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2,2′-Dimethoxybibenzyl

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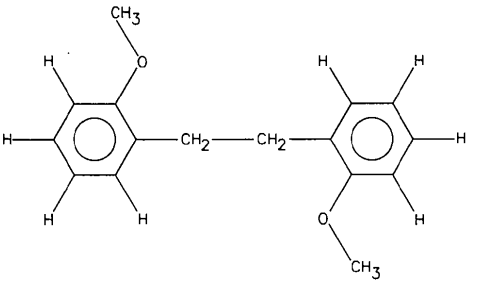
Abstract

The 2′,2′-dimethoxybibenzyl molecule, $C_{16}H_{18}O_2$, has the midpoint of the $—CH_2—CH_2—$ bond on an inversion centre. Each benzene ring deviates slightly from planarity, shows marked angular distortion and is inclined at 81.5 (1)° to the plane of the $C(Ar)—CH_2—CH_2—$ moiety.

Comment

There are only two structures similar to that of the title compound, (I), in the Cambridge Structural Database (1993): dibenzyl (Cruickshank, 1949) and 4,4′-dimethylbibenzyl (Brown, 1954). The structures are similar in that, in each case, the centre of the molecule coincides with a crystallographic centre of symmetry. Both structures have large R factors (15% and 18%, respectively) so that no real comparison with bond lengths and angles can be made, except to say that the current structural details are in broad agreement with those found from the earlier studies.



(I)

The benzene ring is not planar at a 3σ level, the deviations of some of the ring atoms from the ring mean plane being as large as 6σ. The ring also shows a range of angle values which reflect angular distortion due to the ring substituents (Domenicano & Hargittai, 1992). Initially, we were surprised to find a value of 115.28 (10)° for the angle C1—C2—O2, but a search of the April 1993 release of the Cambridge Structural Database for the *o*-$C_6H_4(OMe)CH_2$ fragment yielded ten 'hits' for which the mean value of the C—C—O angle was 115.2° (range 112.0-118.8°). Similarly, the mean values of the torsion angles $CH_2—C—C—O$ and $C—C—O—CH_3$ are 1.7 and 172.6°, respectively. It would appear that the geometry reported here for the title molecule is entirely in accord with that reported in all previous examples.

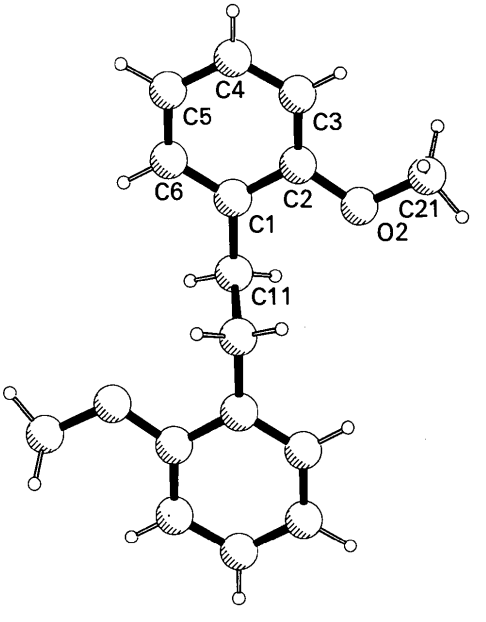


Fig. 1. View of the title molecule.

Experimental

The title compound (m.p. 363-364 K) was obtained as a by-product in the formation of the Grignard reagent from 2-methoxybenzyl chloride in thf. It was recrystallized from ethyl acetate and petroleum ether.

Crystal data

$C_{16}H_{18}O_2$
M_r = 242.31
Monoclinic
$P2_1/n$
a = 7.1487 (24) Å
b = 12.1026 (11) Å
c = 7.8726 (8) Å
β = 99.542 (21)°
V = 671.70 (24) Å^3
Z = 2
D_x = 1.198 Mg m^{-3}
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 24 reflections
θ = 10.00-15.00°
μ = 0.07 mm^{-1}
T = 293 K
Plate
0.67 × 0.50 × 0.33 mm
Colourless

Data collection

Nonius CAD-4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
2074 measured reflections
1944 independent reflections
1343 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.007
θ_{max} = 29.89°
$h = -10 \rightarrow 9$
$k = 0 \rightarrow 17$
$l = 0 \rightarrow 11$
3 standard reflections
frequency: 60 min
intensity variation: < 1%

Refinement

Refinement on F
R = 0.044
wR = 0.069
S = 2.11
1348 reflections
119 parameters
All H-atom parameters refined
$w = 1/[\sigma^2(F) + 0.0008F^2]$
$\Delta\rho_{max}$ = 0.19 e Å^{-3}
$\Delta\rho_{min}$ = −0.17 e Å^{-3}
Extinction correction: Larson (1970)
Extinction coefficient: 0.19 (19) × 10^4
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Table 2.2B)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters* (Å^2)

$U_{eq} = (1/3)\sum_i\sum_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	U_{eq}
C1	0.29089 (16)	0.53098 (10)	0.12087 (16)	0.0479 (6)
C11	0.40879 (18)	0.46976 (10)	0.00939 (19)	0.0528 (7)
C2	0.17137 (15)	0.61770 (10)	0.05199 (16)	0.0461 (6)
O2	0.17646 (13)	0.64255 (9)	−0.11646 (12)	0.0618 (5)
C21	0.0592 (3)	0.72917 (17)	−0.1934 (3)	0.0779 (10)
C3	0.05748 (18)	0.67199 (11)	0.15253 (19)	0.0552 (7)
C4	0.06580 (21)	0.64305 (15)	0.32228 (21)	0.0661 (8)
C5	0.18634 (22)	0.56125 (16)	0.39496 (21)	0.0713 (9)
C6	0.29678 (20)	0.50565 (13)	0.29319 (20)	0.0622 (8)

Table 2. *Selected geometric parameters* (Å, °)

C1—C11	1.5082 (17)	C2—C3	1.3910 (17)
C1—C2	1.4043 (17)	O2—C21	1.4141 (18)
C1—C6	1.3846 (20)	C3—C4	1.3732 (22)
C11—C11^i	1.5238 (25)	C4—C5	1.374 (3)
C2—O2	1.3662 (15)	C5—C6	1.3884 (24)
C11—C1—C2	120.35 (11)	O2—C2—C3	124.10 (11)
C11—C1—C6	122.16 (12)	C2—O2—C21	118.01 (12)
C2—C1—C6	117.49 (12)	C2—C3—C4	119.89 (13)
C1—C11—C11^i	112.98 (10)	C3—C4—C5	120.81 (14)
C1—C2—O2	115.28 (10)	C4—C5—C6	119.10 (14)
C1—C2—C3	120.62 (12)	C1—C6—C5	122.02 (14)

Symmetry code: (i) $1-x, 1-y, -z$.

All H atoms were clearly visible in difference maps at an intermediate stage of the refinement and were refined isotropically. Data collection and refinement: *CAD-4 Software* (Enraf-Nonius, 1989). Data reduction, program used to solve and refine structure and software used to prepare material for publication: *NRCVAX* (Gabe, Le Page, Charland, Lee & White, 1989). Molecular graphics: *PLUTON92* (Spek, 1992).

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: HA1088). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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