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RESEARCH & INNOVATION PROGRAMME ON RAW MATERIALS
TO FOSTER CIRCULAR ECONOMY

ERA-MIN Joint Call 2019

MOSTMEG

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

First Annual Project Report



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A. GENERAL INFORMATION

| | |
|---|---|
| Project acronym | MOSTMEG |
| Project title | <i>Predictive models for strategic metal rich, granite-related ore systems based on mineral and geochemical fingerprints and footprints</i> |
| Project start date (day/month/year) | 01/10/2020 |
| Project end date (day/month/year) | 30/09/2023 |
| Period covered by the report | 01/10/2020 – 30/09/2021 |
| Project website | https://mostmeg.rd.ciencias.ulisboa.pt/ |
| Date of submission of the report | 28/10/2021 |
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| Project Partners (other than Consortium Coordinator) | <i>Project Partner 2 – Faculdade de Ciências da Universidade do Porto Alexandra Guedes E-mail address: aguedes@fc.up.pt female <input checked="" type="checkbox"/> male <input type="checkbox"/> Portugal Partner type: Academia <input checked="" type="checkbox"/> Large Company <input type="checkbox"/> SME <input type="checkbox"/> NGO <input type="checkbox"/></i> |
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| | <i>Project Partner 4 – Laboratório Nacional de Energia e Geologia, I.P. Rute Maria Morgado Salgueiro E-mail address: rute.salgueiro@lneg.pt female <input checked="" type="checkbox"/> male <input type="checkbox"/> Portugal Partner type: Academia <input checked="" type="checkbox"/> Large Company <input type="checkbox"/> SME <input type="checkbox"/> NGO <input type="checkbox"/></i> |
| | <i>Project Partner 5 – Universidade de Coimbra Alcides Pereira E-mail address: apereira@dct.uc.pt</i> |
| | |

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



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| | <p>female <input type="checkbox"/> male <input checked="" type="checkbox"/></p> <p>Portugal</p> <p>Partner type: Academia <input checked="" type="checkbox"/> Large Company <input type="checkbox"/> SME <input type="checkbox"/> NGO <input type="checkbox"/></p> <hr/> <p>Project Partner 6 – Instituto de Geociências da Universidade de São Paulo, Departamento de Mineralogia e Geotectónica e Centro de Pesquisas Geocronológicas</p> <p>Colombo Celso Gaeta Tassinari</p> <p>E-mail address: ccgtassi@usp.br</p> <p>female <input type="checkbox"/> male <input checked="" type="checkbox"/></p> <p>Brazil</p> <p>Partner type: Academia <input checked="" type="checkbox"/> Large Company <input type="checkbox"/> SME <input type="checkbox"/> NGO <input type="checkbox"/></p> <hr/> <p>Project Partner 7 – CNRS - GeoRessources</p> <p>Michel Cathelineau</p> <p>E-mail address: michel.cathelineau@univ-lorraine.fr</p> <p>female <input type="checkbox"/> male <input checked="" type="checkbox"/></p> <p>France</p> <p>Partner type: Academia <input checked="" type="checkbox"/> Large Company <input type="checkbox"/> SME <input type="checkbox"/> NGO <input type="checkbox"/></p> |
| GDPR consent | <p>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.</p> <p>yes <input checked="" type="checkbox"/> no <input type="checkbox"/></p> |

B. INTERNATIONAL COOPERATION

B.1 How was the consortium formed? (Tick all appropriate options)*

Previous connections/collaborations

Networking events including workshops, seminars, scientific conferences

Partner search tool on ERA-MIN website

Web search, social or professional networks

Other:

B.2 Previous experience in working together within the:

None, we are working together for the first time

Yes, with part of the consortium Indicate which partners collaborated previously:

(P1+P4), project MINATURA2020, EU funding; (P1+P2+P4+P6), project NewOres, ERA-MIN; (P2+P7), project DEASPHOR, ERA-MIN; (P1+P6) and (P2+P7) several interchange projects; (P3+P4), EXPLORA, ZOM3D and InCarbon projects, national funding); (P1+P4), Greenfuel, national funding; (P1+P3) KADRWaste, national funding; (P4+P5), Suscity, national funding, and Interreg Sudoe; (P4+P6, as CNRS), SFERAI and EXCITE, EU funding.



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Yes, with the whole consortium

If your previous answer was "Yes", did that experience include applications to grants for transnational research projects?

Yes No

What programme? Multiannual R&D Programme (1990-92), Horizon2020, ERA-MIN

B.3. Were new collaborations established by the consortium or part of the consortium due to project results?

No

Yes

If yes,

National collaborations?

International collaborations?

Other initiatives?

Other ERA-MIN 2 projects? Which ones?

B.4. Did the consortium or part of the consortium applied to other national / transnational calls?

No

Yes

If yes, how many applications in total?

In the past 3 years, one application to a national call (project MINPROM.Pt, involving P1+P2+P3+P4+P5) and one application to the ERA-MIN Joint Call 2021 (project DIESEL.4future, involving P2 and P7). Both proposals were not selected for the second-phase of the calls.

B.5 Has there been any change in the consortium composition since the start of the project?

Yes No

If Yes, please indicate which partner(s), the justification and if there the partner was replaced.



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B.6 Please list the project meetings, workshops held during the implementation period:

| Meeting objective | Partners involved | Date | Place |
|---|-------------------|--------------------|---|
| Steering Group 1st Meeting | All | October 30, 2020 | Video-conference |
| Steering Group 2nd Meeting | All | April 23, 2021 | Video-conference |
| Annual Meeting and Steering Group 3rd Meeting | All | September 18, 2021 | 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. The Annual Meeting and Steering Group 3rd Meeting were preceded by a Joint Fieldtrip (13th-17th September 2021). This Joint Fieldtrip, attended by 11 researchers from 4 project partners (coming from Portugal and France), was instrumental 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; (iii) increase the sampling densification in specific targets to support forthcoming detailed studies (WP3, WP4 and WP5); and (iv) refine several details of the intended research programme, reinforcing the collaborative efforts between the project partners.

C. PROGRESS REPORT

C.1 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 6 | Month 15 |
| 2 | Vectoring metal endowment and critical timing for mineralisation triggering | M2.1 | P1 & P6 | P1, P6, P7 | Month 7 | Month 7 |
| | | M2.2 | P6 & P1 | | Month 15 | Month 18 |
| 3 | Relevant processes for metal concentration and deposition at the ore system scale | M3.1 | P2 & P7 | P1, P2, P7 | Month 7 | Month 7 |
| | | M3.2 | P2 & P7 | | Month 12 | Month 12 |
| 4 | Mineral fingerprints and footprints | M4.1 | P4 | P4, P1, P3 | Month 7 | Month 9 |



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According to the work plan approved for MOSTMEG, the research activities performed during the 1st year were oriented towards the objectives of **WP1** (tasks 1.1, 1.2 and 1.3), **WP2** (tasks 2.1 and 2.2), **WP3** (tasks 3.1, 3.2 and 3.3) and **WP4** (task 4.3). Researchers from all the consortium partners participate in these activities.

C1.1 Milestones (M) accomplishment

M1.1 (WP1) was partly accomplished. Additional fieldwork is necessary to address misgivings raised by the compilation of old data and complete the (GIS-supported) “*Preliminary Database*”, also supporting the intended harmonised geological map for the Segura-Argemela-Panasqueira-Góis strip.

In what concerns the preparatory activities for WP4, the compilation of (un)published literature data has allowed:

(i) A general assessment of the availability and conditions of alluvial heavy minerals (HM) samples, successfully used in the past for Sn-W ore exploration surveys, leading to the selection of the Segura-Salvaterra do Extremo-Zebreira (Sul) region and of cassiterite, wolframite, scheelite, TiO₂ polymorphs and tourmaline as the focus potential indicator minerals of rare metal granite related ore systems to study.

(ii) The production of a guide for HM study and the use of HM as a tool in regional mineral exploration, condensing the information into two tables. Table 1 provides HM data from mineral assemblages forming different rock types and mineralised bodies, along with resumed information about HM morphology, size, and mineral inclusions, as well as their geological setting, location and host structure or lithology. This compilation delivers an initial picture of the spatial distribution of mineralogy and grain population variability in the region, the prevailing HM related to the main (i.e. cassiterite, scheelite and wolframite) and secondary mineralisation and metamorphic halos (e.g. tourmaline) and plausible connections of alluvial HM to their sources. Table 2 includes the (50) HM identified in exposed rocks and alluvial samples already studied, and the occurrence of alluvial HM, along with their chemical formula, specific gravity, magnetic susceptibility, fluorescence, Mohs scale hardness and stability to weathering; these properties make it possible to identify HM and understand their presence/absence in alluvial samples, before binocular microscope analysis during task 4.3 of WP4.

(iii) A data review on alluvial cassiterite, scheelite and wolframite concentration (by a total number of grains from ≈1000 samples) for the Segura-Salvaterra do Extremo-Zebreira (Sul) region. Subsequent numerical handling in GIS yields to generate HM abundance maps (Fig.1), highlighting the most conspicuous mineral anomalies.

(iv) The identification of information gaps to be fulfilled during the project, namely the completion of HM classification and separation (with a binocular microscope, assisted by U.V. lamp, hand magnet, chemical tests whenever needed, and handpicking), further complemented with comprehensive chemical characterisation of statistically representative mineral populations.

The WP1 also included an extensive rock sampling programme, during several field surveys. A collection of samples was obtained from critical and representative exposures of the main geological formations that were complemented with drill-core samples in the case of Panasqueira.

M2.1 (WP2), pertaining the activities related to sample preparation for elemental and isotopic analyses using the appropriate methods, was fully achieved. The work so far developed will be extended, in the following months, to an additional set of samples which represent specific features of several targets (mineralising systems).





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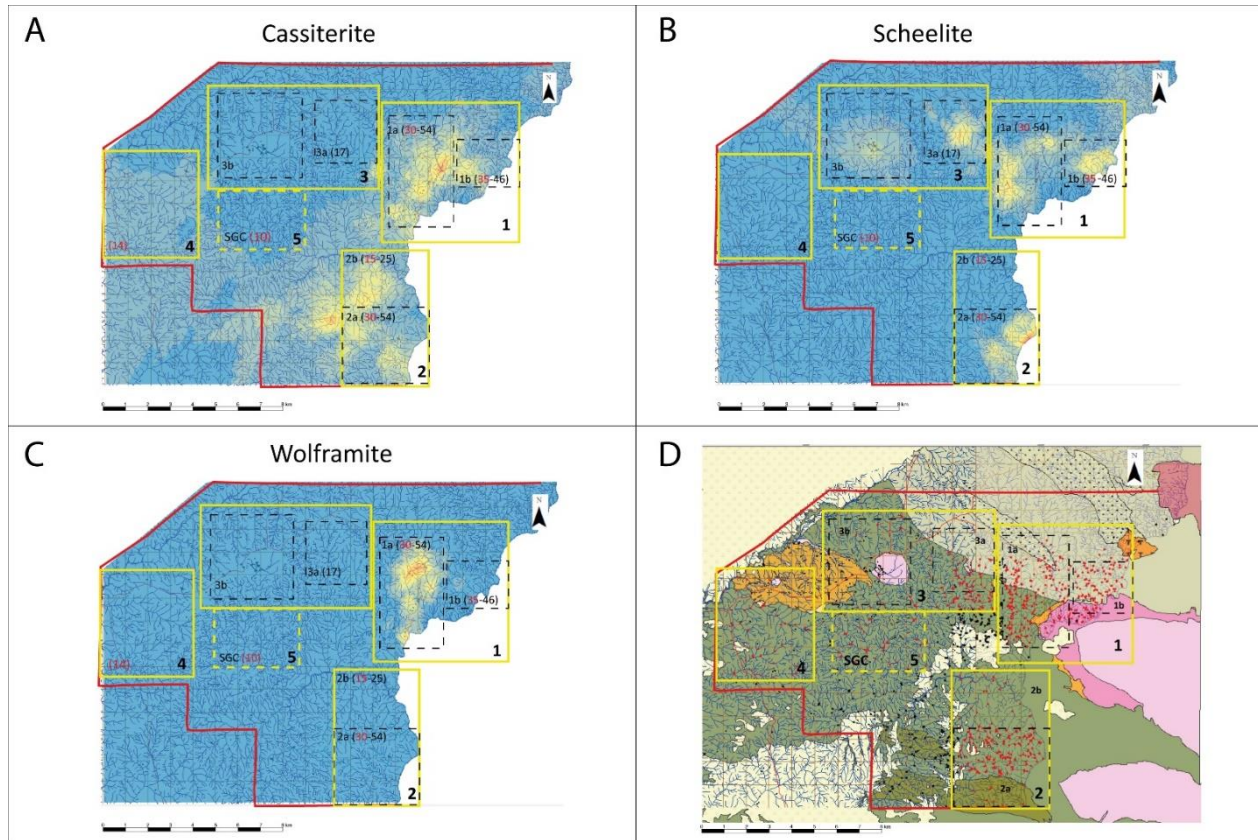


Figure 1: Map of alluvial (A) cassiterite, (B) scheelite and (C) wolframite grains concentration (increasing from yellow to orange) by total number of grains: up to 1930, 505 and 360, respectively, in Zebreira, Salvaterra do Extremo, Zebreira Sul and Segura. Based on 1005 (986 for cassiterite) samples (data from Inverno et al., 2007, and references therein, and Grácio, 2020, after Inverno et al., 2007). The visible (1 to 5) polygons were defined to control the mineral grain anomalies and include the number of samples foreseen for study. Polygon 1 was defined as the priority in this study and polygons 2, 3, 4 and 5 are complementary and dependent on the mineralogical composition of the samples and time spent during WP4. (D) Extract of the geological map, for the same region, produced by the MOSTMEG transnational project team; red dots correspond to sampling points.

M2.2 (Geochemical and isotopic analyses completed) was largely completed. Multi-element whole-rock analyses were obtained for 183 samples. Whole-rock, multi-system (Sr-Nd-Pb) isotopic analyses were concluded for 20 samples. The analytical work is currently in progress for 45 other selected samples (many of them are already prepared for spectrometric measurement). Zircon grains were also extracted from 18 samples of granitoid, aplite and pegmatite rocks representing different geological settings. Further studies, involving additional samples and other analytical methods, will be necessary to achieve the purposes of WP2 and WP3 by the end of the 2nd year, as initially planned.

Whole-rock major and trace element contents for a selection of 70 samples were obtained at Activation Laboratories, Ltd. (www.actlabs.com), using the analytical package 4E-research. Major oxide elements were analysed by ICP-OES. Trace and rare earth elements were obtained by ICPMS and INAA. F, B and FeO contents were measured with KOH-ion chromatography, Prompt gamma neutron activation analysis and titration, respectively. The results obtained for these 70 samples were subsequently used as in-house standards for the remaining 113 samples analysed with XRF at FCUL. Accuracy-related errors in XRF measurements were $\leq 5\%$ for major elements and better than 10% for the most widely used incompatible



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elements. Duplicate measurements also indicate reproducibility-related errors <5% in XRF analysis of both major and trace elements.

Zircon grains were mounted (together with the TEMORA standard) in epoxy to be examined, after polishing and Au-coating, with a FEI-QUANTA 250 scanning electron microscope equipped with secondary-electron and cathode-luminescence (CL) detectors, and further analysed with a sensitive high-resolution ion microprobe (SHRIMP-II). Uranium abundance and U/Pb ratios were calibrated against TEMORA and age calculations were performed with Isoplot software.

M3.1 (*Beginning of sample preparation/polished sections/mineral separation*; WP3) and **M3.2** (*Petrography & sample selection for subsequent analytical methods*; WP3), as well as **M4.1** (*Onset of alluvial sediment study*; WP4), were attained. More than 200 polished thin sections were produced, and are currently under examination. Several bi-polished thick sections were also produced to assist the study of fluid inclusions. Following the work done in task 1.3, the 104 samples of alluvial HM the from polygon 1 (Fig. 1, selected as a priority) were subjected to a preliminary (re-)examination under a binocular microscope to check the presence of TiO₂ polymorphs and mineral phases of interest; anatase and tourmaline are abundant, which is consistent with the available data for polygons 2 and 4. Mounts of TiO₂ polymorphs and tourmaline grains extracted from these mineral concentrates will be analysed with EPMA and LA-ICP-MS.

C1.2 Deliverables (D) accomplishment

Deliverables **D1.1** (*Preliminary Database*) and **D1.2** (*Stratigraphic/structural Map*), both related to WP1, are somewhat delayed, but they will be completed by the end of 2021, unless unpredictable setbacks occur in the meantime. Presently, only the information available for the area between Panasqueira and Góis is not processed, as justified in section C.2. The *Sampling Dataset* (**D1.3**) was completed in September after the densification of the sampling programme in some selected targets. This does not exclude the possibility of gathering additional samples on particular targets (such as Segura, Medelim, Mata da Rainha, Fundão and Argemela), during 5 to 6 specific/short-term field surveys, prudently scheduled for the period ranging from Nov2021 to March/April 2022.

The work required for **D2.1** (*Geochemical database*) and **D2.2** (*Isotopic database*), initially planned for month 15th, is progressing at a good rate with the available samples. Results will provide relevant background information for the Segura-Panasqueira area. Still, additional materials must be collected in the adjoining region, from Panasqueira to Góis, to address the objectives stated in the project proposal duly. Similarly, the work needed to accomplish **D3.2** (*Mineral and fluid chemistry databases*) and **D3.3** (*By-products database*), both scheduled for month 20th, is advancing with the samples collected at the end of 2019, but other critical information must be obtained by studying the samples recently picked (14th-17th Sept21) in various key targets.

C1.3 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. The preliminary geological map and interpretative cross-sections are shown in Fig. 2 and Fig. 3, summarising the results achieved in **tasks 1.1** (**Data compilation**) and **1.2** (**Regional and local tectonic/structural constraints**). The joint interpretation of data collected in previous studies and gathered during additional fieldwork under the scope of MOSTMEG shows that:

1. The cropping out, pre-Ordovician, sedimentary succession of the Beiras Group is recrystallised under P-T conditions of greenschist metamorphic facies and comprises two lithostratigraphic units, labelled as Malpica do Tejo (MT) and Rosmaninhal (R) Formations (F). The MTF, most likely Ediacaran in age, includes





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two main siliciclastic members that are distinguished by the relative predominance and thicknesses of middle-coarse grained greywackes over siltstones/pelites. The RF (late Ediacaran/basal Cambrian?) is composed mainly of monotonous pelitic succession. Considering the sedimentary facies, the RF can be separated into proximal (p) and distal (d) members. Siltstone layers of variable extension, thickness and grain size are increasingly abundant from the lower to the upper member of the proximal facies (Rp¹ and Rp²) and include centimetre-thick discontinuous conglomerate levels in Rp². The reconstructed cartographic pattern suggests a significant lateral facies change, from ESE to WNW, between Rp¹ and Rd. In addition, conceivable unconformities between MT and Rp¹/Rd (intra-Alcudian?) and below Rp² (basal Cambrian?), as well as the “Toledanic” unconformity at the Cambrian-Ordovician transition, can be inferred (Figs. 2, 3).

2. The metasedimentary succession is intruded by voluminous plutonic rocks and different arrays of dykes that can be separated by geometry (orientation and shape) and petrological nature. Metasediments and some granitoid facies preserve evidence of heterogeneous strain accommodation, recording the structural rearrangement of multi-scale effects (rock fabrics/foliations, folding, shear and fault zones, quartz veins and other discontinuities) due to the progression of regional deformation phases. The currently available field data does not exclude the possibility of existing pre-Ordovician structures and low-grade metamorphism (D_n/M_n). Nonetheless, many of the observed features may can be interpreted as miscellaneous effects of early-Variscan (Middle Devonian) deformation and metamorphism (D₁/M₁) overprinting pre-existent anisotropic fabrics generated during the transient rift-to-drift tectonic regime at the end of the Cambrian-Ordovician period and/or to orogenic processes in the Ediacaran (Cadomian orogeny). The later Variscan structures which include the NW-SE to WNW-ESE heterogeneous folds and the conjugated transcurrent shear zones that cut the post-315 Ma granitoids, correlate well with those typifying the Variscan D₃/M₃ regional deformation-metamorphic stage (upper Carboniferous).

3. Plutonic rocks can be grouped into three main suites. The first suite includes different rock types (clearly dominated by granodiorites, biotite quartz-diorites and tonalites) which represent the Lower Palaeozoic magmatic event (peaking at the Cambrian-Ordovician transition), concurrent with the extreme continental stretching related to the Rheic Ocean opening. The main plutons in this suite are Batão, Oledo – Idanha-a-Nova, Zebreira and Fundão. The other two plutonic suites, mainly composed of monzonitic granites (sometimes porphyroid and often bearing biotite and muscovite) but locally including significant leucogranites, document the multiphase emplacement of melts generated during the Variscan orogenic stages (primarily throughout the late Carboniferous and towards the early Permian). These suites comprise some major exposed plutons (such as Segura, Penamacor-Monsanto, Orca and Castelo Branco), besides the partly and completely concealed granite bodies related to the ore-forming systems of Argemela and Panasqueira, respectively.

4. Swarms of subvertical, mesocratic to mafic dykes running from N110°±5° to N121°±5° (mostly fine-grained granodiorites) and N102°±7° (micro-gabbros) occur in several locations, often nearby the Cambrian-Ordovician plutons, the most important exception being the cluster of quartz-diorite/granodiorite dykes labelled as Matos, which also includes a minor laccolith. Subvertical dykes of porphyry tonalites (N148°±5°), porphyry microgranites (N360°±12°) and microgranites (N119°±9°) are also common in the SW part of the study area, particularly in the vicinity of Zebreira where some dykes criss-cross the prevalent granodioritic and biotitic quartz-dioritic facies forming the pluton. Aplite dikes, sometimes occurring along with pegmatites, are mostly confined to the periphery of the Orca and Penamacor-Monsanto plutons, and to the metasedimentary envelope of the Segura granites, displaying a wide range of directions (although dominantly between NE-SW and ENE-WSW) and dips (from subvertical to low-angle, <40°, usually to the NW-WNW). Around the Oledo – Idanha-a-Nova pluton, but mainly on its western border, rhyolitic dykes can also be observed besides those of aplitic and microgranitic nature. All these rocks trace the protracted magmatic activity in the region during the Lower and Upper Palaeozoic periods, concurrently or after to the



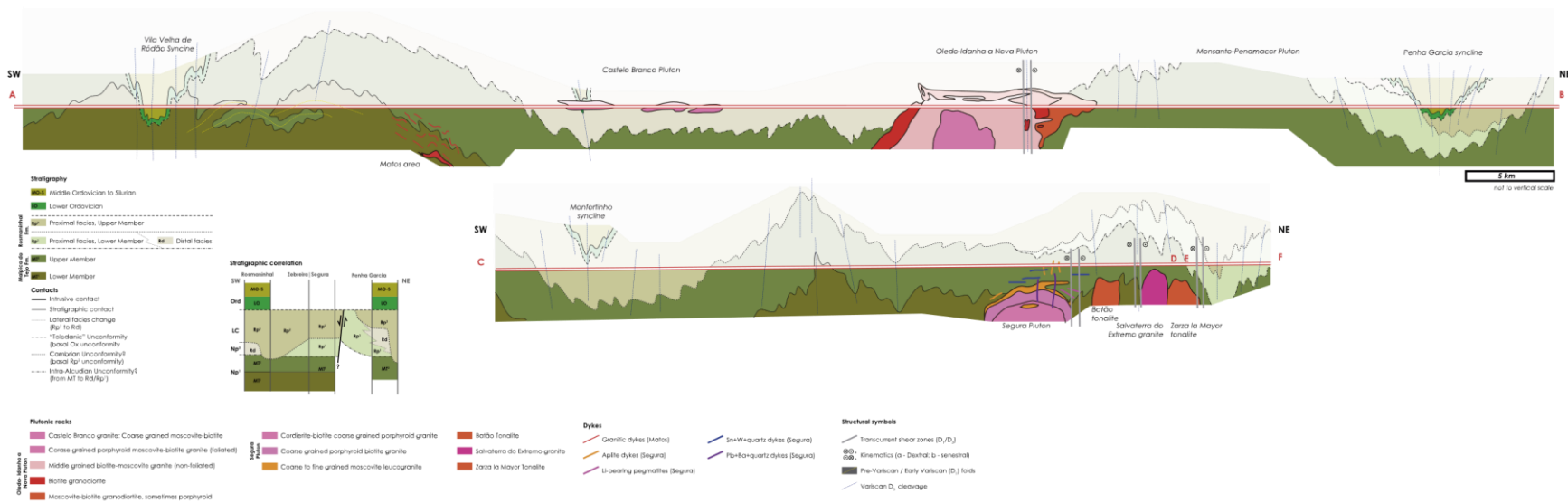
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Figure 3: Interpretative geological cross-sections (for location see the map in Figure 2).



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multistage crustal emplacement of silicate melts related to the development of the plutons as mentioned above. However, convincing mineralisation signs seem to be confined to the aplite/pegmatite arrays so far recognised near Monsanto and Segura, notwithstanding the interesting geochemical indications provided by some aplite bodies sampled elsewhere. In this regard, the geometric and chronological relationships between the aplite dykes distributed across the NE-border of the Orca pluton and the cluster of quartz veins to the S of Mata da Rainha will be investigated in the future, assuming that the latter group of structures represent satisfactorily the tungsten-rich lodes exploited in the past (São Francisco Mine).

5. Many subvertical transcurrent shear zones cut across the study area, mostly running ENE-WSW to \approx ESE-WNW and NNW-SSE to NW-SE, as observed throughout the Variscan orogen. Considering the macroscopic features displayed by these structural corridors, and their kinematics and spatial orientation, a straightforward comparison with the so-called syn-D₁ and syn-D₃ Variscan shear zones can be made. In general, the nucleation/development of these shear zones is related to strain partitioning within metasedimentary successions or along with their contacts with the Cambrian-Ordovician granitoids (at some stage in D₁) and thermo-mechanical contrasts with country rocks during the multiphase emplacement and cooling of Variscan granite bodies (roughly during D₃). So, the main shear zones are: (i) critical conduits for long-lasting crustal fluid flow, concomitant of strain accommodation, as indicated by copious arrays of quartz infillings, occasionally also bearing other accessory mineral phases such as sulphides and gold; (ii) significant mechanical weakening corridors that may influence the rising and emplacement of orogenic silicate melts (as verified for several dyke swarms); and (iii) central to the development of a wide range of subsidiary structures which might act as preferred loci for lode ore-forming processes of magmatic-hydrothermal nature (as likely represented in Mata da Rainha). According to the available data, the leading family of shear-related, subvertical quartz infillings runs $N67^{\circ}\pm 16^{\circ}$. Subsequent reactivation of several shear zone segments in Late-Variscan and Alpine times led to fault zones included in the regional network of strike-slip fault systems which, in the study area, are represented mainly by those oriented $N42^{\circ}\pm 10^{\circ}$. The tectonic inheritance and recurrent reactivation of several families of shear/fault zones explain also the late development of brecciated quartz-rich infillings mineralised with barite and galena ($N113^{\circ}\pm 11^{\circ}$) near Segura.

The purpose of **WP2** is the **vectoring of metal endowment and the definition of the critical timing for mineralisation triggering**. Substantial data collection is still needed to fully accomplish these objectives. Nevertheless, the multi-element whole-rock geochemical data so far obtained under the scope of **task 2.1 (Crustal protoliths for “productive/fertile” granite melts)**, complemented by the preliminary work done in **task 2.2 (Temporal development of “fertile” granite melts and related mineralisation)**, show that:

1. Pelites/siltstones of the MPF and RF are characterised by average concentrations of major (and some minor) elements comparable to those typifying fine-grained and highly mature siliciclastic sediments derived from intensely chemical weathered (median CIA value of 75) felsic to intermediate igneous sources related to continental island arcs. The most significant minor elements are Ba (511 ppm), F (449 ppm), Zr (196 ppm), V (152 ppm), Rb (113 ppm), Cr (101 ppm), Zn (101 ppm), along with trace amounts of B (94 ppm), Li (66 ppm), Ce (61 ppm), Sr (58 ppm), Ni (42 ppm), Cu (32 ppm), Y (29 ppm), La (28 ppm), Ga (22 ppm), Sc (19 ppm), Pb (14 ppm), Nb (12 ppm) and Th (10 ppm). The Average Shale-normalised patterns for major, minor and trace elements are characterised mainly by positive anomalies of Li, Cs, Sn, Hf, Bi, As and Sc, and negative anomalies of Mg, Mn, Ca, S, F, Sr, Nb, Ta, W, Th, U, Y, Ni, Mo, Cd, Cu, Pb, Ge and Ag. These compositional features (Fig. 4) are similar to those reported in literature for lateral equivalent metasedimentary successions in Spain. However, the higher Na₂O and CaO concentrations (often up to 3 wt% and 1 wt%, respectively) in the Spanish sector, and their negative correlation with Al₂O₃ contents, should reflect a mineral assemblage with a higher abundance of plagioclase that possibly signs a closer



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spatial relation with the source regions: a Cadomian island arc, which remnants are located to the south, at present coordinates.

2. The Cambrian-Ordovician plutons are mainly composed of weakly peraluminous I-type (mean ASI value of 1.1), biotite/biotite>muscovite tonalite to granodiorite rocks. These facies have moderate contents of SiO₂ (mean value of 68 wt%), total alkalis (Na₂O+K₂O mean value of 6.85 wt%) and a mean Fe* ratio [(FeO_(t))/(FeO_(t)+MgO)] of 0.66. They follow the compositional features of calcic to calc-alkalic series and magnesian granites, displaying condensed differentiation trends from diorite to normal granite compositions, as observed in many suites of volcanic arc granitoids.

3. Samples representing the Variscan magmatic events are highly peraluminous S-type (median ASI value of 1.26), muscovite>biotite / biotite>muscovite monzogranite to granite rocks that exhibit high median values of SiO₂ (73 wt%), median total alkalis (8.12 wt%) and a median Fe* ratio of 0.8. The compositional attributes of these rocks match those typifying calc-alkalic to alkali-calcic series and magnesian to ferroan granites developed in syn-collisional settings, often recording strongly differentiation paths.

4. The Upper Continental Crust-normalized multi-element patterns for the Cambrian-Ordovician and Variscan granitoids are characterized mainly by positive anomalies of Li, B, Rb, Cs, Ta, Sn, W and U, and negative anomalies of Ba, Sr, Zr, Hf and Th, usually much more distinct for the Variscan samples. The latter rock suites also show positive anomalies in F, Be and Nb, and negative anomalies of Y.

5. Most granitoid samples contain less than 30 ppm Sn, with a range from 30 to 100 ppm in the Segura, Salvaterra do Extremo and Panasqueira granites, as well as in the highest-differentiated granite facies of the Castelo Branco pluton. Tin contents are considerably higher (~400 ppm) in a late leucogranite facies (Medelim) of the Penamacor-Monsanto pluton, rising up to ~1000 ppm in the Argemela leucogranite. These metal grades co-vary positively with F concentrations, which largely exceed 1000 ppm and 2000 ppm in the Medelim and Argemela leucogranites, respectively (Fig. 5).

6. Tungsten contents ranging from 10 to 20 ppm were measured in some samples of granite facies from Orca, Castelo Branco, Panasqueira and Argemela. These values were exceeded only in the two analysed samples representing the Capinha granite (20-30 ppm W).

7. Significant Li contents (100-500 ppm, occasionally up to ~700 ppm) characterise the Variscan granites from Castelo Branco, Segura, Panasqueira, Penamacor-Monsanto, Orca and Capinha. Most Argemela leucogranites are still more Li-rich with values up to ~5000 ppm Li (Fig. 5).

8. Preliminary geothermometric estimates based on zircon solubility indicate higher crystallisation temperatures (> 750°C) for the Cambrian-Ordovician granodiorites, biotitic quartz-diorites and tonalities, as well as for several Variscan monzogranite and granite facies from Castelo Branco and Penamacor-Monsanto plutons. The lowest crystallisation temperatures are found for the Panasqueira (750 – 650°C) and Argemela (660 – 615°C) granites. In addition, considering the Al₂O₃/TiO₂, CaO/Na₂O, Rb/Sr and Rb/Ba ratios, most of the Cambrian-Ordovician granitoids plot within the greywacke/igneous source compositional field, except for the highest fractionated facies of the Zebreira, Oledo – Idanha-a-Nova and Fundão plutons which spread in the metapelite-derived melting compositional field together with all the Variscan granite suites. Therefore, exploratory modelling was performed using the modal mineral abundances for greywackes and pelites (the main difference being their relative abundance in micas and feldspars) and the batch (partial) melting and Rayleigh fractionation equations, taking into account the elemental partition coefficients compiled from several studies for peraluminous melts.



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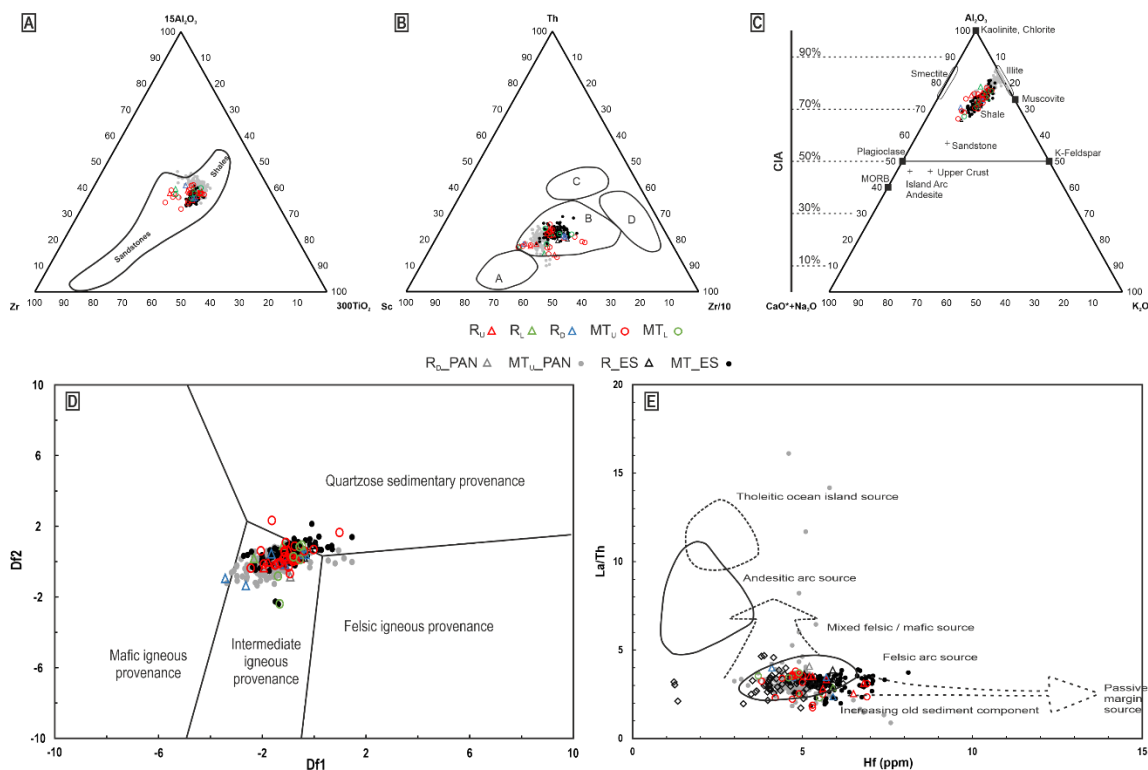


Figure 4: (A) Ternary 15Al₂O₃ – 300TiO₂ – Zr plot evidencing the compositional maturity of the Beiras Group metasedimentary rocks [Rosmaninhal Fm. (R) upper (u), lower (l) and distal (d) members; Malpica do Tejo Fm. (MT) upper (u) and lower (l) members; samples picked in the Panasqueira area (PAN) and in lateral equivalent metasedimentary successions in Spain (ES)]. The solid lines delimit the compositional field of clastic sedimentary rocks (Garcia et al., 1994); (B) Sc – Th – Zr/10 ternary discriminating plot of tectonic settings for the Beiras Group metasedimentary rocks (adapted from Bhatia & Crook, 1986). A – Oceanic Island Arc, B – Continental Island Arc, C – Active Continental Margin and D – Passive Continental Margin; (C) Ternary CN-A-K plot illustrating the spreading of CIA values of the Beiras Group rocks (adapted from McLennan et al., 1990; Goodfellow et al., 2003); Provenance diagrams for metasedimentary rocks using: (D) the discrimination function for major elements ratios from Roser & Korsch, 1988 ($Df1 = 30.638TiO_2/Al_2O_3 - 12.541Fe_2O_3(total)/Al_2O_3 + 7.329MgO/Al_2O_3 + 12.03Na_2O/Al_2O_3 + 35.402K_2O/Al_2O_3 - 6.382$; $Df2 = 56.500TiO_2/Al_2O_3 - 10.879Fe_2O_3(total)/Al_2O_3 + 30.875MgO/Al_2O_3 - 5.404Na_2O/Al_2O_3 + 11.112K_2O/Al_2O_3 - 3.89$); and (E) Hf vs. La/Th trace elements ratios (after Floyd & Leveridge, 1987). For comparison, the composition of stoichiometric minerals (in C), average crustal rocks and data from the Panasqueira Mine samples (PAN) and lateral equivalents to the Beiras Group in Spain (ES) are plotted.



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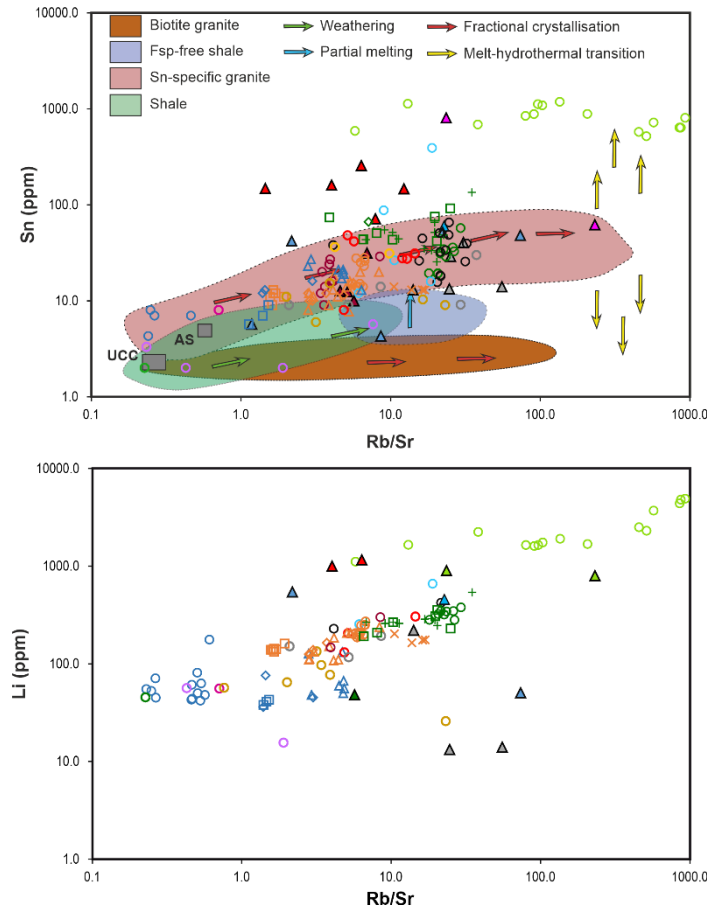
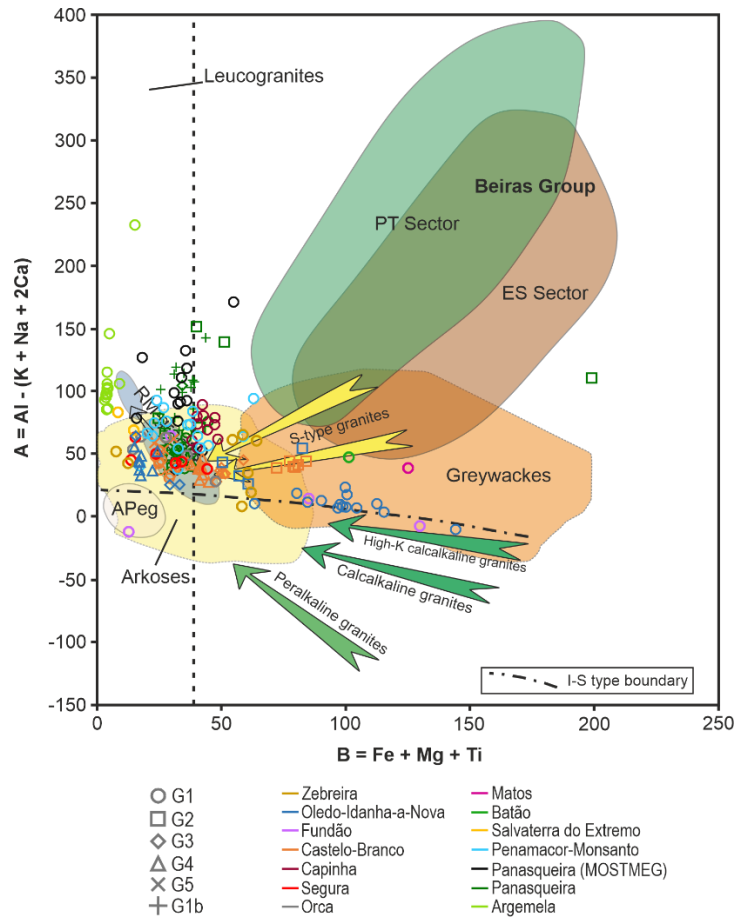


Figure 5: (Left) Distribution of the analysed granitoid samples (excluding dykes) in the B-A diagram of Debon and Lefort (1988), where the compositional fields defined for fine-grained metasediments from the Beiras Group are also indicated. (Right) Rb/Sr vs Sn (Romer, 2020) and vs Li cross-plots illustrating the Sn and Li increase with differentiation for Variscan granites and leucogranites, often followed by aplite/pegmatite dykes (triangles).



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Modelled contents of Rb, Ba and Sr are comparable with the concentration values obtained for these elements in most of the granitoid facies cropping out in the Segura-Panasqueira area. However, there is a significant deviation between modelled and observed abundances of Li, Nb, Sn and Ta, even considering the compositional heterogeneity of the metasedimentary sequences. These discrepancies could result from: (i) underestimation of Nb compatibility and overestimation of Li, Sn and Ta compatibility; and/or (ii) the non-evaluation of the influence of other plausible processes, such as hydrosaline melt immiscibility, high-T alkaline metasomatism and/or late interaction with aqueous hydrothermal fluids. Using the Nb/Ta and K/Rb ratios to separate peraluminous granites affected by significant magmatic-hydrothermal transformations (<5 and <150 , respectively), one may conclude that the majority of the analysed granitoid facies should have been subjected to some kind of compositional readjustment, leading to a progressive enrichment in lithophile elements such as Li, Nb, Sn and Ta. Such a trend is consistent with indications provided by Nb/Ta and Zr/Th ratios, which show in addition that many of the analysed Variscan granite facies spread over the empirical field of Sn-W(-U)-related granites and transition to that of rare metals (Ta-Cs-Li-Nb-Be-Sn-W) related granites, where the Argemela leucogranites plot. Consequently, as future work, improved models of partial melting and fractional crystallisation will be assessed for complex systems, involving interactions between crystals, silicate melts and immiscible aqueous fluids (hydrothermal fluids and hydrosaline melts).

9. Considering the geochemical indications so far assembled, the Variscan granite suites are clearly more fertile than those of the Cambrian-Ordovician age (Fig. 5). Among the Variscan suites, the strongly differentiated and ferroan leucogranites from the Penamacor-Monsanto and Segura plutons trace the most promising targets recognised up to now. For example, the lithium-rich (>1000 ppm Li) aplite/pegmatite samples of Segura display up to 2900 ppm F, 84 ppm Nb, 67 ppm Ta and 256 ppm Sn. The granitic facies of the Penamacor-Monsanto and Segura plutons are characterised by degrees of differentiation (Rb/Sr up to 18.93 and 14.53, respectively) and metal enrichment (up to 391 and 48 ppm Sn, 661 and 305 ppm Li, 27 and 33 ppm Nb, 9 and 10 ppm Ta, respectively) close to those typical of granites related to ore-forming processes at reference places such as Panasqueira (W-Cu) and Argemela (Sn-Li). In Argemela, concentrations of 1500 ppm F, 87 ppm Nb, 114 ppm Ta and 800 ppm Sn are common in lodes displaying up to 900 ppm Li.

10. Zircons extracted from some aplite dykes (such as the case of Gf_MDCH#1 spatially related to the Oledo – Idanha-a-Nova pluton) display evidence of disturbances similar to those caused by metamict (radiation-damage) transformations. In other cases (such as the porphyry dyke and laccolith represented by Gf-Furão#1 and G_MARCELINA#1 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%, respectively), which created additional challenges during the analytical work and increased the uncertainties in age estimation. Even so, the preliminary data so far obtained: (i) confirm the Cambrian-Ordovician ages of the main granitoid facies forming the Batão (508 ± 6 Ma), Fundão (499 ± 5 Ma) and Zebreira (488 ± 3.4 Ma) plutons, disclosing also inherited zircon populations in the former two cases with 554 ± 7 Ma and 527 ± 6 Ma ages; (ii) point to a tentative age of ca. 474 Ma for the quartz-diorite/granodiorite dykes at Matos; (iii) indicate a crystallisation age of 311.2 ± 2.8 Ma for the peripheral granite facies of the Penamacor-Monsanto pluton; and (iv) provide an age of 299.9 ± 7.2 Ma and 306 ± 4.6 Ma for the late-emplaced porphyry dykes and laccoliths nearby Zebreira, respectively, similar to the ages estimated for the internal granite facies of the Orca pluton (304 ± 3 Ma), the pegmatite dykes of Monsanto (308 ± 3 Ma) and the Li-rich aplite/pegmatite lodes of Segura (308 ± 5 Ma).

Additional work will be needed to better constrain some of the ages up to now estimated, implying the extraction of more zircon grains (with different grain sizes and from other samples) and enlarge the database



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for newly formed and inherited zircon populations already detected in: (i) the Salvaterra do Extremo granite (only inherited zircon ages clustering at 559.6 ± 4.1 Ma?); (ii) the Segura granites (ranging from 310 to 300 Ma, besides inherited zircon clusters at ca. 320 Ma and 373 Ma); (iii) the Medelim leucogranite, intruding across the contact between the two main facies of the Penamacor-Monsanto pluton (poorly represented ages around 306 Ma in addition to an inherited zircon population dated of 378.4 ± 4 Ma); (iv) and one of the granite facies sampled at Panasqueira (ill-documented ages of ca. 305-310 Ma, along with zircon populations interpreted as inherited and dated of ca. 320 Ma, 382.5 ± 6.5 Ma, and 532 ± 8.4). Future work will also explore the geological/geochemical meaning related of the U enrichment (co-varying with ^{204}Pb increase?) in zircons included in rocks affected by ore-forming processes.

The main objective of **WP3** is to **investigate the relevant processes for metal concentration and deposition at 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 main results obtained can be summarised as follows:

1. The chemical analyses of granites, aplites and pegmatites collected from previous works on Segura and Argemela (Antunes et al. 2013, Michaud et al. 2020), were re-processed and handled as the data from Panasqueira granites in Marignac et al. (2020). Aplite and pegmatite veins from Segura are slightly less siliceous than the Panasqueira granites, which are highly siliceous (72.5 and 75 wt% SiO_2). For the Segura facies, SiO_2 contents range from 72.2 to 74.0 wt% in granites and 73.3 to 74.8 in aplite-pegmatite rocks, and therefore more siliceous than Argemela facies. The Segura granites display intermediate features between the rather albitic facies from Argemela (66-72% SiO_2) and the most evolved and albitic Panasqueira granites. The $\text{K}_2\text{O}/\text{Na}_2\text{O}$ ratios range between 0.7 and 1.2, less than recorded by the Panasqueira granites (0.5 and 3.1), and higher than the values for the Argemela granites (0.2 to 0.7). The enrichment in Na_2O depicts the differentiation and the more albitic character of the evolved magmas (Fig. 6a).

Mafic components are low to very low. The $\text{FeO}+\text{MgO}+\text{TiO}_2$ sum, ranging from 0.14 to 1.40 wt%, is much lower than in Panasqueira granites (1.71 – 3.12 wt%) and similar or higher than in Argemela granites (0.03 – 0.19 wt%). All the rocks are depleted in magnesium, with the M index ($M=100\text{Mg}/\text{Mg}+\sum\text{Fe}$) between 8 and 45, similar to Panasqueira (15 to 31) and Argemela granites (6 to 46).

All the rocks are distinctly peraluminous, as seen in the $\text{Al}-(\text{K}+\text{Na}+2\text{Ca})$ vs. $\text{Fe}+\text{Mg}+\text{Ti}$ diagram of Debon and Lefort (Fig. 6b). At Panasqueira, the peraluminous parameter $\text{Al}-(\text{K}+\text{Na}+2\text{Ca})$ is, however, increased by alteration effects, very obvious for the most altered facies and perceptible for many of the less altered granite facies (Marignac et al., 2020).

The Segura aplite and pegmatites veins are significantly enriched in phosphorous, with P_2O_5 contents in the 0.36 to 2.23 wt% range, higher than in the Panasqueira granites (0.23 to 0.74 wt%). Compared to the Panasqueira granites (which are dominated by a strong correlation between phosphorous and calcium content, indicating that both elements are entirely incorporated in apatite, Fig. 6c), the Segura aplite-pegmatite veins are characterised by an excess in phosphorus, not linked to apatite. The data plot to the right of the apatite line indicate that, for a given calcium content, the phosphorus is linked to another phosphate (Li, Na or another element, Sr, for instance, dominated phosphate) than apatite.

The magmatic trends are clearer at Segura and Argemela where the parameter A increases at decreasing B, as the parameter Q (silica not linked to feldspars, e.g. quartz) dops at decreasing P (thus, at increasing albitic character) – Fig. 6 d.

2. The mineralogy of the Segura aplite and pegmatite dykes is quite diverse.





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Cassiterite grains in the aplite-pegmatites from the northern zone to the northeast of the Segura granites are euhedral with limited zoning and do not show extensive replacement. They formed, at least, later than the earliest generation of Nb-Ta phases, which they enclose as euhedral grains (Fig.7a,b, c, d).

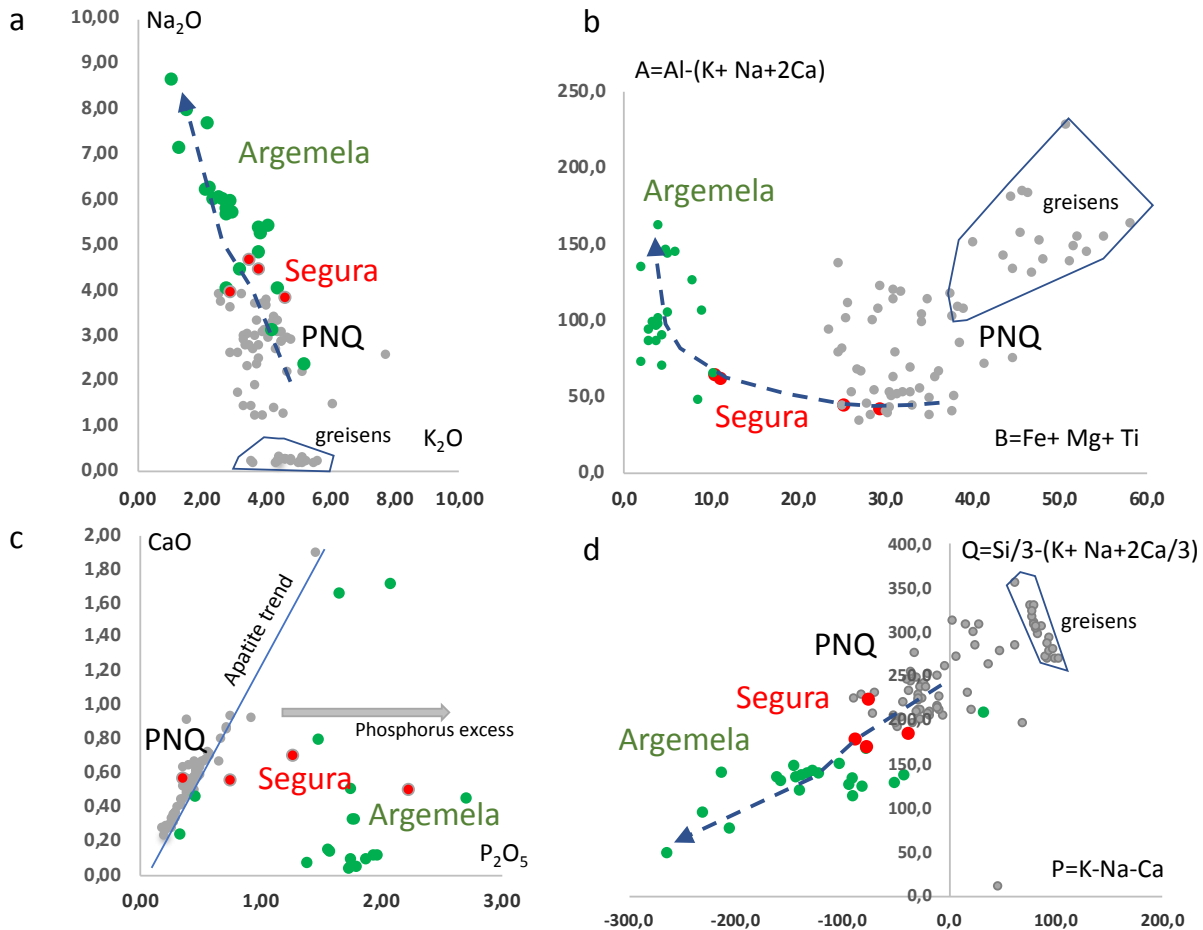


Figure 6: Major element geochemistry of the Segura, Panasqueira (PNQ) and Argemela granite suites; data from Antunes et al (2013), Michaud et al. (2021) and Marignac et al. (2020). **(a):** The Na₂O vs K₂O (wt. %) diagram, showing sodium enrichment with differentiation degree, and leaching in the greisens. **(b)** A-B diagram from Debon and Lefort (1988) showing the decrease of the parameter B as a function of A, a differentiation marker. In most Panasqueira granites, the A parameter is increased by greisenisation process. **(c)** CaO vs P₂O₅ (wt. %) diagram, showing that calcium is essentially incorporated in apatite at Panasqueira but is partly accommodated in other phosphates (including Li phosphates) at Argemela and Segura. **(d)** Q-P diagram showing the enrichment in albite at decreasing amounts of quartz, as well as the quartz + muscovite pseudo-greisen trend. The dashed line depicts a possible magmatic differentiation trend.

Nb-Ta and Sn oxides: columbite-tantalite mainly was observed in the aplite-pegmatites from the northern zone, as individual grains dispersed in aplite-pegmatitic masses. They are not exsolved grains in or from cassiterite, as presented by Antunes et al. (2013). Euhedral grains of Nb-Ta phases vary from few 10 μm to ≥ 100 μm in length, and display spectacular zoning, mostly oscillatory zoning (OZ) on BSE imaging. Three main rims with internal OZ are observed in the overgrowth. The Nb-Ta phases contain a significant amount of W (4 to 5 % WO₃), Mn (10-12%) and are characterised by large oscillations in Nb and Ta (Fig. 7e, f).



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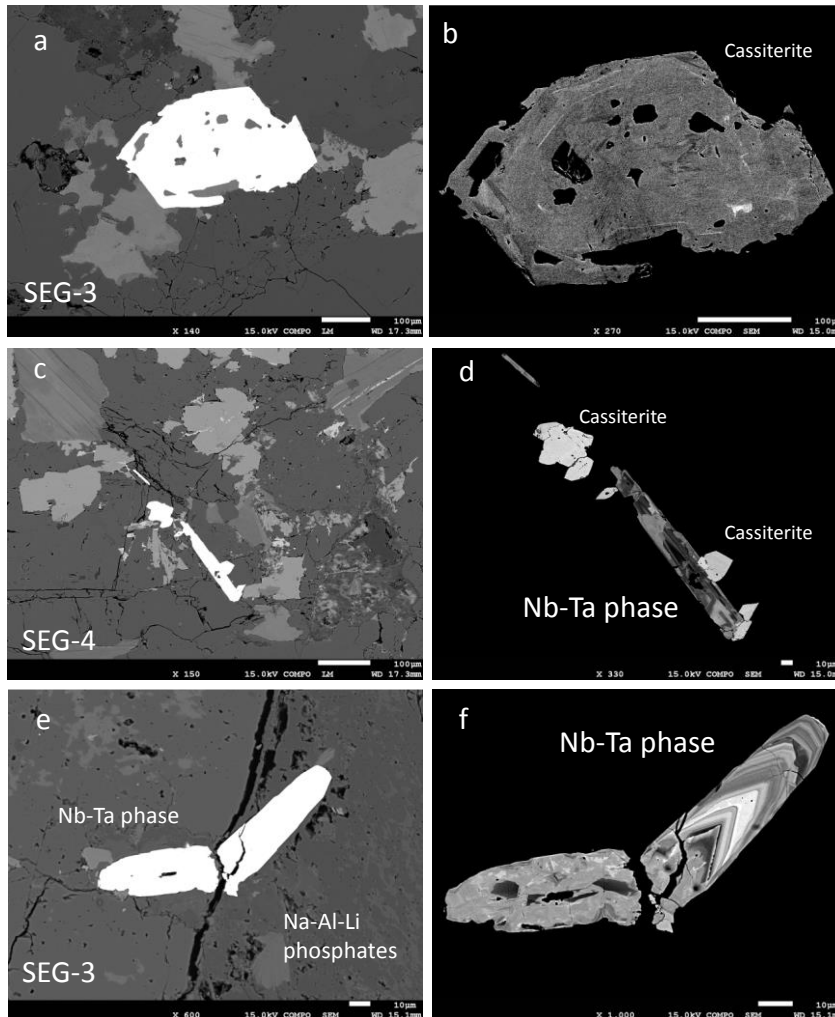


Figure 7: Sn and Nb-Ta phases in Segura aplite and pegmatites. SEG3 **a, b**: cassiterite grain; euhedral with inclusions, **b** reveals the growth bands which are not discriminated by significant substitutions, SEG 4 **c, d**: Nb-Ta phase with euhedral cassiterite crystallised its surface, indicating that cassiterite saturates later than Nb-Ta phase in the magma; SEG 3 **e, f**: Nb-Ta phase crystallised first together with a-quartz and feldspars, showing a zoned overgrowth crystallised in a cavity now filled with Na-Al-Li phosphates (assemblage lacroixite-montebbrasite).

Phosphates and Li-bearing phases: there are two main Li bearing phases, lepidolite and Li (Na, Al)-phosphates. Lepidolite was not studied in detail yet. The main focus was the Li-phosphate as a significant enrichment in phosphorus over what can be ascribed to apatite is obvious from Fig. 6c. The methodology used is original and is based on mapping of phosphorus by micro-XRF, at the thin-section scale, followed by semi-quantitative study of the phosphates by SEM equipped with WDS, which, after due calibration, may provide accurate analyses (Fig. 8).

The phosphates display complex relationships, as the early phosphate phases may be dissolved and replaced by a succession of later phosphates:

- The earliest phosphate phases (Li-Al) occur preferentially formed as large euhedral crystals, sometimes centimetre-sized prisms oriented perpendicular to the aplite layers and growing towards the core of the pegmatite veins. (Fig. 8). They are associated with topaz (Fig 8d);
- They are replaced on a small scale by a pervasive Na-Al-Li phosphate, shown to be a mixture of the two phases (montebbrasite, lacroixite), at the microscale (Fig. 8e);



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- Ca-Sr phosphates crystallise later on, at the expense or onto the two early Li-(Al, Na) phosphates (Fig. 8).

Besides Li-(Al, Na) phosphates, Fe-Mn (Al) phosphates have crystallised as isolated grains, apparently relatively early, as isolated grains (Fig. 8c, g). They show a succession of overgrowths with significant changes in their Fe/Mn ratio. The external rim grows in micro-fissures developed nearby when they had almost no more space to grow (Fig. 8).

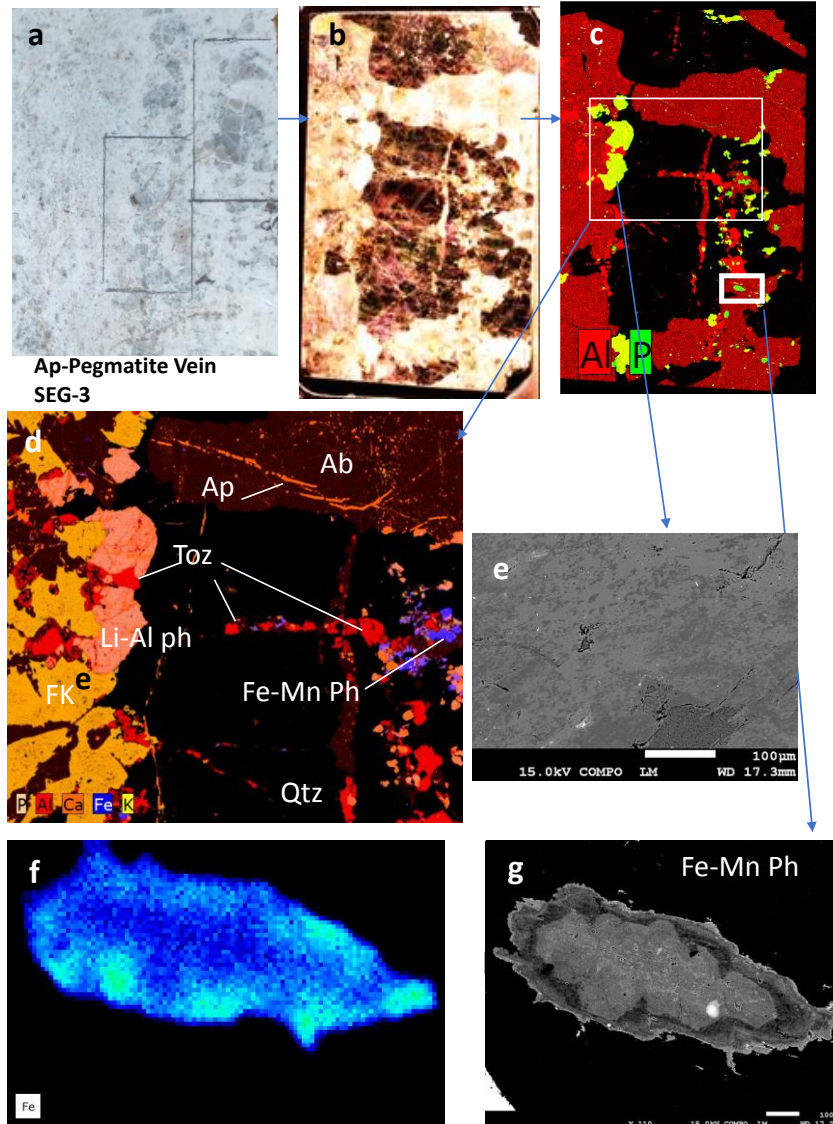


Figure 8: Distribution of major mineral phases in microXRF maps (example of sample SEG 3). **(a)** Macro-photograph of the aplite and pegmatite layers. **(b)** Thin section; **(c)** microXRF map showing most phases: in black: quartz, in red: feldspars (undifferentiated). **(d)** Detail showing the relationships with Li phosphates and topaz (in red), growing onto K-feldspar towards the cavity later filled by quartz. Fe-Mn phosphates are dispersed (blue phases) in quartz. **(e)** Detail of the lacroixite (light grey)-montebrasite (grey) relationships. **(f)** and **(g)** Map of the Fe-Mn phosphate grain from map c), showing a series of overgrowths onto the euhedral crystals. Ap: apatite, Ab: albite, Li-Al ph: LiAl phosphates, Fe-Mn Ph, Fe-Mn phosphates, FK: K-feldspar, Toz: topaz, Qtz : quartz.



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Fine-grained **tourmaline**-bearing aplites to the south and north of Segura also show abundant (Na-Al, and Fe-Mn) phosphates, as small grains dispersed within the rock, in addition to apatite. A future goal will be to establish a paragenetic sequence for these to establish a paragenetic sequence of the phosphate phases and to understand why two distinct phosphates crystallised in addition to apatite, in all facies at the magmatic stage.

3. Petrography, microthermometry and Raman micro-spectrometry were carried out on fluid inclusions (FI) preserved in different quartz aggregates forming a selected group of samples from the Segura aplitite-pegmatite dykes: Q1A, large crystals of clear but fractured quartz; Q1B, a subhedral clear mosaic of quartz slightly recrystallised along the borders; and Q2, anhedral clear quartz with some recrystallisation (Fig. 9).

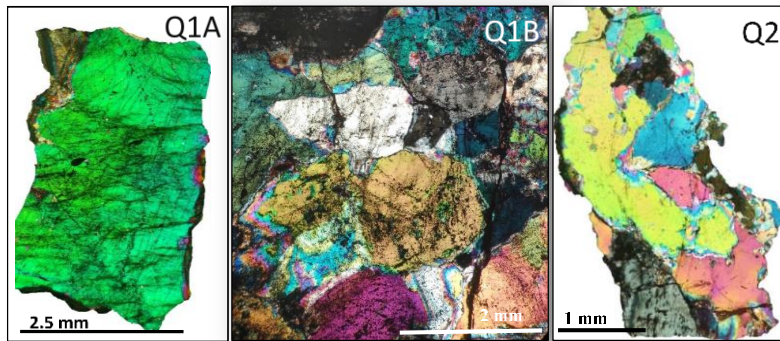


Figure 9: General features of the investigated quartz types.

According to the observed characteristics and following the classification scheme of Boiron et al. (1992), several types of FI were identified: (i) Lw-c, Lc-w, occurring isolated or forming clusters in Q1A and Q1B, and Vc-w and Vc occurring in clusters and fluid inclusion planes (FIP) in Quartz Q1A and Q1B (Fig. 10A); (ii) Lw-c, Lw-m, occurring isolated or developing clusters in Q2; and (iii) Vm-w and Vm occurring as clusters and within FIP in Q2 (Fig. 10B).

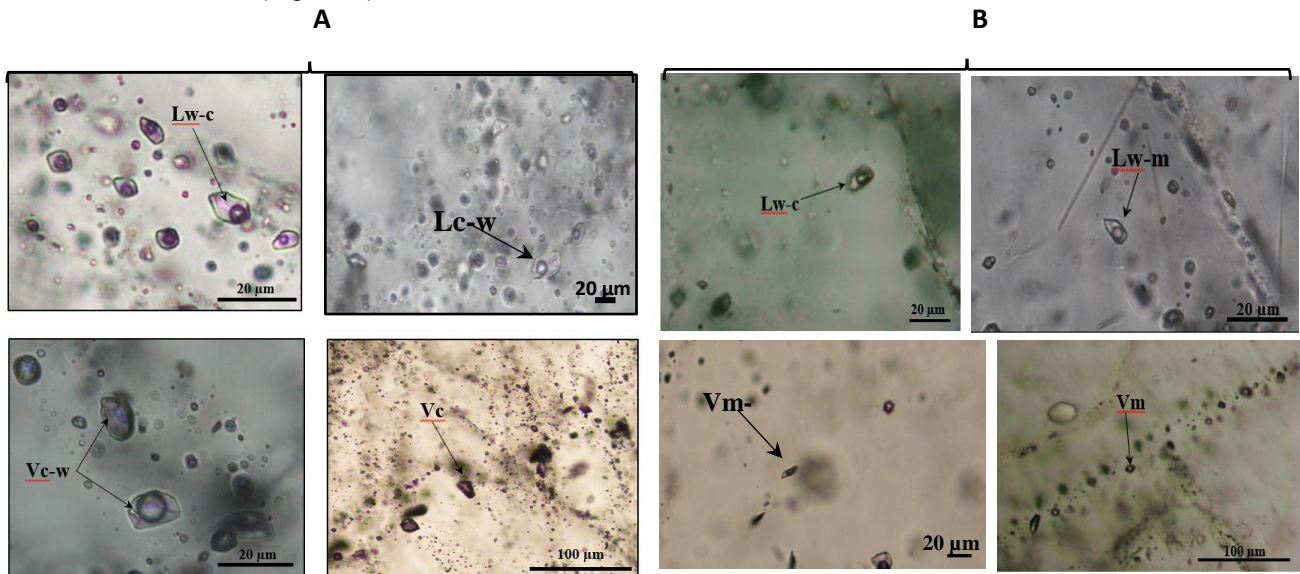


Figure 10: Fluid inclusions preserved in Q1A and Q1B (A) and Q2 (B) from the studied samples.



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Results from exploratory FI microthermometry and Raman micro-spectrometry can be summarised as follows:

- The volatile phase composition of fluids preserved in Q1A and Q1B is dominantly constituted by CO₂ (58 to 89 mol%) with minor amounts of CH₄ (3 to 22 mol%) and N₂ (5 to 24 mol%), whereas in Q2 most of the fluids trapped are mainly composed by CH₄ (51-82 mol%) with amounts of N₂ between 18-49 mol%, excluding those in Lw-c FI (Fig. 11);
- Except for the CO₂-(CH₄-N₂)-rich vapours (Vc FI) and the CH₄-vapours (Vm FI) the main fluid component is water (89.3 to 95.5 mol%), accompanied by minor CO₂, CH₄, N₂ and salt (Fig. 12);
- Using the molar volume, composition data and equations of state, isochores for representative FI within Q1A, Q1B and Q2 form three main clusters (Fig. 13).

Despite these results, it should be noted that, in all the examined samples, most FI are either decrepitated or occur as late FIP dominated by mono-phase methane or carbon dioxide-rich inclusions of low density (Fig. 14). Therefore, it is challenging to place precisely the few bi-phase fluid inclusions in the sequence of fluid events. They are probably earlier than mono-phase low density volatile dominated inclusions. In the southern zone of Segura, only late volatile-rich vapours were found in the samples studied at Nancy.

The significance of the trapped fluids is, at present, difficult to establish. Inclusions can correspond to relatively late fluid trapping after a phase of tectonic deformation after dyke emplacement during which most primary FI have been destroyed. The presence of both aqueous carbonic fluids and methane-rich low-density fluids may indicate that both late fluids were still equilibrated with host metamorphic rocks when they percolated the aplite and pegmatite dykes. Aqueous carbonic fluids may reflect the P-T-X conditions during or after the intrusions of the sills. Volatile vapour FIP's may represent a late event once the system had been decompressed, indicating that the predominant fluid in the host rock at that time was still issued from graphite-water equilibrium (or disequilibrium).

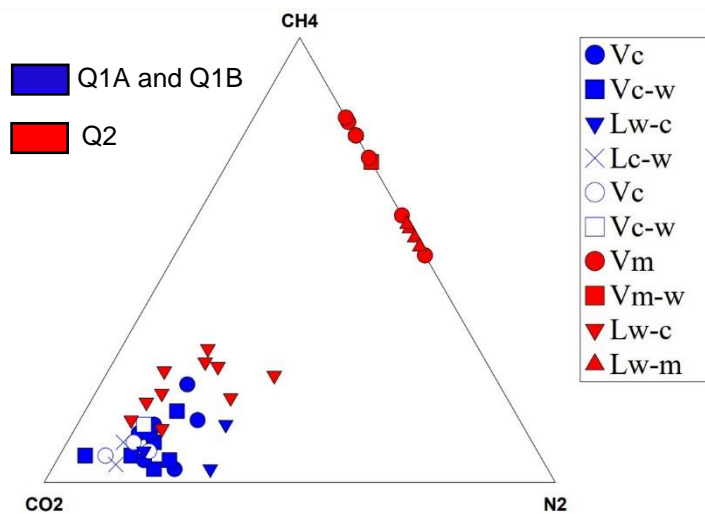


Figure 11: Ternary plot in the CO₂-CH₄-N₂ diagram of the volatile phase composition for the different FI.





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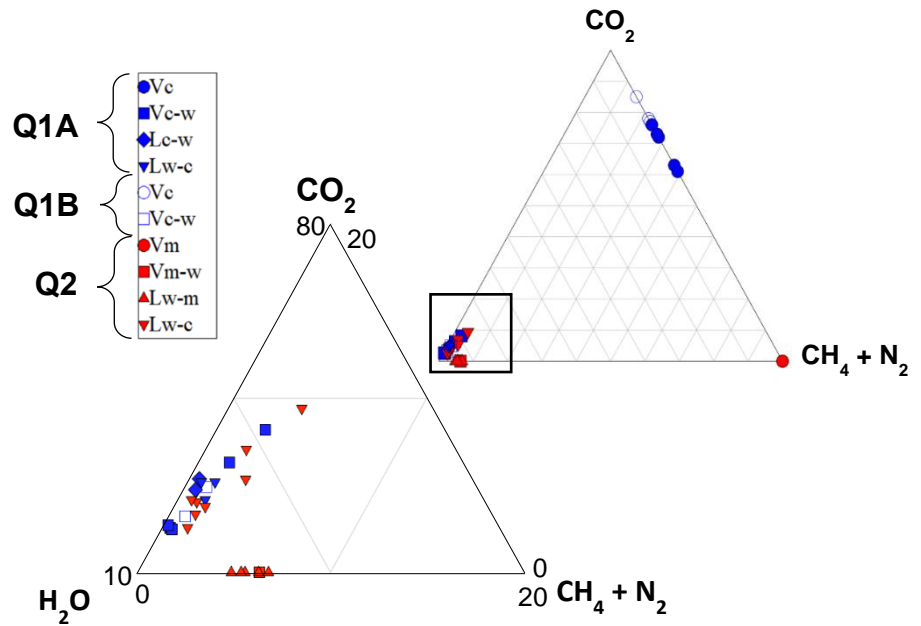


Figure 12: Ternary plot in the H₂O–CO₂–CH₄+N₂ diagram of bulk composition for different FI.

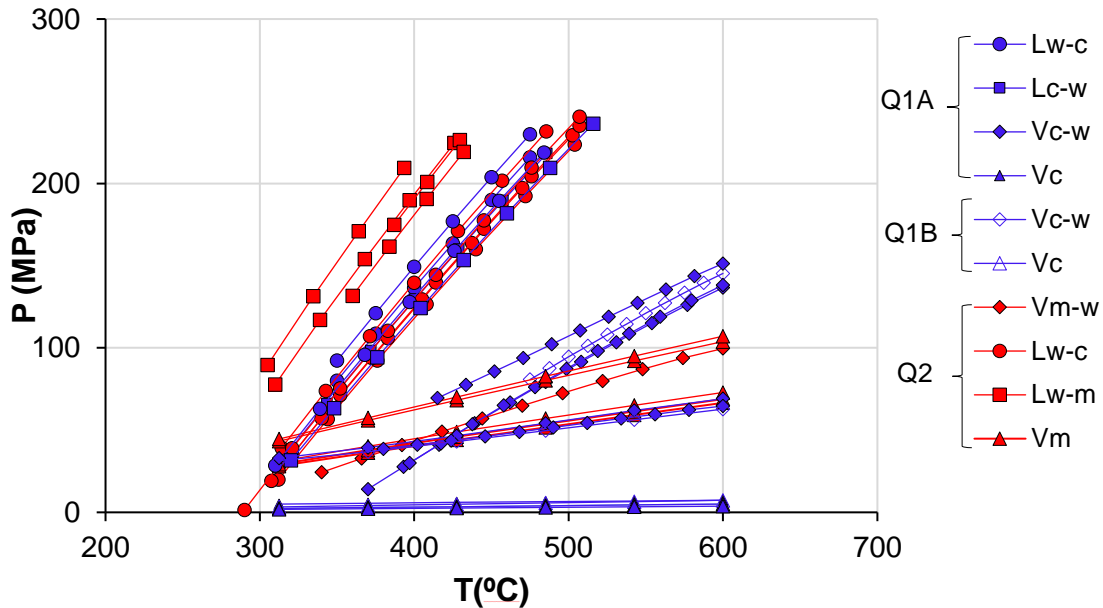


Figure 13: Isochores for FI preserved in Q1A, Q1B and Q2, using total homogenisation temperature (Th) as minimum trapping temperature conditions. P-T pairs of fluid trapping are somewhere along the isochores, and will be defined in the next step by considering independent geothermometric considerations and potential geothermal gradients.



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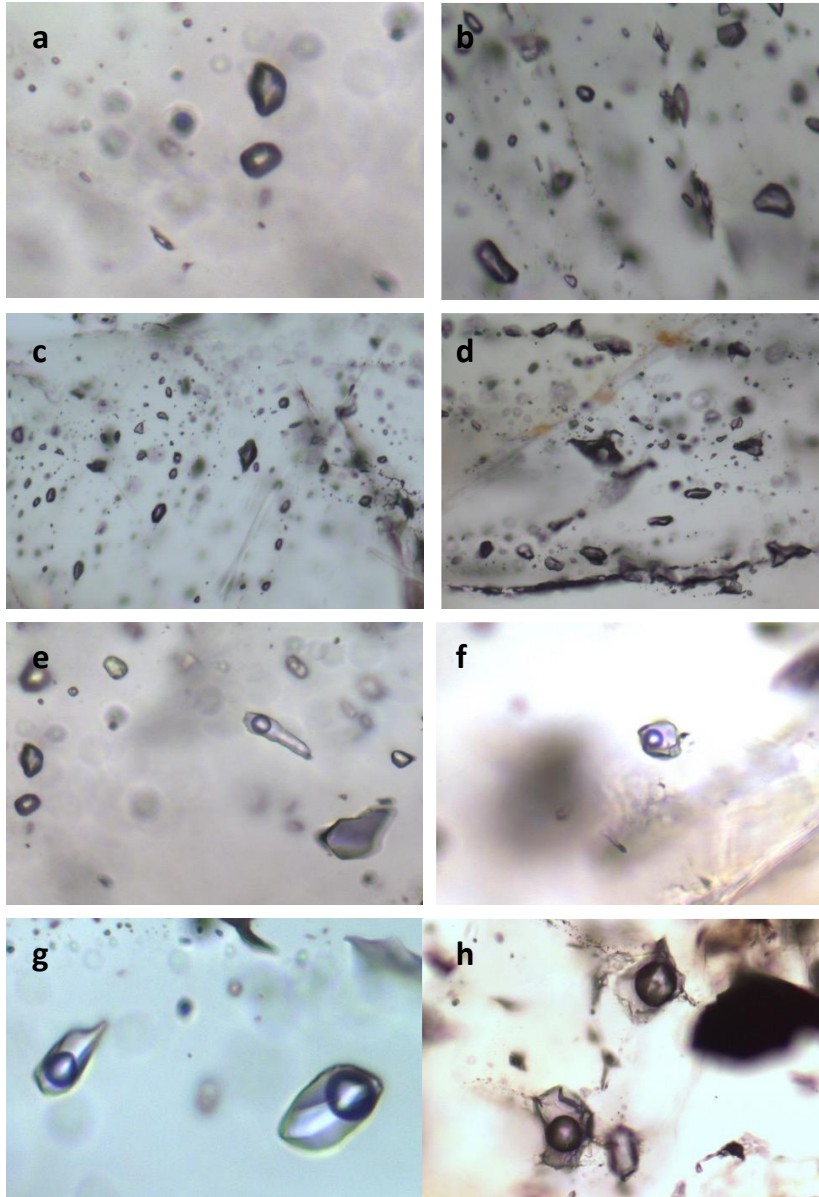


Figure 14. Main fluid inclusion types in quartz from pegmatites of Segura. **(a, b, c, d):** Mono-phase volatile bearing inclusions, prevalent in all samples, **(e):** Coexistence of mono-phase volatile-rich inclusions and bi-phase aqueous-carbonic inclusions. **(f):** Rare bi-phase fluid inclusions with solid, **(g):** Bi-phase aqueous-carbonic inclusions (SEG 4). **(h):** Sizable bi-phase fluid inclusions with significant vapour phase (SEG 4). Most fluid inclusions have a size of around 5 to 20 microns.

In **WP4**, and according to the work plan, activities were mainly directed towards task **4.3 (Re-assessment of alluvial heavy minerals from old exploration surveys)**, despite some exploratory analytical work on some mineral concentrates, during and intertwined with **WP1** (task 1.3).

A preliminary (re-)examination, under the binocular microscope, of 104 samples from polygon 1 (Fig. 1, selected as first priority) confirmed the presence of TiO_2 polymorphs, tourmaline and several other mineral phases of interest. The prevalence of anatase and tourmaline is consistent with the available data for



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polygons 2 and 4. A first insight into the trend of higher mineral concentration domains suggests that: (i) tourmaline should mostly indicate proximity to granitic source rocks and their exocontacts; (ii) anatase is widespread in the region, possibly documenting a regional (metamorphic?) source; and (iii) the relative abundance rutile and brookite emphasise the positive mineral anomalies previously verified (Grácio, 2010).

Forty grains of TiO_2 polymorphs recovered from alluvial samples from the Segura area were analysed with micro-Raman spectroscopy to confirm polymorph assignment based on macroscopic characteristics. The grains were previously embedded in epoxy resin.

A HORIBA XPlora Raman spectrometer, equipped with a 785 nm near-infrared laser and coupled with an Olympus™ microscope, recorded the Raman spectra. The system uses a thermo-electrically cooled charge-coupled device detector (CCD). At least three spectra per sample were acquired in the 100-2000 cm^{-1} range. The 50x objective was used for all samples, whereas the measuring time, laser power, and the number of accumulations being adjusted to avoid thermal damage and to obtain a good signal-to-noise ratio. The instrument itself was controlled using the LabSpec software. The collected Raman spectra were further processed in GRAMS (ThermoFisher Scientific™). The results revealed that polymorph assignment based on macroscopic characteristics is reliable as only around 1% of the titanium oxide grains were misidentified. Micro-Raman spectroscopy also enabled the identification of titanium oxide polymorphs rutile and anatase when such an assignment was not possible using macroscopic observation (Fig. 15).

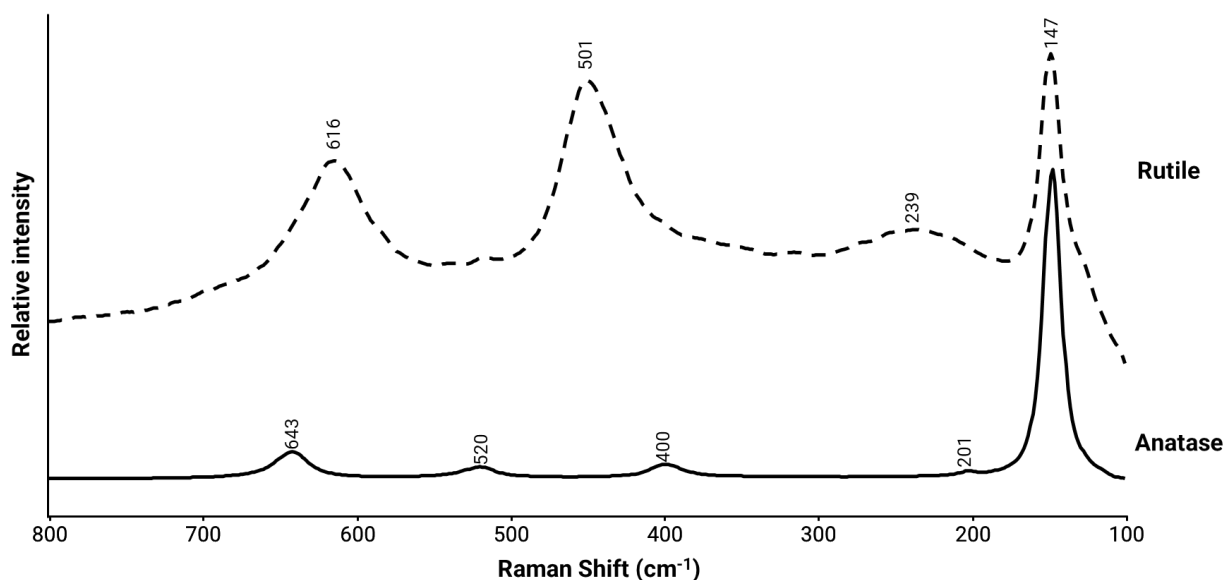


Figure 15: Raman spectra of TiO_2 grains extracted from alluvial samples of the Segura area. Characteristic Raman bands of rutile (top) and anatase (bottom) enabled identification of these two polymorphs.

In addition, thirty-three rutile grains recovered from the same alluvial samples were analysed with LA-ICP-MS for the following elements: Mg, Al, Si, Sc, Ti, V, Cr, Mn, Fe, Cu, Zn, Sr, Y, Zr, Nb, Sn, Ba, La, Ce, Pr, Nd, Sm, Eu, Gd, Yb, Hf, Ta, W, Th and U. The LA-ICP-MS analyses were conducted using a CETAC LSX-213 G2+ laser ablation system coupled to an Agilent™ 8800 Triple Quad ICP-MS. A 100 μm spot size with a frequency of 10 Hz and a laser output energy of 80% was used to analyse 1–3 spots in each rutile grain. Each spot had a total acquisition time of 55 s, including a 15 s washout. With a flow of 0.8 L/min, helium was used as a carrier gas in the LA system, and the ICP-MS data was acquired in MS/MS mode. The NIST



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610 glass standard was used for the calibration of the ICP-MS before the analysis. Fractionation and oxide formation were monitored using the $^{238}\text{U}/^{232}\text{Th}$ ratio and the $^{248}\text{ThO}/^{232}\text{Th}$ ratio, respectively. For the validation of the analytical method (accuracy and precision) and drift correction if needed, two standard reference materials were used – USGS BCR-2G basalt glass standard was used as an external standard for elemental determination, and SRM NIST 610 was used as a “blank” for quality control. Elemental concentrations were determined using off-line calculations and data reduction with the GLITTER® software, using BCR-2G as the primary reference material and TiO_2 as an internal standard for the samples and reference material. TiO_2 values (wt.%) of each rutile sample were previously obtained by EPMA. Detection limits, calculated with the GLITTER® software, average between < 0.001 and 2.628 ppm for elements with an atomic mass higher than 88 (Sr) and between 1.655 and 1877 ppm for all other elements analysed. Precision and accuracy values for all elements are below 5.02 and 0.14%, respectively.

The results obtained suggest that rutile grains recovered from samples sourced by the internal and border granitic facies and aplite-rich zones of the Segura area have different compositions (Fig. 16). These differences indicate that alluvial heavy-minerals may be used to establish mineral fingerprints and footprints to define regional exploration strategies.

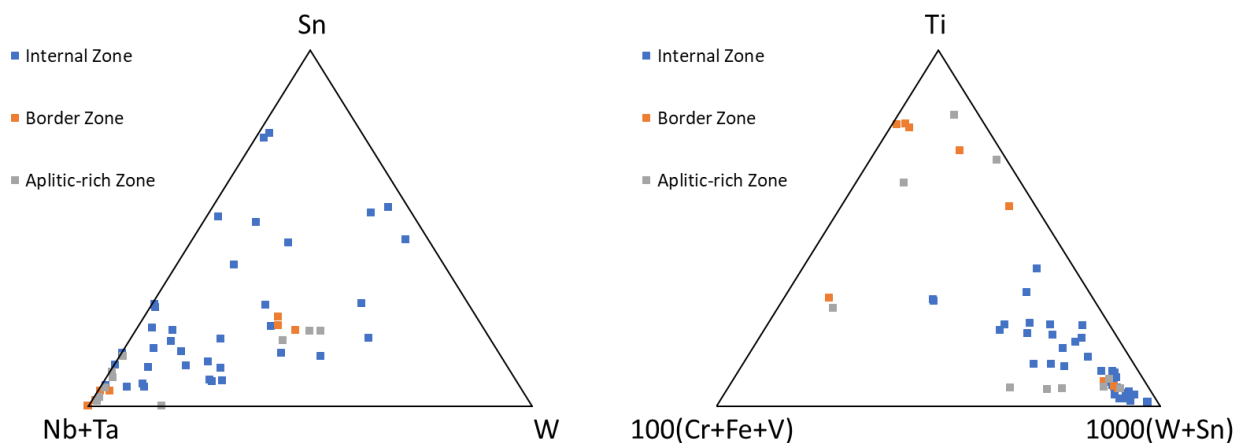


Figure 16: Ternary diagrams Sn-W-Nb+Ta (left) and Ti-1000(W+Sn)-100(Cr+Fe+V) (right), which suggest that the rutile grains recovered from the internal, border and aplite-rich zones of the Segura massif have different compositions.

C.2 Deviations

C2.1 Deviations and difficulties

The main deviations from the work plan and operational difficulties recorded until now were due to circumstances imposed by the COVID-19 pandemic. These circumstances mainly affected the regular scheduling of fieldwork in Portugal (including sampling) and the analytical routines in several laboratories (in Portugal, France and Brazil). Whenever possible (i.e. when circulation across the Portuguese municipalities was allowed and when health safety conditions were ensured), several field surveys of short duration were performed. Similarly, the lab work was resumed whenever access to the facilities in Portugal, France and Brazil was possible (according to the lockdown periods in each of the three countries). Some of the planned work for the Work-Package (WP) 1 was, therefore, delayed. Still, it is our intention to reinforce the endeavours in the following months, particularly on those tasks that are crucial for the consistent development of WP2, WP3 and WP4.

C2.2 Extension of deadlines

Considering the reasons stated above and the results already achieved, **we formally request a 6 month postponement for the remaining milestones and deliverables of the MOSTMEG project** to balance the scheduling uncertainties and operational difficulties due to the COVID-19 pandemic during the 1st year of project implementation.

C.3 Problems in the implementation of the work plan

In case you had any relevant problems with the implementation of the work plan, please select the reason(s) that may explain the problems:

- 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 access restrictions imposed by the COVID-19 pandemic, which varied in time and differed in Portugal, France and Brazil. Delays on the completion of initial tasks also impacted the launch of applications for one MSc contract (LNEG, Portugal) and one Post-doc fellowship (GeoRessources, France).

Comments:

D. RESULTS

D.1. Scientific results

Publications and presentations in scientific meetings are below what was planned, as the highest priority has been the acquisition of new results whenever allowed by the atypical (and unpredictable) rules enforce since the beginning of the COVID-19 pandemic.

| List resulting from jointly conducted work | | | | | |
|---|--------|--|---------------------|----------------------|---------------------------|
| Type of result | Number | Authors, title, year, issue/editor | Partner(s) involved | Open Access (Yes/No) | Website address |
| Peer review papers* | | | | | |
| Books or book chapter | | | | | |
| Publications co-authored by R&D and industrial partners | | | | | |
| Conference proceedings/presentations | 1 | Martins I., Mateus A., Ribeiro da Costa I., Gaspar M., Dias da Silva, Í. (submitted) | P1 | | Abstract submitted to the |





ERAMIN 2

RESEARCH & INNOVATION PROGRAMME ON RAW MATERIALS
TO FOSTER CIRCULAR ECONOMY

| | | | | | |
|------------------------------|--|--|--|--|--------------------|
| | | Geochemistry and ore-forming processes of multi-stage granitic magmatism in the Central Iberian Zone: Segura-Panasqueira Belt (Portugal) case study. | | | SGA Meeting (2022) |
| Other dissemination activity | | 1. Presentation to the public in general at the Centro de Ciência Viva da Floresta (Proença-a-Nova, Sept. 24 th 2021). | | | |

D.2. Innovation oriented results

| List of results | | |
|----------------------------|---------------|-------------|
| Type of result | No. submitted | No. granted |
| Patent applications | | |
| Patents | | |
| International patent | | |
| EU patents | | |
| National patents | | |
| Licences | | |
| New collaborative projects | | |
| Other | | |

D3. Human resources involved

| Academic level | Number of persons involved in project activities | Gender (F/M) |
|-------------------|--|--------------|
| Master Degree | 3 | 1F + 2M |
| PhD degree | 22 | 10F + 12M |
| Post-doc | 2 | 1F + 1M |
| Graduation degree | 1 | F |
| Undergraduate | 0 | 0 |



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ERAMIN 2

RESEARCH & INNOVATION PROGRAMME ON RAW MATERIALS
TO FOSTER CIRCULAR ECONOMY

| Total number of researchers involved | Number of young researchers involved | Number of female researcher involved | Number of permanent jobs created | Number of temporary jobs created |
|--------------------------------------|--------------------------------------|--------------------------------------|----------------------------------|----------------------------------|
| 28 | 6 | 13 | 0 | 2 |

D.4 Dissemination

D4.1. which dissemination tools were used? (Tick all appropriate options and include the link if applicable)

- Project website (<https://mostmeg.rd.ciencias.ulisboa.pt/>)
- Project movie clips to YouTube or another video channel (<https://www.linkedin.com/pulse/first-field-trip-meeting-mostmeg-13-18-sept-2021-icaro>)
- Social and professional networks and blogs
- Other, please specify: LNEG website: <https://www.lneg.pt/en/project/mostmeg-2/>

D.4.2. Have you actively approached or been approached by any companies or stakeholders during the course of your project for exploitation of your project results?

- Yes, we have actively approached
- Yes, we have been approached
- No

Comments: Informal meetings occurred with representatives of the company that has legal exploration rights in the Argemela area. Some other exploratory talks happened by the initiative of a different Portuguese company, interested in lithium exploration. According to the work plan approved for the project, we intend to intensify these contacts in the future, namely through the two Seminars open to academia, industry and stakeholders interested in issues related to the project's main topics.

D.4.3. Have you invited policymakers to project-related events/networking?

- Yes
- No

Comments: Not yet. These kinds of actions are planned to occur in the future, namely when preparing and accomplishing the two Seminars open to academia, industry and stakeholders interested in issues related to the project's main topics.

D.4.4. Have the results of this project contributed to white papers, regulations or standards?



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ERAMIN 2

RESEARCH & INNOVATION PROGRAMME ON RAW MATERIALS
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Yes

No

Comments: The focus of the MOSTMEG project and the intended objectives are not directly related to mineral policies or another type of regulatory instruments.

E. FINANCIAL STATUS

Give an indicative amount of the grant budgets spent by the partners.

| Partner # | 1 | 2 | 3 | 4 | 5 | 6 | 7 |
|---|------------|-----------|-----------|-----------|-----------|-----------|------------|
| TOTAL ALLOCATED BUDGET | 260,274.20 | 49,500.00 | 80,656.50 | 53,852.23 | 28,137.50 | 64.859.00 | 278,715.00 |
| National/regional financing | 75,589.20 | 49,500.00 | 35,437.50 | 35,394.30 | 17,437.50 | 0.00 | 162,815.00 |
| Own financing | 184,685.00 | 0.00 | 45,219.00 | 18,458.23 | 10,070.00 | 64.859,00 | 115,900.00 |
| TOTAL Spent at Annual reporting no. 1 | 72,900.05 | 0.00 | 22,126.75 | 6,152.74 | 3,356.67 | 21,619.67 | 40,532.33 |
| National/regional financing | 26,890.00 | 0.00 | 7,053.75 | 0.00 | 0.00 | 0.00 | 1,899.00 |
| Own financing | 61,561.67 | 0.00 | 15,073.00 | 6,152.74 | 3,356.67 | 21,619.67 | 38,663.33 |
| DEVIATION (against the forecasted expenses at that time) | -15,551.62 | 18,875.00 | 1,658.77 | 5,309.15 | 2,615.22 | 0.00 | 22,523.25 |
| National/regional financing | 11,338.38 | 18,875.00 | 1,658.77 | 5,309.15 | 2,615.22 | 0.00 | 24,422.25 |
| Own financing | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 | 0.00 |

The deviations recorded during the 1st year of the project implementation are due to the operational difficulties briefly described and justified above. In fact, access restrictions to the study area and to the labs imposed by the COVID-19 pandemic (which also varied in time and differed in Portugal, France and Brazil) create several constraints to the planned work, reducing the expended funds. Delays on the completion of initial tasks also



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impacted the launch of applications for one MSc contract (LNEG, Portugal) and one Post-doc fellowship (GeoRessources, France).

F. PUBLISHABLE SUMMARY

The co-occurrence of Sn(-Nb-Ta) and Li(-Sn-Nb-Ta) aplite/pegmatite-types and Sn-Li, Sn(-W), W-Cu(-Sn) quartz vein-types of mineralisation in the Segura-Panasqueira area represents an opportunity to investigate the factors that may rule the (concomitant?) development of distinct ore-forming systems and establish the most promising vectors to be used in further mineral exploration endeavours. During the 1st year of MOSTMEG implementation, several fundamental steps were completed (i) to gradually apprehend the geological and geochemical evolution of granite-related metallogenic processes occurring in a large area, (ii) to better understand the patterns of element distribution in space and time between aplite/pegmatite and quartz lodes during the so-called magmatic-hydrothermal transition, and (iii) to identify the most favourable geochemical/mineralogical fingerprints and footprints of mineralising processes.

The folded pre-Ordovician metasedimentary succession (Beiras Group) is affected by many shear zones and intruded by voluminous plutonic rocks and different arrays of dykes. The composition of metapelites in the Beiras Group show Average Shale-normalised patterns mostly characterised by positive anomalies of Li, Cs, Sn, Hf, Bi, As and Sc, and negative anomalies of Mg, Mn, Ca, S, F, Sr, Nb, Ta, W, Th, U, Y, Ni, Mo, Cd, Cu, Pb, Ge and Ag. The shear zones, typically running ENE-WSW to \approx ESE-WNW and NNW-SSE to NW-SE: (i) include copious arrays of quartz infillings; (ii) may control the rising and emplacement of orogenic silicate melts; and (iii) may act as a preferred loci for lode ore-forming processes. Two main suites of plutonic rocks can be distinguished. The majority of Cambrian-Ordovician tonalities and granodiorites are weakly peraluminous I-type, calcic to calc-alkalic and magnesian rocks. The Variscan (Carboniferous-Permian) monzogranites and granites are highly peraluminous S-type, calc-alkali to alkali-calcic and magnesian to ferroan rocks. The various groups of dykes trace the protracted magmatic activity in the area during the Lower and Upper Palaeozoic periods, being concurrent or after the emplacement of plutonic bodies.

Variscan granite suites are clearly more fertile than those of Cambrian-Ordovician age and, among them, the strongly differentiated and ferroan leucogranites from the Penamacor-Monsanto and Segura plutons trace the most promising targets recognised so far. Granitic facies of the Penamacor-Monsanto and Segura plutons are characterized by degrees of differentiation and metal enrichment close to those of the granites related to ore-forming processes at reference places such as Panasqueira (W-Cu) and Argemela (Sn-Li).

Additional comments to this reporting:

Project Consortium Coordinator Signature:

Date: 28 October 2021



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