13.3–1   SMALL-ANGLE NEUTRON SCATTERING FROM POLY(ethylene terephtalate).* 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 terephtalate) (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.

13.5–2   WATER MOLECULES IN NATURAL ZEOLITES STUDIED BY NEUTRON DIFFRACTION*. By A. 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 $O\cdots O$ distances vary in the ordered zeolites between 2.68 Å to 3.02 Å with $O-H\cdots O$ 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.

13.5–1   SINGLE CRYSTAL NEUTRON DIFFRACTION STUDIES OF $Na_2C_2O_4\cdot H_2O_2$ AND $K_2C_2O_4\cdot H_2O_2$ 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 H_2O_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 H_2O_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 2 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 H_2O_2 in the two compounds are:

	O-O(Å)	O=H(Å)	H···O	φ	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.

13.5–3   SINGLE CRYSTAL STRUCTURE DETERMINATION OF 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.

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 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 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}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 ReO_3 structure undergoes a tetragonal distortion of the lattice involving a

displacement of the oxygen atoms from the linear O-Re-O chains of cubic ReO_3 to a "hinged" arrangement at the oxygens. This necessitates a doubling of the lattice and a change from simple cubic to a body centered geometry.

Until now, due to a large X-ray form factor mismatch, design limitations in X-ray single crystal pressure cells and a limited range in accessible pressure for the neutron powder measurement, no direct evidence of the high pressure ReO_3 structure has been obtained. For the first time, we have been able to obtain direct structural information at 15 kbar using the newly designed single crystal neutron diffractometer at WNR. This was accomplished by developing a novel geometry for the high pressure experiment which maximizes the available information using time-of-flight techniques and does not require the sample to be remounted in order to solve the structure (good for high symmetry systems > orthorhombic, with possible extensions to lower symmetry groups). This method consists of a cylindrical high pressure cell mounted along the beam axis (i.e. mounted on the incident beam collimator) so that the incident beam enters through the bottom of the cell. The sample is isolated inside a hydrostatic fluid chamber and the diffracted beam is scattered at 90° to the incoming beam.

As predicted, ReO_3 does undergo a lattice doubling with a hinging of the oxygen atoms. At ~15 kbar, the Re-O-Re angle is 165.4° (Re-Re-O 7.3°) with almost no change in the Re-O distance from the zero pressure structure. The "compressibility collapse", therefore, results from a rotation of Re-O octahedra.

13.5—4 ANTIFERROMAGNETIC STRUCTURE OF $LaFeO_3$ FROM HIGH RESOLUTION NEUTRON DIFFRACTION. By T. Peterlin-Neumaier and E. Steichele, Fakultät für Physik der Technischen Universität München, E 21, D-8046 Garching, BRD.

The rare earth orthoferrites belong to the orthorhombically distorted perovskite structures (space group D_{2h}^{16} - Pbnm) and show an antiferromagnetic ordering of the iron ions at room temperature. The determination of the Fe^{3+} moment direction from the intensities of magnetic Bragg peaks may become difficult from powder measurements when some of the relevant reflexions overlap. This problem is most serious with the light rare earth perovskites, as here the difference of the cell edges a and b is very small. For $LaFeO_3$ the a/b ratio is only 1.002, compared to 1.0175 for $PrFeO_3$. With a high resolution time-of-flight neutron diffractometer (E. Steichele and P. Arnold, Phys. Letters (1973) 44A, 165) and a carefully prepared sample the orthorhombic splitting could be resolved.

The sample was prepared from a 1:1 molar mixture of La_2O_3 and Fe_2O_3, pressed to pellets with force of 15 tons, subsequently heated twice to 1420° C with grinding of the pellets in between. The diffractometer resolution could be improved to $\Delta d/d \approx 4 \times 10^{-4}$ for the (101) and (011) reflexions, which are purely magnetic peaks.

Diffraction measurements were performed in the fixed backscattering geometry between $\lambda = 2d = 9.2$ Å and $\lambda = 2d = 3.4$ Å, where the experimental resolution was 1×10^{-3}. For higher order reflexions no magnetic contributions could be observed due to the reduced magnetic form factor of the iron ion. Individual peaks were fitted with a combined Gauss-Lorentz function (Lorentzian in the peak and Gaussian in the tails), which approximated well the expected triangular resolution function. The result of such a least squares fit for the (101) and (011) reflexions is shown in the figure below. The experimental linewidth is about twice the instrumental resolution and the relative splitting of the clearly separated peaks is 1.2×10^{-3}.

For a pure G_x - type configuration mostly found in rare earth perovskites one would expect an intensity ratio R = {011} : {101} = 3 : 1, whereas our experimental result is $R_{exp} = 2.32 \pm 0.12$. This implies that the spin must be canted with respect to the x-axis. From the experimental result two extreme orientations can be derived: With the moment in the xz-plane the deviation from the x-axis would be $\approx 27 \pm 4°$ and with the moment in the xy-plane the deviation from the x-axis would be $\approx 19 \pm 4°$. In order to design the spin a unique orientation in space the intensities of other magnetic peaks are being evaluated. We also find that the magnetic peaks are by about 20% wider than the purely nuclear peaks probably due to finite magnetic domain sizes.

T. PETERLIN-NEUMAIER AND E. STEICHELE JULY 1983
$LaFeO_3$