

## Evaluation of the D8 ADVANCE Diffractometer using the NIST LaB<sub>6</sub> Line Position and Profile Shape Standard (SRM 660)

### Abstract

Precise and accurate peak positions along with consistent peak widths are critical to any powder diffraction data. This study evaluates the peak positions and the full width at half maximum (FWHM) recorded by the D8 ADVANCE Diffractometer using the NIST SRM 660 Instrument Line Position and Profile Shape Standard. The D8 ADVANCE provides data that are within  $\pm 0.01^\circ$  of the certified line positions without any data correction, and this data also provides a lattice parameter that is within  $\pm 2\sigma$  of the certified lattice parameter. The FWHM of the peaks increases as a function of  $2\theta$ , as expected. The FWHM of the largest d-spacing peak is  $0.03^\circ$ .

### Introduction

The ability of a powder diffractometer to provide precise and accurate line positions is critical to any experiment. Incorrect line positions will adversely affect the results of applications such as phase identification, unit cell indexing, and stress analysis. The National Institute of Standards has developed a line position standard called SRM 660 that is specifically designed to determine how well an instrument can determine the line positions from a diffraction pattern.

Incorrect peak widths also affect powder diffraction experiments. Applications such as crystallite size and strain as well as structure determination experiments, i.e. Rietveld analysis, can be adversely affected due to inconsistent FWHM. SRM 660 was used in this study to evaluate the D8 ADVANCE Powder Diffractometer for line position and profile shape.

### Experimental Procedures

In order to compare the data with the NIST standard, some calculations must first be done. NIST supplies the certified lattice parameter for the SRM 660 in the Certificate of Analysis as well as the (hkl) values for all of the peaks used in the analysis<sup>1</sup>. Using the following formula the d-spacing of the peaks can be calculated:

$$d_{hkl}^{*2} = (h^2 + k^2 + l^2)a^{*2}$$

Where:

- $d_{hkl}^*$  =  $1/d_{hkl}$
- (hkl) = The Miller Indices of the reflection
- $a^*$  =  $1/a$
- a = The certified lattice parameter for SRM (660) (4.15695Å)<sup>1</sup>

This value can then be used to calculate the 2θ peak positions using Bragg's Law:

$$\lambda = 2d_{hkl}\sin\theta$$

Where:

- $\lambda$  = The wavelength of  $CuK_{\alpha 1}$  radiation
- d = The d spacing calculated from above
- $\theta$  = The angular position of the peaks

The diffraction pattern from the SRM 660 was collected using the instrument configuration in Table 1.

The diffracted peak positions were found by profile fitting the top 25% of the  $CuK_{\alpha 1}$  peak with a split Pearson VII function. The FWHM of the peaks were found by profile fitting the entire peak with a split Pearson VII function.<sup>2</sup>

<b>Table 1. System Configuration</b>	
D8 ADVANCE X-ray Powder Diffractometer	
Incident Beam Optics	0.5° Divergence Slit 2° Vertical soller slits
Sample Stage	Standard sample stage with 3 reference points for sample height reproducibility
Diffracted Beam Optics	0.5° Antiscatter Slit 2° Vertical soller slits
Receiving Slit	0.05mm
Monochromator	Graphite diffracted beam monochromator
Detector	Dynamic Scintillation Detector
Temperature	22°

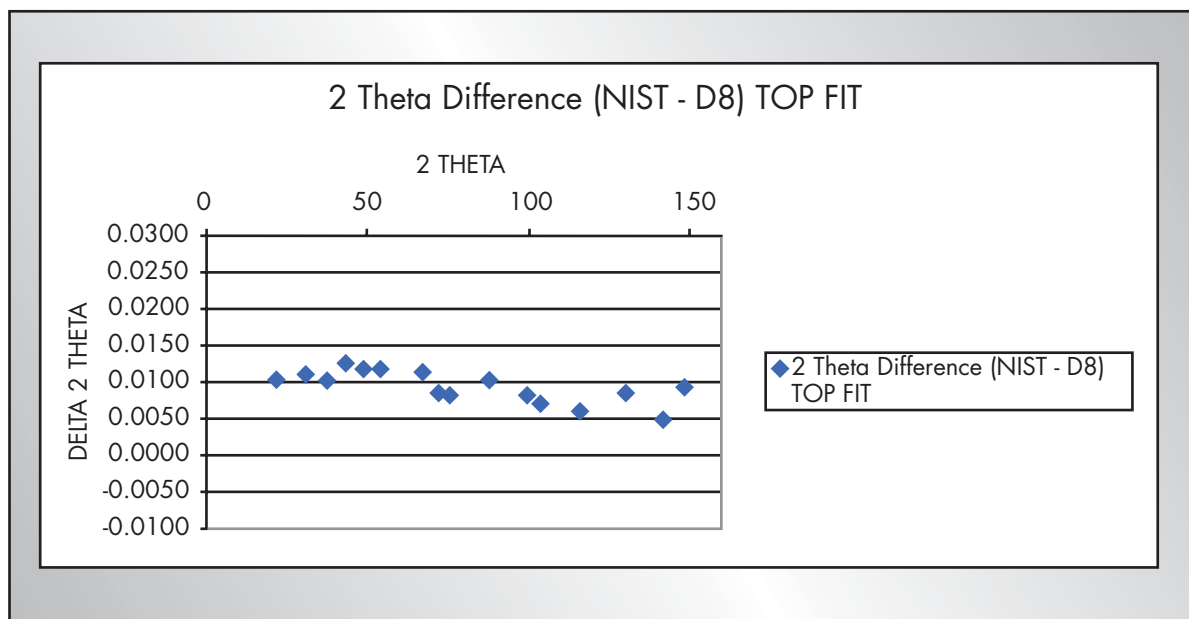


Figure 1. Delta 2θ versus 2θ for SRM 660 on the D8 ADVANCE

### Results

The line positions found by the D8 ADVANCE were subtracted from the certified positions. The difference was then plotted vs. the certified line positions. The results of this difference plot are shown in Figure 1.

The data shows a very small deviation from zero. The largest deviation is 0.0125°. This agrees very well with the data presented by Cheary et al.<sup>2</sup> The quality of this data is excellent considering that the data has not been corrected for any errors known to be associated with X-ray powder diffraction. Usually errors such as sample displacement, instrumental zero offset, sample transparency etc., need to be corrected prior to determining accurate lattice parameters. Without correcting the data, these peak positions were input into the Bruker AXS lattice parameter refinement software program.

This was done to determine how close the measured, uncorrected, peak positions would come to the NIST certified lattice parameter. Again no corrections or calibration curves were applied to the data. The certified value in the NIST Certificate of Analysis is  $4.15695\text{\AA} \pm 0.00006\text{\AA}$ . The calculated lattice parameter from the measured peak positions using the D8 ADVANCE is  $4.15710\text{\AA} \pm 0.0001\text{\AA}$ . This value is within  $2\sigma$  of the certified lattice parameter with no data correction applied.

The FWHM from the full fit data was plotted as a function of the certified peak positions to evaluate the peak width data. This plot is illustrated in Figure 2.

This curve, showing the proper data collection of the instrument, follows a second-degree polynomial function.<sup>3</sup>

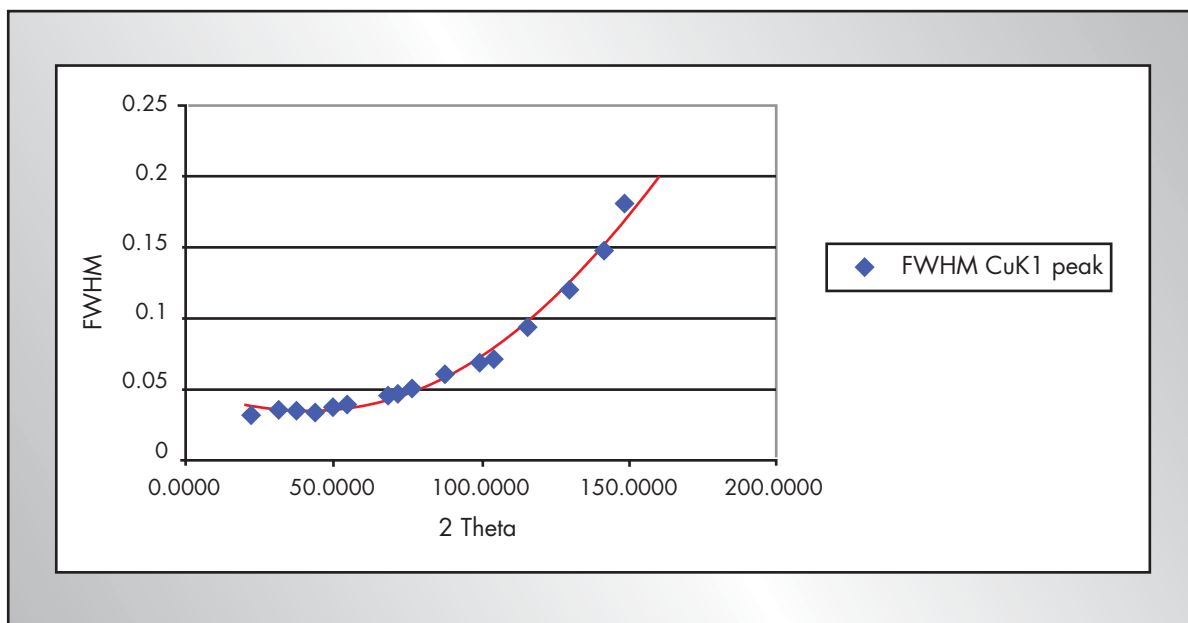


Figure 2. FWHM vs.  $2\theta$  for SRM 660 on the D8 ADVANCE

### Conclusions

The D8 ADVANCE X-ray Powder Diffractometer provides precise, uncorrected, line positions that can be used for all X-ray diffraction applications. The user can be assured that any major line shift in the X-ray pattern is not a function of the instrument. The FWHM of the peaks follow a second-degree polynomial throughout the angular range showing that the D8 ADVANCE is designed to collect high precision data, and that the instrument is functioning properly.

### References

- <sup>1</sup> Rasberry, S. D. (1989), Certificate of Analysis, SRM 660 "Instrument Line Position and Profile Shape Standard for X-ray Diffraction" NIST, Gaithersburg, MD 20899.
- <sup>2</sup> Cheary R.W., Cline J.P. and Anast M. (1997), *Advances in X-ray Analysis* **39**, 579-587.
- <sup>3</sup> Jenkins R., and Snyder R.L. (1996), "Introduction to X-ray Powder Diffractometry", pp. 284-285, John Wiley and Sons, Inc.: New York.

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