange resins.

### Application


Urea can be produced by numerous methods but mainly it is commercially made by reacting carbon dioxide with ammonia at 200 °C (392 °F). After reacting, urea is evaporated and processed by prilling or granulating to produce a solid end product.

### Instrumentation and installation

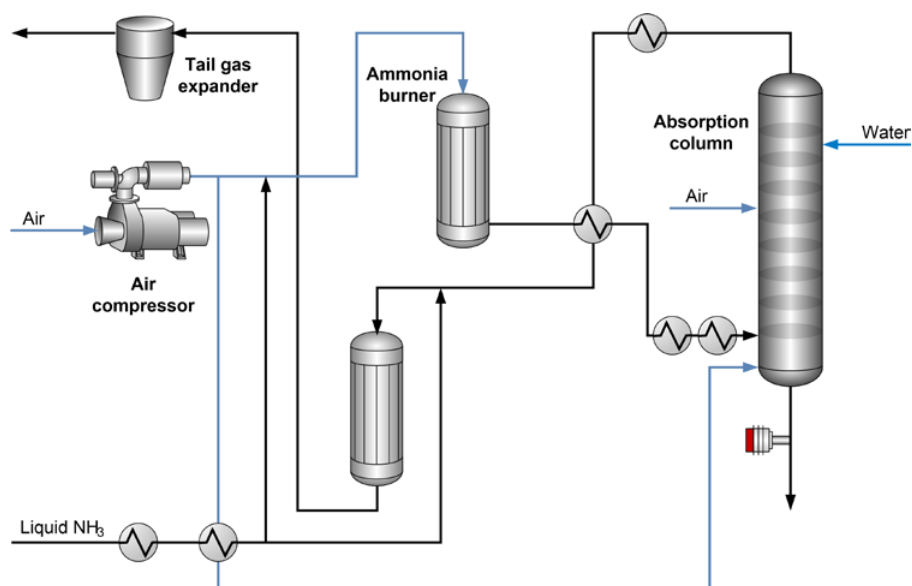
Vaisala K-PATENTS® Process Refractometer PR-43-GP is used for quality control. It monitors the concentration of produced urea after the reactor. Typical measurement range is 65-75 % and the normal process temperature is about 80 °C (176 °F).

The Process Refractometer PR-43-GP is also used to measure the concentration of dissolved urea in water in the manufacture of medicinal insulin.

The refractometer is installed in the recirculation pipe. It is used to control and adjust the quality of the urea-to-water concentration. Typical refractive index range in this application is 1.380-1.395, at a process temperature of around 4 °C (39 °F).

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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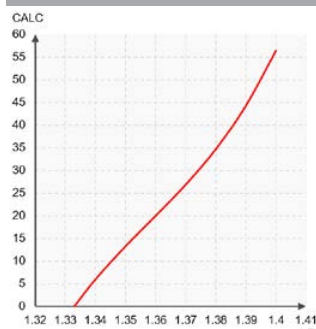


## NITRIC ACID, HNO<sub>3</sub>

### Typical end products

Nitric acid of approximately 60 % concentration by weight.

Chemical curve: Nitric acid R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Nitric acid (HNO<sub>3</sub>), also known as *aqua fortis* or *spirit of nitre*, is a highly toxic and corrosive mineral acid.

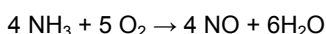
Approximately 70 % of all nitric acid produced is used for the production of ammonium nitrate, which is used in fertilizers. Nitric acid is also a key component in the manufacture of adipic acid and terephthalic acid. Other applications include explosives, mine leaching and stainless steel pickling.

### Application

Nitric acid production can be composed of one or two processes depending on the required final concentration.

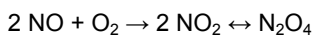
A large portion of the nitric acid manufactured in the world is via the high-temperature catalytic oxidation of ammonia. This process consists of three main steps: ammonia oxidation, nitric oxide oxidation and absorption. Processing can be achieved through single or multiple pressures.

A mixture, composed of a 1:9 ratio of ammonia and air, is oxidized at a temperature close to 760 °C (1400 °F) in a catalytic converter according to the reaction:



The most common catalyst is composed of about 90 % platinum and 10 % rhodium (by weight). The catalyst is formed in the wire gauze and inserted into the converter. The exothermic reaction proceeds to a nitric oxide yield of about 93-98 %.

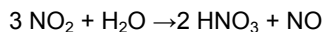
The nitric oxide is cooled (and water condensed) to a temperature of 40 °C (104 °F) or less, at a pressure up to 7.8 bar (115 psi). The nitric oxide reacts (non-catalytically) with oxygen to form nitrogen dioxide and nitrogen tetroxide via the reaction:



This reaction is highly dependent on both temperature and pressure. Low temperatures and high pressures favor the production of nitrogen dioxide (preferred) over nitrogen tetroxide.

After cooling down, the nitrogen dioxide/nitrogen tetroxide mixture enters an absorption column. The gaseous mixture is introduced to the bottom of the



column, while liquid dinitrogen tetroxide and de-ionized water enter from the top. Liquids flow countercurrent to the gases in the system, while the oxidation takes place between the trays while absorption takes place on the trays (usually bubble cap trays). The reaction in the absorption column proceeds by:



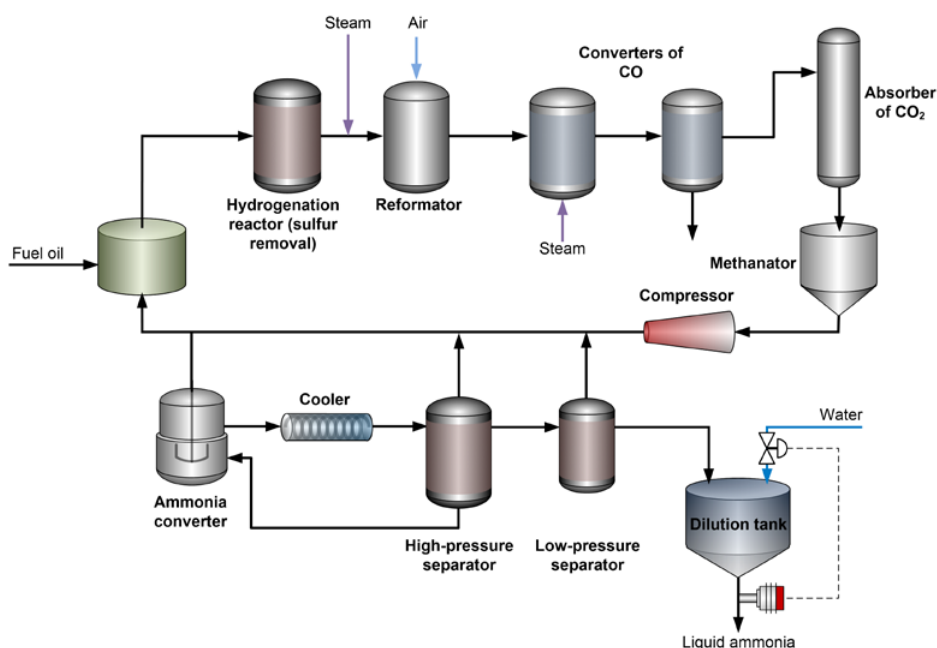
A second air stream entering the column further oxidizes the NO and removes the NO<sub>2</sub> from the product acid. Acid concentrations leaving the absorption tower are typically between 55-65 % by weight.

## Instrumentation and installation

Vaisala K-PATENTS® Saunders Body Refractometer PR-23-W is mounted in the outlet pipe of the absorber to control the absorption process and to get a stable nitric acid concentration value. The Teflon Body Refractometer PR-23-M should be installed in a by-pass.

Instrumentation	Description
	Teflon Body Refractometer PR-23-M. A compact refractometer for chemically aggressive solutions and ultra-pure fine chemical processes. Connected to the process by a G1/2" female or a 1/2" NPT process connection. It has a built-in flow cell designed to keep all metal and other easily corroding parts from coming into contact with the process liquid.
	Saunders Body Refractometer PR-23-W. A heavy-duty refractometer for chemically aggressive liquids in large-scale production and in large pipe sizes (diameter 50, 80 or 100mm/2", 3" or 4"). The Saunders body material is graphite cast iron, which provides a solid mechanical base. A PFA-lining ensures the chemical resistance.
Measurement range	50-65 % Nitric acid. Typical accuracy +/- 0,75 % by weight.

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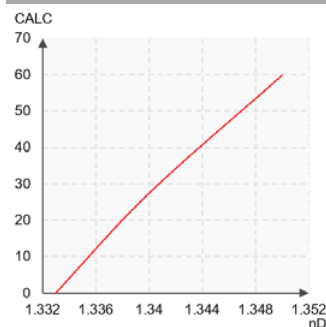


## LIQUID AMMONIA NH<sub>3</sub>, AMMONIUM HYDROXIDE NH<sub>4</sub>OH

### Typical end products

Liquid ammonia, ammonium hydroxide.

Chemical curve: Ammonium hydroxide R.I. per Conc. % b.w. at Ref. Temp. of 20 °C



### Introduction

Ammonia (NH<sub>3</sub>) is a colorless gas, which can easily be dissolved in water. The concentration of ammonia in water is usually 25 %. Ammonium hydroxide (NH<sub>4</sub>OH) is formed during the liquefaction.

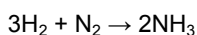
### Application

Ammonia is mainly produced via the *Haber process*. The raw material is natural gas or fuel oil (naphtha), which produces hydrogen (H<sub>2</sub>) for the steam reforming process. Nitrogen is extracted from air.

The process is divided into five steps. Sulfur removal, steam reformation, CO-conversion, purifying and ammonia synthesis.

After sulfur removal, steam is added to the raw gas to produce carbon monoxide (CO). Next, it is reacted with water in the CO-converter to produce H<sub>2</sub> and carbon dioxide (CO<sub>2</sub>). The CO<sub>2</sub> is then removed either by absorption or adsorption.

The purified hydrogen/nitrogen gas enters the ammonia synthesis stage, where the following reaction occurs at 300 bars (4350 psi) and 500 °C (932 °F):



After the ammonia reactor, the gas is cooled and liquefied in a high-pressure separator (HPS). The impurities in the dissolved ammonia liquid evaporate in a low-pressure separator (LPS).


### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP measures the concentration of liquid ammonia after dilution. Final concentration of ammonia is typically around 25 % with measurement taken at ambient temperature.

The refractometer can be easily calibrated either to read NH<sub>3</sub> (ammonia-in-water) or NH<sub>4</sub>OH (ammonium hydroxide) in water.

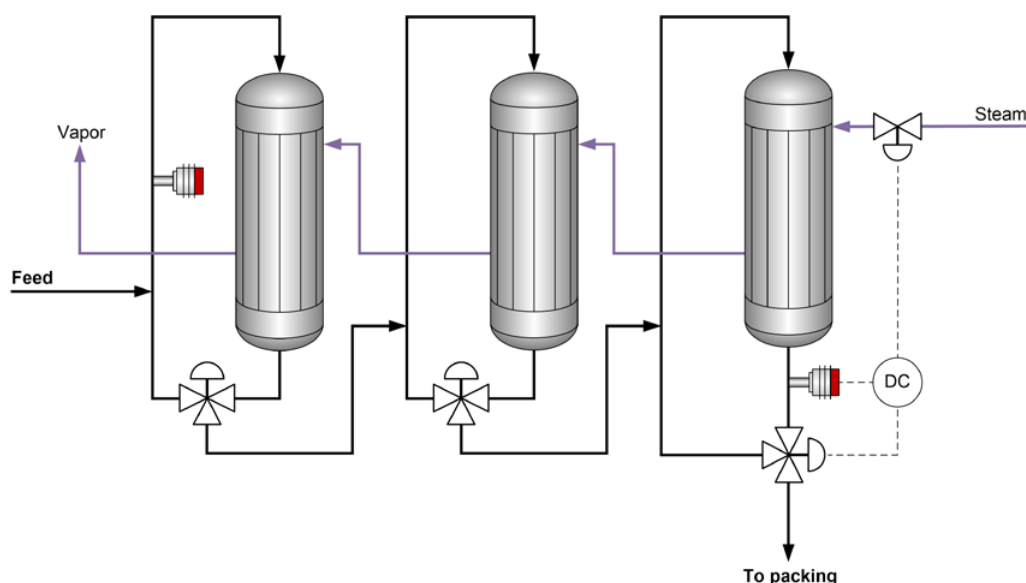
The refractometer provides Ethernet and 4-20 mA output signals that can be used for automatic control of the dilution process. The signal from the refractometer

controls the water feed valve, thus ensuring a correct concentration level in the final product. In certain cases, steam prism wash is recommended.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	<b>Prism wash with steam (optional).</b> The components of a steam wash system are a sensor with integral steam nozzle mounted at the sensor head, a shut-off valve for steam line and an indicating transmitter equipped with relays to drive the wash valves.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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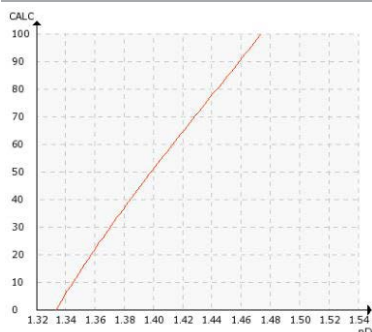


## GLYCEROL (GLYCERIN)

### Typical end products

Pharmaceuticals, humectants, solvent, sweetener, emulsifier, nitroglycerin, glycerol soap, moisturizer.

Chemical curve: Glycerol R.I. per Conc b.w. at Ref. Temp. of 20 °C



Splitting or hydrolysis of oil, under pressure and at high temperature, produces fatty acids and *sweetwater*. The *sweetwater* contains 10–20 % glycerol. Transesterification of oil with methanol, in the presence of a catalyst, yields methyl esters and glycerol. Since the process does not use water, the glycerol concentration is higher. Saponification of an oil/fat with caustic soda forms soap and soap lye. The soap lye formed contains 4-20 % glycerol and is also known as *sweetwater* or *glycerin*.

Sweetwater or glycerin obtained as a co-product from the above three processes contains impurities and must undergo further purification and concentration, for example, by evaporation.

### Introduction

Glycerol (Glycerin) is a clear, nearly colorless and viscous liquid with a very sweet taste. It occurs in combination with various fatty acids (glycerides) in all animal and vegetable fats and oils.

Glycerin is produced organically by a number of different methods and by various synthesis processes.


### Application

Natural glycerin is obtained primarily as a co-product from the production of fatty acids and fatty esters, or soap from oils and fats.

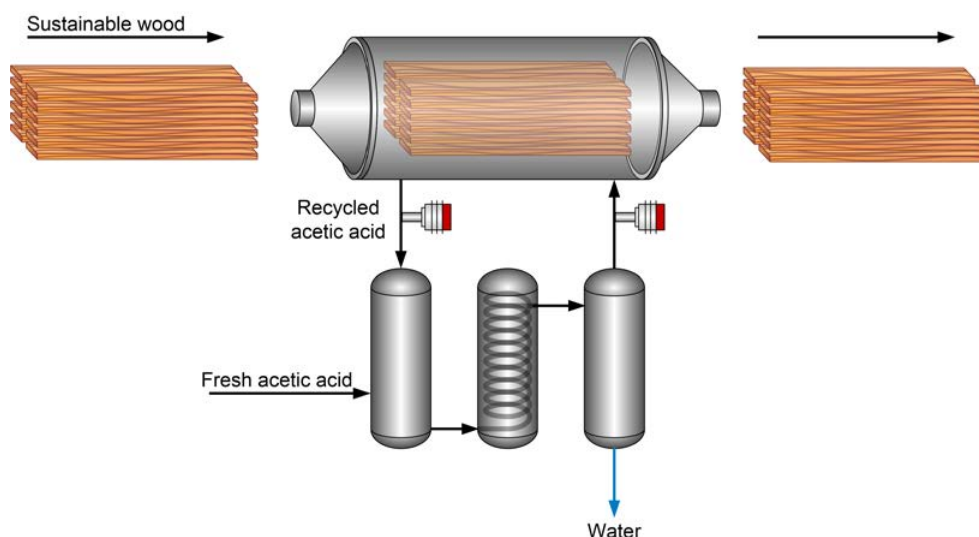
### Instrumentation and installation

Vaisala K-PATENTS® Compact Refractometer PR-43-GC is used to measure the glycerin concentration in evaporation. Automatic control of the evaporator is possible by using the refractometer's output signal.

The typical measurement range is 0-20 % glycerin and the process temperature is about 50 °C (122°F) before evaporation. After evaporation the typical measurement range is 80-100 % glycerin and the process temperature is about 70 °C (158 °F).

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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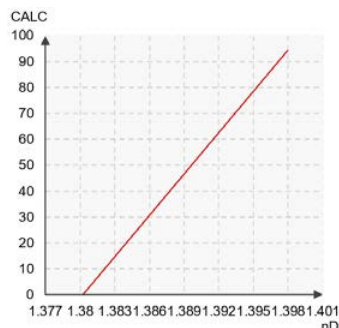


## ACETIC ANHYDRIDE, (CH<sub>3</sub>CO)<sub>2</sub>O

### Typical end products

Acetylated (durable) wood timber for window frames, doors, shutters, cladding, siding, facades, decking and garden furniture.

Chemical curve: Anhydride in Acetic acid R.I.per Conc. % b.w. at Ref. Temp. of 20 °C



Free hydroxyl groups absorb and release water, according to changes in the climatic conditions, which the wood is exposed to. This is the main reason why wood swells and shrinks. It is also believed that the digestion of wood by enzymes initiates at the free hydroxyl sites, which is one of the principal reasons wood is prone to decay.

Acetylation changes the free hydroxyls within the wood into acetyl groups. This is done by reacting the wood with acetic anhydride, which comes from acetic acid (CH<sub>3</sub>COOH) known as *vinegar* when in its dilute form:



### Introduction

Acetylated wood timber is rapid growth wood (generally the cheapest wood available), which has been treated to have better dimensional stability, durability, UV resistance and paint retention.

### Application

The treatment technology is based on the acetylation process. Acetic anhydride (CH<sub>3</sub>CO)<sub>2</sub>O is used as a reagent for this process.

The physical properties of any material are determined by its chemical structure. Wood contains an abundance of chemical groups called *free hydroxyls* (represented as OH).


When a free hydroxyl group is transformed to an acetyl group, the wood's ability to absorb water is greatly reduced, rendering the wood more dimensionally stable and, because it is no longer digestible, extremely durable.

### Instrumentation and installation

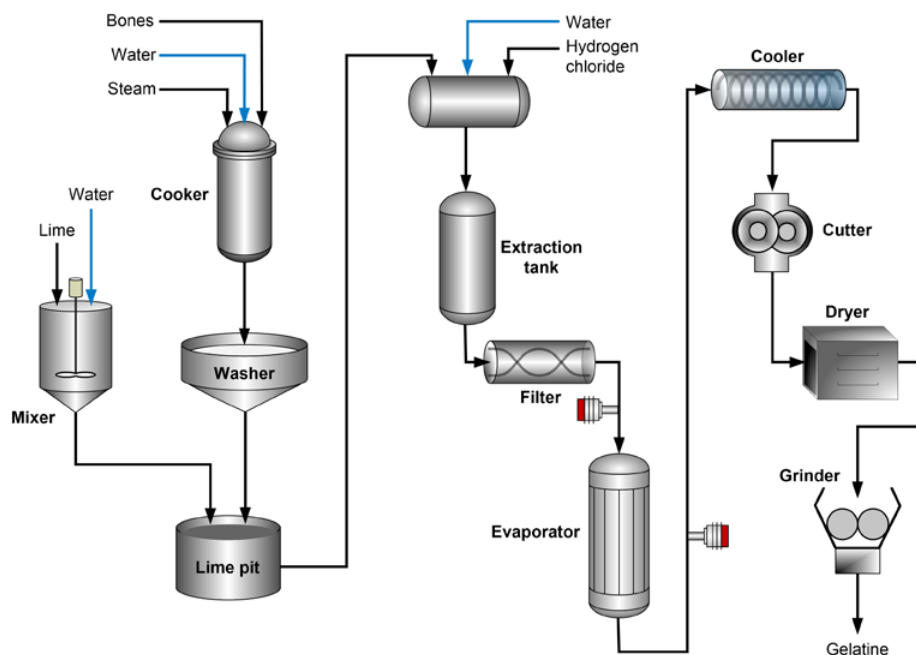
Vaisala K-PATENTS® Process Refractometer PR-43-GC is installed in the reactor feed to measure and maintain the correct concentration of acetic anhydride in acetic acid.

The second installation point is in the recycling plant's distillation columns, where the spent chemical is recycled into the process. The refractometer assists in reducing the operating costs of distillation units via real-time process control and optimization.

The refractometer is uninfluenced by bubbles or wood chips. Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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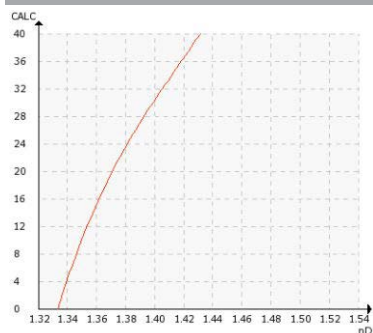


## GELATINE

### Typical end products

Gelling agent in food, pharmaceuticals, photography and cosmetics.

Chemical curve: Gelatine R.I. per Conc. % b.w. at Ref. Temp. of 20° C



### Introduction

Collagen is the main organic component of bone and skin in mammals. Acid and liming process production methods are used to produce gelatine, which is a purified protein derived from the selective hydrolysis of collagen.

Gelatine is an organic, colloidal protein substance, whose principal value depends on its coagulative, protective and adhesive powers. Gelatines swell in cold water but are insoluble in it. They dissolve in hot water to produce very viscous solutions.

Gelatines are manufactured from bones and hides, and are used in different industries: photographic, pharmaceutical and food industries.

### Application

The acid process method consists of placing the bones or skins in a vat containing a dilute solution of acid for a determined period. The solution is then washed in cold water, which reduces the pH to approximately pH 4.



The liming process method consists of placing the bones or hides into liming pits with lime slurry. At the end of this operation, the raw material is washed to remove residual lime and the hydrolysed organic impurities.

The next stage is the most important in providing different qualities of gelatine. At this point, the gelatine extract with a concentration of around 5 % is filtered, deionised in an ion-exchanger and concentrated in multiple-effect vacuum evaporators. A 15 % solution is obtained and filtered through cellulose filter-cakes and evaporated up to 30-40 %. The concentrated solution is then sterilized, cooled and extruded to gelatine sheets. The final gelatine product is dried in a tunnel dryer.

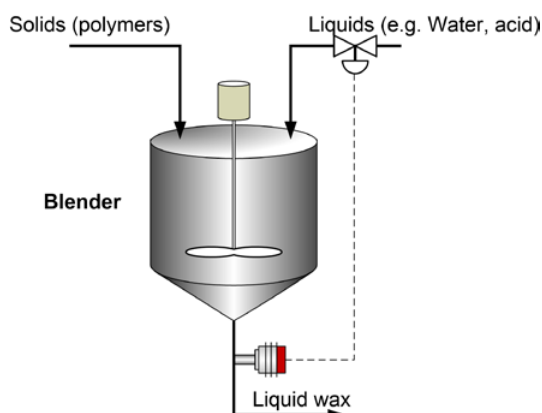
## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-G is used at the most important step, where the quality of the gelatine and the efficiency of subsequent operations are defined. The refractometer is installed between filtration and the evaporation plant, directly in the pipeline after the circulation pump.

The typical measurement ranges for gelatine production are 0-15 % and 0-40 % before and after evaporation, respectively. The process temperature of about 60 °C (140 °F). Steam cleaning is recommended for concentrations above 5 %.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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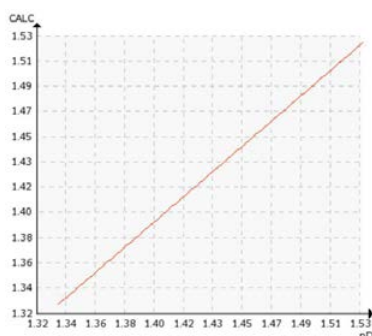


## DETERGENTS

### Typical end products

Floor wax, furniture wax, household detergents.

### Chemical curve: R.I. at a Temp. of 20 °C



## Introduction

Detergents are synthetic and organic cleaning agents, which are liquid-, water- or oil-soluble. Detergents have a wetting and emulsifying properties.

Floor waxes, furniture waxes, deodorants and shampoos are typical household detergents, which are usually manufactured with a batch process.


## Application

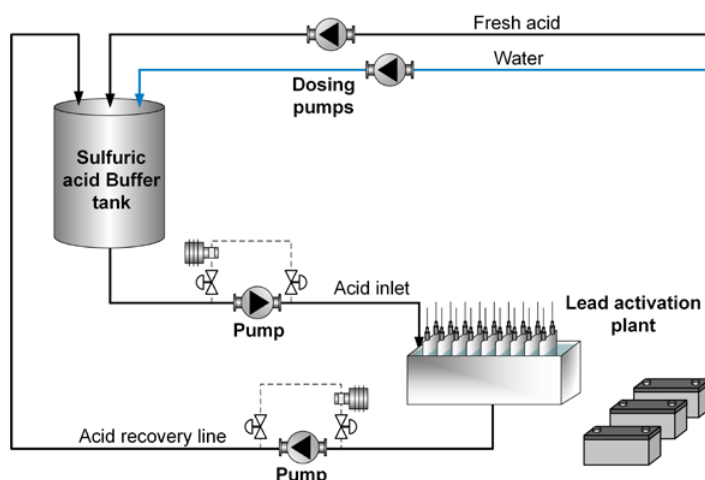
When liquid wax is produced, it consists of two major components. These components are solids or polymers which give the wax a coating effect, and volatile carriers, which are the liquids that keep the polymers in the solution. The volatile carriers will evaporate after use. The ratio between these two components must be closely controlled to obtain a consistent product quality.

## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GC is installed in the outlet pipe after blending, or directly in the vessel. The refractometer measures continuously the percent of dissolved solids in the liquid wax product.

The refractometer provides Ethernet and 4-20 mA output signals that can be used for automatic control of the blending operation. This ensures the target solid levels is always achieved. Typical concentration range in floor wax is 10-30 % solids (corresponding to a refractive index of 1.33-1.40), and the process temperature is about 27 °C (81°F).

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

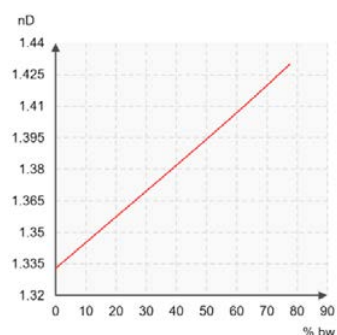


## SULFURIC ACID H<sub>2</sub>SO<sub>4</sub>

### Typical end products

Lead batteries for automobiles and electric vehicles.

Chemical curve: H<sub>2</sub>SO<sub>4</sub> R.I. per Conc.% b.w. at Ref. Temp. of 20°C



### Introduction

Battery manufacturing is the process of producing lead-acid and gel batteries commonly used for automobiles and electric vehicles that need long service periods and durability. Such vehicles are, for example, sweepers, forklifts and cleaning machines.

### Application

Sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) activates the lead elements of the lead battery resulting in the power effect. The

correct effect can be obtained only with the right acid concentration. If the sulfuric acid concentration is too high, it causes corrosion of the lead.


Evaporation of the acid may occur due to the exothermic reaction, which means that the concentration is likely to change during processing.

Fresh acid is mixed with water in a buffer tank and the solution is then pumped into a lead activation plant. After the activation plant the acid is recovered back to the buffer tank.

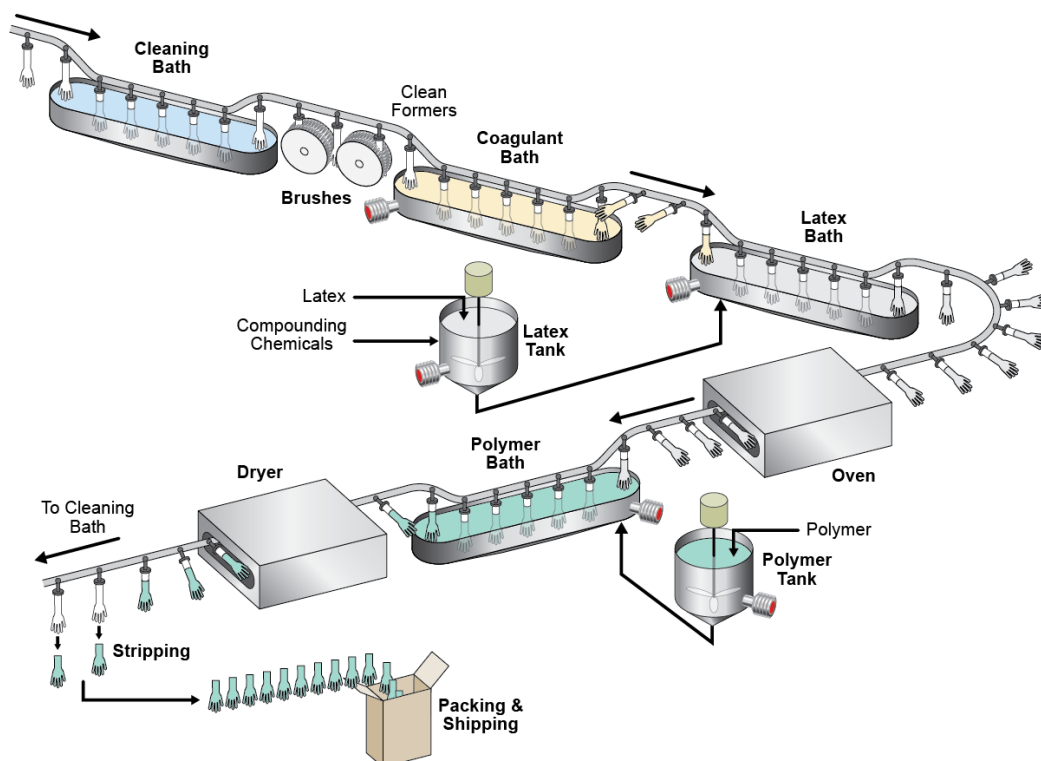
### Instrumentation and installation

Vaisala K-PATENTS® Teflon Body Refractometer PR-23-M measures in-line the sulfuric acid concentration in the acid inlet and recovery lines. A typical mounting point is in a by-pass loop across the pump inlet and outlet. Typical measurement range of sulfuric acid is 1030-1500 g/l (equivalent to 5-60 % by weight).

By measuring the sulfuric acid the correct power activation effect is reached and the potential acid concentration fluctuations are instantly revealed.

Instrumentation	Description
	Teflon Body Refractometer PR-23-M. A compact refractometer for chemically aggressive solutions and ultra-pure fine chemical processes. Connected to the process by a G1/2" female or a 1/2" NPT process connection. It has a built-in flow cell designed to keep all metal and other easily corroding parts from coming into contact with the process liquid.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.



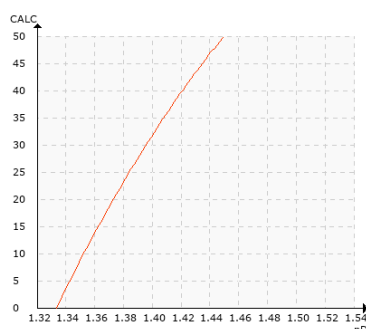


## NATURAL LATEX, NITRILE BUTADIENE RUBBER (NBR), COAGULANT SOLUTIONS, POLYMER SLURRY

### Typical end products

Gloves made of natural or synthetic rubber for different uses in the healthcare, food, chemical, pharmaceutical, automotive finishing and oil and gas industries.

Chemical curve: R.I. vs. Latex % b.w. at Ref. Temp. of 20°C



### Introduction

Rubber gloves are used for protecting skin against physical and chemical injuries as well as against infection by viruses and bacteria. Rubber gloves can be made from natural rubber (*latex*) or from synthetic rubber such as *nitrile butadiene rubber (NBR)* and *chloroprene rubber (CR)*.

Rubber gloves are for different tasks in several industries, ranging from healthcare to food processing and services, automotive to construction and chemical industries.

### Application

#### Natural rubber gloves

Natural rubber gloves are manufactured by a *dipping technology*, where ceramic or metal formers that mimic the shape of a hand are dipped in liquid latex, and then dried. This process has many variations, and production is done in batches or continuously.

The manufacturing process starts with cleaning the formers to remove any residual particle or dirt that could cause defects in the final glove product, and to ensure latex adhesion to the formers during latex dipping. In cleaning, the formers are sunk in acid and alkali baths and are brushed mechanically for thorough cleaning. After that, the formers are rinsed with clean water.

Next, the formers are dipped in a coagulation tank containing a solution of calcium nitrate or calcium carbonate. The coagulant is applied to enhance

polymer flocculation and to ensure the latex is distributed equally on the surface of the formers. The Total Dissolved Solids (TDS) concentration of the coagulation bath is maintained at its optimal level to prevent sedimentation of ingredients, as it could cause weight variation or weak spots on the gloves.

After mild drying, the formers are dipped into the latex dipping tank. This tank contains *compounded mature latex* that has been prepared by mixing latex with various compounding chemicals. The concentration of latex is carefully monitored and controlled as it has a crucial effect on the final quality, durability and product specification. The concentration of latex for dipping should be kept between 10 and 60 %.

A leaching process follows, where the formers are dipped into hot water to dissolve proteins, water-soluble residues and other chemicals from previous steps. Proper leaching of the gloves improves the strength of the latex film but not without replenishment of water to prevent build-up of extracted chemicals.

The next step is vulcanization. In this step, the material is heated at a high temperature to allow the compounding chemicals to form a cross-link with the polymer molecules. Cross-linking gives strength and elasticity to the physical properties of the rubber.

After vulcanization, the surface of the gloves might be treated to facilitate release from the hand former, to prevent gloves adhering to one another, and to ensure the gloves fit smoothly. For instance, the gloves may be dipped in a polymer coating solution for easier donning and to ensure the inner part of the glove is smoother and not sticky. Typical polymer coatings are aqueous dispersions of acrylic or polyurethane. The polymers are diluted and maintained at the required concentration (up to 20 %) to ensure high quality of the glove product.

The last part of the manufacturing process is the stripping phase where the gloves are removed from the formers. The gloves are tested for quality and packed for delivery to the customers.

## Synthetic rubber gloves

The manufacturing process for synthetic rubber gloves is similar to latex production besides that they are manufactured from synthetic materials such as nitrile, vinyl and synthetic polyisoprene.

The main difference is that in this process the synthetic material needs to be created first. The rubber raw material used is commonly a copolymer, a plastic material produced by the copolymerization of two or more different molecules or monomers. In the case

of nitrile butadiene rubber (NBR), the two monomers are butadiene and acrylonitrile. Synthetic rubber is manufactured by emulsion polymerization or polymer emulsification by means of mechanical shearing.

Once the synthetic materials are available, they go to the factory for production. The required processes are relatively the same to those in latex rubber gloves manufacturing, but the dipping line requires different operating parameters.

## Instrumentation and installation


Vaisala K-PATENTS® Process Refractometer PR-43-G is installed at different stages of the rubber gloves line to measure the concentration of dipping baths solutions. The refractometer provides continuous TDS concentration measurement to ensure operation within the specifications and to guarantee high-quality rubber gloves. The in-line measurement by the refractometer eliminates the need for sampling and long laboratory tests, providing immediate data for real-time process control.

The refractometer can be installed directly on a pipe or tank. At the compounding latex, polymer and coagulant preparation steps, the refractometer monitors the desired TDS concentration is consistently used and provides important information to keep it to the pre-determined level. The measurement is also useful for monitoring the concentration of different baths and for adjusting it only when needed, reducing the consumption of raw materials and minimizing operating costs.

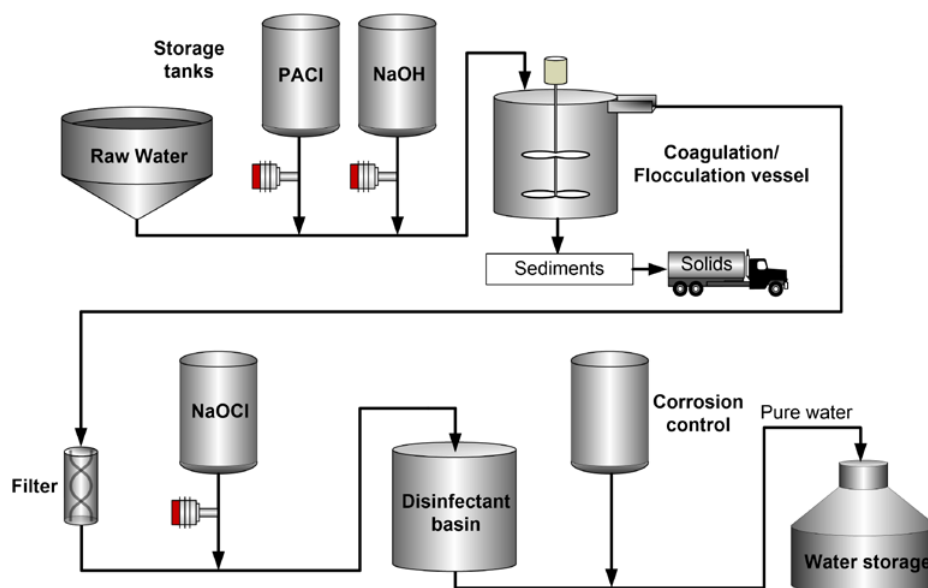
If the factory operates from synthetic rubber made at the same facilities, the refractometer can also be used in the polymerization vessel. The refractive index measurement provided by the refractometer helps to follow in real-time the progress of the reaction and to determine the degree of polymerization. The refractometer can be installed in-line in the bypass line of the reactor or directly at the bottom of the vessel through the steam jacket, eliminating the need for sampling and off-line testing (see also Application Note *Control of Polymerization and Nitrile Butadiene Rubber Production process*).

Automatic prism wash with high pressure water is required in this application for concentrations above 40 % by weight or when flow velocity is below 1.5 m/s. Hazardous and intrinsic safety approvals are available also when required.

The reliable in-line measurement by the refractometer helps rubber glove manufacturers to improve product quality, enhance productivity and reduce production costs.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic Prism Wash	<b>Prism wash with high pressure water:</b> The components of a high-pressure water system are a sensor with integral water nozzle mounted at the sensor head, a high pressure pump together with a power relay unit and an indicating transmitter equipped with relays.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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## POLYALUMINIUM CHLORIDE PACI, SODIUM HYDROXIDE NaOH, SODIUM HYPOCHLORITE NaOCl

### Typical end products

Drinking water, medical, pharmacological, chemical and industrial water applications.

### Introduction

Pure water treatment is the process of removing undesirable chemicals, biological contaminants, suspended solids and gases from raw water. Water purification aims to produce water for a specific purpose, for example, for human consumption and medical or industrial use.

Polyaluminium coagulants are finding increasing use in potable water treatment plants, particularly, for soft, colored surface waters. Polyaluminium chloride (PACI) is gradually replacing the Alum (aluminium sulphate), a commonly used coagulant in water treatment plants. Alum coagulates at a limited pH range (between 5.5 and 6.5), and often requires the addition of alkali to the raw water to achieve the optimum coagulation pH. Furthermore, the alum floc produced is particularly fragile. This is especially important if a coagulant is required to maximize color removal in a microfiltration-based water treatment process.

### Application

Water treatment by chemical precipitation is a complex process. It starts with adding flocculants, specifically, Polyaluminium Chloride (PACI) and Sodium Hydroxide (NaOH) into raw water. PACI is a synthetic polymer dissolved in water. It precipitates in big volumetric

flocs which absorb suspended pollutants in the raw water. The amount of Polyaluminium Chloride to be added to the process is defined by the turbidity of the raw water. In order to keep the flocculation process smooth, PACI concentration must be higher than 10 %. Polyaluminium Chloride is stable in the storage tank, however, it tends to crystallize after a period of time. Vaisala K-PATENTS® refractometer monitors the concentration of PACI to inform about the need for tank or pipe cleaning, thus preventing blockage caused by the PACI crystals.

NaOH regulates pH level, increases alkalinity and neutralizes acids in the water. In alkaline water the coagulation and flocculation processes work more effectively. Moreover, sufficient alkalinity prevents dissolving the lead from pipes and pipe fittings, as well as reducing the corrosiveness effect of the water to iron pipes.

Further, particles suspended in water start to precipitate and agglomerate to form larger particles, known as *flocs*. The flocs are then settled at the bottom forming a sludge. The sediment is removed from the process. After separating most of the floc, the water is filtered to remove the remaining suspended particles and unsettled floc.

In the filtration phase the water goes through the layers of anthracite, sand and gravel. Organic compounds contributing to taste and odor are removed. Other

remaining particles are trapped by adhering to the sand and gravel particles.

After harmful micro-organisms have been filtered, it is necessary to add disinfecting chemicals to the water in order to inactivate any remaining pathogens and potentially harmful micro-organisms. One of the disinfecting chemicals used is Sodium Hypochlorite (NaOCl). This chemical releases chlorine when it is dissolved in water, which is an efficient and safe disinfectant if added in a sufficient amount. Apart from sodium hypochlorite, liquid chlorine and chlorine dioxide may also be used as disinfectants.

Fluoride may also be added to the water with the goal of reducing tooth decay and preventing chronic diseases. However, fluoride in the water must not exceed recommended levels. Excessive levels of fluoride can be toxic or cause undesirable cosmetic effects such as staining of teeth.

Sodium Hypochlorite is unstable and easily decomposes. The stability of NaOCl solution is dependent on the following factors: hypochlorite concentration, temperature of the solution, pH value of the solution, concentration of the impurities catalyzing decomposition, and exposure to light. With the refractometer it is possible to monitor NaOCl concentration and control the disinfection conditions.

The water purification disinfection stage is accomplished in the disinfectant basin. In order to assure high quality of the purified water corrosion control is performed. Finally, the pure water is stored for further consumption.

## Instrumentation and installation


Vaisala K-PATENTS® Teflon Body Refractometer PR-23-M provides in-line measurements of Polyaluminium Chloride and Sodium Hydroxide at the initial stage of the purification process, ensuring the efficient flocculation of undesired particles. By measuring Sodium Hypochlorite and Fluoride at the water disinfection stage a high-quality purified water at the outlet is assured.

The refractometer is installed in three different points in a by-pass loop between each chemical storage tank pump outlet and the treatment point. The refractometer allows monitoring of the chemicals concentration at the exit from the storage tank to the pipe treatment point.

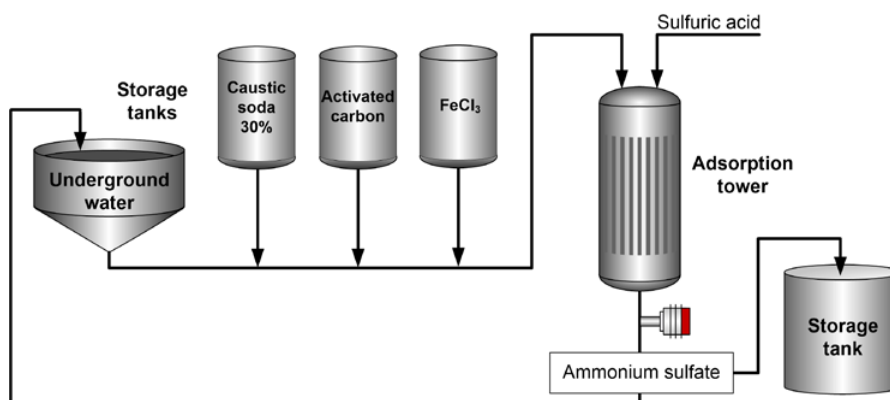
Typical measurement range of PACl is ca. 10-11 %.

Typical measurement range of NaOH is ca. 40-45 %.

Typical measurement range of NaOCl is ca. 8-12 %.

Instrumentation	Description
	Teflon Body Refractometer PR-23-M. A compact refractometer for chemically aggressive solutions and ultra-pure fine chemical processes. Connected to the process by a G1/2" female or a 1/2" NPT process connection. It has a built-in flow cell designed to keep all metal and other easily corroding parts from coming into contact with the process liquid.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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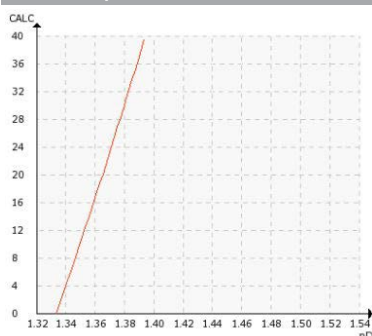


## AMMONIUM SULFATE (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>

### Typical end products

Inorganic salt with a number of commercial uses, e.g. soil fertilizer.

Chemical curve: Ammonium Sulfate, (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>, per Conc. % b.w. at Ref. Temp. of 20 °C



### Introduction

Small amounts of Ammonia (NH<sub>3</sub>) is present in aquifer naturally. Higher content is considered an impurity, often resulting from sewer backflow and drainage system problem, runoff in agricultural areas where it is used as a fertilizer, inadequate wastewater and solid waste disposal and treatment.

Ammonia is undesirable in water because ultimately it gets converted into nitrites and nitrates which can cause vitamin deficiency, and if combined with other components can cause cancer. Moreover, elevated ammonia concentration can create favorable conditions for intensive growth of aquatic organisms, including algae, which leads to deterioration of commodity water quality, especially its clarity, smell, taste as well as bacterial contamination.

### Application

For ammonia treatment to underground water caustic soda (30 %), activated carbon and ferric chloride (FeCl<sub>3</sub>) are used. Caustic soda is a chemical reagent,

pH-regulating, ion exchanger regenerating agent, catalyst, etching or cleaning agent. It leaves no residual colour.

Activated carbon is used as filter media for dissolved organics, as well as for the removal of color-, taste- and odor-causing compounds. It helps to improve the adsorbing capacity of ammonia.

Ferric chloride is a coagulant used for the treatment of turbidity and for further removal of color, natural organic materials, and arsenic from the raw water.


The ammonia is stripped away from the process. Sulfuric acid (50 %) is used as a stripping medium. Sulfuric acid added to the adsorption tower converts ammonia into ammonium sulfate ((NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>). Ammonium sulfate is an inorganic salt which has commercial value. Its most common use is as a soil fertilizer.

### Instrumentation and installation

Vaisala K-PATENTS® Teflon Body Refractometer PR-23-M provides in-line and continuous measurement of ammonium sulfate concentration. If the concentration value is correct, ammonium sulfate can be stored for further use. If the concentration is out of specification, the product is recycled for further processing and the adsorption process is adjusted accordingly.

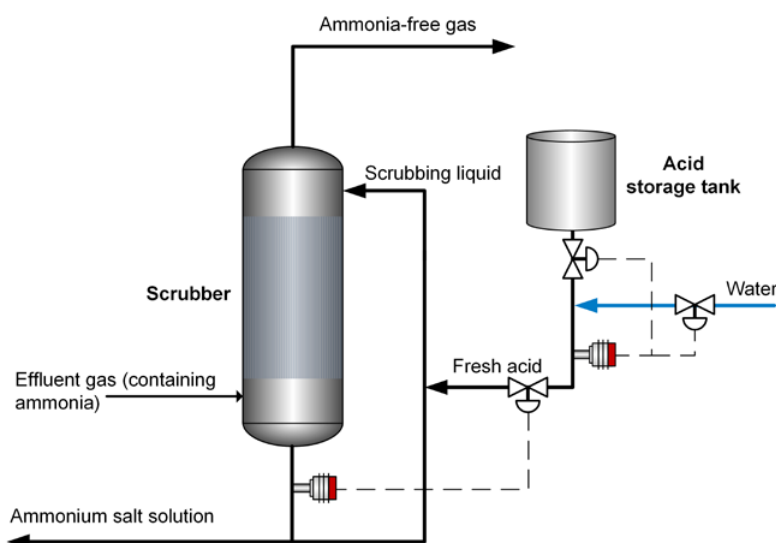
It is important to constantly monitor the ammonium sulfate concentration value in order to regulate the process correctly, to discharge or further recycle the product. Target concentration in this application is about 30 %.

The mounting point of the refractometer is at the outlet of the adsorption tower.

Instrumentation	Description
	Teflon Body Refractometer PR-23-M. A compact refractometer for chemically aggressive solutions and ultra-pure fine chemical processes. Connected to the process by a G1/2" female or a 1/2" NPT process connection. It has a built-in flow cell designed to keep all metal and other easily corroding parts from coming into contact with the process liquid.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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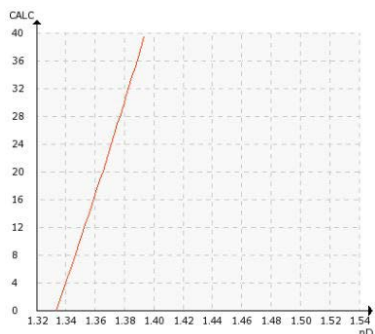


## SULPHURIC ACID (H<sub>2</sub>SO<sub>4</sub>), NITRIC ACID (HNO<sub>3</sub>), PHOSPHORIC ACID (H<sub>3</sub>PO<sub>4</sub>), AMMONIUM SULFATE (NH<sub>4</sub>)<sub>2</sub>SO<sub>4</sub>

### Typical end products

Ammonium salt of the acid, for example, ammonium sulphate, ammonium nitrate or ammonium dihydrogen phosphate, with different uses, such as nitrogen fertilizers.

Chemical Curve: Ammonium sulphate R.I. per % b.w. at Ref. T of 20 °C.



### Introduction

Ammonia is widely used in a variety of industries. Emissions of ammonia are of great concern as they contribute to several environmental problems including eutrophication and contamination of groundwater. Moreover, ammonia is very corrosive and may cause breathing problems at concentrations above 50 ppm and is considered poisonous in greater quantities.

### Application

Wet scrubbing is used successfully to control ammonia emissions, achieving efficiencies up to 99 %. However, wet scrubbers create effluents that need to be treated and disposed.

In acid scrubbing, a strong acid is added to the scrubbing liquid to react with the ammonia and to produce an ammonium salt, which can be safely discharged to a drain or wastewater treatment plant or sold as a liquid or solid fertilizer.

Typical ammonia scrubbers recycle liquid from the bottom to the top of the scrubber. This allows the scrubber to operate at reasonable wetting rates without the need for excessive acid addition or purge of liquid.

In continuous gas-liquid absorption systems, the concentration of the recycled solution is always controlled to ensure the proper acidic condition for scrubbing (usually a pH of 3 and 6) and to maintain the driving force by acid replenishment. The dosage of acid and water is controlled based on the pH and concentration of the scrubbing liquid.

### Instrumentation and installation


Vaisala K-PATENTS® Teflon Body Refractometer PR-23-M monitors continuously the concentration of the bottom product of the scrubber. The reading from the refractometer can be configured to show the preferred control unit, for example, Total Dissolved Solids (TDS) or density.

The refractometer's signal is used to control automatically the concentration of the recycle line to the scrubber by the addition of fresh acid. This ensures the optimal conditions for ammonia absorption.



A second refractometer controls the dilution of the acid. The refractometer's output signal can be used as feedback to control the water and acid feed valves to achieve the target concentration.

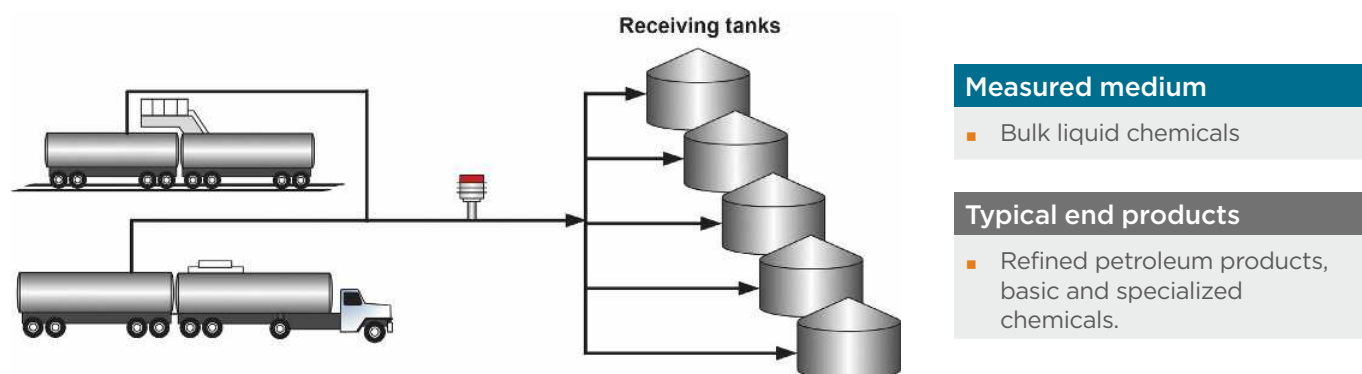
The design of the PR-23-M refractometer makes it safe for use in processes involving corrosive chemicals, such as ammonium salts and strong acids.

Instrumentation	Description
	Teflon Body Refractometer PR-23-M. A compact refractometer for chemically aggressive solutions and ultra-pure fine chemical processes. Connected to the process by a G1/2" female or a 1/2" NPT process connection. It has a built-in flow cell designed to keep all metal and other easily corroding parts from coming into contact with the process liquid.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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## Chemical interface identification at loading and unloading operations

Chemicals and allied I 4.07.01



### Introduction

In chemical plants, different chemicals are loaded and unloaded from trucks or rail cars to receiving or storage tanks. A plant may deal with multiple unloading stations; thus, fast and reliable interface detection is necessary for product identification and safe unloading operation.

Combining an interface detection device with automatic controls can minimize transmix of products, reduce waste, reduce the filling/unloading times, decrease safety risks, reduce sampling and minimize operator errors.

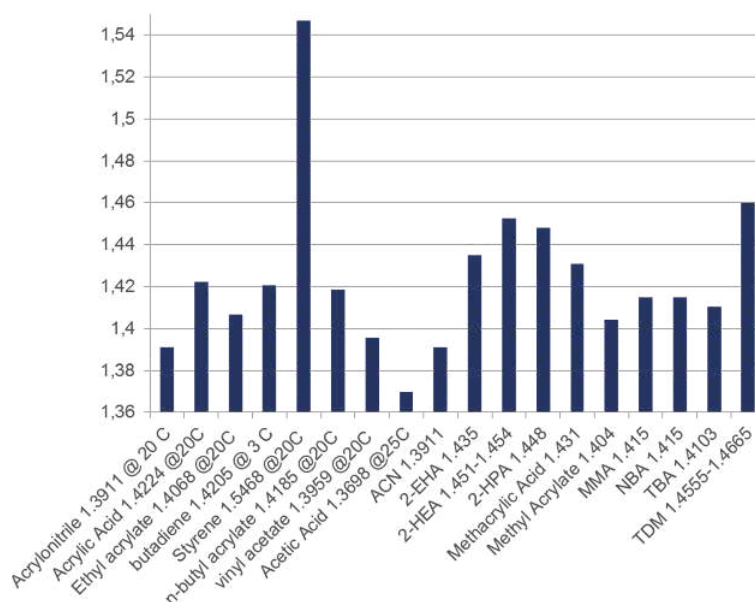


Figure 1. Refractive Index of different chemicals.

### Application

Refractive index is a reliable method for identifying a chemical before unloading it into receiving tank. Refractive index is a property inherent to each chemical and can be used as a fingerprint for product identification (Figure 1). This fingerprint can be applied to a scale to achieve the proper controls.



An example of product identification is chlorite and hypochlorite unloading operation. Each of these chemicals has a distinctive refractive index. The difference in refractive index is wide enough to accurately and reliably distinguish between these two products.

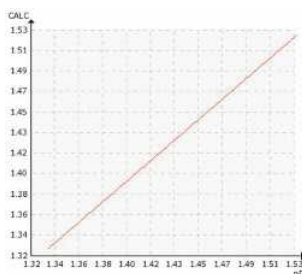
## Instrumentation and installation

Vaisala K-PATENTS Process Refractometer PR-43-G provides repeatable refractive index measurements with accuracy of  $nD \pm 0.0002$ . The refractive index measurement may also be used to provide the Brix value or other engineering scale for ease of use.

The refractometer is typically installed in the main supply line and in the inlet of the product storage. This provides adequate timing for interface detection and complete assurance that the liquid chemical is being delivered to its intended storage tank. Due to the digital sensing technology, small amounts of entrained air or gas will not affect the measurement.

The refractometer is available with stainless steel 316L and higher alloys wetted-parts. Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.
Chemical curve	R.I. at Ref. Temp. of 20°C



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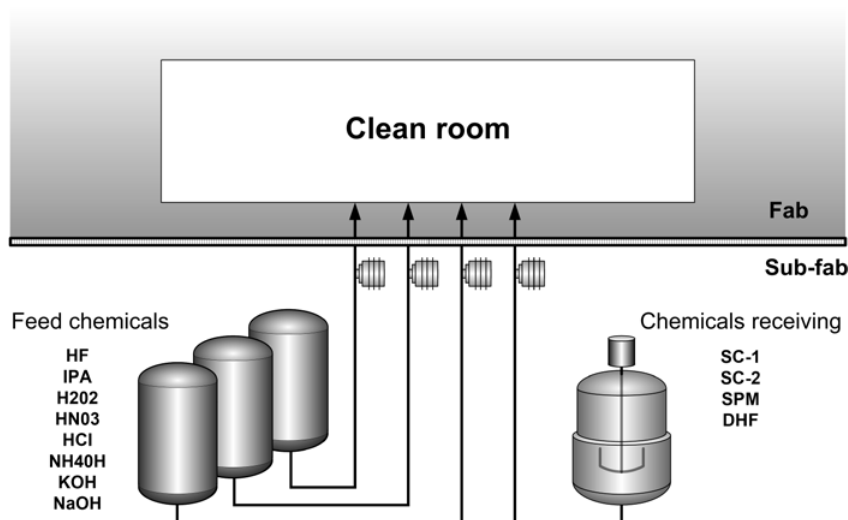
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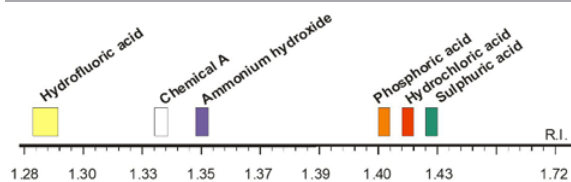


## SEMICONDUCTOR WET CHEMICALS

### Typical end products

Silicon wafers for semiconductors and microelectronics.

### Refractive Index of typical semiconductor chemicals



### Introduction

Typically, the chemical suppliers are responsible for the quality of the incoming chemicals. This leaves the semiconductor fabricators with a very limited capability to detect problems with the process chemicals that they receive from their suppliers. Poor chemical quality is revealed through production line test data or poor e-sort performance. Changes, which can occur during their handling on-site are disregarded and not monitored. Furthermore, factors such as human error in container handling and equipment failure at the distribution point are not taken into account.

Implementation of Vaisala K-PATENTS® Semicon Refractometer is economically feasible (costing less than the labor requirement for correcting a major mishap). It should also be noted, that expertise in the chemical distribution area should not be overlooked or underestimated. Instead this should be treated as a critical tool set, capable of making a significant impact on the fab very quickly.

### Application

The refractometer prevents the application of incorrect bulk chemicals and concentrations into the process. This way it helps to prevent expensive equipment

damage and wafer scrap. The key control function in this application is to provide an alarm as the chemical approaches its out-of-specification limit. In many cases, the cost of the refractometer can be recovered with the prevention of a single mishap.

The refractometer uses refractive index  $nD$  as a measurement principle. The benefit is that one refractometer can detect all chemicals, as every chemical is unique in terms of its physical properties, including its refractive Index value.

The standard refractometer covers the full refractive index  $nD$  range of 1.32 to 1.53 (corresponding to 0-100 % Conc.). This refractive index range can be extended by using Sapphire H74, YAG or GGG as prism material.



### Instrumentation and installation

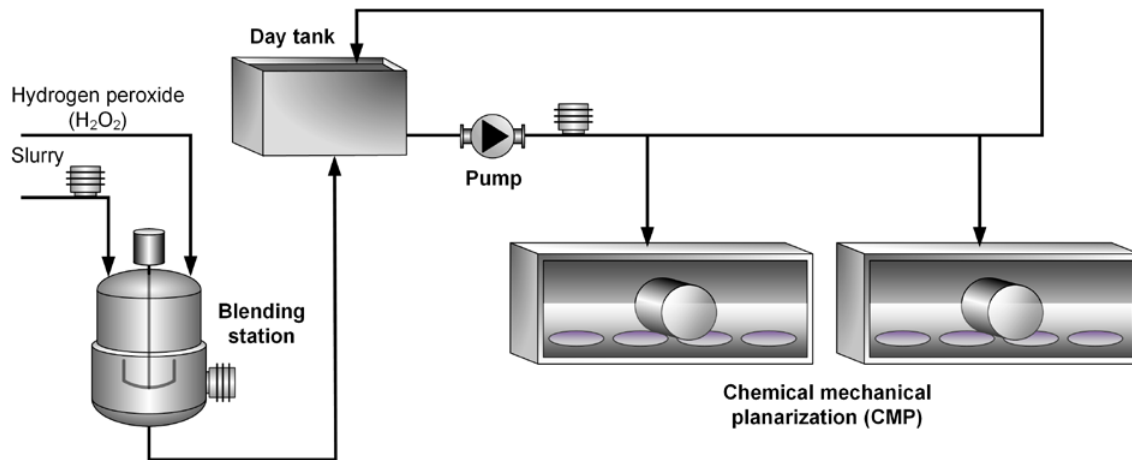
The refractometer measures the concentration of process liquids such as KOH (Potassium hydroxide), H<sub>2</sub>SO<sub>4</sub> (Sulfuric acid), HF (Hydrofluoric acid), NH<sub>4</sub>OH (Ammonium hydroxide), HCl (Hydrochloric acid), IPA (Isopropyl alcohol), Ethylene glycol and others. It also measures such chemical mixtures as SC-1, SC-2, SPM, DHF, etc. In multi-component solutions this is a checksum; if one of the components is wrong, the overall Refractive Index changes. The only chemical parameter, which is not detectable via refractive index  $nD$  is surfactant concentration because its concentrations are only at few part per million (ppm).

Depending on the brand and chemical distribution system, an ideal location and positioning for the refractometer will allow the monitoring of chemical feed to the fab as well as drum changes.

The refractometer provides a continuous 4-20 mA or Ethernet measurement signal. Shutdown set points are determined by the incoming chemical quality. Set points for the shutdown are set according to the tolerance range for the particular chemicals. For example, if the range of assay for sulfuric acid is 95 % to 98 %, the limits for the refractive index system can be 95.5 % to 97.5 %.

A distribution system shutdown takes place as the tolerance limit is approached, preventing violation, which is very similar to how a statistical process control program would function.

Instrumentation	Description
	Semicon Process Refractometer PR-23-MS is a compact, PTFE body refractometer for semiconductor liquid chemical processes. Connected to the process by a flare fitting, G1/2 inch female or a 1/2 inch NPT process connection. Mounted directly in-line without filtering.
	A small footprint, PVDF covered sensor for cleanroom environment and integrated process tools. Monitors the chemical concentrations in real-time and provides an Ethernet output signal and immediate feedback to the control system. Connected through a modified PTFE flow cell body to the process by a 1/4"-1" Nippon pillar or flare fitting.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

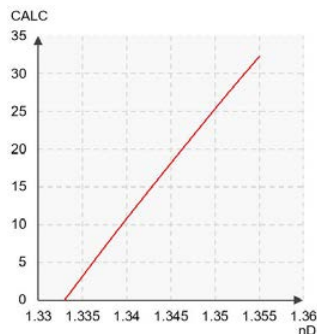


## HYDROGEN PEROXIDE(H<sub>2</sub>O<sub>2</sub>), CMP SLURRY

### Typical end products

Silicon wafers.

Chemical curve: Hydrogen peroxide R.I. per Conc% b.w. at Ref. Temp. of 20° C



### Introduction

Chemical mechanical planarization (CMP) is the process of smoothing and polishing the wafer's surface. This is done with the aid of an oxidizing agent, Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) for example, which contains abrasive particles suspended in the carrier fluid. H<sub>2</sub>O<sub>2</sub> oxidizes the silicon wafer surface to silicon dioxide. The polishing pad is then capable of polishing the wafer surface more efficiently because the newly oxidized layer is much softer than the silica.

The CMP slurries require mixing or dilution before use. Oxide polishing slurries are commonly purchased in concentrated form and diluted with water on-site to minimize shipping and labor costs. Some multi-component polishing slurries may only be blended just prior to their use because of their short post-mix lifespan. In the latter case, it is essential to measure the H<sub>2</sub>O<sub>2</sub> concentration of the mixed slurry, because altering the concentration of the slurry constituents will

affect the chemical reaction rates and wafer polishing rate.

Process tools that drive the semiconductor manufacturing processes, like CMP, are referred to as critical process systems. They are typically fed from CMP slurry delivery systems operated by the fab's facility management team. Automated chemical and slurry handling systems have tremendous implications to the safety, purity and up-time of the fabrication processes.

### Application

A typical blending and distribution system is composed of blend stations working in conjunction with each other to provide a continuous supply of slurry to multiple CMP polishers. Vaisala K-PATENTS® Semicon Refractometer PR-33-S is mounted as an integrated process metrology device, which measures the H<sub>2</sub>O<sub>2</sub> content of the slurry during blending.

The system blends the slurry, DI water and H<sub>2</sub>O<sub>2</sub>, adjusting the mix to the required ratios. The raw slurry is pumped from a supply drum connected directly to the blending tank. The refractometer monitors the quality of the raw slurry and ensures it has a consistent density.

The slurry and other components are directed into the blend tank by weight or flow and recirculated in the blend loop until the mixture becomes homogenized. The blend must pass a quality analysis before the slurry can be distributed to the day tank and/or CMP tools.


With a traditional on-line auto-titrator, it could take anything from minutes to an hour before the slurry

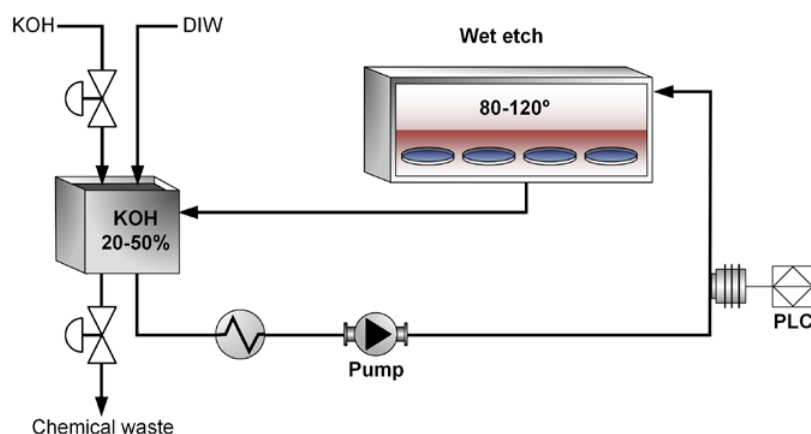
mixture can be validated as a qualified slurry blend. Optimizing the blend recirculation time by using a refractometer will eliminate unnecessary delays between slurry blending steps. Also, the qualification step which determines the CMP slurry's H<sub>2</sub>O<sub>2</sub> concentration can be shortened without sacrificing the slurry quality.

or in the slurry dispensing line. A typical H<sub>2</sub>O<sub>2</sub> concentration is 0-5 % by weight in the slurry and the process takes place at ambient temperature. The sensor can be installed into a vertical or a horizontal pipeline. The sensor should be mounted horizontally, so that the cable connection points downwards.

## Instrumentation and installation

The Semicon Refractometer can be installed in a circulation line at the slurry blending station and/

Instrumentation	Description
	A small footprint, PVDF covered sensor for cleanroom environment and integrated process tools. Monitors the chemical concentrations in real-time and provides an Ethernet output signal and immediate feedback to the control system. Connected through a modified PTFE flow cell body to the process by a 1/4"-1" Nippon pillar or flare fitting.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

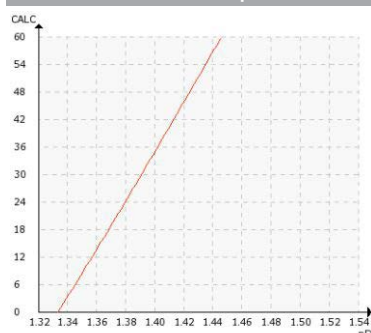


## POTASSIUM HYDROXIDE, KOH

### Typical end products

Silicon wafers for semiconductors and microelectronics, MEMS (microelectromechanical systems), micromachines etc.

Chemical curve: KOH R.I. per Conc. % b.w. at Ref. Temp. of 20 °C



## Introduction

Wet etching is used to chemically remove layers (metal, silicon, photoresist) from the surface of a wafer during manufacturing. Etchants that erode the substrate equally in all directions are called *isotropic*. Modern processes prefer *anisotropic* etches because they produce sharp, well-controlled features. Several anisotropic wet etchants are available for silicon. For instance, potassium hydroxide (KOH) is commonly used for this purpose, especially in the MEMS processing.

## Application

The etch rate of silicon in a KOH bath depends on the bath temperature and the KOH concentration. As etching progresses, some KOH (namely OH<sup>-</sup> ions) is consumed in the process.

Vaisala K-PATENTS® Semicon Refractometer gives a real time indication of KOH concentration and helps to determine the correct etch end-point. This way the bath life can be sustained for much longer, and wafer scrap

and chemical waste can be minimized. In certain cases, it is possible to double the KOH bath life.

## Instrumentation and installation

The refractometer is installed inside an etch cabinet on a circulation line, prior to the etch bath and directly after a pump. Typical KOH concentration is 30-50 % and the process temperature is 80-120 °C (176-248 °F). The temperature can be as high as 160 °C (320°F) for heated KOH etching. The sensor can be installed into a vertical or a horizontal pipeline. The sensor should be mounted in the horizontal axis position. We recommend a minimum flow velocity of 1.5 m/s (5 ft/s).

## Compensation of Silicate

Refractive index gives an indication of total dissolved solids. The etching of silicon using a KOH water mix forms a tertiary solution with the dissolved Silicate. The influence of Silicate needs to be compensated from the KOH concentration reading. We provide a method (patent pending) for compensating this in the refractometer output reading.


Dissolved Silicate increases the concentration reading. This offset can be compensated with our simple balance calculation and without introducing any extra measurements.

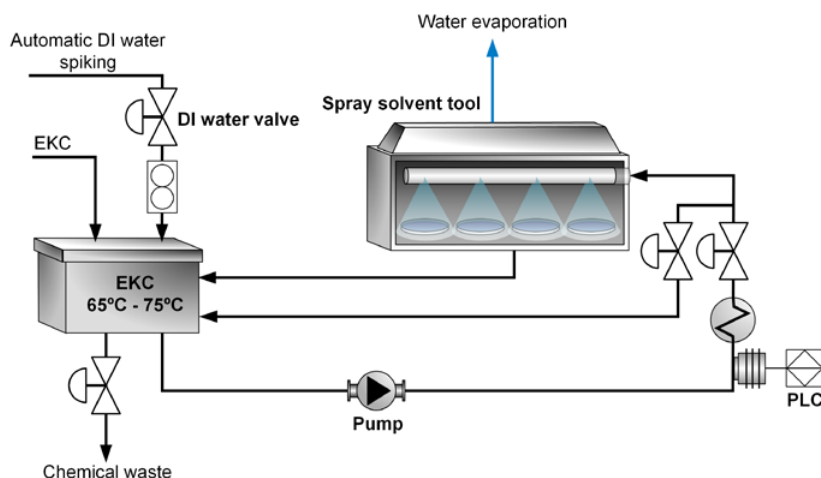
The amount of potassium (K<sup>+</sup>) does not change. The only two variables that change are:

- the amount of water (which may change due to evaporation and in the reaction),
- and the amount of silicon (which depends on the wafer design and etch depth). The amount of silicon can be estimated when the number and type of wafers etched in the solution are known.



The compensation factor can be implemented in the control system. The same phenomenon is seen when etching Silicon nitride with heated KOH.

Instrumentation	Description
	Semicon Process Refractometer PR-23-MS is a compact, PTFE body refractometer for semiconductor liquid chemical processes. Connected to the process by a flare fitting, G1/2 inch female or a 1/2 inch NPT process connection. Mounted directly in-line without filtering.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

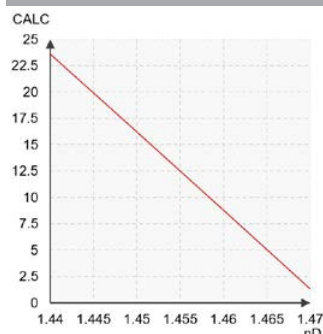


## POLYMER RESIDUE REMOVER, EKC

### Typical end products

Silicon wafers.

Chemical curve: Water in EKC R.I. per Conc% b.w. at Ref. Temp. of 20 °C



### Introduction

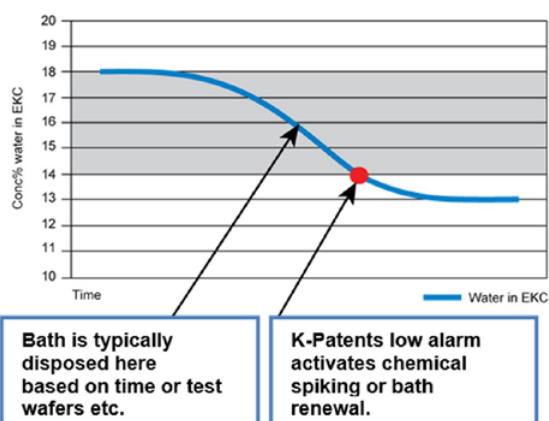
Spray solvent tools are used in the post etch residual polymer removal process to clean the surface of wafers. EKC is a commonly used chemical in the process that employs two tanks. The process operating temperature of EKC is kept between 65 °C (149 °F) and 75 °C (167 °F). The actual residue removal takes place in the spray solvent tool where EKC is fed to, according to a pre-determined cycle of chemical recirculation and wafer run.

### Application

Two control strategies can be adopted with the aid of Vaisala K-PATENTS® Semicon Refractometer PR-33-S. The first one is to monitor water content in EKC, and the second is to maintain the water content at a constant level. The former allows for setting high and low alerts for the amount of water, which warrants safe wafer production. The latter has proved to provide a considerable reduction in the EKC consumption.

The recirculation starts when the wafer run in the spray solvent tool is over and the chemical is delivered to the EKC tank. The tank is partially drained of the chemical and refilled with fresh EKC and water. Water is evaporating through the exhaust system of the spray solvent tool during the spray run.


The amount of water in EKC is maintained with automated chemical spiking after each wafer run. The chemical spiking is activated using the PR-33-S output signal. This arrangement provides the exact set point for water in the EKC concentration and helps to optimize and reduce EKC replenishment.

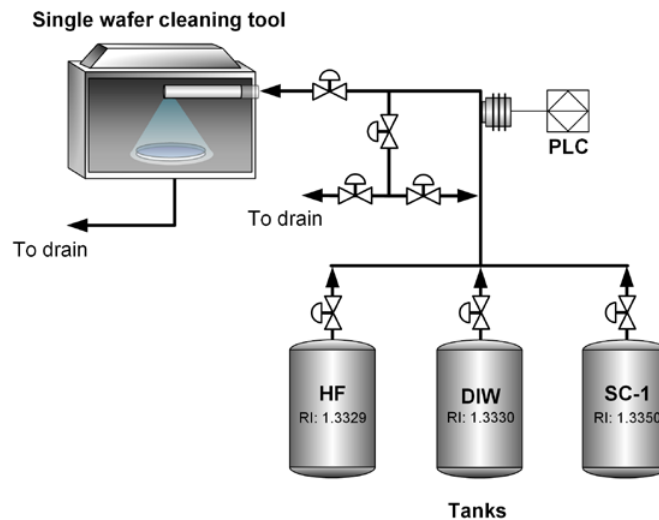


After EKC clean, there is an IPA/Dry process where IPA may get contaminated with EKC traces. The Semicon Refractometer can monitor the concentration of the chemicals to alert of possible mixes. The refractive index values of EKC and IPA differ substantially allowing a very reliable measurement of EKC traces in IPA.

## Instrumentation and installation

The Semiconductor Refractometer PR-33-S is installed in the EKC recirculation loop. The compact instrument's design suits the water level control for EKC perfectly. Data logging software allows on-line printing of concentration and temperature profiles.

Instrumentation	Description
	A small footprint, PVDF covered sensor for cleanroom environment and integrated process tools. Monitors the chemical concentrations in real-time and provides an Ethernet output signal and immediate feedback to the control system. Connected through a modified PTFE flow cell body to the process by a 1/4"-1" Nippon pillar or flare fitting.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

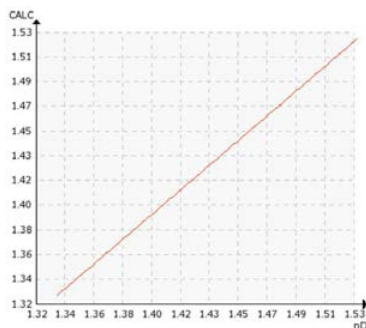


## HYDROFLUORIC ACID (HF), DEIONIZED WATER (DIW), SC-1 (H<sub>2</sub>O<sub>2</sub>, NH<sub>3</sub>)

### Typical end products

Silicon wafers.

Chemical curve: R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Cleaning chemicals are the most consumed liquids in a fab. These include acids and bases to remove organic and inorganic contaminant residues from the wafers. The most commonly used cleaning solutions are *standard clean 1* (SC-1) and *standard clean 2* (SC-2). If the wafers are particularly contaminated, there are additional cleaning steps utilizing a solution known as *Caros* or *Piranha* and diluted hydrofluoric acid (DHF).

Thorough rinsing with deionized water (DIW) is required after each chemical step. This is usually followed by a spin dry process.

Common tools for wafer cleaning are immersion baths and single wafer spray systems. Centrifugal spray

units offer benefits over immersion baths since each wafer is uniformly exposed to uncontaminated, fresh chemicals.

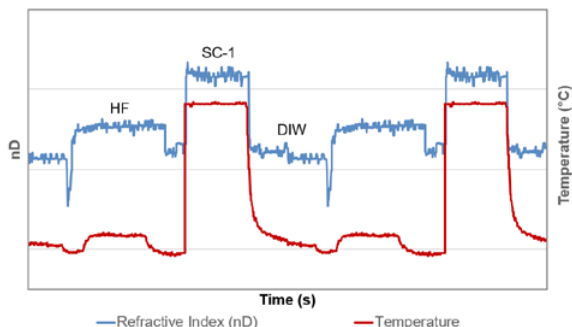
### Application

The spray acid chamber is designed to allow multiple chemicals to be applied on the wafer in-situ through a nozzle or dispenser, providing tight control while eliminating the risk of contamination.

HF and SC-1 are common chemicals used for wafer cleaning. The chemicals are delivered separately with a DIW rinse between steps. These solutions are provided at certain preferred temperatures, in a particular sequence and manner. The time span for each step is only tens of seconds.

In order to maintain a continuous flow, the interface between the liquids should be monitored. Vaisala K-PATENTS® Semicon Refractometer PR-33-S allows for instant switching between cleaning chemicals and DIW by means of refractive index measurement (Figure 1).

Refractive index is an inherent property of all liquids and is unique for each chemical. The refractometer immediately detects the liquid in the line and sends a signal to the PLC to open and close valves and divert the flow as required. This ensures the correct chemical is dispensed and prevents mixing between liquids.



**Figure 1.** Example of interface detection between wafer washing steps with the refractometer.


## Instrumentation and installation

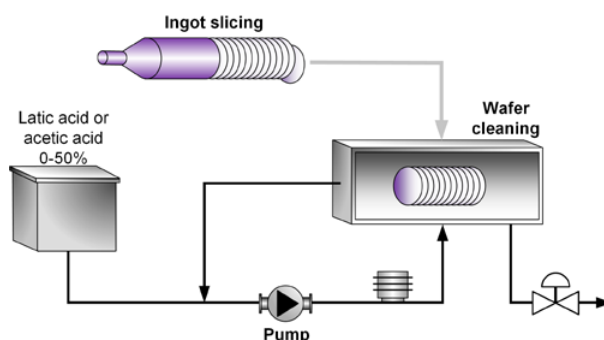
The Semicon Refractometer PR-33-S is mounted as an integrated device in the cleaning tools. The refractometer provides Ethernet and 4-20 mA output

signals that can be connected to the process controller for continuous and accurate control.

Set-points based on the refractive index values of the cleaning chemicals and DIW can be created to control the valves. The refractometer covers the full refractive index range  $nD$  of 1.32 to 1.53, with a measurement accuracy of  $nD \pm 0.0002$ .

Moreover, the refractometer has a built-in temperature sensor to measure the temperature of the surface in contact with the process liquid, providing fast and automatically temperature compensated reading. Typical temperature range in this application is 20-60 °C (68-140 °F).

Instrumentation	Description
	A small footprint, PVDF covered sensor for cleanroom environment and integrated process tools. Monitors the chemical concentrations in real-time and provides an Ethernet output signal and immediate feedback to the control system. Connected through a modified PTFE flow cell body to the process by a 1/4"-1" Nippon pillar or flare fitting.
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

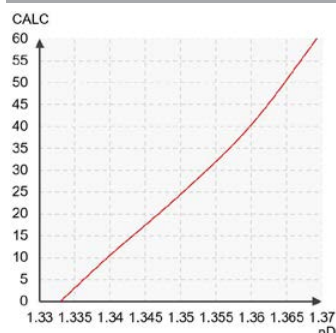


## ACETIC ACID ( $\text{CH}_3\text{COOH}$ ), LACTIC ACID ( $\text{CH}_3\text{CHOHCOOH}$ )

### Typical end products

Solar wafers, solar cells.

Chemical curve: Acetic acid R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Silicon crystal growing and casting plants are either an integrated unit of the photovoltaic manufacturer or independent sub-contract producers. They don't need to be situated in the proximity of solar cell production plants because wafer transportation is inexpensive. Most are though, because the Photovoltaic manufacturers have invested to ensure a secure wafer supply to their cell plants.

Wafer processing starts with cutting ingots into bars, then slicing them with a wire saw into wafers (slightly thicker than a compact disc). The product is then cleaned in preparation for cell manufacturing. The cleaning phase consists of an acetic or lactic acid bath, where the wafers are immersed in order to remove glue and other residues such as Si particles from the wafer surfaces.

Solar cell plants then take the cleaned wafers through a high-technology processing sequence to create the final solar product.

### Application

The lactic or acetic acid bath is typically kept at a concentration between 30 % and 50 %.


Vaisala K-PATENTS® Semicon Refractometer PR-33-S measures the bath concentration and helps to optimize the consumption of chemicals, and also to determine the exact timing for bath replenishment. Subsequently, an important task is to be able to control the concentration of the bath chemistry. As the chemical calibration curve (on the left) shows, the correlation of refractive index versus concentration of the used chemical (acetic acid or lactic acid) is linear.

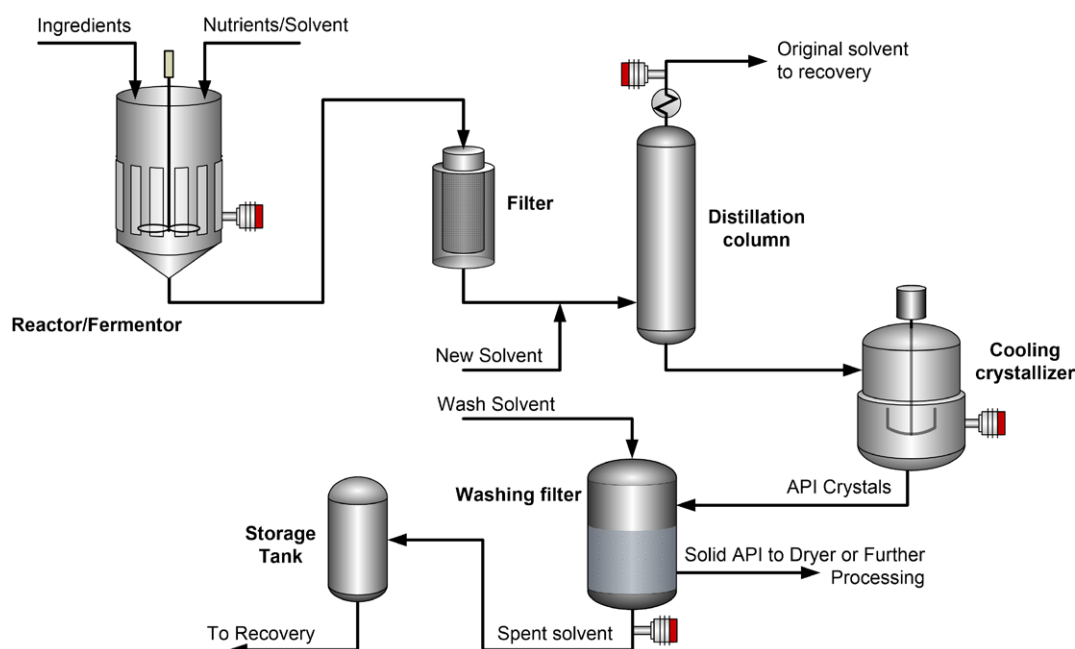
The typical process temperature is 20-40 °C (68-104°F) and it is also measured with the PR-33-S. Automatic temperature compensation is crucial for the concentration measurement, as refractive index is dependent on the process fluid temperature.

### Instrumentation and installation

The Semiconductor Refractometer PR-33-S is installed in the re-circulation loop of the acid bath. Our data logging software via Ethernet allows real-time collection and printing of the concentration and temperature values and sensor diagnostic data.

The sensor output signal is used to establish the optimum level of acid concentration and to trigger the spiking of the fresh chemical.

Instrumentation	Description
	A small footprint, PVDF covered sensor for cleanroom environment and integrated process tools. Monitors the chemical concentrations in real-time and provides an Ethernet output signal and immediate feedback to the control system. Connected through a modified PTFE flow cell body to the process by a 1/4"-1" Nippon pillar or flare fitting.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

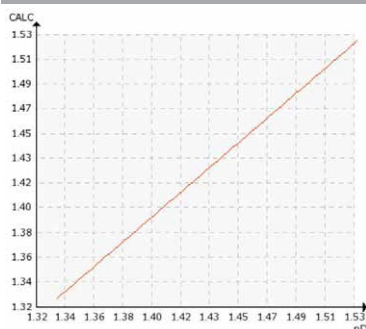


## ACTIVE PHARMACEUTICAL INGREDIENT (API), SOLVENTS

### Typical end products

Active Pharmaceutical Ingredients (APIs) for medical treatment or the manufacture of other drugs.

### Chemical curve: R.I. at Ref. Temp. of 20°C



### Introduction

Active Pharmaceutical Ingredients (API) are specialty chemicals that have a disease-curing value. These APIs are manufactured through different steps usually including reaction, separation and purification.

Traditionally, manufacture has been performed by batch processes because of their simplicity and flexibility. However, as demand for drugs increases, pharmaceutical companies are moving towards continuous operation and larger scale production. Different guideline changes and initiatives have been put into action to reduce production costs while increasing efficiency, productivity and product safety. For instance, the *Process Analytical Technology (PAT)*

initiative promotes the use of in-line measurements throughout the entire manufacturing process.

### Reaction

The API can be obtained by a variety of processes, such as chemical synthesis, natural product extraction and fermentation. The reactions can be carried out in batch or continuous reactors, that are usually equipped with jacket and agitator.

The first step is the addition of the raw materials and solvents to the reactor. These are mixed, cooled or heated, and they are let to react for a certain time to obtain the API product. The ratio of raw materials, process conditions, and reaction time are defined by the specific product formulation.

Vaisala K-PATENTS® Pharma Refractometer provides a real-time refractive index measurement that is used to follow the degree of conversion and determine the end-point of the reaction. In fermentation, the refractometer's signal can also be used to detect the need for the further addition of nutrients and to control automatically the feeding valve (see also application note *Pharmaceutical Fermentation*).

### Separation and purification

After the reactor, the API undergoes a series of steps for its separation and purification. Common downstream operations are solvent swap, crystallization, filter cake washing and drying.



## Solvent swap or solvent exchange

Solvent swap is performed to remove the solvent used in an earlier step (original solvent), and to replace it with another solvent (swap solvent) that is more suitable for the next processing step. This can be done, for example, by batch distillation.

The new solvent is usually added to the product from the reactor, and the mixture is heated. As the components reach their boiling points, the most volatile solvent (the original solvent) is evaporated from the mixture, leaving behind the API mixed with the new solvent. As distillation progresses, the concentration of the solvents should be monitored to ensure a pure product or original solvent.

It can be challenging to measure the concentration of the mixture inside the vessel as the swap takes place. In such cases, the Pharma Refractometer is installed after the condenser to measure the concentration of the condensed vapor. As there is vapor-liquid equilibrium (VLE) inside the vessel, the concentration of the swap solvent can be then determined with the aid of VLE data, the temperature and the information provided by the refractometer (see also application note *Solvent Swap*).

## Crystallization

Recovery of the API to a solid state and further purification is commonly achieved by crystallization. Consistent crystal morphology and good particle size are required when crystallizing the API. Poor quality crystals are difficult to process and may require costly recycling and reprocessing.

The Pharma Refractometer measures continuously the concentration of the mother liquor as it is saturated. The refractometer provides useful information to monitor supersaturation and determine the seeding point. Due to our unique digital sensing technology, the measurement is not affected by bubbles or crystals formed during the process. The accurate information from the refractometer guarantees a consistent product quality.

Moreover, the measurement of the Pharma Refractometer is highly accurate and repeatable and can also be used for obtaining the solubility curve of an API (see also application note *Pharmaceutical crystallization*).

## Filter cake washing

Filtration and washing is required for efficient removal of the mother liquor and to obtain pure solids. Filter cake washing is achieved by filtration of the product from crystallization while applying a separate washing solvent.

Refractive index is a valuable tool for the development, monitoring and control of cake washing operations. At laboratory scale, and at a development stage, the Pharma Refractometer provides valuable information for understanding the washing process, creating washing profiles and to find the most suitable washing solvent.

Because each liquid has a different refractive index value, the refractometer can detect immediately the interface between the original solvent and the washing solvent. This indicates the end-point of washing, thus reducing the amount of solvent used. The refractive index of pure solvent and saturated solvents can also be studied and used as reference to determine if the washing conditions are not optimal, and if some product is washed away. This ultimately increases the yield and productivity (see also application note *Filter Cake Washing*).

## Process fingerprint and troubleshooting

Refractive index has proven to be a useful tool not only for API process monitoring and control, but also for process understanding, evaluation, and troubleshooting.

The precise measurement of the refractometer can be used for finding the *process profile* or *fingerprint*. This is a reference during scale-up to confirm that the process behaves as designed and to assure there is process equivalence.

Refractive index can also provide the basis to understand the interaction between the raw materials, formulation, and conversion yields, as well as to identify Critical Process Parameters (CPPs). The data from the refractometer can be used to set the correct range limits for CPPs and to determine acceptable process variations.

## Instrumentation and installation





Vaisala K-PATENTS® Pharma Refractometer PR-43-PC is the ideal in-line process analyzer for pharmaceutical processing in accordance with the PAT framework. The refractometer provides measurement of the refractive index of the medium, which can be used at any step in the development and production of an API. The scalability feature of the PR-43-PC

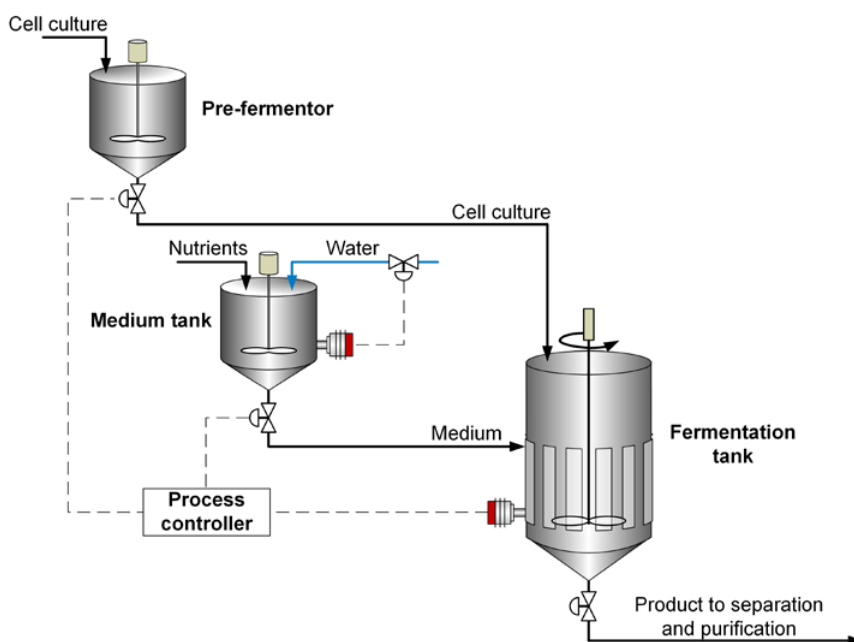
helps the drug manufacturer to reduce development time and improve product reliability throughout the complete process analysis. The measurement by the refractometer is accurate and very repeatable, providing assurance at any scale.

For larger scales, Vaisala K-PATENTS® Sanitary Probe Refractometer PR-43-AP can be used. The refractometer is also available with special materials and alloys for corrosive mediums. The refractometer

is constructed of pharma grade materials, and the product surface finishes are electro-polished.

The refractometer is available with 3-A Sanitary and EHEDG certifications. Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

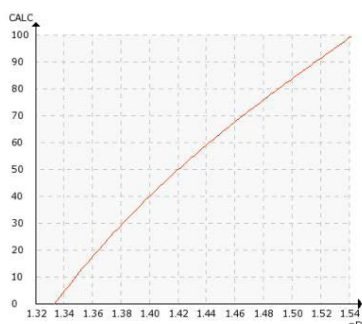


## FERMENTATION BROTH, SUGAR SYRUPS (GLUCOSE, FRUCTOSE)

### Typical end products

Active Pharmaceutical Ingredients (APIs) for the manufacture of different drugs, such as antibiotics, vitamins and enzymes.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Pharmaceutical fermentation is the basis for the production of a wide range of products, such as antibiotics, hormones, vaccines, and specialized proteins. This process involves the conversion of organic materials into simple substances by the action of micro-organisms.

The key elements in fermentation are the selection of the right strain and process medium. The process conditions need to be developed and optimized throughout scale-up to maximize productivity and reduce variation in the product yield.

Biotechnological processes, such as fermentation, are becoming increasingly important for the manufacture of Active Pharmaceutical Ingredients (APIs). There is a necessity in the pharmaceutical industry to standardize and automate the processes to meet the requirements for consistent quality, to accelerate development and production times, to increase yields and to reduce costs.

### Application

Fermentation is a process for cultivating organic materials. The process involves several phases and takes place under strictly controlled conditions.

The first step for fermentation is the preparation of the *inoculum* for the cell culture. The type of micro-organism is selected to yield the right product. For example, to produce penicillin, *Penicillium Chrysogenum* is used, and to produce glutamic acid, *Corynebacterium Glutamicum* is used.

The cell culture is dosed to large vessels known as *fermentation tanks* or *fermenters*, containing the process medium. The medium is an important factor in the process. It is carefully selected to ensure it is easily degradable by the micro-organisms and to guarantee viability of the cells. The reaction regime is also designed to maximize the productivity of the

organism of interest by providing optimal conditions for population growth.

The actual fermentation reaction (or *incubation*) is slow and can take up to several days. During incubation the nutrients in the medium (e.g. glucose) are depleted as the biomass and end product is produced. The concentration inside the tanks is monitored throughout the reaction time to follow the degree of fermentation and detect the end-point of reaction. This is commonly achieved by sampling and laboratory tests, for example by refractive index measurements.

The fermentation product, known as the *fermentation broth* is processed downstream by various technologies for extracting, concentrating and purifying the main product or API from the broth.

## Instrumentation and installation

Vaisala K-PATENTS® Pharma Refractometer PR-43-PC provides accurate and repeatable refractive index measurements throughout the development and production of an API.

At laboratory scale, the refractometer is used as a PAT tool for the creation of fermentation profiles. The correlation between refractive index and nutrient concentration in the fermenter is a valuable tool for the operation at larger scales and to maintain a viable medium for cell incubation. The reaction profile guarantees that the process behaves as designed and is useful for identifying and investigating deviations.




At pilot and full scales, the refractometer is installed directly in the fermentation tank to provide real-time information on the conversion rate and degree of fermentation. The continuous measurement of the refractometer combined with the fermentation profile help to identify the reaction end-point and the need for nutrient addition. In-line measurement reduces sampling and laboratory testing.

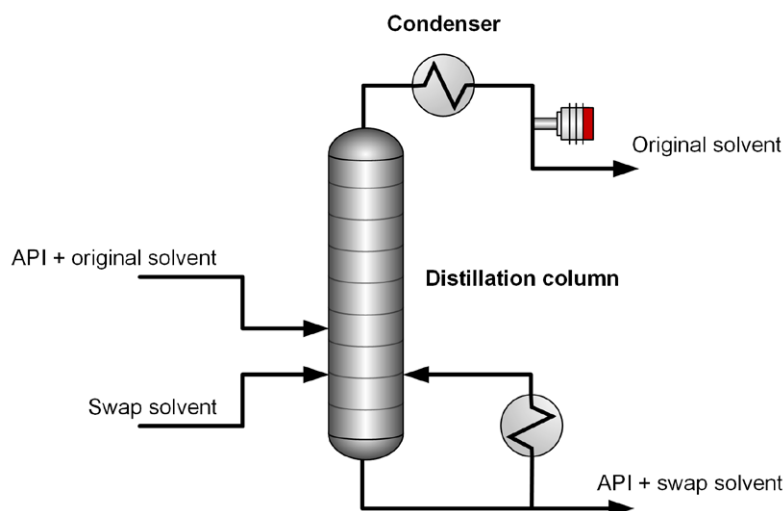
The refractometer provides Ethernet and 4-20 mA output signals that can be connected to the process controller to control automatically the feed valves to the fermenter.

The refractometer is also ideal to monitor and control other operations upstream, for instance, for syrup dilution or nutrient solution preparation. The refractometer is installed directly in the tank or in a bypass line.

Due to its unique digital sensing technology, the refractometer is accurate and does not drift in the presence of bubbles or suspended particles. The refractometer is delivered factory calibrated and does not require re-calibration. Moreover, verification is easily performed using standard refractive index liquids.

When many instruments are required, the Multichannel User Interface (MI) connects up to 4 refractometers, thus reducing the investment cost. The MI provides user authentication, electronic records, data logging, event log and audit trail, all in compliance with pharmaceutical requirements.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

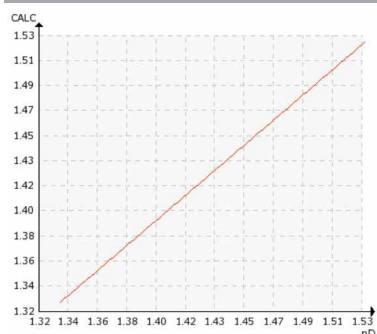


## ACTIVE PHARMACEUTICAL INGREDIENT (API), SOLVENTS

### Typical end products

Active Pharmaceutical Ingredients (APIs) for medical treatment or the manufacture of other drugs.

### Chemical curve: R.I. at Ref. Temp. of 20 °C



### Introduction

Solvents have a multipurpose role in pharmaceutical processing. Different solvents can be used in different processing steps, such as chemical reaction, separation and purification of the Active Pharmaceutical Ingredients (APIs).

Very often, the reaction takes place in a solvent (original solvent) and the next processing step (e.g. reaction, crystallization, extraction or washing) requires a different solvent.

Solvent swap (or solvent exchange) is a common and important task in the production of an API, which is performed to remove the original solvent that is used in an earlier processing step, and to replace it with another solvent (swap solvent) that is more suitable for the next processing step.

### Application

Solvent swap is usually achieved by batch distillation. In this case, the swap solvent is mixed with the original solvent mixture (containing the API) and loaded to a batch distillation column. The original solvent is distilled off the mixture and is collected as the top product of the column. The swap solvent and the API are collected as the bottom product.

Traditionally, solvent swap is performed in a stepwise operation. First the original solvent is concentrated by distillation before the addition of the swap solvent. Both the original solvent and the swap solvent are again distilled to a certain concentration and more swap solvent is added. This sequence is repeated until the original solvent is reduced, and the swap solvent reaches the desired concentration.

The solvent swap operation can be optimized by reducing the amount of solvent used and minimizing the energy consumption, while still obtaining the same total separation. This can be achieved by real-time measurement of solvent concentrations during the swap. The purpose is to determine the right point for swap solvent addition and the point where the desired swap purity is reached.

### Instrumentation and installation

Vaisala K-PATENTS® Pharma Refractometer is usually installed in-line after the distillation column's condenser to measure the concentration of the top product. The refractometer provides Ethernet and 4-20 mA output signals that can be used as feedback for automated control of the solvent swap operation. The real-time measurement of the original

solvent concentration determines the point for swap additions and ensures a consistent and reproducible product. This leads to optimized solvent and energy consumption, and reduced operational costs.




The Pharma Refractometer can be used at any stage during the development and production of an API. The refractometer provides highly accurate and repeatable refractive index measurement to maintain consistency of the process in different scales from development to scale-up and commercial manufacturing.

The refractometer is also used for process understanding and the development of vapor-liquid equilibrium (VLE) data that helps the scale-up process. For instance, at laboratory and pilot scales it can be challenging to measure the concentration of the mixture inside the vessel when the swap takes place. In these cases, the Pharma Refractometer is installed after the condenser to measure the concentration of the condensed vapor. As there is VLE inside the vessel, the concentration of the swap solvent can be

then determined with the aid of VLE data, temperature and the information provided by the refractometer.

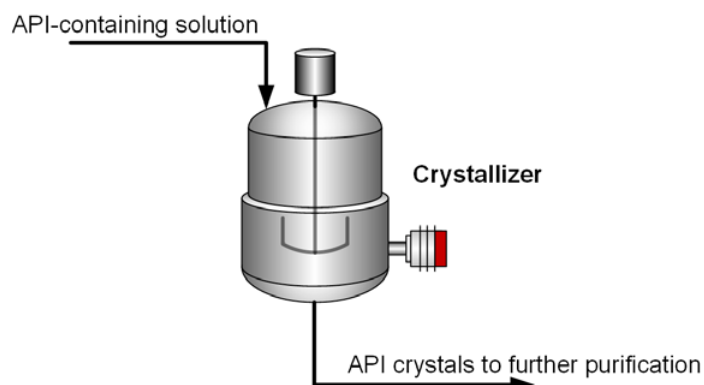
Due to its unique digital sensing technology, the refractometer does not drift in the presence of bubbles or suspended particles. The refractometer is delivered factory calibrated and does not require re-calibration. Moreover, verification is easily performed using standard refractive index liquids.

When many instruments are required, the Multichannel User Interface (MI) connects up to 4 refractometers, thus reducing the investment cost. The MI provides user authentication, electronic records, data logging, event log and audit trail, all in compliance with pharmaceutical requirements.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

Ref. B211936EN-A © Vaisala 2020



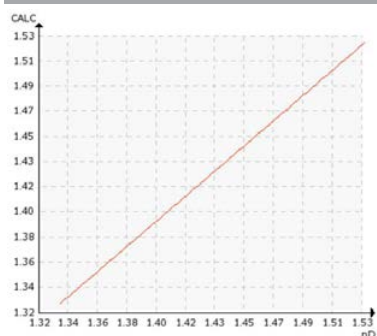


## ACTIVE PHARMACEUTICAL INGREDIENT (API)

### Typical end products

Active pharmaceutical ingredients (APIs), excipients, intermediates and raw material for pharmaceutical drugs.

### Chemical curve: R.I. at Ref. Temp. of 20 °C



## Introduction

Crystallization is an important unit operation within many pharmaceutical processes for the separation and purification of intermediate compounds and active pharmaceutical ingredients (APIs).

The operating conditions of the crystallization process determine the physical properties of the product such as the crystal purity, size, and shape distribution. These properties determine the efficiency of downstream operations, such as filtration, drying, milling and tableting, and the product effectiveness, such as bioavailability and shelf life.

## Application

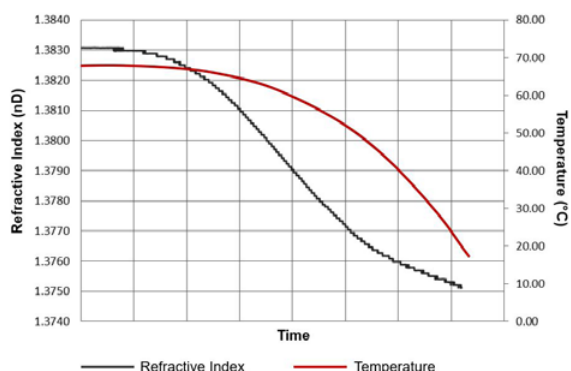
*Supersaturation* is the driving force for nucleation and growth of the crystals. Supersaturation is defined as the state at which any solution contains more dissolved solid (solute) than can be found in saturation conditions. This means that crystallization can only happen if the conditions of the liquid are taken above its saturation point, and by doing this, the excess solute will precipitate. In the crystallizer, a

supersaturation state is created by means of cooling, evaporation or reaction.

Supersaturation has a direct effect on the final crystal and product quality. A high degree of supersaturation accelerates the nucleation and growth rate and will consequently result in agglomeration of the crystals and in a difficult downstream processing. If the supersaturation is too low, it might be insufficient to cause crystal formation. Therefore, close monitoring of the supersaturation is necessary in crystallization control.

Concentration measurement of the mother liquor inside the crystallizer is necessary for determining the correct seeding point and controlling nucleation and growth rates. As the crystals grow, and the solute is transferred to the crystal, the mother liquor concentration will decrease. At this point the mixture inside the reactor is a slurry consisting of solids and liquids. A selective measurement of the liquid phase helps to control the supersaturation.

Figure 1 shows a typical pharmaceutical crystallization process, where supersaturation and temperature are controlled, and a seeding technique is used. The solution containing the API is fed to the crystallizer. Supersaturation is achieved by cooling. Seeding is performed to control the saturation and to start crystallization at the desired level. The concentration, followed by a measurement of refractive index ( $nD$ ), decreases as the crystals form until it stabilizes, marking the end-point of crystallization. The final product consists of crystals of the desired size and morphology.



**Figure 1.** Profile of a cooling crystallization process, with seeding and temperature control.

## Instrumentation and installation

Vaisala K-PATENTS® Pharma Refractometer PR-43 is used to measure in real-time the concentration of the liquor inside the crystallizer. The refractometer is usually mounted directly in the crystallizer, thus eliminating the need for sampling.




The measurement of the refractometer is not influenced by the crystals or bubbles in the liquid. The measurement is selective to the liquid phase, making it ideal to follow liquid-solid operations. The

refractometer can follow the supersaturation level to determine the exact seeding point. This also helps to estimate the correct crystallization parameters in order to achieve a stable operation and high-quality crystals.

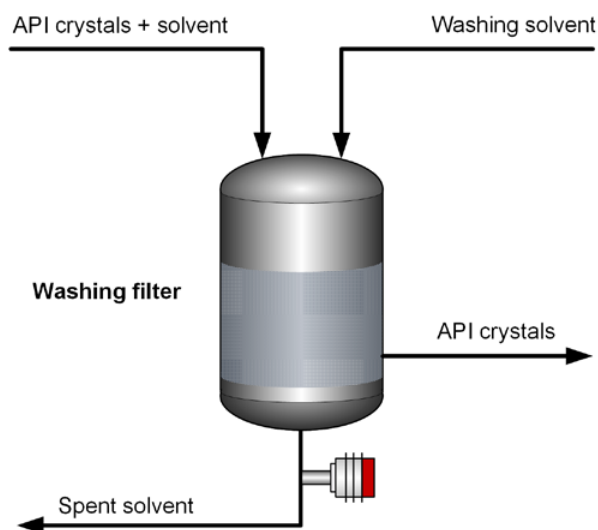
The refractometer provides refractive index measurement of high accuracy and repeatability. The measurement of the refractometer is ideal to create solubility curves of APIs or crystallization profiles at laboratory scale that are valuable for scale-up operations.

The refractometer is delivered factory calibrated and does not require re-calibration. Moreover, verification is easily performed using standard refractive index liquids.

When many instruments are required, the Multichannel User Interface (MI) connects up to 4 refractometers, thus reducing the investment cost. The MI provides user authentication, electronic records, data logging, event log and audit trail, all in compliance with pharmaceutical requirements.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
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	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
<b>User Interface</b>	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
<b>Measurement range</b>	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.



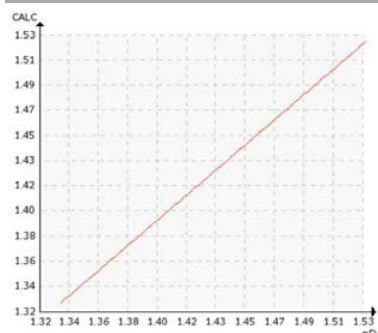


## ACTIVE PHARMACEUTICAL INGREDIENT (API), SOLVENT

### Typical end products

Active Pharmaceutical Ingredients (APIs) for the manufacture of different drugs, such as antibiotics, vitamins and enzymes.

Chemical curve: R.I. at Ref. Temp. of 20 °C



### Introduction

After crystallization, the active pharmaceutical ingredient (API) needs to be separated from the mother liquor and impurities. Separation of the API crystals is usually performed by pressure filtration.

Filter cake washing is an important step in pharmaceutical processing, since high purity is a main requirement. Inefficient washing of the API crystals affects the quality attributes of the final drug and may create problems in downstream processing.

### Application

Filter cake washing removes the impurities and mother liquor from the filter cake by the application of a miscible and pure washing solvent. The number of washing steps required, and the washing solvent are specific for each process.

The design and operation of cake washing requires thorough evaluation. Solubility of the API in the washing solvent is undesirable as it reduces the product yield. Therefore, the washing solvent must be carefully selected to displace the mother liquor from the cake whilst minimizing product dissolution.

Process understanding is also necessary. The development of washing profiles can help to understand the washing regime, and to find the optimal conditions for the highest washing efficiency.

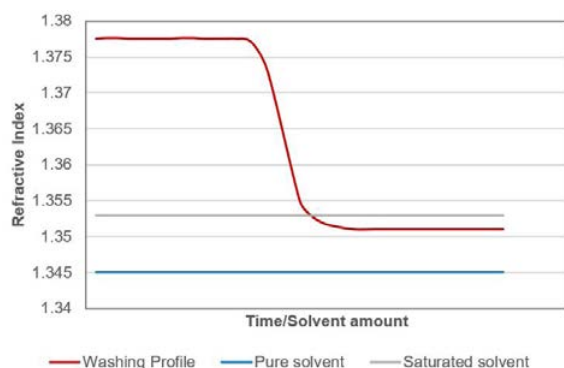
Refractive index has proven to be a valuable tool for the development, monitoring and control of cake washing operations. Refractive index measurement is used, for example, to understand the impact of variations on the process and on the product, to create washing profiles, and to find the most suitable washing solvent.

### Instrumentation and installation

Vaisala K-PATENTS® Pharma refractometer PR-43-PC provides an accurate refractive index measurement with excellent repeatability. The refractometer is ideal for the development and in-line monitoring of cake washing operations.

The optional Laboratory Test Cuvette (LTC) for the Pharma Refractometer offers the opportunity to use the same instrument throughout the entire development process. At laboratory scale, the refractive index of pure solvent and saturated solvents can be studied and used as reference to determine the optimal washing conditions. The refractometer can also be used to find the washing profile of the process, which

can be used as a *fingerprint* of the process during scale-up operations, and as a tool for troubleshooting (Figure 1).



**Figure 1.** Washing profile of an API crystal cake. After installation, the data from the refractometer reveals that the washing solvent and conditions are not optimal, which leads to reduced yield.




At pilot and full-scale, the refractometer is installed after the filter in the spent solvent line to monitor in real-time the refractive index of the liquid. Because each liquid has a different refractive index value, the

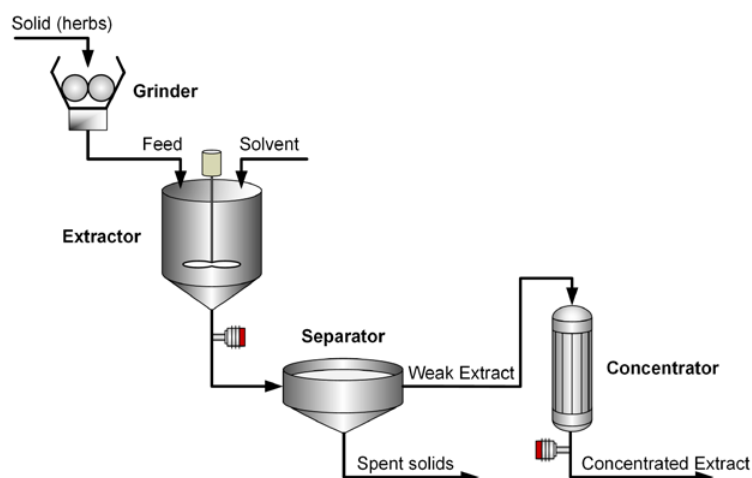
refractometer can detect immediately the interface between the mother liquor and washing solvent. This indicates the end-point of washing and reduces washing time and the amount of solvent used.

The refractometer is delivered factory calibrated and it does not require re-calibration. The measurement by the refractometer is not affected by bubbles or suspended particles, making it perfect for liquid-solid operations. Moreover, verification is easily performed with standard refractive index liquids.

When many instruments are required, the Multichannel User Interface (MI) connects up to 4 refractometers, thus reducing the investment cost. The MI provides user authentication, electronic records, data logging, event log and audit trail, all in compliance with pharmaceutical requirements.

The Pharma Refractometer is the ideal in line process analyzer in accordance with the PAT framework. Monitoring the filter cake washing operation with the refractometer helps to obtain a clean cake while meeting the economic, production and quality requirements of pharmaceutical processing.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
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User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

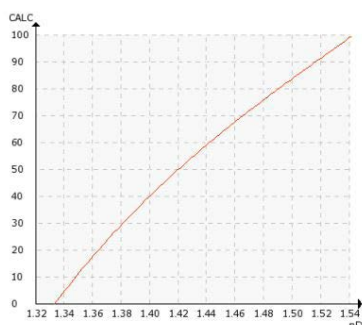


## ACTIVE PHARMACEUTICAL INGREDIENT IN SOLVENT, HERBAL EXTRACT, CHINESE HERBAL MEDICINE

### Typical end products

Chinese medicine, antibiotics and other active pharmaceutical ingredients and herbal-based medicines.

Chemical curve: R.I.per Brix at Ref. Temp. of 20°C



### Introduction

Natural substances such as herbs are an important source of raw materials for the pharmaceutical industry due to their special attributes. Good examples of final drugs are *herbal-based medicines* or *Traditional Chinese Medicines (TCM)*. These have been created and produced through thousands of years and have supported the development of modern pharmaceutical technology.

Nowadays, herbal-based medications can be prepared through a variety of operations, including liquid-solid extraction, distillation, pressing, purification, concentration and fermentation.

### Application

The main operation in the production of herbal-based medicines is extraction. This technique involves

solubilizing the *Active Pharmaceutical Ingredient (API)* and removing it with a suitable extraction solvent. Since the active material is present in the solid (e.g. plants and herbs), and it is extracted by using a liquid phase, the operation is called *solid-liquid extraction* or *leaching*.

The most widely adopted extraction methods are maceration, percolation and Soxhlet extraction. Extraction can be performed in a batch or continuous operation, and in one step with a single solvent or in multiple sequential steps with different solvents.

The process usually starts with cleaning and preparation of the solids. Solid-handling techniques are typically physical methods, such as grinding and milling, that increase surface contact between the solids and extraction solvents.

The treated solid material is then fed to the extraction column or vessel (extractor) together with the solvent. Since most APIs have some aqueous solubility, extraction is typically performed with water or a mixture of water and organic media. The extraction solvent is carefully chosen to present a high-solubilizing power and preferential dissolution for the desired ingredient. The process is carried out for a specified time, and usually aided by heating and agitation to increase extraction efficiency.

During or after extraction, the extract concentration is monitored for extraction efficiency. This is typically achieved by HPLC analysis, laboratory tests or hand-held refractometers. The concentration at the extraction point is usually around 10-15 Brix.

After the operation is completed, the leached or spent solids must be separated from the extract, for example, by settling followed by decantation, filters, centrifuges or thickeners.

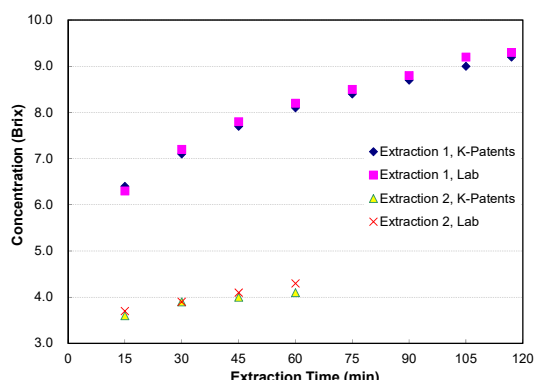
The extract moves on to downstream processing which usually includes a low temperature evaporation step to increase the concentration to 60-80 Brix. The solvent is recovered, and the spent solids may also be treated to recover any solvent imbibed in the slurry.

## Instrumentation and installation

Vaisala K-PATENTS® Pharma Process Refractometer PR-43-PC provides real-time information of the increasing dissolved API content in solvent (Figure 1). This is a required measurement to calculate and monitor extraction efficiency and mass balances, and to continuously optimize the process.

The refractometer is installed directly in the pipeline after the extractor or in a recirculation line. Another refractometer after the concentration step ensures the target concentration is always achieved. In-line measurements reduce the need for sampling and time-consuming laboratory tests and eliminates the risk for product contamination. Automatic prism wash might be required in installations at the evaporator if the process medium is not purified.

The refractometer is also a helpful tool for the development and scale-up of API production processes. The refractive index measurement by the refractometer is accurate, reliable and repeatable, providing the right basis for the development of efficient and cost-effective processes.





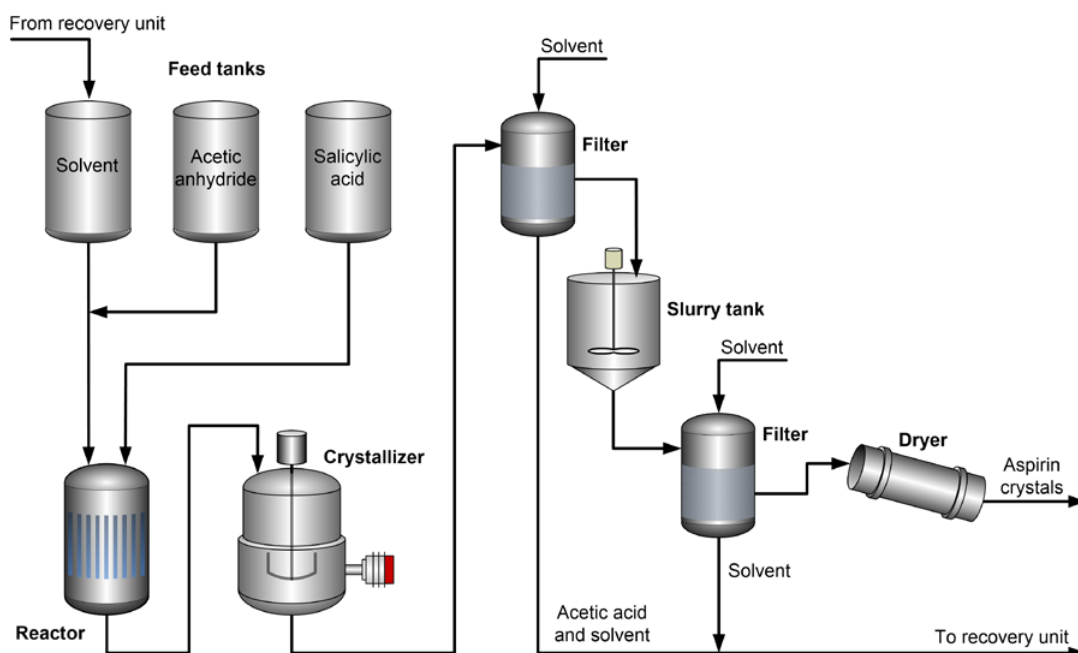
**Figure 1.** Measurement of the dissolved solute concentration during 2 extraction steps. The measurement by the refractometer matches closely the reference value from laboratory.

At the early stages, the refractometer can be used, for instance, to find the correct extraction solvent, residence time, and temperature in order to achieve an optimum way to operate and reduce batch-to-batch variations.

The refractometer is ideal for liquid-solid mass transfer operations as the measurement is not affected by suspended solids or air bubbles in the liquid. The refractometer measures selectively the dissolved material in the liquid phase.

Moreover, the Pharma Refractometer is designed to meet the strict requirements for pharmaceuticals processing and it's an ideal tool in accordance with the PAT framework. The refractometer withstands CIP and SIP operations and it is available with 3-A Sanitary and EHEDG certifications.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

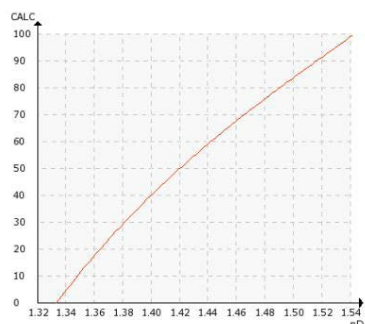


## ACETYSALICYLIC ACID (ASPIRIN)

### Typical end products

Aspirin.

Chemical curve: R.I. per Brix at Ref. Temp. of 20°C



### Introduction

Aspirin, also known as *acetylsalicylic acid*, is the most widely sold over-the-counter drug. The production process involves the reaction of salicylic acid and acetic anhydride in glass lined vessels. The liquid produced is pumped to a crystallizer, where it is cooled down to start the crystallization to Aspirin. The Aspirin is then filtered to remove the acetic acid and solvent, which are recovered and possibly recycled. The crystals are then washed and filtered again. The crystals are sent to sifting, granulating and tableting after drying.


### Application

Vaisala K-PATENTS® Process Refractometer PR-43-AP is highly effective in obtaining the selective measurement of the liquid phase in Aspirine crystallization. The refractometer gives an instant indication of liquid concentration drop, which happens dramatically when crystal formation starts. This allows consistent control of crystallization (see also application note *Pharmaceutical Crystallization*).

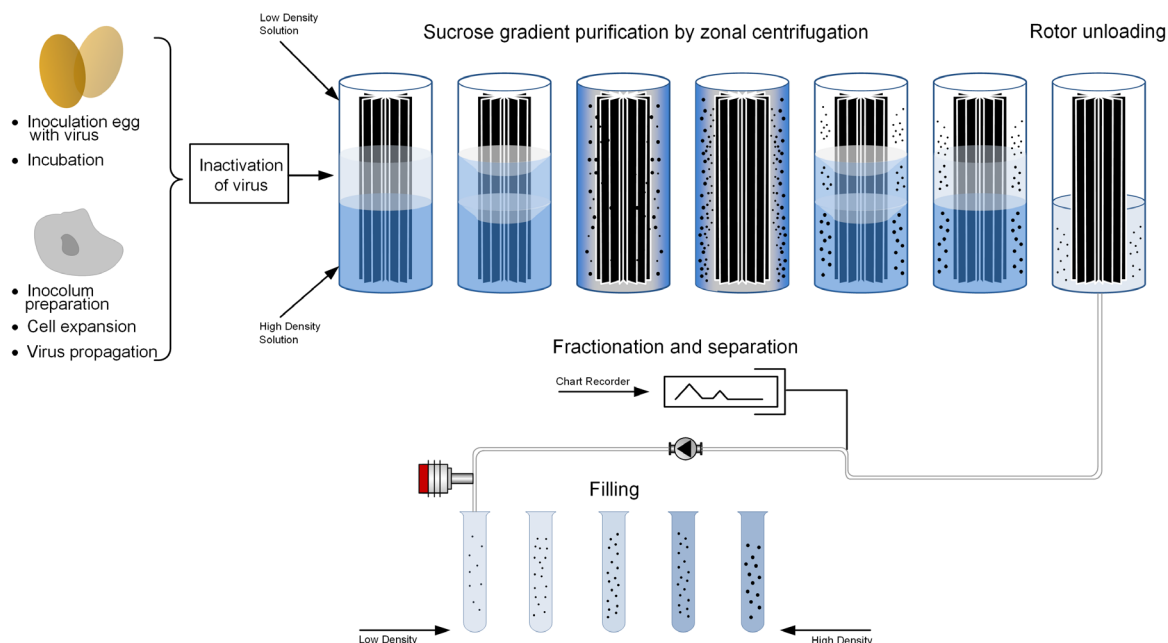
### Instrumentation and installation

The Process Refractometer is installed directly in the vacuum crystallizer. Due to its unique digital operation, the refractometer measures the true concentration of the mother liquor uninfluenced by Aspirin crystals or bubbles present in the pan. Appropriate equipment with hazardous and intrinsic safety approvals are available if required.

The Process Refractometer is an ideal real-time instrument, which complies with pharmaceutical industry standards and regulations, including PAT, GAMP, CIP/SIP, 21 CFR Part 11 and validation. Its ability to measure and control parameters, such as refractive index  $nD$ , contributes significantly to the development of effective drugs and manufacturing processes. The PR-43 refractometers comply with pharmaceutical drug production regulations for process wetted part materials, sealing and surface roughness.

Instrumentation	Description
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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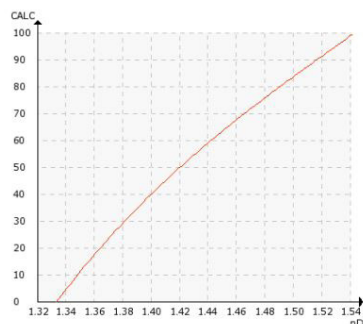


## SUCROSE SOLUTION

### Typical end products

Viral vaccines e.g. seasonal influenza, meningitis, rabies, hepatitis B, polio, measles, mumps, rubella and other vaccines.

Chemical curve: R.I.per Brix at Ref. Temp. of 20°C



### Introduction

Viral vaccines are used globally to protect humans against infections. Vaccines are made by first growing a virus in an egg-based or cell-based process. The virus is then inactivated or attenuated, concentrated and purified, before it is finally blended with other components for making the final vaccine formulation.

Purification is one of the most important steps in vaccine manufacturing, as it removes impurities originating from host cells or culture media and ensures the safety and efficiency of the final product. Common downstream processes for purification of the virus include density gradient ultracentrifugation,

size exclusion chromatography (SEC) and ion-exchange chromatography. The choice of purification technology depends on the type of virus to be purified and the source of the starting material. For example, the separation of influenza viruses in large scales is performed using continuous flow centrifugation of the fluid with a sucrose density gradient.

### Application

Sucrose density gradient has traditionally been used for the production of influenza vaccines, but it can also be used for the purification of other viruses such as Hepatitis B and Rabies. In sucrose gradient centrifugation the separation of the virus and possible impurities may be *rate-zonal* (based on particle size differences), *isopycnic* (based on the particle density differences), or a combination of both.

The concentration and purification of the virus takes place in a centrifuge (e.g a continuous flow ultracentrifuge). First the density gradient, consisting of sucrose solutions, is loaded into the rotor. The concentration range of the density gradient is selected so that it covers the particle of interest, in this case the virus. In the production of influenza vaccines sucrose solutions between 0 and 60% are typically used.

Next the rotor is accelerated. This rotation forms a density gradient in the form of bands in which the sucrose concentration goes from 0 to 60%. The



sample fluid containing the virus is then loaded into the rotor.

The viral particles in the sample move along the density gradient and separate out to form bands according to their sedimentation rates or differences in density. For instance, in isopycnic centrifugation the particles move to a position where their density is equal to that of the solution (buoyant density). This operation is known as *isopycnic banding* as the result is a solution with stable bands.

At the end of the run, the rotor is decelerated to rest and the bands are ready to be unloaded. The separation of different fractions can easily be done by measuring changes in refractive index or Brix with a process refractometer. The refractometer provides real-time readings that help to identify and collect the rich-virus fraction(s).

After the virus is purified, the manufacturing process continues with formulation, quality testing, filling and distribution.

## Instrumentation and installation

The Vaisala K-PATENTS® Pharma Refractometer PR-43-PC has been designed to meet the requirements and needs in biopharmaceuticals processing. In vaccines production, the accurate and repeatable measurement by the refractometer helps to isolate reliably the virus fraction in the purification step, ensuring a safe and high-quality vaccine product, while increasing yield and productivity.

The Vaisala pharma refractometer is installed directly in the unloading line from the vaccine fractionation unit for in-line process control. The output is Ethernet or 4 to 20 mA signals that can be easily configured to provide values of sucrose solution density, concentration, Brix or any other scale preferred by the manufacturer.

The measurement signal is used for reliable and timely determination of the product peak in the density gradient (0 to 60 % w/w sucrose), the subsequent collection of the virus rich fraction (Figure 1) and in diverting the virus rich fraction into the correct container. For instance, the output signal of the refractometer can be used as a relay to control the pneumatic actuator of diverting valve, or can be connected to the PLC for unmanned operation to pump product into one barrel and waste to another.

The Vaisala pharma refractometer is also a valuable PAT tool during research and development phase. The scalability feature of the refractometer helps researchers collect sufficient real-time data during

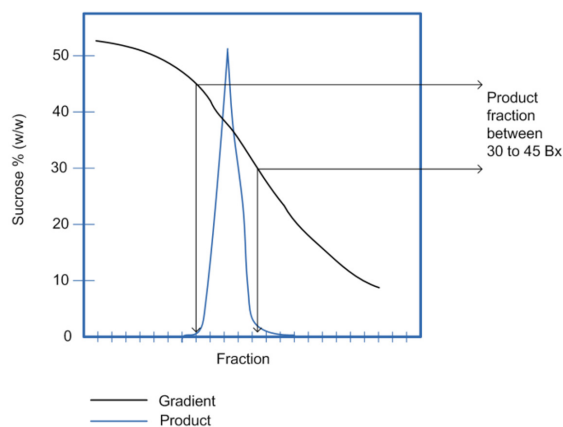


Figure 1. Collection of the virus rich fraction.



early development in the laboratory to gain process understanding. Refractive index measurements are useful to characterize the concentration (or density) of sucrose in each fraction as well as the concentration of the virus sample itself. The information helps to find banding patterns and optimizing the target fraction. Once purification protocols has been developed, the same pharma refractometer can be used in full-scale production for determining the moment when to begin and to stop collecting the target fraction.

The typical system comprises the Vaisala Pharma Refractometer PR-43-PC with a Pharma Mini Flow Cell PMFC that allows the refractometer connection to centrifuge rotor unloading and fractionation phase. The Pharma Mini Flow Cell is ideal for ensuring reliability of the measurement even at small volumes. The standard Ethernet communication solution allows for simultaneous data logging and continuous monitoring of the measurement values and diagnostics by computer via an Ethernet connection.

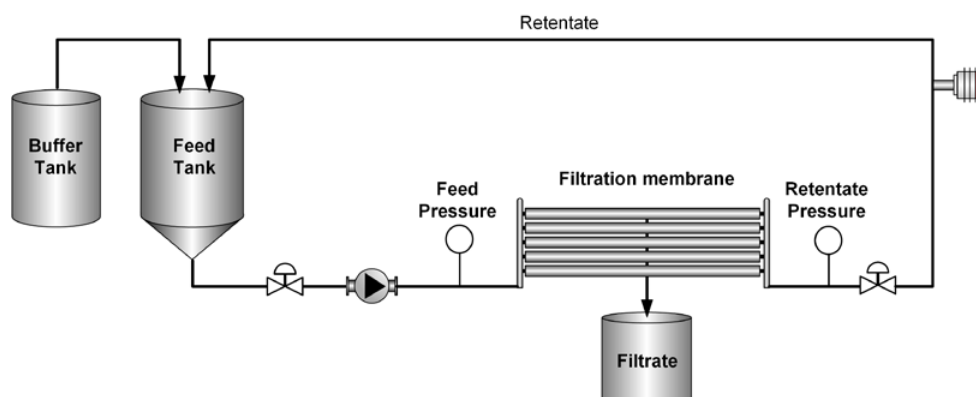
Due to its unique digital sensing technology, the measurement by the refractometer is accurate and does not drift in the presence of bubbles or suspended particles. The refractometer is delivered factory calibrated and does not require re-calibration. Moreover, verification is easily performed using standard refractive index liquids.

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Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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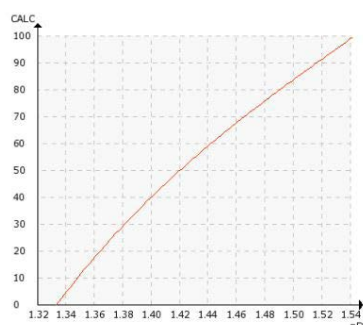


## PROTEIN RETENTATE

### Typical end products

Concentrated and filtered protein.

Chemical curve: R.I.per Brix at Ref. Temp. of 20°C



filtration process the buffer is added into the feed tank at the same flow rate as the filtrate is removed. This allows the volume of the fluid in the process to be kept constant while the smaller molecules pass through the filter and are washed away. Then the retained protein retentate proceeds to the feed tank for further circulation.

It is important to control the retentate concentration as it affects the permeability of the smaller molecules through the membranes. The permeability of a molecule through a membrane is determined by measuring the concentration in the filtrate compared to the concentration in the retentate under specified conditions.

## Introduction

Filtration is a technique using ultrafiltration (UF) membranes to separate the components of solutions and suspensions based on their molecular size, although other factors such as molecular shape and charge can also play a role.

In the UF membrane, molecules larger than the membrane pores will be retained at the surface of the membrane, while solvent and smaller solute molecules will pass freely. This process of molecular exclusion at the UF membrane surface leads to concentration of the protein solute in the retained fraction. The protein solute (retentate) can then be recovered from above the membrane.

## Application

Purified protein solution from the feed tank is pumped to the membranes for filtration. Pressure is applied to force the feed against the membranes. At this point, small molecules are filtered and pass through the membrane pores, while large molecules form the concentrated protein retentate. In a continuous

Depending on the required concentration of biomolecules in the retentate, the process can be either a batch process or a continuous process.

## Instrumentation and installation

Vaisala K-PATENTS® Pharma Refractometer PR-43-PC provides an accurate real-time measurement of the protein concentration. The refractometer aids in controlling and adjusting concentration levels after ultrafiltration.

Refractive index technology is used to replace traditional off-line A280 measurements and to determine highly concentrated protein solutions. The refractometer measures the protein concentration in-line at the manufacturing scale, thus ensuring the capability of reading index values in real-time conditions that correspond to high protein concentrations (>100 g/l). The refractometer can detect inadequate mixing of highly viscous process streams over time through trending process data.

The 4 to 20mA and Ethernet output signals can be connected directly to Data Acquisition Systems allowing for real-time protein concentration monitoring.



Typical installation of the Pharma Refractometer is on a pipe bend. It is either angle mounted directly on the outer radius of the pipe bend or by using a flow cell. Self-cleaning effect can be achieved with proper installation and the best flow conditions.

Prism wash is usually not required in this application, since most UF facilities perform CIP cleaning every 10-20 hours.

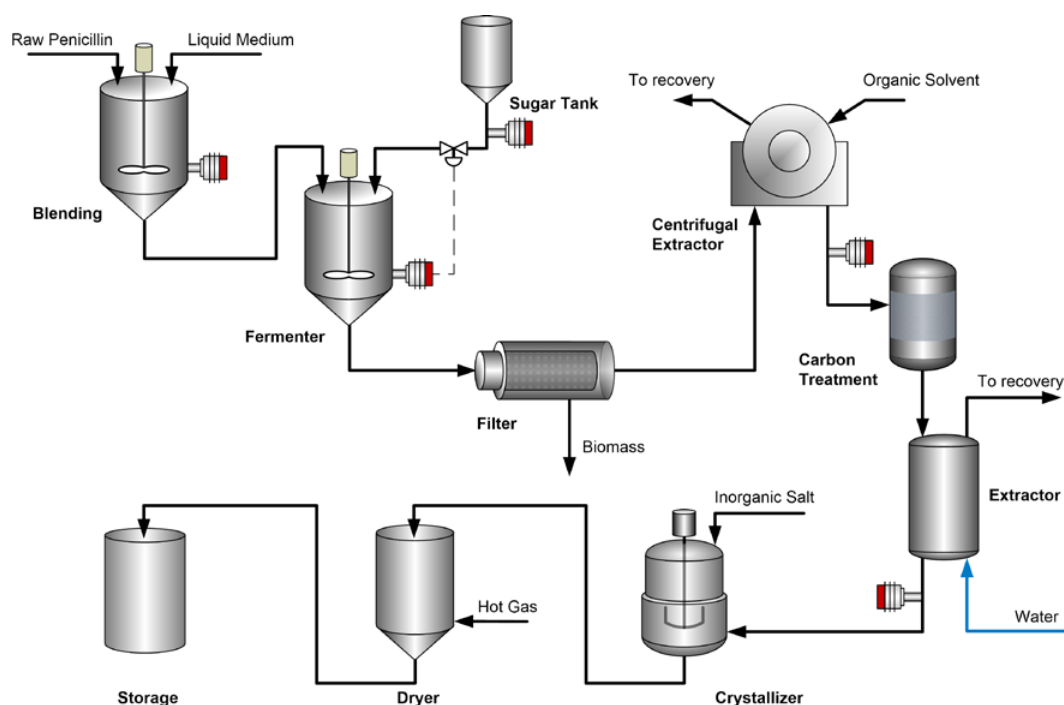
Due to its unique digital sensing technology, the refractometer is accurate and does not drift in the presence of bubbles or suspended particles. The

refractometer is delivered factory calibrated and does not require re-calibration. Moreover, verification is easily performed using standard refractive index liquids.

The high accuracy control achieved with the precise in-line concentration measurements improves end-product quality and reduces operating costs.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix

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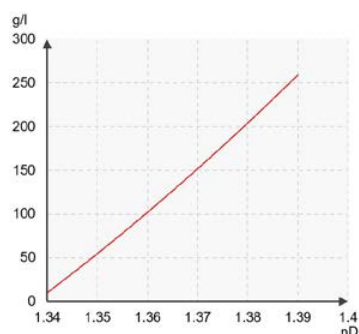


## PENICILLIN IN WATER, PENICILLIN IN SOLVENT

### Typical end products

Penicillin group antibiotics such as Penicillin V, Penicillin O, Ampicillin and Amoxycillin.

Chemical curve: Penicillin G R.I. per g/l in solvent at Ref. Temp. of 20°C



### Introduction

Penicillin is the oldest known group of antibiotics. It is widely used because of its effectiveness against bacteria (e.g. staphylococci and streptococci), which makes it the preferred group of antibiotics for treating bacterial infections such as skin, chest and urinary tract infections.

### Application

The Penicillin production process starts with the preparation of an inoculum of a high yielding strain of *Penicillin Chrysogenum*. This is mixed in a medium

designed to provide the organism with all the nutrients that it requires. The medium contains carbon and nitrogen sources such as glucose, corn steep liquor, lactose, and ammonium sulfate. At the penicillin inlet to the mixing buffer vessel the concentration can range from 180 to 250 g/l.

After the liquor has been mixed, it proceeds to the fermenter, where the medium for fermentation is continuously controlled and maintained by the addition of sugar to ensure a high yield of penicillin. After a certain time, the broth containing about 1 % penicillin goes on to extraction and purification.

The biomass is filtered out for disposal, and the penicillin is extracted from the solution by adding an organic solvent. Common solvents used are n butyl acetate or Methyl Isobutyl Ketone (MIBK). The penicillin containing solvent is treated with activated carbon to remove other impurities, and the penicillin is recovered by precipitation with an inorganic salt.

The sodium or potassium salts of penicillin are further processed to remove impurities. After crystallization and drying, the penicillin salt is ready for storage or for further processing to produce other semi-synthetic antibiotics.

## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43 is designed for aseptic environments and the accurate and precise control required by the Pharmaceutical Industry.

The refractometer is installed at the bottom of the mixing buffer vessel to measure the concentration of raw penicillin.

Another refractometer at the fermenter monitors continuously the fermentation process and maintains the optimal conditions by controlling the sugar feed valve (see application note *Pharmaceutical Fermentation*). This ensures a high yield of penicillin. The refractometer can also monitor the concentration of sugar fed to the fermenter.




Continuous and accurate measurements also ensure high recovery rate of the penicillin ingredient and the organic solvent used for extraction.

The refractometer eliminates the need for manual sampling and reduces the risk for contamination. This provides a tighter control of the product quality.

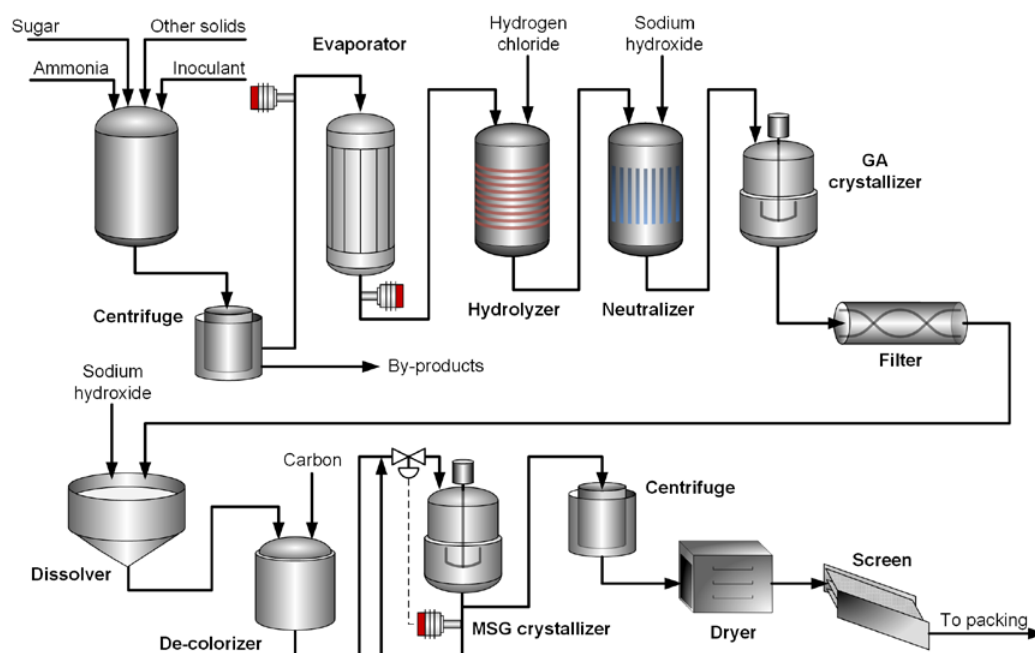
Moreover, the Refractometer withstands CIP and SIP, and is available with 3-A Sanitary and EHEDG certifications.

Due to its unique digital sensing technology, the refractometer is accurate and does not drift in the presence of bubbles or suspended particles. The refractometer is delivered factory calibrated and does not require re-calibration. Moreover, verification is easily performed using standard refractive index liquids.

When many instruments are required, the Multichannel User Interface (MI) connects up to 4 refractometers, thus reducing the investment cost. The MI provides user authentication, electronic records, data logging, event log and audit trail, all in compliance with pharmaceutical requirements.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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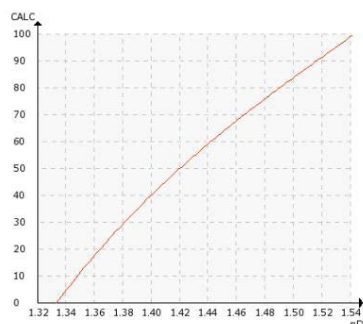


## MONOSODIUM GLUTAMATE, MSG ( $C_5H_8NNaO_4 \cdot H_2O$ )

### Typical end products

MSG for barbecue sauce, salad dressings, snack food, tortilla chips, seasoning mixtures etc.

### Chemical curve: R.I.per Brix at Ref. Temp. of 20 °C



## Introduction

Monosodium glutamate, more commonly referred to as *MSG*, is a sodium salt ( $C_5H_8NNaO_4$ ) of the non-essential amino acid glutamic acid. It is used to intensify the natural flavor of meats and vegetables.

## Application

Several U.S. companies have developed their own microbiological process, others have obtained it from the Japanese. The principal process steps are fermentation, concentration, hydrolysis, neutralization and acidification, crystallization, separation and purification.

One of the most important areas of concern for end-product quality control is crystallization.


The principle measurement is total density, which is measured at the crystallizer outlet. This is used to regulate the flow of fresh liquor into the crystallizer circulation, thereby keeping the supersaturation levels within specifications. This continuous monitoring maintains process consistency.

## Instrumentation and installation

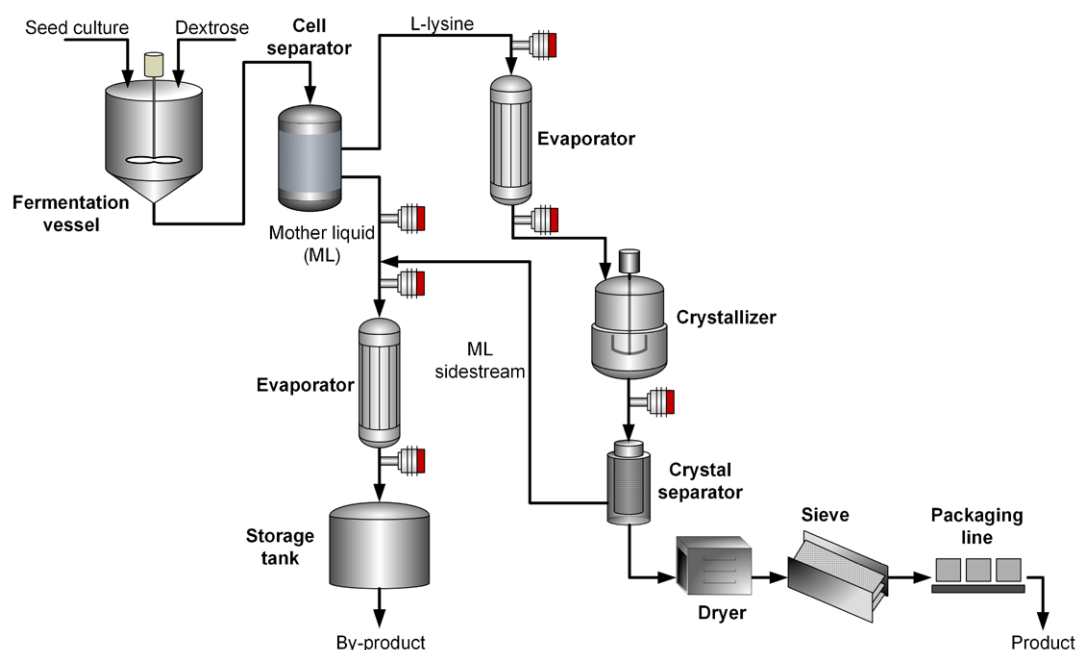
Vaisala K-PATENTS® Sanitary Probe Refractometer PR-43-AP is installed directly in the crystallizer or between the outlet and the heat-exchanger in the crystallizer vessel recirculation-pipe.

The refractometer can also be installed in the feed and outlet lines of the evaporator. The refractometer provides 4-20 mA and Ethernet output signals that can be used for automatic control of the evaporation step to achieve the target concentration and to reduce energy consumption.

The typical measurement range of MSG is 45-65 Brix and the process temperature is about 60 °C (140 °F).

Instrumentation	Description
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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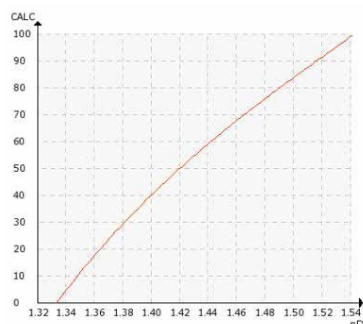


## L-LYSINE

### Typical end products

Feed additive, food material, health supplements.

Chemical curve: R.I.per Brix at Ref. Temp. of 20°C



## Introduction

L-lysine is an amino acid essential for human and animal nutrition. It has a variety of industrial uses, including as a medicament, chemical agent, food material and feed additive.

## Application

L-lysine is manufactured by microbial fermentation in bioreactors. The choice of raw materials for amino acid fermentation is geographically specific: sugar, molasses, dextrose/ glucose or starch as the carbohydrate source are added to the fermentation reactor along with seed culture as feed stock, for example, corn. Corn provides minerals and

growth factors needed for lysine production. During fermentation period the temperature is about 30 to 35 °C (86 to 95 °F).

The lysine accumulates in the fermentation broth as an aqueous solution. After fermentation the lysine feed is separated into lysine broth and mother liquid by-product. The lysine feed is then concentrated by evaporation. The evaporation process is controlled using Vaisala K-PATENTS® Sanitary Compact Refractometer PR-43-AC to ensure a precise concentration level is reached.

The concentrated lysine material is then purified by crystallization to produce higher grades of lysine. The lysine content is typically above 60 % on dry matter. Control over the required grade of lysine after crystallization and before drying is performed with a Process Refractometer PR-43-GP. The product then goes through the sieve to packaging after which it is ready for shipment.

The by-product from lysine broth is concentrated and stored in tanks before shipment. The Process Refractometer PR-43-AC controls the mother liquid feed during the concentration, and Process Refractometer PR-43-GP is used to ensure the required concentration of the product before storage.



## Instrumentation and installation

In the L-lysine production process there are several points where Process Refractometer measurements are used for process control:

### In L-lysine line:

1. Monitoring of the fermentation reaction (see also application note *Pharmaceutical Fermentation*).
2. Measurement of the concentration at the evaporation stage, range 0-100 Brix.
3. Lysine crystallization (see also application note *Pharmaceutical Crystallization*), range 0-100 Brix.



### In by-product line:

4. Mother liquor feed concentration measurement before it is concentrated in the reactor.
5. By-product concentration measurement after the mother liquor side stream has been added to the process before entering the reactor.
6. Final by-product concentration measurement before storage.

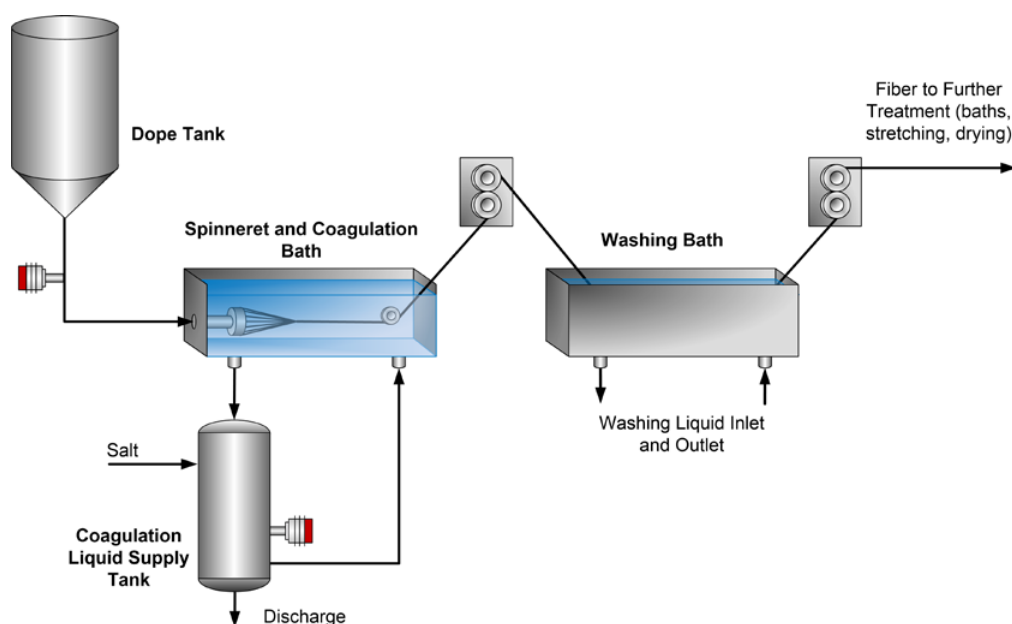
The Sanitary Compact Refractometer PR-43-AC and Process Refractometer PR-43-GP are ideally suited for process control and monitoring in the L-lysine production process.

Due to its unique digital sensing technology, the refractometer is accurate and does not drift in the presence of bubbles or suspended particles. The refractometer is delivered factory calibrated and does not require re-calibration. Moreover, verification is easily performed using standard refractive index liquids.

Concentration monitoring with the refractometer ensures process efficiency, maximizes productivity and reduces operational costs.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	<b>Prism wash with steam.</b> The components of a steam wash system are a sensor with integral steam nozzle mounted at the sensor head, a shut-off valve for steam line and an indicating transmitter equipped with relays to drive the wash valves.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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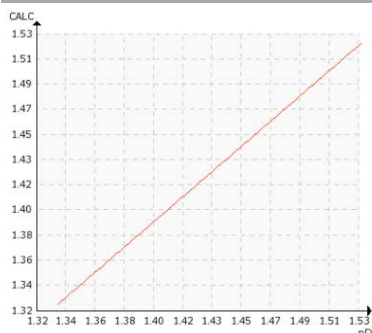


## SODIUM ALGINATE, $\text{NaC}_6\text{H}_7\text{O}_6$ , CALCIUM CHLORIDE, $\text{CaCl}_2$

### Typical end products

Alginate fibers, such as sodium alginate, calcium alginate or hybrids for the production of wound dressings and medical fabrics.

### Chemical curve: R.I. at Ref. Temp. of 25°C



## Introduction

Alginates are biopolymers extracted from brown algae species such as seaweed. They have a variety of uses in different industries because of their unique properties. These materials have been extensively used in pharmaceutical applications as they are gel forming, non-toxic and highly absorbent.

Fibers made of alginate have become very popular for the production of wound dressings. These fibers are biocompatible, have hemostatic properties and accelerate the healing process by creating a gel that keeps a moist interface on the surface of the wound. The composition of the fibers can be modified by a controlled process to enhance their hemostatic

properties and obtain fibers with additional healing effects.

## Application

Alginate fibers are produced by a *wet-spinning process*. It is called wet-spinning because the fibers are extruded directly into a solution or bath. The bath is usually a salt solution or a mixture of salts, containing metal ions, but it can also be an inorganic acid solution or an organic solvent depending on the desired final product. The most common salt is calcium chloride, but salts containing zinc, silver, and other bioactive additives, beneficial for wound healing, are also used.

For the production of the fibers, a *spinning dope* or *spinning solution* is prepared by mixing sodium alginate with water, to form a homogenous solution. The concentration ranges between 5 and 10 %. This solution is then spun directly in the coagulation bath through a spinneret or a nozzle, to convert the solution into fibers. As the sodium alginate contacts the salt bath, water is removed from the formed fiber leaving behind only the biopolymer (alginate).

Coagulation happens when the sodium ions come into contact with the polyvalent ions of the bath (e.g.  $\text{Ca}^{2+}$ ). The sodium ions exchange places with the calcium ions to form calcium alginate, which is not soluble in water. The resulting fiber is washed, stretched and dried to obtain the final product. Additional baths can be used to alter the composition of the fiber and obtain additional wound healing properties.

The concentration of both, the solution dope and the coagulation bath, play an important role in the final product quality. If the concentration of sodium alginate in the solution dope falls too low neither coagulation nor formation of the alginate filament will take place. In addition, the hemostatic property depends on the concentration of the alginate.

As sodium alginate is extruded, the coagulation bath inevitably gets diluted. The concentration of the bath should also be monitored as the morphological structure of the fibers is affected by the composition of the salt.

## Instrumentation and installation



Vaisala K-PATENTS® Sanitary Refractometer PR-43-A provides real-time, accurate and reliable concentration measurement for ensuring the highest quality, purity and consistency in pharmaceutical applications.

A refractometer is installed directly on the filling line to measure continuously and precisely the concentration of sodium alginate solution pumped into the bath. The

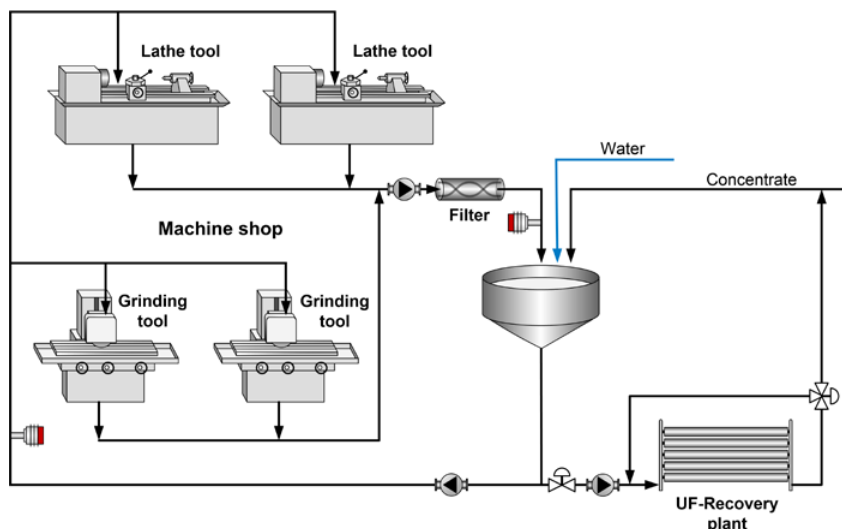
concentration of the dope solution ranges between 5 and 10 %, at a temperature of 35-50 °C (95-122 °F). A second refractometer monitors the concentration of the coagulation liquid as water content builds up. To ensure a high product quality, the water content should be kept under 20 %.

The refractometer provides Ethernet or 4-20 mA output signals for real-time process control. The concentration of the bath can be controlled and kept at its ideal value by a circulation system where more coagulation agent is added to restore the concentration.

The PR-43-A refractometer is designed to meet all the pharmaceutical industry standards and regulations, and it is the ideal in-line process instrument for the Process Analytical technology (PAT) framework.

Instrumentation	Description
	Pharma refractometer PR-43-PC for hygienic installations. The PR-43-PC is installed in the main processing line or vessel and no by-pass arrangements are required. Optional laboratory test cuvette (LTC) for off-line laboratory testing and validation.
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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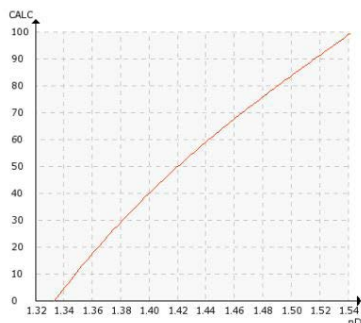


## HYDRAULIC FLUIDS, CUTTING OILS, COOLING LUBRICANTS, WASHING LIQUIDS

### Typical end products

Metal components and parts e.g. engines, drill bits, washers, beverage cans, steel coils, turbine blades, knives, blades, scissors, bearings etc.

### Chemical curve: R.I. per Brix at Ref. Temp. of 20 °C



### Introduction

Metal machining is the process of converting metal blanks into finished components or parts. All metalworking operations involve the contact of two solids, a tool and the work-piece, to create the finished piece. The process involves high friction, high temperatures and high stresses, resulting in tool wear. A *lubricant* or a *metalworking fluid* is used to minimize these effects. Metalworking fluids accomplish this by providing cooling, lubrication and corrosion resistance.

Over time, cutting fluids can become contaminated by chips and fines, tramp oil, bacteria and dissolved salts. Therefore, monitoring the pH, water hardness and fluid concentration is essential in preventing failure of the fluid. Water evaporation should be monitored to ensure the coolant-to-water ratio is

correctly maintained. Vaisala K-PATENTS® Process Refractometer provides accurate and continuous measurement of the coolant-to-water ratio.

Traditional sampling method and analysis by titration is time consuming. Sampling can be minimized with in-line Process Refractometer control, especially when there are multiple stations. Furthermore, portable handheld refractometers are not ideal tools for metalworking fluids, as they give a fuzzy borderline resulting in inaccurate measurements.

Too low a concentration allows bacterial growth, reduces cutting capacity resulting in longer machining time, poor surface finish, ineffective lubrication and debris to tool welding. Too high a concentration means excessive use of lubricant (costly) and grease contamination (difficult to remove).

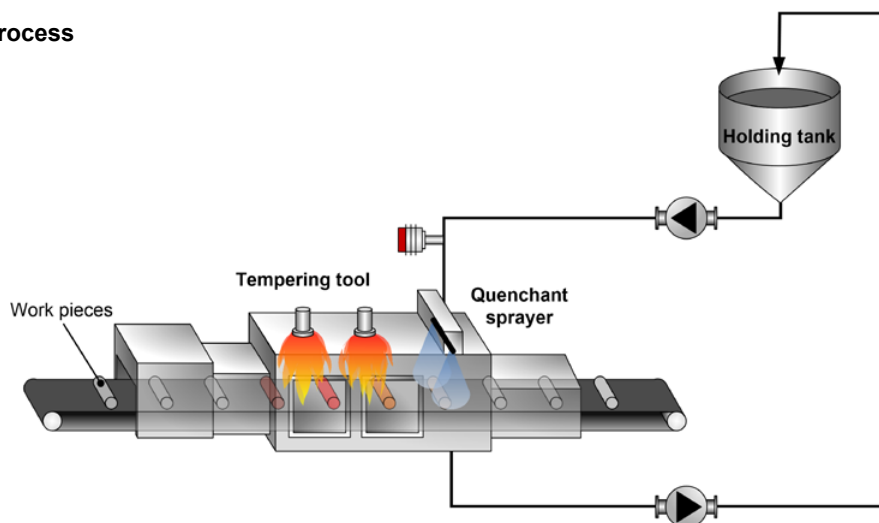
### Application

The Process Refractometer applications can be divided into four areas:

#### 1. Quenching/Tempering Process

The physical property of the metal is changed through quenching. The metal is heat-treated and then cooled rapidly (quenched) with a fluid in order to increase the surface hardness. The concentration of the fluid is one parameter, which affects the final quality. Fluids are also used in the casting process. When the concentration levels are correct, the surface properties of the finished parts are within the specifications.

## Quenching Process



## 2. Cooling/Cutting

During machining, the metal has to be cooled and lubricated. Coolants are often supplied through a centralized system but also by local systems at the point of use. The refractometer is used in both the feed and recycling lines serving machine tools.

## 3. Washing

The primary goal of washing is to clean the machine parts thoroughly of residual oil and dirt. The washing fluid is sprayed onto the parts at high pressure. The fluid forms a film that protects the parts from corrosion during storage.

The refractometer detects the correct concentration to ensure that the excess grease and oil have been completely removed by the washing process.



## 4. Rinsing

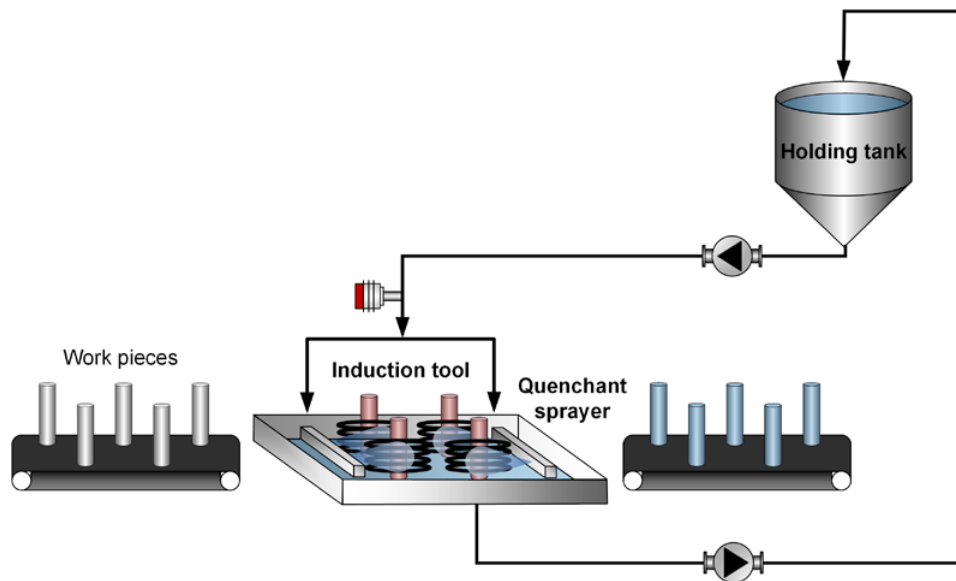
If the parts and components are destined for welding, the protective film must be completely removed as residual wash solution affects the quality of welds. The film is removed in water rinsing baths.

As the water picks up the residual wash liquid, the bath must be replenished as the concentration value reaches its saturation limit. The acceptable limit varies from plant to plant, but typically it is around 0.5 Brix.

## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Probe Refractometer PR-43-AP is ideal for installation in larger pipes or vessels. The Sanitary Compact Refractometer PR-43-AC with a flow-cell is suitable for smaller pipes. Automatic prism wash with steam is recommended in cases, when dirty liquids are being processed.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
	Sanitary Probe Refractometer PR-43-AP for hygienic installations in large pipes, tanks, cookers, crystallizers and kettles and for higher temperatures up to 150°C (300 °F). The PR-43-AP refractometer is installed in the pipe line or vessel through a 2.5 inch or 4 inch Sanitary clamp, I-clamp, APV Tank bottom flange or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % Brix.

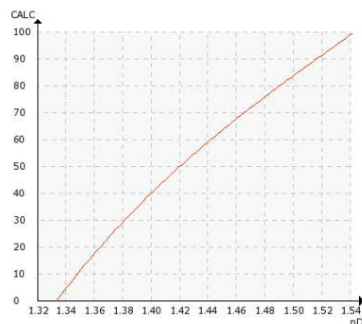


## POLYALKYLENE GLYCOL (PAG), PAG QUENCHANT

### Typical end products

Metal components and parts e.g. engines, drill bits, washers, steel coils, turbine blades, knives, blades, scissors, bearings, etc.

Chemical curve: R.I.per Brix at Ref. Temp. of 20°C



### Introduction

Metal parts often require heat treatment to obtain improved mechanical properties. A common method used is *induction hardening*, in which induced heat is combined with rapid cooling (also known as *quenching*) to increase the hardness and durability of the steel.

Heating by induction is preferred because only the part to be hardened is heated. This is a flame-free, non-contact process that quickly produces intense, localized and controllable heat. Metal induction is used to harden numerous components such as gears, power/transmission parts, crankshafts, driveshafts, torsion bars, valves, rock drills and slewing rings.

### Application

Induction heating tools usually treat one workpiece at the time. The metal part is placed inside a coil (inductor) which generates a magnetic field that results in an induced alternating current in the workpiece. The coil does not touch the piece, and heat is only generated by the induced current flowing into the surface of the workpiece.

The necessary cooling is achieved by quenching. Rapid quenching is performed by spraying the workpiece with a suitable liquid medium. The quenched metal undergoes a martensitic transformation (creation of a very hard form of steel crystalline structure), increasing the hardness and brittleness of the part.

There are a number of quenchants that are used with induction heating and they are selected according to the materials being processed. Polyalkylene Glycol (PAG) is commonly preferred because it exhibits inverse solubility in water, providing great flexibility of cooling rate.

The efficiency of the of the quenching system is dependent on the percentage of PAG in the water. The greater the polymer concentration, the slower the quenching action and the lower the heat removal rate. Since cooling the heated workpieces causes the water to evaporate, the concentration of the quenchant must be constantly monitored. In-line measurement of the quenchant concentration is necessary for automated control to maintain the polymer concentration at the desired level and to optimize the hardening process.

## Instrumentation and installation

Vaisala K-PATENTS® Sanitary Process Refractometer PR-43-AC is installed directly in the pipe before the induction tool to monitor in real-time the concentration of quenchant. Alternatively, the refractometer can be installed in a circulation line from the quenchant storage tank.

The measurement signal by the refractometer is used to ensure the quenchant has the correct concentration before it enters the sprayer. When the concentration levels are correct, the surface properties of the finished parts are within the specifications.

The refractometer's signal can also be connected to a process controller for continuous monitoring of the system and automated control over the concentration replenishment process.


Traditionally, handheld or laboratory Brix refractometers indicating sucrose content on a Brix scale (symbol °Bx), have been used for monitoring PAG quenchant concentrations. The refractometer

provides this same measurement capability in real-time.

The Brix reading can be converted into the quenchant concentration using a specific conversion factor for the quenchant being measured. The factor is typically from 2 to 2.5 for new PAG solutions and is typically provided by the manufacturer of the quenchant.

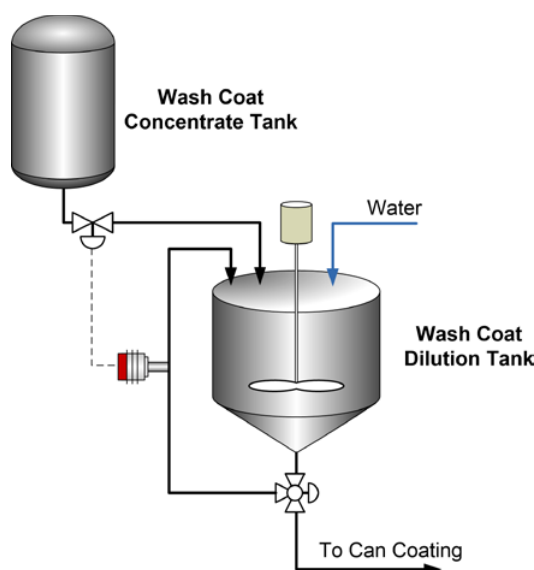
The refractometer is factory calibrated and delivered for PAG applications with a Brix calibration, which is identical to the typical hand-held or lab refractometer. The measured values can be displayed either using sucrose Brix scale or the specific quenchant concentration scale, where the Brix values are multiplied by the conversion factor for the quenchant (see Technical Note 7.01.01 PR-23 Calibration for PAG Quenchants).

Due to its unique digital sensing technology, the measurement by the refractometer is not influenced by suspended particles or bubbles.

Instrumentation	Description
	Sanitary Compact Refractometer PR-43-AC for hygienic installations in small pipe line sizes of 2.5 inch and smaller. The PR-43-AC refractometer is installed in the pipe bend. It is angle mounted on the outer corner of the pipe bend directly, or by a flow cell using a 3A Sanitary clamp, I-clamp or Varinline® connection.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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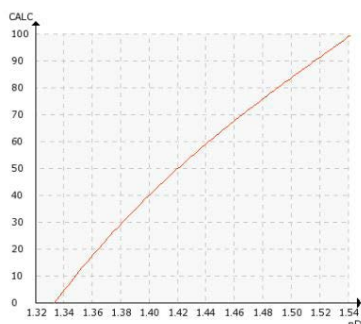


## WASH COAT MATERIAL

### Typical end products

Can Coating.

Chemical curve: R.I.per Brix at Ref. Temp. of 20°C



## Introduction

Canning of foods and beverages is currently considered to be a principle method of food preservation and storage. Thus, it is very important to keep up with protective properties that enhance the performance of cans. In can coating technologies a wash coat is used to provide high chemical resistance, hardness, flexibility and gloss of can caps and closures.

## Application

The wash coat is prepared by diluting an acrylic substance in water. The mixture is applied to the outside of the cans in order to protect them from oxidation before the labels are applied.

For the preparation of a wash coat, the wash coat concentrate is diluted with water in a mixing tank to

a certain concentration. It is important to monitor the wash coat concentration to ensure a high quality coat, good anti-corrosion properties and alkali-resistance, good adhesion properties for labels, and better storage stability.


## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GC is installed in the wash coat dilution tank to monitor continuously the concentration of wash coat material. The refractometer's output signal can be used for automatic control of the dilution operation. The refractometer controls the concentrate feed valve to adjust the amount added to the dilution tank and to ensure the target concentration is always achieved.

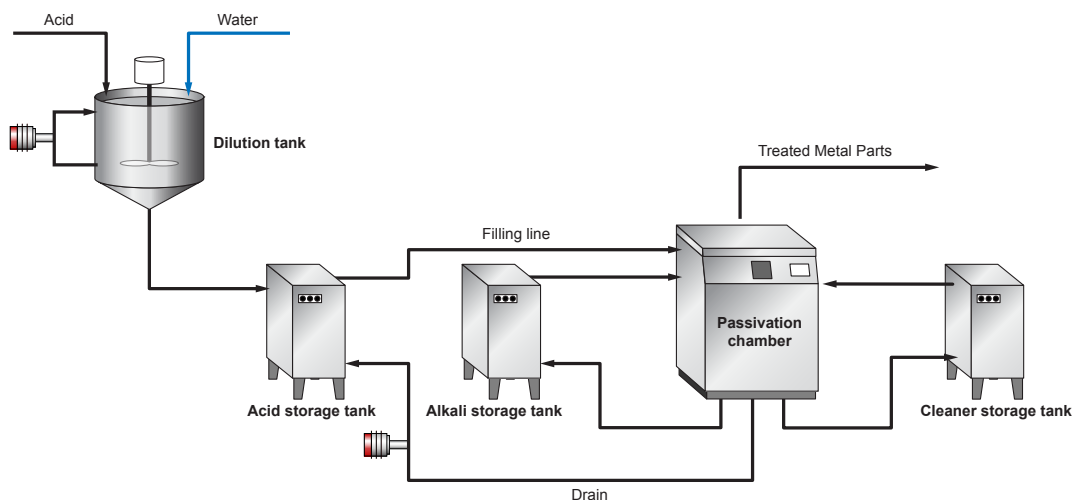
Dilution control with the refractometer guarantees a consistent quality in the application of material and precise film weight of coating. Moreover, it ensures an economical use of coating material, and increases safety in the plant by eliminating handling of the material by the operators.

The Refractometer is not affected by bubbles and is maintenance-free. The instrument's solid CORE-optics module is not affected by vibration and does not drift in the installation point, thus, providing accurate concentration measurement.



Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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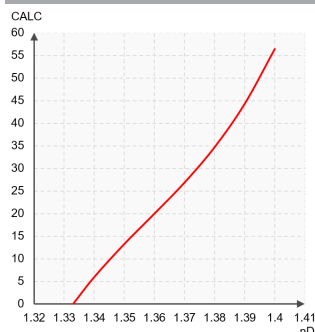


## NITRIC ACID $\text{HNO}_3$ , CITRIC ACID $\text{C}_6\text{H}_8\text{O}_7$

### Typical end products

Stainless steel metal parts for different applications.

Chemical curve: Nitric acid R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Stainless steel (SS) is a steel alloy containing at least 11% chromium. It is well-known for its corrosion resistance properties which derive from its chromium content.

The chromium, in the presence of air (oxygen), forms an inert layer of chromium oxide that covers the surface of the stainless steel and makes it less likely to react with air and cause corrosion. Ideally, a thick oxide layer is formed completely when the pure, cleaned SS is exposed to oxygen. In practice, foreign material on the surface from previous treatments (e.g. welding or grinding) can inhibit the formation of the oxide film which protects SS from corrosion. Therefore, these contaminants need to be removed in order to improve the corrosion resistance properties by a process known as *passivation*.

### Application

The passivation process takes place in a passivation chamber, where the metal parts are treated with different liquids in a series of steps. The first step is always cleaning. Cleaning is important as it removes grease and dirt from the parts, ensuring the acid reaches the entire SS surface. Cleaning is done with a detergent specifically chosen depending on the contaminants.

After cleaning the SS parts are ready for the passivating acid bath. Passivation ideally is done by submerging the metal parts completely in a nitric or citric acid solution. There are different passivation standards (ASTM A967, ASTM A380) and federal specifications (QQ-P-35C) that provide guidance on the proper methods to passivate stainless steel, including composition and concentration of the passivation bath solutions. Some industries also may have their own specifications. For instance, the medical device and aerospace industries require that SS parts are passivated only with a nitric acid solution.

Passivation with nitric acid requires 25-50% concentrations, while with citric acid the concentration is typically 5-15%. During passivation, manufacturers need to carefully control the concentration of the bath as its acid content is reduced gradually during the treatment of the metal parts. Contamination of the acid baths can also change the concentration and cause *flash attacks*, creating an undesirable heavily etched or darkened surface. Continuous monitoring of the concentration and other important parameters such as temperature and immersion time will ensure that the passivated surfaces meet the final requirements for quality.

The passivation acid solutions are usually prepared on site, by diluting the high purity chemical with water. The acid solutions are stored in individual tanks, from where they are pumped into the passivation chamber. The chamber is also connected to other tanks, which supply different cleaning solutions for final finishing of the stainless steel parts.

After passivation the parts are rinsed thoroughly. Rinsing can involve many steps, for example one neutralization step with an alkali (e.g. sodium hydroxide) or post-treatment (typically sodium dichromate), but always consists of a final rinse with clean water.

Finally, the liquid is drained, the parts are dried and tested according to standard specifications. If the passivated stainless steel does not pass the specification tests, it needs to be re-treated.

## Instrumentation and installation

Passivation processes require regular solution monitoring to ensure the acid baths are within the recommended concentration and successful passivation of the SS parts. In-line concentration measurement of the acid bath with the Vaisala K-PATENTS® Teflon Body Refractometer is a safe, reliable and quick method to monitor that the passivation is performed at all times within the recipe specifications.



The Vaisala K-PATENTS Teflon Body Refractometer is designed specifically for measurement of chemically aggressive solutions, ensuring complete compatibility with the acid and long-term performance. The

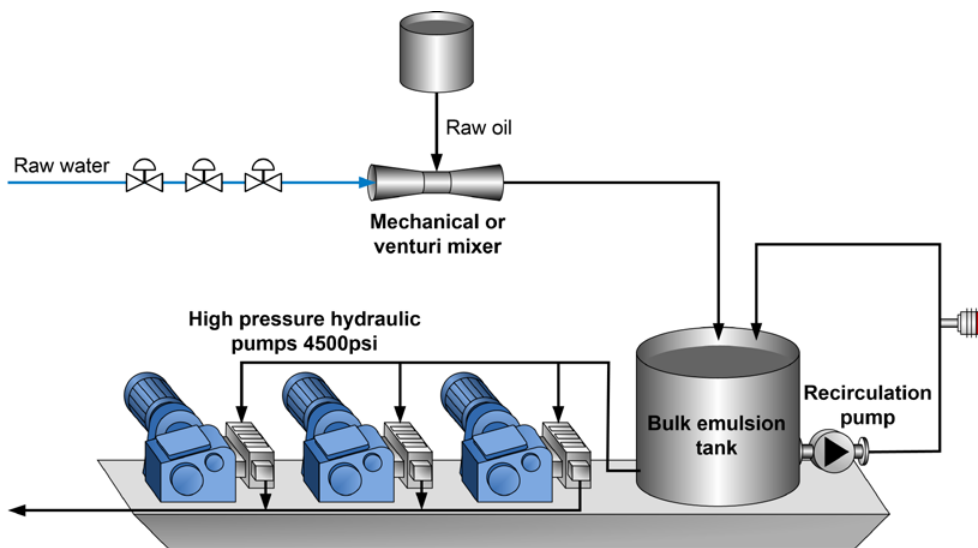
refractometer has a built-in flow cell made of non-metallic materials that ensure there is no contamination to the solutions. Moreover, the design of the Vaisala refractometer has a compact size, making it easy for its installation in the small piping of passivation units. If stainless steel or metal wetted-parts are preferred, the Vaisala K-PATENTS® Compact Process Refractometer PR-43-GC is available with optional higher alloys and special wetted parts materials.

The real-time and accurate acid concentration measurement helps to obtain products that meet the specifications and to avoid deterioration of the metal parts, thus avoiding re-treatment. Refractive index measurements also can serve as an indication of increased dissolved material in the baths or contaminants in the passivation solution, providing important information for replenishment of the baths.

The Vaisala refractometer has mA and Ethernet output signals that can be connected to the control system to maintain records of the concentration of the passivation solution, required during inspection to demonstrate that the specified passivation conditions were maintained for each lot of stainless-steel parts. The sensor requires no recalibration and verification is easily done in the field with standard refractive index liquids.

The Vaisala refractometer provides the required reliable liquid concentration measurement to ensure the chemicals meet the recommended concentration level and that contaminants are within tolerance for optimal passivation and consistency in all the process steps.

Instrumentation	Description
	Teflon Body Refractometer PR-23-M. A compact refractometer for chemically aggressive solutions and ultra-pure fine chemical processes. Connected to the process by a G1/2" female or a 1/2" NPT process connection. It has a built-in flow cell designed to keep all metal and other easily corroding parts from coming into contact with the process liquid.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	30-50 % Nitric acid. Typical accuracy +/- 0.5 % by weight.

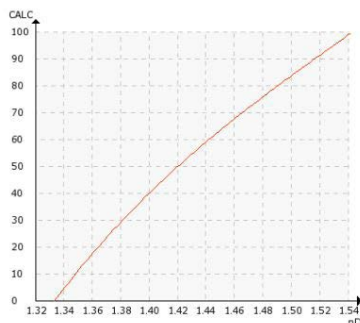


## LONGWALL HYDRAULIC FLUIDS, FIRE RESISTANT EMULSIONS

### Typical end products

Coal.

Chemical curve: R.I.per Brix at Ref. Temp. of 20°C



### Introduction

Longwall mining is an underground coal extraction method, which removes large volumes of coal with minimum impact to the surface environment.

It involves cutting parallel underground roadways to form *blocks*. A coal face is formed between these roadways and the coal is extracted by a mechanical shearer. The longwall advances as the shearer cuts back and forth from one roadway to another. The roof directly above and behind the shearer is supported by hydraulic jacks, creating a safe work area for the machinery and operators. These jacks require an operating pressure of around 4500 psi. The roof jacks contract, extend and edge forward individually, thus maintaining the roof directly in front of the coal face. At the same time, it allows the roof behind the advancing

shearer, where the coal has been extracted, to collapse. Thus, there is no requirement for permanent support. This collapsed area is called the *GOB* and cannot be re-entered.

The underground atmosphere in the mine contains a dangerously explosive mixture of methane and coal dust. Additionally, the water table is sometimes only meters from the coal seam. It is mainly for these reasons that oil based hydraulic fluids are not used. A product referred to as a fire-resistant hydraulic emulsion, also known as *longwall fluid*, is used instead. It is water based, containing special additives.

### Application

Longwall hydraulic fluid loss from the hydraulic circuit is extremely high, requiring continuous replenishment. In older longwall systems as much as 113562 liters (30000 gallons) of neat emulsion can be used per month.

Efficient and reliable dosing of the longwall fluid is required in order to maximize the operating life of the hydraulic components and pump systems. High levels of concentration can cause gasket failure resulting in leakage, while low level concentrations will reduce its corrosion prevention effectiveness, which may result in damage and system failure.

Vaisala K-PATENTS® Process Refractometer is used for precise dosing and mixing of the fluid. The concentration of the longwall fluids ranges between

1 and 5 % and has an approximate Brix gain of 0.6 (where 5 % is around 3.5 Brix).

The concentration measurement is unaffected by oil tank head pressure, water flow, pressure variations, raw oil concentrations or instrument location.

## Instrumentation and installation

There are two typical installation types for the Process Refractometer:

### 1. Underground

The roof supports require hydraulic pumps driven by large electric motors to supply 4500 psi in order to function properly. These pumps are mounted on a pump cart and supplied from a mixed emulsion tank. The emulsion is then recycled from the coal face to the tank. The tank is replenished with fresh emulsion in batch mode when the tank levels drop.

The best installation point is in a dedicated recirculation loop drawing from and returning to the mixed emulsion tank.


### 2. Surface

Some mines prepare emulsion above ground and then transfer the mixed emulsion underground through a pipe.

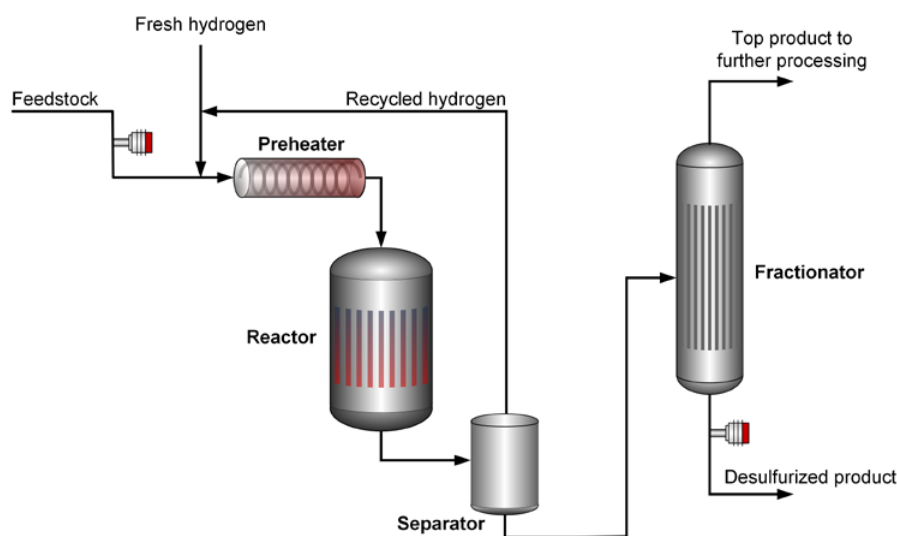
In this installation, as there is no return line, only the fresh emulsion is measured. Provided there is no interference, the fresh emulsion and underground return emulsion should remain at the same concentration.

### Application considerations

- Prism coating occurs even at low concentrations if the flow velocity is less than 1.8 m/s. Sometimes, if the water hardness varies, soap will form and cause prism coating irrespective of velocity. For these reasons automatic prism wash with steam may be needed.
- Explosion protection is typically required. Appropriate equipment with hazardous and intrinsic safety approvals are available when required.
- In some cases, it may be desirable to mount the whole refractometer system in an EXd-enclosure. The PR-43 is a stand-alone device and can be easily operated remotely via Ethernet without having to access the transmitter.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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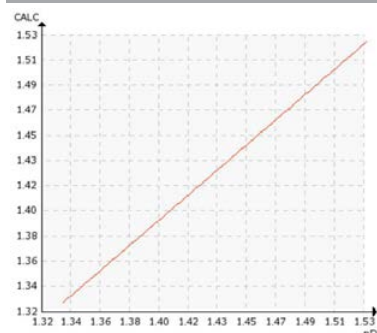


## HYDROCARBON

### Typical end products

High-end and intermediate feedstocks: diesel fuel, jet fuel, naphtha, gas oil, coker feed.

Chemical curve: R.I. at Ref. Temp. of 20 °C



## Introduction

Hydrotreating is a refining process used to purify and saturate olefins and aromatics in all final and intermediate refinery feedstocks. These include diesel, jet fuel, naphtha, gas oil, and coker feed. The impurities removed include sulfur, nitrogen and metals. The purpose of saturating the olefins and aromatics is to reduce their presence in the hydrocarbons, and to improve the feedstock to the other refinery processes such as the catalytic cracker. The process also has the benefit of mild cracking of heavier components.

## Application

The hydrocarbon is mixed either with hydrogen or a hydrogen-enriched gas and is then heated before entering the reactor. There, the hydrogen reacts with the sulfur to form hydrogen sulphide, and with the nitrogen to form ammonia. Any free hydrogen is then


available to react, saturating the aromatic and olefin compounds. This reduces the total percentage of aromatics and olefins in the feed.

Vaisala K-PATENTS® Process Refractometer PR-43-GP provides real-time in-line indication of the aromatic content of the hydrocarbon stream. There is a correlation between the refractive index value and the aromatic content of hydrocarbons. The typical measurement range is between 1.400 and 1.4630 at 25 °C (77 °F).

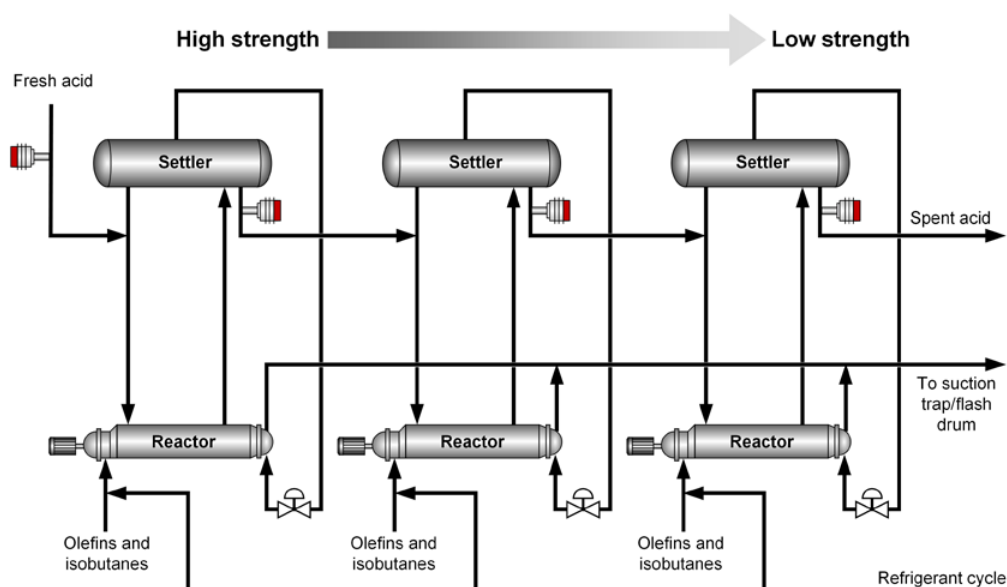
## Instrumentation and installation

The Process Refractometer PR-43-GP is mounted in the hydrotreater feed stream or at the final product line. The refractometer provides Ethernet and 4-20 mA output signals that can be used for real-time control. For instance, the output signal from the refractometer is used to adjust either reactor temperature, hydrogen make-up ratio or the feedstock blend to achieve the required aromatic ratio.

Appropriate equipment with hazardous and intrinsic safety approvals are available when required. For sensor wetted parts stainless steel 316L SS can be used, unless conditions require the use of special materials. Automatic steam prism wash with steam is recommended.

Instrumentation	Description
	Process Refractometer PR-43-GP is a heavy-duty instrument with non-weld body construction for diverse oil and gas industry applications. The refractometer is installed in the main processing line by welding stud and flange connection for 2 inch, 2.5 inch and larger pipe sizes and vessels, or via flange and FTC Flow through cell connection for 0.5 inch, 1 inch, 1.5 inch and 2 inch pipe sizes.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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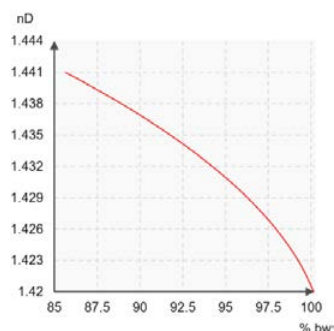


## SULFURIC ACID, H<sub>2</sub>SO<sub>4</sub>

### Typical end products

Alkylate (premium higher-octane gasoline blending stock for motor fuel and aviation gasoline).

Chemical curve: Sulfuric acid 88-100 R.I. per Conc wt.-% at Ref. Temp. of 20 °C



### Introduction

Motor fuel alkylation using sulfuric acid (H<sub>2</sub>SO<sub>4</sub>) or liquid hydrofluoric acid (HF) is one of the oldest catalytic processes used in petroleum refining. The purpose of the alkylation is to improve motor and aviation gasoline properties (higher octane) with up to 90 % lower emissions compared to conventional fuel usage.

The problem with HF is that the catalyst forms a hazardous air pollutant when released as a superheated liquid, while H<sub>2</sub>SO<sub>4</sub> does not. Therefore nearly 90 % of all alky units built since 1990 have adopted the H<sub>2</sub>SO<sub>4</sub> technology.

The leading alkylation unit licensor, with a 90 % share of the market, is DuPont (Stratco®). Another licensor is EMRE (Exxon Mobile Research Engineering, formerly K.W. Kellogg).

### Application

In the process, isobutane is alkylated with low molecular weight olefins (propylene, butylene and pentylene) in the presence of a strong acid catalyst to form alkylate (the premium higher-octane gasoline blending stock). The catalyst (sulfuric acid) allows the two-phase reaction to be carried out at moderate temperatures. The phases separate spontaneously, so the acid phase is vigorously mixed with the hydrocarbon phase to form higher molecular weight isoparaffinic compounds.

After the reactor, the mixture enters a separation vessel where the acid and hydrocarbon separate. The acid is then recycled back to the reactor.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is installed after the settlers to continuously monitor in real-time the concentration of acid in the process.

The concentration of sulfuric acid is critical to achieve the complete consumption of isobutane. A highly variable concentration of isobutane in the feedstock upsets the sulfuric acid content in the process.



It is important to determine the proper quantity of acid that will be fed into the process. This is achieved by combining routine sample titration analysis with continuous acid monitoring by the Process Refractometer. Real-time measurements reduce the need for sampling and laboratory analyses that cause delay in the implementation of any necessary adjustments to the acid flow.

Continuous monitoring removes the uncertainty involved between titration measurements. The refractometer will indicate any gradual fluctuations in the acid flow, allowing precise control over efficient acid consumption and resulting in cost savings. It is also useful in preventing acid runaway, an unwanted situation commonly described as wild acid.

Acid runaway may happen when the acid strength drops below 85-87 %  $H_2SO_4$ . As a result, the reactions between olefins and isobutane turn into reactions of olefins only, producing polymers known as *acid sludge*, *ASO* or *red oil*.

The refractometer is not affected by acid soluble oil (ASO). The refractometer indicates actual acid strength regardless of the amount of hydrocarbons present, which is essential when transferring acid emulsion. It is also an extremely useful tool in real-time process acid strength measurement during agitated conditions.

The initial acid concentration is typically 85-100 % and the temperature is 15 °C (59 °F). The benefits of the refractometer's continuous monitoring system include substantial cost savings due to reduced acid consumption, and smooth alkylate production without acid runaways.

## The Process Refractometer System for Alkylation Acid Measurement Consists of:

1. The Process Refractometer PR-43 for hazardous locations in Zone 2.

or

The PR-43 Intrinsically Safe model for installations in hazardous locations up to Zone 0.

2. Optional parts:

2.1. Different flow cell options for easy sensor installation

2.2. *EXd* enclosure for easy isolator and transmitter mounting

2.3. Parts for a start up


2.4. Spare parts supplied for two years of operation

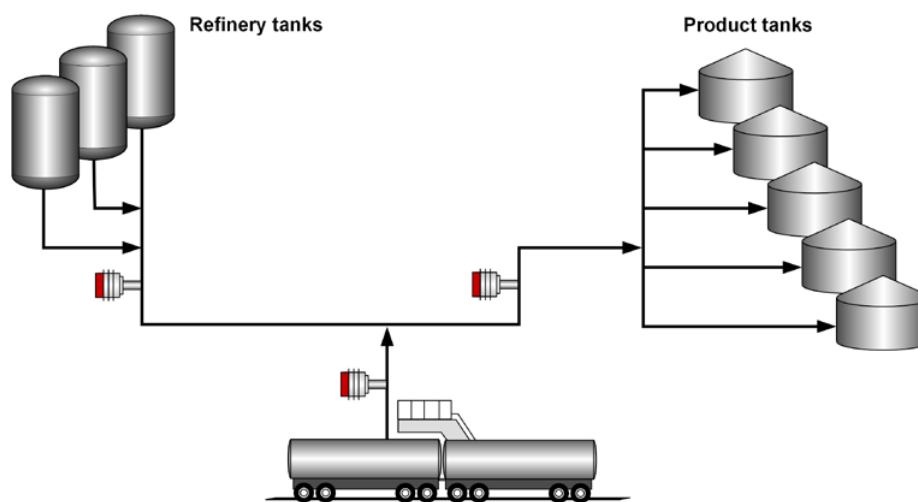
2.5. Start-up and commissioning service

3. User specified tests and documentation.

Alloy C-276/ASTM C276 should be considered as wetted parts material when the acid piping flow velocity is at a maximum of 6 m/s (20 ft/s). Alloy 20 can be considered when acid piping flow velocity is at a maximum of 1.8 m/s (6 ft/s). However, it is the responsibility of the end-user to specify the appropriate material, ensuring that it is satisfactory for the intended operating requirements.

Non-sparking incentive (*Ex nA*) and intrinsic safety (*Ex ia*) approvals are available for hazardous area installations.

Instrumentation	Description
	Process Refractometer PR-43-GP is a heavy-duty instrument with non-weld body construction for diverse oil and gas industry applications. The refractometer is installed in the main processing line by welding stud and flange connection for 2 inch, 2.5 inch and larger pipe sizes and vessels, or via flange and FTC Flow through cell connection for 0.5 inch, 1 inch, 1.5 inch and 2 inch pipe sizes.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

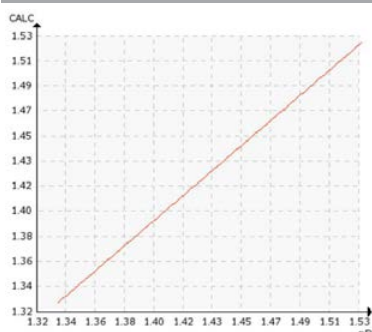


## NATURAL GAS LIQUIDS (NGL), LIQUID HYDROCARBONS

### Typical end products

Natural gas liquids such as n-butane and iso-butane, and other liquid hydrocarbons.

### Chemical curve: R.I. at Ref. Temp. of 20°C



## Introduction

Chemical plants and refineries receive or deliver many different liquid hydrocarbons, including Natural Gas Liquids (NGLs) which are transported via pipelines, railcars, tanker trucks or ships. Many of these products are very similar in properties and appearance, therefore, proper identification of the hydrocarbons and the interface at transport locations that handle multiple products is important. Combining an interface detection device with automatic controls can minimize transmix of products, reduce waste, reduce the filling/unloading times, decrease safety risks, reduce sampling and minimize operator errors.

Refractive index is a reliable method for identifying liquid hydrocarbon products since each hydrocarbon has a different and distinct refractive index value. This refractive index is a property inherent to the hydrocarbon and can be used as a product *fingerprint*

for identification. This fingerprint can be applied to a scale to achieve the proper controls.


## Application

Two typical products that require interface detection are n-butane and iso-butane. These isomers of butane have the same molecular formula but different chemical structures. Since they are isomers, the chemical properties of these products are very similar, in particular, the density. However, the difference in refractive index is wide enough to accurately and reliably distinguish between these two products. Often, the refractive index value is converted to the Brix scale so that whole number values can be used.

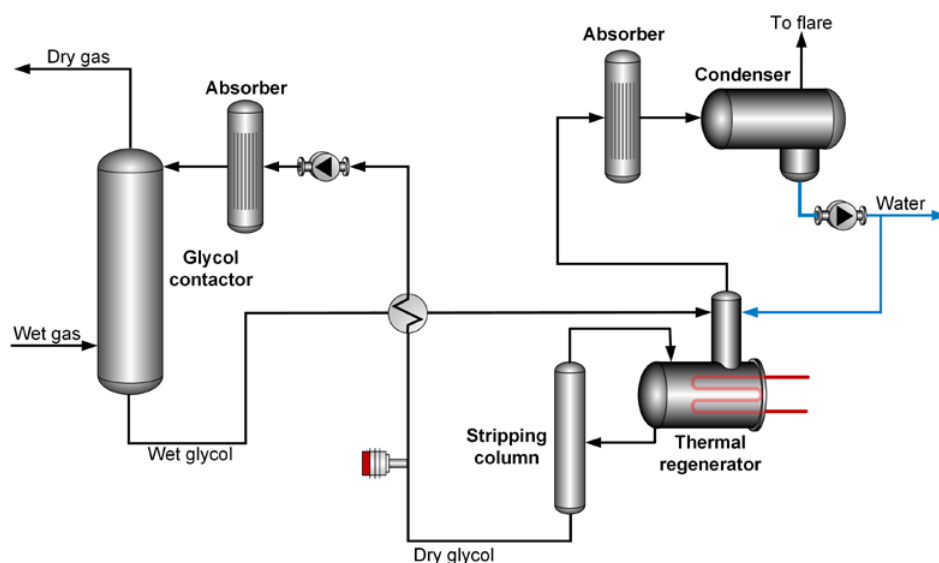
## Instrumentation and installation

Vaisala K-PATENTS® Process refractometer PR-43-GP is typically installed in the main supply line and at the inlet of the product storage. This provides sufficient time for interface detection and assurance of the hydrocarbon product being delivered to the correct storage. The refractometer provides measurement accuracy of  $nD \pm 0.0002$ .

The refractometer should be installed in a location that is constantly wetted. Due to the digital sensing technology small amounts of entrained air or gases will not affect the measurement. Normal wetted parts are stainless steel 316L and higher allows are available on request. Appropriate hazardous and intrinsic safety approvals are available if required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a heavy-duty instrument with non-weld body construction for diverse oil and gas industry applications. The refractometer is installed in the main processing line by welding stud and flange connection for 2 inch, 2.5 inch and larger pipe sizes and vessels, or via flange and FTC Flow through cell connection for 0.5 inch, 1 inch, 1.5 inch and 2 inch pipe sizes.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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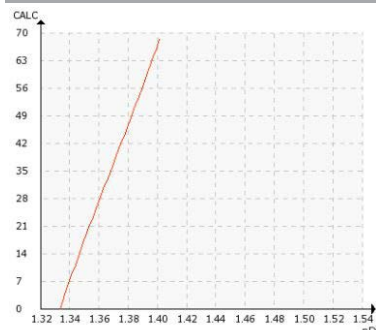


## DIETHYLENE GLYCOL (DEG), TRIETHYLENE GLYCOL (TEG)

### Typical end products

Pipeline quality dry natural gas.

Chemical curve: Ethylene glycol R.I. per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Natural gas processing consists of separating all the various hydrocarbons and fluids from the pure natural gas to produce what is known as *pipeline quality* dry natural gas. It means that before the natural gas can be transported, it must be purified and most of the associated water must be removed.

Most of the liquid (free water) is removed by simple separation methods at, or near, the wellhead. However, the removal of the water vapor, which exists in natural gas solution, requires a more complex treatment. This treatment consists of *dehydrating* the natural gas either by absorption or adsorption.

### Application

An example of absorption dehydration is known as *Glycol Dehydration*. In this process, a liquid desiccant dehydrator serves to absorb water vapor from the gas stream. Glycol, the principal agent in the process, has a chemical affinity to water. During the process, the glycol concentration decreases and water content increases. Due to glycol loss continuous replenishment is required.

The regeneration should be minimized since it involves combustion emission. The refractometer indicates when glycol regeneration is required.

Essentially, a glycol solution is used in glycol dehydration, usually either Diethylene Glycol (DEG) or Triethylene Glycol (TEG), which is brought into contact with the wet gas stream in what is called *the contactor*. The glycol solution will absorb water from the wet gas. Once absorbed, the glycol particles become heavier and sink to the bottom of the contactor, from where they are removed. The natural gas, having been stripped of most of its water content, is then transported out of the dehydrator.


The glycol solution, bearing all the water absorbed from the natural gas, is put through a specialized boiler designed to vaporize only the water out of the solution. While water has a boiling point of 100 °C (212 °F), glycol does not boil until 200 °C (392°F). This differential boiling point makes it relatively easy to remove water from the glycol solution, allowing it to be reused in the dehydration process.

## Instrumentation and installation

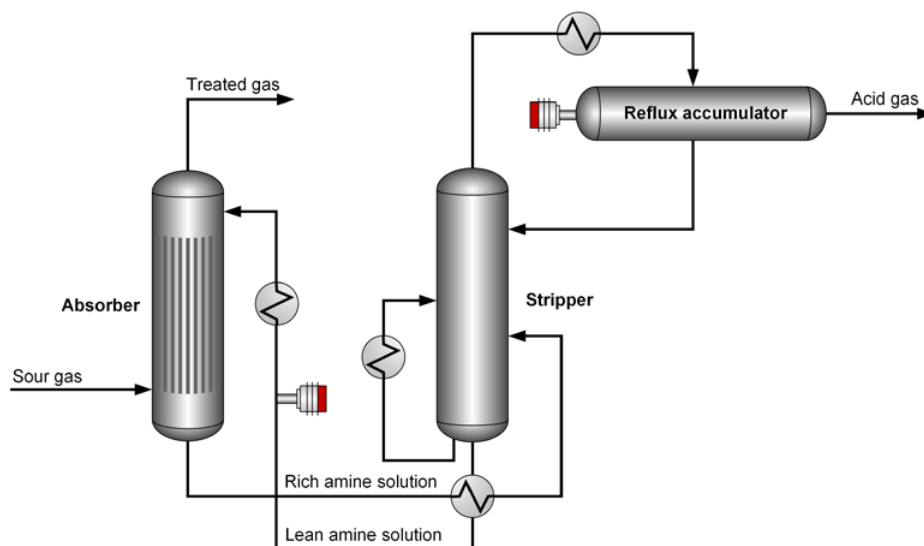
Vaisala K-PATENTS® Process Refractometer PR-43-GP is installed after the stripping column to monitor the concentration of glycol after the regeneration process. The refractometer provides Ethernet and 4-20 mA output signals that can be used as feedback control to adjust the column's parameters, thus ensuring the target glycol specification.

During the dehydration of the gas, the concentration of glycol decreases because of the absorption of water. Another refractometer can monitor the concentration of glycol to ensure an effective process and indicate when regeneration is required.

Standard sensor material can be used in this application. Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a heavy-duty instrument with non-weld body construction for diverse oil and gas industry applications. The refractometer is installed in the main processing line by welding stud and flange connection for 2 inch, 2.5 inch and larger pipe sizes and vessels, or via flange and FTC Flow through cell connection for 0.5 inch, 1 inch, 1.5 inch and 2 inch pipe sizes.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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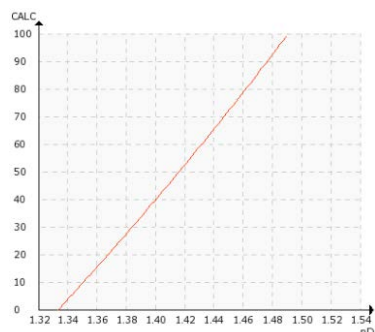


## MONOETHANOLAMINE (MEA), DIETHANOLAMINE (DEA), METHYLDIETHANOLAMINE (MDEA)

### Typical end products

Pipeline quality dry natural gas, liquefied petroleum gas (LPG).

Chemical curve: MDEA R.I. per Conc% b.w. at Ref. Temp. of 20 °C



### Introduction

Natural gas contains significant amount of hydrogen sulfide (H<sub>2</sub>S) and carbon dioxide (CO<sub>2</sub>). Natural gas is also referred to as *sour gas* because of its strong odor, caused by the sulfur content. These sulfur compounds render it extremely harmful, even lethal, to breathe. Natural gas can also be extremely corrosive. Carbon dioxide must be removed before the gas can be transformed into liquid form (liquefaction LNG) for transportation. Liquefaction happens at a extremely low temperature (-161 °C or -258 °F) at which carbon dioxide can freeze and consequently block pipe lines.

Amine gas treating, also known as *gas sweetening* and acid gas removal, refers to a group of processes that use aqueous solutions of various amines to remove H<sub>2</sub>S and CO<sub>2</sub> from gases. It is a common

unit process used in refineries, petrochemical plants, natural gas processing plants and other industries.

The acid gas absorption in amine solution is conducted using a two-column operation: the first column is used to absorb the acid gas into the absorbent amine, the second column is used to regenerate the amine.

The process relies on counter current flow to achieve optimum mixing. A lean solution (low acid gas) enters the top of the absorber and flows to the bottom; acid gas enters the bottom of the absorber tower and bubbles to the top.

The rich amine (high acid gas) enters the stripper, where the acid gases are released, and the clean amine is returned to the absorber. The acid gases collect and exit at the top of the stripper.

### Application

In the regeneration process the amine can degrade or be depleted. In order to achieve the proper acid gas removal, the optimum amine concentration must be maintained. Most acid gas recovery systems use either monoethanolamine (MEA), diethanol-amine (DEA) or methyldiethanolamine (MDEA).

Degraded and corrosive by-products are removed by carbon filters. Carbon filters also remove amine from the solution, therefore continual amine top-up is required.

Filtration is done through a slipstream, so the amine concentration is not totally depleted on each pass. Therefore, the top-up must be based on the quantity filtered.

The amine concentration is traditionally measured by lab titration. This technique assumes that all alkalinity is due to amine and can give false readings because of the many inhibiting factors. Furthermore, unlike periodical sampling, continuous in-line measurement provides an instant feedback to indicate any fluctuations in the process. This instant feedback can be used for real-time process control.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to measure the lean amine concentration of the acid gas from the MEA/DEA regeneration stripper column. By maintaining an optimum amine concentration, the appropriate acid gas removal can be achieved.


Reliable amine concentrations of 18-20 % are an advantage for optimal H<sub>2</sub>S removal. The final amine concentration can be controlled with a top-up, so that the appropriate amount of MEA at 10-15 wt-% is fed into the process to remove CO<sub>2</sub>, as:

- too low a MEA will lower the absorption efficiency
- too high a MEA will increase the corrosion of the process equipment (corrosion protection)

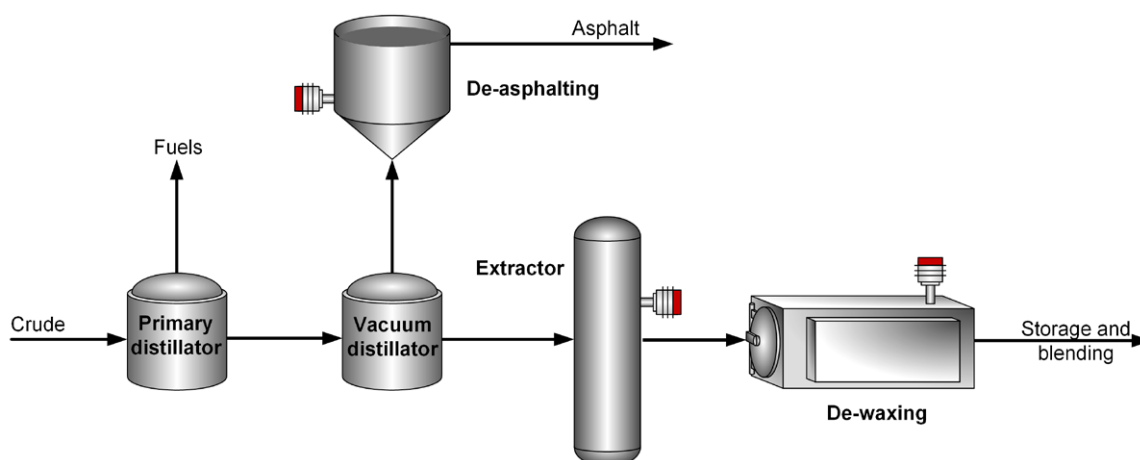
The refractometer is unaffected by alkalinity or any other possible inhibitors present in the process.

Standard sensor material can be used in this application. Silicon oil is often added to the MEA solution to prevent foaming and therefore periodical prism cleaning (every 3-4 weeks) may be needed. Installation in a by-pass is recommended.

Instruments with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a heavy-duty instrument with non-weld body construction for diverse oil and gas industry applications. The refractometer is installed in the main processing line by welding stud and flange connection for 2 inch, 2.5 inch and larger pipe sizes and vessels, or via flange and FTC Flow through cell connection for 0.5 inch, 1 inch, 1.5 inch and 2 inch pipe sizes.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

Ref. B212033EN-A © Vaisala 2020



## LUBE OIL

### Typical end products

Lube oil.

### Introduction

Continuous in-line monitoring of the refractive index in lube oil plants allows highly efficient refining process control. Traditionally, the operators perform refractive index analysis to the waxy raffinates with a laboratory refractometer. This analysis is described in the *ASTM D 1747-89 Standard Test Method for Refractive Index of Viscous Materials*.

When manufacturing different types of Viscosity Index (V.I.) oils, it has been established through laboratory experimentation that the Refractive Index (R.I.) of waxy raffinates should remain within defined limits. The operators need to adjust the process temperatures of the different catalytic beds to maintain the refractive index of the waxy raffinates within these limits.

### Application: Lubricating Base Oil Processes

#### Vacuum Distillation Unit

The first step in the refining process for lubricating oils is separation in the distillation units of the individual fractions according to viscosity and boiling range specifications. The heavier raw stock for lube oil settles at the bottom of the vacuum fractionating tower with the asphaltenes, resins and other undesirable materials.

The raw lube oil fractions from most crude oils contain components, which have undesirable characteristics for finished lubricating oil. These must be removed or reconstituted by processes such as extraction,

crystallization, hydrocracking and hydrogenation. Vacuum distillation separates raw lube oil into two or three grades with increasing viscosity. The heavier grades are derived by removing asphalt from the residue in a de-asphalting unit. The lighter feedstocks are sent directly to solvent extraction. This first stage determines the final base oil viscosity grades.

Further processing is usually done in the following order; de-asphalting, solvent extraction, de-waxing and finishing.

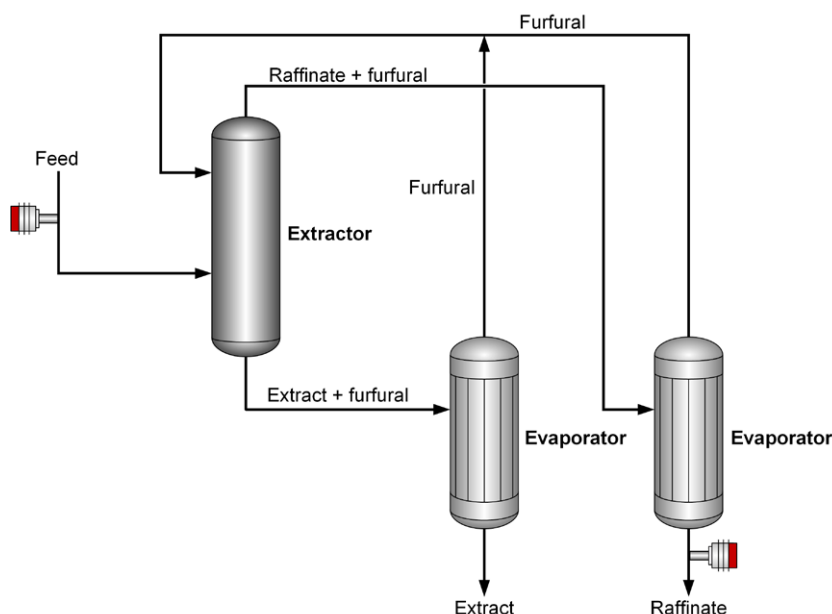
#### De-asphalting

Propane is typically used as the solvent in de-asphalting, but it may also be combined with ethane or butane in order to obtain specific solvent properties. Propane has unusual solvent properties at temperatures from 40 °C (104 °F) to 60 °C (140 °F). Paraffines are very soluble in propane but the solubility decreases as the temperature increases, until the critical temperature of propane (96.8 °C or 206.2 °F), when all hydrocarbons become insoluble. In the range of 40 °C (104 °F) to 96.8 °C (206.2 °F), the higher molecular weight asphaltanes and resins are largely insoluble in propane. Separation by distillation is generally through molecular component weight and solvent extraction through molecule structure type.

#### Solvent Extraction

There are three solvents used for the extraction of aromatics from lube oil feedstocks. The solvent recovery portions of the systems are different for each one. The solvents are furfural, phenol and N methyl-2-pyrrolidone (NMP). The purpose of the solvent extraction is to improve the Viscosity Index (V.I.), oxidation resistance, color of the lube oil base stock,





and to reduce carbon and lubricants' sludge-forming tendencies by separating the aromatic portion from the naphthenic and paraffinic portion of the feedstock.

## Furfural Extraction

The process flow through the furfural extraction unit is similar to that of the propane de-asphalting unit, except for the solvent recovery section, which is more complex. The oil feedstock is introduced to a continuous counter-current extractor at a temperature, which is the function of the feed's viscosity; the greater the viscosity, the higher the temperature used.

## Phenol Extraction

The process flow for the phenol extraction unit is somewhat similar to that of the furfural extraction unit, but differs markedly in the solvent recovery section because phenol is easier to recover than furfural.

## NMP Extraction

NMP extraction uses N-methyl-2-pyrrolidone as the solvent to remove the condensed aromatics and polar components from the lubricating oil distillate brightstocks. This process was developed as a replacement for phenol extraction because of health, safety and environmental problems associated with phenol use.

## De-waxing

All lube oil stocks, except those from a relatively few highly naphthenic crude oils, must be de-waxed or they will not flow properly at ambient temperatures.

De-waxing is one of the most important and most difficult processes in lubricating oil manufacture.

## Hydrofinishing


Hydrotreating of de-waxed lube oil stocks is needed to remove chemically active compounds that affect the color and color stability of the lube oils.

## Instrumentation and installation

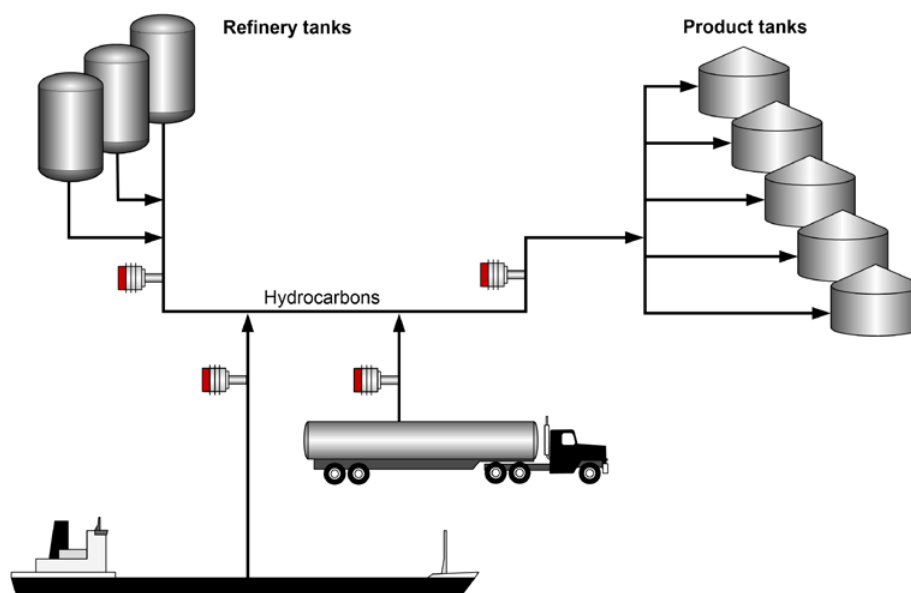
A typical measurement point for Vaisala K-PATENTS® Process Refractometer PR-43-GP is in-line in the heat exchanger outlet, where off-line lab samples are usually taken from. The refractometer is typically installed in an insulated by-pass line with temperature control. A flow velocity in the sampling loop of 1.5 m/s (5 ft/s) or higher is recommended.

The Process Refractometer eliminates sampling, improves product consistency and helps to optimize the process. A uniform product can be achieved through continuous monitoring.

Unlike periodic sampling, continuous monitoring can provide instant feedback on process fluctuations. This instant feedback is used to control the process in real-time.

Instrumentation	Description
	Process Refractometer PR-43-GP is a heavy-duty instrument with non-weld body construction for diverse oil and gas industry applications. The refractometer is installed in the main processing line by welding stud and flange connection for 2 inch, 2.5 inch and larger pipe sizes and vessels, or via flange and FTC Flow through cell connection for 0.5 inch, 1 inch, 1.5 inch and 2 inch pipe sizes.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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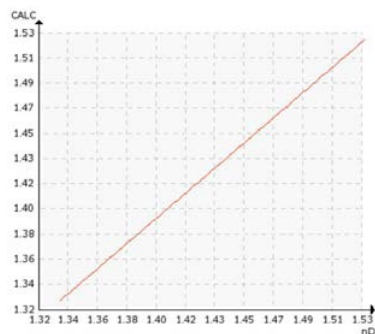


## CRUDE OIL, FUEL OIL, DIESEL, MULTI-PRODUCT INTERFACES

### Typical end products

Crude oil, fuel oil, hydrocarbons.

Chemical curve : R.I. at Ref. Temp. of 20°C



### Introduction

Fuel oils are transported from oil refineries to end users via a complex distribution system. Identifying the fuel type is a necessity, as different types of fuels are handled and passed through the distribution lines. The fuels are directed into the appropriate product tanks at the discharge end of the system. Accurate and reliable interface detection is required to ensure that each product ends up in the correct tank, as product contamination can be very costly.

### Application

The refinery and pipeline operating personnel identify the interface visually, through color variation. These

visual indicators are sometimes enhanced by the use of markers such as dyes (pigs). Sampling is also done to verify the identification.

The time it takes for the interface to pass is extremely short, typically less than five minutes. The exact time will depend on the flow rate. This requires a rapid response in order to make the appropriate selections.

It is also important for the user to be able to differentiate between the various crude oil types.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP provides an in-line and real-time indicator for interface detection. The difference between the refractive indexes for different fuels is wide, so the interface can be detected reliably (see Table 1). The typical refractive index range is between 1.4000 and 1.4630 at a temperature of 25 °C (77 °F). Crude oils of different origins have different refractive indexes. The PR-43-GP can also provide information to the operator on the type of crude oil.


**Table 1.** Typical values of transportation fuels.

Fuel	API Density	Brix	n <sub>D</sub> @ 25°C
Gasoline	55-65	45-51	1.4098–1.4222
Jet Fuel	50-40	64	1.4511
Diesel	30-40	71	1.4679

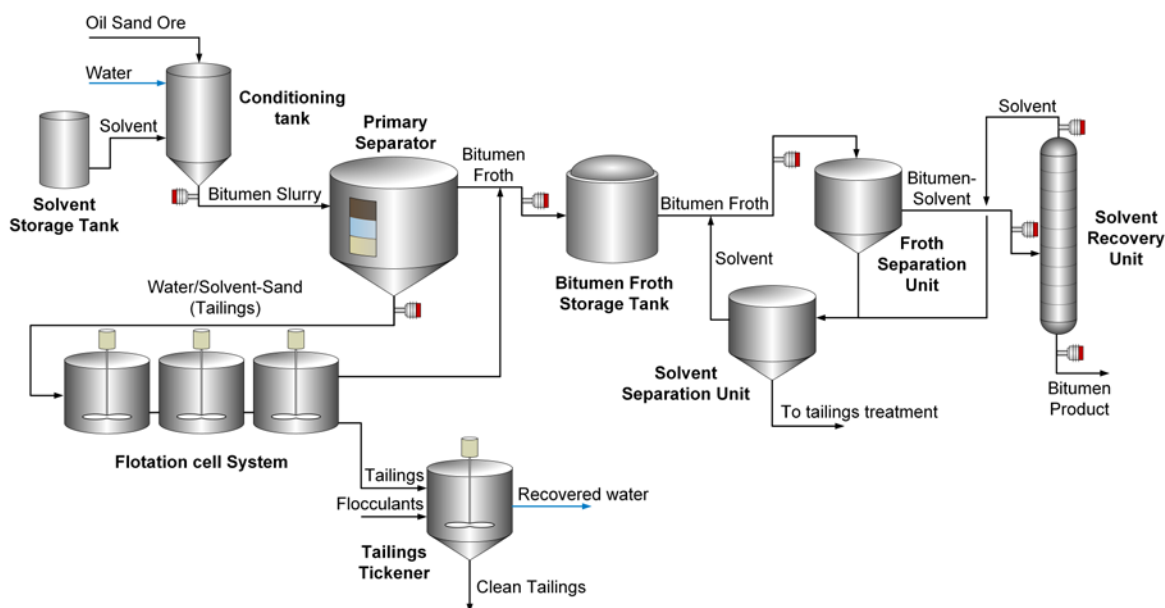
If the pipeline operates at a very high pressure, it might require the refractometer to be mounted on to a slip stream or on the low-pressure side of the pipeline.

wetted parts if the service conditions do not demand the use of special materials.

Appropriate equipment with hazardous and intrinsic safety approvals are available when required. Stainless steel 316L SS can be specified for sensor

Instrumentation	Description
	Process Refractometer PR-43-GP is a heavy-duty instrument with non-weld body construction for diverse oil and gas industry applications. The refractometer is installed in the main processing line by welding stud and flange connection for 2 inch, 2.5 inch and larger pipe sizes and vessels, or via flange and FTC Flow through cell connection for 0.5 inch, 1 inch, 1.5 inch and 2 inch pipe sizes.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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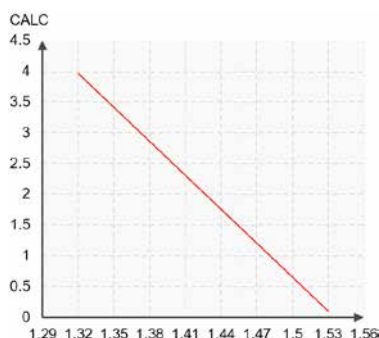


## DILUTED BITUMEN SLURRY AND FROTH, SOLVENT

### Typical end products

Synthetic crude oil, upgraded bitumen.

Chemical curve: R.I. per Solvent/Bitumen Ratio at Ref. Temp. of 20°C



### Introduction

Oil sands, tar sands or heavy oils are a mixture of water, sand, clay and bitumen. The bitumen can be extracted from the mixture and be upgraded into a higher-value synthetic crude oil for producing the same petroleum products as from conventional oil. Bitumen content in the sands is usually between 1 and 20 %.

Oil sands can be recovered by surface mining or from subsurface reservoirs by steam injection and they can be classified in two types. If the bitumen is separated from the sand by a film of water, the oil sands are called *water-wet*. The other type is *oil-wet*, where the

bitumen is bonded directly to the sand, or with little water between them.

Because bitumen content is low in the oil sand, it is important to keep tight control during the extraction process to maximize bitumen recovery and quality.

### Application

The bitumen extraction process starts by milling the mined ore and mixing it with a solvent in a large vessel to create a slurry. This slurry is agitated to incorporate air bubbles.

For the extraction of bitumen from water-wet sands, the *Clark Extraction Process* is commonly used, where the solvent is hot water. For extraction from oil-wet sands, the hot water is replaced by a water/solvent solution (usually alkali), or completely by an organic solvent, e.g. citrus based.

In hot water extraction, the excess of water separates the bitumen from the water and sand. Because the bitumen is hydrophobic, it attaches to the air bubbles created by agitation and floats on the surface. In the case of oil-wet sand, the solvent dissolves in the bitumen and immediately separates the bitumen from the insoluble ore materials.

Next, the slurry is pumped into a large vessel referred to as the *Primary Separation Cell* to allow physical separation.

The aerated bitumen instantly floats on the top when it enters the vessel and it can be easily skimmed off. This bitumen froth usually contains 60 % bitumen, 30 % water and 10 % fine solids. The sand (tailings) sink to the bottom of the vessel and are removed as an underflow to the tailings treatment plant. A middle phase (middlings) is also formed consisting in fines that are too light to sink, and too heavy to float. The middlings have a higher content of bitumen than the underflow normally between 1-4 %.

The separated fractions move on for further treatment. The bitumen froth is mixed with a light hydrocarbon solvent (naphtha-based or paraffinic) to reduce its viscosity and to allow further separation of the fine solids. After this, the bitumen may be further upgraded by distillation to meet the required specifications. The water or solvent in tailings is recovered for reuse and the solids are transported back to fill the mine.

## Instrumentation and installation


Vaisala K-PATENTS® Process Refractometer PR-43-GP is an ideal heavy-duty instrument for oil sands extraction and bitumen processing. The refractometer provides an accurate and continuous measurement of bitumen froth and solvent concentrations at any stage of the process. The measurement of the refractometer is selective to the liquid phase and is not influenced by bubbles, suspended solids or the color of the mixture.

The refractometer is installed directly in the pipe or vessels, and provides Ethernet or 4-20 mA output signals for real-time process control. The signal from the refractometer can be used, for example, to monitor the effectiveness of the gravity separation by installing refractometers in the bitumen slurry, bitumen froth and underflow lines of the Primary Separation Cell.

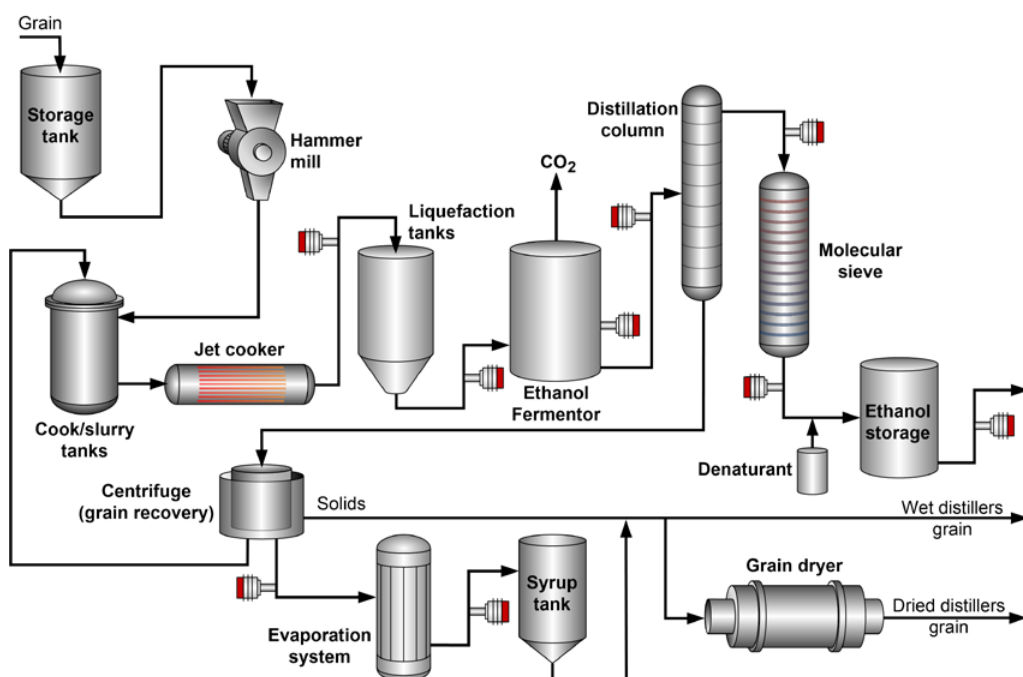
The refractometer can be calibrated to read the Solvent:Bitumen (S:B) ratio in the Froth Treatment Process. Real-time and precise measurement of the S:B ratio prevents exceeding the capacity of the vessel, minimizes the amount of solvent in the tailings stream and ensures enough asphaltene is precipitated to meet the final product specifications.

Finally, the refractometer monitors the distillation operation to guarantee that the final product meets the strict requirements by the refineries.

The precise measurement by the refractometer improves productivity and quality of the bitumen product with less costs and reduced environmental impacts. Automatic prism wash may be required in this application.

Instrumentation	Description
	Process Refractometer PR-43-GP is a heavy-duty instrument with non-weld body construction for diverse oil and gas industry applications. The refractometer is installed in the main processing line by welding stud and flange connection for 2 inch, 2.5 inch and larger pipe sizes and vessels, or via flange and FTC Flow through cell connection for 0.5 inch, 1 inch, 1.5 inch and 2 inch pipe sizes.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	Prism wash system components are a refractometer with integral wash nozzle mounted at the refractometer probe or in a flow cell, wash supply line components and a Multi user interface MI with relay module for prism wash diagnostics and control. Alternative wash media can be used for wash, e.g. steam, high-pressure water and warm water (hot condensate).
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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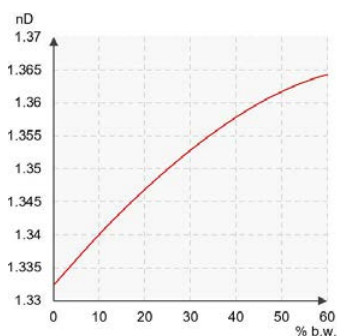
## ETHANOL (C<sub>2</sub>H<sub>5</sub>OH)

### Typical end products

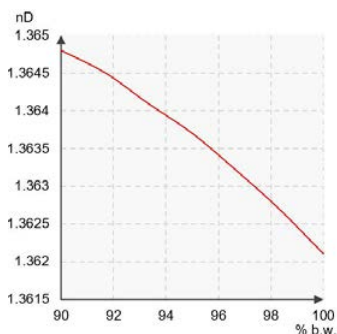
Bioethanol fuel (ethyl alcohol) additive for gasoline.

Chemical curve: Ethanol R.I. per Conc% b.w. at Ref. Temp. of 20 °C

### Range 0-60 %



### Range 90-100 %



## Introduction

The main fuel used as gasoline substitute for road transport vehicles is bioethanol. Ethanol or ethyl alcohol (C<sub>2</sub>H<sub>5</sub>OH) fuel is mainly produced by the sugar fermentation process.

Fuel and energy crops are the main sources of sugar required to produce ethanol. These crops are specifically grown for energy use and include corn, maize, wheat, reed canary grass and cord grass. Waste straw, willow and popular tree sawdust are also used. Development is also proceeding for the utilization of municipal solid wastes in ethanol fuel production.

## Application

The Dry Mill process of producing Motor Fuel Grade Ethanol (MFGE) using corn as the feedstock consists of six steps.

### 1. Grinding

Incoming grain is screened and ground to a coarse flour in either a hammer mill or a roller mill. Hammer mills are most commonly used. Maintaining a consistent grain size is important in enabling the proper breakdown of starch into simple sugar for efficient separation downstream.

## 2. Cooking

The ground flour is mixed with water and enzymes. The temperature and the pressure are raised to 120 °C (248 °F) and 1-3 bar (10-40 psig) for partial sterilization. The mixture temperature is then kept elevated at 85-90 °C (185-194 °F) for 2-4 hours so the enzymes break the starch down into simple sugar. The resulting product is called *mash*.

## 3. Fermentation

The mash is added to the fermentation tanks, along with large amounts of yeast. The yeast converts the sugar into ethanol, CO<sub>2</sub> and heat. This step can take between 40-70 hours. The resulting product is called *beer*.

## 4. Distillation

The beer is then distilled by the application of heat to boil off the ethanol and separate it from the non-fermentable content.

## 5. Dehydration

Distillation produces ethanol, which contains 5 % water (190 proof). The water is then removed by passing the fermented solution through columns packed with molecular sieves to produce the 100 % (200 Proof) ethanol.

## 6. Non-fermentables

The non-fermentable components are separated after distillation using a centrifuge. After the centrifuge, the solids can be sold as *wet distiller grains* or dried to

produce *dried distiller grains*. The remaining liquid called thin stillage is recycled back to the slurry tanks.

## Instrumentation and installation



Vaisala K-PATENTS® Process Refractometer PR-43-G is installed directly in the main line or in a by-pass. The refractometer provides continuous indication of the concentration throughout the process with excellent repeatability.

When bioethanol is produced from molasses, the refractometer can be used for molasses dilution. The automatic control provided by the refractometer ensures the optimal concentration for fermentation is always achieved.

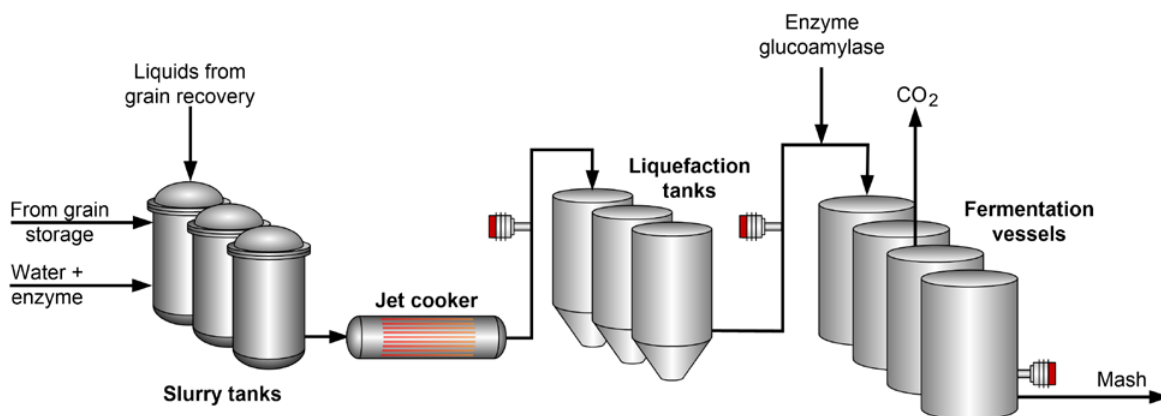
Another refractometer installed directly in the fermenter to provide real-time information on the degree of fermentation and end-point of the reaction.

The refractometer is also used to monitor and control different purification operations. For instance, the refractometer measures the concentration of the top and bottom products of the distillation columns and sends an instant signal to the process controller to adjust the reflux and boil-up as necessary. This optimizes distillation efficiency and ensures the product specifications are met.

Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Low: 0-60 % C <sub>2</sub> H <sub>5</sub> OH. High: 90-100 % C <sub>2</sub> H <sub>5</sub> OH.





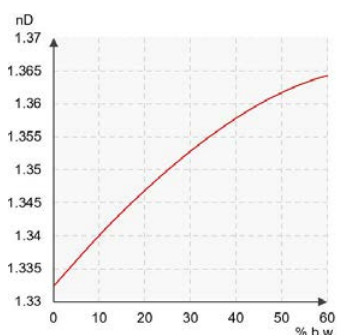
## ETHANOL (C<sub>2</sub>H<sub>5</sub>OH)

### Typical end products

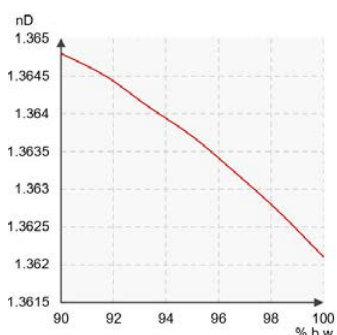
Bioethanol fuel (ethyl alcohol) additive for gasoline.

Chemical curve: Ethanol R.I. per Conc% b.w. at Ref. Temp. of 20 °C

### Range 0-60 %



### Range 90-100 %



## Introduction

After the corn is ground to the correct size, water and the first enzyme (alpha-amylase) are added to breakdown the starch. During the first step, called *gelatinization*, hot water (85 °C or 185 °F) is mixed with the flour for 20-60 min, followed by the addition of superheated water at 105 °C (221 °F). During this

step the starch absorbs water, swells up and loses its compact crystalline structure. This enables the first enzyme, Alpha-amylase, to break the long starch chains into smaller variable length chains called *dextrins* via hydrolysis. This process usually takes between 2-4 hours to complete and is referred to as *liquefaction*.

The second enzyme, glucoamylase, is added during the passage to the fermentation tank. This enzyme breaks the dextrins into individual glucose molecules. It usually takes 40 hours or more to complete this stage of the process. This step is called *Simultaneous Saccharification Fermentation* (SSF).

During fermentation the yeasts convert the simple sugar into ethanol. This step takes between 40-70 hours.

## Application

The dissolved sugar is monitored in the liquefaction tanks by Vaisala K-PATENTS® Process Refractometer PR-43-GP to ascertain the required hold time. Also, a refractometer can be used to monitor and control the quantity of the required Alpha-amylase enzyme.



The ethanol and/or sugar concentration is measured during fermentation to ensure the maximum yield of ethanol. The ethanol concentration is typically 0-15 % by weight.

## Instrumentation and installation

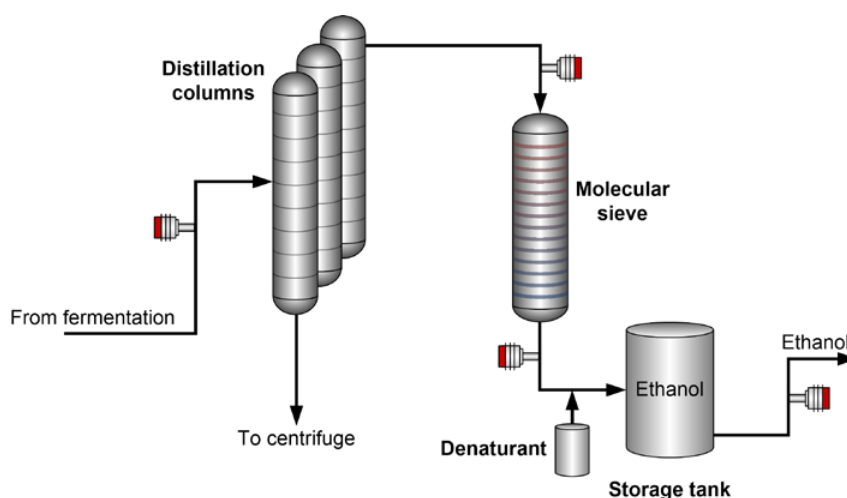
The Process Refractometer PR-43-G is used to minimize the holding time and enzyme use. The refractometer is installed on either side of the liquefaction tanks. The typical measurement range is between 14-35 Brix at a temperature of 85 °C (185 °F).

At the fermentation tanks, the refractive index of the liquid changes because of the conversion of sugars to Ethanol. The refractometer is installed directly in the fermentation tanks to monitor progress of the reaction and ethanol level. Typical measurement range is 0-15 % ethanol.

Automatic prism wash with high-pressure water is recommended in this application.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Low: 0-60 % C <sub>2</sub> H <sub>5</sub> OH. High: 90-100 % C <sub>2</sub> H <sub>5</sub> OH.

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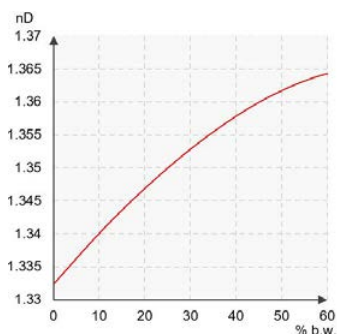
## ETHANOL (C<sub>2</sub>H<sub>5</sub>OH)

### Typical end products

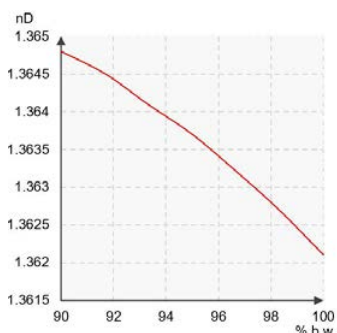
Bioethanol fuel (ethyl alcohol) additive for gasoline.

Chemical curve: Ethanol R.I. per Conc% b.w. at Ref. Temp. of 20°C

### Range 0-60 %



### Range 90-100 %



## Introduction

Following fermentation, further ethanol concentration is required. This is achieved through distillation and by using molecular sieve columns. Once the ethanol is 100 % pure, a denaturant, usually 2-5 % gasoline,

is added to render the alcohol unfit for human consumption. It is then ready to be placed in storage tanks to wait for transportation and distribution.

## Application

Downstream from the fermenters, the ethanol concentration is typically 10-15 % by weight, and needs to be concentrated to 95 % (190 Proof) in a distillation column. This usually takes between 20-24 stages and is limited by an azeotrope. The ethanol concentration is monitored at the feed and product lines of the distillation process to increase efficiency by enabling less energy consumption.

Since 100 % purity cannot be accomplished with distillation, molecular sieve columns are used to remove the remaining water content. This is called dehydration. The ethanol concentration must be free of water for gasoline to be mixed with it. The ethanol concentration is monitored following the molecular sieving to ensure that the ethanol produced is 100 % (200 Proof).

The ethanol concentration should also be monitored before getting the product ready for transportation. This ensures high quality of the delivered product.

## Instrumentation and installation


Vaisala K-PATENTS® Process Refractometer PR-43-GP is installed on the feed to distillation. Typical measurement range is 10-15 % b.w. A high-pressure prism wash may be required since this stream still contains non-fermentables. The refractometer also monitors the concentration of the top product (ethanol), providing essential information for distillation control and optimization. The typical

measurement range is 93-97 %. A prism wash is not required in this installation point.

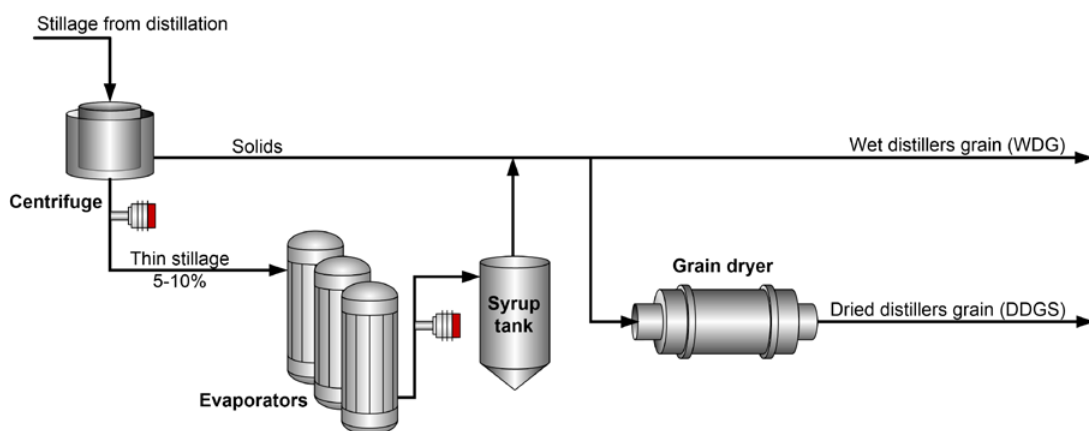
The refractometer is also installed following the molecular sieves, for monitoring the concentration of the final product. The measurement range in this point is usually 98-100 %. The refractometer can also be

used for quality control. The refractometer is installed after the storage tank to guarantee the correct concentration of the delivered product.

Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Low: 0-60 % C <sub>2</sub> H <sub>5</sub> OH. High: 90-100 % C <sub>2</sub> H <sub>5</sub> OH.

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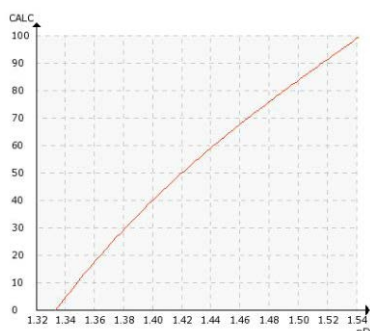


## ETHANOL (C<sub>2</sub>H<sub>5</sub>OH)

### Typical end products

Thin stillage for distiller's dried grains (DDGS), cattle feed.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20 °C



### Introduction

The remaining stillage or residues left from the distillation process contain nutrients, minerals and unfermented sugars. These are valuable by-products, providing an additional source of revenue as animal feed. The average stillage amount produced in the bioethanol process is approximately 13 h/L per h/L of bioethanol.

Decanting centrifuges mechanically dewater the spent grains carried in the stillage. Thin stillage, containing both dissolved and suspended solids, is produced as the *centrate*. Some of the thin stillage is recycled as backset to the fermentation process. The remaining thin stillage, normally with a concentration ranging between 5 and 10 % b.w., is concentrated in an evaporator to produce the condensed soluble ingredient of distiller's dried grains (DDGS).

The decanting centrifuges simultaneously produce a wet cake of spent grains, or WDG (wet distiller's grains), which can be marketed as cattle feed or

processed further in a dryer creating DDGS, a higher revenue product.

### Application


Falling film evaporators are used to pre-concentrate the thin stillage. The evaporators normally achieve a final product concentration between 28 % and 42 %. For optimization of the evaporation process, the syrup concentration should be monitored at the evaporator inlet and outlet, resulting in reduced energy consumption and costs.

### Instrumentation and installation

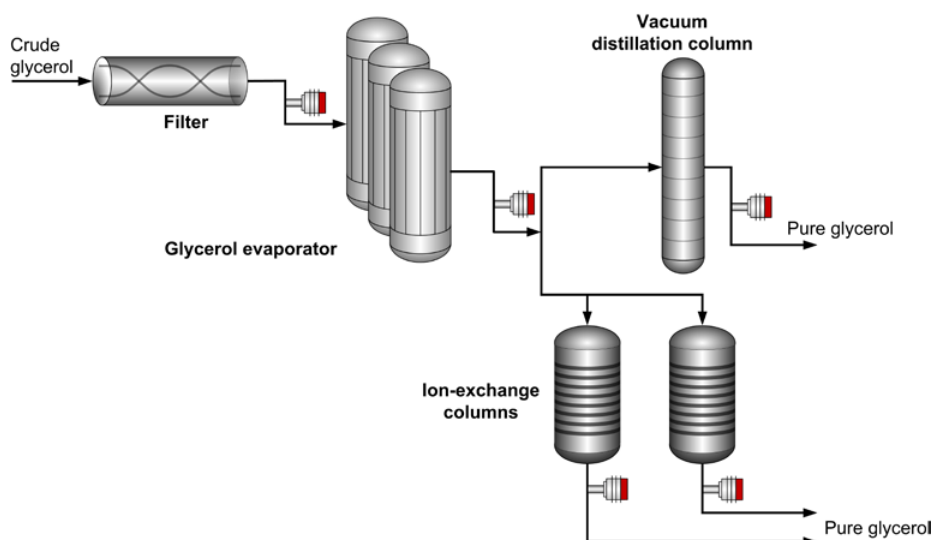
Vaisala K-PATENTS® Process Refractometer PR-43-GP provides accurate concentration measurement of the thin stillage and syrup products. The refractometer is installed in-line on both inlet and outlet streams of the evaporation station.

The refractometer provides Ethernet and 4-20 mA output signals that can be used for real-time control of the evaporators. The signal from the refractometer is used to automatically adjust the inlet flow or the heat-flow to the evaporators in order to obtain the desired concentration continuously.

Typical measurement range at the inlet is 5-10 % and outlet range is 28-42 %.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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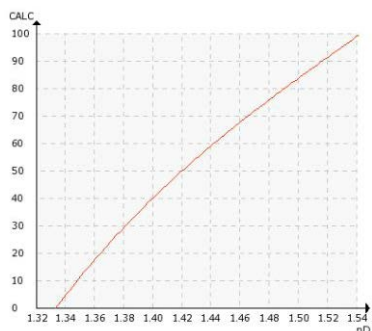


## GLYCEROL (GLYCERIN), C<sub>3</sub>H<sub>5</sub>(OH)<sub>3</sub>

### Typical end products

High purity glycerol for food, drugs and cosmetics.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



## Introduction

Glycerol (Glycerin) is a by-product of the petrochemical, animal fat and vegetable oil-based biodiesel industries. However, crude glycerol derived from biodiesel production is of low value because of impurities. Further refining of crude glycerol depends on the economics and the availability of facilities. Larger scale biodiesel producers refine their crude glycerol and market it to other industries.

For every 9 kg (19.8 lbs) of biodiesel produced, about 1 kg (2.2 lbs) of a crude glycerol by-product is formed. Effective usage, or conversion, of crude glycerol with specific products will offset biodiesel production costs.

Glycerol is generally treated and refined through filtration, chemical additions and fractional vacuum distillation, yielding a variety of commercial grades. When destined to be used in food, cosmetics,

and drugs, further purification such as bleaching, deodorizing, and ion-exchange will be required to remove trace properties.

## Application

### Physical Refining

The first step in physical refining is to remove fatty, insoluble or precipitated solids by filtration and/or centrifugation. This removal may require pH adjustment.

Then, the water is removed by evaporation. All physical processing is typically conducted at 65-90 °C (149–194 °F) as glycerol, at these temperatures, is less viscous but remains stable.

### Glycerol Purification

The final glycerol purification is completed by using vacuum distillation with steam injection, followed by an activated carbon bleaching. This is a well-established technology but has the disadvantage of being capital and energy intensive. Vacuum distillation of glycerol is best suited in plants with a production above 25 kilotons per day.

Ion-exchange purification of glycerol is an attractive alternative to vacuum distillation for smaller capacity plants. The ion-exchange system uses cation, anion and mixed bed exchangers to remove catalyst and other impurities. For this, the glycerol is first diluted with soft water to a 15 % to 35 % glycerol-to-water solution. The ion-exchange is followed by vacuum


distillation or flash drying. After water removal, there is often a partially refined glycerol of 85 %.

## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is installed at the inlet and the outlet of the evaporator. The continuous information from the refractometer helps to achieve the target glycerol concentration, improve evaporation efficiency and reduce energy cost.

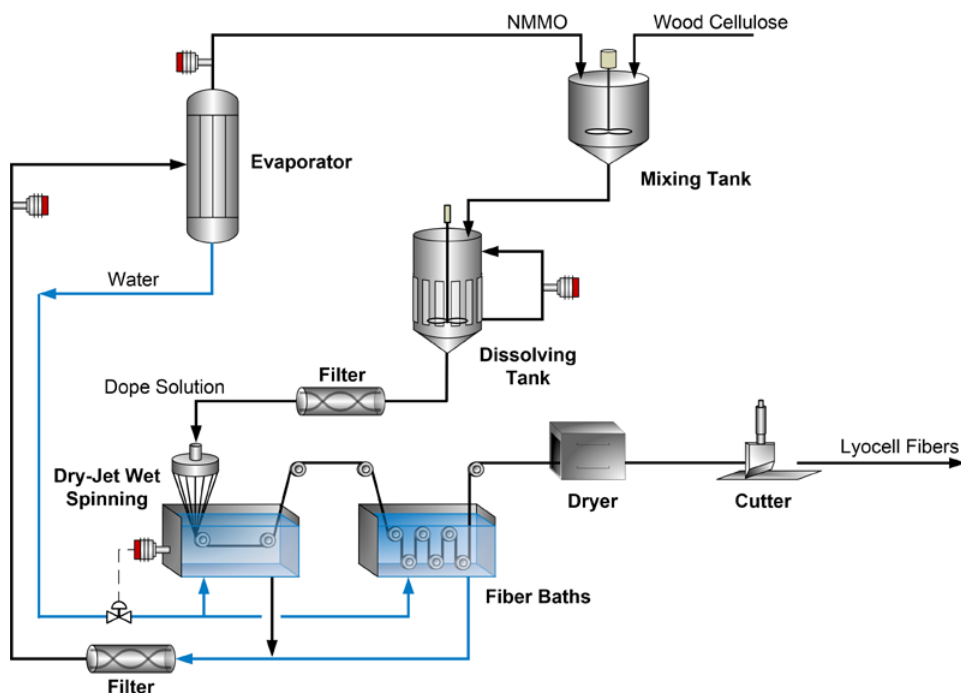
The refractometer can also be installed in the distillation or ion-exchange step to monitor the product concentration.

If ion-exchange columns are used, the refractometer can be used to detect the liquid interface during the regeneration process, thus allowing for an automated operation.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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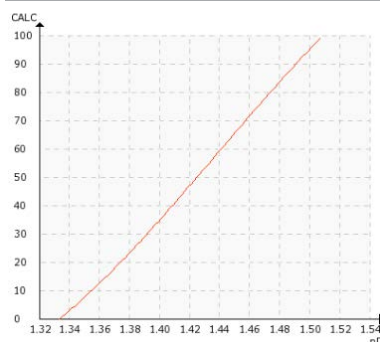


## N-METHYLMORPHOLINE N-OXIDE (NMMO)

### Typical end products

Lyocell fibers for the manufacture of apparel and non-apparel products.

Chemical curve: NMMO R.I. per Conc. % b.w. at Ref. Temp. of 20°C



### Introduction

Regenerated cellulose fibers (also known as *rayon fibers*) are made by chemical modification of the cellulose contained in wood pulp, so that it can be dissolved and spun into fine fibers in a solution.

Typical rayon fibers are viscose, cellulose acetate and Lyocell fibers. Lyocell fibers stand out from this group as they are smoother, stronger and greater wet-strength. These excellent physical properties make them suitable for industrial applications as well as for uses in apparel production.

### Application

The Lyocell Process is more straightforward than other regenerated fibers processes as it does not involve the formation of a chemical derivative. In this process, the wood pulp is dissolved directly in an organic solvent. The solvent used for Lyocell production is N-methylmorpholine N-oxide (NMMO). At a temperature of 100 °C (212 °F), an aqueous solution of NMMO is a very effective solvent for cellulose.

The Lyocell Process starts by debarking and chipping wood. Wood chips are mixed with an aqueous solution of NMMO to dissolve the cellulose. The concentration and temperature of the solution play a significant role in the process, e.g. if the water content is too high (above 15 %) cellulose will not dissolve.

The solution is initially prepared to 50-60 % of NMMO and then concentrated by evaporation in a heated dissolving vessel to give a dope solution of composition NMMO:water:cellulose (about 76:10:14). The temperature is kept between 90 and 120 °C.

After a filtration step, the dope solution moves on to dry-jet wet spinning where the solution is extruded through a spinneret to make filaments. The filaments are formed in an air gap before entering a spinning bath for complete coagulation. The spinning solution can be water or a very dilute solution of NMMO.

The fibers are then washed, dried, and cut to obtain the final Lyocell fibers. The coagulating liquid is purified and concentrated to recover the NMMO for reuse in the process. Recovery rate can be up to 99.5 % of the solvent.

## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-G continuously monitors the concentration of the dope solution and the solvent at various stages of the process. This guarantees maximum productivity and high fiber quality.



The refractometer is installed in the dissolving tank outlet or directly in the dissolving tank where the cellulose is dissolved in NMMO while the solution is heated and concentrated.

Changes in the refractive index value of the solution are used to monitor the dissolving process. This ensures that the correct concentration and complete cellulose dissolution is achieved. Undissolved cellulose in the spinning dope results in poor spinnability and lower fiber quality.

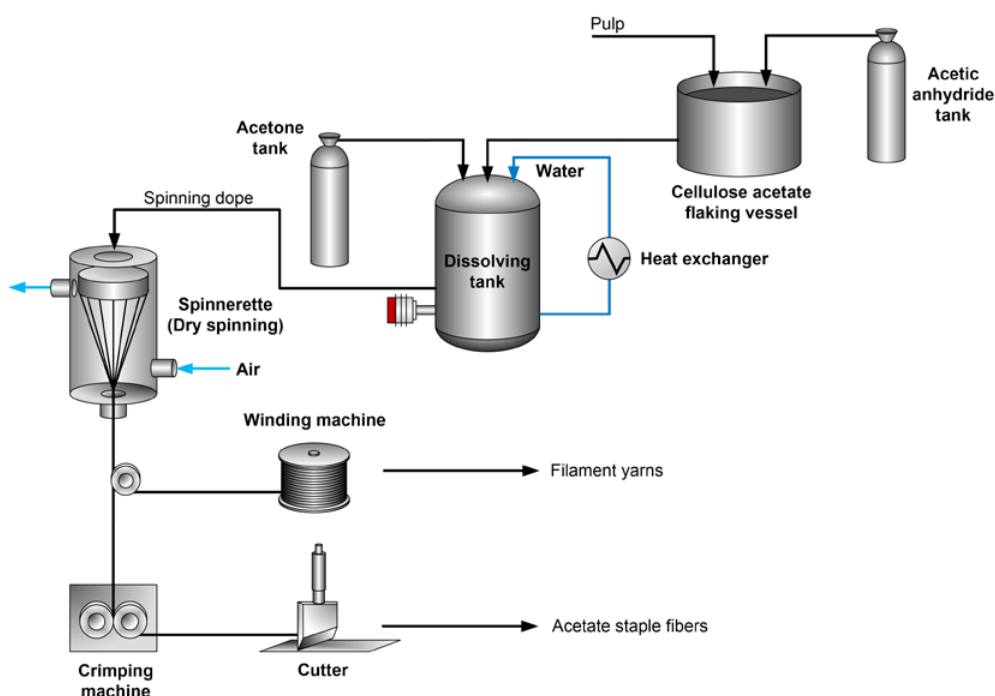
During coagulation, the solvent diffuses out from the doping solution increasing the concentration of the bath. The PR-43-G continuously measures the spinning bath concentration as the fibers are produced. The refractometer's signal is used to maintain the bath at the optimal concentration through the addition of water.

NMMO is a very costly solvent, and its recovery is essential for the economic viability of the process. The refractometer can also be installed in the solvent recovery phase to ensure the correct concentration, and to maximize the solvent recovery rate for later reuse in the process.

The refractometer can be installed in large and small size pipes or directly in vessels. The refractometer also provides Ethernet and 4-20 mA output signals for real-time and automated process control.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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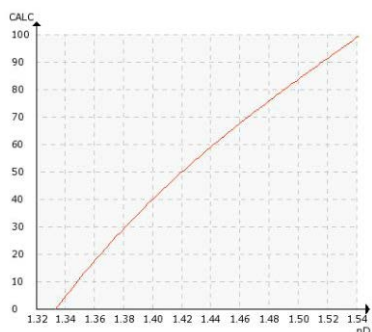


## CELLULOSE ACETATE, CELLULOSE TRIACETATE (CTA)

### Typical end products

Cellulose acetate fibers.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

There are two types of cellulose-based fibers: regenerated or pure cellulose (such as the fibers from the cupro-ammonium process) and modified cellulose (such as the cellulose acetates and rayon).

Acetate fiber is a synthetic fiber, in which the forming substance is cellulose acetate. When no less than 92 % of the hydroxyl groups are acetylated, the term triacetate may be used as a generic description for the fiber.

### Application

Acetate is derived from cellulose by breaking down wood pulp (dissolving pulp) into purified cellulose. Cellulose acetate dope is produced by reacting the purified cellulose with acetic acid and acetic anhydride, whilst using sulfuric acid as a catalyst. The cellulose acetate flakes are then dissolved into acetone for extrusion. Then, filaments emerge from the spinneret and the solvent is evaporated in warm air.

### Production process

Purified cellulose from wood pulp or cotton linters is mixed with glacial acetic acid, acetic anhydride and a catalyst (sulfuric acid). The mixture is put through a controlled 20 hour partial hydrolysis to remove the sulfate and the required amount of acetate molecules to obtain the product's desired properties. In this step a precipitate of acid-resin flakes is obtained. The flakes are dissolved in acetone and the solution is filtered.

The resulting solution is the *spinning dope* or *spinning solution*. The spinning solution is extruded in a column to form filaments which are dried with dry air. The solvent is recovered and purified for reuse. The filaments are stretched and wound onto beam cones or bobbins to obtain the final product.

### Fiber Formation Method: Extrusion and Spinning

After being formed, cellulose acetate is dissolved into acetone for extrusion. As the filaments emerge from the spinneret, the solvent is evaporated in warm air (dry spinning) producing fine filaments of cellulose acetate.

The liquid substance of cellulose is forced through a metal cap, or nozzle, called a spinneret. The spinneret is perforated with small holes and a filament is extruded through each one. The extruded filament gets solidified by a liquid bath as it emerges from the spinneret. The number of perforations in a spinneret varies from 1 to 20000 and filaments of equal gauge are produced simultaneously. Subsequently, filaments are twisted together to form yarn.

As the filaments emerge from the holes in the spinneret, the liquid polymer becomes rubbery and then solidifies. This process of extrusion and solidification of endless filaments is called *spinning*. It should not be confused with the operation by the same name, used for producing natural yarn, where the shorter lengths of natural fiber are twisted into yarn. There are four methods of spinning synthetic fiber filaments: wet, dry, melt and gel spinning.

### Stretching and Orientation

While extruded fibers are solidifying, or in some cases even after they have hardened, the filaments may be drawn to impart strength. Drawing pulls the molecular

chains together and orientates them along the fiber axis, creating a considerably stronger yarn.


### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is used in the dissolving tank to monitor the concentration of the dope solution prior to the spinning of the fibers. The cellulose is dissolved in a dope solution, which consists of dissolved cellulose in acetone. Traditionally, the concentration of the dope is measured by taking samples and analyzing them in the laboratory. This method is not optimal as acetone evaporates quickly, giving false results.

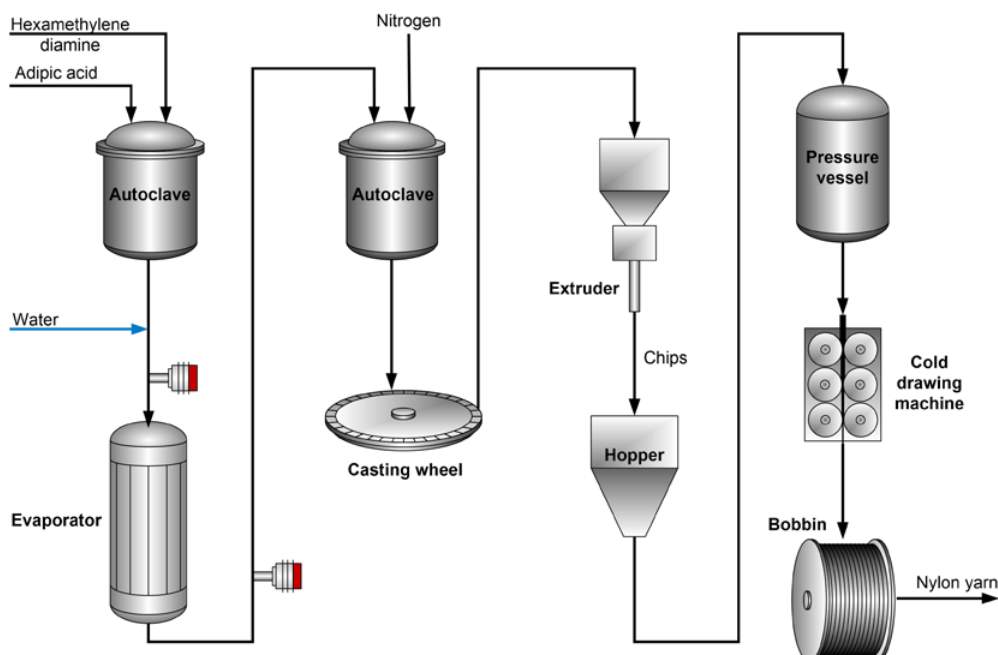
The refractometer is installed in-line for continuous measurement and control of the dope concentration. A product of high quality is obtained only if the refractive index value is maintained within a pre-determined limit. The refractometer is installed either in a by-pass loop with an external heat exchanger or directly in the dissolving tank.

Another refractometer can be installed in the solvent recovery area to maximize acetone recovery and reduce operation costs.

Typical measurement range in this application is 20-30 % and the process temperature is about 20-60 °C (68-140 °F). Appropriate equipment with hazardous and intrinsic safety approvals are available when required.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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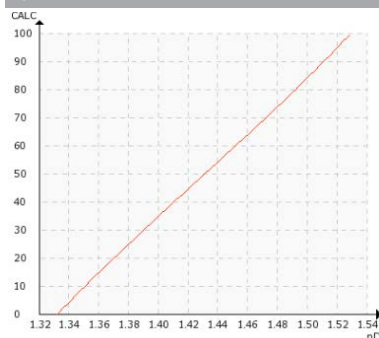


## POLYAMIDE FIBER, NYLON SALT

### Typical end products

Synthetic fibers, Nylon fibers, Nylon 6, Nylon 6-6.

Chemical curve: Nylon salt R.I.per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

Nylon 6-6 was the first commercially made all-synthetic fiber. The product resulting from the polymerization reaction of adipic acid and hexamethylene diamines is called Nylon 6-6. The name comes from the molecular chains of the two raw chemical components, containing six carbon atoms each.

### Application

The reaction between adipic acid and hexamethylene diamine produces hexamethylene diammonium adipate, commonly called *nylon salt*. It is essential for the material to be polymerized so that high quality fibers, with very few impurities, can be achieved.

Different types of nylons can be made by using a variety of processes.

In the batch process, the hexamethylene diammonium adipate solution is concentrated in an evaporator and acetic acid is added to stabilize the chain length. After evaporation, the salt solution is heated and the remaining water removed.  $\text{TiO}_2$  dispersion is added and the polymerization takes place. After the polymerization is completed, the molten viscous polymer is forced out through the bottom of the autoclave, onto a casting wheel and extruded as rapidly as possible.

Nylon is also produced by continuous processing, which is more economical for large quantity production, whereas the flexibility of the batch process allows end-product variations.


Nylon 6, or *caprolactam*, is a polymeric fiber derived from only one constituent, caprolactam. It has a lower melting point than nylon 6-6 but has superior dyeability, elasticity and resistance to light, etc. Other types of nylons also have useful differing properties.

### Instrumentation and installation

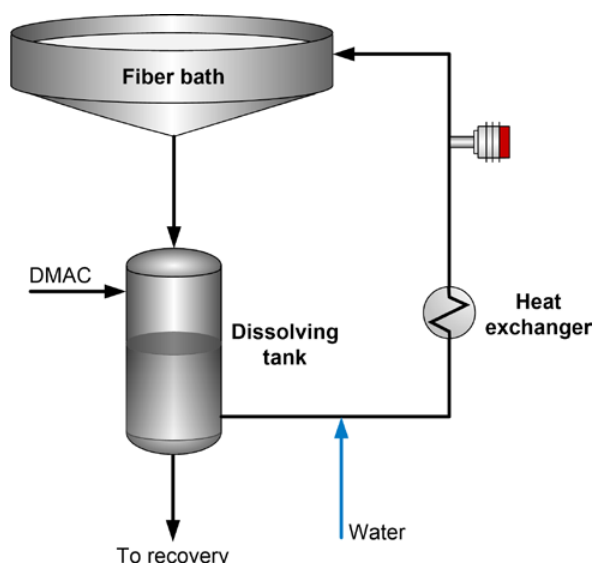
Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to monitor and control the nylon salt, caprolactam and polymer solution concentrations.

The refractometer is installed after the evaporator to ensure the target concentration is achieved. Another refractometer can be installed before the evaporator for real-time monitoring of the evaporation efficiency. The refractometer provides Ethernet and 4-20 mA output signals that can be used for automatic control of evaporation, for instance, by regulating heat-flow or the feed to the evaporation.

Typical refractive index measurement range for nylon salt is 1.3300-1.4100, and the process temperature is 60 °C (140°F). Typical refractive index measurement range for caprolactam is 1.3370-1.3570.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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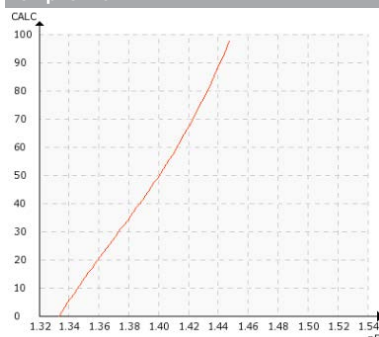


## DIMETHYL ACETAMIDE (DMAC)

### Typical end products

Polyurethane elastic fibers, elastane, spandex.

Chemical curve: Dimethyl acetamide R.I.per Conc% b.w. at Ref. Temp. of 20°C



Next, the polymer is dissolved into dimethyl acetamide (DMAC) and then chain-extended or coupled through the use of glycol, diamine or water. Other solvents can also be used, for example, dimethylformamide (DMF) and nitric acid (HNO<sub>3</sub>). The final polymer is converted into fibers by a *spinning process*.

DMAC is an excellent solvent for a large variety of organics and is widely used as such for fibers, adhesives and dyes.

### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-G is perfectly suited to control the spinning bath concentration.

The refractometer measures continuously the concentration of the bath in order to keep it at the optimum level and avoid a decrease in product quality.



Typical measurement range in this application is 40-60 %.

### Introduction

Spandex is the generic name for the synthetic fiber, whose fiber-forming substance is a long chain of a synthetic polymer. It comprises of at least 85 % of segmented polyurethane. Common trade names for these fibers are *LYCRA* (DuPont), *DORLOSTAN* (Bayer), *SPANZELLE* (Acordis) and *VYRENE* (US Rubber).

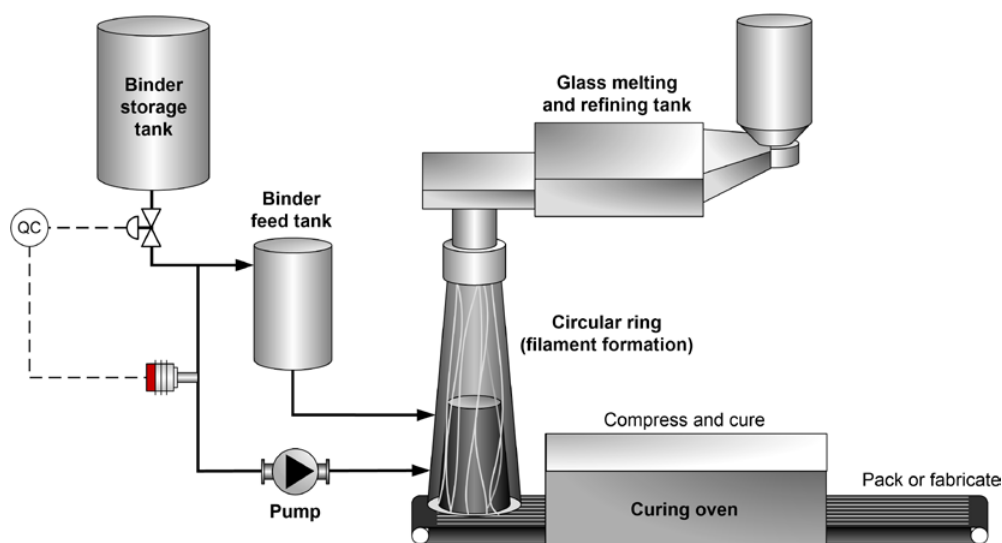
### Application

Typically, the spandex fiber structure is achieved by reacting di-isocyanates with long chain glycols, which are usually polyesters or polyethers.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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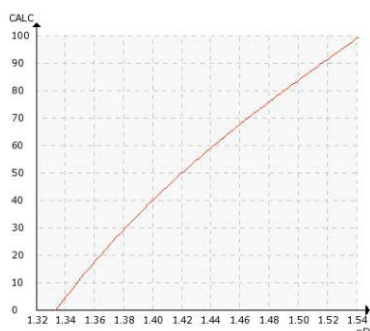


## FIBERGLASS BINDER SOLUTION

### Typical end products

Fiberglass.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Fiberglass, or glass fiber wool, is a material made from extremely fine short fibers of glass. These fibers are produced by spinning or blowing molten glass (silica).

### Application

In the manufacture of fiberglass wool, an adhesive binding solution is applied to hold the fibers together. These binders are also used as a coloring agent for the product. This coloring provides brand identification in the highly competitive insulation materials market.

The fiberglass strands, which make-up the insulation wool, are produced at the forefront of the glass furnace by flowing a stream of molten glass vertically down through a circular ring. Then, air is blown through nozzles to atomize the molten glass into fine strands, onto which the binder is sprayed. The final

bonded wool is collected and packaged to be used as insulation. The residual binder waste solution is collected and pumped back to a holding tank, where it is allowed to settle and is then filtered several times to remove particles.

Once the filtering process is complete, a dissolved solids measurement is taken to determine the quantity of the resin, ammonium sulfate, phenol and other components present, so the filtered residual binder solution can be accurately remixed into the fresh binding solution.

Spent binder solution cannot be released into the local sewage systems, due to the color agents being difficult to neutralize. Spent binder solution is therefore collected from the production lines for recycling.


### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP is used to determine the amount of dissolved solids in the spent binder solution in order to control the quantity of fresh binder to be added. Knowing the spent solution concentrations makes it possible to compute the appropriate amount of new binder in order to maintain the correct formulation. This reduces wastage, resulting in lower production costs. The binder is a water-soluble acrylic substance, which possesses a direct correlation between refractive index and binder concentration.

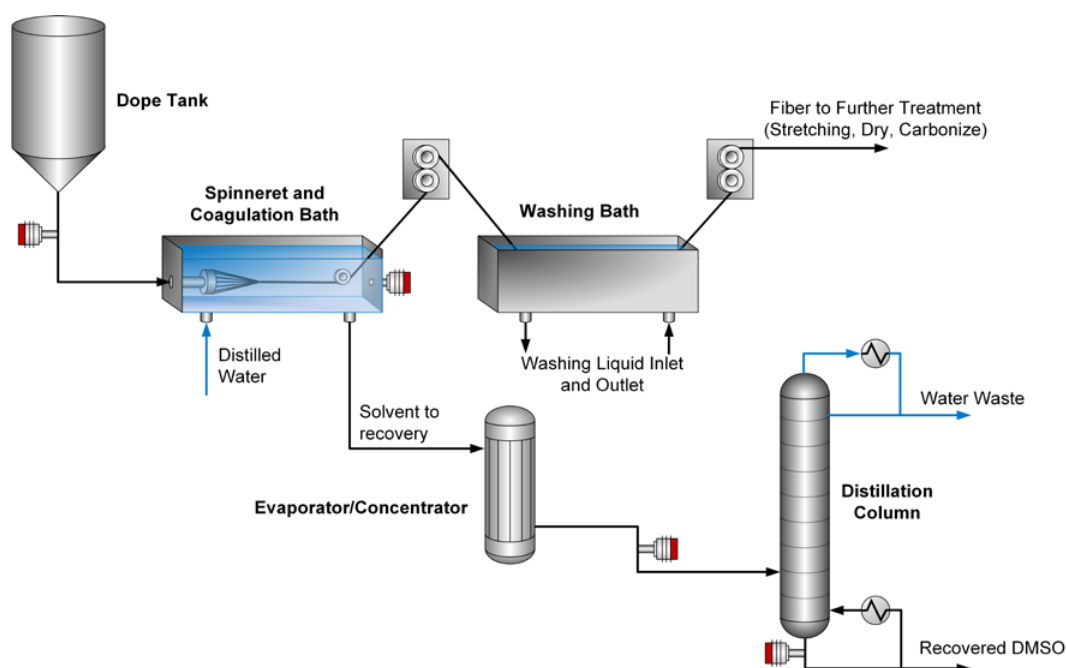
The refractometer is mounted in the recycle line to the binder feed tank. The output signal from the refractometer is used to control the addition of fresh binder to the static mixer in order to maintain the final

wash coat concentration at approximately 8-10 % b.w. The process temperature is between 32 °C and 40 °C (90-104°F).

An automatic high-pressure water prism wash system is recommended due the adhesive nature of the binder.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic prism wash	<b>Prism wash with high pressure water.</b> The components of a high-pressure water system are a sensor with integral water nozzle mounted at the sensor head, a high-pressure pump together with a power relay unit and an indicating transmitter equipped with relays.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.

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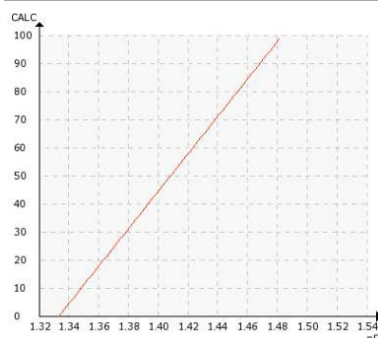


## DIMETHYL SULFOXIDE (DMSO), DIMETHYL FORMAMIDE (DMF) AND DIMETHYL ACETATE (DMAC)

### Typical end products

Carbon fibers for different applications such as aerospace, sporting goods, wind turbine blades, automotive.

Chemical curve: Dimethyl Sulfoxide R.I. per % b.w. at Ref. Temp. of 20 °C



### Introduction

Carbon fiber is a long, thin strand of material that contains over 90 % of carbon by weight. The raw material used to make carbon fiber is known as the *precursor*.

Carbon fibers are obtained by pyrolysis of an appropriate precursor fiber. Polyacrylonitrile (PAN) is the predominant precursor for carbon fiber due to a superior strength and stability, and higher carbon yield. About 90 % of the carbon fiber produced is

made from PAN and the remaining 10 % is made from rayon or petroleum pitch.

PAN-based carbon fiber is widely used in many industries such as aviation, aerospace, sporting goods, and construction.

### Application

The first step in the production of PAN-based carbon fibers is spinning the PAN co-polymer to form the fibers. Because PAN decomposes before melting, it is necessary to make a spinning solution or *dope solution* with a solvent in order to be able to spin the material into a fiber. Common solvents are dimethyl sulfoxide (DMSO), dimethyl formamide (DMF), dimethyl acetate (DMAc) and sodium thiocyanate. The concentration of the dope solution is usually 15-25 wt-% and should be carefully controlled to ensure its ability to form filaments of high mechanical strength.

Spinning can be done through different methods, such as dry-jet wet spinning or wet spinning. However, only the wet spun PAN fiber is used as a precursor for carbon fiber, as it contains a co-polymer (e.g. itaconic acid) that helps the carbonation process.

In wet spinning, the dope solution is passed through a *spinneret* into a coagulation bath to form filaments.

The fibers solidify when the solvent diffuses away, leaving behind the PAN fibers.

The spinning step is important because the internal atomic structure of the fiber is formed during this process. The quality of the final fiber depends on different process parameters, such as the composition of the dope and the temperature and concentration of the coagulation bath.

After the fibers are formed, they are further treated by stretching, stabilization, carbonation and graphitization. The final carbon fiber is then ready for use in different applications. The solvent-water mixture from the coagulation bath is sent to the recovery area where water and solvent are separated. The recovered solvent is used again in the dope preparation to save operating costs.

## Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GP monitors in-line the concentration of solvent at different stages of the process.


The refractometer is installed directly on the pipe after the dope tank to measure the concentration of the PAN solution before it is pumped into the coagulation

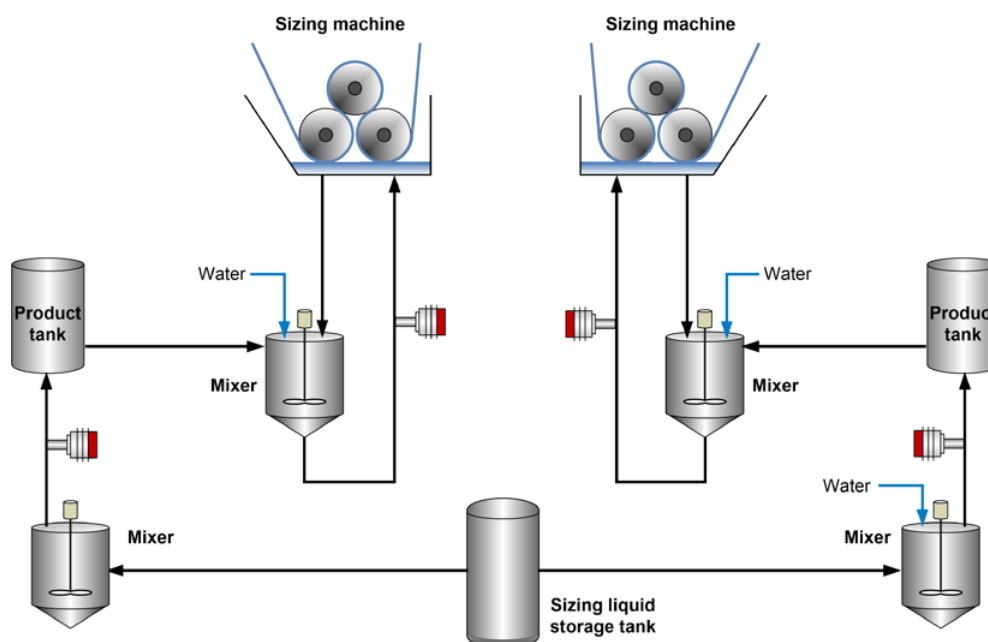
bath. The real-time information by the refractometer helps to maintain the dope concentration within the desired range and to obtain precursor fibers with excellent properties.

As the fibers are formed and passed through the coagulation bath, the solvent diffuses out changing the concentration of the bath. The refractometer continuously monitors the solvent concentration in the bath, and provides real-time information to keep the concentration at optimal through the addition of water.

At the solvent recovery stage, the refractometer monitors the concentration after evaporation and distillation. The refractometer provides Ethernet and 4-20 mA output signals that can be used as feedback for automated control. The instant and accurate measurement from the refractometer ensures the target concentration is always achieved while reducing energy costs.

The refractometer reduces the need for sampling and laboratory tests. The refractometer is factory calibrated and does not require re-calibration. Automatic prism wash may be needed in this application.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

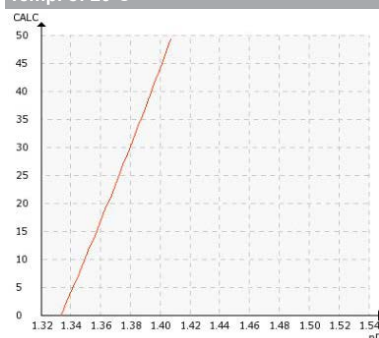


## SIZING AGENTS, STARCH, CARBOXYMETHYL CELLULOSE (CMC), POLYVINYL ALCOHOL (PVA)

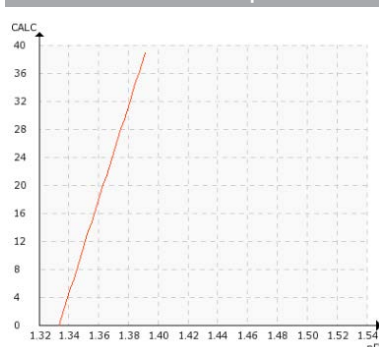
### Typical end products

Textile yarn.

Chemical curve: Polyvinyl alcohol R.I.per Conc% b.w. at Ref. Temp. of 20°C



Chemical curve: Starch R.I.per Conc% b.w. at Ref. Temp. of 20°C



### Introduction

The yarn sizing process is essential in reducing breakage and thus avoiding stoppages during weaving. Improved quality, as well as smoother surface finish, will be achieved by sizing the strength and abrasion resistance of the yarn. Different types of water soluble polymers known as textiles sizing agents/chemicals are used to protect the yarn. Some examples are modified starch, polyvinyl alcohol (PVA), carboxymethyl cellulose (CMC) and acrylates. Mixtures of the former mediums and other chemical components are also used.

### Application

Before the yarn can be woven, it needs to be strengthened to withstand the stress sustained in weaving on a high-speed industrial looms. For this purpose, the yarn is passed through a sizing bath that contains the sizing medium, mixed with water and other additives depending on the formula.

The sizing medium is mixed to achieve the correct level of concentration before loading it into the sizing bath. If starch is used, the mixture has to be cooked before it can be used. Usually the cooking or mixing station is situated close to the sizing bath and may serve several sizing lines.



After the initial mixing, the product is pumped into a product tank. From there, it passes to a final mixing tank, where it is mixed to the required concentration level before loading into the sizing bath. The mixing tanks are equipped with a load gauge so a specific quantity of the medium can be mixed for each textile batch. After the initial mixing and cooking, the concentration of the medium is typically 16-18 % and after the final mixing it is at 6-10 %. Recycled sizing medium can sometimes be added to the final mixing to adjust the concentration of specific yarns.

in a circulation loop to provide adequate product flow across the prism. Typical concentration levels are 5-15 %.

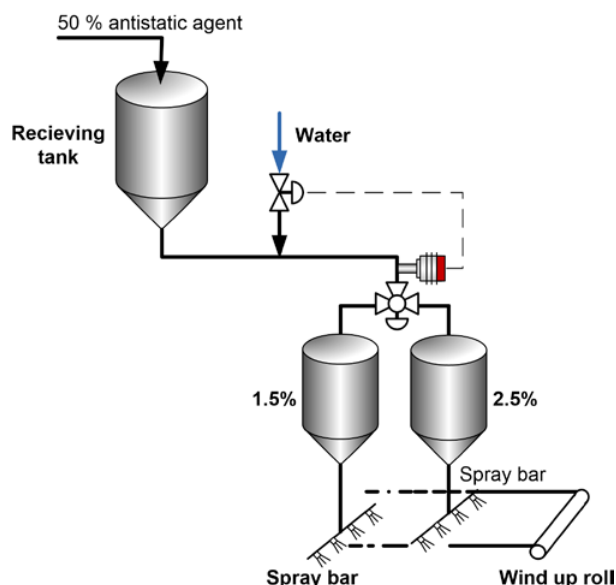
The refractometer is also used to measure concentration in the product tanks, or during mixing, to ensure correct concentration levels. A steam cleaning system is recommended for the above installation positions.

## Instrumentation and installation

The concentration of the sizing baths need to be monitored as it may fluctuate depending on evaporation and yarn absorption. Vaisala K-PATENTS® Process Refractometer PR-43-G is usually mounted

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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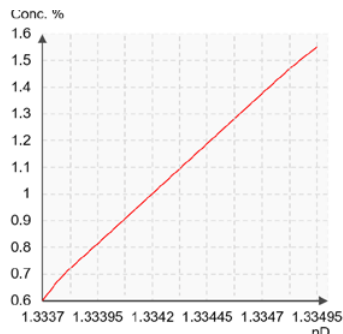


## ISOPROPYL ALCOHOL BASED SOLUTION

### Typical end products

Housewrap, vehicle covers, envelopes, medical and industrial packaging, protective apparel.

Chemical curve: Isopropyl alcohol based solution R.I. per Conc% b.w. at Ref. Temp. of 25°C



### Introduction

Polyethylene fiber is known for its high strength and durability features. The material is used in a variety of applications, for example, Tyvek® HouseWrap, vehicle covers, envelopes, medical and industrial packaging, and as protective apparel.

### Application

The polyethylene sheet is breathable and water repellent, prevents infiltration of air and water and allows vapor to pass through. The material is also strong, durable and puncture resistant. The sheet is made by spinning extremely fine high-density polyethylene fibers fused together to produce a strong uniform web.

Inside the sheet antistatic agent is applied as a thin microporous film on a coarse fabric consisting of millions of small pores. An isopropyl alcohol-based solution (Zelec®) is used as the antistatic agent.

Before the antistatic agent is applied, it must be diluted to a 1.5 or 2.5 % solution by the addition of water.


### Instrumentation and installation

Vaisala K-PATENTS® Process Refractometer PR-43-GC is used to measure the concentration of antistatic agent after it is diluted, and before it is applied to the polyethylene sheet.

The refractometer is installed in a pipe bend to control the concentration of the diluted agent before it is forwarded into 1.5 % or 2.5 % solution tanks. The antistatic solution is then applied to the polyethylene sheet to form a thin film.

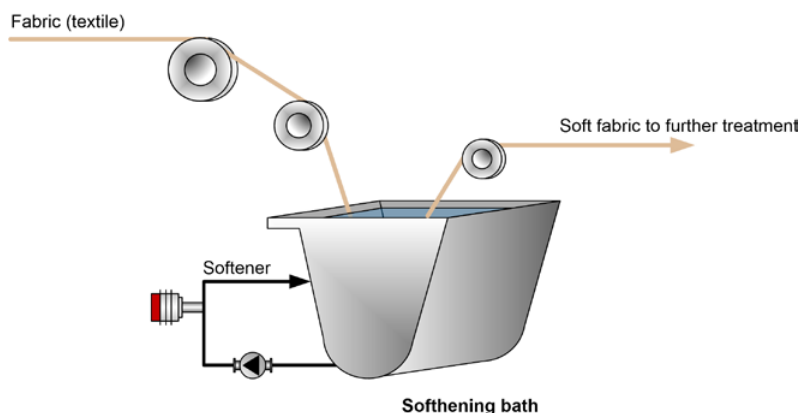
The output signal from the refractometer can be used to control the water feed valve for an automated dilution control. The accurate measurement from the refractometer ensures the target concentration is achieved.

The measurement using ultrasonic flow meter has proved to be inaccurate and unreliable. Moreover, it is maintenance intensive. Hand held and lab sample refractometry have proved to be time consuming and disruptive. The Process Refractometer is maintenance-free and provides an accurate and reliable continuous measurement of the agent solution in-line.

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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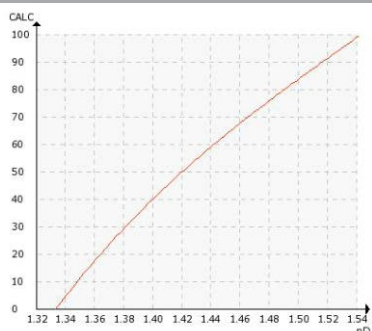


## TEXTILE SOFTENER

### Typical end products

Textile fabrics, fibers, yarn and sewing thread for different apparel items, such as leggings, stockings and nightwear.

Chemical curve: R.I. per BRIX at Ref. Temp. of 20°C



### Introduction

Textile softening is an essential finishing process in the making of fabrics. Softening treatment improves fabric's aesthetic properties and makes further processing easier. A nice, soft handle is often the decisive criteria for buying a textile.

### Application

In softening, the fabric is passed through an immersion bath containing a softener solution via rollers.


The concentration of the bath must be monitored and kept constant in order to obtain a consistent end-product quality. During the treatment, the fabric may bring some water with it (e.g. if it comes from a previous dyeing step), thus diluting the bath. In such cases, the softener needs to be replenished to bring its concentration back to the optimal level. On the other hand, if dry fabric is treated, it will absorb some water and increase the concentration of softener.

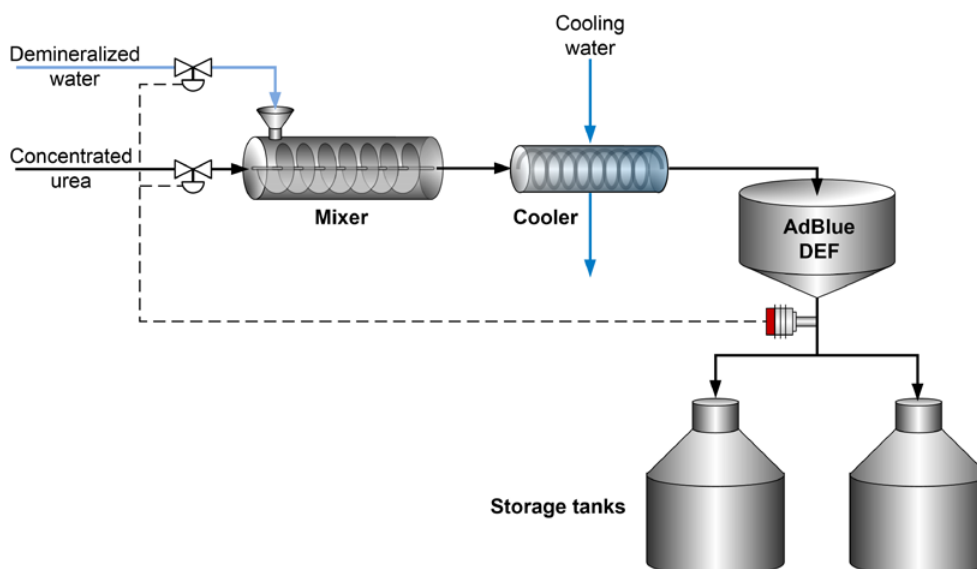
### Instrumentation and installation

Vaisala K-PATENTS® Refractometer PR-43-GC is installed in the recycling line of the bath after the pump. The refractometer is mounted on a pipe bend, for a better flow velocity and self-cleaning effect.

The refractometer provides continuous measurement of the softener concentration to alert the operators of when the concentration needs to be replenished. During the addition of softener, the refractometer also indicates when the optimal concentration has been reached in order to prevent excess use of material.

The refractometer provides Ethernet and 4-20 mA output signals that can be used for real-time control of the softening bath concentration. An automated process ensures consistent product quality and reduces operating costs.

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

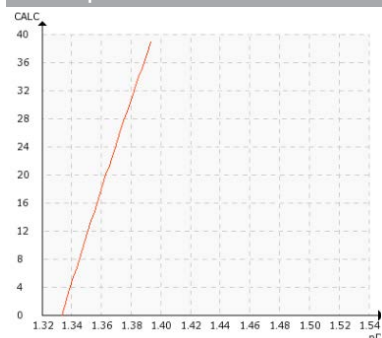


## ADBLUE (AUS32) UREA SOLUTION, DIESEL EXHAUST FLUID (DEF)

### Typical end products

Automotive grade urea solution.

Chemical curve: Automotive grade urea R.I. per Conc% b.w. at Ref. Temp. of 20 °C



### Introduction

The automotive urea AdBlue, also known by the generic name Diesel Exhaust Fluid (DEF), is the registered trademark for AUS32 (Aqueous Urea Solution 32.5 %).

AdBlue is used as a reagent to reduce the harmful emissions from the internal diesel combustion engines. In order to use AdBlue, the vehicle must be equipped with a *Selective Catalytic Reduction* (SCR) system. The fluid is passed through the SCR and into the exhaust.

### Application

As the name AUS32 suggests, AdBlue is made by using urea mixed with demineralized water resulting in a 32.5 % aqueous urea solution. It is colorless, non-toxic and safe to handle.

A stream of concentrated urea is blended with demineralized high purity water in a mixer. The ratio between the streams is carefully controlled to achieve the desired concentration. The process temperature is 25-50 °C (77-122 °F). After mixing, the urea solution AdBlue 32.5 % is cooled and transferred to the storage tanks.

### Instrumentation and installation

Vaisala K-PATENTS® refractometer PR-43-GP is used to measure the concentration of urea solution AdBlue 32.5 % after the heat exchanger before it is transferred to storage tanks.


The refractometer is mounted in the outlet pipe before storage to control the concentration of the urea mix. The concentration is maintained between 31.8 % and 33.2 % b.w. (32.5 % optimum) with automatic adjustment of the demineralized water feed. The lowest freezing point for urea is around -11 °C (12 °F) at the concentration of 32.5 % by weight.

The refractometer provides Ethernet and 4-20 mA output signals for automatic and

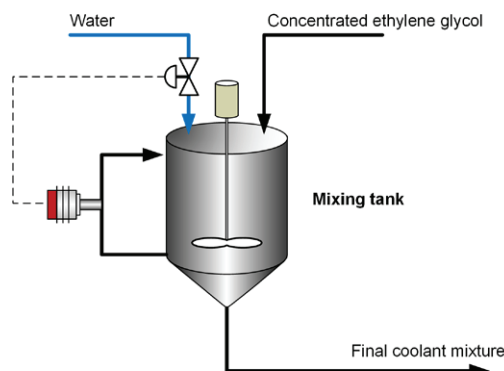
real-time control of the valves of concentrated urea and demineralized water streams. The control provided by the refractometer ensures the optimal concentration is always achieved.

Typical measurement range in this application is 0-40 %.

When two or more measurements are needed, the Multichannel User Interface (MI) provides multiple connectivity with one transmitter (up to 4 refractometers), thus reducing the investment cost.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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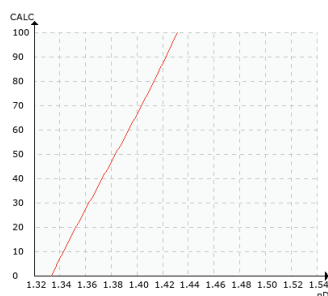


## ETHYLENE GLYCOL $C_2H_6O_2$ , PROPYLENE GLYCOL $C_3H_8O_2$

### Typical end products

Automotive antifreeze, coolant, de-icing, hydraulic brake fluids.

Chemical curve: Ethylene glycol R.I. per Conc. % b.w. at Ref. Temp. of 20 °C



### Introduction

Ethylene glycol is a clear viscous liquid frequently used in many industrial and commercial applications. One of the main uses for ethylene glycol is in the automotive industry as an antifreeze and coolant because it has a much lower freezing point than water. Ethylene glycol in the car's cooling system manages the engine temperature by removing, circulating dissipating and controlling heat.

Apart of being a key ingredient in automotive antifreeze and coolant solutions, ethylene glycol is also a major component of deicing solutions used in a variety of transportation applications, including cars, boats and aircraft, as well as on airport runways during the cold winter months.

### Application

An important feature of ethylene glycol is that it lowers the freezing point of water. For example, while pure water freezes at 0 °C, a typical concentration of automotive antifreeze/coolant mixture of 60% ethylene glycol and 40% water freezes at -45 °C

(-49 °F). The concentration of the mixture should be controlled carefully to keep it to the optimal ethylene glycol content for certain cold temperature in order to ensure the functionality of the engines and avoid damages. For car manufacturers, using coolants with an inappropriate concentration would even result in costly recalls.

The preparation of the coolants is very simple. Ethylene glycol is usually available on site as a concentrated coolant solution which is diluted in a mixing tank with water according to the technical regulations in each country. The recommended water-coolant ratio is specific to the cold weather (Table 1), and it must be followed carefully as it has a direct effect on the efficiency and/or service life of the cooling system and engine:

**Table 1.** Protection temperatures and coolant properties.

Outside temperature protection		Composition (%)		Refractive index at 20 °C (nD)
°C	°F	Ethylene glycol	Water	
-15	5	30	70	1.3625
-24	-11	40	60	1.3728
-45	-49	60	40	1.3931

After mixing, the coolants must be tested for quality (e.g. for freezing point) through different standard procedures such as ASTM D3321-19 and ASTM D1177-17. The final product is sold to the consumer or used directly to fill the engines of vehicles before they leave the factories.

Most conventional heavy-duty coolant or antifreezes use ethylene glycol, however, propylene glycol may also be used.



## Instrumentation and installation

The Vaisala K-PATENTS® process refractometer is the ideal solution for measuring the concentration of glycol-based products, such as antifreeze, coolant, windshield cleaners and deicing fluids. The refractometer is installed in the mixing tank's recirculating line or directly in the tank with a probe type or flush mounted version. The in-line measurement of the refractometer provides continuous measurement of glycol concentration during blending and reduces the need for taking samples for laboratory or handheld refractometers tests, which expose personnel to hazardous chemicals.

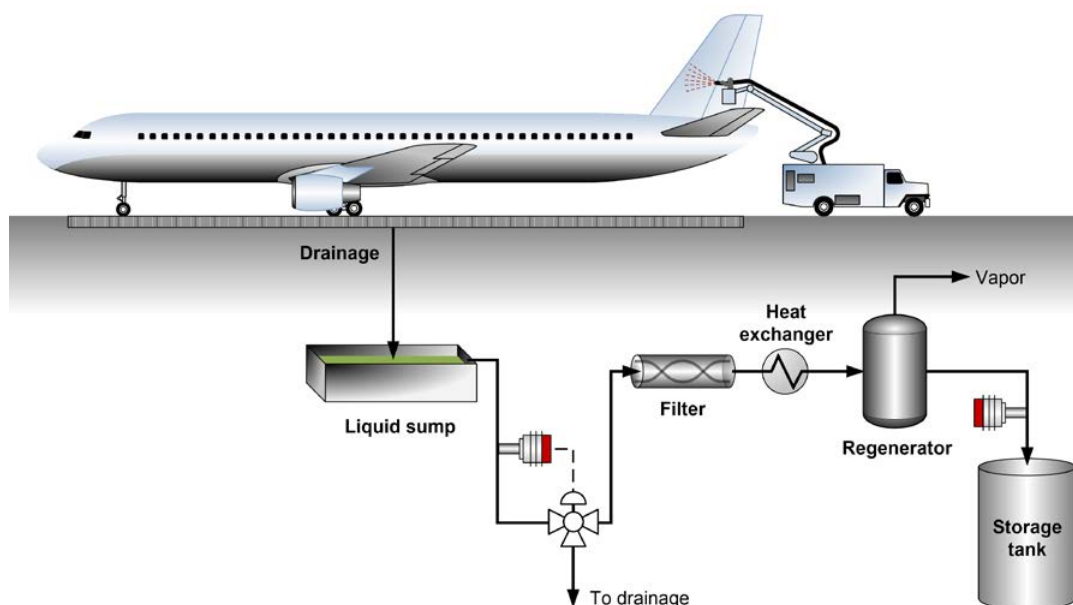
The measurement by the Vaisala K-PATENTS refractometer is not affected by air bubbles created

during mixing, and it can be easily configured to show information about glycol concentration, specific gravity or even freezing point. The refractometer provides Ethernet and mA output signals that can be used for complete automation of the coolant dilution process, creating an efficient and safe process while optimizing raw material use.

The Vaisala K-PATENTS refractometer is delivered factory calibrated and has no measurement drift. This accurate and reliable measurement assures manufacturers of antifreeze/coolants and vehicles a final coolant with the correct mixture ratio of glycol and water, as well as peace of mind of engines fully protected from overheating in hot weather and freezing in extreme cold weather.

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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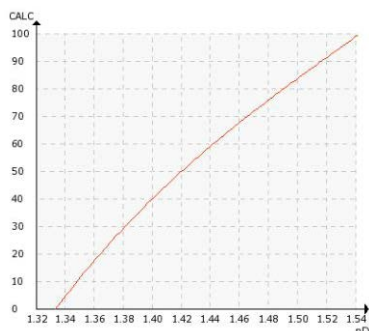


## ETHYLENE GLYCOL $\text{CH}_2\text{OH}_2$ , PROPYLENE GLYCOL $\text{C}_3\text{H}_8\text{O}_2$

### Typical end products

Airplane de-icing.

Chemical curve: R.I. per Brix at Ref. Temp. of 20 °C



### Introduction

Aviation de-icing and anti-icing fluids, such as ethylene glycol (EG) or propylene glycol (PG), keep atmospheric ice from accumulating on an aircraft's flying and control surfaces while in flight. The effects of ice accretion on an aircraft can cause loss of control, resulting in catastrophic flight events.

De-icing on the ground is usually done by spraying the aircraft with a de-icing fluid. The operational procedures are continually checked and updated by an international group of experts under the auspices

of the Society of Automobile Engineers (SAE) G-12 Committee on Aircraft Ground De-icing/Anti-icing.

The de-icing fluids must be used with a containment system to capture the used liquid, preventing ground and streams contamination. Airport storm water discharges containing de-icing fluids are the focus of numerous regulatory actions.

### Application

#### 1. Spraying

De-icing is typically accomplished with the application of a glycol and water mixture via spray nozzles to the aircraft's fuselage and flying surfaces. The de-icing fluids are applied through spray nozzles in the apron area either from fixed application platforms or from mobile tanker trucks. To ensure that the fluid performs effectively, operators must perform quality control checks with regard to the fluid's viscosity and Refractive Index.

The fluid's freezing point, that is -36 °C (-32.8 °F), rises with the addition of water (e.g. through contamination or precipitation), reducing its effectiveness. Therefore, regular checking of the fluids' freezing point is necessary to ensure safe operations. The freezing point can be measured directly using the ASTM D1177

-method. However, this method is cumbersome for field application.

The refractive index value is related to the concentration of ethylene glycol, the freezing point depressant contained in the de-icing fluid. Therefore, refractive index is also related to its freezing point. In the field, glycol's freezing point can effectively and easily be monitored by measuring the fluid's refractive index with Vaisala K-PATENTS® Compact Process Refractometer PR-43-GC. The acceptable refractive index range at 20 °C (68°F) is 30.5–33.5 °Brix. The refractometer is automatically temperature compensated and covers the full refractive index range, which is measured in Brix.

Hydrometers are unsuitable for calculating the freezing point for the aircraft de-icing formulations because they are not sufficiently accurate. Air bubbles are often present in the de-icing/anti-icing fluid. The refractometer is uninfluenced by the presence of bubbles.

## 2. Recovery

After application, the de-icing fluid and water mixture is either disposed through natural rainwater drainage channels, or recycled via centralized de-icing fluid recovery and recycling facility. A drainage system, which is incorporated into the concrete or asphalt apron, collects the de-icing fluid along with melted ice and snow and directs the liquid into a sump.

Upon accumulating a sufficient quantity of the used and diluted de-icing fluid, it is pumped to a sludge tank. Solids and free water are separated by settling and filtration processes in the sludge tank. The filtered de-icing fluid mixture is heated through sequential heat exchangers and directed into a regenerator. The de-icing fluid mixture is leaned with a dehydration process in the regenerator, involving water and water condensate evaporation in a reflux stripping column. Then, it is passed on to an accumulator.


The heated, lean de-icing fluid is pumped from the accumulator back through the sequential heat exchangers and then returned to the storage tank for subsequent reuse.

## Instrumentation and installation

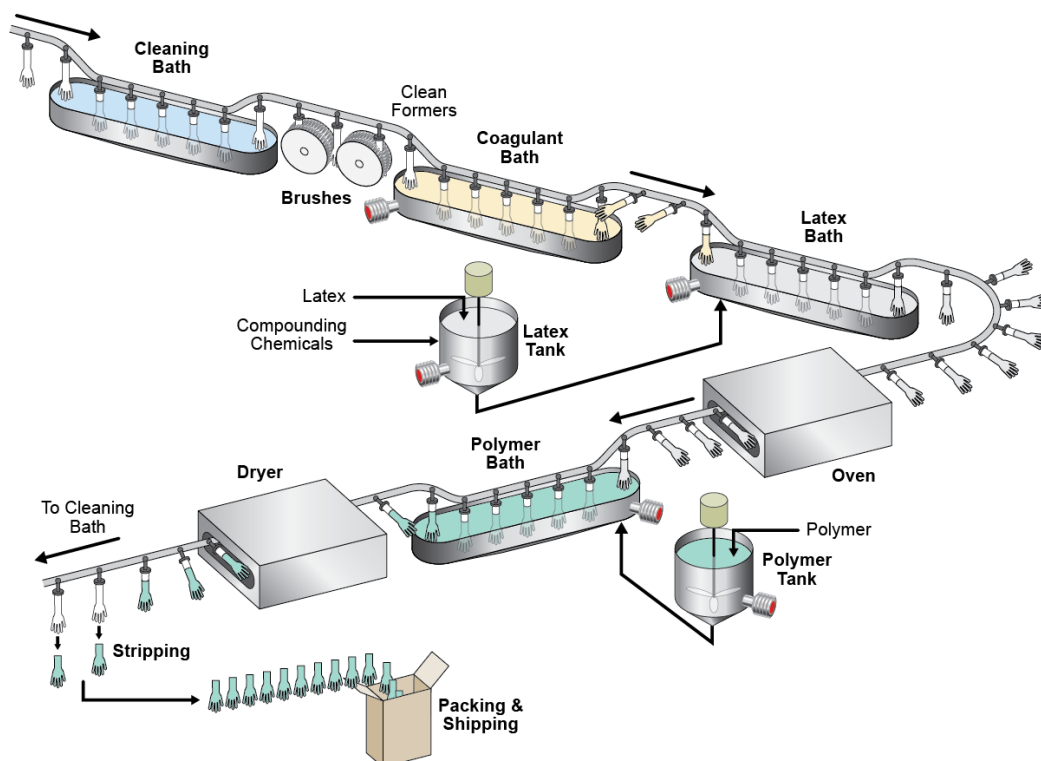
The refractometer is used to measure the concentration of glycol from spraying platforms and mobile units, as a quality and safety check.

Another process control area is in the recovery system. It monitors the concentration of collected fluids in order to direct pure water to drainage and diluted glycol to the sludge tank.

The refractometer also measures the final concentration after the dehydration process before the glycol is returned to storage.

Instrumentation	Description
	Process Refractometer PR-43-GC is a compact refractometer for smaller pipe sizes in general industrial applications. Available in 2 inch and 2.5 inch process connections and via reducing ferrule in 1.5 inch process connection. The refractometer is installed directly in a pipe elbow by an L coupling connection or in a straight pipe via a Wafer flow cell or a Pipe flow cell.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 Brix.



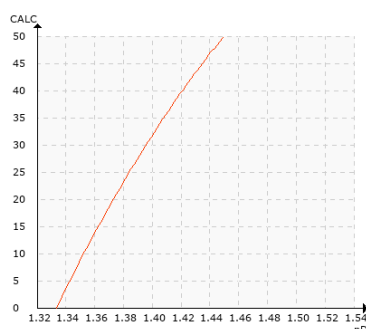


## NATURAL LATEX, NITRILE BUTADIENE RUBBER (NBR), COAGULANT SOLUTIONS, POLYMER SLURRY

### Typical end products

Gloves made of natural or synthetic rubber for different uses in the healthcare, food, chemical, pharmaceutical, automotive finishing and oil and gas industries.

Chemical curve: R.I. vs. Latex % b.w. at Ref. Temp. of 20°C



### Introduction

Rubber gloves are used for protecting skin against physical and chemical injuries as well as against infection by viruses and bacteria. Rubber gloves can be made from natural rubber (*latex*) or from synthetic rubber such as *nitrile butadiene rubber (NBR)* and *chloroprene rubber (CR)*.

Rubber gloves are for different tasks in several industries, ranging from healthcare to food processing and services, automotive to construction and chemical industries.

### Application

#### Natural rubber gloves

Natural rubber gloves are manufactured by a *dipping technology*, where ceramic or metal formers that mimic the shape of a hand are dipped in liquid latex, and then dried. This process has many variations, and production is done in batches or continuously.

The manufacturing process starts with cleaning the formers to remove any residual particle or dirt that could cause defects in the final glove product, and to ensure latex adhesion to the formers during latex dipping. In cleaning, the formers are sunk in acid and alkali baths and are brushed mechanically for thorough cleaning. After that, the formers are rinsed with clean water.

Next, the formers are dipped in a coagulation tank containing a solution of calcium nitrate or calcium carbonate. The coagulant is applied to enhance



polymer flocculation and to ensure the latex is distributed equally on the surface of the formers. The Total Dissolved Solids (TDS) concentration of the coagulation bath is maintained at its optimal level to prevent sedimentation of ingredients, as it could cause weight variation or weak spots on the gloves.

After mild drying, the formers are dipped into the latex dipping tank. This tank contains *compounded mature latex* that has been prepared by mixing latex with various compounding chemicals. The concentration of latex is carefully monitored and controlled as it has a crucial effect on the final quality, durability and product specification. The concentration of latex for dipping should be kept between 10 and 60 %.

A leaching process follows, where the formers are dipped into hot water to dissolve proteins, water-soluble residues and other chemicals from previous steps. Proper leaching of the gloves improves the strength of the latex film but not without replenishment of water to prevent build-up of extracted chemicals.

The next step is vulcanization. In this step, the material is heated at a high temperature to allow the compounding chemicals to form a cross-link with the polymer molecules. Cross-linking gives strength and elasticity to the physical properties of the rubber.

After vulcanization, the surface of the gloves might be treated to facilitate release from the hand former, to prevent gloves adhering to one another, and to ensure the gloves fit smoothly. For instance, the gloves may be dipped in a polymer coating solution for easier donning and to ensure the inner part of the glove is smoother and not sticky. Typical polymer coatings are aqueous dispersions of acrylic or polyurethane. The polymers are diluted and maintained at the required concentration (up to 20 %) to ensure high quality of the glove product.

The last part of the manufacturing process is the stripping phase where the gloves are removed from the formers. The gloves are tested for quality and packed for delivery to the customers.

## Synthetic rubber gloves

The manufacturing process for synthetic rubber gloves is similar to latex production besides that they are manufactured from synthetic materials such as nitrile, vinyl and synthetic polyisoprene.

The main difference is that in this process the synthetic material needs to be created first. The rubber raw material used is commonly a copolymer, a plastic material produced by the copolymerization of two or more different molecules or monomers. In the case

of nitrile butadiene rubber (NBR), the two monomers are butadiene and acrylonitrile. Synthetic rubber is manufactured by emulsion polymerization or polymer emulsification by means of mechanical shearing.

Once the synthetic materials are available, they go to the factory for production. The required processes are relatively the same to those in latex rubber gloves manufacturing, but the dipping line requires different operating parameters.

## Instrumentation and installation


Vaisala K-PATENTS® Process Refractometer PR-43-G is installed at different stages of the rubber gloves line to measure the concentration of dipping baths solutions. The refractometer provides continuous TDS concentration measurement to ensure operation within the specifications and to guarantee high-quality rubber gloves. The in-line measurement by the refractometer eliminates the need for sampling and long laboratory tests, providing immediate data for real-time process control.

The refractometer can be installed directly on a pipe or tank. At the compounding latex, polymer and coagulant preparation steps, the refractometer monitors the desired TDS concentration is consistently used and provides important information to keep it to the pre-determined level. The measurement is also useful for monitoring the concentration of different baths and for adjusting it only when needed, reducing the consumption of raw materials and minimizing operating costs.

If the factory operates from synthetic rubber made at the same facilities, the refractometer can also be used in the polymerization vessel. The refractive index measurement provided by the refractometer helps to follow in real-time the progress of the reaction and to determine the degree of polymerization. The refractometer can be installed in-line in the bypass line of the reactor or directly at the bottom of the vessel through the steam jacket, eliminating the need for sampling and off-line testing (see also Application Note *Control of Polymerization and Nitrile Butadiene Rubber Production process*).

Automatic prism wash with high pressure water is required in this application for concentrations above 40 % by weight or when flow velocity is below 1.5 m/s. Hazardous and intrinsic safety approvals are available also when required.

The reliable in-line measurement by the refractometer helps rubber glove manufacturers to improve product quality, enhance productivity and reduce production costs.

Instrumentation	Description
	Process Refractometer PR-43-GP is a general industrial refractometer for pipes and vessel installations. The PR-43-GP can be installed with 2, 3 and 4 inch flange and 3 inch Sandvik L coupling process connections and a variety of flow cells for pipe sizes of 1 inch and larger.
User Interface	Selectable multichannel MI, compact CI or a web-based WI user interface options allow the user to select the most preferred way to access and use the refractometer measurement and diagnostics data.
Automatic Prism Wash	<b>Prism wash with high pressure water:</b> The components of a high-pressure water system are a sensor with integral water nozzle mounted at the sensor head, a high pressure pump together with a power relay unit and an indicating transmitter equipped with relays.
Measurement range	Refractive Index (nD) 1.3200 – 1.5300, corresponding to 0-100 % by weight.

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