Looking beneath the Stawell and Bendigo zones in Victoria, Australia: A view through the granite window

J. D. Clemens

Department of Earth Sciences, University of Stellenbosch, Private Bag X1, Matieland 7602, South Africa

jclemens@sun.ac.za

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J. D. Clemens: http://orcid.org/0000-0002-8748-1569

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Supplementary paper

A1 Analytical details

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Sr and Nd Isotope analyses by MC-ICP-MS at the University of Cape Town

For each sample, approximately 50 mg of powder was weighed into a Teflon beaker and about 4 mL 4:1 concentrated 2B HF:HNO₃ was added. The beaker was then capped and left to digest on a hot plate for at least 2 days. The resulting solution was dried and re-dissolved in 6M 2B HNO₃, twice. After the second drying, the sample was taken up in 1.5 mL of 2M 2B HNO₃. After centrifuging, the supernatant was loaded onto a cleaned and pre-conditioned column of 0.2 mL Sr.Spec resin (Eichrom). The column was washed with 2M 2B HNO₃ and the Sr fraction collected in 0.02M 2B HNO₃ (after Míková & Denková, 2007; Pin & Zalduegui, 1997; Pin, Briot, Bassin, & Poitrasson, 1994). Following the final drying, these fractions were re-dissolved in 2 mL of 0.2% HNO₃. Based on the XRF values for Sr concentrations of the original samples, 3 mL of 200 ppb Sr solutions were prepared for analysis using the same 0.2% HNO₃.

Analyses were performed using a NuPlasma HR MC-ICP-MS (Nu Instruments, Wrexham, Wales, UK) in the Africa Earth Observatory Network, EarthLAB Facility in the Department of Geological Sciences at the University of Cape Town. The approximately 200 ppb Sr sample and standard solutions were aspirated into the plasma through a microcyclonic spraychamber. The on-peak background was measured for 120 seconds while aspirating the same 0.2% HNO₃ used to dilute the sample and standard solutions. These background measurements, including any krypton (⁸⁴Kr and ⁸⁶Kr) present in the argon gas, were subtracted from the measured signals. Instrumental mass fractionation was corrected using the exponential law and a fractionation factor based on the measured ⁸⁶Sr/⁸⁸Sr ratio and the accepted value of 0.1194. The ⁸⁷Rb contribution to the 87 amu signal was calculated and subtracted using this fractionation factor, the exponential law, the measured ⁸⁵Rb signal and a ⁸⁵Rb/⁸⁷Rb ratio of 0.3856.

To assess instrument tuning and stability, a 200 ppb Sr solution of the NIST SRM987 international Sr isotope standard was analysed twice, prior to any samples. The external, measured 2 σ reproducibility of SRM987 was 0.000019 (n = 3) on an average ⁸⁷Sr/⁸⁶Sr ratio of 0.710277. All ⁸⁷Sr/⁸⁶Sr data were normalised to 0.710255, the in-house long-term average, which agrees with published results. The average ⁸⁴Sr/⁸⁶Sr ratio was 0.05643 ± 0.00012 (n = 4), also in agreement with published values. Further queries regarding analytical methods should be addressed to AEON Laboratories at the University of Cape Town. Initial ⁸⁷Sr/⁸⁶Sr ratios and their 2 σ uncertainties were calculated from the measured Rb and Sr contents (with 1 σ errors) and measured, present-day ⁸⁷Sr/⁸⁶Sr values (with 2 σ measurement variations) using a Monte Carlo simulation routine. Overall uncertainties on the calculated initial ⁸⁷Sr/⁸⁶Sr ratios also incorporate an age uncertainty of ± 2 Ma.

Nd isotope analyses were carried out using the same multi-collector ICP-MS instrument that was used for the Sr isotope determinations, using the same primary solutions that were used for the Sr isotope work. Nd isotopes were analysed in 1.5 mL of 50 ppb Nd 2% HNO₃ solutions using a Nu Instruments DSN-100 desolvating nebuliser. JNdi-1 was used as bracketing standard, and all Nd isotope data presented are referenced to this standard, using a ¹⁴⁴Nd/¹⁴³Nd ratio of 0.512115 (Tanaka *et al.,* 2000). All Nd isotope data were corrected for Sm and Ce interferences and for instrumental mass fractionation, using the exponential law and a ¹⁴⁶Nd/¹⁴⁴Nd value of 0.7219. For further details of the analytical techniques see Will, Frimmel, Zeh, Le Roux, and Schmädicke (2007). Further queries regarding analytical methods should be addressed to AEON Laboratories at the University of Cape Town. Two-stage, depleted-mantle, Nd model ages were calculated using the methods of Keto and Jakobsen (1987), with present-day depleted mantle values of ¹⁴³Nd/¹⁴⁴Nd = 0.513151 and ¹⁴⁷Nd/¹⁴⁴Nd = 0.2136.

Sr and Nd isotope analysis by ID-TIMS analyses at the University of Melbourne

Other Rb–Sr and Sm–Nd isotope analytical work was carried out at the University of Melbourne, following procedures described in detail by Maas *et al.* (2005). The isotopic data were obtained with a NU Plasma multi-collector ICP-MS coupled to a CETAC Aridus desolvating nebuliser operated at an uptake rate of 40 µL/min. Typical sensitivity in this set-up is in the range of 100–130 V/ppm Sr and Nd. Instrumental mass bias was corrected by normalising to ⁸⁸Sr/⁸⁶Sr = 8.37521 and ¹⁴⁶Nd/¹⁴⁵Nd = 2.0719425 (equivalent to ¹⁴⁶Nd/¹⁴⁴Nd = 0.7219) (Vance & Thirlwall, 2002), using the exponential law

as part of an on-line iterative spike-stripping/internal normalisation procedure. Data are reported relative to the accepted values for the standards: La Jolla ¹⁴³Nd/¹⁴⁴Nd = 0.511860 and SRM987 ⁸⁶Sr/⁸⁷Sr = 0.710230. This secondary normalisation yields the following results for the international standards ($\pm 2\sigma$): BCR-1 ¹⁴³Nd/¹⁴⁴Nd = 0.512641 \pm 18, BHVO-1 = 0.512998 \pm 18, JNdi-1 = 0.512113 \pm 22, and for ⁸⁶Sr/⁸⁷Sr, Sr = 0.708005 \pm 47, BCR-1 = 0.705016 \pm 46, BHVO-1 = 0.703478 \pm 36. These results compare well with data based on TIMS from other laboratories (Maas & McCulloch, 1991; Raczek, Jochum, & Hofmann, 2003; Tanaka *et al.*, 2000). Typical within-run precisions (2Se) are \pm 0.000010 (¹⁴³Nd/¹⁴⁴Nd) and $\leq \pm$ 0.000020 (⁸⁶Sr/⁸⁷Sr), while external precision (reproducibility, 2 σ) based on the results for secondary standards are \pm 0.000020 (Nd) and \pm 0.000040 (Sr). External precision for ⁸⁷Rb/⁸⁶Sr and ¹⁴⁷Sm/¹⁴⁴Nd obtained by isotope dilution is \pm 0.5% and \pm 0.2 %, respectively. Parent/daughter ratios obtained by Q-ICP-MS (Monash University) for unspiked samples have uncertainties of \pm 2% to \pm 3% for ⁸⁷Rb/⁸⁶Sr and \pm 3% to \pm 5% for ¹⁴⁷Sm/¹⁴⁴Nd, respectively.

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