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THE CRYSTAL STRUCTURE OF BICYCLO[3.3.3]UNDECANE-1,5-DIOL AND THE CONFORMATION OF BICYCLO[3.3.3]UNDECANE (MANXANE).

P. MURRAY-RUST,* J. MURRAY-RUST and C. I. F. WATT†
Department of Chemistry, University of Stirling, Stirling, Scotland, FK9 4LA

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Abstract —The crystal structure of bicyclo[3.3.3]undecane-1,5-diol has been determined. It is monoclinic, $P2_1/c$, $a = 12.99(2)$, $b = 14.16(2)$, $c = 12.50(1)$ Å, $\beta = 112.42(2)°$, with two independent molecules in the asymmetric unit. One of these is disordered, but the other has almost exact C_{3h} symmetry and its conformation and precise molecular geometry agree well with previous calculations by molecular mechanics. The molecule shows considerable angle strain, having bridge angles in the range 118–121°.

Bicyclo[3.3.3]undecane,[1] **I**, is an intriguing molecule since, although it comprises cyclo-octane rings, inspection of molecular models shows that its possible conformations are not flexible, unlike those of most monocyclic 8-membered rings. One of its conformations **II** (with C_{3h} symmetry) is particularly attractive, and its resemblance to the triskelion crest (Fig. 1) of the Isle of Man suggested the trivial name 'manxane' for the parent hydrocarbon.

The conformations of manxane and its derivatives have been studied by dynamic NMR[1] and by molecular mechanics.[2] The calculations favour **II** as the most stable conformation, but indicate that even this arrangement is highly strained. Confirmation of the high ground state strain has been provided by experimental measurements of the heat of formation.[3] One structural manifestation of the strain is a flattening of the bridgehead systems, and the calculations showed that conversion of a bridgehead to a trigonal center would result in relief of strain. Enhanced cation and radical reactivity at these sites has been linked to relief of strain.[4]

Both 1-aza[5] and 1,5-diazamanxane[6] have been described. The structure of 1-azabicyclo[3.3.3]undecane has been determined[7] and provides qualitative confirmation of the molecular mechanics calculations in that the molecule has C_3 symmetry, C–C–C angles increased (up to 120° see Table 4) and there is measurable flattening at the bridgeheads. No structural data is yet available for the hydrocarbon, and as the nitrogen force field differs from that of carbon, no detailed comparison is possible.

Manxane itself is not a good subject for crystallographic investigation since it is disordered, sublimes readily, and reacts rapidly with atmospheric oxygen. We have therefore prepared a crystalline derivative by oxidising manxane with oxygen to a mixture of the bridghead peroxides and hydroperoxides and reducing the mixture to manxan-1-ol **VI** and manxan-1,5-diol **VII** (Scheme). These were separated chromatographically and, as the alcohol **VI** was found to be disordered, the crystal structure of **VII** was undertaken.

Crystallography of bicyclo[3.3.3]*undecane*-1,5-*diol* (*VII*). Large prismatic crystals were produced as below

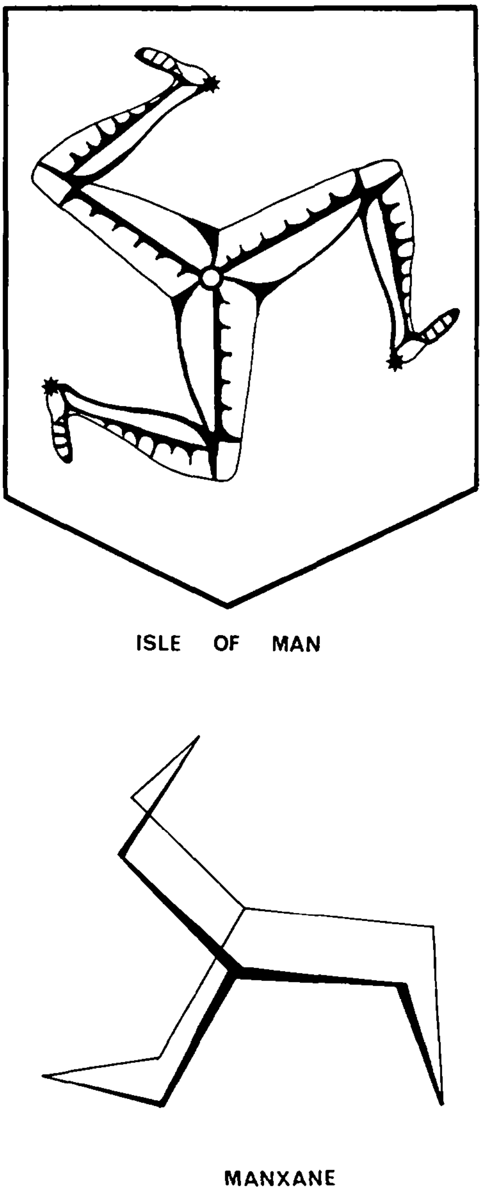

Fig. 1. The crest of the Isle of Man and its relationship to manxane.

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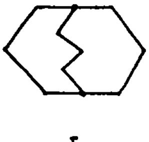
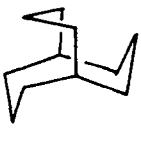
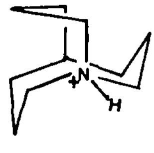
†Present address: Department of Chemistry, University of Manchester



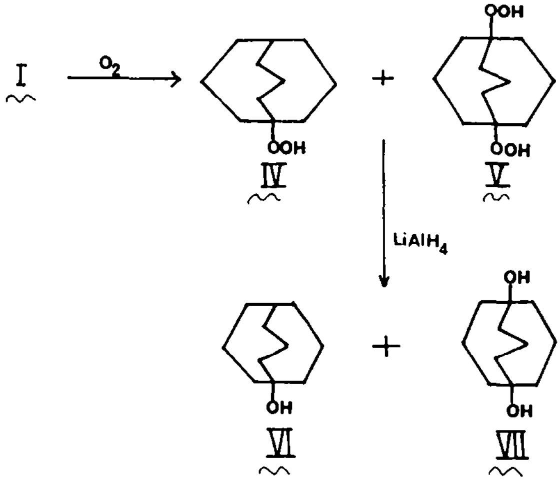
I



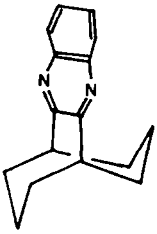
II



III



VIII



IX

and mounted on a glass fibre. No sublimation or decomposition seemed to take place. Weissenberg and precession photography gave the following information:

Crystal data. $C_{11}H_{20}O_2$, $M_r = 184.2$. Monoclinic, space group $P2_1/c$. $a = 12.99(2)$, $b = 14.16(2)$, $c = 12.50(1)$ Å; $\beta = 112.43(2)$; $U = 2124$ Å^3, $Z = 8$. Mo$K\bar{\alpha}$ radiation, $\lambda = 0.7107$ Å; $\mu = 0.43$ cm^{-1}.

Data were collected from a crystal about $0.5 \times 0.3 \times 0.3$ mm^3 on a Hilger-Watts linear diffractometer for layers $h0$–$10l$ (2969 reflexions, of which 1421 had $I \geqslant 3\sigma(I)$) and 0–$6kl$ (2328 reflexions, of which 1058 had $I \geqslant 3\sigma(I)$). After layer scales had been calculated and applied, the reflexsions were merged ($R = 0.053$ on I) to give 1762 unique data.

Structure solution by direct methods was carried out with MULTAN[8] and all non-H atoms for two independent molecules were revealed in the E-map with the highest figure of merit (25 of the first 26 peaks being interpretable as atoms). This structure could not be refined below $R = 0.30$ with isotropic temperature factors and it seemed possible that a false solution had been found. Repeated attempts over several years to find alternatives all gave this single solution. The E-map was re-examined manually and it was then seen that some of the peaks in one molecule (A) were highly distorted into thin ellipsoids. The original structure was then refined[9] with anisotropic thermal parameters for the heavy atoms and R dropped to 0.159 in a single cycle. H atoms with isotropic temperature factors were placed in geometrically calculated positions throughout (C–H = 1.08 Å) and the refinement (with unit weights and two blocks in the matrix) converged after four more cycles at $R = 0.0842$ (271 parameters). The atoms in molecule B (which had approximate C_{3h} symmetry) had normal thermal ellipsoids, but molecule A approximated to D_{3h} symmetry and the bridge atoms exhibited severe tangential anisotropy, though they were still positive definite. The anisotropy was greatest for the mid-bridge atoms (C(3), C(7) and C(10)).

It is clear that molecule A is suffering from either static or dynamic disorder. On energetic grounds (Discussion) it seemed unlikely that the model produced by refinement was chemically acceptable and disordered arrangements were considered. Disorder might arise from a mixture of different conformers (e.g. with C_s or D_3 symmetry) in the crystal at the A site, but this was felt to be unlikely since **II** appears to be the most stable conformer[2] by about 6 kcal mol^{-1} Orientational disorder of **II** seems the most probable since the molecule is globular with no strong directional packing forces. Accordingly site A was represented by two molecules (with fractional occupancies summing to unity) sharing the same local 3-fold axis but oriented in opposite directions (Fig. 2). Since this model could produce singularities in the matrix (e.g. C(2′) and C(2″) are close together) constrained refinement was used (C–C in molecule A = 1.53(2) Å). H atoms (disordered) were placed in calculated positions on molecule A and all disordered atoms were given isotropic temperature factors. The B molecule was treated normally. This

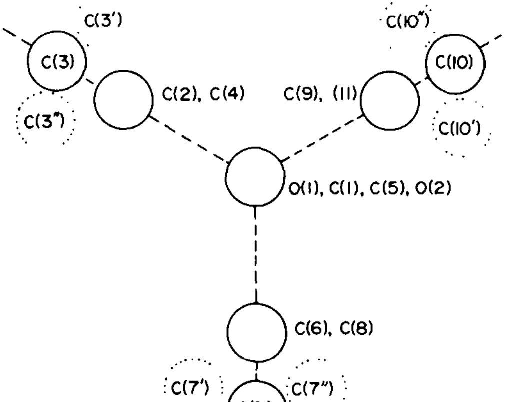


Fig. 2. Schematic view along the approximate 3-fold axis of the initial model used for refinement of disordered molecule A. The molecule had refined to a configuration with 3 approximate mirror planes (dashed lines). Atoms on the 3-fold axis (C(1), O(1), C(5) and O(2)) were given unit occupancy. Each other atom X was replaced by two part atoms X′ and X″ whose occupancies summed to unity. The mid-bridge atoms (C(3′) etc.) were displaced from the mirror plane by an amount estimated from models. The remaining bridge atoms X′, X″ (C(2′) etc.) initially both had the same position as X, but by constrained refinement with SHELX (all C–C = 1.53(0.02 Å) positions off the mirror plane were found.

model (280 parameters and 24 equations of constraint) refined to $R = 0.0950$; there were some quite large elements in the correlation matrix. Comparison of R factors shows that this disordered model is *statistically* worse than the ordered anisotropic one. For this reason tables of final parameters (Table 1†), bond lengths (Table 2), bond angles (Table 3) and torsion angles (Table 4) are based on the anisotropic model. This is not because the anisotropic model is more likely to be *physically* correct (we argue in the Discussion that a disordered model is preferable on chemical grounds) but because it better describes the scattering contribution from molecule A and hence allows better refinement of molecule B.

Table 1. Fractional atomic coordinates ($\times 10^4$) with c.s.d.s. in brackets

	x	y	z
C(1A)	4223(4)	4762(4)	7021(5)
C(1B)	9655(4)	2284(3)	6529(4)
C(2A)	5054(6)	5305(4)	8024(6)
H(2AA)	4575(6)	5675(4)	8458(6)
H(2AB)	5435(6)	5822(4)	7647(6)
C(2B)	8905(4)	1994(4)	7173(5)
H(2BA)	8061(4)	2108(4)	6590(5)
H(2BB)	9100(4)	2472(4)	7910(5)
C(3A)	5954(8)	4880(5)	8931(7)
H(3AA)	6626(8)	5385(5)	9057(7)
H(3AB)	5686(8)	4947(5)	9649(7)
C(3B)	8982(5)	0993(5)	7636(5)
H(3BA)	8878(5)	0532(5)	6925(5)
H(3BB)	8303(5)	0912(5)	7922(5)
C(4A)	6541(7)	4005(4)	9192(6)
H(4AA)	7331(7)	4147(4)	9111(6)
H(4AB)	6700(7)	3864(4)	10094(6)
C(4B)	10023(5)	0664(4)	8629(5)
H(4BA)	10178(5)	1167(4)	9336(5)
H(4BB)	9848(5)	-0016(4)	8896(5)
C(5A)	6104(4)	3088(4)	8544(5)
C(5B)	11115(5)	0572(4)	8441(4)
C(6A)	4994(5)	2765(5)	8563(6)
H(6AA)	5079(5)	2836(5)	9459(6)
H(6AB)	4923(5)	2036(5)	8326(6)
C(6B)	11659(5)	1455(4)	8781(5)
H(6BA)	12430(5)	1424(4)	8341(5)
H(6BB)	12334(5)	1427(4)	9711(5)
C(7A)	3973(7)	3181(8)	7892(14)
H(7AA)	3515(7)	3082(8)	8445(14)
H(7AB)	3677(7)	2711(8)	7172(14)
C(7B)	11298(5)	2427(4)	8520(5)
H(7BA)	11945(5)	2945(4)	8964(5)
H(7BB)	10686(5)	2436(4)	8909(5)
C(8A)	3540(5)	4042(4)	7366(6)
H(8AA)	2856(5)	3881(4)	6597(6)
H(8AB)	3258(5)	4418(4)	7970(6)
C(8B)	10720(4)	2777(3)	7293(5)
H(8BA)	10536(4)	3527(3)	7354(5)
H(8BB)	11320(4)	2728(3)	6887(5)
C(9A)	4702(5)	4384(5)	6187(5)
H(9AA)	4978(5)	5016(5)	5863(5)
H(9AB)	4026(5)	4067(5)	5521(5)
C(9B)	9837(5)	1498(4)	5785(4)
H(9BA)	9880(5)	1842(4)	5032(4)
H(9BB)	9137(5)	1050(4)	5562(4)
C(10A)	5590(12)	3738(10)	6452(7)
H(1AA)	5332(12)	3308(10)	5707(7)
H(1AB)	6274(12)	4203(10)	6462(7)
C(10B)	10870(5)	0875(4)	6286(5)
H(1BA)	10937(5)	0484(4)	5591(5)
H(1BB)	11592(5)	1359(4)	6634(5)
C(11A)	6167(5)	3084(5)	7344(6)
H(5AA)	5899(5)	2408(5)	6999(6)
H(5AB)	7054(5)	3176(5)	7510(6)
C(11B)	10970(5)	0176(4)	7254(5)
H(5BA)	10238(5)	-0236(4)	6973(5)
H(5BB)	11697(5)	-0252(4)	7389(5)
O(1A)	3430(3)	5504(3)	6396(4)
O(1B)	9059(3)	3034(2)	5724(3)
O(2A)	6908(3)	2386(3)	9220(4)
O(2B)	11770(4)	-0134(3)	9274(4)

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†Tables of structure factors have been deposited. . .

Table 2. Bond distances (Å) with e.s.d.s. in brackets

C(1A)	- C(2A)	1.515(8)
C(1A)	- C(8A)	1.517(9)
C(1A)	- C(9A)	1.501(10)
C(1A)	- O(1A)	1.471(6)
C(1B)	- C(2B)	1.537(9)
C(1B)	- C(8B)	1.518(6)
C(1B)	- C(9B)	1.526(8)
C(1B)	- O(1B)	1.464(6)
C(2A)	- C(3A)	1.415(10)
C(2B)	- C(3B)	1.520(9)
C(3A)	- C(4A)	1.426(10)
C(3B)	- C(4B)	1.519(7)
C(4A)	- C(5A)	1.521(8)
C(4B)	- C(5B)	1.528(10)
C(5A)	- C(6A)	1.520(9)
C(5A)	- C(11A)	1.534(10)
C(5A)	- O(2A)	1.456(6)
C(5B)	- C(6B)	1.537(8)
C(5B)	- C(11B)	1.530(8)
C(5B)	- O(2B)	1.460(6)
C(6A)	- C(7A)	1.400(11)
C(6B)	- C(7B)	1.533(8)
C(7A)	- C(8A)	1.398(13)
C(7B)	- C(8B)	1.511(7)
C(9A)	- C(10A)	1.410(16)
C(9B)	- C(10B)	1.526(8)
C(10A)	- C(11A)	1.423(13)
C(10B)	- C(11B)	1.531(9)

EXPERIMENTAL

(a) *Preparation of* 1,5-*dihydroxybicyclo*[3.3.3]*undecane*

Oxidation of bicyclo[3.3.3]*undecane.* Dry O_2 was bubbled through a soln of bicyclo[3.3.3]undecane (0.15 g) in pentane (50 ml) until all the hydrocarbon was consumed (9 hr glc monitoring). The mixture was then cloudy and gave a strongly positive starch iodide peroxide test. Evaporation of solvent under reduced pressure at 10° gave a white sticky mass which was taken up in dry ether (50 ml). LAH (0.2 g) was added and the mixture was refluxed for 1 hr. The mixture was then cooled and quenched by the addition of Na_2SO_4 aq. The organic layer was decanted, dried (Na_2SO_4) and evaporated to give a waxy solid (0.12 g) which on tlc examination (silica eluting with ether) showed the presence of 2 major components. Preparative tlc on silica yielded 1-hydroxybicyclo[3.3.3]undecane as the faster moving component (0.047 g) and 1,5-dihydroxybicyclo[3.3.3]undecane as the slower moving component (0.036 g). Sublimation of the latter and recrystallization from acetonitrile gave crystals m.p. 217–18° (sealed tube), m^+ 184.1469 Calc. for $C_{11}H_{20}O_2$: 184.1463. ν_{max} (KBr disc) 3370, 2920, 1420, 1452, 1420, 1359, 1343, 1270, 1240, 1226, 1181, 1162, 1090, 978, 882, 861, 810 cm^{-1} δH (CD_3OH, 300 MHz) 1.78 (m unresolved). δC^{13} (CD_3OH) 20 MHz 22.47 (t) 40.80 (t), 76.53 (s).

DISCUSSION

The structure contains two crystallographically independent molecules with two A and two B molecules being linked by a cycle of four H-bonds (Fig. 3). This results in H-bonded sheets parallel to $(\bar{1}01)$. The ring of

Table 3. Bond angles (degrees) with e.s.d.s. in brackets

C(8A)	- C(1A) -	C(2A)	114.5(6)
C(9A)	- C(1A) -	C(2A)	113.8(6)
C(9A)	- C(1A) -	C(8A)	113.6(5)
O(1A)	- C(1A) -	C(2A)	102.2(4)
O(1A)	- C(1A) -	C(8A)	105.3(5)
O(1A)	- C(1A) -	C(9A)	106.0(5)
C(8B)	- C(1B) -	C(2B)	113.7(4)
C(9B)	- C(1B) -	C(2B)	113.6(4)
C(9B)	- C(1B) -	C(8B)	113.7(5)
O(1B)	- C(1B) -	C(2B)	106.2(4)
O(1B)	- C(1B) -	C(8B)	102.4(4)
O(1B)	- C(1B) -	C(9B)	105.9(4)
C(3A)	- C(2A) -	C(1A)	123.9(6)
C(3B)	- C(2B) -	C(1B)	119.1(5)
C(4A)	- C(3A) -	C(2A)	139.4(7)
C(4B)	- C(3B) -	C(2B)	120.4(5)
C(5A)	- C(4A) -	C(3A)	124.1(6)
C(5B)	- C(4B) -	C(3B)	119.3(5)
C(6A)	- C(5A) -	C(4A)	114.3(6)
C(11A)	- C(5A) -	C(4A)	112.1(6)
C(11A)	- C(5A) -	C(6A)	114.8(5)
O(2A)	- C(5A) -	C(4A)	103.5(4)
O(2A)	- C(5A) -	C(6A)	105.1(5)
O(2A)	- C(5A) -	C(11A)	105.7(5)
C(6B)	- C(5B) -	C(4B)	114.2(5)
C(11B)	- C(5B) -	C(4B)	113.7(4)
C(11B)	- C(5B) -	C(6B)	113.5(5)
O(2B)	- C(5B) -	C(4B)	105.8(5)
O(2B)	- C(5B) -	C(6B)	103.0(4)
O(2B)	- C(5B) -	C(11B)	105.2(4)
C(7A)	- C(6A) -	C(5A)	123.5(8)
C(7B)	- C(6B) -	C(5B)	118.3(5)
C(8A)	- C(7A) -	C(6A)	140.2(8)
C(8B)	- C(7B) -	C(6B)	121.4(5)
C(7A)	- C(8A) -	C(1A)	123.0(7)
C(7B)	- C(8B) -	C(1B)	117.9(5)
C(10A)	- C(9A) -	C(1A)	125.9(6)
C(10B)	- C(9B) -	C(1B)	119.2(4)
C(11A)	- C(10A)-	C(9A)	138.3(11)
C(11B)	- C(10B)-	C(9B)	119.6(6)
C(10A)	- C(11A)-	C(5A)	123.7(8)
C(10B)	- C(11B)-	C(5B)	118.2(5)

Table 4. Torsion angles (°) (e.s.d.'s *ca.* 1)

				Molecule	
				(A)	**(B)**
C(8)	-C(1)	-C(2)	-C(3)	**62.0**	**-95.9**
C(9)	-C(1)	-C(2)	-C(3)	**-71.2**	**36.3**
0(1)	-C(1)	-C(2)	-C(3)	**175.1**	**152.3**
C(2)	-C(1)	-C(8)	-C(7)	**-74.4**	**37.9**
C(9)	-C(1)	-C(8)	-C(7)	**58.6**	**-94.2**
0(1)	-C(1)	-C(8)	-C(7)	**174.3**	**152.0**
C(2)	-C(1)	-C(9)	-C(10)	**60.8**	**-94.9**
C(8)	-C(1)	-C(9)	-C(10)	**-72.6**	**37.2**
0(1)	-C(1)	-C(9)	-C(10)	**172.1**	**148.9**
C(1)	-C(2)	-C(3)	-C(4)	**15.4**	**68.2**
C(2)	-C(3)	-C(4)	-C(5)	**-17.8**	**-66.9**
C(3)	-C(4)	-C(5)	-C(6)	**-58.1**	**93.4**
C(3)	-C(4)	-C(5)	-C(11)	**74.7**	**-39.0**
C(3)	-C(4)	-C(5)	-0(2)	**-171.8**	**-154.0**
C(4)	-C(5)	-C(6)	-C(7)	**75.3**	**-38.1**
C(11)	-C(5)	-C(6)	-C(7)	**-56.2**	**94.5**
0(2)	-C(5)	-C(6)	-C(7)	**-172.0**	**-152.4**
C(4)	-C(5)	-C(11)	-C(10)	**-61.3**	**95.0**
C(6)	-C(5)	-C(11)	-C(10)	**71.3**	**-37.8**
0(2)	-C(5)	-C(11)	-C(10)	**-173.3**	**-149.6**
C(5)	-C(6)	-C(7)	-C(8)	**-25.9**	**-68.4**
C(6)	-C(7)	-C(8)	-C(1)	**24.0**	**68.4**
C(1)	-C(9)	-C(10)	-C(11)	**17.2**	**69.7**
C(9)	-C(10)	-C(11)	-C(5)	**-16.8**	**-68.3**

four H-bonded O atoms is not planar, and has approximate S_4 symmetry. The angles in the H-bonding scheme are sketched in Fig. 3(b) and suggest that the H one e.g. O(2A) is aligned towards O(1A). There are no short C C contacts which could distort the observed geometries.

Molecule B has near-perfect non-crystallographic C_{3h} symmetry, (Figs. 4 and 5) in agreement with this being the conformation of lowest energy. The observed bond angles follow the trends observed in **III** and suggested by force-field calculations for **II**, and they are compared in Table 5. It can be seen that agreement is good, with perhaps a hint that the force field produces values for C–C–C angles slightly too large at the bridgehead and too small in the centres of the bridges. However, librational effects may be important in the crystallographic data and these will tend to increase the apparent angles in the bridges, so that overall the force field provides a good model for the manxane system. The torsion angles are probably a more stringent test of the usefulness of the force field but, unfortunately, these were not published for the calculations. Nevertheless, the general effects discussed by Chang and Schleyer[2a] can be seen clearly in Fig. 5 where the distortions from an unstrained model are shown. A manxane molecule with tetrahedral bond angles can be constructed with no angle strain but there are severe H...H repulsions [(about 1.3 Å)] between neighbouring bridges. The torsion angles (*ca* 90°) in the bridges are also not optimum. By flattening the bridges somewhat (or "straightening the knee" in the Manx crest!) the repulsions are lessened and the torsion angles approach 60°. In the present X-ray structure (B molecule) the H...H distances between bridges are in the range 1.95 Å–2.05 Å, very similar to those in of cyclododecane-1,6-diol[10] where the same triangular arrangement occurs. The torsion angles are likewise more favourable (Table 4), but this can only be achieved at the expense of some angle strain. By comparing molecules with a bridged cyclooctane ring we can see an increase in angle strain with size of bridge. In bicyclo[3.3.1]nonane **VIII** and its derivatives the conformation is chair–chair and the angles in the bridges are normal (about 111–112°).[11] In the bicyclo[3.3.2]decane derivative **IX**,[12] however, the cyclooctane ring is boat-chair (as in the present structure) and the bridge angles are 116–117°, whilst in the present skeleton (Table 5) they are 120°.

Molecule A approximates to D_{3h} symmetry with nearly planar bridges (Fig. 4). However, Chang and Schleyer[2a] have calculated that this conformation is 45 kcal mol^{-1} less stable than that with C_{3h} symmetry and it is therefore likely that molecule A is disordered in the crystal. The nature of this disorder has not been satisfactorily revealed by the refinement but, since the activation energy for interconversion of enantiomeric C_{3h} conformers of **I** is found to be 11 kcal/mol^{-1}, it is unlikely that this dynamic process occurs in the crystal. It is probable that since the molecule is globular it can take up different orientations with little difference in

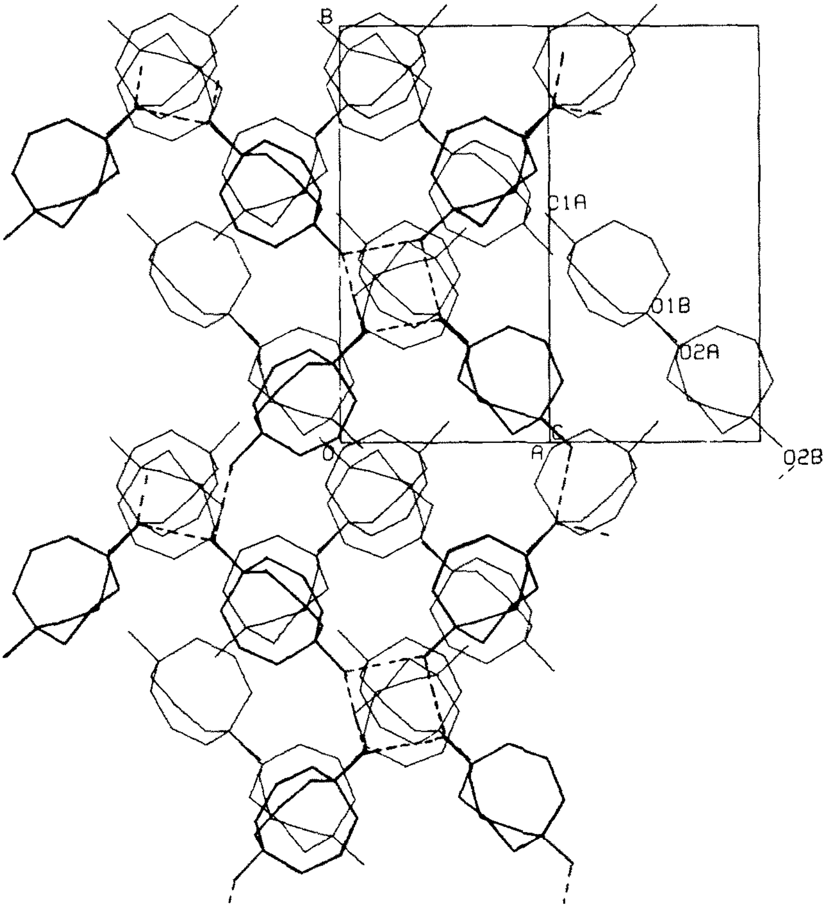


Fig. 3(a). A view perpendicular to (101) of the crystal packing showing the hydrogen-bonded sheets of molecules. The intra-sheet hydrogen bonds (shown for one sheet only as dashed lines) form four-membered rings, details of which are given in Fig. 3(b). The sheets (one of which is shown in heavy outline with hydrogen bonds) have *pseudo*-tetragonal symmetry and are related by symmetry to give a *pseudo* body-centred arrangement where molecules on one sheet fit into holes in the sheets above and below.

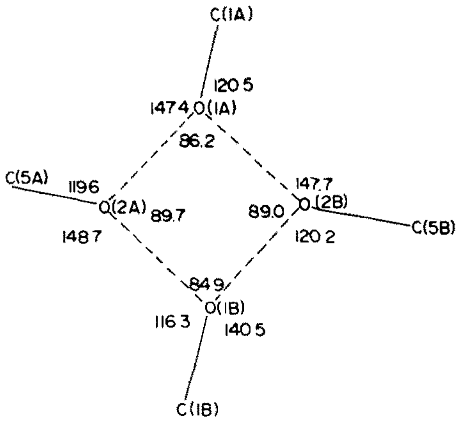


Fig. 3(b). The hydrogen bonding scheme. Bond lengths are O(2A)–O(1A) ($1 - x$, $y - \frac{1}{2}$, $1\frac{1}{2} - z$) = 2.761(6) Å; O(2B)–O(1A) ($1 + x$, $\frac{1}{2} - y$, $\frac{1}{2} + z$) = 2.756(6) Å; O(2B)–O(1B) ($2 - x$, $y - \frac{1}{2}$, $1\frac{1}{2} - z$) = 2.809(6) Å; O(2A)——O(1B) (x, $\frac{1}{2} - y$, $\frac{1}{2} + z$) = 2.772(6) Å.

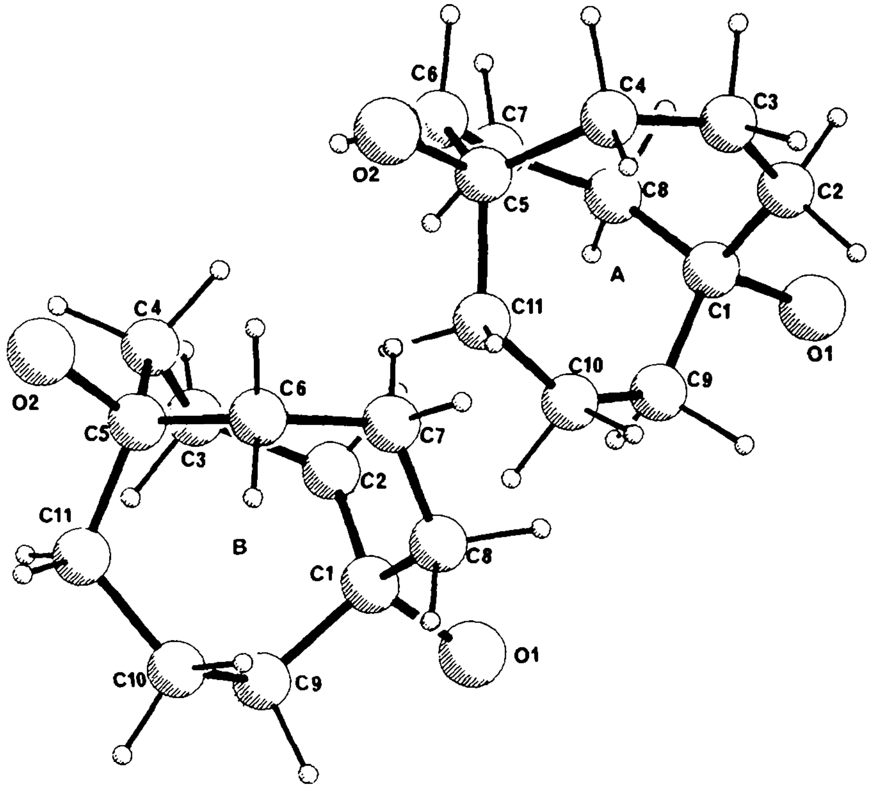


Fig. 4. The two independent molecules of bicyclo[3.3.3]undecane-1,5-diol in the crystal. Molecule B is ordered and has well defined C_{3h} symmetry. Molecule A, though almost certainly disordered, has been refined to a configuration with near D_{3h} symmetry.

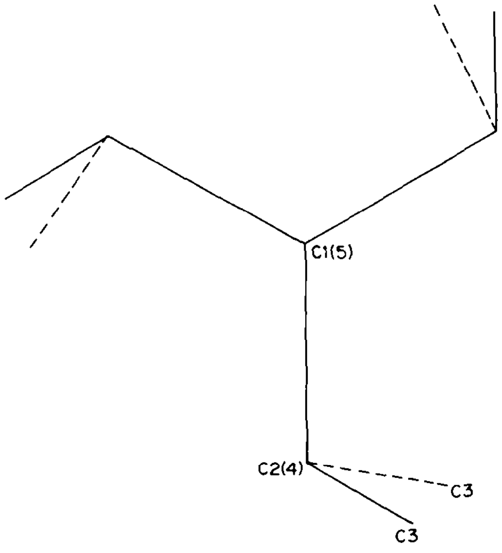
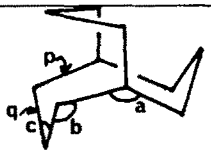


Fig. 5 View down threefold axis of manxane skeleton in (a) Molecular model (with tetrahedral carbon atoms), dotted lines. Dihedral angle of "thigh" to "shin" = 96°. (b) In the ordered (B) molecule of manxane diol, solid lines, dihedral angle = 121°.

Table 5. Comparison of bond lengths and angles in bicyclo[3.3.3]undecane systems. Column (i) represents values observed in the carbon skeleton of **VII** (this work, B molecule only). Column (ii) represents those from the X-ray analysis of **III**. Column (iii) represents those calculated by Chang and Schleyer.[2a] Column (iv) Average of (i) and (ii).



	(i)	(ii)	(iii)	(iv)
a(°)	113.6, 113.6 113. 7	113.5, 114.8 113.5	115.2	113.8 (5)
b(°)	119.1, 119.3 118.3, 117.9 119.2, 118.2	117.4, 118.3 118.7	118.8	118.5 (6)
c(°)	120.4, 121.4 119.6	119.5, 119.6 120.1	118.3	120.1 (7)
p (Å)	1.518, 1.526 1.537	1,536, 1.547 1.550	1.542	1.536 (12)
q (Å)	1.519, 1.520 1.511, 1.533(6) 1.526, 1.531	1.523, 1.510 1.505, 1.516 1.522, 1.498	1.535	1.518 (10)

intermolecular interactions, and that several orientations are present. (This type of disorder is probably also present in the crystals of **I** and **VII**).

We would like to dedicate this structure to the memory of the late Professor William Parker who first synthesised and named the manxane system.

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