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MINERALOGY AND PETROLOGY ATLAS OF SEDIMENTARY ROCKS FROM STRATIGRAPHIC WELL BARNICARNDY 1 USING THE TESCAN INTEGRATED MINERAL ANALYZER (TIMA)

Z Li, BIA McInnes and LS Normore

Department of **Energy, Mines, Industry Regulation and Safety**

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ARAHAN MARA John de Laeter Centre

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Isotope and element analyses were conducted using the GeoHistory laser-ablation ICP-MS facility at the John de Laeter Centre (JdLC), Curtin University, with the financial support of the Australian Research Council and AuScope (auscope.org.au) and the Australian government via the National Collaborative Research Infrastructure Strategy (NCRIS).

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We respectfully acknowledge Aboriginal peoples as the Traditional Custodians of this land on which we deliver our services to the communities throughout Western Australia. We acknowledge their enduring connection to the lands, waterways and communities and pay our respects to Elders past and present.

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Cover image One of the largest and most distinctive metagranitic units in the Gascoyne Province, the Davy Well Granite emerges from the water of the Yinnetharra Pool along the Gascoyne River. Photo by Angela Riganti

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Mineralogy and petrology atlas of sedimentary rocks from stratigraphic well Barnicarndy 1 using the TESCAN Integrated Mineral Analyzer (TIMA)

Z Li*, BIA McInnes* and LS Normore

Abstract

The TESCAN Integrated Mineral Analyzer (TIMA) has been used to establish a petrographical atlas of 47 sedimentary rock samples from stratigraphic well Barnicarndy 1, located in the Barnicarndy Graben, western Canning Basin. The samples were analysed by scanning electron microscopy/energy dispersive X-Ray spectrometry (SEM/EDX) techniques which generated 2D maps displaying mineralogical and compositional information. Modal mineral abundances, grain-size distributions, and mineralogy models are provided. Samples containing minerals suitable for in situ radiometric dating (e.g. ankerite, apatite, biotite, calcite, dolomite, rutile and zircon) have been identified for potential follow-on geochronology studies. Initial porosity estimates from TIMA analysis can be used to prioritize high-value reservoir samples for more detailed 3D porosimetry testing. The establishment of a Barnicarndy 1 sample atlas will be useful in understanding the geological evolution of the western Canning Basin and Paterson Orogen regions.

KEYWORDS: automated quantitative mineralogy, Barnicarndy Graben, Canning Basin, Paterson Orogen, porosity, TESCAN Integrated Mineral Analyzer, SEM/EDX

Introduction

A multiphase research project between the Geological Survey of Western Australia (GSWA) and Curtin University was designed to build a detailed understanding of the mineralogy, petrology, and geochronology of stratigraphic well Barnicarndy 1, Barnicarndy Graben, western Canning Basin (Fig. 1) to further promote exploration for mineral and energy resources and test the potential for carbon capture and storage in this region. This report presents the results of Phase 1 of the project utilising a non-destructive automated scanning electron microscopy/energy dispersive X-Ray spectrometry (SEM/EDX) technique for 2D mineralogical characterization of 47 sedimentary rock samples. An objective of Phase 1 was the creation of a petrological atlas for sedimentary rocks from the well, including quantitative mineralogy, grain size and porosity data.

Previous collaborative work between GSWA and Curtin University has resulted in the integration of automated mineralogy and in situ geochronological analysis to determine the stratigraphic ages of sedimentary rocks (Li et al., 2023). TESCAN Integrated Mineral Analyzer (TIMA) petrology provides high-resolution mineralogic characterization and enables effective sample/domain screening of minerals for in situ laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS) uranium– lead (U–Pb) of carbonate minerals, zircon, apatite and rutile, and rubidium–strontium (Rb–Sr) dating of biotite and muscovite. In this study, the TIMA petrology maps allow for the identification of mineral grains (e.g. ankerite, apatite, biotite, calcite, dolomite, rutile and zircon) suitable for radiometric dating.

Geology

Barnicarndy 1 is the only continuously cored deep stratigraphic well (2680.5 m) in the Barnicarndy Graben, western Canning Basin (Normore et al., 2023, 2024). The samples returned represent an excellent natural laboratory to determine basin depositional histories using automated petrology and radiometric dating methods. The stratigraphy consists of 96 m of Cenozoic regolith unconformably overlying 759 m of Carboniferous to lower Permian Grant Group to 855 m depth. The Grant Group unconformably overlies a thick section of Ordovician rocks to 2585 m, comprising in descending order, the Barnicarndy, Nambeet and Yapukarninjarra Formations. Another major unconformity marks the base of the Canning Basin at 2585 m, with 95 m of weathered siltstone and dolomite of the Neoproterozoic Yeneena Basin cored to the bottom of hole at 2680.5 m (Fig. 2).

Samples

Forty-seven core plugs from the Barnicarndy 1 core over a depth range of $599.87 - 2679.72$ m were extracted for petrographic (Appendix 1) and routine core analysis (Appendix 2; Normore et al., 2023). Core plug stub ends, or thin-section stubs (Table 1) were used for TIMA analysis to provide direct comparison with petrographic and routine core analysis (RCA) data (Fig. 2). This study included a wide variety of sedimentary rock types including arkose to quartz arenite sandstone, mudstone, siltstone, limestone, dolomite, diamictite and volcanic ash. Two samples of an ash bed at 1891.87 and 1891.95 m, within the Samphire Marsh member of the Nambeet Formation, reported ultraprecise chemical abrasion isotope dilution thermal ionization *John de Laeter Centre, Faculty of Science and Engineering, Curtin

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Figure 1. Regional map of the southwestern Canning Basin, showing the location of deep stratigraphic well Barnicarndy 1, Kidson deep crustal seismic line 18GA-KB1 (red), first vertical derivative magnetic anomalies (background image), tectonic subdivisions, major crustal boundaries, 1:250 000 map sheet boundaries, local surface faults, nearby mineral deposits and relevant petroleum exploration wells

Figure 2. Stratigraphic column modified from Normore et al., 2023, showing TIMA, petrography, hylogger data, routine core analysis, geochronology, and thermochronology sample locations

GSWA sample #	Depth (m)	Rock type	TIMA resolution	RCAØ%	TIMA Ø %
251816	745.73	muddy sandstone	dot mapping	10.8	4.55
251817	799.17	sandy mudstone	dot mapping	8.4	1.97
251818	846.79	sandstone	dot mapping	23.8	5.02
251819	871.64	sandstone	dot mapping	16.1	19.50
251820	922.27	sandstone	high resolution	17.4	20.34
251821	970.60	sandstone	dot mapping	15.7	19.94
251822	1026.37	sandstone	dot mapping	15.4	16.88
251823	1070.47	sandstone	dot mapping	21.3	27.05
251824	1127.08	sandstone	high resolution	24.7	13.82
251825	1172.89	sandstone	dot mapping	16.4	18.90
251826	1228.04	sandstone	dot mapping	16.7	21.59
251827	1269.34	sandstone	dot mapping	18.2	22.57
251828	1320.12	sandstone	dot mapping	17.7	20.32
251829	1360.54	sandy mudstone	dot mapping	6.7	2.08
251830	1421.52	sandy fossiliferous dolomitic mudstone	dot mapping	8.4	1.74
251831	1500.78	calcareous sandy mudstone	dot mapping	N/A	0.74
251832	1528.53	calcareous mudstone	dot mapping	5.6	0.37
251833	1569.77	muddy sandstone	dot mapping	9.9	20.01
251834	1604.20	sandy fossiliferous mudstone	high resolution	7.6	2.40
251835	1700.58	sandy calcareous mudstone	dot mapping	N/A	0.54
251836	1801.68	sandy mudstone	dot mapping	N/A	0.25
251837	1891.87	ash bed	dot mapping	N/A	1.69
251838	1900.32	sandy calcareous mudstone	dot mapping	3.3	0.26
251839	2000.05	sandy calcareous mudstone	dot mapping	N/A	0.32
251840	2098.80	sandy mudstone	dot mapping	N/A	0.10
251841	2200.26	sandy limestone interbedded with mudstone	dot mapping	N/A	0.10
251842	2267.30	sandy mudstone	Both HR and DM	N/A	1.20
251843	2296.88	sandstone	Both HR and DM	2.2	1.99
251844	2321.42	muddy sandstone	dot mapping	3.9	0.61
251845	2346.58	sandstone	dot mapping	9.1	10.09
251846	2378.33	muddy sandstone	dot mapping	3.8	0.32
251847	2411.30	sandy mudstone	dot mapping	1.8	0.18
251848	2436.58	muddy sandstone	high resolution	$\mathbb O$	0.84
251849	2460.36	muddy sandstone	dot mapping	0.7	0.61
251850	2476.60	ash bed	dot mapping	N/A	1.05
251851	2487.48	sandstone	dot mapping	18.4	18.05
251852	2509.31	sandstone	dot mapping	12.4	13.48
251853	2530.63	sandstone	dot mapping	7.8	11.00
251854	2545.20	sandstone	dot mapping	3.4	4.62

Table 1. Summary of 47 samples from the Barnicarndy 1 well for TIMA analysis. RCA and TIMA porosity results are listed for comparison

2563.56 sandstone high resolution 9.6 14.20

mass spectrometry (CA-ID-TIMS) zircon U–Pb dates of 477.24 ± 0.58 and 477.24 ± 0.41 Ma (2σ) (Normore et al., 2023) interpreted as the maximum depositional age.

Methods

Petrologists use SEM techniques to determine the chemical composition and morphology of samples at the microscopic scale. Compositional data is obtained using EDX which measure the spectrum of X-rays generated during the interaction between the electron beam and the analysed mineral. The mineral's spectral signature is characteristic of the atoms making up its crystallographic structure. The spectrum of an unknown mineral can be compared to a library of known mineral spectra, and where there is a match, the unknown mineral can be positively identified. The robotic automation of this analytical process allows for the rapid and accurate identification of rock samples at the microscopic scale and is termed automated petrology.

The automated petrology system at the John de Laeter Centre (JdLC), Curtin University, was manufactured by TESCAN. TIMA is comprised of a field emission SEM equipped with four silicon EDX. Samples suitable for TIMA analysis include polished whole rock, heavy or light mineral separates, or drill cuttings. They can be prepared as round mounts of 25 or 30 mm in diameter, rock slabs or thin sections with dimensions up to 27 x 46 mm or blocks up to 50 x 75 mm (Fig. 3).

Sample preparation

Due to the non-standard size/shape of core plug ends and thin-section billets, sample preparation was conducted at JdLC, where samples were cut to size, mounted, and polished prior to SEM/EDX analyses. The polished samples were then placed into the vacuum chamber of a Cressington carbon evaporative coater, and a 10 nm carbon coating was deposited on the sample surface. The samples were then taken directly to the TIMA instrument for quantitative analysis.

SEM/EDX analytical conditions

The analytical conditions for this TIMA project included an accelerating voltage of 25 kV, probe current of 3.2 – 5.5 nA, beam intensity of $17.1 - 18.9$, spot size of $35.7 - 83.8$ nm, and a nominal working distance of 15.0 mm. A total of 47 samples were analysed (Table 1): 1) Thirty-nine samples using dot mapping (DM) mode at a back-scattered electron (BSE) pixel spacing of 3.0 μm and EDX dot spacing of 27 μm; 2) eight samples using high-resolution mapping (HR) mode at a 3.0 μm pixel size for both the BSE and EDX data collection. In the DM mode, the combination of the high-resolution BSE image and the lower-resolution EDX data is used to greatly improve the analytical efficiency with reasonable data collection time. This mode can still provide minerals/phases segmentation results because the X-ray data from zones of similar BSE and EDX signals are summed to produce single higher quality spectra for each final segment (Hrstka et al., 2018). The HR mode can provide improved results because the X-ray spectroscopic data are

collected with the same high resolution as the BSE image, however this method requires a much longer period of data acquisition. The interpretation of the raw TIMA data was undertaken using the TIMA 2.6.1 software. The subroutine 'Auto identify phases' (Auto-ID; for 32 out of 47 samples) and 'Field stitching' (for all the samples) operations were carried out to improve the TIMA analyses of fine-grained sediments and 2D porosities.

Unclassified phase

The category of unclassified refers to analyses where the measured X-ray spectra did not match any of the spectra in the TIMA mineral library. This is a common phenomenon in fine-grained sedimentary rocks, where clay mineral grain dimensions (<20 μm) are often smaller than the electron beam interaction volume. Consequently, the X-ray spectrum generated from ultrafine- grained rocks is potentially that of a heterogenous mineral assemblage that cannot be resolved as a single mineral. To minimise the reporting of unclassified phases, two procedures were adopted: 1) use of HR acquisition mode on select lithologies which can generate improved results albeit over a much longer required period of collection; 2) utilization of Auto-ID mode on samples reporting >5% unclassified phases. TIMA measurements before/after Auto-ID are stored separately in the source data (Fig. 4). Once Auto-ID has been performed the remaining unclassified phase reported can be attributed to fine-grained mineral assemblages (Fig. 5).

Porosity assessment

The TIMA method can generate estimates of rock porosity based on the detection of holes or gaps in the sample surface. Thus Holes (%) reported in the modal mineral abundance data (Appendix 3) represents areas where minerals are absent in two dimensions. An algorithm in the TIMA software propagates this information into a 3D volume estimate if the hole represents a spherical volume and approximates the sample's subsurface porosity.

Results

The entire datasets of all 47 samples are provided in Appendix 3 with high-resolution images and quantitative mineralogy spreadsheets in Appendix 4. Supplementary raw TIMA source data related to Appendices 3 and 4 is over 280 GB in size and can be obtained by request from GSWA.

2D TIMA porosity

TIMA-derived porosity data are presented in Figures 6 and 7 and listed in Table 1. TIMA porosity results can be compared to porosity data measured using conventional 3D mercury/ helium porosimetry techniques carried out separately on Barnicarndy 1 core plug samples in previous RCA (Fig. 6). The TIMA 2D porosity estimates roughly correlate with conventional porosity determinations (Fig. 7), with differences in results likely attributable to sample preparation artefacts (e.g. artificial fracturing and/or plucking of grains

Figure 3. Samples suitable for TIMA analysis: a) round mounts of 25 or 30 mm in diameter; b) rock slabs or thin-section stubs with dimensions up to 27 x 46 mm; c) polished blocks up to 50 x 75 mm

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Figure 4. Example of TIMA measurements before/after Auto-ID operation

Figure 5. Grain-size distribution of Unclassified phases in the Barnicarndy 1 samples after Auto-ID operation

Figure 6. TIMA 2D porosity estimation vs 3D mercury/helium porosity from routine core analysis. The three samples in red are outliers which are discussed in the main text

Figure 7. Stratigraphic column modified from Normore et al., 2023, showing the comparison of routine core analysis and TIMA porosities of core samples from the Barnicarndy 1 well in the Canning Basin

from epoxy), natural porosity variation between sample aliquots and deficiencies in the assumption that 2D surface data can be reliably propagated into spherical 3D volumes.

Three outliers of TIMA 2D porosity results (Barnicarndy 1 well samples 251818, 251824, and 251833; Fig. 6) exemplify the differences of porosity estimates generated by the TIMA and RCA techniques. The artificial rock/grain fracturing and grain plucking during sample preparation is observed in core plugs 251818 and 251824 (Fig. 8; Appendix 3), which may have changed some pores into void spaces connected to the outer epoxy in the mount/section. Such connected spaces were not included in the calculations of Holes (%) in the modal mineral abundance results due to the limitation of TIMA Background and Porosity Mode, resulting in the underestimation of TIMA 2D porosity. The significant internal inhomogeneity in core plug 251818 (Fig. 8; bimodal size distributions of sediments; see Appendix 1) could have also contributed to the differences between the TIMA and helium porosities, because the exposed surface for TIMA analysis may not be representative of the entire core plug sample. On the other hand, the removal of sand grains from the epoxy-impregnated muddy sandstone 251833 during sample sawing and/or grinding could have resulted in the exceptionally large artificial pores in this case, leading to the overestimation of TIMA 2D porosity (Fig. 9).

TIMA analysis can provide a rapid and practicable assessment of total porosities of rock samples (Table 1) if high-precision mercury/helium porosity techniques are not available. The accuracy of TIMA 2D porosity, however, needs to be assessed by comprehensive consideration of the quality of prepared samples, the likelihood of porosity inhomogeneity in samples, and TIMA imaging resolution. We propose that the TIMA porosity data be reviewed early in the analytical workflow, and used as a guide as to which samples should be submitted for higher precision 3D porosimetry testing.

TIMA automated 2D mineralogy

The TIMA automated mineralogy results are presented in the following order for each sample: 1) back-scattered electron (BSE) image; 2) secondary electron (SE) image; 3) mineralogical model; 4) grain-size analysis (all phases); 5) grain-size analysis (dominant phase); 6) grain-size distribution of mineral candidate(s) for in situ geochronology.

The TIMA outputs for a calcareous sandy mudstone sample (GSWA 251831 from 1500.78 m depth within the Samphire Marsh Member of the Nambeet Formation) is illustrated in Figures 10 and 11 and are discussed as an example of the results of this technique. Panorama BSE and SE electron images are provided in Figures 10a,b. The mineralogical model (Fig. 10c) consists of a panoramic image of the primary phases and a legend of the modal mineral abundance (volume %). Grain-size analysis includes the grain-size distribution of all mineral phases (Fig. 11a) followed by the dominant phase (Fig. 11b). The grain-size

statistical output, including median size of grains and P80 in microns (the latter represents the size of grains, below which 80% of the selected grain population resides), is also provided for each grain-size chart. The grain-size estimates are based on the actual measured area of individual grains, however for comparative purposes these areas are converted into circular equivalents, with the diameter of the circle roughly equivalent to the grain dimensions.

In situ geochronology candidates

A panoramic image of each potential geochronometer for in situ radiometric dating is provided in TIMA outputs. For example, mudstone sample 251831 has two potential geochronology candidates: biotite (Fig. 12) and calcite (Fig. 13). In addition to the biotite plus BSE image (Fig. 12a), a grain-size distribution chart of the biotite grains (Fig. 12b) is shown adjacent to images of the larger individual biotite grains which are over 175.43 μm (Fig. 12c). This sample contains 124 biotite grains with grain sizes suitable for in situ Rb–Sr dating using LA-ICP-MS. Laser ablation spot size for in situ biotite Rb-Sr dating is generally > 50 µm but may be > 100 µm depending on expected sample age and radioisotope content. The calcite grains from this sample are shown in Figure 13 with 78 calcite grains having diameters exceeding 139.82 µm, a mineral size which may be suitable for in situ U–Pb dating by LA-ICP-MS. Laser ablation spot size for in situ calcite U–Pb dating is generally > 75 um but may be > 100 µm depending on expected sample age and radioisotope abundance. Full results of potential in situ geochronology candidates are provided in Appendices 3 and 4.

Conclusions

The foundations of a TIMA sedimentary rock atlas have been established using 47 samples from the deep stratigraphic well, Barnicarndy 1. The TIMA instrument enabled rapid determination of mineralogy, mineral associations, modal mineral abundances, grain size, morphology, porosities, and textures. The TIMA 2D porosity values are comparable with conventional 3D mercury/helium porosimetry values. Future analytical workflows should use TIMA 2D porosity measurements as a screening guide for samples requiring higher precision 3D porosimetry testing.

Automated 2D mineralogy using the TIMA has demonstrated it as an effective screening and selection tool for in situ geochronology on a variety of mineral grains. This includes ankerite, calcite, dolomite, biotite, apatite, zircon and rutile that are amenable to U–Pb, fission track, (U–Th)/He and Rb–Sr geo/thermochronology techniques. The in situ nature of this analysis in polished sections, retains the petrographic context of the minerals being dated and can assist with the interpretation of the age results applicable to metamorphism, hydrothermal alteration, timing of mineralization events and paragenetic sequence.

Outliers: GSWA No. 251818 (underestimated TIMA 2D porosity)

Figure 8. An example of underestimated TIMA 2D porosity resulting from sample preparation artefacts and rock inhomogeneity

Outliers: GSWA No. 251833 (overestimated TIMA 2D porosity)

Figure 9. An example of overestimated TIMA 2D porosity resulted from the exceptionally large artificial pores produced during sample preparation

Figure 10. TIMA automated 2D mineralogy results for GSWA sample 251831 from 1500.78 m in the Barnicarndy 1 well: a) BSE image; b) SE image; c) TIMA mineralogical model mapping with modal mineral abundance

Grain-size distribution: quartz

Figure 11. Grain-size analysis of mineral phases in GSWA sample 251831 from 1500.78 m in the Barnicarndy 1 well: a) all phases; b) dominant phase (quartz)

Figure 12. Grain-size distribution of biotite in GSWA sample 251831 from 1500.78 m in the Barnicarndy 1 well: a) panorama of biotite grains overlying BSE image; b) biotite grain-size distribution chart; c) images of individual biotite grains with grain sizes over 175.43 μm

Figure 13. Grain-size distribution of calcite in GSWA sample 251831 from 1500.78 m in the Barnicarndy 1 well: a) panorama of calcite grains overlying BSE image; b) calcite grain-size distribution chart; c) images of individual calcite grains with grain sizes over 139.82 μm

Figure 14. Stratigraphic column showing recommended candidate samples for in situ geochronology from the Barnicarndy 1 well

Recommendations

X-ray computed tomography (XCT) scans are recommended for each of the major rock types (including sandstone, siltstone, mudstone, carbonate) encountered in the Barnicarndy 1 samples. The integration of XCT 3D scanning and TIMA 2D mineralogical characterization are necessary to build a multimodal mineralogy database of sedimentary rocks in the Canning Basin. The resultant multimodal mineralogy will enable the 3D visualization and volumetric reconstruction of mineralogical phases and pore space in samples and can be used to characterize quantitatively the diagenesis and cementation in seal and reservoir rocks in the Canning Basin.

Nineteen samples are recommended for in situ geochronology based on the distribution, quality, and quantity of suitable minerals (Table 2). The Barnicarndy 1 stratigraphic well already has a wealth of geochronological and biostratigraphic information, however the ages of two

new Formations defined in this well, the Barnicarndy and Yapukarninjarra Formations, and the underlying Yeneena Basin basement are currently constrained by detrital zircon geochronology and thermochronology. Two Barnicarndy 1 samples in the Samphire Marsh Member of the Nambeet Formation yielded CA-ID-TIMS zircon U–Pb dates of 477.24 +/‒ 0.58 and 477.24 +/‒ 0.41 Ma (Normore et al., 2023). These accurate dates provide excellent baseline age information useful in interpreting subsequent in situ geochronology of the various mineral phases. There is also excellent biostratigraphic control in the Nambeet Formation of Barnicarndy 1 with three conodont biozones (Zhen et al., 2022) and three trilobite assemblages identified (Smith and Allen, 2023). This sample set will help define the depositional, structural and diagenetic history of this unique part of the Canning Basin with implications for diagenesis, fluid flux and base metal mineralization in the graben bounded by the Barnicarndy, Parallel Range and Anketel– Samphire faults.

Table 2. Summary of 19 samples from the Barnicarndy 1 well recommended for in situ geochronology

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