09.2-34 THE LOW TEMPERATURE (-100°C) STRUCTURE OF A 'STRAPPED' PORPHYRIN. S.A. Einstein and T. Jones, Department of Chemistry, Simon Fraser University, Burnaby, B.C., Canada, V5A 1S6, and D. Dolphin, Department of Chemistry, The University of British Columbia, Vancouver, B.C., Canada, V6T 1W5.

The strapped porphyrin 7,17-diethyl-2,8,12,18-tetramethoxy-3,13-naphthalene porphyrin was prepared by "3 + 2 + 2 synthesis" of the appropriate dipyrromethane. It was expected the nine-carbon 'strap' would cause non-planarity of the porphyrin core, thus providing a model to examine the importance of doping in the metalloloporphyrin oxygenation process.

The crystals belong to space group P21/c with Z = 4. The cell dimensions at -100°C are: a = 14.774(3), b = 13.828(3), c = 17.654(2) Å, β = 103.31(1)°. Intensities were measured, at -100°C, for 312 independent reflections of which 2349 were classed observed [I > 2σ(I)] and used in refinement. The structure was solved using MULTAN, hydrogen atoms were easily located and behaved well when coordinates and isotropic thermal factors were refined. All non-hydrogen atoms were refined anisotropically yielding a final R-value of 0.036. The porphyrin core was indeed distorted; the angle between the planes of rings 1 and 3 being 112° compared to 18° between 2 and 4. The two pyrrole hydrogens are found to be attached to the "unstrapped" pyrrole rings (2 and 4). Bond distances and angles have errors < 0.005 Å and < 0.4°, respectively.


Two isomers of CsH48O4 are obtained by hydrogenation of the product of the reaction of furane and acetone with Li2O in acid medium. One of them forms a complex with two Li+ ions in solution while the other does not. We have determined the crystal structure of the former. Crystal data: CsH48O4 from ethylbenzene, M = 648,7, α = 10.906(5), b = 13.356(5), c = 10.958(4), α = 96.54(3), β = 13.68(3), γ = 181.74(4). Space group P1. Intensities were measured on a Syntax P21 diffractometer with graphite-monochromatized MoKα radiation. 2430 of the 4504 reflections measured had F > 2σ(F) and were accepted as observed. The structure was solved using MULTAN80 and refined by full-matrix methods to R = 0.058.

The molecule has a non-crystallographic two-fold axis through one pair of opposing dimethylmethylene groups. The tetrahydrofurane groups have the envelope conformation with the O atom as the flap and are all cis-substituted. The free electron pairs of the 0 atoms of opposing tetrahydrofurane rings are on the same side of the molecule and could be expected to chelate to a Li+ ion.

09.2-36 THE CRYSTAL AND MOLECULAR STRUCTURES OF A NOVEL TRICYCLIC CYLINDRICAL POLYETHER IN UNCOMPLEXED AND COMPLEXED FORMS. By R.G. Battenhammer, D.N. Walba, and R.H. Richards, Department of Chemistry, University of Colorado, Boulder, Colorado 80309, U.S.A.

In the initial phase of a project directed towards the synthesis of cylindrical organic hosts possessing hydrophobic interiors, the tricyclic polyether I, containing the novel tetrahydroxymethylethylene (THME) unit, has been synthesized. The THME unit was envisioned as ideal for incorporation into such hosts because of its rigidity and lack of stereochemistry. The host I is shown to form the novel and provocative complex 1I, containing a tightly bound water molecule within a cavity composed of two K+ ions and twelve encircling oxygens. The neutral guest is believed to be disordered with the K+ ions and 12-O-bonding to the other oxygens.

The complexed host I, [K2Cs2H8O4(0.4)(PtCl3(CH3)2SO)]2-2CH2Cl2, the complexed ligand II, crystallizes in space group $P2_1/a$, $a = 10.297(6)$, $b = 25.36(2)$, $c = 12.050(4)$, $\alpha = 113.99(4)$, $\beta = 109.2(4)$, $\gamma = 109.2(4)$. The structure, solved by a combination of heavy atom techniques and direct methods, was refined as above. For 1293 observed structure amplitudes measured with MoKα radiation using 0-20 scans to 40.0 deg, the final residuals were R = 0.0156 and wR = 0.047.

The complexed ligand and the toluene solvate are both located about inversion centers. While this posed no difficulty for the uncomplexed ligand it required that the toluene be disordered. The compound crystallizes with four of the twelve oxygens oriented such that they are within the cage, four outside and four between the inside and outside. Examination of models suggested flexibility for the compound and it appeared that it could coordinate to a metal through the ether oxygen atoms or possibly through the double bonds.