Summary

An existing low temperature diffractometer was extended with a CCD area detector. This enables electron density determinations at 20 K within one weak, if crystals with excellent quality are available.

A Huber 4-circle goniometer equipped with a closed cycle cryostat (APD) was used. The extension with a Bruker-Apex detector was possible through DFG funding. This required a new goniometer control, that is now completely included in the Bruker software. The obtainable data quality was not sufficient with former used Beryllium cylinder, because of its highly structured scattering. To obtain the required data quality for electron density determinations, a Kapton vacuum cylinder was constructed at the beginning of reconstruction. This cylinder is built by 0.125 mm Kapton film only. This result in strongly reduced background. In addition Kapton film produces nearly unstructured scattering. Till now only Mo-Kα radiation was used for electron density determinations. Ag radiation would also be possible, but is problematic because of the lower intensity. Graphite is used in the monochromator. To enhance the primary intensity a focusing collimator from XOS was installed short ago.

In scope of this work the electron densities of Strychnine, a [1.1.1]-Propellane-derivate, Adenosinmonophosphat and a Dithiolate-Zink-complex were experimentally evaluated.

The comparative study on Strychnine shows, that four different measurements with different instruments can give well reproducible electron densities. These measurements show a small deviation to theoretical calculations.

The experimental electron density determination of a [1.1.1]-Propellane-derivate was only possible at 100 K using synchrotron. All bonds of this highly strained system could be characterised. A bond between the bridgehead atoms was located, what justifies the description as an inverted carbon atom. This bond was predicted by theoretical calculations since a while, but is characterised in detail experimentally first.

The electron density determination of Adenosinmonophosphat shows problems in the multipol model for phosphorus atoms. In comparison to a 100 K measurement the lower temperature of 25 K seems to be of great advantage for changing of the model. Only the 25 K measurement together with modified models to roughly compares with theoretical calculations. The modelling of phosphorus atom remains problematic an further improvement would be helpful.

Hydrogen parameters obtained by a neutron diffraction experiment from Dr. Kloosters could be used in the refinement of the Dithiolat-Zink-complex. The Zink atom shows a remarkable deformation of the 3d and 4s shell, that could not be deducted ea-

sily by the coordination geometry. In addition the deformation is structured in a too complicated way for the available multipolar model.

Measurements of numerous substances were performed in the last two years to determine their experimental electron densities with the new developed instrument. This includes some amino acid formiates, Tripeptides, Opiods, as well as Strychnine, Thymidine, Adenosinmonophosphate and one metal complex at temperatures around 20 K. In addition DL-Serin was measured at three different temperatures (298, 100 and 25K).