

The Ongoing Challenge of Representative Sampling of Bulk Mineral Commodities

Ralph J Holmes

E-mail: ralph.holmes@csiro.au

Sampling of bulk mineral commodities for international trade, such as iron ore, coal and a wide variety of mineral concentrates, is generally carried out in accordance with National or more commonly International (ISO) Standards developed to provide representative samples for subsequent analysis and payment. Because commercial transactions are involved, clearly getting the sampling right is critically important, and poor sampling practices can potentially lead to substantial financial losses for one of the parties involved. The “golden rule” for correct sampling is that “all parts of the material being sampled must have an equal probability of being collected and becoming part of the final sample for analysis”. If this rule is not respected, then bias is easily introduced and samples are not representative.

While on-site observations indicate that the adoption of good sampling practices is improving, ensuring that samples are representative continues to be an ongoing challenge. This is often due to cost-cutting measures where sampling facilities, equipment and operations are the first to suffer, or it may just simply be due to ignorance of the requirements for collecting representative samples despite the existence of National and International Standards as well as high level sampling courses presented by international experts. More often than not, the company focus is on maximizing production tonnage rather than product quality and its measurement. Areas where significant issues continue to occur include:

- **Primary cutter design for ever increasing high-capacity streams**
- **Correct operation of cross-stream secondary cutters**
- **Crusher performance and ongoing maintenance, particularly in relation to product particle size**
- **Retained sample mass versus particle size**
- **Extraction and handling of moisture samples**
- **Equipment maintenance.**

Timely ongoing maintenance of sample stations is critical and needs to be a high priority to ensure correct performance. A “set and forget” strategy simply does not work. Sampling needs to be given the commitment it deserves by company management, particularly through correct sample plant design, timely equipment maintenance, and appropriate staff training and awareness.

Introduction

The accurate sampling of bulk mineral commodities, such as iron ore, coal and a wide variety of mineral concentrates, is critically important where commodities are changing hands between trading parties. The sampling is generally carried out in accordance with National or more commonly International (ISO) Standards that are designed to provide representative samples for subsequent analysis and payment. Unfortunately, poor sampling practices can lead to substantial financial losses for one of the parties involved, so clearly such practices need to be avoided. The “golden rule” for correct sampling is that “all parts of the material being sampled must have an equal probability of being collected and becoming part of the final sample for analysis” (Gy, 1982a and 1982b; Pitard, 1993, 2005 and 2019; Holmes, 2004, 2005, 2007, 2010, 2013, 2015, 2017 and 2019). If this rule is not observed, bias is easily introduced and the samples collected are not representative. Even a relatively small bias can have significant economic consequences (Holmes, 2021), and no amount of replicate analysis of the material collected will eliminate the bias once bias is present.

While the adoption of good sampling practices in the mineral industry continues to improve, representative sampling of mineral commodities remains an ongoing challenge. This is often due to cost-cutting measures where sampling facilities and procedures are the first to suffer and second-rate facilities end up being constructed. Alternatively, the cause may be a poor knowledge of representative sampling requirements by those charged with designing, constructing and operating sampling facilities despite the existence of National and International Standards as well as high level sampling courses presented by international experts. All too often the main driving force is maximizing the production tonnage rather than product quality and its measurement. Areas where significant issues occur usually include primary cutter design for high-capacity streams, correct operation of cross-stream secondary cutters, crusher performance, retained sample mass, extraction and handling of moisture samples, and overall equipment maintenance. Clearly, sampling needs to be given the commitment it deserves by company management through correct sample plant design, timely equipment maintenance, and appropriate staff training and awareness.

The best sampling location for mineral commodities is at a transfer point between conveyor belts (Holmes, 2017 and 2019), where the full cross-section of the stream can be conveniently intercepted at regular intervals, enabling representative samples to be obtained. However, it is impossible to take a representative sample in-situ from a stockpile (see Figure 1) or a ship, because it is impossible to gain access to the material in the centre of the stockpile or deeper within the hold of the ship. In these two cases, samples must be taken while stockpiles are being built up or broken down, or while ships are being loaded or unloaded.



Figure 1. Sampling from the side of a stockpile is problematic and will not provide a representative sample.

After satisfying the above requirements for correct sampling, attention then needs to be given to the following important sampling principles (Holmes, 2019):

- Collecting sample masses that are large enough taking into account the particle size to reduce the fundamental, grouping and segregation errors (Gy, 1982a; Pitard, 1993 and 2019) to acceptable levels.
- Taking a sufficient number of primary increments to reduce the long-range quality fluctuation error to an acceptable level.
- Selecting sampling locations that avoid the presence of periodic variations in quality, eg, due to equipment items such as bucket wheel reclaimers and centrifugal pumps.
- Eliminating accessory errors, such as sample contamination, sample spillage (see Figure 2), particle degradation and operator mistakes.



Figure 2. Accessory errors such as sample loss due to holes in chutes need to be eliminated.

Primary cutter design and operation

Extraction of primary increments by the primary cutter in a sample station is the first step in obtaining representative samples from mineral commodities. Consequently, the design and operation of the primary cutter is critical, the key requirements being to ensure correct increment delimitation and increment extraction (Gy, 1982a; Pitard, 1993 and 2019). The requirements for primary cutter design and operation are summarised in the relevant National and International Standards for mineral commodities. For example, the general requirements for iron ore largely taken from ISO 3082 (Iron ores – Sampling and sample preparation procedures) are as follows:

- There shall be no overflow or spillage of sample or loss of ultra-fines.
- There shall be no impedance to flow of sample material through the sample cutter at the maximum flow rate.
- Bucket-type cutters shall be of sufficient capacity to accommodate the increment mass obtained at the maximum flow rate without any sample loss.
- There shall be no clogging or retention of residual material in the sample cutter, i.e. the cutter shall be self-clearing.

- There shall be no contamination or introduction of material other than the sample into the sample cutter.
- There shall be no significant change of the quality of the sample while taking increments, e.g. degradation of the constituent particles if the sample is taken for size determination or change in moisture content if the sample is taken for moisture determination.
- The sample cutter shall take a complete cross-section of the stream, both the leading and trailing edges clearing the stream in one path.
- The sample cutter shall intersect the stream either in a plane perpendicular to or along an arc normal to the mean trajectory of the stream.
- The sample cutter shall travel through the stream at a uniform speed, not deviating by more than $\pm 5\%$ at any point.
- The geometry of the cutter aperture shall be such that the cutting time at each point in the stream is equal, not deviating by more than $\pm 5\%$, e.g. straight-path cutters shall have parallel cutter lips and rotary cutters shall have radial cutter lips.
- The plane of the cutter aperture shall not be vertical or near vertical to avoid sample loss from particles striking the inside edge of the cutter aperture and bouncing downwards to reject.

Expanding on a number of key aspects in the above list, the following requirements are essential to ensuring correct increment delimitation and increment extraction (Holmes, 2019):

- The sample cutter must take a complete cross-section of the process stream with both the leading and trailing edges of the cutter completely clearing the stream at the end of each traverse.
- The length of the cutter aperture must be large enough to intercept all of the material in the stream, including particles bouncing off the inside edges of the cutter aperture.
- The cutting time at each point in stream must be equal. Flap or diverter type cutters do not respect this condition.
- The cutter must travel through the stream at a uniform speed, subject to a maximum cutter speed of 0.6 m/s, accelerating up to its cutting speed before entering the stream and decelerating to a stop only after leaving the stream.
- The cutter must have sufficient power for its duty. Electric cutter drives are best, although hydraulic drives are acceptable if well maintained. Pneumatic cutter drives are not satisfactory, because it is very difficult to maintain constant cutter speed while traversing the stream.
- Belt scrapings should fall within the area traversed by the cutter.
- The sample cutter must be non-restrictive and self-clearing, discharging completely each increment without any reflux, overflow or hang-up in the cutter aperture. Hence, the cutter body should be large and streamlined in design to eliminate reflux at high flow rates.
- The cutter aperture must be at least three times the nominal top size (d) of the material being sampled, i.e. $3d$ to prevent preferential loss of the larger particles, subject to a minimum of 10 mm for dry solids.

The main problems often observed with primary cutters in sampling facilities operating in the minerals industry are failure to take a complete cross-section of the stream, undersized cutter apertures, excessive cutter speed, and sample reflux from cutter apertures at the ever-increasing flow rates encountered in industry. An example of a poorly designed primary cutter that does not correctly take a complete cross-section of the stream is shown in Figure 3 (Holmes, 2019). The primary cutter pivots around an axis on the side of the conveyor belt and hence the cutting time at each point in the stream is not equal, leading to potential bias. This problem does not occur when full cross-stream primary cutters are installed.



Figure 3. Poorly designed primary cutter that pivots around an axis on the side of the conveyor belt, so the cutting time at each point in the stream is not equal.

As already indicated above, sample reflux from the primary cutter aperture can be a significant problem at high flow rates (typically greater than 10,000 t/hr), particularly for fine products that have a tendency to hang up in the cutter aperture especially when they are slightly moist. This is evident in Figure 4, where massive sample reflux from the top of the primary cutter aperture and hence sample loss is clearly visible. The solution is to redesign the primary cutter to provide a much larger cutter body and capacity able to accommodate large primary increments, with a sloping rear section that directs the incoming sample down the sample delivery chute and away from the incoming sample material, thereby eliminating sample reflux. A well-designed primary cutter conforming to these requirements is shown in Figure 5. The end result is a primary cutter that is able to correctly sample high-capacity streams without sample reflux as shown in Figure 6.



Figure 4. Sample reflux from a poorly designed primary cutter with a narrow body and limited sample capacity.



Figure 5. Well-designed primary cutter with a large body for sampling high-capacity streams without reflux from the cutter aperture.



Figure 6. Sampling a high-capacity stream with a well dimensioned primary cutter displaying no sample reflux.

Operation of cross-stream secondary cutters

Another common ongoing problem in sampling bulk mineral commodities is the operation of the secondary cutter, which often is a cross-stream cutter (see Figure 7). The relevant ISO Standards specify the minimum number of secondary cuts (or increments) to be taken for division of primary increments to a suitable mass for subsequent preparation, which commonly is specified as follows:

- A minimum of 4 cuts for mass-basis sampling where constant mass division is required.
- A minimum of 5 cuts for increments of average mass for time-basis sampling where proportional division is required.

It is essential that the cuts taken by the secondary cutter are spread over the entire primary increment to be divided. However, due to incorrect timing of the secondary cuts, unfortunately it is quite common to see substantial sections of the primary increment feed stream totally ignored after the minimum number of cuts has been taken, which clearly is totally unacceptable and likely to introduce significant bias. Equally bad, only one or two cuts may be taken from the primary increment, once again due to incorrect timing between the secondary cutter operation and the feeding of primary increments to the secondary cutter. Getting the operation of the secondary sampling stage right requires special attention and should not pose too many difficulties given the engineering control systems that are now readily available.



Figure 7. Cross-stream secondary cutter designed to collect separate increments for chemical/moisture analysis and sizing.

Crusher performance and retained sample mass

Crushers are very often key equipment items in sample stations for mineral commodities, particularly for lump products where particle size reduction is required to enable sample masses to be safely reduced to facilitate subsequent sample

preparation while keeping the fundamental error variance under control (Gy, 1982a; Pitard, 1993, 2019). The relevant ISO Standards contain tables specifying the minimum mass of samples for this purpose, an example of which is given in Table 1 for iron ores. Similar tables are provided in ISO 13909-2 for coal and ISO 12743 for copper, lead, zinc and nickel concentrates. Sampling regimes are designed around these tables, all of which rely on the crushers installed in sample stations performing their required duty. However, sadly this is not always the case and many instances are observed in operating sample stations where crusher products are much coarser than the design specification due to wear of crusher components and poor maintenance. This is illustrated in Figure 8 where the crusher product is meant to be less than 10 mm nominal top size, but unfortunately it is more like 20 mm or more, which has a big impact on the minimum sample mass that needs to be retained and hence the overall precision of sampling. Clearly this is not acceptable, so crushers in sample stations need special attention and adequate resources need to be devoted to performance checks and maintenance to ensure that crusher products meet particle size requirements in the design of the sample station. More specifically, regular crusher checks are required and crusher gaps need to be adjusted and/or the crushers overhauled to maintain performance.

Table 1. Minimum mass of divided gross sample for iron ore.

Nominal top size (mm)	Minimum mass of divided gross sample (kg)	
	$\sigma_D = 0.1\% \text{ Fe}$	$\sigma_D = 0.05\% \text{ Fe}$
40	325	1,300
31.5	180	710
22.4	75	300
10	10	40
6.3	3.2	13
2.8	0.5	1.7
≤ 1.4	0.5	0.5



Figure 8. Nominal top size check of crusher product in a sample station. In this case the target nominal top size of 10 mm was not achieved.

Moisture samples

Extraction of moisture samples for bulk commodities is particularly problematic. Unlike chemical analyses which do not change during the sampling process (unless for example a sample is overheated when it is pulverised prior to analysis which may change its chemical composition), significant changes in moisture content can occur in moisture samples during sampling, sub-sampling and storage prior to moisture determination. Consequently, it is vital that moisture samples are extracted quickly with the minimum number of sampling stages, collected in sealed containers, and preferably returned to the laboratory immediately for determination of moisture content. Trickling a moisture sample through a multi-stage sample plant with lots of exposure to ambient air must be avoided at all cost. Notwithstanding these precautions, inevitably some change in moisture content will almost certainly occur and a common practice is to apply a correction factor to compensate for the measured change in moisture content determined for individual sample plants.

Equipment maintenance and precision checks

The maintenance of sampling facilities is critical and needs to be given high priority and management commitment to ensure correct performance. A “set and forget” approach simply does not work. Unfortunately, many examples of poor maintenance continue to be found in key sampling installations for determining the value of bulk mineral commodities at the point of sale, including worn cutter lips (see Figure 9), partially blocked primary cutter chutes (see Figure 10), sample build-up in secondary cutter apertures (see Figure 11), blockages of Vezin cutter apertures (see Figure 12) and sample spillage and

sample loss. Fixing such problems is not technically complex, but requires strong management commitment to conducting regular inspections of sampling equipment and rectifying identified problems in a timely manner. In more detail, key items that need to be checked include:

- Size and geometry of cutter apertures, including checking that cutters intercept the complete stream.
- Cutter speed and uniformity while cutting the ore stream.
- Condition of cutter lips, including wear and missing cutter lips.
- Build-up and/or blockages in cutter apertures and chutes.
- Reflux from cutter apertures, particularly at high flow rates and for fine moist material.
- Ingress of extraneous material when the cutter is parked.
- Location of belt scrapers and whether scrapings are sampled.
- Increment and sample mass.
- Number of primary, secondary, tertiary increments, etc.
- Holes in cutters, chutes and bins resulting in sample loss.
- Crusher performance, particularly product particle size.
- Condition of vibratory feeders.
- Sample mass as a function of nominal top size at each sampling and sample preparation stage.



Figure 9. Primary cutter showing signs of significant wear of the cutter lips.



Figure 10. Partially blocked primary cutter chute.



Figure 11. Sample build-up in the secondary cutter apertures.



Figure 12. Partially blocked cutter aperture in a Vezin divider.

Finally, it is highly desirable to regularly check the sampling, sample preparation and measurement precisions actually achieved in practice, which can be determined using duplicate “interleaved” sampling, e.g. using ISO 3085. If necessary, action can then be taken to address the largest contribution(s) to the overall variance and hence optimise the sampling regime.

Conclusion

Accurate sampling of bulk mineral commodities, such as iron ore, coal and a wide variety of mineral concentrates, is critically important when commodities are changing hands between trading parties, because poor sampling practices can lead to substantial financial losses for one of the parties involved. The “golden rule” for correct sampling is that “all parts of the material being sampled must have an equal probability of being collected and becoming part of the final sample for analysis”. If this rule is not observed, bias is easily introduced and the samples collected are not representative.

While the adoption of good sampling practices in the mineral industry continues to improve, representative sampling of mineral commodities is an ongoing challenge due to either cost-cutting measures where sampling facilities and procedures are the first to suffer or a poor knowledge of representative sampling requirements by those charged with designing, constructing and operating sampling facilities. This occurs despite the existence of National and International Standards, as well as high level sampling courses presented by international experts. Often the main driving force is maximizing production tonnes rather than product quality and its measurement. Areas where significant issues occur usually include primary cutter design for high-capacity streams, correct operation of cross-stream secondary cutters, crusher performance, retained sample mass, extraction and handling of moisture samples, and timely equipment maintenance. It is essential that

sampling is given the management commitment it deserves through correct sample plant design, timely equipment maintenance, and appropriate staff training and awareness.

References

- Gy, P M, 1982a. *Sampling of Particulate Materials - Theory and Practice*, 2nd Edition (Elsevier: Amsterdam).
- Gy, P M, 1982b. Sampling from high capacity streams, in *Proceedings First Australian International Bulk Materials Conference*, Sydney, Australia, pp 407-423.
- Holmes, R J, 2004. Correct sampling and measurement – The foundation of metallurgical accounting, *Chemometrics and Intelligent Laboratory Systems*, **74**: 71-83.
- Holmes, R J, 2005. Design of sample plants – Getting it right first time, in *Proceedings Second World Conference on Sampling and Blending (WCSB2)*, Sunshine Coast, Australia, pp.103-110 (The Australasian Institute of Mining and Metallurgy: Melbourne).
- Holmes, R J, 2007. Best practice in sampling iron ore, in *Proceedings Third World Conference on Sampling and Blending (WCSB3)*, Porto Alegre, Brazil, pp.
- Holmes, R J, 2010. Sampling mineral commodities – the good, the bad, and the ugly, *Journal of the Southern African Institute of Mining and Metallurgy*, **110**: 1-8.
- Holmes, R J, 2013. The importance of sampling in the mineral industry, in *Proceedings Metallurgical Plant Design and Operating Strategies (MetPlant 2013)*, July 2013, Perth, Australia, pp. 34-49.
- Holmes, R J, 2015. Sample station design and operation, in *Proceedings Seventh World Conference on Sampling and Blending (WCSB7)*, June 2015, Bordeaux, France, TOS Forum, Issue 5, pp. 119-128.
- Holmes, R J, 2017. Common pitfalls in sampling iron ore, in *Proceedings Eighth World Conference on Sampling and Blending (WCSB8)*, May 2017, Perth, Australia, pp. 261-264 (The Australasian Institute of Mining and Metallurgy: Melbourne).
- Holmes, R J, 2019. Best practice in sampling iron ore shipments, in *Proceedings Ninth World Conference on Sampling and Blending (WCSB9)*, May 2019, Beijing, China, pp. 51-62.
- Holmes, R, 2021. Incorrect sampling practices always have significant economic consequences – and never more so than where tonnages are large, *Spectroscopy Europe*, Vol 33, No 6, 84.
- ISO 3082, 2017. Iron ores – Sampling and sample preparation procedures (ISO: Geneva).
- ISO 3084, 2002. Iron ores – Experimental methods for checking the precision of sampling, sample preparation and measurement (ISO: Geneva).
- ISO 12743, 2021. Copper, lead, zinc and nickel concentrates – Sampling procedures for determination of metal and moisture content (ISO: Geneva).
- ISO 13909-2, 2016. Hard coal and coke – Mechanical sampling – Part 2: Coal – Sampling from moving streams (ISO: Geneva).
- Pitard, F F, 1993. *Pierre Gy's Sampling Theory and Sampling Practice*, 2nd Edition (CRC Press Inc: Florida).
- Pitard, F F, 2005. Sampling correctness – A comprehensive guideline, in *Proceedings Second World Conference on Sampling and Blending*, Sunshine Coast, Australia, pp. 55-66 (The Australasian Institute of Mining and Metallurgy: Melbourne).
- Pitard, F F, 2019. *Theory of Sampling and Sampling Practice*, 3rd Edition (CRC Press Inc: Florida).