

06.2-25 THE ELECTRON DENSITY IN BERYLLIUM USING 0.12A γ -RADIATION, N.K. Hansen, Laboratoire de minéralogie et cristallographie. Université de Nancy 1 France, J.R. Schneider, Hahn-Meitner - Institut für Kernforschung, Berlin, Germany, W.H. Pearson and W.B. Yelon, University of Missouri research reactor, Coulumbia, Missouri, U.S.A.

Single crystal structure factors for Be have been measured at room temperature on the γ -ray diffractometer at the University of Missouri at Columbia. The experiment was done on a box-shaped sample with approximate dimensions of 2 x 2 x 2 mm, which is the same sample as was used by Larsen, Lehmann & Mericale (Acta Cryst. A36, 159-163, 1980). The experimental setup is such that the crystal is fully bathed in the homogeneous γ -ray beam. All Bragg reflections corresponding to $\sin\theta/\lambda \leq 1.1\text{\AA}^{-1}$ have been measured, and the intensities for 35 equivalent groups were obtained with sufficient accuracy for further analysis. These data are treated as X-ray diffraction measurements: Due to the very low background level, the integrated intensities are very easily obtained from the step scanned profiles.

Absorption in the sample, mainly caused by incoherent scattering, is weak: Transmission coefficients vary between 0.953 and 0.960. An advantage of using short wavelength (0.12A) radiation is that secondary extinction will be of little importance.

Using the reflections with $\sin\theta/\lambda \geq 0.65\text{\AA}^{-1}$, a least squares refinement of the vibrational amplitudes leads to values in close agreement with the neutron diffraction analysis. In addition to a more detailed presentation of the analysis of the experimental structure factors, charge density maps based on these will be shown.

This study shows that, using a γ -ray facility like the one at the University of Missouri, accurate γ -ray and neutron diffraction studies can be made on one and the same sample.

06.2-26 ELECTRONIC DENSITY STUDY OF ACETYLENE DERIVATIVES COMPLEXED TO $\text{Co}_2(\text{CO})_6$. By F. Baert and A. Guelzim, U.E.R. de Physique Fondamentale, U.S.T.L., 59655 Villeneuve d'Ascq, France.

Preliminary study of three dicobalt hexacarbonyl acetylene complexes $\text{Co}_2(\text{CO})_6\text{R}_2\text{C}_2$ (Baert, Guelzim and Coppens, Acta Cryst., submitted) shows that the metal-acetylenic ligand distances decrease with increasing electronegativity of R while the acetylenic C-C distance increases, indicating the strengthening of the Co-C_{ac} bonds to be due to increased back donation. Electronic density study where $\text{R} = \text{C}(\text{CH}_3)_3$ (Gregson, Howard and Baert, in preparation) and $\text{R} = \text{CF}_3$ is presented in order to understand the interactions between the acetylene derivatives and the $\text{Co}_2(\text{CO})_6$ fragment.

The deformation density calculated with X-N and X- X_{HO} formalisms at a resolution of 0.90\AA^{-1} ($I > 1\sigma(I)$) shows three peaks of different heights around the Co atoms. The acetylenic bond is largely bent with an important peak located outside the line connecting the two carbon atoms.

Multipole refinements with different models has been done using the Stewart modified by Coppens formalism.

The best results have been obtained with the $4s^2$ incorporated in the core and the $3d^7$ represented by the monopole 1. There is good agreement between the model dynamic maps and the experimental ones. The d-orbital occupancies have been calculated from the multipole parameters. In each model the d_{z^2} orbital (along the Co-Co bond) is slightly depopulated relative to the spherical d^7 configuration in agreement with the long metal-metal distance (2.47 Å). Results from the study of the complex with the more electronegative substituent CF_3 will be compared to those of the previous compound.

06.2-27 THE ELECTRON DENSITY DISTRIBUTION OF THE OXALIC ACID MOLECULE IN VARIOUS CRYSTALLINE ENVIRONMENTS By S. Harkema, G. van Hummel and D. Feil, Chemical Physics Laboratory, Twente University of Technology, Enschede, the Netherlands

The oxalic acid molecule forms strong O-H...O hydrogen bonds to neighbouring molecules in oxalic acid dihydrate, oxalic acid urea 1:1 and oxalic acid urea 1:2 (Dam, Harkema & Feil, Acta Cryst. B39 (1983) 760; Harkema & ter Brake, Acta Cryst. B35 (1979) 1011; Harkema, Bats, Weijenberg & Feil, Acta Cryst. B29 (1973) 143). The three complexes differ in the weak hydrogen bonds, crystal packing and space group.

Accurate X-ray diffraction data have been collected for the three compounds. Neutron diffraction data on the first two compounds provided information on the positional and thermal parameters. Hirshfeld analyses of the electron density distributions were performed. Difference densities for the three compounds are compared. The electron density distribution of the oxalic acid molecule does not seem to depend much on the difference in crystalline environment in the different complexes. The features of the strong hydrogen bond are in agreement with a Hartree-Fock-Slater calculation using an extensive basis set of Slater orbitals.