

SADABS -- Instructions --- Beginner

Notes and user input are in red and italicized.

SADABS-2006/1 - Bruker AXS area detector scaling and absorption correction

Answer N for Expert mode

Expert mode (Y or N) [N]:

Change the listing file name

Enter listing filename [sad.abs]: *KBABS.TXT*

Laue group numbers:

[1] -1	[8] -3m (rhombohedral axes)
[2] 2/m (Y unique)	[9] -31m (Z unique)
[3] mmm	[10] -3m1 (Z unique)
[4] 4/m (Z unique)	[11] 6/m (Z unique)
[5] 4/mmm (Z unique)	[12] 6/mmm (Z unique)
[6] -3 (rhombohedral axes)	[13] m3
[7] -3 (Z unique)	[14] m3m

*The unit cell 12.20440 17.67740 15.67050 90.0000 91.4210 90.0000
This is Laue Class 2/m*

	<i>a</i>	<i>b</i>	<i>c</i>	α	β	γ	Laue		
Triclinic							-1		
Monoclinic				90		90	2/m		
Orthorhombic				90	90	90	mmm		
Tetragonal	a=b	a=b		90	90	90	4/m	4/mmm	
Trigonal	a=b	a=b		90	90	120	-3	-3m1	-31m
Hexagonal	a=b	a=b		90	90	120	6/m	6/mmm	
Cubic	a=b=c	a=b=c	a=b=c	90	90	90	m-3	m-3m	

[0] to write list of equivalent indices for Laue/point groups to listing file

Enter Laue group number [2]: *2*

Read reflection files written by EVALCCD (with extension .sad specified) or by SAINT (extension .raw, default if no extension, or .ram for incommensurate structures). Either individual files for each scan or a single merged file may be read. It is important that all files are from the same crystal and that reflections have been indexed consistently, i.e. that the orientation matrices are similar (no rows with signs reversed)! Note that XPREP can re-index a .raw or .sad file transforming the direction cosines

Enter filename (/ if no more) []: *datam.raw*

I have entered the merged file generated directly from SAINT. You may want to input the files one by one i.e. data1.raw, data2.raw etc.

Mean and maximum errors in direction cosine check function = 0.001 0.005 The mean error should not exceed 0.005, and is usually caused by matrix changes during data processing.

If the mean error is greater than 0.005 then you should run saint again with the datam.p4p file.

Approximate wavelength, cell and maximum 2-theta (from cosines etc.):

0.71108 12.233 17.707 15.705 90.006 91.533 90.032 55.22

This should match your cell and radiation (0.71 or 1.54Å)

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PART 1 - Refinement of parameters to model systematic errors

Thresholds should now be specified for excluding reflections from the parameter refinement; these reflections may still be corrected and included in the final output .hkl file

49162 Reflections of which 8087 unique; 16.29 data per frame

Redundancy:	1	2	3	4	5	6	7	8	9+
Number of groups:	56	158	462	998	1584	1951	905	800	1173

Mean(I/sigma):	-inf	0	1	2	3	5	10	15	20	+inf
Number of groups:	383	428	416	454	411	2130	1379	900	1586	

If -inf is much larger than the rest of the numbers your data set is very bad.

The following restraint esd could be increased for strong absorbers.

Restraint esd for equal consecutive scale factors [0.005]: *0.005*

Increase this number for third row (main) and second row transition elements.

Number of refinement cycles [15]: 15

15 is normally ok. Use 20 or more for poorly converging L.S.

Weak (W), moderate (M) or strong (S) empirical absorption correction or face-indexed numerical (N) absorption correction [M]: W

Use M for moderate, S for strong and N for face-indexed corrections. If you choose N then you MUST have the FACE commands in the P4P file!

42746 Reflections employed for parameter determination

Effective data to parameter ratio = 11.72

R(int) = 0.1042 (selected reflections only, before parameter refinement)

Cycle R(incid) R(diffr) Mean wt.

1	0.0786	0.0609	0.9132
2	0.0569	0.0551	0.9306
3	0.0542	0.0538	0.9326
4	0.0533	0.0533	0.9333
5	0.0530	0.0531	0.9336
6	0.0527	0.0529	0.9338
7	0.0526	0.0528	0.9340
8	0.0525	0.0527	0.9340
9	0.0525	0.0527	0.9341
10	0.0524	0.0527	0.9341
11	0.0524	0.0526	0.9341
12	0.0524	0.0526	0.9341
13	0.0524	0.0526	0.9342
14	0.0524	0.0526	0.9342
15	0.0523	0.0526	0.9342

R(int) = 0.0526 (selected reflections only, after parameter refinement)

Refinement has converged. If the you do not see convergence then repeat refinement with more cycles until you do!

Repeat parameter refinement (R) or accept (A) [A]: A

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PART 2 - Reject outliers and establish error model

Rejected reflections are ignored in the statistics and Postscript plots (except the detector diagnostics) and in the output .hkl file.

Before applying rejections there are:

49162 total and 8087 unique reflections assuming Friedel's law.

48883 total and 8087 unique reflections left after $|I - \langle I \rangle|/su$ test

$g = 0.1401$ gives best error model.

This value will be about the value you see on the WGHT line in SHELX.

Enter new value for g or $\langle CR \rangle$ to accept:

Run	2-theta	R(int)	Incid. factors	Diffr. factors	K	Total	I>2sig(I)
1	-28.0	0.0529	0.807 - 0.994	0.877 - 1.309	0.693	9799	9282
2	-28.0	0.0579	0.768 - 0.931	0.908 - 1.322	0.761	9714	9079
3	-28.0	0.0547	0.759 - 1.086	0.877 - 1.336	0.708	9750	9170
4	-28.0	0.0520	0.870 - 1.123	0.908 - 1.286	0.661	9833	9276
5	-28.0	0.0526	0.849 - 1.122	0.887 - 1.333	0.668	9787	9232

The R(int) is important. It should be small less than 0.2 for OK and 0.1 for good data. All of the Ks and the I > 2 sig(I)s should be more or less equal

$su = K * \text{Sqrt}[\text{sigma}^2(I) + (g\langle I \rangle)^2]$ where $\text{sigma}(I)$ is estimated by SAINT

The above statistics are based on all non-rejected data, ignoring reflections without equivalents when estimating R(int) and K.

Repeat parameter refinement (P) or accept (A) [A]: **A**

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PART 3 - Output Postscript diagnostics and corrected data

Write Postscript diagnostic file (Y or N) [Y]: **N**

At this stage you can write a postscript file to exam for errors. If SADABS is doing OK this file is not necessary.

Repeat (R), write unmerged .hkl (W), merged .hkl (M), .sca (S), XD format (D), testxtl.dat (BioXhit) (T) or quit (Q) [W]: **W**

Reflection output file [sad.hkl]: **KB.hkl**

Write the hkl file that you will input to XPREP. This file does not contain directional cosines and is merged.

Lambda/2 correction factor (0 if no monochromator [0.0015]): **0.0015**

This corrects for the $n\lambda$ where $n=2$ and not 1. This will only help! Keep it!

48883 Corrected reflections written to file KB.HKL

Estimated minimum and maximum transmission: 0.3207 0.7453

These numbers can be directly input into the final CIF.

The ratio of these values is more reliable than their absolute values!

Repeat (R), write unmerged .hkl (W), merged .hkl (M), .sca (S), XD format (D),
testxtl.dat (BioXhit) (T) or quit (Q) [Q]: *Q*

At this point you can output an unmerged file for absolute configuration.