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REPORT  
190

# MRIWA REPORT NO. 462 MULTI-SCALED NEAR SURFACE EXPLORATION USING ULTRAFINE SOILS

by R Noble, I Lau, R Anand and T Pinchand



Geological Survey of Western Australia



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**PERTH 2018**



**Geological Survey of**  
**Western Australia**

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**About this publication**

This Report presents the results of the Minerals Research Institute of Western Australia (MRIWA) Project M462 'Multi-scaled near surface exploration using ultrafine soils'. The Geological Survey and Resource Strategy Division (GSRSD) is releasing the Report as part of its Report Series to ensure a wider distribution for the results of the Ultrafine+ method that demonstrates the geochemical analysis of <math><2 \mu\text{m}</math> particle size fraction of soils and sediments, and its application for mineral system signatures. Although GSRSD provided support to the project, the scientific content of the Report, and the drafting of figures, has been the responsibility MRIWA. No editing has been undertaken by GSRSD.



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**Cover photograph:** Ferricrete capped mesas, south of Halls Creek, Western Australia

MRIWA REPORT NO. 462

# Multi-scaled near surface exploration using ultrafine soils

MRIWA Project No. M462 - 2016 to 2018

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Authors

Ryan Noble, Ian Lau, Ravi Anand, Tenten Pinchand

October 2018



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Western Australia**



REPORT NO. 462

## Multi-scaled near surface exploration using ultrafine soils

Results of research carried out as MRIWA Project M462

at CSIRO Mineral Resources, Kensington, Western Australia

by

Ryan Noble, Ian Lau, Ravi Anand, Tenten Pinchand

October 2018

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# Executive Summary

The M462 Project was conceived to develop and test a new analytical workflow to separate the <2 µm soil and sediment fractions for multielement analysis along with other, commonly not utilised physico-chemical parameters that should aid exploration. The project delivered the method, workflow, and commercialised platform (UltraFine+ certified trademark pending); and demonstrated our success in experiments, orientation field surveys and new regional geochemical map products for Western Australia. This report condenses the two-year effort into two journal-style papers.

Part 1 - Method Optimisation Report *Refining fine fraction soil extraction and analysis for mineral exploration* by Noble, R.R.P., Lau, I.C., Anand, R.R. and Pinchand, G.T.

Part 2 – Site Studies and Regional Maps *Application of ultra fine fraction soil extraction and analysis for mineral exploration* by Noble, R.R.P., Morris, P.A., Anand, R.R., Lau, I.C. and Pinchand, G.T.

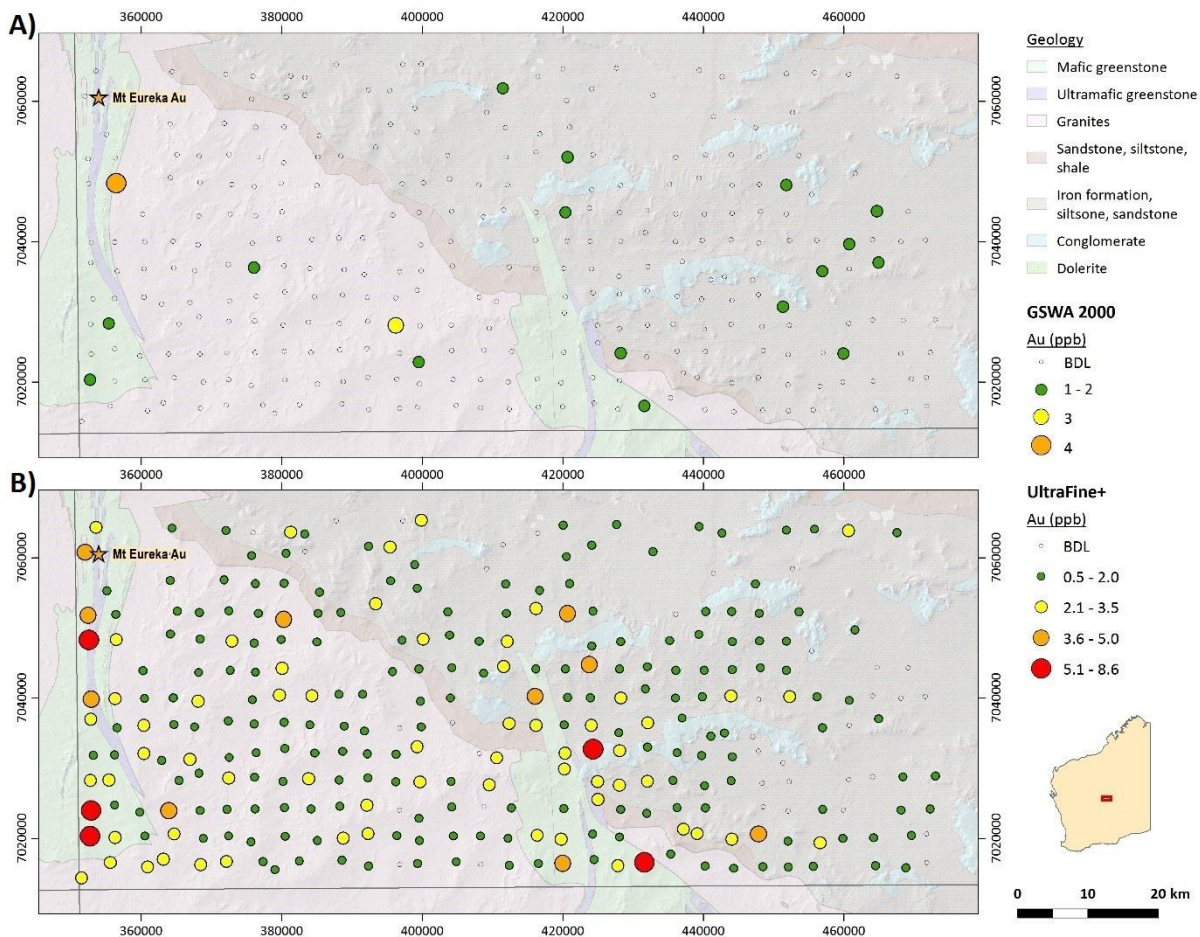
Additional data products and the public data release for the regional maps are linked in the Appendices for cloud data delivery to sponsors; and the data and report will also be hosted in GSWA publications repository and GeoView.

Greenfields exploration in Australia is in decline, and the technical challenge of exploring in deeply weathered and covered regions has not been fully addressed; yet exploration success in these areas is critical to the future economy. Commonly, soil sampling is paired with acid digestion and multi-element measurement. This established approach has not changed significantly over the past 30 years: that is, digest the <250 µm soil fraction and analyse the solution for elemental concentrations. In transported cover, the mobile element signature is contained in the smallest size fractions, so we tested the clay size fraction (<2 µm) as an improved sample medium for mineral exploration.

A series of experiments were conducted to demonstrate the value of using <2 µm fractions for exploration geochemistry. Twenty seven bulk reference soils were collected in the vicinity of known mineral deposits (including background areas) that reflect the common soil types of Western Australia. By analysing fine fractions (<2 µm) we produced reproducible, reliable results, with higher concentrations than from the <250 µm fraction (average increase of 100-250%). Key benefits were the reduction of nugget effects (in Au) and the challenges with detection limits in materials that are dominated by quartz sand. Testing sub-micron fractions showed that although <0.2 µm fraction was slightly different to the <2 µm and <0.75 µm fractions there was not significant additional value. The <2 µm fraction represent the most effective and cost efficient sample medium to use. The overall method development showed that ultrasonics were not required, a dispersant was critical for solid recovery and that Na-hexametaphosphate (technical or laboratory grade) was the most effective dispersant. The developed method proved the use of a small weight for analysis was effective (0.2 g) and microwave assisted aqua regia was the best analytical method for Au detection. Our research shows obvious benefits in using fine fractions for Au. Copper and Zn were consistently and abundantly extracted from the fine particle size fraction.

We applied the UltraFine+ workflow to a number of small orientation site studies in Western Australia, and reprocessed archived regional soil samples from the Geological Survey of Western Australia to test the method to improve exploration targeting. The orientation program involved ~200 samples from the Leonora and Sir Samuel 1:250k map sheets, an area that hosts known major Au and base metal deposits. We then applied this approach to the Kingston 250k map sheet area, analysing a further 300 samples in a region on the Yilgarn Craton margins that is essentially greenfields. There has been little exploration in the region, and the original geochemical survey data was heavily censored due to the dominance of transported regolith dominated by quartz-rich sand. Of most relevance, the study revealed a marked decrease in censored results for Au (~67% to 10% below detection limit) using historic samples, and re-assaying them enabled us to produce a new geochemistry map of the Kingston 1:250,000 map sheet.





**Gold (ppb) in soils in the Kingston 1:250 000 map sheet. A) Original GSWA data with only a few detectable Au values, B) the new results of the Ultrafine+ method developed in this MRIWA project using the same samples, clearly showing the vast improvement in Au information. Mt Eureka is the only known small Au deposit in the region (mined in the 1930s). Geology is generalised and based on the data from GSWA (2014).**

The new maps show geochemistry, some example indices for mineral exploration, and lithology indicators through cover, as well as map products of new interpretations using the additional spectral mineralogy proxies and particle size measurements. Adding spectral mineralogy, particle size and other physico-chemical parameters to this style of mapping is valuable, but is not commonly done, and is certainly not integrated, currently.

The application of the <2 µm particle size separation and the UltraFine+ workflow demonstrate the importance of the additional value from (re-)assaying regional soil and sediment samples to generate new targets and improve regional geochemical maps (see figure above). This is an exercise that can be applied to new greenfields surveys, and when exploration budgets are lean, applied to abundant, historically collected samples.

The developed workflow was transferred to the commercial laboratory partner (Lab West Pty Ltd) and is now being used by a number of sponsors of the project. UltraFine+ will now be available to all industry and commercial laboratories. The commercialisation of the UltraFine+ workflow (logo below) was nearly as important as the development and proof of the technique. The technique was designed to be robust for industry and streamlined enough to be economically viable.

The project has completed all its deliverables. Over the course of the project, we have determined that a number of additional developments will ensure this process is the world leader for providing better high-quality data in a useable format for future explorers. The next iteration of this workflow should improve the UltraFine+ method, particularly estimating organic C as well as building algorithms and machine learning to cloud-process the various data streams; and this should be part of the service from commercial laboratories in the future. We envisage a second project of similar size will realise the full potential of the workflow

developed in this project over the next few years, and lead to a subsequent improvement to the WA greenfields exploration success rate.



# 1 M462 Deliverable: Part 1 - Method Optimisation Report

## 1.1.1 M462 Deliverable: Part 1 - Method Optimisation Report

### Refining fine fraction soil extraction and analysis for mineral exploration

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### 1.1.2 Abstract:

Commonly, mineral exploration soil and regolith samples are sieved to <250 µm or <180 µm to remove larger gravels and coarse sand and later pulverized for analysis, but no other refinement is used with the soil. In transported cover, the soils that host the mobile element signature are the smallest size fractions, so we should look to concentrate the clay size fraction (<2 µm) as an improved sample medium. However, industry is unlikely to fully adopt these recommend changes without proof of the benefit of refining the size fraction. A series of experiments were conducted to demonstrate the value of using <2 µm fractions for exploration geochemistry. Twenty seven bulk reference soils were collected in the vicinity of known mineral deposits (including background areas) that reflect the common soil types of Western Australia. A selection of these soils was used in replicate testing to assess differences in particle size, sample weight, dispersants and how this relates to the geochemistry with intent to optimise the extraction and speed of ultra-fine fraction recovery. Using multiple analysis of variance tests the sub-micron fractions showed that although the <0.2 µm fraction was slightly different to the <2 µm and <0.75 µm fraction, there was not additional value in the extra effort of extracting these sub-micron fractions, and the <2 µm favoured. The overall method development showed ultrasonics and rinsing were not required for Au analysis, no Au is lost in solution and only a small fraction of the overall Cu (0.5%) was lost. Using a dispersant was critical for solid recovery and that Na-hexametaphosphate (technical or laboratory grade) was found to be the most effective reagent for dispersing the sample material. The developed method proved that the use of a small sample weight for analysis of Au was effective (0.2 g) with no significant differences for assays using 0.1 g, 0.2 g, 0.5 g, 1 g and 4g (p <0.05) with a microwave assisted aqua regia digestion being the best analytical method for Au recovery compared to certified reference materials. In addition to the geochemistry, the spectral reflectance variation was investigated with respect to particle size and the integration of multiple parameters was used to generate a new workflow called UltraFine+. This workflow provides much more data than previous commercial analytical offerings (40 elements, spectral mineralogy, particle size distribution, pH, EC, specific surface area) and offers a refined data set to improve mineral exploration using soil and regolith samples.

**KEYWORDS:** *spectral mineralogy, regolith, ultrafine+, particle size, geochemistry*

## 2 Introduction

Greenfields exploration in Australia is in decline and directly hindered by the transported overburden that blankets the underlying rock and masks ore bodies. The technical challenge of exploring in such deeply weathered and covered regions has not been fully addressed, and yet exploration success in these areas is critical to the future economy. Commonly near surface soil sampling is paired with acid digestions and multi-element measurement. This “standard” approach has not significantly changed over the past 30 years that is, digest the <250  $\mu\text{m}$  soils and analyse the solution for elemental concentrations. The change to the technology improving to analyse the solutions has evolved, e.g., AA to ICP-MS, improving the detection limits and number of elements detected and occasionally the extraction solutions (partial digestions) are varied with some success (Chao, 1984; Mann *et al.*, 1998; Williams & Gunn, 2002; Dalrymple *et al.*, 2005; Noble & Stanley, 2009).

Commonly, exploration soil samples are sieved to <250  $\mu\text{m}$  or <180  $\mu\text{m}$  to remove larger gravels and coarse sand and later pulverized for analysis, but no other refinements are undertaken on the soil samples. Firstly, why does the exploration industry and analytical service providers (commercial laboratories) use this soil fraction? It is because the standard chemical analysis for soils (in agriculture) was deemed to use this fraction when a lot of these techniques were derived (Jackson, 1958). Subsequently analytical laboratories routinely used these methods and as exploration geochemistry developed, laboratories that had used this fraction for agriculture shifted focus/clients and used the same methods for mineral exploration. Secondly, is this the appropriate size fraction for mineral exploration? In residual terrains the <250  $\mu\text{m}$  soils may be effective because all materials present are related to the local geology, but in covered terrains, where the origin of the surface materials are transported, materials do not represent the local underlying rocks and so only a mobile dispersion signature of target and pathfinder elements should be studied as demonstrated by Anand *et al.* (2014, 2015), particularly in pit experiment studies. These mobile elements of interest are adsorbed onto soil exchange sites and not part of the matrix. The size of the soil particles is inversely related to the surface area and the associated exchange capacity of the particle, i.e., smaller particles have the greatest surface area and ability to adsorb other ions in the soil solution (Table 1). Clays, organic compounds and various oxides/oxyhydroxides dominate this fine fraction and have a greater exchange capacity than the larger minerals like quartz (Table 2; Hawkes and Web, 1962; Hall, 1998). Hence, the soils that host the mobile element signature are the smallest size fractions, so we would argue that for exploration purposes we should look to concentrate the clay size fraction (<2  $\mu\text{m}$ ) as an improved sample medium. However, industry are unlikely to fully adopt these recommend changes without proof of the benefit of refining the size fraction and that there is benefit by simplifying the process. This paper shows a series of experiments to demonstrate the value of using <2  $\mu\text{m}$  fractions for mineral exploration geochemistry.

**Table 1. Approximate particle size, surface area and cation exchange capacity (CEC) of soil. Note CEC of sand and silts is likely due to coatings of finer particles and the values are influenced greatly by clay type and pH.**

Size fraction	Diameter ( $\mu\text{m}$ )	Surface area ( $\text{m}^2/\text{g}$ )	CEC ( $\text{cmol}^+/\text{kg}$ )
Sand	50-2000	0.04	1-4
Silt	2-50	0.1	1-10
Clays	< 2	5 - 800	3-150

**Table 2. Typical cation exchange values of important soil minerals that constitute much of the soil fine fraction <2 µm. Values reported are estimates (at pH =7) from McQueen 2008 and Rowell 1993.**

Clay mineral	CEC (cmol+/kg)	Other key minerals	CEC (cmol+/kg)
Kaolinite	3-15*	Goethite	<100
Illite	10-40**	Hematite	<100
Smectite	80-150	Ferrihydrite	<500
Chlorite	10-40	Birnessite	240
Vermiculite	100-150	Organic matter	130-500

\* CEC increases with decreasing crystallinity

\*\* Pure illite CEC is <5, but increases with increasing interstratified smectite

Microparticulate (<2 µm) and even nanoparticulate (<0.2 µm) separations are performed for clay analysis in engineering and agriculture related pursuits. This technique is slow and laborious and provides the weight of each fraction and sometimes the mineralogy. The related geochemistry is not tested. Small Au particles (<2 µm) in regolith have been observed using scanning electron microscopy (Hough *et al.*, 2008), but the amount of micro and nanoparticulate Au cannot be quantified by this method. Silt and clay fraction (c. <50 µm) separation and geochemical analysis has been used for geochemical exploration in several studies, with the < 75, <63 or <50 µm size fractions used to enhance target and pathfinder element contents (Anand *et al.*, 2014; Morris, 2013, Scott & van Riel, 1999; Arne & MacFarlane, 2014; Baker, 2015). Although intermittently tested by industry and researchers it is not a routine method as the potential benefits and how to use the data is not as well understood or familiar as general <250 µm analyses. For this reason, fine fractions (<2 µm) for mineral exploration is a specialised application that has not been commercially available.

A demonstration study by Noble *et al.* (2013) showed the separation and quantification of micro and nanoparticulate Au in 14 transported soils from the East Wongatha region of Western Australia where a regional regolith geochemical survey was carried out by the Geological Survey of Western Australia (Morris, 2013). The results revealed that the Au was highly anomalous in the micro and nanoparticulate size range where it was close to, or below, detection limits (1 ppb) in the bulk material. The importance of improving detection limits for Au to avoid so many results below detection has been emphasized (Leybourne and Rice, 2013). Quartz (SiO<sub>2</sub>), with practically no exchange capacity and makes up much of the sand fraction (Table 1), is the major mineral in most exploration soil samples, and is a major dilutant to the trace element chemistry of interest. Another demonstrative study published by Anand *et al.* (2014) shows the <53 µm had much greater concentrations of Au over the Moolart Well deposit whereas the commonly used <250 µm fraction was ineffective.

In this paper, we assess differences in particle size and how this relates to the geochemistry with intent to optimise the extraction and speed of fine fraction recovery. In addition to the geochemistry, the spectral reflectance variation is investigated with respect to particle size and the integration of multiple parameters is used to generate a new workflow called UltraFine+.

# 3 Methods

## 3.1.1 Soil samples

Twenty seven bulk reference soils were collected from ten prominent exploration and mining regions in Western Australia to provide a background set of samples to test various separation and analysis techniques. Figure 1 shows the location of these sites. Table 3 shows key features of the soil types/target commodity. At each reference site a number of samples from the area (Table 3) were combined. These soils were in background areas as well as above mineralisation. Samples were homogenized to provide c. 5 kg of material to test.



Figure 1. The location of the bulk reference soil samples used in this study.

**Table 3. Bulk reference soils and key parameters. n = number of samples that were combined to represent that site. \* a single sample collected as a 10 kg composite from an area of ~1 m. Note: clay and silt % underestimated, refer to main text.**

Deposit	Region	Target commodity	Soil colour	%C	%OC	CEC cmol+/kg	EC $\mu$ S/cm	pH	%Clay	%Silt	%Sand	%Gravel	n	Lat	Long
Gruyere	Yamarna	Au	5YR 4/4	0.2	0.2	0.64	39	5.74	1.8	1.1	97.1	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	7.5YR 3/2	0.3	0.3	1.47	99	6.83	1.2	1.0	97.8	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	7.5YR 3/4	0.2	0.2	0.86	44	5.92	1.4	1.1	97.5	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	5YR 4/4	0.2	0.2	0.79	52	5.56	1.2	0.8	98.0	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	-	0.2	0.2	0.64	66	5.63	1.3	0.9	97.8	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	5YR 4/4	0.2	0.2	0.54	86	5.48	1.5	0.7	97.8	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	7.5YR 4/2	0.2	0.2	0.75	76	6.29	1.3	0.6	98.2	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	5YR 3/2	0.2	0.2	0.78	47	6.1	1.2	0.8	98.1	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	5YR 3/4	0.2	0.2	0.54	28	5.79	1.4	0.5	98.1	0.0	1*	-27.98	123.84
Gruyere	Yamarna	Au	5YR 3/4	0.2	0.2	0.61	35	5.8	1.3	0.7	98.0	0.0	1*	-27.98	123.84
Calibre	Paterson	Cu-Au	5YR 4/4	0.2	0.2	0.54	47.	5.42	0.7	1.4	97.7	0.1	15	-20.77	122.20
Calibre	Paterson	Cu-Au	5YR 4/4	0.1	0.1	0.44	44	5.43	0.6	1.5	97.8	0.0	15	-20.77	122.20
Bentley	NE Yilgarn	Zn	5YR 5/6	0.4	0.2	1.36	341	4.86	2.9	0.8	75.2	21.2	22	-28.44	121.15
Bentley	NE Yilgarn	Zn	5YR 5/6	0.4	0.4	1.7	491	5.13	3.4	1.2	80.9	14.5	24	-28.44	121.15
Bentley	NE Yilgarn	Zn	5YR 5/6	0.5	0.4	1.56	387	5.13	4.7	3.5	63.3	28.5	6	-28.44	121.15
Boddington	SW Yilgarn	Au	10YR 5/4	12.7	5.1	1.27	933	4.77	2.5	2.3	27.3	67.9	15	-32.77	116.38
Boddington	SW Yilgarn	Au	10YR 5/4	13.8	5.2	10.79	641	6.4	1.0	5.6	40.7	52.7	56	-32.77	116.38
DeGrussa	Bryah Basin	Cu	5YR 5/6	0.5	0.4	1.36	575	4.93	1.6	4.4	58.3	35.8	38	-25.53	119.32
Kintore	Kalgoorlie	Au	7.5YR 6/4	3.4	0.5	22.5	27020	8.36	3.5	6.5	43.2	46.7	28	-30.59	121.01
Kintore	Kalgoorlie	Au	5YR 6/6	1.8	0.5	60.44	45660	8.14	2.9	4.2	66.9	25.9	42	-30.59	121.01
Kopai	Kalgoorlie	Au	5YR 5/4	2.8	0.4	16.17	5370	9.34	1.4	7.4	59.9	31.3	28	-30.80	121.25
Kopai	Kalgoorlie	Au	5YR 4/4	2.7	0.3	16.04	5951	9.16	1.9	7.1	68.3	22.6	41	-30.80	121.25
Moolart Well	NE Yilgarn	Au	-	1.1	0.6	1.42	937	4.89	4.3	3.8	80.8	11.1	58	-27.62	122.35
Area 7	Yandal	Au	5YR 5/6	0.4	0.4	3.72	26800	6.55	4.2	3.9	80.6	11.4	20	-26.47	120.69
Area 7	Yandal	Au	5YR 5/6	0.3	0.4	2.52	7503	5.78	2.1	5.9	76.7	15.4	20	-26.47	120.69
Tropicana	Albany-Fraser	Au	7.5YR 3/2	1.6	1.4	3.89	889	6.74	1.3	1.4	95.1	2.1	54	-29.24	124.54
Tropicana	Albany-Fraser	Au	7.5YR 3/2	1.7	1.1	3.68	1353	7.12	1.5	1.5	95.1	1.9	26	-29.18	124.52





### 3.1.2 Separation methods and materials

Soil was added to deionised (DI) water at a ratio of 1:5 in 120 or 240 mL high-density polyethylene (HDPE) bottles with screw-on lids. Sodium-hexametaphosphate (35.5 g/L) with Na<sub>2</sub>CO<sub>3</sub> (5 g/L) was used as a dispersant and added at a rate of 5 mL/100 mL solution. Samples were rolled for 24 hours and then stood approximately 4 hours dependant on laboratory temperature. The solution of <2 µm was drawn from the top 5 cm depth based on Stokes Law:  $F_d = 6\pi \eta R v$

where  $F_d$  is the frictional force in newtons (= kg m s<sup>-2</sup>)– acting on the interface between the fluid and the particle,  $\eta$  in Pa·s is the dynamic viscosity (= kg m<sup>-1</sup> s<sup>-1</sup>),  $R$  is the radius in metres of the spherical object,  $v$  is the flow velocity relative to the object in m/s. The solution was then dried at 60° C for ~24 hr in the same HDPE bottle.

Variations on the separation method tested different dispersants (None, analytical, laboratory and food grade Na-hexametaphosphate, 0.5M ammonium solution, 0.5M sodium carbonate; Table 4) and the application of ultrasonic vibration for 30 minutes and DI rinse steps. Other tests compared the weight of sample used for digestion and the particle size differences. Experiments are shown in Table 4.

Centrifugation was employed to separate the sub-micron fractions, also using Stokes Law, with c. 14 mins @ 3000 RPM for <0.75 µm and 16 minutes @ 8800 RPM for the <0.2 µm fractions using a Sorvall RC 6+ centrifuge and accounting for tube shape and rpm/slow down conditions.

Additionally, all bulk reference soils were subject to dry sieving and weighing of the various larger size fractions based on the methods described by Gee & Bauder (1986).

**Table 4. Various tests performed and referenced in this paper with the adjusted variables.**

Test	Experiment	Variables tested
1	Soil weight	0.1 g, 0.2 g, 0.5 g, 1 g, 4 g
2	Ultrafine size	2 µm, 0.75 µm, 0.2 µm
3	Solubility of ultrafine particles	Supernatant of 2 µm, 0.75 µm, 0.2 µm fractions
4	Extraction	4 acid, microwave aqua regia, aqua regia, dilute aqua regia, hydroxylamine
5	Dispersants	3 x Na-hexametaphosphate, ammonia, Na-carbonate
6	Separation	Dispersant only or “none”, dispersant + ultrasonics “ultrasonics” and dispersant + 2 DI water rinses “rinsed”
7	Alternative laboratories	ALS clay separation compared to UltraFine+ at Lab West
8	Spectral reflectance response	Reflectance of bulk and ultrafine sizes using visible to shortwave infrared spectrometer and shortwave to thermal infrared fourier transform infrared (FTIR) spectrometer

### 3.1.3 Laboratory analysis

The standard extraction for the method testing was MAR-04 offered through LabWest Pty Ltd, Perth, Australia as documented below along with additional extractions. All partial extractions of the soils were analysed for a multi-element suite of c. 40 elements using ICP-OES (Perkin Elmer Optima 7300DV) and ICPMS (Perkin Elmer Nexion 300Q).

- 1) **Microwave-assisted aqua regia digestion (LabWest MAR04).** Soil samples (0.2 g) were subjected to an aqua regia digestion with a 100% mixture of 3:1 concentrated HCl: HNO<sub>3</sub> and heated to 180 °C in a closed Teflon tube in a microwave (Anton Paar Multiwave PRO Microwave Reaction System). The detection limits are reported in Table 5.
- 2) **Aqua regia digestion (LabWest PL05s).** Soil samples (0.2 g) were subjected to an aqua regia digestion with a 100% mixture of 3:1 concentrated HCl: HNO<sub>3</sub> for 24 hours with some agitation. The samples are centrifuged and supernatant analysed.
- 3) **Weak aqua regia digestion (LabWest PL05).** Soil samples (0.2 g) were subjected to an aqua regia digestion with a 10% mixture of 3:1 HCl: HNO<sub>3</sub> for 24 hours with some agitation. The samples are centrifuged and supernatant analysed. This partial extraction has lower detection limits than those stronger acid extractions with 0.1 ppb Au.
- 4) **Weak (cold) hydroxylamine hydrochloride leach (LabWest PL04).** Soil samples of 0.2 g were weighed into a 50 ml polypropylene centrifuge tube with 45 ml of 0.01 M hydroxylamine hydrochloride. Each sample was then shaken for 24 h at room temperature, centrifuged for 10 min (4500 rpm), and the supernatant was decanted into test tubes for analysis. This extraction is interpreted to selectively digest elements bound in amorphous Mn oxide, and to a lesser extent amorphous Fe oxide, fractions of soil samples (Chao, 1984; Gray *et al.*, 1999).
- 5) **Microwave-assisted four acid digestion (LabWest MMA04).** Soil samples (0.2 g) were subjected to a HF/multiacid aqua regia digestion and heated to 180 °C in a closed Teflon tube in a microwave (Anton Paar Multiwave PRO Microwave Reaction System). The detection limits are slightly higher than those in Table 5 and, importantly, the Au detection limit is 5 ppb.

**Table 5. Detection limits for soil sample analysis by microwave assisted multi-acid digestion (MAR-04). Detection limits are in ppm unless shown otherwise**

LabWest Code MAR04 (42 elements)							
<b>Ag</b>	0.01 - 100	<b>Cr</b>	2 - 1,000	<b>Mo</b>	0.1 - 1,000	<b>Se</b>	0.05 - 1,000
<b>Al</b>	0.001% - 10%	<b>Cs</b>	0.1 - 1,000	<b>Na</b>	0.001% - 10%	<b>Sn</b>	0.2 - 500
<b>As</b>	0.5 - 5,000	<b>Cu</b>	0.2 - 10,000	<b>Nb</b>	0.5 - 100	<b>Sr</b>	0.1 - 1,000
<b>Au</b>	0.5 - 5000 ppb	<b>Fe</b>	0.01% - 50%	<b>Ni</b>	2 - 10,000	<b>Te</b>	0.02 - 1,00
<b>Ba</b>	0.2 - 1,000	<b>Hg</b>	0.05 - 500	<b>P</b>	0.001% - 1%	<b>Th</b>	0.02 - 1,000
<b>Be</b>	0.2 - 1,000	<b>In</b>	0.01 - 1,000	<b>Pb</b>	0.2 - 1,000	<b>Tl</b>	0.1 - 1,000
<b>Bi</b>	0.1 - 10,000	<b>K</b>	0.001% - 10%	<b>Rb</b>	0.1 - 1,000	<b>U</b>	0.02 - 1,000
<b>Ca</b>	0.001% - 10%	<b>La</b>	0.05 - 1,000	<b>Re</b>	0.01 - 500	<b>V</b>	2 - 1,000
<b>Cd</b>	0.05 - 1,000	<b>Li</b>	0.05 - 10,000	<b>S</b>	0.005% - 10%	<b>Y</b>	0.05 - 1,000
<b>Ce</b>	0.05 - 1,000	<b>Mg</b>	0.001% - 10%	<b>Sb</b>	0.1 - 1,000	<b>Zn</b>	0.20- 10,000
<b>Co</b>	0.2 - 1,000	<b>Mn</b>	2 - 1,000	<b>Sc</b>	1 - 1,000		

### 3.1.4 Spectral analysis

Visible-near to shortwave infrared reflectance measurements were acquired on the bulk and separate samples using a Malvern Panalytical Analytical Spectral Devices (ASD) FieldSpec4 standard resolution spectroradiometer. The ASD FieldSpec4 collects in the 350 to 2500 nm wavelength region. For the initial testing of the bulk and separated samples reflectance data was obtained using a muglight accessory and black anodized aluminium sample cups with 25 mm sapphire glass windows. A reference measurement was made on a plug of sintered polytetrafluoroethylene (PTFE, aka. Spectralon™) sitting inside the cup which was ratioed with the sample measurement to obtain a relative reflectance. The relative reflectance was converted to absolute reflectance in post processing by a reflectance correction factor for the Spectralon™ plug.

For the testing of the separates for measurement using a commercial laboratory method the reflectance measurements of the <2 µm separations were performed using a bifurcated optical fibre probe supplied by Malvern Panalytical ASD. One of the bifurcated ends, containing 18 400 µm fibres, was attached to an ASD Illuminator accessory, which houses a quartz halogen lamp. The other end, containing a single large fibre, was mated to the ASD FieldSpec4 optic fibre ferrule using a joiner. A spacer ring was mounted on the common end of the probe to allow a gap of 5 mm between the sample surface and the tip of the fibres. A calibrated piece of PTFE was used as the reflectance standard and measured before each set of soil measurements by raising the white reference standard on a lab jack located directly below the downward facing probe. The samples were measured in their plastic centrifuge vials after being gently crushed with a glass stirring rod. Care was taken not to collect the reflectance of the plastic of the vial. After each measurement the probe was cleaned with a wipe with a Kimwipe and jet of compressed air. A total of 40 scans were averaged into a single measurement. Each measurement took approximately 10 seconds.

Mid to longwave infrared diffuse reflectance measurements were acquired on the bulk and clay-separate samples using a Bruker Vertex 80v fourier transform infrared (FTIR) spectrometer with a Bruker A562 Au integrating sphere attachment. A glow bar internal source and KBr beam splitter was used in the foreoptics of the Bruker Vertex, with a 2x2 mm MCT detector attached to the baffled exit port of the integrating sphere. The samples were placed in a black anodised aluminium 25mm x 3 mm sample cup and the surface gently smoothed with a glass slide. The sample cup was placed in the bottom port of the integrating sphere and a background reference measurement of the sphere wall was collected before the internal mirror in the sphere was manually switched to point the incoming beam down towards the lower port containing the sample. The diffuse reflectance between 7000 and 500 wavenumbers (cm<sup>-1</sup>) was acquired and converted into approximately 1428 to 16667 nm (due to data being noisy below 600 cm<sup>-1</sup>).

### **3.1.5 Additional analysis**

Total Carbon was measured using an Elementar CS Cube combustion method, with organic C measured using the same device, but samples were pre-treated with HCl to destroy any carbonate C.

Prior to settling or separation methods, the pH (TPS 900-P) and electrical conductivity (TPS K=1 sensor) of 1:10 w/w soil to water ratio slurries were measured at CSIRO laboratories, Kensington for all reference samples collected in this study. Using the Ultrafine+ developed workflow, similar measurements were made at Lab West using a TPS AQUA-CP/A Waterproof Conductivity/TDS/Sal/pH/Temperature meter.

Particle size analysis was measured using a Malvern Mastersizer 3000 in both dry mode using the AeroS attachment and in suspension using the HydroMV unit. Both techniques assumed non-spherical particles and kaolinite as the dominant mineral with a refractive index of 1.45 (this is not dissimilar to the value for quartz or Fe oxides which also are common minerals with 1.51 and 1.456, respectively).

### **3.1.6 Quality control and data treatment**

A certified reference soil (OREAS 250) was incorporated into all analyses as a geochemical standard. Laboratory duplicates were analysed at a rate of 1 per 20 samples (n=5 per extraction test). Percent half absolute relative difference (HARD) was determined on the standard soil to be acceptable if it was <10% for the aqua regia, multiacid and hydroxylamine hydrochloride extractions. The HARD (Stanley & Lawie, 2007) was calculated as a percentage for all compounds using method and field duplicate samples:

$$\text{HARD} = ((\text{assay1} - \text{assay2}) / (\text{assay1} + \text{assay2})) \times 100.$$

Quality control on the spectral reflectance measurements involved the measurement of an internal sand standard of quartz (from Lucky Bay, Western Australia) and on KGa-1b (Clay Mineral Society well crystalline kaolinite from Georgia, USA) at the start, middle and end of daily measurements.

## 4 Experiments and Results

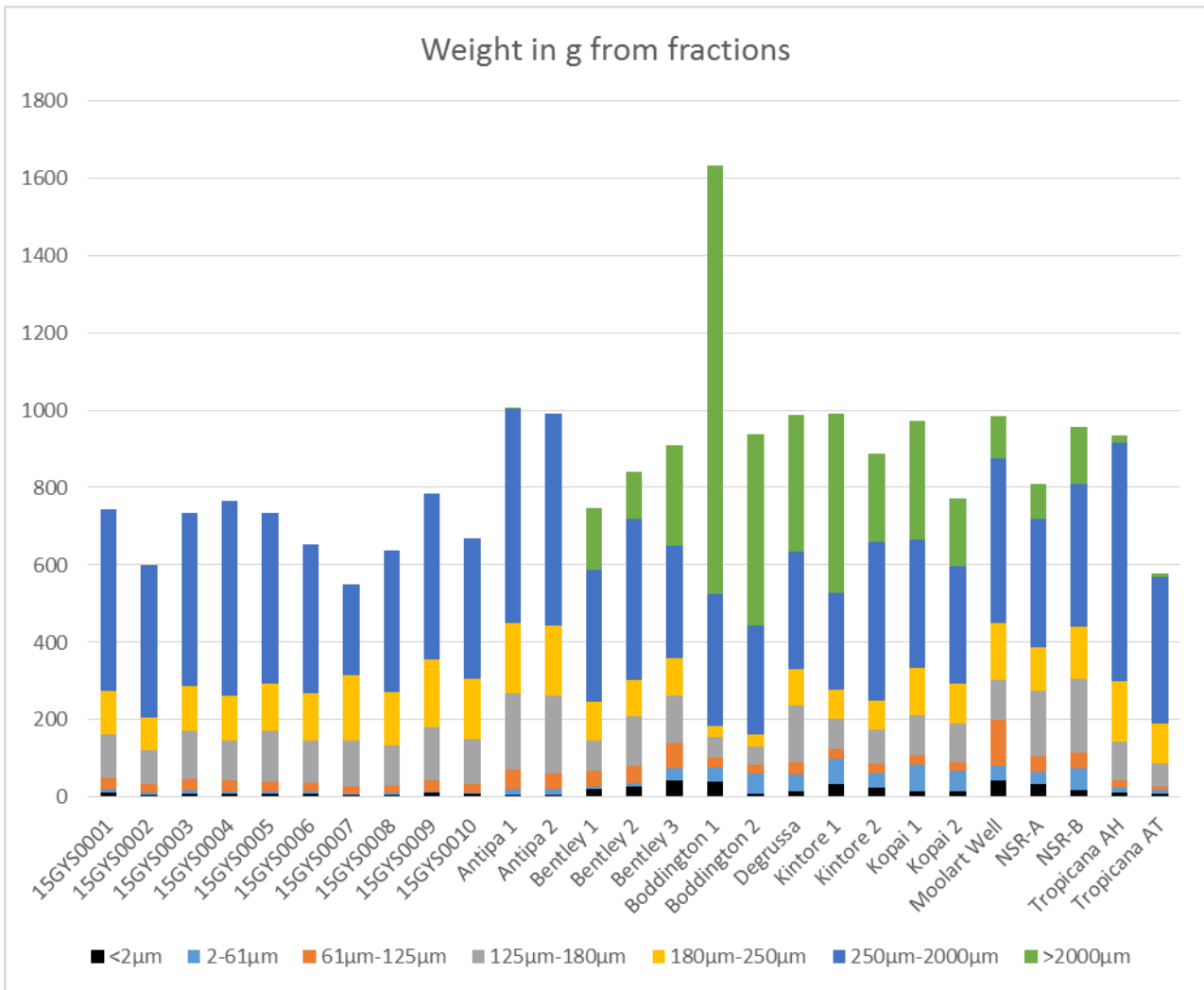
The bulk reference samples were dominated by sand size fractions. Figure 2 shows the particle size distribution by weight, with the medium to coarse sand (>180 µm) dominating most of the soil mass as is common in many Western Australian soils. This distribution was determined by dry sieving, and so some clay coatings contribute to the weight of the larger particles. This partly explains a discrepancy between the particle size analyses determined using the laser scattering and the plot below. Dry sieving only gets to the silt fraction (~<61 µm in this study). To calculate the proportion of silt and clay, the proportions between these two fractions in the laser scatter analysis was used and applied to split the dry weight recovered. Again, this slightly underestimates the fine materials. The results of the Au analysis for the reference sites and the other size fractions show that Au and Cu is commonly higher in the <2 µm fraction (Table 6 and Table 7). Only one sample was at the detection limit of 0.5 ppb for Au (Antipa 2) and this sample is from shallow regolith of Quaternary and Tertiary sand cover that sits 60 m above prospective lithology, and these regolith materials have little Au or other trace elements of interest in this part of the sequence.

**Table 6. Gold concentration in ppb of bulk reference samples by particle size.**

Sample	<2µm	2-61µm	61-125µm	125-180µm	180-250µm	250-2000µm	>2000µm
15GYS0001	4	1.2	3	1.7	1.8	0.8	
15GYS0002	4.7	3	1.1	1	1.9	0.5	
15GYS0003	2.4	1	0.9	0.5	2	0.5	
15GYS0004	5.3	3.2	0.6	2.2	1	2.4	
15GYS0005	4.02	2.4	3.3	0.8	0.5	0.5	
15GYS0006	2.9	1.7	0.5	0.7	0.5	1.1	
15GYS0007	1.5	0.5	8.7	0.5	1.4	1.4	
15GYS0008	2.7	0.5	0.5	0.5	1.3	1.4	
15GYS0009	2.3	3.2	1.4	0.9	1.3	3.4	
15GYS0010	2.6	1	1.7	0.5	1.8	0.5	
Antipa 1	1.87	2.1	1.8	1.5	2.1	2	
Antipa 2	0.5	1.3	0.5	0.5	0.7	0.5	
Bentley 1	2.5	4	3.8	3.1	1	1	4.6
Bentley 2	1.8	3.3	3.7	0.5	2.5	1.8	1.3
Bentley 3	2.7	6.4	1.6	0.7	0.5	1.4	1.5
Boddington 1	15.3	22.7	19.2	10.3	8.7	14.1	5.1
Boddington 2	52.95	38.2	23.4	24.1	25.6	30.2	12.2
Degrussa	3.72	3.7	0.8	0.5	1	1.4	1.6
Kintore 1	110	101	65.9	64.7	55.6	51.6	22.9
Kintore 2	119	120	79.9	78.5	63.3	53.8	50.4
Kopai 1	37.52	29.2	18.4	13.1	14.8	11.4	22
Kopai 2	28.7	39.9	25.3	18.9	13.2	11.3	31.7
Moolart Well	2.8	1.2	1.4	0.7	0.9	2.4	2.3
NSR-A	8.30	8.7	3.8	3.5	1.5	3.4	6.4
NSR-B	7.1	7.2	2.3	4.7	1.8	5.1	5.7
Tropicana AH	6.9	6.8	1.3	1.9	1.1	1.2	2.8
Tropicana AT	2.5	3.7	0.8	0.5	1.1	0.5	2.7

**Table 7. Copper concentration in ppm of bulk reference samples by particle size.**

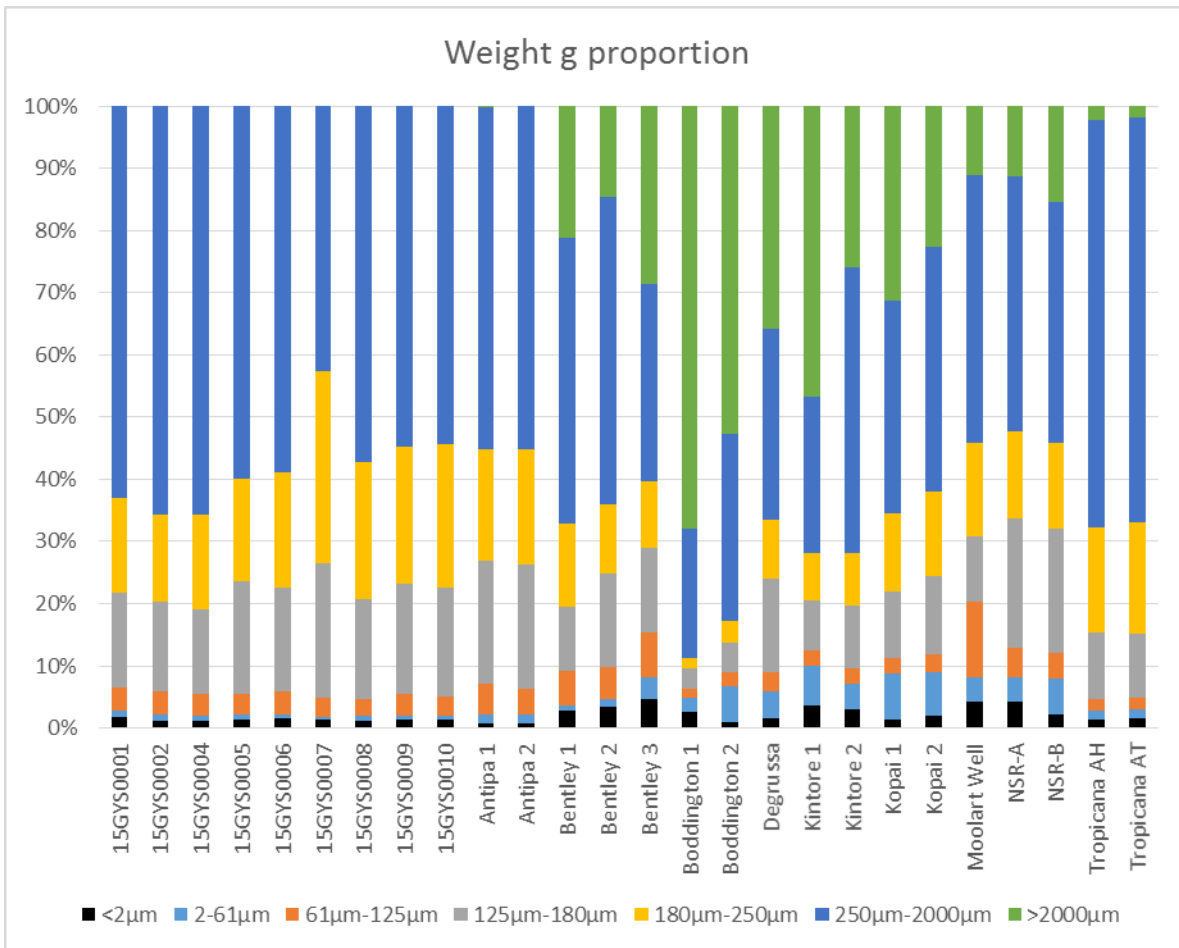
Sample	<2µm	2-61µm	61-125µm	125-180µm	180-250µm	250-2000µm	>2000µm
15GYS0001	27.5	17.7	10.5	10.7	6.4	6.1	
15GYS0002	27.9	18.1	11.5	8.7	10	3.9	
15GYS0003	24.9	16.3	10.2	11.1	5.7	5.5	
15GYS0004	25.8	18.6	10.9	8.3	9.8	2.9	
15GYS0005	26.87	11.7	9.6	9.1	5.3	5.6	
15GYS0006	27.1	14.9	9.2	7.4	9.3	3.7	
15GYS0007	25	12.2	8.9	8.9	4.3	6.1	
15GYS0008	23.4	12	8.9	6.9	9.1	3.3	
15GYS0009	25.8	16.6	9.4	10.6	5	6	
15GYS0010	26	12.8	9	6.7	8.1	3.2	
Antipa 1	15.33	7.4	5.8	6.5	3.7	4.5	
Antipa 2	9.6	7.4	9.4	4.5	6.5	1.8	
Bentley 1	45.9	49	41.3	38	34.1	47.5	58.3
Bentley 2	44.7	44.5	38.8	32.9	29.8	31.4	56.9
Bentley 3	53.6	51.7	37.7	36.2	33	51	62.5
Boddington 1	38.8	55.6	38.1	28.2	32.3	26.1	20.7
Boddington 2	120.83	93.8	76.9	66.4	57.2	72	45.3
Degrussa	65.87	57	41	37.2	34.2	52.5	63
Kintore 1	59.7	52.7	47.5	47	41.7	45.8	76.3
Kintore 2	64.1	63.2	57.7	57.3	53.7	48.1	63.7
Kopai 1	46.93	38.4	38.3	33.2	23.8	23.3	26.7
Kopai 2	53.7	38.5	39.2	33.2	27.7	22.6	22.8
Moolart Well	43.6	39.8	21.7	24.6	21.6	53.3	93.7
NSR-A	45.42	44.1	29.4	26.8	27.3	58	140
NSR-B	52.7	47.4	33.4	28.7	26	54.9	143
Tropicana AH	19.3	20.1	10.3	9.5	12	3.5	11.5
Tropicana AT	24.2	21.9	10.3	16.8	6.6	7.8	13.6



**Figure 2. Bulk reference samples percentage fractions of larger particle size fractions by weight. Weight was determined by dry sieving and then by calculation using a proportion of the laser scatter particles size to ratio the remaining <61 µm material. GY samples are Gruyere, NSR are Area 7 from Northern Star Resources. As expected the finer fraction comprises <10% of the soil.**

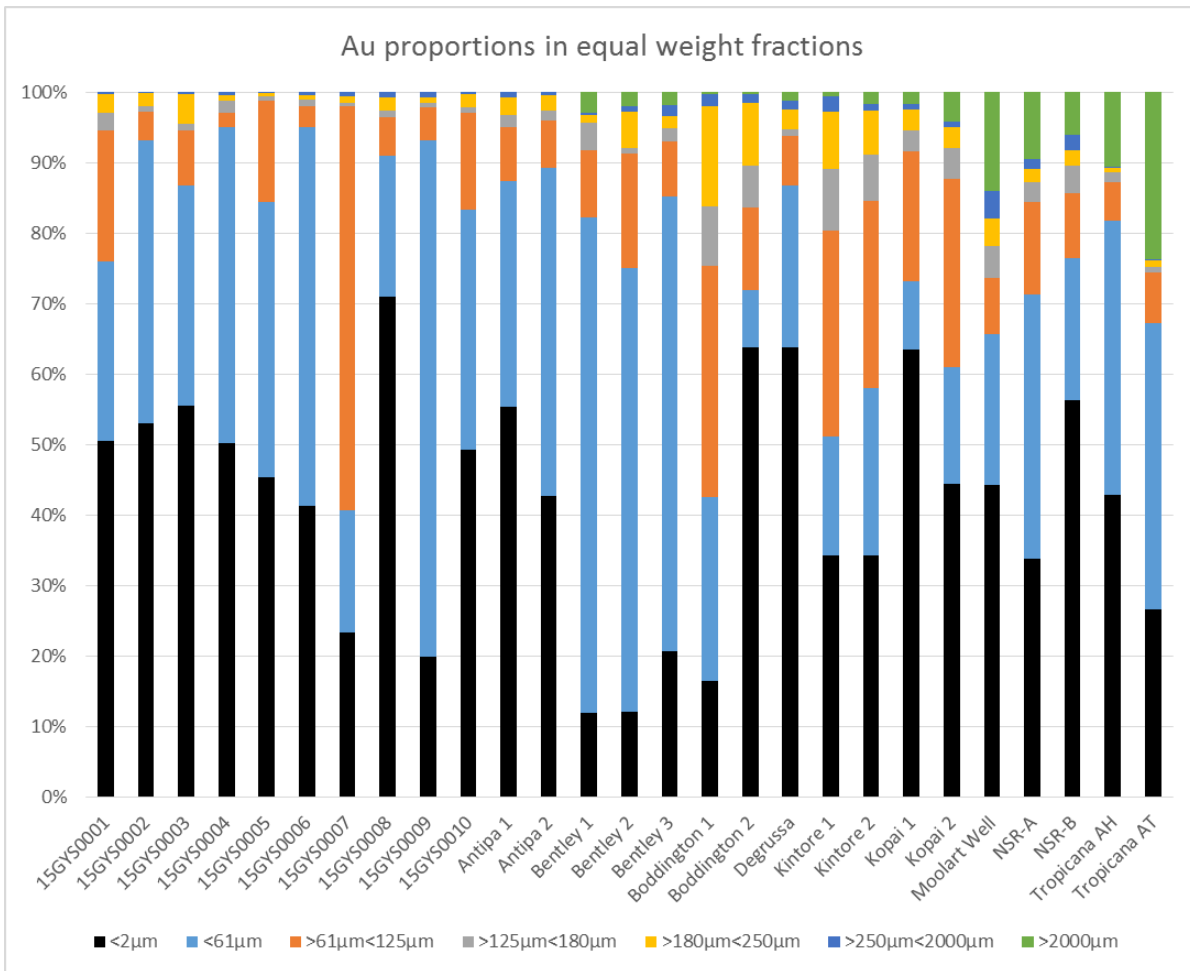
Scaling the various weights of the size fractions to 100% provides a more even comparison of the bulk reference soils. For many samples the fine to medium sand is quite similar (yellow and grey coloured bars in Figure 3), with the biggest differences highlighting those soils with a significant coarse soil component (green colour in Figure 3) such as Boddington that has a lot of pisolith gravels in the surface environment. The Boddington site was also unusual in not having the sand fraction that most other sites had and the large organic component (Table 3). All of the samples used in this study as reference samples had <5% clays and a similar proportion of silts (2-61 µm, black and light blue in Figure 3). These minor components by weight were determined to have a similar or greater concentration of trace elements of interest.

The results will focus on Au and Cu analysis as these were considered the most important commodity targeting elements. Adjusting the Au concentration by assuming all the soil size fractions were equally proportioned highlights the dominance of the Au (Figure 4) and Cu (Figure 5) in the size fractions where the fine fractions and those size fractions that have the most exchange sites (Table 3). This result highlights the value of concentrating and analysing the fine fractions (or any fraction) separately – it provides a more level comparison.

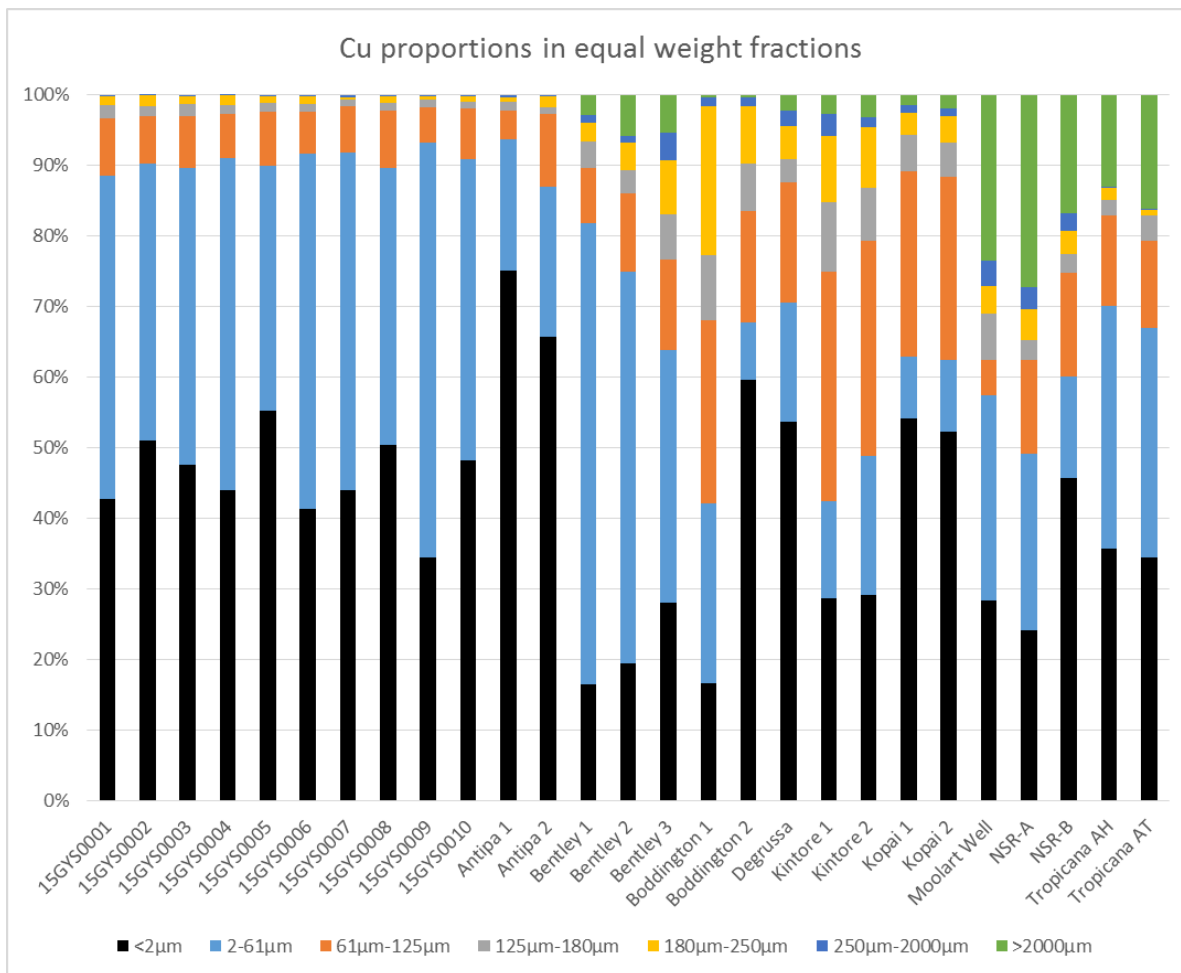


**Figure 3. Bulk reference samples distribution of Au in each fractions of larger particle sizes. GY samples are Gruyere, NSR are Area 7 from Northern Star Resources. The finer fraction commonly hosts more Au than most other fractions. And lower Fig Bulk reference samples distribution of Au in each fractions of larger particle sizes. GY samples are Gruyere, NSR are Area 7 from Northern Star Resources. The finer fraction commonly hosts more Au than most other fractions. If adjusted for weight the bulk of the Au is in the finer fractions. The <2µm fractions cannot be weight adjusted with the present series of tests.**





**Figure 4. Bulk reference samples distribution of Au in each fractions of larger particle sizes adjusted for the proportional weight of each fraction (i.e. if particle sizes were in equal proportions). The finer fractions (<125 µm) commonly hosts more Au than most other fractions. GY samples are Gruyere, NSR are Area 7 from Northern Star Resources.**



**Figure 5. Bulk reference samples distribution of Cu in each fractions of larger particle sizes adjusted for the proportional weight of each fraction (i.e. if particle sizes were in equal proportions). The finer fractions (<125 µm) commonly hosts more Cu than most other fractions. GY samples are Gruyere, NSR are Area 7 from Northern Star Resources. Results were similar for Zn.**

#### 4.1.1 Test 1 Sample weight required for analysis

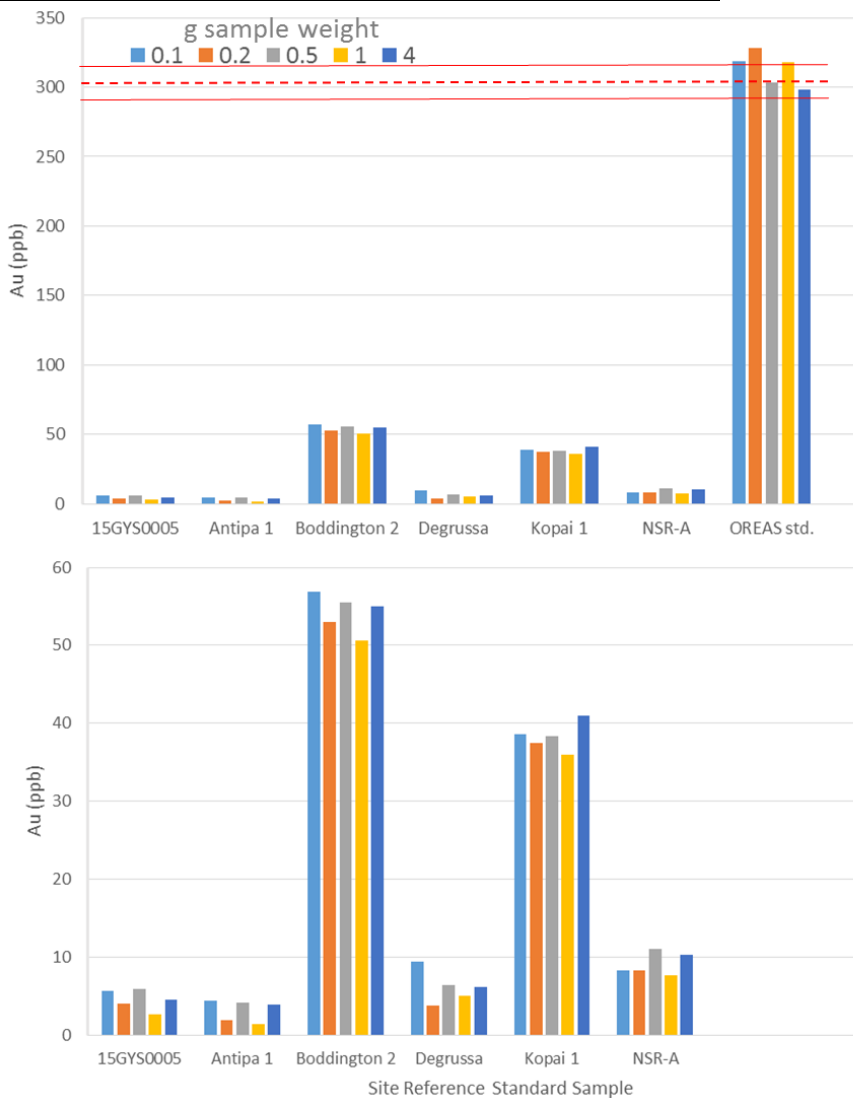
The results from six replicates of seven reference samples using five different weights were tested using the MAR04 (microwave assisted aqua regia extraction) analytical method from Lab West. The weights were 4.0 g, 1.0 g, 0.5 g, 0.2 g and 0.1 g. A multiple analysis of variance (MANOVA) test showed no significant differences in the results for all weights tested for Au (

Table 8; Figure 6) and minor differences for Cu (Figure 7) and Zn.

Figure 7 shows that even when significant differences are observed between the weights used the differences are not extreme and do not greatly influence the interpretation of the data in practice (e.g. orientation surveys, Noble *et al.*, Part 2). Not surprisingly the MANOVA also showed significant differences between all sites and nearly all elements – since the samples are from distinct regions this is to be expected and any similarities are coincidental. The replicates were very uniform and the reproduction of the OREAS 250 standard (303 ppb Au) matched exceptionally well (Figure 6). Based on these findings, the use of very small weights for the analysis is suitable and preferred as it requires less starting material, lower energy and consumption of acids to complete the digestion. From this finding the following experimental comparisons (extractions, settling chemicals, method variations) used the 0.2 g sample weights. The 0.2 g or 0.5 g are presently the best sizes for sample precision and accuracy for most elements of interest.

**Table 8. MANOVA comparisons of sample weights for Au. All Pairwise Multiple Comparison Procedures (Holm-Sidak method). Overall significance level = 0.05.**

Comparison (g)	Diff of Means	t	P	P<0.050
0.100 vs. 4.000	3.286	1.52	0.753	No
0.100 vs. 1.000	2.964	1.371	0.817	No
0.200 vs. 4.000	2.526	1.168	0.894	No
0.100 vs. 0.500	2.493	1.153	0.867	No
0.200 vs. 1.000	2.205	1.02	0.891	No
0.200 vs. 0.500	1.733	0.802	0.937	No
0.500 vs. 4.000	0.793	0.367	0.993	No
0.100 vs. 0.200	0.76	0.351	0.979	No
0.500 vs. 1.000	0.471	0.218	0.97	No
1.000 vs. 4.000	0.321	0.149	0.882	No



**Figure 6. Comparing weight of the fine fraction sample used in the analysis of Au at six references sites and using OREAS 250 standard with reported value as a solid line (303 +/- 13 or 1 standard deviation –red dashed lines (top)); the same data with the standard removed and rescaled to see the lower concentrations of some sites more clearly. No significant differences occur between any of the weight classes. GY samples are Gruyere, NSR are Area 7 from Northern Star Resources.**

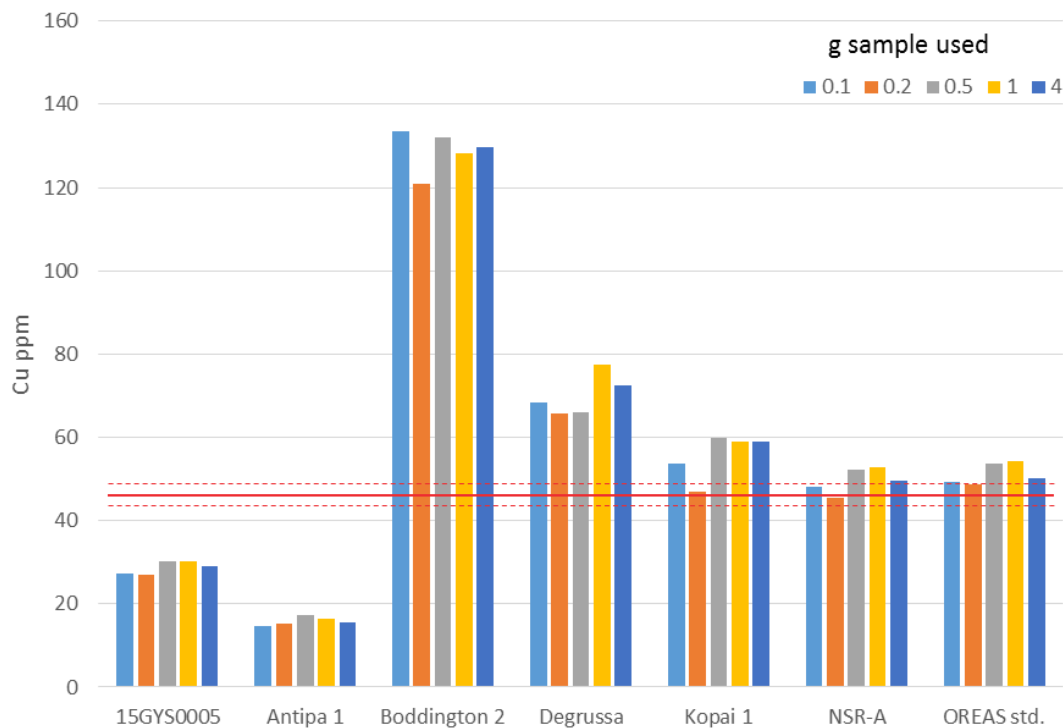


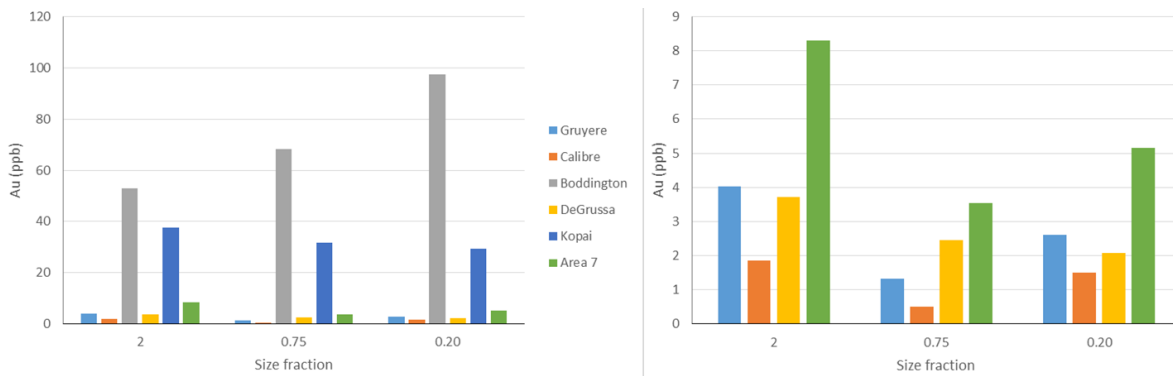
Figure 7. Comparing weight of the fine fraction sample used in the analysis of Cu at six references sites and using OREAS 250 standard with reported value as a dashed line (44.7 +/- 1.3 or 1 standard deviation –red solid lines. Some differences occur between most of the weight classes, but visually any differences observed are small. GY samples are Gruyere, NSR are Area 7 from Northern Star Resources.

#### 4.1.2 Test 2 Ultrafine sample size fraction

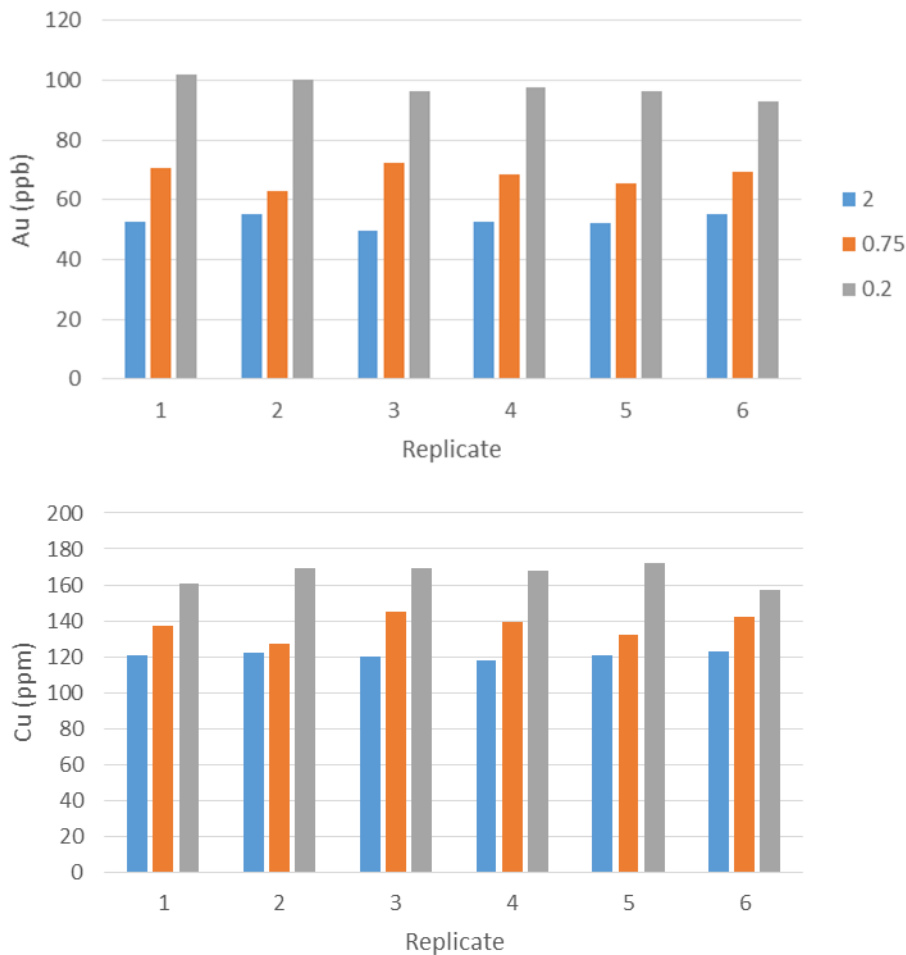
The results from six replicates of six reference samples using three different (ultrafine) particle size fractions were tested using the MAR04 analytical method from Lab West. The size fractions were separated using settling and centrifugation to 2 µm, 0.75 µm and 0.2 µm. Results show there is no additional benefit to going to smaller than 2 µm clay fractions (sub-micron) with the 0.75 µm and 0.2 µm showing very little differences to the larger and more easily extracted <2 µm fraction (e.g. Table 9). Combining all six sites there was no significant differences between 2 µm and 0.75 µm, although both of these were different to the very small 0.2 µm fraction. These differences tended to be more pronounced at the sites that were more challenging from a separation aspect, Boddington with much greater organic content (Table 3) and Kopai that had a higher salt content and very little easily separated clay fraction. These sites also had the highest Au concentrations (10s ppb Au). The 2 µm fraction on average tended to have slightly greater Au (Figure 8) although not always as is evident at Boddington. The extraction results were consistent, especially at the Boddington site (Figure 9). All fractions do not show evidence of nugget effects, although some reproducibility of Au varied 100% at the lower concentrations e.g. 4 ppb and 8 ppb replicates for Au, but none of the samples varied from near detection to many 10s or 100s of ppb Au as had been observed with nugget effects in other samples and analytical techniques. Other elements are much more stable with Cu being very consistent in all of the size fractions tested (Figure 9).

**Table 9. MANOVA comparisons of size fractions for Au at all sites combined and then some of the individual sites. All Pairwise Multiple Comparison Procedures (Holm-Sidak method). Overall significance level = 0.05.**

Sites	Comparison	Diff of Means	t	P	P<0.050
All	0.200 vs. 0.750	5.042	10.073	<0.001	Yes
All	0.200 vs. 2.000	4.961	9.912	<0.001	Yes
All	2.000 vs. 0.750	0.0806	0.161	0.872	No
Calibre	2.000 vs. 0.750	1.35	1.101	0.617	No
Calibre	0.200 vs. 0.750	0.983	0.802	0.669	No
Calibre	2.000 vs. 0.200	0.367	0.299	0.766	No
DeGrussa	2.000 vs. 0.200	1.633	1.332	0.461	No
DeGrussa	2.000 vs. 0.750	1.267	1.033	0.516	No
DeGrussa	0.750 vs. 0.200	0.367	0.299	0.766	No
Gruyere	2.000 vs. 0.750	2.683	2.189	0.091	No
Gruyere	2.000 vs. 0.200	1.4	1.142	0.447	No
Gruyere	0.200 vs. 0.750	1.283	1.047	0.298	No



**Figure 8. Comparing sizes of the fine fractions and the average concentration of Au extracted. Results are average of six replicates at six references sites (left); the same data with the greater Boddington and Kopai site concentrations removed and rescaled to see the lower concentrations at some sites more clearly (right). Few significant differences occur between any of the size classes.**



**Figure 9. Comparing sizes of the fine fractions and the concentration of Au (top) and Cu (bottom) extracted. Results show the consistency of the analysis of six replicates at the Boddington site. The results were uncharacteristic of most sites in that it that showed greater concentrations in the <0.2  $\mu\text{m}$  fraction and not the <2  $\mu\text{m}$ .**

#### 4.1.3 Test 3 Solubility of ultrafine particles into supernatant

As an additional check on the effectiveness of the separation and analysis, the supernatant fluid was also analysed. It was expected that some highly soluble metals or nanoparticulate “floating” Au might remain in the solution. The supernatant had very little soluble material. No Au was detected in the discarded solutions in these tests and only trace amounts of Cu was in the solution. The trace soluble Cu was equivalent to 0.2% of the total extracted in the solid phase and did not influence the interpretation of the data. The difference in the mean values among the different size fractions separated and concentrations in the remaining supernatant is not great enough to exclude the possibility that the difference is just due to random sampling variability after allowing for the effects of differences in sites. There is not a statistically significant difference ( $P = 0.120$ ) and the individual comparison tests are shown in Table 10.

**Table 10. MANOVA comparisons of size fractions and the remaining solution for Au at all sites combined. All Pairwise Multiple Comparison Procedures (Holm-Sidak method). Overall significance level = 0.05.**

Comparison	Diff of Means	t	P	P<0.050
0.200 vs. 2.000	0.0061	2.082	0.123	No
0.750 vs. 2.000	0.00417	1.433	0.292	No
0.200 vs. 0.750	0.00193	0.5	0.619	No

#### 4.1.4 Test 4 Extraction comparison Soil chemistry

Five replicates of five bulk reference soils including a standard reference soil were subject to extraction from five different solutions to provide an indication of the best method to partner with the 2  $\mu\text{m}$  particle size

extraction. All extractions and analyses were conducted by LabWest, Malaga. The results showed that aqua regia related extractions are effective for Au solubilisation, with microwave- assisted aqua regia multi-element analysis by ICP-MS/OES the preferred method (MAR-04 in the LabWest scheme). Testing a known standard, OREAS 250 (Figure 10) all the strong acid digests were effective. The microwave-assisted extraction reports greater concentrations than any other method (Figure 10) and this is attributed to no “normal” loss from volatile phases in the closed and heated vessel compared the more traditional heating block vessels. The 100% aqua regia does not extract all the Au, although both the 100% and 10% aqua regia do a reasonable job (Figure 10a). The four acid digestion is probably the most accurate at this concentration range, however is should be evident in Figure 11 that many sites have much lower Au concentrations in soil. In samples that are low in Au concentration the four-acid digest is limited as the detection limit of 5 ppb may cause samples to report below detection. This 5 ppb detection limits would prove problematic in other regional studies (Noble *et al.*, 2018a; Noble *et al.*, 2018b)

The hydroxylamine hydrochloride (HA) is not effective (Figure 10 and Figure 11). In the ultrafine fractions separation, there is not a lot of Mn oxides that would be reduced and in turn liberate metals in this phase. Reducing Au solid particles (0 valence state) is also not effective in solubilising Au that ideally resides in a +1 or +3 state. As a result, HA is not a viable extraction method for these soils and this workflow.

Although strongly focused on Au to avoid the nugget effect and the variants of aqua regia extraction that are known to be most effective at Au solubilisation, the extraction of Cu is also effective using the microwave aqua regia and strong Cu recovery is achieved (Figure 12).

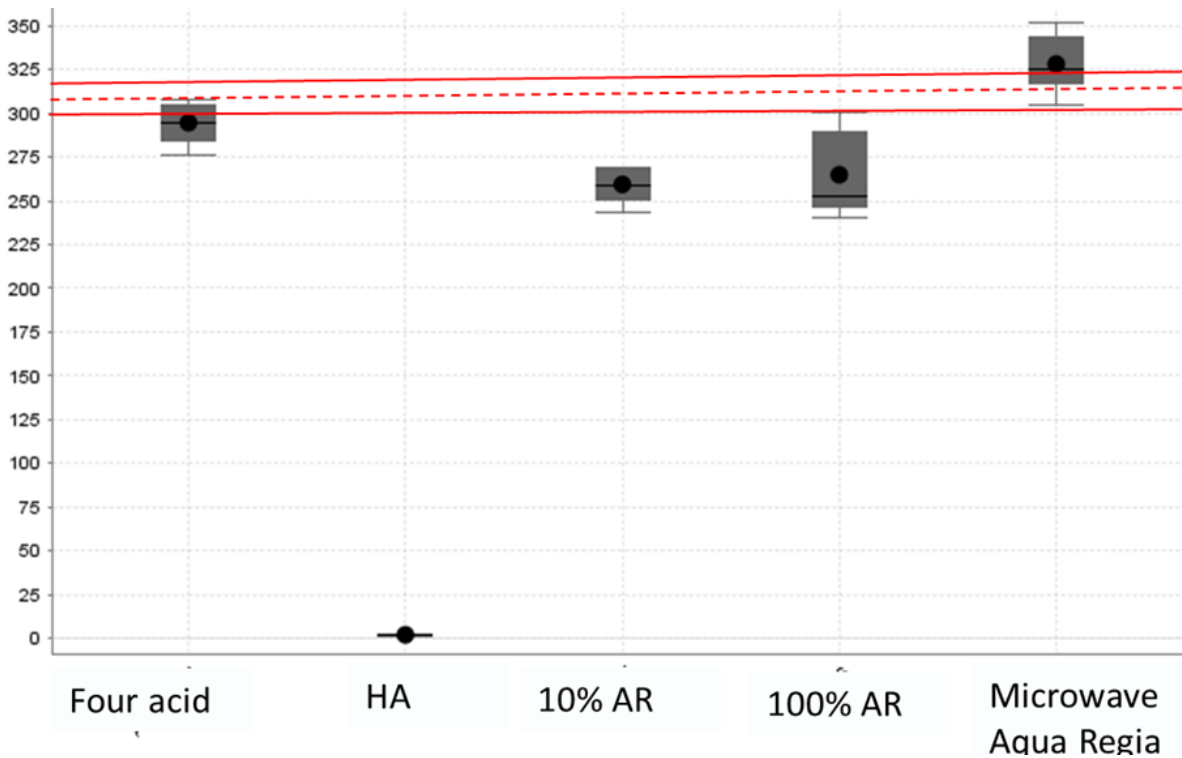


Figure 10. Comparison of the Au extracted from the OREAS 250 standard using the five different extractions tested. The reported value in concentrated aqua regia is the solid line (303 +/- 13 or 1 standard deviation –red dashed lines).

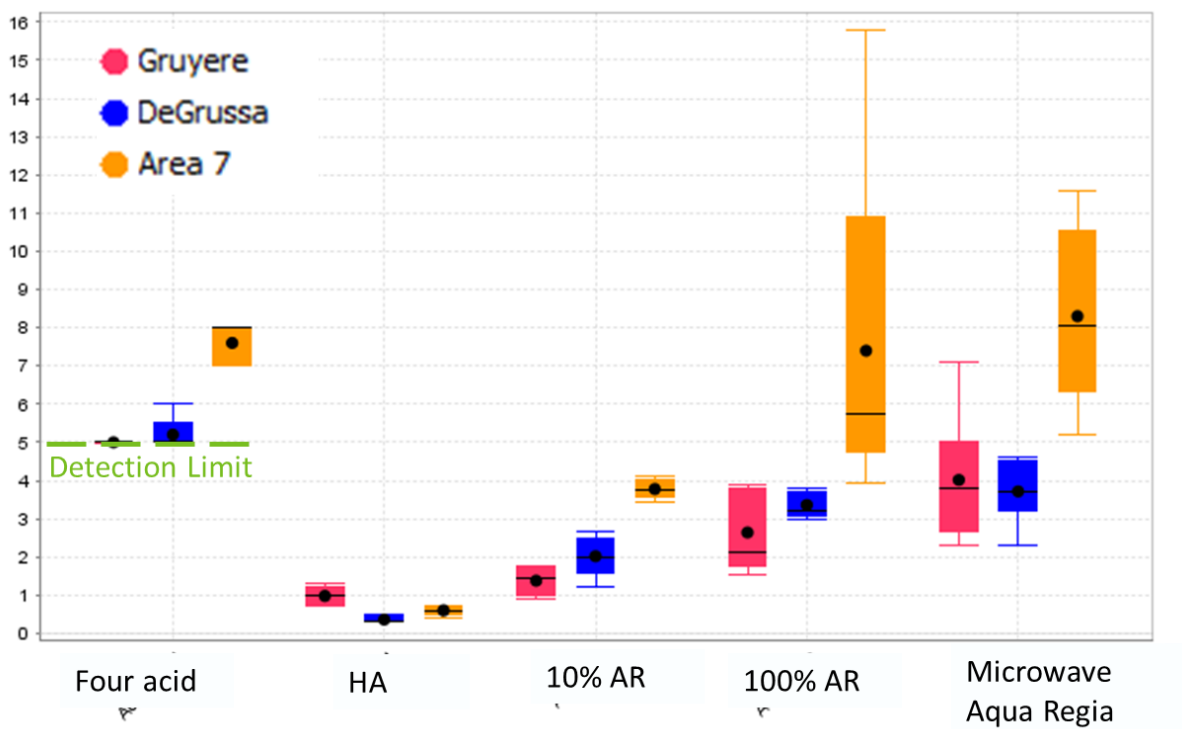


Figure 11. Comparison of the Au extracted from the bulk reference samples from DeGrussa, Gruyere and Area 7 using the five different extractions tested. Refer to Table 3 for more details about the reference samples and sites



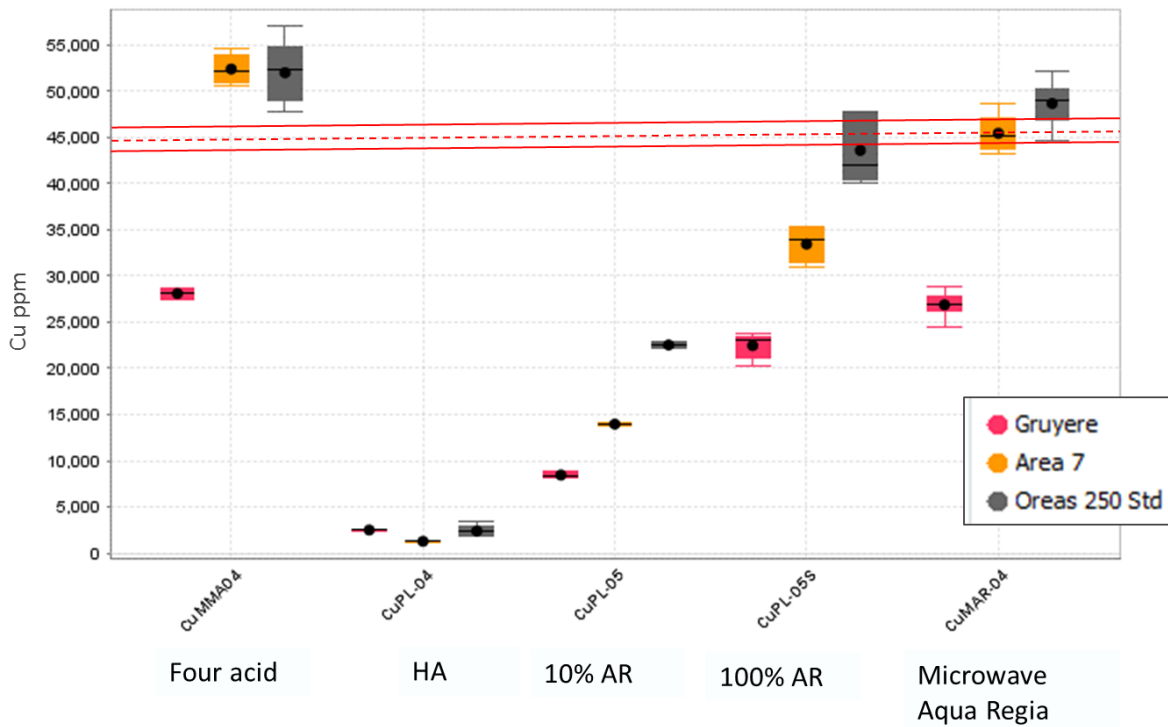


Figure 12. Comparison of the Cu extracted from the OREAS 250 standard, and reference samples from Gruyere and Area 7 in the eastern Yilgarn Craton using the five different extractions tested. The reported value in concentrated aqua regia is the solid line (44.7 +/- 1.3 or 1 standard deviation –red dashed lines).

#### 4.1.5 Test 5 Comparison of dispersants

For the ultrafine fractions separation, early testing had indicated that for Western Australian soils, a dispersant was valuable to ensure adequate recovery of the fine particles. A number of dispersants are viable and this experiment was designed to compare three different grades of Na hexametaphosphate, Na carbonate and ammonia. For this test two bulk reference soils (DeGrussa and Kopai) were used and each separation was done with five replicates. A MANOVA test showed the main effects of the dispersants cannot be properly interpreted if significant interaction is determined with the different sites. This is because the size of the dispersants effect depends upon the site, i.e., the sites respond very differently and variably to the dispersants. These soils are distinctly different and there is a statistically significant interaction between Site and Dispersant, ( $P < 0.001$ ).

Table 11 shows the dispersants are significantly different, except for the bulk and laboratory grade Na-hexametaphosphate. The results of the DeGrussa soils only shows that there is little difference between the dispersants except for the ammonia (

Table 11). Ammonia extracted slightly more Au than the other dispersants for both reference soil types (Figure 13). The biggest contrast in treatments was the Na-carbonate that was reasonably effective at DeGrussa, but performed poorly for Au and Cu at Kopai. The Kopai site is the most alkaline soil tested (pH =9.3; Table 3) and, clearly, adding a carbonate solution did not disrupt the standard conditions of that soil (Figure 13).

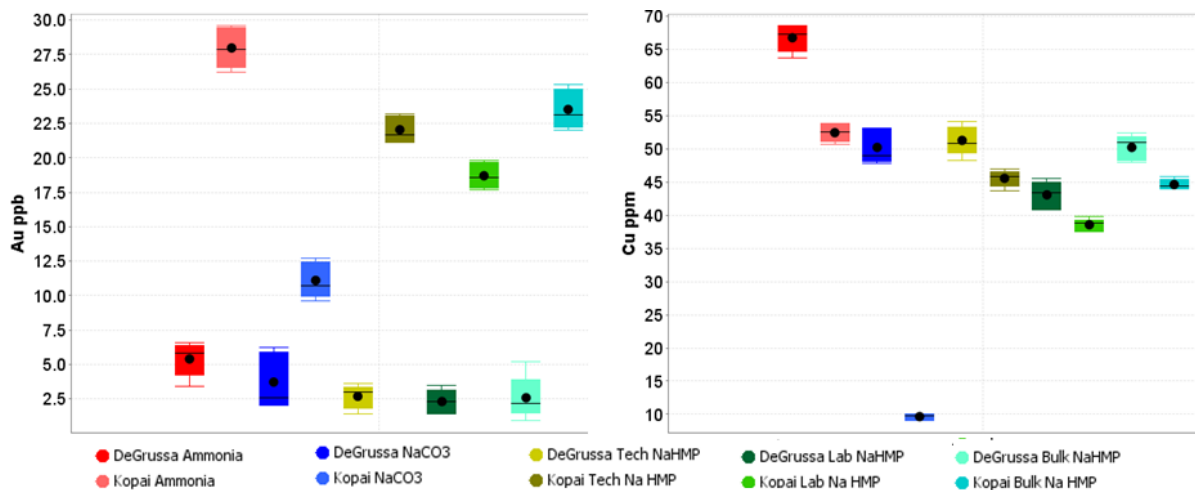
**Table 11. MANOVA comparisons of dispersant types and grades with respect to Au concentration using two reference soils and 5 replicates of each treatment. All Pairwise Multiple Comparison Procedures (Holm-Sidak method). Overall significance level = 0.05.**

Comparison	Diff of Means	t	P	P<0.050
<b>Degrussa and Kopai soils n=10</b>				
Ammonia soln vs. Na Carbonate	9.28	15.787	<0.001	Yes
Ammonia soln vs. Lab gr Na-hex	6.19	10.53	<0.001	Yes
Bulk commerc Na- hex vs. Na Carbonate	5.64	9.594	<0.001	Yes
Technical gr Na-hex vs. Na Carbonate	4.95	8.421	<0.001	Yes
Ammonia soln vs. Technical gr Na-hex	4.33	7.366	<0.001	Yes
Ammonia soln vs. bulk commerc Na-hex	3.64	6.192	<0.001	Yes
Lab gr Na-hex vs. Na Carbonate	3.09	5.257	<0.001	Yes
Bulk commerc Na-hex vs. Lab gr Na-hex	2.55	4.338	<0.001	Yes
Technical gr Na-hex vs. Lab gr Na-hex	1.86	3.164	0.006	Yes
Bulk commerc Na-hex vs. Technical gr Na-hex	0.69	1.174	0.247	No
<b>DeGrussa soils only n=5</b>				
Ammonia soln vs. Lab gr Na-hex	3.1	3.729	0.006	Yes
Ammonia soln vs. bulk commerc Na-hex	2.82	3.392	0.014	Yes
Ammonia soln vs. Technical gr Na-hex	2.72	3.272	0.018	Yes
Ammonia soln vs. Na Carbonate	1.68	2.021	0.302	No
Lab gr Na-hex vs. Na Carbonate	1.42	1.708	0.452	No
Bulk commerc Na- hex vs. Na Carbonate	1.14	1.371	0.625	No
Technical gr Na-hex vs. Na Carbonate	1.04	1.251	0.626	No
Technical gr Na-hex vs. Lab gr Na-hex	0.38	0.457	0.957	No
Bulk commerc Na-hex vs. Lab gr Na-hex	0.28	0.337	0.931	No
Bulk commerc Na-hex vs. Technical gr Na-hex	0.1	0.12	0.905	No

Another test of the dispersants looked at the effectiveness of disaggregating the clays and fine particles and the % recovery. This is a % of the total bulk soil for comparison of the dispersants and not the total % of the fine fraction that is recovered. Table 12 indicated that the Na-hexametaphosphate was a much better dispersant, even if the ammonia solution had extracted more Au in this separation. With the laboratory grade reagent significantly more expensive, using one of the lesser quality Na-hexametaphosphate could be beneficial (Table 12), but the amount used in the technique is small, so the better grade material is preferred. Another consideration is that the better quality reagents (laboratory and technical grades) were much better to make up the bulk solutions, with an expected increase of impurities in the bulk commercial grade material.

**Table 12. Comparison of dispersants for weight of material dispersed and the approximate economic costs. nd = not determined.**

Dispersant	Avg. < μm wt. (g)	SD	% recovered	Cost \$ per eq. unit
Laboratory grade Na-hex	2.5	0.39	12.5	0.84
Technical grade Na-hex	2.1	0.54	10.5	0.5
Bulk commercial grade Na-hex	2	0.33	10	0.07
Ammonia solution	1.2	0.4	6	nd
Sodium carbonate	0.8	0.48	4	nd



**Figure 13. Box plots comparing the five different dispersants and two soil types compared to the concentration of Au and Cu assayed in the fine separates. n=5.**

#### 4.1.6 Test 6 Separation comparison

The process of separation requires Na-hexametaphosphate to maximise the fine fraction recovery in the soils used in this study. Ultrasonics alone were not effective in consistently separating the fine materials (not shown). Testing of ultrasonics in addition to dispersants and rinsing of dispersants was completed. Rinsing did show some slight variation to the other treatments (none and ultrasonics) for some elements, but it was a minor. The Boddington site with greater organics, was strongly influential in the statistical tests. Four replicates of five reference samples using three treatments (dispersant only or “none”, dispersant + ultrasonics “ultrasonics” and dispersant + two DI water rinses “rinsed”) were tested using the MAR04 analytical method from Lab West. A multiple analysis of variance (MANOVA) test showed no significant differences in the results for all treatments tested for Au if the Boddington samples were excluded (Table 13). The difference in the mean values among the different treatment is not great enough to exclude the possibility that the difference is just due to random sampling variability after allowing for the effects of differences in sample sites. There is not a statistically significant difference for Au ( $P = 0.765$ ). The effect of different sites does not depend on what level of treatment is present. There is not a statistically significant interaction between site and the treatment ( $P = 0.928$ )

Only minor differences for most metals and metalloids including Cu were observed (Table 14). Consistently there was no significant difference between “none” and “ultrasonics” indicating that the use of ultrasonics in the Ultrafine+ workflow adds unnecessary time to the separation method (Table 13 and Table 14). Not surprisingly the MANOVA also showed significant differences between all sites and nearly all elements – since the samples are from distinct regions this is to be expected and any similarities is purely coincidental.

**Table 13. MANOVA comparisons of fine fractions separation treatments for Au (Boddington excluded). All Pairwise Multiple Comparison Procedures (Holm-Sidak method). Overall significance level = 0.05.**

Comparison	Diff of Means	t	P	P<0.050
Ultrasonics vs. Rinsed	0.47	0.684	0.873	No
None vs. Rinsed	0.395	0.575	0.814	No
Ultrasonics vs. None	0.075	0.109	0.914	No

**Table 14. MANOVA comparisons of fine fractions separation treatments for Cu (Boddington excluded). All Pairwise Multiple Comparison Procedures (Holm-Sidak method). Overall significance level = 0.05.**

Comparison	Diff of Means	t	P	P<0.050
Rinsed vs. None	1.455	2.918	0.016	Yes
Rinsed vs. Ultrasonics	0.805	1.614	0.214	No
Ultrasonics vs. None	0.65	1.304	0.199	No

#### 4.1.7 Test 7 Comparison to other commercial separation techniques

Although the workflow developed as part of the Ultrafine+ service is the first of its kind, there are other commercial analytical laboratories that will perform a clay or fine particle separation and geochemical analysis. Both Bureau Veritas and ALS provide this service (both in their Vancouver laboratories) and to better understand the results obtained from our study the same reference samples were sent to ALS to compare results. We were not aware of the BV option until recently, hence only one laboratory comparison was made. The ALS method used was ME-MS41L which is an aqua regia digestion with “super trace” best detection limits available using ICP-MS. This method is very comparable to the technique employed for the rest of the analysis in this project (MAR-04 from Lab West). The ALS results did not use the OREAS 250 standard as it was not a sufficient amount and the E-MS41L technique has a caveat that Au determinations are semi-quantitative due to the small sample weight used (0.5g). Regardless of the caveat, the duplicate data was excellent, with an average %HARD of 3 and most elements <2% among our blind duplicates (n=3). One initial observation with the ALS extraction is the initial sample size is much larger (~300 g) yet the average % material recovered for analysis was 1.1% which is much less than the 3.75% using the methods described in this paper (data not shown).

Gold (Figure 14) and Cu correlate very closely between the two labs and the trace elements are similar. Differences are evident in the major elements and this is likely a product of the dispersants used in the separation process. The difference in the mean Au concentrations from the different laboratory methods is not great enough to exclude the possibility that the difference is just due to random sampling variability after allowing for the effects of differences in sites. There is not a statistically significant difference ( $P = 0.455$ ) for Au analysis between the laboratories. This is not the case for Cu, with the Ultrafine+ method reporting an average 24% more Cu. The linear trend is consistent however, so the methods relate closely even if the data is not truly the “same” as it was for the Au. Correlation analysis for Cu was strong ( $R^2 = 0.95$ ). These closely aligned results are expected as the initial separation method is similar and quite standard practice with the exception of dispersants used that may influence some elements.

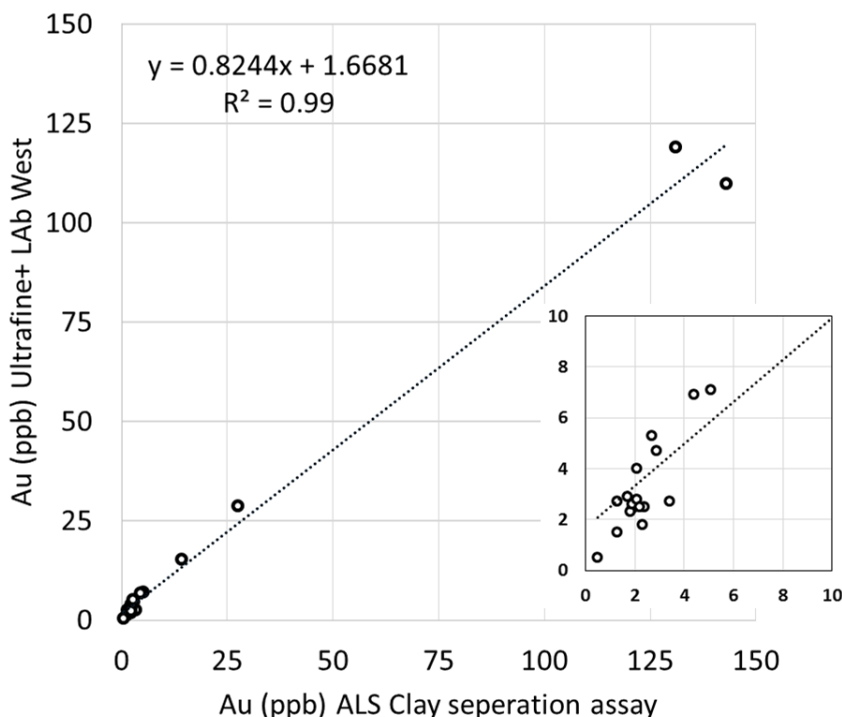


Figure 14. Scatterplot of the Au concentration extracted from the Ultrafine+ method at Lab West and the Clay separation at ALS that correlate well and show a little more variation at the low end (inset).

#### 4.1.8 Test 8 Variation in spectral reflectance from fine fraction materials

Testing to optimise the spectral mineralogy was also conducted. Using the Bentley 3 <2 µm sample 0.2 g, 0.3 g, 0.45 g, 0.55 g and 1.0 g were weighed out and measured with the ASD between each cumulative addition of more material. Each sample measurement was replicated six times. Although the 0.2 g sample (the preferred amount of material for chemical digestion analysis) was only a very thin layer of sample in the sample holder and light was visible from underneath when placed on the vertical upward shining light of the muglight apparatus, the spectral reflectance results were highly comparable to the 1.0 g amount of sample (Figure 15). The standard deviation varied with the different sample weights, with the 0.2 g sample having the most variance

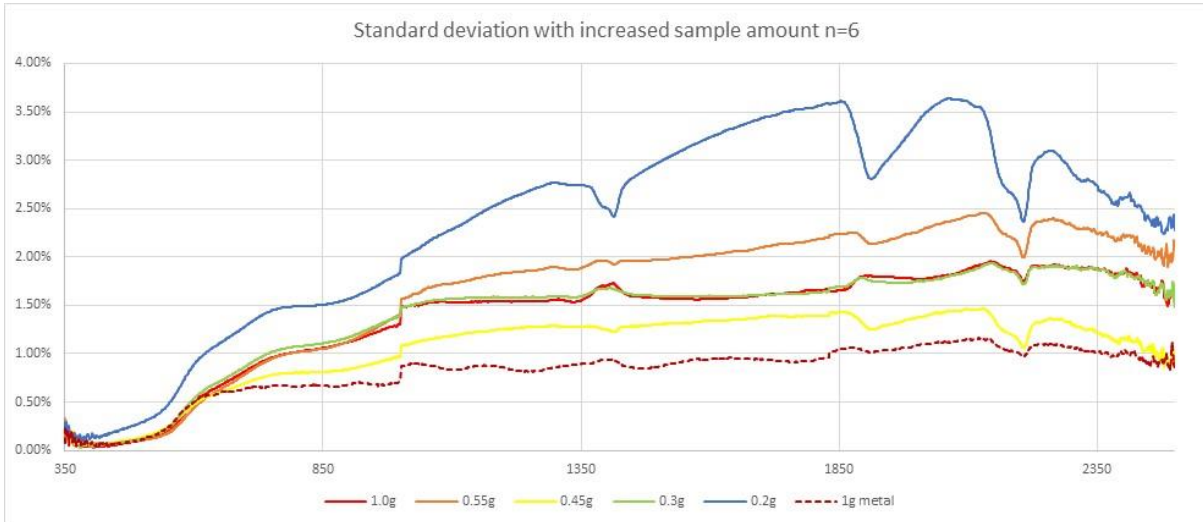


Figure 16

Error! Reference source not found.) between measurements (due to not filling the complete field of view measured by the FieldSpec4 when using the muglight). Overall, there was confidence that 0.2-0.5g of sample could be measured consistently with the ASD FieldSpec4 using the muglight and sapphire glass spectral sampling cups. However, it is noted that removal of the sample from the tubes and placing in the spectral sampling cups (sapphires glass bottom) was required and then the sample was tipped back into the tubes. An additional time restriction of cleaning the spectral sampling cups was also found to be problematic. Thus a new method for spectral measurement was proposed for the UltraFine+ method.

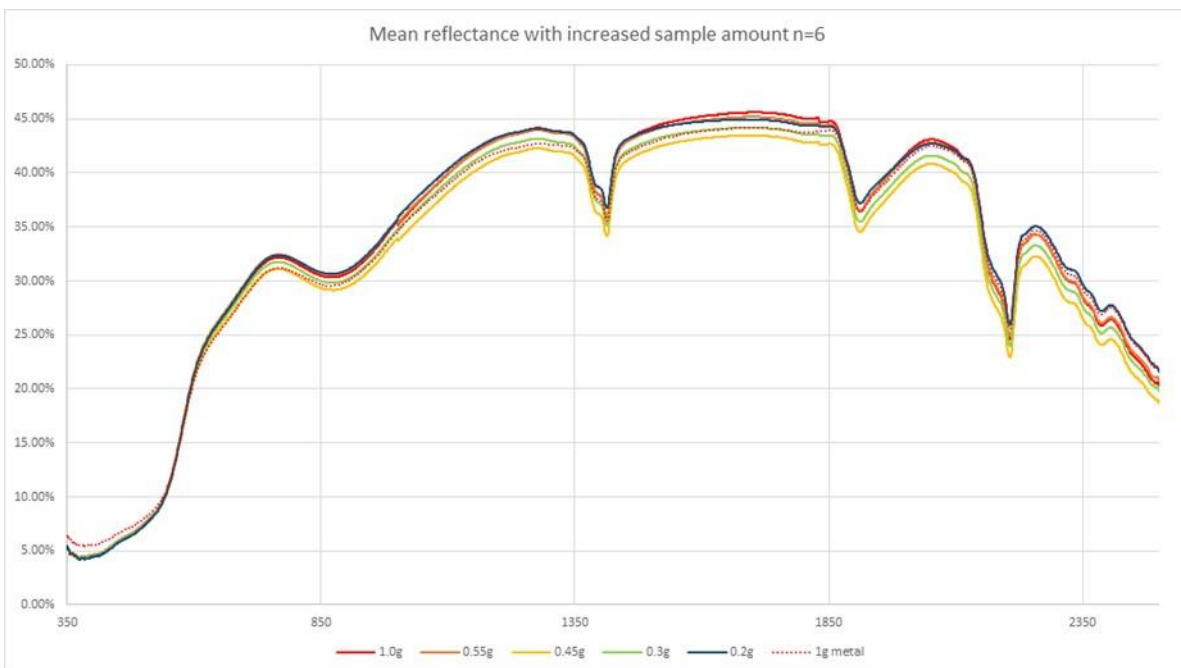
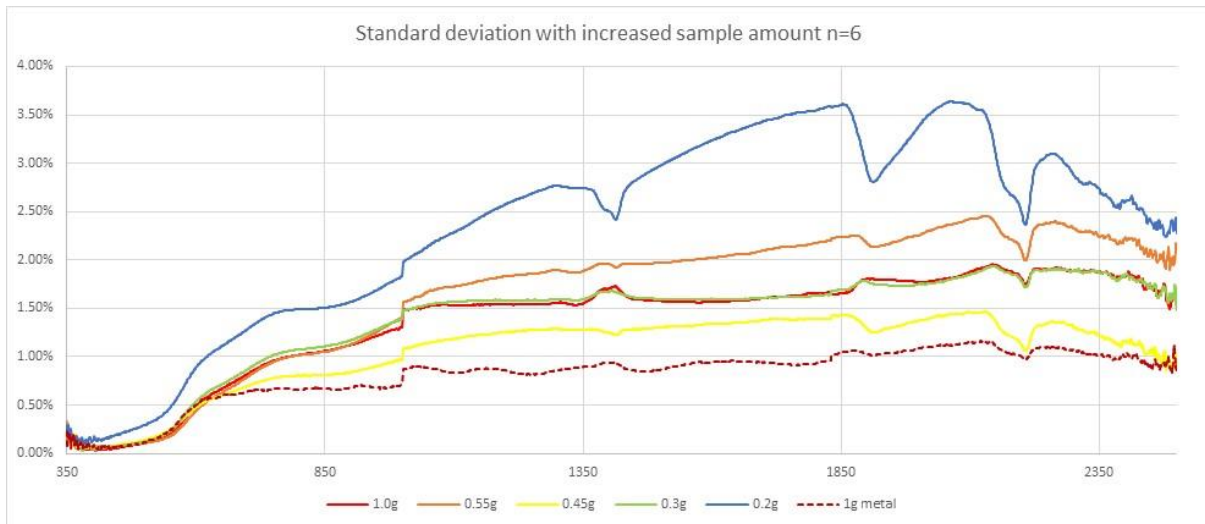


Figure 15. The Bentley 3 sample <2 µm measured with an ASD FieldSpec4 using different sample weights of 0.2g, 0.3g, 0.45g, 0.55g and 1g with a muglight and sampling cups. Each sample measurement was replicated six times.



Figure

16. The standard deviation of the measurements performed in Figure 15 above at the different spectral wavelengths.

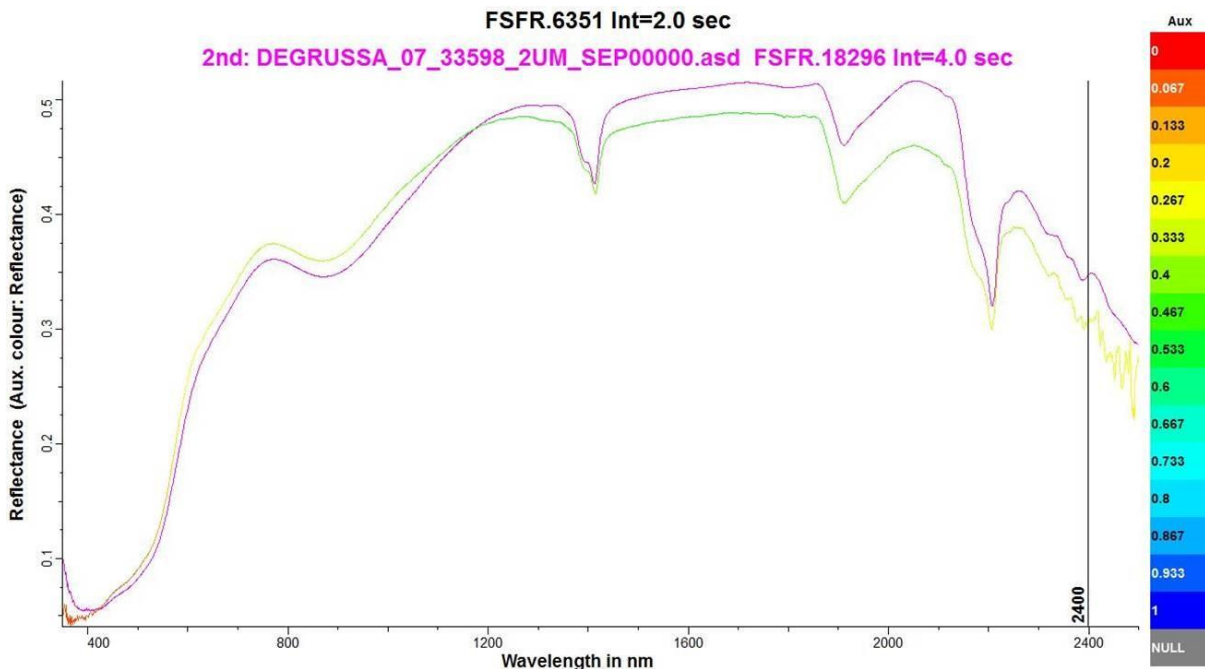


Figure 17. The standard set up and spectral analysis using the ASD (pink line) with a lot of sample in a muglight sample cup compared with the new test probe method (rainbow coloured line).

As a result of the additional time required with sample transfer to the sampling cups, a new fibre optic dip probe was tested. A small (~0.5g) amount of <2 μm DeGrussa sample was placed in a conical centrifuge flask and was tested in comparison to large (3g) amount of sample using the normal ASD muglight sample cups. The spectra of the small sample had slightly more noise in the longer wavelength regions (>2400 nm), where the energy is starting to get weak (Figure 17Error! Reference source not found.). However, this region is not used much for clay mineralogy and is unlikely to be influential for identifying the presence of the dominant regolith minerals reported in the samples.

Spectral measurements using the ASD and FTIR were collected on the various sieved size fractions (>2000 μm, 2000-250 μm, 250-180 μm, 180-125 μm, 125-61 μm and <61 μm, as well as the < 2 μm separation. The spectral results found the >2000 μm fraction to typically contain the most variation within a site due to the presence of large rock fragments, whereas the spectra of the size fractions from 2000-250 μm to <61 μm were largely similar. The <2 μm spectra were noticeably different from the <61 μm. Pulverisation of the <2 μm separation samples caused the spectral features to become less distinct and more rounded in shape, making identification of the mineral phases more difficult.



The mineralogy of the bulk samples identified the presence of hematite, goethite (VNIR), kaolinite, montmorillonite (SWIR), quartz (FTIR). Minor carbonate, feldspar were seen in some samples using the FTIR. Gibbsite was the dominant SWIR mineral in the Boddington samples. On first glance of the spectra of the bulk samples, besides the Boddington samples, there appears to be little difference in the spectral features. However, the use of scalars to calculate the depth and wavelength position of absorption features identifies the samples containing stronger iron oxide and kaolinite features, as well as those with higher water content.

The relative proportions of hematite to goethite was spectrally estimated using the wavelength of the ferric oxide absorption feature around 850-950 nm, with shorter wavelengths representing a greater proportion of hematite and longer wavelengths more goethite. The relative abundance of these iron oxide/oxyhydroxides (FeOx) was estimated based on the depth of this feature. Although the XRD results for Boddington1 shows a large amount of hematite (31.2%), the VNIR spectra show almost no FeOx absorption, which could be due to the FeOx minerals being present inside the cemented matrix of the weathered material (pisoliths), rather than present as coatings on quartz grains, which is the typical form for soils. In the bauxitic Boddington samples, the grains could be coated in gibbsite. The <2 mm bulk samples from Kintore, Moolart Well, Bentley, DeGrussa, Kopai and NSR all displayed iron oxide XRD concentrations of >4% and typically exhibit absorptions in the reflectance spectra in the iron oxide region. The samples where XRD identified greater content of goethite (i.e. Kintore, DeGrussa and Bentley) also correspond to samples with longer wavelength feature for FeOx (i.e. >895 nm), indicating that the wavelength position of the iron oxide is a good indicator of the proportions of goethite to hematite.

All <2mm bulk samples displayed kaolinite absorption features (asymmetrical 2207 nm absorption with inflection at ~2170 nm), with Kintore and Kopai samples displaying deep 1900 nm and asymmetric 1400 nm absorptions corresponding to the presence of water. This may be due to the presence of an aluminium bearing clay mineral (montmorillonite).

The FTIR measurements of the bulk <2 mm samples found the presence of an absorption at ~2550 nm (just outside the wavelength region of the ASD) which is related to the presence of carbonate for the Kintore and Kopai samples. These samples also display a strong asymmetric absorption due to carbonate at 3970 nm and a peak at ~6400 nm, which has been used to identify the presence of calcite. All samples display prominent quartz absorptions at ~4465 nm and 8640 nm except the Boddington, Kopai and Kintore samples, which have only small absorptions. These samples were found to have lower amounts of quartz in the XRD analysis. All samples have an absorption feature at 9000 nm which could either be attributed to kaolinite or feldspar, however, all but the Boddington and Kopai samples have a 10960 nm peak, which would indicate the presence of kaolinite.

The ASD spectra for the <2 µm samples displayed variation in the iron oxide, kaolinite and water absorptions. The Kintore sample was very different in appearance and possessed deep water features, with absorptions similar to a sulfate mineral, possibly gypsum. The XRD results for the Kintore samples found sodium chloride and Thenardite (sodium sulfate).

Similarly to the bulk measurements, the samples with a longer hematite-goethite wavelength index corresponded to the higher XRD goethite abundances. The spectra for Kopai and Boddington are different in their appearance to the other samples which are kaolinite dominated. The Boddington <2 µm samples display gibbsite absorptions and minor kaolinite, whereas the Kopai has a spectrum more like an aluminium smectite. It also has a weak absorption in the 2335 nm region, which could be carbonate. The spectra for Kopai also have a strong decrease in reflectance towards 2500 nm, which could be due to a carbonate absorption at longer wavelengths than the ASD wavelength coverage.

The FTIR spectra for the <2 µm samples do not contain any quartz or feldspar features and are dominated by kaolinite. The exception are the Boddington, Kintore and Kopai samples. The Kintore spectra have an unusual 4735 nm absorption feature, which could be a sulfate, whereas the Kopai samples have a 3970 nm carbonate absorption. Owing to the fine grain size of the carbonate in the <2 µm samples for Kopai, the carbonate spectrum is dominated by volume scattering in the >6500 nm region. The Boddington spectra are weak and display numerous broad absorptions and peaks, but do not display diagnostic kaolin, goethite or gibbsite in the TIR wavelength region.

## 5 Discussion

The study in the East Wongatha region of WA showed that the Au was hosted in different size fractions (Noble *et al.*, 2013). Depending on which size fraction was viewed spatially, a number of different targets were identified i.e., exploration priorities changed based on the size fraction analysed. If applied to industry operations, significant expense and effort could be directed in very different areas depending on which size fraction was analysed. From this preliminary research the question is raised: is this important for exploration, and if so, how many similar samples have been overlooked in bulk analyses by the exploration industry? These questions clearly demonstrate it is important to be able to justify which size fraction presents the best targets in a given setting. The limitation of the East Wongatha study was the small number of test samples and the limited variation in scale (proximal versus distal versus regional with limited context to deposits).

The results of this research demonstrates the value of concentrating and analysing the fine fractions (or any fraction) separately, it provides a more level comparison. This effect is most strongly evident in the finest fractions. For example, if the coarse fractions have minimal Au, a sample may have 10 ppb Au but only have 2% of this fine material. If another sample in the same survey contains 10% clay and is 20 ppb Au, the likely scenario is that the explorer is drawn to the greater number, however, if we adjust for the clay proportion, the first sample had an equivalent 100 ppb Au to the second sample, and potentially is a 5x stronger anomaly that is overlooked. This action may not always be the best choice, but it is this adjustment that needs to be carefully considered in the exploration industry, and has been principally ignored. The same could be said for stronger integration of physico-chemical parameters and mineralogy that greatly influence the soil, regolith and sediment chemistry. The UltraFine+ workflow developed and tested with these key reference samples is a solid base to develop this application into future exploration. To better evaluate this approach in the field Noble *et al.*, 2018a has applied this method to a range of prospects and (re-)assaying regional surveys samples from the Geological Survey of Western Australia.

This approach has been promoted by others in the past, but the combination of separation and chemical analysis with a range of other data in the same workflow is an advancement. The separation of finer materials has been used in different exploration environments (Van Geffen *et al.*, 2012; Robertson, 1999; Carlson, 2016; Stewart *et al.*, 2017; Sader *et al.*, 2018) and all have had some degree of success. More routinely geochemists are looking at the <63  $\mu\text{m}$  fraction that is easily dry sieved e.g. Anand *et al.* (2014) shows the <53  $\mu\text{m}$  had much greater concentrations of Au over the arid Moolart Well deposit whereas the commonly used <250  $\mu\text{m}$  soils were ineffective, whereas others have showed benefit for the <63  $\mu\text{m}$  in till settings for base metal, uranium and REE exploration (Hashmi *et al.*, 2018; McClenaghan & Paulen, 2018).

The results of our study support the application of this fine separation for base metals and Au, with a significant enrichment in both. The average increase in Au and Cu using the <2  $\mu\text{m}$  fraction is 188% and 195% increase, respectively, in the bulk reference samples compared to the other fractions. The East Wongatha study by Noble *et al.*, 2013 showed samples are predominantly coarse materials, and initially report Au concentrations of less than detection (0.5 ppb) up to about 30 ppb. This was very much aligned with the bulk reference samples studied in this paper. When the fine fraction is concentrated the detection limits become obsolete, as the concentrations are commonly well above this. The results for the new testing support the increase in concentrations, but not to the extent shown in the earlier study, where Au concentrations were in some cases an order of magnitude or more greater in the < 2  $\mu\text{m}$  fractions.

The enrichment of Au and Cu is certainly valuable, the benefit for Au is greatly supported in the <2  $\mu\text{m}$  fraction with the removal of nugget effects. Micro- and nano-particulate “invisible” Au has been problematic to study (Hough *et al.*, 2008). One particular observation was that nanoparticulate populations seem to increase in number and decrease in size as the detection/visualisation technology improves (Hough *et al.*, 2008; 2011; Noble *et al.*, 2009). The populations appear almost fractal in nature and no research has evaluated what this means for exploration. For both resource definition and mineral exploration Au is notoriously difficult to analyse due to its “nuggety-nature” (Stanley, 2006). The results of our study show no “nuggety” results using the <2  $\mu\text{m}$  fraction and this is supported by other results published by Arne & MacFarlane (2014) who removed nugget effects in stream sediment samples by separating and analysing the <4  $\mu\text{m}$  material in approximately 10 samples as a demonstration. The results of our study potentially contradict or at least put a limit on the push to smaller size fractions. We did not observe a marked benefit from separating to the sub-

micron level, even though Hough *et al.* (2008; 2011) show more Au in these morphologies. Our results indicate that the <2 µm fraction captures all this data effectively, and the complications with separating to smaller fractions (0.75 µm and 0.2 µm) was not required.

Sader *et al.* (2018) found the <2 µm fraction has significantly better anomaly to background contrast ratios for Au and Ag (37% and 16%, respectively) compared to the <180 µm fraction and that the reproducibility was better, supporting the growing examples in our study and others where nugget effects have been reduced or removed by using smaller particle sizes (e.g. Arne & MacFarlane, 2014).

Dispersants were important for the disaggregation of WA soils and not all performed the same. Sodium hexametaphosphate was preferred and this was also shown in fine fraction separation and testing comparing Acumer™ and Calgon® (commercial grade Na-hexametaphosphate) for sediments related to uranium exploration (Kyser, 2015 unpublished). The results showed the Acumer™ extraction did not successfully disaggregate particles and with similar centrifuge times to the Calgon® treated soils, and this resulted in more coarse material being included in the final digestion. Other evidence suggests that in till soils or other heavy clay materials, dispersants are not necessary and may provide further complications (Sader 2018 *pers. comm.*) based on work by the commercial laboratory, Bureau Veritas. This seems plausible for high clay soils, but is definitely not the case for the soils used in this study and representative of many semi-arid climate soils. Another difference observed in this study compared to the other commercial offerings was the use and recovery of fine material from a small initial bulk sample. Typically, 20-40 g were used for the UltraFine+ workflow with recovery of 0.5-2 g in many of these soils. The UltraFine+ method is nearly an order of magnitude less starting material compared to the 300 g required by ALS for their clay separation and analysis. Presently, we are unaware of the starting soil weight used by Bureau Veritas in their clay separation procedure.

From this initial research into particle size separation, the UltraFine+ workflow evolved to produce results for 40 element concentrations, spectral mineralogy, particle size distribution and estimates of other key physicochemical parameters such as pH, EC, specific surface area and cation exchange capacity. The next iteration of this workflow will aim to add organic carbon estimations to fully characterise the major metal exchange phases in soils that are likely influencing anomalous metal signatures. With this step-change in data offering as a standard analytical procedure, the exploration industry will need to be able to integrate many variables for exploration assessment. Another likely, flow-on effect, will be the application of machine learning and integration of other spatial data to produce exploration indices and other ranking tools to assist the used. With the next development of this workflow, it is likely that the person submitting their samples for analysis e.g. UltraFine+ will be able to click a button to add interpretation and by providing spatial coordinates, freely available data along with the output of the analytical procedure will be used with machine learning to generate additional rankings, estimations of uncertainty and a range of additional information that has not been available to the general exploration geologist.

## 6 Conclusions

The study into fine soil separation (<2 µm) for mineral exploration aimed to greatly refine analytical methods and enhance surface exploration success. By analysing fine fractions (<2 µm) we produced reproducible, reliable results, with bigger concentrations. Key benefits were the removal of nugget effects (in Au) and the challenges with detection limits in materials that are dominantly quartz sand. Testing sub-micron fractions showed that although <0.2 µm fraction was slightly different to the <2 µm and 0.75 µm fractions there was not significant additional value in going to this extra effort, so the <2 µm is favoured as the best fine fraction separate. The overall method development showed ultrasonics were not required, a dispersant was critical for solid recovery and that Na-hexametaphosphate (technical or laboratory grade) was the most effective. The developed method proved the use of a small weight for analysis was effective (0.2 g) and microwave assisted aqua regia was the best analytical method for Au. This research shows obvious benefits to using fine fractions for Au, however, there is no loss of benefit to using this fraction to analyse for base metals and other pathfinder elements, Cu and Zn were very consistently and abundantly extracted from the fine particle size fraction.

The UltraFine+ workflow produces results for 40 elements, spectral mineralogy, particle size distribution and estimates of other key physicochemical parameters in soils. Just as exploration companies (and supporting laboratories) went from using one target element to multielement interpretation, the research in this paper supports that the next step is to add numerous other parameters to the mix and the UltraFine+ workflow developed to separate fine fractions is a strong move to progress exploration in this sphere.

## 7 References

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# 8 M462 Deliverable: Part 2 – Site Studies and Regional Maps

## 8.1.1 M462 Deliverable: Part 2 – Site Studies and Regional Maps

### Application of ultra fine fraction soil extraction and analysis for mineral exploration

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### 8.1.2 Abstract:

The need to innovate to advance exploration success is ongoing and while regional geochemical surveys are common, increasing the data we collect and improving survey design has stagnated. A new adaption of existing methods known as UltraFine+ extracts the <2 µm fraction of soils and sediments, which is then analysed and combined with spectral mineralogy proxies and physico-chemical properties to improve targeting for Au and base metal exploration. We applied the UltraFine+ workflow to a number of small orientation site studies in Western Australia, and reprocessed archived regional soil samples from the Geological Survey of Western Australia to test the method. The orientation program (~200 samples) involved samples from the Leonora and Sir Samuel 1:250k map sheets, an area that hosts known major Au and base metal deposits. We then applied this approach to the Kingston 250k map sheet area, analysing a further 300 samples in a region on the Yilgarn Craton margins that is essentially greenfields, as there has been little exploration and the original geochemical survey data was heavily censored due to the dominance of transported regolith dominated by quartz-rich sand. The archived samples were specifically selected soils (sheetwash or sand plain) with a component of Quaternary/Tertiary transported cover. Results at a number of small orientation test sites were promising. Importantly, the study revealed a marked decrease in censored results for Au (63% to 10% below detection limit). The new data show geochemistry and some example indices for mineral exploration and lithology mapping, as well examples map products of new interpretations using the additional spectral mineralogy proxies or particle size variation. The application of the <2 µm particle size separation and the UltraFine+ workflow importantly demonstrates the additional value from (re-)assaying regional soil and sediment samples to generate new targets and improve regional geochemical maps. This is an exercise that can be applied to new greenfields surveys, and when exploration budgets are lean, applied to historically collected samples.

**KEYWORDS:** *clay, spectral mineralogy, regolith, ultrafine+, particle size*

## 9 Introduction

Traditional, near surface geochemical techniques have been reasonably effective in mineral discovery but to explore in deeper transported cover and find new resources, as demanded by a growing global population, innovative methods are required. Recently a new method or adaption of methods to extract the ultrafine fraction of soils and sediments, assay the geochemistry and combine this is with additional spectral and physico-chemical properties (Noble et al., 2018a) has shown some major benefits for Au and Cu exploration. This UltraFine+ workflow uses less sample, provides more data, a reduced nugget effect with solid reproducibility, and a small proportion of Au below detection due to the ability to avoid dilution from the bulk Si-rich matrix. However, it has not been applied to exploration examples such as orientation studies or regional geochemical surveys.

Regional geochemical surveys are conducted routinely with sample densities from one per km<sup>2</sup> to one per 100 km<sup>2</sup> (Salminen, 2011) by government organisations, companies and individuals in all parts of the world. Many of these surveys use traditional methods which aids comparison particularly across state and country borders or with other data sets (Min et al., 2014; Reimann & de Caritat, 2012; Smith et al., 2009).

The resulting surveys and data are instrumental in providing context and background for a range of applications, environmental baseline, remediation, agriculture, forestry, and mineral exploration. These surveys are common first-pass approaches in mineral exploration when exploring for bedrock-hosted mineralisation (Morris, 2013). However, to improve the application for mineral exploration in particular, especially with respect to cover, these results may not provide the best information of trace and pathfinder elements.

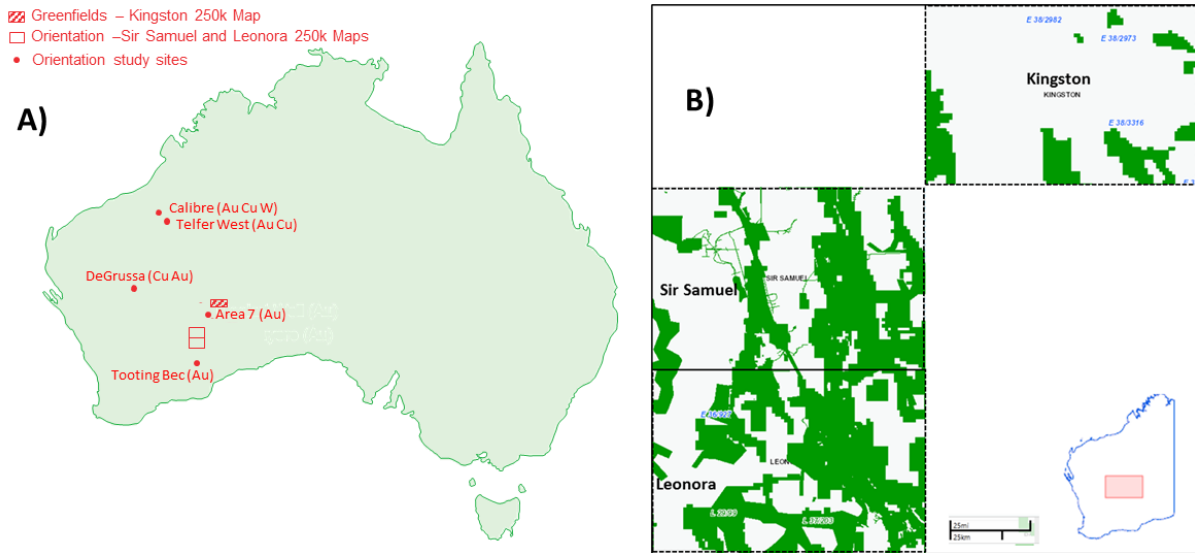
Regarding mobilisation of trace metals through cover, results show these transported elements will be small, seasonally-variable (Anand et al., 2014) and commonly positively charged. These metals and metalloids will subsequently be adsorbed to clays, organic complexes and Fe oxides as these phases are common and have the ability to exchange these charged small particles (Hawkes and Webb, 1962; Hall, 1998). Separating these small fractions (<4 µm) to improve results has been shown to be valuable (Noble et al., 2018a, 2014; Sader et al., 2018; Carlson et al., 2016; McClenaghan & Paulen, 2018; van Geffen et al., 2012).

By adjusting methods for regional surveys, significant improvements can be made for mineral exploration. The Au data for a regional stream sediment survey in Canada was deemed useless as the nugget effect was too extreme and reproducibility almost non-existent in the bulk sediments. However refining the sample particle size to the <4 µm demonstrated greatly improved reproducibility on a select few samples that could be scaled up to the survey (Arne & MacFarlane, 2014). Also, by using newer technology and analytical sensitivity, new targets and detecting a major deposit that was missed in earlier regional geochemical surveys, shows value in re-assaying historic samples for a stronger exploration focus (Baker et al., 2015).

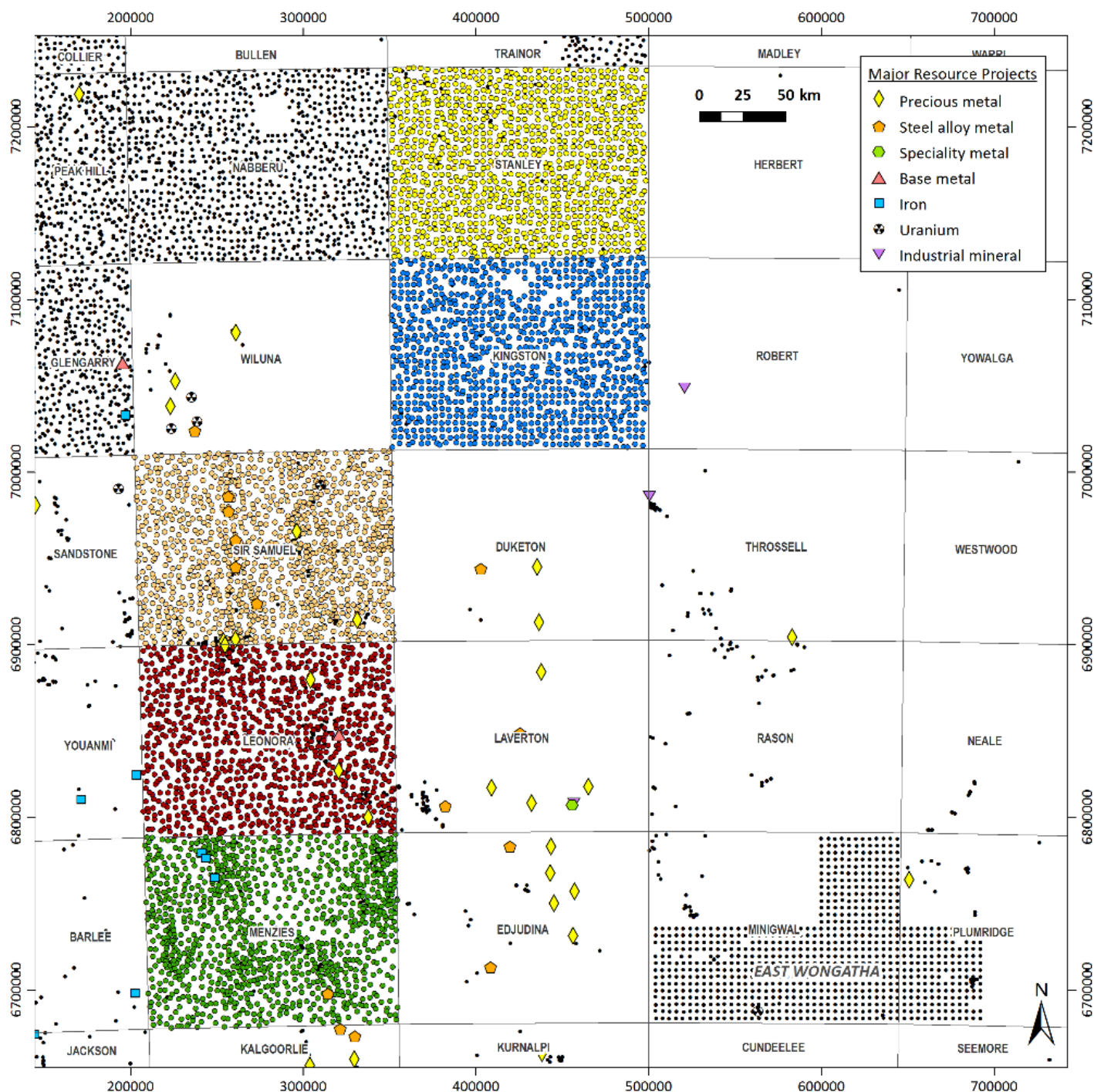
The nugget effect is well known in mineral exploration and processing. The nugget effect means that however well-mixed samples are, there will be big variations in value. This variability is most problematic where there are small-scale structures such as small-scale veining or coarse gold resulting in biased estimations for exploration potential or resource definition (Harris, 1982; Nichol et al., 1989; Dominy et al., 2001). While statistical approaches are used to minimise this effect (e.g. Stanley, 2006) there is a lot of potential to adjust the physical sample particle size down to exclude “nuggets” to be analysed and for increased reproducibility. Detection limits have often been problematic in regional surveys, with Au values below detection that result in highly skewed data, and create problems for statistical analysis (Leybourne and Rice 2013). For example, the Mount Phillips survey comprised 89% censored data of samples that were below detection for Au (Sanders et al., 1997). The results from decades previously are much more problematic with numerous key elements not analysed or detection limits that were so high the majority of data are censored and those that are reported are just around the detection limits that can cause interpretation issues. A common practice to deal with data below detection for statistical evaluation is to replace censored data with a value equal to half the lowest detection limit (Grunsky 2010; Reimann et al., 2010).



Other regional surveys of the past have suffered from censored data such as the GSWA’s Kingston 1:250,000 map sheet that was sampled, analysed and reported by Pye et al. (1996). The Kingston map extends NE of the Yilgarn Craton and into the Earraheedy Basin. The region is underexplored based on tenement uptake (Figure 18) compared to Sir Samuel (Kojan et al., 1996) and Leonora (Bradley et al., 1995) projects, SW of the Earraheedy basin. Here, there are more samples with detectable Au, some outcrop and residual regolith and numerous large and well known gold deposits in the area to keep exploring further. Figure 18 shows the location of these key regions that are part of the broader regional analysis conducted in this study. Figure 19 shows the map sheets discussed, surrounding areas, major deposits and the samples that are potentially available for further analyses.



**Figure 18. A) Location showing the major map sheet areas as well as the approximate location of orientation sites used in this study. B) The map sheets with green shading representing current active tenements (Tenement map from GeoView, GSWA accessed Sept 12, 2018).**



**Figure 19. Regional geochemical samples from GSWA data base that related to this current project and potential analysis using the UltraFine+ method. Major resource projects are mapped and note there is nothing in the Kingston map sheet.**

The large number of samples in Figure 19 all have multi-element geochemistry associated with them, but minimal additional data excluding regolith and geological description. While these are of utmost importance, adding spectral mineralogy, colour and other parameters to geochemistry is valuable and is not commonly done, and certainly not integrated. Examples of regional geochemistry and the addition of spectral mineral proxies have been tested (Reeve & Smith, 2009), resulting maps combined with geochemistry have been shown to have value in agriculture. The use of reflectance spectroscopy in digital soil mapping (pedometrics) has increased greatly in recent years, with soil carbon, pH, CEC, Eh and moisture predicted using partial least squares regression (PLSR) from spectral measurements on soils (for a recent review see Fang et al., 2018). This same approach has not really been done in mineral exploration and is eminently achievable.

Noble et al. (2018a) demonstrates an innovative shift in method development and analytical considerations with using fine soil fractions. In this paper, we apply the new UltraFine+ method to a number of exploration sites and importantly demonstrate the additional value to be gleaned from (re-)assaying regional soil samples with newer and improved methods to generate new targets and regional geochemical maps.

# 10 Geological Settings

## 10.1.1 Sir Samuel/Leonora Region

Sir Samuel lies between latitudes 27°00'S and 28°00'S and longitudes 120°00'E and 121°30'E, and includes major mining regions for gold such as Agnew, Bronzewing and Darlot, with significant Ni endowment at Leinster and Mount Keith among others. The map sheet takes its name from the abandoned mining town of Sir Samuel (Kojan et al., 1996; Figure 18; Figure 19). The Leonora region located immediately south of the Sir Samuel map sheet lies between latitudes 28°00'S and 29°00'S and longitudes 120°00'E and 121°30'E, and includes major mines for Au Sons of Gwalia (Leonora), Emu (Lawlers), Tarmoola - King of the Hills being the largest when the initial samples were collected (Bradley et al., 1995; Figure 19). The sheet is named after the biggest population centre in the region – the township of Leonora.

The geology of this area is typical of the northern Yilgarn Craton and is comprised of variably distributed Archaean-aged (2.6 Ga) granites and granitic gneiss with extensive NE trending, elongate greenstone belts (Williams 1975; Myers 1997). The dominantly mafic and ultramafic volcanic greenstones occur in a series of belts including the well-endowed Norseman-Wiluna belt, along with the Yandal and Dingo Range belts (Myers & Hocking 1998; Morris & Sanders 2001). The elevation ranges from 437 to 612 m a.s.l. (Bradley et al., 1996). The landscape topography is gently undulating, with upland areas that have extensive ferruginisation or silcrete armouring that lead to erosional breakaways and stony plains, with much of the lower, broad depositional plains of colluvial/alluvial sheetwash plains, sand plains and drainage channels.

The soils are dominated by kaolinite and quartz with small amounts of goethite, hematite and muscovite, with weak structure and show little development of distinct A and B horizons (Anand & Paine, 2002). The annual rainfall is highly variable and approximately 200-210mm. Rain falls more frequently in the first half of the year and tied to northern cyclonic activity (Anand & Paine, 2002; Bradley et al., 1996). The flora is dominated by mulga (*Acacia* sp.) shrub-lands with spinifex (*Triodia* sp.) on sandplains, and *Eucalypt* sp. in the ephemeral drainages. In the broad saline drainage channels, halophilic saltbush, bluebush and samphire species are present.

## 10.1.2 Kingston Region

The Kingston map sheet lies between latitudes 26°00'S and 27°00'S and longitudes 121°30'E and 123°00'E, and includes no major mining regions compared to the other regional sites used. Mount Eureka on the western edge of the greenstone belt of the same name produced 20 kg of gold in the early 1930s as the only record of sizeable metal extraction (Pye et al., 2000; Figure 18; Figure 19).

The geology of this area includes the NE Yilgarn Craton with Archaean-aged (2.6 Ga) granites and granitic gneiss and greenstone belts (Williams 1975; Myers 1997). This is overlain by 5 km thick sedimentary rocks of the Proterozoic 1.6-1.8 Ga Earahedy Basin. Other dolerite and Patterson formation rocks are present to the north, but do not cover the area studied here. The Frere Formation covers the mapped basin sediments in the southern section of the Kingston map sheet and this unit consists of shales interbedded with iron formation and chert, and uncommon lenses of limestone (Pye et al., 2000).

The elevation ranges from 440 to 600 m a.s.l. The landscape topography is gently undulating, and is broadly classified into four regions by Pye et al. (2000): 1) upland areas corresponding to resistant Proterozoic rocks, Permian rocks, and Archaean greenstone; 2) areas of sandplain and aeolian material developed over Archaean/Permian rocks, particularly in the southwest; 3) broad sheetwash plains and valleys; and 4) areas proximal to and including salt lakes.

The soils are commonly more poorly developed, with little horizon definition and pedogenesis, and rarely show development of distinct A and B horizons. Soils are siliceous, and individual commonly coated with clay-rich or iron-rich material (Pye et al., 2000). The annual rainfall is highly variable and approximately 200-250mm. Rain falls more frequently in the first half of the year and tied to northern cyclonic activity although winter depressions may also provide precipitation to the region (Anand & Paine, 2002; Pye et al., 1996). The flora is dominated by mulga (*Acacia* sp.) shrub-lands with spinifex (*Triodia* sp.) on sandplains, and Mallee *Eucalypt* sp. in other areas. The southern third of the map sheet and many of the samples used in this study

are on a sandplain regolith setting. Similar to the Sir Samuel area, halophilic saltbush, bluebush and samphire species are present in the broad saline drainage channels.

### 10.1.3 Telfer West

Telfer West prospect is located 25 km north-west of Newcrest's major Au-Cu mine at Telfer (Figure 18). The potential mineralisation zone is under 50 m to 80 m of transported cover. The cover is commonly Quaternary/Tertiary dune sands and underlying this is Permian glacial sediments that are highly weathered. Beneath the cover is some supergene Au in the weathered oxide zone. Recent and historic drilling indicates the area contains several zones of high grade gold mineralisation within a substantial volume of stockwork style gold mineralisation. Geochemistry of the Proterozoic rocks indicates elevated As, Bi and Co as potential pathfinder elements. Exploration at Telfer West is targeting Au mineralisation in a similar stratigraphy to the host units at Telfer. A dome structure has a core of Isdell Formation overlain by the Malu Formation, Telfer Formation and sediments of the Puntapunta Formation. These units are the main hosts of gold-copper mineralisation at Telfer (Encounter Resources, 2018). The vegetation comprises sparse shrub-steppe with the dominant *Triodia basedowii* ('hard spinifex') and *Eucalyptus* sp. communities, and grasslands on alluvial channel sediments (Kendrick 2001). The broad depositional alluvial/colluvial/aeolian plains have a mix of *A. ancistrocarpa* and *Triodia* sp. The climate is semi-arid with average maximum and minimum temperatures of 34.1°C and 19.4°C, respectively. The annual rainfall averages 367 mm with most rain in summer (Telfer weather station; Bureau of Meteorology 2013).

### 10.1.4 Tooting Bec

The Tooting Bec Prospect, 2 km south west of the Cannon Gold Mine and is approximately 30 km east of Kalgoorlie (Figure 18) in the in the Yilgarn Craton and part of a major Archean age greenstone belt c. 2.7 billion years old. The region is typical of the orogenic Kalgoorlie Au deposits, with the target mineralisation in strongly sheared, mafic and ultramafic rocks which are structurally controlled and strike north-easterly and dips to the west. The mineralisation is associated biotite, calcite, chlorite, albite and pyrite alteration. High-MgO basalts, komatiites and related intrusives underlie the area. Black shale horizons, monzogranite, diorite dykes are present. The prospect is covered by shallow calcareous and/or ferruginous soil, the regolith profile is shallow and it is commonly less than 20 m thick over the saprock. Calcrete nodules are common in profile. The area is semi-arid with average maximum and minimum temperatures of 25.3 °C and 11.7 °C, respectively. The rainfall averages 267 mm annually (Kalgoorlie-Boulder airport weather station; Bureau of Meteorology 2018a). The area consists of eucalypt dominated open woodland with an understorey of shrubs and grass.

### 10.1.5 DeGrussa

DeGrussa is located in the Proterozoic (c. 2 Ga) Bryah Basin that lies adjacent to the Archaean Yilgarn Craton to the south and the Archaean granite Marymia Inlier to the north, and is part of the Capricorn Orogen (Figure 18; Noble et al., 2017a). The geology has been discussed more by Adamides (1998) and Hawke et al. (2015). The dominant underlying lithology is sedimentary and volcanic rocks with argillaceous, sandstone and conglomerates (Hawke et al. 2015; González-Álvarez et al., 2015). The elevation of the area is generally between 560 to 570 m a.s.l. The deposit is high grade Cu-Au in sulphide assemblages and also has a sizeable supergene oxide resource. DeGrussa is located on the edge of a palaeochannel in a colluvial/alluvial plain. The relatively flat surface hides a more complex regolith and the overlying transported units varying in thickness from a few metres directly over DeGrussa, to c. 30 m. The palaeochannel is up to 100 m in thickness. Residual uplands occur to the east and south of the deposit. Soils are dominantly acidic, sandy with red-brown silicified hardpans and lack significant calcrete horizons except in palaeochannels. Prominent soil minerals include quartz, kaolinite and Fe oxides. The regolith in this region is similar to that of the northern Yilgarn as seen in the Sir Samuel/Leonora region, with the DeGrussa region receiving more rainfall. Approximately 240 mm of rain annually. Vegetation is *Acacia aneura* dominated shrub land with some seasonal grasses, especially in the alluvial channels.

### 10.1.6 Area 7

Located in the northern Yandal greenstone belt, the regional geology is similar to Sir Samuel, previous section. The local greenstone succession is dominated by mafic-ultramafic rocks and dolerite sills. Approximately 50 km east of Wiluna, the target mineralisation is the orogenic lode-gold style in which Au distribution is controlled by brittle fault-fractures and located in narrow, discontinuous ore lenses. Proximal alteration is characterised by a quartz-sericite-ankerite-sulphide (pyrite ± arsenopyrite ± chalcopyrite) assemblage.

Regolith cover for the sampled area is a mix of fine soils from colluvial flats, sheetwash plains and clayplans, with a transition to more coarse colluvium towards the north and eastern edges of the survey.

Lateritic duricrust is common in the region, but not at the local scale where the remaining terrain is dominated by mixed soil types derived from the deeper erosion of weathered bedrock units. Colluvium is the dominant soil parent material with poor particle sorting and angular fragments. A ferruginous hardpan is present. The depth of transported cover is likely to only be a few metres at this site, but it is not confirmed.

The climate is semi-arid with average maximum and minimum temperatures of 29.2°C and 14.3°C, respectively. The annual rainfall averages 261 mm with most rain in summer (Wiluna weather station; Bureau of Meteorology 2018b). Vegetation is *Acacia* sp. dominated shrub land with some seasonal grasses and Mallee Eucalypt sp., especially in the alluvial channels. Spinifex grasses (*Triodia* sp.) are common on sandplains.

### 10.1.7 Calibre

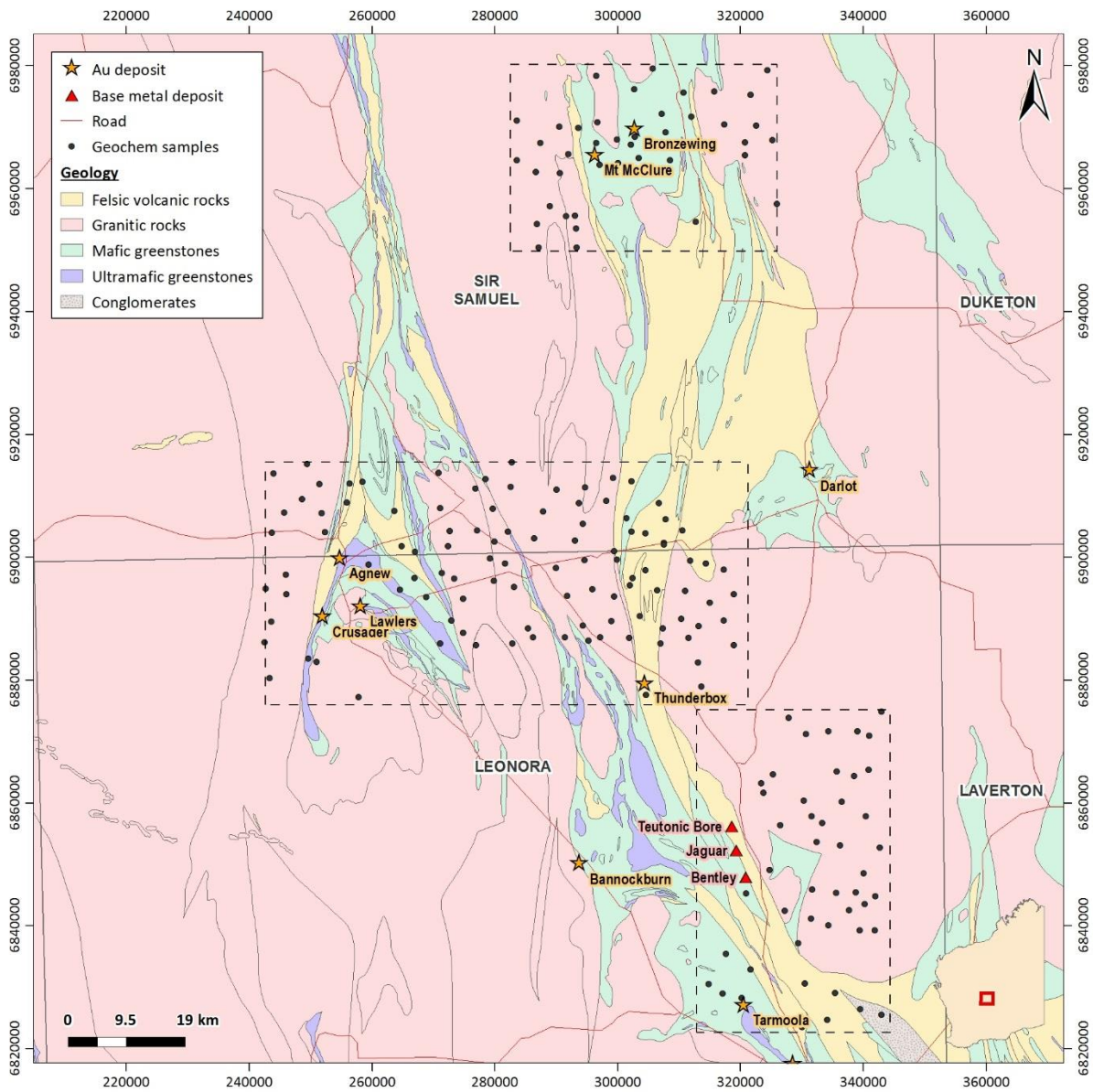
Calibre is very similar to Telfer West prospect described above. The Calibre Au-Cu-Ag-W deposit (Figure 18) is located approximately 100km north of Newcrest's Telfer Au-Cu mine in the Paterson Province. The Proterozoic basement and general geological setting and regolith is also very similar. A thick blanket of Permian glacial sediments and an upper cover sequence of Tertiary and Quaternary transported regolith. The depth of cover is approximately 80 m. Vegetation and climate conditions are also the same as those described in the Telfer West description.

# 11 Methods

## 11.1.1 Soil and regolith samples

For the large regional maps, more than 500 soil samples were sought from the stored historic materials at the Geological Survey of Western Australia's (GSWA) core storage facility in Carlisle, W.A. Using the GSWA GeoView spatial data we isolated a total of 200 soil samples that were located around major mineral deposits in the Agnew, Leonora and Yandal greenstone belt regions (Figure 20). We then determined another 300 samples in the Kingston map sheet over the Earacheedy Basin and covered approximately 50% of that 1:250,000 map sheet as a "greenfields" test case. Figure 19 shows the number of available samples in the areas considered for this study, with Figure 21 showing the final selected samples and the only mineralisation known in the area.

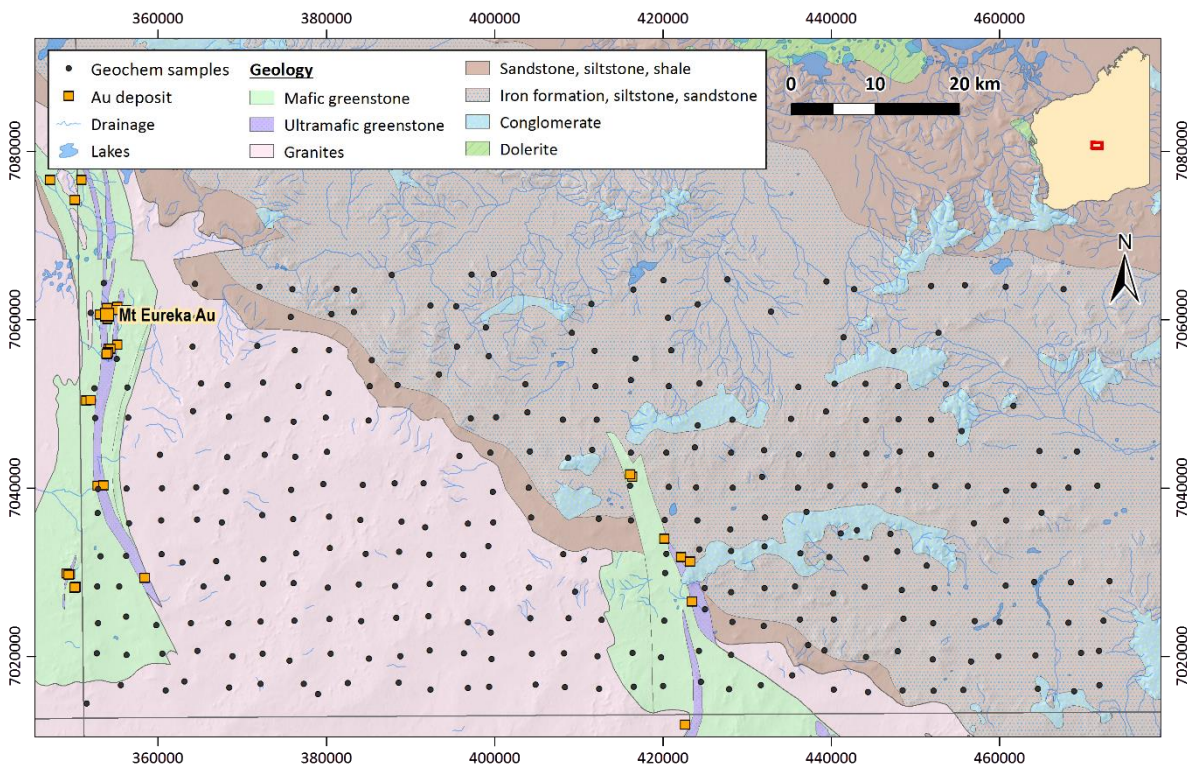
Regional orientation samples crossed two major field work programs by GSWA – the Leonora and Sir Samuel 1:250,000 map sheets that are summarised in reports by Bradley et al. (1995) and Kojan et al. (1996), respectively. Samples were filtered to proximity and then selecting only samples that were clearly not residual regolith/outcrop or stream drainages. Many of these samples were labelled as sand plain, sheetwash plain or soil. The stream sediment analysis in the Leonora and Sir Samuel region revealed some Au and base metal anomalies near the deposits and we did not want to duplicate this obvious result, rather, test the new method where previously results were ineffective when mapped in the original reports (Bradley et al., 1995; Kojan et al., 1996). Figure 20 depicts the location of the orientation samples. The Kingston map showed few detectable Au samples and no major Au resources (Figure 18), however, for consistency the samples were filtered the same as the other orientation surveys to remove vastly different sample types (outcrop regolith or stream sediments).



**Figure 20. Selected samples for the orientation test case, filtered by sample type and proximity to some prominent gold and base metal deposits and areas of interest (dashed boxes). Inset box shows the same area but with all the smaller mineralisation areas clearly depicting this as a resource-rich area (data from GSWA 2014).**

Once the ID of the samples required were ascertained, these were sub-sampled (approx. 300 g) from these old 3-5 kg sample bags. Of the 200 samples collected as part of the orientation map, <5 looked to be unusual samples and may be indicative of proximal colluvium or possibly stream drainages. These were dominantly gravels and were noted during collection. All soil samples were screened to < 2 mm for the UltraFine+ analysis.





**Figure 21. Selected samples for the Greenfield regional map covering the Kingston map sheet. The only mined gold deposit is labelled with other small, known Au mineralisation in the region.**

Other soils and regolith profile materials were collected from a number of orientation sites as part of the M462 project. These sites varied in sample number, target mineralisation and knowledge of underlying mineralisation (Table 15). Locations are noted in the geological settings and Figure 18. These soils were submitted to a commercial laboratory using the UltraFine+ method. The method is fully documented in Noble et al. (2018a). In brief, the method uses approximately <40 g of soil from the bulk (< 2 mm) material. Gravity settling following dispersion of clays is used to separate the <2 µm size fraction. The separated fine soil fraction is analysed for a number of physico-chemical properties such as pH, EC, particle size distribution, spectral reflectance mineralogy and then a microwave aqua regia digestion and analysis of the solution for approximately 40 elements using ICPOES and ICPMS.

**Table 15. Details for the various smaller orientation surveys conducted using the UltraFine+ method.**

Orientation Site	Size	Target mineralisation	Cover	Type of cover	Samples
DeGrussa	Large mine	Cu-Au	2-30 m	Transported residuum. Quaternary/Tertiary	Two soil traverses
Telfer West	Prospect	Polymetallic	30-60 m		Two soil traverses
Area 7	Prospects	Au	?		Four soil traverses
Calibre	Resource?	Cu-Au	80 m	Transported. Tertiary over Permian	Regolith profiles targeting interfaces in cover
Tooting Bec	Prospect	Au	?		Four soil traverses

### 11.1.2 Laboratory analysis

The standard UltraFine+ extraction was MAR-04 (microwave-assisted aqua regia) offered through LabWest Pty Ltd, Perth, Australia. All partial extractions of the soils were analysed for a multi-element suite of c. 40 elements using ICP-OES (Perkin Elmer Optima 7300DV) and ICPMS (Perkin Elmer Nexion 300Q). The microwave-assisted aqua regia digestion (LabWest MAR04) uses 0.2 g of soil subjected to an aqua regia digestion with a 100% mixture of 3:1 concentrated HCl: HNO<sub>3</sub>. This is heated in a closed Teflon tube in a microwave (Anton Paar Multiwave PRO Microwave Reaction System).

Visible-near infrared reflectance measurements were acquired on the bulk and separate samples using a Panalytical ASD FieldSpec4 standard resolution spectroradiometers. The ASD FieldSpec4 collects in the 350 to 2500 nm wavelength region, with a resolution of 3 nm at 700 nm and 10 nm at 1400 and 2100 nm. The spectral bandwidths of the FieldSpec4 are 1.4 nm at 350-1000 nm and 1.1 nm at 1001-2500 nm, which are resampled to 1 nm to provide 2151 bands. A calibrated piece of PTFE (Spectralon) was used as the reflectance standard and measured before each set of soil measurements by placing raising the white reference standard on a lab jack directly below the downward facing probe. The samples were measured in their plastic centrifuge vials and 40 scans were averaged into a single measurement. Processing of spectra was done using The Spectral Geologist (TSG) software to extract the main features reported as part of the UltraFine+ workflow.

Conductivity and pH measurements were made at Lab West using a TPS AQUA-CP/A Waterproof Conductivity/TDS/Sal/pH/Temperature meter of 1:5 w/w soil to water ratio slurries for all samples collected in this study.

Particle size analysis was measured using a Malvern Mastersizer 3000 in suspension using the HydroMV unit and dry bulk using the Aero S attachments. Both techniques assumed non-spherical particles and kaolinite as the dominant mineral with a refractive index of 1.45 (this is not dissimilar to the value for quartz or Fe oxides which also are common minerals with 1.51 and 1.456, respectively).

### **11.1.3 Quality control and data treatment**

For GSWA's regolith geochemistry assessment of precision a value <20% RSD (relative standard difference) was considered acceptable if the values of the elements detected were at least three times greater than the detection limit (Morris, 2013; Morris & Verren, 2001). The certified reference soil OREAS 250 was incorporated into all geochemistry analyses for this study as a standard replicate to assess accuracy. The half absolute relative difference (HARD; Stanley & Lawie, 2007) was calculated as a percentage for all analytes using duplicate samples:

$$\text{HARD} = ((\text{assay1} - \text{assay2}) / (\text{assay1} + \text{assay2})) * 100.$$

Percent HARD was determined on the standard soil to be good if it was <10% for elements of interest, and useable if <20%. Gold was <2% different to the OREAS standard, Cu <6%, Ag<10% and Zn <8% (n=26). Elements with HARD> 20% were Al, Cs, Ga, Ge, K, Sb and Ti, that were all >20%. Not surprisingly many of these are highly resistant to full digestion via aqua regia extraction or in the case of Sb were just above the 3x detection limit cut-off and so the reported 0.7 ppm compared to 0.4 ppm CRM is probably acceptable with the HARD of 26%. Aluminium and K were known to be problematic for accurate determination with the UltraFine+ method possibly due to partial digestion of the clay and are of limited use.

Elements that were all close to, or below the detection limit included Bi, In, Re, Hg, Ta, Te and W. These elements were also at similar concentrations in the full data set and are not used further.

Laboratory duplicates were analysed at a rate of 1 per 20 samples to assess precision and all elements achieved the <20% HARD criteria above. All elements except Al, Ga, Ge, Hf, Sr Ti and Zr were <10%. Gold, unexpectedly, showed a slight "nuggetty" nature if the samples near detection limits were included (HARD> 40%) in these regional samples where it had previously been much better in other testing (Noble et al., 2018a). Many duplicates were close to the detection limit and when restricting to the samples that were 3x the detection limit or more the HARD was 28% with one sample skewing the data (a duplicate of 17.6 and 2.3 ppb). With this outlier excluded the results were an acceptable 12% HARD.

# 12 RESULTS AND DISCUSSION

## 12.1.1 Sir Samuel/Leonora Region

The region has numerous Au deposits and the results showed only 8.8% data in the <2 µm fraction was below detection for Au (Table 16) compared to 74% of data for the historical analysis of this same material. Both datasets have similar LLDs for Au, (0.5 ppb for this work and 1 ppb for historical data). The GSWA size fraction for analysis was 2000 – 450 µm using Fire Assay-ICPMS. The previous results from GSWA for both map sheets resulted in 74% of results below detection or 1559 samples out of 2114, providing limited value for the interpretation in an area that is a major gold mining region.

Gold in the <2 µm fraction ranged between 0.5 and 17.6 ppb, with mean and median values of 2.36 and 1.8 ppb, respectively (Table 16). Censored data was greater than expected given the past samples and test work in other regions by Noble et al. (2018a; 2014), but is better than the historic data. The distribution of Au picks out a number of the major mineralised areas effectively and shows areas like Tarmoola/King of the Hills (Figure 22) to be prospective with 5.4 ppb Au = 11x detection limit and >95<sup>th</sup> percentile, when the previous data from Bradley et al. (1995) was 1 ppb, the detection limit and 70<sup>th</sup> percentile (Figure 23). The results show the Mt McClure region in the Yandal greenstone belt as elevated with respect to Au (concentrations >5 ppb). The results do not highlight the Bronzewing deposit as much as expected. The area is certainly prospective with consistent Au above 1.5 ppb, but no bigger values like those observed in the Agnew Lawlers area. The data is somewhat noisy as not all samples were highly anomalous near major gold deposits. The historic GSWA results failed to show Bronzewing as a target (Figure 23). The major Agnew Lawlers Au camp is the largest and also the one that is most prominent in the both soils data sets (Figure 22 and Figure 23).

**Table 16. Summary statistics for the Sir Samuel and Leonora regional soils using the UltraFine+ method. Elemental concentrations are in ppm except Au and Pt in ppb. EC in µS/cm. Spectral scalars are standard output, refer to Noble et al., 2018a. n=193. \*Particle size distribution is for comparison only and currently over-estimates clay and silt %. SSA = specific surface area.**

	% Censored	Min	Max	Mean	Median	StDev	95 pctl
Ag	0.0	0.02	0.14	0.04	0.04	0.010	0.05
Al	0.0	34900	171000	95675	93700	24409	142000
As	0.0	3.4	32.1	7.72	7.3	2.934	10.83
Au	8.8	0.5	17.6	2.36	1.8	2.113	5.46
Ba	0.0	31.2	250	67	62	27	110.2
Be	0.0	0.7	2.8	1.62	1.6	0.342	2.2
Bi	0.0	0.2	1.3	0.47	0.4	0.161	0.8
Ca	0.0	310	5320	1835	1920	905	3543
Cd	46.1	0.05	0.16	0.08	0.07	0.025	0.135
Ce	0.0	29.7	113	60.05	58.4	13.684	84.28
Co	0.0	4.7	92	12.77	10.2	8.785	26.63
Cr	0.0	74	501	130	119	50	213.3
Cs	0.0	1.6	6.9	4.88	4.8	0.768	6.1
Cu	0.0	11.5	77.1	42.38	42.9	12.675	62.73
Fe	0.0	30100	92800	58037	57400	9717	74950
Ga	0.0	8.38	47.2	26.17	25	6.006	40.53
Ge	0.0	0.11	0.77	0.37	0.36	0.141	0.62
Hf	0.0	0.07	1.07	0.33	0.26	0.204	0.75
In	0.0	0.03	0.11	0.08	0.08	0.011	0.1
K	0.0	2300	9290	4586	4580	1124	6577
La	0.0	15.4	76.5	39.58	38.2	10.214	59.69

	% Censored	Min	Max	Mean	Median	StDev	95 pctl
Li	0.0	12.3	52	27.85	26.9	6.524	39.92
Mg	0.0	687	4900	1733	1690	696	3100
Mn	0.0	138	1540	483	465	227	887.7
Mo	0.0	0.6	19.9	2.01	1.9	1.347	2.73
Nb	0.5	0.5	1.4	0.94	0.9	0.134	1.1
Ni	0.0	10	1020	41	31	74	75.9
Pb	0.0	13	42	19.27	19.2	3.379	25.44
Pt	13.5	1	5	2.08	2	0.912	4
Rb	0.0	23.7	102	65	63.7	11	81.9
S	0.0	140	637	282	247	107	532.1
Sb	0.0	0.3	1.6	0.45	0.4	0.116	0.6
Sc	0.0	6	38	20	19	5	28
Se	0.0	0.49	1.95	1.19	1.18	0.196	1.539
Sn	0.0	0.9	3.6	2.54	2.5	0.328	3
Sr	0.0	12.7	60.9	34	35	9	49.2
Th	0.0	5.87	49	15.18	14.2	4.850	28.18
Ti	0.0	343	1530	713	688	155	996.5
Tl	0.0	0.2	0.6	0.41	0.4	0.075	0.5
U	0.0	0.76	6.24	3.08	3.09	0.804	4.308
V	0.0	64	204	135	135	21	171
W	0.0	0.2	0.9	0.36	0.3	0.100	0.6
Y	0.0	5.53	47.7	24.24	24.5	8.176	38.13
Zn	0.0	31.8	242	76	74.8	26	106
Zr	0.0	3	27	11.98	10	6.148	23
EC	0.0	7.33	484.3	30	22.36	40	68.783
pH	0.0	3.76	6.4	5.16	5.16	0.530	5.995
<2 µm*	0.0	10	87	62	65	16	82
2-50 µm*	0.0	9	81	28	25	12	48
50 -125 µm*	0.0	0	8.7	0.4	0.0	0.9	1.8
125 - 250 µm*	0.0	0	2.6	0.3	0.0	0.5	1.5
250 -2000 µm*	0.0	0	50.2	9.5	5.0	11.3	31.3
> 2000 µm*	0.0	0	1.5	0.2	0.0	0.3	0.8
SSA* cm <sup>2</sup> /g	0.0	1544	70612	39874	40296	16284	64002
kaolin abundance	0.0	0.125	0.327	0.25	0.244	0.035	0.311
ferric oxide abundance	0.0	0.0484	0.123	0.09	0.09	0.015	0.1143
hem/goe	0.0	896.33	910.26	901	900.37	3	906.709

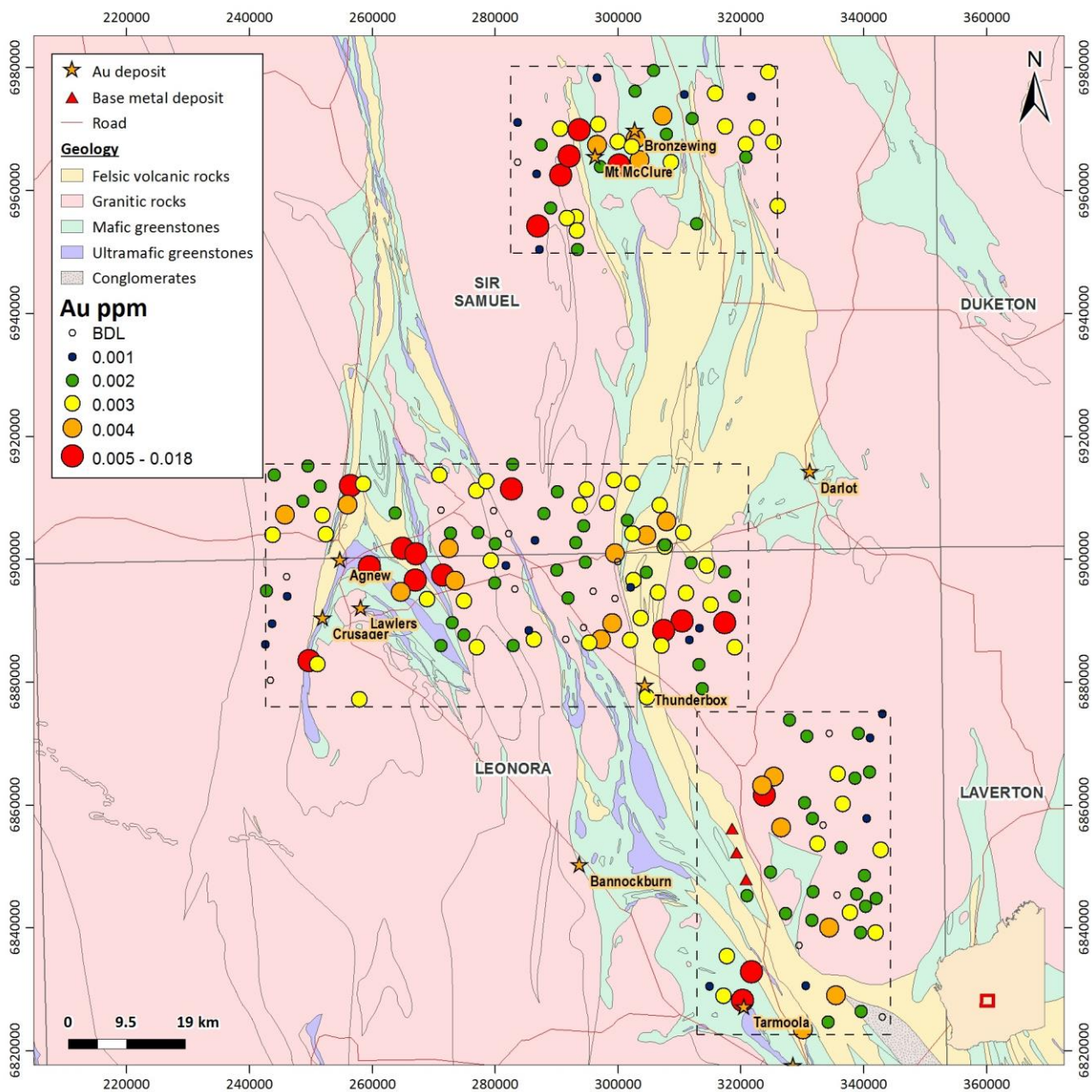
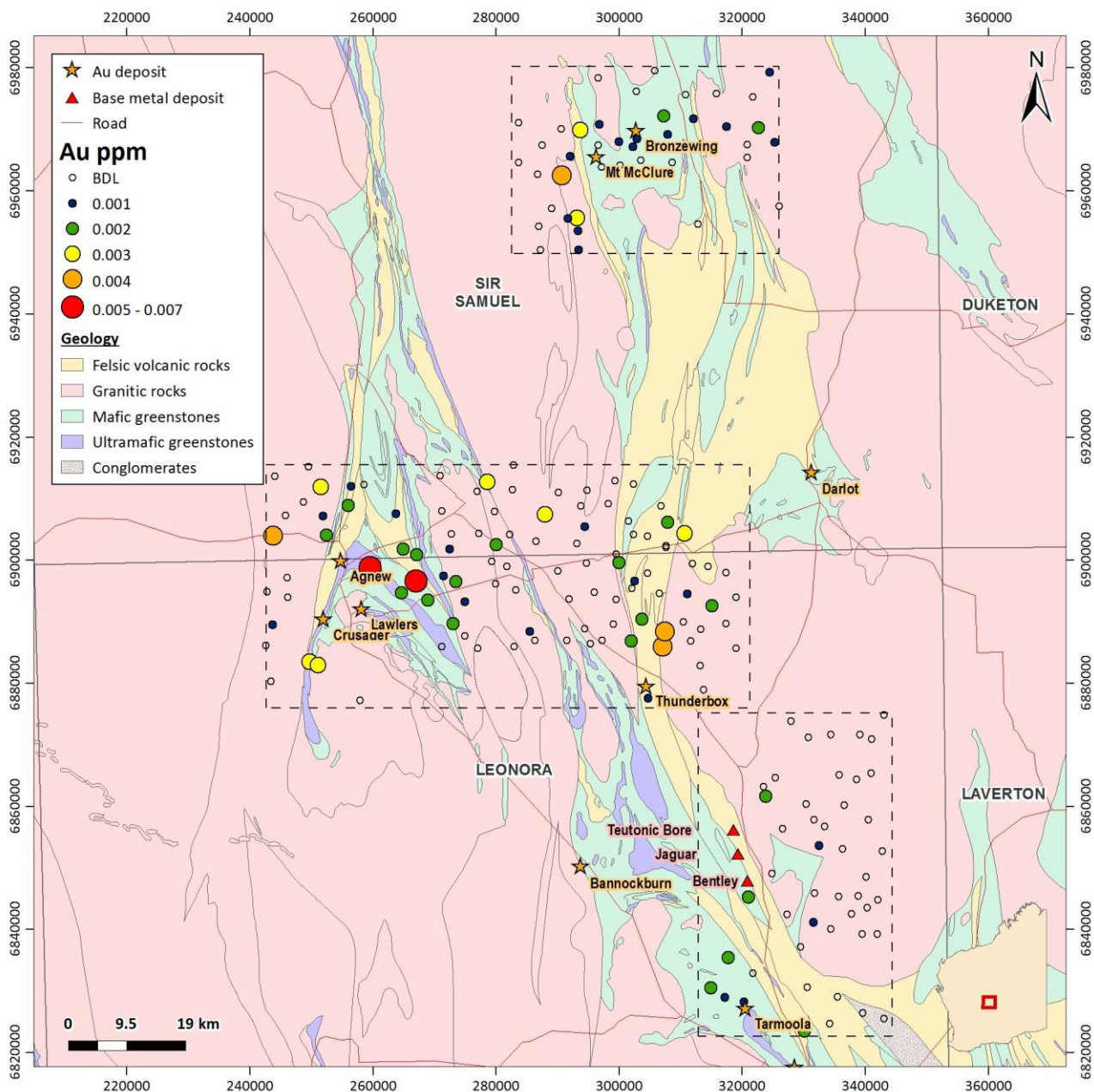
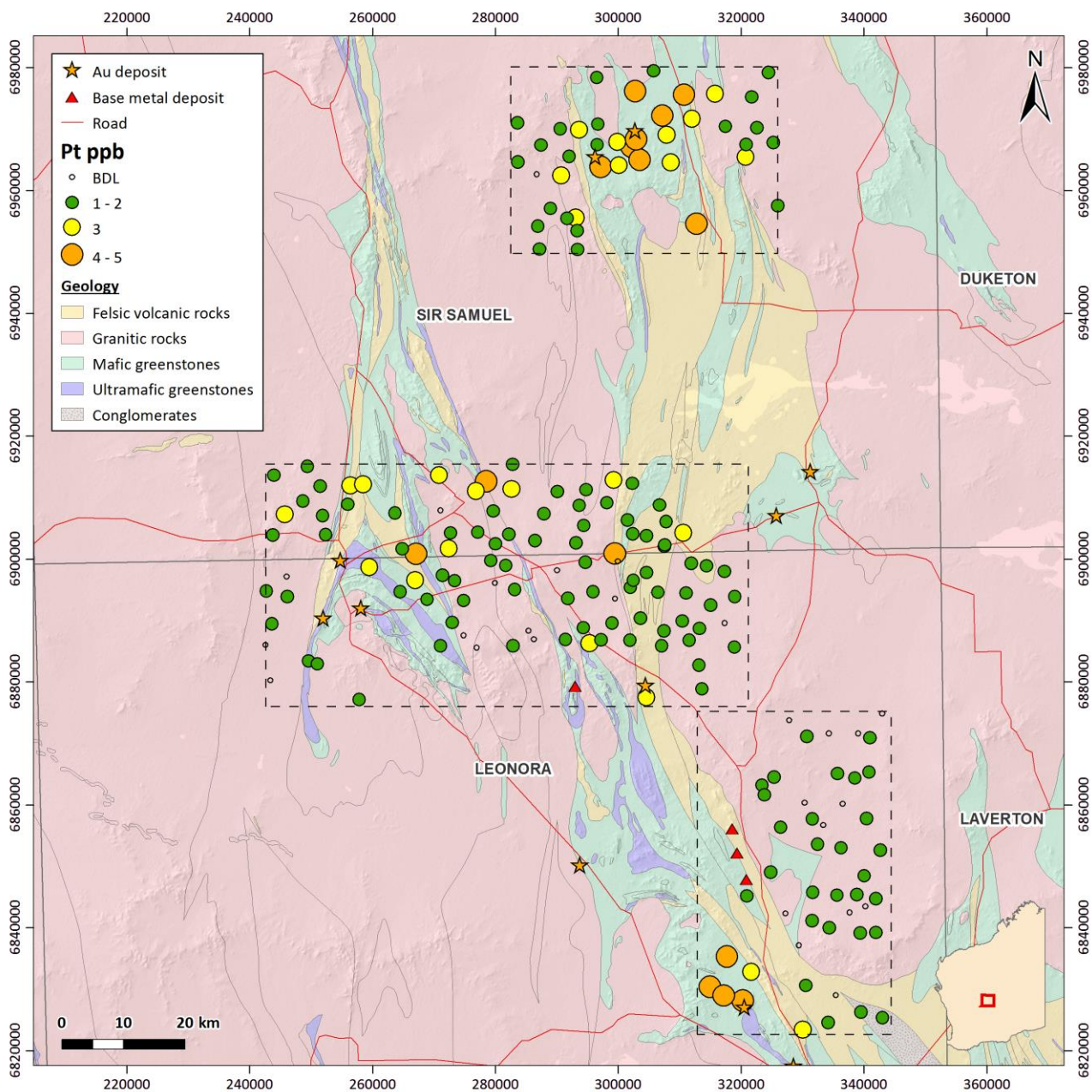


Figure 22. Gold (ppm) in the <2 μm fraction from archived regional soil samples around significant Au mining areas in the Leonora and Sir Samuel 1:250,000 map sheets. Geology is generalised and based on the data from GSWA (2014).



**Figure 23. Gold (ppm) in the archived regional soil sample data from Bradley et al. (1995) and Kojan et al. (1996) in the Leonora and Sir Samuel 1:250,000 map sheets. Geology is generalised and based on the data from GSWA (2014).**

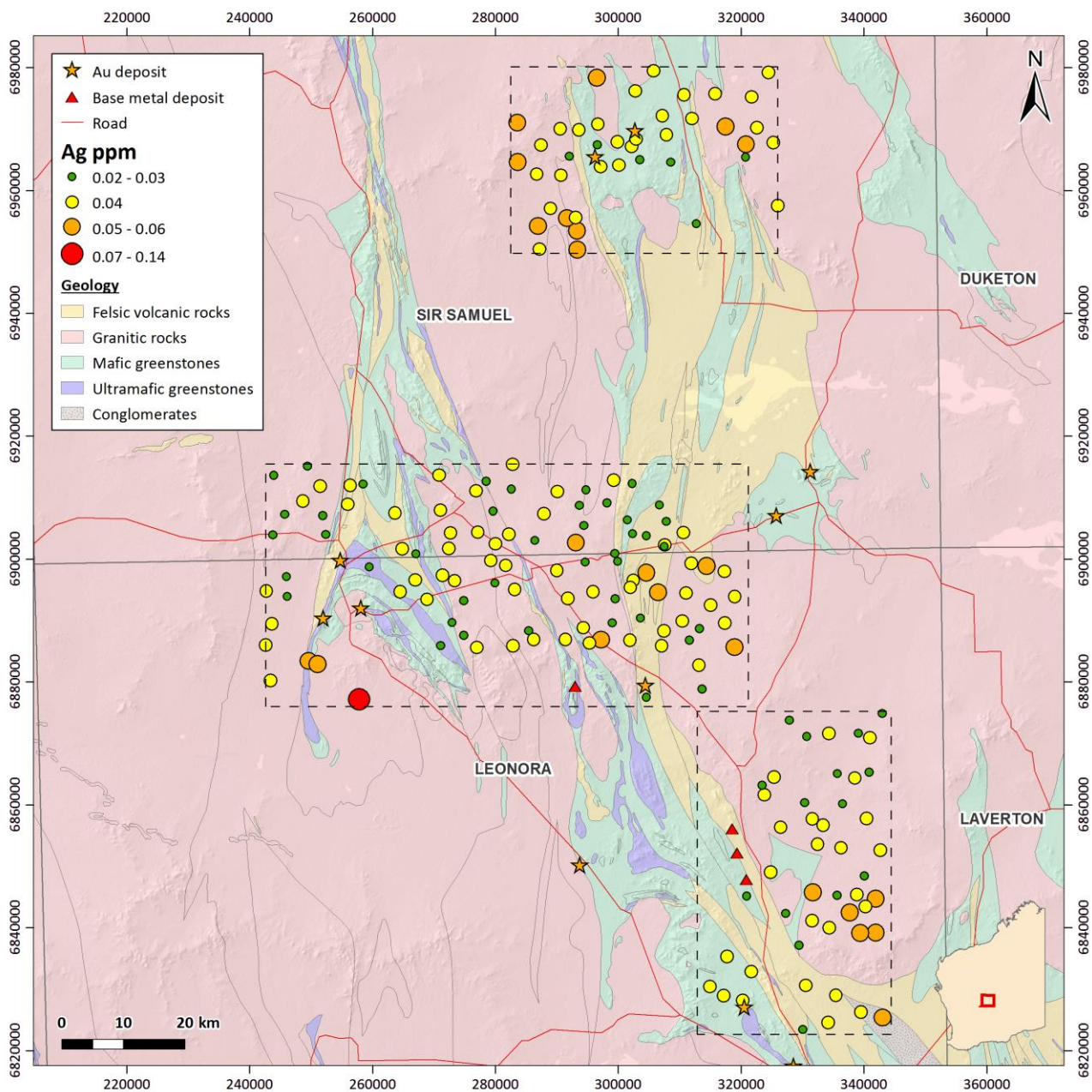
An additional value-add for the UltraFine+ package is the ability to measure Pt in these soils. The QAQC indicates the data are robust, even though most concentrations are only 1-5x the detection limit. In this part of the Yilgarn Craton, Pt may be a useful pathfinder for Ni. Nickel is abundant in the Agnew Wiluna greenstone belt e.g., Leinster and Mt Keith are world-class deposits in the region. The sampled regional map areas did not focus on Ni, so there is not a great overlap with major deposits (not shown). The marked improvement from the % censored data (below detection) in the analysis of both the Leonora and Sir Samuel map sheets was 13.5% with the new method compared to 75% in the previous work with the same samples (Table 16; Bradley et al., 1995, Kojan et al., 1996). The Pt concentrations are still very low, mean 2 ppb, with a maximum of 5 ppb and 95<sup>th</sup> percentile value of 4 ppb. In general, Pt may provide more useful exploration information with the higher Pt numbers indicative of greenstones compared to a pathfinder for Ni, but it is not clear.



**Figure 24. Platinum (ppb) in the <2 μm fraction from archived regional soil samples around significant Au mining areas in the Leonora and Sir Samuel 1:250,000 map sheets. Geology is generalised and based on the data from GSWA (2014).**

Cadmium is a problem for the new method and its analysis as has been a problem in the past. A lot of the data (46%) were below detection limits, and if Cd is deemed the best pathfinder element for a specific type of deposit, the UltraFine+ method may not be appropriate. Historically, the region has a very lower natural abundance of Cd (Table 16; Bradley et al., 1995, Kojan et al., 1996).

Silver should also be an effective trace metal of interest that will be extracted by the UltraFine+ method. Noble et al. (2018a) tended to focus on Au, Cu and Zn analyses, and Ag was not tested extensively, partly because the results for the soils studied in that paper and in these regional results were all close to the detection limits, although there were no censored data. The precision was very good between duplicate samples (HARD = 3.2%) and again is a dramatic improvement to earlier analyses that had 67% of Ag data censored using the same materials (Table 16; Bradley et al., 1995, Kojan et al., 1996). The distribution of Ag in the region does not add a lot of value for exploration (Figure 25). Many of these orogenic deposits are not Ag-rich, so the mapped results are an accurate reflection of this mineralisation style. It would be of interest to test the UltraFine+ method on regional soils in an area that has traditionally used Ag as a successful pathfinder or target element.



**Figure 25. Silver (ppm) in the <2 μm fraction from archived regional soil samples around significant Au mining areas in the Leonora and Sir Samuel 1:250,000 map sheets. Geology is generalised and based on the data from GSWA (2014).**

The censored data of Au, Ag, Pt and Cd will continue to be a challenge for many geochemical soil samples, especially in transported cover that is abundant in Australia. Slight improvements to the UltraFine+ method are expected, and there is also potential for improvements to be gained in the ICP-MS technology. High resolution instruments will become more and more available for standard testing like acid digestions of soils. The challenge is to reduce the TDS of the analytical solutions while retaining the low level precious metals.

Siderophile elements like Cr and V, perhaps to a lesser extent Ni, show the greenstones through the cover. This trend was noted in the earlier published work by GSWA (Bradley et al., 1995; Kojan et al., 1996) and has since been observed in groundwater geochemistry for this region as well (Gray et al., 2016). Chromium and V have similar concentrations in the <2 μm fraction chemistry, with Cr and V mean concentrations of 130 ppm and 135 ppm, respectively (Table 16). The spatial distribution of the two mafic/ultramafic rock indicators is not the same and it seems like high Cr more likely to reflect ultramafic rocks compared to mafic rocks, e.g., high Cr over ultramafic SW of Agnew (Figure 26).



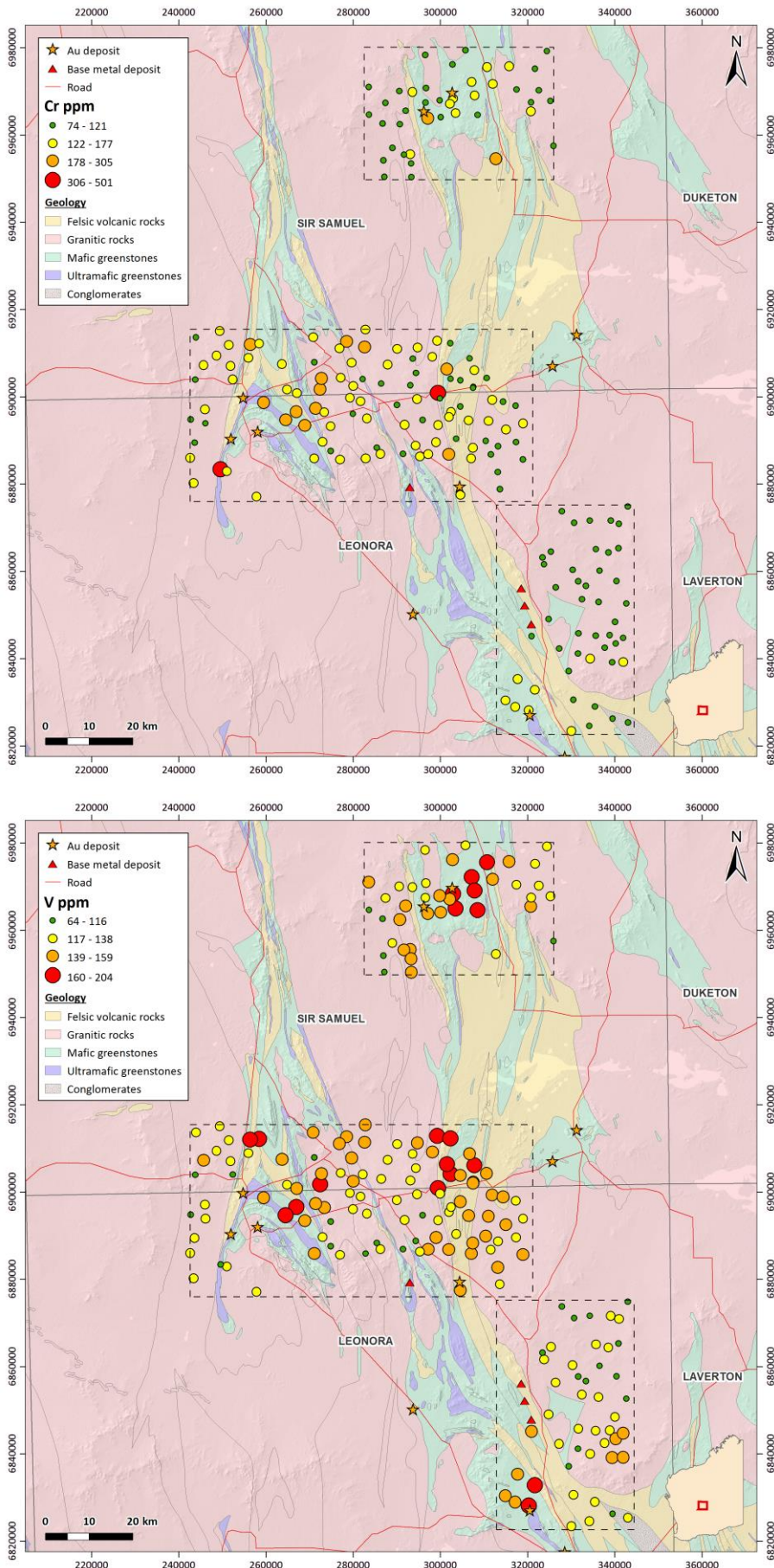


Figure 26. Chromium (top) and V (bottom) in the <2 μm fraction from archived regional soil samples around significant Au mining areas in the Leonora and Sir Samuel 1:250,000 map sheets. Concentrations are ppm. Geology is generalised and based on the data from GSWA (2014).

Lithophile elements do not add a lot of value to assessing the exploration potential for Au and base metals in the areas tested. Future planned processing of these data using machine learning and precompetitive spatial data may add value to the UltraFine+ geochemistry of the lithophile elements.

In contrast to the association of siderophile elements and lithology that has been well established, yet only briefly explored here, chalcophile elements show little relationship with lithology in most cases. Silver was discussed previously and is quite uniform across the region, Cd is below detection and Bi is also inconsistent and at low concentrations. The historic samples also showed 41% of sample results for Bi were censored. For the <2 µm soil fraction Bi concentrations ranged between 0.2 and 1.3 ppm with a detection limit of 0.1 ppm (Table 16).

Copper and Zn results are elevated in the areas of base metal deposits in the study area, but are not stand out anomalies. These elements tend to be a little inconsistent for targeting or at a general background level. As noted by Noble et al. (2018a), there is significant upside for Au using the <2 µm fraction, but Cu and Zn are also effectively measured in the silt sized fraction, too. As a result the concentrations in the finer fractions are greater than the historic data, but the patterns for exploration targeting are similar. The sample <1 km to the south of the Bentley deposit is in the highest quartile, but not the biggest concentrations measured.

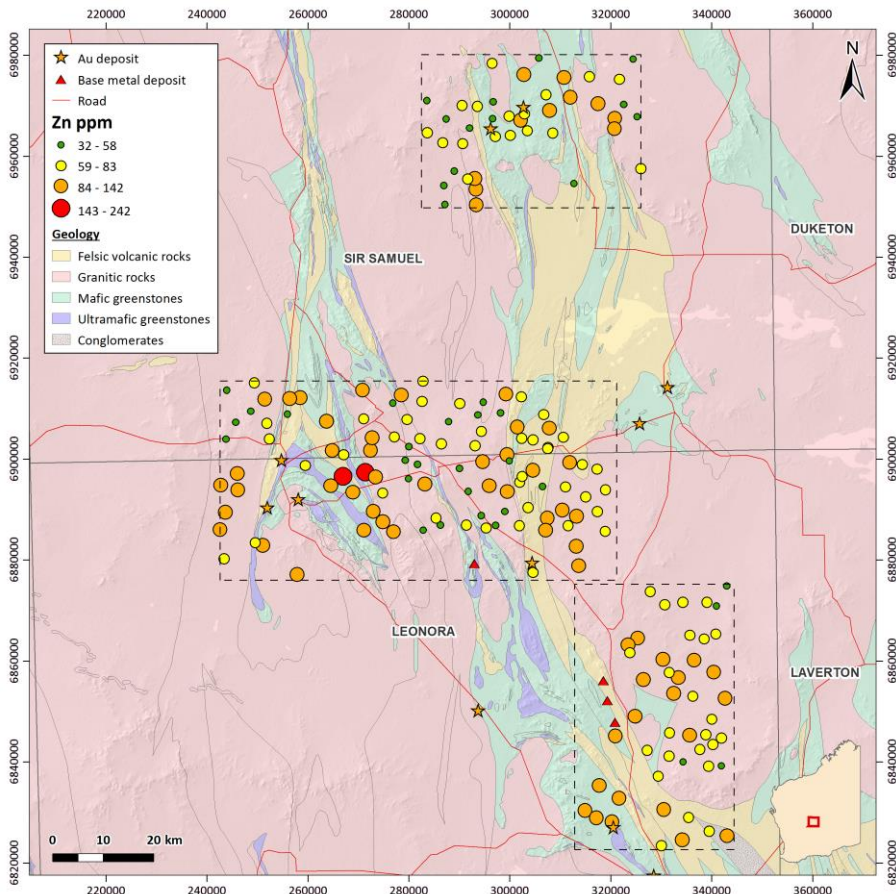
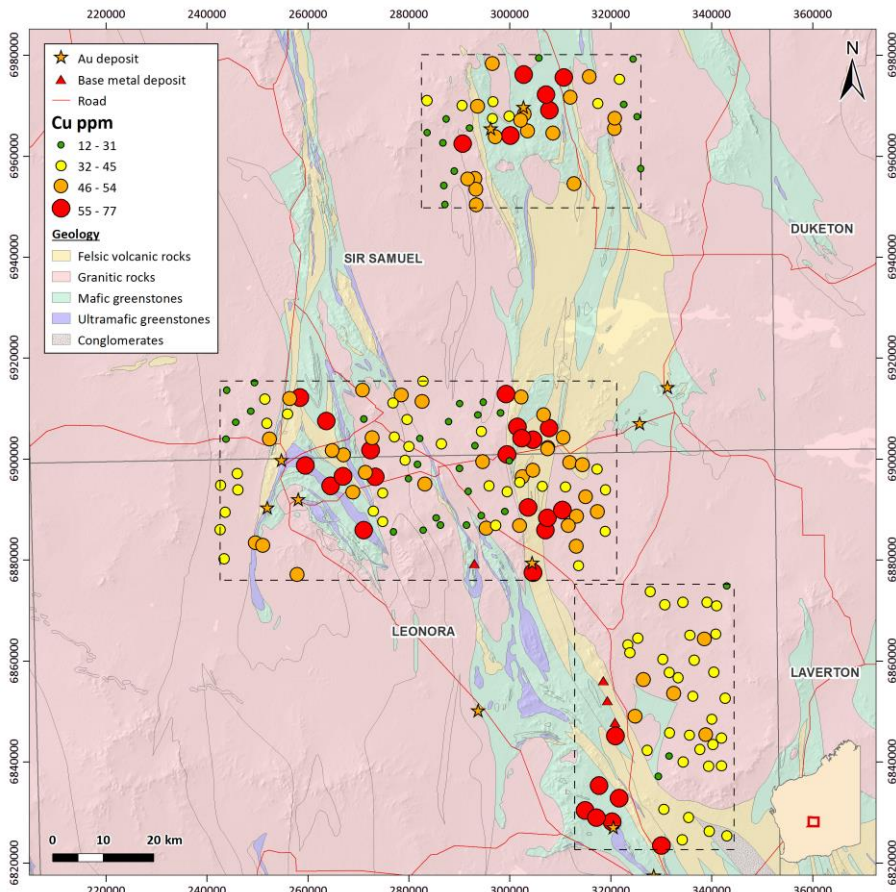


Figure 27. Copper (top) and Zn (bottom) in the <2 μm fraction from archived regional soil samples around significant Au mining areas in the Leonora and Sir Samuel 1:250,000 map sheets. Concentrations are ppm. Red triangles are base metal deposits of note. Geology is generalised and based on the data from GSWA (2014).

The Leonora/Sir Samuel results show quite a lot of value to improving the results of historic samples. It also suggests that clusters of elevated Au or other target and pathfinder elements are prospective on a regional scale.

### Kingston Region

The Kingston region is a greenfields exploration area, and although taking in the northern margin of the highly prospective Yilgarn Craton, there is only one known gold deposit (Mt Eureka; Figure 21)

Summary statistics of the results of the UltraFine+ analytical method applied to 300 soils from the region are shown in Table 17.

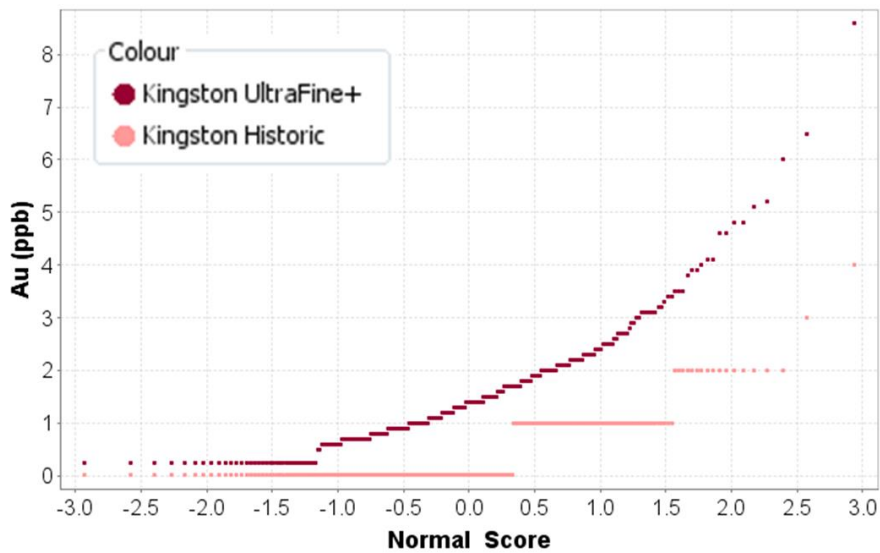
**Table 17. Summary statistics for the Kingston regional soils using the UltraFine+ method. Elemental concentrations are in ppm except Au and Pt in ppb. EC in  $\mu\text{S}/\text{cm}$ . Spectral scalars are standard output, refer to Noble et al., 2018a. n=302. \* Particle size distribution is for comparison only and currently over-estimates clay and silt %. SSA = specific surface area.**

	% censored	Min	Max	Mean	Median	StDev	95 pctl
Ag	0.3	0.01	0.13	0.03	0.03	0.01	0.05
Al	0.0	30100	158000	89355	88250	22117	130850
As	0.3	1.5	19.4	7.61	7.5	2.27	11.29
Au	12.3	0.5	8.6	1.76	1.5	1.09	3.9
Ba	0.0	25.7	629	108	86.15	79	219.85
Be	0.0	0.5	3	1.43	1.4	0.38	2.1
Bi	0.0	0.2	2.7	0.43	0.4	0.15	0.5
Ca	0.0	163	81100	1956	1015	5477	4313.5
Cd	85.4	0.05	0.21	0.08	0.06	0.04	0.1875
Ce	0.0	17.5	168	52.18	45.5	21.48	100.715
Co	0.0	2.7	27.9	9.37	8.9	4.36	16.64
Cr	0.0	45	765	89.40	84	44.87	110
Cs	0.0	2	14.2	6.47	6.1	1.73	9.885
Cu	0.0	8.8	67.6	28.82	27.9	7.73	41.01
Fe	0.0	20400	71500	53879	54400	8609	66900
Ga	0.0	8.67	47.9	30.23	30.65	5.78	38
Ge	1.7	0.05	0.74	0.34	0.33	0.12	0.54
Hf	0.0	0.02	1.17	0.43	0.42	0.23	0.82
In	0.0	0.03	0.12	0.08	0.08	0.01	0.1
K	0.0	950	19900	5603	4880	2854	11325
La	0.0	9.51	93.6	29.97	26.95	11.07	49.725
Li	0.0	7.1	86.5	30.63	29.75	11.22	50.01
Mg	0.0	281	15200	1889	1300	1910	6109.5
Mn	0.0	92	4160	431.72	317	395.20	1115.5
Mo	0.0	0.6	5.2	1.54	1.5	0.47	2.385
Nb	0.3	0.5	1.5	0.93	0.9	0.15	1.2
Ni	0.0	8	85	22.40	21	8.10	37
Pb	0.0	8.8	42.3	23.25	22.4	5.62	33.785
Pt	13.9	1	4	1.57	1	0.66	3
Rb	0.0	19	118	65.37	63.1	17.21	99.47
S	0.0	84	3590	375	301	366	851.65
Sb	0.0	0.2	1.3	0.50	0.5	0.11	0.685

	% censored	Min	Max	Mean	Median	StDev	95 pctl
Sc	0.0	5	25	14.08	14	3.11	20
Se	0.0	0.49	2.23	1.11	1.095	0.22	1.4585
Sn	0.0	1.2	4.3	2.87	2.9	0.48	3.685
Sr	0.0	7.6	162	33.71	27.85	19.54	72
Th	0.0	6.28	41.2	15.86	15.7	3.38	20.385
Ti	0.0	243	927	595	588.5	116	776
Tl	0.0	0.2	0.7	0.50	0.5	0.09	0.6
U	0.0	0.49	9.68	1.87	1.72	0.75	2.9685
V	0.0	43	168	118	121	17	142
W	0.0	0.2	0.6	0.37	0.4	0.06	0.4
Y	0.0	4.5	44.3	15	13.3	7	30.48
Zn	0.0	13.8	175	53	47.65	22	97.365
Zr	0.3	1	35	15	15	8	28
EC	0.0	9.75	3530	91	32.31	311	230.55
pH	0.0	4.08	8.18	4.92	4.745	0.67	6.3595
<2 µm*	0.0	5	81	52	54	16	74
2-50 µm*	0.0	11	86	33	29	14	64
50 -125 µm*	0.0	0	11	1	1	1	3
125 - 250 µm*	0.0	0	2	0	0	0	1
250 -2000 µm*	0.0	0	53	13	12	11	32
> 2000 µm*	0.0	0	3	0	0	0	1
SSA*	0.0	697	71932	35716	38326	16492	57812
kaolin abundance	0.3	0.069	0.352	0.227	0.229	0.047	0.305
ferric oxide abundance	0.0	0.005	0.143	0.085	0.086	0.020	0.114
hem/goe	0.3	890	913	899	899	3	904

The results showed 12.3% data in the <2 µm fraction was below detection for Au (Table 17). The historical analysis of this same 302 samples with a similar detection limit (1 ppb versus 0.5 ppb now), resulted in 63% of results below detection or 191 samples out of 302, providing more limited use in Au exploration. The difference (and benefit of the re-assayed data) is evident in Figure 29 (a versus c).

Gold in the <2 µm fraction ranged between 0.5 and 8.6 ppb, with mean and median values of 1.76 and 1.5 ppb, respectively (Table 17). Censored data was similar to the Leonora/Sir Samuel soil chemistry results, with the overall Au concentrations lower, which was expected given this a region of more cover in the Earaaheedy Basin. The results of the UltraFine+ method are a significant improvement on the historic data (Figure 28).



**Figure 28. Probability plot showing the Au data from the UltraFine+ method and the historic data from Pye et al. (2000).**

The initial data only had a few samples above detection, with the highest value located approximately 10 km south of the only mined gold in the area (Figure 29a). This sample sits on the edge of the greenstone. The rest of the survey is not useful for Au exploration if only using gold as the target element in these soil samples. The re-assayed results are a major improvement with Au detected in nearly all samples, and the greenstones much more pronounced in terms of prospectivity and these appear to extend further into the basin cover. It is likely the Au concentrations show a lithological control as well as any potential effects of mineralisation. The results are compared by using the same scale as the historic data and a more standard scale (Figure 29b, c).

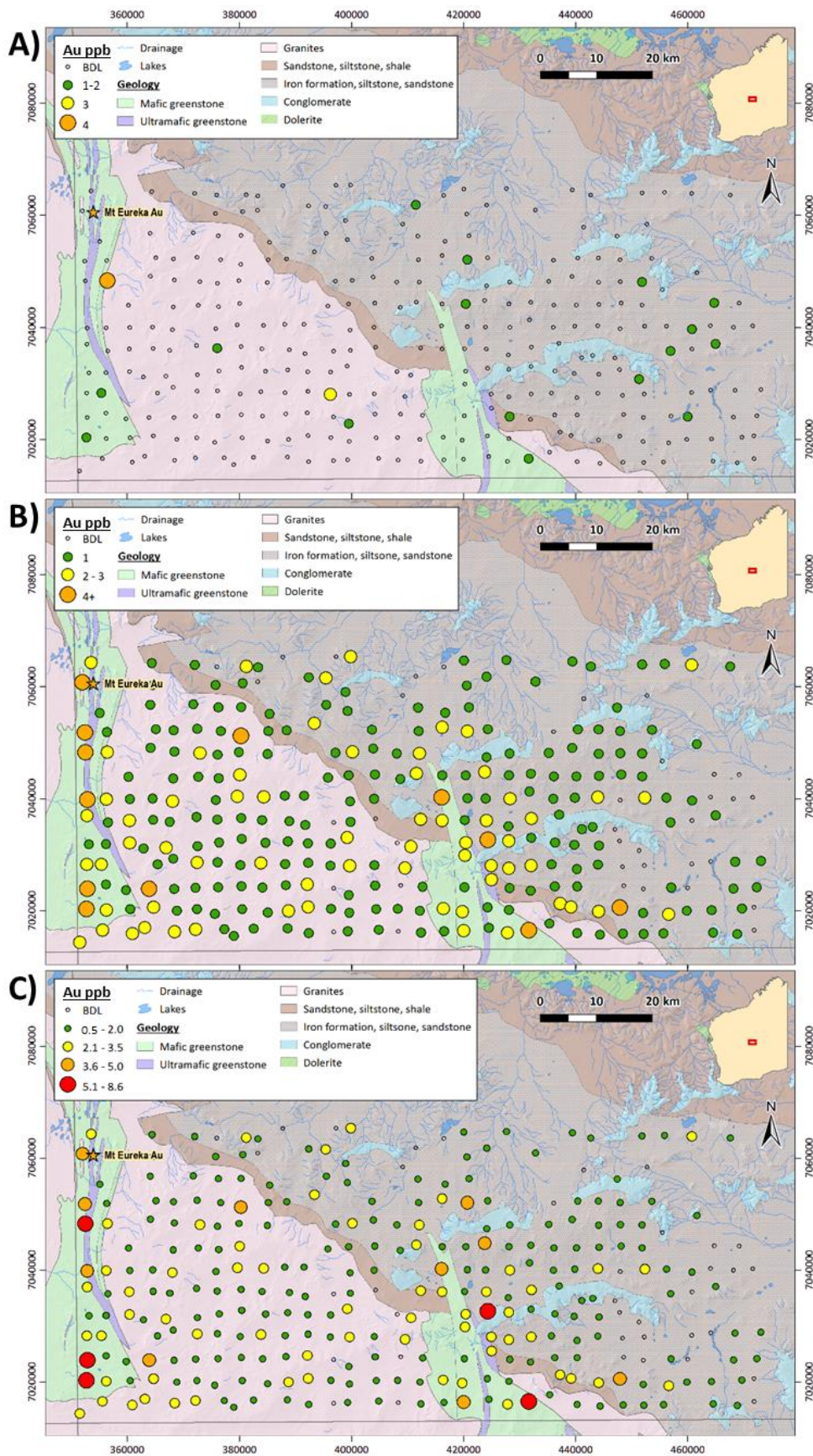
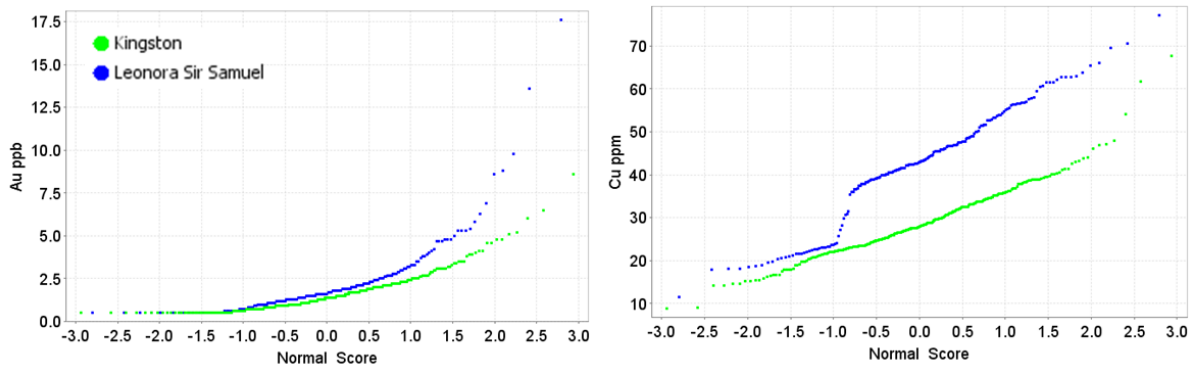


Figure 29. Gold (ppb) in soils in the Kingston 1:250 000 map sheet. A) Original data with only a few detectable Au values, B) the ultrafine fractions Au using the same scale, C) the Ultrafine fractions with natural breaks in scale to better identify anomalous areas. The star represents the (small) Mt Eureka Au deposit. Geology is generalised and based on the data from GSWA (2014).

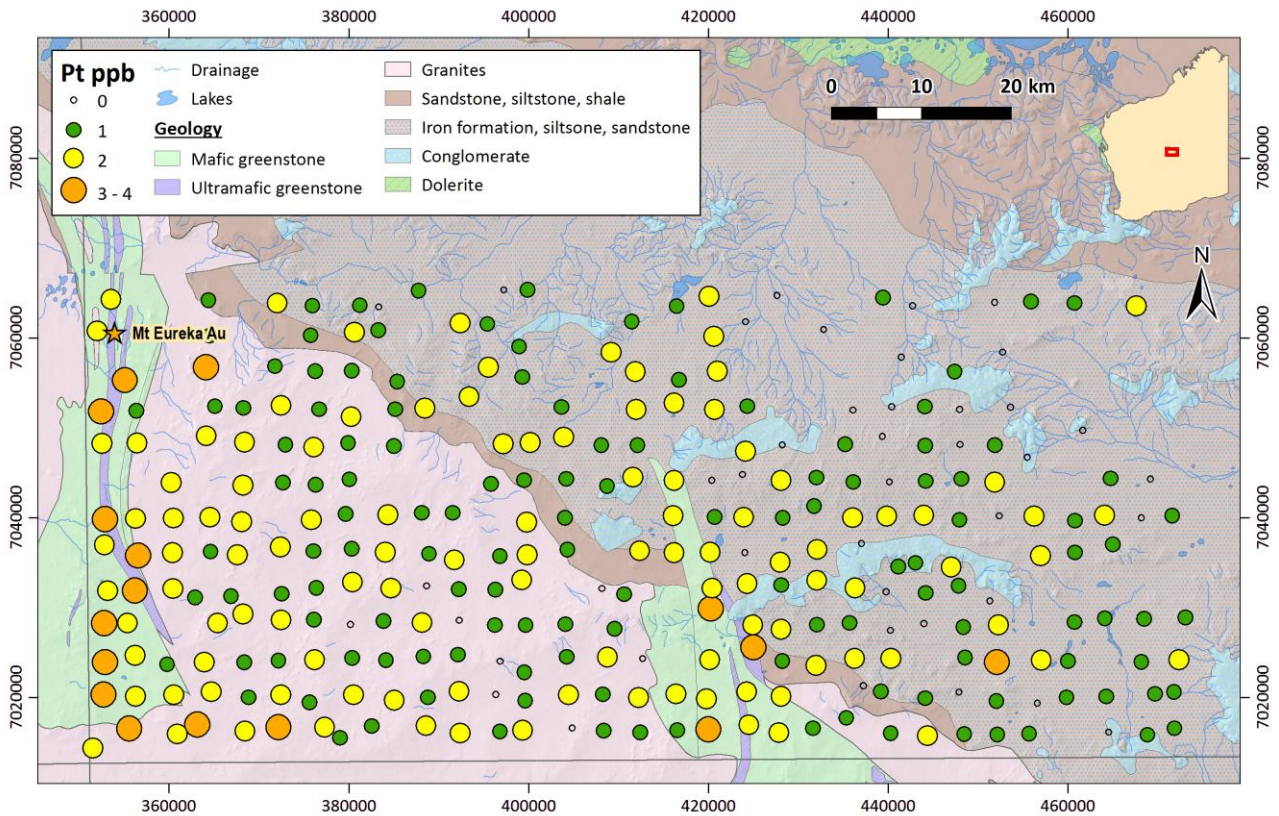
The Au (and Cu) results are commonly lower than those concentrations measured in the <2  $\mu\text{m}$  fraction in Leonora and Sir Samuel soils. This is well represented in Figure 30 and gives a potential indication of proximity to Au deposits. Given there were a number of deposits in the Leonora/Sir Samuel region that were identified with concentrations of Au of 3.5-7 ppb using the same method, sample sites represented by similar concentrations in Figure 29 are perhaps just as prospective, and clearly much less explored.



**Figure 30. Probability plot comparing Au and Cu concentrations extracted using the UltraFine+ method in the <2  $\mu\text{m}$  fraction for both Leonora/Sir Samuel and Kingston map sheets.**

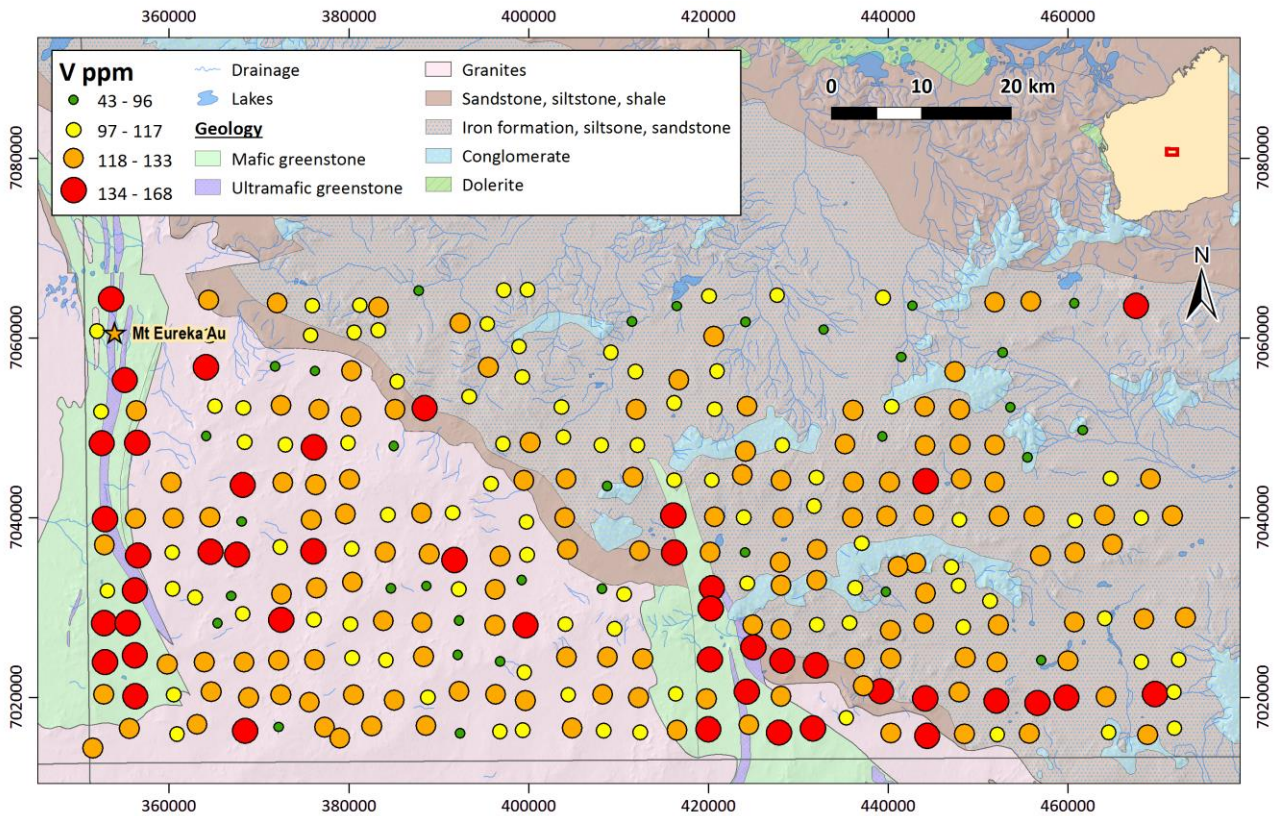
As in the orientation regional data set, Ag and Pt were consistently measured above detection limits using the <2  $\mu\text{m}$  fraction geochemistry (Table 17), a marked improvement on the past data, with historic data for Ag and Pt being censored 64% and 81%, respectively, in the same soils (Pye et al., 2000). Even with the additional new data, it is unclear if these elements present additional targets or indicators for mineralisation potential in the region. As stated earlier, Ag is not really associated with Au deposits of the Yilgarn Craton, but potentially may be a more viable target in the basin setting. Platinum changes are subtle and tend to highlight the greenstones in the Kingston region (Figure 31).





**Figure 31. Platinum (ppb) in the <2 μm fraction from archived regional soil samples in the Kingston 1:250,000 map sheet. Geology is generalised and based on the data from GSWA (2014).**

As is observed in the orientation regional sites, siderophile elements Cr, Ni and V (Figure 32) also tend to be found in greater concentrations in soils that are located near the greenstones. The concentrations of these elements have similar ranges, but are about 30% less than those in Leonora/Sir Samuel map sheets and this is attributed to fewer ultramafic and mafic rocks, as well as the thicker transported cover and increased sandplain areas over the Earahedy basin. These mafic/ultramafic rock indicator elements in the soil are in strong contrast to Rb concentrations that map out the basin and sedimentary rocks much more effectively. The Rb is not as high over the granitic rocks as might be expected and this is postulated to be due to the regolith over the granites being mostly in situ and strongly weathered. Some other lithophile elements e.g. Li (not shown) also replicate this pattern, but not as strongly as that observed in Rb (Figure 33). Combining these variables in a simplistic manner (V+Cr-Rb) as the statistical range of these variables is similar (Table 17) is a very effective way to map the lithology in this region (Figure 34). Using inverse weighted distances to extrapolate these data produce a very effective map of the Yilgarn Margin into the Earahedy Basin under cover (Figure 34). Whereas this can be seen in other data such as magnetics and gravity (Figure 34), it is good to see the geochemistry from the surface materials reflecting the underlying rocks and providing evidence that materials are mobilised from depth through the cover as has been suggested by numerous studies in Australia (Morris 2013; Noble et al., 2017a, 2018b; Anand et al., 2014, 2016a) and globally (Kelley et al., 2003; Wang et al., 2007; van Geffen 2015).



**Figure 32. Vanadium (ppm) in the <2 μm fraction from archived regional soil samples in the Kingston 1:250,000 map sheet. Geology is generalised and based on the data from GSWA (2014).**

A strong anomaly is present in the SW corner of this lithology ratio map that is attributed to an extreme Cr concentration (765 ppm, 99<sup>th</sup> percentile) with very low Rb (23 ppm, 1<sup>st</sup> percentile). This could be a sliver of unmapped greenstone in the Yilgarn Craton, but it is unusual that it does not show in the magnetics data (Figure 34). This anomalous sample has some Au (2.9 ppb, 87<sup>th</sup> percentile), but is not anomalous. The Cr concentration is more than two times greater than the next greatest value in the survey (303 ppm).

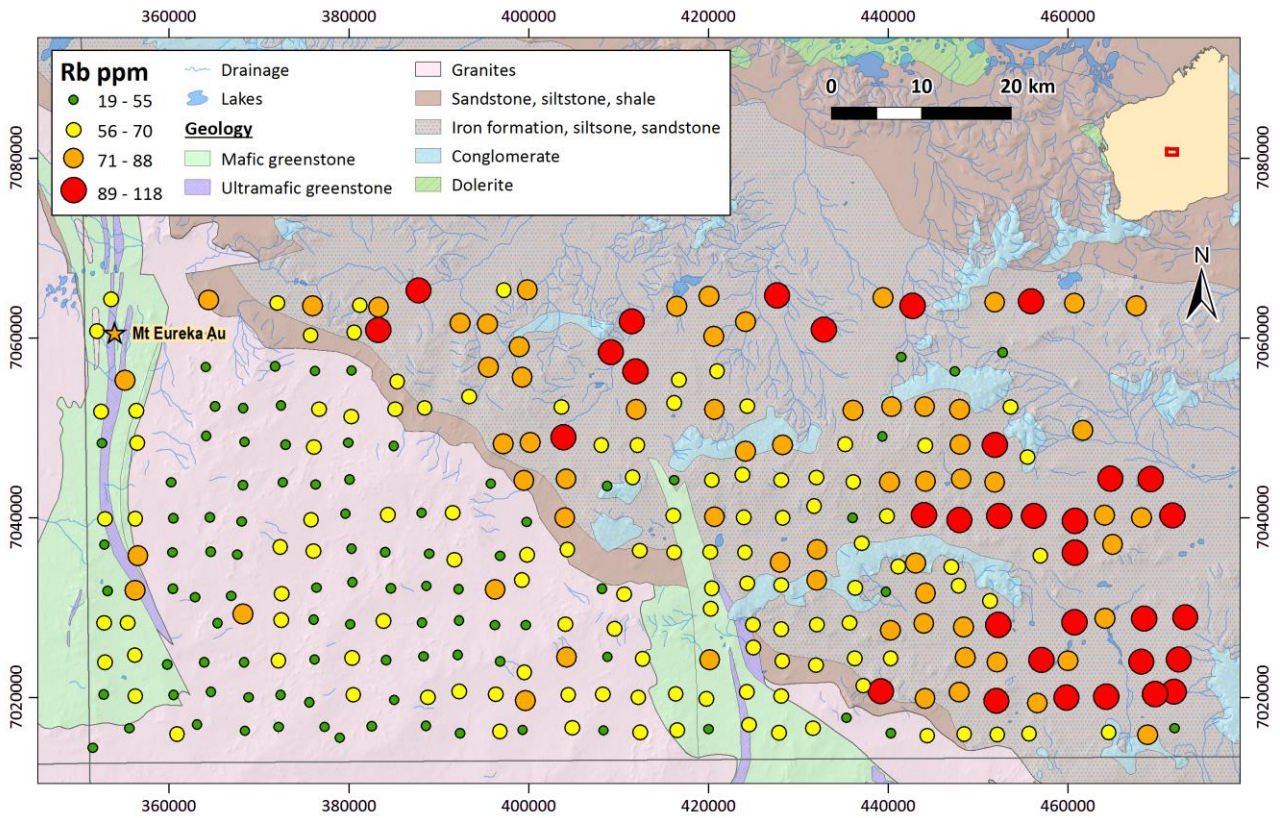


Figure 33. Rubidium (ppm) in the <2 μm fraction from archived regional soil samples in the Kingston 1:250,000 map sheet. Geology is generalised and based on the data from GSWA (2014).

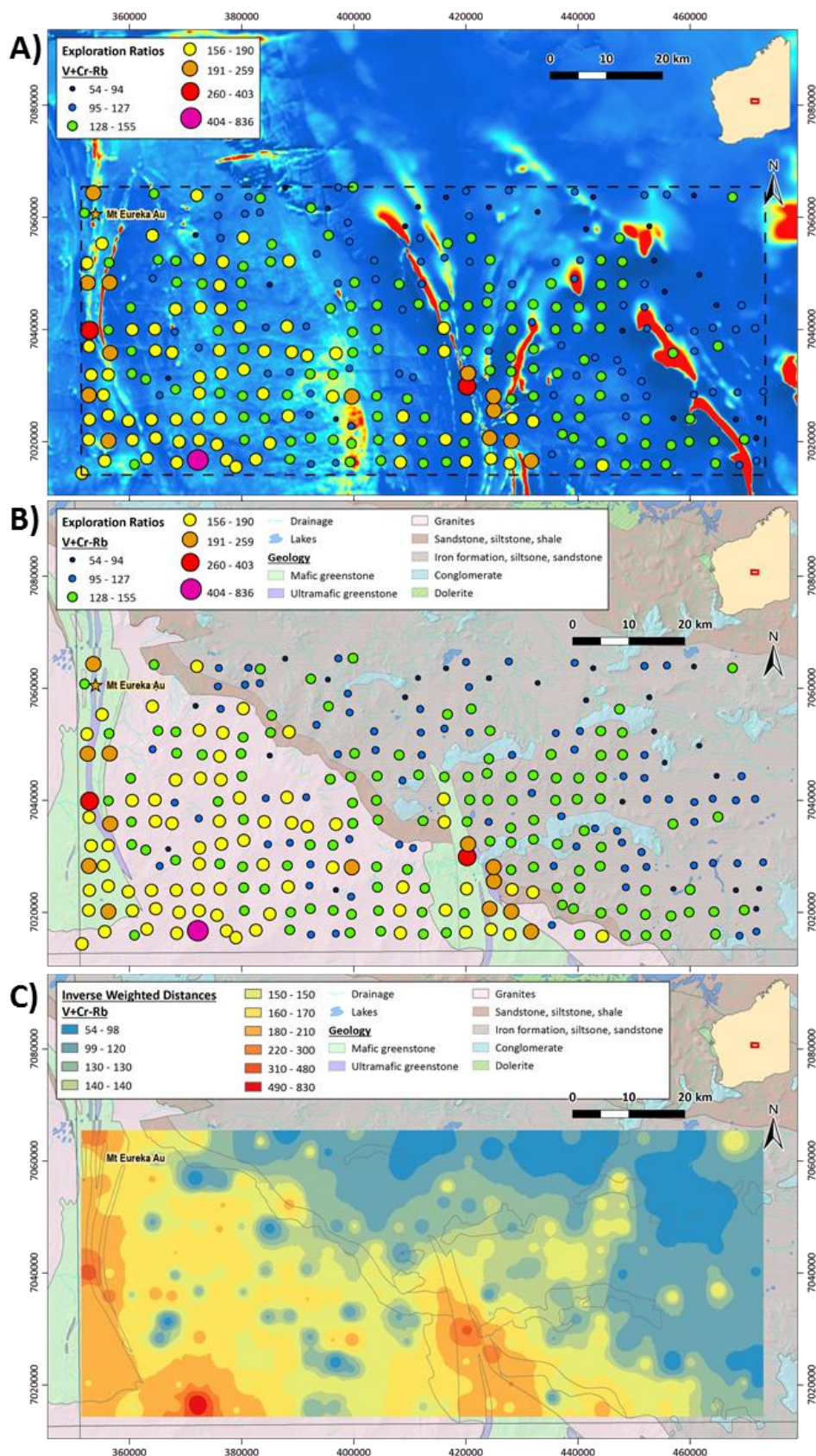
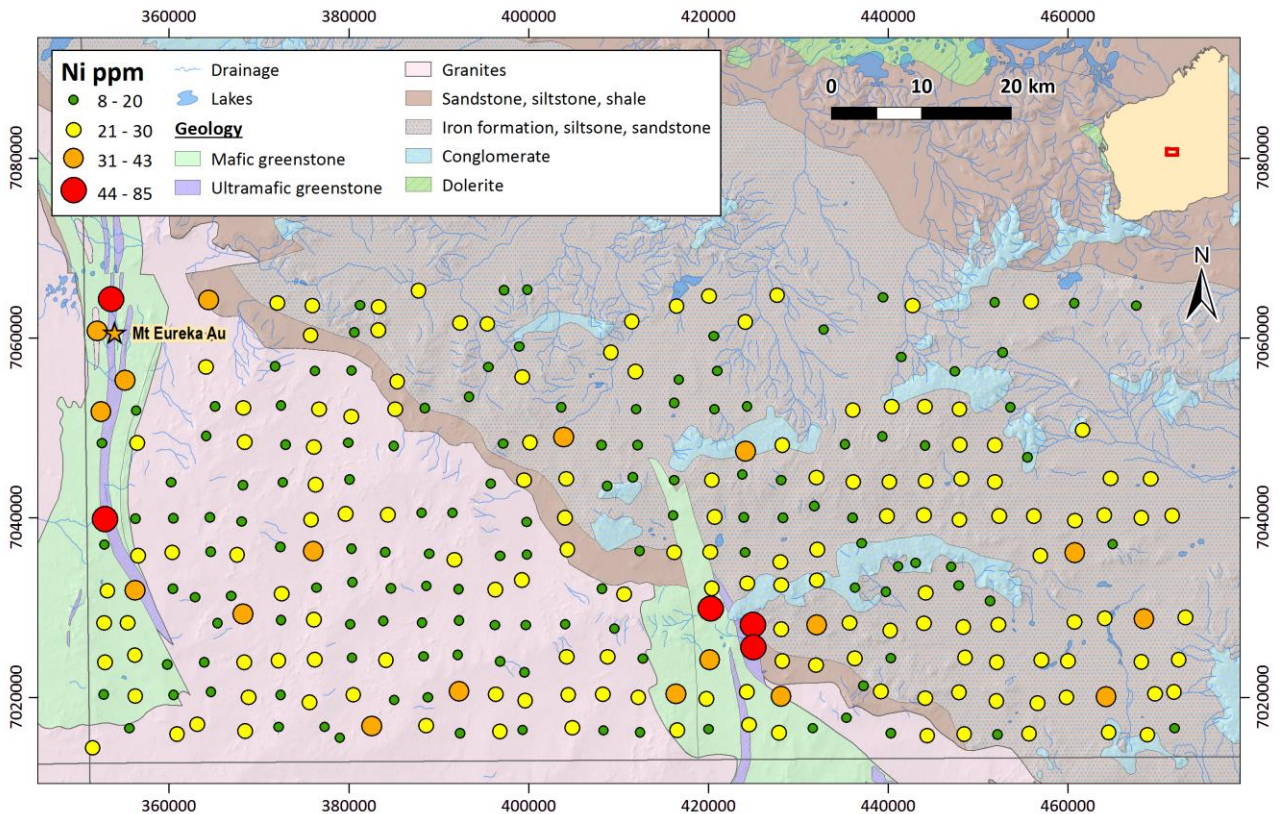


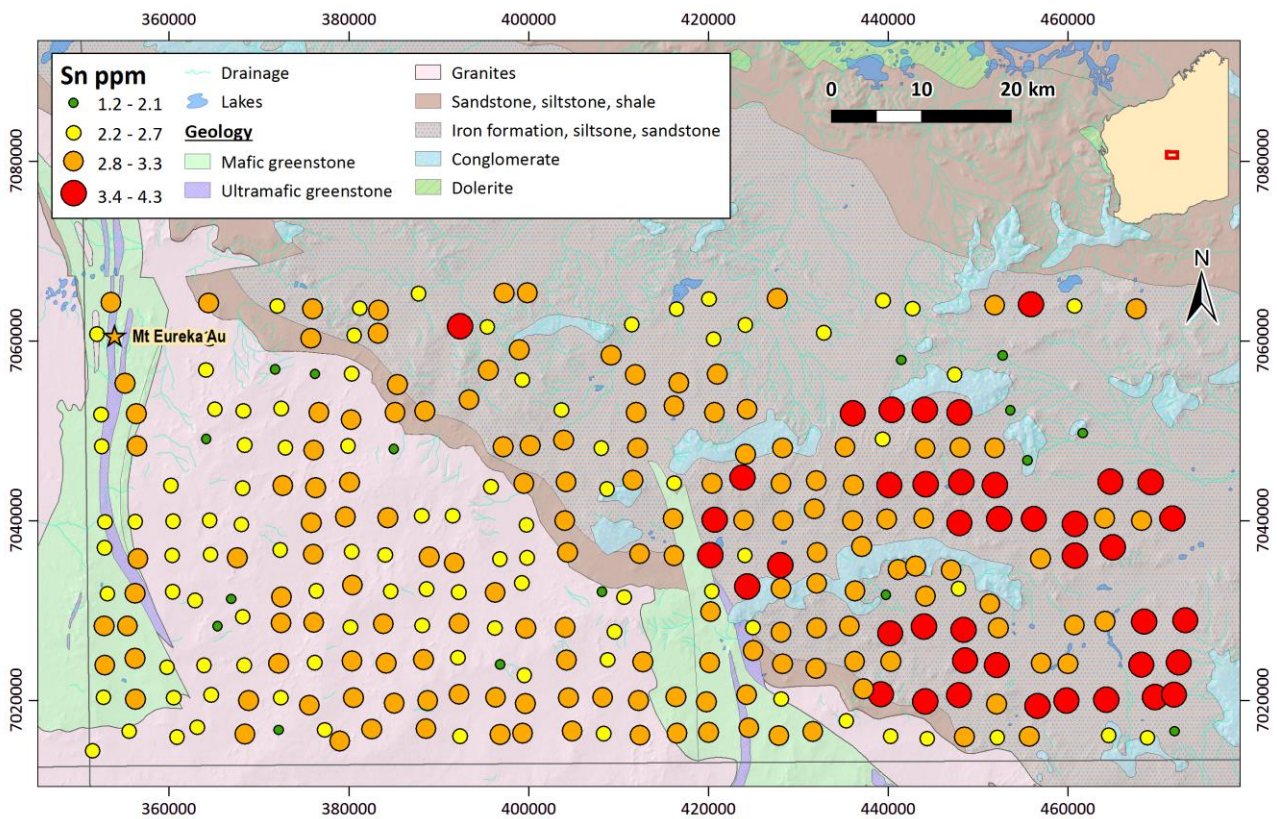
Figure 34. A) Lithology ratio using V, Cr and Rb data from the <2  $\mu$ m fraction analysis shown over the magnetics; B) the ratio over the geology map; C) the inverse weighted distance interpolation of this data and the outlines of the major geology units underneath showing a very close association. Geology is generalised and based on the data from GSWA (2014).

For exploration purposes, Ni shows a few strong anomalies in a small clustered area at the edge of the greenstone which is possibly an extension of the Duketon greenstone belt that has a number of small, Ni deposits associated with it (e.g. Rosie, C2; Godel et al., 2012). The historic soil Ni data (Pye et al., 2000) is very similar in patterns to those determined using the <2 µm fraction and the UltraFine+ method (Figure 35).



**Figure 35. Nickel (ppm) in the <2 µm fraction from archived regional soil samples in the Kingston 1:250,000 map sheet. Geology is generalised and based on the data from GSWA (2014).**

In contrast to the association of siderophile and lithophile elements, chalcophile elements show little relationship with lithology in most cases with a number of these elements poorly extracted using the UltraFine+ method. Arsenic, Bi, Cd, Se are all close to detection limits (Table 17). Cadmium was rarely detected. Others such as Ga, Ge (and W) has been shown to have poorer reproducibility and some association with Ti and Si, and were not used. Interestingly the Sn (and to a lesser extent Li and Rb) values are quite unusual and provide very different information to the earlier survey results. Tin (Figure 36) shows only a small distribution range from 1.2-4.3 ppm (Table 17), but a cluster of the larger values are in the far eastern section of the survey and weakly correlated to Rb ( $R=0.61$ ) possibly reflecting the lithological differences, however, the Sn distribution is not as wide-spread in the Earahedy basin, does not seem to be associated with anything else, and does not match the historic results at all.



**Figure 36. Tin (ppm) in the <2 μm fraction from archived regional soil samples in the Kingston 1:250,000 map sheet. Geology is generalised and based on the data from GSWA (2014).**

Base metals such as Cu and Zn tend to be slightly elevated along the greenstones, but also show higher concentrations in the soils in the basin. The historic data shows lower concentrations than the <2 μm fraction results in general. This is evident comparing the plots with the same scale (Figure 37 a and b). The rescaled newer data compared to the old data (Figure 37 a and c) shows a more consistent background Cu concentration across the Kingston area. The Cu results perhaps highlight some additional areas with higher Cu in the basin setting (central north and far east), but these are far from anomalous concentrations. Copper correlates with Sc ( $R = 0.71$  and  $0.85$ ) in both Kingston and Sir Samuel Leonora, respectively, and likely reflects a broad lithological control. A weak positive trend is associated between Cu and Zn. Zinc shows similar results with respect to comparison to older data and also the results tend to show more a background response (Figure 38). The <2 μm fraction extracts more Zn, yet it is not showing more anomalous results than the traditional extraction and analysis conducted in 2000 by GSWA (Pye et al., 2000).

Even though a few elements have not really added a lot of value or change by being processed with the UltraFine+ method in this greenfields setting, there has been no negative impact by re-assaying these samples, with significant upside for Au. Gold is providing new information and is not associated with any other variable determined in the UltraFine+. The other major value of the workflow applied here is the additional information provided (including spectral mineralogy and particle size analysis) that we don't routinely use and therefore don't intuitively apply. Although the current methodology over estimates the fine materials, the results provide a variable for comparison, an estimate of exchange capacity of mobile metals which should be used to interpret the regional geochemical patterns. Results for the Kingston map sheet using the UltraFine+ method shows the selected soils have quite a range in their Specific Surface Area (SSA) with a mean of  $35,716 \text{ cm}^2/\text{m}$  and a standard deviation of  $16,492 \text{ cm}^2/\text{cm}$  (Table 17) using the wet separation sample. The SSA is calculated based on the clay particle size distribution and so the concentrations of clays  $\ll 2 \mu\text{m}$  greatly influence this result.

Another major metal exchange phase is Fe oxides, and the spectral results for Fe oxide abundance are also valuable information for assessing anomalous results. The Fe oxide abundance has a correlation coefficient of 0.68 with SSA, 0.65 with clays  $< 2 \mu\text{m}$  and 0.61 with clays  $< 0.2 \mu\text{m}$ . Using SSA or perhaps the amount of fine clays ( $< 0.2 \mu\text{m}$ ) may be useful as these will have more exchange surfaces. Ratios of elemental

concentrations with the total % clay should not be used as the UltraFine+ effectively separates this fraction only, and in effect is levelling all soil geochemical signatures based on the clay content. The results at Tooting Bec (later) show this specific benefit. The best algorithms to integrate this data with the geochemistry for exploration are not known, and further testing will assist in refining the method and interpretation. As an example of potential future map products that could be developed using this workflow, we created two example indices. One uses Au in a ratio with SSA, the other is a multielement (Au, Bi, Cu, Zn, Ni, Pb, Pt) combination that is adjusted for exchange phases (clay and Fe oxide abundance, Figure 39).

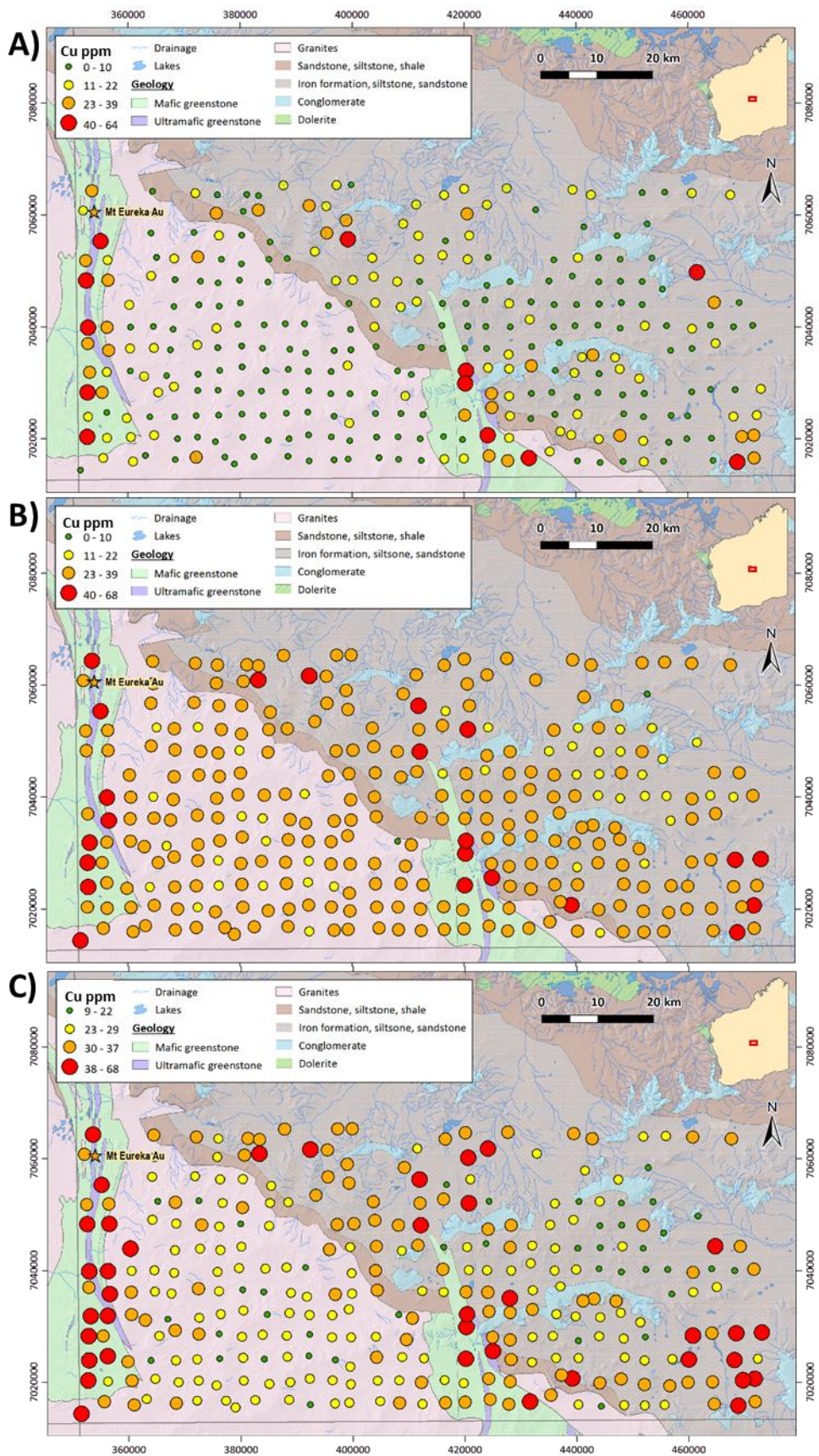
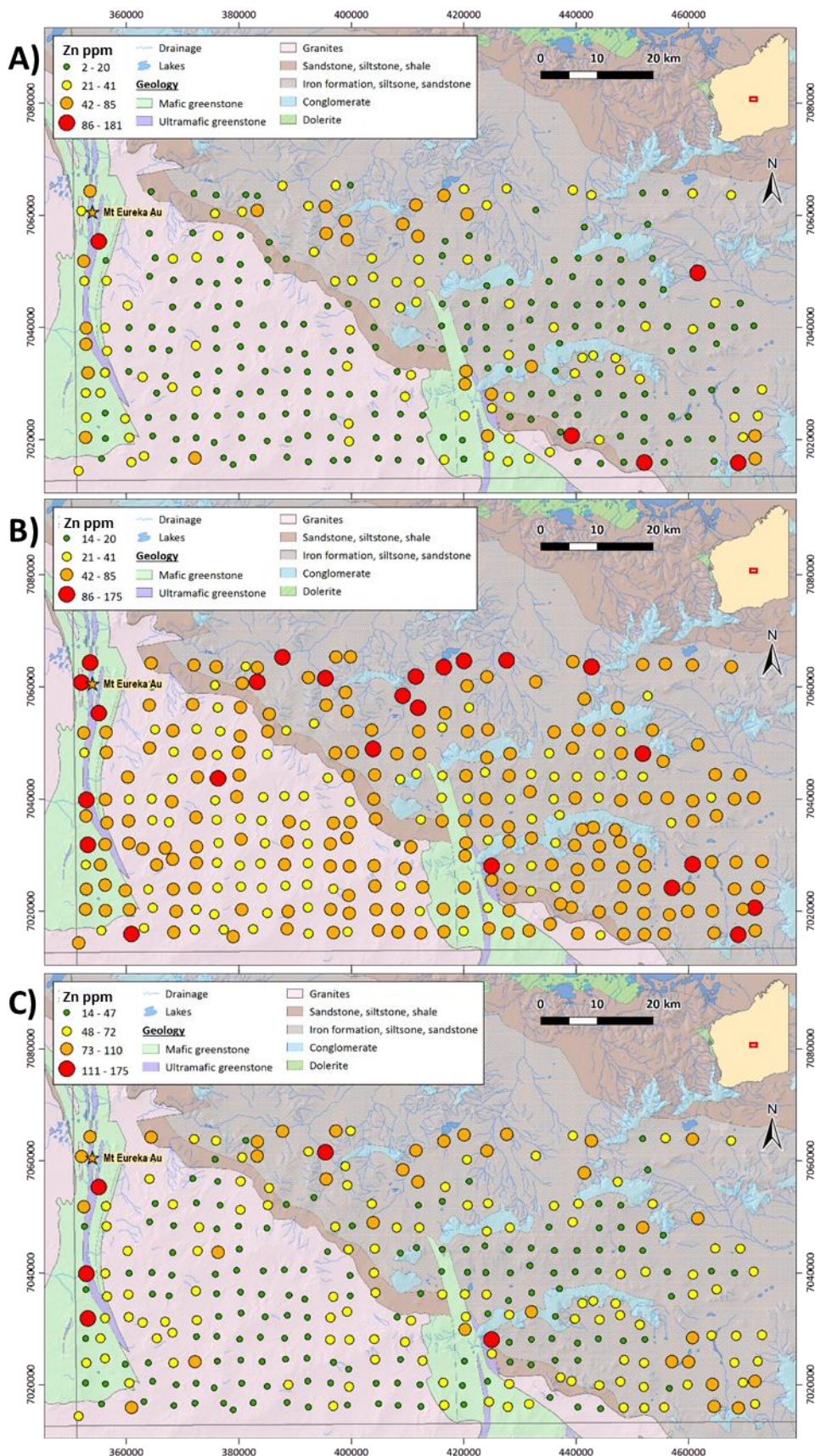


Figure 37. Copper (ppm) in Kingston soils. A) historic data (Pye et al., 2000), B) the >2 μm fractions Cu using the same scale as the historic data, C) the >2 μm fractions with natural breaks in scale to better identify anomalous areas.

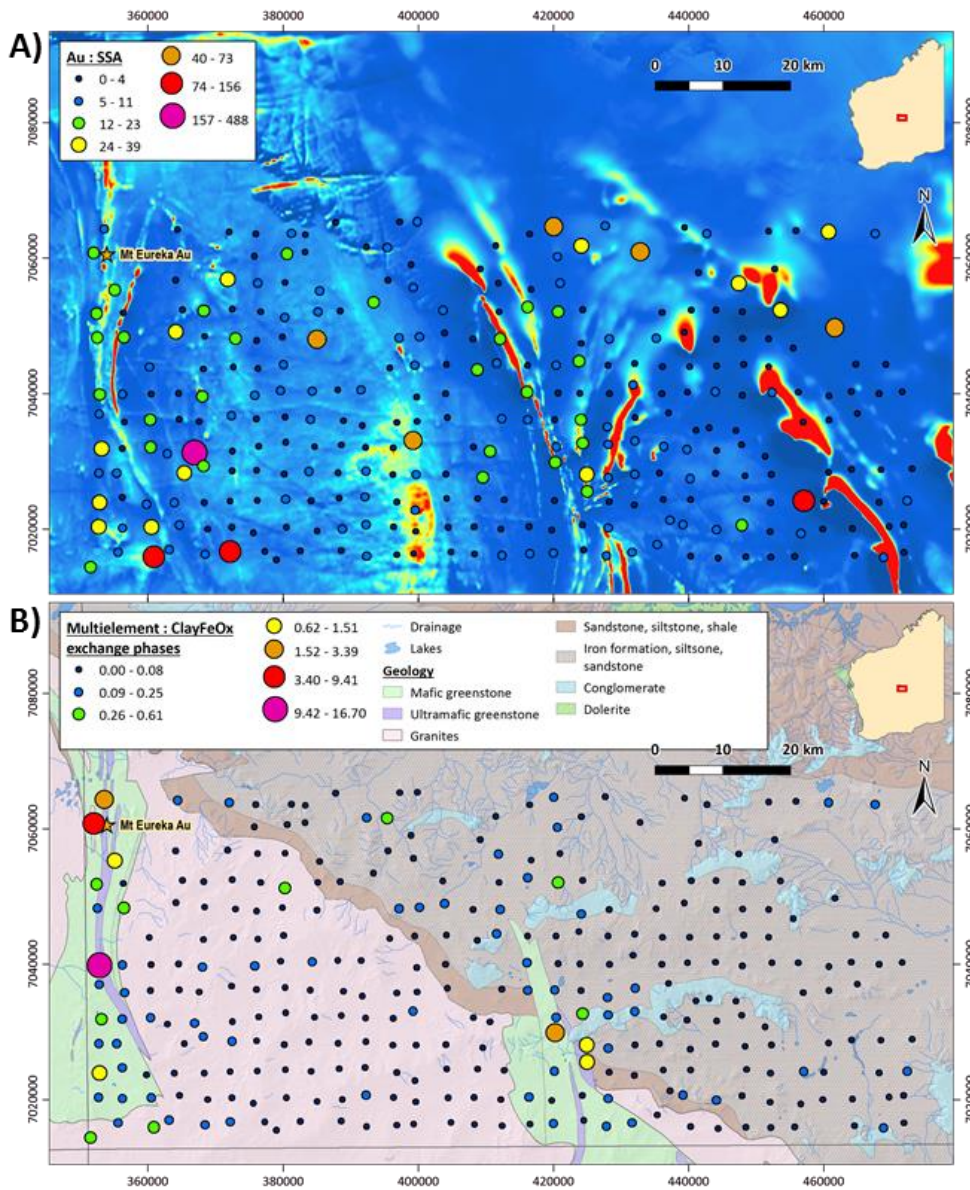




**Figure 38. Zinc (ppm) in Kingston soils. A) historic data (Pye et al., 2000), B) the  $>2 \mu\text{m}$  fractions Zn using the same scale as the historic data, C) the  $>2 \mu\text{m}$  fractions with natural breaks in scale to better identify anomalous areas.**

The results of these new maps are the change in focus potentially for some of the Au regions of interest. In Kingston this may not prove that valuable, but could certainly prove effective elsewhere, especially when soil types vary significantly and can create a lot of false positives. The other exploration ratio shown here as

an example does pick out the Mt Eureka region and the further extent of that greenstone belt as prospective, but it also highlights the western edge of the other greenstone (Figure 39). This area was of interest in a number of elements (Au, Ni, Pt), so while it may be identified as an area of interest using single elements, there is a lot of potential to use multiple variable and additional information such as particle size and mineralogy proxies to improve exploration and discern false positives (or negatives).



**Figure 39. New potential map products using the UltraFine+ workflow. A) Gold adjusted for the specific surface area of the samples overlying the magnetics. B) A typical multielement index that is now adjusted with respect to an increase in exchange phases with clay and Fe oxides. Geology is generalised and based on the data from GSWA (2014).**

### 12.1.2 Telfer West prospect

The two soil sample lines completed at Telfer West were compared to the nearby drilling data that included composite downhole geochemistry and geology logs. The cover is approximately 50 m to some supergene Au in the weathered oxide zone. Other target elements of interest, that are somewhat enriched at depths include As, Bi and Co. From the surface soils analysis using the <2 µm material, the highest As (11-12 ppm) concentrations in the fine fraction align with the more As-rich oxidised Proterozoic rocks below from drilling data drill holes ETG 0029 and 0030. The lowest concentrations are 7 ppm and although it can be highlighted when scaling the data in percentiles or similar, the results are essentially background. Bismuth is also a vector element at this prospect, but the Bi data in the <2 µm fraction was at the detection limit and not used. Gold in the surface is very low concentrations, not anomalous, and the highest concentrations of 2.7 ppb. Anecdotally, the world class Cu-Au Telfer deposit was reported, in hindsight, to have been outlined by a 2

ppb gold anomaly in soil originally (K. Hodgson *pers. comm.*). Even though this subtle geochemical gradient did not provide the discovery, it does show that these subtle surface features may still have value in these settings of thick cover. No clear evidence exists in linking the surface expression to Au at depth, and the cover is thick in this region (50 m).

Cobalt, Cu, Ni, Pb, Rb and K all are at their highest concentrations toward the southernmost part of the survey (Figure 40). The elevated values are not highly anomalous by any means with the range of Cu 22.4-35.5 ppm, all essentially background values for soils. These parameters also correlate to the spectral response of the hematite/goethite ratio, providing additional information from the UltraFine+ method to assess these values that would not be available to standard geochemical assays. The congruent increase in K may be indicative of clay changes. The spectral response for the 2250d spectral feature that is potentially related to Fe substitution into kaolinite (not shown), is the other parameter that provides evidence of these changing soil conditions. In general, this indicates that this southern elevated soil chemistry may be a false positive, just a soil type transition, and not a location to prioritize for follow up sampling. This site, although not clearly seeing through cover, highlight the value of the addition spectral scalars to assist the geochemical interpretation. These higher elemental values are in the thickest cover of the region (nearly 80 m, log from ETG0028). Adjusting the results for the hematite/goethite shift suppresses these values somewhat and it also further enhances the results for S and Sb, and what appears to broaden the Sb dispersion at surface (Figure 40). The S and Sb results were the only other interesting feature in the UltraFine+ soil data at this site. Most pathfinders have the greater concentrations in the same samples, Sb and S are distinctly different with an anomaly that most closely aligns with the potential mineralisation (drill hole ETG0027). Antimony in soils is not highly anomalous, it is also not clearly anomalous in the limited down-hole, whole rock geochemistry available. Presently, this appears to be a weak concentration gradient at the surface and unrelated to mineralisation.

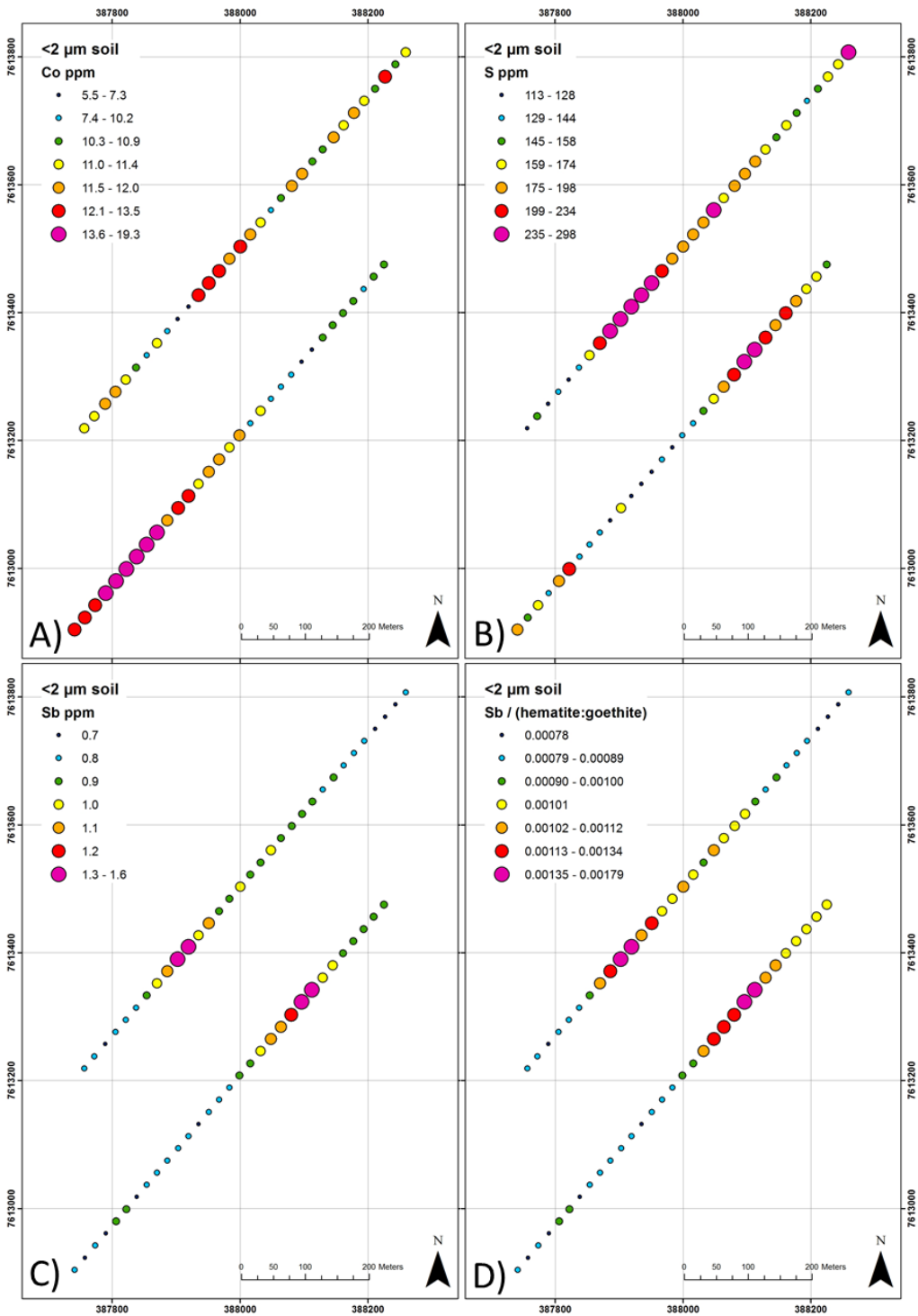


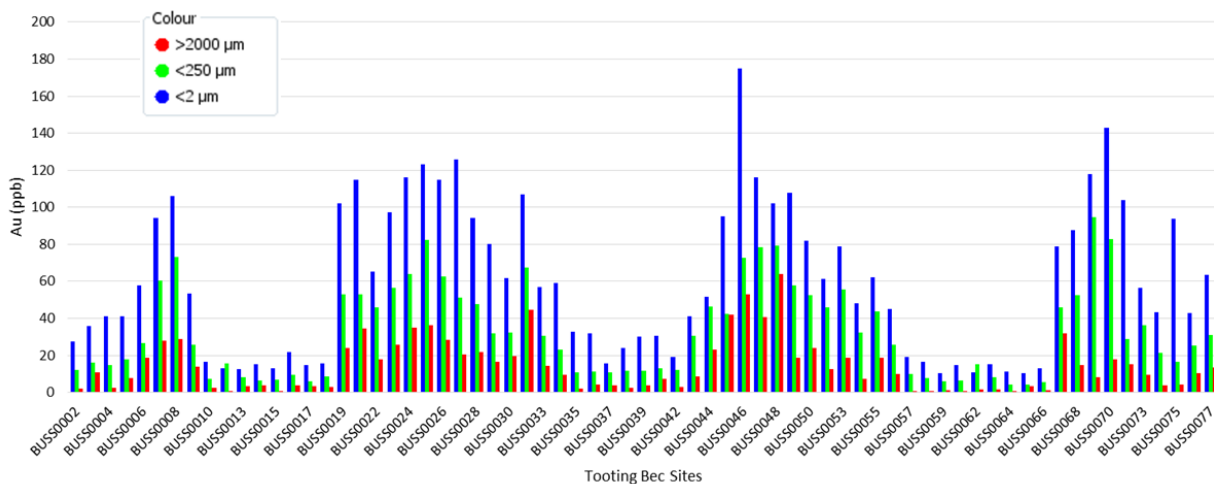
Figure 40. Surface geochemistry at Telfer West. A) Co ppm, B) S ppm, C) Sb ppm, D) Sb adjusted for the change in hematite to goethite ratio from spectral mineralogy.

### 12.1.3 Tooting Bec prospect

Tooting Bec as a prospect probably best represents exploration soil sampling in the southern Yilgarn. At this site the underlying target was not well defined, but the central section of the sampling was most likely the target area and is evident in the sample spacing. This site specifically looked at the comparison of three size fractions <2 μm, <250 μm and <2000 μm.

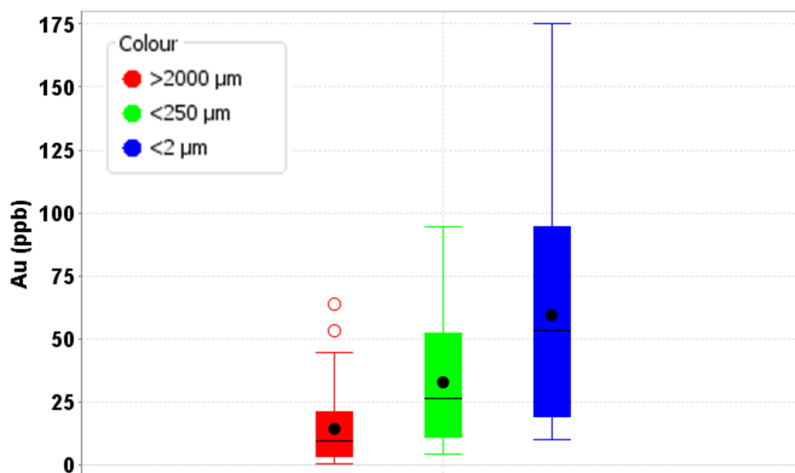
Other studies have shown the coarse pisolitic lag can sometimes be an effective sample media in the Yilgarn Craton, and concentrate Au particularly in the cortices of the pisoliths (Anand & Butt, 2010; Anand et

al., 2016b). At Tooting Bec, however, the fine fraction consistently had much greater Au concentrations ~250% more (Figure 41).



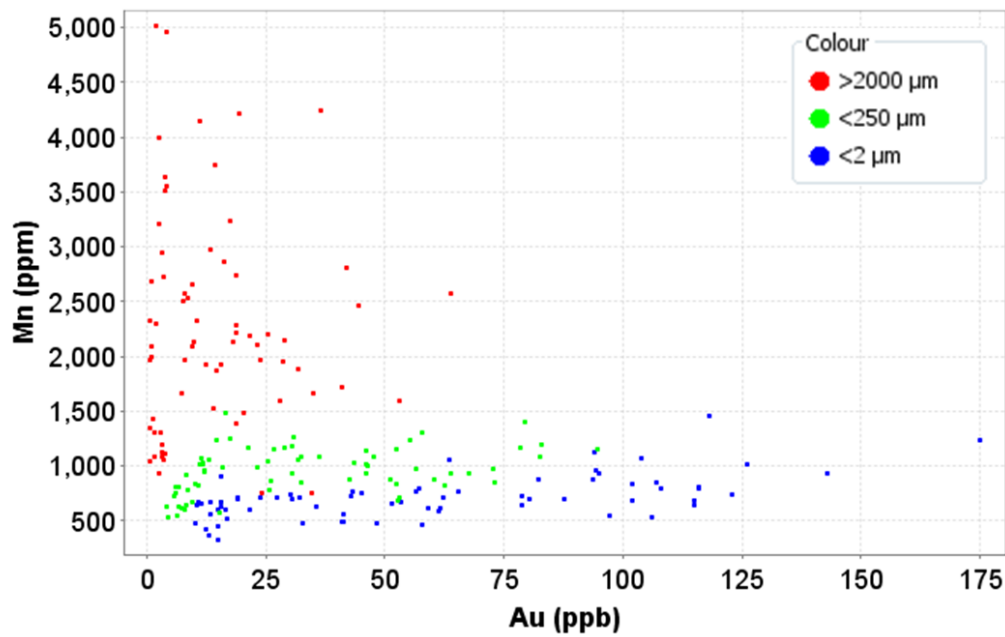
**Figure 41. Individual sample comparison of Au in the different particle sizes analysed.**

The average HARD for Au was 5% in the <2 µm fractions and no samples were below detection or close to the detection limit of 0.5 ppb (Figure 42). The Au concentrations in the <2 µm fractions at this site range from 10.3 to 175 ppb with a mean of 59.5 and a median of 53.4 ppb. The box plots don't show the individual sample comparison and Figure 41 better shows this comparison at each sample point. The pattern is very consistent and obvious for Au. Arsenic, another common pathfinder in this region, is also slightly greater in the <2 µm fraction. For Cu and Zn the fractions seem similar which was observed in the method development (Noble et al., 2018a). Although similar, the coarse fraction tends to have slightly more Cu and Zn in selected samples.



**Figure 42. Boxplot showing the data distribution for each of the three separated size fractions at Tooting Bec. The black bar is the median and the black dot is the mean.**

Iron and Mn oxides can influence trace metal distributions and have been used as a target phase for partial extractions (Chao & Zhou, 1983; Hall et al., 1996). The results at this site show the Mn (Figure 43) and Fe (not shown) do not vary much in the <2 µm fraction and do not have a relationship to Au. As the size fraction increases, the Mn (and Fe) concentrations vary much more. Iron is sometimes used as an element to level data and these results show that the UltraFine+ method would not require this adjustment and performs potentially better than the coarser fractions at Tooting Bec.



**Figure 43. Gold and Mn scatterplot with results coloured by size fraction.**

Fine fractions Au, and Cu and Zn are anomalous over the central region and most prospective target (Figure 44). This is similar in the other fractions, too, although numbers are not as great, so there is less contrast in the coarser fractions. The increased contrast at this site provides more evidence for the advantage of the <2 µm fraction analysis and the UltraFine+ method. The greatest Au concentrations in the soils at Tooting Bec trend in a NW/SE direction and may be related to landform more than the possibility of reflecting the inferred strike of mineralisation which is believed to trend more N-S. With limited additional site data at present, the <2 µm fraction analysis performs better than the other two size fractions for the geochemistry alone.

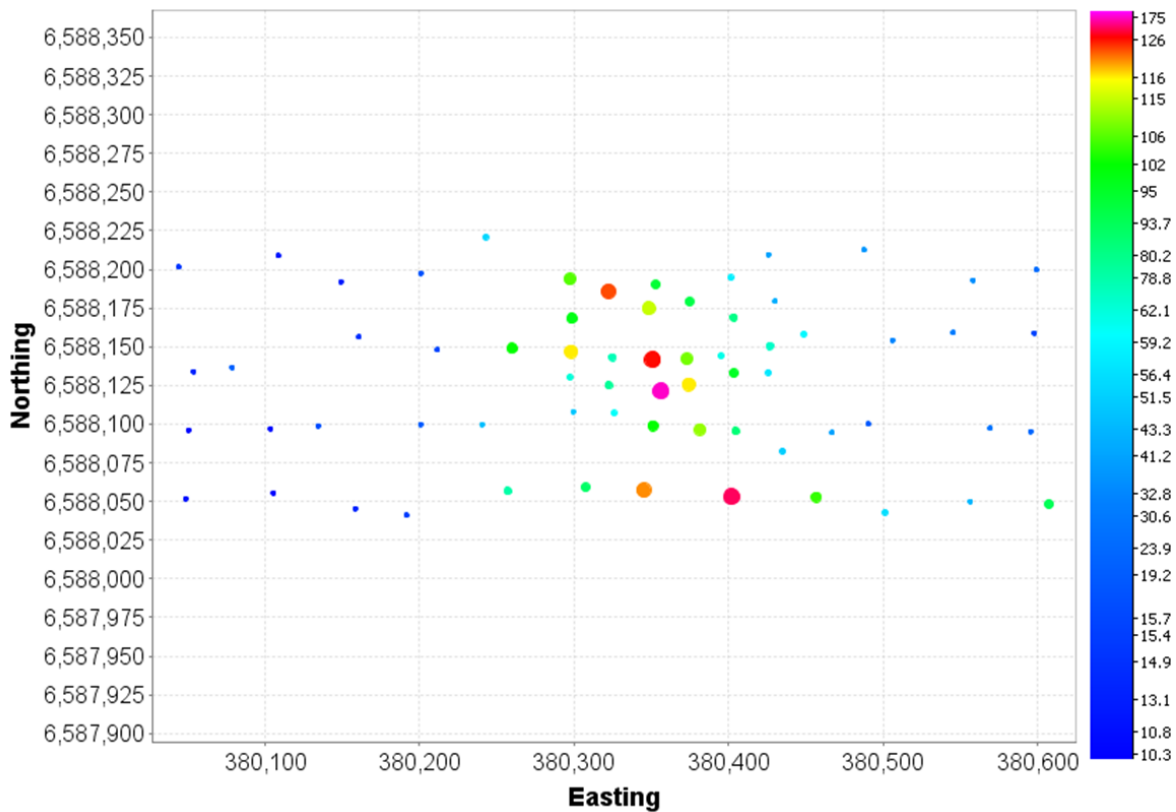


Figure 44. Gold from the <2 μm fraction at Tooting Bec.

#### 12.1.4 Area 7 prospect

In a similar test as Tooting Bec a number of previous analyses of the site compared particle size fractions and the best geochemical response. This site is in the Northern Yilgarn Craton, further north than the Bronzewing deposit results that were mentioned in the Sir Samuel regional study. These areas have been successfully explored using laterite samples, and subsequently coarse grained pisoliths would be an effective sample medium (Anand & Butt, 2010). This is in agreement with the previous work where the coarse grained soils were deemed the most effective exploration medium in this area. The previous work used the following size fractions with fine being 250 μm to 500 μm, medium 500 μm to 800 μm, and coarse being 800 μm to 1200 μm. Clearly, much larger than the <2 μm fraction analysed using the UltraFine+ method in this research. The new method extracting the <2 μm particle size, somewhat unexpectedly shows a similar result to the coarse fraction i.e., providing as good or better results than previous surveys although at lower concentrations with the coarser fractions having ~50% more Au. The explanation for the similarity may be linked to the research that has shown pisolith rinds to sequester Au quite effectively (Anand et al., 2016b). These rinds, although now very resistive to weathering, are made up of fine Fe oxides and clays. The same material targeted in the UltraFine+ extraction. We believe the extraction is getting some of this signature off the coarse material coatings, just not as much as is the case when these grains are crushed and metals liberated. This is one example where the dilution effect of the matrix is not a problem. Figure 45 shows the results of the <2 μm and the two main prospects in the area – Drifter and Le Blaireau. The target to the south of the survey (red outline in Figure 45) was not detected in any method applied and the fine transported soil cover in this area does not appear to have a signature from the subsurface.

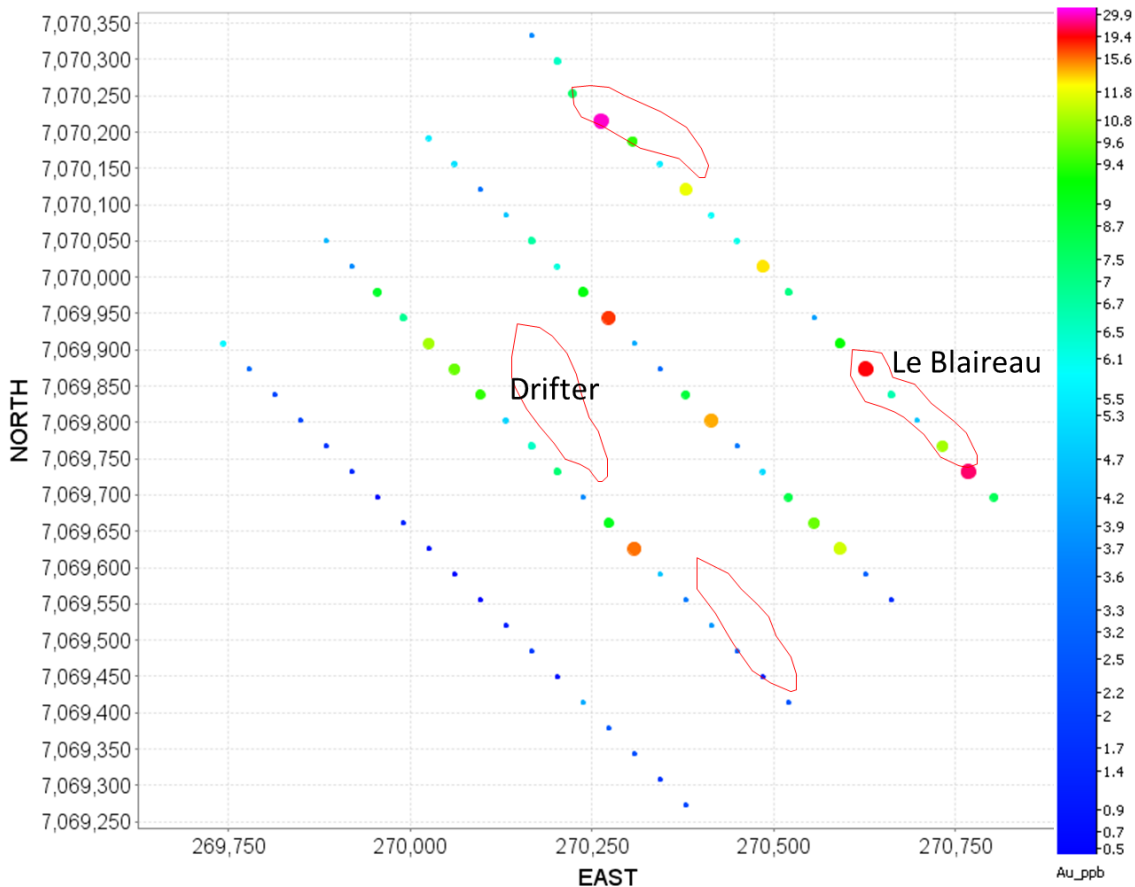


Figure 45. Distribution of Au (ppb) extracted using the UltraFine+ method at the Area 7 prospects.

### 12.1.5 DeGrussa deposit

The DeGrussa site was previously sampled, analysed and reported by Noble et al. (2017a) investigating a variety of sample media and the mechanisms of metal migration at this site. Soil orientation traverses used the coarser <250  $\mu\text{m}$  fraction and aqua regia extraction, which is directly comparable to the <2  $\mu\text{m}$  UltraFine+ extraction method, with the main difference being the sample grain size. The comparison test shows that both extractions effectively identify the mineralisation using both Au and Cu (Figure 46). The concentrations extracted in the <2  $\mu\text{m}$  fraction is greater and the size of the anomaly a bit wider, making it arguably a better method (Table 18; Figure 46). The increased size of the anomaly more closely matches the dispersion model suggested by Noble et al. (2017a) where Au was mobilised in the lateritic materials towards the palaeochannel to the north and outside the oxidised Au zone, whereas Cu was more uniformly dispersed in the clays and Fe oxides, and matched the secondary Cu zone offset towards the south. The <250  $\mu\text{m}$  fraction had slightly more contrast (lower background) and in effect, the methods are very similar (Figure 46). The advantage of the UltraFine+ with additional parameters such as pH, EC, particle size and spectral mineralogy did not add more to the exploration potential with this first-pass assessment.



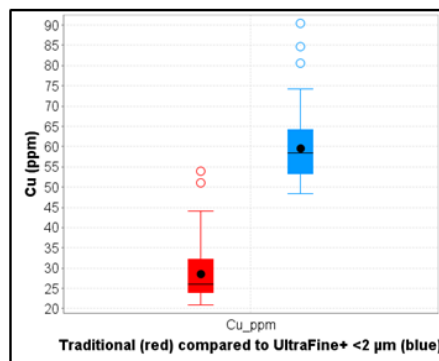
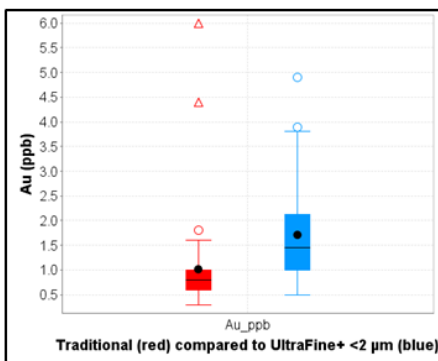
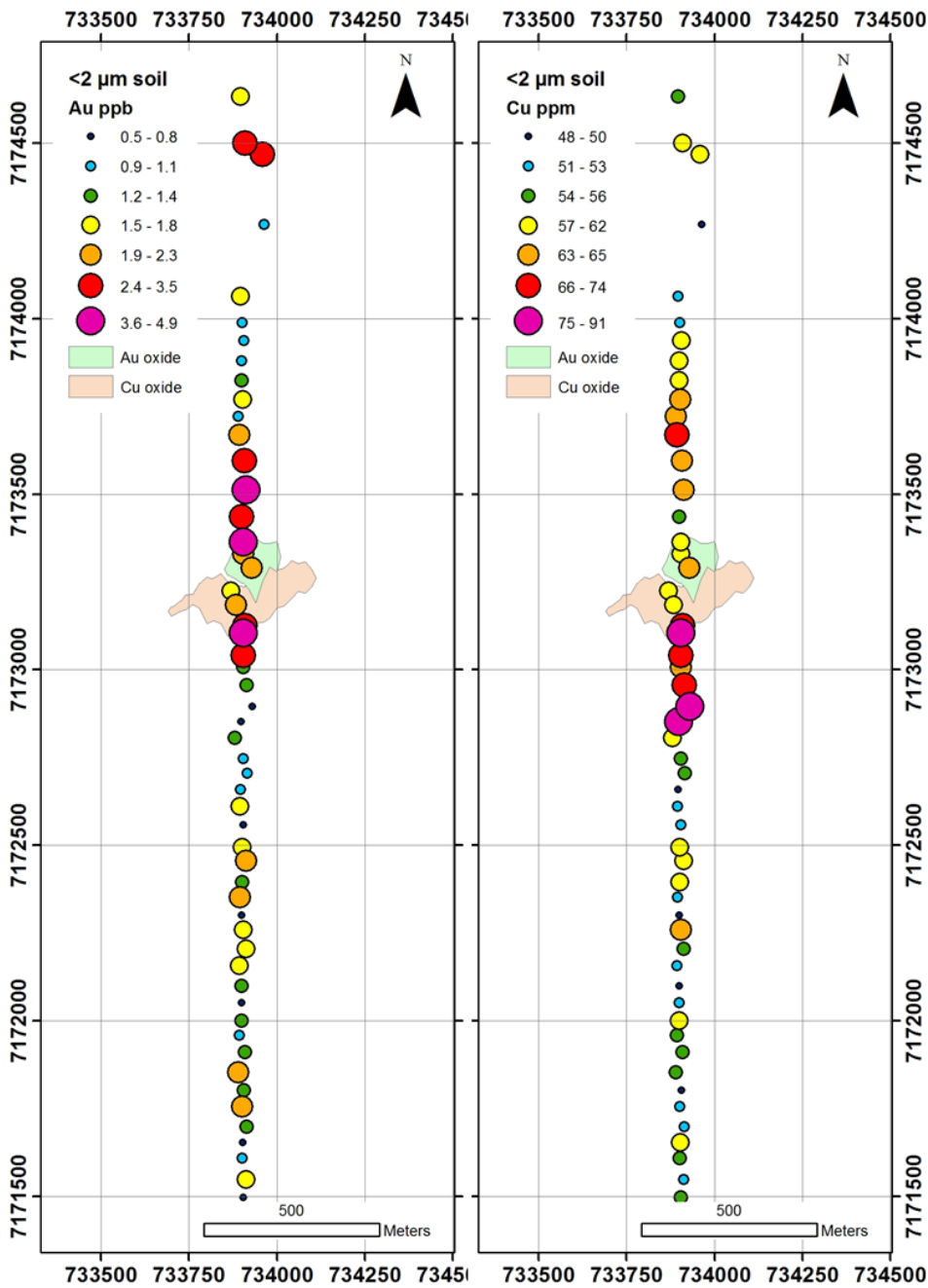


Figure 46. Orientation traverse at DeGrussa with results for the UltraFine+ shown for Au (ppb) and Cu (ppm). Box plots inset show the differences in concentrations between UltraFine+ and standard aqua regia methods for both elements. The secondary dispersion of ore-grade supergene Au and Cu depicts the mineralised zone.

**Table 18. Summary statistics. n= 54, 0% censored. All values in ppm except Au in ppb.**

Aqua regia with <250 µm soil fraction						
	Min	Max	Mean	Median	StDev	95 pctl
Ag	0.006	0.024	0.011	0.01	0.004	0.02
As	3.1	5.7	4.01	3.9	0.55	5.1
Au	0.3	6	1.02	0.8	0.90	2.5
Cu	21	54	28.5	26	7.3	45.8
S	50	400	104.6	50	94.3	362.5
Zn	12	47	19.3	18	6.2	32.3
UltraFine+ <2 µm soil fraction						
Ag	0.04	0.11	0.054	0.05	0.012	0.08
As	6.1	9.9	8.02	8.1	0.91	9.4
Au	0.5	4.9	1.71	1.45	0.95	3.8
Cu	48.3	90.5	59.5	58.4	8.7	81.6
S	127	1050	445.4	404.5	215.4	895.5
Zn	67.4	350	116.3	103.5	50.3	233.8

The surface signature of the secondary, supergene mineralisation at the DeGrussa Cu-Au sulphide deposit is evident in both methods tested and other sample media (Noble et al., 2017a). The transported cover is shallow here, (<10 m), and testing this in the deeper cover (30 m) further south should be considered. Earlier studies had observed a subtle signature at this depth and estimate the depth limitation in this environment and many others in Western Australia to be <10 m e.g., the Jaguar and Moolart Well deposits (Anand et al. 2007, 2009). During weathering the Au and Cu have mobilised separately forming different dispersion patterns and the <2 µm fraction analyses is as suitable as any in detecting this signature.

### 12.1.6 Calibre deposit

The surface geochemistry of Au, Cu, As, Mo, Sb and W shows very low concentrations at Calibre and surrounding regions. From the other study at Telfer West, the soil chemistry seems consistent, although previous MMI analysis seemed to show much greater concentrations that were not clearly aligned to the deposit. The UltraFine+ method has previously been applied to soil and one shallow profile at North Mittel (not shown), so this site was a different application where the analysis was applied to deep profiles through cover. A number of profiles show elevated metals in the interface between the ferricrete and the Permian sediments (Figure 47). Copper and As are 200% more than the background at the interface. Other profiles further away from the mineralisation at this site also show elevated concentrations and there is no consistent anomaly immediately above mineralisation. Looking at slices through the regolith over the interface (not shown), concentrations >40 ppm Cu are evident in one particular hole over mineralisation, but also one sample approximately 400 m away from the central mineralised zone. A Cu concentration of 40 ppm would be considered background in many other settings, but in the Patterson Province in these sand plain areas, these concentrations may be anomalous. At this site, 95<sup>th</sup> percentile results in the upper regolith profile (i.e., excluding bedrock interface) are 2.5 ppb Au, 26.5 ppm Cu and 35.1 ppm As (n=107).

This pattern of enrichment in the ferricrete-Permian interface has been observed in a near identical cover sequence in the Yamarna belt more than 800 km SSE and also shows this same trend with respect to Au (Salama & Anand, 2018). The key challenge here is to identify the association of these elevated element concentrations with the underlying mineralisation. Research at the Kintyre U deposit in the Patterson Province with a similar cover sequence also noted some anomalism at this interface, but concluded it was not associated with the underlying mineralisation and rather shedding from adjacent outcropping mineralisation and subsequent lateral dispersion along the interface aligned with the water table (Noble et al., 2017b). At Kintyre, the interface with enriched target element concentration in the profile was just coincidentally aligned with another underlying mineralised zone.

The research at Calibre cannot confirm nor reject the possibility of mobilisation of metals from depth. However, it is likely that the elevated Cu and other target or pathfinder elements in the interface are part of a much broader geochemical pattern, perhaps providing a broad-scale shallow drilling sample media that could be analysed with the UltraFine+ workflow, but not directly targeting the underlying orebody. This potential pattern would require further research and analysis to establish the extent of element dispersion at the interface.

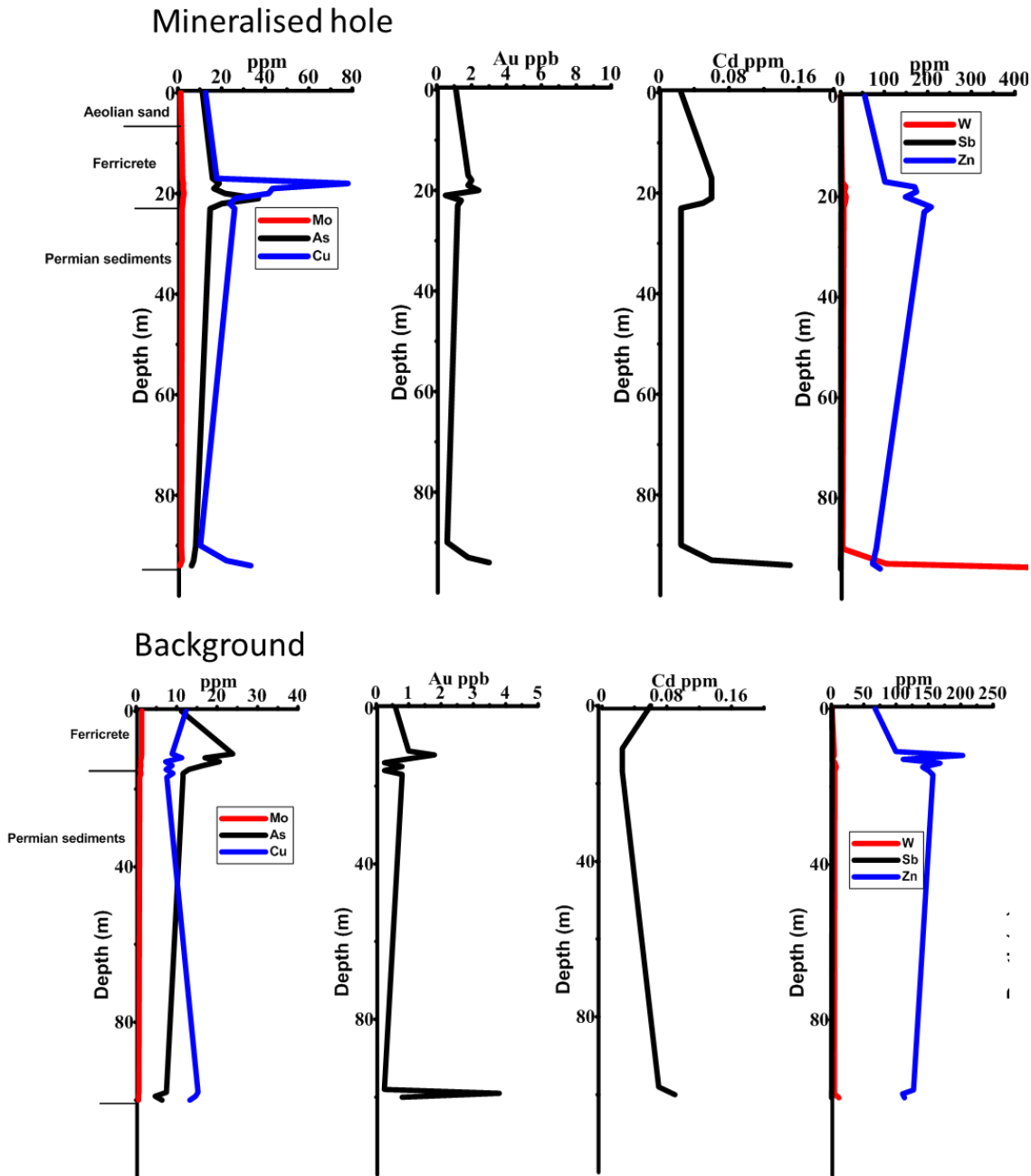


Figure 47. Profile geochemistry using <math><2 \mu\text{m}</math> fraction. The top profile is over the main Au-Cu-W mineralised zone whereas the lower profile is further away. Both profiles are in thick cover over. Note the concentration scale on the x-axes are not identical.

### 12.1.7 Other studies and the application to the UltraFine+ method

Although focusing on exploration through cover, the application of fine fractions to stream sediment sampling shows the value and confidence in reproducibility the fine fraction can provide. A Canadian stream

sediment survey wasted vast resources and money, in the analysis of stream sediments for Au, when the reproducibility of the selected analytical method showed extremely poor reproducibility (Arne & MacFarlane, 2014). The results delayed gold exploration in the region by many years; an area that is now known as the White Gold region and hosts numerous gold deposits e.g. Coffee (Wainwright et al., 2010; MacKenzie et al., 2015). A test of just a selection of these sediment samples shows reproducibility is poor with a number of other standard methods used in the industry such as BLEG or a slightly finer fraction (<177 µm), but the very fine fraction (<4 µm, mostly clay) produced excellent reproducibility of Au (Arne and MacFarlane, 2014). The result demonstrates the immediate impact improved methods can have on previously collected samples and regional survey data. Although the reproducibility was solid in the earlier regional survey work by GSWA in Western Australia, the change in the amount of data reported for Au (major decrease in censored data) has seen major improvements realised in the samples used as part of this study, too.

The preliminary analysis of East Wongatha sandplain soils showed samples are predominantly coarse material, and initially report Au concentrations of less than detection (0.5 ppb) up to about 30 ppb (Noble et al., 2013). Separation and concentration of the <2 µm samples proved the detection limits were obsolete as the concentrations were much greater, i.e. in this example there was no censored data. The results of Noble et al. (2018a) and the results in this paper support the observed increase in concentrations reported by Noble et al. (2013), but not to the extent shown in the earlier study. The average increase in Au using the <2 µm fraction is in the order of a 150-250% increase. While this is not as great an increase as earlier results had suggested, it is important to recognize that from approximately 1000 different WA soil samples, most from transported cover, <10% were below detection for gold; an exceptionally valuable contribution for exploration soil sample geochemistry in areas of dominantly inert quartz-rich cover. Previous analysis of these soils would have resulted in >70% of samples below detection limit for Au based on the three regional geochemical surveys used in this study (Sir Samuel, Leonora and Kingston; Bradley et al., 1995; Kojun et al., 1996; Pye et al., 2000). These surveys are representative of soils sampled in the Northern Yilgarn Craton, the Capricorn Orogen and various basins to the north and further east in Western Australia where transported cover thickens and becomes a major challenge for mineral exploration. An additional benefit to this reduction in censored Au data is to statistical interpretation, as the method shifts a lot of geochemical data to a normal distribution where previously it is heavily skewed due to samples being at or below detection limits. This specific issue is further discussed by Leybourne & Rice (2013), albeit for general geochemical data and not fine particle material analysis. The results of the research presented here and in other referenced studies supports the idea that the <2 µm geochemistry significantly reduces the nugget effect, even though precision is not always as tight as expected (Noble et al., 2018a).

Nugget effects are notoriously a problem for both resource definition and mineral exploration gold (Stanley, 2006). The research presented in this paper has not attempted to test the UltraFine+ workflow for resource definition. And while it may be beneficial, the design of the workflow is very much aligned with near surface soil sampling for exploration. The integration of spectral data and other physico-chemical parameters are not likely to improve resource definition or the nugget effect influence further, yet they are seen as the next generation of routinely collected data. Much like explorers went from single element assays to multi-element assays, we believe the next decades of exploration will see multielement data integrates with spectral mineralogy and other parameters as part of the “standard assessment”. Spectral data was collected extensively in this study, yet has been a lesser component of the evaluation. Like the industry, research is also attempting to catch up with the development of big/more data and how we can best use it. Spectral data did show evidence of mapping lithology trends in some cases, picking up felsic volcanics in the Leonora map sheet (not shown), but this is likely to be the next progression of this workflow, stronger integration of the “other” data.

Other data can also map lithological changes and integrating these is important to future applications of exploration geochemistry. Morris (2013) showed aeromagnetic signatures of greenstones extended under cover and the <50 µm soils tended to pick up this signature where it was not seen in other geochemical results. The distribution of samples with higher concentrations of fine-fraction Au, Cr and V in soils shows a strong relationship to Yilgarn Craton greenstones which is consistent with the established connection between Au mineralization and greenstones established elsewhere in the Yilgarn Craton (e.g. McCuaig et al. 2010; Blewett et al. 2010; Morris 2013). The numerous observations of bedrock or even bedrock-hosted

mineralisation being identified through transported cover (Kelley et al., 2003; Wang et al., 2007; Morris 2013; Anand et al., 2014; van Geffen et al., 2015; Noble et al., 2017, 2018b) does imply some migration of these materials through the cover. In the Kingston map sheet, drainage is minimal and not related to the signatures of greenstones in the soils (Figure 34) which would imply coincident shedding of greenstone materials from those belts subcropping further south.

We see these greenstone extensions in the Kingston map using the <2 µm fraction. These extensions trend north under the Earraheedy basin and should be considered for future exploration. Major exploration success in recent times in Western Australia has come from explorers stepping out into the lesser known and more covered greenstone belts in Western Australia. Examples of Gruyere Au deposit in the Yamarna greenstone belt and the Nova Bollinger Cu deposit in the Albany Fraser region are clear examples of industry successfully pushing into lesser explored terrains. The edges of the Yilgarn Craton, particularly the NE as investigated in this study are newish prospective terrains. The techniques and results developed in this project should assist more exploration in the Kingston area and other Greenfields settings.

The study by Noble et al. (2013) provided early indications that the very fine (<2 µm) particle size fraction may be important for exploration, and provide a significant advantage over the standard <250 µm assays. This study, for both regional and site based orientation, confirms that the finer fraction is indeed providing much more information. These results are also supported by other research (Arne & MacFarlane, 2014; van Geffen et al., 2012; Robertson, 1999; Carlson, 2016; Sader et al., 2018). Another demonstrative study published by Anand et al. (2014) shows the <53 µm had much greater concentrations of gold over the Moolart Well deposit whereas the commonly used <250 µm soils were ineffective, and likewise the results of Morris (2013) suggest a similar size fraction in East Wongatha identified other targets through transported cover. Although not as fine, the <53 µm is an improvement and it seems the <2 µm is a step change in near surface exploration geochemistry.

Much of this research has focused on the challenges of gold exploration, the same potential challenges and opportunities are available in exploration for base metals, REE and more, as these commodities also commonly favour regional soil sampling and geochemistry to refine targets. Recent studies in thick transported cover for base metal exploration in the Kalahari Desert show the greater metal content is in the fine fractions, with the bulk material showing Pb and Zn concentrations near detection limits and being comprised of coarse, quartz-dominant sands. Analysing fine fractions results in samples that are orders of magnitude above detection limits (Salama et al., 2016).

Evidence and examples of the very fine particle size geochemistry greatly improving results, particularly in areas where the amount of censored data are problematic, the question now is, how many similar samples have been overlooked in bulk analyses by the exploration industry? Immediate impact of the improved methods developed by Noble et al. 2018a could be realised by industry and surveys re-testing their historic samples; an immense resource that remains in many industry and government survey storage areas.

As a parallel example, another recent study demonstrated the value of re-assaying approximately 100 regional samples with improved methods for Cu-Au exploration. Baker (2015) showed that the Wafi Golpu area in Papua New Guinea stood out clearly using finer size fractions (<75 µm) and better detection limits with a multi-element anomaly where it was overlooked in earlier sampling and analysis. Not only does this demonstrate the immediate value that can be recognised in re-assaying samples with an improved method such as UltraFine+, but Baker et al. (2015) go further to demonstrate there are more than 10,000,000 samples/geochemical results with open access and there are many times this in archives of industry that could benefit from this type of research and application. The opportunity for industry to regenerate exploration interest using the UltraFine+ workflow is highly significant, just based on samples in storage alone. However, the industry needs to be shown the value of such an exercise, and this study has delivered the initial demonstration in areas of cover in Western Australia and has transferred the technology to a commercial laboratory for easy access. It is envisaged that more commercial laboratories will take up this workflow for future offering to industry to improve mineral exploration globally.

## 13 Conclusions

The new adaptation of methods known as UltraFine+ extracts the  $<2\ \mu\text{m}$  fraction of soils and sediments, provides an assay of the geochemistry and combines this with additional spectral mineralogy proxies and physico-chemical properties to improve soil analysis for Au and base metal exploration. Applying this to exploration examples showed promising results at a number of small orientation test sites. Of most relevance, the study revealed a marked decrease in censored results for Au (~70% to 10% below detection limit) using historic samples and re-assaying them to produce a new geochemistry map of the Kingston 1:250,000 map sheet. The new maps show geochemistry and some example indices for mineral exploration and lithology mapping, as well as examples of new interpretations using the additional spectral mineralogy proxies or particle size variation. Adding spectral mineralogy, particle size and other physico-chemical parameters to this style of mapping is valuable and is not commonly done, and certainly not integrated. Future research will improve the UltraFine+ method, particularly building algorithms to cloud-process the various data streams, and this should be part of the service from commercial laboratories in the future. The application of the  $<2\ \mu\text{m}$  particle size separation and the UltraFine+ workflow importantly demonstrate the additional value from (re-)assaying regional soil and sediment samples to generate new targets and improve regional geochemical maps. This is an exercise that can be applied to new greenfields surveys, and when exploration budgets are lean, applied to abundant, historically collected samples.

## 14 References

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# Appendices

All the data for the project is provided in the multiple tabs of the following Excel spreadsheet

M462 Sponsors Final Data Release Sept 2018.xlsx

This file “MRIWA M462 Report Multi-scaled near surface exploration using ultrafine soils” has been published via the CSIRO Data Access Portal (DAP) at

It is available to view at <https://data.csiro.au/dap/landingpage?pid=csiro:36621>.

Persistent Link: <https://doi.org/10.25919/5bd139f3a66fa>

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Access: The metadata and files (if any) are available to the public.

It is expected this data will also be accessible from the GeoView platform of GSWA within 4 months of this report being published.

Orientation sites do not have the sponsor sample locations provided specifically. Consult Table 3 for site locations.



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### **Mineral Resources**

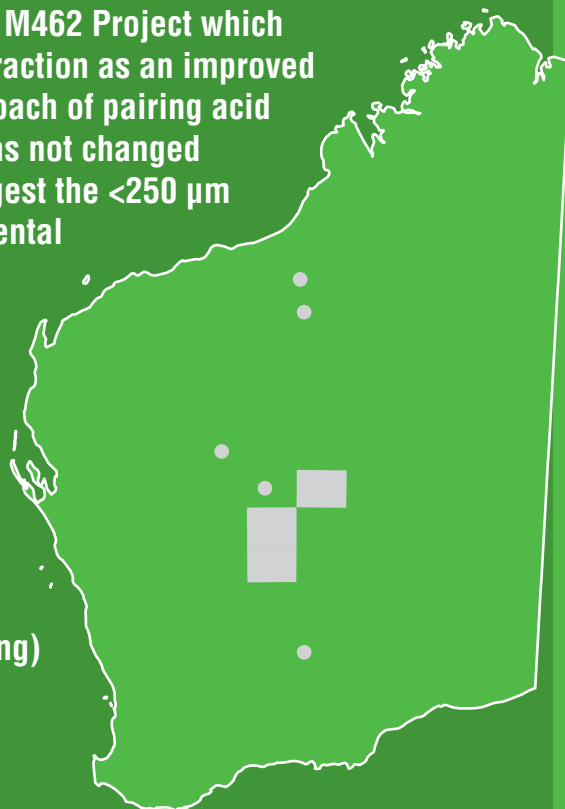
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This Report presents the results of the MWIRA M462 Project which was designed to test the  $<2 \mu\text{m}$  soil clay size fraction as an improved sample medium for metal detection. The approach of pairing acid digestion and multi-element measurements has not changed significantly over the past 30 years: that is, digest the  $<250 \mu\text{m}$  soil fraction and analyse the solution for elemental concentrations. In transported regolith cover, the mobile element signature is contained in the smallest size fractions. UltraFine+ provides a new analytical workflow and methodology to separate the  $<2 \mu\text{m}$  soil and sediment fractions for multi-element analysis along with other, commonly not utilized physico-chemical parameters that should aid exploration. UltraFine+ is a commercialized platform (UltraFine+ certified trademark pending) with demonstrated success in experiments, orientation field surveys and new regional geochemical map products.



Further details of geological products and maps are available from:

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