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## Plan Overview

*A Data Management Plan created using DMPonline*

**Title:** Spodumene pegmatite haloes: characterization, exploration vectoring and ore potential

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**Template:** Health Research Board DMP Template

### Project abstract:

The growing demand for lithium is driven by the need for a clean energy and transportation transition (Bibienne et al., 2019). To date, the majority of lithium is extracted from salars, their occurrence however is in the main part restricted to South America and subordinately China. Hard-rock lithium sources such as Li-Cs-Ta (LCT) pegmatites (e.g. Selway et al. 2005) or rare metal granites (RMG) have a wide distribution globally and in Europe specifically (e.g. Gourcerol et al., 2019). Spodumene is a Li-Al pyroxene ( $\text{LiAlSi}_2\text{O}_6$ ) and spodumene pegmatites are the main hard-rock source for lithium. Often not or poorly exposed, the first step in successful Li exploitation is the establishment of quick and comparably cheap exploration tools. This project aims to target the metasomatic haloes surrounding the pegmatites and employ the geochemical halo signature in certain minerals as a tool to vector towards spodumene pegmatites. The preliminary research hypothesis states that the parental liquids of the spodumene expel a- or multiple pulses of a hydrothermal fluid at the transition between magmatic and hydrothermal conditions. The process by which this fluid separates from the late-stage magmatic liquid may be liquid immiscibility (Kaeter et al., 2018). The expelled fluid forms chemical haloes around the pegmatite intrusion, the intensity of this halo signature decreases with increasing distance from the pegmatite. The main study area is located in Ireland in the southern part of Leinster, where both barren as well as mineralized pegmatites intruded the East Carlow Deformation Zone along the margins of the ~400 Ma (Fritschle et al., 2018) Leinster granite. The objectives of the research projects are both scientific as well as economic in nature. Scientifically, the process of halo formation is aimed to be constrained from the chemistry of certain mineral phases and subsequently connected to the processes of pegmatite crystallization. The question whether halo formation should be seen as single- or multi scale process is central. The second part of the project is economically in nature and aims to establish effective tools for future exploration of hard-rock lithium sources. A hand-held Laser Induced Breakdown Spectrometry (LIBS) method is to be developed, which aims to make detection of the halo signature on drill cores and outcrops further away from the pegmatite possible. For the same reason geochemical analysis of typical halo minerals in stream sediments is planned in order to detect mineralized pegmatites upstream.

References cited:

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5. Selway, J. B. A Review of Rare-Element (Li-Cs-Ta) Pegmatite Exploration Techniques for the Superior Province, Canada, and Large Worldwide Tantalum Deposits. *Exploration and Mining Geology* 14, 1–30 (2005).

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# Spodumene pegmatite haloes: characterization, exploration vectoring and ore potential

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## Data description and collection or re-use of existing data

### How will new data be collected or produced and/or how will existing data be re-used?

New data collected for this project can be grouped into two main categories: *A) geochemical data* and *B) observational and descriptive data*.

#### *A) Geochemical data*

The acquisition of geochemical data will produce three types of datasets: *a) whole rock geochemical data*, *b) quantitative in situ data* and *c) semi quantitative in situ data*.

The first step for all three data types involves sampling from outcrops and/or drill core.

For (*a*), a portion of each sample is labelled, packaged, and sent to an external laboratory (ALS) for sample preparation and analysis.

Sample preparation includes crushing and grinding the rock into a fine, homogeneous powder.

- For major and minor element analysis, the powder is fused with lithium borate to form a glass disk which is then analysed by X-ray fluorescence (XRF).
- For trace element analysis, the powder is subjected to four-acid digestion, and the resulting solution is analysed using ICP-OES trace elements with high abundance and ICP-MS for low concentration trace elements.

The data is returned as .csv files containing elemental concentrations reported as wt% oxides (XRF) and ppm or element % for high-concentration trace elements (ICP-OES/ICP-MS).

Data quality control is internally monitored by ALS through the measurement of standards and blanks and externally monitored through hidden duplicates.

For all *in situ analyses*, thin sections are prepared by cutting a small piece of the sample into a slab, mounting it on a glass slide, and polishing it to ~30 µm thickness. Samples intended for major and minor element analysis are carbon-coated prior to measurement.

#### *Major and minor element analysis:*

Quantitative and semi-quantitative major element in situ data are collected using three electron-beam instruments: a Tescan TIGER Mira III field-emission SEM (equipped with dual Oxford X-MaxN 150 mm<sup>2</sup> EDS detectors) at the iCrag laboratory at the Trinity college Dublin (TCD), a Cameca SC 100 EPMA operating in wavelength-dispersive mode at the Joint Laboratory of Electron Microscopy and Microanalysis of the Department of Geological Sciences, Masaryk University, Brno, Czech Republic and a JEOL JXA-8530F EPMA at the faculty of geosciences of Utrecht University, Netherlands.

Both SEM and EPMA require samples to be loaded into a vacuum chamber. The electron beam interacts with the sample, producing secondary and backscattered electrons. Chemical information is collected via EDS (SEM) or WDS (EPMA).

- Quantitative spot analyses are obtained from defined locations using:
  - SEM: 20 kV accelerating voltage, 300 pA beam current, 1 µm beam diameter.
  - EPMA: 15 kV, 10 nA, and spot sizes between 2–10 µm.
- Semi-quantitative elemental maps capture relative variations in selected elements across defined surface areas.

Quantitative analyses are calibrated against mineral and glass standards. Data output is in .csv or text format, representing either per-spot elemental concentrations or per-pixel relative abundances, which can be converted into compositional maps.

### *Trace element analysis:*

In situ trace element analysis was performed using LA-ICP-MS (Laser Ablation – Inductively Coupled Plasma – Mass Spectrometry) on the same spots and areas previously analyzed for major elements. Polished thin sections were loaded into a gas-purged ablation cell, where a 193 nm ArF excimer laser ablated material from the surface. The aerosol was carried by a helium–argon gas flow into a high-temperature plasma for ionization, and the resulting ions were analyzed in a quadrupole mass spectrometer based on their mass-to-charge ratio.

Two similar instrument setups were used:

- First setup: Photon Machines Analyte Excite laser with a HeLex two-volume cell, coupled to an Agilent 7900 ICP-MS at the iCrag laboratory in Trinity College Dublin

Second setup: Teledyne Analyte G2 laser with a HeLex cell, coupled to a Thermo iCAP Qc ICP-MS at the National Centre for Isotope Geochemistry (NICIG) at University College Dublin.

Both quantitative and semi-quantitative data required external standards (NIST 610, GSE-1G, BCR-2G). Quantitative spot analysis additionally used an internal standard, derived from prior major element data (e.g., Ca or Si).

- Mapping parameters: 10  $\mu\text{m}$  laser spot, 65 Hz repetition rate, 3.0 J/cm<sup>2</sup> fluence; maps were acquired via adjacent line scans with a square aperture.
- Spot analysis: 15–25  $\mu\text{m}$  spot size, 11 Hz repetition rate, 2.0 J/cm<sup>2</sup> fluence.

Raw data were exported as .csv files and processed in Iolite software to generate semi-quantitative trace element maps and quantitative point analyses.

### *B) Observational and descriptive data*

Observational data is acquired in the form of pictures of samples, pictures of microstructures and minerals via a microscope-camera set up and scans of entire thin sections. Descriptive data is acquired in the form of field book entries, sample descriptions and descriptions of the petrography of thin sections.

A dataset of whole-rock analyses previously acquired for the GREENPEG project will be reused to formulate ideas and generate figures for this project. Sampling, sample preparation and analysis are comparable to the method for whole-rock analysis described above.

**What data (for example the kind, formats, and volumes), will be collected or produced?**

|                               | Type of data                              | Data collection                                                                                                                                   | Purpose                                                    | File formats   | Volume |
|-------------------------------|-------------------------------------------|---------------------------------------------------------------------------------------------------------------------------------------------------|------------------------------------------------------------|----------------|--------|
| Geochemical data              | Numerical – quantitative                  | XRF, ICP-MS, ICP-OES                                                                                                                              | Understand the broader distribution of halo mineralization | .csv           |        |
| Numerical – quantitative      | EPMA, SEM, LA-ICP-MS                      | Characterize the mineralogy and chemistry of minerals affected by or crystallized through the interaction with pegmatitic fluids in the host rock | .csv                                                       |                |        |
| Numerical – semi quantitative | EPMA, SEM, LA-ICP-MS                      | Characterize micro-reaction textures and chemical variations within grains                                                                        | .csv<br>.jpg<br>.png                                       |                |        |
| Non-geochemical data          | Descriptive data                          | Microscope observations                                                                                                                           | Characterize mineral assemblages and microtextures         | .xlsx<br>.docx |        |
| Drill core observations       | Characterize halo distribution and extend | Field book entries<br>.xlsx                                                                                                                       |                                                            |                |        |
| Observational data            | Microscope pictures                       | Visualize mineral assemblages and microtextures                                                                                                   | .jpg<br>.png                                               |                |        |
| Thin section scans            |                                           |                                                                                                                                                   |                                                            |                |        |
| Drill core images             | Visualize halo distribution and extend    |                                                                                                                                                   |                                                            |                |        |

## Documentation and data quality

**What metadata and documentation (for example the methodology of data collection and way of organising data) will accompany data?**

Additional to the metadata specific to the individual types of data, an overview metadata table will be provided specifying for each sample, sampling strategy, sampling date, drill core ID (if applicable), longitude and latitude, the drill core inclination (if applicable), depth interval sampled, the lithology as well as an overview of the analytical methods applied to each sample and to which step the sample is prepared (powder, covered thin section, uncovered thin section etc.)

| <b>All samples</b>                             |                         |                              |                        |                        |
|------------------------------------------------|-------------------------|------------------------------|------------------------|------------------------|
| Geographic information                         |                         |                              |                        |                        |
| Sample number                                  | Drill core ID           | Sample/drill core lat.-long. | Drill core inclination | Depth interval sampled |
| Sample type                                    |                         |                              |                        |                        |
| Sample type (drill core, boulder, outcrop)     | Chemical classification | Lithology                    | Alteration status      |                        |
| <b>In situ analytical sites</b>                |                         |                              |                        |                        |
| Sample type (thin section, thick section etc.) | Analytical method       | Analytical method code       | Mineral specimen       | Lithology              |

[There seems to be no widely accepted metadata standard for geochemical data, or for petrographic data. I think you can answer this by creating a version of the range of metadata listed in Chamberlain et al. (2021). For geographical metadata on drill core samples you should also include latitude, longitude, azimuth (compass direction) and inclination from horizontal of the hole, and the position in metres of the sample within the drill core. The same metadata will apply to most of your datasets, so a table method by method would be unnecessarily repetitive. For rocks, indicate the lithology and unit to which it belongs. For minerals, give the mineral name, and the rock type it occurs in. Put the lab-related things in the next section of the plan.

It would be a good idea to add a second table dealing with curation of physical samples. This should include sample number, type of material (rock specimen, mineral specimen, thin section, etc.), lithology of rock or name of mineral, approximate size or quantity of rock or mineral specimen, for thin section whether covered, uncovered, polished, approximate thickness and what it has been used for (optical, SEM, EPMA, LA-ICP-MS, etc.). The storage location after your project ends should also be included as an item, even though you won't know where that is.]

## What data quality control measures will be used?

|                                                    |                    |                    |                    |                       |            |        |
|----------------------------------------------------|--------------------|--------------------|--------------------|-----------------------|------------|--------|
| Laboratory information                             |                    |                    |                    |                       |            |        |
| Analyst and laboratory                             | Analytical code    | Instrument used    | Measured parameter | Technique description |            |        |
| Data quality control for each analytical procedure |                    |                    |                    |                       |            |        |
|                                                    | External standards | Internal standards | Uncertainty        | Detection limit       | Duplicates | Blanks |
| EPMA                                               | Yes                | -                  | -                  | Yes                   | No         | No     |
| SEM                                                | Yes                | -                  | -                  | Yes                   | No         | No     |
| LA-ICP-MS                                          | Yes                | Yes                | 2SE, 2SD           | Yes                   | No         | No     |
| XRF                                                | Yes                | -                  | Yes                | Yes                   | Yes        | Yes    |
| ICP-MS<br>ICP-OES                                  | Yes                | -                  | Yes                | Yes                   | Yes        | Yes    |

## Storage and backup during the research process

### How will data and metadata be stored and backed up during the research process?

All data is stored in the google drive account of the iCRAG google account of the data manager ([elena.geiger@icrag-centre.org](mailto:elena.geiger@icrag-centre.org)). Individual datasets are stored on the local drive of the work computer of the data manager. Those are regularly backed up into the drive folder.

The entire folder containing all data and metadata related to this project is additionally backed up monthly on an external hard drive.

State what you are doing at the moment for storage and backup. Note the UCD guidance on Google Drive for file storage. Then we can review it and see if any change to practice is needed.

### How will data security and protection of sensitive data be taken care of during the research?

Operating system and application updates are installed on the used laptops when they are available, Sophos endpoint protection software is installed on the used computers as well. The laptop of the data manager is password protected at all times. The external hard drive containing monthly backups is stored in a locked cabinet in the office of the data manager. Files confidential to companies involved in the project are especially protected.

Talk about password protection on your laptop, and of specific files if they are confidential to Blackstairs or anyone else. However, apart from company files, there is no serious worry here about data confidentiality - we don't have to protect personal details, or anything related to national security! See also the text below in the example for more things to include.

## **Legal and ethical requirements, codes of conduct**

### **If personal data are processed, how will compliance with legislation on personal data and on security be ensured?**

No personal data will be used in the project.

### **How will other legal issues, such as intellectual property rights and ownership, be managed? What legislation is applicable?**

It is anticipated that intellectual property generated by the project will be made available as open access scholarly publications. Where it would be desirable to include pre-existing IP of mineral exploration companies, their permission will be sought before including it in scholarly publications.

### **What ethical issues and codes of conduct are there, and how will they be taken into account?**

There are no ethical issues relating to people or animals.

Both UCD researchers named on this project have passed the Epigeum Research Integrity online course organised through UCD.

Ethical considerations apply to the use of proprietary data originating in exploration companies. Such data will remain confidential to the two researchers unless and until the owner of the data gives permission for wider dissemination, which may include presentation at conferences, inclusion in scholarly publications, or possibly even inclusion in final archiving of project data at the end of the project.

## **Data sharing and long-term preservation**

### **How and when will data be shared? Are there possible restrictions to data sharing or embargo reasons?**

There are no data that must be destroyed for any reason.

All data generated by the project will be made publically available. Data used by the project but originating elsewhere may be made publically available if the owners of the data agree and it is useful to do so, i.e. the data are not publically available elsewhere. Much of the data will be published in open

access peer-reviewed scientific papers, including supplementary data. Whilst some should be published before the end of the project, the time required for write up, review and publication means that some will be published after the end of the project.

### **How will data for preservation be selected, and where data will be preserved long-term (for example a data repository or archive)?**

When all data worthy of publication has been published, all project data, whether in peer-reviewed publications or not, will be uploaded to Zenodo (<https://zenodo.org>), a multi-disciplinary open repository maintained by CERN. A digital object identifier (DOI) is automatically assigned to all Zenodo files and Zenodo provides detailed guidance on file naming and structuring, and on metadata.

### **What methods or software tools are needed to access and use data?**

All data is stored in the google drive account of the iCrag google account of the data manager ([elena.geiger@icrag-centre.org](mailto:elena.geiger@icrag-centre.org)). Individual datasets are stored on the local drive of the work computer of the data manager. Those are regularly backed up into the drive folder.

The entire folder containing all data and metadata related to this project is additionally backed up monthly on an external hard drive.

The google drive account is password protected, as is the laptop with the desktop shortcut to the drive folder.

The external hard drive is at all times stored in a locked cabinet to which the data manager only has the keys.

Individual datasets that belong to companies might need to be retained, there is no need to destroy any data.

The data will be made available when all publishable data has been published and will be uploaded to Zenodo.

This seems to repeat an earlier section, but put it in here too and again refer to the UCD guidance to the right of this panel.

In here you should also think about chemical mapping data. I suggest you first talk to Maurice Brodbeck to find out what he is doing to archive your data. If there is a pathway in what he is doing to allow your raw data from chemical mapping to be made public, you can refer to that. More likely, he is just archiving it for our own use and we then have to think about it some more!

### **How will the application of a unique and persistent identifier (such as a Digital Object Identifier (DOI)) to each data set be ensured?**

A DOI will automatically be attached to each data set when it is archived with Zenodo.

### **Data management responsibilities and resources**

**Who (for example role, position, and institution) will be responsible for data management (i.e. the data steward)?**

During the lifetime of the project, Elena Geiger is responsible for data capture, metadata production, data quality, storage and backup, data archiving, and data sharing, under the guidance of Julian Menuge. When the project ends, Julian Menuge will be responsible for all data management and stewardship roles.

The DMP will be constructed and maintained by Elena Geiger, under the guidance of Julian Menuge, for the duration of the project. Any updating of the DMP required after the end of the project will be carried out by Julian Menuge. It is anticipated that Julian Menuge will retire from UCD in September 2028, two years after the planned end of the project. It is likely that all project data will be archived by Zenodo well before September 2028.

**What resources (for example financial and time) will be dedicated to data management and ensuring that data will be FAIR (Findable, Accessible, Interoperable, Re-usable)?**

The time required for data management will be reckoned as part of the PhD research time of Elena Geiger and the UCD employment of Julian Menuge. The iCFAG grant supporting the project includes money for some open access peer-reviewed publication, and additional open access charges for publication are expected to be met by the agreements between UCD and major publishers. There are no other foreseen financial requirements; indefinite archiving on the Zenodo site is free of charge.