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Structure of *cis*-Bicyclo[3.3.0]oct-3-ene-2,7-dione 7-(2,2-Dimethyltrimethylene Acetal)

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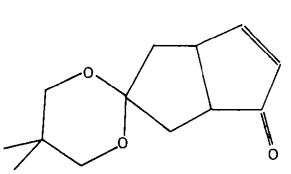
Abstract. $C_{13}H_{18}O_3$, $M_r = 222{\cdot}28$, orthorhombic, *Pbca*, $a = 19{\cdot}633\,(3)$, $b = 12{\cdot}211\,(2)$, $c = 10{\cdot}147\,(2)$ Å, $V = 2433\,(1)$ Å^3, $D_x = 1{\cdot}214\,(1)$ Mg m^{-3}, $Z = 8$, λ(Mo $K\alpha$) = 0·71069 Å, μ(Mo $K\alpha$) = 0·091 mm^{-1}, $F(000) = 976$, room temperature, $R = 0{\cdot}048$ for 1045 observed reflections. The six-membered ring has a chair conformation. The two five-membered rings have a skew-envelope and an envelope form. Most of the bond distances and angles are normal; deviations are due to electronic conjugations and steric effects.

Introduction. The title compound (I) was synthesized by Carceller, Moyano & Serratosa (1984) and it is a synthetic intermediate in which all eight C atoms are properly activated. This compound can undergo a series of chemo-, regio- and stereoselective reactions useful for the synthesis of either natural or non-natural polyfused cyclopentanoid systems.



(I)

Experimental. Colourless prism (0·1 × 0·1 × 0·2 mm). Philips PW 1100 diffractometer. Unit-cell parameters from 25 reflections ($4 \le \theta \le 11°$). Mo $K\alpha$, graphite monochromator, ω-scan technique, scan width 1°, scan speed 0·03° s^{-1}. 1076 independent reflections ($2 \le \theta \le 25°$); 1045 with $I \ge 2{\cdot}5\sigma(I)$. Max. $h,k,l = 20,13,12$. Three reflections every 2 h as control, significant variations not observed. Lorentz–polarization correction; no absorption or extinction correction. Attempts to solve the structure by direct methods failed. A model was derived from a Patterson synthesis using *SHELX*76 (Sheldrick, 1976) and *DIRDIF* (Beurskens, Bosman, Doesburg, Gould, van der Hark, Prick, Noordik, Beurskens & Parthasarathi, 1981). This model was introduced into *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), using the Debye normalization. Refinements by full-matrix least-squares method, using *SHELX*76 and minimizing $\sum w||F_o| - |F_c||^2$, with $w = [\sigma^2(F_o) + 0{\cdot}026|F_o|^2]^{-1}$. H from $\Delta\rho$ map and refined with an overall isotropic temperature factor; non-hydrogen atoms refined as anisotropically vibrating; f, f' and f'' were taken from *International Tables for X-ray Crystallography* (1974). Final $R = 0{\cdot}048$ ($wR = 0{\cdot}054$). $(\Delta/\sigma)_{max} = 0{\cdot}13$ for x coordinate of C(4). Max. and min. peaks in final $\Delta\rho$ map 0·1 [0·93 Å from H(C14)′] and −0·2 e Å^{-3}, respectively.

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Table 1. *Final atomic coordinates* ($\times 10^4$) *and equivalent isotropic thermal parameters* (Å^2)

$$B_{eq} = \tfrac{8}{3}\pi^2 \sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i . \mathbf{a}_j .$$

	x	y	z	B_{eq}
O(1)	7912 (1)	1822 (2)	7269 (2)	3·56 (11)
C(2)	8446 (2)	1347 (3)	6488 (4)	3·93 (17)
C(3)	9131 (2)	1378 (3)	7199 (4)	3·84 (17)
C(4)	9019 (2)	862 (3)	8548 (4)	3·97 (18)
O(5)	8462 (1)	1380 (2)	9234 (2)	3·85 (12)
C(6)	7840 (2)	1331 (3)	8535 (4)	3·38 (15)
C(7)	7304 (2)	1983 (3)	9253 (4)	3·79 (17)
C(8)	6628 (2)	1546 (3)	8706 (4)	3·99 (17)
C(9)	6382 (2)	2128 (3)	7462 (4)	4·39 (19)
C(10)	6312 (2)	1306 (3)	6416 (4)	4·33 (19)
C(11)	6531 (2)	340 (3)	6845 (4)	4·25 (19)
C(12)	6763 (2)	355 (3)	8261 (4)	3·81 (17)
C(13)	7533 (2)	165 (3)	8457 (4)	3·92 (17)
C(14)	9638 (2)	696 (4)	6429 (5)	5·55 (26)
C(15)	9373 (3)	2557 (4)	7371 (6)	5·49 (23)
O(16)	6274 (2)	3107 (3)	7361 (4)	6·67 (18)

Table 2. *Bond lengths* (Å) *and angles* (°)

C(2)–O(1)	1·436 (4)	C(13)–C(6)	1·548 (5)
C(6)–O(1)	1·425 (4)	C(8)–C(7)	1·533 (5)
C(3)–C(2)	1·527 (5)	C(9)–C(8)	1·527 (6)
C(4)–C(3)	1·524 (5)	C(12)–C(8)	1·546 (5)
C(14)–C(3)	1·514 (6)	C(10)–C(9)	1·468 (6)
C(15)–C(3)	1·525 (6)	O(16)–C(9)	1·218 (5)
O(5)–C(4)	1·443 (5)	C(11)–C(10)	1·330 (6)
C(6)–O(5)	1·413 (4)	C(12)–C(11)	1·507 (6)
C(7)–C(6)	1·507 (5)	C(13)–C(12)	1·542 (5)
C(6)–O(1)–C(2)	113·6 (2)	C(13)–C(6)–C(7)	103·8 (3)
C(3)–C(2)–O(1)	111·9 (3)	C(8)–C(7)–C(6)	104·2 (3)
C(4)–C(3)–C(2)	106·6 (3)	C(9)–C(8)–C(7)	114·3 (3)
C(14)–C(3)–C(2)	108·7 (3)	C(12)–C(8)–C(7)	106·5 (3)
C(14)–C(3)–C(4)	109·3 (3)	C(12)–C(8)–C(9)	104·5 (3)
C(15)–C(3)–C(2)	110·6 (3)	C(10)–C(9)–C(8)	108·0 (3)
C(15)–C(3)–C(4)	109·4 (4)	O(16)–C(9)–C(8)	125·6 (4)
C(15)–C(3)–C(14)	112·0 (4)	O(16)–C(9)–C(10)	126·4 (4)
O(5)–C(4)–C(3)	111·2 (3)	C(11)–C(10)–C(9)	109·8 (4)
C(6)–O(5)–C(4)	113·2 (3)	C(12)–C(11)–C(10)	113·6 (4)
O(5)–C(6)–O(1)	110·4 (2)	C(11)–C(12)–C(8)	103·8 (3)
C(7)–C(6)–O(1)	106·5 (3)	C(13)–C(12)–C(8)	105·8 (3)
C(7)–C(6)–O(5)	109·8 (3)	C(13)–C(12)–C(11)	114·7 (3)
C(13)–C(6)–O(1)	112·3 (3)	C(12)–C(13)–C(6)	104·4 (3)
C(13)–C(6)–O(5)	113·6 (3)		

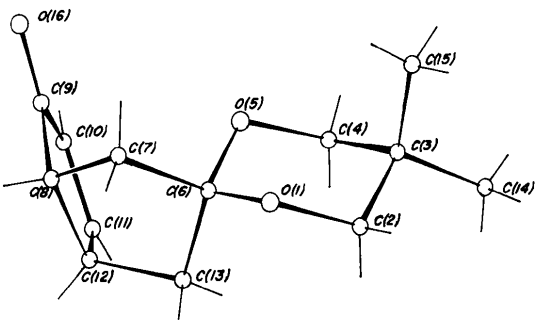

Fig. 1. View of the molecule with the numbering of the atoms.

Discussion. Final atomic coordinates and bond lengths and angles are listed in Tables 1 and 2, respectively.* A view of the molecule with the numbering of the atoms is shown in Fig. 1. The six-membered ring has a chair conformation [deviations of the two atoms from the mean plane C(3) = 0·679 (5) and C(6) = −0·624 (5) Å]. The O(1)–C(6) and O(5)–C(6) bonds have an antiperiplanar form, and the six-membered ring has the C(6)–C(7) as equatorial plane. The shortening of the equatorial plane [C(6)–C(7) 1·507 (5) Å] with respect to the axial bond length [C(6)–C(13) 1·548 (5) Å] has also been observed in the three isomers with formula $C_{15}H_{20}O_3$ (Carceller, Moyano, Serratosa, Solans & Font-Altaba, 1985). This explains the widening of the C(13)–C(6)–O bond angles [average value 112·9 (6)°]. The two five-membered rings have a skew-envelope [C(6),C(7),C(8),C(12) and C(13)] and an envelope [C(8),C(9),C(10),C(11) and C(12)] form, with C(6) and C(8) out of plane [deviation: −0·574 (6) and −0·081 (6) Å, respectively]. The conformation of the second ring explains the shortening of the C(9)–C(10) and C(11)–C(12) bond lengths, which are due to electronic conjugation of these bonds with bonds C(10)–C(11) and C(9)–O(16). The remaining bond lengths and angles have normal values [weighted mean values: C–C 1·526 (8), C–O 1·429 (10), C=O 1·218 (5) Å, C–C–C 109·2 (11), C–C–O 111·5 (3), C–O–C 113·3 (2) and C–C=O 126·0 (5)°]. Different molecules are linked by van der Waals forces.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and distances and angles involving H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 42347 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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