Appendix A. Description of Goniometers and Aspects of Data Collection

Tables A.I and A.II list the details of the experimental conditions for the X-ray measurements. Briefly, a 4-axis (Φ, χ, Ω, 2Θ) goniometer was employed for the stress determinations using both the Ω- and Ψ-goniometer geometries [15]. During scanning, the specimens were oscillated ± 2 mm in plane to improve particle statistics. No stress determinations were performed on the 2-axis (Θ–Θ) goniometer.

Specimen alignment was accomplished using a dial gauge probe, which was accurate to $\pm 5\,\mu$m and a telescope. Here, the relative distance to the center of rotation is known, and the diffracting surface is positioned accordingly. Further, a telescope was initially employed in specimen alignment. The position of the specimen was confirmed by rotating 180° about an axis parallel to the diffracting surface of the specimen and observing that this surface was coincident with the horizontal cross hair at both -90 and $+90°$ χ. Goniometer alignment was ensured by examining LaB_6 powder on a zero background plate. The maximum observed peak shift for the (510) reflection of LaB_6 (141.7° 2Θ) was less than 0.03° 2Θ for Ω and χ tilting as described in Table A.I.

Table A.I. Experimental conditions of the X-ray measurements 4-axis unit.*

Parameter	Condition
Equipment	PTS goniometer Spellman DF3 series 4.0 kW generator Liquid N_2-cooled Ge detector
Power	1.32 kW; 40 kV, 33 mA (long fine focus 0.4x12mm)
Radiation	Cu, λ = 1.54056 Å
Incidence slit divergence	0.12°
Receiving slit acceptance	0.25°; radial divergence limiting (RDL) Soller slit
Source to specimen distance	290 mm
Specimen to back slit distance	290 mm
Tilt axis and angles	χ & Ω; 0, ±55°
Scans	0.02 & 0.05 °2Θ/step

*Software: DMSNT v1.39-1B (build 125).

Table A.II. Experimental conditions of the X-ray measurements 2-axis unit.**

Parameter	Condition
Equipment	Θ-Θ goniometer Seifert ID-3000, 3.5kW generator Bicron scintillation detector with curved graphite diffracted beam monochromator***
Power	1.8 kW; 45 kV, 40 mA (normal focus 1 x 10 mm)
Radiation	Cu, λ = 1.54056 Å
Incidence slit divergence	0.7°
Receiving slit acceptance	0.3 mm
Source to specimen distance	250 mm
Specimen to back slit distance	250 mm
Scans	0.01 °2Θ/step; 1-2°/min.

**Software: DMSNT v1.37.
*** Advanced Ceramics Corp., pyrolytic graphite, ZYA grade, (002) oriented, 0.4° ±0.1° mosaic spread, r = 225 mm.

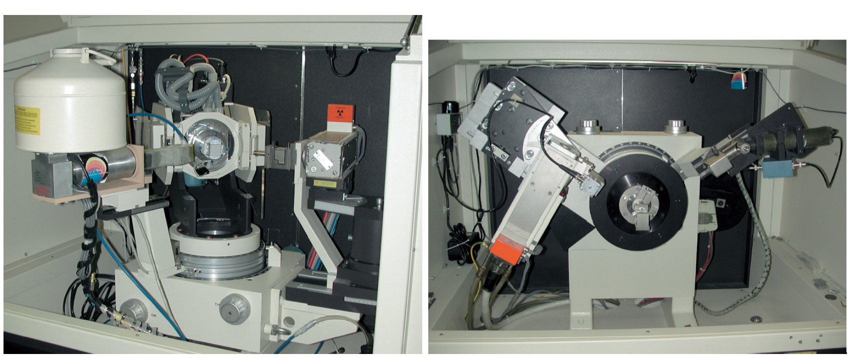
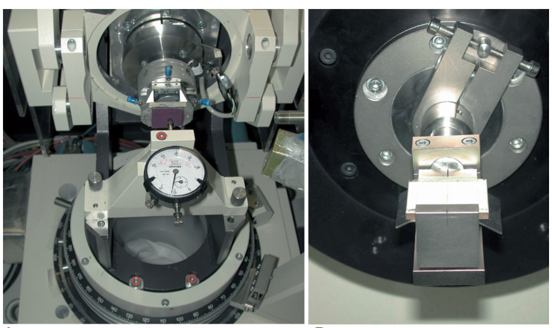


Fig. A1. 4-axis (base Θ–2Θ) and 2-axis (Θ–Θ) goniometers.

Fig. A2. 4-axis unit: Goniometer movements for χ tilting and symmetric diffraction ($\Omega=2\Theta/2$ always). Left to right χ: -55, 0, $+55°$.

Fig. A3. 4-axis unit: Goniometer movements for Ω tilting and asymmetric diffraction ($\Omega\neq2\Theta/2$). Left to right Ω: 25.9, 70.9, 115.9° corresponding to Ψ: -55, 0, $+55°$.



A. B.

Fig. A4. Sample mounts for the (A) 4-axis and (B) Θ–Θ units. The 4-axis unit uses a dial gage probe with $\pm5\,\mu$m precision while the Θ–Θ unit uses a fiducial surface with no easy adjustments.

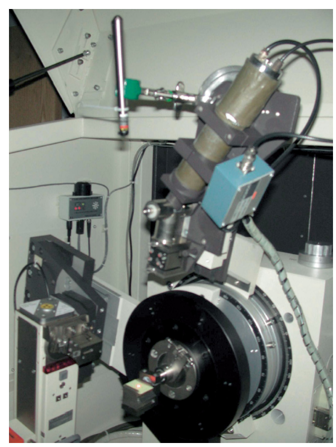


Fig. A5. Θ–Θ unit: Photo showing the utilization of a laser pointer to find the center of rotation of the goniometer.

Appendix B. Diffraction Angle Calibration

PURPOSE

The Diffraction Angle Calibration provides a means to correct the angular position of the diffraction lines for both instrumental and physical aberrations. This correction is useful when accurate peak positions are to be determined, e.g., calculating lattice parameters, as well as improving phase identification results.

CALIBRATION STEPS

1. Prepare a slurry mount of a standard/reference powder –325 mesh particle size (e.g., LaB_6, Si, Al_2O_3) with either methanol or acetone and spread on a sample support, preferably a zero background plate. An Al_2O_3 or quartz plate each of random/near random texture may also be used. For the high temperature unit, the support will be a properly mounted heater strip.

2. Select the divergence and receiving slits typical of those used for samples to be measured on the unit. The irradiated sample length

Table B.I. Lattice parameter refinement of LaB_6 data in Fig. B1 taken on the 4-axis unit.

[D07893.raw] Lab6 on zero bkgd plate — **Cell Refinement Report**

SCAN: 15.0/155.0/0.02/0.2(sec), Cu, I(max)=5129, 06/21/01 08:30

PEAK: 25-pts/Parabolic Filter, Threshold=1.0, Cutoff=0.05%, BG=3/1.0, Peak-Top=Summit

NOTE: Intensity = Counts, 2T(0)=0.0(°), Wavelength to Compute d-Spacing = 1.54056A (Cu/K-alpha1)

Cell Type = Cubic, Pm3m (221)
Refined Cell = 4.15984(0.000402) []
Vol= 71.98 A^3, Density(c)= 4.7002 (Chemical Formula =LaB6, Z=1.0)
2-Theta Error Window = 0.3(°), Zero Offset = 0.0(°), Displacement = 0.0(°)
ESD of Fit = 0.0882(°), IDelta 2-ThetaI = 0.0749(°), IDelta dI = 0.00317(A), F(24) = 13.4(24)
Intensity Weighting = Sqrt(I%), 2-Theta Range = 15.0/155.0(°), (x) Outlier Rejection at 2.0 sigmas
2T(cor) = 2T(obs) - Zero Offset - Displacement (cal=Calculated, obs=Observed, cor=Corrected)

#	(h k l)	2T(cal)	2T(cor)	2T(obs)	Delta	d(cal)	d(cor)	d(obs)	Del-d	I%
1	(1 0 0)	21.342	21.185	21.185	0.157	4.15984	4.19028	4.19028	-0.03043	56.9
2	(1 1 0)	30.362	30.209	30.209	0.153	2.94145	2.95602	2.95602	-0.01457	97.7
3	(1 1 1)	37.414	37.278	37.278	0.136	2.40168	2.41013	2.41013	-0.00844	47.0
4	(2 0 0)	43.473	43.354	43.354	0.119	2.07992	2.08537	2.08537	-0.00545	25.4
5	(2 1 0)	48.920	48.809	48.809	0.111	1.86034	1.86430	1.86430	-0.00397	49.0
6	(2 1 1)	53.946	53.844	53.844	0.102	1.69825	1.70122	1.70122	-0.00298	30.8
7	(2 2 0)	63.167	63.059	63.059	0.108	1.47073	1.47298	1.47298	-0.00226	10.3
8	(2 2 1)	67.492	67.388	67.388	0.104	1.38661	1.38849	1.38849	-0.00188	28.6
9	(3 1 0)	71.685	71.593	71.593	0.093	1.31546	1.31693	1.31693	-0.00147	19.5
10	(3 1 1)	75.779	75.702	75.702	0.077	1.25424	1.25532	1.25532	-0.00108	14.3
11	(2 2 2)	79.800	79.720	79.720	0.080	1.20084	1.20184	1.20184	-0.00100	2.3
12	(3 2 0)	83.771	83.694	83.694	0.077	1.15373	1.15460	1.15460	-0.00087	9.9
13	(3 2 1)	87.711	87.717	87.717	-0.005	1.11176	1.11171	1.11171	0.00005	100.0
14	(4 0 0)	95.579	95.514	95.514	0.065	1.03996	1.04050	1.04050	-0.00054	4.2
15	(3 2 2)	99.544	99.490	99.490	0.054	1.00891	1.00931	1.00931	-0.00040	19.1
16	(3 3 0)	103.555	103.509	103.509	0.045	0.98048	0.98079	0.98079	-0.00031	15.0
17	(3 3 1)	107.635	107.603	107.603	0.032	0.95433	0.95453	0.95453	-0.00019	7.0
18	(4 2 0)	111.810	111.784	111.784	0.026	0.93017	0.93031	0.93031	-0.00014	10.5
19	(4 2 1)	116.110	116.087	116.087	0.023	0.90775	0.90787	0.90787	-0.00011	18.4
20	(3 3 2)	120.576	120.578	120.578	-0.002	0.88688	0.88687	0.88687	0.00001	10.2
21	(4 2 2)	130.228	130.267	130.267	-0.039	0.84912	0.84899	0.84899	0.00014	8.4
22	(4 3 0)	135.595	135.654	135.654	-0.059	0.83197	0.83179	0.83179	0.00018	8.9
23	(5 1 0)	141.535	141.625	141.625	-0.090	0.81581	0.81559	0.81559	0.00022	36.2
24	(3 3 3)	148.382	148.421	148.421	-0.039	0.80056	0.80048	0.80048	0.00008	11.9

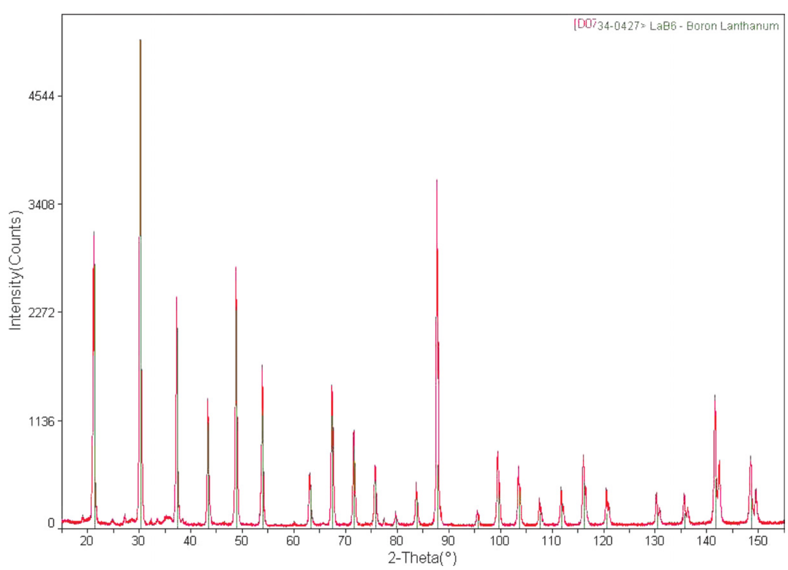


Fig. B1. 4-axis unit: Diffraction pattern of LaB_6 shows peak positions are systematically low relative to PDF card #34-427.

Table B.II. Lattice parameter refinement of LaB_6 data in Fig. B2 taken on the Θ–Θ unit.

[X01593.raw] LaB6 std., Sollers in place — **Cell Refinement Report**

SCAN: 10.0/159.99/0.01/0.2(sec), Cu, I(max)=1866, 06/22/01 15:24

PEAK: 21-pts/Parabolic Filter, Threshold=1.0, Cutoff=0.05%, BG=3/1.0, Peak-Top=Summit

NOTE: Intensity = Counts, 2T(0)=0.0(°), Wavelength to Compute d-Spacing = 1.54056A (Cu/K-alpha1)

Cell Type = Cubic, Pm3m (221)
Refined Cell = 4.15179(0.000177) []
Vol= 71.57 A^3, Density(c)= 4.7276 (Chemical Formula =LaB6, Z=1.0)
2-Theta Error Window = 0.3(°), Zero Offset = 0.0(°), Displacement = 0.0(°)
ESD of Fit = 0.0118(°), IDelta 2-ThetaI = 0.0058(°), IDelta dI = 0.00014(A), F(16) = 171.0(16)
Intensity Weighting = Sqrt(I%), 2-Theta Range = 10.0/159.99(°), (x) Outlier Rejection at 2.0 sigmas
2T(cor) = 2T(obs) - Zero Offset - Displacement (cal=Calculated, obs=Observed, cor=Corrected)

#	(h k l)	2T(cal)	2T(cor)	2T(obs)	Delta	d(cal)	d(cor)	d(obs)	Del-d	I%
1	(1 0 0)	21.384	21.384	21.384	0.000	4.15179	4.15171	4.15171	0.00008	56.2
2	(1 1 0)	30.422	30.426	30.426	-0.004	2.93576	2.93541	2.93541	0.00035	100.0
3	(1 1 1)	37.489	37.499	37.499	-0.010	2.39704	2.39643	2.39643	0.00061	61.1
4	(2 0 0)	43.562	43.569	43.569	-0.007	2.07590	2.07558	2.07558	0.00032	30.1
5	(2 1 0)	49.021	49.028	49.028	-0.007	1.85674	1.85648	1.85648	0.00026	79.7
6	(2 1 1)	54.059	54.062	54.062	-0.003	1.69496	1.69488	1.69488	0.00009	37.8
7	(2 2 0)	63.304	63.307	63.307	-0.003	1.46788	1.46782	1.46782	0.00006	16.5
8	(2 2 1)	67.640	67.642	67.642	-0.002	1.38393	1.38390	1.38390	0.00003	49.2
9	(3 1 0)	71.846	71.846	71.846	-0.001	1.31291	1.31290	1.31290	0.00001	32.8
10	(3 1 1)	75.952	75.950	75.950	0.002	1.25181	1.25184	1.25184	-0.00003	19.7
11	(2 2 2)	79.986	79.987	79.987	-0.001	1.19852	1.19851	1.19851	0.00001	4.1
12	(3 2 0)	83.970	83.966	83.966	0.004	1.15150	1.15154	1.15154	-0.00004	12.8
13	(3 2 1)	87.925	87.920	87.920	0.005	1.10961	1.10967	1.10967	-0.00005	23.2
14	(4 0 0)	95.824	95.809	95.809	0.015	1.03795	1.03807	1.03807	-0.00013	3.5
15	(3 2 2)	99.806	99.793	99.793	0.014	1.00696	1.00706	1.00706	-0.00010	19.6
16	(3 3 0)	103.837	103.821	103.821	0.016	0.97859	0.97869	0.97869	-0.00011	16.9
17	(3 3 1)	107.939	107.913	107.913	0.026	0.95249	0.95264	0.95264	-0.00015 x	6.6
18	(4 2 0)	112.138	112.111	112.111	0.028	0.92837	0.92852	0.92852	-0.00015 x	10.8
19	(4 2 1)	116.467	116.431	116.431	0.037	0.90600	0.90618	0.90618	-0.00018 x	19.2
20	(3 3 2)	120.966	120.921	120.921	0.045	0.88517	0.88536	0.88536	-0.00020 x	9.1
21	(4 2 2)	130.709	130.629	130.629	0.080	0.84748	0.84775	0.84775	-0.00027 x	5.5
22	(4 3 0)	136.142	136.035	136.035	0.107	0.83036	0.83067	0.83067	-0.00031 x	7.2
23	(5 1 0)	142.176	142.026	142.026	0.150	0.81423	0.81460	0.81460	-0.00037 x	23.7
24	(3 3 3)	149.176	148.945	148.945	0.230	0.79901	0.79946	0.79946	-0.00044 x	12.5

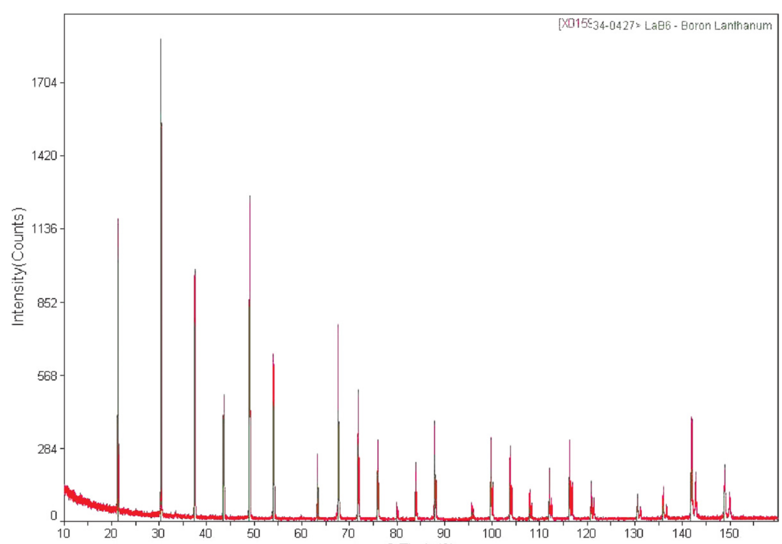


Fig. B2. Θ–Θ unit: Diffraction pattern of LaB_6 with the PDF card # 34-427 superimposed.

Table B.III. Lattice parameter refinement of LaB_6 data in Fig. B3 taken on the 4-axis unit after alignment.

[D07914.raw] Lab6 on a zero bckgd plate | **Cell Refinement Report**

SCAN: 15.0/155.0/0.02/0.2(sec), Cu, I(max)=4748, 06/24/01 09:11

PEAK: 25-pts/Parabolic Filter, Threshold=1.0, Cutoff=0.05%, BG=3/1.0, Peak-Top=Summit

NOTE: Intensity = Counts, 2T(0)=0.0(°), Wavelength to Compute d-Spacing = 1.54056A (Cu/K-alpha1)

Cell Type = Cubic, Pm3m (221)
Refined Cell = 4.15659(0.000018) []
Vol= 71.81 A^3, Density(c)= (Chemical Formula =(Unknown), Z=0.0)
2-Theta Error Window = 0.1(°), Zero Offset = 0.0(°), Displacement = 0.0(°)
ESD of Fit = 0.003(°), lDelta 2-Thetal = 0.0024(°), lDelta dl = 0.00003(A), F(19) = 413.3(19)
Intensity Weighting = Sqrt(I%), 2-Theta Range = 15.0/155.0(°), (x) Outlier Rejection at 2.0 sigmas
2T(cor) = 2T(obs) - Zero Offset - Displacement (cal=Calculated, obs=Observed, cor=Corrected)

#	(h k l)	2T(cal)	2T(cor)	2T(obs)	Delta	d(cal)	d(cor)	d(obs)	Del-d	I%
1	(1 0 0)	21.359	21.337	21.337	0.022	4.15659	4.16089	4.16089	-0.00430 x	55.1
2	(1 1 0)	30.386	30.377	30.377	0.009	2.93915	2.94002	2.94002	-0.00086 x	100.0
3	(1 1 1)	37.444	37.435	37.435	0.009	2.39981	2.40036	2.40036	-0.00055 x	47.9
4	(2 0 0)	43.509	43.508	43.508	0.001	2.07830	2.07835	2.07835	-0.00005	22.7
5	(2 1 0)	48.960	48.963	48.963	-0.003	1.85888	1.85879	1.85879	0.00009	65.7
6	(2 1 1)	53.992	53.992	53.992	0.000	1.69692	1.69692	1.69692	0.00001	33.2
7	(2 2 0)	63.222	63.228	63.228	-0.006	1.46958	1.46945	1.46945	0.00012	12.1
8	(2 2 1)	67.552	67.550	67.550	0.001	1.38553	1.38556	1.38556	-0.00003	32.4
9	(3 1 0)	71.750	71.752	71.752	-0.002	1.31443	1.31439	1.31439	0.00004	23.3
10	(3 1 1)	75.849	75.852	75.852	-0.003	1.25326	1.25321	1.25321	0.00005	16.8
11	(2 2 2)	79.875	79.855	79.855	0.020	1.19990	1.20015	1.20015	-0.00025 x	3.5
12	(3 2 0)	83.851	83.851	83.851	0.000	1.15283	1.15283	1.15283	0.00000	9.8
13	(3 2 1)	87.798	87.794	87.794	0.004	1.11090	1.11093	1.11093	-0.00004	21.2
14	(4 0 0)	95.678	95.677	95.677	0.001	1.03915	1.03916	1.03916	-0.00001	4.1
15	(3 2 2)	99.649	99.652	99.652	-0.002	1.00812	1.00810	1.00810	0.00002	19.8
16	(3 3 0)	103.668	103.670	103.670	-0.001	0.97972	0.97971	0.97971	0.00001	16.2
17	(3 3 1)	107.757	107.763	107.763	-0.006	0.95359	0.95355	0.95355	0.00004	6.6
18	(4 2 0)	111.942	111.940	111.940	0.002	0.92944	0.92945	0.92945	-0.00001	10.5
19	(4 2 1)	116.254	116.256	116.256	-0.002	0.90704	0.90703	0.90703	0.00001	21.6
20	(3 3 2)	120.733	120.733	120.733	0.000	0.88619	0.88619	0.88619	0.00000	9.5
21	(4 2 2)	130.421	130.418	130.418	0.004	0.84846	0.84847	0.84847	-0.00001	7.7
22	(4 3 0)	135.815	135.822	135.822	-0.008	0.83132	0.83130	0.83130	0.00002 x	9.5
23	(5 1 0)	141.792	141.790	141.790	0.002	0.81517	0.81518	0.81518	-0.00001	36.1
24	(3 3 3)	148.700	148.696	148.696	0.004	0.79994	0.79994	0.79994	-0.00001	21.9

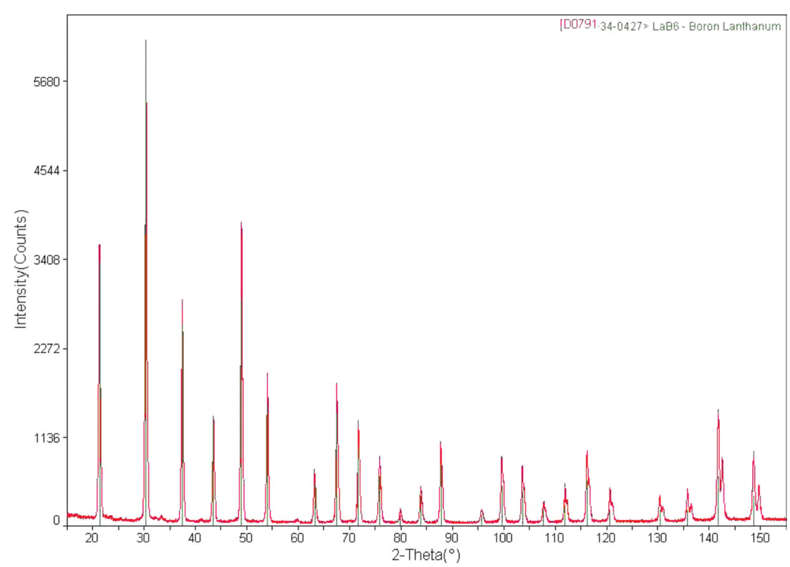


Fig. B3. 4-axis unit: After alignment, diffraction pattern of LaB_6 shows peak positions are near PDF card #34-427.

Table B.IV. Lattice parameter refinement of LaB_6 data in Fig. B3 taken on the Θ–Θ unit after alignment.

[X01604.raw] LaB6 std., Sollers in place	**Cell Refinement Report**
SCAN: 10.0/159.99/0.01/0.2(sec), Cu, I(max)=2072, 07/06/01 21:43	
PEAK: 21-pts/Parabolic Filter, Threshold=1.0, Cutoff=0.05%, BG=3/1.0, Peak-Top=Summit	
NOTE: Intensity = Counts, 2T(0)=0.0(°), Wavelength to Compute d-Spacing = 1.54056A (Cu/K-alpha1)	
Cell Type = Cubic, Pm3m (221) Refined Cell = 4.15686(0.00013) [] Vol= 71.83 A^3, Density(c)= 4.7103 (Chemical Formula =LaB6, Z=1.0) 2-Theta Error Window = 0.1(°), Zero Offset = 0.0(°), Displacement = 0.0(°) ESD of Fit = 0.0143(°), IDelta 2-ThetaI = 0.0114(°), IDelta dI = 0.00023(A), F(21) = 88.0(21) Intensity Weighting = Sqrt(I%), 2-Theta Range = 10.0/159.99(°), (x) Outlier Rejection at 2.0 sigmas 2T(cor) = 2T(obs) - Zero Offset - Displacement (cal=Calculated, obs=Observed, cor=Corrected)	

#	(h k l)	2T(cal)	2T(cor)	2T(obs)	Delta	d(cal)	d(cor)	d(obs)	Del-d	I%
1	(1 0 0)	21.358	21.360	21.360	-0.003	4.15686	4.15637	4.15637	0.00049	43.1
2	(1 1 0)	30.384	30.393	30.393	-0.009	2.93934	2.93849	2.93849	0.00085	100.0
3	(1 1 1)	37.441	37.453	37.453	-0.012	2.39996	2.39924	2.39924	0.00072	43.9
4	(2 0 0)	43.506	43.523	43.523	-0.017	2.07843	2.07765	2.07765	0.00079	37.0
5	(2 1 0)	48.957	48.969	48.969	-0.012	1.85900	1.85857	1.85857	0.00043	65.0
6	(2 1 1)	53.988	53.999	53.999	-0.010	1.69703	1.69673	1.69673	0.00030	39.6
7	(2 2 0)	63.218	63.216	63.216	0.002	1.46967	1.46971	1.46971	-0.00004	12.3
8	(2 2 1)	67.547	67.549	67.549	-0.002	1.38562	1.38558	1.38558	0.00004	34.7
9	(3 1 0)	71.745	71.746	71.746	-0.001	1.31451	1.31449	1.31449	0.00002	26.9
10	(3 1 1)	75.843	75.844	75.844	-0.001	1.25334	1.25333	1.25333	0.00001	14.6
11	(2 2 2)	79.869	79.864	79.864	0.005	1.19998	1.20004	1.20004	-0.00006	2.5
12	(3 2 0)	83.844	83.842	83.842	0.003	1.15291	1.15294	1.15294	-0.00003	9.8
13	(3 2 1)	87.790	87.784	87.784	0.007	1.11097	1.11104	1.11104	-0.00007	20.4
14	(4 0 0)	95.670	95.653	95.653	0.016	1.03922	1.03935	1.03935	-0.00013	4.0
15	(3 2 2)	99.641	99.623	99.623	0.018	1.00819	1.00832	1.00832	-0.00013	17.3
16	(3 3 0)	103.659	103.641	103.641	0.018	0.97978	0.97990	0.97990	-0.00012	12.6
17	(3 3 1)	107.747	107.724	107.724	0.024	0.95365	0.95379	0.95379	-0.00014	6.7
18	(4 2 0)	111.931	111.908	111.908	0.023	0.92950	0.92963	0.92963	-0.00013	8.2
19	(4 2 1)	116.242	116.219	116.219	0.023	0.90710	0.90722	0.90722	-0.00011	17.5
20	(3 3 2)	120.720	120.694	120.694	0.025	0.88625	0.88636	0.88636	-0.00011	7.5
21	(4 2 2)	130.405	130.371	130.371	0.035	0.84852	0.84863	0.84863	-0.00012 x	4.8
22	(4 3 0)	135.796	135.788	135.788	0.008	0.83137	0.83140	0.83140	-0.00002	4.9
23	(5 1 0)	141.771	141.725	141.725	0.046	0.81523	0.81534	0.81534	-0.00011 x	20.2
24	(3 3 3)	148.673	148.622	148.622	0.051	0.79999	0.80009	0.80009	-0.00010 x	11.4

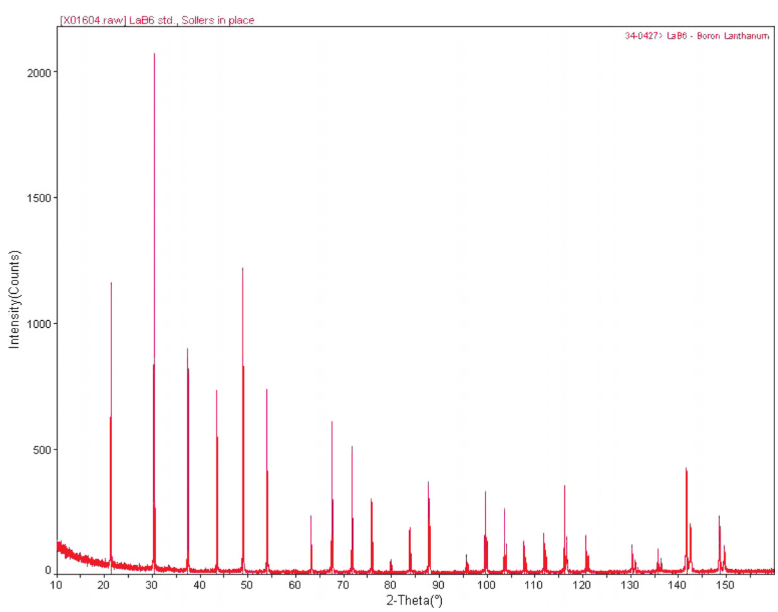


Fig. B4. Θ–Θ unit: After alignment, diffraction pattern of LaB_6 shows peak positions are near PDF card #34-427.

should be <20 mm at the lowest 2Θ of the scan in order to keep the beam on the sample. (If several slit combinations are used, two or three scans with different slits may be required.)

3. Collect a Θ–2Θ powder diffraction pattern in continuous scan mode from ~15° 2Θ (Cu Kα radiation assumed) to the maximum possible diffraction angle of the unit at a step size of 0.02° and a total scan time of at least 4 hours.

4. Use profile-fitting software to obtain the position of each of the diffraction peaks.

5. Use the cell refinement option of your software to determine the calibration curve and save the calibration parameters.

6. Compare the calibration results with those obtained previously. If changes greater than 0.02° occur in any of the peaks, then a reason for the change must be determined before assuming that the calibration, sample mount, or instrument alignment is acceptable.

TEST RECORD

1. Record that the test was performed and indicate the average deviation from the calibration curve in the instrument maintenance and instrument/calibration logbook. Date and sign each entry.

Appendix C. Intensity, FWHM/Resolution and X-ray Wavelength Contamination Tests

PURPOSE

The diffracted intensity and profile breadth tests are used to monitor both the performance of the X-ray tube and the alignment of the goniometer.

BACKGROUND

The intensity and spectrum of the X-ray beam produced from an X-ray tube deteriorates with normal use over time. The high-energy electrons impinging on the target cause erosion of the anode surface, producing a crater. As this depression grows, the intensity of X-rays produced is reduced by the partial absorption of the X-ray by the crater's shoulder. This results in a loss of diffracted intensity and potentially an increase in the breadth of the peak profile as the target focal area becomes larger.

In addition, the high temperature of the tungsten cathode leads to vaporization of tungsten;

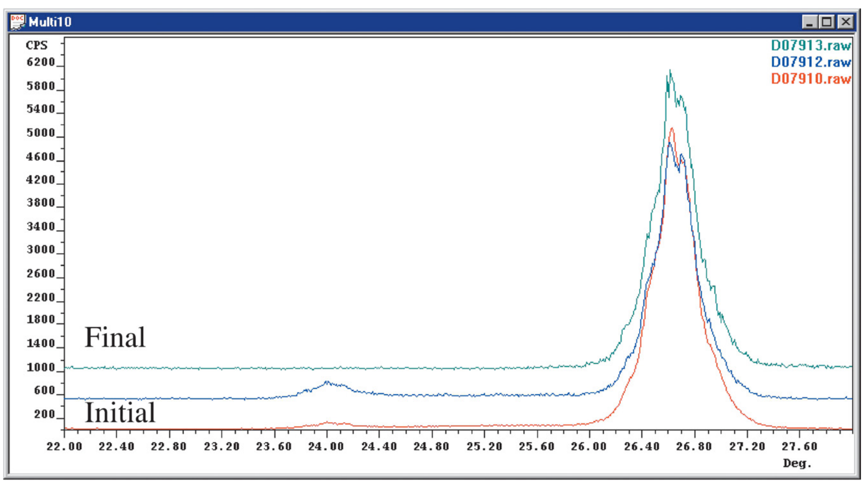
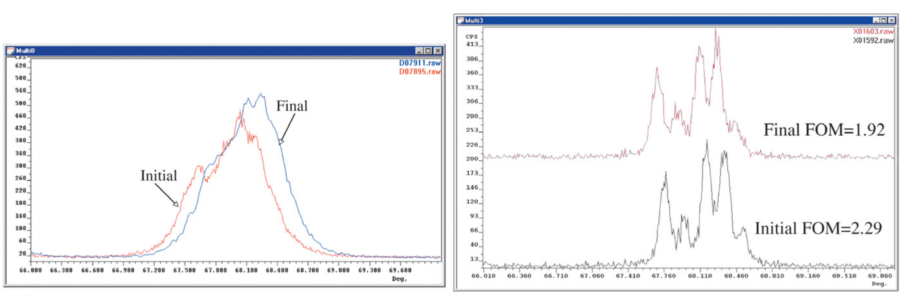


Fig. C1. 4-axis unit: Diffraction pattern of (101) quartz shows reasonable peak intensity and no W or kβ lines.



(A) (B)

Fig. C2. 4-axis and Θ–Θ units: The (A) mitten and (B) five fingers of quartz, respectively.

tungsten then deposits on colder surfaces inside the tube, including the target and the beryllium windows. When this occurs, tungsten L-alpha radiation is produced and emitted in the beam along with the target K-alpha lines and the Bremsstrahlung. This deposited tungsten also reduces the intensity of the target K-alpha lines by absorption of the electron beam as well as the excited X-rays and cannot be discriminated against by the electronics because of the close wavelength of the two.

PROCEDURE

1. Set generator (kV and mA) to normal power level .
2. Install typical slits and record their values.
3. For a Θ–2Θ goniometer with single sample holder attachment mount the quartz plate in the standard manner. For a Θ–Θ goniometer with a high temperature furnace attachment mount the heater reference strip with quartz sample attached and adjust the height of the chamber until the (101) peak is in correct two-theta position.
4. Collect data from a 5° range centered on the (101) and the (212) theoretical peak locations with a maximum step size of 0.01° and a continuous scan rate of $<$1.0°/min.
5. Plot the data for the (101) peak so the peak intensity is full scale. Examine the region for evidence of contaminant radiation peaks. For copper radiation, Cu Kβ occurs at 24.04° and the W Lβ occurs at 25.52° 2Θ.
6. Profile fit the raw data for each of the regions and compare the intensity of the (101) peak with those recorded earlier. If the intensity drops dramatically from the previous value, or has declined to less than 50% of the tube's original values, then replacement of the X-ray tube should be considered.
7. Calculate the figure of merit of the resolution of the "five fingers of quartz" [2]. The figure of merit (FOM) is the intensity of the (212) minus the background intensity divided by the average intensity of the valleys surrounding the (212) line [i.e., trough between $(212)_{\alpha_1-\alpha_2}$, $(212)_{\alpha_2}$–$(203)_{\alpha_1}$, $(301)_{\alpha_1-\alpha_2}$] minus the background intensity. *Generally, an acceptable value for the FOM should be greater than 2.*

Appendix D. Detector Linearity Calibration (Dead Time Correction)

PURPOSE

The purpose of Detector Linearity Calibration is to measure the detector linearity at various count rates, and determine calibration parameters to correct for the intensity data that exceeds the detector linearity limit. The lack of dead-time correction can lead to improper intensity and FWHM values for intense peaks, which partially saturate the detector.

BACKGROUND

At high count rates, some X-ray detectors (e.g., liq. N_2 cooled HPGe and PSD detectors) and associated electronics cannot process each incident photon fast enough to prevent overlap of signals. Missing these counts leads to errors in intensity readings. In other words, at very high count rates, the number of events recorded by the detector is lower than the true number of incident events. Measuring the dead-time parameter permits the system to correct the observed count rate for these effects, up to a maximum observed count rate established during the calibration.

CAUTION!

The measurement of the dead-time should only be performed by the custodian of the X-ray system. The power settings (kV times mA) for the particular X-ray tube should not exceed the recommended maximum settings provided by the tube manufacturer.

PROCEDURE

• The dead-time correction should be determined for each detector on an annual basis, or when experimental conditions have changed (i.e. repair or replacement of detector or detector electronics, etc.).

• The dead-time correction should be determined as per manufacturer software and on-line help if available. If unavailable, the dead-time correction function should be determined manually and recorded.

For Manufacturer Software Equipped Systems:

- The maximum allowable potential (kV) setting for the particular X-ray tube is set, and the current (mA) setting is incremented per software window instructions.

For Systems without Software Packages:

- The dead time, t_d, of the detector and associated electronics is determined by measuring count rate (i.e., measured intensity, I_m) as a function of generator current for the (101) and (202) reflections of quartz.
- At least four scans of these reflections should be made at four "low" generator currents, which result in low count rates (typically $<10^4$ cps).
- These data should be in the linear portion of

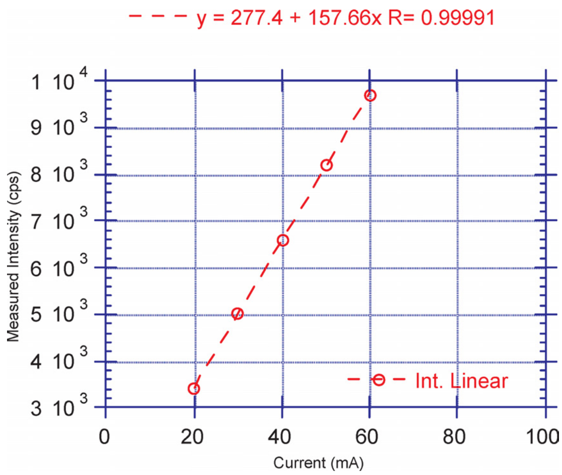


Fig. D1. Measured intensity, I_m, as a function of generator current for the (101) reflection of quartz (linear portion of the curve).

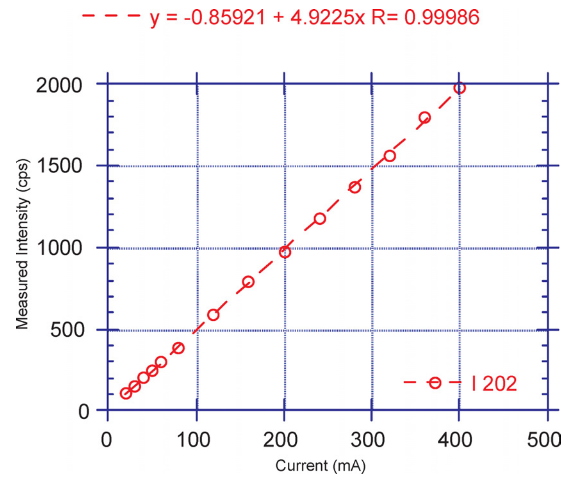


Fig. D3. Measured intensity as a function of generator current for the (202) reflection of quartz.

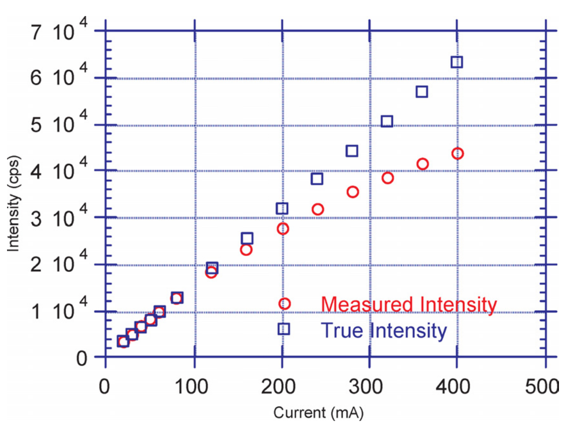


Fig. D2. Measured and true intensities as a function of generator current for the (101) reflection of quartz.

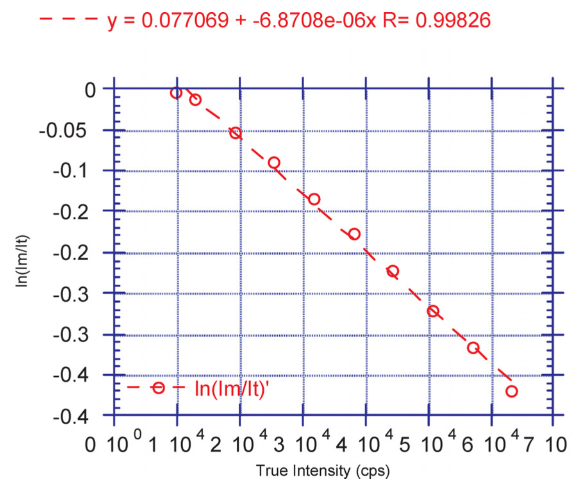


Fig. D4. $\ln(I_m/I_t)$ as a function of I_t for the (101) reflection of quartz (non-linear portion of the curve).

the count rate versus generator current curve.

- These data are then fitted with straight lines in order to predict the true count rates, I_t, at larger generator currents, where dead time errors can occur for high intensity reflections (see Fig. D1).
- Both reflections should also be measured at four "high" generator currents, such that the count rates of the (101) and (202) reflections were non-linear and linear, respectively, with generator current (see Figs. D2 and D3, respectively). This "linear" count rate for the (202) reflection will demonstrate that the detector can be linear at higher generator currents.
- The dead time can be estimated using the following relation:

$$I_m/I_t = \exp(-I_t t_d),$$

where It is the true intensity.

- The dead-time is determined from the slope of Fig. D4 and in our example was found to be 6.9 μs. The data is then corrected using [2]:

$$I_{corr} = I_m/(1 - I_m t_d)$$

Example Measurement

- The example intensities were measured using the dead-time correction program in DMSNT as well as independently. The results from each data set were similar. In Table D.I, the true intensity originates from the linear extrapolation of the data as shown in Fig. D2. The percent fit can be improved if the True intensity is plotted as a function of Measured and fit with either a power law or polynomial function.

Table D.I. The raw and predicted dead-time data.

Generator Current (mA)	True Intensity (cps)	Measured Intensity (cps)	Percent Difference¥	"Manual" Correction*	Percent Fit¥¥
400	63341	43793	31	60115	5
360	57035	41601	27	56060	2
320	50729	38625	24	50787	0
280	44422	35556	20	45611	-3
240	38116	31911	16	39782	-4
200	31809	27768	13	33543	-5
160	25503	23320	9	27262	-7
120	19197	18210	5	20528	-7
80	12890	12713	1	13801	-7
60	9737	9701	0	10322	-6
50	8160	8187	0	8625	-6
40	6584	6602	0	6884	-5
30	5007	5035	-1	5197	-4
20	3431	3394	1	3467	-1

¥ Percent Difference=$(I_t - I_m)/I_t$
* Manual correction= $I_m/(1- I_m t_d)$
¥¥ Percent Fit=$(I_t-I_{predicted})/I_t$

Table D.I. (cont'd).

Power Law Fit**	Percent Fit¥¥	Polynomial Fit***	Percent Fit¥¥
58412	8	62294	2
54840	4	57441	-1
50060	1	51183	-1
45218	-2	45131	-2
39590	-4	38469	-1
33371	-5	31594	1
26928	-6	25036	2
19871	-4	18555	3
12777	1	12840	0
9165	6	10262	-5

** Power Law Fit=$AI_m^B = 0.11565\ I_m^{1.2288}$

*** Polynomial Fit=$A+BI_m+CI_m^2 = 1072+0.6515\ I_m + 1.75\times10^{-5}\ I_m^2$