APPLICATION NOTE

The Gandolfi Stage: a novel approach for the analysis of single crystals and small volume samples with ARL EQUINOX Series

Author: Gregory Schmidt
XRD Applications Scientist

Introduction

The Gandolfi Stage, developed for use in conventional powder diffractometers, enables the analysis of single crystal or small volume samples without the need of a dedicated single crystal diffractometer. The stage effectively turns the diffractometer into a three-circle system, allowing for the fast analysis of samples previously not suitable for conventional X-ray diffraction (XRD) systems. Combined with proper software applications, the Gandolfi Stage allows for a variety of analyses from phase identification to structure solution.

Instrumentation

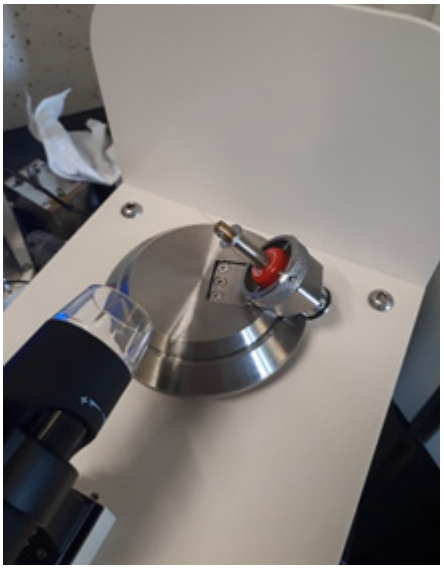
The Thermo Scientific™ ARL™ EQUINOX 3000/5000 Diffractometers utilize a unique tall vertical goniometer. Their features include a large choice of X-ray sources and higher resolution detectors. The large sample compartment accommodates a large array of sample sizes and sample handling devices such as large assemblies, auto samplers, and furnaces. Software capabilities allow expansive analysis capabilities including: crystal structure analysis, qualitative analysis, degree of crystallinity determination, phase identification and quantification, uniaxial stress, phase transition on various environments and thin film analysis.

Figure 1: ARL EQUINOX 3000 X-ray Diffractometer.

The ARL EQUINOX 3000/5000 Diffractometers provide faster data collection when compared to other diffractometers due to its unique curved position sensitive detector (CPS) that measures all diffraction peaks simultaneously and in real time. It is therefore well suited for both reflection and transmission measurements.

The Gandolfi Stage consists of two motors and a goniometer head with a standard brass pin, which is used along with glass fibers to mount the crystal(s) (Figure 2). Alignment is performed using the USB camera, connected to the controlling computer, and associated software on the stage holder. The stage is then transferred to the diffractometer and rotated during data collection to maximize the peaks and their respective intensities collected.

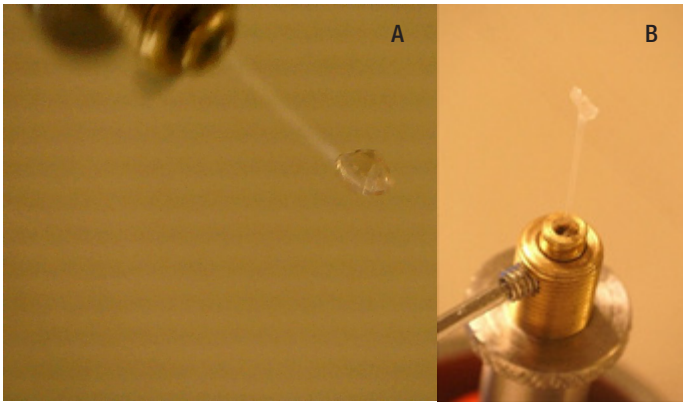
Figure 2: Gandolfi Stage on centering station.



Experimental

Examples of potential applications range from non-destructive phase determination of low volume samples all the way to the structure solution of a single crystal. In each case, the crystal can be mounted on a glass fiber (Figure 3 A & B), centered and placed in the instrument. Data was collected for five minutes and one hour, respectively, under Cu Kα radiation at 30 kV, 40 mA using a CPS 120 detector.

Figure 3: Gemstone (A) and a single crystal (B) mounted on glass fiber.



Results

Phase identification

Collected intensities were imported into MDI JADE 2010 for phase identification. After performing a search-match, the material was identified as the mineral elbaite (Figure 4). Elbaite is the most commonly used member of the tourmaline family in jewelry, ranging from dark purplish-red to colorless (Figure 5). The skewed background is due, at least in part, to the large crystal size.

Figure 4: Unknown gemstone sample showing elbaite fit.

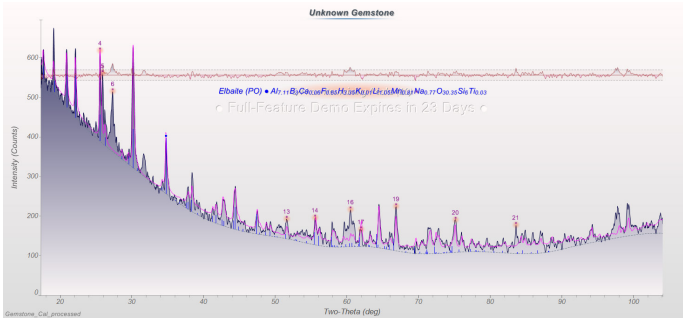


Figure 5: Polished elbaite from Mozambique.

thermoscientific

Single crystal

Intensities were extracted from a one-hour data set (Figure 6). Utilizing any of an array of software solutions (FOX, EXPO2014, GSAS-II, etc.) the ab initio structure can be solved and refined using Rietveld methods (Figure 7).

Figure 6: One-hour single crystal raw data.

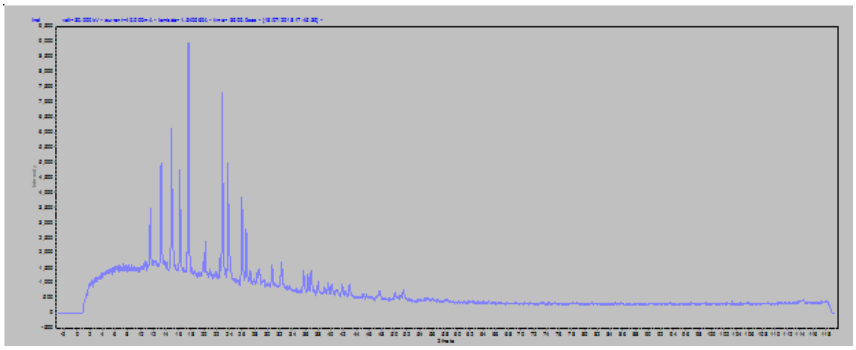
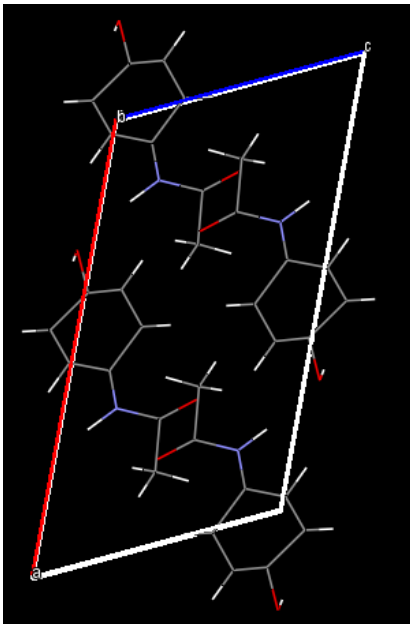


Figure 7: Final wireframe structure.



Conclusion

The Gandolfi Stage, developed for use in conventional powder diffractometers, enables the analysis of single crystal or small volume samples without the need of a dedicated single crystal diffractometer. Combined with the speed and resolution of the ARL EQUINOX 3000/5000 Diffractometers, it allows for the analysis of single crystals or small clusters from phase identification through *ab initio* structure solutions and expands the capability of a single system to include routine analyses of a variety of sample types and quantities.

Acknowledgements

The author would like to express his thanks to Dr. Henry Pilliere and Raphael Yerly for collecting the data on the provided samples used in this application note.

Find out more at thermofisher.com/xrd

ThermoFisher SCIENTIFIC