

# Data description of EPMA, LA-ICP-MS U-Pb and bulk rock composition datasets of metapelites from the Austroalpine Unit north of Graz

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## 1. General description

### 1.1. Short description

This data publication contains the supporting datasets of the publication “Pressure-temperature-time and REE-mineral evolution in low to medium grade polymetamorphic units (Austroalpine Unit, Eastern Alps)” by Hollinetz et al. (2024) published in the European Journal of Mineralogy. We investigated 12 metapelites sampled in two nappes of the Austroalpine Unit north of Graz. The present datasets include (1) compositional data measured by electron probe microanalyzer (EPMA) of main phases (biotite, chlorite, chloritoid, garnet, ilmenite, plagioclase, staurolite and white mica; Table S1-S8) and rare earth element (REE)-bearing phases (allanite, REE-bearing epidote, monazite, xenotime, thorite and apatite; Tables S9-S11), (2) LA-ICP-MS U-Th-Pb data of allanite and monazite (Tables S12a-d) and (3) effective bulk rock composition data for calculation of pseudosection diagrams (Tables S13-S15).

## 1.2. Content of the data publication

1. EPMA data of biotite	TableS1_EPMA_Bt.csv
2. EPMA data of chlorite	TableS2_EPMA_ChI.csv
3. EPMA data of chloritoid	TableS3_EPMA_Cld.csv
4. EPMA data of garnet	TableS4_EPMA_Grt.csv
5. EPMA data of ilmenite	TableS5_EPMA_Ilmen.csv
6. EPMA data of plagioclase	TableS6_EPMA_Pl.csv
7. EPMA data of staurolite	TableS7_EPMA_St.csv
8. EPMA data of white mica	TableS7_EPMA_Ms_Pg.csv
9. EPMA data of allanite and epidote	TableS9_EPMA_Aln.csv
10. EPMA data of monazite, xenotime and thorite	TableS10_EPMA_Mnz_Xtm_Thr.csv
11. EPMA data of apatite	TableS11_EPMA_Ap.csv
12. LA-ICP-MS data of allanite in sample Hi-1	TableS12a_LAICPMS_AlnHi-1.csv
13. LA-ICP-MS data of allanite in sample Ra-3	TableS12b_LAICPMS_AlnRa-3.csv
14. LA-ICP-MS data of allanite in sample Ra-4	TableS12c_LAICPMS_AlnRa-4.csv
15. LA-ICP-MS data of monazite in sample Ro-2	TableS12d_LAICPMS_MnzRo-2.csv
16. Bulk rock composition	TableS13_bulk.csv
17. Bulk rock composition of sample Hi-1	TableS14_bulkHi-1.csv
18. Bulk rock composition of sample Hi-1	TableS15_bulkRo-1.csv
19. Sample coordinates	TableS16_samples.csv

## 2. Methodology

### 2.1 Electron microprobe analysis

Quantitative mineral chemistry analyses were determined on polished thin sections using a field emission gun Cameca SXFiveFE electron probe microanalyzer (EPMA) at the University of Vienna (Austria). The instrument is equipped with a Schottky field emission gun electron source and five wavelength-dispersive spectrometers (WDS). Operating conditions were 15 kV acceleration voltage and a beam current of 20 nA for garnet, staurolite, chloritoid, ilmenite and plagioclase. Phyllosilicates were measured with a beam current of 10 nA and a defocused beam diameter of 5  $\mu\text{m}$  to minimize beam damage. Plagioclase was measured with a defocused beam diameter of 7  $\mu\text{m}$  and 10 s peak counting time to avoid Na-loss. Counting times for other elements are 20 and 10 s at peak position and background, respectively. Natural and synthetic minerals were used as standards (Ntaflos et al., 2017). Major element compositions of biotite, chlorite, chloritoid, garnet, ilmenite, plagioclase, staurolite and white mica and calculated mineral formulae are reported in Tables S1-S8. Major and trace element compositions of allanite, monazite, xenotime and thorite were analyzed using 20 kV acceleration voltage and 100 nA beam current. For allanite, the beam was defocused to 3-5  $\mu\text{m}$ . La, Ce, Nd, Eu, Dy, and Y were measured using the  $L\alpha$  line and

25 s peak counting time. Pr, Sm, Gd and Er were measured using the L $\beta$  line and 30 s peak counting time. For Th and U, the M $\alpha$  and M $\beta$  lines and 35.5 s and 30 s peak counting time were used, respectively. For Pb, the L $\alpha$  line was measured for 40 s at peak position at two spectrometers simultaneously. For fluorapatite analyses 20 kV acceleration voltage, 20 nA beam current and a defocused beam diameter of 8  $\mu$ m was used. Na, F and Cl were measured first with 10 s peak counting time. Synthetic glasses were used for calibration. Results of REE-minerals are in Tables S9-S11. The PAP method (Pouchou and Pichoir, 1991) was used for matrix correction for all data. The laboratory internal standard yields a relative error below 1% (Ntaflos et al., 2017).

## 2.2 Laser ablation inductively coupled plasma mass spectrometry (LA-ICP-MS)

The U-Th-Pb analyses were performed using a Resonetics M-50 193 nm ArF Excimer laser ablation system coupled to an Agilent 7700x quadrupole ICP-MS system at the University of New Brunswick (UNB), Canada, following the protocol of McFarlane and Luo (2012). Samples and standards were loaded in a Laurin Technic Pty S-155 two-volume cell with a working ablation volume of < 1 cm<sup>3</sup>. Reference material NIST610 with isotope values for Pb/U from Horn and von Blanckenburg (2007) was used as primary standard. Prior to each analytical session, tuning of the ICP-MS on NIST610 glass to achieve  $^{248}\text{ThO}/^{232}\text{Th} < 0.2\%$ ,  $^{238}\text{U}/^{232}\text{Th} \sim 1.05$ , and  $^{44}\text{Ca}^{++}/^{44}\text{Ca}^{+} \leq 0.2\%$  was carried out to maximize sensitivity and minimize interference-related biases in final corrected ages.

Target grains were localized based on scanning electron microscopy (SEM) documentation in combination with micro X-ray fluorescence compositional maps produced by a Bruker M4 Tornado (UNB). Allanite / REE-epidote was analyzed using a 24  $\mu$ m crater diameter, 1.2 to 1.5 J/cm<sup>2</sup> laser fluence, 3 Hz repetition rate and 25 s of sample ablation preceded by 30 s background collection. Allanite was standardized against NIST610 according to the protocol of McFarlane (2016). To monitor reproducibility, Siss3 allanite (c. 33 Ma, Oberli et al., 2004) and Hartfield intrusion allanite (c. 414 Ma, McFarlane, 2016) were used as reference material. Monazite was ablated using a 10  $\mu$ m crater diameter, 3 J/cm<sup>2</sup> laser fluence, 4 Hz repetition rate and 30 s background measurement preceding 30 s sample ablation. GSC8153 monazite (c. 507 Ma) was used as a primary standard and 44069-monazite (c. 425 Ma, Aleinikoff et al., 2006) was used as a secondary standard. The ablated material was transferred in a carrier gas composed of 930 mL/min Ar and 300 mL/min He via a Laurin Technic Pty Peek™ Y-connector and ‘squid’ smoothing device to the ICP-MS. The following masses were analyzed with dwell times in ms given in brackets:  $^{202}\text{Hg}$  (20),  $^{204}\text{Pb}$ , Hg (60),  $^{206}\text{Pb}$  (50),  $^{207}\text{Pb}$  (70),  $^{208}\text{Pb}$  (10),  $^{232}\text{Th}$  (10) and  $^{238}\text{U}$  (20). Additionally, the following masses were monitored using a dwell time of 10 ms:  $^{31}\text{P}$ ,  $^{44}\text{Ca}$   $^{49}\text{Ti}$ ,  $^{56}\text{Fe}$ ,  $^{90}\text{Zr}$ ,  $^{89}\text{Y}$ ,  $^{139}\text{La}$  and  $^{146}\text{Nd}$ . Concentrations of these

elements are calculated without internal standard but are typically within 20% of the true value, thus sufficient for the purpose of detecting inclusions and assigning individual U-Pb analyses to chemical domains of the analyzed unknowns.

Data reduction was carried out offline using *Iolite* v3.7 (Paton et al., 2011) in combination with *VizualAge* v2015.06 (Petrus and Kamber, 2012) running under *Wavemetrics IgorPro* v6.22. The U-Pb geochronology data reduction scheme of *Iolite* (Paton et al., 2010) performs a down-hole Pb/U fractionation correction, followed by a drift correction, reference material normalization and uncertainty propagation. Analyses exhibiting obvious heterogeneity in the time-resolved U-Pb concentrations were removed during this step. Common Pb corrected values for monazite of sample Ro-2 based on measured net  $^{204}\text{Pb}$  were calculated using *Isoplot* 3.71 (Ludwig, 2012).

### 2.3 Bulk rock composition for pseudosection calculation

Molar proportions of the effective bulk rock composition used as input for pseudosection calculations (Table S13) are derived from whole rock analyses. Samples selected for whole rock analyses were cleaned from macroscopic signs of alteration, crushed and ground to a fine powder using an agate disc mill. Major and trace elements were determined by inductively coupled plasma mass spectrometry (ICP-MS) of fused beads at Activation Laboratories Ltd. (Ancaster, Canada) using the 4 Litho package. Analyses of internal duplicates yield analytical uncertainties of <2.6% for major elements (except MnO, 3.4%) and <7.6% for REEs, Th and U. Results of whole rock analyses are reported in Table 2 in Hollinetz et al. (in press). Sample Hi-1 exhibits small-scale compositional layering which indicates limited equilibration, therefore the effective bulk composition was calculated from average mineral compositions measured by EPMA and mineral proportions estimated from petrography (Table S14). For sample Ro-1 we calculated a bulk rock composition considering 2.5 mol% garnet fraction (denoted as Ro-1\* in Table S15), following Evans (2004). The composition of the fractionated garnet corresponds to average compositions of the garnet core (XAlm – 0.81, XSps – 0.06, XPrp – 0.08, XGrs – 0.05).

Due to the lack of  $\text{Fe}^{3+}$ -bearing oxides (e.g. magnetite, hematite) and very minor amounts of  $\text{Fe}^{3+}$  calculated in the structural formulae of chloritoid, staurolite, garnet and ilmenite, we assume reducing conditions at the metamorphic peak, consistent with the common occurrence of carbonaceous material. Therefore, the amount of O was calculated assuming all Fe is present as  $\text{Fe}^{2+}$ . To calculate the effective Ca concentration ('Ca corrected' in Table S13), the measured CaO content was corrected to account for CaO corresponding to apatite, which is not considered in the model calculations (Hollinetz et al., 2022). This approach yields negative Ca values for some samples (Hi-4, Hi-5, Hi-6), which show evidence of open system behavior of

Ca postdating the metamorphic peak. Given the uncertainties related to the effective bulk Ca concentration at the metamorphic peak, we decided to exclude Ca entirely from the compositional space for the Hirschkogel sample group (Hi-1 to Hi-6). The final Ca content used for pseudosection calculations is given in the column 'Ca\*' in Table S13. For more details on choices regarding the compositional space, refer to Hollinetz et al. (in press).

### 3. Detailed descriptions of datasets

#### 3.1. General remarks and abbreviations

Mineral abbreviations are used after Warr (2021): allanite – Aln, apatite – Ap, biotite – Bt, chlorite – Chl, chloritoid – Cld, epidote – Ep, ilmenite – Ilm, monazite – Mnz, muscovite – Ms, paragonite – Pg, plagioclase – Pl, quartz – Qz, rutile – Rt, staurolite – St, thorite – Thr, xenotime – Xtm, zircon – Zrn.

The EPMA datasets (Tables S1-11) contain EPMA measurements in oxide weight percent (wt%) left of the column 'Total', followed by the calculated structural formulae in atoms per formula unit (apfu). Values that are below detection limit (bdl) or not determined (nd) for all samples are indicated. Al<sub>IV</sub> and Al<sub>VI</sub> correspond to Aluminium in the tetrahedral and octahedral lattice position, respectively. Rare earth elements (REE) are grouped in light (LREE), medium (MREE) and heavy (HREE) subgroups. Molar proportions indicated by the variable X are defined as  $X_{Mg} = Mg/(Mg+Fe)$  and  $X_{Na} = Na/(Na+K+Ca)$ . The column 'type' in EPMA datasets refers to different mineral types that were identified based on SEM documentation. Mineral types may refer to microstructural characteristics such as the orientation with respect to planar fabrics (denoted as S1, S2, S3 or S<sub>n</sub>), the relationship between the analyzed mineral and a different phase (e.g. replacement, inclusion) or indicate if several populations of the analyzed mineral exist (e.g. Cld1, Cld2). See Hollinetz et al. (in press) for detailed explanation on the significance of the various types.

The LA-ICP-MS datasets (Tables S12a-d) contain approximate concentrations of additional elements in parts per million (ppm) and isotopic ratios and error correlations ( $\rho$ ) for Concordia diagrams in Wetherill and Tera-Wasserburg space. The allanite datasets (Table S12a-c) additionally contain isotopic ratios for the joint isochron regression method (Vermeesch, 2020). The presence of inclusions inferred from elevated Ti or Zr contents is indicated in the column 'inclusion'. Moreover, analyses were grouped in populations according to microstructural or chemical properties of the target mineral (see Hollinetz et al., in press for details). The full datasets, including analyses that were omitted from age calculations in Hollinetz et al. (in

press) are reported. Omitted analyses are indicated in the columns 'rejected' (Table S12a-c) and 'comment' (Table S12d).

### 3.2. Content of the datasets

#### **Table S1**

Table S1 contains biotite EPMA data in wt% . Structural formulae [apfu] are calculated for 11 oxygen using MINSORT (Petrakakis and Dietrich, 1985). M1 is the trioctahedral mica component; A is the sum of K + Na + Ca on the interlayer site.

#### **Table S2**

Table S2 contains chlorite EPMA data in wt%. Structural formulae [apfu] and endmember proportions (XClc – clinochlore, XAme – amesite, XSud – sudoite) are calculated for 14 oxygen following the method of Vidal et al. (2005).

#### **Table S3**

Table S3 contains chloritoid EPMA data in wt%. Structural formulae [apfu] are calculated for 8 cations using MINSORT (Petrakakis and Dietrich, 1985). R3 and R2 is the sum of trivalent and divalent cations, respectively. Fe<sub>2</sub>O<sub>3</sub> was determined from stoichiometry.

#### **Table S4**

Table S4 contains garnet EPMA data in wt%. Structural formulae [apfu] and endmember proportions (XAlm – almandine, XSps – spessartine, XPrp – pyrope, XGrs – grossular, XAdr – andradite) are calculated for 12 oxygen using MINSORT (Petrakakis and Dietrich, 1985). Fe<sub>2</sub>O<sub>3</sub> is estimated iteratively until sum of trivalent cations is 2. Measurements corresponding to the profile shown in Fig. 11a in Hollinetz et al. (in press) are highlighted.

#### **Table S5**

Table S5 contains ilmenite EPMA data in wt%. Structural formulae [apfu] are calculated for 3 oxygen. Some analyses that show anomalously high Ti contents indicating alteration were excluded from Fig. 11b in Hollinetz et al. (in press).

#### **Table S6**

Table S6 contains plagioclase EPMA data in wt%. Structural formulae [apfu] and endmember proportions (XAn – anorthite, XAb – albite, XOr – orthoclase) are calculated for 8 oxygen using MINSORT (Petrakakis and Dietrich, 1985).  $\text{Fe}_2\text{O}_3$  is determined from stoichiometry, assuming total Fe is present as  $\text{Fe}^{3+}$ .

#### **Table S7**

Table S7 contains staurolite EPMA data in wt%. Structural formulae [apfu] are calculated for 46 oxygen using MINSORT (Petrakakis and Dietrich, 1985). R3 and R2 is the sum of trivalent and divalent cations, respectively.  $\text{Fe}_2\text{O}_3$  is determined from stoichiometry.

#### **Table S8**

Table S8 contains white mica EPMA data in wt%. Structural formulae [apfu] and endmember proportions (XMs – muscovite, XPg – paragonite, XCel – celadonite, XPrl – pyrophyllite, XTri – trioctahedral white mica, XMrg – margarite, XTims – Ti in muscovite) are calculated for 11 oxygen following the method of Parra et al. (2002).

#### **Table S9**

Table S9 contains allanite and REE-epidote EPMA data in wt%. Structural formulae [apfu] are calculated following the approach of Ercit (2002) that accounts for vacancies (vac) on the A site, with the following site occupancies: T site: Si - As, M site: Ti - Mg. A site: Ca – vac.

#### **Table S10**

Table S10 contains monazite, xenotime and thorite EPMA data in wt%. Structural formulae [apfu] are calculated for 4 oxygen. TGH98 is the temperature calculated with the monazite thermometer of Gratz and Heinrich (1997) at a pressure of 0.4 GPa. TSG02 is the temperature calculated with the monazite thermometer of Seydoux-Guillaume et al. (2002) at a pressure of 0.2 GPa.

#### **Table S11**

Table S11 contains apatite EPMA data of sample Ra-4 in wt% (bdl – below detection limit). Structural formulae [apfu] are calculated following Ketcham (2015). Anomalously high F contents and negative values for OH are due to beam damage.

#### **Table S12a**

Table S12a contains results of LA-ICP-MS U-Th-Pb geochronology of allanite in sample Hi-1.

**Table S12b**

Table S12b contains results of LA-ICP-MS U-Th-Pb geochronology of allanite and REE-epidote in sample Ra-3.

**Table S12c**

Table S12c contains results of LA-ICP-MS U-Th-Pb geochronology of allanite and REE-epidote in sample Ra-4.

**Table S12d**

Table S12d contains results of LA-ICP-MS U-Th-Pb geochronology of monazite in sample Ro-2. The fraction of radiogenic Pb is given in the column 'Pb\* [%]'. The  $^{204}\text{Pb}$  correction is applied to common Pb corrected values (Pbc). Analyses that were discordant after the  $^{204}\text{Pb}$  correction are rejected. Chemical domain refers to the composition on a La-Th-U ternary diagram. See Figure 13b in Hollinetz et al. (in press) for significance of 'mixed' and 'chemical outlier'.

**Table S13**

Table S13 contains effective bulk rock compositions [mol] as input for pseudosection calculation. See section 2.3 for details on the calculation method.

**Table S14**

Table S14 contains estimated minerals modes [mol%] and average phase compositions [w%] measured by EPMA used for calculation of Hi-1 effective bulk rock composition with the Rock Maker spreadsheet (Büttner, 2012). The density was taken from the Rock Maker mineral dataset.

**Table S15**

Table S15 shows the calculation of the bulk composition of sample Ro-1 considering 2.5 mol% garnet fractionation (denoted as 'Ro-1\*' in Table S13).

**Table S16**

Table S16 contains sample names, coordinates in UTM N33 (EPSG 32633), lithostratigraphic and tectonic units and lithologies. 'Alias' is the sample name in the database of the Geosphere Austria. Samples KP02, KP06 and KP07 are from Schantl et al. (2015).



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