the glide plane. All non-H atoms were refined with anisotropic thermal parameters and H atoms were placed in calculated positions and allowed to ride. An absorption correction was not applied. Full-matrix least-squares refinement on F using the weighting scheme $w = [\sigma^2(F) + 0.0014F^2]^{-1}$ converged to give $R = 0.0582$, $wR = 0.0722$ and $S = 1.49$, data/parameter ratio = 7.8. No shift in the final refinement (max. $\Delta/\sigma = 0.001$, mean $\Delta/\sigma = 0.000$). The highest unassigned electron density peaks (0.22–0.38 e Å^{-3}) were near the anion.

Fractional atomic coordinates are given in Table 1,* and selected bond distances and bond angles in Table 2. Fig. 1 shows the thermal ellipsoids drawn at the 30% probability level for the title compound and Fig. 2 illustrates the packing and hydrogen-bonding networks.

Related literature. The structure of 2,2,6,6-tetramethyl-4-piperidone hydrochloride (Rees & Weiss, 1971) contains N—H···Cl interactions and in 2,2,6,6-tetramethyl-4-piperidone monohydrate (De Camp, Micovic & Pelletier, 1974) there is evidence of hydrogen bonding from the water molecule. The title complex shows hydrogen bonding from N—H to the adjacent carbonyl O atom of another cation and two types of interactions to fluorines of the anion.

References

De Camp, W. H., Micovic, I. V. & Pelletier, S. W. (1974). *Cryst. Struct. Commun.* **3**, 427–431.

Rees, B. & Weiss, R. (1971). *Acta Cryst.* **B27**, 932–940.

Sheldrick, G. M. (1987). *SHELXTL-Plus*. Release 3.4 for Nicolet *R3m/V* crystallographic system. Nicolet Instrument Corporation, Madison, Wisconsin, USA.

---

* Lists of structure factors, anisotropic thermal parameters, bond lengths and angles, and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54824 (15 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HE0037]

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Structure of an (*E*)-17-(Diethylphosphonoisocyanomethylene) Steroid

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Abstract. (*E*)-17-(Diethylphosphonoisocyanomethylene)-3-methoxy-19-norandrosta-1,3,5(10)-triene, $C_{25}H_{34}NO_4P$, $M_r = 443.52$, monoclinic, $P2_1$, $a = 7.244$ (1), $b = 34.769$ (8), $c = 9.759$ (2) Å, $\beta = 105.70$ (1)°, $V = 2366.3$ (8) Å^3, $Z = 4$, $D_x = 1.245$ g cm^{-3}, λ(Mo $K\overline{\alpha}$) = 0.71073 Å, $\mu = 1.4$ cm^{-1}, $F(000) = 952$, $T = 130$ K, $R_F = 0.044$ for 3939 unique observed reflections with $I \geq 2.5\sigma(I)$ and 762 parameters. Two crystallographically independent molecules (*A* and *B*) of the title compound are present in the asymmetric unit. Their conformational flexibility, as measured by the different torsion angles about the P—O bonds, indicates that the crystal lattice plays a role in determining the molecular conformation. Both the exocyclic double bond of *A* as well as the exocyclic double bond of *B* exhibit *E* stereochemistry.

Experimental. The title steroid was prepared as one of a series from a 17-oxo steroid (Stoelwinder, van Zoest & van Leusen, 1992). A suitable colourless plate-like crystal of dimensions 0.10 × 0.25 × 0.43 mm, which was obtained by slow evaporation of a petroleum ether (b.p. 313–333 K) solution, was glued on top of a glass fiber and transferred into the cold nitrogen stream of the low-temperature unit mounted on an Enraf–Nonius CAD-4F diffractometer interfaced to a VAX-11/730 computer. Unit-cell dimensions and their standard deviations and the orientation matrix were determined from the setting angles of 22 reflections in the range $14.49 < \theta < 21.36°$ in four alternative settings (de Boer & Duisenberg, 1984). Intensity data of 4690 reflections were collected [h: $-1\rightarrow 8$; k: $0\rightarrow 42$; l: $-12\rightarrow 11$; $1.17 \leq \theta \leq 26.00°$; $\omega/2\theta$-scan mode; $\Delta\omega = (0.75 + 0.34\text{tg}\theta)°$; graphite-monochromated Mo $K\overline{\alpha}$ radiation]. The unit cell was identified as monoclinic, space group $P2_1$: the *E* statistics unambiguously showed a non-centrosymmetric space group (Snow & Tiekink, 1988). This assignment was confirmed by the solution and the successful refinement in this space group. Reduced-cell calculations did not indicate any

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higher metric lattice symmetry (Spek, 1988) and examination of the final atomic coordinates of the structure did not yield extra metric symmetry elements (Le Page, 1987, 1988). Crystal and/or instrumental instability was monitored by measurement of the intensities of three reference reflections that were collected after every 3 h of X-ray exposure time; there was no indication of crystal decomposition. The reference reflections showed a long-term variation of less than 3%. Intensities were corrected accordingly, and for Lorentz and polarization effects, but not for absorption. Standard deviations in the intensities based on counting statistics were increased according to an analysis of the excess variance (McCandlish, Stout & Andrews, 1975) of the three reference reflections: $\sigma^2(I) = \sigma^2_{cs}(I) + (0.061I)^2$. Equivalent reflections were averaged resulting in 3940 reflections satisfying the $I \geq 2.5\sigma(I)$ criterion of observability.

The structure was solved by direct methods with *SHELXS*86 (Sheldrick, 1986) and the positional and anisotropic thermal displacement parameters for the non-H atoms were refined with block-diagonal least-squares procedures using *CRYLSQ* (Olthof-Hazekamp, 1990) minimizing the function $Q = \sum_{\mathbf{h}}[w(|F_o| - |F_c|)^2]$. Subsequent difference Fourier maps led to the location of all the H-atom positions, which were included in the refinement. Final refinement on F_o, with unit weights, by full-matrix least-squares techniques with anisotropic thermal displacement parameters for the non-H atoms and one common isotropic thermal displacement parameter for the H atoms converged at $R_F = 0.044$ ($wR = 0.047$, $S = 1.219$). The polarity of the structure was tested by refinement with $i\Delta f''$ and $-i\Delta f''$ values, respectively, giving only marginally different R and S values (values of the alternative structure: $R_F = 0.044$, $wR = 0.048$, $S = 1.223$; there is no atom with strong anomalous-dispersion factors in the molecule). The reflection 100, which showed strong extinction effects, was excluded from the refinement. Fractional atomic coordinates and equivalent isotropic thermal displacement parameters of the non-H atoms are presented in Table 1. Selected molecular geometry data are collected in Table 2.* Scattering factors (Cromer & Mann, 1968) and anomalous-dispersion factors (Cromer & Liberman, 1970) were included in F_c. All calculations were carried out on the CDC-Cyber 962-31 computer of Groningen University with the program packages *XTAL* (Hall & Stewart, 1990), *PLATON* (Spek, 1990; calculation of geometric data) and a locally modified version of the program *PLUTO* (Meetsma, 1991; preparation of

Table 1. *Final fractional atomic coordinates and equivalent isotropic thermal displacement parameters for non-*H *atoms with e.s.d.'s in parentheses*

$$U_{eq} = (1/3)\textstyle\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}(Å^2)
Molecule *A*				
P(1)	0.04835 (19)	0.35060	0.56522 (14)	0.0233 (4)
O(1)	−0.1538 (5)	0.34556 (12)	0.4875 (5)	0.0370 (14)
O(2)	0.1168 (6)	0.33278 (11)	0.7184 (4)	0.0335 (14)
O(3)	0.2001 (5)	0.33087 (11)	0.5018 (4)	0.0246 (11)
O(4)	0.1539 (5)	0.71101 (11)	0.3761 (5)	0.0342 (13)
N(1)	0.3120 (6)	0.40515 (12)	0.6593 (4)	0.0204 (11)
C(1)	0.2779 (7)	0.61188 (15)	0.4648 (5)	0.0197 (14)
C(2)	0.2922 (7)	0.65087 (15)	0.4449 (5)	0.0240 (17)
C(3)	0.1267 (7)	0.67259 (15)	0.3953 (5)	0.0245 (17)
C(4)	−0.0510 (7)	0.65498 (15)	0.3690 (5)	0.0219 (17)
C(5)	−0.0652 (7)	0.61537 (14)	0.3894 (5)	0.0170 (12)
C(6)	−0.2631 (7)	0.59817 (15)	0.3668 (6)	0.0242 (17)
C(7)	−0.2691 (7)	0.55454 (15)	0.3550 (5)	0.0199 (14)
C(8)	−0.1059 (6)	0.53608 (14)	0.4707 (5)	0.0171 (12)
C(9)	0.0877 (6)	0.54949 (14)	0.4499 (5)	0.0161 (12)
C(10)	0.1018 (7)	0.59304 (15)	0.4371 (5)	0.0163 (12)
C(11)	0.2557 (7)	0.53118 (14)	0.5617 (5)	0.0198 (14)
C(12)	0.2449 (7)	0.48701 (14)	0.5639 (5)	0.0166 (12)
C(13)	0.0511 (7)	0.47336 (14)	0.5812 (5)	0.0150 (12)
C(14)	−0.1099 (6)	0.49275 (14)	0.4639 (5)	0.0177 (12)
C(15)	−0.2892 (7)	0.47039 (16)	0.4689 (6)	0.0233 (16)
C(16)	−0.2169 (7)	0.42865 (15)	0.4850 (6)	0.0247 (16)
C(17)	−0.0017 (7)	0.43087 (15)	0.5545 (5)	0.0195 (16)
C(18)	0.0372 (8)	0.48214 (16)	0.7339 (5)	0.0235 (16)
C(19)	0.1167 (7)	0.40057 (14)	0.5891 (5)	0.0181 (12)
C(20)	0.4725 (8)	0.40470 (15)	0.7179 (6)	0.0270 (17)
C(21)	0.0264 (10)	0.3464 (2)	0.8271 (7)	0.044 (2)
C(22)	0.1721 (13)	0.3517 (3)	0.9579 (9)	0.070 (3)
C(23)	0.1970 (9)	0.3363 (2)	0.3514 (6)	0.0326 (19)
C(24)	0.3791 (11)	0.3566 (3)	0.3508 (8)	0.056 (3)
C(25)	−0.0139 (9)	0.73398 (17)	0.3242 (8)	0.041 (2)
Molecule *B*				
P(2)	0.85133 (19)	0.25733 (5)	0.05531 (15)	0.0224 (4)
O(5)	1.0540 (5)	0.26653 (11)	0.1139 (4)	0.0319 (12)
O(6)	0.7084 (5)	0.27295 (11)	0.1379 (4)	0.0280 (11)
O(7)	0.7642 (5)	0.27387 (12)	−0.0980 (4)	0.0291 (12)
O(8)	1.0980 (5)	−0.09856 (11)	−0.1428 (4)	0.0289 (11)
N(2)	0.6212 (6)	0.19615 (12)	−0.0242 (4)	0.0195 (12)
C(26)	0.8741 (7)	−0.00605 (15)	−0.1330 (5)	0.0194 (16)
C(27)	0.9000 (8)	−0.04467 (15)	−0.1554 (5)	0.0222 (14)
C(28)	1.0811 (7)	−0.06065 (15)	−0.1077 (5)	0.0220 (14)
C(29)	1.2317 (8)	−0.03830 (16)	−0.0308 (5)	0.0244 (16)
C(30)	1.2041 (7)	0.00098 (14)	−0.0064 (5)	0.0181 (12)
C(31)	1.3721 (7)	0.02284 (15)	0.0874 (6)	0.0262 (17)
C(32)	1.3479 (7)	0.06665 (14)	0.0774 (6)	0.0200 (14)
C(33)	1.1461 (7)	0.07821 (15)	0.0805 (5)	0.0184 (12)
C(34)	0.9977 (7)	0.06100 (15)	−0.0501 (5)	0.0179 (14)
C(35)	1.0251 (7)	0.01744 (15)	−0.0602 (5)	0.0175 (12)
C(36)	0.7943 (7)	0.07370 (15)	−0.0559 (5)	0.0190 (12)
C(37)	0.7724 (7)	0.11752 (15)	−0.0520 (5)	0.0199 (14)
C(38)	0.9163 (6)	0.13512 (14)	0.0786 (5)	0.0165 (12)
C(39)	1.1206 (6)	0.12187 (14)	0.0763 (5)	0.0172 (12)
C(40)	1.2541 (7)	0.14674 (15)	0.1903 (5)	0.0211 (14)
C(41)	1.1538 (7)	0.18612 (15)	0.1641 (6)	0.0218 (16)
C(42)	0.9459 (7)	0.17825 (14)	0.0878 (5)	0.0153 (12)
C(43)	0.8661 (8)	0.12366 (16)	0.2168 (6)	0.0234 (17)
C(44)	0.8139 (7)	0.20552 (14)	0.0414 (5)	0.0185 (12)
C(45)	0.4599 (9)	0.19078 (17)	−0.0792 (6)	0.0356 (17)
C(46)	0.7355 (9)	0.2631 (2)	0.2881 (6)	0.0332 (19)
C(47)	0.5612 (10)	0.2431 (2)	0.3026 (7)	0.043 (2)
C(48)	0.5637 (8)	0.28460 (16)	−0.1647 (6)	0.0292 (17)
C(49)	0.5166 (10)	0.2708 (2)	−0.3152 (7)	0.047 (2)
C(50)	1.2818 (9)	−0.11610 (17)	−0.0938 (7)	0.037 (2)

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* Lists of structure factors, anisotropic thermal displacement parameters, H-atom parameters, and bond lengths and angles involving H atoms have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54817 (42 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. *Selected bond lengths* (Å), *bond angles* (°), *torsion angles* (°) *and hydrogen bonds* (Å, °) *with e.s.d.'s in parentheses*

Molecule *A*		Molecule *B*	
P(1)—O(1)	1.466 (4)	P(2)—O(5)	1.460 (4)
P(1)—O(2)	1.569 (4)	P(2)—O(6)	1.571 (4)
P(1)—O(3)	1.559 (4)	P(2)—O(7)	1.567 (4)
P(1)—C(19)	1.804 (5)	P(2)—C(44)	1.821 (5)
O(2)—C(21)	1.468 (8)	O(6)—C(46)	1.466 (7)
O(3)—C(23)	1.474 (7)	O(7)—C(48)	1.470 (7)
N(1)—C(19)	1.404 (7)	N(2)—C(44)	1.407 (7)
N(1)—C(20)	1.149 (7)	N(2)—C(45)	1.161 (8)
C(17)—C(19)	1.343 (7)	C(42)—C(44)	1.336 (7)
C(21)—C(22)	1.431 (11)	C(46)—C(47)	1.481 (10)
C(23)—C(24)	1.497 (11)	C(48)—C(49)	1.494 (9)

O(1)—P(1)—O(2)	117.4 (2)	O(5)—P(2)—O(6)	117.1 (2)
O(1)—P(1)—O(3)	117.1 (2)	O(5)—P(2)—O(7)	113.3 (2)
O(1)—P(1)—C(19)	112.5 (2)	O(5)—P(2)—C(44)	111.1 (2)
O(2)—P(1)—O(3)	97.4 (2)	O(6)—P(2)—O(7)	102.2 (2)
O(2)—P(1)—C(19)	104.6 (2)	O(6)—P(2)—C(44)	105.9 (2)
O(3)—P(1)—C(19)	106.1 (2)	O(7)—P(2)—C(44)	106.2 (2)
P(1)—O(2)—C(21)	118.2 (4)	P(2)—O(6)—C(46)	120.2 (4)
P(1)—O(3)—C(23)	121.1 (4)	P(2)—O(7)—C(48)	127.6 (3)
C(19)—N(1)—C(20)	172.7 (5)	C(44)—N(2)—C(45)	175.9 (5)
C(13)—C(17)—C(19)	127.0 (5)	C(38)—C(42)—C(44)	127.3 (5)
C(16)—C(17)—C(19)	125.4 (5)	C(41)—C(42)—C(44)	124.3 (5)
P(1)—C(19)—N(1)	112.2 (3)	P(2)—C(44)—N(2)	111.8 (3)
P(1)—C(19)—C(17)	126.1 (4)	P(2)—C(44)—C(42)	126.8 (4)
N(1)—C(19)—C(17)	121.6 (5)	N(2)—C(44)—C(42)	121.4 (4)
O(2)—C(21)—C(22)	108.7 (6)	O(6)—C(46)—C(47)	108.6 (5)
O(3)—C(23)—C(24)	106.7 (5)	O(7)—C(48)—C(49)	106.8 (5)

O(1)—P(1)—O(2)—C(21)	−60.6 (5)	O(5)—P(2)—O(6)—C(46)	−57.0 (5)
O(3)—P(1)—O(2)—C(21)	173.6 (4)	O(7)—P(2)—O(6)—C(46)	178.5 (4)
C(19)—P(1)—O(2)—C(21)	64.8 (5)	C(44)—P(2)—O(6)—C(46)	67.5 (4)
O(1)—P(1)—O(3)—C(23)	51.1 (5)	O(5)—P(2)—O(7)—C(48)	−152.4 (4)
O(2)—P(1)—O(3)—C(23)	177.0 (4)	O(6)—P(2)—O(7)—C(48)	−25.4 (5)
C(19)—P(1)—O(3)—C(23)	−75.4 (4)	C(44)—P(2)—O(7)—C(48)	85.4 (5)
C(18)—C(13)—C(17)—C(19)	−86.3 (6)	C(43)—C(38)—C(42)—C(44)	−90.1 (6)
C(15)—C(16)—C(17)—C(19)	179.1 (5)	C(40)—C(41)—C(42)—C(44)	−177.1 (5)
C(13)—C(17)—C(19)—P(1)	176.4 (4)	C(38)—C(42)—C(44)—P(2)	179.2 (4)
C(13)—C(17)—C(19)—N(1)	1.0 (8)	C(38)—C(42)—C(44)—N(2)	.4 (8)
C(16)—C(17)—C(19)—P(1)	−1.5 (8)	C(41)—C(42)—C(44)—P(2)	1.1 (8)
C(16)—C(17)—C(19)—N(1)	−176.9 (5)	C(41)—C(42)—C(44)—N(2)	−177.6 (4)
N(1)—C(19)—P(1)—O(1)	−178.0 (3)	N(2)—C(44)—P(2)—O(5)	−175.8 (3)
N(1)—C(19)—P(1)—O(2)	53.6 (4)	N(2)—C(44)—P(2)—O(6)	56.0 (4)
N(1)—C(19)—P(1)—O(3)	−48.8 (4)	N(2)—C(44)—P(2)—O(7)	−52.2 (4)
C(17)—C(19)—P(1)—O(1)	6.3 (5)	C(42)—C(44)—P(2)—O(5)	5.3 (5)
C(17)—C(19)—P(1)—O(2)	−122.2 (5)	C(42)—C(44)—P(2)—O(6)	−122.8 (5)
C(17)—C(19)—P(1)—O(3)	135.5 (4)	C(42)—C(44)—P(2)—O(7)	129.0 (5)
C(22)—C(21)—O(2)—P(1)	−137.3 (6)	C(47)—C(46)—O(6)—P(2)	−121.1 (5)
C(24)—C(23)—O(3)—P(1)	116.2 (6)	C(49)—C(48)—O(7)—P(2)	−138.7 (5)

Intra- and intermolecular hydrogen bonds

D—H···*A*	*D*···*A*	*D*—H	H···*A*	*D*—H···*A*
C(12)—H(122)···N(1)[a]	2.993 (6)	0.93 (6)	2.63 (6)	104 (4)
C(21)—H(211)···O(7)[b]	3.355 (8)	0.98 (6)	2.46 (6)	151 (5)
C(22)—H(223)···O(8)[c]	3.462 (1)	1.01 (6)	2.52 (6)	155 (5)
C(23)—H(231)···O(8)[d]	3.389 (8)	1.11 (6)	2.35 (6)	154 (4)
C(23)—H(232)···O(5)[e]	3.321 (7)	1.00 (6)	2.46 (6)	144 (5)
C(24)—H(242)···O(1)[f]	3.307 (9)	1.14 (6)	2.57 (6)	121 (4)
C(37)—H(372)···N(2)[a]	2.985 (7)	0.94 (7)	2.56 (6)	108 (4)
C(46)—H(461)···O(1)[f]	3.436 (8)	0.88 (6)	2.58 (6)	166 (5)
C(49)—H(493)···O(3)[g]	3.255 (8)	0.92 (6)	2.52 (6)	137 (5)

Symmetry codes: (*a*) x, y, z; (*b*) $-1+x, y, 1+z$; (*c*) $1-x, \frac{1}{2}+y, 1-z$; (*d*) $1-x, \frac{1}{2}+y, -z$; (*e*) $-1+x, y, z$; (*f*) $1+x, y, z$; (*g*) $x, y, -1+z$.

illustrations). The identification of the atoms and the configuration are shown in the *PLUTO* drawings of Fig. 1; the packing and the arrangement of the molecules in the unit cell are shown in Fig. 2. Each asymmetric unit contains two complete molecules (*A* and *B*) with no atom at a special position, separated by normal van der Waals distances (Bondi, 1964) (Fig. 2) and some weak hydrogen bonds (Berkovitch-Yellin & Leiserowitz, 1984) (Table 2).

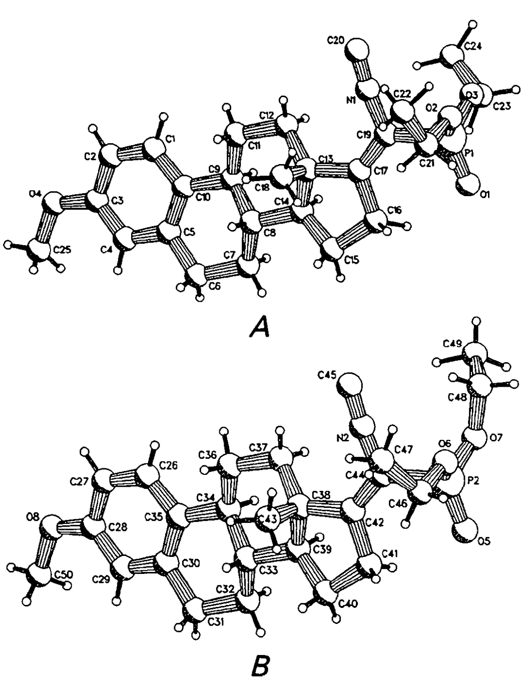

Fig. 1. *PLUTO* drawings of the two independent molecules *A* and *B* of the title compound, showing *E* stereochemistry for both C(17)—C(19) and C(42)—C(44).

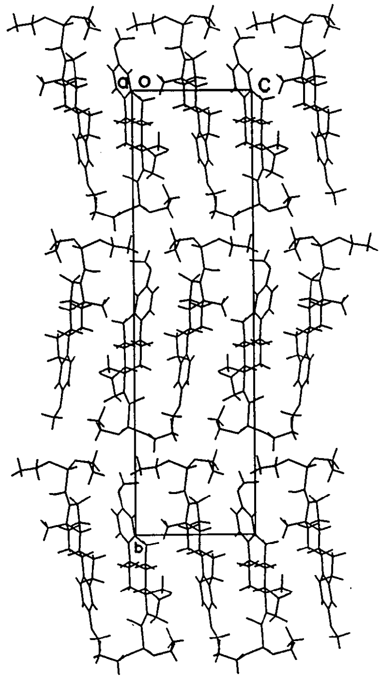

Fig. 2. Projection of the crystal structure down [100].

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 isocyano groups. The structural parameters of the diethylphosphono group can be compared with those reported by Espenbetov, Chernega, Struchkov, Dzhiembaev & Abiyurov (1985).

References

Berkovitch-Yellin, Z. & Leiserowitz, L. (1984). *Acta Cryst.* B**40**, 159–165.

Boer, J. L. de & Duisenberg, A. J. M. (1984). *Acta Cryst.* A**40**, C410.

Bondi, A. (1964). *J. Phys. Chem.* **68**, 441–451.

Britton, D., Gleason, W. B. & Glick, M. (1981). *Cryst. Struct. Commun.* **10**, 1497–1500.

Busetta, P. B., Courseille, C. & Hosptial, M. (1973). *Acta Cryst.* B**29**, 298–313.

Cromer, D. T. & Liberman, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.

Cromer, D. T. & Mann, J. B. (1968). *Acta Cryst.* A**24**, 321–324.

Debaerdemaeker, T. D. J. (1972). *Cryst. Struct. Commun.* **1**, 39–42.

Espenbetov, A. A., Chernega, A. N., Struckov, Yu. T., Dzhiembaev, B. Zh. & Abiyurov, B. D. (1985). *Izv. Akad. Nauk. Kaz.* p. 65.

Hall, S. R. & Stewart, J. M. (1990). Editors. *XTAL*3.0 *Reference Manual.* Univs. of Western Australia, Australia, and Maryland, USA.

Le Page, Y. (1987). *J. Appl. Cryst.* **20**, 264–269.

Le Page, Y. (1988). *J. Appl. Cryst.* **21**, 983–984.

McCandlish, L. E., Stout, G. H. & Andrews, L. C. (1975). *Acta Cryst.* A**31**, 245–249.

Meetsma, A. (1991). Extended version of the program *PLUTO*. Groningen Univ., The Netherlands. (Unpublished.)

Olthof-Hazekamp, R. (1990). *CRYLSQ. XTAL*3.0 *Manual*, edited by S. R. Hall & J. M. Stewart. Univs. of Western Australia, Australia, and Maryland, USA. Perth: Lamb.

Sheldrick, G. M. (1986). *SHELXS*86. Program for crystal structure solution. Univ. of Göttingen, Germany.

Snow, M. R. & Tiekink, E. R. T. (1988). *Acta Cryst.* B**44**, 676–677.

Spek, A. L. (1988). *J. Appl. Cryst.* **21**, 578–579.

Spek, A. L. (1990). *Acta Cryst.* A**46**, C-34.

Stoelwinder, J., van Zoest, W. J. & van Leusen, A. M. (1992). *J. Org. Chem.* In the press.

Acta Cryst. (1992). C**48**, 1141–1143

Structure of Hexaacetyl-D-mannitol

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Abstract. *n*-Hexyl hexaacetate, $C_{18}H_{26}O_{12}$, $M_r = 434.4$, orthorhombic, $P2_12_12_1$, $a = 8.934\,(2)$, $b = 11.961\,(7)$, $c = 19.764\,(3)$ Å, $V = 2112.0$ Å^3, $Z = 4$, $D_x = 1.366$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 1.09$ 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, $[\alpha]_D$ +18.6 (*c* 0.22 in CH_2Cl_2). Recrystallization from acetone. Crystal size 0.5 × 0.3 × 0.2 mm, cell dimensions from setting angles of 22 reflections in the range $8 < \theta < 12°$, space group by systematic absences. Data collected on an Enraf–Nonius CAD-4 diffractometer with graphite-monochromated Mo $K\alpha$ radiation, $\theta_{max} = 25°$, 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 $\bar{4}\bar{2}4$) 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 $I > 3\sigma(I)$ were used in refinement, no corrections for absorption or secondary extinction.

The structure was solved by direct methods (*SHELXS*86; Sheldrick, 1985), and refined by least squares (*SHELX*76; 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 $w(\Delta F)^2$, with $w = 1.43/\sigma^2(F_o)$ ($w = 0$ 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 $\Delta/\sigma < 0.3$, residual densities in Fourier maps

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