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2-Methoxynaphthalene at 173 K

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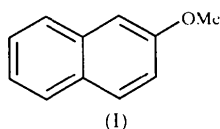
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Abstract

2-Methoxynaphthalene, C₁₁H₁₀O, crystallizes as a nearly planar molecule, with the methoxy group adopting a synperiplanar conformation with respect to the C_α atom. A search of the Cambridge Structural Database [Allen & Kennard (1993). *Chem. Des. Autom. News*, **8**, 31–37] reveals that this conformation is characteristic of the α-methoxynaphthalene moiety, because, for each of the four exceptions found, a valid explanation can be given as to why they display a different conformation.

Comment

The title compound, (I), is a common moiety in crystallographically well explored compounds, many of which have properties of potential use in medical treatment. Some of these compounds exhibit anti-inflammatory, analgesic and antipyretic activities (Ravikumar *et al.*, 1985), some display oestrogenic activity (Stora & Rerat, 1974; Cox & Sim, 1978) and others show promise in the field of cancer research (Wadhawan & Sikka, 1976; McKenna *et al.*, 1991).



We retrieved a total of 62 fragments containing a β-methoxynaphthalene moiety, with H atoms at the α and γ positions of the aromatic ring, by searching the Cambridge Structural Database (CSD, Version 5.15, April 1998; Allen & Kennard, 1993). Bond lengths and angles of the almost planar [r.m.s. deviation 0.014 (2) Å] title compound (Table 1) agree well with the mean values of the structures retrieved from the CSD: O—CH₃ 1.42 (2), O—C_{ar} 1.36 (2) Å, C—O—C 117 (1), O—C_β—C_α 124 (4), O—C_β—C_γ 124 (4)°.

A surprising aspect of almost all these reported molecules is the fact that the conformation of the methoxy group attached to the naphthalene moiety is *syn* with respect to the C_α atom. 58 of the structures from the CSD showed the 2-methoxynaphthalene moiety in the aforementioned conformation. In three of the four exceptions (Fridrichsons & Mathieson, 1955; Wadhawan & Sikka, 1976), the retrieved fragment was found to be part of a phenanthrene moiety, and in the fourth structure (Caira *et al.*, 1995) the conformation of the fragment is obviously influenced by a cyclodextrin host molecule, because the crystal structure of the guest without a host (Ravikumar *et al.*, 1985) shows the expected conformation. Therefore, these exceptions cannot really be compared with the other structures.

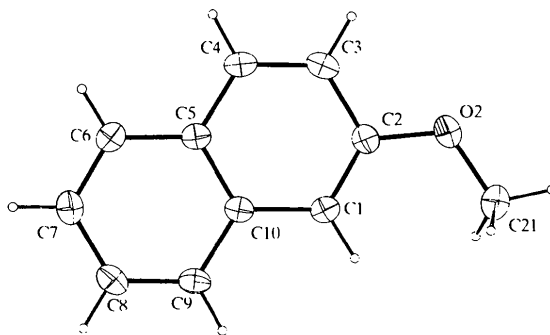


Fig. 1. A perspective view of the title compound with the atom-numbering scheme; displacement ellipsoids are at the 50% probability level.

Experimental

2-Methoxynaphthalene was first reported by Staedel (1882), who described a synthesis similar to our procedure: the only differences are that he used methyl iodide instead of dimethyl sulfate and methanol instead of water as the solvent. The procedure used here is as follows: the title compound was obtained by adding dimethyl sulfate to an aqueous solution of β-naphthol in 2 M NaOH at 313 K. The solution was then heated to 373 K and stirred continuously for 30 min at this temperature. After cooling to room temperature, the precipitated substance was isolated by filtration and purified by recrystallization from ethanol. 2-Methoxynaphthalene is a colourless compound which undergoes slow decomposition when exposed to daylight under atmospheric conditions, leaving a brown residue. This kind of chemical behaviour was observed during our attempts to obtain suitable crystals for the X-ray structure analysis. These attempts were carried out with several solvents, of which the following proved to be useful: hexane, cyclohexane, hexane/dichloromethane and 2-propanol. The crystals were formed during slow evaporation of the solvent. The main difficulty was that the title compound showed a great tendency to form thin plates. In the end, we used the crystals from the 2-propanol solution.

Crystal data

$C_{11}H_{10}O$
 $M_r = 158.19$
 Monoclinic
 $P2_1/c$
 $a = 17.104 (1) \text{ \AA}$
 $b = 6.012 (1) \text{ \AA}$
 $c = 8.110 (1) \text{ \AA}$
 $\beta = 95.46 (1)^\circ$
 $V = 830.16 (18) \text{ \AA}^3$
 $Z = 4$
 $D_r = 1.266 \text{ Mg m}^{-3}$
 D_m not measured

Mo $K\alpha$ radiation
 $\lambda = 0.71073 \text{ \AA}$
 Cell parameters from 8192 reflections
 $\theta = 1-25^\circ$
 $\mu = 0.080 \text{ mm}^{-1}$
 $T = 173 (2) \text{ K}$
 Plate
 $0.80 \times 0.65 \times 0.20 \text{ mm}$
 Colourless

Data collection

Siemens CCD three-circle diffractometer
 ω scans
 Absorption correction: *SADABS* (Sheldrick, 1996)
 $T_{\min} = 0.939$, $T_{\max} = 0.984$
 15 038 measured reflections
 2075 independent reflections
 1734 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.018$
 $\theta_{\max} = 28.95^\circ$
 $h = -22 \rightarrow 22$
 $k = -7 \rightarrow 8$
 $l = -10 \rightarrow 10$
 1310 standard reflections
 frequency: 1320 min
 intensity decay: none

Refinement

Refinement on F^2
 $R[F^2 > 2\sigma(F^2)] = 0.038$
 $wR(F^2) = 0.112$
 $S = 1.072$
 2075 reflections
 109 parameters
 H atoms: see below
 $w = 1/[\sigma^2(F_o^2) + (0.064P)^2 + 0.1403P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$
 $\Delta\rho_{\max} = 0.266 \text{ e \AA}^{-3}$
 $\Delta\rho_{\min} = -0.212 \text{ e \AA}^{-3}$
 Extinction correction: none
 Scattering factors from *International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (\AA , $^\circ$)

C2—O2	1.3749 (11)	O2—C21	1.4250 (14)
O2—C2—C1	125.18 (9)	C1—C2—C3	120.80 (9)
O2—C2—C3	114.01 (8)	C2—O2—C21	117.22 (8)

The data collection nominally covered a sphere of reciprocal space, by a combination of eight sets of exposures; each set had a different φ angle for the crystal and each exposure covered 0.3° in ω . The crystal-to-detector distance was 5.8 cm. Coverage of the unique set is complete to at least 26° in θ . Crystal decay was monitored by repeating the initial frames at the end of data collection and analysing the duplicate reflections. H atoms were located by difference Fourier synthesis and refined with fixed individual displacement parameters [$U(H) = 1.5U_{\text{eq}}(\text{C}_{\text{methyl}})$ or $U(H) = 1.2U_{\text{eq}}(\text{C})$], using a riding model with C—H(methyl) = 0.98 or C—H(aromatic) = 0.95 \AA .

Data collection: *SMART* (Siemens, 1995). Cell refinement: *SMART*. Data reduction: *SAINT* (Siemens, 1995). Program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a). Program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997b). Molecular graphics: *PLUTON* (Spek, 1990).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SK1220). Services for accessing these data are described at the back of the journal.

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Bromo(ethyl)hydridobis(3,4-dimethylpyridine-*N*)silicon Bromide at 173 K

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Abstract

In aprotic solvents, dibromoethylsilane (HSiEtBr_2) and 3,4-dimethylpyridine form a stable 1:2 crystalline complex. X-ray structure analysis reveals an $[\text{HSiEtBr}(\text{3,4-dimethylpyridine})_2]^+$ ($\text{C}_{16}\text{H}_{24}\text{BrN}_2\text{Si}^+\cdot\text{Br}^-$) complex, with one Br^- ion in the outer coordination sphere. The ligands bound directly to the Si centre exhibit almost trigonal-bipyramidal geometry. The geometry of the title compound compares well with that of bromo(hydrido)methylbis(4-methylpyridine-*N*)silicon bromide.