SAMPLING SPECIAL SECTION

Laboratory test sample representativity: an easily neglected aspect in consignment and its economic impact

Li Huachang and Zhu Mingwei
BGRIMM MTC Technology Co. Ltd, China

An unrepresentative sample is unable to reflect the true quality of materials and goods, and will eventually cause laboratory chemical analysis results that cannot substantiate trade settlements between buyers and sellers. In quality inspection of mineral products and metals, sampling errors account for ~80 % of the total error, with sample preparation errors responsible for ~15 % and analytical errors accounting for only 5 %. If sampling is poorly represented, no matter how accurate the sample preparation and chemical analysis, quality grading can be severely compromised. Ignoring primary sampling, there are still significant representativity problems arising from sample preparation causing all parties difficulty when trying to find an answer to the crucial question: "where did the money go?". Thus, at the second and third stage sampling levels also, huge economic losses can occur for the buyer *or* the seller. Three cases from the international copper industry sector are presented.

Example 1: Significant trade settlement impact from sampling and grading of high purity copper cathode material

Copper cathode material is usually divided into three categories according to the content of impurities. Impurity element concentrations of Class A cathode copper shall not exceed 0.0065 % in total, while lead (Pb) must not exceed 0.0005 % and iron (Fe) 0.0010 %. Reliable control of misrepresentation of samples used for laboratory testing has a great impact on quality grading and pricing of high pure cathode copper.

Africa is rich in non-ferrous mineral resources, especially copper mineral reserves.

Many Chinese companies have started operating in Africa, building mining plants, concentrating mills and smelters, and eventually smelting and producing copper cathode material and selling it globally. Copper cathode material is usually produced and traded directly in the original size format of 80 × 80 cm square plates with a thickness of about 1 cm, which weighs ~200 kg per piece. To ease transportation, copper cathode plates are usually strapped together using high strength steel bands into bundles suitable for loading weights of typically 1–2 tons each.

If the Class A copper cathode sampling process is contaminated by strap steel bands, as shown in Figure 1, it will lead to an excessive iron content, resulting in a grade reduction of the copper cathode products. Each quality grade class reduction results in a price reduction of ~$30 per ton. For a smelter with an annual output of 200,000 tons of copper cathode with, say, 10 % of samples contaminated, the annual output value is reduced up to **$600,000**, calculated as follows:

$$\text{Economic Loss} = 30 \times 200{,}000 \times 10\,\% = \$600{,}000$$

Figure 1. Copper cathode plates contaminated by steel band straps. © The authors

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Product degradation will not only bring *pro rata* economic losses to the seller, but also affects its reputation as the performance stipulated in the contract turns out to be difficult or impossible to achieve, ultimately leading to a reduction in the seller's market share, with reduced corporate profits of the whole enterprise.

Example 2: Representative sampling of copper concentrates in ton bag packaging directly determines the procurement risk of the smelter

Due to poor resource endowment, low grade, difficulties in exploitation and process, Chinese copper concentrates are far from meeting the needs of domestic copper smelters. A large amount of copper concentrates are imported. Consignment copper concentrates are packaged either in bulk or in ton bags. The economic results for bulk copper concentrate consignments are relatively stable and less controversial because of the relatively easy operability of the sampling methods employed. However, due to very uneven quality fluctuation distribution *between* bags, copper concentrates packaged in *ton bags* often have large deviations (gaps) between the in-material quality (analytical results) when determined in loading and unloading ports, gaps which exceed the "reasonable" error range assumed in contracts etc.

A large smelting enterprise in China needs to import more than 500,000 tons of copper concentrates every year. Due to the type of inconsistent sampling approaches just described at loading and unloading, final copper grade difference of the two ports was up to 1.5%. In order to identify the *cause* of this apparent poor quality match, 20 tons was randomly selected from a shipment of 500 tons to serve as a basis for detailed investigations of the between-bag quality fluctuations. The resulting test results are shown in Table 1.

Between-bag copper variations (never zero) ranged up to a maximum gap of 11.16 %. If the between-bag coefficient of variation (CV%) is not carefully controlled, this may easily propagate into a large compositional gap in the final results. The domestic smelter, as the buyer, takes account of large material quantities for which the compositional estimations must be determined with a very high accuracy and precision.

As an example, the smelter used copper concentrates transported by sea, with 20,000 tons per delivery; the relevant London Metal Exchange's copper price was about $9400 per ton. If the copper shipment unloading port's estimated copper concentration was 1.5 % *higher* than the loading port, regardless of the impact of other valuation elements and processing fee deductions, a value of **$2.82 million** was due to the contested differently indicated amounts of copper alone.

Example 3: A detail from blister copper sampling and preparation

China has a huge need for copper raw materials. In addition to directly importing copper concentrates as raw materials for domestic production of copper cathodes, Chinese companies also construct copper smelting facilities overseas to obtain copper products such as copper blister, copper anode and copper cathode, which are also sold domestically.

The copper content in copper blister is usually around 99 %. For trade purposes, copper blister is usually delivered in the form of ingots or anode plates. Generally, the content of copper, gold and silver is used as the characteristic pricing elements of the product—sometimes including other specific impurity components. The analysis is preceded by a sampling procedure which generally includes the following steps: randomly pick out a defined number of ingots from a consignment, further select a few points on each ingot for drilling out and collect all *cuttings* to become a *composite sample*, which is milled (ground) and

Table 1. Copper content in randomly selected copper concentrate bags.

Bag No.	Cu (%)	Bag No.	Cu (%)
1	16.36	10	21.35
2	18.58	11	25.69
3	19.06	12	22.64
4	18.65	13	19.45
5	18.45	14	19.70
6	24.14	15	26.08
7	18.74	16	23.68
8	27.52	17	16.82
9	22.43	18	19.20
Mean	21.07		
Range	11.16		

Figure 2. Copper blister. © The authors

from which a test sample is produced for analysis of the content of each element involved in the contractual specifications.

Both parties in the trade use analysis results as the basis for a fair trade settlement—which should always cause no issues *were* trading parties using only one analytical facility. But when using two, the road is open for possible *deviating analytical results*, which at first are sometimes difficult to understand as they manifestly represent the *same consignment*. But there is always a rational explanation, an example of which is shown below.

Blister copper sampling procedure

Occasionally when enterprises sell blister copper, the two parties agree on a proscribed sample preparation method in the trade contract as follows. Drill blister copper ingots, grind all the collected cuttings, followed by *screening* by a 40-mesh sieve, followed by further grinding of the left behind, over-sized sample part again, until the complete composite sample has passed through the screen.

However, the mandated method in Chinese domestic industry is to *separate* the material of the up-sieve and down-sieve size bins into identified sub-samples. According to the screened mass ratio, weighing is also carried out of the separated up-sieve and the down-sieve sub-samples, which are then analysed for copper content.

As an internal control, after a batch of samples are sieved, sub-samples of these particle size bins are tested separately to obtain their specific copper contents, as shown in Table 2. The reason for the resulting diverging results may be due to the different constituent particle sizes, or it may be a result of the repeated grinding operations, which causes the material to be *oxidised*, resulting in a lowered pure copper content for the small particle sizes.

Table 2. Copper content in different particle size categories.

Particle size bin	Mean copper %
>40 mesh	99.15
<40 mesh	98.85

A large copper smelter established overseas by China has an annual output of about 20 tons of copper blister. If the sample preparation method of all 40-mesh sieves is used in trade accounting, this alone may bring about a 0.3 % reduction in copper content. The unit price of copper blister at the time of writing is US$9400 per ton, regardless of the influence of other pricing elements. Thus, for this smelter, this single detail of sample preparation procedures alone may represent a loss of up to **US$5.64 million** in trade **per year**. Every *detail* matters in global commodity trade ….

Conclusions

These consignment examples demonstrate the economic importance of even the smallest differences in laboratory preparation and analysis approaches. Sub-sampling, sample preparation, transportation and sample storage processes may all have significant effects on the quality and representativity of samples that eventually enter the analytical instruments. Only by careful and strict control of each operation can the test samples ultimately used for analysis be qualified as representing the full, comprehensive quality of commodities and the goods—equally in the interest of both buyers and sellers. The apparent *minute* issues treated here for a large-volume bulk commodity, may quickly lead to surprisingly large, added or lost, values which are far too large to overlook in consignment economics.

Between the laboratory and management

The reader is referred to two earlier Sampling Columns dealing with how to run a commercial analytical laboratory notably with, or without, the TOS on the agenda: Does management have the necessary foresight to accept the challenge of also caring for "the customers of the customer of the laboratory"? This is an exciting two-part story, both of which are just a click away.

K.H. Esbensen, "A tale of two laboratories I: the challenge", *Spectrosc. Europe* **30(5),** 23–28 (2018). https://doi.org/10.1255/sew.2018.a3

K.H. Esbensen, "A tale of two laboratories II: resolution", *Spectrosc. Europe* **30(6),** 26–28 (2018). https://doi.org/10.1255/sew.2018.a4