VAISALA

TECHNICAL NOTE

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Understanding the refractometer measurement performance

Introduction

Measurement accuracy is one of the most important properties of any measurement instrument. Unfortunately, reliable accuracy information is difficult to obtain, as the instrument manufacturers state the accuracy in different ways. The terms accuracy, repeatability, reproducibility, resolution and sensitivity may be neither defined nor correctly used.

This technical note clarifies different aspects of measurement performance of Vaisala's process refractometers making comparisons to other instruments easier.

The actual measurements presented here have been carried out with PR-43 refractometer models.

Terminology

This document uses the generally accepted terminology used in measurement technology. It should be noted that all of the following terms are very often used to signify other meanings, as well.

Measurement uncertainty is a quantitative value that describes the range of measurement points around the true value. It is an umbrella term for all different temporal and spatial uncertainty sources of the measurement, related to either the instrument and the measurement setup.

Accuracy is defined as the difference between the value indicated by the instrument and the absolute correct value of the sample. The accuracy specification for a measurement instrument helps determining the uncertainty of a measurement. A classic way to illustrate accuracy is with a dartboard, such as figures 1 and 2, where the center is considered as the true value.

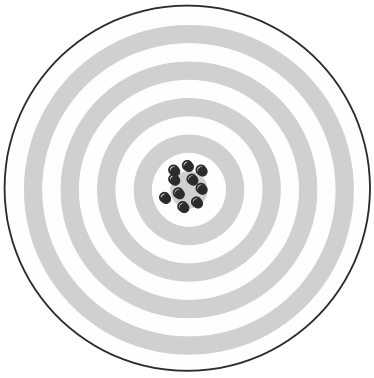

Figure 1. High accuracy – measurements hit close to the true value.

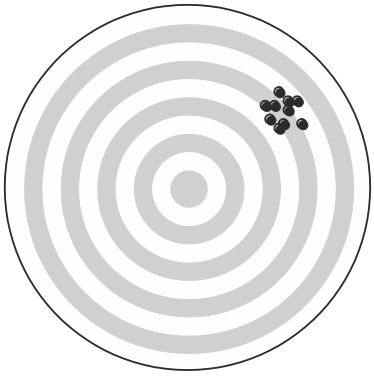

Figure 2. High repeatability but low accuracy – repeated measurements are consistent but far from the true value.

The true accuracy is often very difficult to determine, as the true value is always subject to uncertainty.

The accuracy is limited by systematic (offset) and random (noise) errors.

Repeatability is the variation between instrument readings when the same sample is measured repeatedly within a short period of time in the same conditions.

Reproducibility is the variation between readings when similar samples are measured multiple times with a change in some of the reproducibility factors, such as different instrument, operator, measurement time, place or replacing sample.

In addition to these well-defined concepts, the concepts of *resolution* and *sensitivity* are often used. Unfortunately, it is often difficult to find out how each instrument manufacturer has defined them. The following descriptions give some possible meanings for these terms.

Resolution may mean the smallest change the display shows. For example, a digital display with three decimal places (0.123) has a resolution of ±0.001. This interpretation is not very meaningful with modern digital instruments whose internal resolution is much higher than the actual measurement accuracy or precision. Though the readout of the display could be changed to show several more digits of resolution, such an increase would not make the instrument more accurate, it would simply increase the apparent variation in readings.

Resolution may also be defined as the smallest change in input signal which can be seen. In this case, the resolution depends on the instrument artefacts (noise, drift, etc.). This is the definition of resolution used in measurement technology, but it is very difficult to determine reliably in and thus seldom used in practice.

Sensitivity is sometimes used instead of resolution in the meaning of "smallest detectable change".

Systematic error

There are several non-random error sources in the refractometric concentration measurement (figure 3).

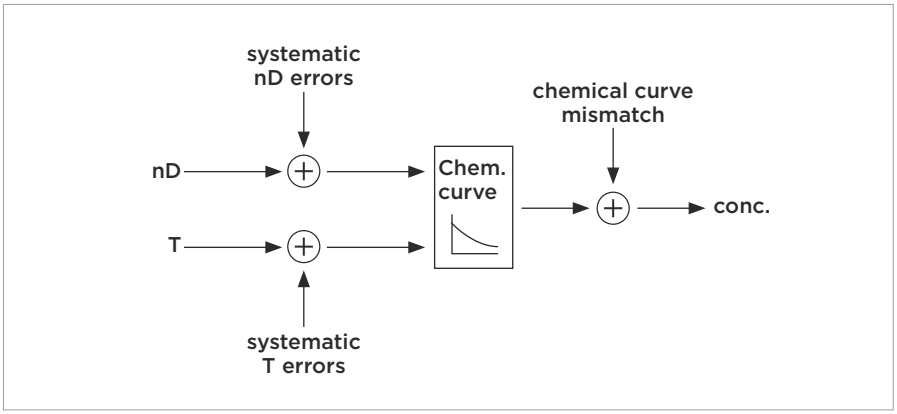

Figure 3. Systematic errors affecting the refractometer concentration reading.

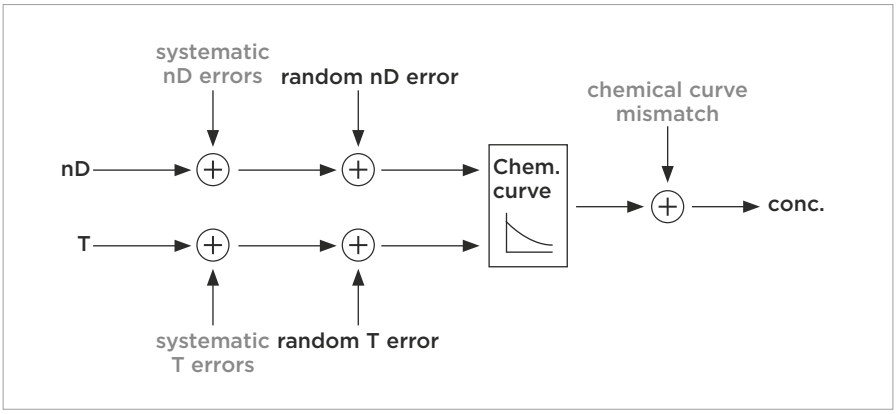

Figure 4. Random error (noise) is present in the refractometer nD and T measurements.

In order to measure concentration, a refractometer has to measure two physical quantities; refractive index (nD) and temperature (T). Based on the measured quantities, a chemical curve calculates the liquid concentration value. All three factors contain error sources for the concentration measurement:

- **Systematic nD measurement errors may arise** due to, for example, uncertainty in the instrument's calibration or thermal effects or defects in the optical system. These errors, however, are reproducible in the sense that if the same instrument is used in the same conditions with the same sample, it will give the same nD value.

- **Systematic temperature measurement errors are** caused by three different factors.

First, the temperature of the temperature measurement element may not be the same as that of the liquid that flows on the prism. This depends on the flow profile and other process-related factors. This error may be up to a few degrees Celsius, and this is usually the dominant temperature measurement error source.

Second, the Pt1000 temperature elements in Vaisala's refractometers have small variation between different units. The maximum error is ±0.15 °C.

Third, the resistance measurement electronics and digital linearization have a combined error of well less than ±0.1 °C.

- **Chemical curve mismatch** produces deviation if the process liquid composition does not exactly match the liquid samples used in the creation of the chemical curve. Moreover, measurements outside the chemical curve's concentration or temperature compensated range may lead to measurement errors. Chemical curve mismatch errors, however, are reproducible and can be corrected by using the field correction or creating a new chemical curve.

As a rough rule of thumb, a temperature measurement error of 1 °C offsets the concentration reading in the most common liquids (brix, black liquor, etc.) by 0.1 % by weight. This number, however, depends heavily on the liquid.

Random error

In addition to systematic error (offset), there is always some random noise present in the measurement system.

The total error of a single measurement is a sum of the systematic and random errors (figure 4).

The different types of noise in a refractometer are:

- **nD measurement noise** originates from the optical noise in the CCD camera image. The magnitude of this noise depends on the process medium (soft optical image produces more noise than a sharp one). In general, the noise is more pronounced close to the lower nD limit of the instrument and smallest in the middle of the range. The actual magnitude of the noise depends on the instrument and optics, but in general the standard deviation for a single measurement is below 0.0001 nD. (See below for ways to improve this.)

- **Temperature measurement noise** comes mainly from the measurement electronics. This noise is negligible compared to the other error sources.

The nD noise in Vaisala's refractometers is wellbehaving in the sense that it can easily be reduced by filtering. For example, a 4-second linear filter approximately halves the noise.

Process-related errors

In practice, the most significant measurement errors are usually process-related.

In some cases the process flow velocity is very low, or the instrument is not in contact with the liquid. If this happens, the process medium near the prism may not be a representative sample. The instrument measures this sample, and thus the measurement result is not reliable.

It is also possible that some material is deposited on the prism (a.k.a. coating or scaling). The instrument starts to measure the nD of this film instead of the nD of the process medium. The onset of this process may be gradual and seen as a drift in the measurement result. Typical drift due to prism coating is in the order of several percents in concentration.

Flow conditions may lead to a situation where the sample on the prism is not well-defined. A poorly mixed flow may not provide a homogenous sample on the prism. Moreover, a too high flow rate may lead to turbulent flow and cavitation. In both cases, the measurement result is unreliable.

It should be noted that process-related errors are often an order of magnitude worse than instrument errors.

Dynamic behavior and response time

The instrument carries out two separate measurements; nD and temperature. These measurements have different dynamic behavior.

The nD measurement is carried out once per second. Due to processing and communication delays the response time to a step change in nD is from 200 to 1200 ms. The nD measurement does not have any other time lag, and thus it is not meaningful to define any half-time or time constant for the measurement.

The temperature measurement is carried out several times per second. Its time constant is dictated by the thermal time constant of the instrument. The time constant depends on the instrument model and process conditions, but the half-time is approximately 6 seconds (time constant 9 seconds, 90 % step change in 20 seconds).

Most of the time the instrument is faster than the process and the dynamic behavior does not introduce any significant measurement error. However, if there are fast step changes in the process, the different time constants may become visible.

Figure 5 shows the (rare) case where the concentration is constant but the temperature changes stepwise 10 degrees Celsius. It can be seen that the indicated concentration jumps slightly for some time as the temperature compensation does not receive accurate temperature information.

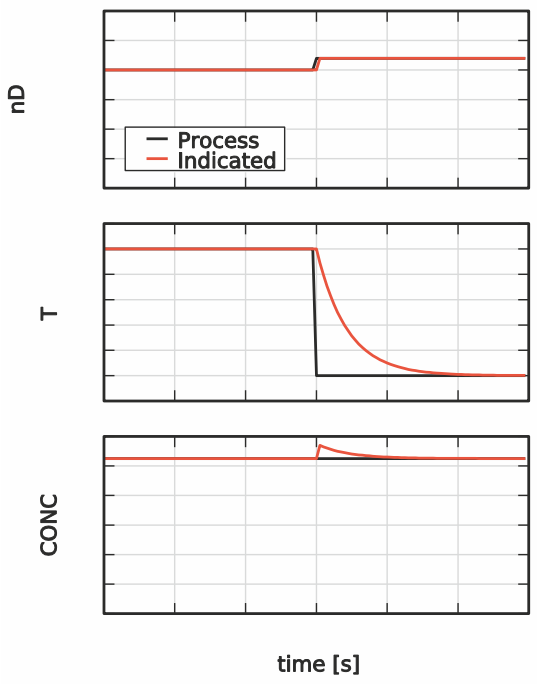

Figure 5. Step change in temperature (no change in concentration)

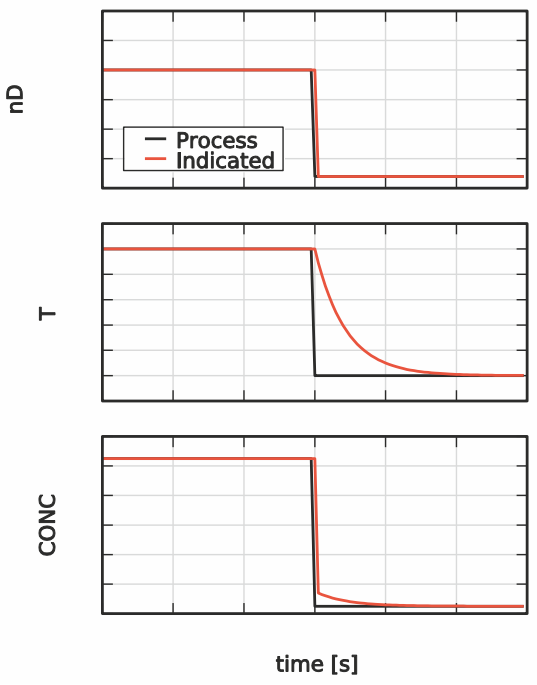

Figure 6. Simultaneous step change in concentration and temperature

Figure 6 shows a slightly more likely situation where the temperature change is accompanied by a concentration change of 10 %. It can be seen that the slower temperature response introduces an error of approximately 1 % for a few seconds after the step change, but the error is only a small part of the step change.

It should be emphasized that this phenomenon is only visible in some interface detection applications. As a rule of thumb, if the rate of change of temperature is less than several degrees Celsius per minute, this effect is negligible.

Filtering

The amount of noise in the measurement result can be reduced by using filtering (damping). This happens at the expense of increasing the response time. In most processes, the process is very slow compared to the instrument, so that a moderate amount of damping does not deteriorate the measurement accuracy.

In Vaisala's refractometers the filtering is applied to the output value (concentration in most cases). There are two different filtering methods: exponential and linear. The exponential filtering is the most common damping method used in the industry. Linear filtering is a moving average of the output signal, and it can only be practically made with digital signal processing.

Figure 7 shows the response of the two filtering methods. It can be seen that the exponential filter has an infinite response time (the

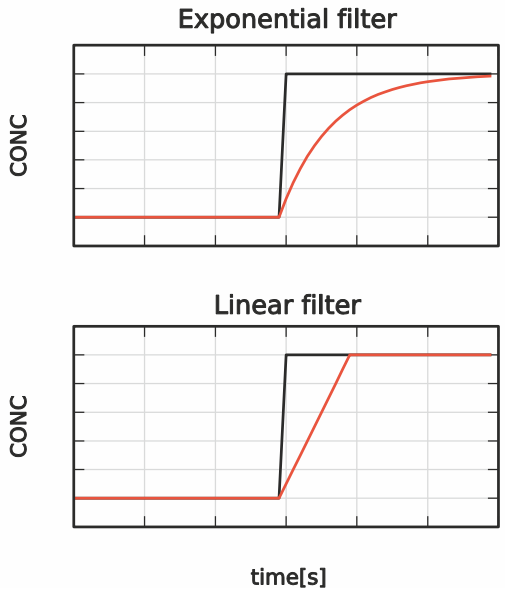

Figure 7. Step change after filtering with a 5-second half-time exponential and a 10-second linear filter.

output value never reaches the input), whereas the linear filter reaches the input very soon. The filter lengths are chosen so that both give approximately the same noise reduction.

With linear filtering, the filter length defines the averaging time. If the filter length is 20 seconds, a step change will take this time. With exponential filtering, the filter time is the half-time of a step change. A 10-second exponential filter needs 10 seconds to change the output to 50 % of the input step.

Exponential filtering, however, provides a smoother result that behaves better with especially the derivative term of PID controllers.

Increasing the filter length decreases the random noise. However, the systematic error sources usually set their limits so that after a certain point increasing the filter length will not remove any noise. There are no hard limits, but in most cases filters longer than 30 seconds are not very useful.

Repeatability test results

In order to determine the short-term stability of the instrument, a series of tests were run with several PR-43 refractometer units, with a specified accuracy of 0.0002 nD.

One of the tests ran a sugar solution in an increasing temperature for several hours. The increase in temperature translated into a decrease in nD, so that a slow gradual nD change could be seen. In the test the change was approximately 3 % of the full measurement range.

The test solution was chosen so that its nD is close to water where the noise performance is at its worst. Higher nD values give less noise.

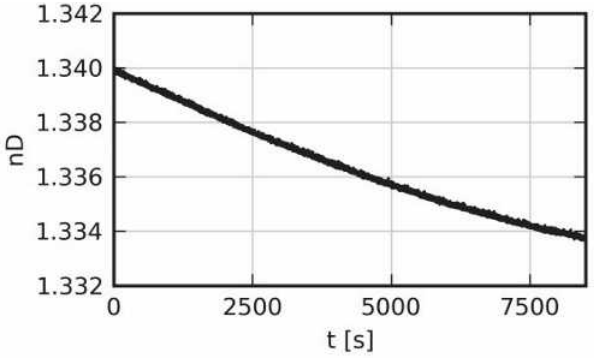

Figure 8. Unfiltered nD measurement points with a slowly rising temperature

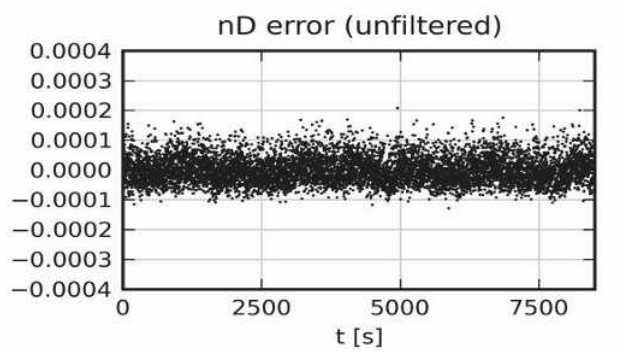

Figure 9. Unfiltered nD measurement error with the effect of temperature removed.

The nD data for a test described above is shown in figure 8. All nD data points measured by the instrument are shown without any filtering.

In order to see the noise better, the effect of the temperature change is removed in figure 9, i.e. only the noise remains.

In this unfiltered data, vast majority of points are within 0.0001 nD (corresponding to 0.05 Bx) of the average. The standard deviation of the noise is approximately 0.000045 nD (corresponding to 0.025 Bx). Often the repeatability of an instrument is defined by the “two-sigma” value which is in this case 0.00009 nD (less than 0.05 Bx).

If the fast response time is not required, filtering improves the situation as shown in figure 10 where a 10-second linear filter is applied to the same data.

The remaining standard deviation of the noise is approximately 0.000017 nD (less than 0.01 Bx) after filtering. However, the data starts to show that while most of the noise is gone, there are some slower changes in the data.

This phenomenon can be shown better by increasing the filter length to 30 seconds as in figure 11.

The standard deviation with a 30-second filter is 0.000013 nD. The improvement over the 10-second filter is fairly modest, as the slow changes start to dominate.

The performance of the instrument with a 10-second filter can be seen in figure 12 which shows the last ten minutes of the run.

It can be seen that changes at the level of 0.00005 nD can be distinguished from the noise.

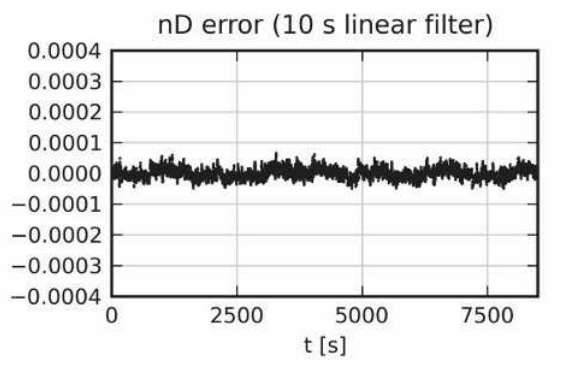

Figure 10. nD measurement noise with 10-second linear filtering

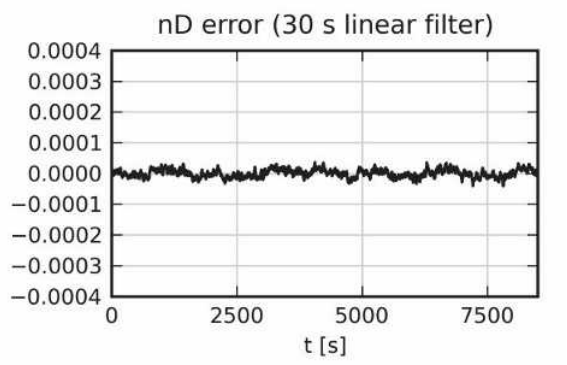

Figure 11. nD measurement noise with 30-second linear filtering

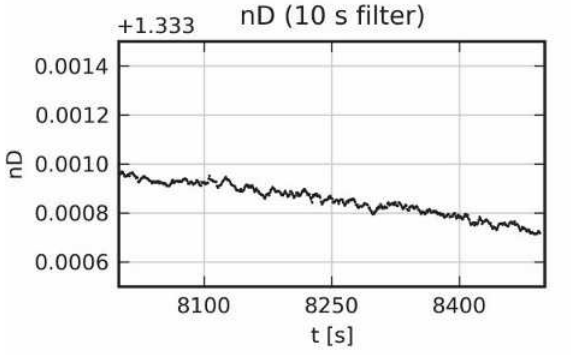

Figure 12. nD measurement with 10-second linear filtering

Conclusions

While the accuracy specification of the PR-43 refractometers is ±0.0002 nD, the practical measurement performance in favourable conditions is often better. The measurement bias type errors can be mitigated using the field correction or creating a new chemical curve, and noise can be reduced with filtering.

The measurement results show that the refractometer's repeatability may be even an order of magnitude better than the accuracy. This is important to acknowledge in process control applications where observing the trend and changes reliably is of more importance than the absolute accuracy in the chemical concentration.

In general, many uncertainty sources related to the measurement conditions, external from the refractometer, can play a major role in the total uncertainty of measurements. The following factors should be taken in account:

- Constant process temperature (or only slow temperature changes in a narrow temperature range)
- Narrow concentration range
- Homogeneous and clear process liquid
- Sufficient flow conditions for prism self-clean effect
- Slow nD changes in the process (allowing for filtering)

If these conditions are met, the measurement is very likely to perform much better than the stated accuracy would predict. Many measurement uncertainty issues are not a result of the refractometer nD accuracy or repeatability performance, but come from external factors such as the process conditions.