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U-Pb Geochronology of Zircon by LA-ICPMS in samples from the Grenville Province (La Tuque Area, Quebec)

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1. Objectives

In this report, are presented U-Pb isotopic and electron beam imaging data from seven samples that were collected during the fieldwork session July – August 2017, in the La Tuque area (Lac St-Jean), and from a sample of the Labelle shear zone that was collected along the highway 117. The U-Pb isotopic data were collected using Laser Ablation – High Resolution – Inductively Coupled Plasma – Mass Spectrometry (LA-HR-ICP-MS: Photon-Machines G2 and Nu Attom instruments) and the imaging data using cathodoluminescence and backscatter electron microscopy (Hitachi S-3400N SEM/BSE-CL). Normalisation was done by bracketing unknowns with international standard zircon 91500 (Wiedenbeck et al. 1995), reduced in Lolite v3 (Paton et al. 2010) and further processed in Isoplot (Ludwig, 1999) to generate Concordia diagrams. Zircon grains from the samples 16GC1200, 17AM061, 17AM101, 17AM105, 17GC1055, and 17GC1086 were processed following conventional gravitational and magnetic separation methods and were mounted in epoxy resin. Zircon and monazite grains from the samples 16GC1051 and KP17LSZ were analysed in-situ on polished thin sections. In the following sections are presented the U-Pb analytical and SEM imaging data with reference also to complementary data (trace elements in zircon and hafnium in zircon) collected from the examined samples.

References

- Wiedenbeck M., Allé P., Corfu F., Griffin W.L., Meier M., Oberli F., von Quadt A., Roddick J.C. and Spiegel W. (1995) Three natural zircon standards for U-Th-Pb, Lu-Hf, trace element and REE analyses. *Geostandards Newsletter*, 19, 1-23.
- Paton, C., Woodhead, J., Hellstrom, J., Hergt, J., Greig, A. & Maas, R (2010) Improved laser ablation U-Pb zircon geochronology through robust down-hole fractionation correction. *G Cubed*, 11, doi:10.1029/2009GC002618
- Ludwig K. R. (1999) *User's Manual for Isoplot/Ex Version 2, A Geochronological Toolkit for Microsoft Excel*. Berkeley Geochronology Center Spec. Pub. 1a:47 p., Berkeley, CA, USA

2. U-Pb geochronology

Sample 16GC1051 (thin section) – Granitic pegmatite

The sample 16GC1051 hosts mm-scale subhedral to euhedral, zircon grains that exhibit in cathodoluminescence images sector and growth (i.e. oscillatory) zoning (**Fig. 1a-d**). The zircon grains are commonly fractured, host silicate inclusions, and are texturally overgrown by monazite grains. In one zircon grain, is observed an inherited component, expressed as dark core in CL imaging mode, and overgrown by a oscillatory zoned domain. The zircon grains are observed commonly in contact with mm-scale allanite grains. The three zircon grains that were analysed show variably high U contents from 352 to 1222 ppm and Th/U ratios from 0.44 to 0.79. The Th/U range falls within the range of zircons with magmatic origin but the extremely high U contents are indicative of metamict alteration. The examined grains are variably reversely discordant (-2.5% to -20.5%) with the two least discordant analyses yielding a $^{206}\text{Pb}/^{238}\text{U}$ date of 1085 ± 31 Ma (2σ , $n = 2$, $\text{MSWD} = 1.02$) that is tentatively suggested as the best approximate of zircon crystallization.

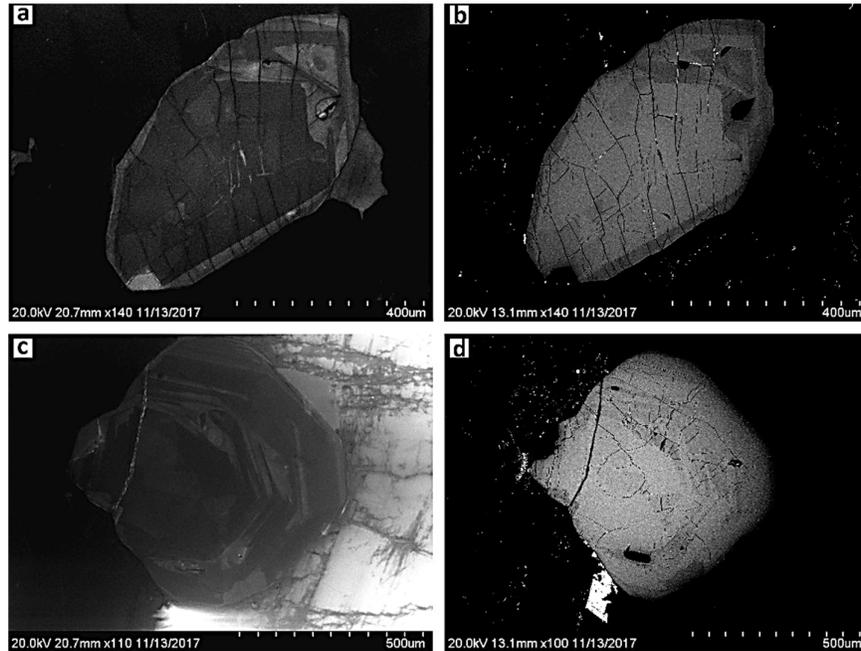


Figure 1. Cathodoluminescence (a and c) and backscatter electron (b and d) images of zircon grains from the sample 16GC1051. Note that the zoning patterns are best visible in the CL imaging mode

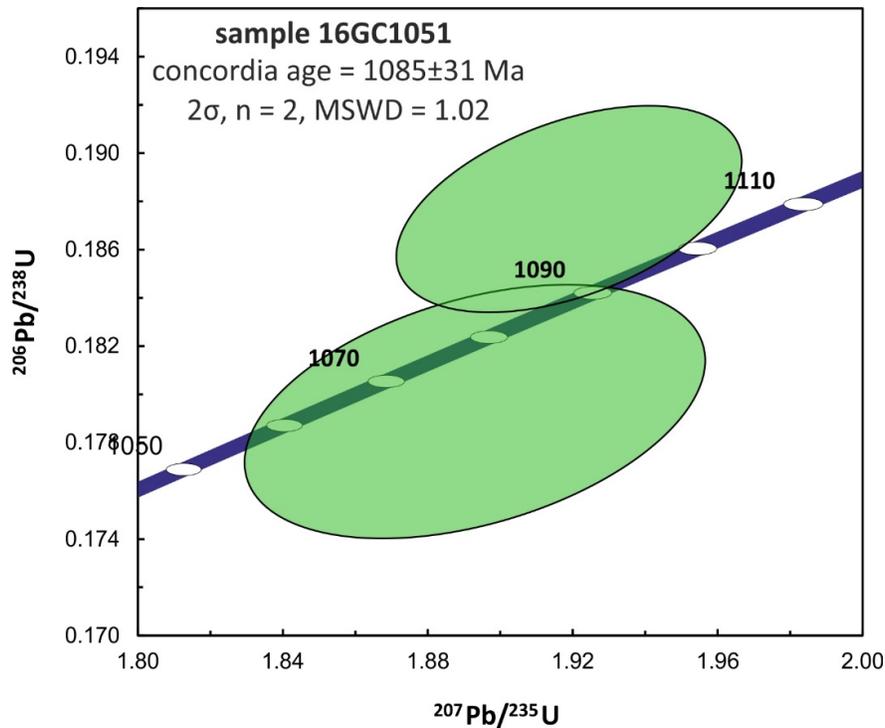


Figure 2. Concordia diagram that presents the two least discordant analyses of zircon grains from the sample 16GC1051

Sample 16GC1200 – Monzonite

Zircon grains from the sample 16GC1200 are subhedral to anhedral with aspect ratios 1:1 to 3:1. Internal CL imaging shows that the examined zircon grains show distinct zoning patterns with dark core domains, possibly locally xenocrystic (**Fig. 3c**), and overgrowths with oscillatory and sector zoning (**Fig. 3b-3f**). Resorbed domains with no zoning (**Fig. 3d-3g**) and thin rims (i.e. $<10 \mu\text{m}$) that are darker in cathodoluminescence images, are also commonly observed (**Fig. 3d – 3e**). Sixty-five ($n = 65$) U-Pb isotopic analyses targeted on domains with oscillatory zoning show that the U contents in these domains vary from 16 to 123 ppm with the Th/U ratios varying from 0.26 to 0.71. The U-Pb isotopic data plotted in concordia diagram define an upper intercept date of 1071 ± 11 Ma that is interpreted as the crystallization age of the zircon domains with magmatic, growth zoning. It is worth noting, that the U-Pb age dating of domains with different CL signature (e.g. dark cores, resorbed domains) yield U-Pb dates that overlap within uncertainty with the U-Pb date 1071 ± 11 Ma, indicating contemporaneous syn-magmatic processes expressed with distinct zoning patterns. The lower intercept is anchored to the date 0 ± 100 Ma assuming recent Pb loss.

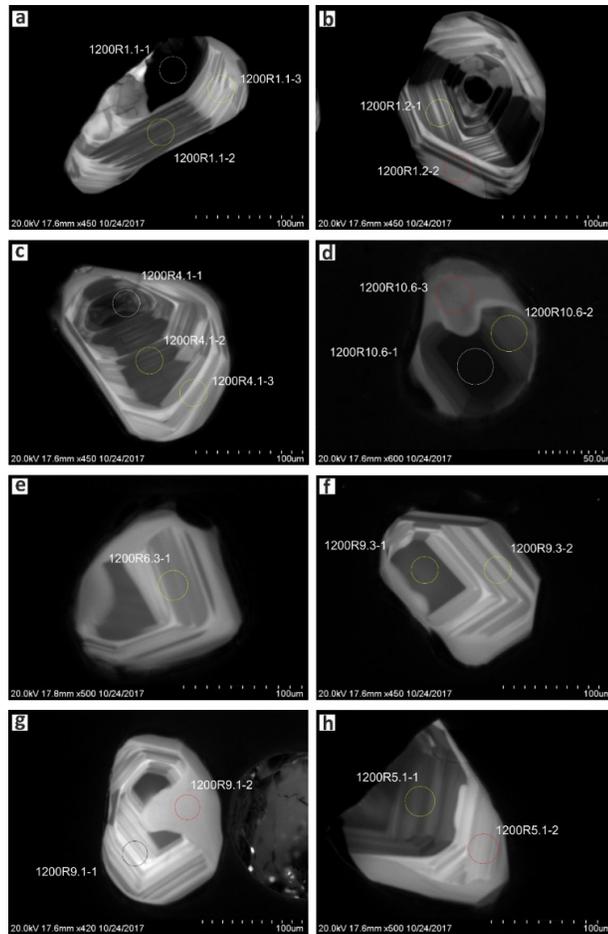


Figure 3. Cathodoluminescence images with spot domains for microanalysis in zircons from the sample 16GC1200

Sample 17AM061 - Syenogranite

Zircons grains from the sample 17AM061, are characterised in CL images, chiefly, by oscillatory and sector zoned domains indicative of magmatic crystallization and dark in CL metamorphic rims with thickness 10-20 μm (**Fig. 5**). Most of the grains are subhedral with aspect ratios 1:1 to 3:1. The oscillatory-zoned intragranular domains show U contents that vary from 27 to 140 ppm with Th/U ratios between 0.42 and 1.16, indicative also of magmatic petrogenetic origin. The U-Pb isotopic data yield in concordia space an upper intercept date of $1069 \pm 8.2 \text{ Ma}$ (2σ , $n = 65$, $\text{MSWD} = 1.3$) that is interpreted as the timing of primary crystallization taking in consideration the zoning patterns of the targeted analytical domains. The lower intercept is anchored to the date $0 \pm 100 \text{ Ma}$ assuming recent Pb loss. The majority of the analyses are sub-concordant with a few analyses slightly reversely discordant.

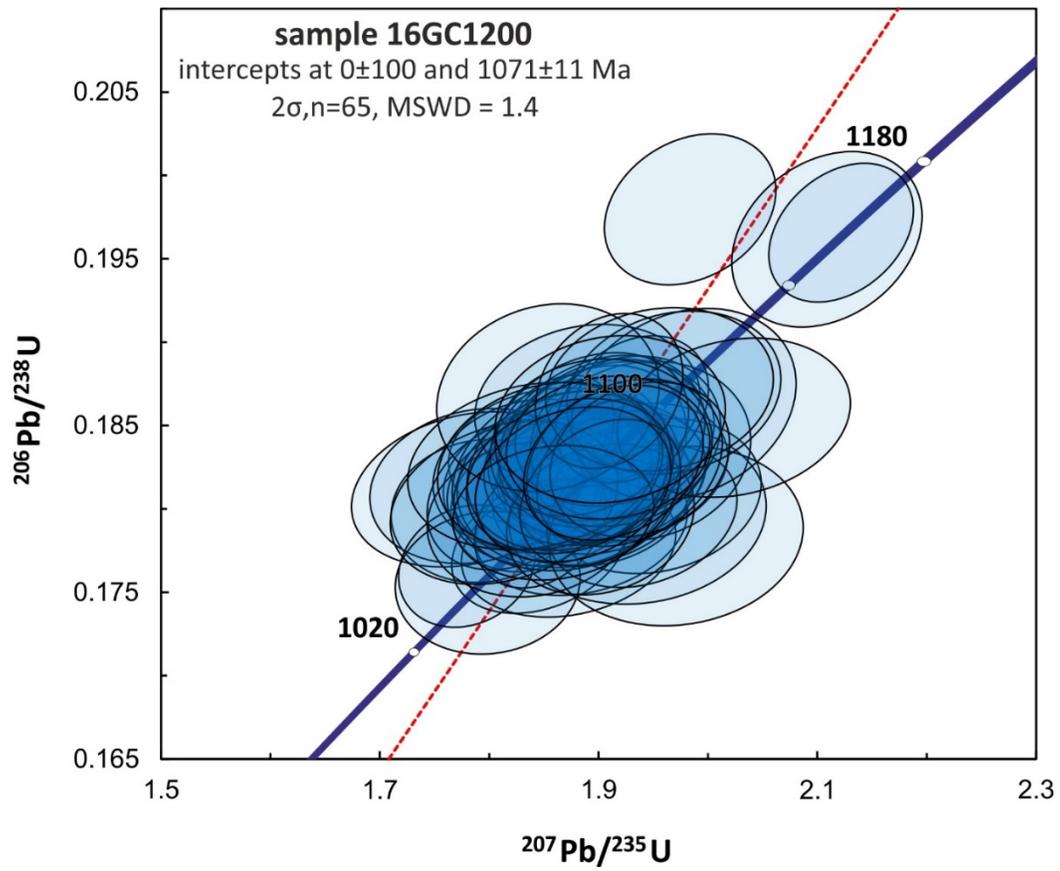


Figure 4. Concordia diagram with U-Pb analytical data from zircons grains of the sample 16GC1200

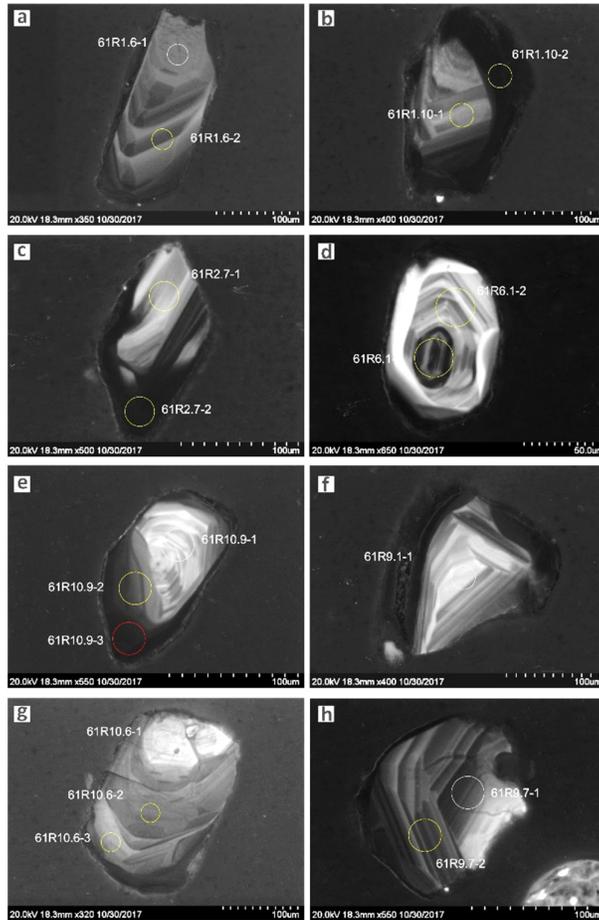


Figure 5. Cathodoluminescence images with spot domains for microanalysis in zircons from the sample 17AM061

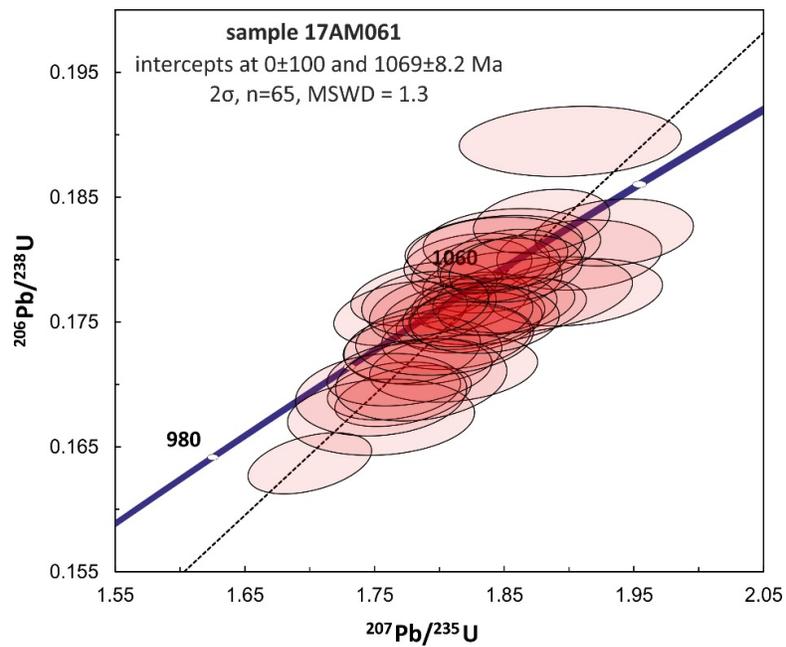


Figure 6. Concordia diagram with U-Pb analytical data from zircons grains of the sample 17AM061

Sample 17AM101 - Mangerite

Zircon grains from the sample 17AM101 exhibit dark cores in CL images with overgrowth domains characterised chiefly by oscillatory and sector zoning (**Fig. 7**). Zircon grains with turbulent zoning and resorbed domains are also observed (**Fig. 7c and 7e**). Thin metamorphic rims (<10 μ m) with dark CL signature is a ubiquitous feature in most of the grains. Morphologically, the zircon grains are euhedral to subhedral with aspect ratios 1:1 to 4:1. The Th/U ratios in the examined domains, that were selected for U-Pb dating, vary from 0.76 to 1.66 that indicate magmatic growth of these domains. The U-Pb microanalysis, of domains with oscillatory zoning, yielded an upper intercept date of 1076.2 ± 8.4 Ma anchored to present day Pb loss of 0 ± 100 Ma. This date, taking in consideration the zoning patterns and Th/U ratios is interpreted as the timing of primary igneous crystallization of the monzogranite.

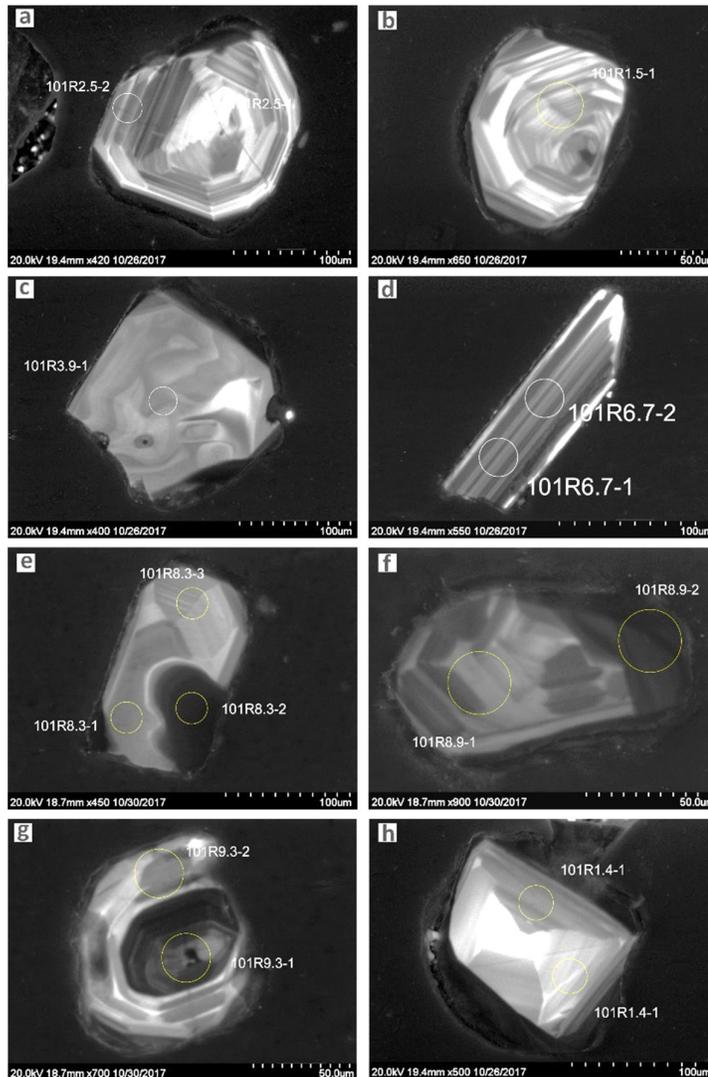


Figure 7. Cathodoluminescence images with spot domains for microanalysis in zircons from the sample 17AM101

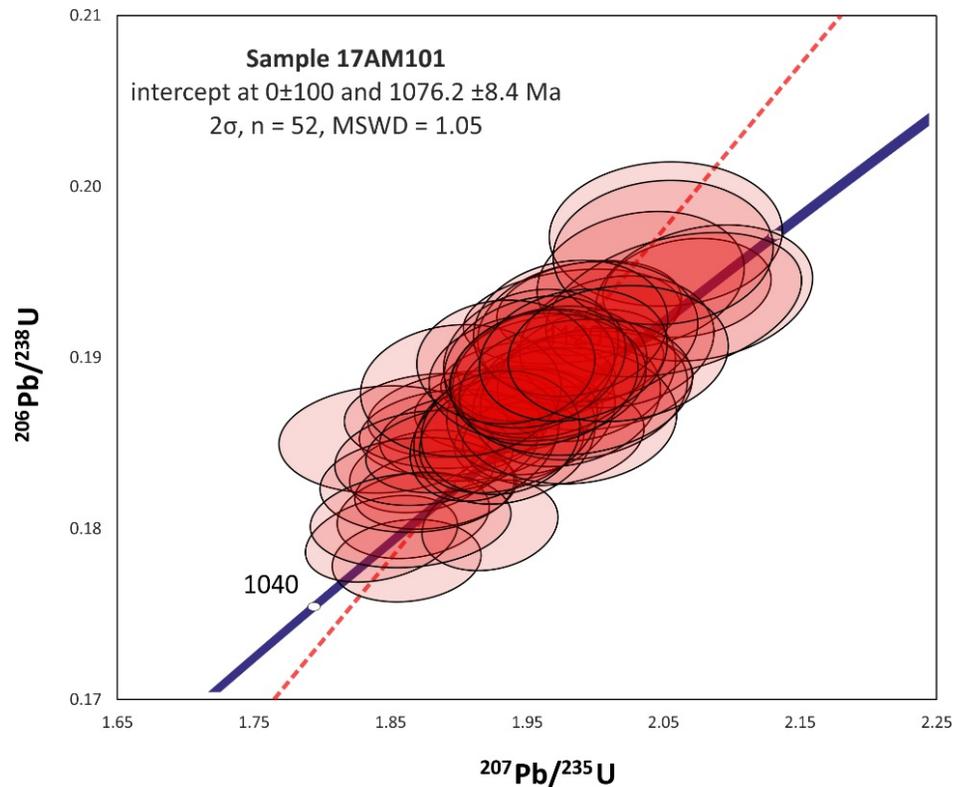


Figure 8. Concordia diagram with U-Pb analytical data from zircons grains of the sample 17AM101

Sample 17AM105 – Syenogranite

The sample 17AM105 contains chiefly subhedral zircon grains with aspect ratios 1:1 to 3:1. The CL imaging shows that the examined grains exhibit a variety of zoning patterns, commonly with mixed signature. In more detail, the majority of grains show oscillatory and sector zoned domains (**Fig. 9a-9d**) but also are observed grains with patchily zoned areas. It is worth noting the presence of intragranular domains with homogeneous dark CL signature and resorbed – amoeboid morphology (**Fig. 9a**). Metamorphic rims are not present in all of the examined grains, but where present, they have 5 to 10 μm thickness (**Fig. 9g**). The Th/U ratios of the analysed domains vary from 0.16 to 0.48 with U contents between 66.4 and 231.1 ppm. Analyses in domains that are characterised as dark cores or rims in CL images, show significantly lower Th/U ratios (i.e. 0.02 - 0.05; metamorphic origin) since the U contents of these domains are much higher (125 – 495 ppm). In the sample 17AM105 zircon grains with distinct CL domains are not systematically related with discrete age domains. The clustering though of U-Pb analyses, in concordia space, yields an age population of 1354.2 ± 9.5 Ma (upper intercept date; 2σ , $n=64$, $MSWD = 0.54$) anchored to 0 ± 100 Ma assuming recent Pb loss. The zircon rims define a younger concordia age of 1055 ± 13 Ma (2σ , $n = 3$, $MSWD = 3.5$). A tentative interpretation is that the upper intercept date represents an event of primary magmatic crystallization with the youngest age of the rim domains possibly representing a later metamorphic event.

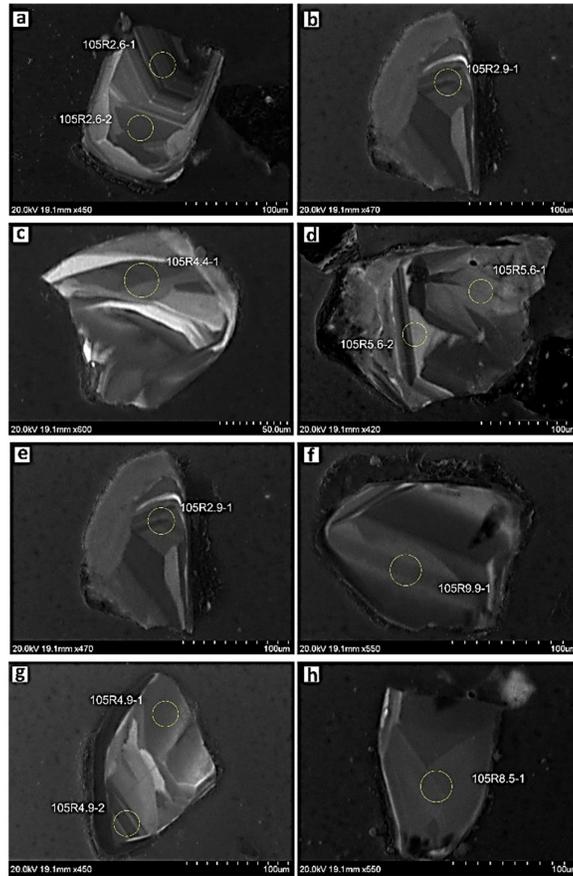


Figure 9. Cathodoluminescence images with spot domains for microanalysis in zircons from the sample 17AM105

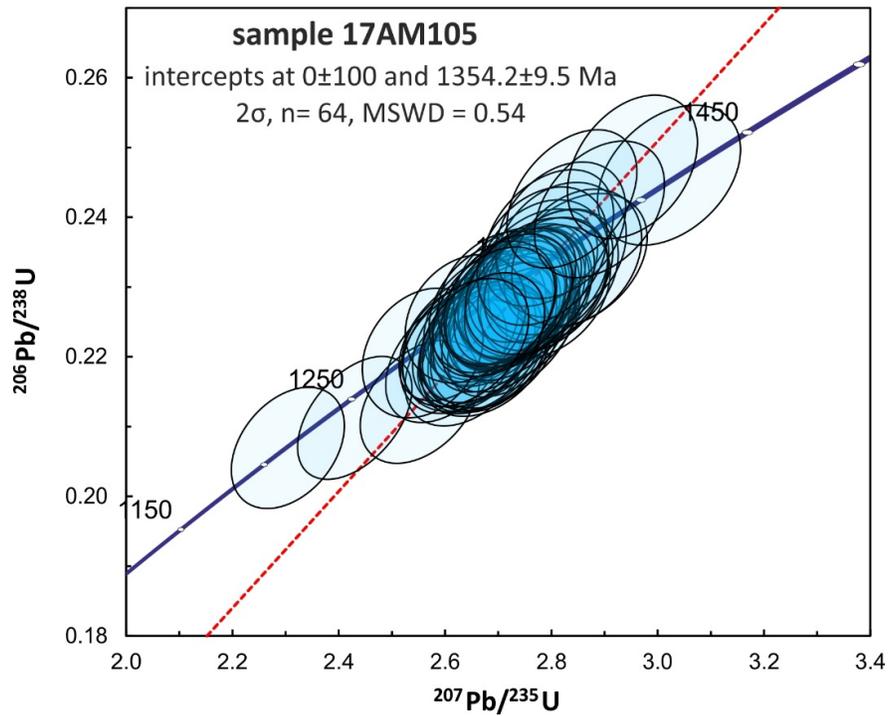


Figure 10. Concordia diagram with U-Pb analytical data from zircons grains of the sample 17AM105

Sample 17GC1055 – Orthopyroxene syenite

Zircon grains from the sample 17GC1055 are euhedral to subhedral with aspect ratios 1:1 to 4:1. The zircon grains in CL images show dark cores with oscillatory and sector zoned overgrowths (**Fig. 11**). Thin, dark in CL, metamorphic rims are not ubiquitously present as in zircon grains from the other samples. The Th/U ratio of the analysed grains vary from 0.32 to 0.98 with the U contents varying between 39.5 to 252.9 ppm. The zoning patterns in CL images do not systematically correlate with discrete age components in concordia space. Consequently, the majority of the analyses overlap within uncertainty and yield a concordia age of 1358.9 ± 3.5 Ma (2σ , $n = 67$, MSWD = 0.03; **Fig 12**). The latter date is interpreted as the timing of primary magmatic crystallization. The low MSWD indicates that the uncertainties in this case are overestimated since the data are far less variable than their assigned uncertainties.

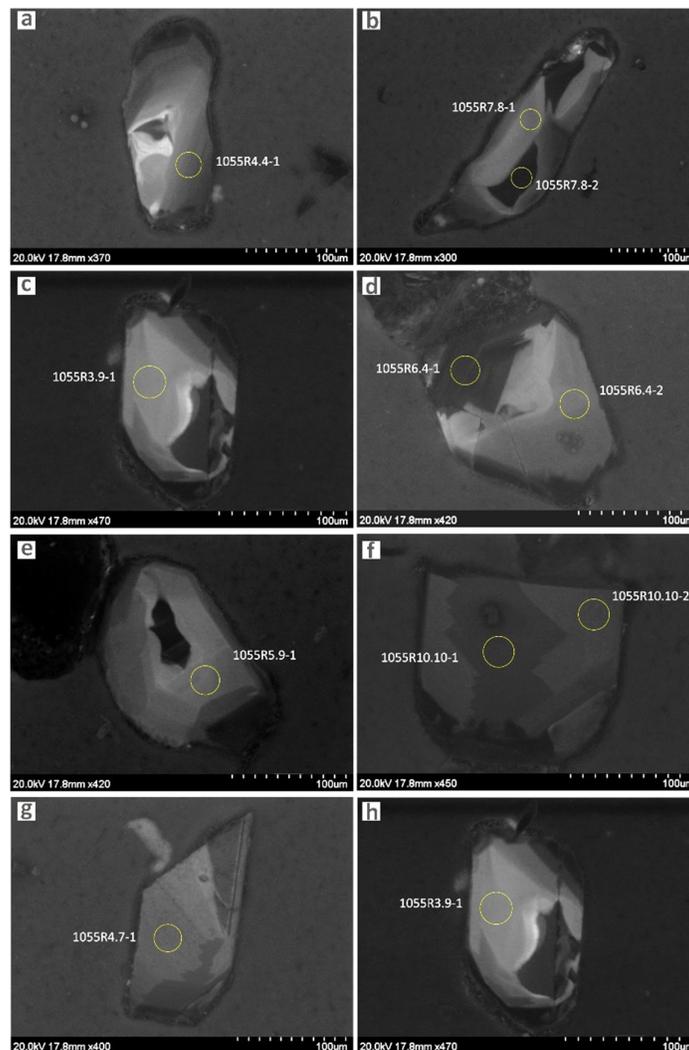


Figure 11. Cathodoluminescence images with spot domains for microanalysis in zircons from the sample 17GC1055

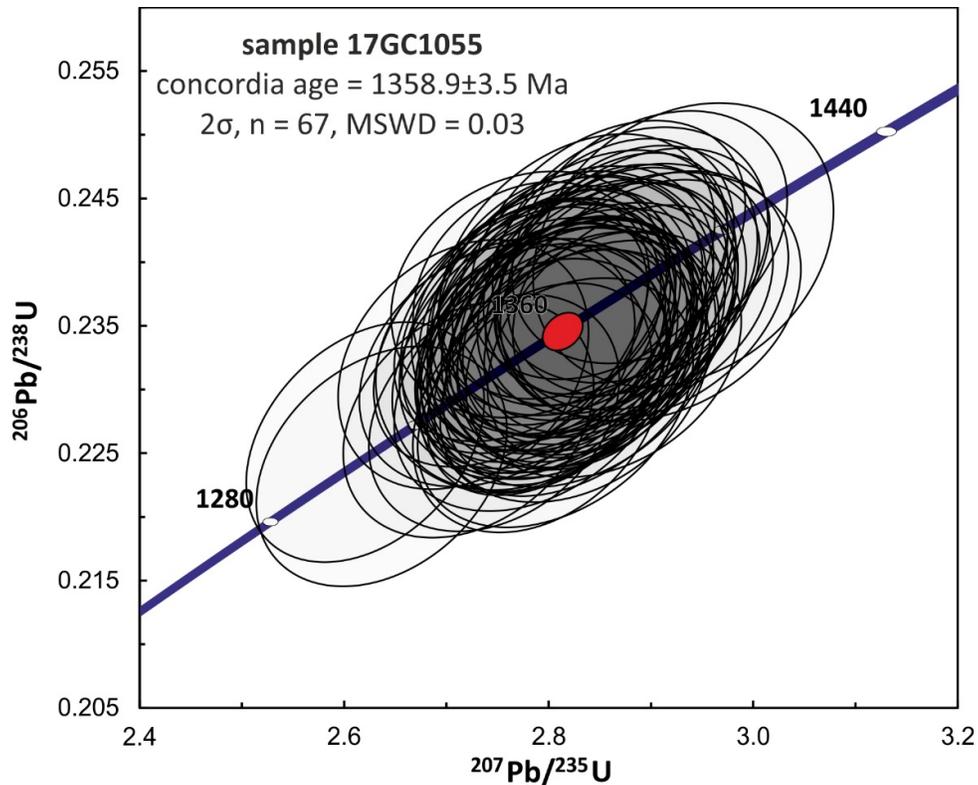


Figure 12. Concordia diagram with U-Pb analytical data from zircons grains of the sample 17GC1055

Sample 17GC1086 – Quartzite

The sample 17GC1086 contains zircon grains with aspect ratios 1:1 to 3:1 with most of the grains having long axis of ca. 80 – 100 μm . The majority of the imaged grains are rounded, and exhibit overgrowth domains with dark CL signature and brighter core domains with oscillatory and sector zoning (**Fig. 13a -13h**). The zircon grains show Th/U ratios that vary from 0.1 to 1.5 and U contents from 29 to 637 ppm. The U-Pb microanalysis of domains with sector or oscillatory zoning show that most of the analyses are up to 10% discordant, with analyses higher than 10% discordant not taken into consideration on the interpretation of the data. In U-Pb concordia space and Pb-Pb histograms (**Fig. 14**) the analyses show clustering with discrete peaks between 3 – 2.5 Ga, 1.9 – 1.8 Ga, 1.7 – 1.6 Ga, and 1.5 – 1.4 Ga. The highest peak in a relative probability Pb-Pb histogram, that represents the most abundant population of detrital zircons, is observed between 1.5 and 1.4 Ga (**Fig. 14**). These age populations agree, within uncertainty, with the whole rock Nd model ages that place constraints on the pre-collisional crustal evolution of the Grenville Province. On another note, the four youngest concordant detrital zircon grains yield a date of 1389 ± 22 Ma (2σ , n = 4, MSWD = 1.3), that represents

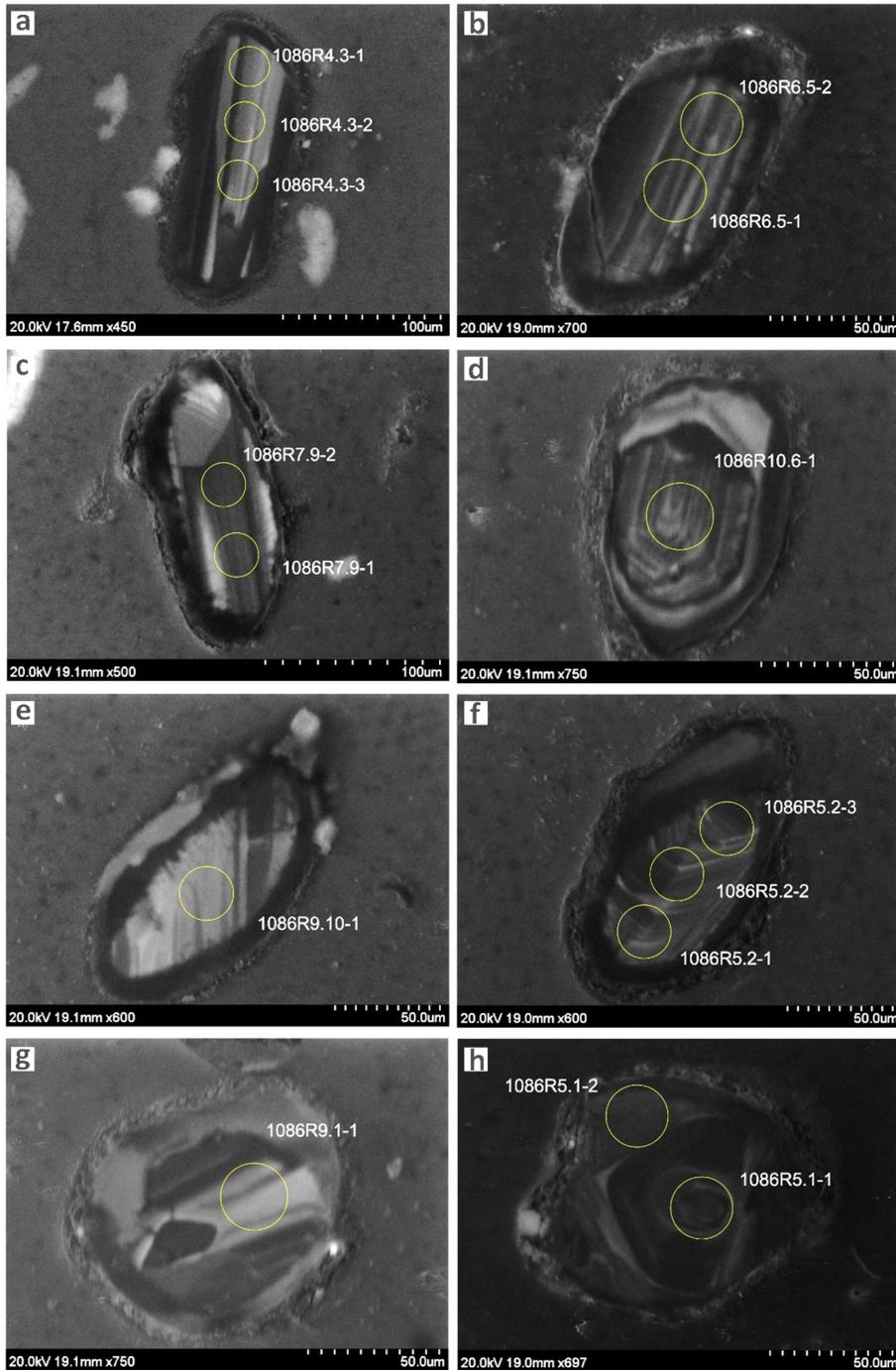


Figure 13. Cathodoluminescence images with spot domains for microanalysis in zircons from the sample 17GC1086

the maximum depositional age of the quartzite (**Fig. 15**). Taking into consideration that the sample is associated with the Montauban volcanoclastics then the date of ca. 1.39 place new age constraints on the deposition of supracrustal rocks in the Montauban basin. Preliminary results from additional analyses, with focus on the Hf isotopic composition of the examined zircon grains, are briefly mentioned in the section “complementary trace element and isotopic data”.

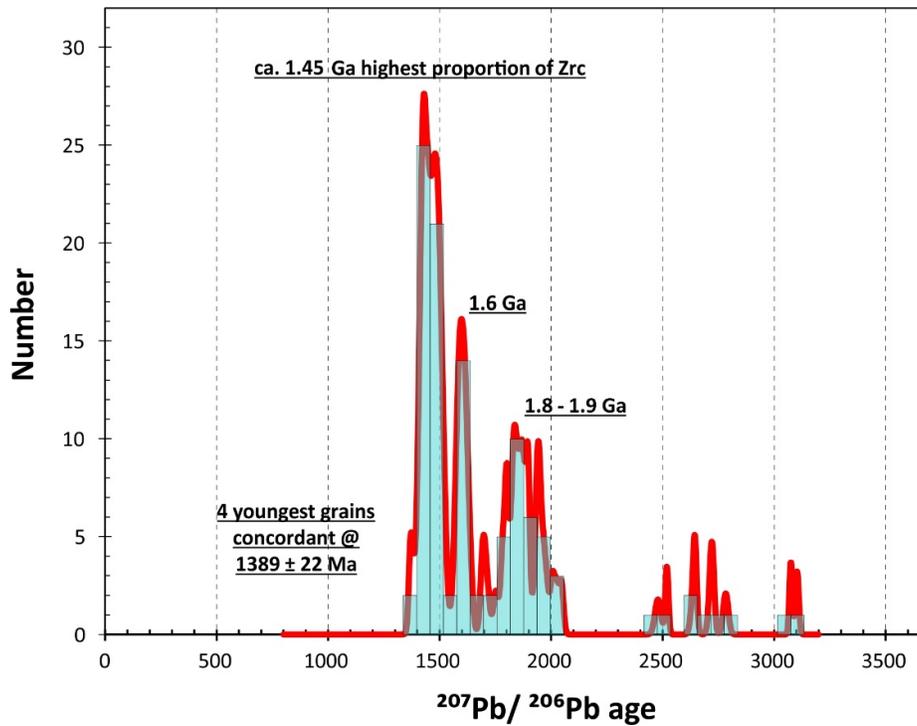


Figure 14. Pb-Pb histogram that shows the distribution of age populations in detrital zircons from the sample 17GC1086

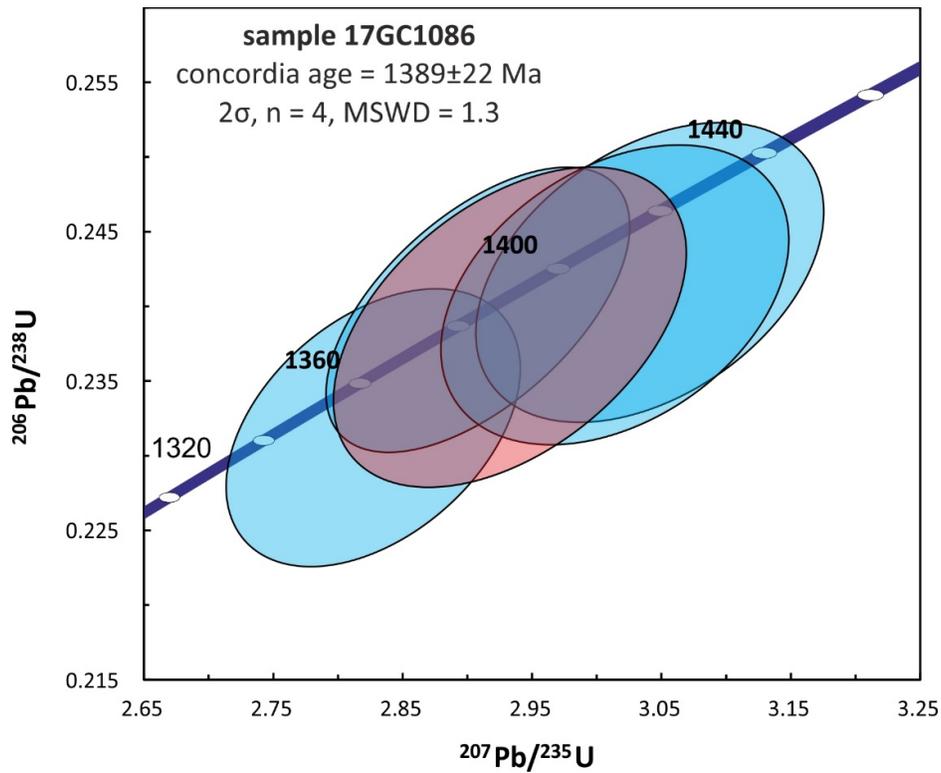


Figure 15. Concordia diagram with U-Pb isotopic data from the four youngest concordant zircons of the sample 17GC1086

Sample KP17LSZ2 – Migmatitic leucosome

The sample KP17LSZ is characterised by the assemblage: Opx-Kfs-Hbl-Ilm-Zrc±Opq±Mnz±Ap. The monazite and zircon grains are observed either as inclusion within orthopyroxene grains or within the feldspar-rich matrix. In more detail, the monazite grains exhibit patchy zoning and show distinct compositional domains that have embayed morphology in backscatter electron (BSE) images (**Fig. 16**). Qualitative microchemical mapping shows that the patchily zoned, brighter and darker, BSE domains are associated with variations in the abundance of Y (yttrium). The detected monazite grains in U-Pb concordia space are systematically reversely discordant yielding a U-Pb date of 1089 ± 15 Ma (2σ , $n = 36$, MSWD = 1.09) and a weighted average $^{207}\text{Pb}/^{206}\text{Pb}$ date of 1044 ± 10 Ma (2σ , $n = 36$, MSWD = 1.4; **Fig.17**). Reverse discordance is a common phenomenon in monazite that can be induced either by analytical (e.g. laser-mineral coupling) or geological factors (e.g. dissolution-precipitation reactions). Small spot size (i.e. $8\mu\text{m}$) LA-ICP-MS analysis of zircon grains detected in thin section from a sample of the Labelle shear zone yielded low precision results (upper intercept U-Pb date of 1348 ± 140 Ma). Despite the low precision, the latter date could be associated with zircon grains related with the emplacement of the pre-sheared protolith (e.g. Lacoste complex).

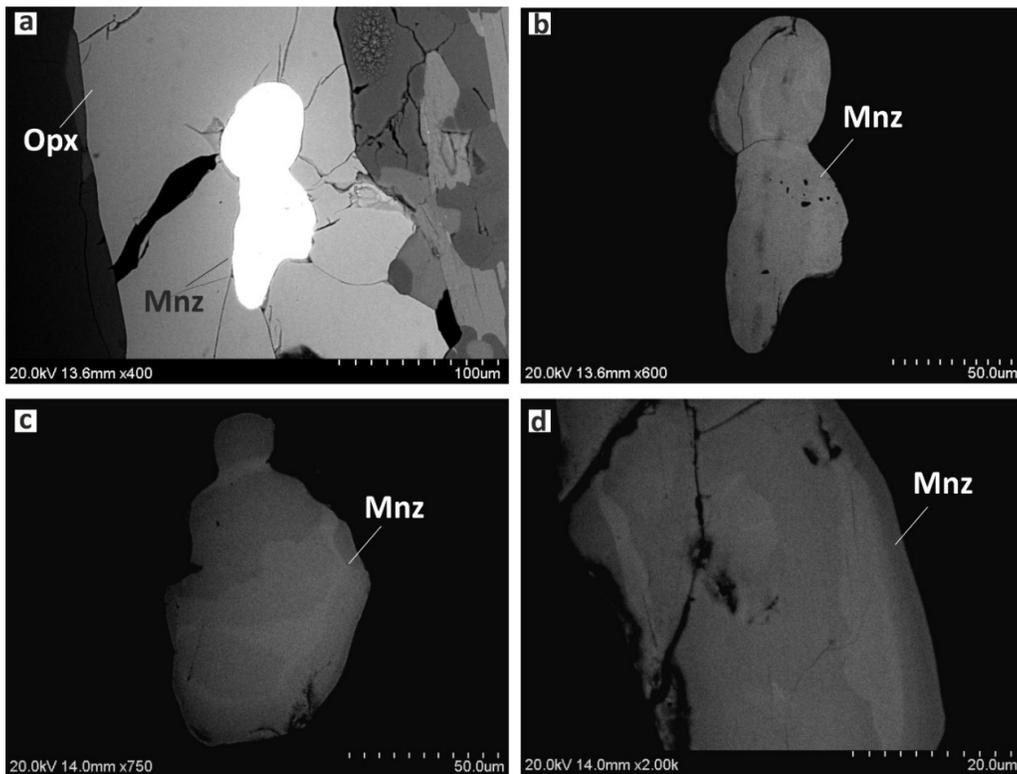


Figure 16. Backscatter electron images of monazite grains from the sample KP17LSZ

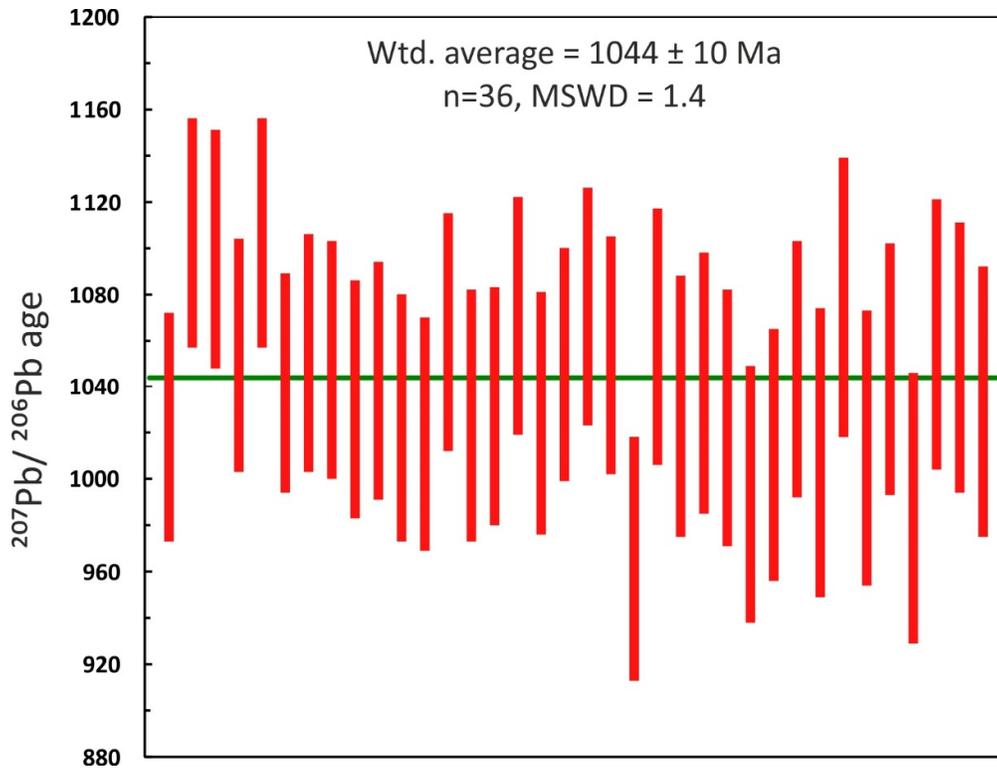


Figure 17. Weighted average Pb-Pb diagram from monazite grains of the sample KP17LSZ (Labelle shear zone)

3. Complementary trace element and isotopic data

The U-Pb isotopic dating of zircon grains from (metamorphosed) igneous lithologies shows two predominant age populations of ca. 1.06 – 1.08 Ga (samples 16GC1200, 17AM061, 17AM101) and 1.36 – 1.37 Ga (samples 17AM105 – 17GC1055). Trace element microanalysis of the same or adjacent zircon domains, that were selected for U-Pb dating, aims to compare crystallization temperatures of the zircon grains from the two groups (i.e. Ti-in-zircon thermometry), detect variations indicative of distinct petrogenetic processes using REE and other tracer element ratios (e.g. U/Yb, Nb/Yb), and explore in more detail potential effects of high-temperature metamorphism on trace element systematics. Moreover, the Hf isotopic composition of detrital grains shows that the latter are characterised predominantly by juvenile (mantle-derived) signature expressed by positive epsilon Hf (ϵHf) values. The latter constraints will provide a direct comparison with existing databases of whole-rock Nd model ages with main aim to understand better the pre-collisional accretionary stages of the Grenvillian orogeny.

**Supplementary report for samples
17AM105 and 17GC1055**

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GEOTOP-UQAM

Introduction

In this brief supplementary report are provided revised plots for the sample 17AM105 and complementary U-Pb isotopic data for the sample 17GC1055 with further explanations and descriptions of the acquired data.

Sample 17GC1055

For the sample **17GC1055** forty-six (n=46) new analyses were performed on fifty newly mounted zircon grains with main aim to verify the previous isotopic data (report of May 2018) that yielded a U-Pb concordant date of 1358.9 ± 3.5 Ma (2σ , n = 67, MSWD = 0.03). The U-Pb isotopic data were filtered based on the Th/U ratio assuming typical magmatic zircon Th/U ratios between 0.4 and 1 (Hoskin and Ireland., 2000), and the zoning pattern in CL images of each zircon grain. Moreover, during the picking of the zircon grains under the binocular microscope were selected grains with varying aspect ratios (i.e. long to short axis) and optical features. In the cathodoluminescence images the zircon grains exhibit either patchy or homogeneous dark-grey zoning (**Fig. 1**).

Overall, thirty analyses (n=30) with Th/U ratios between 0.42 and 0.91 were accepted with the majority of them being subconcordant with discordance values between -2.18 and 6.25. The thirty analyses yielded an upper intercept date of **1346.6 ± 9.9 Ma (Fig.2; 2σ , n=30, MSWD = 0.72)** assuming recent Pb loss and anchoring the lower intercept at 0 ± 100 Ma. This date is identical within uncertainty with the previous concordant U-Pb date of 1358.9 ± 3.5 Ma (2σ , n = 67, MSWD = 0.03). Relaxing the constraint of an anchored lower intercept at 0 ± 100 Ma (i.e. recent Pb loss) the analyses yield an upper intercept date of **1381 ± 54 Ma** (2σ , n = 30, MSWD = 0.54) with a lower intercept date of 714 ± 480 Ma.

Sample 17AM105

For the sample **17AM105** are provided new concordia plots with a revised interpretation of the data set. The U-Pb data plotted in the concordia diagram (**Fig.3**) define a discordia or mixing line with an upper intercept date of 1417 ± 37 Ma and lower intercept date of 988 ± 55 Ma. The U-Pb isotopic data were further inspected and filtered based on the Th/U ratios of the analysed domains, the zoning type in cathodoluminescence images, and the intragrain location of the analytical spots. The analytical domains with oscillatory or sector zoning and Th/U values higher than 0.4 yielded a concordant date of **1336.7 ± 9 Ma** (2σ , n=8, MSWD = 0.73) whereas zircon rims with Th/U < 0.05 (metamorphic signature) a concordant date of 1055 ± 13 Ma (2σ , n= 3, MSWD = 3.5). Is important to note that the

U-Pb dates of zircon grains with magmatic features in the samples 17AM105 and 17GC1055 overlap within uncertainty.

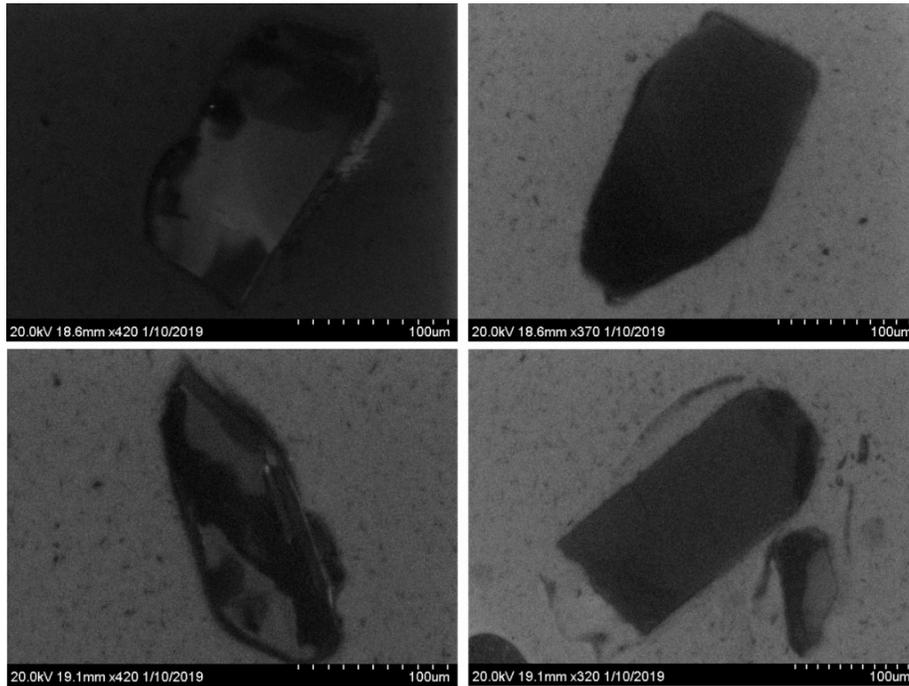


Figure 18. Representative cathodoluminescence images of zircon grains from the sample 17GC1055

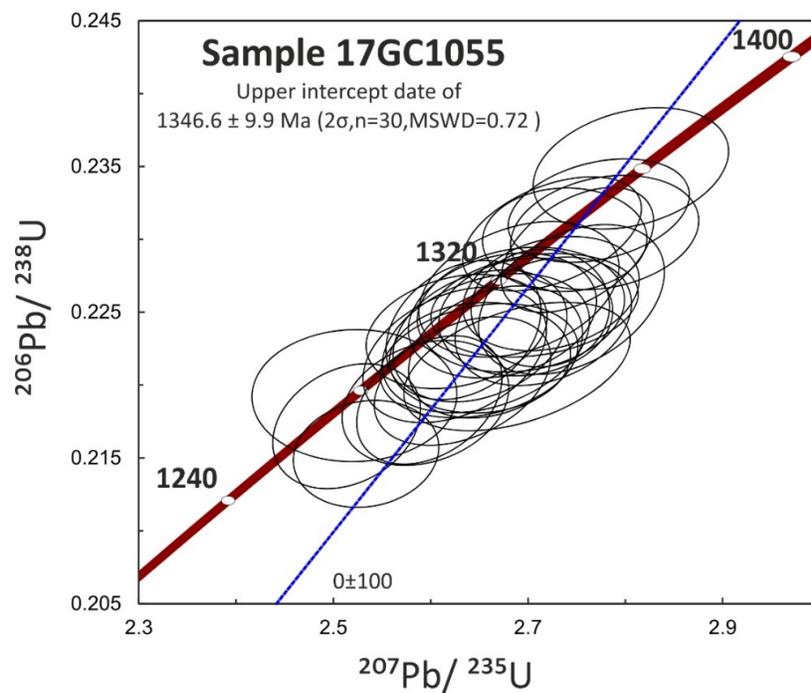


Figure 2. U-Pb concordia diagram for zircon grains of the sample 17GC1055

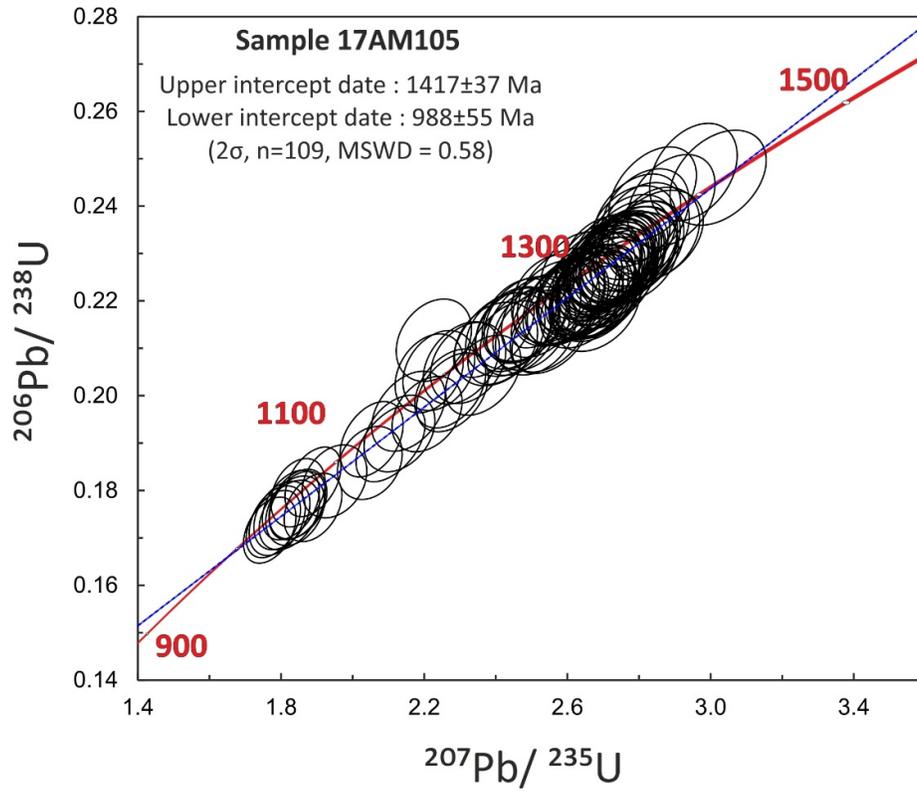


Figure 3. U-Pb isotopic data from all the selected zircons of the sample 17AM105

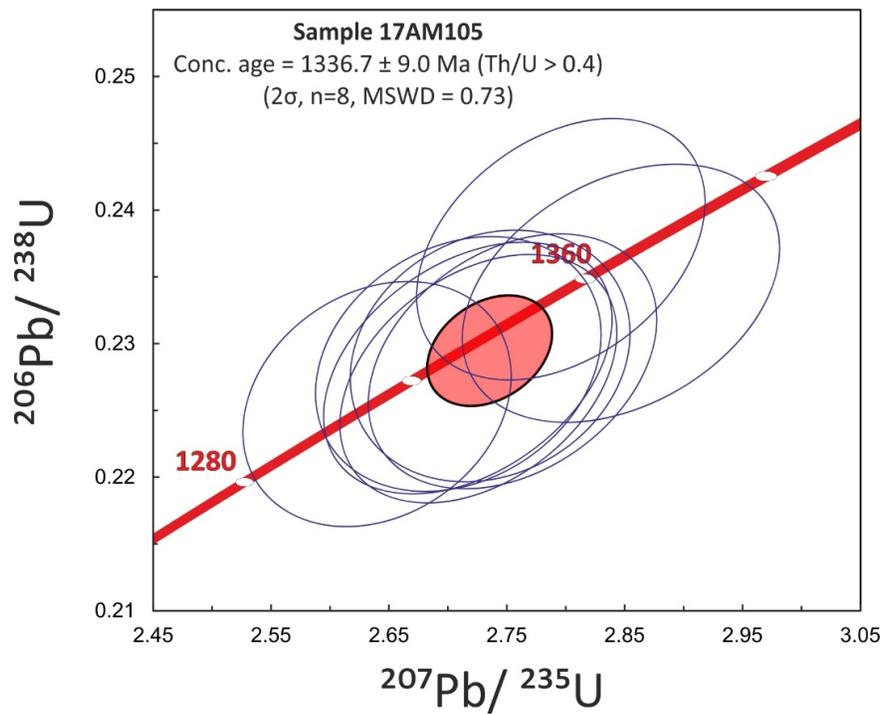


Figure 4. U-Pb concordia diagram from zircon grains of the sample 17AM105 that show magmatic Th/U values and oscillatory or sector zoning

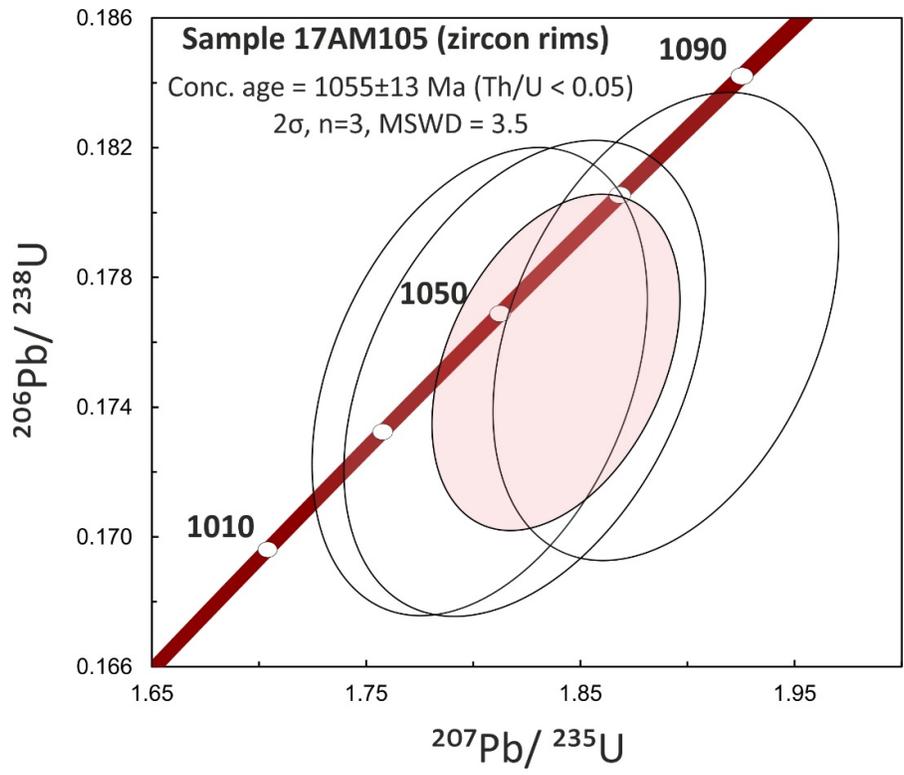


Figure 4. U-Pb isotopic data from rim domains of zircon grains of the sample 17AM105