

# Dynamic structural studies of pharmaceutical products using the ARL EQUINOX Series X-ray Diffractometers

## Introduction

X-ray diffraction is the gold standard of techniques used in the structural and phase characterization of pharmaceutical products. XRD is one of the most commonly used techniques in the pipeline from drug discovery to preformulation and formulation, through to stability and quality control. XRD has applications in studying the solid dosage forms, crystallinity and bioavailability of APIs.

In particular, XRD is routinely applied in the study of polymorphs, salts and co-crystals of a particular API. In addition XRD is also used to follow the crystallization of the product, for the determination of the crystalline percentage of the final product, the stability of the formulations and their reactivity in a controlled environment.

Depending on the scope of applications and the extent of measurements needed, different types of XRD instruments and configurations are used. For example, a bench-top XRD instrument would be the most cost effective solution for initial polymorph and salt screening, in QA/QC laboratories, and in determining the percentage of crystallinity of a substance. A stand-alone high resolution XRD would be needed for more extensive research studies of the pharmaceutical product.

One of the most desirable features of the entire ARL EQUINOX XRD line is its unique curved position sensitive (CPS) detector, enabling acquisition over a full range of  $2\theta$  (from  $90$  to  $120^\circ$  according to the considered CPS model) in real time to study the structural and crystallographic changes of the API and related products. Very fast measurements can be performed on the same sample as a function of different parameters enabling the analyst to make quicker decisions and/or process more samples.



Figure 1: Real-time XRD detector



## Instrument

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, high performance, research grade systems for investigative laboratories.

Thermo Scientific™ ARL™ EQUINOX 100 and Thermo Scientific™ ARL™ EQUINOX 1000 are bench-top XRD models utilizing different X-ray power sources, offering different levels of convenience and performance. The ARL EQUINOX 100 employs a custom-designed 60 W micro X-Ray source with micro-focus X-Ray tube (Cu or Co) which does not require an external water chiller. The same unit can be transported between laboratories or into the field and does not require any special infrastructure. There are also options available on the ARL EQUINOX 100 to study the reactivity and stability using a controlled environment cell. Both transmission and reflection mode measurements can be done on the same bench-top instrument.

The ARL EQUINOX 1000 on the other hand uses a standard 3500 W X-Ray source requiring chilled water cooling to the tube. The ARL EQUINOX 1000 is the only bench-top XRD with high power and standard XRD tubes (Cu or Co) ensuring optimum performance both in terms of resolution and sensitivity.

Users can choose from either Ge monochromator (for high resolution) or HOPG monochromator (for high intensity) or both in a unique optional twin monochromator system (SIAM X), which allow the ARL EQUINOX 1000 to provide performance comparable to bigger and higher priced instruments. The ARL EQUINOX 100 and ARL EQUINOX 1000 provide very fast data collection times compared to other diffractometers due to their unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time.

Figure 2: Paracetamol 250 mg measured in reflection mode 40 kV / 1 mA, scan from 2° to 100°

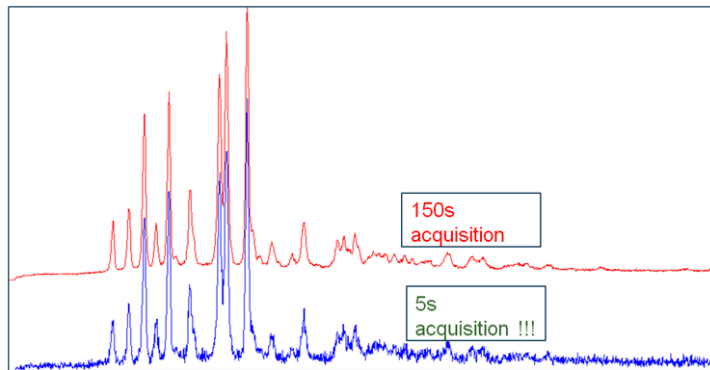
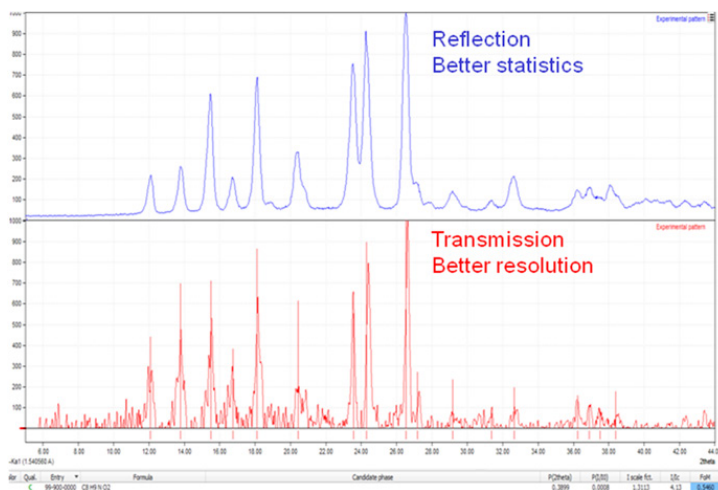


Figure 3: Paracetamol - comparison of analysis mode (reflection versus transmission)



### Software and Data Processing

The ARL EQUINOX XRD Series diffractometers come with a fully integrated data acquisition and processing software and can also interface with third party programs depending on the information to be obtained.

### Conclusion

The ARL EQUINOX 100 and ARL EQUINOX 1000 models are fast, convenient and high performance X-ray diffraction instrument perfectly suited for routine applications in

In addition to standard XRD measurements under ambient conditions, both ARL EQUINOX 100 and ARL EQUINOX 1000 can be fitted with optional sample stages (Figure 4) such as capillary stage, controlled atmosphere cell, transmission stage and sample spinners which provide lot more flexibility even with these bench-top XRDs. Some of these options are unique to ARL EQUINOX 100 and ARL EQUINOX 1000 bench-top models and are generally not available with other bench-top XRD instruments.

Figure 4: Standard sample holders

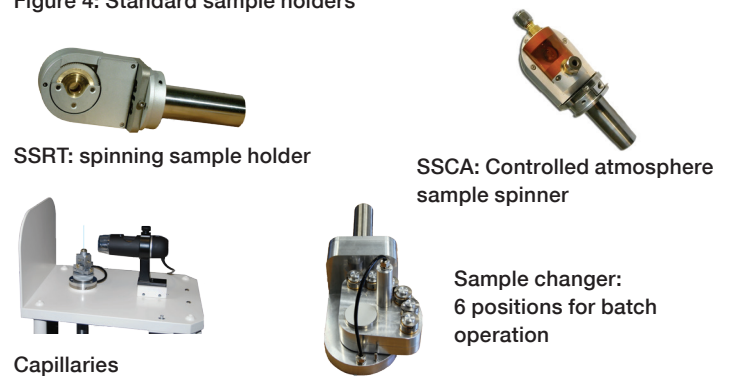
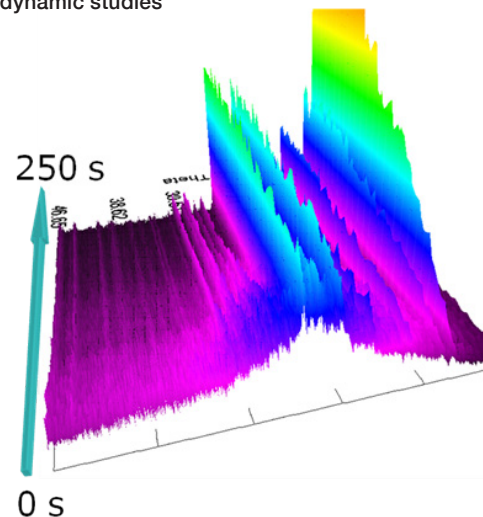


Figure 5: Crystallization of pharmaceutical product: real-time dynamic studies



pharmaceutical industry. Although these are bench-top designs, they can accommodate different sample stages to extend the measurements for different applications.

For more investigative research oriented applications, the ARL EQUINOX 3500 is preferable owing to various options and flexibility that a floor-standing platform can offer.

Thanks to the simultaneous acquisition of the full 2θ range, data can be generated and processed much faster than conventional XRD systems.

Find out more at [www.thermofisher.com/xrd](http://www.thermofisher.com/xrd)

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