LABNOTE

ELECTRON-DENSITY DETERMINATION OF A BOROXINE COMPOUND FROM HIGH-RESOLUTION X-RAY DATA MEASURED WITH A STOE IPDS 2T

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The boroxine compound shown in Figure 1 originally served as a model compound to exemplify concepts of ringstrain and electron deficiency.[1]

Its electron-density (ED) distribution was derived from a high-resolution X-ray data set of 286975 reflections collected at 90 K on an IPDS 2T that allowed 2θ-movement to reach high regions in reciprocal space. A typical frame of the measurement is presented in Figure 2.

3082 frames with an exposure time of one minute were collected, using ω-scans with an ω increment of 0.3 degrees. To gain a better signal to noise ratio for the weak reflections at high 2θ angles, a second data set of 1932 frames with an exposure time of five minutes was collected. Both data sets were scaled together and used for the ED determination.

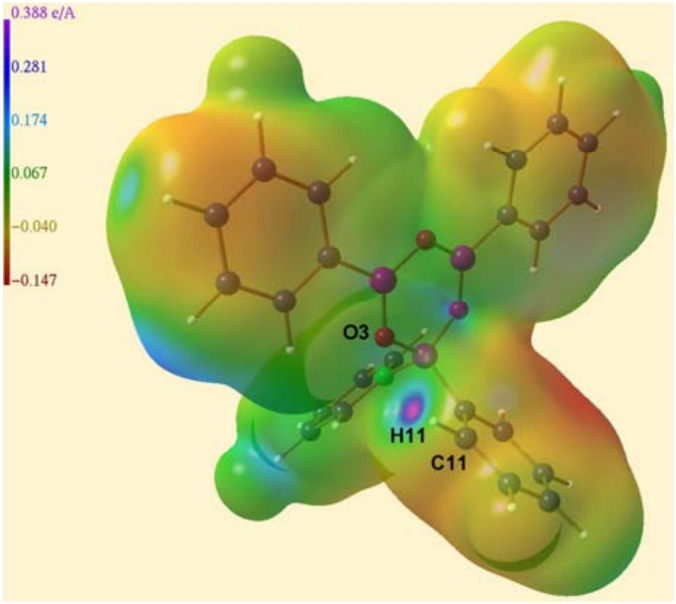

Fig. 1: Molecular structure and electrostatic potential (esp) of the examined boroxine compound.

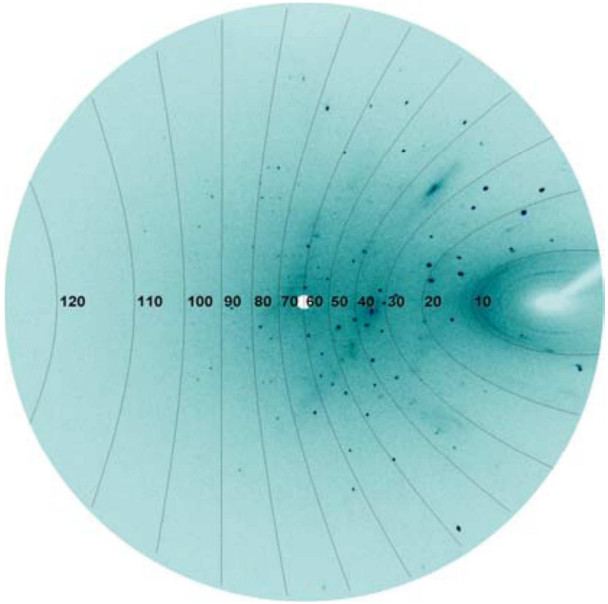

Fig. 2: Typical frame of the measurement with an IPDS 2T

Hence, a maximum resolution of $\sin\theta_{max}/\lambda$ = 1.22 Å^{-1} ($2\theta_{max}$ = 120°), an overall completeness of 97.5% and a redundancy of 9.1 could be reached. The data set, reduced to 30876 unique reflections (R_{int} = 4.1%), was properly suited to model successfully the ED distribution using the multipole formalism as implemented in XD2006[2]: 778 parameters, $wR_{mult}(F^2)$ = 1.5 %, GooF = 2.8.

Figures 1, 3 and 4 show the esp and deformation densities as first ED results. An intra-molecular hydrogen bond C11-H11…O3 could be found that exhibits a strong electrostatic nature, see Fig. 1. The deformation densities show the shape of bonding and non-bonding effects. For example, the oxygen lone pairs are clearly developed in the boroxine plane, see Fig. 4.

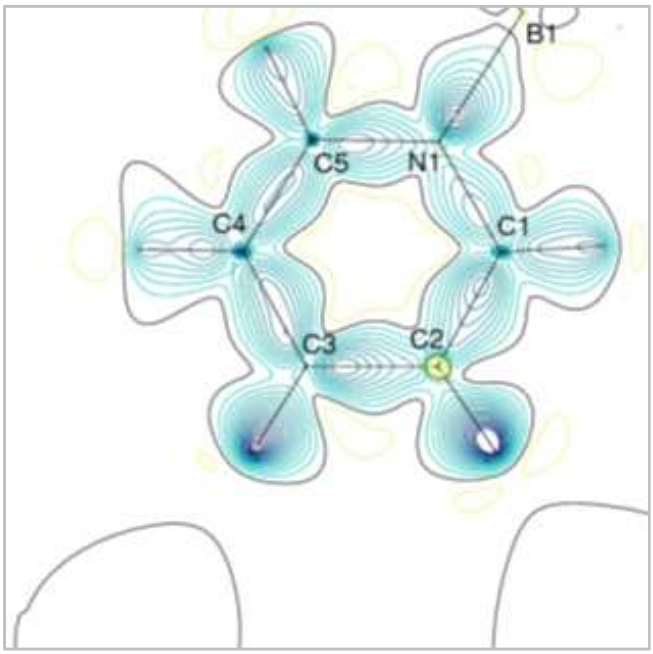

Fig. 3: Static deformation density map in the plane of the pyridine ring.

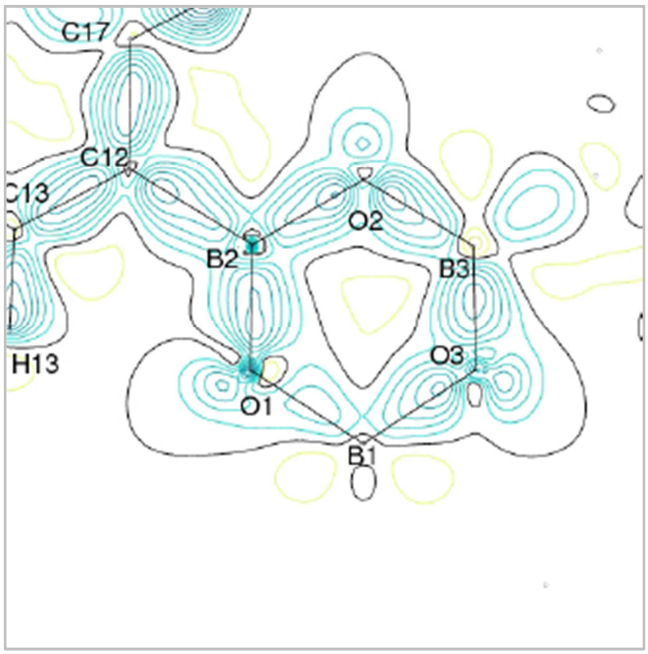

Fig. 4: Static deformation density map in the plane of the boroxine ring.

As conclusion, the STOE IPDS 2T has proven as a powerful instrument in the collection of high resolution and high quality data for electron density determination.

[1] J. Beckmann, D. Dakternieks, A. Duthie, A. E. K. Lim, E. R. T. Tiekink, J. Organomet. Chem. 2001, 633, 149.
[2] A. Volkov, P. Macchi, L. J. Farrugia, C. Gatti, P. Mallinson, T. Richter, T. Koritsanszky, XD2006