

Sample station design and operation

Ralph J Holmes

CSIRO Mineral Resources Flagship, Private Mailbag 10, Clayton South, Victoria 3169, Australia. E-mail: ralph.holmes@csiro.au

Accurate sampling practices in the mineral industry are critical for determining the chemical, mineralogical and physical characteristics of ores and mineral products for resource evaluation and utilisation, feasibility studies, process design and optimisation, quality control, metallurgical accounting, and ultimately commercial sales. Sampling is the first step in the measurement chain and is where the measurement process all begins, so if the sample that is collected is not representative, then the whole measurement chain is compromised at the outset. However, frequently the responsibility for sampling is entrusted to personnel who do not fully appreciate the significance and importance of collecting representative samples for analysis, and quite often everyone seems satisfied as long as some material is collected and returned to the laboratory for analysis. In the case of sample stations, cost is often the main consideration rather than sampling correctness (unbiasedness), which is unacceptable and needs to change. It is important that sampling experts are involved in the design stage at the outset to avoid structural design flaws and the subsequent need for expensive retrofits to address major and sometimes even fatal problems. Furthermore, ongoing audits of performance need to be conducted to ensure sample stations are adequately maintained and continue to conform to correct sampling principles. Provision also needs to be made for duplicate sampling to monitor the precision achieved in practice on an ongoing basis for quality assurance purposes. The examples used and commented upon here relate to one of the more difficult industry sectors with respect to correct sampling practices, material and constituent type (e.g. ores, concentrates and mineral aggregates), tonnages, process stream flow rates, and wear and tear, and as such provides the ideal showcase for the intended message which applies essentially to all technologies and industries.

Introduction

Samples are taken from many different locations in the mineral industry for optimising resource utilisation, process and grade control, metallurgical accounting and ultimately commercial transactions^{1,2}. These locations include diamond and percussion drill holes, blast holes, feed and product streams, conveyor belts, trucks, railway wagons and stockpiles, a number of which present major, if not impossible, problems in extracting representative samples, e.g. in-situ sampling from a large stockpile. Notwithstanding this, it is surprising how frequently sampling is left to personnel who do not understand its critical importance in providing representative samples for analysis, and quite often everyone is happy as long as just *some* material is collected and sent back to the laboratory for analysis. This approach is totally wrong and completely unacceptable. Representative samples are essential to obtaining meaningful analyses that can be relied upon to make correct resource and quality control decisions and ensure equitable payment for the sale of mineral commodities. Sampling is where the measurement chain begins and the whole measurement process is corrupted at the outset if all samples are not representative. Furthermore, accurate analysis of non-representative samples submitted to the laboratory can very often be a waste of time, leading to reduced mine life, poor recovery in processing plans, and loss of sales revenue. It is therefore critical to ensure that the samples collected are free of significant bias and that the overall precision of the final analyses is appropriate for the required task, both of which are important in the design and operation of sample stations, which is the focus of this paper.

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”, i.e. the Fundamental Sampling Principle (FSP in the Theory of Sampling).¹⁻⁷ If this golden rule is respected at the outset, then extraction of representative samples is largely assured. Otherwise, a sampling

bias is easily introduced, which is particularly serious because no amount of replicate sampling and analysis is able to reduce bias once it is present, far less eliminate it¹. There is no point in being “precisely” incorrect. As pointed out by Gy⁴, the sources of bias that can be eliminated include incorrect delimitation of sample “increments” (i.e. incorrect cutter geometry), incomplete extraction of sample increments, preferential exclusion of specific size fractions, sample loss and sample contamination, while other errors due to the fundamental, grouping, segregation, long-range quality fluctuation, periodic quality fluctuation and weighting errors can never be totally eliminated, but they can be minimised or at the very least reduced to acceptable levels. Unfortunately, many of these requirements are frequently ignored in the design of sample stations to reduce capital costs, which is a dangerous false economy because the samples taken are likely to be seriously biased, the precision may be compromised, and the subsequent cost of retrofitting a correct sampling system can be large. The design of subsequent sampling stages is also very important, particularly in terms of the relationship between particle size and the sample mass that needs to be retained to achieve acceptable precision.

Sample station design

While samples are taken from many locations in mineral processing plants, by far the best method is to sample a moving stream at the discharge point at the end of a conveyor belt or at the end of a slurry pipe.^{1-3, 8, 9} Here the process stream can be intersected at random or regular times or tonnages, and sample “increments” can be collected by taking a full cross-section of the stream with a sample cutter such as shown in Figure 1, and subsequently combining them into representative composite samples for specified time periods or tonnages of material passing through the processing plant. This is guaranteed to satisfy the Fundamental Sampling Principle. Having satisfied this requirement, the sample mass collected then needs to be large enough taking into account the particle size of the

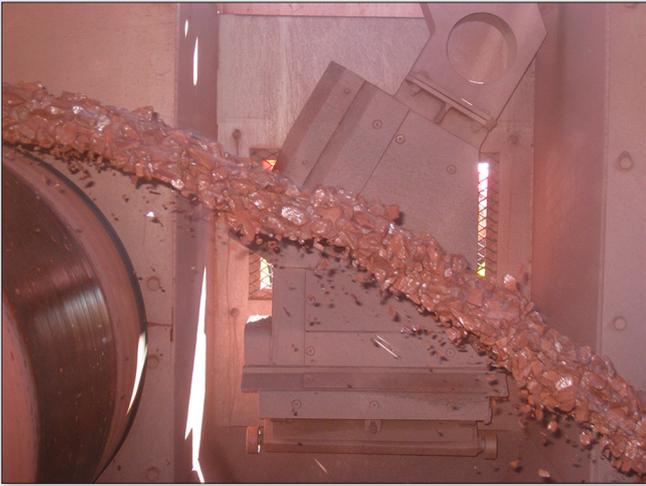


Figure 1. Cross-stream sample cutter (background) designed for taking a full cross-section of an ore stream at the discharge point of a conveyor belt.

material being sampled to reduce the fundamental, grouping and segregation errors to acceptable levels and sufficient increments need to be taken to reduce the long-range quality fluctuation error to an acceptable level. In addition, the sampling location should be selected to avoid the presence of periodic variations in quality due to equipment such as bucket wheel reclaimers and centrifugal pumps. Clearly, accessory errors⁴ such as sample contamination, sample spillage, particle degradation and operator mistakes also need to be eliminated at the outset. An example of such an error is shown in Figure 2, where the sample mass collected exceeded the minimum mass requirement by a large margin, and, instead of using a rotary sample divider or a riffle to reduce the sample mass, the operator simply tipped out part of the sample on the ground. Such practices are clearly unacceptable in the context of representative sampling, but it does indicate the need to design the sampling regime to generate samples of manageable mass for operators and provide lifting aids if required.



Figure 2. Accessory error caused by an operator tipping out part of a sample on the ground instead of using a rotary sample divider or a riffle to reduce its mass. The supervisor is not doing his/her job.



Figure 3. Manual sampling from the top of a conveyor belt most emphatically does not sample the complete ore stream and raises serious safety concerns.

In contrast to full cross-stream sampling, examples of poor plant sampling practices include *scooping* material from the surface of a conveyor belt (see Figure 3), intercepting only part of a falling ore stream (see Figure 4), taking cuts from a fixed location within a launder or extracting slurry from a fixed position within a pipe as shown in Figure 5. Segregation occurs both vertically and horizontally across a conveyor belt due to the action of the idlers and the manner in which the material is fed onto the conveyor, and particles suspended in a slurry segregate under the effects of gravity and centrifugal forces. Consequently, partial stream cuts or extracting only part of the stream are structurally unable to provide representative samples. In Figure 4, the primary cutter is pivoted on the side of the head chute. Consequently, when the cutter is rotated into the ore stream, it does not traverse the complete ore stream and hence increments are extracted from only part of the falling stream, which is clearly incorrect.

Focussing on sampling at the discharge point of a conveyor belt or chute where the complete stream can be intersected with comparable ease at regular intervals, an important consideration is the design of the sample cutter, which must satisfy a number of requirements to eliminate both increment delimitation and extraction errors.⁴



Figure 4. Example of a poorly designed sample cutter that does not traverse the full ore stream.



Figure 5. Pressure pipe samplers do not extract a full cross-section of the slurry stream so the samples collected can never be representative.



Figure 7. Vezin cutter aperture that is no longer radial due to poor maintenance.

Correct increment delimitation

One of most important requirements for correct *increment delimitation* is that 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. Furthermore, the length of the cutter aperture must be large enough to intercept all the material in the stream, including particles that bounce off the inside edges of the cutter aperture in the direction of its long axis.

The cutter aperture must also be designed so that the cutting time at each point in the stream is equal. To achieve this, the cutter lips must be parallel for linear-path cutters, while the cutter lips

must be radial for cutters travelling in an arc such as “Arcual” and “Vezin” cutters, where Arcual cutters rotate about their axis back and forth through the stream being sampled with the leading and trailing edges of the cutter completely clearing the stream at the end of each traverse while Vezin cutters rotate continuously in the one direction only. A correctly designed Vezin cutter with radial cutter lips is shown in Figure 6, while the original correct design of the Vezin cutter shown in Figure 7 has been compromised through poor maintenance practices, i.e. the cutter lips closer to the axis of rotation are no longer radial. An alternative radial cutter design that is also acceptable is the “rotating tube sampler”, which consists of a tubular distributor rotating around a vertical axis that feeds the material being sampled across a stationary radial cutter aperture as shown in Figure 8. In contrast, flap or diverter type cutters that divert one side of the stream for a longer period of time than the other do not satisfy the requirement that the cutting time at each point in the stream is equal and hence are also structurally unable to provide representative samples.

A further requirement is that the cutter must travel through the stream at a uniform speed, accelerating up to its cutting speed before entering the stream and decelerating to a stop only after



Figure 6. Example of a correctly designed radial Vezin cutter aperture just before interacting with the vertical falling stream of ore.

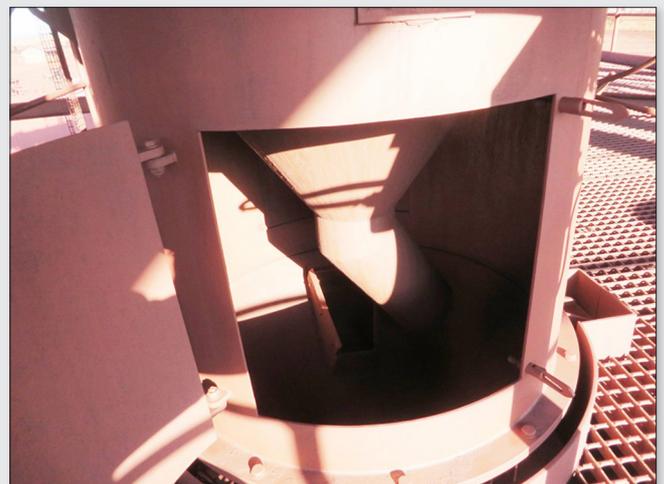


Figure 8. Example of a rotating tube sampler.



Figure 9. Belt scraper on a head pulley. The primary cutter needs to be moved closer to the head pulley or the cutter aperture extended to intersect all the belt scrapings.

leaving the stream cross-section, thereby ensuring that the cutting time at each point in the ore stream is equal. Consequently, the cutter drive must have sufficient power to ensure that the cutter does not slow down as it enters the stream and/or speed up as it leaves the stream. Electric cutter drives are best for ensuring uniform cutter speed, although hydraulic drives can also be satisfactory provided they are well maintained. Pneumatic drives are not recommended, because gas is compressible and hence it is usually impossible to adequately control the cutter speed.

If a belt scraper is required to remove material adhering to the belt, the scraped material must fall within the area traversed by the cutter, although in many instances the amount of material removed by belt scrapers is negligible, particularly for dry materials. A belt scraper installed on the head pulley in an iron ore sample station is shown in Figure 9. In this case the belt scrapings are quite significant and the primary cutter needs to be moved closer to the head pulley or alternatively the length of the cutter aperture extended to intersect all the belt scrapings.

Correct increment extraction

A key requirement for correct *increment extraction* is that 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. This is particularly important for so-called "reverse spoon" type cutters, where the material being sampled has to change direction as it strikes the back of the cutter body, which can cause sample reflux at high flow rates if the cutter does not have sufficient capacity. Furthermore, fine material that is damp has a tendency to hang-up in the cutter aperture, resulting in blockages and subsequent sample reflux. A bad case of sample reflux from a primary cutter in an iron ore sample station is shown in Figure 10. This problem can be overcome by incorporating generously large cutter bodies and chutes in the sample station design as well as setting the angle of the back of the cutter to deflect material down and away from the incoming stream (see Figure 11), thereby avoiding sample reflux and overflow from the cutter aperture. It should be noted, however, that the material removed by the belt scraper in Figure 11 is not sampled by the cutter in this case, so there is still room for improvement in this particular design. For sticky materials,



Figure 10. Massive reflux from a poorly designed primary cutter aperture at high flow rates.

steep chute angles ($>60^\circ$) and stainless steel or polythene chute linings are generally used to reduce adhesion, and the cutter aperture is often increased above the minimum to prevent bridging of the aperture.

An additional important requirement is that the cutter aperture must be at least 3 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 fine dry solids⁴. However, the cutter aperture is often significantly increased above this minimum to make absolutely sure that no large particles are excluded from the sample. In addition, the cutter should intersect the stream either in a plane normal to, or along an arc normal to, the mean trajectory of the stream to reduce the distance that particles bounce along the length of the cutter aperture after striking the inside edge of the cutter lips and consequently need to be collected as part of the sample. Notwithstanding this, the plane of the cutter aperture must not be vertical or near vertical, because particles that strike the inside edge of the cutter aperture and which should therefore end up in the sample are deflected downwards and away from the



Figure 11. Cross-stream cutter with a large cutter body to eliminate sample reflux at high flow rates.

cutter aperture by gravity into the reject stream, resulting in sample loss^{1,2,6}.

The cutter speed is also an important consideration and according to Gy⁴ must not exceed 0.6 m/s unless the cutter aperture exceeds 3d, because the “effective” cutter aperture decreases as the cutter speed increases, leading to the preferential exclusion of the coarser particles and hence the introduction of bias. However, Gy and Marin¹⁰ showed experimentally on a sample of calcined bauxite at low flow rates that when the cutter aperture (w) is increased above the minimum cutter aperture w_0 (i.e. 3d or 10 mm, whichever is the greater), the maximum cutter speed (v_c) could be increased as follows, subject to an absolute maximum of 1.2 m/s:

$$v_c = 0.3 \left(1 + \frac{w}{w_0}\right) \quad (1)$$

Notwithstanding the above relationship, the maximum cutter speed is usually limited to 0.6 m/s in the design of sample stations to allow for the high ore flow rates now routinely encountered and provide a reasonable safety margin.

Recently a few criticisms have appeared in the literature, and at World Sampling and Blending conferences, that the above cutter speed stipulations developed by Gy⁴ may not always apply as the variety and type of materials sampled around the world increases. Needless to say there may be exceptions for some materials and flow regimes, but the above cutter speed guidelines are designed to provide a safe approach to sample station design for the majority of materials and applications.

While cutter-chute type sample cutters need to be designed to be non-restrictive and self-clearing, bucket-type cutters must have sufficient capacity to accommodate the entire increment mass extracted at the maximum flow rate of the stream without any reflux or overflow of sample from the cutter aperture. In addition, care needs to be taken to ensure that the gate on the bottom of the cutter bucket does not jam in the open position while traversing the stream or in the closed position when parked, and that no sample is lost from the bucket during each traverse. An example of a poorly designed and maintained bucket-type cutter used for secondary sampling is shown in Figure 12. The gap between the gate and the



Figure 12. Poorly designed and maintained secondary cross-stream bucket cutter resulting in sample loss during its traverse.

bottom of the cutter is much too large and part of the sample collected is lost while traversing the stream.

The final design requirement for sample cutters is that no materials other than the sample must be introduced into the cutter or the sample delivery chute, and there must be no loss of sample from the sample delivery chute(s) or change in quality of the sample. If necessary, the sample cutter needs to be covered in the parked position between increments to prevent ingress of dust or spillage from within the sample station. Furthermore, possible sample loss due to the action of wind and air currents in sample stations needs to be eliminated by sealing and/or covering feeders, crushers, sample transfer conveyors and chutes, particularly when fine particles are being sampled, and any holes in chutes need to be rectified without delay to eliminate sample loss.

Cross-belt cutters

The sample cutters discussed so far have been cross-stream cutters where the cutter passes through a falling stream at the discharge point of a conveyor belt, chute or pipe. Provided suitable access is provided, it is reasonably straightforward to visually check that the cutter intercepts the complete stream and that increment delimitation and extraction are correct, thereby providing confidence that the samples collected are representative.

On the other hand, cross-belt cutters that take samples directly off conveyor belts are also used in the mineral industry. However, it is virtually impossible to check visually whether cross-belt cutters are operating correctly and remove a complete and correctly delimited cross-section of material from the conveyor belt. Consequently, while they may be less expensive to install than cross-stream cutters, cross-belt cutters have major deficiencies and are not recommended for the following reasons, particularly for high capacity streams:

- Cross-belt cutters tend to leave a layer of material on the conveyor belt if the profile of the conveyor belt is not matched to the path of the tip of the cutter or the skirts at the bottom of the cutter are not correctly adjusted as they gradually wear out. Furthermore, the wear of the skirts may not be uniform, resulting in gaps between the tip of the cutter and the conveyor belt, and maintenance staff often deliberately increase the gap between the cutter skirts and the conveyor for fear of damaging the conveyor belt. In each case the increment extraction is incorrect. Consequently, cross-belt cutters can be seriously biased, because the material on the bottom of the belt can be different in grade from the bulk of the material on the conveyor belt.

- As already pointed out above, it is virtually impossible to check visually whether a cross-belt cutter is performing correctly in terms of correct increment delimitation and increment extraction.

A typical example of a cross-belt cutter installation is shown in Figure 13. For safety reasons the cutter is fully enclosed, so it is impossible to visually check its operation. Figure 14 shows an ore stream after taking a cross-belt sample cut using a similar cutter to that in Figure 13, which indicates that almost certainly the cutter did not remove a full cross-section of ore from the conveyor belt, while Figure 15 is a photograph of an actual cross-belt cutter showing the poor condition of the rubber skirt on the bottom of the cutter. The sample cut shown in Figure 14 is clearly unsatisfactory. Cross-stream cutters must therefore be recommended in preference to cross-belt cutters to be sure of obtaining representative samples.



Figure 13. Typical fully enclosed cross-belt sampler installation. While 'hidden' from view, the very poor sampling performance with respect to extraction of a complete cross-section of the stream remains.



Figure 14. Sample cut taken by a cross-belt sampler indicating that a full cross-section of ore was not removed from the conveyor belt.



Figure 15. Poor condition of the rubber skirt on the bottom of a cross-belt cutter.

Increment mass

Returning to cross-stream cutters, the increment mass m_i (kg) collected by such cutters is determined by the cutter aperture A (m), the cutter speed v_c (m/s), and the flow rate of the stream G (tonnes/hr) as follows:^{8,11}

$$m_i = \frac{GA}{3.6 v_c} \quad (2)$$

Consequently, for a given flow rate, the smallest increment mass that can be taken and conform to correct sampling principles is determined by the minimum cutter aperture (3d) and the maximum cutter speed (usually limited to 0.6 m/s). While increments of larger mass can be taken using a larger cutter aperture and/or a lower cutter speed, it is not possible to take unbiased increments of smaller mass unless the flow rate is reduced or the material being sampled is crushed prior to sampling so that the cutter aperture can be safely reduced. Sample stations therefore need to be designed taking into account these important requirements. Contrary to what may be found in a number of old national and international sampling standards, there is no absolute minimum increment mass for a given particle size, just the correct increment mass determined by the flow rate, cutter aperture and cutter speed.

The "extraction ratio" is a very useful parameter for checking the design and operation of sample cutters,¹² ie, the ratio of the actual increment mass collected to the calculated increment mass using equation (2). If this ratio is significantly less than one, then the cause needs to be identified and corrective action taken to rectify the problem. Possible problems include reflux from the cutter aperture, hang-up in the cutter chute due to capacity problems or blockages in the cutter chute. The extraction ratio should be determined as a function of flow rate, because problems with reflux and hang-up in cutters become more serious as the flow rate increases.

Minimum sample mass

In contrast to increment mass, there is a minimum sample mass that needs to be extracted and retained for a given particle size to control the fundamental error variance⁴, which is determined by the particulate nature of the material being sampled, in particular the variation in quality between individual particles. Clearly, the fundamental error variance can be progressively reduced by including more and more particles in the sample that is collected, i.e. by increasing the sample mass. This is a very important sampling requirement, *which applies and needs to be checked at every stage of the sampling flowsheet*, i.e. at the primary, secondary, tertiary and if necessary quaternary stages of sampling, to ensure that the total sample mass collected at each stage meets the minimum requirement for the particle size at that stage.

However, unfortunately the minimum sample mass requirements are often ignored in the design of sample stations to reduce the masses that sampling personnel need to carry back to the sample preparation laboratory. While this might be desirable from the occupational health and safety perspective, it will seriously compromise the integrity of the sample. The correct approach is to crush the sample to a smaller particle size, thereby enabling the sample mass to be reduced by correct sample division (sub-sampling). An alternative is to provide mechanical lifting aids for sampling personnel to avoid the appalling situation shown earlier in Figure 2 where part of the sample is tipped out on the ground before taking the remaining sample material back to the laboratory for analysis.

There are several ways of determining the minimum sample mass that needs to be retained for a given particle size. One approach is to experimentally determine the precision by analysing replicate samples for a range of sample masses and particle sizes.¹¹ The relationship between sample mass and particle size that provides the required precision can then be plotted. An example of this approach may be found in ISO 3082 (Iron ores – Sampling and sample preparation procedures),¹⁴ where an equation and a table are provided for determining the minimum mass of divided sample as a function of nominal top size and division precision. While not as rigorous as for iron ore, the minimum sample mass requirements as a function of particle size for other commodities are specified in their respective national and international (ISO) standards, e.g. ISO 13909 (Hard coal and coke – Mechanical sampling – Part 2: Coal – Sampling from moving streams)¹⁶, and these mass requirements must be observed when designing sample stations. The alternative is to estimate the minimum sample mass from the well-known fundamental error (σ_{FE}) equation first derived by Gy⁴ and subsequently expanded on by Pitard⁶, ie, for a “binary” type ore when the divided sample mass is much less than the initial sample mass:

$$m_s = \frac{c \ell f g d^3 a^2}{\sigma_{FE}^2} \quad (3)$$

where m_s = divided sample mass (g)

σ_{FE} = fundamental error as a fractional concentration

c = mineralogical composition factor

ℓ = liberation factor

f = particle shape factor, which can usually be taken to be 0.5

g = size range factor, usually between 0.25 and 1.0.

d = nominal top size of the material (cm)

a = fractional concentration of the component of interest.

Further details on this approach together with worked examples are provided in text books by Gy⁴ and Pitard,⁶ as well as publications by other authors such as François-Bongarçon¹⁵ and Holmes.^{1,3}

Number of increments

Assuming the sample cutters have been designed to eliminate increment delimitation and extraction errors, and that the minimum sample mass requirements have been determined to reduce the fundamental error variance to acceptable levels, a sufficient number

of increments now need to be taken to reduce the long-range quality fluctuation error variance to the desired level. A number of methods are used to determine the required number of increments.

For iron ores, the standard deviation of individual primary increments within strata is determined experimentally using ISO 3084 (Iron ores – Experimental methods for evaluation of quality variation).¹⁷ This parameter is known as the quality variation σ_w , and the number of increments n required to achieve the desired primary sampling precision β_s , i.e. $2\sigma_s$, is calculated using the following equation:

$$n = \left(\frac{2\sigma_w}{\beta_s} \right)^2 \quad (4)$$

The ISO standard for sampling iron ore (ISO 3082)¹⁴ also provides a table specifying the minimum number of primary increments required to achieve the required sampling precision for large, medium and small quality variation, which is reproduced in Table 1.

On the other hand, the required number of increments for sampling coal is determined experimentally from the variance V_i of successive primary increments using the method specified in ISO 13909-2.¹⁶ The number of primary increments n to be taken from each sub-lot is then calculated for the desired overall precision P_L (95% confidence limit) after correcting for the sample preparation and analysis variance V_{PT} using the following equation:

$$n = \frac{4V_i}{mP_L^2 - 4V_{PT}} \quad (5)$$

where m is the number of sub-lots in the lot

V_{PT} is the preparation and analysis variance

While the above methods for determining the number of increments differ in detail, the general approach is similar, i.e. the required number of increments is determined by dividing the variance between individual increments by the required sampling variance.

In principle, the same approach can be used at the secondary, tertiary and quaternary sampling stages, and this approach is described in detail in ISO 12743 (Copper, lead, zinc and nickel concentrates – Sampling procedures for determination of metal and moisture content),¹⁸ but the variance of individual cuts is hardly ever determined. Instead, the number of cuts is usually set at a minimum

Table 1. Example from ISO 3082 of the minimum number primary of increments required to achieve specific sampling precisions (sS) for iron ore¹⁴.

Mass of lot (1000 t)		Sampling precision (σ_s)			Number of primary increments		
Over	Up to	Fe, SiO ₂ or moisture content	Al ₂ O ₃ content	P content	Quality variation Large (L), Medium (M) or Small (S)		
					L	M	S
270		0.155	0.045	0.00115	260	130	65
210	270	0.16	0.045	0.0012	240	120	60
150	210	0.17	0.05	0.00125	220	110	55
100	150	0.175	0.05	0.0013	200	100	50
70	100	0.185	0.055	0.00135	180	90	45
45	70	0.195	0.055	0.00145	160	80	40
30	45	0.21	0.06	0.00155	140	70	35
15	30	0.225	0.065	0.0017	120	60	30
0	15	0.25	0.07	0.00185	100	50	25

of approximately four, although in many cases Vezin dividers are used for subsequent sampling stages, particularly at the tertiary and quaternary stages, and hence in practice a much larger number of cuts are taken.

Sampling regime

Assuming that the sample cutters to be used are correctly designed, that the relationship between particle size and minimum sample mass has been established for the desired fundamental error has been established for sample division, and that the number of increments required to achieve the required sampling precision has been determined, there are usually a range of sampling regimes that can be successfully used in sample station design.

The usual strategy after collecting the primary increments is to crush the increments first so that they can then be safely divided down to a smaller sample mass. However, it may be beneficial to divide primary increments down to a smaller sample mass first, particularly when sampling high capacity streams where the primary increment mass can be quite large (possibly as much as 1,000 kg or more), provided of course that the minimum sample mass requirement for the composite sample comprising all increments is respected. This approach can be beneficial in reducing the load on crushers in the sample station, thereby reducing wear and tear and significantly reducing the need to adjust crusher gaps to ensure that the required particle size of crusher products meets design specifications. This is particularly important in sample stations, because if the particle size of the crusher product increases due to wear and tear and is not re-adjusted, then the sample mass retained after crushing and subsequent division will almost certainly not meet the minimum sample mass requirements for the larger particle size. This is a common fault in sample stations, so crushers need to be selected that can comfortably perform their duty and their performance needs to be carefully monitored and adjustments made if required.

As alluded to above, it is critical that the minimum sample mass requirements be respected at all subsequent sampling stages as well, e.g. that the sum of the masses of all secondary increments collected to constitute an analysis sample meets the minimum sample mass requirements for the particle size of the material at that stage which may be preceded by a crushing step so that the sample mass can be safely reduced. It is also good practice to ensure that the secondary, tertiary and if required quaternary cutters are triggered to take their first cut independent of the timing of operation of the preceding cutter.

Example. As an example of the design of a sampling regime, assume that a 180,000 tonne shipment of iron ore lump is being sampled according to ISO 3082.¹⁴ The particle size of the ore is $-31.5 + 6.3$ mm and the quality variation is assumed to be "small". Hence, according to Table 1, 55 primary increments are required.

(a) Primary stage

The primary cutter is a cross-stream sample cutter

Flowrate = 12,000 tonnes/hr

Cutter aperture = 0.15 m

Cutter speed = 0.4 m/s

From equation 2, the primary increment mass = 1,250 kg
Consequently, the total sample mass collected at the primary stage = $1,250 \times 55 = 68,750$ kg, which far exceeds the minimum sample mass of 180 kg in ISO 3082¹⁴ for a division precision of 0.1% Fe (see Table 2).

Table 2. Examples from ISO 3082 of minimum mass of divided gross sample for moisture and/or chemical analysis of iron ore¹⁴.

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
0.50	0.5	0.5
0.25	0.5	0.5

(b) Secondary stage

Because the primary sample mass is very large, use a cross-stream sample cutter taking 5 cuts from each primary increment to reduce the sample mass without crushing.

Flowrate = 10t/hr to completely clear the sample station between primary increments

Cutter aperture = 0.15 m

Cutter speed = 0.4 m/s

From equation 2, the secondary increment mass = 1.04 kg
Consequently, the total sample mass collected at the secondary stage = $1.04 \times 5 \times 55 = 286$ kg, which safely exceeds the minimum sample mass of 180 kg for a nominal top size of 31.5 mm in ISO 3082¹⁴ for a division precision of 0.1% Fe (see Table 2).

(c) Tertiary stage

Because the sample mass cannot be reduced much below 286 kg at a nominal top size of 31.5 mm, a cone crusher is used to reduce the nominal top size of the lump ore to 6.3 mm prior to division using a Vezin divider with a single radial cutter dimensioned to extract 5% of the sample fed to the divider.

Total secondary sample mass = 286 kg

Nominal top size after crushing = 6.3 mm

Consequently, the total divided sample mass at the tertiary stage = $286 \times 0.05 = 14.3$ kg, which safely exceeds the minimum sample mass of 3.2 kg for a nominal top size of 6.3 mm in ISO 3082¹⁴ (see Table 2).

This sample mass is also suitable for transfer to the laboratory for subsequent sample preparation and analysis.

Performance verification

Verification of the correct performance of sample stations is an important part of initial and ongoing quality assurance. For this purpose, comprehensive check lists are available,¹² including in a number of ISO standards, eg, for iron ore¹⁴ and coal and coke.¹⁹ Consequently, as pointed out by Pitard,⁷ large and readily accessible inspection ports are required to enable inspection of sample cutters to ensure that they intercept the whole stream and are in good condition and free of build-up and blockages. Unfortunately, practical experience indicates that this is not always the case and inspection ports are often non-existent, inconveniently and/or inappropriately located, or bolted shut on safety grounds. However,



Figure 16. Excessive sample build-up and partial blockage of a secondary cutter aperture.



Figure 17. Duplicate sampling system installed in a sample station.

safety concerns associated with inspection ports can be overcome by installing steel mesh on the inside of inspection ports behind the access doors to prevent physical access. This enables inspection of cutters “live” as they intercept the stream to validate correct operation, provided of course that the steel mesh provides good visibility of cutter operation. Inspection ports should also be provided for checking chutes, crushers and sample dividers, such as Vezin dividers and rotating tube samplers, for blockages and maintained condition. An example of excessive sample build-up and partial blockage of a bucket cutter is shown in Figure 16. Furthermore, the ability to monitor increment mass and/or the extraction ratio also provides valuable information for checking performance.

When conducting routine inspections and verifying the performance of sample stations, the key items that need to be checked include the following:

- The size and geometry of cutter apertures, including checking that cutters take a complete cross-section of the stream being sampled
- The cutter speed and its uniformity while cutting the ore stream
- The condition of cutter lips, including identifying any missing cutter lips
- The presence of build-up on cutter lips and/or blockages in cutter apertures and chutes
- Sample reflux from cutter apertures, particularly at high flow rates and for fine moist materials
- Ingress of extraneous material into the cutter aperture when the cutter is parked
- The location of belt scrapers and whether material removed by belt scrapers is significant and if so intercepted by the sample cutter
- The increment mass and whether it corresponds with the calculated increment mass
- The number of primary, secondary and tertiary cuts depending on the number of sampling stages
- Holes in cutters, chutes and bins, as well as the action of excessive air currents or wind, resulting in sample loss
- Crusher performance, in particular blockages and whether the product particle size conforms to specification
- The condition of vibratory feeders

- Sample mass as a function of particle size at each sampling stage to ensure that it conforms to minimum sample mass requirements.

Overall precision

The overall precision of sampling, sample preparation and analysis must be appropriate for the required task and decided at the outset so that an appropriate sampling regime can be designed. For example, it is impossible to control plant, stockpile or shipment grades to high precision if the overall precision of measurement is poor and instances of plant operators responding to apparent changes in grade that are no more than measurement “noise” are not uncommon. Furthermore, target grades can be moved closer to contract specifications without incurring penalties if the overall precision of grade measurements is high, thereby significantly improving resource utilisation.

The actual precision achieved in practice can be determined via duplicate “interleaved” sampling, where alternate primary increments are directed to duplicate samples A and B, which are subsequently prepared and analysed in duplicate under strictly identical conditions.²⁰ This enables separate estimates of the precision of sampling, sample preparation and analysis to be obtained. Consequently, duplicate sampling facilities should be incorporated into sample stations at the outset (see Figure 17) so that the precision achieved in practice can be determined and monitored on an ongoing basis. A number of well-designed, efficient and user-friendly approaches to this type of “agreement analysis” are available²¹, which enable a full range of precision assessments to be made for quality control purposes.

Conclusion

Accurate sampling practices are critical for characterising ores and mineral products in the mineral industry for resource evaluation, resource utilisation, feasibility studies, process design and optimisation, quality control, metallurgical accounting, and ultimately commercial sales. Sampling is the first step in the measurement chain, so if the sample that is collected is not representative, the whole measurement chain is compromised at the outset. On the other hand, it is still surprising how often sampling is entrusted to personnel who are not appropriately trained or do not fully

appreciate its importance, and everyone seems satisfied as long as just some material is collected and dispatched to the laboratory for analysis. Cost is often the overriding consideration instead of sampling correctness (unbiasedness) when designing sample stations, which is unacceptable. Sampling experts need to be fully involved in the design of sample stations and have the final sign-off to avoid structural flaws and the subsequent need for expensive retrofits to address major problems. Provision also needs to be made for duplicate sampling to monitor the precision achieved in practice on an ongoing basis for comprehensive quality assurance. After commissioning sample stations, regular performance audits need to be conducted to ensure they are adequately maintained and continue to conform to correct sampling principles. It is high time that sampling is given the necessary attention by company management right through to sample station operators as the first critical step in the quality measurement chain.

References

1. R.J. Holmes, "Correct sampling and measurement – The foundation of metallurgical accounting", *Chemometrics and Intelligent Laboratory Systems*, **74**: 71-83 (2004).
2. R.J. Holmes, "Sampling mineral commodities – the good, the bad, and the ugly", *Journal of the Southern African Institute of Mining and Metallurgy*, **110**: 1-8 (2010).
3. R.J. Holmes, "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) (2005).
4. P.M. Gy, *Sampling of Particulate Materials - Theory and Practice*, 2nd Edition (Elsevier: Amsterdam) (1982).
5. P.M. Gy, "Sampling from high capacity streams", in *Proceedings First Australian International Bulk Materials Conference*, Sydney, Australia, pp 407-423 (1982).
6. F.F. Pitard, *Pierre Gy's Sampling Theory and Sampling Practice*, 2nd Edition (CRC Press Inc: Florida) (1993).
7. F.F. Pitard, "Sampling correctness – A comprehensive guideline", in *Proceedings Second World Conference on Sampling and Blending (WCSB2)*, Sunshine Coast, Australia, pp. 55-66 (The Australasian Institute of Mining and Metallurgy: Melbourne) (2005).
8. R.J. Holmes, "Sampling", Chapter 4 of "An Introduction to Metal Balancing and Reconciliation", pp. 141-170 (Julius Kruttschnitt Mineral Research Centre, The University of Queensland) (2008).
9. R.J. Holmes, "The Importance of Sampling in the Mineral Industry", in *Proceedings MetPlant 2013*, Perth, Australia, pp. 34-49 (The Australasian Institute of Mining and Metallurgy: Melbourne) (2013).
10. P.M. Gy and L. Marin, L, "Unbiased sampling from a falling stream of particulate material", *International Journal of Mineral Processing*, **5**: 297-315 (1978).
11. R.J. Holmes, "Best Practice in Sampling Iron Ore", in *Proceedings Third World Conference on Sampling and Blending (WCSB3)*, Porto Alegre, Brazil, pp.416-429 (2007).
12. J. Docherty, "Mechanical sample plants", in *Proceedings Second World Conference on Sampling and Blending (WCSB2)*, Sunshine Coast, Australia, pp. 83-93 (The Australasian Institute of Mining and Metallurgy: Melbourne) (2005).
13. G.K. Robinson and R.J. Holmes, "Iron ores – Results of testwork on sample division for iron ores", ISO/TC 102 – Iron ores and direct reduced iron, *TC 102 Technical Committee Report No. 9*.
14. ISO 3082, Iron ores – Sampling and sample preparation procedures (ISO: Geneva) (2009).
15. D. François-Bongarçon, "The modelling of the liberation factor and its calibration", in *Proceedings Second World Conference on Sampling and Blending (WCSB2)*, Sunshine Coast, Australia, pp. 11-13 (The Australasian Institute of Mining and Metallurgy: Melbourne) (2005).
16. ISO 13909, Hard coal and coke – Mechanical sampling – Part 2: Coal – Sampling from moving streams (ISO: Geneva) (2009).
17. ISO 3084, Iron ores – Experimental methods for evaluation of quality variation (ISO: Geneva) (1998).
18. ISO 12743, Copper, lead, zinc and nickel concentrates – Sampling procedures for determination of metal and moisture content (ISO: Geneva) (1996).
19. ISO 21398, Hard coal and coke – Guide to the maintenance and inspection of sampling systems (ISO: Geneva) (2006).
20. ISO 3085, Iron ores – Experimental methods for checking the precision of sampling (ISO: Geneva) (2002).
21. M. Pitard, "Agreement analysis – Testing the boundaries between producers and consumers", *TOS Forum Issue 2*, pp. 12-15 (2014).