



Article



XRD or XRF?

Deciding Which Technique is Best for Your Material Characterization Needs

For years, X-ray analysis has been used by manufacturers to study and characterize materials for research and quality assurance. Two popular techniques that incorporate X-ray analysis for sample analysis are X-ray Fluorescence (XRF) and X-ray Diffraction (XRD). Both XRD and XRF techniques measure a sample's response to the X-rays, but provide different types of measurements.

XRF provides the elemental data for chemical composition without differentiating between the different chemical compounds that are present in the sample. XRD identifies and measures the presence and amounts of minerals and their species in the sample. XRD is also used to analyze phase composition and structure.

Although XRF and XRD provide different information, they complement one another and give a total picture of sample composition. A major advantage of both XRF and XRD compared to other elemental analysis techniques is that they are non-destructive. This means that the sample can be recovered for other confirmatory tests.

First, in order to better understand the differences between XRF and XRD it is important to have clarity on the two techniques.

Why use XRD for material characterization?

All matter is composed of atoms, which show a degree of repetition in their structural arrangements. The scattering of a homogeneous X-ray beam by collision with electrons of atoms results in a diffraction pattern. This diffraction pattern is dependent on the wavelength of X-rays and the distance between the planes of the atoms arranged in such arrays.

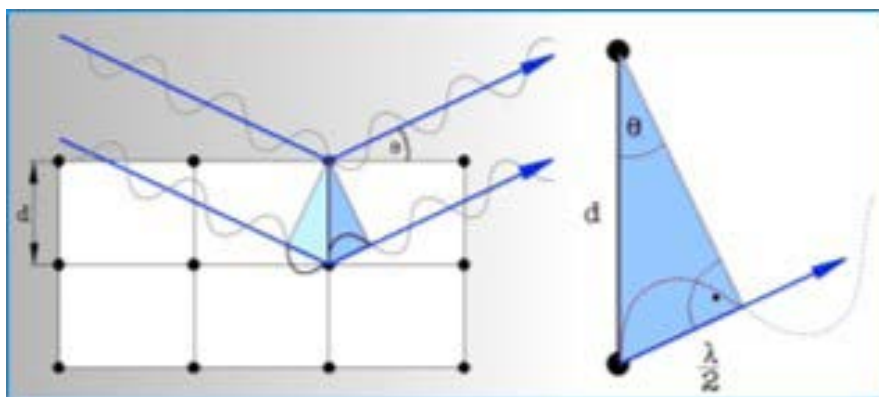


FIGURE 1: BRAGGS LAW PROVIDES A SIMPLISTIC MODEL TO UNDERSTAND WHAT CONDITIONS ARE REQUIRED FOR DIFFRACTION.

The study of the diffracted X-ray patterns provides valuable details on structural arrangements of crystals in unit cells or lattices unlocking the material's atomic structure. Qualitative analysis is possible by comparing the XRD pattern of an unknown material with a library of known patterns.

Each of these angles will produce a maximum (peak) on the diffraction picture (diffractogram) and for every mineral there exists a set of characteristic peaks, by which

the mineral can be identified (see Figure 1). Both the diffraction angle and the intensity of each of the diffracted beams (diffraction peaks) are then measured, processed, and counted. The intensity of diffracted X-rays is plotted as a function of diffraction angle 2θ .

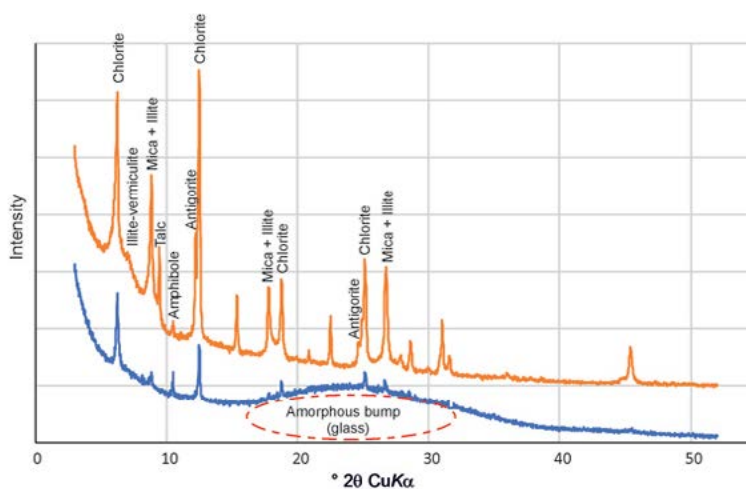


FIGURE 2: DIFFRACTOGRAMS OF MINERAL MIXTURES

XRD is a proven technique for characterizing materials for a wide range of applications such as:

What is in my sample?	Identification of single or multiple phases in an unknown sample.
How much is there?	Quantifying the amount of crystalline phases present in a mixture and amorphous content evaluation.
What is the crystallite shape?	Microstructure analysis.
What is the crystal structure?	Structure determination and refinement

Where is XRD routinely used?

XRD is utilized in a wide range of industries, including geology & mining, cement, ceramics & refractories, pharmaceuticals, polymers, paints & coatings, environmental, and forensics.

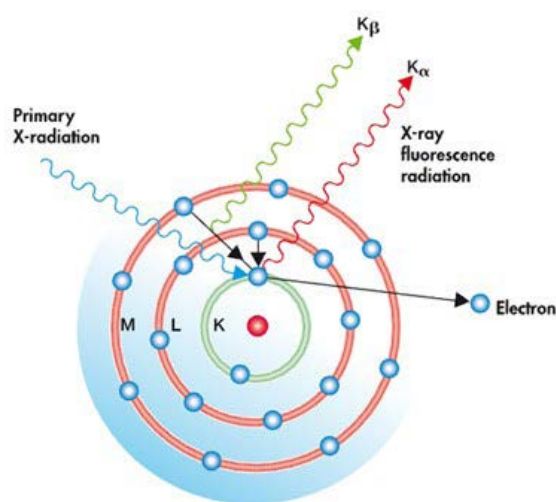
What are the advantages and disadvantages of XRD?

Advantages	Disadvantages
<ul style="list-style-type: none">• Fast identification of materials• Easy sample preparation• Large database of known crystalline structures• Computer-aided material aided material identification	<ul style="list-style-type: none">• Impossible to identify directly amorphous materials• Quantitative analysis needs grinding and is limited down to 0.1 w%• Does not determine chemistry of a sample

Why use XRF for material characterization?

We understand that XRD is a useful tool for measuring minerals and phases, but it does not determine the chemistry. This is a strong benefit to using XRF for material characterization. XRF spectrometers determine the elemental composition of a sample by measuring the fluorescent (or secondary) X-rays emitted from a sample when it is excited by a primary X-ray source.

FIGURE 3: ATOMIC MODEL FOR THE X-RAY FLUORESCENCE ANALYSIS METHOD



Each element in a sample produces a set of characteristic X-ray photons that are unique for a specific element, presenting a specific energy (keV). This is the equivalent to a fingerprint. The peak energy identifies the element, and the peak height / intensity is indicative of its concentration.

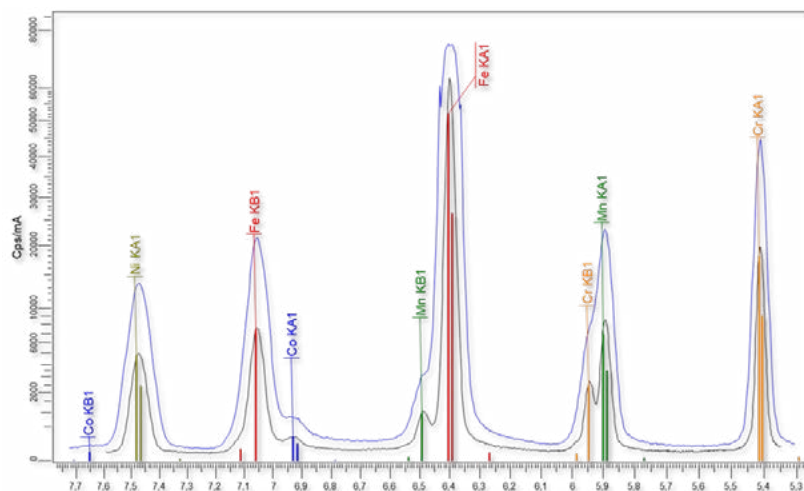


FIGURE 4: EXAMPLE OF XRF SPECTRA

XRF permits qualification and quantification of both metallic and nonmetallic elements of the periodic table from beryllium (Be) to americium (Am) in concentration ranges from 100 wt % to sub-ppm levels. XRF analysis is a robust technique, combining high precision and accuracy with straightforward, fast sample preparation. No acids and chemicals are necessary since the samples do not need to be dissolved into a liquid or diluted.

Where is XRF routinely used in materials analysis?

Materials testing has been dominated by atomic absorption spectrometry (AAS) and inductively coupled plasma (ICP) techniques for several years. But as industries look for alternative elemental analysis techniques that are less time consuming and require less sample preparation, this has created a strong value proposition for the use of XRF. XRF has a long history analyzing materials in industrial settings, such as metals and alloy manufacturing, geology and mining, cement manufacturing, petroleum and chemicals, plastics and polymers, food and agriculture, environmental, and much more.

What are the advantages and disadvantages of XRF?

Advantages	Disadvantages
<ul style="list-style-type: none">• XRF is a fast analysis (seconds up to 10 min), making it a useful research and development tool.• Quantitative from ppm to 100%.• Can analyze both solids and liquid samples.• It is non-destructive, so you can test sample materials without posing any risk to them.• XRF allows for simple and fast sample preparation, and it has low running costs.• XRF spectroscopy instruments do not require daily re-calibration.	<ul style="list-style-type: none">• The reliance of XRF on quantity can limit measurements, with 5 to 10 mL being the typical volume required for best performance.• Limitations on measuring lighter elements.• The highest accuracy measurements require reference standards similar in composition and/or thickness to the test sample.

Is XRD or XRF better for material characterization?

Before beginning to analyze a material, it is important to consider what you want to determine from the analysis. Successful analysis depends on the interpretation and understanding of the data. Manufacturers are increasingly developing instruments that are easier to operate. Therefore, the value of a technique lies in its rigorous interpretation, something that can only be achieved by an understanding of the scientific principles behind the analysis. Ideally a variety of techniques would be used together as they build a more complete understanding of the range of materials present. And until a single analytical technique becomes capable of identifying all materials, consideration should be given to seeing the complementary nature of XRF and XRD techniques.

References:

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