British Crystallographic Association

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Industrial Group

UK Intensity Round Robin

A Guide to self testing your Diffractometer

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Introduction

An Intensity Round Robin was completed in the UK in 1997. It covered Philips and Siemens (now Bruker) diffractometers and generated a useful resource for the evaluation of instrument performance. To make this data widely available so that anyone could test their own machine a series of self test files were produced. This guide explains how to use these files to run the tests in your own laboratory.

Equipment required

A diffractometer to test.
A NIST SRM 1976 alumina plate standard to fit the instrument.
A computer running Word 6 or later, Excel 5 or later and the ability to obtain files by ftp (and unzip them) or e-mail capable of receiving attached .doc and .xls files.

Self test files

File	size	Use
•README.TXT	3kb	explains tests
•RRint.PDF	224kb	UK Round Robin report
•RROBCUTP.XLS	153kb	Cu tube Data Template
•RROBCOTP.XLS	152kb	Co tube Data Template
•UKDEMO.XLS	154kb	A completed data example

Available by download or as e-mail attachments from djtaylor@lineone.net

Running the tests

Print a copy of RRREPT.DOC and RROBAPND.XLS and combine the output to assemble the UK Round Robin report.
Follow the instructions in Appendix 1 of the report to run the tests. If time is limited then at least run test 1c and test 8.

Data Extraction

The data is extracted from a raw data file listing. The extracted data is typed into the input sheet of the Excel file appropriate for your X-ray tube (e.g. RROBCUTP.XLS). It is useful to print a copy of the INPUT sheet of this file and use it as a form to fill in the acquired data.

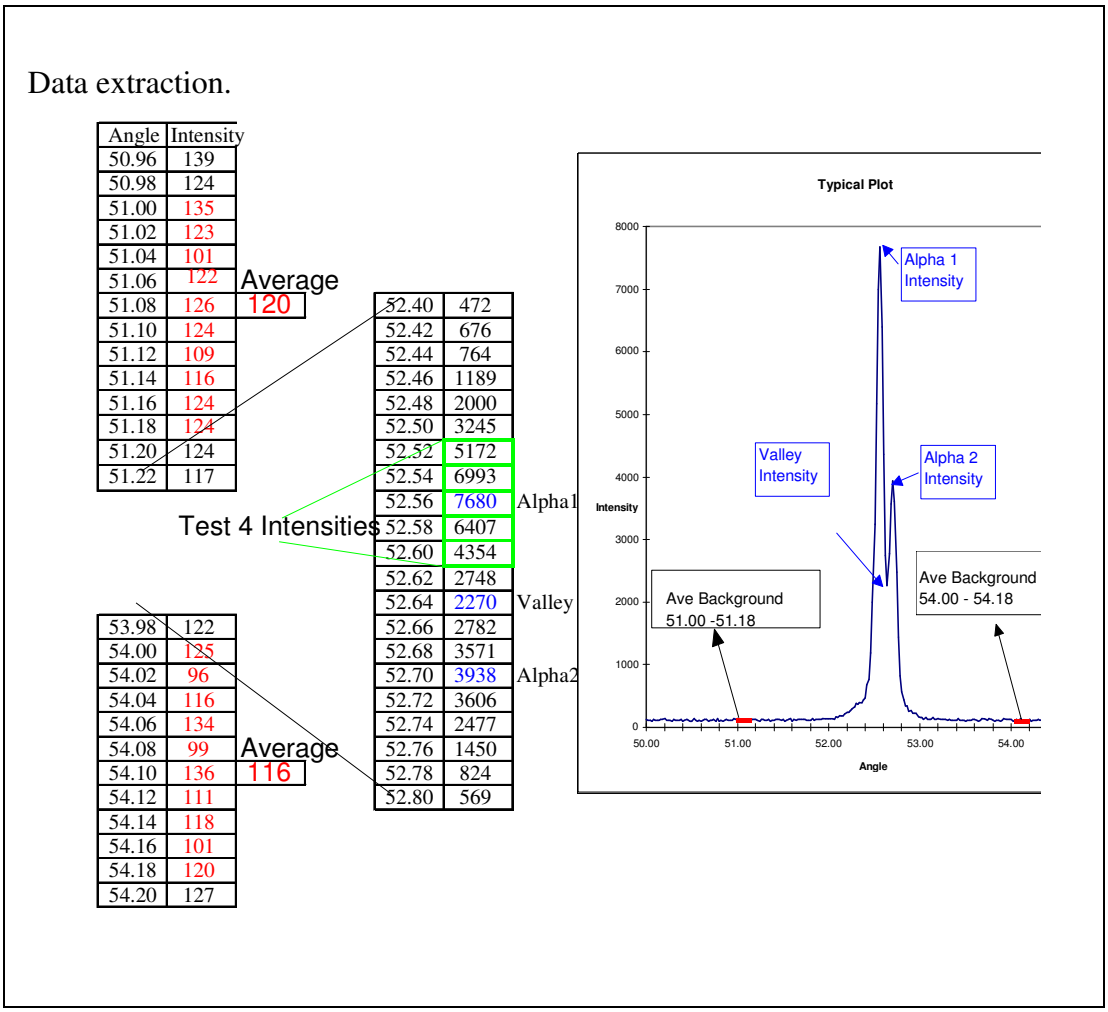

Table 1.

Table 1 shows parts of a typical result from Test 1 to 6. The text panels show the information you need to extract. Take the average backgrounds of the data from 51.00° to 51.18° and 54.00° to 54.18°. Find the maximum intensity at about 52.5° and record its angular position and intensity value. In table 1 these are 52.56° and 7680 counts. Next find the minimum intensity in the valley between the alpha1 and alpha 2 peaks, in this example the value is 2270 counts. The maximum intensity of the alpha 2 peak is also recorded, in this case it is 3938 counts.
This procedure is repeated for each test and the values obtained are entered into the INPUT sheet of the appropriate Excel spreadsheet (RROBCUTP.XLS or RROBCOTP.XLS) For test 1c you also need to calculate the full width at half height (FWHM) of the alpha 1 peak. You can do this manually or more accurately by profile fitting. In the example in table 1 the peak height is 7680 with an average background of 118 giving a net height of 7562 counts. The half height of 3781 is between 52.50° (3245counts) and 52.52°(5172counts). Assuming a linear slope, the 0.02° step covers 1827 counts (5172-3245=1827) and 3781 counts would be at **52.5059°** (3781-3245=536, 536/1827*0.02+52.50). Similarly the half height at the high angle side is between 52.60° (4354) and 52.62°(2748). So 3781-2748=1023, 1023/(4354-2748)*0.02+52.60= **52.6127°** and the **FWHM** = 52.6127-52.5059 = **0.1068°**

For Test 4 the procedure is different. First find the maximum alpha 1 intensity, record its angular position and then record the five intensity values that span the peak. These are entered across the spreadsheet with the lowest angle first. In this example they are, 5172, 6993, 7680, 6407, 4354.

Test 7 is a weak peak at about 41.7 degrees and differs in that only one background is averaged from 41.00 to 41.18 degrees.

Test 8 is a long scan and the individual background counts are recorded at the intervals specified in the INPUT sheet. Also the angular position and intensities of the specified peaks are recorded. You should also check the scan for any spurious peaks that may be due to tube lines or other instrument artefacts.

Entering the data.

Round Robin Data Input Copper Tube Type your data in the empty white cells

User ID	Name	,Company	Standard	Instrument	FWHM Test1c	0.09					
31	ANON			Philips Expert							
				Test Param	Alpha 1 Angle	Intensity Alpha1	Intensity Valley	Intensity Alpha2	Bkg sum 41.00>.18	Bkg sum 54.00>.18
kV	45	1a	mA	10	52.56	2421	697	1340	335	354
RS	0.2	1b		20	52.56	5084	1391	2570	693	768
CT	4	1c		40	52.56	9900	2830	5285	1447	1459
mA	20	2a	kV	30	52.56	2621	734	1347	385	372
RS	0.2	2b		40	52.56	4212	1122	2209	628	611
CT	4	2c		45	52.56	5013	1429	2663	712	717
		2d		50	52.56	5806	1592	2970	838	876
kV	45	3a	RS	0.1	52.56	4998	1089	2591	555	667
mA	35	3b		0.2	52.54	8464	2411	4422	1226	1269
CT	4	3c		1.8	52.56	12321	6610	6642	2523	2479
					Peak Angl	Intensity	Intensity	Intensity	Intensity	Intensity
kV	45	4a	Step size	0.01	52.55	7850	8482	8724	8446	7482
mA	35	4b		0.02	52.56	6626	8372	8464	6209	3600
RS	0.2	4c		0.04	52.56	2490	6593	8391	3600	2663
CT	4	4d		0.06	52.54	620	2401	8538	3844	3376
		4e		0.08	52.52	306	912	6416	4020	4476
					Angle	Alpha1	Valley	Alpha2	51.00>.18	54.00>.18
kV	45	5a	Div slit	0.25	52.56	2642	801	1391	326	366
mA	35	5b		0.50	52.56	4984	1490	2570	721	701
CT	4	5c		1.0	52.54	8486	2460	4396	1241	1254
		6	No	Soller	52.54	15575	5184	8028	2471	2492
		7a	Time	1	41.68	310	139	166	313	41.00
		7b		9	41.68	2673	1267	1513	2956	>0.18
Bkg angle	1.0	2.0	5.0	10.0	20.0	30.0	40.0	48.0	55.0	63.0
Bkg ht rav	1968690	17983	328	154	92	110	62	62	67	64
Bkg angle	73.0	82.0	93.0	107.0	123.4	134.0	147.0	158.0		
Bkg ht rav	59	41	52	32	59	59	61	53		
Theor ar	25.58	35.16	43.36	52.56	57.5	68.2	76.88	84.36		
Meas. ang	25.58	35.16	43.36	52.56	57.5	68.22	76.86	84.36		
Peak Ht	5761	16641	8556	4382	14593	2560	5806	310		
Theor ar	95.24	101.08	116.56	127.68	136.06	145.2				
Meas. ang	95.24	101.08	116.59	127.68	136.08	145.16				
Peak Ht	1289	1858	1840	1362	1129	745				

Table2

The data is entered into the spaces in the INPUT sheet. A completed example is shown in table 2. This sheet has links to other sheets in the workbook and reports and plots are automatically generated.

Results.

Your spreadsheet results can be compared to those in the UK report as follows:-

Sheet	**Report**	**Test**
UKSumry	Appendix 5	All
Bkg	Appendix 9	8
FOM plot	Page 8	1c
Displacement	Appendix 10	8
Intensity(Fixed Slit)*	Appendix 8	8
Intensity(Variable Slit)*	Appendix 8	8

* Use the one appropriate for your system!

A REPORT sheet is also produced and a printout may be useful in comparing data directly with the UK report. Some averages for other manufacturers from the USA tests are added in the extended report shown below in Table 3. The result from Test 8 is split into Fixed and Variable slit values and the appropriate value for your set up should be used.

ICDD INTENSITY ROUND ROBIN - UK Preliminary Test results

User: ANON
User ID: 31 Instrument Type: Philips Expert

					Mean Philips		Mean Siemens					
	Test		**User 31**		**USA**	**UK**	**USA**	**UK**	**Nicolet USA**	**Scintag USA**	**Rigaku USA**	**Dron USA**
Test1	Tube Current	c/s/mA	**63**		148	60	56	42	0	47	41	41
		Dead Time	**5**		13	10	7	12	0	21	17	0
		Pk/Bk	**69**		72	65	72	56	24	74	73	32
Test2	Tube Voltage	Bk incr with kV	**1.72**		1.57	2.15	2	1.89	0	0.94	1.63	3.92
		Pk/Bk (40kV)	**67**		62	67	72	62	34	78	75	45
		Exponent Value	**1.17**		1.03	1.06	1.28	1.22	1.48	0.89	1.05	0.74
Test3	Receiving Slit	Int Ratio0.20/0.10	**1.69**		1.93	1.86	1.67	2.83	2.94	2.53	1.47	3.33
		Int Ratio1.80/0.10	**2.45**		2.61	2.72	2.99	5.66	0	2.07	3.01	0
Test4	Step Size	Pk Shift 0.01/0.02	**0.0011**		-0.0004	-0.0006	0.0020	0.0002	-0.0070	-0.0006	-0.0028	0.0100
		Pk Shift 0.01/0.04	**0.0070**		0.0023	0.0008	0.0049	0.0050	-0.0100	0.0071	0.0037	0.0010
		Pk Shift 0.01/0.08	**0.0496**		0.0447	0.0250	0.0381	0.0426	0.0300	0.0426	0.0318	0.0430
Test5	Divergence Slit	FOM Small	**18.88**		20.65	17.34	10.51	12.55	0	13.27	21.83	12.1
		FOM Mid	**25.9**		32.2	27.37	18.35	20.3	0	18	29.07	17.1
Test6	Sec Collimator	Intensity gain Wo/W	**1.80**		1.68	1.86	3.20	2.78	0	2.51		2.62
		Change Peak/Valley	**0.83**		0.89	0.84	0.99	0.73	0	0.74		0.77
		Change Bk	**1.95**		1.69	2.1	4.08	2.95	0	1.38		3.2
Test7	Count Time	Improvement factor	**0.95**		1.01	1.03	0.94	0.96	0.97	1.03	0.98	0
			Fixed slit	Var slit								
Test8	Sensitivity	Average Intensity	**0.97**	**0.78**	0.94	1.015	1.05	1.051	1	0.95	0.98	0.97
		Standard Deviation	**0.08**	**0.42**	0.11	0.107	0.14	0.092	0.13	0.09	0.1	0.05

Table 3.

Comparing Results.

Your results can be compared with those in table 3 above and the plots against the UK report. The intensity plot from Test 8 should not show any pattern and no point should deviate above 1.0785 or below 0.9215 if your instrument is in control. In the FOM plot the higher the score the better. The value of the dead time can only be used as a guide and if doubted should be measured with improved counting statistics.

Conclusions.

A method to test the intensity of a diffractometer has been described. It is based on the UK tests in an International Round Robin Exercise. It has been established in the belief that allowing users to test their own systems will help them identify and correct potential sources of error in their data.