Revision 2

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Petrogenesis of the Kulyk Lake monazite-apatite-Fe(Ti)-oxide occurrence revealed using in-situ LA-(MC)-ICP-MS trace element mapping, U-Pb dating, and Sm-Nd isotope systematics on monazite.

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Abstract

The high-grade metamorphic metasedimentary rocks that comprise the Wollaston Domain, northern Saskatchewan, are host to numerous REE-mineralized pegmatite bodies, including the Kulyk Lake monazite-apatite-Fe(Ti)-oxide occurrence. This occurrence, which is defined by a 3-5cm wide sinuous zone of granoblastic monazite, apatite and titanomagnetite, is enclosed within aplitic monzonite and granitic pegmatite dykes. Monazite in this dyke was studied in detail using *in-situ* laser-ablation inductively-coupled plasma mass spectrometry (LA-ICP-MS) and LA-multi-collector (MC)-ICP-MS. A combination of *in-situ* LA-ICP-MS trace element mapping, trace-element quantification and U-Pb dating were used to identify a significant volume of partial resorbed xenocyrstic monazite and zircon cores within the monazite-apatite-Fe(Ti)-oxide zone. This xenocrystic monazite is locally characterized by anomalously high As, V, Mo, and Eu concentrations and high $(La/Yb)_{CN}$ (i.e., >1000) consistent with their derivation from metalliferous black shales. The U-Pb age distribution of the inherited monazite and zircon populations matches that of the middle- to upper-Wollaston Group sedimentary succession. The latter was confirmed by *in-situ* Sm-Nd isotope systematics measured by LA-MC-ICP-MS that

24   yielded $\varepsilon Nd_{(1830Ma)}$ between -5.0 and -5.7 consistent with derivation from Wollaston Group

25   metasediment. The crystallization of ~1830 Ma anatectic overgrowths on xenocrystic cores is

26   indistinguishable from monazite crystallization in the aplitic monzonite dyke hosting the

27   monazite-apatite-Fe(Ti)-oxide bodies.   This study reveals the potential importance of

28   metalliferous monazite-rich lithologies in the anatectic zone to these pegmatite-hosted REE

29   occurrences and suggests that entrainment and magmatic segregation mechanisms may have

30   helped to concentrate monazite, apatite, and Fe(Ti)-oxide prior to final emplacement of the

31   aplite-pegmatite dykes. Similar processes may have occurred regionally and in other high-grade

32   metamorphic terrains worldwide that are endowed with metalliferous metasedimentary

33   protoliths.

34   Keywords: monazite, LA-ICP-MS, monazite-apatite-Fe(Ti)-oxide, Trans-Hudson, detrital

35   monazite, *in-situ* geochronology

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Introduction

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38   Recent advances in laser ablation inductively coupled plasmas mass spectrometry (LA-ICP-MS)

39   methods applied to *in-situ* analyses for U-Pb geochronology (Petrus and Kamber, 2012), trace-

40   element analysis and trace element mapping (Paton et al., 2011), and Sm-Nd isotope systematics

41   using laser ablation-multi-collector (MC)-ICP-MS (Fisher et al., 2011) allow us to exploit

42   monazite as a geochronometer and petrogenetic tracer at spatial resolutions (~15 μm diameter)

43   nearing the typical excitation volumes of high-voltage electron microbeam microanalytical

44   applications.   The LA-(MC)-ICP-MS technique also allows us to routinely analyze elements

45   present at <10 ppm concentrations and measure radiogenic isotope ratios with high precision for

46   geochronological and isotope tracer investigations.

47   This contribution focuses on the *in-situ* application of these LA-(MC)-ICP-MS

48   techniques to the Kulyk Lake monazite-apatite-Fe(Ti)-oxide occurrence. This occurrence is

49   hosted by upper amphibolite to granulite-facies metasedimentary rocks that comprise the

50   Wollaston Domain, within the central Hearne Province of northern Saskatchewan. The region

51   hosts a variety of U-Th ± REE mineralized veins and pegmatite-hosted dykes. Those associated

52   with granitic dykes, such as the occurrence at Kulyk Lake, are intimately associated with the

53   adjacent pegmatitic to aplitic host rocks and display textures reminiscent of both hydrothermal

54   deposition and igneous crystallization. As a result, the timing of crystallization of these unusual

55   lithologies relative to their pegmatite host, geochemical trends diagnostic of magmatic or

56   hydrothermal deposition processes, and the isotope systematics relevant to reconstructing

57   possible source rocks for these bodies needs to be reconciled with competing models for their

58   origin. Monazite is well-suited to answer these questions because of its robustness as a U-Pb

59   geochronometer (Alagna et al., 2008; Aleinikoff et al., 2006; Bosch et al., 2002; Cherniak et al.,

60   2004; Fitzsimons et al., 1997; Forbes et al., 2007; McFarlane and Harrison, 2006) and Nd isotope

61   tracer (Fisher et al., 2011).   Monazite can also crystallize from REE-saturated anatectic melts

62   (Kelsey et al., 2008; Montel, 1993) and can undergo solid-state hydrothermal alteration and

63   recrystallization leading to resetting of U-Pb ages (Seydoux-Guillame et al., 2002; Williams et

64   al., 2011).   The trace element chemistry, internal zoning, and stable isotope systematics of

65   monazite may also be used to reveal details of monazite petrogenesis over a broad range of

66   metamorphic *P-T* conditions (Ayers et al., 2006; Breecker and Sharp, 2007; Williams et al.,

67   2007).   Integrating textural, geochemical and isotope datasets for monazite can, therefore, help

68   build petrogenetic models to explain the origin of unusual phosphatic rocks and the LA-(MC)-

69   ICP-MS methodologies applied here are particularly well-suited to this goal.

70   **Regional geology**

71   The Wollaston Domain (Figure 1) is one of several tectonic blocks that comprise the Hearne

72   Province of the Churchill Craton in Saskatchewan (Bickford et al., 1990; Hoffman, 1989;

73   Hoffman, 1990; Lewry and Collerson, 1990).   It is comprised of a northeast-trending, tightly

74   folded linear belt containing Archean granitoid rocks preserved in structural domes, and

75   unconformably overlying Paleoproterozoic metasedimentary rocks that were deformed and

76   metamorphosed during the Trans Hudson Orogeny (Lewry and Sibbald, 1980; Tran and Yeo,

77   1997).   Partial melting during granulite-facies metamorphism of the Wollaston Group

78   supracrustal rocks produced migmatitic paragneisses and anatectic granitic rocks that are

79   constrained to the latter stages of Trans-Hudson deformation (Annesley et al., 2005; Bickford et

80   al., 1990; Bickford et al., 2005; Chiarenzelli, 1989; Chiarenzelli et al., 1998; Yeo and Delaney,

81   2007).

82   The Wollaston Group supracrustal rocks in the Kulyk Lake region overly Archean

83   granites that range between 2650 Ma and 2500 Ma based on U-Pb zircon thermal ionization

84   mass spectrometry (TIMS) analyses (Annesley et al., 1992; Annesley et al., 2005; Ray and

85   Wanless, 1980; Rayner et al., 2005). A detailed study by Tran et al. (2010) described the

86   regional volcano-sedimentary stratigraphy of the Wollaston Group and established the relative

87   contributions from Archean and Paleoproterozoic detritus using whole-rock Sm-Nd data and

88   detrital zircon analyses. The metasdimentary cover at Kulyk Lake (McKeough et al., 2010) is

89   described as a Paleoproterozoic syn-rift passive margin and foreland sequences (Tran et al.,

90   2003; Tran et al., 2008): they are divided into Lower and Upper subgroups that are separated by

91   a regional unconformity. The lower succession includes a basal graphitic pelitic unit overlain by,

92   and in part interleaved with, arkose, conglomerate, quartzite, and calcareous sedimentary rocks.

93   The upper part of the succession includes conglomerates, immature arkosic rocks and calcsilicate

94   rocks and is thought to represent subsequent infilling of a foreland basin. The provenance of

95   these metasedimentary sequences falls into three main zircon populations including: 1) >2400

96   Ma grains that represent detritus shed from the neighbouring Hearne craton; 2) 2100 Ma

97   components thought to represent reworking of syn-rift volcanic rocks, and; 3) 1920-1880 Ma

98   detritus shed from advancing volcanic arc terrains of the western Churchill Province (Su et al.,

99   2012). The lower succession of the Wollaston Group appears to have detritus sourced primarily

100   from Archaean protoliths (2600 to 2850 Ma) with locally minor Paleoproterozoic (~2400 Ma)

101   input.   In contrast, the middle and upper successions contain significant Paleoproterozoic

102   components (2100 to 1880 Ma) and only minor Archean detritus (Hu et al., 2011).

103   The Paleoproterozoic metasedimentary gneisses are intruded by north-, east- and east-

104   northeast-trending, variably radioactive leucogranite dykes, sills and lenses (Mawdsley, 1957;

105   McKeough and Lentz, 2011; McKeough et al., 2010).   U-Th-REE mineralization in the Kulyk

106   Lake area is primarily fracture-controlled within prospective folded and faulted rocks of the

107   basal Wollaston sequences, particularly in the leucogranitic pegmatite dykes that intrude the

108   metasedimentary rocks and locally along the contacts between the host gneisses and pegmatite

109   intrusions.   Previous geochronological studies on syn-tectonic diatexite, amphibolite, and

110   numerous foliated and cross-cutting 'Hudsonian' granite plutons that intrude the Wollaston

111   group reveal a prolonged history of Trans-Hudson metamorphism, anatexis, and pegmatite and

112   aplite emplacement between ~1840 and ~1770 Ma (Hou et al., 2012).   The onset of partial

113   melting under granulite-facies conditions and the generation of restitic diatexitic gneisses

114   occurred between 1840 to 1830 Ma in response to crustal thickening in the domain (Annesley et

115   al., 2005).   Emplacement of Hudsonian granitioid rocks peaked between 1830 and 1815 Ma

116   following the initiation of exhumation and decompressional melting.   The final transpressional

117   stages of Trans-Hudson orogenesis involved strike-slip deformation, emplacement of

118   undeformed 1780-1760 Ma granitic pegmatite bodies that cross-cut the regional gneissic fabrics,

119   followed by rapid cooling.   The Wollaston Group rocks were near or at the surface by 1650 Ma

120   when mature quartz-rich sediments at the base of the Athabasca basin were deposited (Hou et al.,

121   2012).

Description of the occurrence
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124   The Kulyk Lake monazite-apatite-Fe(Ti)-oxide occurrence was first described by Watkinson &

125   Mainwaring (1976). The deposit occurs within northeast-striking and steeply southeast dipping

126   upper amphibolite- to granulite-facies calc-silicate paragniesses with assemblages comprised of

127   biotite-hornblende-clinopyroxene-quartz-K-feldspar-plagioclase with variable biotite/hornblende

128   and local magnetite-bearing zones. Psammitic gneisses to the east and west of the calc-silicate

129   gneiss hosting the deposit also contain abundant concordant and locally cross-cutting pegmatitic

130   bodies and granitic sheets.

131   The area of granitoid dykes hosting the monazite-apatite-Fe(Ti)-oxide zone is located

132   ~65 m east of Kulyk Lake (Figure 1b). This zone of granitoid dykes is sub-vertical and ~15 m in

133   length, locally extending as lenses discordant to the north-northeast strike of the regional fabric

134   observed in the adjacent calc-silicate gneisses. The host intrusive unit ranges from granitic

135   pegmatite to monzonitic aplite, with the monazite-apatite-Fe(Ti)-oxide mineralization distributed

136   primarily within the 10-15cm wide aplitic phase of the dyke system. The aplitic monzonite

137   displays a seriate to locally porphyritic texture with a hypiodiomorphic granular groundmass of

138   0.2 to 0.5mm perthitic K-feldspar, plagioclase, and minor (<5%) interstitial quartz. Grain

139   boundaries in the groundmass are sutured and locally blurry and are typical of dynamically-

140   recrystallized rocks. Monazite, apatite, and Fe(Ti)-oxide are ubiquitous accessory minerals in

141   the aplitic monzonite and together comprise ~1% modal abundance in thin section. The Fe(Ti)-

142   oxide mineral in the monzonite displays micron-scale exsolution lamellae of rutile and ilmenite.

143   Larger 2 to 4 mm phenocrysts of perthitic K-feldspar host inclusions of subhedral to oval

144   monazite, euhedral to subhedral apatite, and ovoid Fe(Ti)-oxide.

145   The phosphate-rich zone was emplaced along the central part of the host monzonite aplite

146   dyke, and forms concordant but locally tortuous contacts with it (Figure 2a). It is comprised of

147   0.5 to 5cm thick bands of granoblastic fine- to medium-grained reddish brown monazite, pinkish

148   to beige apatite, and an Fe(Ti)-oxide component of medium- to coarse-grained titanomagnetite.

149   The titanomagnetite is typically disseminated throughout the monazite-apatite bands, but may

150   also form as a thick outer rind surrounding monazite-apatite in sharp contact with the aplitic

151 phase of the granitic dyke. The phosphate minerals comprise 70 to 90% of these zones. It is

152 important to note that quartz is absent in the phosphatic zones and only trace amounts of feldspar

153 have been encountered. The monazite-apatite-Fe(Ti)-oxide zone also locally forms lobate

154 embayments or injections into the adjacent monzonitic aplite dyke giving rise to textures

155 reminiscent of magma mingling. These lobate domains are characterized by stromatic Fe(Ti)-

156 oxide layers rimmed by narrow monazite-rich selvages that separate an apatite-rich inner zone

157 from the host aplite dyke. In other parts of the monazite-apatite-Fe(Ti)-oxide occurrence,

158 alternating monazite-rich and apatite-rich bands and schlieren give the zone a layered appearance

159 with no obvious differentiation (Figure 2b). A reddish earthy hematite alteration affected the

160 monazite-apatite-Fe(Ti)-oxide occurrence and the adjacent aplitic and pegmatitic wall rocks.

161   McKeough & Lentz (2011) have also shown that the aplitic monzonite dyke hosting the

162 phosphate zone is also enriched in Nb, Y, REE, U, Th and Zr (NYF-type) albeit at lower

163 absolute concentrations. The monazite-rich zone and its host monzonite dyke also display

164 similar trace element fractionation trends including sub-parallel Chondrite-normalized REE

165 fractionation patterns.

Methodologies

Sampling

168 Samples of the granitic host rocks and the monazite-apatite-Fe(Ti)-oxide mineralization were

169 obtained by channel sampling along two transect through the occurrence using a portable

170 diamond saw. The study focuses on channel samples of: 1) layered monazite-apatite-Fe(Ti)-

171 oxide rocks from the southern (MMKJ037) and northern (MMKJ006) ends of the occurrence,

172 and 2) a fine-grained granoblastic monzonitic aplite dyke (DFKJR006) immediately adjacent to

173   the phosphatic zone at the northern end of the occurrence. Other sample locations shown in

174   Figure 2 are part of a larger regional study by McKeough et al. (2010). The bulk of the

175   analytical work described below was carried out on normal-thickness (~30 μm) polished thin

176   sections.

Electron microscopy

178   After optical imaging in transmitted and reflected light using a Zeiss AxioImager A1M

179   polarizing microscope, monazite was imaged and analyzed using a JEOL6400 scanning electron

180   microscope (SEM) equipped with an Oxford Prism PGT energy dispersive spectrometer (EDS)

181   and a Gatan Chroma-CL cathodoluminescence system. Backscattered electron (BSE) and

182   cathodoluminescence (CL) images were acquired at 10-15 nA and 15kV with a focused beam.

183   Major element data for monazite was collected on a JEOL733 electron probe microanalyzer

184   (EPMA) equipped with four wavelength dispersive spectrometers (WDS) and Geller dQuant

185   automation software. The analyses were performed at 15kV and 30nA and synthetic $REEPO_4$

186   glasses used as primary standards for the REE.

LA-ICP-MS

188   All analyses were carried out using a Resonetics M-50 193nm ArF (excimer) laser ablation

189   system attached to an Agilent 7700x quadrupole ICP-MS with two external rotary pumps and

190   using Iolite™ Trace Elements Data Reduction Scheme (Paton et al., 2011) and VizualAge™ for

191   U-Pb geochronology (Petrus and Kamber, 2012).

192   For trace element mapping, the laser ablation software (Geostar v. 6.23) was used to

193   define a rectangular area within which a number of linear raster scan were defined. Each raster

194   line of the pattern was programmed to run with a crater size of 17 μm, a scan speed of 8 μm/sec,

195   and a laser pulse rate of 10 Hz. The fluence on target was regulated at 4J/cm^2 and the inline

196   smoothing device (the 'squid') between the laser and ICP-MS was removed to ensure fastest

197   possible washout from the cell. Ablated aerosol was transported out of the cell using 750 mL/min

198   He and was combined downstream with a 750 mL/min Ar carrier gas flow. A background delay

199   of 20sec was for used for each raster line. Concentration standards (NIST610) and quality

200   control standards (GSE-1G) were ablated before and after the map pattern using the same

201   analytical conditions. A typical trace element map acquisition defined in this way took between

202   30-40 minutes to run. The ICP-MS was tuned using NIST610 in order to maximize sensitivity

203   while also maintaining oxide production (monitored as $^{248}ThO^+/^{232}Th^+$ <0.3%), doubly-charged

204   ions (measured as $^{22}M^+/^{44}Ca^+$), and $^{238}U^+/^{232}Th^+ \approx 1.05$. The latter is a measure of plasma

205   robustness. The number of analytes and their dwell times were selected to ensure that the total

206   ICP-MS sweep time was <0.5 sec. Individual analyte dwell times were assigned to be

207   approximately inversely proportional to the concentration in the target grain. High concentration

208   elements (P, LREE, Th) were set with 0.01 sec dwell times whereas lower concentration

209   elements were set with 0.02 to 0.05 sec dwell times. The total ICP-MS acquisition time was set

210   to correspond to the total time to run through the predefined laser ablation sequence and the two

211   instruments were started simultaneously. At the end of the acquisition, the Geostar laser log file

212   and the Agilent time series data (cps versus time) were combined offline using the Iolite

213   automatic integrations function and the data reduced. The independently (electron microprobe)

214   measured concentration (wt%) of Nd in the monazite target was used as an internal standard.

215   Trace element data for Kulyk Lake monazite was reduced assuming a Nd concentration of

216   9.0wt%. Data for Trebilcock and 44069 monazite were analyzed assuming Nd concentrations of

217   10wt% and 9.8wt% respectively. Quality of the analytical method and data reduction scheme

218   was checked by comparing the measured/accepted values obtained for GSE-1G standard (**Table**

219   **1**). Absolute values and detection limits for As are anomalously high and are probably related to

220   a wide variety of argide interferences (e.g., $^{36}Ar^{38}Ar^{1}H^{+}$, $^{40}Ar^{35}Cl^{+}$, $^{36}Ar^{39}K^{+}$) present in the gas

221   background and produced during ablation.   As a result, As concentrations were adjusted

222   downward by ~10% (As* in Tables 3, 4, 5, and 6) to match the upper error limit in GSE-1G (350

223   ppm).   The trace element maps presented in Figures 6 and 10 show semi-quantitative

224   concentrations that are calculated without using an internal standard but instead are based on the

225   analyte intensity in the unknown compared to its intensity in NIST610.   The semi-quantitative

226   concentrations are typically within 20-30% of the true value and so serve as a reasonable

227   approximation for visualizing different compositional domains.

228   For U-Pb geochronology, the ICP-MS was first tuned using NIST610 to obtain oxide

229   production and U/Th values described above whereas doubly-charged ion production was not

230   strictly monitored. Monazite U-Pb geochronology by LA-ICP-MS was carried out using 14, 17

231   or 26 μm diameter craters depending on the size of the compositional zoning domain targeted for

232   analysis.   Ablation was carried out at 4 Hz and 3 J/cm^2 with a 30 sec background and 30 sec

233   ablation. External standardization to correct for laser-induced Pb/U fractionation and mass-bias

234   in the ICP-MS was accomplished by replicate ($n \geq 15$) measurements of either 44069 (425 Ma)

235   or Trebilcock (272 Ma) monazite.   The 44069 standard was used primarily for the lowest U

236   concentration domains encountered in the monazite-apatite-ilmenite dyke owing to the better

237   matrix matching compared to the much higher-U Trebilcock standard. Analyses of NIST610 at

238   the start and end of the ablation sequence were additionally used as concentration standards. The

239   ICP-MS was tuned, using a raster scan on NIST610, to achieve maximum sensitivity for ^{207}Pb by

240   adding 2.8 mL/min N$_2$ to the He + Ar carrier gas and by using the second external rotary pump

241   on the ICP-MS. The analytes and dwell times (in milliseconds) chosen were: ^{31}P (10), ^{146}Nd

242   (10), ^{204}Pb(80), ^{206}Pb (30), ^{207}Pb (80), ^{208}Pb (10), ^{232}Th (10) and ^{238}U (20). No significant ^{204}Pb

243   above the gas blank of ca. 160cps was encountered and so common-Pb corrections are

244   considered to be negligible. The data were processed offline using Iolite™ and VizualAge™.

245   When necessary the data were also corrected for common-Pb using the correction routine

246   described by Andersen (2002) and implemented in VizualAge™ which avoids inaccuracies

247   associated with measuring low concentrations of ^{204}Pb by LA-ICP-MS (Storey et al., 2006).

248   Common-Pb corrected data are highlighted in Table 7. Data quality was confirmed by

249   measuring replicate analyses of one or more in-house standards such as Thompson Mine-2

250   monazite (1765 Ma).

LA-MC-ICP-MS for Sm-Nd

252   Following optical microscopy and backscattered electron imaging to identify suitable targets, 1

253   cm diameter round target areas were drilled out of a 60μm thick thin section and mounted in

254   epoxy along with LREE-glass standards described by Fisher et al. (2011) to correct for Sm/Nd

255   fractionation. Measurements were carried out at the Memorial University of Newfoundland

256   CREAiT facility on a GeoLas 193nm laser ablation system connected to a Thermo Neptune MC-

257   ICP-MS. The outermost portions of large monazite grains were analyzed using a 45 μm

258   diameter crater at a repetition rate of 5 Hz and fluence of approximately 5 J/cm^2. Replicate

259   analyses of LREE-glass ($n = 4$) and Trebilcock monazite ($n = 6$) were used to correct for Sm/Nd

260   bias and to verify that the measured ^{143}Nd/^{144}Nd and ^{145}Nd/^{144}Nd values for the standard were

261   within error of the true values. The data were processed offline using an in-house data reduction

262   macro (MCTool-Nd) that was modified from LAMTool-Hf written by J. Kosler, University of

263   Bergen. Corrections for instrumental mass-bias followed the methodology outlined in Fisher et

264   al. (2011). Initial εNd(t) values were calculated on the basis of measured U-Pb ages.

Results

Petrographic relationships

267   The monazite-apatite-Fe(Ti)-oxide zones display an internal differentiation, with interleaving

268   monazite-rich and apatite-rich bands (Figure 2b). Monazite-rich areas occur as lenses and

269   stringers with a granoblastic interlocking texture and grains sizes between 0.1 to 2 mm. In thin

270   section, boundaries between monazite masses and neighbouring apatite-rich layers are typically

271   sharp, but locally show a gradational transition (Figure 2c). Whereas apatite and monazite form

272   discrete layers, titanomagnetite occurs either as elongate cm-scale clots or as weakly foliated

273   xenomorphic grains interstitial to monazite and apatite (Figure 2c and 3). Grain boundaries

274   between adjacent monazite and apatite are typically straight to curvilinear and well defined

275   triple-junctions exist between the major phases in the rock. Whereas monazite is subhedral to

276   xenomorphic, apatite locally forms euhedral to subhedral hexagonal grains enclosing zircon

277   grains that display core-overgrowth textures (Figure 3a). Radial fractures emanating from zircon

278   inclusions in apatite and titanomagnetite are abundant and this texture is typically associated

279   with volume expansion during metamictization of zircon. Titanomagnetite defines a weak

280   alignment and displays fine (1 to 5 μm wide) exsolution lamellae of ilmenite and rutile. Apatite

281   locally occurs as inclusions in large ilmenite grains whereas monazite inclusions in ilmenite have

282   not been observed. Similarly, apatite inclusions within monazite are rare.

283   Accessory zircon is abundant and is observed primarily along grain boundaries and triple

284   junctions between monazite, apatite and titanomagnetite in addition to being included in every

285   major mineral in the monazite-apatite-Fe(Ti)-oxide zones. Zircon occurs as sub-rounded equant

286   grains up to 75μm in diameter most of which display a thin 10-20 μm rim that is visible in

287   reflected light, BSE, and CL images (Figure 4).

Major and trace element zoning and chemistry

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289 A combination of BSE and CL imaging were used to examine internal zoning features in

290 monazite, apatite, and titanomagnetite within the mineralized zones. Apatite displays faint

291 patchy zoning in BSE images but no systematic zoning is obvious. Detailed CL imaging of

292 apatite did reveal the presence of numerous darker-CL healed fractures and a cryptic network of

293 dark CL domains that concentrated around zircon grains (Figure 3b). In contrast, backscattered-

294 electron images (Figure 5) of monazite-rich layers reveal at least three compositional domains:

295 1) rare bright-BSE cores surrounded by; 2) darker-BSE oscillatory-zoned domains, and 3)

296 uniform medium-BSE overgrowths that form the interlocking texture noted earlier.

297 The EPMA was used to analyze each of these compositional domains and establish major

298 element monazite compositions that could be used as internal standards for subsequent LA-ICP-

299 MS analyses (Table 2). The LREE (La, Ce, Pr, Nd) abundances uniform and independent of the

300 compositional zoning revealed by BSE imaging. The bulk of the variation in BSE brightness is

301 attributable to the Th content of each domain which varies from 3.3wt% Th in the bright-BSE

302 inner cores to 2.8wt% in dark-BSE areas of oscillatory-zoned domains to 4.3% in the uniform

303 overgrowths. Uranium concentrations measured by electron microprobe were typically below

304 detection limit (<0.02wt%) under the analytical conditions employed for EPMA major-element

305 analyses.

306 Three LA-ICP-MS maps were constructed to help guide the location of *in-situ* U-Pb

307 geochronology measurements. These maps (Figure 6) not only revealed the presence of low-U

308 cores that correspond to the dark-BSE domains in Figure 5, they also show that within these core

309 domains exist partially-resorbed monazite characterized by anomalously high V, As, and Mo as

310   well as distinct $(La/Yb)_{CN}$ and Th/U. Based on the LA-ICP-MS maps it is, therefore, possible to

311   further sub-divide the cores into several sub-domains based on their trace element composition.

312   Once the LA-ICP-MS mapping session was complete, the thin sections were removed

313   from the laser ablation cell and re-polished using 3μm diamond paste to remove the bulk of the

314   surface damage induced by the laser raster pattern used to generate the trace element maps. The

315   samples were reloaded into the cell and images showing either the As or V concentration were

316   overlain on the reflected light image of the target grain captured by the laser ablation software.

317   This allowed specific compositional domains revealed in the LA-ICP-MS maps to be targeted for

318   trace element spot analyses using 13μm craters. Figure 7 shows how an overlay showing the

319   distribution of V in monazite grain 2 was used to carefully position the location of craters used to

320   characterize the compositional variability of monazite and corresponding U-Pb ages.

321   The results of the LA-ICP-MS spot analyses are shown in Tables 3, 4, and 5. For

322   comparison, two widely-used monazite standards, Trebilcock monazite (Tomascak et al., 1998)

323   and 44069 (Aleinikoff et al., 2006) were also analyzed for comparison and the results are given

324   in Table 6. Data for the Chondrite-normalized (McDonough and Sun, 1995) REE show only

325   minor variations among the different compositional domains identified.   Taken as a group,

326   monazite from the phosphate-Fe(Ti)-oxide zones displays strongly-fractionated REE patterns

327   (Figure 8) with $(La/Yb)_{CN}$ ranging from 1000 to 1400 compared to ~125 and ~200 for Trebilcock

328   and 44069 respectively. Kulyk Lake monazite also contains ~1000 ppm Eu and displays a small

329   (0.25) negative Eu/Eu* anomaly. The LA-ICP-MS data also confirm the low concentrations of

330   both U (75-350 ppm) and Th (2.2 to 5.5 wt%). The domains containing elevated V, As, and Mo

331   display concentrations in the 100-350 ppm range for V, 1200-1750 ppm for As, and 4 to 9 ppm

332   Mo. These domains also contain low and variable amounts of Ba and Nb. For comparison, the

333   pegmatite-hosted Trebilcock monazite does not contain V, Mo, Nb, and Ba above detection

334   limits (1.4, 0.34, 0.18 and 0.21 ppm respectively) and has ~589 ppm As. The 44069 standard

335   does contain measureable V (2.8 ppm) and has a somewhat lower As content of ~470 ppm.

U-Pb geochronology

337   Results of *in-situ* U-Pb dating of different compositional domains in monazite revealed by BSE

338   imaging and mapped by LA-ICP-MS in grains 1, 2, and 3 as well as data for the host aplitic

339   monzonite are presented in Table 7. The partially resorbed domains characterized by elevated

340   As, V, and Mo define an array of near-concordant data with $^{207}Pb/^{206}Pb$ ages ranging from ~1870

341   to ~2000 Ma with significant populations at ~1880 Ma and 1975 Ma (Figure 9 probability

342   density).

343   The measured $^{207}Pb/^{206}Pb$ ages are strongly correlated with compositional zoning in the

344   monazite grains for which LA-ICP-MS trace element maps were constructed. Figure 10 shows

345   the V, Th/U and Mo semi-quantitative compositional maps for monazite grain 3 as well as the

346    $^{207}Pb/^{206}Pb$ age. Domains with elevated Mo up to 10 ppm and intermediate Th/U gave somewhat

347   near-concordant common-Pb corrected data with $^{207}Pb/^{206}Pb$ dates in the range of 1876 to 1920

348   Ma. The compositional domain characterized by the lowest Th/U value display a similarly

349   narrow range of near-concordant data with $^{207}Pb/^{206}Pb$ dates between 1870 to 1897 Ma. This

350   grain contains a distinct core characterized by low V, Mo and U as well as distinctly higher Th/U

351   compared the other compositional domains. Two craters on this domain produced discordant U-

352   Pb data with common-Pb corrected $^{207}Pb/^{206}Pb$ dates of ~1850 Ma suggesting it my represent an

353   embayment of younger overgrowth material.

354   The results for the uniform-BSE overgrowths (Figure 11a and 11b) also display a

355   spectrum of near-concordant ages ranging from ~1770 to ~1850 Ma with two populations of

356   overlapping data at 1768 ± 10 Ma (n = 5) and 1838 ± 10 Ma (n = 7).

357   The host monzonitic aplite dyke to the monazite-apatite-Fe(Ti)-oxide occurrence also

358   contains abundant and large monazite grains with similar core-overgrowth relationships as

359   imaged by BSE. In order to constrain the crystallization age of the aplite dyke, uniform-BSE

360   overgrowths were specifically targeted to avoid mixing between older inherited components.

361   The results of this dating, shown in Figure 11c and 11d are identical within error to those

362   obtained for the monazite within the mineralized zone: a cluster of overlapping dates at 1787 ± 8

363   Ma (n = 4) and another at 1827 ± 8 Ma (n = 4).

364   Zircon cores and overgrowths were also dated by LA-ICP-MS to help identify their

365   origin. The results of zircon dating, compiled in Table 8, reveal a similarly wide spectrum of

366    $^{207}Pb/^{206}Pb$ ages ranging from ~1800 to 2350 Ma with populations at 1895 Ma and 1975 Ma.

367   Higher reflectance zircon overgrowths shown in Figure 4 are not obviously younger. Rather,

368   these overgrowths and the cores they surround are both locally reset to ~1800 Ma.

369   **Sm-Nd isotope systematics**

370   The uniform overgrowths on Kulyk Lake monazite have uniform $^{143}Nd/^{144}Nd$ as measured by

371   LA-MC-ICP-MS with εNd(t) values calculated at 1830 ± 10 Ma between -5.0 and -5.7 (Table 9).

372   There is very little intergranular variation in $^{147}Sm/^{144}Nd$ and depleted mantle model ages (T_{DM})

373   average 2400 Ma.

Discussion

Timing of and temperature of emplacement

376 Both the monazite-apatite-Fe(Ti)-oxide occurrence and the adjacent aplitic dyke contain

377 monazite overgrowths with statistically indistinguishable U-Pb monazite ages. In both rocks, the

378 age of crystallization of compositionally uniform overgrowths is 1830 ± 5 Ma. This date falls in

379 the range of estimates for the timing of granulite-facies metamorphism in the region related to

380 Trans-Hudson crustal thickening and orogenesis (Annesley et al., 2005).   This timing of

381 crystallization is consistent with the oxygen-isotope temperature estimates of >850°C described

382 by McKeough (2013) for monazite-apatite and monazite-titanomagnetite fractionation.   This

383 high temperature estimate is also consistent with the presence of exsolved ilmenite and rutile in

384 the Fe(Ti)-oxide phase.   The discordant nature of the host granitic dykes with respect to the

385 regional gneissic fabric shown in Figure 2 would reflect emplacement of anatectic melts derived

386 from deeper levels into syn-kinematic dilatational jogs developed at higher structural levels (e.g.,

387 Marchildon and Brown, 2003; Solar et al., 1998). The younger 1770 ± 10 Ma population in both

388 rocks matches the timing of pegmatite emplacement associated with the late stages of isothermal

389 decompression and transpressive deformation at amphibolite-facies conditions (Annesley et al.,

390 2005; Hou et al., 2012). The localized resetting of U-Pb ages measured in monazite and zircon is

391 most likely associated with this post-peak metamorphic reworking. Evidence for the localized

392 nature of this overprint may be recorded by the cryptic dark-CL network in apatite that suggests

393 these zones were locally recrystallized in the vicinity of zircon and along fractures that were

394 subsequent healed. Hence, it appears that whereas the monazite overgrowths in the monazite-

395 apatite-Fe(Ti)-oxide zones and the adjacent aplitic monzonite co-crystallized at 1830 ± 5 Ma, the

396 entire sequence was later affected by dynamic recrystallization and localized Pb-loss along a

397 cryptic fracture-controlled network.

398 **Significance of As, V, Mo in inherited cores**

399   A large volume fraction of the monazite in the phosphate-Fe(Ti)-oxide zone is inherited from the

400   Wollaston Group metasediments. The *in-situ* Sm-Nd isotope systematics measured on 1830 ± 5

401   monazite domains have $\varepsilon Nd_{(1830)}$ ranging from -4.3 ± 0.8 to -5.0 ± 0.8 and these fall within the

402   range of wholerock $\varepsilon Nd_{(1830)}$ values (-4.1 to -7.6) documented by Tran et al. (2010) for the

403   Wollaston Domain.   This confirms their derivation from reworked Wollaston Group

404   metasedimentary rocks and implicates high-temperature anatexis as a source of the granitic

405   melts. It is not possible to identify exactly which part (i.e., upper or lower succession) of the

406   volcano-sedimentary succession the rocks were derived from, although the dominantly

407   Paleoproterozoic detrital monazite and zircon population and less evolved $\varepsilon Nd_{(1830)}$ signature are

408   broadly consistent with an upper Wollaston Group source. Based solely on the areal-percentages

409   of oscillatory zoned and dark-BSE cores in Figure 5, 15 - 20% by volume of the monazite is

410   inherited. An even larger volume fraction of zircon is detrital in origin based on the proportion

411   of narrow overgrowths to resorbed cores. The provenance of these components broadly matches

412   the detrital zircon populations measured elsewhere in the middle succession of the Wollaston

413   domain (Su et al., 2012).

414   The xenocrystic components in monazite (i.e., >1880 Ma) have trace element

415   compositions that are anomalously enriched in As, V, and locally Mo, Nb, and Ba compared to

416   monazite found in peraluminous granitic pegmatites (Trebilcock) and associated with

417   amphibolite-facies psammitic and pelitic metasedimentary gneisses (44069). The Kulyk Lake

418   monazite also displays strongly-fractionated REE patterns with $(La/Yb)_{CN} > 1000$ compared to

419   values of <200 for Trebilcock and 44069. Additional compositional anomalies in the inherited

420   cores such as a small negative Eu-anomaly (0.25) and Th/U between 250 and 550 also contrast

421   with monazite in the limited number studies in which it was analyzed by LA-ICP-MS in

422   metamorphic and magmatic systems (Bea and Montero, 1997; Bea et al., 1994; Gregory et al.,

423   2009; Hermann and Rubatto, 2003; McFarlane and Frost, 2008; Rubatto et al., 2001) . These

424   observations are most compatible with a portion of the inherited monazite being derived from

425   metalliferous black shales that share similar enrichment in Mo, V, As, and Ba (e.g., Breit et al.,

426   1989; Desborough et al., 1989) as well elevated LREE/HREE (Lehmann et al., 2007). The

427   formation of authigenic monazite nodules in a variety of Ca-poor sedimentary lithologies

428   (Borisova et al., 2010; Bouch et al., 2002) and especially REE-enriched metalliferous black

429   shales (Alipour-Asll et al., 2012) provides a viable mechanism for the formation of abundant As,

430   V,   Mo, and LREE-enriched monazite prior to partial melting associated with peak-

431   metamorphism during the Trans-Hudson orogeny.

432   The existence of abundant xenocrystic monazite and zircon components, mineralogical

433   similarities with the neighbouring monzonite dyke, timing with respect to peak Trans-Hudson

434   metamorphism, and high-temperature crystallization inferred from oxygen-isotope data

435   (McKeough, 2013) preclude hydrothermal vein deposition processes for the monazite-apatite-

436   Fe(Ti)-oxide occurrence. The abundance of inherited monazite and zircon in the monazite-

437   apatite-Fe(Ti)-oxide zone leaves little doubt that entrainment of either detrital or diagenetic

438   phosphates was an important process in the source region to these rocks. Entrainment of

439   xenocrystic monazite and zircon have been identified elsewhere in pegmatite-related U-Th-REE

440   occurrences in the region such as Frasers Lake (McKechnie et al., 2013) and Yellow (Karin)

441   Lake (McKeough et al., 2010). In these occurrences monazite, apatite, zircon, Fe(Ti)-oxide and

442   biotite typically occur as glomerophorphyric masses, locally forming cm-scale lenses. Although

443   a detrital origin for some part of the apatite in the phosphatic zone at Kulyk Lake could not be

444   confirmed, detrital and diagenetic apatite are both common constituents in marine sediments

445   (Bouch et al., 2002; Morton and Yaxley, 2007).   Reconnaissance LA-ICP-MS trace element

446   mapping of apatite failed to reveal core-overgrowth relationships similar to those preserved by

447   monazite and zircon and the locally euhedral habit of apatite (e.g., Figure 3a) and the more

448   widespread occurrence of zircon inclusions in apatite compared to monazite suggests that the

449   bulk of the apatite may have crystallized from the anatectic melt.

450   Partial melting of source rocks already enriched in either detrital or diagenetic phosphates

451   is, therefore, a potentially viable mechanism to account for the observed volume fraction of

452   inherited material in the monazite-apatite-Fe(Ti)-oxide zone.   Given the limited solubility of

453   monazite and apatite in low-aH$_2$O metaluminous (ASI ~ 1) to weakly peraluminous (ASI < 1.3)

454   granitic liquids derived from biotite-dehydration melting at granulite-facies conditions (Montel,

455   1993; Rapp and Watson, 1986; Watson et al., 1989; Wolf and London, 1994; Wolf and London,

456   1995) the preservation of inherited xenocrystic phosphate components is likely. Entrainment of

457   xenocrystic components was apparently followed by magmatic (cotectic) crystallization of

458   monazite, zircon, and apatite followed by emplacement coevally with the granitic dykes. The

459   observed textural relationships between monazite, apatite, zircon, and titanomagnetite suggest

460   that cotectic crystallization of overgrowths on monazite and zircon occurred prior to

461   titanomagnetite crystallization, the latter typically occurring interstitial to monazite but locally

462   occluding apatite and zircon. Apatite grain morphology varies from euhedral to anhedral,

463   suggesting that it may have crystallized over a wider range of conditions. The interstitial and

464   locally oikocrystic nature of titanomagnetite in the phosphatic zones (see Figure 2c) is

465   reminiscent of an intercumulus texture.

466   **Revealing source controls on pegmatite fertility**

467 The in-situ LA-(MC)-ICP-MS techniques applied here have revealed compelling evidence for

468 the involvement of potentially metalliferous protoliths in the anatectic source region to the

469 pegmatite bodies at Kulyk Lake. This evidence is preserved primarily as complex intragranular

470 zoning in monazite of elements such as V, As, Mo, Nb, Ba, Eu, (La/Yb)$_{CN}$, and Th/U. Many of

471 these elements occur at concentrations (<10 ppm) well below practical operating conditions for

472 EPMA and so LA-(MC)-ICP-MS trace element mapping is a critical first step in assessing

473 evidence for inheritance in these S-type magmatic systems. Linking high-precision U-Pb isotope

474 geochronology with compositional zoning for these low-abundance trace elements is also critical

475 for resolving inherited xenocrystic components from those that crystallized from the

476 differentiated granitic liquid.

477   The trace element composition of 1830 Ma magmatic overgrowths analyzed by LA-ICP-

478 MS spot analyses may also reveal details of magmatic differentiation and fractionation processes

479 in contaminated peraluminous magmas. For example, discontinuous 1830 Ma overgrowths have

480 higher absolute concentrations of U and Mo compared to the xenocrystic domains. This

481 enrichment of incompatible U and Mo is typically associated with fractional crystallization of

482 granitic magmas (e.g., Blevin and Chappell, 1995). The concentrations of V, As, Nb, Mo, and

483 Ba encountered in the magmatic overgrowths at Kulyk Lake are also higher than those measured

484 for either the monazite from the Trebilcock pegmatite (Tomascak et al., 1998) or from regional

485 metamorphic monazite (44069) from the Wilmington Complex, Delaware (Aleinikoff et al.,

486 2006). Hence, monazite that crystallized from variably fractionated granitic liquid at the Kulyk

487 Lake monazite-apatite-Fe(Ti)-oxide occurrence appears to faithfully record anomalous

488 geochemical signatures related to source-rock entrainment and partial assimilation processes

489 (McKeough, 2013). Source rock heterogeneity of this nature has been identified in other granitic

490 systems on the basis of textural, major-element, and isotopic heterogeneity (Clemens et al.,

491 2010). Monazite zoning patterns and concentration anomalies for the broad range of trace

492 elements measureable using LA-ICP-MS could provide another method of exploring source

493 heterogeneity in peraluminous granitic melts.

Implications

495 This study illustrates how the integration of *in-situ* LA-(MC)-ICP-MS trace element mapping, U-

496 Pb geochronology, trace-element spot analyses, and *in-situ* Nd-isotope systematics on monazite

497 may be combined to reveal details of REE-phosphate occurrences associated with granitoid

498 intrusions. In particular, the integration of LA-ICP-MS trace element mapping and high-spatial

499 resolution compositionally-guided U-Pb geochronology may be used to distinguish between

500 xenocrystic and magmatic components in these systems that may otherwise be missed using

501 conventional approaches. The results obtained here for the monazite-apatite-Fe(Ti)-oxide

502 occurrence also suggests that the prospectivity of REE-enriched pegmatite corridors in the

503 Wollaston Domain and, by extension, other high-grade metsedimentary belts, could be identified

504 using monazite geochemistry as a tracer of fertile metalliferous source rocks at depth. Applying

505 similar techniques to other unusual phosphatic deposits may help identify processes related

506 source lithology, xenocryst entrainment, assimilation, and fractional crystallization that are

507 preserved as relict trace element and Nd-isotope zoning features now easily revealed by LA-

508 (MC)-ICP-MS.

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Figure Captions

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741   Figure 1.   Regional and local geology of the Kulyk Lake monazite-apatite-ilmenite REE

742   occurrences relative to the major lithotectonic domains comprising the Hearne Province in

743   northern Saskatchewan.   A) Regional lithotectonic subdivisions identified within the Hearne

744   Domain of the Western Churchill Province in Saskatchewan (Bickford et al., 1990; Hoffman,

745   1990).   B) Local geology adjacent to Kulyk Lake showing the location of the monazite-apatite-

746   Fe(Ti)-oxide mineralization relative to regional amphibolite-facies paragneiss and syn- to post-

747   tectonic granite and pegmatite bodies.

748   Figure 2. A) Detail geology of the Kulyk Lake monazite-apatite-Fe(Ti)-oxide occurrence.   The

749   mineralization occurs in association with an aplitic leucogranite dyke which is in turn enclosed

750   by a locally pegmatite leucogranite dyke.   B) A polished slab of sample MMKJG037 showing

751   the distribution of granoblastic monazite-rich and apatite-rich zones within the mineralized zone.

752   Dark clots of titanomagnetite are heterogeneously distributed.   The area of the occurrence is

753   overprinted by an earthy hematite alteration that appears to have infiltrated along fracture planes.

754   C) portion of a polished thin section obtained from MMKJG037 showing the granoblastic nature

755   of monazite and apatite with titanomagnetite forming either interstitial to the phosphates or as

756   larger poikilitic grains enclosing apatite.   Zircon occurs as a ubiquitous accessory mineral and is

757   included within all of the other major minerals in the rock.

758   Figure 3.   A) Reflected light image showing grain boundary relationship between monazite,

759   apatite, titanomagnetite (T-mag), and zircon.   Apatite locally forms euhedral to subhedral

760   hexagonal grains.   Zircon grains included in apatite locally display well-developed radial

761   fractures typically associated with positive volume change in zircon during accumulation of

762   radiation damage (metamictization).   This image also shows the interstitial nature of

763   titanomagnetite which locally forms thin meniscuses between apatite and monazite. Exsolution

764   of rutile (Rt) and abundant micron-scale ilmenite are also visible within the titanomagnetite host.

765   B) Cathodoluminescence image of a portion of a single apatite grains. Arrows point to dark-CL

766   healed fractures that are not visible in optical or BSE images. A dark-CL network of potentially

767   recrystallized apatite also envelopes occluded zircon grains and those decorating the grain

768   boundary with the adjacent grain below.

769   Figure 4. Reflected light photomicrograph mosaic showing details of a zircon-rich portion of

770   monazite-apatite-Fe(Ti)-oxide zone. Zircon occurs primarily along grain boundaries or at triple

771   junctions in the monazite-apatite aggregate. A lower proportion of zircon grains occur included

772   within the cores of apatite and rarely within monazite grains. A large fraction of the zircon

773   displays higher reflectance overgrowths.

774   Figure 5. Backscattered electron (BSE) image of a monazite-rich portion of the occurrence.

775   BSE-imaging revealed the presence of three distinct compositional domains: 1) bright-BSE

776   cores; 2) dark-BSE oscillatory zoned core domains, and; 3) uniform-BSE overgrowths.

777   Figure 6.   Trace-element maps measured by LA-ICP-MS showing the semi-quantiative

778   concentrations of U, V, As, Th/U and $(La/Yb)_{CN}$ in monazite grains1 and 2. In both case the

779   monazite grain is surrounded by apatite. Hot colours correspond to high concentrations whereas

780   cold colours correspond to low concentrations. Semi-quantitative concentration scale is given

781   for each element.   Low-U cores contains domains with anomalously high As and V

782   concentrations in addition to discrete high Th/U domains and minor $(La/Yb)_{CN}$ zoning.

783   Figure 7. Reflected light live video image from the laser (background) with superimposed semi-

784   transparent trace element map showing the distribution of vanadium. The sample was polished

785   with 3 μm diamond past after raster mapping to remove most of the laser ablation damage (some

786   is still visible). Dark circles are craters left by ablations with 13 μm and 17 μm diameter spot

787   used for U-Pb geochronology. Red circles are the locations of targets for full trace element

788   quantification.

789   Figure 8. Chondrite-normalized REE patterns for monazite grains1, 2, and 3 with data for

790   Trebilcock and 44069 standards for comparison. Slightly higher Er value is probably a result of

791   a small^{150}(Nd+Sm)^{16}O$^+$ interference on ^{166}Er. The data were calculated using Nd as an internal

792   standard at concentrations previously determined by EPMA. The Kulyk Lake monazite displays

793   very high LREE/HREE fractionation with (La/Yb)$_{CN}$ values > 1000.

794   Figure 9. Histogram and probably density plot of ^{207}Pb/^{206}Pb ages for inherited components in

795   monazite grains 1, 2, and 3. The provenance of the inherited domains is dominated by

796   Paleoproterozoic ages with major peaks at 1880 Ma and 2150 Ma and a broad spectrum of ages

797   centered at about 2050 Ma.

798   Figure 10. Compilation of trace element compositional zoning for V, Th/U, and Mo in monazite

799   grain3 with superimposed ^{207}Pb/^{206}Pb ages measured using 17 μm diameter craters. Absolute 2σ

800   errors (not shown for clarity) are 15 to 17 Ma (i.e., ~1%). There is a close correspondence

801   between compositional zoning and ^{207}Pb/^{206}Pb age with Mo-enriched domains preserving 2020

802   Ma to 2150 Ma ages whereas patchy low Th/U domains are 1870 Ma to 1900 Ma. The resorbed

803   low-V and low-Mo nucleus preserves older Paleoproterozoic ages and is interpreted to be

804   detrital.

805   Figure 11. Conventional Concordia diagrams showing the results of in-situ U-Pb geochronology

806   applied to monazite in the monazite-apatite-ilmenite vein (A, B) and in the aplitic host to the

807   vein (C,D). Black squares are the absolute positions of the measured ratios which are used to

808   help identify data clusters. Both samples contain higher-U overgrowths that crystallized at 1828

809    $\pm$ 6 Ma. Both rocks were also affected by a younger thermal resetting event between 1770 Ma to

810   1790 Ma.

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Table 1. LA-ICP-MS trace element quality control on GSE-1G

Analyte	GSE-1G Mean (ppm)	1σ	GSE-1G Ref. value (ppm)	1σ	Average D.L. (ppm)
^{44}Ca	52750	2100	52887	2140	890
^{51}V	446	20	440	20	1.39
^{75}As	392	14	260	90	8.72
^{89}Y	415	40	410	30	0.16
^{93}Nb	441	11	420	40	0.18
^{98}Mo	401	11	390	30	0.34
^{137}Ba	364	11	427	5	0.21
^{139}La	391	11	392	4	2.01
^{140}Ce	411	11	414	4	0.81
^{141}Pr	464	27	460	10	1.10
^{146}Nd I.S.			453		
^{147}Sm	489	42	488	5	0.35
^{153}Eu	411	9	410	20	0.06
^{157}Gd	527	18	490	70	0.48
^{159}Tb	511	12	480	20	0.05
^{163}Dy	550	14	524	6	0.12
^{165}Ho	514	53	489	6	0.02
^{166}Er	586	14	595	6	0.04
^{169}Tm	531	38	500	20	0.01
^{173}Yb	535	13	520	5	0.09
^{175}Lu	538	55	518	6	0.01
^{208}Pb	382	10	378	12	0.36
^{232}Th	387	24	380	20	0.04
^{238}U	431	11	420	30	0.03

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Table 2. EPMA major element chemistry of BSE imaged compositional domains

El. Wt%	Cores - Bright BSE oscillatory zones		Cores - Dark BSE oscillatory zones		Overgrowths	
	Mean (*n* = 5)	1σ	Mean (*n* = 7)	1σ	Mean (*n* = 7)	1σ
P	13.2	0.2	13.3	0.1	12.8	0.06
Si	0.27	0.0	0.22	0.0	0.49	0.05
Ca	0.56	0.1	0.55	0.0	0.41	0.08
La	14.0	0.3	14.3	0.3	13.9	0.39
Ce	27.1	0.4	27.1	0.5	27.2	0.28
Pr	2.20	0.1	2.21	0.1	2.28	0.17
Nd	9.08	0.2	9.04	0.3	9.05	0.24
Y	0.27	0.2	0.23	0.2	0.20	0.21
Th	3.32	0.5	2.86	0.4	4.41	0.46

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Table 3. Monazite grain1, LA-ICP-MS trace element data

Analysis#	Map1, High-V cores				Map 1, higher-U uniform overgrowths				
	1	2	3	4	1	2	3	4	5
^{44}Ca	5140	4850	4560	5900	3390	3760	3440	3510	3600
^{51}V	310	277	274	252	60	110	107	132	103
^{75}As*	1214	1167	1607	1265	1034	1145	969	1109	1092
^{89}Y	5129	4971	4190	4830	4660	4459	4850	4340	4746
^{93}Nb	<0.18	<0.18	<0.18	0.25	<0.18	0.22	<0.18	0.19	<0.18
^{98}Mo	<0.34	<0.34	<0.34	0.34	<0.34	0.32	0.81	0.67	0.49
^{137}Ba	3.01	5.09	2.53	29.0	0.67	4.10	0.51	0.57	0.01
^{139}La	158900	159200	150800	148300	123200	137600	135700	141600	137000
^{140}Ce	299500	296200	293400	291200	258000	273900	269100	282300	276600
^{141}Pr	30500	29920	29830	29970	28140	29710	28470	29540	28720
^{146}Nd I.S.	90000	90000	90000	90000	90000	90000	90000	90000	90000
^{147}Sm	9940	9540	9580	9830	10680	10160	10360	9840	10310
^{153}Eu	1057	1054	998	1063	1129	1105	1094	1049	1122
^{157}Gd	6059	5840	5680	5792	6040	5760	6070	5550	5910
^{159}Tb	434	414	380	414	426	409	435	390	418
^{163}Dy	1552	1486	1330	1439	1479	1386	1527	1354	1476
^{165}Ho	201	191	167	188	181	178	186	172	186
^{166}Er	360	349	294	338	306	297	322	296	319
^{169}Tm	29.4	28.4	23.0	27.2	22.7	22.6	25.2	23.2	23.5
^{173}Yb	113	105	84.8	103	81.8	81.9	88.1	86.5	87.4
^{175}Lu	13.0	13.0	16.5	17.0	19.1	24.0	23.2	27.2	24.3
^{208}Pb	4230	3680	6720	7480	3652	6440	4890	6410	6420
^{232}Th	26110	22720	39800	44500	23240	36520	35000	38900	39510
^{238}U	78	79	96	96	126	159	153	171	156
Eu/Eu*	0.25	0.26	0.25	0.26	0.26	0.26	0.25	0.26	0.26
(La/Yb)$_{CN}$	960	1029	1208	983	1023	1141	1046	1112	1065
Th/U	335	289	415	464	184	230	229	228	254

816   Values in ppm

817

Table 4. Monazite grain2, LA-ICP-MS trace element data

Analysis#	Map2, High-Vanadium cores			Map 2, higher-U uniform overgrowths		
	1	2	3	1	2	3
^{44}Ca	4650	4160	3880	4200	3930	4260
^{51}V	334	240	277	107	112	111
^{75}As*	1758	1516	1656	994	961	995
^{89}Y	3855	4220	4042	3909	3903	3939
^{93}Nb	<0.18	0.93	<0.18	<0.18	0.23	<0.18
^{98}Mo	<0.34	0.42	<0.34	0.61	0.56	0.52
^{137}Ba	62.8	21.4	0.33	0.46	0.21	0.28
^{139}La	147600	149000	152300	143900	145600	145500
^{140}Ce	296300	283100	294100	290800	286100	286600
^{141}Pr	29940	29310	29660	29530	29540	29300
^{146}Nd I.S.	90000	90000	90000	90000	90000	90000
^{147}Sm	9720	9630	9670	9730	9490	9590
^{153}Eu	1000	988	995	979	968	967
^{157}Gd	5310	5358	5218	5123	5137	5123
^{159}Tb	374.1	383.1	366	357.7	349.4	359.8
^{163}Dy	1247	1349	1280	1203	1216	1231
^{165}Ho	157	173	159	154	152	153
^{166}Er	268	301	273	263	265	269
^{169}Tm	20.7	23.8	20.1	20.3	20.6	20.8
^{173}Yb	71.2	88.7	74.1	75.0	77.6	74.3
^{175}Lu	6.07	8.22	6.33	6.74	6.9	6.84
^{208}Pb	8420	8670	8100	4340	4490	4890
^{232}Th	49300	55100	50200	27170	27850	30620
^{238}U	97	107	91	127	136	143
Eu/Eu*	0.25	0.25	0.25	0.25	0.25	0.25
(La/Yb)$_{CN}$	1408	1141	1396	1303	1275	1330
Th/U	507	515	549	215	206	214

818   Values in ppm

819

Table 5. Monazite grain3, LA-ICP-MS trace element data

Analyte	Map 3, high-Mo core domains					Map 3, low Th/U			Low-V
	Core	Core-1	Core-2	Core-3	Core-4	Og1	Og1-1	Og1-2	
^{44}Ca	7620	8050	5690	5490	6110	5320	5600	4880	5390
^{51}V	151	159	148	150	139	181	185	189	120
^{75}As*	1153	1118	1052	1090	978	1062	1022	974	1405
^{89}Y	4239	4241	4151	4124	4165	4207	4262	4015	4108
^{93}Nb	0.46	0.66	0.47	0.34	0.35	0.50	0.44	0.46	0.18
^{98}Mo	9.05	9.27	4.98	4.40	4.20	3.67	3.28	2.59	<0.34
^{137}Ba	1.15	2.40	7.70	7.10	0.64	1.49	0.41	0.52	0.42
^{139}La	15640	15760	15670	15520	15450	158600	15690	15960	15610
^{140}Ce	29900	30310	29640	29530	29720	301900	29970	30080	29640
^{141}Pr	30580	30600	30180	30090	30310	30700	30600	30300	30620
^{146}Nd I.S.	90000	90000	90000	90000	90000	90000	90000	90000	90000
^{147}Sm	9280	9520	9460	9380	9420	9490	9460	9330	9350
^{153}Eu	972	988	983	995	995	999	979	923	987
^{157}Gd	6252	6126	6175	5997	5965	5954	5983	5767	6230
^{159}Tb	398	394	392	395	389	390	393	372	398
^{163}Dy	1325	1322	1308	1312	1323	1334	1336	1248	1294
^{165}Ho	167	164	163	164	166	168	170	156	161
^{166}Er	293	284	284	287	286	296	300	277	274
^{169}Tm	23.7	23.3	22.4	23.1	21.8	23.7	24.1	22.2	21.0
^{173}Yb	87.6	90.3	84.9	85.2	84.3	90.2	92.5	84	75.8
^{175}Lu	8.22	8.24	8	7.54	7.79	8.24	8.56	7.65	6.71
^{208}Pb	8400	8160	7930	7880	7640	8300	8030	8320	8380
^{232}Th	49730	48520	47160	45680	45370	49080	48180	49480	50010
^{238}U	179	177	178	177	175	326	329	247	134
Eu/Eu*	0.25	0.25	0.25	0.25	0.25	0.25	0.25	0.24	0.25
(La/Yb)$_C$	1213	1186	1254	1237	1245	1194	1152	1291	1399
Th/U	278	275	265	258	259	151	146	200	374

820   Values in ppm

821

Table 6. LA-ICP-MS trace elements of monazite standards

Analyte	Trebilcock ($n = 5$) Average	1σ	44069 ($n = 4$) Average	1σ
^{44}Ca	12548	2015	4160	239
^{51}V	<1.39	-	2.80	0.12
^{75}As*	589	10	470	5
^{89}Y	22520	2615	15990	679
^{93}Nb	<0.18	-	<0.18	-
^{98}Mo	<0.34	-	<0.34	-
^{137}Ba	<0.21	-	<0.21	-
^{139}La	73600	5616	84925	2076
^{140}Ce	186900	8472	173750	592
^{141}Pr	24806	470	21938	244
^{146}Nd I.S.	100000		98000	
^{147}Sm	36108	1099	16483	306
^{153}Eu	173	27	1996	95
^{157}Gd	23854	1338	12535	539
^{159}Tb	2712	206	1372	42
^{163}Dy	9164	886	5788	185
^{165}Ho	813	93	705	14
^{166}Er	1197	162	1148	44
^{169}Tm	101	15	85.2	8
^{173}Yb	407	57	294	41
^{175}Lu	32.3	5	24.2	4
^{208}Pb	3728	559	579	37
^{232}Th	162140	24559	17173	1301
^{238}U	5971	897	1920	262
Eu/Eu*	0.01	0	0.28	0
(La/Yb)$_{CN}$	123	20	196	33
Th/U	27	2	9	2

822   Values in ppm

823

Table 7. Results of in-situ LA-ICP-MS U-Pb monazite geochronology

Comments	%Pb*	$^{207}Pb/^{235}U$	2σ	$^{206}Pb/^{238}U$	2σ	Err. corr.	$^{207}Pb/^{206}Pb$ age (Ma)	2SE	%Disc
Host aplite dyke – thermal resetting									
Mnz5b	100.0	4.80	0.08	0.3193	0.0036	0.41	1782	15	-0.2
Mnz2b	100.0	4.81	0.08	0.3184	0.0035	0.68	1791	12	0.5
Mnz15-core	99.8	4.81	0.09	0.3191	0.0060	0.58	1792	15	0.1
Mnz4	99.8	4.85	0.09	0.3200	0.0048	0.42	1798	15	0.4
Host aplite dyke – crystallization age									
Mnz3b	99.8	5.04	0.08	0.3264	0.0041	0.45	1829	11	0.4
Mnz17a	99.9	5.04	0.12	0.3263	0.0068	0.59	1826	20	0.2
Mnz15-ovrgr	100.0	5.05	0.10	0.3269	0.0055	0.57	1814	15	-0.7
Mnz22	100.1	5.06	0.08	0.3279	0.0045	0.49	1821	11	-0.4
LA-ICP-MS map-guided spots									
Map1_core-1	100.1	5.68	0.11	0.3468	0.0042	0.19	1925	21	0.3
Map1_core-2	99.9	5.87	0.11	0.3512	0.0046	0.42	1963	19	1.2
Map1_core-3	99.9	6.03	0.11	0.3587	0.0046	0.55	1979	15	0.2
Map1_core-4	99.9	5.36	0.01	0.3372	0.0046	0.36	1873	21	0.1
Map1_core-5	99.6	5.32	0.09	0.3347	0.0040	0.33	1878	17	1.0
Map2_core-1*	97.9	4.77	0.11	0.3141	0.0048	0.98	1831	26	-2.6
Map2_core-2*	98.7	5.39	0.13	0.3358	0.0051	0.96	1912	23	2.2
Map2_core-3*	98.9	5.20	0.12	0.3292	0.0051	0.97	1886	23	2.3
Map3_highMo-1*	97.5	5.20	0.11	0.3275	0.0046	0.97	1895	27	0.9
Map3_highMo-2*	97.5	5.13	0.10	0.3245	0.0042	0.98	1876	24	1.4
Map3_highMo-3*	98.1	5.20	0.10	0.3294	0.0045	0.96	1894	24	1.2
Map3_highMo-4*	98.3	5.26	0.12	0.3304	0.0050	0.97	1905	26	3.0
Map3_highMo-5*	98.7	5.39	0.11	0.3328	0.0045	0.96	1919	24	3.1
Map3_highMo-6*	98.6	5.17	0.10	0.3273	0.0045	0.95	1890	24	3.4
Map3_lowV_core*	94.6	4.90	0.09	0.3144	0.0043	0.99	1847	16	2.5
Map3_lowV_core*	94.6	4.91	0.09	0.3139	0.0045	0.99	1851	15	3.8
Map3_ovrgr-1	100.0	5.60	0.07	0.3442	0.0035	0.45	1897	14	-0.5
Map3_ovrgr-2	100.0	5.45	0.06	0.3399	0.0037	0.48	1870	14	-0.9
Map3_ovrgr-3	99.7	5.41	0.06	0.3351	0.0035	0.30	1883	13	1.1
Map3_ovrgr-4	99.9	5.46	0.07	0.3408	0.0038	0.61	1880	15	-0.5
Map3_ovrgr-5	99.8	5.56	0.06	0.3436	0.0037	0.24	1889	15	-0.9
Map3_ovrgr-6	99.9	5.59	0.07	0.3446	0.0038	0.31	1875	12	-1.8
BSE-guided spots – thermal resetting									
Ovrgr	100.0	4.72	0.11	0.3152	0.0071	0.60	1780	23	0.9
Oscll core	100.0	4.73	0.34	0.3150	0.0063	0.35	1763	69	-0.1
Ovrgr	100.0	4.65	0.13	0.3126	0.0063	0.36	1794	28	2.3
Ovrgr	99.7	4.69	0.12	0.3164	0.0050	0.42	1776	24	0.2
Ovrgr	100.0	4.76	0.14	0.3168	0.0049	0.39	1772	27	-0.1
BSE-guided spots – crystallization age									
Ovrgr	99.9	5.08	0.12	0.3287	0.0055	0.50	1829	18	-0.3
Ovrgr	100.0	5.11	0.16	0.3293	0.0049	0.34	1809	32	-1.5
Ovrgr	99.9	5.10	0.25	0.3298	0.0060	0.13	1839	41	0.1
Ovrgr	100.0	5.09	0.17	0.3299	0.0067	0.56	1832	28	-0.3
Ovrgr	100.0	5.01	0.13	0.3278	0.0067	0.46	1846	27	1.1

824   * analyses corrected for common-Pb using the Andersen (2002) correction routine.

Table 8. Results of in-situ LA-ICP-MS U-Pb zircon geochronology

Comments	%Pb*	$^{207}Pb/^{235}U$	2σ	$^{206}Pb/^{238}U$	2σ	Err. corr	$^{207}Pb/^{206}Pb$ age (Ma)	2σ	%Disc
z-30	100.6	4.82	0.13	0.3198	0.0110	0.75	1797	26	0.5
z-20-2	99.3	4.83	0.10	0.3176	0.0081	0.52	1797	31	1.1
z-12	99.6	4.96	0.07	0.3138	0.0083	0.10	1871	19	5.8
z-13	99.6	4.91	0.11	0.3089	0.0100	0.87	1871	47	7.3
z-28	98.7	4.65	0.09	0.2932	0.0083	0.56	1848	30	10.3
z-11	98.7	4.74	0.08	0.2950	0.0083	0.65	1900	22	12.3
z-3-1	97.2	4.61	0.14	0.2866	0.0100	0.75	1906	28	14.8
z-18	98.2	4.38	0.09	0.2633	0.0077	0.70	1961	27	23.2
z-1	96.7	4.40	0.08	0.2632	0.0080	0.57	1971	28	23.6
z-14	97.0	4.50	0.07	0.2638	0.0070	0.47	1998	26	24.5
z-27	96.5	4.36	0.09	0.2609	0.0084	0.76	1979	26	24.5
z-25	96.8	6.29	0.21	0.3160	0.0130	0.92	2345	20	24.6
z-15	96.7	4.37	0.11	0.2611	0.0100	0.62	1989	30	24.8
z-8	97.0	4.28	0.09	0.2548	0.0068	0.39	1972	34	25.8
z-26-2	96.7	3.75	0.10	0.2326	0.0075	0.78	1897	29	28.9
z-17	96.6	3.96	0.12	0.2381	0.0092	0.81	1943	31	29.2
z-6	96.4	4.00	0.09	0.2382	0.0065	0.60	1971	30	30.1
z-3-2	97.2	4.03	0.12	0.2349	0.0081	0.62	2049	42	33.6
z-8b	97.0	4.07	0.08	0.2344	0.0071	0.55	2051	37	33.8

825

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Table 9. Summary of in-situ LA-MC-ICP-MS Sm-Nd isotope data

Spot	$^{147}Sm/^{144}Nd$	2S.E.	$^{143}Nd/^{144}Nd$	2S.E.	$^{145}Nd/^{144}Nd$	2S.E.	eNd(t)	±	T_{DM}
1.1	0.0584	0.0004	0.510732	0.000017	0.348444	0.000012	-4.73	0.73	2405
2.1	0.0579	0.0003	0.510719	0.000018	0.348433	0.000013	-4.86	0.72	2410
3.1	0.0570	0.0003	0.510703	0.000022	0.348446	0.000011	-4.96	0.83	2411
4.1	0.0598	0.0001	0.510774	0.000016	0.348427	0.000014	-4.22	0.63	2386
5.1	0.0582	0.0001	0.510715	0.000022	0.348431	0.000016	-5.00	0.78	2418
6.1	0.0588	0.0001	0.510759	0.000022	0.348444	0.000012	-4.28	0.79	2385
7.1	0.0575	0.0001	0.510719	0.000018	0.348424	0.000012	-4.77	0.66	2404
8.1	0.0577	0.0000	0.510717	0.000024	0.348448	0.000016	-4.85	0.81	2409
9.1	0.0572	0.0001	0.510725	0.000024	0.348438	0.000012	-4.59	0.83	2393
Spot	$^{147}Sm/^{144}Nd$	2S.E.	$^{143}Nd/^{144}Nd$	2S.E.	$^{145}Nd/^{144}Nd$	2S.E.			
Treb1.1	0.2217	0.0003	0.512616	0.000017	0.348444	0.000016			
Treb2.1	0.2208	0.0002	0.512581	0.000022	0.348440	0.000015			
Treb3.1	0.2208	0.0002	0.512613	0.000021	0.348448	0.000017			
Treb4.1	0.2211	0.0001	0.512621	0.000032	0.348441	0.000020			
Treb5.1	0.2214	0.0001	0.512592	0.000030	0.348430	0.000020			
Treb6.1	0.2203	0.0003	0.512600	0.000014	0.348432	0.000015			
Ref. value	0.2167	0.0125	0.512616	0.000011	0.348418	0.000008			

827

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A — Legend:
- ⊙ Township
- ⨉ Mine Location
- ★ U-Th+/-REE-Y-Nb Showing
- — Needle Falls Shear Zone (NFSZ)

B Kulyk Lake Geology
Lithological Units
- Psammitic to Semi-pelitic Gneiss
- Arkosic Gneiss
- Calc-silicate Paragneiss
- Granite
- Pegmatite
- Regional Foliation

A

X

MMKJR006

56

85

43

DFKJR006

76

74

MMKJG037
Fig.2B-C

75

0   2   4 m
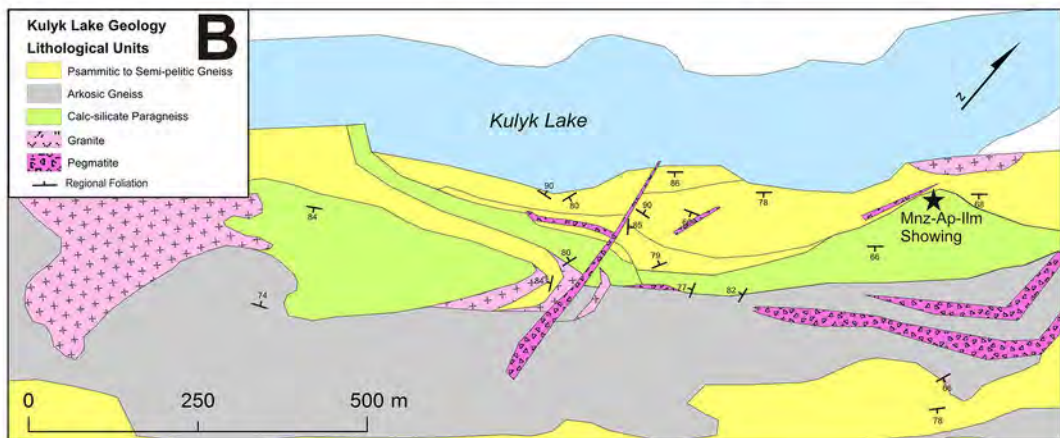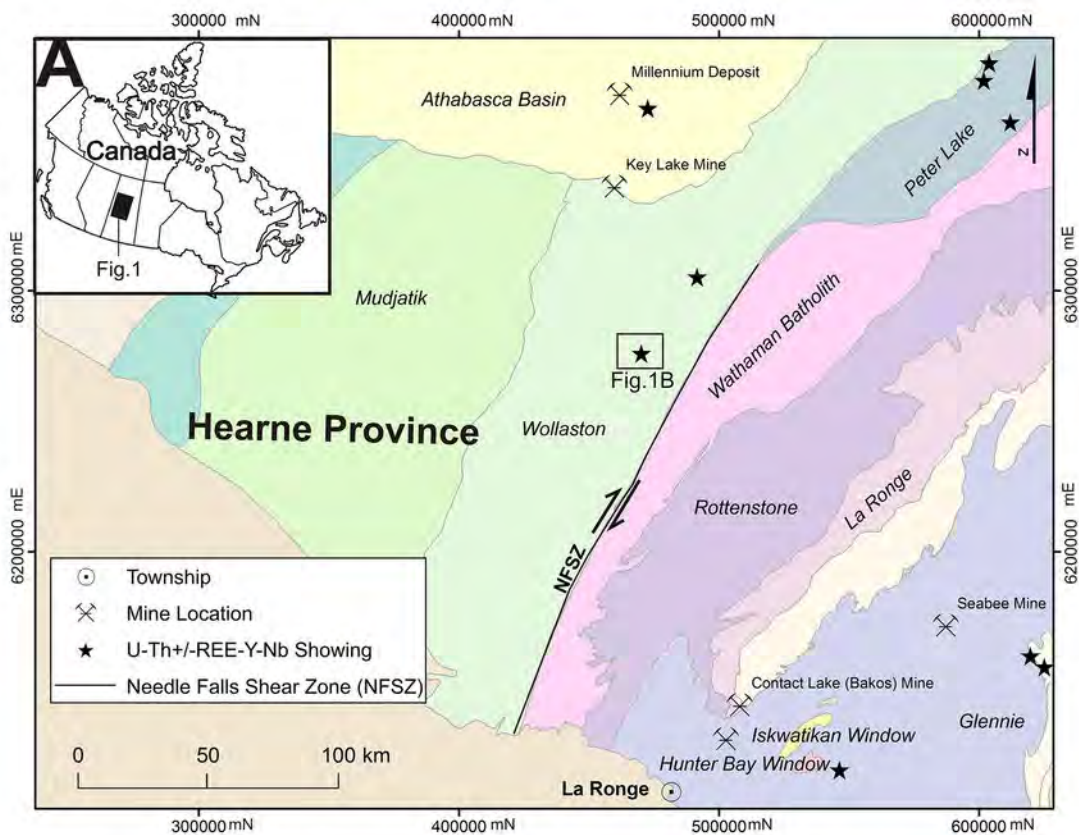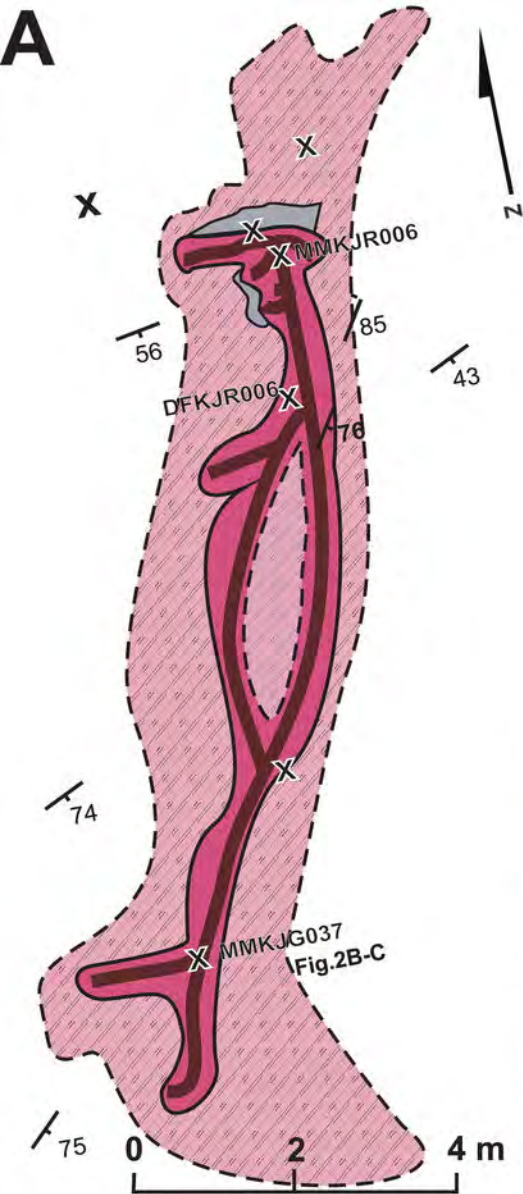
Lithological Units

	Calc-silicate Paragneiss
	Pegmatitic- to granitic- leucocratic dyke
	Aplitic leucocratic dyke
	Monazite-apatite-titanomagnetite mineralization
	Biotite-shist (metasomatic?) zone
	Regional Foliation

B

Area of Fig. 2c

apatite

titano-magnetite + hematite

monazite

titanomagnetite

~3cm

C

Apatite

Mnz

Mnz

Zircons

Tmt

Zircons

Apatite

Mnz

Tmt

Tmt

6.5 mm

A

Mz

Zrn

Ap

Ap

Mz

Rt

T-mag

Ap

Mz

100 μm

B

Ap

Zrn
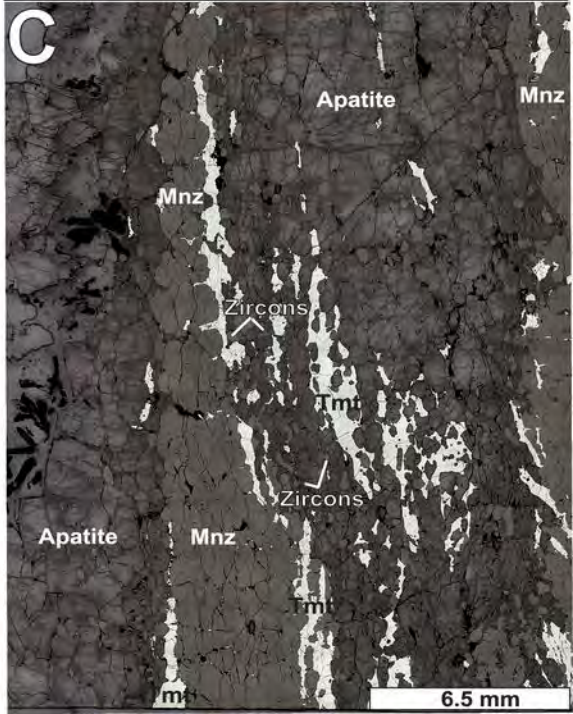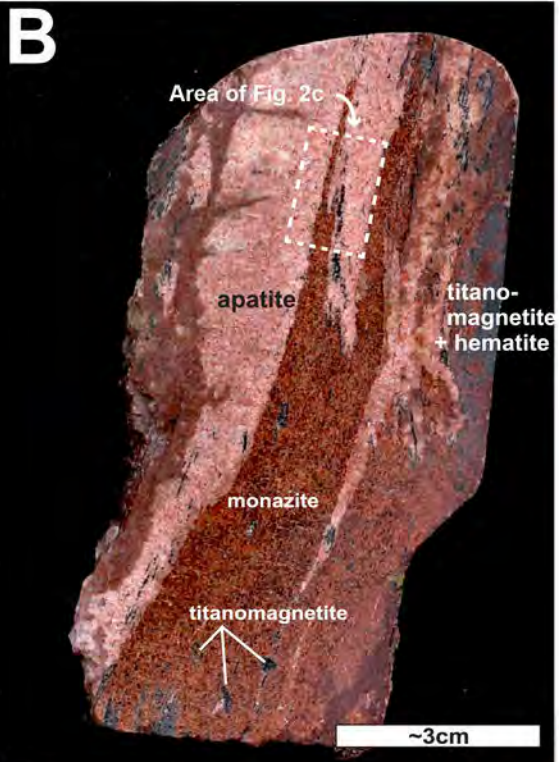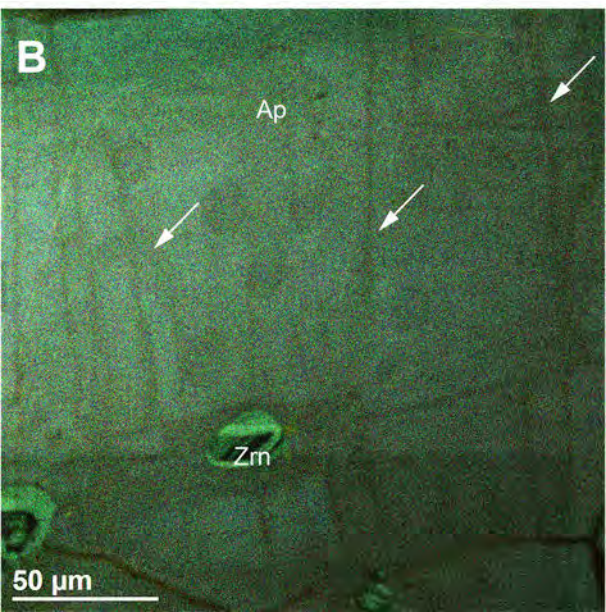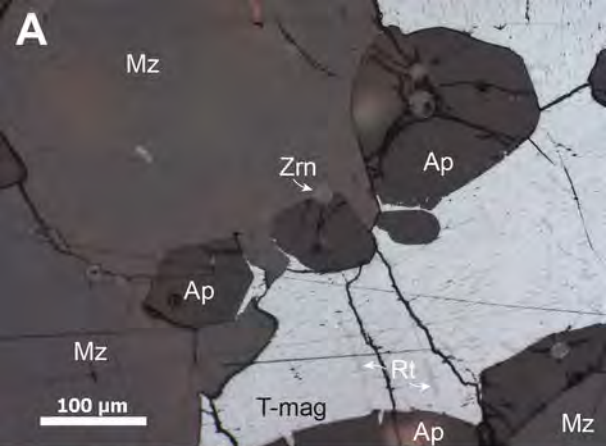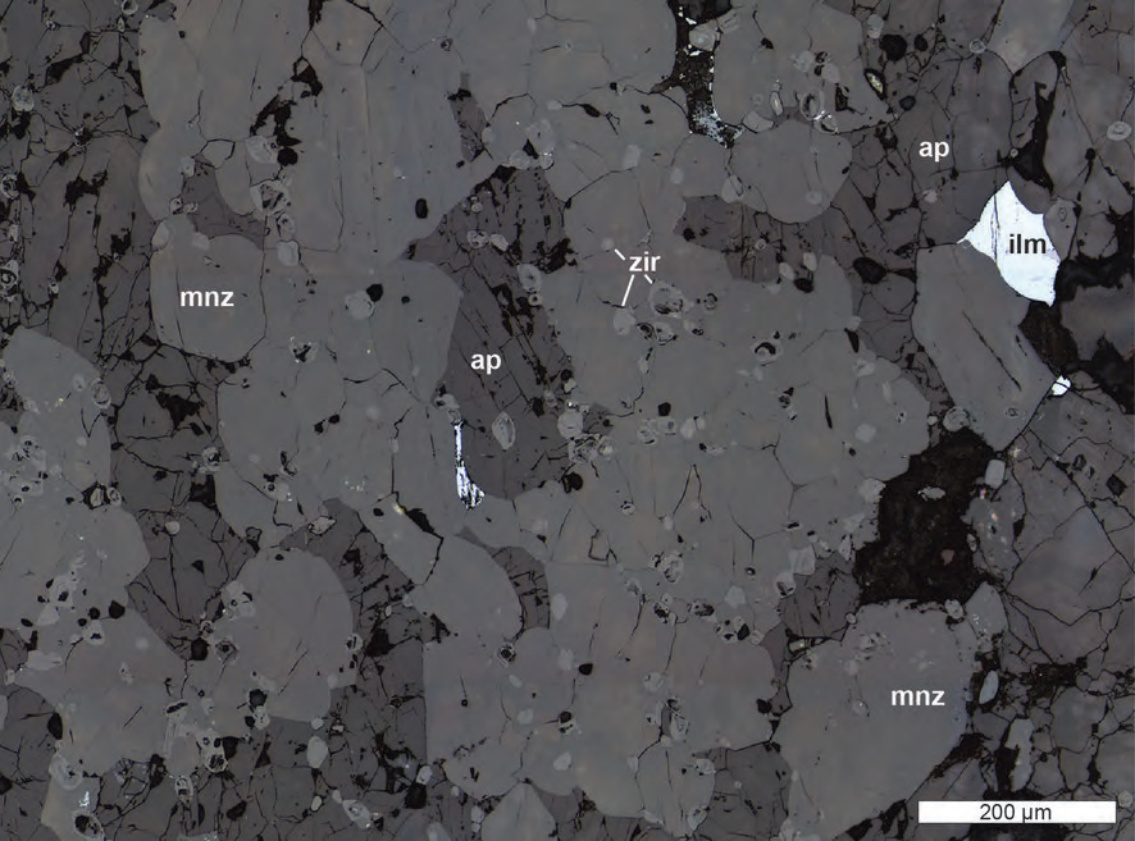
50 μm
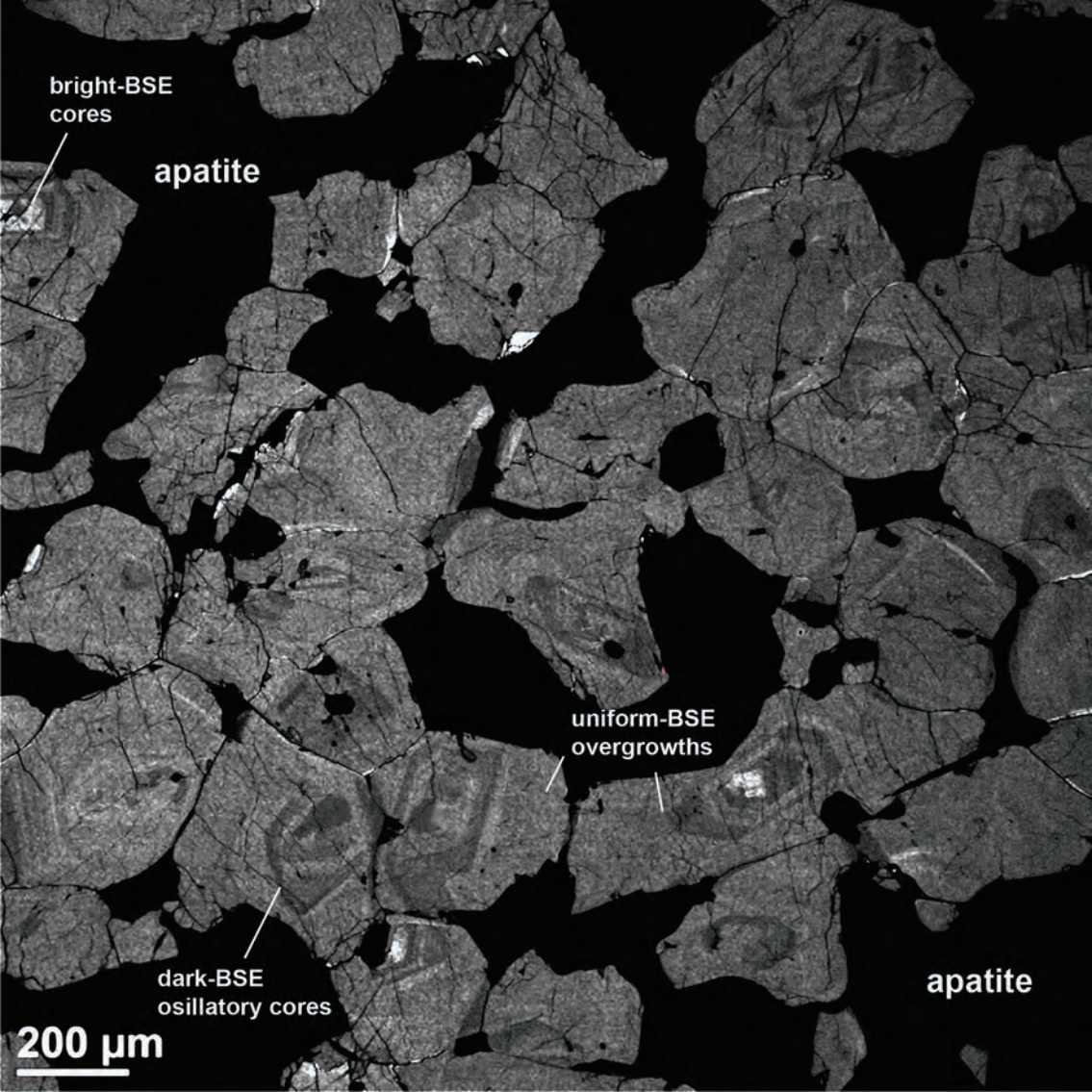
bright-BSE cores

apatite
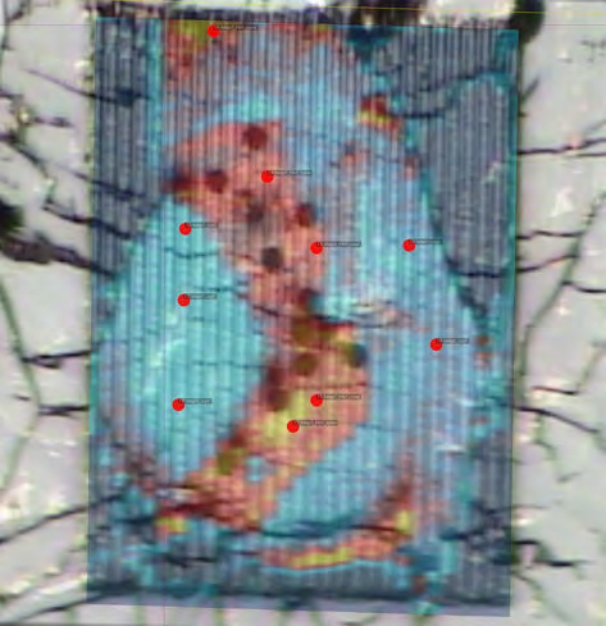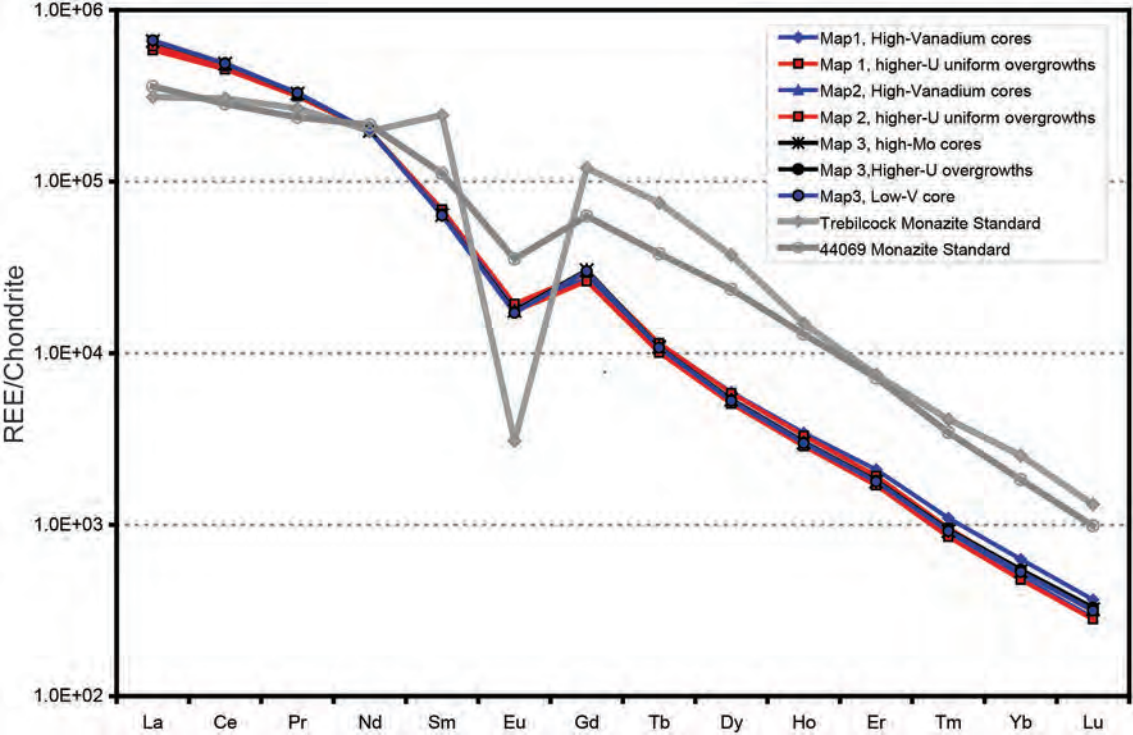
uniform-BSE overgrowths

dark-BSE osillatory cores

apatite
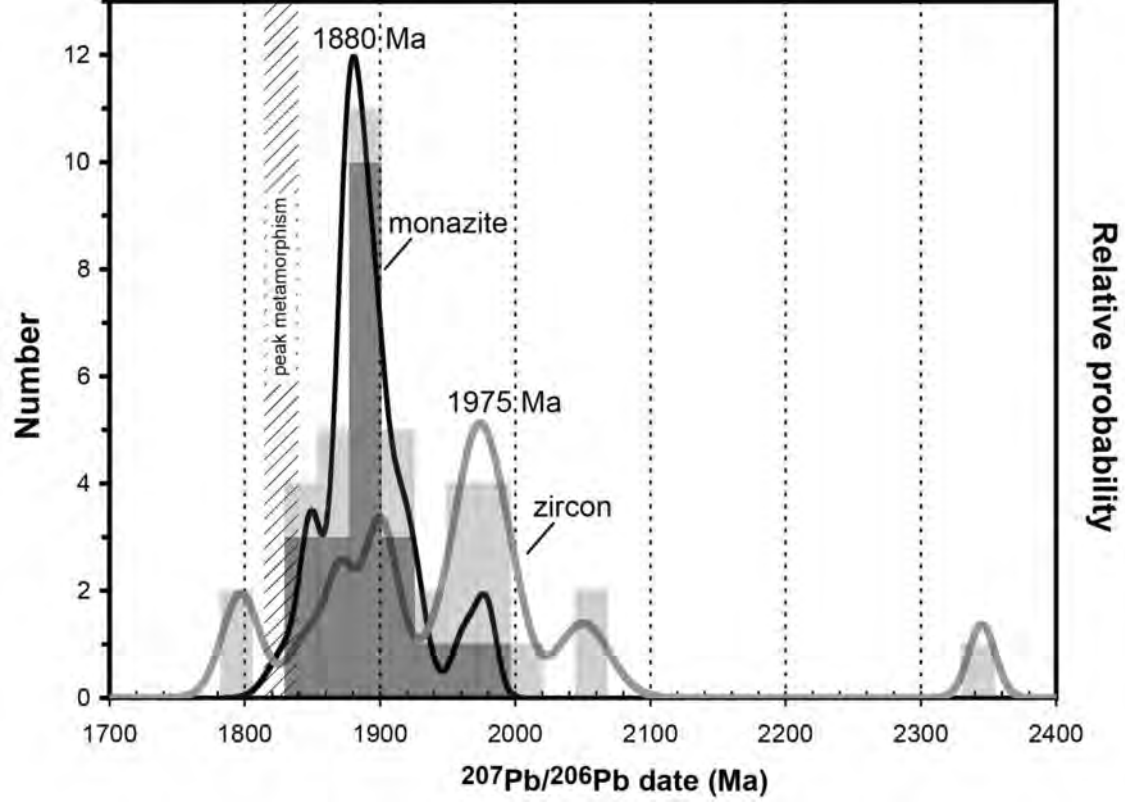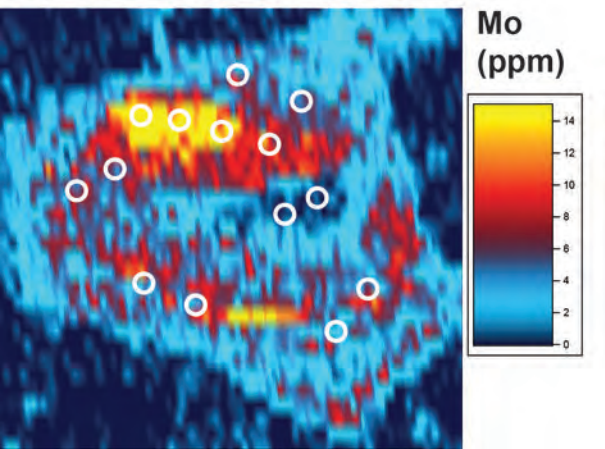
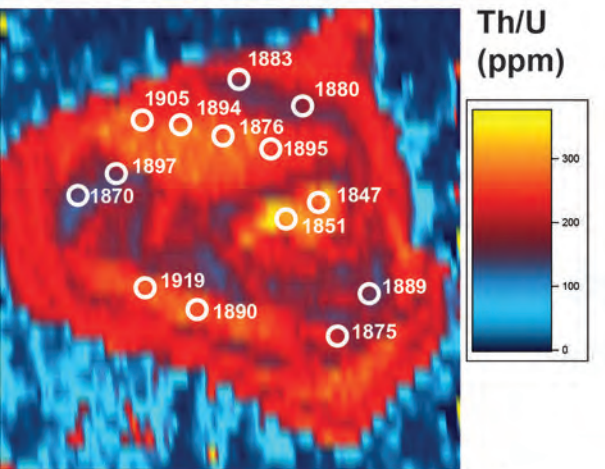200 µm

REE/Chondrite chart with legend:
- Map1, High-Vanadium cores
- Map 1, higher-U uniform overgrowths
- Map2, High-Vanadium cores
- Map 2, higher-U uniform overgrowths
- Map 3, high-Mo cores
- Map 3, Higher-U overgrowths
- Map3, Low-V core
- Trebilcock Monazite Standard
- 44069 Monazite Standard

V (ppm)

1883
1880
1905 1894 1876
1897 1895
1870 1847
1851
1919 1889
1890 1875

140 μm

Th/U (ppm)

1883
1905 1894 1880
1876
1897 1895
1870 1847
1851
1919 1889
1890 1875

Mo (ppm)

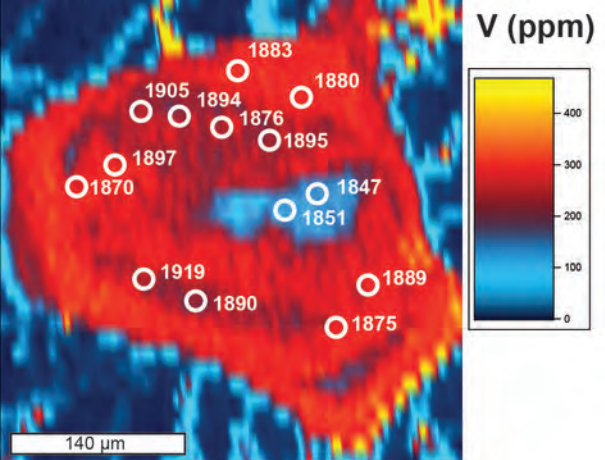**A** — Monazite in phosphate occurrence - overgrowths

Concordia Age = 1829.6 ±9.0 Ma
(2σ, decay-const. errs included)
MSWD (of concordance) = 0.53,
Probability (of concordance) = 0.47
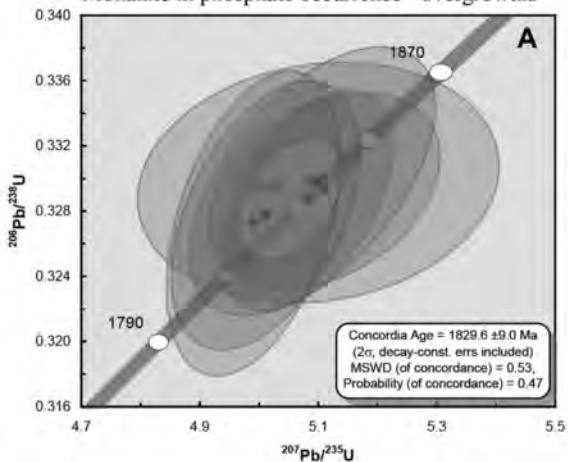
B — Monazite in phosphate occurrence - resetting

Concordia Age = 1768.0 ±9.7 Ma
(2σ, decay-const. errs included)
MSWD (of concordance) = 0.026,
Probability (of concordance) = 0.87
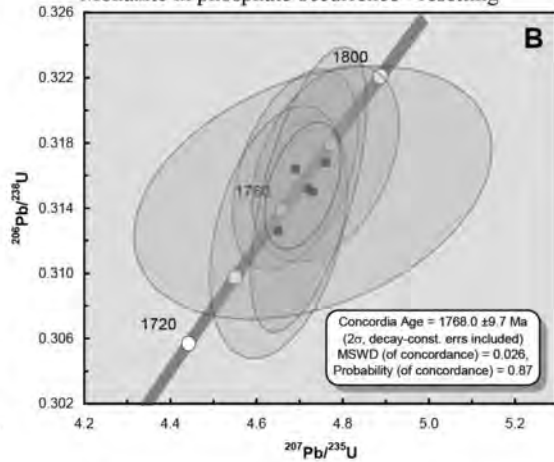
C — Monazite in monzonite host - overgrowths

Intercepts at
2±2 & 1832±15 [±16] Ma
MSWD = 0.0046

Concordia Age = 1826.9 ±7.8 Ma
(2σ, decay-const. errs included)
MSWD (of concordance) = 0.53,
Probability (of concordance) = 0.47
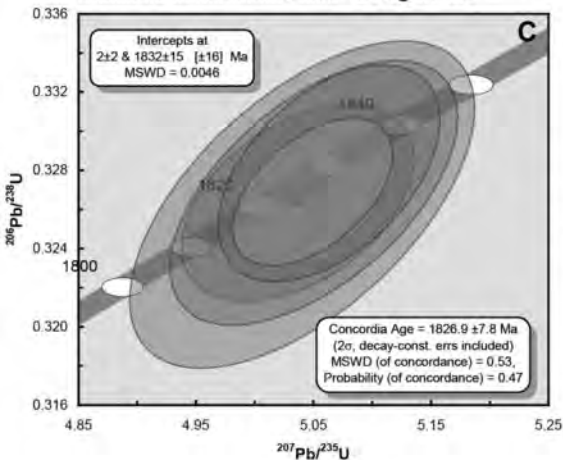
D — Monazite in monzonite host - late thermal

Concordia Age = 1787.4 ±7.6 Ma
(2σ, decay-const. errs included)
MSWD (of concordance) = 0.41,
Probability (of concordance) = 0.52