

Related literature. A view of the complex with the atomic numbering scheme is depicted in Fig. 1. The final atomic fractional coordinates* with their standard deviations and equivalent isotropic thermal parameters (Hamilton, 1959) are given in Table 2. Selected bond lengths and bond angles are given in Table 3. Fig. 2 shows the contents of the cell. The packing differs from that observed for the complex formed by triphenylarsine oxide and 4-nitrophenol (Lariucci, de Almeida Santos & Lechat, 1986) in that it consists of alternate layers of NP and TPPO molecules parallel to the *ab* plane. This difference is attributed to the solvent used for crystallization and is reflected in the plate shape of the crystals, whereas complexes grown in less-polar or non-polar solvents usually have more uniform dimensions (Lariucci, de Almeida Santos & Lechat, 1986; Etter & Baures, 1988).

The molecular parameters of TPPO and NP in the complex are comparable to those reported for the

* List of structure factors, anisotropic thermal parameters, bond distances and angles, and fractional H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55092 (20 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CD0076]

free molecules (Bandoli, Bartolozzo, Clemente, Croatto & Panattoni, 1970; Spek, 1987).

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Structure of 2,5:3,4-Dianhydro-D-altritol

BY J. GABRIEL GARCIA, RONALD J. VOLL, FRANK R. FRONCZEK AND EZZAT S. YOUNATHAN*

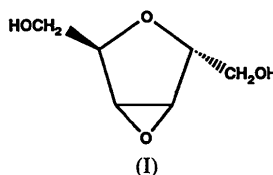
Departments of Biochemistry and Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1806, USA

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Abstract. 3,6-Dioxabicyclo[3.1.0]hexane-2,4-dimethanol, C₆H₁₀O₄, *M_r* = 146.1, orthorhombic, *P*2₁2₁, *a* = 7.6209 (2), *b* = 9.1292 (3), *c* = 9.6135 (5) Å, *V* = 668.8 (1) Å³, *Z* = 4, *D_x* = 1.451 g cm⁻³, λ(Mo *K*α) = 0.71073 Å, μ = 1.15 cm⁻¹, *F*(000) = 312, *T* = 298 K, *R* = 0.029 for 1280 observations with *I* > 3σ(*I*) (of 1695 unique data). The tetrahydrofuran ring has the envelope conformation, ^o*E*, with *P* of 94.3° and τ_{*m*} = 24.0°. C atoms deviate from their best plane by ± 0.006 (1) to 0.010 (1) Å, and the O atom lies 0.331 (1) Å from that plane. The epoxide O atom is *syn* to the tetrahydrofuran O atom. Each hydroxy group is involved in intermolecular hydrogen bond-

ing both as donor and acceptor. The two hydrogen bonds have O...O distances of 2.743 (1) and 2.729 (1) Å, and angles about H of 166.3 (12) and 172 (2)°, respectively.

Experimental. The title compound (I) was prepared as described in the literature (Guthrie, Jenkins, Waters, Wright & Yamasaki, 1982) from 2,5-anhydro-D-mannitol.



* Author to whom correspondence should be addressed.

Suitable crystals deposited from the reaction flask and had m.p. 349–351 K; a clear, colorless crystal with dimensions $0.30 \times 0.33 \times 0.45$ mm was used for data collection on an Enraf–Nonius CAD-4 diffractometer with Mo $K\alpha$ radiation and a graphite monochromator. Cell dimensions were determined from setting angles of 25 reflections having $13 > \theta > 10^\circ$. The ω - 2θ scans were designed for $I = 50\sigma(I)$, subject to maximum scan time of 180 s, with scan rates varied from 0.32 to $3.30^\circ \text{ min}^{-1}$. Two octants of data having $1 < \theta < 35^\circ$, $0 \leq h \leq 12$, $0 \leq k \leq 14$, $-15 \leq l \leq 15$ were measured and corrected for background, Lorentz and polarization effects. Three standard reflections (200, 040, 002) increased in intensity by 2.4% during data collection, and a linear correction was applied. No absorption correction was made. A total of 3260 data was measured. The two equivalent octants were averaged ($R_{\text{int}} = 0.013$) yielding 1695 unique data. Systematic absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd indicated space group $P2_12_12_1$. The structure was solved by direct methods using *MULTAN80* (Main *et al.*, 1980) and refined by full-matrix least squares based on F , using data for which $I > 3\sigma(I)$, weights $w = 4F_o^2[\sigma^2(I) + (0.02F_o^2)^2]^{-1}$ using the *MoIEN* programs (Enraf–Nonius, 1990), scattering factors of Cromer & Waber (1974), and anomalous coefficients of Cromer (1974). The absolute configuration was assumed to correspond to that of the starting material. Heavy-atom coordinates were refined with anisotropic thermal parameters; H-atom coordinates were located by ΔF synthesis and refined with isotropic thermal parameters. Final $R = 0.029$ for 1280 observed data (0.047 for all 1695 data), $wR = 0.034$ and $S = 1.677$ for 132 variables. Maximum shift in the final cycle was 0.01σ , maximum residual density was 0.22, minimum was $-0.18 \text{ e } \text{\AA}^{-3}$. An extinction coefficient $g = 1.7(3) \times 10^{-6}$, where the factor $(1 + gI_c)^{-1}$ was applied to F_c . The fractional coordinates of the title compound are given in Table 1. Fig. 1 is a perspective drawing showing the atom numbering. Fig. 2 illustrates the unit cell. Bond distances and angles, and selected torsion angles are presented in Table 2.*

Related literature. Structures of 2,5-anhydro-D-mannitol (Watkins, Abboud, Voll, Koerner & Younathan, 1983), methyl 3,4-anhydro-1,6-bis-*O*-(*p*-tolylsulfonyl)- β -D-tagatofuranoside (Guthrie, Jenkins,

* Lists of anisotropic thermal parameters, H-atom parameters, bond distances and angles involving H atoms, torsion angles and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55075 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HH0600]

Table 1. Coordinates and equivalent isotropic thermal parameters (\AA^2)

$$B_{\text{eq}} = (8\pi^2/3) \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	B_{eq}
O1	0.33327 (8)	0.32320 (9)	0.82080 (7)	2.79 (1)
O2	0.6565 (1)	0.4059 (1)	0.98312 (9)	3.53 (2)
O3	-0.0441 (1)	0.2881 (1)	0.87091 (8)	4.19 (2)
O4	0.3475 (1)	0.3360 (1)	0.54152 (7)	4.02 (2)
C1	0.5973 (1)	0.4705 (1)	0.8572 (1)	3.19 (2)
C2	0.5022 (1)	0.3621 (1)	0.7650 (1)	2.77 (2)
C3	0.4637 (2)	0.4256 (2)	0.6241 (1)	3.55 (2)
C4	0.2771 (2)	0.4581 (1)	0.6186 (1)	3.13 (2)
C5	0.1988 (1)	0.4101 (1)	0.75486 (9)	2.62 (2)
C6	0.0355 (1)	0.3181 (2)	0.7401 (1)	3.49 (2)

Table 2. Bond distances (\AA), bond angles ($^\circ$) and selected torsion angles ($^\circ$)

O1—C2	1.439 (1)	C1—C2	1.514 (2)
O1—C5	1.443 (1)	C2—C3	1.502 (2)
O2—C1	1.420 (1)	C3—C4	1.454 (2)
O3—C6	1.423 (1)	C4—C5	1.504 (1)
O4—C3	1.444 (2)	C5—C6	1.508 (2)
O4—C4	1.442 (1)		
C2—O1—C5	109.64 (8)	C2—C3—C4	107.59 (9)
C3—O4—C4	60.51 (8)	O4—C4—C3	59.80 (8)
O2—C1—C2	112.3 (1)	O4—C4—C5	111.71 (9)
O1—C2—C1	111.81 (9)	C3—C4—C5	107.28 (9)
O1—C2—C3	104.87 (8)	O1—C5—C4	105.13 (8)
C1—C2—C3	111.6 (1)	O1—C5—C6	108.76 (9)
O4—C3—C2	113.4 (1)	C4—C5—C6	114.05 (9)
O4—C3—C4	59.69 (7)	O3—C6—C5	112.08 (9)
C5—O1—C2—C3	-23.8 (1)	C2—O1—C5—C4	22.7 (1)
O2—C1—C2—C3	-171.6 (1)	O1—C2—C3—C4	15.2 (1)
C2—C3—C4—C5	-1.8 (1)	C3—C4—C5—O1	-12.3 (1)
O1—C5—C6—O3	69.2 (1)	C4—C5—C6—O3	-173.9 (1)

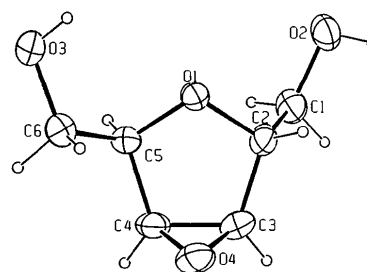


Fig. 1. ORTEP drawing (Johnson, 1965) of the molecule, representing C atoms as 40% probability ellipsoids and H atoms as circles of arbitrary radius.

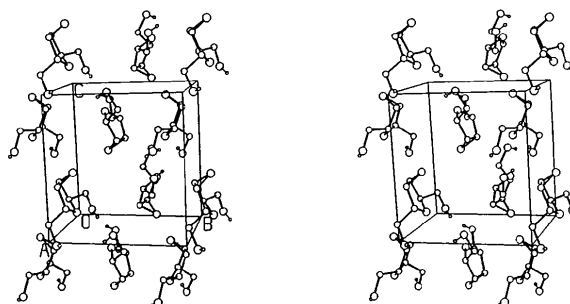


Fig. 2. Stereoview of the unit cell.

Yamasaki, Skelton & White, 1981), 6-chloro-6-cyano-1,5-dimethyl-3,8-dioxatricyclo[3.2.1.0^{2,4}]-octane (Cossu, Viani, Lapasset, Aycard, Marfisi & Bodot, 1982), 1-(β -D-2',3'-anhydroribofuranosyl)-3-methyluracil and *O*⁶,5'-anhydro-1-(β -D-2',3'-anhydroribofuranosyl)-3,5-dimethyluracil (Marton-Merész, Kuzmann, Pelczar, Parkanyi, Koritsanszky & Kalman, 1983), and eremofortin D (Arnoux, Pascard & Moreau, 1977) have been reported. Calculation of ring conformation parameters is described by Altona & Sundaralingam (1972).

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Structure of 1*H*-Isoindole-1,3(2*H*)-dione (Phthalimide)

BY SEIK WENG NG

Institute of Advanced Studies, University of Malaya, 59100 Kuala Lumpur, Malaysia

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Abstract. C₈H₅NO₂, *M_r* = 147.13, monoclinic, *P*2₁/*n*, *a* = 3.8036 (7), *b* = 7.643 (1), *c* = 22.818 (5) Å, β = 91.36°, *V* = 663.1 (2) Å³, *Z* = 4, *D_x* = 1.474 g cm⁻³, $\lambda(\text{Mo } K\alpha)$ = 0.71073 Å, μ = 1.91 cm⁻¹, *F*(000) = 304, *T* = 298 K, *R* = 0.038 for 770 reflections [*I* ≥ 3σ(*I*)]. The flat molecule is centrosymmetrically hydrogen bonded.

Experimental. A crystal measuring approximately 0.10 × 0.20 × 0.20 mm was mounted on an Enraf–Nonius CAD-4 diffractometer. Unit-cell parameters were fixed from 25 strong reflections in the 17 ≤ θ ≤ 19° thin shell. Intensity data were collected up to 2 θ _{max} = 50° (collection range: *h* 0–2, *k* 0–5, *l* –16–16; ω –2 θ –scan mode); 1167 reflections were collected, of which 770 obeyed the *I* ≥ 3σ(*I*) criterion. Three reflections were used to monitor the intensity and showed no significant decay of the crystal. All 11 non-H atoms were located by using the *SIMPEL* (Peschar & Schenk, 1987) direct-methods program, and then refined anisotropically. A

weighting scheme, $w = [\sigma(F)^2 + (0.02F)^2 + 1]^{-1}$ (Killean & Lawrence, 1969), was introduced. The H atoms were then obtained from a difference Fourier synthesis and refined isotropically. Refinement based on *F* converged at *R* = 0.038, *wR* = 0.040 for 120 variables refined; Δ/σ = 0.01; *S* = 0.54; $\Delta\rho_{\text{max}}$ = 0.54 (2) e Å⁻³. Scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1). All computations were performed using the *MolEN* structure determination package (Fair, 1990) on a DEC MicroVAX minicomputer. The atomic coordinates are listed in Table 1* and the asymmetric unit shown in Fig. 1. Bond distances and angles are listed in Table 2.

* Lists of structure factors, anisotropic thermal parameters, H-atom positional parameters and bond distances and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 55062 (9 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: AS0549]