12.X–8 APPLICATION OF STRUCTURE REFINEMENT BY THE RIETVELD METHOD TO CRYSTAL CHEMISTRY. BY A.W. Hewat, Institut Laue-Langevin, BP156X, Grenoble 38042 FRANCE.

The use of the Rietveld method of structure refinement has become common over the past ten years for both neutron and X-ray powder diffraction. What more then can be done ? Firstly, the instrument resolution continues to increase, extending powder techniques further into the realm of what was conventional crystallography. This will be illustrated by results for the resonance structure of terephthalic acid, with the new high resolution diffractometer D2B at the ILL compared to pulsed neutron machines (P. Fischer, P. Zolliker, A.W. Hewat & J. Jorgensen, J. Solid State Chem. (1984) submitted). With neutrons, powder diffraction has now largely taken over all such relatively simple structural studies. With X-rays, and especially with synchrotron sources, the resolution can be even higher, and the structures more complex. Secondly, structures are now routinely studied as a function of temperature - the investigation of "the" crystal structure at standard temperature and pressure is not enough. This will be illustrated by a temperature dependent study of the dynamic Jahn-Teller effect for Cu(II)-oxygen bonding in Tutton's salts (B.J. Hathaway & A.W. Hewat, J. Solid State Chem. (1984) in press). Thirdly, the precision of the structures obtained, especially by neutron powder diffraction, now compares well with standard single crystal techniques, and allows comparison and calculation of bonding effects. This precision will be further illustrated by a study of the electronic and magnetic transitions in the Fe^{++}/Fe^{+++} minerals Ilvaite and Magnetite, using Zachariasen and Brown-Shannon valence sum rules (Subrata Ghose, A.W. Hewat & M. Marezio, Physics & Chem. Minerals (1984) in press). Finally, the posibilities for direct methods of structure solution is mentioned.

12.1–2 SHORT TIME X-RAY POWDER DIFFRACTION USING SYNCHROTRON RADIATION. BY K.Kosten and H.Arnold, Institut für Kristallographie der TH Aachen, Germany.

At HASYLAB, Hamburg, a De Wolff monochromator and a Guinier camera is installed in order to obtain diffraction patterns of phase transitions and chemical reactions. The shortest exposure time obtained was 10 sec. Low temperature devices and high temperature furnaces are available. Several phase transitions and chemical reactions have been studied. As examples the hydration of $CaSO_4 \cdot 1/2\ H_2O$ to gypsum and the decomposition and phase transitions of $Na_2MoO_4 \cdot 2H_2O$ are shown.

12.1–1 FILM READER PROGRAM FOR GUINIER POWDER PATTERNS. By T. Evans, M. K. Hanafey, and C. M. Foris, Central Research and Development Dept., E. I. du Pont de Nemours & Company, Experimental Station, Wilmington, Delaware 19898, U.S.A.

A computer program has been developed for collecting digitized intensity/position data from x-ray powder diffraction films obtained with a Guinier-type focusing camera. This program utilizes the capabilities of the Optronics P-1700 Photomation and an Advance Electronic Design (AED) Model 512 color terminal and is operating on a Digital Electronics PDP 11/60 (RSX-11M) computer. Through a series of interactive commands the absorbance data for a specified area of the film (e.g., a narrow center strip) can be read, stored and displayed. The AED 512 screen display reproduces, or artificially enhances, the film image. Both hardware and software magnification (zoom) aid cursor positioning of points to define a line, according to a least-squares fit, through the center of curvature of the reflections recorded on the film. A specified number of absorbance data values surrounding the defined line are then averaged at each vertical scan position. Finally, a disk file of averaged absorbance and film position (mm, film length) is created. This data file is used for 2Θ calculation (internal standard reference) and peak-finding. The program is not specific to Guinier-type films and has been used to collect absorbance vs. position (2-dimensional) data for other types of diffraction films, such as those obtained with fibers and polymer materials.

12.1–3 THE AMORPHOUS CHARACTER AND PARTICLE SIZE DISTRIBUTIONS OF POWDERS PRODUCED WITH THE MICRONIZING MILL FOR QUANTITATIVE X-RAY POWDER DIFFRACTOMETRY. By B.H. O'Connor and W-J. Chang, Department of Applied Physics, Western Australian Institute of Technology, Bentley, Western Australia.

The rapid rise in the popularity of rod micronizing mills for powder preparation in quantitative analytical x-ray powder diffractometry (XRPD) has lead to the present critical examination of material produced by the method.

Micronizing mill specimens of α-quartz were produced for a range of milling times under wet- and dry-grinding conditions.

Tests were conducted to infer the amorphous content of the specimens with a procedure similar to that described by Altree-Williams, Byrnes and Jordon (Analyst (1981), 106, 69). Measured and calculated Bragg-Brentano XRPD intensities for milled α-quartz and reference corundum specimens, and measured XRPD data for material acid-washed after milling, were used to provide an estimate of amorphous content.

Particle size distributions were estimated using scanning electron microscopy (SEM) and sedimentation techniques.

The results show that the micronizing mill can produce XRPD specimens of α-quartz for assay work which adequately satisfy particle size criteria, and for which negligible amorphous content is introduced by grinding.