Références

BOGER, D. L. & WEINREB, S. M. (1987). *Organic Chemistry*, Tome 47, ch 9, p. 261. London, New York: Academic Press.

PRADÈRE, J. P., ROZÉ, J. C., QUINIOU, H., DANION-BOUGOT, D., DANION, D. & TOUPET, L. (1986). *Can. J. Chem.* **64**, 597.

TEA, C. G., PRADÈRE, J. P. & QUINIOU, H. (1986). *Synth. Commun.* **16**, 79.

TEA, C. G., PRADÈRE, J. P., QUINIOU, H. & TOUPET, L. (1985). *J. Chem. Soc. Perkin* I, p. 1875.

VERBOOM, W., HAMZINK, M. R. J., REINHOUDT, D. N. & VISSER, R. (1984). *Tetrahedron Lett.* **25**, 4309.

Acta Cryst. (1989). C**45**, 1255–1256

2,7-Dimethoxynaphthalene

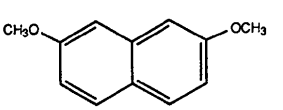
BY PHILIPPE PRINCE, FRANK R. FRONCZEK AND RICHARD D. GANDOUR*

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, *USA*

(*Received* 11 *January* 1989; *accepted* 6 *March* 1989)

Abstract. $C_{12}H_{12}O_2$, $M_r = 188{\cdot}2$, orthorhombic, $P2_12_12_1$, $a = 6{\cdot}109\ (3)$, $b = 8{\cdot}235\ (2)$, $c = 19{\cdot}713\ (3)$ Å, $V = 991{\cdot}8\ (9)$ Å^3, $Z = 4$, $D_x = 1{\cdot}260$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0{\cdot}79$ cm^{-1}, $F(000) = 400$, $T = 293$ K, $R = 0{\cdot}047$ for 737 observations (of 1337 unique data). The average deviation from planarity is 0·013 (3) Å with a maximum of 0·027 (3) Å for the fused rings. Steric interaction between the methyl groups and the adjacent H atoms is responsible for opening the angles between the methoxyl groups and the rings. The CH_3O–C–C angles are 125·5 (3)° [125·0 (3)°] where the methyl group is *syn* to the hydrogen, and 114·3 (3)° [114·2 (3)°] where it is *anti*.

Experimental. Colorless plates of (1), m.p. 412–413 K, were isolated by recrystallization in dichloromethane/hexane from the crude reaction product of 2,7-naphthalenediol and dimethyl sulfate with sodium hydroxide in water at reflux temperature (Johansson, Mellin & Hacksell, 1986). Crystal size 0·13 × 0·43 × 0·45 mm, capillary-mounted, space group from systematic absences $h00$ with h odd, $0k0$ with k odd and $00l$ with l odd, cell dimensions from setting angles of 25 reflections having $10 < \theta < 11°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time = 180 s, scan rates varied 0·39–4·00° min^{-1}. Two octants of data having $2 < 2\theta < 55°$, $-7 \le h \le 7$, $0 \le k \le 10$, $0 \le l \le 25$ measured. Data corrected for background, Lorentz and polarization effects, not for absorption. Standard reflections 102, 020, 006 varied randomly 2·4%, and no decay correction was applied. The two octants (2486 measurements) merged, $R_{int} = 0{\cdot}029$, to yield 1337 unique data, 737 observed with $I > 1\sigma(I)$. Structure solved by direct methods, using *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), refined by full-matrix least squares based upon F with weights $w = 4F_o^2Lp[S^2(C + R^2B) + (0{\cdot}02F_o^2)^2]^{-1}$, where S = scan rate, C = integrated count, R = scan time/background time, B = background count, using Enraf–Nonius *SDP* (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous coefficients of Cromer (1974). Non-H atoms refined anisotropically; H atoms located by ΔF map and included as fixed contributions with C–H 0·95 Å and $B = 1{\cdot}3B_{eq}$ of the bonded C atom. Final $R = 0{\cdot}047$, $wR = 0{\cdot}042$, $S = 1{\cdot}561$ for 127 variables. Max. shift $0{\cdot}02\sigma$ in the final cycle, max. residual density 0·22, min. −0·16 e Å^{-3}. Coordinates† are given in Table 1; bond distances and angles are given in Table 2. The molecule is illustrated in Fig. 1.



(1)

Related literature. Space group of 2,7-naphthalenediol: Ahmed (1978). Macrocycles containing the 2,7-dioxynaphthyl group: Hamilton & Van Engen (1987); Muehldorf, Van Engen, Warner & Hamilton (1988).

Support for this work is provided by a grant from the National Institutes of Health.

* To whom correspondence should be addressed.

† Tables of H-atom coordinates, least-squares planes, anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51814 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/89/081255-02$03.00

Table 1. *Coordinates and equivalent isotropic thermal parameters*

The equivalent isotropic thermal parameter, for atoms refined anisotropically, is defined by the equation: $B_{eq} = 8\pi^2(U_{11} + U_{22} + U_{33})/3$.

	x	y	z	B_{eq}(Å^2)
O1	0·4356 (4)	0·2007 (3)	0·6606 (1)	5·38 (6)
O2	0·1963 (4)	−0·0296 (3)	1·01088 (9)	5·44 (6)
C1	0·3250 (6)	0·2048 (4)	0·7201 (1)	4·29 (7)
C2	0·1334 (6)	0·3012 (4)	0·7187 (2)	5·31 (9)
C3	0·0051 (7)	0·3105 (4)	0·7741 (2)	5·56 (9)
C4	0·0543 (6)	0·2246 (4)	0·8342 (2)	4·34 (8)
C5	−0·0819 (6)	0·2285 (4)	0·8919 (2)	5·57 (9)
C6	−0·0307 (6)	0·1439 (4)	0·9487 (2)	5·47 (9)
C7	0·1639 (6)	0·0506 (4)	0·9509 (2)	4·57 (8)
C8	0·2997 (5)	0·0425 (4)	0·8961 (1)	3·88 (7)
C9	0·2494 (6)	0·1310 (3)	0·8361 (1)	3·75 (7)
C10	0·3821 (6)	0·1220 (4)	0·7781 (1)	3·90 (7)
C11	0·6100 (7)	0·0888 (4)	0·6554 (2)	6·0 (1)
C12	0·3946 (7)	−0·1203 (5)	1·0187 (2)	6·4 (1)

Table 2. *Bond distances* (Å) *and angles* (°)

O1	C1	1·353 (4)		C4	C5	1·411 (4)	
O1	C11	1·413 (4)		C4	C9	1·420 (4)	
O2	C7	1·369 (4)		C5	C6	1·354 (5)	
O2	C12	1·432 (4)		C6	C7	1·416 (5)	
C1	C2	1·414 (5)		C7	C8	1·364 (4)	
C1	C10	1·376 (4)		C8	C9	1·422 (4)	
C2	C3	1·347 (4)		C9	C10	1·405 (4)	
C3	C4	1·412 (4)					
C1	O1	C11	117·2 (3)	C4	C5	C6	121·2 (3)
C7	O2	C12	117·8 (3)	C5	C6	C7	119·9 (3)
O1	C1	C2	114·3 (3)	O2	C7	C6	114·2 (3)
O1	C1	C10	125·5 (3)	O2	C7	C8	125·0 (3)
C2	C1	C10	120·2 (3)	C6	C7	C8	120·9 (3)
C1	C2	C3	119·9 (3)	C7	C8	C9	120·1 (3)
C2	C3	C4	121·9 (3)	C4	C9	C8	118·9 (3)
C3	C4	C5	122·7 (3)	C4	C9	C10	119·4 (3)
C3	C4	C9	118·3 (3)	C8	C9	C10	121·7 (3)
C5	C4	C9	119·0 (3)	C1	C10	C9	120·3 (3)

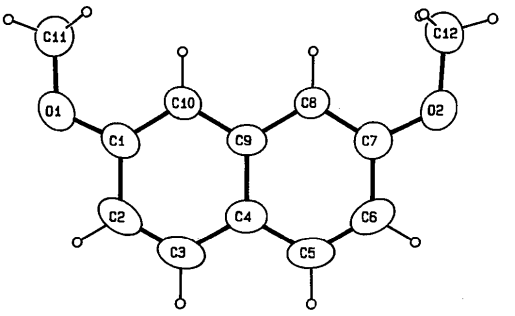


Fig. 1. Numbering scheme and thermal ellipsoids drawn at the 40% probability level. H atoms are drawn as circles with the same arbitrary radius.

References

AHMED, N. A. (1978). *Egypt. J. Phys.* **9**, 67–68.

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

CROMER, D. T. & WABER, J. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2B. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft, The Netherlands.

HAMILTON, A. D. & VAN ENGEN, D. (1987). *J. Am. Chem. Soc.* **109**, 5035–5036.

JOHANSSON, A. M., MELLIN, C. & HACKSELL, U. (1986). *J. Org. Chem.* **51**, 5252–5258.

MAIN, P., FISKE, S. J., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCQ, J.-P. & WOOLFSON, M. M. (1982). *MULTAN*11/82. *A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.

MUEHLDORF, A. V., VAN ENGEN, D., WARNER, J. C. & HAMILTON, A. D. (1988). *J. Am. Chem. Soc.* **110**, 6561–6562.

Acta Cryst. (1989). C**45**, 1256–1258

1-Acetyl-2,7-dimethoxynaphthalene

BY PHILIPPE PRINCE, FRANK R. FRONCZEK AND RICHARD D. GANDOUR*

Department of Chemistry, Louisiana State University, Baton Rouge, LA 70803-1804, *USA*

(*Received* 18 *January* 1989; *accepted* 6 *March* 1989)

Abstract. $C_{14}H_{14}O_3$, $M_r = 230·3$, monoclinic, $P2_1/c$, $a = 8·8107$ (9), $b = 18·372$ (3), $c = 7·7512$ (11) Å, $\beta = 98·49$ (1)°, $V = 1240·9$ (5) Å^3, $Z = 4$, $D_x = 1·232$ g cm^{-3}, λ(Mo $K\alpha$) = 0·71073 Å, $\mu = 0·80$ cm^{-1}, $F(000) = 488$, $T = 293$ K, $R = 0·047$ for 1909 observations (of 2848 unique data). The average deviation from planarity is 0·017 (2) Å with a maximum of 0·0285 (15) Å for the fused rings. The dihedral angle between the naphthalene system and the acetyl group is 117·91 (6)°. The methoxyl group *ortho* to the acetyl adopts a conformation with the methyl group *anti* to the neighboring α-carbon of the ring, with a C—C—O—C torsion angle of −178·7 (2)°. The other methoxyl group has the methyl *syn* to the neighboring α-carbon, with a C—C—O—C torsion angle of −1·3 (3)°.

* To whom correspondence should be addressed.

0108-2701/89/081256-03$03.00