

## Mid-Devonian basaltic magmatism and associated sedimentation: the Ooloo Hill Formation, central-eastern South Australia

C. Wade<sup>a,b</sup>, A. J. Reid<sup>a, b</sup>, E. A. Jagodzinski<sup>b</sup>, and M. J. Sheard<sup>c</sup>

<sup>a</sup>Geological Survey of South Australia, Adelaide, Australia; <sup>b</sup>Department of Earth Sciences, The University of Adelaide, Australia; <sup>c</sup>Morphettville, Adelaide, Australia

C. Wade <http://orcid.org/0000-0003-2569-2771>

A. J. Reid <http://orcid.org/0000-0002-9435-9342>

E.A. Jagodzinski <http://orcid.org/0000-0002-0151-2337>

**Contact** C. Wade email [Claire.wade@sa.gov.au](mailto:Claire.wade@sa.gov.au) mail Geological Survey of South Australia, GPO Box 320, Adelaide, SA 5001, Australia

### SUPPLEMENTAL DATA

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**Appendix 1.** Analytical methods for K–Ar geochronology, SHRIMP U–Pb geochronology and whole-rock geochemistry.

**Appendix 2.** Whole-rock geochemical major- and trace-element data for standards BCR-2, BIR-2, and BHVO-2. (excel file)

**Appendix 3.** U–Pb zircon geochronology data for two sedimentary samples from Ooloo Hill Formation. (excel file)

## Appendix 1. Analytical methods

### K–Ar analytical method

Five basaltic samples were analysed for whole-rock K–Ar geochronology. The K–Ar standard methods used in this study are described in Zwingmann and Mancktelow (2004). K content was determined by atomic absorption. The pooled error of duplicate K determinations on several samples and standards is better than 2.0%. Ar isotopic determinations were performed using a procedure similar to that described by Bonhomme *et al.* (1975). Samples were pre-heated under vacuum at 80 °C for several hours to reduce the amount of atmospheric Ar adsorbed onto the mineral surfaces during sample preparation. Ar was extracted from the mineral fractions by fusing samples using a low blank resistance furnace within a vacuum line serviced by an on-line  $^{38}\text{Ar}$  spike pipette. The  $^{38}\text{Ar}$  spike was calibrated against GA1550 biotite (McDougall & Roksandic, 1974). The isotopic composition of the spiked Ar was measured with an on-line VG3600 mass spectrometer via Faraday cup. The released gases were subjected to a two-stage purification procedure via CuO and Ti getters. Blanks for the extraction line and mass spectrometer were systematically determined and the mass discrimination factor was determined by airshots. About 25 mg of sample material was required for Ar analyses. During the course of the study, the international standards HD-B1 and LP6 were measured several times (Table 1). The error for Ar analyses is below 1.00% (Table 1). The K–Ar ages were calculated using  $^{40}\text{K}$  abundance and decay constants recommended by Steiger and Jäger (1977).

### SHRIMP U–Pb analytical methods

Two sedimentary samples were selected for SHRIMP U–Pb detrital zircon geochronology and processed by Geoscience Australia Geochronology Laboratories. Several hundred zircons were extracted from samples 1837679 and 1837680 through standard crushing, density and magnetic separation procedures and hand picking and were encapsulated together with zircon reference standards in a 25 mm epoxy resin disc, and polished to expose the interiors of those crystals. Zircons were photographed in transmitted and reflected light, and imaged by cathodoluminescence (CL), to ensure that analyses were made on discrete growth phases. U–Pb isotopic results were collected on the SHRIMP IIe instrument at Geoscience Australia in Canberra using a primary oxygen ion beam of ca 2.5–4.5 nA and ~20 microns in diameter. Secondary ions were collected on a single electron multiplier via cycling of the magnet through 5 scans across 10 mass stations of interest.

Absolute U and Th concentrations were estimated by comparison with the M257 zircon standard (840 ppm  $^{238}\text{U}$ ).  $^{206}\text{Pb}/^{238}\text{U}$  ratios were determined relative to the Temora zircon standard ( $^{206}\text{Pb}/^{238}\text{U} = 0.0668 [416.8 \pm 0.3 \text{ Ma}]$ ; Black *et al.*, 2003), analyses of which were interspersed with those of unknown zircons, based on the power law relationship  $^{206}\text{Pb}^+/\text{U}^+ = a(\text{UO}^+/\text{U}^+)^2$ . Temora 2 was also used to monitor isobaric interference at the  $^{204}\text{Pb}$  mass peak. The OG1 standard ( $3465.4 \pm 0.6 \text{ Ma}$ ; Stern *et al.*, 2009) was used to monitor  $^{207}\text{Pb}/^{206}\text{Pb}$  reproducibility and accuracy. Data were reduced and analysed using *Squid 2.5* and *Isoplot 3* (Ludwig, 2008, 2009), using decay constants recommended by Steiger and Jäger (1977). Correction for initial or common Pb was made using measured  $^{204}\text{Pb}/^{206}\text{Pb}$  and contemporaneous common-Pb isotopic compositions determined according to the model of Stacey and Kramers (1975). Weighted mean ages are reported with 95% confidence intervals.

The two samples were analysed concurrently during a single session, then additional analyses of 1837679 were obtained during a second session. Eighteen analyses of the Temora standard obtained during the first session indicated a  $^{238}\text{U}/^{206}\text{Pb}^*$  calibration uncertainty of 0.16% ( $1\sigma$ ) with a 1% external spot to spot error assigned. Eleven analyses of OG1 yielded a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of  $3466.0 \pm 2.1 \text{ Ma}$ . Ten analyses of the Temora standard obtained during the second session indicated a  $^{238}\text{U}/^{206}\text{Pb}^*$  calibration uncertainty of 0.49% (1.29% external spot to spot error). Five analyses of OG1 yielded a weighted mean  $^{207}\text{Pb}/^{206}\text{Pb}$  age of:  $3468.2 \pm 4.6 \text{ Ma}$ .

Zircons older than 1100 Ma are reported as  $^{207}\text{Pb}/^{206}\text{Pb}$  ages. For zircons younger than 1100 Ma,  $^{206}\text{Pb}/^{238}\text{U}$  ages have been used as the  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios become too imprecise to be of any use. Additionally, for zircons older than 1100 Ma, the  $^{207}\text{Pb}/^{206}\text{Pb}$  and  $^{206}\text{Pb}/^{238}\text{U}$  ratios can be used to

access the level of concordance of each analysis, and thereby determine the degree to which a given zircon has remained a closed system. For zircons older than 1100 Ma, a cut-off of 10% discordance has been used to exclude poor analyses from age considerations. For zircons younger than 1100 Ma, concordance cannot be properly determined because the  $^{207}\text{Pb}/^{206}\text{Pb}$  ratios are too imprecise.

Therefore, the degree of concordance cannot be used to identify and remove poor analyses. Only two Phanerozoic grains containing high common radiogenic Pb are rejected (47.1.1 and 39.1.1 in sample 1837680 from RD/DD94WB3). U–Pb zircon data are presented in Appendix 3.

### Whole-rock geochemistry method

Approximately 100 mg of sample was weighed into clean 15 mL Savillex Teflon beakers and the mass recorded to four decimal places. Samples were digested using a 1:1 mixture of HF (Merck, Suprapur grade) and Teflon distilled  $\text{HNO}_3$  (Merck) at 120° C for 24 hours, then dried down and repeated. Samples were then digested in a mix of 2 mL HF (Merck Suprapur) and 10 drops  $\text{HClO}_4$  (Merck Suprapur) overnight and dried down gradually to remove fluoride complexes. A further digest in 6N HCL and then 6N  $\text{HNO}_3$  was performed before samples were dissolved in a 2%  $\text{HNO}_3/0.5\%$  HF for analysis.

1:1000 dilutions of each sample were then individually spiked with a 20  $\mu\text{L}$  aliquot of a solution of  $^6\text{Li}$ , As, Rh, In and Bi in 2%  $\text{HNO}_3$ . Samples and standards were analysed on an Agilent 7500cs ICPMS. BCR-2 was used as a calibration standard. Standards BIR-1 and BHVO-2 were also analysed in a 1:1000, 1:2000, and 1:5000 dilution.

Blank was subtracted using the 2%  $\text{HNO}_3/0.5\%$  HF rinse.

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