

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.

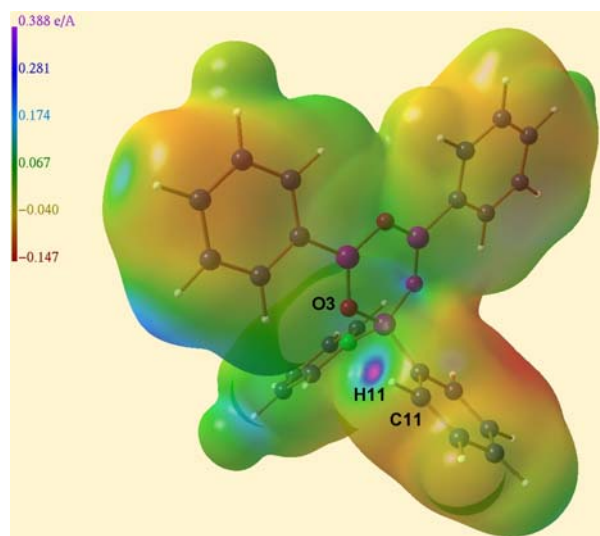


Fig. 1: Molecular structure and electrostatic potential (esp) of the examined boroxine compound.

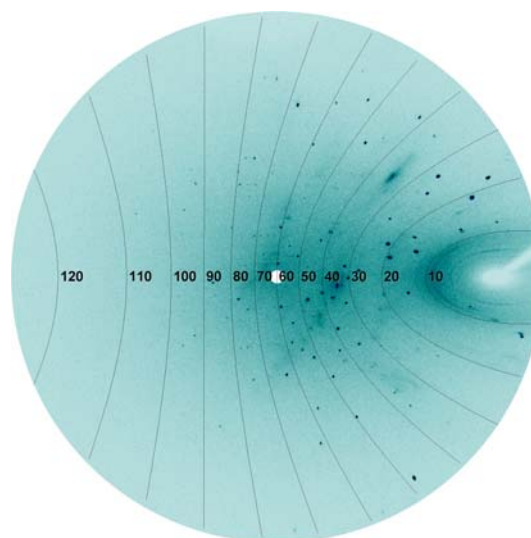


Fig. 2: Typical frame of the measurement with an IPDS 2T

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

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, 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, Fig. 4.

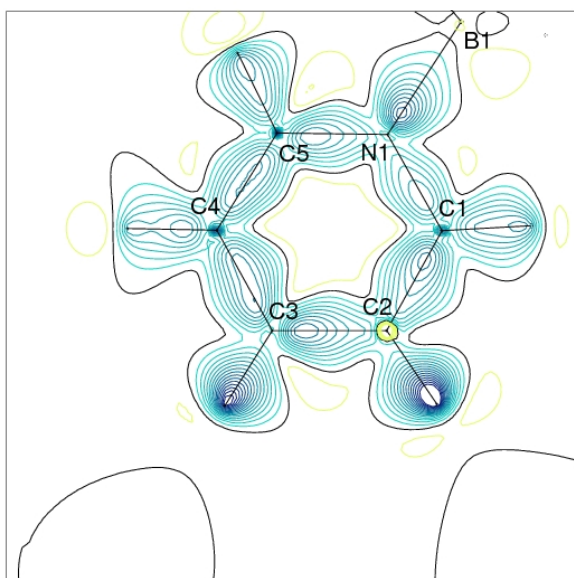


Fig. 3: Static deformation density map in the plane of the pyridine ring

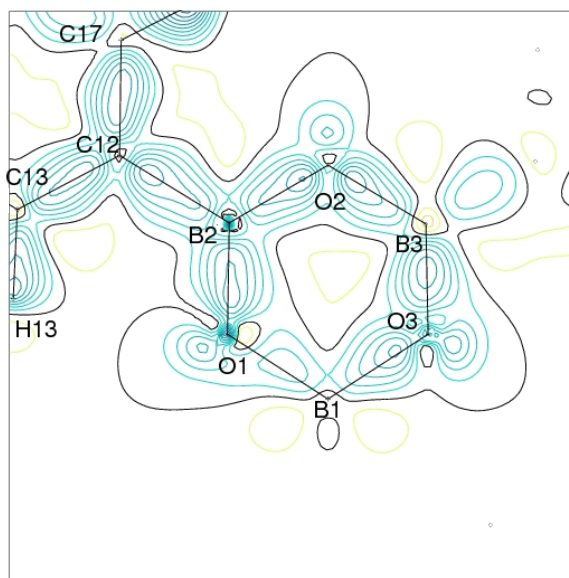


Fig. 4: Static deformation density map in the plane of the boroxine ring

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