

13.3-1 SMALL-ANGLE NEUTRON SCATTERING FROM POLY(ethylene terephthalate).\* By G.D. Wignall, National Center for Small-Angle Scattering Research, Oak Ridge National Laboratory, and K. McAlea, J.M. Schultz, University of Delaware, and K.H. Gardner, E.I. duPont de Nemours Co.

Small-angle neutron scattering (SANS) experiments were performed on poly(ethylene terephthalate) (PET) with the Oak Ridge National Laboratory 30-m SANS instrument. An improved sample preparation technique which produces void free films was developed. Single chain dimensions in amorphous undrawn films were measured for different label (deuterated chain) concentrations. The SANS radius of gyration was compared to the ideal chain value and the SANS molecular weight was compared to the GPC molecular weight. The extent of the ester interchange reaction, both in the solid and the melt (temperatures from 200 to 300°C), was measured by following changes in the scattering curves over a period of time. Preliminary experiments on drawn PET samples were also performed as background for future work.

\*Research sponsored by U.S. Army Research Office Grant No. DAAG-29-81-C-0187, and the National Science Foundation Grant No. DMR-77224459 through Interagency Agreement No. 40-636-77 with the U.S. Department of Energy under contract DE-AC05-84-OR21400 with Martin Marietta Energy Systems, Inc.

13.5-1 SINGLE CRYSTAL NEUTRON DIFFRACTION STUDIES OF  $\text{Na}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$  AND  $\text{K}_2\text{C}_2\text{O}_4 \cdot \text{H}_2\text{O}$  AT 123 K AND 60 MW. By Berit F. Pedersen, University of Oslo, P.B.Box 1033, Blindern, 0315 Oslo 3, Norway and Åke Kvick, Chemistry Department, Brookhaven National Laboratory, Upton, New York 11973, USA

Previous investigations (Pedersen & Pedersen, Acta Chem. Scand. (1964) 18, 1454-1468 and (1967) 21, 779-790) by X-ray and NMR techniques gave a dihedral angle of 180.0° for  $\text{H}_2\text{O}_2$  in the sodium compound and 102.5° in the potassium compound. To get more detailed information on the molecular conformation, in particular the hydrogen atom positions of  $\text{H}_2\text{O}_2$ , a 3-dimensional neutron diffraction study of the two compounds have been undertaken at the HFBR reactor at 123 K. We have measured 1830 reflexions for the Na- and 2196 reflexions for the K-compound. The structures were refined by least squares methods to  $R_w(F^2)$  values of 5.1 and 4.1 % respectively, conventional  $R$  values of 3.5 and 2.2 %.

The dimensions of  $\text{H}_2\text{O}_2$  in the two compounds are:

	O-O(Å)	O-H(Å)	H...O	$\phi$	O-O-H
Na	1.4670(10)	1.0094(9)	1.5821(11)	180.0	99.95(8)
K	1.4578(6)	1.0117(5)	1.5791(6)	101.6	100.69(3)

The O-O distance is significantly longer in the planar than in the synclinal conformation.

The most interesting feature of the oxalate ion is the long C-C distance of 1.5640(10) and 1.5675(5)Å which are both significantly longer than in oxalic acid, 1.544(1)Å. We have collected 3-dimensional X-ray data for the compounds at the same temperature and are studying the experimental electron density distribution. We have also started ab initio calculations on the molecule situated in the experimentally determined crystal field.

The ND research was carried out at Brookhaven National Laboratory, and I wish to thank for the hospitality and assistance received during my stay.

13.5-2 WATER MOLECULES IN NATURAL ZEOLITES STUDIED BY NEUTRON DIFFRACTION\*. By Å. Kvick, Chemistry Department, Brookhaven National Laboratory, Upton, NY 11973 and G. Artioli, J. J. Pluth and J. V. Smith, The Department of Geophysical Sciences, The University of Chicago, IL 60637.

A series of neutron diffraction experiments using natural zeolites have been performed at the Brookhaven National Laboratory High Flux Beam Reactor in order to study the bonding of the water molecules and the cations in the zeolitic cavities. The ultimate aim of the studies is to develop models that will be useful for the understanding of reversible dehydration and ion-exchange properties of zeolite molecular sieves.

Precise structural data for natrolite, edingtonite, thomsonite and brewsterite collected in the temperature range 20K to 423K have yielded important information on the water molecules and their interaction with the cations and the framework oxygens. The hydrogen bonding is usually weak and the molecules adapt well to the local charge distribution in the framework. The O...O distances vary in the ordered zeolites between 2.68 Å to 3.02 Å with O-H...O angles between 140 to 178°. The stronger bonds are accepted by oxygen atoms bonded to both Si and Al. In brewsterite where there is Si/Al and cation disorder a complex hydrogen bond scheme is observed. The water oxygen atoms have fixed positions whereas the hydrogen atoms assume alternative positions to adjust to the varying local charge distribution caused by the disorder.

\* Research carried out under contract with the U.S. Department of Energy and supported in part by its Office of Basic Energy Sciences.

13.5-3 SINGLE CRYSTAL STRUCTURE DETERMINATION OF  $\text{ReO}_3$  AT 15 KBAR. By R. W. Alkire, Allen C. Larson, P. J. Vergamini, Los Alamos National Laboratory, Neutron Scattering Group, Los Alamos, NM 87545 and J. Schirber and B. Morosin, Sandia National Laboratory, Albuquerque, NM 87123.

$\text{ReO}_3$  is a bright red metal which exhibits conductivity within a factor of six of copper at room temperature. Intense interest has developed in  $\text{ReO}_3$  due to a novel second-order phase transition in the metallic perovskite structured compound. The most unusual property of this transition is that the high pressure phase has a much larger (by a factor of seven) compressibility than the normal volume phase; this transition has been referred to as a "compressibility collapse".

The  $\text{ReO}_3$  Fermi surface above the pressure-induced transition has been derived from simple cubic band structure, and experiments designed to pinpoint the onset of the phase change as well as characterize the resulting structure and corresponding properties include: Fermi surface pressure derivative measurements at 2K, single crystal and powder X-ray studies of the lattice constant as a function of pressure at room temperature (J. E. Schirber and B. Morosin, Phys. Rev. Lett. (1979) 42, 1485-1487), de Haas-van Alphen and NMR measurements ( $^{187}\text{Re}$ ) as a function of pressure and time-of-flight neutron powder diffraction (~9 kbar) measurements (J. E. Schirber, L. J. Azevedo, A. Narath and B. Morosin, Materials Science and Engineering (1981) 47 P7-P10 J. E. Schirber and L. F. Mattheiss, Phys. Rev. B. (1981) 24, 692-697).

This massive amount of information leads indirectly to the conclusion that the  $\text{ReO}_3$  structure undergoes a tetragonal distortion of the lattice involving a