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# Portable X-ray Fluorescence (pXRF) detectORE<sup>™</sup> Analysis for Au in Soils from the Archean Pilbara Craton, Western Australia

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#### INTRODUCTION

In this paper, we provide one of the first discussions of a new technology (detectORE<sup>TM</sup>) for preconcentrating Au in the field and measuring its concentration to low ppb levels using field-portable XRF (pXRF). This approach can provide geochemical data immediately during and/or following sample collection, meaning that exploration programs can be modified in close to real time, as opposed to the delays of weeks or even months when samples must be shipped to laboratories for analysis. Field-portable XRF is already a widely used approach to achieving real time geochemical data. A range of elements from Mg (z = 12) to U (z = 92) can be measured in seconds to minutes, to varying degrees of precision and accuracy, and varying lower levels of detection (LLD; Hall *et al.* 2014). Although continuing hardware and software development has increased both the number of elements that can be measured and the quality of data (e.g. Piercey and Devine 2014; Simandl *et al.* 2014), some target elements such as Li remain beyond the capabilities of pXRF. Other elements such as Au, can be measured in most crustal rocks and surficial materials. This issue is related to low count rates but can be exacerbated by spectral interference (Hall *et al.* 2014). In the absence of precise and accurate low-level Au data needed for effective mineral exploration, pXRF has relied on the analysis of pathfinder (e.g. chalcophile elements) and alteration-related elements such as Ca, K, Al, Rb, Fe, and Sr.

The vexing issue of obtaining pXRF Au analysis at suitably low levels has been recently addressed by detectORE<sup>™</sup> technology, a field-based preconcentration technique. Using this technology, Au in unconsolidated samples is released during partial leaching and concentrated on a collector device which is then analysed by pXRF (Lintern 2018; Lintern and Bolster 2018). The patented detectORE<sup>™</sup> technology was invented at Australia's National Science Agency, CSIRO, between 2015 and 2018. It has been exclusively licensed for commercialization to Portable PPB Pty Ltd.

The detectORE<sup>™</sup> - pXRF Au analytical methods discussed here were generated from a soil sampling program on exploration tenement E46/1026 in the eastern part of the Pilbara Craton of Western Australia (Fig. 1, 2). These data identified a broad zone of anomalism coincident with a regional structure (Blue Spec Fault; Fig. 3) hosting mineralization to the east of the tenement at Blue Spec and Gold Spec (Fig. 2). Based on the pXRF Au results from the soil samples, a drilling program in late 2022 confirmed the presence of Au mineralization, with intercepts including 6 m @ 40.15 g/t Au from 38 m depth, 7 m @ 5.42 g/t Au from 46 m depth, and 41 m @ 2.37 g/t Au from 32 m depth (Calidus Resources Ltd. 2023).

#### **REGIONAL GEOLOGY**

Calidus Resources Ltd ('Calidus') is exploring for Au on tenement E46/1026 with Gondwana Resources Ltd., who is the tenement holder. The tenement lies within siliciclastic metasedimentary rocks of the Mesoarchean Mosquito Creek Basin on the exposed eastern edge of the Pilbara Craton. The basin is about 60 km long from east to west and 30 km wide from north to south and overlies Paleoarchean granite–greenstone belts of the East Pilbara Terrane along a sheared unconformity (Fig. 1, 2; Hickman 1990; Van Kranendonk *et al.* 2004).

The lower part of the basin fill comprises medium- to coarse-grained, poorly sorted and chemically immature sandstone and conglomerate and komatiitic and tholeiitic basalt of the Coondamar Formation, which is overlain by thin-bedded sandstone, siltstone, and shale of the Mosquito Creek Formation (Hickman 2021; Fig. 2). Units of the Mosquito Creek Formation display graded bedding (as part of well-developed Bouma sequences), local cross-bedding, and sole marks (Hickman 2021). The Mosquito Creek Basin has been interpreted as an intramontane basin with coarse-clastic fan-delta marginal facies rimming an east-west basin centre with predominantly finer grained mass flow-dominated sediments (Nijman *et al.* 2010).

The age of the Coondamar Formation is poorly constrained, but that of the Mosquito Creek Formation is constrained by a maximum depositional age of 2926 ± 29 Ma (U-Pb detrital zircon date; Bagas *et al.* 2004) and a minimum deposi-



tional age provided by a Pb-Pb model age of 2905 ± 9 Ma from galena associated with Au mineralization (Thorpe *et al.* 1992).

All units of the Mosquito Creek Basin were overprinted by regional metamorphism of zeolite to lower greenschist facies and up to six deformation events (Farrell 2006) during the Mosquito Creek Orogeny (Hickman 2021). The most important events consist of major east–northeast-trending folds, thrusts/reverse faults, and a penetrative slaty cleavage, and later dextral movement along east- to northeast-trending fault zones. Thrust and reverse faults are north-verging in the north of the basin and south-verging in the south of the basin (Farrell 2005; Nijman *et al.* 2010) leading Nijman *et al.* (2010, p. 269) to suggest that the architecture of the folds and thrusts could be the result of "…transpressive push-up or positive flower structure."

Over almost 150 years, 42 t of Au or 50% of the total production from the Pilbara Craton has come from the Mosquito Creek Formation (Bagas *et al.* 2008). Mineralization is controlled by east–west fault zones roughly parallel to major fold axes. Two principal zones of mineralization, the Blue Spec and Middle Creek zones, are recognized (Fig. 2). The style of



Fig 2. The 1:500 000-scale map outlining the components of the Mosquito Creek Basin with interpreted structure. The purple dots show the locations of gold deposits and prospects, most of which lie on major faults of the Middle Creek Fault zone or Blue Spec zone. The Blue Spec and Gold Spec mines are shown, as well as the outline of tenement E46/1026 (data source: Department of Mines, Industry Regulation and Safety (www.dmirs.wa.gov.au)).



Fig. 3. A 1:100 000-scale surface geology and structures for tenement E46/1026 (data source: Department of Mines, Industry Regulation and Safety (www.dmirs.wa.gov.au)): a) Au (ppb) and b) Sb (ppm) in the <425 μm fraction of stream sediment samples. Symbols shown in orange and red have statistically anomalous element concentrations.

mineralization in both zones is similar, but the strong association of Sb with Au in the Blue Spec zone is not seen in the Middle Creek zone.

The historic Blue Spec mine (Fig. 2) has accounted for 2 t of Au, at an average grade of >18 g/t Au as well as concentrates containing 1500 t of Sb from >110 000 t of ore (Hickman 1983; Ferguson and Ruddock 2001). Mineralization at Blue Spec is hosted in a vertical to steeply south-dipping 15–20 m wide fault zone, characterized by quartz veining, and sporadically associated with carbonate alteration (Morrison 1978). Panning of some drill spoil on E46/1026 revealed free gold or gold liberated from weathered sulphides. DetectORE<sup>™</sup> and pXRF analysis of drill spoil produced Au (ppb) concen-

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trations that were approximately 50% of fire assay determinations, suggesting that some gold is hosted in silicates or quartz veins. In addition to gold, the quartz veins contain stibnite (Sb<sub>2</sub>S<sub>3</sub>), aurostibite (AuSb<sub>2</sub>), pyrite, pyrrhotite, with minor scheelite, arsenopyrite, marcasite, sphalerite, chalcopyrite, magnetite, calaverite (AuTe<sub>2</sub>), rickardite (Cu<sub>7</sub>Te<sub>5</sub>), and gudmundite (FeSbS). Bagas *et al.* (2008) argued that the mineral assemblage is indicative of relatively shallow-level, epizonal orogenic Au mineralization.

#### **EXPLORATION ON TENEMENT E46/1026**

In late 2020, Calidus commenced exploration on E46/1026 (Fig. 2) to search for Au on structures extending west from Blue Spec and Gold Spec as well as subparallel structures to the north of the fault zone, and folded rocks to the south.

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A stream sediment sampling program (76 sample sites) in the second half of 2021 (Calidus Resources Ltd. 2022), confirmed the potential of the Blue Spec Fault area, with maximum Au fire assay/AAS (30 g sample; SGS code FAM303) concentrations of 326 ppb (Fig. 3a). Although anomalous Au values associated with structures to the north of the Blue Spec Fault are less common, the potential for mineralization was indicated by anomalous Sb values of up to 51.7 ppm (Fig. 3b, four-acid digest and ICP-MS finish, SGS code ICM40Q). Samples from a small area of folded rocks to the south of the Blue Spec Fault returned few anomalous Sb or Au values. Multi-element analysis of stream sediment samples confirmed field observations that elevated Au concentrations are commonly associated with chalcophile enrichment, potassic alteration, carbonation or ferruginization (Calidus Resources Ltd. 2022).

Based on the results of the stream sediment chemistry, a follow up soil sampling program was carried out in mid-2022 (Fig. 4), focusing on the Blue Spec Fault area and the structures to the north, but including the less prospective area to the south. Samples were collected from sites on north-south transects spaced at either 100 m or 200 m, with samples spaced along lines at either 20 m or 50 m intervals. The transect and sample spacing reflects perceived prospectivity indicated by stream sediment chemistry. The soil sampling covers an area of approximately 6 km<sup>2</sup>.

Twelve hundred and thirty-two samples were analysed by pXRF using the detectORE<sup>™</sup> method (www.portableppb.com). Three hundred and sixty-eight samples were analysed for Au by 30 g fire assay/ICP-AES (method Au-ICP21) to a lower limit of detection (LLD) of 1 ppb, as well as for 48 additional elements by four-acid digest/ICP-MS (method ME-MS61) at ALS Laboratories (Perth, Western Australia). Of these 368 samples, 26 do not have corresponding pXRF data (Fig. 4, yellow symbols).

#### **GOLD ANALYSIS OF SOIL SAMPLES**

#### Sampling

Tenement E46/1026 is characterized by low, east-trending ridges separated by broad floodplains. Soils consist of disconnected patches of thin lithic-rich, sandy material in areas of outcrop, or sandy colluvium and less common sheetwash approaching drainage channels. Sample sites are largely confined to upland areas, where soil consists of moderately weathered coarse to fine sand with lithic fragments, and minor silt or clay. Due to the dominantly erosional landscape and immaturity of soils, organic matter is usually absent.

Soil sampling was carried out over about four weeks in June and July 2022. At each sample site, the top 5 cm of soil was removed to avoid any anthropomorphic contamination or windblown material. Where possible, each sample was collected at a uniform depth of 25 cm using a small metal entrenching shovel. For most sample sites, the selected <180  $\mu$ m grain size fraction was screened at the time of sampling. In some cases, the rough terrain slowed the sampling rate, and in these cases, approximately 3–4 kg of <2 mm material was collected, with further screening carried out at the field



camp. For each sample, approximately 400 g of the <180 µm (-80 mesh) fraction of soil was collected using a plastic sieve and collection pan. This amount is sufficient for detectORE<sup>™</sup> digestion (250 g), with the remainder (i.e. 150 g) for fire assay and multi-element geochemistry at a laboratory, if needed.

The screened material was placed in a 25 x 38 cm 125  $\mu$ m thick plastic bag sealed with a plastic cable tie. A unique site identification number was written on the bag with an indelible marker. In order to ensure that the site information was not rubbed off the bag during transit, the sample bag was placed in a 45 x 30 cm drawstring calico bag, also annotated with the unique site number. After completion of sampling, any residual material was removed from the sieve, collection pan and shovel using a stiff nylon brush. At designated sites, a second sample (site duplicate) was collected from within a 50 cm radius of the original sample using the same sampling protocol. At these sites, the letter "D" was appended to the site number written on the bag.

#### **pXRF** Analysis

The patented detectORE<sup>™</sup> technology involves a leach and collect process using a proprietary non-toxic, non-hazardous reagent called GLIX-20<sup>™</sup> and a collector device (CD). Sample preparation, carried out at the field camp at Marble Bar (Western Australia), involved partial digestion of approximately 250 g of unconsolidated sample material for 6 hours in a vessel containing 500 ml of GLIX-20<sup>™</sup>, and a collector device (CD) on which the released Au is collected. Following digestion, the CD was removed, rinsed and dried, then analysed by a pXRF device in detectORE<sup>™</sup> mode loaded with Portable PPB's pLIMS<sup>™</sup> software. Results are reported in detectORE<sup>™</sup> Units *continued on page 11* 





(dU). A detectORE<sup>™</sup> unit represents the µg of Au per kg of sample that is extracted from a sample; henceforth, the concentration of Au measured by pXRF in this study is discussed in terms of parts per billion (ppb).

#### Grain size selection orientation study

As the detectORE<sup>™</sup> technology involves a partial digest of the sample, it is likely that a better signal-to-noise ratio is achieved with finer grain sizes, as a greater surface area of the sample is exposed to the lixiviant (e.g. Leybourne and Rice 2013). Accordingly, 250 g of the <1 mm fraction (-18 mesh, i.e. coarse sand and finer) and a similar amount of the <150 m (-100 mesh; fine sand and finer) fractions were screened from each of 31 samples in the northeast of the sampling area for an orientation survey (Fig. 4, black square). As each of the 31 sample sites are only 50 m apart, and because the area is underlain by only turbiditic sediments, the effects of bedrock variations are reduced.

A bivariate plot of Au (ppb) according to grain size shows a high level of correlation ( $r^2 = 0.87$ ), but the Au concentration is the same or higher in the <150 µm fraction of all but four of the 31 samples (Fig. 5). Accordingly, the <150 µm fraction was selected for pXRF Au analysis in the remaining soil samples. Because a 150 µm screening cloth was not available for the main sampling program, each of the 1,232 soil samples was screened to -80 mesh (<180 µm), with sufficient screened material retained for laboratory analysis, if required.

#### Sample Digestion and Analysis

Soil samples were leached mostly in batches of 90 samples and the CDs analysed by pXRF at the field camp, using an Olympus Vanta M-series pXRF (serial number 801756). Leaching took place typically within 48 hours of sample collection. The CDs were analysed the day after digestion, which allowed sufficient time for them to dry; advice from PortablePPB is that any moisture retained by the CD can affect the Au analysis. In each batch, two reference materials were analysed. These reference materials, supplied by Portable PPB, contain a precise mass of a Certified Reference Material diluted with barren regolith material.

#### Site duplicates

A second soil sample—a site duplicate—was collected at 59 sites to test within-site homogeneity. A bivariate plot of Au (ppb) in the primary and site duplicate (Fig. 6a) plot around a 1:1 line, with  $r^2 = 0.886$ . There is more scatter at concentrations <20 ppb.

#### Collector device duplicate analysis

Fifty-two CDs were analysed twice, with a different part of the continued on page 12



Fig. 6. Plots showing a) pXRF Au (ppb) for parent and site duplicate samples (n = 59).  $r^2$  is 0.886 and b) duplicate analysis of pXRF Au (ppb) of CDs (n = 52) shown as first scan versus rescan.  $r^2$  is 0.99. The dashed line has a slope of 1 and a y intercept of 0.

CD exposed to the pXRF. The second CD measurement (i.e. rescan) was carried out immediately following the first scan. A bivariate plot (Fig. 6b) shows excellent agreement, with r<sup>2</sup> close to 1 and analyses plotting close to a 1:1 line. These results indicate Au is homogeneously distributed in the CD.

#### Fire assay analysis

The -80 mesh (<180  $\mu$ m) fraction of 368 samples was analysed for Au by fire assay/ICP-AES (method Au-ICP21; LLD = 1 ppb;) and for multielements by four-acid digest/ICP-MS (method ME-MS61) by ALS (Perth, WA). Prior to analysis, each sample was milled to nominal <75  $\mu$ m in a low-Cr steel ring mill (method PUL-31L). Soil samples were analysed along with reference materials (most of which have certified values for Au), sample duplicates (i.e. an analysis of a second aliquot of the sample pulp), and a silica pulp blank with a target range documented by ALS of <0.001–0.002 ppm (i.e. <1–2 ppb) for Au.

#### Reference materials

Four of the five reference materials have certified values for Au (GPP-14 (Geostats), KIP-19 (OREAS), TAZ-20 (OREAS), and OREAS 254b) ranging between 0.909 and 2.52 ppm. The fifth (AMIS0448; African Mineral Standards) has a provisional Au value of 1.31 ppm. Where reference materials have been analysed at least three times, the percent relative standard deviation (%RSD) has been calculated. It is <5 % in all cases, indicating acceptable precision. The certified or provisional Au value has been compared with the average Au concentration from at least three analyses using 100HARD (i.e.  $100 \times [(x1 - x2)/(x1 + x2)]$ ; Stanley and Lawie 2007). The 100HARD values lie between +5 and -5, indicating acceptable accuracy. Despite the acceptable precision and accuracy results, the data are disappointing in that they have limited application in terms of understanding the accuracy and precision of soil analysis, as only three soil samples have Au concentrations within the range of reference materials (i.e. 0.909–2.52 ppm, or 909–2520 ppb).

#### Duplicate and blank analyses

A second pulp aliquot for nine soil samples, with Au concentrations between 4 and 421 ppb, were randomly chosen by ALS for Au duplicate analysis. The Au concentration of the parent and duplicate samples were compared using 100HARD. Six of the parent samples have Au concentrations of more than 10x the LLD, so the effects of poorer precision close to the LLD are minimized. A 100HARD comparison for these pairs

produced values between -10 and +10, which is taken as acceptably precise. Two other parent-duplicate pairs with <10 ppb Au also produced 100HARD values in this range, showing that good precision persists to low concentration levels. Eleven analyses of a silica blank were carried out, with values of <1 ppb for all but one determination (1 ppb), indicating compliance with the target range.

#### COMPARISON OF detectORE<sup>™</sup> pXRF AND FIRE ASSAY Au DATA

A bivariate plot of Au according to method (Fig. 7, n = 342) shows a strong correlation ( $r^2 = 0.902$ ). The slope of a best fit line is 1.63, and assuming that FA/ICP-AES is a 'total' approach, these data show that detectORE<sup>TM</sup> - pXRF has

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determined approximately 60% of the available Au. Log-scale box and whisker plots for samples analysed by both pXRF and FA/ICP-AES (n = 342; Fig. 8a, b) show that both techniques have the same number of samples (35) with statistically anomalous Au concentrations (>144 ppb for pXRF and >258 ppb for FA/ICP-AES), and of these, 21 samples have extreme values (>228 ppb for pXRF and 408 ppb for FA/ICP-AES). For all pXRF data (n = 1232, Fig. 8c), 172 samples (14%) have anomalous Au concentrations >49 ppb, and of these 118 samples (10%) have extreme concentrations (i.e. >76 ppb). For FA/ICP-AES data (n = 368, Fig. 8d), a similar proportion of samples (10%) have anomalous Au concentrations (>245 ppb, n = 37), and of these, 23 have extreme concentrations (>386 ppb).

The spatial distribution of all samples symbolized according to Au concentration is shown in Figure 9. Samples with anomalous Au concentrations, as identified by the box and whisker plots (Fig.



Fig. 7. Plot of Au concentrations determined by FA/ICP-AES (ppb) versus Au by pXRF (ppb). The solid line is a calculated best fit, and the dashed line has a slope of 1 and a y intercept of 0.

8c,d), are shown as orange and red dots. The distribution of Au is similar for pXRF and FA/ICPMS, with a high proportion of anomalous samples on or near to the Blue Spec Fault. A few anomalous samples plot approximately 200 m north of, and subparallel to, the Blue Spec Fault, and other samples with anomalous Au concentrations coincide with sub-parallel faults in the central west of the tenement.

#### DISCUSSION

Both detectORE<sup>™</sup> - pXRF and FA/ICP-AES Au analysis of soil samples on tenement E46/1026 have confirmed the potential for Au mineralization on the Blue Spec Fault, and sub-parallel structures to the north. A considerable time saving was achieved using the detectORE<sup>™</sup> - pXRF approach compared to laboratory-based FA/ICP-AES analysis: sample collection and detectORE<sup>™</sup> pXRF analysis of the soil samples was completed on-site in 36 days, compared to about 90 days from the submission of samples for FA/ICP-AES analysis to receipt of results. Of note is that the initial drilling

program, involving 31 shallow reverse circulation holes with hole collars based on the detectORE<sup>TM</sup> - pXRF results, was completed by the time the FA/ICP-AES analyses were obtained from the laboratory. This drilling program returned significant intercepts in 25 holes, including 6 m @ 40.15 g/t Au (with 1 m @ 220.17 g/t) in one hole, and several holes containing multiple intercepts (Calidus Resources Ltd. 2023).

The receipt of detectORE<sup>™</sup> results within 48 hours of sample collection allowed for samples on infill lines and extensions to existing lines to be collected while the sampling crew was still in the field. Therefore, the cost of mobilizing and demobilizing the sampling crew months later for a second round of sampling was avoided.

Once an area of interest is identified for drilling, a Program of Work must be submitted to and approved by the Department of Mines, Regulation and Safety (DMIRS) and a heritage survey carried out to ensure that no Aboriginal cultural heritage is damaged. Given the demands on Aboriginal organizations for heritage surveys, it is quite likely that without the detectORE<sup>™</sup> results, a survey would have had to wait until after the hiatus of the summer wet season. The timeframe for conducting the earthworks and securing a drill rig would have meant that, if soil samples had been sent only to a laboratory, drilling probably would not have commenced until April or May 2023, a delay of five to six months.

![](_page_7_Figure_12.jpeg)

Fig. 8. Log-scale box and whisker plots for paired data (n = 342) and all data (pXRF n = 1232), FA/ICP-AES n = 368). The black and grey dots are, respectively, outlier and extreme values. Anomalous samples have Au (ppb) concentrations >[(1.5 \* IQR) +  $75^{th}$  percentile]. The division between outlier and extreme is at [( $3*(IQR) + 75^{th}$ percentile], as shown by a dotted line in each plot. The black square shows the median value. IQR is the interquartile range, calculated as the  $75^{th} - 25^{th}$  percentile values (Reimann et al. 2008).

![](_page_8_Figure_3.jpeg)

*Fig. 9. Dot maps showing Au (ppb) according to method for all samples: a) pXRF b) FA/ICP-AES. Classes are minimum to 25<sup>th</sup> percentile, 25 to 50<sup>th</sup> percentile (median), 50 to75<sup>th</sup> percentile, 75<sup>th</sup> percentile to outlier, outlier to extreme (orange dots), and greater than extreme to the maximum value (red dots).* 

Aside from the direct application to the Au exploration on tenement E46/1026, the pXRF data discussed here have highlighted some issues with the broader applicability of the detectORE<sup>TM</sup> pXRF approach in Au exploration, particularly the vectoring towards mineralization from areas of background. Of the 1,232 samples analysed by pXRF, 57% (i.e. 698 samples) returned <10 ppb Au. For these 698 samples, Au by FA/ICP-AES ranged from 2 to 67 ppb, with 59 samples having values of >10 ppb. At the reconnaissance stage of exploration, samples above 10 ppb by a method with an LLD of 1 ppb (i.e. at or above 10x the LLD) would attract interest (e.g. Noble *et al.* 2018), yet if only pXRF data for these samples are considered the data are unremarkable. To expand the application of the detectORE<sup>TM</sup> approach to the early stages of exploration, the significance of sub-40 ppb pXRF data requires clarification. For the data discussed here, this is difficult to assess, as QC data do not include analyses of certified reference materials and blanks, crucial for estimating precision

and accuracy and providing an estimate of a LLD. An impediment to routine generation of CRM data is the 250 g sample size required by detectORE<sup>™</sup>, making robust QA/QC programs a costly exercise.

A comparison of pXRF and FA/ICP-AES Au data must take into account the fundamental differences in the analytical approach. The detectORE<sup>™</sup> methodology is a partial digest, and although the composition of the GLIX lixiviant is not documented, it is fair to assume that it would be ineffective in liberating refractory Au — that is, Au encapsulated in silicates. In contrast, fire assay collection is an aggressive digestion approach, which is relatively unaffected by matrix issues (Hall and Pelchat 1994). The geology of the E46/1026 tenement consists of siliciclastic sedimentary rocks, with soils having undergone limited chemical weathering. As matrix effects can result in variable Au recovery (Chao 1984; Chao and Sanzalone 1992; Hall 1998), caution should be exercised in translating the level of Au recovery by detectORE<sup>™</sup> within and between different projects.

A further consideration in comparing pXRF and FA/ICP-AES data in this study is that the coarse grain size of samples digested in the detectORE<sup>TM</sup> process (<180 µm) relative to the nominal <75 µm grain size for fire assay Au collection. The grain size orientation data discussed here indicate that the detectORE<sup>TM</sup> approach benefits from finer grain size samples, where presumably more Au is exposed to the lixiviant. However, the 250 g sample size currently required by detectORE<sup>TM</sup> also has implications for using a fine grain size fraction, such as silt and clay, to optimes the signal-to-noise ratio. Soils on tenement E46/1026 are lithic-rich and typically

sand dominated, with only a few percent silt and clay. To extract sufficient mass of silt and clay for analysis would require collection of an estimated 4–5 kg sample of <180  $\mu$ m material at each site, followed by screening to <63  $\mu$ m, introducing a significant time and cost factor.

#### CONCLUSIONS

The detectORE<sup>TM</sup> - pXRF approach to the determination of ppb-level Au concentrations in soils from the Calidus tenement in the eastern part of the Pilbara Craton of Western Australia has been evaluated using comparative FA/ICP-AES data. In terms of validating the results of stream sediment chemistry and identifying areas of anomalous Au concentrations, both detectORE<sup>TM</sup> - pXRF and FA/ICP-AES data are valid approaches, although Au determined by detectORE<sup>TM</sup> in siliciclastic rocks on E46/1026 is approximately 60% of that determined by fire assay/ICP. However, detectORE<sup>TM</sup> - pXRF has the major advantage of substantial time savings, providing the opportunity to react to results as they are generated in the field. The work on tenement E46/1026 puts pXRF in a strong position to directly evaluate not only Au, but Au pathfinder elements (e.g. Sb, As) and elements related to alteration (Ca, K, Al, Rb, Fe).

The detectORE<sup>™</sup> approach could have a wider applicability in the earlier stages of mineral exploration if the significance of Au reported at levels <40 ppb can be better quantified. A current drawback of the detectORE<sup>™</sup> approach to tackling this issue is the large sample size required for leaching (250 g), which makes routine analysis of low-Au CRMs costly. This sample size also has cost and time implications for exploration where large volumes of material must be screened in order to produce sufficient fine-grained material appropriate to a partial digestion approach.

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#### REFERENCES

Bagas, L., Farrell, T.R. and Nelson, D.R. 2004. The age and provenance of the Mosquito Creek Formation. Western Australia Geological Survey, Annual Review 2003-4, 62–70.\*

- Bagas, L., Bierlein, F.P., Bodorkos, S. and Nelson, D.R. 2008. Tectonic setting, evolution and orogenic gold potential of the late Mesoarchean Mosquito Creek Basin, North Pilbara Craton, Western Australia. Precambrian Research, 160, 227–244.
- Calidus Resources Ltd, Report to the Australian Stock Exchange March 21 2022. Strong exploration results highlight growth potential of Blue Spec Project. 15 p. www.asx.com.au
- Calidus Resources Ltd, Report to the Australian Stock Exchange 18 January 2023. High-grade zone in Felix discovery at Blue Spec Project. 16 p. www.asx.com.au
- Chao, T.T. 1984. Use of partial dissolution techniques in geochemical exploration. Journal of Geochemical Exploration, 20, 101–135.
- Chao, T.T. and Sanzalone, R.F. 1992. Decomposition techniques. Journal of Geochemical Exploration, 44, 65–106.
- Farrell, T.R. 2005. Eastern Creek, W.A. (Version 2.0). Western Australia Geological Survey, 1:100 000 Geological Series Sheet 3054.
- Farrell, T.R. 2006. Geology of the Eastern Creek 1:100 000 sheet, Western Australia. Western Australia Geological Survey, 1:100 000 Geological Series Explanatory Notes, 33 p.
- Ferguson, K.M. and Ruddock I. 2001. Mineral occurrences and exploration potential of the east Pilbara. Western Australia Geological Survey, Report 81, 114 p.\*
- Hall, G.E.M. 1998. Analytical perspective on trace element species of interest in exploration. Journal of Geochemical Exploration, 61, 1–19.
- Hall, G.E.M. and Pelchat, J.C. 1994. Analysis of geological materials for gold, platinum and palladium at low ppb levels by fire assay-ICP mass spectrometry. Chemical Geology, 115, 61–72.
- Hall, G.E.M., Bonham-Carter, G.F. and Buchar, A. 2014. Evaluation of portable X-ray fluorescence (pXRF) in exploration and mining: Part 1, control reference materials. Geochemistry: Exploration, Environment, Analysis, 14, 99–123.
- Hickman, A.H. 1983. Geology of the Pilbara Block and its environs. Western Australia Geological Survey Bulletin, 127, 268 p.\*
- Hickman, A.H. 1990. Geology of the Pilbara Craton. In: Ho S.E., Glover J.E., Myers J.S. and Muhling J.R. (eds), Third International Archaean Symposium Excursion Guidebook No. 5: Pilbara and Hamersley Basins. University of Western Australia, Geology Department and University Extension, Publication 21, 2–13.
- Hickman, A.H. 2021. East Pilbara Craton: a record of one billion years in the growth of Archean continental crust. Western Australia Geological Survey Report 143, 187 p.\*
- Leybourne, M.I. and Rice, S. 2013. Determination of gold in soils and sediments by fire assay or aqua regia digestion: choosing the optimal method. Explore, 158, 1–10.
- Lintern, M. 2018. Field analysis of low ppb gold using pXRF and new detectORE technology. ASEG Extended Abstracts, 2018:1, 1–5.
- Lintern, M. and Bolster, S. 2018. Low level field analysis of gold using pXRF. 2018 International Applied Geochemistry Symposium Abstracts, No 1609.
- Morrison, R.J. 1978. The Blue Spec gold-antimony mine. Annual Report for the Year 1978, Western Australia Geological Survey, 74–79.\*
- Nijman, W., Clevis, Q. and de Vries, S.T. 2010. The waning stages of a greenstone belt: The Mesoarchean Mosquito Creek Basin of the East Pilbara Craton, Western Australia. Precambrian Research, 180, 251–271. *continued on page 17*

- Noble, R.R.P., Morris, P.A., Anand, R.R., Lau, I.C. and Pinchand, G.T. 2018. Application of ultrafine soil extraction and analysis to mineral exploration. Geochemistry: Exploration, Environment, Analysis, 20, 129–154.
- Piercey, S.J. and Devine, M.C. 2014. Analysis of powdered reference materials and known samples with a benchtop, field portable X-ray fluorescence (pXRF) spectrometer: evaluation of performance and potential applications for exploration lithogeochemistry. Geochemistry: Exploration, Environment, Analysis, 14, 139–148.
- Reimann, C., Filzmoser, P., Garrett, R.G. and Dutter, R. 2008. Statistical Data Analysis Explained. John Wiley and Sons, Chichester. 343 p.
- Simandl, G.J., Fajber, R. and Paradis, S. 2014. Portable x-ray fluorescence in the assessment of rare earth element-enriched sedimentary phosphate deposits. Geochemistry: Exploration, Environment, Analysis, 14, 161–169.
- Stanley, C.R. and Lawie, D. 2007. Average relative error in geochemical determinations: clarification, calculation and a plea for consistency. Exploration and Mining Geology, 16, 267–275.
- Thorpe, R.I., Hickman, A.H., Davis, D.W., Mortensen, J.K. and Trendall, A.F. 1992. Constraints to models for Pb evolution from precise U/Pb geochronology for the Marble Bar region, Pilbara Craton, Western Australia. *In:* Glover J.E. and Ho S.E. (eds), The Archaean Terrains: Processes and Metallogeny. Geology Department and University Extension, The University of Western Australia, Publication 22, 395–406.
- Van Kranendonk, M.J., Smithies, R.H., Hickman, A.H., Bagas, L., Williams, I.R. and Farrell, T.R. 2004. Event stratigraphy applied to 700 million years of Archean crustal evolution, Pilbara Craton, Western Australia. Western Australia Geological Survey, Annual Review 2003-2004, 49–61.\*

\*Available to download free of charge from the eBookshop at www.dmirs.wa.gov.au.

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