

Basics of Jana2006 to process high-pressure single-crystal data

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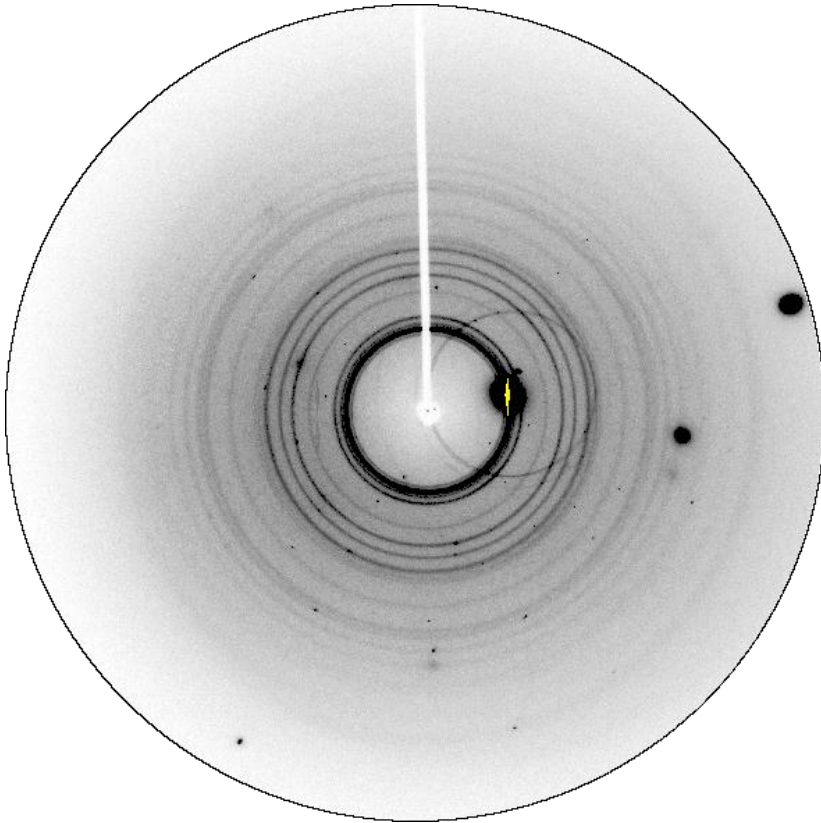
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³ Jülich Centre for Neutron Science, Germany

⁴ Institute of Physics, Czech Academy of Sciences, Prague, Czech Republic

Problems of the high-pressure single-crystal diffraction data measured in diamond anvil cells with area-sensitive detectors



- ▶ Limited resolution in $\sin \theta / \lambda$
- ▶ Overlap of reflections with powder rings due to the gasket and backing plates
- ▶ Overlap of reflections with those due to the diamonds
- ▶ Shadowed reflections by the diamond cell
- ▶ Diamond “dips”

Identification of outliers

- On the basis of symmetry equivalent reflections

The more equivalent reflections there are to average, the easier to find the outliers

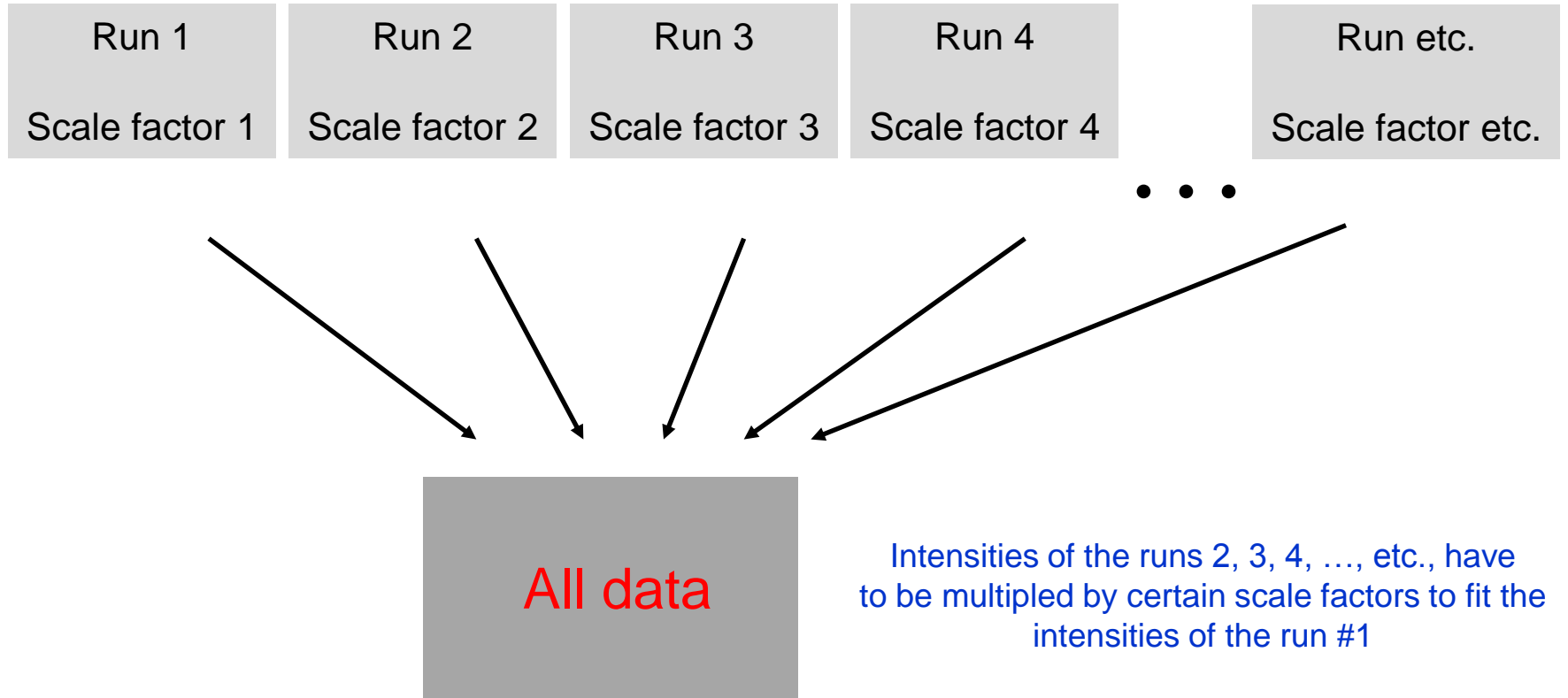
→ the higher the redundancy the better

In the initial stages of data reduction, one can use “approximate” symmetry to find the outliers → Laue symmetry

- On the basis of the refinement

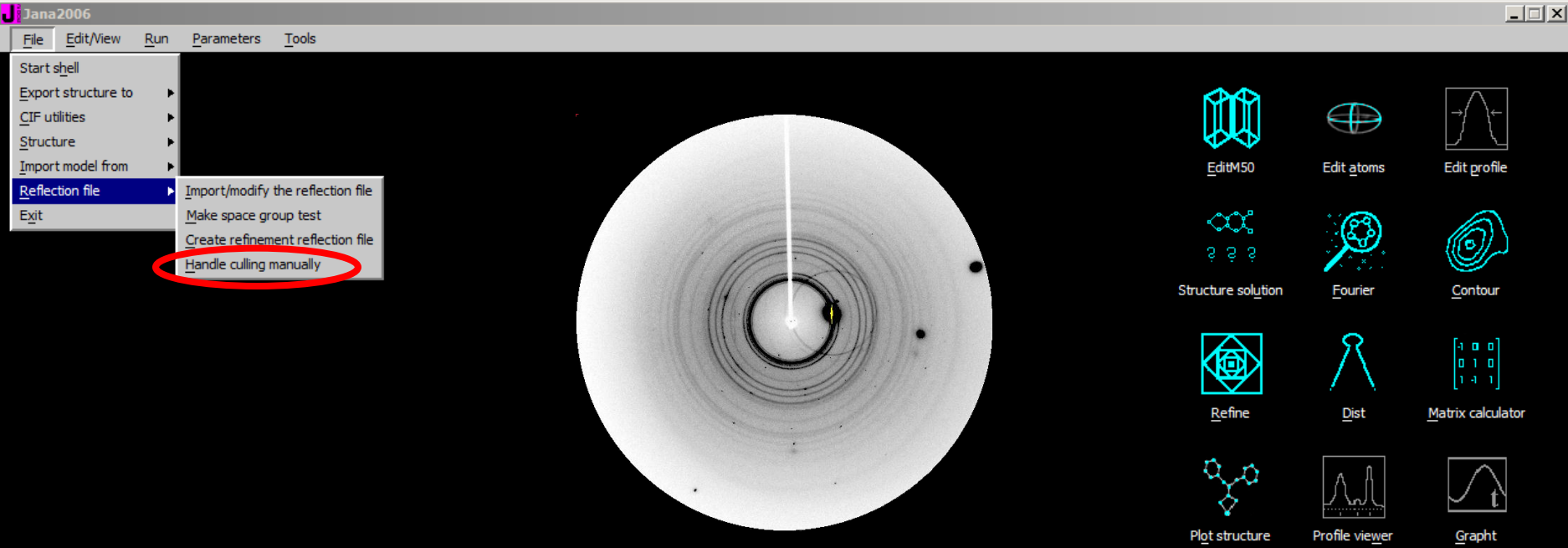
$F_{\text{obs}} / F_{\text{calc}}$ plots

Scaling of several datasets



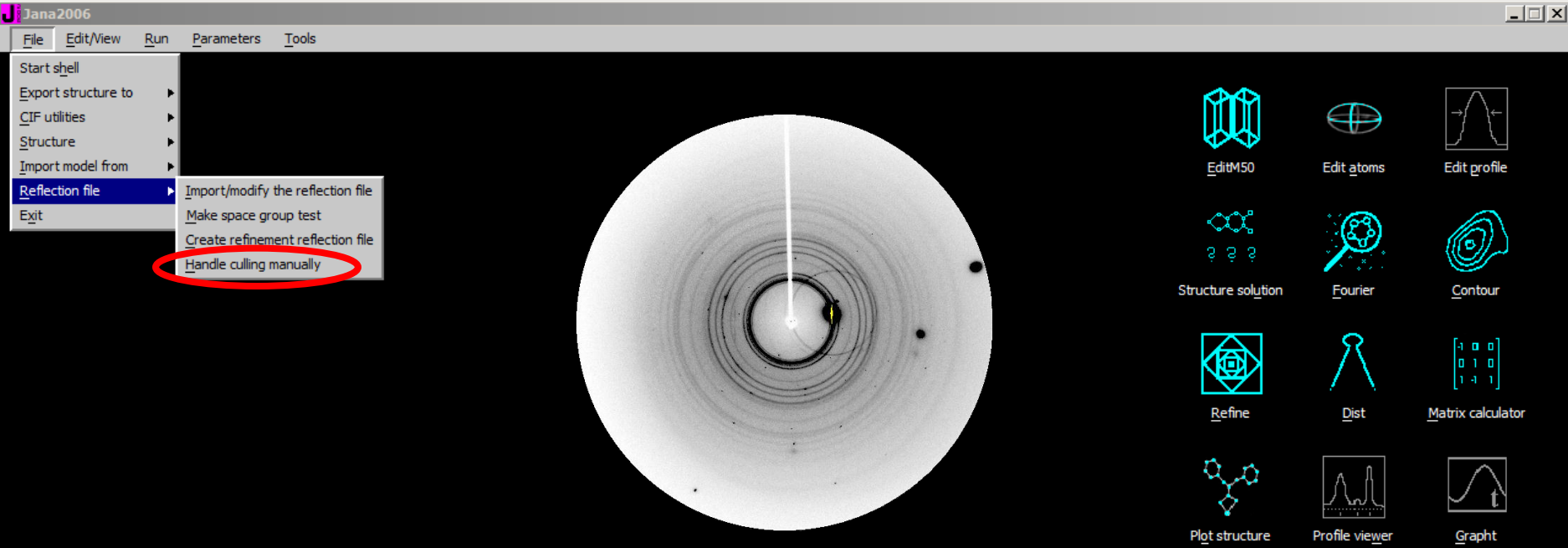
Wrong reflection intensities introduce errors in the scale factors.

Any error in any of the scale factors influences all reflections in the data !!!



High-pressure single-crystal x-ray data in Jana2006:
the utility **File** → **Reflection file** → **Handle culling manually**
as an aid to identify all outliers and to scale the data

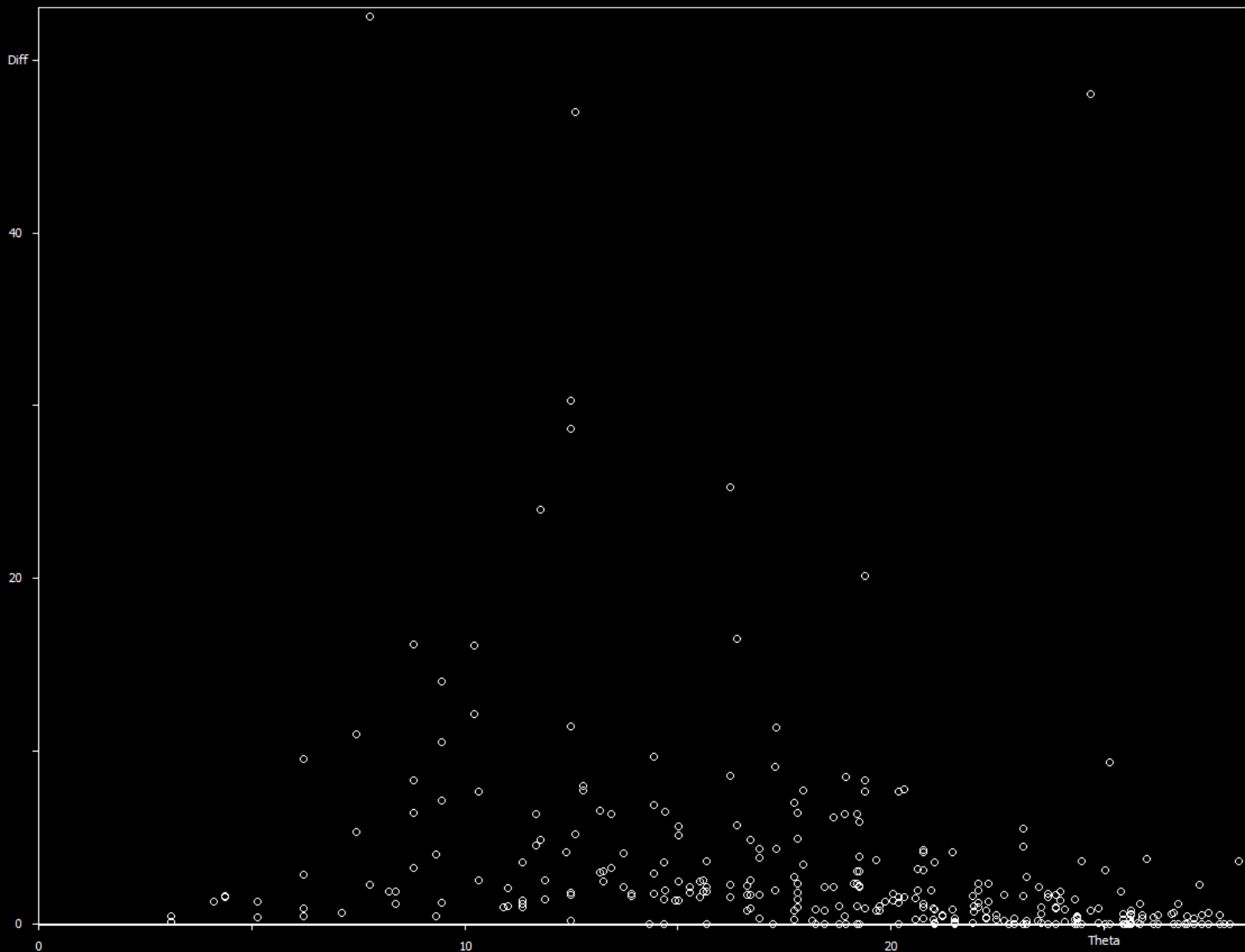




The utility **File** → **Reflection file** → **Handle culling manually** was originally developed for processing high-pressure single-crystal synchrotron data from HASYLAB.

It could also be applied to any data measured at atmospheric pressures as well as to modulated structures.





Quit

X=I(Min)

X=I(ave)

X=I(max)

X=theta

Linzoom

Run average

Average report

Edit atoms

Edit m40 file

Run refine

Refine report

Refine commands

Run Fourier

Fourier commands

Run Contour

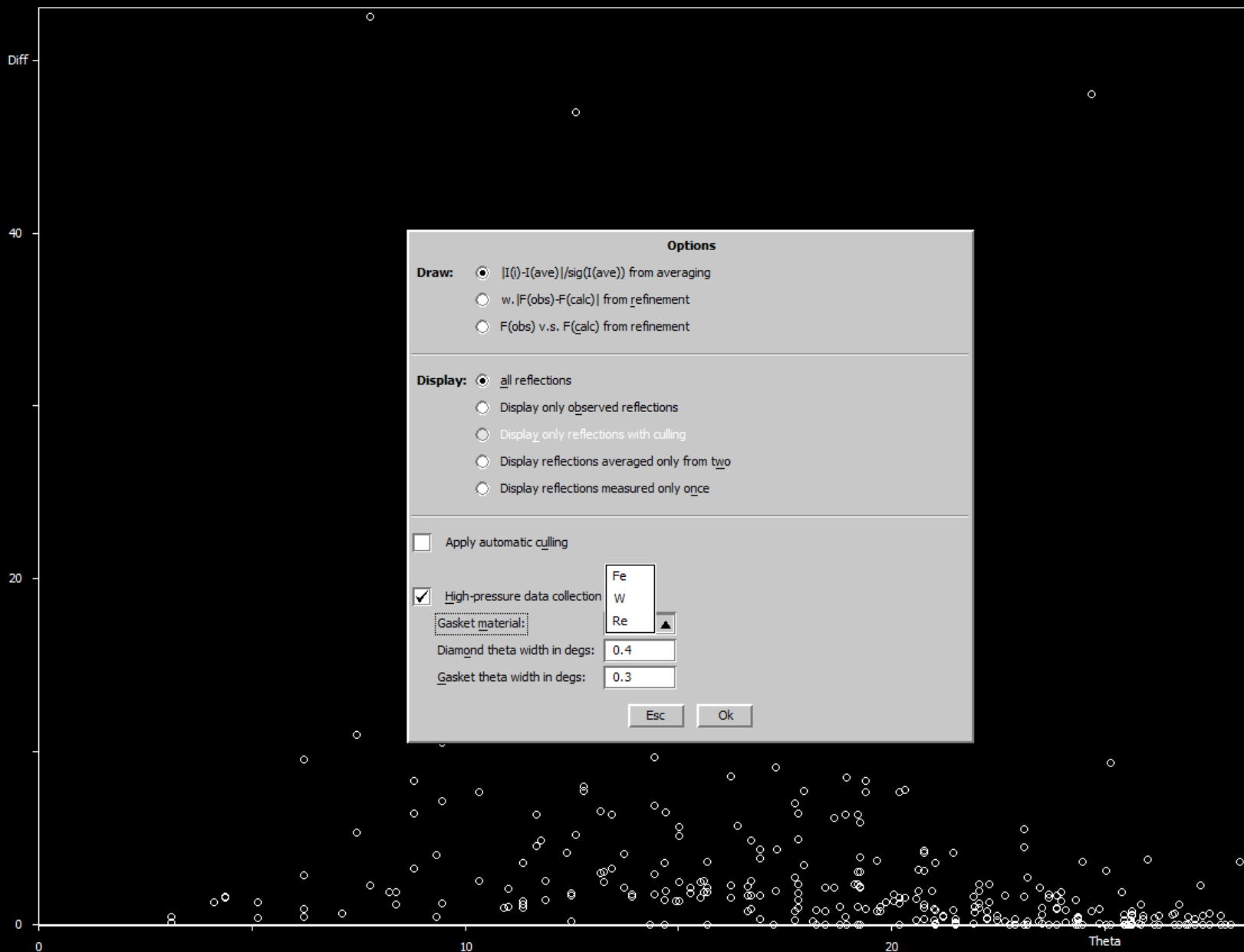
Reset culled

Satellite filter

Options

Rint(obs)/Rint(all) : 10.96/12.71 N(obs)/N(all) : 144/318 Redundancy : 6.016 Culled(man)/Culled(auto) : 0/0

R(obs)/R(all) : 10.94/34.69 Rw(obs)/Rw(all) : 10.48/11.63 GOF : 3.220



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Options

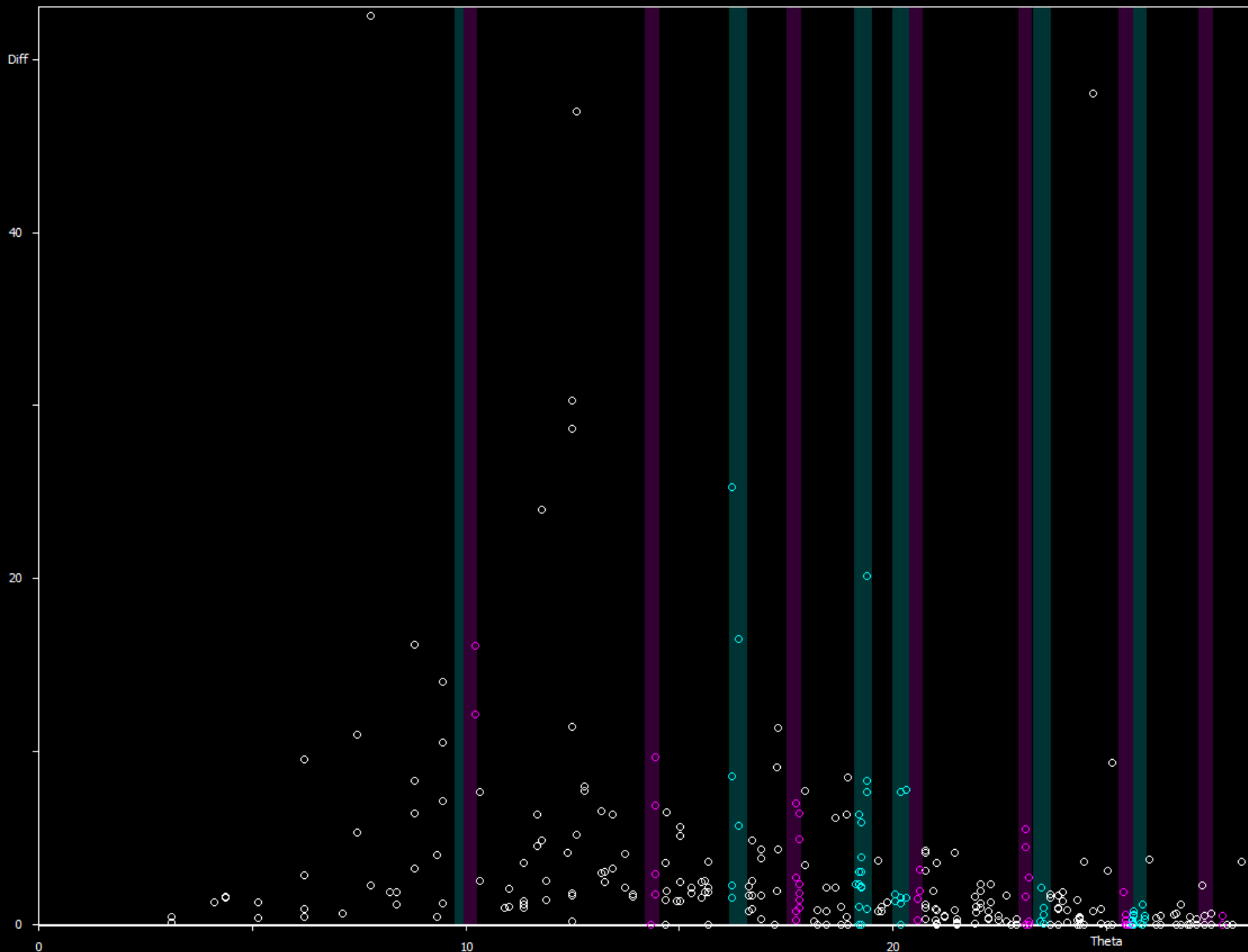
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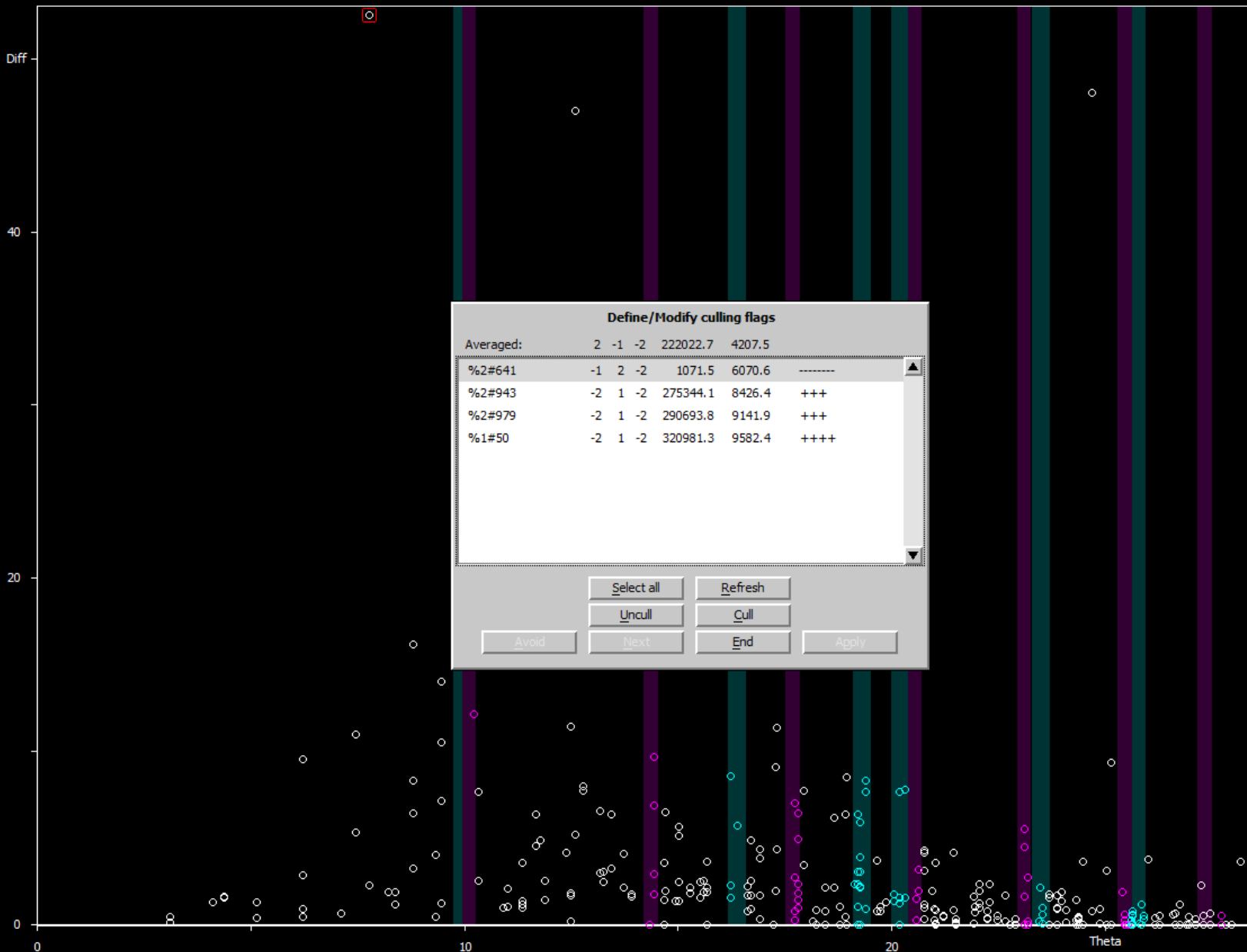
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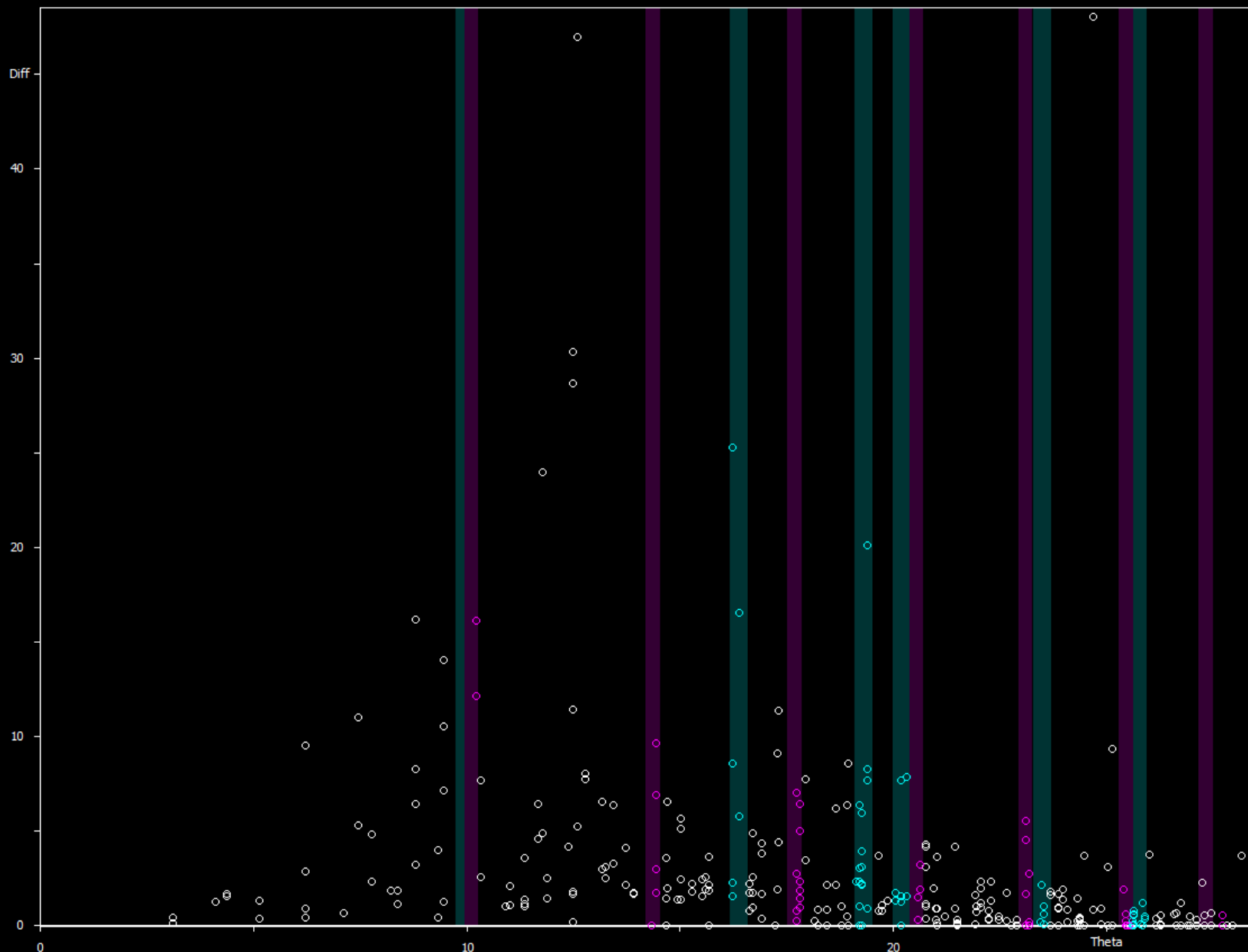
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- Refine commands
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- Satellite filter
- Options



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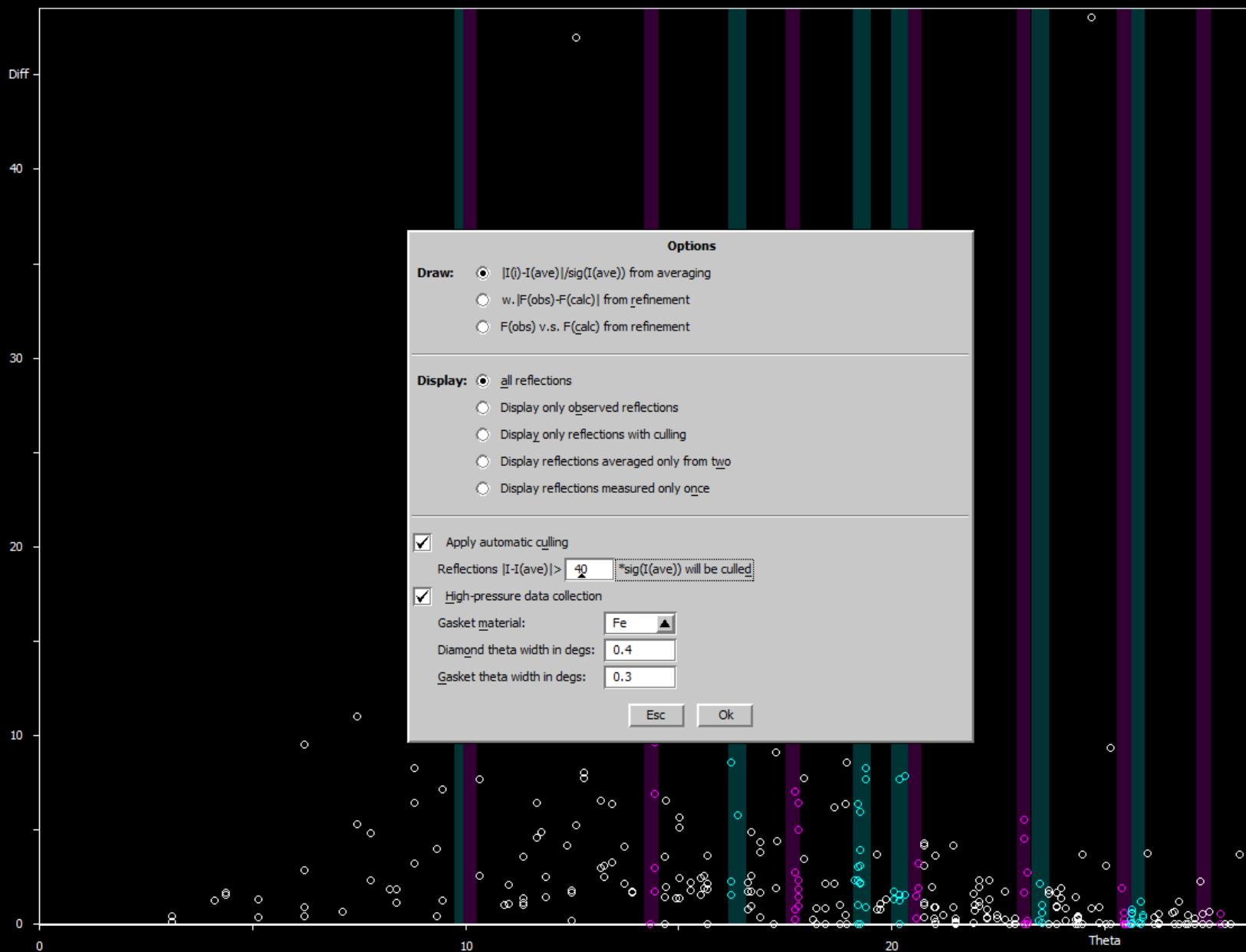
Rint(obs)/Rint(all) : 10.49/12.25 N(obs)/N(all) : 144/318

Redundancy : 6.016 Culled(man)/Culled(auto) : 1/0

R(obs)/R(all) : 10.80/34.53

Rw(obs)/Rw(all) : 10.29/11.46

GOF : 3.180



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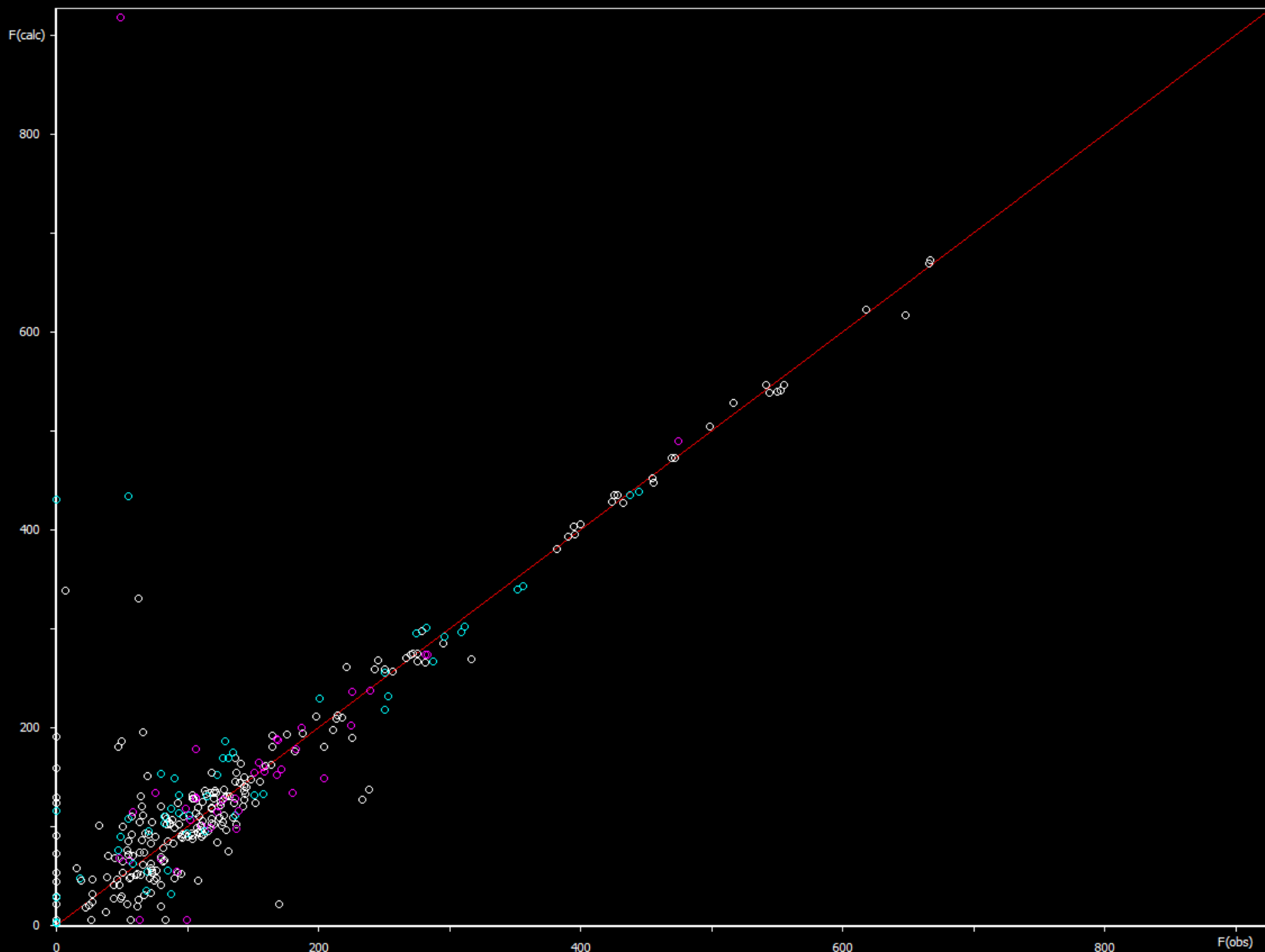
Redundancy : 6.016 Culled(man)/Culled(auto) : 1/0

R(obs)/R(all) : 10.80/34.53

Rw(obs)/Rw(all) : 10.29/11.46

GOF : 3.180

F(obs) versus F(calc)



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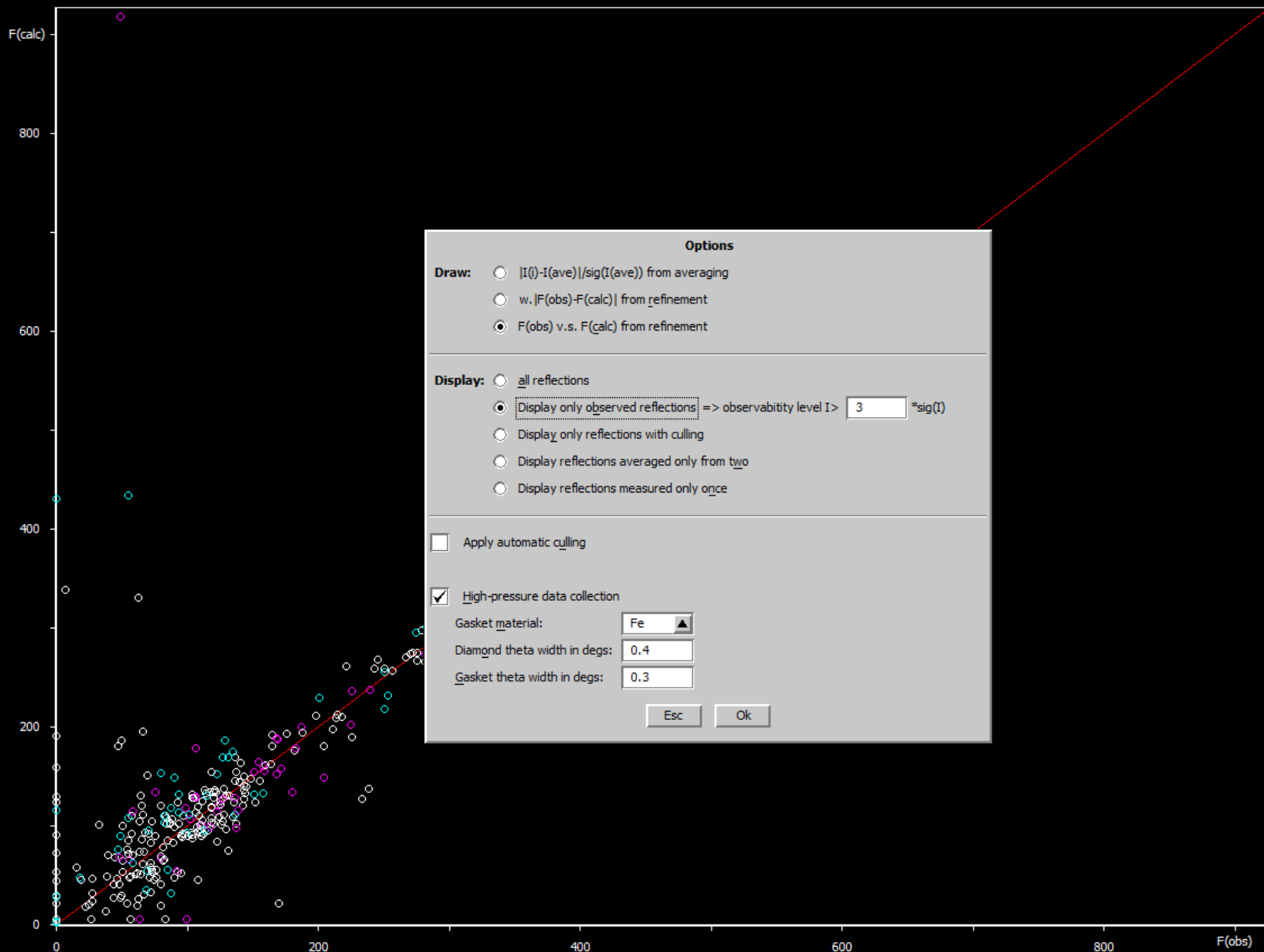
Reset culled

Satellite filter

Options

Rint(obs)/Rint(all) : 9.34/11.16 N(obs)/N(all) : 143/317 Redundancy : 6.016 Culled(man)/Culled(auto) : 4/0
R(obs)/R(all) : 6.28/21.61 Rw(obs)/Rw(all) : 5.64/6.69 GOF : 1.840

F(obs) versus F(calc)



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R(obs)/R(all) : 6.28/21.61 Rw(obs)/Rw(all) : 5.64/6.69 GOF : 1.840

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Run refine

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Refine commands

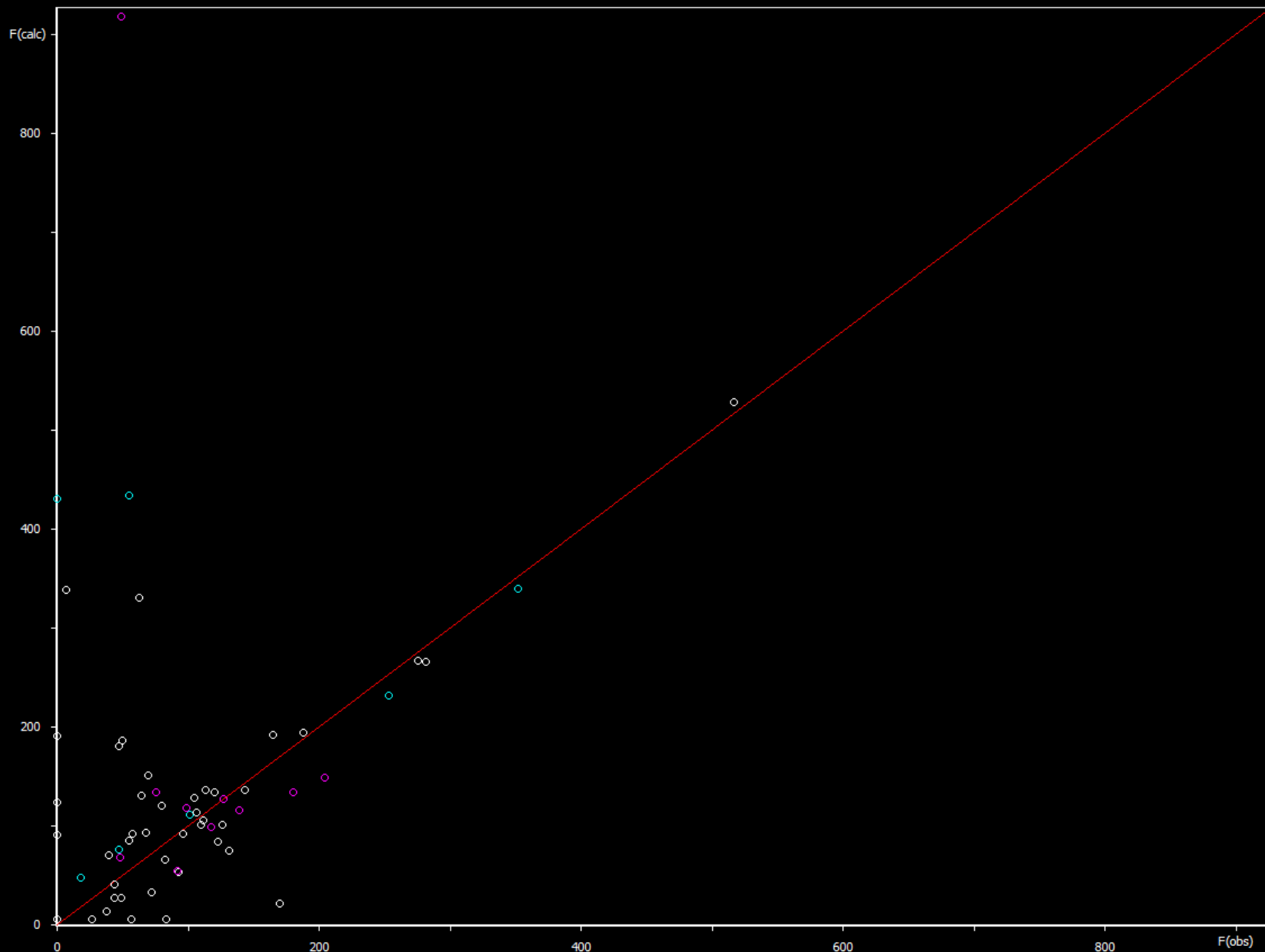
Run Fourier

Fourier commands

Run Contour

Reset culled

Options



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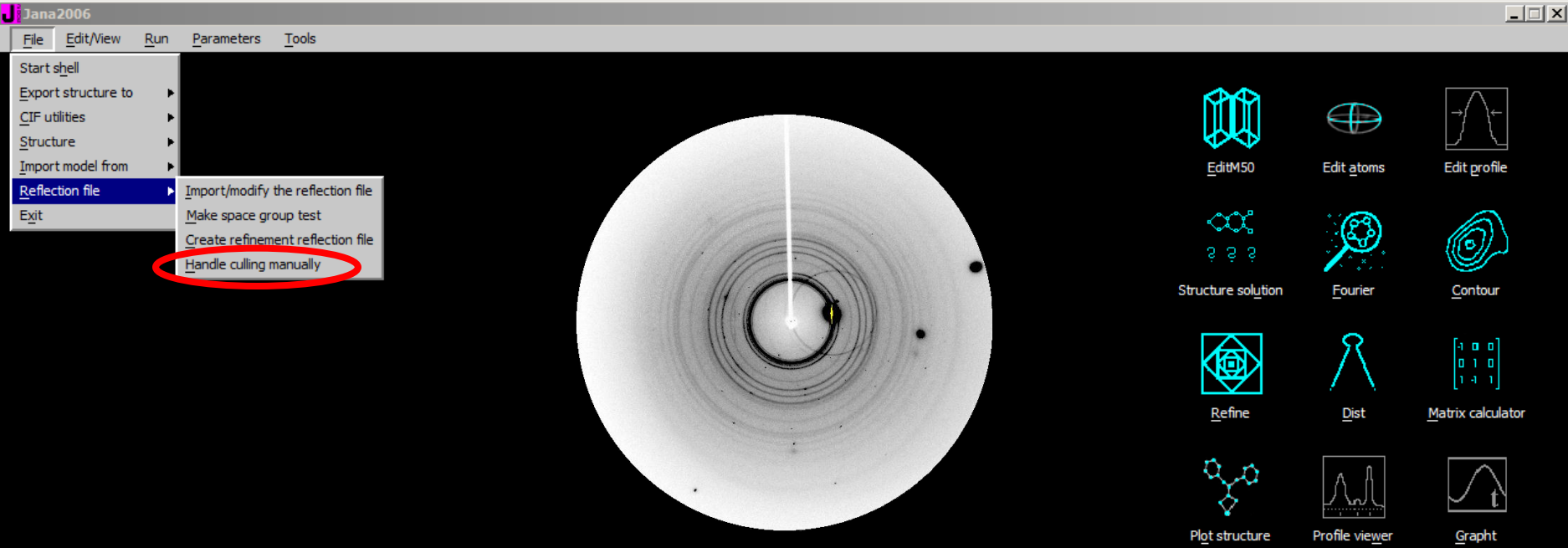
Run Contour

Reset culled

Satellite filter

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R(obs)/R(all) : 6.28/21.61 Rw(obs)/Rw(all) : 5.64/6.69 GOF : 1.840



Warning: The manual culling in Jana2006 must be handled with care.

While using this utility, it is too easy to fit the data to one's favorite structural model.

The best way to identify the outliers is to visually inspect the reflections on the images.

