XRD investigations in geological and mineralogical fields

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Figure 1. ARL X'TRA Companion X-ray diffraction system with θ/θ Bragg-Brentano geometry, perfect for all geological and mineral analyses.

Introduction

X-Ray diffraction (XRD) is a widely utilized analytical technique for determining the phase composition of crystalline materials. It is particularly relevant in the fields of mineralogy and geology, where it is used to identify the mineralogical content of geological samples, including sediments. Typically, XRD is a bulk analysis method, examining areas several square millimeters in size. However, specialized instruments are available that can perform XRD on very small regions, known as microdiffraction.

What are the principles of XRD?

X-ray diffraction operates on the principle that when X-rays pass through a crystal, they interact with the atoms within the lattice, leading to constructive and destructive interference of the X-ray waves. This interaction produces a distinctive pattern of the diffracted X-rays, which can be detected and analyzed based on **Bragg's Law (figure 2).**

Each crystalline mineral has a unique and repeating atomic structure. The specific arrangement and types of atoms within the mineral create a distinct structural pattern.

XRD can be used to determine the lattice parameters, which serve as a unique fingerprint for each mineral, enabling precise identification.

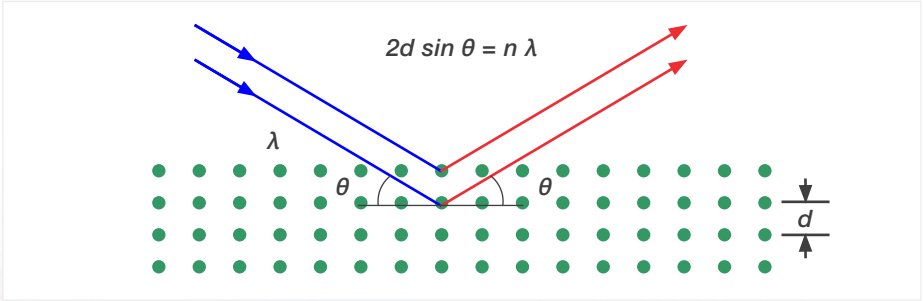

Figure 2. Bragg's law schema.
λ: wavelength, d: d spacing, θ: diffraction angle, n: diffraction order

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What type of instrumentation is used?

The benchtop X-ray diffractometer, **Thermo Scientific™ ARL™ X'TRA Companion** system (see Figure 1), is the ideal companion for XRD analysis in geological and mineralogical fields. Robust, very safe, and easy to use, it enables the establishment of a simple and relevant experimental protocol for high-quality data acquisition in a short time. The results obtained are easily interpretable thanks to open-source software and free crystallographic databases.

Ideally suited for continuous high-throughput operation, this system integrates automation solutions to minimize operator intervention. It can be configured to operate in batch mode, providing one-click quantification results based on the Rietveld method, with the option for automated transmission of results to a LIMS.

What are these main characteristics?

The **ARL X'TRA Companion** XRD system features a θ/θ goniometer (see figure 3) with a 160 mm radius in Bragg-Brentano geometry.

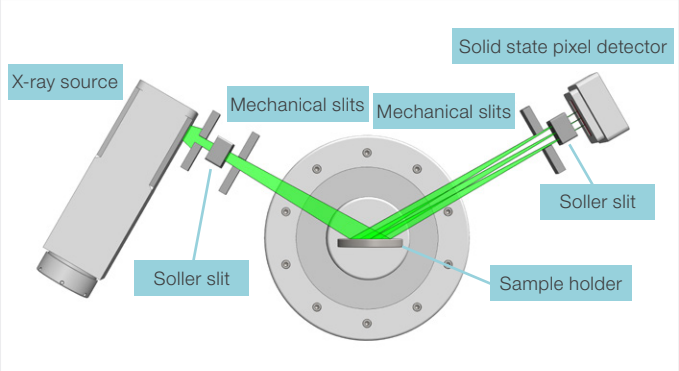

Figure 3. θ/θ geometry of the goniometer.

It is equipped with a 600 W X-ray source (available in either Cu or Co) and could be used with either a single sample holder or a sample changer according to the analysis flow. Then, various sample supports are available to accommodate different types and shapes of samples. The instrument could also include an integrated water cooler, making it fully autonomous.

Equipped with a innovative semiconductor pixel detector with a 55 x 55 µm pitch, the **ARL X'TRA Companion** XRD enables very fast and high-resolution data collection, ensuring reliable data interpretation.

How to prepare the sample?

Rock samples are crushed and ground to a particle size of approximately 100 µm, providing a test sample that is more representative of the entire sample. The resulting powder is then carefully placed in a suitable sample holder. This step is crucial because minerals with specific morphologies, such as clays with plate-like particles, can inadvertently orient themselves, leading to a preferred orientation that can distort quantitative results.

Figure 4. Sample collection before laboratory analysis.

How to perform phase identification and quantification?

XRD software includes search and match capabilities that interface with crystallographic databases of known minerals, such as ICDD and COD, to facilitate the identification of minerals based on the diffractogram generated by your sample.

Quantitative analysis relies on the relative intensity of the peaks corresponding to each phase or mineral present. The relative proportions of each phase can be accurately determined using the Rietveld analysis.

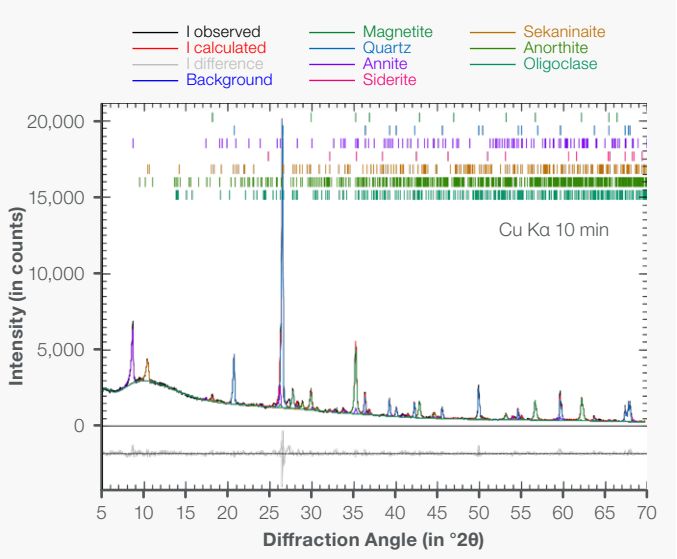

Figure 5. Determine total Fe and Fe^{2+} / Fe^{3+} ratio with ease.

Phase	wt. %	Description
Magnetite	15.50	Fe oxide
Quartz	52.60	
Annite	6.81	Fe bearing minerals
Siderite	0.23	
Sekaninaite	2.89	
Anorthite	15.30	Feldspars
Oligoclase	6.73	

Table 1. Mineralogy.

	wt. %
Fe total	13.84
Fe^{3+}	7.48
Fe^{2+}	6.36

Table 2. Fe species.

What would be the minimal configuration of the X'TRA Companion XRD for this work?

To ensure optimal performance with minimal requirements, the configuration of the ARL X'TRA Companion XRD will be relatively simple:

- **ARL X'TRA Companion** advanced benchtop XRD system for geological analysis
- Standard single sample holder or sample changer following the sample flow
- Open-source PROFEX software for free access to all users
- ICDD or COD* crystallographic databases
  ** Free open database*
- Additional dedicated software: MATCH! for the search match, MAUD or FULLPROF (free) for the Rietveld method

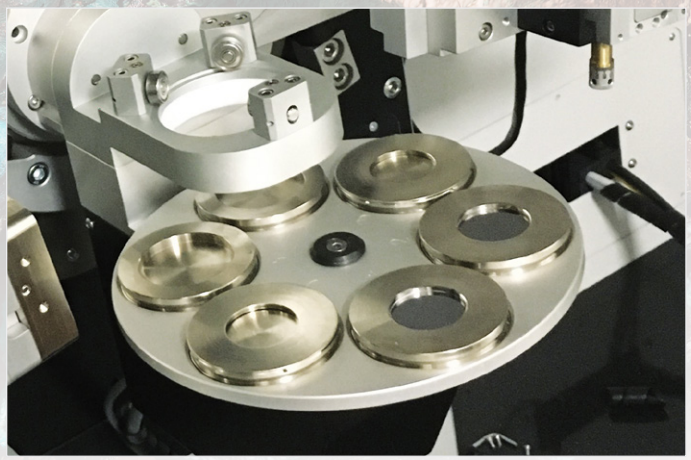

Figure 6. 6-position sample changer.

Do you know any complementary technique?

Although both techniques use X-rays and have similarities, XRF and XRD produce different but complementary data. XRD identifies minerals and determines phase assemblages, while XRF determines elemental compositions. XRF detects elements such as aluminum (Al) and silicon (Si) and quantifies their concentrations, often down to ppm levels, but it does not reveal specific compounds. XRD, however, can identify compounds such as SiO2, Al2O3, or Al2O3·2SiO2·2H2O (kaolinite) and quantify their percentages using Rietveld analysis. Therefore, XRF can be combined with XRD to simplify the identification of unknown phases and provide more accurate quantitative results through Rietveld refinement.

What would be your conclusion about this XRD instrument?

The **ARL X'TRA Companion** XRD benchtop system is a powerful tool in geology, specifically designed to assist researchers and geologists in understanding the mineralogical composition of geological materials. Suitable for use in both academic and industrial settings, its robustness, high-quality results, and user-friendly interface make it ideal for experiments focused on identifying and quantifying crystalline phases. The data obtained with the ARL X'TRA Companion XRD is valuable across all geological sectors, from academic research aimed at better understanding our environment to exploration geology and the quality control of finished products.

Data can be easily combined with those obtained using the **Thermo Scientific™ ARL™ QUANT'X** Benchtop EDXRF Spectrometer.

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