dimensions $0.56 \times 0.36 \times 0.30$ mm. Systematic absences $h0l$, $h + l \neq 2n$; $0k0$, $k \neq 2n$ indicating space group $P2_1/n$ (non-standard setting of $P2_1/c$).

Heavy atoms located from a Patterson synthesis and the light atoms then found on successive Fourier syntheses, including one molecule of solvent CH_2Cl_2. Anisotropic temperature factors for all non-H atoms. H atoms given fixed isotropic temperature factors, $B = 5.0 \text{ Å}^2$, inserted at calculated positions and not refined. Final refinement on F by large-block least-squares methods. Largest positive and negative peaks on a final difference Fourier synthesis of height $\pm 5 \text{ e Å}^{-3}$ produced by diffraction ripples from U. A weighting scheme of the form $W = X.Y$ applied, where $X = 1.0$ for $(\sin\theta/\lambda) \geq 0.35 \text{ Å}^{-1}$ and $Y = 1.0$ or $110.0/F_{obs}$ if $F_{obs} \geq 110.0$, and shown to be satisfactory by a weighting analysis. Max. Δ/σ in final cycle 0.9. Computing with $XRAY76$ (Stewart, 1976) on Burroughs B6800. Scattering factors in the analytical form and anomalous-dispersion factors taken from $International$ $Tables$ for X-ray $Crystallography$ (1974). Final atomic coordinates are given in Table 1, and bond lengths and angles in Table 2.* Fig. 1 shows a view of the molecule.

Related literature. $[UO_2(OPPh_3)_2(NO_3)_2]$ (Alcock, Roberts & Brown, 1982); $[UO_2(OPPh_3)_2Cl_2]$ (Bombieri, Forsellini, Day & Azeez, 1978).

We thank SERC for studentship support (DJF).

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* Lists of structure factors, anisotropic thermal parameters and phenyl-group dimensions have been deposited with British Library Lending Division as Supplementary Publication No. SUP 42707 (33 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
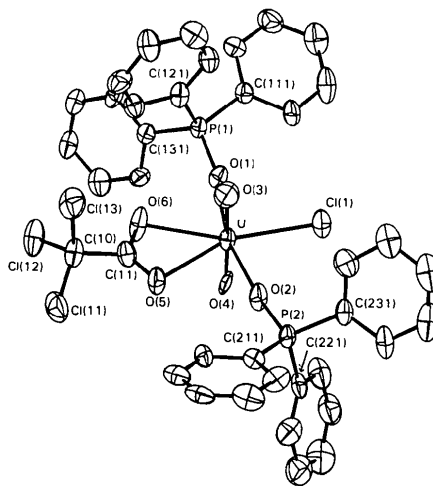

Fig. 1. View of the molecule, showing the atomic numbering (H atoms omitted for clarity).

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Structure of 1,3:2,5:4,6-Tri-O-methylene-D-mannitol*

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Abstract. $C_9H_{14}O_6$, $M_r = 218.2$, orthorhombic, $P2_12_12_1$, $a = 14.323$ (2), $b = 15.754$ (2), $c = 4.292$ (1) Å, $V = 968.5 \text{ Å}^3$, $Z = 4$, $D_m = 1.50$ (1), $D_x = 1.496 \text{ g cm}^{-3}$, Cu $K\alpha$ $(\lambda\alpha_1 = 1.54056 \text{ Å})$, $\mu = 10.4 \text{ cm}^{-1}$, $F(000) = 464$, $T = 296$ K, final $R = 0.040$ for 538 unique observed reflections. The analysis confirms the formulation. The molecule has a non-crystallographic twofold rotation axis passing through the 2,5-O methylene C atom and the mid-point of the C(3)–C(4) bond. The torsion angle $\varphi[C(2)C(3)C(4)C(5)]$ of this bond is 48.8 (6)°; the dioxepane ring is in the twist-chair conformation.
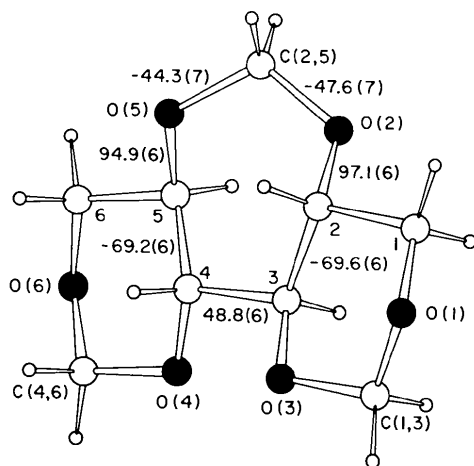
Fig. 1. A view of the molecule. O atoms are represented by filled circles; H atoms (small open circles) are in assumed positions. Torsion angles (°) are given for the bonds of the dioxepane ring.

Table 1. *Atomic coordinates (e.s.d.'s) and equivalent isotropic temperature factors for the non-H atoms*

$$B_{eq} = (8\pi^2/3)(U_{11} + U_{22} + U_{33}).$$

	x	y	z	$B_{eq}(\text{Å}^2)$
C(1)	0·1565 (4)	0·0476 (4)	0·9398 (18)	4·3
C(2)	0·0724 (3)	0·1051 (3)	0·9735 (16)	3·0
C(3)	−0·0145 (3)	0·0576 (3)	0·8570 (13)	2·5
C(4)	−0·1049 (3)	0·1055 (3)	0·8995 (14)	2·5
C(5)	−0·1013 (3)	0·1972 (3)	0·7841 (15)	2·5
C(6)	−0·1988 (3)	0·2355 (3)	0·8073 (16)	3·2
O(1)	0·1397 (3)	−0·0294 (2)	1·1134 (11)	4·5
O(2)	0·0878 (2)	0·1757 (2)	0·7664 (11)	3·1
O(3)	−0·0214 (2)	−0·0200 (2)	1·0420 (11)	3·5
O(4)	−0·1759 (2)	0·0610 (2)	0·7347 (11)	3·0
O(5)	−0·0419 (2)	0·2462 (2)	0·9823 (11)	3·3
O(6)	−0·2642 (2)	0·1826 (2)	0·6488 (10)	3·7
C(1,3)	0·0593 (4)	−0·0698 (4)	0·9917 (17)	4·2
C(2,5)	0·0517 (4)	0·2532 (4)	0·8736 (17)	4·0
C(4,6)	−0·2642 (3)	0·1001 (3)	0·7755 (16)	3·2

Table 2. *Bond lengths (Å) and angles (°) and their e.s.d.'s (see remark in text concerning e.s.d.'s)*

C(1)–C(2)	1·514 (8)	O(3)–C(3)	1·461 (6)
C(2)–C(3)	1·536 (7)	O(3)–C(1,3)	1·414 (6)
C(3)–C(4)	1·510 (7)	O(4)–C(4)	1·423 (6)
C(4)–C(5)	1·528 (7)	O(4)–C(4,6)	1·418 (6)
C(5)–C(6)	1·525 (7)	O(5)–C(5)	1·429 (7)
O(1)–C(1)	1·444 (8)	O(5)–C(2,5)	1·424 (7)
O(1)–C(1,3)	1·416 (7)	O(6)–C(6)	1·426 (7)
O(2)–C(2)	1·441 (7)	O(6)–C(4,6)	1·409 (7)
O(2)–C(2,5)	1·403 (7)		
C(2)–C(1)–O(1)	108·7 (5)	C(6)–C(5)–O(5)	107·0 (4)
C(1)–C(2)–C(3)	108·8 (5)	C(5)–C(6)–O(6)	109·8 (4)
C(1)–C(2)–O(2)	106·3 (5)	C(1)–O(1)–C(1,3)	108·8 (5)
C(3)–C(2)–O(2)	107·4 (5)	C(2)–O(2)–C(2,5)	114·4 (5)
C(2)–C(3)–C(4)	114·3 (4)	C(3)–O(3)–C(1,3)	109·0 (4)
C(2)–C(3)–O(3)	106·6 (4)	C(4)–O(4)–C(4,6)	111·2 (4)
C(4)–C(3)–O(3)	107·1 (4)	C(5)–O(5)–C(2,5)	114·0 (5)
C(3)–C(4)–C(5)	113·9 (4)	C(6)–O(6)–C(4,6)	110·8 (4)
C(3)–C(4)–O(4)	107·8 (4)	O(1)–C(1,3)–O(3)	111·1 (5)
C(5)–C(4)–O(4)	109·2 (4)	O(2)–C(2,5)–O(5)	112·8 (4)
C(4)–C(5)–C(6)	108·8 (4)	O(4)–C(4,6)–O(6)	110·7 (4)
C(4)–C(5)–O(5)	109·7 (4)		

Experimental. The material was synthesized by Dr M. A. Ragan, of this laboratory, following Ness, Hann & Hudson (1943) and crystallized (colourless needles [001]) from the reaction mixture. Specimen dimensions $0.05 \times 0.07 \times 0.14$ mm, density by flotation in aqueous KI solution. Picker four-circle diffractometer with Ni-filtered Cu $K\alpha$ radiation, θ–2θ scan, $2\theta < 130°$. Cell constants from 30 reflections, $107 < 2\theta < 129°$. Max. $(\sin\theta)/\lambda = 0.588$ Å^{-1}, $h = 0$ to 16, $k = 0$ to 18, $l = 0$ to 5. One standard reflection (120) measured every 60 reflections, with mean intensity 40243(520) counts; no indication of specimen decay. 1003 unique reflections scanned, 465 deemed to be unobserved $[I_{net} < 3\sigma(I_{net})$; σ evaluated from counting statistics]. Absorption corrections not applied. Net intensities derived by analysis of individual reflection profiles (Grant & Gabe, 1978). Structure solved by direct methods (*MULTAN78*; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978), and refined by block-diagonal least squares, minimizing $\sum w\Delta F^2$, where $w^{-1} = \sigma^2(F_o) + 0.0007F_o^2$. H atoms assigned reasonable parameters (C–H = 1·08 Å; $U = 0.04$ Å^2). Parameters refined: scale factor, secondary-extinction coefficient (1·09 $\times 10^{-4}$ mm; Larson, 1970), atomic coordinates and anisotropic temperature factors for C and O. Final $R = 0.040$ (0·11 including unobserved reflections); $wR = 0.048$, $S = 1.17$. Ratio of max. parameter shift to corresponding e.s.d. in final cycle 0·05. Density in final difference-Fourier synthesis did not exceed the limits ± 0.16 e Å^{-3}. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974). The computer program system has been described by Larson & Gabe (1978). The structure is defined in Fig. 1 and Tables 1 and 2.* A comparison of chemically equivalent quantities in Table 2 suggests that the e.s.d.'s have been underestimated by a factor of two.

Related literature. The first isolation is described by Schulz & Tollens (1896). Of the possible conformational isomers, that found for the crystal structure was correctly inferred from synthetic studies (Ness *et al.*, 1943) and from proton NMR studies (Grindley, Stoddart & Szarek, 1969).

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Structures of Two $C_{20}H_{24}O_4$* Dimers of 3-Acetyl-6,6-dimethylfulvene Epoxide

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Abstract. Passage of the hydroperoxide (III) over silica gel leads to the formation of two *endo,syn* diepoxide dimers [(I), (II)] [Thomas, Perret & Bernardinelli (1985). *Chimia*, **39**, 228–229] of 3-acetyl-6,6-dimethyl-fulvene epoxide (IV), the crystal structures of which are reported here. (I): $C_{20}H_{24}O_4$, m.p. 411 K, $M_r = 328\cdot4$, monoclinic, $P2_1/n$, $a = 13\cdot100$ (3), $b = 8\cdot6517$ (11), $c = 15\cdot805$ (3) Å, $\beta = 93\cdot69$ (2)°, $V = 1787\cdot6$ (6) Å^3, $Z = 4$, $D_x = 1\cdot220$ Mg m^{-3}, λ(Mo $K\alpha$) = $0\cdot7107$ Å, $\mu = 0\cdot0784$ mm^{-1}, room temperature, $R = 0\cdot044$ for 1193 observed reflections [$|F_o| \geq 3\sigma(F_o)$ and $|F_o| \geq 7\cdot0$]. (II): $C_{20}H_{24}O_4$, m.p. 425 K, $M_r = 328\cdot4$, orthorhombic, $P2_12_12_1$, $a = 6\cdot9764$ (17), $b = 14\cdot892$ (2), $c = 17\cdot043$ (5) Å, $V = 1770\cdot6$ (7) Å^3, $Z = 4$, $D_x = 1\cdot232$ Mg m^{-3}, λ(Mo $K\alpha$) = $0\cdot7107$ Å, $\mu = 0\cdot0791$ mm^{-1}, room temperature, $R = 0\cdot055$ for 1134 observed reflections [$|F_o| \geq 4\sigma(F_o)$]. Apart from the position of the acetyl group on the double bond C(9)–C(10), the two isomers reveal no quantifiable differences in their individual conformation. There are no unusual distances or angles.
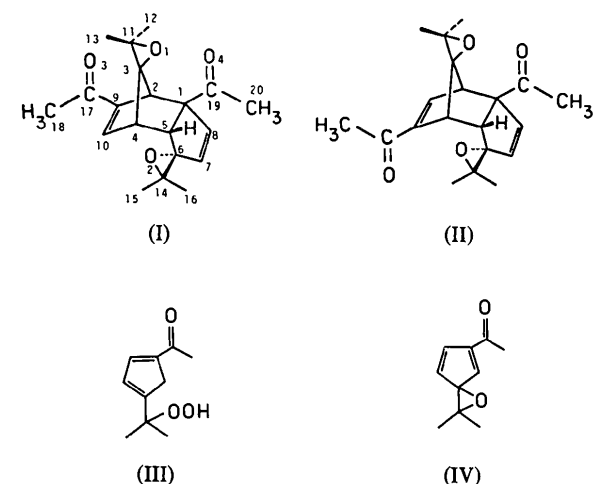
Experimental. Experimental data and structure refinement are summarized in Table 1.† Both crystals were furnished by Firmenich S. A. Research Laboratories. Philips PW 1100 diffractometer, graphite-mono-chromated Mo $K\alpha$; $\omega/2\theta$ scans; Lorentz–polarization correction; no absorption correction; structures solved by *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Atomic scattering factors from *International Tables for X-ray Crystallography* (1974); no secondary-extinction correction; all calculations performed with a local version of *XRAY*76 (Stewart, Machin, Dickinson, Ammon, Heck & Flack, 1976) and *ORTEP*II (Johnson, 1976). Table 2 gives the positional parameters and Table 3 bond distances and torsion angles. Fig. 1 shows stereoviews of the two isomers.

(I)

(II)

(III)

(IV)

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* 3a′,5′-Diacetyl-3a′,4′,7′,7a′-tetrahydro-3,3,3″,3‴-tetramethyl-dispiro[oxirane-2,1′-[4,7]-methano-[1*H*]-indene-8′,2″-oxirane] (I) and 3a′,6′-diacetyl-3a′,4′,7′,7a′-tetrahydro-3,3,3″,3‴-tetramethyl-dispiro[oxirane-2,1′-[4,7]-methano-[1*H*]-indene-8′,2″-oxirane] (II).

† Lists of structure factors, atomic positional and thermal parameters for all atoms and other information in the printed form of the Standard Crystallographic File Structure of Brown (1985) have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42706 (67 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.