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# LITHOS

## Cryptic crustal growth identified through Variscan post-collisional lamprophyre-granite composite dykes, French Massif Central. --Manuscript Draft--

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| Abstract:             | <p>The processes that control crustal growth by addition of new mantle-derived material to the crust and the recycling of crustal components that were introduced into the mantle during subduction remain poorly understood in collisional orogens. This is largely due to the fact that the sub-orogenic mantle has been fertilized by the introduction of crustal components during previous subduction events, and lacks the typical depleted mantle signature. Here, we present a study of lamprophyric-granitic composite dykes from the northern border of the post-collisional Variscan Aigoual pluton, French Massif Central (FMC). At Aigoual, lamprophyres and granites have gradational contacts and evidences in the field and similar geochemical features that they coexisted indicate that they are comagmatic. U-Pb dating of zircon yields crystallisation ages between <math>311 \pm 2.9</math> Ma and <math>313 \pm 3.0</math> Ma for dykes, synchronous within uncertainty with the ages of <math>312 \pm 3.1</math> Ma and <math>313 \pm 3.2</math> Ma obtained for the emplacement of the Aigoual pluton. The lamprophyres are metaluminous to slightly peraluminous and display enrichment in both compatible (Fe, Mg, Ni, Cr) and incompatible elements (K, <math>^{20}\text{O}</math>, LILE, LREE) and have crustal isotopic signatures in both radiogenic (Sr, Nd and Hf) and stable (O) isotope systems. This dual crust/mantle signature of the lamprophyres indicates partial melting, within the spinel-lherzolite field, of an enriched mantle that was metasomatised by sediment-derived melts or fluids related to subduction preceding continental collision. The granites are peraluminous, display high K, <math>^{20}\text{O}</math> and have shoshonitic affinities. Their trace elements patterns are similar to the lamprophyres. The isotopic compositions of the granites overlap those of the lamprophyres. The high Cr, Ni, FeO, and MgO contents of the granites are consistent with a mantle component in the magmas. Based on our results, we suggest that between 65 and 80% of material in the lamprophyres and granites was derived from the depleted mantle, and the rest from recycled crustal material. Although blurred from an isotopic point of view, this magmatism contributes with crustal growth in the post-collisional magmatism of the FMC. Coupled with their high preservation potential, post-collisional sites might represent significant sites of crust growth.</p> |
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***Title: Cryptic crustal growth identified through Variscan post-collisional lamprophyre-  
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## Abstract

The processes that control crustal growth by addition of new mantle-derived material to the crust and the recycling of crustal components that were introduced into the mantle during subduction remain poorly understood in collisional orogens. This is largely due to the fact that the sub-orogenic mantle has, in many instances, been fertilized by the introduction of crustal components during previous subduction events, and lacks the typical depleted mantle signature. Here, we present a study of lamprophyric–granitic composite dykes from the northern margin of the post-collisional Variscan Aigoual granite, French Massif Central (FMC). At Aigoual, lamprophyres and granites have gradational contacts and similar geochemical characteristics which indicate that the two were co-magmatic. U–Pb dating of zircon yields crystallisation ages between  $311 \pm 3$  Ma and  $313 \pm 3$  Ma for dykes, identical within uncertainty with the ages of  $312 \pm 3$  Ma and  $313 \pm 3$  Ma obtained for the emplacement of the Aigoual granite. The lamprophyres are metaluminous to slightly peraluminous and display enrichment in both compatible (Fe, Mg, Ni, Cr) and incompatible elements ( $K_2O$ , LILE, LREE) and have crustal isotopic signatures in both radiogenic (Sr, Nd and Hf) and stable (O) isotope systems. This dual crust/mantle signature of the lamprophyres can be interpreted as partial melting, within the spinel stability field, of an enriched mantle that was metasomatised by the addition of subducted sediments. The granites are peraluminous, display high  $K_2O$  and have shoshonitic affinities. Their primitive mantle-normalized trace-element patterns are similar to the lamprophyres and the isotopic compositions of the two rock types overlap. The high Cr, Ni, FeO, and MgO contents of the granites are consistent with a mantle component in the magmas. Our results suggest that between 65 and 80 wt% of material in the lamprophyres and granites was derived from the depleted mantle, and the rest from recycled crustal material. Although blurred from an (conventional) isotopic point of

24 view, this magmatism contributed to the crustal growth via the post-collisional magmatism of the  
25 FMC. Coupled with their high preservation potential, post-collisional sites might thus represent  
26 significant sites of crustal growth.

27

28 **Keywords:** Composite Dykes; Crustal Growth; Variscan Belt; Lamprophyre; Metasomatised  
29 mantle

### Highlights

- Lamprophyre-granite composite dykes from late-Variscan Aigoual pluton, French Massif Central;
- Mantle-crust interaction in a post-collisional setting;
- Sr-Nd-Hf and O isotopes display a cryptic crustal growth.

[Click here to view linked References](#)

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## Abstract

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view, this magmatism contributed to the crustal growth via the post-collisional magmatism of the FMC. Coupled with their high preservation potential, post-collisional sites might thus represent significant sites of crustal growth.

**Keywords:** Composite Dykes; Crustal Growth; Variscan Belt; Lamprophyre; Metasomatised mantle

## 1. Introduction

The process that allow continental crust to be generated and evolve involve the addition of new, mantle-derived magma to the crust; the recycling and melting of oceanic crust and sediments at subduction sites and the addition of these magmas to the continental crust; as well as the anatectic reworking of the continental crust in collisional environments (Dhuime et al., 2011; Jacob et al., 2021; Moyen et al., 2021). The continental crust grows when new, purely mantle-derived material is added to the continents, not when continental crustal components introduced into the mantle are returned to the crust; this is simply crust coming home. Growth of the continental crustal occurred extensively in arc settings throughout geological time, where large amounts of mantle-derived mafic and intermediate magmas (and their differentiates) were added to the crust. Nevertheless, this does not necessarily imply significant long-term crustal-growth, because arc settings display poor preservation potential. A high proportion of the crust generated in this setting may be recycled back to the mantle shortly after formation (Kay and Kay, 1993; Condie, 2014; Scholl and von Huene, 2009). Although less efficient than arc settings in producing new continental crust, post-collisional sites display the greatest potential for preservation in the geological record (Hawkesworth et al., 2009; Spencer et al., 2015). Post-collisional settings are characterized by orogenic collapse, potential delamination of the lithosphere and hence asthenospheric upwelling. This may produce a range of magmas, including granites, from both mantle and crustal sources (Bonin, 2004; Clemens et al., 2009). Thus, post-collisional settings represent a significant opportunity for crustal growth in an environment with potential for long-term preservation (Couzinié et al., 2016; Moyen et al., 2017; Gómez-Frutos et al., 2023).

69 In this context, high-K–Mg mantle-derived magmatic suites occur systematically in post-  
70 collisional settings. Although relatively scarce, the suites are considered a significant contributor  
71 to crustal growth throughout geological time (Prelević et al., 2012; Scarrow et al., 2008; Murphy,  
72 2020). Post-collisional high-K–Mg mafic rocks have been described in the Variscan belt of  
73 Western Europe (e.g. Turpin et al., 1988; Janoušek & Holub, 2007; Couzinié et al., 2016; von  
74 Raumer et al., 2014). These rocks are characterized by a significant range of SiO<sub>2</sub> contents (45–  
75 65 wt%), high-K, through shoshonitic to ultrapotassic affinity (K<sub>2</sub>O = 1.5–6.0 wt%), and high  
76 maficity (FeO<sub>T</sub> + MgO up to 25 wt%). They are enriched in volatiles, mantle-compatible (Mg, Ni,  
77 Cr) and incompatible elements (e.g., K, Rb, Ba, Sr).

78 The French Massif Central is a segment of the Variscan Belt, in which high-K–Mg mafic  
79 rocks are spatially and temporally associated with the peak of granite magmatism (335 to 300  
80 Ma; Couzinié et al., 2014; Laurent et al., 2017). These mafic rocks occur as either enclaves to  
81 decametric small bodies in felsic igneous rocks and migmatites, or as lamprophyric dykes  
82 (Couzinié et al., 2014; 2016; Sabatier, 1991). Despite being well defined in space and time, the  
83 role of this mafic magmatism in the post-orogenic phase of the Variscan orogeny is still debated  
84 (e.g. Tabaud et al., 2015; Couzinié et al., 2016). More specifically, it is unclear whether the  
85 volumetrically more significant granites represent differentiated magmas from the mafic rocks  
86 (Clemens et al., 2021; Moyen et al., 2017) or the products of hybridization between the mafic and  
87 crustal melts (Laurent et al., 2017, Solgadi et al., 2007, Yamasaki, 2022). In turn, the balance  
88 between crustal growth by addition of new mantle-derived material and crustal recycling into the  
89 mantle in post-collisional sites is poorly understood (Jacob et al., 2021; Storck et al., 2021, and  
90 references therein). To contribute to this debate, we selected co-magmatic lamprophyres and  
91 granites in a unique system of parallel dykes associated with the northern boundary of the late-

Variscan Aigoual granite, French Massif Central, to investigate the petrogenesis of both rock types, explore the potential genetic links between them and to constrain the contribution of mantle-derived material to these post-collisional magmas.

## **2. Geological setting**

### **2.1 The French Massif Central**

The orogenic Variscan Belt in central and Western Europe formed between c. 400 and 280 Ma in a complex history of collision between two main continents, Gondwana and Laurussia, as well as intervening micro-continents and oceanic basins, resulting in the formation of the Pangea supercontinent at the end of the Paleozoic (Kröner and Romer, 2013; Stampfli et al., 2013; Matte, 2001). One of the largest exposures of the Variscan Belt in Western Europe is preserved in the French Massif Central (FMC) (Vanderhaeghe et al., 2020). Igneous and metamorphic rocks here represent the exhumed root zone of the orogen (Lardeaux et al., 2014), constructed over the northern margin of Gondwana (Fig. 1b).

From the Ordovician to the late Carboniferous, the FMC experienced several tectonometamorphic–magmatic events, with opening of ocean basins (ca. 500–450 Ma) and subsequently closure and subduction between 420 and 360 Ma, to continental collision between ca. 350 and 320 Ma. Orogenic collapse occurred in the late Carboniferous (ca. 320–300 Ma) (Faure et al., 2009; Vanderhaeghe et al., 2020). The eastern FMC (E-FMC) consists of a south-verging stack of nappes that are composed of four main metamorphic units (Faure et al., 2009; Vanderhaeghe et al., 2020).

In the southeastern part of the FMC, a low-grade metamorphic unit called the Parautochthonous Unit represents the lowermost unit of the nappe pile. It exposes predominantly metasediments (micaschists, quartzites and graphitic metapelites) (Faure et al., 1999) deposited

between the Neoproterozoic and early Cambrian (Melleton et al., 2010), minor orthogneisses formed in the late Cambrian to Early Ordovician (490–485 Ma), and metarhyolites. These rocks underwent polyphase deformation and metamorphism between ca. 340 and 310 Ma under greenschist- to amphibolite-facies conditions (Faure et al., 1999) (Fig. 1b). This unit was intruded by the Aigoual–Saint Guiral–Liron Pluton (ASGL) at shallow depth (1–2 kbar; Najoui et al., 2000) developing a contact metamorphic aureole. Based on U–Pb dating in zircon and monazite, and  $^{40}\text{Ar}/^{39}\text{Ar}$  dating in biotite, the main intrusion occurred between 305 and 310 Ma. Thus it was synchronous with the late stage of migmatization at lower structural levels and the late Variscan extensional event in this area (Brichau et al., 2008).

## 2.2 Magmatic relations in the E-FMC and Aigoual granite

The main phase of igneous activity in the French Massif Central occurred during the transition from mid-Carboniferous collision to the syn- and post-orogenic collapse of the orogen in late-Carboniferous to early-Permian times (Faure et al., 2009; Laurent et al., 2017). The plutonic rocks of E-FMC can be subdivided in four main groups (Chantraine et al., 1996; Barbarin, 1999). (1) Two-mica or muscovite-bearing peraluminous granitoids (“MPG”) cropping out as small plutons and dykes. These rocks are granites *sensu stricto* with  $\text{SiO}_2$  (>70 wt%); their chemistry and isotopic characters indicate mature crustal source (Pin and Duthou, 1990); (2) Cordierite-bearing peraluminous granitoids (“CPG”), which are granodiorites to granites with biotite and cordierite ranging from abundant to rare. These rocks are characterized by  $\text{K}_2\text{O}$  contents in the range of 3–7 wt%, with  $\text{SiO}_2$  > 65wt%. Isotopic characteristics suggest melting of local crust as the source of the magmas that formed these rocks (Pin and Duthou, 1990; Williamson et al., 1992). The compositions of the CPG are consistent with higher melting temperatures ( $\geq 850$  °C, biotite breakdown) than the MPG (Laurent et al., 2017; Villaros et al.,

2018) and/or greater proportions of orthogneisses in the source (Downes et al., 1997; Moyen et al., 2017), although with limits imposed by their isotopic compositions. (3) K-feldspar-phyrlic, biotite  $\pm$  amphibole-bearing granites, granodiorites and quartz diorites (“KCG”) are high-K calc-alkaline, metaluminous to weakly peraluminous and form large plutons (e.g., Aigoual–Saint Guiral–Liron) or enclaves in the Velay Complex. In general, they are more mafic than the CPG, having  $\text{FeO}^t + \text{MgO} \sim 4$  wt%, with  $\text{SiO}_2$  contents averaging around 69 wt%. (4) A suite of biotite–amphibole  $\pm$  clinopyroxene-bearing mafic to intermediate rocks that occur throughout the Variscan Belt (von Raumer et al., 2014) and are locally known as vauagnerites (Couzinié et al., 2016; Sabatier, 1991). The vauagnerites are metaluminous with low silica ( $44 < \text{SiO}_2 < 63$  wt%), high  $\text{FeO}^t + \text{MgO}$  (10–24 wt%), Cr (200–600 ppm) and Ni (100–220 ppm) contents,  $\text{Mg\#}$  (molecular  $\text{Mg}/(\text{Mg} + \text{Fe}) > 0.65$ ). They also have high  $\text{K}_2\text{O}$  contents (1.6–6.7 wt%) and high Ba and Sr contents (1000–2000 ppm; Couzinié et al., 2016). In this contribution, we refer to these rocks as lamprophyres.

The Aigoual granite forms the northern end of the L-shaped Aigoual–Saint Guiral–Liron (AGSL) Pluton in the southeastern FMC. The ASGL rocks are classified as K-feldspar-phyrlic calc-alkaline granitoids (KCG) (Chantraine et al., 1996). The northern termination of the Aigoual granite consists of a swarm of NE–SW to NNE–SSW-trending lamprophyre–granite composite dykes, which are the subject of this study (Fig. 1c).

### 3. Analytical methods

This study has integrated information on field relations, petrography, mineral chemistry, whole-rock major and trace element compositions, whole-rock Nd, Sr and Hf isotopic ratios, U–Pb dating and Lu–Hf analyses in zircon and oxygen isotopes in quartz and K-feldspar, from the Aigoual lamprophyre–granite composite dykes and three samples from the Aigoual granite. The

whole-rock major- and trace-element data information of mineral major element compositions, as well as U–Pb dating of zircon, were undertaken at the Central Analytical Facilities at Stellenbosch University, South Africa. The whole-rock Nd, Sr and Hf isotope analyses and quartz and K-feldspar oxygen isotope ratio analyses were undertaken at the University of Cape Town. Lu–Hf analyses in zircon were carried out at Universidade Federal de Ouro Preto, Brazil. The analytical details for each method are described in the Supplementary Material 1.

## **4. Results**

### **4.1 Field relations**

At the northern end of the Aigoual granite, several 100–1000 m long, NE-trending dykes intrude the Cévennes micaschists, part of the Parautochthonous Unit described above. Most dykes are composite and display gradation between granitic and lamprophyric components. The wider dykes (described by Sabourdy, 1975) display lamprophyric dark borders with aphanitic textures, progressively grading into porphyritic microgranites with some mafic enclaves in the cores. In general, bulk compositional gradients that are interpreted to reflect different mixing ratios, are aligned parallel to the dyke walls. The dyke illustrated in Fig. 2a varies from lamprophyre to granite (SiO<sub>2</sub> 57.20 to 67.81 wt%) from left to right in the image. An increase in number and size of K-feldspar phenocrysts, from lamprophyric to granitic end-members, was observed in the field (Fig. 2a). In other outcrops, lamprophyric and granitic components form independent dykes that were observed to intrude one another (Fig. 2b).

The Aigoual granite proper contains many typically rounded, 1 to 3dm in diameter (Fig. 2c), lamprophyric enclaves with partly resorbed phenocrysts of K-feldspar. In combination, the compositionally gradational dykes and the existence of lamprophyric enclaves in the granites that include crystals likely to have been captured from the granite magma. In combination, this

mineral textural and field evidence demonstrates the coeval nature of the granitic and lamprophyric magmas.

The sample list and the coordinates of the sample points are presented in Supplementary Data 2.

## **4.2 Petrography and mineral chemistry**

### **4.2.1 The composite dykes**

The lamprophyres have panidiomorphic–porphyritic textures with phenocrysts of euhedral biotite (~ 25–30 vol%), plagioclase (~ 20%), quartz (~ 5%) and K-feldspar (~ 5%), set in a fine matrix composed of quartz, alkali-feldspar, plagioclase, biotite, ± amphibole, ± carbonate (Fig. 3). Olivine and pyroxene were not observed but pseudomorphs indicate the former presence of one or more ferromagnesian phases in some lamprophyric samples, as also reported by Sabourdy (1975) from the same outcrops. Accessory minerals are apatite, zircon, ilmenite, ± allanite, ± rutile.

A characteristic of the lamprophyres is the presence of carbonate in three different textural forms (Fig. 3): 1) globular, ocellar; 2) interstitial to the silicate minerals, and 3), intergrown with biotite/phlogopite. The carbonate-mica intergrowths appear to pseudomorph a prior blocky mineral.

Carbonate (dolomite) was observed mantling corroded quartz that varies from 0.4 to 0.6 mm in size (Supplementary Figure 1a). Ocellar carbonate aggregates vary from 0.2–1.6 mm in diameter; the ocelli also contain very fine-grained alkali-feldspar (Fig. 3). The matrix surrounding the ocelli is aphanitic and composed of quartz, plagioclase, biotite (partially altered to chlorite in some places). Matrix plagioclase laths display an orientation tangential to the

margins of the carbonate globules indicating that the ocelli structures formed part of the magma (Supplementary Figure 1b-c). Interstitial carbonate is present among euhedral quartz, plagioclase and biotite (Supplementary Figure 1d). The interstitial character of the carbonate relative to euhedral quartz, biotite and plagioclase crystals is shown in the Si X-ray map of Supplementary Figure 1e. Plagioclase crystals associated with the carbonate display normal magmatic zoning with Na content increasing towards the rim (Ab<sub>56</sub> to Ab<sub>75</sub>) (Supplementary Figure 1f). The carbonate associated with biotite/phlogopite (interpreted as pseudomorphing a high-temperature ferromagnesian phase) was observed in both granitic and lamprophyric samples (Supplementary Figure 1g, h). In these textures, calcite or dolomite cores are rimmed by blocky phlogopite and both replace a former phenocryst with rectangular habit, possibly pyroxene.

Granitic samples from composite dykes display seriate porphyritic textures with extreme average crystal size variations between the phenocrysts and the matrix. In general, the phenocrysts (up to 5 cm) of plagioclase (~ 30%), K-feldspar (~ 30%), quartz (~ 20%) biotite (~ 5–10%) and ± clinopyroxene (~ 3%) are set in a fine- to medium-grained matrix composed of quartz, K-feldspar ± plagioclase. Accessories are apatite, zircon, ± allanite, ± titanite ± ilmenite. In some samples, phenocrysts of plagioclase display oscillatory zoning (Fig. 3j) and some K-feldspar phenocrysts have corroded margins (Fig. 3l).

#### **4.2.2 Aigoual granite**

The Aigoual granite proper consists predominantly of strongly porphyritic, coarse-grained granite with phenocrysts of K-feldspar reaching 5–8 cm. The granite contains phenocrysts of K-feldspar (~ 30%), plagioclase (~ 20%) and quartz (~ 15%) in a coarse matrix of quartz, plagioclase and K-feldspar (~20%), mica (~7%) and clinopyroxene (~5%).

#### **4.2.4 Mineral Chemistry**

Mineral chemistry data is presented in Supplementary Data 4, Table S1. Euhedral crystals of mica from the lamprophyres have Mg# varying from 0.47 to 0.80, corresponding to a transition between biotite (Mg# < 0.65) and phlogopite (Mg# > 0.65) (Fig. 4a). Biotite/phlogopite has low to moderate Cr<sub>2</sub>O<sub>3</sub> content (0–0.05 apfu), high Ti (0.37–0.97 apfu) and in some samples displays zoning in BSE images with dark grey cores and light gray discordant rims (e.g. Fig. 3g). The cores have lower Ti (0.42–0.59 apfu) than the rims (0.78–0.97 apfu) and biotite in the matrix shows similar compositions to the rims with high Ti (0.69–0.94 apfu). In the FeO<sup>t</sup> vs. Al<sub>2</sub>O<sub>3</sub> and TiO<sub>2</sub> vs. Al<sub>2</sub>O<sub>3</sub> mica discrimination diagrams, the biotite/phlogopite follows a minette trend, typical of calc-alkaline lamprophyres (Fig. 4b–c; Rock, 1991). Dark micas in the granites from the composite dykes are generally less magnesian than those in the lamprophyres, with an average of Mg# ~ 0.52 and are thus classified as biotite. A few analyses with Mg# > 0.65 fall in the phlogopite field (Fig. 4a). Biotite in the Aigoual granite proper has Mg# ~ 0.52.

Feldspar compositions are illustrated in Supplementary Figure 2a. Feldspar in the lamprophyres is predominantly plagioclase, which ranges in composition from An<sub>10</sub> to An<sub>60</sub>. Alkali feldspar is a relatively minor component in these rocks and ranges in composition from Or<sub>5</sub> to Or<sub>40</sub>. Feldspar in the Aigoual granite consist of plagioclase with a narrow compositional range clustered around An<sub>30</sub> and K- feldspar which varies in composition from Or<sub>80</sub> and 92. In the granite dykes the plagioclase compositional range is similar to that exhibited by the lamprophyric dykes. However, at low An contents significantly more K<sub>2</sub>O (typically up to Or<sub>30</sub>) is present in some feldspar crystals with significant Ca, which plot in the fields of oligoclase and anorthoclase. The more common alkali-feldspar compositions in these dykes vary from Or<sub>60</sub> to Or<sub>100</sub>.

All pyroxene in the Aigoual granite and in the granitic dykes is augite and clusters in a narrow range of compositions characterized by Ca contents in the range of Di<sub>50</sub> to 60 and with Mg#s between 60 and 75 (Supplementary Fig. 2b). All amphibole from the lamprophyres is actinolite with high Si contents (7.53–7.90 apfu) and Mg# (0.62–0.88), as is typical of secondary amphiboles in lamprophyres (Supplementary Fig. 2c and d).

### 4.3 Zircon U–Pb ages and Lu–Hf isotopes

In this work, the geochronological data are from individual composite dykes, where lamprophyre and granite occur in the same outcrop with gradational transition from one to the other. The zircons are euhedral to subhedral, 50–450 µm across. The crystals have prismatic, subrounded prismatic, pyramidal to bipyramidal tips and aspect ratios from 4:1 to 1:1. Under CL, they exhibit strong to weak oscillatory zoning, diffuse zoning or no luminescence at all (Supplementary Figure. 3). Many grains show a core–rim texture with diffusely or oscillatory zoned core surrounded by a dark rim. Inherited cores display bright, diffuse to oscillatory, truncated zoning with dark rims.

The <sup>206</sup>Pb/<sup>238</sup>U dates show a large range of concordant ages, scattering from 340 to 300 Ma (Fig. 5). However, this large dispersion displays no correlation with position of the analysis spot in different zircon crystals. Older ages, between 320 and 340 Ma, were obtained from both dark rims and CL-dark grain interiors with unusually high U contents (> 1000 ppm). Due to the high U content, these zircons might no longer be crystalline (Ewing et al., 2003). The absence of a correlation between age and Hf isotopic composition indicates that the age dispersion has no geological significance. The older ages, between 320 and 340 Ma, are likely to be artefacts potentially caused by inaccurate correction for downhole fractionation in these high U crystals and strongly radiation damaged zircon. Accordingly, the Concordia ages were calculated using

data from zircons that were more likely to retain coherent crystallinity ( $U < 1000$  ppm) and KDE variation, which displays a peak at 315 - 310–Ma for most of the samples (Fig. 6).

Eleven concordant analyses from lamprophyres and granites of dyke 5 yielded a Concordia date of  $^{206}\text{Pb}/^{238}\text{U} = 313 \pm 3$  Ma (MSWD conc. + equiv. = 1.7) (Fig. 5a). Inherited grains were largely found in the granite component and yielded  $^{206}\text{Pb}/^{238}\text{U}$  ages of  $537 \pm 10$ ,  $541 \pm 14$ ,  $545 \pm 9$ ,  $649 \pm 10$  and  $673 \pm 7$  Ma. Younger ages of  $296 \pm 4$  and  $291 \pm 5$  Ma are interpreted as reflecting Pb loss. Magmatic zircons from dyke 5 have  $\varepsilon_{311}^{\text{Hf}} = -1.6$  to  $-8.1$  ( $\pm 1.2$ ),  $T_{\text{DM}} = 1.7$  to 1.3 Ga] (Fig. 6). The inherited zircons yielded  $\varepsilon_{311}^{\text{Hf}}$  of  $+6.8$  to  $+0.9$ .

Thirty-three concordant analyses from lamprophyres and granites of dyke 10 yielded a Concordia date of  $^{206}\text{Pb}/^{238}\text{U} = 311 \pm 3$  Ma (MSWD conc. + equiv. = 1.5) (Fig. 5b). A large population of inherited grains found in the granite component yielded several date groups from Paleoproterozoic (2490–2040 to 2480 Ma), through early Neoproterozoic (920–1000 Ma) to late Neoproterozoic and early Paleozoic (450–730 Ma). Zircons from dyke 10 have  $\varepsilon_{311}^{\text{Hf}} = +3.6$  to  $-7.7$  ( $\pm 1.5$ ) (Fig. 6), with  $T_{\text{DM}} = 1.7$  to 1.1 Ga. Inherited grains with  $^{206}\text{Pb}/^{238}\text{U}$  ages of 450–600 Ma gave  $\varepsilon_{311}^{\text{Hf}}$  of  $+2.5$  to  $0.0$ , whereas inherited grains ( $> 600$  Ma) yielded  $\varepsilon_{311}^{\text{Hf}}$  of  $+2.9$ – $-13.5$ .

Samples 12A and 12B represent the eastern margin of Aigoual granite with microgranite and monzonite occurring as part of the body of the granite, in contrast to all the other granite samples, which form part of the composite dykes. Isotopic data from 10 concordant analyses display a Concordia date of  $312 \pm 3$  Ma (MSWD conc. + equiv. = 1.7) (Fig. 8c). No inherited grains were observed. Zircon grains from margin of Aigoual granite (locality 12) have  $\varepsilon_{311}^{\text{Hf}} = 0.4$  to  $-6.8$  ( $\pm 1.4$ ),  $T_{\text{DM}} = 1.6$  to 1.2–Ga] (Fig. 6).

Zircons from the Aigoual granite proper yield a Concordia date of  $313 \pm 3$  Ma (MSWD conc. + equiv. = 1.3; n = 7) (Fig. 8d) with  $\varepsilon_{311}^{Hf} = -5.6$  to  $-3.3 (\pm 1.1)$ ,  $T_{DM} = 1.4\text{--}1.5$  Ga] (Fig. 6).

#### 4.4 Whole-rock major- and trace-element chemistry

In the TAS system of Le Maitre et al. (2002), the rocks of this study would be classified as granites, quartz monzonites and monzonites, but of course such alkali rich compositions are not amenable to classification according composition discrimination diagrams. The compositions of the three Aigoual granite samples are very similar and fall consistently between the compositions of the granitic and lamprophyric portions of the composite dykes on plots of all elements vs.  $\text{SiO}_2$  (Fig. 7). The dykes show a wide range in  $\text{SiO}_2$ , varying from 53.16 to 72.57 wt%, recalculated on a volatile-free basis. Lamprophyric and granitic samples are high-K (4.08–6.55 wt%) with shoshonitic affinities (Fig. 8a) and high  $\text{Al}_2\text{O}_3$  (13.63–17.18 wt%). In the A/NK vs. A/CNK [molar  $\text{Al}_2\text{O}_3/(\text{Na}_2\text{O} + \text{K}_2\text{O})$  vs.  $\text{Al}_2\text{O}_3/(\text{CaO} + \text{Na}_2\text{O} + \text{K}_2\text{O})$ ] diagram of Shand (1943), lamprophyric samples classify as metaluminous to slightly peraluminous with higher A/NK values, whereas granitic samples are weakly to moderately peraluminous with some exceptional cases having A/CNK > 1.3 (Fig. 8b).

A striking feature of the bulk rock chemistry of the entire sample set is that Cr, Ni and Ba are high (30–573 ppm, 15–149 ppm and 404–2416 ppm, respectively), but variable and with only a vague inverse correlation with  $\text{SiO}_2$ . Mg# varies from 41 to 71 and along with  $\text{TiO}_2$  and  $\text{P}_2\text{O}_5$  is one of the components which shows the best developed inverse correlation with  $\text{SiO}_2$  concentration. For the incompatible elements, which commonly are positively correlated with  $\text{SiO}_2$  content,  $\text{Na}_2\text{O}$  demonstrates this behavior, despite a significant amount of scatter, for the lamprophyres. However, for the granites, if there is any trend its flat. Similarly, over the whole

range of compositions  $K_2O$  shows no correlation with  $SiO_2$ , with the majority of the rocks falling between 5 and 6 wt%  $K_2O$ .

The lamprophyric–granitic rocks from composite dykes and the Aigoual granite are enriched in large ion lithophile elements (LILE, e.g. Cs, Rb, Ba, Th, U; Fig. 9a) and Pb relative to high field strength elements (HFSE, e.g. Nb, Ta, P, Zr and Ti). The lamprophyres show negative Nb, Ta and Ti anomalies, but Ta anomalies are not observed in the granites. In general, the lamprophyres are richer in REE and mantle-incompatible elements than the granites, as is commonly observed in these types of rocks (e.g. Couzinié et al., 2016; Fowler et al., 1996).

In the primitive mantle -normalized (Sun and McDonough 1989) REE multielement plot (Fig. 9b), lamprophyres and granites from composite dykes as well as the Aigoual granite samples show similar patterns. They are enriched in LREE ( $La_N = 24\text{--}147$ ) and depleted in HREE ( $La_N/Yb_N = 3.34\text{--}22.81$ ). In general, lamprophyres show smaller negative Eu anomalies ( $Eu/Eu^* = 0.60\text{--}0.85$ ) than granites ( $Eu/Eu^* = 0.38\text{--}0.86$ ).

#### 4.5 Whole-rock Sr–Nd–Hf isotopes

Lamprophyres and granites from the composite dykes show little variation in Sr, Nd and Hf isotope ratios. Initial  $^{87}Sr/^{86}Sr$  ratios of lamprophyres and granites overlap and vary from 0.7080 to 0.7106. Similarly, the Nd isotope ratios of the lamprophyres and granites overlap ( $\epsilon_{311}^{Nd} = -3.60$  to  $-5.39$  and  $\epsilon_{311}^{Nd} = -4.62$  to  $-5.39$  respectively). The Hf isotope ratios for both rock types also overlap, with lamprophyres varying from  $\epsilon_{311}^{Hf} = -0.26$  to  $-4.92$  and the granites varying from  $\epsilon_{311}^{Hf} = -2.31$  to  $-3.47$  (Fig. 14) (Table S2 from Supplementary Data 3). On average, lamprophyres have slightly more radiogenic Nd and Hf than granites ( $\epsilon_{311}^{Nd} = -4.40$  and  $-5.13$ , and

340  $\varepsilon_{311}^{Hf} = -2.81$  and  $-3.20$ , respectively). The Aigoual granite has an  $(^{87}\text{Sr}/^{86}\text{Sr})_i$  of  $0.70941$ ,  $\varepsilon_{311}^{Nd} = -$   
 341  $5.43$  and  $\varepsilon_{311}^{Hf} = -3.71$ .

## 342 4.6 Quartz, and K-feldspar O isotopes

343 The  $\delta^{18}\text{O}$  values of quartz and K-feldspar ( $\delta^{18}\text{O}_{\text{Qtz}}$  and  $\delta^{18}\text{O}_{\text{Fsp}}$  respectively) from composite  
 344 dykes are listed in Table 3 from Supplementary Data 4 and illustrated in Fig. 10. The  $\delta^{18}\text{O}_{\text{Qtz}}$   
 345 values vary from  $10.76$  to  $11.21\text{‰}$  ( $n = 9$ ; average =  $10.97\text{‰}$ ) and, despite their narrow range,  
 346 show a positive correlation with whole-rock  $\text{SiO}_2$  ( $r^2 = 0.72$ ) (Fig. 10a) and a negative one with  
 347  $\text{Cr} + \text{Ni}$  ( $r^2 = -0.66$ ) (Fig. 10b). The  $\delta^{18}\text{O}_{\text{Fsp}}$  vary from  $6.4$  to  $13\text{‰}$  ( $n = 9$ ; average =  $9.4\text{‰}$ ) and  
 348 are thus much more variable than for quartz. There is no correlation between  $\delta^{18}\text{O}_{\text{Fsp}}$  and  $\delta^{18}\text{O}_{\text{Qtz}}$   
 349 (Fig. 10c). The  $\delta^{18}\text{O}_{\text{Qtz}}$  values display no correlation with whole-rock  $(^{87}\text{Sr}/^{86}\text{Sr})_i$  ( $r^2 = 0.13$ ) (Fig.  
 350 10d),  $\varepsilon^{Nd}$  ( $r^2 = 0.17$ ; not shown) and  $\varepsilon^{Hf}$  ( $r^2 = 0.23$ ; not shown). The three lamprophyre samples  
 351 plot between the diffusion closure and crystallization isotherms on a  $\delta$ - $\delta$  plot (Fig. 10c). The  
 352 remaining samples show a wide range in feldspar  $\delta^{18}\text{O}$  values that are both higher and lower than  
 353  $\delta^{18}\text{O}_{\text{Qtz}}$ . The wide range in  $\delta^{18}\text{O}_{\text{Fsp}}$  values is consistent with interaction with fluids at a range of  
 354 temperatures.

## 355 5. Discussion

### 356 5.1 Timing of magma emplacement

357 U-Pb analyses in zircon from lamprophyres and granites from dykes 5 and 10 yielded  
 358  $^{206}\text{Pb}/^{238}\text{U}$  ages of  $313 \pm 3$  Ma and  $311 \pm 3$  Ma, respectively. Locality 12, in the Aigoual granite  
 359 at its eastern border, gave a  $^{206}\text{Pb}/^{238}\text{U}$  age of  $312 \pm 3$  Ma and the granite from the center of the  
 360 body has a  $^{206}\text{Pb}/^{238}\text{U}$  age of  $313 \pm 3$  Ma. Therefore, all samples of dykes and the Aigoual granite  
 361 show the same crystallization ages, within uncertainties. These are statistically indistinguishable

from the U–Pb age of  $304 \pm 12$  Ma, reported by Brichau et al. (2008), from the southern portion of the ASGL Pluton.

The alignment of the high-K–Mg rocks in FMC in along NE–SW trends (Fig. 1b), that are also parallel to major lithological boundaries, suggests that emplacement followed zones of weakness in the crust. Emplacement was possibly also facilitated by NW–SE directed extensional associated with crustal thinning of the Variscan Belt, during the syn- to post-collisional period (Faure et al., 2009; Talbot et al., 2005). Dykes from northern Aigoual granite display an AMS fabric indicative of NE–SW stretching. However, this fabric was controlled by magmatic flow rather than regional extension, suggesting a southwestward source feeding the magmatic system (Talbot et al., 2005).

The lack of inherited or xenocrystic zircon grains in lamprophyres can be explained by zircon under-saturation due to the high temperature and mafic composition of these magmas (Laurent et al., 2017). Inherited grains in granitic samples display a wide spread of Paleoproterozoic to late-Ordovician ages (Fig. 8b) and  $\mathcal{E}^{\text{Hf}}$  signatures from -13 to +6.8, consistent with age distribution and isotopic signatures from Variscan ortho- and paragneisses in the FMC (Chelle-Michou et al., 2017).

Dating of granites that were proposed to be crustally derived and mantle-derived mafic magmas in the E-FMC (Couzinié et al., 2014; Laurent et al., 2017) showed that the two were related in space and time. They marked a protracted (40 Myr) magmatic period, in which granites ( $337 \pm 1$  to  $299 \pm 2$  Ma), emplaced coevally with vaugnerites ( $336 \pm 2$  and  $299 \pm 1$  Ma – Laurent et al., 2017). The age distribution displays migration of magmatism toward the south, where the younger ages of 307 and 298 Ma were reported from the KCG plutons in the Cévennes area (Couzinié et al., 2014; Laurent et al., 2017). These are substantially younger than the ages

reported in the current work. Late Carboniferous southward delamination of the lithospheric mantle and asthenosphere upwelling may explain the long-lived lithospheric-scale thermal anomaly that could have produced melts of both crustal and mantle sources (Laurent et al., 2017).

## **5.2 Origin of the lamprophyres and granites**

### **5.2.1 Origin of lamprophyres**

The lamprophyres have moderate to high compatible element contents, e.g. Cr (74–572 ppm) and Ni (23–148 ppm), coupled with relatively high Mg# (up to ~ 70), consistent with a mantle source for at least this component of the composite system. However, the lamprophyres also display elevated concentrations in LILE (K<sub>2</sub>O, Sr, Rb, Ba,), LREE and depletion in HFSE such as Nb, Ta and Ti, in relation to the primitive mantle. Whole-rock Sr–Nd–Hf and in-zircon Hf isotopes display crust-like signatures with subchondritic  $\epsilon^{\text{Nd}}$  and  $\epsilon^{\text{Hf}}$  values. Also the  $\delta^{18}\text{O}$  values for the lamprophyres are on average 5‰ higher than the expected value for depleted mantle (5.2‰, Eiler, 2001). The crust-like signatures of the lamprophyres can result from crustal assimilation accompanying fractional crystallization of a primitive basaltic liquid (AFC process) or partial melting of an enriched mantle source, that was metasomatised by interaction with crustal material during the subduction stage that preceded the continental collision (e.g., Becker et al., 1999; Janoušek and Holub, 2007; Lexa et al., 2011; Schulmann et al., 2014; Couzinié et al., 2016).

The absence of correlations between Mg#, Cr, Ba, Ni and  $^{87}\text{Sr}/^{86}\text{Sr}_i$ ,  $\epsilon^{\text{Nd}}$ ,  $\epsilon^{\text{Hf}}$  suggests that the isotopic enrichment did not result from assimilation of continental crust by a primitive mafic end-member magma. Moreover, the most mafic members display crust-like isotopic signatures, similar to the intermediate members and even to the granites from composite dykes. An alternative interpretation that the lamprophyres are the result of assimilation of Fe-Mg rich crustal

rocks of suitable composition, e.g. the graphitic metapelites of the Parautochthonous Unit (Faure et al., 2001), does not seem viable, as such sediments are unlikely to be sufficiently rich in Cr and Ni. Furthermore, lamprophyres and other high-K–Mg rocks referred to as vagnerites are ubiquitous in the FMC, where they have intruded several different rock types of the nappe pile (Montel & Weisbrod, 1986; Sabatier, 1991; Couzinié et al., 2016). These other high-K–Mg rocks have chemical and isotopic signatures similar to the lamprophyres reported in this work, pointing to the enriched mantle as the most likely source (Couzinié et al., 2016).

Magmas derived from mantle sources modified by subducted components usually differ from MORBs and OIBs by enrichment in Th and Nb compared to Yb (Pearce, 2008) (Fig. 11a). As is the case for modern sediments (GLObal Subducted Sediments – GLOSS; Plank and Langmuir, 1998), the studied calc-alkaline lamprophyres show jagged spidergrams marked by low Nb–Ta–Ti concentrations and elevated Th, U and Pb. All these geochemical features coupled with low Nb/La ratios, enrichment in LILE, LREE and depletion in HFSE in primitive-mantle-normalized multi-element diagrams suggest that the enriched lithospheric mantle was the most likely source for the lamprophyres. Likewise, the relatively low  $^{87}\text{Sr}/^{86}\text{Sr}_i$  ratios (0.7080– 0.7106) are consistent with recent mantle enrichment, probably during the Silurian–Devonian subduction.

The presence of phlogopite identified in the lamprophyres and also the elevated Rb/Sr and low Ba/Rb ratios strongly indicate that phlogopite was the dominant hydrous phase in the mantle source (Fig. 11b) (e.g., Butvina et al., 2021; Condamine et al., 2022). Moreover, high-K<sub>2</sub>O magmas with shoshonitic affinities are a typical product of partial melting of phlogopite-bearing lithospheric mantle that has been metasomatised by fluids or melts derived from the continental crust or marine sediments during subduction (Conceição and Green, 2004; Förster et al., 2019; Prelević et al., 2012). (Fig. 11c).

The  $\delta^{18}\text{O}_{\text{Qtz}}$  values can be used as a proxy for the original magma  $\delta^{18}\text{O}$  values (e.g., Harris et al., 2005). The parental magma would be about 1‰ lower than  $\delta^{18}\text{O}_{\text{Qtz}}$  values (e.g. Fourie and Harris, 2011). The calc-alkaline lamprophyres display high  $\delta^{18}\text{O}_{\text{Qtz}}$  values ( $\delta^{18}\text{O} = 10.76$  to 10.80), suggesting an average  $\delta^{18}\text{O}$  value of  $\sim 10\text{‰}$  for the original magma. This is close to the boundary between I- and S-type granites suggested by Harris et al. (1997) (Fig. 12). The  $\delta^{18}\text{O}$  values expected for an uncontaminated mantle-derived magma is about 5.7‰ (Eiler, 2001) and fractional crystallization from a primitive magma can at most change  $\delta^{18}\text{O}$  values by  $\sim 1.0\text{‰}$  for a 20% increase in  $\text{SiO}_2$  content (Bindeman, 2008; Bucholz et al., 2017). Either a very high  $\delta^{18}\text{O}$  contaminant or a very high proportion of crustal assimilation would have been required to raise the  $\delta^{18}\text{O}$  value from 5.7 to 10‰. Simple mixing calculations between mantle and average of upper continental crust ( $\delta^{18}\text{O} = 15\text{‰}$ ) show that  $\sim 50\%$  of crustal assimilation would be necessary to raise  $\delta^{18}\text{O}$  values above 10‰ (Fig. 13). This high degree of crustal assimilation is inconsistent with the high compatible element contents and the relatively low  $^{87}\text{Sr}/^{86}\text{Sr}_i$  ratios of the lamprophyres which point to little or no crustal assimilation being involved in the origin of lamprophyres. Therefore, we suggest that the high  $\delta^{18}\text{O}$  values in lamprophyres result from contamination with high  $\delta^{18}\text{O}$  material already in the mantle source.

High  $\delta^{18}\text{O}$  values are common in sedimentary rocks. Oceanic carbonate-bearing sediments have  $\delta^{18}\text{O}$  values of 25 to 32‰ and pelagic clays from 15 to 25‰ (Eiler, 2001). Values from the silicate upper continental crust are around 10 and 20‰ (Taylor and Shepard, 1986). Extremely high  $\delta^{18}\text{O}$  values have been reported in peraluminous granite in ophiolite as a product of the melting of subducted pelagic sediments, leading to magma  $\delta^{18}\text{O}$  values above 20‰ (Spencer et al., 2017) (Fig. 12). In this work, the high  $\delta^{18}\text{O}$  values from lamprophyres ( $> 10\text{‰}$ ) indicate that

the crustal component that metasomatised the mantle source was dominated by sedimentary material.

To quantify the nature of the mantle source, mixing calculations using Sr, Nd and O isotopic systems were applied. Simple mixing calculations between mantle and potential contaminants indicate that ~ 20% of oceanic carbonate-bearing sediment ( $\delta^{18}\text{O} = 28\text{‰}$ ) or ~ 35% of pelagic clay ( $\delta^{18}\text{O} = 20\text{‰}$ ) would be necessary to increase  $\delta^{18}\text{O}$  values to  $> 10\text{‰}$  (Fig. 13).

Following the same approach, simple magma mixing calculations using  $^{87}\text{Sr}/^{86}\text{Sr}_i$  and  $\epsilon^{\text{Nd}}$  from the Variscan mantle and sedimentary material were applied. A proxy for the original, subduction-modified depleted Variscan mantle can be derived from Devonian arc-related magmatism and ophiolites of the Variscan Belt. Arc magmatism, resulting from fluid-fluxed melting of the mantle wedge should have  $\epsilon^{\text{Nd}}$  close to the canonical depleted mantle values but  $^{87}\text{Sr}/^{86}\text{Sr}$  offset to higher values (e.g., Bouilhol et al., 2013). The Devonian rocks of Massif Central have  $\epsilon^{\text{Nd}}$  up to +7 (Pin and Paquette, 2002). They also have  $^{87}\text{Sr}/^{86}\text{Sr}$  ratios that are invariably higher than the expected value of 0.703 on the “mantle array” for rocks with  $\epsilon^{\text{Nd}} = +7$ , as expected for arc magmas and likely reflecting some continental crustal component. Consequently, we estimate the Variscan mantle composition at that time to be  $\epsilon^{\text{Nd}} = +7$ ,  $^{87}\text{Sr}/^{86}\text{Sr} = 0.703$ .

In the case of FMC, the pre-Variscan metasediments are compositionally close to sediments from the former northern Gondwana margin (Chelle-Michou et al., 2017; Couzinié et al., 2017). We modelled binary mixing with several end-members, covering the range of Variscan metasediments (Solgadi et al., 2007; Williamson et al., 1992) and GLOSS (Plank and Langmuir, 1998). Mixing calculations indicate that the amount of sedimentary material mixed with mantle

required to model the crustal isotopic signatures in Sr–Nd isotopic systems is 25–40% (Fig. 14a), depending on the composition of the sediment end-member.

Lu–Hf and Sm–Nd isotopic systems display similar behavior during most magmatic processes and the composition of mantle and crust are correlated, evolving in a “mantle array” trend (Chauvel et al., 2008). Subchondritic whole-rock  $\epsilon^{\text{Nd}}$  and  $\epsilon^{\text{Hf}}$  imply mantle enrichment with crustal materials.

The textural evidence from the lamprophyres and granites argues for the involvement of a carbonate component in the magmas and the existence of an immiscible carbonate liquid in the lamprophyres during crystallization. Ocellar carbonates are commonly described as the products of immiscibility between silicate and carbonate liquids in potassic, alkaline, mantle-derived igneous rocks (e.g., Huang et al., 2002 and references therein). The presence of carbonates in lamprophyre might indicate that the crustal component was a carbonate-bearing sediment. This composition corroborates the high oxygen fugacity identified in the mantle beneath FMC, which is attributed to the addition of volatiles, including CO<sub>2</sub>, in the mantle during Variscan subduction, triggering CO<sub>2</sub>, H<sub>2</sub>O and K metasomatism (Martin et al., 2017).

After inferring the probable nature of the contaminant, the partial melting mechanism that generated the lamprophyres was investigated. Non-modal partial melting models of a calculated source with 65–80% of DM and 20–35% of a contaminant (GLOSS composition) show that melts produced by 10 to 20% partial melting match the trace-element composition of lamprophyres and associated granites (Fig. 15a, b). This model resembles results of experimental melting of metasomatised mantle by Condomine et al. (2022).

The high HREE contents are consistent with shallow melting of a spinel peridotite, i.e., in the absence of garnet. Lamprophyres and granites display higher LREE contents than the modelling

predicts, suggesting a pelagic clay richer in Rb and Ba than the average global sediments used in the calculation (Plank and Langmuir, 1998), or melts derived from sediments, more enriched in LREE than GLOSS.

In summary, the evidence from this study indicates that the magmas that produced the lamprophyric component of the composite dykes was produced by 10–20% melting of a metasomatised mantle source in the spinel-lherzolite field. This CO<sub>2</sub>-, H<sub>2</sub>O-, K-enriched metasomatised mantle was phlogopite-bearing and resulted from reaction with sediment-derived melts or fluids that were probably introduced into the mantle during Silurian–Devonian subduction.

#### **5.2.2 Origin of granites**

The studied dykes are composite and include lamprophyric and granitic components, which display field evidence indicating that they coexisted. They are also characterized by very similar geochemical features. Thus, the lamprophyres and granites of the composite dykes were comagmatic and show a mixture of “crust-like” and “mantle-like” geochemical features. Three main hypotheses or combinations thereof are plausible for the origin of the granites:

1. The lamprophyres and the granites are related by differentiation processes, such as fractional crystallization, although remelting of underplated lamprophyres to produce the granites would result in broadly the same patterns;
2. The lamprophyres and the granites represent distinct magmas, an enriched-mantle derived magma for the lamprophyres and a crust-derived magma for the granites, that mingled and mixed;
3. The granite and the lamprophyres are both direct products of melting of the enriched mantle source but reflect different degrees of partial melting. This could be achieved by

different source compositions, different temperatures of partial melting, different volatile contents (more CO<sub>2</sub> in the lamprophyre source), or some combination.

We briefly discuss each hypothesis below.

The granites in the dykes share many geochemical characteristics with the lamprophyres, including nearly identical isotopic ratios and trace-element concentrations. However, the granites and the lamprophyres are not connected by differentiation trends, as illustrated by the fact that the lamprophyres are richer in most incompatible elements than the granites. This enrichment is inconsistent with a simple model of fractional crystallization of mafic into felsic magmas and is also inconsistent with the granites being the product of partial melting of the lamprophyres.

Major elements such as K<sub>2</sub>O, Na<sub>2</sub>O and Al<sub>2</sub>O<sub>3</sub> are relatively constant with increasing SiO<sub>2</sub> content, implying no significant fractionation of K-feldspar, plagioclase and biotite in high-K magmas (e.g., Wang et al., 2021) (Fig. 6a, b, c). The increase in volume and size of phenocrysts of K-feldspar in the granitic component demonstrate that a considerable decrease in K<sub>2</sub>O is expected during fractional crystallisation. Furthermore, a fractionation process involving plagioclase would display similar behavior for CaO and Sr on the binary plots against SiO<sub>2</sub> in Figure 6 display distinct behavior from CaO and Sr against SiO<sub>2</sub> contents, demonstrating that fractional crystallization from the lamprophyres is unlikely to generate the granites.

In general, fractional crystallization involving clinopyroxene would remove compatible elements such as Cr and Ni. The high Cr (30–140 ppm; 80.9 on average and in cases over 100 ppm in granite with SiO<sub>2</sub> = 70 wt%) and Ni (14–40 ppm; 24.4 on average) content in the granites are inconsistent with fractional crystallisation of clinopyroxene playing an important role in the formation of granites. Moreover, no cumulate textures are observed in lamprophyres and no pyroxenites are described in the area. Therefore, this fractionation model is unlikely.

Cogenetic granite may result from mixing between lamprophyric and crust-derived magmas at crustal conditions. Petrological evidence favoring this model consists of the presence in ca. 10% of zircon xenocrysts in granites from composite dykes and the existence of corroded quartz xenocrysts mantled by carbonate and amphibole in lamprophyres. Quartz xenocrysts rimmed by amphibole and carbonate are interpreted as typical magma-mixing texture (e.g., Janoušek et al., 2004). In this way, we consider that assimilation of a crustal component is involved in the petrogenesis of the granites from the composite dykes.

Linear trends observed in many (but not all) binary chemical plots between lamprophyre and granite might indicate magma mixing between these two end members. However, similar trends are observed in fractional crystallization or partial melting processes (Janoušek et al., 2016). Radiogenic isotopes are generally not fractionated during partial melting or fractional crystallization/accumulation and, therefore, are the best tools for deciphering open-system processes such as magma mixing (e.g., Sun et al., 2010).

Large variations in zircon  $\epsilon^{\text{Hf}}$  in granitic rocks have been attributed to magma mixing between crustal and mantle-derived magmas (Griffin et al., 2002; Storck et al., 2021; Yang et al., 2007) or inheritance of heterogeneities in detrital zircon record from the sedimentary source of anatectic granites (e.g., Villaros et al., 2012).

In the present study, zircon  $\epsilon^{\text{Hf}}$  values from lamprophyres and granites vary from  $-7.8$  to  $+0.4$  with more than 90% of the data falling between  $-5.0$  and  $-1.6$ , i.e. within less than 4  $\epsilon^{\text{Hf}}$  units. This is a limited variation with respect the analytical uncertainty ( $c. \pm 1.5$  on average). In the case of mixing of magmas with typical mantle and crustal Hf isotopic compositions, a substantially wider range in  $\epsilon^{\text{Hf}}$  values is expected (up to 10  $\epsilon^{\text{Hf}}$  units). Also, crustal and mantle signatures would be more prominent, i.e., mantle component migrating towards positive and

crustal component towards negative  $\epsilon^{\text{Hf}}$  values (e.g., Storck et al., 2021). Likewise, magmatic rims mantling an old inherited core display different Hf isotopic signatures than cores, which indicates that the inheritance does not control the isotopic composition of the magmatic rim. The  $\epsilon^{\text{Hf}}$  values from lamprophyres and granites are similar and overlap, suggesting that mixing within different and contrasting magmas did not play an important role.

In terms of oxygen isotopes, the lack of correlation between  $\delta^{18}\text{O}$  values and  $^{87}\text{Sr}/^{86}\text{Sr}_i$ ,  $\epsilon^{\text{Nd}}$  and  $\epsilon^{\text{Hf}}$  renders the slight correlation between  $\text{SiO}_2$  and Cr or Ni meaningless. Therefore, the small difference in  $\delta^{18}\text{O}$  values between lamprophyres and granites (less than 0.5‰) is consistent with source-related variation or homogenization of the magma than mixing between two different and contrasting magmas.

Although crustal melts are regionally widespread in FMC, the ASGL Pluton lacks obviously crustally-derived granites, such as strongly peraluminous cordierite- or muscovite-bearing granites that are ubiquitous elsewhere in the FMC. In addition, the lack of well-defined mixing trends for major elements, trace elements and isotopes suggests that magma mixing was not a dominant process. Therefore, we favor only local and limited assimilation.

The comagmatic nature of the lamprophyres and granites, as well as their very similar isotopic signatures, indicates that a third hypothesis needs to be evaluated: That the granites and lamprophyres were both produced by directly by partial melting of metasomatised mantle. Mantle peridotite or lherzolite metasomatised by either subducted metasediment or melts derived from such metasediments is widely accepted to be the source of post-collisional K-rich magmatism (e.g. Förster et al., 2019, 2020, 2021; Mallik et al., 2015). More specifically, recent studies reported that mixing between peridotite and sediment may produce rocks that are likely to be

quartz normative (Förster et al., 2020, 2021) with major- and trace-element patterns similar to those in the Aigoual dykes.

The available data clearly rule out the possibility that the granites formed by fractional crystallization of a lamprophyric parent magma. It is also very unlikely that the granites formed predominantly from a crustally derived magma because the granites have typical SiO<sub>2</sub> contents, yet are strongly enriched in mantelitic elements. They also have isotopic compositions that are very similar to the lamprophyres. The ages of the inherited zircon crystals in the granites do indicate some assimilation of Variscan basement, but the range of  $\epsilon^{\text{Hf}}$  values (approximately -8 to +4) for these zircons relative to  $\epsilon^{\text{Hf}}$  in the magmatic zircons (90% between approximately -5 and -2) clearly indicates that this Variscan basement was not a major source of magma to the granites. The data are consistent with the granite magma forming via the partial melting of a mantle source metasomatised in the manner defined, with a minor amount of assimilation of Variscan crust to explain the inherited zircons. As the granite and lamprophyre magmas are isotopically very similar at source, the major and trace element arrays defined by the granitic and lamprophyric components may be shaped by mixing between these magmas that is invisible from an isotopic perspective.

### **5.2.3 Texture of the microgranites**

The preservation of the studied composite dykes and the fine-grained, quench textures in the granites may be a direct consequence of the presence of carbonate in the system. The absence of clinopyroxene in the lamprophyres and its presence in the granites may be explained by the textural association of biotite/phlogopite with carbonate pseudomorphing a prior mafic mineral in the lamprophyres. This reaction replacing clinopyroxene and/or olivine can be explained as a response to increasing  $a\text{CO}_2$  and  $a\text{H}_2\text{O}$  in the lamprophyric magma during crystallization (Blank

and Brooker, 1994; Solomatova et al., 2020). As the presence of carbonate facilitates partial melting of the mantle (e.g., Dasgupta et al., 2013), carbonated portions of the metasomatised mantle source are inferred to have melted preferentially to produce the lamprophyres.

The CO<sub>2</sub> plays a fundamentally different role in the melting of mantle and crustal rocks and its solubility is strongly dependent of pressure, melt composition, particularly SiO<sub>2</sub> content (Blank and Brooker, 1994; Ni and Keppler, 2013). During melting of mafic and ultramafic rocks at mantle pressures, the solubility of CO<sub>2</sub> increases with increasing pressure and decreasing SiO<sub>2</sub> contents (Brooker et al., 2001). CO<sub>2</sub> has a very low solubility in felsic melts at crustal pressures, whilst H<sub>2</sub>O solubility is high, consistent with the powerful effect of H<sub>2</sub>O in depressing the granite solidus (Blank and Brooker, 1994; Brooker et al., 2001). The consequence of CO<sub>2</sub> being present in these systems is to raise the solidus temperature by lowering the water activity (Scaillet et al., 1997). Therefore, CO<sub>2</sub>-fluids derived from cooling mantle-derived magmas that intruded the crust may limit the possibilities for complete mixing between the mafic and felsic magma at shallow levels. Our study potentially records an example of this behavior, in which the mantle-derived lamprophyre magmas quenched the granitic magmas with which they mingled, once carbonate saturation of the mafic magma occurred.

### **5.3 Implications for crustal growth**

In any magmatic episode, the ratio of new mantle-derived magma relative to that derived from the partial melting of older crustal material, determines the amount of crustal growth achieved. In the case of the FMC, a large proportion of the magmatic rocks (MPG, CPG and Velay migmatites) were derived from melting of local crust. Therefore, these rocks did not contribute to crustal growth and corresponded to pure crustal recycling (e.g. Moyen et al., 2017). On the other hand, vauquerite and the KCG granites contain a contribution of mantle-derived

material and thus, to some degree, represent the formation of new continental crust (Couzinié et al., 2016; Moyen et al., 2017). Couzinié et al. (2016) has demonstrated that Hf–O isotopes in zircon in this case do not identify additions of new mantle-derived material to the crust. Therefore, the process of crustal growth may be underestimated throughout geological time, since many studies were carried out relying on the zircon record (e.g., Belousova et al., 2010; Hawkesworth and Kemp, 2006).

This study has illuminated the reasons for this, clearly mantle derived magmas such as the vaugnerites in this study, are derived from mantle source rocks with sufficient degrees of metasomatism by a subducted crustal component that conventional radiogenic isotopic systems, such as Rb–Sr, Sm–Nd and Lu–Hf, as well as O isotope ratios, attain values consistent with their derivation from the continental crust. The high  $\delta^{18}\text{O}$  of clay-rich sediments and the low abundance the relevant radiogenic precursor isotopes in the mantle, result in a relatively small proportion of sediment-rich crustal material (< 30%) recycled into the mantle being sufficient to control all incompatible elements contents and form the “crust-like” isotopic signature observed in mafic post-collisional magmatism (Couzinié et al., 2016; Jacob et al., 2021). However, in detail, the calculated mantle:crust ratio may be dependent on the element, or isotopic system used, as well as the exact composition of deep crustal contaminant and the mechanism of metasomatism (Couzinié et al., 2016; Janoušek et al., 2020; 2021).

Our results demonstrate that between 20 and 35% of the budget of Sr, Nd, Hf and other incompatible elements comes from the crust and therefore does not constitute additions of new material. However, at the same time, the budget of compatible elements such as Mg, Fe, Ni, Cr is strongly controlled by the mantle component and represents addition of new material to the crust. In the lamprophyres, the mafic character and dual geochemical signature displays mantle and

crustal contributions. Even though the mantle contribution is hidden from an isotopic point of view, between 65 to 80% of material comes from the mantle. In the case of the Aigoual composite dykes, this broad finding applies to modelled mantle crust ratios constrained on the basis of radiogenic isotope ratios, trace element compositions and oxygen isotope ratios (Fig. 13, 14 and 15).

Although less evident, the same process occurs in the granites of composite dykes. Assimilation of a minor amount of crustal material is likely to be involved in their petrogenesis, but the relatively high Cr, Ni, also FeO, and MgO contents in relation to purely crustally-derived granites in the FMC reflects the mantle component involved in their genesis. The intimate relation (physically and geochemically) between lamprophyre and granite described in this work from the composite dykes at northern border of the Aigoual granite provides constraints on the mantle contribution in post-collisional granitoids from the FMC. The fact that the chemistry of the Aigoual granite lies at the mafic end of the trend defined by the granites from the composite dykes, as well as the common occurrence of rounded lamprophyric enclaves in the granite, suggests that it formed by mixing with approximately the same amount of lamprophyric magma as the most mafic granites in the composite dykes.

High-K–Mg mafic rocks are low in volume in the FMC, forming ~ 10% outcrops, and therefore their contribution to crustal growth may be relatively minor. However, the findings of this study demonstrate a significant fraction of mantle material in the granites of the composite dykes, in the Aigoual granite and, thus, likely also in other KCG granites in the FMC (ca. 25% of outcrop area). Considering that between 65% and 80% of the material from lamprophyres and KGC granites came from the mantle and both represent < 35% of granitoids outcrops in the FMC, approximately 22 to 28% of the magmatic material represented by the KGC likely came

from the mantle and, despite the crustal-like incompatible element contents and isotopic signatures, represents a substantial new addition to the crust.

## **6. Conclusions**

In summary, lamprophyric-granitic composite dykes from the northern border of the Aigoual granite (southeastern Massif Central, France) record interaction between the mantle and crust in the source of the magmas. The intrusion of these magmas into the late/post-orogenic continental crust represents significant long-term additions of new crust with excellent potential for preservation. Their intrusion was coeval with the emplacement of Aigoual granite between 311 and 313 Ma, which shares their source but underwent more complete homogenization because the magmas that formed the granite did not freeze in their conduits. Crystallization of the granitic component of the composite dykes in the conduits was likely a consequence CO<sub>2</sub>-saturation of the lamprophyric component and consequent quenching of the granite portion. Lamprophyres display crust-like signatures in their Sr, Nd, Hf, O isotopes and other incompatible elements, and were the product of 10–20% of partial melting from metasomatised mantle. This mantle can be modelled by mixing between 20 and 35% of sediments from northern Gondwana margin with 65 to 80% of depleted mantle. The melting processes occurred at shallow depths in the spinel stability field. Granites have a mantle-derived component, evident from their high Cr, Ni, FeO and MgO contents compared with typical anatectic granites in the FMC. In terms of crustal growth, the results imply that between 22 and 28% of magmatic material in the FMC was mantle-derived. This study demonstrates that, although hidden by crustal signatures in terms of radiogenic isotopes and incompatible elements, the mantle contribution in post-collisional magmatism can be significant. Considering their greater preservation potential throughout

geological time, post-collisional sites represent a meaningful contributor to long-term crustal growth.

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#### 1043 **Figure Captions**

1044 Figure 1. (a) General location of Variscan belt in Western–Central Europe. (b) Geological map of  
 1045 the eastern French Massif Central modified from Moyen et al. (2017). Post-collisional mafic  
 1046 magmatic rocks (vaugnerites) occurrences from Couzinié et al. (2016). (c) Geological map of  
 1047 Aigoual granite and composite dykes modified from Talbot et al. (2005). At the scale illustrated,  
 1048 it is not possible to differentiate the different lamprophyric components of the dykes from the  
 1049 granites.

1050

1051 Figure 2. Selected field photos illustrating key field relations (a) A section of a composite dyke  
 1052 with the field of view orientated perpendicular to the strike of the dyke. A typical gradational  
 1053 contact from lamprophyre (left) to granite (right) is illustrated, also showing the increasing  
 1054 proportion and size of phenocrysts of K-feldspars; (b) Field relationships between lamprophyre  
 1055 and granite showing that two different generations of lamprophyre dykes intrude a granite dyke.  
 1056 The contacts in this case are sharp with no evidence of mingling; (c) Aigoual granite with a

typical rounded enclave of lamprophyre cut by a later thin aplitic dyke. The larger pale coloured inclusions in the enclave represent partially resorbed K-feldspar phenocrysts.

Figure 3. Photomicrographs and BSE images of lamprophyres and granites from composite dykes. (a–b) Euhedral–subhedral biotite in lamprophyre; (c) Interstitial dolomite in lamprophyre associated with euhedral quartz, plagioclase and biotite; (d) Calcite associated with biotite in granite; (e) Ocellar carbonate and microcrysts of K-feldspar in lamprophyre; (f) Plagioclase and biotite phenocryst in lamprophyre; (g–h) BSE images with compositional variation in biotite/phlogopite from lamprophyre; (i) Fine-grained granite from composite dykes; (j–l) Textural variation in granite from composite dykes with phenocrysts of zoned plagioclase, K-feldspar (j), quartz (k) and corroded megacryst of K-feldspar (l). Mineral abbreviations from Warr (2021).

Figure 4. (a) Al – Mg – Fe<sup>2+</sup> ternary diagram showing compositional variations of biotite to phlogopite. Minettes and lamproites fields after Rock (1991). (b) Al<sub>2</sub>O<sub>3</sub> vs. FeO<sup>T</sup> and (c) Al<sub>2</sub>O<sub>3</sub> vs. TiO<sub>2</sub> discrimination diagram for micas after Mitchell and Bergman (1991). Abbreviations: Es = Eastonite, Ph = Phlogopite, Sd = Siderophyllite, and An = Annite.

Figure 5. Zircon U–Pb Tera–Wasserburg concordia diagrams for composite dykes and Aigoual granite. Data-point error ellipses are 2σ.

Figure 6. Diagram of <sup>206</sup>Pb/<sup>238</sup>U ages (Ma) versus ε<sup>Hf</sup><sub>(t)</sub> from zircons of composite dykes and Aigoual granite. ε<sup>Hf</sup><sub>(t)</sub> lower and upper limit for Depleted Mantle (DM) are from Næraa et al.

(2012) and Griffin et al. (2004). The KDE curve (light pink) portrays the  $^{206}\text{Pb}/^{238}\text{U}$  age distribution used to calculate the Concordia ages.

Figure 7. (a)  $\text{SiO}_2$  vs.  $\text{K}_2\text{O}$  classification diagram of Peccerillo and Taylor (1976); (b) A/CNK vs. A/NK diagram.

Figure 8. Whole-rock major- and trace-element compositions of lamprophyre and granite from composite dykes and Aigoual granite.

Figure 9. (a) Primitive mantle-normalized rare earth element (REE) patterns from Sun and McDonough (1989); (b) Multi-element normalized to Primitive mantle from Sun and McDonough (1989). GLOSS composition from Plank and Langmuir (1998); PCMM – post-collisional mafic magmatism from FMC of Couzinié et al. (2016); CAL – China – Calc-alkaline lamprophyre derived by partial melting of subducted sediments from Su et al. (2017).

Figure 10. (a)  $\text{SiO}_2$  versus  $\delta^{18}\text{O}$  quartz; (b)  $\log(\text{Ni} + \text{Cr})$  versus  $\delta^{18}\text{O}$  quartz; (c)  $\delta^{18}\text{O}$  quartz versus  $\delta^{18}\text{O}$  feldspar. Quartz–feldspar isotherms for 550 °C and 950 °C calculated using the calibrations of Chiba et al. (1989).

Figure 11. (a) Nb/Yb vs. Th/Yb diagram after Pearce (2008) suggesting the involvement of continental crust in the formation of the magmas that produced the composite dykes. (b) Ba/Rb vs. Rb/Sr diagram (after Furman and Graham, 1999) suggesting the presence of phlogopite in the mantle source region as a consequence of the metasomatism by a crustal component; (c) La/Yb

vs. Nb/La diagram (Abdel and Nassar, 2004). Granites are also plotted (in pale colors), as they are comagmatic and may be derived from similar source rocks. Primitive mantle (PM) values from Hofmann (1988).

Figure 12. Histogram of  $\delta^{18}\text{O}$  quartz analyses from this study. Mantle whole-rock O isotopic value ( $\sim 5.7\text{‰}$ ) from Eiler (2001); I- and S-type granites from Harris et al. (1997) and peraluminous granite from Oman-United Arab Emirates (UAE) ophiolite from Spencer et al. (2017).

Figure 13. (a) Results of mixing models between mantle ( $\delta^{18}\text{O} = 5.7\text{‰}$  and  $^{87}\text{Sr}/^{86}\text{Sr}_{\text{initial}} = 0.703$ ) and three different contaminants all characterised by  $^{87}\text{Sr}/^{86}\text{Sr}_{\text{initial}} = 0.715$  (GLOSS, Plank and Langmuir 1998) but having different  $\delta^{18}\text{O}$  values: carbonate-bearing sediments (28‰), pelagic clays (20‰) and average continental crust (15‰) (Eiler, 2001).

Figure 14. (a)  $\epsilon^{\text{Nd}}_{\text{initial}}$  vs.  $^{87}\text{Sr}/^{86}\text{Sr}_{\text{initial}}$  plot for composite dykes and Aigoual granite. Initial ratios calculated at 311 Ma. Lithospheric mantle melting field from Davis and Blanckenburg (1995); Vaugnerites French Massif Central (FMC) from Moyen et al. (2017); Variscan lamprophyres from Soder and Romer (2018). The mixing calculations used the Variscan mantle composition from Pin and Paquette (1997), GLOSS from Plank and Langmuir (1998) and FMC paragneiss compositions from Moyen et al. (2017); (b)  $\epsilon^{\text{Hf}}_{\text{initial}}$  vs.  $\epsilon^{\text{Nd}}_{\text{initial}}$  plot for composite dykes and Aigoual granite. Data from MORB and OIB are taken from Stracke (2012) and mantle array regression line from Chauvel et al. (2008). Global kimberlites field from Tappe et al. (2018). DM = depleted mantle; OIB = ocean island basalt.

1126

1127 Figure 15. (a) La/Yb vs. Yb plot with batch melting models for phlogopite-bearing spinel and  
1128 garnet peridotite. The calculated sources correspond to Depleted Mantle (Workam and Hart  
1129 2005) mixed with 20% to 30% GLOSS (Plank and Langmuir 1998). Modal proportions in the  
1130 residue are recalculated from Condamine and Médard (2014) for spinel peridotite (olivine 0.69,  
1131 orthopyroxene 0.15, clinopyroxene 0.08, phlogopite 0.07, spinel 0.01) and Thibault et al. (1992)  
1132 for garnet peridotite (olivine 0.25, orthopyroxene 0.50, clinopyroxene 0.15, phlogopite 0.03,  
1133 garnet 0.07). Partition coefficients for mafic and intermediate rocks from Laurent (2012). (b)  
1134 Multielement patterns of lamprophyres and granites and calculated compositions generated by 5,  
1135 10 and 20% of partial melting of a phlogopite-bearing spinel peridotite source. Black and gray  
1136 continuous lines represent partial melting of a calculated source with DM + 20% GLOSS and  
1137 blue dashed lines of DM + 35% GLOSS. Partition coefficients from mafic and intermediate rocks  
1138 from Laurent (2012). The melt composition from calculated source replicates the multi-element  
1139 patterns exhibited by lamprophyres and granites well, except for the LREE.

1140

1141     **Supplementary Material**

1142     Supplementary Figure 1. BSE images and X-ray maps of three different magmatic textures of  
1143     carbonate.

1144

1145     Supplementary Figure 2. (a) Ternary diagram of classification of feldspars; (b) Ternary diagram  
1146     of classification of clinopyroxene; (c) (d) Amphibole classification.

1147

1148     Supplementary Figure 3. Selected CL images of zircon from composite dykes and Aigoual  
1149     granite with U–Pb age and  $\epsilon^{\text{Hf}}$  values.

1150

1151     Supplementary Data 1. Analytical methods.

1152     Supplementary Data 2. Coordinates and lithologies of the samples taken from composite dykes in  
1153     the Aigoual granite.

1154

1155     Supplementary Data 3

1156     Supplementary Data 3 Table S1. Whole-rock major- (wt.%) and trace-element (ppm)  
1157     compositions of composite dykes from Aigoual granite, French Massif Central.

1158     Supplementary Data 3 Table S2. Whole-rock Sr, Nd, Hf isotopes.

1159     Supplementary Data 3 Table S3.  $\delta^{18}\text{O}$  values from quartz and feldspar from lamprophyre and  
1160     granite of composite dykes and Aigoual granite (AG14A).

1161

1162     Supplementary Data 4

1163     Supplementary Data 4 Table S1. Zircon LA-SF-ICP-MS U–Pb geochronology data.

1164    Supplementary Data 4 Table S2. Analytical results for zircon Lu–Hf isotopes determinations.

1165

1166    Supplementary Data 5

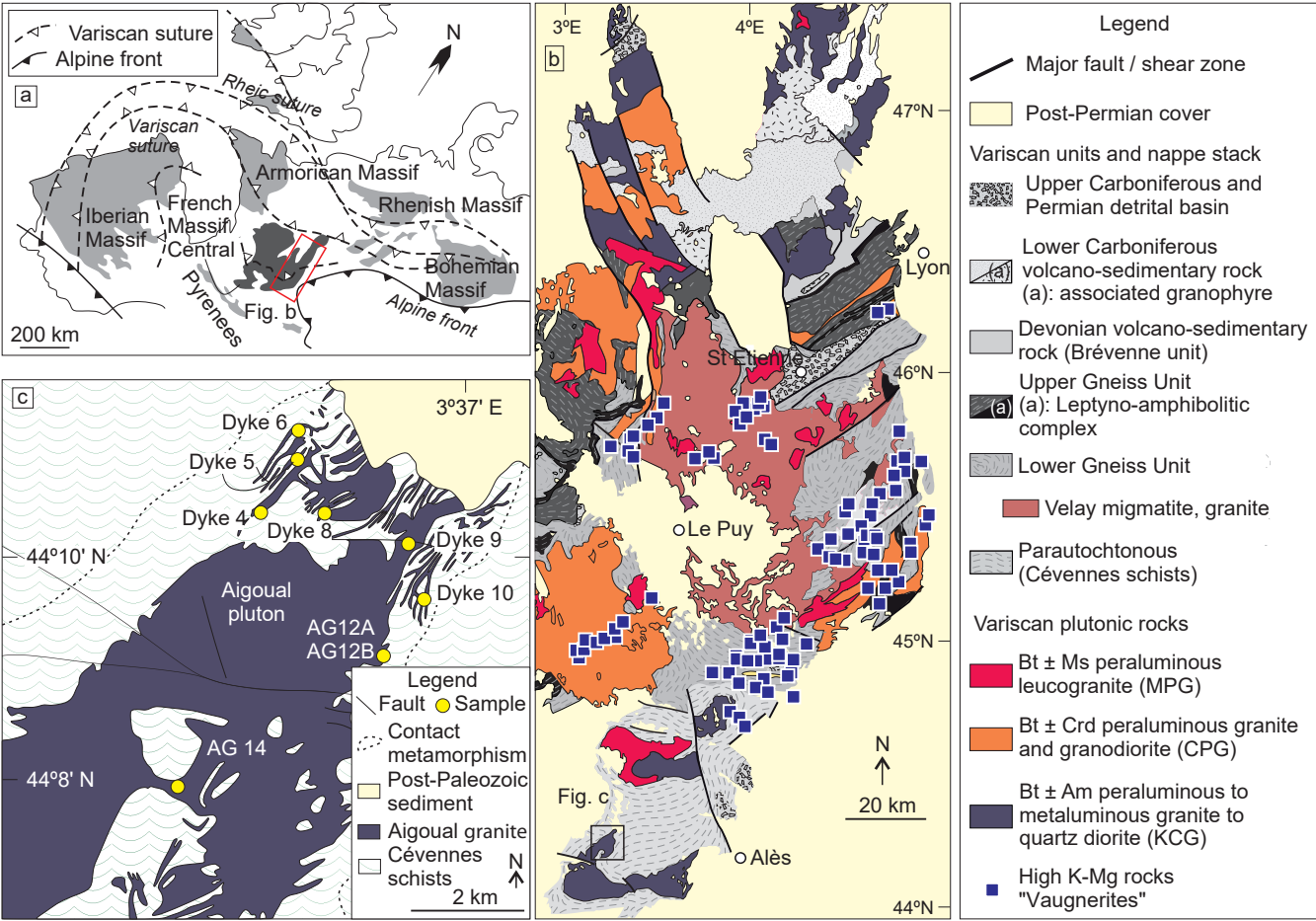
1167    Supplementary Data 5 Table S1. Mineral chemistry of biotite/phlogopite

1168    Supplementary Data 5 Table S2. Mineral chemistry of feldspars

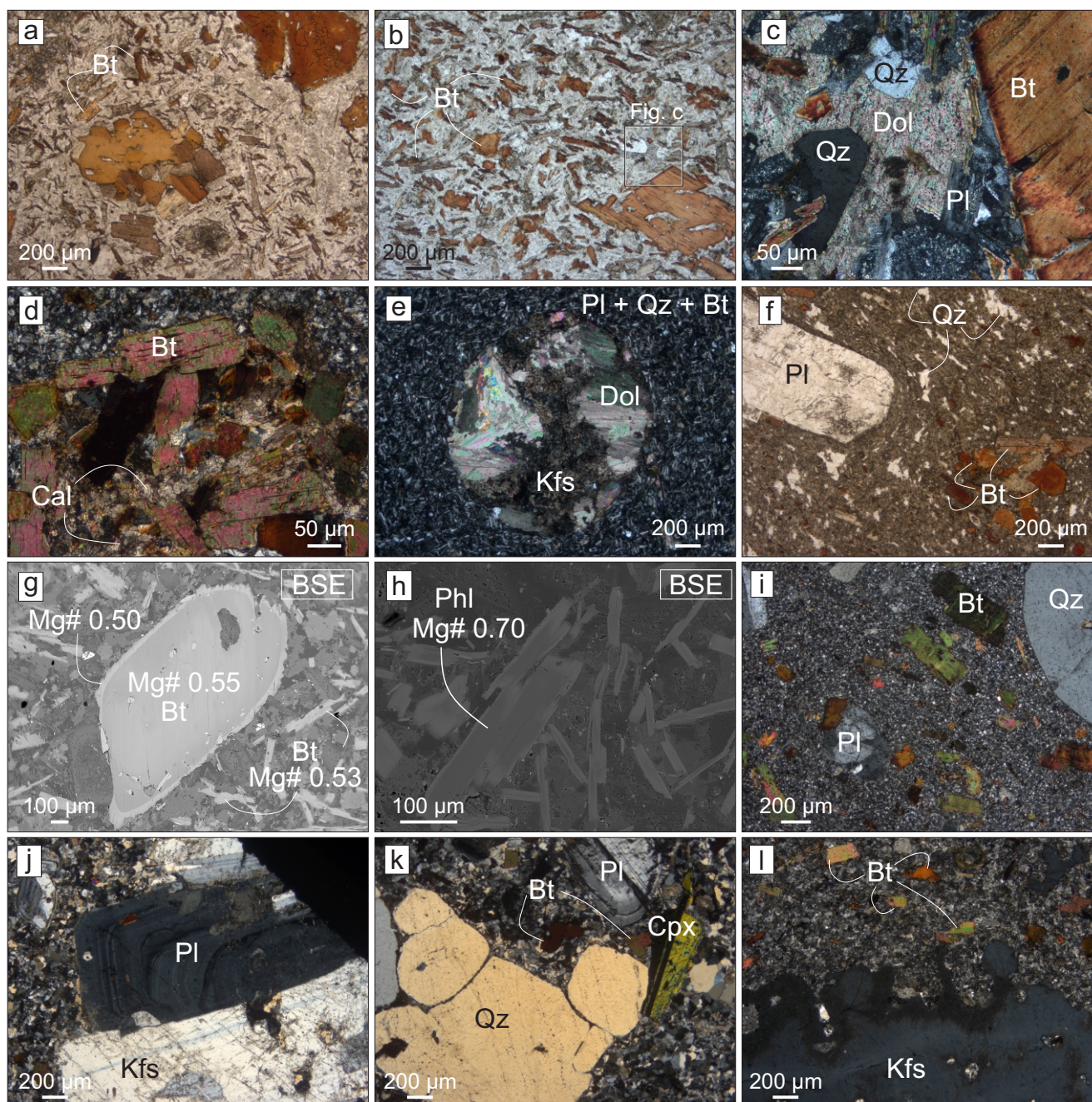
1169    Supplementary Data 5 Table S3. Mineral chemistry of clinopyroxene

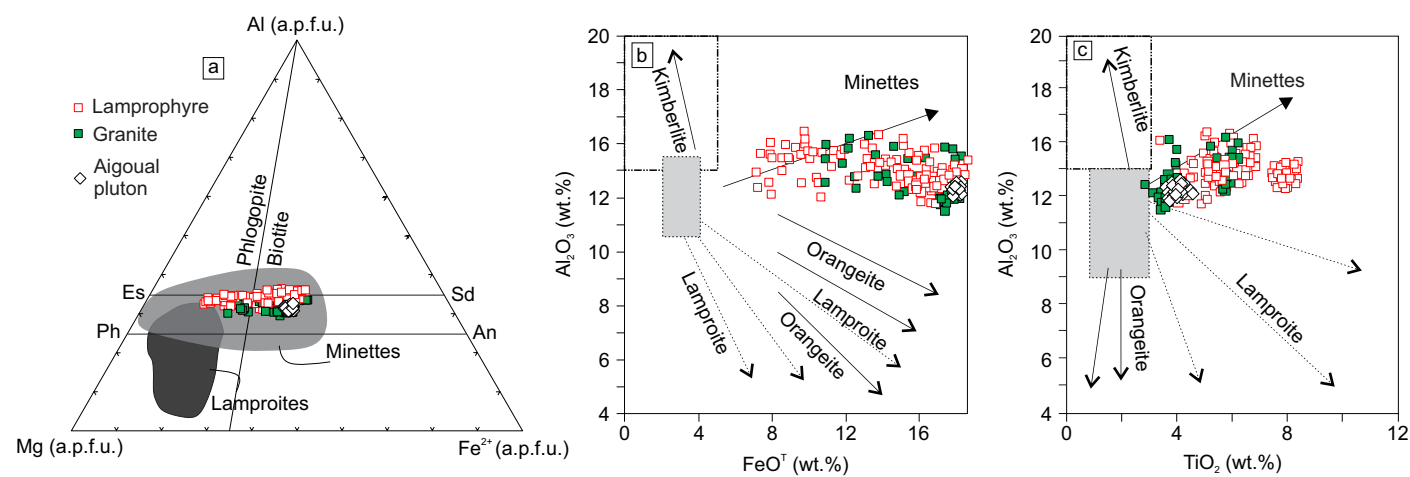
1170    Supplementary Data 5 Table S4. Mineral chemistry of amphibole

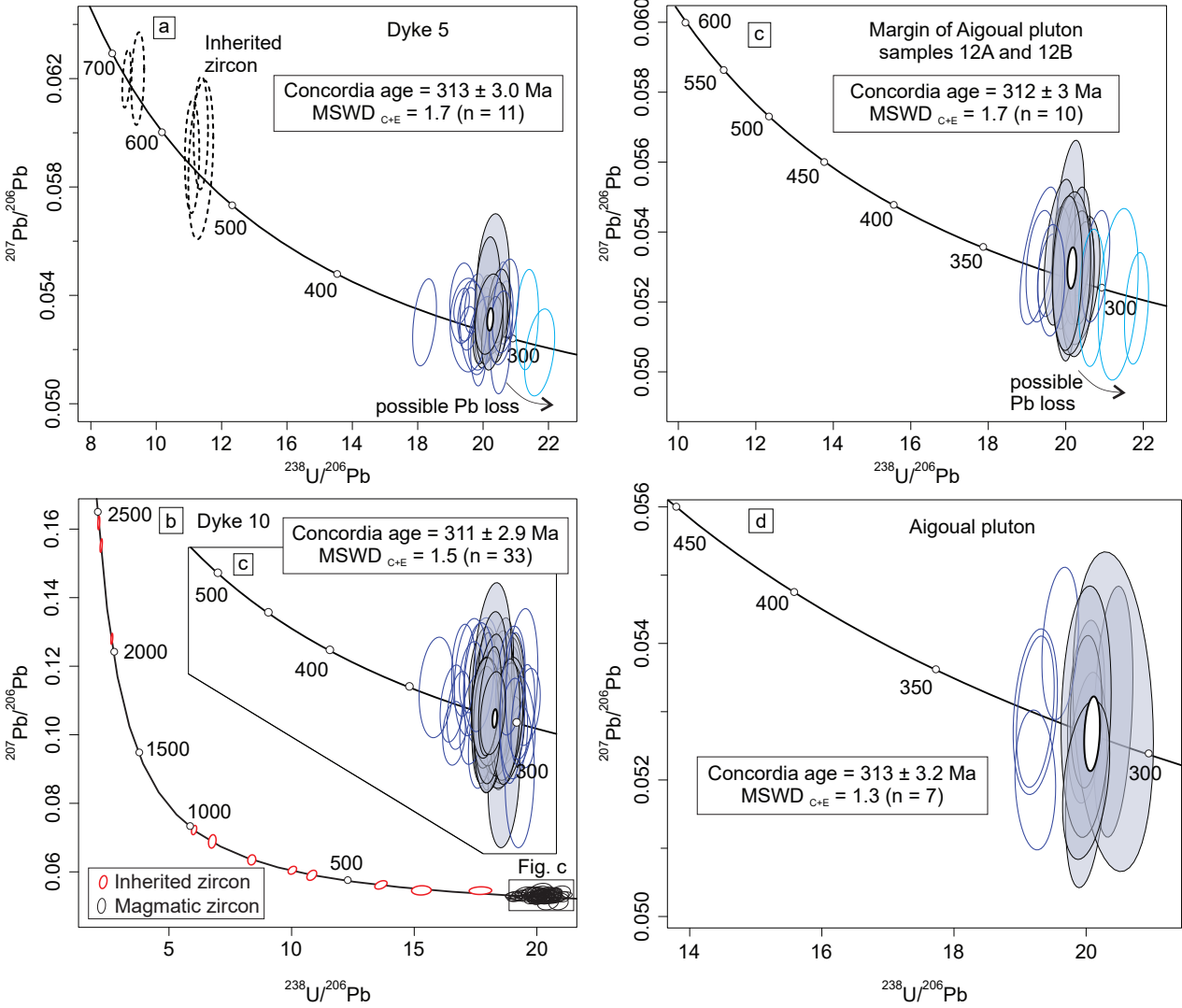
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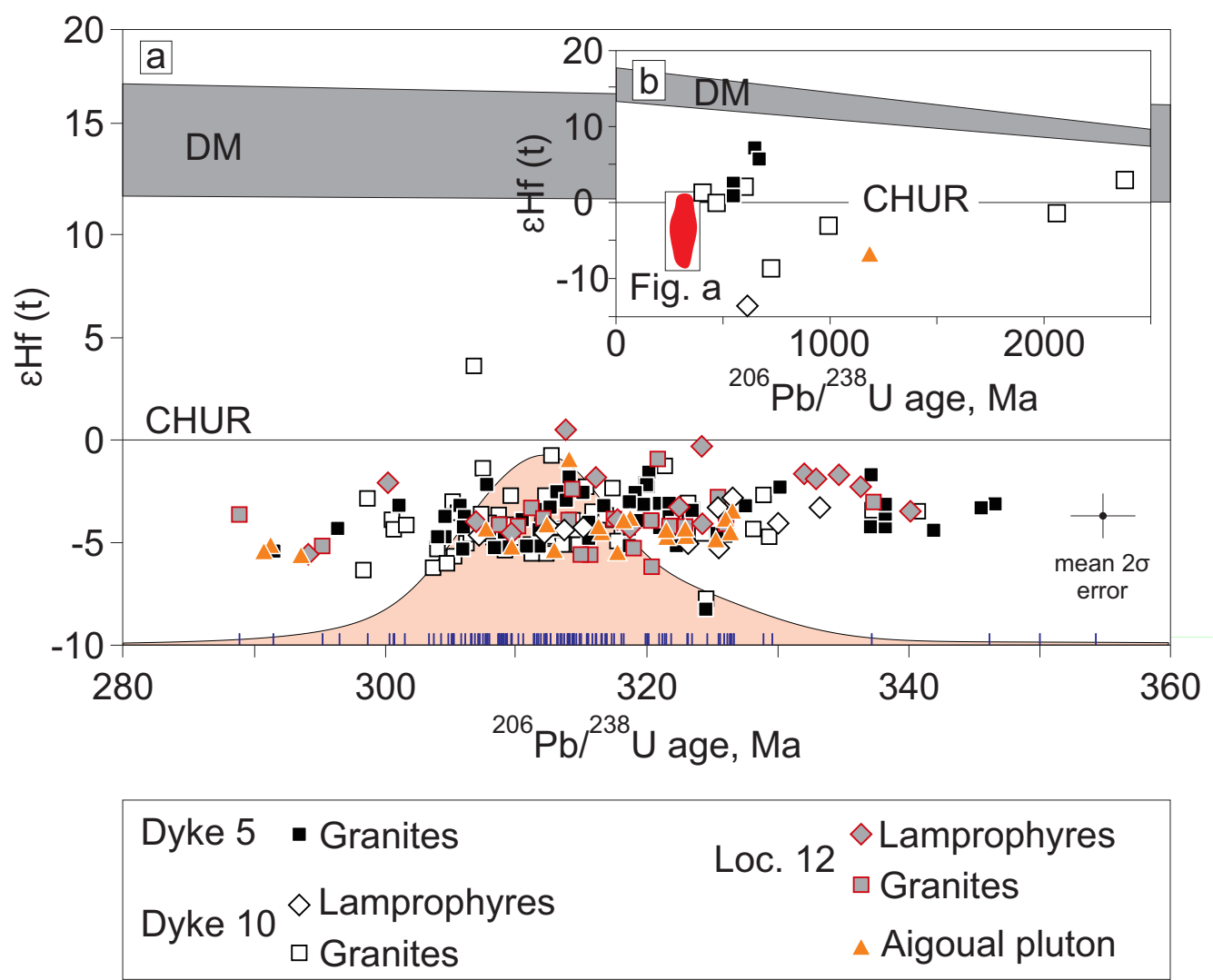


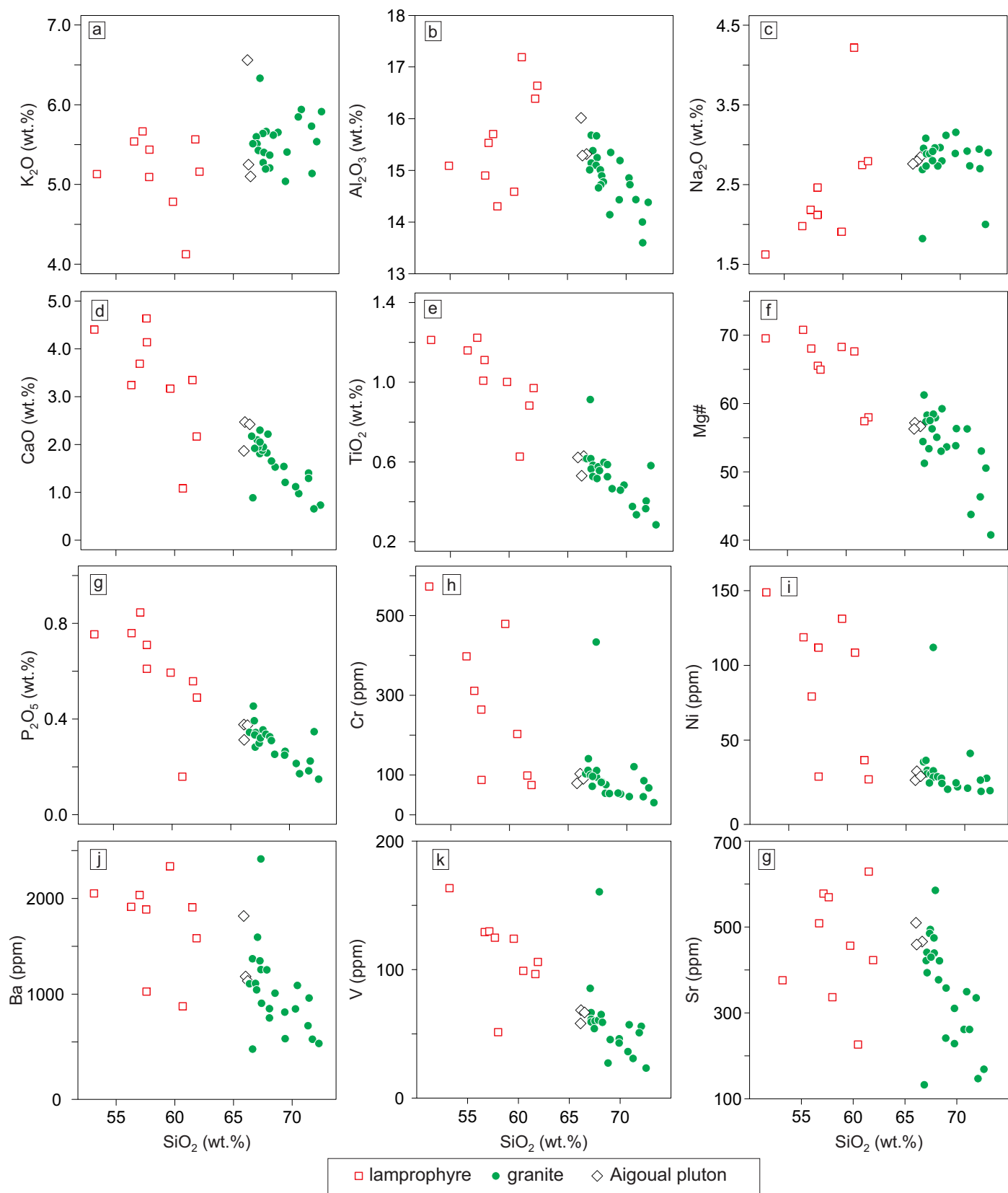


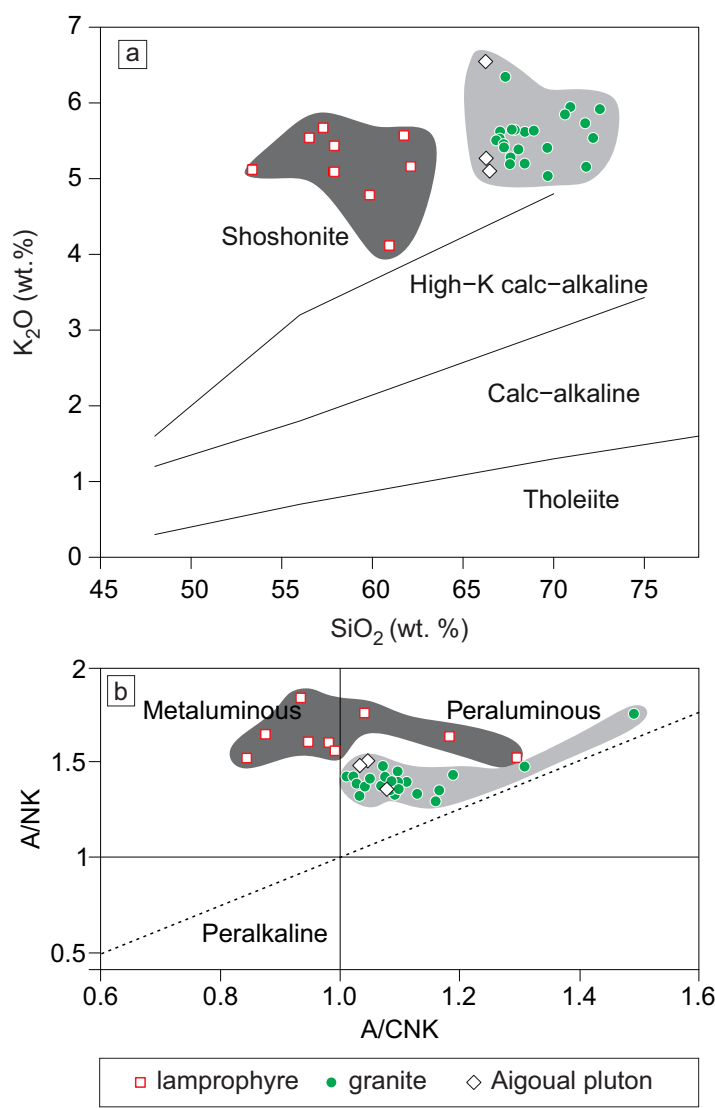


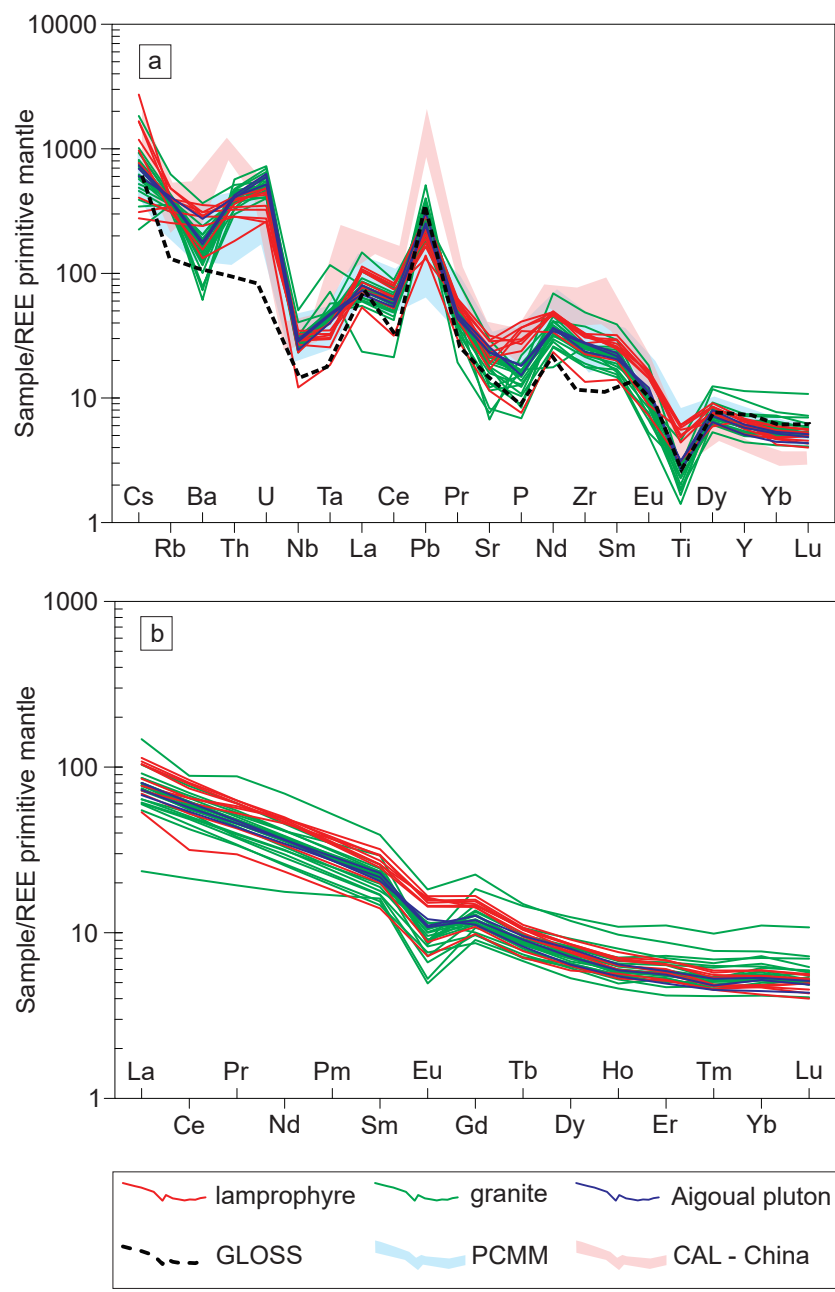


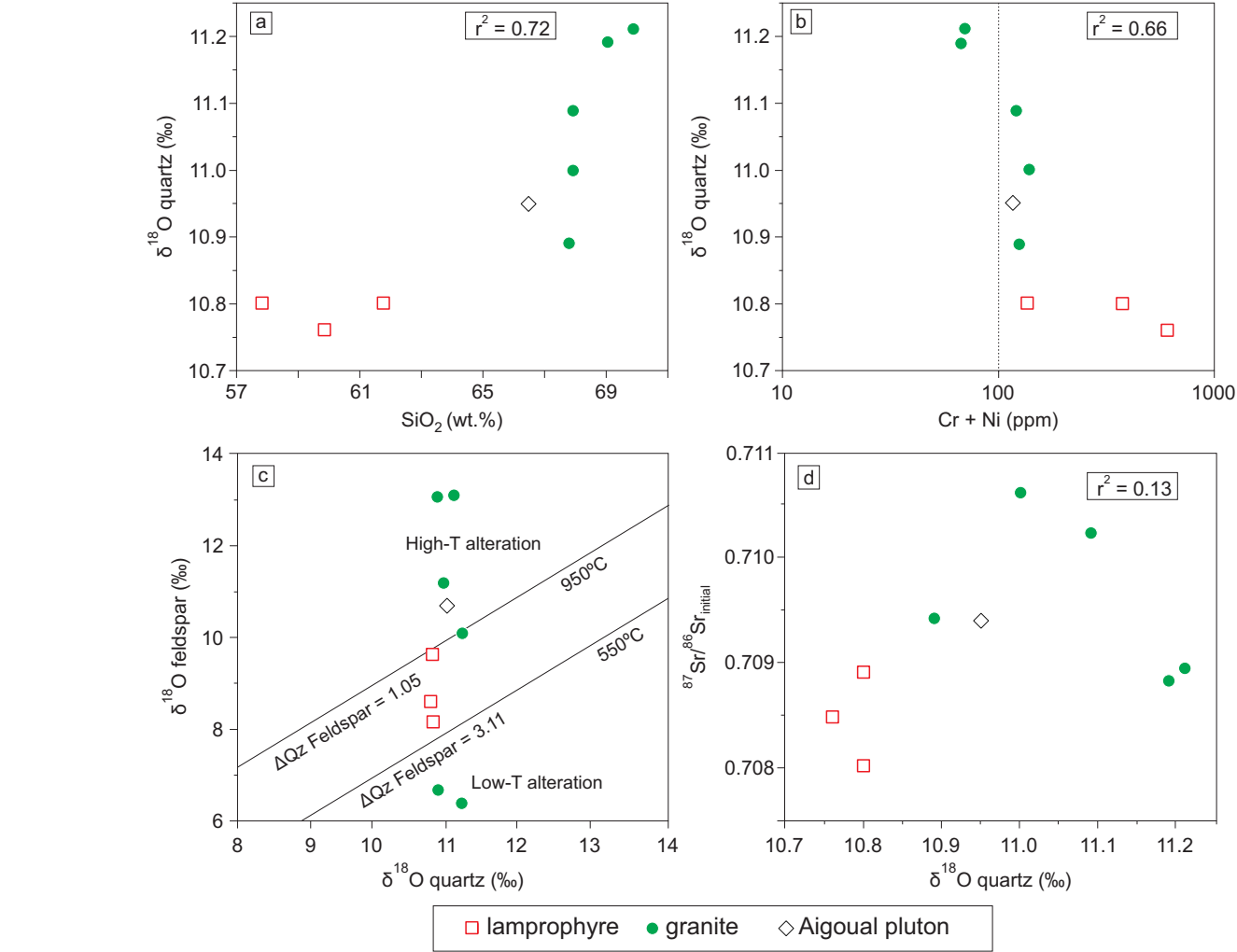


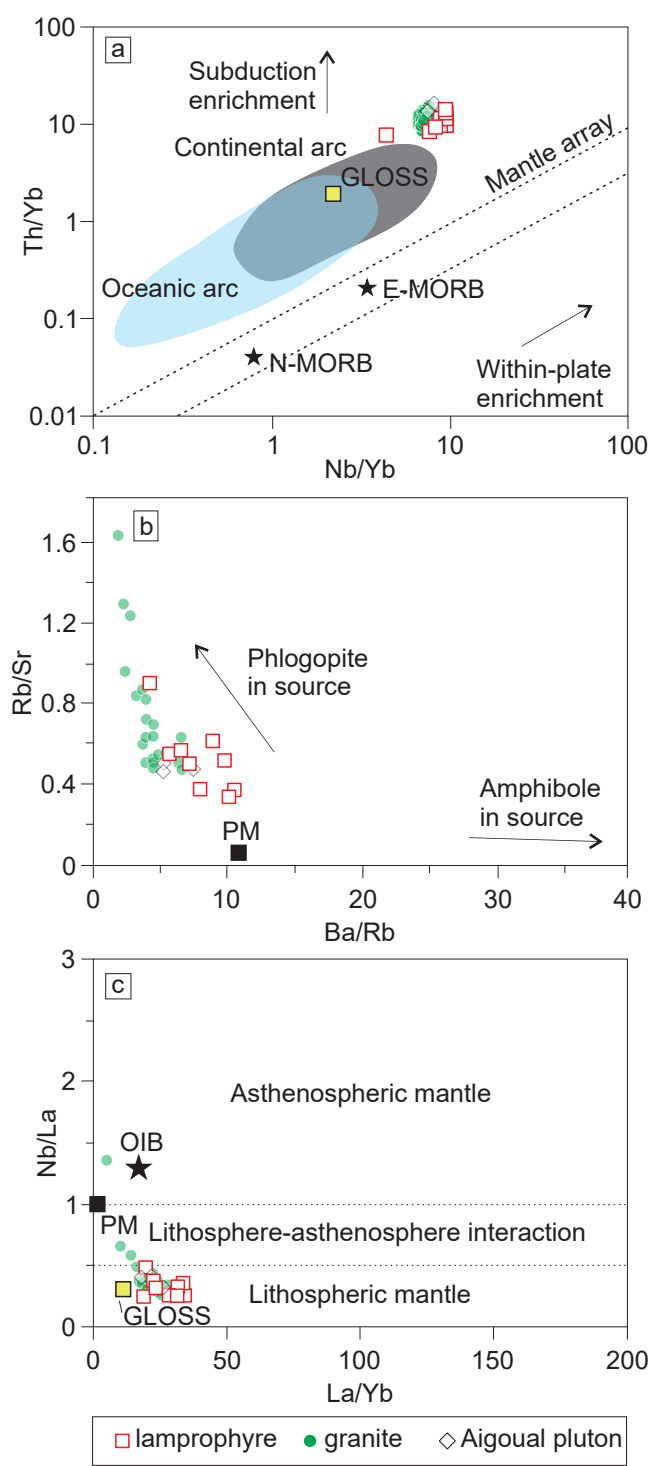


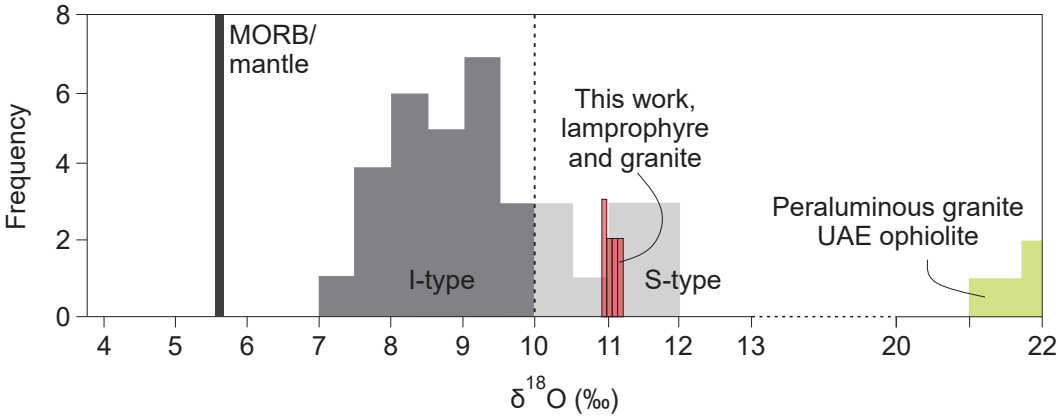


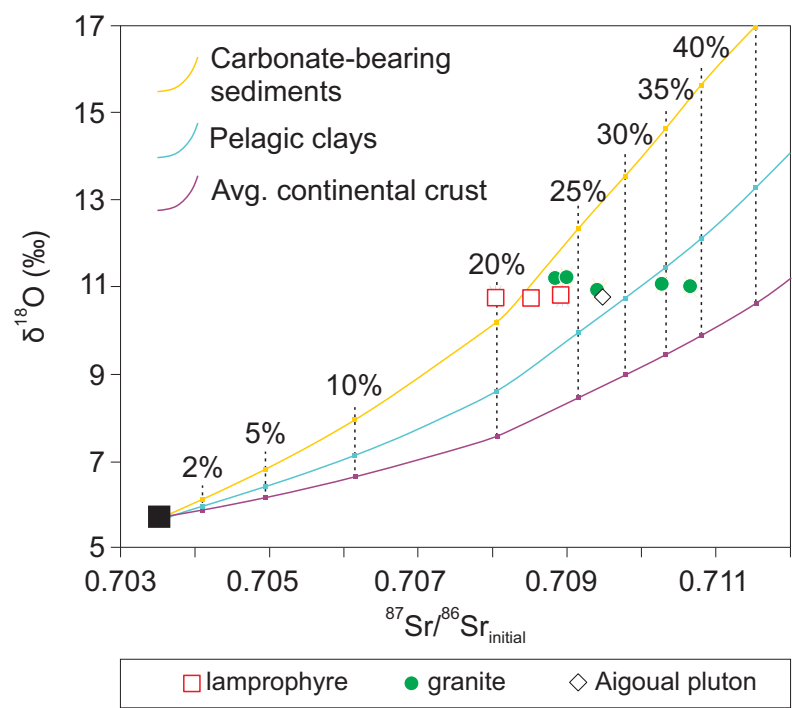


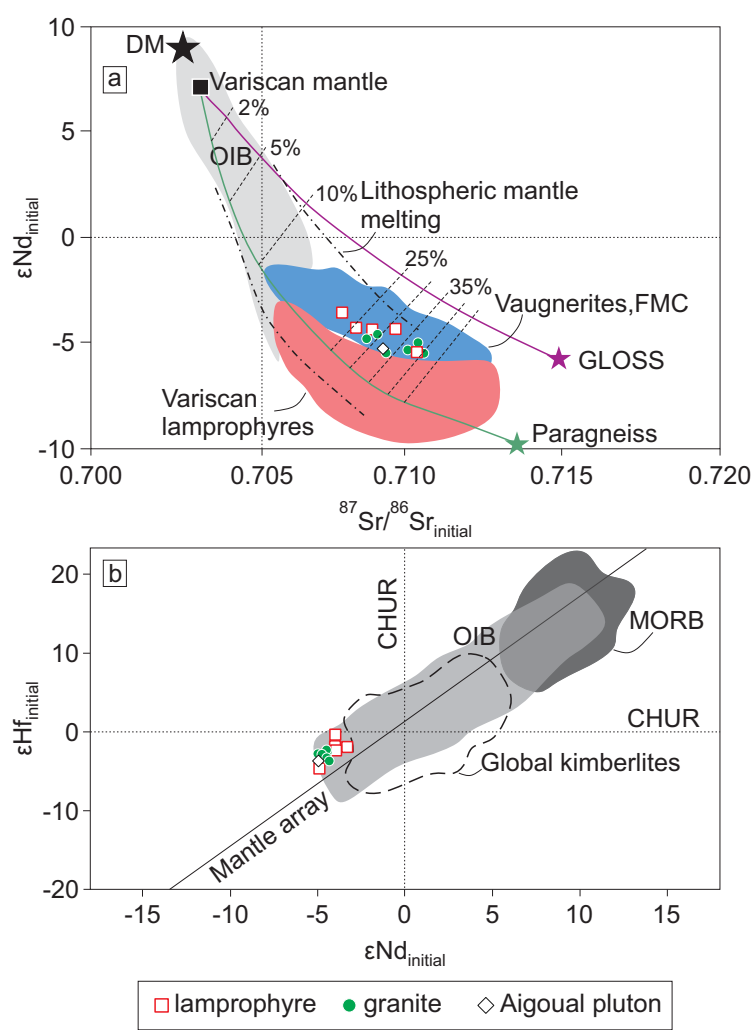




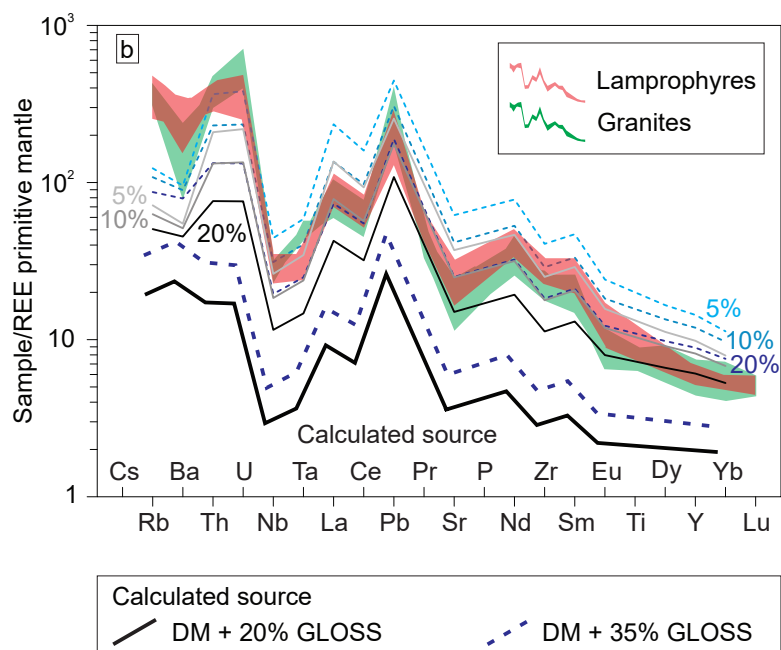


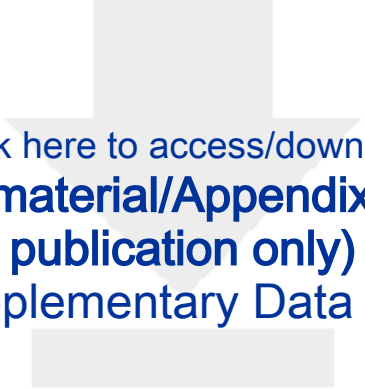






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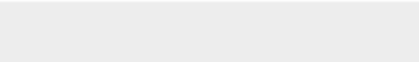
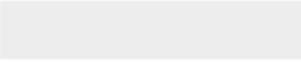




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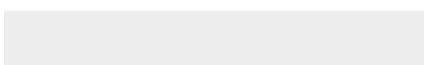
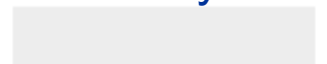




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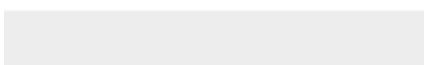
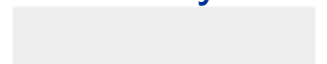




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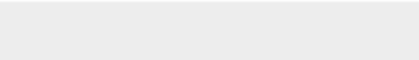




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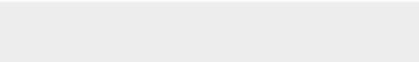




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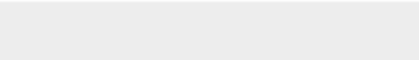


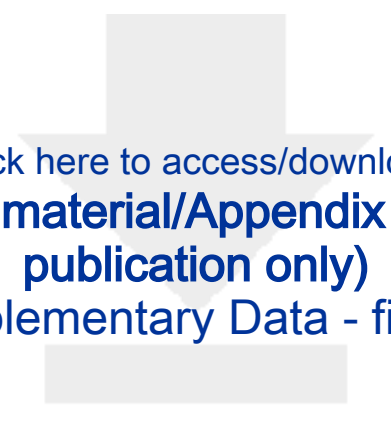


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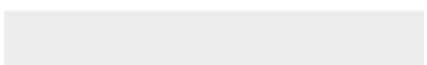




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☒ The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

☐The authors declare the following financial interests/personal relationships which may be considered as potential competing interests: