APPLICATION NOTE

XRD analysis in forensic drug investigation using benchtop ARL EQUINOX 100 X-ray Diffractometer

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Introduction

Due to emerging availability and consumption as well as development of drugs, their analysis plays a key role in modern forensic science. To trace the source of drugs it is important to determine their type, form and composition. XRD (X-ray diffraction) analyses are a crucial step in the analytical workflow carried out by forensic labs for the investigation of powder and bulk samples not only for drug related samples but additionally for various other samples like textiles or soils. XRD analyses are capable of confirming findings from complementary analytical techniques like mass spectrometry and providing additionally information about the crystalline structure which allows conclusions like phases quantities from even complex mixtures.

As a scientist in forensics analyzing drug related samples using XRD there are three main questions to answer:

1. Can I confirm the findings from related techniques, e.g., is the preliminary test result showing heroine valid?

2. What chemical form of drug is present, e.g., the base or hydrochloride hydrate form of heroine?

3. Can I (a) identify and (b) quantify additional phase as well as the main drug component? This is important for tracing and clustering drug samples.

This application note will answer all three questions using various test samples.

Figure 1: ARL EQUINOX 100 diffraction system

Instrument

The Thermo Scientific™ ARL™ EQUINOX Series represent a portfolio of XRD instruments from simple, easy to use bench-top systems for routine analysis to more advanced floor-standing, research grade systems.

The Thermo Scientific™ ARL™ EQUINOX 100 employs a custom-designed 50 W (Cu or Mo) or 15 W (Co) high-brilliance micro-focus tube with mirror optics which does not require an external water chiller. The unit can be transported between laboratories or into the field without requiring any special infrastructure.

The ARL EQUINOX 100 (c.f. Figure 1) provides very fast data collection compared to other diffractometers due to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time.

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Experimental

Powdered drug samples were provided by and evaluated with the assistance of the scientific police. The measurement was made in reflection using an ARL EQUINOX 100 with Cu Kα radiation.

Results

For many drug related compounds different chemical forms like salts, hydrochlorides and others as well as stereoisomers exist. As they could have different physical effects and for sample tracing it is important to identify them. Figure 2 unequivocally shows Heroine in its Hydrochloride Monohydrate form compared to the base form. This allows to compare with other samples and trace their origin. As there are no diluents detectable this sample was most likely not designated for end customers distribution.

The content of active ingredient in drugs varies as well as the type of diluents. Therefore, a crucial part in the drug analysis process is identifying the phases and their respective quantification. Figure 3 and 4 show two different types of Cocaine Hydrochloride samples. Both are mainly diluted with α-Lactose and contain varying amounts of other drug related compounds and Caffeine. Sample 1 exhibits a Cocaine content of ~35 wgt%, whereas Sample 2 has a content of >90 wgt%.

Conclusion

The ARL EQUINOX 100 XRD is capable of measuring drug related samples within 10 min measurement time. The combination of MDI JADE 2010 and ICDD pdf 4 Organics database allows qualitative and quantitative phase analysis (WPF Rietveld refinement) of the samples. The results clearly show type, form and composition of the samples and therefore enable tracing and identification of drugs.
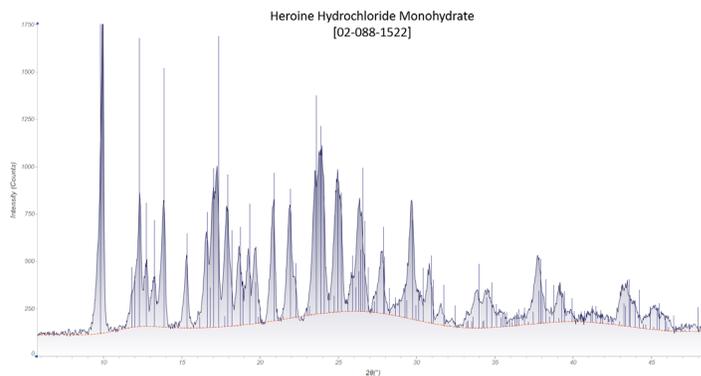

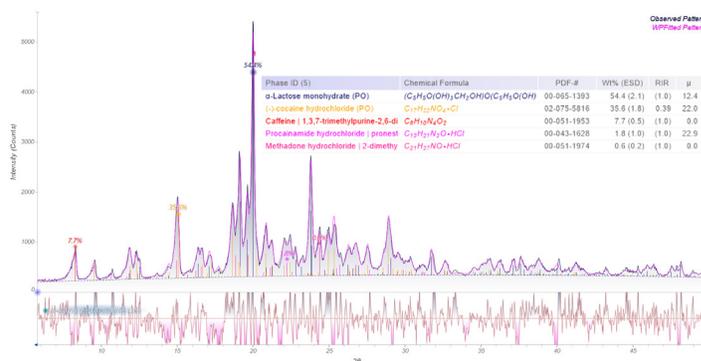Figure 2: Heroine Hydrochloride Monohydrate (10 min measurement time)
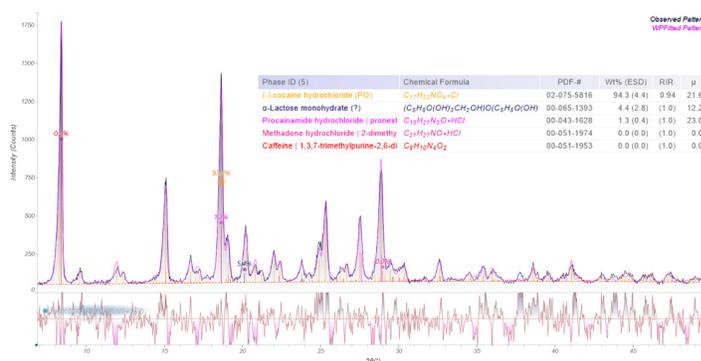

Figure 3: Cocaine blend Sample 1 (10 min measurement time)

Figure 4: Cocaine blend Sample 2 (10 min measurement time)

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