

Using ANA to correct data taken on ID3

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These are some quick notes on the settings to be used in ANA when analysing data collected on the ID3 beamline at the ESRF. These notes will hopefully save some time, but are no substitute for knowing what you are doing - in particular you should consult the anarod documentation [1], or the source code.

As far as I know the settings are correct, but you use them at

your own risk. If you find any errors please let me know.

Eventually the information included in this document should be included in the ANA manual, but until then, the latest version can be found at <http://www.chiark.greenend.org.uk/~chrisw/science/anarod/ana-tips.pdf>.

1 Initial setup

1.1 set read

Item	ID3 Value	9.4 value	Comment
filename	Filename	basename	Filename of data file
mcol	$\neq 0$	3	to normalise to the monitor counts (if 0, counts are not normalised to the monitor). ANA will automatically pick the column titled monitor (you see the value change after reading in a spec scan).
xcol	1	1	Column number of “X” in data file - usually 1 as spec puts the scanned motor in this column, but may be different for an hklscan along l for example.
norm	arbitrary	arbitrary	Monitor counts per second halfway through the refill is a good choice, and means that the counts bear some relationship to real counts ($Correctedcounts = Counts \times \frac{M_{norm}}{Monitor}$)
Yname	<i>det_corr</i>	detector	name of detector column
Ycol	-	5	Not needed for id3 as it is calculated automatically from the <i>Yname</i> . Not sure about 9.4
Solid	No	Yes	Solid state detector at 9.4 always records 1 count - subtract 1 from detector counts to correct for this
<i>type of file</i>	id03	srs ¹	set type of file to read in

1.2 set cor

Item	ID3		9.4	Comment
	EH1	EH2		
“geometric”	zaxis	zaxis	zaxis	Zaxis geometry - see discussion below
lingamma	No	Yes	No	Yes on EH2 diffractometer, no on EH1 diffractometer
Hpolar	0	1	0.95	In EH1, the diffractometer is rotated 90 degrees from the normal geometry and puts the polarisation in the plane of the sample. The beam on 9.4 is not perfectly horizontally polarised, hence the value of 0.95
“scan type”				
rscan	θ scan		ϕ scan	rotating about the sample normal ² .

¹Apparently still says file of id03 type - but as you read data using *read srs*, this isn't a problem

²On 9.4 ϕ is approximately the sample normal.

Item	ID3		9.4	Comment
	EH1	EH2		
refrock sprdscan hklscan	ϕ or χ rocking on specular	? hklscan		specular rocking scans specular rod scan diffraction index (reciprocal space) scan ³
Slit settings ⁴				
hslit vslit dslit hbeam	pssvg psshg manual detsl pssv _{FWHM}	pssg ssg manual detsl pssp _{FWHM}	EOBHOR EOBVER 2.4mm	no vertical pre sample slits in EH2 post sample slits in delta direction. These set the sample area. pssvg and pssg respectively closed when performing scan. Daresbury figures from http://srs.dl.ac.uk/station/9.4/beamline/profile.htm
vbeam	pssh _{FWHM}	ssp _{FWHM} ???	0.6mm	
radius				sample radius (circular samples)
beamcor	yes	yes		Use numerical beam correction - see notes.
gammafix	0	0	0/15	Angle of manual gamma arm (9.4 only)
oslit	dsl	dsl		Oslit only used in hzsixc and vzsixc geometry - not in zaxis geometry. detector slits giving l resolution.

Zaxis mode should be used for the diffractometers in EH1 and EH2 of beamline ID3 at the ESRF.

The classic six circle diffractometer has the sample normal horizontal, so the scattering plane of “inplane” peaks is mainly vertical. This fits the narrow divergence of the beam vertically where peaks are sharp, and the more diffuse nature of the peaks in l . The diffractometer in EH2 of ID3 is such a diffractometer. This has shaped the naming of some of the slits etc - eg hpolar the horizontal polarisation in fact is the amount of polarization parallel to the sample normal.

The large UHV diffractometer in EH2 has a linear gamma stage, which adds an extra $\cos\gamma$ term to the correction factor, but is otherwise standard.

The diffractometer in EH1 has the same six circle zaxis geometry as that of EH2, but is rotated by 90° and has a “proper” gamma circle. This results in a sample normal that is vertical. This has various advantages in fitting heavy chambers, or studying liquid layers on surfaces.

Correction factors are therefore best done by considering this as rotating the plane of polarisation of the light by 90° and think-

³Doesn't apply area correction factor for each data point, so only valid for scans where this doesn't change significantly

⁴These are explained in more detail in “from scans to structure factors”

ing of *HPOLAR* as the fraction of light polarized parallel to the sample normal.

It is also possible to use the *hzsixc* (EH1 our “vertical zaxis” diffractometer) and *vzsixc* (EH2) corrections - the only difference seems to be the use of analytic area correction parameters instead of numerical but with no possibility of taking beam profile into account. Secondly there is the addition of an incorrect $1/\beta_{in}$ factor - though this is not an issue for data taken with the same incident angle throughout.

Station 9.4 at Daresbury, is a 5 circle zaxis diffractometer with the sample normal horizontal. The geometry is similar to the big UHV diffractometer in EH2 at ID3. 9.4 however has an additional gamma arm that can be moved by hand between two positions, this gives it the ability to go significantly further out in l , than would otherwise be the case.

1.2.1 slit settings

More detail on slit settings can be found in “from scans to structure factors”. In particular note that *dslit* is the post sample slit (ie the slit immediately after the sample), and not the slit immediately before the detector. This value is used to determine the active sample area. So if one would use a 2 mm slit close to the sample and, e.g., 4 mm close to the detector, the delta-resolution is determined by the 4 mm slit, but the value one should use is 2 mm, since this determines the part of the sample that is observed.

1.2.2 Numerical beam profile correction

This corrects for two effects:

- beam profile correction: corrects deviations from an infinitely large and infinitely homogeneous beam.
- Finite sample size: This is useful if the area of the sample illuminated by the beam and seen by the slits extends off the sample. None of the routines deal with square samples, though of course you can check how big an effect it is by using the sample radius calculations.

For glancing incoming angles and not too small scattering angles, there is no need to worry about inhomogeneities in the profile of the X-ray beam or about the finite size of the sample. When this is not the case however, the beam profile correction flag has to be set. The beam profile in this case is determined by four parameters: vertical and horizontal Gaussian widths (fwhm) and vertical and horizontal slit size, which are essentially cut-offs of the Gaussians. This allows one to describe Gaussian profiles, rectangular profiles and mixtures of the two. Note that the slit size is meant to describe the profile at the sample position, and does not necessarily equal the setting of the pre-sample slits in the beam line. The size of the in-plane detector slits (slit1) is also required, because this determines which fraction of the sample is observable. The finite sample size correction is currently only implemented in its simplest form: a circular sample

1.3 Misc

Usually the incident angle is kept fixed - either significantly above the critical angle, or at the critical angle. The same consideration should also be given to the exit angle (usually γ) - particularly if there is a significant miscut.

1.4 set int

Item	Value	Comment
transmat	Transformation matrix	from the <i>hkl</i> used in data collection to the <i>hkl</i> to be used in analysis (which is usually only integers)
auto	Yes	use <i>nbackgr</i> points from scan ends to calculate background
auto	No	use user specified background (or if you put -1, will use background calculated from fit).

2 Tips

I usually write a macro (or series of macros) to integrate my scans - that way it is easy to redo. I usually plot the fits in multiplot mode to give some visualisation that the scans have been correctly integrated.

2.1 Averaging using AVE

Don't forget to put lbragg in - you can use a macro if you are integrating scans on the fly.

References

- [1] E. Vlieg, O. Svensson, and R. Wilke. Anarod. http://www.esrf.fr/computing/scientific/joint_projects/ANA-ROD/index.html.