DEVELOPMENT AND TESTING OF DOWN-THE-HOLE DEPLOYABLE X-RAY FLUORESCENCE SPECTROMETER

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ABSTRACT

To allow a truly in-situ elemental analysis, an Australian resource company commissioned the development and building of a down-the-hole deployable ED-XRF sensor. The instrument was designed to be deployable up to a depth of 30 m for drill holes of varying diameters as well as being run in a bench-top mode.

Following the development and upon commissioning, the ED-XRF system was investigated in the laboratory for its suitability for use in the Iron Ore industry by using well characterised crushed iron ore samples, standards made from analytical reagents and large iron ore rocks that had been sectioned to provide a curved surface for presentation to the XRF. The effects of operating conditions and other parameters were studied using the standards made from the analytical reagents. It was found that the system could be used to determine the elemental Fe content with reasonable accuracy when used on crushed powder samples. The curved surfaces of the sectioned rocks increased the variability of the amount of Fe detected.

Two trace elements, Al and Si were also investigated using the same methodology. It was found that the ED-XRF system was not suitable for distinguishing the Al and Si .

This paper outlines the ED-XRF system used, the testing methodology employed and the results of the laboratory testing.

Keywords: Down-The-Hole, X-Ray Fluorescence Spectroscopy, Field deployable

INTRODUCTION

X-Ray Fluorescence (XRF) Spectroscopy has been used widely in geology and mining for elemental analysis of samples across diverse mineralogical spectrum for a large number of years. The advent of new Energy Dispersive X-Ray Fluorescence (ED-XRF) Spectrometers has allowed for transportable, smaller bench-top spectrometers to be developed. Coupled along with the development of hand-held mineral analysers, this has meant that analysis in the field were able to be completed without the need to transport samples to testing laboratories. However both methods have not allowed a truly in-situ elemental analysis.

The case for in-situ elemental analysis is well made by Argyraki [1] and the use of field deployable bench top instruments in more general terms by Gore [2]. Frost [3] outlines
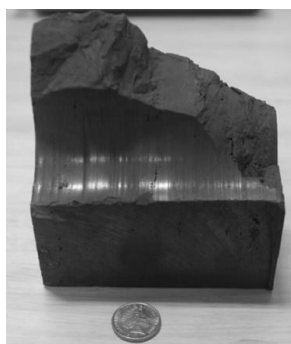
the niche that hand-held mineral analysers have in the nickel industry within exploration and mining groups.

The culmination of the two interests has resulted in the development of an XRF system that can be deployed in a down-the-hole (DTH) mode as well as run in a bench-top configuration. This system gives the flexibility of utilising both DTH information generation as well as being able to be used in conjunction with a small sample crushing system to allow for analysis of traditional XRF pressed pellet or cup samples.

Developed by a specialist manufacturer DTH-XRF beta unit was assessed for its operational ability with specific reference to use in iron ore exploration, however in addition a more general assessment of the technology was undertaken. This was achieved by using selected samples from a production mine as well as making use of synthetic ores made from reagent grade chemicals. Final work up of the laboratory assessment made use of rock samples sectioned so as to provide a curved surface for presentation of the rock surface to the analysis window of the instrument (see Figure 1-(A)). The specific operational parameters assessed were effects of sampling time, instrument warm-up time, and reproducibility and accuracy of analysis along with operational ease for field deployment.

The system utilises a 40 kV rhodium target X-ray generator and Si-pin detector, housed in a 48 mm diameter 475mm long housing (see Figure 1-(B)). The housing is connected via an umbilical to the control panel, the umbilical contains the communication cables as well as a helium line. The helium line is used to purge instrument window area for analysis. Moxtex polypropylene X-ray window is used (see Figure 2 (A)). The window is recessed partially to provide protection, additionally a rubber sealing pad is incorporated when used down-the-hole for better contact with the surface of the drill hole. The system incorporates a Geostuff BHG-2 clamping actuator in the casing allowing use in drill holes up to 480 mm diameter.

The signal system consists of an Amptec ADMCA and associated software. The spectra is analysed using Spectrax de-convolution software. The system allows for variations in elemental analysis as well as for use of multiple correction factors to translate intensity to concentration. The data after de-convolution is saved as a simple text file that can be data-transferred or read on the control laptop.

A   B

Figure 1 (A) Cut sample showing curved surface used for analysis (coin is Aus $2 for size). (B) Probe system showing probe and control panel

A   B

Figure 2 (A) X-ray window showing polypropylene and helium purge. (B) Bench top configuration.

EXPEREMENTAL

The DTH-XRF instrument was operated in its bench-top mode analysing Fe_2O_3 powder samples initially (see Figure 2 (B)). The samples were reagent grade powders that were certified 99% pure, the Fe_2O_3 was mixed with SiO_2 at varying concentrations to make a sample set that covered the range from 0 to 50 wt% SiO_2. The samples were weighed out using a five decimal balance, following by hand-ground in an agate mortar with an agate pestle. Once ground, the samples were placed into sample cups that have 4 µm polypropylene film supplied by the instrument manufacturer.

The DTH-XRF was operated in line with the manufacturer's instructions of having one cubic foot of He flow as displayed by the flow meter on the instrument panel. An air purge of five minutes was initiated for the warm-up cycle with 2 second purge after each sample change.

The investigation into warm-up requirements made use of a 50/50 wt% reagent grade SiO_2/Fe_2O_3 sample and included sample analysis at commencement of operations, after 4 hours of operation, and after 6 hours of operation. The collection time used was 30 seconds per spectrum with a total collection time of 600 seconds giving 20 spectrum per sample collection period. Figure 3 shows that no real dependence on warm-up is notable and or any drift. The error bars displayed are one standard deviation of the 20 collected spectra whilst the value is the mean of the 20 spectra. The actual value for the Fe content is displayed for comparison.
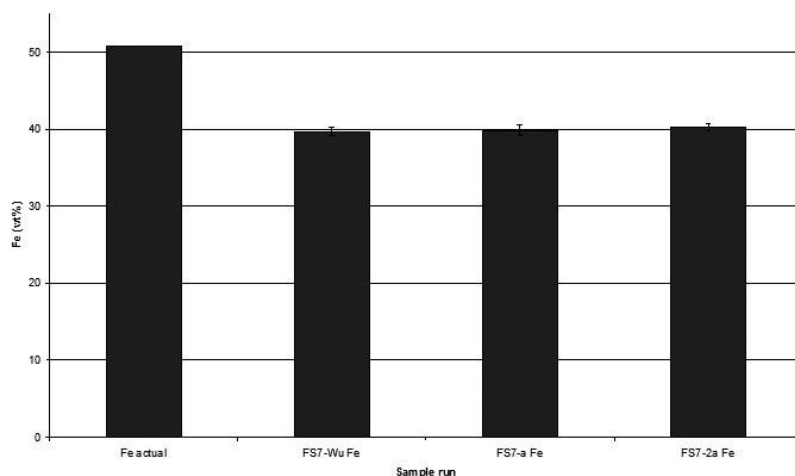
Figure 3 Warm up dependence comparison (error bars are 1 stdev)
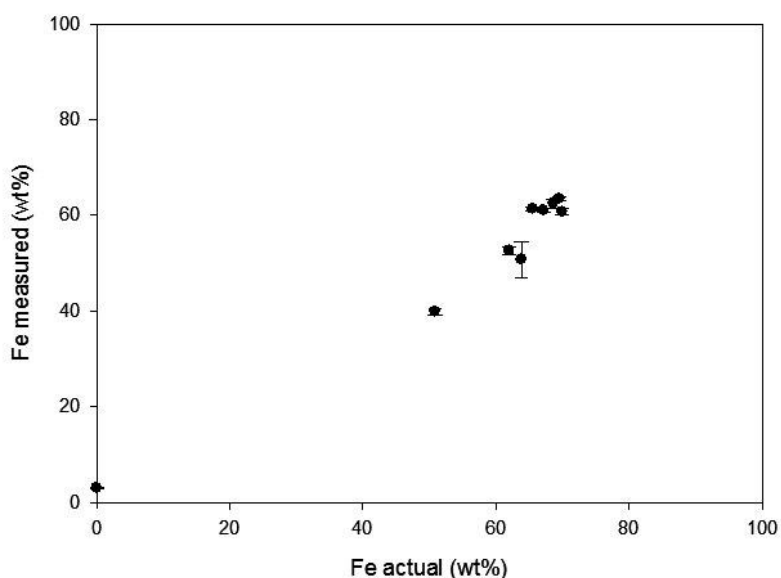

Figure 4 Fe actual vs. measured for Si cut samples including zero Fe (error bars which may be too small to see are 1 stdev).

The Fe content of the samples was investigated (see Figure 4). It was impossible from this to discern any trend within the data. The error bars indicated that a large variability within the spectrum from the same samples was present in some instances. Standard deviations of greater than 3% are common from this data set with the largest being over 5%, the variability within the 20 spectra appears to be of the same order as that seen when taking three different sets of 20 spectra from the same sample at different operating times.

The Si values were plotted (measured against actual values), the variability in results within samples was in excess of ± 5% with the samples of less than 2% Si having lower

variability but the standard deviations returned were larger than the actual values of elemental Si within the sample.
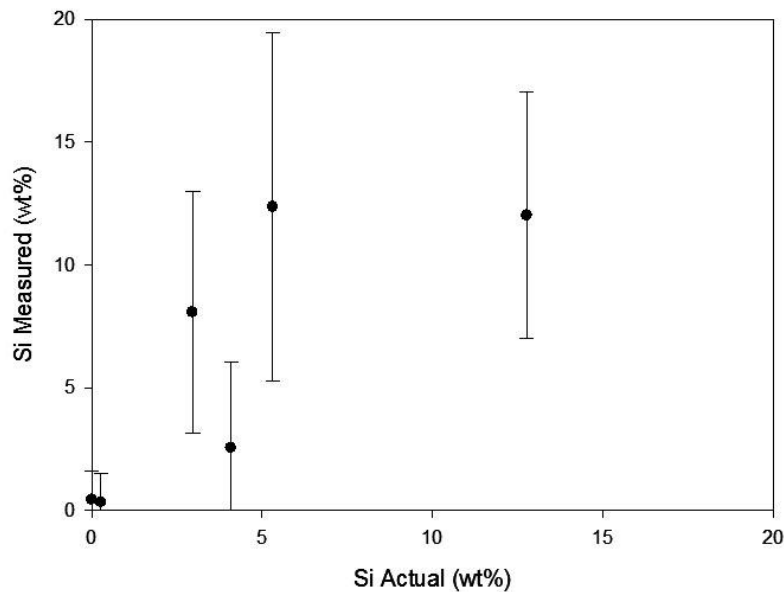

Figure 5 Si measured vs. Si actual in Fe samples (error bars are 1 stdev)

To investigate if this variability within the data from a single sample was due to matrix effects and/or if the Si was dominating the results, a sample set was produced that had Fe_2O_3 mixed with Al_2O_3. Again the samples were manufactured from AR grade reagents using a 5 decimal place balance and then hand-ground using an agate motor and pestle. The samples were analysed using the same configuration as that used previously for the Fe-Si work. Lower variability from within a single sample was noted for the Al Fe matrix (Figure 6). The standard deviations were still above 2% Fe, however the large extreme variations were not evident.
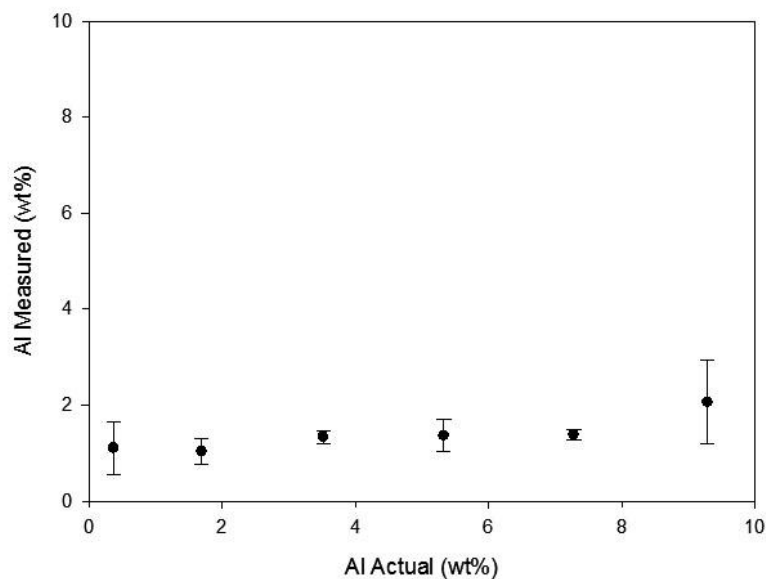

Figure 6 Al measured vs. Al actual (in Fe/Al samples) (error bars are 1 stdev)

Natural Iron Ore samples were analysed using the DTH-XRF and a commercial laboratory-based standard XRF fusion bead system.

Table 1 actual vs. measured for natural Iron Ore sample

% actual/measured		
Fe (actual 57.3 %)	SiO_2 (actual 5.6 %)	Fe2O3 (actual 2.6 %)
90	-10769	445

The data shows that the DTH-XRF instrument can be used with some confidence for the major element, however the trace elements as shown in the synthetic samples are poorly analysed.

The DTH-XRF instrument was then assessed against using a sectioned sample presenting a curved surface to the instrument. The curved section that was removed as part of rock sectioning was crushed and analysed using a commercial laboratory-based standard XRF fusion bead methods as well as by the DTH-XRF. Table 2 shows the results from this work, the DTH-XRF returned a result in line with the actual/measured (90%) previously seen, what is telling is that the variability of the curved surface as determined by the standard deviation is significantly higher. This is somewhat in line with expectations as the comparison is being made to a well crushed and homogenized sample which elevates any variation that may be present in the rock strata and other performance parameters that are obvious to XRF practitioners like particle size and such.

Table 2 Fe Curved surface, crushed and commercial laboratory

Fe % Uncertainty are 1 stdev		
Curved	Crushed Bench top mode	Commercial Laboratory
63 ± 7.8	63 ± 26	58 ± 1.0

CONCLUSIONS

The DTH-XRF instrument analysed at Curtin University of Technology was found to perform well for elemental Fe analysis, however its performance when analysing SiO_2 and Al_2O_3 as trace elements in Fe 2O3 was poor. However the authors feel that the results of the reported work show that the DTH-XRF holds great promise for further development for use in the exploration arena were flexible deployment (down-the-hole

or bench-top) as well as positive results for major element make it a potentially attractive tool.

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