

Use of Field Portable X-Ray Fluorescence Spectrum Analysers for Grade Control — A Presentation of Case Studies

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ABSTRACT

This paper presents recent case study data collected by field portable x-ray fluorescence spectrum analysers (FPXRF) as part of grade control activities at three separate (nickel, copper and copper/gold) mining operations. FPXRF has been under utilised for grade control in recent years, largely because the success of early versions of FPXRF was limited to a few metals (eg tin). Efforts to utilise these early versions for other metals was largely unsuccessful and created a perception that FPXRF could not be utilised more broadly within the mining sector and specifically the area of grade control. The structural setting of the analytical function within mining operations has also contributed to the under utilisation of more recent FPXRF instrumentation. The concept of FPXRF as a grade control tool straddles the boundaries of the geology and laboratory departments. As such, this option is often not considered as part of an assessment of grade control procedures for new and existing mining operations. This paper assesses data collected by Australian and New Caledonian miners using FPXRF technology as part of the grade control function and concludes that FPXRF can provide operational and cost benefits to certain mining operations.

INTRODUCTION

Field portable x-ray fluorescence spectrum analysers (FPXRF) have played an integral role in the alloy, contaminated soil and lead paint industries for many years. These instruments have been shown to provide accurate, quantitative chemical analysis of metal alloy, bulk (soil and dust) and paint samples in less than 60 seconds. FPXRF delivers these industries rapid access to chemical data and as a consequence, significant project management and cost benefits. In contrast, FPXRF has been much less utilised in the mining industry. Through the presentation of case study data (from Ernest Henry Copper/Gold and Thalanga Copper Mines in North Queensland and the Falconbridge Koniombo Nickel laterite deposit in New Caledonia), this paper evaluates the potential of FPXRF technology to play a role in the area of mine grade control.

FPXRF AND MINING

Fixed laboratory x-ray fluorescence spectrum analysers (XRF) are a common laboratory tool that have been used globally for many decades within the mining and mineral exploration industries. Portable versions of these analysers were first released in the mid- to late-1960s principally for the differentiation of alloys, as part of positive material identification (PMI) regimes in plants and refineries. To date, they have been employed by very few mining and exploration companies.

The limited use of FPXRF in the resources sector is a result of a combination of factors. Early versions of FPXRF have been trialled over the last 20 - 30 years with very limited success. The failure of the early FPXRF units created a widespread perception within the industry that the technology was not capable of being successfully transferred to the mining industry and specifically

the area of grade control. This widely held view may have been valid for FPXRF instruments that were commercially available up until recently.

The limited use of FPXRF within the mining industry can also be understood by observing the structural setting of the analytical laboratory within the mineral extraction industry. Traditionally, the analytical laboratory provides data analysis services to various departments within any mining operation. The main recipients of the mine laboratory services are the metallurgical and geological divisions, with the laboratory generally under the supervision of the metallurgical department. Using sound economic rationale, metallurgical samples generally take precedence over geological samples. Accordingly, on-site laboratories tend to maintain a closer relationship with the metallurgy department and have a better understanding of that department's operational requirements.

Whether these analytical services are outsourced/contracted or the mine operates its own laboratory, the relationship is generally one of supplier and client. It is not commonplace in this relationship for the laboratory (or, in this case, the supplier) to review the methods employed by their client (in this case, the geology department) or to investigate potential strategies to improve established mine grade control procedures. This responsibility remains firmly with mine geologists. This area of potential use for FPXRF in mine grade control straddles the boundaries of the geology and laboratory departments and as such, may not always come under the singular scrutiny of staff investigating innovative methods to achieve improved operational efficiencies.

With the take up of any new technologies, there is a lag time between the development of the technology and a general awareness in the industry regarding the effectiveness and availability of the technology. When this 'technology lag' is combined with the past perceptions of FPXRF and the established structural setting of the analytical function within the industry, it is not difficult to observe the reasons why FPXRF has been under-utilised for grade control to this point in time.

RECENT FPXRF DEVELOPMENTS

All the case study work forming the bulk of this paper was undertaken using US manufactured NITON FPXRF instruments. Two different model FPXRF units were used, an XL-502S and an XLi 969. NITON instruments were first released in 1994, to analyse (*in situ*) the content of lead in painted surfaces. They were further developed to accurately determine the content of multi elements (including lead, copper, zinc and arsenic) in soil and other 'bulk' materials. The main feature of the technology is the ability to obtain chemical analysis of samples on-site, in real time in less than 60 seconds.

The principal market for portable XRF analysers in bulk materials is the area of contaminated soil assessment. The United States Environment Protection Agency (USEPA) formally recognised the use of FPXRF in the field of contaminated site assessment in 1998 by publishing USEPA Method 6200, 'Field Portable XRF Spectrometry for the Determination of Elemental Concentrations in Soil and Sediment' (USEPA, 1998). By the late-1990s, FPXRF technology was being utilised to investigate and remediate contaminated sites around the world. Australian

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examples have included sites owned by BHP, Dulux, Normandy, Orica, Carter Holt Harvey and the Department of Defence. A substantial body of accuracy and precision data has been published on the successful use of FPXRF in assessing contaminated soils (Argyaki, Ramsey and Potts, 1997; Davis and Bawden-Smith, 1999; Shefsky, 1997; Ridings, Shorter and Bawden-Smith, 1999).

To be of use to the contaminated soil industry, FPXRF instruments were required to be able to differentiate between 'clean' soil and contaminated material. To do this, software programs were developed to ensure instrument accuracy in the low parts per million (ppm) range, typically less than 100 ppm. However, these software programs were largely ineffective in soils and bulk materials at concentrations above one to two per cent due to the inability to automatically compensate for the variations in matrix typically encountered in different soil types. This includes variations in elemental ratios, bulk density and particle size.

In 2000, portable XRF were released with two different software programs for analysis of bulk materials. The original software program (Compton Normalisation or CN) for measuring multi elements at ppm levels was retained and a second program, Fundamental Parameters (FP) Bulk, introduced. FP Bulk used similar software to that used for alloy analysis, however could not automatically compensate for changes in the matrix type as CN could. However, the precision of the instruments is such that errors are generally linear, which allowed the introduction of a calibration capability.

As a result, FP Bulk was adapted to include a simple two-point calibration. This provided a simple technique that enabled the FPXRF to be calibrated to individual matrix (or ore) types. This is done by testing samples of known concentrations and effectively teaching the FPXRF the typical concentrations of light elements in any specific ore type. (FPXRF cannot detect elements lighter than atomic number 19). In addition, the calibration capability could also be used to take into account field interferences such as particle size and moisture, typical physical attributes of samples collected from the field in a mining scenario.

CASE STUDIES

In 2000, the development of FP Bulk software for FPXRF with a capability for calibration to specific ore and sample types suggested that the limitations of the technology for use in mine grade control could be overcome. Case studies are presented that detail the following:

1. mine grade control data reported by companies currently using FPXRF for grade control as part of operations; and
2. trial data reported as part of feasibility assessments into the potential application of FPXRF at existing Australian mines.

CASE STUDY 1

Falconbridge NC SAS, a division of Toronto-based Falconbridge Limited, is currently conducting feasibility studies concerning the possible construction of a Nickel Laterite Mine and pyrometallurgical processing plant in the North Province of New Caledonia, capable of producing 60 000 tonnes per year of nickel in ferronickel.

From September 2001 to May 2002, an open pit test mining program was conducted over the Koniambo Ni Laterite deposit with a Liebherr excavator operating on two different bench levels. To direct trial mining operations, an average of four to five channel samples were collected per day. To ensure that machinery was efficiently utilised and not idle, the operation required less than one day turnaround on nickel (Ni) and iron

(Fe) assays. It was not possible to obtain this turnaround by sending samples offsite, with the nearest lab 350 km away in the capital, Noumea. After trialling a FPXRF on-site for several weeks, the operation purchased an instrument for use as the principal grade control tool. Speed of analyses was the primary reason for selecting a FPXRF to carry out this role.

As the mining fronts advanced, routine channel sampling was conducted to determine waste, sub-ore and ore for mining/processing purposes. Channel sample chips (collected by hammer) averaged 3 - 4 kg of material (wet), and were subjected to full sample preparation on-site. This sample preparation involved:

- drying in Clayson oven for 12 hours at 105°C;
- crushing all dry material to 2 - 3 mm (95 per cent passing 5 mm) in standard jaw crusher;
- pulverising split to less than 100 microns in Labtech ball pulveriser; and
- producing 125 gram sample pulps for assay.

FPXRF determinations for per cent Ni and per cent Fe were conducted on the freshly made sample pulps. A minimum of two analyses per pulp were taken for 60 seconds each and the average of the two readings reported. Values were then reported to the mining fronts and spray painted onto the face (<18 hour total turnaround). Every tenth reading undertaken using FPXRF was carried out on a prepared nickel standard to check for accuracy and drift in the instrument.

Sample pulps were then sent to SGS, Perth for complete analyses of Ni and major oxides by borate fusion XRF. The comparative results for Ni and Fe are presented in Figures 1 to 4.

Data summary – Nickel

Good linear correlation was demonstrated between the laboratory and FPXRF data sets, reporting an r^2 value of 0.9 at a slope of 0.88.

78.9 per cent (138) of all samples tested (175) using the FPXRF were within 10 per cent of the laboratory value reported for that sample. On average, the FPXRF reported nickel concentrations 0.09 per cent lower than the participating laboratory with an average relative per cent difference (RPD) of +2.6 per cent.

Data summary – Iron

Excellent linear correlation was demonstrated between the laboratory and FPXRF data sets, reporting an r^2 value of 0.98 at a slope of 0.9.

90.9 per cent (159) of all samples tested (175) using the FPXRF were within ten per cent of the laboratory value reported for that sample. On average, the FPXRF reported iron concentrations 0.23 per cent lower than the participating laboratory with an average RPD of +0.5 per cent.

Case study 1 – Findings

The FPXRF achieved the operators aim of rapid turnaround on grade control samples. Data quality was considered adequate for the mining method employed and was found to expeditiously determine waste from sub-ore and ore. The operators have stated they intend incorporating FPXRF into mining operations should they commence as planned in mid-2004.

CASE STUDY 2

In late-2001, a comprehensive FPXRF trial had been undertaken at the Mt Lyell copper operation in Tasmania – owned and operated by Sterlite Industries India Limited. This trial assessed

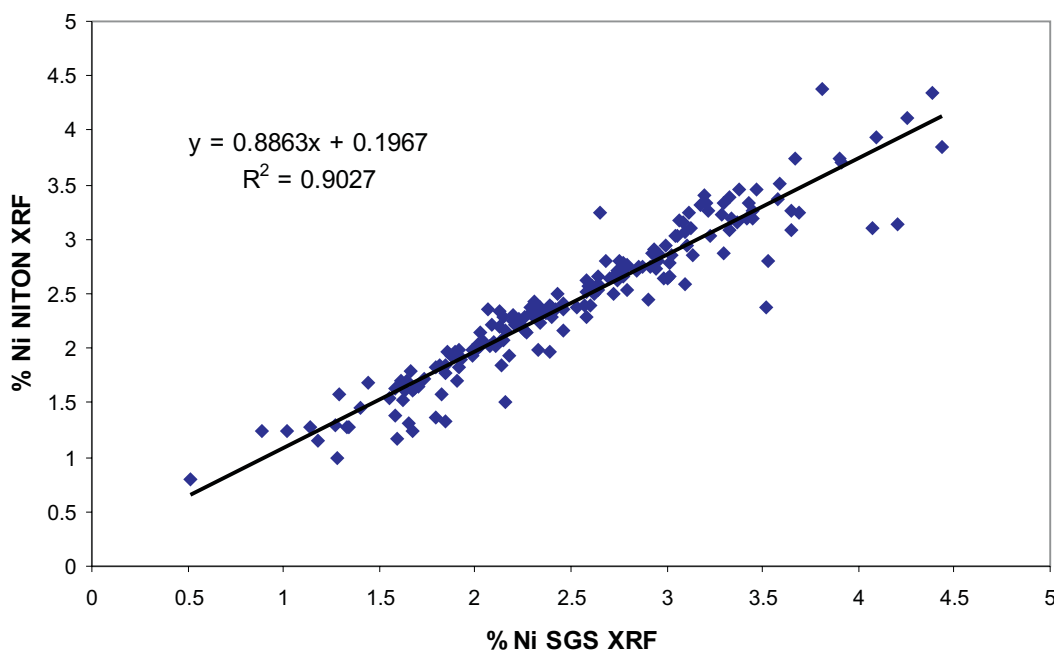


FIG 1 - Nickel Channel sample comparison – Koniambo Ni laterite (n = 175).

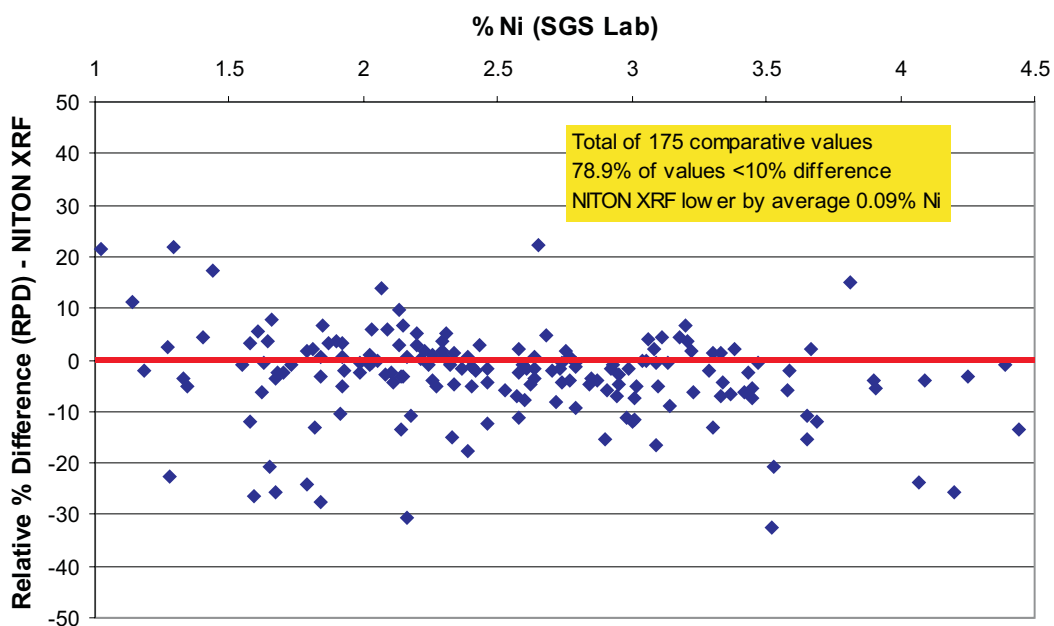


FIG 2 - Nickel Channel sample per cent difference – Koniambo Ni laterite (n = 175).

the impact of sample preparation on FPXRF data quality by testing unprepared bogger samples, samples sieved to 2 mm and fully prepared pulps. The results of this trial are graphically presented in Figure 5 and demonstrated good correlation with laboratory check assays, especially on pulps. FPXRF was not employed at Mt Lyell due to operational changes, however it was considered appropriate for another of Sterlites Australian operations – the Highway Reward Copper Mine.

The Highway Reward Copper Mine is an underground operation located to the south of Charters Towers in North Queensland. The Mount Windsor Joint Venture (MWJV) operates the mine. This is comprised of 70 per cent Thalanga Copper Mines (a subsidiary of Sterlite Industries India Limited) and 30 per cent Grange Resources. The mine has been in operation since 1997, with initially open pit resources, and currently underground resources being extracted. The current

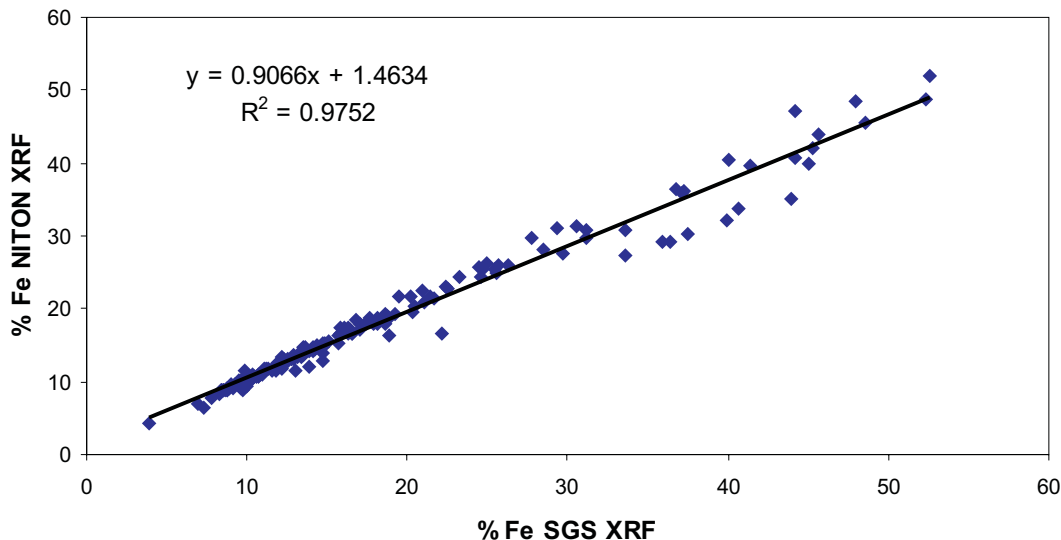


FIG 3 - Iron Channel sample comparison – Koniambo Ni laterite (n = 175).

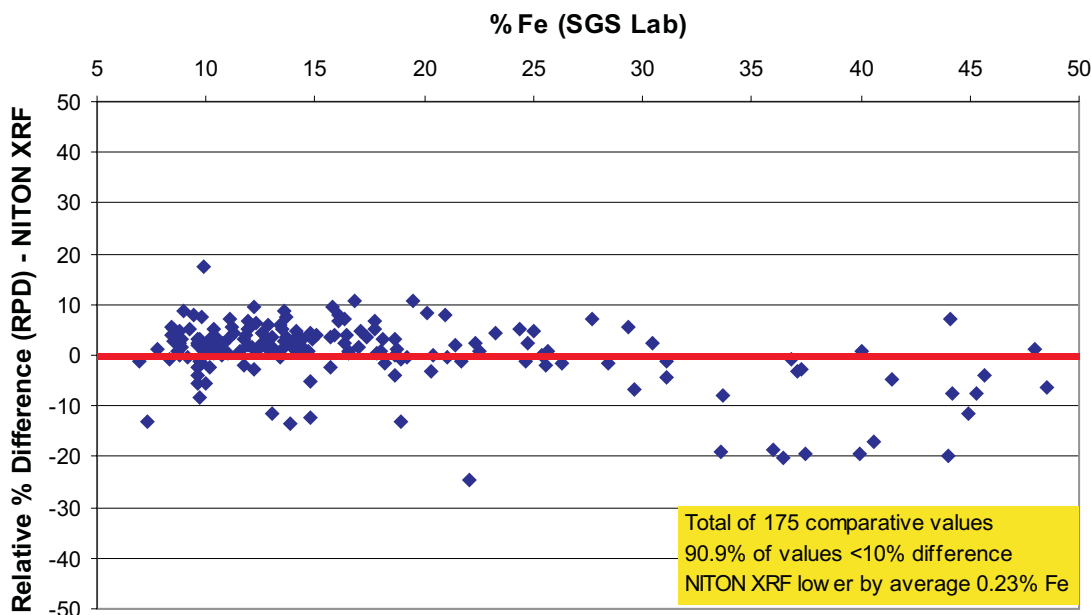


FIG 4 - Iron Channel sample per cent difference – Koniambo Ni laterite (n = 175).

underground resources are being mined using both up-hole benching and sublevel caving techniques. The resource mineralisation style is a volcanic hosted massive sulfide (VHMS) deposit, comprising close to 100 per cent massive pyrite (ie iron sulfide) ‘pipes’. The copper ore is primary chalcopyrite (ie copper sulfide) hosted in high-grade lenses within the massive pyrite ‘pipes’.

Thalanga Copper Mines began to investigate alternative, cost-effective strategies for the rapid assay of underground grade control samples for three principal reasons:

1. the relocation of commercial laboratory services from Charters Towers to Townsville;
2. the quick turn around necessary on daily grade control samples from a sublevel caving operation; and
3. the difficulty in visually differentiating massive pyrite waste from chalcopyrite rich ore.

In early-2002, a trial was undertaken at Thalanga Copper Mines and later that year it became the first mining operation in Australia to purchase and commission a FPXRF for use as the primary analytical grade control tool.

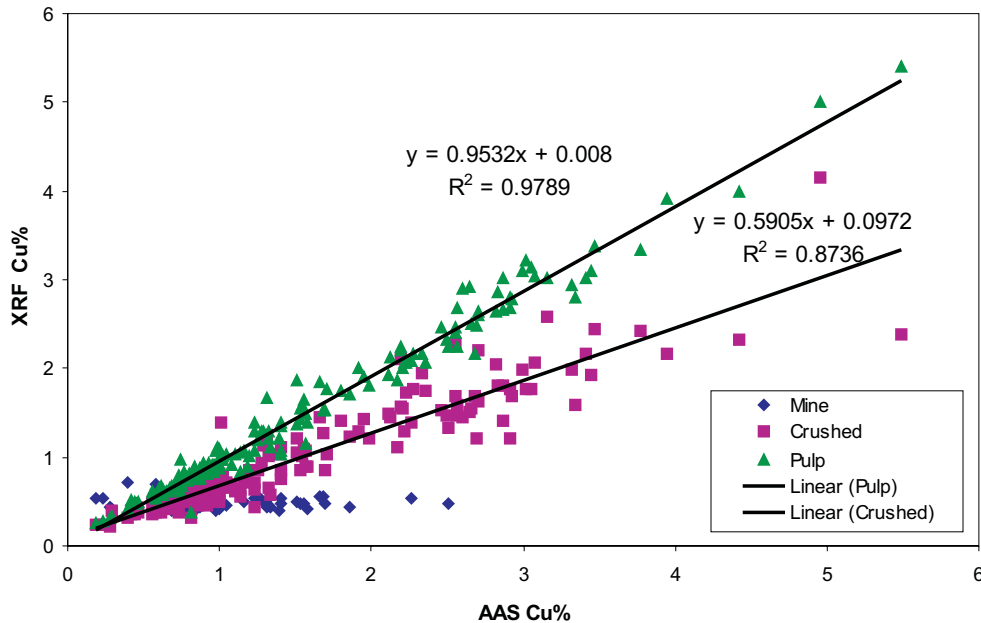


FIG 5 - Copper sample comparison – Mt Lyell (n = 178).

Samples were collected from underground faces at the Highway Mine and transported approximately 100 km to the Thalanga concentrator. Samples were subjected to full preparation through:

- oven drying for a minimum of four hours;
- jaw crushing to less than 5 mm;
- pulverising to less than 100 microns; and
- producing 500 gram sample pulp for analysis.

Analysing samples for copper determination using FPXRF was undertaken using a two-stage method. Stage 1 involved testing samples using calibration factors that were designed to assign each sample to an appropriate copper interval. Stage 2 involved the re-analysis of each sample using calibration factors specifically designed for accurate analysis of copper concentrations within a specific interval. The intervals established were zero to 1.5 per cent, 1.5 to five per cent, five to ten per cent and >10 per cent.

Data reported during FPXRF commissioning on-site proved successful and demonstrated excellent correlation with laboratory data ($r^2 = 0.97$ at a slope of 0.97) and acceptable RPD (83.3 per cent of samples within ten per cent of reported laboratory values). This data is presented in Figures 6 and 7.

A standard operating procedure was developed that included sample preparation and FPXRF analysis. An interval standard was established for each copper interval and tested prior to the analysis of mine samples and at every tenth reading.

Sample pulps were also sent to Australian Laboratory Services (ALS) for check analysis using a Nitric/Hydrofluoric/Perchloric Acid digest and read by Atomic Absorption Spectroscopy (AAS). The first six months worth of comparative data for copper are presented in Figures 8 and 9.

Data summary

Excellent linear correlation was demonstrated between the laboratory and FPXRF data sets, reporting an r^2 value of 0.97 at a slope of 0.93.

However, only 52.9 per cent (973) of all samples tested (1841) using the FPXRF were within ten per cent of the laboratory value reported for that sample. On average, the FPXRF reported copper concentrations 0.09 per cent higher than the participating laboratory with an average RPD of +30.7 per cent.

Thalanga's copper cut-off grade is two per cent. If all data less than 1.5 per cent is excluded from the data set, the average RPD between FPXRF and laboratory data is +5.2 per cent, substantially less than +30.7 per cent. This is due to high RPD at low copper concentrations where the absolute difference between values is relatively small. The significant fall in RPD values by the exclusion of this data demonstrates that the majority of very high RPD values reported, were from sub grade material.

Variation between FPXRF and laboratory check assays was not occurring on an ad hoc basis. That is, the FPXRF would report data of acceptable accuracy for a three to four week period, then report data above acceptable limits for the following three to four weeks.

Case study 2 – Findings

Thalanga Copper Mines geology team believed the principal reason for the mixed results reported by the FPXRF over the first six months of use, arose through the absence of a technically competent person to be responsible for the operation and overall management of the FPXRF on a day-to-day basis.

Staff employed at Thalanga during this period, were too far away to be 'hands-on' in monitoring FPXRF performance (in the case of the Highway Reward mine geologists being 100 km from the plant), or their existing work schedules were too demanding to provide adequate involvement in the project (in the case of the Thalanga concentrator staff). Daily sample testing was carried out by a single field assistant. This person was thoroughly trained in the FPXRF operating procedure, however, was untrained to take responsibility for the entire process, which must include FPXRF versus laboratory data comparison and review. This resulted in analytical discrepancies between FPXRF and laboratory assay going unrecognised for relatively long periods of time. Consequently, solutions could not be investigated and introduced in a timely manner.

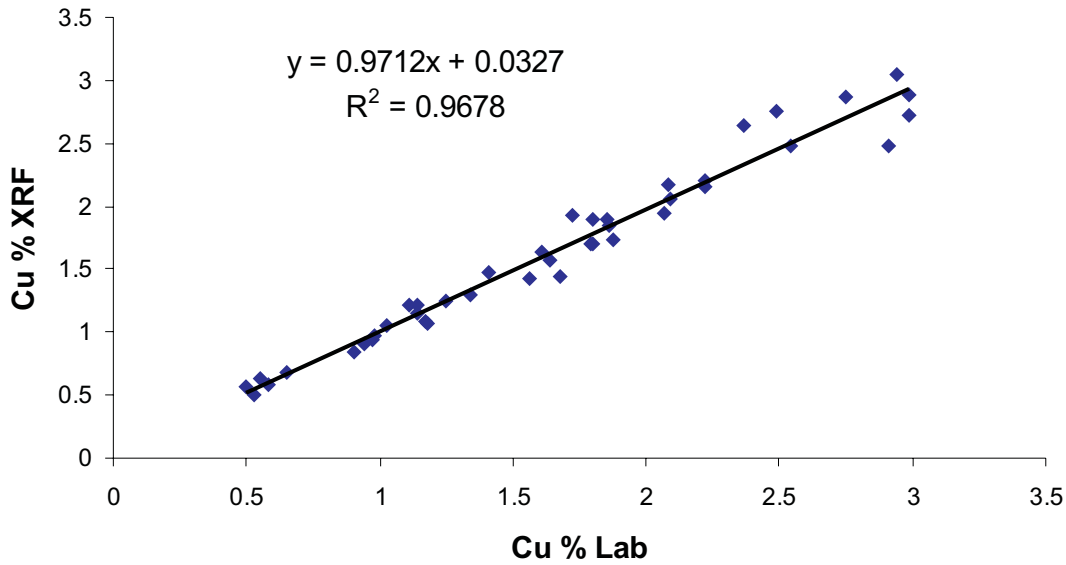


FIG 6 - Copper sample comparison during FPXRF commissioning – Thalanga (n = 42).

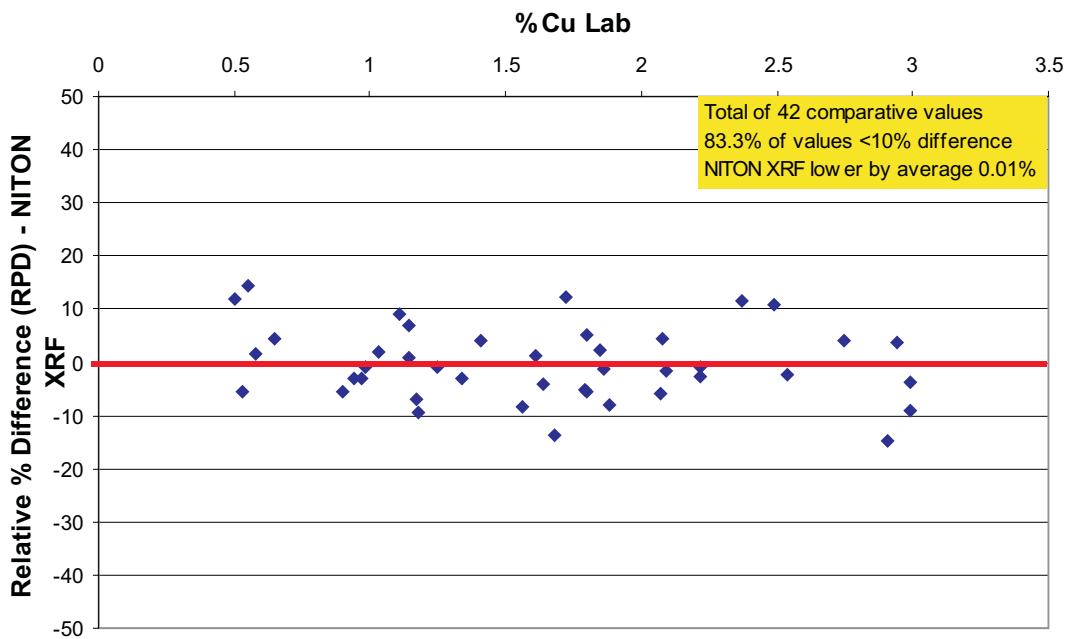


FIG 7 - Copper sample comparison during FPXRF commissioning – Thalanga (n = 42).

Worn sample preparation pulverising bowls (and resultant increase in particle size of samples) were suspected as the cause for a period of time where XRF and laboratory assay comparisons were poor (February, 2003). As soon as the new bowls and puck were installed, assay comparisons returned to within five per cent RPD. It is believed that if a single technically competent person had been given ownership of the entire process (sample prep, FPXRF testing and data review), the cause of the problem would have been identified sooner and solutions could have been introduced without a major impact on mine to mill reconciliation.

As of July 2003, six months after commissioning of FPXRF, the Highway Reward mine geology department remained confident in the ability of their FPXRF unit to provide consistently accurate and reliable grade control data. At the time of writing, the company were in the process of establishing the most efficient way to ensure the FPXRF obtains the appropriate level of technical support required to achieve this.

This case study suggests that the successful implementation of FPXRF within a mining operation should be accompanied by the participation of suitably qualified personnel who can allocate a small amount of time on a regular basis for the purposes of data

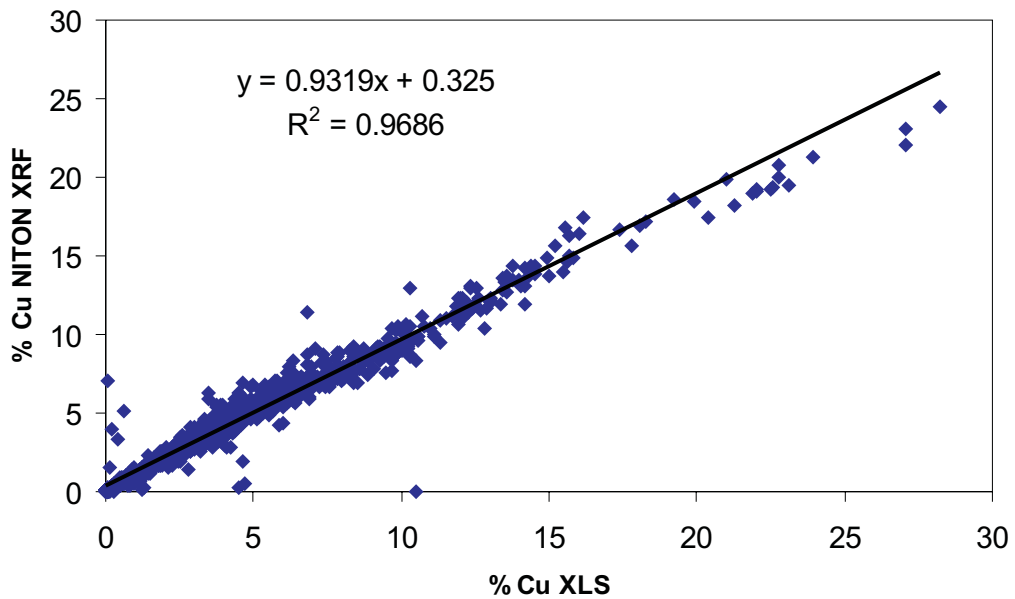


FIG 8 - Copper sample comparison – Thalanga Copper Mine (n = 1841).

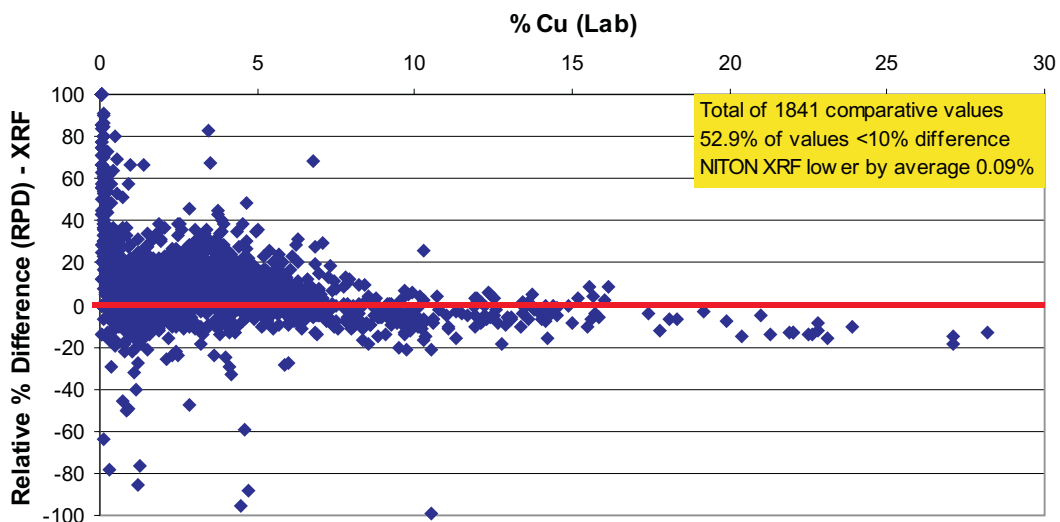


FIG 9 - Copper sample per cent difference – Thalanga Copper Mine (n = 1841).

review. Given the logistics of this particular operation, it appears preferable for a single person to take full responsibility and ownership for all stages of the grade control analysis procedure, from sample preparation through to data analysis and comparison.

CASE STUDY 3

The Ernest Henry Iron-oxide Copper-Gold mine is located 38 kilometres north-east of Cloncurry in north-west Queensland. The mine is owned by Ernest Henry Mining Pty Ltd (EHM), a subsidiary of MIM Holdings Limited. The Ernest Henry deposit, being close to the surface, is amenable to low cost, large scale, open pit truck/shovel operation. Over the 15 years of the

currently planned mine life, in excess of 700 million tonnes of ore and waste will be mined. The mine plant provides for up to ten million tonnes of ore to be extracted each year from the open cut operation to produce approximately 360 000 tonnes of concentrate containing 100 000 tonnes of copper and 125 000 ounces of gold.

Since commissioning in 1997, EHM has carried out grade control via a staggered 15 × 15 m grid reverse circulation (RC) drill pattern. Holes are typically 48 m deep with 2 m sample intervals. An on-site laboratory assays all grade control and blasthole samples. However, in early-2003, an assessment of grade control drilling versus blasthole sampling was commissioned. As part of this assessment a trial of the effectiveness of an FPXRF was undertaken.

The trial involved the analysis of unprepared blasthole cuttings (hole diameter 270 mm) using the FPXRF directly in calico sample bags collected from the open pit. The aim of the trial was to assess the feasibility for the FPXRF to provide preliminary grade control data within 12 - 24 hours of collection to allow the mine geology department to quickly define the ore/waste boundaries in the pit. Sample turnaround from the on-site laboratory varies depending on sample throughput, but is in the order of two to seven days for base metal results. The opportunity to obtain reliable data within 12 - 24 hours would allow the mine geology department to insert FPXRF data into the grade control model as 'preliminary' assay results and direct in pit operations with confidence.

To overcome problems of obtaining a representative sample from a blasthole, a composite sample is compiled from a total of

four full profile subsamples (one from each quadrant of the blasthole). For the purpose of this trial, blasthole samples were collected as per normal operations and then riffle split several times. One sample was dispatched to the on-site laboratory for analysis in duplicate, another sample was dispatched to Australian Laboratory Services (ALS) and the remaining sample retained for FPXRF analysis. No preparation was carried out on any samples prior to FPXRF analysis. To overcome sample heterogeneity issues, each sample was tested directly in its calico bag four times for 30 seconds and the average of the four readings reported. Site-specific standards were run prior to and at the end of each days testing.

Comparative copper data as reported by the FPXRF and the average of the laboratory readings is presented in Figures 10 and 11.

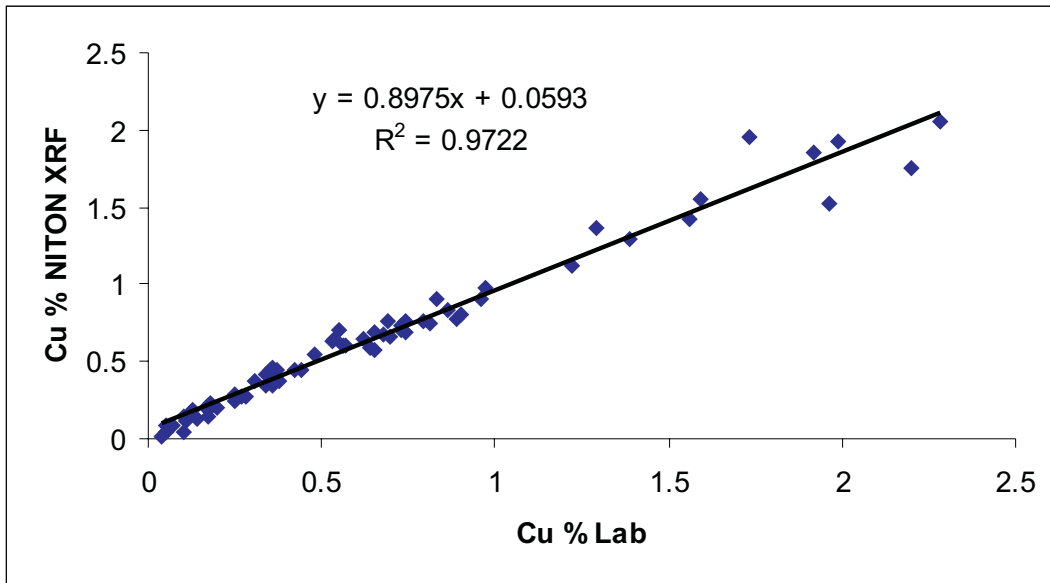


FIG 10 - Copper blast hole sample comparison – Ernest Henry Mine (n = 67).

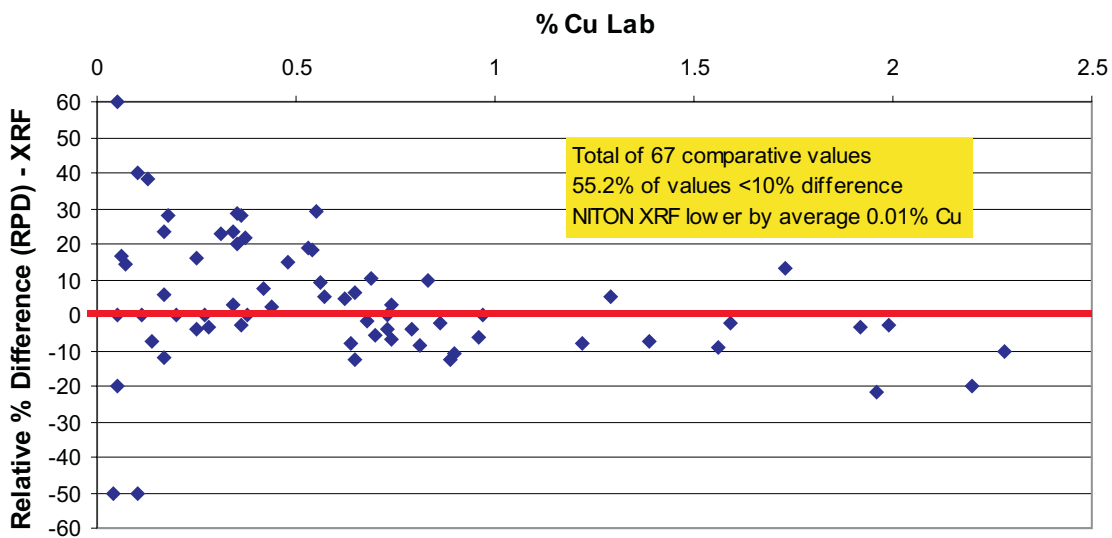


FIG 11 - Copper blast hole sample per cent difference – Ernest Henry Mine (n = 67).

Data summary

Excellent linear correlation was demonstrated between the laboratory and FPXRF data sets, reporting an r^2 value of 0.97 at a slope of 0.92.

55.2 per cent (37) of all samples tested (67) using the FPXRF were within 10 per cent of the laboratory value reported for that sample. On average, the XRF reported copper concentrations 0.01 per cent lower than the participating laboratory with an average RPD of +3.42 per cent.

79.1 per cent (53) of all samples tested (67) using the FPXRF were within 20 per cent of the laboratory value reported for that sample. This represents a good result given no sample preparation was carried out prior to analysis.

Case study 3 – Findings

The cut-off grade used at EHM is 0.3 per cent. Despite greater RPD at relatively low concentrations (<0.55 per cent Cu), the FPXRF was able to differentiate waste from ore and provide relatively consistent and accurate data (± 10 per cent) above 0.55 per cent copper.

No samples classified as waste through laboratory analysis were reported as ore by the FPXRF. Likewise, no samples reported as ore by the laboratory were reported as waste by the FPXRF. This suggests that the FPXRF may be suitable for use as a preliminary tool for the differentiation of ore boundaries in pit. Moisture contents of blastholes will increase as the depth of the pit increases and will rely heavily upon dewatering the previous staged pit. This may impact on data quality for FPXRF with excessive moisture contents. However, unpublished trial data undertaken on behalf of Queensland Nickel suggests that, as with particle size, the interferences on FPXRF data quality as a result of moisture can be overcome if the water content is relatively consistent.

CONCLUSIONS

The case study data presented herein indicates that there is definite potential for FPXRF to play a role as a rapid analytical tool to assist mine grade control sample turnaround where this can improve operational efficiency. The data presented indicates that in the operational settings discussed:

- FPXRF recorded nickel in pulps to within ten per cent of the reported concentration in 79 per cent of the samples tested;
- FPXRF recorded copper in pulps to within ten per cent of the reported concentration in 53 per cent of the samples tested (despite acknowledged deficiencies in program supervision); and

- FPXRF recorded copper in unprepared blast hole cuttings to within 20 per cent of the reported concentration in 79 per cent of the samples tested. The FPXRF was also able to delineate waste from ore in 100 per cent of the blast hole samples tested.

These case studies suggest that FPXRF can provide operational and cost benefits to some mining operations. The type of operation to benefit from FPXRF will be dependant on factors unique to that operation. These may include:

- the mining method employed and necessity for rapid access to geochemistry data;
- numbers of grade control samples collected per shift;
- existing laboratory turnaround time;
- the availability of suitable staff to oversee any FPXRF program;
- data quality required to make informed decisions; and
- the amount of sample preparation needed to achieve data of a specified standard.

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