DR 2015<mark>138</mark>

Supplimentary information for:

EVIDENCE FOR PROTRACTED HALIP MAGMATISM IN THE CENTRAL SVERDRUP BASIN FROM STRATIGRAPHY, GEOCHRONOLOGY, AND PALEODEPTHS OF SAUCER-SHAPED SILLS

Carol A. Evenchick, William J. Davis, Jean H. Bédard, Nathan Hayward, and Richard M. Friedman

GEOCHEMISTRY

Analytical methods for mineral and glass analyses

Minerals were analyzed using a 5 micron beam at 15kV and 20nA. A 20 s counting time was used for the peak, with 10 s for the background. For glass analyses, conditions were initially set as they were for the mineral analyses. However, count rates for Na₂O and K₂O dropped rapidly during analysis due to volatilization. Six glass analyses were redone using a lower setting of 10 nA and shorter count times of 10 sec to minimize this effect. The two sets of data were compared and regressed one against the other, and the initial set of glass analyses were corrected using this regression. Despite this correction, values of alkalies in glasses probably represent minima. Totals are typically low (82-96%), suggesting high volatile contents. Four types of glass were detected.



Figure DR1. (a) Tb/Yb vs La/Sm and (b) Nb/Th vs Th/Yb, all normalized to Primitive Mantle (PM) of Sun and McDonough (1989). Fields marked JA and JT are alkaline and tholeiitic HALIP data (respectively) from Jowitt et al. (2014). N-MORB and mantle components (black squares)

are also from Jowitt et al. (2014). Black dots are HALIP data from this study. 69 is sample 11EP069A1.

GEOCHRONOLOGY - U-PB ANALYTICAL TECHNIQUES

Analytical Methods - University of British Columbia

Chemical Abrasion Thermal Ionisation Mass spectrometry

CA-TIMS procedures described here are modified from Mundil et al. (2004), Mattinson (2005), and Scoates and Friedman (2008). After rock samples have undergone standard mineral separation procedures zircons are handpicked in alcohol. The clearest, crack- and inclusion-free grains are selected, photographed and then annealed in quartz glass crucibles at 900°C for 60 hours. Annealed grains are transferred into 3.5 mL PFA screwtop beakers, ultrapure HF (up to 50% strength, 500 μ L) and HNO₃ (up to 14 N, 50 μ L) are added and caps are closed finger tight. The beakers are placed in 125 mL PTFE liners (up to four per liner) and about 2 mL HF and 0.2 mL HNO₃ of the same strength as acid within beakers containing samples are added to the liners. The liners are then slid into stainless steel ParrTM high pressure dissolution devices, which are sealed and brought up to a maximum of 200°C for 8-16 hours (typically 175°C for 12 hours). Beakers are removed from liners and zircon is separated from leachate. Zircons are rinsed with >18 M Ω .cm water and subboiled acetone. Then 2 mL of subboiled 6N HCl is added and beakers are set on a hotplate at 80°-130°C for 30 minutes and again rinsed with water and acetone. Masses are estimated from the dimensions (volumes) of grains. Single grains are transferred into clean 300 µL PFA microcapsules (crucibles), and 50 µL 50% HF and 5 µL 14 N HNO3 are added. Each is spiked with a ²³³⁻²³⁵U-²⁰⁵Pb tracer solution (EARTHTIME ET535), capped and again placed in a Parr liner (8-15 microcapsules per liner). HF and nitric acids in a 10:1 ratio, respectively, are added to the liner, which is then placed in a Parr high pressure device and dissolution is achieved at 240°C for 40 hours. The resulting solutions are dried on a hotplate at 130° C, 50 µL 6N HCl is added to microcapsules and fluorides are dissolved in high pressure Parr devices for 12 hours at 210°C. HCl solutions are transferred into clean 7 mL PFA beakers and dried with 2 µL of 0.5 N H₃PO₄. Samples are loaded onto degassed, zone-refined Re filaments in 2 µL of silicic acid emitter (Gerstenberger and Haase, 1997).

Isotopic ratios are measured using a modified single collector VG-54R or 354S (with Sector 54 electronics) thermal ionization mass spectrometer equipped with analogue Daly

photomultipliers. Measurements are done in peak-switching mode on the Daly detector. Analytical blanks are 0.2 pg for U and for Pb up to 2.0 pg. U fractionation was determined directly on individual runs using the ²³³⁻²³⁵U tracer, and Pb isotopic ratios were corrected for fractionation of 0.23%/amu, based on replicate analyses of the NBS-982 Pb reference material and the values recommended by Thirlwall (2000). Data reduction employed the excel based program of Schmitz and Schoene (2007). Standard concordia diagrams were constructed and regression intercepts, weighted averages calculated with Isoplot (Ludwig, 2003). Unless otherwise noted, all errors are quoted at the 2 sigma or 95% level of confidence. Isotopic dates are calculated using the uranium decay constants $\lambda_{238}=1.55125 \times 10^{-10} \text{ y}^{-1}$ and $\lambda_{235}=9.8485 \times 10^{-10} \text{ y}^{-1}$ (Jaffey et al., 1971). EARTHTIME U-Pb synthetic solutions are analysed on an on-going basis to monitor the accuracy of results.

Analytical Methods - Geological Survey of Canada

Heavy mineral concentrates were prepared by standard techniques (crushing, grinding, WilfleyTM table, heavy liquids), and sorted by magnetic susceptibility using a FrantzTM isodynamic separator.

Thermal Ionisation Analyses

Isotopic and U-Pb compositional data were determined by isotope dilution thermal ionization mass spectrometry at the Geochronology laboratory, Geological Survey of Canada. Sample dissolution and chemical methods are described in Parrish et al. (1987). Individual crystals were selected under binocular microscope to avoid inclusions and other imperfections, annealed at 1000°C and leached in HF-HNO3 following procedures modified from Mattinson (2005), spiked with a mixed 205 Pb- 233 U- 235 U tracer solution calibrated to ±0.1% against a gravimetric solution, and dissolved in high-pressure bombs in HF-HNO3. Data reduction and error propagation follow methods outlined in Roddick (1987). U and Pb isotopic ratios were measured using a Triton mass spectrometer operated in either static multi-collection mode or using a secondary electron multiplier and ion counting system. The 205 Pb, 207 Pb, and 208 Pb isotopes were measured simultaneously in Faraday collectors, with 204 Pb in an axial secondary electron multiplier gain was monitored and corrected by peak jumping 205 Pb into the axial cup. A Pb mass fractionation correction of 0.1± 0.04%/amu was applied as determined by replicate analyses of the NBS981 standard. U fractionation was corrected using the 233 U- 235 U double spike and was typically in the range of 0.12%/amu. Deadtime for the ion

counting system was determined by replicate analyses of the NBS982 solution. Accuracy and reproducibility were monitored by analyses of the GSC standard 6266 with a 206 Pb/ 238 U ratio of 0.0963.

SHRIMP Analyses

SHRIMP analytical procedures followed those described by Stern (1997). Briefly, zircons were cast in 2.5 cm diameter epoxy mounts along with fragments of the GSC laboratory standard zircon (z6266, with 206 Pb/ 238 U age = 559 Ma). The mid-sections of the zircons were exposed using 9, 6, and 1 µm diamond compound, and the internal features of the zircons (such as zoning, structures, alteration, etc.) were characterized in back-scattered electron mode (BSE) utilizing a Zeiss Evo 50 scanning electron microscope. Mount surfaces were evaporatively coated with 10 nm of high purity Au. Analyses were conducted using an ¹⁶O⁻ primary beam, projected onto the zircons at 10 kV. The sputtered area used for analysis was ca. 25 µm in diameter with a beam current of ca. 8 nA. The count rates at ten masses including background were sequentially measured over 5 scans with a single electron multiplier and a pulse counting system with deadtime of 23 ns. Off-line data processing was accomplished using SQUID ver 2.2 and utilized the Temora 2 standard with an accepted age of 416.8 Ma (Black et al., 2004). The 1σ external errors of ${}^{206}\text{Pb}/{}^{238}\text{U}$ ratios reported in the data table incorporate a ± 1.3 % error in calibrating the standard zircon. No fractionation correction was applied to the Pb-isotope data; the common Pb correction utilized the Pb composition of the surface blank (Stern, 1997). U concentration was calculated based on a value of 910 ppm for the 6266 standard. Isoplot v. 3.00 (Ludwig, 2003) was used to generate concordia plots and calculate weighted means. The error ellipses on the concordia diagrams, and the weighted mean errors are reported at 2σ .

Analyses of a secondary internal zircon standard (z9910) were interspersed between the sample analyses to verify the accuracy of the U-Pb calibration. This zircon has a 206 Pb/ 238 U age of 441± Ma. Using the calibration defined by the Temora standard, the weighted mean 206 Pb/ 238 U age of thirteen SHRIMP analyses of 9910 zircon is 443.1 ± 4.6 Ma (MSWD = 1.4) within error of the accepted age.

REFERENCES CITED

Black, L.P., Kamo, S.L., Allen, C.M., Davis, D.W., Aleinikoff, J.N., Valley, J.W., Mundil, R., Campbell, I.H., Korsh, R.J., Williams, I.S., and Foudoulis, C., 2004, Improved ²⁰⁶Pb/²³⁸U microprobe geochronology by monitoring of a trace-element-related matrix effect; SHRIMP, ID-TIMS, ELA-ICP-MS and oxygen isotope documentation for a series of zircon standards: Chemical Geology, v. 205, p. 115-140.

- Gerstenberger, H., and Haase, G., 1997, A highly effective emitter substance for mass spectrometric Pb isotope ratio determinations: Chemical Geology, v. 136, p. 309–312.
- Jaffey, A.H., Flynn, K.F., Glendenin, L.E., Bentley, W.C., and Essling, A.M., 1971, Precision measurement of half-lives and specific activities of 235U and 238U: Physical Review C, v. 4, 1889–1906.
- Jowitt, S.M., Williamson, M.-C., and Ernst, R.E., 2014, Geochemistry of the 130 to 80 Ma Canadian High Arctic Large Igneous province (HALIP) event and implications for Ni-Cu-PGE prospectivity: Economic Geology, v. 109, p. 281-307.
- Ludwig, K.R., 2003, User's manual for Isoplot/Ex rev. 3.00: A Geochronological Toolkit for Microsoft Excel: Berkeley Geochronology Center Special Publication 4, Berkeley, CA, 70 p.
- Mattinson, J.M., 2005, Zircon U-Pb chemical abrasion ("CA-TIMS") method: Combined annealing and multi-step partial dissolution analysis for improved precision and accuracy of zircon ages: Chemical Geology, v. 220, p. 47–66.
- Mundil, R., Ludwig, K.R., Metcalfe, I., and Renne, P.R., 2004, Age and Timing of the Permian Mass Extinctions: U/Pb Dating of Closed-System Zircons: Science, v. 305, p. 1760-1763.
- Parrish, R.R., Roddick, J.C., Loveridge, W.D., and Sullivan, R.W., 1987, Uranium-lead analytical techniques at the geochronology laboratory, Geological Survey of Canada, *in*Radiogenic Age and Isotopic Studies: Report 1, Geological Survey of Canada Paper 87-2, p. 3-7.
- Roddick, J.C., 1987, Generalized numerical error analysis with applications to geochronology and thermodynamics: Geochimica et Cosmochimica Acta, v. 51, p. 2129-2135.
- Scoates, J.S., and Friedman, R.M., 2008, Precise age of the platiniferous Merensky Reef, Bushveld Complex, South Africa, by the U-Pb zircon chemical abrasion ID-TIMS technique: Economic Geology, v. 103, p. 465-471.
- Schmitz, M.D., and Schoene, B., 2007, Derivation of isotope ratios, errors, and error correlations for U-Pb geochronology using 205Pb-235U-(233U)-spiked isotope dilution thermal

ionization mass spectrometric data: Geochemistry Geophysics Geosystems, v. 8, Q08006, doi:10.1029/2006GC001492.

- Stern, R.A., 1997, The GSC Sensitive High Resolution Ion Microprobe (SHRIMP): analytical techniques of zircon U-Th-Pb age determinations and performance evaluation: *in* Radiogenic Age and Isotopic Studies, Report 10, Geological Survey of Canada, Current Research 1997-F, p. 1-31.
- Sun, S.S., and McDonough, W.F., 1989, Chemical and isotopic systematics of oceanic basalts:
 Implications for mantle composition and processes, *in* Saunders, A.D., and Norry, M.J., eds.,
 Magmatism in the Ocean Basins: London, Geological Society [London] Special Publication 42, p. 313–345.
- Thirlwall, M.F., 2000, Inter-laboratory and other errors in Pb isotope analyses investigated using a ²⁰⁷Pb–²⁰⁴Pb double spike: Chemical Geology, v. 163 p. 299–322.