



XRD SHOWS IT ALL

X-ray diffraction (XRD) is among the most powerful non-destructive techniques for analyzing crystalline materials. It provides the molecular composition of a wide range of samples, in areas as diverse as mining, metallurgy, pharmaceutical, nanotechnologies and aerospace. XRD can also determine the three-dimensional structure of a substance at the atomic level, for isolated crystals.

Crystalline and amorphous materials

Since XRD can only be used with crystalline materials, it is important to clarify what a crystal is: it is a solid material whose atoms or molecules are organized in a regular three-dimensional arrangement. Some well-known examples of crystals include diamond, salt and silica. Materials that do not show a long-scale periodical structure are called amorphous; glass is such a material, as are some polymers.

Working principle

In a diffractometer (Figure 1), X-rays bombard the sample and are diffracted at different angles, as a function of the crystalline planes of the sample. The diffractometer has a detector that measures the intensity of the diffracted X-rays as a function of the angle θ , while the sample and the detector turn synchronously. The data collected are then plotted in a graph of the intensity as a function of 2θ , which gives a series of “peaks” that we usually call a diffraction pattern or spectrum (Figure 2)

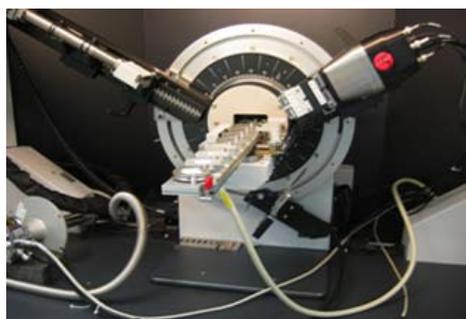


Figure 1: Diffractometer with X-ray source (to the left), a sample holder (in the center) and a detector (to the right).

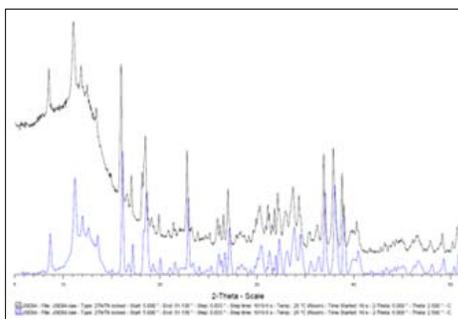


Figure 2: Diffraction spectrum of a mineralogical sample composed mainly of zinc chloride.

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XRD IN A FEW WORDS...

Sample type:

almost any type of solid (polymer, thin films, metals, minerals, etc).

Elements detected:

XRD does not detect specific elements but precise phases. For instance, XRD will differentiate zinc chloride from zinc oxide.

Detection limit:

typically about 1%.

Uncertainty:

The uncertainty can vary with the sample, but it is typically of the order of $\pm 5\%$.

Each chemical compound yields a diffraction pattern that is unique to its structure, much like a fingerprint. Accordingly, a mixture of compounds will give a complex diffraction diagram that shows the sum of the individual compounds. The work of the analyst consists of matching the experimental data with the diffraction spectra found in a database of known compounds. Oftentimes, the diffraction peaks do not match exactly the spectra of the database, and the judgement of a competent analyst therefore becomes critical.

Applications

- › Quantification of phases in minerals or metals
- › Polymorph analysis in the pharmaceutical industry
- › Identification of the preferred orientation of thin films
- › Analysis of the structure of vitamins, proteins and drugs
- › Phase identification
- › Semiconductor analysis

Advantages

- › Non-destructive
- › Quantitative measurements
- › Minimal sample preparation
- › Analysis under ambient conditions (no vacuum required)

WHY X-RAYS AND NOT LIGHT?

X-rays are used to produce a diffraction pattern because their wavelength is typically of the same order of magnitude as the spacing between crystalline planes. According to diffraction theory, to obtain a high intensity diffraction signal, the spacing between the diffracting structure and the wavelength of the incident electromagnetic wave must be similar. The wavelength of visible light is too long to produce an intense diffraction pattern in crystals.

CASE STUDY: ANALYSIS OF FABRIC FINISH

Recently, a company contacted the GCM to determine the metals composition of a fabric finish used in the textile industry. Preliminary analysis had revealed the presence of titanium and calcium. The customer suspected the presence of silicon and magnesium, at concentrations higher than 1%, which was ideal for X-ray diffraction analysis. The sample also contained polymers that would be invisible to XRD, due to their amorphous nature, but this aspect did not matter for this project.

The Centre de caractérisation et de synthèse moléculaire, an associate laboratory of the GCM, located at Université de Montréal, obtained the diffraction spectrum shown in Figure 3, in black. The blue lines show the diffraction pattern of CaCO_3 (calcite) while the red lines show that of titanium oxide (TiO_2). These patterns account for 36 of the 43 peaks observed, which is sufficient to conclude that CaCO_3 and TiO_2 are the main crystalline compounds of the sample. The remaining peaks (about 7) are probably due to a polymer or a silicon-based compound.

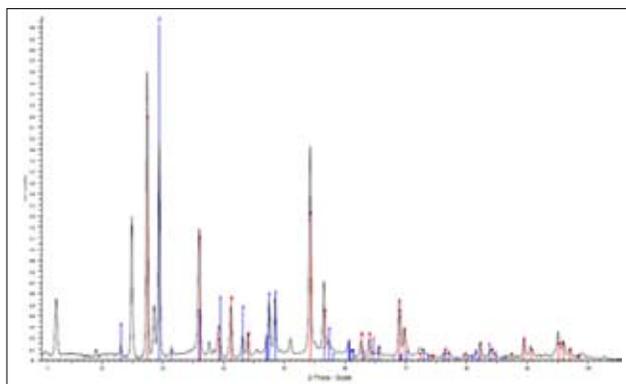


Figure 3: Diffraction spectrum of a fabric finish.