What does X-Ray Diffraction do and what can we offer?

Wendy Hulse & Jade Hollis

Introduction to X-Ray Diffraction

The physical form of ingredients used in both food and pharmaceutical products can have a major impact on the desired properties of the product.

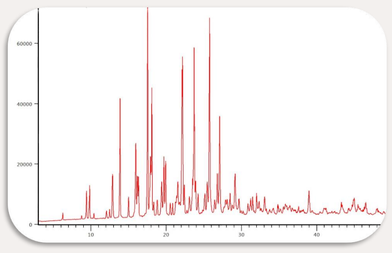
For example, conversion may occur from the least stable to the most stable form through processing or storage.Therefore, it is very important to understand the physical form of the ingredients throughout processing, formulation and storage of the final product.

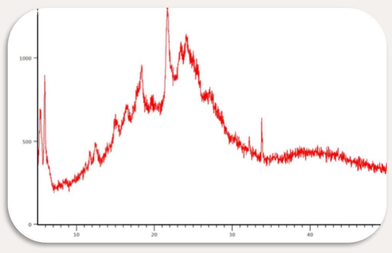
X-ray diffraction (XRD/XRPD) is the gold standard technique for physical form identification and can be used to ensure that any changes in physical form are highlighted. This can prevent product recalls and show instability before other techniques.
XRD can be used in conjunction with DSC, TGA, DVS and various microscopy techniques to highlight polymorphic forms and instabilities. It can be used throughout the entire product lifecycle from formulation to stability and beyond to release testing.

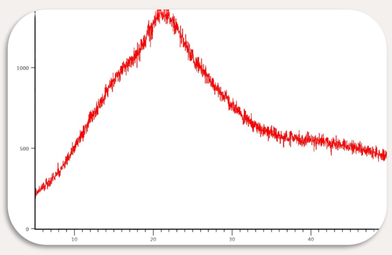
Commercial uses of XRPD – Common Issues

Basics of XRD - understanding what we can assess

Shown are three figures ranging from crystalline to amorphous with a mixed system in between. We can assess the level of crystallinity within a compound versus the desired attributes.







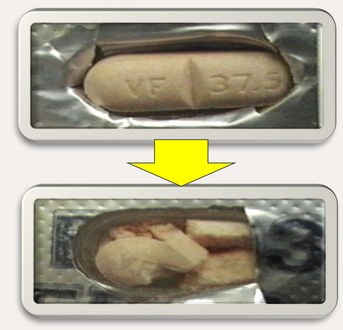
For example;

- Is the compound amorphous but degrading over time?
- Which polymorph is crystalizing out?
- Is the excipient or API crystalizing out?

XRD is a powerful tool to determine these factors this is particularly important in nasal/inhalation formulations as this can impact the dosing to patients.

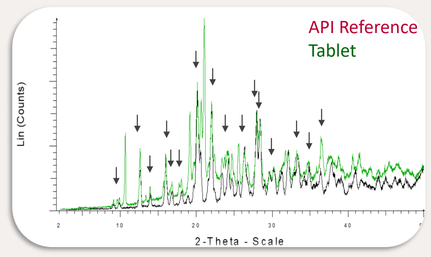
Basics of XRD – formulation applications

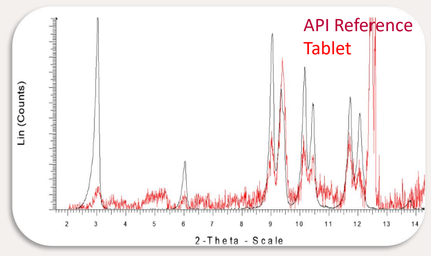
The identification of the physical form of an API in a tablet formulation can also be performed. This is important as formulation processes can cause changes to the physical form of the API which may affect efficacy and/or tablet stability



Likewise, the solvent used during formulation of manufacturing can have a large impact in determining the polymorph - RSSL can provide insights on this, and we offer polymorph screening

The two images below show API reference diffractograms against the tablet showing how we can identify API and interactions with excipients.

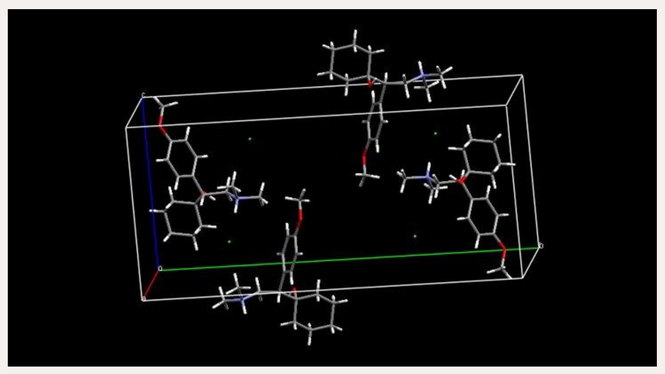


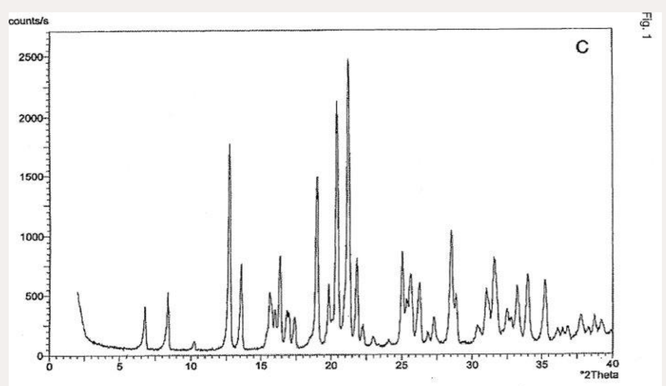


For high API dose strengths, it can often be very simple to identify the API and compare against a reference diffractogram. However, for lower API concentrations identifying the API diffraction peaks can be difficult. We can offer the complex method development required to identify an X-ray region in which the excipients do not interfere with the API peaks allowing fast identification and quantification.

Summary of service offerings at RSSL

- API solid form determination
- Stability testing and trending of degradation/crystallinity percentage over time
- Release testing
- Characterisation of amorphous solid dispersions – nasal/inhalation devices
- API polymorph and salt screening to identify the optimal physical form
- Identification of possible costly food and pharmaceutical processing issues before they arise. For example, tablets breakages and/or change in characteristics.
- Food and Pharmaceutical troubleshooting from formulation to consumer complaint
- Method development/validation for crystalline content and identification





Reading Scientific Services Ltd
The Reading Science Centre, Whiteknights Campus, Pepper Lane, Reading, Berkshire RG6 6LA • Tel: **+44 (0)118 918 4000** • Email: **enquiries@rssl.com** • **www.rssl.com**