

## X-ray diffraction

# Analysis of Li hard rock ores for mining exploration using ARL X'TRA Companion X-ray Diffractometer

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Global Application XRD

**Introduction**

Lithium (Li) hard rock ores, such as spodumene, petalite, and lepidolite, are primary sources for lithium extraction, crucial for various applications including batteries for electric vehicles and portable electronics. These ores are typically mined from pegmatite deposits and require efficient processing to extract lithium. Spodumene, the most common lithium ore, contains up to 3.73% lithium and undergoes a series of processes including crushing, grinding, and flotation to concentrate the lithium content.

X-ray diffraction (XRD) is an essential analytical technique in the analysis and processing of lithium hard rock ores. XRD provides detailed information on the mineralogical composition and crystallographic structure of the ores, which is crucial for optimizing beneficiation processes. By identifying and quantifying the minerals present, XRD aids in determining the most effective extraction methods. To determine the lithium content, XRD can be used to identify the specific lithium-bearing phases and quantify their abundance. This information allows for the calculation of the lithium content based on the known stoichiometry of the identified minerals. Additionally, XRD can monitor phase changes during thermal treatments, ensuring the consistency and quality of the final lithium product. XRD also helps in detecting impurities and optimizing the overall efficiency of the extraction process.

## Instrument & software

The Thermo Scientific™ ARL™ X'TRA Companion X-ray Diffractometer (c.f. Figure 1) is a simple, easy-to-use benchtop XRD instrument for routine phase analysis as well as more advanced applications. The ARL X'TRA Companion XRD uses a  $\theta/\theta$  goniometer (160 mm radius) in Bragg-Brentano geometry coupled with a 600 W X-ray source (Cu or Co). The radial and axial collimation of the beam is controlled by divergence and Soller slits, while air scattering is reduced by a variable beam knife. An integrated water chiller is available on demand. Thanks to the innovative solid state pixel detector (55 x 55  $\mu\text{m}$  pitch), the ARL X'TRA Companion XRD provides very fast data collection and comes with one-click Rietveld quantification capabilities and automated result transmission to a LIMS (laboratory information management system).



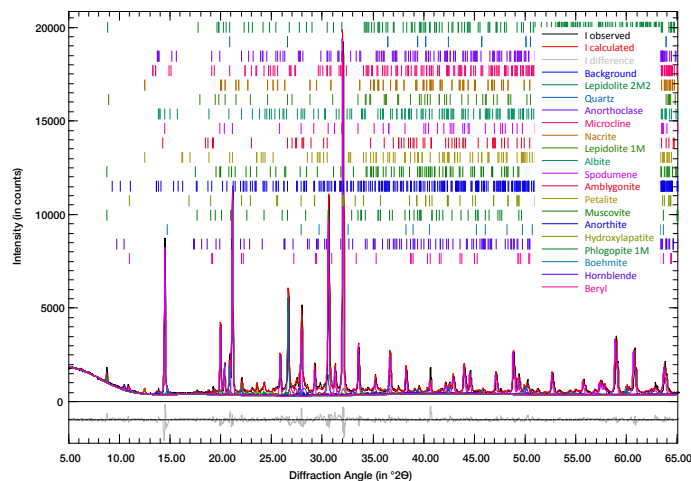
**Figure 1:** ARL X'TRA Companion X-ray diffractometer system.

## Experimental

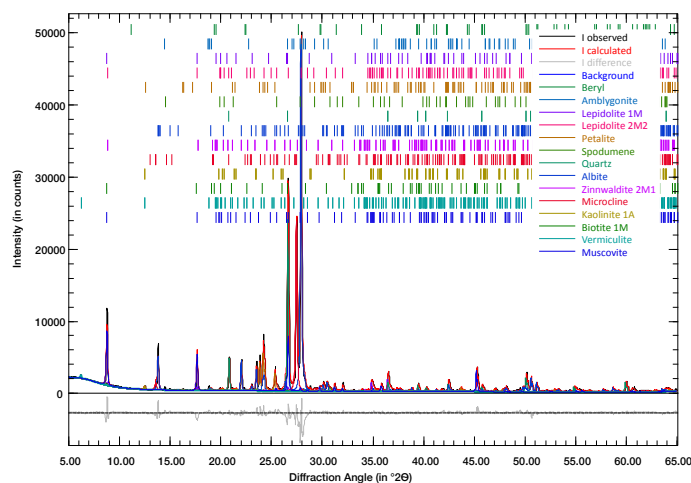
A quantitative XRD analysis of three Li hard rock ore samples with spodumene (sample 1), petalite (sample 2), and lepidolite (sample 3) as the main Li-bearing phases was carried out. The samples were milled and loaded in backloading sample cups, and measurements in reflection mode using Cu K $\alpha$  (1.541874 Å) radiation with sample spinning were performed (30 minutes) (c.f. Figures 2, 3, 4). A Rietveld refinement in Profex software was carried out to quantify the constituents of the mixture and consequently the chemical composition (Table 1).

## Results & Discussion

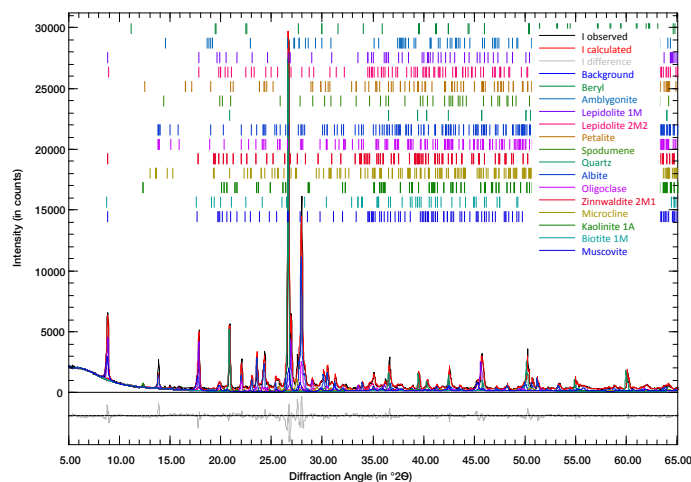
By identifying and quantifying the constituents of the Li ores, it is possible to determine their mineralogical composition. The chemical formulas of the phases are a prerequisite in a Rietveld refinement, which allows to calculate the elemental composition of the mixture. Often the exact composition of phases is unknown due to existing solid solutions and therefore such calculations bear some uncertainties. Nonetheless, in planning the beneficiation process, it is useful to use such calculated values. Here, we calculate Li content of 2.5 wt% (sample 1, Figure 2), 0.7 wt% (sample 2, Figure 3), and 0.7 wt% for sample 3 (Figure 4), which are representative of high-, and lower-grade Li ores.



**Figure 2:** XRD pattern of sample 1 with a Li content of 2.5 wt% calculated by refinement.



**Figure 3:** XRD pattern of sample 2 with a Li content of 0.7 wt% calculated by refinement.



**Figure 4:** XRD pattern of sample 3 with a Li content of 0.7 wt% calculated by refinement.

Table 1: Results of quantitative analysis (Rietveld method) of 3 Li hard rock ore samples).

Sample 1				Sample 2			Sample 3		
	Phase	Wt%	Li	Phase	Wt%	Li	Phase	Wt%	Li
Minerals with Li	Amblygonite	2.1	0.10	Amblygonite	3.1	0.15	Amblygonite	2.9	0.14
	Lepidolite1M	0.3	0.00	Lepidolite1M	3.4	0.09	Lepidolite1M	8.4	0.23
	Lepidolite 2M2	6.0	0.14	Lepidolite 2M2	1.8	0.04	Lepidolite 2M2	7.4	0.17
	Petalite	0.4	0.01	Petalite	12.3	0.28	Petalite	1.4	0.03
	Spodumene	60.5	2.26	Spodumene	4.2	0.16	Spodumene	2.0	0.07
	Zinnwaldite 2M1	0	0	Zinnwaldite 2M1	1.9	0.01	Zinnwaldite 2M1	0.8	0.01
	Quartz	5.6		Quartz	20.4		Quartz	25.1	
	Albite	5.3		Albite	36.1		Albite	16.5	
	Muscovite	3.6		Muscovite	11.2		Muscovite	12.4	
	Microcline	4.4		Microcline	3.0		Microcline	6.1	
	Beryl	0.9		Beryl	0.1		Beryl	0.1	
	Nacrite	1.5		Kaolinite1A	1.8		Kaolinite1A	1.3	
	Anorthoclase	1.8		Biotite1m	0.8		Biotite1m	0.1	
	Anorthite	0.9		Vermiculite	0.1		Vermiculite	8.8	
	Hydroxyapatite	1.7							
	Phlogopite 1M	1.4							
	Boehmite	1.0							
	Hornblende	2.7							
	Sum	100	2.51		100	0.73		100	0.65

## Your Benefits

The ARL X'TRA Companion XRD yields data perfectly suited to quantify the constituents in Li hard rock ore samples. Utilizing a Rietveld refinement in a one-click refinement yields results in minutes, enables ease-of-use for operators and relieves training constraints. XRD allows assessing the grade of Li ores during exploration and beneficiation while reducing sample preparation efforts compared to elemental analysis techniques.

Learn more at [www.thermofisher.com/xtra](http://www.thermofisher.com/xtra)

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