

Determination of micro and nanoparticulate fraction gold in regolith

INTRODUCTION

Small gold particles ($<2\ \mu\text{m}$) in regolith have recently been observed using field emission gun scanning electron microscopy (FEGSEM) (Hough et al. 2008), but the amount of micro and nanoparticulate gold cannot be quantified by this method. Knowing the amount of micro ($<2\ \mu\text{m}$) and nanoparticulate ($<0.2\ \mu\text{m}$) gold in soils and regolith in areas of mineralisation is important to understanding the formation and discovery of economic deposits, and contributes to knowledge of how secondary supergene enrichment and surface geochemical anomalies form.

A method to quantify gold in the microparticulate and nanoparticulate fractions was tested using soil samples from the East Wongatha area of Western Australia, where a

regional regolith geochemical survey was carried out by the Geological Survey of Western Australia (Morris 2013). This survey showed that the $<50\ \mu\text{m}$ fraction of some regolith samples hosted appreciable gold content determined using an aqua regia digestion, when most samples were expected to be below detection limit ($<1\ \text{ppb}$). The East Wongatha area is composed of Archean rocks of the Yilgarn Craton, and Archean – Proterozoic rocks of the Albany-Fraser Orogen, both of which are unconformably overlain by Proterozoic - Paleozoic rocks of the Gunbarrel Basin (Spaggiari et al. 2011; Fig. 1). Unconformity-hosted uranium, gold and base metal mineralisation has been reported from the margin of the Gunbarrel Basin, and elsewhere in the Yilgarn Craton, where gold mineralisation is spatially related to volcanic rocks (greenstones).

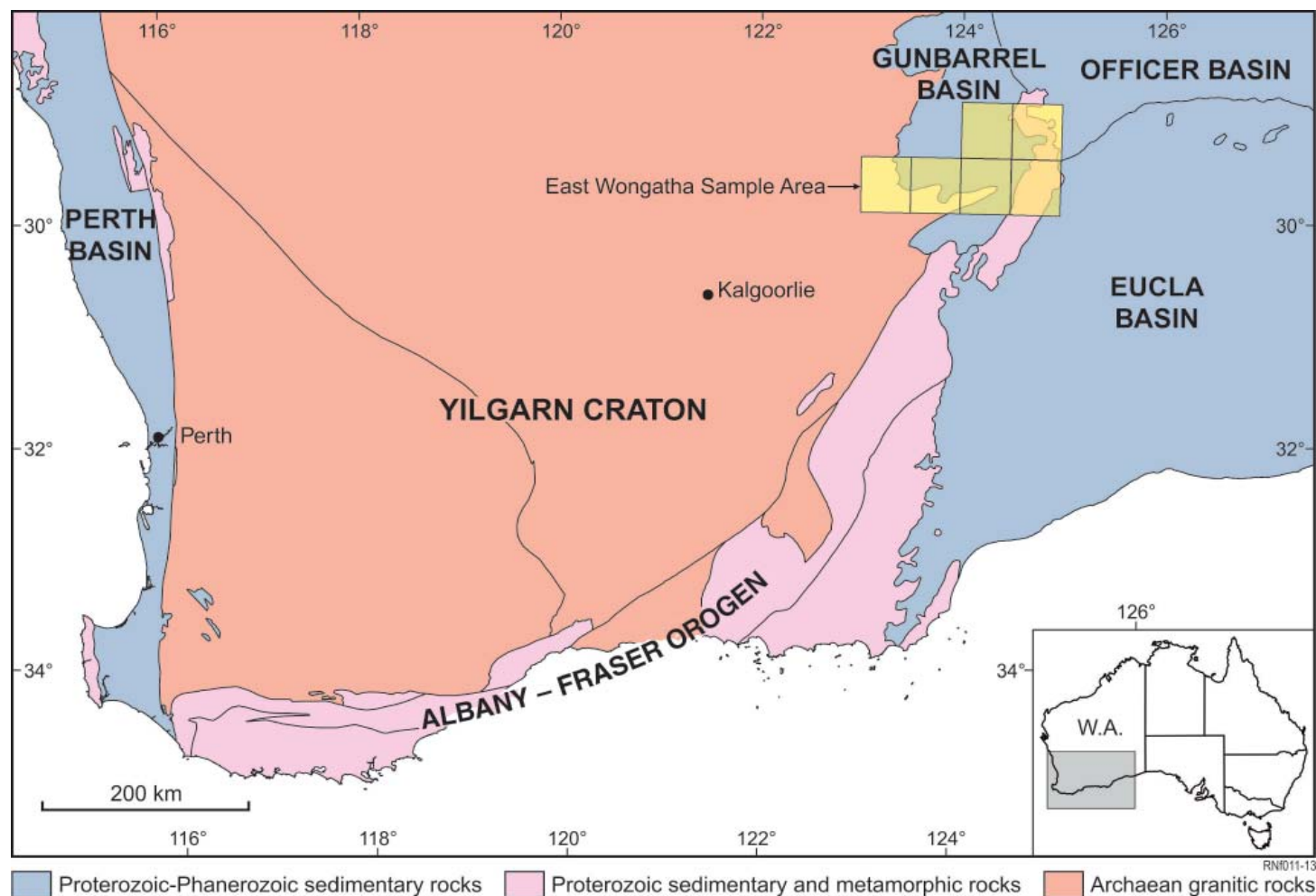


Figure 1. Samples used in this study are from the Gunbarrel Basin approximately 900 km ENE of Perth, Western Australia. East Wongatha area and interpreted 1:500,000 scale bedrock geology from Morris et al. (2013).

continued on page 3

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 1

Mechanisms of metal enrichment leading to anomaly formation through soil are important to understand. Gold in soils could have been transported by wind, or regionally (laterally and/or vertically) by mechanisms such as gas, vegetation, seismic pumping, hydromorphic or mechanical dispersion. These mechanisms have all been considered viable in some settings (Aspandiar et al. 2004; Anand et al. 2007; Anand et al. 2009; Cameron et al. 2004; Klusman 1993; Noble et al. 2013a, b). The smaller-fraction gold in regolith of the East Wongatha area is believed to have a component associated with localised migration.

Supergene gold deposits form a significant resource globally, but understanding the previously termed “invisible” gold in the micro and nanoparticulate size range has been problematic (Hough et al. 2008). Hough et al. (2008, 2011) and Noble et al. (2009) described the morphology of this “invisible” gold, and observed nanoparticulate populations that seem to decrease as the detection/visualisation technology improves (Fig. 2). The populations appear to have an almost fractal nature. Gold as a target element for both resource definition and mineral exploration is notoriously difficult to analyse due to its nuggety nature (Stanley

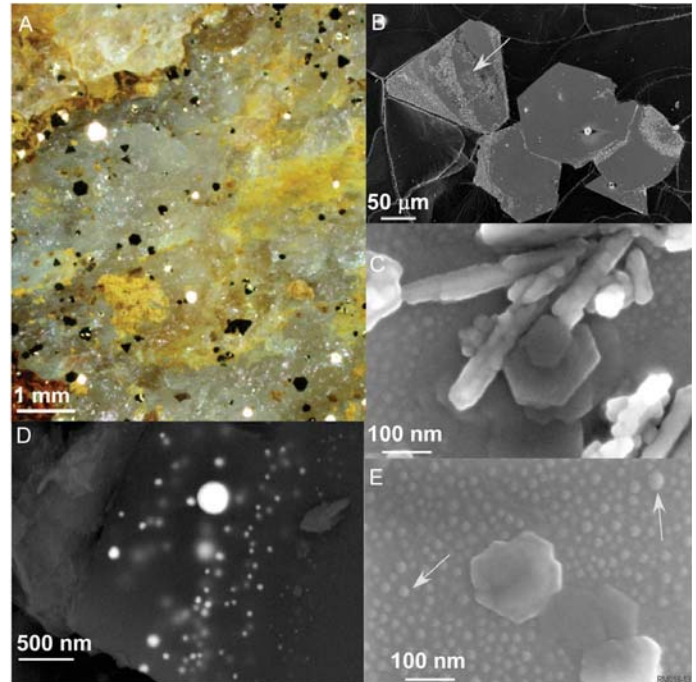


Figure 2. Multiple sizes and generations of gold particles on the surface of a weathered quartz fracture shows the supergene nanoparticulate gold population hosted on larger gold particles. A) light microscope image of black and shiny gold triangular and hexagonal crystals; B) FEGSEM image of gold microparticles (hexagons and triangles); C) a close-up of the darker band arrowed in B with nanoparticulate gold (hexagons) and halloysite (rods); D) gold nanospheres; E) 20 nm nanospheres arrowed in the background next to coarser gold nanoparticles are near the limits of present FEGSEM imaging capabilities (from Hough et al., 2011).

continued on page 4

Note: This EXPLORE article has been extracted from the original EXPLORE Newsletter. Therefore, page numbers may not be continuous and any advertisement has been masked.

Determination of micro and nanoparticulate fraction gold in regolith...*continued from page 3*

2006). To determine the nature of gold at the nanoparticle scale and, if it is nuggety, requires separation and quantification of gold in different size fractions.

Recently, Leybourne & Rice (2013) emphasised the importance of improving detection limits for gold, particularly for exploration through cover where signatures are subtle and poorly understood. Lower detection limits are vital for analysing the transported signature in soils, however, a large portion of each of these samples is made up of/composed of coarse, quartz-rich material with only a small number of target and pathfinder elements associated with this fraction (bound to cutans of goethite and hematite). The abundance of quartz dilutes the already low gold signature. To minimise this effect, a number of partial extractions may be used (Kelley et al. 2003; Noble 2012; Noble & Stanley 2009; Mann et al. 1998, 2005; Williams & Gun 2002). Partial extractions are more likely to release a significant proportion of the gold from the bulk sample especially if it is in an ionic form, or the digest (e.g., cyanide) specifically targets gold. If the gold is associated with the cutans on coarse grains, the partial extractions will work well. However, if the gold is associated with the smaller particles, an alternative is to concentrate the size fraction that contains the gold.

“Fine fraction” analysis of soil and regolith (i.e., silt particle size and smaller) has been used for geochemical exploration in several studies, with the <63 or $<50 \mu\text{m}$ size fractions used to enhance target and pathfinder element contents (Morris 2013; Noble 2012; Scott & van Riel 1999). Most of the loosely bound ions will be adsorbed to particles in the smaller size fractions because the clays, organic compounds and various oxides that dominate this fraction have a greater exchange capacity than the larger minerals such as quartz (Hawkes & Web 1962; Hall 1998).

These smaller fractions clearly influence the content of metals such as gold. Common questions arise as to which size fraction is optimal, and how fine is fine? We will not tackle the first question, but in this paper we will provide a method to quantify the gold content of the micro and nanoparticulate fractions in soil samples from the East Wongatha area of Western Australia and demonstrate the remarkably different gold contents associated with these fractions compared to the bulk regolith.

METHODS

Fourteen soil samples from a large regional regolith survey in the East Wongatha region of Western Australia (Morris 2013; Fig. 1) are discussed here. The $<50 \mu\text{m}$ fraction of these samples had been analysed for gold by the Geological Survey of Western Australia, following aqua regia digestion of 1 g of sample, producing gold contents from <1 ppb to 29 ppb (Morris 2013). All soil samples are quartz-rich, and from a region of largely allochthonous thick transported regolith. In most cases, the $<50 \mu\text{m}$ fraction is $<5\%$ of the bulk soil. Approximately 5 kg of bulk soil was collected at a depth of about 90 cm at each site using a power auger. A split of c. 1 kg from each sample was used in this study.

Five fractions were separated from each bulk sample (e.g., Figs. 3, 4 and 5) and the average weight % of each fraction was determined (Table 1). Bulk sample dry weight was determined and the material was then soaked in deionized water overnight. Following this, the soil suspension was placed in an ultrasonic bath for 20 minutes and then poured through nylon mesh sieves at 2 mm, $250 \mu\text{m}$ and $53 \mu\text{m}$. The suspension that passed through all sieves ($<53 \mu\text{m}$) was placed in a 1 L graduated cylinder and filled to 1L with

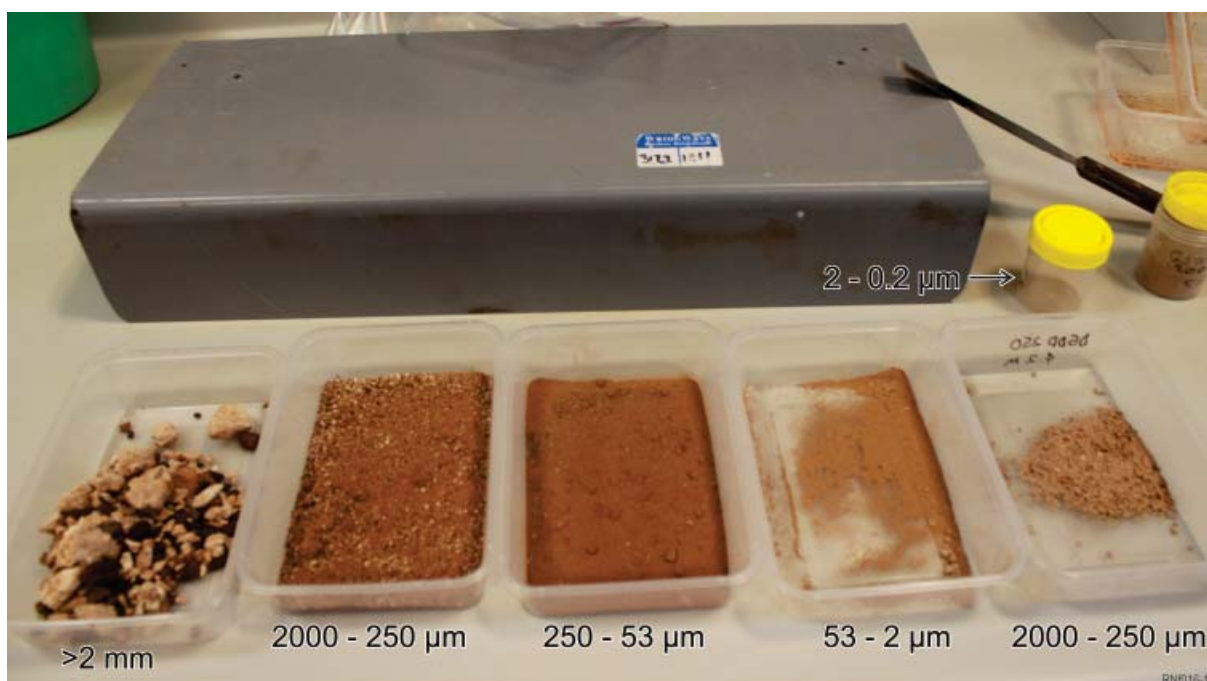


Figure 3. Examples of the 2 mm to the $<2 \mu\text{m}$ size fractions separated from the 1 kg bulk soil sample using wet sieving.

continued on page 6

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 4

.....



Figure 4. Using centrifugation to separate the <0.2 μm (or 200 nm) size fraction results in dry films (dark red material) of soil weighing between 0.1 and 0.5 g that were then subject to aqua regia digestion.

deionized water. This fraction was then mixed and left standing. After 5.5 hours (at 25° C) the top 8 cm was drawn off (~ 200 mL with only <2 μm sized particles in solution), and the remainder along with the other sieved fractions was dried in an oven at 80°C for approximately 1 week. The samples were then cooled to room temperature and weighed. Full details for particle size separation are available in Gee & Bauder (1986).

The <2 μm solution drawn off the top of the 1 L cylinder was placed in a centrifuge tube and spun at >3000 Relative Centrifugal Force (RCF) for 1 hr at 25°C. The particles remaining in solution were approximately <0.2 μm using a conservative adjustment of the methods by Gee & Bauder (1986) and Kilmer & Alexander (1949). This solution (<0.2 μm) and the separate fraction (<2 μm to 0.2 μm) at the bottom of the tube were then dried and weighed in the same manner as the larger fractions (Fig. 4). A flow chart of the procedure traces the sample separation steps (Fig. 5).

All fractions were subjected to an aqua regia digestion at CSIRO Laboratories, Kensington, Western Australia. For the <2 μm fraction and coarser soil samples, 0.8 ± 0.01 g of soil were subjected to a conventional aqua regia

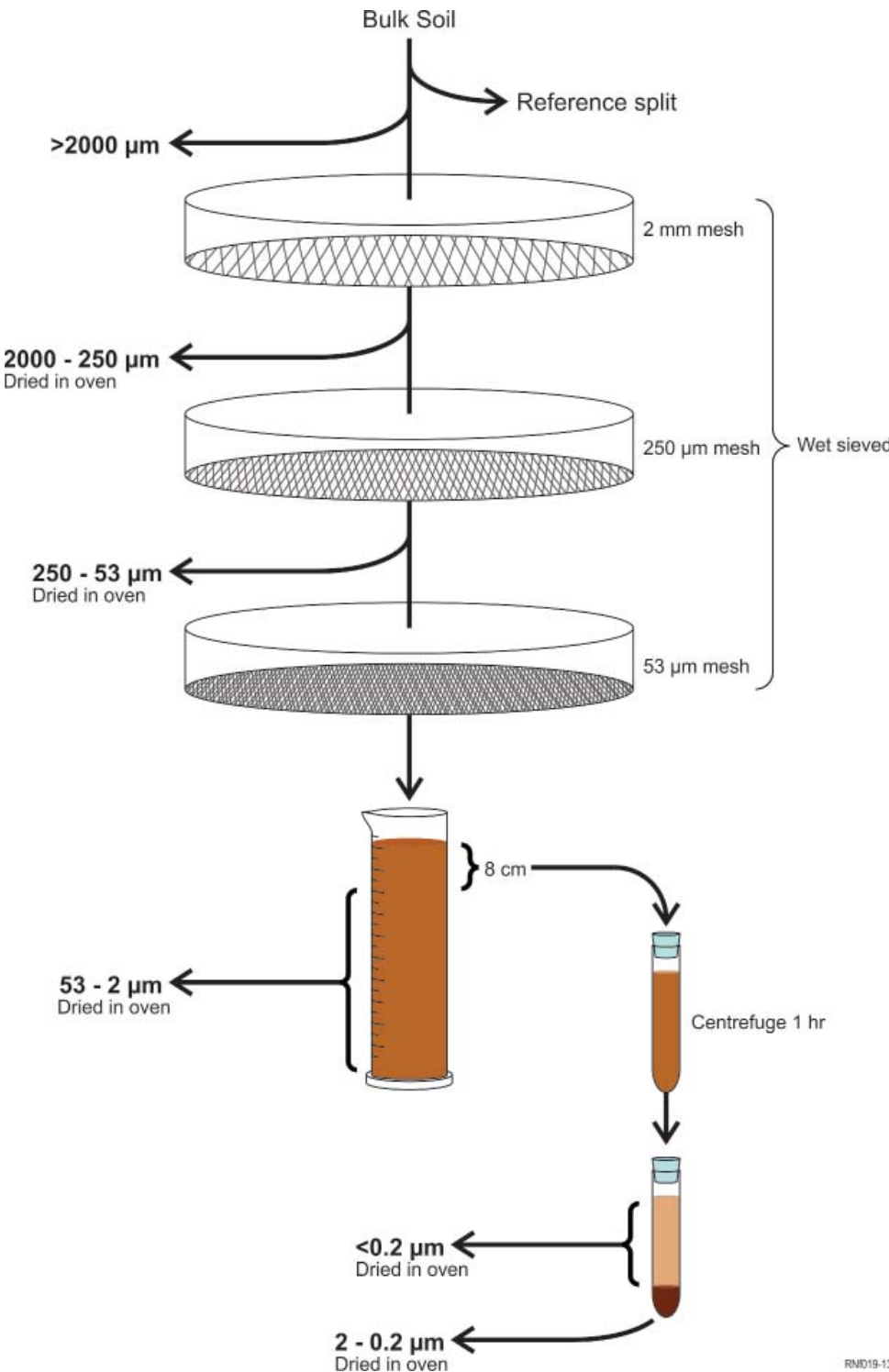


Figure 5. Flow chart of the method used to separate the particle size fractions used in this study.

Table 1. Mean data and standard deviation for the weight (g) of the 14 samples used for particle size separation in this study.

Fractions	Coarse			Medium	Microparticulate	Nano-particulate	
Size	>2 mm	2000-250 μm	250 -53 μm	53 -2 μm	2 – 0.2 μm	<0.2 μm	Lost
Mean Wt.(g)	175.2	694.8	200.9	84.4	16.4	0.18	13.1
Std Deviation	281.9	342.9	69.4	92.9	13.3	0.12	26.3
Mean Wt. %	14.80	58.69	16.97	7.13	1.38	0.02	1.11

continued on page 7

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 6

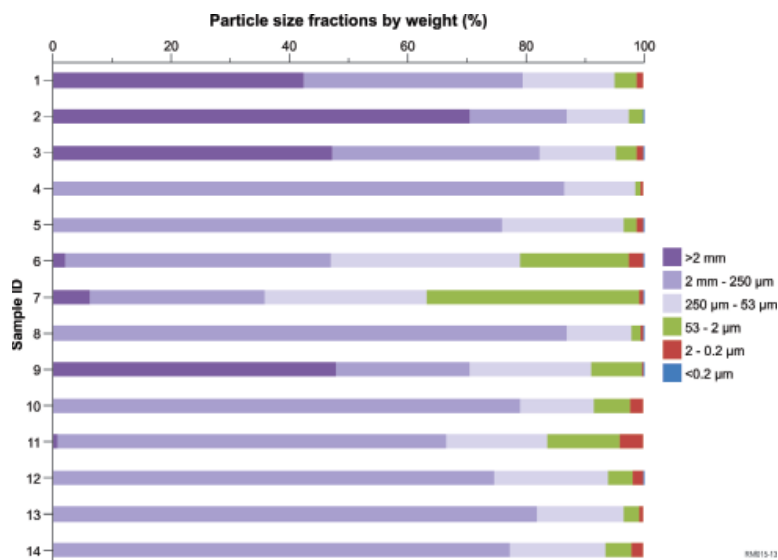


Figure 6. Size fractions by weight % for the samples used in this study.

(3:1 concentrated HCl:HNO₃, respectively) digestion (Chen & Ma 2001) to determine their readily soluble bulk chemical composition. For the nanoparticulate fraction (<0.2 µm), the remaining sample available was much smaller. As a result a smaller sample weight of 0.1g to 0.4 g was digested, with most samples c. 0.2 g.

Each sample was weighed into 250 ml glass conical flasks, 25 ml of aqua regia was added, watch glass lids were placed on top, and the flasks placed on hotplates at 110°C for 3 h. After evaporation to near dryness, the sample was diluted with 20 ml of 2% (v/v with H₂O) HNO₃ and the samples were transferred into a 100 ml volumetric flask after filtering through Whatman no. 42 paper and diluted to 100 ml with milliQ water. This extraction was used to determine the contents of elements in soil particles, excluding those substituted within the crystal lattice of highly resistant minerals. The solutions were directly analysed using an ICP-MS (Perkin Elmer Optima 7500A) by Bureauveritas Pty Ltd, Perth, Australia.

For all analyses, blanks and analytical duplicates were included. The half absolute relative difference (HARD) error was calculated as a percentage for all compounds using duplicates according to the methods described by Stanley & Lawie (2007):

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

RESULTS

Approximately 75% of the weight of most samples was comprised of particles >250 µm, with two exceptions that had approximately 45% of their weight in this fraction (samples #6 and 7; Fig. 6). Mineralogically, the microparticulate and nanoparticulate fractions consist of kaolinite, goethite and hematite along with quartz. Although the soils tend to be dominated by the coarse, quartz-rich soil fraction, the gold analysis on all the size fractions shows that the majority of the gold is small and hosted in the <53 µm fraction. The average total gold distribution within the size fractions for all samples used in this study is shown in Figure 7. The gold

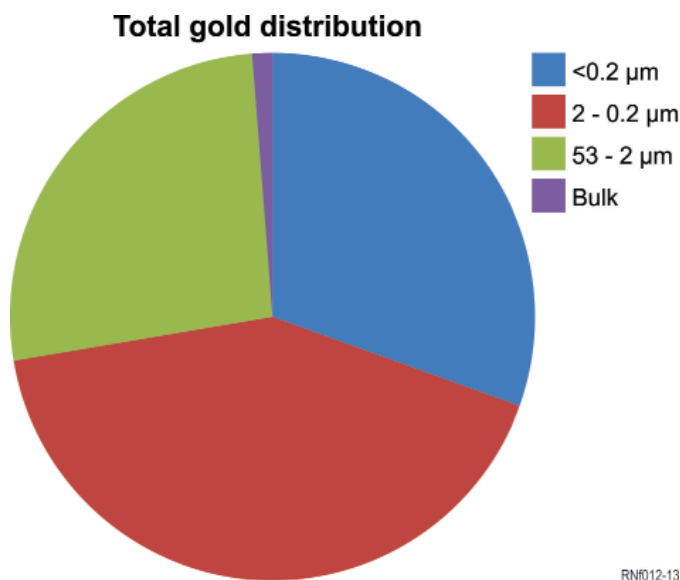


Figure 7. Average total gold distribution by size fraction. A large proportion of the gold is in the microparticulate (<2 µm) and nanoparticulate (<0.2 µm) fractions. The bulk category (purple) represents all the combined fractions and is dominated by particles > 53 µm.

associated with the bulk, coarse fraction dominated materials is less than 2% and, on average, almost insignificant (Fig. 7).

Gold is not consistently hosted within a specific size fraction, i.e., significant variability exists between the samples analysed (Fig. 8). Proportionally, the majority of gold is associated with the silt size fraction (2-53 µm) such as in samples #1 and 4 (Fig. 8), whereas other samples exhibit the majority of gold in the clay-sized microparticulate fraction (<2 µm), such as samples #3, 9 and 10. Three out of the fourteen samples have the majority of their gold hosted in the ultrafine, nanoparticulate (<0.2 µm) size fraction (Samples #2, 5 and 8; Fig. 8). One of those samples (#8) has over 95% of its gold in the nanoparticulate fraction. We have presented gold proportions, but actual contents of gold are commonly large (>100 ppb) in the two finest fractions (Fig. 9). Analysing the bulk soil and the medium fraction (<53 µm) for gold resulted in samples that were below detection (1 ppb) or in the low 10s of ppb range for gold. However, analysis of the micro (<2 µm) and nanoparticulate (<0.2 µm) soil fractions revealed contents commonly in the 100s of ppb range and in some cases, in the ppm concentrations of range for gold (Fig. 9). Selecting and analysing the nanoparticulate gold fractions via particle size separation results in gold concentrations that span three orders of magnitude. It should be noted that these results are determined by digesting a sample size that is smaller than normal (~0.2 g compared to a few grams in most aqua regia digests), so there are increased errors in accuracy, but these trends of increased gold in the smaller particle size fractions are expected to be real. Table 2 shows the percentage half absolute relative difference between duplicate analyses, with significant variation recorded. The duplicates could

continued on page 8

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 7

.....

not be prepared for the nanoparticulate fraction due to the limited sample mass recovered, but a potentially larger variability is expected in this fraction. The limited sample size has an increased propagation of error when calculating the mg/kg of gold in soil.

Spatially, the results plot differently and do not give a clear indication of which sample is the most prospective for mineralisation in the East Wongatha area as the different fractions do not show the same distribution of samples with the greatest gold contents. The sample with the greatest bulk gold content does not contain the greatest amount of gold in the micro and nanoparticulate fractions (Fig. 10). Normalised ratios of the gold content in each fraction to the weight of that specific fraction recovered only change the patterns slightly from the direct gold content plots of the micro and nanoparticulate fractions (Fig. 10) Orientation surveys are required to assess the exploration benefit of this technique. The area studied has U, REE and Ni prospects

and deposits adjacent to the samples, but known gold occurrences are commonly further away to the northeast and to the south (Fig. 10; Morris 2013). The micro and nanoparticulate fractions are remarkably different, with no single sample showing larger contents in both of these fractions compared to the rest of the samples assayed (Fig. 10). This indicates that gold tends to be nuggety regardless of the size fraction analysed.

DISCUSSION

Tens of ppb gold in soils may be considered anomalous when exploring in transported, covered areas of Western Australia. In this study, we have shown that the weight percent of the fine size fractions may significantly influence the gold content and perhaps smoothing the data based on particle size is warranted. Research has shown gold to be preferentially concentrated in the fine (<63 µm) size fraction of soils and using this size fraction improved explora-

Table 2. Percentage half absolute relative difference (HARD) results for four fractions with duplicates. Note these were full method duplicates subject to the full separation technique prior to chemical digestion and analysis.

Sample ID	Size fraction duplicated	% HARD
3	53 -2 µm	8.8
4	53 -2 µm	37.4
6	2 – 0.2 µm	14.3
11	2 – 0.2 µm	86.5

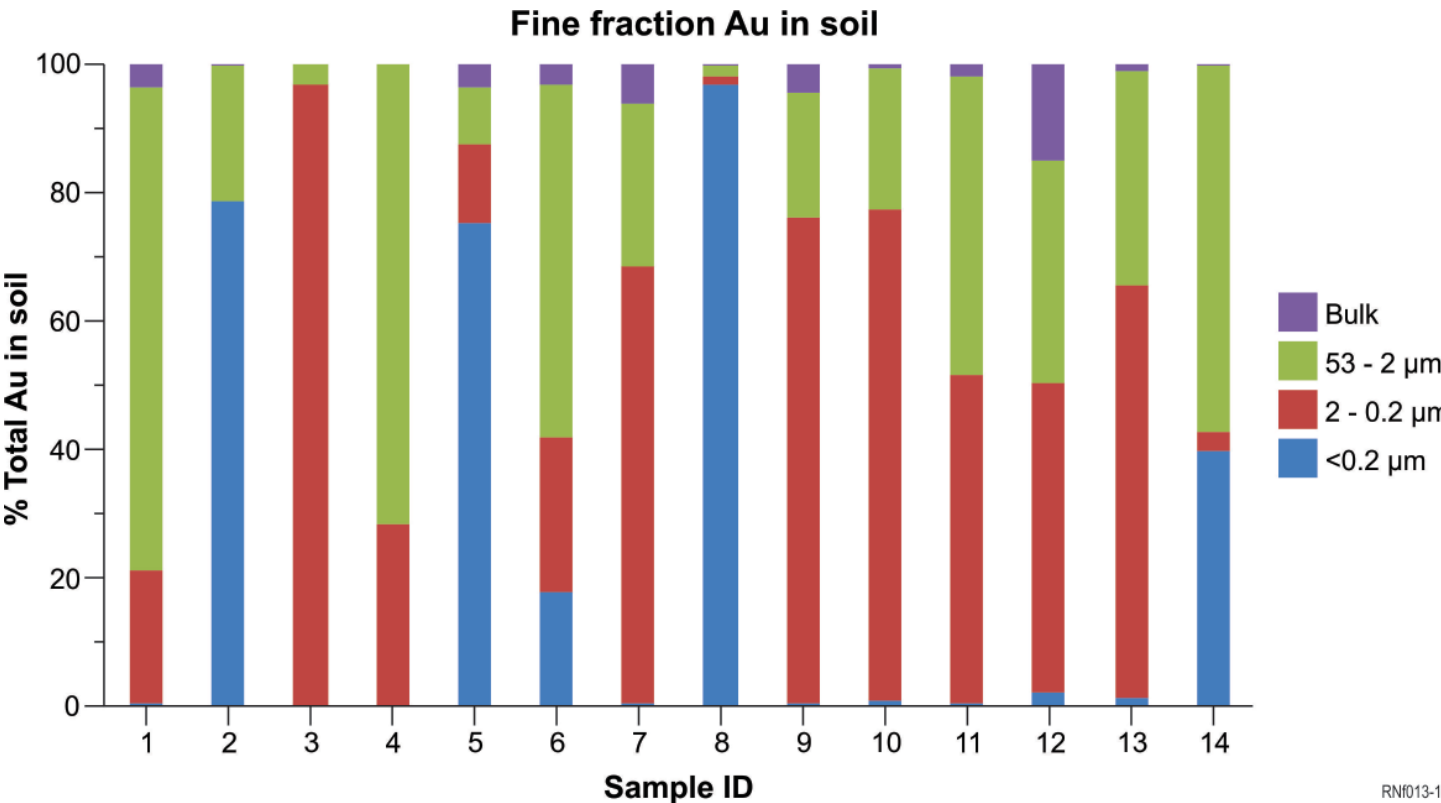


Figure 8. The percentage total gold in each size fraction for the individual samples. A large, but not uniform, proportion of the gold is in the fine (<2 µm) and ultrafine (0.002 µm) fractions. The bulk is all the combined fractions and dominated by particles > 53 µm.

continued on page 9

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 8

.....

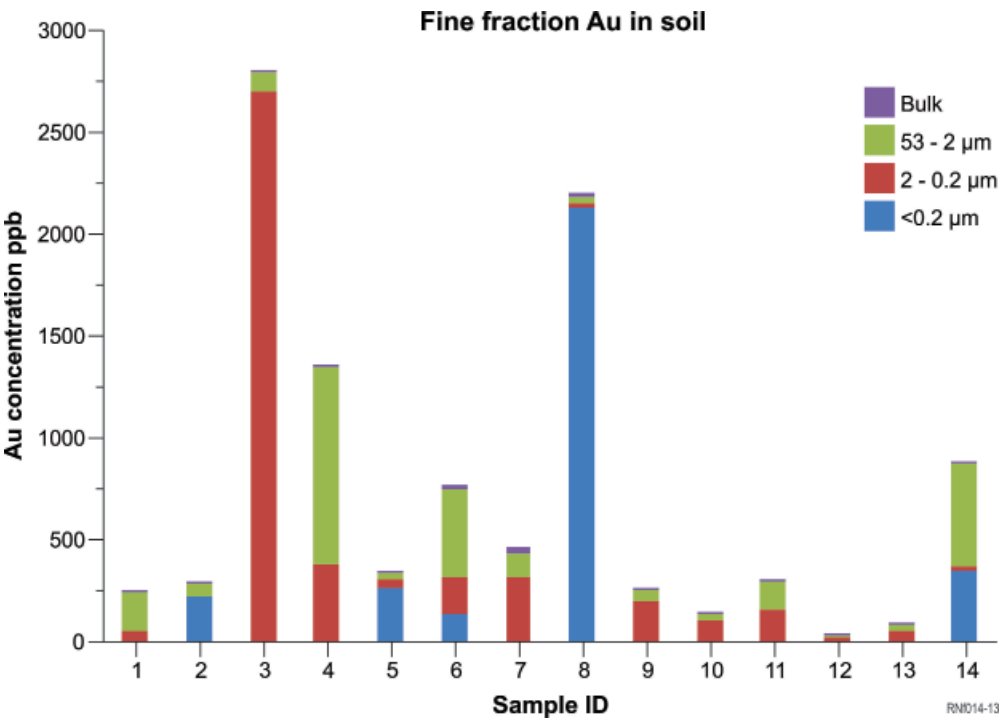


Figure 9. Gold contents in each sample separated by size fractions. The bulk category represents all the combined fractions and is dominated by particles > 53 µm.

tion through cover at the Goornong South gold deposit in northern Victoria, Australia (Scott & van Riel 1999). It is not known if exploration potential is improved by using the ultrafine fractions as samples over known mineralisation sources were not part of this study i.e., no orientation survey. An assessment of the gold content in the fractions should be normalised by the mass or calculated surface area of that fraction in the sample to truly test the exploration potential because just using the finer fractions tends to elevate the content of gold (and other pathfinder metals) in all samples including background samples contents. The normalisation in this study only slightly changed the results from the direct fraction contents. Without an orientation survey in areas with known gold deposits, the effectiveness of this normalisation cannot be assessed. For bulk gold results,

continued on page 10

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 9

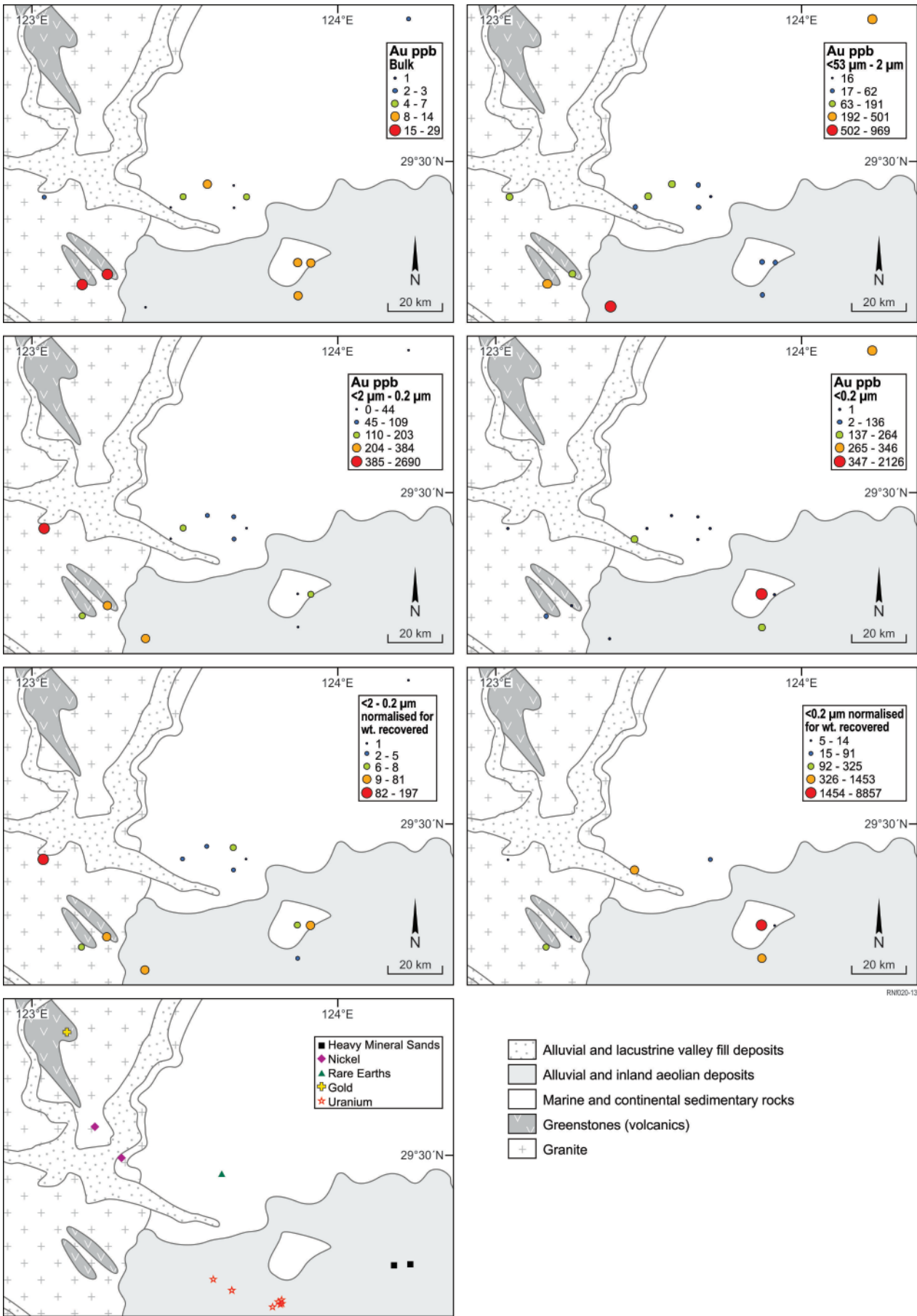


Figure 10. Spatial plots for the gold content by size fractions and the microparticulate and nanoparticulate fractions normalised to the weight of that fraction recovered. Targeting by a specific fraction may give significantly different targets. See Figure 3 of Morris (2013) for explanation of regional geology.

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 10

the greatest contents are located near mapped greenstones that are generally the host lithology of gold deposits in the Yilgarn Craton. However, the gold content of the micro and nanoparticulate fractions (and the normalised results) highlight other areas, mostly in basins with deeper cover that possibly represent other mineralisation targets. The elevated gold content in the micro and nanoparticulate fractions could be indicative of aeolian transport of fine fraction gold (false anomalies) or more extensive local mobility of the smaller fraction as suggested by Morris (2013).

The results of Lintern (2007) also support the work of Morris (2013) in that gold in sand dunes from South Australia was dominantly microparticulate and spatially associated with buried mineralisation. In this South Australian study, it was determined that vegetation cycled gold from deeper beneath the dunes via evapotranspiration, particularly in drier years when the plants were required to tap the groundwater supply as the shallow regolith pore water was unavailable (Lintern 2007). These and other studies show micro and nanoparticulate gold (Hough et al. 2011) as well as ionic gold (Lintern et al. 2009) are the common forms and that cycling between both types is viable. Noble et al. (2009) showed experimentally that both forms can result from secondary mobilisation.

In the samples used in this study, cycling between ionic and particulate gold would influence the gold contents associated with the various fractions. Ionic gold would be solubilised in the water during sieving and centrifuging and could increase the gold content of the nanoparticulate fraction, although the original ionic crystal may have been much larger *i.e.*, $>2\ \mu\text{m}$. For example, results by Noble et al. (2009) showed that AuCl crystallised along with the pure metal particles. The ionic gold salts were micron-sized, so this potential increase into the nanoparticulate fraction may be viable, although kinetic studies of the dissolution process would be required to confirm the assumption that this leads to greater nanoparticulate fraction gold. Support for the presence of ionic gold is provided by results of deionized water digestion of $< 50\ \mu\text{m}$ fraction regolith from the East Wongatha area, which showed a strong positive correlation with aqua regia gold (Morris 2013).

Both the potentially larger ionic gold and the nanoparticulate gold are likely to be highly mobile in the surficial weathering environment. The mechanisms of gold mobility could be driven by vegetation (Lintern 2007; Anand et al. 2007), groundwater (Grimes et al. 1995; Gray 2001) or carrier gases (Cao et al. 2009; Gao et al. 2011). Understanding these processes is not part of this study, but how gold mobility affects particle size of the gold should be pursued in future studies.

Excluding the aeolian transport mechanism, the size of gold particles in soils in Western Australia does not necessarily indicate distance from source. Experiments have shown that the size of gold precipitating from solutions in proxy experiments for supergene mobilisation and recrystallization varies, so the size of the gold fractions in these transported soils is not necessarily indicative of distance from ore deposits (Noble et al. 2009; Hough et al. 2011).

The methods presented in this paper show a large and variable amount of micro and nanoparticulate gold. The visualisation of these size populations and the apparent nanoparticulate population of gold $<0.20\ \mu\text{m}$ (Hough et al. 2008; Hough et al. 2011; Fig. 2) are corroborated in this research with significantly large gold contents found in the nanoparticulate fractions. The results of the study by Hough et al. (2011) suggest that with increasing technology developments improving the resolution of scanning electron microscopy, soil and regolith samples will also host a significant population of nanoparticulate gold at the $0.02\ \mu\text{m}$ size and smaller. With increased centrifugation of the technique reported in our methods, these even smaller fractions can be analysed separately, too.

The particles remaining in solution for the nanoparticulate fraction are approximately $<0.2\ \mu\text{m}$ using a conservative adjustment of the methods by Gee & Bauder (1986) and Kilmer & Alexander (1949). A number of assumptions are used to relate the particle size and density in the above methods and a direct calculation using Stoke's Law and the assumption of quartz mineral density of $2.65\ \text{g/cm}^3$ do not directly agree (J. Robertson *pers. comm.*, unpublished data). Future research will image the dried suspension using FEGSEM and determine the maximum particle size in each step, removing the need for the previously applied theoretical assumptions.

CONCLUSIONS

This study assessed soil size fractions from bulk soil to the nanoparticulate ($<0.2\ \mu\text{m}$) fraction and the associated gold contents. It has demonstrated the method to quantify gold in these very small size fractions and shown there are differences in the micro and nanoparticulate fractions compared to the bulk or medium fractions that are routinely analysed in mineral exploration. The study is limited by the fact that the samples are not oriented with respect to known gold mineralisation and by the small number of samples and the small weight of nanoparticulate sample size material recovered. The small volumes of material analysed result in increased errors, but has effectively demonstrated that the size fractions can be separated, analysed and provide different information. The results are plotted spatially as an example for exploration feasibility, but do not assess which fraction provides the best exploration sample medium. Future research requires a more rigorous assessment of the method to exactly quantify the nanoparticulate size fraction separated, along with more focused orientation surveys to determine if significant benefit exists for analysing the micro and nanoparticulate fractions of soils for gold exploration; a process that is not currently conducted by industry.

Acknowledgements

Michael Verrall (CSIRO) assisted in SEM work. Funding for this work was provided by CSIRO Minerals Down Under Internship program. Ravi Anand, Matt Leybourne and Beth McClenaghan are thanked for reviewing and improving this manuscript.

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 11

References

- Anand, R.R., Cornelius, M. & Phang, C. 2007. Use of vegetation and soil in mineral exploration in areas of transported overburden, Yilgarn Craton, Western Australia. *Geochemistry, Exploration, Environment and Analysis*, **7**, 267-288.
- Anand, R.R., Lintern, M.J., Townley, B. Noble, R.R.P., Wakelin, S., Macfarlane, C., Hough, R., Smith, P., Aspandiar, M., Carr, G., Korsch, M., Reith, F., Phang, .Reid, N., Gray, D.J., S. Soongpankhao, C. Soto, F. López, R. Luca, Hill, S., Pinchand, T., Gregg, A. & Reich, M. 2009. AMIRA P778 Final Report: Predictive geochemistry in areas of Transported overburden. Final Report. CSIRO Report P2009/1788.
- Aspandiar, M.F., Anand, R.R., Gray, D. & Cucuzza, J. 2004. Mechanisms of metal transfer through transported overburden within the Australian regolith. *EXPLORE*, **125**, 9-12.
- Cameron, E.M., Hamilton, S.W., Leybourne, M.I., Hall, G.E.M. & McClenaghan, M.B. 2004. Finding deeply buried ore deposits using geochemistry. *Geochemistry: Exploration, Environment, Analysis*, **4**, 7-32.
- Cao, J., Hu, R., Liang, Z. & Peng, Z. 2009. TEM observations of geogas-carried particles from the Chankeng concealed gold deposit, Guangdong Province, South China. *Journal of Geochemical Exploration*, **101**, 247-253.
- Chen, M. & Ma, L.Q. 2001. Comparison of three aqua regia digestion methods for twenty Florida soils. *Soil Science Society of America Journal*, **65**, 499-510.
- Gao, Y., Wang, M. & Zhang, D. 2011 Application of 'metals-in-soil-gas' techniques to mineral exploration in exotic overburden. *Geochemistry: Exploration, Environment, Analysis*, **11**, 63-70.
- Gee, G.W. & Bauder, J.W. 1986. Particle size analysis. In: *Methods of Soil Analysis*. Klute, A (Ed). American Society of Agronomy, Madison, Wisconsin, U.S.A.
- Gray, D.J. 2001. Hydrogeochemistry in the Yilgarn Craton. *Geochemistry: Exploration, Environment, Analysis*, **1**: 253-264.
- Grimes, D.J., Ficklin, W.H., Meier, A.L. & McHugh, J.B. 1995. Anomalous gold, antimony, arsenic, and tungsten in ground water and alluvium around disseminated gold deposits along the Getchell Trend, Humboldt County, Nevada. *Journal of Geochemical Exploration*, **52**, 351-371.
- Hall, G.E.M. 1998. Analytical perspective on trace element species of interest in exploration. *Journal of Geochemical Exploration*, **61**, 1-19.
- Hawkes, H.E. & Webb, J.S. 1962. *Geochemistry in Mineral Exploration*. Harper and Row, New York.
- Hough, R.M., Reich, M. & Noble R.R.P. 2011. Noble metal nanoparticles in ore systems. *Ore Geology Reviews*, **42**, 55-61.
- Hough, R. M., Noble, R.R.P., Hitchen, G.J., Hart, R., Reddy, S.M., Saunders, M., Clode, P., Vaughan, D., Lowe, J. Gray, D.J., Anand, R.R., Butt, C.R.M. & Verrall, M. 2008. Naturally occurring transparent gold nanoplates and particles. *Geology*, **36**, 571-574.
- Kelley, D.L., Hall, G.E.M., Clos, L.G., Hamilton, I.C. & McEwen, R.M. 2003. The use of partial extraction geochemistry for copper exploration in northern Chile. *Geochemistry: Exploration, Environment, Analysis*, **3**, 85-104.
- Kilmer, V.J. & Alexander, L.T. 1949. Methods of making mechanical analyses of soils. *Soil Science*, **68**, 15-24.
- Klusman, 1993. *Soil Gas and related Methods for Natural Resource Exploration*. Wiley, New York.
- Leybourne, M.I. & Rice, S. 2013. Determination of gold in soils and sediments by fire assay or aqua regia digestion: Choosing the optimal method. *EXPLORE*, **158**, 1-10.
- Lintern, M.J. 2007. Vegetation controls on the formation of gold anomalies in calcrete and other materials at the Barns Gold Prospect, Eyre Peninsula, South Australia. *Geochemistry: Exploration, Environment, Analysis*, **7**, 249-266.

continued on page 13

Determination of micro and nanoparticulate fraction gold in regolith...continued from page 12

Lintern, M.J., Hough, R.M., Ryan, C.G., Watling, J. & Verrall, M. 2009. Ionic gold in calcrete revealed by LA-ICPMS, SXRF and XANES. *Geochimica et Cosmochimica Acta*, **23**, 1666-1683.

Mann, A.W., Birrell, R.D., Mann, A.T., Humphreys, D.B. & Perdrix, J.L. 1998. Application of the mobile metal ion technique to routine geochemical exploration. *Journal of Geochemical Exploration*, **61**, 87-102.

Mann, A.W., Birrell, R.D., Fedikow, M.A. & de Souza, H.A.F. 2005. Vertical ionic migration: mechanisms, soil anomalies, and sampling depth for mineral exploration. *Geochemistry: Exploration, Environment, Analysis*, **5**, 201-210.

Morris, P.A. 2013. Fine fraction regolith chemistry from the East Wongatha area, western Australia: tracing bedrock and mineralization through thick cover. *Geochemistry: Exploration, Environment, Analysis*, **13**, 21-40.

Noble, R.R.P. 2012. Transported cover in northwestern Victoria, Australia - An impediment to geochemical exploration for gold. *Journal of Geochemical Exploration*, **112**, 139-151.

Noble, R.R.P. & Stanley, C.R. 2009. Traditional and novel geochemical extractions applied to a Cu-Zn soil anomaly: a quantitative comparison of exploration accuracy and precision. *Geochemistry: Exploration, Environment, Analysis*, **9**, 159-172.

Noble, R.R.P., Hough, R.M. & Grenik, E.M. 2009. Natural and experimental clues to understanding the transport and deposition of supergene gold in Western Australia. *EXPLORE*, **142**: 1-8.

Noble, R.R.P., Lintern, M.J., Gray, D.G., Reid, N. & Anand, R.R. 2013a. Metal migration at the North Miitel Ni sulphide deposit in the southern Yilgarn Craton: Part 1 regolith and groundwater. *Geochemistry: Exploration, Environment, Analysis*, DOI: 10.1144/geochem2012-132.

Noble, R.R.P., Lintern, M.J., Townley, B., Anand, R.R., Gray, D.G. & Reid, N. 2013b. Metal migration at the North Miitel Ni sulphide deposit in the southern Yilgarn Craton: Part 3 gas and overview. *Geochemistry: Exploration, Environment, Analysis*, DOI: 10.1144/geochem2012-131.

Scott, K.M. & van Riel, B. 1999. The Goornong South gold deposit and its implications for exploration beneath cover in Central Victoria, Australia. *Journal of Geochemical Exploration*, **67**, 83-96.

Spaggiari, C. V., Kirkland, C. L., Pawley, M. J., Smithies, R. H., Wingate, M. T. D., Doyle, M. G., Blenkinsop, T. G., Clark, C., Oorschot, C. W., Fox, L. J. & Savage, J. 2011. The geology of the east Albany-Fraser Orogen – a field guide. Geological Survey of Western Australia Record, 2011/23.

Stanley, C.R. 2006. On the special application of Thompson-Howarth error analysis to geochemical variables exhibiting a nugget effect. *Geochemistry: Exploration, Environment, Analysis*, **6**, 357-368.

Stanley C.R. & Lawie, D. 2007. Average relative error in geochemical determinations: classification, calculation, and a plea for consistency. *Exploration and Mining Geology*, **16**, 267-275.

Williams, T.M. & Gunn, A.G. 2002. Application of enzyme leach soil analysis for epithermal gold exploration in the Andes of Ecuador. *Applied Geochemistry*, **17**, 367-385.

Ryan R.P. Noble¹, Marco Cavaliere^{1,2}, Paul A. Morris³, G. Tenten Pinchand¹ and Robert M. Hough¹

¹CSIRO Earth Science and Resource Engineering
PO BOX 1130
Bentley, Australia 6102
Email: ryan.noble@csiro.au

²University of Naples- Dipartimento Scienze della Terra
Via Mezzocannone 8
80134 Napoli, Italy

³Geological Survey of Western Australia
100 Plain Street, East Perth, Australia 6004

