A new approach to implement quality assurance and quality control to technological innovations – quality of spectral data capture and processes in the minerals industry

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ABSTRACT

Technological innovations and emerging analytical capabilities are transforming the way drill samples are analysed, with real-time mineralogical analysis made possible. In addition, new business/technical requirements are challenging long standing processes to incorporate more sources of information during planning in order to improve compliance to plan during mining and processing. Unfortunately, the new sources of information that are added do not always have adequate protocols in place to ensure quality data is collected.

Spectral data covering the visible – near – short wave – midwave – longwave range are routinely collected by the Geoscience and Exploration department at BHP Billiton Iron Ore for accurate mineralogical identification. These data are used for geological interpretations (logging and modelling) and prediction of physical variables (metallurgical and geotechnical properties).

This paper introduces how sampling and quality assurance / quality control concepts have been applied in the spectral data process. The described routine for sampling, data collection/management and reconciliation has been developed to track system precision and accurately and consistently ensure the data is of high quality before it is used across the mining chain.

INTRODUCTION

Technology is defining new ways to satisfy the demand for fast and accurate geological information in the mining industry. Hyperspectral sensing in the near and long wave infrared is increasingly used for mineralogical characterisation by mining companies as an objective and cost-efficient alternative to X-ray diffraction or QEMSCAN analysis (eg Asadzadeh and Roberto de Souza Filho, 2016; Haest et al, 2012; Ramanaidou et al, 2008, 2015; Schodlok and Ramanaidou, 2011). Infrared spectral imaging of mineralogy from satellite or airborne platforms has been routine practice over the last two decades for mineral exploration and environmental surveys (eg Hewson et al, 2005; Van Ruitenbeek et al, 2012). Its routine application in laboratory analysis is gaining significant traction thanks to the introduction of new and fast infrared analysis tools (eg the Hylogger™ systems developed by the Commonwealth Scientific and Industrial Research Organisation).

Mineral characterisation from infrared spectra is based on absorptions of photons by electronic transitions or molecule bond vibrations/rotations at characteristic energies across the electromagnetic spectrum (Clark et al, 1990). The characteristic wavelength position, width and depth of these absorptions are referred to as spectral parameters. At BHP Billiton Iron Ore, these spectral parameters are used in conjunction with assays to produce mineral predictions using a set of automated algorithms (Haest et al, 2012; Haest, Vitins and Hacket, 2013).

The ability to quantitatively track spectral data quality and correct spectra for instrument drift is crucial for successful inference of geological information from these data (Kerekes, Cisz and Simmons, 2005; Haest, Mittrup and Dominguez, 2015; Laukamp et al, 2010). This paper presents an automated approach, using internal calibration standards and quality control samples, to track quality of the infrared lab data and correct the spectra for instrument drift, back to the historic average.

METHODOLOGY

The Geoscience and Exploration department has embedded full range visible to longwave infrared scanning of pulverised diamond core and pulverised chip samples from reverse
circulation (RC) drilling as a routine component in the BHP Billiton Iron Ore analytical suite.

All drill samples are crushed, pulverised and barcoded according to BHP Billiton Iron Ore standard practise. Infrared analysis and elemental analysis using X-ray fluorescence are completed on the same samples. These samples retain a unique sample number and are kept together in the same jobs to ensure an exact match between geochemical and infrared spectral data sets (Haest, Mittrup and Dominguez, 2015). In addition, pulverisation of all samples prior to infrared analysis:

- ensures seamless correlation between spectra collected from diamond core and RC chip samples
- removes the effect of grain size variations on spectral response, resulting in more accurate predictions of mineralogy and physical parameters (Asadzadeh and Roberto de Souza Filho, 2016)
- results in an optimal representative sample (rather than scanning of diamond or chip sample surfaces).

Infrared spectral data collection is performed with a Hylogger™ and with a Fourier transform infrared spectrometer (FTIR), capturing the visible-near to short wave (VNIR-SWIR) and the short wave, midwave to longwave (SWIR-MWIR-LWIR) respectively. The individual pulp samples for Hylogger™ analysis are sampled into standard chip trays, whereas FTIR samples are pressed into metal disks and analysed with the FTIR spectrometer. Both infrared and element analysis are completed at one commercial laboratory in Perth.

The quality assurance and quality control systems in place are the same as for sample collection and assaying done by the Geoscience and Exploration department of BHP Billiton Iron Ore. Company controls are inserted to monitor precision (duplicates), contamination (blanks) and accuracy (company standards). In addition, to control the infrared sensing process, spectralon plates (described below) are used to monitor the precision and accuracy of the Hylogger™ on a daily basis and to correct raw spectra back to the long-term historic average, in order for spectra to align through time in terms of wavelength and reflectance. The laboratory developed its own proprietary process to track the precision of the FTIR sensor, using a combination of assay prediction models of duplicates and routine samples, with automated comparison against actual X-ray fluorescence results for the same samples.

Duplicates and blank samples are also measured with the infrared spectral instruments for further data quality analysis.

**INFRARED QUALITY CONTROL PROCESSES**

Data validation is essential during infrared spectral data collection. A variety of error sources can result in large wavelength shifts, poor signal-to-noise ratios and spectrum offsets. Some of these sources of error include: poor light alignment, wear and ageing of parts, changes to environmental conditions such as temperature or humidity.

The precision of the Hylogger™ instrument is controlled on a daily basis by scanning doped spectralon, a mylar sheet draped over a spectralon plate, a multireflectance spectralon plate and a Munsell colour chart (Figure 1) at the start and end of each shift. The first three targets are mounted together for scanning efficiency, while the Munsell colour chart is scanned independently (Figure 2). These standards have the following uses:

**FIG 1** – Standard Spectralon plate covering absorption features in the VNIR-SWIR (visible and near-infrared–short wave infrared) wavelength range.

**FIG 2** – Munsell colour chart for the calibration of Hylogger™ high resolution red, green, blue (RGB) camera based on hue, intensity and saturation.
• doped spectralon – sharp absorption features in the VNIR and short wavelength range SWIR for tracking wavelength precision
• Mylar draped over spectralon – sharp absorption features in the long wavelength range SWIR for tracking wavelength precision
• Multireflectance spectralon plate – reflectance variations from near complete absorption to near complete reflection across the VNIR-SWIR in four steps (used to control reflectance levels)
• Munsell colour chart – track precision of the high resolution RGB camera.

To monitor the lab performance and tracking the stability of each individual analytical component of the system, a set of spectral parameters is extracted from these standard measurements. These are compared against expected values that are based on long-term averages of previous analyses. The precision of the three analytical components on the Hylogger™ system is assessed, including: VNIR-SWIR spectrometer, laser profile-meter and RGB image camera. Five main parameters are monitored consistently to capture system drift (Figure 3).

**Figure 3** – Tracking sheet for the assessment of the analytical components of the Hylogger™.
This short-term quality assurance / quality control (QA/QC) process can identify operative errors at the lab and ensures spectral data collection is within set specification before the onset of data collection. This process has proven quite robust over the last years, helping to rectify issues mostly related to instrument alignment.

The raw infrared spectra delivered by the laboratory are processed with further quality controls in place at BHP Billiton Iron Ore. The daily standard measurements over the VNIR to SWIR wavelength range are correlated against a set of reference standards developed for these standards. Calibration coefficients are extracted from this correlation and are used to adjust individual raw infrared spectral measurements scanned during that shift to align against the reference standard measurements. The differences are corrected through a linear wavelength shift and a mean reflectance dependent shift in absolute reflectance.

As with every instrument, the Hylogger™ undergoes regular preventative maintenance services. During instrument maintenance, a different sensor is used to continue data collection using the same spectrolon. Significant wavelength shifts were noted to no surprise, between the Hylogger™ standard measurements before, during and after instrument maintenance (Figure 4). The spectrolon data acquired from the substitute appliance was cross-calibrated to adjust shifts in band positions and upon return of the maintained (recalibrated) spectrometer, a different set of wavelength shifts were observed that needed to be cross-corrected as well. These wavelength shifts demonstrate the importance of having a robust QA/QC process in place.

Extensive quality checks for FTIR data have been developed by the laboratory, involving generic element prediction models and comparisons against results from element analysis. A final check before the data is released for mineralogy and geomeallurgical purposes involves the matching of the Hylogger™ and FTIR captured spectral parameters aligning in the SWIR range. The match over a specific set parameter is monitored within a specific tolerance to detect false spectral alignments between the two instruments that could result from either instrument having an error or (more likely) a mismatch between samples.

**USE OF INFRARED DATA FOR OTHER QUALITY CONTROL APPLICATIONS**

Coarse blanks are routinely used to detect Fe contamination in the standard X-ray fluorescence spectroscopy (assay) sample preparation process at laboratories. The red, green, blue (RGB) images collected with the Hylogger™ are utilised to confirm contamination of blanks detected through assay QA/QC procedures. For a contamination assessment, the

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**FIG 4** – Wavelength shifts of diagnostic absorption features (doped and mylar spectrolon plate) from before and after Hylogger™ maintenance.
RGB colour image is set to detect Fe contamination, which deviates from the greyish colour of the blank (Figure 5).

Sample sequencing errors can equally be validated using results from infrared analysis. For example, the blanks that are inserted in the process have quite unique mineralogy (chlorite and amphibole). When these minerals are detected for normal iron ore samples, which should be dominated by hematite, goethite, kaolinite and/or quartz, this is often a clear indication that a sample swap has occurred.

An automated process has been developed for the conversion of raw pulp spectra and laboratory assays into quantitative mineralogy. The mineralogy script precision and repeatability is determined by comparing the calculated mineralogy from the spectral infrared results of primary and field duplicates. By measuring the precision of assay reconciled spectral mineralogy, the duplicate precision (within ten per cent relative difference) is obtained for a combined geochemical/ infrared process, which is the ultimate input into geology and geometallurgy.

CONCLUSION
Implementation of a robust QA/QC process for infrared analysis at BHP Billiton Iron Ore has ensured accurate, consistent and reliable information can be extracted from this novel data set and can be applied across the mining value chain. Collection of spectral standards (wavelength and reflectance standards) form the corner stone of quality control in the visible-near to short wave infrared, while predictions of element analysis and mineralogy on samples, standards and blanks are used to check precision across the complete wavelength range (VNIR-SWIR-MWIR-LWIR).

Integrating increasingly higher resolution data sets such as spectral infrared and assays, and potentially geophysical information, benefits not only mineralogy determination for geological modelling, but it is also an effective evaluation across the business to help with geometallurgical analysis and resource evaluation estimates. To deal with these large and continuous streams of sample data, quality control is largely automated and consistency in terms of input data format is of fundamental importance.

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REFERENCES


