Supplementary data for this paper are available from the IUCr electronic archives (Reference: BM1174). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1857–1859

Bis(3,4-dimethoxybenzyl) Ether and Tetramethoxy-4,4′-(2,3-dimethyltetramethylene)dipyrocatechol†

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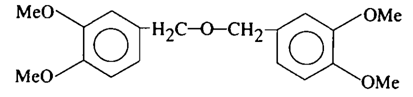
(*Received 14 April 1997; accepted 2 July 1997*)

Abstract

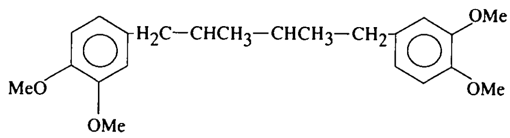
In molecules of both bis(3,4-dimethoxybenzyl) ether, $C_{18}H_{22}O_5$, (I), and *meso*-tetramethoxy-4,4′-(2,3-dimethyltetramethylene)dipyrocatechol, $C_{22}H_{30}O_4$, (II), the methoxy groups are almost coplanar with the attached benzene rings. In (I), the two benzene rings make a dihedral angle of 17.20 (5)°, whereas in (II), they are almost perpendicular [94.8 (4)°] to one another. The crystal structure is stabilized by van der Waals interactions in both compounds.

Comment

The structure determinations of the title compounds, (I) and (II), represent part of an investigation of a series of methoxybenzenes.



(I)



(II)

The mean C_{sp^2}—O [1.366 (2) Å] and C_{sp^3}—O [1.423 (3) Å] bond distances in the methoxy groups agree with values observed in other methoxybenzene derivatives (Fun, Chinnakali, Sivakumar, Sam & How, 1997; Bryan & White, 1982). The methoxy groups are almost coplanar with the attached benzene rings but significant displacements from their adjacent phenyl rings are observed for C15 (0.120 Å) and C18 (0.239 Å) in (I),

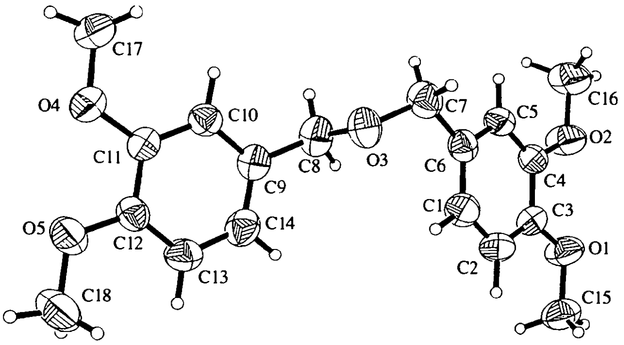


Fig. 1. View of (I) showing the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

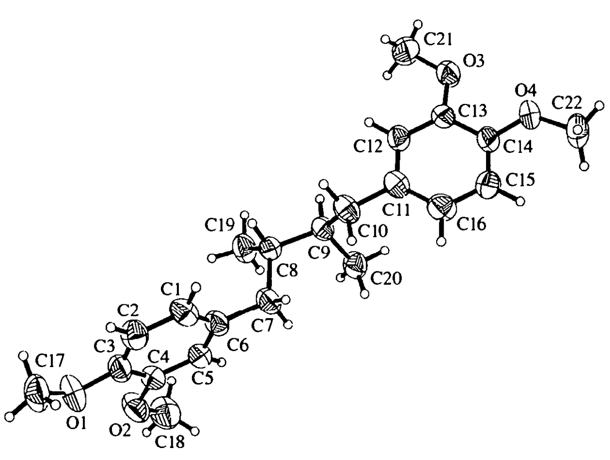


Fig. 2. View of (II) showing the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

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† Alternative name: 1,1′,2,2′-tetramethoxy-4,4′-(2,3-dimethyltetramethylene)dibenzene.

‡ On leave from: Department of Physics, Anna University, Chennai 600 025, India.

and for C17 (0.266 Å) and C22 (0.269 Å) in (II). The dihedral angle between the two benzene ring planes in (I) is 17.20 (5)° and these planes are almost normal to that formed by atoms C7, C8 and O3 [dihedral angles 86.89 (1) and 87.28 (8)°]. The two benzene rings in (II) are almost perpendicular [94.8 (4)°] to one another.

The shortest intermolecular contacts observed are C2···O3$(\frac{1}{2}-x, \frac{1}{2}+y, \frac{1}{2}-z)$ of 3.368 (2) Å and C18···O4$(x, y, z-1)$ of 3.395 (2) Å in compounds (I) and (II), respectively.

Experimental

Compounds (I) and (II) were prepared from their hydroxy precursors by methylation with dimethyl sulfate in the presence of anhydrous potassium carbonate, using dry acetone as solvent. Single crystals were obtained by slow evaporation from ethyl acetate solutions.

Compound (I)

Crystal data

$C_{18}H_{22}O_5$
$M_r = 318.36$
Monoclinic
$P2_1/n$
$a = 10.399 (1)$ Å
$b = 5.806 (1)$ Å
$c = 28.126 (2)$ Å
$\beta = 93.02 (1)°$
$V = 1695.8 (2)$ Å^3
$Z = 4$
$D_x = 1.247$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 52 reflections
$\theta = 2.77–12.34°$
$\mu = 0.090$ mm^{-1}
$T = 293 (2)$ K
Rectangular
0.68 × 0.62 × 0.26 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5423 measured reflections
3911 independent reflections
2158 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.034$

$\theta_{\rm max} = 27.50°$
$h = -1 \rightarrow 13$
$k = -1 \rightarrow 7$
$l = -36 \rightarrow 36$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.040$
$wR(F^2) = 0.110$
$S = 0.865$
3911 reflections
297 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0553P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\rm max} < 0.001$

$\Delta\rho_{\rm max} = 0.14$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.12$ e Å^{-3}
Extinction correction: *SHELXL*93 (Sheldrick, 1993)
Extinction coefficient: 0.0216 (15)
Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. *Selected geometric parameters (Å, °) for (I)*

O1—C3	1.362 (2)	O4—C11	1.368 (2)
O1—C15	1.424 (2)	O4—C17	1.421 (2)
O2—C4	1.366 (2)	O5—C12	1.363 (2)
O2—C16	1.422 (2)	O5—C18	1.431 (2)
O3—C8	1.424 (2)	C6—C7	1.509 (2)
O3—C7	1.425 (2)	C8—C9	1.496 (2)
C8—O3—C7	112.47 (12)	O3—C7—C6	114.00 (14)
O1—C3—C2	125.23 (14)	O3—C8—C9	108.15 (12)
O2—C4—C5	124.65 (14)	O4—C11—C10	125.31 (14)
O2—C4—C3	115.38 (13)	O5—C12—C13	124.84 (14)
C8—O3—C7—C6	−73.3 (2)	C7—O3—C8—C9	−177.41 (14)
C5—C6—C7—O3	153.05 (14)	O3—C8—C9—C10	85.1 (2)

Compound (II)

Crystal data

$C_{22}H_{30}O_4$
$M_r = 358.46$
Triclinic
$P\overline{1}$
$a = 6.274 (1)$ Å
$b = 10.571 (1)$ Å
$c = 15.426 (1)$ Å
$\alpha = 86.49 (1)°$
$\beta = 80.78 (1)°$
$\gamma = 87.72 (1)°$
$V = 1007.5 (2)$ Å^3
$Z = 2$
$D_x = 1.182$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 38 reflections
$\theta = 5.33–12.54°$
$\mu = 0.080$ mm^{-1}
$T = 293 (2)$ K
Rectangular
0.80 × 0.70 × 0.26 mm
Colourless

Data collection

Siemens *P*4 diffractometer
$\theta/2\theta$ scans
Absorption correction: none
5828 measured reflections
4591 independent reflections
2912 reflections with $I > 2\sigma(I)$
$R_{\rm int} = 0.019$

$\theta_{\rm max} = 27.50°$
$h = -1 \rightarrow 8$
$k = -13 \rightarrow 13$
$l = -20 \rightarrow 20$
3 standard reflections every 97 reflections intensity decay: <3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.043$
$wR(F^2) = 0.126$
$S = 0.949$
4591 reflections
355 parameters
All H atoms refined
$w = 1/[\sigma^2(F_o^2) + (0.0735P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\rm max} = 0.001$
$\Delta\rho_{\rm max} = 0.13$ e Å^{-3}
$\Delta\rho_{\rm min} = -0.22$ e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for Crystallography* (Vol. C)

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

O1—C3	1.369 (2)	C6—C7	1.511 (2)
O1—C17	1.413 (2)	C7—C8	1.538 (2)
O2—C4	1.367 (2)	C8—C19	1.515 (2)
O2—C18	1.428 (2)	C8—C9	1.545 (2)
O3—C13	1.366 (2)	C9—C20	1.520 (2)
O3—C21	1.421 (2)	C9—C10	1.534 (2)
O4—C14	1.368 (2)	C10—C11	1.512 (2)
O4—C22	1.424 (2)		
O1—C3—C2	125.20 (14)	C20—C9—C10	111.12 (12)
O2—C4—C5	124.68 (14)	C20—C9—C8	113.26 (11)
C6—C7—C8	114.79 (12)	C11—C10—C9	112.97 (11)
C19—C8—C7	111.72 (12)	C16—C11—C12	118.05 (12)
C19—C8—C9	111.15 (12)	O3—C13—C12	125.34 (12)
C7—C8—C9	112.18 (11)	O4—C14—C15	125.22 (12)
C5—C6—C7—C8	97.2 (2)	C8—C9—C10—C11	−173.98 (12)
C6—C7—C8—C9	−179.90 (13)	C9—C10—C11—C16	−109.1 (2)
C7—C8—C9—C10	−74.5 (2)		

The structures were solved by direct methods and refined by full-matrix least-squares techniques. The H atoms were located from difference Fourier maps and refined isotropically.

Data collection, cell refinement and data reduction: *XSCANS* (Siemens, 1994). Program(s) used to solve structure: *SHELXTL/PC* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL/PC*. Geometrical calculations: *PARST* (Nardelli, 1983).

The authors would like to thank the Malaysian Government and the Universiti Sains Malaysia for research grant R&D No. 190-9609-2801 and short-term grant No. 131-0250-0530. KC thanks the Universiti Sains Malaysia for a Visiting Postdoctoral Fellowship.

Supplementary data for this paper are available from the IUCr electronic archives (Reference: MU1334). Services for accessing these data are described at the back of the journal.

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Acta Cryst. (1997). C**53**, 1859–1862

Substituted Methoxybenzene Derivatives: $C_8H_9NO_4$, $C_9H_{11}NO_5$ and $C_{13}H_{18}O_4$

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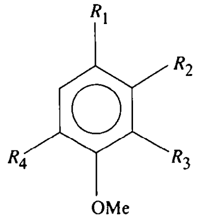
(*Received 14 April 1997; accepted 2 July 1997*)

Abstract

The structures of three methoxybenzenes, namely 1,2-dimethoxy-4-nitrobenzene, $C_8H_9NO_4$, (I), 1,2,3-trimethoxy-5-nitrobenzene, $C_9H_{11}NO_5$, (II), and 1-(2,4,5-trimethoxyphenyl)-1-butanone, $C_{13}H_{18}O_4$, (III), are reported. Molecules of (I) and (III) are planar, but one of the three methoxy groups of (II) is twisted out of the phenyl ring plane as a result of steric hindrance. In all three solids, the molecules are linked to form centrosymmetrically hydrogen-bonded dimers; they are packed in parallel layers in (I) and (II), but in zigzag layers in (III).

Comment

This paper reports an investigation of a series of methoxybenzenes, (I), (II) and (III), which was carried out since such moieties often occur in natural products.



	R_1	R_2	R_3	R_4
(I)	NO_2	H	OMe	H
(II)	NO_2	H	OMe	OMe
(III)	Butanone	OMe	H	OMe

The mean lengths of the C_{sp^2}—O [1.361 (3) Å] and O—CH_3 [1.425 (3) Å] bonds in the methoxy groups of these compounds agree with values observed for related structures (Bryan & White, 1982). Molecules of (I) and (III) are essentially planar; the steric interactions of the methoxy groups in (II) cause C8 to deviate by 1.173 (2) Å from the mean plane formed by the remaining non-H atoms.

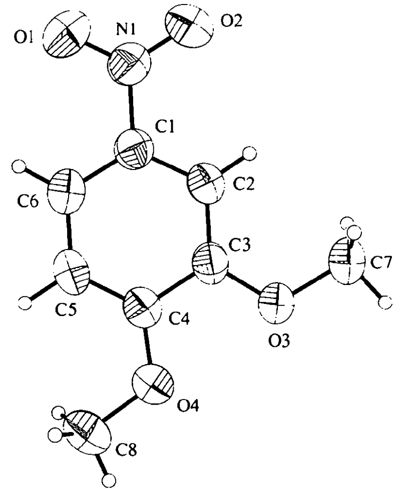


Fig. 1. View of (I) showing the atom-numbering scheme; displacement ellipsoids are drawn at the 50% probability level.

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Acta Crystallographica Section C
ISSN 0108-2701 © 1997