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# Polyphased rare-element magmatism during late orogenic evolution: geochronological constraints from NW Variscan Iberia

Jérémie Melleton[1,3,*], Eric Gloaguen[1,2], Dirk Frei[3,4], Alexandre Lima[5,6], Romeu Vieira[7] and Tania Martins[8]

[1] BRGM, Georesources Division, ISTO, UMR 7327, 3 avenue Claude Guillemin, B.P.6009, 45060 Orléans Cedex 2, France
[2] Université d'Orléans, ISTO, UMR 7327, 45071 Orléans, France
[3] Department of Earth Sciences, University of Western Cape, Cape Town, South Africa
[4] GEUS, Østervoldgade 10, DK-1350 Copenhagen K, Denmark
[5] Department of Geosciences, Environment and Spatial Planning, Faculty of Sciences, University of Porto, Porto, Portugal
[6] ICT (Institute of Earth Sciences), Porto pole, Portugal
[7] Fortescue Metals Group, Perth, Australia
[8] Manitoba Geological Survey, 360-1395 Ellice Av., Winnipeg, MB, Canada

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**Abstract –** Rare-element granites and pegmatites represent important sources of raw materials for "clean, green and high technologies", such as lithium and tantalum, for example. However, mechanisms of rare-element granites and pegmatite's origin are still far from being fully understood. Several rare-element pegmatite fields and a rare-element granite are known in the Variscan realms located in Iberia (Spain and Portugal), enhancing the interest of this area for studying the formation of these extremely fractionated melts. In situ U-Pb dating by LA-SF-ICP-MS of columbite-group minerals from rare-element granites and pegmatites of the Iberian Variscan belt provides new constraints on the generation of rare-element melts. Three events have been recognized: (i) Emplacement of the Argemela rare-element granite, in the Central Iberian Zone (CIZ), with an age of 326 ± 3 Ma; (ii) Emplacement of rare-element pegmatites from the Galicia-Trás-os-Montes Zone (GTOMZ), at an average age of 310 ± 5 Ma; (iii) Emplacement of rare-element pegmatites in the CIZ and in the southern GTOMZ at about 301 ± 3 Ma. These two last events are coeval with the two peaks of ages for the late orogenic magmatism at ca. 308 Ma and 299 Ma, and all dated rare-element pegmatites clearly emplaced during the late-orogenic evolution of the Variscan belt. Contemporaneous fields of rare-element pegmatites are arranged in belts following those formed by similar granitoid suites. Pegmatite fields from both the GTOMZ and the CIZ reveal a southward propagation of ages of emplacement, which matches the observed propagation of deformation, metamorphism and magmatism in the two different geotectonic zones. Existence of three successive rare-element events in the Iberian Massif argues against the involvement of lower crustal HP-HT metamorphism in the generation of rare-element melts. Possible sources of rare-element-enriched melts are more likely located in the middle to upper crust, as are the major components of granitic magmatism. Analyses of U and Pb isotopes from columbite-group minerals are very robust and reproducible, making them good candidates for dating ore deposits related to peraluminous magmatism as well as REE- and Nb-bearing deposits.

**Keywords:** rare-element granite and pegmatite / columbite group minerals / LA-SF-ICPMS dating / Variscan orogeny / Iberian Massif

**Résumé – Magmatisme à métaux rares polyphasé au cours de l'évolution tardi-orogénique : contraintes géochronologiques depuis le NO de l'Ibérie varisque.** Les granites et pegmatites à métaux rares représentent des ressources importantes en matières premières minérales utilisées par les technologies de la transition énergétique et de la télécommunication, comme le lithium ou le tantale. Cependant, les processus géologiques à leur origine sont encore peu compris. Plusieurs champs de pegmatites et un granite à métaux rares sont connus dans les formations varisques de l'Ibérie (Espagne et Portugal), ce qui en fait une

*Corresponding author: j.melleton@brgm.fr

aire d'étude d'intérêt pour l'étude de la formation de ces magmas extrêmement différenciés. La datation U-Pb in situ par LA-SF-ICP-MS des minéraux du groupe de la colombite extraits de granites and pegmatites à métaux rares de la chaine varisque ibérique apporte de nouvelles contraintes sur la genèse des magmas à métaux rares. En effet, trois événements distincts ont été reconnus : (i) La mise en place du granite à métaux rares d'Argemela dans la zone centre ibérique (CIZ) à 326 ± 3 Ma ; (ii) La mise en place de pegmatites à métaux rares dans la zone de Galice-Trás-os-Montes (GTOMZ), à un âge moyen de 310 ± 5 Ma ; (iii) La mise en place de pegmatites à métaux rares dans la CIZ et le sud de la GTOMZ à environ 301 ± 3 Ma. Ces deux derniers événements sont contemporains des deux pics de magmatisme tardi-orogénique à environ 308 Ma and 299 Ma. Toutes les pegmatites à métaux rares datées au cours de cette étude se sont mises en place durant l'évolution tardi-orogénique de la chaine varisque. Les champs de pegmatites à métaux rares contemporains sont organisés en ceintures similaires à celles des suites de granitoïdes. Les champs de la GTOMZ et de la CIZ montrent une propagation vers le sud des âges de mises en place, ce qui est cohérent avec la propagation observée pour la déformation, le métamorphisme et le magmatisme dans les deux domaines géotectoniques. L'existence de trois épisodes successifs de magmatisme à métaux rares dans le massif ibérique est un argument contre la participation de processus métamorphiques de hautes pressions et hautes températures infracrustaux dans la genèse des magmas à métaux rares. Leurs sources possibles sont plus probablement localisées dans la croute moyenne à supérieure, comme le magmatisme granitique contemporain. Les analyses des isotopes de l'uranium et du plomb des minéraux du groupe de la colombite sont robustes et reproductibles, ce qui en fait un excellent outil pour la datation des gisements à métaux rares.

**Mots clés :** granites et pegmatites à métaux rares / minéraux du groupe de la colombite / LA-SF-ICPMS / orogénèse varisque / Massif Ibérique

## Introduction

Lithium and tantalum are key metals for the development of new technologies. Rare-element granites and pegmatites are amongst the most important sources of raw materials for these metals (Linnen *et al.*, 2012). They are also storehouses for many industrial minerals such as feldspar, quartz, mica and kaolin (Glover *et al.*, 2012). Rare-element granites and pegmatites generally form volumetrically small bodies, corresponding to small batches of enriched magma. Magmatic processes can lead to rare-element-enriched magma in two ways: (A) low melting rate resulting in small melt fractions from a crustal source with a restricted composition or partial melting of rare-element rich sediments in shear zone context (Stewart, 1978; Melleton *et al.*, 2012; Deveaud *et al.*, 2013, 2015; Shaw *et al.*, 2016; Simmons *et al.*, 2016; Müller *et al.*, 2017; Konzett *et al.*, 2018a, b; Gourcerol *et al.*, 2019; Webber *et al.*, 2019; Ballouard *et al.*, 2020; Lv *et al.*, 2021b); or (B) a late withdrawal of residual liquid from the crystallization of parent granite (Černý *et al.*, 2005; Novak *et al.*, 2013; London, 2018; Roda-Robles *et al.*, 2018; Neiva *et al.*, 2019; Garate-Olave *et al.*, 2020; Lv *et al.*, 2021a). This latter process is the most accepted genetic model for rare-element granites and pegmatites, although fractionation between "parent" granites and rare-element magmatic bodies still has to be explained (London, 2008).

Another explanation for rare-element enrichment has been proposed: the extraction of metasomatic fluids during lower crustal granulite metamorphic processes (High Pressure-High Temperature, HP-HT) was suggested to explain the abnormal enrichment of fluxing elements (Li, F, B, Be) and High Field Strengh Elements (HFSE, mainly Nb and Ta) (Christiansen *et al.*, 1988; Marignac and Cuney, 1999; Cuney *et al.*, 2002; Cuney and Barbey, 2014; Simons *et al.*, 2016). This hypothesis is based mainly on the observation of extreme depletion of Cl, F, Large-Ion Lithophile Elements (LILE) and rare-metals in granulite and on geochronological results ($^{40}$Ar-$^{39}$Ar on lepidolite and muscovite) from the North French Massif Central, where rare-element bodies were emplaced contemporaneously at about 310 Ma, at the same time as HP-HT metamorphism affecting the lower crust of the French Massif Central.

In the northwestern Variscan realms of Iberia, rare-element pegmatites are widespread (Cotelo Neiva, 1944; Neiva, 1975, 1977; Gomes and Nunes, 1990; Charoy *et al.*, 1992; Martín-Izard *et al.*, 1992; Roda, 1993; Fuertes Fuente and Martin-Izard, 1998; Neiva *et al.*, 2008, 2012; Neiva and Ramos, 2010; Antunes *et al.*, 2013; Lima *et al.*, 2014; Roda-Robles *et al.*, 2016, 2018). In addition, the Argemela granite, studied by Charoy and Noronha (1991, 1996), Michaud *et al.* (2020) and Michaud and Pichavant (2020) presents all the characteristics of a rare-element granite. Rare-element pegmatites occur in geographically restricted fields and, for this reason, at least seven main rare-element pegmatite fields are recognized. Previous studies, based mainly on geochemical data, suggested a genetic relationship between rare-element pegmatites and surrounding granitoids (Gomes and Nunes, 1990; Fuertes Fuente and Martin-Izard, 1998; Lima, 2000; Almeida *et al.*, 2002; Neiva *et al.*, 2008, 2012; Neiva and Ramos, 2010; Roda-Robles *et al.*, 2018). Due to the very low number of available geochronological data on rare-element pegmatites in Iberia, as highlighted by Roda-Robles *et al.* (2018), one of the first questions is on the general timing of rare-element magmatism: is there one general event or is it polyphased? Currently, only three studies provided radiometric ages in an attempt to date emplacement of granitic pegmatites in the Iberian segment of the Variscan belt. Two of them focused on a single pegmatite field, the Fregeneda-Almendra field (Portugal and Spain) for the first one ($^{40}$Ar-$^{39}$Ar on micas; Roda *et al.*, 2009; Vieira, 2010), the second at the Tres Arroyos field (CIZ, Spain) (U-Pb by LA-ICP-MS on columbite-group minerals; Garate-Olave *et al.*, 2020). The other study (U-Pb on zircon and monazite; Lima *et* al., 2012) focuses on the Pavia field composed of barren pegmatites (*i.e.*, without minerals typical of rare-elements pegmatites, Lima *et al.*, 2014) located in the Ossa-Morena zone. In the the Fregeneda-Almendra field, authors concluded that Li-bearing pegmatites are not genetically related to their nearest granite; their conclusion is also supported by earlier structural, geochemical and mineralogical data (Roda *et al.*, 1999). For the Pavia field, pegmatites are contemporaneous to some neighbouring granites (Lima *et al.*, 2012, 2014).

Age of rare-element pegmatite and granite is usually obtained using $^{40}Ar$-$^{39}Ar$ method on micas. However, posterior disturbances can easily affect this isotopic system (Miller and Morton, 1980). Zirconium activity in rare-element granites and pegmatites is generally very low and, when zircon is present, it shows generally high U contents that lead to advanced metamictization (Raimbault, 1998). It is then difficult to use zircon to date emplacement of rare-element granite and pegmatite by U-Pb isotopes measurements. Monazite, another mineral widely used for U-Pb dating of magmatic rocks, is unfortunately rare in the pegmatites of the Li-Cs-Ta (LCT) family (Černý and Ercit, 2005) and related peraluminous rare-element granites. However, columbite group minerals (CGM) are common accessory phases of rare-element pegmatites and granites. They are ideally suited for U-Pb dating since they easily accept U in their structures and can therefore contain a significant amount of radiogenic Pb (Romer and Wright, 1992). Moreover, Pb diffusion is considered to be very low, making these minerals resistant to isotopic system resetting, together with generally very low amount of common Pb (Romer and Wright, 1992; Melleton *et al.*, 2012; Legros *et al.*, 2019). In this context, CGM are the most suitable magmatic minerals for constraining emplacement ages of rare-element granites and pegmatites and is more and more used (Romer and Wright, 1992; Romer and Smeds, 1994, 1996, 1997; Melcher *et al.*, 2008; Melleton *et al.*, 2012; Deng *et al.*, 2013; Van Lichtervelde *et al.*, 2017; Yan *et al.*, 2018; Legros *et al.*, 2019; Feng *et al.*, 2020; Garate-Olave *et al.*, 2020).

In this paper, we present new in-situ U-Pb ages obtained by laser ablation-single collector magnetic sectorfield-inductively coupled plasma-mass spectrometry (LA-SF-ICP-MS) on CGM (both columbite and tantalite) from selected rare-element granites and pegmatites of the Iberian Massif. We discuss the timing of rare-element magmatic events in the Variscan belt, temporal relationships between pegmatites and granites, and the crustal level of the magmatic sources involved. To conclude, we highlight the fact that all CGM compositions show similar behaviour of U-Pb isotopes, making the entire group suitable for dating several types of ore deposits.

## Geological setting

Located along the western border of the Iberian Peninsula, the Iberian Variscan belt represents a large segment of the European Variscan belt. The Variscan belt remnants extend for more than 8000 km from the Appalachians to the Caucasus. The belt was produced by the Paleozoic convergence and collision of two megacontinents, Gondwana in the south and Laurussia in the north, with the involvement of several microcontinents and the closure of several oceanic domains (Matte, 1986, 1991; Martínez Catalán *et al.*, 2021 and references therein).

The northwest part of the Iberian Variscan belt is subdivided into four main zones (Fig. 1): (i) the Cantabrian domain; (ii) the West Asturian Leones Zone; (iii) the Galicia-Trás-os-Montes Zone (GTOMZ) (Farias *et al.*, 1987); (iv) the Central Iberian Zone (CIZ) (Julivert *et al.*, 1974; Farias *et al.*, 1987).

Northwestern Iberia records three main phases of deformation (D1, D2 and D3), part of a long-lived deformation history developed in response to Variscan subduction, continental collision and final post-thickening extension (Matte, 1986). The ages of the three main deformation events are diachronous in time and space as a result of deformation propagation (Dallmeyer *et al.*, 1997).

All pegmatites studied are emplaced within the GTOMZ or the CIZ. The GTOMZ (Fig. 1) comprises mainly schists of the para-autochthon and allochthonous complexes. In general, D1 and pre-D1 events are preserved in allochthonous complexes. Paleozoic metasedimentary rocks from the para-autochthon have been affected mainly by D2 and D3 events and by the emplacement of numerous granitoids. The D2 event (~ 375–340 Ma) is related chiefly to final nappe emplacement, prograde metamorphism and local extensional movements. The strong E-W compressive D3 event (~ 340–310 Ma) produced upright folds at all scales and local subvertical S3 foliation. This event is related to high-T and medium-P metamorphism producing partial melting.

The CIZ (Fig. 1) is characterized by autochthonous metasedimentary formations with the predominance of a thick, monotonous Precambrian to Cambrian metapelite and greywacke sequence (Schist-Greywacke Complex) covered by Lower Ordovician quartzite (Arenig facies). Three main Variscan tectono-metamorphic stages are recognized (Ábalos *et al.*, 2002): (i) the D1 event, corresponding to the formation of straight folds associated with prograde Barrovian metamorphism and dated to about 360 Ma (Dallmeyer *et al.*, 1997); (ii) the D2 extensional event (~ 340–325 Ma), marked by local development of extensive shear zones and sub-horizontal folds and associated with varying degrees of metamorphism; and (iii) the D3 stage (~ 325–305 Ma), related to dextral and sinistral strike slip and marked by development of vertical shear zones and emplacement of synkinematic granitoids.

In northwestern Iberia, Variscan granitoids have been classified according to their age or chemical composition (review in Ábalos *et al.*, 2002). Basically, they can be subdivided into three series: i) early and late peraluminous tonalites, granodiorites and biotite granites; ii) peraluminous leucogranites; and; iii) biotite ± cordierite granites. Early peraluminous granodiorites represent a syntectonic, late-D2 to syn-D3 event, peraluminous granites, a syn-D3 event, biotite ± cordierite granites, a late-D3 event, and late peraluminous granodiorites, a post-D3 event. This is coherent with the proposed classification by Roda-Robles *et al.* (2018), *i.e.*, the five following types: i) two-mica peraluminous leucogranites; ii) P-rich highly peraluminous granites; iii) P-poor moderately peraluminous granites; iv) moderately to low peraluminous granites and v) I-type low peraluminous granites.

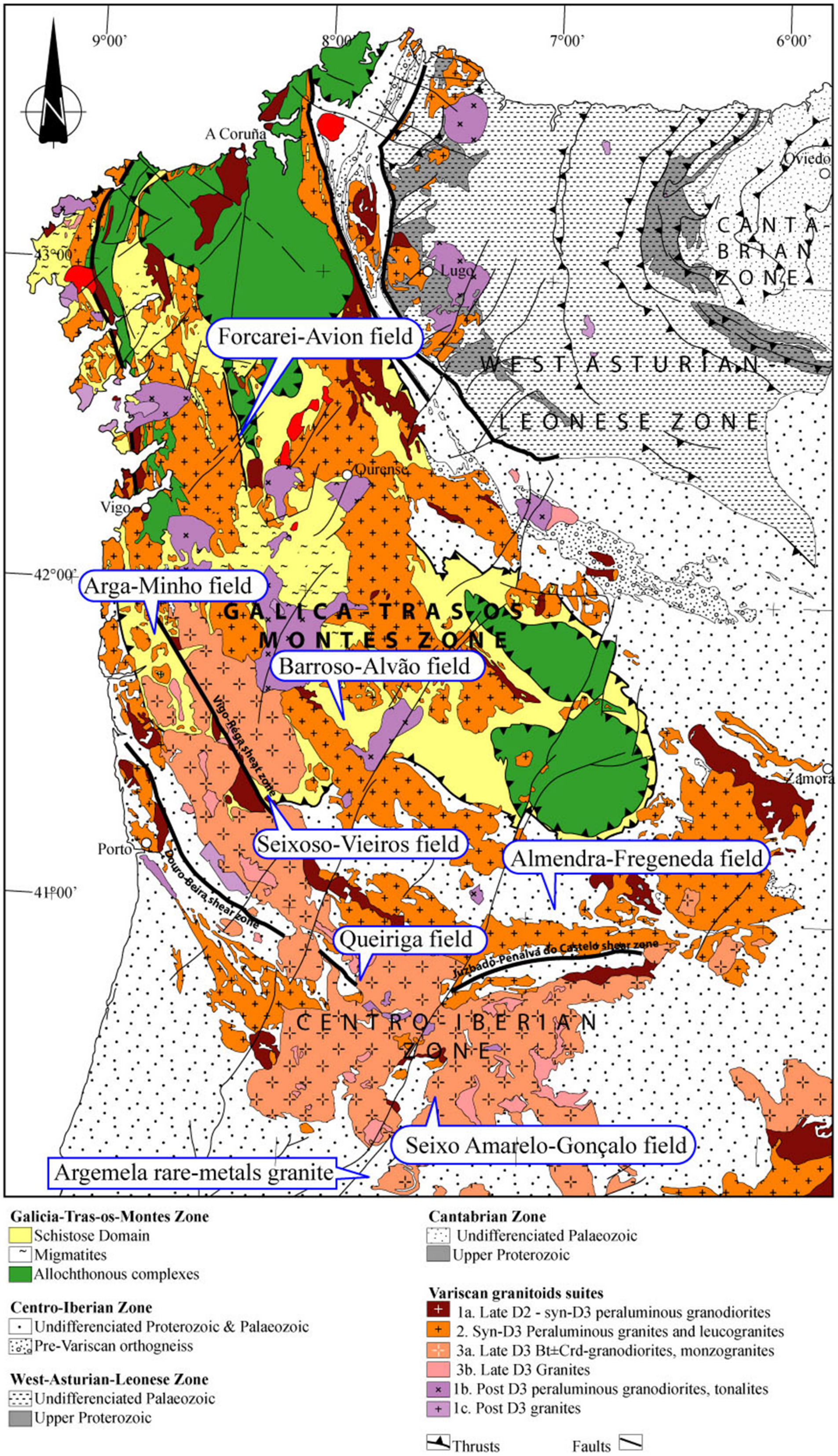


**Fig. 1.** Location of sampled pegmatite fields within the Iberian Massif. Geology modified from Julivert *et al.* (1974); granitoid classification according to Castro *et al.* (2002).

**Table 1.** GPS location of studied samples (geographic co-ordinates, WGS84 datum) and summary of their main characteristics.

| Sample | Longitude | Latitude | Name | Type | location | Tectonic zone |
|---|---|---|---|---|---|---|
| FO1001 | W8°20'23.5″ | N42°30'12.7″ | Alfonsín | Spd-pegmatite | Avíon-Forcarei field | GTOMZ |
| BA1002 | W7°39'43.9″ | N41°36'12.9″ | Adagóï | Spd-pegmatite | Barroso-Alvão field | GTOMZ |
| BA1001 | W7°49'48.4″ | N41°37'20.3″ | Lousas | Pet-pegmatite | Barroso-Alvão field | GTOMZ |
| SA1001 | W8°37'32.9″ | N41°50'00.3″ | Formigoso | Pet-pegmatite | Arga-Minho field | GTOMZ |
| SE1001 | W8°06'46.5″ | N41°21'03.8″ | Outeiro | Pet-granite | Seixoso-Vieiros field | GTOMZ |
| SE1003 | W7°59'34.6″ | N41°19'18.1″ | Vieiros | Pet-Spd-pegmatite | Seixoso-Vieiros field | GTOMZ |
| QU1001 | W7°44'18.7″ | N40°47'11.9″ | Mina do Rebentão | Pet-pegmatite | Queiriga field | CIZ |
| GO1001B | W7°20'01.6″ | N40°26'11.5″ | Gonçalo | Lep-pegmatite | Seixo Amarelo-Gonçalo field | CIZ |
| AR1001A | W7°36'10.0″ | N40°09'21.8″ | Argemela | Rare-element granite | | CIZ |

## Local setting of the study areas

Sampled ore bodies are emplaced within two different geotectonic zones and exhibit different relationships with respect to main deformation phases and emplacement conditions (ductile *versus* fragile). This section briefly describes the local setting of each pegmatite field. All pegmatites studied belong to the LCT family, following the scheme of Černý and Ercit (2005). Figure 1 shows the location of the pegmatite fields studied and of the Argemela rare-element granite. Details of sampled ore bodies including locations are presented in Table 1.

### Pegmatites from the Galicia-Trás-os-Montes Zone-Schistose Domain (GTOMZ-SD)

#### The Forcarei-Avíon pegmatite field

The pegmatite fields of the Galicia-Trás-os-Montes area are emplaced within the Schistose Domain of the GTOMZ. This domain is a large, trapezoidal area bounded to the west and east by peraluminous syn-D3 granite batholiths. Autochthons and the bases of para-autochthon units were strongly affected by D3 metamorphism that caused partial melting, and migmatites are therefore observed on the edges of D3 synforms in the northwestern, southwestern and southeastern parts of the GTOMZ-SD. A large-scale, left-lateral, N170°E-trending shear zone, the Serra do Suido shear zone (Fig. 1), is developed at the contact between the two-mica Cerdedo granite and the para-autochtonous metasedimentary rocks west of the GTOMZ-SD (Gloaguen *et al.*, 2014). At least three pegmatite fields are currently recognized within the GTOMZ-SD: the Forcarei-Avíon field, the Lalín field and the Boborás field (Fuertes-Fuente, 1996; Fuertes-Fuente and Martin-Izard, 1998). The most important one extends within the Forcarei-Avíon region and contains numerous rare-element-bearing pegmatites. One petalite-subtype pegmatite from this field, which lies at the end of Devesa de Abaixo on the road to Alfonsín, was sampled for this work (Tab. 1).

#### The Barroso-Alvão pegmatite field

The Barroso-Alvão pegmatite field is also part of the GTOMZ, located close to the thrust that represents the southern boundary with the Central Iberian Zone and bounded to the east by the Vila Real fault (one of the major NNE-SSW families of faults that affect the region). This pegmatite field has been extensively studied by several authors (Charoy *et al.*, 1992; Lima, 2000; Martins and Lima, 2011; Martins *et al.*, 2011; Silva *et al.*, 2018). Another remarkable feature is the proximity of the pegmatite field to the Vila Real fault, one of the major NNE-SSW families of faults that affect the region. Pegmatites intruded metasedimentary rocks, which exhibit N120°E-trending S2 subhorizontal cleavage locally overprinted by the D3 event. Deformation event D3 produces a locally strong, penetrative S3 crenulation cleavage that is preferentially associated with ductile shear zones (Noronha *et al.*, 1981). This event occurred during the final Variscan ductile upright folding event that resulted in the 120°-trending folds, which overprinted earlier deformation events.

For our geochronological study, we selected two pegmatite bodies classified as complex type, petalite and spodumene subtype: the Lousas petalite-subtype pegmatite and the Adagói spodumene-subtype pegmatite (Tab. 1).

#### The Arga-Minho pegmatite field

The Arga-Minho pegmatite field extends north to Ponte de Lima, east to the Vila Verde thrust and west to the Vigo-Régua shear zone. It comprises mainly Silurian metasedimentary formations of the para-autochton unit, mostly andalusite-bearing micaschists. It is intruded by the Serra de Arga peraluminous granite, which is considered to represent a syn-D3 event (Gomes and Nunes, 1990). Previous studies linked the development of the Arga-Minho pegmatite field with emplacement of the Serra de Arga peraluminous granite, mainly because of the presence of Li, F and Nb anomalies in the southwestern and southeastern parts of the pluton (Gomes and Nunes, 1990; Nunes and Gomes, 1994; Gomes, 1995). In this study, we collected samples from the Formigoso petalite-subtype pegmatite described by Gomes and Nunes (1990).

#### The Seixoso-Vieiros field

The Seixoso-Vieiros field contains numerous granitic pegmatite-aplite bodies (Seixoso and Vieiros pegmatites). The area is bordered to the northwest by the Variscan Celorico de Basto granitic massif, and to the southeast by the syntectonic Felgueiras granodiorite. In the Seixoso area, a specialized heterogeneous granitic intrusion also outcrops as two main apices in the Seixoso and Outeiro granite cupolas (Lima *et al.*, 2009). These apices have a biotite-bearing facies at depth and two-mica or muscovite ± tourmaline facies on their roofs (Helal *et al.*, 1993).

During this study, we dated the Outeiro Mine specialized granite and the Vieiros Mine albite-type pegmatite (Tab. 1). The Outeiro granite contains granitic facies, heterogeneously distributed layered facies and pegmatitic segregations. Lithium-bearing minerals, petalite and spodumene have been observed in the pegmatitic segregations (Lima *et al.*, 2009). The mineral assemblage typically matches that of albite-type pegmatites (Černý and Ercit, 2005).

### Pegmatites and rare element granite from the Central Iberian Zone (CIZ)

#### The Queiriga pegmatite field: Mina do Rebentão

The Queiriga pegmatite field, like other pegmatite occurrences of this area (*e.g.*, Penalva do Castelo field: Venturinha beryl-pegmatite, Correia Neves, 1964), is located along the border of the large, left-lateral, N130°E-trending Douro-Beira crustal-scale shear zone (Valle Aguado *et al.*, 2005). We sampled the Mina do Rebentão pegmatite (Tab. 1), emplaced within the porphyric Cota-Viseu late-D3 granite, which has been dated at 306 ± 4 Ma (Valle Aguado *et al.*, 2005) by conventional U-Pb dating of zircon and monazite. The Mina do Rebentão pegmatite was likely emplaced under brittle conditions and contains numerous metric granodiorite enclaves with sharp contacts in its upper part.

#### The Seixo Amarelo-Gonçalo pegmatite field

The Seixo Amarelo-Gonçalo pegmatite field (Guarda-Belmonte area, east central Portugal) is located in the CIZ of the Iberian Massif. It was described extensively by Ramos (1998, 2007) and Neiva and Ramos (2010). In this area, numerous aplite-pegmatite sills intrude the porphyritic biotite > muscovite Guarda granite, which is classified as a late-D3 granite (Ferreira *et al.*, 1987; Neiva *et al.*, 2009; Neiva and Ramos, 2010). The last authors distinguished four two-mica granites in the Guarda-Belmonte area: the Guarda granite G1 is dated at 304 ± 4 Ma (SHRIMP U-Th-Pb on monazite; Neiva *et al.*, 2009; Neiva and Ramos, 2010), the fine- to medium-grained porphyritic biotite > muscovite granite (G2) at 300 ± 1 Ma, the coarse grained porphyritic biotite > muscovite granite (G3) at 301 ± 2 Ma and the medium- to coarse-grained muscovite > biotite granite (G4) at 299 ± 3 Ma (ID-TIMS on zircon and monazite; Neiva and Ramos, 2010; Neiva *et al.*, 2011). According to these authors, aplite-pegmatite sills would belong to a magmatic serie related to G2 and G3. However, the marked variation in geochemical results from their analyses of aplite-pegmatite (especially for Li, Sn, Rb and Ta) raises some doubts about their interpretations.

Because of the sharp contacts between pegmatite sills and the host granite and the scarcity of deformation patterns, emplacement likely occurred under brittle conditions at an upper level during compressive motion (reverse faulting).

For this study, we chose to date the Gonçalo lepidolite-subtype pegmatite, which outcrops about 1.5 km northeast of the town of Gonçalo.

#### The Argemela rare-element granite

The Argemela rare-element granite is a small elliptical body in central Portugal and showing extreme enrichment in incompatible elements (Charoy and Noronha, 1996). This rare-element granite intruded into Upper Precambrian, low-grade schists and metagreywackes, along a N165°E-trending right-lateral vertical shear zone that also slightly deformed its eastern border (Michaud *et al.*, 2020). Its very fine texture and the presence of rounded quartz suggest very fast cooling consistent with upper-level emplacement (Charoy and Noronha, 1991). Several generations of steeply dipping quartz veins with montebrasite, wolframite, and scarce cassiterite, crosscut the granitic body (Michaud *et al.*, 2020). Genetic relationships between rare-element granites, intragranitic veins and with the neighbouring amblygonite-cassiterite-bearing quartz-vein swarm mined for tin in the past have been investigated recently (Michaud and Pichavant, 2020; Michaud *et al.*, 2020). These results highlighted the effect of the continuity of magmatic to hydrothermal processes for the origin of the different mineralizations. Following Michaud *et al.* (2020) interpretation, CGM disseminated in the granite crystallized at the magmatic stage whereas scarce CGM also occur in late intragranitic veins.

## Analytical methods

Pegmatites are usually very heterogeneous bodies and representative samples must be selected with care. However, the studied pegmatites are not all zoned, and in most cases, observed zoning mostly concerned crystal sizes rather than significant mineralogical changes. Macroscopic observations during fieldwork allowed us to recognize blocks with abundant oxides (mainly cassiterite and CGM). All study samples were selected from the oxide-rich zones of the pegmatites.

CGM grains were handpicked under a binocular microscope after densimetric separation. To distinguish these grains from cassiterite grains, they were tested (tinning test) by placing them inside a zinc cup and covering them with hydrochloric acid (Parfenoff *et al.*, 1970). CGM grains do not change during the tinning test, whereas cassiterite grains become covered by a thin layer of zinc, giving them a silvery aspect. Grains were mounted in epoxy blocks and subsequently ground and polished to obtain a uniform surface. Backscattered electron (BSE) images were acquired at the BRGM (Orléans, France) and at the Géosystèmes research unit (UMR 8217) in Lille, France.

In-situ U-Pb isotopic data on single grains of CGM were acquired by laser ablation (NewWave UP213)-single collector magnetic sectorfield-inductively coupled plasma-mass spectrometry (LA-SF-ICP-MS, ThermoFinnigan Element2) at the Geological Survey of Denmark and Greenland in Copenhagen. At the time of analyses, no CGM international standard was available, possibly because of the marked compositional variability of these minerals. However, Melcher *et al.* (2008) did not observe matrix-dependent U-Pb fractionation during CGM dating, and chose to use a zircon standard to correct the matrix effect on fractionation and an extensive study shows that matrix effect are mainly observed for tantalite crystals

(Legros *et al.*, 2019). Frei and Gerdes (2009) and Gerdes and Zeh (2006) described in detail the methods used for the analysis and processing of data from zircon U-Pb dating, and we applied these methods to process the U-Pb data acquired on CGM. All age data presented herein were obtained by single spot analyses with a spot diameter of 45 μm and a crater depth of approximately 15 to 20 μm. The Plešovice (Sláma *et al.*, 2008) and M127 (Nasdala *et al.*, 2008) zircon standards were repeatedly analyzed during data acquisition for quality control, and the results were consistently within 2σ of the published ID-TIMS ages. Isoplot/Ex 3.0 (Ludwig, 2003) was used to calculate concordia ages and to plot concordia diagrams.

Electron-microprobe analyses of CGM grains from the studied pegmatites were carried out in the wavelength-dispersion mode on a CAMECA SXFive instrument (University of Orléans-CNRS-BRGM common laboratory). Analyses were performed with an accelerating voltage of 20 kV. The beam diameter was 2 μm. All samples were examined for chemical heterogeneities by back-scattered electron imaging. The number of points and grains analysed per sample depended upon the degree and scale of chemical zoning. Matrix corrections were made with the phi-rho-Z computing program PAP (Pouchou and Pichoir, 1984). Major and trace element were analysed with a beam current of 200 nA. Ti Kβ, Nb Lα, Zr Lα were measured on PET, Hf Mβ on TAP, Fe Kα, Mn Kα on LiF, Sn Lα, U Mβ, Sc Kα on LPET and W Lα, Ta Lα on LLiF. Counting times on peak and background were 10 s for Ti, Fe, Mn; 40 s for Sn, Nb, W, Zr, U, Hf and 60 s for Ta. Standards of calibration were natural mineral (cassiterite for Sn), synthetic oxides ($MnTiO_3$ for Ti, $Fe_2O_3$ for Fe, $LiNbO_3$ for Nb, $UO_2$ for U, $ScPO_4$ for Sc), and pure elements (Ta, Zr, Hf and W). According analytical setting and element concentration within CGM for each analysis, detection limits were in between 267 and 305 ppm for Sn, 49 and 56 ppm for Sc, 198 and 223 ppm for Ti, 325 and 366 ppm for Fe, 125 and 142 ppm for Mn, 321 and 688 ppm for Nb, 266 and 347 ppm for Zr, 254 ppm for Hf, 382 ppm for W, 245 and 443 ppm for Ta, 306 and 348 ppm for U.

## Geochronological results

Isotopic data are presented in Tables S1 and S2 (e-components) and plotted in Figures 2–10. All age and error calculations were performed at the 2σ level. Errors ellipses were plotted at the 2σ level in all diagrams.

For most samples, few analyses produced strongly fractionated transient signals unusable for age calculations. Such signals are interpreted to be due to the presence of micro-inclusions (quartz, albite, micas or U-rich minerals such as uraninite) that were not detected during the examination of SEM pictures prior to the analyses.

### Pegmatites from the the Galicia-Trás-os-Montes Zone-Schistose Domain (GTOMZ-SD)

Forcarei-Avíon pegmatite field: Alfonsín petalite-subtype pegmatite (FO1001): BSE images showed various types of grains: homogenous, with patchy zoning, or with dark cores surrounded by lighter rims (Fig. 2). Crystal sizes range from 300 to 500 μm. In total, twenty-three analyses were performed on six columbite-(Mn) and columbite-(Fe) grains. Results from three analyses were rejected because of irregular, strongly fractionated, unusable transient signals. The remaining analyses gave concordant or nearly concordant results and yielded an age of 312 ± 4 Ma, with an MSWD value of 0.73 (n = 20) (Fig. 2). Uranium and lead concentrations range from 291 to 1248 ppm and from 15 to 61 ppm respectively (see Tab. S1, e-components).

Barroso-Alvão pegmatite field: Three grain populations from the Lousas petalite-subtype pegmatite (BA1001) were observed in BSE images (Fig. 3). The first is represented by slightly homogenous grains, the second, by grains mainly showing patchy zoning, and the third, by grains with oscillatory zoning. Uranium-rich mineral inclusions are common and are particularly abundant in grain 22 (Fig.). Crystal sizes range from 300 to 800 μm. In total, twenty-two analyses were performed on seventeen columbite-(Mn) and columbite-(Fe) grains (Fig. 3). Results from two analyses were rejected because of irregular and highly fractionated transient signals, and from three others because of significant radiogenic Pb loss. The remaining analyses yielded an age of 311 ± 4 Ma (Fig. 3), with an MSWD of 0.19 (n = 16). Uranium and lead concentrations range from 104 to 3241 ppm and from 5 to 156 ppm respectively (see Tab. S1, e-components).

Two populations of grains from the Adagói spodumene-subtype pegmatite (BA1002) were observed in BSE images (Fig. 4). The first is composed of heterogeneous grains with cores showing patchy zoning and rims with oscillatory zoning. The second comprises grains having homogenous cores and rims with oscillatory zoning. Uranium rich mineral inclusions are common. Crystal sizes vary from 100 μm to 1 mm.

In total, twenty-one analyses were carried out on seventeen columbite-(Fe) and tantalite-(Fe) grains (Fig. 4). Three analyses were rejected because of irregular, highly fractionated transient signals, and four more because of significant radiogenic Pb loss. The remaining analyses yielded an age of 307 ± 5 Ma (Fig. 4), with an MSWD of 0.30 (n = 14). Uranium and lead concentrations range from 69 to 1390 ppm and from 3 to 69 ppm respectively (see Tab. S1, e-components).

Arga-Minho pegmatite field: Formigoso petalite-subtype pegmatite (SA1001): In total, eight analyses were carried out on one columbite-(Fe) grain and one tantalite-(Fe) grain. BSE imaging of the tantalite-(Fe) grain shows oscillatory zoning; the columbite-(Fe) grain shows sector zoning (Fig. 5). Crystal sizes are about 150 μm (Fig. 5). Results from one analysis were rejected because of an irregular, highly fractionated transient signal, and from another because of small, but significant radiogenic Pb loss. One analysis provided concordant results, whereas five others provided results that plot along a mixing line with common Pb values (Fig. 5) that yield an age of 304 ± 9 Ma, with an MSWD of 1.0 (n = 6). Uranium and lead concentrations range from 240 to 1386 ppm and from 12 to 65 ppm respectively (see Tab. S1, e-components).

Seixoso-Vieiros pegmatite field: Two grain populations from the Outeiro Mine granite (SE1001) were observed in BSE images (Fig. 6). The first is composed of poikilitic grains with patchy and oscillatory zoning and numerous inclusions of U-rich minerals, and the second, of grains with mainly oscillatory

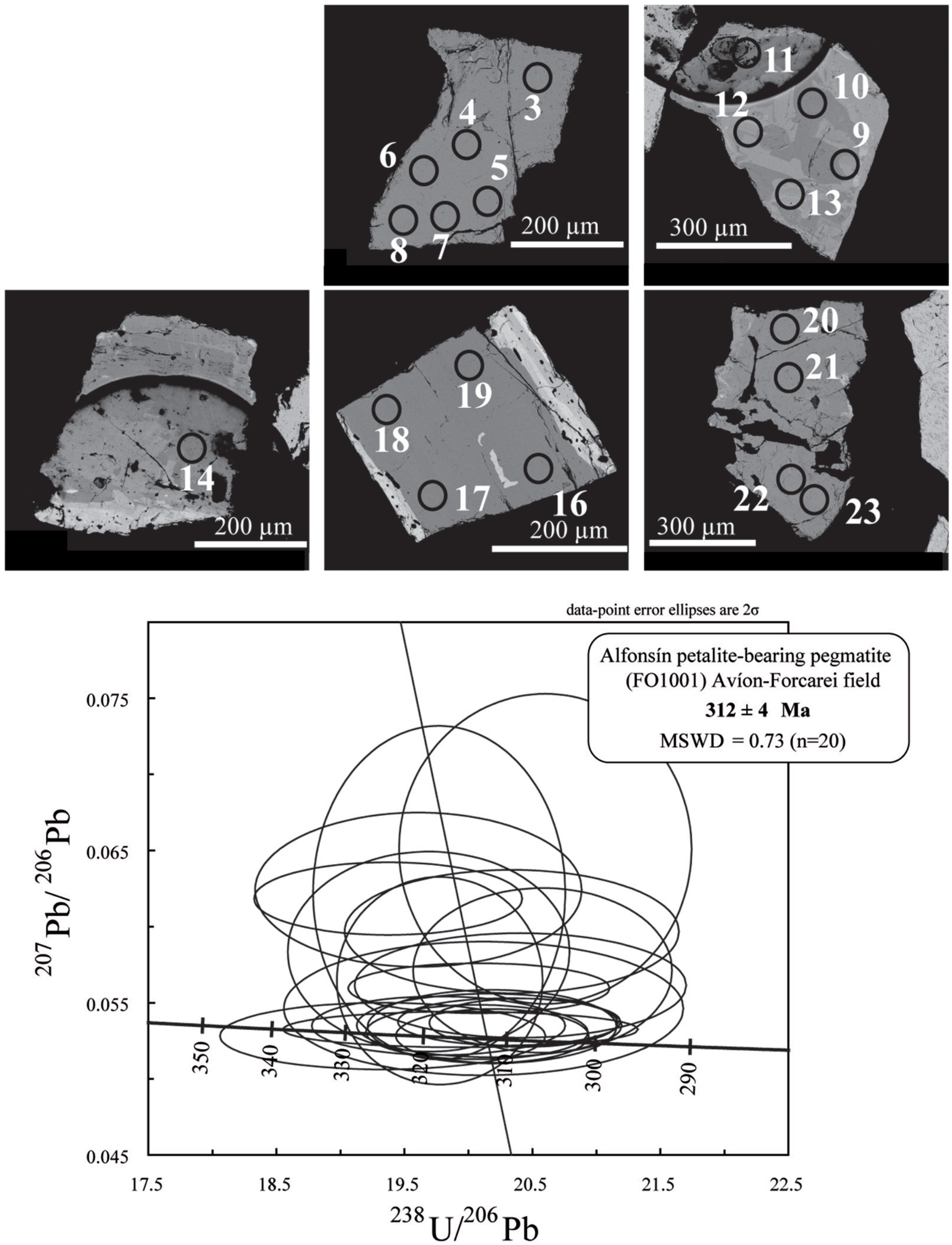


**Fig. 2.** Inverse concordia diagrams reporting CGM U-Pb data for the sample from the Alfonsín petalite-bearing pegmatite (FO1001A; Forcarei-Avíon field). BSE images of the dated grains.

zoning and no inclusions. Crystal sizes range from 100 to 500 µm. In total, fourteen analyses were performed on five columbite-(Fe) grains (Fig. 6). Two analyses (2 and 12) of grain cores (Fig. 6) yielded low U concentrations (between 225 and 266 ppm) and an age of 316 ± 9 Ma. A second analysis population yielded an age of 301 ± 5 Ma, with an MSWD of 0.43 (n = 9). Uranium and lead concentrations range from 225 to 1129 ppm and from 8 to 54 ppm respectively (see Tab. S1, e-components).

Tantalite-(Fe) grains from the Vieiros pegmatite (SE1003) show patchy zoning and crystal sizes ranging from 700 to 900 µm (Fig. 7). In total, nineteen analyses were performed on two tantalite-(Fe) grains (Fig. 7). Results from seven analyses were rejected because of irregular, highly fractionated transient signals. Results from the remaining analyses are concordant or plot along a mixing line with common Pb values and yield an age of 301 ± 4 Ma (Fig. 7), with an MSWD of 0.44 (n = 12). Uranium and lead concentrations range from 112 to 218 ppm

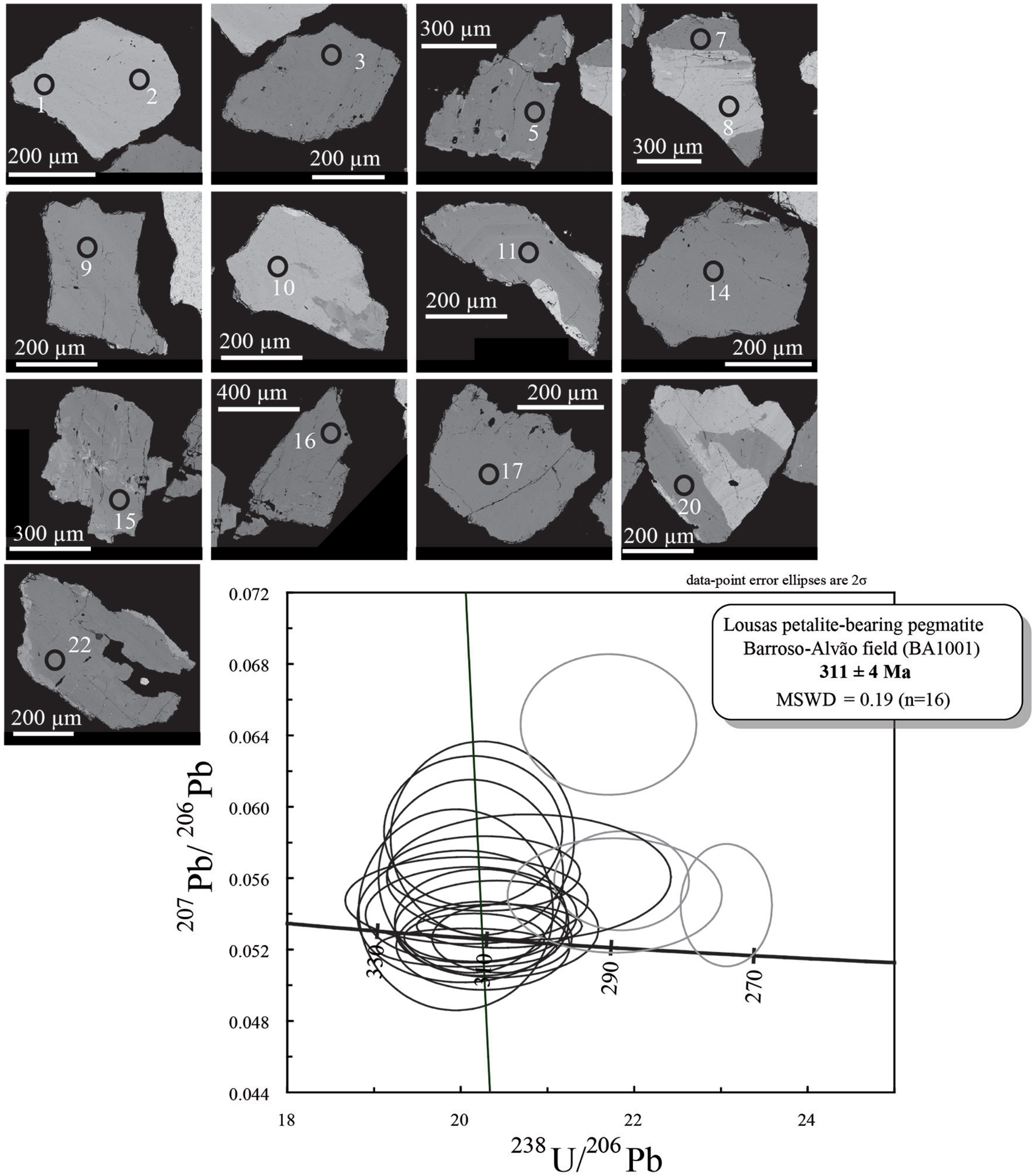


**Fig. 3.** Inverse concordia diagrams reporting CGM U-Pb data for the sample from the Lousas petalite-bearing pegmatite (BA1001; Barroso-Alvão field). BSE images of the dated grains.

and from 5 to 11 ppm respectively (see Tab. S1, e-components).

## Pegmatites from the Central Iberian Zone (CIZ)

Mina do Rebentão petalite-subtype pegmatite (QU1001, Queiriga field): In total, nineteen analyses were performed on seventeen columbite-(Mn) and tantalite-(Mn) grains (Fig. 8). BSE images showed very heterogeneously textured grains. Homogenous grains or grains with slight rectilinear zonations seem to form a first population. Very complex grains with patchy and/or oscillatory zoning form a second heterogeneous population (Fig. 8). Crystal sizes range from 200 to 500 µm (Fig. 8). Results from three analyses were rejected because of irregular, highly fractionated transient signals, and from three more because of significant radiogenic Pb loss.

Results from the remaining analyses are concordant or plot along a mixing line with common Pb values and yield an age of 300 ± 4 Ma, with an MSWD of 0.47 (n = 16) (Fig. 8). Uranium concentrations range from 510 to 4453 ppm. Consequently, Pb concentrations range from 23 to 210 ppm (see Tab. S2, e-components).

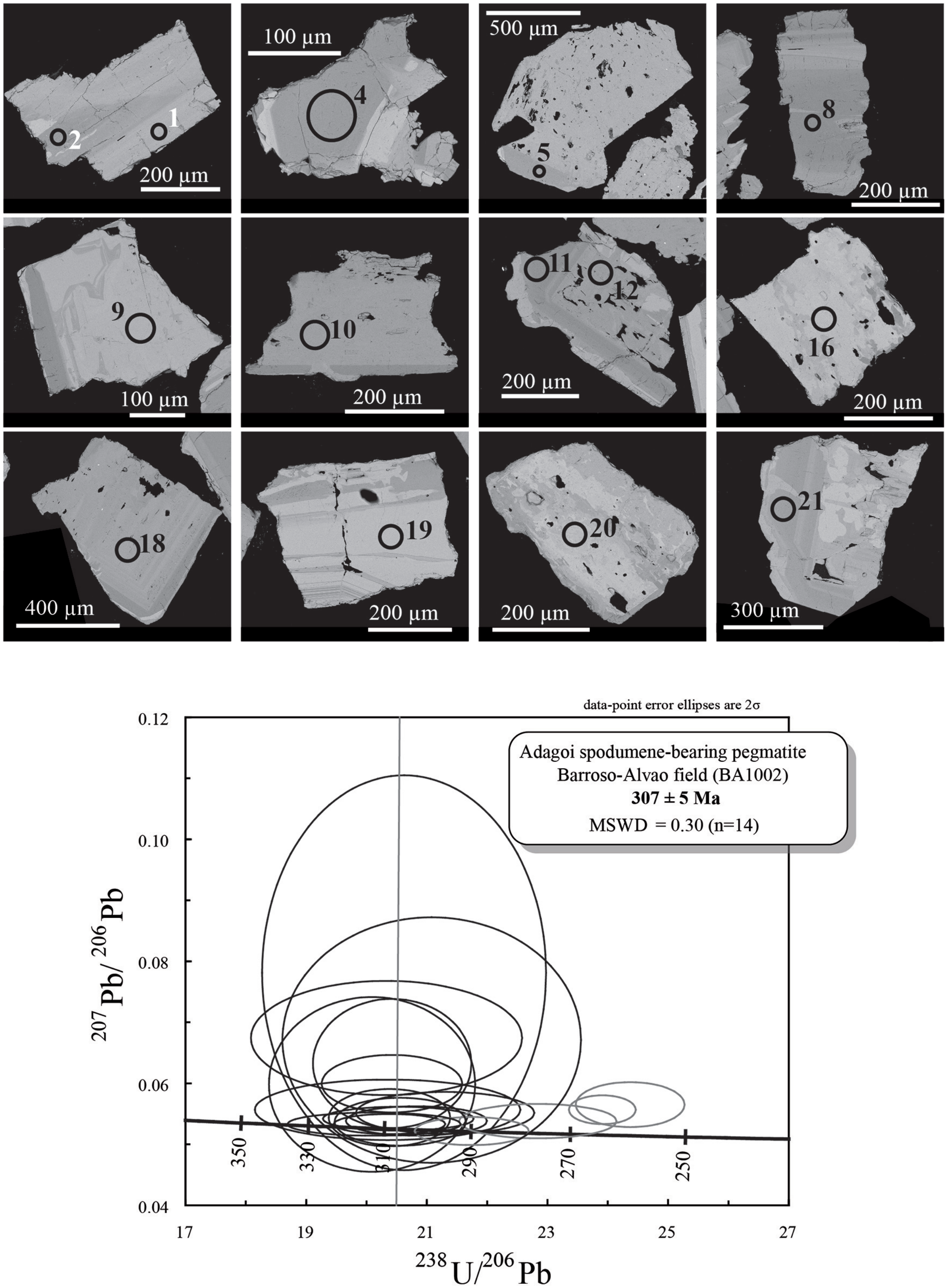


**Fig. 4.** Inverse concordia diagrams reporting CGM U-Pb data for the sample from the Adagói spodumene-bearing pegmatite (BA1002; Barroso-Alvão field). BSE images of the dated grains.

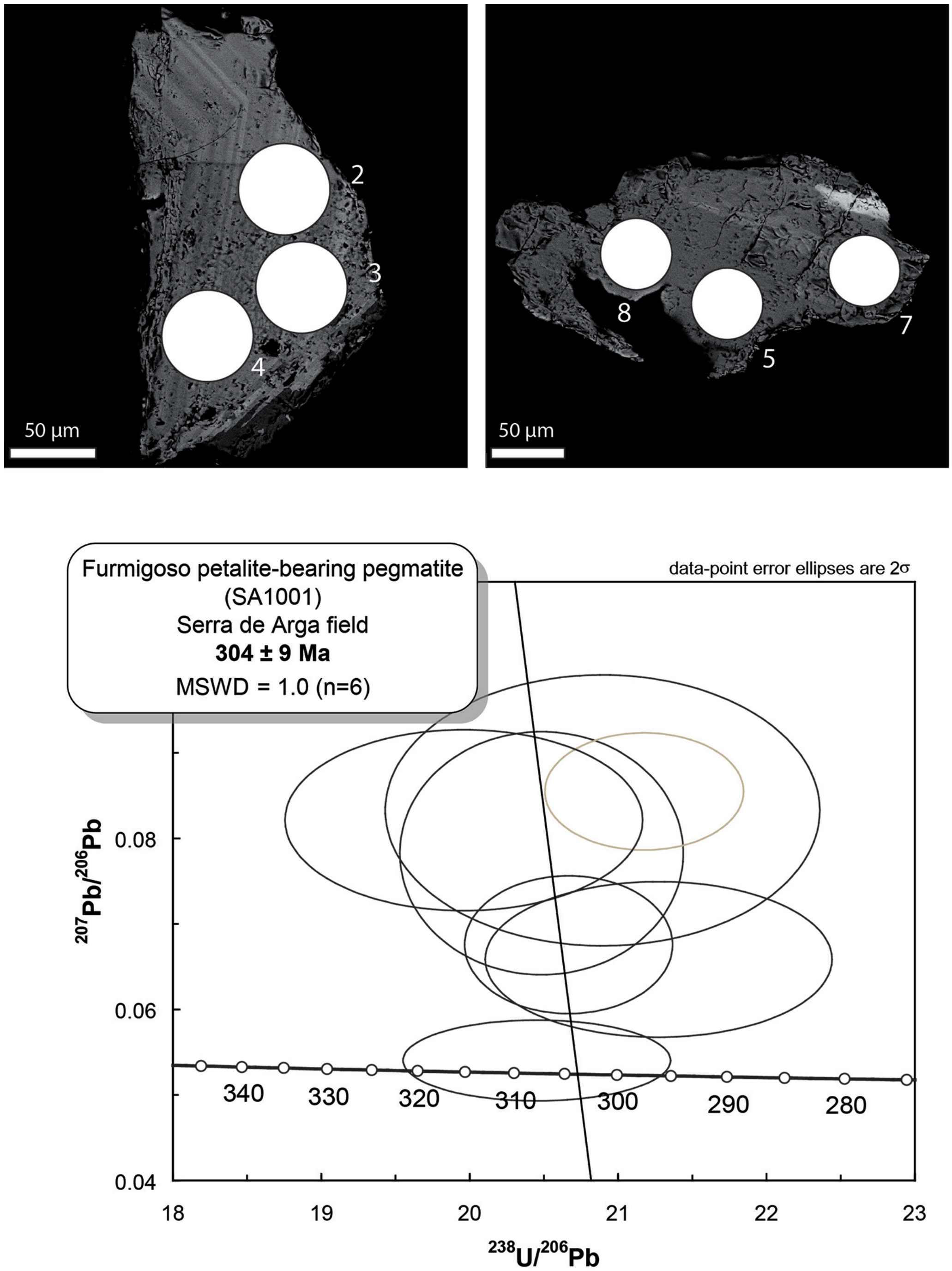


**Fig. 5.** Inverse concordia diagrams reporting CGM U-Pb data for the sample from the Formigoso petalite-bearing pegmatite (SA1001; Arga-Minho field). BSE images of the dated grains.

The Seixo Amarelo-Gonçalo pegmatite field: Two grain populations from Gonçalo lepidolite-subtype pegmatite (GO1001B) were observed in BSE images (Fig. 9). The first is slightly homogeneous, whereas the second is composed of complex grains with patchy and/or oscillatory zoning. Most of the grains analyzed are between 100 and 200 µm in size; one grain measured nearly 700 µm.

In total, twenty-nine analyses were performed on eighteen columbite-(Mn) and tantalite-(Mn) grains, representative of the two textural types. Results from three analyses were rejected because of irregular, highly fractionated transient signals, and from a fourth because of significant radiogenic Pb loss. The remaining analyses yielded an age of 301 ± 3 Ma (Fig. 9), with an MSWD of 0.29 (n = 25). Uranium and lead

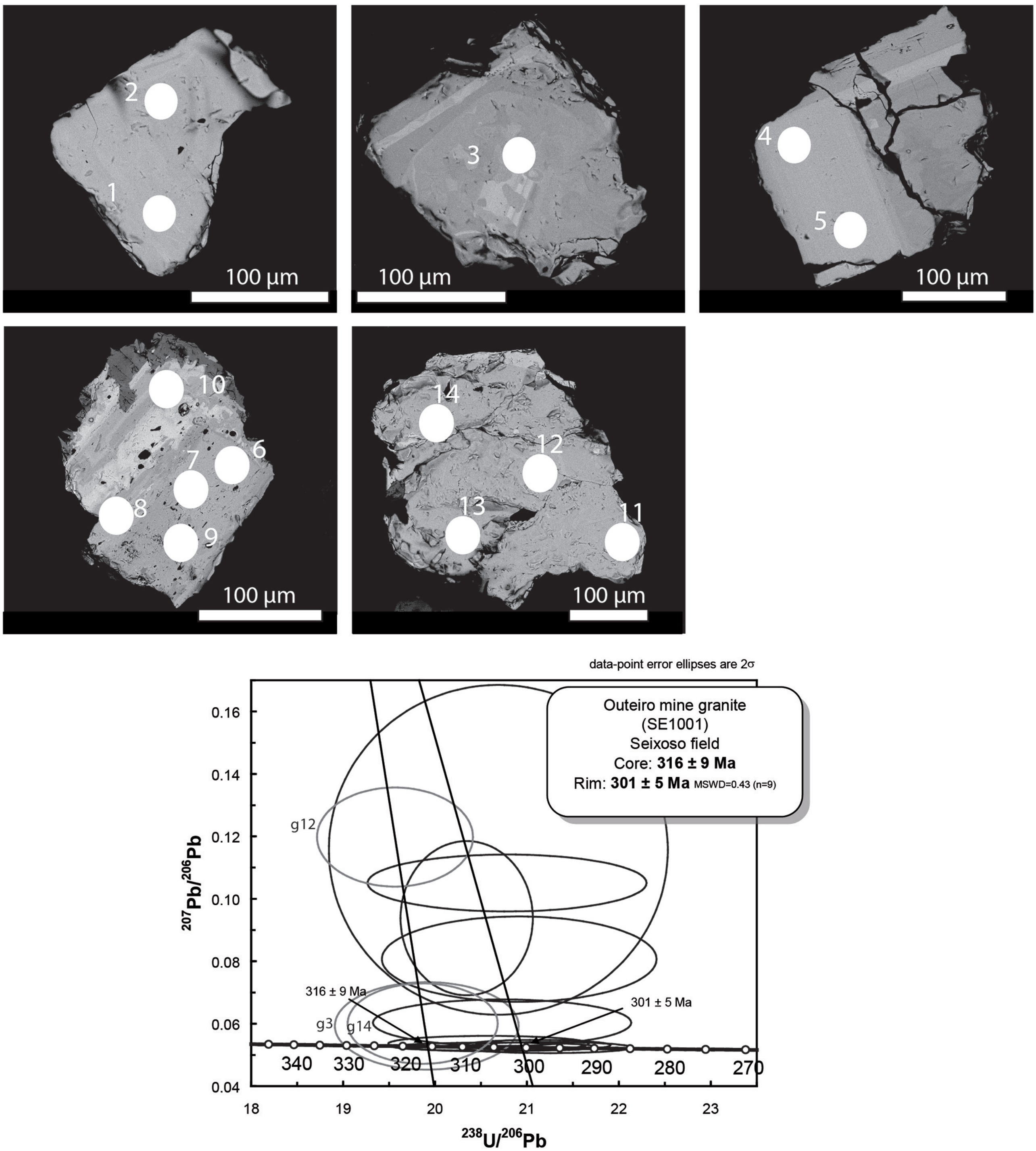


**Fig. 6.** Inverse concordia diagrams reporting CGM U-Pb data for the sample from the Outeiro Mine granite (SE1001; Seixoso-Vieiros field). BSE images of the dated grains.

concentrations range from 66 to 921 ppm and from 3 to 44 ppm respectively (see Tab. S2, e-components).

Argemela granite (AR1001A): BSE images (Fig. 10) showed several types of columbite-(Mn) grains: homogenous grains, grains with a heavier rim and a dark core, and more complex grain populations with patchy zoning. Sizes range from 300 to 700 µm. In total, twenty analyses were performed on eleven columbite-tantalite grains. Results from five analyses were rejected because of irregular, highly fractionated transient signals. Results from most of the remaining analyses are highly concordant, whereas results from three analyses plot along a mixing line with common Pb values and gave an age of 326 ± 3 Ma,

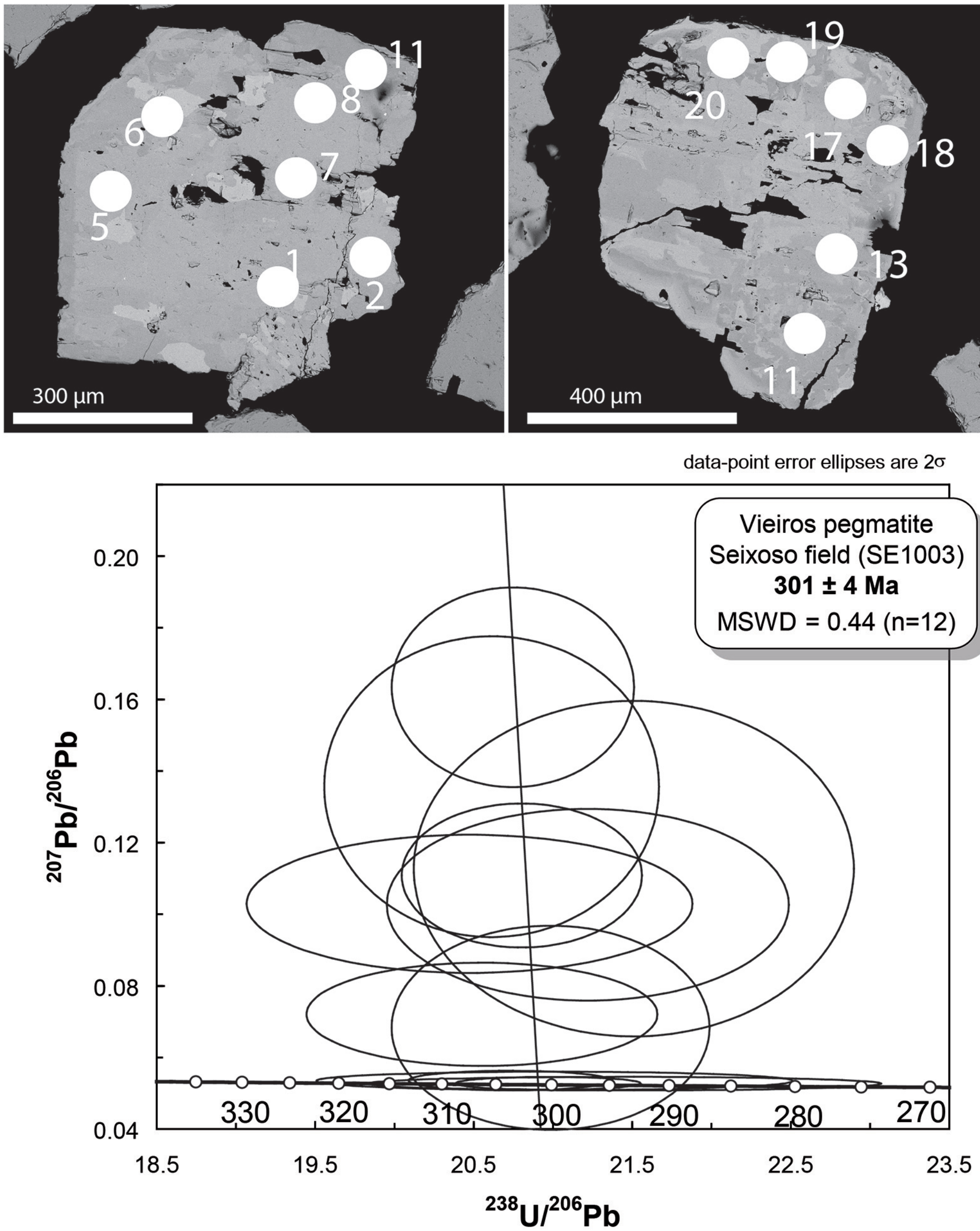


**Fig. 7.** Inverse concordia diagrams reporting CGM U-Pb data of the sample from the Vieiros petalite-bearing pegmatites (SE1003; Seixoso-Vieiros field). BSE images of the dated grains.

with an MSWD of 0.22 (n = 15) (Fig. 10). Uranium and lead concentrations range from 72 to 969 ppm and from 4 to 50 ppm respectively (see Tab. S2, e-components).

## Chemical compositions of dated CGM

Table S3. (e-components) and Figure 11A show that almost all parts of dated CGM grains correspond to columbite-(Mn) (Outeiro granite SE1001, Formigoso petalite-subtype pegmatite SA1001, Adagói spodumene-subtype pegmatite BA1002, Alfonsín petalite-subtype pegmatite FO1001A, Rebentão petalite-subtype pegmatite QU1001) or columbite-(Fe) (FO1001A, AR1001, Lousas petalite-subtype pegmatite BA1001 and Gonçalo lepidolite-subtype pegmatite GO1001B). Only grains from the Vieiros pegmatite (SE1003) and scarce parts of grains correspond to tantalite-(Fe) (Adagói spodumene-subtype pegmatite BA1002) or tantalite-(Mn) (Rebentão petalite-subtype pegmatite QU1001, Argemela rare metal granite AR1001). The composition of some tantalite-(Fe) from the Vieiros petalite-subtype pegmatite (SE1003) located within the boundaries of the tantalite-tapiolite gap are probably the result of a disequilibrium growth, remaining metastable and not exsolved into ferrotapiolite-tantalite pairs (Cerny *et al.*, 1992). Trace

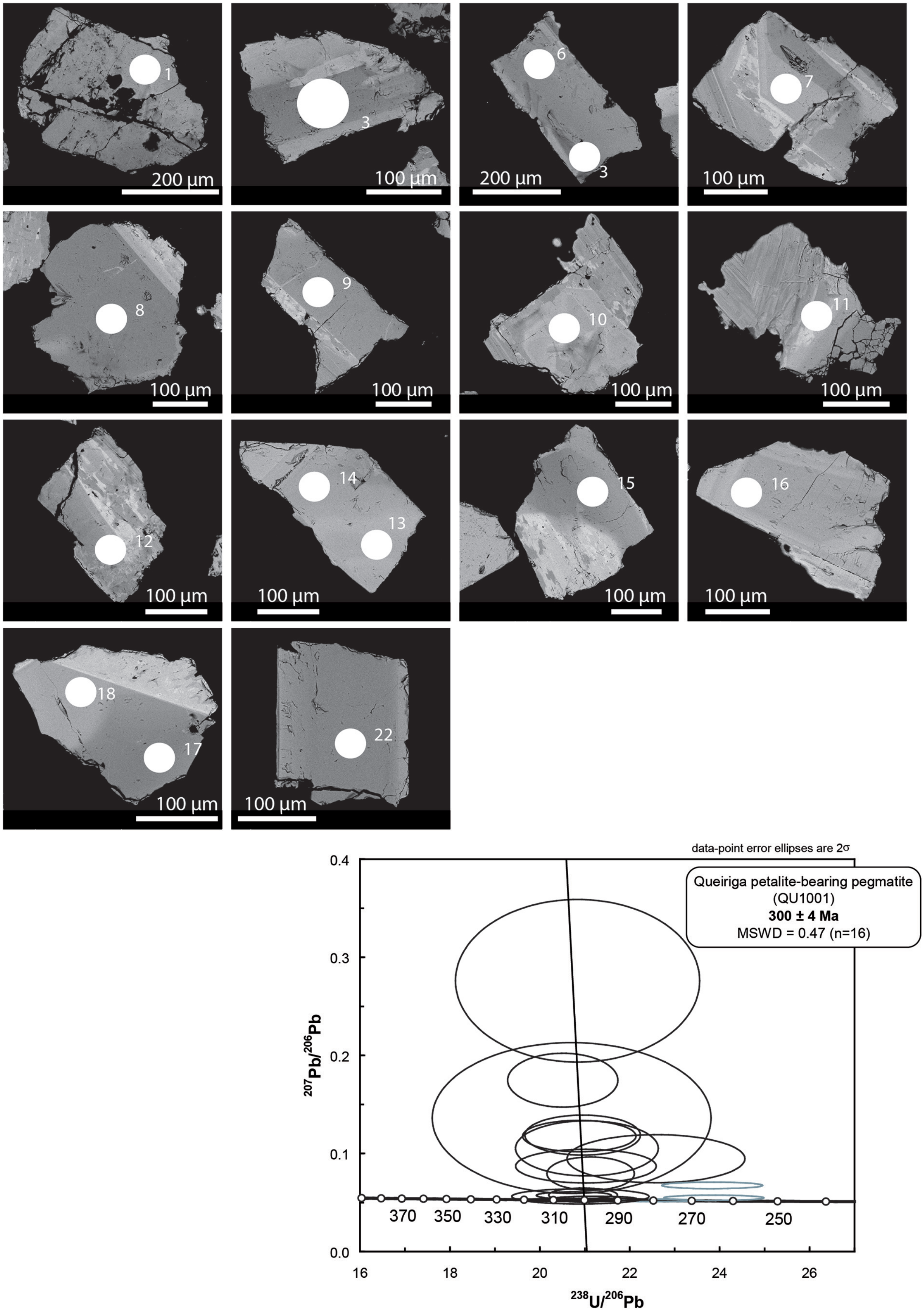


**Fig. 8.** Inverse concordia diagrams reporting CGM U-Pb data for the sample from the Mina do Rebentão pegmatite (QU1001; Queiriga field). BSE images of the dated grains.

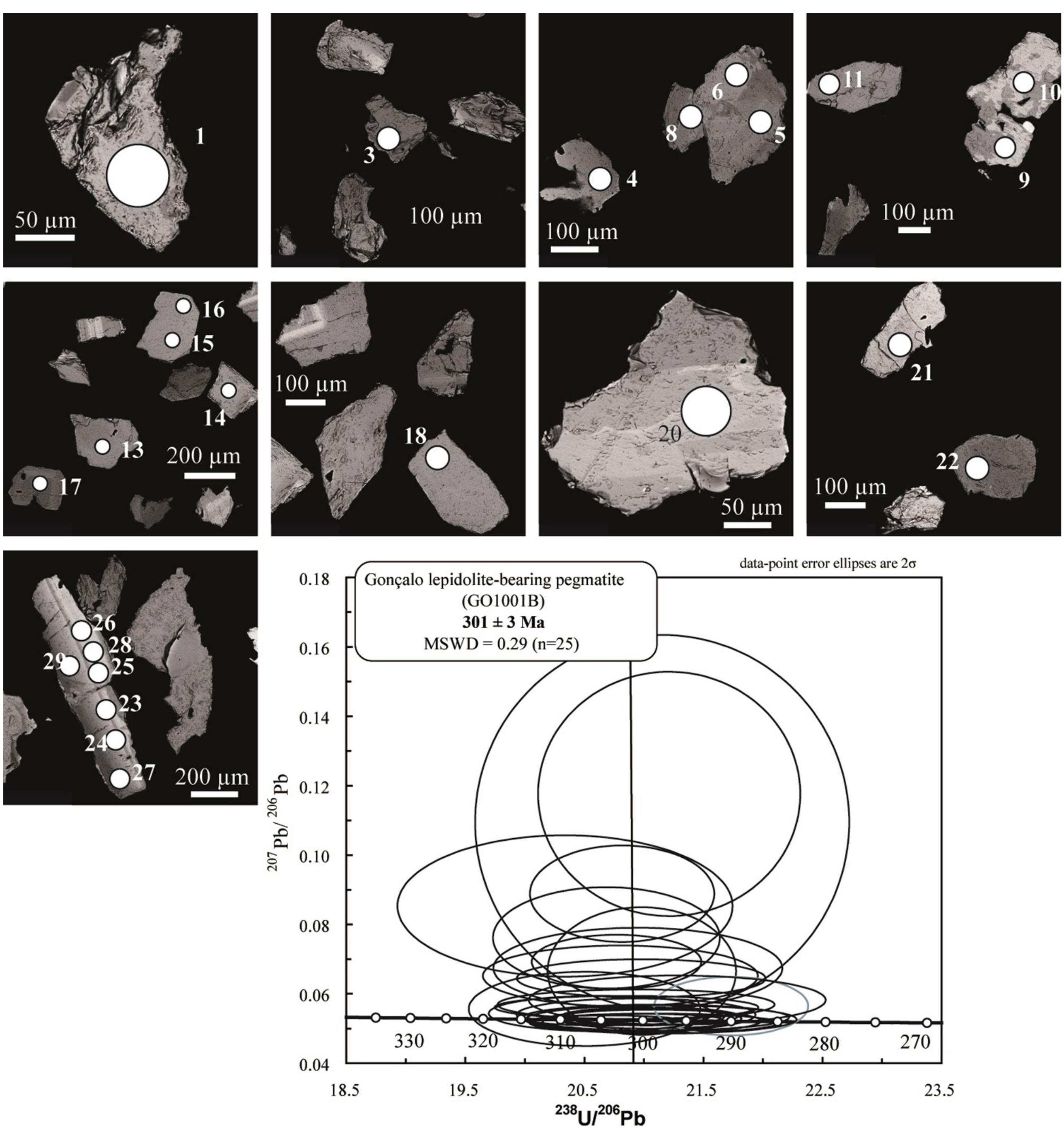


**Fig. 9.** Inverse concordia diagrams reporting CGM U-Pb data for the sample from the Gonçalo lepidolite-bearing pegmatite (Seixo Amarelo-Gonçalo field). BSE images of the dated grains.

element within studied CGM are mainly represented by Sn, Ti, Zr, Sc and U. Elements Hf and W are generally below the detection limits. The major element *versus* trace element binary diagrams (Fig. 11C and zoom on Fig. 11D) underline the anti-correlation between the two groups. CGM grains from SA1001, SE1001, GO1001B and FO1001A follow the 1:1 substitution trend, indicating a $M^{2+} + M^{5+} \rightarrow M^{3+} + M^{4+}$ substitution vector (*e.g.*, Ercit, 1994; Černý *et al.*, 2007). Conversely, CGM grains from others samples follow the 3:2 substitution trend, indicating a $M^{2+} + 2M^{5+} \rightarrow 3M^{4+}$ substitution vector. For all samples, Sn, Ti and Zr are well correlated and their quantities decrease with the Mn/(Mn + Fe) ratio (Fig. 11B) whereas U content is not correlated with Sn (Fig. 11F), thus not correlated with others $M^{4+}$.

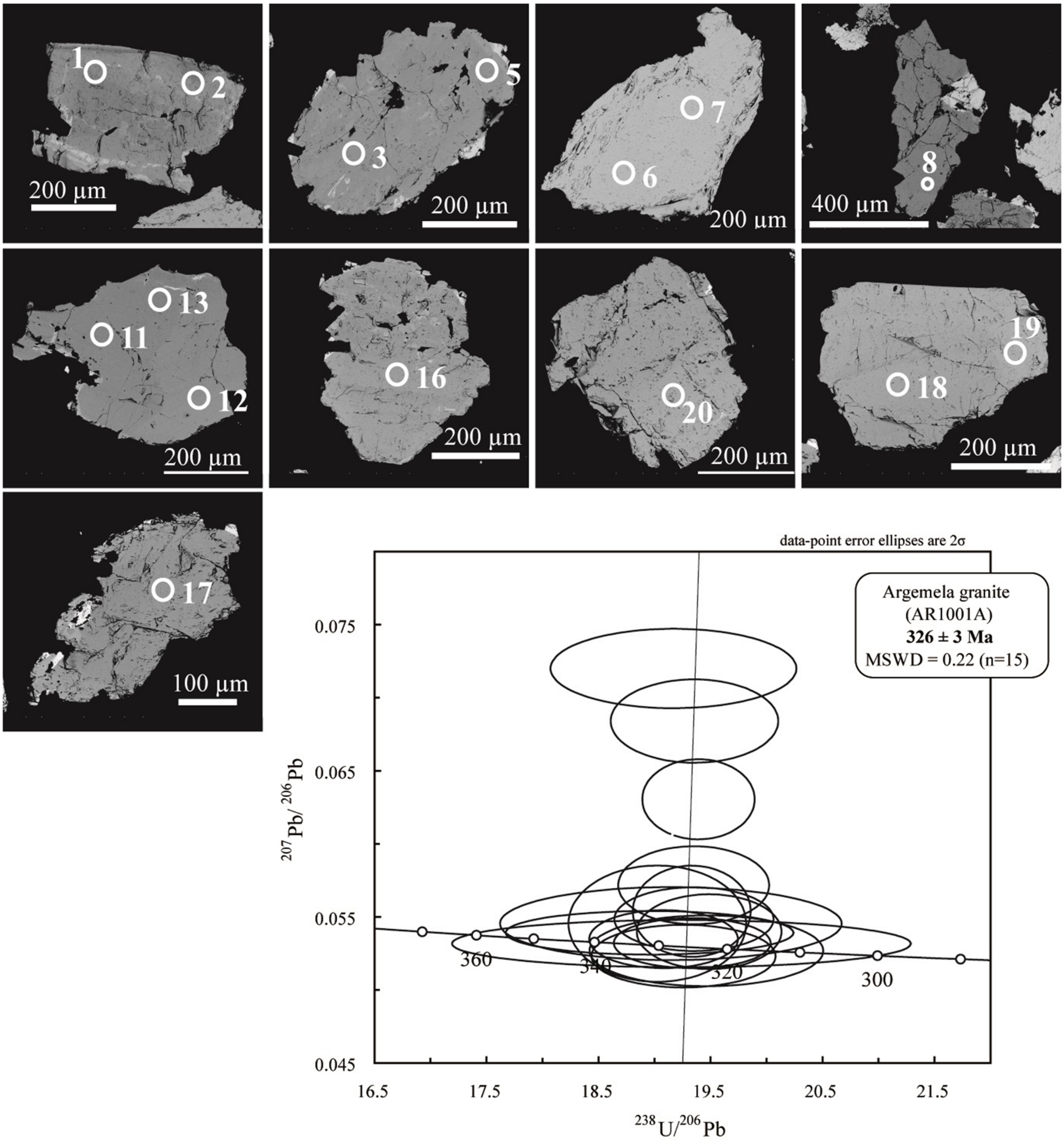


**Fig. 10.** Inverse concordia diagrams reporting CGM U-Pb data for the sample from the Argemela rare-element granite. BSE images of the dated grains.

## Discussion

This paper provides ten new U-Pb ages on CGM from rare-element granites and pegmatites of the Iberian Variscan belt (summarized in Figs. 12 and 13). With the exception of the Outeiro Mine granite (Seixoso-Vieiros field), for which two different ages can be clearly established, all the samples provided homogeneous results (within error). For all the studied samples (except Outeiro Mine), the absence of textural features that could indicate posterior disturbance events suggest that the age obtained for each sample corresponds to the age of magmatic emplacement. In the following section, we first discuss the implications of these results for the chronology of rare-element magmatism and the evolution of the Late Variscan continental crust. Consequences for rare-element enriched peraluminous melts are also debated. To

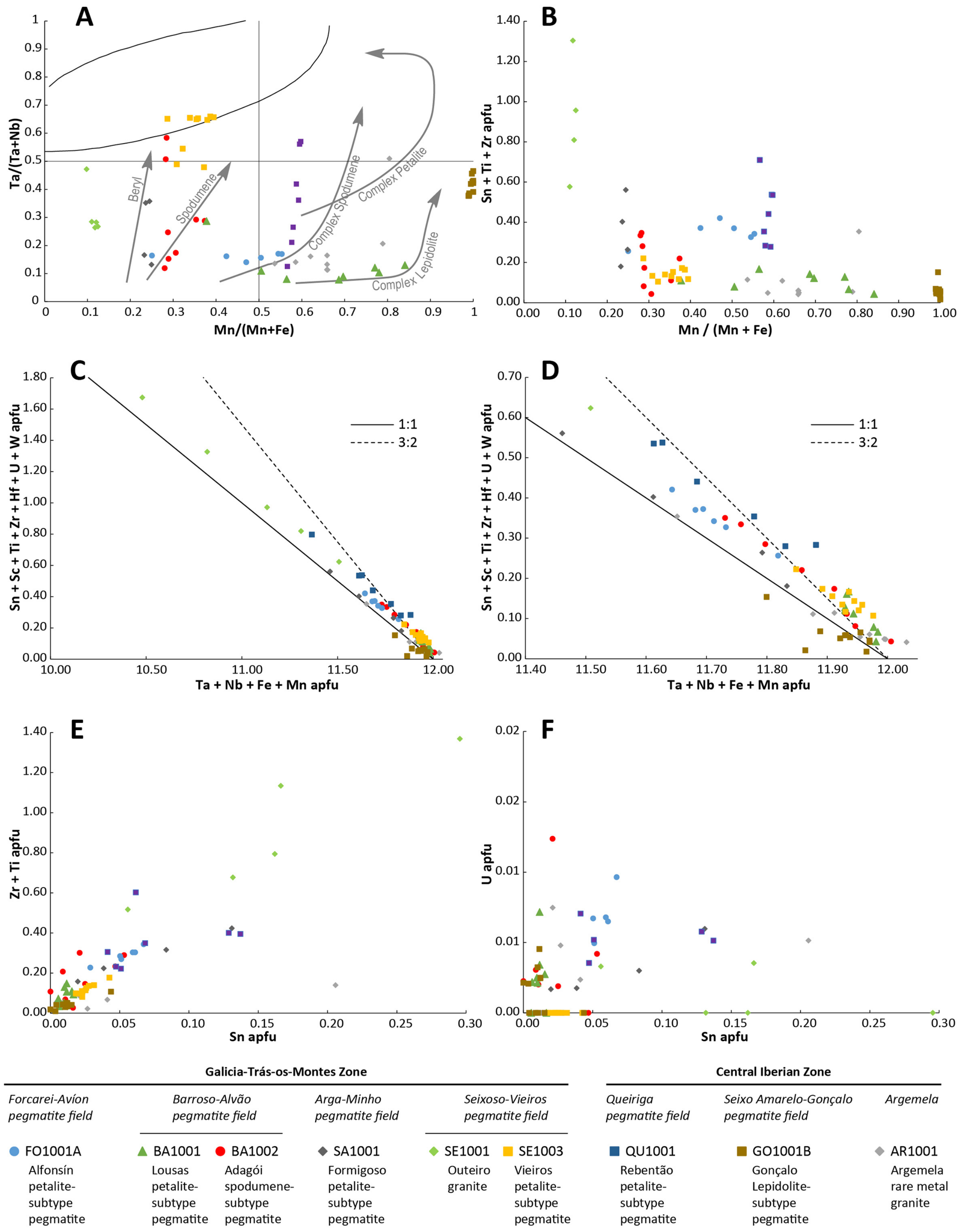


**Fig. 11.** (A) Composition of studied CGM grains in the quadrilateral Ta/(Ta + Nb) *versus* Mn/(Mn + Fe) diagram. Enrichment tends of Ta and Nb (grey arrows) observed for CGM from various types of pegmatites according to Černý (1989). (B) Sn + Ti + Zr *vs.* Mn/(Mn + Fe) (atoms per formula unit, apfu) binary diagram showing a poorly defined general trend of $M^{4+}$ traces impoverishment with increase of (Mn/Mn + Fe) ratio. (C and D) Trace element *vs.* Ta + Nb + Mn + Fe (apfu). (E) Zr + Ti *vs.* Sn (apfu) binary diagram showing the correlation between Zr, Ti and Sn in columbite-tantalite grains within almost all pegmatites fields. (F) U *vs.* Sn (apfu) binary diagram showing the lack of correlation between the uranium and the tin contents within studied columbite-tantalite grains.

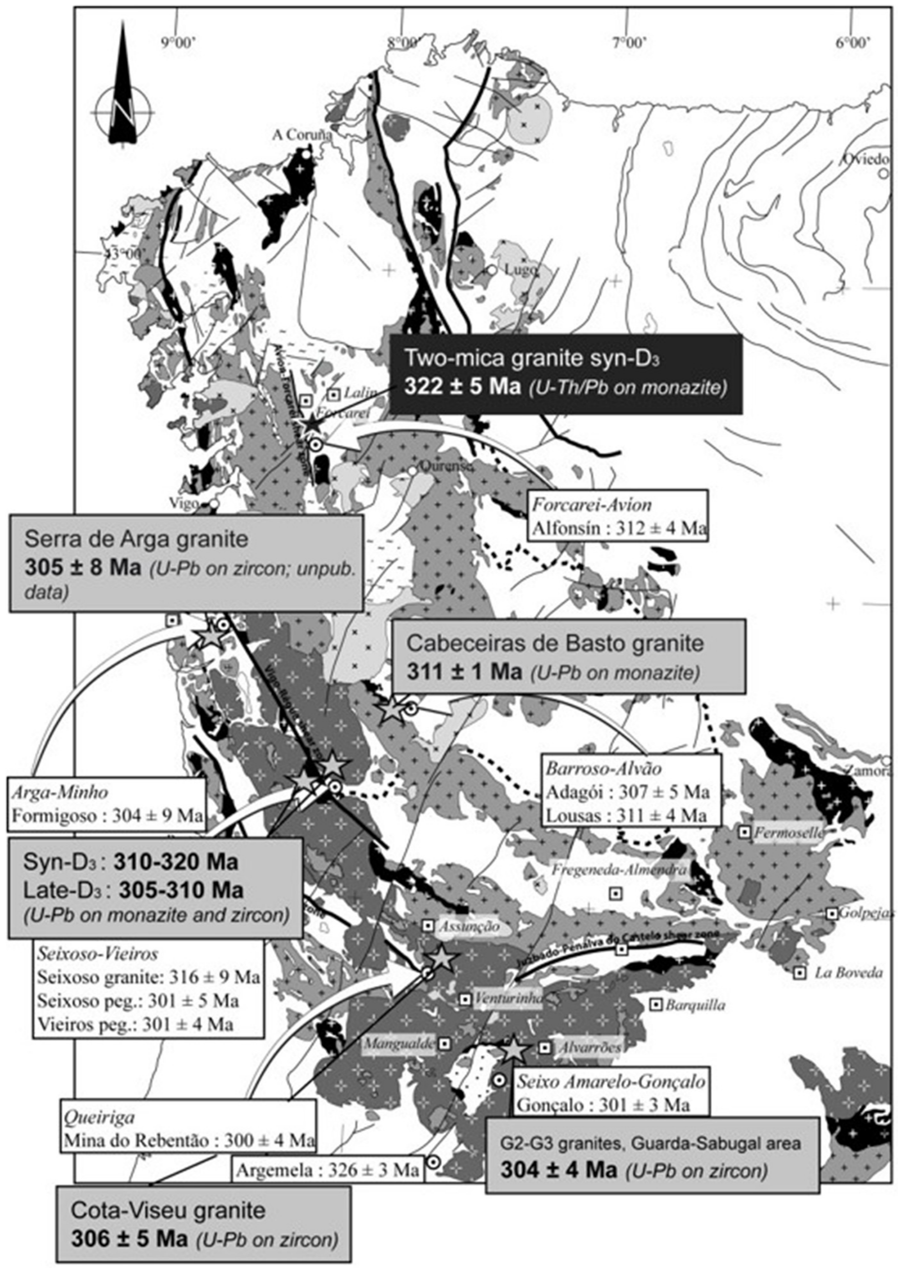


**Fig. 12.** Results from pegmatite dating showing a spatial organization of ages of emplacement. Other pegmatite fields are included to show the spatial relationship between most of the pegmatite fields and shear zones. Dark grey stars and boxes represent supposed "parent" granites that are not contemporaneous with neighbouring rare-element pegmatite fields. Light grey stars and boxes represent supposed "parent" granites that are contemporaneous with neighbouring rare-element pegmatite fields.

conclude, we argue for the relevance of using CGM to date ore deposits related to rare-element granite and pegmatite.

### Early rare-element granite emplacement

Granitoids emplaced during the last ductile deformation phase D3 predominate in the CIZ. Given the current state of knowledge, geochronological data indicate two main periods of rare-element pegmatite and granite emplacement, a first event at 330–320 Ma with a peak at 320 Ma, and a second event at 314–299 Ma, with a paroxysm at around 308 Ma (Fig. 12) (Dias *et al.*, 1998; Valle Aguado *et al.*, 2005; Zeck *et al.*, 2007; Ruiz *et al.*, 2008; Antunes *et al.*, 2008, 2013; Solá *et al.*, 2009; Carracedo *et al.*, 2009; Martins *et al.*, 2009, 2013; Neiva *et al.*, 2009, 2011, 2012; Diaz-Alvarado *et al.*, 2011; Teixeira *et al.*, 2011, 2012; Orejama *et al.*, 2012; Carvalho *et*

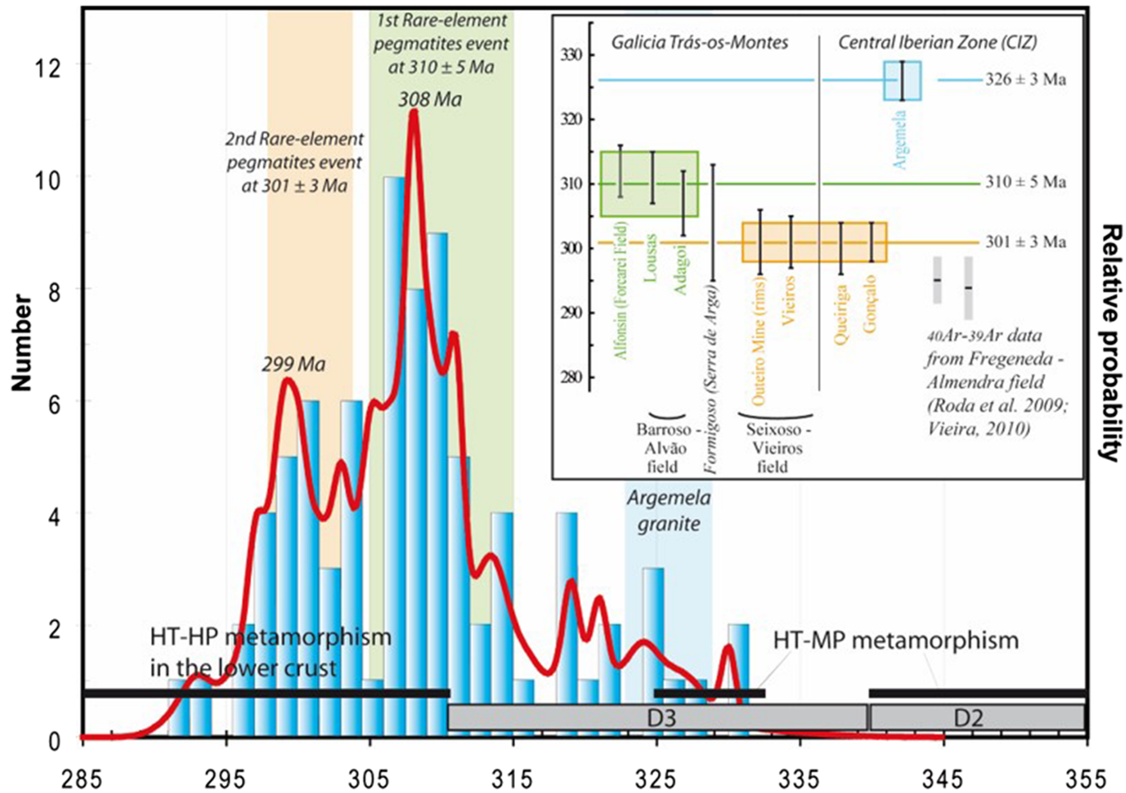


**Fig. 13.** Summary of results from the geochronological study and comparison with emplacement ages of granites (compilation of geochronological data from Dias *et al.*, 1998, 2002; Valverde-Vaquero *et al.*, 1999; Fernández-Suárez *et al.*, 2000; Martin-Izard *et al.*, 2000; Valle Aguado *et al.*, 2005; Bea *et al.*, 2006; Zeck *et al.*, 2007; Ruiz *et al.*, 2008; Antunes *et al.*, 2008, 2013; Carracedo *et al.*, 2009; Solá *et al.*, 2009; Martins *et al.*, 2009, 2013; Neiva *et al.*, 2009, 2011, 2012; Diaz-Alvarado *et al.*, 2011; Gutiérez-Alonso *et al.*, 2011; Teixeira *et al.*, 2011, 2012; Orejama *et al.*, 2012), tectonic phases, HT-MP metamorphic events (data from Montero *et al.*, 2004 and Bea *et al.*, 2009) and the HT-HP metamorphic event (Fernández-Suárez *et al.*, 2006).

*al.*, 2012). Three main granitic suites have been described: (i) the early syn-D3 peraluminous granodiorite-granite suite, derived from melting of mixed mafic and felsic lower crust components, or mixing of felsic crustal melts with mantle-derived magmas (Valle Aguado *et al.*, 2005; Teixeira *et al.*, 2011); (ii) the syn-D3 peraluminous leucogranite suite, mainly originating from a moderate degree of partial melting under water-undersaturated conditions of metasedimentary crustal sources (Beetsma, 1995; Teixeira *et al.*, 2012); and (iii) the late- to post-D3 biotite ± cordierite-bearing granodiorite-granite suite, forming large, composite batholiths and possibly originating from mixing of mantle-derived magmas and crustal felsic melts during petrogenesis (Azevedo and Nolan, 1998; Castro *et al.*, 1999; Dias *et al.*, 2002; García Moreno and Corretgé, 2007).

Dating columbite-(Mn) from the Argemela rare-element granite gave an age of 326 ± 3 Ma, which corresponds to the earliest rare-element event highlighted by our study (Fig. 13). Given the current state of knowledge about the chronology of Variscan magmatic activity within the CIZ, no other contemporaneous granitic body has been found in the vicinity of the Argemela rare-element granite. However, Teixeira *et al.* (2012) dated several two-mica granites near Carrazeda de Ansiães, about 150 km to the North, and obtained similar ages of 324 ± 4 Ma and 324 ± 2 Ma. These granites are interpreted to be derived from partial melting of heterogeneous metasedimentary material. To the west, along the Porto-Tomar fault zone, migmatitization is dated at 335 ± 4 Ma (U-Pb SHRIMP on zircon overgrowths (Ordóñez-Casado, 1998) and granites between 335 and 318 Ma (Pereira *et al.*, 2010 and references therein).

Charoy and Noronha (1991, 1996) showed that the Argemela rare-element granite was emplaced at an upper level in the crust. This means that this portion of the Iberian Variscan Massif was not thickened during the Variscan orogeny. In the northern French Massif Central, the Beauvoir and Montebras rare-element granites were also emplaced at a high level in the crust, but are significantly younger with an age of 308 ± 2 Ma (Cheilletz *et al.*, 1992; Cuney *et al.*, 2002). Therefore, on the basis of our results, the Argemela granite was emplaced almost 20 Ma earlier and is the earliest rare-element granite currently known in the Variscan belt.

## Temporal relationships between rare-element pegmatites and granitic magmatism

The generally preferred genetic model for rare-element pegmatites recognizes a spatial and genetic relationship between fertile "parent" granite and a swarm of increasingly fractionated pegmatites with distance from the granite (*e.g.*, Černý *et al.*, 2005; London, 2008). In this model, granites and pegmatites are supposed to have the same ages within the uncertainties of the currently available dating methods.

Granite batholiths can be built during several successive pulses spread over more than 10 Ma (Coleman *et al.*, 2004; Neiva *et al.*, 2012; Lima *et al.*, 2012; Teixeira *et al.*, 2012; Carvalho *et al.*, 2012). Conversely, several authors have argued for very fast emplacement and crystallization of granitic intrusions (Bea *et al.*, 2007; Michel *et al.*, 2008), *e.g.*, over less than 1 Ma. Because the uncertainties of U-Pb dating are generally greater than 1 Ma, we make the assumption that rare-element granites and pegmatites and their supposed "parent" granites, when their ages differ within uncertainties, are not petrogenetically related.

Most of the studied areas show contemporaneous emplacement of granitoids and pegmatites. The two types of rare-element pegmatites from the Barroso-Alvão pegmatite field provide ages of 311 ± 4 Ma, for the Lousas petalite-bearing pegmatite, and of 307 ± 5 Ma, for the Adagói spodumene-bearing pegmatite. Several authors consider the Cabeceiras de Basto granite, which shows little enrichment in Li, Sn W and F and is located south of the Barroso-Alvão pegmatite field, as a potential source for these rare-element pegmatites (Lima, 2000; Almeida *et al.*, 2002). The ages obtained are consistent with the 311 ± 1 Ma (U-Pb on monazite) minimum age of crystallization of the Cabeceiras de Basto granite proposed by Almeida *et al.* (2002). However, mineral study and spatial statistical analyses discarded spatial relationships between the Barroso-Alvão pegmatite and the Cabeceiras de Basto granite (Martins *et al.*, 2012; Silva *et al.*, 2018).

CGM from the Formigoso pegmatite (Serra de Arga field) gave an age of 304 ± 9 Ma. In this case, the greater uncertainties are due to the limited number of analyses (*i.e.*, six analyses). Previous work linked the development of the Arga-Minho pegmatite field with emplacement of the Serra de Arga granite (Gomes and Nunes, 1990; Nunes and Gomes, 1994; Gomes, 1995), however published geochronological data for the area are lacking. Gomes (1995) suggested an age of 305 ± 6 Ma (unpublished data) for emplacement of the Serra de Arga granite. In this case, the two ages are coeval.

CGM from the Outeiro Mine granite gave ages of 316 ± 9 Ma and 301 ± 5 Ma. There are two possible interpretations for these two ages: (i) the Outeiro Mine granite was emplaced at 316 ± 9 Ma and was affected by post-crystallization disturbance at 301 ± 5 Ma that caused partial lead loss or solution/recrystallization; or (ii) the Outeiro Mine granite formed at 301 ± 5 Ma and incorporated older inherited grains from its source or as a result of crustal contamination. Several arguments favour the first interpretation. CGM are not that common in crustal rocks. It is therefore very unlikely that the magma of the Outeiro Mine granite contained inherited xenocrysts. Moreover, older ages were obtained from two grains cores (Fig. 6) that have lower U concentrations than domains with younger ages. This is consistent with easier loss of radiogenic Pb in domains with high U concentrations during a later disturbance. The Vieiros pegmatite from the same field gave an age of 301 ± 4 Ma, interpreted as the age of emplacement. The Seixoso pegmatite, located 100 m from the Outeiro Mine granite, shows structural and mineralogical features similar to those of the Vieiros pegmatite. Therefore, emplacement of these pegmatites and associated fluids could have caused the disturbance that affected the Outeiro Mine columbite-tantalite. The Felgueiras syn-D3 granodiorite, located to the south, had not been dated directly. Dias *et al.* (2002) dated the Celorico de Basto late-D3 granite, located to the north, and obtained an age of 308 ± 4 Ma, making it contemporaneous with the Vieiros pegmatite. The older Outeiro Mine granite could be related to syn-D3 magmatic activity.

CGM from the Gonçalo lepidolite-subtype pegmatite gave an age of 301 ± 3 Ma. This pegmatite crosscuts the Guarda granite, which has been dated at 304 ± 4 Ma (SHRIMP U-Th-Pb on monazite; Neiva *et al.*, 2009). However, Neiva and Ramos (2010) argue that these pegmatites are related to later porphyritic biotite > muscovite granites, which has been dated at about 300 ± 1 Ma and 301 ± 2 Ma (U-Pb on zircon and monazite; Neiva *et al.*, 2011). The data obtained are therefore in good agreement with these observations.

The Mina do Rebentão petalite-subtype pegmatite from the Queiriga pegmatite field was dated at 300 ± 4 Ma, which is the same age as the Cota-Viseu granite (306 ± 5 Ma; Valle Aguado *et al.*, 2005) whose edge is crosscut by the pegmatite. However, the Mina do Rebentão pegmatite was emplaced under brittle conditions, which argues against the existence of a genetic link between this pegmatite and the host granite.

Finally, only one pegmatite lead to an age that suggests unrelated emplacement of pegmatites and granitoids. Indeed, analyses of CGM from the Alfonsín pegmatite (Forcarei-Avíon field) provided an age of 312 ± 4 Ma. Using geochemical arguments, several authors suggested that a relationship exists between aplites-pegmatites from the area and syn-D3 peraluminous two-mica granites (Fuertes-Fuente, 1998; Fuertes-Fuente and Martín-Izard, 1998). However, Gloaguen (2006) dated emplacement of the syn-D3 peraluminous two-mica granite at 322 ± 5 Ma (U-Th/Pb on monazite). Thus, our results suggest that no temporal relationship exists between pegmatites and neighbouring granite.

In conclusion, our results suggest that there are no links between the rare-element pegmatites and their neighbouring late-D3 biotite ± cordierite granites. This is in good agreement with field observations from the Barroso-Alvão field, where pegmatites are clearly deformed (Martins, 2009).

## Three rare-element events in the Iberian Massif

The U-Pb dating of CGM from nine rare-element pegmatites and granites from the Iberian Massif provides the first evidence that rare-element magmatism occurred at least during three temporal events in this part of the Variscan belt (Fig. 13).

In the CIZ, the Argemela granite was emplaced during an early event at 326 ± 3 Ma. As highlighted previously, granitoids of this age are rare in the CIZ (Fig. 13) and are located more in the north. This magmatism was related to a first melting event that occurred at the beginning of the D3 deformation phase.

In the GTOMZ, dated pegmatites from both the Forcarei-Avíon field and the Barroso-Alvão field gave an average age of 310 ± 5 Ma (Fig. 13). Emplacement of these rare-element pegmatites was contemporaneous with the main granitoid emplacement event, which occurred at the end of the D3 tectonic phase at about 308 Ma (Fig. 13). Given the uncertainties, this second event could be contemporaneous with either the early syn-D3 granodiorite-granite suites or the syn-D3 leucogranite suites. During our work, we did not observe any evidence of this event in the CIZ. However, obtained age at 305 ± 9 Ma for the Tres Arroyos aplo-pegmatites (U-Pb on columbite group minerals; Garate-Olave *et al.*, 2020), localized in the southern part of the CIZ, could be part of this event or related to a third later event.

The third event corresponds to the emplacement of pegmatites from the Seixoso-Vieiros (GTOMZ) and the Queiriga and Gonçalo (CIZ) fields at 301 ± 3 Ma (Fig. 13). These results and field observations clearly show the absence of relationship between this event and emplacement of the late- to post-D3 biotite ± cordierite-bearing granite suite. The rare-element pegmatites of the Fregeneda-Almendra field have been dated at about 300 Ma by $^{40}$Ar-$^{39}$Ar dating on micas (Roda *et al.*, 2009; Vieira, 2010). Emplacement of these bodies could be related to the event at 301 ± 3 Ma highlighted by our study. Emplacement of this rare-element pegmatite was contemporaneous with a second peak of emplacement of granitoids at about 299 Ma (Fig. 13).

The age 304 ± 9 Ma obtained for the Formigoso pegmatite from the Arga-Minho field is difficult to associate to either of the two events because the uncertainly is too large (Fig. 13). The Arga-Minho field is bordered to the east by the Vigo-Régua shear zone (Fig. 12), as is the Seixoso-Vieiros field. One possibility is that this fault played a role in the emplacement of the two pegmatites fields. If such is the case, the two fields could be contemporaneous, with emplacement of the pegmatites of the Arga-Minho field occurring during the third event at 301 ± 3 Ma. On the other hand, the presence of early-D3 granodiorite-granite suites emplaced along the Vigo-Régua shear zone shows that this structure was active during the first stage of the D3 event (Pamplona *et al.*, 2006). The rare-element Arga-Minho pegmatites could therefore also be related to the 310 ± 5 Ma event.

Granitoids from northwestern Iberia generally have asymmetric, elongate shapes parallel to the main structural features of the belt (Fig. 1). Some of them, for example the early D3 granodiorite-granite suite, show strong spatial relationships with major shear zones. In comparison, contemporaneous pegmatite fields dated during this study appear to be distributed following the two geotectonic domains of the inner part of the Iberian Variscan massif, the GTOMZ and the CIZ. Emplacement ages of rare-element pegmatites decrease toward the outer part of the belt, with younger ages in the CIZ (Fig. 12). This agrees with the documented propagation of deformation, metamorphism and magmatism in the different geotectonic zones as proposed by Dallmeyer *et al.* (1997). However, additional geochronological and structural studies are needed to confirm this observation.

As mentioned previously, granitoids are widespread within the Variscan Iberian massif. In the CIZ and in particular during the D3 event, large-scale emplacement of peraluminous bodies occurred over almost 30 Ma (Fig. 12).

Given our results, it is surprising that emplacement of rare-element pegmatites or granites does not appear to be related to one specific event of granite emplacement; on the contrary, it was widespread over time. Moreover, no specific type of "parent" peraluminous granite has been identified in the Variscan Iberian massif, although some similarities do exist in terms of chemical and mineralogical composition between rare-element pegmatites of different ages, for example between the rare-element pegmatites of the Barroso-Alvão field (about 310 Ma) and those of the Fregeneda-Almendra field (about 295–300 Ma, Roda *et al.*, 2009). In the CIZ, Roda-Robles *et al.* (2018) proposed that lithium-rich pegmatites are related to two micas peraluminous leucogranites in its northern part and to P-rich highly peraluminous granites in its southern part on the basis of spatial and geochemical relationships.

In addition, in the French Massif Central and the South Brittany Variscan domain, where peraluminous granites with ages equivalent to those of peraluminous granites from the GTOMZ and CIZ are also widespread, rare-element pegmatites are essentially absent, with only one field known in the Mont d'Ambazac (Limousin, France, Raimbault, 1998). The situation is completely different in the Iberian massif where several very important fields of rare-element pegmatites are known.

High-temperature, medium- to low-pressure metamorphism (HT-MP) documented in the Iberian massif occurred from about 360 to 340 Ma during the D2 phase and from 332 to 325 Ma during the D3 phase (Montero *et al.*, 2004; Bea *et al.*, 2009 and references therein). The Argemela granite was emplaced at the end of the second event. The two rare-element pegmatite emplacement events are not related to any documented HT-MP metamorphic event. The situation is different in the Moldanubian domain of the Variscan belt (Eastern Europe), where rare-element pegmatites are contemporaneous with HT-MP metamorphic events, during the main orogenic activity of this part of the Variscan belt (Melleton *et al.*, 2012). However, the contemporaneity of rare element magmatic events in Iberia and in the French Massif central is remarkable. Indeed, dating of several rare elements pegmatites and granites lead to similar middle to late Pennsylvanian ages (Cheilletz *et al.*, 1992; Melleton *et al.*, 2015; Marcoux *et al.*, 2021).

## Implications for rare-element melts generation

Several studies (Marignac and Cuney, 1999; Cuney *et al.*, 2002; Cuney and Barbey, 2014) argued that rare-element granite and pegmatite emplacement events in the French Massif Central, dated at 308 ± 2 Ma (Cheilletz *et al.*, 1992), are coeval with HP-HT metamorphism of the lower crust. This metamorphism would result in devolatilization of the lower crust, producing fluids enriched in F, Li and other rare elements. These fluids would induce a low degree of partial melting of an unknown protolith of the middle crust. The produced melts would then be collected and channelled along vertical structures in the crust.

A number of observations favour a middle- to upper-crust source origin for the rare-element melts of the Iberian Massif:

-The existence of at least three distinct rare-element magmatic events in the Iberian Massif, in particular two events separated by almost 30 Ma in the CIZ. Our dataset strongly argues against the involvement of lower crustal processes, because devolatilization of the lower crust can be considered as an irreversible, secular event occurring during a relative short time span. Moreover, Fernández-Suárez *et al.* (2006) dated lower crustal granulite xenoliths from the Spanish Central system by U-Pb on zircon and concluded that the Iberian lower crust recorded HT metamorphic conditions only between 310 Ma and 280 Ma during the Variscan orogeny. Some rare-element pegmatites and granites dated in our study predate this metamorphic event (Fig. 12) and thus cannot have been generated by the mechanisms proposed by Marignac and Cuney (1999) and Cuney *et al.* (2002).

-In the Iberian Massif, rare-element pegmatites and granite suites were emplaced under similar spatial and temporal conditions. Because of their strong crustal affinities, the leucogranite suites are considered the best candidates for the generation of LCT-type pegmatites (review in Černý *et al.*, 2005, and London, 2008). Neiva and Ramos (2010) reached the same conclusion for the rare-element pegmatites of the Seixo Amarelo-Gonçalo field. Castro *et al.* (1999) suggested that sources of peraluminous leucogranites could be gneiss and greywackes. Their experimental work also highlighted that the amount of melt needed to generate the large outcropping plutons would be produced at low pressure, during decompression. This suggests that potential sources of the rare-element-enriched melts, such as peraluminous two-mica granites, would more likely be located in the upper to middle crust. In Central and Northern Portugal, recent studies argued for mid-crustal metasedimentary sources for the two micas leucogranite (Carvalho *et al.*, 2012; Costa *et al.*, 2014). On the other hand, our dataset does not allow us to recognize the genetic relationships between rare-element pegmatites and granodiorite-granite suites or two-mica peraluminous granite suites. Both types of granitic magma were emplaced over a short period and mainly simultaneously, indicating that meta-igneous and metasedimentary sources melted contemporaneously.

The existence of at least three different rare-element events between 330 and 300 Ma in the Iberian Variscan Massif strongly argues against any genetic relationships between lower crustal processes and the origin of rare-element melts. Additional geochemical and geochronological investigations of migmatites and high-grade formations of the GTOMZ and the CIZ are needed to study this hypothesis. In particular, investigations of melt inclusions in peritectic minerals from a metasedimentary protolith that underwent a very low degree of melting could provide important constraints on the chemistry of the first melts generated (Bartoli *et al.*, 2013). New experimental petrology studies are also required to definitely eliminate (Černy *et al.*, 2005; London, 2008) or confirm this hypothesis. On the other hand, the scarcity of rare-element granites and pegmatites and their location in restricted geographic areas of an orogenic belt strongly suggest that particular pre-enriched fertile sources are a prerequisite for the generation of rare-element melts. The role of deformation in the segregation, migration and emplacement of rare-element-enriched melts is also of first importance to understand the generation of these magmas. Several recent studies highlighted the role of deformation structures in the emplacement of rare-element pegmatites (Demartis *et al.*, 2011; Deveaud *et al.*, 2013). As discussed earlier, because of the apparent spatial relationships of several rare-element pegmatite fields in the Iberian Variscan belt, structural studies dedicated to these fields could provide additional constraints on the mechanisms that gave rise to rare-element melts.

### U-Pb dating of columbite-group minerals to date Li-, Be-, Sn-, W-, Ta-, Nb- or REE-bearing ore deposits

Establishing geochronological constraints is a key feature to understand the genesis of most ore and mineral deposits. However, in many cases, this can prove very difficult because of the particular nature of some mineral assemblages. Previous geochronological studies based on U-Pb dating of CGM demonstrated the validity of the method used to date emplacement of pegmatite from the Paleoproterozoic to the Cretaceous (Romer and Wright, 1992; Romer and Smeds, 1994, 1996, 1997; Romer and Lehmann, 1995; Glodny *et al.*, 1998; Melcher *et al.*, 2008; Melleton *et al.*, 2012; Deng *et al.*, 2013; Che *et al.*, 2015, 2019; Van Lichtervelde *et al.*, 2017; Zhou *et al.*, 2016; Legros *et al.*, 2019; Feng *et al.*, 2020; Garate-Olave *et al.*, 2020). Moreover, assimilation of common Pb and loss of radiogenic Pb seem to occur more rarely in CGM than in zircon (Melleton *et al.*, 2012). During our study, we dated several grains of columbite-(Mn), columbite-(Fe), tantalite-(Mn) and tantalite-(Fe). Our results did not reveal any differences in the behaviour of the U-Pb system in the four minerals of the columbite group, and we were able to use these minerals to date the emplacement of several distinct ore bodies. This is in agreement with observation of other studies dedicated to columbite-group minerals dating (Che *et al.*, 2019; Feng *et al.*, 2020). Moreover, metamictization processes do not seem to affect U-Pb isotopic systematics in CGM (Feng *et al.*, 2020).

Because columbite-group minerals occur in a wide range of ore deposits related to peraluminous magmatism (rare-element pegmatites and granites, Linnen *et al.*, 2012; Sn-W deposits, Lerouge *et al.*, 2007) and also in some REE- and Nb-bearing carbonatites (Melcher *et al.*, 2008), they appear to provide useful and powerful geochronological constraints on the formation of these ore deposits. Nevertheless, age uncertainties need to be strongly reduced in order to decipher chronological relationships in geological settings with long-lived magmatic activities, such as the Variscan orogeny.

## Conclusions

In situ U-Pb dating by LA-SF-ICP-MS of columbite-group minerals from Iberian rare-element granites and pegmatites provides new constraints on the generation of rare-element melts during the Variscan orogeny. Three events have been recognized. The Argemela granite, in the Central Iberian Zone (CIZ), with an age of 326 ± 3 Ma, provides some of the oldest evidence of magmatic activity in this part of the Variscan belt. Rare-element pegmatites of the LCT family from the Galicia-Trás-os-Montes Zone (GTOMZ), which gave an average age of 310 ± 5 Ma, contemporaneous with the early syn-D3 granodiorite-granite suite or the syn-D3 leucogranite suite, represent the second event. The third event corresponds to the emplacement of rare-element pegmatites of the LCT family in the CIZ and in the southern GTOMZ at about 301 ± 3 Ma, which corresponds to the end of late- to post-D3 granitic suites emplacement.

Contemporaneous fields of rare-element pegmatites are arranged in belts similar to those formed by granitoid suites. Pegmatites fields from both the GTOMZ and the CIZ reveal a southward younging trend of emplacement ages, which matches the propagation of deformation, metamorphism and magmatism in the different geotectonic zones.

The existence of three different rare-element events in the Iberian Massif argues against the involvement of lower crustal HP-HT metamorphism in the generation of rare-element melts as the emplacement of rare-element pegmatites does not appear to have been coeval with documented HT-MP metamorphism. Possible sources of rare-element-enriched melts are more likely located in the middle to upper crust, as are the major components of granitic magmatism.

The reproducibility of analyses of U and Pb isotopes observed during this study is a good argument to consider that columbite-group minerals are good candidates to perform geochronological study of ore deposits related to peraluminous magmatism and some REE- and Nb-bearing deposits.

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## Supplementary material

**Table S1.** LA-ICP-MS U-Pb isotope data for columbite and tantalite from rare-element pegmatites and granites from the Galicia-Trás-os-Montes Zone.
**Table S2.** LA-ICP-MS U-Pb isotope data for columbite and tantalite from rare-element pegmatites and granites from the Central Iberian Zone.
**Table S3.** Chemical compositions of dated CGM grains measured by EPMA.

The Supplementary Material is available at http://www.bsgf.fr/10.1051/bsgf/2022004/olm.

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