# ERA-MIN Joint Call 2019

*MOSTMEG*

*Predictive models for strategic metal rich, granite-related ore systems based on mineral and geochemical fingerprints and footprints*

Second Annual Project Report

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# GENERAL INFORMATION

| | |
|---|---|
| **Project acronym** | MOSTMEG |
| **Project title** | *Predictive models for strategic metal rich, granite-related ore systems based on mineral and geochemical fingerprints and footprints* |
| **Project start date (day/month/year)**<br><br>**Project end date (day/month/year)** | 01/10/2020<br><br>30/09/2023 |
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| **Project website** | https://mostmeg.rd.ciencias.ulisboa.pt/ |
| **Date of submission of the report** | 31/10/2022 |
| **Consortium Coordinator** | *Project Partner 1 – Faculdade de Ciências da Universidade de Lisboa and IDL*<br>*António Manuel Nunes Mateus*<br>*E-mail address: amateus@fc.ul.pt*<br>*female ☐ male ✗*<br>*Portugal*<br>*Partner type 1: Academia ✗ Large Company ☐ SME ☐ NGO ☐* |
| **Project Partners (other than Consortium Coordinator)** | *Project Partner 2 – Faculdade de Ciências da Universidade do Porto*<br>*Alexandra Guedes*<br>*E-mail address: aguedes@fc.up.pt*<br>*female ✗ male ☐*<br>*Portugal*<br>*Partner type: Academia ✗ Large Company ☐ SME ☐ NGO ☐* |
| | *Project Partner 3 – Universidade de Évora, Geociências - HERCULES*<br>*Patrícia Moita*<br>*E-mail address: pmoita@uevora.pt*<br>*female ✗ male ☐*<br>*Portugal*<br>*Partner type: Academia ✗ Large Company ☐ SME ☐ NGO ☐* |
| | *Project Partner 4 – Laboratório Nacional de Energia e Geologia, I.P.*<br>*Rute Maria Morgado Salgueiro*<br>*E-mail address: rute.salgueiro@lneg.pt*<br>*female ✗ male ☐*<br>*Portugal*<br>*Partner type: Academia ✗ Large Company ☐ SME ☐ NGO ☐* |

[1] Academia (universities, other higher education institutions or public research organizations-PRO)

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|---|---|
| | *Project Partner 5 – Universidade de Coimbra*<br>*Alcides Pereira*<br>*E-mail address: apereira@dct.uc.pt*<br>*female ☐ male ✗*<br>*Portugal*<br>*Partner type: Academia ✗ Large Company ☐ SME ☐ NGO ☐* |
| | *Project Partner 6 – Instituto de Geociências da Universidade de São Paulo, Departamento de Mineralogia e Geotectónica e Centro de Pesquisas Geocronológicas*<br>*Colombo Celso Gaeta Tassinari*<br>*E-mail address: ccgtassi@usp.br*<br>*female ☐ male ✗*<br>*Brazil*<br>*Partner type: Academia ✗ Large Company ☐ SME ☐ NGO ☐* |
| | *Project Partner 7 – CNRS - GeoRessources*<br>*Michel Cathelineau*<br>*E-mail address: michel.cathelineau@univ-lorraine.fr*<br>*female ☐ male ✗*<br>*France*<br>*Partner type: Academia ✗ Large Company ☐ SME ☐ NGO ☐* |
| **GDPR consent** | I, as coordinator of this project, accept that my name and email address are public at ERA-MIN 2 media channels for dissemination and exploitation purposes.<br>*yes ✗ no ☐* |

# A. PROGRESS REPORT

### A.1 Please list the project meetings and workshops held during the implementation period:

| Meeting objective | Partners involved | Date | Place |
|---|---|---|---|
| Steering Group 4th Meeting | All | March 4, 2022 | Video-conference |
| 2nd Annual Meeting and Steering Group 5th Meeting | All | October 03, 2022 | Hybrid attendance (virtual and in-person) |

In addition to the listed meetings, several others occurred involving different subgroups of partners (and researchers) to discuss in detail specific problems and results meanwhile obtained. A Joint Fieldtrip (May 30 – June 2, 2022), involving three researchers from FCUL and two from IGc-USP, was organised to: (i) provide the main highlights on the geological background of the Segura-Argemela area; (ii) observe and discuss data provided by key-exposures of mineralised structures (arrays of aplite/pegmatite veins and quartz lode systems) and their host rocks; and (iii) increase the sampling densification in specific targets to support forthcoming detailed studies (WP2 and WP5). The 2nd Annual Meeting and Steering Group 5th Meeting were preceded by a Joint Fieldtrip (September 29 – October 2, 2022) attended by 14 researchers from 4 project partners (CNRS-GeoRessources, FCUL, UCoimbra and LNEG). This Joint Fieldtrip was instrumental: (i) to realise some particular geological features of the Góis-Segura strip western segment, namely in the sector of Figueiró dos Vinhos – Pedrogão Grande – Castanheira de Pêra; (ii) to discuss the recently obtained field data and complement the sampling programme in the Argemela and Mata da Rainha areas, consolidating the ongoing research related to WP3, WP4 and WP5; and (iv) to refine several details of the intended research plan, reinforcing the collaborative efforts between the project partners.

### A.2 Progress

| WP no. | Work package title | Milestones | Work package leader | Participating partners | Planned delivery date | Effective delivery date |
|---|---|---|---|---|---|---|
| 1 | Re-assessment and harmonisation of the available data, structural analysis and sampling | M1.1 | P5 & P1 | P1, P5, P4 | Month 15 | Month 20<br>(including detailed geological mapping of selected targets) |
| 2 | Vectoring metal endowment and critical timing for mineralisation triggering | M2.2 | P6 & P1 | P1, P6, P7 | Month 15 | Month 24 |
| 3 | Relevant processes for metal concentration and deposition at the ore system scale | M3.2<br>M3.3<br>M3.4 | P2 & P7<br>P2 & P7<br>P2 & P7 | P1, P2, P7<br>P1, P2, P7<br>P1, P2, P7 | Month 12<br>Month 20<br>Month 24 | Month 24<br>Month 29<br>Month 33 |
| 4 | Mineral fingerprints and footprints | M4.2<br>M4.3 | P3 & P7<br>P3 & P7 | P4, P1, P3<br>P3, P7, P1, P4 | Month 16<br>Month 30 | Month 25<br>Month 38 |
| 5 | Geochemical fingerprints and footprints | M5.1<br>M5.2 | P1& P6<br>P1 & P6 | P1, P6, P7<br>P1, P6, P7 | Month 13<br>Month 24 | Month 22<br>Month 33 |

During the 2nd year, research activities were mainly oriented towards the objectives of WP2 (tasks 2.1 and 2.2), WP3 (tasks 3.1, 3.2 and 3.3) and WP4 (task 4.3). Detailed geological surveys and sampling, complementing the "regional data" gathered in WP1 (tasks 1.1, 1.2 and 1.3), were performed in the Argemela, Mata da Rainha, Medelim and Segura areas. Planned activities related to tasks 5.1 and 5.2 (WP5) continued. Researchers from all the consortium partners participate in these activities. The justification for the "*effective delivery dates*" indicated in the table is provided in the following sections, supporting the nine-month deadline extension formally requested for the MOSTMEG project.

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### A.2.1 Milestones (M) accomplishment

**M1.1** (WP1) is accomplished, just awaiting the incorporation of data provided by the UCoimbra partner, thus completing the GIS-supported database for the Góis-Segura strip. Meanwhile, detailed fieldwork was performed in the Argemela, Mata da Rainha, Medelim and Segura areas. Considering the available results, we intend to detail the geological survey of the Fundão pluton and its neighbouring envelope. Additional work was also carried out to refine the alluvial cassiterite, wolframite, and scheelite concentration map interpretation for the Segura region (Polygon 1a, b; priority study area).

**M2.1** (WP2) was fully achieved. Additional samples representing specific features of several mineralising systems were collected on May/June 2022 and September 2022. Selected samples (complementary to the existing ones) will be picked in the forthcoming months to address specific questions and complete the intended geochemical and mineralogical databases.

**M2.2** (WP2) was largely completed. Multi-element whole rock analyses were obtained for an extensive collection of samples (>220). Whole-rock, multi-system (Sr-Nd-Pb) isotopic analyses were concluded for 75 samples. Zircon grains were extracted from 28 selected samples (different granitic facies, aplite and pegmatite bodies), examined with cathode-luminescence and analysed with SHRIMP over the first two years of the project. Further studies involving additional samples (already collected) and/or different analytical methods (e.g. Ar-Ar, Rb-Sr) will be done in the following months to achieve the purposes of WP2 and WP3 fully.

The intended goals for **M3.1/3.2** (WP3) were mainly attained. The petrographic examination of a large number (> 250) of thin polished sections was performed, followed by a systematic mineral compositional analysis in selected sample sets using EPMA (FCUL) and ICP-MS-LA (CNRS-GeoRessources). The work needed to accomplish **M3.3** (WP3), previewed for month 20th, is behind schedule due to equipment problems in the Raman Spectroscopy laboratory at FCUP and traveling difficulties that delayed the sampling program carried out by the CNRS-GeoRessources team. This delay caused problems in the accomplishment of **M4.2** and **M4.3**.

The planned aims for **M4.1** (WP4) were achieved. Part of the reported gaps in estimating cassiterite grains in alluvial samples from the Segura region were completed (LNEG, T4.3), strengthening the required map interpretation, which preliminary version was finished in December 2021. The initial inspection of heavy mineral fractions in alluvial samples, followed by exploratory EPMA chemical characterization of $TiO_2$ grain phases and tourmaline, was also done at LNEG and FCUL. Unfortunately, it was not possible to start the planned ICP-MS-LA data acquisition due to equipment malfunctions at the HERCULES Lab - Univ. Évora. This affected the intended progression of activities under **M4.2** and **M4.3**.

Although with some delay, caused by the accumulation of deferrals affecting intertwined previous milestones, **M5.1/M5.2** (WP5) were partially completed.

### A.2.2 Deliverables (D) accomplishment

The **D1.1/D1.2** (WP1) are still delayed due to the reasons pointed out above. The Sampling Dataset (**D1.3**) has been gradually updated (May/June 2022, September 2022) in selected targets, taking advantage of the joint field trips which took place in the meantime. Using an old but well-referenced collection existing at the

Univ. Porto, five samples from underground mining works (presently inaccessible) of Pedra Alta (Permit nº1, Argemela) were collected for detailed mineralogical and fluid inclusion studies of quartz lodes with Li-Sn mineralisation. Twenty-seven core samples from drill holes S1, S4, S8, S9, S11 and S12, representing quartz lodes and breccias with W-Sn mineralisation (Vale Pião, Góis), were also chosen for fluid inclusion studies, thus complementing the available geological information of this system. The extensive sampling programme so far completed does not exclude the possibility of adding extra samples, mainly collected during short-term field surveys in specific areas, such as Medelim, Mata da Rainha and Fundão.

The **D2.1/D2.2** (WP2) were completed nine months after the original plan, including some more samples than initially intended. Several other samples, already collected or to be picked soon, will complement the databases, providing relevant background information for the Góis-Segura strip and duly achieving the objectives stated in the project proposal.

The work needed to accomplish **D3.1/D3.2/D3.3** (WP3), previewed for month $20^{th}$, is delayed due to the reasons pointed out above for the corresponding milestones. Even so, some advances in data acquisition were recorded (as demonstrated in section **A.2.3**), allowing preliminary advancements towards **D4.1** and **D4.2**.

The work carried out in Task 4.3 has been proceeded to meet the objectives designed for **D4.3** (initially planned for month 30) on time. The identification, separation and characterization of heavy minerals from 51 samples collected in the priority area (Polygon 1, Segura region) are almost reaching the proposed goal (65 samples), which represents half of the minimum total samples expected to study (*i.e*. 100). For mineral exploration purposes, mineral physical properties (e.g. colour, shape, habit, size, pleochroism, colour zoning, and inclusions) beyond chemical composition are good indicators that can be quickly inspected by visual means. Several grain populations were identified and characterized under a binocular microscope. In addition, 493 grains of anatase, cassiterite, tourmaline, wolframite, garnet and ilmenite, representing distinct populations, were handpicked from different sampling points, mounted in epoxy, polished, and analysed with EMPA (1089-point analysis) and LA-ICP-MS (234-point analysis). Finally, regional distribution maps of mineral populations were produced, from where preliminary interpretations were made. However, additional critical information must be obtained to suitably characterise more alluvial heavy minerals and finish the study of the priority area; and, if possible, the analysis of the complementary regions (4 potential polygons) depending on the mineralogical content of the samples and the available time to do it under the scope of MOSTMEG.

Delays with publications and presentations at scientific meetings, reported for the 1st year, were partly overcome. Although behind our planned schedules, we are confident that the estimated figures will be fully attained.

### A.2.3 Results

The aim of **WP1** is the **re-assessment and harmonisation of the available data, structural analysis and sampling** in the Segura-Argemela-Panasqueira-Góis WNW-ESE strip. Following the preliminary regional assessment reported in October 2021, it was decided to densify the existing geological information for the Argemela, Mata da Rainha, Medelim and Segura areas, all of them selected for comprehensive studies in

WP3 and WP4. These targets were mapped in detail, and the new lithological and structural data were added to the GIS information layers compiled under the scope of MOSTMEG.

The **Argemela area** hosts exposures of a rare-metal granite at Cabeço de Argemela and different sets of mineralised quartz veins. Some of these quartz veins are part of the structural arrays exploited in the past at the Pedra Alta mine, which is included in the Li-Sn permit recently assigned to the PANNN Company (https://files.dre.pt/2s/2021/12/242000000/0010000100.pdf). The geological map produced (**Fig. 1**), the first to integrate data collected in Cabeço de Argemela, Pedra Alta and in the area surrounding the two preferential targets, together with the systematic structural survey carried out (**Figs. 2** and **3**), shows that:

(1) Some quartz infillings of NW-SE dextral shears were tentatively explored in the past, revealing distinct attributes of quartz veins in Cabeço de Argemela and Pedra Alta;

(2) The various groups of veins recognized in Cabeço de Argemela and Pedra Alta share several features, but straightforward comparisons between them should be avoided because some vein sets in the latter site preserve evidence of a long evolution, suggesting spatial superposition of effects due to diachronic mineralizing events;

(3) The hydrothermal alteration intensity experienced by metasediments adjoining the Cabeço de Argemela granite is strong, leading to significant compositional changes, and obliterating an early-developed (andalusite?) blastesis ascribable to contact metamorphism;

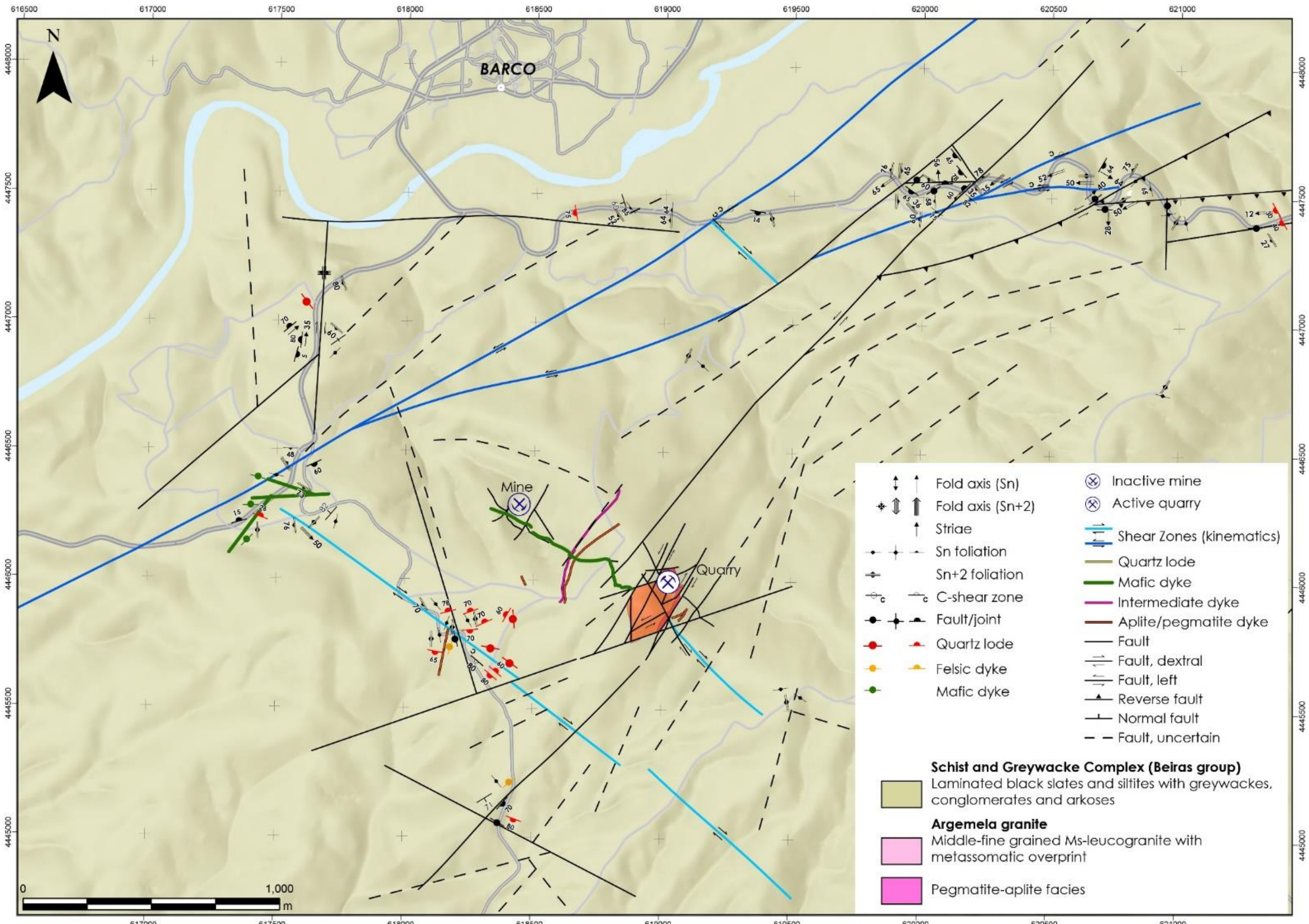
(4) This alteration, conceivably related to the mineralising events (or, at least, some of them) and expressed mainly by the growth of fine-grained mica (*bt*>*ms*) aggregates variably enriched in tourmaline, is identifiable macroscopically until ca. 25-30 m away from the contact with the granite;

(5) The ENE-WSW sinistral shear- or fault-bounded blocks positioned ca. 300-400 m to the North and East of the Cabeço de Argemela and Pedra Alta mineralised centres comprise metasediments variably affected by contact metamorphism, possibly denoting the influence of primary composition on andalusite (and cordierite?) blastesis, besides differences on the proximity to a granite batholith in depth; and

(6) The mineral blasts in these spotted schists are notably retrogressed, but there are no evident signs of *bt* mica or tourmaline.

Further work in the Argemela area will focus on the compositional and textural transformations recorded by metasedimentary rocks aiming at recognising of footprints for concealed mineralising systems, therefore completing the available (already published) information about Cabeço de Argemela and Pedra Alta.

The **Mata da Rainha area** encloses different types of old exploitation works (**Fig. 4**), the most important of which are Sn-W quartz lodes subjected to underground mining during the 2nd World War period, largely hosted in strongly modified (tourmaline-enriched) spotted schists near an *ms*-rich (greisen-like) facies. The latter is distinct from the two granite facies that form the core and the border of the Orca pluton. This “greisen” defines the outermost rim of this pluton in Mata da Rainha, and comprises clusters of tourmaline-rich aplite dykes (potentially carrying cassiterite), frequently coupled with quartz-tourmaline veins in the granite and metasediments, which show signs of artisanal mining. Future work will explore: (1) the geochemical differences of the mapped granite facies and aplite dykes, complementing the existent database and

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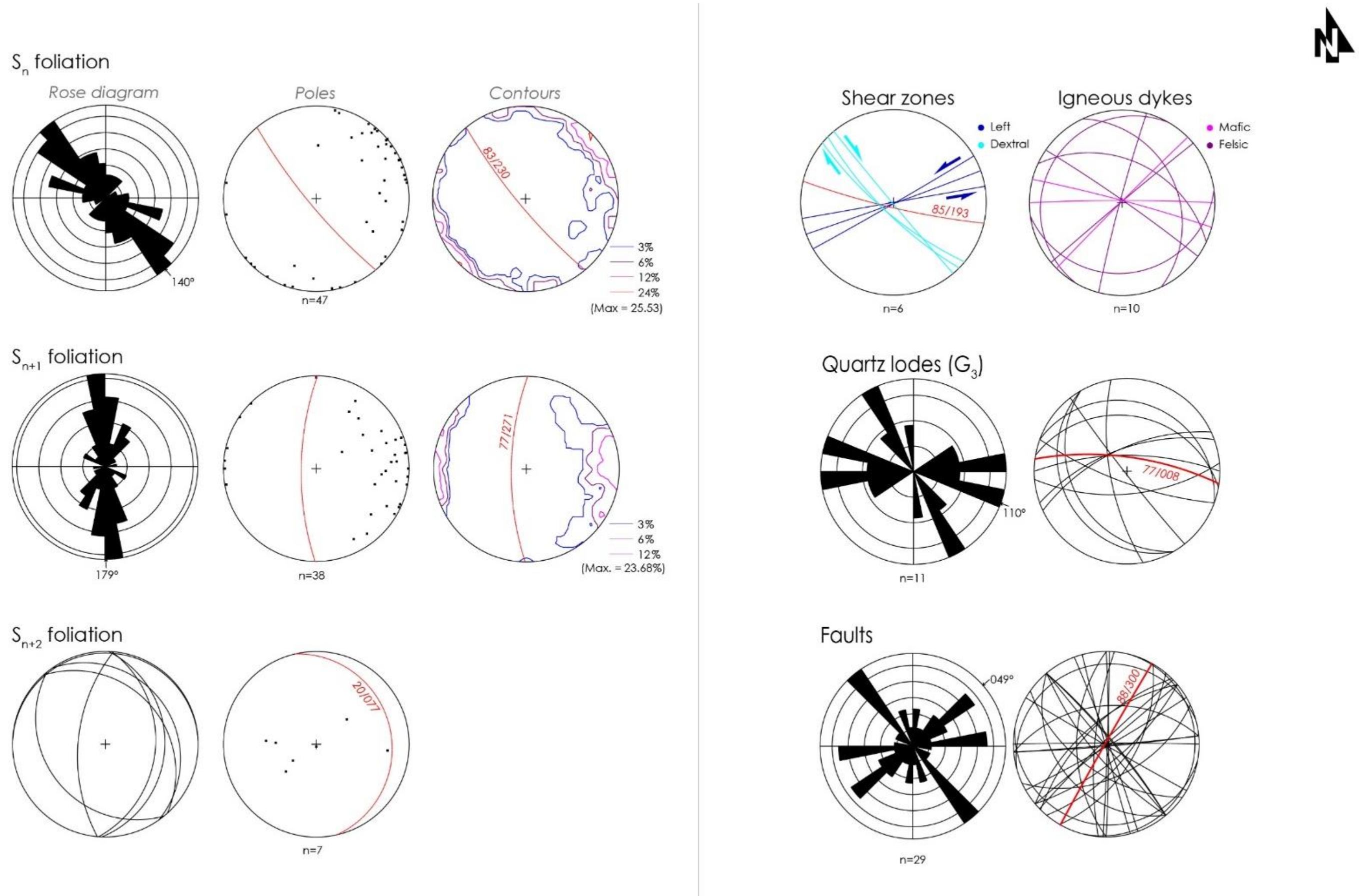


**Fig.1** – Geological map of the Argemela area (previous page) and summary of the most relevant structural elements (orientation and stereographic plots). The geological map indicates Pedra Alta and Cabeço de Argemela as “mine” and “quarry”, respectively.

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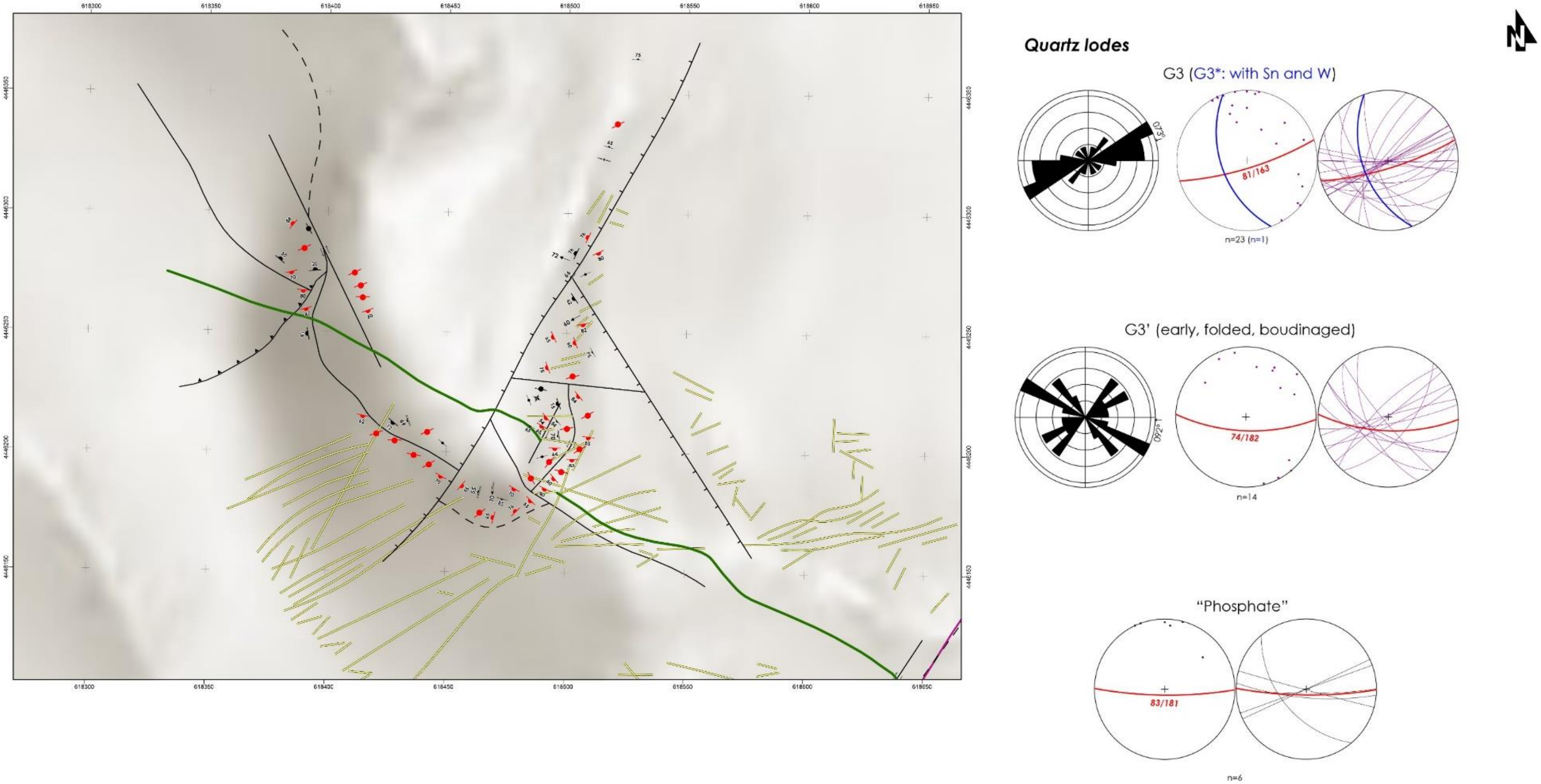


**Fig.2** – Prevalent quartz veins observed in the accessible exposures of Pedra Alta (left – in red, following the symbols used in Fig. 1) and summary of the most relevant structural elements (orientation and stereographic plots, to the right). The yellow lines in the map represent a compilation of quartz lodes data made by PANNN for the same area, using the information from old surveys. Note the strong consistency between both structural datasets. G3 = milky quartz veins.

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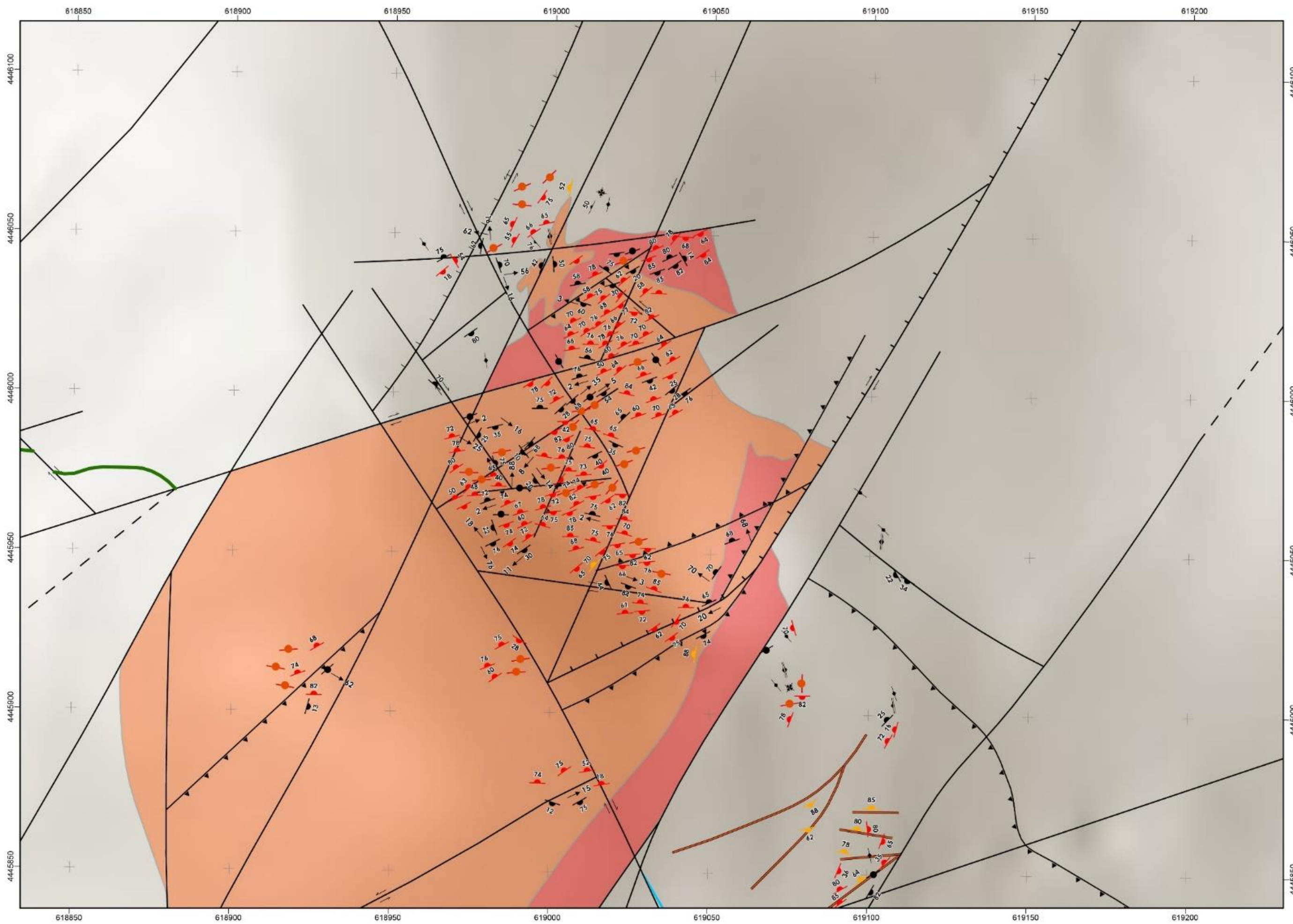


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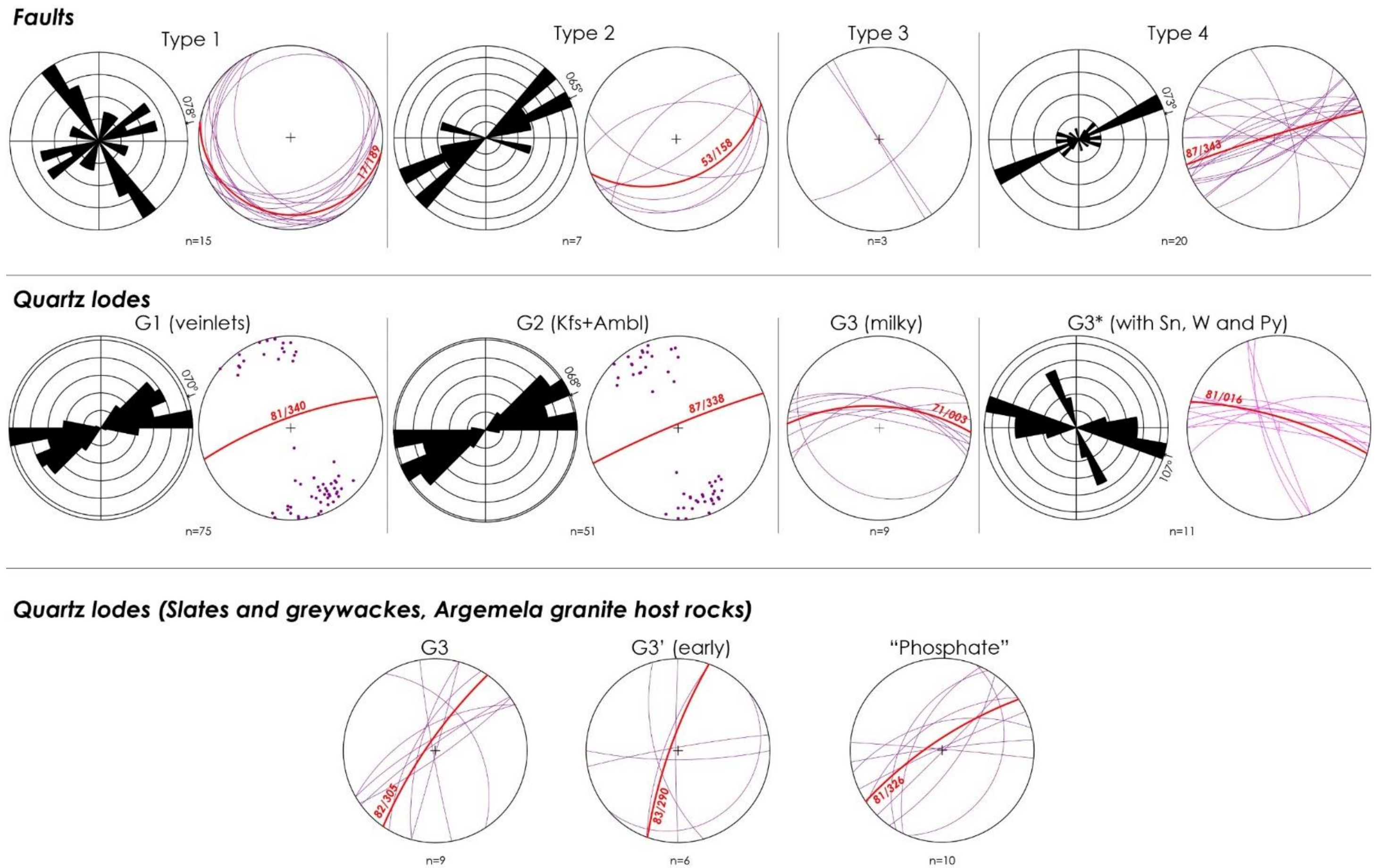


**Fig.3** – Geological map of the Cabeço de Argemela area (previous page) and summary of the most relevant structural elements (orientation and stereographic plots). Colours and symbols as in Fig.1.

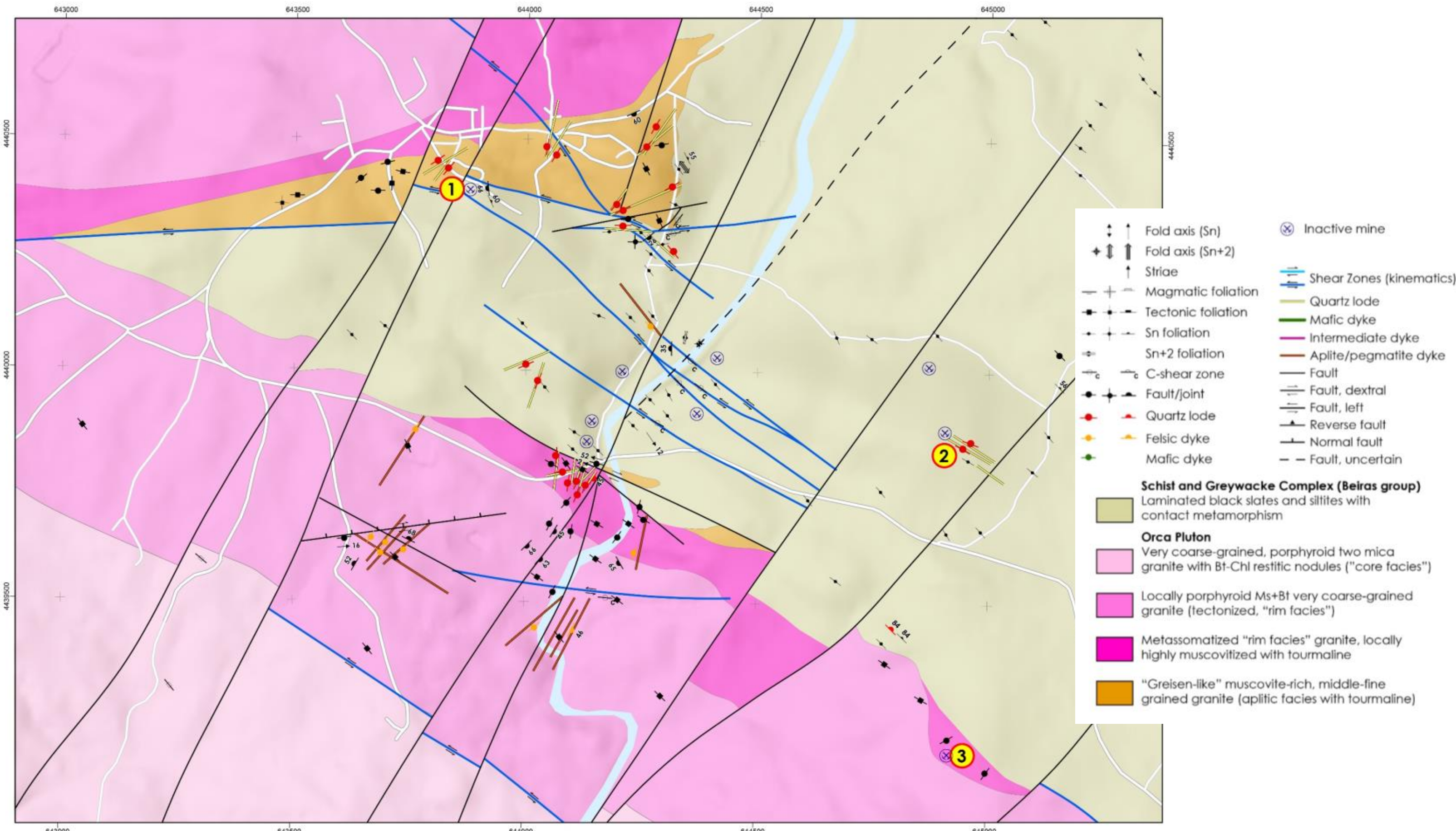


**Fig.4** – Geological map of the Mata da Rainha area. The numbers in the map indicate key sampling sites.

verifying if they are consistent with the compositional trends towards fertile systems; (2) the structural control of these vein and dyke arrays, possibly regulated by the intersection of syn-D3 shear zones (some of themalready recognized in the field); (3) the factors ruling the unusual tourmalinization of the granitic and metassedimentay host of mineralized quartz lodes; and (4) the composition of fluid inclusions potentially preserved in coarse-grained quartz infillings.

The **Medelim area** hosts an anomalously Sn-rich (ca. 400 ppm) coarse-grained, *ms*-rich granite that intrudes the border facies of the Penamacor-Monsanto granite body. The *ms*-rich granite includes a fine-grained leucogranite within which a dense swarm of aplite dykes was also delimited during geological mapping (1:1,000). The following work plan for the area will investigate the geochemical differences of all these (highly differentiated) granite facies and aplite dykes, supplementing the current database and verifying if they match the compositional trends towards fertile systems.

Fieldwork and detailed geological mapping (1:5.000) carried out in the **Segura area** was the basis for an extensive database on the swarms of Li-bearing aplite and pegmatite dykes, and a complete revision of the exposed granite facies, the metasedimentary units, and the contact metamorphic aureole associated with the Segura-Cabeza de Ayara pluton (**Fig. 5**). These data represent a fundamental background to support an integrative (multi-scale) interpretation of the geochemical and mineralogical information gathered in the mineralised systems encircling the granite batholith, and of the late transformations experienced by the spotted schists forming the contact metamorphic aureole. These issues will be addressed below when reporting results of WP3.

The refinement of alluvial cassiterite, wolframite and scheelite abundance maps for the Segura area showed that the concentration of cassiterite grains defines a concentric anomalous halo that surrounds the Segura granitic intrusion, encompassing its endo- and exo-contact (**Fig. 6**). The highest abundance of cassiterite grains is confined to the western (and northern) part of the halo (up to ≥ 5000 grains), overlapping the sites where W-Sn mineralised quartz veins were exploited in the past. The contribution of these sources to cassiterite in the alluvium deposits must have been more significant than the one potentially related to the minor W-Sn quartz veins and Li-Sn aplite-pegmatite bodies recognised in the eastern part of the anomalous halo. The anomaly of alluvial wolframite (up to < 300 grains) develops mainly to the west (and north) of the Segura intrusion border and its exo-contact, defining an N-S trend; in addition, the prevalence of wolframite over cassiterite grains tends to increase with distance from the Segura granite, following the metallogenic zoning proposed for the regional W-Sn mineralisation. Regarding alluvial scheelite, its higher abundance (up to >500 grains) defines a discreet concentric anomalous halo surrounding the exo-contact of the granitic intrusion, with a distal position relative to the cassiterite-enriched halo. In summary, the alluvial cassiterite, wolframite, and scheelite anomaly pattern halos can be correlated with distinct W-Sn metallogenetic events, interpreted to have different origins and control factors (e.g. structural, lithological, magmatic/hydrothermal, timing), conceivably superimposed mostly in the western part of the Segura area.

The purpose of **WP2** is the **vectoring of metal endowment and the definition of the critical timing for mineralisation triggering**. Complementing the whole-rock geochemical data reported in October 2021, an extensive multi-system (Pb-Sr-Nd) isotopic database was obtained for metasedimentary and granitic rocks. Numerical handling of this database is in progress, but preliminary results for samples representing the main metasedimentary units (Malpica do Tejo and Rosmaninhal *Fm*s.) confirm: (1) the inferences made based

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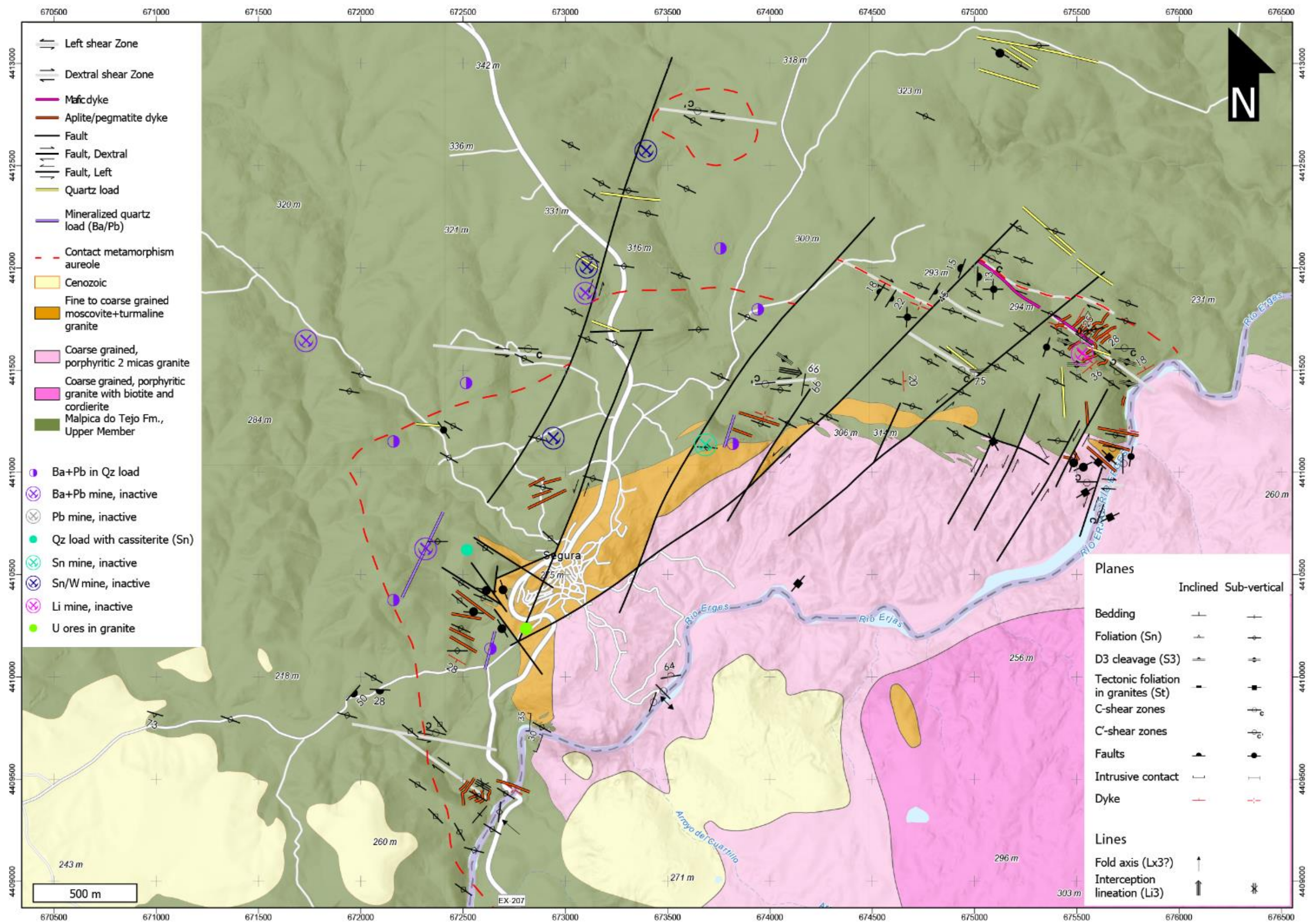


**Fig.5** – Geological map of the Segura area.

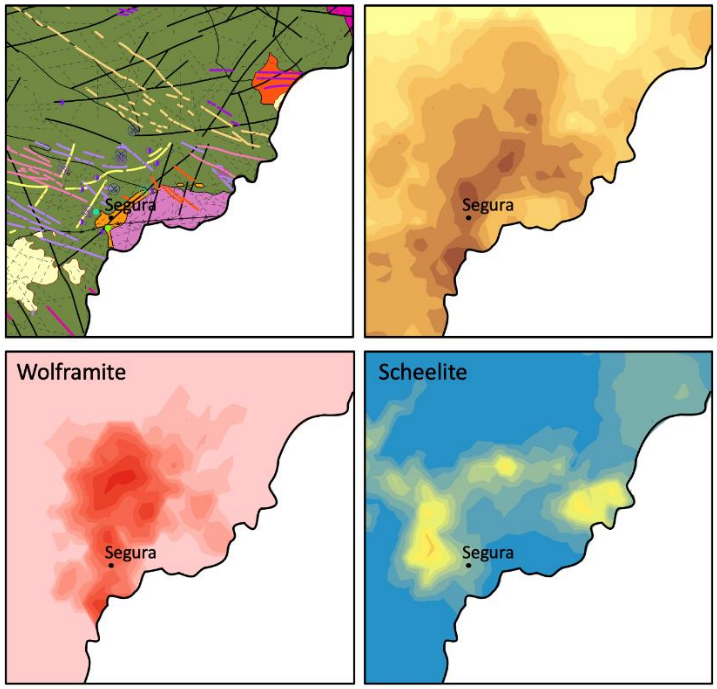


**Fig. 6** – Alluvial cassiterite, wolframite and scheelite abundance maps for the Segura area.

on multi-element geochemistry about the principal sources involved in the formation of the Beiras Group sediments; and (2) the usefulness of geochemical criteria to distinguish the Malpica do Tejo and Rosmaninhal *Fm*s., the latter more radiogenic, though isotopically closer to the upper member of the former formation. Implications to different metal endowments and the generation of granite melts variably enriched in Sn (Nb, Ta), Li (Cs, Rb) or W (as a function of melting temperatures, a relative abundance of fluxing elements and other factors) are presently under study (task 5.2, **WP5**).

Regarding the timing of mineralization events in the Góis-Segura strip, the SHRIMP U-Pb zircon data gathered so far indicate, as best estimation, the ca. 310-290 Ma time window, which includes the Concordia ages obtained for the pegmatite dykes of Monsanto (308±3 Ma), the Li-rich aplite/pegmatite lodes of Segura (308±5 Ma), the *ms*-rich granite of Segura (307.6±2.5 Ma), and the (drilled) two-mica granite of Panasqueira (304±5.8 Ma). This time window, consistent with other published geochronological information, also includes the Concordia ages provided by zircon grains extracted from the internal granite facies of the Orca pluton (304±3 Ma) and the porphyry laccoliths (306±4.6 Ma) and dykes (299.9±7.2 Ma) cropping out in the Zebreira area.

The main objective of **WP3** is to **investigate the relevant processes for metal concentration and deposition at the ore scale**. During the 1st year of MOSTMEG implementation, Segura and Argemela were selected as the main targets to initiate the studies planned for **tasks 3.1** (***Rare metal enrichments in (aplite-pegmatite systems***) and **3.2** (***High-grade, (magmatic-)hydrothermal ore systems***). The following paragraphs summarise the advancements and the results obtained after October 2021.

Aiming the characterisation of mineral phases forming the quartz lodes at Pedra Alta (Permit nº 1, **Argemela**) and of the fluids involved in the Li-Sn mineralising processes, detailed petrography and mineralogical inspection of the lodes were performed together with a microthermometry and Raman micro-spectrometry study of fluid inclusions (FI) in quartz, amblygonite and cassiterite.

The studied samples are characterized by massive amblygonite white to blue/greenish, in well-developed crystals, mostly centimetres in size. Due to the dimension of amblygonite crystals and the size of the studied samples, it was impossible to correctly assess the chronological relationship between amblygonite and other mineral phases (**Fig. 7**). Anhedral to subhedral quartz crystals with variable size and wavy extinction, some of them displaying comb-textural arrangements (QZI), were also recognised, besides the mosaic-textured quartz aggregates (QZII) (**Fig. 8**).

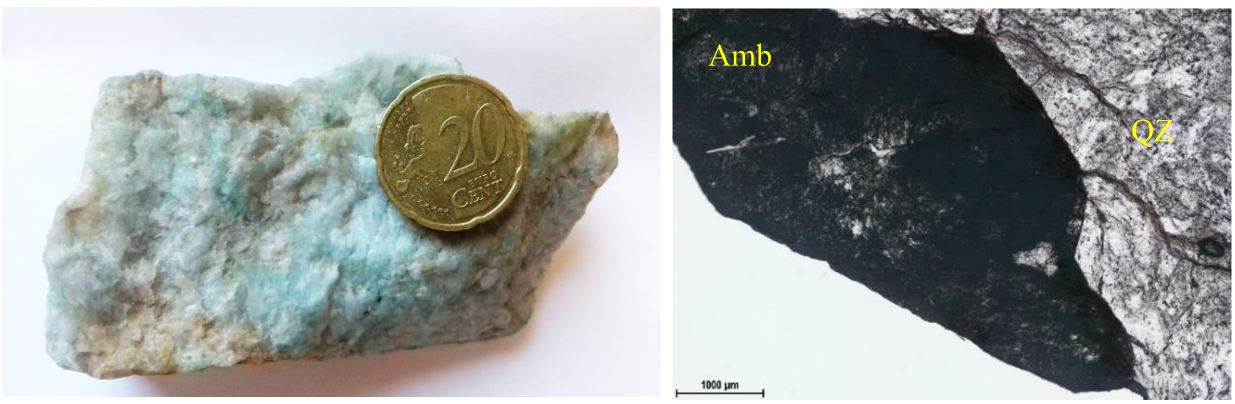


**Fig. 7** – Massive amblygonite (left) and photomicrograph of amblygonite (Amb) and quartz (QZ) (right) from one of the studied samples from Pedra Alta.

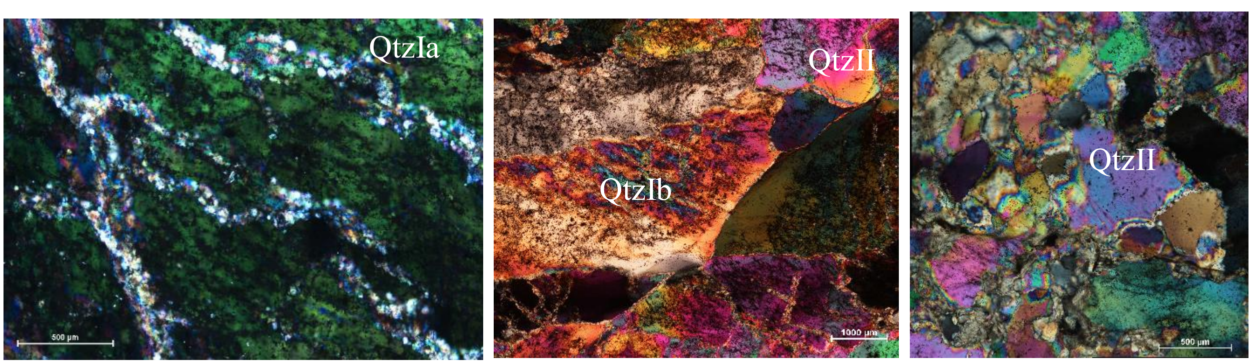


**Fig. 8** – Quartz types forming the Pedra Alta lodes.

In the Pedra Alta lodes, cassiterite, following quartz deposition, is anhedral to euhedral and generally zoned (**Fig. 9**). No Ta was detected, and Nb contents are low (up to 2.07 wt. %). The identified columbo-tantalite phases are enclosed in cassiterite grains and show some compositional zoning, reflecting the variation of the Ta/Nb ratio (**Fig. 10**). Additionally, some minor mineral phases of the Zr-Hf series were also found (**Fig. 11**). Native bismuth, bismuthinite and silver-bearing sulphosalts occur in the quartz lodes and fill cracks in cassiterite (**Figs. 12** and **13**). These mineral phases should have formed after the development of Sn-bearing phases since they also occur enclosed in stannite (**Fig. 13**). Chalcopyrite is also observed and formed later than stannite. Other mineral phases in the Pedra Alta quartz lodes were described by Inverno et al. (2019).

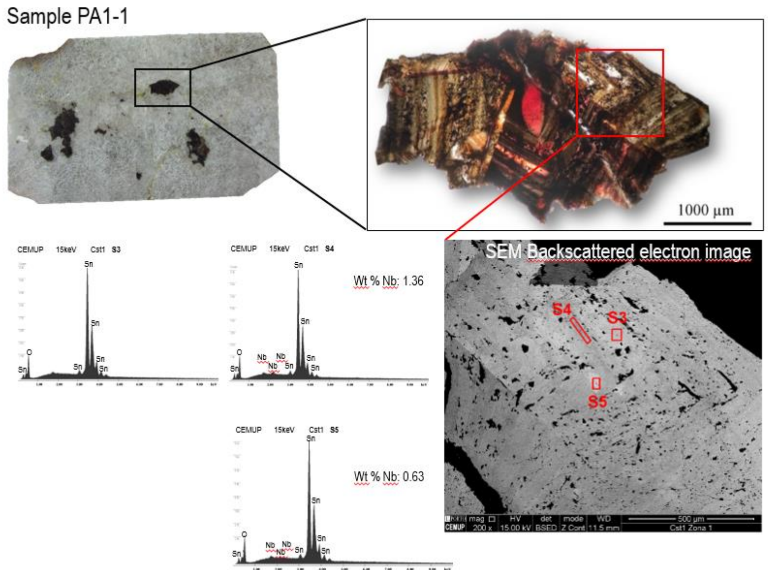


**Fig. 9** –SEM backscattered electron image with EDS of cassiterite with variable Ta/Nb ratio along the crystal zoning.

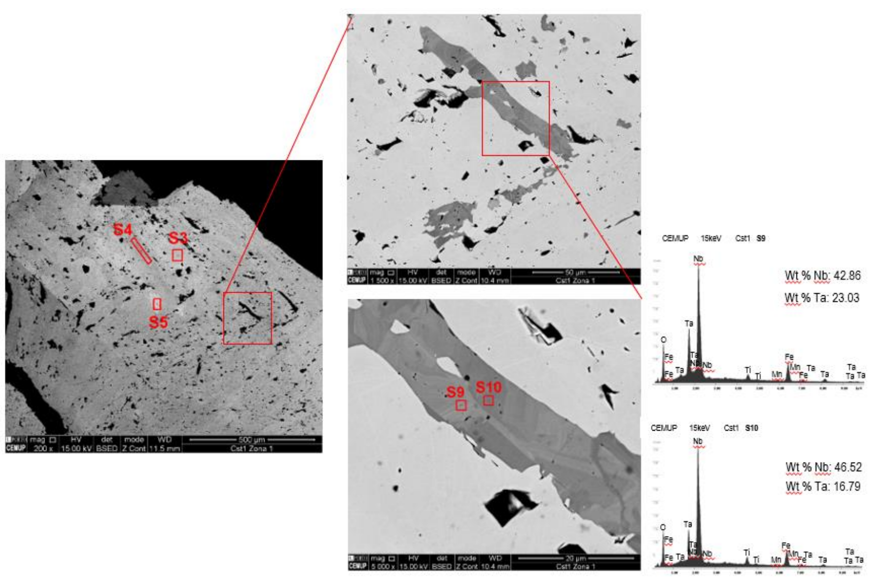


**Fig. 10** – SEM backscattered electron image with EDS of columbo-tantalite phases enclosed in cassiterite and variable Ta/Nb ration along the crystal zoning.

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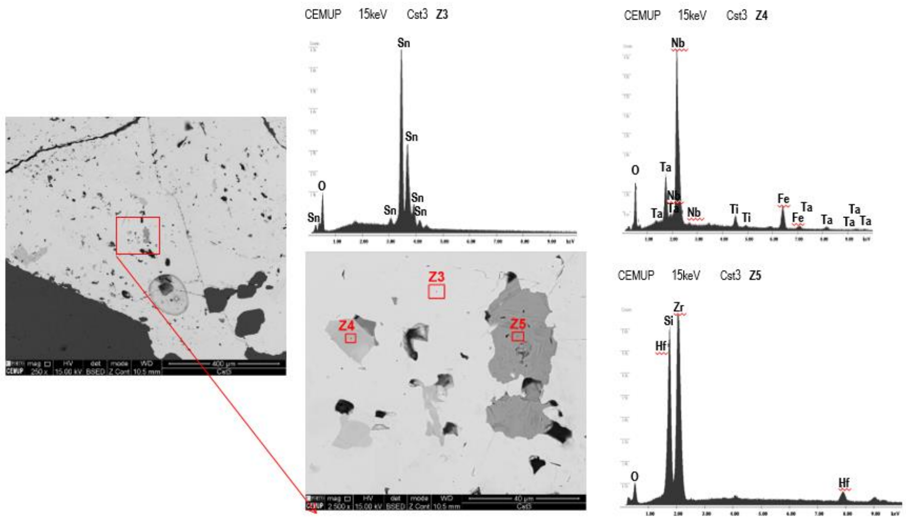


**Fig. 11** – SEM backscattered electron image with EDS of Nb-Ta (Z4) and Zr-Hf (Z5) mineral phases within cassiterite (Z3).

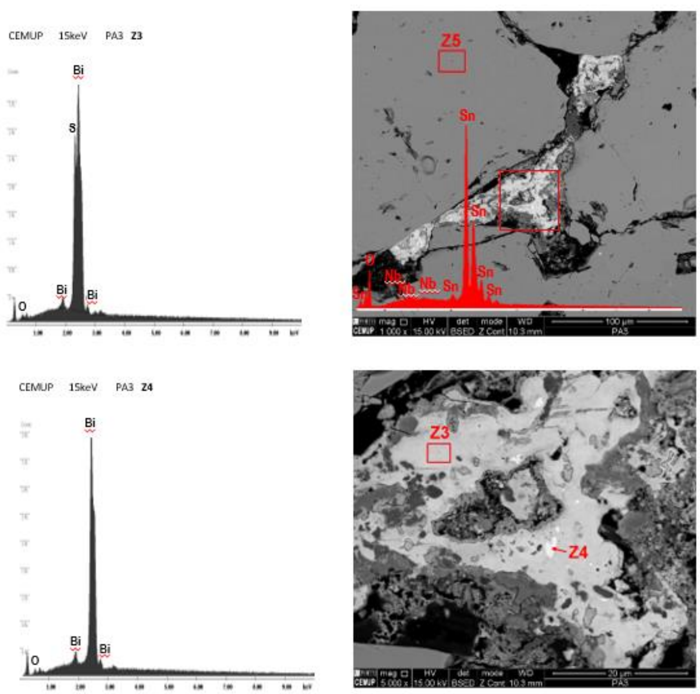


**Fig. 12** – SEM backscattered electron image of bismuthinite (Z3) and native Bi (Z4) filling cracks of cassiterite (Z5).

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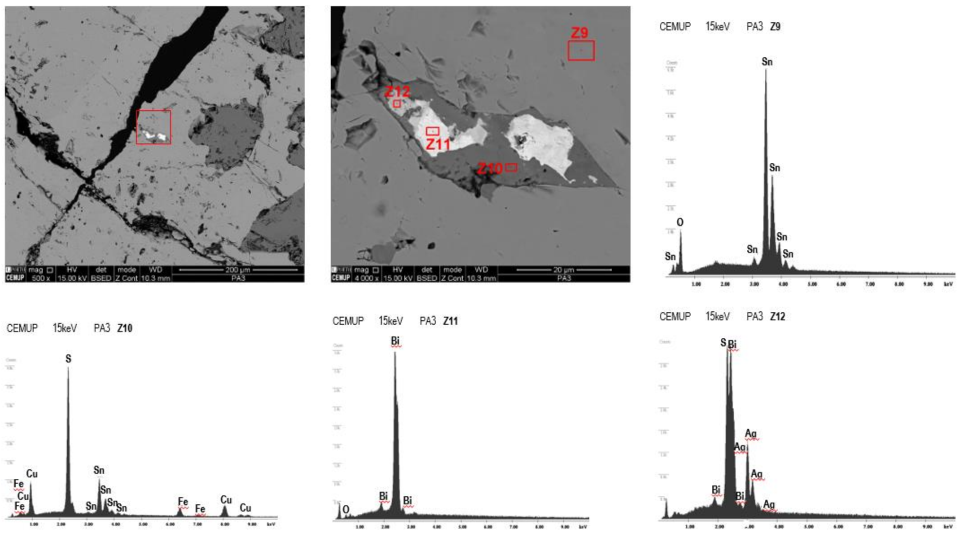


**Fig. 13** – SEM backscattered electron image with EDS of stannite (Z10), native bismuth (Z11) and silver-bearing sulphosalts (Z12) identified in crack-infillings affecting cassiterite (Z9).

According to the observed characteristics and following the classification scheme of Boiron et al. (1992), several types of fluid inclusions (FI) were identified in the examined samples (**Fig. 14**):

- Lw-c, Lc-w, in amblygonite, as isolated FI or clusters of FI with rounded and negative crystal shapes, and 5 to 15 µm in size, displaying a degree of filling (Flw) between 0.55 and 0.7.
- Lw-c, Lc-w, in QZII, as isolated FI or clusters of FI with rounded and negative crystal shapes, and 10 to 20 µm in size, showing an Flw between 0.50 and 0.60.
- Lw-c, Lc-w and Lw-m, in cassiterite, as isolated and negative crystal shapes, distributed along the growth zone, varying in size from 5 to 20 µm and in Flw from 0.70 to 0.75; or as isolated FI displaying similar ranges in dimension and Flw between 0.60 and 0.70. Trails of pseudo-secondary FI with 5-10 µm sizes are also common.

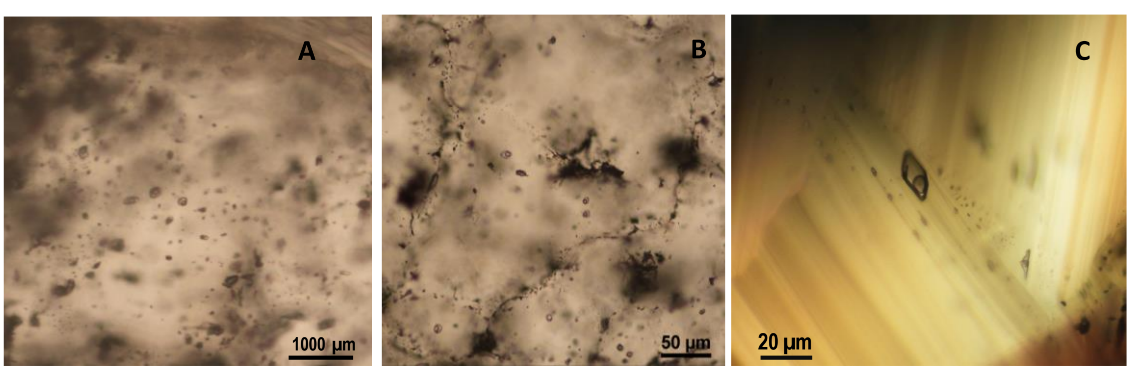


**Fig. 14** – Fluid inclusions in amblygonite (A), quartz II (B) and cassiterite (C) from the studied quartz lodes.

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The obtained microthermometry results for the studied FI are summarized in **Table 1**.

**Table 1 –** Microthermometric data for different FI types in Pedra Alta lode samples.

| Mineral | FI type | TmI (º C) | TmCl (º C) | $TmCO_2$ | $ThCO_2$ | TH |
|---|---|---|---|---|---|---|
| Cst | Lw-c | -2.7/-1.1 | 5.6/15.1 | | | 234/310 (L) |
| | Lw-m | -2.9/-1.3 | 4.1/16.8 | | | 240/329 (L) |
| | Lc-w | -2.9/-1.6 | 11.1/14.3 | -65.3/-62.9 | 2.3/3.8 (C) | 293/329 (C) |
| Qz | Lw-c | -2.2/-1.1 | 9.4/16.4 | | | 311/333 (L) |
| | Lc-w | -3/-1.2 | 3.8/14.2 | -61.7/-60.2 | -10/11.2 (V) | 313/357 (C) |
| Amb | Lw-c | -3.7/-2.1 | 6.8/10.3 | | | 280/290 (L) |
| | Lc-w | -3.5/-2.1 | 9.3/10.3 | -59.6/-58.5 | 16.4/21.5 (L) | 289/315 (L) |

$TmCO_2$: $CO_2$ melting temperature; TmI: ice melting temperature; TmCl: melting temperature of clathrate; $ThCO_2$: $CO_2$ homogenisation temperature; TH: total homogenisation. L: liquid; V: vapour.

The obtained bulk and volatile phase compositions (**Fig. 15**) show that water is the main component of FIs trapped in different mineral phases from Pedra Alta lodes; the $CO_2$, $CH_4$, $N_2$ and salt components are always minor. Nonetheless, fluids with a $CO_2$ -dominated volatile phase were trapped in amblygonite and quartz, deviating from fluids trapped in cassiterite which show variations from $CO_2$-dominated to $CH_4$-dominated. All the fluids have a low $N_2$ content in their volatile phase.

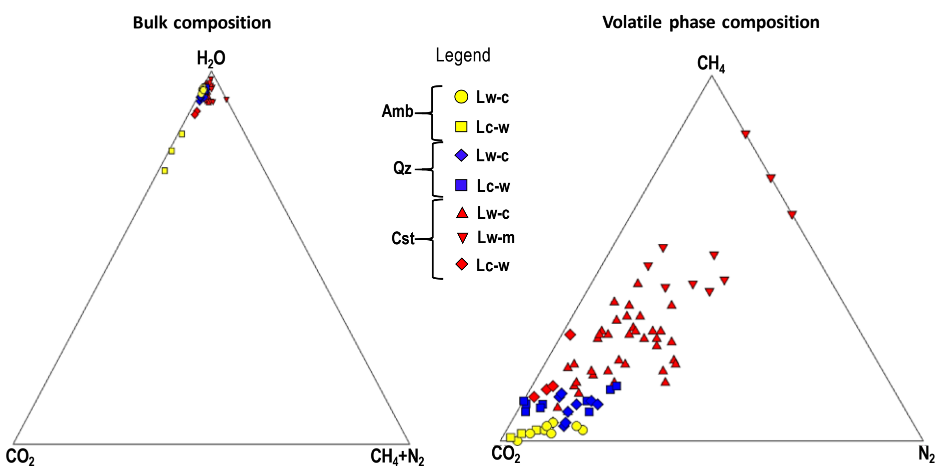


**Fig. 15** – Ternary plot in the $H_2O$–$CO_2$–($CH_4$+$N_2$) diagram of bulk composition and in $CO_2$–$CH_4$–$N_2$ diagram of the volatile phase composition for the different FI studied in amblygonite, quartz and cassiterite.

A series of representative aplite and pegmatite dykes from the **Segura area** (close to the Segura granite contact but hosted in metasedimentary rocks) were sampled and analysed. The new chemical analyses of granite (SEG Inner facies), aplites and pegmatites (Segura dykes) were compared to those from previous works (Antunes et al.,2013) on the same zone and similar intrusions elsewhere, such as the Argemela granite (Michaud et al., 2020) and the Panasqueira granite facies (Marignac et al. (2020). The available data show that aplite and pegmatite dykes from Segura are very close to Argemela granites and more albitic than the samples described by Antunes et al. (2013). The enrichment in $Na_2O$ depicts the high degree of differentiation and the albitic feature of the evolved magmas (**Fig. 16a**).

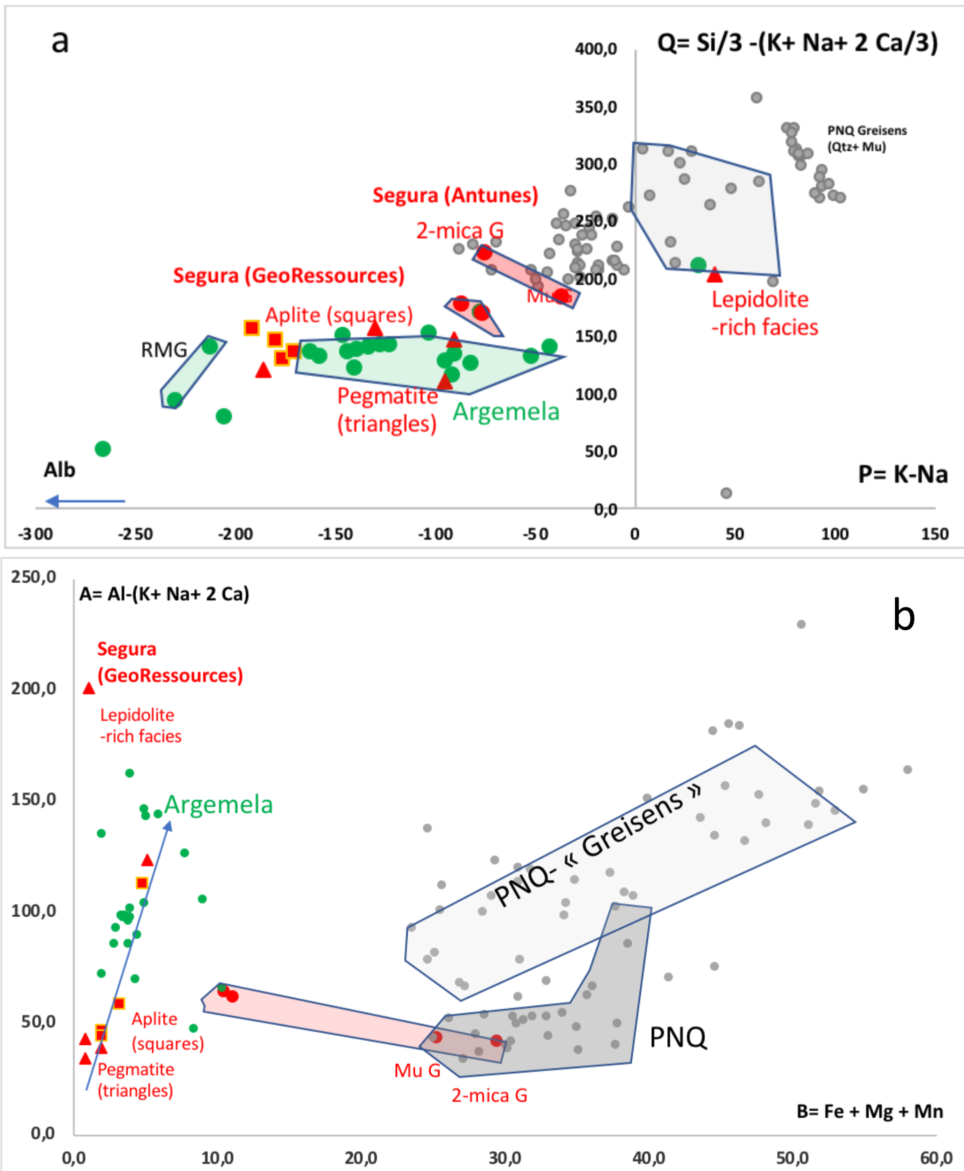


**Fig. 16 –** Major element geochemistry of the new pegmatite and aplite samples from Segura compared to the former data from Antunes et al. (2013) on the same region and data on other differentiated granites as the Panasqueira (PNQ) and Argemela granite suites (Michaud et al., 2020; Marignac et al.. 2020). **a**: Q = Si/3 – (K+ Na= 2Ca/ 3), P= K-Na+

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Ca, showing the enrichment in albite at decreasing amounts of quartz: the lepidolite facies in an aplite from Segura (northern dykes) is a quartz + lepidolite association similar to a greisen., **b**: *A-B* diagram from Debon and Lefort showing the decrease of the parameter B as a function of A as a marker of the differentiation. Data from Antunes are red dots, and newly analysed samples from Segura are in red triangles (pegmatites) and squares (aplites). Based on literature data, the possible magmatic differentiation trend of the aplite and pegmatite is distinct from the inner granite to pegmatite trend. It is almost superimposed on the Argemela trend.

In the Segura aplite and pegmatite dykes, mafic components are low to very low, and the *B* parameter is lower than 6, close to the values of Argemela. All the rocks are magnesium poor and not so peraluminous, as seen in the Al-(K+Na+2Ca) vs Fe+Mg+Ti diagram of Debon and Lefort, where most aplites display *A* values lower than 60 (**Fig. 16b**) due to the prevalence of feldspars. In reverse, the lepidolite rock facies has a very high *A* parameter, around 200, and may correspond to a late conversion of feldspars into Li-rich micas, similar to a greisenisation process. At Panasqueira, the peraluminous parameter Al-(K+Na+2Ca) is, however, increased by alteration effects of the greisen but conversely is correlated to an increase of the *B* parameter as a consequence of Fe-rich micas crystallisation along with micro-sulphides (Marignac et al., 2020).

The Segura aplite and pegmatite dykes are significantly enriched in phosphorous, with $P_2O_5$ contents in the 1.7 to 2.7 wt% range, higher than the previous data. Compared to the Panasqueira granites, which are dominated by a strong correlation between phosphorous and calcium content, meaning that the two elements are both entirely contained in apatite, the Segura aplite-pegmatite veins are characterised by an excess of phosphorus, not linked to apatite, but amblygonite-montebrasite series, and to a Fe-Mn rich phosphate (see the report from the first year).

The characterisation of the Sn, Nb-Ta and W-bearing phases in these dykes has been completed. In the lepidolite facies, cassiterite enriched in Nb, Ta and W was found, as shown in **Fig. 17**. Regarding the Nb-Ta and Sn oxides in the aplite-pegmatites, the available data show that:

(1) **Nb-Ta oxides** do not form only exsolution grains in or from cassiterite, as Antunes et al. (2013) reported. In samples from the northern zone, euhedral elongated grains of Nb-phases are a few 10 µm to 100 µm and more in length and display BSE imaging spectacular zoning, mostly oscillatory zoning (OZ) (**Fig. 17a, c, d, e, f**). Acicular Nb-phases contain a significant amount of W (4 to 5 % $WO_3$) and Mn (10-13%) and are characterised by oscillations in Ta from 10 to 20% (**Fig. 17e, f**). Overgrowths of Ta-Nb-phases with significant oscillation of Nb-Ta formed then on the initial core rich in Nb. In the southern zone, elongated acicular Nb-Ta phases with zonation have also been found (SEG2).

(2) **Cassiterite** is generally euhedral and crystallised onto the thin prisms of Nb-Ta phases, confirming earlier observations (**Fig. 17a, b, d**). Although the previous report indicated poor compositional zonings due to a low substitution level, new samples show very complex textures with significant substitution, particularly in the samples from the lepidolite facies. Cassiterite may contain up to 8% $Ta_2O_5$, and 1% $WO_3$. Cassiterite contains tiny inclusions of Ta-(Nb) phases (tantalite with 60% $Ta_2O_5$) and variable $SnO_2$ content up to 12%. It is unclear whether these inclusions could be considered exsolutions. They appear primarily as inclusions of the latest growing zones of the complex geometry of the Ta phase overgrowths.

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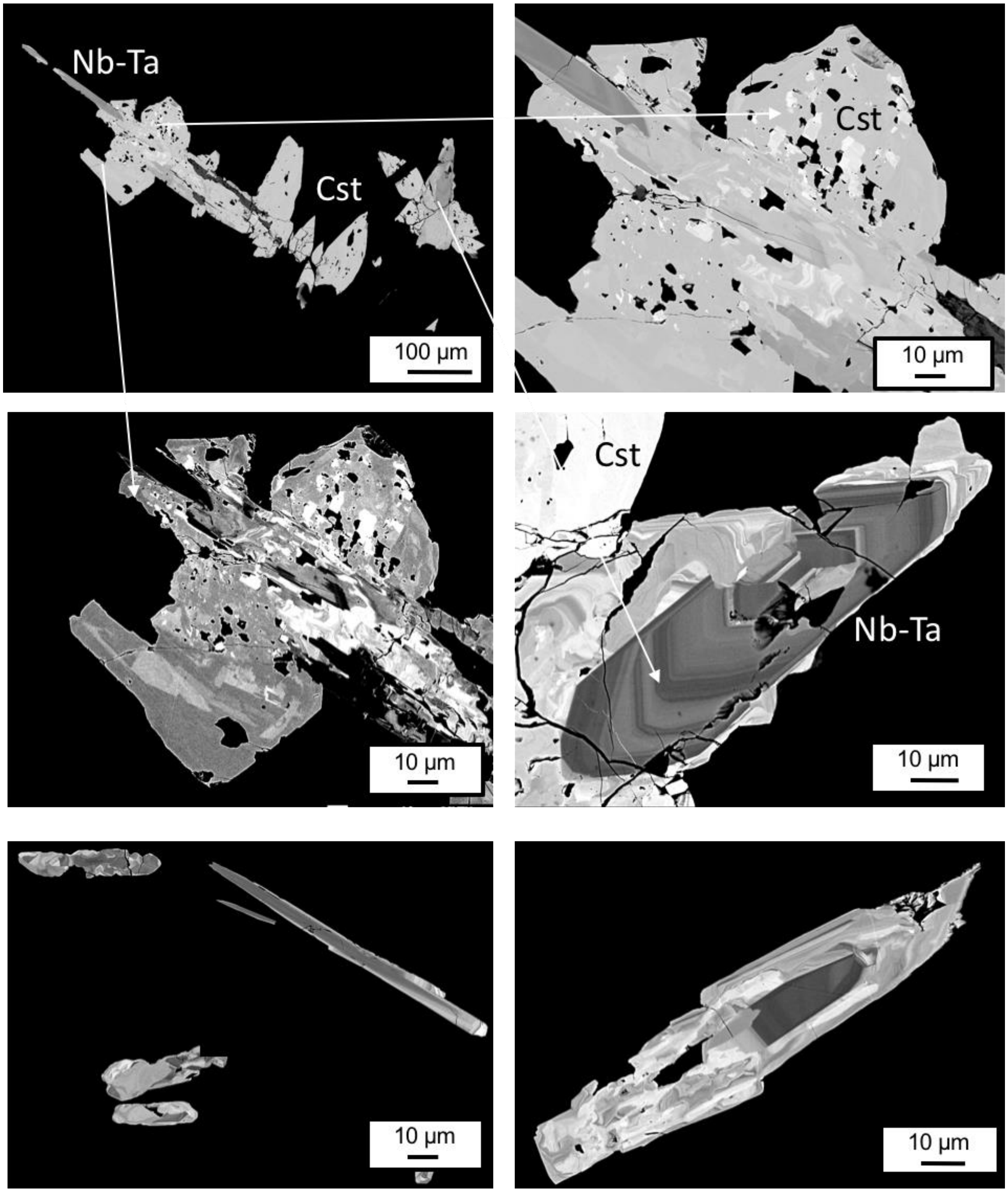


**Fig. 17 –** Sn and Nb-Ta phases in Segura aplite and pegmatites. SEG-4-(5) (sept21) **a**: euhedral cassiterite grains crystallised onto Nb-Ta acicular crystals; **b**: detail of the cassiterite; **c**: detail of the Nb-Ta phase; **d**: relationships between the two minerals; **e, f**: other euhedral acicular Nb-Ta phases.

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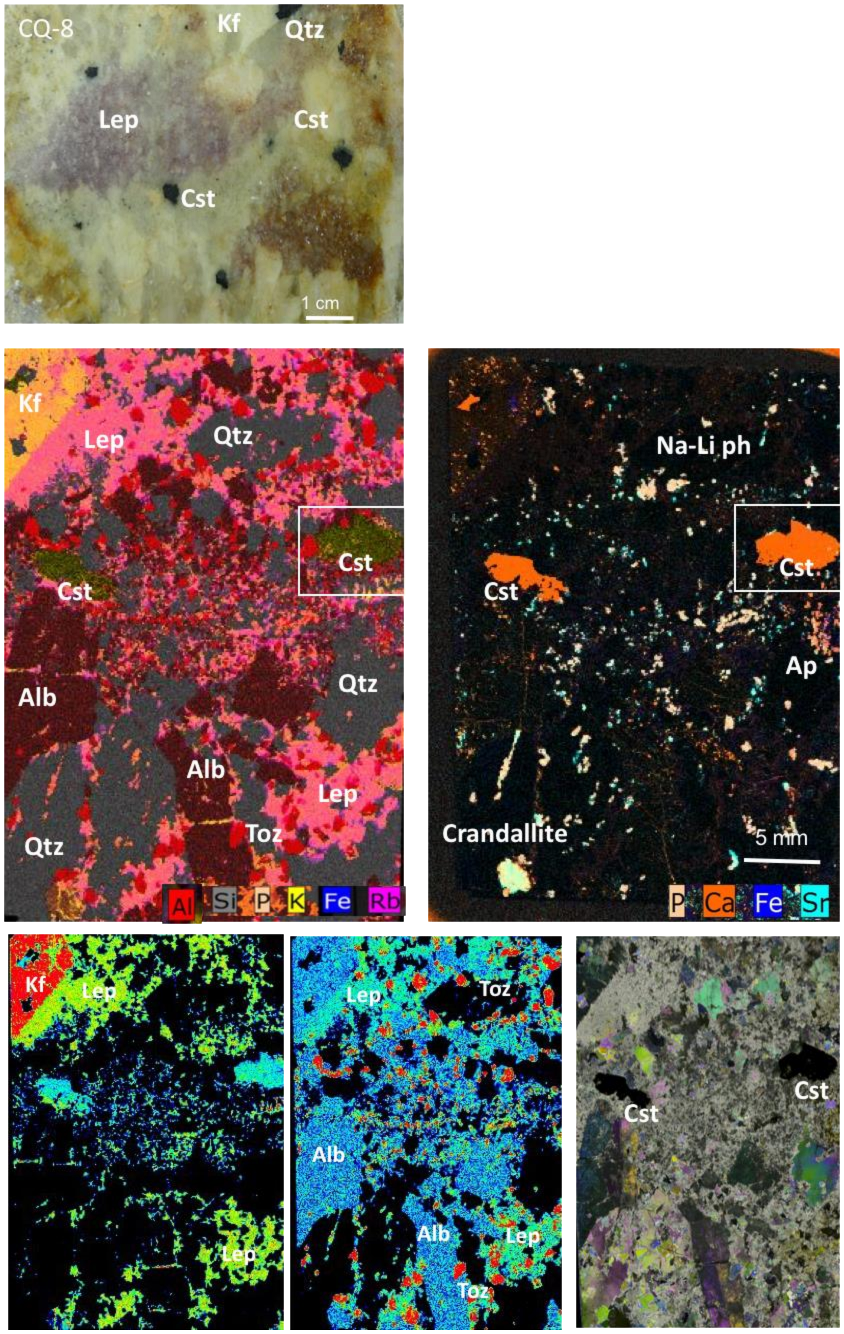


**Fig. 18 –** Cassiterite in the Segura aplite sampled in September 2021 (CQ8) euhedral cassiterite grains crystallised in the lepidolite-rich facies and micro-XRF maps of the corresponding thin section showing the abundance of lepidolite and topaz around the cassiterite grains. Phosphates such as apatite, amblygonite and crandallite are also dispersed in the rock.

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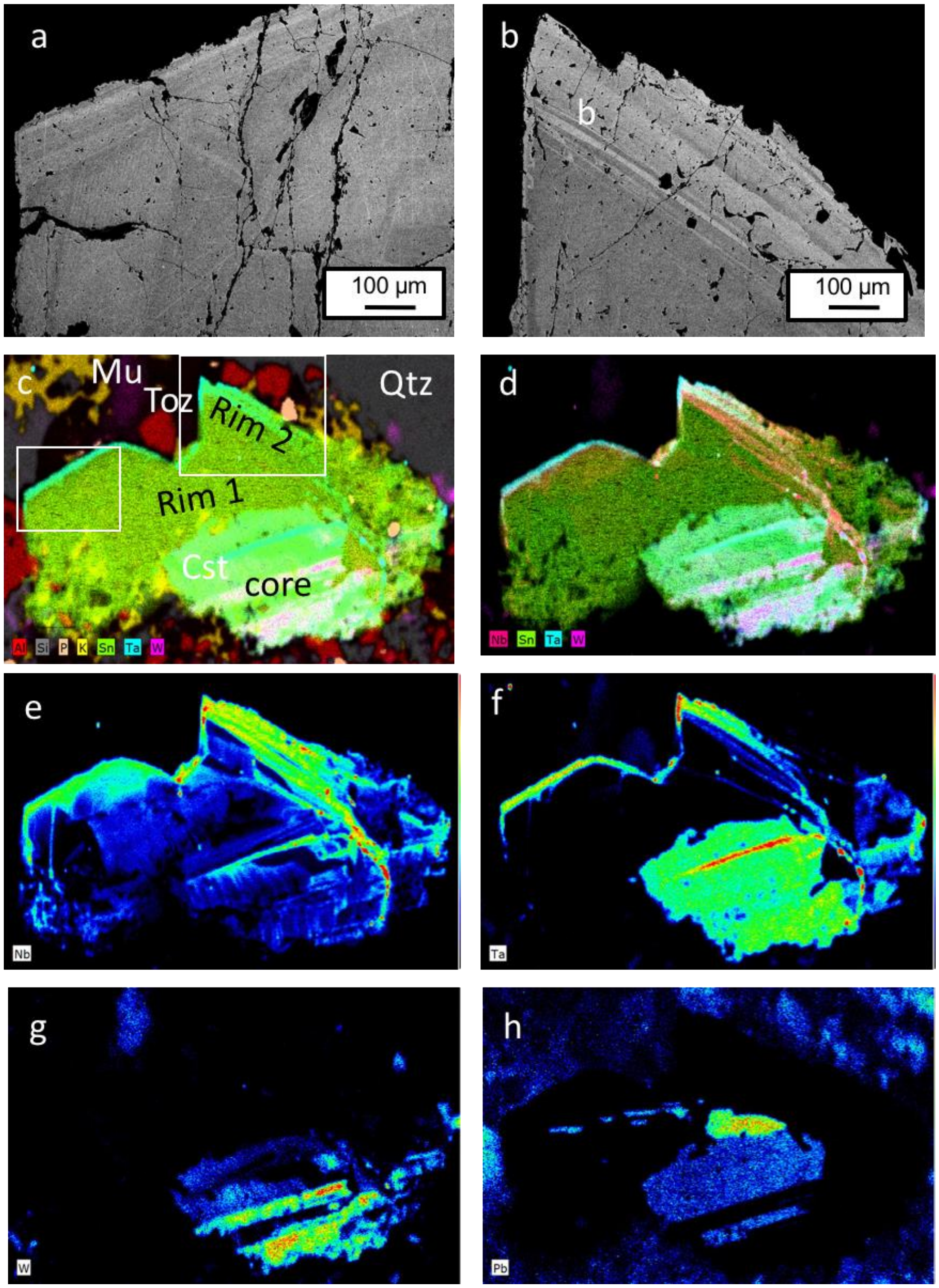


**Fig. 19 –** Cassiterite in the Segura aplite sample CQ8 (cassiterite at the right-hand side from Fig. 3). Details of euhedral cassiterite grains (BSE images) showing the external growth bands. The location of the two images, a and b, are indicated in squares from c. c: composite chemical map showing the core and two rims of the cassiterite, d: same image with Nb-Ta, Sn, W, and e,f,g,h, are the individual chemical maps (Nb, Ta, W, Pb) with scales of signal intensity.

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The general trend during the crystallisation of pegmatite dykes from Segura is the formation of Nb phases, followed by mineral phases increasingly rich in Ta. Cassiterite then formed as euhedral crystals onto the complex assemblages. The large cassiterite grains found in the lepidolite (**Fig. 18**) are also characterised by complex zonations, although the level of substitutions is less than in SEG4. One crystal from sample CQ8 (lepidolite facies) has a core with some substitutions W (up to 1% $WO_3$), Nb (0.3-0;4 $Nb_2O_5$) and Ta (0,7 $Ta_2O_5$). After, an overgrowth with poor substitutions is finally followed by a late overgrowth with increased content in Nb and Ta, and oscillatory zoning with variable Ta content from 1 to 4 % $Ta_2O_5$ (**Fig.19**). In the lepidolite sample, topaz is very abundant, as shown by the Al map (red dots; **Fig. 18**).

Trace elements in muscovite could be used as an index of granite fertility. So far, micas have been comprehensively studied in magmatic series from the Segura area and compared with other muscovite-rich facies (Medelim, Penamacor-Monsanto granite body). Muscovites in granites are frequently zoned, such as those from the muscovite-rich granite from Segura pluton or Medelim or Monsanto granites (**Fig.20**). There are often cores and a surrounding rim which is particularly enriched in Li, Rb, Cs, Sn indicative of the existence of fluids (or magmas) enriched in incompatible elements at the end of the crystallization of such facies. Similar muscovite-rich facies from Medelim and Monsanto present micas with similar compositional zonings and elemental enrichments.

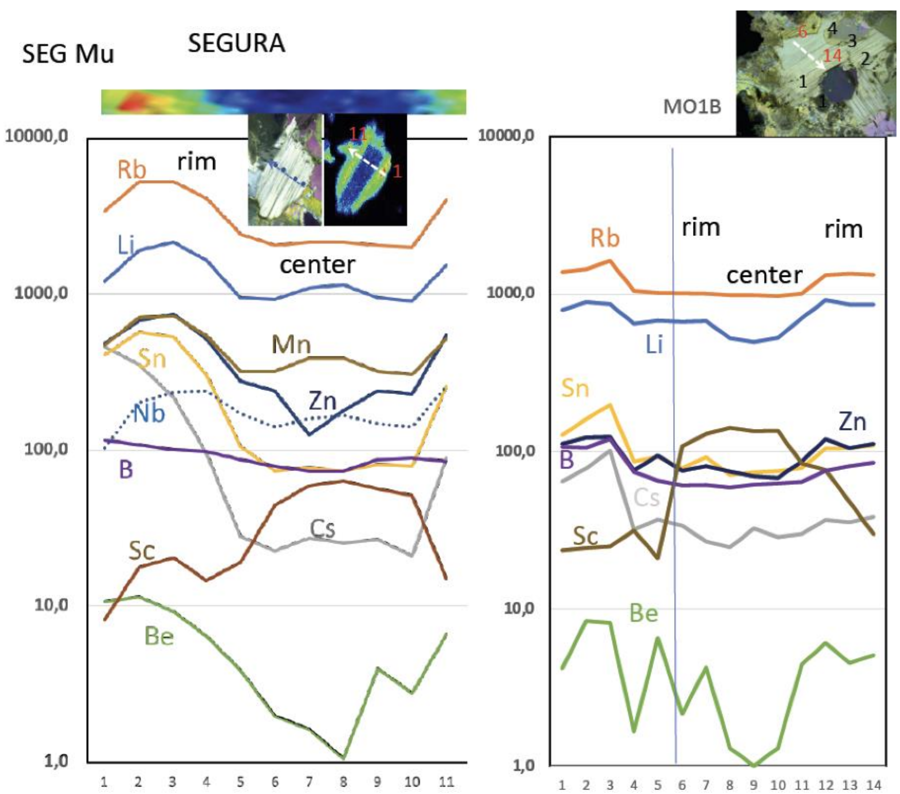


**Fig. 20 –** Muscovites from Segura muscovite-granite, and from Monsanto. Profiles, made from the crystal rim to its core, show the enrichment in Pb, Li, Sn, Mn, Be and Zn in the external rim.

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This study aims to compare micas in different magmatic suites and discuss the relative enrichment in trace elements (such as Sn, Li and Rb) as a function of the bulk whole-rock geochemistry and other features (age, isotopic data).

Another research line currently explored is the imprint of contact metamorphic aureoles by hydrothermal processes. In the Segura area, the micaschists are spotted within a distance of around 500m to a few kilometres from the granite boundary. It is generally admitted that spots from spotted schists are constituted of cordierite and develop in particular in the slate bands (aluminous sediments made initially of clays) and much less in the sandy or silty bands. There are, however, very few available descriptions of preserved cordierite, which are generally entirely retrograded into chlorite or other phyllosilicates.

Thick sections were examined using reflected/transmitted light and scanning electron microscopy (TESCAN VEGA3 and JEOL J7600F, providing backscattered electron mode images at an acceleration voltage of 20 kV) to characterise mineral assemblages and parageneses. Semi-quantitative analyses were carried out on microsites identified from optical and scanning electron microscopy, with analytical conditions identical to those used for EPMA analysis. Chemical micro-XRF maps were acquired using the M4 TORNADO analyser with beam conditions of 50 kV and 200 mA, 25 µm spatial resolution and 12 ms/pixel dwell time. The obtained results show that:

(1) Spots, presumably cordierite nodules linked to contact metamorphism, are presently replaced by well-crystallised mineral assemblages ("pinite") related to retrograding processes. The relatively preserved samples have nodules where biotite and albite form individual crystals and are cemented by a Fe-Mg phase retromorphosed into chlorite. The replacement of cordierite occurred then, probably above 400°C, as the biotite-albite assemblage is predominant.

(2) Tourmaline appears within the matrix in highly hydrothermal zones, and quartz is more abundant than in the spots (**Fig. 21**). In hydrothermal zones, or zones close to late dyke intrusions, the rock matrix may be entirely replaced by muscovite (yellow colour in the composite chemical maps of **Fig. 21**). The spot itself could be partially replaced by muscovite.

So, if spots may be considered related to the proximity of intrusions, the muscovite or tourmaline replacements may be regarded as a proxy to (significant) hydrothermal fluid flow.

A new series of samples was collected along two profiles from the Segura granite to the unspotted metamorphic rocks. Samples were first investigated using microscopy and micro-XRF. A series of representative schists (12 samples) was impregnated (epoxy resin), and thick sections were manufactured. The map of the micro-XRF images on the twelve samples is given in **Fig. 22**. Powders of these sample series were sent to SARM in April 2022 for whole rock analyses. The whole-rock geochemistry of the same samples is awaited due to delays from the SARM laboratory in Nancy after technical problems during this summer.

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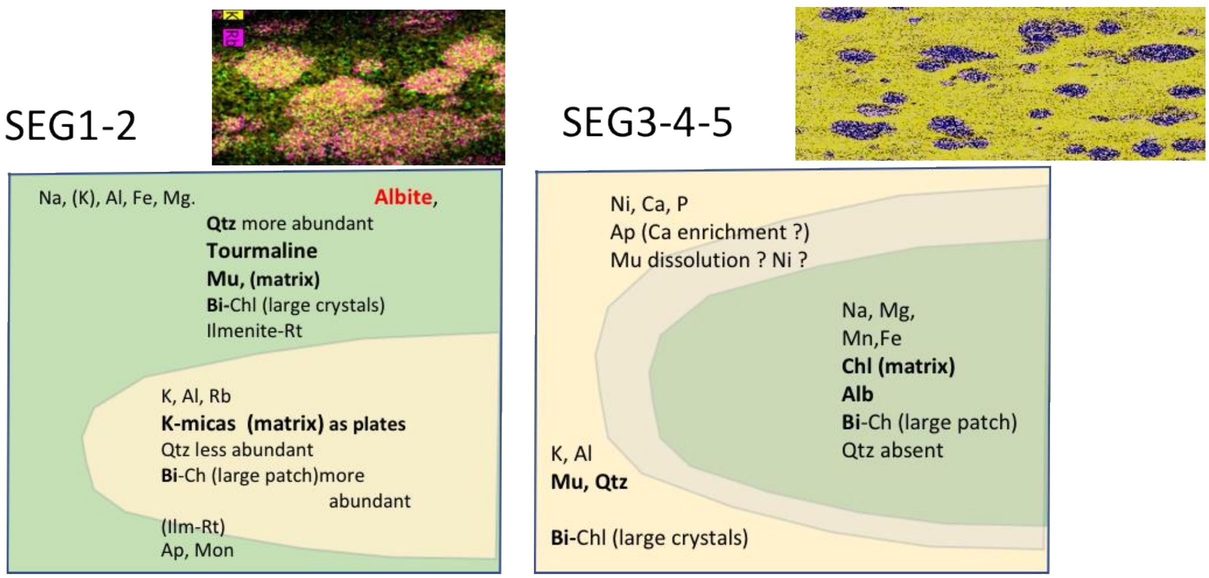


**Fig. 21 (next page) –** Micro-XRF maps of the spotted schists in the vicinity of the Segura granite and aplite dykes. The matrix may be entirely "muscovitised", as in the sample on the right-hand side, taken close to the northern dykes from the Colina Queimada. Alternately, only spots may be replaced by muscovite, as in the schist from the southern part of the Segura area at the vicinity of the dykes. The conceptual drawings below summarise the mineralogical observations made in the matrix and nodules.

Activities planned for WP3 also include the characterization of fluid inclusions in metasedimentary rocks and subsequent geothermometric approaches. A series of representative quartz veins (highly deformed quartz affected by Dn and Dn+1 folding, quartz in the foliation planes, veins crosscutting the $S_n$ and $S_{n+1}$ foliations) present in the mica-schists were prepared for fluid inclusions studies. The objective was to get data on the regional fluids trapped in the available quartz, whatever the early history of the quartz, as quartz could be micro-fractured and healed during brittle episodes synchronous with the principal ore mineralising events. The early history of the quartz is generally lost, but the idea was to examine a potential trap for post-$D_{n+1}$ fluid circulations.

Around 30 thick sections were prepared and examined in detail using a microscope, permitting zoom between magnifications of x200 and x1000. Results were well below what was expected as inclusions are, in most samples, very tiny (around or less than 5 $\mu$m) and very difficult to study or interpret as several processes of necking and deformation have entirely obliterated the message from fluid inclusions. Contrarily to what was expected, the quartz veins did not act as a satisfactory trap for fluids during the last stages of Variscan deformation, synchronous of granite intrusions and formation of the W-Sn ores.

Notwithstanding the difficulties, the inventory of textures and the feasibility of microthermometric investigations have been undertaken. Studies will be carried out by Raman spectroscopy and microthermometry next winter. Nonetheless, the size of the inclusions will forbid the use of LA-ICP-MS in

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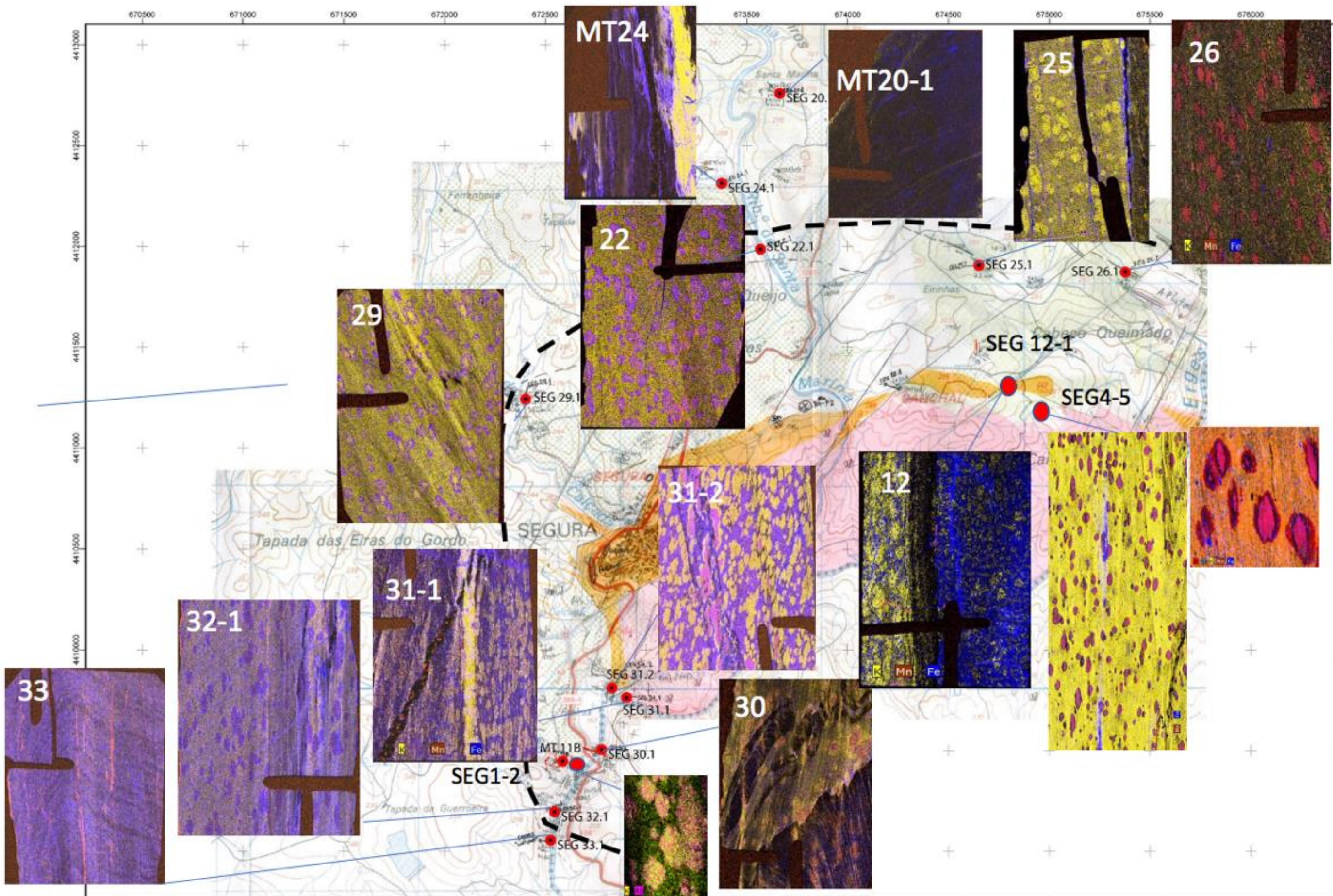


**Fig. 22 –** Micro-XRF maps of the spotted schists in the vicinity of the Segura granite and aplite dykes reported on the geological map. The matrix appearing in blue is dominated by (variably chloritized) biotite and, alternately, the samples with yellow colour show progressive intensity in “muscovitisation” (K is in yellow, Fe in blue, Mn in red-brown), beginning with spots and ending with the entire matrix.

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most cases. Representative examples of textures in the main sites investigated (Perais, Castelo Branco, Segura old Sn-mine, Medelim, Salvaterra, Monsanto, Mata Rainha) are displayed in **Fig. 23**. A new series of wafers is under preparation to search for fluid inclusions more suitable in size.

In **WP4**, a significant part of the activities developed during the 2nd year of project implementation was directed towards task **4.3** (***Re-assessment of alluvial heavy minerals from old exploration surveys***) through the study of heavy minerals' physical/chemical properties extracted from alluvial samples in the Segura area. Several grain populations were identified and characterized. The main results include the following:

(1) Definition of 9 cassiterite populations (**Fig. 24A**) based on colour (yellow, beige, white, red, brown, and black shades; some with colour zonation), diaphaneity and pleochroism. The variability in terms of physical properties is a striking feature of Segura's alluvial cassiterite specimens. It may reflect distinct compositional and environmental conditions of the multiple Sn mineralization events and/or their distinct evolving stages. Zoned cassiterite grains, showing reddish-brown to colourless pleochroism (Type 4), are alike of Nb-Ta-rich cassiterites reported for the Segura Li-bearing granitic aplite-pegmatite veins and, despite their wide occurrence, a spatial association with exposures of these ore bodies (in the eastern part of the area) is recognised (**Fig. 24A**).

(2) Distinction of 7 major rutile populations, arranged into 4 Groups, according to their habit (prismatic – group A; anhedral – group B; anhedral or acicular polycrystalline aggregates – group C; bipyramidal and others undifferentiated – group D); Group B habit dominates in samples from the eastern part of the Segura area (**Fig. 24B**). The two anatase grain populations, both with variable colour, were identified according to their habit, namely the most common tetrahedral bipyramids (Type 1) and the basal pinacoids with pyramidal faces (Type 2); Type 1 tends to dominate in samples near or within the granitic border, mainly in the eastern part of the Segura area (**Fig. 24C**). Brookite grains can be principally distinguished by its orange, brownish orange (Type 1) or orange, greenish-yellow colour (Type 2), the former tending to dominate in the relative proximal samples of the Segura granite exocontact (**Fig. 24D**).

(3) Discrimination of 9 tourmaline populations by their colour (several shades of brown, green, blue, orange-red and pink; **Fig. 24E**). Brown (Type 1) and green (Type 2) tourmaline are dominant in samples sourced to the northern and eastern lithological outcrops, further away from Segura granite (**Fig. 24E**). Pink tourmaline (Type 6) was identified only in samples collected at the NW extreme of the Segura area (**Fig.24E**). The alluvial, mixed, grains of tourmaline-cassiterite intergrowths reflect the common association between tourmaline and the W-Sn regional mineralizing events. The fine-grained/acicular tourmaline-phyllosilicate aggregates may correspond to relics of the tourmalinisation processes experienced by metasedimentary rocks adjoining the ore veins or granitic rocks, as commonly observed in many places. Preliminary chemical data of alluvial tourmaline composition suggest the co-existence of 3 main compositional trends, Sn-enrichment, Sn+Li-enrichment and Li-enrichment (**Fig. 25A**), creating reasonable expectations on their use as vectors to ore-forming systems, after an improved critical data analysis.

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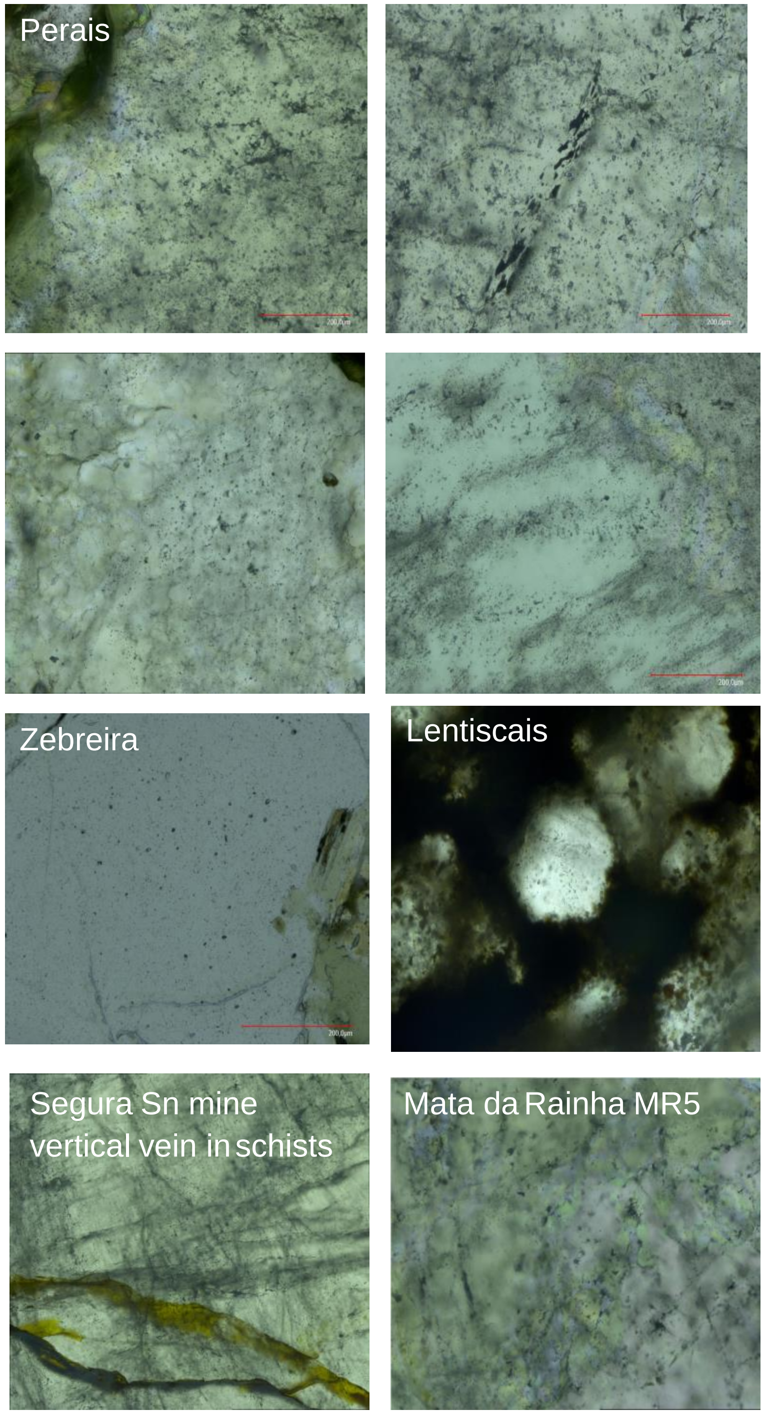


**Fig. 23 –** Photographs of fluid inclusions assemblages in quartz lenses from the metamorphic series in the Segura – Castelo Branco region: Perais, Zebreira, Lentiscais and Segura surroundings.

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(4) Distinction of 2 garnet populations: euhedral grey to orange spessartine (Type 1), usually full of dark inclusions, and anhedral fragments of hyaline pale rose to slightly orange almandine (Type 2). Type 1 garnet grains show a widespread (regional) spatial distribution. In contrast, Type 2 is mainly sourced by the Segura granitic rocks and adjoining exo-contacts or other granitic rocks in alluviums along the WNW-ESE direction (**Fig. 24F**). The regional distribution, mineralogical and chemical preliminary data gathered for Type 1 and 2 garnet grains (**Fig. 25B**) show a positive correlation with the Mn-enriched spessartine observed in the contact metamorphic aureole contiguous to the Salvaterra do Extremo granite (to the north of Segura) and with the almandine found in Garnet-Cordierite Granite Porphyry dykes related to the Cabeza de Araya batholith (in Spain), the southeastern extension of the Segura granite.

(5) Scheelite from some alluvial samples show a predominance of colourless-white to creamy semi-translucent grains. Still, one colourless hyaline scheelite grain population stands out in samples collected in the western part of the surveyed area. However, until now, the response to UV light does not evidence any significant variance (bluish colour) in these two scheelite populations suggesting no significant difference in their Mo content. For wolframite, no population distinction has been identified yet, in opposition to ilmenite which could be grouped into three grain populations.

During the 2$^{nd}$ year of project implementation some other advancements were accomplished in mineralogical and geochemical proxies and vectors to granite-related mineralization, preliminary addressing tasks **4.1/4.2 (WP4)** and **5.1 (WP5)**. Some of these results were already presented in an international scientific meeting (Martins et al., 2022a; Gaspar et al., 2022) and, in part, published (Martins et al., 2022b) – see section B.1 of this report.

Summing up the whole-rock geochemical data so far obtained it was possible to verify that, as expected, highly fractionated granites and related magmatic-hydrothermal ore-forming processes can be traced by elemental ratios such as Nb/Ta, K/Rb, Y/Ho, Sr/Eu, Eu/Eu*, Zr/Hf, and Rb/Sr. The utility of the lanthanide "tetrad effect" parameter ($TE_{1,3}$) as a geochemical fingerprint of highly fractionated granites was successfully tested (Martins et al., 2022a, b), revealing that the increase in $TE_{1,3}$ values co-vary with magmatic differentiation and metal-enrichment. The Argemela Li-Sn-bearing rare metal granite and the Segura Li-phosphate-bearing aplite–pegmatite dykes deviate from this geochemical trend, displaying $TE_{1,3}$ $< 1.1$ but high $P_2O_5$ contents. This suggests that mineralized rocks related to peraluminous-high-phosphorus Li-Sn granite systems are typified by $TE_{1,3} < 1.1$, whereas those associated with peraluminous-high-phosphorus Sn-W-Li (lepidolite) and peraluminous-low-phosphorus Sn-Ta-Nb granite systems display $TE_{1,3} > 1.1$, reaching values as high as 1.4 and 2.1, respectively.

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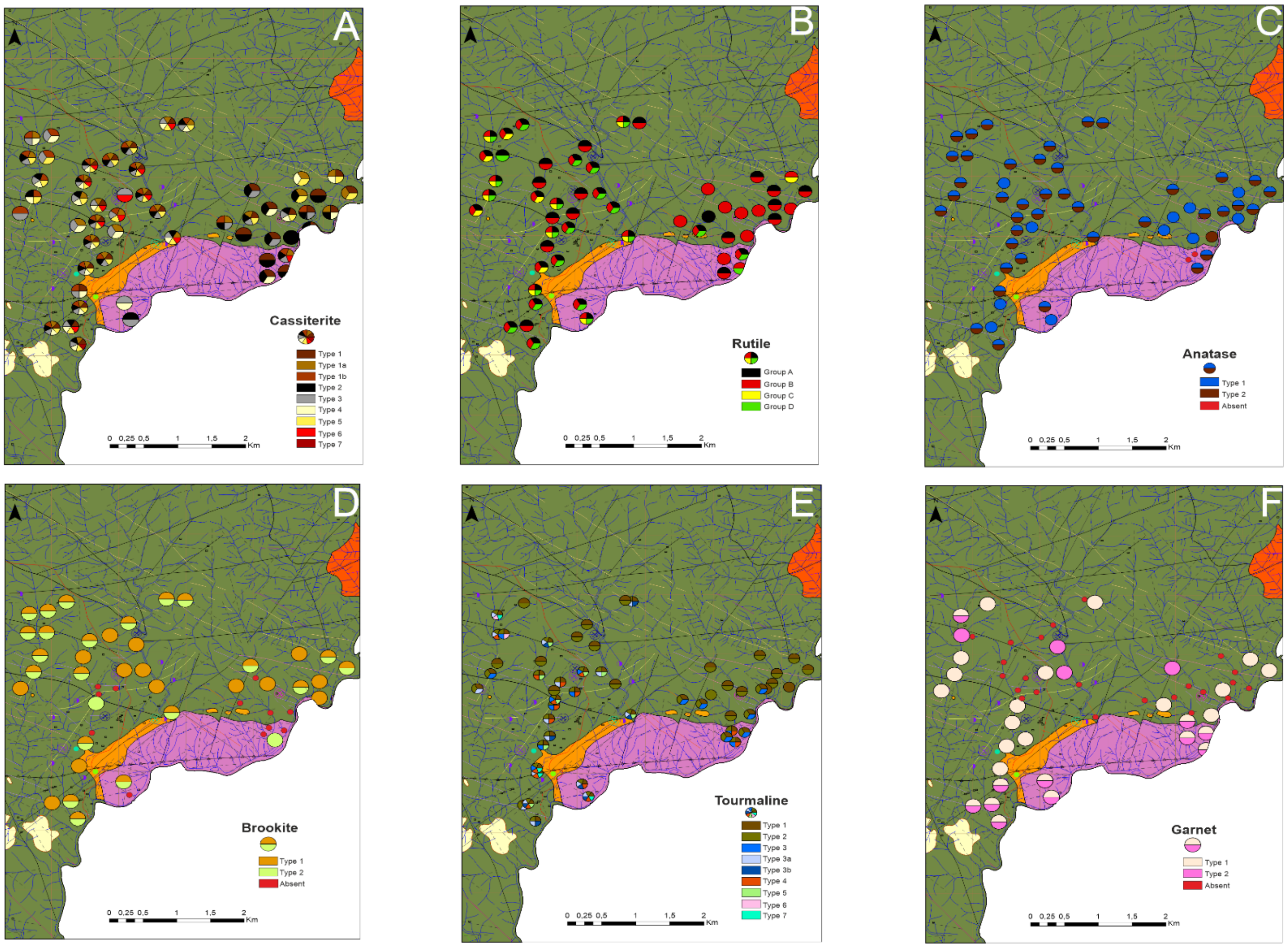


**Fig. 24** – Preliminary regional distribution of the alluvial heavy mineral grain populations in Segura, plotted over the geological map produced under the scope of the MOSTMEG project (for details see legend of Fig. 5). Grain populations for: A) Cassiterite, defined mainly by colour (see text; mostly similar to the graphic marker of each Type), diaphaneity and pleochroism; B) Rutile, defined by habit (Group A: prismatic; Group B: anhedral; Group C: acicular polycrystalline aggregates; Group D: bipyramidal and others undifferentiated); C) Anatase, defined by habit (Type 1: bipyramidal; Type 2: basal); D) Brookite, defined by colour (see text; primarily similar to the graphic marker of each Type); E) Tourmaline, defined by colour (see text; mainly similar to the graphic marker of each Type); F) Garnet (Type 1: spessartine; Type 2: almandine); red dots: Absent (samples without the mineral phase).

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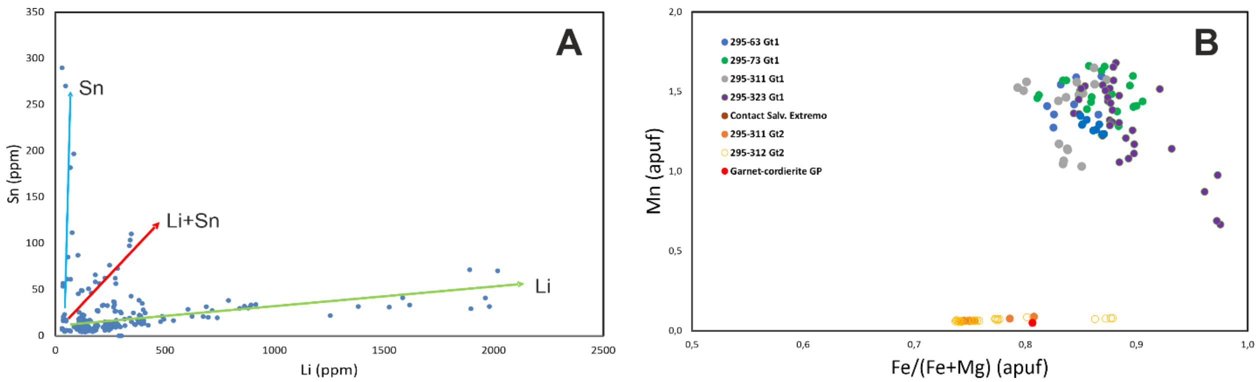


**Fig. 25** – (A). Sn vs Li diagram for alluvial tourmaline from the Segura area, showing the trends according to Sn-enrichment, Sn+Li- enrichment and Li-enrichment (LA-ICPMS data). (B). Mn vs Fe/(Fe+Mg) diagram for alluvial garnet populations (Gt1: Type 1, spessartine; Gt2: Type 2, almandine; EMPA data) from Segura. Spessartine data from contact metamorphism halo metasedimentary rocks in adjacent Salvaterra do Extremo region (to N of Segura) and almandine data from Garnet-Cordierite Granite Porphyry dyke associated with Cabeza de Araya batholith (Spanish territory; Corretgé and Suárez,1994), were also plotted, showing the positive correlation with alluvial garnet Type 1 and 2 from Segura, respectively.

The assessment of tourmaline composition as a vectoring tool for Sn-W deposits was also successfully tested with some detail making use of ca. 2000 EPMA data compiled for tourmaline from the Góis-Panasqueira-Segura Sn-W strip (Gaspar et al., 2022). Granite tourmalines are typically schorlitic with low #Mg ($Mg/(Mg+Fe_T)$) and #Ca (Ca/(Ca+Na+K)), whereas metasediment tourmalines are dravitic with higher #Mg and #Ca. The #Mg *vs* #Ca cross-plots show (1) magmatic differentiation trends for composite granites, with a progressive decrease in #Mg and #Ca, (2) compositional variability within metasediments and (3) mixing lines for each hydrothermal system between tourmaline compositions buffered by metasediments and by magmatic-hydrothermal fluid sources. The mixing line spread discloses the hydrothermal system magnitude (time, space, and mineralizing fluid volume), as shown by the wider trend for the Panasqueira deposit compared to smaller deposits such as Góis or Segura. However, complete interpretation of mixing lines, or deviations from it, require detailed petrography and careful microprobe spatial and compositional control to address aspects such as multiple tourmaline generations, compositional zoning, or even diffusion mechanisms. In general, hydrothermal tourmaline documents a progressive increase in #Mg and #Ca, reflecting a simultaneous rise of the magmatic-hydrothermal fluid component. Except for Panasqueira, tourmalinites at the Penamacor-Monsanto batholith exo-contact record the lowest #Mg values denoting their proximity to the intrusion. For systems like Góis, where no granites are known in the vicinity, the trend starts at higher #Mg values, and a more distal position relative to a causative granitic intrusion is believed.

Zircon grains from some aplite dykes display disturbance effects similar to those caused by metamict (radiation-damage) transformations. In other cases (such as the porphyry laccoliths and dykes nearby Zebreira, but also documented for many zircons extracted from highly differentiated granites or granitic facies affected by mineralising fluids), the measured contents of U and $^{204}Pb$ are quite high, ranging from 500 to 10000 ppm, and often above 1 wt%, respectively. Future work will explore the geological meaning of this U enrichment (co-varying with $^{204}Pb$ increase?) in zircons included in rocks affected by ore-forming processes. This approach will be developed together with the analysis of REE contents in zircon, which

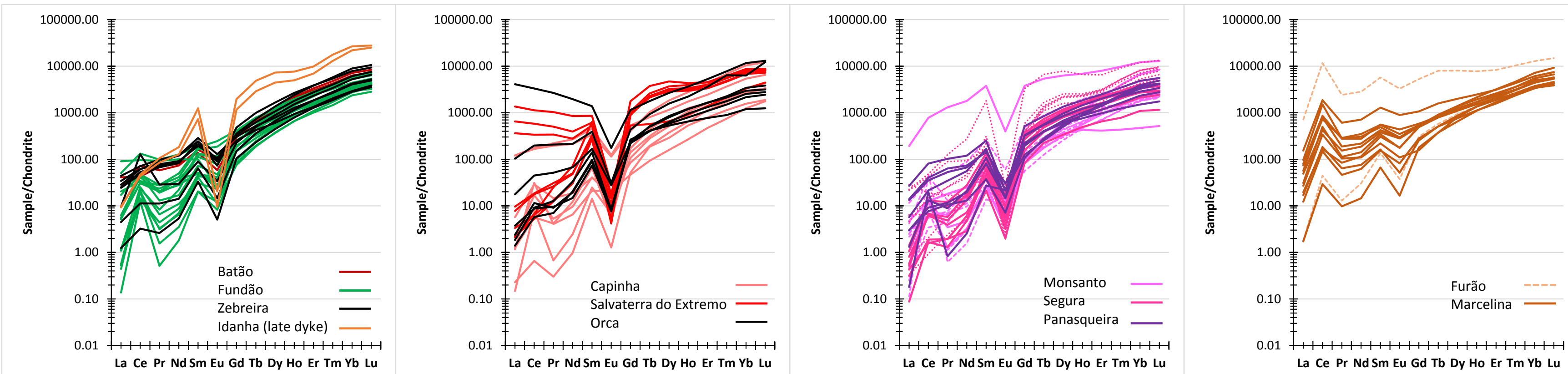


**Fig. 26** – CN-normalised REE patterns for zircon grains extracted from samples representing different granite facies and aplite/pegmatite dykes exposed in the Argemela-Segura region.

preliminary results reveal (**Fig. 26**): (1) an evident LREE-HREE fractionation, together with positive Ce anomalies and negative Eu anomalies in non-altered zircons from granite rocks; (2) a significant LREE enrichment, notwithstanding the typical Ce and Eu anomalies, in non-altered zircons from late porphyry rocks; and (3) a prominent LREE enrichment along with evident fading or even disappearance of the Ce (and sometimes Eu) anomaly in zircon grains variably affected by high-temperature hydrothermal processes.

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## A.3 Deviations

### A.3.1 Deviations and difficulties

Some deviations from the work plan due to circumstances imposed by the COVID-19 pandemic (reported in October 2021) have been overcome. However, delays with sampling densification and several operational difficulties with analytical work to be carried out by some Consortium Partners have slowed down the expected progress in various tasks, mostly in those associated with WP3 and WP4. This is a common impact in work plans designed to take advantage of tight cooperation between partners.

As comprehensively explained in the formal request to the ERA-MIN Secretariat for a nine-month deadline extension, the full operability of the Raman Spectroscopy laboratory at FCUP was only guaranteed on 16$^{th}$ February 2022, after the malfunction detection and report to the equipment's technical support on 21$^{st}$ June 2021. We, therefore, needed to wait ca. eight months to resume fluid inclusion analyses at FCUP under the scope of WP3. The situation with the Laser Ablation system and the low energy EDS detector at HERCULES Lab - Univ. Évora was much critical. In what concerns LNEG, the major deviations from the work plan and operational difficulties recorded until now were due to delays in the hiring of a research fellowship for Master (BI) (14 months), which only occurred on 21/03/2022, after the request to change (06/12/2021) the hiring typology from “MSc contract” (14 months) of the original proposal. The administrative process of this “MSc contract” has proven to be highly complex and time-consuming, requiring a series of prior authorizations (following Law nº 35/2014) and thus not expected to be completed in good time for the development of the project (as justified to FCT on November 2, 2021). Delays in the completion of initial tasks also impacted the launch of applications for the Post-doc fellowships planned for HERCULES Lab - Univ. Évora and CNR-GeoRessources.

### A.3.2 Extension of deadlines

In October 2021, a six month extension of the original deadline was considered acceptable to balance the scheduling uncertainties and operational difficulties due to the COVID-19 pandemic during the 1st year of project implementation. At FCUL, it was possible to recover a significant part of the accumulated delay, but the progression of several other activities depended on the results obtained by different partners, as usual in a collaborative project. Consequently, delays in specific sampling for WP3/WP4 by the CNRS-GeoRessources team and supplementary sampling for WP2/WP3 by the IGc-USP team have impacted the completion of some milestones and deliverables. In addition, malfunctions of analytical equipment at FCUP and HERCULES Lab and, above all, the delays with equipment repair (mainly due to the long wait for the delivery of components - much longer than usual) significantly hampered the achievement of the expected results. With a nine-month extension of the original deadline, already formally requested, all the intended data and information will be collected to suitably characterize the geological background of the Góis-Segura strip and the selected targets in it (such as Vale Pião, Argemela, Mata da Rainha and Segura mineralizing systems). This will permit a thorough analysis of the obtained databases, providing the means to proceed confidently in the tasks planned for WP5 and WP6. In addition, this will increase the impact of the MOSTMEG deliverables, including the two Seminars open to the scientific community, companies and mining authorities.

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### A.4 Problems in the implementation of the work plan

*☒ Difficulties in recruiting personnel*

*☐ Poor communication between project partners*

*☐ Change of one project partner*

*☐ One or more partners underperforming*

*☒ Experimental/technical difficulties*

*☒ Other, please specify:*

Operational difficulties related to travel restrictions imposed by the COVID-19 pandemic hampering the completion of fieldwork activities and sampling surveys as initially planned. Operational difficulties related to lab access restrictions imposed by the COVID-19 pandemic varied in time and differed in Portugal, France and Brazil. Malfunctions of analytical equipment at FCUP and HERCULES Lab and, above all, the delays with equipment repair (mainly due to the long wait for the delivery of components - much longer than usual). Fortunately, the difficulties are now overcome or being resolved.

Comments:

The delays in recruiting personnel mentioned in section A.3 are currently resolved.

## B. RESULTS

### B.1. Scientific results

| List resulting from jointly conducted work | | | | |
|---|---|---|---|---|
| **Type of result** | **Authors, title, year, issue/editor** | **Partner(s) involved** | **Open Access (Yes/No)** | **Website address** |
| Peer review papers* | 1. Martins I., Mateus A., Cathelineau M.; Boiron M.-C.; Ribeiro da Costa I., Gaspar M., Dias da Silva I. (2022) The lanthanide tetrad effect as an exploration tool for granite-related rare metal ore systems: exemples from Iberian Variscides. Minerals 12(9), 1067<br>2. Gaspar, M.; Grácio, N.; Salgueiro, R.; Costa, M. Trace element geochemistry of alluvial $TiO_2$ polymorphs as a proxy for Sn and W deposits. Minerals 2022, 12(10), 1248. | P1, P7<br>P1, P3, P4 | Yes<br>Yes | https://doi.org/10.3390/min12091067<br>https://doi.org/10.3390/min12101248 |
| Books or book chapter | 1.<br>2.<br>n. | | | |

| | | | | |
|---|---|---|---|---|
| Publications co-authored by R&D and industrial partners | 1.<br>2.<br>n. | | | |
| Conference proceedings/ presentations | 1. Martins I., Mateus A., Ribeiro da Costa I., Gaspar M., Dias da Silva I. (2022) The lanthanide tetrad effect as an exploration tool for granite-related rare metal ore systems: examples from Iberian Variscides. SEG 2022 Conference: *Minerals for Our Future*. Theme: *Critical Minerals for Our Energy Future: Geology and Ore Deposit Models*. ID4112. | P1 | Yes | https://www.segweb.org/SEG-2022/SEG-Conference/SEG-2022/Home.aspx |
| | 2. Gaspar M., Ribeiro da Costa I., Mateus A., Martins I., Rodrigues P. (2022) Assessment of tourmaline composition as a vectoring tool for Sn-W deposits – the Góis-Panasqueira-Segura Belt (Central Portugal). SEG 2022 Conference: *Minerals for Our Future*. Theme: *Recent Innovations, Integrated Methods, and Case Studies*. ID4317 | P1 | Yes | https://www.segweb.org/SEG-2022/SEG-Conference/SEG-2022/Home.aspx |
| | 3. Yakovenko A., Guedes A., Boiron M.-C., Cathelineau M., Martins I., Mateus A. (2022) Fluid inclusion studies in quartz from the Li-rich pegmatite veins from Segura. Jornadas ICT, 10-11 February 2022. Book of Abstracts, p. 43 | P2, P7, P1 | Yes | https://www.icterra.pt/index.php/2022/01/07/jornadas-ict-10-e-11-de-fevereiro-2022/ |
| | 4. Martins I., Mateus A., Ribeiro da Costa I., Gaspar M., Dias da Silva, Í. (2022) Geochemistry and ore-forming processes of multistage granitic magmatism in the Central Iberian Zone: Segura-Panasqueira Belt (Portugal) case study. 16th SGA Biennial Conference 2022: *The critical role of minerals in the carbon-neutral future* (28-31 March). | P1 | Yes | https://www.aig.org.au/events/16th-biennial-meeting-sga-2022/ |
| | 5. Yakovenko, A., Guedes, A. 2022. Mineralogy and fluid inclusion studies in quartz from the Li-rich pegmatite veins from Segura. Young Researcher Meeting - IJUP. 4-6 May. Book of abstracts, p. 343 | P2 | Yes | |

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| | | | | |
|---|---|---|---|---|
| Other dissemination activity | 1.<br>2.<br>n. | | | |

**B.2. Innovation oriented results**

| List of results | | | | |
|---|---|---|---|---|
| **Type of result** | **Title** | **Authors** | **Partner(s) involved** | **Date submission/granted** |
| Patent applications | | | | |
| Patents | | | | |
| International patent | | | | |
| EU patents | | | | |
| National patents | | | | |
| Licenses | | | | |
| New collaborative projects | | | | |
| Other | | | | |

***Not applicable to MOSTMEG project***

**B.3. Human resources involved**

| Academic level | Name of persons involved in project activities |
|---|---|
| Master Degree | **3** |
| PhD degree | **22** |
| Post-doc | **2** |
| Graduation degree | **1** |
| Undergraduate | **0** |

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# C. FINANCIAL STATUS

| Partner # 1 | Faculdade de Ciências da Universidade de Lisboa and IDL |
|---|---|
| TOTAL ALLOCATED BUDGET | 260,274.20 |
| National/regional financing | 75,589.20 |
| Own financing | 184,685.00 |
| TOTAL Spent at Annual reporting no. 2 | 124,234.30 |
| National/regional financing | 49,361.99 |
| Own financing | 74,872.30 |
| DEVIATION (against the forecasted expenses at that time) | 0.00 |
| National/regional financing | 0.00 |
| Own financing | 0.00 |

| Partner # 2 | Faculdade de Ciências da Universidade do Porto |
|---|---|
| TOTAL ALLOCATED BUDGET | 49,500.00 |
| National/regional financing | 0.00 |
| Own financing | 0.00 |
| TOTAL Spent at Annual reporting no. 2 | 23,346.66 |
| National/regional financing | 23,346.66 |
| Own financing | 0.00 |
| DEVIATION (against the forecasted expenses at that time) | 14,302.40 |
| National/regional financing | 0 |
| Own financing | 0 |

Deviations recorded by FCUP reflect mostly the setbacks occurred during the 1st year of the project implementation, already described in the 1st annual progress report.

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| Partner # 3 | Universidade de Évora, Geociências - HERCULES |
|---|---|
| TOTAL ALLOCATED BUDGET | 47,025.00 |
| National/regional financing | 47,025.00 |
| Own financing | 0.00 |
| TOTAL Spent at Annual reporting no. 2 | 6,821.25 |
| National/regional financing | 6,821.25 |
| Own financing | 0.00 |
| DEVIATION (against the forecasted expenses at that time) | 0.00 |
| National/regional financing | 0.00 |
| Own financing | 0.00 |

| Partner # 4 | Laboratório Nacional de Energia e Geologia, I.P. |
|---|---|
| TOTAL ALLOCATED BUDGET | 53,852.23 |
| National/regional financing | 35,394.30 |
| Own financing | 18,458.23 |
| TOTAL Spent at Annual reporting no. 2 | 18,483.71 |
| National/regional financing | 12,247.88 |
| Own financing | 6,235.83 |
| DEVIATION (against the forecasted expenses at that time) | 17,754.18 |
| National/regional financing | 17,837.27 |
| Own financing | -83.09 |

The main deviations recorded by LNEG during the 2nd year of the project implementation are due to the change of the hiring typology from “MSc contract” to a Research Fellowship for Master (BI) and the delay on hiring the Research Fellowship for Master (BI), which happened on March 21st, 2022. This had a great impact on the expenses since the budget for “Human Resources” has a high weight in LNEG´s total budget.

| Partner # 5 | Universidade de Coimbra |
|---|---|
| TOTAL ALLOCATED BUDGET | 28,137.50 |
| National/regional financing | 17,437.50 |
| Own financing | 10,070.00 |
| TOTAL Spent at Annual reporting no. 2 | 3,356.67 |
| National/regional financing | 2,313.02 |
| Own financing | 0.00 |
| DEVIATION (against the forecasted expenses at that time) | 0.00 |
| National/regional financing | 0.00 |
| Own financing | 0.00 |

| Partner # 6 | Instituto de Geociências da Universidade de São Paulo, Departamento de Mineralogia e Geotectónica e Centro de Pesquisas Geocronológicas |
|---|---|
| TOTAL ALLOCATED BUDGET | 64,859.00 |
| National/regional financing | 0.00 |
| Own financing | 64,859.00 |
| TOTAL Spent at Annual reporting no. 2 | 21,619.67 |
| National/regional financing | 0.00 |
| Own financing | 21,619.67 |
| DEVIATION (against the forecasted expenses at that time) | 0.00 |
| National/regional financing | 0.00 |
| Own financing | 0.00 |

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| Partner # 7 | CNRS - GeoRessources |
|---|---|
| TOTAL ALLOCATED BUDGET | 278,715.00 |
| National/regional financing | 162,815.00 |
| Own financing | 115,900.00 |
| TOTAL Spent at Annual reporting no. 2 | 45,418.19 |
| National/regional financing | 6,754.86 |
| Own financing | 38,633.33 |
| DEVIATION (against the forecasted expenses at that time) | 15,768.14 |
| National/regional financing | 22,523,25 |
| Own financing | 0.00 |

## D. PUBLISHABLE SUMMARY

According to the available SHRIMP U-Pb zircon data, the mineralization events within the Góis-Segura strip are scattered in the ca. 310-290 Ma time window.

Aplite-pegmatite dykes from Segura are compositionally very close to the Argemela granites, depicting the involvement of highly differentiated and $Na_2O$ enriched magmas. Compared to the Panasqueira granites, the Segura aplite-pegmatite dykes are characterised by an excess of P not linked to apatite but to amblygonite-montebrasite series and to a Fe-Mn rich phosphate. These dykes also include Nb-Ta- and Sn-oxides, often characterised by complex compositional zonings. In general, the highly fractionated granites and related magmatic-hydrothermal ore-forming processes can be traced by Nb/Ta, K/Rb, Y/Ho, Sr/Eu, Eu/Eu*, Zr/Hf, and Rb/Sr elemental ratios. Similarly, the lanthanide "tetrad effect" parameter ($TE_{1,3}$) co-varies positively with the magmatic differentiation and metal-enrichment.

Fluid inclusions in different minerals forming the Li-Sn quartz lodes of Pedra Alta (Argemela) document the prevalence of water-dominated, low salinity fluids with bulk homogenization temperature ranging from ca. 230 to 350ºC. These fluids display a $CO_2$-dominated volatile phase when trapped in amblygonite and quartz, and show variations from $CO_2$-dominated to $CH_4$-dominated when confined to cassiterite.

Muscovite in granites is frequently zoned and the rims incorporate significant amounts of Li, Rb, Cs and Sn, reflecting the existence of fluids (or magmas) enriched in incompatible elements at the end of the crystallization of *ms*-rich facies (e.g. border of the Segura pluton; Medelim and Monsanto margin facies of the Penamacor body). Tourmaline also displays variable composition when included in granitic (schorlitic with low #Mg [$Mg/(Mg+Fe_T)$] and #Ca [Ca/(Ca+Na+K)]) or metasedimentary (dravitic with higher #Mg and #Ca) rocks. The #Mg *vs* #Ca cross-plots show (1) magmatic differentiation trends for composite granites, with a progressive decrease in #Mg and #Ca, (2) compositional variability within metasediments and (3) mixing lines for each system between tourmaline compositions buffered by metasediments and by magmatic-hydrothermal fluid sources. The mixing line spread discloses the hydrothermal system

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magnitude, as shown by the wider trend for the Panasqueira deposit compared to smaller deposits (Góis, Segura).

Spotted-schists forming the contact metamorphic aureoles encircling “fertile” granites are being examined carefully because the replacements of cordierite/andalusite spots by late mica or tourmaline may be regarded as a proxy to (significant) hydrothermal fluid flow.

In Segura, the alluvial cassiterite, wolframite, and scheelite anomaly pattern halos can be correlated with distinct W-Sn metallogenic events, interpreted to have different origins and control factors (e.g. structural, lithological, magmatic/hydrothermal, timing), conceivably superimposed mostly in the western part of the surveyed area.

Additional comments to this reporting:

**Project Consortium Coordinator Signature:** **Date: 31 October 2022**

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