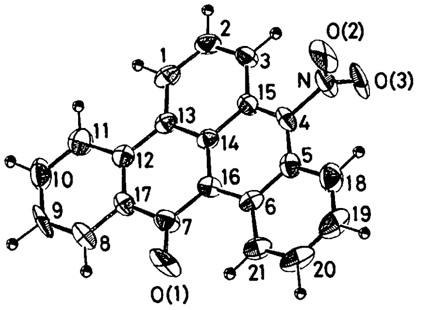

Fig. 1. Perspective drawing of the molecule. 50% probability ellipsoids are shown.

1831 with $|F_o| > 3\sigma(|F_o|)$ considered observed and used for structure determination; corrections for Lorentz and polarization, absorption ignored; direct methods (*MULTAN*78; Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978) and subsequent difference calculation; block-diagonal least squares (*HBLS*; Ohashi, 1975) with anisotropic thermal parameters for all non-H atoms; H atoms derived geometrically (C—H 1·08 Å) and refined isotropically; $\sum w(|F_o| - |F_c|)^2$ minimized with $w = [\sigma^2(F_o) + (0{\cdot}03F)^2]^{-1}$; max. (Δ/σ) 0·15; final R = 0·059 and wR = 0·066; S = 5·6; $\Delta\rho$ excursions in final difference map 0·3 e Å^{-3}; atomic scattering factors from *International Tables for X-ray Crystallography* (1974); calculations carried out on the HITAC M-680 computer at the Computer Center of the University of Tokyo. The final atomic parameters for non-H atoms are in Table 1.* A perspective drawing of the molecule with the numbering scheme is shown in Fig. 1. The bond lengths and angles are listed in Table 2.

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* Lists of structure factors, anisotropic thermal parameters for non-H atoms, positional and thermal parameters for H atoms and equations of mean planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44014 (9 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of Methyl 3,4-Epoxy-5,5-ethylenedioxy-*exo*-7,*endo*-11-dihydroxytricyclo[7.2.1.0^{4,10}]dodecane-8-carboxylate

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Abstract. $C_{16}H_{22}O_7$, M_r = 326·35, monoclinic, $P2_1/n$, a = 6·520 (1), b = 15·119 (4), c = 15·584 (5) Å, β = 96·82 (2)°, U = 1525·3 (7) Å^3, Z = 4, D_x = 1·421 g cm^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, μ = 1·0 cm^{-1}, $F(000)$ = 696, T = 295 K, R = 0·044 for 2064 reflections with $I \geq 2{\cdot}5\sigma(I)$. The structure contains one intramolecular and one intermolecular H bond. Hydrogen bonding links the molecules into infinite chains that run in the **b** direction.

Experimental. X-ray data for a plate-shaped transparent colourless crystal (0·075 × 0·50 × 0·50 mm), glued on top of a glass fibre, were collected on an Enraf–Nonius CAD-4*F* diffractometer using Zr-filtered Mo $K\alpha$ radiation. Lattice parameters and their estimated standard deviations were derived from the setting angles of 20 reflections (10 < θ < 12°). The space group was determined from the observed systematic absences. A total of 3745 unique reflections [θ < 27·5°; $\omega/2\theta$ scan; $\Delta\omega$ = 0·70 + 0·35tan(θ)°; $-8 \leq h \leq 8$, $0 \leq k \leq 19$, $0 \leq l \leq 20$] were scanned. Two reference reflections ($\bar{2}08$, $1\bar{6}\bar{2}$) showed no decay during the 67 h of X-ray exposure time. The intensities were corrected for Lp but not for absorption. Variance $\sigma^2(I)$ calculated based on counting statistics plus a term $(PI)^2$, where P (=0·012) is the instability constant as

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derived from the excess variance in the reference reflections (McCandlish, Stout & Andrews, 1975). $\sum\sigma/\sum I = 0{\cdot}02$. The 2065 reflections with $I \geq 2{\cdot}5\sigma(I)$ 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 blocked full-matrix least-squares techniques on F with *SHELX*76 (Sheldrick, 1976). All non-hydrogen atoms were refined with anisotropic thermal parameters. H atoms were located from a difference Fourier map and their positional and individual isotropic thermal parameters refined, except for the two H atoms bonded to C(16) that were introduced on calculated positions and refined with fixed geometry with respect to their carrier atom ($U = 0{\cdot}20$ Å^2). Convergence was reached at $R = 0{\cdot}044$ [$wR = 0{\cdot}036$, $w^{-1} = \sigma^2(F)$, 2064 reflections, 235 parameters, $S = 2{\cdot}64$; $(\Delta/\sigma)_{\rm av} = 0{\cdot}05$]. A final difference Fourier map did not show features outside the range of 0·25 and $-0{\cdot}24$ e Å^{-3}. One extinction-affected reflection ($\bar{1}12$) was left out of the final refinement cycles. C(16) 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

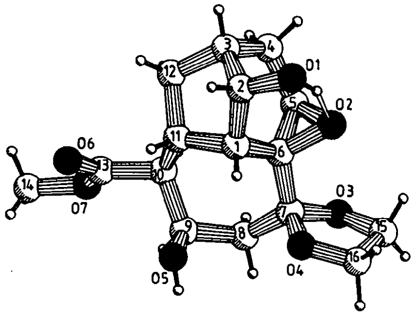

Fig. 1. View of the molecule with adopted numbering.

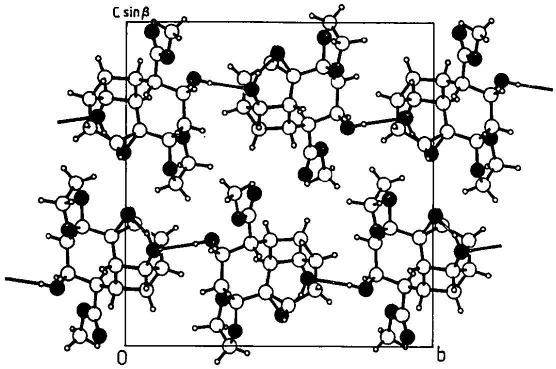

Fig. 2. Projection of the structure down **a** illustrating the hydrogen bonding that links the molecules into infinite chains running in the **b** direction.

Table 1. *Final coordinates and equivalent isotropic thermal parameters of the non-*H *atoms with 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)	0·4576 (2)	0·0918 (1)	0·2928 (1)	0·0547 (6)
O(2)	0·2563 (2)	0·0078 (1)	0·4039 (1)	0·0440 (5)
O(3)	0·0948 (2)	−0·1431 (1)	0·4540 (1)	0·0434 (5)
O(4)	0·3075 (2)	−0·1843 (1)	0·3551 (1)	0·0487 (5)
O(5)	0·0474 (2)	−0·2196 (1)	0·1755 (1)	0·0528 (5)
O(6)	−0·2453 (2)	−0·0827 (1)	0·0299 (1)	0·0677 (6)
O(7)	−0·4830 (2)	−0·1330 (1)	0·1083 (1)	0·0534 (5)
C(1)	0·2033 (3)	−0·0304 (1)	0·2457 (1)	0·0328 (6)
C(2)	0·2815 (3)	0·0648 (1)	0·2348 (1)	0·0403 (6)
C(3)	0·0849 (3)	0·1182 (1)	0·2382 (1)	0·0410 (6)
C(4)	0·0098 (3)	0·1160 (1)	0·3273 (1)	0·0467 (6)
C(5)	0·0411 (3)	0·0292 (1)	0·3745 (1)	0·0402 (6)
C(6)	0·1429 (3)	−0·0444 (1)	0·3352 (1)	0·0313 (6)
C(7)	0·1184 (3)	−0·1397 (1)	0·3645 (1)	0·0372 (6)
C(8)	−0·0647 (3)	−0·1887 (1)	0·3185 (1)	0·0455 (6)
C(9)	−0·1124 (3)	−0·1851 (1)	0·2204 (1)	0·0395 (6)
C(10)	−0·1657 (3)	−0·0922 (1)	0·1856 (1)	0·0322 (6)
C(11)	0·0146 (3)	−0·0301 (1)	0·1754 (1)	0·0347 (6)
C(12)	−0·0589 (3)	0·0683 (1)	0·1705 (1)	0·0460 (6)
C(13)	−0·2970 (3)	−0·1004 (1)	0·0981 (1)	0·0392 (6)
C(14)	−0·6241 (4)	−0·1468 (2)	0·0300 (2)	0·063 (1)
C(15)	0·2940 (4)	−0·1614 (2)	0·5003 (1)	0·057 (1)
C(16)	0·4113 (5)	−0·2027 (3)	0·4364 (2)	0·102 (2)

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

O(1)—C(2)	1·434 (2)	C(2)—C(3)	1·521 (3)
O(2)—C(5)	1·460 (2)	C(3)—C(4)	1·527 (2)
O(2)—C(6)	1·459 (2)	C(3)—C(12)	1·525 (2)
O(3)—C(7)	1·422 (2)	C(4)—C(5)	1·506 (2)
O(3)—C(15)	1·436 (3)	C(5)—C(6)	1·466 (2)
O(4)—C(7)	1·428 (2)	C(6)—C(7)	1·526 (2)
O(4)—C(16)	1·392 (4)	C(7)—C(8)	1·512 (2)
O(5)—C(9)	1·421 (2)	C(8)—C(9)	1·524 (2)
O(6)—C(13)	1·183 (2)	C(9)—C(10)	1·531 (2)
O(7)—C(13)	1·336 (2)	C(10)—C(11)	1·527 (3)
O(7)—C(14)	1·453 (3)	C(10)—C(13)	1·527 (2)
C(1)—C(2)	1·543 (2)	C(11)—C(12)	1·562 (2)
C(1)—C(6)	1·509 (2)	C(15)—C(16)	1·465 (4)
C(1)—C(11)	1·547 (3)		

C(5)—O(2)—C(6)	60·3 (1)	O(3)—C(7)—C(6)	110·8 (1)
C(7)—O(3)—C(15)	107·6 (1)	O(4)—C(7)—C(6)	107·0 (2)
C(7)—O(4)—C(16)	109·5 (2)	C(6)—C(7)—C(8)	115·2 (1)
C(13)—O(7)—C(14)	116·5 (2)	O(3)—C(7)—O(4)	106·2 (1)
C(2)—C(1)—C(6)	111·0 (1)	O(3)—C(7)—C(8)	106·0 (1)
C(2)—C(1)—C(11)	99·5 (1)	O(4)—C(7)—C(8)	111·3 (1)
C(6)—C(1)—C(11)	112·5 (2)	C(7)—C(8)—C(9)	120·9 (1)
O(1)—C(2)—C(1)	116·5 (1)	O(5)—C(9)—C(8)	114·0 (2)
O(1)—C(2)—C(3)	116·2 (1)	C(8)—C(9)—C(10)	113·5 (1)
C(1)—C(2)—C(3)	101·6 (1)	O(5)—C(9)—C(10)	108·1 (1)
C(2)—C(3)—C(4)	112·5 (1)	C(9)—C(10)—C(11)	117·1 (2)
C(2)—C(3)—C(12)	99·5 (1)	C(9)—C(10)—C(13)	108·8 (1)
C(4)—C(3)—C(12)	112·4 (2)	C(11)—C(10)—C(13)	108·3 (1)
C(3)—C(4)—C(5)	115·1 (1)	C(1)—C(11)—C(10)	118·8 (1)
C(4)—C(5)—C(6)	119·9 (1)	C(1)—C(11)—C(12)	104·5 (1)
O(2)—C(5)—C(4)	114·6 (2)	C(10)—C(11)—C(12)	110·8 (2)
O(2)—C(5)—C(6)	59·8 (1)	C(3)—C(12)—C(11)	106·1 (1)
O(2)—C(6)—C(1)	115·9 (1)	O(6)—C(13)—C(10)	126·5 (2)
O(2)—C(6)—C(5)	59·9 (1)	O(7)—C(13)—C(10)	110·1 (1)
O(2)—C(6)—C(7)	110·7 (1)	O(6)—C(13)—O(7)	123·4 (2)
C(1)—C(6)—C(5)	117·4 (1)	O(3)—C(15)—C(16)	104·7 (2)
C(1)—C(6)—C(7)	117·2 (1)	O(4)—C(16)—C(15)	107·3 (3)
C(5)—C(6)—C(7)	121·5 (1)		

D—H···A	D···A	D—H	H···A	∠D—H···A
O(1)—H(101)···O(2)	2·622 (2)	0·89 (3)	1·82 (3)	149 (3)
O(5)—H(105)···O(1$^{\rm i}$)	2·895 (2)	0·82 (3)	2·08 (3)	172 (2)

Symmetry code (i): $\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z$.

worthwhile to investigate the exact nature of this disorder. Fig. 1 shows the molecule with adopted numbering. The packing and hydrogen bonding are illustrated in Fig. 2. 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 were performed and illustrations drawn 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).

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 structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43963 (26 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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3,8,11,16-Tetrakis(trifluoromethyl)-1,2,4,7,9,10,12,15-octaazacyclohexadeca-2,8,10,16(1)-tetraene

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Abstract. $C_{12}H_{12}F_{12}N_8$, $M_r = 496{\cdot}3$, orthorhombic, $P2_12_12_1$, $a = 8{\cdot}572\,(2)$, $b = 9{\cdot}509\,(3)$, $c = 23{\cdot}502\,(5)$ Å, $V = 1915{\cdot}7$ Å^3, $Z = 4$, $D_x = 1{\cdot}72$ Mg m^{-3}, $\lambda(\mathrm{Mo}\,K\alpha) = 0{\cdot}71069$ Å, $\mu = 0{\cdot}141$ mm^{-1}, $F(000) = 992$, $T = 293$ K, $R = 0{\cdot}051$ for 818 unique reflexions $[F > 3\sigma(F)]$. The ring is composed of two distorted *anti* 1,3,4,6-tetraaza-2,4-hexadiene moieties linked by two ethylene bridges, one *anti* and the other *gauche*, so that an approximate twofold axis passes through the C—C bonds.

Experimental. The title compound was obtained on mixing 2,5-dichloro-1,1,1,6,6,6-hexafluoro-3,4-diaza-2,4-hexadiene with 1,2-ethanediamine in molar ratio 1:2 in diethyl ether at 273 K for 30 min followed by recrystallization from 50:50 petroleum ether (b.p. 313–333 K) and chloroform. Crystals suitable for X-ray work were obtained from aqueous ethanol (10:90 H_2O: EtOH).

Crystal size $0{\cdot}4 \times 0{\cdot}4 \times 0{\cdot}2$ mm; Enraf–Nonius CAD-4 diffractometer, graphite-monochromatized Mo $K\alpha$ radiation; unit-cell dimensions from setting angles of 25 accurately centred reflexions ($5{\cdot}0 < \theta < 15{\cdot}8°$); ω–2θ scan mode used to measure 1194 reflexions with $I > 0$, ω-scan width of $(1{\cdot}00 + 0{\cdot}35\tan\theta)°$ and scan speed ranging from 0·3 to 5° min^{-1} according to the intensity gathered in a pre-scan; $0 \le h \le 9$, $0 \le k \le 10$, $0 \le l \le 26$; $0 < \theta < 25°$; 818 unique structure amplitudes with $F > 3\sigma(F)$; negligible change in intensity standards ($\bar{1}\bar{2}\bar{3}$; $\bar{1}\bar{2}0$; $\bar{1}\bar{2}4$) measured every 2 h; Lorentz and polarization corrections but absorption ignored; structure solution *via* *MULTAN*80 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1980), all non-H atoms found in a Fourier map, H atoms constrained to chemically reasonable positions except for those attached to N atoms, which were located from a difference Fourier map; full-matrix least squares based on F using