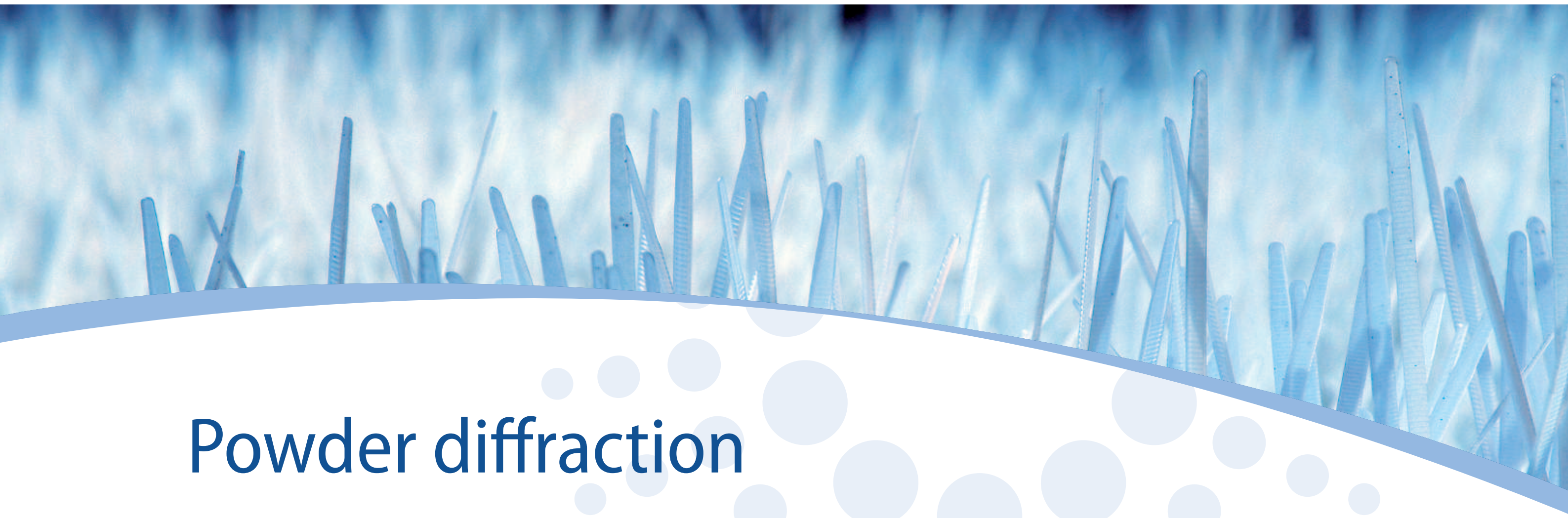


Powder diffraction

The technology at a glance

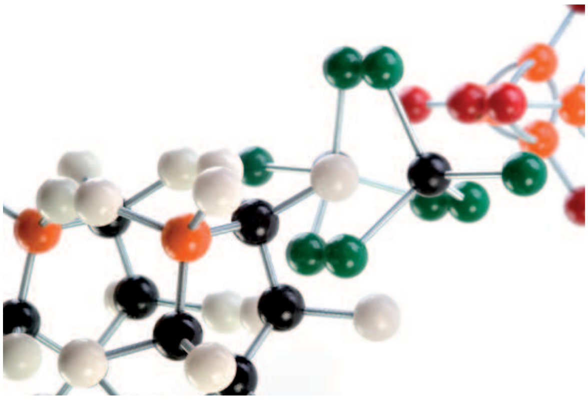
Many bulk solids are composed of microcrystals, which are hard to study by standard single-crystal techniques. Powder diffraction makes it possible to analyse the structures of such materials under a wide range of conditions, e.g. while heating or cooling, or under different atmospheric conditions. The positions, intensities and shapes of the peaks in the powder diffraction pattern reveal information about the microscopic structure and strain state of a sample, and can also be used to identify which substances are present in a (possibly complex) mixture. Such information is crucial for understanding the properties and behaviour of materials.

The added value of the ESRF powder diffraction facilities

At the ESRF, the synchrotron X-ray powder diffraction beamline delivers very accurate, high-resolution data, with the choice of a wide range of wavelengths, which can be used to investigate complex and highly absorbing materials. Furthermore, an automatic sample changer means that a series of samples can be quickly and efficiently studied. Finally, users have the possibility of coming to the ESRF and carrying out the experiment themselves with the support of the beamline staff or, for a small number of samples, can benefit from the facility's mail-in service, thus eliminating the need to travel and be present when the measurements are carried out.

"We can see things in the samples that people haven't even imagined were there."

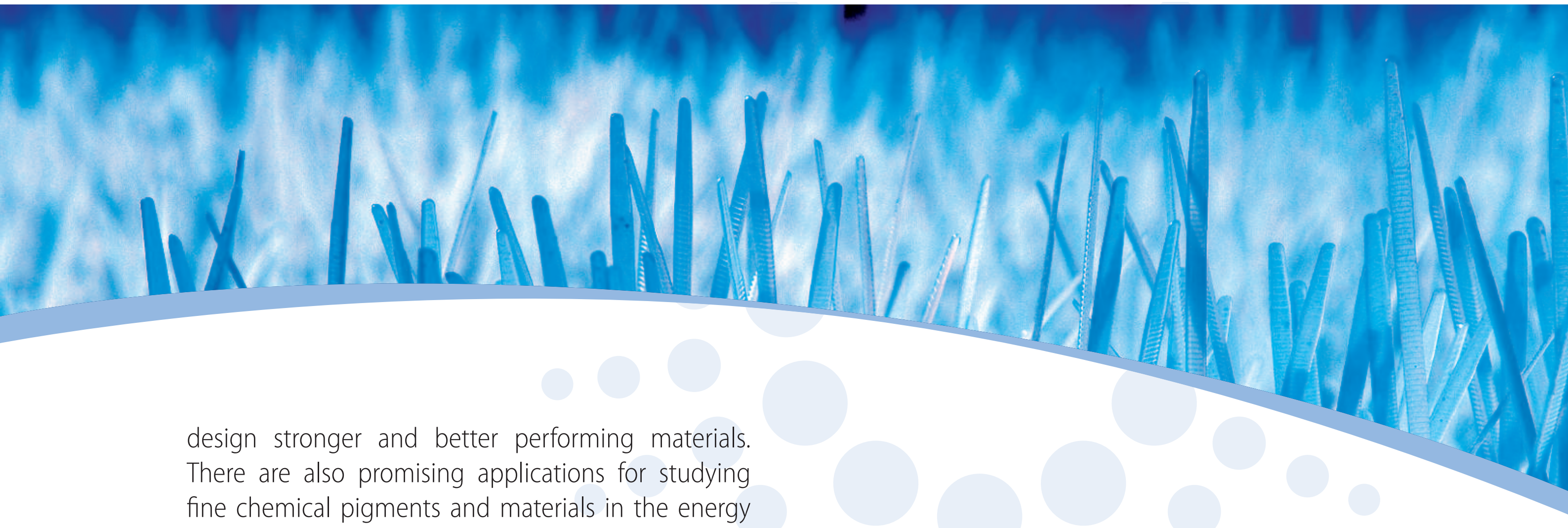
- Andy Fitch, Scientist in charge of ID31, the ESRF powder diffraction beamline



Fields of application

Pharmaceutical companies regularly use ESRF powder diffraction techniques to characterise pharmacologically-active components and other ingredients in formulations e.g. to investigate polymorphism and hence enable them to comply with regulatory requirements and to protect their intellectual property.

Steelmakers, aerospace companies, car manufacturers and other metallurgy-based industries use the facility to examine alloy structures, carry out stress and fatigue tests and thereby help



design stronger and better performing materials. There are also promising applications for studying fine chemical pigments and materials in the energy sector, such as for batteries, hydrogen storage and superconductors, as well as in nano-technology and other related materials.

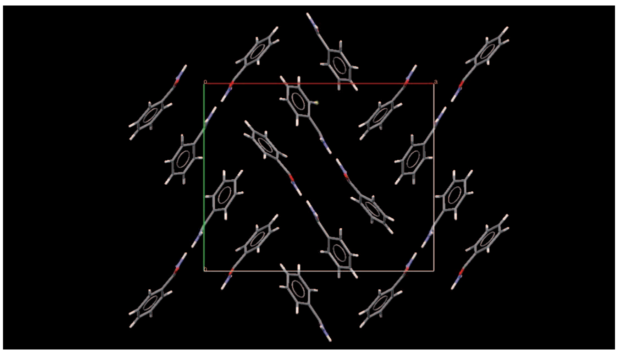
"ID31 gives me quality data that I can get nowhere else – *incomparable* ! "
- Sanofi-Aventis (Montpellier, France)

"When we come to the ESRF, we are always satisfied with the efficiency of data collection, but above all we really appreciate the great technical support we get here. When we can't come to the ESRF, we ship samples to them and we know they will run tests thoroughly for us. Last, but not least, the ESRF respects our intellectual property. Companies are really at home here and free to develop their technologies."
- a formulation scientist from a "Big Pharma" company, USA

CASE STUDY

Powder diffraction reveals the crystal structure of the metastable polymorph of benzamide.

The challenge: To obtain a crystal structure of metastable benzamide, unsolved for over 170 years.
Background: Understanding the influences of structural, thermodynamic and kinetic factors that control crystallisation processes is important for fields such as pharmaceuticals, health care, optoelectronics and speciality chemicals.
A metastable transient Form II of benzamide has been known since 1832, but the crystal structure proved intractable until very recently.
Results: The crystal structure was solved from high-resolution powder diffraction data from ID31. Numerous powder diffraction patterns were collected to monitor Form II in the sample, which also contained Form I. The structure showed that the polymorphism arises from the disorder of one of the independent benzamide molecules in the unit cell and highlights the delicate balance between kinetics and thermodynamics in the appearance of polymorphs.
How did the synchrotron help? Rapidly collected high-resolution powder diffraction patterns were the key to solving this structure, together with the *in situ* kinetic and temperature control of the sample. The very high angular resolution of the ID31 diffractometer allowed high-quality data to be collected with good separation of the powder peaks, which led to the final detailed crystal structure.
Reference: Blagden et al. Crystal Growth & Design **5** (2005), 2218-2224.



Crystal structure of Form II of benzamide showing the noncentric molecular dimer determined from the ID31 powder data.