REPORT NO. 543

Field-based XRF for prompt Au analysis

Results of research carried out as MRIWA Project M0543

Portable PPB Pty Ltd

by

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April 2022

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EXECUTIVE SUMMARY

Portable PPB Pty Ltd (PPPB) has developed detectORE™ Technology that will analyse gold in the field within a day of sample collection, with extensive testing in the laboratory and field demonstrating the method to be safe, robust, practical, and fit for purpose. A proof-of-concept detectORE™ was developed and patented by CSIRO, with PPPB obtaining the exclusive worldwide license to the early technology and considerably advancing it through this project. Future developments for detectORE™ include analysing other elements and expanding the applicability of detectORE™ to other parts of the gold value chain.

The detectORE™ Technology is comprised of a Method and a Process; the detectORE™ Method relates to the analysis and the Process relates to its application to exploration. For the Method, PPPB uses a unique reagent which has been developed and exhaustively tested. The reagent, called GoldELIX3™ (now GLIX2020™), partially dissolves the gold from a geological sample from where it is simultaneously adsorbed onto a proprietary collector device. GoldELIX3™ has been precisely formulated through experimentation to ensure suitability for a variety of geological materials. Mixing the sample is critical to the dissolution-adsorption process, and so machinery and mixing parameters (such as speed, time and type of agitation) have been optimised. This resulted in a reduced time of analysis down to 6 hours, from the initial prototype 24 hours by CSIRO. Furthermore, the Method (including reagent and analysis) is stable over a range of temperatures commonly encountered in the field. The analysis of the collector device by pXRF determines the amount of gold in the sample; PPPB tested several pXRF instruments (including multiple machines from the same company) and found most could determine gold using detectORE™ proprietary software, demonstrating independent advancements by PPPB from the limitations of the original software. To ensure validation of the detectORE™ Method, laboratory standards and certified reference materials (CRMs) were used. Linear calibration curves on laboratory standards, and the analysis of CRMs, indicate that gold was determined from ~10-20 ppb concentrations up to tens of ppm.

The detectORE™ Process of analysing gold in the field empowers the user to make decisions on-the-fly. For example, a sample collected in the morning may be analysed by the afternoon so that a decision can be made on where to collect samples the next day based on the results of the previous day. This approach is called Reactive Sampling™. To optimise the samples used in the detectORE™ Method, tests were undertaken on particle fineness, weight and sample type. It was shown that sample sizes of 250 g were optimal for the detectORE™ Process, providing enough gold to be detected and reducing the inhomogeneity around gold commonly encountered in field samples. Furthermore, PPPB demonstrated through extensive experimentation that a representative sample for the detectORE™ Method could be obtained by sieving through a 1 mm sieve; this approach was applicable to soils and drill spoil alike. Minimising the amount of sample preparation and maximising the sample size are key features of the detectORE™ Process and make it suitable for field use. Many different sample types were tested from around the world. Samples sourced from Africa, North America and Australia using different gold mineralisation styles and sample types indicate the Method has a wide range of applicability. Samples containing refractory gold commonly cause extraction problems with analytical techniques such as aqua regia. The detectORE™ Method is no different but testing has shown that, even with samples of this type, there is indicative gold recorded by the Method.

The detectORE™ Technology will potentially save exploration companies considerable resources. With knowledge of gold concentrations in the field, samples can be sent for further analysis (if showing anomalous gold), given a lower priority or ignored if they show low gold concentrations. This is particularly important if the normal analytical laboratory is distant or, as in the current circumstances, overwhelmed with samples causing considerable delays in results turnaround. Such delays may have significant monetary impact on a company budget.

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

MRIWA Project M0543

Field-based XRF for prompt Au analysis

1 INTRODUCTION

1.1 Project background

CSIRO mineral research divisions have been at the forefront of mineral exploration scientific applications to assist industry and multi-billion-dollar advances in geochemistry, geophysics and chemical analysis have made Australia more competitive on a world scale. The advancement of gold (Au) exploration is no exception and has greatly benefitted from applied CSIRO research including breakthroughs in laterite and calcrete methodologies. It is from this successful scientific stable that another novel piece of research has emerged with the potential to revolutionise the way we explore for Au.

In 2015, Dr Mel Lintern of CSIRO had an idea for a rapid and robust field Au analysis technology which he termed detectORE™. The detectORE™ Method processes a geological sample both collecting and concentrating the Au using a proprietary process. The Au is analysed using an off-the-shelf portable X-Ray fluorescence machine and an Au measurement reported using proprietary software. The idea was incubated over three-years and a patent was applied for and granted. The invention was advertised for worldwide tender by CSIRO for expression of interest (EOI) in a company interested in developing and commercialising the detectORE™ technology. Portable PPB Pty Ltd (PPPB) was incorporated in June 2017 with the intention of applying for the EOI and was subsequently selected as the successful bidder for the exclusive global license. The detectORE™ research and its potential to benefit mineral exploration has been presented at several conferences (AIG 2017, AEGC Sydney 2018, MEGWA Perth November 2017, RFG Vancouver 2018, Gold18@Perth, AGCC, AIG Exploration Geochemistry Seminar 2018) and gained considerable interest from mineral exploration companies. Most of these conferences have required abstracts and extended abstracts that have been peer reviewed and published e.g. Lintern, 2018[1]. These presentations described the preliminary research undertaken by CSIRO on detectORE™.

PPPB were confident that the CSIRO data indicated the technique was viable and that product development had the potential to translate into a commercial product; thus detectORE™ for PPPB had every chance of success. Nevertheless, comprehensive research was required to substantiate the preliminary data, interpret substantial new data, assemble case studies around the new technique to fully realise its potential, and to establish QA/QC around the analytical method. The CSIRO research formed the basis for a program of further research up to the stage of commercialisation. Initial results from this new disruptive technology suggested there was strong opportunity to realise this game-changing goal. Each analysis takes less than 24 hours, greatly increasing the effectiveness of Au exploration programs and decreasing the time to discovery. To the best of PPPB's knowledge, no other activity was being currently undertaken in this field of research. An earlier attempt to develop a viable field method using different technology by DET CRC had failed and been abandoned.

The PPPB Research Laboratory Facility was commissioned in February 2019 at 18 Sorbonne Crescent, Canning Vale, Western Australia, with its purpose to investigate the feasibility of field portable methods for the rapid and robust analysis of trace elements in geological and environmental samples. Field Au analysis and reactive sampling™ was determined as being of critical importance to exploration and consequently MRIWA and several industry sponsors

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[1] https://www.publish.csiro.au/ex/pdf/ASEG2018abT7_4D

commenced supporting research and development of detectORE™ with MRIWA Project M0543. The project was scheduled to run for 12 months.

1.2 Project aims and objectives

The project had the primary objective of developing a method that analysed Au in the field which, at the time, could not be done. It was to be a robust, safe, field analytical method for Au exploration capable of achieving sensitivities of assay to approximately 10 ppb Au.

Secondary objectives included:

1. Analysis of certified reference materials (CRMs) and laboratory standards using a range of high-quality commercial CRMs to assess the accuracy and precision of the detectORE™ Method, expanding upon the initial tests undertaken by CSIRO.

2. Employing the detectORE™ Method to an extensive range of exploration sample materials, sourced from Western Australian mineral deposits and prospects with a preference given to those provided by the project sponsors. Test samples included were to be surface soils, augered sub-surface soils including lateritic residuum and calcrete, stream sediments, drill hole cuttings from RAB, AC and RC, saprolite and sap-rock. Sample preparation techniques, designed for the field, were to be developed tailored to these different sample media. Duplicate samples were to be analysed externally by traditional laboratory methods.

3. Evaluating the detectORE™ Method under field conditions. Suitable Western Australian mine site(s) and exploration project(s) were selected to test the robustness of the detectORE™ Method under typical conditions found in a field camp.

In addition to the practical experimental program of work, evaluation and further development of the software, used in the collection of the detectORE™ analytical data, was completed. The detectORE™ Method employs an off-the-shelf portable X-Ray fluorescence (pXRF) machine. Raw output from the pXRF is processed to maximise precision and accuracy of the Au determination. The optimisation of machine count rates, counting statistics, noise reduction, energy window parameters, analysis time and competing interference minimisation were fine-tuned to provide the best precision and detection limits.

Feedback from the practical test work was used to aid the development and deployment of detectORE™, and industry uptake. It was highly likely that changes to the detectORE™ methodology would arise out of the experimental work leading to improvements and optimisation of the original and preliminary CSIRO method, with the aim to produce a commercially viable product that would be a great benefit to those exploring in remote parts of WA and other parts of the world.

1.3 Project work plan

A step-by-step research plan was implemented to provide the road map for the evaluation and advancement of detectORE™ (Figure *1*). A literature search was undertaken to ascertain if there was any similar work that had been, or was being, undertaken, and was ongoing throughout the project. In summary, no research on direct Au analysis using pXRF for mineral exploration was identified from the literature search.

CSIRO was commissioned to undertake parcels of work, given its specific capability around metallurgy and with the determination of specific analytes pertinent to the efficacy of detectORE™.

1.3.1 *Data collection and evaluation*

The research project generated considerable amounts of data. The raw data generated by the pXRF machine are outputs in the form of an energy vs counts spectra for each analysis. These spectral data sets were used to improve the signal to noise ratio of the Au analysis particularly for low Au concentrations, where distinguishing the Au excitation energy peak from background noise and competing interferences was important for determining detection limits. Software generated by CSIRO and included with the license agreement assisted in completing this task but advances in this aspect of the research were undertaken as needed.

Data generated from each analysis and counts were compared with:

- (i) counts from detectORE™ Au standards
- (ii) CSIRO software data output and
- (iii) data outputs internally generated by algorithms from the pXRF instrument.

Sample integrity in collection and process, integral with quality assurance and quality control are critical for any analytical project, particularly for a new method. Release of data prematurely and without rigorous statistical analysis would jeopardise trust in the results making it difficult to recover integrity and increase risk for product acceptance. Correlation, Student t-test and regression analysis were used to compare measurements between detectORE™ and other analytical methods.

1.3.2 Expected outcomes

The nature of research is unpredictable, but a step-by-step approach was adopted in the research work plan to assist in isolating the variation when compared with other data. However, PPPB were optimistic and expected to confirm the promising preliminary results by CSIRO and generate considerable additional data, for example, from new experiments using specific sample preparation procedures. Testing detectORE™ on a range of geological and regolith materials was expected to generate considerable data. Obtaining precision with Au analyses is inherently difficult even with traditional laboratories and, thus, some detectORE™ measurements were anticipated to be better than others; e.g., samples with coarse particulate Au produce more erratic data than samples with finely dispersed or nanoparticulate Au.

1.3.3 Deliverables

1. A series of analytical reports delivered as quarterly reports and summarising progress towards milestones
2. A final presentation to sponsors of the project
3. A Final Report
4. A peer reviewed publication (post project)

1.4 Acknowledgement of sponsors

We acknowledge that support for the project has come from many companies and institutions. The Minerals Research Institute of Western Australia (MRIWA) sponsors for the Project were:

Government of Western Australia (MRIWA)
Agnico Eagle Mines Ltd
Bellevue Gold Ltd
Gold Road Resources Ltd
Gold Fields Australia Pty Ltd
Barrick Gold Corporation
Centerra Madencilik A.S
Perseus Mining Ltd
Newcrest Operations Limited

In addition, logistic and monetary support was provided by non-MRIWA sponsor exploration companies:

Exore Minerals
Jerrett Canyon Gold Llc
Great Boulder Resources Ltd
Riversgold Ltd
Titan Minerals Ltd
White Rock Minerals Ltd

Additional input and support was received from:
OREAS
Geostats Pty Ltd
Olympus Instruments
Portable Spectral Services
SciAps Inc
Bruker Corporation
Thermo Fisher Scientific
Portable Analytical Solutions Pty Ltd
CSIRO

All this support is received with thanks.

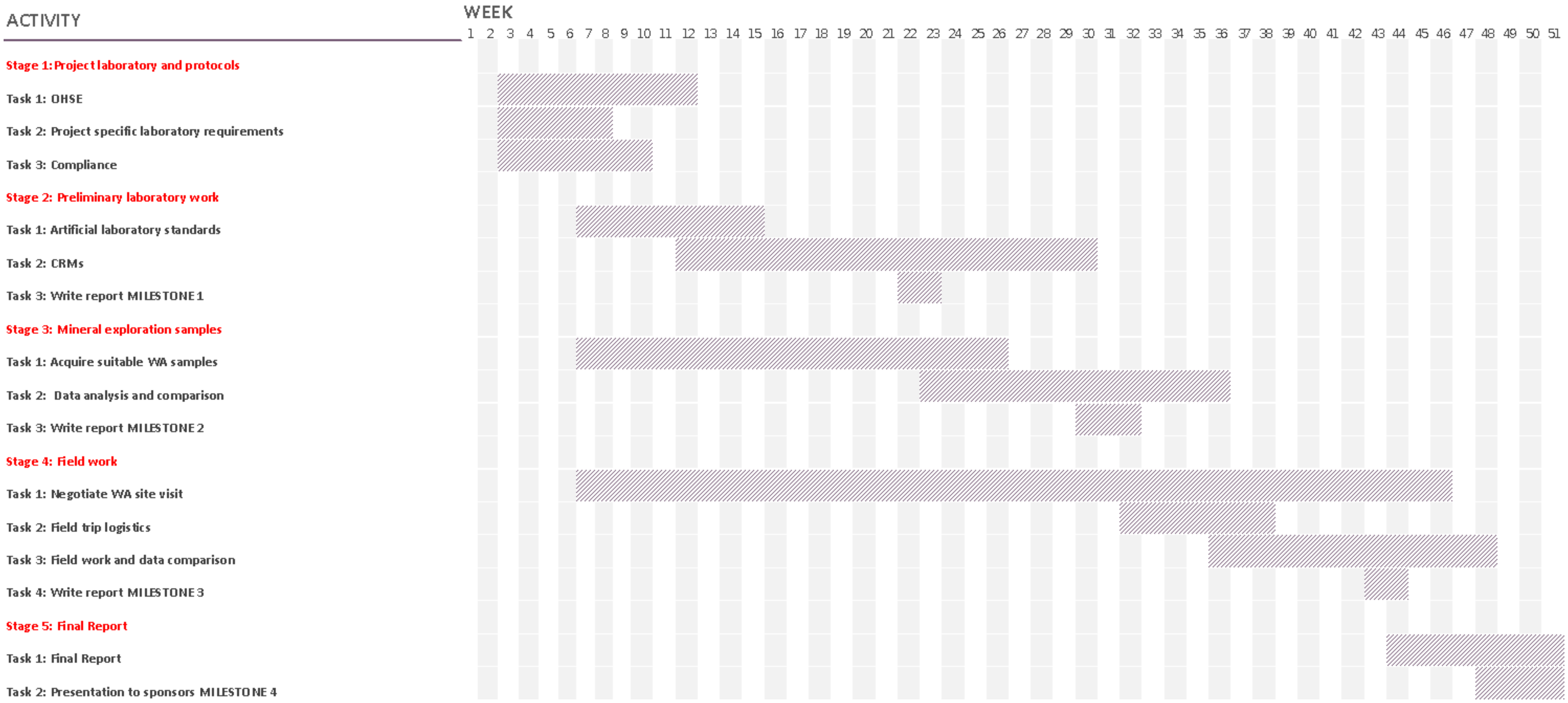


Figure 1: Gantt chart showing timeline of activities

2 EXPERIMENTAL METHODS AND DETAILED FINDINGS

2.1 Introduction to methodology

The detectORE™ Process is aligned with the principles of exploration geochemistry. DetectORE™ is a fit for purpose technology designed to maximise the strengths of adaptive or 'reactive sampling™'. Reactive sampling™ is a term coined by Mel Lintern at PPPB and describes a field methodology whereby samples collected and analysed one day can be acted upon the next by collecting and analysing adjacent sampling areas. Anomalous soils or drill cuttings can be followed up faster than background; this is not to say that other areas will be excluded from sampling and analysis, but the priority for exploration can be more flexible. Prioritising targets is particularly important for companies working on a low to medium-sized budget or working in areas where exploration is remote, costly and samples difficult to analyse.

Ideal characteristics required of an analytical method and to implement reactive sampling™ include fast, safe, cost effective, precise and ease of field use; detectORE™ aspires to possess these characteristics. Users of detectORE™ must be confident that an analysis is precise (with accuracy a lesser concern) since exploration geochemistry is a discipline where differences are important so that areas with higher grades (anomalies) and low grades (background) can be differentiated. Typically, anomalous areas may be followed up with further sampling in order to define areas of the highest grades, which may lead to commercially significant discoveries. With detectORE™ the pathway to discovery is accelerated because the follow up sampling of anomalous areas can be conducted rapidly (Figure 2).

Figure 2: The detectORE™ Process

2.2 The detectORE™ Method

The detectORE™ Method can be described in four parts (Figure 3):

1) A geological sample e.g. 250 g is weighed into a container. To this is added a pre-weighed 'puck' of chemicals, water and a collector device (CD); alternatively, a liquid reagent containing the dissolved chemicals is added. The reagent and CD are referred to collectively as the detectORE™ 'widget'. Compositional details of the widget are proprietary to PPPB.
2) Several tens of samples are processed for each batch in the mixer.
3) The containers are mixed for a period e.g. 6 or 24 hours using fit-for-purpose mixers.
4) The CD is removed and washed with water to remove adhering geological sample; excess water is removed and drying between tissue paper is found to be sufficient.
5) The CD is scanned by pXRF, data exported, analysed using detectORE™ software and the Au content measured; the CD is unaffected by the pXRF analysis and can be re-run multiple times if required.

The experiments were designed to evaluate and substantially improve upon the detectORE™ Method initially developed at CSIRO.

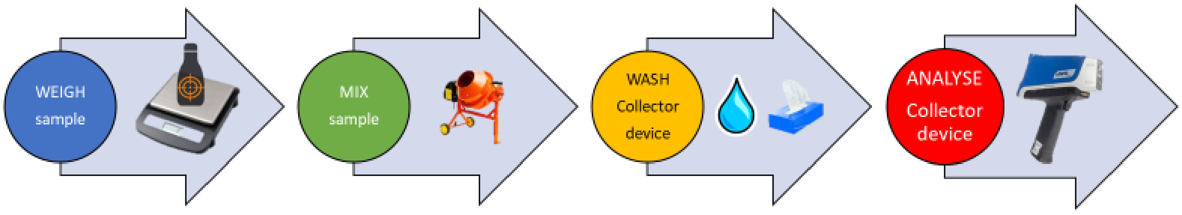


Figure 3: The detectORE™ 4-step Method

2.3 DetectORE™ uses a partial extraction method

Despite its use of penetrating x-ray technology, the detectORE™ Process is not a total analysis like fire assay or neutron activation (see 'The detectORE™ Method'). It is important to distinguish accuracy from precision. For exploration geochemistry, the emphasis is on precision rather than accuracy (Figure 4). Geochemical exploration does not require samples to be analysed for their total element content and so with Au other more cost-effective methods are commonly employed including aqua regia or cyanide digestion (partial digestions) rather than fire assay.

DetectORE™ uses a similar aqueous partial digestion approach. Partial digestions are commonly used in geochemical exploration for investigating more labile elements which are indicative of active dispersion mechanisms that may be used to vector towards mineralisation. Partial digestion methods, however, may not analyse some Au in the sample and therefore underrepresent the strength of the anomaly. This can happen in several ways for example:

1) The Au is locked within quartz or sulphides and are poorly leached, particularly if the sample is not finely ground;
2) The strength of the reagent is reduced by the sample;
3) The form of the Au cannot be leached e.g. Au as a telluride.

While these factors may increase the risk of not identifying the full tenor of mineralisation or an anomaly in a particular sample using partial extraction techniques, it is arguable that adjacent samples in a drill hole may be more amenable to extraction because of slightly differing mineralogy or increased weathering so that the drill hole itself is classified as anomalous. Risk increases for mineral exploration drilling in a greenfield prospect where little is known about a mineralisation style and samples are tested and misclassified e.g. false negative. However, despite these concerns, other partial extraction methods such as aqua regia digest, that has been used for decades, is still probably the most common analytical method used in mineral exploration for Au.

Another related risk factor particularly for Au analysis is the 'nugget effect'. Gold naturally forms accumulations during mineralisation causing problems with representative sampling. Samples with coarse Au are rarely analysed accurately or precisely with a single analysis and commonly laboratories undertake multiple analyses if high concentrations of Au are encountered in order to get a more precise estimation of the actual concentration of the sample. Typically, several kilograms of sample are pulverised in a laboratory and from that 25 g, 40 g or 50 g is extracted for the analysis. Taking larger samples such as 250 g would be much more representative but it is not practical to apply aqua regia to such amounts. Previously, astute explorers may have tried other partial digestion techniques such as cyanide digestion which use larger sample sizes in order to attain better precision. DetectORE™ typically uses 250 g of material for its analysis.

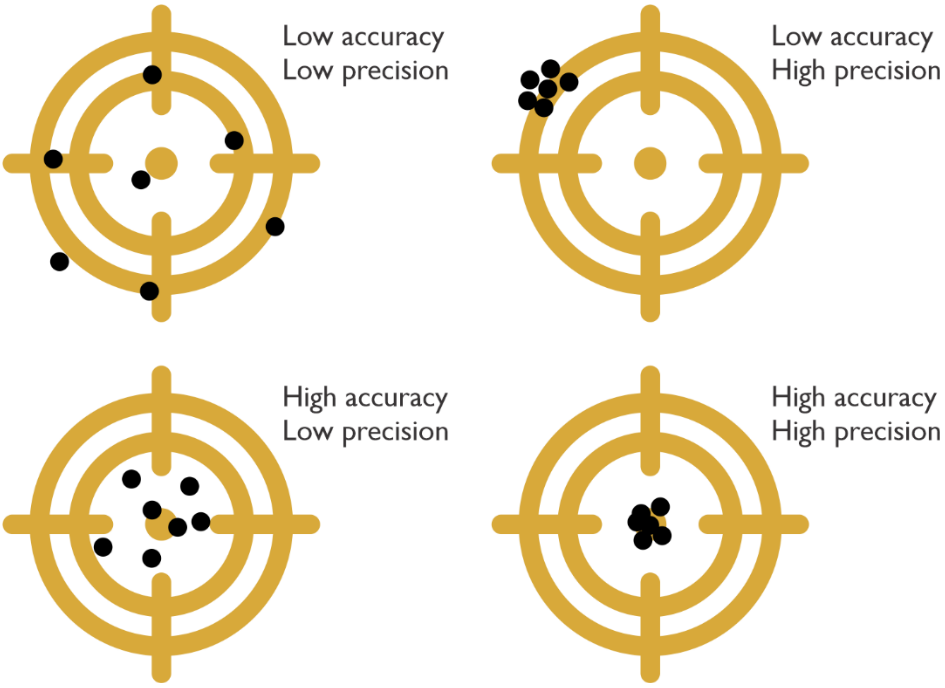


Figure 4: Comparison of precision and accuracy.

2.4 Optimisation of CDs and chemistry: the Widget

Method efficacy for detectORE™ was tested in several ways. Firstly, the ability of a CD to adsorb (recover) a range of added Au concentrations was assessed for precision and ability to produce a predictable linear or first order polynomial fit standard (calibration) curve; a CD that demonstrated poor precision in a wide calibration range of synthetic Au standard solutions was not considered further - a CD was rejected early in the evaluation procedure because of failure with precision expectations. Secondly, the digest solution (reagent solution) needed to satisfy the criteria of rapidly dissolving Au, ease of use, robustness, handling, safety and toxicity. An upper limit of 24 hours for the detectORE™ process was in place for samples using Widget 1 (W1). Thirdly, a CD and digest solution were tested together using CRMs to ensure performance was not affected by combining the two components e.g. W1 digest may not work well with W2 CD. Fourthly, the Widget was tested against the complete range of CRMs (over 40). If metals or other components were dissolved and were concentrated on the CD that interfered with the pXRF analysis for Au, the Widget had limited use or may only be used for samples not containing these interferences. Once the Widget had satisfied requirements, final testing was undertaken on various sponsor samples; as these were in limited supply, these samples were used judiciously.

DetectORE™ experiments were designed to improve field practicality, quantify precision, expand upon the number of sample types and lower detection limits. Evaluation of an experiment was based on mixing the sample with the Widget (reagent and CD), concentrating the Au, and analysing the CD for Au by pXRF; the improvement in refinement in the Widget chemistry could be measured as output from the pXRF. Any experimental variable could be changed and then samples re-tested to measure a change in precision or leaching efficiency as output from the pXRF. As with reactive sampling™, experiments or testing were iterative, and results obtained rapidly, with results from sample analyses or changes to method interpreted within 24 hours. Spiked addition-recovery is another experimental tool commonly used in analytical chemistry where a known amount of analyte e.g., Au is added to a sample or reagent, so the amount of additional Au “recovered” on the CD is measured by pXRF and compared with expected results; this is particularly important to monitor the effects of sample on the Method.

2.5 Certified Reference Materials

PPPB purchased from OREAS (oreas.com.au) most of the CRMs that contain certified Au concentrations and a series of uncertified reference materials. OREAS CRMs are commonly made by blending Au containing high grade material with barren pulverised rocks. Two high Au grade OREAS CRMs (62f and 216b) were selected to exhaustively test the efficacy of the detectORE™ experiment batches.

OREAS 62f was prepared from coarse reject splits of Cracow Au-Ag ore samples blended with Blair Duguid barren andesite. Cracow is a low sulphidation epithermal deposit hosted by meta-andesitic volcanics. High grade Au mineralisation occurs within fissure quartz veins and is associated with zones of silicification, present as quartz lode breccia and as quartz vein breccia. It has an Au concentration of 9.71 ppm.

OREAS 216b was prepared from a blend of Archean greenstone-hosted Wilber Lode primary ore from the Andy Well Gold Mine and barren Cambrian greenstone sourced from a quarry north of Melbourne, Australia. The Wilber Lode is a shear-hosted, narrow vein, quartz lode-style Au deposit situated within the Meekatharra-Wydgee greenstone belt in the Archean Yilgarn Craton of Western Australia. It has an Au concentration of 6.66 ppm Au

These two particular CRMs (25 g) were diluted (10:1) with barren regolith materials (three types, 225 g to make 250 g in each container) to reduce research costs tenfold during process evaluation. A selection (sometimes all) of OREAS standards is typically run through advanced new widget formulations.

2.6 Sample processing focussed on field analysis

Theoretically, by avoiding pulverising in the field, the sample preparation procedure for detectORE™ would be simple, fast and cost effective. A protocol was developed by preparing drill cutting and soil samples for analysis that involved sieving (Figure 5), to reduce the amount of coarse material in a sample to be analysed (Figure 6). However, samples containing coarse Au are less likely to be accurately or precisely analysed by this process in comparison to a sample that was pulverised. The question was whether the sieving approach would provide enough extractable Au to be indicative of mineralisation and identify an anomaly. The protocol was tested using a series of case studies to determine whether sieving provided suitable data for the effective implementation of the reactive sampling™ method.

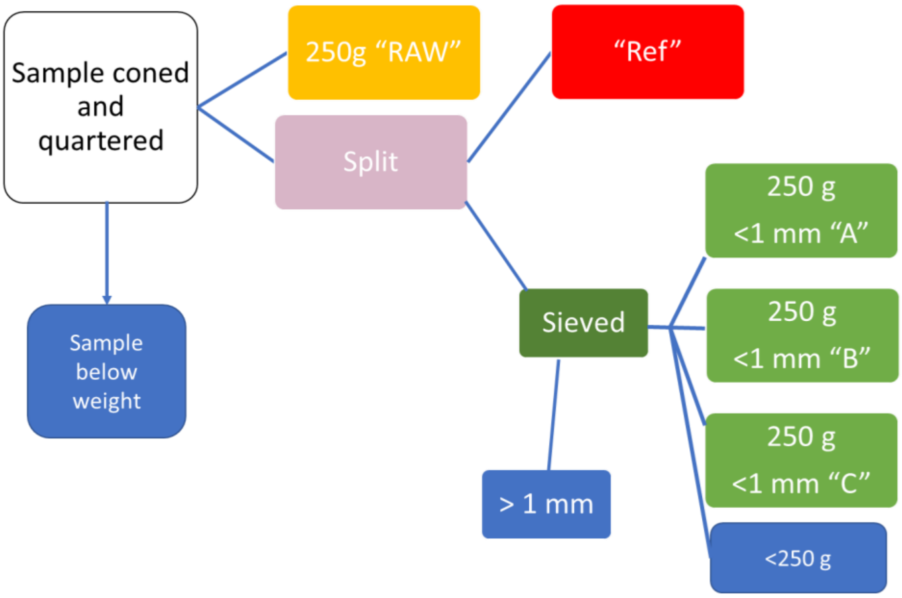


Figure 5: Protocol for field preparation of samples in the field. If sufficient sample was available more than one raw (or un-sieved) sample and/or more than three sieved samples were processed. If insufficient sample was available the raw sample was not processed.

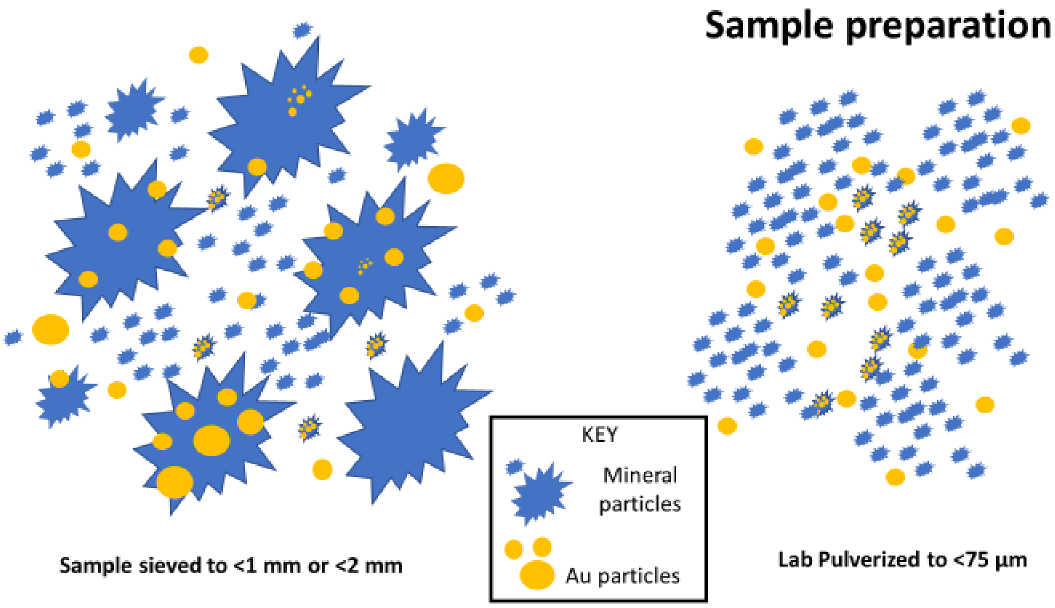


Figure 6: Sample containing Au can be pulverised or sieved. The DetectORE™ system is focused on field work and to try to avoid the requirement to pulverise a sample as occurs in a laboratory.

2.7 Several pXRF machines tested

We acquired 4 Olympus Vanta M machines for undertaking the bulk of the analyses. Six machines were tested from four manufacturers during the project. Agreements made between machine manufacturers and PPPB do not permit manufacturer names or the machines to be disclosed to the project. Machines were 'off the shelf' and not research style and can be purchased from the manufacturers. Results were supplied to manufacturers of their machine performance.

2.8 Laboratory standards and samples

Calibration or standard curves are commonly employed in analytical method evaluation to ensure that measurements undertaken are predictable, have a known precision and the range of analysis is known. The detectORE™ laboratory standards were CDs with a pre-loaded amount of Au adsorbed from a certified standard. The response of the pXRF and processed through detectORE™ software was related to the Au loaded on the CD; the original Au concentrations in the solutions were equivalent to 0, 1, 2, 5, 10, 20, 50, 100, 500 and 1000 ppb. The laboratory standards were run throughout PPPB experimental batches to assess the stability and precision of the CDs (and monitor the pXRF performance) over time.

The laboratory standards were routinely tested in each batch of experiments that were processed; they were used as a measure of pXRF performance, principally precision. Concentrations of Au for the CD standards are different to concentrations of Au determined in geological samples. This is because a laboratory standard CD accumulates Au from a 1 L (~1 Kg) solution of a known concentration of Au (made from 0-1000 ppb Au solutions). For samples, the Au is extracted from 250 g (or 25% of the equivalent standard solution) of sample. Furthermore, a laboratory standard has typically accumulated Au over a period longer than 24 hours while a sample digest has accumulated Au over a much shorter period e.g., 6 hours. The mass of Au on the CD was analysed by the pXRF and relates to the mass and geometry of the CD. The geometry of the CD approximated a constant and so the counts by the pXRF relate to the concentration (mass) of Au on the CD. It means, for example, that the 1000 ppb Au standard potentially contains the same amount of Au from a fully extracted 250 ppb Au sample. In practice the amount of Au on the CD generated from solution for the laboratory standards was much smaller than for the CRMs and samples since we did not extract all the Au for the following reasons:

1) The extraction time is much shorter (24 hours or less).
2) The sample may contain some refractory (or unextractable) Au.
3) The sample itself may contain material that affects adsorption of the Au on the CD (physical or chemical interference).
4) The CD may adsorb elements that interfere with the Au signal reported by the pXRF.

For this reason, we could only compare CD readings from the pXRF for samples that had been through the same conditions e.g. reagent type, solution volume, time of extraction, count time by pXRF, container type, mixing conditions. By including CRMs into the experimental sets we could compare values to expected CRM values to the non-CRM samples.

After the CDs had been read, they were stored in paper envelopes and may be reanalysed. At PPPB, we have a collection of many thousand CDs as part of a library of CRMs, standards and samples.

2.9 Improvements to software

The original software from CSIRO has been modified and adapted to suit new data acquired during the course of the project. As the number and variety of samples has increased, the complexity of the analyses has changed. From a simple analysis of the data with a limited suite of samples, the project has grown to testing a multiplicity of samples (from different geological settings), modified samples, and CRMs from around the world.

New proprietary software has been used for analysing data automatically from the pXRF machines. A specific program was constructed to enable the data to be processed from the raw data output from the machines. This software has undergone a series of upgrades as feedback from the experiments (precision and detection limit) was integrated back into the program (Figure 7). Modifications to the software have been undertaken to enable a work around solution for processing data from other instruments. For example, output from some instruments is as single files rather than composited data from a set of analyses into a single file.

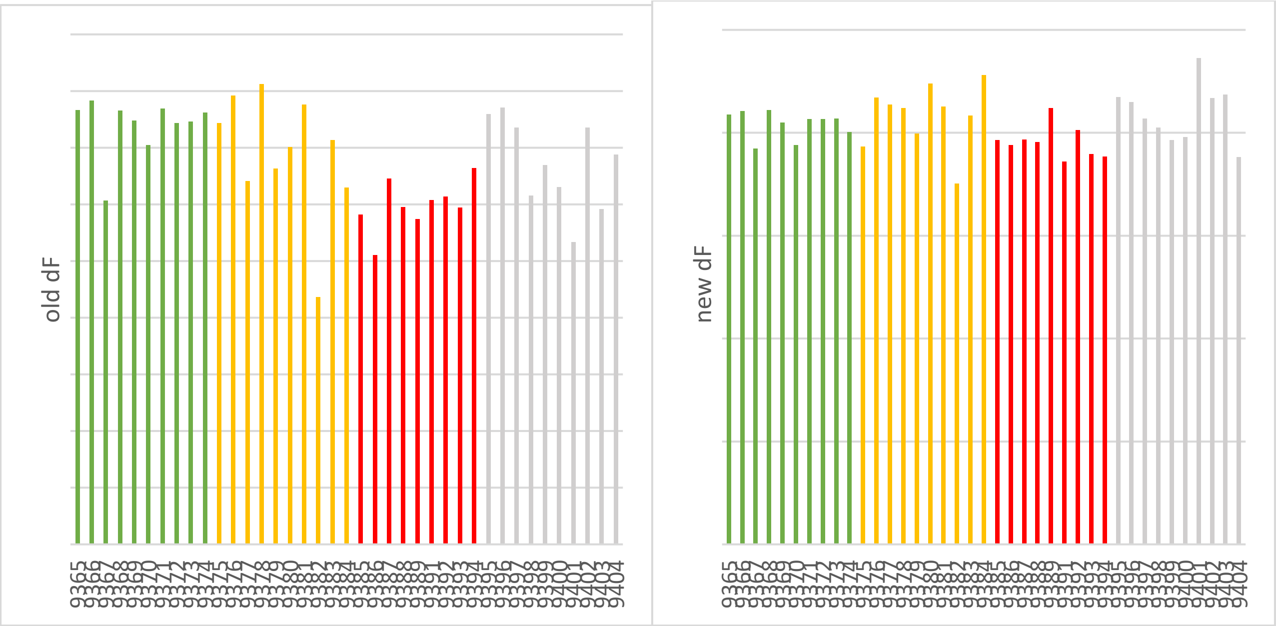


Figure 7: Two sets of samples (10 replicates each of 4 samples containing Au) analysed with two versions of the software. The new software has led to an improvement in precision. An example of data from old software compared with data from new software.

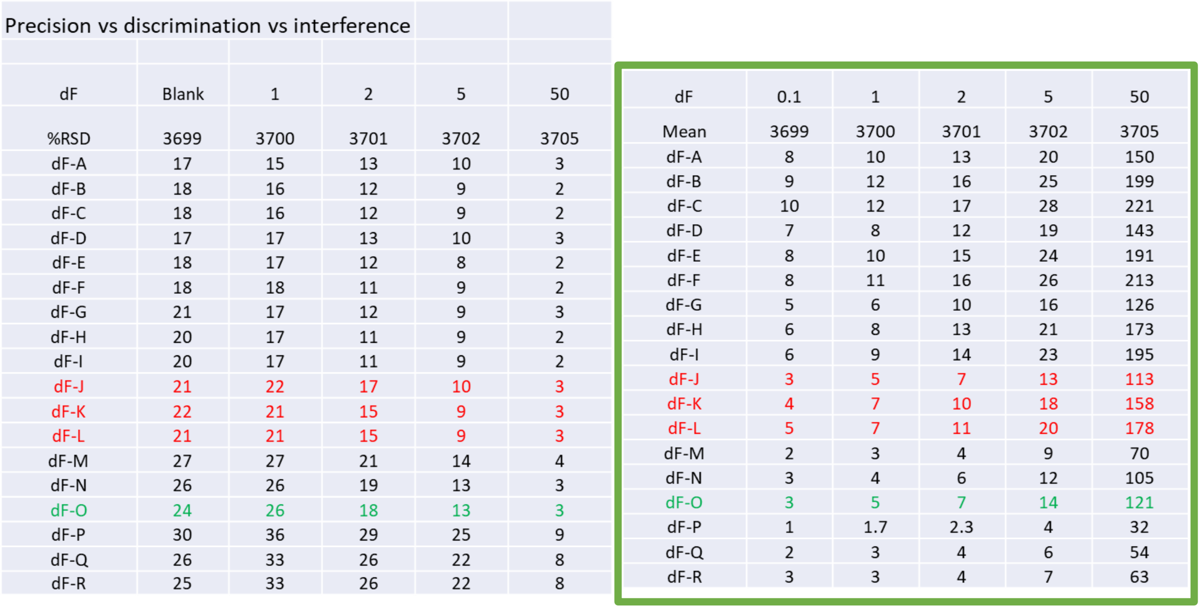
As the project developed it became evident that the measurements were more accurately reported as ‘detectORE™ factor’ or ‘dF’ rather than a concentration result. A dF is determined from the raw counts data. There are many different dF calculations possible depending on the statistical treatment of the raw data (Figure 8). Several dFs were tested, modified and/or rejected during the course of the project and this aspect of the project is ongoing.

For early stages of the project, concentrations were reported (rather than a dF) and compared to commercial laboratory results. Commonly, detectORE™ data were lower than the corresponding laboratory data since detectORE™ rarely extracts all of the Au from a sample. In order to avoid confusion (i.e., “why are my Au results low?”) we decided to report results as a dF. Since detectORE™ data are about comparative geochemistry the units themselves are not relevant (ppb or dF) but the comparative magnitude is.

For laboratory experiments too, data are ‘measurements of µg of Au on the CD’. So, if we changed our experimental conditions e.g. extended the time of extraction or changed the widget reagent, we would have another set of measurements which would only be comparative to a laboratory standard that had been prepared in exactly the same way. This would lead to a multiplicity of laboratory standards specifically tuned to the conditions of each experiment. Each experimental change would require new standards to calculate a Au concentration.

Therefore, measurements by the pXRF *stricto senso*, are now routinely reported as dF and their magnitude comparable to other results using a particular set of conditions used in the experiments. For any set of experiments or analysis of samples or CRMs, conditions are constant and individual results are comparable. It is only once we have locked in the conditions for detectORE™, after exhaustively testing all the variables, that samples will be able to be compared between sets or runs.

As the numbers of samples increased so did the appearance of interfering peaks from elements that had dissolved and accumulated with the Au on the CD or from sample contamination of the CD itself from incomplete CD washing. Interference peaks were not unexpected and are from elements with similar energy values to those of Au. Accordingly, new dFs were generated to eliminate these whilst minimising the effect on the Au signal. Changing the dF to allow for this circumstance commonly led to a lower Au signal (higher detection limit) and poorer precision. Thus, the dF maximising the greatest Au signal or the highest precision may not be the most appropriate to use because of the interfering elements which may lead to spurious Au results.



Precision vs discrimination vs interference

dF	Blank	1	2	5	50
%RSD	3699	3700	3701	3702	3705
dF-A	17	15	13	10	3
dF-B	18	16	12	9	2
dF-C	18	16	12	9	2
dF-D	17	17	13	10	3
dF-E	18	17	12	8	2
dF-F	18	18	11	9	2
dF-G	21	17	12	9	3
dF-H	20	17	11	9	2
dF-I	20	17	11	9	2
dF-J	21	22	17	10	3
dF-K	22	21	15	9	3
dF-L	21	21	15	9	3
dF-M	27	27	21	14	4
dF-N	26	26	19	13	3
dF-O	24	26	18	13	3
dF-P	30	36	29	25	9
dF-Q	26	33	26	22	8
dF-R	25	33	26	22	8

dF	0.1	1	2	5	50
Mean	3699	3700	3701	3702	3705
dF-A	8	10	13	20	150
dF-B	9	12	16	25	199
dF-C	10	12	17	28	221
dF-D	7	8	12	19	143
dF-E	8	10	15	24	191
dF-F	8	11	16	26	213
dF-G	5	6	10	16	126
dF-H	6	8	13	21	173
dF-I	6	9	14	23	195
dF-J	3	5	7	13	113
dF-K	4	7	10	18	158
dF-L	5	7	11	20	178
dF-M	2	3	4	9	70
dF-N	3	4	6	12	105
dF-O	3	5	7	14	121
dF-P	1	1.7	2.3	4	32
dF-Q	2	3	4	6	54
dF-R	3	3	4	7	63

Figure 8: Different dFs were tested to look at precision (expressed as a percentage of relative standard deviation, %RSD) and discrimination between concentrations for 50 analyses. Data from laboratory standards 3699 (blank), 3700 (1 ppb), 3701 (2 ppb), 3702 (5 ppb) and 3705 (50 ppb) using different dFs (A-R) to examine which provided the least variation and the best discrimination. Not considered here (these are laboratory standards) is the effect of interfering peaks on the Au data in some samples. Consideration of interferences led to dFJ-L (red) and dF-O (green) for further evaluation.

2.10 Quality assurance and quality control

Critical for any analytical method is to be assured that the results reported reflect the concentrations of the analyte being measured. DetectORE™ is no different from normal laboratory methods except that the sample analysis is being performed by the explorer or agent. The data generated from the explorer are analysed by PPPB to determine whether supplied laboratory standards and CRMs are being analysed correctly. If these are analysed correctly, the assumption is that the samples are also being analysed correctly.

Errors occur for any analytical method, and it is important to quantify these so that an understanding of the magnitude of these errors is understood. Errors are erroneous measurements reported which differ from the true value. For detectORE™, Au measurements are reported as a dF value which is proportional to the concentration. Although the power of detectORE™ relies on comparisons between anomalous measurements and background, it is important that differences between samples are not missed because of reading precision e.g., the signal is stronger than, and distinguishable from, the noise.

Errors can be classified as followed: systematic (determinate), random (indeterminate) or gross (e.g., human). Potential errors in the detectORE™ process that can affect accuracy and precision include:

- gold concentration in natural sample variability e.g., due to coarse or nuggety Au in a sample (random);
- measurement errors in weighing out sample or dispensing reagents volumes (random, human);
- process errors (digestion, widget, human) – random and systematic;
- instrumental errors (systematic), S/N ratio (random);
- reporting errors (gross, human).

Errors can be reduced by:

- identification of potential errors through exhaustive testing of standards and samples;
- automation of procedures;
- exhaustive testing of processing samples;
- effective bar coding through processing samples;
- quantification of the systematic errors;
- inclusion of laboratory standards and CRMs;
- replicate analyses.

2.11 Laboratory standards and detection limits

The laboratory standards (from 1 ppb to 1000 ppb) show a quasi-linear standard curve (Figure 9). Linear calibration curves simplify determinations so that a simple correction is required between determination and concentration. In addition, the slopes of the standard curves assisted in evaluating different machines. The steeper the standard curve the more sensitivity the machine has and a potentially lower detection limit. The standards were used to compare before and after a pXRF detector window change. They were analysed for each batch run on the pXRF and showed high precision (commonly <2 standard deviation when compared to long term mean) throughout the project. They were analysed as a check on the pXRF machine functionality and as a check to ensure that the machine users placed the CDs on the pXRF correctly.

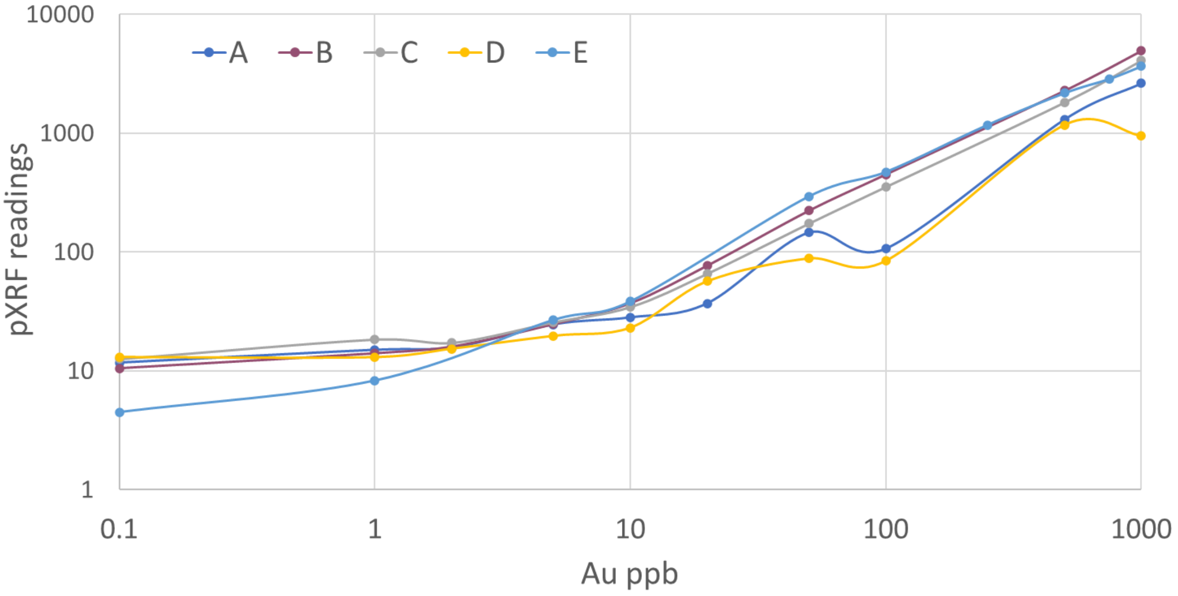


Figure 9: Calibration curves of 5 different CDs. Crucial to detectORE™ is the ability to discriminate between low concentrations of Au. Collector device E shows the best ability to do so in this experiment.

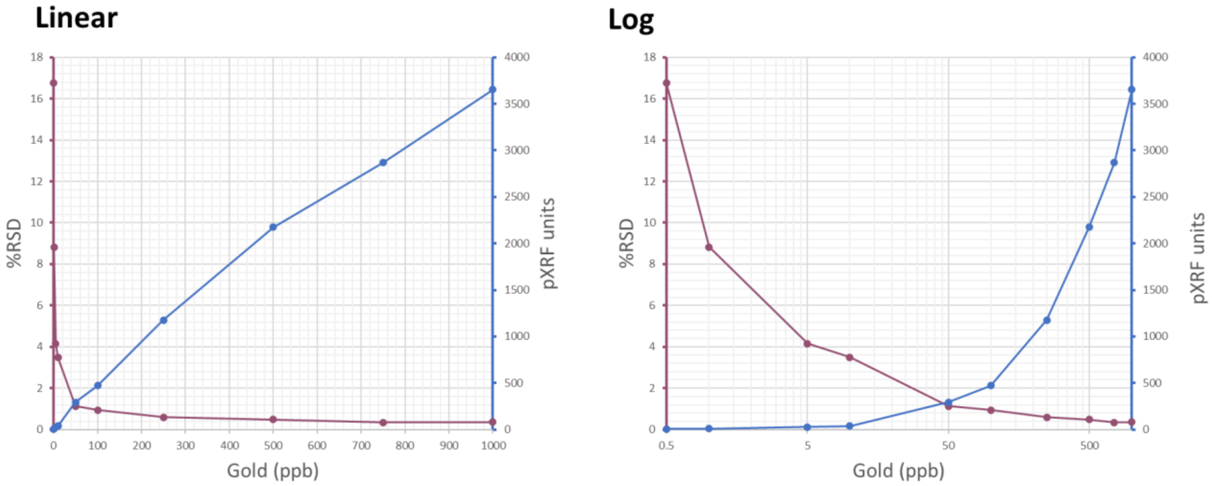


Figure 10: The calibration curves for W1 suggest a linear relationship over a wide concentration range (50 repeat analyses). The greater the Au concentration on the CD the more precision. Precision at 1 ppb Au using W1 is about 9% relative standard deviation (100xstandard deviation/mean).

Precision is measured as relative standard deviation after dividing by the mean (RSD) and is expressed as a percentage (%RSD) to enable comparisons. For example, for W1 standards, for 1 and 10 ppb, %RSD is ~9% and 4%, respectively; low concentration Au laboratory standards have lower precision than higher concentrations (Figure 10). Errors in making laboratory standards included pipetting errors, volume errors and adsorption differences of Au on the CD. Cross-checking and determination of these low amounts of Au on CDs was problematic as it would involve destructive analytical testing. A set of secondary Au standards was made using a standard addition of Au to solutions, to demonstrate the variability of Au measurements (Figure 11); secondary standards were not used in pXRF calibrations.

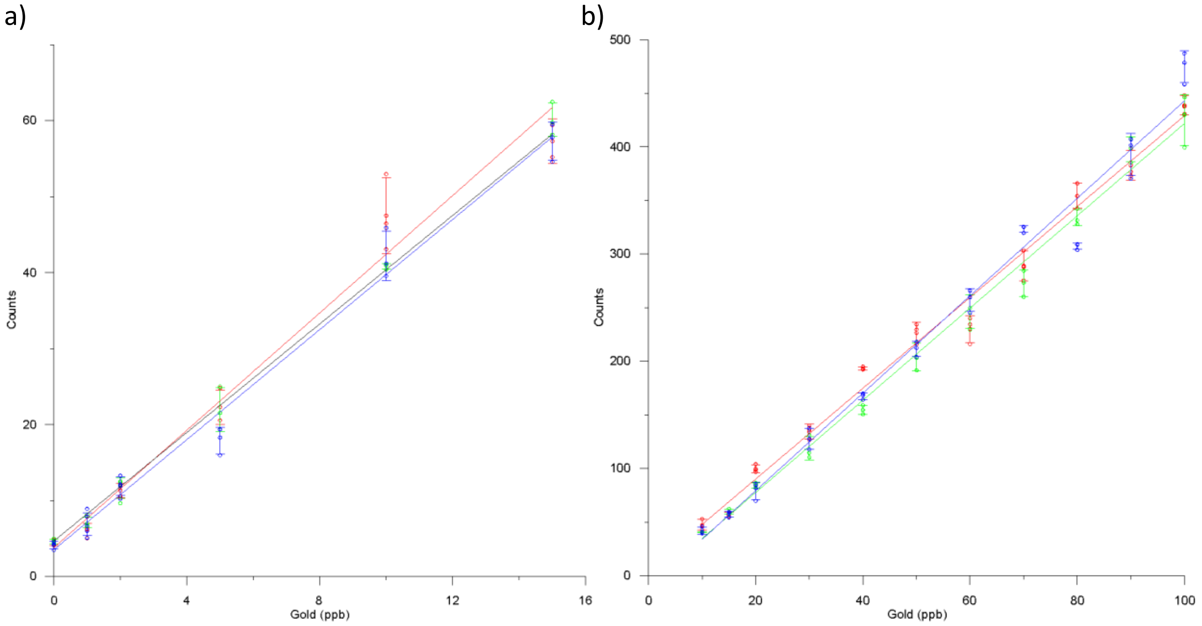


Figure 11: Calibration or standard curves for three sets of laboratory 'secondary' standards (red, green and blue) for a) 1, 2, 5 10 and 15 ppb and b) 10-100 ppb. Collectively they show linearity over a broad concentration range. However, differences between sets suggesting variation in the amounts of Au adsorbed on the CDs. The secondary standards are not used for any calculations but are shown here to illustrate the variability. Counts (y axes) refer to dF units.

For laboratory standards determining detection limits is relatively straightforward. Errors are well characterised since there is no interference from either geological sample from an improperly washed CD or other elements that may be adsorbed onto the CD which serve to interfere with the Au peak. There are several ways that 'detection limits' may be determined including LOB (highest measurement result that is likely to be observed for a ‘blank’ sample), LOD (lowest concentration that can detect analyte but not determine its amount) and LOQ (lowest concentration that can determine the concentration of an analyte). The relationship between the three measurements is shown in Figure 12. The LOQ may be expressed as a quantification limit (QL) where:

$$QL=10\sigma/S \qquad (1)$$

where σ can be estimated from the calibration curve or the standard deviation of the blank, and S is the slope of calibration curve. For W1 and 30 second counts, the LOD and LOQ for the laboratory standards was 0.5 ppb and 1.4 ppb, respectively (using the calibration curve method) and 0.8 ppb and 2.5 ppb, respectively, for the ‘blank’ method. For 60 second pXRF read times, the detection limit for laboratory standards was ~1 ppb. The blank CD reported a small amount of Au.

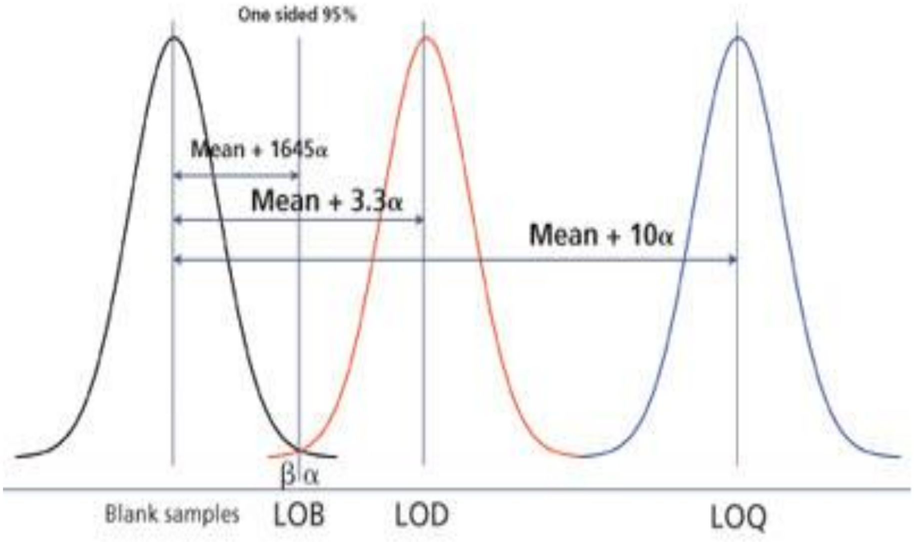


Figure 12: Theoretical relationship between Blank, LOB, LOD and LOQ measurements. For detectORE™ measurement data are compared between samples so, at the lower Au measurements, the veracity of the difference between the blank and the sample measurement is important.

For samples, the precision (standard deviation) was often higher than for laboratory standards because of the effect of chemically or physically adsorbed material on the CD. Interaction of the ‘x-rays’ with non-analyte material caused a loss of counts e.g. caused by deflection or attenuation of the returning waves to the detector. These were difficult to quantify for each sample because they are highly variable and would require many reading replicates and or increased analytical times. Alternatively, a standard or spiked addition of analyte approach in the sample may be routinely adopted but this would also lead to increased turnaround of results and higher costs.

In the field, ranking and classifying data for a given sample set can be applied in the same way that other geochemical data sets are already classified. For example, natural breaks in the data set can determine background (no Au detected), threshold(s) and anomalous dF values. This is better explained with real examples (see later sections). The discrimination between low concentrations is important for detectORE™ because it could spatially increase the size of anomalies in the field in samples that contain low ppb Au e.g., soil samples, and therefore broaden the anomalous target or allow increased sample spacing.

Low Au laboratory standards were analysed multiple times and at different count times. For 1 minute count time using collector W2, it was difficult to discriminate 1 ppb from the blank and 2 ppb was also not easily discriminated (Figure 13). For longer count times e.g., 5 minutes, the signal to noise was improved and the possibility of discerning Au at these low concentrations was improved (Figure 14).

Collector W1 was more sensitive than W2 and so lower Au concentrations could be better determined and discriminated (Figure 15).

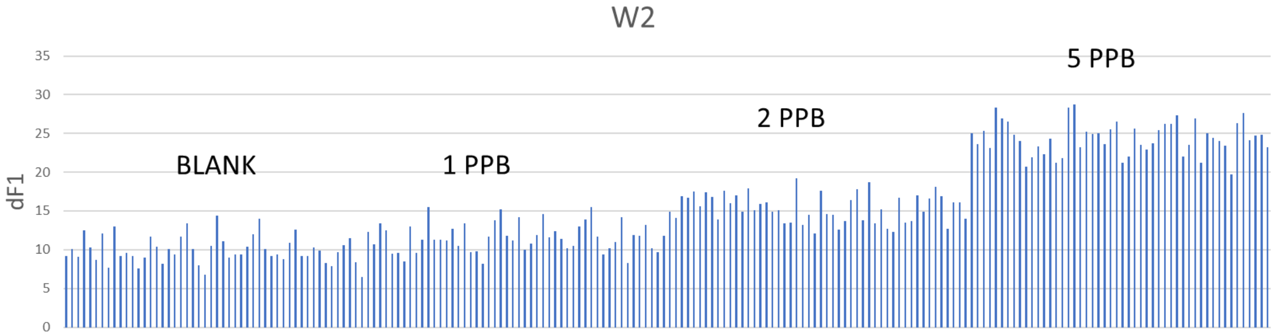


Figure 13: Replicate readings of low Au concentration (measured as dF) W2 laboratory standards showing lack of discrimination between blank and 1 ppb, and poor discrimination between 1 ppb and 2 ppb for 1 minute count times.

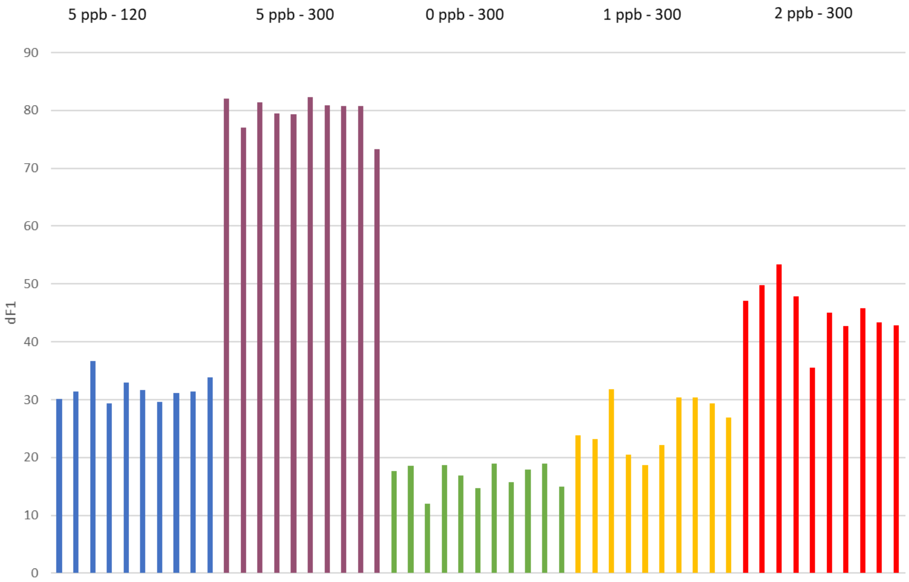


Figure 14: Replicate readings of low Au concentration laboratory W2 standards showing better discrimination between blank and 1 ppb, and for 1ppb and 2 ppb with 120 second or 300 second count times. 2 ppb in a laboratory standard is equivalent to 8 ppb in a 250 g sample.

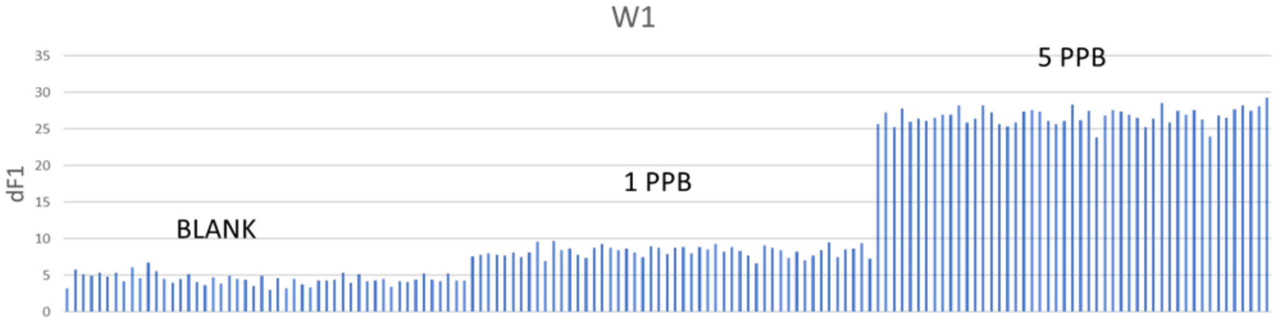


Figure 15: Replicate readings of low Au concentration laboratory W1 standards for 1 minute count times.

The beneficial effect of increasing count times on low Au laboratory standards using 50 replicate readings may be seen in Figure 16. Examples of analysing samples containing low Au for 1 minute and then 5 minutes are shown in Figure 17. Samples and laboratory standards were routinely run for

60 seconds using the Olympus Vanta M and were a compromise between precision and sample throughput.

A more robust design of laboratory standards was tested recently for field use and to be more precisely located on the pXRF window; replicates of these standards are shown in Figure 18.

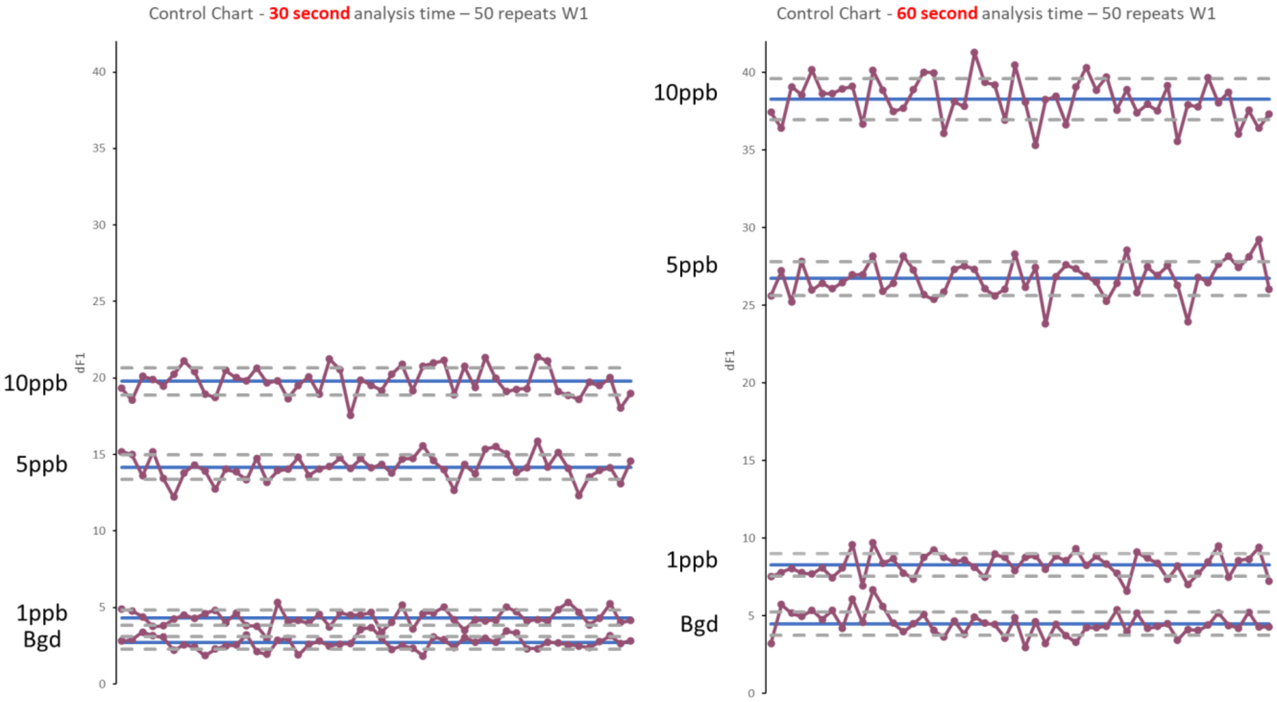


Figure 16: Gold analyses of synthetic Au standards using W1 show acceptable precision at both 30 second (left hand side) and 60 second (right hand side) count time by pXRF. Discriminating between 1 ppb and background (blank) CDs is better at 60 second count time compared with 30 seconds. The data demonstrate the stability of the machines being tested.

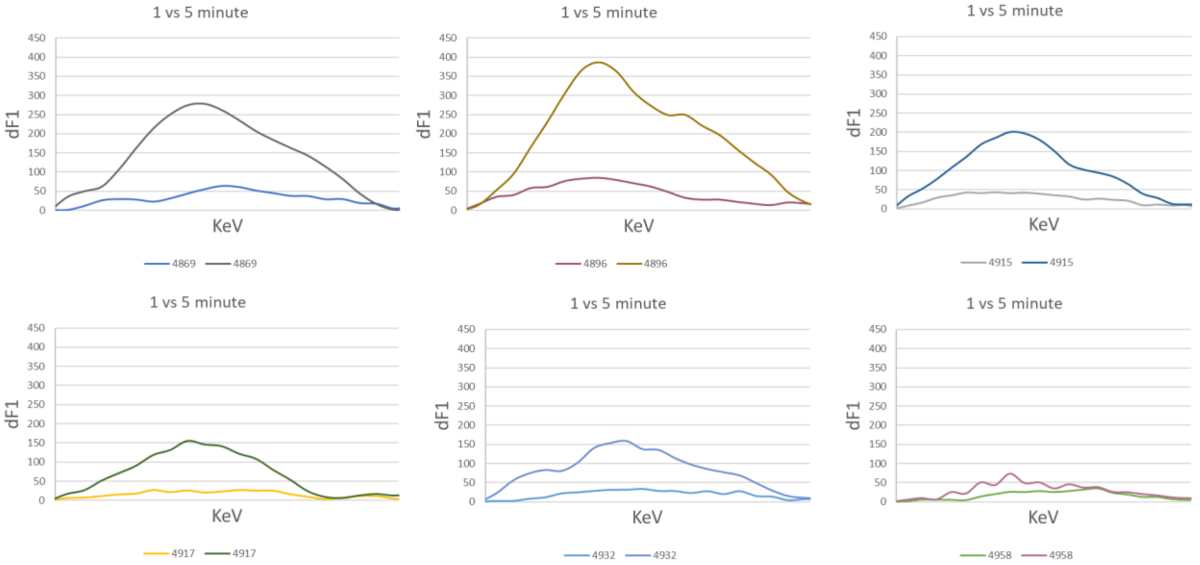
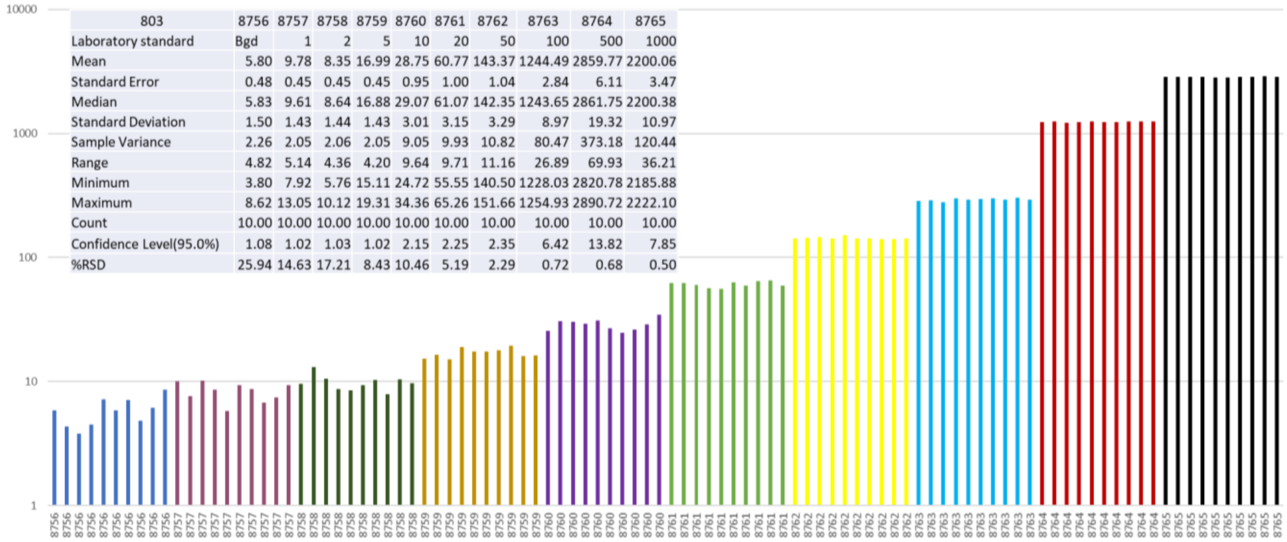


Figure 17: Longer count times increase the signal to noise ratio. Six examples shown here of 1 vs 5 minute counting times for samples. 5 minute count times show better defined peaks than 1 minute.



803	8756	8757	8758	8759	8760	8761	8762	8763	8764	8765
Laboratory standard	Bgd	1	2	5	10	20	50	100	500	1000
Mean	5.80	9.78	8.35	16.99	28.75	60.77	143.37	1244.49	2859.77	2200.06
Standard Error	0.48	0.45	0.45	0.45	0.95	1.00	1.04	2.84	6.11	3.47
Median	5.83	9.61	8.64	16.88	29.07	61.07	142.35	1243.65	2861.75	2200.38
Standard Deviation	1.50	1.43	1.44	1.43	3.01	3.15	3.29	8.97	19.32	10.97
Sample Variance	2.26	2.05	2.06	2.05	9.05	9.93	10.82	80.47	373.18	120.44
Range	4.82	5.14	4.36	4.20	9.64	9.71	11.16	26.89	69.93	36.21
Minimum	3.80	7.92	5.76	15.11	24.72	55.55	140.50	1228.03	2820.78	2185.88
Maximum	8.62	13.05	10.12	19.31	34.36	65.26	151.66	1254.93	2890.72	2222.10
Count	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00	10.00
Confidence Level(95.0%)	1.08	1.02	1.03	1.02	2.15	2.25	2.35	6.42	13.82	7.85
%RSD	25.94	14.63	17.21	8.43	10.46	5.19	2.29	0.72	0.68	0.50

Figure 18: Ten replicate readings (different colours) of a recently made set of laboratory standards (from left to right: blank/background, 1, 2, 5, 10, 20, 50, 100, 500, 1000 ppb). They are more robustly made than the original set and show comparable precision. Note the log scale.

2.12 CRMs – Certified Reference Materials

The CRMs were an important tool to measure performance between methods. Two 'workhorse RMs' (RMs are derived from CRMs) were manufactured which were used throughout the project and added continuity to the verification and comparative processes during the experimental work. The RMs were diluted from commercially available CRMs and so the reliability of the assay is assured and equivalent in QC performance as the commercially available CRM. It is intended that the RMs will be available from PPPB as we move forward and commercialise the detectORE™ technology.

OREAS and GeoSTATS CRMs were tested using detectORE™ and compared with other data. At CSIRO, detectORE™ was compared with GeoSTATS standards containing Au for the initial appraisal process for detectORE™. Good agreement between the two methods was found, which led to the detectORE™ Method being patented and licensed to PPPB (Figure 19). A variant of W1 was used for these tests either using 25 g of CRM or 250 g of CRM.

At PPPB, most test work was completed using OREAS standards to follow on from the CSIRO work which used GeoSTATS CRMs. DetectORE™ is compared with OREAS standards analysed by two methods (fire assay and aqua regia) in Figure 20 to Figure 23. Good correlation was found between the three methods (aqua regia, fire assay and detectORE™), remembering that detectORE™ results are reported as dF and not Au concentration. The data supports the findings from early CSIRO data (Figure 19). Some comparisons between CRMs, detectORE™, and laboratory analyses did not agree, and this has subsequently been shown to be with certain CRMs containing suspected refractory Au or high sulphide content. Samples with these same characteristics will be affected by recovery and report lower Au content (as dF) than for fire assay. Ongoing research within PPPB is addressing these issues.

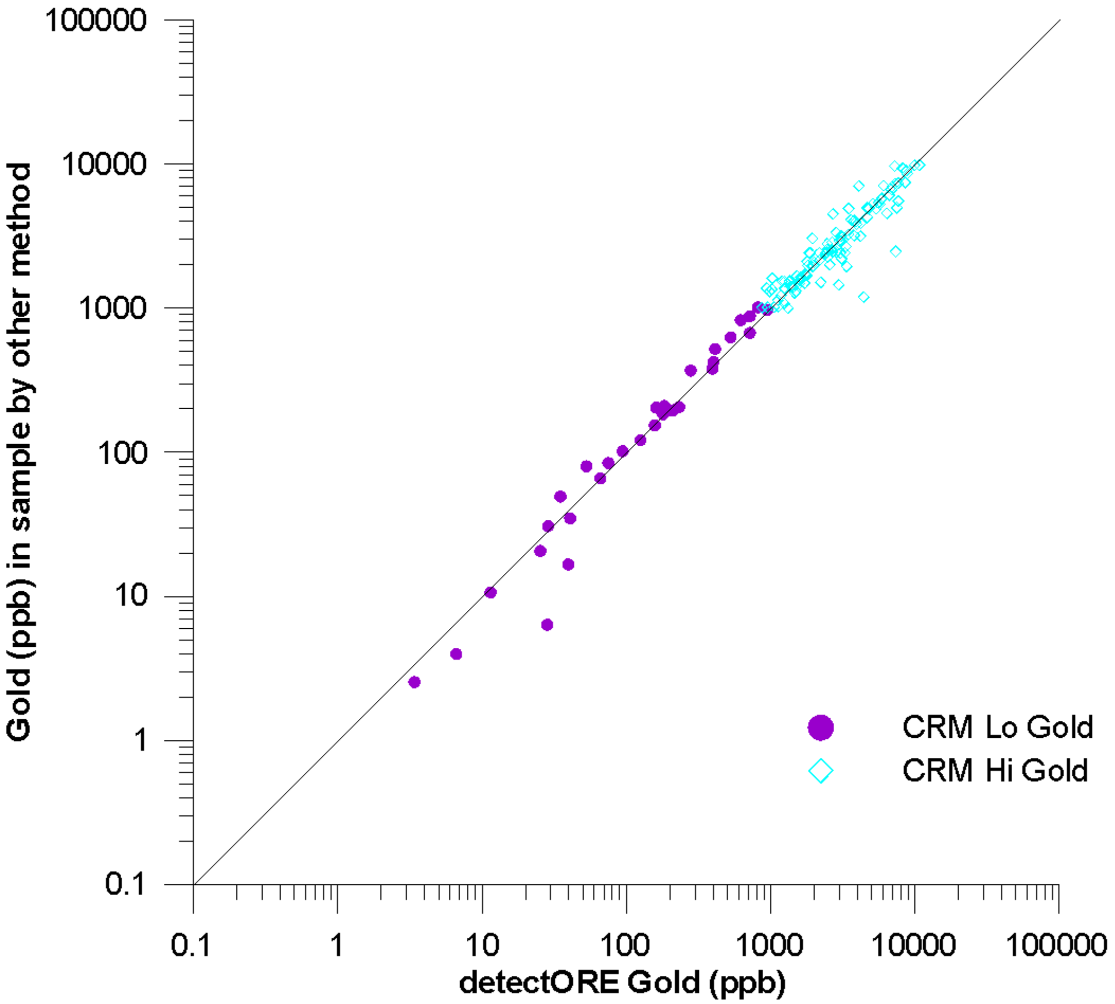


Figure 19: Early comparison between detectORE™ W1 vs GeoSTATS standards containing Au (fire assay and aqua regia) (from Lintern, 2018: https://www.publish.csiro.au/ex/pdf/ASEG2018abT7_4D). 'Lo Gold' was using 250 g of CRM and 'Hi Gold' was using 25 g of CRM.

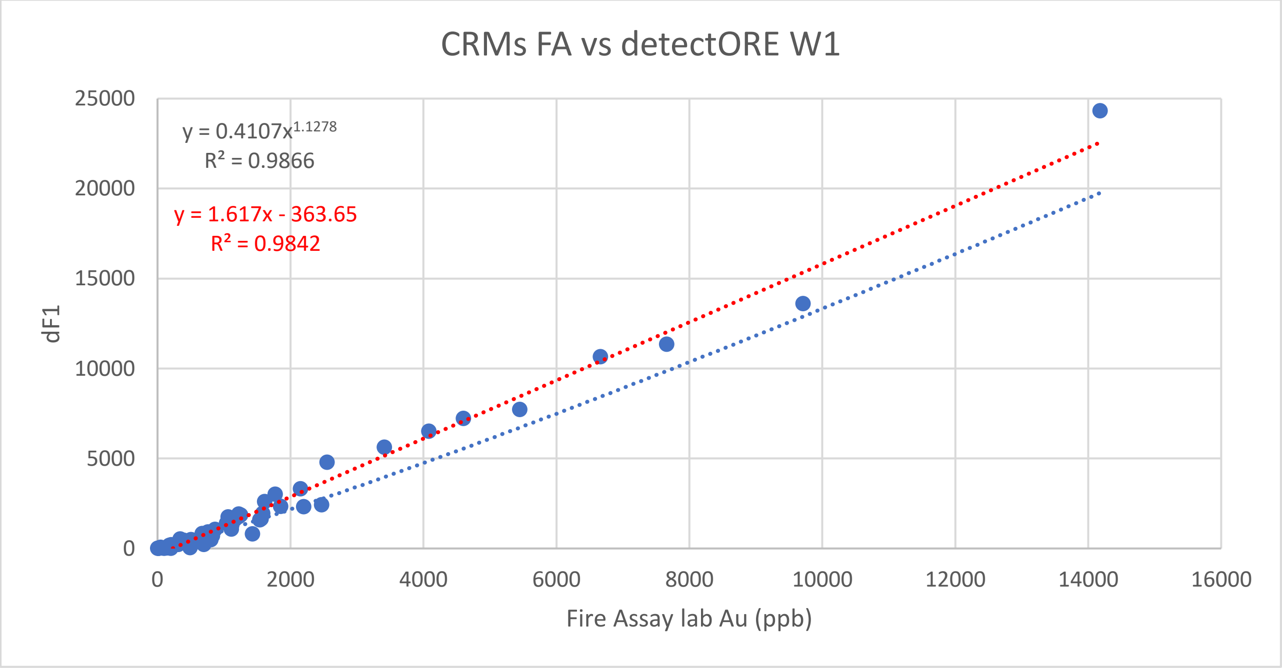


Figure 20: Scatter plot of OREAS Au standards analysed after fire assay versus detectORE™ W1 as dF1. Axes are linear. Two lines of best fit are shown.

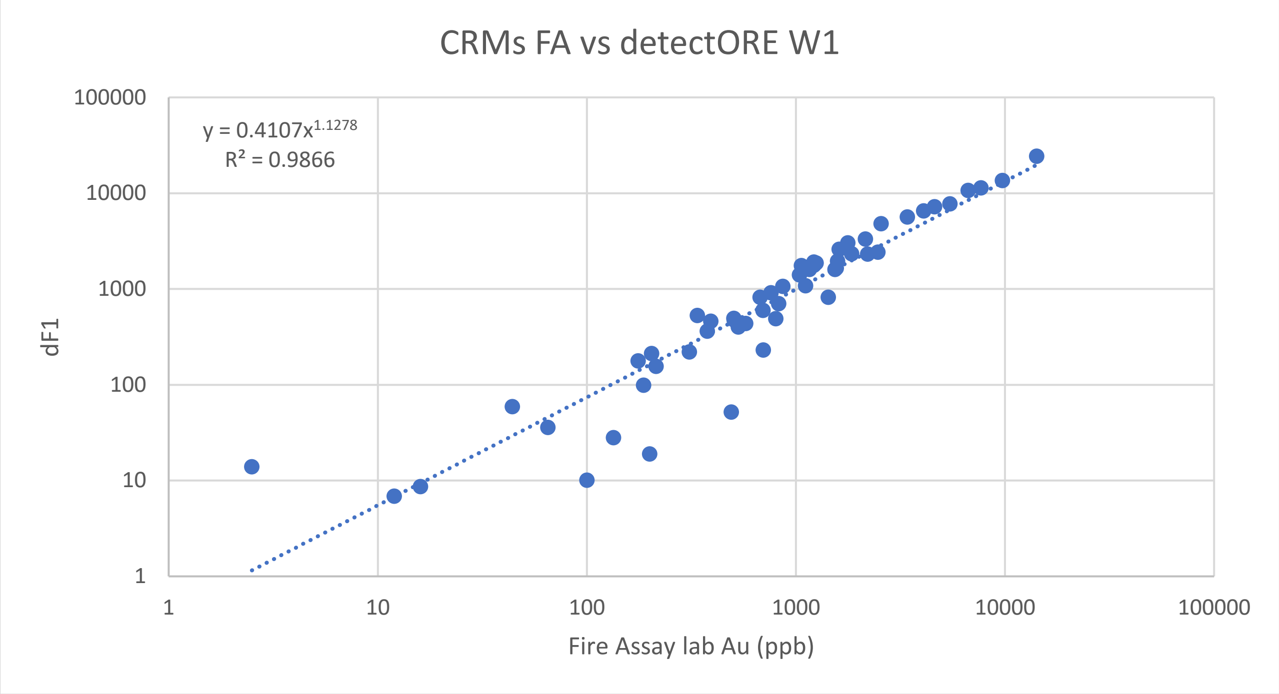


Figure 21: Scatter plot of OREAS Au standards analysed after fire assay versus detectORE™ W1 as dF1. Axes are logarithmic.

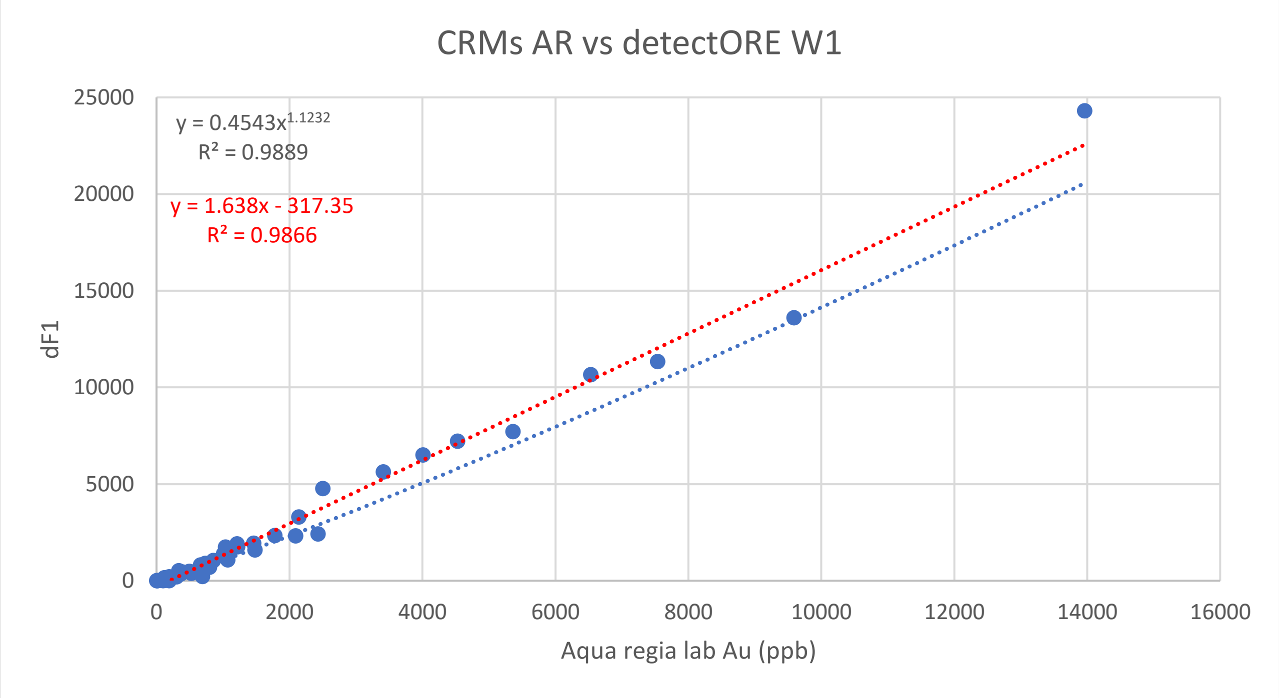


Figure 22: Scatter plot of OREAS Au standards analysed after aqua regia versus detectORE™ W1 as dF1. Axes are linear. Two lines of best fit are shown.

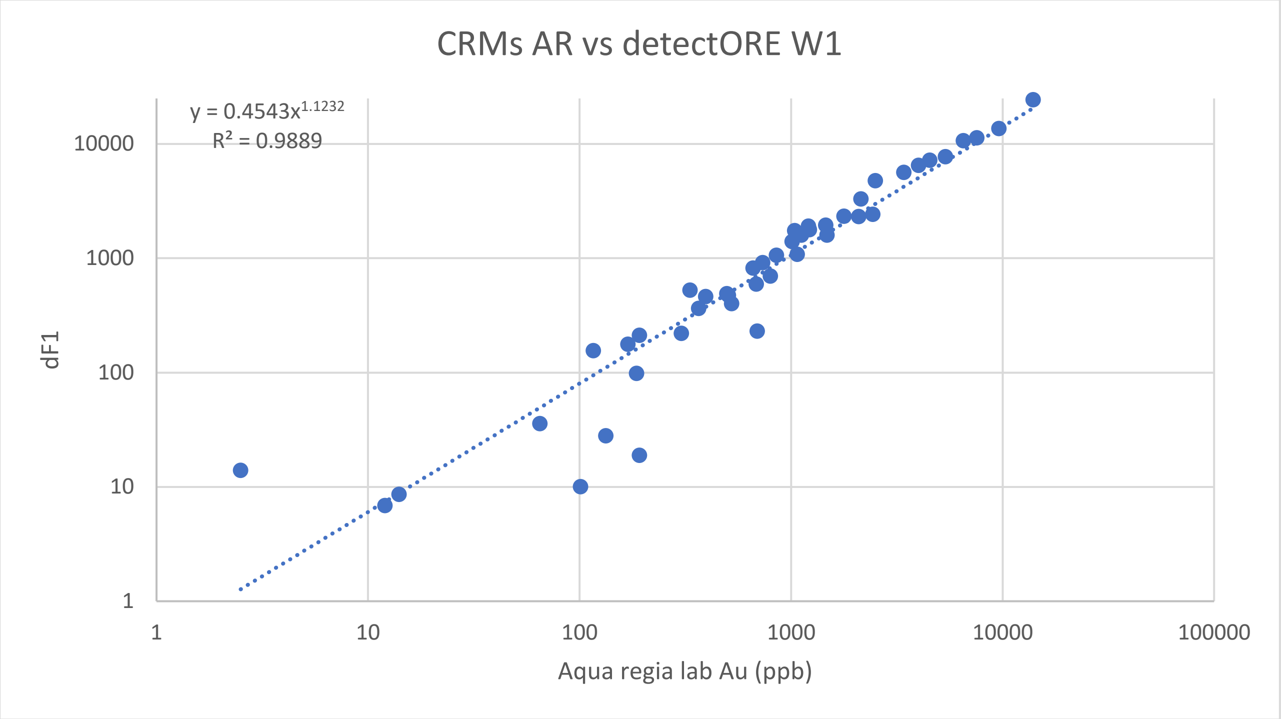


Figure 23: Scatter plot of OREAS Au standards analysed after fire assay versus detectORE™ as W1 dF1. Axes are logarithmic.

2.13 The pXRFs

Numerous pXRFs were used at PPPB. However, four Olympus Vanta M pXRfs were used for the majority of the test work and all field work. The Vanta machine outputs from laboratory standards were compared with each other and their stability was also tested at higher ambient temperatures. Several other machines from different companies were evaluated and their performance measured against the Vanta. Typically, the machines were borrowed over a few days and laboratory standards used to test the raw data output. The raw data output were analysed using our software. The machine identification has been kept confidential out of respect to the manufacturers who kindly supported our endeavours by lending them to us free of charge.

The figures that follow contain details of the test work undertaken in the captions and represent a selection of the experiments undertaken. A pXRF was run at ambient (warehouse) temperatures of around 20℃ and was then moved to a hot sunny position outside where temperatures were over 40℃ and the internal pXRF temperature reached nearly 70℃. The pXRF reading of a laboratory standard hardly changed during the entire 150 readings (Figure 24, four-hour period).

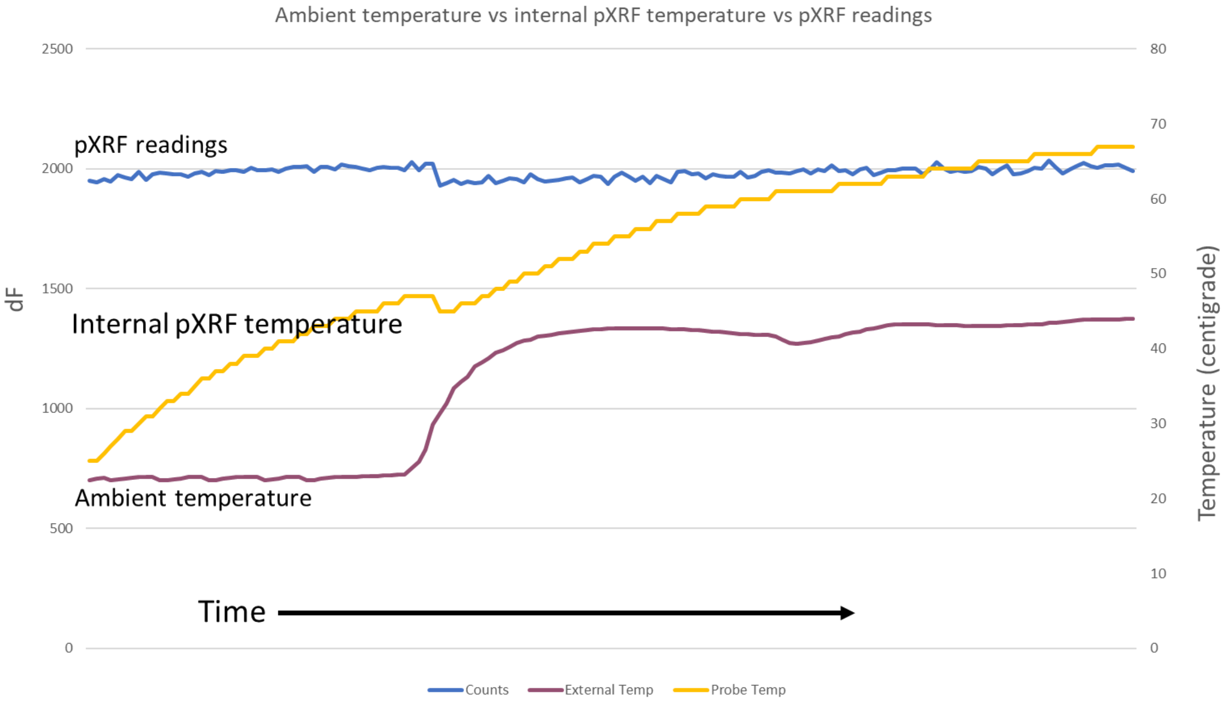


Figure 24: This chart shows what happens to pXRF data when outside temperatures are increased and sustained over several hours. Ambient temperatures were raised from 23ºC to 42ºC and then to 45ºC during the analysis of the same laboratory standard over a four hour period, no significant difference was noted between the 150 pXRF readings. The upper maximum temperature of the pXRF exceeded its upper rated temperature of 60ºC but still produced precise analyses in these harsh conditions.

The four Olympus Vantas were run for fifty readings each across the full range of laboratory standards (blank, 1-1000 ppb). Little difference was recorded between the mean readings for each concentration (Figure 25). The data suggests the Vanta M is fully interchangeable with one another allowing batches of samples to be run on different machines without applying a significant calibration correction for each instrument.

Several pXRFs were evaluated. The brands and manufacturers are not disclosed as part of agreements between PPPB and the manufacturers and/or providers. Characteristics important for detectORE™ are machine stability, sensitivity (limits of detection), ease of use, robustness and precision. As the loan of some of these instruments was for only a few days, some of the characteristics were not fully evaluated. Typically, repeat readings were undertaken with the laboratory standards and mean and standard deviation was recorded and charted for each concentration (Figure 26).

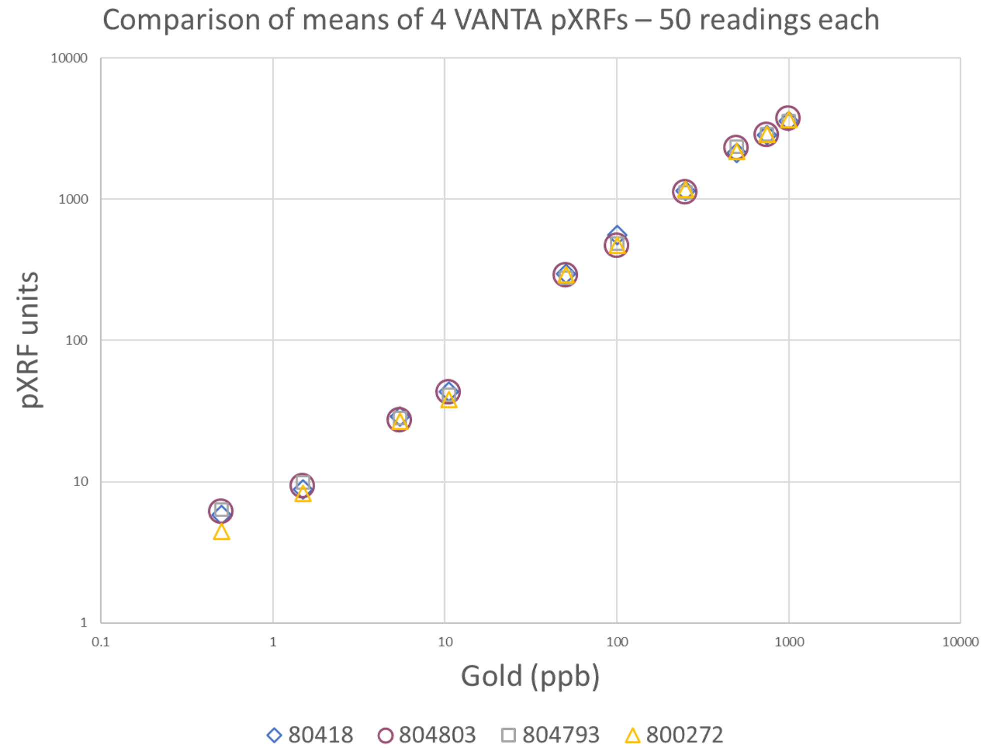


Figure 25: Four Olympus Vanta M machines (identified as 80418, 804803, 804793 and 800272) were tested using the same test conditions using a range of laboratory standards. Fifty replicate analyses for each Vanta at each concentration had no statistical difference. This suggested that our Vantas could be used interchangeably.

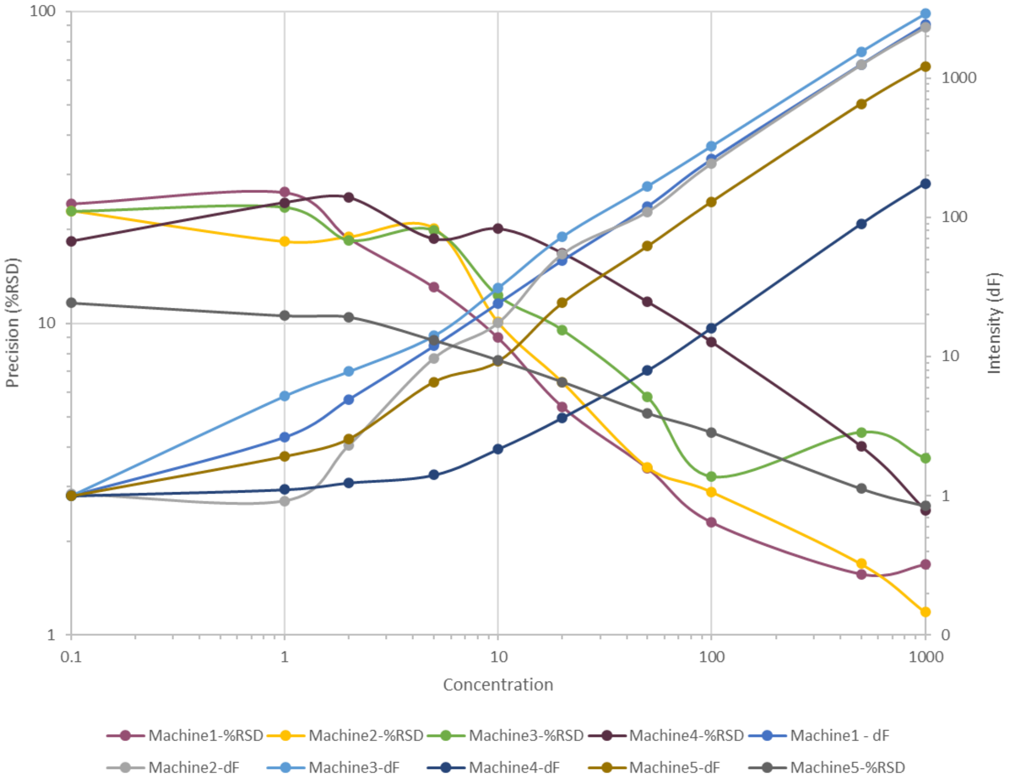


Figure 26: Comparison of five pXRF machines for precision (%RSD) vs concentration. Seven different pXRFs from 3 different manufacturers. Multiple tests (between 10 and 50 repeats were used at each concentration). Clearly Machine 4 has lower sensitivity and is not as useful at discriminating between concentrations. PPPB will recommend machines suitable for use.

PPPB originally had four Vantas and currently has three. Their performance was monitored twelve months apart using the same laboratory standards. The mylar film protecting the detector was replaced during this period however no change in the readings was noted between measurement dates (Figure 27).

To simulate contamination of the mylar detector window, a few sand grains (YS, ~20) were placed on the mylar film and 10 readings collected using a laboratory standard before and after the sand grains were added (Figure 28).

Laboratory standards were routinely analysed with each sample batch run on the pXRF. The dF (with means and standard deviations) was recorded for each pXRF (272, 793, 803, 818); the control charts for standard 3708 for the four instruments are shown in Figure 29.

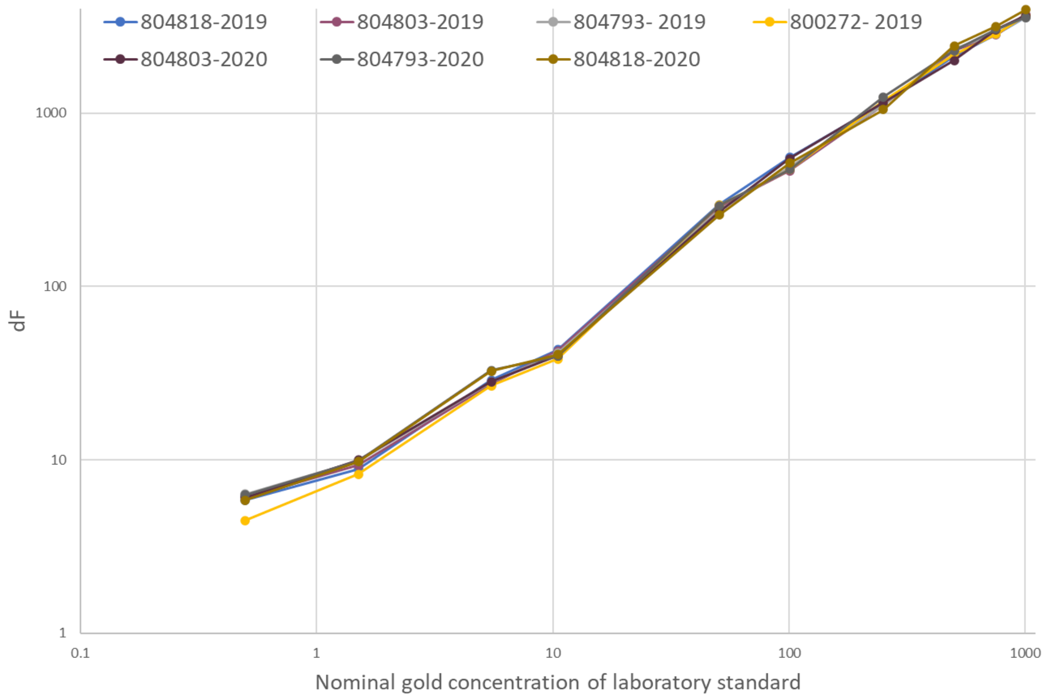


Figure 27: Instrument readings in February 2019 compared with February 2020. Three instruments (804818, 804803, 804793) have had a new mylar film installed on the pXRF. Instrument 800272 was loaned out at the beginning of 2020 before the testing was undertaken. Sensitivity has not changed over 12 months.

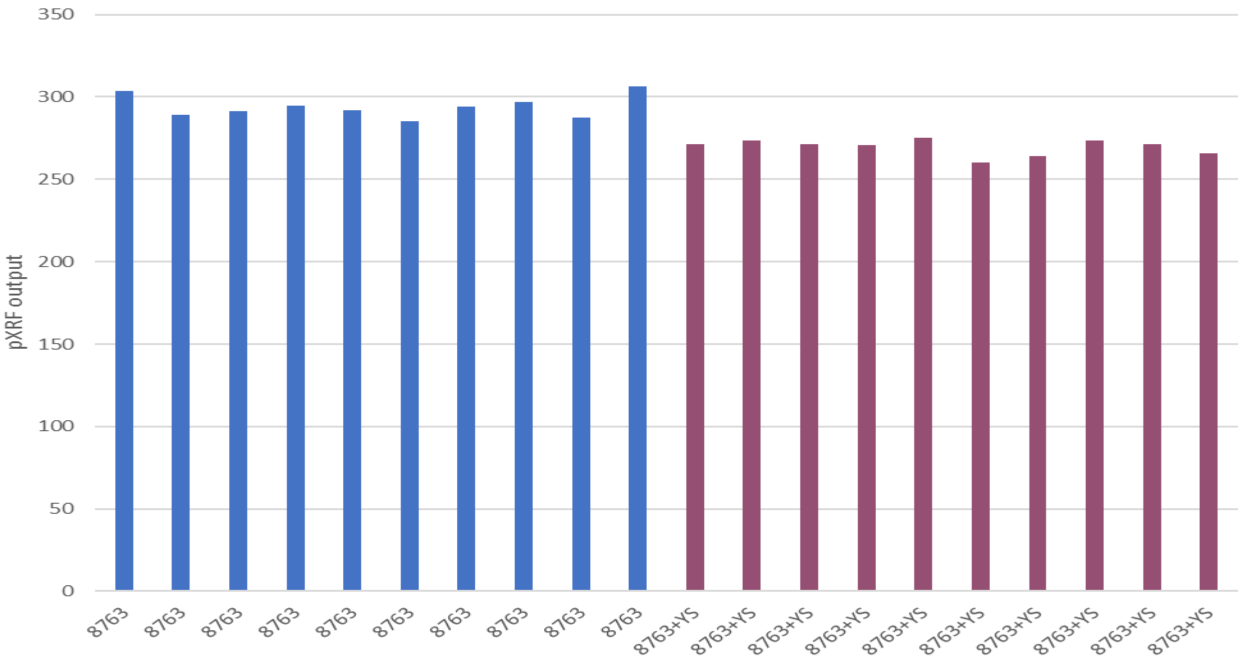


Figure 28: About 20 sand grains were placed on mylar film to see the effect of dust on the pXRF Au signal. A laboratory standard was used (50 ppb Au) to measure the effect. Material on the mylar film lowers the Au signal and users should be vigilant against dust and other material accumulation during analyses over a period. We recommend the use of a mini portable air gun to periodically clean the surface.

Figure 29: Control charts showing data for 1000 ppb Au laboratory standard (3708) for four different pXRF instruments (272, 793, 803 and 818) over a 12-month period. Blue line and grey dashed lines show mean and standard deviations (1 and 2 SD), respectively. These charts incorporate the new film for the last few readings for 793, 803 and 818.

2.14 Physical processing test work

The physical testing of the detectORE™ Method involved mixing parameters, containers, washing of CDs and is ongoing (as part of research within PPPB) to maximise sensitivity and precision; also important is simplicity, robustness and cost-effectiveness. These experiments were performed over the period of the project; early experimental data were generally less precise than recent experimental data.

Towards the end of the project, PPPB acquired a new research mixer that enabled speed, rotational direction, and tilting to be performed. Mixing of sample is critical to the detectORE™ Method and may be affected by several factors that are not well understood. The approach taken with this project was to test a parameter and record data, then make changes to the parameter and compare the data.

The captions in Figures 30 - 34 and Table 1 that follow, contain details of the test work undertaken.

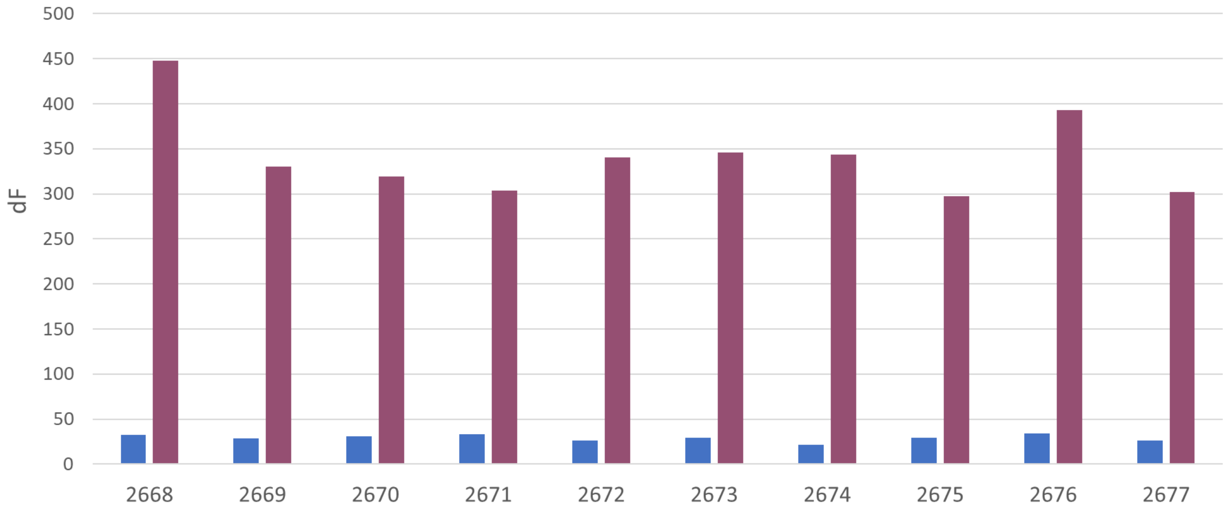


Figure 30: An early project experiment examined the effect of mixing. Ideally, if mixing could be avoided altogether then we could avoid using a mixer and lower power consumption and transport volume. In this experiment, ten CRM replicates in separate containers were left static for a 24-hour period (blue columns) and the CDs removed and measured. Another CD was put in and the sample mixed for a further 24 hours (maroon columns). Clearly, mixing increases the sensitivity of the method as an order of magnitude more Au accumulates on the CD.

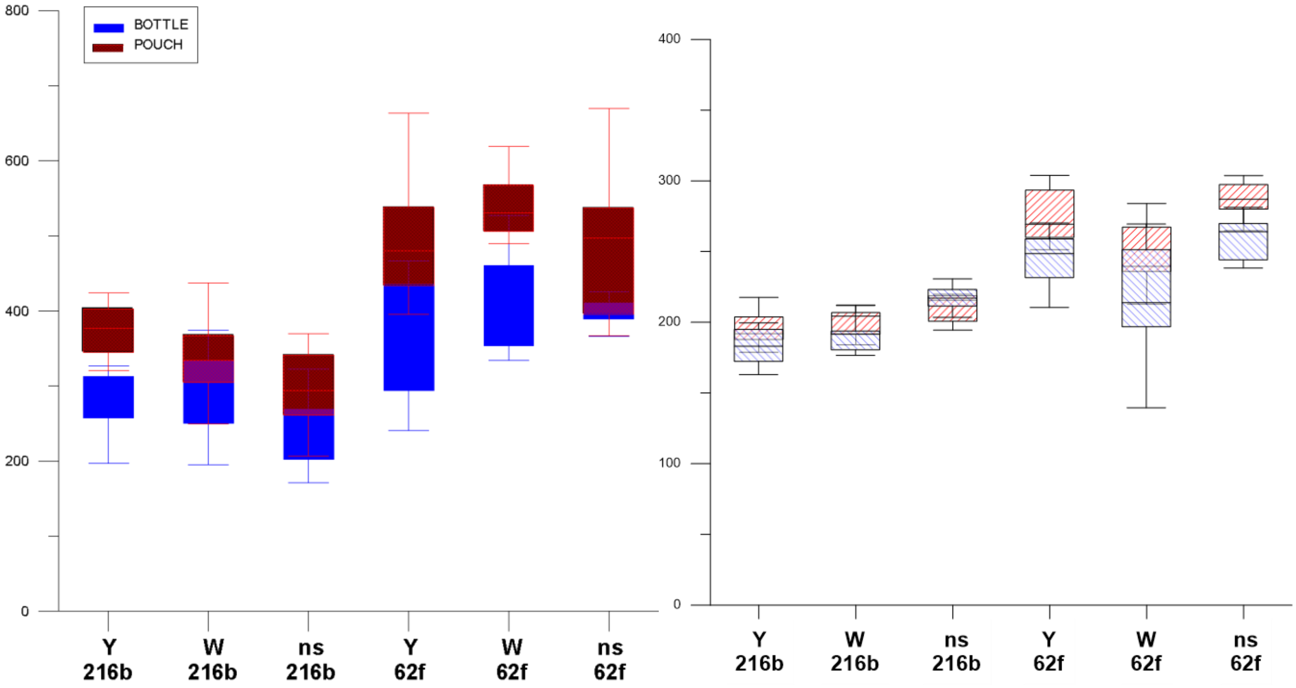


Figure 31: Comparison between early project results (LEFT) and later project results (RIGHT) for testing of containers: pouches versus bottles. The development of new mixers, containers and mixing process has led to improvements in precision.

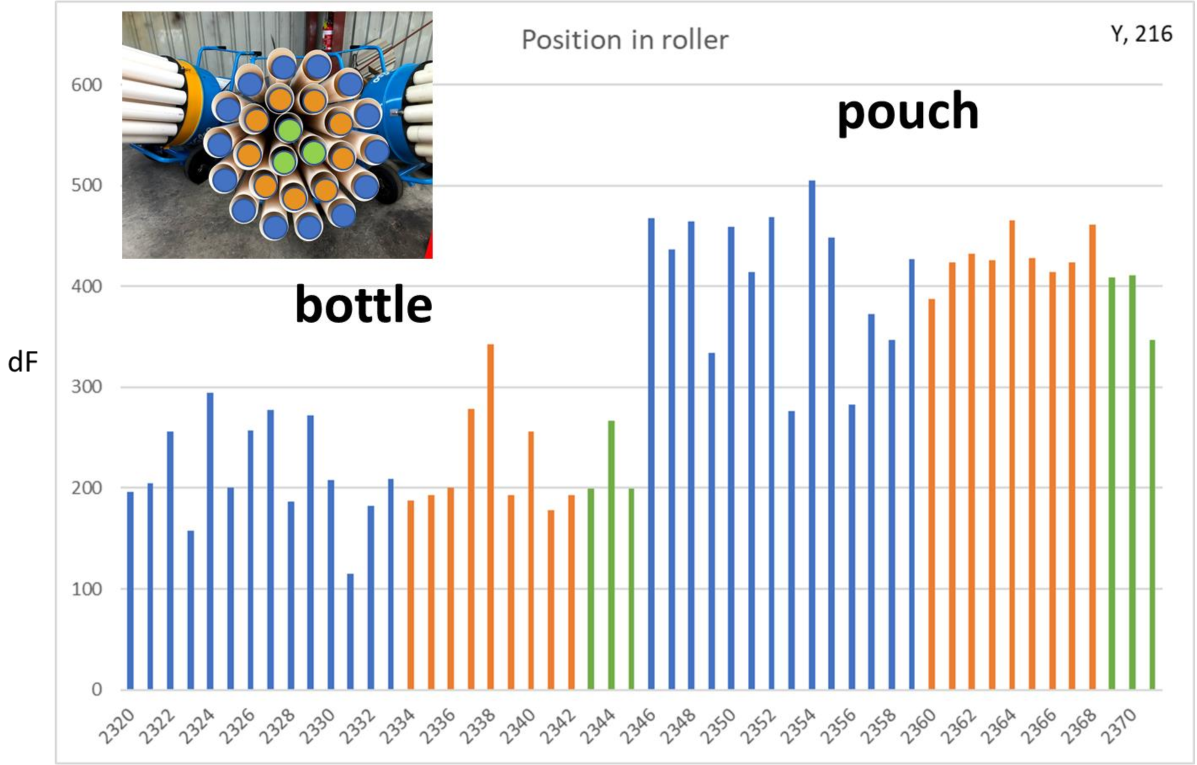


Figure 32: A test was performed to determine whether there were any differences with placement position in the mixer. Bottles and pouches were used with CRM216b diluted with yellow sand. There was no difference found between position. However, surprisingly, pouches had greater Au adsorbed when compared with bottles. Pouches probably had more turbulent flow compared with more laminar flow in the bottles.

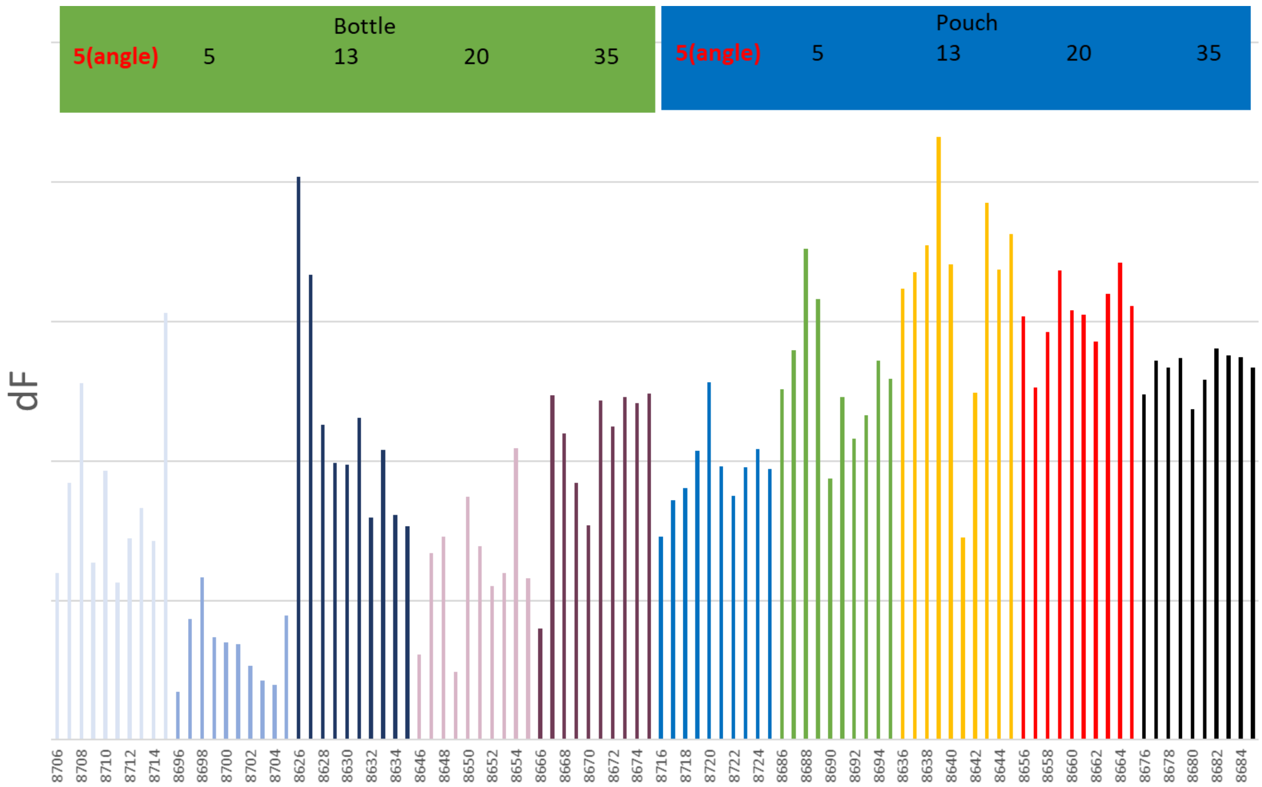
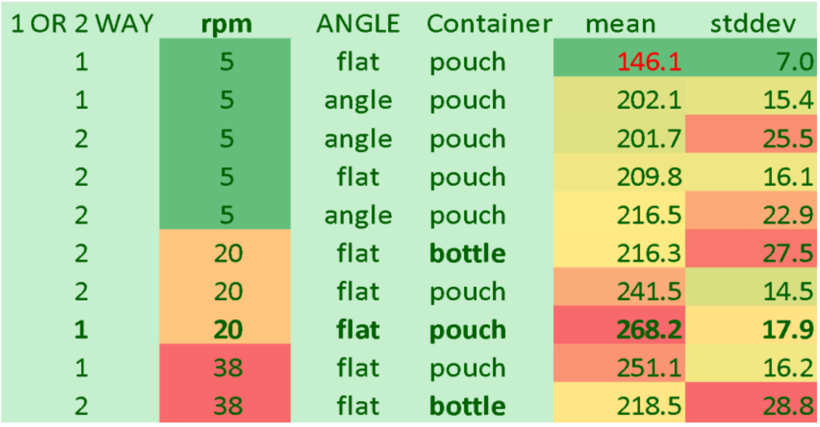


Figure 33: In these experiments the new research flatbed mixer was used. RPMs shown at the top of the figure for pouch (blue) and bottle (green). Angle refers to an angled mixer similar to the original mixer design. Unsurprisingly, increased RPM produced more precise data and/or accumulated more Au on the CD. The angled mixer produced some surprising results, bottles tested produced higher readings (compared with the flat mixer), whilst with pouches little difference was noted. Higher RPM were generally more precise and/or accumulated the most Au (greater sensitivity) on the CD.

Table 1: Summary of means and standard deviations from the new mixer tests. The new mixer arranged flat with a pouch and rotating one way produced the greatest mean and therefore the highest sensitivity of the parameters tested. Bottle containers generally produce lower precision than the pouches.



1 OR 2 WAY	**rpm**	ANGLE	Container	mean	stddev
1	5	flat	pouch	146.1	7.0
1	5	angle	pouch	202.1	15.4
2	5	angle	pouch	201.7	25.5
2	5	flat	pouch	209.8	16.1
2	5	angle	pouch	216.5	22.9
2	20	flat	**bottle**	216.3	27.5
2	20	flat	pouch	241.5	14.5
1	**20**	**flat**	**pouch**	**268.2**	**17.9**
1	38	flat	pouch	251.1	16.2
2	38	flat	**bottle**	218.5	28.8

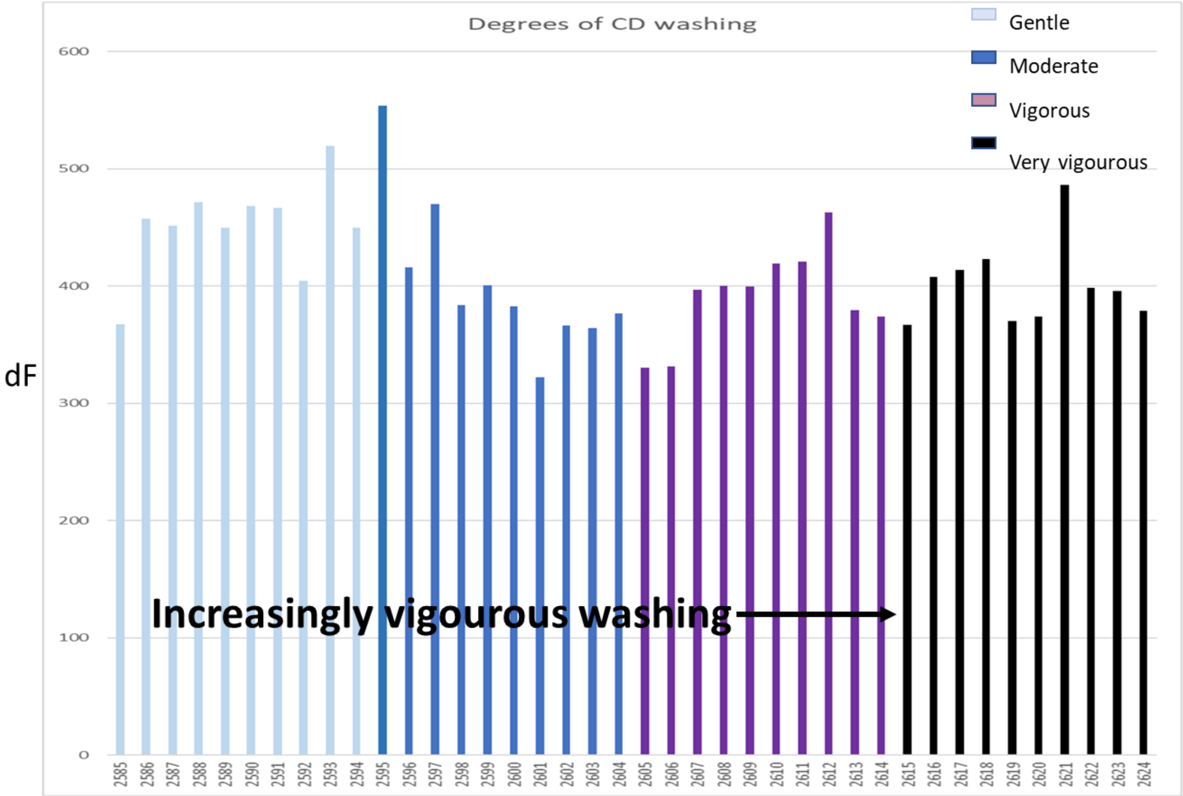


Figure 34: After the mixing/digestion, the CD requires washing to remove extraneous geological sample material. To determine whether Au was being physically removed from this washing process, CDs containing Au were subjected to various degrees of washing – 10 replicates of each. Very vigorous washing involved a powerful jet of water sprayed onto the CD. A small difference was noted between gentle and other washing procedures.

2.15 Reagent chemistry used in digest of samples

Different reagents were tested during the project. Experiments designed to investigate reagent dissolution characteristics were performed in combination with a specific CD i.e., reagent efficacy was not independent of the CD. Reagent and CD recipes and how they are applied are not disclosed. Desirable reagent characteristics are to:

1) dissolve the metal of interest e.g., Au in a reasonable time frame,
2) allow dissolved metal to be adsorbed by the CD,
3) not dissolve chemical species that interfere with ‘target’ analysis,
4) be safe to use and transport,
5) have low toxicity for the environment,
6) be relatively inexpensive (compared to other methods), and
7) have a reasonable ‘shelf life’ effectiveness.

Experiments showed that technical grade quality chemicals could be used to substitute for analytical grade, reducing costs.

Several reagents were tested but no single reagent positively addressed all the above criteria. The most important criteria were those relating to safe-use and being environmentally friendly, to conform with PPPB values. These reagents were not necessarily the best at dissolving Au but nevertheless were considered to be at acceptable levels.

The rate of dissolution of Au varied for samples and for CRMs with the latter having faster dissolving Au suggesting that it is more amenable to dissolution by the reagents. The experimental dilemma for testing reagents was therefore about selecting ‘reference’ material that was homogenous in Au (no nugget effect and reproducible in assay), available in large quantities but would have dissolution characteristics similar to ‘real samples’. Diluted versions of our ‘workhorse’ CRMs (62f and 216b) were first used to perform the bulk of the reagent test work. Later, reagents were then tested on different CRMs and then on samples where comparative data were available from other methods.

The three most effective reagent-CD combinations tested were designated W1, W2a and W2c. The CRMs using W1, W2a and W2c are shown in Figures 20 - 23, Figures 35 - 36, and Figures 37 - 38, respectively. Correlation coefficients (r^2) are greater than 0.9 for W1 and W2c. A few CRMs were in poor agreement between laboratory and detectORE™ reagents and were due to pXRF interfering peaks and/or samples containing refractory Au.

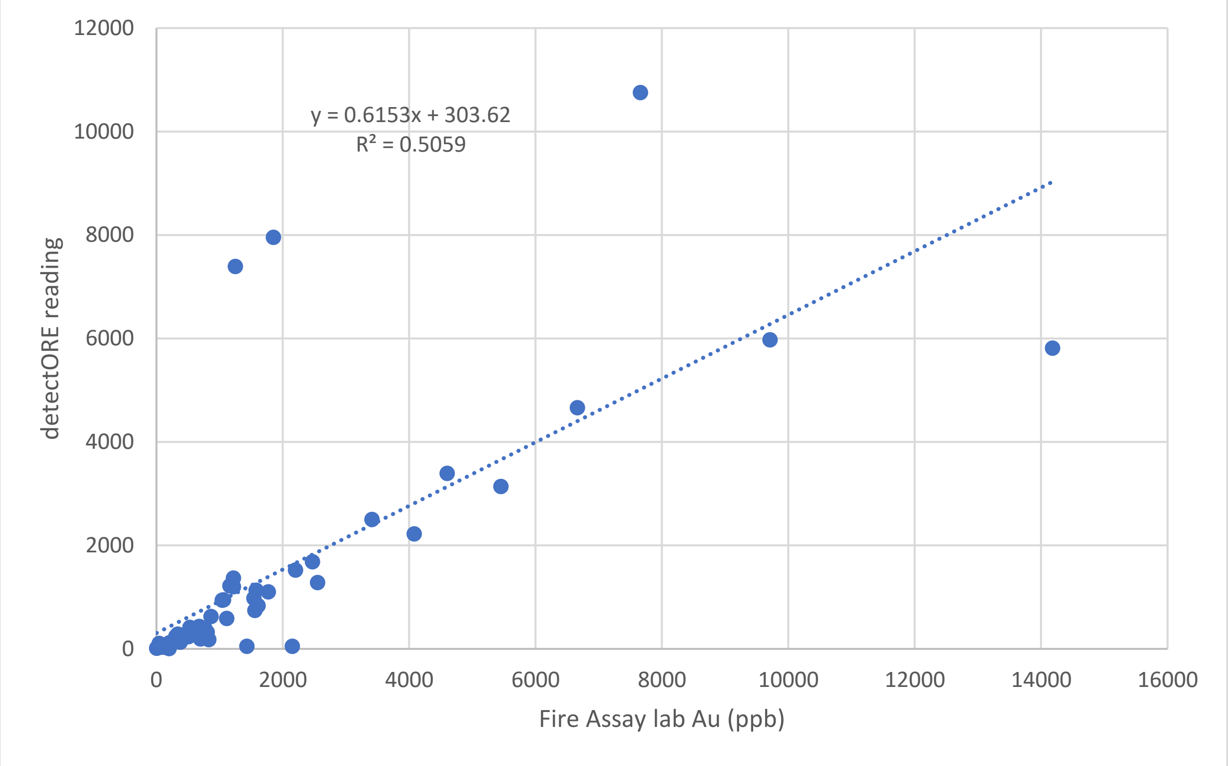


Figure 35: Scatter plot of OREAS Au standards analysed after fire assay versus detectORE™ as W2a dF1.

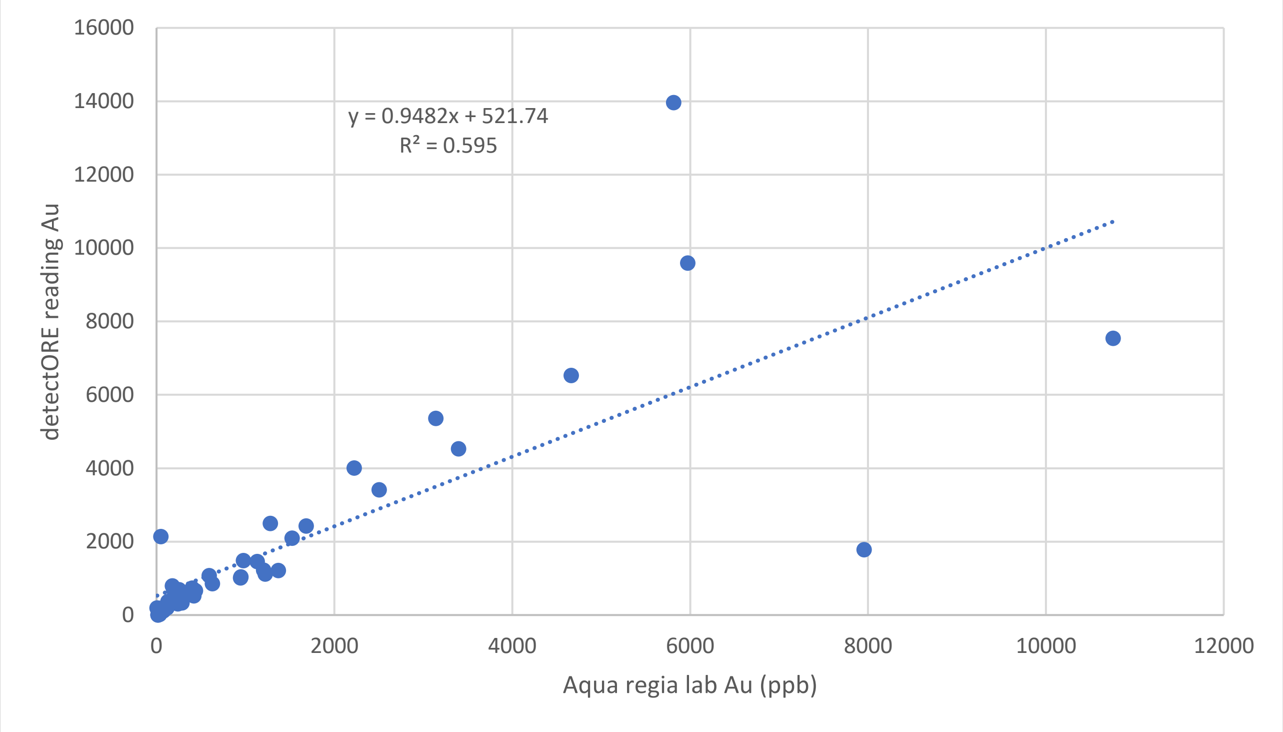


Figure 36: Scatter plot of OREAS Au standards analysed after aqua regia versus detectORE™ as W2a dF1.

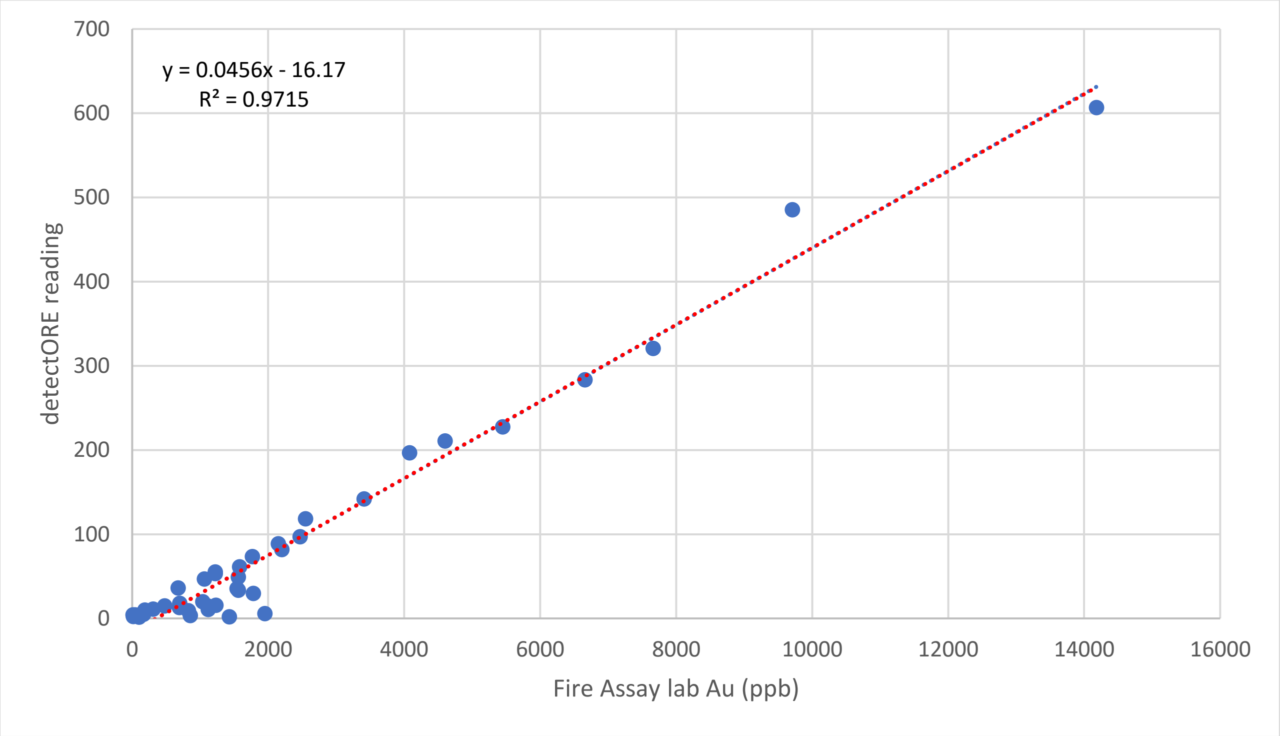


Figure 37: Scatter plot of OREAS Au standards analysed after fire assay versus detectORE™ as W2c dF1.

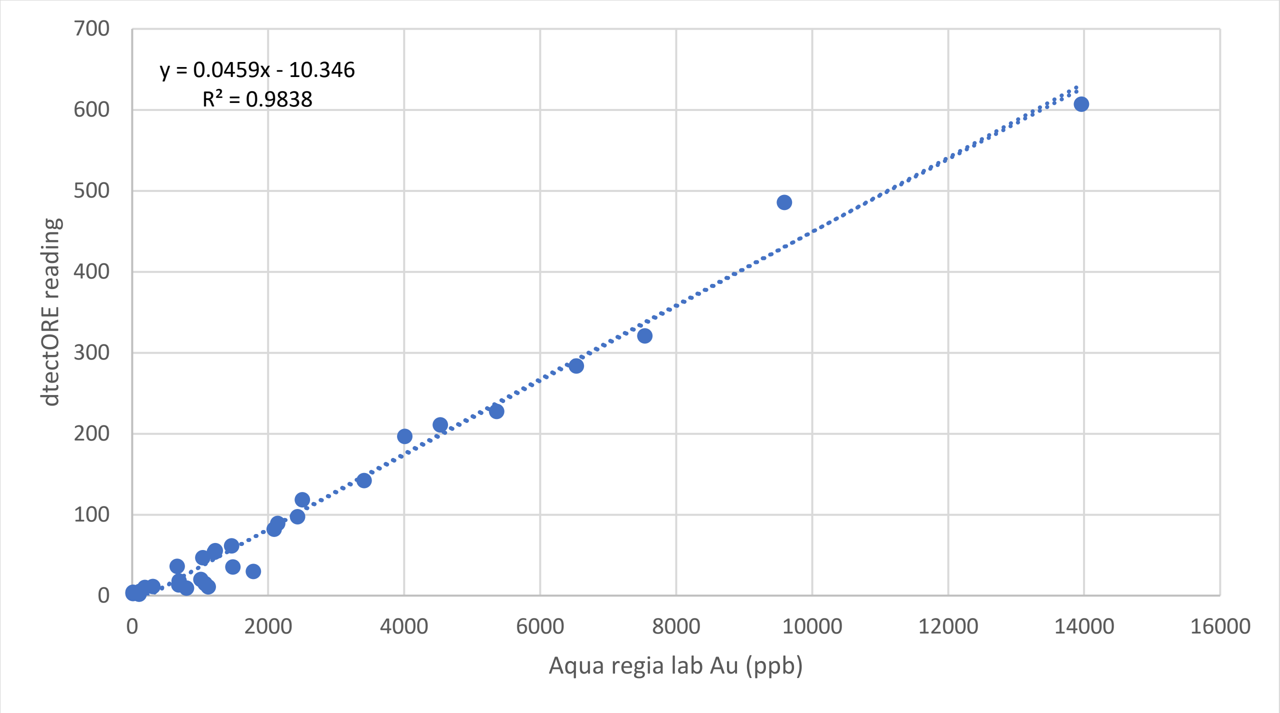


Figure 38: Scatter plot of OREAS Au standards analysed after aqua regia versus detectORE™ as W2c dF1.

Interfering peaks for pXRF from other elements are few in type and occurrence. Spectral software is limited in its ability to deconvolute or remove grossly interfering peaks particularly when the elements, or element combinations, are in high concentrations relative to the amount of analyte; interfering peaks can be identified using the detectORE™ software and thus provide a 'red flag' to the user that there may be an analyte determination issue or that a higher detection limit needs to be applied. Thus, samples containing low levels of Au and high levels of interfering elements are the most challenging to analyse using detectORE™. There are fewer interferences using W1 (Figure 39) compared with W2a (Figure 40) and W2c.

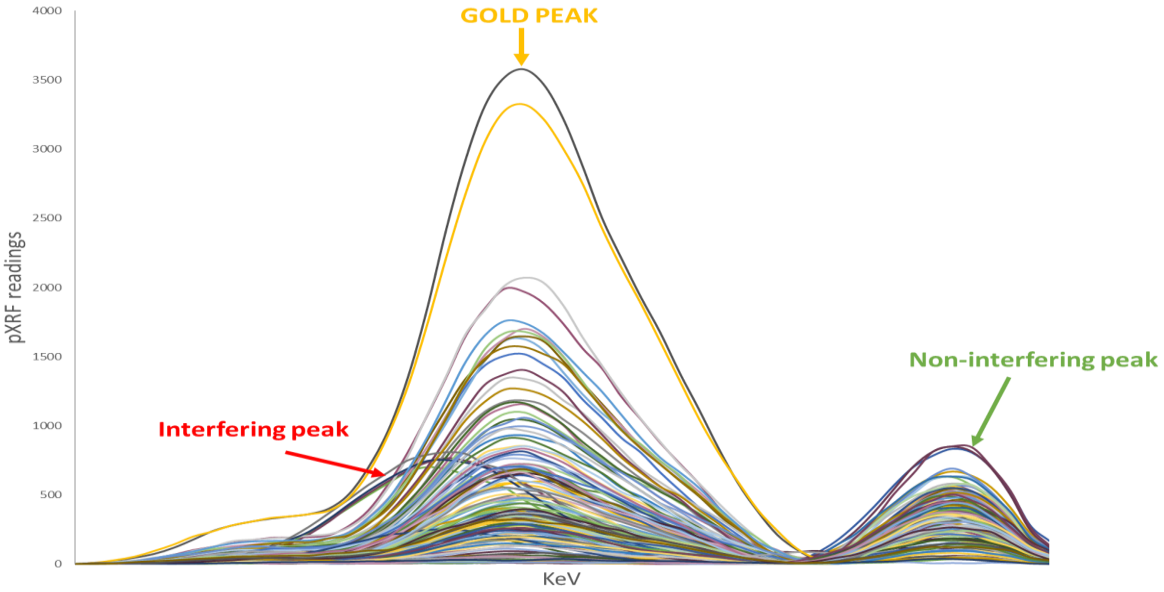


Figure 39: Overlaid spectra for CRMs using W1. Interfering peaks will affect the quantification of Au with low level Au concentrations and high concentrations of interfering elements the most difficult for a determination.

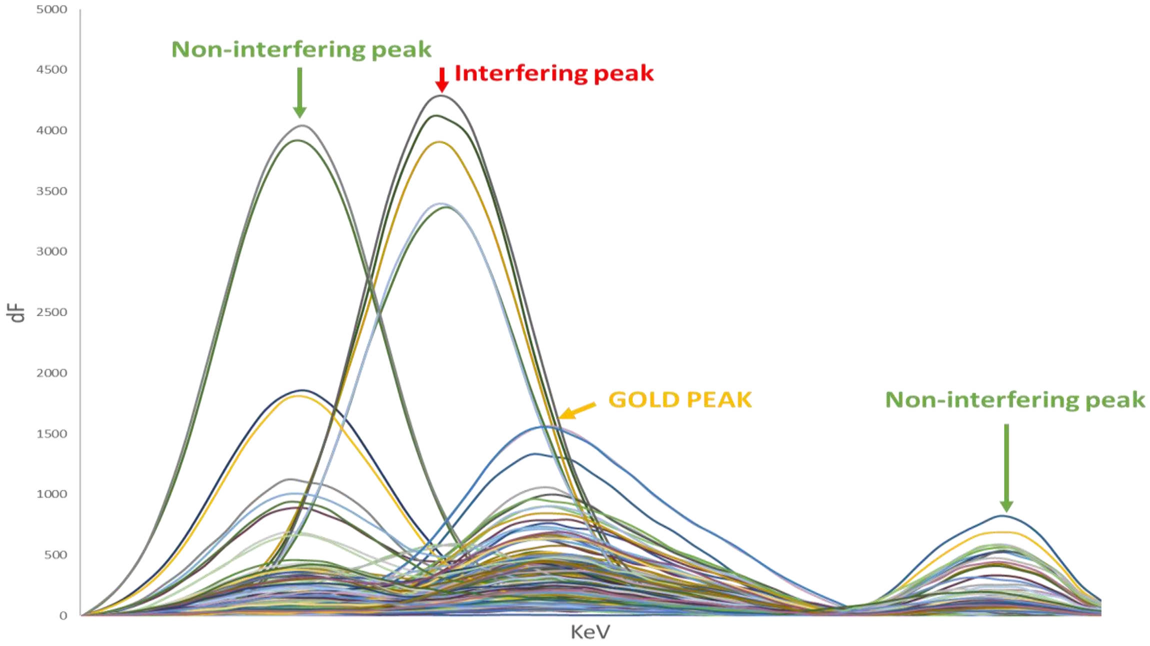
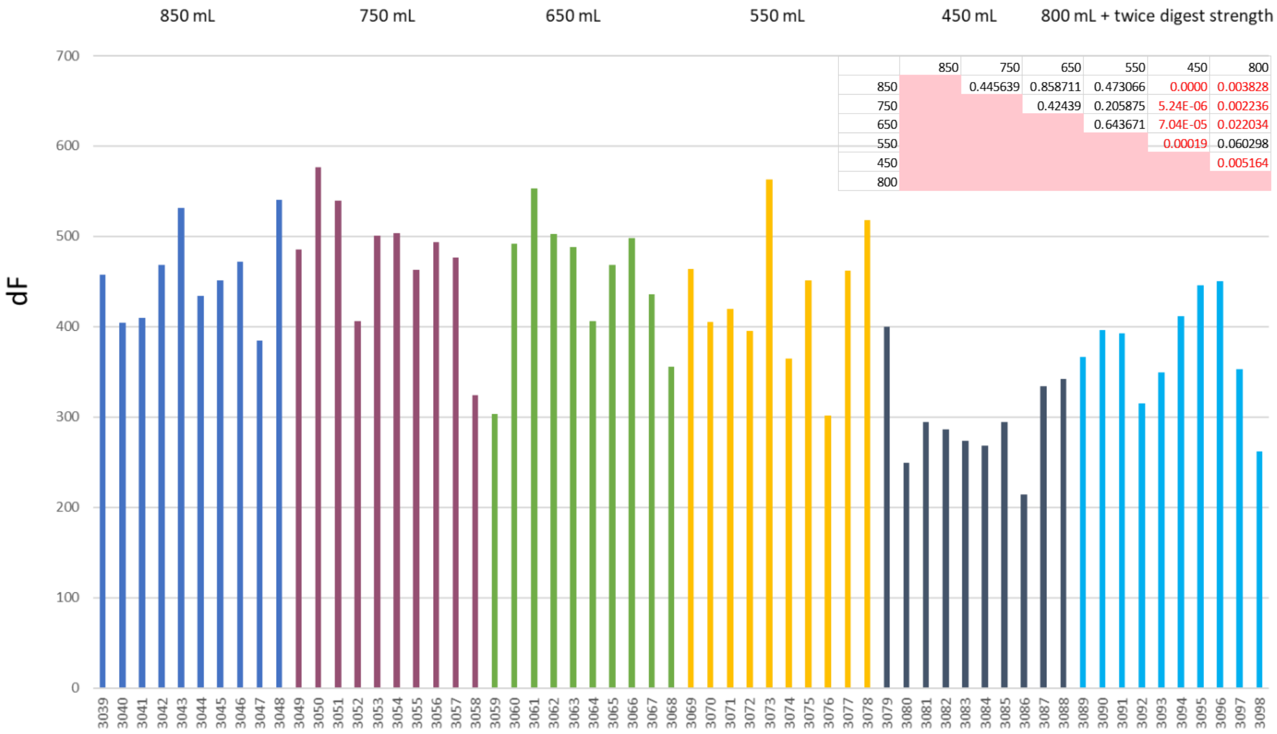


Figure 40: Overlaid spectra for CRMs using W2a. Interfering peaks will affect the analysis of Au with low level Au concentrations and high concentrations of interfering elements the most difficult for a determination.

The volume of reagent was also tested. Volume of reagent affects several factors including how viscous the digest is, how dilute the extracted Au dissolved the reagent (low volumes will be more concentrated), the ability of the CD to adsorb Au, the type of sample (to some degree) and the way the sample is mixed which is, in turn, affected by the size and shape of the container. The aim of the experimental work is to maximise precision and sensitivity. Examples of the experiments are shown in Figure 41 and Figure 42.



	850	750	650	550	450	800
850		0.445639	0.858711	0.473066	0.0000	0.003828
750			0.42439	0.205875	5.24E-06	0.002236
650				0.643671	7.04E-05	0.022034
550					0.00019	0.060298
450						0.005164
800						

Figure 41: Different volumes of W1 reagent were used in 1 L bottles containing a CRM (216b) diluted with yellow sand. Ten replicates and coloured coded for each set. A low volume of reagent measures less Au by pXRF. In the last set, twice the digest strength was used with a significantly reduced effect on Au measurement. The inset table shows the student-t statistic comparing each set; those in red are statistically significant.

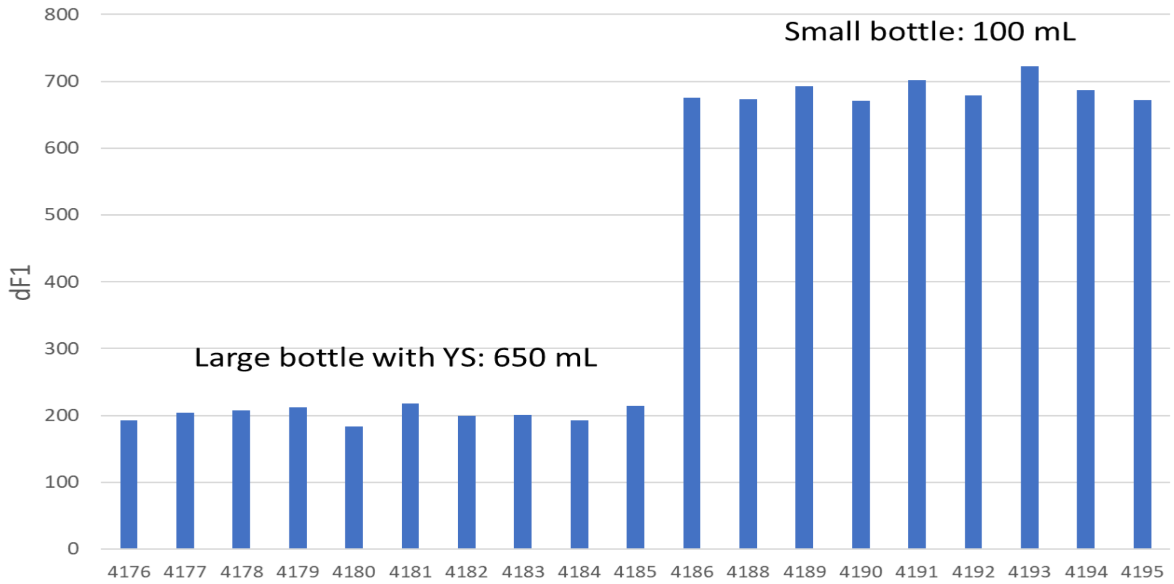


Figure 42: Using the same amount of Au (25 g of CRM62f) but in different containers (1 L and 125 mL) and reagent volumes (650 mL and 100 mL) led to different measured Au on the CD. The small bottle CD result indicates three times as much Au as the large bottle. Ten replicates of each and YS refers to yellow sand used as a 10:1 diluent with the CRM.

The effect of temperature (including extremes) was tested on the dry W2c reagent. The detectORE™ Process will be used in all continents so consideration was given to testing the effect of prolonged exposure (70 days) through method efficacy as measured by the pXRF. Four temperature conditions were tested on the dry reagent designated as sea box (~10 to 60ºC), freezer (-15ºC), warehouse (15-35ºC) and fresh. No statistical difference was recorded between the different temperatures (Figure 43).

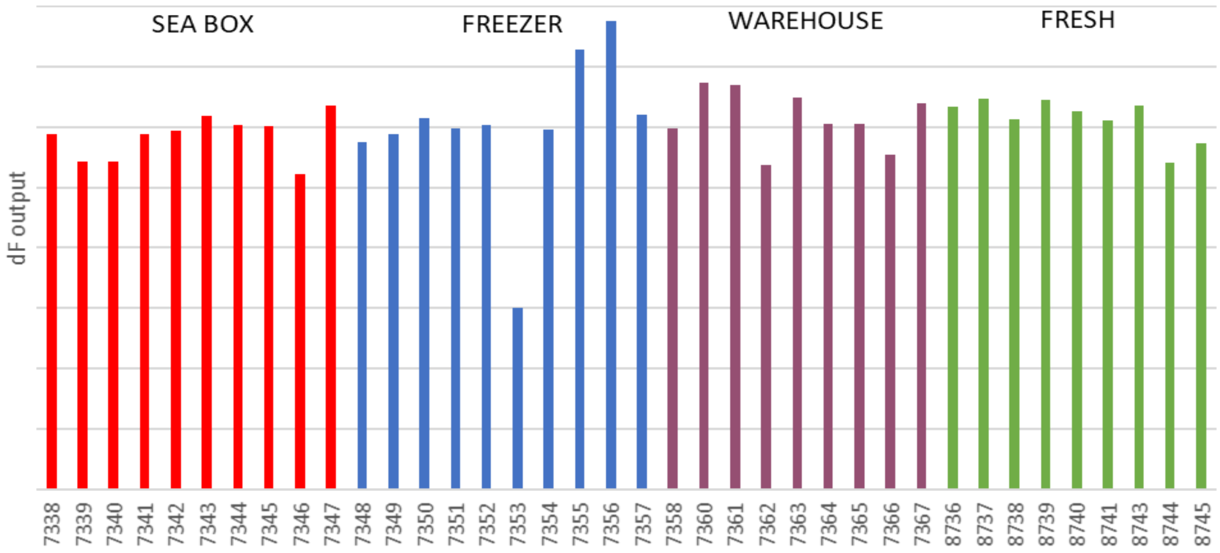


Figure 43: Reagent dry chemicals stored in bottles under various temperature conditions from 4/2/2020 to 15/4/2020. Sea Box (sea container) would have experienced variable temperature ranges (~10-60 degrees). Freezer was at -15 degrees. Warehouse at ambient (10-40 degrees). Ten replicates of reagent chemicals were stored in the different temperature conditions and then each combined with CRM and CD and processed to examine the efficacy of the 'aged' chemicals.

In a similar experiment, ‘aged’ W2c reagent solution from six batches (batch 6 to 11, kept aside over several months) was tested; this experiment was conducted towards the end of January 2020. Differences between batches (except Batch 9) were not significant (Figure 44).

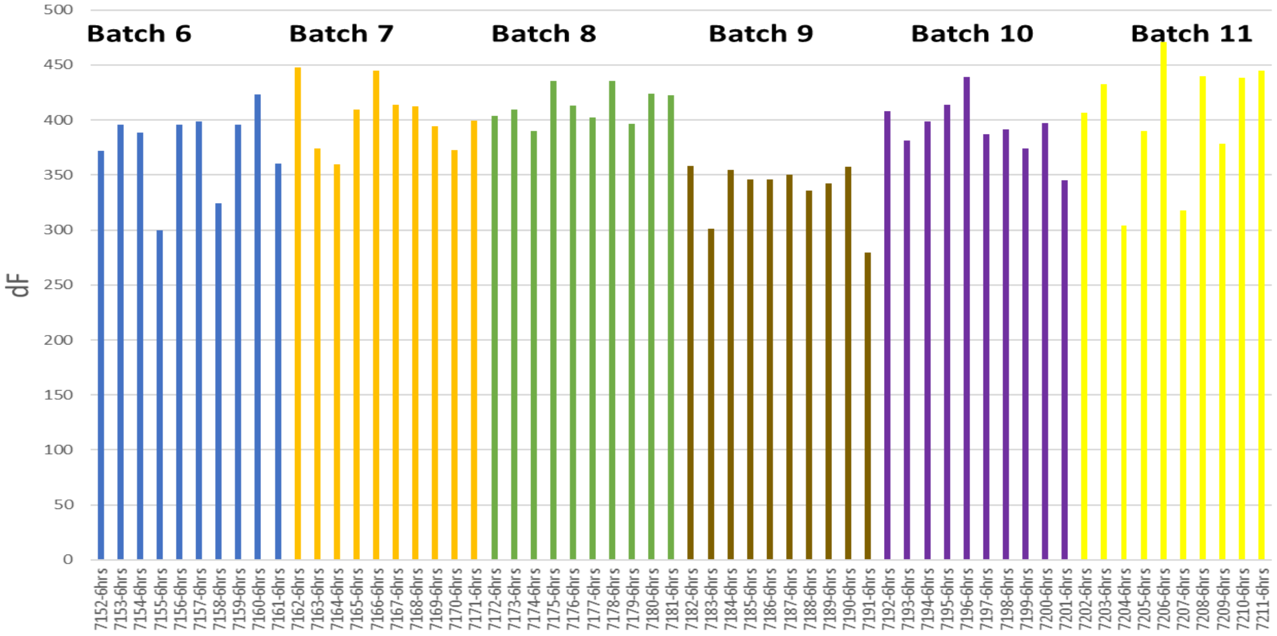


Figure 44: Aged liquid reagents (designated with batch numbers 6-11 and batch date labelled in table inset) were tested on its efficacy for the detectORE™ method towards the end of January 2020. Ten replicates of 100 mL of reagent mixed with dilute CRM 62f and using a 125 mL bottle were tested and showed little difference between batches indicating that the ‘made-up’ reagent is effective over several months in ambient conditions.

The timing of field receival and subsequent processing of samples is frequently unpredictable particularly for reactive sampling™. Samples may need processing overnight in order for results to be delivered the following dawn prior to setting the next day field sampling plan. To simulate the effect of ad hoc processing on detectORE™ 50 samples were digested and after 6 hours (and every subsequent 6 hours) 10 CDs were removed up to 30 hours after the experiment commenced. Thus, the 30 hour samples had been mixed for 6 hours but then left static in reagent for a further 24 hours. Little difference was found between these samples (Figure 45).

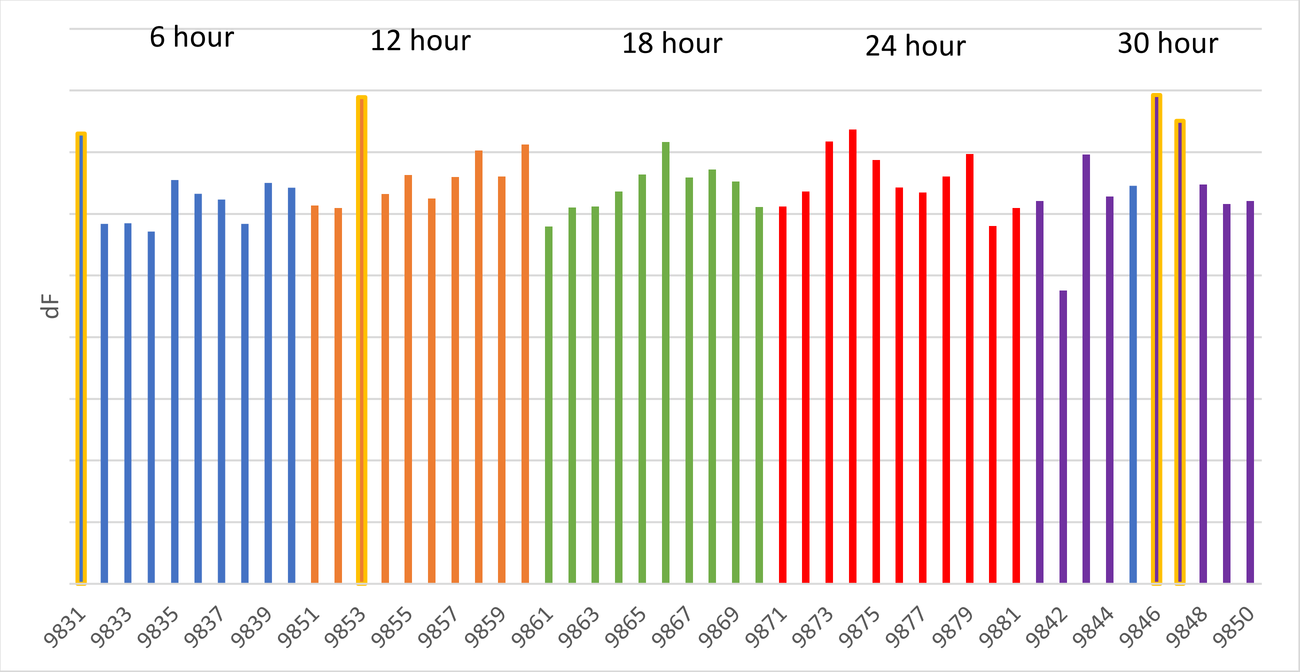


Figure 45: These 6-hour digests (n~50, using diluted CRM216b) had their CDs left in their pouch with reagent and removed after set periods of time (12, 18, 24 and 30 hours). This implies that CDs can be extracted at a later period (eg digested in the evening and processed in the morning) without affecting the results. 4 samples with yellow bars had interferences.

2.16 Collector devices (CDs)

A variety of CDs were tested with different reagents. Collector device "A" may work well with reagent "A", whilst CD "B" may work poorly with reagent B, even though the latter may be excellent at dissolving Au quickly. Thus, the combinations of many CDs with many reagents led to a multiplicity of experiments. A selection of these experiments is shown below.

The pH was adjusted in one reagent and while it worked well with CD A1, for other CDs (A2, A4 and A5) it was not as good. For CD A3, there was an intermediate performance which was optimal at pH10.

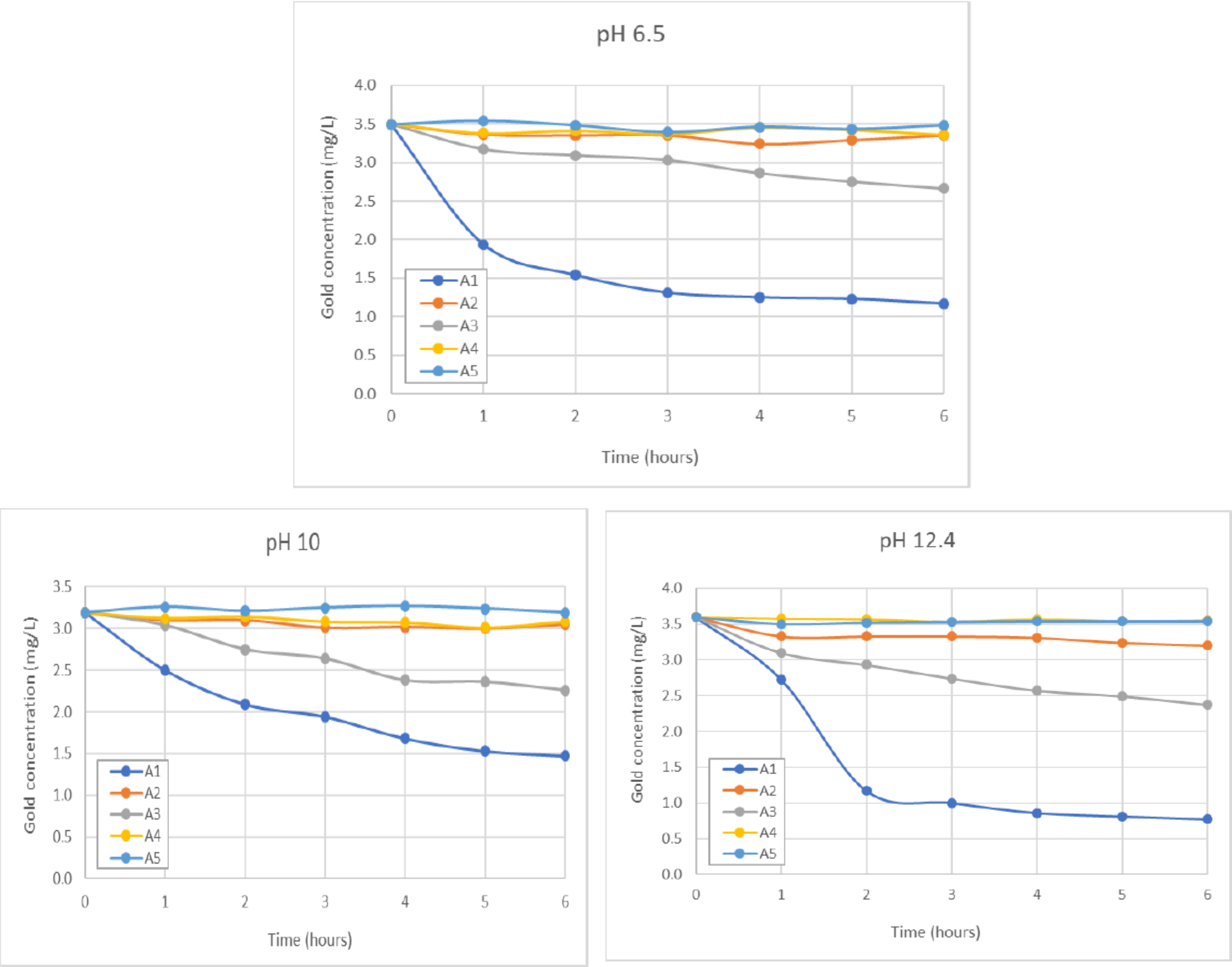


Figure 46: Gold adsorption kinetics for different CDs (A1-A5) with different pH conditions (pH 6.5, 10 and 12.4) for one of the lixiviants (reagents). CD A1 was the best performing as the Au concentration was the lowest in solution after 6 hours (CSIRO).

The effect of storing a CD (A) in an oven over several weeks to simulate acute environmental temperatures was examined. CD A was dried in an oven at 60℃ for up to 4 weeks and the adsorption of Au performance was measured and compared with CD B (Figure 47).

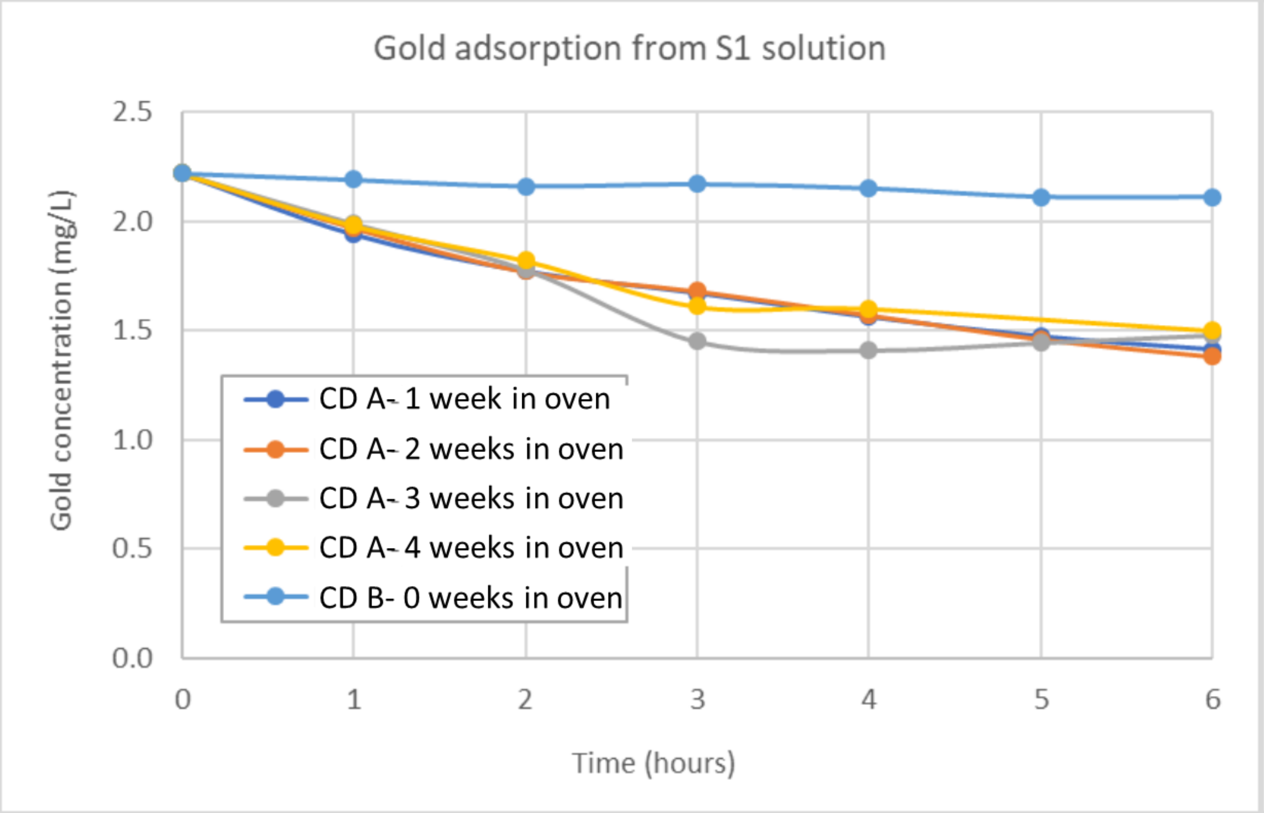


Figure 47: There was no difference in performance by heating CD A for up to 4 weeks compared with 1 week. CD B was poor at adsorbing Au in comparison. Gold concentrations measured in solution reduced with time (up to 6 hours) (CSIRO).

CD W2 was put under extreme temperature conditions (e.g. freezing to 60°C) for 7 days and Au adsorption was measured using pXRF. Noticeably, heating reduced the performance of W2 (measured as dF) compared with freezing (Figure 48).

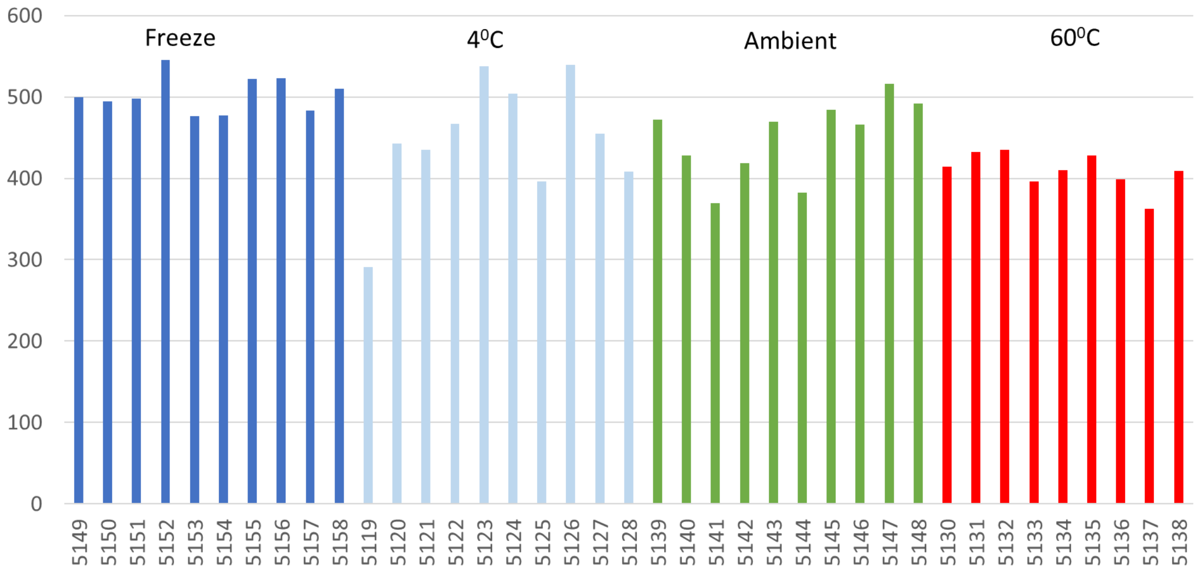


Figure 48: Effect of temperature extremes on CD Au absorbance performance (measured as dF). Ten CD replicates were left for 7 days and Au measured (on the CDs) by pXRF.

A solution containing 3.5 ppm Au was mixed with a CD and different concentrations of reagent. The stronger the reagent concentration the lower the adsorption of Au on the CD (Figure 49). However, stronger reagent concentrations lead to greater leaching of Au from the ore (more and faster). Thus, it is balance between leaching and adsorption that is important for reagent strength.

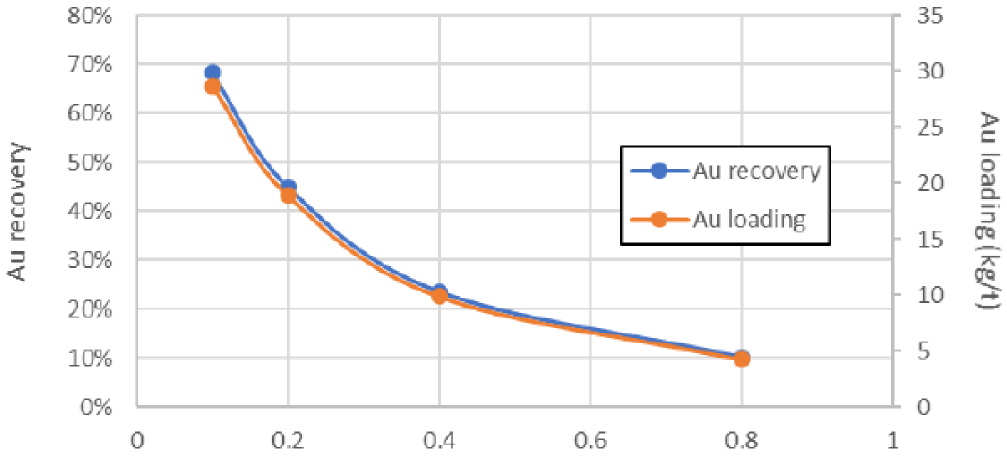


Figure 49: Gold adsorption equilibrium for the CD as a function of reagent concentration (0-1 nominal units). The higher the reagent concentration e.g., 0.8 the lower the Au loading (10%) on the CD. However, other experiments indicate that higher reagent concentrations leach more Au. (CSIRO).

During mixing the CD may become partially coated by the digest slurry and must be physically cleaned; we found no difference between CDs (with adsorbed Au) that had been gently rinsed compared with high pressure washing (Figure 34).

The stability of the CD Au readings over 12 months was described earlier (Figure 29). Measurements were taken over replicate readings and where CDs were removed from the pXRF detector window and then returned and re-analysed. Typical repeat readings of CDs across a range of concentrations return a correlation coefficient r^2 >0.95 indicating excellent agreement. However, good placement of the CD on the pXRF detector window is important (Figure 50). PPPB investigated several holders for the pXRF platform to ensure consistency of CD placement from sample to sample. The same CD in different holders can generate quite different results (Figure 51).

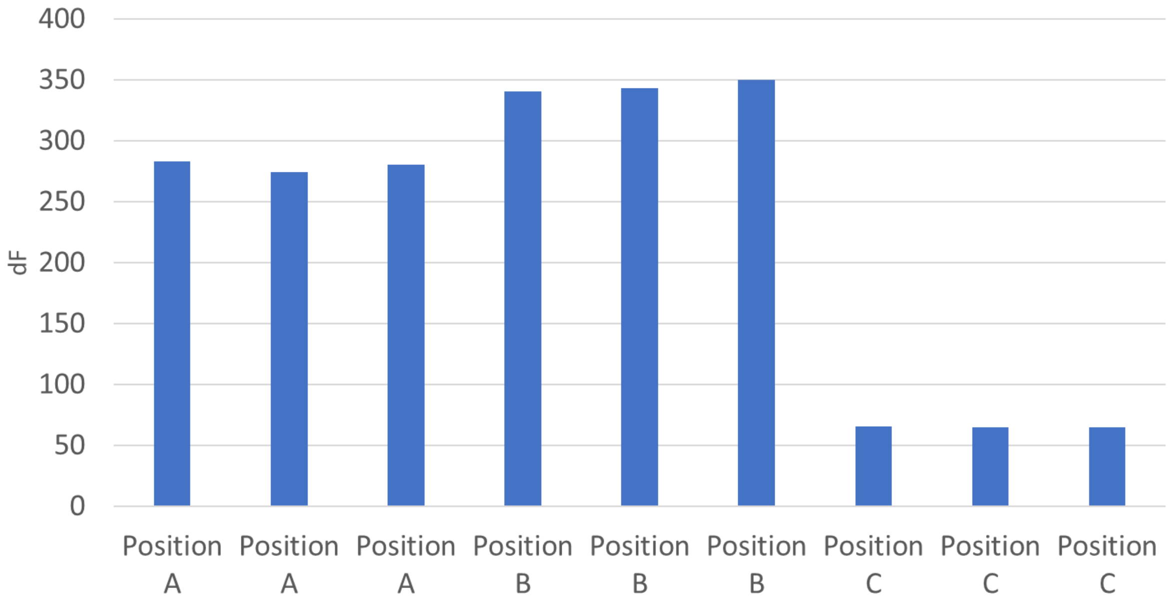


Figure 50: Three positions of the CD (A, B and C) over the window indicates that it must completely cover the pXRF window for reproducible and optimal data. Triplicate data shown.

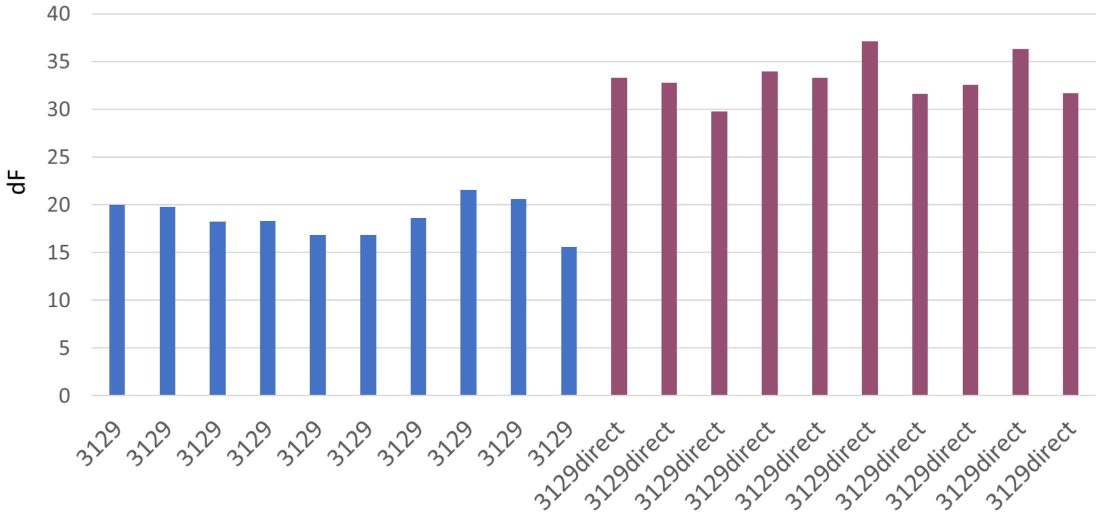


Figure 51: Comparison of two types of CD housings demonstrating the importance of correct presentation of the CD to the pXRF. Here, 10 replicate readings of a 5 ppb laboratory standard are compared side by side.

Laboratory standards were analysed as dry, wet or very wet on the pXRF. When CDs are removed from the sample containers (pouches or bottles) they contain adsorbed sample material which needs to be washed off. The CDs are wet after this process and are normally dried before placing on the pXRF for analysis. The results of this experiment indicate that moisture on widget 1 CD does not affect the Au readings at higher concentrations (Figure 52). At lower concentrations, there is some effect of moisture that will create higher detection limits.

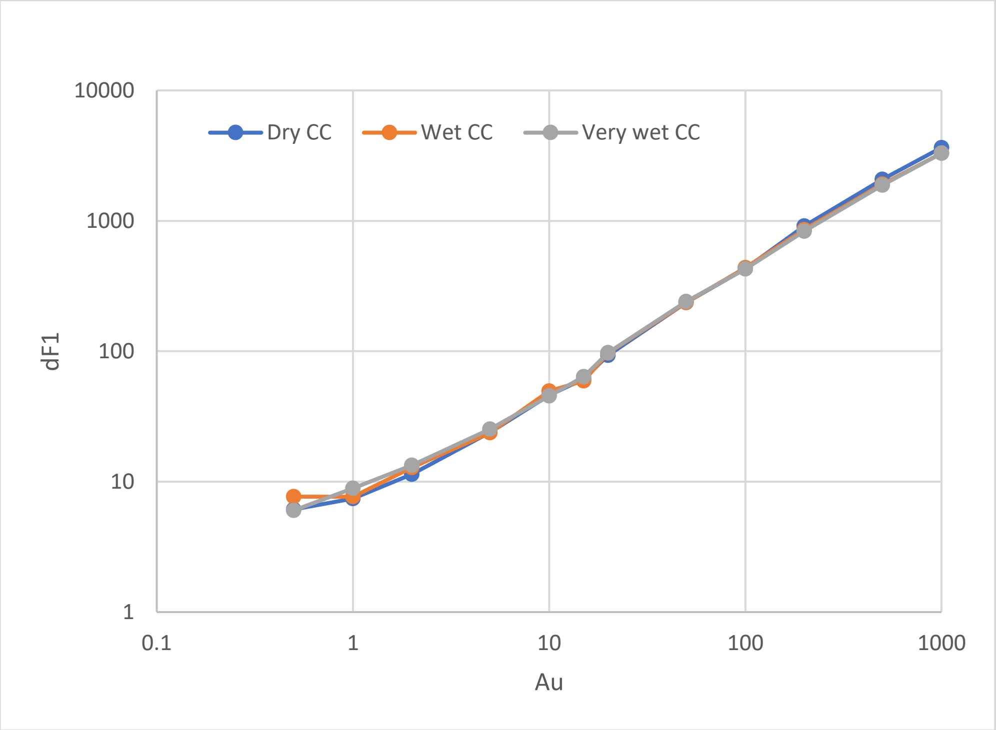
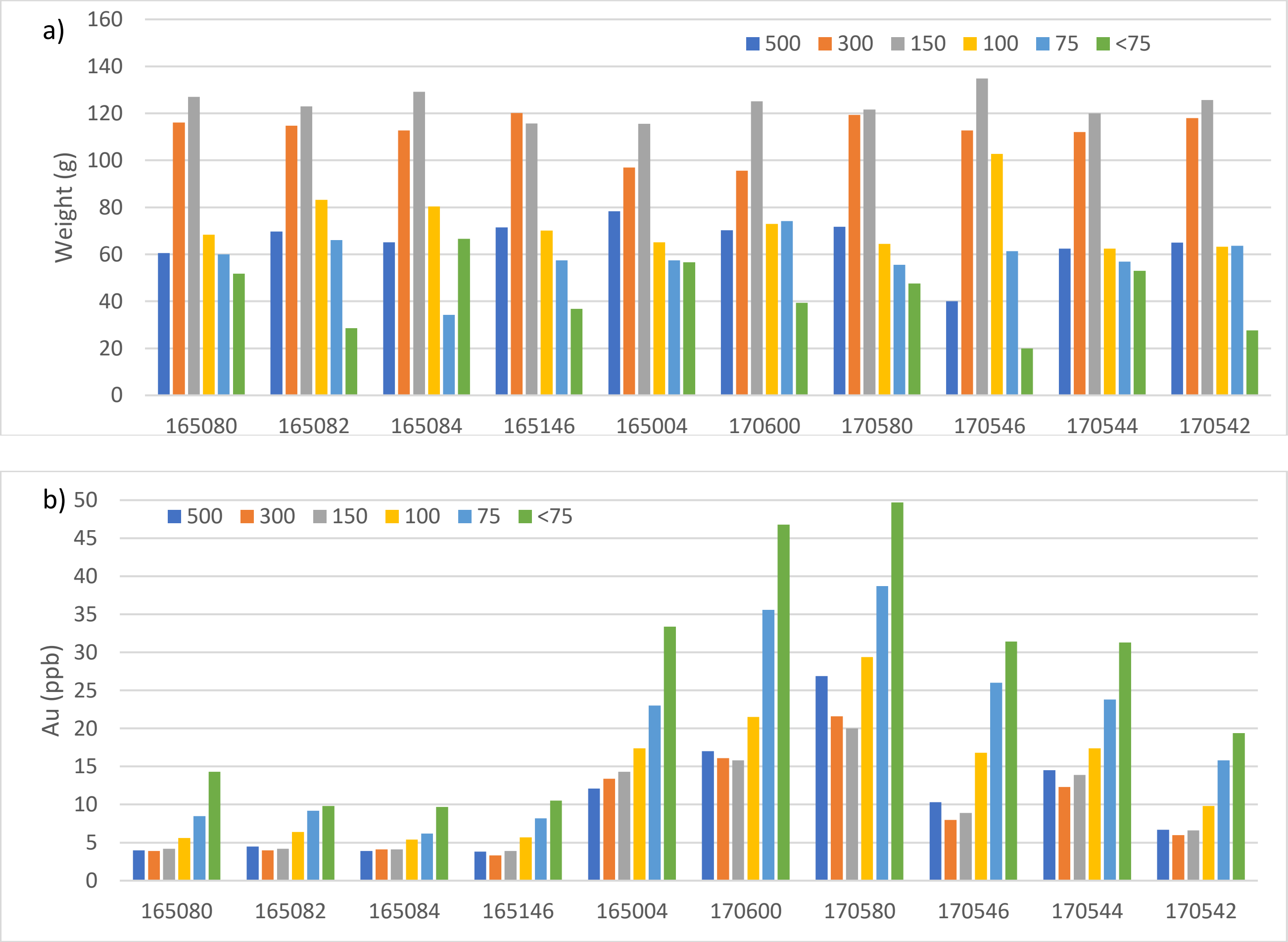


Figure 52: Effect of moisture on the CD readings (widget 1). Whether the CD is wet or dry there is little difference in the readings of Au (df1) at higher concentrations. At very lower concentrations there may be some effect and so, for consistency, CDs are recommended to be dried before reading.

2.17 Sample preparation

For detectORE™ Au is measured using a much larger sample size (e.g. 250 g) than techniques such as aqua regia and fire assay. The weight used in detectORE™ is like a bulk leach extractable Au (BLEG) analysis, in which in-sample variability is likely to be less than aqua regia or fire assays using smaller weights, but not eliminated. However, the detectORE™ sample is not pulverised. PPPB's field practical sample preparation technique involves sieving to remove coarse particles that are inaccessible to the reagent for leaching Au. Ultimately, it will be the choice of the client on how samples are prepared and may be dependent on local environmental conditions; for example, after rain, sieving through a very fine sieve is problematic as particles become trapped within the mesh material and are difficult to remove. With very wet samples it is necessary to analyse the sample 'as is' or 'raw'. PPPB has undertaken case studies to demonstrate potential outcomes of the field-practical sample preparation procedure. The working hypothesis is that a 250 g sieved sample can identify anomalies in the field as effectively as laboratory procedures that will pulverise, sub sample and analyse a smaller 25 g of material. We are cognisant that remote field camps have few sample preparation facilities such as sample pulverisers. Pulverisers also introduce the potential for cross contamination of samples caused by smearing of gold particles on the pulveriser which are difficult to remove even with 'quartz rinsing' between samples.

Ten samples of 500 g of <1 mm calcrete were sieved through a series of sieves (mesh sizes 500 µm, 300 µm, 150 µm, 100 µm, and 75 µm) and the weights recorded and analysed (Figure 53). Individual size fractions were analysed by a mini BLEG (25 g, without pre-pulverising) at a commercial laboratory. The Au distribution was not unexpected and indicated that the finer fractions have higher concentrations of Au but that coarser fractions yielded the most mass of sample. The data provides an indication as to how much and type of calcrete field sample may need to be collected and sieved with enough Au content for detectORE™ to measure.



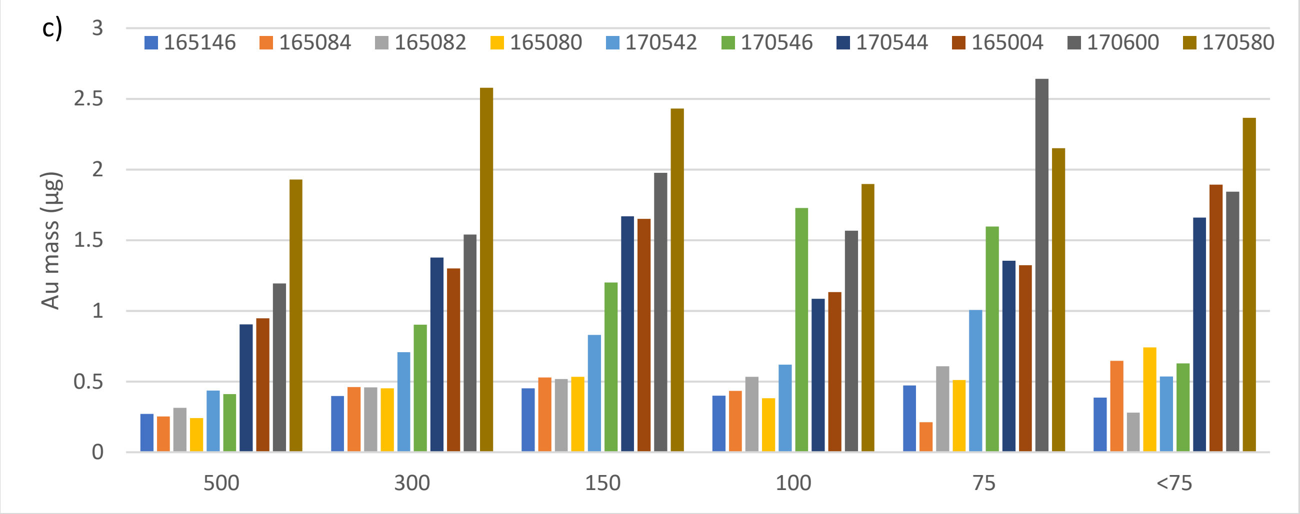


Figure 53: Ten calcrete samples were sieved into six size fractions and analysed for Au by BLEG without further processing: a) sieving indicated a bias of material towards the coarse size fractions; b) Au concentrations indicate a strong and systematic skewness towards the fine size fractions; c) calculations of the mass of Au in each size fraction indicate that despite samples containing predominantly coarse material, most of the Au (by mass) is found in the fine fractions.

In another evaluation of the sieving method, five kilos each of six RC (reverse circulation) drill cutting samples were sieved using the same mesh sizes as above. All samples contained the most mass in the 300-150 µm range (Figure 54); subsequent experiments indicate that extra time on the sieve shaker may have yielded more finer material. Each size fraction was analysed without further pulverising using detectORE™, with results suggesting that fine fractions usually contain the most Au, although very coarse material may have high Au concentrations (Figure 55).

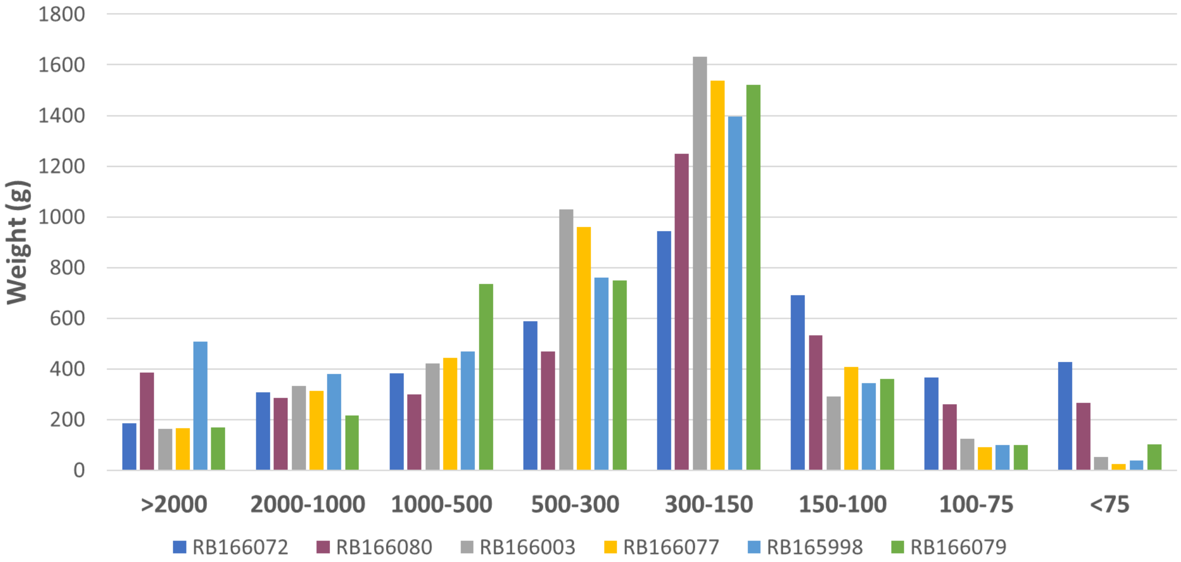


Figure 54: Five kg of six samples were sieved through a multi-stack sieve shaker yielding results that indicate that the 300-150 µm size fraction is dominant.

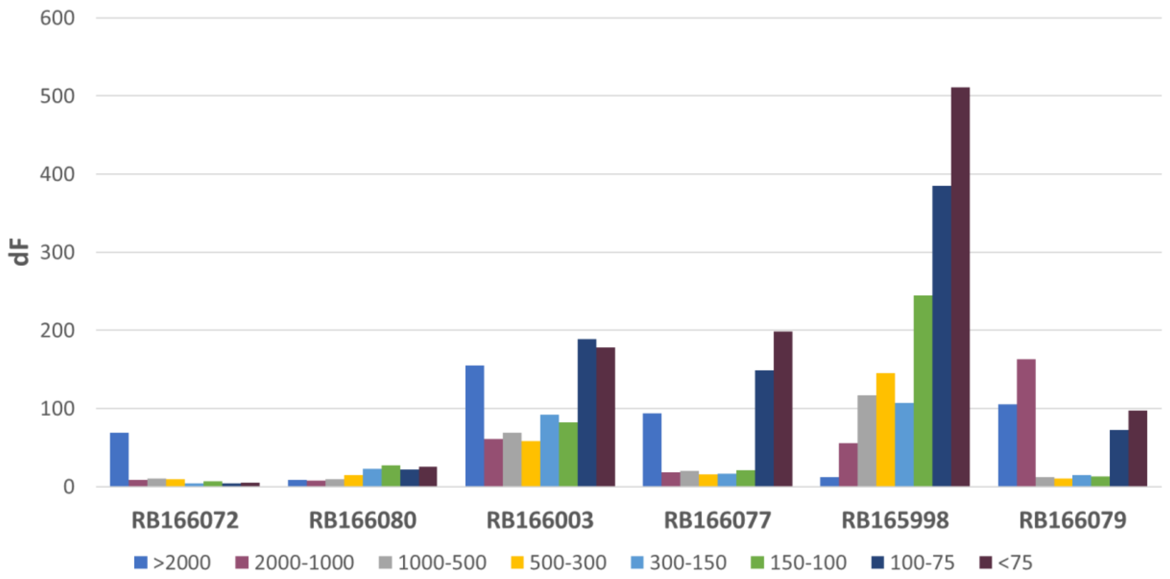


Figure 55: The fine fraction usually contained the highest Au concentrations of the eight size fractions analysed.

2.18 Laboratory based case studies using sponsor supplied exploration materials

A comparison of 130 NE Yilgarn soils from 7 traverses (2 prospects) between laboratory analyses and detectORE™ indicate good agreement over 3 of the eastern traverses (r^2=0.88; B, D and F), and reasonable agreement (r^2=0.46, outliers removed) over the remaining 4 western traverses (A, C, E and G). Samples from the western part of the traverses contain possible particulate Au which may lead to variable Au content in samples.

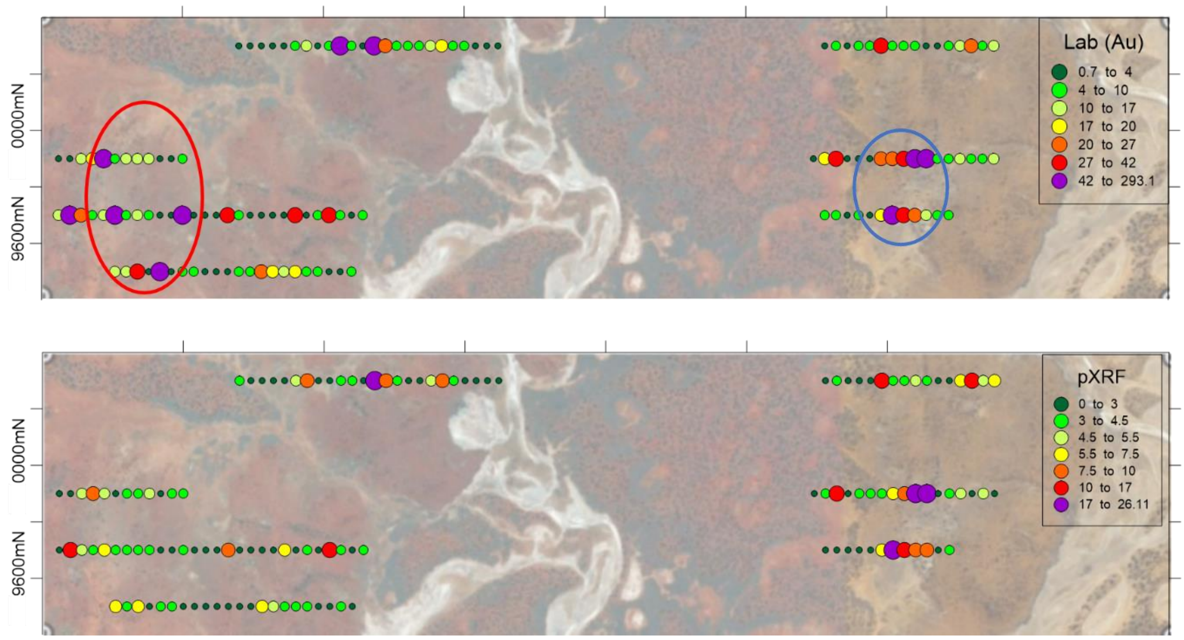


Figure 56: A NE Yilgarn soil survey consisting of seven traverses and 130 samples provided an initial test of the capabilities of the detectORE™ method. There is good agreement between the two most northern and the eastern traverses; the blue circle shows a cluster of samples that would have been worth following up with reactive sampling™ methodology. The three western traverses are in reasonable agreement but punctuated with high Au values (circled in red) most probably due to nuggety-type samples. The pXRF values are in nominal units (similar to dF) and are compared with laboratory ppb Au.

A set of RC samples from near the same location as Figure 56 were analysed by fire assay, and compared with detectORE™ before and after sieving at <1 mm. Unprocessed samples (RAW) gave reasonable agreement with sieved (<1 mm) samples for detectORE™ but less so with fire assay (FA) data (Figure 57 to Figure 59).

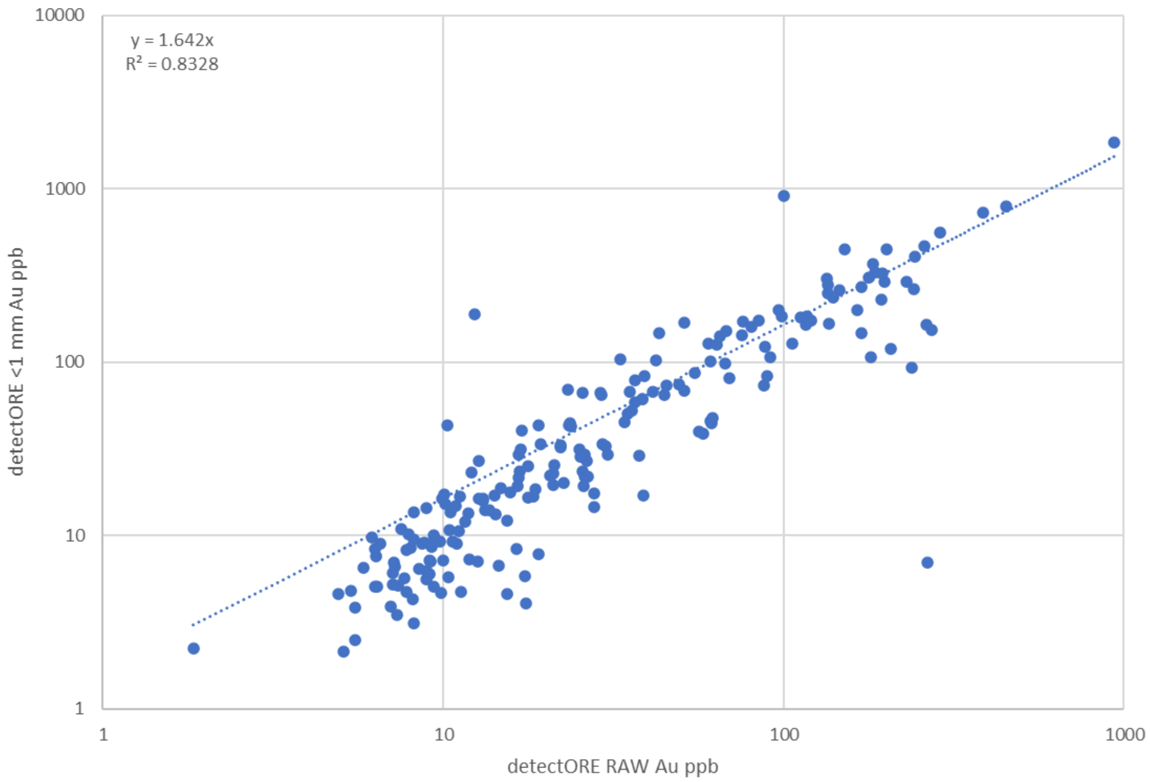


Figure 57: Comparison of unprocessed versus sieved (<1 mm) RC samples from NE Yilgarn by detectORE™.

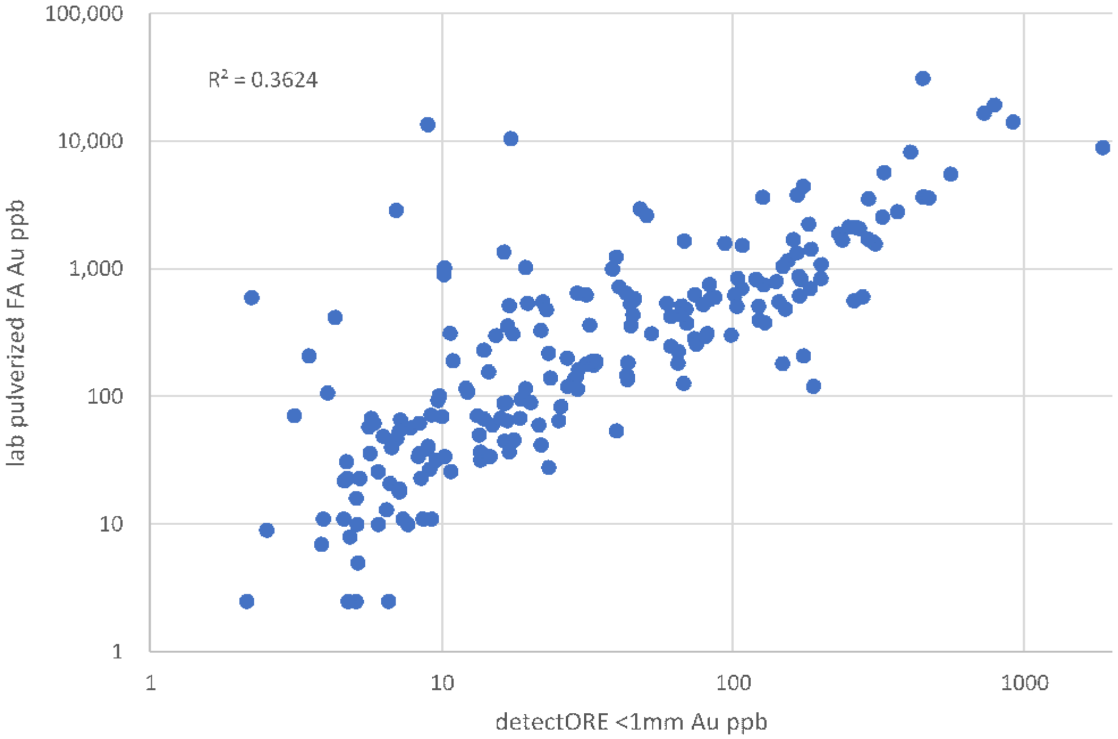


Figure 58: Comparison of detectORE™ with FA for sieved (<1 mm) CDI RC samples.

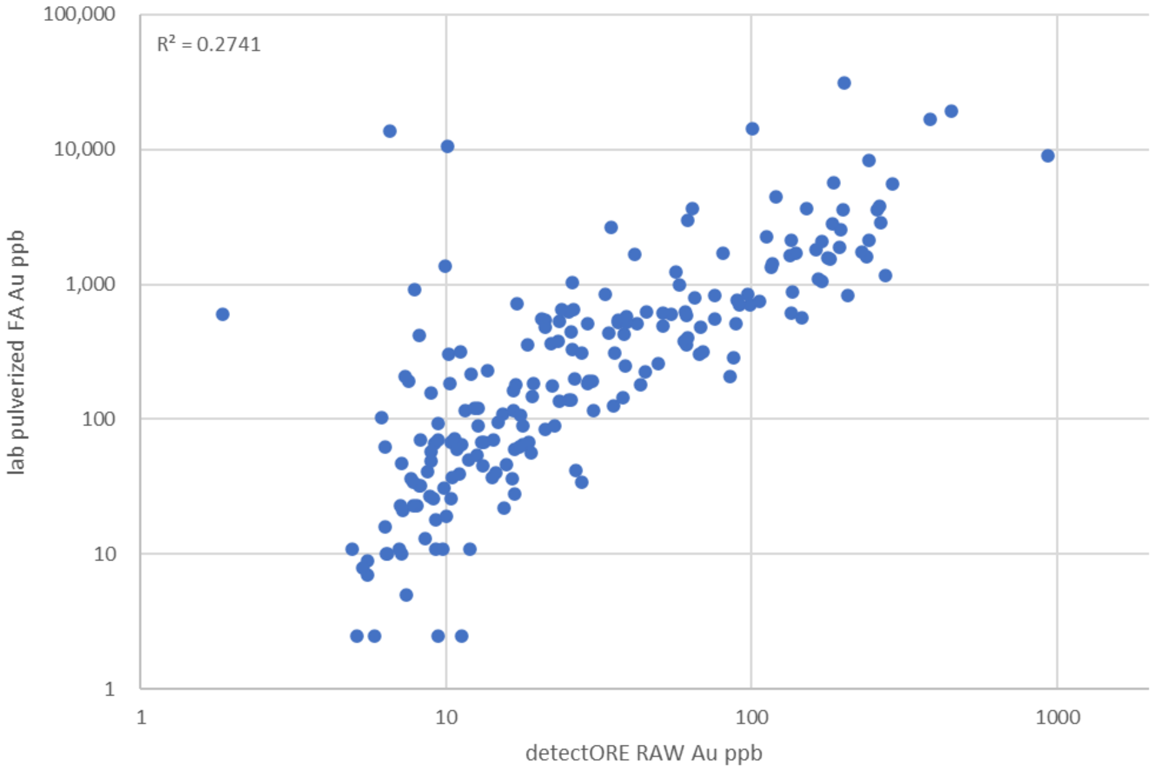


Figure 59: Comparison of detectORE™ with FA for unprocessed RAW detectORE™ sample.

Samples containing (i) carbonaceous ore, (ii) sulphide ore and/or (iii) coarse Au present challenges for both commercial testing laboratories and for detectORE™ technology. Refractory Au is also a challenge for processing of ores of this type. A total of 320 RC samples were received from West Africa (Cote d'Ivoire, CDI); many of the samples were black or very dark grey indicating carbonaceous and/or sulphide material. These samples were generally >1 kg so that multiple test work could be performed using the drill cutting procedure described above. Company fire assay data were provided with the samples enabling comparisons with different detectORE™ widgets and BLEG performance. Enough material was available to investigate i) two widgets (W1 and W2) and ii) two digest times on some samples, to compare with fire assay (FA). Examples of the comparisons are shown in Figure 60 to Figure 68.

Comparing RAW versus sieving gave reasonable agreement with the CDI samples (Figure 60). Using W1, there was a reduced Au detectORE™ factor (dF) response on some samples compared with FA mostly due to carbonaceous and/or sulphide ore present. However, anomalous intervals in the drill cuttings identified by FA coincided with those found with detectORE™. By using a 6-hour W2 and a 18-hour W2 on the drill hole with samples that had the poorest agreement between FA and W1 (r^2=0.24) correlation was improved significantly (r^2=0.75 and r^2=0.86, respectively) over W1. Samples contained arsenic (As) and carbonaceous material. Some samples with high As had poor agreement between FA vs dF and FA vs BLEG; the poor agreement may not be caused by As *per se* but by the presence of carbon, with As content serving as an indicator of poor recovery. Comparisons between FA vs detectORE™ and FA vs BLEG were not as good as detectORE™ vs BLEG. The detectORE™ factor dF31 improved agreement with BLEG compared with dF1. High As on the CD may give a false positive for Au particularly if using dF1 (compared with dF31) but, coincidentally, may 'compensate' for the poorer extraction/Au precipitation dF value giving an ostensibly better agreement with BLEG (i.e., a coincident false positive).

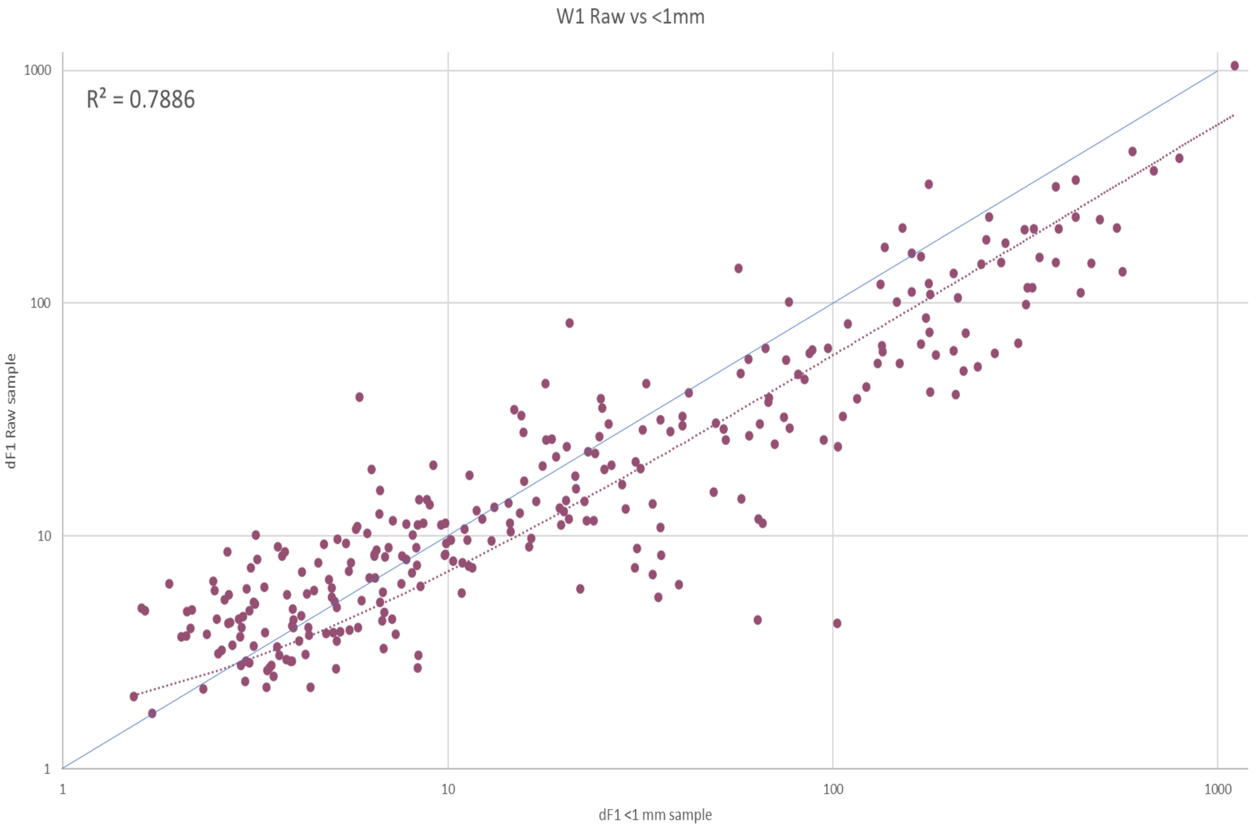


Figure 60: Comparison of Raw vs sieved (1mm) for detectORE™ on 320 RC samples (from Cote d'Ivoire) using widget W1. Reasonable agreement between Raw (unsieved) vs <1 mm using W1. The blue line represents the line of equality and suggests more Au is found in the <1 mm compared with the raw sample. This is not surprising as the raw sample is likely to contain more coarser material than the <1 mm and thus less amenable to extraction.

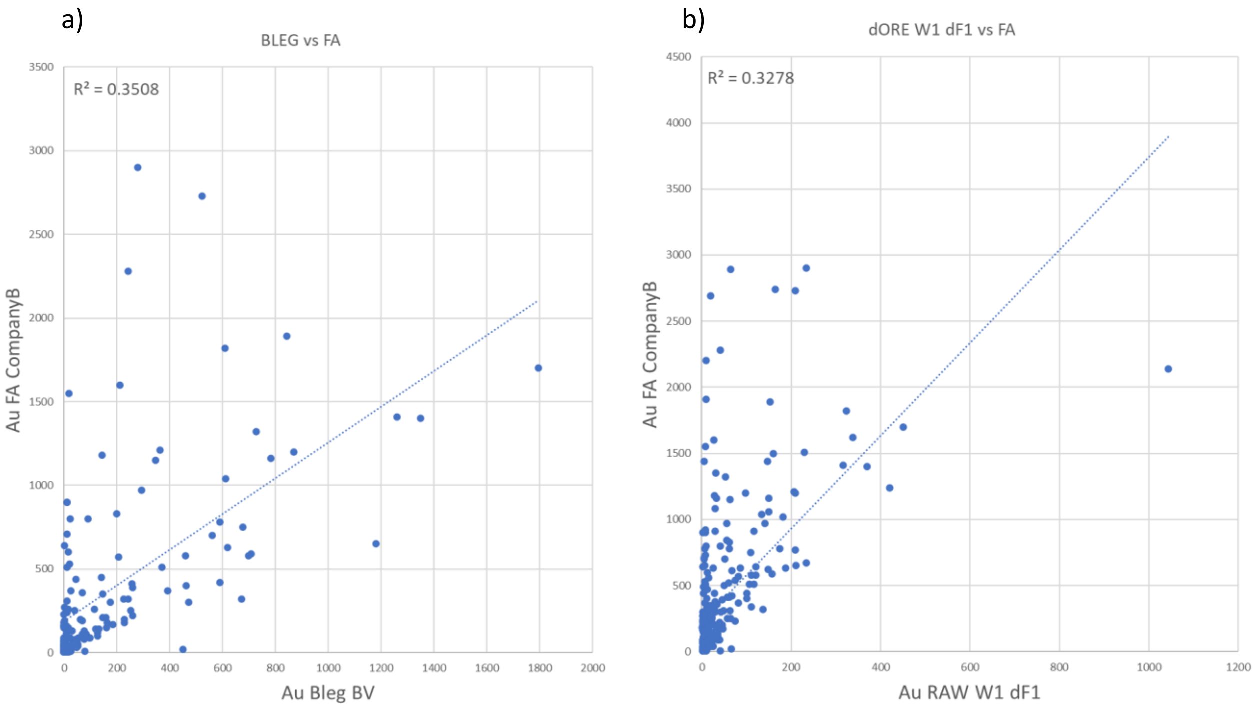


Figure 61: 320 Cote d'Ivoire (CDI) RC samples a) BLEG (Au ppb) versus fire assay (FA, conducted by the company, Au ppb) for 320 CDI samples; b) unsieved sample detectORE™ (dF1, W1) versus fire assay (Au ppb).

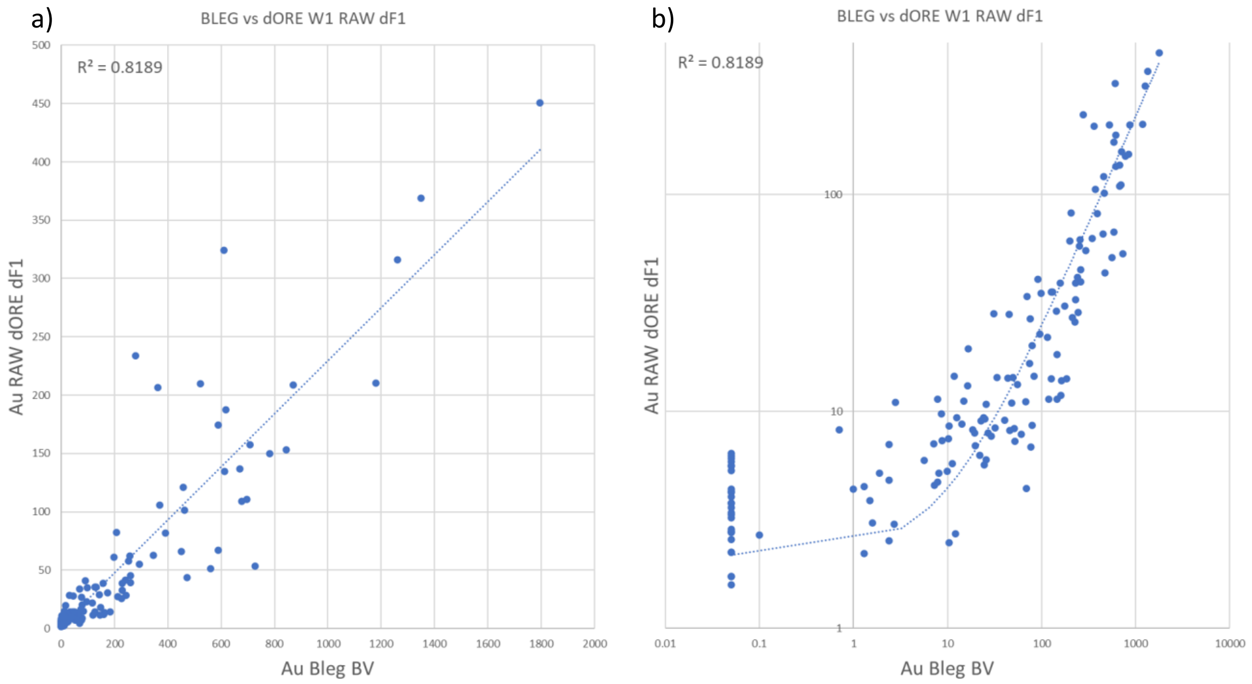


Figure 62: 320 Cote D'Ivoire RC samples a) BLEG (Au ppb) versus unsieved detectORE™ (dF1); same as a) but b) log-log plot BLEG versus unsieved detectORE™.

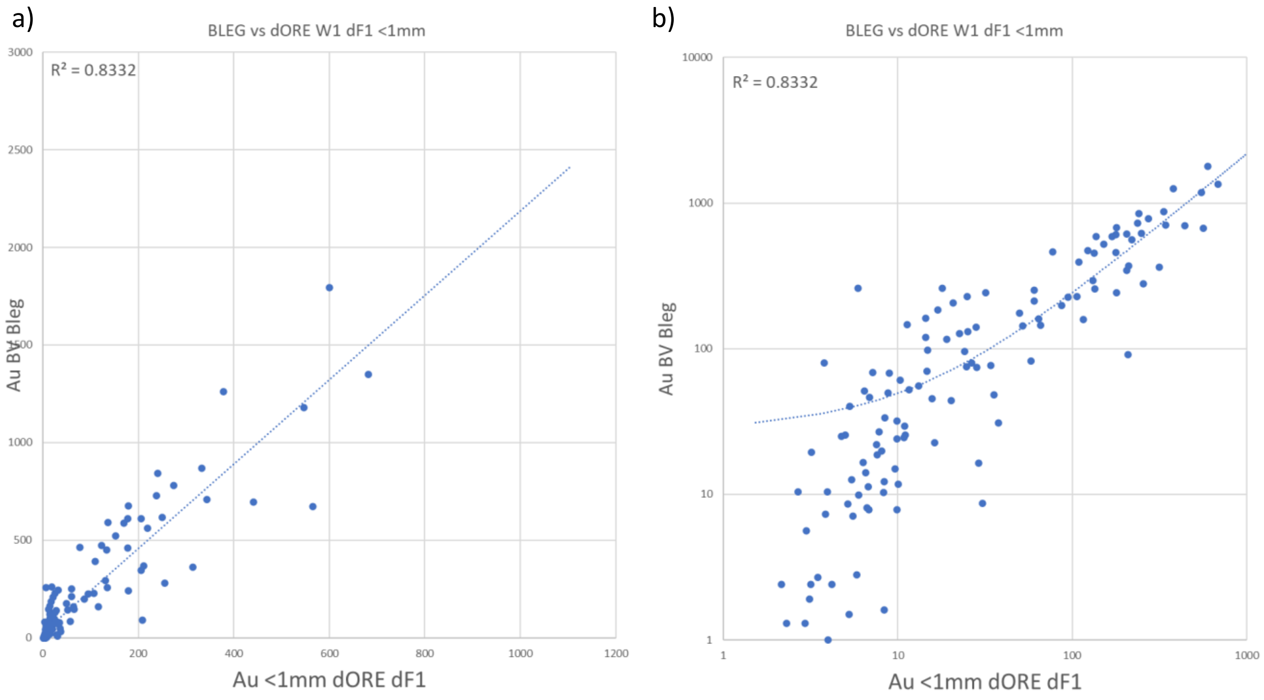


Figure 63: 320 Cote d'Ivoire RC samples a) BLEG (Au ppb) versus sieved detectORE™ (dF1, W1); same as a) but b) log-log plot BLEG versus sieved detectORE™

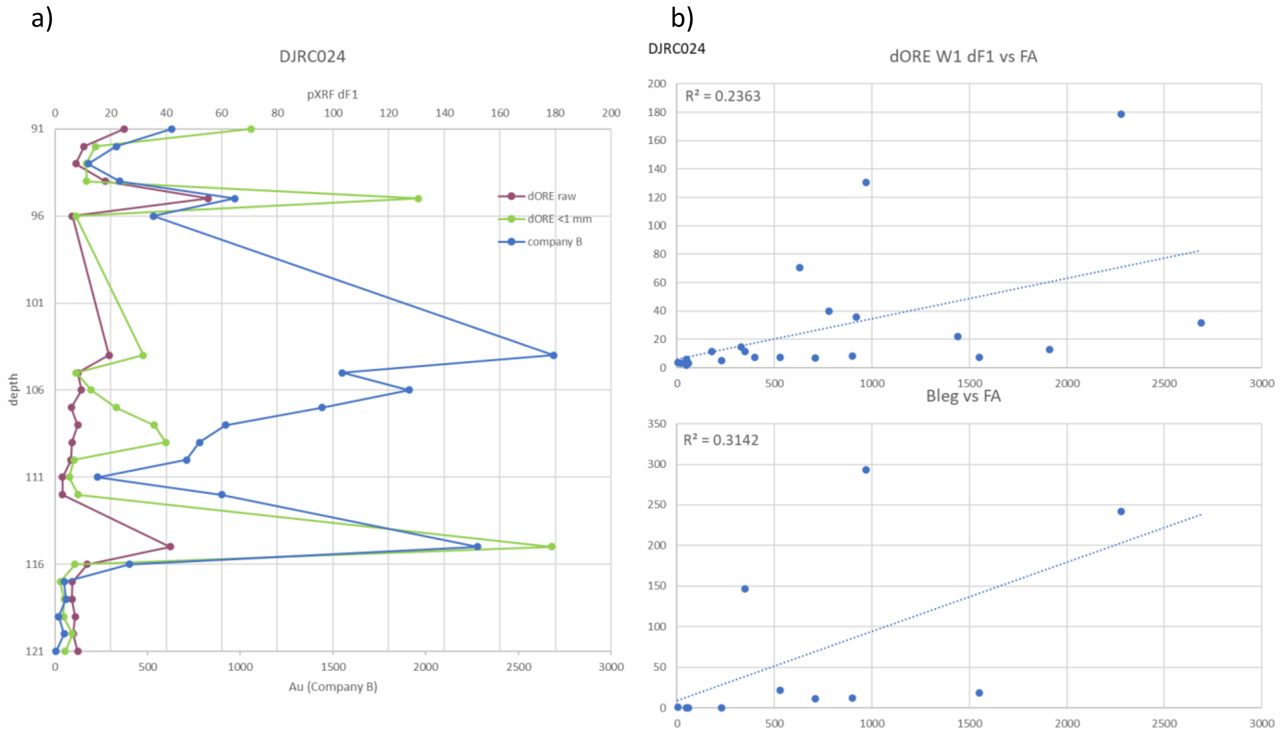


Figure 64: Cote d'Ivoire RC hole DJR024. There is poor agreement between company B fire assay results (ppb) and detectORE™ using W (compare with Figure 65); a) down hole plot with detectORE™ (dF1) unsieved, sieved and fire assay (Au ppb).

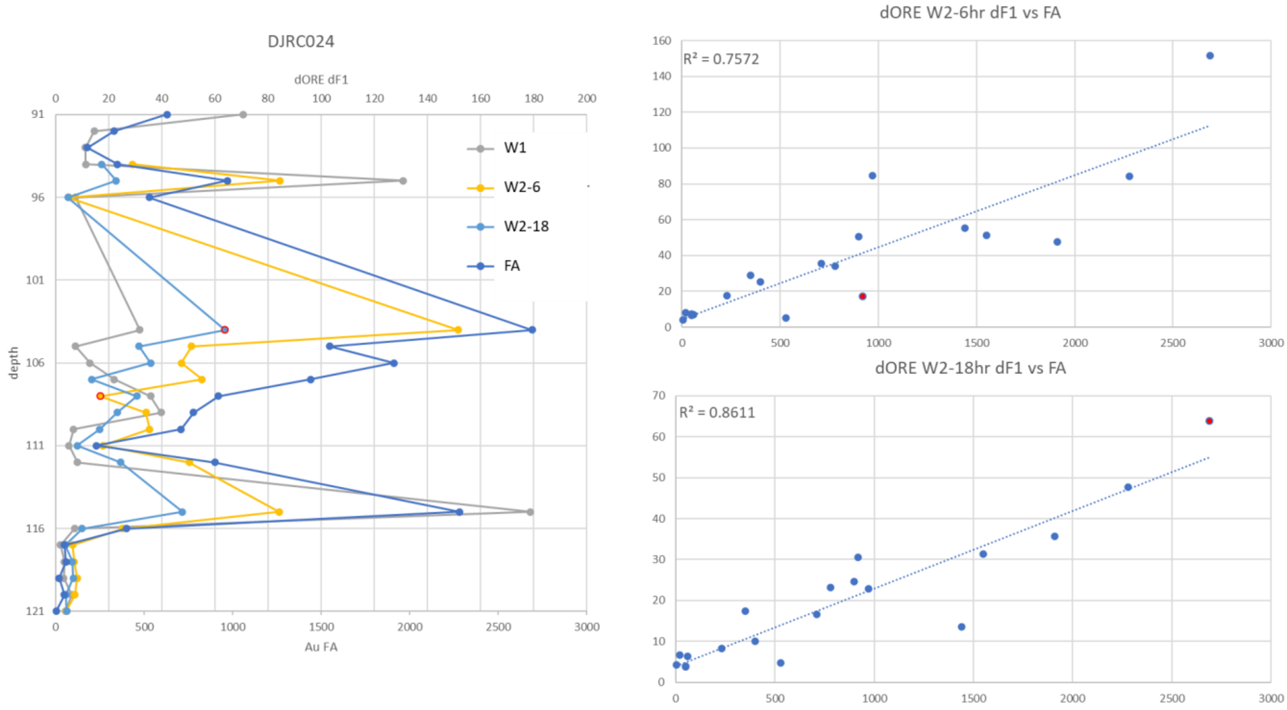


Figure 65: Cote d'Ivoire RC hole DJR024 contains samples that resulted in poor agreement between W1 (dF1) and FA (company B FA, Au ppb) (see Figure 64). But, with W2, there is much better agreement between the two methods both for a 6 hr digest process and an 18 hr process time. The red circles indicate a problem with the CD that may have affected the Au result.

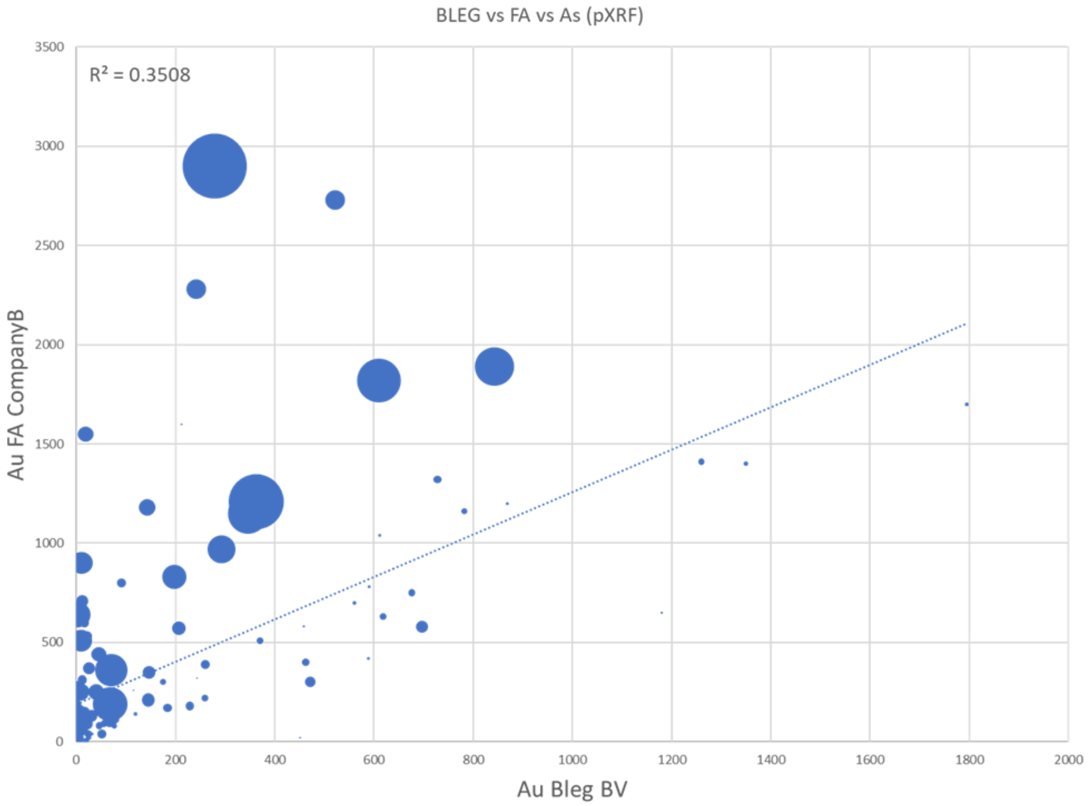


Figure 66: Fire assay vs BLEG for samples from Cote d'Ivoire. Symbol diameter size is related to As content. Note how samples with higher As content have less recoverable Au (poorly extracted or re-adsorbed) than samples with lower As content as indicated by the BLEG results. The As may be indicative of samples containing more refractive Au.

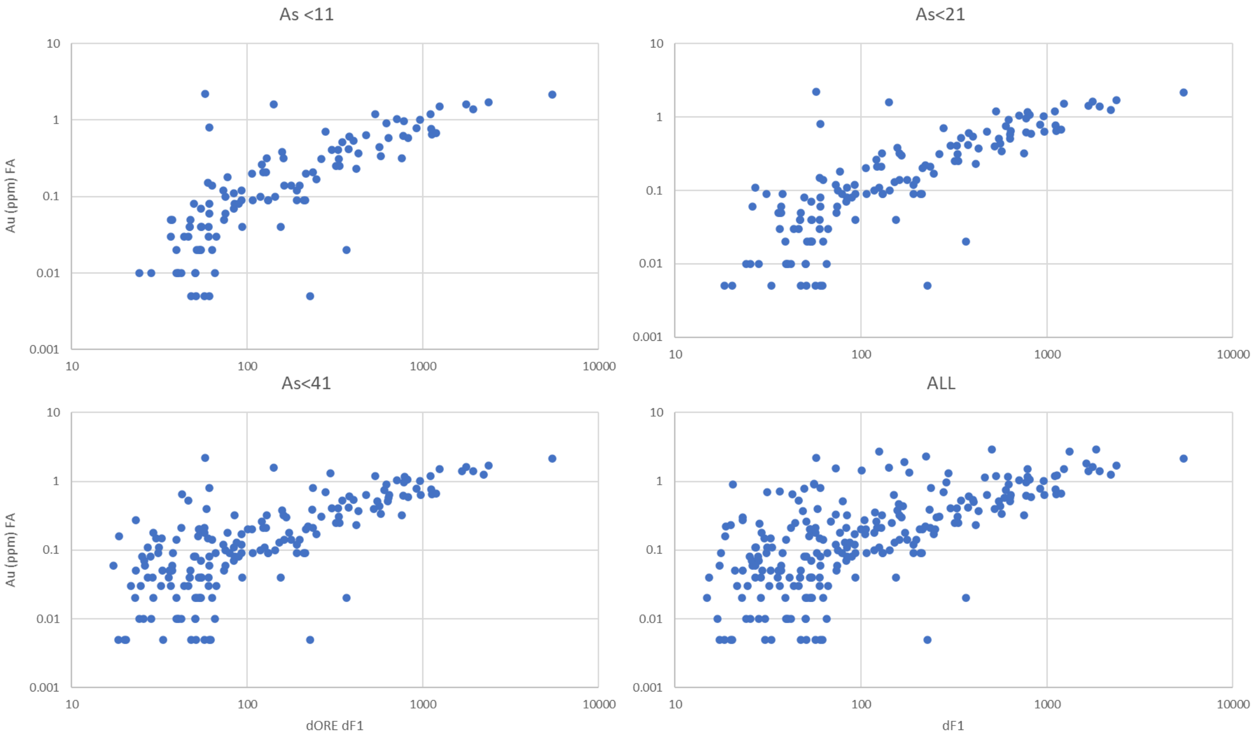


Figure 67: Increasing As (from <11 ppm to >41 ppm (ALL) as measured on the CD by pXRF) with these particular samples from Cote d'Ivoire leads to increasingly poor agreement with fire assay and detectORE™ (dF1, W1). Arsenic is a symptom of the poor agreement between FA and detectORE™ rather than the cause.

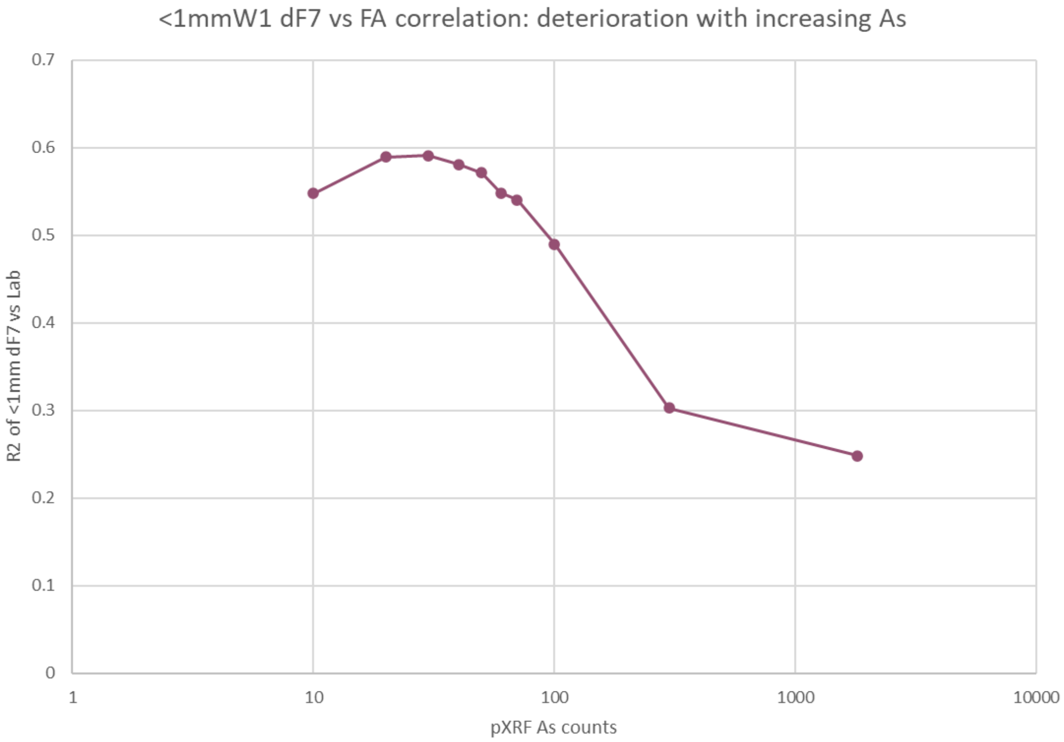


Figure 68: Correlation between detectORE™ (W1) and FA decreases with increasing As recorded on the CD by the pXRF.

For Cote d'Ivoire laterite augered and soil samples, coarse sized (some >10 mm), reddish-brown lateritic material was augered with the corresponding soil samples (n=139) from two traverses. Samples were pulverised prior to further testing work. Samples were rarely >1 kg and so only limited comparative trials could be performed. Many 25 g aqua regia (AR) lab pulverised replicates on different samples indicated nuggety Au. For example, one sample returned 29 ppb, 136 ppb, 540 ppb for triplicate analyses from a commercial laboratory (Figure 69). The PPPB test work with 25 g and 250 g samples was performed using W1 only.

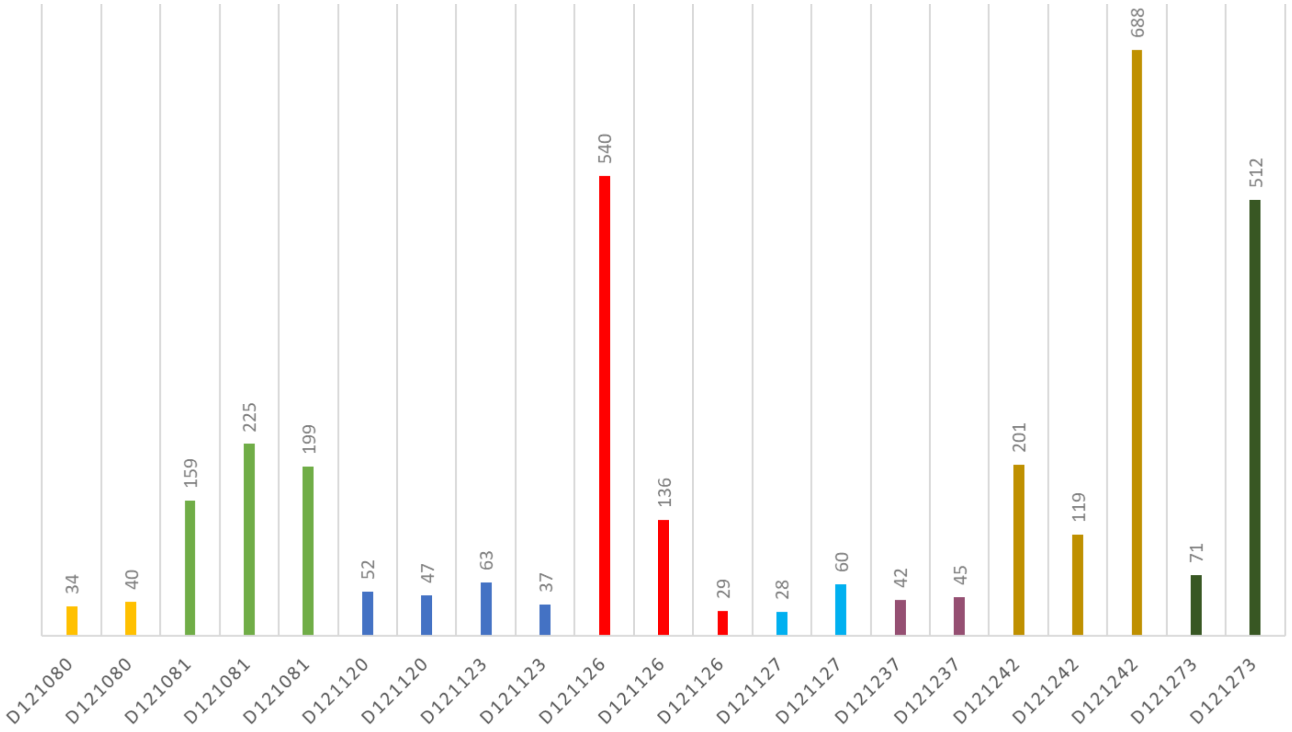


Figure 69: Commercial laboratory replicate Au analyses (ppb, shown at top of columns) of aqua regia digested and ICPMS analysed soil and augered samples from Cote d'Ivoire. Poor agreement between some samples indicates the presence of particulate Au.

Augered samples by aqua regia (BV) and by detectORE™ from the two traverses showed coincident anomalous areas (Figure 70). There was better agreement between W1 (250 g) and AR (BV) than between W1 (25 g) and AR (BV). The anomalous areas identified by AR (BV) results (around 207500E:773200N and 208500E:773600N) were found by W1 (250 g) but less convincingly so by W1 (25 g). Starred highly anomalous areas (top 5% of data) were identified by all three methods. The AR (BV) point anomaly at 208000E, 773600N was not identified by either W1 method, and additional single point anomalies found by W1 (25 g) were not found by W1 250 g or AR (BV). Background areas were in better agreement between AR (BV) and W1 250 g than with AR (BV) and W1 25 g. Many of the differences between the data could be explained by the particulate Au.

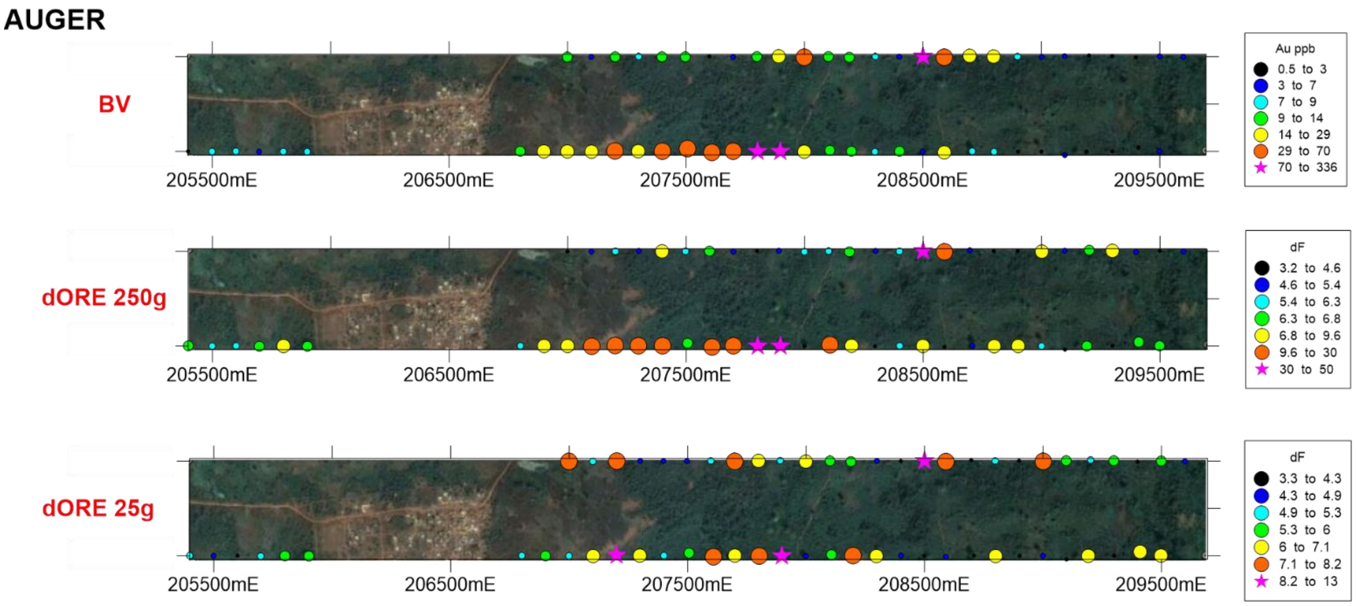


Figure 70: Augered samples from two traverses are compared by three analytical methods: a) BV indicates AR digest using 25 g; b) dORE 250 g indicates W1 using detectORE™; c) dORE 25 g indicates W1 using detectORE™.

Soil sample results (aqua regia and 250 g W1) indicated an anomalous area towards the centre of the southern line which is coincident with the augered sample results; however, soil data were in poor agreement with the other data (Figure 71).

In conclusion, PPPB found contrast between background and anomalous areas was greatest with 250 g samples (vs 25 g samples), and with auger samples vs soils (Figure 72 - Figure 73). There was also better agreement between 25 g AR results and the larger weight 250 g detectORE™ Process than with 25 g detectORE™. Despite the presence of erratic, low precision replicate results from the aqua regia digest, the anomalous areas identified using aqua regia were coincident with those found by detectORE™ .

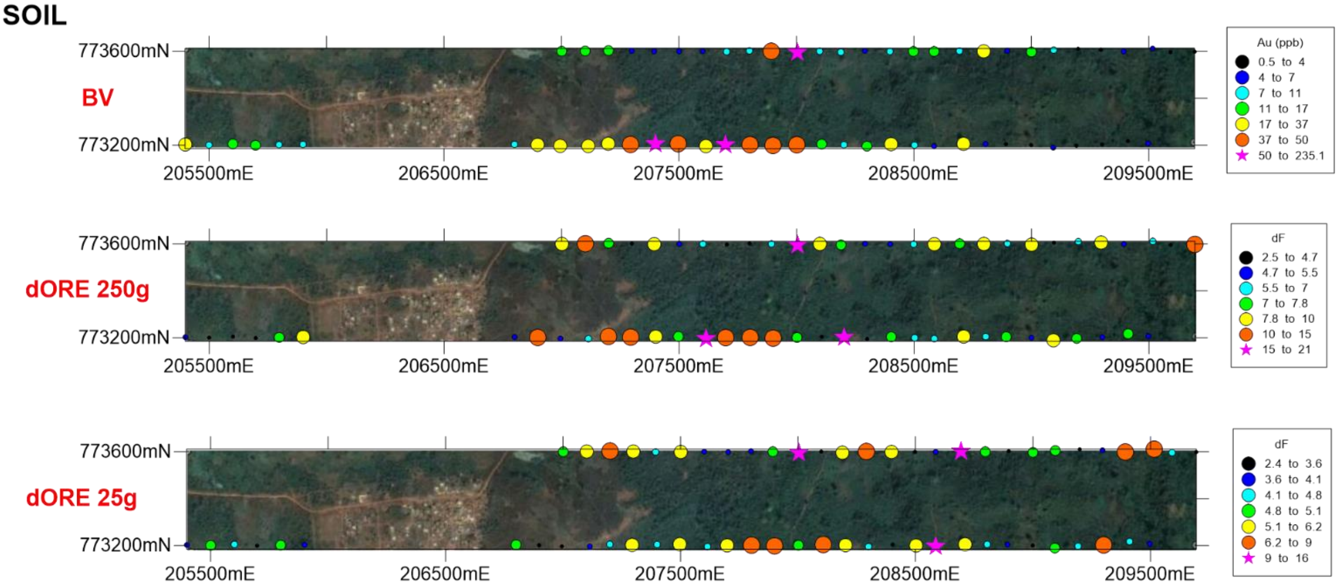


Figure 71: Soil samples from two traverses are compared by three analytical treatments: a) BV indicates AR digest using 25 g; b) dORE 250 g indicates W1 using detectORE™; c) dORE 25 g indicates W1 using detectORE™.

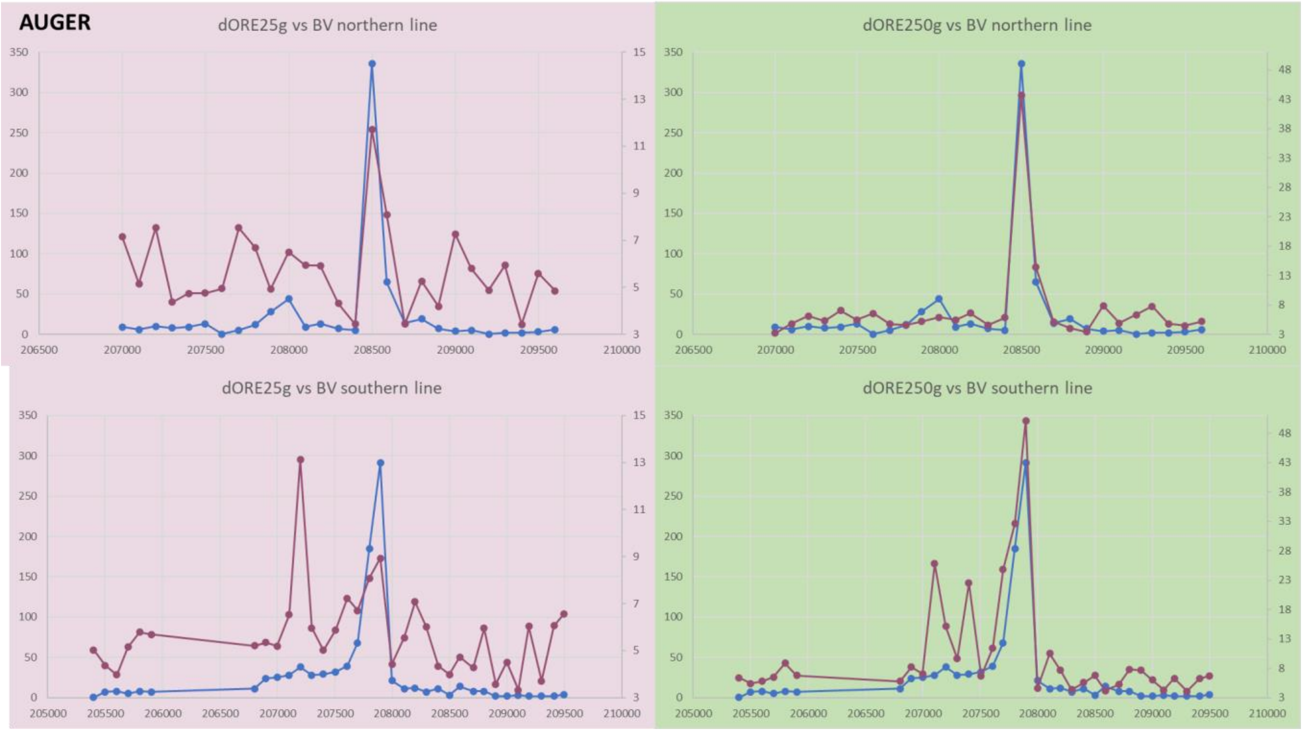


Figure 72: Line plots of augered sample data shown in Figure 70. The left hand axis is for the aqua regia data (ppb Au) and the right hand axis is the dF value recorded by detectORE™. Green background signifies 250 g and pink background 25 g.

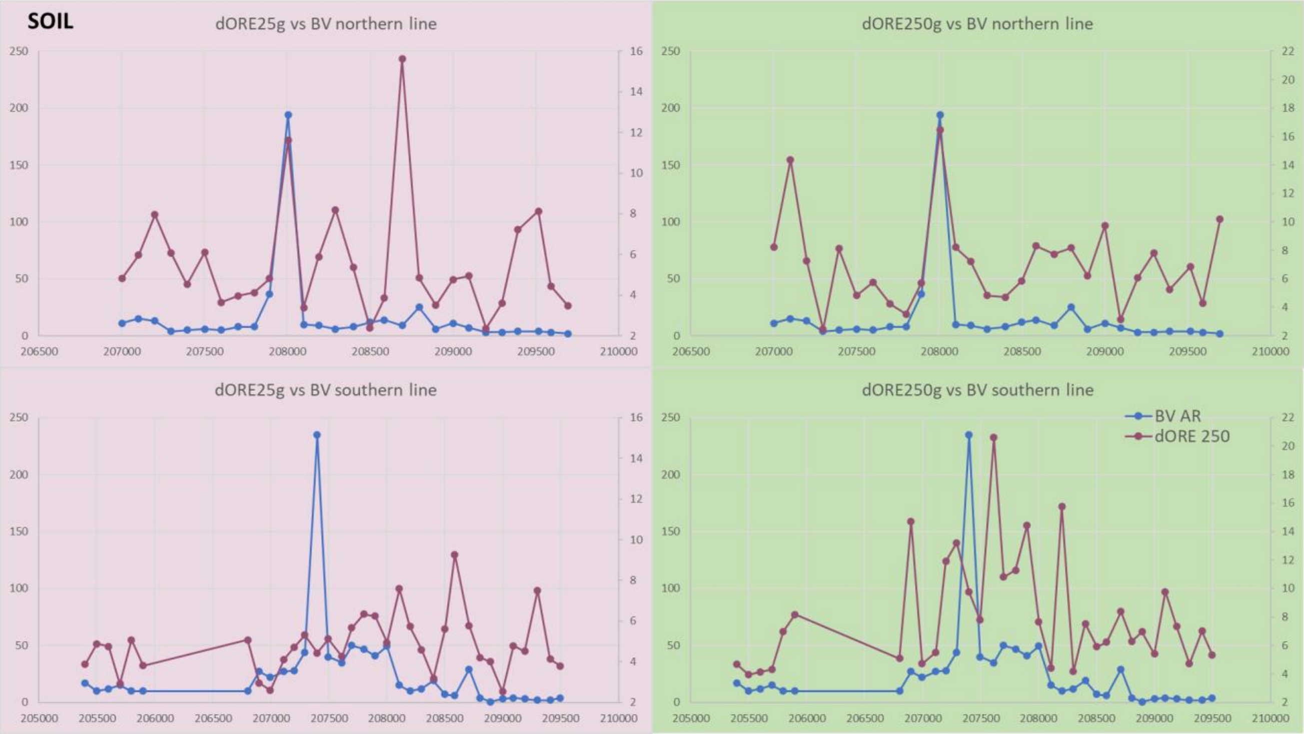


Figure 73: Line plots of the soil sample data shown in Figure 71. The left hand axis is for the aqua regia data (ppb Au) and the right hand axis is the dF value recorded by detectORE™.

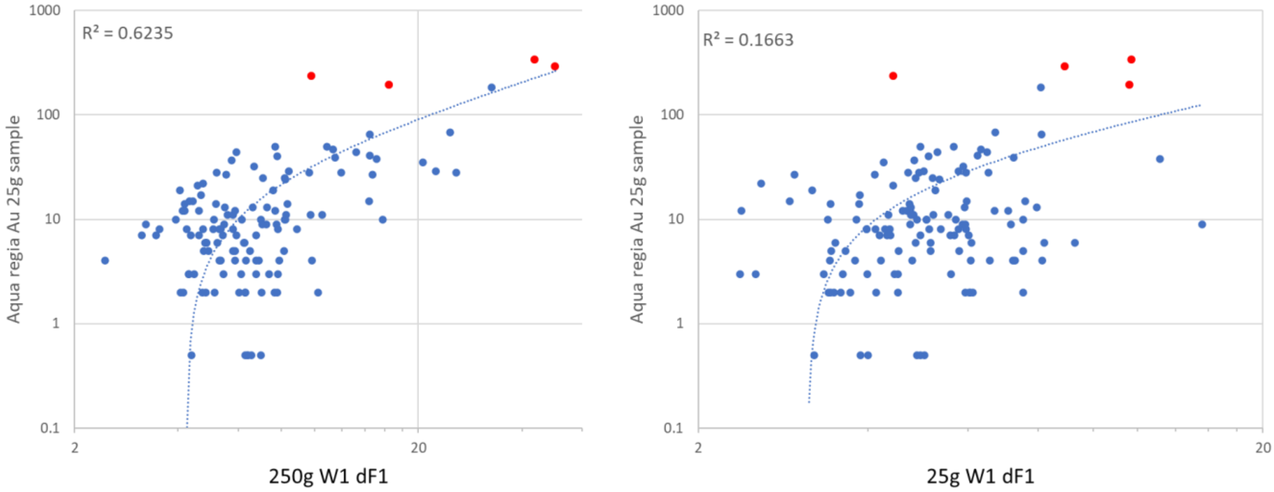


Figure 74: Comparison between aqua regia vs detectORE™ using either 250 g (left hand side plot) and 25 g (right hand side plot). Correlation is much better with aqua regia using the larger sample weight for detectORE™ which is better for particulate Au samples.

Calcrete and soil samples (n=240) from a single soil traverse in the Eastern Goldfields were received unsieved and contained coarse materials (some >100 mm). Ordinarily, when Au is present, coarse calcrete is elevated in Au but coarse material is unsuitable for detectORE™ analyses presenting a sample preparation dilemma. Usually, detectORE™ in field conditions requires samples to be sieved to <1 mm prior to processing but, for these samples, the coarse calcrete was removed during sieving. The assumption tested was that there would be sufficient calcareous material (containing Au) within the fine-sieved material to detect an anomaly.

Test work for these samples included a comparison between two different types of widget (W1 and W2), a 25 g aqua regia (AR) digest following pulverising to 75 µm and a 250 g BLEG followed by

ICPMS. For the auger samples, the analyses by aqua regia indicated that Au concentrations were low averaging 14 ppb and more elevated in the western part of the traverse (maximum of ~ 80 ppb) (Figure 75). BLEG results using 250 g of <1 mm indicated elevated Au in the western part of the traverse (as with AR) with a maximum of 50 ppb that was not coincident with the position of the AR maximum. The two detectORE™ digests produced data elevated in the western part but the contrast was not as great as that for AR and BLEG. The maximum for BLEG, W1 and W2 were coincident (Figure 75). For soil samples, the Au was highest in the western part of the line but the strongest anomaly was in the centre of the line. Insufficient material prevented a BLEG digest for all the samples of the traverse and no AR data were available from the company.

In conclusion, despite there being low Au for these sieved calcrete samples, detectORE™ produced anomalies consistent with the laboratory data. Auger and soils produced different anomalies indicating that either sample media could be used for exploration and generate areas of interest for follow up work. PPPB was not provided with any data indicating the location of any mineralisation found as a result of any subsequent drilling undertaken.

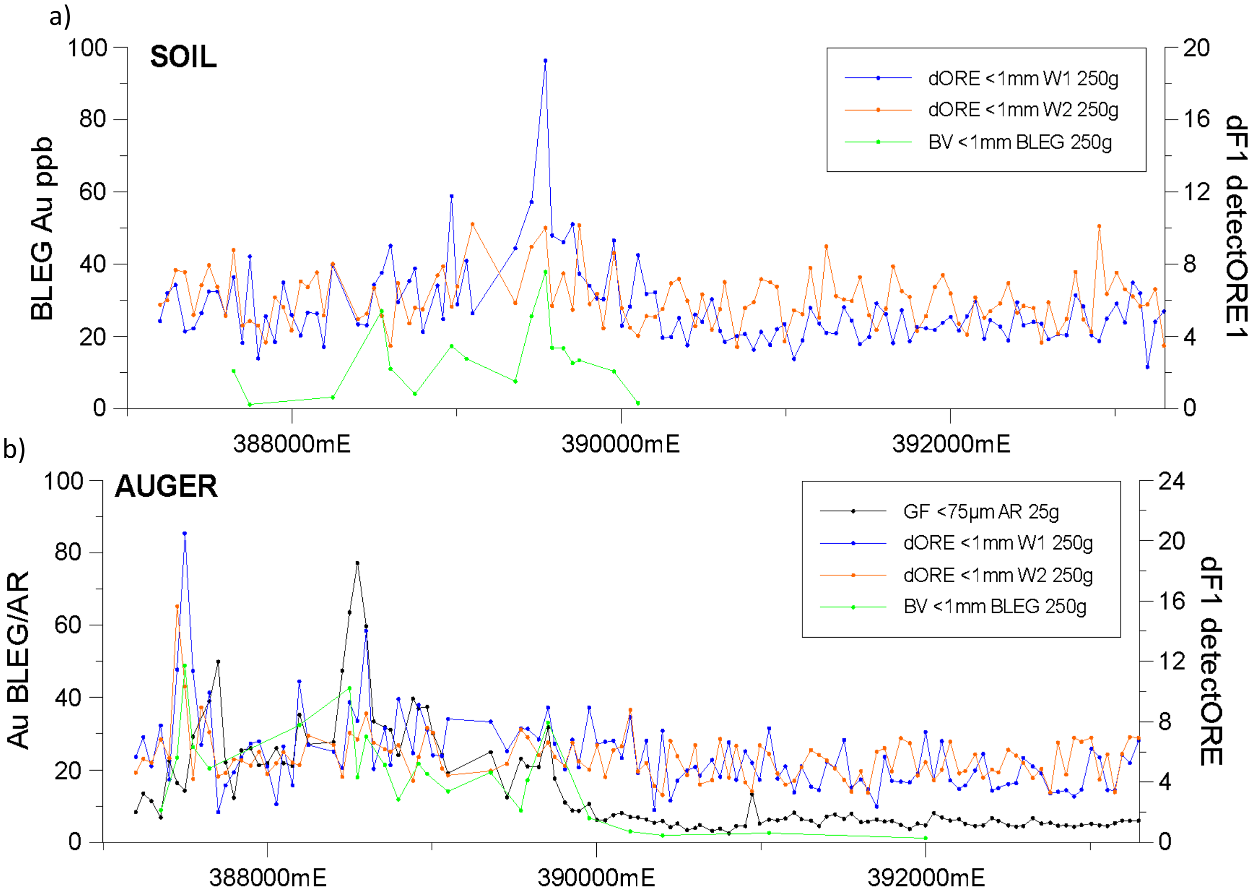


Figure 75: Comparison between a) soil and b) auger using aqua regia, detectORE™ (W1 and W2) and BLEG. Gold results are generally low (average <14 ppb for augered samples with soils lower still) but comparison between the methods produced similar trends with some coincident maxima.

2.19 Field work

PPPB collected and analysed 250 soil samples from the NE Yilgarn. Soil samples were sieved in the field and brought back to a base camp for analysis. Samples were put through detectORE™ process over a 24 hour period. One person using detectORE™ could process in the order of 100 sieved samples per day and provide analyses so that decisions could be made on site for the following day of sampling.

Two areas of interest (west and east) were initially targeted based on information provided by the company (Figure 76). The western area already had previous exploration activity and was initially targeted. The eastern area had undergone less previous exploration. Over a six day ‘reactive sampling™’ lines of strike and anomalous zones were determined in the field using detectORE™ (Figure 76). The field trip was restricted to six days and results were to be followed up in a subsequent field trip and are not reported here. Reference materials (216b and 62f) were run with each sample batch analysed and the results indicated satisfactory precision (relative standard deviation for 216b and 62f was 10.1% and 8.2%, respectively; Figure 77).

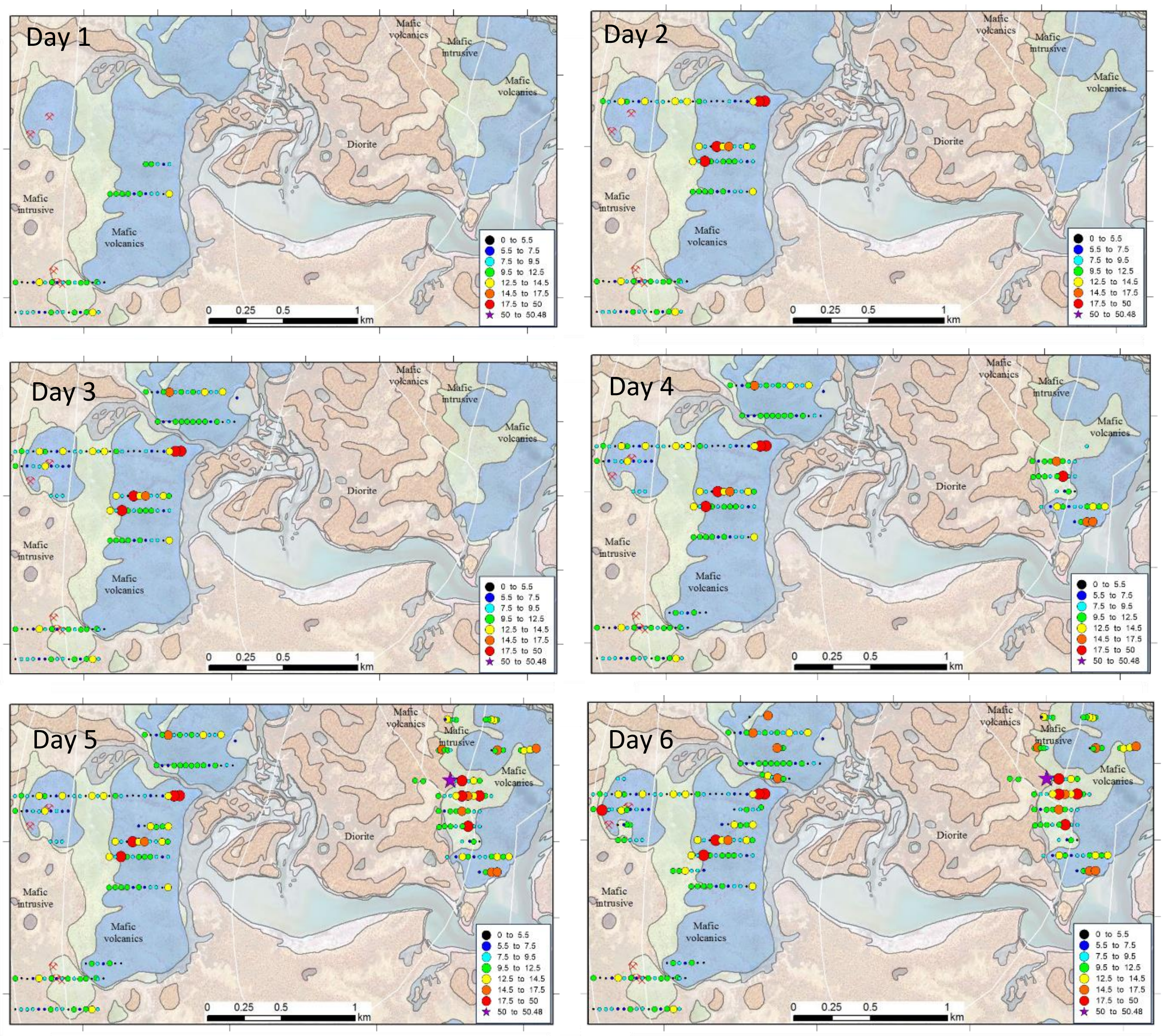


Figure 76: Example of reactive sampling™ using detectORE™ from a field trip conducted by PPPB in northern Yilgarn. From arrival on site (Day 1) to Day 6 the geochemical map was iteratively generated in two areas. Lines of strike and anomalous zones to be followed up in subsequent field trips. Gold data reported as dF.

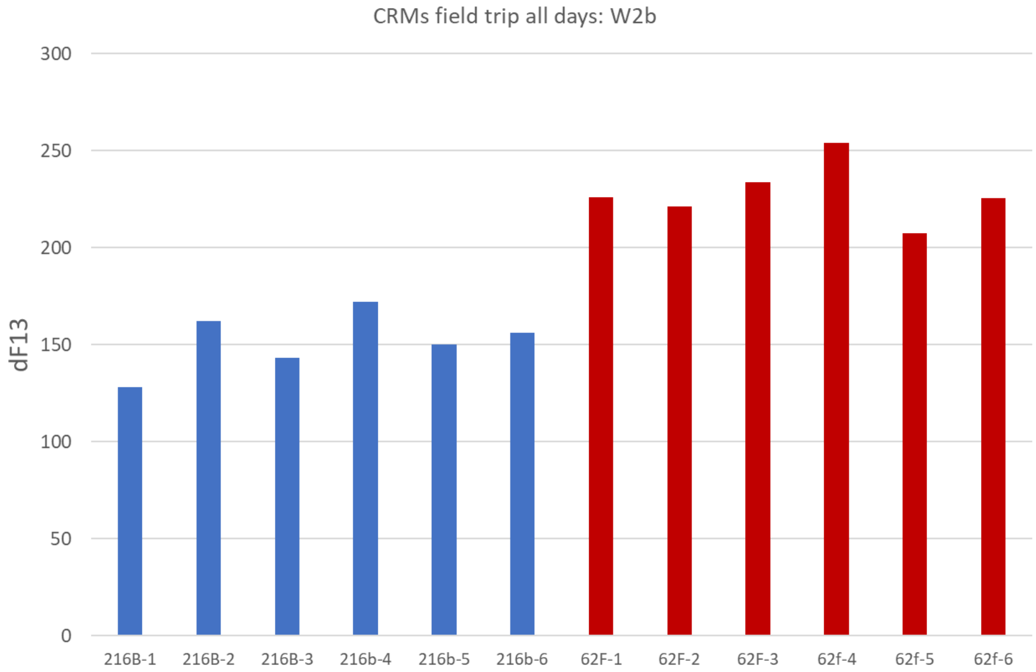


Figure 77: Reference materials (216b and 62f) were run for each batch analysed. Precision was satisfactory.

3 DISCUSSION

The detectORE™ technology over the course of this project has evolved to a rapid, robust, safe and fit for purpose field analytical method for Au. The technology uses a Method (how the Au is extracted from the sample, concentrated on the CD and read by the pXRF) and a Process (how reactive sampling™ for Au will lead to a paradigm shift to accelerate Au exploration).

Early experiments set up physical parameters for the analysis of Au in CRMs based on experiments conducted in CSIRO. Data from these early experiments verified the CSIRO results using widget W1. An early decision was made to develop a method for another widget (W2), principally driven by safety (Table 2). Widget W1 is classed as (i) 'dangerous goods' and not easily transported by air, (ii) hazardous and (iii) toxic requiring treatment before disposal. The W1 will still have important applications in a field laboratory facility or a mine site laboratory.

Table 2: Comparison of characteristics of widget W1, and two types of widget W2 (2a and 2c). Although the efficacy of W1 is the best of the three other characteristics pertaining to safety and transport are less desirable. Widget W2c was *selected* as the best widget overall.

Property	W1	W2a	W2c
Dangerous goods	Yes	Yes/No	No
Toxicity	Yes	Moderate	Slight
Hazardous	Yes	Moderate	No
Efficacity	Best	Good	Good
Time	24 hour	24 hour	8 hour

Optimisation experiments benefited from the immediacy of the pXRF analysis and the rapidity of the detectORE™ Process, and ensured that the research progressed dynamically and in-house (without having to wait for analyses from commercial laboratories to verify data); for example, experiments performed on Day 1 can be used to design experiments on Day 2 and is akin to the detectORE™ reactive sampling™ in the field. During the project, the Au dissolution time was reduced from 24 hours to 8 hours (Table 2). This has many advantages for exploration since a true turnaround time of less than 24 hours can be realised; as a result, the additional time saving may be used for other tasks such as preparing the next batch of samples, processing data and, above all, setting the sampling strategy for the next day.

Most (if not all) analytical methods involving aqueous dissolution of sample with reagents such as aqua regia will not dissolve the entire sample and may leave part of the analyte undissolved and therefore not reported in the concentration result (Table 3). Some partial extraction methods for Au include BLEG, aqua regia and MMI; aqua regia will leach a relatively high proportion of Au in finely ground samples but will usually leach less if the sample is coarse grained and contains Au encapsulated in minerals such as quartz or sulphides. Furthermore, samples may also contain carbonaceous materials so that even if reagents are robust e.g., aqua regia, Au can re-adsorb back onto the sample from the digest and not get reported in the concentration result. The metallurgist is very familiar with this problem of 'preg-robbing' which can result in significant Au production losses. The detectORE™ Method uses reagents that partially extract the Au but information on its extractability of Au is less well known than with other more familiar digestion methods. PPPB has tested many hundreds of samples from different countries and deposit styles and is satisfied that Au can be dissolved from samples and that the propensity for it to be adsorbed on carbonaceous material is weaker than for a BLEG digest. In addition, to simplify the field procedures, the detectORE™ Method uses sieved sample material in order to avoid having to pulverise samples in the field; BLEG is another digestion method that typically uses sieved rather than pulverised material. The detectORE™ Method, like BLEG, attempts to 'compensate' for this lack of pulverising and to gain better representativity by using a very large sample size (e.g., 250 g of sample or more) in comparison to aqua regia or fire assay (e.g., 25, 40 or 50 g). Gold is notorious for being too difficult to precisely determine concentration because of its particulate habit of occurrence; the most common way of reducing this so-called 'nugget effect' is to use larger samples.

Table 3: Comparison of some conventional digestion procedures and methods for Au with detectORE™.

Analytical method/digest	% of Au analysed	Sample size	Adsorbtion	Ease of use; requirements, cost	Field friendly	Cost per sample
Aqua regia	High	Small	Low	Heat, acid	Low	$
BLEG	Medium	Large	Medium	Cyanide permit; DG; toxic; hazard	Low	$$
MMI	Very low	Small	?	Certified lab use; mixer	Low	$$
Fire Assay	Very high	Small	N/A	High power, furnace, $$	Low	$$$
Photon Assay	High	Large	N/A	High power, radiation, $$$	Low	$$$
detectORE™	Low-medium	Large	Low	Mixer	High	$$

At the commencement of the project, the hypothesis that a partial digest method was suitable for Au exploration required testing; such a hypothesis pertains to exploration rather than a simple comparison between other analytical methods. Extensive laboratory testing of samples from a variety of Au projects demonstrated that it was possible to identify geochemical anomalies (for soil maps or drilling programs) using the detectORE™ Method. For example, preliminary laboratory analyses on samples sent to us from the northern Yilgarn was followed up with a field trip to the site where they were collected. This confirmed that a total Au analysis in a sample is not necessary and the detectORE™ Method is suitable for Au exploration to identify new anomalies and confirm existing ones.

PPPB found that detectORE™:

1) Partial digest results provide sufficient information to minimise false negative anomalies during exploration.
2) When used *in the field* with the reactive sampling™ process it enhances single point Au anomalies into multi-point robust anomalies.
3) Identified false positive anomalies by follow up analyses; exploration effort is commonly wasted on samples that have been contaminated (lab or field) or showing spuriously high results for unknown reasons. False positives can be verified within 24 hours.
4) Identified background samples so that transport to, and analysis at, a distant laboratory could be avoided. Low Au concentrations generally do not require systematic further analytical confirmation; if these samples were identified in the field, then this alone would amount to considerable cost savings for an exploration program.

The reactive sampling™ approach used in the detectORE™ Process will transform an 'old method' mineral exploration program into one that provides in-field feedback to a company using it, allowing for rapid decision making and exploration.

Typically, Au data from an exploration program (e.g., soils or drilling) are rarely normally distributed. Au concentrations in samples are commonly markedly skewed towards lower concentrations with some relatively high to very high Au concentrations indicating mineralised samples. Comparing methods of Au analysis statistically may be problematic since detectORE™ is a partial extraction method; notwithstanding, PPPB has attempted to do this for the case studies. Comparative analyses using scatter plots (and/or with statistical methods) are excellent at visualising the data. However, linear axes scatter plots over a wide range of Au concentrations tend to ‘hide’ the multitude of lower concentrations compared with the few higher concentrations; an axis break may counteract this issue. Logarithmic scatter plots, on the other hand, tend to overstate the lower concentration differences and understate the higher concentrations. PPPB has found that using both these plots is valuable. Scatter plots provide an instant visual appraisal of *all* the data without reducing the relationship to a single statistical value e.g., a correlation coefficient.

The detectORE™ data are reported as a detectORE™ Factor, or dF, rather than a ppb concentration. The dF is a pXRF measurement value created from PPPB software. It is a measurement of the amount of Au on the CD which in turn is directly proportional to the amount of Au extracted from the sample. From analysing sponsor samples and communications with them, PPPB chose to report values in this way to avoid any confusion with actual ppb concentrations of the sample from, for example, a fire assay which will be significantly greater than the partial extraction method used by detectORE™. For soils, the dF values may still be directly plotted as values on geochemical maps. The dF values plotted in this way can discriminate between anomalous and background areas as if it were a ppb measurement. Kriging and contouring is possible if data are plentiful and regularly dispersed such as soil or auger survey data, or class coded if data are available as traverses only. For down hole, plots of data are better at visualising and assessing anomalies and thus a better representation of the results for use with in-field reactive sampling™ scenarios, as envisaged for the detectORE™ application. Good examples of soils and drill hole data were shown in the case studies and are beneficial to identify and visualise anomalies.

As with any other analytical technique, checks are mandatory to validate the data. Mineral explorers commonly include blind standards in a submission of samples. For the detectORE™ Method verification is required for the digestion, mixing and Au adsorption on the CD since something may have affected the process eg. incorrect reagent mix, mixer brake down, CDs being affected by environmental factors or exposure to deleterious chemicals. Manufactured dry pouched geochemical standards (known as RMs) were included with each sample run typically at a rate of about 3-5% of total samples analysed. Reagent and CDs were added to a standard pouch containing the reference material or RM, with the RMs containing commercial CRM diluted with background regolith material. RMs that were put through the detectORE™ Method were analysed at the same time as samples.

As part of the QC procedure, a set of Certified Collector Devices or CCDs were included with each analysis using the pXRF. The CCDs were analysed at least ten-fold each and have expected dF readings associated with them. The purpose of the CCDs is to ensure that the pXRF instrument is optimised and used correctly to analyse the known amount of Au present. Typically, between 4 and 8 CCDs were analysed with each batch of samples.

4 CONCLUDING REMARKS AND RECOMMENDATIONS

The primary objective of the project was to develop *'a method that analyses Au in the field which, currently, cannot be done. It is to be a robust, safe, field analytical method for Au exploration capable of achieving sensitivities of assay to approximately 10 ppb Au*'.

This objective has been achieved. Detection limits around 10-20 ppb Au were achieved, going down to <1 ppb in Au adsorbed from laboratory standards. The technology has been tested in the field and has been shown to be safe and able to withstand the harsh conditions of the Australian bush.

The secondary objectives were also achieved, as follows:

1) *Analysis of certified reference materials (CRMs) and laboratory standards using a range of high-quality commercial CRMs to assess the accuracy and precision of the detectORE™ Method, expanding upon the initial tests undertaken by CSIRO*. All the OREAS Au standards were analysed by detectORE and found to be in reasonable agreement with Au fire assay results. Some of the OREAS standards contained high levels of metals known to interfere with normal pXRF signals but because of the detectORE Method these did not interfere with Au determinations.

2) *Employing the detectORE™ Method to an extensive range of exploration sample materials, sourced from Western Australian mineral deposits and prospects with a preference given to those provided by the project sponsors. Test samples included surface soils, augered sub-surface soils including lateritic residuum and calcrete, stream sediments, drill hole cuttings from RAB, AC and RC, saprolite and sap-rock. Sample preparation techniques, designed for the field, were to be developed tailored to these different sample media. Duplicate samples were to be analysed externally by traditional laboratory methods*. Various exploration sample media from Western Australia (and around the world) were tested using detectORE and Au successfully determined (as measured against laboratory analyses). A method was developed whereby samples were sieved to <1 mm before the reagent was added. This negated the use of pulverising equipment and simplified field procedures.

3) *Evaluating the detectORE™ Method under field conditions. Suitable Western Australian mine site(s) and Exploration Project(s) selected to test the robustness of the detectORE™ Method under typical conditions found in a field camp*. Two field trips were successfully executed to test detectORE™ by the end of the MRIWA M0543 project, with several others completed since. The field trips demonstrated the utility of reactive sampling™ and how considerable resources can be saved by exploration companies by reacting to positive or negative Au results on the fly.

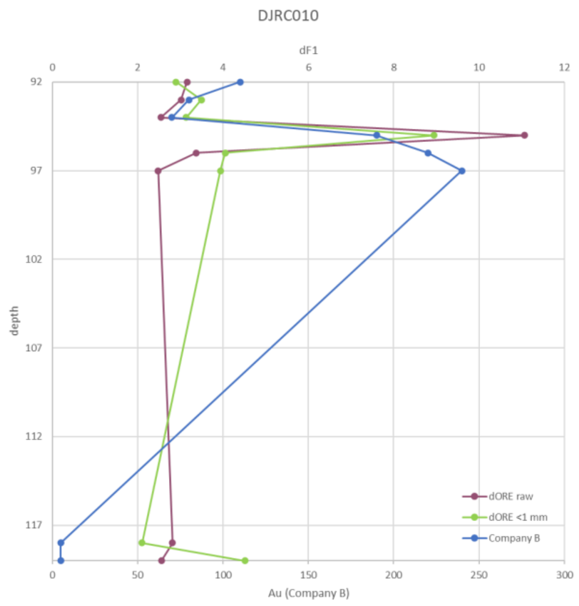
5 RECOMMENDATIONS FOR FUTURE WORK

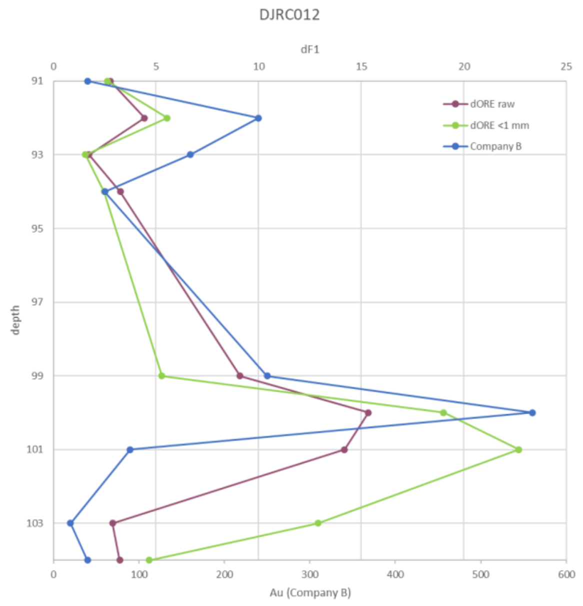
The outlook for detectORE™ is very positive. Sponsor companies have enthusiastically received the results that have emerged during the project. We are encouraged by this positive support and more case studies are planned both for the laboratory and the field. Additional research is to be completed for continuous improvement of the detectORE™ product, with the potential to extend its use to more elements such as silver and copper to broaden the appeal. PPPB is also aware of interest in other applications of detectORE™ for the Au value chain.

Commercialisation is now the logical step forward for detectORE™. The 'widget' system is well suited as a saleable product and its ease of use, safety and robustness of the advanced prototype has since been proven by sponsor uptake of the Method in the field. Commercialisation of detectORE™ is planned in the coming months on the basis of this work and other research that has been completed over the past twelve months.

6 APPENDIX

The following graphs show down hole data from Cote D'Ivoire drill hole samples (similar to Figure 64). The data show raw (RC samples as received), RC samples sieved to <1 mm and fire assay data (labelled 'Company B'). The provided samples are from drill holes that pass through mineralised 'anomalous' zones with the elevated Au generally been identified by the detectORE™ technology.


DJRC010
dF1
0 2 4 6 8 10 12
depth
92 97 102 107 112 117
0 50 100 150 200 250 300
Au (Company B)
dORE raw
dORE <1 mm
Company B


DJRC012
dF1
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depth
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0 100 200 300 400 500 600
Au (Company B)
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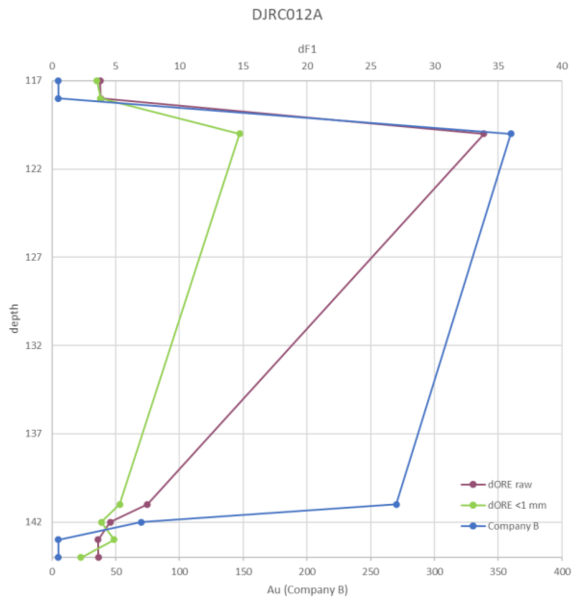
dORE <1 mm
Company B


DJRC012A
dF1
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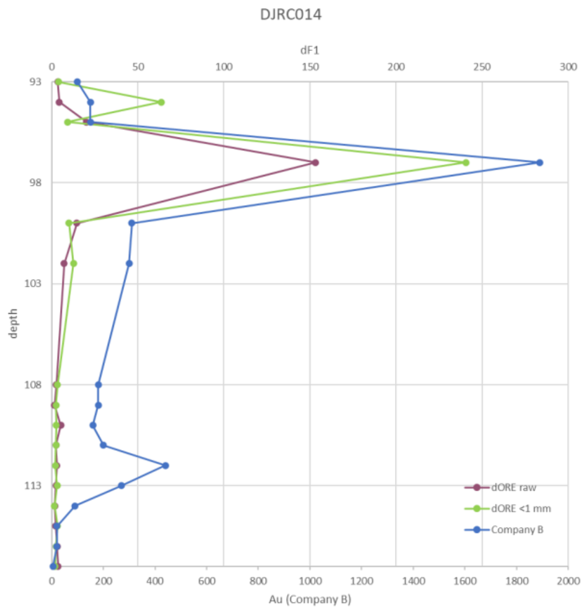
depth
117 122 127 132 137 142
0 50 100 150 200 250 300 350 400
Au (Company B)
dORE raw
dORE <1 mm
Company B


DJRC014
dF1
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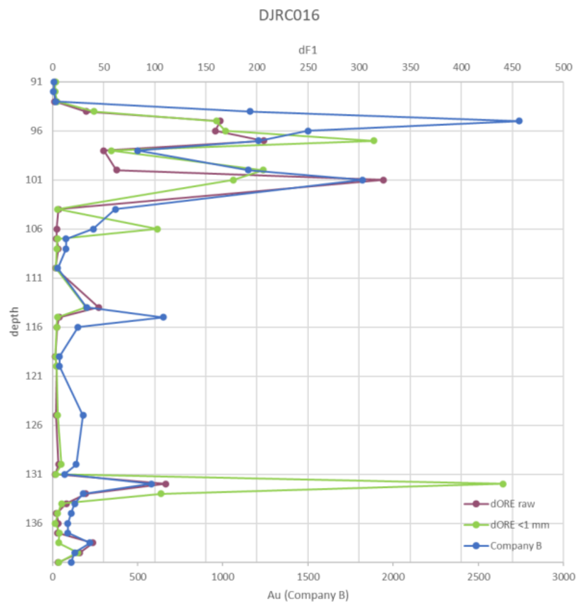
depth
93 98 103 108 113
0 200 400 600 800 1000 1200 1400 1600 1800 2000
Au (Company B)
dORE raw
dORE <1 mm
Company B


DJRC016
dF1
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depth
91 96 101 106 111 116 121 126 131 136
0 500 1000 1500 2000 2500 3000
Au (Company B)
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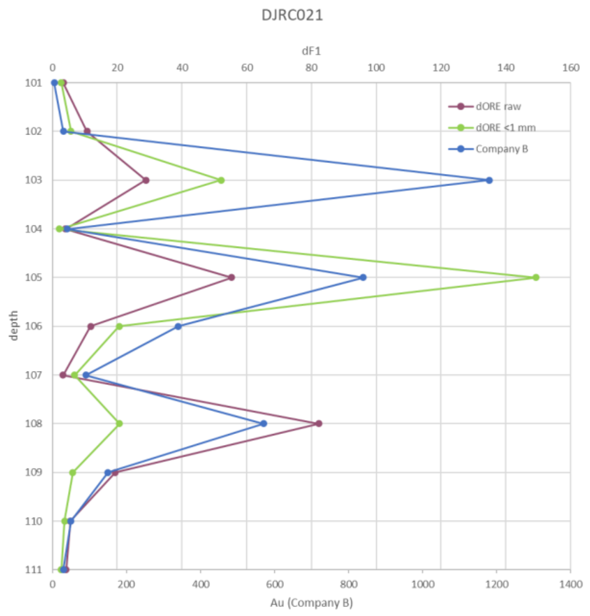
dORE <1 mm
Company B


DJRC021
dF1
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depth
101 102 103 104 105 106 107 108 109 110 111
0 200 400 600 800 1000 1200 1400
Au (Company B)
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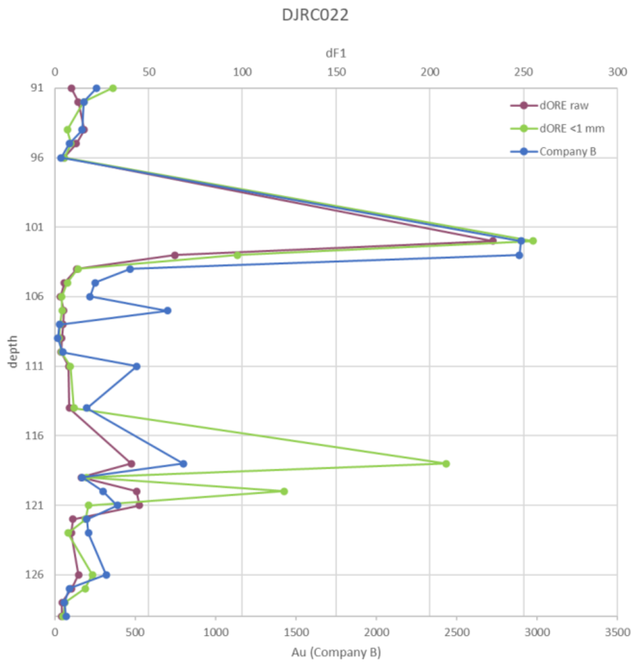
dORE <1 mm
Company B


DJRC022
dF1
0
50
100
150
200
250
300
91
96
101
106
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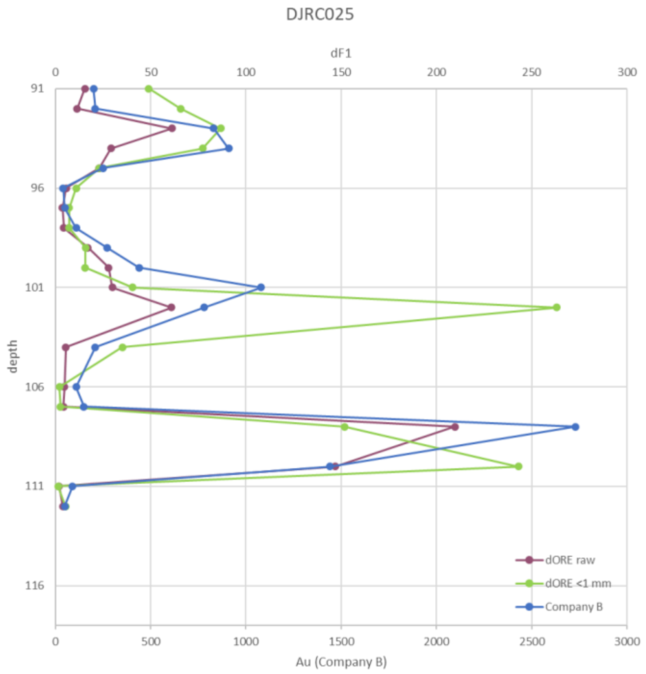
121
126
depth
dORE raw
dORE <1 mm
Company B
0
500
1000
1500
2000
2500
3000
3500
Au (Company B)


DJRC025
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100
150
200
250
300
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96
101
106
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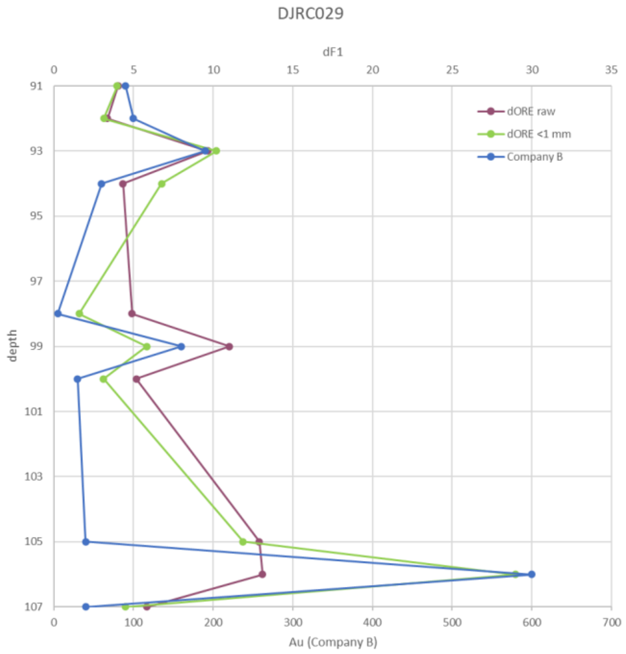
116
depth
dORE raw
dORE <1 mm
Company B
0
500
1000
1500
2000
2500
3000
Au (Company B)


DJRC029
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25
30
35
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93
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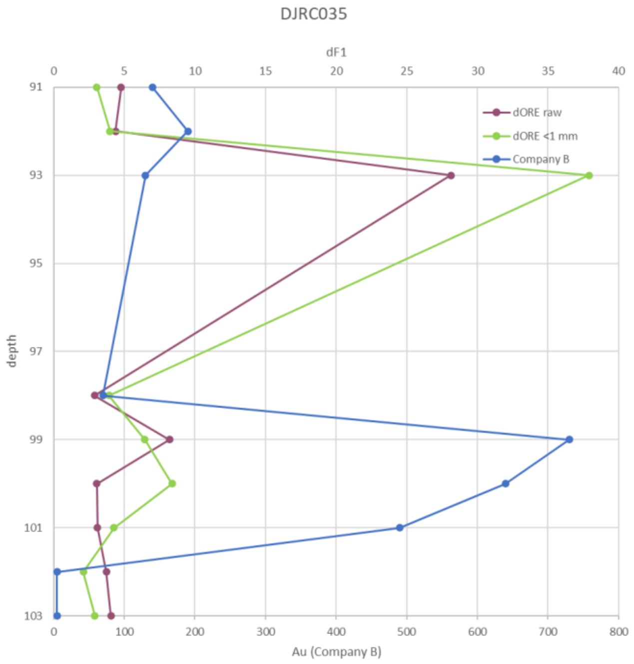
97
99
101
103
105
107
depth
dORE raw
dORE <1 mm
Company B
0
100
200
300
400
500
600
700
Au (Company B)


DJRC035
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97
99
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103
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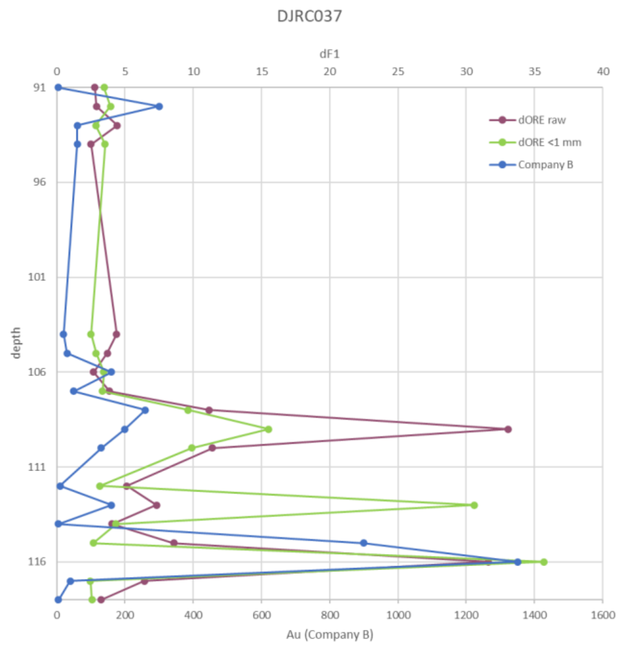
dORE <1 mm
Company B
0
100
200
300
400
500
600
700
800
Au (Company B)


DJRC037
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101
106
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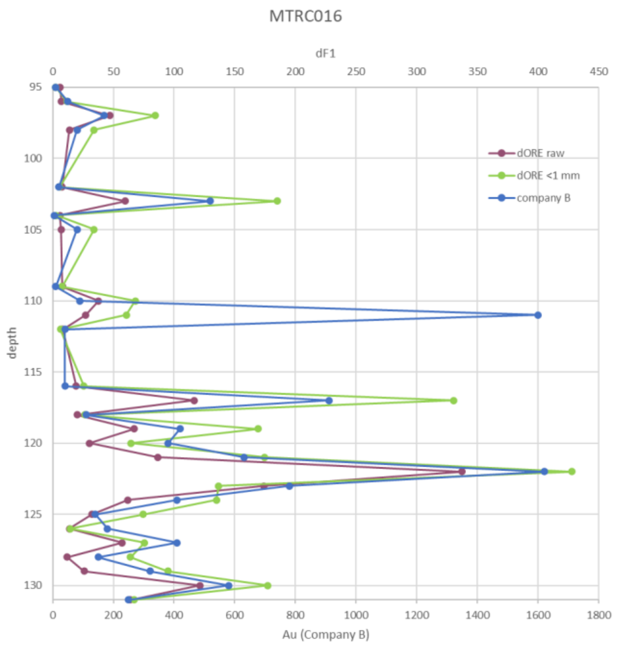
dORE <1 mm
Company B
0
200
400
600
800
1000
1200
1400
1600
Au (Company B)


MTRC016
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105
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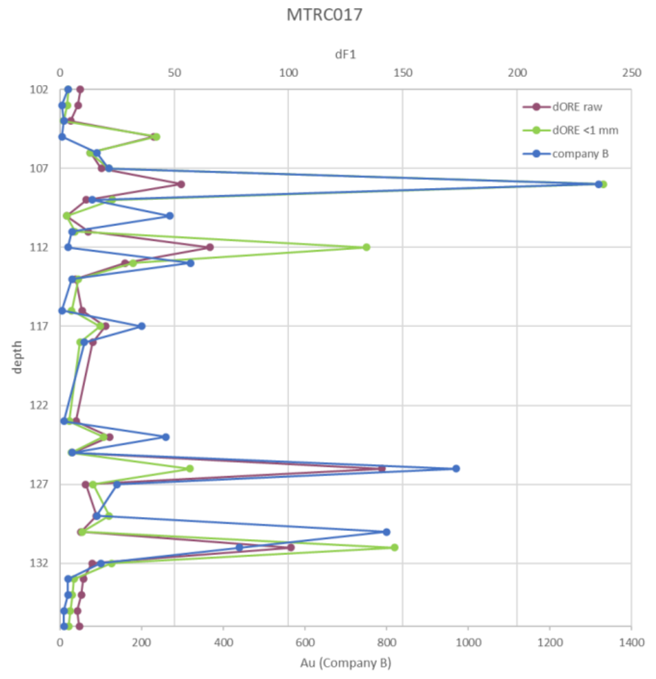
115
120
125
130
depth
dORE raw
dORE <1 mm
company B
0
200
400
600
800
1000
1200
1400
1600
1800
Au (Company B)


MTRC017
dF1
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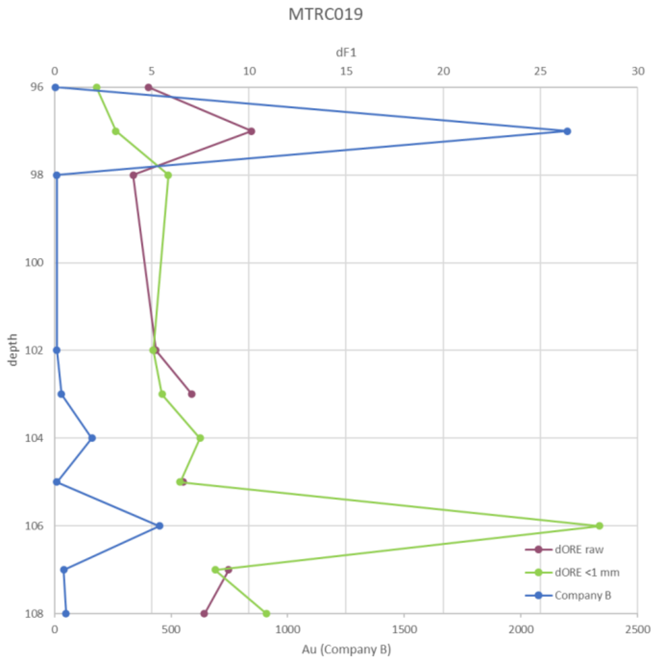
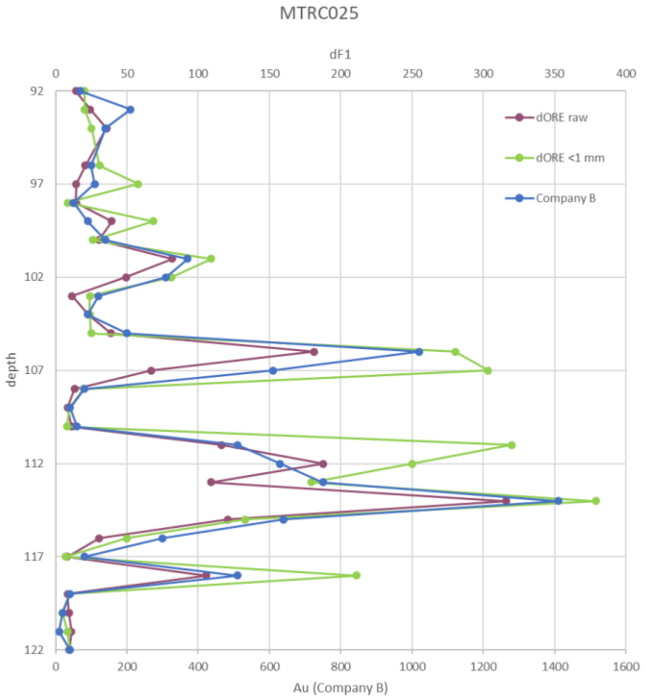
100
150
200
250
102
107
112
117
122
127
132
depth
dORE raw
dORE <1 mm
company B
0
200
400
600
800
1000
1200
1400
Au (Company B)


MTRC019
dF1
0
5
10
15
20
25
30
96
98
100
102
104
106
108
depth
dORE raw
dORE <1 mm
Company B
0
500
1000
1500
2000
2500
Au (Company B)


MTRC025
dF1
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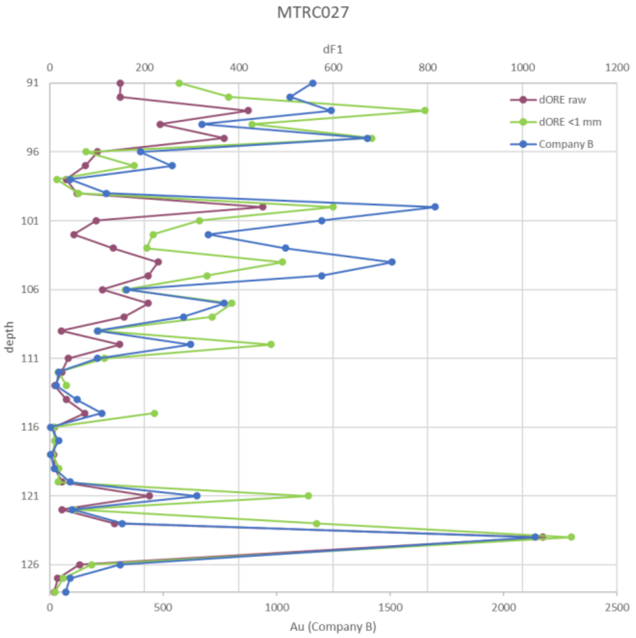
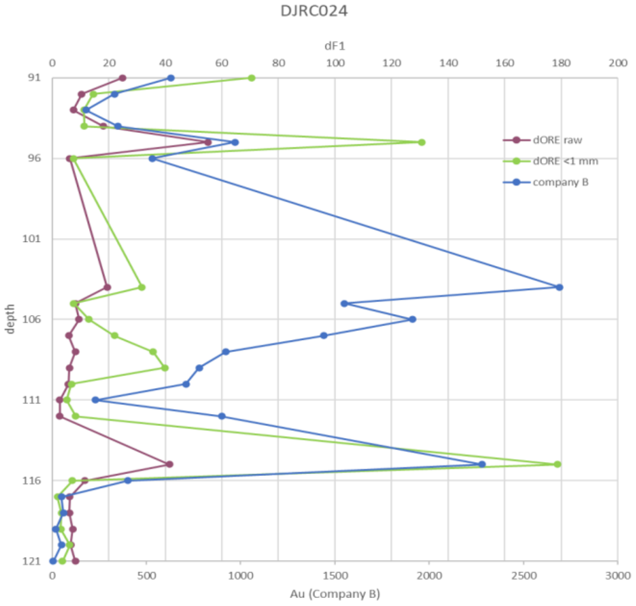
100
150
200
250
300
350
400
depth
92
97
102
107
112
117
122
dORE raw
dORE <1 mm
Company B
0
200
400
600
800
1000
1200
1400
1600
Au (Company B)


MTRC027
dF1
0
200
400
600
800
1000
1200
depth
91
96
101
106
111
116
121
126
dORE raw
dORE <1 mm
Company B
0
500
1000
1500
2000
2500
Au (Company B)


DJRC024
dF1
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60
80
100
120
140
160
180
200
depth
91
96
101
106
111
116
121
dORE raw
dORE <1 mm
company B
0
500
1000
1500
2000
2500
3000
Au (Company B)