

# Improvement practices in process industry—the link between process control, variography and measurement system analysis

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In modern process industry it is necessary to find “smart” ways to continuously satisfy customers’ demands, strive for more efficient production and prioritise the focus on safety, health and environment, allowing dedicated companies to obtain a competitive advantage. To achieve individual goals in these areas, companies increasingly implement a so-called business system approach, which ensures systematic, continuous improvement initiatives in all sectors. Most business systems address improvement in process control, since in many cases there is a significant positive economic potential which often only requires minor capital investments. This is also the case for Glencore Nikkelverk, Norway, where it has been decided to transform a former project-based improvement framework into a holistic business system, the Nikkelverk Business System (NBS). One pillar consists of different tools to improve process performance, with the objective to identify critical processes and process stages a.o. and also to ensure that the measurement systems in use can be geared, and validated, to mainly capture process variations, allowing to document valid process improvements. At Nikkelverk there are between 6000 and 7000 measurement points, of which one-third are used for process regulation purposes. It is therefore critically important to be able to monitor and verify that measurement system uncertainty is suitably low—ideally at a level of 10% relative or below compared to the variation of the process being monitored. This will become a key priority. An optimal measurement system should therefore not contribute significantly to the total apparent process variation as revealed by raw process data that are used to track when a process is deviating from steady state or dangerously close to control limits, or gets outside relevant safety limits. Critical processes must be kept under constant monitoring and control to ensure that all improvement attempts work from a solid database. As but two examples, energy and material consumption, can be fine-tuned to a lowest possible level *only* when both processes themselves and the measurement systems are monitored and controlled properly. This not only contributes to the cost efficiency of the company, but enforces process monitoring and control to take a leading role. We here focus specifically on the relative merits of variographic analysis (Theory of Sampling) and measurement system analysis (Six Sigma) and show how the former can function as a very effective screening for the much more costly latter.

## Historical background

Since the 1930s, processes have mainly been monitored by control charting, starting out with the well-known Shewhart chart, a concept of quality control in manufacturing which was first advanced by Walter Shewhart.<sup>1–4</sup> Many graphical methods have been developed under the umbrella of “statistical process control” (SPC), for example the Quesenberry chart, Moving Range chart, EMWA chart etc., which all have the purpose to make room for the voice of the process. But with such a wide range of possible methods, one can at times be bewildered instead of informed. It is quite a task to find suitable (and simple) tools that give you good enough results to work on for continuous improvement of key processes.

It should not, however, be necessary to be an expert in statistics to be able to improve an industrial process. Tools and methods used must be relatively easy to grasp for process technicians, enabling involvement of as many stakeholders as

possible to create a common understanding of the current process state. Hence the best analysis is the one that provides the greatest insight with the simplest approach. But this organisational challenge will not be further discussed in this paper, which instead focuses on two salient technical issues, measurement system analysis (MSA) and variographic process characterisation.

## Introduction

Glencore Nikkelverk is situated on the sea-side of the city of Kristiansand at the south tip of Norway. Since 1910, nickel and other metals have been refined, produced and exported from the production plant in Kristiansand. Nikkelverk’s history ties in with the development of industrialisation in Norway at the beginning of the 1900s. Today, Nikkelverk produces 92,000t of nickel, 39,000t of copper, 4700t of cobalt and 115,000t of sulphuric acid per year, and is one of the most efficient nickel refineries in the world. In the process industry, stabilisation and control of the process is

a key factor to continue production as a sustainable company.

Proper monitoring and control of the measurement system also play important roles for all industrial process performance. Therefore, the magnitude of the contribution to the total observable process variation stemming from the measurement system itself has to be known in order for it to be compensated for when trying to perform the most efficient process control. This can be determined by conducting a measurement system analysis (MSA) in which one splits the measurement system into suitable compartments and determines their individual specific variation by involving several operators to repeat the same procedure several times over. The variation stemming from the measurement system should be kept to a minimum in order to be able to achieve production under stable conditions.

Measurement system analysis is a vital part of the Six Sigma philosophy, which was introduced at Nikkelverk in the first years of the current millennium. Experiences from

this period were that the MSA process was rather cumbersome and burdened the operators with significant additional workloads. Application of the knowledge obtained by a MSA study was also sometimes questionable. It was estimated that the time spent for one MSA could range between one-half and three days, excluding reporting. Approximately 2000 measurement points/systems are currently used in process regulation at Nikkelverk; application to all would therefore lead to a resource expenditure of several years purely for MSA fieldwork. This does not seem a feasible work load as seen from a management point of view and it is certainly even harder to sell this level of many MSA studies to the personnel involved. Even if we only focus on the critical processes identified, the number of MSA studies necessary would be overwhelming. Something had to give—enter variographic characterisation. Notably also, apart from being a very time-consuming task, the result from such studies only reveal the variation of the measurement system at the moment of the test. Concluding that the measurement system is not a significant contributor to the process variation based on single MSA studies therefore runs risks with respect to generalisation over long production periods.

What is the objective of a MSA study? The goal is to record process facts in a database with which to evaluate the adequacy of a particular working measurement system in relation to getting access to the

reliable information regarding the process variability proper.

What if one can in fact do this quality check for each measurement system only, or mainly, by using online data and/or the extensive historical process database?

What if one could initially classify measurement systems as capable, conditionally capable or capable, compared to the process variation only involving stored *online data*?

What if this type of control on the measurement systems could be performed automatically, on *routine* on-line process data?

Such an approach has in fact existed for quite a while, as used within *geostatistics* and within the *Theory of Sampling* (TOS) regimen; an approach called *variography*. At Nikkelverk we see variography as a skilled detective for both processes measurement and process characterisation. The following sections will outline and explain *why*.

### Variography vs MSA

The first issue on the agenda is to clarify the difference between variographics and a MSA study in order to describe how they may complement each other in relation to improving industrial processes.

### The variogram—absolute and relative

Figure 1 shows a simple example of an increasing variogram and how the lag distance affects the resolution of observable

process variability. To develop a variogram one needs a time series with equidistant sampling. By calculating the average of the variances characterising *pairs of observations* spaced by different *lags* (distance between samples) a variogram is obtained with relative ease. Several literature resources exist which explain how to do this and how to interpret variograms,<sup>5-8</sup> making it unnecessary to repeat this here.

The master formula for an *absolute variogram* based on absolute concentration values is:

$$V_a(j) = \frac{1}{2(N_u - j) \cdot a_L^2} \sum_m (a_{m+j} - a_m)^2 \quad (1)$$

where  $(N_u - j)$  is the number of sample pairs and  $N_u$  the total number of datapoints;  $j$  is the contemporary *lag* distance;  $a_m$  is the concentration value at position  $m$  in the time series and  $a_{m+j}$  is the value of the datapoint at position  $(m + j)$ . The squared estimated average  $a_L$  from all data is part of the denominator in order to scale the variance measure for lag  $j$ , so that  $V(j)$  becomes a unitless variance estimate.

The formula for the relative variogram based on *heterogeneity contributions* at each sampling location in the measurement series,  $h_j$ , is shown in equation 2:

$$V_r(j) = \frac{1}{2(N_u - j)} \sum_m (h_{m+j} - h_m)^2 \quad (2)$$

The absolute variogram,  $V_a(j)$ , is convenient when comparing the information with actual measurements and specification

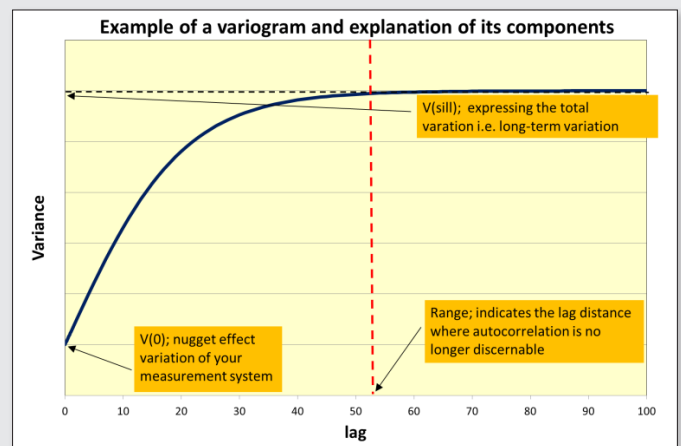
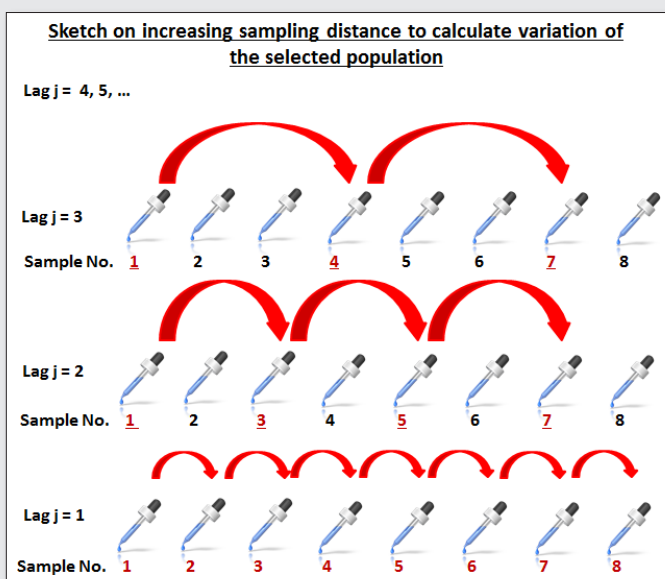


Figure 1. Example of an increasing variogram (right), illustrating the nugget effect,  $V(0)$ , the range and the  $V(sill)$  as well as how the contemporary lag distance reflect the sampling resolution employed.<sup>5-8</sup>

limits, while the relative variogram,  $V_r(j)$ , is more suitable when comparing process information from one variogram with another; for example, from two (or more) measurement locations/measurement systems or from two variograms at different process times.

The variogram example in Figure 1 shows  $V(0)$ , the *nugget effect*, which expresses the total error variance of the measurement system (containing contributions from all incorrect sampling errors, all correct sampling errors as well as the total analytical error).<sup>5-8</sup> After a certain lag distance (about lag 50 in the example presented), termed the *range*, the variogram approaches the total variation of the process,  $V(\text{sill})$ , also called the long-term variation. This level is called the variogram *sill*.

There are further sources of variation, e.g. process cycles and/or linear trends, which can be deduced (as well as their proportion of the total variances) and quantitatively estimated from a full variographic analysis. These variance source effects are easily incorporated in the subject matter described here. However, these variations are not the focus of this article and will not be addressed further here; see Reference 8 for details.

### Measurement system analysis (MSA)

The purpose of a measurement system analysis (MSA) is to evaluate and verify the measurement system by quantifying its accuracy, precision and stability.<sup>9</sup> Thereby one is able to check if the variation of the measurement system is “small” compared to the process variations themselves. Depending on the industry sector and the specific process involved, an often stated rule-of-thumb is that the ratio between measurement system variation and process variation will be lower, or between 1/10 and 3/10.

If one wants to control process variation using a particular measurement system as a basis for decision making, process data should of course not be overshadowed by variations stemming from the measurement system. A MSA study is a vital part of a Six Sigma project. There are several approaches on how to conduct a MSA. For a study on variations regarding a sampling system which delivers samples to a laboratory for analysis, the steps sketched in Figure 2 can, for example, be the sampling procedure (step 1), the sampling reduction (step 2) and the sample preparation

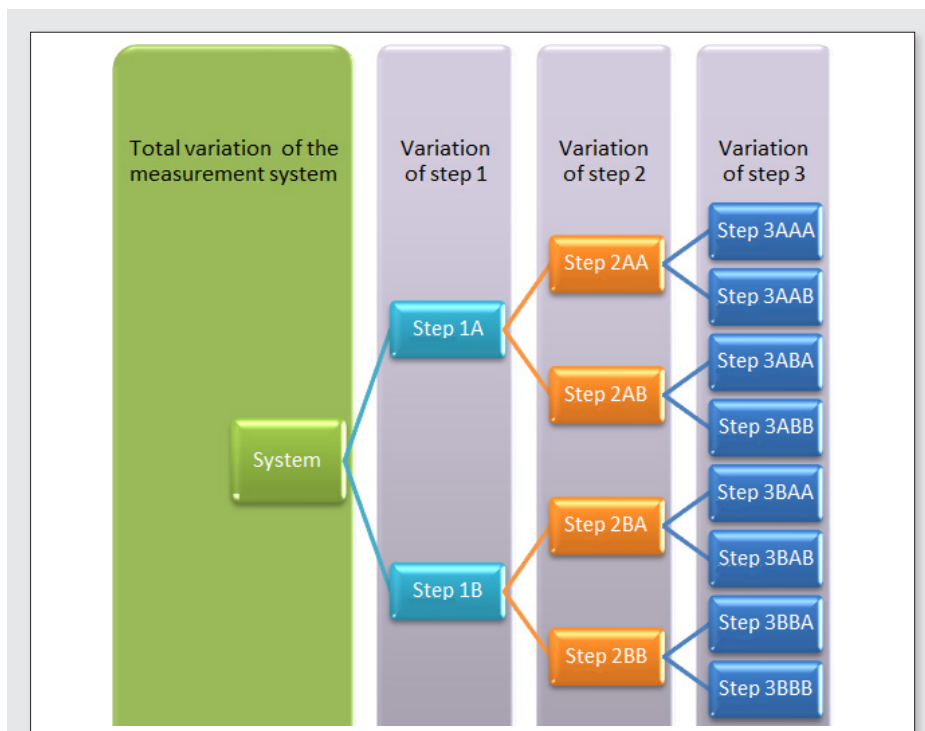


Figure 2. Generic illustration of a MSA study where *parallel samples* for each stage allow determination of the effective variations ascribable to each step. The illustration amounts to a duplicate split design, but more intense sampling replication is an option as well.

(step 3). Additionally, a step 4 would be the analytical instrument on which the samples are analysed.

In a MSA the measurement system is divided into sequential steps and parallel samples (replicates) are obtained to identify the resulting variations in each step. Care must be observed that all variance contributing factors are taken into account in the respective steps, for example operator errors, sampling errors, crushing and weighing errors etc. all of which contribute to the total observable variation.

This master design can be done in different optional ways, for example by using more parallels at each stage. The designer of the MSA in collaboration with the operative personnel has to decide why and when what level of replication is necessary.

Observe that this general MSA setup is equally applicable if the “sampling” consists of a direct process measurement instead of extricating a sample to be analysed in the laboratory. Esbensen and Paasch-Mortensen describe the duality of such process sampling with the physical sampling extraction, and show that there are no essential differences, both approaches give rise to the potential full complement of sampling errors (incorrect sampling as well as correct sampling errors).<sup>6</sup>

Another way to set up a MSA would be to focus on the reproducibility and repeatability of the final analytical results when different operators, instruments or other devices are used. The possible sources of the total observed process variation can then be decomposed as:

$$\sigma_{\text{Observed Process}}^2 = \sigma_{\text{Actual Process}}^2 + \sigma_{\text{Measurement System}}^2$$

$$\sigma_{\text{Measurement System}}^2 = \sigma_{\text{Repeatability}}^2 + \sigma_{\text{Reproducibility}}^2$$

where the variation due to *repeatability* and *reproducibility* are considered as the main contributors to the variation of the measurement system. The results of these kinds of MSA are often reported as “Gauge R&R” numbers. The stability and accuracy of the analytical measurement system can be determined by the use of *control samples* and/or *certified reference materials* where available. It is here necessary to distinguish between the analytical part of the measurement system in the strict sense and the total, effective measurement system, which specifically also includes the sampling system(s).<sup>†</sup>

<sup>†</sup>Observe that these attributes are only defined for *analytical methods/analytical systems*, but that there is no carrying-over option to the *sampling process*, see Esbensen & Wagner (2014),<sup>10</sup> who describe these crucial differences in full detail.

For a MSA there are several established statistical tools (*linearity, stability, gauge repeatability and reproducibility, GRR*) that can be deployed to ensure that measurement setups are within acceptable conditions to meet manufacturing capabilities and customer requirements. Observe, however, that this approach does not recognise issues arising from sampling process variances. The central issue is that TOS shows how the influence of so-called “incorrect sampling errors” lead to a *sampling bias* which is **inconstant**, and which therefore cannot be straightjacketed into the standard analytical variance decomposition scheme above, see References 6 and 10.

### Variographics and MSA complement one another

At Nikkelverk we would like to use the variogram as a *screening and grading tool* of the measurement system performance in order to decide if application of the tedious MSA is necessary. Figure 3 shows where the variogram and the MSA work well individually and where their scope coincides. Figure 3 shows that the total observed process variance, either delineated by a variogram or via a MSA can be decomposed.

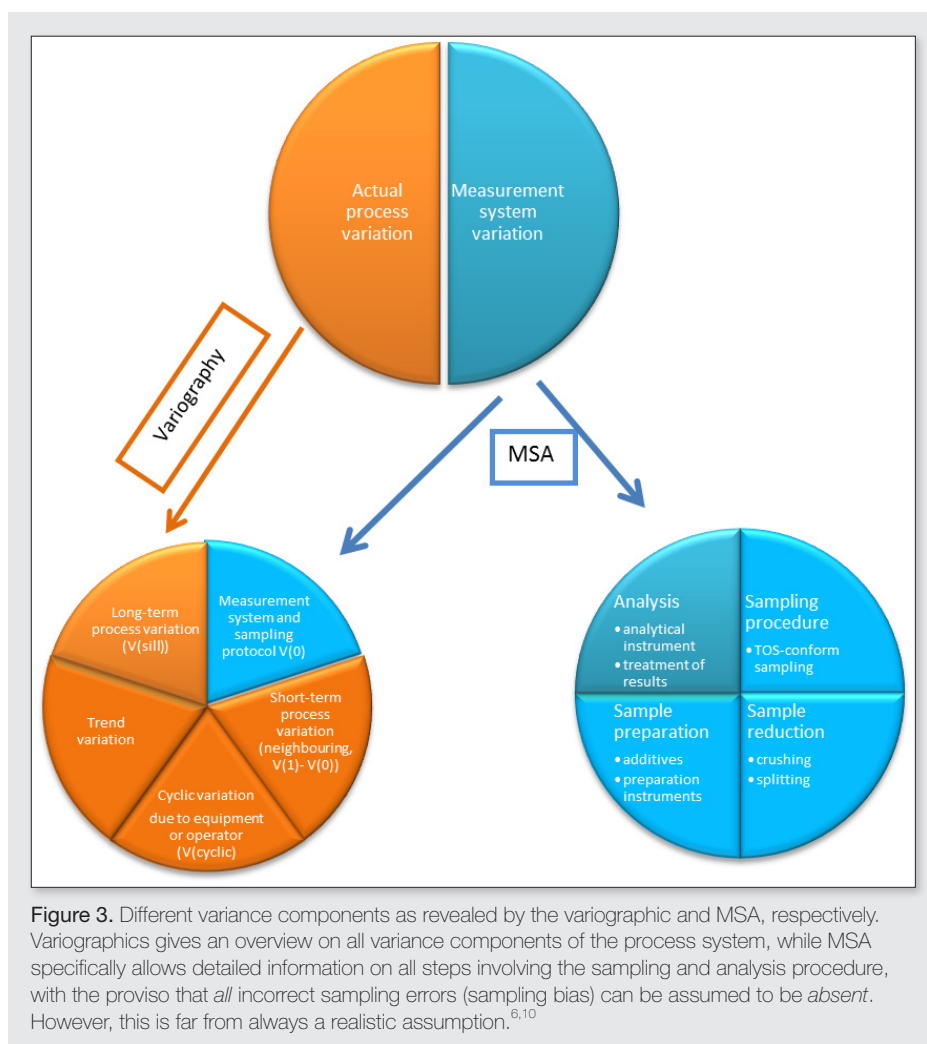
### Examples of effectively categorising measurement system conditions

The first step in a MSA is to understand how the results appear under normal operating conditions (NOC). In the software used for this feature this can be done either by looking at the time series and checking the distribution pattern of the data series by means of a histogram or by comparing a Q-Q plot against a fitted normal distribution.

It is recommended to test the measurement system with a slightly oversampled unit lag distance when performing a variographic characterisation; in this way an optimal correct lag distance can be derived.<sup>6</sup> This optimised lag distance should then be used when calculations are performed to determine the status on the measurement system in question (each day, each week or whatever is appropriate to the process). Below we present three examples of how variography can be applied to check particular measurement systems at Nikkelverk.

#### pH measurements

Measurement of pH is one of the most important measurements at Nikkelverk (where nickel refining is performed by a



**Figure 3.** Different variance components as revealed by the variographic and MSA, respectively. Variographics gives an overview on all variance components of the process system, while MSA specifically allows detailed information on all steps involving the sampling and analysis procedure, with the proviso that *all* incorrect sampling errors (sampling bias) can be assumed to be *absent*. However, this is far from always a realistic assumption.<sup>6,10</sup>

complex hydro metallurgical process) and therefore serves as a major indicator. Pilot studies of the use of variograms have been started regarding monitoring of the variation stemming from the measurement system itself. The software used for detailed study to calculate variograms is EMPV (Effective Management of Process Variability), from Francis Pitard Sampling Consultants.

There are many process steps at Nikkelverk which are controlled by pH measurements. A typical sampling station is presented in Figure 4a. The electrodes are immersed in the process reactor solutions, and they are cleaned every hour by lifting the electrodes into an acidic solution (Figure 4b).

Figure 5 shows a time series plot of on-line pH measurements at reactor 1, and a conventional statistical distribution of the measurement data from the selected time series is shown in Figure 6. The lag “distance” is 4 minutes and 19 seconds, corresponding to a complete series of 72 hours; the 1000 measurements used here were

obtained from the historical process system database.

The resulting variogram (absolute variogram) is shown in Figure 7. The sill,  $V(sill)$  is defined by the horizontal dashed line in black. The variance for the measurement system,  $V(0)$ , is defined the intersection of the back-projected curve with the y-axis corresponding to lag=0.

pH measurements obtained for six different weeks with the same time periode of 72 hours are extracted from the process database for reactors 1 and 2. The variograms of all weeks for each reactor are normalised with respect to the largest sill variance allowing all variograms to be presented and easily compared in one graph, as shown in Figure 8 for reactor 1 and Figure 10 for reactor 2. The coloured areas in the graphs indicate if the variance of the measurement system is well below company threshold limits to ensure that the process is the main contributor to the observed variations. These characterisations follow the Six Sigma approach.





Figure 4. a: Sampling station for pH measurement; b: lifted pH electrodes during the cleaning cycle.

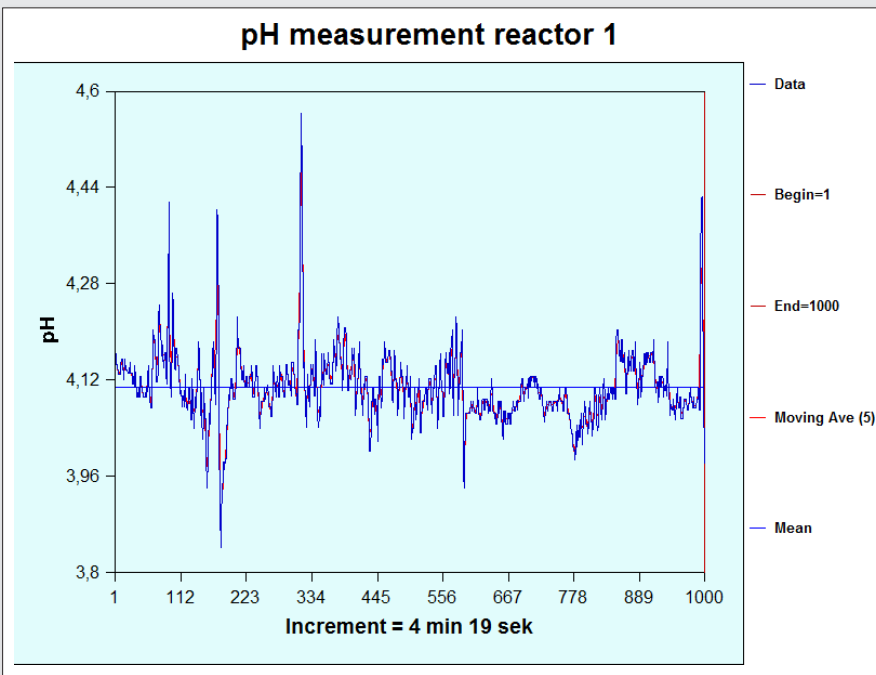


Figure 5. Time series plot of pH measurement of reactor 1 for three consecutive days in week 49, 2015.

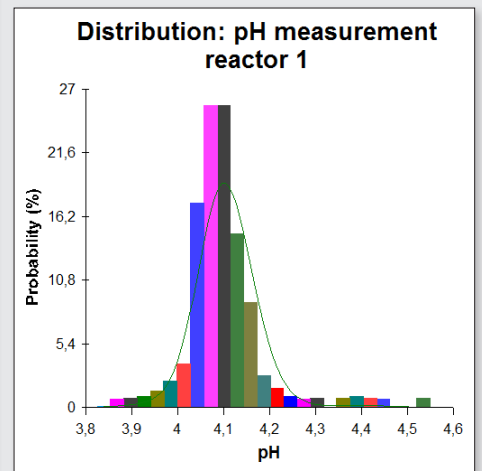


Figure 6. Distribution of the pH measurements presented in Figure 10.

In the study at Nikkelverk, classification of the different capability levels of a measurement system follows from terms used in MSA studies, Figure 9. The measurement systems for pH are therefore classified either as “capable” (green area) where the variation of the

measurement system,  $V(0)$ , is 10% or below compared to the process variation, “conditionally capable” (yellow area) where the variation of the measurement system is between 10% and 30% relative to the process variation and “not capable” (red area) where the measurement

system variation is above 30% compared to the process variation.

The picture of the sampling station for the pH electrodes, Figure 4a, shows several measurement points for the different reactors. Spillage of solution from one to another measurement system is unfortunately not

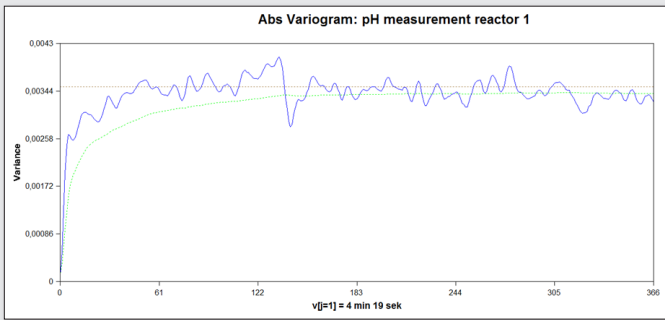


Figure 7. Absolute variogram of pH measurements presented in Figure 5. The green dashed line represents the first order integral  $W$  that is used to extrapolate the variogram back to  $V(0)$  and the red dashed line represents the total variance of your process,  $V(sill)$ .

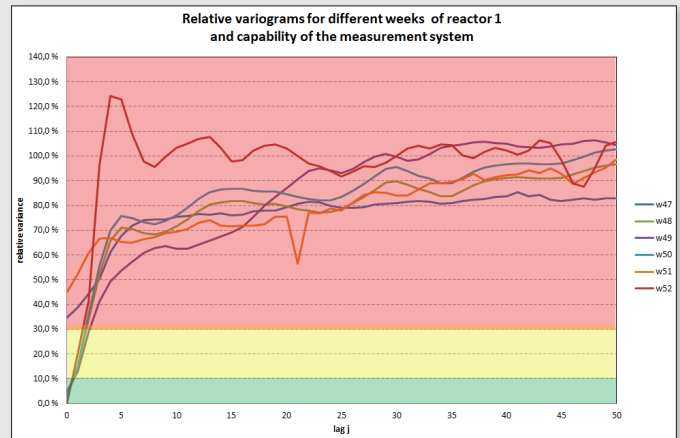


Figure 8. Six consecutive one-week variograms of pH measurements in reactor 1. The variance (y-axis) is normalised with regards to the largest  $V(sill)$ .

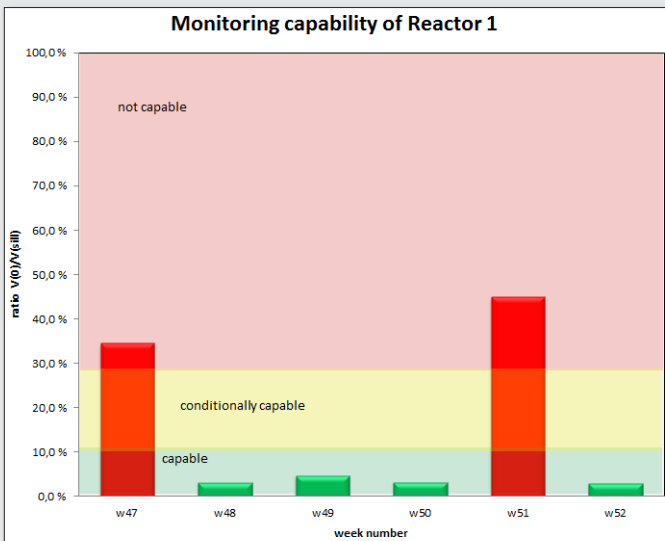


Figure 9. Measurement system *conditions* of pH in reactor 1 presented as ratio of  $V(0)/V(sill)$  per week showing capable, conditionally capable and not capable conditions following the Six Sigma approach.

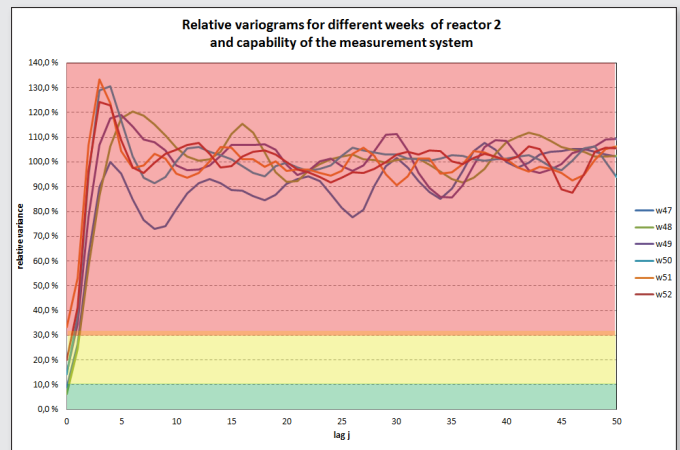


Figure 10. Variograms of pH measurements in reactor 2 per week presented over 6 weeks—one variogram for each week. This presentation uses the relative variograms.

completely ruled out, by an inconvenient design (it is, however, minimised). If something is wrong in the sampling station and the monitoring of the capability shows that the capability is in the red zone for parallel positions, this might be an indication of cross-contamination by spillage and can be checked by visual observation as well as corrected for.

*Alternative variographic characterisation of the measurement system*

DS 3077 (2013),<sup>5</sup> Esbensen and Romach<sup>11</sup> describe how the fraction of  $V(0)$  of the sill variance level, expressed as a percentage, can be used as a grading facility for the measurement system delivering the on-line time series involved. In this

approach a single threshold demarcation is recommended; measurements systems must not give rise to a  $V(0)/sill$  fraction larger than 30% in order for the measurement system to be reliable for valid insight into the process variances proper.

This furthers an alternative on-line measurement system characterisation that does not involve an experimental design and active intervention, as is the case for MSA. The six-week time series for pH measurements from reactor 2 is shown in Figure 10, here based on the relative variograms. It is easy to augment the variographic threshold 30% with the three-fold variance fraction classification system employed in MSA, as is shown in Figure 11.

From Figure 9 it is observed that for both weeks 47 and week 51 the measurement system in reactor 1 contributed to well above the specified limits for “not capable”. For reactor 2, Figure 11, this is only the case in week 51, while the variation of the measurement system seems to be at least “conditionally capable” in weeks 49, 50 and 52 or even “capable” in week 47 and 48.

Whether to use absolute or relative variograms can be debated, but in the present context both will lead to a measurement system classification that can be appreciated within the three-fold MSA brackets. The main issue is that it is possible to *grade* any process measurement system based directly on routine on-line process data. This is a huge efficiency improvement of the

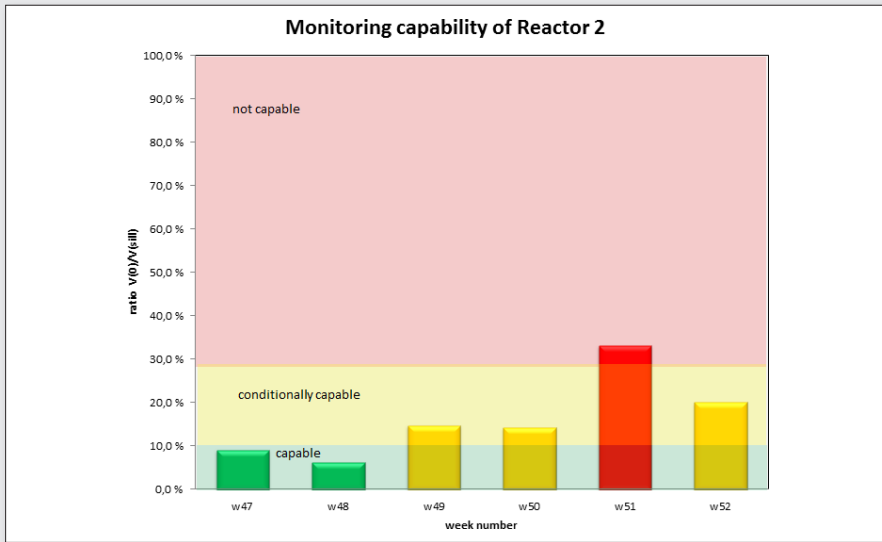


Figure 11. Measurement system condition of pH in reactor 2 presented as ratio of  $V(0)/V(sill)$  per week. The three-level grading classification system is adopted from Six Sigma MSA.

measurement system check over having to instigate the cumbersome full MSA procedure for all measurement locations.

### Online process analysis of Pb

There are different chemical elements which must be monitored closely to ensure high quality products at the end of Nikkelverk's production line. One of these elements is lead, which has to comply with very strict, low concentration specifications in the final nickel product. Formerly, lead was monitored by using polarography with mercury electrodes. However, recent restrictions by the authorities on using mercury in workplaces started a project in which a new method for lead analysis should be found.

One of the tentative new test systems is shown in Figure 12, which is installed close to the process in the so-called at-line configuration. There are several systems installed through the full process line and two examples of process data for reactor 1 and 2 are shown in Figure 12.

The sample is withdrawn in batch mode from the process, with a time interval of several minutes. The measurements are recorded continuously in the company database.

For reactor 1, a time series of 7 days is presented in Figure 13. Due to known external disturbances in the system, the first 120 measurements have been excluded from the calculations producing the variogram.

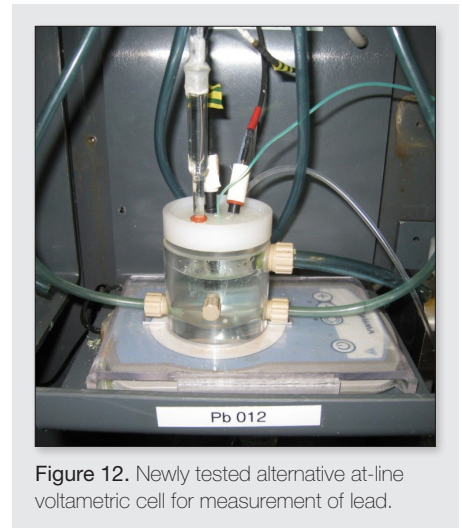


Figure 12. Newly tested alternative at-line voltametric cell for measurement of lead.

Since the measurement period is several minutes, a lag distance of 20 minutes was chosen to be used for the variogram shown in Figure 14 in concordance with the intimate general process knowledge accumulated over several decades.

For reactor 2 the same time frame and starting date are used to draw the time series shown in Figure 15. As for reactor 1 the time series for reactor 2 was also reduced to 380 measurement points, here to eliminate the highly irregular outlier shown in Figure 15. The resulting variogram is shown in Figure 16.

From these variographic analyses it easily appreciated that i) both measurement systems operate with a low  $V(0)$  relative to the sill level (very clearly below 10%), which is highly satisfactory; and ii) the largest contributor to the overall process variance is a cyclic phenomenon. So the challenge

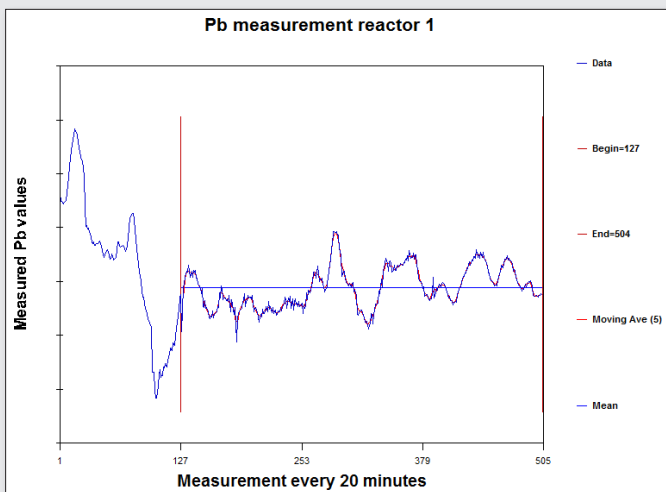


Figure 13. Time series of Pb measurement in reactor 1 (the first 120 minutes were excluded due to know irregularities).

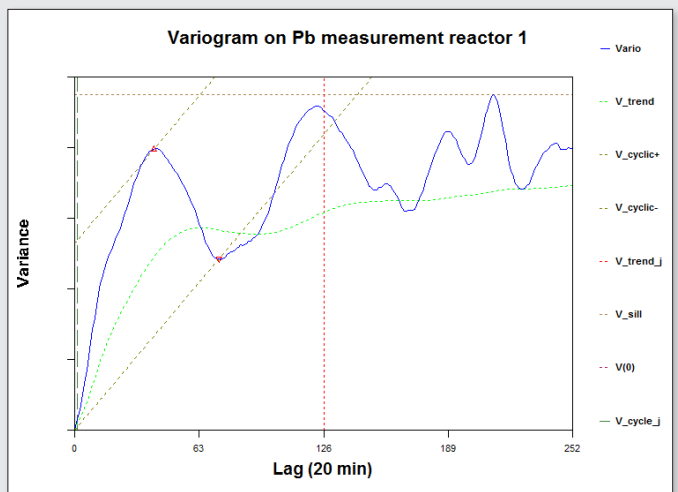


Figure 14. Relative variogram on Pb measurement in reactor 1 with lag distance 20 minutes.

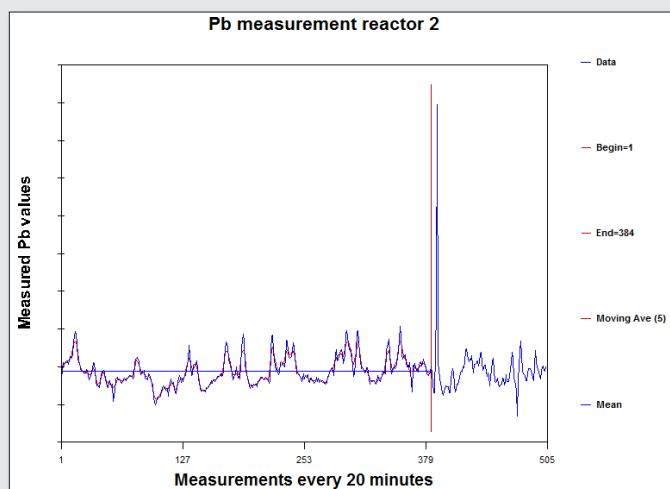


Figure 15. Time series of Pb measurement in reactor 2.

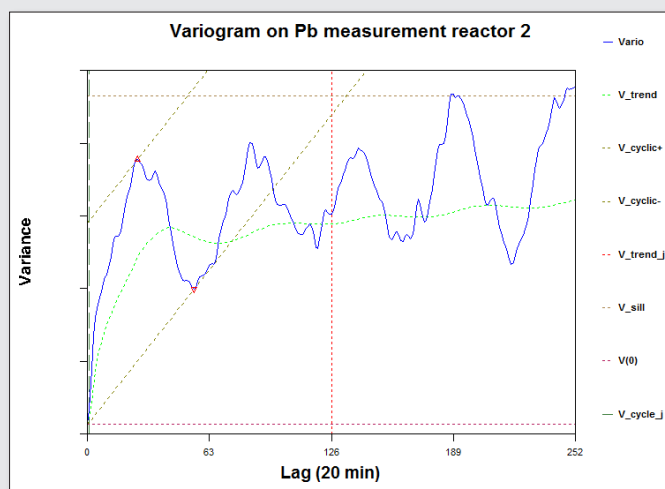


Figure 16. Relative variogram on Pb measurement in reactor 2 with lag distance of 20 minutes.

for this new alternative lead analysis is not the measurement system performance but the observed process cyclicality with a frequency of 20–22 hours. It is critical to identify the root cause of this behaviour since it contributes with ~50% to the total process variation. Nevertheless, the intention to use the  $V(0)/V(\text{sill})$  ratio to express the condition of the measurement system can still be upheld as it is not influenced by the large cyclicality. For the two reactors, the lead measurement systems are both capable due to both  $V(0)/V(\text{sill})$  being considerably smaller than 10%.

This verification of course has to be repeated after the observed cyclicality is reduced or has been completely removed from the process.

### Redox measurement

As for pH measurement, redox measurements are used extensively to determine

the status of the process. The measured mV signal gives an indication of the degree of process reaction equilibrium. The redox electrodes are installed at the same sampling station as the pH electrodes (Figure 4a). For reactor 1 a time series of a mV signal from a redox electrode is shown in Figure 17. As for the other examples it is important to check whether the data series includes significant shifts or if pervasive trends are present during the measurement interval.

The variogram of the redox measurements in Figure 18 show a cyclic behaviour with a distinct periodicity of one hour which is clearly a representation of the washing cycle of the bank of electrodes. The intersection with the y-axis determining  $V(0)$  is again low compared to the total process variation; the ratio is ~8% which signifies a “capable” status of the redox probe at the time.

### Synoptic overview of measurement system status

To illustrate the overall benefits of using the rapid on-line variographic measurement system, data series for time frames of three day’s duration were extracted from the reactor 2 process database, resulting in seven separate time series equal to the example presented for the pH measurements. The resulting capability classification is shown in Figure 19.

In Figure 19, as well as Figures 9 and 11 for the pH measurements, the ratio between  $V(0)$  representing the variation of the measurement system and  $V(\text{sill})$  representing the variation of the process are shown and company-developed MSA terms applied to the three ratio levels: <10%, 10–30% and >30% following the Six Sigma approach.

This is the same measurement system *quality index* approach described in DS 3077,<sup>5</sup> with the mandate that this index

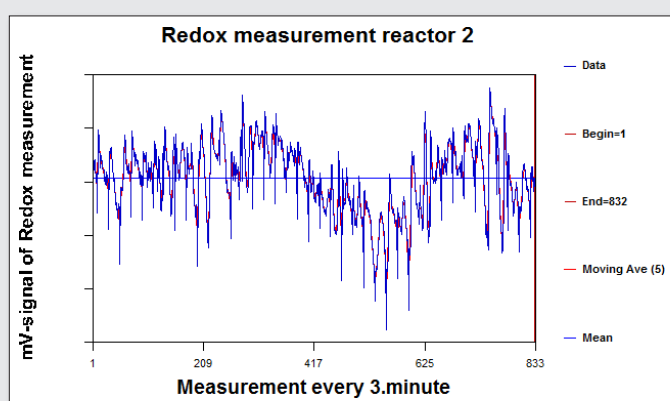


Figure 17. Time series of redox measurement in reactor 2.

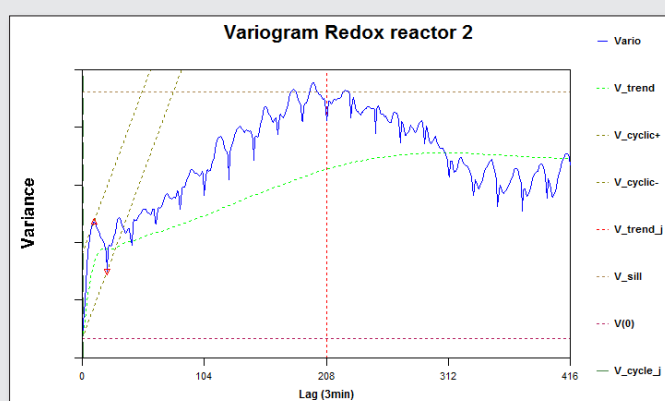


Figure 18. Variogram of redox measurement in reactor 2 shown in Figure 17.



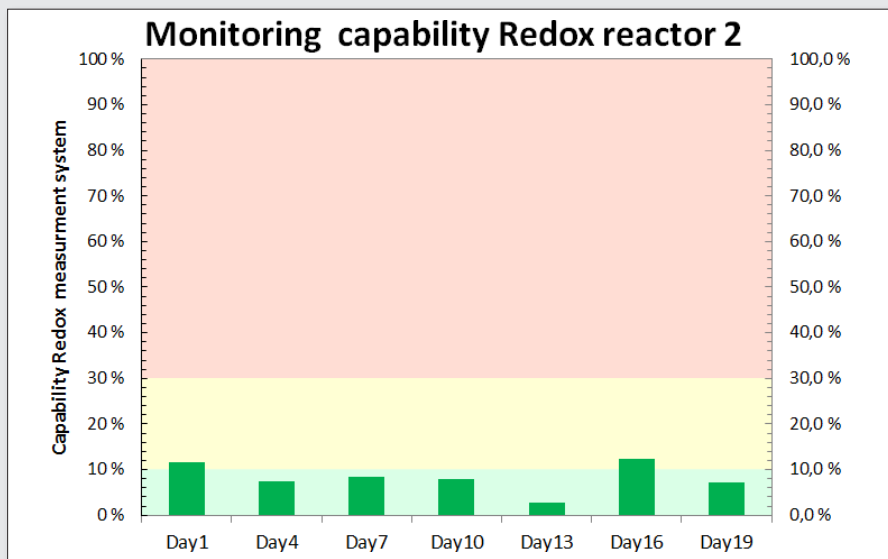


Figure 19. Monitoring overview graph to classify the capability of the redox measurement for reactor 2 with a time interval of 3 days for each bar.

must be made public in order for the quality department (or any other user of the process data or by product end-users for that matter) to get the necessary *insight* into the measurement system performance in relation to the total magnitude of the observable process variations. A parallel example, although applied to a very different industrial process (mixing in pharmaceutical productions) was presented by Esbensen and Wagner using the same approach as the one adopted here.<sup>11</sup> This parallels how complete process-and-measurement system evaluation can be achieved by simply “piggy-backing” on the existing process data acquisition, i.e. no specific MSA experimentation necessary.

## Discussion and conclusion

Figures 9, 11 and 19 show how the condition of a measurement system can be derived from on-line data and compared to the magnitude of the contemporary process variation.

The specific time period selected for variographic characterisation is important. Too long lag distances may lead to the impression that the measurement system is a major source of variation. Too short distances between the measurements can give several data points with the same value following each other, since the process system is programmed to not change the stored data if the measured data does not deviate from previous data. The choice of the time period to monitor the variation of a measurement system must be competently

and carefully defined for each system separately, always based on the most comprehensive process experience, and validated and revised at regular intervals.

Measurement system changes (electrode replacements, setups etc.), for example due to maintenance, should show up and will be documented with the conventional monitoring graphs and will thus give an easy understanding on why systems might “suddenly” show a higher variation. When skilled at working with these monitoring graphs (e.g. Figures 9 and 11), specific measurement system patterns can be detected, for example as for the pH measurements in week 51 for reactors 1 and 2 which were defined “not capable”. Operators and process engineers familiar with the design of the process know that both pH electrodes used in this study are situated at the same measurement station. At the measurement station several reactor liquids are collected in defined compartments running with a steady flow and pH for all reactors are measured with separate pH electrodes. However, cleaning routines are common for all pH electrodes. Assuming that these routines are **not** followed (for a variety of *possible* reasons), the pH electrode measurement system variances at the measurement station,  $V(0)$ , might increase and this could be easily observed with monitoring graphs of the types shown in Figures 9, 11 and 19.

It will also be possible to review all preventive maintenance intervals in which exchange of parts in measurement systems have taken place to inspect the resulting

performance quality index changes—and to adjust the maintenance intervals, for example, if changes are unnecessarily made too often. Since the redox electrodes are located at the same sampling station as the pH electrodes, combining the results from both measurement types might even give an indication on what might be the root cause if direct faults are discovered.

It would appear that evaluation (grading) of measurement systems by on-line variographic analysis can be very helpful in selecting which measurement systems are in need of a more thorough evaluation (MSA). Variographic process variability characterisation will easily show whether a critical process measurement system has a  $V(0)$  of 10% or below, relative to the contemporary process variation  $V(\text{sill})$ , fully qualifying the existing system for duty. Including the  $V(0)$  and  $V(\text{sill})$  values in the monitoring chart should be considered in order to reveal sudden shifts in time series data which would be camouflaged in a high  $V(\text{sill})$ -value and resulting in a low  $V(0)/V(\text{sill})$  ratio.

Further work for Nikkelverk with this monitoring opportunity will include development of a standard operating procedure (SOP) on how to estimate the correct time range for the process where the measurement system variation is best investigated. Today operators/process engineers acquire knowledge of system performance by manually checking *individual* data series with standard SPC charts and/or (recently) by using variographic analysis. One has to be aware of the consequences of too-small time intervals when extracting data from the process monitoring system. The responsible operator/process engineer has to have been properly trained, have proper experience and has to know the historical circumstances of the measurement system—as well as its interaction with the process. It is necessary to work as a *team* with the operators in the field. This type of team approach has been well described, for example, in chapter two of the standard work *Process Analytical Technology*.<sup>6</sup>

At Nikkelverk, weekly monitoring has to be setup automatically in a thoroughly user-friendly context to ensure that system characterisations, and therefore also system benefits, are easy to acquire and understand, and appropriate graphs must be actively used by all operators and process engineers, as well as by maintenance personal. An ideal example is shown in Figure 20.

Process A		Day 1	Day 2	Day 3	Day 4	Day 5	Day 6	Day 7	Day 8	Day 9	Day 10	Day 11	Day 12	Day 13	Day 14
1	pH														
	Redox														
	Temp														
	Flow														
2	pH														
	Redox														
	Temp														
	Flow														
3	pH														
	Redox														
	Temp														
	Flow														
4	pH														
	Redox														
	Temp														
	Flow														

Figure 20. Schematic example of measurements system capability overview.

An example of an overview sheet for different measurement systems is shown in Figure 20. This synopsis allows an easy overview of all measurement systems performance and conditions and will help to find the root cause of the compound process variation.

As in earlier figures, colour codes show if the graded measurement system is capable (green), conditionally capable (yellow) or not capable (red) in the time period selected.

Furthermore, standard operation procedures (SOP) giving out-of-control-action plans also have to be developed to specify in all necessary detail what to check and what to do when a measurement system shows up as “not capable”.

It seems obvious that this type of variographic monitoring will help to stabilise processes at Nikkelverk further and increase our understanding of what to address when tending to run out of control. Measurement system analysis is still viable and available, but can now optimally be conducted **only** where it proved necessary—either due to high variations of the measurement system or at process locations which are not monitored frequently by on-line or laboratory measurements. The use of the recently introduced variographic characterisation is clearly a further step forward as part of Nikkelverk’s policy for controlling all critical processes with the ICCM (identify, control, capable and maintain) approach.

The application of variography as a method in process industry is endless. It

is simple to *dare* just to start using it—and consider every irregularity (and many will always be observed) as a golden opportunity to learn more about the process(es), the measurement system(s) and their critical role in the broader company landscape.

Consider the case in which measurements systems problems are **not** discovered by the suggested frequent variographic scanning of the full array of systems. The consequences would be that the total variation cannot be understood with certainty, but would very often likely be attributed to the process alone since in this case there would be no fact-based evidence that the measurement systems are not working properly. This will invariably lead to *unnecessary* correction of the process parameters, or lead to faulty and quite unnecessary “corrections” of the measurement system. As an example, an undiscovered error in a pH measurement by a 0.2 pH unit over several days will in such a case lead to an excess use of correcting chemicals which is in reality completely unnecessary. It is critically important to control both process and your measurement system with help of variography.

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