Case study - Improved sampling of iron sludge at Glencore Nikkelverk

Eirik Djuve[a*] and Elke Willam Thisted[b]

[a]**Glencore Nikkelverk AS, Vesterveien 31, P.O.boks 604, 4604 Kristiansand, Norway. E-mail: Eirik.Djuve@glencore.no**
[b]**Glencore Nikkelverk AS, Vesterveien 31, P.O.boks 604, 4604 Kristiansand, Norway. E-mail: Elke.Thisted@glencore.no**

Glencore Nikkelverk produces nickel, copper and cobalt with high purity. This demands several leaching and filtration steps removing contaminants to achieve a high-quality metal via electrolysis. In one of the process steps iron is oxidized to its trivalent state and formsa slurry precipitate, which contains different iron components described as $Fe_xO_y(OH)_z$. The slurry is very fine-grained, corrosive and has a miry consistency resembling muddy clay. Filter presses with up to 90 frames separate the process solution from the iron slurry, which undergoes a washing cycle. Due to the slurry's fine-grained nature, this washing cycle is challenging with uneven distribution of the washing water and results in loss of valuable dissolved Nickel chloride ($NiCl_2$) which is left in the slurry. The slurry discharges of the filter presses are stored in Nikkelverk's underground landfills and leads to a yearly loss of approximately 20 million Norwegian kroner. In 2017, the R&D department at Nikkelverk took spear samples for over a period of nine month to map the Nickel content in the material prone to be discharged to the underground landfill. The results showed that the Nickel lost was approximately 40% higher than reported from the samples taken directly from a frame by the operators. In 2018, a mapping of several press frames by handheld X-ray analysis showed that the Nickel content, depending on the position of the sample taken in a frame, varies. Since the physique of the operators determines where the sample is prone to be taken, this will add to the variation in the analytical result of Nickel lost. It can be assumed that this variation completely overshadows the process variation resulting in lost Nickel. In summary, the current sampling protocol gives a high variation and a wrong content of Ni in the slurry. Nikkelverk's commitment to its business system and thereby involvement of resources on any level of the company has resulted in formations of process teams which shall approach such issues and find solutions fit-for-purpose. Therefore, a process team was initiated to develop an automatic sampler in-house for the iron slurry. Their goal was to achieve samples that are more representative as the ones retrieved by today's protocol. There are six filter presses where slurry is separated from the filtrate. Each filter press releases its slurry on a conveyor belt, which transports the slurry to a collecting chute. The idea is to mount an automatic sample in the collecting chute prior to the slurry compiled for transport to the underground landfill. The article will describe in detail the pre-studies of the problem, the development of the automatic sampler for the collecting chute and discuss its conformity regarding the theory of sampling.

Introduction

Since 1910 Nikkelverk has refined, produced, and exported nickel and other metals from the production plants in Kristiansand. The environmental, energy and process technology used at Nikkelverk has made the plant one of the most effective and technologically advanced refineries in the world. An important part of the extensive value chain in the Integrated Nickel Operation, it is of the utmost importance to handle the intermediate raw materials responsibly and avoid losses at the refinery. Achieving a high material yield is desirable both from an economic and environmental point of view. It is also vital to reconsider traditional waste streams occurring during production. If properly managed regarding specifications, waste from one company can be of value as raw material for another. When striving for a sustainable operation to serve social, environmental, and economic purposes, one goal must be to revise existing sampling procedures and check their conformity towards the theory of sampling (TOS). This is both valid for physically withdrawn samples and sensors representing process analytical technology. Any kind of sampling in the process industry should be fit-for-purpose, representing the actual condition of your operation. Only then one can place confidence in the resulting data and the decisions made upon them ensuring a sustainable production.

During the last century Nikkelverk has developed towards a highly technological company through engaged staff and high competency in several disciplines. Productivity tools like Six Sigma and Lean were applied to systemize work and utterly improve performance of the process. To achieve maximum efficiency, both in terms of staff performance, management and processes, concepts on how to organize improvement work developed further. The latest productivity tool currently used at Nikkelverk, Nikkelverk Business System (NBS), encourages to form temporary working groups of employees from different areas and with different backgrounds to solve identified problems. The investigations and planning on the automatic sampler described in this paper were done in such a group.

Background

At Nikkelverk, the sulphide-based matte is processed in several leaching and purification steps to high purity metals needed in a modern society as simplified illustrated in figure 1. Impurities are separated from the nickel containing filtrate by precipitation. It is important to keep process parameters at an optimum to ensure maximum output of metals in the filtrate..
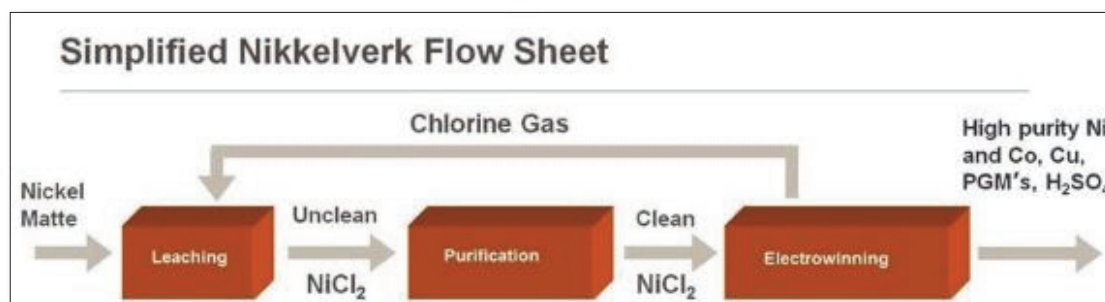

Figure 1. Simplified flow sheet for the Glencore Nikkelverk production process.

Separation of the filtrate from the precipitate is mainly done using filter presses. The filter presses are filled with the liquid/solid mixture, and thereafter pressure is applied to remove the liquid phase to be sent to the next process step. The remaining solid phase undergoes a washing cycle with water to remove the remaining liquid phase containing metals for production. When completed, the press frames are emptied sequentially on a conveyor belt, the content is collected in the basement and transported to a storage facility, figure 2. Depending on the content of the solid phase, it is either returned to a prior process step or stored at the disposal site. Samples taken from the solid phase of each filter press whilst emptying, indicate the performance of the washing cycle.
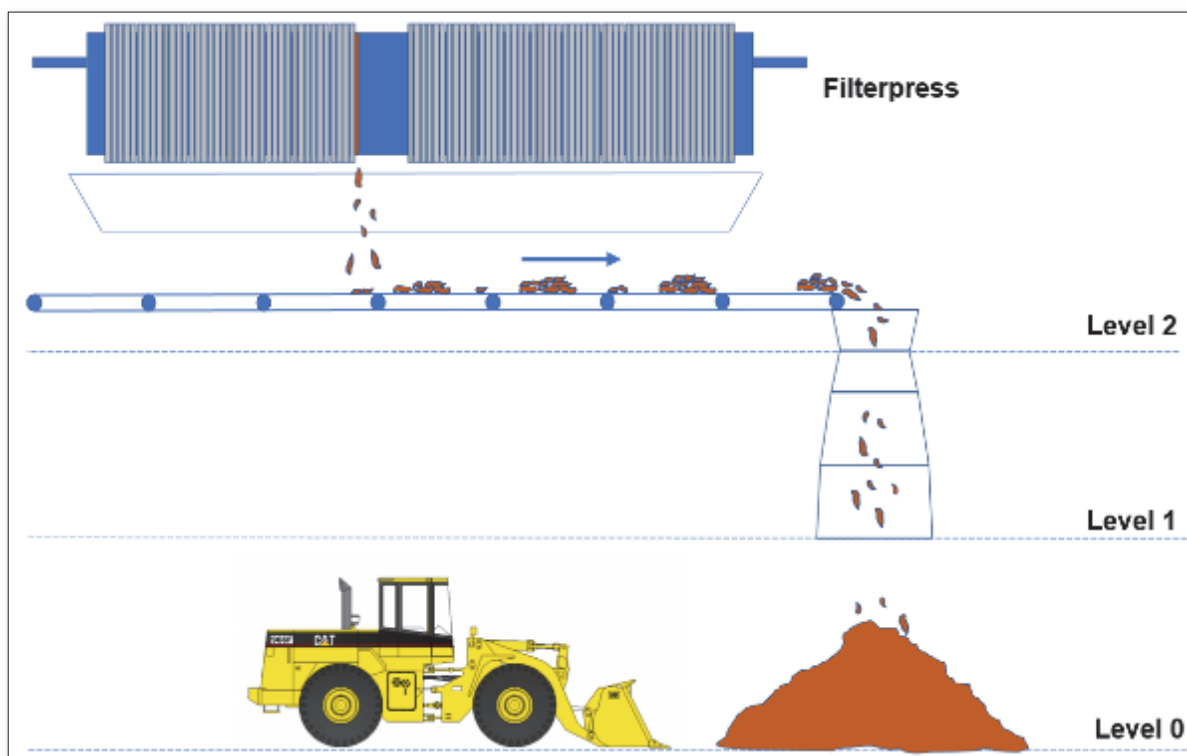

Figure 2. Schematic diagram of emptying a filter press and transportation of solid phase to the storage facility.

In the process step where iron sludge is removed from the process stream several filter presses are in operation. Each filter press contains over 90 frames with a height of 180cm and a width of 100cm of the filtration surface. Though presentations on the internet from filter press suppliers show an evenly distribution of the liquid/solid mixture, the filling of a filter press with iron sludge will certainly lead to a heterogeneous filter cake for each frame and for the whole press. The current sampling routine is done manually by taking samples on one of the frames of the filter press frame chosen by each operator. The press frames are emptied on a conveyor belt and the total content of the filter presses is transported to a storage facility. The manual sample of an arbitrarily chosen frame is most likely taken from the region marked with the red frame in figure 3. Operators take a piece of the sludge from the filter press frame as shown in figure 4, and in addition to bagging a lab sample, they often take a conductivity test. This is done by mixing a fixed amount of sludge and water, using a kitchen hand-mixer. The conductivity of the resulting solution is measured using an inductive conductivity meter. The conductivity comes from water soluble salts in the iron sludge which is mainly based on $NiCl_2$ in the sample. Therefore, this indirect measurement serves as an indicator of the remaining Nickel content in the sludge and can help operation to adjust parameters in the filtration process.

Figure 3. One of the filter press frames with iron sludge. The red framed area marks the most likely reachable region for the manual sample taken by the operators.

Figure 4. Manual sampling from the filter press frame.

Studying and understanding the problem

The sludge samples, originating from grab sampling, are sent to the laboratory, and analyzed by XRF to determine their Nickel content. During an arbitrarily chosen two years' period, the resulting Nickel content shows a relative variation of 110% (u = 2 std), figure 5. This indicates either that the process is not under control or that the samples taken are not fit-for-purpose where the latter might the most relevant issue to look at first.
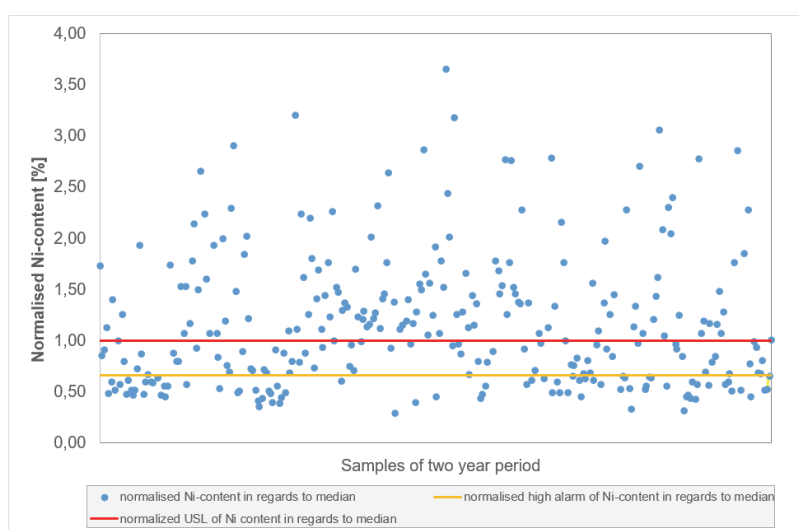

Figure 5. Normalized Nickel content compared to median over a period of two years.

Apart from the probable issues during filling and cleaning of the filter sludge in the filter press, there exist a certain likelihood that during the water wash cycle channels are formed where the water can easily go through the filter cake without washing all of the sludge content in the frame. Independent of the root causes leading to this event, it increases the variation of Nickel content from frame to frame.

A sample taken from an arbitrary filter press frame represents the Nickel content of the whole filter press with over 90 frames. Since the position of where to withdraw the sample was not standardized in the beginning of investigating the sampling problem, an additional contribution to the large variation could be assigned to the different height of the operators determining where it is most comfortable to take a sample. Additionally, sampling is usually done by hand with protective working gloves which are also used elsewhere in the process and can at times be contaminated with Nickel salt. This raises the concern that samples are unwillingly cross contaminated and wrongly contribute to the increasing variation in the resulting Nickel content. As recent as February 2022, a procedure for manual sampling was introduced. This helps the situation somewhat by ensuring that the sample is always taken from the same couple of frames, and it also emphasizes the use of clean gloves to avoid contamination.

Investigation of the filter cake content 2017/2018

In 2017, an attempt was made to determine how much Nickel was lost to storage by investigating the Nickel content in the sludge pile at ground level (Level 0, figure 2). Spear samples from the storage pile were removed over half a year and the results were compared to the corresponding, manual grab samples from the filter press frames. The same analytical method at the main laboratory was used for both sample types to determine the Nickel content.

Figure 6. Illustration of spear sampling the iron sludge pile at ground level.

The comparison showed that on average approximately 25% more Nickel was lost to storage than registered via the standard operating procedure with grab samples from the filter press frames. Though spear sampling might not give a fully representative answer[2], the results indicated that the current sampling grab sampling procedure do not represent the actual content of Nickel in the sludge, figure 7.
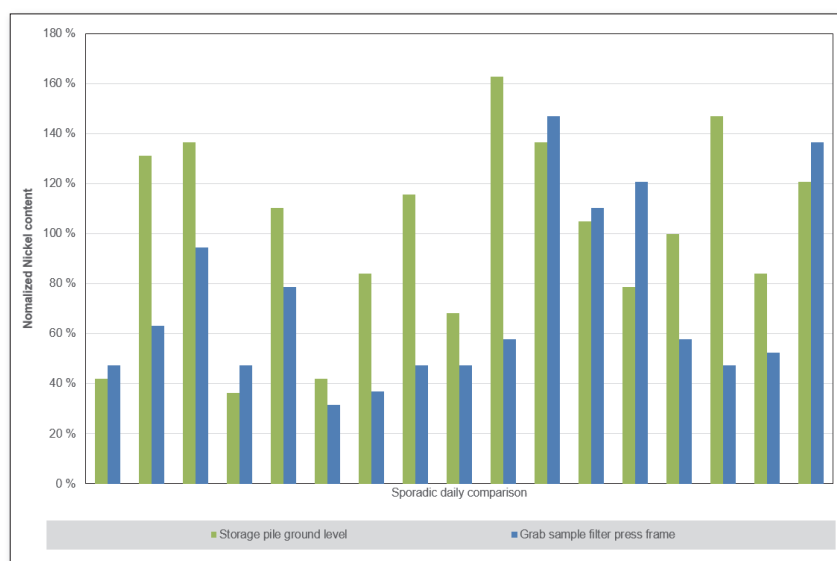

Figure 7. Comparison between grab sampling filter press frame vs spear samples storage pile over half a year, normalized according to the average of each sampling series.

In May 2018, another investigation was undertaken to prove the heterogenous distribution of the Nickel content and map the distribution of Nickel in selected filter press frames. Suppliers of filter presses present the filling procedure as an operation where

slurry is distributed evenly on the whole frame[6].. However, in reality the sludge will be affected by gravity and fill from the bottom up rather than distribute the sludge homogenously over the filter frame. Since there can be deviation from an optimal washing and airdrying cycle of the filter press, Nickel might be more prone to remain in certain parts of the frame.

It was planned to measure the sludge in the filter press frame directly before discharge to the conveyor belt. A handheld X-ray instrument (HH-XRF) from Niton, XL3t-GOLDD+ from 2018 with an Ag-Xray tube and an SSD-detector was used. The most appropriate program on the instrument was chosen. This was the mining programme with four separate programs analysing the sludge sample for 30 seconds for each program, determining the elemental content ranging from heavy to light elements. The total analysis time is two minutes and makes it challenging to keep the HH-XRF at its position on the frame. Additionally, several positions on the frame should be analysed qualitatively, preferably five to six positions, which challenged the need to free the filter press for production purposes. Therefore, on site, another strategy was chosen, where physical grab samples from the filter press frames were placed on filter press clothes placed aside to reproduce their actual position. This secured both continuous production and enough time to analyse the samples withdrawn from the selected filter press frames.
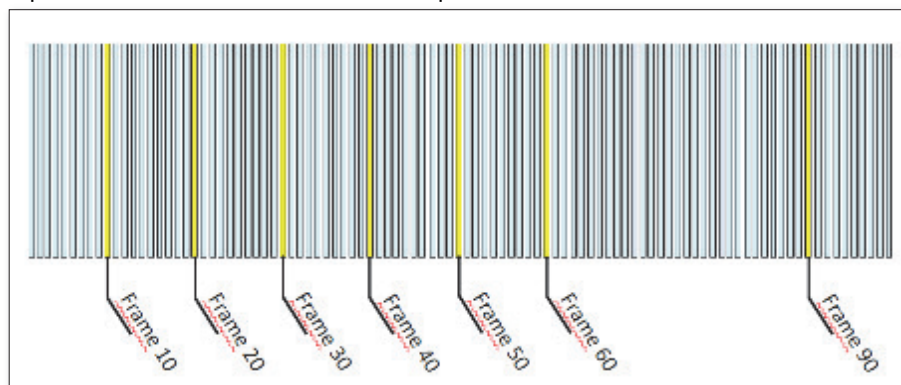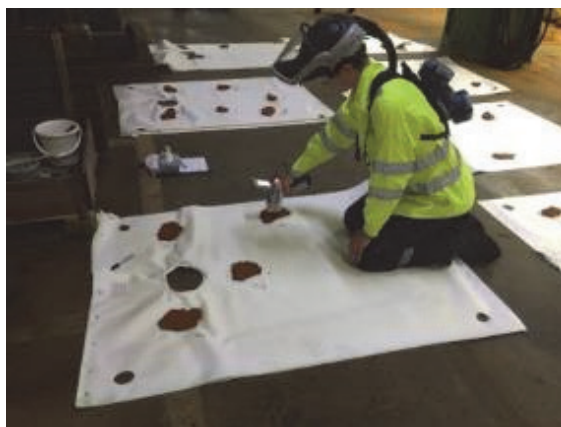

Figure 8. Illustration of the sampled filter press frames selected for more thorough analyses of the distribution of the Nickel content.

Seven filter press frames were chosen as shown in figure 8 and for each frame samples were placed approximately on the place of withdrawal, figure 9 and 10.

Figures 9 and 10. Samples positioned on filter press clothes for each frame selected for more thorough analyses of the distribution of the Nickel content with HH-XRF.

Depending on the element to be analysed one must consider if the element to be analysed is properly represented in an x-ray context. For Nickel, as one of the heavier elements the periodic table of elements, the penetration depth in the sample should be sufficient. As the filter sludge containing mainly a type of iron hydroxide, the matrix of the samples is considered medium heavy. However, the penetration depth of X-rays is theoretically calculated to be only a few millimetres in this matrix which cannot ensures a proper presentation of the Nickel content in the whole sample. Nevertheless, it should be kept in mind that the main goal of the investigation was to prove that there exists an uneven distribution of Nickel in the filter press frames.
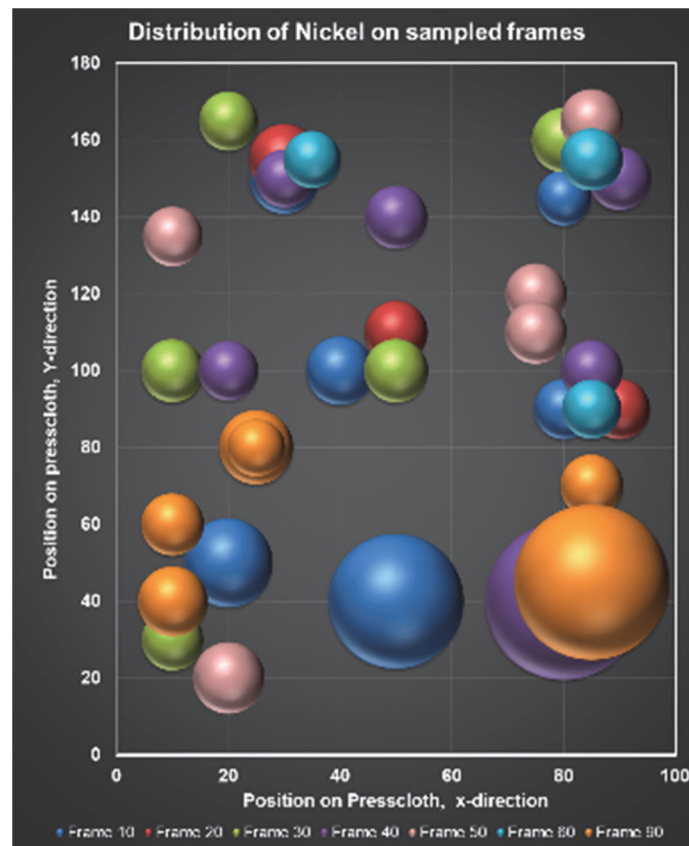
Figure 10. Illustrated distribution of the Nickel content in seven filter press frames. Size of circles is related to the Nickel content.

There are hotspots of high Nickel content at one corner bottom of the frames at the day of sampling, figure 10. The root cause(s) could be the washing cycle procedures and so forth. It is expected that minor Ni-components, which are not water-soluble, will not be removed from the sludge. However, efforts are made to reduce the loss of Nickel in the sludge to the absolute minimum. To achieve this, the control measurements of Nickel content in the sludge must be improved. An automated sampling system is planned to be installed where the conformity towards the theory of sampling (TOS) must be considered.

How to plan a sampler from scratch

According to NBS, a process team was formed with shift operators, mechanical and project engineers as well process metallurgists and electric and instrument engineers on request. Their expertise on the process, material to be sampled and mechanical components was assumed to be sufficient to build a robust automatic sampler. It might seem as an ambitious and maybe even imprudent assignment to plan a sampling system from scratch with inhouse resources. However, one must understand that Nikkelverk has had several mediocre experiences with external suppliers over the years, promising plug and play solutions and resulting in equipment with insufficient performance, extended workload on our inhouse resources and thereby lost value for the company. The main reason for failure of purchased equipment from suppliers is probably the unusually corrosive solutions and sludges being processed at Nikkelverk, which require very robust equipment made of corrosion resistant materials. Therefore, a determined attitude developed over the years to primarily fix issues and challenges mainly with own resources. The Nikkelverk business system also encourages to engage people from several disciplines to solve such problems together as a team. This gives each team member the chance to broaden their knowledge on other areas and motivates them to contribute to a sustainable solution. When implementations of solutions show better results for process control and so forth, the team will additionally be rewarded with a proud feeling having contributed to improved performance of their company. Unfortunately, the team had close to zero knowledge of TOS and had to rely on past experiences and common sense instead. This is obviously affected the samplers' coformity to TOS.

Considering the development of a sampling system for the iron sludge, several workshops and brainstorming sessions in the process team were held to understand the scope of the problem and the challenges involved. Many ideas were discussed, but two possible concepts were considered more thoroughly. The first solution was a sampling device cutting through the falling stream back and forth. This idea was considered to be the least viable, since the discharge chute had to be reconstructed to fit the sampler. The second idea resembled an input feeder which is a well-known technique for transportation of materials at Nikkelverk. Placing it across part of the falling stream seemed to be more robust and easier to implement then the first solution. Emphasis was on the flow behaviour of the material and practical issues involving sample extraction and the functional reliability of the new sampling equipment. In the initial stages, less thought was given to the representativity of the samples extracted representing the sludge content of one filter press. This is mainly because the slurry is sticky and corrosive, so creating a robust and reliable automatic sampler in such an environment is hard. If the sampler works well also gives a more representative sample than before, this would already be a great success and a hard enough challenge even though there is still much more potential for increasing the representativeness of the sampler. The idea considered was a tube placed across the discharge chute as shown in figures 11 – 13.

Placed in a fixed position in the falling stream of the iron sludge, an opening on top of the tube going over the whole length of the falling stream, should ensure that parts of the discharge were collected. The extraction of the sample from the tube was planned to be done with a piston pushing "increments" towards the end of the tube and into a container placed beside the chute.

Figure 11. Picture showing the discharge chute for the iron sludge.

Figure 12. Illustration of planned automatic sampler with collection container besides the chute.
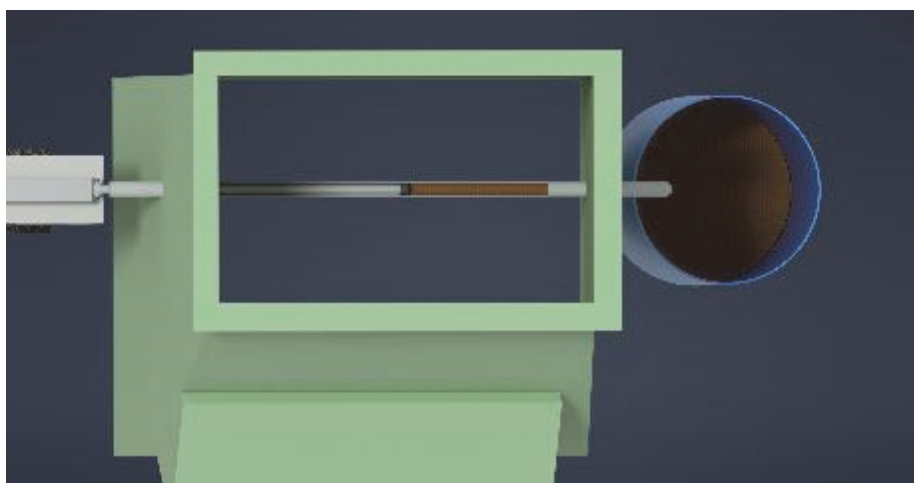

Figure 13. Top view on the planned automatic sampler with collection container besides the chute.

Prior to implementing the complete sampling system, manual tests are planned to check if the concept will work properly and manages to extract parts of the falling stream during the filter press discharge operation.

The chosen sampler's conformity to TOS

As shown in the previous sections of the paper, substantial work has been done to prove that the previous sampling procedure was unfit and had to be redesigned. In the previous section the chosen sampling system design was presented which shall be compared to the principles of the theory of sampling. As for any sampling and reduction system, one has to know all possible errors which can occur. There are errors due to the material, the process, and the sampling system itself. As stated in the literature and courses related to TOS[3, 4, 5], the first thing to keep at a minimum – and optimally avoid – are the incorrect sampling errors due to the sampling system, which are the increment delimitation error (IDE), the increment extraction error (IEE), the increment preparation error (IPE) and the increment weighing error (IWE).

Increment deliminitation error

Since the mass stream consists of a slurry, preferably with low liquid content, the material will agglomerate in large lumps. The size of the lumps may depend on size distribution of individual particles, chemical composition, and solid fraction among others. Hence, there might already be a correlation between the flow properties and the parameter that should be measured. As a consequence, one shall expect a variation in the Nickel content of the slurry leading to a bias in the result. Therefore, designing an equipment that maintains the equiprobability principle is important. Each particle in the mass stream has to have the same likelihood to be sampled. The proposed design, figure 12 and 13, extracts samples only at a fixed position and violates thereby the equiprobability principle. Ideally, the sampler should cut through the entire falling stream at a constant speed and keep the geometry of the cut constant to avoid or at least minimize the IDE – strike 1 for the chosen sampling design.

Increment extraction error

Next, the sample must be extracted correctly to minimize the IEE. It is important that the rule of the centre of gravity is followed and thereby ensures that all fragments can fall randomly on either side – into or outside the sampler. Since the slurry lumps have a maximum diameter of about 5-6 cm, the cutter opening shall ensure that the largest fragments can be collected as well. A rule of thumb is to have a cutter opening at least three times larger than the largest fragment of the increment i.e. at least 15 to 18 cm in the case of the planned sampler. As for the current setup, the cutter opening of the sampling tube has a width of 2,9 cm (see figure 14) which means that lumps will break when falling onto the sampler's opening. In addition, the fixed sampling tube has not enough volume to capture the sample fragments and will be filled rather fast building up lumps on top. The sampler should ideally only be 1/3 filled when cutting through the falling stream ensuring that the fragments sampled cannot escape the sample container after being collected. Hence, the suggested design has insufficient capacity to cope with the flow rate.

For moving the slurry lumps from the cutter to the collection container, the sampler should ideally be equipped with a transportation belt or screw inside the cutter suitable for sampling of non-free-flowing materials[5]. Since the slurry may vary in its flow characteristics due to varying water content, this transportation device must be designed to cope with all possible extreme cases concerning the slurry texture. Concerning the shape of the cutter edges, those should be designed in such a way to avoid build-up of the material. As for the chosen design with no cutter edge this means strike 2 according to TOS.
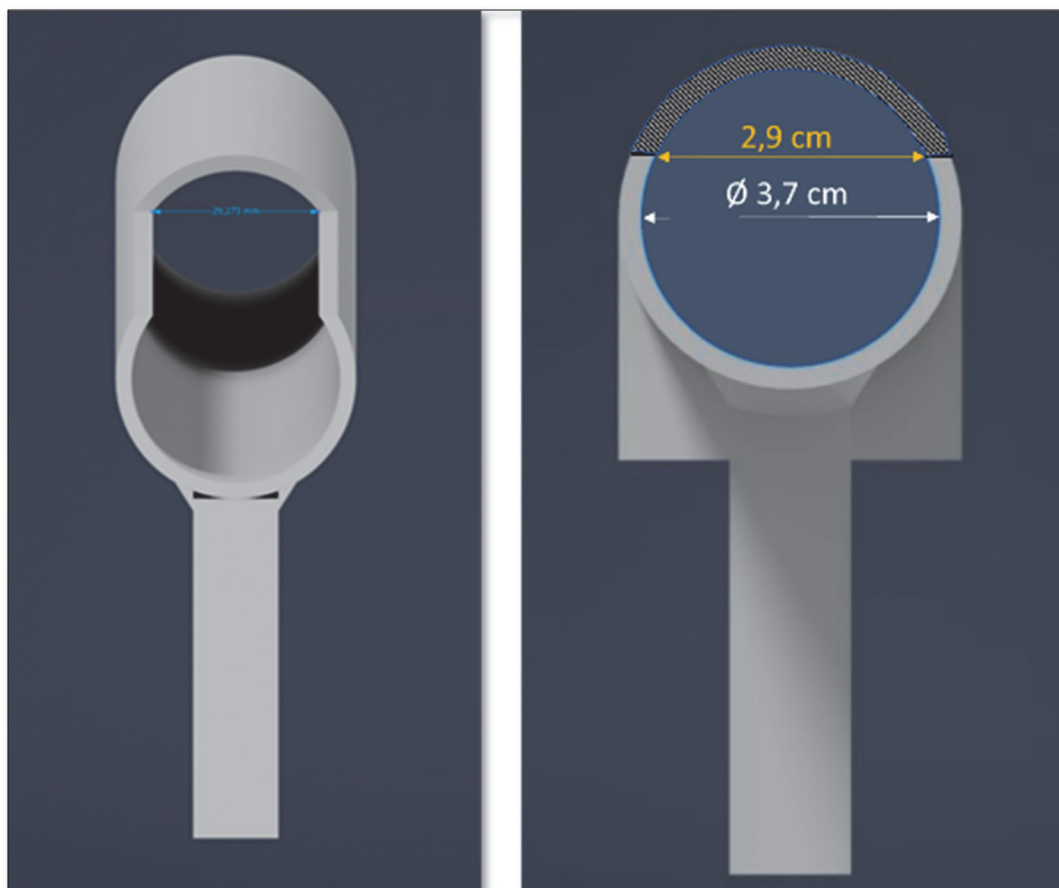

Figure 14. Illustrations of a section from the sampling collection tube to the left and cross section with measures to the right.

Increment preparation error

As for the IPE, it is of the utmost importance to preserve the increment's integrity when extracted from the mass stream. Contamination of the sample by equipment due to abrasion or corrosion or by contamination through the surroundings should be kept at an absolute minimum. As for the planned sampler - though the prototype will be built in stainless steel - the final design will be built out of titanium which is a much-used material at Nikkelverk withstanding corrosive process environments. Thereby contamination of the equipment itself is avoided, however one must keep in mind that the environment around the filter presses might contain contaminants. After all, it is Nickel which is produced at the plant.

Though the designed sampler has already shown several faults according to TOS, the planned extraction of the sample from the sampling tube should be examined with regards to IPE. A piston in the sampling tube is supposed to push the sample out of the cylindric formed tube and into a container. The sample extraction was theoretically thought to be viable, since the slurry contains approximately between 35 – 45wt% of water. Therefore, sliding the sample through the sampling tube seemed to be feasible. However, late field tests with a prototype showed that most of the slurry collected escaped the sampling tube and was lost before being pushed into the container. Loosing large parts of the increment during extraction will increase the IPE considerably. As follows, this is strike 3 for the chosen design. As for the moment, unfortunately no further sampling reduction after the primary sampling has been planned. This leaves the primary sample exposed to the idea of grab sampling – possibly strike 4.

Increment weighting error

Since emptying of the filter press frames can vary in time from frame to frame, the weight of the mass stream on the transport belt is not constant. For the planned sampler with a fixed position in the chute, this does represent a similar challenge as for a cross stream cutter. If the sampler would be built in a more traditional cross-stream cutter manner, care should be taken to avoid an unnecessary IWE originating from an uneven mass flow rate.

The correct sampling errors, like the fundamental sampling error (FSE), grouping & segregation error (GSE), process trend error (PTE) and process periodicity error (PPE)3, are not evaluated in the paper since the incorrect sampling error mentioned above should be minimized first.

Conclusion - Engineering versus TOS

The automatic sampler currently designed and planned is witness to a shift from manual to automated grab sampling still containing major faults according to TOS and not giving a representative sample as an optimum. Emphasis has been on reducing the workload on the operators and on improving the sampling procedure, and not on TOS-conforming sampling. The process team, planning the automatic sampler, evaluated several design possibilities and rated them according to four criteria: ease of construction, EHS-considerations, reliability & low maintenance as well as sample quality in reference to the manual grab sampling. The planned design fulfils the requirements of the first three criteria. Unfortunately, the lack of TOS awareness in the selected team caused them to focus mainly on the engineering aspects. This is a typical example and a common problem in all industries basing decisions on data originating from samples – physical or by sensors. A main reason for this is the lack of awareness on TOS and unsuccessful communication and knowledge sharing from the sampling community acquainted with TOS.

A traditional mindset in industries is to achieve their goals with the least necessary effort. A known expression is "achieving 80% effect with 20% effort". This type of philosophy promotes a quick response on occurring problems saving resources for other tasks. However, it will not make the sampling world a better place and contribute to a more sustainable future. Even though not all projects at a company can demand 100% effort from available resources, critical projects should aim for maximum impact with highest possible precision with regards to representative sampling. Engineers – like all others – should team-up with the other academic disciplines and keep in mind that equipment should not only work, but it should work correctly in all aspects, especially when it comes to representative sampling.

Learning curve – is it the whole story?

Sampling of filter cake is obviously a demanding task. During the study there were several discoveries which showed the complexity of the problem. For example, in the process step of filling the filter press with iron sludge and starting the cleaning cycle with water, channels can form in the filter cake. This phenomenon leads to poor washing of the iron sludge in certain frames and leaves water soluble Nickel components in the filter cake, increasing the amount of Nickel unnecessarily lost. Therefore, a proper sample from the filter press might reveal such process problems faster and save valuable material. Furthermore, the texture of the iron sludge to be sampled can vary a lot, due to several process parameters and therefore, as discussed in the previous section, the material flow on the conveyor belt is not constant. These facts need to be taken into consideration when revising the automatic sampler.

Since the conductivity of the iron slurry sampled is an easy measurement done by the operator in the field and a sufficient indicator if soluble Nickel components remain in the filter cake, there should be a correlation between these two variables. However, as for today, this correlation is not existing due to many parameters distorting the relationship – one being the current sampling procedure.

In summary one must appreciate the will and effort to understand and improve the sampling procedure of the iron sludge. It takes time to grasp all aspects of a complicated problem and ensure that the chosen solution will work properly. The experience gained in a "trial & error" fashion will give a much more long-lasting knowledge for the involved parties than merely stating the theories in meetings and expect them to be applied in the field. To allow testing of ideas, even though they are not TOS compliant, can be of value on a personal and company level. Since the presented automatic sampler has not yet been built in full scale, reconsideration of the design will be necessary to incorporate the principles of TOS.

As new improvement projects steadily occur, and representative sampling might be one of the main goals to achieve, the team working on the problem should neither approach the challenge from a purely engineering perspective or a TOS angle alone. The team should be a multi-faceted group with different academic disciplines to cope with the complexity of the problem as well as to

find an approach with is easy to maintain and understand. Since the knowledge on TOS in companies is far from widespread, finding resources with TOS knowledge is like finding a needle in a haystack. And even though large efforts are made to circulate this knowhow in a broader community, there are still few recipients compared to the many companies existing. A challenge is still to promote the benefits of applying TOS in an easy and comprehendible manner and thereby increasing the impact field of TOS-supporters and the sustainable gains thereof.

ORCID iDs
B. Elke Thisted: https://orcid.org/0000-0001-5798-5144
Eirik Djuve: https://orcid.org/0000-0002-3559-4625

References

1. K.H. Esbensen and C. Wagner, "Theory of sampling (TOS): the missing link before analysis", *Spectroscopy Europe* **26,** 19–21 (2014). https://www.spectroscopyeurope.com/sampling/theory-sampling-tos-missing-link-analysis

2. K.H. Esbensen and C. Wagner, "Spear sampling—a bane at all scales", *Spectroscopy Europe* **28/5**, 26–29 (2016). https://www.spectroscopyeurope.com/system/files/pdf/Sampling_Column_28-5.pdf

3. K.H. Esbensen, *Introduction to the Theory and Practice of Sampling*. IM Publications Open (2020). https://doi.org/10.1255/978-1-906715-29-8

4. F. Pitard, *Theory of Sampling and Sampling Practice*, 3rd ed., Boca Raton, FL: CRC Press, https://doi.org/10.1255/tosf.120

5. G.J. Lyman, *Theory and Practice of Particulate Sampling: An Engineering Approach*. Materials Sampling & Consulting Pty Ltd., 43-44 (2019), ISBN 978-164633382-0, https://doi.org/10.1255/tosf.121

6. https://mwwatermark.com/articles/what-is-a-filter-press/