

DRILLING POWDERS PRODUCED DURING DIAMOND DRILLING OFFER AN OPPORTUNITY FOR ANALYSES IN NEAR REAL TIME

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Introduction

The common practice in current exploration diamond drilling campaigns is to drill a hole, collect the core, log the core, split the core and send samples from selected intervals for analysis in a commercial lab. This results in a turnaround time of a few weeks or months, and the data obtained is a conventional assay data on a number of chemical elements. Rarely, if ever, a few samples are analysed by some other techniques, for example X-Ray Diffraction (XRD). Powders produced during diamond drilling show high potential to act as a representative sample media of the lithology intersected by the drill hole. The drilling powders are transported back to the surface by the drilling fluid providing an opportunity of being sampled and analysed in real time at a resolution defined by a geologist on site. Moreover, this novel sample media requires little or no sample preparation before it can be analysed.

Here, we demonstrate application of using diamond drilling powders collected while drilling to decipher geochemistry and mineralogy in near real time. We also show that sampling depth can be determined accurately and precisely within a narrow range and the depth fidelity excellent as shown by the comparison to the corresponding core.

This is a first study of a novel sampling media and although it is applied to conventional diamond drilling, in future the approach will be incorporated for other drilling types such as coil tube (CT) drilling, which will only return a powdered sample to the surface (Hillis et al. 2014).

Methodology

Sample collection and preparation

The samples studied here are from a diamond drill hole at the Brukunga pyrite mine, South Australia, closed in 1970s and currently being used as a Deep Exploration Technologies Cooperative Research Centre Drilling Testing and Training Facility. It is located 47 km east from Adelaide, South Australia. The rocks intersected by the drill hole are metasediments of the Nairne Pyritic Formation comprised of fine-grained greywackes, quartzites and siltstones and pyrite- and pyrrhotite-bearing beds siltstones of Cambrian age (Skinner 1958; Graham 1978). The drill hole intersected the main ore body from 130 to 280 m, and a number of thin zones with iron sulfide veins. The rocks of the Nairne Pyritic Formation are intruded by altered dolerite dikes.

During drilling of the DETBrukunga2 hole, drill fines brought up to the surface by drilling fluids were passed through the Imdex Solid Recovery Unit (SRU) and collected post centrifuge every 1.5 m of drilling. The sampled material was dried in a laboratory oven at 50°C to avoid any mineralogical damage.

Particle size distribution analysis

The nature of the sample passed through the SRU was carefully examined. The particle size distribution study was done as dry sieving and careful weighing of all fractions. Figure 1 shows an SEM image of the material and corresponding particle size distribution plots. As seen from Figure 1, the material sampled at the back of the SRU from DETBrukunga 2 is fine-grained with most particles being less than 38 µm. Note that this type of material does not require any further sample preparation prior to being introduced to XRF and XRD sensors and is of similar quality as pulps prepared by commercial laboratories.

Portable X-ray Fluorescence Spectrometry

Portable XRF analyses were performed on drilling powders using an Olympus X-5000 (25 mm² detector, 50kV, 10Watt Ta anode) and an Olympus Delta Premium (30 mm² detector, 40kV, 4Watt Rh anode) instruments. Samples were analyzed in Soil, Mining Plus and Geochem modes. Prior to use, the instruments were standardized using a 316-steel plate. An instrument check was done prior to measurement and then after every 15 samples using standards NIST2702, NIST2709, NIST 2781, NIST 2710a, NIST 2711a and a SiO₂ blank. Detection limits were estimated on the basis of reproducibility of the measurements for certified reference materials (NIST standards). The samples were placed in plastic vials, with the opening covered with Kapton tape. A small amount of material is required for this analysis, 10 g samples were used in this study. The vial was then placed on the instrument window (tape side down) and analyzed with reading time on each beam of 60 seconds. Both Mining Plus and Geochem modes have two beams, and the Soil

mode has three beams, with the beam energy stepping down with each subsequent beam. The counting time was set up at 60 seconds, and this resulted in total run times of 120 seconds for the Mining Plus and Geochem modes, and 180 seconds for the Soil mode. The data were processed off-line by quality assurance and quality control (QA/QC) procedures based on the standard performance and application of appropriate calculated correction factors. The correction factors were developed based on the analysis of certified reference materials NIST2702, NIST2709, NIST 2781, NIST 2710a, NIST 2711a, and comparison of the expected and measured concentrations for each standard.

To compare the results of the pXRF on drill powders produced during diamond drilling and separated from drilling fluids by the SRU to the typical results that would be achieved through lab analysis of core, core material was sent to a commercial laboratory for analysis (ACME Labs, Vancouver, Canada). Core intervals 1.5-m-long and corresponding to the same 1.5-m-long intervals of composite powders were analysed using a set of analyses including XRF, lithium metaborate/lithium tetraborate fusion ICP-MS, 4-acid digest ICP-MS and fire assay fusion by ICP-ES for major and trace elements.

Powder X-ray diffraction

The same samples of drilling powders were subsequently analysed by X-ray diffraction (XRD) using two instruments: (1) a lab-based Bruker D4 Endeavor AXS instrument, operating with Co radiation; data collection range of 2θ angle from 5 to 90° , with step size of 0.2° ; data collection time of 7 minutes per sample; and (2) an Olympus Terra portable XRD instrument, operating with Co radiation; data collection range of 2θ angle from 5 to 55° , with increment of 0.25° ; data collection time of 10 minutes per sample. Sample preparation for the analysis on the Bruker instrument required the material to be mixed with ethanol to form a paste and applied to a glass slide in order to obtain random orientation of mineral crystallites, particularly of platy shape. No additional sample preparation was required for the Terra pXRD instrument as the particles were less than $130\text{ }\mu\text{m}$, and the pulps were loaded into the piezo-harmonic, Vibrating Sample Holder (VSH) of the Terra instrument (Sarrazin et al. 2005). The vibration frequencies are within the kHz range (Sarrazin et al. 2005). For both Bruker and Terra XRD analyzers 100 to 200 mg of material were used. Quartz contained in all analysed samples was used as an internal standard to verify mineral peak positions and correct for any displacement if required.

The XRD patterns obtained from both instruments were processed and mineral assemblages were quantitatively estimated using a Bruker DIFFRAC.EVA software package which is based on the Reference Intensity Ratio (RIR) method (Smith et al. 1987). Each peak above the background was examined and mineral identification in the analyzed samples was done manually to ensure that all

components present in the multiphase mixtures are identified and accounted for, as the quantification data are normalized to 100%.

Quality assurance of data collected and processed was performed using a workflow and standards outlined in Uvarova et al. (2014). Artificial mixtures were prepared by weight using well-characterised mineral standards (i.e., quartz, albite, muscovite, biotite, hornblende and pyrite) and these mixtures were analyzed using the same procedures as the samples. The obtained diffraction patterns were processed and mineral abundances were estimated and compared to the expected percentages demonstrating close agreement (Uvarova et al. 2014).

Results

Particle size distribution study shows that powders produced during diamond drilling and collected post-SRU are uniform and fine-grained providing an opportunity to be introduced to various sensors with no additional sample preparation.

Selected elemental contents determined by pXRF are shown in Figure 2 and are correlated with the lithologic log for the drill hole. The drill hole from which the samples were collected for this study intersected a zone of stratiform sulfide mineralization from 130 to 260 m, a few narrower intervals (5 cm to 1 m) with sulfide veins (e.g., at 65, 100, 103, 275 - 285, and at 300 m) and a few dolerite dikes (48 - 50, 288 - 291, 292 - 293, 295 - 298, 302 - 304, and 315 - 318 m) (Figure 2). The pXRF data show that sulfide veins and main mineralization are evident as sharp increases in Fe and S contents, whereas dolerite dikes are distinguished by well-defined peaks in Ca (Figure 2).

Comparison of the lab results with pXRF performed with Olympus Delta instrument are shown in Figure 3. Comparison of the lab results done on the core and pXRF results analyses done on the drilling powders shows that the results compare very well in their spatial correlation, and concentration variations match peak for peak (Figure 3).

X-Ray diffraction analysis provides information on mineral assemblage and percentage of each phase present in the sample. The assemblage quartz – feldspar - biotite – muscovite \pm actinolite \pm chlorite \pm pyrite \pm pyrrhotite was identified in all samples studied. The main mineralisation zone and thinner sulphide veins were obvious from the identified pyrite and pyrrhotite, with their total abundances closely matched to those estimated by a geologist during core logging (Figure 2). Altered dolerite dikes are prominent from the presence of actinolite (Figure 2). As quantitative XRD data provides information on the amount of every crystalline mineral phase present in the sample above 3 vol.%, it can be further used to calculate the density of the rock based on the analysis of the corresponding diamond drilling powder (as shown in Figure 2).

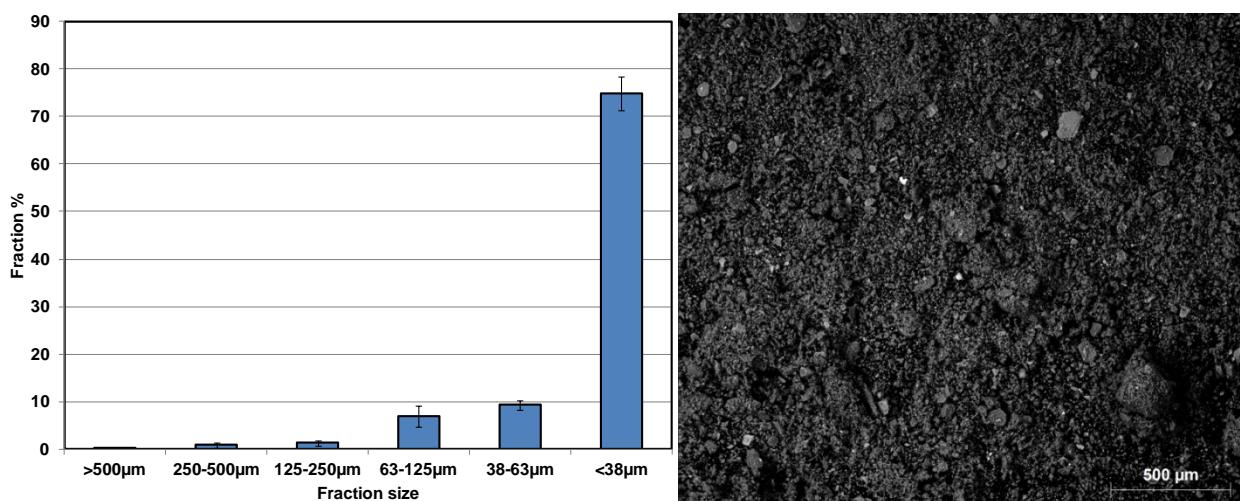


Figure 1. (a) Back-scattered electron image of the material from the DETBrukunga2 drill hole, and (b) particle size distribution for drilling powders collected at the back of SRU.

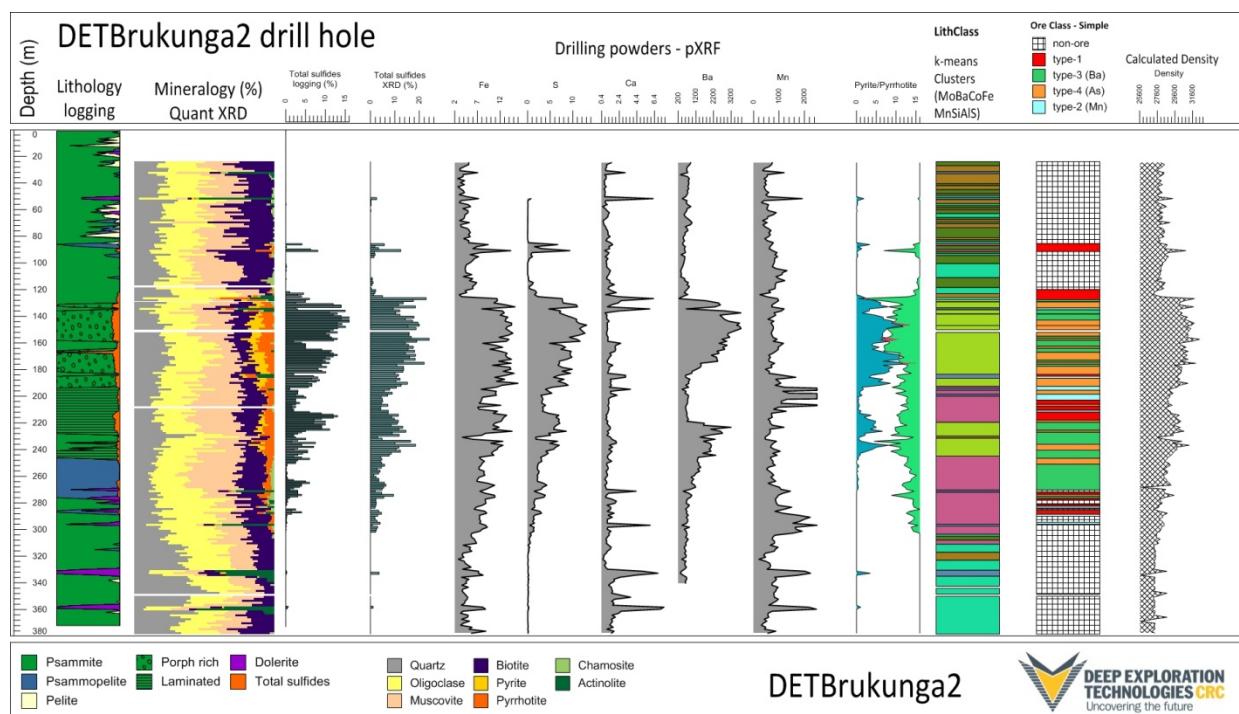


Figure 2. DETBrukunga2 drill hole data: geologic log, results of quantitative XRD and pXRF analyses, lithochemical classifications and density calculated based on QXRD interpretations.

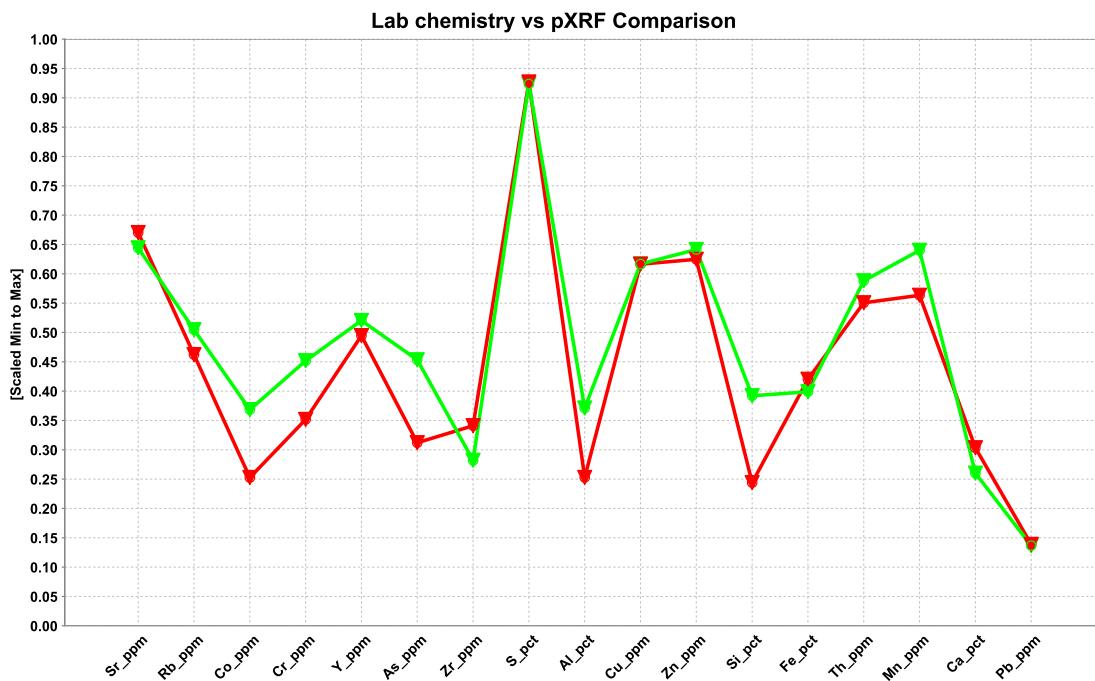


Figure 3. Comparison of selected elemental concentrations determined by pXRF in drilling powders (in green) and commercial lab assays done on the corresponding diamond core (in red) from the DETBrukunga2 drill hole.

Discussion and Conclusions

The current study illustrates that diamond drilling cuttings previously considered to be ‘waste’, show high potential to act as a representative sample media of the lithology intersected by the drill hole and can be successfully used for analyses in real time. The approach undertaken here is to collect diamond drilling cuttings brought up to the surface with drilling fluids from well constrained depth intervals; separate the diamond drilling powders from the fluids using an SRU; dry the powders and analyse them. The sample has proven to be homogeneous and any observed heterogeneity was within analytical precision. The drilling powders are transported to the surface fast enough to avoid any lag that can potentially create smearing in the data. Moreover, the sampling depth can be determined accurately and precisely within a narrow range. In the first instance we suggested analyses by a combination of XRF and XRD, as these portable sensors are well developed, have an excellent performance and produce data of high quality. Comparison of XRF and XRD results for powders with existing logging of the corresponding core showed that drilling powders are consistent with the lithologies intersected by the drill hole. Comparison of pXRF analyses results done on powders are comparable with core assays done by a commercial lab on the corresponding core.

The approach suggested here of combined pXRF-pXRD analyses can be performed on a large set of complex geological samples and the techniques complement each other. Portable XRF results can be used to verify the results of portable XRD and vice versa. Our study also shows the viability and usefulness of portable XRD analysis that is currently underused. Portable XRD analysers can provide data comparable to modern lab-based instruments and the novel, innovative, and unique convectional sample chamber of the Olympus Terra analyser is ideally suited for analysis of samples with minerals with preferred crystallographic orientation such as mica-group minerals, chlorites, and amphiboles (Uvarova et al. 2014). A small amount (less than 5 - 10 g) of sample is required for coupled pXRF-pXRD analysis that can be performed with currently available portable instruments in less than 15 minutes for both measurements. Drying of this amount of material requires little time (up to 30 minutes).

Application of the workflow that we used at Brukunga for drilling powders collected from a 380-m deep diamond drill hole results in full chemical and mineralogical analyses by the time the drill hole is completed, providing ‘objective logging’ and an opportunity to make a decision in due course of an exploration campaign in real time.

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References

- GRAHAM, J. 1978. Manganochromite, palladium antimonide, and some unusual mineral associations at the Nairne pyrite deposit, South Australia. *American Mineralogist*, **63**, 1166-1174.
- HILLIS, R.R., GILES, D., VAN DER WIELEN, S.E., BAENSCH, A., CLEVERLEY, J.S., FABRIS, A., HALLEY, S.W., HARRIS, B.D., HILL, S.M., KANCK, P.A., KEPIC, A., SOE, S.P., STEWART, G. & UVAROVA, Y. 2014. Coiled tubing drilling and real-time sensing – enabling prospecting drilling in the 21st century? *Society of Economic Geologists, Special Publication* **18**, 243-259.
- SARRAZIN, P., CHIPERA, S., BISH, D., BLAKE, D. & VANIMAN, D. 2005. Vibrating sample holder for XRD analysis with minimal sample preparation. *Advances in X-ray Analysis*, **48**, 156-164.

SKINNER, B.J. 1958. The geology and metamorphism of the Nairne Pyritic Formation, a sedimentary sulphide deposit in South Australia. *Economic Geology*, **53**, 546-562.

SMITH, D.K., JOHNSON, G.G.J., SCHEIBLE, A., WIMS, A.M., JOHNSON, J.L. & ULLMANN, G. 1987. Quantitative X-Ray powder diffraction method using the full diffraction pattern. *Powder Diffraction*, **2**, 73-77.

UVAROVA, Y., CLEVERLY, J., BAENSCH, A. & VERRALL, M. 2014. Coupled XRF and XRD analyses for rapid and low-cost characterization of geological materials in the mineral exploration and mining industry. *EXPLORE Newsletter for the Association of Applied Geochemists*, **162**, 1-14.