

16.2-5 RECENT DEVELOPMENTS IN CRYSTAL STRUCTURE ANALYSIS WITH SYNCHROTRON RADIATION INVOLVING A 5-CIRCLE DIFFRACTOMETER. By V. Kupcik, M. Wendschuh-Josties, A. Wolf, R. Wulf, Mineralogisch-Kristallographisches Institut der Universität, V.M. Goldschmidt-Str. 1, D-3400 Göttingen, FRG

Using the properties of synchrotron radiation, such as a continuous spectrum with high intensity in a wide spectral range and low divergency, measurements for example on extremely small crystals (R. Bachmann, H. Kohler, H. Schulz, H.-P. Weber, V. Kupcik, M. Wendschuh-Josties, A. Wolf, R. Wulf, *Angew.Chem.Int.Ed.Engl.* 22 (1983) No.12), distinction of atoms with similar scattering power (V. Kupcik, M. Wendschuh-Josties, A. Wolf, R. Wulf, *HASYLAB Jahresbericht 1983*, 165 pp) and phase determination by use of anomalous scattering are possible.

On the other hand these properties make a special experimental apparatus necessary. A single crystal diffractometer was installed in the HASYLAB and modified for the special conditions inherent with the use of synchrotron radiation. The following system was developed in the last two years:

The desired radiation and suppression of higher harmonics is obtained using a tunable fixed exit monochromator which can be adjusted to compensate for the slightly varying position of the synchrotron radiation beam. This is accomplished most efficiently by using a double crystal monochromator in which both crystals can be independently adjusted. Due to the high intensity of the synchrotron radiation, parts of the apparatus must be cooled and shielded.

Due to the high polarisation of the synchrotron radiation the diffractometer is operated in the vertical position. Its application can be further increased by rotating the diffractometer around the beam. The angle resolution can be adjusted at increments of  $10^{-3}$  ( $\approx 4''$ ). Previous systems can not be directly used in this manner without modifications of both electrical and mechanical functions.

Since the radiation varies in position, polarisation and intensity the beam must be continually monitored.

The detector used should be selective for specific wavelengths in order to suppress background effects (fluorescence radiation, higher harmonics, etc.). In this respect, a solid state detector is most efficient but in practice difficult to employ as a result of the necessary cooling system. On the other hand, a position sensitive detector will reduce considerably the time for data collection.

16.2-6 A SELF TESTING DIFFRACTOMETRIC ELECTRONIC. By M. Steifa and W. Saenger, Institut für Kristallographie, Freie Universität Berlin, Takustr. 6, D-1000 Berlin 33.

The commercial development of an intelligent stepping motor control system served as a starting point and enabled the construction of a new diffractometer control system with the following advantages:

- The motor motion and angle evaluation are performed independently allowing a check after every motor movement
- the control system gives no discrepancies when the electronics and mechanics are operating error free
- four computer independent angle displays for the arcs,  $2\theta$ ,  $\omega$ ,  $\chi$ ,  $\phi$
- minimal computer burden because of the independent arc positioning
- no need for microprocessors
- no loss of speed while operating the stepping motors in the half step mode
- the electronics are adaptable to other computer systems
- every Eulerian cradle, to whose motor an encoder can be attached, may be used
- is ideal for the updating of older systems.

16.2-7 EXPERIMENTAL DETERMINATION OF TRIPLET PHASES FOR A NON-CENTROSYMMETRIC STRUCTURE: L-ASPARAGINE. By H. Billy, H. Burzlaff and K. Hümmer, Institut für Angewandte Physik, Lehrstuhl für Kristallographie, Loewenichstr.22 Universität Erlangen-Nürnberg, FRG.

It was shown by theoretical and experimental investigations (H.J. Juretschke, *Physics Lett.* (1982) 92A, 183; K. Hümmer and H. Billy, *ECM8-Liège* (1985), paper 4.03-0) that informations on structure invariants even for non-centrosymmetric structures can be derived by special scanning procedures through multiple diffraction positions.

As it is difficult to realize these conditions using conventional single crystal diffractometers a new six circle diffractometer was constructed. This device contains two circles with axes perpendicular to each other for the detector and four axes for the crystal. The first crystal axis is parallel to the first detector axis in  $\omega$ - $2\theta$ -relation. Perpendicular to  $\omega$  a second axis for  $\psi$ -rotation is installed, this axis bears an Eulerian cradle with motions  $\chi$  and  $\phi$ . Thus an arbitrary scattering vector  $h$  can be aligned to the  $\psi$  axis and a complete  $\psi$ -scan is allowed. The device will be demonstrated in the Non-Commercial Exhibition.

Experimental results will be compared with phases calculated from the refined crystal structure.