BRITISH CRYSTALLOGRAPHIC ASSOCIATION

Charity Registration Number 284718

UK Instrument Sensitivity Round Robin

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Introduction.

The seeds of the Round Robin exercise were sown at the Spring meeting at Cardiff in 1995 during discussions with Ron Jenkins of ICDD. It stemmed from a need to evaluate an individual instrument's performance against other instruments, of similar configuration, and set a sensitivity benchmark against which any instrument could be judged. A similar exercise had been completed in the USA and plans were well advanced for other countries to run the same exercise. At the Industrial Group Committee meeting on 2nd May 1995 it was decided to run the USA exercise in the UK. Ron Jenkins supplied details of the experiments, a Quattro Pro spreadsheet to evaluate the measured data and details of the USA results. The event was advertised in Crystallography News and IG Newsletter and the first registration was received in July 1995. In total 35 instruments were registered for the exercise and 33 tests were completed by January 1997 and are included in this report.

Basis of Tests.

To ensure that the participant did not introduce any sample preparation error into the tests the NIST SRM1976 Alumina plate was chosen as the specimen. Test standards were kindly provided by both Philips and Siemens with some participants using their own standard. Participants were required to run a series of 22 scans with prescribed settings of tube current, voltage, step size, slit size and count times. One scan covered the range 1° to 160° with the others just covering a single reflection. The measurement details supplied to complete the Round Robin are given in Appendix 1. The original US Quattro Pro spreadsheet was converted into Excel 5 for UK data evaluation.

Data Extraction

The required data was manually extracted from ASCII files submitted by participants, using Excel 5, as shown below. For tests 1, 2, 3, 5 and 6 average backgrounds from 51.0° to 51.18° and 54.0° to 54.18° were taken. The position and intensity values of the (024) alpha 1 peak and the intensities of alpha 2 peak and valley between the peaks were extracted. For test 4 the five most intense points over the alpha 1 peak and the peak position were taken. Test 7 was performed on the weak peak at 41.7° with a single background (41 to 41.18°) and the same peak information as test 1. In test 8 peak positions and intensities were taken for most peaks along with background intensities at specific angles.

Data extraction.

Angle	Intensity
50.96	139
50.98	124
51.00	135
51.02	123
51.04	101
51.06	122
51.08	126
51.10	124
51.12	109
51.14	116
51.16	124
51.18	124
51.20	124
51.22	117

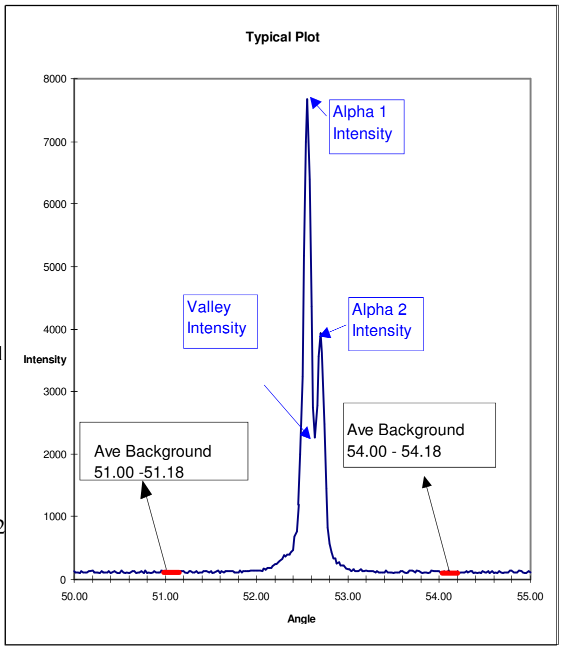
Average
120

Test 4 Intensities

52.40	472	
52.42	676	
52.44	764	
52.46	1189	
52.48	2000	
52.50	3245	
52.52	5172	
52.54	6993	
52.56	7680	Alpha1
52.58	6407	
52.60	4354	
52.62	2748	
52.64	2270	Valley
52.66	2782	
52.68	3571	
52.70	3938	Alpha2
52.72	3606	
52.74	2477	
52.76	1450	
52.78	824	
52.80	569	

53.98	122
54.00	125
54.02	96
54.04	116
54.06	134
54.08	99
54.10	136
54.12	111
54.14	118
54.16	101
54.18	120
54.20	127

Average
116



<u>Participants.</u>

Data for Thirty-three instruments were submitted for inclusion. A list of participants is given in Appendix 2 , some included more than one instrument. Each participant was given a unique number (starting at 20) to ensure their anonymity and yet still allow them to identify their data in this report. Both Philips (19) and Siemens (13) instruments are represented with one Picker 2 circle. A range of instrument parameters was used across the instruments with both Copper and Cobalt tubes, primary and secondary beam monochromators and both fixed and variable slits. A detailed list of the usual Instrument parameters used on each instrument is given in Appendix 3.
Table 1 below summarises the instrument settings used. It shows the variety of slits used, especially for Siemens instruments, but overall it is not difficult to specify a set of generic instrument parameters as shown in bold characters. This provides a useful starting point for instrument settings for new diffraction users.

Table 1

Instrument Statistics

		Philips	Siemens	
Model	PW1050	10		
	PW18XX	5	9	D500
	X-Pert	4	4	D5000
Tube	Co LFF	2	2	
	Cu Normal	2	2	
	Cu Broad	2		
	Cu LFF	**13**	**8**	
	Cu AEG		1	
Radius	200	1	1	
	250	2	2	
	410		2	
	Standard	**16**	**8**	
Sec Monochrom	None	2	3	
	Kevex ED		1	
	Graphite	**17**	**9**	
Geometry	Theta/Theta	1	1	
	T/2T	**18**	**12**	
Divergence Slit	Variable	6	2	
	0.3		3	
	0.5	2		
	1	**11**	**7**	
	2		1	
Receiving Slit	0.05		4	
	0.1	6		
	0.15		3	
	0.2	**12**	3	
	1	1	2	
	2		1	
Detector	Proportional	**19**		
	Scintillation		**12**	
	Kevex ED		1	

Results.

The exercise was divided into eight tests with each test focusing on a particular instrument parameter. The spreadsheet calculations reduced the data to a suitable figure to allow easy comparison between instruments. The calculations performed within the spreadsheet to arrive at these numbers are detailed in Appendix 4. A summary of all the calculated test results for all instruments is given in Appendix 5.

Test 1 mA variation.

This test was used to give the comparative counts per second per mA value. The countrates at all three mA setting have been plotted against intensity for all instruments and are shown for each instrument in Appendix 6. The plots have been individually scaled for intensity so beware of direct comparison. A straight line between zero and the highest value has been added. The data should fall in straight lines if the detector is not being saturated and the dead time circuitry is functioning correctly. Non linearity with primary beam monochromators is an indication of misalignment. The plots for users 23, 38, 44, 48, and 50 are not linear and indicate problems. The test 1 data was also used to estimate deadtime. The counting statistics were not ideal to give an accurate determination. It is recommended that users with a Not Detm comment in the summary determine the deadtime more accurately for their system. Also in test 1 a Peak to background ratio (pk/Bg) was measured, a similar determination was made in test 2. For most applications a high pk/Bg is beneficial.

Test 2 increasing kV

The increase in background with increased kV was determined. Lower values for the calculated ratio are preferred. The results are comparable with the US data. The high UK Philips average is the result of exceptionally high values obtained by users 22 and 27. An exponent value (Exp) is also calculated from the peak intensities. It should be just greater than one for copper radiation.

Test 3 Receiving slit variation.

Participants were asked to scan using three different receiving slit sizes. A wide range of slits was used in this test and in test 5 where the divergence slit was varied. A summary of the slits employed for both tests along with the test results is shown in table 2. The intensity ratio values quoted in the summary sheet (med/sm, lg/sm) should approximate the ratio of the slit sizes used. Low values indicate that over illumination of the slit by the beam has occurred, and that the optimum slit size has been exceeded.

Test 5 Divergence slits.

Participants were asked to use the kVand mA settings of test 3 and their usual receiving slit. Three scans were performed with 1/4, 1/2, and 1° divergence slits. The higher the reported Figure of Merit (FOM) the better the instrument performance. The UK data is comparable with USA data with Philips instruments giving larger FOM's.

Table 2

User	Test 3 Receiving Slit size			Test 3 Results		Test 5 Divergence Slit size			Test 5 Results	
	Small	Medium	Large	med/sm	lg/sm	Small	Medium	Large	Small	Medium
21	0.1	0.2	1.57	**1.60**	**2.51**	0.17	0.50	1.00	**0.88**	**0.14**
22	0.1	0.2	0	**2.04**	**0.00**	0.25	0.50	1.00	**0.73**	**0.51**
23	0.1	0.3	3	**2.81**	**13.49**	0.30	1.00	0.00	**0.87**	**0.14**
24	0.1	0.2	4	**1.90**	**2.78**	0.25	0.50	1.00	**0.99**	**0.17**
25	0.05	0.1	1.8	**2.45**	**5.62**	0.25	0.50	1.00	**0.95**	**0.18**
26	0.1	0.2	1.8	**1.73**	**2.36**	0.17	0.50	1.00	**0.84**	**0.16**
27	0.1	0.2	0.3	**1.70**	**2.23**	0.25	0.50	1.00	**0.80**	**0.38**
28	0.05	0.1	3	**1.85**	**4.84**	0.25	0.50	1.00	**1.10**	**0.43**
29	1	3	0	**0.99**	**0.00**				**0.74**	**0.56**
30	0.018	0.05	0.6	**3.12**	**5.04**	0.30	0.60	1.00	**0.88**	**0.18**
31				**1.69**	**2.44**	0.25	0.50	1.00	**0.86**	**0.15**
32	0.018	0.05	0.6	**3.36**	**5.48**	0.25	0.50	1.00	**1.01**	**0.23**
33	0.05	0.1	6	**1.89**	**5.19**	0.25	0.50	1.00	**0.77**	**0.27**
34	0.1	0.2	3	**2.27**	**3.46**	0.10	0.30	1.00	**0.88**	**0.16**
35	0.1	0.2	2	**1.97**	**5.82**	0.60	1.00	2.00	**0.29**	**0.42**
36	0.1	0.3		**2.03**	**No Data**	0.25	0.50		**1.13**	**0.48**
37	0.018	0.05	0.6	**2.74**	**5.39**	0.10	0.30	1.00	**0.68**	**0.49**
38	0.1	0.20	0.30	**1.75**	**2.22**	0.25	0.50	1.00	**0.82**	**0.32**
39										
40	0.1	0.3		**2.00**	**No Data**	0.25	0.50		**1.04**	**0.37**
41	0.018	0.05	0.6	**2.54**	**3.88**	0.10	0.30	1.00	**1.10**	**0.43**
42	0.05	0.1	0.2	**1.69**	**3.03**	0.25	0.50	1.00	**0.86**	**0.17**
43	0.1	0.2	0.3	**1.69**	**2.09**	0.25	0.50	1.00	**0.67**	**0.49**
44	0.018	0.5	6	**8.39**	**11.68**	0.25	0.50	1.00	**0.93**	**0.21**
45				**No Data**	**No Data**				**0.90**	**0.18**
46	0.1	0.2	1	**1.57**	**2.03**	0.25	0.50	1.00	**0.88**	**0.19**
47	0.1	0.2	0.3	**1.86**	**2.46**	0.25	0.50	1.00	**0.90**	**0.15**
48	0.018	0.05	0.6	**2.93**	**4.23**	0.25	0.50	1.00	**0.89**	**0.16**
49	0.05	0.10	3.00	**1.60**	**3.84**	0.25	0.50	1.00	**0.64**	**0.48**
50	0.01	0.05	0.2	**5.39**	**9.37**	0.04	1.00	0.00		
51										
52	0.1	0.2	1	**1.85**	**2.65**	0.25	0.50	1.00	**0.65**	**0.45**
53	0.05	0.1	2	**1.83**	**4.54**	0.60	1.00	2.00	**0.81**	**0.14**
54	0.05	0.1	0.6	**1.93**	**5.43**	0.25	0.50	1.00	**1.03**	**0.17**
55	0.1	0.2	0.3	**2.39**	**2.44**	0.50	1.00	0.00	**0.65**	**0.45**

Test 4 Step size

Participants were asked to use the kVand mA settings of test 3 and their usual receiving slit and perform scans with 0.01, 0.02, 0.04, 0.06 and 0.08 step sizes. This test identifies the maximum step size which can be used without adversely affecting the calculated peak position. The figures quoted are the peak shifts relative to the smallest step size. It shows that 0.02 steps are safe for all instruments and 0.04 on some. Above 0.04, shifts of 0.03° to 0.04° are obtained on the 52.5° peak. The results for the UK and USA show the same trend, with little difference between manufacturers.

Test 6 No Secondary Soller Slit

Most participants were reluctant to remove the Soller slits. Only nine data sets are available. The data shows the significant peak intensity gain obtained (Wo/W) with only a small loss of resolution(p/v). The background (Bg) also increases in most cases with the same ratio as the peak. If low intensity is a problem then removal of the Secondary Soller slit is in most cases recommended. The UK and USA data is comparable and shows that the increase in intensity is bigger on Siemens' instruments.

Test 7 Count time on weak peak

The effect of increasing count time per step on the weak peak at 41.7° was measured. One second and nine second times were compared for net peak intensity. The data shows that for qualitative analysis a one second counting time is adequate for detecting weak peaks. The USA and UK results are in good agreement.

Test 8 Long scan

A scan from 1° to 160° (or as wide a range as possible) using 45kV and 35mA, 0.02 steps, 2 seconds per step and the user's usual slits was requested. The data extracted were peak positions, peak intensities and background intensities. Intensity data and peak position data from the NIST instrument in the USA were used as the standard against which all other data sets were compared. If necessary the intensity data was converted from variable to fixed slit values so that direct comparison of the intensities of all instruments could be made. Each peak intensity was scaled against the strongest peak and given a percentage relative intensity ratio. This relative intensity ratio was divided by that obtained from the NIST data set to give a ratio that should ideally be unity. This comparative data for all the peaks in the scan was averaged (Av) and the standard deviation (sd) calculated and these figures are reported in the Summary. Average intensities are lower in the UK and standard deviations are a factor of 3 worse in the UK.

Appendix 7 shows a plot of intensity ratio for all instruments. The scale on the y-axis is 0.3 to 1.9 plotted against angle on the x-axis. It is clear from this plot that many instruments show the same pattern of deviation from unity. This points to an error in the NIST intensities possibly introduced in the conversion of the spreadsheet to Excel. The peak intensity data was recalculated using the certified peak intensity data provided on the NIST SRM1976 data sheet. This removed the pattern observed in the previous data and put most instruments within the 1+/-0.0785 specified on the standard certificate for an instrument in control. The modified intensity ratio plots are shown in Appendix 8 and the individual intensity values are given in Table 3 below. The new intensity ratios also improve the averages and standard deviations and bring them into line with the US values as shown.

		Average	Std Dev
	UK	0.86	0.31
Philips	UK Mod	1.02	0.09
	US	0.94	0.11
	UK	0.84	0.27
Siemens	UK Mod	1.05	0.11
	US	1.05	0.14

It is now clear to see that some instruments fitted with variable divergence slits show intensity problems. The data for Cobalt tubes all show the same trend of increasing intensity deviation with angle. This is being investigated by Ron Jenkins and will be discussed at the Leeds workshop.

Table 3 Modified Intensity Ratios

Angle	25.58	35.16	43.36	52.56	57.5	68.2	76.88	95.24	101.08	116.56	127.68	136.06	145.2	Average	Std Dev
21	1.060	1.000	1.034	1.077	1.039	1.022	1.035	1.099	1.128	0.981	1.118	1.058	1.081	**1.056**	**0.044**
22Co	1.019	1.000	0.954	0.949	0.877	0.910	0.969	1.229	1.279					**1.021**	**0.139**
23	0.969	1.000	1.108	1.142	1.032	0.977	1.030	0.987	1.058	0.851	0.992	1.045	1.082	**1.021**	**0.073**
24	1.093	1.000	1.007	0.999	0.955	1.005	0.978	1.145	1.067	1.197	1.145	1.231	1.211	**1.079**	**0.097**
25	0.875	1.000	0.905	0.872	0.886	0.919	0.980	1.027	1.046	0.958	0.995	0.982	1.053	**0.961**	**0.064**
26	1.002	1.000	0.966	0.944	1.036	0.964	0.988	1.016	1.009	1.013	0.988	1.046		**0.998**	**0.030**
27	0.954	1.000	1.009	1.059	1.024	1.065	1.093	1.142	1.131	1.172	1.122			**1.070**	**0.068**
28v	1.061	1.107	1.105	1.034	1.000	0.962	1.019	0.924	0.944	0.974	0.863	0.819	0.873	**0.976**	**0.091**
29	1.153	1.000	0.949	1.019	0.941	0.998	0.616	1.499	1.755					**1.103**	**0.336**
30	0.955	1.000	1.018	1.039	1.058	1.057	1.123	1.081	1.147	1.149	1.082	1.029	1.002	**1.057**	**0.059**
31	1.040	1.000	1.030	1.045	1.049	0.912	1.009	1.070	1.019	1.096	1.083	1.036	1.102	**1.038**	**0.049**
32	0.977	1.000	0.956	0.979	0.978	1.028	1.117	1.154	1.166	1.213	1.286	1.300	1.478	**1.126**	**0.161**
33v	1.061	1.000	0.966	0.959	1.020	1.003	0.965	0.933	0.934	1.022	0.949	0.948		**0.980**	**0.040**
34	1.006	1.000	1.016	1.020	1.041	1.129	1.065	1.133	1.070	1.064	1.112	0.986	1.151	**1.061**	**0.056**
35	1.035	1.000	1.011	1.074	1.052									**1.034**	**0.030**
36v	1.144	1.025	1.009	1.050	1.000	0.889	1.046	0.990	0.995	0.977	0.943	0.944	0.984	**1.000**	**0.062**
37Co	1.075	1.000	0.924	0.966	0.991	0.936	1.098	1.278	1.347					**1.068**	**0.151**
38v	1.365	1.285	1.178	1.101	1.000	1.021	0.854	0.717	0.719	0.608	0.566	0.531		**0.912**	**0.287**
40v	1.139	1.078	1.004	1.005	1.000	0.988	0.939	0.904	0.922	0.726	0.866	0.797	0.872	**0.942**	**0.112**
41	0.995	1.000	0.972	1.015	0.989	0.996	1.069	1.157	1.195	1.276	1.325	1.260	1.427	**1.129**	**0.154**
42	0.873	1.000	0.924	0.888	0.994	0.982	1.039	1.007	0.940	0.979	0.949	0.992	1.030	**0.969**	**0.051**
43vCo	0.852	1.000	1.033	1.060	1.026	1.056	1.119	1.278	1.476					**1.100**	**0.180**
44	0.958	1.000	1.014	0.803	1.023	0.929	1.099	1.285	1.126	1.181	1.198	1.206	1.224	**1.081**	**0.140**
45	1.006	1.000	1.010	1.067	1.044	1.114	1.089	1.053	1.092	1.109	1.034	1.057	1.106	**1.060**	**0.040**
46	1.006	1.000	1.010	1.067	1.044	1.114	1.089							**1.047**	**0.045**
47	1.039	1.000	0.990	0.996	0.969	0.873	0.976	1.056	0.943	1.003	1.006	1.030	1.119	**1.000**	**0.058**
48	0.995	1.000	1.053	0.984	1.027	0.938	1.086	1.122	1.126	1.091	1.121	1.095	1.013	**1.050**	**0.061**
49v	1.320	1.280	1.070	1.070	1.000	1.010	0.900	0.860	0.770					**1.031**	**0.182**
50	0.951	1.000	1.046	1.046	1.044	1.155	0.952	0.976	1.062	0.842	1.072	1.148	1.013	**1.024**	**0.084**
52	0.965	1.000	0.954	0.965	0.971	1.002	1.044	1.151	1.072					**1.014**	**0.065**
53	1.050	1.000	0.963	0.963	1.026	0.872	0.884	0.943	0.951	0.910	0.952	0.966	0.975	**0.958**	**0.051**
54v	1.106	1.069	0.999	0.975	1.000	1.085	0.998	0.927	0.893	0.978	0.856			**0.990**	**0.078**
55	1.042	1.000	0.977	0.960	0.980	1.056	0.992	1.032	1.076					**1.013**	**0.040**

Average Siemens	**1.051**	**0.107**
Average Philips	**1.015**	**0.092**

Appendix 8 shows the background data for all instruments. The scales are; intensity ratio to the NIST instrument for the y-axis and angle for the x-axis. Even a range of 0 to 3.5 in the ratio is not wide enough to fit all the data from some instruments. The data from 22,35 and 47 show the high background values at low angle expected from the use of a beta filter monocromator. The data from 29 shows a peak at 10° with a 7.2 ratio. The data from 21,27 and 52 show high values of background intensity ratio at 5° with 19, 27 and 29 ratios respectively, possibly due to sample holder scatter.

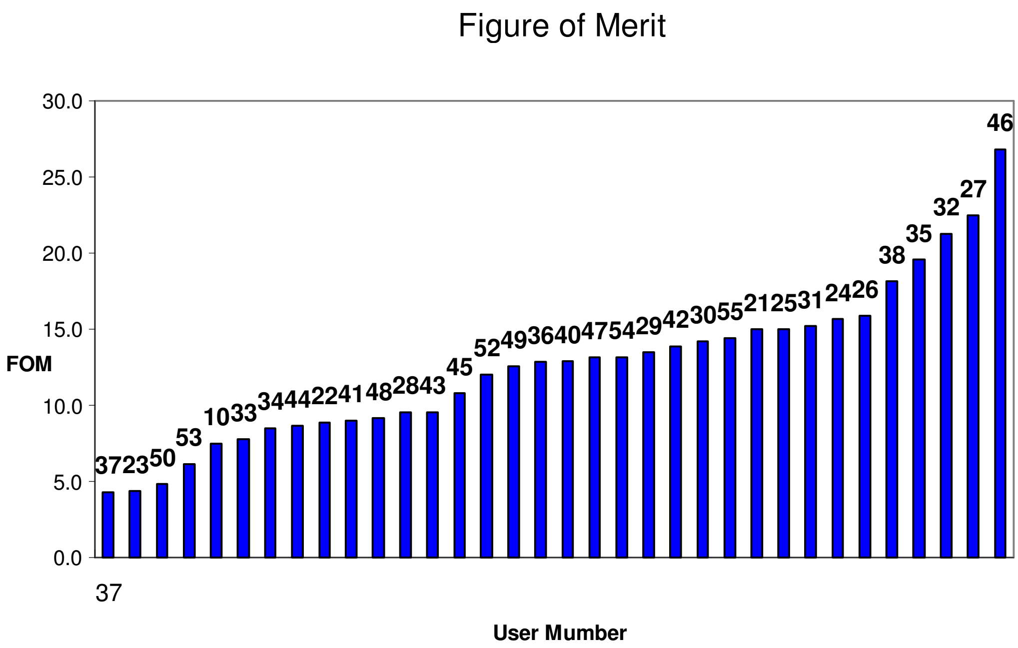
Each participant was provided with a plot showing peak displacement relative to the NIST data. The plots show that many instruments have displacement problems. These plots are shown in Appendix 10. They all are on the same scale of displacement -0.12° to +0.12° on the y-axis and angle on the x-axis. The user number is given at the top of each plot, the addition of “Co” signifies data from a Cobalt tube and “v” that a variable divergence slit was employed. The data shows that many instruments have angular displacement problems. Some have a simple displacement over the whole angular range e.g. user 40 which is easily corrected. Others have displacement that varies with angle e.g. user 29 which is symptomatic of a one to two theta alignment error.

Instrument Benchmark

From the US data Jenkins and Schreiner derived a Figure of Merit (FOM) to test instrument sensitivity - it is fully explained in a book by Jenkins and Snyder(ref 1). I have taken Test 1c

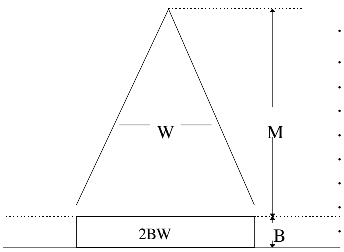
UK data to calculate the FOM. this gives participants a benchmark to test their instrument sensitivity against other similar instruments
The data for 10 and 46 are from the same instrument with a change in X-ray tube, all other parameters remained the same. The 46 data set was obtained with a new X-ray tube, of the same specification (Cu LFF), as an old one used for the 10 data.

User ID	FOM
37	4.3
23	4.4
50	4.8
53	6.1
10	7.5
33	7.8
34	8.5
44	8.6
22	8.9
41	9.0
48	9.1
28	9.5
43	9.5
45	10.8
52	12.0
49	12.5
36	12.8
40	12.9
47	13.1
54	13.2
29	13.5
42	13.9
30	14.2
55	14.4
21	15.0
25	15.0
31	15.2
24	15.7
26	15.9
38	18.1
35	19.6
32	21.2
27	22.5
46	26.8



Calculating the FOM

Instrument Parameter Figure of Merit



$$FOM = \frac{MW}{\sqrt{W(M + 4B)}}$$

M = Net Intensity cps
B = Average Background
W = FWHM

The higher the value of the FOM the better the sensitivity. The FOM's derived form a useful basis for testing the performance of any instrument. The value calculated for an individual instrument can be used to validate that any changes in alignment or measurement parameters have improved performance.

Conclusions

There is a wide spread of performance, many instruments have angular displacement problems which are easily overcome. The errors found demonstrate the need to frequently check goniometer angles and calibrate at more than just the single angular position which is common practice amongst the diffraction community. Countrates are significantly lower than in the US. This could be due to X-ray tube degradation. Perhaps routine instrument checks should be more widely used to ensure that equipment performs to manufacturer's specification and that X-ray tube degradation is monitored. Routine checks should monitor overall or angular dependent loss in intensity or shifts in 2 theta values and changes in peak shape. The NIST SRM 1976 is a useful standard to use for such tests especially where they are performed as part of an accreditation procedure. Test 8 could be used as the basis for the test but modified to use your normal power setting. These data could then be used to generate the displacement, background and intensity plots and the 52.5° peak could be used to give the instrument sensitivity FOM. All instrument manufacturers should supply an instrument routine check programme and certified standard with their systems.

Reference

1. *Introduction to X-Ray Powder Diffractometry - Jenkins & Snyder - John Wiley & Sons Inc. p224-226 ISBN 0-471-51339-3*

APPENDIX 1

Measurement instructions UK Sensitivity Round Robin

USER NUMBER.......... *a unique identifier supplied by the co-ordinator*

Serial Number of SRM 1976 used ....(on front of standard box)

All measurements to be made using NIST SRM1976 Alumina plate

All angles quoted are for a Copper tube and will require conversion for other anode materials.

Where possible the following file name convention should be used:-

****uk??** (Where ** is User identity number and ?? is test number e.g. 1a)

For tests 1 to 5 the following conditions apply:-

Scan 50-55° , Step size 0.02 ° (except test 4), count time 4 seconds/step

MAKE SURE YOU DO NOT EXCEED YOUR TUBE LOADING.

Test 1 Calculate X from X= Max. tube loading in watts ÷ 180
e.g. for 2 kW tube X= 2000 ÷ 180 = 11.11
X is rounded to a mA interval suitable for your generator (e.g. for 11.11 use 10 or 11 mA)
Use your usual slits and 45 KV for this test

Divergence slit used...........*Receiving slit used............Detector type............ *eg Scint*

**Mark VAR for variable divergence slit*

1a	Scan at 1X mA (eg 1.8 kW tube use 10 mA)	***mA used..........File..........***
1b	Scan at 2X mA (eg 1.8 kW tube use 20 mA)	***mA used..........File..........***
1c	Scan at 4X mA (eg 1.8 kW tube use 40 mA)	***mA used..........File..........***
Test 2	Use your usual slits and 20 mA	
2a	Scan at 30 KV	***File name................***
2b	Scan at 40 KV	***File name................***
2c	Scan at 45 KV	***File name................***
2d	Scan at 50 KV	***File name................***
Test 3	Use your usual divergence slit, 45 KV and 35 mA (or as close as your generator will allow). ***Values used KV........... mA...........***	
3a	Scan using smallest available receiving slit	***Value used..........File.........***
3b	Scan using next smallest avail receiving slit	***Value used.........File..........***
3c	Scan using largest available receiving slit	***Value used.........File..........***
Test 4	Use your usual slits and Test 3 power	
4a	Use 0.01° step size	***File name................***
4b	Use 0.02° step size	***File name................***
4c	Use 0.04° step size	***File name................***
4d	Use 0.06° step size	***File name................***
4e	Use 0.08° step size	***File name................***

Test 5	**For fixed divergence slit systems only**	
	Use usual receiving slit and Test 3 power	
5a	Scan with 1/4° divergence slit	***File name***................
5b	Scan with 1/2° divergence slit	***File name***................
5c	Scan with 1° divergence slit	***File name***................
Test 6	Remove secondary soller collimator (if possible) and repeat test 4b	***File name***................
Test 7	Obtain profile of weak peak at 41.7° by scanning 41° to 42.5° step size 0.02° , your usual slits and Test 3 power	
7a	Scan at 1 second/step	***File name***................
7b	Scan at 9 second/step	***File name***................
Test 8	Perform as long a scan as possible starting at 1° 2 Θ and scanning to say 160° 2 Θ. Use Test 3 power, 0.02° step size, 2 seconds / step and your usual slits.	***File name***................

Reporting results - For all tests:-

1 Plot each scan with the largest peak at about full scale For test 8 split into 20° segments and scale individually

2 Computer printout of raw count data vs. 2Θ (Disable any automatic angular correction made by your system) or raw data onto IBM PC compatible floppy

APPENDIX 2

UK Round Robin Participants *Italics Siemens* / Normal- Philips + Picker 2 circle

D Beveridge - Ilford Ltd
Mr Bruce Fox - Raychem Ltd
Ms Michele Leigh- De Beers Industrial Diamond Division
Mr NL Andrew - Philips Research Laboratories
Mr R R Giles - Alcan Chemicals Ltd
Dr Andries Bosland /Mr Jes Brown - Alcan International Ltd
Dr Craig Adam - Staffordshire University
Mr F L Cullen - AEA Technology
Mr Eric Kelly - 44 Penymynydd Rd
Dr J M Parker - Dept of Engineering Materials Sheffield University
Dr Mary Vickers - Cambridge University
Dr Mary Vickers - Cambridge University
Dr Mary Vickers - Cambridge University
Dr Tim Hyde - Johnson Matthey Technology Centre
Dr Robin Payne - Physical Sciences Group Zeneca
Mr Mike Edmondson - Research Dept Zeneca
Mr Mike Edmondson - Research Dept Zeneca
Mr Ian Tucker Sharon Evans - Unilever Research
Dr J A Jutson- BICC Cables Ltd
Mr T J Carter - AWE Aldermaston
Mrs J Blake- ICI C&P Ltd Runcorn
Dr David Dyson - British Steel Technical Rotherham
Mr David L Rush - Hepworth Refractories Ltd
Mr David J Taylor - Pilkington
Dr S V Norval- ICI Wilton
Ms Cerys Oliver - Cookson Technology Centre
Miss Sue Staniforth - GalaxoWellcome
Mr Colin Small - Rolls-Royce PLC
Mr P G Lake - Glaxo Wellcome
Dr Christian Lehmann - Chemistry University of Durham
Dr R Alan Howie - University of Aberdeen
Mr A Calder - Dept of Geology St Andrews University
Dr J Ian Langford - School of Physics Birmingham

APPENDIX 3

See Excel spreadsheet rrobapnd.xls after appendix 4

APPENDIX 4

Spreadsheet Calculations

Test1

c/s/mA - This is the background subtracted, dead time corrected, counts per second per milliamp.

Dead Time - This is the counter dead time calculated from the peak intensities at different mA settings. Note that the counting statistics are not ideal for the calculation. If your data looks odd you should recalculate with better statistics.

Pk/Bk - This is calculated from test 1b as (I-B)/B
where I = peak Intensity (cps) B = Ave Background (cps) . The background is averaged from 51.0 to 51.18 and 54.0 to 54.18 degrees.

Test2

Bk inc with kV - This is ((A-B)/B)/(C-D)
where A is Ave background/kV from 45 kV data
where B is Ave background/kV from 30 kV data
Where C is kV -8.86 for 45kV data
Where D is kV -8.86 for 30kV data

Pk/Bk (40kV) - This ((A-B)/40)/(B/40)
where A is peak intensity from 40 kV expt
where B is average background intensity from 40 kV expt.

Exponent Value - This is (I1-I2)/(k1-k2)
where I1 is log peak intensity from 45kV
where I2 is log peak intensity from 40kV
where k1 is log (45-8.86)
where k2 is log (40-8.86)

Test3
Int Ratio x/y this is the Peak-Bk intensity of slit x divided by the Peak-Bk of slit y

Test4
Pk Shift 0.01/0.02 - The alpha 1 peak intensity = T with the adjacent intensities on the low angle side = R and S and on the high angle side U and Vall in counts per second.
Pk Shift = A-B
where A = C-(D/E)*F for 0.02 step measurements
C is peak position as 2 theta angle
D is -0.2R-0.1S+0.1U+0.2V(1st derivative)
E is 0.285714R-0.142857S-0.285714T-0.14857U+0.285714V (2nd derivative)
F is step size
B = C-(D/E)*F for 0.01 step measurements
The other Pk Shift data is calculated as above but substituting the 0.04 and 0.08 for the 0.02 step data.

Test5
FOM Small -

$A1*\sqrt{((1/(A2/V)/(A1+4*B))}$
where A1 is alpha1 net peak intensity for small divergence slit
where A2 is alpha2 intensity
where V is valley intensity
where B is the average background intensity
FOM Mid - as above but using middle divergence slit data

Test6
Intensity gain Wo/ - c/s/mA without collimator divided by c/s/mA with collimator
Change Peak/Valley - (A/V)/(B/W)
where A is alpha2 intensity without collimator
where V is valley intensity without collimator
where B is alpha2 intensity with collimator
where W is valley intensity with collimator
Change Bk - average background without divided by average background with sec collimator

Test7
Improvement factor - $(A/B)/\sqrt{t}$
where t is 9 second count time
A is $(I*\sqrt{t}/\sqrt{C})$ for 9 second data
where I is net peak intensity
where t is count time
where C is average background
B is $(I*\sqrt{t}/\sqrt{C})$ for 1 second data

Test8
Peak Intensities were corrected :- Icor = I * sin(rad(A/2)) and scaled to give I/Imax*100
where A is the peak position as 2 theta angle and I is peak intensity.
Your relative intensities were then ratioed to the intensities obtained on the NIST instrument .
The **Average intensity** reported is the mean of all your individual test 8 relative intensity ratios and the **Standard deviation** is the 1 sigma value of this intensity data.

Appendix 3 & 5 to 10

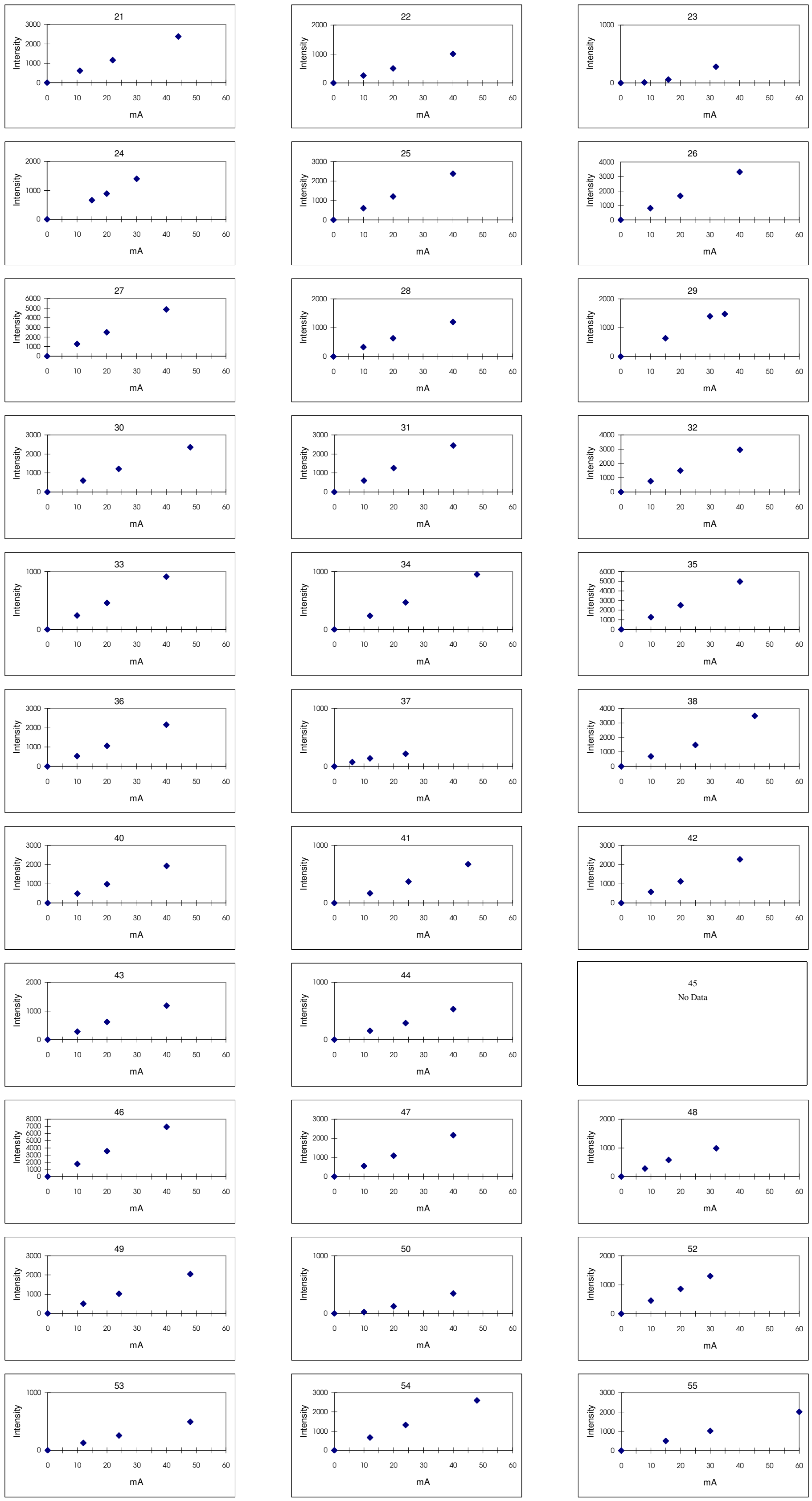
These pages are from the Excel spreadsheet rrobapnd.xls and follow on the next page of this PDF version.

ID	Manufact	Model	Anode	Filament	TakeOff	Radius	PB mono	SB moNone	Geometry	Div	Rec	Counter
21	hybrid	PW1050	Cu	Normal	6	Standard		graphite	T/2T	1	0.2	prop
22	Philips	PW1820	Cobalt	LFF	6	Standard		None	T/2T	1	0.1	prop
23	Siemens	D500	Cu	Normal	6	250	Quartz	None	T/2T	1	1	Scint
24	Philips	PW1050	Cu	Broad	6	Standard		graphite	T/2T	1	1	prop
25	Philips	X'pt PW3020	Cu	LFF	6	Standard		Graphite	T/2T	0.5	0.2	prop
26	Philips	PW1820	Cu	LFF	6	250		graphite	T/2T	1	0.2	prop
27	Philips	PW1050	Cu	LFF	6	Standard		graphite	T/2T	1	0.2	prop
28	Philips	X-Pert MPD	Cu	LFF	6	200		graphite	Theta/Theta	var	0.2	prop
29	Siemens	D500	Cobalt	FF	6	Standard		None	T/2T	1	0.05	Scint
30	Siemens	D500	Cu	LFF	6	Standard		graphite	T/2T	1	0.05	Scint
31	Philips	X-pert	Cu	LFF	6	Standard		graphite	T/2T	1	0.2	prop
32	Siemens	D500	Cu	Normal	6	250		graphite	T/2T	1	0.15	Scint
33	Siemens	D5000	Cu	LFF	6	200.5		graphite	Theta/Theta	var	0.2	Scint
34	Siemens	D500	Cu	LFF	6	Standard		graphite	T/2T	0.3	1	Scint
35	Siemens	D5000	Cu AEG	fk600412	6	414		None	T/2T	2	2	Scint
36	Philips	PW1800	Cu	LFF	6	Standard		graphite	T/2T	var	0.1	prop
37	Siemens	D500	Cobalt	LFF	6	Standard		graphite	T/2T	1	0.05	Scint
38	Philips	PW1050	Cu	LFF	6	Standard		graphite	T/2T	var	0.2	prop
39	*Philips*	*PW1050*	*Cu*	*Broad*	*6*	*Standard*		*graphite*	*T/2T*	*No DATA Supplied*		
40	Philips	PW1800	Cu	LFF	6	Standard		graphite	T/2T	var	0.1	prop
41	Siemens	D500	Cu	FF	6	Standard		graphite	T/2T	0.3	0.15	Scint
42	Philips	PW1050	Cu	LFF	6	Standard		graphite	T/2T	1	0.1	prop
43	Philips	PW1820	Cobalt	LFF	6	Standard		graphite	T/2T	var	0.1	prop
44	Siemens	D500	Cu	LFF	6	Standard		graphite	T/2T	0.3	0.15	Scint
45	Philips	PW1050	Cu	LFF	6	Standard		graphite	T/2T	0.5	0.2	prop
46	Phillips	1050mod	Cu	LFF	6	Standard		graphite	T/2T	1	0.2	prop
47	Philips	PW1050/80	Cu	Normal	6	Standard		None	T/2T	1	0.1	prop
48	Siemens	D500	Cu	LFF	6	410		graphite	T/2T	1	0.05	Scint
49	Philips	PW3040	Cu	LFF	6	250		graphite	T/2T	var	0.2	prop
50	Picker	2-Circle	Cu	LFF	3	145.5	Ge 111	None	T/2T	2	0.05	scint
52	Philips	PW1050	Cu	LFF	6	Standard		graphite	T/2T	1	0.2	prop
53	Siemens	D5000	Cu	LFF	6	Standard		graphite	T/2T	1	0.2	Scint
54	Siemens	D5000	Cu	LFF	6	Standard		ED -Det	T/2T	var	0.2	ED Kevex
55	Philips	PW1050/81	Cu	Broad	6	Standard		graphite	T/2T	1	0.2	prop

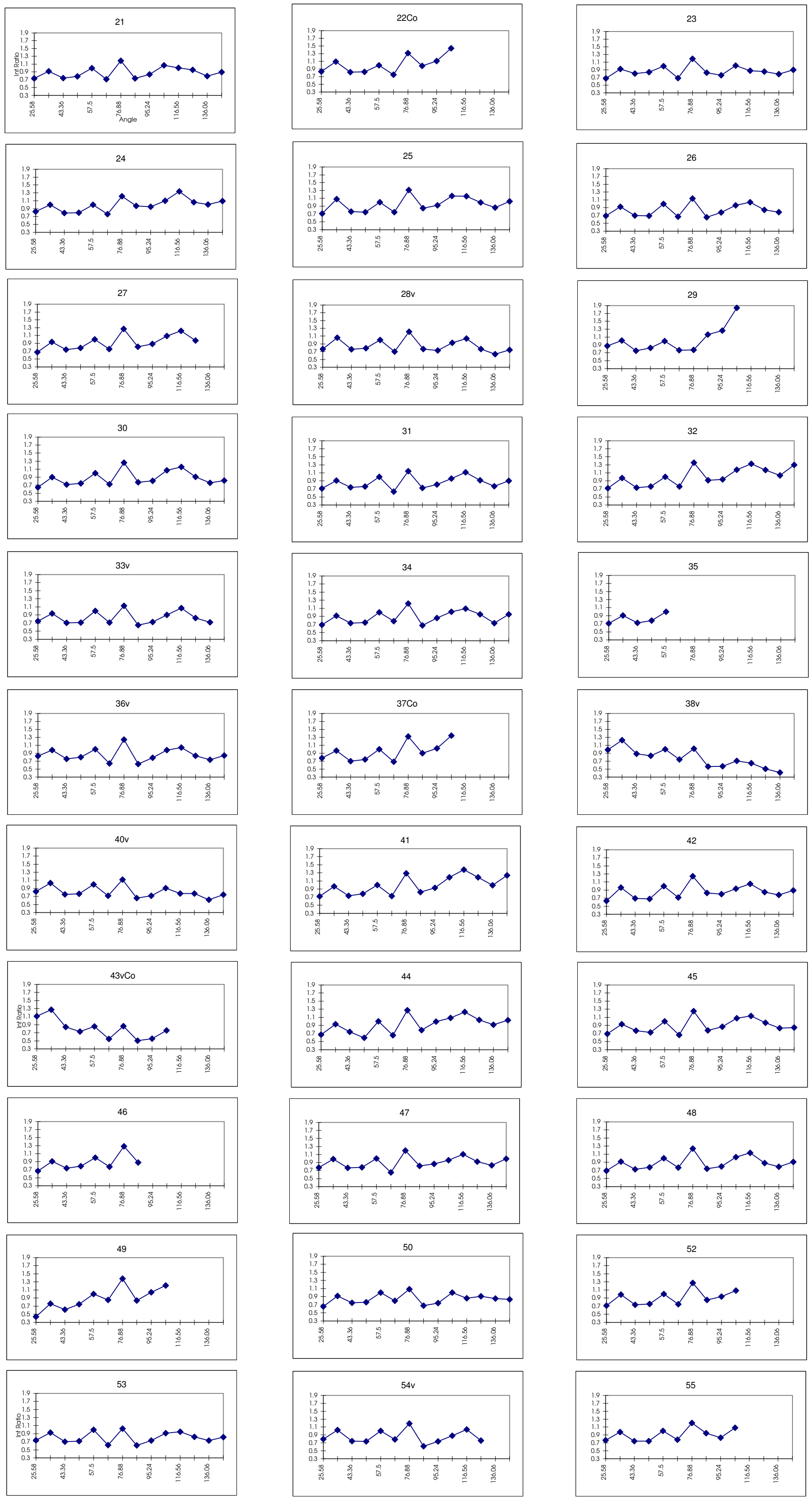
UK Intensity Round Robin
Data Summary

Appendix 5 ID	Type	Test #1 c/s	#1 DT	#1 Pk/Bg	#2 Bg/kV	#2 Pk?bg	#2 Exp	#3 med/sm	#3 lg/sm	#4 1/2	#4 1/4	#4 1/8	#5 med/sm	#5 lg/sm	#6 Wo/W	#6 P/V	#6 P/B	#7 IF	#8 Av	#8 SD
21	Philips	58	22	58	1.32	64	0.83	1.60	2.51	0.0019	0.0012	0.0386	12.50	21.05	No Data	No Data	No Data	1.00	0.88	0.14
22	Philips Co	26	15	27	7.28	37	1.03	2.04	0.00	-0.0167	-0.0184	-0.0212	No Data	No Data	No Data	No Data	No Data	1.37	1.02	0.23
24	Philips	42	Not Detm	47	1.84	47	0.97	1.90	2.78	0.0071	0.0296	0.0407	18.32	26.62	No Data	No Data	No Data	1.06	0.99	0.17
25	Philips	62	11	67	1.53	65	1.01	2.45	5.62	0.0011	0.0044	0.0338	21.10	34.62	No Data	No Data	No Data	1.01	0.95	0.18
26	Philips	82	Not Detm	69	1.19	68	1.03	1.73	2.36	-0.0017	-0.0068	0.0356	14.32	28.58	1.78	0.75	2.00	0.91	0.84	0.16
27	Philips/hybrid	109	11	61	1.58	59	1.20	1.70	2.23	0.0075	-0.0053	-0.0393	No data	32.49	No Data	No Data	No Data	1.06	0.93	0.19
28	Philips	34	Not Detm	82	3.07	73	1.42	1.85	4.84	0.0077	0.0186	0.0428	No data	No data	1.95	0.88	2.24	1.02	0.85	0.17
31	Philips	63	5	69	1.71	67	1.17	1.69	2.44	0.0011	0.0270	0.0496	13.70	25.94	1.80	0.83	1.96	0.95	0.86	0.15
36	Philips	53	Not Detm	83	1.07	87	1.19	2.03	No Data	-0.0008	0.0009	0.0342	No Data	No Data	No Data	No Data	No Data	0.96	0.86	0.17
38	Philips	57	Not Detm	49	1.24	62	0.86	1.75	2.22	-0.0092	-0.0158	0.0149	No Data	No Data	No Data	No Data	No Data	0.93	0.78	0.24
40	Philips	50	7	84	3.31	80	1.09	2.00	No Data	-0.0023	-0.0020	0.0523	No Data	No Data	No Data	No Data	No Data	1.01	0.81	0.15
42	Philips	58	10	67	1.17	66	1.35	1.69	3.03	-0.0032	0.0037	0.0316	15.97	23.42	No Data	No data	No Data	1.14	0.86	0.17
43	Philips Co	30	6	107	0.41	110	0.61	1.69	2.09	-0.0028	-0.0086	0.0337	No Data	No Data	No Data	No Data	No Data	1.04	0.80	0.25
45	Philips	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	No Data	0.90	0.18
46	Philips	181	5	69	1.26	68	0.90	1.57	2.03	-0.0025	-0.0103	0.0155	31.69	45.41	No Data	No Data	No Data	0.98	0.88	0.19
47	Philips	57	13	32	5.39	38	1.14	1.86	2.46	0.0061	0.0148	0.0370	14.87	20.44	No Data	No Data	No Data	1.06	0.90	0.15
49	Philips	43	Not Detm	87	1.64	94	0.88	1.60	3.84	-0.0003	0.0011	0.0278	No Data	No Data	No Data	No Data	No Data	1.08	0.89	0.28
52	Philips	45	<10	47	No Data	48	No Data	1.85	2.65	0.0066	-0.0122	0.0002	15.29	21.67	No Data	No Data	No Data	0.98	0.91	0.19
55	Philips	34	8	62	1.50	64	1.26	2.39	2.44	-0.0100	-0.0074	0.0224	15.69	20.78	1.92	0.88	2.19	1.02	0.91	0.16
30	Siemens	52	14	69	1.39	73	0.83	3.12	5.04	0.0020	-0.0088	0.0327	22.12	28.67	2.90	0.65	4.08	1.07	0.88	0.18
48	Siemens	40	Not Detm	75	2.69	76	1.82	2.93	4.23	0.0005	-0.0061	0.0363	13.90	24.71	No Data	No Data	No Data	0.99	0.89	0.16
54	Siemens	57	17	81	1.45	90	0.73	1.93	5.43	0.0092	0.0207	0.0470	9.10	17.29	2.76	0.61	3.23	1.09	0.86	0.17
35	Siemens	136	4	14	0.87	15	1.18	1.97	5.82	-0.0028	0.0313	0.0256	25.73	32.30	No soller	used in all	tests	No Data	0.82	0.13
33	Siemens	24	Not Detm	64	2.47	72	0.68	1.89	5.19	0.0007	0.0072	0.1721	9.23	12.63	2.89	0.76	1.55	0.92	0.84	0.16
44	Siemens	12	Not Detm	51	0.17	56	1.44	8.39	11.68	-0.0006	0.0115	0.0330	19.00	26.33	2.57	0.91	3.21	0.89	0.93	0.21
32	Siemens	80	14	35	1.05	34	0.95	3.36	5.48	0.0001	0.0106	0.0208	15.61	28.08	No Data	No Data	No Data	0.96	1.01	0.23
41	Siemens	15	Not Detm	45	2.03	48	1.03	2.54	3.88	-0.0045	-0.0031	0.0343	2.88	21.44	No Data	No Data	No Data	0.88	1.10	0.43
34	Siemens	20	Not Detm	73	1.79	74	0.69	2.27	3.46	0.0000	-0.0074	0.0343	9.57	17.48	No Data	No Data	No Data	0.98	0.88	0.16
29	Siemens Co	80	Not Detm	30	3.25	32	1.10	0.99	0.00	-0.0023	-0.0017	0.0352	No Data	No Data	No Data	No Data	No Data	1.13	1.03	0.33
37	Siemens Co	17	Not Detm	15	0.51	82	1.44	2.74	5.39	-0.0004	0.0095	0.0362	3.23	5.19	No Data	No Data	No Data	1.07	0.95	0.24
23	Siemens pbm	2	Not Detm	99	6.24	92	2.83	2.81	13.49	0.0014	0.0008	0.0030	No Alpha2	No Alpha2	No Data	No Data	No Data	0.87	0.87	0.14
53	Siemens	11	Not Detm	78	0.67	66	1.09	1.83	4.54	-0.0002	0.0008	0.0434	7.71	9.14	2.76	0.72	2.66	0.63	0.81	0.14
50	Picker pbm	4	Not Detm	75	-1.73	59	1.35	5.39	9.37	-0.0060	-0.0100	0.0120	No Alpha2	No Alpha2	4.22	No Alpha2	6.20	0.82	0.85	0.13
Average	**Philips UK**	**60**	**10**	**65**	**2.15**	**67**	**1.06**	**1.86**	**2.72**	**-0.0006**	**0.0008**	**0.0250**	**17.34**	**27.37**	**1.86**	**0.84**	**2.10**	**1.03**	**0.89**	**0.18**
Average	**Philips USA**	**148**	**13**	**72**	**1.57**	**62**	**1.03**	**1.93**	**2.61**	**-0.0004**	**0.0023**	**0.0447**	**20.65**	**32.20**	**1.68**	**0.89**	**1.69**	**1.01**	**0.94**	**0.11**
Average	**Siemens UK**	**42**	**12**	**56**	**1.89**	**62**	**1.22**	**2.83**	**5.66**	**0.0002**	**0.0050**	**0.0426**	**12.55**	**20.30**	**2.78**	**0.73**	**2.95**	**0.96**	**0.91**	**0.21**
Average	**Siemens USA**	**56**	**7**	**72**	**2.00**	**72**	**1.26**	**1.67**	**2.99**	**0.0020**	**0.0049**	**0.0381**	**10.51**	**18.35**	**3.20**	**0.99**	**4.08**	**0.94**	**1.05**	**0.14**

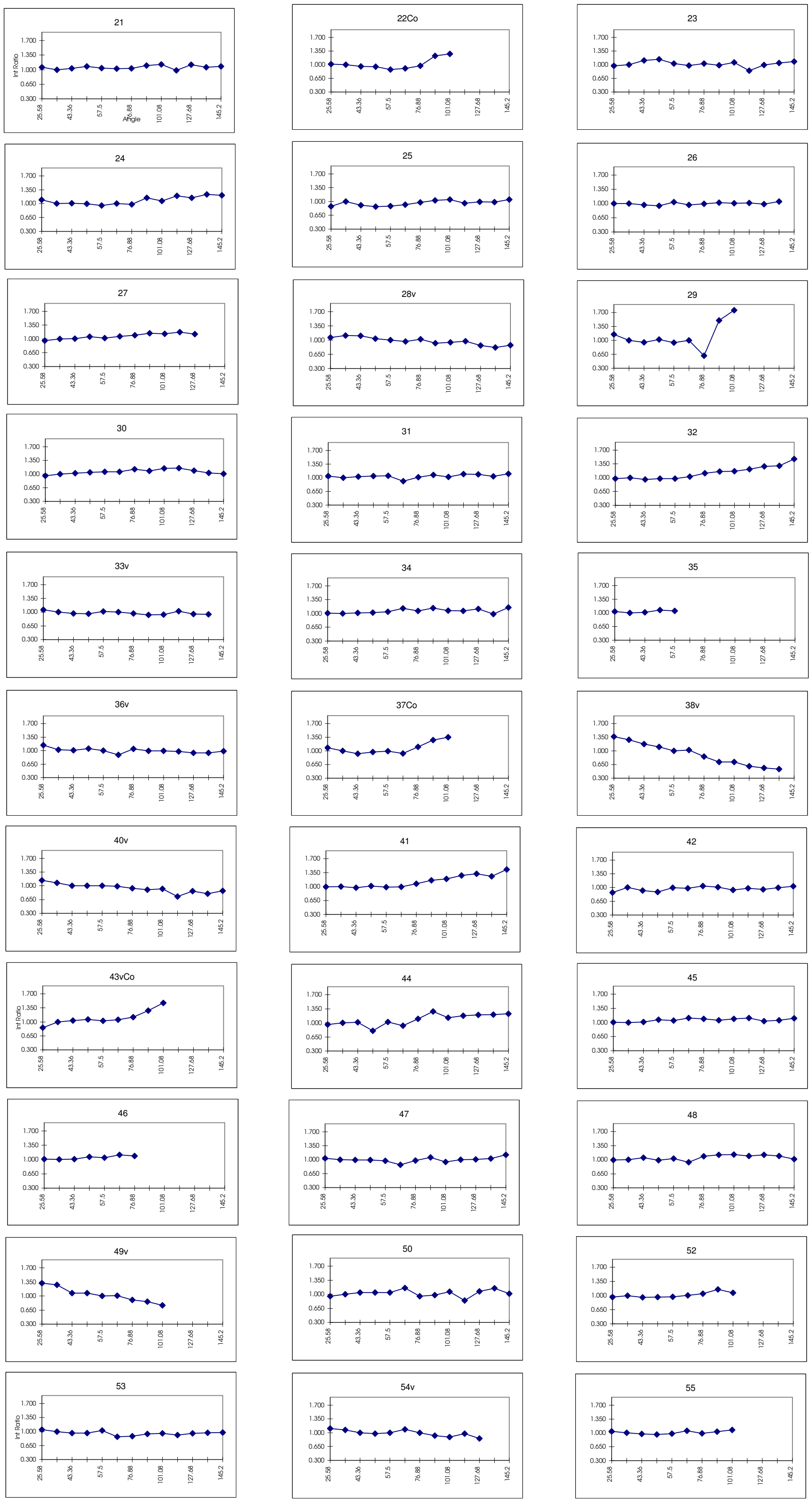
Test 1 Intensities



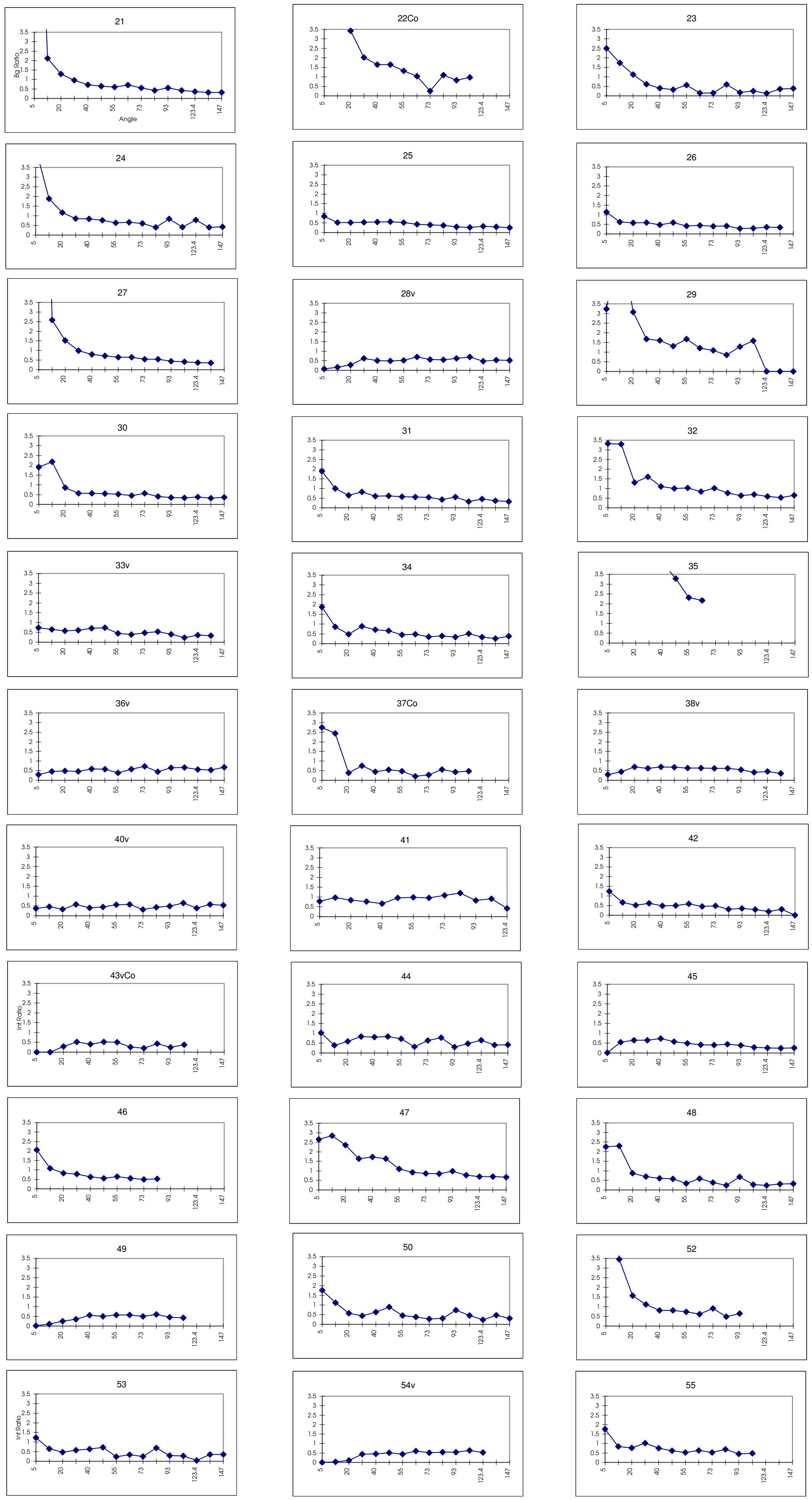
APPENDIX 7 Intensity Ratio



Intensity Ratio (Modified)



Background Ratio



APPENDIX 10 Angular Displacement