# VAISALA

Advantages of liquid concentration over density measurement in the chemical industry

eBook for process engineers

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### Introduction

The chemical industry is one of the largest global manufacturing sectors. It produces chemicals that are used as intermediates by other manufacturing sectors (such as the pharmaceutical and food industries) and to make products produced by other branches of the chemical industry. Water, air, fossil fuels, minerals, and metals are the typical raw materials used by the chemical industry.

Depending on their final purpose, chemical industry products can be divided into bulk or commodity chemicals, specialty chemicals, and fine chemicals. The manufacturing of products according to predefined specifications ensures that they are of high quality, that raw materials are used efficiently, and that production costs are optimized. Given that production processes routinely involve the handling of hazardous materials, safety is the number one priority.

As an energy-intensive industry, the chemical industry is undergoing a transformation driven by the need to decarbonize and find new ways to save energy during chemical processing.

Substantial energy savings can be achieved through the optimization of common chemical manufacturing processes by taking into use modern process control tools. This eBook is intended for process engineers, technology managers, instrumentation engineers, and quality control and assurance managers. It includes a review of a selection of typical chemical industry processes and an analysis of liquid and density measurements as a means of process control. It concludes with practical examples of the technology used in various chemical applications.

Whenever there is liquid in a process, it must be measured. Liquid concentration often changes during the production process, and these changes have a direct impact on both the final product quality and the overall efficiency and sustainability of the process. This makes liquid concentration measurement a key parameter for controlling and adjusting processes to ensure the desired output.

When choosing a process control method, it is important to take all relevant factors into consideration, including process conditions, installation options, environmental impact, accuracy, and total cost of ownership.

There are many different analytical process instruments for measuring liquid concentration. This eBook focuses on comparing two of the most common methods used in industrial applications: refractive index technology (refractometer) and density technology (Coriolis, ultrasonic, nuclear, and microwave).



Liquid concentration measurement for industrial process control – general aspects to consider

When choosing the right analytical instrumentation for ensuring product quality, process safety, and overall control, it is essential to evaluate a solution's capital cost and overall cost of ownership. This includes engineering, installation, calibration, and maintenance costs. Additional practical aspects to gauge are process conditions that can have an impact on the performance and measurement accuracy of such things as impurities, entrained gas, pressure, temperature, and flow.

#### Accuracy and repeatability

How accurate does the instrument need to be? Does the measurement uncertainty need to be traceable or is excellent repeatability more important for the intended application? In many cases, inline instruments can be field-adjusted against, for example, laboratory reference sample measurements, and so the fact that the inline measurement is repeatable and consistent is more important than standalone accuracy. Learn more from our <u>accuracy statement</u>.

#### Long-term stability

How much measurement drift can be tolerated, and how often is it affordable to recalibrate or maintain the sensor? It may be better to have a measurement technology that is robust and retains its promised accuracy long term rather than opting for one with a superior accuracy specification that doesn't provide trustworthy readings after a certain period of time. The stability of measurement accuracy can be affected by internal measurement drift mechanisms and also by external error sources. For example, corrosion and wear can cause drift in some measurement instruments.



#### Installation options

Is the intention to measure from a pipe or a tank? Is it preferable to install the instrument directly in the main line, or can it be installed on a side stream? Some instruments and measurement techniques are strictly limited to pipe installation or even specific pipe orientations or sizes, whereas others offer more flexible installation options; this may include the possibility to retract the instrument from the pipe without having to interrupt the process.

## Cost of ownership and return on investment

A more accurate and stable measurement instrument may often cost more, but it is important to take into account recalibration and maintenance costs. Moreover, in many industrial measurement applications accurate, stable measurements can provide greater savings by reducing raw material or energy use, eliminating product loss, or increasing throughput or yield.



Installation options for the Vaisala Polaris process refractometer.

Tank bottom flange



Long probe for large pipes and vessels

## What is a process refractometer?

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A process refractometer is based on the principle of refractive index measurement, which is a highly accurate measurement of the dissolved components in a liquid. Inline measurement with a refractometer eliminates the risk of solution contamination associated with manual sampling.

The measurement principle behind a refractometer is critical angle measurement. There are three main components in a refractometer: a light source, a prism, and an image detector. The light source sends light rays at different angles to the prism and process interface. Rays with a steep angle are partly reflected into the image detector and partly refracted by the process. Rays with a low angle are completely reflected into the detector. The angle from which total reflection starts is called the critical angle.

The image detector detects a bright field and a dark field corresponding to partially reflected light and fully reflected light. The position of the borderline between the bright and the dark areas correlates with the critical angle, which is a function of the refractive index – and therefore correlates with the concentration of the solution.



A built-in temperature sensor measures the temperature (T) on the interface of the process liquid. The sensor converts the refractive index (nD) and temperature into concentration units.

The Vaisala Polaris process refractometer can indicate different scales, for example Brix, liquid density, and concentration by weight. The diagnostics program ensures that the measurement is reliable. Because the measurement is based on critical angle measurement it is not influenced by crystals, particles, bubbles, or the color of the liquid, making it an ideal solution for chemical manufacturing needs.

Vaisala Polaris PR53GP probe process refractometer

## A look inside the inline Polaris process refractometer

The inline Polaris PR53GP is a general-purpose probe refractometer for measuring concentrations of various solutions. It can be installed directly in a pipeline or tank and is suitable for production and quality-control applications in the chemical industry.

Outstanding long-term stability provides years of accurate, continuous, fast, and stable concentration measurement directly in the process stream. Inline process refractometers are easy to install and have no moving parts that require regular maintenance.

The Vaisala Polaris PR53GP includes the CORE-optics module, a rigid unit that consists of the main measuring components: the light source, prism, temperature sensor, and image detector. Because the module is mechanically isolated from external forces, measurement is not disturbed by vibrations.

The PR53GP continues the success of the Vaisala K-PATENTS process refractometer series. Based on 40 years of experience and continuous development, the PR53 family is the latest generation of digital process refractometers.

#### Read more about the PR53GP



The CORE-optics module is mechanically isolated from external forces and vibrations and requires no mechanical adjustments.

#### An optical window into the process

Once installed, the Vaisala Polaris process refractometer provides remote access to data and an overview of the process. When paired with a Vaisala Indigo520 transmitter, it includes features such as data storage, a graphical interface, and an analog and digital interface. The Indigo520 transmitter is required when the application or the installation position requires washing to control the process. Changing settings, measurement parameters, or other servicing updates can be done directly from the Indigo520 or through a USB cable using Vaisala software.





Distinctive features of the Vaisala Polaris process refractometer



# What is a density meter?

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Density meters can determine liquid density through different measurement techniques such as Coriolis, ultrasonic, microwave, or nuclear. Each of these has various strengths and weaknesses.

#### **Coriolis meters**

Inline Coriolis meters measure mass flow. The instrument has internal oscillating tubes, configured for example in a U-shape. The tubes cause the liquid flowing through them to create a twisting force due to the Coriolis effect. This force is measured by extremely sensitive sensors in the tubes, and the result is used to calculate the mass flow rate, and optionally, the liquid density.

The tubes can be coated with a protective layer to increase tolerance to corrosive chemicals. An advantage of Coriolis meters is that multiple parameters can be measured by a single instrument, including mass flow rate, density, and volume flow. One of the disadvantages of a Coriolis meter is that it is an indirect measurement of liquid concentration. The density reading is impacted by slurries and bubbles, and the instrument is calibrated for concentration typically only at a nominal temperature.

Another disadvantage is that the force sensors in the instrument are prone to vibrations from external sources such as pumps. The process liquid can coat, clog, or corrode the tube wall, affecting the resonating properties and causing measurement error. Wear of the moving tubes and possible protective coatings also cause drift in the measurement, and the protective coating does not tolerate low pressures. Moreover, the instrument cost for large pipe diameters and double or triple U-tube configurations is relatively expensive.



#### **Ultrasonic meters**

Ultrasonic meters measure the propagation of sound waves in liquid. As the liquid density affects the sound velocity, an ultrasonic meter can be calibrated to indicate the liquid density. The measurement probe can be configured as a metallic fork type and can be coated with a protective layer to increase tolerance to corrosive chemicals.

The denser the liquid, the more the sound waves moving through it attenuate. Therefore, ultrasonic density meters work best at specific ranges, typically in liquids with low density or low dissolved solids content.

While ultrasonic meters are relatively inexpensive and can be installed vertically or horizontally, bubbles and suspended particles attenuate the meter's ultrasonic waves, creating noise and reducing measurement accuracy. Furthermore, the technique is not optimal for high concentrations. Corrosive chemicals or abrasive particles in the process liquid can coat or corrode the probe, causing measurement error.

#### Microwave density meters

Microwave density meters measure the propagation velocity of microwaves in the process medium. The dielectric constant of the liquid determines the propagation velocity. Because the dielectric constant of solids (dissolved or suspended) is significantly different from that of water, the velocity can be used to calculate the liquid density.

While microwave density meters can work well in applications with challenging high-turbidity and high total solids liquids, the equipment is relatively expensive, measurement is disturbed by slurries and bubbles, and as a liquid concentration measurement technique this method has limited sensitivity and accuracy for low concentrations and small changes. Additionally, pipe coatings can cause measurement drift.

#### Nuclear density meters

As with microwave density meters, nuclear density meters utilize the propagation velocity of radiation in the process medium to determine the liquid density. A significant advantage of nuclear density meters is that they can measure without having to penetrate the pipe. However, nuclear equipment is potentially hazardous and thus operating it requires strict safety protocols. Monitoring and disposing of the equipment according to these protocols can be very complex and costly, and the degradation of its nuclear radiation source reduces the instrument's accuracy over time.



## Comparison of liquid concentration and density measurement technologies

Density and refractive index are physical parameters used to measure liquid concentration and composition. Density meters can be affected by air bubbles, particles, impurities, deposits, solids, changes in flow, and turbulence. Temperature changes also affect density meters as they need to achieve thermal equilibrium before the measurement is accurate again.

A density meter measurement is based on the assumption that the volume in the pipe is the same at all times.

In contrast, measurements based on refractive index – such as with the Vaisala Polaris process refractometer – avoid these drawbacks. Air bubbles, particles, impurities, deposits, solids, changes in the flow, and turbulence have no impact on the accuracy of measurement. Likewise, comparative analysis of refractive index (RI)based measuring devices with regard to long-term stability, recalibration, and verification all indicate as-good or better efficiency and performance compared to density meters (see performance analysis table on the next page).



## Comparison of liquid concentration and density measurement technologies

Features	<b>Refractive Index by</b> Vaisala inline process refractometer	Density				
		Coriolis	Microwave	Ultrasonic	Nuclear	
Process medium						
Gas (bubbles) or suspended solids (particles) in liquid	No effect, selective measurement of liquid phase.	Affect twisting Coriolis force of the solution and therefore impacts density reading.	Affect microwave propagation and therefore impacts density reading.	Affect ultrasonic propagation and therefore impacts density reading.	Affect nuclear radiation propagation and therefore impacts density reading.	
Pipe deposits	No effect, high flow velocity provides self-clean effect. Prism wash options available for harsh environments.	Affect the resonance frequency and therefore cause drift. May be plugged with heavy slurries.	Attenuate microwave propagation and therefore cause drift to measurement.	Attenuate ultrasonic propagation and therefore cause drift to measurement.	Attenuate nuclear radiation and therefore cause drift to measurement.	
Color of the liquid	No effect	No effect	No effect	No effect	No effect	
Conductivity of the liquid	No effect	No effect	Affects microwave propagation and therefore causes measurement error.	No effect	No effect	
Process operation						
Flow changes, turbulence	No effect	Sensitive to flow velocity changes.	No effect	May create errors in the measurements.	No effect	
Temperature shocks	Compensation needed. Built-in T measurement compensation.	Compensation needed. Temperature changes cause error due to impact on the resonant frequency of the sensor.	Compensation needed. Temperature and density are inversely proportional.	Compensation needed. Temperature and density are inversely proportional.	Compensation needed. Temperature and density are inversely proportional.	
Pressure shocks	No effect thanks to unique CORE- optics design.	Pressure compensation may be necessary.	May create errors in the measurements.	Pressure compensation may be necessary.	No impact, instrument outside pipe.	
Vibration		Vibrations cause noise to the Coriolis force measurement.	Little or no effect.	Vibration may cause noise to the sound measurement.	Little or no effect.	

Features	<b>Refractive Index by</b> Vaisala inline	Density				
	process refractometer	Coriolis	Microwave	Ultrasonic	Nuclear	
Instrument attributes	Instrument attributes					
Installation	Flexible installation options inline, directly to small or larger pipelines, tanks or vessels.	Limited to inline, and bypasses only in large pipes or tanks.	Directly in the pipeline.	Directly in the pipeline or tank.	Around the pipe. No need to penetrate pipe.	
Maintenance	Maintenance-free	Little maintenance	Little maintenance	Maintenance-free	Maintenance and monitoring required.	
Maximum operating temperature	150 °C	200 °C	100 °C	120 °C	Any	
Operating pressure	Max. 40 bar	Max. 500 bar In Iow pressures/vacuum. Protective coatings may not tolerate vacuum.	Max. 85 bar	Max. 250 bar	Any	
Typical liquid concentration accuracy	±0.1%	± 0.1 0.05%	±0.1%	±0.05%	±1%	
Long-term stability	Excellent. No drift mechanisms that degrade accuracy thanks to unique CORE-optics design.	Poor. Pipe deposits, wear, and other drift mechanisms degrade accuracy over time.	Average. Possible drift due to microwave radiation source and detector, and measurement impacted by pipe deposits.	Average. Possible drift in ultrasonic source and detector, and measurement impacted by pipe deposits.	Poor. Notable drift due to radiation source degradation.	
Size and weight	Robust compact or long probe model ranging from 1.6 to 2.9 kg.	From few kg (U-tube) up to 300 kg models for large pipe sizes.	Starting from 6 kg	Starting from 4 kg	Varies by source, detector, and accessories configuration. From few kg to 50 kg and over.	
Recalibration	Factory calibrated, no need for recalibration.	Required due to poor long-term stability. Frequent re-calibration is costly and time consuming.	Recalibration may be needed if measurement impacted by e.g., pipe deposits, wear, or sensor drift.	Recalibration may be needed if measurement impacted by e.g., pipe deposits, wear, or sensor drift.	Recalibration needed due to source degradation.	
Verification	Easy on-site verification, traceable to international standards according to ISO 9000.	Traditional methods for verification are both time consuming and disruptive.	Difficult to arrange known reference.	One-point verification possible with water.	Verification difficult due to safety reasons.	

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# Turning challenges into opportunities for the chemical industry

The chemical industry deals with various liquids that change in concentration as they are being processed. As the example below shows, accurate measurement of liquid concentration enables significant cost savings.

#### Example revenue optimization calculation for a chemical plant

For a chemical plant producing 2 million tons of NaOH a year, improving the accuracy of its liquid concentration measurement by 0.1% can reduce product wastage by 2000 tons. This adds up to an annual saving of USD 700,000-1,200,000 (2,000 x USD 350-600\*).

\*Price per ton of NaOH based on data from <u>www.made-in-china.com</u>, accessed on March 10, 2022.

Learn about the chlor-alkali process in the application note.



It is important to measure the liquid composition of both the raw materials and the finished goods in order to deliver quality products.

Achieving the right concentration values will ensure consistent production of high-quality product, reducing wastage and increasing revenue.

The inline Vaisala Polaris process refractometer is ideal for chemical processes as it withstands temperatures of up to 200 °C and pressure of up to 40 bar, and enables remote control and adjustment of the process via its remote diagnostics feature. The refractometer does not drift as a result of vibration. Although most applications do not require regular prism cleaning, the Vaisala Polaris process refractometer can include a built-in wash nozzle for applications where chemicals may cause heavy coating of the prism.

Typical chemical industry operations and installations where the Vaisala Polaris process refractometer has been successfully deployed include:

- Reactor, reaction degree, and endpoint determination
- Evaporation
- Dissolving tank or vessel
- Dilution, mixing, or blending
- Solid-liquid extraction
- Absorbers and wet scrubbers
- Ion exchangers
- Distillation

- Interface detection and product identification of product-to-product interfaces in loading/unloading operations
- Quality control

In all of these operations it is vitally important to accurately measure and identify the chemical in question in order to achieve high product quality, increase operational safety, enable cost efficiency, reduce downtime, and ensure raw materials are used efficiently.

Liquid density is typically used to measure changes in liquid concentration. There are various ways to measure density. When comparing measurement solutions, it is important to consider the influence of process or ambient temperature, air bubbles, and impurities on the measurement.

The application cases below illustrate some of the typical processes in the chemical industry where an inline refractometer is an ideal solution for liquid concentration measurement.

The Vaisala Polaris process refractometer is designed to withstand harsh, corrosive environments and is available with components made from special wetted parts materials and with intrinsically safe and hazardous area certification.



## Application case: Manufacturing sulfuric acid by contact process

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Sulfuric acid  $(H_2SO_4)$  and oleum are produced industrially in contact plants from sulfur-containing gases resulting from such processes as sulfur burning, acid regeneration, or metallurgical operations. The process consists of the catalytic oxidation of sulfur dioxide  $(SO_2)$  to  $SO_3$ , and the hydration of  $SO_3$  to  $H_2SO_4$  by absorption in concentrated acid. Depending on the number of absorption steps, the contact plants are classified as being either single or double contact process.

Learn about sulfuric acid production by contact process in the application note.

In the production of oleum, the final product is viscous with a temperature of 80 °C (176 °F) and contains 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.

Ilfuric acid and oleum	Refractometer installation point	Measurement value
oncentration of sulfuric id, (H <sub>2</sub> SO <sub>4</sub> )	At drying tower outlet. At primary and final absorber outlet. After oleum dilution tank. In a control loop	<ul> <li>Monitoring and control of 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.</li> <li>Measuring the concentration of acid as it gets blended or concentrated.</li> </ul>
		• Control of the acid circulation to the towers to ensure operation within the optimal concentration range, and to maximize the absorption.
	Dry SO- can	Image: Production of



*Image: Production of sulfuric acid by contact process* 

## Application case: Nitrile butadiene rubber (NBR) production by polymerization

Nitrile Butadiene Rubber (NBR) is considered to be the keystone for industrial and automotive rubber products, such as synthetic latex. NBR is produced in an emulsion polymerization system. To ensure the required product properties, the polymerization process must be accurately monitored.

The refractometer output signal indicates the degree of polymerization. Each individual polymerization vessel requires the installation of a refractometer to accurately monitor the conversion rate from monomer to polymer.

Learn about NBR production by polymerization in the application note.

Learn how the inline refractometer is utilized to control degree of polymerization in the application note.



Image: Production of NBR by polymerization

#### Application case: Chlor-alkali process

Chlor-alkali is the industrial process for electrolysis of sodium hydroxide solutions. Brine (NaCl in water) electrolysis produces chloride and hydrogen, along with the alkali hydroxide.

Sodium hydroxide is delivered in 30-40% NaOH concentrations to customers, who then further dilute it with water for use at concentrations of 14-15% NaOH.

The Vaisala Polaris process refractometer is used to measure the concentration of brine at influx and outflux, which is usually between 190-320g/l. If measured with a density meter, the tube must be very thin to ensure enough sensitivity for accurate density measurement – but this tube easily gets blocked with the impurities in the brine. While coating of the Vaisala Polaris process refractometer prism can occur, this can be remedied with its built-in nozzle that provides an automatic wash with high pressure water.

In the chlor-alkali process the main product is sodium hydroxide (NaOH). The inline refractometer can also be used to measure the concentration of hydrochloric acid (NaCl), hydrogen chloride (HCl), sulfuric acid and sodium hypochlorite (NaClO).

The Vaisala Polaris process refractometer withstands strong alkali and highly corrosive environments. It is a perfect device for chemically aggressive solutions and ultra-pure fine chemical processes.

Electrolysis of brineInflux and outflux of brine.Measuring concentration of brine.Before the electrolytic cell, and in brine recirculation line.Measuring concentration of: • Hydrochloric acid (NaCl)	kali	
In the product feedline to the dryer.• Hydrogen chloride (HCl)Prior to and after the evaporators.• Sulfuric acid• Sodium hydroxide (NaOH)• Sodium hypochlorite (NaClO)	/sis of brine	n of brine. n of: NaCl) [HCl) NaOH) e (NaClO)



# Application case: Chemical interface and product identification in loading / unloading operations

A common application in the chemical industry involves chemical interface detection and product identification during liquid chemicals custody transfer. When receiving or discharging chemicals from a railcar, ship or truck, it is critical to ensure that the right chemical with the right specifications is stored in the right storage tank.

The inline refractometer identifies the chemicals according to their distinctive Refractive Indices. For example, the figure below illustrates the distinctive refractive indices of hydrocarbons.

The inline process refractometer provides temperature compensated chemical identification with an accuracy of nD + -0.0002 across the full measurement range of nD = 1.3200 - 1.5300 corresponding to 0-100% by weight.

Learn how the Vaisala Polaris process refractometer supports efficient and accurate chemical interface documentation and product identification from the brochure <u>Chemical identification</u> and interface detection.

<u>Read more</u> about how our customers achieve cost savings, reduce product loss, and eliminate workplace safety risks with the help of inline refractometer measurement.





Did you know that by installing the Vaisala Polaris process refractometer for product interface detection you can decrease product waste by up to 30%?

Image: Chemical interface and product identification in loading operations with the Vaisala Polaris process refractometer.

## Application case: Caprolactam $(C_6H_1NO)$ production process

Caprolactam ( $C_6H_{11}NO$ ) is the raw material for Nylon-6 plastics and fiber engineering.

Learn about the caprolactam production process in the application note.

Caprolactam	Refractometer installation point	Measurement value
Aqueous caprolactam solution	After the initial extraction.	To control and maintain high extraction efficiency.
Evaporation process control	In the outlet of the evaporator.	Provides a signal to a controller to regulate the concentration value by varying the inlet flow through the evaporators.



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## Application case: Ammonium nitrate production process

Ammonium nitrate  $(NH_4NO_3)$  is a salt consisting of ions of ammonium and nitrate. It is mostly used in agriculture as a high-nitrogen fertilizer. Ammonium nitrate is produced by reacting nitric acid with ammonia. The resulting solution is concentrated to 97.5-98% in a final concentrator.

Learn about ammonium nitrate production in the application note.

Ammonium nitrate	Refractometer installation point	Measurement value
Ammonium nitrate concentration measurement	On the concentrator outflow. On the slurry tank outflow.	<ul> <li>This measurement is critical to create a uniform prill and to prevent the need for reprocessing.</li> <li>NH<sub>4</sub>NO<sub>3</sub> solution is 90-98%, at a process temperature of 160-180 °C (320-356 °F).</li> <li>NH<sub>4</sub>NO<sub>3</sub> solution is 90-98% and the process temperature is 150-160 °C (302-320°F).</li> </ul>

Solution Final concentrator Additives Water 24 Prill tower Scrubber Filler Cooler Fattening drum ...... Screen Fines recycle Small fines Coatin drum To storage

The Vaisala Polaris process refractometer provides a direct measurement of ammonium nitrate concentration which can be sent to the control room through 4-20 mA output signals. The refractometer's signal warns the operators of changes in the process and allows for real-time adjustments of the process.

> *Image: Ammonium nitrate production optimized with inline refractometer measurement*

## How Vaisala can help with your measurement needs

Vaisala has extensive knowledge on how to optimize chemical production to be more efficient.

Read our application examples or contact us directly to discuss your process and measurement needs.

<u>Contact us</u> to discuss your application and measurement needs.







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