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Process Refractometry –

Everything from Physical Basics to Innovative Solutions for the Food and Pharmaceutical Industries

White Paper

turning science into solutions

Contents

1. Introduction

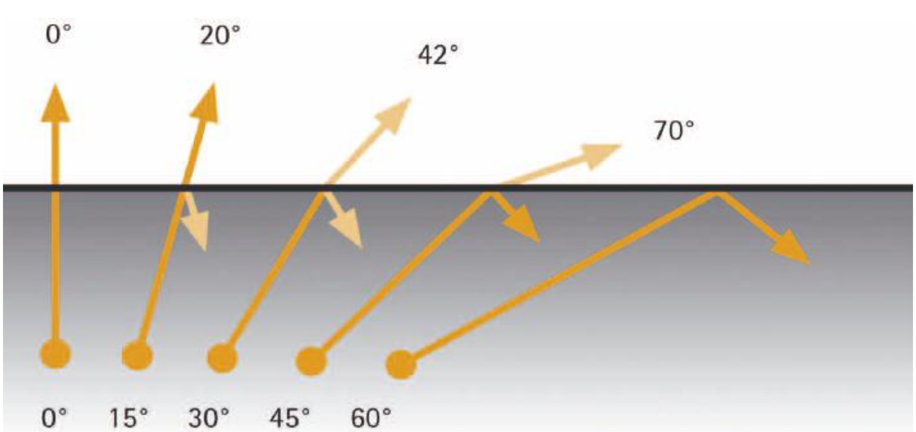
Process measuring instruments are essential tools for monitoring and controlling critical process parameters. Risk analysis identifies critical process parameters which must be monitored and controlled during the production process in order to achieve a specified product quality. Depending on how important a process step is, comprehensive monitoring and control are essential in order to ensure that the process parameters continue to meet specifications.

Inline and online measuring instruments provide real-time data for process control, allowing for high production quality. Furthermore, comprehensive monitoring and control of process parameters and subsequent documentation ensure that legal and commercial criteria are met.

Process refractometers were developed for monitoring and controlling liquid and pasty process flows. They can be used for continuous, extremely accurate, real-time substance identification and determination of critical factors such as the concentration and purity of solutions.

2. The Basic Principle: Optical Refraction and Reflection

Refractometers measure the refractive capabilities of liquid and pasty process flows and use this data to determine the concentration of a dissolved substance. To do so they use the effect of optical refraction, which occurs when an object passes from one medium to another, e.g., from air into water. From a certain angle of incidence the light is reflected where the two media meet instead of refracting:



3. The Physics of Refractometry

The speed of light depends on the medium in which it propagates. In pure water, for example, the speed of light is faster than in water with other dissolved substances. The speed of light in a vacuum, c_0, is the fastest speed at which light can propagate:

$$c_0 = 2{,}99792458 \cdot 10^8 \text{ m/s}$$

Wavelength is defined through the following relationship:

$$\lambda = \frac{c}{\nu}$$

The variable ν is the frequency of light in optics. The speed of light in a medium depends on the wavelength, material, and its temperature. The unitless material constant, refractive number n, or refractive index n_D, is the quotient of the speed of light in a vacuum (c_0) and the speed of light in the medium (c):

$$n = \frac{c_0}{c}$$

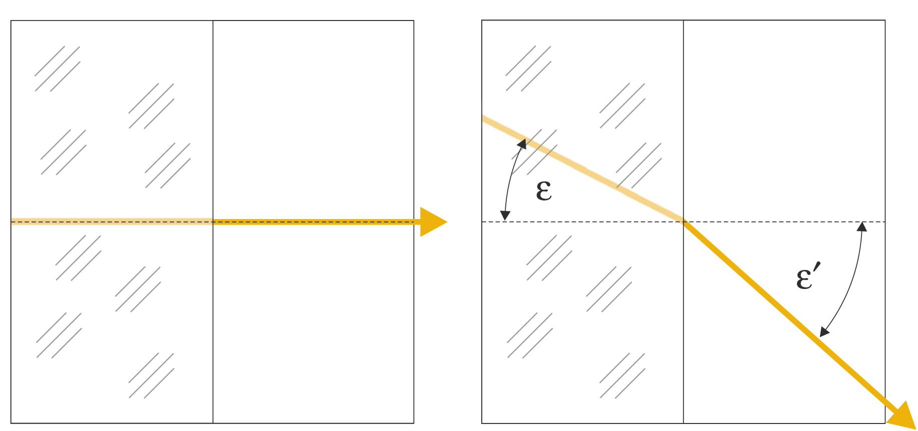
Because c is always less than c_0 , the refractive index can only have values greater than 1. The following table provides an overview of the refractive index measurements of some of the materials which are relevant for subsequent observations.

Refractive index at 20° for λ = 589.3 nm:

Material	Water	Ethanol	Sapphire	YAG	Diamond
Refractive Index	1.33	1.36	1.76	1.82	2.42

A refractive index of 1.33 means that the speed of light in a vacuum is 1.33 times faster than in the medium being examined.

The higher the refractive index, the more optically dense a medium is; thus the lower the refractive index, the less optically dense a medium is. When an object passes from a medium with greater optical density to a medium with less optical density, the light is refracted from the axis of incidence, as shown in the following figure:

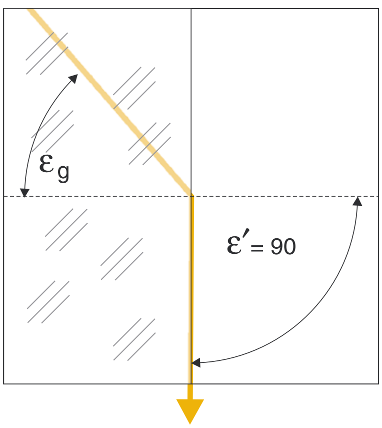


This relationship is expressed mathematically using Snell's law, or the law of refraction:

$$n \cdot \sin \varepsilon = n' \cdot \sin \varepsilon'$$

ε – angle of incidence
ε' – angle of emergence | refraction
n – refractive index of medium in which light is propagating
n' – refractive index of emergence medium

When light propagates below the critical angle of incidence ε_g, an angle of refraction of $\varepsilon' = 90°$ is achieved, meaning that the refracted beam hits the interface plane.



The law of refraction can be simplified under this condition:

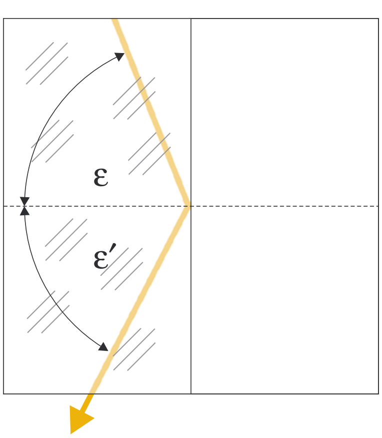
$$\sin \varepsilon_g = \frac{n'}{n} \cdot \sin 90° = \frac{n'}{n}$$

Which results in the critical angle of total reflection:

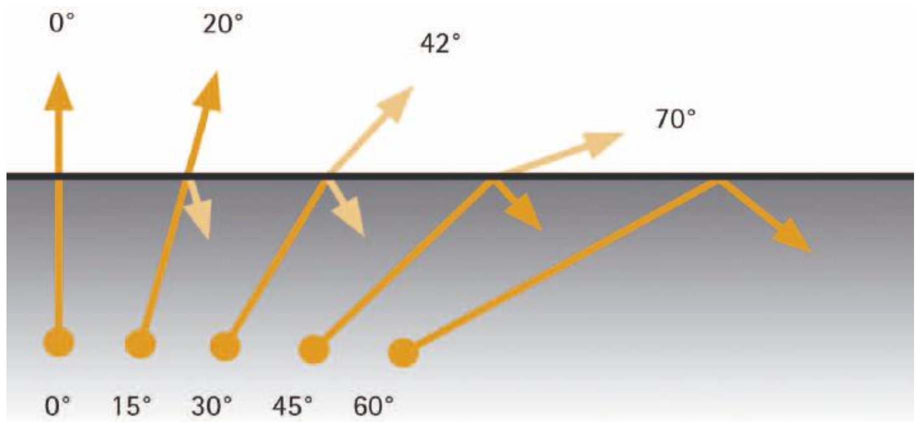
$$!_g = \arcsin \frac{n'}{n}$$

When the angle of incidence $\varepsilon > \varepsilon_g$, the beam of light entering the medium is inevitably totally reflected. The law of reflection, angle of incidence = angle of reflection, applies:

$$\varepsilon = \varepsilon'$$



When light travels from one medium to another, part of the light is refracted, and the other part is reflected depending on the refractive index difference and angle of incidence. With unpolarized light, the proportion of reflected light constantly increases with the angle of incidence up to the critical angle of total reflection. This relationship is shown in the following figure:



4. The Effect of Temperature

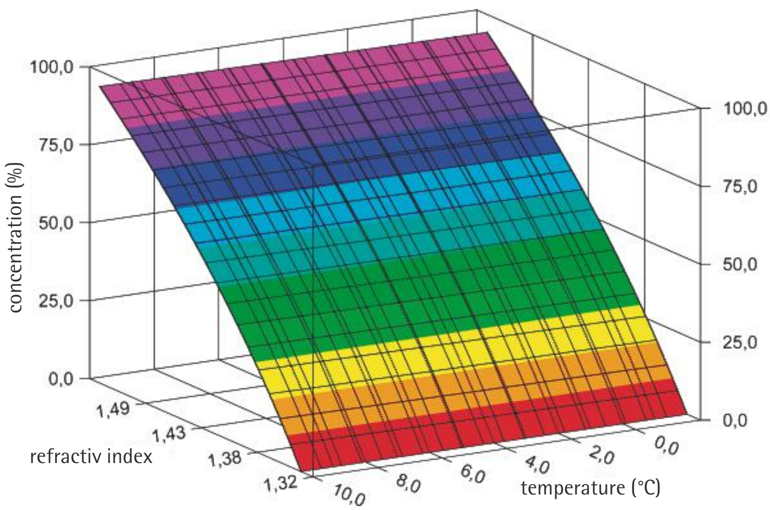
The exact refractive index of a sample depends both on the temperature of the sample and that of the prism.
For this reason, it is very important that the temperature measurement is accurate.
There are essentially three different measurement methods:

- Measurement with temperature sensor in the process flow
- Measurement in the sensor interior
- Measurement in the prism very close to the process flow

Measuring in the process flow or in the sensor interior can only give an estimate of the existing temperature (on the surface of the prism in the process flow) during refractive index measurement. For this reason, measuring the temperature directly in the prism very close to the process flow is the preferred method for temperature compensation.

Due to its dependence on temperature, the refractive index alone cannot directly indicate the concentrations in a solution. Mathematical functions, also known as scales, incorporating both the refractive index and the temperature, can be used to find measured values that are not temperature-dependent. Scales such as the Brix, Oechsle, and Baumé scales, for example, have become standard in the food industry. The Brix scale is most widely used. It was developed for use in determining the concentration of saccharose in pure water. However, it can also be used to measure other dissolved substances. In other words: The Brix value indicates the percentage of saccharose in a mixture of pure water and saccharose (20 Brix is equal to 20% saccharose). The Brix scale can be used for process monitoring and also with mixtures with multiple substances. For example, if the measured Brix value was linked to the results of the reference analysis for optimization of the concentration process, the concentration can be determined during the process using the Brix value and then the process can be monitored and controlled accordingly.

Application-specific, user-defined scales can also be created. To create a user-defined scale, measurements of reference samples with known concentrations at different temperatures are required. If the refractive index is determined using the concentration and temperature, a surface is created:

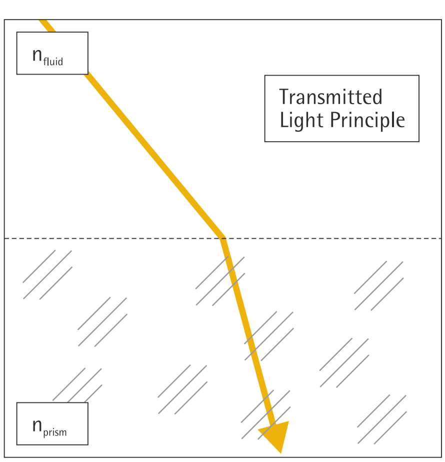


To create a scale, this surface must be adapted using a polynomial. The quality of this polynomial determines the measurement accuracy of the refractometer. A high refractive index resolution is useless if the polynomial is of low quality. To achieve a high degree of measurement accuracy and reliability, it is absolutely essential that the developed user-defined scales are very complex and sophisticated.

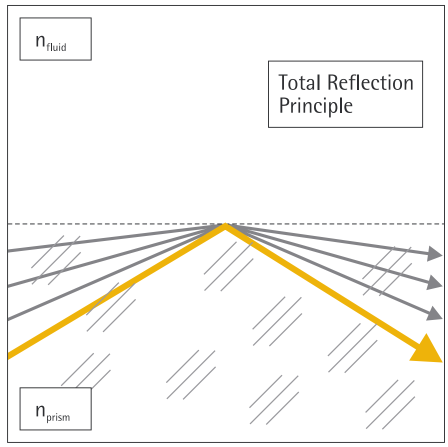
5. Functional Principle of Refractometers

Most refractometers can be assigned to one of two measurement principles: transmitted light and total reflection.

– With transmitted light, the interface plane between both media is illuminated from the direction of the sample with a parallel beam and the change in the direction of the light as it propagates is observed.



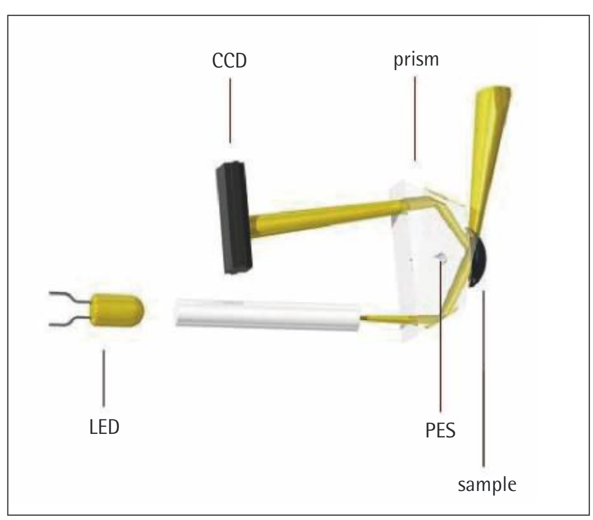
– With total reflection, the sample is illuminated from the direction of the prism using divergent light. The critical angle of total reflection is detected.



Most process refractometers function on the principle of total reflection. Only one prism is needed, used for both illumination and observation. In contrast to the transmitted light principle, the total reflection measurement process can also be used for colored and very cloudy liquids, because absorption by the sample does not affect the measurement. Process refractometers which function on the principle of total reflection are known above all for being easy to integrate into processes and very simple to clean.

6. Process Refractometer Design and Components

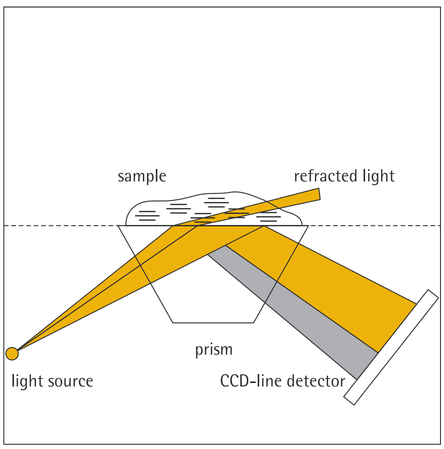
The following figure shows the structure of a process refractometer:



They do not have any moving parts, making them very robust and resistant to defects. To a great extent, the quality of the central components determines the measurement accuracy and reliability of a refractometer

6.1 CCD Detector

A CCD line is used as an optical detector in the refractometer. Light that enters at an angle greater than the critical angle of total reflection is completely reflected and detected on the CCD line. The light–dark transition point changes on the CCD line, depending on the critical angle of total reflection:



If a CCD line is used for detection, the temperature must be precisely measured in order to maintain the accuracy of the refractometer.

6.2 Light Source

One factor the refractive index depends on is the wavelength of the beam of light. Measurements in refractometry are taken at 589 nm as standard. If the interface plane between the sample and prism is illuminated with a wide-band source, the critical angle of total reflection becomes an angle range, thus resulting in an imprecise measurement. In order to achieve high-resolution measurements, the wavelength range must be as small as possible. A narrow-band light source with a wavelength of 589 nm is thus required for refractometer illumination. LEDs are known to be highly efficient with a long service life and are available with a wavelength of 589 nm. However, they have a bandwidth of 20 – 30 nm. Integrating a filter with a bandwidth of 5 nm increases the resolution of the refractometer. Due to the relatively low bandwidth of LEDs, only a small amount of intensity is lost during filtering compared to thermal emitters. An LED requires less power and cooling than a thermal emitter at the same emission strength.

6.3 Prism

Samples are placed on the prism of the refractometer in order to determine the refractive index. The prism must meet criteria in terms of refractive index, dispersion, and resistance.

From the equation for the critical angle of total reflection, it can be taken that the refractive index of the prism must be greater than the refractive index of the sample. According to this requirement, a prism with a high refractive index must be chosen in order to achieve a large measurement range. Sartorius refractometers have integrated YAG (yttrium aluminum garnet) and sapphire prisms which have a higher refractive index than almost any liquid.

In optics, dispersion is the wavelength dependency of the refractive index. Dispersion has almost no effect on Sartorius refractometers, because it concerns single-wavelength devices with a bandwidth of only 5 nm.

The surface of the prism should be scratch-free and chemical-resistant. The surface of the prism must not be damaged during cleaning or by a sample flowing past it during the measurement process. Both YAG and sapphire have a very high degree of hardness: YAG has a hardness of 8.5 and sapphire has a hardness of 9 on the Mohs scale, making them more durable than stainless steel.

7. The Limitations of Refractometry

Refractometry technology is limited in certain ways, which must be taken into account when using a refractometer.

To achieve reliable measured values, the sample being measured must be mixed homogenously, because a heterogeneous sample would result in deviations in the measurements. Furthermore, the refractive index gives the results for an examined medium as a whole. If a mixture contains several substances, the refractive index for the mixture as a whole is measured. Therefore, it is not possible to identify individual substances in a solution and determine their respective proportions without further targeted examination.

From a technical point of view, it should be noted that components in the process flows may cause deposits to build up on the prism. This in turn results in the refractometer measuring the refractive index of the deposit rather than that of the sample. High-quality process refractometers can detect deposit buildup on the prism and then automatically start cleaning the prism using an integrated control unit.

8. Refractometry in the Food and Pharmaceutical Industries

There are a number of applications in the food and pharmaceutical industries in which process refractometers are put to good use.

8.1 Mixing Processes

Errors in the addition or mixing of new substances in a solution can have drastic consequences both on product quality and on production costs. Process refractometers can be used during these processes to check for errors. Linking a reference analysis and measured refractometer values during the optimization of the process allows the production process to be monitored and controlled using process refractometers.

8.2 Concentration Processes

Deviations in raw ingredients are a general problem for products based on natural ingredients. The concentration process for fruit juices involves removing a certain portion of water, which is generally achieved through evaporation. This evaporation process can be effectively controlled using process refractometers. Deviations in the water content of the raw ingredient are automatically detected so that consistent quality can be achieved in the end product.

8.3 Cleaning Inspection

If a process system is used in the production of different products, it is important to ensure that no cross-contamination occurs. Process refractometers can be used to check for residual product traces in real time. In this way, the cleaning process can be optimally monitored and controlled based on purity levels

8.4 Crystallization

Crystallization plays a central role during the purification of solids in the production of many pharmaceuticals. Monitoring and controlling concentration levels during the supersaturation phase is essential for inoculation and the subsequent crystallization process. By measuring in real time, process refractometers help operators optimally control these crystallization processes.

9. Conclusion

Refractometry is an analysis process used to quickly, reliably, and very accurately identify a sample and determine the concentration and purity levels in a sample. Refractometers measure the refractive index and temperature of liquid and pasty process flows and use mathematical functions – scales – to calculate concentration levels. Process refractometers are particularly useful in the food and pharmaceutical industries. Various criteria are set for the quality of the hardware and software of process refractometers in order to ensure that they can be used for the precise monitoring and control of process flows. Only the high quality of all central components ensures a high degree of measurement accuracy and reliability.

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