

# If Mahomet Won't Come to the Mountain, the Mountain Must Come to Mahomet: Transforming Sampling and Preparation Services for Circular Economy Materials through a Specialised TOS Compliant Mechanical Sampling Hub

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## 1. Introduction

This paper demonstrates the transformative potential of a specialised mechanical sampling hub concept for offering the highest accuracy, precision, and robustness of sampling and sample preparation services in the realm of circular economy materials, with a specific focus on Incinerator Bottom Ash (IBA). Capitalizing on the Theory of Sampling (TOS), Alfred H Knight (AHK) has inaugurated a cutting-edge sampling hub in the Netherlands. This innovation confers significant economic, operational, and trust advantages to a broad spectrum of stakeholders, including IBA suppliers and copper smelters. By reversing the traditional third-party inspection model, i.e., by transporting the material to a specialized facility instead of sending an inspector to the site, the sampling hub approach secures reliable representativity by eliminating incorrect sampling errors, while reducing correct sampling errors to acceptable levels for the parties of the trade. This revolutionary, independent third-party sampling hub approach not only speeds up payment cycles and reduces the technical risk of being wrong, but also minimizes the environmental impact of material transportation and metal production relative to virgin metal production, thus aligning with UN World Development Goals numbers 9 and 12.

## ABSTRACT

Incinerator Bottom Ash (IBA) is a by-product formed at the base of waste incineration furnaces during the combustion of waste materials, e.g. municipal solid waste. Comprised primarily of metal, glass, and mineral species, the Heavy Non-Ferrous fraction of IBA (HNF) is especially rich in valuable metal concentrations of copper, gold, silver, platinum, and palladium. Serving as an optimal feedstock for metal smelters, this IBA fraction presents distinct environmental advantages as secondary, circular metals requires fewer production steps and reduced energy consumption compared to their virgin counterparts derived from natural ore and mineral concentrates. Because of its intrinsic high value, accurate and representative sampling and testing of IBA is essential for equitable commercial transactions. The highly heterogeneous nature of IBA presents complex sampling, testing, and analysis challenges, which require strict adherence to sampling approaches in complete compliance with the Theory of Sampling (TOS).

This paper outlines the technical foundations of the mechanical sampling system that has been established and implemented by AHK in the Netherlands. This new approach introduces the innovative sample preparation technique of homogenization through melting of a large bulk sample of IBA, elucidates the associated quality control mechanisms to establish system robustness, and highlights its many multi-stakeholder advantages. In doing so, it hopes to lay a compelling case for broader adoption of this approach in the global circular economy industry elsewhere in the world and for other metal recovery products as well, all contributing to responsible and sustainable sourcing of critical metals for electrification and battery technologies.

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## 2. Urban Mining

Municipal Solid Waste (MSW) is mainly comprised by household refuse or similar waste appropriate for incineration. The incineration process converts waste to energy through combustion. Incineration Bottom Ash (IBA) is the primary solid byproduct of this process, accounting for approximately 80% of all incineration residues by weight (Chimenos et al., 1999). The global production of IBA is substantial, particularly in Europe (Dou et al., 2017; CEWEP, 2023). For each tonne of MSW incinerated, approximately 150–250 kg of IBA is produced (Hyks and Hjelmar, 2018). In 2021, there are over 800 waste incinerators worldwide and over 400 in Europe. MSW incineration plants in the European Union process around 62 million tonnes of MSW annually, yielding about 12 million tonnes of untreated IBA, which represents 26% of the annual incinerated waste by mass (Eurostat, 2023).

Municipal Solid Waste Incineration (MSWI) serves not only to harness the energy content of waste, but increasingly also facilitates the recovery of various valuable components. This makes MSWI an integral component of the circular economy (Van Caneghem et al., 2019; Pan et al., 2015; Malinauskaite et al., 2017). The composition of untreated IBA varies according to the MSW feedstock, combustion technology, and operational conditions at the incineration facilities. IBA is a highly heterogeneous material primarily comprised by complex inorganic mixtures of melted products, minerals, metallic compounds, ceramics, and glass. Specifically, the mineral fraction constitutes 80–85% of the bulk mass of untreated IBA, while the remaining 10–12% is made up of combined ferrous (Fe) and non-ferrous (NF) metals.

Specifically, attention will here be centered on the fraction consisting of heavy and non-ferrous metals, designated as the Incinerator Bottom Ash Heavy Non-Ferrous Metals Fraction (IBA-HNF). Serving as an alternative feedstock for copper smelters and precious metals refining plants, IBA-HNF can substitute for mineral concentrates and even eliminate certain production steps otherwise necessary for generating virgin metal from mining resources. Recovery rates stand at approximately 90% for copper, 70% for silver, and 80% for gold contained in untreated IBA. Beyond the direct energy efficiencies in the production process, there are also substantial transport-related savings, especially pertinent when considering that copper ores are primarily sourced and concentrated in distant regions or countries beyond, for example, Europe.

## 3. Sampling Hub The Netherlands

The growing understanding of the value and potential associated with the product, IBA-HNF, clearly highlights the need for precise quantification and qualification. This is essential for transferring ownership and settling financial transactions. Multiple stakeholders participate in the process from Municipal Solid Waste Incineration to the smelting and refining of metals such as copper, gold, silver, platinum, and palladium. Like traditional commodities, IBA-HNF is bought and sold based on its verified mass and elemental composition—copper, gold, silver, platinum, and palladium—determined through critical sampling and analysis.

To ensure commercial settlements between IBA-HNF producers and receiving copper smelters are accurate, this circular economy industry needs representative samples. However, the representativeness of an individual sample is not discernible from the sample's own characteristics. Instead, the focus must be on specifying the qualities a sampling process must have to be considered representative. According to TOS, a representative sample results from a representative sampling process. Therefore, a sample is either representative or not; an unrepresentative process can only produce 'specimens' with an unknown provenance, making them unsuitable and unreliable for analysis and crucial decision-making in various sectors.

For a sampling process to be qualified as representative, active steps must be taken to eliminate or minimize both bias and precision. While most sampling standards and their respective sections focus primarily on procedures to minimize total effective precision, it is crucial that efforts to eliminate sampling process bias are not overlooked (DS3077, 2013; Esbensen, 2020; Pirtard, 2019; Lyman 2020). In fact, guidelines often emphasize the importance of eliminating bias, but often lack specific procedures to accomplish this task. Failure to comply with these essential steps, whether intentionally or inadvertently, constitutes a breach of due diligence in designing, preparing for, and executing a documentable, representative sampling process.

This sets the stage for the introduction of the solution for weighing, sampling, sample preparation, and testing that Alfred H. Knight has developed for IBA-HNF in the Netherlands; this is termed the Sampling Hub.

#### 4. If Mahomet Won't Come to the Mountain, the Mountain Must Come to Mahomet

Focusing first on the relationship with the elimination of sampling process bias, it is important to emphasize the persistent heterogeneity in Municipal Solid Waste (MSW) and the resulting untreated Incineration Bottom Ash (IBA).



**Fig. 1:** Typical heterogeneity manifestation of a 25-tonne lot shipment of IBA-HNF. Note how larger particles accumulate at the bottom and smaller particles remain at the top due to segregation during stockpile build-up.

This heterogeneity remains largely unmitigated even after minerals and metals have been separated and concentrated in IBA-HNF. Typically, individual traded shipments are around 25 tonnes, but it is important to note that these smaller tonnage shipments are actually composites, blending IBA-HNF streams originating from different concentration and beneficiation stages and from different Municipal Solid Waste Incinerators (MSWI) across Europe. There is thus no 'typical' IBA-HNF material.

Although IBA-HNF constitutes only 1-4% of untreated IBA by mass, this concentrated product on closer examination reveals phenomena such as grouping, segregation, and nugget effects, which are common in stockpiles of solid bulk particulate materials. The nominal top size of IBA-HNF is 19 mm, and with a moisture content of less than 3%, the material is mostly free-flowing. However, the presence of non-ferrous metals from cables and wires may cause material aggregation, much like yarn forming a ball. Furthermore, during stockpiling operations, larger particles tend to concentrate at the bottom and smaller particles rise to the top (Figure 1).



**Fig. 2:** Close-up photo depicting free gold particle from IBA-HNF material attesting to highly significant heterogeneity at particular scales.

Considering the variations in the composition of distinct particle sizes and the evident nugget effect in cases like gold particles (Figure 2), it becomes clear that manual sampling of a three-dimensional lot (3D) will fail to meet the core principle of The Theory of Sampling (Gy, 1979). Specifically, this framework advocates that “For precision and accuracy, it is essential that increments (or cuts) are extracted in such a way that all particles from the lot have the same probability of being selected and becoming part of the final sample for testing, irrespective of their shape, size, mass or density.”

Previously, sampling of IBA-HNF shipments for copper smelters and refiners in Europe was carried out using the following traditional techniques:

1. Utilization of an excavator or wheel loader for mechanical *quartering-and-coning* of the entire 25-tonne lot, followed by the extraction of a primary sample using a bucket excavator. A sub-sample was then acquired through *manual shoveling* for further preparation and analysis.
2. Mechanical increment sampling was executed by driving a shovel attached to a wheel loader into various positions around the circumference of the 25-tonne 3D stockpile. The sample mass was subsequently reduced via mechanical *quartering-and-coning* using a mini bucket excavator.
3. The 25-tonne 3D stockpile was reshaped into a flat, rectangular surface of uniform thickness. The rectangle was divided, often into a 4 x 5 grid, and increments were extracted using a sided sampling shovel, as frequently depicted in certain ISO standards. This method is akin to increment division or the Japanese slab cake division technique, but write large.
4. A flap sampler was used to divert a stream of IBA-HNF to obtain a sample.

While methods 1–3 offer the practical advantage of allowing the sampler to approach the stockpile directly and perform in situ sampling, their inability to eliminate sampling bias undermines their reliability (Gy, 1979; Esbensen, 2019). Flap gate samplers, as outlined in method 4, act essentially as single-edge cutters and are inherently flawed in terms of increment delimitation, with no viable options for bias mitigation (*ibid*; Pitard, 2020). Such biased sampling techniques produce only ‘specimens,’ rendering them fundamentally unsuitable for commercial analysis. Therefore, it should not be surprising that these approaches have led to many analytical and valuation discrepancies between the producer and the receiver of IBA-HNF, inevitably resulting in frustrations, financial settlement delays, and a loss of trust among the trading parties.

In a newly devised approach, Alfred H Knight was tasked with establishing a centralized sampling hub situated strategically within the logistical supply chain between the IBA-HNF supplier and receiver. At this designated facility, each 25-tonne lot is subjected to weighing, sampling, and final sample preparation for subsequent analysis. The analysis samples are then accepted and trusted by both the supplier and receiver for transaction settlements. This is a radical departure from traditional methodologies, where the sampler would go to the stockpile at either the IBA-HNF production site or the smelter for in situ weighing and sampling. Instead, this innovative approach requires the complete 25-tonne lot to be conveyed to the centralized sampling hub—embodying the concept that “the mountain must come to Mahomet.” This single site approach was inspired by the comprehensive analysis of the conventional “Assay Exchange” paradigm (Esbensen & Vogel, 2023), which was shown to contain inherent weaknesses due to two sampling procedures whose principal uncertainties have been left out of consideration.

In this paper, the focus shall specifically be on issues related to sampling and sample preparation, the steps depicted in Figure 3.

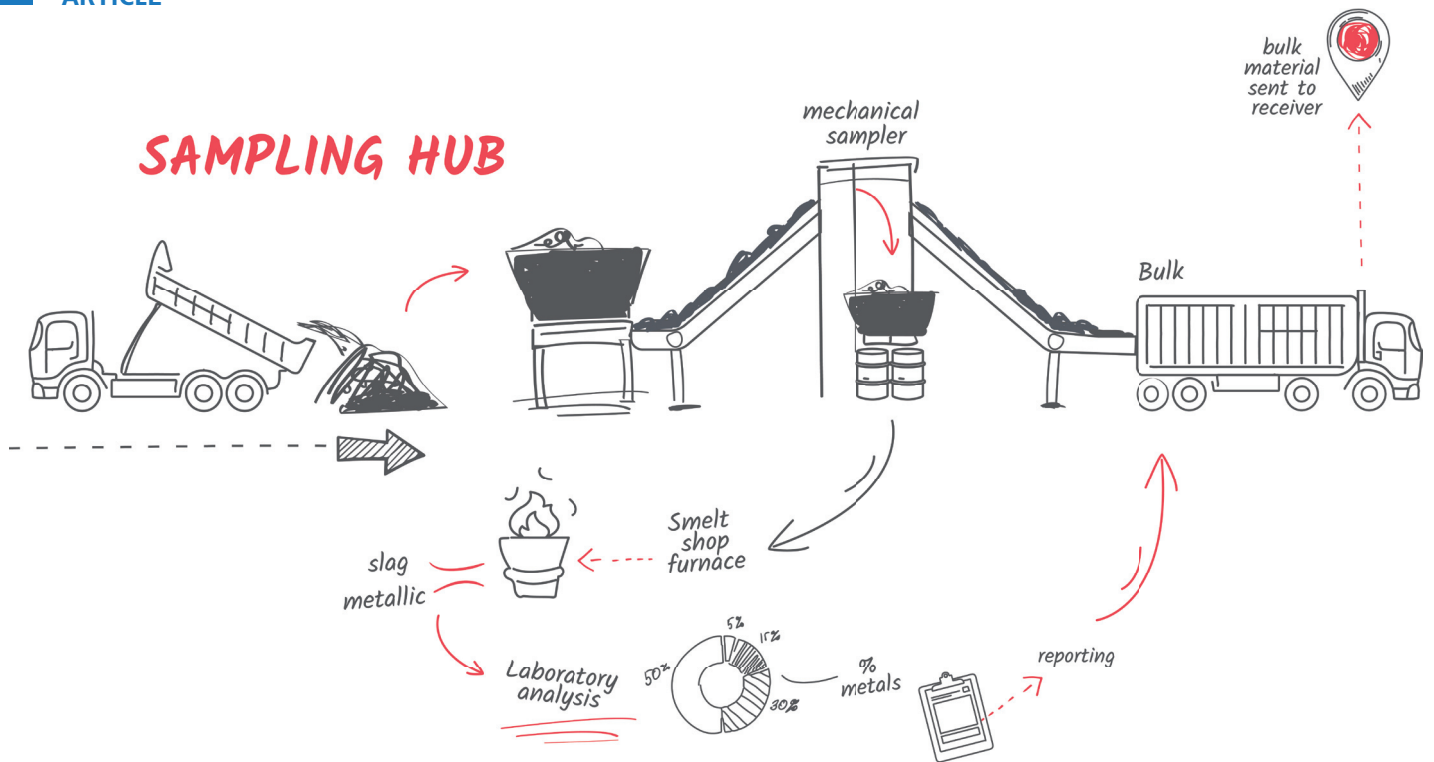


Fig. 3: Graphical illustration of the Sampling Hub in the Netherlands.

## 5. Optimizing One-Dimensional Lot Configuration for Bias-Free Increment Extraction

Bias elimination is achieved when incorrect sampling errors are meticulously eliminated. At the Netherlands-based sampling hub for IBA-HNF, lots of nominally 25 tons are reconfigured into a one-dimensional (1-D) form. Specifically, the IBA-HNF is transported on a conveyor belt in a manner where its length and surface area vastly exceed its width and height, as elaborated in Esbensen (2020). This ensures complete accessibility of the entire lot for increment extraction. Increment slices from this 1-D stream are obtained using a Vezin sampler, meeting the necessary-and-sufficient criteria for removing increment delimitation error (IDE), increment extraction error (IEE), and weighting error (IWE) as outlined by Vogel (2017).

### 5.1 Ensuring Unbiased Sampling through Rigorous Quality Control Measures

As effective quality control to demonstrate that the active mechanical sampling system (MSS) remains free from IDE, IEE and IWE caused by e.g. cutter stopping or slowing down in the stream, a blockage with metal wires, or fluctuating flow-rate of the stream itself, the m/m sampling ratio is monitored on a continuous basis, as per ISO 11790 (2017).

The sampling ratio,  $R_s$ , serves as a critical parameter for assessing the reliability of the sampling process. It is calculated by dividing the actual mass of the sample  $m_A$  in kilograms by the mass of the material it represents  $m_{SL}$  in tonnes multiplied by 1000 as shown in the equation [1]:

$$R_s = \frac{1000 m_A}{m_{SL}} \quad [1]$$

The control chart for the sampling ratio plots this ratio (Figure 5). As all system settings are constant—such as cutter apertures, lot size, and mass flow rate, the sampling ratio demonstrates absence of bias at the MSS of the sampling hub, by:

1. Consistency: A stable sampling ratio plotted on a control chart, suggests a consistent sampling approach. If  $R_s$  remains stable within control limits, the sampling process can be considered unbiased.
2. Process Control: A sampling ratio control chart can quickly highlight instances where the ratio goes out of control, thereby signaling a need for investigation and corrective action. This reactive approach helps in maintaining an unbiased system.



Credit: Alfred H Knight, Holland; used with permission.

Fig. 4: Vezin type sampler in rotating motion, obtaining a TOS-correct full cut of the falling IBA-HNF stream.

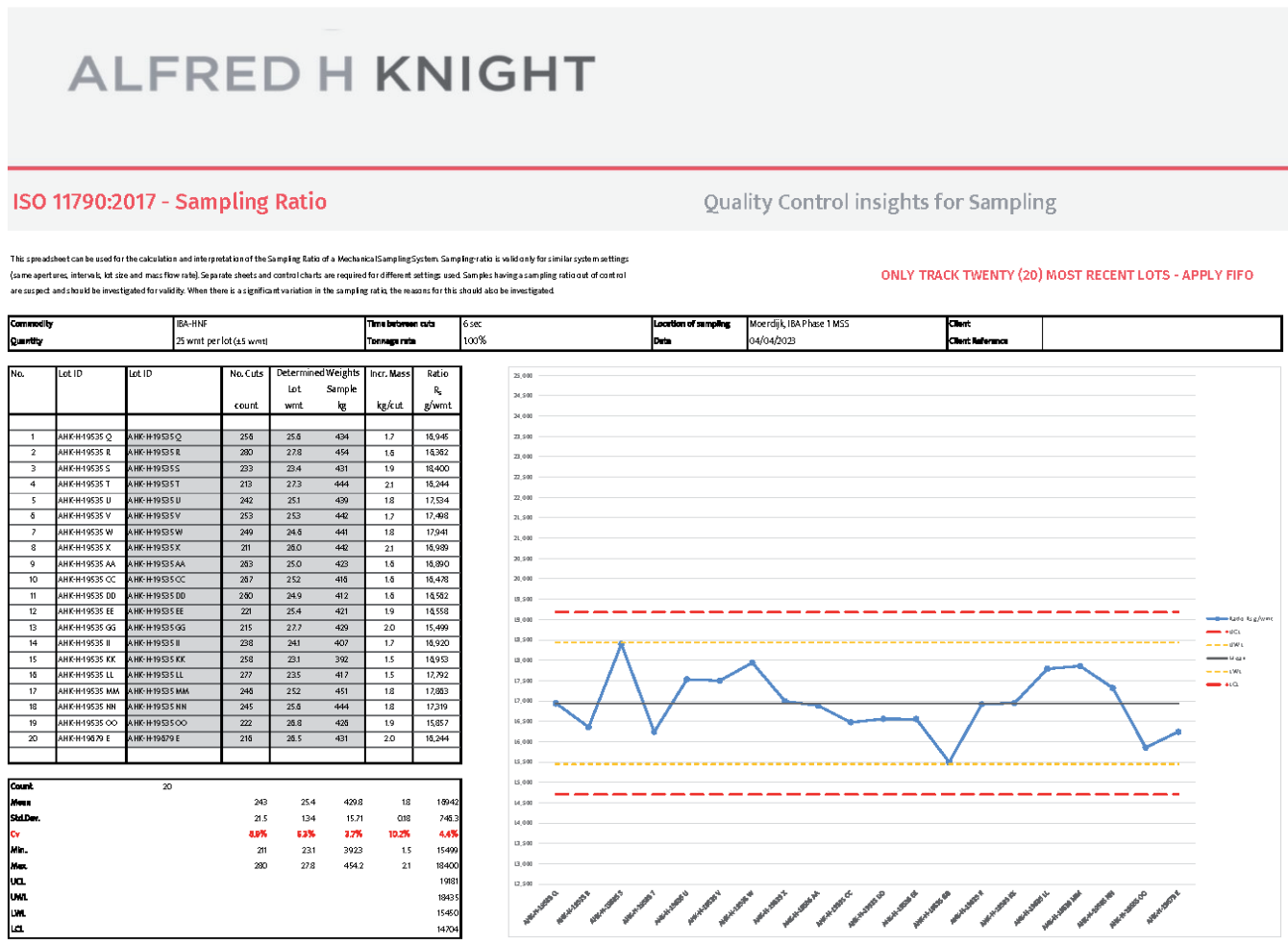


Fig. 5: Sampling Ratio control chart

## 6. Optimization and Validation of Sampling and Sample Preparation in IBA-HNF Trade: An ISO 3085 (2019) Based Approach

Given the distinctive composition of each IBA-HNF shipment and the substantial variances between shipments, variographic analysis is unsuitable for sampling process evaluation, as shipments originate from a myriad of concentration levels, varying beneficiation stages, and multiple Municipal Solid Waste Incinerators (MSWI) throughout Europe. Therefore, to evaluate the precision of the sampling process in practice rather than theory at the sampling hub, duplicate samples A and B are formed by alternating cuts of the Vezin sampler.

To establish appropriate overall precision checks in both sampling and subsequent sample preparation, we diverge from the famous Gy's formula and apply ISO 3085 (2019) instead. This standard accommodates variations in lot quality through a practical statistical framework. In the present new methodology, we perform routine sampling and record data for thirty lots on a first-in, first-out basis. Primary sampling cuts by the MSS of each lot are alternated to produce two gross sample portions (composite samples), each of which are independently prepared through induction furnace melting after which a key quality characteristic—metal yield adjusted for metal contained in slag—is determined. It is recognised that this approach may not estimate for the overall precision for certain parameters, such as gold. However, as quality control is consistently applied to every lot and the IBA-HNF industry has predefined *target masses*, it is claimed that the new processing facility operates within a regime that allows acceptable quality parameter checks.

Data for this process, captured in April 2023, is collected in Table 1. The relationship between these precision values is shown in equation [2]. The mean and the range of each pair of measurements is calculated as per equations [3] and [4], and the overall mean and estimated value of overall standard deviation follow by equations [5] and [6] where  $n$  is the number of lots (here 30). The resulting overall precision of sampling, preparation and measurement ( $\beta_{SPM}$ ) is estimated to be twice the overall standard deviation and considers that each sample portion is half of the routine sample and therefore applying division factor of  $\sqrt{2}$ , as per ISO 3085 (2019) as shown in equation [7].

Finally, a statistical upper control limit,  $D_4$ , is applied in equation [8] with a value of 3.47 (the 99% limit for the difference between two independent normally distributed measurements).

By employing this methodology, the calculated  $\beta_{SPM}$  can confirm that our process is tightly controlled within statistically defined limits.

$$\hat{\sigma}_{SPM}^2 = \hat{\sigma}_S^2 + \hat{\sigma}_P^2 + \hat{\sigma}_M^2 \quad [2]$$

$$\bar{x} = \frac{1}{2}(x_1 + x_2) \quad [3]$$

$$R = |x_1 - x_2| \quad [4]$$

$$\bar{\bar{x}} = \frac{1}{n} \sum \bar{x} \quad [5]$$

$$\hat{\sigma}_{SPM}^2 = \frac{1}{2n} \sum R^2 \quad [6]$$

$$\beta_{SPM} = \sqrt{2\hat{\sigma}_{SPM}^2} \quad [7]$$

$$UCL = D_4 \hat{\sigma}_{SPM} \quad [8]$$

Thus calculated, the obtained overall precision for the sample mass stands at 4.20 kg, while for metal yield, it is 2.04%. The upper control limits are 10.30 kg for the range of gross sample mass between portions and 5.00% for the range of metal yield between portions. These results not only meet, but exceed the industry-defined Key Performance Indicators (KPI) for the sampling, preparation, and measurement processes conducted at the AHK Sampling Hub.

**Tab. 1:** Interleaved Sampling Data for Quality Control: Comparing Portion Mass and Metal Yield, 30 IBA-HNF Shipments, April 2023.

AHK Ref.	Mass A (kg)	Mass B (kg)	Mean <sup>x̄</sup> (kg)	Range R (kg)	Yield A%	Yield B%	Mean <sup>x̄</sup> (%)	Range R (%)
19679 H	242.0	243.6	242.8	1.6	93.6	92.3	92.9	1.3
19679 I	215.5	217.1	216.3	1.6	93.6	94.5	94.1	0.9
19679 J	196.8	200.8	198.8	4.0	93.6	93.3	93.4	0.3
19679 K	242.2	238.8	240.5	3.4	79.7	81.0	80.3	1.3
19679 L	236.1	244.7	240.4	8.6	81.4	83.4	82.4	2.0
19679 M	223.4	236.1	229.8	12.7	77.3	80.0	78.7	2.7
19679 N	224.4	223.1	223.8	1.3	81.9	77.6	79.7	4.3
19679 O	225.7	222.3	224.0	3.4	88.4	90.9	89.6	2.5
19679 P	222.9	222.9	222.9	0.0	79.0	79.2	79.1	0.2
19679 BB	215.8	216.4	216.1	0.6	84.4	86.2	85.3	1.7
19679 CC	211.8	212.3	212.1	0.5	82.8	83.0	82.9	0.2
19679 JJ	218.1	220.3	219.2	2.2	81.7	85.2	83.4	3.5
19679 KK	217.0	208.7	212.9	8.3	82.4	84.7	83.5	2.3
19679 M2	199.9	202.2	201.1	2.3	89.0	90.1	89.5	1.1
19679 N2	214.8	213.2	214.0	1.6	88.2	88.4	88.3	0.3
19679 O2	224.5	224.3	224.4	0.2	89.9	90.4	90.1	0.5
19776 A	204.0	207.9	206.0	3.9	90.8	91.8	91.3	1.0
19776 B	214.2	213.1	213.7	1.1	89.8	93.0	91.4	3.2
19776 C	205.2	205.0	205.1	0.2	87.3	93.1	90.2	5.8
19776 E	209.1	208.1	208.6	1.0	81.9	81.1	81.5	0.8
19776 G	206.0	204.1	205.1	1.9	83.8	81.0	82.4	2.8
19776 H	202.1	197.4	199.8	4.7	80.3	80.6	80.5	0.3
19776 I	210.6	211.8	211.2	1.2	90.4	88.8	89.6	1.6
19776 J	206.1	205.5	205.8	0.6	89.1	89.2	89.2	0.1
19776 K	213.6	210.0	211.8	3.6	86.7	85.8	86.3	0.9
19776 L	235.2	243.6	239.4	8.4	89.7	90.4	90.1	0.7
19776 N	205.8	208.7	207.3	2.9	80.8	80.0	80.4	0.8
19776 O	200.6	205.2	202.9	4.6	78.3	76.7	77.5	1.6
19776 P	203.1	203.2	203.2	0.1	84.1	83.7	83.9	0.4
19776 Q	220.9	218.2	219.6	2.7	89.8	88.7	89.2	1.1
$\bar{\bar{x}}$			215.9				85.9	
$\sum R^2$				528.76				124.35



## 7. Conclusion

In this study, we have presented a comprehensive methodology for optimizing and validating the precision of a new TOS-correct sampling and sample preparation approach in the Incinerator Bottom Ash – Heavy Non-Ferrous (IBA-HNF) trade. Beginning with transformation of the 3D lot to a 1D stream, the methodology eliminates common bias-generating sampling errors such as IDE, IEE, IWE (Gy, 1979), by enabling representative sampling by a TOS-correct Vezin cutter. The mass of the resulting sample is at an industrial scale, and its subsequent homogenization process via smelting delivers consistency to the industry. By eliminating bias and adapting the ISO 3085 (2019) standard to demonstrate precision, we achieved a noteworthy 2.04% precision for overall metal yield, which exceeds industry benchmarks and our KPI target. This level of precision instills trust in the commercial settlement values for elements such as copper, gold, silver, platinum, and palladium. These trustworthy values are essential for both the IBA-HNF concentrators and the European smelters engaged in this circular economy material.

This study serves as a critical reference for stakeholders in the IBA-HNF industry, offering insights and actionable solutions for achieving reliable, bias-free, and precise outcomes in sampling and sample preparation. Moreover, the financial implications of this one-stop methodology could potentially improve settlement agreements, contributing positively to the overall trade (Esbensen & Vogel, 2023).

As for future work, we suggest the exploration of this methodology's applicability to other circular economy and electrification metals. The integration of automated systems for further optimization will also be considered. By adhering to the Theory of Sampling (TOS), we point to how this development work and this study contributes its part to the United Nation Sustainable Development Goals 9 and 12, promoting innovation, sustainability, and responsible consumption and production.

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