Sampling of Mineral Commodities – Where Everything Begins

Minerals Down Under

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CSIRO Minerals Down Under Flagship
Chair ISO/TC 102/SC 1 – Sampling Iron Ore
Chair ISO/TC 27/SC 4 – Sampling Coal and Coke
Introduction

• **Samples are taken for a number of purposes**
  - Exploration
  - Ore reserve/resource estimation
  - Mine development
  - Grade/quality control
  - Commercial transactions

• **Sampling locations include:**
  - Drill holes
  - Blast holes
  - Feed and product streams
  - Conveyors
  - Trucks and wagons
  - Stockpiles

• **Two important requirements**
  - Samples need to be *free of significant bias*
  - Overall precision needs to be adequate for the required task
Introduction

- Financial consequences of poor sampling practices can be huge
  - Poor investment decisions
  - Poor estimates of ore reserves/resources
  - Poor utilisation of ore resources
  - Reduced mine life
  - Poor recovery in processing plants
  - Metallurgical balancing problems
  - Loss of sales revenue

- Despite the serious financial consequences, poor sampling practices are still common in industry
  - Sampling is often left to personnel who do not understand its importance
  - In many instances, everyone seems to be happy as long as some material is sent back to the laboratory for analysis!!
Introduction

- Poor sampling practices need to be addressed
  - Ensure that sampling is given the attention it deserves
  - Improve staff training and awareness
  - Ensure samples are representative
- Otherwise the entire measurement chain is corrupted at the outset
- No point in accurately analysing samples in the laboratory if they are not representative in the first place
- Sampling is where everything begins
Golden Rule of Correct Sampling

- 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
- Need to eliminate key sampling errors such as:
  - Incorrect delimitation of increments
  - Incomplete extraction of increments
  - Preferential exclusion of specific size fractions
  - Sample loss and contamination
Components of Sampling Error

The key components of sampling error are as follows (Gy, 1982):

- Fundamental, grouping and segregation errors
- Long-range quality fluctuation error
- Periodic quality fluctuation error
- Weighting error
- Increment delimitation error
- Increment extraction error
- Accessory errors
Components of Sampling Error

- Some error components lead to bias and need to be eliminated
  - Accessory errors
  - Delimitation and extraction errors
  - Weighting errors
- The others need to be reduced to achieve acceptable precision
  - Fundamental, grouping and segregation errors
  - Long-range quality fluctuation error
  - Periodic quality fluctuation error
- Minimising or eliminating bias is critical, because bias cannot be eliminated once it is present
  - No point in being “precisely incorrect”
  - Sources of bias that can be eliminated
    - Sample spillage and contamination
    - Incorrect delimitation and extraction of increments
  - Sources of bias that need to be minimised
    - Change in moisture content
    - Dust loss, etc
Accessory Errors

- Sample contamination
- Loss of sample material
- Alteration of chemical composition
- Loss of moisture
- Particle degradation
- Operator mistakes
- Fraud and sabotage

Hole in side of chute

Moisture loss

Sample loss due to wind
Delimitation and Extraction Errors

- Delimitation and extraction errors arise from incorrect increment delineation/sample cutter design and operation.
- The delimitation error is eliminated if increments are correctly delineated, e.g., a parallel section of ore on a conveyor belt or radial cutter lips on a Vezin divider.
- The extraction error is eliminated if increments are completely extracted without any sample loss, e.g., no reflux from the cutter aperture.
Sampling Drill Holes

- Drill hole types
  - Diamond core
  - Conventional percussion
  - Reverse circulation (RC)
  - Blast hole

- Need to extract the full vertical section without sample loss, e.g., due to dust loss or fractures in the ground

- RC drilling better than conventional percussion drilling

- Automatic sampling of cuttings OK, provided all the cuttings are collected and passed through the sample divider
Blast Hole Sampling

- Blast holes of varying sizes are sampled
Blast Hole Sampling

- Cones are often poorly formed and quite heterogeneous
Blast Hole Sampling

• Vertical slices or channel samples are currently taken, but problems with:
  • Segregation of drill cuttings
  • Non-uniform thickness of cuttings
  • Allowance for sub-drill
• Extraction of a number of complete “sectors” of the blast hole cone is a better method
Blast Hole Sampling

- Sampling using sector sample trays
Blast Hole Sampling

- RC drilling and automatic sampling
Sampling from Moving Streams

- The discharge end of a conveyor is the best location
- The cutter must take a complete stream cross-section
- The cutting time at each point in stream must be equal
- The cutter should intersect the stream in a plane normal to the stream trajectory
- The sample cutter must be non-restrictive, self-clearing and discharge completely each increment
- The plane of the cutter aperture must not be vertical
- The cutter speed must be uniform
- The cutter aperture must be at least 3d
- The cutter speed must not exceed 0.6 m/s unless the cutter aperture exceeds 3d
- Bucket-type cutters must have sufficient capacity
- Material from belt scrapers must be sampled
- There must be no contamination of the sample or change in its quality
Sampling from Moving Streams

- The cutter must take a complete stream cross-section of the stream.

(a) Taking all of the stream part of the time

(b) Taking part of the stream all of the time (always incorrect)

(c) Taking part of the stream part of the time (always incorrect)
Sampling from Moving Streams

• Sampling from the top of a conveyor belt does not provide a representative sample
Sampling from Moving Streams

• The discharge point of a conveyor belt is the most suitable sampling location, but a complete cross section of the ore stream must be taken.
Sample Cutter Design

- Correct sample cutter design is critical
Sample Cutter Design

- Large cutter body and streamlined design required to eliminate reflux at high flow rates
Sample Cutter Design

- Vezin samplers/dividers need to have radial cutter lips
Sample Cutter Design

• Vertical cutter lips must be avoided
Sample Cutter Design

- Poorly designed secondary cutter bucket resulting in sample loss
Sample Cutter Design

- Partially blocked cutter chute causing sample reflux
Sample Cutter Design

- Belt scrapers need to be located so that the scrapings are intersected by the sample cutter
Sample Cutter Design

Good

Poor

Bad
Cross-stream vs Cross-belt Cutters

• **Cross-stream cutters**
  - Relatively easy to visually check correct operation, ie, correct increment delineation and extraction
  - Much less expensive than bias tests

• **Cross-belt cutters**
  - Very difficult to check visually, so bias tests required
  - Often leave a layer of material on the conveyor belt due to wear/incorrect adjustment of skirts on bottom of cutter
  - Not recommended for high capacity iron ore streams
Cross-belt Cutters

- Range of different designs, some of which are extremely bad
Sampling Dry Concentrate

- Sampling dry concentrate pneumatically from the side of a feed tube does not extract a full cross-section of the concentrate stream
Sampling Slurries

• As for particulate material, a complete cross-section of the slurry stream needs to be taken.
Sampling Slurries

- Taps from the side of pipes and pressure pipe samplers are incorrect and do not extract a full cross-section of the slurry stream.
Sampling from Railway Wagons

- Sampling from the tops of railway wagons does not provide representative samples
Sampling from Vessels

• Dip sampling does not extract a full vertical cross-section of ore, concentrate or slurry from the vessel
Sampling from Stockpiles and Ships

• Impossible to take a representative sample in-situ from a stockpile or ship

• Samples must be taken:
  • While the stockpile is being built up or broken down
  • While the ship is being loaded or unloaded
The fundamental error variance $\sigma_{FE}^2$ identified by Gy is due to the particulate nature of the material being sampled and is given by:

$$\sigma_{FE}^2 = \frac{Cd^3a^2}{m_S}$$

where $C = \text{the sampling constant}$
$d = \text{nominal top size (cm)}$
$m_S = \text{divided sample mass (g)}$
$a = \text{fractional concentration}$

The fundamental error leads to the minimum sample mass to achieve the required precision by transposing the above equation:

$$m_S = \frac{Cd^3a^2}{\sigma_{FE}^2}$$
Fundamental Error

- The sample mass cannot be reduced below the minimum sample mass for a given precision until the sample is crushed.
- This is a critical sampling requirement that must not be ignored if good division precision is required.
- While in principle the minimum sample mass can be calculated, the best approach is to determine the minimum sample mass experimentally.
- Test programs have been conducted by several ISO subcommittees to establish the minimum sample mass as a function of nominal top size and required precision:
  - ISO/TC 27/SC 4 (Sampling coal and coke)
  - ISO/TC 102/SC 1 (Sampling iron ores)
- In the case of ISO/TC 102/SC 1, a comprehensive international test work program was conducted, the main contributors being:
  - Australia
  - Brazil
  - Japan
Minimum Sample Mass

- Minimum mass of divided sample for iron ore from ISO 3082

<table>
<thead>
<tr>
<th>Nominal top size (mm)</th>
<th>Minimum mass of divided gross sample (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$\sigma_D = 0.1%$ Fe</td>
</tr>
<tr>
<td>40</td>
<td>325</td>
</tr>
<tr>
<td>31.5</td>
<td>180</td>
</tr>
<tr>
<td>22.4</td>
<td>75</td>
</tr>
<tr>
<td>10</td>
<td>10</td>
</tr>
<tr>
<td>6.3</td>
<td>3.2</td>
</tr>
<tr>
<td>2.8</td>
<td>0.5</td>
</tr>
<tr>
<td>1.4</td>
<td>0.5</td>
</tr>
<tr>
<td>0.50</td>
<td>0.5</td>
</tr>
<tr>
<td>0.25</td>
<td>0.5</td>
</tr>
</tbody>
</table>

- The masses specified in ISO 3082 are conservative, because the ISO Standard is designed to cover iron ores from around the world, including those with low grade and high constitution heterogeneity.
Minimum Sample Mass

- Minimum sample mass requirements apply at each stage of sampling/sample preparation

- Vigilance required to keep sample masses above the “safety line”
Sample Preparation

• There must be no loss of sample from conveyors or chutes
• Crushers must have sufficient capacity
• Minimum divided sample mass requirements must be met
• Other sample preparation issues to be avoided
  • Discarding part of the sample, eg, too heavy
  • Incorrect use of sample dividers, eg, riffles
  • Sample loss in dust extraction systems
  • Cross contamination between samples
Robotic Sample Preparation

- Robotic sample preparation systems are increasingly being used to overcome occupational health and safety restrictions on sample masses
- Still need to be carefully designed to meet all the requirements for correct sample preparation, eg,
  - Particle size after crushing
  - Minimum sample mass requirements
  - Minimal sample loss in rotary driers
  - Minimal cross contamination of samples
System Verification

• Need to be able to readily verify conformance to correct design principles
• Need large and easily accessible inspection hatches
• Monitoring sampling ratio and increment mass/extraction ratio also provides valuable information
System Verification

- Key items that need to be checked include:
  - Cutter speed
  - Uniformity of cutter speed while cutting the ore stream
  - Number of cuts
  - Size and geometry of cutter apertures
  - Worn and/or missing cutter lips
  - Build-up and/or blockages in cutter apertures and chutes
  - Reflux from cutter apertures
  - Ingress of extraneous material when the cutter is parked
  - Holes in chutes and bins resulting in sample loss
  - Increment/sample mass
  - Particle size
Precision Achieved in Practice

- The actual precision achieved in practice can be determined via duplicate “interleaved” sampling.
- One approach is to use ISO 3085 (Iron ores – Experimental methods for checking the precision of sampling, sample preparation and analysis).
  - Alternate primary increments from at least 20 lots are directed to duplicate subsamples A and B.
  - Subsamples prepared and analysed in duplicate to determine the separate precisions of sampling, sample preparation and analysis.
Iron Ore Case Study

- ISO 3082 (Iron ores – Sampling and sample preparation procedures) was developed by international consensus between iron ore producers and consumers by ISO/TC102/SC 1.

- Apart from specifying design criteria for sample cutters, the Standard specifies target values for sampling precision and thus the number of primary increments for ores with “large”, “medium” and “small” quality variation.
Iron Ore Case Study

- The quality variation, \( \sigma_W \), is defined as the standard deviation of a given constituent within sampling strata, i.e., the short range standard deviation of primary increments.
- For Fe content, the \( \sigma_W \) ranges for high, medium, and small quality variation are >2.0% Fe, 2.0-1.5% Fe, and <1.5% Fe, respectively.
- To optimise a sampling regime, the actual value of \( \sigma_W \) should be measured using the procedures specified in ISO 3084 (Iron ores – Experimental methods for determining quality variation).

<table>
<thead>
<tr>
<th>Mass of lot (1000 t)</th>
<th>Sampling precision (( \sigma_S ))</th>
<th>Number of primary increments</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Fe, SiO(_2) or moisture content</td>
<td>Al(_2)O(_3) content</td>
</tr>
<tr>
<td>Over</td>
<td>Up to</td>
<td>L</td>
</tr>
<tr>
<td>270</td>
<td>270</td>
<td>0.155</td>
</tr>
<tr>
<td>210</td>
<td>270</td>
<td>0.16</td>
</tr>
<tr>
<td>150</td>
<td>210</td>
<td>0.17</td>
</tr>
<tr>
<td>100</td>
<td>150</td>
<td>0.175</td>
</tr>
<tr>
<td>70</td>
<td>100</td>
<td>0.185</td>
</tr>
<tr>
<td>45</td>
<td>70</td>
<td>0.195</td>
</tr>
<tr>
<td>30</td>
<td>45</td>
<td>0.21</td>
</tr>
<tr>
<td>15</td>
<td>30</td>
<td>0.225</td>
</tr>
<tr>
<td>0</td>
<td>15</td>
<td>0.25</td>
</tr>
</tbody>
</table>
Iron Ore Case Study

- For iron ore, the overall precision in ISO 3082 is expressed as:

\[
\sigma_{SPM} = \sqrt{\sigma_S^2 + \frac{\sigma_P^2}{k} + \frac{\sigma_M^2}{kr}}
\]

where \(\sigma_S\) is the primary sampling precision, \(\sigma_P\) is the sample preparation precision, \(\sigma_M\) is the measurement (analysis) precision, including selection of the final test portion, \(k\) is the number of sub-lot samples and \(r\) is the number of replicate analyses of each analysis sample.

- The target overall precision values \((1\sigma)\) specified in ISO 3082 are:

<table>
<thead>
<tr>
<th>Quality characteristic</th>
<th>Iron content</th>
<th>Silica content</th>
<th>Alumina content</th>
<th>Phosphorus content</th>
<th>Moisture content</th>
</tr>
</thead>
<tbody>
<tr>
<td>Mass of lot (tonnes)</td>
<td>(0.17)</td>
<td>(0.175)</td>
<td>(0.185)</td>
<td>(0.0017)</td>
<td>(0.17)</td>
</tr>
<tr>
<td>Over 270,000</td>
<td>(0.175)</td>
<td>(0.185)</td>
<td>(0.19)</td>
<td>(0.00175)</td>
<td>(0.175)</td>
</tr>
<tr>
<td>210,000 to 270,000</td>
<td>(0.185)</td>
<td>(0.19)</td>
<td>(0.20)</td>
<td>(0.00185)</td>
<td>(0.185)</td>
</tr>
<tr>
<td>150,000 to 210,000</td>
<td>(0.19)</td>
<td>(0.20)</td>
<td>(0.21)</td>
<td>(0.0019)</td>
<td>(0.19)</td>
</tr>
<tr>
<td>100,000 to 150,000</td>
<td>(0.20)</td>
<td>(0.21)</td>
<td>(0.225)</td>
<td>(0.0020)</td>
<td>(0.20)</td>
</tr>
<tr>
<td>70,000 to 100,000</td>
<td>(0.21)</td>
<td>(0.225)</td>
<td>(0.245)</td>
<td>(0.0020)</td>
<td>(0.21)</td>
</tr>
<tr>
<td>45,000 to 70,000</td>
<td>(0.225)</td>
<td>(0.245)</td>
<td>(0.275)</td>
<td>(0.0021)</td>
<td>(0.225)</td>
</tr>
<tr>
<td>30,000 to 45,000</td>
<td>(0.245)</td>
<td>(0.275)</td>
<td>(0.275)</td>
<td>(0.00225)</td>
<td>(0.245)</td>
</tr>
<tr>
<td>15,000 to 30,000</td>
<td>(0.275)</td>
<td>(0.275)</td>
<td>(0.275)</td>
<td>(0.0024)</td>
<td>(0.275)</td>
</tr>
<tr>
<td>Less than 15,000</td>
<td>(0.275)</td>
<td>(0.275)</td>
<td>(0.275)</td>
<td>(0.0024)</td>
<td>(0.275)</td>
</tr>
</tbody>
</table>
Iron Ore Case Study

- A total of 1,216 shipments from Australia were sampled routinely using duplicate interleaved sampling assuming “small” quality variation.
- The precisions of sampling, sample preparation and analysis were determined in accordance with ISO 3085 and were much better than the target values.

### Table 4. Precision achieved in practice for the Fe content of five different iron ore types at two different Australian ports.

<table>
<thead>
<tr>
<th>Ore Type</th>
<th>Port</th>
<th>( \sigma_S ) (%Fe)</th>
<th>( \sigma_P ) (%Fe)</th>
<th>( \sigma_M ) (%Fe)</th>
<th>( \sigma_{SPM} ) (%Fe)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>A</td>
<td>0.051</td>
<td>0.042</td>
<td>0.028</td>
<td>0.071</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>0.080</td>
<td>0.035</td>
<td>0.031</td>
<td>0.093</td>
</tr>
<tr>
<td>2</td>
<td>A</td>
<td>0.053</td>
<td>0.034</td>
<td>0.024</td>
<td>0.067</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>0.082</td>
<td>0.043</td>
<td>0.028</td>
<td>0.097</td>
</tr>
<tr>
<td>3</td>
<td>A</td>
<td>0.055</td>
<td>0.034</td>
<td>0.027</td>
<td>0.070</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>0.068</td>
<td>0.038</td>
<td>0.027</td>
<td>0.082</td>
</tr>
<tr>
<td>4</td>
<td>A</td>
<td>0.059</td>
<td>0.043</td>
<td>0.027</td>
<td>0.077</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>0.068</td>
<td>0.037</td>
<td>0.021</td>
<td>0.080</td>
</tr>
<tr>
<td>5</td>
<td>A</td>
<td>0.023</td>
<td>0.024</td>
<td>0.023</td>
<td>0.040</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>0.021</td>
<td>0.027</td>
<td>0.022</td>
<td>0.041</td>
</tr>
</tbody>
</table>

### Table 5. Comparison of precisions achieved in practice with target precisions in ISO 3082 for the Fe content of five different iron ore types at two different Australia ports.

<table>
<thead>
<tr>
<th>Precision measure</th>
<th>Target precision range (%Fe)</th>
<th>Precision range achieved in practice (%Fe)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Overall precision</td>
<td>0.185 – 0.245</td>
<td>0.040 – 0.097</td>
</tr>
<tr>
<td>Sampling precision</td>
<td>0.17 – 0.225</td>
<td>0.021 – 0.082</td>
</tr>
</tbody>
</table>
Conclusions

• Special attention needs to be given to sampling, sample preparation and analysis for resource development, quality control, metallurgical accounting and commercial transactions to ensure that:
  • The final analyses are unbiased
  • The analyses are sufficiently precise for their intended purpose

• To achieve this objective
  • Management commitment to correct sampling practices is required
  • Sampling equipment and procedures need to be designed to eliminate bias at the outset
  • Sampling regimes need to provide the required precision and be designed with duplicate sampling capabilities to enable measurement of the precision achieved in practice
  • Participate in ISO Standard development
Thank you