**Results**

Diffraction data collected with a 2D image detector records the Debye-Scherrer rings. These are integrated to give a 1D pattern and analysed to determine the crystal structure.

1. The sample is illuminated by a monochromatic X-ray beam and the diffraction is measured using a 2D detector.
2. From the 2D detector, a series of Debye-Scherrer rings are recorded.
3. The 2D rings are integrated to give a series of Bragg peaks in the corresponding 1D diffraction pattern.
4. Analysis of the diffraction pattern allows the crystal structure to be derived.

**Applications**

Total scattering and pair distribution function analysis have allowed powder diffraction to extend beyond crystalline solids, to short range order, for example in nanocrystalline or nanoporous materials. Further, powder diffraction may be applied to investigate in situ or in operando reactions. This is useful for understanding catalysts, fuel cells, batteries, pharmaceutical compounds and magnetic materials. Additionally, in the pharmaceutical industry, powder diffraction is considered the gold standard for characterizing pharmaceutical solid materials.

**Reproducibility and data deposition**

Determining the accuracy of a crystal structure is referred to as structure validation. Several approaches may be used for structure validation. For example, the quality of a refinement may be assessed using statistical calculations. Alternatively, experimental data can be compared to one of several online repositories, which together contain nearly two million reported structures. To upload data to a repository, the crystal structure must be converted to a Crystallographic Information File (CIF) format, which is then made available to the wider scientific community. Both published and unpublished datasets can be uploaded to these databases.

**Limitations and optimizations**

Powder diffraction is inherently limited because it compresses 3D, spatial data into a 1D pattern. The main consequence of this is peak overlap. As a result, there is a limit on the amount of information that can be extracted from the data, particularly for nanostructure features. Experimental optimization involves tailoring the instrumentation to the intended purpose, for example by changing the wavelength, step size or counting time.

**Outlook**

A key advantage of diffraction is that it may be used to structurally characterize a material during operation or under reaction conditions. This results in a large amount of experimental data. Future work will likely see an increase in advanced data processing techniques. In addition, as more structures are refined, diffraction databases continue to grow. New releases of, for example, the Powder Diffraction File database, provide greater confidence in both phase identification and structure validation.

**Experimentation**

Crystallography involves the diffraction of X-ray’s, neutrons or electrons to elucidate the structure of a crystalline material. The most common approach uses monochromatic X-rays as the incident beam. For a crystalline material, the most prominent features in the diffraction pattern are the Bragg peaks, the positions of which are determined by the Bragg equation:

\[ n \lambda = 2d \sin \theta \]

Where \( \lambda \) is the wavelength, \( d \) the spacing between crystal planes, \( 2\theta \) the diffraction angle and \( n \) the order of diffraction.

Different instrument geometries can be used to measure the angle of diffraction. Two common approaches are the Bragg-Brentano and Debye-Scherrer geometries. Both methods mount an X-ray source — often copper, cobalt, chromium, molybdenum or silver — to the incidence side of the instrument and an X-ray detector on the opposite side. Depending on the diffractometer configuration, a different specimen holder is required. This includes flat surfaces, thin sheets and filled capillaries.

However, for all experimental set-ups, the ideal specimen is a fine, smooth, homogeneous powder with randomly distributed crystallite orientations.

**Analysis of a diffraction pattern provides an insight into atomic arrangement, crystallite size, strain and texture. For a mixture of phases, powder diffraction gives qualitative information on phase identification and quantitative data about the concentration of each species present.**