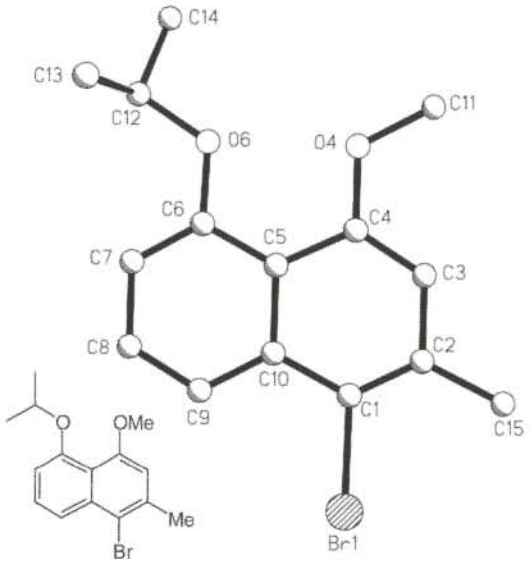
Crystal structure of 1-bromo-5-isopropoxy-4-methoxy-2-methyl-naphthalene, $C_{10}H_4(OC_3H_7)(OCH_3)(CH_3)Br$

K. Peters*[I], E.-M. Peters[I], W. Saeb[II] and G. Bringmann[II]

[I] Max-Planck-Institut für Festkörperforschung, Heisenbergstraße 1, D-70506 Stuttgart, Germany
[II] Universität Würzburg, Institut für Organische Chemie, Am Hubland, D-97074 Würzburg, Germany

Received December 9, 1999, CCDC-No. 1267/352



Abstract

$C_{15}H_{17}BrO_2$, monoclinic, $P12_1/n1$ (No. 14), a = 10.912(2) Å, b = 8.690(2) Å, c =15.112(3) Å, β = 98.98(2)°, V = 1415.4 Å^3, Z = 4, $R_{gt}(F)$ = 0.085, $wR(F)$ = 0.070, T = 293 K.

Source of material

The title compound, a synthetic precursor to several naphthyl-isoquinoline alkaloids [1], was synthesized by reduction of the corresponding bromomethyl naphthalene derivative (prepared from the hydroxymethyl analog [2]) using $LiAlH_4$ in THF.

Table 1. Data collection and handling.

Crystal:	colourless plate, size 1.3 × 1.35 × 0.25 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	29.00 cm^{-1}
Diffractometer, scan mode:	Bruker AXS P4, ω
$2\theta_{max}$:	55°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	4074, 3022
Criterion for F_{obs}, $N(hkl)_{gt}$:	$F_{obs} > 3\,\sigma(F_{obs})$, 1920
$N(param)_{refined}$:	163
Program:	SHELXTL-plus [3]

Table 2. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{iso}
H(3)	4e	–0.0752(6)	0.2763(7)	0.9987(4)	0.08
H(7)	4e	0.3013(7)	–0.1253(7)	1.2860(4)	0.08
H(8)	4e	0.4465(6)	–0.1240(7)	1.1857(5)	0.08
H(9)	4e	0.3976(7)	–0.0084(8)	1.0457(5)	0.08
H(11A)	4e	–0.2136(6)	0.2365(8)	1.1824(5)	0.08
H(11B)	4e	–0.2111(6)	0.1965(8)	1.0815(5)	0.08
H(11C)	4e	–0.1499(6)	0.3491(8)	1.1224(5)	0.08
H(12)	4e	0.1506(6)	–0.1712(7)	1.3432(4)	0.08
H(13A)	4e	0.2067(7)	–0.0352(8)	1.4735(4)	0.08
H(13B)	4e	0.1544(7)	0.1168(8)	1.4260(4)	0.08
H(13C)	4e	0.2715(7)	0.0374(8)	1.3978(4)	0.08
H(14A)	4e	–0.0101(7)	–0.1528(8)	1.4298(4)	0.08
H(14B)	4e	–0.0687(7)	–0.1570(8)	1.3283(4)	0.08
H(14C)	4e	–0.0563(7)	–0.0002(8)	1.3802(4)	0.08
H(15A)	4e	0.1122(7)	0.2982(8)	0.8291(4)	0.08
H(15B)	4e	0.0310(7)	0.4119(8)	0.8759(4)	0.08
H(15C)	4e	–0.0261(7)	0.2569(8)	0.8354(4)	0.08

Table 3. Atomic coordinates and displacement parameters (in Å^2).

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
Br(1)	4e	0.31369(9)	0.1488(1)	0.89818(5)	0.1100(7)	0.1395(8)	0.1010(7)	–0.0157(6)	0.0575(5)	0.0036(5)
C(1)	4e	0.1949(6)	0.1509(7)	0.9798(4)	0.076(5)	0.075(4)	0.069(4)	–0.011(4)	0.032(4)	–0.003(3)
C(2)	4e	0.0894(7)	0.2259(7)	0.9547(4)	0.090(6)	0.075(4)	0.057(4)	–0.010(4)	0.009(4)	–0.001(3)
C(3)	4e	0.0038(6)	0.2274(7)	1.0158(4)	0.059(4)	0.079(4)	0.066(4)	0.002(4)	0.003(3)	0.002(3)
C(4)	4e	0.0301(6)	0.1613(7)	1.0981(4)	0.070(5)	0.069(4)	0.066(4)	–0.009(4)	0.019(3)	–0.018(3)
O(4)	4e	–0.0528(4)	0.1655(5)	1.1586(3)	0.059(3)	0.122(4)	0.070(3)	0.022(3)	0.017(2)	–0.001(3)
C(5)	4e	0.1416(6)	0.0773(6)	1.1262(4)	0.047(4)	0.064(4)	0.062(4)	–0.002(3)	0.012(3)	–0.009(3)
C(6)	4e	0.1753(6)	0.0022(7)	1.2087(4)	0.056(4)	0.067(4)	0.069(4)	0.003(3)	0.020(3)	–0.002(3)
O(6)	4e	0.0896(4)	0.0063(5)	1.2651(3)	0.067(3)	0.099(3)	0.066(3)	0.008(3)	0.021(2)	0.010(2)

* Correspondence author
(e-mail: peters@vsibm1.mpi-stuttgart.mpg.de)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(7)	4*e*	0.2827(7)	–0.0724(7)	1.2298(4)	0.074(5)	0.085(4)	0.076(5)	0.004(4)	0.018(4)	0.005(3)
C(8)	4*e*	0.3678(6)	–0.0739(7)	1.1695(5)	0.056(4)	0.096(5)	0.103(6)	0.012(4)	0.021(4)	–0.005(4)
C(9)	4*e*	0.3396(7)	–0.0030(8)	1.0873(5)	0.073(5)	0.095(5)	0.093(5)	0.004(4)	0.039(4)	0.001(4)
C(10)	4*e*	0.2297(6)	0.0746(7)	1.0636(4)	0.053(4)	0.069(4)	0.077(4)	–0.005(3)	0.015(3)	–0.009(3)
C(11)	4*e*	–0.1658(6)	0.2430(8)	1.1343(5)	0.067(5)	0.116(6)	0.112(5)	0.024(5)	0.023(4)	–0.018(4)
C(12)	4*e*	0.1098(6)	–0.0743(7)	1.3478(4)	0.077(5)	0.084(4)	0.071(4)	0.003(4)	0.026(4)	0.004(3)
C(13)	4*e*	0.1933(7)	0.0198(8)	1.4177(4)	0.116(7)	0.112(6)	0.071(5)	0.010(5)	0.010(5)	0.017(4)
C(14)	4*e*	–0.0182(7)	–0.0983(8)	1.3739(4)	0.094(6)	0.136(6)	0.078(5)	–0.021(5)	0.040(4)	–0.004(4)
C(15)	4*e*	0.0478(7)	0.3056(8)	0.8655(4)	0.123(7)	0.118(5)	0.075(5)	–0.029(5)	0.031(5)	0.003(4)

References

1. Bringmann, G.; Pokorny, F.: The Naphthylisoquinoline Alkaloids. In: *The Alkaloids* (Ed. G. A. Cordell), 46, p. 127-271. Academic Press, New York 1995.
2. Bringmann, G.; Holenz, J.; Weirich, R.; Rübenacker, M.; Funke, C.; Boyd, M. R.; Gulakowski, R. J.; François, G.: First Synthesis of the Antimalarial Naphthylisoquinoline Alkaloid Dioncophylline C, and its Unnatural Anti-HIV Dimer, Jozimine C. Tetrahedron **54** (1998) 497-512.
3. Sheldrick, G. M.: Program Package SHELXTL-Plus. Release 4.1, Siemens Analytical X-Ray Instruments Inc., Madison (WI 53719), USA 1990.