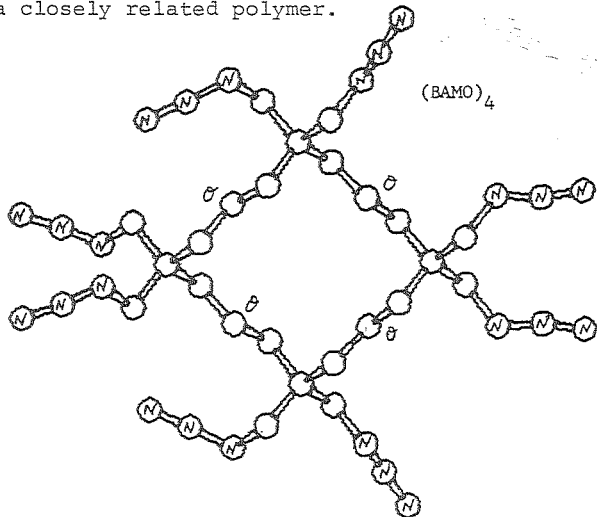


Finally, a study of the X-ray diffraction of the linear (as opposed to cross-linked) polymer of BAMO is in progress. Low angle ($2\theta \leq 40^\circ$) powder patterns can be obtained from untreated samples, indicating a tendency towards crystallization. Partially aligned fibrous samples have been obtained by drawing out films which were heated to the melting (softening) point. The fiber patterns, which are limited to a few strong reflections, appear to be monoclinic (or possibly triclinic) with a fiber repeat of $c = 4.6 \text{ \AA}$. This short repeat is consistent with a non-helical planar zigzag backbone, as is found in some forms of fibrous polyoxetane, a closely related polymer.



09.2-26 THE CONFORMATION OF HETEROCYCLIC SPIRO COMPOUNDS. X. THE STRUCTURE OF N-(γ -HYDROXYPROPYL)-GRANATANINE-3-SPIRO-5'-HYDANTOIN MONOHYDRATE ($C_{13}H_{23}N_3O_4$).

By F. Florencio, P. Smith-Verdier and S. García-Blanco. Departamento de Rayos X, Instituto Rocasolano, CSIC, Serrano 119, Madrid (6), Spain.

This structure has been determined to check the conclusions drawn by Avendaño and Bellanato (1980) from IR studies which showed that the N atom belonging to the two piperidine rings was a quaternary N. In all other compounds of this series this N atom is ternary. The structure was solved by use of the program MULTAN (Main, Woolfson, Lessinger, Germain, Baggio and Declercq, 1978). The refinement was carried out by several cycles of full-matrix least squares with isotropic and anisotropic thermal parameters. The H atoms were located in a difference map (except the water H atoms) and included in the refinement with isotropic temperature factors. The final R values were $R=0.052$ and $R_w=0.066$. The bicyclo nonane system adopts a boat-chair conformation.

The quaternary character of the piperidinic N deduced from IR studies has been confirmed. This quaternization is due to proton transfer from the hydantoin ring to the basic piperidine N. Consequently a "zwitterion" structure is present. This structure is stabilized by the water molecule.

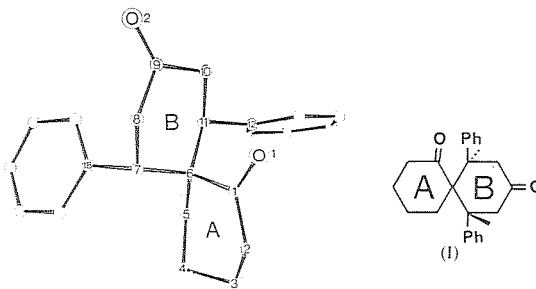
An intricate three dimensional network holds the molecules together.

09.2-27 THE CRYSTAL STRUCTURE OF 7,11-DIPHENYLSPIRO [5,5] UNDECANE-1,9-DIONE. Michael Laing, Department of Chemistry, University of Natal, Durban, Republic of South Africa.

7,11-diphenylspiro[5,5] undecane-1,9-dione(I) reacts readily with bromine to yield a variety of products, four of whose crystal structures have been determined (Sommerville and Laing, Acta Cryst., B34, (1978) 670; 672; 674; 676). While one was a simple tribromo-substitution product, three were oxygen-bridged derivatives resulting from attack by the keto oxygen (1) of ring A on the carbon atoms (9) or (10) of ring B. The non-bonded separations between these atoms in the parent compound(I) were of interest, and the crystal structure was therefore determined.

The unit cell is monoclinic, C_2 , $a = 18.52(1)$, $b = 9.770(5)$, $c = 20.24(1) \text{ \AA}$, $\beta = 101.68(5)^\circ$, $V = 3586.2 \text{ \AA}^3$. $Z = 8$, $D_m = 1.23 \text{ g cm}^{-3}$, i.e. two molecules per asymmetric unit. Data were collected on a Philips four-circle diffractometer (C.S.I.R., Pretoria) with graphite-monochromated $Mo K\alpha$ radiation for θ between 3 and 25° . Of 3275 reflections measured, 1820 were classed as observed; $I > 1.65\sigma(I)$. The structure was solved after some difficulty by Direct Methods, and refined anisotropically by block-diagonal least squares to a final R of 0.062 (H atoms included).

The two molecules in the asymmetric unit are a racemic pair. Other than the chirality, there are no major differences between the two molecules; even the torsion angles are of almost identical magnitude. The six-membered rings A and B are both slightly distorted chairs; the phenyl ring at C(7) is axial, while that at C(11) is equatorial. The non-bonded contacts between the keto oxygen on ring A and the pertinent carbon atoms on ring B are short; $O(1) \cdots C(8) = 3.31, 3.30$; $\cdots C(9) = 3.36, 3.40$; $\cdots C(10) = 2.88, 2.90 \text{ \AA}$. The analogous distances in the tribromo derivative are 3.03, 3.33 and 2.89 \AA respectively. It is quite clear that



it is these close contacts that allow the facile O-bridging to occur, and that substitution of hydrogen by bromine atoms alpha to the keto groups causes surprisingly little change to the conformation of the rings.

There are no close intermolecular contacts. However, in projection down either x or y, the molecules are related by a pseudo mirror, with the phenyl rings parallel to the plane of projection. This parallelism causes a regularity in the arrangement of the atoms and hence has a marked effect on the intensity statistics. This was a cause of difficulty in the solution of the structure. This case of a racemic pair of molecules per asymmetric unit in space group C_2 is certainly unusual, if not unique.