Crystal structure of tetramethyl (1α,2β,3α,5α,6β,7α,8β,9α,11α,12β)-4,10-dioxahexacyclo[5.5.1.$0^{2,6}$.$0^{3,5}$. $0^{8,12}$.$0^{9,11}$]tridecane-3,5,9,11-tetracarboxylate, $C_{19}H_{20}O_{10}$

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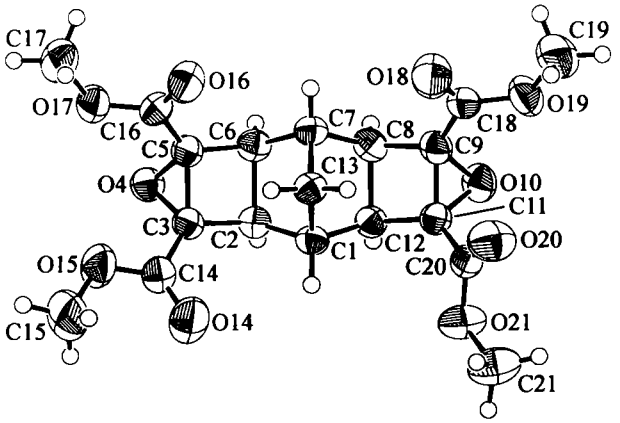

Abstract

$C_{19}H_{20}O_{10}$, monoclinic, $P12_1/c1$ (No. 14), a = 7.309(2) Å, b = 12.471(3) Å, c = 20.930(7) Å, β = 95.96(2)°, V = 1897.5 Å^3, Z = 4, $R_{gt}(F)$ = 0.076, $wR_{ref}(F^2)$ = 0.226, T = 293 K.

Source of material

The compound was prepared as described in the literature [1]. Colourless crystals were obtained from the slow evaporation of a dichloromethane/hexane solution of the compound; mp 469 K – 471 K.

Experimental details

The C-bound H atoms were placed in their geometrically calculated positions and included in the final refinement in the riding model approximation. The crystal chosen for the analysis was weakly diffracting and the structure determination is less than optimal. Nevertheless, the molecular connectivity has been established unambiguously.

Discussion

The molecular structure of the title compound was determined as part of a synthetic program designed to generate functionalised cavity structures [1-3]. The X-ray analysis shows an absence of crystallographically imposed symmetry. However, approximate mirror symmetry is found (the C1, C7 and C13 atoms define a pseudo mirror plane) with the major deviation been found in the orientation of the C14 and C20 ester groups. The four carbonyl groups lie to one side of the molecule and the two epoxides to the other.

Table 1. Data collection and handling.

Crystal:	colourless needle, size 0.05 × 0.16 × 0.39 mm
Wavelength:	Mo K_α radiation (0.7107 Å)
μ:	1.17 cm^{-1}
Diffractometer, scan mode:	Rigaku AFC6R, $\omega/2\theta$
$2\theta_{max}$:	55.2°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4943, 4388
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 1536
$N(param)_{refined}$:	263
Programs:	SIR92 [4], teXsan [5], SHELXL-97 [6], DIBABS [7]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(1)	4e	0.3881	0.7865	–0.2271	0.091(4)
H(2)	4e	0.7318	0.7391	–0.2370	0.091
H(6)	4e	0.7614	0.5465	–0.2410	0.091
H(7)	4e	0.4427	0.4610	–0.2376	0.091
H(8)	4e	0.5603	0.5477	–0.3390	0.091
H(12)	4e	0.5198	0.7404	–0.3330	0.091
H(13a)	4e	0.2156	0.6122	–0.2332	0.091
H(13b)	4e	0.3511	0.6140	–0.1686	0.091
H(15a)	4e	0.7229	0.8000	0.0555	0.109(5)
H(15b)	4e	0.5273	0.8129	0.0179	0.109
H(15c)	4e	0.6858	0.8922	0.0049	0.109
H(17a)	4e	0.8690	0.4037	0.0342	0.109
H(17b)	4e	0.7657	0.3323	–0.0196	0.109
H(17c)	4e	0.6552	0.4135	0.0187	0.109
H(19a)	4e	–0.1668	0.3912	–0.4641	0.109
H(19b)	4e	–0.2010	0.4107	–0.3923	0.109
H(19c)	4e	–0.0622	0.3220	–0.4094	0.109
H(21a)	4e	–0.0622	0.9791	–0.3453	0.109
H(21b)	4e	–0.1540	0.8854	–0.3102	0.109
H(21c)	4e	–0.1667	0.8876	–0.3855	0.109

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Table 3. Atomic coordinates and displacement parameters (in $Å^2$).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
O(4)	4*e*	0.8679(4)	0.6405(2)	–0.1307(2)	0.044(2)	0.051(2)	0.061(2)	–0.004(2)	0.003(2)	–0.002(2)
O(10)	4*e*	0.2727(4)	0.6347(3)	–0.4087(2)	0.071(2)	0.068(2)	0.041(2)	–0.001(2)	0.011(2)	0.001(2)
O(14)	4*e*	0.5241(6)	0.8370(4)	–0.1057(2)	0.104(3)	0.120(4)	0.073(3)	0.061(3)	–0.003(2)	–0.022(3)
O(15)	4*e*	0.7106(5)	0.7486(3)	–0.0349(2)	0.104(3)	0.099(3)	0.051(2)	0.036(3)	–0.007(2)	–0.010(2)
O(16)	4*e*	0.5299(5)	0.4354(3)	–0.1030(2)	0.057(2)	0.071(2)	0.076(3)	–0.016(2)	–0.005(2)	0.019(2)
O(17)	4*e*	0.7947(4)	0.4842(3)	–0.0467(2)	0.072(2)	0.062(2)	0.052(2)	–0.006(2)	–0.005(2)	0.006(2)
O(18)	4*e*	0.1016(5)	0.4244(3)	–0.3115(2)	0.095(3)	0.077(3)	0.086(3)	–0.032(2)	–0.012(2)	0.022(3)
O(19)	4*e*	0.0368(5)	0.4696(3)	–0.4142(2)	0.070(2)	0.072(3)	0.067(2)	–0.018(2)	–0.008(2)	–0.011(2)
O(20)	4*e*	–0.0814(5)	0.6927(3)	–0.3449(2)	0.064(3)	0.074(3)	0.127(4)	–0.009(2)	0.012(2)	–0.001(3)
O(21)	4*e*	0.0799(4)	0.8425(3)	–0.3445(2)	0.053(2)	0.052(2)	0.109(3)	0.002(2)	0.027(2)	0.005(2)
C(1)	4*e*	0.4395(6)	0.7177(4)	–0.2390(2)	0.049(3)	0.040(3)	0.046(3)	0.002(2)	0.008(2)	–0.003(2)
C(2)	4*e*	0.6435(6)	0.6996(4)	–0.2138(2)	0.044(2)	0.047(3)	0.044(3)	–0.001(2)	0.009(2)	0.000(2)
C(3)	4*e*	0.6926(6)	0.6936(4)	–0.1417(2)	0.038(3)	0.046(3)	0.049(3)	0.000(2)	0.004(2)	–0.003(2)
C(5)	4*e*	0.7080(6)	0.5733(4)	–0.1440(2)	0.039(3)	0.047(3)	0.051(3)	0.001(2)	0.002(2)	–0.002(3)
C(6)	4*e*	0.6615(6)	0.5720(4)	–0.2171(2)	0.046(3)	0.047(3)	0.053(3)	0.001(2)	0.008(2)	–0.002(2)
C(7)	4*e*	0.4711(6)	0.5366(4)	–0.2447(2)	0.045(3)	0.041(3)	0.051(3)	0.002(2)	0.007(2)	–0.002(2)
C(8)	4*e*	0.4579(6)	0.5711(4)	–0.3156(2)	0.048(3)	0.051(3)	0.049(3)	–0.004(2)	0.010(2)	–0.006(3)
C(9)	4*e*	0.2679(7)	0.5654(4)	–0.3538(2)	0.058(3)	0.052(3)	0.037(3)	–0.001(3)	0.009(2)	–0.003(2)
C(11)	4*e*	0.2433(6)	0.6827(4)	–0.3481(2)	0.056(3)	0.046(3)	0.040(3)	0.001(2)	0.005(2)	0.003(2)
C(12)	4*e*	0.4329(6)	0.6978(4)	–0.3109(2)	0.046(3)	0.045(3)	0.045(3)	–0.002(2)	0.005(2)	0.000(2)
C(13)	4*e*	0.3429(6)	0.6181(3)	–0.2151(2)	0.044(2)	0.044(3)	0.051(3)	–0.003(2)	0.007(2)	0.004(2)
C(14)	4*e*	0.6316(7)	0.7669(4)	–0.0921(3)	0.045(3)	0.062(3)	0.053(3)	0.003(3)	0.008(2)	–0.002(3)
C(15)	4*e*	0.657(1)	0.8194(5)	0.0151(3)	0.143(6)	0.133(6)	0.062(4)	0.034(5)	0.002(4)	–0.032(4)
C(16)	4*e*	0.6643(6)	0.4907(4)	–0.0959(2)	0.045(3)	0.050(3)	0.054(3)	0.006(2)	0.004(2)	0.005(3)
C(17)	4*e*	0.7690(9)	0.4013(4)	0.0007(3)	0.101(5)	0.079(4)	0.060(3)	–0.009(4)	0.000(3)	0.021(3)
C(18)	4*e*	0.1268(7)	0.4791(4)	–0.3566(3)	0.054(3)	0.046(3)	0.060(4)	0.004(3)	0.000(3)	–0.005(3)
C(19)	4*e*	–0.1107(8)	0.3918(5)	–0.4205(3)	0.073(4)	0.085(5)	0.113(5)	–0.018(4)	–0.011(4)	–0.015(4)
C(20)	4*e*	0.0628(7)	0.7392(4)	–0.3460(2)	0.055(3)	0.048(3)	0.056(3)	–0.006(3)	–0.003(2)	0.004(3)
C(21)	4*e*	–0.0903(7)	0.9039(4)	–0.3466(3)	0.062(4)	0.067(4)	0.127(6)	0.015(3)	0.040(4)	0.007(4)

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