

Structure of a Substituted Octane

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Abstract. 4,5-Bis(1-hydroxy-1-methylethyl)-2,7-dimethyloctane-2,7-diol, $C_{16}H_{34}O_4$, $M_r = 290.44$, monoclinic, $C2/c$, $a = 16.411$ (9), $b = 6.669$ (4), $c = 16.245$ (9) Å, $\beta = 96.01$ (5)°, $V = 1768.2$ Å³, $Z = 4$, $D_m = 1.08$, $D_x = 1.09$ Mg m⁻³, $\lambda(\text{Cu K}\alpha) = 1.54178$ Å, $\mu = 0.61$ mm⁻¹, $F(000) = 648$, $T = 293$ K. Final $R = 0.061$ for 819 unique observed reflections. The asymmetric molecule of $C_{16}H_{34}O_4$ is related by a twofold axis through the bond C(12)–C(12') [1.566 (3) Å] to the other half. Intermolecular contacts involve hydrogen bonds of the type O(11)···O(21) 2.741 (3) Å with angle O(11)–H(11)···O(21) 173 (3)°. These produce chains in the [100] direction.

Experimental. Crystals of the title compound crystallized from ethanol solution as the subsidiary product in a photosynthesis of dimethylvinylmethanol. The formula $C_{16}H_{34}O_4$ was determined by means of chemical analysis. On the basis of X-ray analysis, the material was identified as 4,5-bis(1-hydroxy-1-methylethyl)-2,7-dimethyloctane-2,7-diol which can be considered as a dimer of 2,5-dimethylhexane-2,5-diol.

Colourless crystal with dimensions $0.25 \times 0.25 \times 0.75$ mm; D_m by flotation; monoclinic space group $C2/c$ (No. 15). Many single crystals examined were of poor quality, but a satisfactory specimen was examined with a Syntex $P2_1$ diffractometer. Triclinic Bravais translation lattice given by program *UB* (Sivý, Sivý & Koreň, 1987). Data collection was performed with lattice parameters $a = 6.669$ (4), $b = 8.858$ (7), $c = 16.245$ (11) Å, $\alpha = 84.45$ (6), $\beta = 89.94$ (5), $\gamma = 67.94$ (5)°, Cu $K\alpha$ radiation, graphite monochromator, $\theta/2\theta$ scan, $2\theta_{\max} = 110^\circ$, time per reflection ca 60 s, two standard reflections, variation 1.0%; 25 reflections with $10.9 < 2\theta < 29.0^\circ$ used for refinement of lattice parameters; min. and max. transmission

factors are the same and equal 0.8475 (absorption correction not applied); index range $0 \leq h \leq 5$, $-8 \leq k \leq 8$, $-16 \leq l \leq 16$; 2072 reflections measured.

At this stage a second crystal was examined and diffractometrically gained refined angles gave a new monoclinic solution (Sivý, Sivý & Koreň, 1987) which was confirmed from Weissenberg photographs. The transformation matrix $(\bar{1}20/100/00\bar{1})$ between new and old lattice parameters was found by program *TRALAP* (Sivý, 1987). Intensities from the triclinic measurement of the single crystal were used further. The index range is now $0 \leq h \leq 16$, $0 \leq k \leq 6$, $-16 \leq l \leq 16$; 910 unique reflections, 819 reflections observed with $I > 2.0\sigma(I)$, 91 unobserved. Data reduction carried out with program *XP21* (Pavelčík, 1987). Initially, structure solution was attempted unsuccessfully with *MULTAN80* (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). For a second attempt a five-carbon tetrahedral unit was input to program *PATSEE* (Egert, 1985). Its located position, input to *SHELXS86* (Sheldrick, 1986), gave the positions of all the non-H atoms. These were isotropically refined as C atoms by full-matrix least squares using F values (unit weights); the O atoms were selected on the basis of thermal motion according to the lowest values of the thermal coefficients ($U = 0.0080\text{--}0.0176$ Å²). After anisotropic least-squares refinement H-atom positions were calculated and isotropically refined (159 parameters), all by program *SHELX76* (Sheldrick, 1976). Scattering factors and f' , f'' from *International Tables for X-ray Crystallography* (1974). Max. positive and max. negative electron density in final difference Fourier synthesis 0.18 and -0.29 e Å⁻³; $(\Delta/\sigma)_{\max} = 0.013$ (H atom) in final refinement cycle; final $R = 0.061$, $wR = 0.073$, weighting scheme $w = 1.0/[\sigma^2(F_o) + 0.014F_o^2]$. Calculations performed using an

EC-1045 computer, J. A. Komenský University, Bratislava, Czechoslovakia.

The numbering of atoms is shown in Fig. 1 and a projection of the crystal structure in Fig. 2. Fractional atomic coordinates for non-H atoms are in Table 1;* bond distances and angles are in Table 2.

Related literature. Other tetrahedral conformations have been studied for 2,5-dimethylhexane-2,5-diol (at 110 K) by Helmholdt & Reynaers (1976) and for the tetrahydrate by Jeffrey & Shen (1972).

*Lists of structure amplitudes, anisotropic thermal parameters, H-atom parameters and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51461 (11 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

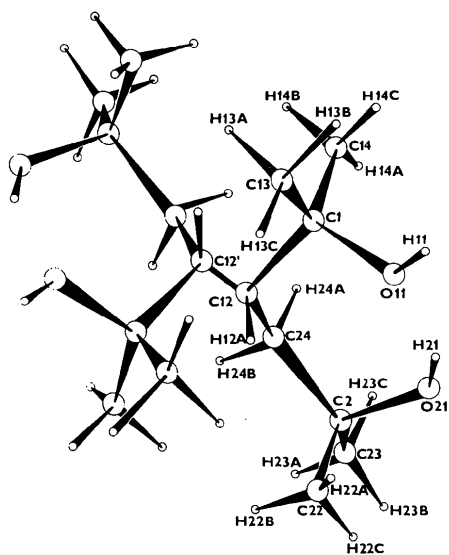


Fig. 1. Structure of the title compound showing the atomic numbering.

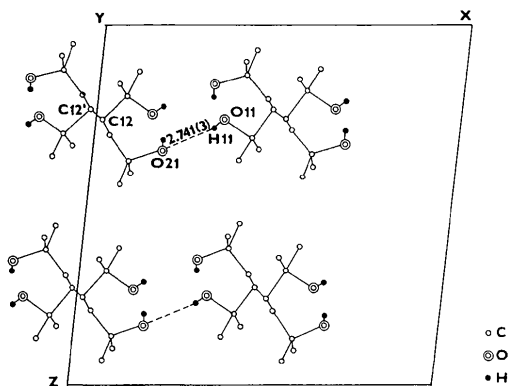


Fig. 2. Projection of the crystal structure along the y axis.

Table 1. Fractional atomic coordinates and equivalent isotropic thermal parameters (\AA^2) with e.s.d.'s in parentheses for non-H atoms

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	x	y	z	U_{eq}
C(1)	0.1015 (2)	0.4178 (4)	0.1997 (2)	0.038 (1)
O(11)	0.1791 (1)	0.4585 (4)	0.2463 (1)	0.051 (1)
C(12)	0.0468 (2)	0.3382 (4)	0.2656 (2)	0.032 (1)
C(13)	0.0710 (2)	0.6191 (6)	0.1628 (3)	0.060 (1)
C(14)	0.1149 (3)	0.2725 (7)	0.1318 (3)	0.067 (2)
C(2)	0.1436 (2)	0.1293 (4)	0.3783 (2)	0.043 (1)
O(21)	0.2225 (1)	0.1677 (3)	0.3497 (1)	0.048 (1)
C(22)	0.1309 (2)	0.2803 (7)	0.4455 (2)	0.068 (2)
C(23)	0.1512 (3)	-0.0843 (7)	0.4107 (3)	0.068 (2)
C(24)	0.0754 (2)	0.1367 (4)	0.3062 (2)	0.037 (1)

Table 2. Bond distances (\AA) and angles ($^\circ$) with e.s.d.'s in parentheses for non-H atoms

C(1)—O(11)	1.438 (4)	C(2)—O(21)	1.444 (4)
—C(12)	1.560 (5)	—C(22)	1.516 (5)
—C(13)	1.533 (5)	—C(23)	1.519 (6)
—C(14)	1.502 (6)	—C(24)	1.534 (5)
C(12)—C(24)	1.548 (4)		
C(13)—C(1)—C(14)	110.2 (3)	O(21)—C(2)—C(22)	108.0 (3)
—C(12)	112.2 (3)	—C(23)	103.5 (3)
—O(11)	106.0 (3)	—C(24)	110.9 (3)
C(12)—C(1)—C(14)	115.1 (3)	C(22)—C(2)—C(23)	112.7 (3)
—O(11)	104.2 (3)	—C(24)	112.6 (3)
O(11)—C(1)—C(14)	108.6 (3)	C(23)—C(2)—C(24)	108.8 (3)
C(1)—C(12)—C(24)	114.7 (3)	C(12)—C(24)—C(2)	121.2 (2)

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