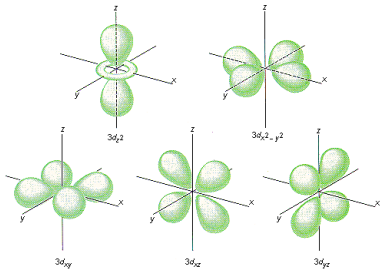
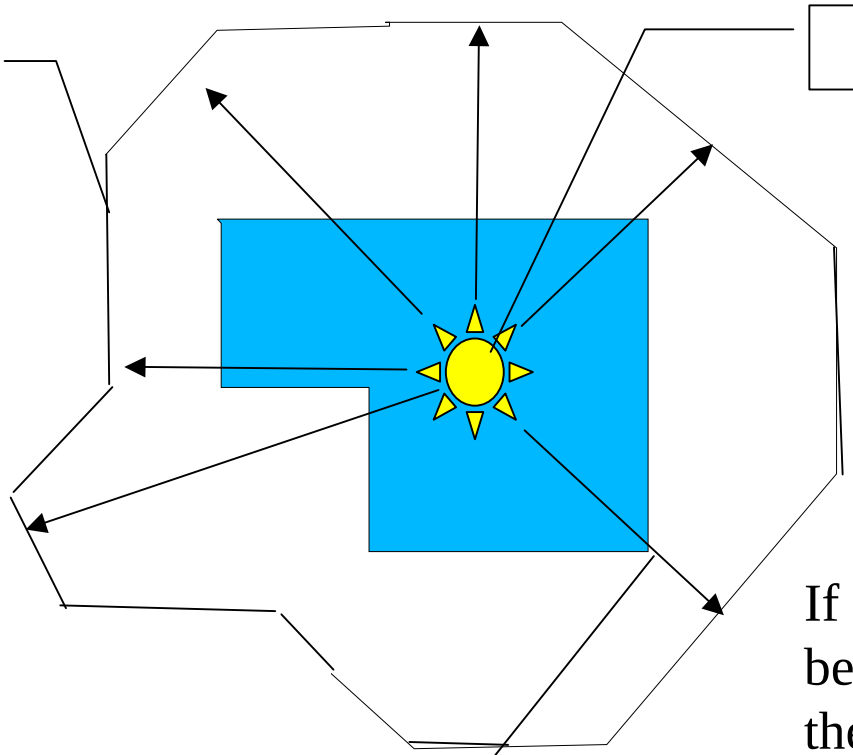
SADABS – Siemens Area Detector ABSorption correction program

- Author : G. Sheldirck
- B. Blessing, *Acta Cryst*. **A51** (1995) 33-38
  - Spherical Harmonic Function $I_c = I_o S_n P(u,v,w)$
- Input : SAINT – RAW files
  - *m.raw or *1.raw, *2.raw etc
  - NOT *t.raw



- Output : 3I4,2F8.2 format h,k,l,F^2,σF^2

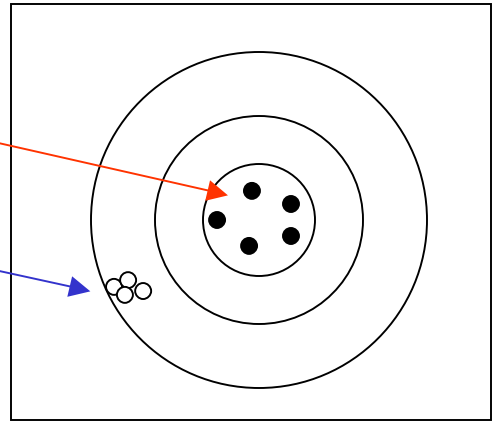
Absorption Surface



If the surface can be modeled then the intensities can be corrected so as to reconstruct the intensity source

Data Errors

- Random
- Systematic



- Increasing ***repetitive sampling*** will increase accuracy (random errors) and increase precision (systematic errors)

SADABS

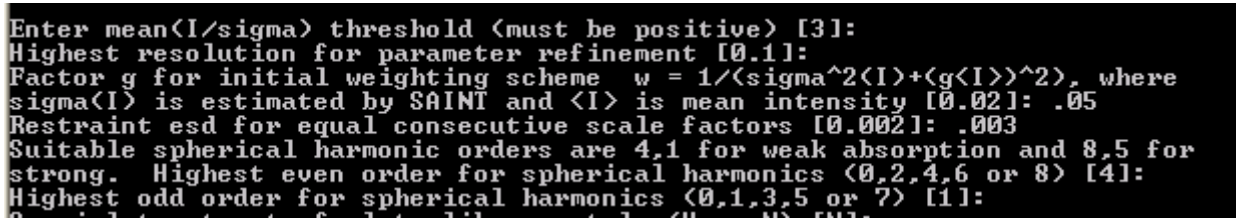
- Systematic error correction program
  - Variation in irradiated crystal volume
    - Large crystals
    - Misaligned crystals
  - X-ray beam inhomogeneity
  - X-ray absorption
  - Crystal decay

Steps

1. Model absorption (systematic errors)
2. Error analysis and derivation of corrected e.s.d.’s
3. Output of corrected data and diagnostics (post script)

Parameters for Part 1

Part 1 - Correction Model



$$w = \left[\sigma^2(I) + \left(g\langle I_c \rangle\right)^2\right]^{-1}$$

g = [0.02] For poor data raise g (similar to the weighting scheme found in XL)

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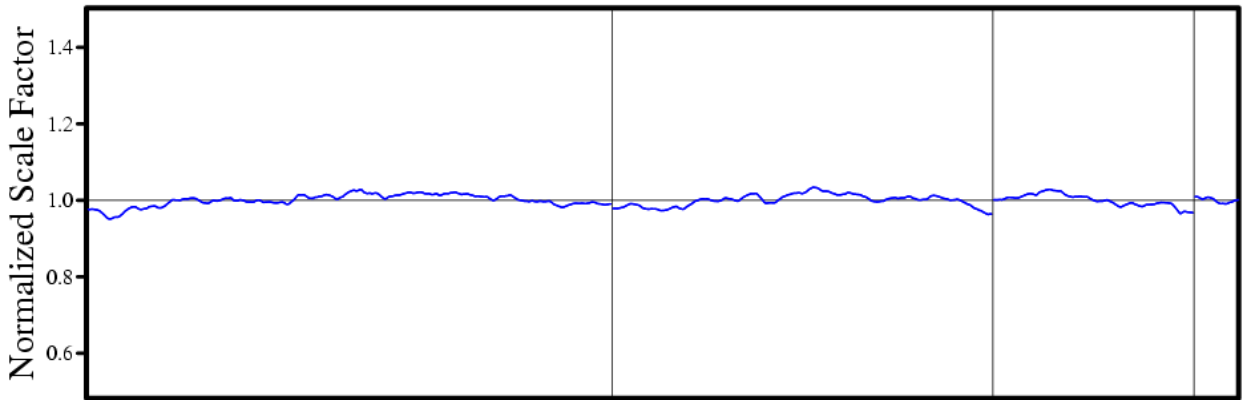
esd restraints = [0.002] 0.001 to 0.005

smooth no-smooth

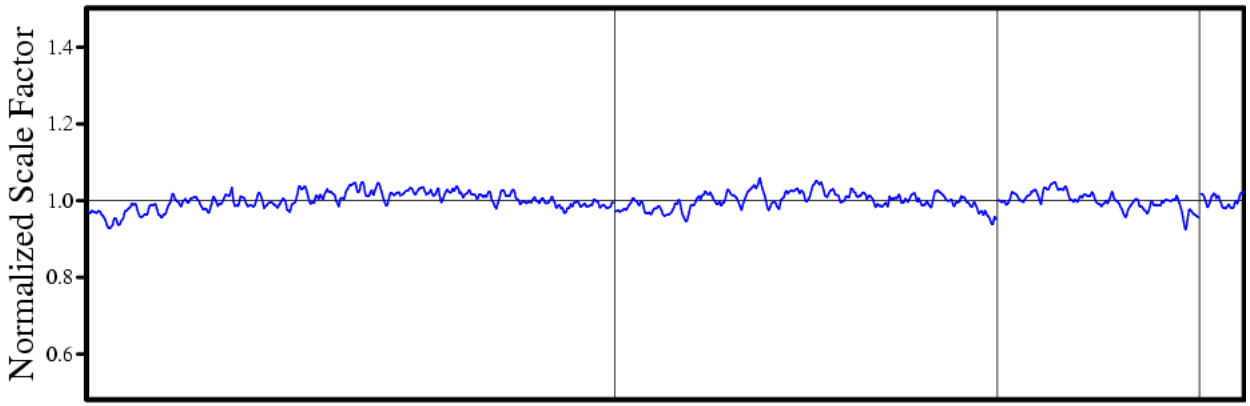
If the esd restraint is too low then the data will be over restrained (high R_{merge}) if too large then the data will be over-fitted (low R_{merge}).

Over restrained/Over fitted

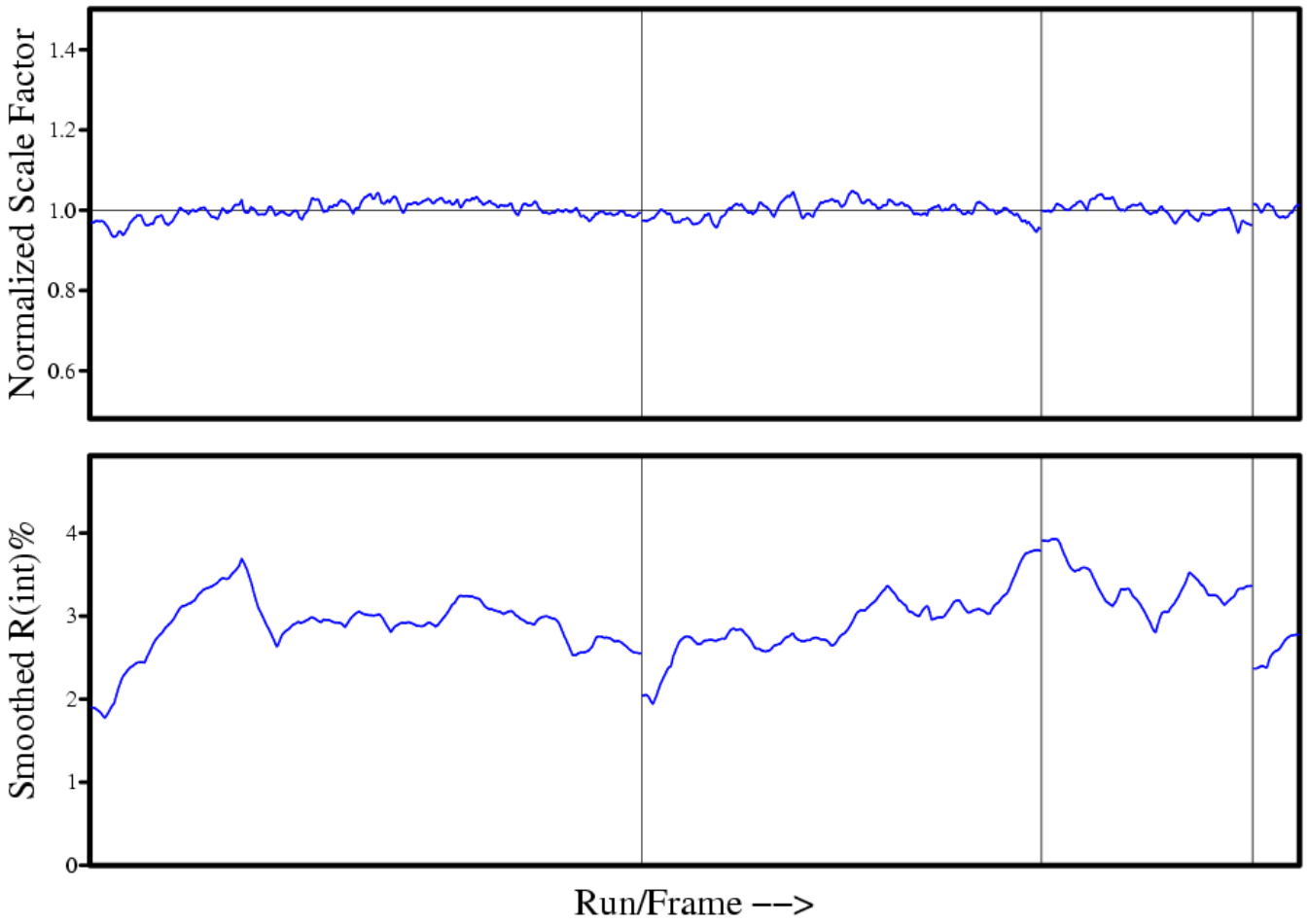
Over restrained = 0.001 g = 0.05



Over fitted = 0.005 g = 0.05



OUTPUT (post script)



Part 1. Refinement

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Suitable spherical harmonic orders are 4,1 for weak absorption and 8,5 for
strong.  Highest even order for spherical harmonics (0,2,4,6 or 8) [4]:
Highest odd order for spherical harmonics (0,1,3,5 or 7) [1]:
Special treatment of plate-like crystals (Y or N) [N]:
Number of refinement cycles [10]:

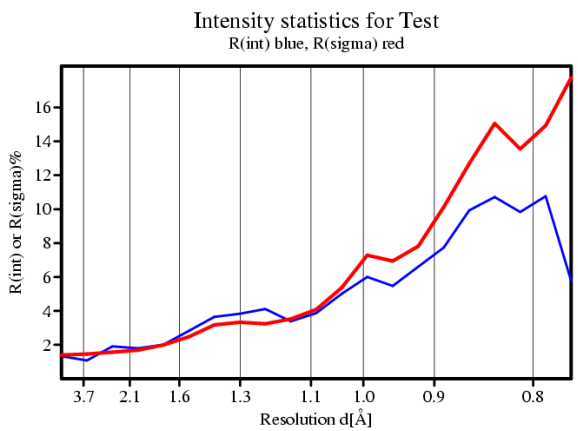
   3109 Reflections employed for parameter determination
Effective data to parameter ratio =    1.55

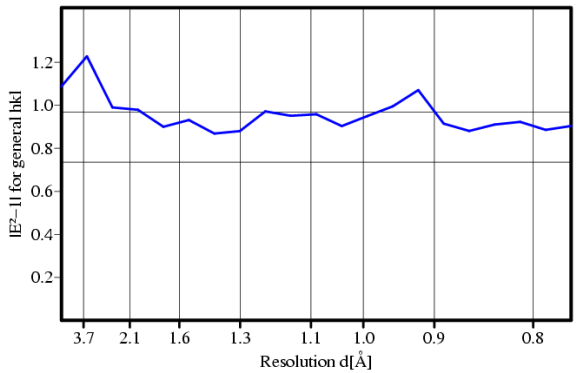
R(int) =  0.0340 (selected reflections only, before parameter refinement)

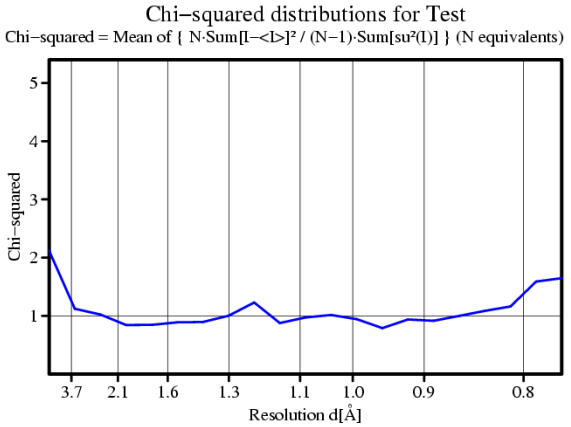
Cycle  R(incid)  R(diffr)  Mean wt.
   1    0.0297    0.0289    0.9715
   2    0.0280    0.0279    0.9720
   3    0.0279    0.0277    0.9721
   4    0.0278    0.0277    0.9721
   5    0.0278    0.0277    0.9721
   6    0.0278    0.0277    0.9721
   7    0.0278    0.0277    0.9721
   8    0.0278    0.0277    0.9721
   9    0.0278    0.0277    0.9721
  10    0.0278    0.0277    0.9721

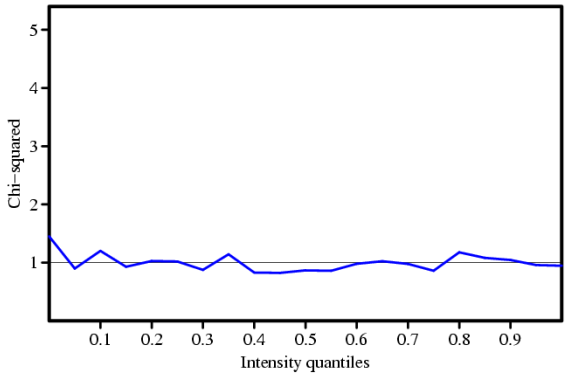
R(int) =  0.0277 (selected reflections only, after parameter refinement)
Repeat parameter refinement (R) or accept (A) [A]: _
```

OUTPUT (Rint / χ^2 distribution)









Part 2. Rejection

```
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:
   4308 total and   1655 unique reflections assuming Friedel's law.

High resolution limit [0.1]:
|I-<I>|/su ratio for rejection [4.0]:

g-value for use in:  su^2 = sigma^2 + (g<I>)^2  (sigma(I) from SAINT). This
is only used for rejections, not for final sigma(I) values [0.10920]: .1

   4308 total and   1655 unique reflections left after |I-<I>|/su test

Repeat parameter refinement (P), repeat rejections (R) or accept (A) [A]:

g = 0.1092  gives best error model.
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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:
   4308 total and   1655 unique reflections assuming Friedel's law.

High resolution limit [0.1]:
|I-<I>|/su ratio for rejection [4.0]:

g-value for use in:  su^2 = sigma^2 + (g<I>)^2  (sigma(I) from SAINT). This
is only used for rejections, not for final sigma(I) values [0.01000]:

   4303 total and   1655 unique reflections left after |I-<I>|/su test

Repeat parameter refinement (P), repeat rejections (R) or accept (A) [A]:

** Impossible to obtain satisfactory error model **

g = 0.0500  gives best error model.

Enter new value for g or <CR> to accept: _
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Uncertainties (e.s.ds)

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Repeat parameter refinement (P), repeat rejections (R) or accept (A) [A]:

g = 0.1123  gives best error model.

Enter new value for g or <CR> to accept:

 Run 2-theta  R(int)  Incid. factors  Diffr. factors    K     Total I>2sig(I)
   1  -28.0  0.0282   0.934 - 1.039   0.950 - 1.028   0.457    1983    1835
   2  -28.0  0.0285   0.953 - 1.046   0.961 - 1.025   0.468    1428    1321
   3  -28.0  0.0331   0.927 - 0.994   0.974 - 1.029   0.466     741     685
   4  -28.0  0.0252   0.929 - 0.963   0.950 - 1.014   0.398     154     144

su = K * Sqrt[ sigma^2(I) + (g<I>)^2 ]  where 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), repeat rejections (R) or accept (A) [A]:
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$$\sigma^2(I_c) = K[\sigma^2(I) + (g\langle I_c \rangle)^2]$$

Part 3. Output of Data/ Diagnostics

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PART 3 - Output Postscript diagnostics and corrected data

Write Postscript diagnostic file (Y or N) [Y]:
Enter name of Postscript file [sad.eps]: test.eps
Short (<21 chars) title for Postscript plots [Test]: new data
Spatial display of (I-<I>)/su greater than [3.0] (0 for none):

Repeat (R), write .hkl file (W), or quit (Q) [W]:
Enter name of output .hkl file [sad.hkl]: test.hkl
 Mu*r of equivalent sphere for additional spherical absorption correction.
Enter <CR> if none: .07
Lambda/2 correction factor (0 if none, e.g. for MWPC!) [0.0015]: 0.002
    4306 Corrected reflections written to file test.hkl

Ratio of minimum to maximum apparent transmission:  0.923742
Repeat (R), write .hkl file (W), or quit (Q) [Q]:
```

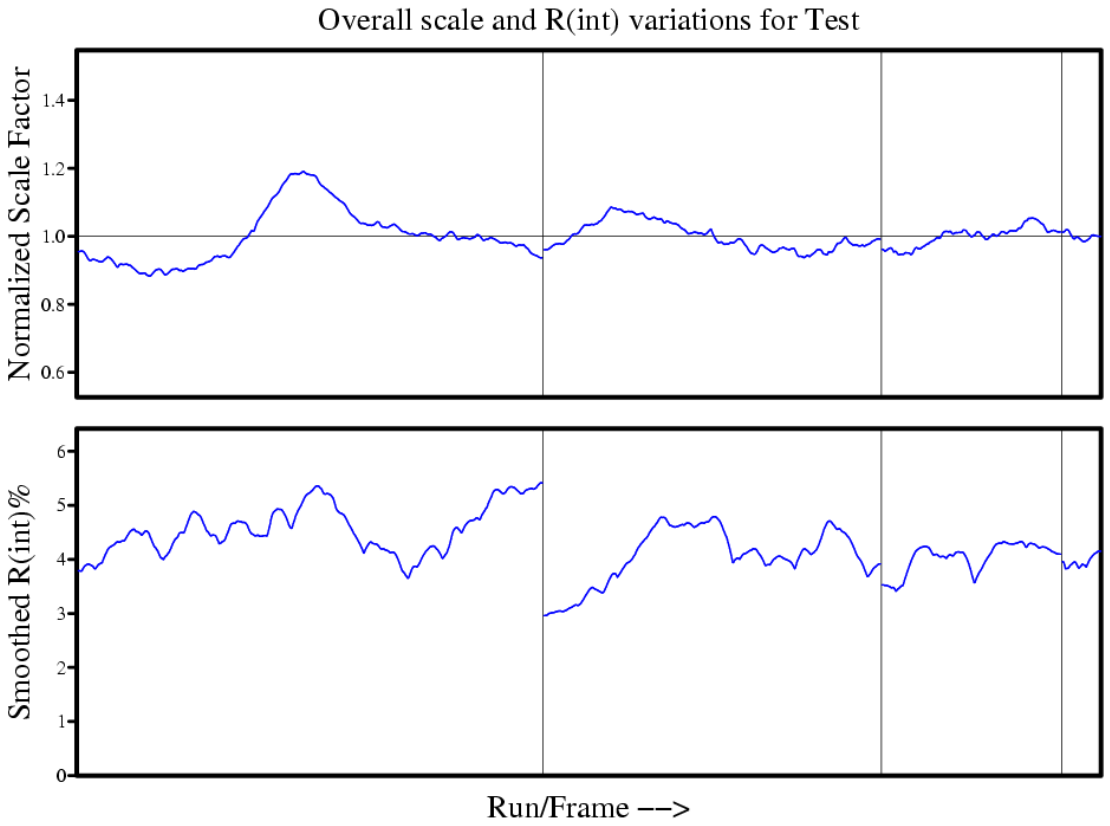
Spherical correction : increases isotropic displacement

- increase in case of n.p.ds

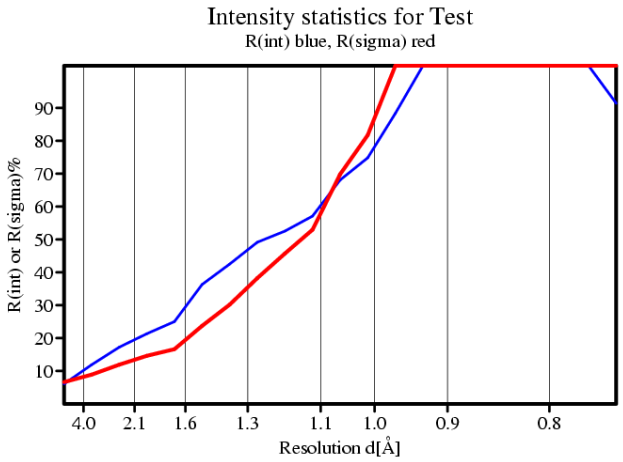
Lambda/2 correction (002/004/008 etc)

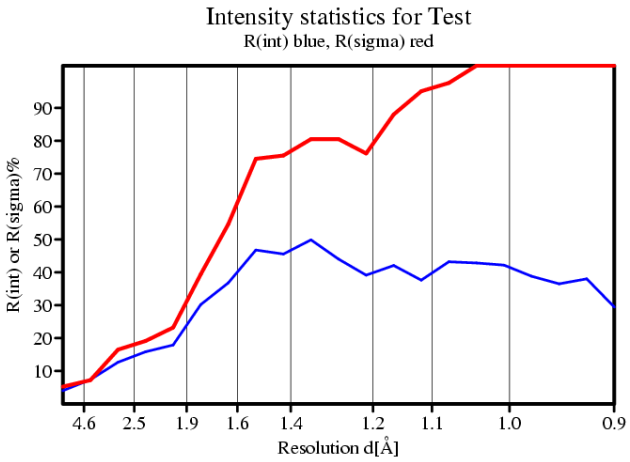
- do not use for multi-wire
- Kirschbaum, Martin & Pinkerton, *J. Appl. Cryst.* **30** (1997) 514-516

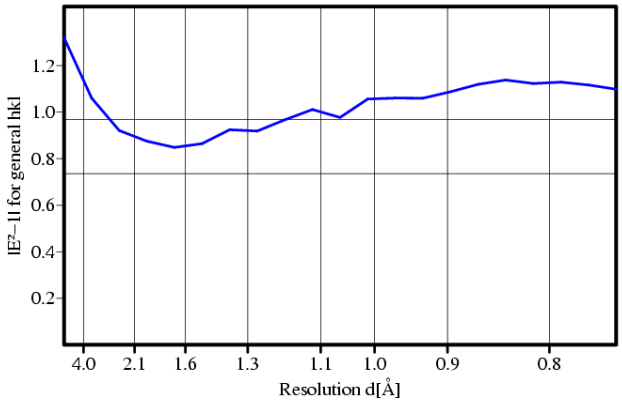
Bad Frame Set

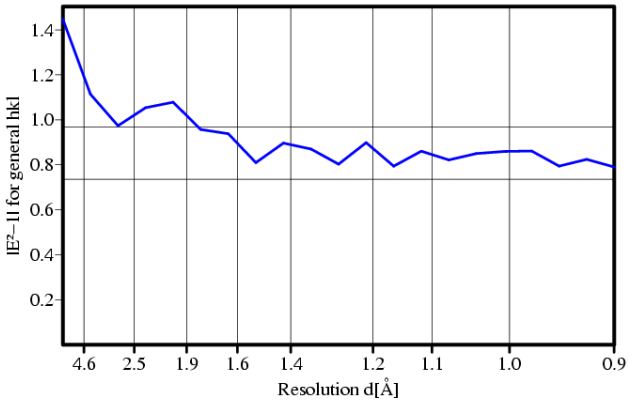


Bad Data

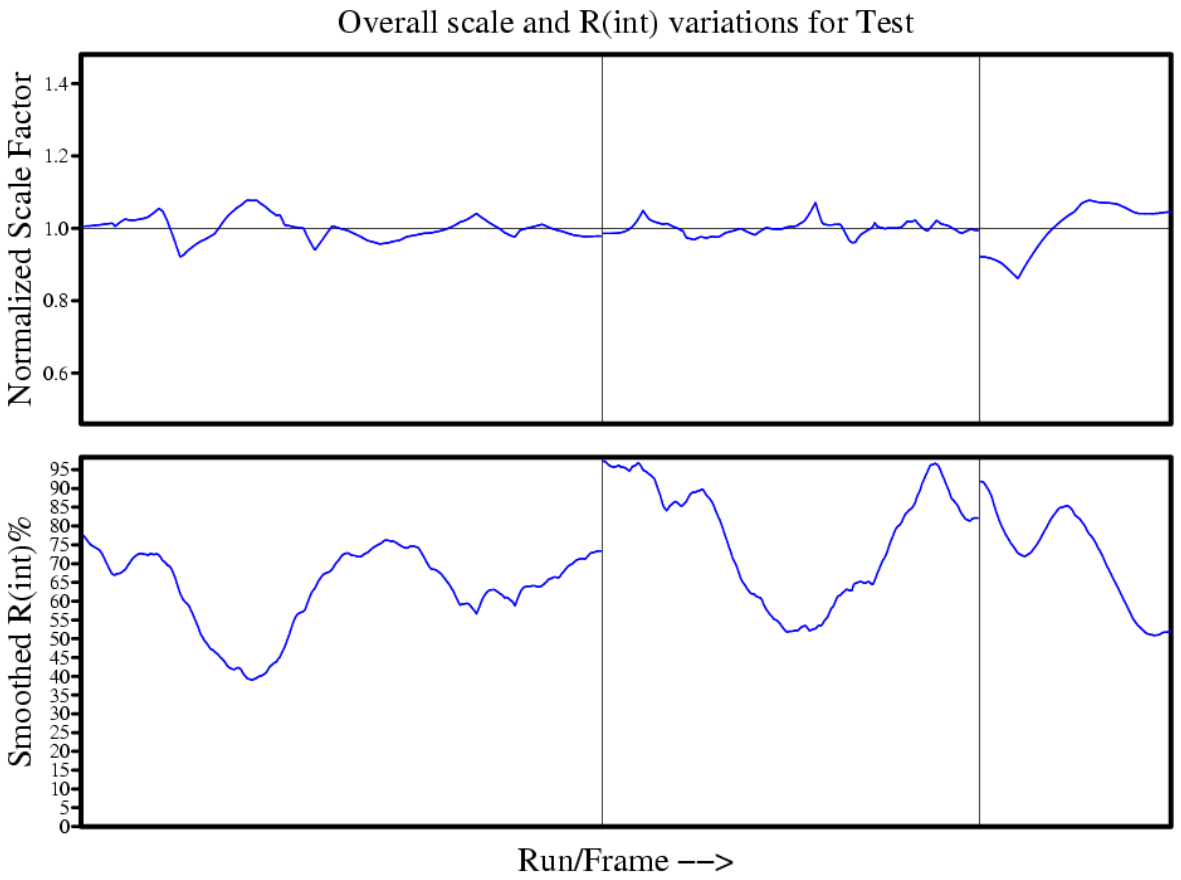

Intensity statistics for Test
R(int) blue, R(sigma) red
R(int) or R(sigma)%
90
80
70
60
50
40
30
20
10
4.0
2.1
1.6
1.3
1.1
1.0
0.9
0.8
Resolution d[Å]


Intensity statistics for Test
R(int) blue, R(sigma) red
R(int) or R(sigma)%
90
80
70
60
50
40
30
20
10
4.6
2.5
1.9
1.6
1.4
1.2
1.1
1.0
0.9
Resolution d[Å]

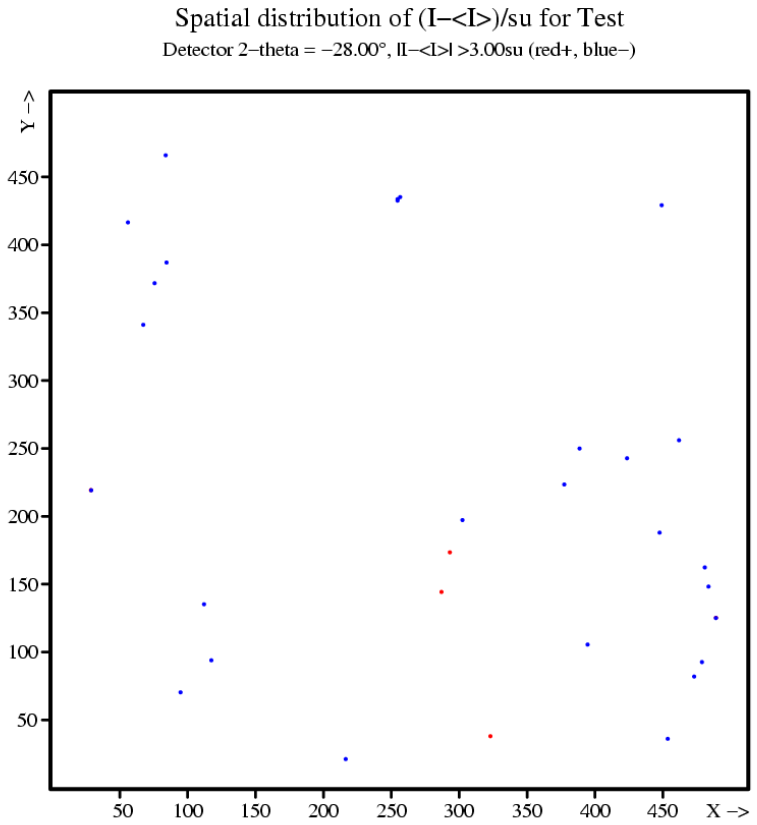

|E²−1| for general hkl
1.2
1.0
0.8
0.6
0.4
0.2
4.0
2.1
1.6
1.3
1.1
1.0
0.9
0.8
Resolution d[Å]


|E²−1| for general hkl
1.4
1.2
1.0
0.8
0.6
0.4
0.2
4.6
2.5
1.9
1.6
1.4
1.2
1.1
1.0
0.9
Resolution d[Å]

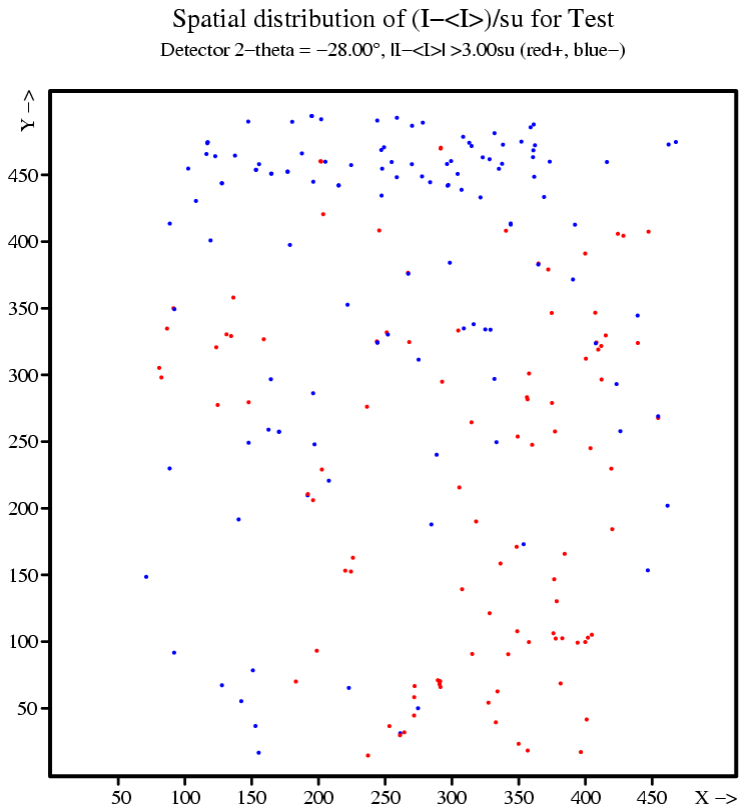
Real BAD DATA

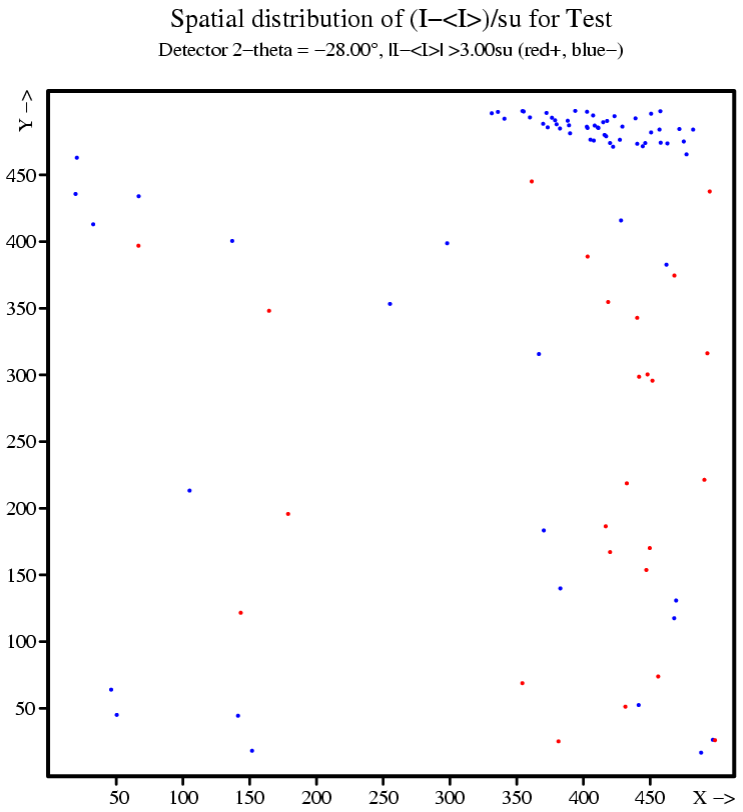


FRAME ERRORS



Low Temperature Obstruction





Very Weak Data