Using TESCAN TIMA for automated mineralogy characterization of lithium ore

Introduction:

Lithium has become a crucial element in modern industry, particularly in the production of batteries for electric vehicles and renewable energy storage. As demand for lithium continues to rise, it is essential to develop efficient and sustainable methods for its extraction from lithium-bearing ores, such as spodumene and lepidolite pegmatites. Characterizing these ores using a classical electron microscopy approach presents a challenge because the lithium cannot be observed directly with energy dispersive spectroscopy (EDS). X-ray diffraction (XRD) is used frequently to quantify the abundance of spodumene, but it does not provide any information on the texture and grain size distribution of this phase. XRD quantification of lithium-bearing micas is even more challenging because there are only very subtle differences between the lithium-bearing (e.g. lepidolite*) and lithium-free micas (typically muscovite) in the diffraction patterns. This application note presents the use of a SEM-based automated mineralogy technique to characterize samples of spodumene/lepidolite pegmatitic ore from both compositional and textural perspectives.

Materials and Methods:

A lithium-cesium-tantalum (LCT) pegmatite ore dominated by spodumene was analyzed. The source locality for the sample cannot be disclosed. Epoxy blocks representing four size fractions were provided (−250, 250−500, 500−1000, +1000).

TESCAN TIMA was used to perform automated mineralogy characterization (modal composition, liberation degree and association). TIMA mineral maps and corresponding grain coordinates were used to localize representative analytical points for laser ablation inductively coupled plasma mass spectrometry (LA ICP-MS) which was used to determine the actual lithium, rubidium and cesium content of the phases.

Lithium bearing minerals can be identified indirectly in TESCAN TIMA by relying on the characteristic ratio of other elements present, like Al, Si, and F, which can be detected and quantified by EDS.

Wet chemical analysis was used to quantify elements in the bulk sample which allowed verification of the bulk chemistry calculation using TIMA.

Results and Discussion:

Modal mineralogy:

The composition of the sample was dominated by spodumene followed by gangue minerals such as quartz, albite, potassium feldspar (microcline), muscovite and lepidolite; also accessory minerals dominated by manganotantalite, cassiterite and triphylite (see Table 1). Automated Mineralogy is the only technique that can identify and quantify economically significant accessory minerals in the range between ppm and 1 wt. %.

Bulk chemistry

Using the mass percentage of minerals and their chemical compositions as input, TIMA performs quantification of elements present in a bulk sample. For the final calculation, the necessary chemical composition of each mineral can be derived directly using the offline automated mineralogy data from the sample (see Table 2). It is common for either an LA ICP-MS or an electron microprobe analyzer to be utilized to detect elements like lithium, cesium and rubidium, which EDS cannot detect due to their low concentrations or the technique's limitations. Average concentration of the above-mentioned elements is listed in Table 1. The accuracy of the bulk composition calculation performed by TIMA was validated by comparing it with the results obtained by wet chemical analysis (see Figure 1).

Lithium deportment:

Three lithium bearing phases were identified in the sample: spodumene, lepidolite and triphylite (see Figure 2). Spodumene is the dominant lithium host (88.1 %) but a significant portion of lithium is held by lepidolite (11.4%). The remaining <0.5% of lithium is hosted by accessory triphylite. The spodumene in our study is close to its ideal composition, with only minor amounts of iron and sodium (see Table 2).

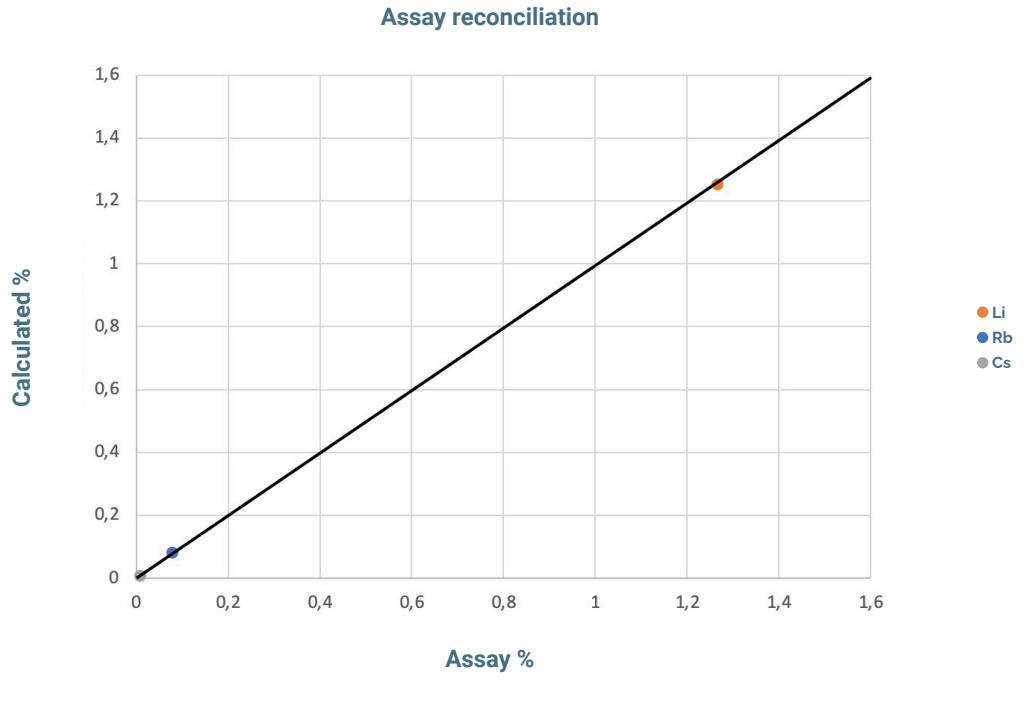
** Lepidolite is a mineral species discredited by International Mineralogical Association, but the term is still widely used for Li micas such as trilithionite or polylithionite that cannot be distinguished in the field.*

Mineral	Ideal formula	Mean Li content in wt. % (LA ICP MS)	Mean Rb content in wt. % (LA ICP MS)	Mean Cs content in ppm (LA ICP MS)	Content of mineral in wt. %.
Quartz	SiO_2	-	-	-	38.922
Spodumene	$LiAlSi_2O_6$	3.69	-	-	29.992
Albite	$NaAlSi_3O_8$	-	-	-	12.875
Microcline	$KAlSi_3O_8$	-	0.38	23	8.114
Muscovite	$KAl_2(AlSi_3O10)(F,OH)_2$	-	0.28	158	4.783
Lepidolite	$KLi_2AlSi_4O_{10}F(OH)$	2.38	0.85	1825	4.038
Cassiterite	SnO_2		-	-	0.681
Tantalite-(Mn)	$Mn(Ta, Nb)_2O_6$	-	-	-	0.470
Triphylite	$LiFePO_4$	4.43	-	-	0.126
Total					**100.000**

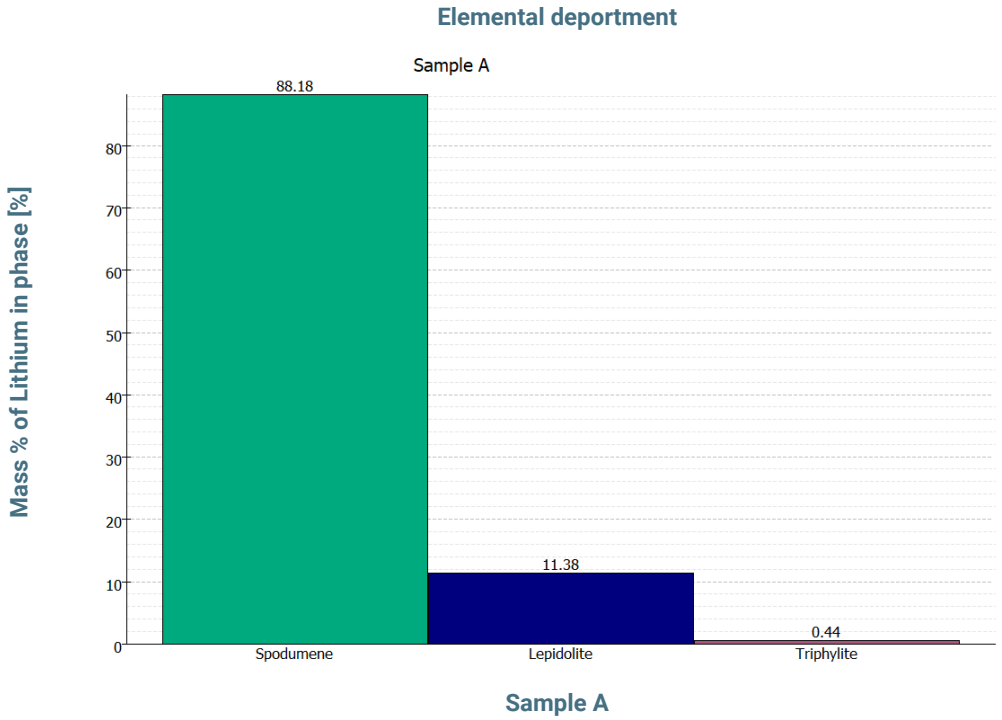
▲ **Table 1:** Abundance of minerals present in the sample and their average Li, Cs, and Rb content as obtained by LA ICP-MS.

	Li	O	Na	Al	Si	Fe
Average concentration	3.69%	51.08%	0.80%	14.54%	29.70%	0.17%
Standard deviation	0.01%	0.09%	0.12%	0.10%	0.10%	0.09%

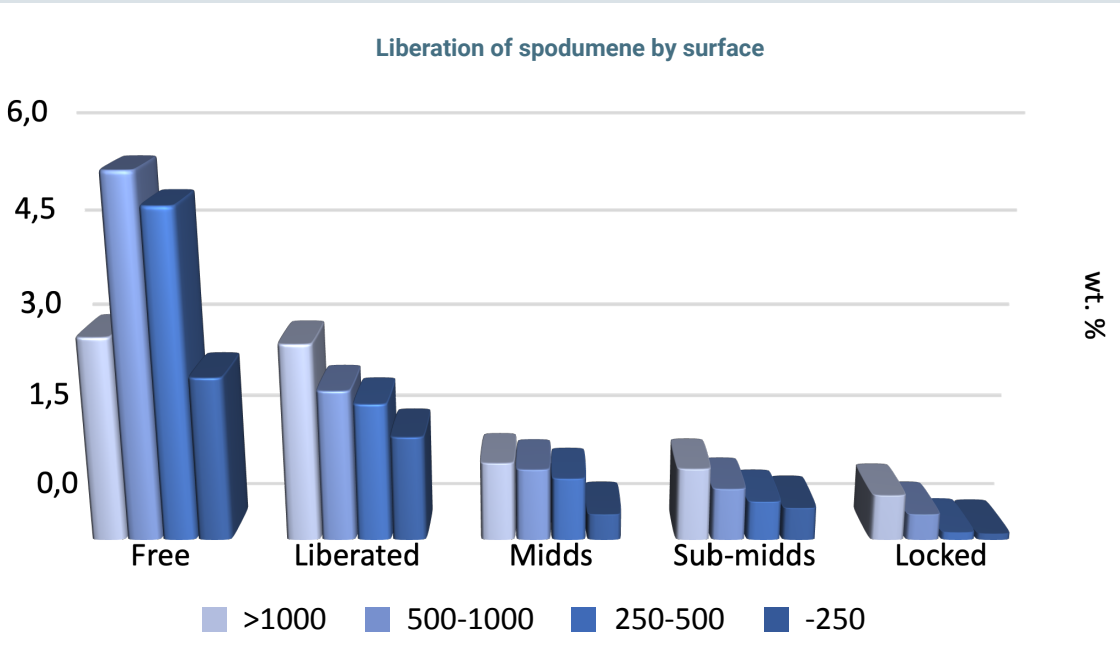
▲ **Table 2:** Average chemical composition of spodumene based on stoichiometric calculations using EDS data.



▲ **Figure 1:** Assay reconciliation of Li, Cs, and Rb.



▲ **Figure 2:** Elemental deportment of lithium.

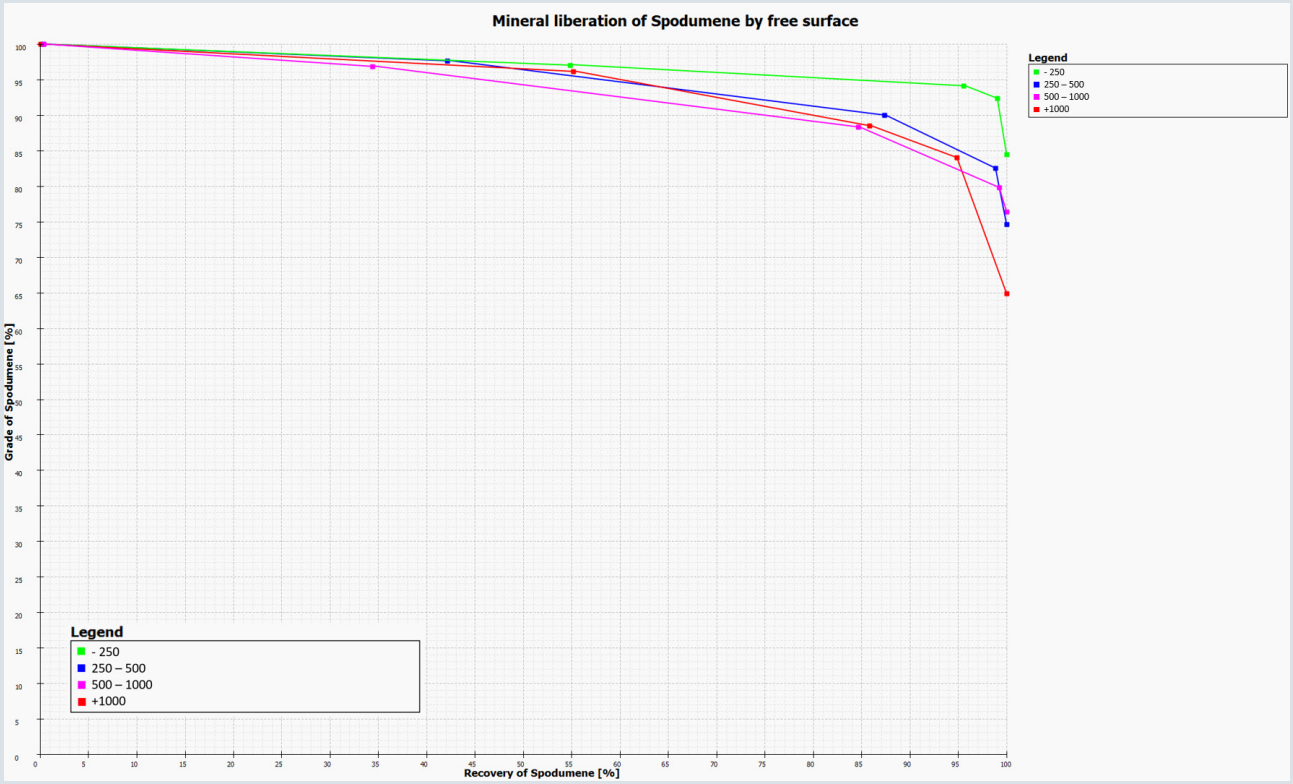


▲ **Figure 3:** Surface liberation of spodumene expressed in weight %.

Liberation and theoretical grade recovery curve

TESCAN TIMA analyses samples at the particle-by-particle level. Based on this data, it is possible to categorize particles containing minerals of interest into specific surface (or volume) liberation categories such as free (> 95 %), liberated (80 – 95 %), middling (30 – 80 %) and locked (< 30 %). This approach helps to predict the behavior of the ore throughout the beneficiation process. In the given sample, the majority (78%) of the spodumene belongs to the well-liberated categories (see Figure 3). The middling categories are formed primarily by intergrowths with quartz and alkaline feldspars. Lepidolite is also well-liberated and only a small portion intergrows with spodumene; thus, it is possible to concentrate lepidolite from the tailings of the spodumene processing.

TESCAN TIMA automated mineralogy data also generates theoretical grade-recovery curves, which reflect the highest possible recovery rate for a given mineral at a specific grade of the product, based on the surface liberation and grind size. The curves' positioning on graphs are indicators of the expected grade and recovery, with curves to the right indicating optimal recovery. (see Figure 4).

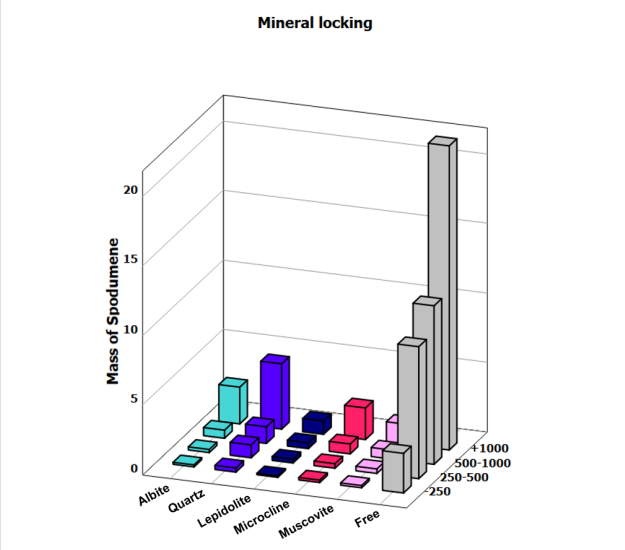


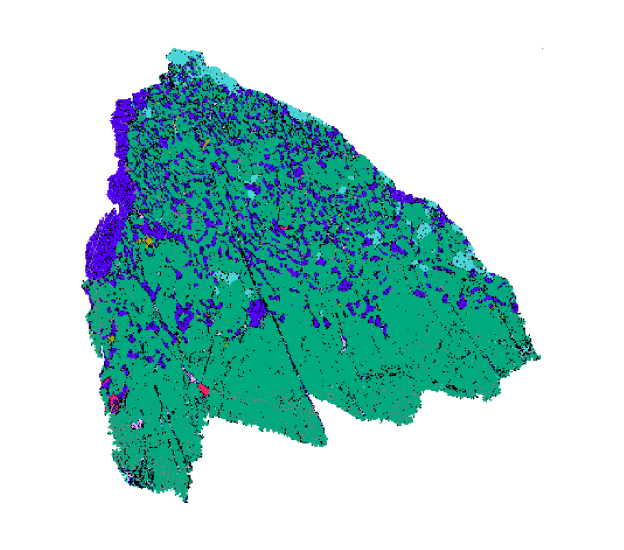
▲ **Figure 4:** Theoretical grade-recovery curve for analyzed size fractions.

Mineral locking

Mineral locking is used to characterize particles hosting the mineral of interest. It provides the mass proportion for the exposed mineral (free surface) versus locked minerals. In this case, the spodumene is highly liberated but a certain proportion is locked with gangue minerals such as feldspars, quartz, and micas (see Figure 5).

In the finer size fractions, the locking becomes dominated by quartz at the expense of another gangue. This is caused by the presence of a specific textural type of spodumene containing exsolutions of quartz (see Figure 5).



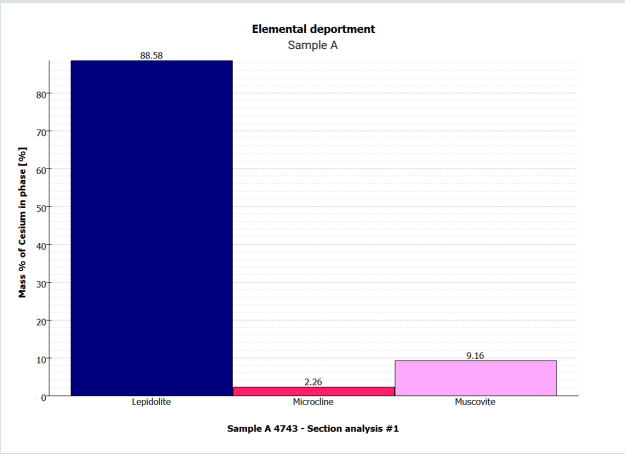


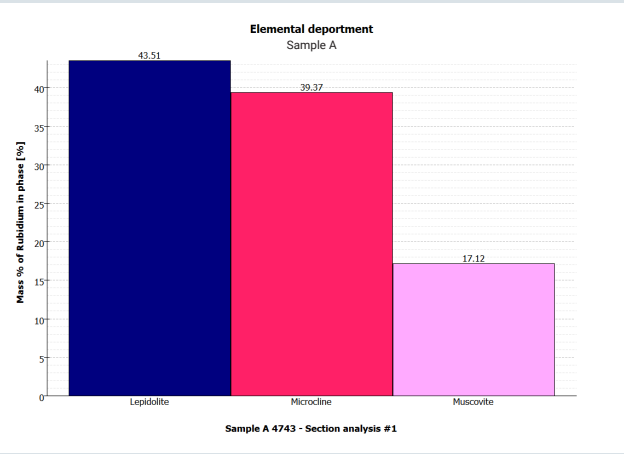
▲ **Figure 5:** Mineral locking of spodumene (left) and an example of a spodumene-rich (green) particle containing fine quartz (blue) exsolutions.

Other valuable components:

Other potentially economically significant minerals are the tin and tantalum oxide minerals cassiterite and tantalite-(Mn). However, these are typically fully locked in albite and quartz, and they are very fine grained (generally below < 40 µm), so further grinding would be necessary to liberate them.

Heavy alkaline metals rubidium and cesium are potentially interesting by-products of lepidolite processing. Lepidolite hosts nearly 90% of cesium (see Figure 6). Rubidium is also dominantly hosted by lepidolite but about 40% is hosted by microcline without a chance of reporting to lepidolite concentrate (see Figure 6).





▲ **Figure 6:** Cesium (left) and rubidium (right) deportment.

Conclusion and Outlook:

Automated Mineralogy is invaluable for characterizing the lithium-bearing ores because, unlike any other technique, it can provide information on textural properties such as liberation degree, particle/grains size, distribution, and association on top of the modal composition. The results demonstrate the potential for efficient beneficiation and thus, for extraction and upgrade of lithium from the ore.

Automated Mineralogy in connection with other techniques such as LA-ICP MS furthered our understanding of deportment of lithium and that of trace elements which are typically below EDS detection limits, for example Rb and Cs in micas and feldspars. This way, it was possible to evaluate the recoverable Rb together with lepidolite versus the Rb hosted by feldspars and thus lost to tailings.

Automated Mineralogy facilitated the identification of additional valuable elements like tantalum and tin, which can be recovered through further processing. Similarly, Automated Mineralogy makes it possible to focus on the presence of impurities, like iron oxides in the case of high grade spodumene concentrates used in ceramics production, and minerals like calcite affecting chemical treatment of the ore.

The role of automated mineralogy for optimizing lithium extraction processes and managing impurities is crucial to promote sustainable and economical production of high-grade lithium concentrates.

Further reading:

Aylmore, M. et al. (2018): Assessment of a spodumene ore by advanced analytical and mass spectrometry techniques to determine its amenability to processing for the extraction of lithium. Minerals Engineering 119, 137-148 p.

Aylmore, M. et al. (2018): Applications of advanced analytical and mass spectrometry techniques to the characterization of micaceous lithium-bearing ores. Minerals Engineering 119, 182-195 p.

Di Feo, A. et al. (2023): Applications of Automated Mineralogy in Beneficiation of the Sayona's Lithium Deposit in the Abitibi Region of Québec, Canada. In book: Proceedings of the 62nd Conference of Metallurgists, COM 2023.

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