

## Sampling in a precious metals refining plant – A practical approach for a complex problem

Bert Pauels

Umicore Precious Metals Refining, Hoboken (Belgium), E-mail: bert.pauels@umicore.com

**Umicore Precious Metals Refining is one of the world's largest precious metals recycling facilities. This plant recycles very diverse waste streams containing precious and other non-ferrous metals. More than 200 different types of raw materials are being processed here, varying from complex mining concentrates and refining residues coming from the mining industry and other smelters up to precious metals containing production scraps and end-of life materials such as electronic scrap and car catalysts. All these materials need to be sampled and analysed, of course, to determine the metal content (and so the value) of each individual lot. It is clear that heterogeneity rules in this process! Next to the obvious heterogeneity of the material itself, there is also a huge variety e.g. in lot size, in packaging, in physical appearance of the different materials, ... which is also to be taken into account during the sampling process. To take all these hurdles, the necessary resources are – unfortunately... – not unlimited. In this article we will gain more insight on how this complex problem is tackled in practice within Umicore, with respect for the Theory of Sampling. A couple of major specific sampling lines have been developed and designed in particular to be able to deal with the variety in physical appearance of the materials itself. Additionally, a large number of sampling procedures are made up in a modular way, to be able to cope with the necessary flexibility demanded for. Some examples will be illustrated and described more in detail, with specific attention to the most important influencing circumstances. Furthermore, the way the overall performance and quality of this whole process is supervised, will be explained. And finally, we'll discuss some special cases where “nothing seems to be what expected for”...**

### Umicore Precious Metals Refining

Precious metals have been used since the beginning of time for their beauty and nobility in jewellery, but today their technical properties are very important as well. Examples of these properties are firstly excellent electrical conductivity, therefore utilized in electronics, and secondly unique catalytic properties as used for e.g. in car catalysts or petrochemical catalysts. As global demand for these metals continues to rise, more low-quality ores are mined, leading to an overall decrease in average ore grade and a subsequent increase in energy use for the extraction of these metals. Thanks to their limited reactivity however, precious metals are endlessly recyclable, without loss of quality. Recycling these metals can be done using much less energy than is needed during primary production<sup>1</sup>. A major reason for this favourable energy balance is the relative richness of “ores” such as waste electronic scrap and spent automotive catalysts.

Since 1887 in Hoboken (Belgium), near the port of Antwerp, Umicore has had a long tradition in refining precious metals. In the last decades, the plant went through an extreme makeover, transitioning from a complex Pb-Cu-Ni concentrate smelter and refiner into one of the world's largest recycling facilities for precious metals. Due to its unique and innovative technology, UPMR is able to treat a wide range of complex precious metal bearing materials and recover 17 different metals efficiently, while applying world class environmental standards.

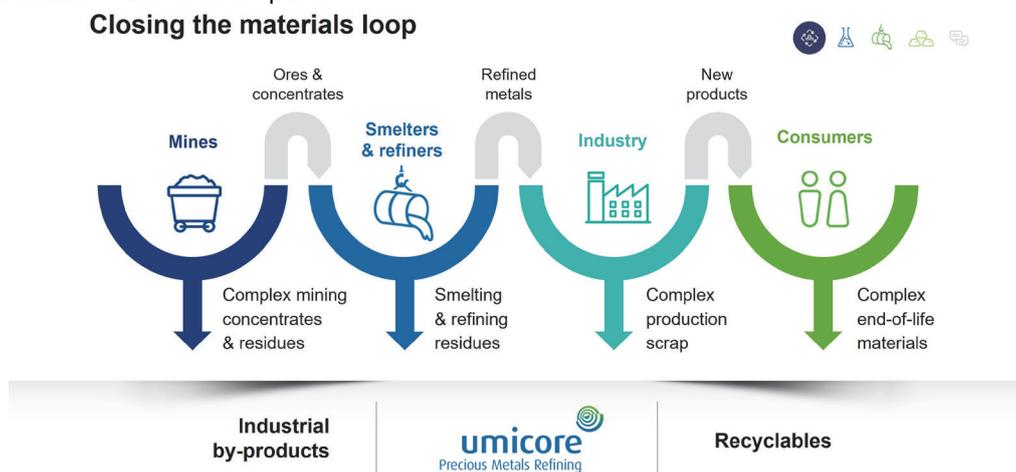


Figure 1. Umicore Precious Metals Refining, Hoboken – Belgium

## Incoming materials

UPMR is a world market leader in the assay-based recycling of complex waste streams containing precious and other non-ferrous metals. The plant offers eco-efficient smelting and refining services for precious metal bearing materials such as industrial by-products coming from other non-ferrous industries and, on the other hand, recyclables consisting of a large variety of end of life products containing precious metals.

Typically, the treated industrial by-products include: dross, precious metals-containing slag, matte, flue dust, hydrometallurgical residue, filter cake, tankhouse slimes and copper cement. Those products do not only come from other non-ferrous industries such as the copper, lead and zinc industry but may also come from other precious metals industries. Also complex concentrates (with a high degree of impurities) and by-products (such as gold on carbon or sludges) coming from mining operations are treated in the Hoboken plant.



**Figure 2. Overview of incoming materials**

Nowadays, precious metals are crucial, yet often invisible in our everyday life. They are key materials that enable consumer electronics to function, found in our mobile phones and computers. They also keep our air clean in automotive catalysts or support key industrial processes through industrial catalysts. As explained above, UPMR has evolved over time to efficiently recycle the precious metals coming of those end of life products from the consumer market, and is so turning the waste of yesterday into the materials of tomorrow. Typically, the recyclables treated in the plant are products such as electronic scrap (e-scrap), spent automotive catalysts, spent industrial catalysts and bottom ashes coming from incineration furnaces.

## Sampling and assaying

The wide variety and complexity of incoming materials mean that sampling and assaying are of crucial importance for reliable and efficient recycling. An accurate determination of the composition of the incoming materials is not only the base for the refining agreement with the customer (his final financial yield after all is based on the valuable metal content of the incoming material), but it also strengthens the knowledge about the exact composition of the feed, allowing the plant to tune its processes and to define optimal processing routes for every kind of feed material.

The full range of industrial by-products and recyclable materials are sampled on site at Hoboken, close by the operations. By means of an "à la carte" approach, each and every material gets its own dedicated sampling process, mostly in dedicated equipment. Often, these processes and technologies are in-house developed. This will be explained more in detail further below.

## Heterogeneity rules

As already described, a wide variety of materials arrives at the Hoboken plant. Yearly, about 8000 lots of more than 200 different types of raw materials are entering the plant, and need to be sampled before being processed in the smelter. Next to this enormous diversity of materials, there is of course also the obvious heterogeneity within each type of material. Both constitutional and distributional heterogeneity<sup>2,3</sup> play of course an important role in how the material is finally sampled. But there is a lot more which is influencing the final way of sampling.



**Figure 3. Diversity within incoming recyclables**

Heterogeneity is also very much observed within the variety of the intrinsic value of the incoming material. This value can vary from nearly 1 €/kg up to more than several thousands €/kg, depending of course on the kind and amount of precious metals the material contains. To reduce financial risks, both for the customer as for the refiner, lot sizes are also determined by this value, and will be limited to a certain threshold, mostly depending on the type of material, the yearly number of lots for this specific contract and/or the financial possibilities of the customer. Consequently, this lot size can vary as well very significantly. Fig. 4 shows this variation typically within different types of e-scrap. In general, the lot size can vary for 1 lot from about 50 kg up to more than 500 t of material!

**umicore**  
Precious Metals Refining

Printed circuit boards	10 t	
Cell phones & other small IT devices	2 t	
CPU, IC, connectors	1 t - 5 t	
Laptops without batteries & screen	10 t	
Shredded fractions with printed circuit boards	25 t - 100 t	

**Figure 4. Variability within incoming E-scrap**

Of course, this lot size equally has its impact on the way the incoming material is handled and shipped. Large lots are often being handled in bulk, and shipped in tipping trucks, containers or even ships, as the plant is located at the river Schelde which connects it to the port of Antwerp. Smaller lots are, in general, mostly packaged in sealed drums, boxes or big bags and are mostly being transported by means of trucks and containers. Due to more and more strict internationally environmental regulations, there is an increasing trend towards packaged goods, also for larger lots, which has of course its influence in the way of handling the material at unloading and may even influence the final way of sampling.

Last but not least, the physical appearance of the incoming materials can be just about anything: dry lumpy material, sludges, filter cakes, metallic scrap, plastic boards, dry powders, ... and anything in between. As we are dealing with precious metals containing materials, a very important feature to determine in most cases for all those materials is the moisture content, ranging all the way from zero to sometimes up to ninety-five percent! Even some types of e-scrap can contain an important amount of water (even ~20%), depending on the way they have been pre-processed before being shipped.

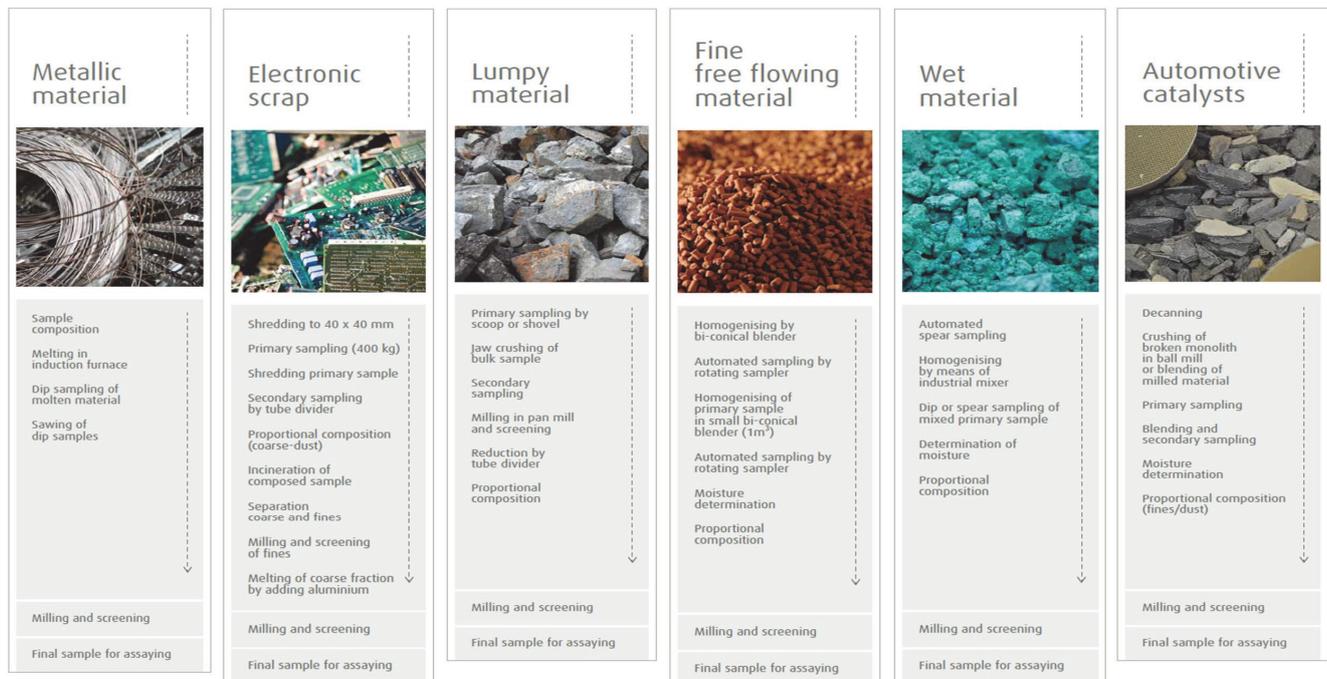
### Sampling at UPMR

As already described above, because of the very diverse and complex incoming materials, sampling and assaying are a key success factor in determining the exact metal content and, as a consequence, the final financial value of each lot. Therefore, UPMR allocates a substantial amount of its operating budget to the sampling and assaying services. At present, about 230 people are working within the sampling department, and about 100 in the laboratory. Yearly, next to the 8000 incoming lots, more than 2000 internal lots (mostly intermediates) are equally being sampled in these facilities. In the laboratory, about 70000 samples are analysed on a yearly base, including all the necessary samples for the follow-up of all the refining and production activities in the plant.

Most of the sampling and assaying processes and technologies are in-house developed, and over the years, TOS also came more and more into the spotlight next to the use of state-of-the art technology. With a mindset on automation, efficiency

and fast throughput, human intervention and sampling risks are minimized. Based mainly on the physical characteristics of the material to be sampled, UPMR has set-up a number of main sampling lines, each of them especially suited for a specific type of material. In this way, dedicated sampling lines have been developed to deal specifically with following type of materials, some of these lines will be described more in detail below:

- Electronic scrap
- Metallic scrap
- Lumpy/rocky materials
- Fine, free flowing materials
- Wet/sticky materials
- Automotive catalysts



**Figure 5. Dedicated Sampling Lines @UPMR**

### Evolution over time towards TOS principles

In the past 2 decades, the sampling department got a total make-over and further automation was introduced where possible, respecting the TOS principles on the one hand but focusing on greater efficiency and shorter lead times on the other hand. E-scrap nowadays is treated on a big shredder line, including a wind-shifter to remove possible damaging out-of-spec heavy (mostly metallic) pieces, a 2-axis pre-shredder followed by the 4-axis final shredder, both slow rotating shredders to minimize dust formation. The scrap is shredded at a flow rate of up to 10t/h into pieces of 40 mm before going to a 2-stage stream-sampling station, a Vezin-sampler followed by a rotary tube divider, resulting in a primary sample of 400kg. At least 1000 increments are guaranteed in this sampling stage to compose the primary sample. In the early days, e-scrap used to be sort out manually to compose a primary sample which was then further burnt in a furnace for further sample preparation. Both the way of sampling as its environmental impact have been significantly improved by installing these new lines!

Concerning lumpy/rocky materials, the sampling department used to have a jaw crusher with a static (!) sampler until about only 10 years ago. At present, these products are treated now in a 2-stage jaw crusher line, where the particle size is reduced down to 20 mm and where afterwards the primary sample is taken by means of an automated cross-cut stream sampler. Afterwards, this sample is further milled in an automated pan mill-line, mostly in 2-3 stages down to a particle size of < 3 mm. The volume of the sample is reduced after each stage of milling by means of a rotary tube divider. Only 10 years earlier, this sample size reduction still used to be done by means of coning & quartering...



**Figure 6. Pan Mill – Before: Manual screening followed by coning a quartering – After: automated sampling after milling with rotary tube divider**

Another example of this TOS increasing insight has been introduced for the sampling of automotive catalysts. Though a first automation line had been installed, the analysis results on the final samples were not robust enough and also led to quite some complaints. On this old line, the catalyst material was crushed to a size  $< 7$  mm and then sampled by means of a double rotary tube divider into a sample of a few kg, which was further prepared in the final preparation area into the final lab samples. But due to the very special nature of this material, where a very thin high value precious metals containing layer is coated on to a carrier substrate, this material – especially after crushing – is very heterogeneous: especially the dust fraction will contain preferentially substantially more precious metals than the larger particles. For this reason, the size reduction of the catalyst material is nowadays done by means of a ball mill and also milled to a much smaller particle size  $< 1$  mm. The milled material is then sampled by means of a rotary tube divider, and also all dust which is generated before this sampling step is collected separately in a dust filter. It will make a separate fraction of the sample, to be taken into account proportionally. But it was not only the constitutional heterogeneity which had a strong influence on the way of sampling – also the distributional heterogeneity played a significant role in the older days. As it is a recycling business, the feed of the installation can be very diverse: so the amount of precious metals used for the coating of the catalyst converter, can differ very widely from type to type. This resulted often in a somewhat layered primary sample, leading to inaccurate end samples.. So it was also important to introduce a mixing step for the primary sample, which is done by mixing it in a Nauta type vertical cone screw blender before being sampled through a rotary tube divider. The speed of this rotary tube dividers has been also reduced to avoid the possible impact of turbulence on the dust fraction<sup>3</sup>. The dust generated in this sampling step before the sampler is likewise collected separately and will be also taken into account proportionally to the end sample.



**Figure 7. Nauta type vertical cone screw blender for automotive catalysts**

### Other influencing factors

But of course, there is always an economic trade off to be made: resources are - unfortunately - not unlimited! Therefore, the way of sampling is not only determined by the material characteristics, however, there are also other important influencing factors. As explained already before, lot size will be one of these other determining factors. Smaller lots do have mostly a higher value, and will be unloaded directly into the secured sampling department. Mostly they will be sampled completely, for 100%, directly on the most suited sampling line. On the contrary, the larger lots have a lower intrinsic value and will be mostly unloaded outside of the sampling department. For these large volume lots, a primary sample will be taken at first, often in a more manual way, e.g. by means of spear sampling the incoming big bags, or by taking increments with a sample shovel from each unloaded truck. The number of spears/shovels is strongly determined on the intrinsic value of the material: the higher the value of the material, the more increments are being taken. This results more or less in a relative stable amount of primary sample as the lot value is inversely proportional to the lot volume. When these large volume lots do contain more lumpy and/or rocky material the primary sample will be taken during the transfer of the lot with a payloader by collecting half a scoop as a 10 % primary sample at each 5<sup>th</sup> scoop taken during the displacement of the material. All payloaders at UPMR are therefore equipped with a dynamic weighing system to be able to control this process: the drivers are even guided by an onboard IT system which ensures that the driver will perform this way of sampling in an accurate way. On the one hand, this 'payloader' technique is used because it is impossible to sample this type of material in a representative way with a shovel, because of the larger particle size of most of the material, and on the other hand – in such cases – it would be too expensive for these larger lot volumes to crush the material for 100%. This will only be done for high grade, often also heterogeneous materials, or also when this is required for the further refining of the material in the plant. In all of the above described cases, the collected primary samples will afterwards be brought to the sampling department where they then will be further processed on the dedicated sampling lines.

This way of sampling is revealing also another important influencing factor: the cost of sampling. One could also sample the lot for 100% on the dedicated sampling lines, but then it would ask too much time (and so also the overall sampling cost would increase significantly). Another consequence is that in that case the overall lead times for sampling would increase significantly, and would also have an impact on other lots, waiting for sampling. As a company, a trade-off is then to be made to either invest in extra equipment, or to simplify somewhat the way of sampling, depending of course on the financial risks this simplification can imply. Sampling overall lead times need to be under control, as the customer is awaiting the final outcome of the sampling process, the analysis, to get paid for his material delivered.

A nice example of this trade off can be found in the way UPMR organised its e-scrap sampling. For larger lots (mostly >30 t for e-scrap, or > 1 truck load), it is contractually demanded that the material should already be pre-shredded. As this material is mostly low grade material, a primary sample will be taken after the unloading of the material. For very low grade e-scrap, this will be done directly to 400 kg, for more valuable material a 10% primary sample will be collected. Smaller lots (more valuable material, or medium/high grade e-scrap) will be mostly packaged and discharged entirely within the sampling department. These lots will be treated for 100 % on the dedicated sampling lines. On these lines, a primary sample of 400 kg will be collected as well. This means that, regardless of the value of the lot, the size of the primary sample will be always 400 kg. This is a setting the operator needs to adjust, each time when starting a new lot on the sampling line. But as the value of the material is inversely proportional to the lot size, the size of the primary sample will be relatively more important for smaller, high value lots. The reason why the primary sample needs to be 400 kg is because of the standardisation of the following sampling step, where the 400 kg sample is further shredded down to < 7mm. After this shredding step, a secondary sample of 4 kg is then taken by means of a rotary tube divider. This sample will be adjusted proportionally with the during this processing step generated dust. Further preparation of this sample is then done by burning this sample, screening the ashes on 75 $\mu$ . The fines are then blended and distributed, packaged and labelled into a first sample fraction, whereas the coarse fraction is melted in an electrical furnace by adding aluminum. The result of this melt is a kind of brittle, crystalline metallic ingot, which can be grinded and milled down to 180 $\mu$  to result in a second sample fraction for the original e-scrap lot.



- Shredding to 40 x 40 mm
- Primary sampling (400 kg)
- Shredding Primary sample to 7 x 7 mm
- Secondary sampling by rotary tube divider
- Proportional composition coarse sample with dust
- Incineration of composed sample
- Separation coarse and fines
- Milling and screening of fines (1)
- Melting of coarse fraction by adding Aluminum
- Milling and screening (2)
- Final samples (1) + (2) for assaying



**Figure 8. Sampling of e-scrap @UPMR**

Standardising those secondary sampling steps, has the advantage to be able to have more or less fix lead times for this part of the sampling process of e-scrap. In case everything would be organised based proportionally on the incoming volume of material, too much time would be spent on dealing with the lower grade materials, where the financial risks are in any

case (much) lower than for high grade materials. Another advantage is that quite a lot of the final steps in the final preparation could be automated, and are nowadays performed automatically: a robot distributes the samples from one handling station to another, and at the end the final samples are automatically bagged and labelled. By introducing this automated installation, an increased robustness could be reached: the focus here is on achieving maximal accuracy and repeatability by minimizing the human interaction, and of course on reducing the throughput times.



**Figure 9. Automated final preparation @UPMR**

### Modular Sampling Procedures

As the e-scrap example shows, it is not only the material characteristics on themselves which will influence the final way of sampling the material. Of course, they remain the basic foundation for the sampling method to be followed, but there is also a substantial need for increased flexibility, taking into account financial risks and costs and focusing on efficiency and shorter lead times. Therefore, starting from the basic dedicated sampling lines as shown in Fig. 5, UPMR has set-up a modular system of sampling procedures to cope as much as possible with all those sometimes somewhat contradicting needs.

Today, about three hundred different sampling procedures are used. These procedures are, in fact, partial procedures for a certain sampling step (or sub-procedures), mostly related to the installation being used for the specific sampling step and the nature of the material being dealt with. They are subdivided into three major categories:

- Primary sampling procedures (Pxx)
- Secondary sampling procedures (Gyyy)
- Final sample preparation procedures (Fzzzz)

With these three categories of sub-procedures, a number of combinations can be made (if justified, of course), to obtain a specific full procedure (Pxx.Gyyy.Fzzzz) by which means a material will be sampled. On top of that, customer-specific demands can be satisfied by an “à la carte” approach, in which the sampling procedure is – within certain (contractual) limits – developed further in close collaboration with the supplier of a material.

### Sampling Process @UPMR

Keeping all this in mind, the sampling of incoming materials starts in fact already at the moment of the signing of the contract with the customer. Here, packaging requirements are discussed, as certain sampling installations can have constraints on maximum dimensions or weights which can be treated, but also to facilitate the logistics for unloading and safe handling of the materials. Material characteristics will be checked with the sampling experts, and, in general, the most appropriate standard combination of sub-procedures is chosen as the sampling procedure and linked to the contract.

Subsequently, at arrival each incoming lot is controlled and inspected directly after unloading to control if the shipment is complete as indicated by the customer. The next check is on the physical appearance of the material and is mostly documented with pictures to address the following concerns: Is the material really as stated in the contract? Is the initially foreseen sampling procedure suited for the material being delivered? If not, the procedure may be slightly adapted (often in close collaboration with the customer or his representative on site). In other cases, a non-conformity report will be created, pointing out all the issues to the customer and proposing solutions. Meanwhile, the material will be blocked for sampling and further treatment until an agreement has been reached with the customer on how to proceed.

Once the material has been inspected and the sampling procedure has been validated, the material is released for the actual sampling. A primary sample is taken, which is then further treated on one of the many sampling lines. All relevant weighing information is registered. During this treatment, samples are generated, collected and brought to the final preparation area. Samples are then further prepared, packed and labelled and will be sent to UPMR’s laboratory (and the customer) for detailed analysis. A weighing and sampling report is made up, containing all relevant sampling information about the lot at hand, including: net wet weight delivered, moisture content, description and composition of the different samples/sample fractions.

## Quality assurance

Due to the multitude of products and procedures on the one hand, and the financial risks on the other hand, it is obvious that the quality of the sampling process is crucial for the success and continuity of the company. The overall quality is therefore continuously monitored and supervised, through internal and external, independent assessments, often also in close collaboration with the customers.

As explained above, the first monitoring step takes place at the start of the contract, where the material characteristics are discussed as well as the logistical preconditions for the material to be sent. At arrival, the material is then checked in detail to confirm ultimately the exact way of sampling, sometimes after having made the necessary adjustments in agreement with the customer. For some type of products (e.g. e-scrap), a reserve (primary) sample will always be put aside until the whole sampling process has ended, or sometimes even until final analysis is known. This is done likewise for materials, where financial risks are estimated to be high, i.e. mostly for very heterogeneous materials. These reserve samples can sometimes be used already during the first sampling process, when one discovers further in the sampling process that the chosen way of sampling is not the best way to obtain an optimal result. The reserve sample will then be started up, and can – in agreement with the customer - even be sampled according to an adjusted sampling procedure which fits better for the material at hand. Also in case of a mistake or any other problem somewhere in the sampling process, the reserve sample will function as a kind of insurance for both parties, customer and refiner.

## Monitoring analysis results

Samples are sent to the laboratory and to the customer for analysis. These analysis results are monitored for each type of product and contract, and where possible, compared with historic data and/or pre-shipment analysis coming from the customer. If anything unusual is detected, the laboratory will question this to the sampling department. In some cases, it can also be the customer who is questioning the final analysis results. In the sampling department, all the necessary information to these questions is then gathered and examined further in detail. In most cases, the answer can be found in deviating material characteristics which have been detected and documented inside of the sampling department. In some cases it is not directly clear, what the reason of deviation could be. In such cases, and when there is still a reserve sample available, this will then also be sampled to recheck the final analysis results. Yearly there are more than 150 of these questions to be examined, resulting in about 10% of extra rechecks to be done. In some cases, the customer will not agree with the final analysis results, and can request – under certain conditions – for an official resampling of the material (starting from the reserve primary sample). In such cases, further agreements are made upfront in detail with the customer how to cope with the final result after the resampling.

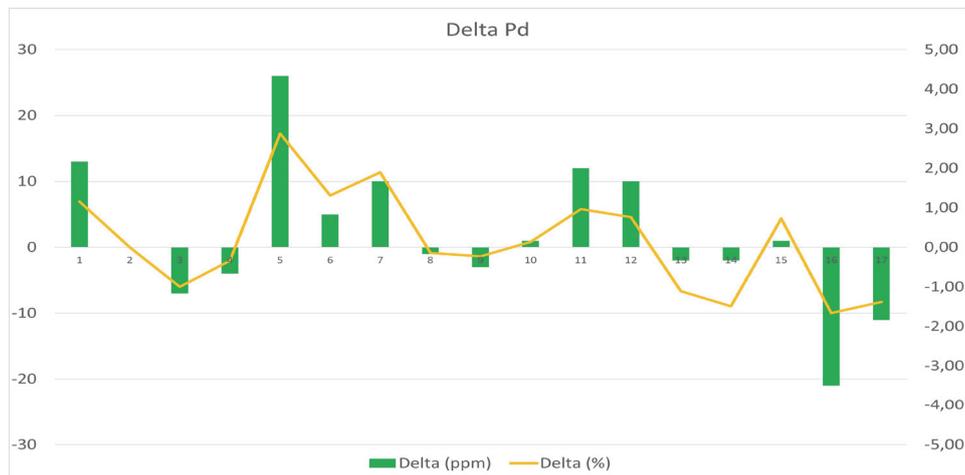
## Quality control programme

Next to these rechecks "on demand", the sampling department carries out on a yearly base an intensive quality control programme. In this programme, about 50 lots are sampled in double to follow up the performance on the major sampling lines. Where possible, the total lot will be resampled a second time, after the official sampling. For e-scrap, the reserve 400 kg primary sample will be further sampled to compare the analysis results with the first official sampling. All results of the first and second sampling are then compared with each other. The differences should be between certain limits, depending on the type of material and the sampling procedure that was followed.



**Figure 10. Quality Control Programme 2021 Shredder 3 – Au result**

This type of control is also used when new equipment is installed, or new sampling procedures are introduced. Lots will be sampled on a well-known sampling line, or according to a well-known sampling procedure, and will then be 100% resampled on the new sampling line, or according to the new procedure proposed. After final analysis, the results of the lots sampled on the new sampling line or following the new sampling procedure are compared with the results of the sampling the same lots on the well-known sampling line or according to the well-known sampling procedure. The results for the new line or method should fall within the same accuracy ranges as the results after sampling on the well-known lines or following the usual sampling procedure. Fig. 11 shows the test results for milled automotive catalysts being sampled according to a new continuous way vs. the well-known batch-wise approach.



**Figure 11. Comparison continuous sampling vs. batchwise sampling of milled automotive catalysts – Pd results**

### Inventory process

Sampling also plays a crucial role in the plant's inventory process. Due to the large amount of precious metals on site, it is very important that the stock at inventory time is determined in an accurate way. Not only do weights need to be registered correctly, but also moisture content and composition of the materials (often intermediates) need to be determined in a representative way. At UPMR, a detailed inventory is carried out twice a year.

In this exercise the plant checks the evolution of the stocks of metals over a 6 months period: the metals stock from 6 months ago, increased by the incoming metals over 6 months and reduced by the outgoing metals in the same 6 months should be equal to the metals stock actually on site. At these moments, the sampling department needs to sample > 300 lots of intermediate materials to determine their metals content. The final outcome of this exercise needs of course to be between narrow ranges, depending on the type of metal, to ensure the financial health of the company.

### Some special cases...

Despite the fact everything seems to be prepared upfront in detail – at least on paper, reality however often shows a large discrepancy from what was promised before. This can really be about anything you could imagine, which is illustrated by underneath cases. As you will see, in those cases “nothing seems to be what it was expected for”...

#### Filter cake

During the check of the physical appearance, typically a few packages per lot are opened, to verify the material inside. For this shipment, 9 containers with 214 pallets each containing 4 drums, 18 lots had to be sampled. After opening some of the drums, it appeared that some of these drums did not only contain filter cake, but were covered under a layer of white sand. Apparently (after questioning the customer), for transport reasons this material needed to be covered with this sand. Of course, this was never discussed before...

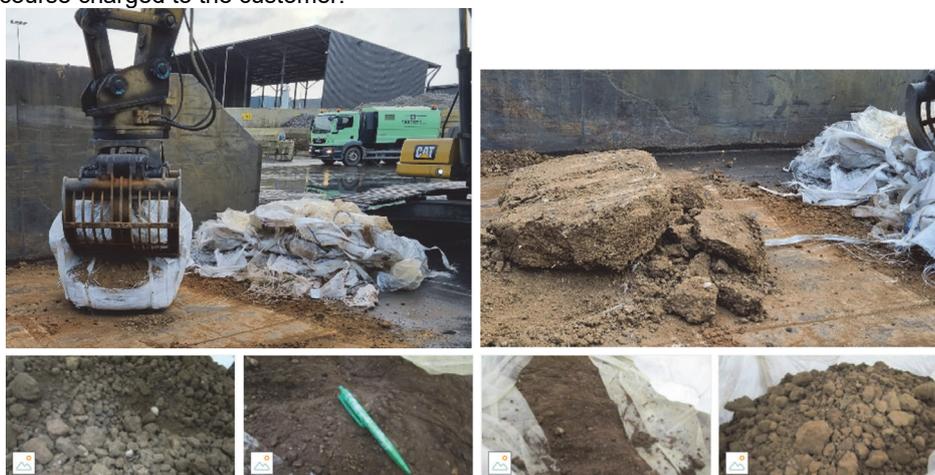
The normal foreseen sampling procedure for this filter cake was: spear sampling each drum five times, and then mixing the primary sample in the mixing installation. A trial for 100% mixing was done on 1 lot, but it appeared to be very difficult to nicely homogenise the sand together with the filter cake. Next to this fact, also the throughput time would be very high to treat all this material for 100%. It was then decided to screen the whole shipment by means of a vibrating 10 mm screen. The oversize material was collected per lot into big bags, and could then be spear sampled; the undersize was treated as 1 extra single lot and was manually primarily sampled during transfer by means of a sampling shovel.



**Figure 12. Filter Cake with white sand – Mixing result**

### Ag-Au concentrate

For this shipment, 7 containers each containing 22 big bags, had to be spear sampled as 1 lot, 5 spears per big bag. But, the material was to the large extent hardened, and couldn't be spear sampled at all. Therefore, an alternative sampling method imposed itself. Due to the nature of the product, unloading of the big bags needed to be performed by means of a grab crane. The material needed to be broken into pieces and more or less pulverised by the crane before further manual primary sampling was possible by means of a sampling shovel. Moisture samples were separately collected per container, and further reduced through grid sampling, moisture content was subsequently determined in double for each container. The primary sample was afterwards treated in the pan mill for further sample preparation. All the extra costs for these activities were of course charged to the customer.



**Figure 13. Ag-Au Concentrate – Discharge with crane**

### Ashes

This material is coming very regularly from another refining plant, and is also sampled at their premises. But, when the final outcome of the analysis was compared, there were from time to time large discrepancies between the analysis based on the sampling Hoboken compared with the initial analysis found at the other plant. So, an extensive investigation was started, both at the supplier's side as at the refiner's side.

The material was prepared by the supplier to a particle size  $< 300\mu$ . By auditing each other's processes, it was soon detected that the primary sampling at the supplier's side was done by means of a screw sampler in the outgoing stream of a blender! So this sampler was only covering part of the outgoing stream of material after blending, which conflicts with the Fundamental Sampling Principle (FSP = "All potential increments of a lot shall have identical non-zero probability and practical possibility to end up as the physically extracted increment"<sup>2,3,4</sup>). So, especially with heterogeneous materials, this was clearly the reason behind the sometimes widely diverging analysis results.

But as it was an extensive exercise, also some extra reserve samples were taken during sampling at UPMR, and analysed accordingly. For some materials, however, a far larger spread in results was found also between these similar samples as normal for the sampling line used. That's why the final samples were screened on  $300\mu$ , before being further prepared to laboratory samples. And yes, it appeared that more than 2% of the material was a  $>300\mu$  coarse metallic fraction, which contained 5 times more precious metals as the fines fraction! So this material was definitely not the same as in most other

cases. After further investigation at the supplier's side, this material turned up to be ashes containing also crushed refractory brick material. So, in this case also the constitutional heterogeneity of the material played an important role for some lots: to cope with this phenomenon, the final sampling stage of this lot has been adapted and includes now a grinding step where the total sample needs to be grinded  $< 300\mu$  before being further reduced.

Ashes - Screw sampler



Ashes - Fines



Coarse fraction



**Figure 14. Screw sampler – Coarse fraction within ashes**

## Conclusion

Sampling in a precious metals refining company can be very diversified (and challenging!) as a consequence of the very different and often complex materials that are dealt with. As sampling is a key success factor for the sustainable recycling of precious metals, it deserves significant attention. It is however not only influenced by TOS, there is always an economic trade-off to be made to which extent these rules prevail to be able to reduce on the one hand the financial risks for both the customer as well as the refiner, and on the other hand to guarantee high efficiency and short lead times. UPMR's vision in this matter illustrates this: Master Complexity, Foster Flexibility and Ensure Reliability<sup>1</sup>.

## ORCID iDs

Bert Pauels: <https://orcid.org/0000-0002-2815-3688>

## References

1. F. Vanbellen & M. Chintinne, "Extreme makeover: UPMR's Hoboken plant", *Proceedings of EMC 2007 – Volume 1, GDMB*, pp. 371-380 (2007). ISBN 978-3-940276-04-9
2. 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>
3. F.F. Pitard, "The Theory of Sampling and Sampling Practice, Third Edition", *CRC Press*, (2019). <https://doi.org/10.1201/9781351105934>
4. DS 3077, "Representative sampling—Horizontal Standard", *Danish Standards* (2013). <http://www.ds.dk>