

Supporting information for paper “Reliable structural data from Rietveld refinements *via* restraint consistency”

Statistical analysis

The formal derivation of $\mathcal{K}_p(N)$ is not straightforward, but it can easily be calculated for small values of N relevant for our task using random number generator. The saw-like pattern of $\mathcal{K}_{p,N}$ is an unavoidable result of order statistics employed; the easiest way to use it is to rely on a pre-calculated table (provided in Table S1 using 1×10^6 trials for each N). We chose $\mathcal{K}_{0.05,N}$ (resulting in 95% confidence in the significance of an outlier) for all further calculations.

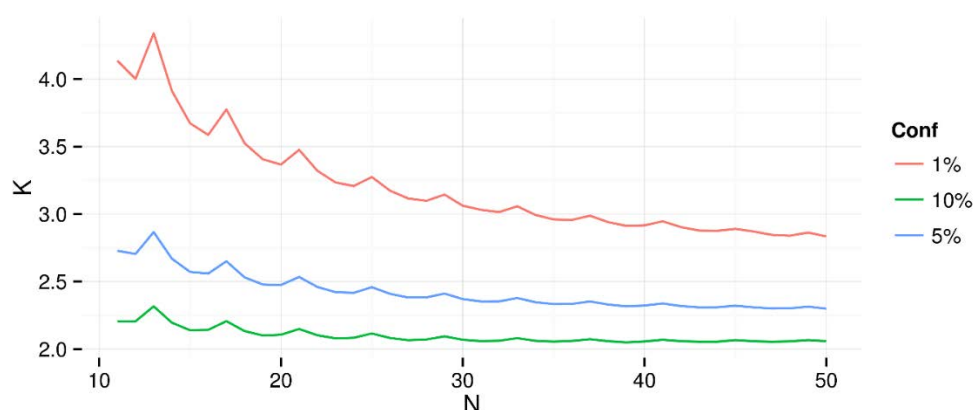


Figure S1. Dependence of $\mathcal{K}_{p,N}$ on N for practically important values of confidence (defined as $1 - p$).

Table S1. Pre-calculated values of $\mathcal{K}_{0.05,N}$

5	5.626193
6	3.596898
7	2.99684
8	2.914194
9	3.37232
10	2.931963

11	2.727716
12	2.704177
13	2.865769
14	2.669211
15	2.570927
16	2.558814
17	2.649954
18	2.530756
19	2.477919
20	2.474183
21	2.533845
22	2.459834
23	2.421229
24	2.415943
25	2.457848
26	2.408792
27	2.380904
28	2.382168
29	2.410482
30	2.369893
31	2.35215
32	2.35247
33	2.37772
34	2.346434
35	2.332696
36	2.333793
37	2.352136
38	2.329313
39	2.31586

40	2.322118
41	2.337192
42	2.318222
43	2.308369
44	2.309286
45	2.320534
46	2.30881
47	2.29984
48	2.300816
49	2.314276
50	2.29827

Table S2. HUW_i values for S-vario-f dataset.

<u>Bond</u>	<u>Lower</u>	<u>Upper</u>	<u>Ideal</u>	<u>HUW</u>
O1 C2b	1.426844	1.467469	1.437	0.020313
O1 C1	1.385906	1.451531	1.432	0.032813
O1b C1b	1.362031	1.458906	1.43	0.048438
O2 C2	1.404094	1.493156	1.433	0.044531
O2b C2b	1.398844	1.422281	1.409	0.011719
O2b C5b	1.417844	1.467844	1.453	0.025
O4b C4b	1.365594	1.449969	1.432	0.042188
O3b C3b	1.341031	1.426969	1.409	0.042969
O3 C3	1.380656	1.491594	1.433	0.055469
O4 C4	1.363094	1.455281	1.417	0.046094
O5 C1	1.395469	1.448594	1.415	0.026562
O5 C5	1.412844	1.494094	1.448	0.040625
O6 C6	1.344531	1.469531	1.425	0.0625
O6b C6b	1.389469	1.511344	1.434	0.060938
C1b C2b	1.471281	1.544719	1.508	0.036719
C1 C2	1.478344	1.548656	1.526	0.035156
C3b C4b	1.511969	1.569781	1.519	0.028906
C2 C3	1.471719	1.559219	1.51	0.04375
C2b C3b	1.532844	1.560969	1.543	0.014063
C3 C4	1.490094	1.549469	1.519	0.029688
C4 C5	1.510656	1.582531	1.513	0.035938
C4b C5b	1.481844	1.519344	1.517	0.01875
C5b C6b	1.492281	1.542281	1.504	0.025
C5 C6	1.504719	1.606281	1.518	0.050781

Measurement details

The institution details are provided in the Acknowledgements section, here only the initials and surnames of study participants are listed.

Refined white sugar from local suppliers was used in all measurements. Prior to all discussed experiments, the sugar crystals were ground using an agate mortar and pestle under a hexane layer until full hexane evaporation (≈ 15 min). Smaller grinding times lead to significant divergence of line intensities from expected values due to unsatisfactory particle statistics (Alexander et al., 1948). All measurements were performed at r.t. Naming convention for datasets is *S-instrument-sample holder-special details*.

S-vario-f (own data): measured on a Bruker D8 Advance Vario diffractometer using $\text{CuK}\alpha^1$ radiation (Ge(111) monochromator) and LynxEye 1D position-sensitive detector in transmission mode ($R=217.5$ mm), with specimen placed between Kapton foils, with rotation. Absorption correction $A(\theta) = \frac{t}{\cos\theta} \exp\left(-\frac{s}{\cos\theta}\right)$ as per International Tables for Crystallography (Prince et al., 2006) eq. 2.3.1.20, spherical harmonics of 4th order (Järvinen, 1993) used for PO correction.

S-vario-cap-vct (own data): measured on a Bruker D8 Advance Vario diffractometer using $\text{CuK}\alpha^1$ radiation (Ge(111) monochromator) and LynxEye 1D position-sensitive detector in capillary mode, with X-ray beam focusing on the detector (so-called “low intensity–high resolution” setup). The sample was placed in a 0.9 mm glass capillary and rotated; standard variable counting time strategy was employed. Absorption correction performed using Sabine approximation (Sabine et al., 1998), spherical harmonics of 2nd order (Järvinen, 1993) used for PO correction.

S-d8-zbg-v20 (own data): measured on a Bruker D8 Advance diffractometer using $\text{CuK}\alpha$ radiation (Ni filter) and LynxEye 1D position-sensitive detector in reflection mode (θ - θ Bragg-Brentano geometry, $R=300$ mm), specimen dispersed in hexane on a flat zero-background Si holder, with rotation and variable slits (irradiated zone ≈ 20 mm). Standard absorption correction employed, spherical harmonics of 4th order (Järvinen, 1993) used for PO correction.

S-ultimaIV-zbg (*dataset provided by M.G. Krzhizhanovskaya*): measured on a Rigaku Ultima IV diffractometer using CuK α radiation (Ni filter) and D/tex Ultra position-sensitive detector in reflection mode (θ - θ Bragg-Brentano geometry, R=285 mm), specimen dispersed in hexane on a flat zero-background Si holder, with rotation and 0.5 mm fixed slits. Standard absorption correction employed, March-Dollase PO correction in (001) direction.

S-phaser-zbg (*dataset provided by M.G. Krzhizhanovskaya*): measured on a Bruker D2 Phaser benchtop diffractometer using CuK α radiation (Ni filter) and LynxEye 1D position-sensitive detector in reflection mode (θ - θ Bragg-Brentano geometry, R=140 mm), specimen dispersed in hexane on a flat zero-background Si holder, with rotation and fixed slits. Standard absorption correction employed, spherical harmonics of 4th order (Järvinen, 1993) used for PO correction.

S-phaser-thick (*dataset provided by M.G. Krzhizhanovskaya*): same setup as described for **S-phaser-zbg**, with sample deposited in a standard 2 mm deep plastic holder. Standard absorption correction employed, spherical harmonics of 4th order (Järvinen, 1993) used for PO correction.

S-discover-cap (*dataset provided by I. A. Kasatkin*): measured on a Bruker D8 Advance diffractometer using CuK α^1 radiation (double-monochromated parallel beam) and LynxEye 1D position-sensitive detector in transmission geometry, R=300 mm.

S-stadiP-f (*dataset provided by E. Allig and M. U. Schmidt*): measured on a STOE Stadi P diffractometer, Ge(111) monochromator, linear position-sensitive detector, CuK α^1 radiation, in transmission geometry with specimen placed between polyacetate films, psd-step 0.2, R=130 mm. Absorption correction $A(\theta) = \frac{t}{\cos\theta} \exp\left(-\frac{s}{\cos\theta}\right)$ as per International Tables for Crystallography (Prince et al., 2006) eq. 2.3.1.20, spherical harmonics of 4th order (Järvinen, 1993) used for PO correction.

S-stadiP-cap (*dataset provided by E. Allig and M. U. Schmidt*): measured on a STOE Stadi P diffractometer, Ge(111) monochromator, linear position-sensitive detector, CuK α^1 radiation, in transmission geometry with specimen placed in a 1.0 mm capillary, psd-step 0.2, R=130 mm.

S-stadiMP-cap (dataset provided by S. Correll): measured on a STOE Stadi MP diffractometer, Ge(111) monochromator, linear position-sensitive MYTHEN detector, CuK α ¹ radiation, in transmission geometry with specimen placed in a 0.2 mm capillary, R=140 mm. The data were recorded by summing up of five identical measured ranges, each prepared by merging three recorded patterns with the same internal data point increment of 0.015° 2 θ but different starting values of 6.000, 6.005, and 6.010° 2 θ , thus giving a final data point increment of 0.005° 2 θ for the total measurement. Spherical harmonics of 4th order (Järvinen, 1993) used for PO and line width anisotropy correction.

References

- Alexander, L., Klug, H. P., & Kummer, E. (1948). *J. Appl. Phys.* **19**, 742.
- Järvinen, M. (1993). *J. Appl. Cryst.* **26**, 525–531.
- Prince, E., Fuess, H., Hahn, T., Wondratschek, H., Müller, U., Shmueli, U., Prince, E., Authier, A., Kopský, V., Litvin, D. B., et al. (2006). *International Tables for Crystallography Vol C: Mathematical, Physical and Chemical Tables*, Chester, England: International Union of Crystallography.
- Sabine, T. M., Hunter, B. A., Sabine, W. R., & Ball, C. J. (1998). *J. Appl. Cryst.* **31**, 47–51.