Table 3. *Hydrogen-bond distances* (Å) *and angles* (°)

X—H···O	X···O	H···O	X—H···O
O(2)—H(22)···W(4$^{\mathrm{i}}$)	2.693 (4)	1.74 (4)	167 (4)
O(3)—H(33)···O(9)	2.764 (4)	1.87 (3)	153 (3)
O(4)—H(44)···O(8$^{\mathrm{ii}}$)	2.608 (4)	1.67 (4)	161 (4)
O(5)—H(55)···O(9$^{\mathrm{iii}}$)	2.934 (4)	2.01 (3)	158 (4)
W(1)—H(W11)···O(9$^{\mathrm{iv}}$)	2.928 (4)	1.98 (3)	166 (4)
W(1)—H(W12)···O(8$^{\mathrm{v}}$)	2.823 (4)	1.87 (3)	165 (3)
W(2)—H(W21)···O(7$^{\mathrm{iv}}$)	2.919 (4)	1.98 (4)	163 (4)
W(2)—H(W22)···W(3)	2.872 (5)	2.00 (4)	149 (4)
W(3)—H(W31)···O(7$^{\mathrm{v}}$)	2.926 (5)	1.98 (3)	165 (3)
W(3)—H(W32)···O(9$^{\mathrm{iv}}$)	2.869 (4)	1.93 (3)	161 (3)
W(4)—H(W41)···O(7)	2.769 (4)	1.83 (3)	161 (4)
W(4)—H(W42)···W(2$^{\mathrm{vi}}$)	2.724 (4)	1.88 (3)	144 (4)
W(5)—H(W51)···W(3$^{\mathrm{i}}$)	2.908 (4)	1.99 (3)	156 (4)
W(5)—H(W52)···O(7$^{\mathrm{vii}}$)	2.884 (4)	1.92 (2)	175 (5)
C(6)—H(6)···O(8$^{\mathrm{i}}$)	3.223 (5)	2.29 (3)	143 (3)

Symmetry code: (i) $x-1$, y, z; (ii) $-x$, $y+0.5$, $1-z$; (iii) $-x$, $y-0.5$, $1-z$; (iv) $1-x$, $y-0.5$, $1-z$; (v) $1-x$, $y+0.5$, $1-z$; (vi) x, y, $z-1$; (vii) x, y, $1+z$.

O(ester)—P—O bond angle [O(1)—P—O(8) = 102.1 (2)°]. Two terminal phosphate O atoms [O(7) and O(9)] form (as acceptors) four hydrogen bonds and are therefore formally pentacoordinated (Table 3). Although this arrangement does not appear to have been observed previously in phosphate esters a few examples of formal pentacoordination of phosphate O atoms are already known (Weichsel & Lis, 1990).

All OH groups participate in hydrogen bonding (see Fig. 2 and Table 3). There is also an intermolecular hydrogen bond between terminal phosphate atoms O(8) and C(6) according to the criterion of Taylor & Kennard (1982).

There are two crystallographically independent Na^+ ions in the structure. The Na(1) atom (Table 2) is seven coordinate [Na—O distances of 2.378 (3)–2.623 (3) Å], binding to three independent fructose moieties. One of these is tridentate, binding to Na(1) through O(1$^{\mathrm{i}}$), O(5$^{\mathrm{i}}$) and O(6$^{\mathrm{i}}$), and two are bidentate, binding through O(3) and O(4) and through O(4$^{\mathrm{ii}}$) and O(5$^{\mathrm{ii}}$). The Na(2) atom has a distorted octahedral coordination formed by two sugar O atoms O(2$^{\mathrm{iii}}$) and O(3$^{\mathrm{iii}}$) and four water molecules at Na—O distances of 2.352 (4)–2.795 (3) Å. The phosphate O atoms do not participate in the coordination spheres of the Na^+ cations as observed previously in Na_2(F6P).$7H_2O$ (Lis, 1986) and Na_3H(F1,6-dp).$8H_2O$ (Narendra, Seshadri & Viswamitra, 1985*a*; Cerrini, Coiro, Lamba & Bisso, 1986).

References

Cerrini, S., Coiro, V. M., Lamba, D. & Bisso, G. M. B. (1986). *Carbohydr. Res.* **147**, 183–190.

Cremer, D. & Pople, J. A. (1975). *J. Am. Chem. Soc.* **97**, 1354–1358.

Koerner, T. A. W. Jr, Voll, R. J., Cary, L. W. & Younathan, E. S. (1980). *Biochemistry*, **19**, 2795–2801.

Lis, T. (1986). *Acta Cryst.* C**42**, 1745–1747.

Narendra, N., Seshadri, T. P. & Viswamitra, M. A. (1985*a*). *Acta Cryst.* C**41**, 31–34.

Narendra, N., Seshadri, T. P. & Viswamitra, M. A. (1985*b*). *Acta Cryst.* C**41**, 1612–1614.

Sheldrick, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.

Starynowicz, P. (1986). *Tenth European Crystallographic Meeting, Collected Abstracts*, p. 174. Wrocław, Poland.

Takagi, S. & Jeffrey, G. A. (1977). *Acta Cryst.* B**33**, 3510–3515.

Taylor, R. & Kennard, O. (1982). *J. Am. Chem. Soc.* **104**, 5063–5070.

Weichsel, A. & Lis, T. (1990). *Acta Cryst.* C**46**, 962–965.

Weichsel, A., Lis, T. & Kuczek, M. (1989). *Carbohydr. Res.* **194**, 63–70.

Acta Cryst. (1992). C**48**, 696–699

Structure of 1,3,4,5-Neopentanetetrayl Tetrakis[3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionate]

By Roberto Millini* and Gastone Del Piero

Eniricerche SpA, Via F. Maritano 26, *I*-20097 *San Donato Milanese, Milano, Italy*

and Michela Bracci

Enichem Synthesis SpA, Via F. Maritano 26, *I*-20097 *San Donato Milanese, Milano, Italy*

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Abstract. $C_{73}H_{108}O_{12}$, $M_r = 1177.65$, monoclinic, Cc, $a = 25.442$ (8), $b = 10.051$ (3), $c = 27.062$ (10) Å, $\beta = 94.5$ (1)°, $V = 6898.5$ Å^3, $Z = 4$, $D_x = 1.1339$ Mg m^{-3}, λ(Cu $K\alpha$) = 1.54178 Å, $\mu = 5.64$ cm^{-1}, $F(000) = 2568$, $T = 293$ K, $R = 0.094$ for 5060 observed reflections. The molecular structure

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* Author to whom correspondence should be addressed.

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consists of four chemically equivalent chains which branch out tetrahedrally from the central C(1) atom. One pair of chains has a conformation slightly different from the other, giving rise to a pseudo-binary symmetry through the C(1) atom. The crystal structure is stabilized by weak intermolecular hydrogen bonds [O(3)···O(11)(x, $y-1$, $z+\frac{1}{2}$) 3.14 (1) and O(6)···O(8)(x, $1-y$, $z-\frac{1}{2}$) 3.17 (1) Å].

Introduction. The title compound is industrially produced and used as an antioxidant for the polyolefins (Dexter, Spivack & Steinberg, 1966). Although it is widely used for different commercial polymers, no information on its crystal structure is available. The compound crystallizes in different phases, depending on the solvent used for crystallization. Ethanol gives rise to a phase melting at 385–387 K (Hasui, Furihata, Takanashi & Ohmori, 1981) characterized by a powder diffraction pattern which can be indexed on the basis of a tetragonal unit cell (Millini, 1990). A different phase, in the form of single crystals was obtained by recrystallization from *n*-hexane (m.p. 394–396 K). This paper reports the results of the crystal structure analysis of this phase.

Experimental. The title compound was obtained by transesterification of methyl 3-(3,5-di-*tert*-butyl-4-hydroxyphenyl)propionate with pentaerythritol and subsequent purification by recrystallization from *n*-hexane. Colorless crystals, m.p. 395 K (determined by DSC). A parallelepiped crystal, approximately 0.4 × 0.5 × 0.7 mm in size, was used for data collection on a Siemens AED diffractometer. Ni-filtered Cu $K\alpha$ radiation ($\lambda = 1.54178$ Å) was used. Unit-cell parameters were determined by least-squares refinement from 30 reflections collected in the range $7 \leq 2\theta \leq 22°$. Systematic absences indicated space group $C2/c$ or Cc. The noncentrosymmetric space group Cc (origin on glide plane c) was confirmed by structure solution and least-squares refinement. Data were collected to $\theta_{\max} = 65°$, using θ–2θ scans, index range h ±28, k 0→11, l 0→30. The intensity of the 004 reflection, monitored every 50 measurements, varied within ±3%. 6328 unique reflections were measured, 5060 with $I > 2\sigma(I)$ were used for structural analysis. Intensities were not corrected for absorption effects. The structure was solved by direct methods using *SIR*88 (Burla, Camalli, Cascarano, Giacovazzo, Polidori, Spagna & Viterbo, 1989). Because of high thermal parameters, some C atoms belonging to *tert*-butyl groups were refined with fixed isotropic $U = 0.06$ Å^2. Positions of H atoms were calculated according to the trigonal or tetrahedral geometry of C atoms and kept fixed with $U = 0.06$ Å^2 during the block-matrix anisotropic least-squares refinement (694 parameters) (Immirzi, 1973). The function minimized was $\sum w(|F_o| - |F_c|)^2$, where $w = 17 + F_o + 0.011F_o^2$. The refinement converged to $R = 0.094$, $wR = 0.098$ and $S = 1.956$, with a maximum shift/e.s.d. ratio of 0.44. We attribute the relatively high R factors to the disorder affecting some *tert*-butyl groups [mainly C(52)–C(54), C(66)–C(69) and C(70)–C(73)]. The residual electron density in the final difference map was within ±0.32 e Å^{-3}. Calculations were performed on an IBM 4381 computer. Atomic scattering factors were from *International Tables for X-ray Crystallography* (1974, Vol. IV). Other computer programs used: *PLUTO* (Motherwell & Clegg, 1978) and *PARST* (Nardelli, 1983).

Final positional and equivalent isotropic thermal parameters are reported in Table 1; Table 2 lists interatomic distances and selected valence and dihedral angles.*

Discussion. The molecular conformation and the labelling scheme adopted are shown in Fig. 1. The molecule contains four chains of equal composition, tetrahedrally linked to the C(1) atom. No anomalies in bond lengths and angles were observed. Inspection of the dihedral angles (Table 2) revealed the presence of a pseudo-binary symmetry through the C(1) atom, arising from the slightly different conformation

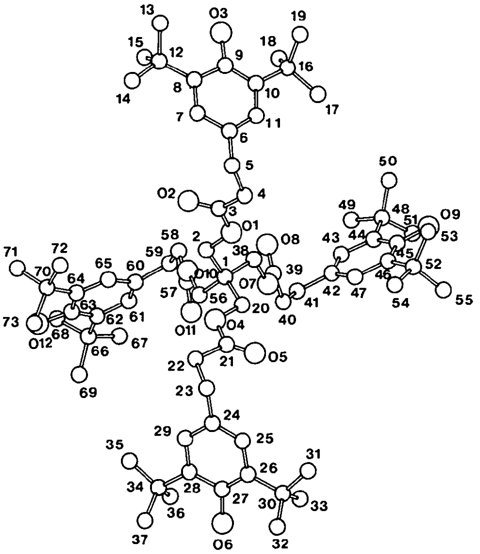

Fig. 1. *PLUTO* (Motherwell & Clegg, 1978) view of the molecule, showing the numbering scheme adopted.

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* Lists of structure factors, valence and dihedral angles, anisotropic thermal parameters, H-atom coordinates, and mean planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 54642 (33 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: GE0280]

Table 1. *Atomic coordinates and equivalent isotropic thermal parameters* (Å^2) *with e.s.d.'s in parentheses*

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

	x	y	z	U_{eq}/U_{iso}
C(1)	0.8134 (4)	0.5031 (4)	0.2675 (3)	0.030 (1)
C(2)	0.7823 (2)	0.4134 (6)	0.3000 (2)	0.035 (1)
O(1)	0.8188 (2)	0.3361 (4)	0.3318 (2)	0.038 (1)
C(3)	0.7968 (2)	0.2474 (5)	0.3618 (2)	0.034 (1)
O(2)	0.7493 (2)	0.2424 (5)	0.3630 (2)	0.043 (1)
C(4)	0.8350 (2)	0.1623 (6)	0.3878 (2)	0.038 (1)
C(5)	0.8119 (3)	0.0741 (5)	0.4261 (2)	0.041 (2)
C(6)	0.7993 (2)	0.1417 (5)	0.4731 (2)	0.029 (1)
C(7)	0.7475 (2)	0.1328 (6)	0.4886 (2)	0.035 (1)
C(8)	0.7349 (2)	0.1880 (5)	0.5336 (2)	0.034 (1)
C(9)	0.7773 (3)	0.2522 (6)	0.5624 (2)	0.039 (1)
C(10)	0.8272 (2)	0.2689 (6)	0.5467 (2)	0.037 (1)
C(11)	0.8355 (2)	0.2078 (5)	0.5026 (2)	0.035 (1)
O(3)	0.7636 (2)	0.3090 (6)	0.6064 (2)	0.056 (1)
C(12)	0.6788 (2)	0.1742 (7)	0.5491 (2)	0.044 (2)
C(13)	0.6744 (3)	0.1188 (10)	0.6022 (3)	0.072 (3)
C(14)	0.6433 (3)	0.0914 (9)	0.5140 (3)	0.060 (2)
C(15)	0.6527 (3)	0.3092 (10)	0.5497 (4)	0.069 (3)
C(16)	0.8700 (3)	0.3463 (6)	0.5779 (2)	0.042 (1)
C(17)	0.9209 (3)	0.3549 (9)	0.5504 (3)	0.059 (2)
C(18)	0.8518 (3)	0.4875 (7)	0.5879 (3)	0.054 (2)
C(19)	0.8843 (3)	0.2719 (8)	0.6280 (3)	0.056 (2)
C(20)	0.8467 (2)	0.4223 (5)	0.2338 (2)	0.031 (1)
O(4)	0.8108 (1)	0.3344 (4)	0.2052 (1)	0.034 (1)
C(21)	0.8343 (2)	0.2509 (5)	0.1747 (2)	0.028 (1)
O(5)	0.8793 (2)	0.2455 (5)	0.1715 (2)	0.044 (1)
C(22)	0.7921 (2)	0.1634 (6)	0.1458 (2)	0.039 (1)
C(23)	0.8168 (3)	0.0696 (6)	0.1092 (2)	0.036 (1)
C(24)	0.8306 (2)	0.1380 (5)	0.0615 (2)	0.035 (1)
C(25)	0.8797 (2)	0.1300 (5)	0.0462 (2)	0.033 (1)
C(26)	0.8935 (2)	0.1844 (6)	0.0017 (2)	0.037 (1)
C(27)	0.8555 (2)	0.2512 (5)	−0.0268 (2)	0.035 (1)
C(28)	0.8024 (2)	0.2656 (5)	−0.0127 (2)	0.033 (1)
C(29)	0.7907 (2)	0.2077 (6)	0.0323 (2)	0.034 (1)
O(6)	0.8619 (2)	0.3059 (5)	−0.0736 (2)	0.055 (1)
C(30)	0.9499 (3)	0.1722 (8)	−0.0141 (3)	0.048 (1)
C(31)	0.9855 (3)	0.0958 (9)	0.0249 (3)	0.065 (2)
C(32)	0.9502 (3)	0.0893 (9)	−0.0612 (3)	0.058 (2)
C(33)	0.9742 (4)	0.3136 (10)	−0.0167 (4)	0.076 (3)
C(34)	0.7574 (3)	0.3396 (6)	−0.0435 (2)	0.046 (2)
C(35)	0.7079 (4)	0.3448 (11)	−0.0158 (3)	0.068 (3)
C(36)	0.7736 (4)	0.4845 (8)	−0.0528 (3)	0.070 (2)
C(37)	0.7450 (3)	0.2726 (8)	−0.0928 (2)	0.053 (2)
C(38)	0.8523 (2)	0.5913 (5)	0.3006 (2)	0.032 (1)
O(7)	0.8759 (2)	0.6871 (4)	0.2697 (1)	0.038 (1)
C(39)	0.8791 (2)	0.8128 (5)	0.2856 (2)	0.033 (1)
O(8)	0.8633 (2)	0.8483 (4)	0.3240 (2)	0.050 (1)
C(40)	0.8976 (2)	0.9036 (6)	0.2488 (2)	0.038 (1)
C(41)	0.9138 (2)	1.0414 (6)	0.2675 (2)	0.041 (2)
C(42)	0.9623 (2)	1.0480 (5)	0.3029 (2)	0.034 (1)
C(43)	0.9705 (2)	1.1597 (5)	0.3319 (2)	0.033 (1)
C(44)	1.0160 (2)	1.1795 (6)	0.3624 (2)	0.036 (1)
C(45)	1.0549 (2)	1.0773 (6)	0.3644 (2)	0.038 (1)
C(46)	1.0469 (2)	0.9620 (6)	0.3373 (2)	0.039 (2)
C(47)	1.0004 (2)	0.9485 (5)	0.3052 (2)	0.031 (1)
O(9)	1.0985 (2)	1.0953 (6)	0.3964 (2)	0.060 (1)
C(48)	1.0216 (3)	1.3096 (8)	0.3941 (3)	0.064 (2)
C(49)	0.9739 (5)	1.4054 (9)	0.3815 (5)	0.092 (4)
C(50)	1.0254 (6)	1.2813 (12)	0.4482 (4)	0.071 (3)
C(51)	1.0686 (4)	1.3924 (7)	0.3814 (4)	0.067 (3)
C(52)	1.0887 (3)	0.8495 (6)	0.3369 (3)	0.060
C(53)	1.1052 (4)	0.8052 (7)	0.3903 (4)	0.060
C(54)	1.0670 (4)	0.7292 (9)	0.3065 (4)	0.060
C(55)	1.1375 (5)	0.9035 (10)	0.3133 (4)	0.060
C(56)	0.7765 (2)	0.5892 (6)	0.2344 (2)	0.041 (1)
O(10)	0.7517 (2)	0.6861 (4)	0.2643 (1)	0.040 (1)
C(57)	0.7497 (2)	0.8113 (5)	0.2481 (2)	0.034 (1)
O(11)	0.7618 (2)	0.8441 (5)	0.2073 (2)	0.050 (1)
C(58)	0.7296 (2)	0.9018 (6)	0.2886 (2)	0.039 (1)
C(59)	0.7150 (2)	1.0394 (6)	0.2680 (2)	0.040 (1)
C(60)	0.6665 (2)	1.0479 (5)	0.2313 (2)	0.029 (1)
C(61)	0.6577 (2)	1.1630 (6)	0.2031 (2)	0.037 (1)
C(62)	0.6122 (3)	1.1810 (6)	0.1722 (2)	0.044 (2)
C(63)	0.5752 (2)	1.0808 (7)	0.1710 (2)	0.044 (2)
C(64)	0.5816 (2)	0.9607 (6)	0.1992 (2)	0.035 (1)
O(12)	0.5262 (2)	1.0969 (6)	0.1443 (2)	0.071 (2)
C(65)	0.6278 (2)	0.9522 (5)	0.2274 (2)	0.035 (1)
C(66)	0.5999 (3)	1.3055 (6)	0.1412 (3)	0.060
C(67)	0.6447 (5)	1.4077 (9)	0.1505 (5)	0.060
C(68)	0.5480 (5)	1.3674 (8)	0.1559 (5)	0.060
C(69)	0.5944 (4)	1.2689 (7)	0.0858 (4)	0.060
C(70)	0.5383 (3)	0.8562 (6)	0.1981 (3)	0.060
C(71)	0.4881 (4)	0.9199 (7)	0.2159 (4)	0.060
C(72)	0.5567 (5)	0.7421 (9)	0.2333 (5)	0.060
C(73)	0.5270 (5)	0.8021 (10)	0.1447 (5)	0.060

Table 2. *Geometrical data* (Å, °) *with e.s.d.'s in parentheses*

C(1)—C(2)	1.524 (10)	C(1)—C(20)	1.527 (9)
C(1)—C(38)	1.558 (10)	C(1)—C(56)	1.516 (11)
C(2)—O(1)	1.442 (8)	O(1)—C(3)	1.352 (8)
C(3)—O(2)	1.213 (7)	C(3)—C(4)	1.439 (9)
C(4)—C(5)	1.517 (9)	C(5)—C(6)	1.499 (8)
C(6)—C(7)	1.417 (8)	C(6)—C(11)	1.346 (8)
C(7)—C(8)	1.398 (8)	C(8)—C(9)	1.433 (9)
C(8)—C(12)	1.526 (9)	C(9)—C(10)	1.381 (9)
C(9)—O(3)	1.390 (7)	C(10)—C(11)	1.373 (8)
C(10)—C(16)	1.536 (9)	C(12)—C(13)	1.553 (13)
C(12)—C(14)	1.508 (11)	C(12)—C(15)	1.511 (12)
C(16)—C(17)	1.546 (11)	C(16)—C(18)	1.524 (9)
C(16)—C(19)	1.566 (12)	C(20)—O(4)	1.450 (7)
O(4)—C(21)	1.350 (6)	C(21)—O(5)	1.156 (7)
C(21)—C(22)	1.551 (8)	C(22)—C(23)	1.537 (8)
C(23)—C(24)	1.528 (9)	C(24)—C(25)	1.349 (8)
C(24)—C(29)	1.421 (9)	C(25)—C(26)	1.393 (9)
C(26)—C(27)	1.365 (9)	C(26)—C(30)	1.534 (9)
C(27)—C(28)	1.440 (8)	C(27)—O(6)	1.402 (8)
C(28)—C(29)	1.403 (9)	C(28)—C(34)	1.552 (10)
C(30)—C(31)	1.540 (13)	C(30)—C(32)	1.523 (11)
C(30)—C(33)	1.554 (13)	C(34)—C(35)	1.517 (12)
C(34)—C(36)	1.540 (11)	C(34)—C(37)	1.505 (9)
C(38)—O(7)	1.438 (8)	O(7)—C(39)	1.335 (7)
C(39)—O(8)	1.198 (8)	C(39)—C(40)	1.456 (9)
C(40)—C(41)	1.520 (9)	C(41)—C(42)	1.502 (9)
C(42)—C(43)	1.376 (8)	C(42)—C(47)	1.391 (8)
C(43)—C(44)	1.382 (9)	C(44)—C(45)	1.424 (9)
C(44)—C(48)	1.564 (10)	C(45)—C(46)	1.378 (8)
C(45)—O(9)	1.364 (9)	C(46)—C(47)	1.418 (9)
C(46)—C(52)	1.553 (9)	C(48)—C(49)	1.565 (15)
C(48)—C(50)	1.487 (15)	C(48)—C(51)	1.518 (13)
C(52)—C(53)	1.538 (11)	C(52)—C(54)	1.540 (9)
C(52)—C(55)	1.539 (9)	C(56)—O(10)	1.443 (7)
O(10)—C(57)	1.332 (8)	C(57)—O(11)	1.215 (8)
C(57)—C(58)	1.542 (8)	C(58)—C(59)	1.526 (10)
C(59)—C(60)	1.524 (9)	C(60)—C(61)	1.394 (9)
C(60)—C(65)	1.375 (8)	C(61)—C(62)	1.385 (10)
C(62)—C(63)	1.377 (10)	C(62)—C(66)	1.525 (10)
C(63)—C(64)	1.431 (11)	C(63)—O(12)	1.400 (9)
C(64)—C(65)	1.352 (9)	C(64)—C(70)	1.521 (9)
C(66)—C(67)	1.540 (11)	C(66)—C(68)	1.540 (10)
C(66)—C(69)	1.539 (11)	C(70)—C(71)	1.540 (9)
C(70)—C(72)	1.540 (11)	C(70)—C(73)	1.549 (10)

C(2)—C(1)—C(38)	109.9 (2)	C(2)—C(1)—C(56)	110.7 (3)
C(2)—C(1)—C(20)	111.6 (2)	C(20)—C(1)—C(56)	107.3 (3)
C(20)—C(1)—C(38)	106.8 (2)	C(38)—C(1)—C(56)	110.5 (2)
C(1)—C(2)—O(1)	108.9 (2)	C(2)—O(1)—C(3)	115.7 (2)
O(1)—C(3)—O(2)	120.5 (2)	O(1)—C(3)—C(4)	113.0 (2)
O(2)—C(3)—C(4)	126.5 (3)	C(3)—C(4)—C(5)	113.6 (2)
C(4)—C(5)—C(6)	115.8 (2)	C(1)—C(20)—O(4)	106.7 (2)
C(20)—O(4)—C(21)	114.5 (2)	O(4)—C(21)—O(5)	124.3 (3)
O(4)—C(21)—C(22)	109.8 (2)	O(5)—C(21)—C(22)	125.8 (2)
C(21)—C(22)—C(23)	111.8 (2)	C(22)—C(23)—C(24)	113.7 (2)
C(1)—C(38)—O(7)	108.8 (2)	C(38)—O(7)—C(39)	117.6 (2)
O(7)—C(39)—O(8)	123.0 (3)	O(7)—C(39)—C(40)	112.8 (2)
C(39)—C(40)—C(41)	115.9 (2)	O(8)—C(39)—C(40)	123.7 (2)
C(40)—C(41)—C(42)	116.1 (2)	C(1)—C(56)—O(10)	109.4 (2)
C(56)—O(10)—C(57)	117.4 (2)	O(10)—C(57)—O(11)	123.3 (3)
O(10)—C(57)—C(58)	109.2 (2)	C(57)—C(58)—C(59)	111.1 (2)
O(11)—C(57)—C(58)	127.4 (2)	C(58)—C(59)—C(60)	116.7 (2)

C(1)—C(2)—O(1)—C(3)	−177.5 (7)	C(1)—C(20)—O(4)—C(21)	−177.2 (6)
C(2)—O(1)—C(3)—C(4)	172.7 (7)	C(20)—O(4)—C(21)—C(22)	−179.4 (6)
O(1)—C(3)—C(4)—C(5)	172.8 (7)	O(4)—C(21)—C(22)—C(23)	178.1 (6)
C(3)—C(4)—C(5)—C(6)	−75.4 (9)	C(21)—C(22)—C(23)—C(24)	−78.2 (8)
C(4)—C(5)—C(6)—C(7)	124.6 (8)	C(22)—C(23)—C(24)—C(25)	126.2 (8)
C(4)—C(5)—C(6)—C(11)	−57.6 (10)	C(22)—C(23)—C(24)—C(29)	−55.6 (9)
C(1)—C(38)—O(7)—C(39)	135.1 (7)	C(1)—C(56)—O(10)—C(57)	134.4 (7)
C(38)—O(7)—C(39)—C(40)	−172.9 (6)	C(56)—O(10)—C(57)—C(58)	−171.6 (6)
O(7)—C(39)—C(40)—C(41)	−167.0 (7)	O(10)—C(57)—C(58)—C(59)	−167.4 (6)
C(39)—C(40)—C(41)—C(42)	67.7 (9)	C(57)—C(58)—C(59)—C(60)	69.3 (8)
C(40)—C(41)—C(42)—C(43)	−161.4 (7)	C(58)—C(59)—C(60)—C(61)	−166.0 (7)
C(40)—C(41)—C(42)—C(47)	22.3 (10)	C(58)—C(59)—C(60)—C(65)	20.1 (10)

between the two pairs of chains C(2)–C(19), C(20)–C(37) and C(38)–C(54), C(56)–C(73). The phenyl rings show deviations from planarity: r.m.s.d.'s 0.0255 [C(6)–C(11)], 0.0069 [C(24)–C(29)], 0.0168 [C(42)–C(47) and 0.0074 Å [C(60)–C(65)].

The steric hindrance induced by the presence of two *tert*-butyl moieties in *ortho* positions to each phenolic OH group prevents the formation of strong intermolecular hydrogen bonds. However, two weak intermolecular interactions between phenolic OH and ester O=C groups [O(3)···O(11)(x, $1-y$, $z+\frac{1}{2}$) 3.14 (1) and O(6)···O(8)(x, $1-y$, $z-\frac{1}{2}$) 3.17 (1) Å] are responsible for the formation of layers parallel to the *ab* plane. No hydrogen bond is formed between different layers.

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References

Burla, M. C., Camalli, M., Cascarano, G., Giacovazzo, C., Polidori, G., Spagna, R. & Viterbo, D. (1989). *J. Appl. Cryst.* **22**, 389–393.

Dexter, M., Spivack, J. D. & Steinberg, D. H. (1966). US Patent No. 3285855.

Hasui, H., Furihata, T., Takanashi, K. & Ohmori, M. (1981). European Patent No. 32459.

Immirzi, A. (1973). *J. Appl. Cryst.* **6**, 246–249.

Millini, R. (1990). Unpublished results.

Motherwell, W. D. S. & Clegg, W. (1978). *PLUTO*. Program for plotting molecular and crystal structures. Univ. of Cambridge, England.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

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Structure of an Organic Phosphate: $O{=}P(OCH_2)_3CCH_2CH_3$

By B. D. Santarsiero*

Structure Determination Laboratory, Department of Chemistry, University of Alberta, Edmonton, Alberta, Canada T6G 2G2

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Abstract. 4-Ethyl-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane 1-oxide, $C_6H_{11}O_4P$, $M_r = 178.13$, orthorhombic, *Pbca*, $a = 11.356$ (1), $b = 11.771$ (1), $c = 12.290$ (1) Å, $V = 1642.8$ (2) Å^3, $Z = 8$, $D_x = 1.440$ g cm^{-3}, λ(Mo $K\alpha$) = 0.7107 Å, $\mu = 2.91$ cm^{-1}, $F(000) = 752$, $T \simeq 295$ K, goodness of fit = 1.76, $R = 0.047$, $wR = 0.059$ for 802 reflections with $I > 3\sigma(I)$. The P=O bond length is 1.454 Å, and the average P—O, O—C and C—C bond lengths are 1.583, 1.468 and 1.541 Å (all corrected for librational motion). The average O=P—O and O—P—O angles are 114.3 and 104.2°.

Introduction. Phosphines and phosphites are commonly employed as ligands in organometallic synthesis, and a number of organometallic–phosphite complexes have been prepared with use of the ligand 4-ethyl-2,6,7-trioxa-1-phosphabicyclo[2.2.2]octane, $P(OCH_2)_3CCH_2CH_3$. It is easily oxidized to the phosphate in air. The phosphite and the phosphate triesters have, nominally, C_{3v} symmetry. Since no report on the structure has appeared, we report herein on a precise determination of the metrical details of this $O{=}P(OR)_3$ structure.

Experimental. Single clear colorless crystals from a cold solution of toluene/hexane. Specimen, 0.12 × 0.20 × 0.32 mm, mounted on a glass fiber with epoxy. Space group *Pbca*, No. 61 (systematic absences: $0kl$, k odd, $h0l$, l odd, and $hk0$, h odd). Diffractometry: Enraf–Nonius CAD-4 automated diffractometer, 295 K, Mo $K\alpha$ radiation with graphite monochromator, $\lambda = 0.7107$ Å. Cell constants and orientation matrix by least-squares refinement of the setting angles of 22 reflections, determined at $\pm\theta$, in the range $19.9 < \theta < 25{\cdot}1°$. Intensity data with ω–2θ scans at 2.0° min^{-1} (in θ), ω-scan width = $(0.60 + 0.347 \tan\theta)°$, maximum $2\theta = 60°$, ($\pm h$, $-k$, $\pm l$, in the range h 0–15, k 0–16, l 0–17). Three check reflections every 120 min, average variation approximately 4.6%; no decay correction employed. A total of 6455 intensities were collected, and reduced to F_o's. Structure derivation: *MITHRIL* (Gilmore, 1983). Refinement: full-matrix least squares on F, weight $w = 4F_o^2/\sigma^2(F_o^2)$, analytic scattering factors with f' and f'' components of anomalous dispersion (*International Tables for X-ray Crystallography*, 1974, Vol. IV, Table 2.2B) included in the calculations of all non-H atoms.

The H atoms were generated at idealized calculated positions by assuming a C—H bond length of 0.95 Å and the appropriate sp^3 geometry. On the methyl group, they were idealized by least-

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* Present address: Molecular Structure Corporation, Macromolecular Crystallography Division, 3200 Research Forest Drive, The Woodlands, TX 7738-4238, USA.

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