fixed; $\sum w(|F_o| - |F_c|)^2$ minimized; $w = 4F_o^2/(\sigma F_o^2)^2$, where $(\sigma F_o^2)^2 = [s^2(c + R^2B) + (pF_o^2)^2]/\mathrm{Lp}^2$ and p (ignorance factor used to downweight intense reflections) = 0·065 (I) or 0·050 (II) with $I > 3\sigma(I)$ (Corfield, Doedens & Ibers, 1967); no absorption corrections applied; features in final $\Delta\rho$ map $\pm 0{\cdot}20$ e Å^{-3}; scattering factors and anomalous-dispersion values were taken from standard sources (*International Tables for X-ray Crystallography*, 1974; Stewart, Davidson & Simpson, 1965). (I): R(109 variables) = 0·058 and wR = 0·087 for 1041 independent reflections; S = 0·06; $(\Delta/\sigma)_{\max}$ = 0·001. (II): R(163 variables) = 0·066 and wR = 0·068 for 961 independent reflections; S = 0·2; $(\Delta/\sigma)_{\max}$ = 0·001. Final atomic coordinates are listed in Table 1,* and bond lengths and angles in Table 2. Figs. 1 and 2 show perspective views of (I) and (II) with atomic numbering.

Related literature. The inclination angles between the least-squares planes of the cyclohexane and carbamate groups, 87·5 (I) and 82·3° (II), show similarities to structures reported by Jasinski, Desper, Zentner, Butcher & Day (1988). Other related molecules reported by Gardner & Blackwell (1980), Forcier & Blackwell (1981) and Born, Hocker, Paulus & Wolfel (1981) differ in this comparison but show a number of other similarities.

We thank Louis Carreiro, US Army Materials Technology Laboratory (MTL), for providing NMR conformation on the CHDI samples. JPJ wishes to thank MTL and the US Army for an IPA fellowship and support of this work.

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters and packing diagrams have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44745 (21 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C**44**, 1139–1141

Structure of Methyl 8,9-Epoxy-5,5-ethylenedioxy-7-oxo-11-methoxytricyclo[7.2.1.0^{4,10}]dodec-3-ene-8-carboxylate

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(*Received* 6 *November* 1987; *accepted* 23 *February* 1988)

Abstract. $C_{17}H_{20}O_7$, M_r = 336·34, monoclinic, $C2/c$, a = 15·059 (6), b = 6·888 (2), c = 30·557 (12) Å, β = 94·30 (3)°, U = 3161 (2) Å^3, Z = 8, D_x = 1·413 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·0 cm^{-1}, F(000) = 1424, T = 295 K, R = 0·059 for 1169 reflections with $I \geq 1{\cdot}5\sigma(I)$. The carboxylate moiety is coplanar with the adjacent C—O bond of the epoxy group. The bond lengths and angles are as expected.

Experimental. X-ray data for a plate-shaped transparent colourless crystal (0·07 × 0·45 × 0·55 mm), glued on top of a glass fibre, were collected on an Enraf–Nonius CAD-4F diffractometer using Zr-filtered Mo $K\alpha$ radiation. The crystals were found to be poorly

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Table 1. *Final coordinates and equivalent isotropic thermal parameters of the non*-H *atoms and their e.s.d.'s in parentheses*

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

	x	y	z	$U_{\rm eq}$(Å^2)
O(1)	0·8898 (2)	0·2277 (6)	0·1627 (1)	0·042 (2)
O(2)	0·6947 (2)	−0·0149 (6)	0·0489 (1)	0·044 (2)
O(3)	0·6631 (2)	−0·0821 (6)	0·1949 (1)	0·046 (2)
O(4)	0·5846 (2)	0·1920 (7)	0·2100 (1)	0·050 (2)
O(5)	0·5724 (3)	−0·2381 (7)	0·0963 (2)	0·057 (2)
O(6)	0·5698 (3)	0·2089 (8)	−0·0012 (1)	0·065 (2)
O(7)	0·4789 (2)	0·2070 (6)	0·0538 (1)	0·047 (2)
C(1)	0·7505 (3)	0·1044 (8)	0·1243 (2)	0·029 (2)
C(2)	0·8401 (3)	0·2045 (9)	0·1217 (2)	0·037 (2)
C(3)	0·8114 (4)	0·398 (1)	0·1022 (2)	0·044 (3)
C(4)	0·7672 (4)	0·5185 (9)	0·1363 (2)	0·044 (2)
C(5)	0·7024 (3)	0·3993 (9)	0·1606 (2)	0·036 (2)
C(6)	0·6948 (3)	0·209 (1)	0·1561 (2)	0·031 (2)
C(7)	0·6241 (4)	0·090 (1)	0·1763 (2)	0·039 (2)
C(8)	0·5481 (3)	0·037 (1)	0·1417 (2)	0·038 (2)
C(9)	0·5775 (3)	−0·065 (1)	0·1018 (2)	0·038 (2)
C(10)	0·6173 (3)	0·0622 (9)	0·0676 (2)	0·034 (2)
C(11)	0·7065 (3)	0·1450 (9)	0·0793 (2)	0·033 (2)
C(12)	0·7435 (4)	0·335 (1)	0·0643 (2)	0·042 (2)
C(13)	0·5544 (4)	0·171 (1)	0·0348 (2)	0·041 (2)
C(14)	0·4106 (4)	0·311 (1)	0·0266 (2)	0·063 (3)
C(15)	0·6518 (6)	−0·079 (1)	0·2383 (3)	0·132 (5)
C(16)	0·6216 (4)	0·116 (1)	0·2505 (2)	0·067 (4)
C(17)	0·9338 (4)	0·054 (1)	0·1767 (3)	0·074 (3)

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

O(1)–C(2)	1·419 (7)	C(1)–C(11)	1·507 (8)
O(1)–C(17)	1·418 (8)	C(2)–C(3)	1·510 (9)
O(2)–C(10)	1·438 (6)	C(3)–C(4)	1·524 (9)
O(2)–C(11)	1·443 (7)	C(3)–C(12)	1·548 (9)
O(3)–C(7)	1·422 (8)	C(4)–C(5)	1·512 (8)
O(3)–C(15)	1·35 (1)	C(5)–C(6)	1·322 (9)
O(4)–C(7)	1·414 (7)	C(6)–C(7)	1·512 (8)
O(4)–C(16)	1·419 (7)	C(7)–C(8)	1·543 (8)
O(5)–C(9)	1·206 (8)	C(8)–C(9)	1·502 (9)
O(6)–C(13)	1·171 (7)	C(9)–C(10)	1·521 (8)
O(7)–C(13)	1·338 (7)	C(10)–C(11)	1·478 (7)
O(7)–C(14)	1·461 (7)	C(10)–C(13)	1·524 (8)
C(1)–C(2)	1·523 (7)	C(11)–C(12)	1·507 (9)
C(1)–C(6)	1·513 (8)	C(15)–C(16)	1·47 (1)

C(2)–O(1)–C(17)	112·0 (5)	C(6)–C(7)–C(8)	110·9 (5)
C(10)–O(2)–C(11)	61·7 (3)	C(7)–C(8)–C(9)	114·7 (4)
C(7)–O(3)–C(15)	107·5 (5)	O(5)–C(9)–C(8)	123·8 (6)
C(7)–O(4)–C(16)	107·0 (4)	O(5)–C(9)–C(10)	119·9 (6)
C(13)–O(7)–C(14)	115·4 (4)	C(8)–C(9)–C(10)	116·3 (6)
C(2)–C(1)–C(6)	110·7 (5)	O(2)–C(10)–C(9)	115·7 (5)
C(2)–C(1)–C(11)	101·5 (4)	O(2)–C(10)–C(11)	59·3 (3)
C(6)–C(1)–C(11)	105·7 (4)	O(2)–C(10)–C(13)	113·7 (5)
O(1)–C(2)–C(1)	114·6 (5)	C(9)–C(10)–C(11)	117·0 (5)
O(1)–C(2)–C(3)	111·4 (5)	C(9)–C(10)–C(13)	118·5 (4)
C(1)–C(2)–C(3)	101·1 (4)	C(11)–C(10)–C(13)	118·6 (5)
C(2)–C(3)–C(4)	109·7 (5)	O(2)–C(11)–C(1)	118·0 (5)
C(2)–C(3)–C(12)	101·6 (5)	O(2)–C(11)–C(10)	58·9 (3)
C(4)–C(3)–C(12)	111·5 (5)	O(2)–C(11)–C(12)	119·8 (5)
C(3)–C(4)–C(5)	111·8 (5)	C(1)–C(11)–C(10)	118·6 (5)
C(4)–C(5)–C(6)	122·8 (5)	C(1)–C(11)–C(12)	106·9 (5)
C(1)–C(6)–C(5)	119·4 (5)	C(10)–C(11)–C(12)	127·7 (5)
C(1)–C(6)–C(7)	116·5 (5)	C(3)–C(12)–C(11)	104·7 (5)
C(5)–C(6)–C(7)	123·6 (5)	O(6)–C(13)–O(7)	127·0 (6)
O(3)–C(7)–O(4)	107·9 (5)	O(6)–C(13)–C(10)	124·8 (5)
O(3)–C(7)–C(6)	109·4 (5)	O(7)–C(13)–C(10)	108·2 (5)
O(3)–C(7)–C(8)	109·9 (5)	O(3)–C(15)–C(16)	109·1 (6)
O(4)–C(7)–C(6)	112·1 (5)	O(4)–C(16)–C(15)	102·9 (6)
O(4)–C(7)–C(8)	106·6 (4)		

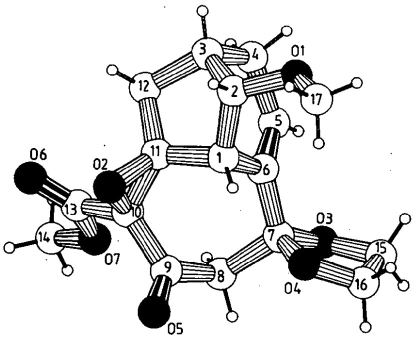

Fig. 1. View of the molecule with adopted numbering.

reflecting with structured reflection profiles. Lattice parameters and their e.s.d.'s were derived from the setting angles of 20 reflections ($12 < \theta < 18°$). The space group was determined from the observed systematic absences as *Cc* or *C*2/*c*. The choice *C*2/*c* was confirmed by a satisfactory structure determination in that space group. 3491 reflections [$\theta < 25°$; $\omega/2\theta$ scan; $\Delta\omega = 0{\cdot}70 + 0{\cdot}35\tan(\theta)°$; $-17 \leq h \leq 17$, $0 \leq k \leq 8$, $0 \leq l \leq 36$] were scanned. Reflections were measured following the *A*-vector procedure (Duisenberg, 1983) at ψ values that minimize the observed reflection splitting. Two reference reflections ($\bar{4}0\bar{8}$, $\bar{5}\bar{1}5$) showed a small decay of 1% during the 58 h of X-ray exposure time. The intensities were corrected for Lp and the small decay but not for absorption. Variance $\sigma^2(I)$ calculated based on counting statistics plus a term $(PI)^2$, where P ($= 0{\cdot}017$) is the instability constant as derived from the excess variance in the reference reflections (McCandlish, Stout & Andrews, 1975). The 1169 reflections with $I \geq 1{\cdot}5\sigma(I)$ were used in the subsequent analysis. The structure was solved with *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). Subsequent refinement was carried out by full-matrix least-squares techniques on *F* with *SHELX*76 (Sheldrick, 1976) on a microVAX-II. All non-H atoms were refined with anisotropic thermal parameters. Most H atoms could be located from a difference Fourier map but, in view of the limited data/parameter ratio, all were included at calculated positions and refined with fixed geometry with respect to their carrier atom [d(C—H) = 0·95 Å] and with isotropic thermal parameters. Convergence was reached at $R = 0{\cdot}059$ [$wR = 0{\cdot}036$, $w^{-1} = \sigma^2(F) + 0{\cdot}0001F^2$, 1169 reflections, 235 parameters, $S = 2{\cdot}38$; $(\Delta/\sigma)_{\rm max} = 0{\cdot}07$]. A final difference Fourier map did not show features outside the range of 0·31 to −0·29 e Å^{-3}. C(15) in the dioxolane moiety is probably slightly disordered as indicated by its relatively high anisotropic thermal parameters with the largest main axis perpendicular to the ring plane and short distances to the neighbouring C and O atoms. It was considered not worthwhile to investigate the exact nature of this disorder. Fig. 1 shows the molecule with adopted numbering. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.* Data on the geometry are assembled in Table 2. Scattering factors of Cromer & Mann (1968) were used. Geometrical calculations and illustrations were made with the programs *PLATON* and *PLUTON* of the *EUCLID* package (Spek, 1982).

Related literature. The title compound was isolated as an intermediate in the total synthesis of C_{19}-diterpene alkaloids (van Beek, van der Baan, Klumpp & Bickelhaupt, 1986). The related structure of methyl 3,4-epoxy-5,5-ethylenedioxy-*exo*-7,*endo*-11-dihydroxytricyclo[7.2.1.$0^{4,10}$]dodecane-8-carboxylate has been reported previously (Spek & van Eijck, 1987).

Crystals were kindly provided by Dr van der Baan and Professor Bickelhaupt. The investigations were supported in part by the Netherlands Foundation for Chemical Research (SON) with financial aid from the Netherlands Organization for Advancement of Pure Research (ZWO).

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* Lists of anisotropic thermal parameters, H-atom parameters and observed and calculated structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44809 (17 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Acta Cryst. (1988). C**44**, 1141–1143

Methyl 2-[2-(2-Hydroxyphenyl)ethyl]benzoate

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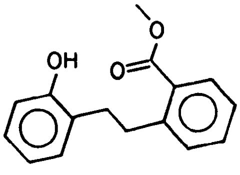
(*Received* 23 *December* 1987; *accepted* 26 *January* 1988)

Abstract. $C_{16}H_{16}O_3$, $M_r = 256{\cdot}3$, monoclinic, $P2_1/n$, $a = 12{\cdot}308\,(3)$, $b = 7{\cdot}300\,(1)$, $c = 16{\cdot}034\,(2)$ Å, $\beta = 110{\cdot}17\,(2)°$, $V = 1352{\cdot}2\,(9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}259$ g cm^{-3}, $\lambda(\text{Mo }K\alpha) = 0{\cdot}71073$ Å, $\mu = 0{\cdot}8$ cm^{-1}, $F(000) = 544$, $T = 295\,(1)$ K, $R = 0{\cdot}033$ for 1605 reflections with $I > 3{\cdot}0\sigma(I)$ (2368 unique observations). The molecule is a substituted dibenzyl [staggered 1,2-diphenylethane, $\omega(\text{C—C}) = 176{\cdot}1\,(1)°$, with substituents (hydroxyl and methoxycarbonyl) in the 2- and 2′-ring positions. The rings are nearly parallel [dihedral angle $6{\cdot}0\,(4)°$] and the methoxycarbonyl group is twisted $18{\cdot}7\,(1)°$ out of the plane of its attached phenyl ring in order to form a weak intramolecular hydrogen bond: $\text{O}\cdots\text{O} = 2{\cdot}788\,(2)$ Å, $\text{O—H}\cdots\text{O} = 159\,(1)°$, $\text{H}\cdots\text{O} = 1{\cdot}91\,(2)$ Å.

Experimental. The compound (I) was obtained by refluxing 2-[2-(2-hydroxyphenyl)ethyl]benzoic acid in acidic methanol and recrystallizing from hexane. A colorless prismatic crystal was mounted with epoxy on a glass fiber in random orientation. Details of data collection and structural refinement are given in Table 1.



(I)

The structure was solved using *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980). All 19 non-H atoms were located from an *E* map. H atoms were located by difference Fourier synthesis and their positions and isotropic thermal parameters were refined. All non-H atoms were treated anisotropically. The structural model was refined by full-matrix least squares with Enraf–Nonius *SDP* (Frenz, 1978), in which the function minimized was

0108-2701/88/061141-03$03.00