

data). Refinement minimizing $\sum w\Delta^2$ to R 0.062, wR 0.076. All non-H atoms anisotropic; H atoms in calculated positions, $U(H)$ refined to 0.083 (9) Å² for CH₂, 0.073 (11) Å² for CH; constant weights, 49 refined parameters, max. Δ/σ 0.002; max. features in final difference map 0.163, -0.180 e Å⁻³. Atomic scattering curves from *SHELX76*. Final coordinates are given in Table 1,* with bond lengths and angles in Table 2. The molecule is shown in Fig. 1.

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44178 (5 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Related literature. The synthesis and properties of this compound will be reported by Schroth in a subsequent publication.

References

- MOTHERWELL, W. D. S. & CLEGG, W. (1978). *PLUTO78*. Program for plotting crystal and molecular structures. Univ. of Cambridge, England.
 ROBERTS, P. & SHELDRIK, G. M. (1975). *XANADU*. Program for crystallographic calculations. Univ. of Cambridge, England.
 SCHOMAKER, D. P. & TRUEBLOOD, K. N. (1968). *Acta Cryst.* **B24**, 63–72.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SHELDRIK, G. M. (1986). *SHELXS*. Program for crystal structure determination. Univ. of Göttingen, Federal Republic of Germany.

Acta Cryst. (1987). **C43**, 2246–2247

2-(1,4-Dimethoxy-2-naphthyl)-2-phenylacetonitrile

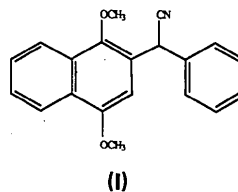
BY UPALI SIRIWARDANE, SUBHASH P. KHANAPURE, SHIRLEY S. C. CHU* AND EDWARD R. BIEHL*

Departments of Chemistry and Electrical Engineering, Southern Methodist University, Dallas, Texas 75275, USA

(Received 21 January 1987; accepted 15 June 1987)

Abstract. C₂₀H₁₇NO₂, $M_r = 303.36$, triclinic, $P\bar{1}$, $a = 7.885$ (4), $b = 9.515$ (5), $c = 11.591$ (6) Å, $\alpha = 84.19$ (4), $\beta = 69.95$ (4), $\gamma = 99.74$ (4)°, $V = 793.2$ (7) Å³, $Z = 2$, $D_x = 1.27$ g cm⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu(\text{Mo } K\alpha) = 0.76$ cm⁻¹, $F(000) = 320$, $T = 295$ K. Final $R = 0.038$ for 1631 observed reflections. The naphthyl, phenyl, nitrile and hydrogen moieties are arranged in a distorted tetrahedron with respect to the α -carbon atom. The angle between the planes formed by the phenyl and naphthyl rings is 78.91 (6)°. The bond distances are O—CH₃(ave.) = 1.426 (3), (CH₃)O—C(ave.) = 1.372 (2), C≡N = 1.133 (3), C—CN = 1.472 (3) and (CN)C—C(ave.) = 1.524 (3) Å. The structure confirms simple anion addition of the sodium salt of phenylacetonitrile to 3,8-dimethoxynaphthene in the reaction.

Experimental. Recently, we found a simple method for the preparation of (I) in 70% yield and a variety of structurally related 2-alkyl- and 2-aryl-2-(1,4-dimethoxy-2-naphthyl)acetonitriles in high yields by the addition of sodium alkyl nitriles to the aryne intermediate generated from the appropriate haloarene (Khanapure & Biehl, 1987).



Crystals of (I) are colorless rhombohedral plates; a single crystal of dimensions 0.40 × 0.60 × 0.24 mm was mounted on a goniometer head with an epoxy resin; unit-cell parameters obtained by least-squares refinement of 15 reflections in the range $10 < 2\theta < 25^\circ$, automatic Syntex *P2*, diffractometer, graphite-monochromated Mo $K\alpha$ radiation, $\theta/2\theta$ scan mode, variable scan rate (3.0–14.7° min⁻¹, depending on intensity), 2358 measured reflections, 2094 independent reflections in the range $3 < 2\theta < 45^\circ$, $R_{\text{int}} = 0.016$, hkl range $h -8 \rightarrow 8$, $k -10 \rightarrow 10$, $l 0 \rightarrow 12$, 1631 observed reflections with $I > 3\sigma(I)$, $\sigma(I)$ from counting statistics; three standard reflections remeasured after every 100 reflections did not show any significant change (~3%) in intensity during data collection; Lorentz-polarization correction, no absorption or extinction corrections. Direct-methods *MULTAN78* (Main, Hull, Lessinger, Germain, Declercq & Woolfson, 1978),

* To whom correspondence should be addressed.

Table 1. Fractional atomic coordinates with equivalent isotropic thermal parameters for the non-H atoms (e.s.d.'s in parentheses)

	x	y	z	$U_{eq}(\text{\AA}^2)^*$
O(1)	0.5724 (2)	0.1132 (1)	0.1387 (1)	0.0536 (3)
O(2)	0.1701 (2)	0.5411 (1)	0.2111 (1)	0.0649 (3)
N	0.6863 (2)	0.4302 (2)	0.4320 (2)	0.0744 (5)
C(1)	0.5052 (2)	0.2182 (2)	0.3659 (2)	0.0460 (4)
C(2)	0.4386 (2)	0.2769 (2)	0.2658 (2)	0.0412 (4)
C(3)	0.4733 (2)	0.2211 (2)	0.1591 (2)	0.0422 (4)
C(4)	0.3989 (2)	0.2666 (2)	0.0693 (2)	0.0422 (4)
C(5)	0.4243 (3)	0.2054 (2)	-0.0394 (2)	0.0564 (5)
C(6)	0.3497 (3)	0.2507 (2)	-0.1226 (2)	0.0688 (6)
C(7)	0.2490 (3)	0.3583 (2)	-0.1028 (2)	0.0670 (6)
C(8)	0.2199 (3)	0.4194 (2)	0.0017 (2)	0.0549 (5)
C(9)	0.2954 (2)	0.3746 (2)	0.0902 (2)	0.0422 (4)
C(10)	0.2678 (2)	0.4351 (2)	0.2008 (2)	0.0446 (4)
C(11)	0.3354 (2)	0.3849 (2)	0.2865 (2)	0.0452 (4)
C(12)	0.3454 (2)	0.1220 (2)	0.4760 (2)	0.0458 (4)
C(13)	0.2652 (3)	0.1679 (2)	0.5865 (2)	0.0555 (5)
C(14)	0.1184 (3)	0.0754 (3)	0.6824 (2)	0.0655 (6)
C(15)	0.0510 (3)	-0.0613 (2)	0.6670 (2)	0.0646 (5)
C(16)	0.1290 (3)	-0.1065 (2)	0.5562 (2)	0.0664 (6)
C(17)	0.2767 (3)	-0.0159 (2)	0.4609 (2)	0.0557 (5)
C(18)	0.7653 (3)	0.1672 (2)	0.0662 (2)	0.0674 (6)
C(19)	0.1448 (3)	0.6101 (3)	0.3167 (2)	0.0750 (6)
C(20)	0.6067 (3)	0.3377 (2)	0.4041 (2)	0.0522 (4)

* The standard deviations of the U_{eq} 's were calculated according to Schomaker & Marsh (1983).

refinement by full-matrix least squares using *SHELX76* (Sheldrick, 1976), non-hydrogen atoms anisotropic; H atoms located in difference Fourier maps, H isotropic; $w = 1/[\sigma^2(F) + 0.000248F^2]$, $\sum w(|F_o| - |F_c|)^2$ minimized, $R = 0.038$ and $wR = 0.041$ for 1621 observed reflections and 276 parameters, $(\Delta/\sigma)_{max} = 0.01$, $\Delta\rho_{(max, min)} = 0.13, -0.08 \text{ e \AA}^{-3}$ in final difference Fourier map. Atomic scattering factors for C, H, N and O used were taken from *International Tables for X-ray Crystallography* (1974). The final atomic parameters of the non-H atoms are given in Table 1.* The identification of the atoms and the configuration of the title compound are shown in the *ORTEP* (Johnson, 1965) drawing of Fig. 1. Bond lengths, bond angles and selected torsion angles with their standard deviations are given in Table 2.

Related literature. The title compound (I) is representative of a class of methoxy-substituted naphthylacetonitriles that serve as valuable precursors in the synthesis of anthracyclinone intermediates. The usual method for preparing substituted naphthylacetonitriles is given by Parker (Parker & Iqbal, 1980).

* Lists of structure factors, hydrogen parameters, least-squares planes and anisotropic temperature factors and a stereoscopic drawing of the unit cell have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44152 (19 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 2. Bond lengths (\AA), selected bond angles ($^\circ$) and torsion angles ($^\circ$)

O(1)—C(3)	1.384 (2)	C(5)—C(6)	1.361 (3)
O(1)—C(18)	1.434 (3)	C(6)—C(7)	1.389 (3)
O(2)—C(10)	1.360 (2)	C(7)—C(8)	1.362 (3)
O(2)—C(19)	1.417 (3)	C(8)—C(9)	1.416 (3)
N—C(20)	1.133 (3)	C(9)—C(10)	1.422 (3)
C(1)—C(2)	1.523 (3)	C(10)—C(11)	1.361 (3)
C(1)—C(12)	1.525 (3)	C(12)—C(13)	1.372 (3)
C(1)—C(20)	1.472 (3)	C(12)—C(17)	1.384 (3)
C(2)—C(3)	1.355 (3)	C(13)—C(14)	1.387 (3)
C(2)—C(11)	1.405 (3)	C(14)—C(15)	1.373 (3)
C(3)—C(4)	1.424 (3)	C(15)—C(16)	1.368 (3)
C(4)—C(5)	1.408 (3)	C(16)—C(17)	1.380 (3)
C(4)—C(9)	1.407 (3)		
C(3)—O(1)—C(18)	113.2 (1)	O(1)—C(3)—C(2)	120.7 (2)
C(10)—O(2)—C(19)	117.3 (2)	O(1)—C(3)—C(4)	118.8 (2)
C(2)—C(1)—C(12)	111.2 (2)	O(2)—C(10)—C(11)	125.4 (2)
C(2)—C(1)—C(20)	110.4 (2)	C(1)—C(12)—C(13)	122.8 (2)
C(12)—C(1)—C(20)	112.3 (2)	C(1)—C(12)—C(17)	117.7 (2)
C(1)—C(2)—C(3)	119.9 (2)	N—C(20)—C(1)	179.1 (2)
C(1)—C(2)—C(11)	119.8 (2)		
O(1)—C(3)—C(2)—C(1)	-1.4 (3)	C(4)—C(3)—O(1)—C(18)	87.6 (2)
O(1)—C(3)—C(2)—C(11)	-179.0 (2)	C(11)—C(2)—C(1)—C(20)	-52.9 (2)
O(1)—C(3)—C(4)—C(5)	-0.3 (3)	C(11)—C(10)—O(2)—C(19)	3.4 (3)
C(2)—C(3)—O(1)—C(18)	-96.0 (2)	C(13)—C(12)—C(1)—C(20)	21.6 (3)
C(3)—C(2)—C(1)—C(20)	129.5 (2)	C(17)—C(12)—C(1)—C(20)	-160.2 (2)

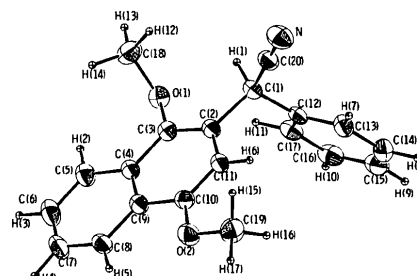


Fig. 1. *ORTEP* (Johnson, 1965) drawing of the molecule. Thermal ellipsoids scaled to enclose 30% probability. Hydrogen atoms are represented as spheres of arbitrary radii.

This work was supported by the Welch Foundation under grants Nos. N-118 and N-495.

References

- International Tables for X-ray Crystallography* (1974), Vol. IV. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
- JOHNSON, C. K. (1965). *ORTEP*. Report ORNL-3794. Oak Ridge National Laboratory, Tennessee, USA.
- KHANAPURE, S. P. & BIEHL, E. R. (1987). *J. Org. Chem.* In the press.
- MAIN, P., HULL, S. E., LESSINGER, L., GERMAIN, G., DECLERCO, J.-P. & WOOLFSON, M. M. (1978). *MULTAN78. A System of Computer Programs for the Automatic Solution of Crystal Structures from X-ray Diffraction Data*. Univs. of York, England, and Louvain, Belgium.
- PARKER, K. A. & IQBAL, T. (1980). *J. Org. Chem.* **45**, 1149–1151.
- SCHOMAKER, V. & MARSH, R. E. (1983). *Acta Cryst.* **A39**, 819–820.
- SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.