APPLICATION NOTE

Small angle diffraction capabilities of the ARL EQUINOX 100 X-ray diffractometer using Ag-behenate

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Introduction

The measurement of reflections at low angles is crucial for various applications, which involve compounds with large-scale structural features. Such compounds are of interest in polymer research and production, catalyst development or some mining applications. A widely used reference compound for low angle diffraction experiments is Ag-behenate as it consists of well-aligned fatty acid chains, which form a large-scale network (c.f., Figure 1).

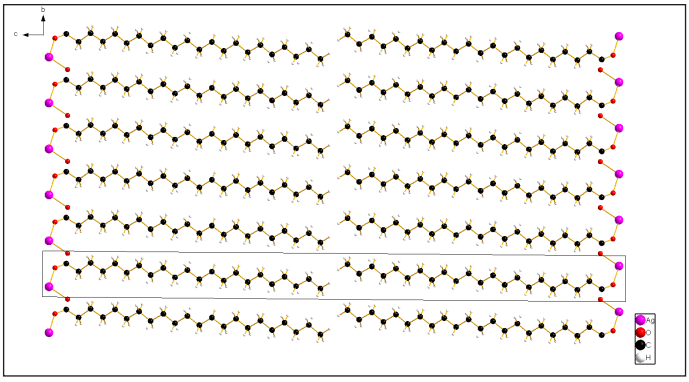

Figure 1: Projection of the crystalllographic structure of Ag-behenate along the *b*-axis.

Instrument

The Thermo Scientific™ ARL™ EQUINOX 100 employs a custom-designed Cu (50 W) or Co (15 W) micro-focus X-ray tube with mirror optics. The low power consumed by the unit allows it to be completely tranportable, not requiring an external water chiller. The same unit is capable of being transported between laboratories without the need for special infrastructure.

Figure 2: ARL EQUINOX 100 X-ray diffractometer

The ARL EQUINOX 100 provides very fast data collection times compared to other diffractometers due to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time and is therefore well suited for both reflection and transmission measurements (Figure 2).

Experimental

A sample of Ag-behenate was filled in a glass capillary (0.5 mm diameter; 10 min) and measured in transmission mode using Cu Ka radiation as well as in a reflection cup (3 mm depth; 5 min) with Co Ka radiation.

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Results

Using transmission mode it is possible to clearly resolve the [001] reflection of Ag-behenate at 1.51°2θ (d = 58.35 Å) up to the [009] reflection at 13.65°2θ (d = 6.48 Å) with an expected FWHM of ~0.25°2θ (c.f., Figure 3).

Results in reflection mode (1° incidence angle) are similar whereas the expected FWHM is ~0.35°2θ but the measurement time is reduced by a factor of two (c.f., Figure 4).

Conclusion

The ARL EQUINOX 100 is a suitable instrument to measure reflections at low angles down to ~1.5°2θ as shown using the example of Ag-behenate, which enables the analysis of polymers for phase determination.

Additionally it is possible to assign the structure of mesostructured compounds, which are often used as catalysts or substrates. The structure directly influences the catalytic activity of such compounds.

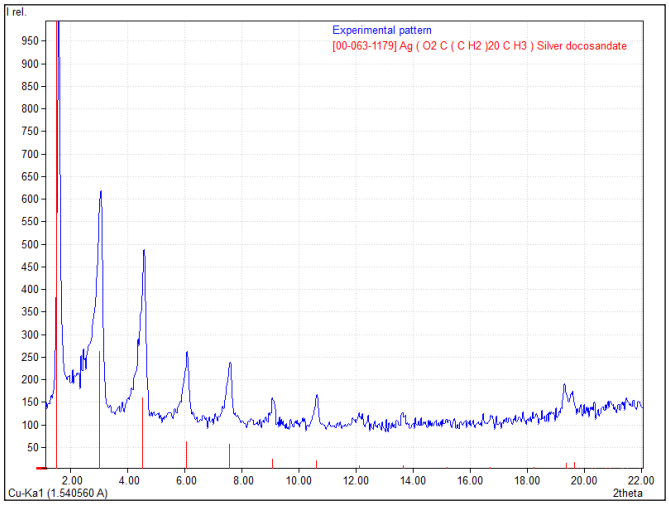

Figure 3: Measurement of Ag-behenate in transmission mode (0.5 mm capillary, 10 min measurement time).

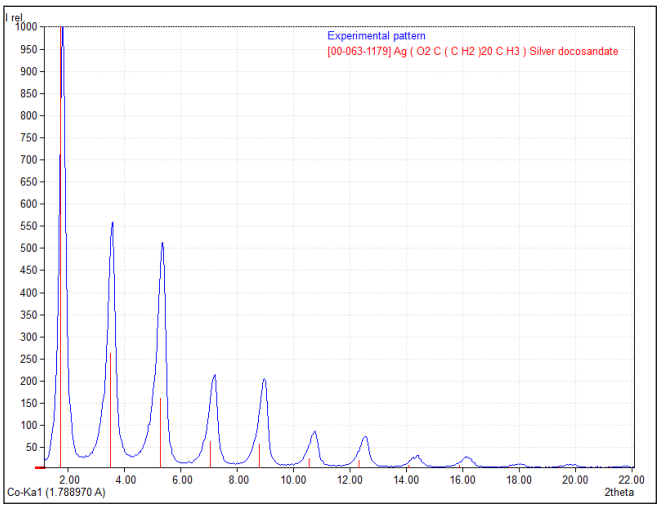

Figure 4: Measurement of Ag-behenate in reflection mode (3 mm cup, 5 min measurement time).

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