Crystal structure of 2,2-dibenzyl-1,3-dimethyloxypropane, $C_{19}H_{24}O_2$

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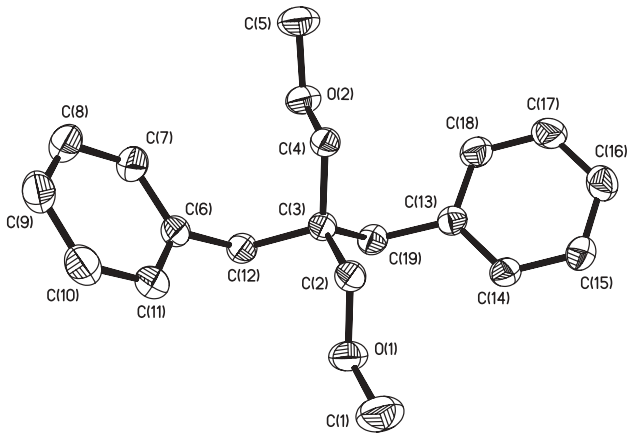
Received March 31, 2003, accepted and available on-line June 7; CCDC-No. 1267/1073



Abstract

$C_{19}H_{24}O_2$, monoclinic, $P12_1/n1$ (No. 14), a = 8.408(2) Å, b = 13.915(3) Å, c = 14.456(3) Å, β = 93.15(3)°, V = 1688.8 Å^3, Z = 4, $R_{gt}(F)$ = 0.042, $wR_{ref}(F^2)$ = 0.111, T = 293 K.

Source of material

The title compound was synthesized similarly to a method described in the literature [1]. Alkylation of $CH_2(COOEt)_2$ by $C_6H_5CH_2Br$ in EtOH containing EtONa gave $(C_6H_5CH_2)_2C(COOEt)_2$, which was reduced by $LiAlH_4$ in Et_2O to give $(C_6H_5CH_2)_2C(CH_2OH)_2$. Methylation of the diol by MeI in THF gave 2,2-dibenzyl-1,3-dimethoxypropane. Colourless prismatic crystals for the X-ray structure determination were obtained by recrystallization from methanol.

Discussion

Electron donors play a fundamental role in modern Ziegler-Natta catalyst systems for the polymerization of propene [2]. A novel and simplified generation of $MgCl_2$-supported catalysts used as internal donors was developed with the discovery of 1,3-diethers [2]. These donors are known for having the property to produce highly active and stereospecific catalysts without any external donors. Herein we report the structure of a diether moleculse of 2,2-dibenzyl-1,3-dimethyloxypropane. In the title compound, the centre C(3) atom which links two methoxymethyl groups and benzyl groups has sp^3 hybrid geometry with C–C–C at 107° – 111°. The atoms C(1), O(1), C(2), C(3), C(4), O(2) and C(5) are coplanar with rms deviation being 0.03 Å. The molecule exhibits essentially butterfly shape with dihedreal angle of 59.28(5)° between wings of benzyl groups. The distance between the O atoms of methoxy groups is 4.789(2) Å, which is longer the one of 3.662(2) Å between the O atoms of β-diol in 2-(hydroxymethyl)-1,3-propanediol [3].

Table 1. Data collection and handling.

Crystal:	colourless prism, size 0.10 × 0.21 × 0.32 mm
Wavelength:	Mo K_α radiation (0.71069 Å)
μ:	0.71 cm^{-1}
Diffractometer, scan mode:	Rigaku R-AXIS RAPID, ω
$2\theta_{max}$:	54.96°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	3870, 3870
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 2098
$N(param)_{refined}$:	190
Programs:	SHELXS-97 [4], SHELXL-97 [5]

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

Atom	Site	x	y	z	U_{iso}
H(1A)	4*e*	–0.3622	0.0509	–0.3324	0.162
H(1B)	4*e*	–0.2946	–0.0224	–0.2578	0.162
H(1C)	4*e*	–0.4229	0.0532	–0.2318	0.162
H(2A)	4*e*	–0.1254	0.0340	–0.1326	0.070
H(2B)	4*e*	–0.2452	0.1155	–0.1078	0.070
H(4A)	4*e*	–0.0584	0.1734	0.0137	0.067
H(4B)	4*e*	0.0583	0.0900	–0.0105	0.067
H(5A)	4*e*	0.2954	0.2569	0.1078	0.144
H(5B)	4*e*	0.1180	0.2366	0.1298	0.144
H(5C)	4*e*	0.2341	0.1508	0.1138	0.144
H(7)	4*e*	–0.0375	0.3849	0.0018	0.094
H(8)	4*e*	–0.2253	0.4466	0.0967	0.111
H(9)	4*e*	–0.4904	0.4141	0.0678	0.111
H(10)	4*e*	–0.5713	0.3233	–0.0590	0.106
H(11)	4*e*	–0.3859	0.2623	–0.1548	0.091
H(12A)	4*e*	0.0271	0.3130	–0.1413	0.076
H(12B)	4*e*	–0.1084	0.2768	–0.2112	0.076
H(14)	4*e*	0.0487	–0.0234	–0.2726	0.078
H(15)	4*e*	0.1498	–0.1756	–0.2505	0.090
H(16)	4*e*	0.3506	–0.2038	–0.1403	0.096
H(17)	4*e*	0.4502	–0.0791	–0.0510	0.106
H(18)	4*e*	0.3519	0.0735	–0.0730	0.094
H(19A)	4*e*	0.0847	0.1506	–0.2512	0.074
H(19B)	4*e*	0.2060	0.1884	–0.1737	0.074

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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(1)	4*e*	–0.2097(1)	0.10728(7)	–0.24063(6)	0.0924(7)	0.0774(6)	0.0600(6)	–0.0116(5)	–0.0164(5)	0.0039(5)
O(2)	4*e*	0.1614(1)	0.21653(6)	–0.00020(6)	0.0800(6)	0.0743(6)	0.0559(5)	–0.0112(5)	–0.0050(4)	–0.0023(4)
C(1)	4*e*	–0.3322(2)	0.0421(1)	–0.2679(1)	0.115(1)	0.105(1)	0.100(1)	–0.029(1)	–0.030(1)	–0.007(1)
C(2)	4*e*	–0.1582(2)	0.09951(9)	–0.14639(8)	0.0694(8)	0.0541(7)	0.0524(7)	0.0022(6)	0.0012(6)	0.0036(6)
C(3)	4*e*	–0.0191(1)	0.16772(8)	–0.12549(8)	0.0650(8)	0.0481(6)	0.0447(6)	0.0014(6)	0.0054(5)	0.0042(5)
C(4)	4*e*	0.0300(2)	0.15638(8)	–0.02346(8)	0.0673(8)	0.0521(7)	0.0490(7)	0.0004(6)	0.0016(6)	0.0023(5)
C(5)	4*e*	0.2059(2)	0.2152(1)	0.0955(1)	0.112(1)	0.107(1)	0.0649(9)	–0.011(1)	–0.0208(9)	–0.0052(9)
C(6)	4*e*	–0.1908(2)	0.31499(9)	–0.08676(8)	0.087(1)	0.0485(7)	0.0531(7)	0.0111(7)	–0.0015(7)	0.0066(6)
C(7)	4*e*	–0.1448(2)	0.3716(1)	–0.0110(1)	0.100(1)	0.0595(8)	0.075(1)	0.0085(8)	–0.0029(8)	–0.0077(7)
C(8)	4*e*	–0.2579(3)	0.4087(1)	0.0461(1)	0.130(2)	0.073(1)	0.073(1)	0.023(1)	0.002(1)	–0.0161(8)
C(9)	4*e*	–0.4157(2)	0.3900(1)	0.0288(1)	0.112(2)	0.085(1)	0.081(1)	0.042(1)	0.009(1)	0.0031(9)
C(10)	4*e*	–0.4636(2)	0.3356(1)	–0.0463(1)	0.086(1)	0.096(1)	0.084(1)	0.0300(9)	–0.0002(9)	0.0063(9)
C(11)	4*e*	–0.3517(2)	0.2988(1)	–0.1037(1)	0.088(1)	0.0754(9)	0.0637(8)	0.0198(8)	–0.0112(8)	0.0007(7)
C(12)	4*e*	–0.0677(2)	0.27324(9)	–0.14721(8)	0.0847(9)	0.0533(7)	0.0508(7)	0.0016(6)	0.0028(6)	0.0064(6)
C(13)	4*e*	0.1878(2)	0.04263(9)	–0.17449(8)	0.0612(8)	0.0619(7)	0.0487(7)	0.0015(6)	0.0113(6)	–0.0012(6)
C(14)	4*e*	0.1299(2)	–0.03381(9)	–0.22741(9)	0.0709(9)	0.0715(9)	0.0528(7)	0.0083(7)	–0.0008(6)	–0.0072(7)
C(15)	4*e*	0.1907(2)	–0.1252(1)	–0.2143(1)	0.083(1)	0.0674(9)	0.075(1)	0.0115(8)	0.0031(8)	–0.0153(7)
C(16)	4*e*	0.3099(2)	–0.1421(1)	–0.1489(1)	0.087(1)	0.078(1)	0.076(1)	0.0266(9)	0.0107(8)	–0.0012(8)
C(17)	4*e*	0.3691(2)	–0.0679(1)	–0.0961(1)	0.080(1)	0.106(1)	0.077(1)	0.0306(9)	–0.0150(8)	–0.0128(9)
C(18)	4*e*	0.3093(2)	0.0235(1)	–0.1091(1)	0.0701(9)	0.085(1)	0.078(1)	0.0102(8)	–0.0098(7)	–0.0192(8)
C(19)	4*e*	0.1207(2)	0.14254(9)	–0.18681(8)	0.0739(9)	0.0593(7)	0.0519(7)	–0.0033(6)	0.0115(6)	0.0025(6)

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