

Related literature. The structural parameters of the steroid skeleton can be compared with those found for estrone (Debaerdemaeker, 1972; Busetta, Courseille & Hospital, 1973). Britton, Gleason & Glick (1981) have reported the structure of xanthocillin, an antibiotic compound containing two isocyanato groups. The structural parameters of the diethylphosphono group can be compared with those reported by Espenbetov, Chernega, Struchkov, Dzhimbaev & Abiyurov (1985).

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Structure of Hexaacetyl-D-mannitol

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Abstract. *n*-Hexyl hexaacetate, C₁₈H₂₆O₁₂, *M_r* = 434.4, orthorhombic, *P*2₁2₁2₁, *a* = 8.934 (2), *b* = 11.961 (7), *c* = 19.764 (3) Å, *V* = 2112.0 Å³, *Z* = 4, *D_x* = 1.366 g cm⁻³, λ(Mo *Kα*) = 0.7107 Å, μ = 1.09 cm⁻¹, *F*(000) = 920, *T* = 298 K, final *R* = 0.047 and *wR* = 0.048 for 1232 observed unique reflections. The planar acetate groups are all aligned approximately parallel to the (001) plane of the crystal, intramolecular interactions between them determining the detailed conformation of the central C chain.

Experimental. During an investigation of metabolites of marine organisms, the title compound was obtained after acetylation of a water soluble material extracted from the *Cystoseira Tamariscifolia* algae; m.p. 390–392 K, [α]_D + 18.6 (*c* 0.22 in CH₂Cl₂). Recrystallization from acetone. Crystal size 0.5 × 0.3 × 0.2 mm, cell dimensions from setting angles of 22 reflections in the range 8 < θ < 12°, space group by systematic absences. Data collected on an Enraf-Nonius CAD-4 diffractometer with graphite-mono-

chromated Mo *Kα* radiation, θ_{max} = 25°, ω–2θ scans, constant scan speed 4° min⁻¹, scan angle (0.90 + 0.35tanθ)°, *h* = 0 to 10, *k* = 0 to 14, *l* = 0 to 23. Three reflections (018, 402 and $\bar{4}24$) monitored periodically during data collection indicated no crystal deterioration. 1935 unique measured reflections (out of a total of 2156 reflections measured) of which 1232 with *I* > 3σ(*I*) were used in refinement, no corrections for absorption or secondary extinction.

The structure was solved by direct methods (*SHELXS86*; Sheldrick, 1985), and refined by least squares (*SHELX76*; Sheldrick, 1976), on *F*, including positional and anisotropic thermal parameters of the non-H atoms. All H atoms were added in calculated positions, the methyls being refined as rigid groups. Minimization of *w*(Δ*F*)², with *w* = 1.43/σ²(*F_o*) (*w* = 0 for two strong reflections, 051 and 006, which appear to be significantly affected by extinction), converged at *R* = 0.047, *wR* = 0.048; 289 parameters refined. Final Δ/σ < 0.3, residual densities in Fourier maps

Table 1. Atomic coordinates and equivalent isotropic displacement coefficients (\AA^2)

U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

	x	y	z	U_{eq}
C(1)	0.2872 (8)	0.8197 (5)	0.6813 (4)	0.0549 (25)
C(2)	0.2114 (7)	0.8919 (5)	0.7342 (3)	0.0457 (23)
C(3)	0.2808 (7)	0.8862 (4)	0.8050 (3)	0.0416 (21)
C(4)	0.1924 (6)	0.9525 (5)	0.8565 (4)	0.0468 (23)
C(5)	0.2237 (7)	0.9166 (5)	0.9304 (3)	0.0466 (24)
C(6)	0.1250 (8)	0.9710 (6)	0.9830 (4)	0.0583 (27)
O(7)	0.4445 (5)	0.8449 (3)	0.6791 (3)	0.0545 (17)
C(8)	0.5388 (8)	0.7569 (6)	0.6698 (4)	0.0557 (27)
O(9)	0.4961 (6)	0.6659 (4)	0.6678 (4)	0.1054 (30)
C(10)	0.6957 (7)	0.7942 (7)	0.6656 (4)	0.0698 (33)
O(11)	0.2155 (5)	1.0081 (3)	0.7163 (2)	0.0482 (15)
C(12)	0.0987 (8)	1.0476 (6)	0.6789 (4)	0.0520 (27)
O(13)	-0.0008 (6)	0.9872 (4)	0.6611 (3)	0.0837 (24)
C(14)	0.1112 (8)	1.1694 (5)	0.6677 (4)	0.0615 (26)
O(15)	0.2801 (4)	0.7698 (3)	0.8225 (2)	0.0468 (15)
C(16)	0.4158 (8)	0.7205 (5)	0.8358 (3)	0.0449 (23)
O(17)	0.5300 (5)	0.7716 (4)	0.8349 (3)	0.0698 (21)
C(18)	0.3943 (8)	0.6008 (5)	0.8499 (4)	0.0628 (27)
O(19)	0.0334 (4)	0.9306 (3)	0.8479 (2)	0.0456 (16)
C(20)	-0.0574 (8)	1.0208 (6)	0.8341 (4)	0.0516 (27)
O(21)	-0.0131 (5)	1.1138 (4)	0.8290 (3)	0.0642 (17)
C(22)	-0.2133 (8)	0.9814 (6)	0.8245 (4)	0.0710 (31)
O(23)	0.3776 (5)	0.9452 (3)	0.9425 (2)	0.0520 (16)
C(24)	0.4624 (9)	0.8696 (6)	0.9783 (4)	0.0582 (29)
O(25)	0.4144 (6)	0.7817 (4)	0.9957 (3)	0.0784 (21)
C(26)	0.6143 (8)	0.9142 (7)	0.9919 (4)	0.0736 (34)
O(27)	0.1361 (5)	1.0901 (4)	0.9780 (2)	0.0592 (19)
C(28)	0.0104 (9)	1.1478 (6)	0.9907 (4)	0.0587 (29)
O(29)	-0.1051 (6)	1.1026 (5)	1.0053 (4)	0.1088 (28)
C(30)	0.0312 (11)	1.2695 (7)	0.9823 (4)	0.0845 (37)

ranging from -0.22 to $0.18 e \text{\AA}^{-3}$. Elongated atomic thermal ellipsoids suggest minor structure modulation along the c axis of the crystal. Atomic scattering factors were taken from *International Tables for X-ray Crystallography* (1974, Vol. IV).

Atomic parameters are listed in Table 1; bond distances, bond angles and selected torsion angles (*PARST*; Nardelli, 1983) are presented in Table 2. The molecular structure and the atom-labelling scheme are shown in Fig. 1; Fig. 2 (*ORTEPII*; Johnson, 1976) illustrates the crystal packing and the alignment of the planar acetate groups parallel to the (001) lattice planes.*

Related literature. Crystal structure determinations of D-mannitol (Berman, Jeffrey & Rosenstein, 1968; Kim, Jeffrey & Rosenstein, 1968), DL-mannitol

* Lists of anisotropic thermal parameters, H-atom coordinates and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54847 (10 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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

C(1)—C(2)	1.516 (9)	C(12)—O(13)	1.198 (8)
C(1)—O(7)	1.438 (8)	C(12)—C(14)	1.478 (9)
C(2)—C(3)	1.532 (8)	O(15)—C(16)	1.374 (7)
C(2)—O(11)	1.435 (7)	C(16)—O(17)	1.189 (8)
C(3)—C(4)	1.513 (8)	C(16)—C(18)	1.471 (8)
C(3)—O(15)	1.435 (6)	O(19)—C(20)	1.377 (8)
C(4)—C(5)	1.548 (9)	C(20)—O(21)	1.185 (8)
C(4)—O(19)	1.454 (6)	C(20)—C(22)	1.483 (10)
C(5)—C(6)	1.511 (9)	O(23)—C(24)	1.376 (8)
C(5)—O(23)	1.437 (7)	C(24)—O(25)	1.186 (8)
C(6)—O(27)	1.431 (8)	C(24)—C(26)	1.483 (10)
O(7)—C(8)	1.361 (8)	O(27)—C(28)	1.342 (9)
C(8)—O(9)	1.154 (8)	C(28)—O(29)	1.200 (9)
C(8)—C(10)	1.473 (9)	C(28)—C(30)	1.477 (11)
O(11)—C(12)	1.363 (8)		
C(2)—C(1)—O(7)	109.8 (5)	O(11)—C(12)—C(14)	111.4 (6)
C(1)—C(2)—O(11)	111.7 (5)	O(11)—C(12)—O(13)	121.2 (6)
C(1)—C(2)—C(3)	115.1 (5)	O(13)—C(12)—C(14)	127.3 (7)
C(3)—C(2)—O(11)	105.0 (4)	C(3)—O(15)—C(16)	117.3 (5)
C(2)—C(3)—O(15)	105.2 (5)	O(15)—C(16)—C(18)	109.8 (6)
C(2)—C(3)—C(4)	112.3 (5)	O(15)—C(16)—O(17)	122.3 (6)
O(4)—C(3)—O(15)	110.1 (5)	O(17)—C(16)—C(18)	127.9 (7)
C(3)—C(4)—O(19)	109.7 (5)	C(4)—O(19)—C(20)	117.2 (5)
C(3)—C(4)—C(5)	113.3 (5)	O(19)—C(20)—C(22)	109.3 (6)
C(5)—C(4)—O(19)	103.7 (5)	O(19)—C(20)—O(21)	123.7 (6)
C(4)—C(5)—O(23)	105.3 (4)	O(21)—C(20)—C(22)	127.0 (7)
C(4)—C(5)—C(6)	115.1 (5)	C(5)—O(23)—C(24)	117.1 (5)
C(6)—C(5)—O(23)	110.0 (5)	O(23)—C(24)—C(26)	111.2 (6)
C(5)—C(6)—O(27)	109.9 (6)	O(23)—C(24)—O(25)	122.2 (7)
C(1)—O(7)—C(8)	116.6 (5)	O(25)—C(24)—C(26)	126.7 (7)
O(7)—C(8)—C(10)	111.2 (6)	C(6)—O(27)—C(28)	116.2 (6)
O(7)—C(8)—O(9)	122.0 (7)	O(27)—C(28)—C(30)	112.4 (7)
O(9)—C(8)—C(10)	126.7 (7)	O(27)—C(28)—O(29)	122.2 (7)
C(2)—O(11)—C(12)	116.7 (5)	O(29)—C(28)—C(30)	125.4 (8)
O(7)—C(1)—C(2)—O(11)	65.6 (7)	O(15)—C(3)—C(4)—O(19)	-72.4 (6)
O(7)—C(1)—C(2)—C(3)	-53.9 (7)	C(3)—C(4)—C(5)—C(6)	-173.2 (5)
C(1)—C(2)—C(3)—C(4)	-175.3 (5)	O(19)—C(4)—C(5)—O(23)	-175.6 (4)
O(11)—C(2)—C(3)—O(15)	-178.7 (4)	C(4)—C(5)—C(6)—O(27)	-56.2 (7)
C(2)—C(3)—C(4)—C(5)	159.8 (5)	O(23)—C(5)—C(6)—O(27)	62.5 (7)

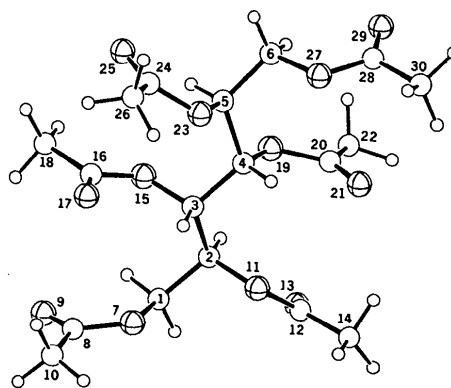


Fig. 1. Molecular structure of D-mannitol hexaacetate with the atom-labelling scheme used. The O atoms are indicated by crossed circles.

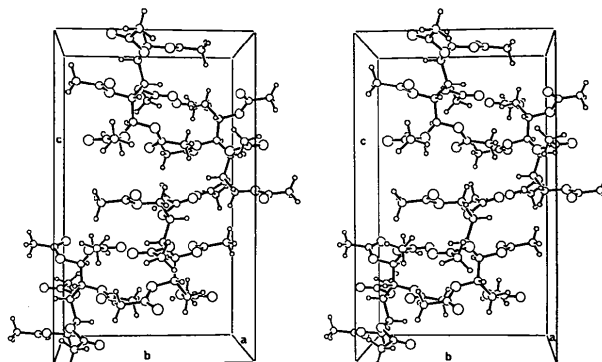


Fig. 2. Stereoview of the crystal packing down the a axis.

(Kanters, Roelofsen & Smits, 1977), and a very small number of their acyclic derivatives, e.g. D-dibromomannitol (Simon & Sasvari, 1973) have been reported. A tetraacetate cyclic derivative of D-mannitol has also been reported (Cameron, Cordes & Grindley, 1977). The detailed conformation of the title compound is determined primarily by steric and stereoelectronic (Bürgi, Dunitz & Shefter, 1974) intramolecular interactions between the carbomethoxy substituents.

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Structure of 2-[(3-Nitrophenylimino)methyl]phenol

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Abstract. C₁₃H₁₀N₂O₃, $M_r = 242.23$, orthorhombic, $Pca2_1$, $a = 26.579$ (2), $b = 3.946$ (4), $c = 10.756$ (2) Å, $V = 1128.1$ (1) Å³, $Z = 4$, $D_x = 1.426$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 0.10$ mm⁻¹, $F(000) = 504$, $T = 298$ K, $R = 0.0731$ for 985 observed reflections [$I > 2.5\sigma(I)$]. The two phenyl rings are rotated slightly about C—N with a torsion angle of 6.6 (2)°. The C—N distance [1.281 (8) Å] is indicative of a double bond while the N atom is intramolecularly hydrogen bonded to the hydroxyl group with N⋯H = 1.8 (1) Å and N⋯O 2.657 (9) Å.

Experimental. The *N*-(3-nitrophenyl)salicylideneamine was prepared by a previously reported method

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(Srivastava & Chauhan, 1977). Preliminary data obtained from Weissenberg and Buerger precession photographs yielded approximate cell dimensions and showed orthorhombic symmetry, space group $Pca2_1$ or $Pcam$ from systematic absences $0kl$, $l = 2n + 1$; $h0l$, $h = 2n + 1$. Data collection on a crystal $0.3 \times 0.2 \times 0.2$ mm was performed on a $P2_1$ diffractometer in $2\theta/\theta$ mode ($0 < h < 26$, $0 < k < 4$, $0 < l < 13$) to $2\theta_{\text{max}}$ of 52.0°. Variable scan speed of 5.0–29.3° min⁻¹ was used. Lattice parameters refined using 30 reflections in the range $25 < 2\theta < 35^\circ$. Standard reflection (4 $\bar{1}$ 2) checked every 50 reflections: no significant deviation. The data were corrected for Lorentz and polarization effects. 1384 reflections were collected, 1186 unique ($R_{\text{int}} = 0$), of which 985 observed reflections with $I > 2.5\sigma(I)$ were