SINCE 1887

THE NEW STOE SPHERES

A Convenient and Elegant Visualization Tool

Manual Scaling – Is It Really Necessary?

Scaling is an important step in diffraction data processing and is an integral part of every diffractometer software. It corrects systematic intensity errors thus avoiding subsequent refinement problems. Many measurements are fine if only a basic frame scaling, or the default scaling method, is employed. There are however occasions (e.g., anisotropic crystal shape, icing, decay, etc.) where manual scaling is crucial.

The quality of the direction-dependent scaling (as *e.g.* Beam(out) Scaling) could so far be judged in LANA by clicking the "Diagnosis" and subsequently the "Beam (in, out)" button, leading to arrays of polar coordinates (a typical array is shown in Fig. 1). Evaluating data quality by looking at a chart of numbers is however not very convenient. Choosing the option "Spheres" instead of "Diagnosis" leads to an elegant spherical representation of direction-dependent scaling quality.

Number of reflections:

4	7	9	5	7	2	2	3
24	56	54	40	18	-	-	6
66	106	102	59	25	3	2	15
131	182	129	87	47	13	25	54
179	218	154	62	37	31	57	97
128	149	75	26	30	31	59	98
72	75	35	14	19	29	39	57
15	15	11	6	3	13	10	11

Figure 1: Example of "Beam(out)" scaling diagnosis in polar coordinates.

When Elegance Meets Function

Fig. 2 shows a platelet shaped crystal of YbB_6. Crystals with shapes deviating so strongly from the ideal sphere are classical candidates requiring manual adjustment of the scaling. The typical SPHERES window with scaling parameters slightly weaker than standard ones (FS[1] = 2, BOS[2] = 2/0, R_{int} = 0.18) applied to this dataset can be seen in Fig. 3.

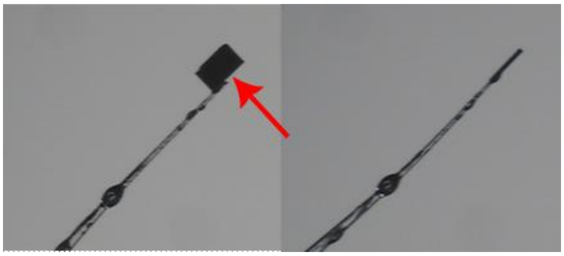

Figure 2: The original sample in two different orientations.

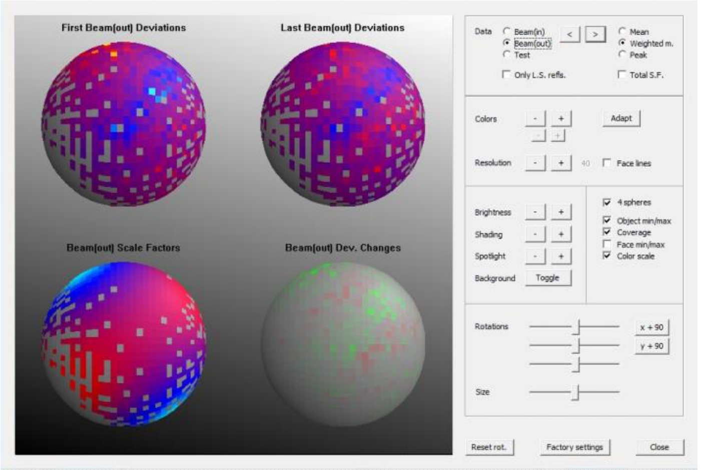

Figure 3: Typical window of STOE SPHERES with a data set with insufficient scaling applied.

The facet[3] colours indicate the average deviation[4], where magenta is zero, yellow is positive and blue is a negative deviation for the upper two spheres. For the lower left sphere, which represents the scale factors, colours range as well from yellow (maximum) to light blue (minimum). Due to the crystal shape, a part of the diffracted beams was systematically weakened by passing through the long axes of the platelet (indicated by the red arrow in Fig. 2), requiring the scale factors of these reflections to be larger. The right side of Fig. 2 displays the orientation of the crystal, which also corresponds to the orientation of the spheres in Fig. 3 and Fig. 4. Since facets bundle reflections being diffracted in the same direction, the colour distribution of the *Scale Factors* sphere should mirror the shape of the crystal.

On the *Deviation Changes* sphere, facets should be green (improvement of deviations) or grey (no change). Red facets indicate a deterioration of the deviations. Judging from the colours of the spheres, the currently applied scaling is insufficient.

[1] FS = Frame Scaling
[2] BOS = Beam(out) Scaling
[3] A facet represents a family of reflections which were diffracted in similar directions.
[4] Here "deviation" means the (weighted) difference between the individual intensity of a reflection and the average intensity of the corresponding family of symmetry-related reflections.

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Fig. 4 shows the spheres of the same data set with a stronger scaling applied (FS = 5, BOS = 10/7, R_{int} = 0.13). Here the colours of the *Last Beam(out) Deviations* sphere have already become more uniform; the scale factors resemble closer the shape of the crystal and the *Beam(out) Dev. Changes* sphere shows more green facets.

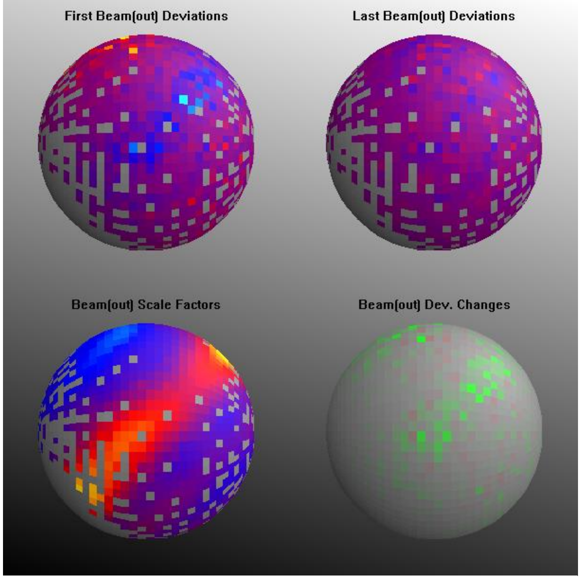

Figure 4: STOE SPHERES after applying a stronger scaling.

A last step to improve the data is the outlier rejection. Reflections with an intensity too far away from the average intensity of their family of symmetry-related reflections will be discarded in this step. The result of an outlier rejection (with coefficients 0.01/0/0/0.2) is shown in Fig. 5. Since this data set was measured with a large redundancy, a strong outlier rejection could be performed without losing unique reflections. Now the *Last Beam(out) Deviations* sphere (upper right) is almost uniformly magenta coloured while the *Beam (out) Dev. Changes* sphere (lower right) shows barely any red facets.

Through these corrections, the reflection intensities in the dataset are trustworthy and later problems with ADP size or element assignment due to falsified intensities can be avoided.

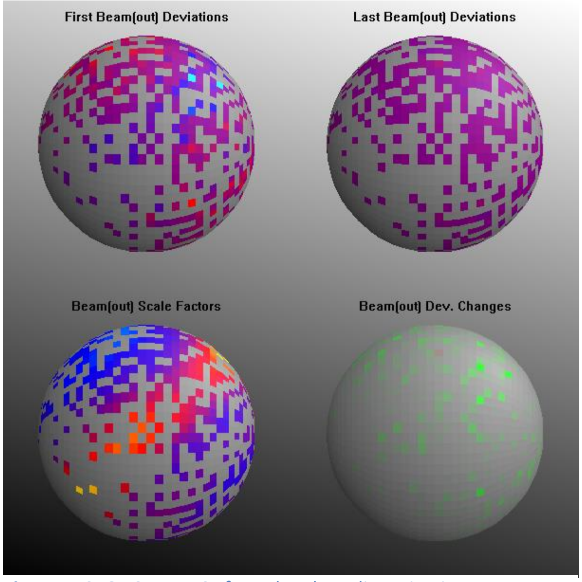

Figure 5: STOE SPHERES after a harsh outlier rejection.

Note that underscaling might be what most of us think of regarding scaling issues, however it is also possible to overscale data. Noticing overscaling in a data set is equally important for a smooth structure solution and refinement process.

In Summary

Checking the quality of the data scaling and outlier rejection can save you a lot of headache in the long run. Doing so with the STOE SPHERES application allows to judge the condition of your data at a glance!

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