K-Patents Refractometer Accuracy Statement

K-Patents PR-43 refractometer

| Accuracy: | nD ±0.0002 (corresponds to 0.1 Bx, typical 0.1 wt%) |
|-----------|------------------------------------------------------|
| Repeatability: | nD ±0.00004 (corresponds to ±0.02 Bx, typical 0.02 wt%) |

The stated accuracy is provided across the full measurement range of nD 1.32 – 1.53 (corresponds to 0–100 Bx, typical 0–100 wt%).

High accuracy version -HAC
in the range of 0–30 Brix and 4–30°C:
±0.05 Brix or % by weight.
±0.02 Brix or % by weight (in set-point applications).

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 are neither defined nor correctly used.

This technical note attempts to clarify the situation by giving a brief introduction into the different aspects of measurement accuracy of K-Patents refractometers. The information given in this document should make comparisons between 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.

Accuracy is defined as the difference between the value indicated by the instrument and the absolute correct value of the sample. The true accuracy is often very difficult to determine, as the true value is not known to high enough accuracy.

The accuracy is limited by random variation (noise) and repeatable non-random variation (bias).

Figure 1 illustrates high accuracy.

Figure 1. High accuracy

Precision is limited by the random variation of measurement results. An instrument may have high precision but still display results wide off the mark, as bias does not deteriorate the precision. Precision is often stated in terms of repeatability and reproducibility (see below).

Figure 2 illustrates high precision (but poor accuracy).

Figure 2. High precision (low accuracy)

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 with similar samples when the readings are taken a long time (weeks, months, years) apart possibly with different environment conditions.

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”.

The scientific definition of “sensitivity” is related to binary measurements which give a “yes” or “no” as an answer and is not applicable to measurements giving a number as the result.

MEASUREMENT BIAS

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

- **nD measurement bias** may arise due to uncertainty in instrument calibration, thermal effects in the optical system, etc. In the PR-43 product line these errors are within ±0.00004 nD. These errors 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.
- **Temperature measurement bias** is caused by three different factors.
  
  First, the temperature of the temperature measurement element may not be the same as that of the flow on the prism. This depends on the flow profile and other process-related factors. This error may be up to a few centigrades, and this is usually the dominant temperature measurement error source.

  Second, the Pt1000 temperature elements have small variation between different units. The maximum error is ±0.15 centigrades.

  Third, the resistance measurement electronics and digital linearization have a combined error of well less than ±0.1 centigrades.
- **Chemical curve mismatch** produces some bias if the process liquid is not exactly the same as that used when creating the chemical curve. The chemical curve may also have some error, if the temperature and/or concentration range is outside of the range used in creating the chemical curve. This error is completely 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 centigrade offsets the concentration reading in the most common liquids (brix, black liquor, etc.) by 0.1 %. This number, however, depends heavily on the application.

MEASUREMENT NOISE

In addition to reproducible errors (bias), there is always some random noise present in the measurement system.

The total error of a single measurement is a sum of the non-random measurement errors and the instrument noise (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 K-Patents instruments is well-behaving 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 is very low, or the instrument is not in the flow. 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. Typically, the drift is in the order of several percents in concentration.

If the instrument is installed in a poorly mixed flow, the sample on the prism is not well-defined. The reading may be stable in a stable flow, or it may start fluctuating in a more turbulent flow. 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. Step change in temperature (no change in concentration)

Figure 6. Simultaneous step change in concentration and temperature

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 K-Patents instruments 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 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.
EXPERIMENTAL FILTERING EFFECTS

A step change after filtering with a 5-second half-time exponential and a 10-second linear filter.

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.

In general, the linear filter is better in removing noise. It can be shown to remove most random noise with least effect on the response time. The exponential filter option is available mostly because some users are more accustomed to it.

Increasing the filter length decreases the random noise. However, the non-random 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 optical repeatability and noise performance of the instrument, a series of tests are run with several units.

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

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

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.

CONCLUSIONS

While the accuracy of the instrument is stated to be ±0.00004 nD (corresponding to 0.02 Bx), it can perform much better if the conditions are favourable. As can be seen from the test measurement results, the repeatability may be even an order of magnitude better than the accuracy.

In general, the following factors contribute to high accuracy:

- Constant process temperature (or only slow temperature changes in a narrow temperature range)
- Narrow concentration range
- Clear (non-emulsion, well-mixed) process liquid
- Stable flow
- 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.

It should also be kept in mind that most measurement accuracy problems experienced by users are not related to instrument accuracy but are related to the process and instrument installation.