Crystal structure of methyl (1*R*,2*S*,3*R*,4*S*)-1-phenyl-3,4-epoxi-1,2,3,4-tetrahydronaphthalene-2-carboxylate, $C_{18}H_{16}O_3$

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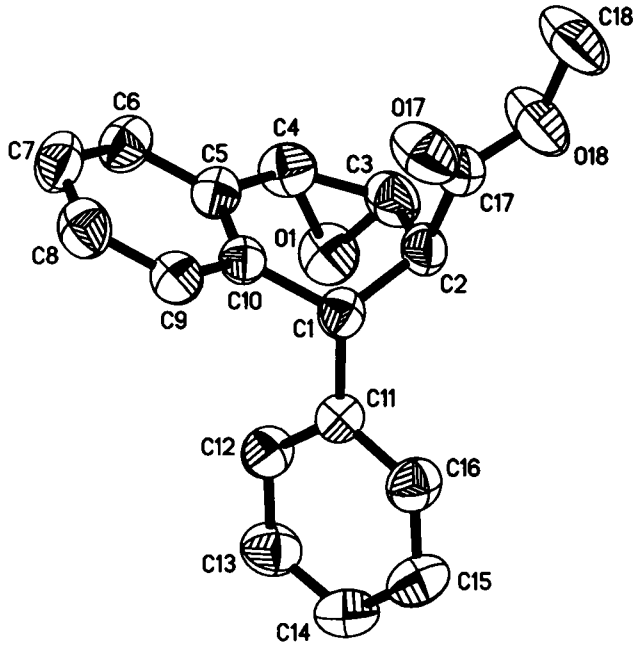

Abstract

$C_{18}H_{16}O_3$, monoclinic, $P12_1/c1$ (no. 14), a = 14.954(3) Å, b = 8.088(2) Å, c = 11.973(2) Å, β = 93.50(1)°, V = 1445.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.083, $wR_{ref}(F^2)$ = 0.249, T = 293 K.

Source of material

The title compound was prepared by kinetic resolution by Jacobsen epoxidation of the corresponding alkene [1].

Experimental details

The crystals could not be cleaved without deterioration of their quality. Therefore, they were used as grown, irrespective of their unusual size.

Discussion

In the molecule the atoms C2, C3, C4 and C5 nearly form a plane, the corresponding torsion angle is of −1.5°. The dihedral angle between this plane and the three-membered ring is of 73.6°.

Table 1. Data collection and handling.

Crystal:	colorless lath, size 0.25 × 0.45 × 1.8 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	0.87 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3142, 3014
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2204
$N(param)_{refined}$:	191
Programs:	SHELXS-97 [2], SHELXL-97 [3]

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

Atom	Site	x	y	z	U_{iso}
H(1)	4*e*	0.2310	−0.0419	0.7674	0.08
H(2)	4*e*	0.2982	−0.3007	0.7119	0.08
H(3)	4*e*	0.3998	−0.2778	0.5618	0.08
H(4)	4*e*	0.3792	−0.0605	0.4342	0.08
H(6)	4*e*	0.3270	0.2224	0.3984	0.08
H(7)	4*e*	0.2606	0.4617	0.4573	0.08
H(8)	4*e*	0.1819	0.4609	0.6159	0.08
H(9)	4*e*	0.1681	0.2205	0.7185	0.08
H(12)	4*e*	0.1095	−0.0921	0.5119	0.08
H(13)	4*e*	−0.0198	−0.2473	0.4845	0.08
H(14)	4*e*	−0.0629	−0.4248	0.6225	0.08
H(15)	4*e*	0.0219	−0.4415	0.7900	0.08
H(16)	4*e*	0.1492	−0.2857	0.8177	0.08
H(18A)	4*e*	0.5819	−0.2563	0.8335	0.08
H(18B)	4*e*	0.5217	−0.1498	0.9086	0.08
H(18C)	4*e*	0.5594	−0.0726	0.8009	0.08

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(1)	4*e*	0.2794(2)	−0.2228(2)	0.4771(2)	0.070(1)	0.048(1)	0.067(1)	0.0014(9)	0.000(1)	−0.0161(8)
C(1)	4*e*	0.2315(2)	−0.0794(3)	0.6896(2)	0.049(1)	0.043(1)	0.038(1)	−0.0029(9)	−0.0046(9)	0.0002(9)
C(2)	4*e*	0.3141(2)	−0.1909(3)	0.6847(2)	0.048(1)	0.038(1)	0.061(1)	0.0000(9)	−0.009(1)	0.005(1)
C(3)	4*e*	0.3458(2)	−0.2108(3)	0.5683(3)	0.049(1)	0.045(1)	0.082(2)	0.009(1)	0.001(1)	−0.008(1)
C(4)	4*e*	0.3329(2)	−0.0745(3)	0.4879(2)	0.056(2)	0.053(1)	0.057(2)	0.001(1)	0.011(1)	−0.008(1)
C(5)	4*e*	0.2873(2)	0.0770(3)	0.5228(2)	0.051(1)	0.042(1)	0.043(1)	−0.0028(9)	−0.0019(9)	−0.0025(9)
C(6)	4*e*	0.2950(2)	0.2216(3)	0.4626(2)	0.067(2)	0.054(2)	0.047(1)	−0.009(1)	0.000(1)	0.005(1)

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Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	4*e*	0.2555(2)	0.3645(3)	0.4979(3)	0.074(2)	0.042(1)	0.065(2)	−0.008(1)	−0.013(1)	0.011(1)
C(8)	4*e*	0.2085(2)	0.3639(3)	0.5927(3)	0.064(2)	0.038(1)	0.074(2)	0.004(1)	−0.008(1)	−0.006(1)
C(9)	4*e*	0.2002(2)	0.2197(3)	0.6544(2)	0.051(1)	0.044(1)	0.055(1)	0.000(1)	−0.001(1)	−0.007(1)
C(10)	4*e*	0.2398(2)	0.0750(3)	0.6204(2)	0.042(1)	0.038(1)	0.042(1)	−0.0023(8)	−0.0044(9)	−0.0014(9)
C(11)	4*e*	0.1448(2)	−0.1749(3)	0.6670(2)	0.047(1)	0.041(1)	0.039(1)	−0.0016(9)	0.0037(9)	−0.0029(8)
C(12)	4*e*	0.0924(2)	−0.1630(3)	0.5679(2)	0.054(1)	0.051(1)	0.043(1)	−0.006(1)	−0.001(1)	0.000(1)
C(13)	4*e*	0.0148(2)	−0.2560(4)	0.5515(2)	0.053(2)	0.068(2)	0.055(2)	−0.009(1)	−0.005(1)	−0.007(1)
C(14)	4*e*	−0.0114(2)	−0.3613(4)	0.6340(3)	0.050(1)	0.065(2)	0.068(2)	−0.014(1)	0.009(1)	−0.008(1)
C(15)	4*e*	0.0394(2)	−0.3717(4)	0.7336(3)	0.065(2)	0.067(2)	0.059(2)	−0.013(1)	0.017(1)	0.006(1)
C(16)	4*e*	0.1159(2)	−0.2788(3)	0.7497(2)	0.059(2)	0.064(2)	0.041(1)	−0.007(1)	0.003(1)	0.004(1)
C(17)	4*e*	0.3903(2)	−0.1246(3)	0.7616(2)	0.046(1)	0.045(1)	0.068(2)	0.000(1)	0.000(1)	0.005(1)
O(17)	4*e*	0.3907(2)	0.0026(3)	0.8117(2)	0.056(1)	0.074(2)	0.096(2)	0.005(1)	−0.010(1)	−0.024(1)
O(18)	4*e*	0.4584(2)	−0.2261(3)	0.7683(3)	0.057(1)	0.071(1)	0.130(2)	0.017(1)	−0.031(1)	−0.016(1)
C(18)	4*e*	0.5369(2)	−0.1717(6)	0.8332(4)	0.053(2)	0.098(3)	0.130(3)	0.015(2)	−0.032(2)	−0.013(2)

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