

## Testing a rapid sampling and analysis workflow in the remote Nullarbor Plain, Australia

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### INTRODUCTION

Winterburn *et al.* (2019) categorised the past decadal advances in exploration geochemistry in four areas; 1) understanding metal mobility and mechanisms, 2) rapid geochemical analyses, 3) improved data access, integration and interoperability, and 4) innovation in laboratory-based methods. Items 2 and 3 can be addressed with new technology and have the potential to improve efficiency and decision making in the field for large economic benefits and efficiencies. However, it can be hard to disrupt traditional work routines to realise these benefits. This paper specifically addresses items 2 and 3, with the objective to demonstrate some approaches used to rapidly sample, analyse and integrate data to geochemically map a region in remote South Australia. In a recent surface geochemical characterisation study, the Geological Survey of South Australia (GSSA) required a protocol for quick and efficient sampling, and with minimal environmental impact. The opportunity within this study was that there was no set protocol to adhere to, leaving the GSSA and CSIRO to test or adapt new rapid sampling technologies. The key criterion was based on the following question: can we geochemically characterise a region of 4000 km<sup>2</sup>, by sampling and analysing 300 sites in 7 days using a single field team?

Physical sample collection time is difficult to reduce significantly, with motorised augers/drills/clippers and manual shovels used as needed in current practice. However, sample/data handling and field analysis techniques can be changed and improved. Recording data electronically in the field provides several advantages over pen and paper. Consistency between sampling teams is maintained, transcription errors are negated and the time taken at each individual sample location is significantly reduced. The less glorified technology changes such as data handling, sample tracking and similar management tasks are often overlooked.

Field digital notebooks are commonly known and used devices for field data capture and a number of platforms and examples exist with geological surveys using them for mapping. Adaption of the platforms for mineral exploration sampling is achieved through modified Electronic Laboratory Notebooks (ELNs – tablet-style computers). Scientists willingly make use of generic notebooking software to aid their work, yet most ELNs lack the required functionality to meet the needs of specific fields (Kanza *et al.* 2017). In mineral exploration, commercial offerings are usually linked to specific analytical inputs (e.g., IMDEXHUB-IQ™ or Vanta™ XRF software). Often, these field data are later transferred and adapted to a specified company database. Tracking of samples past this point is tied to the individual company. One option for future-proofing sample provenance is the use of International Geo Sample Numbers (IGSN) that has become standard use for CSIRO sampling. IGSN are persistent (digitally long-lived), globally unique identifiers of samples. Unique identification of samples also assists the tracking of samples through various institutions, repositories and laboratories (Devaraju *et al.* 2016). IGSN can link to other samples (e.g. sub-samples), sampling features which are identified by an IGSN (e.g. drill holes, outcrops), connect the sample to data identified by Digital Object Identifiers (DOI), and link to descriptions and interpretations of the sample and data in the literature. This way of identifying and connecting research resources will help to maintain traceability and reproducibility of research into the future (Stall *et al.* 2019).

While protocols for field sample identification and data tracking have not been as rapidly taken up by industry, industry has certainly adapted quickly to rapid geochemical analyses, principally through the application of field portable X-ray fluorescence instrumentation (fpXRF; Arne *et al.* 2014; Sterk *et al.* 2018). Workflows and QAQC protocols are available for fpXRF (Fisher *et al.* 2014; Hall *et al.* 2012; 2014; Lemièrè 2018) but not always applied. One critical issue is the quality of sample preparation, or the portability of sample preparation devices required to prepare samples of appropriate quality for fpXRF (and other instrument) analysis.

With a mandate to rapidly sample and characterise a remote region of Australia, we developed a sampling and analysis workflow to maximise efficiency, and appropriate field preparation and analytical processes to generate quality results as the campaign evolved. This paper demonstrates the components of both field sampling and analysis conducted on the Nullarbor Plain of South Australia in November 2017.

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### SETTING

The study area is the Coompana region of the Nullarbor Plain, located in the southern central part of Australia, and has minimal topographic variation (Fig. 1).

The Nullarbor Plain stretches over the states of South Australia and Western Australia and is the surface expression of the Miocene (~23-5 Ma) Nullarbor Limestone. It is one of the largest limestone outcrops on Earth (>300,000 km<sup>2</sup>; O'Connell et al., 2012) and currently experiences semi-arid climatic conditions (annual precipitation ~150-400 mm and evaporation of ~2,000-3,000 mm; Bureau of Meteorology 2013). The study area shows a northwest-southeast elevation trend increasing to the north-west by ~100 m over the ~80 x ~50 km study area. The landscape surface above these sedimentary formations comprises a thin

cover of shallow, red-brown, calcareous, loamy to sandy soil with occasionally outcropping limestone covered by a variably thick (a few metres), hard calcrete layer (Fig. 2). Vegetation here in the Coompana area is dominated by low shrubland of chenopods, mostly pearl bluebush (*Maireana sedifolia*) and bladder saltbush (*Atriplex vesicaria*) and minor samphire (*Sarcocornia* sp.; Dunn & Waldron 2014; Fig. 2).

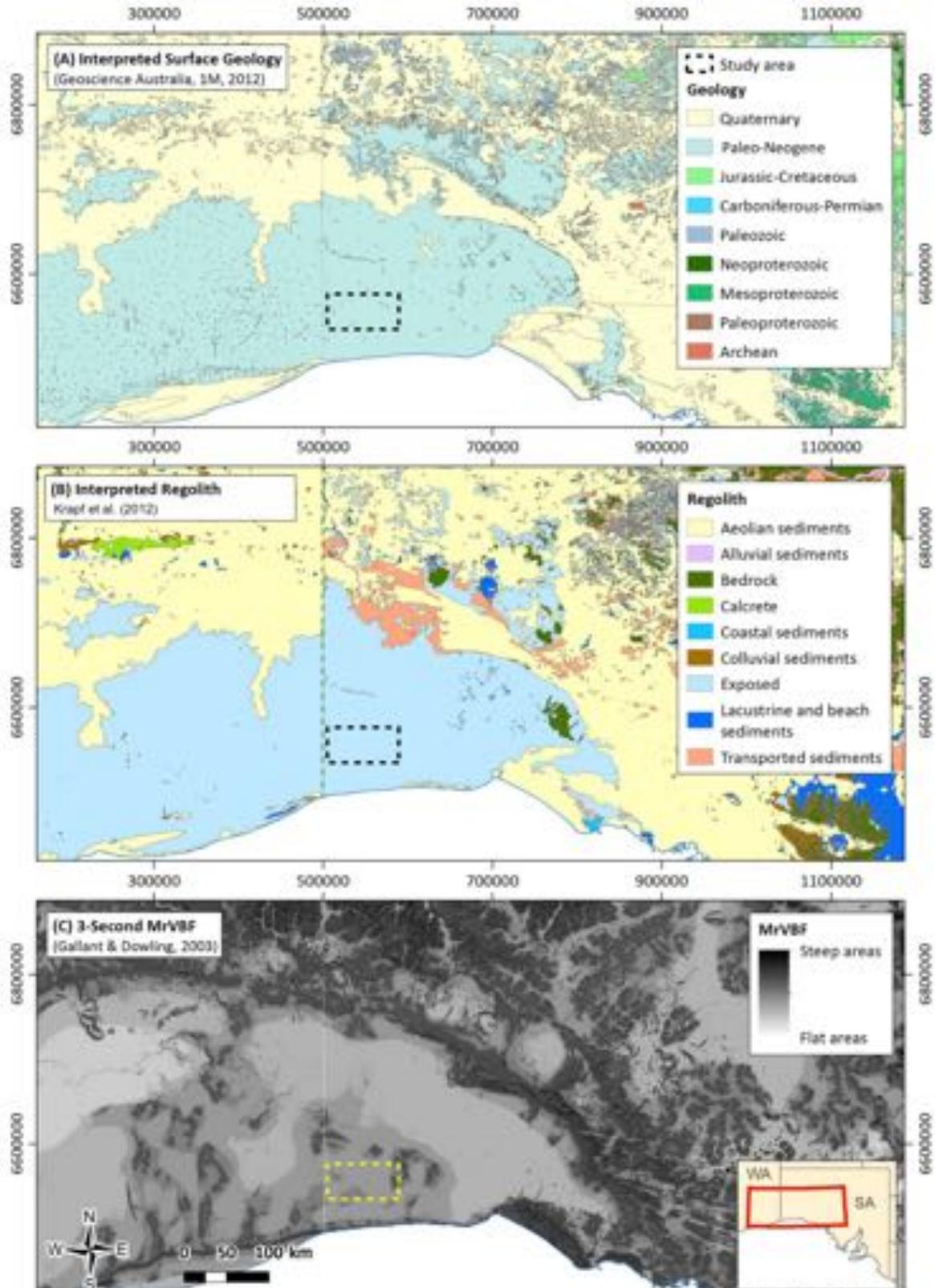


Figure 1. Reference regional maps for the Coompana region in Australia. (A) Interpreted age of surface geology units; (B) interpreted regolith map of the region; and (C) gradient slope map (Multiresolution Index of Valley Bottom Flatness, MrVBF) and inset showing the location of the region in Australia. Coordinates UTM GDA 94, Zone 52.

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The region was selected for the survey because it was a priority region of the South Australian Government as a new frontier for exploration, with little existing geological knowledge. Recognition of the area as a potential subduction driven, migrating magmatic arc with an age profile similar to some other well-endowed regions in South Australia and Western Australia (Dutch *et al.* 2018a,b) has generated interest for the exploration of magmatic nickel-copper sulphide deposits in particular (Lawley 2017).

### METHODS

#### Rapid Field Sample Collection

Sample sites were selected based on being representative of the area (Fig. 2a, c) and commonly away from outcropping rocks or extensive clay pans (Fig. 2b) that represent shallow low-lying areas and possibly have a greater contribution of alluvial materials. Teams of three people collected samples on a grid at 4 km intervals (~280 sites) over a region of 4000 km<sup>2</sup> using a helicopter. This spacing was chosen to highlight regional geochemical variations and not specifically

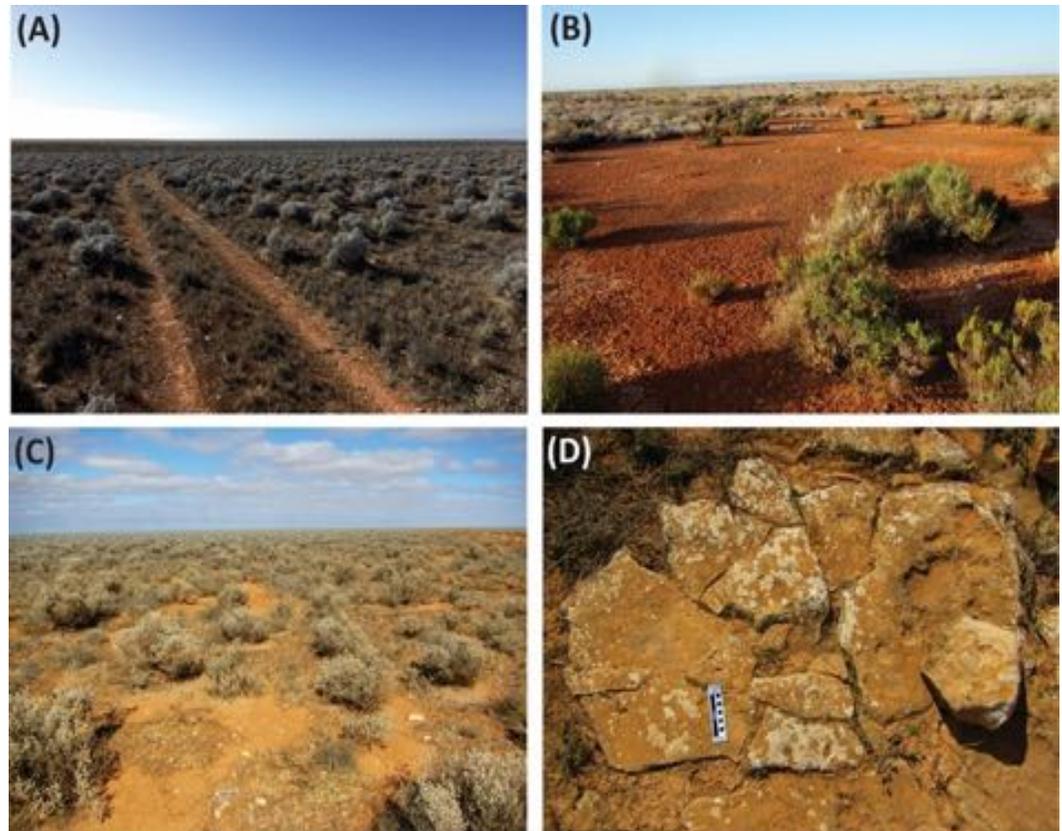
target mineral deposits or large systems. Field sampling times were 5-6 minutes per site to collect five sample media. With travel and refueling time factored in, this equated to 10 minutes per sample site over the 7 days of sampling. Five sample media were collected at each site:

- shallow soil sample (200-400g) from approximately 2-10 cm depth
- surface crust (<3 mm) approximately 20-30 g that is bound by cryptogams (moss/algae/lichens)
- carbonate/limestone lithic lag sample (100-300 g)
- bladder salt bush (*Atriplex vesicaria*) vegetation ~100 g of each (stems and leaves)
- pearl blue bush (*Maireana sedifolia*) vegetation ~100 g of each (stems and leaves)

Nearly all sites were able to be sampled for all five sample types, with the exception of approximately 10 sites that did not have blue bush present in the immediate area from the soils sample location (<50 m radius). Field duplicates were collected every 30 samples sites.

#### Rapid Field Data Collection

For each sample site, the Field Acquired Information Management System Project (FAIMS) application using the camera on a portable device such as a tablet or phone is used to rapidly scan the sample IDs, generate required information fields, take GPS locations, take photos and link the photos to the specific samples. One person is usually using the Android tablet (we used Samsung TAB A 10.5) and collecting site information including landscape setting and photographs and digging the hole for the dry soil sample (in this setting the hole was 25 cm in diameter and typically



*Figure 2. Examples of regolith landform types in the Coompana area: (A) Very gently undulating limestone plain on which a variably thick (less than a few meters) calccrete has developed, overlain by shallow calcareous loamy soil, sometimes with a sparse mantle of limestone and calccrete fragments. Dense vegetation of low shrubland of pearl bluebush and bladder saltbush cover most of the area. (B) Erosional residual plain with clay pans randomly distributed throughout. Vegetation is very scattered and dominated by samphire and minor grassland. (C) Erosional residual plain with NW-SE aligned vegetation. Very gently undulating limestone plain with abundant sub-/outcrop of Nullarbor Limestone and calccrete and/or mantle of mixed-sized rock fragments. NW-SE aligned vegetation of low shrubland and/or grassland. (D) Typical outcrop of Nullarbor Limestone within erosional residual plain.*

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10-20 cm deep with no horizonation excluding the organic crust). The other person was responsible for collecting the mineral materials and bagging them. The third person conducted the vegetation sampling. The results presented in this article only relate to the soil and lag samples that were processed and analysed in the field. Full details of all sampling and analysis procedures and equipment are available in Noble et al. (2018).

Samples were documented and labeled in the field using the FAIMS application, an open source platform (Ballsun-Stanton et al. 2018). The application was developed with Macquarie University and is available through the Google Play Store and [www.fedarch.org](http://www.fedarch.org). The system was adapted to scan and print bar code labels, auto fill tables and display the appropriate drop-down menus (Fig. 3 and Fig. 4). The geochemical sampling customisation features an interactive GIS component overlying maps (as GeoTIFF) that can be pre-loaded to assist in sample location, although this was not needed as we preloaded sample points into the helicopter navigation system. QA-QC samples were incorporated into the existing automated data listing that is generated in the field as a separate sample ID with a very similar location (a few metres apart).

Figure 3. Soil sample site information with dropdown options and site specific parameters. Photos taken are also linked specifically to the site.

To assist the sample documentation and tracking, the FAIMS application was updated to include a barcode reader. Further modification included a field printer and using data matrix labels for scanning (Fig. 4). Sample labels were pre-printed with sample identifiers that were also encoded as a data matrix barcode (Fig. 4). By scanning the pre-printed label, the field team did not have manual data entry and saved a lot of time with in sample documentation. For increased efficiency, the sample labels were printed the evening before and placed on the sample bags. These were then put in numerical order so that a “site batch” of bags could be grabbed together easily. Field duplicate samples used two batches per site and required an additional 3 minutes in sample collection. The FAIMS application works with bags in different order (i.e., you can sample the deeper soil before the surface crust), but the information fields are locked by the sample type (e.g., you can’t enter a sample depth into the surface crust sample information). Scanning the first sample (crust and plant) bags prior to landing at the site decreased the time needed by 1 minute per site. For this task, two tablets were used at each site. By switching screens off between sites, the power would last all day, although a backup and emergency charger were also carried.

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All samples had field sample labels in the IGSN format; these were registered later once connected to the Internet from the office. A field portable server operating with Wi-Fi connectivity ensured all data were effectively captured, backed up and transferred in a useable format to the standard laptop PCs running the portable laboratory and generating data products like maps.

The equipment used in this study performed well in the dry, dusty, arid conditions. Extreme high and low temperatures may cause some problems. The GPS in the devices does not perform well in temperatures  $>50^{\circ}\text{C}$ , such as the dashboard of a vehicle, so we keep devices shaded when not in use. The rugged cases are effective for high moisture conditions such as tropical environments. We are yet to test these in extreme cold conditions and expect they would be well suited to most geochemical sampling conditions.



Figure 4. Soil sampling showing the field printed labels and data matrix bar-code for scanning the sample identifier into the mobile application.

### *Rapid Field-Portable Laboratory Preparation and Analysis*

Analytical preparation and processing time of four minutes per sample was achieved with a three-person team (soil and lithic lag samples only). Sample preparation is identical for both the soil and the lithic lag samples. A field portable sample preparation laboratory (crush, mill and pellet press) was set up and coupled with fpXRF and Analytical Spectral Device (ASD) analysis (Fig. 5). All soil samples collected were prepared and analysed within 24 hours. Only

a few samples required drying (for a few hours) which was managed using a commercial pie oven. Basic requirements for the laboratory were protection from weather, availability of power and working bench space/tables. Samples were not sieved as the soils were not gravelly and were similar in their particle size distribution. Samples were split equally using a riffle splitter with half retained as a bulk reference sample (~150 g) and archived. The split was crushed using a mobile crusher (Reflex™ Crusher model) and milled to  $<100\ \mu\text{m}$  using a mobile disk mill (Reflex™ Mill model) with disk gap set to the approximate width using provided sample spacing tools (metal strips). Approximately 25 g of the milled soil was pressed into a pellet using a press (Reflex™ Press model) to provide a smooth, uniform surface for analysis with the fpXRF and ASD. Silica blanks were analysed approximately every 40 samples. Analytical duplicate sample analyses

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Figure 5. The portable laboratory at Coompana. From left to right, riffle splitter, crusher and mill on one table, pellet press on the old desk, the fpXRF and ASD (inside cardboard box, far right) on the other table.

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were made approximately every 10 samples to assess instrument precision and detection limits. Data calibration of fpXRF measurements against known standard compositions was performed using custom CSIRO software that uses additive log-ratio transformation of data to account for compositional system closure, and a Bayesian linear regression algorithm to robustly estimate and propagate uncertainties (Fig. 6). A stand-mounted Olympus Vanta M-series fpXRF (50 kV X-ray tube and fitted with a large-area silicon drift detector) operated for a total of 60 seconds (30 seconds for each of two beams of different energy levels). All samples and standards were covered by a 4 µm polypropylene film. An ASD (Field Spec 3) with a high intensity contact probe was set up in the field laboratory for visible-near to shortwave infrared reflectance measurements of the pressed pellets. A piece of Spectralon was used as the white reference. Each measurement consisted of an average of 30 scans. Processing of the data consisted of correcting the spectra to absolute reflectance by applying the Spectralon calibration reflectance factor using The Spectral Geologist (TSG™ version 7). Automated feature extraction was performed to estimate the proportions of minerals using TSG® software. QA-QC duplicate samples were incorporated into the existing automated data listing by manually adding an “a” to the ID prior to analysis of that duplicate sample.

Additional analyses and data were also collected using more traditional laboratory decomposition and instrumental methods and are reported in Noble et al. (2018). Major element oxide analysis from LabWest Minerals Analysis, Malaga, Western Australia, was carried out for comparison with fpXRF data. A Li borate fusion (Code AF-02) was used to ensure total recovery of highly refractory mineral phases and silica. Major element oxide analysis was performed using ICP-OES (Perkin Elmer Optima 7300DV) and ICP-MS (Perkin Elmer Nexion 300Q) instrumentation.

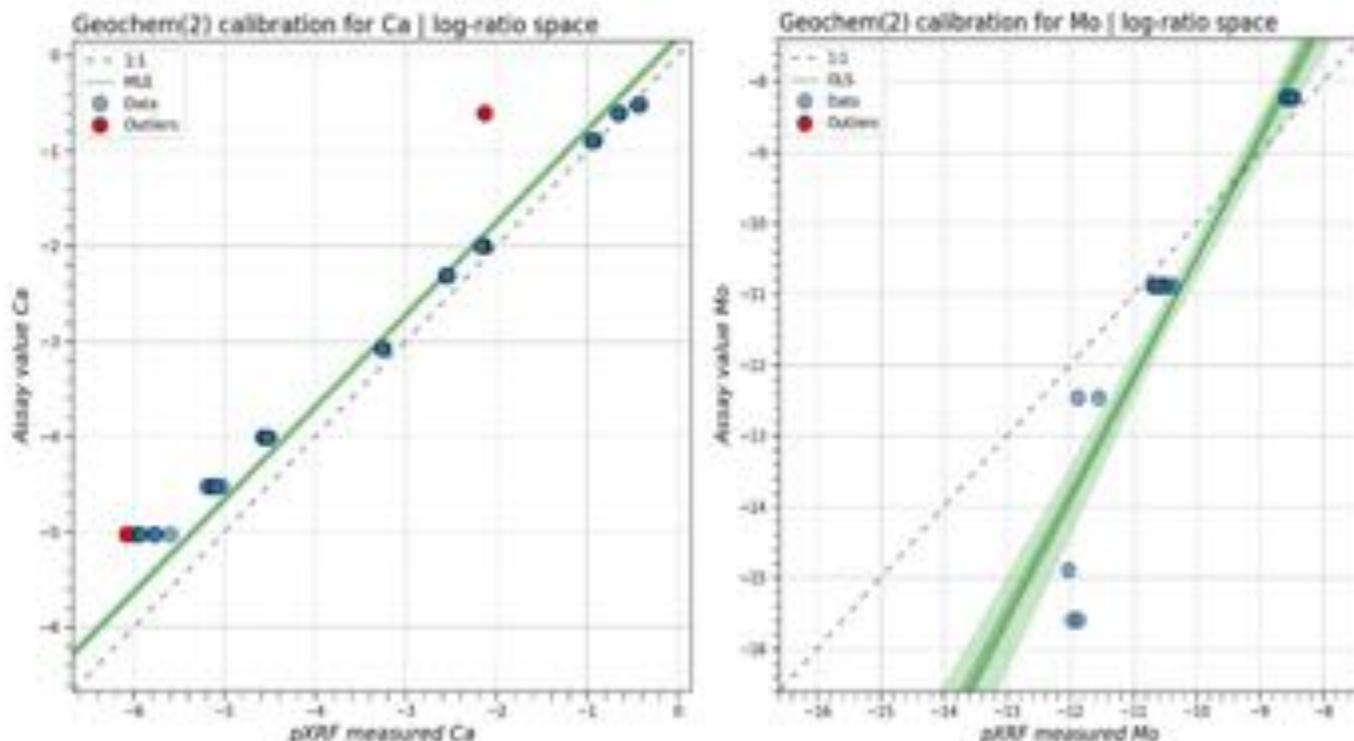


Figure 6. Examples of the CSIRO fpXRF calibrator output showing the data used for standards (blue dots) and rejected outlier data (red dots), 1:1 line and the derived calibration line (green). Laboratory analysis on the y-axis, fpXRF measurement on the x-axis for Ca (left) and Mo (right).

## RESULTS

### Workflow

The regional geochemistry sampling workflow established from this project is best represented by a flow chart (Fig. 7). Although a number of these points on the flow chart are flexible and will not fit all projects, the general steps are shown. The benefit of obtaining field geochemical data was greatly improved by using analytical standards based on the initial reconnaissance investigation. Machine learning models were tested in this project and the adaption of them to the workflow is shown in Fig. 7, but this aspect of the work is not covered in this paper.

### Field Geochemistry and Mineralogy

Over 300 sites were sampled including a targeted infill sampling (36 additional sites at closer spacing) to generate regional geochemical and proxy-mineralogical maps of soil and rock over an area of nearly 4000 km<sup>2</sup> on the Nullarbor

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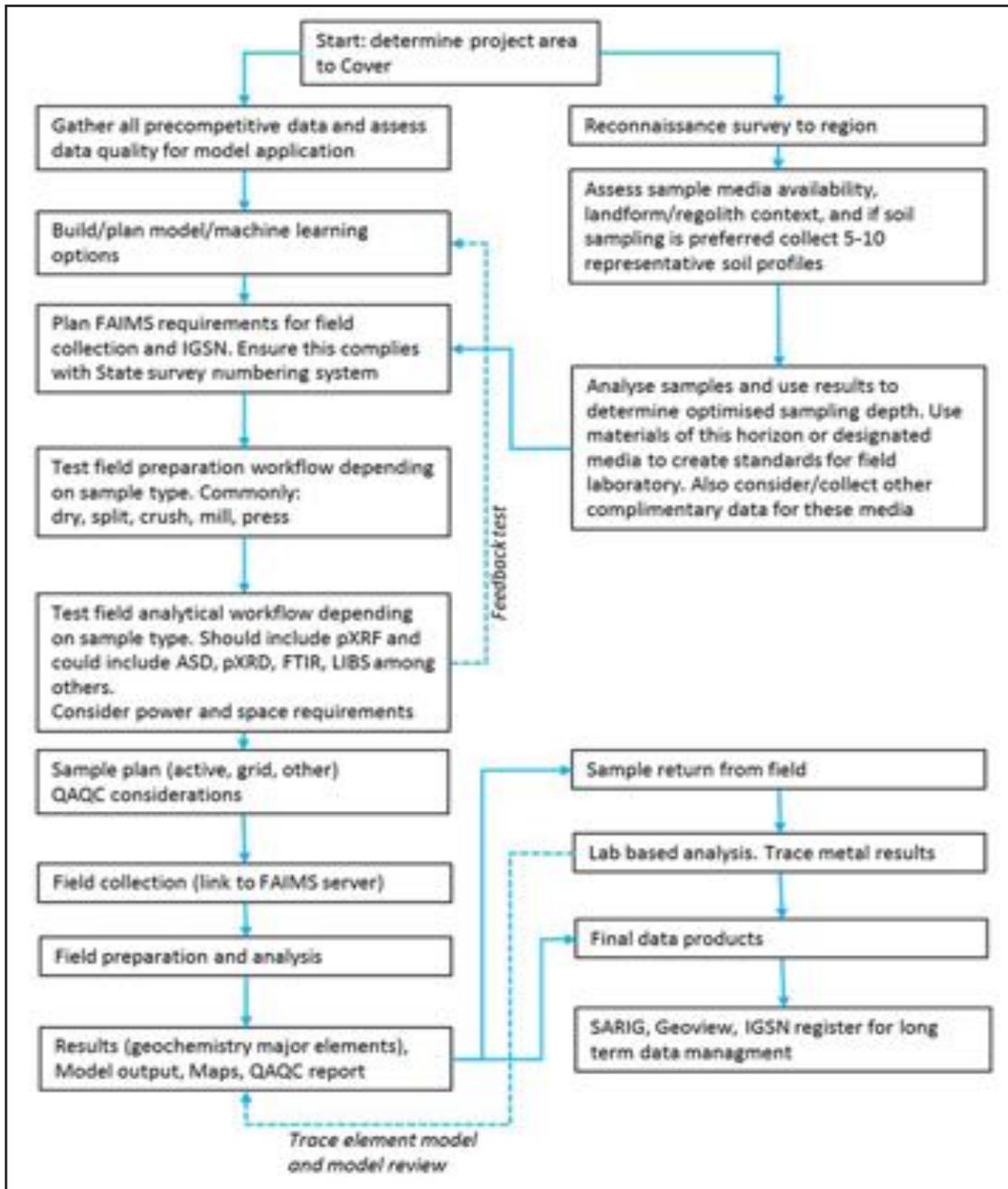


Figure 7. Regional geochemistry sampling workflow for optimised regional sampling and analysis as demonstrated on the Nullarbor Plain in South Australia. SARIG and Geoview are Australian State Geological Survey Data retrieval and visualisation platforms.

Plain. Elemental results from soil and lag were obtained using the fpXRF and mapped for approximately 20 elements and another 10 spectral mineralogical proxies were determined using the ASD.

A negative correlation between Al and Ca in the soils is interpreted as a result of increased clay minerals in ratio to carbonates. An increase of soil Ca in the SE of the region (Fig. 8) was identified as the results were being presented in element maps each evening. This pattern was assessed to relate to a change in the vegetation type and distribution (*Casuarina* sp. and *Eucalyptus* sp. trees were present in a nearly treeless plain) and an increase in some pedogenic carbonate. This observation would not have been recalled except for the near real-time results. Overall, soil Ca measured in the field by fpXRF was 8 to 14 wt.% (Fig. 8).

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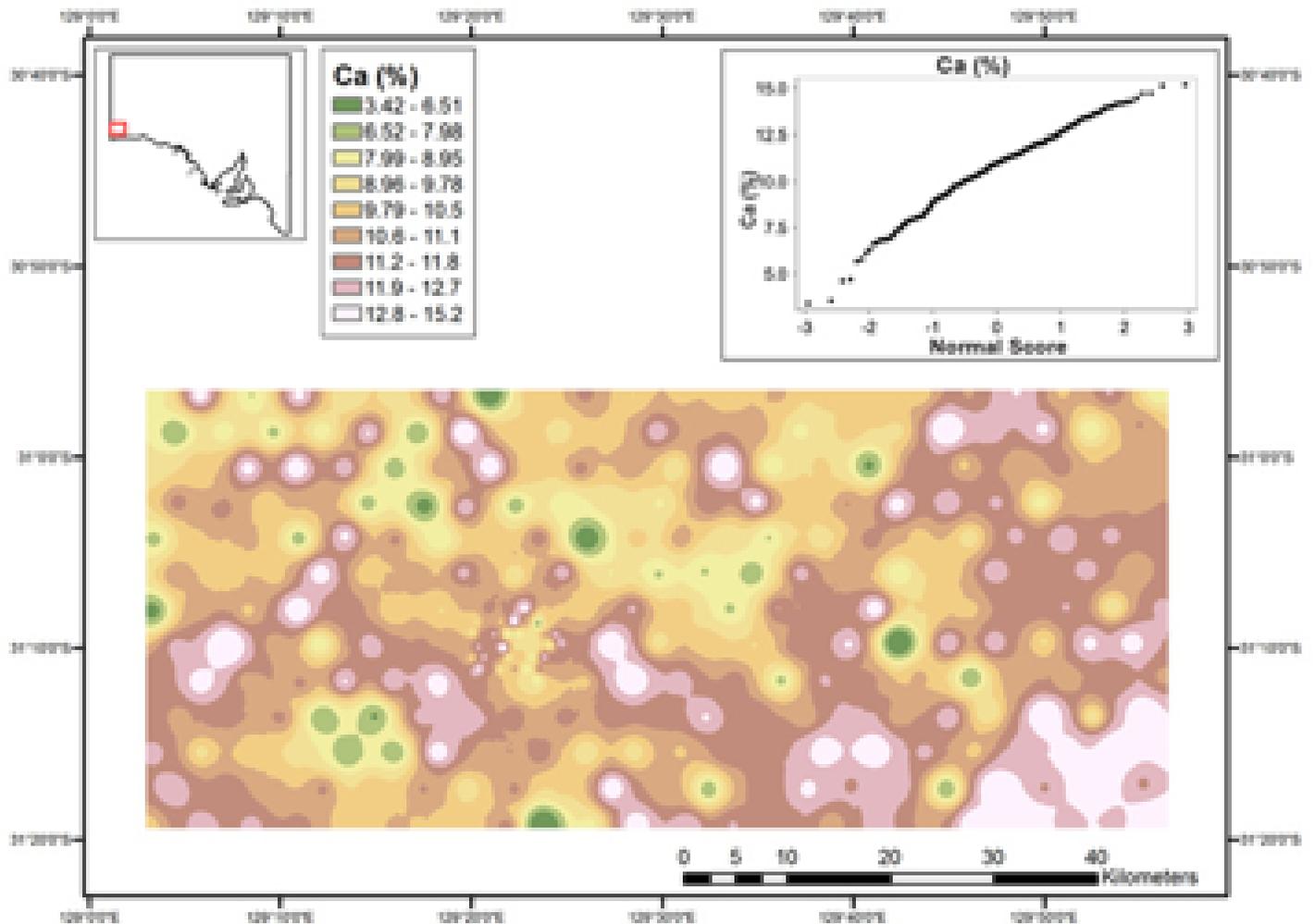


Figure 8. Calcium concentration in shallow soils measured using fpXRF. Data are contoured using inverse weighted distances. Insets show the distribution of the data and the site location with the state of South Australia as the reference.

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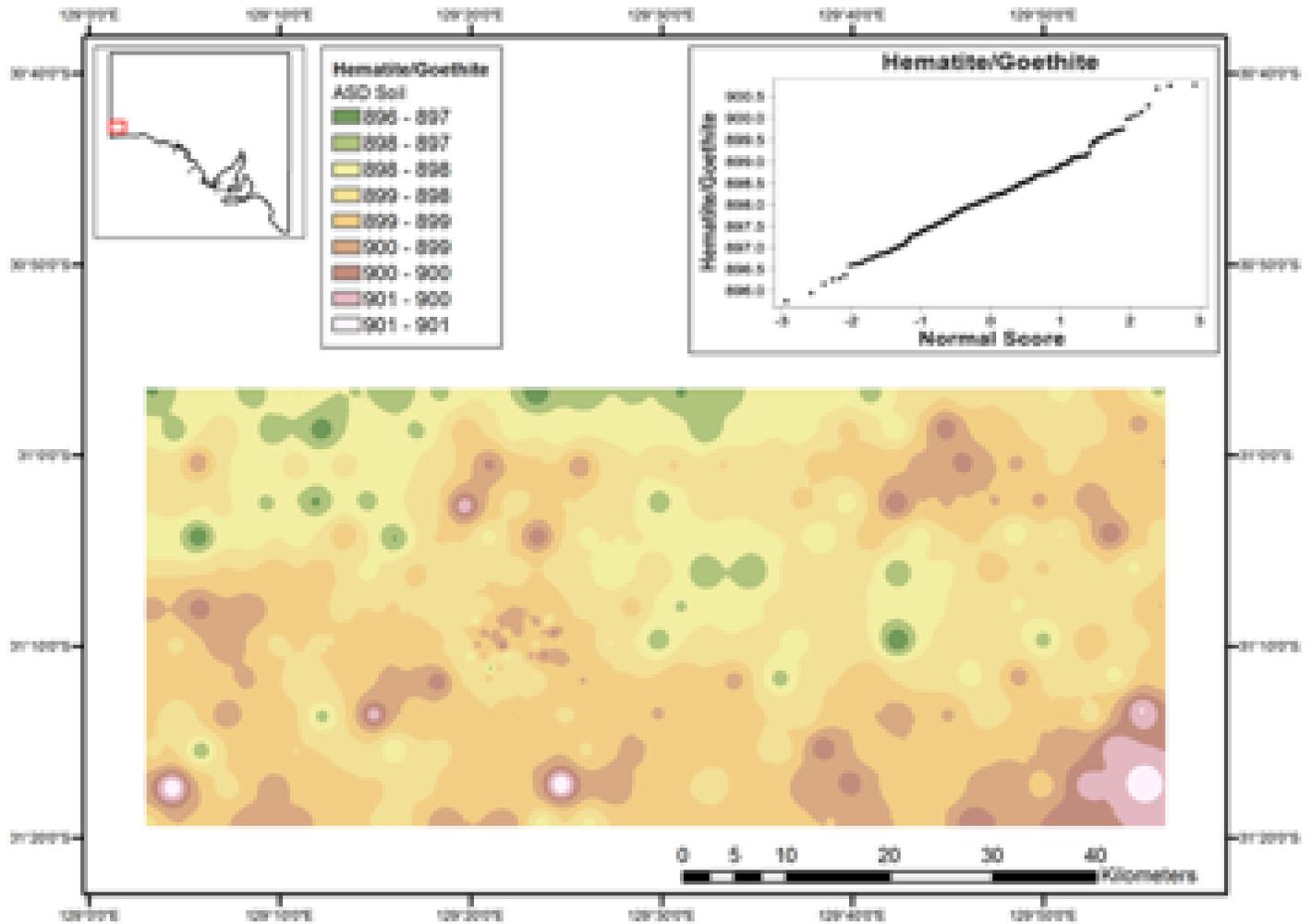


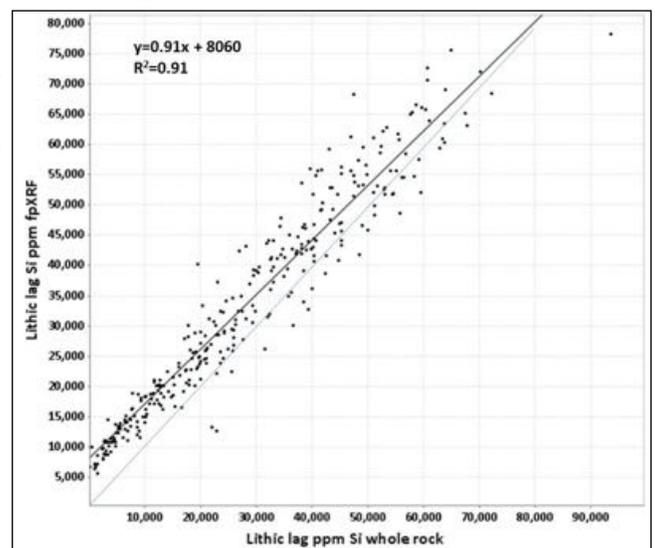
Figure 9. Hematite:goethite using the wavelength of the absorption feature around 900 nm in shallow soils measured using ASD for the Coompana region. Data are contoured using inverse weighted distances. Insets show the distribution of the data and the site location with the state of South Australia as the reference.

Field ASD soil results showed trends that correspond to kaolinite abundance being greater towards the northern extent of the surveyed area. This pattern is similar to the ferric oxide abundance and in contrast to the hematite:goethite ratio (Fig. 9) maps generated in the field. These results suggest subtle pedogenic changes (increased weathering) has occurred to the north of the survey area that are not evident in the surface landscape or vegetation. The Nullarbor Plain soils were all geochemically similar and there were no broad surface anomalies in the spectral reflective properties and element concentrations.

Lithic lag chemistry results showed little variation in major element composition across the area, which is expected given there is a large, relatively uniform, limestone sedimentary layer just beneath the surface. The fpXRF data for major elements (Ca, Al, Fe, K and Si) collected in the field are consistent with the commercial laboratory data (Table 1 and Fig. 10). This similarity was the case for both soil and lithic lag sample media with only few minor differences as noted in Table 1.

Other media collected and analysed at each site included a soil crust/cryptogam sample and the two dominant plant species pearl blue bush (*Maireana sedifolia*) and salt bush (*Atriplex vesicaria*). These results are available in Noble *et al.* (2018).

Figure 10. Scatter plot showing the results for Si determined by fpXRF and in the laboratory - major element oxides (whole rock). The blue line is the 1:1 line and the black line shows the linear regression line of best fit.



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Table 1. Geochemical comparison between major elements determined by fpXRF (acquired in the field) and Li borate fusion ICPMS/OES (laboratory). Regression results are for lithic lag analysis.

Element	Results
Al	Equivalent for spatial patterns and concentrations. $R^2 = 0.88$ ; fpXRF = 0.76 laboratory result + 1584
Ca	Equivalent for spatial patterns and concentrations. $R^2 = 0.82$ ; fpXRF = 1.03 laboratory result + 69325
Fe	Equivalent for spatial patterns and concentrations. $R^2 = 0.91$ ; fpXRF = 1.22 laboratory result + 1188
K	Equivalent results for spatial patterns. Concentrations near detection limit in fpXRF with less precision. $R^2 = 0.91$ ; fpXRF = 0.90 laboratory result + 296
Mg	pXRF not equivalent as Mg content was below detection limits for most samples using fpXRF. $R^2 = 0.01$
Mn*	Equivalent results for spatial patterns. Concentrations near detection limit in fpXRF with less precision. fpXRF did identify the anomalously high Mn value. $R^2 = 0.00$ * Mn assayed by 4-acid digest and ICP-MS outperformed both fpXRF and major element oxide analyses (not shown)
S	Equivalent results for spatial patterns. Concentrations with fpXRF had less precision. $R^2 = 0.81$ ; fpXRF = 0.81 laboratory result + 369
Si	Equivalent for spatial patterns and concentrations. $R^2 = 0.91$ ; fpXRF = 0.91 major oxide assay + 8060

## DISCUSSION

Significant cost benefits were realised in this project via the use of portable laboratories. The reliability and reproducibility of fpXRF data were excellent and comparable to laboratory instruments for major elements (Ca, Al, Fe, K and Si; Table 1; Noble et al., 2018), meaning that the additional commercial analysis for major elements was not needed and would, in future work, significantly reduce the analytical budget. Analytical costs would have been reduced by > 60% (even with the consideration of mobilizing equipment and additional people on site) should we have had this comparative assessment knowledge prior to this project.

A few other more advanced rapid analytical platforms adjacent to drilling samples have been published such as Lab-At-Rig for drilling muds (Fabris 2017; Lemièrè & Uvarova 2017) or fluid analysis (Fabris 2017; Reid 2016), and this concept is broadly accepted as a positive transition for the industry. Greenfields rapid analysis has been primarily driven by individual handheld devices with “point and shoot” style analyses. Inconsistency in sampling with pXRF is a major problem that is often overlooked in the exploration industry. The very fact that we have used a portable field laboratory, rather than just “pointing and shooting” the instrument, is an example of how to address this problem.

The transition from more routine data capture and sample labelling processes is less well published. In our study, it was estimated that field time was reduced from approximately 15 minutes to 6 minutes per site. With limitations on helicopter flying time and related costs, the use of the faster workflow saved 10 days of field time and \$150,000. In addition, the data transfer onto other devices saved at least a few hours per day of manual data entry. This saving is not a large economic one, but it enabled the team to quickly process data in the evening (and not miss out on sleep). The benefit of this savings became quickly apparent when the team was reviewing data in the evening, in some cases only a few hours after collection, and was able to make interpretations based on their recent memory (i.e. few larger trees in the area of increased pedogenic carbonate). This information would not have been captured if the review of these data occurred a month or so later from the

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city-based office. Although this survey was not a true mineral exploration targeting exercise, the comparison of the team's interpretations of changes in landscape setting with the geochemistry, and corollary to changing mineral exploration soil sampling patterns/locations in the field based on new information is clear. Infill sampling was conducted on 1 km spacing (36 sites) on the final day, to effectively demonstrate the agile sampling approach based on the results of the regional geochemistry maps being produced in nearly real-time. This study provided a demonstration of using a portable laboratory to focus exploration without the need for later remobilisation (second sampling trip). Remobilisation can be a major cost. In this project, it was a minimum of \$30,000, with much of that attributed to getting a helicopter to such a remote area (Perth is approximately 1600 km away and Adelaide 1400 km from the study site).

The region is an unknown for mineral potential, although a number of exploration licences have been granted with companies focused for Cu and Ni magmatic systems (Lawley 2017). The soils and the lithic lag do not highlight anomalous surface geochemical results for Cu or related pathfinders. The detection limits with fpXRF also limit the effectiveness of the field results for trace metal analysis, even though this is improving regularly. The most problematic elements based on this work were Mg and Mn for the major elements and Ag, As, Bi, Co, Mo, Sn and Zn being effectively below detection in this setting. Copper and Ni results were of marginal value and also approaching detection limits. This region is particularly difficult to characterize as the soils show low total base metal abundance (Noble *et al.* 2018). Due to the thickness the underlying sedimentary rocks any possible metal dispersion from the potential magmatic prospective sequences at depth are not reflected in the surface media as indicated by Gonzalez-Alvarez *et al.* (2018; 2019). In other regions where pathfinder or target elements were present and detected with fpXRF due to greater concentrations, (e.g., Sarala 2016; Sterk *et al.* 2018), this field-based approach could drive exploration targeting.

Using the ASD and spectral mineral proxies features such as soil colour, hematite, goethite and recognised accessory mineral phases were an effective way to detect atypical samples. Application of spectral mineralogy is expanding rapidly with major laboratories now offering this type of analysis. Presently, the geoscience community does not commonly merge the spectral mineralogy with the elemental chemistry for interpretation, but it is likely that this will become more widely used and manageable in the future. This study did not integrate the spectral data with the geochemistry in the tested machine learning model (see Noble *et al.* 2018). Other studies have used machine learning approaches to handle more geochemical data and gain different insight for mineral exploration or geological mapping (Zuo & Ziong 2018; Kirkwood *et al.* 2016), but the interoperability with other data types is not well developed. Easily adapted portable or field technologies include pXRF, pXRD, pNIR-SWIR,  $\mu$ Raman, and LIBS (Lemièrè & Uvarova 2017). It is expected future projects will adapt this workflow further to provide other portable instrument data, and combine this with precompetitive data and data analytics to better predict the geochemistry mineralisation targets in the field.

### CONCLUSIONS

As a demonstration of rapid characterisation akin to greenfields regional exploration, the study showed that quick and efficient sampling and field analyses could be successfully achieved with minimal environmental impact. To generate new targets using standard regional surface geochemistry, three key activities commonly take place. Firstly, uniform sampling (using grids or cells) is conducted over a broad area, secondly, analyses of samples are conducted to establish geochemical backgrounds, and finally areas of interest are identified for further investigation. This process commonly takes months and is widely practiced. However, huge efficiencies can be delivered using straight-forward technologies such as tablets and Apps for rapid data gathering and transfer coupled with a portable preparation and analysis laboratory, using pXRF and ASD, to produce nearly real-time geochemical and mineralogical maps. We believe the next iteration of efficient regional sampling should arise through the development of "smart" or "active" sampling that will use other spatial data and models (machine learning) to inform sampling locations to maximise the information/minimize uncertainty. However, that requires further research and development.

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