Moretti, C., Polonsky, J., Vuilhorgne, M. & Prange, T. T. *Tetrahedron Lett.* **23**, 647–650.
Polonsky, J. (1985). *Forschr. Chem. Org. Naturst.* **47**, 221–264.
Polonsky, J., Bhatnagar, S. & Moretti, C. (1984). *J. Nat. Prod.* **47**, 994–996.
Sheldrick, G. M. (1976). *SHELX76. Program for Crystal Structure Determination.* Univ. of Cambridge, England.
Sheldrick, G. M. (1985). *SHELXS86. Program for the Solution of Crystal Structures.* Univ. of Göttingen, Germany.
Walker, N. & Stuart, D. (1983). *Acta Cryst.* A**39**, 158–166.

Acta Cryst. (1994). C**50**, 797–798

2-Acetoxy-7-methoxynaphthalene

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

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

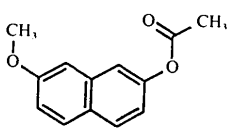
(*Received* 14 *May* 1993; *accepted* 23 *September* 1993)

Abstract

The naphthalene ring system of the title compound (7-methoxy-2-naphthyl acetate, $C_{13}H_{12}O_3$) is slightly non-planar, the average deviation being 0.025 (1) Å, with a maximum deviation of 0.041 (1) Å for the C atom carrying the methoxy group. The methoxy group has the methyl *syn* to the neighboring α-C atom, and is nearly coplanar with the ring, with a C—C—O—C torsion angle of −5.2 (2)°. The dihedral angle between the naphthalene ring system and the acetoxyl group is 115.8 (1)°, with a C—C—O—C torsion angle of 67.2 (2)°.

Comment

The title compound, (1), was prepared by acetylation of 7-methoxy-2-naphthol in acetic anhydride (Gorelic, Reznichenko, Andronova & Luk'yanets, 1983), as an intermediate in the synthesis of new binaphthylacetylenes. Crystals of (1), m.p. 397.5–398.5 K, were isolated by slow evaporation of methanol.



(1)

The methoxy O atom is closer to C2 than C10 as indicated by the angle difference of about 5° from the theoretical 120° for O1—C1—C2 and O1—C1—C10 [114.4 (1) and 125.2 (1)°, respectively]. This is explained by steric interaction between the methyl group and H10. This is observed in similar 2-methoxynaphthalene structures (Prince, Fronczek & Gandour, 1989, 1991*a*, 1991*b*). The carbonyl group is displaced from the plane of the naphthalene system to avoid steric interaction with the H atoms on the ring.

A search of the January 1992 version of the Cambridge Structural Database (Allen, Kennard & Taylor, 1983) revealed no compound with an acetoxy substituent on position 2 or position 7 of naphthalene. Structures containing the 2,7-dioxynaphthyl fragment were found (see Prince, Fronczek & Gandour, 1989, 1991*a*, 1991*b*, and references therein; Watson, Nagl & Eduok, 1989).

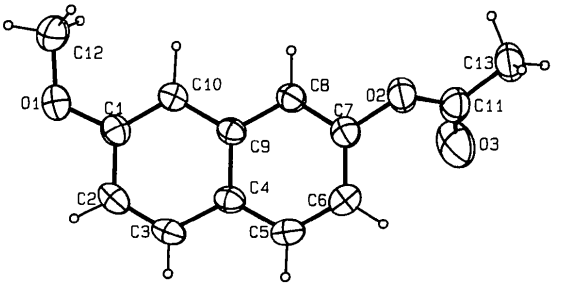


Fig. 1. View of the title compound showing the numbering scheme with displacement ellipsoids drawn at the 40% probability level. H atoms are drawn as circles of arbitrary radii.

Experimental

Crystal data

$C_{13}H_{12}O_3$
M_r = 216.2
Orthorhombic
$P2_12_12_1$
a = 5.8414 (5) Å
b = 7.9263 (10) Å
c = 23.776 (4) Å
V = 1100.8 (4) Å^3
Z = 4
D_x = 1.305 Mg m^{-3}
Cu $K\alpha$ radiation
λ = 1.5418 Å
Cell parameters from 25 reflections
θ = 25–30°
μ = 0.72 mm^{-1}
T = 295 K
Rectangular prism
0.70 × 0.47 × 0.28 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical
T_{min} = 0.9499, T_{max} = 0.9983
2657 measured reflections
2269 independent reflections
2219 observed reflections [$I > 3\sigma(I)$]
R_{int} = 0.014
θ_{max} = 75° (2 octants)
h = 0 → 7
k = 0 → 9
l = −29 → 29
3 standard reflections
frequency: 166.6 min
intensity variation: <2%

† Current address: Department of Chemistry, Virginia Polytechnic Institute and State University, Blacksburg, VA 24061-0212, USA.

Refinement

Refinement on F
$R = 0.032$
$wR = 0.048$
$S = 3.513$
2219 reflections
194 parameters
All H-atom parameters refined
$w = 4F_o^2/[\sigma^2(I) + (0.02F_o^2)^2]$
$(\Delta/\sigma)_{max} = 0.03$
$\Delta\rho_{max} = 0.22$ e Å^{-3}
$\Delta\rho_{min} = -0.19$ e Å^{-3}
Extinction correction: $(1 + gI_c)^{-1}$ applied to F_c
Extinction coefficient: $1.22\,(4) \times 10^{-5}$
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV, Tables 2.2B and 2.3.1)

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

$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij} a_i^* a_j^* \mathbf{a}_i.\mathbf{a}_j$.

	x	y	z	B_{eq}
O1	0.8821 (2)	0.4411 (1)	0.61592 (3)	4.78 (2)
O2	0.5904 (2)	0.4696 (1)	0.32018 (3)	4.53 (2)
O3	0.8189 (3)	0.2979 (2)	0.27142 (5)	7.97 (3)
C1	0.9240 (2)	0.4735 (1)	0.56055 (4)	3.68 (2)
C2	1.1318 (2)	0.5605 (2)	0.55015 (5)	4.07 (2)
C3	1.1936 (2)	0.6025 (1)	0.49715 (5)	3.94 (2)
C4	1.0500 (2)	0.5637 (1)	0.45061 (4)	3.42 (2)
C5	1.1020 (2)	0.6157 (1)	0.39506 (5)	4.05 (2)
C6	0.9572 (2)	0.5820 (1)	0.35143 (5)	4.13 (2)
C7	0.7536 (2)	0.4933 (1)	0.36270 (5)	3.75 (2)
C8	0.6963 (2)	0.4398 (1)	0.41525 (4)	3.49 (2)
C9	0.8428 (2)	0.4751 (1)	0.46109 (4)	3.21 (4)
C10	0.7835 (2)	0.4291 (1)	0.51684 (4)	3.47 (2)
C11	0.6402 (2)	0.3708 (2)	0.27583 (5)	4.61 (2)
C12	0.6694 (3)	0.3677 (2)	0.63046 (6)	5.36 (3)
C13	0.4466 (3)	0.3680 (2)	0.23515 (5)	5.66 (3)

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

O1—C1	1.363 (1)	C4—C5	1.417 (2)
O1—C12	1.415 (2)	C4—C9	1.421 (1)
O2—C7	1.402 (1)	C5—C6	1.365 (2)
O2—C11	1.346 (2)	C6—C7	1.407 (2)
O3—C11	1.198 (2)	C7—C8	1.362 (1)
C1—C2	1.418 (2)	C8—C9	1.414 (1)
C1—C10	1.370 (1)	C9—C10	1.418 (1)
C2—C3	1.353 (2)	C11—C13	1.488 (2)
C3—C4	1.422 (2)		
C1—O1—C12	118.1 (1)	O2—C7—C6	120.3 (1)
C7—O2—C11	119.7 (1)	O2—C7—C8	116.91 (9)
O1—C1—C2	114.4 (1)	C6—C7—C8	122.6 (1)
O1—C1—C10	125.2 (1)	C7—C8—C9	119.78 (9)
C2—C1—C10	120.4 (1)	C4—C9—C8	118.58 (9)
C1—C2—C3	120.7 (1)	C4—C9—C10	119.94 (9)
C2—C3—C4	121.0 (1)	C8—C9—C10	121.45 (9)
C3—C4—C5	122.42 (9)	C1—C10—C9	119.78 (9)
C3—C4—C9	118.22 (9)	O2—C11—O3	122.5 (1)
C5—C4—C9	119.31 (9)	O2—C11—C13	110.7 (1)
C4—C5—C6	121.3 (1)	O3—C11—C13	126.7 (1)
C5—C6—C7	118.5 (1)		
C10—C1—O1—C12	−5.2 (2)	C7—O2—C11—O3	1.6 (2)
C6—C7—O2—C11	67.2 (2)		

Programs used include *MULTAN*11/82 (Main, Fiske, Hull, Lessinger, Germain, Declercq & Woolfson, 1982), *MolEN* (Fair, 1990) and *ORTEP* (Johnson, 1965). The two octants, inequivalent in point group 222, were not averaged. Refinement of the inversion-related structure led to slightly worse agreement, $wR = 0.04803$ *versus* 0.04786.

Support for this work was provided by a grant from the National Science Foundation.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and geometry, least-squares-planes data and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP71678 (19 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: CR1081]

References

Allen, F. H., Kennard, O. & Taylor, R. (1983). *Acc. Chem. Res.* **16**, 146–153.
Fair, C. K. (1990). *MolEN. An Interactive Intelligent System for Crystal Structure Analysis.* Enraf–Nonius, Delft, The Netherlands.
Gorelic, A. M., Reznichenko, A. V., Andronova, N. A. & Luk'yanets, E. A. (1983). *J. Org. Chem. USSR*, **19**, 183–189.
Johnson, C. K. (1965). *ORTEP.* Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
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.
Prince, P., Fronczek, F. R. & Gandour, R. D. (1989). *Acta Cryst.* C**45**, 1255–1256.
Prince, P., Fronczek, F. R. & Gandour, R. D. (1991*a*). *Acta Cryst.* C**47**, 2218–2220.
Prince, P., Fronczek, F. R. & Gandour, R. D. (1991*b*). *Acta Cryst.* C**47**, 2226–2227.
Watson, W. H., Nagl, A. & Eduok, E. (1989). *Acta Cryst.* C**45**, 303–306.

Acta Cryst. (1994). C**50**, 798–801

11-Ketoprogesterone

VIVEK K. GUPTA, RAJNIKANT AND K. N. GOSWAMI*

Department of Physics, University of Jammu, Jammu Tawi 1, India

S. K. MAZUMDAR

Crystallography and Molecular Biology Division, Saha Institute of Nuclear Physics, Calcutta 64, India

K. K. BHUTANI

Regional Research Laboratory, Jammu Tawi 1, India

(*Received* 24 *February* 1992; *accepted* 25 *June* 1993)

Abstract

In the title compound, 4-pregnen-3,11,20-trione, $C_{21}H_{28}O_3$, ring *A* exists in a sofa conformation.