09.4-53 THE CRYSTAL STRUCTURE OF SODIUM TRIAQUA (ETHYLENEDIAMINETETRAACETATE) SAMARATE (III) PENTAHYDRATE BY NEUTRON DIFFRACTION. By DW. Engel, Physics Department, University of Durban-Westville, Durban, South Africa, and TE. Koetzle, Chemistry Department, Brookhaven National Laboratory, Upton, New York 11973, USA.

The crystal structure of $Na^{+}[Sm(C_{10}H_{12}N_2O_8).3H_2O]^{-}.5H_2O$ has been determined from low temperature neutron diffraction data at a wavelength of 1.300 Å at 37 K. The space group is Fdd2 with a = 19.428(10), b = 35.334(15) and c = 12.014(6) and Z = 16. The final unweighted R value based on F^2 is 0.058 for 1795 independent reflections, the refinement being partially anisotropic with 449 parameters.

All the hydrogen atoms have been located revealing an extensive network of hydrogen bonds. Two of the water molecules around the twofold axis are disordered. A model for the disorder is proposed with two distinct positions with occupancy 0.5 for each of the two molecules. The disordered water molecules form an integral part of the hydrogen bonding scheme.

09.4-54 CRYSTAL AND MOLECULAR STRUCTURE OF DINITRATOBIS(TRI-n-BUTYLPHOSPHINE OXIDE)DIOXOURANIUM(VI), By John H. Burns, Chemistry Division, Oak Ridge National Laboratory, Oak Ridge, TN 37830.

Tri-n-butylphosphine oxide (TBPO) is typical of a class of neutral organophosphorous compounds which are versatile solvent-extraction reagents for uranium (Blake, Baes and Brown, Ind. Eng. Chem. (1958), 50, 1763), both alone and in synergistic combination with alkyl phosphoric acids. As part of a study of this extraction process, structural studies are being made of compounds representing the extraction complexes. The title compound is the first of these to be completed.

Crystals suitable for X-ray diffraction were prepared by reacting a 2:1 mixture of TBPO and $UO_2(NO_3)_2 \cdot 6H_2O$. The structure was determined from 1648 X-ray intensities by the heavy-atom method. Least-squares refinement of all atomic positions and anisotropic thermal parameters resulted in an agreement index of 0.047.

The symmetry is monoclinic, C2/c, with unit-cell parameters of a = 8.496(2), b = 29.630(7), c = 14.063(3) Å and β = 94.92(2)°. Each cell contains two centrosymmetric molecules. The uranyl ion is linear with a U-O distance of 1.734(8)Å, and there are six nearly coplanar O atoms around its equator. These are provided by alternating nitrate ions and phosphine oxide molecules at U-O distances of 2.53Å and 2.35Å, respectively. The P coordination is tetrahedral; two of the attached butyl groups are in a trans conformation, one is cis. There are no unusually short distances indicative of bonding between molecules.

Research sponsored by the Division of Chemical Sciences of the U.S. Department of Energy under contract (W-7405-eng-26) with the Union Carbide Corporation.

09.4-55 THE DISORDERED STRUCTURE OF THE ADDITION COMPOUND OF EUROPIUM PERCHLORATE WITH DIPHENYLPHOSPHINAMIDE. By E.E. Castellano, G. Oliva, G. Vicentini and L.R.F. De Carvalho, Institute of Physics and Chemistry of São Carlos, USP, 13560 São Carlos, S.P., Brazil.

The crystal structure of the title compound, $Eu^{3+}(ClO_4)_3^{-}.6(C_6H_5)_2NH_2PO$, has been determined as part of a series of studies on the coordination of lanthanide adducts and their optical properties (Castellano and Becker, Acta Cryst. (1981), in press). Diffractometric X-ray data gave 420 independent reflections with intensities above background. Space group and unit cell dimensions are F23 and a=20.29(1)Å, with Z=4. The Eu atom was placed on the special position 1/4, 1/4, 1/4. All other atoms were located from difference synthesis except those of the phenyl groups which were found by translating and rotating the groups through a set of stereochemically permissible positions around the phosphorous atoms, until a minimum R factor was obtained. Considerations on the occupancy of atoms other than Eu, P and O, and small violations to some systematic extinctions clearly indicated disorder. Constrained least squares refinement, based on the known configuration of the $(C_6H_5)_2NH_2PO$ groups (Oliva and Castellano, Acta Cryst. (1981), in press) gave a final R value of 12%. The phosphine oxygens lay in the set of special positions x, 1/4, 1/4 in a perfect octahedral configuration. In the light of this result, the occurrence of a strong fluorescence band corresponding to the transition $^5D_0$-$^7F_2$ (Carvalho, Vicentini and Zinner, J. Inorg. Nucl. Chem., (1981), in press) was interpreted as a consequence of vibronic coupling. This is further supported by the fact that the relative intensity of this band diminishes markedly from room to liquid nitrogen temperature.

Work supported by FAPESP and CNPq