Application Brief

Mining and Energy

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Assay of Alkali Metals in Pegmatite and Spodumene Ores by ICP-OES

Fast analysis of metals in fusion ore-digests using the Agilent 5800 Vertical Dual View ICP-OES

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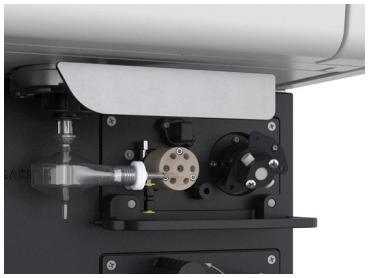
Surge in mining of pegmatite and spodumene ores to meet demand for lithium for use in lithium-ion batteries.



Close-up of the integrated Agilent AVS 6 switching valve.

High global demand for lithium

Mining of lithium (Li) deposits is increasing to meet demand for the metal from the lithium-ion battery (LIB) sector. Once extracted, ICP-OES is often used for the rapid, onsite analysis of representative Li ores. The technique offers excellent sensitivity and selectivity for Li, a high level of robustness, and stability over long runs. Typically ores are prepared using a fast, low-cost sample preparation procedure, such as sodium peroxide (Na_2O_2) fusion. The fusion digestion method is versatile, can break down some refractory minerals, and is commonly used at mine sites.

An Agilent 5800 VDV ICP-OES fitted with a six-port Advanced Valve System (AVS 6) switching valve was used for the accurate analysis of alkali group metals in Li ore samples. The 5800 was fitted with a SeaSpray concentric nebulizer, double pass cyclonic spray chamber, and an Easy-fit fully demountable, 1.8 mm injector torch. Li, potassium (K), and rubidium (Rb) were measured in radial view mode using a plasma RF power of 0.9 kW and a nebulizer flow of 0.85 L/min.

Five Li ore certified reference materials (CRMs) containing various amounts of spodumene, petalite, or lepidolite were used to evaluate the method. The CRMs included NIST 182, NIST 183, and OREAS 752, OREAS 148, OREAS 999 from Ore Research. Concentrations of Li_2O, K_2O, and Rb_2O in the CRMs ranged from 0.03 to 8%.

Simple and effective sample preparation method

Fusion was done in zirconium (Zr) crucibles with 0.125 g of sample and 1.5 g of ≥97% pure Na_2O_2. The mixture was fused just above the melting point of Na_2O_2 for 30 s. Once cooled, it was solubilized with deionized (DI) water, acidified with 10 mL of aqua regia, diluted to a final volume of 100 mL with DI water, and filtered.

Fast, safe, robust sample handling

The CRMs were rapidly prepared using the Na_2O_2 fusion method. Despite the high levels of silica (SiO_2) and alumina (Al_2O_3) in the CRMs (~70% and 15% respectively), excellent recoveries were achieved for group 1 elements using the Na_2O_2 fusion method (Tables 1 and 2).

The integrated AVS 6 sampling valve of the 5800 VDV ICP-OES reduced the exposure of the sample introduction system to the high matrix samples, enhancing the robustness and speed of the analytical method.

Accurate quantitative results

Fusion-digests of the five ore CRMs were introduced to the 5800 ICP-OES by the AVS 6 and the Agilent SPS 4 autosampler. The quantitative results in Tables 1 and 2 show excellent recoveries (98 to 108%) for Li, K, and Rb in the five CRMs. The recoveries confirm the suitability of the fusion sample preparation procedure and the accuracy of the 5800 VDV ICP-OES method for the measurement of the three elements in Li ore. The Na_2O_2 flux contains small amounts of K and Rb impurities, which may limit the accuracy for these elements when present at a low level in the samples.

Table 1. Average measured results for Li_2O and K_2O in the fused CRMs.

CRM	Certified Value (%)	Measured Value (%)	Recovery (%)
	*Li_2O		
NIST 182 (Li ore, petalite)	4.34	4.37	101
NIST 183 (Li ore, lepidolite)	4.12	4.09	99
OREAS 752 (Li pegmatite)	1.52	1.49	98
OREAS 148 (Li ore, spodumene)	1.03	1.01	98
OREAS 999 (spodumene concentrate)	5.76	5.77	100
CRM	**K_2O		
NIST 182 (Li ore, petalite)	0.1	0.1	100
NIST 183 (Li ore, lepidolite)	8	8	100
OREAS 752 (Li pegmatite)	2.53	2.55	101
OREAS 148 (Li ore, spodumene)	1.81	1.89	105
OREAS 999 (spodumene concentrate)	0.63	0.62	99

**Li 610.365 and 670.783 wavelengths used for MultiCal calibration with fitted background correction (FBC). **K 769.897 wavelength with FBC.*

Table 2. Average measured results for Rb_2O in the five fused CRMs.

CRM	Certified Value (%)	Measured Value (%)	Recovery (%)
	*Rb_2O		
NIST 182 (Li ore, petalite)	0.03	0.03	100
NIST 183 (Li ore, lepidolite)	3.5	3.5	100
OREAS 752 (Li pegmatite)	0.072	0.075	103
OREAS 148 (Li ore, spodumene)	0.149	0.150	101
OREAS 999 (spodumene concentrate)	0.046	0.050	108

**Measured as Rb 780.026 using Fast Automated Curve-fitting Technique (FACT) correction. The FACT algorithm in the Agilent ICP Expert software can automatically correct for emissions from interfering elements that could impact analytes of interest.*

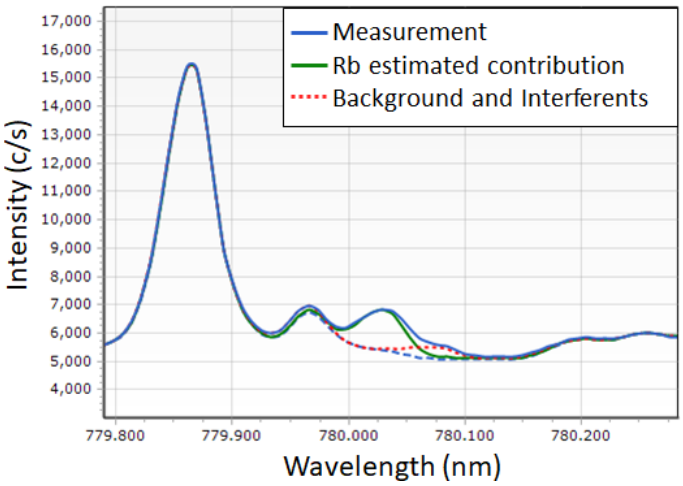


Figure 1. FACT provides an accurate model for correction of Rb 780.026, enabling the measurement of low concentrations of Rb_2O in rock ores.

Reliable routine ICP-OES method

The study has shown the excellent accuracy and precision of the Agilent 5800 VDV ICP-OES for the determination of Li_2O, K_2O, and Rb_2O in hard rock lithium samples.

The samples were prepared within a few minutes using Na_2O_2 fusion and aqua regia. The method produced highly accurate recoveries of Li, K, and Rb in the five ore CRMs—all within 100±8%.

Combined with the Agilent AVS 6 sampling valve, the 5800 ICP-OES provides mining facilities with the methodology, including effective background correction, needed for the routine, onsite analysis of Li, K, and Rb in geological samples.

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