On the power of laboratory powder data: anisotropic refinement of C and isotropic refinement of H atoms

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Laboratory X-ray powder diffraction data can be more powerful than usually assumed. We used a standard STOE Stadi-P diffractometer, equipped with a Ge(111) monochromator and a silicon-strip detector (Mythen 1K) to record the diffractogram of naphthalene with Cu-Kα_1 radiation in transmission mode at room temperature. The measurement was performed in an almost routine manner, just with a larger 2θ range (2-100°), and an extended measurement time (8 hours). The sample was contained in a 0.7 mm glass capillary. From these data, the crystal structure of naphthalene ($C_{10}H_8$, $P2_1/c$, Z = 2) was refined with TOPAS, including a free refinement of all C and H positions, the anisotropic(!) displacement parameters of C and isotropic displacement of H atoms. All parameters were refined simultaneously. Only one restraint was applied (U_{iso}(H2) = U_{iso}(H5)). The resulting structure agrees well with single-crystal X-ray data, see Figure 1. Presumably, the remaining signals in the Rietveld plot are mainly caused by a slight spottiness.

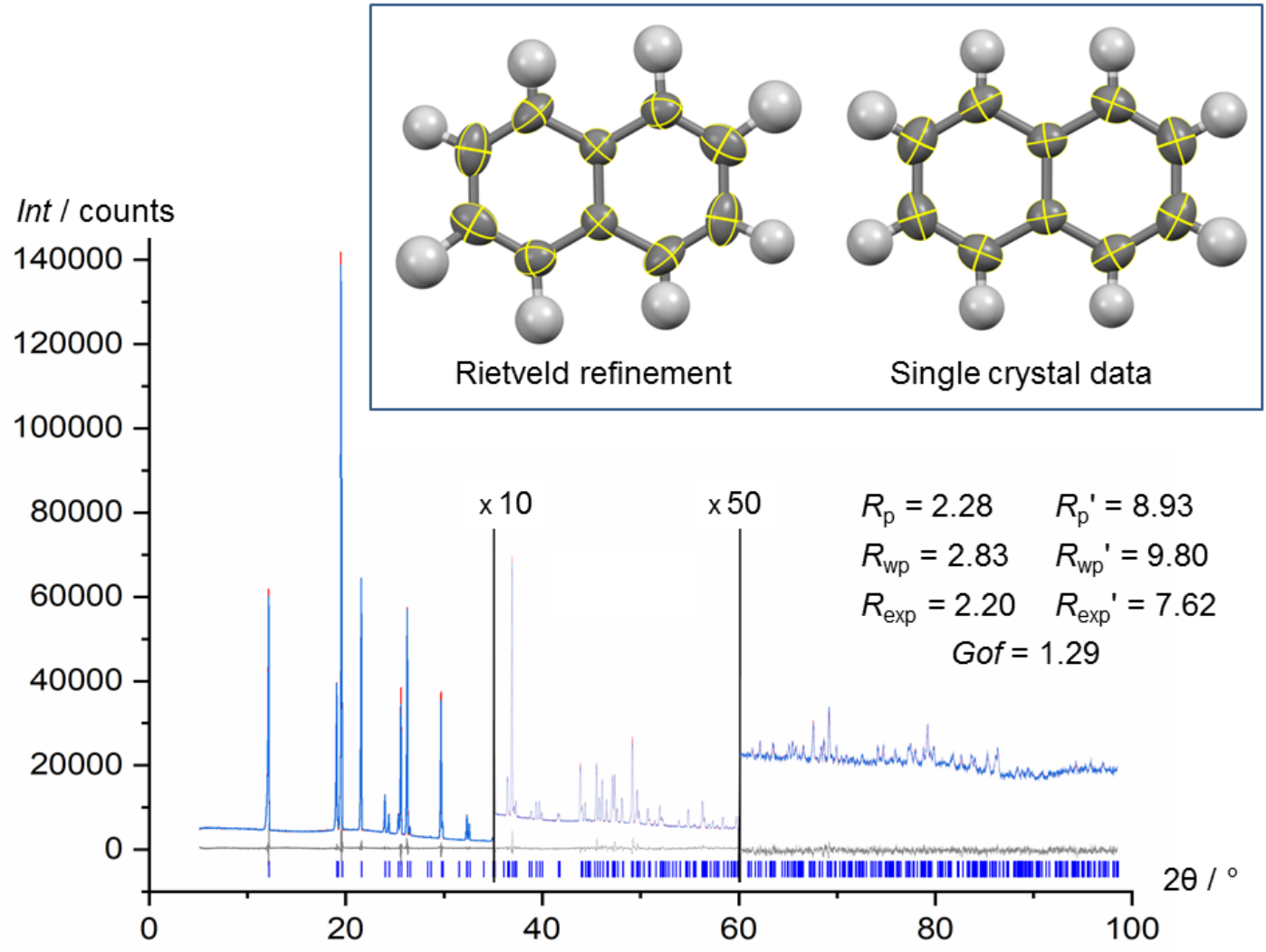


Figure 1. *Rietveld plot, and molecular structure of naphthalene from Rietveld refinement and single-crystal analysis.*

[1] J. Elsass, M.U. Piepenbring, *in preparation*