Supplementary Document 1: Analytical Techniques

Electron Microprobe analyses

(MINGHUA please write)

Whole rock major and trace element compositions:

Whole rock geochemical analyses were performed at ALS Geochemistry, Vancouver, BC Canada. Whole rock samples were crushed and pulverized in an agate ring mill to 85% < 75 micron. Major elements (SiO2, Al2O3, TiO2, Fe2O3, MnO, MgO, CaO, Na2O, K2O, P2O5, Cr2O3, BaO and SrO) were determined by Inductively Coupled Plasma - Atomic Emission Spectroscopy (ICP-AES) following lithium metaborate/lithium tetraborate (LiBO2/Li2B4O7) fusion. The resultant melt was digested in 4% HNO3/2% HCl. Loss on ignition was determined by difference in weight after 1 g sample was heated at 1000°C for 1 hour. Trace elements, including Rare Earth elements, were determined by Inductively Coupled Plasma – Mass Spectrometry (ICP-MS) using the same digestion solutions as for the major elements. Total C analyses were performed on samples that were heated in a LECO induction furnace, with analysis of the generated CO2 by infrared detection. Whole rock samples were also analyzed following a four-acid “total” digestion (perchloric, nitric, hydrofluoric and hydrochloric; highly resistant phases such as chromite and zircon may not quantitatively be digested). The resultant fluid was analyzed by ICP-MS and ICP-AES for Ag, As, Be, Bi, Cd, Co, Cs, Cu, Ge, In, Li, Nb, Ni, Pb, S, Sb, Sc, Se, Sn, Ta, Te, Tl, and Zn. Certified reference materials were analyzed as unknowns, with additional CRMs and blanks analyzed. In some cases, analytes are measured by more than one of the techniques above; only data for the most robust method is reported. With only a couple of exceptions, all analytes are within the target range for the CRMs. Blank values are all below the target range.

Sr-Nd-Pb isotopic analyses

Samples were dissolved with concentrated HF for four days at 160°C on the hot plate. Digested samples were dried, taken up in 2N HNO3, and slowly dried at 80°C over night. The nitrates were taken up in 6N HCl and dried. Sr and Nd were separated and purified using ion-exchange chromatography as described in Romer et al. (2005) and Romer and Hahne (2010). Pb was separated using the HBr-HCl ion-exchange procedure described in Romer et al. (2001, 2005).

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| 87Sr/86Sr and 143Nd/144Nd, normalized to 86Sr/88Sr = 0.1194 and 146Nd/144Nd = 0.7219, respectively,  were obtained on a Thermo-Triton mass-spectrometer using dynamic multi-collection.  Analytical uncertainties are given at 2m level. |
| 87Sr/86Sr690 and Nd690 were calculated for the age of the predominant zircon population,  using 87Rb = 1.42E-11 y-1 and  147Sm = 6.54E-12 y-1, (147Sm/144Nd)0CHUR = 0.1967, and  (143Nd/144Nd)0CHUR = 0.512638, respectively, and the concentration data given in Supplementary  Table 2. |
| Lead isotope data were obtained on a Thermo-Triton mass-spectrometer using static  multi-collection and corrected for instrumental mass discrimination with 0.1% / A.M.U.  Accuracy at the 2 level is better than 0.1%. |
| Lead isotope data recalculated to the age of the predominant zircon population, using  contents of Pb, Th, and U from Supplementary Table 2 and decay constants  recommended by the IUGS:  238U = 1.55125E-10 y-1;  235U = 9.848E-10 y-1;   232Th = 4.9475E-11 y-1. |

U-Pb zircon geochronology:

For U-Pb zircon age determinations, zircons were separated using crushing, heavy liquids and magnetic separation. Zircon from the least magnetic fractions were handpicked under a binocular microscope to obtain the most transparent and inclusion-free grains. U-Th-Pb analyses were performed by secondary ion mass spectrometry (SIMS) using a CAMECA IMS-1280 instrument at the Swedish Museum of Natural History, Stockholm, Sweden (Nordsim facility) closely following methods outlined by Whitehouse and Kamber (2005). Zircon grains from the non-magnetic fractions were mounted on glass slides by using double – sided tape, cast in epoxy and polished to expose their zoning. Prior to the analysis the zircon grains were photographed at high magnification and imaged by cathodoluminescence (CL) using a scanning electron microscope. Grain mounts were then washed, rinsed in distilled water, dried in a vacuum oven, and coated with gold. An O-2 primary ion beam of 23 KV with 10 nA O-2 ion was focused on the surface to a ≤ 25 μm diameter spot, and the zircon sputtered under an oxygen flooding to enhance the secondary ion yield of Pb. Measurements were made at mass resolutions of 5000. Pb/U ratios were calibrated using an empirical power law relationship between measured Pb/U and UO2/U ratios. The 1065 Ma Geostandards zircon 91500 (Wiedenbeck et al, 1995), which was used both for Pb/U calibration and U concentration estimates, was analysed frequently during the analytical session. A total of 41 spots were analysed and analytical uncertainties are listed in the Table 1 and plotted as two-sigma error ellipses on concordia diagrams (Tera and Wasserburg, 1972). Ages mentioned in the text are given with two sigma error and MSWD values on concordia ages are those of combined concordance and equivalence. Calculations use the routines of Isoplot (Ludwig, 2001).

Zircon oxygen isotopes:

Oxygen isotope ratios of zircon grains analyzed during this study by SIMS for U-Pb ages, were measured using a CAMECA IMS-1280 instrument at the Swedish Museum of Natural History, Stockholm, Sweden. Analytical procedures follow the scheme of Be'eri-Shlevin et al. (2010). Ion-microprobe δ18O was performed on thirty zircon grains from three samples of the Gabal Atud complex (AT-4, At-9 and AT-40) in the same U-Pb dated spots or in very close proximity. Prior to ion-microprobe δ18O analysis, the U-Pb analysis spots were removed from the zircons by polishing followed by recoating with ~ 30 nm gold. The analytical results are presented in Table 2. Geostandards zircon 91500 (mean = 9.86 ± 0.14; n = 13; MSWD = 0.84) was analyzed frequently during the analytical session. The grains were in very poor shape with a lot of surface relief, so some of this excess spread could result from poor sample surface condition. Quite a few had inclusions too. The extreme value of 10 per mil in the gabbro norite could be on a crack/inclusion.

References

Ludwig, K.R., 2001. Users manual for Isoplot/Ex version 2.05. Berkeley Geochronology Center, Special Publication No 1a, Berkeley, CA, 48 pp.

Romer, R.L. and Hahne, K. (2010) Life of the Rheic Ocean: Scrolling through the the shale record. Gondwana Res., 17: 236-253.

Romer, R.L., Heinrich W., Schröder-Smeibidl, B., Meixner, A., Fischer, C.-O., and Schulz, C., 2005. Elemental dispersion and stable isotope fractionation during reactive fluid-flow and fluid immiscibility in the Bufa del Diente aureole, NE-Mexico: Evidence from radiographies and Li, B, Sr, Nd, and Pb isotope systematics. Contrib. Mineral. Petrol., 149: 400-429.

Romer, R.L., Förster, H.-J., and Breitkreuz, Chr., 2001. Intracontinental extensional magmatism with a subduction fingerprint: the late Carboniferous Halle Volcanic Complex (Germany). Contrib. Mineral. Petrol., 141: 201-221

Tera, F. and Wasserburg, G.J., 1972. U-Th-Pb systematics in three Apollo 14 basalts and the problem of initial Pb in lunar rocks. Earth and Planetary Science Letters, 14, 281-304.

Whitehouse, M.J., Kamber, B.S., 2005. Assigning dates to thin gneissic veins in high-grade metamorphic terranes: A cautionary tale from Akilia, Southwest Greenland. Journal of Petrology 46 (2), 291–318.

Wiedenbeck, M., Allé, P., Corfu, F., Griffin, W.L., Meier, M., Oberli, F., Von Quadt, A., Roddick, J.C., Spiegel, W., 1995. Three natural zircon standards for U-Th-Pb, Lu-Hf, trace element and REE analyses. Geostandards Newsletter 19 (1), 1-23.