Characterization @ UBC

Round robin study: X-ray Diffraction of LaB_6 Standard Reference Material

Peak position and peak width analysis

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Characterization @ UBC
Research Excellence Cluster

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Summary and significance of the study

As a Characterization@UBC research excellence cluster activity, this inter-laboratory round-robin study benchmarked powder X-ray diffraction (XRD) performance using NIST Standard Reference Material 660a (LaB_6). Six diffractometer configurations across three facilities were compared and evaluated, within the Electron Microscopy Laboratory (EM Lab), the Electron Microscopy and X-ray Diffraction Facility (EMXDF), and the Department of Chemistry. For each instrument, peak positions relative to the certified reference values were compared to confirm instrument accuracy, and instrumental peak broadening was quantified using the full width at half maximum (FWHM) and the Caglioti function parameters were determined to compare the instrumental resolution functions.

During the first phase of the study, one diffractometer configuration (MTRL D6 with the original Universal stage, Uni-Old) exhibited a systematic angular offset of about 0.13°, despite the instrument having been commissioned only a couple of years ago. The phase one data indicated that this specific instrument had a hardware issue, which was unknown to the instrument users. The findings were communicated to the instrument manufacturer, and this resulted in replacement of the Universal stage (under warranty). The LaB_6 measurements were subsequently repeated using the replacement stage (Uni-New), demonstrating substantial improvement in both peak position accuracy and peak-width behavior, and now the instrument is in agreement with the performance of the other participating facilities (and the documentation associated with the standard).

Beyond comparing instruments, the study demonstrates how cross-facility benchmarking can serve as an effective quality-assurance process by detecting hidden faults, verifying corrective maintenance, and generating reference data for future performance checks. We encourage XRD facilities across UBC to use comparable standard reference material-based measurements routinely, particularly after major service, optics changes, or suspected instrument drift. This round-robin exercise also provided an opportunity to build community, share know-how, and highlight the benefits of working together across the UBC research environment towards an improved understanding of characterization technologies

This work advances Characterization@UBC's goals of connecting complementary facilities, sharing technical expertise, and strengthening shared research infrastructure. The resulting common benchmark and repeatable quality assurance workflow support more consistent user service and greater confidence in XRD data produced across UBC.

1. Introduction

The accuracy and precision of XRD measurements are critically dependent on the alignment and condition of the diffractometer, the choice of optics, and the measurement parameters employed. Uncertainty in these measurements can affect all forms of associated XRD analysis, including approaches such as phase classification, lattice parameter determination, and Rietveld analysis. Inter-laboratory comparisons using certified standard reference materials (SRMs) are an established means of verifying instrument performance and ensuring data quality across different facilities and instrument types. We used lanthanum hexaboride (LaB_6) that is NIST-certified Standard Reference Material 660a, which is widely used for calibrating both peak positions and instrumental line profiles in laboratory powder diffractometers.

The present study was coordinated across three facilities under the umbrella of Characterization@UBC, each operating one or more Bruker diffractometers with different detector/source/optics configurations. The comparison allows assessment of the relative performance of each instrument, identification of systematic measurement errors, and verification of instrument health using a common certified reference material. An additional goal was to establish benchmark datasets that could support long-term quality assurance across Characterization@UBC facilities and other XRD instruments on campus. As demonstrated in this work, such benchmarking can reveal subtle hardware issues that may remain undetected during routine operation, while also supporting interoperability, confidence in results, and ease of access to the instrumentation.

2. Objectives

The following were the key objectives of this round-robin study:

1. To generate benchmark data and support a collective effort to share expertise amongst the facilities and cluster members.
2. To identify any systematic errors/anomalies in the instrument configurations and hardware.
3. To assess the peak position accuracy of the instruments against the LaB_6 database values.
4. To quantify instrumental peak broadening (via FWHM) at multiple Bragg reflections across a reasonable 2θ angular range.
5. To fit the Caglioti function[1] to the measured FWHM data and report the U, V, W parameters.
6. To support Characterization@UBC's efforts to build community, reduce barriers to access instrumentation, and improve our understanding of characterization approaches at UBC.

[1] The Caglioti function fits instrument broadening as a function of diffracting angle, and for example this assists in Rietveld refinement analysis. For more information on the function, see Caglioti et al. (1958) [1]

3. Sample

The sample used throughout this study was NIST Standard Reference Material 660a – Line Position and Line Profile Standard. This is a high-purity lanthanum hexaboride (LaB_6) powder that is widely used in calibration and performance verification of X-ray powder diffractometers. The XRD sample from this powder was prepared in the form of a smear on a low-background quartz plate, cased in a standard Bruker sample puck and stored under ambient lab conditions. The same SRM sample puck was circulated to all participating facilities to reduce costs and eliminate any contribution from sample-to-sample variability.

Figure 1: SRM used for the study (left) and prepared sample (right)

4. Participating facilities and instruments

Six diffractometer configurations across three facilities participated in this study. The diffractometers are shown in Figure 2.

Phase I

- MTRL EMLAB - D6 Phaser (Universal stage - old)
- MTRL EMLAB - D6 Phaser (Fixed stage)
- EOAS EMXDF - D8 Advance
- EOAS EMXDF - D8 Endeavor
- Chemistry - D8 Advance

Phase II – after stage replacement

- MTRL EMLAB - D6 Phaser (Universal stage - new)

Figure 2: X-ray diffractometers

Note: The "Fixed stage" in the D6 Phaser is designed for a single sample mounted in a 51 mm sample ring, with the sample height defined by calibrated height plates and no sample-spinning capability. It can be used in the standard horizontal Bragg–Brentano reflection geometry or rotated by 90° for foil transmission measurements. In contrast, the Universal stage adds independent omega (ω) rotation and Z translation, allowing control of the incident angle and sample height. These additional degrees of freedom enable more advanced measurements such as grazing-incidence diffraction (GID), X-ray reflectivity (XRR), stress, and texture analysis, while samples are mounted using interchangeable Z-stage attachments

5. Methodology

All participating facilities measured the same prepared NIST SRM 660a LaB_6 specimen using their routine powder XRD configurations. Although the instruments differed in X-ray source, optics, detector, stage, and acquisition settings, a common analysis workflow was used to compare peak positions against reference values and quantify instrumental peak broadening. Co-source measurements were converted to Cu-Kα_1-equivalent 2θ-positions before comparison, and FWHM values were fitted using the Caglioti relation to characterize each configuration's resolution function. Key acquisition parameters are summarized in Table 1.

Table 1: Scan parameters for all participating instruments

Parameters	**MTRL D6 (Uni-Old)**	**MTRL D6 (Uni-New)**	**MTRL D6 (fixed)**	**EMXDF Advance**	**EMXDF Endeavor**	**CHEM D8 Advance**
Source (λ, Å)	Cu-Kα_1 (1.5406)	Cu-Kα_1 (1.5406)	Cu-Kα_1 (1.5406)	Co-Kα_1 (1.78897)	Co-Kα_1 (1.78897)	Cu-Kα_1 (1.5406)
Voltage & Current	40 kV, 15 mA	40 kV, 15 mA	40 kV, 15 mA	35 kV, 40 mA	35 kV, 40 mA	40 kV, 40 mA
2θ Range (°)	5 to 120	10 to 105	10 to 100	4 to 120	4 to 125	5 to 110
Step size (°)	0.01	0.0085	0.01	0.01	0.01	0.01
Primary Soller (°)	n.a.	n.a.	n.a.	4.1	2.5	4.0
Secondary Soller (°)	2.5	2.5	n.a.	4	2.5	4.0
Detector opening	4.94°	4.94°	4.94°	3.188°	4.107°	12.57 mm
Stage rotation	Static	Static	Static	Spinning	Spinning	Spinning
Detector type	LYNXEYE XE-T	LYNXEYE XE-T	LYNXEYE XE-T	LYNXEYE	LYNXEYE	LYNXEYE

5.1. Scan parameters

XRD analysis focusses on the relationship between the incident radiation (and it's wavelength, λ); the interplanar spacing of the $\{hkl\}$ diffracting plane d_{hkl}; and the Bragg angle; θ, for diffraction (see Figure 3); and n is the diffraction order (usually $n = 1$), via the Bragg equation:

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

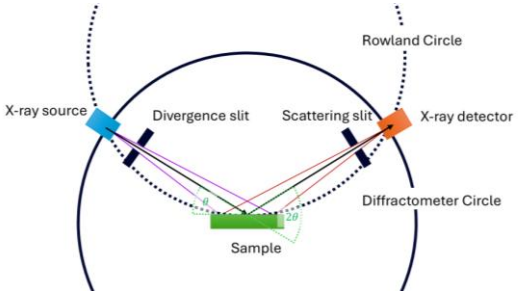

Figure 3: XRD schematic

5.2. Wavelength conversion

Notably, the MTRL and Chemistry instruments are configured to use Cu-Kα_1 radiation (λ = 1.5406 Å), while the EMXDF instruments used Co-Kα_1 radiation (λ = 1.78897 Å). Different radiation sources are selected for instruments depending on the target application area, as the incident radiation energy can affect fluoresces, absorption, penetration and usable angular range.

As different instruments used Cu or Co as radiation source, for this report all Co-source 2θ positions were converted to equivalent Cu-Kα_1 values prior to comparison using the following conversion:

$$2\theta_{\mathrm{Cu}} = \frac{360}{\pi}\left(\arcsin\left(\frac{\lambda_{\mathrm{Cu\ K\alpha1}}}{\lambda_{\mathrm{Co\ K\alpha1}}}\right)\sin\left(\frac{2\pi\theta_{\mathrm{Co}}}{360}\right)\right)$$

5.3. Peak position and FWHM determination

Peak positions and full widths at half maximum (FWHM) were determined for eight LaB_6 reflections: {100}, {110}, {111}, {200}, {210}, {211}, {300}, and {310}. The 2θ vs. intensity dataset was loaded from instrument output files in Excel. Each reflection was isolated within a defined angular window (e.g., 21°–22° for the {100} reflection). The maximum intensity and its angular position were identified as I_{max} and the peak position p, as shown in Figure 4. The FWHM was calculated in MATLAB.

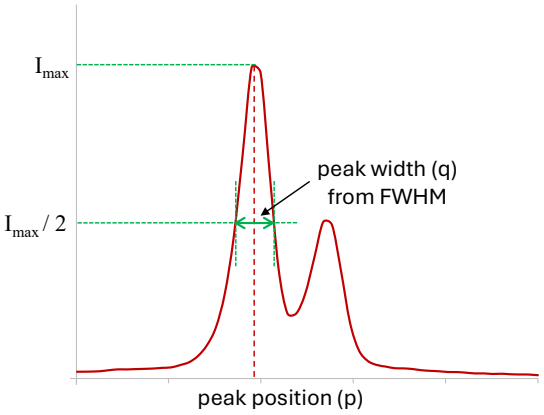

Figure 4: Peak analysis method

For pristine crystalline materials, experimental broadening contributions that influence the peak width are related to the so-called 'instrument broadening' terms.

Characterizing instrumental broadening is necessary to distinguish peak-width contributions arising from the diffractometer from those intrinsic to a sample, such as finite crystallite size, microstrain, and defects. An instrument-specific, angle-dependent resolution function enables more reliable line-profile and Rietveld analyses, while also providing a sensitive benchmark for monitoring alignment, optics, and overall instrument performance over time. Note that the instrument broadening terms can be influenced by physical instrument configuration, but also other parameters such as the slit opening configuration.

5.4. Caglioti function fitting

The angular dependence of instrumental peak broadening can be described using the Caglioti relation, which models FWHM as a second-order polynomial in $\tan\theta$:

$$\beta^2 = U\tan^2\theta + V\tan\theta + W$$

where β is the FWHM expressed in radians, θ = ½ x (2θ) = Bragg angle, and U, V, and W are instrument-specific fitting parameters determined by regression. All FWHM values were converted from degrees to radians prior to fitting.

6. Results

6.1. Diffraction data – overview

Normalized diffraction patterns (counts per second) acquired on all six instrument configurations are shown in Figure 5 in 2θ range of 15° to 90°.

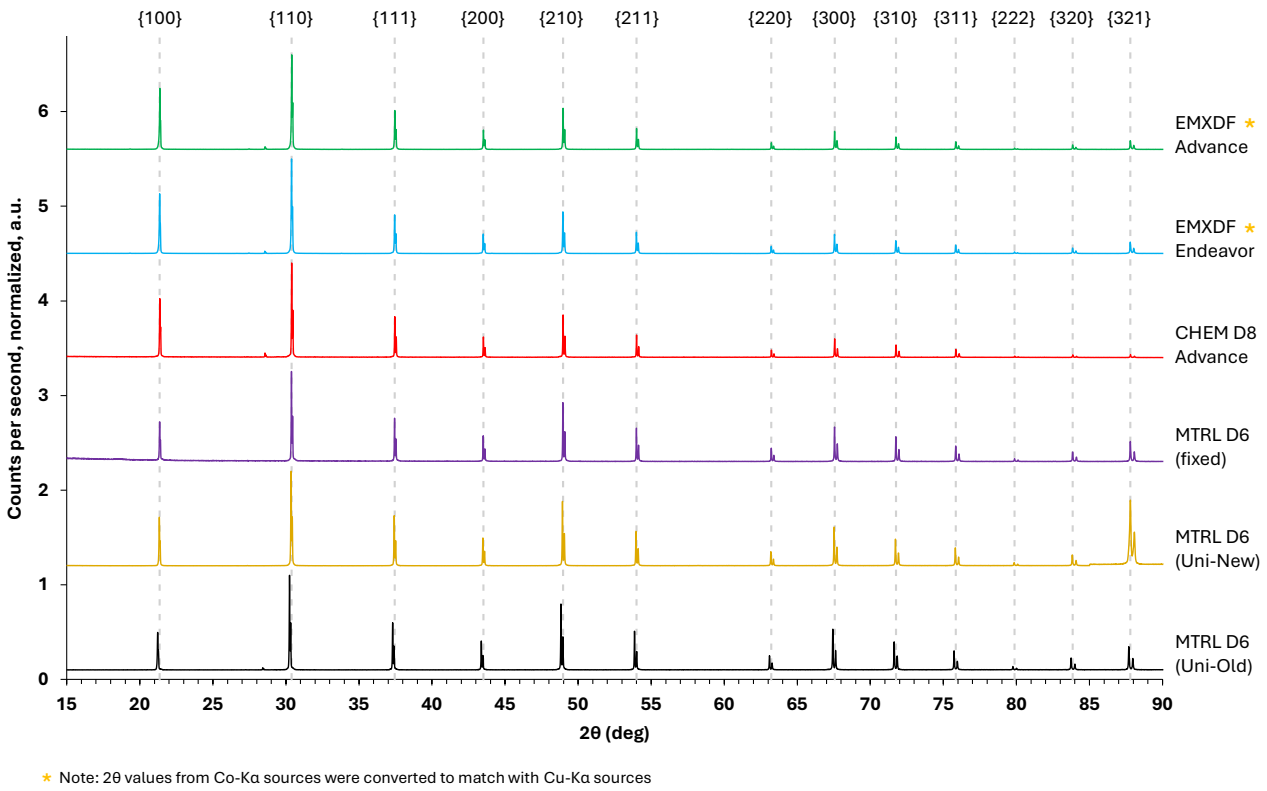

Figure 5: Normalized XRD patterns for all instruments referenced to the LaB_6 reference peak positions in dashed line (PDF 00-034-0427). Reflections from {100} to {321} are indicated. Note that the 2θ scan range presented here is limited to 90°.

6.2. Peak positions

Table 2 presents the measured 2θ peak positions (in degrees) for the eight LaB_6 reflections from each instrument configuration, all referenced to Cu-Kα_1 radiation. The PDF reference value is provided as a benchmark, also included in Appendix A: Figure A1. Figure 6 provides a zoomed-in view on the isolated peaks for better visual comparison.

Table 2: Measured peak positions (2θ, degrees, Cu-Kα$_1$ equivalent) for eight LaB_6 reflections

Reflection (hkl)	LaB_6 reference	MTRL D6 (Uni-Old)	MTRL D6 (Uni-New)	MTRL D6 (fixed)	EMXDF Advance	EMXDF Endeavor	CHEM D8 Advance
100	21.354	21.220	21.337	21.351	21.373	21.358	21.366
110	30.387	30.246	30.352	30.378	30.398	30.385	30.390
111	37.445	37.307	37.411	37.439	37.452	37.445	37.451
200	43.517	43.370	43.484	43.501	43.522	43.502	43.515
210	48.969	48.825	48.931	48.957	48.967	48.959	48.971
211	53.995	53.858	53.957	53.990	54.000	53.986	54.000
300	67.564	67.435	67.518	67.546	67.558	67.548	67.561
310	71.757	71.634	71.721	71.745	71.750	71.749	71.759

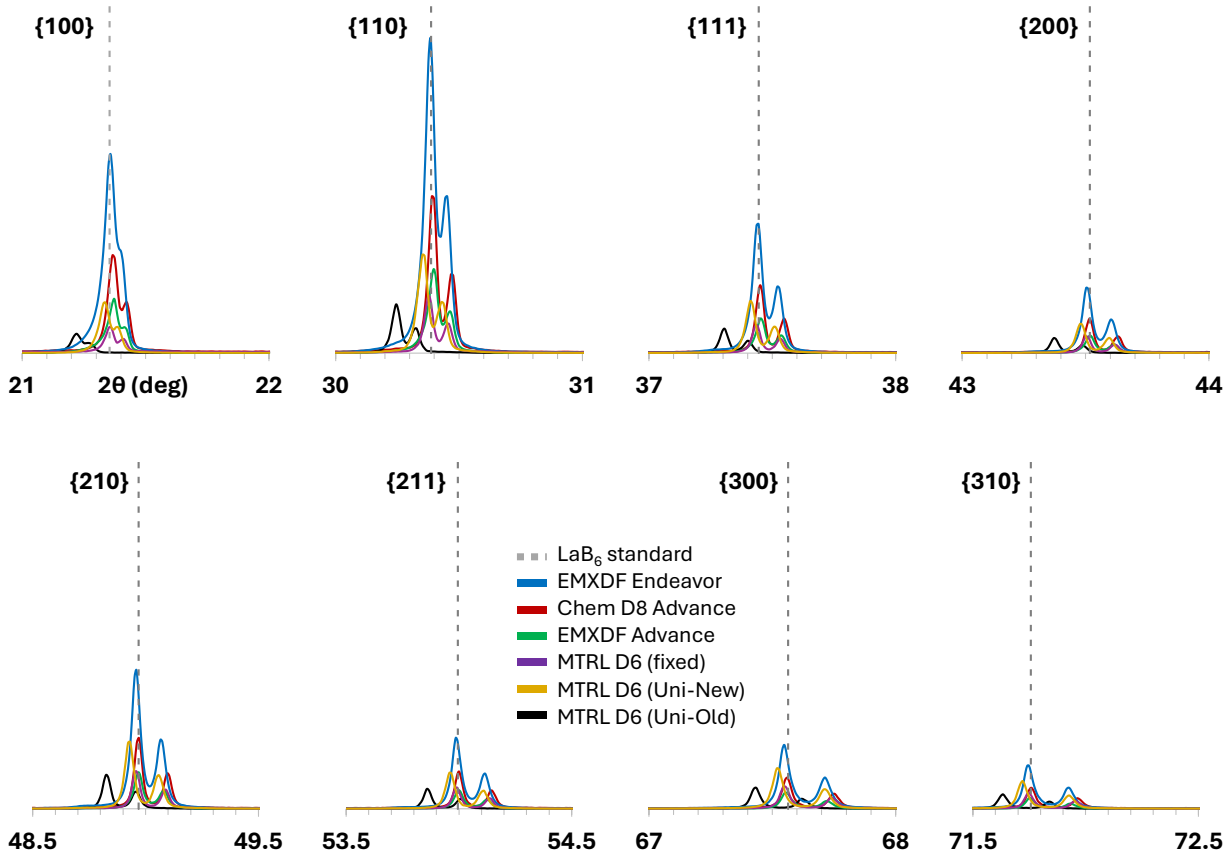

Figure 6: Peak to peak comparison for eight different reflections

Excluding the MTRL D6 (Universal-Old) configuration, all participating instruments demonstrate excellent agreement with the LaB_6 standard peak positions, with average deviations ranging from −0.035° to +0.004° (Table 3). In contrast, the original Universal stage

in MTRL D6 (Uni-Old) showed a consistent negative offset of approximately −0.137° across all reflections, e.g. as shown for {110} in Figure 7. This systematic lag is constant in magnitude across the 2θ range, which upon further investigation was found to be associated with stage-related malfunction.

Based on these findings, the Universal stage assembly was replaced by the manufacturer and repeat measurements acquired using the replacement Universal stage (Uni-New) show a marked reduction in the systematic offset, improving the average deviation from −0.137° (Old) to −0.035° (New).

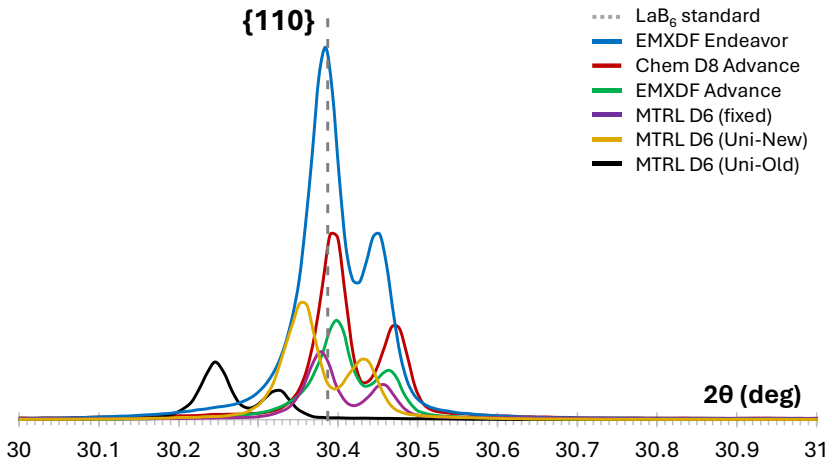

Figure 7: Peak to peak comparison for {110} reflection

Table 3: Measured difference in peak positions with respect to reference values (degrees)

Reflection (hkl)	MTRL D6 (Uni-Old)	MTRL D6 (Uni-New)	MTRL D6 (fixed)	EMXDF Advance	EMXDF Endeavor	CHEM D8 Advance
100	-0.134	-0.017	-0.003	0.019	0.004	0.012
110	-0.141	-0.035	-0.009	0.011	-0.002	0.003
111	-0.138	-0.034	-0.006	0.007	0	0.006
200	-0.147	-0.033	-0.016	0.005	-0.015	-0.002
210	-0.144	-0.038	-0.012	-0.002	-0.01	0.002
211	-0.137	-0.038	-0.005	0.005	-0.009	0.005
300	-0.129	-0.046	-0.018	-0.006	-0.016	-0.003
310	-0.123	-0.036	-0.012	-0.007	-0.008	0.002
Average difference	**-0.137**	-0.035	-0.010	0.004	-0.007	0.003
Standard deviation	0.007	0.008	0.005	0.008	0.007	0.004

6.3. Peak widths (from FWHM)

Table 4 summarize the FWHM values measured for each reflection and instrument. The FWHM values represent the combined instrumental broadening at each Bragg angle.

Table 4: Measured FWHM (degrees) for eight LaB_6 reflections across all instruments.

Reflection (hkl)	MTRL D6 (Uni-Old)	MTRL D6 (Uni-New)	MTRL D6 (fixed)	EMXDF Advance	EMXDF Endeavor	CHEM D8 Advance
100	0.081	0.055	0.048	0.053	0.074	0.051
110	0.043	0.050	0.041	0.049	0.050	0.042
111	0.042	0.046	0.041	0.046	0.049	0.041
200	0.042	0.047	0.042	0.046	0.047	0.041
210	0.044	0.048	0.043	0.045	0.047	0.042
211	0.043	0.050	0.043	0.048	0.049	0.043
300	0.048	0.051	0.048	0.052	0.050	0.047
310	0.051	0.052	0.049	0.051	0.054	0.049

At low 2θ angles for the {100} reflection at ~21°, the MTRL D6 (Uni-Old) and EMXDF Endeavor showed elevated FWHM (0.081° and 0.074° respectively) compared to all other instruments (~0.048–0.054°). This relates to the incompletely resolved first ($K\alpha_1$) and second ($K\alpha_2$) peak from {100} reflection, as shown in Figure A2 in Appendix-A.

6.4. Caglioti function parameters

For peak broadening analysis, the Caglioti function ($\beta^2 = U\tan^2\theta + V\tan\theta + W$) was fitted for the eight measured FWHM data points. A tabulation of each instrument's fitting parameters is provided in Table 5, and the fitted curves are shown in Figure 8.

Table 5: Calculated Caglioti fitting parameters (x 10^{-6})

Instrument	U	V	W
EMXDF Advance	2.70	-2.45	1.20
EMXDF Endeavor	5.35	-5.33	1.96
CHEM D8 Advance	3.29	-2.96	1.17
MTRL D6 (fixed)	2.41	-2.00	0.94
MTRL D6 (Uni-New)	2.54	-2.36	1.23

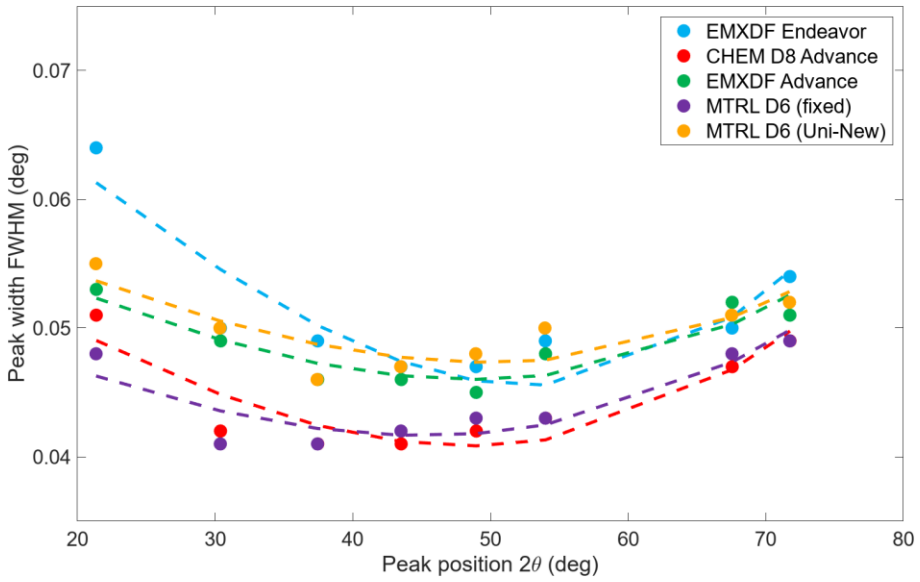

Figure 2: FWHM (degrees) vs. peak position 2θ (degrees) for all instruments, with dashed lines representing the fitted Caglioti curves for each configuration.

The MTRL D6 (fixed stage) returns the smallest U and W values, indicating the narrowest overall instrumental broadening function among the instruments tested. The EMXDF Endeavor have large widths at low angles, strongly influencing its fitting curves (see appendix-A, Figure A2 and A5). The majority of instruments' configurations in this comparison demonstrate excellent peak position accuracy, with offsets below ±0.01° from the certified standard values. Following replacement of the Universal stage, the Caglioti parameters obtained from the MTRL D6 (Uni-New) measurements are comparable to those of the others.

7. Conclusions

The following are the conclusions from the inter-lab comparison study:

7.1. Four of the five instrument configurations tested in Phase-I demonstrated peak position accuracy within ±0.01° of the LaB_6 reference values. This level of agreement is consistent with well-aligned and maintained diffractometers operating under routine laboratory conditions [2, 3].

7.2. The MTRL D6 Universal-old stage configuration exhibited a systematic angular lag of ~0.13° across all measured reflections, attributable to a mechanical malfunction in the stage drive. This work motivated the MTRL EM Lab to seek assistance from the supplier, and a replacement stage was obtained. Repeat measurements with the new stage confirmed that the systematic peak position errors observed in the original measurements had been successfully resolved.

7.3. Beyond benchmarking instrument performance, this study demonstrates the value of collaborative inter-laboratory comparisons. Periodic measurements using certified reference materials can identify hidden instrument issues and improve confidence in diffraction data generated across multiple facilities.

Additionally, the report provides tabular data and analysis of the instrumentation available within the cluster. For users of these instruments, we hope that this data provides valuable information and improves understanding of the capabilities. For facility operators, this data provides a timely snapshot of the equipment across campus and highlights areas where work could be focused (e.g. ensuring that the instruments maintain these calibrations).

The resulting dataset provides a practical basis for ongoing quality assurance across UBC XRD facilities. Periodic measurement of a common certified reference material—particularly after major service, changes to optics or detector configuration, or suspected drift—will enable facilities to monitor peak-position accuracy and instrumental resolution, identify emerging issues early, and maintain confidence in user data.

Acknowledgements

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Author contributions

M. Haroon Qaiser – data analysis, report writing, experiments (MTRL D6), coordination
Cole Maws – sample preparation, XRD data collection on EMXDF Advance & Endeavor.
Heli Eunike – XRD data collection on MTRL D6.
Brian Patrick – XRD data collection on CHEM D8.
Joey Yeap – supervision, report review and editing.
Ben Britton – conceptualization, supervision, report review and editing.

References

[1] Caglioti, G., Paoletti, A., & Ricci, F. P. (1958). Choice of collimators for a crystal spectrometer for neutron diffraction. Nuclear Instruments, 3(4), 223–228. doi.org/10.1016/0369-643X(58)90029-X

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[3] Beckers, D., Kharchencko, A., Gateshki, M., Götz, D., & Fransen, M. (2013). Critical parameters for instrument alignment and performance validation of XRD instrumentation [slides]. www.nist.gov/system/files/documents/mml/2-5_MFransen_APD_IV.pdf

Further information & facility access:

All three facilities that participate in this study provide user access for members of the UBC community, and more information can be found here:

EOAS EMXDF - eoas.ubc.ca/research/facilities/epma-xrd
Chemistry XRD - chem.ubc.ca/x-ray-crystallography
Materials EM Lab - emlab.mtrl.ubc.ca/

Industrial partners can seek access either directly via each individual facilities, or can find out more about access via the UBC imaging labs:

Imaging Labs: imaginglabs.ubc.ca/

Appendix-A

Pattern: PDF 00-034-0427 Radiation: 1.54060 Quality: Star (*)

Formula	LaB6
Name	Boron Lanthanum
Name (mineral)	
Name (common)	
Status	Alternate
Ambient	Yes

Lattice:	Cubic	**Mol. weight =**	203.77
S.G.:	Pm-3m (221)	**Volume [CD] =**	71.83
		Dx =	
		Dm =	

a =	4.15690		
a/b =	1.00000	**Z =**	1.00
c/b =	1.00000		

Additional Patterns: See PDF 00-006-0401 and 00-059-0332
Color: Violet-black
Sample Source or Locality: The sample was obtained from Koch-Light Laboratories, Colnbrook Bucks, England, UK, and donated by Gobel, H., Munich, Germany
Structures: The structure was determined by von Stackelburg and Neumann (1932)
Unit Cell Data Source: Powder Diffraction

Primary Reference
Publication: Natl. Bur. Stand. (U. S.) Monogr. 25
Detail: volume 20, page 62 (1984)
Publication: Z. Phys. Chem. (Leipzig)
Detail: volume B19, page 314 (1932)
Authors: von Stackelburg, M., Neumann.

Radiation:	CuKα1	**Filter:**	M
Wavelength:	1.54060	**d-spacing:**	
SS/FOM:	F(24)= 179.2 (0.0056, 24)		

d	2θ	I fix	h	k	l	d	2θ	I fix	h	k	l
4.15766	21.354	55	1	0	0	1.69687	53.995		1	-2	-1
4.15766	21.354		0	1	0	1.69687	53.995		1	1	-2
4.15766	21.354		0	0	1	1.69687	53.995		1	-1	-2
2.93918	30.387	100	1	1	0	1.46970	63.218	8	2	2	0
2.93918	30.387		1	-1	0	1.46970	63.218		2	-2	0
2.93918	30.387		1	0	1	1.46970	63.218		2	0	2
2.93918	30.387		0	1	1	1.46970	63.218		0	2	2
2.93918	30.387		0	1	-1	1.46970	63.218		0	2	-2
2.93918	30.387		1	0	-1	1.46970	63.218		2	0	-2
2.39980	37.445	41	1	1	1	1.38534	67.564	23	3	0	0
2.39980	37.445		1	-1	1	1.38534	67.564		0	3	0
2.39980	37.445		1	1	-1	1.38534	67.564		2	2	1
2.39980	37.445		1	-1	-1	1.38534	67.564		2	-2	1
2.07798	43.517	22	2	0	0	1.38534	67.564		2	1	2
2.07798	43.517		0	2	0	1.38534	67.564		1	2	2
2.07798	43.517		0	0	2	1.38534	67.564		2	-1	2
1.85862	48.969	46	2	1	0	1.38534	67.564		1	-2	2
1.85862	48.969		1	2	0	1.38534	67.564		0	0	3
1.85862	48.969		2	-1	0	1.38534	67.564		2	2	-1
1.85862	48.969		1	-2	0	1.38534	67.564		2	-2	-1
1.85862	48.969		2	0	1	1.38534	67.564		2	1	-2
1.85862	48.969		0	2	1	1.38534	67.564		1	2	-2
1.85862	48.969		0	2	-1	1.38534	67.564		2	-1	-2
1.85862	48.969		1	0	2	1.38534	67.564		1	-2	-2
1.85862	48.969		0	1	2	1.31435	71.757	16	3	1	0
1.85862	48.969		0	1	-2	1.31435	71.757		1	3	0
1.85862	48.969		2	0	-1	1.31435	71.757		3	-1	0
1.85862	48.969		1	0	-2	1.31435	71.757		1	-3	0
1.69687	53.995	24	2	1	1	1.31435	71.757		3	0	1
1.69687	53.995		1	2	1	1.31435	71.757		0	3	1
1.69687	53.995		2	-1	1	1.31435	71.757		0	3	-1
1.69687	53.995		1	-2	1	1.31435	71.757		1	0	3
1.69687	53.995		1	1	2	1.31435	71.757		0	1	3
1.69687	53.995		1	-1	2	1.31435	71.757		0	1	-3
1.69687	53.995		2	1	-1	1.31435	71.757		3	0	-1
1.69687	53.995		1	2	-1	1.31435	71.757		1	0	-3
1.69687	53.995		2	-1	-1	1.25329	75.849	10	3	1	1

Figure A1: Reference card for PDF 00-034-0427

The FWHM for each peak is presented in the plots below.

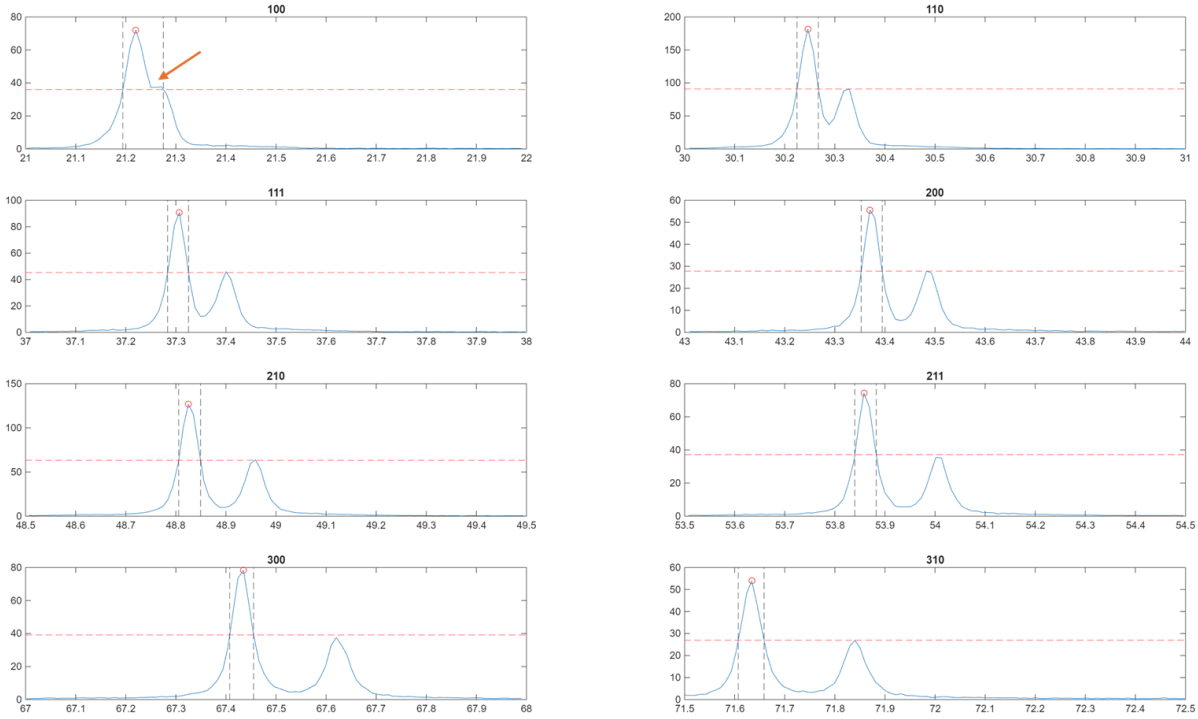

Figure A2: MTRL D6 (Universal stage - old)

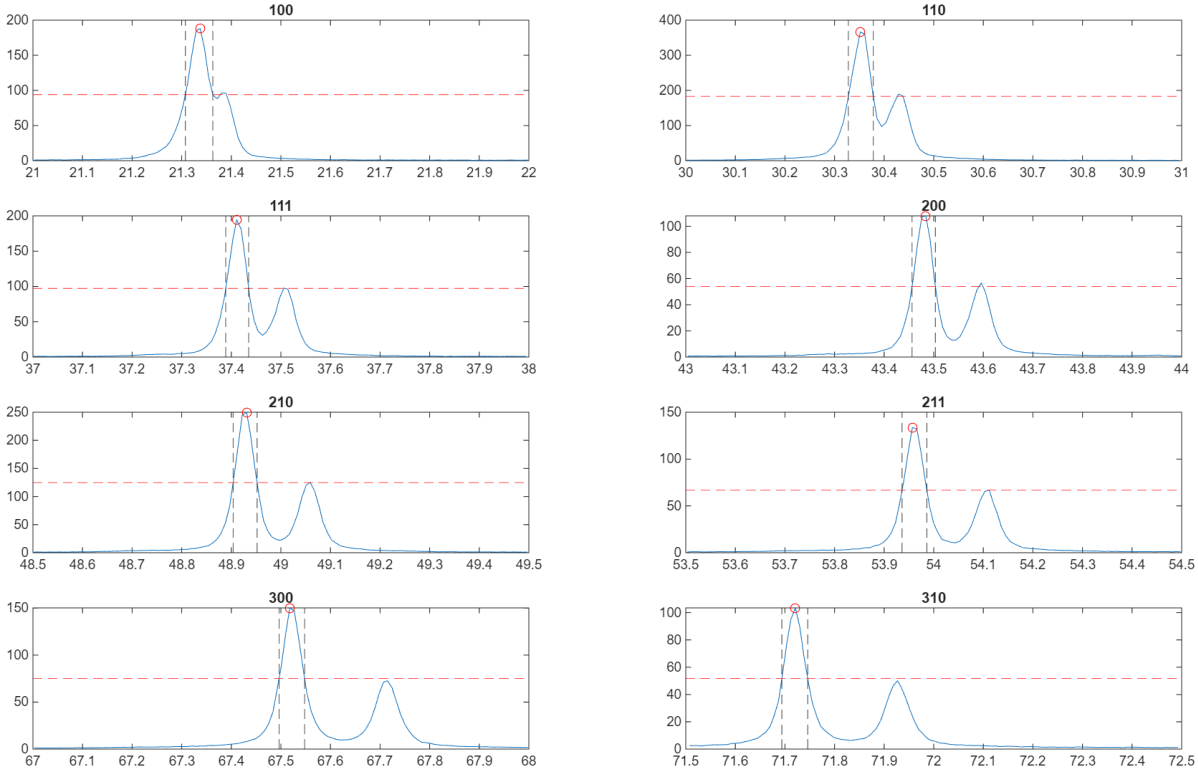

Figure A3: MTRL D6 (Universal stage - New)

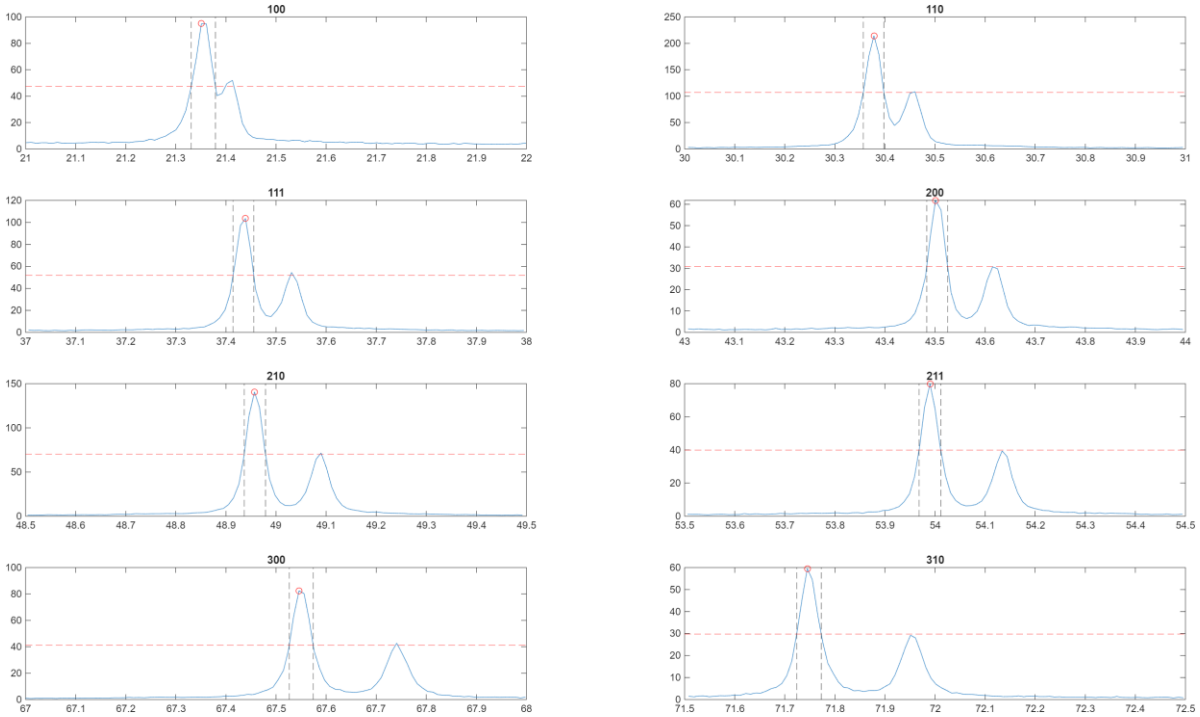

Figure A3: MTRL D6 (fixed stage)

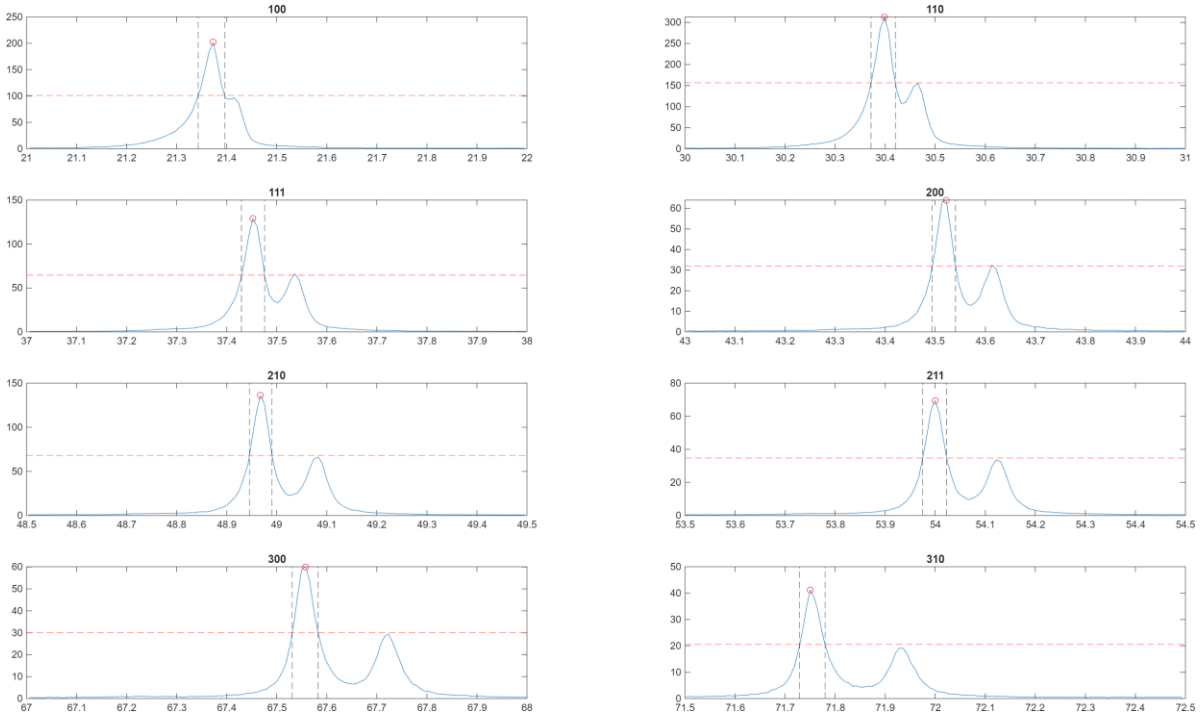

Figure A4: EMXDF Advance

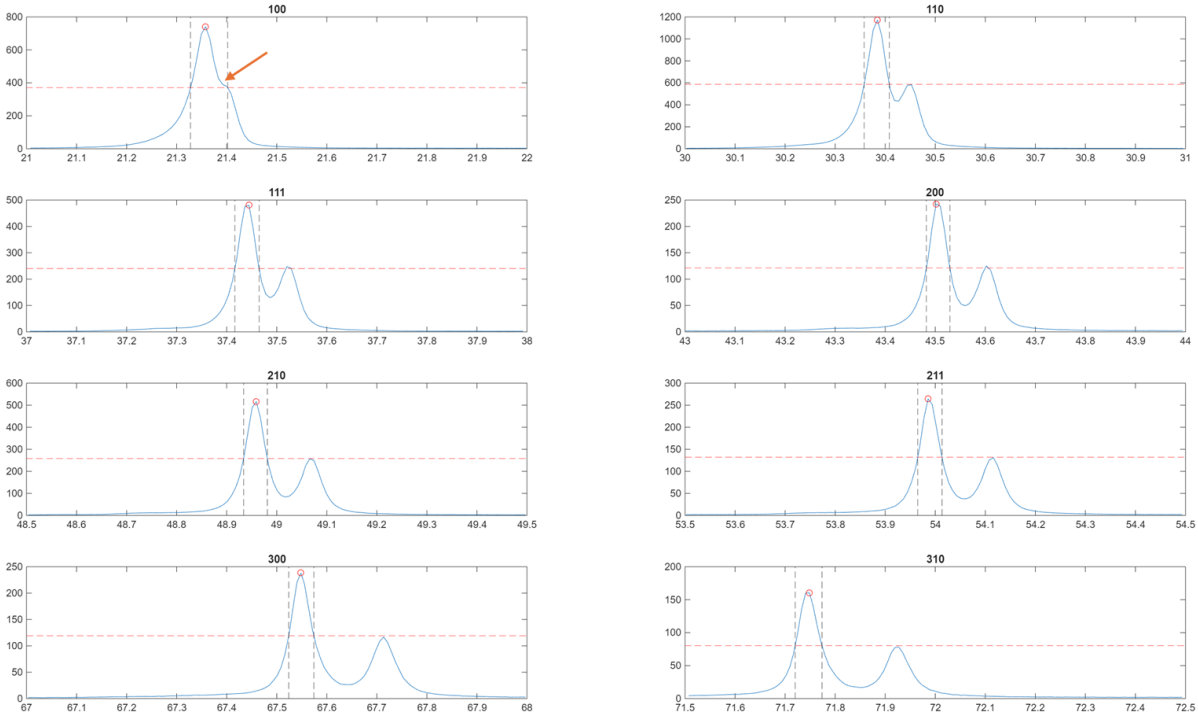

Figure A5: EMXDF Endeavor

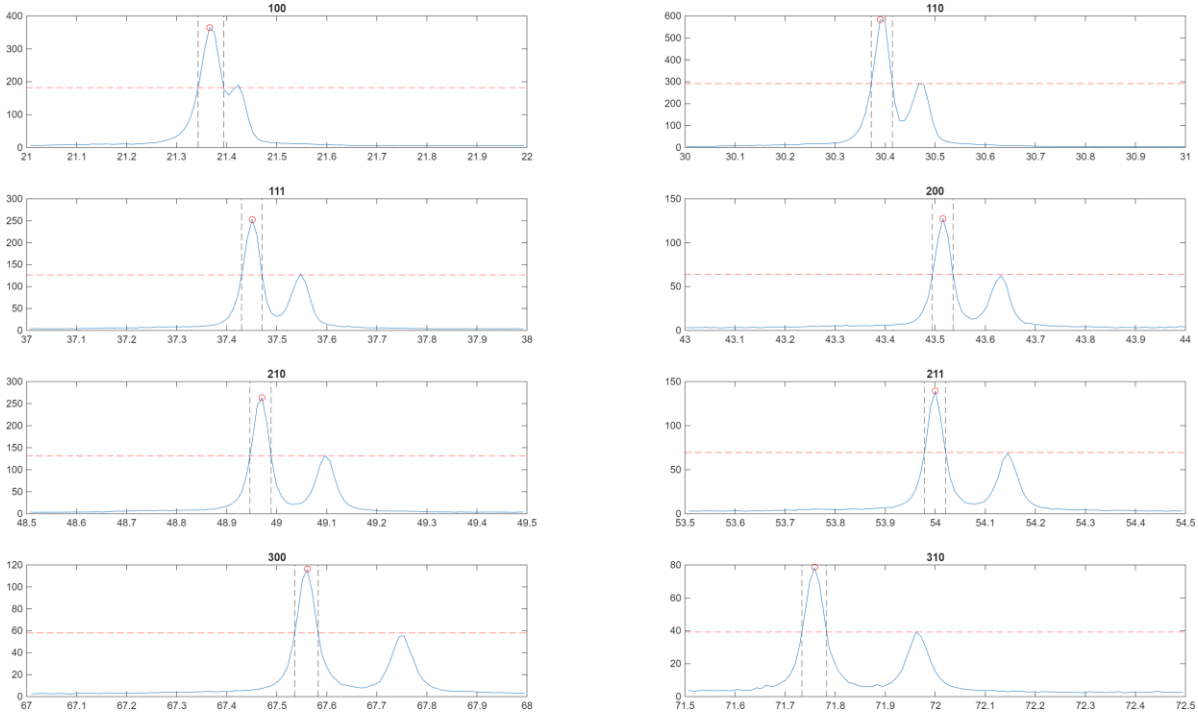

Figure A6: Chem D8