## Appendix EA 4-1: Analytical Methods

*Lithogeochemistry of intrusive suites*

Thirty-six samples of least- to highly altered intrusive rock were sent to Activation Laboratories in Ancaster, Ontario for crushing, pulverising, and whole-rock geochemical analysis. The analytical package used was 4Lithoresearch (Fusion XRF and ICP-OES). Lab certificates and QA-QC of the output data indicate no analytical issues for the results presented herein. Estimates of the errors for the major and minor elements are 0.5 to 10 % whereas for the trace elements it is 0.24 to 10 %.

*40Ar/39Ar* *geochronology*

Samples analyzed by the 40Ar/39Ar method at the University of Nevada Las Vegas were wrapped in Al foil and stacked in 6 mm inside diameter sealed fused silica tubes. Individual packets averaged 2 mm thick and neutron fluence monitors (FC-2, Fish Canyon Tuff sanidine) were placed every 5-10 mm along the tube. Synthetic K-glass and optical grade CaF2 were included in the irradiation packages to monitor neutron induced argon interferences from K and Ca. Loaded tubes were packed in an Al container for irradiation. Samples irradiated at the Oregon State TRIGA Reactor, Corvallis, OR were in-core for 9.1 hours in the F-12 position, In-Core Irradiation Tube (ICIT) of the 1 MW TRIGA type reactor. Correction factors for interfering neutron reactions on K and Ca were determined by repeated analysis of K-glass and CaF2 fragments. Measured (40Ar/39Ar)K values were 1.32 (± 4.62%) x 10-1. Ca correction factors were (36Ar/37Ar)Ca = 2.11 (± 0.78%) x 10-4 and (39Ar/37Ar)Ca = 6.32 (± 0.57%) x 10-4. J factors were determined by fusion of 6-10 individual crystals of neutron fluence monitors which gave reproducibility’s of 0.09% to 0.11% at each standard position. Variation in neutron fluence along the 100 mm length of the irradiation tubes was <4%. Matlab curve fit was used to determine J and uncertainty in J at each standard position. No significant neutron fluence gradients were present within individual packets of crystals as indicated by the excellent reproducibility of the single crystal fluence monitor fusions.

Irradiated FC-2 sanidine standards together with CaF2 and K-glass fragments were placed in a Cu sample tray in a high vacuum extraction line and were fused using a 20 W CO2 laser. Sample viewing during laser fusion was by a video camera system and positioning was via a motorized sample stage. Samples analyzed by the furnace step heating method utilized a double vacuum resistance furnace similar to the Staudacher et al. (1978) design. Reactive gases were removed by three GP-50 SAES getters prior to being admitted to a MAP 215-50 mass spectrometer by expansion. The relative volumes of the extraction line and mass spectrometer allow 80% of the gas to be admitted to the mass spectrometer for laser fusion analyses and 76% for furnace heating analyses. Peak intensities were measured using a Balzers electron multiplier by peak hopping through 7 cycles; initial peak heights were determined by linear regression to the time of gas admission. Mass spectrometer discrimination and sensitivity was monitored by repeated analysis of atmospheric argon aliquots from an on-line pipette system. Measured 40Ar/36Ar ratios were 378.16± 0.09% during this work, thus a discrimination correction of 0.7814 (4 AMU) was applied to measured isotope ratios. The sensitivity of the mass spectrometer was ~6 x 10-17 mol mV-1 with the multiplier operated at a gain of 36 over the Faraday. Line blanks averaged 1.40 mV for mass 40 and 0.01 mV for mass 36 for laser fusion analyses and 6.29 mV for mass 40 and 0.02 mV for mass 36 for furnace heating analyses. Discrimination, sensitivity, and blanks were relatively constant over the period of data collection. Computer automated operation of the sample stage, laser, extraction line and mass spectrometer as well as final data reduction and age calculations were done using LabSPEC software written by B. Idleman (Lehigh University). An age of 28.02 Ma (Renne et al., 1998) was used for the Fish Canyon Tuff sanidine fluence monitor in calculating ages for samples.

For 40Ar/39Ar analyses a plateau segment consists of 3 or more contiguous gas fractions having analytically indistinguishable ages (i.e. all plateau steps overlap in age at ± 2σ analytical error) and comprising a significant portion of the total gas released (typically >50%). Total gas (integrated) ages are calculated by weighting by the amount of 39Ar released, whereas plateau ages are weighted by the inverse of the variance. For each sample inverse isochron diagrams are examined to check for the effects of excess argon. Reliable isochrons are based on the MSWD criteria of Wendt and Carl (1991) and, as for plateaus, must comprise contiguous steps and a significant fraction of the total gas released. All analytical data are reported at the confidence level of 1σ (standard deviation).

*U-Pb zircon geochronology: zircon extraction*

Twelve samples of intrusive dikes were sent to Overburden Drilling Management (ODM) Laboratories in Ottawa, Canada. Prior to disaggregation, all rock samples are rinsed and scrubbed with a silicone brush to remove any soil or adhering particles.

Samples are ideally disaggregated to 80-90%, -1 mm by electric-pulse disaggregation (“EPD”), however, the amount disaggregated is dependent on: (a) rock type; (b) grain size; (c) and abundance of sulphides, soluble mineral and graphite. The disaggregation chamber is filled with water and a rock sample is placed on a 1 mm sieve. The rock is subjected to pulses of ~200,000 V of electricity.

EPD is much cleaner than mechanical crushing because the disaggregation takes place in water and creates no dust. The disaggregation chamber is lined with a new sample bag for every sample which, along with thorough cleaning procedures, essentially eliminates the possibility of carry-over. A quartz-vein blank is run between every sample. Normally, 1-2 kg of sample is sufficient for most applications. Larger samples may be required if the targeted mineral is not abundant (i.e. in visible concentrations).

The disaggregated samples are pre-concentrated on a shaking table to produce a low-grade table concentrate. Most zircon grains are very small (<50 µm) and are recovered by micropanning the table concentrate. A zircon-rich pan concentrate is produced at this stage.

To concentrate other minerals, the -1 mm table concentrate is sized and further refined by heavy liquid (methylene iodide) separation in to produce a heavy mineral concentrate (“HMC”). The density of the heavy liquid can be lowered to any specific gravity depending on the targeted mineral. The HMC can be further refined using one or more of the following methods: (a) ferromagnetic separations; (b) electromagnetic separations; (c) sizing (sieving); (d) friction ramp refining; (e) electromagnetic separations; (f) additional heavy liquid separations; and (g) hand-picking of mineral grains.

*U-Pb zircon geochronology: CA-TIMS*

Zircon grains were separated from rocks as described above at ODM labs prior to being sent to Boise State University, Idaho, USA. The zircons were annealed at 900oC for 60 hours in a muffle furnace, and mounted in epoxy and polished until their centres were exposed. Cathodoluminescence (CL) images were obtained with a JEOL JSM-1300 scanning electron microscope and Gatan MiniCL. Zircon was analysed by laser ablation inductively coupled plasma mass spectrometry (LA-ICPMS) using a ThermoElectron X-Series II quadrupole ICPMS and New Wave Research UP-213 Nd:YAG UV (213 nm) laser ablation system. In-house analytical protocols, standard materials, and data reduction software were used for acquisition and calibration of U-Pb dates and a suite of high field strength elements (HFSE) and rare earth elements (REE). Zircon was ablated with a laser spot of 25 µm wide using fluence and pulse rates of 5 J/cm2 and 10 Hz, respectively, during a 45 second analysis (15 sec gas blank, 30 sec ablation) that excavated a pit ~25 µm deep. Ablated material was carried by a 1.2 L/min He gas stream to the nebulizer flow of the plasma. Dwell times were 5 ms for Si and Zr, 200 ms for 49Ti and 207Pb, 80 ms for 206Pb, 40 ms for 202Hg, 204Pb, 208Pb, 232Th, and 238U and 10 ms for all other HFSE and REE. Background count rates for each analyte were obtained prior to each spot analysis and subtracted from the raw count rate for each analyte. Ablations pits that appear to have intersected glass or mineral inclusions were identified based on Ti and P. U-Pb dates from these analyses are considered valid if the U-Pb ratios appear to have been unaffected by the inclusions. Analyses that appear contaminated by common Pb were rejected based on mass 204 being above baseline. For concentration calculations, background-subtracted count rates for each analyte were internally normalized to 29Si and calibrated with respect to NIST SRM-610 and -612 glasses as the primary standards. Temperature was calculated from the Ti-in-zircon thermometer (Watson et al., 2006). Because there are no constraints on the activity of TiO2, an average value in crustal rocks of 0.8 was used.

Data were collected in one experiment in March 2018. For U-Pb and 207Pb/206Pb dates, instrumental fractionation of the background-subtracted ratios was corrected, and dates were calibrated with respect to interspersed measurements of zircon standards and reference materials. The primary standard Plešovice zircon (Sláma et al., 2008) was used to monitor time-dependent instrumental fractionation based on two analyses for every 10 analyses of unknown zircon.

Radiogenic isotope ratio and age error propagation for all analyses includes uncertainty contributions from counting statistics and background subtraction. For groups of analyses that are collectively interpreted from a weighted mean date (i.e., igneous zircon analyses), a weighted mean date is first calculated using Isoplot 3.0 (Ludwig, 2003) using errors on individual dates that do not include a standard calibration uncertainty, and then a standard calibration uncertainty is propagated into the error on the weighted mean date. This uncertainty is the local standard deviation of the polynomial fit to the interspersed primary standard measurements versus time for the time-dependent, relatively large U/Pb fractionation. This uncertainty is 1.4% (2) for 206Pb/238U. Age interpretations are based on 206Pb/238U dates. Errors on the dates are at 2.

U-Pb dates were obtained by the chemical abrasion isotope dilution thermal ionization mass spectrometry (CA-TIMS) method from analyses composed of single zircon grains (Table 1), modified after Mattinson (2005). Zircon was removed from grain mounts after LA-ICPMS analysis. Grains were selected based on LA-ICPMS dates and CL images.

Zircon was put into 3 ml Teflon PFA beakers and loaded into 300 l Teflon PFA microcapsules. Fifteen microcapsules were placed in a large-capacity Parr vessel and the zircon partially dissolved in 120 l of 29 M HF for 12 hours at 190°C. Zircon was returned to 3 ml Teflon PFA beakers, HF was removed, and zircon was immersed in 3.5 M HNO3, ultrasonically cleaned for an hour, and fluxed on a hotplate at 80°C for an hour. The HNO3 was removed and zircon was rinsed twice in ultrapure H2O before being reloaded into the 300 l Teflon PFA microcapsules (rinsed and fluxed in 6 M HCl during sonication and washing of the zircon) and spiked with the Boise State University mixed 233U-235U-205Pb tracer solution. Zircon was dissolved in Parr vessels in 120 l of 29 M HF with a trace of 3.5 M HNO3 at 220°C for 48 hours, dried to fluorides, and re-dissolved in 6 M HCl at 180°C overnight. U and Pb were separated from the zircon matrix using an HCl-based anion-exchange chromatographic procedure (Krogh, 1973), eluted together and dried with 2 µl of 0.05 N H3PO4.

Pb and U were loaded on a single outgassed Re filament in 5 µl of a silica-gel/phosphoric acid mixture (Gerstenberger and Haase, 1997), and U and Pb isotopic measurements made on a GV Isoprobe-T multicollector thermal ionization mass spectrometer equipped with an ion-counting Daly detector. Pb isotopes were measured by peak-jumping all isotopes on the Daly detector for 100 to 160 cycles, and corrected for 0.16 ± 0.03%/a.m.u. (1 sigma error) mass fractionation. Transitory isobaric interferences due to high-molecular weight organics, particularly on 204Pb and 207Pb, disappeared within approximately 60 cycles, while ionization efficiency averaged 104 cps/pg of each Pb isotope. Linearity (to ≥1.4 x 106 cps) and the associated deadtime correction of the Daly detector were determined by analysis of NBS982. Uranium was analyzed as UO2+ ions in static Faraday mode on 1012 ohm resistors for 300 cycles, and corrected for isobaric interference of 233U18O16O on 235U16O16O with an 18O/16O of 0.00206. Ionization efficiency averaged 20 mV/ng of each U isotope. U mass fractionation was corrected using the known 233U/235U ratio of the Boise State University tracer solution.

U-Pb dates and uncertainties were calculated using the algorithms of Schmitz and Schoene (2007), 235U/205Pb of 77.93 and 233U/235U of 1.007066 for the Boise State University tracer solution, U decay constants recommended by Jaffey et al. (1971), and 238U/235U of 137.818 from (Hiess et al., 2012). 206Pb/238U ratios and dates were corrected for initial 230Th disequilibrium using DTh/U = 0.20 ± 0.05 (1) and the algorithms of Crowley et al. (2007), resulting in an increase in the 206Pb/238U dates of ~0.09 Ma. All common Pb in analyses was attributed to laboratory blank and subtracted based on the measured laboratory Pb isotopic composition and associated uncertainty. U blanks are estimated at 0.013 ± 0.009 pg (1).

Weighted mean 206Pb/238U dates were calculated from equivalent dates (probability of fit (pof) >0.05) using Isoplot 3.0 (Ludwig, 2003). Errors on the weighted mean dates are given as ± x / y / z, where x is the internal error based on analytical uncertainties only, including counting statistics, subtraction of tracer solution, and blank and initial common Pb subtraction, y includes the tracer calibration uncertainty propagated in quadrature, and z includes the 238U decay constant uncertainty propagated in quadrature. Internal errors should be considered when comparing our dates with 206Pb/238U dates from other laboratories that used the same Boise State University tracer solution or a tracer solution that was cross-calibrated using EARTHTIME gravimetric standards. Errors including the uncertainty in the tracer calibration should be considered when comparing our dates with those derived from other geochronological methods using the U-Pb decay scheme (e.g., laser ablation ICPMS). Errors including uncertainties in the tracer calibration and 238U decay constant (Jaffey et al., 1971) should be considered when comparing our dates with those derived from other decay schemes (e.g., 40Ar/39Ar, 187Re-187Os). Errors for weighted mean dates and dates from individual grains are given at 2.

*U-Pb zircon geochronology: LA-ICP-MS:*

Zircon grains were separated from rocks as described above at ODM labs prior to being sent to the University of Alberta. Zircons were mounted and subsequently studied under cathodoluminescence (CL) for determination of analysis locations. A New Wave Research Nd: YAG laser ablation system (frequency-quintupled, λ = 213 nm) coupled with a Nu Plasma multiple collector (MC)-ICP-MS was used for in situ analysis.

*Zircon Trace Element Analysis: LA-ICP-MS:*

*Analytical setup*: Zircon U-Th-Pb, trace element (TE), and Hf isotope analyses were conducted at the Mineral Exploration Research Centre isotope Geochemistry Lab (MERC-IGL), at Laurentian University. Laser ablation sampling was performed using a Photon Machines Analyte G2 ArF excimer laser, with 193 nm wavelength, <5 ns pulse width, and HelEx II cell. Hf isotope measurements were conducted using a Thermo Scientific Neptune Plus multicollector (MC) ICP-MS, equipped with Jet interface and nine Faraday cups. All MC-ICP-MS analyses were conducted in low resolution, static mode to ensure maximum sensitivity and stability. U-Pb and TE measurements were conducted using a Thermo Scientific iCap-TQ ICP-MS in single quad mode to ensure maximum sensitivity on the low to intermediate mass range.

U-Th-Pb and TE measurements were conducted simultaneously with Hf isotope analyses on the iCap-TQ and Neptune Plus, respectively, by splitting the ablated aerosol downstream of the sample cell (LASS; e.g. Kylander-Clark et al., 2013). Helium carrier gas flows through the ablation cell were 0.85 l/min (cup) and 0.15 l/min (cell), with 1.1 l/min Ar and 9 ml/min N2 makeup gas added downstream of the cell. Spots with diameter of 40 µm were ablated for 60 seconds, with laser fluence of 6 J/cm2 and 7 Hz repetition rate, leaving estimated ablation pit depths of ~15 µm. Sixty seconds of background were measured at the beginning and end of the analytical session, with 30 seconds of background measured between each ablation.

For U-Pb and trace element analysis on the iCap-TQ, masses measured and associated dwell times are shown in Table X. Cool gas, auxiliary gas, and RF power were set at 14 L/min, 0.8 L/min, and 1550 W, respectively, with Ar makeup gas flows between 0.15 l/min. Parameters associated with Hf isotope measurements on the Neptune Plus are shown in Table EA 4-1X. The cup configuration for Hf isotope analysis on the Neptune Plus is shown in Table EA 4-1Y, and all cups were coupled with 1011  amplifiers. Daly ion beam drift is typically <0.05amu, with flat top peak width typically >0.175 amu, ensuring stable on-peak measurements throughout long-duration analytical sessions. Cool gas flows and RF power were set at 16 L/min and 1200 W, with Ar auxiliary gas flow of 0.8 L/min and makeup gas flow between 0.07 l/min.

*Data Reduction*: The raw data were synchronized in Iolite, along with the laser log file, prior to processing, so that the same timeslices (selections) for each analysis were used in the calculations. Baseline subtraction, instrumental drift, and other corrections were performed with the data reduction schemes (DRS) implemented within Iolite v3.6 (Paton et al., 2010, 2011), and reference materials (RM) shown in Table X. Reference materials were analyzed multiple times at the beginning and end of each session, and once every ten unknowns throughout the session. Three seconds at the beginning and two second at end of the ablation period were excluded from the selections in order to minimize potential fractionation effects, leaving ~55 seconds of signal for integration. Within run variance in the measured ratios for the primary RMs (i.e. the additional percent error required to achieve MSWD = 1) was propagated into the 2SE uncertainty for U-Pb and Hf isotope ratios of all unknowns. Verification RMs were analyzed during each session to ensure accuracy of the data, with values listed in Table X. For Hf isotope analyses, instrumental mass bias and interference correction factors were determined within each session through iterative calculation of the effective 176Yb/173Yb ratio required to yield identical 176Hf/177Hf ratios for MUN1 and MUN3. The effective (mass bias corrected) within session 176Yb/173Yb was then applied in the interference correction for all analyses as part of the DRS.

**Table EA 4-1Y**:

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|  | **L4** | **L3** | **L2** | **L1** | **C** | **H1** | **H2** | **H3** | **H4** |
| **U-Th-Pb** | 204Pb | 206Pb | 207Pb | 208Pb | ~220.65 |  | 232Th |  | 238U |
| **Hf** | 171Yb | 173Yb | 174Hf | 175Lu | 176Yb,Lu/Hf | 177Hf | 178Hf | 179Hf |  |

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