Crystal structure of 2-bromo-1,4,5,7,8-pentamethoxynaphthalene, $C_{10}H_2Br(OCH_3)_5$

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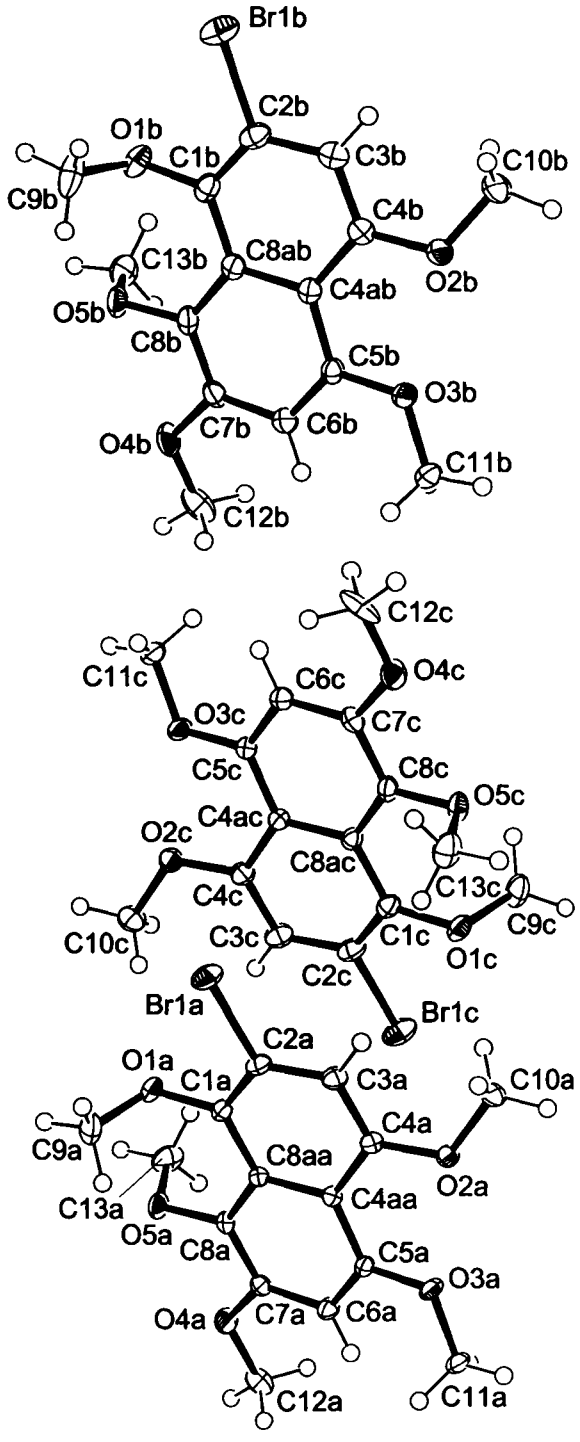
Received May 11, 2006, accepted and available on-line August 4, 2006; CCDC no. 1267/1784



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Abstract

$C_{15}H_{17}BrO_5$, monoclinic, $P12_1/n1$ (no. 14), a = 11.295(2) Å, b = 23.628(5) Å, c = 16.610(3) Å, β = 92.145(5)°, V = 4429.7 Å^3, Z = 12, $R_{gt}(F)$ = 0.038, $wR_{ref}(F^2)$ = 0.105, T = 173 K.

Source of material

The title compound was prepared by methylation of 2-bromo-5,7,8-trimethoxy-naphthalene-1,4-diol with dimethyl sulfate and aqueous sodium hydroxide solution (2.5 M) in dichloromethane and purified by chromatography on silica gel (hexane/ethyl acetate, 3:1) as described in [1]. Light brown crystals were obtained from chloroform (m.p. 372-373 K [2]).

Discussion

The crystal structure proves the constitution of the title compound particularly with regard to the position of the bromo substituent at C2. The crystal structure is built up by three crystallographically independent molecules whose naphthalene planes are arranged approximately perpendicular to the b axis. Equivalent bonding distances and angles are congruent within the range of their standard deviations and show no remarkable deviations from expected values. The methyl groups exhibit relatively large anisotropic displacement parameters due to their flexibility around the O—C ring axes. Bond distance corrections [3,4] of the differing O—CH_3 bond lengths yield acceptable congruent values.

Table 1. Data collection and handling.

Crystal:	colorless prism, size 0.10 × 0.25 × 0.53 mm
Wavelength:	Mo K_α radiation (0.71073 Å)
μ:	28.03 cm^{-1}
Diffractometer, scan mode:	Bruker SMART CCD, φ/ω
$2\theta_{max}$:	61.04°
$N(hkl)_{measured}$, $N(hkl)_{unique}$:	54835, 13518
Criterion for I_{obs}, $N(hkl)_{gt}$:	$I_{obs} > 2\,\sigma(I_{obs})$, 10264
$N(param)_{refined}$:	583
Programs:	SHELXS-97 [5], SHELXL-97 [6], ORTEP-3 [7]

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

Atom	Site	x	y	z	U_{iso}
H(4A)	4e	0.5190	0.0263	0.4018	0.021
H(11A)	4e	1.0471	0.0033	0.2534	0.020
H(1A)	4e	0.6012	−0.0773	0.1221	0.043
H(2A)	4e	0.5043	−0.0594	0.0541	0.043
H(3A)	4e	0.4634	−0.0787	0.1411	0.043

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(5A)	4e	0.6197	0.0899	0.4792	0.032
H(7A)	4e	0.7179	0.0737	0.5473	0.032
H(6A)	4e	0.6167	0.0291	0.5217	0.032
H(10A)	4e	1.0848	−0.0394	0.3697	0.032
H(8A)	4e	1.0875	−0.0087	0.4556	0.032
H(9A)	4e	1.1098	0.0271	0.3758	0.032
H(13A)	4e	1.1218	0.0400	0.1387	0.035
H(14A)	4e	1.1310	0.0084	0.0540	0.035
H(12A)	4e	1.1273	−0.0276	0.1355	0.035
H(17A)	4e	0.6673	0.0772	0.0760	0.044
H(16A)	4e	0.6950	0.0533	−0.0114	0.044
H(15A)	4e	0.8002	0.0777	0.0457	0.044
H(4B)	4e	0.5285	0.2111	0.9702	0.027
H(11B)	4e	1.0385	0.1376	0.8260	0.026
H(1B)	4e	0.5620	0.1188	0.6652	0.063
H(2B)	4e	0.4574	0.1528	0.6190	0.063
H(3B)	4e	0.4384	0.1283	0.7074	0.063
H(5B)	4e	0.6562	0.2598	1.0534	0.044
H(7B)	4e	0.7368	0.2295	1.1220	0.044
H(6B)	4e	0.6168	0.2001	1.0891	0.044
H(8B)	4e	1.0665	0.0969	0.9464	0.036
H(9B)	4e	1.0756	0.1316	1.0291	0.036
H(10B)	4e	1.1055	0.1620	0.9463	0.036

Table 2. Continued.

Atom	Site	x	y	z	U_{iso}
H(14B)	4e	1.1250	0.1804	0.7122	0.050
H(13B)	4e	1.1326	0.1470	0.6288	0.050
H(12B)	4e	1.1202	0.1127	0.7111	0.050
H(17B)	4e	0.7036	0.2534	0.6417	0.045
H(16B)	4e	0.7387	0.2275	0.5568	0.045
H(15B)	4e	0.8398	0.2423	0.6236	0.045
H(4C)	4e	0.4880	0.1424	0.1657	0.027
H(11C)	4e	−0.0363	0.1526	0.3218	0.022
H(2C)	4e	0.5286	0.2417	0.4226	0.043
H(3C)	4e	0.5278	0.2180	0.5129	0.043
H(1C)	4e	0.4055	0.2318	0.4652	0.043
H(5C)	4e	0.3860	0.1428	0.0459	0.041
H(7C)	4e	0.2915	0.0951	0.0200	0.041
H(6C)	4e	0.3945	0.0813	0.0858	0.041
H(9C)	4e	−0.1021	0.1285	0.2001	0.036
H(8C)	4e	−0.0822	0.1634	0.1192	0.036
H(10C)	4e	−0.0775	0.1951	0.2044	0.036
H(12C)	4e	−0.1127	0.1789	0.4426	0.045
H(14C)	4e	−0.1058	0.1448	0.5259	0.045
H(13C)	4e	−0.0980	0.1116	0.4423	0.045
H(16C)	4e	0.2113	0.0744	0.5169	0.043
H(17C)	4e	0.3164	0.0948	0.5774	0.043
H(15C)	4e	0.3448	0.0753	0.4879	0.043

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(1A)	4e	0.35252(2)	0.00159(1)	0.26995(2)	0.01208(9)	0.0340(1)	0.0315(1)	−0.00136(8)	0.00052(8)	0.00151(9)
O(1A)	4e	0.5316(1)	−0.00098(7)	0.13926(9)	0.0166(7)	0.0250(8)	0.0178(7)	−0.0005(6)	−0.0059(5)	−0.0018(6)
O(2A)	4e	0.7425(1)	0.03665(7)	0.44172(8)	0.0146(6)	0.0274(8)	0.0118(6)	0.0029(6)	0.0018(5)	−0.0017(6)
O(3A)	4e	0.9411(1)	0.00638(7)	0.38901(9)	0.0113(6)	0.0369(9)	0.0124(6)	0.0050(6)	−0.0014(5)	−0.0007(6)
O(4A)	4e	0.9736(1)	0.00424(7)	0.09991(9)	0.0180(7)	0.037(1)	0.0142(7)	0.0004(6)	0.0030(6)	−0.0039(6)
O(5A)	4e	0.7445(1)	0.00139(7)	0.07814(9)	0.0211(7)	0.0295(9)	0.0122(7)	−0.0005(6)	−0.0025(5)	−0.0028(6)
C(1A)	4e	0.5870(2)	0.00460(9)	0.2141(1)	0.0141(8)	0.0160(9)	0.0166(9)	−0.0003(7)	−0.0027(7)	0.0005(7)
C(2A)	4e	0.5195(2)	0.00933(9)	0.2807(1)	0.0114(8)	0.019(1)	0.022(1)	−0.0003(7)	0.0001(7)	0.0016(8)
C(3A)	4e	0.5689(2)	0.02041(9)	0.3578(1)	0.0141(9)	0.020(1)	0.019(1)	−0.0003(7)	0.0026(7)	0.0017(8)
C(4A)	4e	0.6901(2)	0.02262(9)	0.3691(1)	0.0145(8)	0.0150(9)	0.0138(9)	0.0001(7)	0.0010(7)	0.0023(7)
C(4AA)	4e	0.7670(2)	0.01236(8)	0.3034(1)	0.0132(8)	0.0135(9)	0.0133(8)	−0.0005(7)	0.0001(7)	−0.0001(7)
C(5A)	4e	0.8933(2)	0.00840(9)	0.3124(1)	0.0136(8)	0.017(1)	0.0122(8)	0.0008(7)	−0.0013(7)	−0.0009(7)
C(6A)	4e	0.9634(2)	0.00517(9)	0.2462(1)	0.0126(8)	0.024(1)	0.0141(9)	−0.0003(7)	0.0012(7)	0.0000(8)
C(7A)	4e	0.9108(2)	0.00457(9)	0.1682(1)	0.0162(9)	0.019(1)	0.0128(9)	0.0011(7)	0.0030(7)	−0.0018(7)
C(8A)	4e	0.7888(2)	0.00417(9)	0.1566(1)	0.0166(9)	0.0163(9)	0.0119(8)	−0.0005(7)	−0.0007(7)	−0.0016(7)
C(8AA)	4e	0.7140(2)	0.00682(8)	0.2235(1)	0.0135(8)	0.0135(9)	0.0137(8)	0.0012(7)	−0.0003(7)	−0.0004(7)
C(9A)	4e	0.5246(2)	−0.0587(1)	0.1120(2)	0.028(1)	0.030(1)	0.028(1)	−0.006(1)	−0.0040(9)	−0.010(1)
C(10A)	4e	0.6683(2)	0.0591(1)	0.5023(1)	0.0198(9)	0.030(1)	0.0149(9)	0.0049(8)	0.0048(7)	−0.0017(8)
C(11A)	4e	1.0657(2)	−0.0045(1)	0.3983(1)	0.0121(9)	0.034(1)	0.0178(9)	0.0051(8)	−0.0020(7)	−0.0024(9)
C(12A)	4e	1.0978(2)	0.0064(1)	0.1076(1)	0.028(1)	0.026(1)	0.017(1)	−0.0005(9)	0.0127(8)	0.0003(8)
C(13A)	4e	0.7252(2)	0.0568(1)	0.0444(1)	0.028(1)	0.041(2)	0.019(1)	0.007(1)	−0.0013(9)	0.010(1)
Br(1B)	4e	0.36160(2)	0.21437(1)	0.83479(2)	0.0196(1)	0.0372(2)	0.0467(2)	0.0012(1)	−0.0026(1)	0.0053(1)
O(1B)	4e	0.5398(2)	0.19702(8)	0.7061(1)	0.0271(8)	0.0318(9)	0.0253(8)	−0.0017(7)	−0.0088(7)	0.0054(7)
O(2B)	4e	0.7487(1)	0.19470(7)	1.01374(9)	0.0242(8)	0.0300(9)	0.0158(7)	0.0070(7)	0.0018(6)	−0.0006(6)
O(3B)	4e	0.9334(1)	0.15049(7)	0.96051(9)	0.0209(7)	0.0282(8)	0.0145(7)	0.0059(6)	0.0000(6)	0.0015(6)
O(4B)	4e	0.9763(2)	0.14912(8)	0.6719(1)	0.043(1)	0.039(1)	0.0138(7)	0.0046(8)	−0.0026(7)	−0.0049(7)
O(5B)	4e	0.7499(2)	0.17236(7)	0.64712(9)	0.0334(9)	0.0265(9)	0.0151(7)	0.0006(7)	−0.0065(6)	−0.0030(6)
C(1B)	4e	0.5931(2)	0.19125(9)	0.7815(1)	0.023(1)	0.017(1)	0.023(1)	−0.0014(8)	−0.0056(8)	0.0010(8)
C(2B)	4e	0.5272(2)	0.2007(1)	0.8475(2)	0.0184(9)	0.019(1)	0.032(1)	−0.0007(8)	−0.0024(8)	0.0028(9)
C(3B)	4e	0.5765(2)	0.2021(1)	0.9263(1)	0.022(1)	0.019(1)	0.026(1)	−0.0001(8)	0.0039(8)	0.0014(9)
C(4B)	4e	0.6945(2)	0.19028(9)	0.9393(1)	0.022(1)	0.016(1)	0.018(1)	0.0002(8)	0.0019(8)	0.0023(8)
C(4AB)	4e	0.7679(2)	0.17475(9)	0.8733(1)	0.0201(9)	0.0140(9)	0.0167(9)	−0.0008(7)	−0.0009(7)	0.0007(7)
C(5B)	4e	0.8893(2)	0.15779(9)	0.8838(1)	0.0208(9)	0.017(1)	0.0148(9)	−0.0002(7)	−0.0009(7)	0.0008(7)
C(6B)	4e	0.9581(2)	0.1487(1)	0.8181(1)	0.022(1)	0.024(1)	0.019(1)	0.0036(8)	0.0003(8)	−0.0006(8)
C(7B)	4e	0.9097(2)	0.1559(1)	0.7398(1)	0.027(1)	0.021(1)	0.016(1)	0.0024(8)	0.0022(8)	−0.0033(8)
C(8B)	4e	0.7913(2)	0.16908(9)	0.7265(1)	0.028(1)	0.018(1)	0.0154(9)	−0.0011(8)	−0.0035(8)	−0.0022(8)
C(8AB)	4e	0.7172(2)	0.17802(9)	0.7925(1)	0.022(1)	0.0131(9)	0.020(1)	−0.0003(7)	−0.0021(8)	0.0001(8)
C(9B)	4e	0.4959(3)	0.1451(1)	0.6717(2)	0.046(2)	0.052(2)	0.028(1)	−0.022(1)	−0.013(1)	−0.001(1)
C(10B)	4e	0.6846(2)	0.2233(1)	1.0743(1)	0.031(1)	0.040(2)	0.017(1)	0.008(1)	0.0062(9)	0.000(1)

Table 3. Continued.

Atom	Site	x	y	z	U_{11}	U_{22}	U_{33}	U_{12}	U_{13}	U_{23}
C(11B)	4e	1.0548(2)	0.1340(1)	0.9714(1)	0.022(1)	0.031(1)	0.019(1)	0.0083(9)	−0.0028(8)	0.0008(9)
C(12B)	4e	1.0971(3)	0.1471(1)	0.6817(2)	0.048(2)	0.028(1)	0.025(1)	−0.001(1)	0.019(1)	−0.000(1)
C(13B)	4e	0.7587(2)	0.2283(1)	0.6147(2)	0.037(1)	0.034(1)	0.019(1)	0.003(1)	−0.0024(9)	0.005(1)
Br(1C)	4e	0.65635(2)	0.16509(1)	0.29650(2)	0.0145(1)	0.0388(2)	0.0393(1)	−0.00483(9)	−0.00099(9)	0.0073(1)
O(1C)	4e	0.4822(1)	0.16053(7)	0.4297(1)	0.0209(7)	0.0195(8)	0.0251(8)	0.0006(6)	−0.0097(6)	0.0019(6)
O(2C)	4e	0.2649(1)	0.12880(7)	0.12772(9)	0.0158(7)	0.0334(9)	0.0157(7)	0.0001(6)	0.0033(5)	−0.0047(6)
O(3C)	4e	0.0655(1)	0.14937(8)	0.18444(9)	0.0139(7)	0.039(1)	0.0135(7)	0.0045(6)	−0.0003(5)	−0.0032(6)
O(4C)	4e	0.0421(1)	0.15245(8)	0.47444(9)	0.0243(8)	0.038(1)	0.0160(7)	−0.0036(7)	0.0029(6)	−0.0035(7)
O(5C)	4e	0.2712(1)	0.15136(7)	0.49344(9)	0.0272(8)	0.0198(8)	0.0150(7)	−0.0019(6)	−0.0058(6)	−0.0010(6)
C(1C)	4e	0.4242(2)	0.15645(9)	0.3551(1)	0.0180(9)	0.0134(9)	0.023(1)	0.0003(7)	−0.0052(8)	0.0008(8)
C(2C)	4e	0.4894(2)	0.1549(1)	0.2879(1)	0.0138(9)	0.021(1)	0.030(1)	−0.0024(8)	−0.0013(8)	0.0031(9)
C(3C)	4e	0.4389(2)	0.1456(1)	0.2107(1)	0.0160(9)	0.025(1)	0.026(1)	−0.0022(8)	0.0026(8)	0.0029(9)
C(4C)	4e	0.3182(2)	0.14120(9)	0.2006(1)	0.0167(9)	0.019(1)	0.019(1)	−0.0012(7)	0.0007(7)	0.0007(8)
C(4AC)	4e	0.2424(2)	0.14795(9)	0.2679(1)	0.0145(8)	0.0158(9)	0.0158(9)	0.0006(7)	−0.0012(7)	−0.0007(7)
C(5C)	4e	0.1157(2)	0.14933(9)	0.2606(1)	0.0159(9)	0.018(1)	0.0148(9)	0.0007(7)	−0.0010(7)	−0.0019(7)
C(6C)	4e	0.0477(2)	0.15143(9)	0.3279(1)	0.0156(9)	0.022(1)	0.0163(9)	0.0014(7)	0.0005(7)	−0.0013(8)
C(7C)	4e	0.1020(2)	0.15188(9)	0.4051(1)	0.0209(9)	0.017(1)	0.0148(9)	−0.0006(7)	0.0031(7)	−0.0014(7)
C(8C)	4e	0.2243(2)	0.15225(9)	0.4149(1)	0.0215(9)	0.016(1)	0.0144(9)	0.0004(7)	−0.0039(7)	−0.0009(7)
C(8AC)	4e	0.2971(2)	0.15227(9)	0.3473(1)	0.0170(9)	0.0134(9)	0.0186(9)	0.0007(7)	−0.0040(7)	−0.0005(7)
C(9C)	4e	0.4864(2)	0.2176(1)	0.4600(2)	0.032(1)	0.023(1)	0.030(1)	−0.0064(9)	−0.010(1)	−0.0031(9)
C(10C)	4e	0.3402(2)	0.1106(1)	0.0649(1)	0.022(1)	0.042(2)	0.019(1)	0.003(1)	0.0072(8)	−0.004(1)
C(11C)	4e	−0.0588(2)	0.1599(1)	0.1764(1)	0.0153(9)	0.038(1)	0.019(1)	0.0049(9)	−0.0027(8)	−0.0055(9)
C(12C)	4e	−0.0767(2)	0.1465(1)	0.4711(1)	0.049(2)	0.022(1)	0.021(1)	0.007(1)	0.030(1)	0.0045(9)
C(13C)	4e	0.2872(2)	0.0945(1)	0.5211(1)	0.037(1)	0.024(1)	0.023(1)	−0.000(1)	−0.0083(9)	0.0071(9)

Acknowledgments. Support of this work by the Deutsche Forschungsgemeinschaft, the Fonds der Chemischen Industrie (Kekule fellowship for SS) and the Schering AG is most gratefully acknowledged.

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