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REFLECTION ZONE PLATE SPECTROMETER FOR LI K EMISSION DETECTION IN LITHIUM COMPOUNDS

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Motivation:

What?

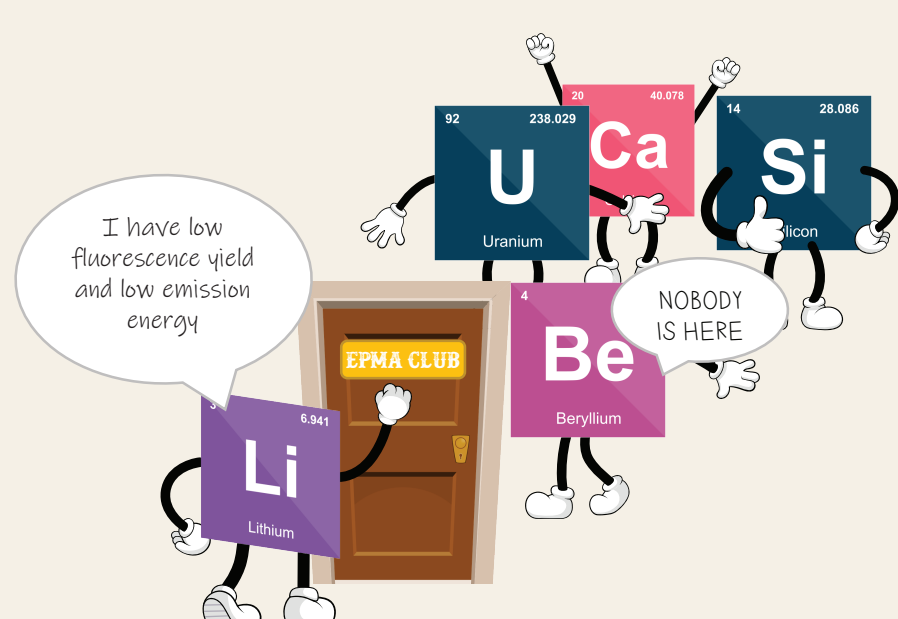
Unlock new frontier by allowing the **quantification of lithium** in complex materials such as lithium bearing minerals and materials used for **lithium batteries** with a technique that is both accessible and cost-effective.

The challenges:

- Low fluorescence yield
 - Low energy emission and
 - Strong self-absorption
- Make the detection with a **non destructive technique** an unresolved issue.

How?

- Probing the fine electronic structure of the valence band using soft x-ray emission spectroscopy technique (SXES) integrated in EPMA
- Tracking the changes in the chemical environment and emission spectrum (Intensity, shape, shift, band width...)



Can we detect lithium and study its electronic structure in complex materials?

Yes!

Methods:

The x-ray emission process:

1. **Excitation:** The sample is being bombarded with and accelerated beam of charged particles (electrons).
2. **Ionization:** The interaction of the beam of electrons with matter leads to the ionization of the core states.
3. **Emission:** the vacancy is filled with an electron from an outer shell, resulting in the emission of an x-ray photon that correspond to the energy difference between the two states.

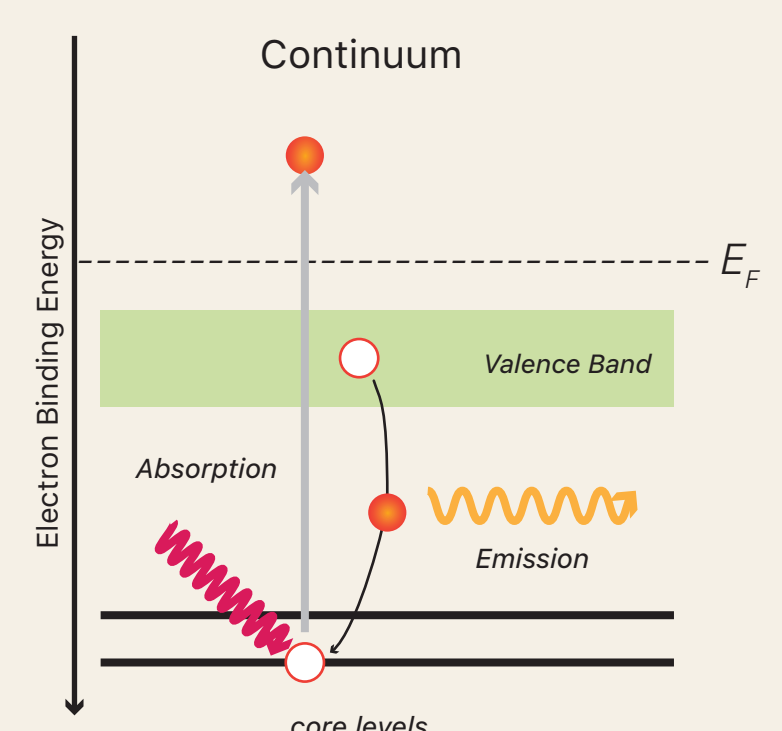


Figure 1: Illustration of the X-ray emission process

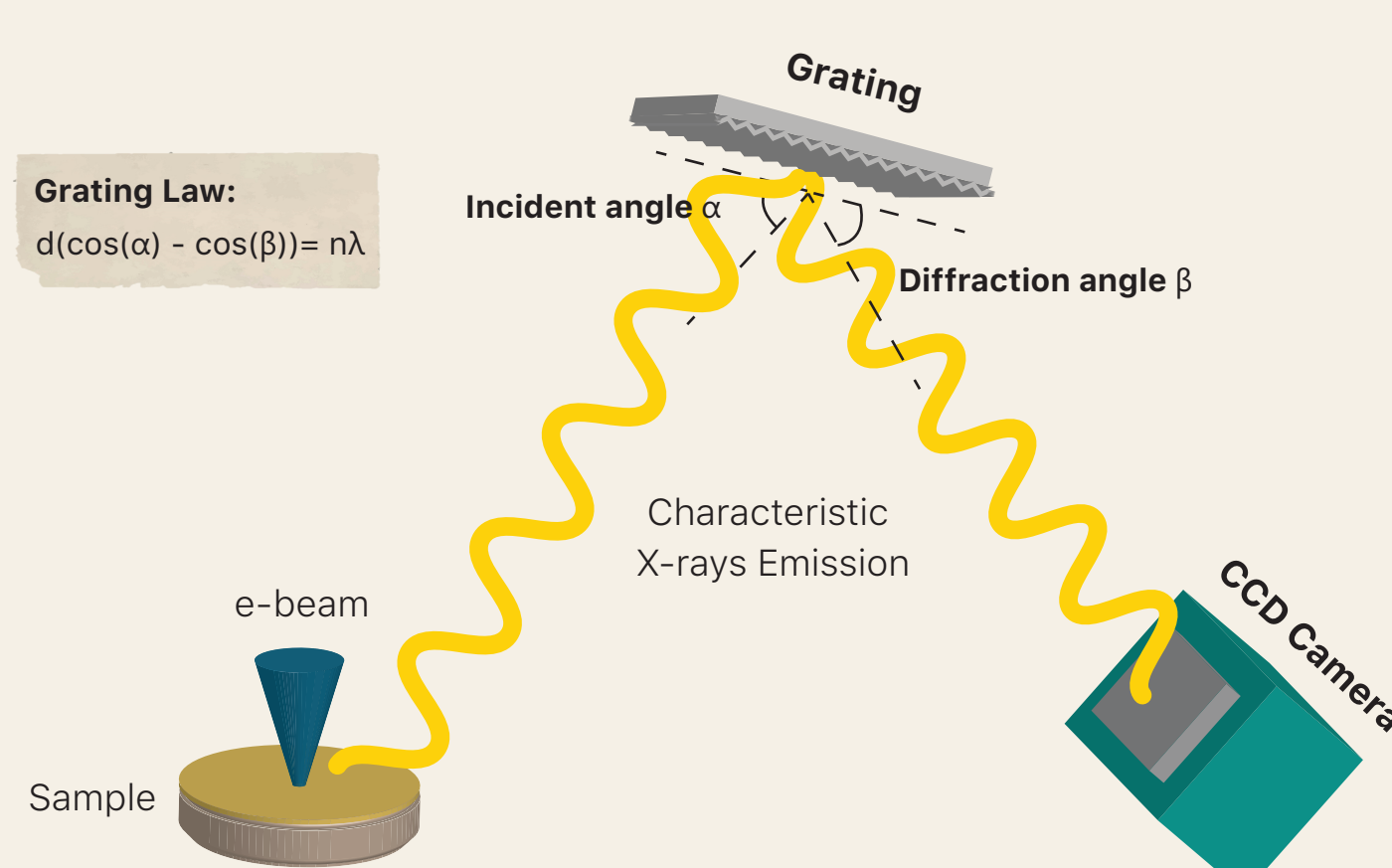


Figure 2: Wavelength dispersive spectroscopy (WDS) technique implemented inside the EPMA to acquire emission spectra

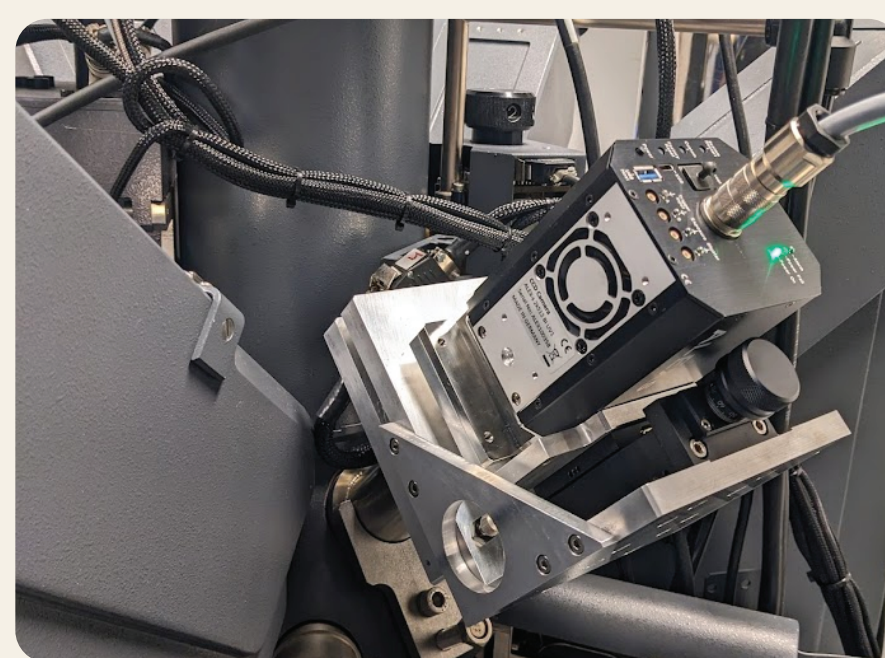


Figure 3: Image of reflection zone plate spectrometer mounted on a electron probe microanalyser (Cameca SX100).

Why RZP instead of Multilayers?

- No scanning mechanism
- Higher resolution (~0.5 at Al L2,3 edge and ~0.25 eV at Mg L2,3 edge)
- Cover wider energy range

Detection:

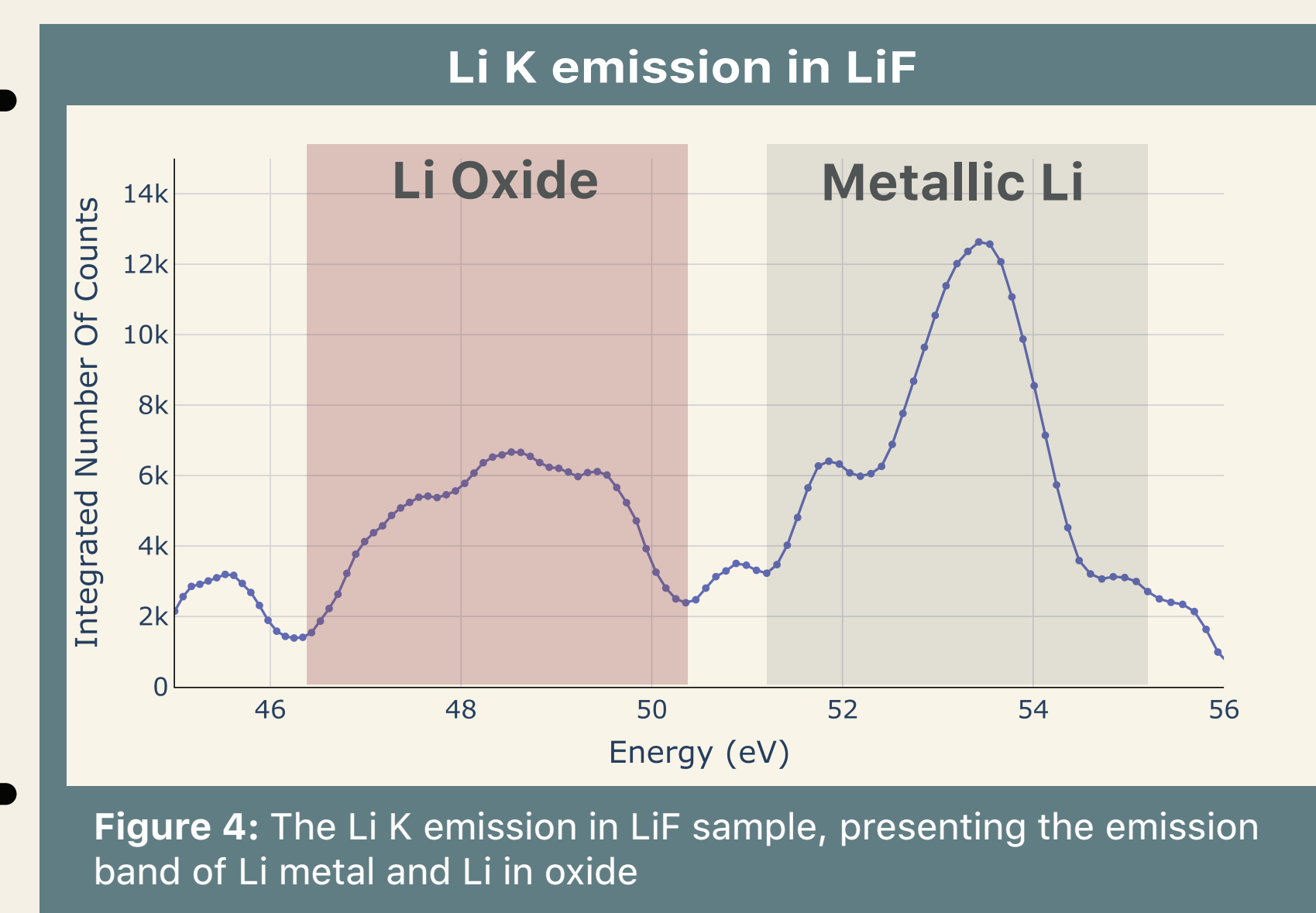
Reflection zone plates (RZP) are used in XES as a wavelength-dispersive element [1]. They consist of a special lamellar groove profile, each with a slightly different thickness. When x-rays interact with the zone plate, the different zones act as diffraction gratings, resulting in the x-rays diffracting and interfering with each other while focusing them at the same time.

Sample analysis in three easy steps:

1. **Polishing**
To remove the surface contaminations
2. **Coating**
Sputter coating for non conducting samples
3. **Analyzing**
Introducing the sample into the microprobe

Results:

The Li K emission occurs when a **2p** valence states transition to **1s** core states in lithium atoms and therefore the spectrum reflects the distribution of the p states within the valence band [2]. The Li K emission of lithium in LiF, Lepidolite $K(\text{Li,Al})_3(\text{Si,Al})_4\text{O}_{10}(\text{OH})_2$, Bikitaite $\text{LiAlSi}_2\text{O}_6(\text{OH})_2$, and Amblygonite $(\text{Li,Na})\text{AlPO}_4(\text{F,OH})$ is presented below.



Accumulation of Li on the surface after F being driven off the sample resulting to an emission identical to Li metal.

Figure 4: The Li K emission in LiF sample, presenting the emission band of Li metal and Li in oxide

X-ray emission spectra of minerals

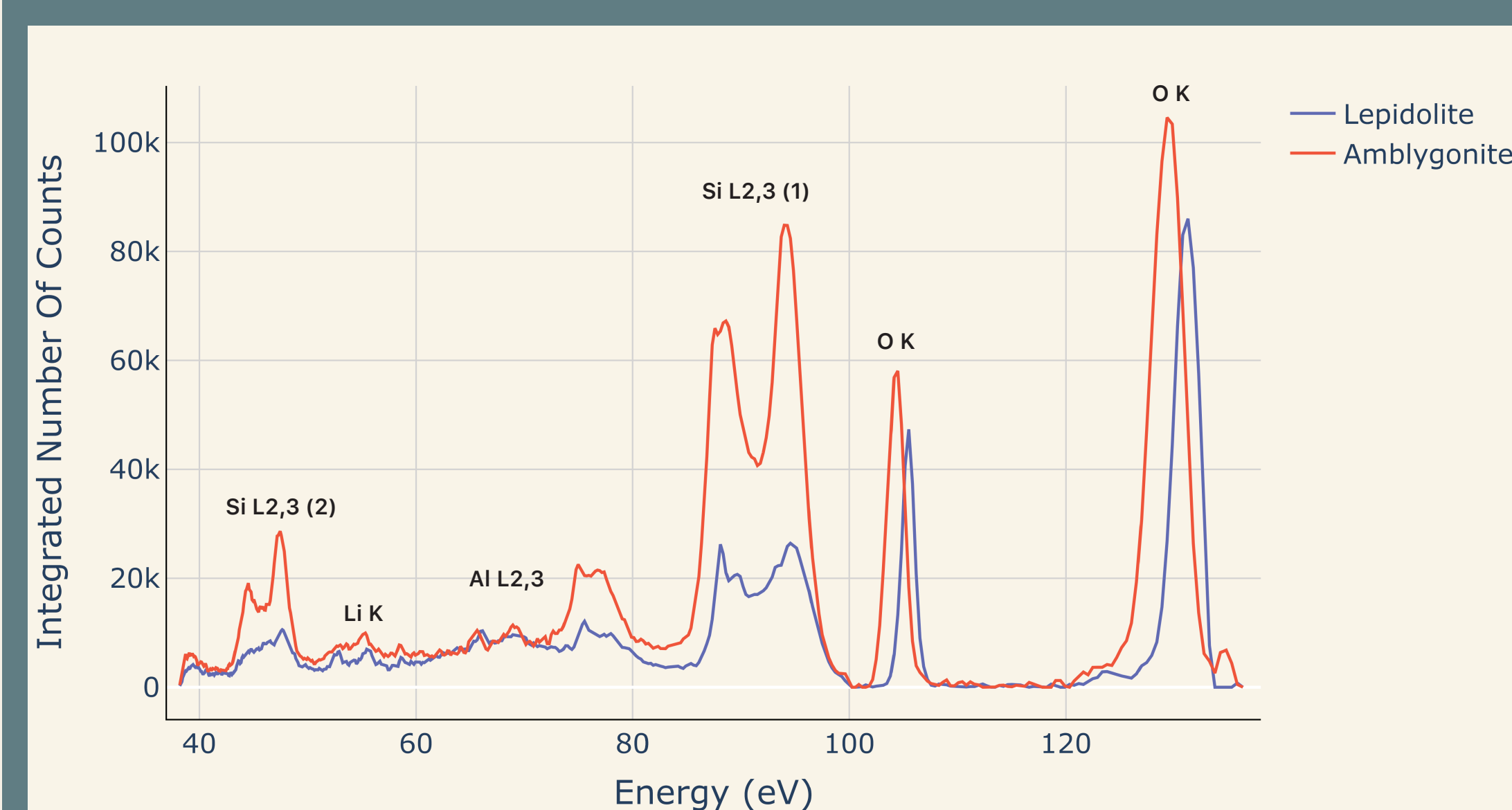
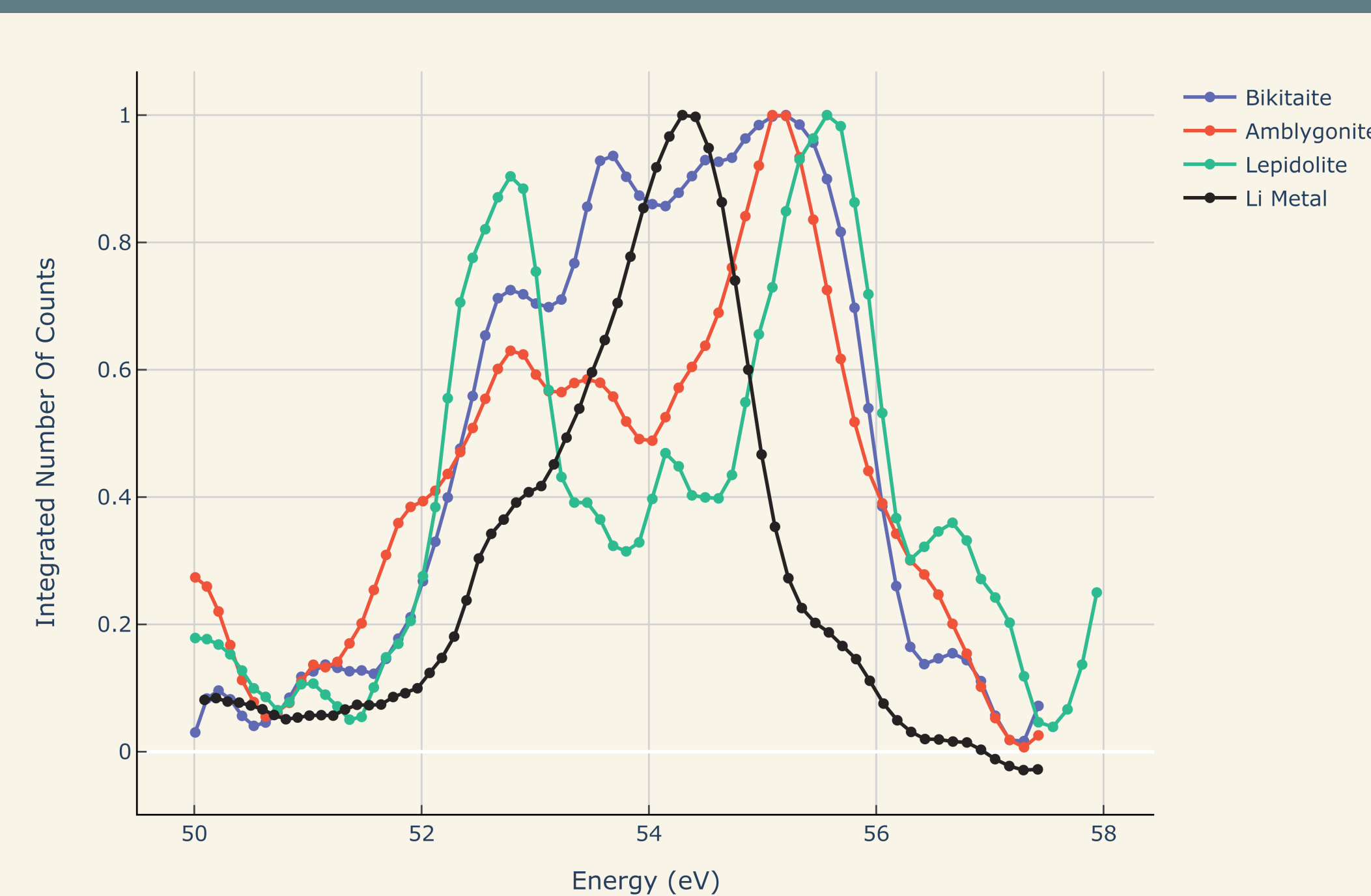


Figure 5: The x-ray emission spectra of Lepidolite and Amblygonite in the full range of the spectrometer

Li K emission reported for the first time in minerals

Figure 6: The Li K emission in the minerals: Lepidolite, Amblygonite, and Bikitaite.



Conclusion:

We demonstrate the possibility to spectroscopically analyze Li K emissions in different compounds using a non destructive and accessible technique. **We also report for the first time the Li K emission bands in minerals.** Our results unlocked potential for various applications to study in details the fine structure of valence band of lithium.

References:

- [1] A. Erko et al., Opt. Express, vol. 22, no. 14, p. 16897, Jul. 2014, doi: 10.1364/OE.22.016897.
 [2] K. Hassebi et al., X-Ray Spectrometry, doi: 10.1002/xrs.3329



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