Data collection

Enraf–Nonius CAD-4 diffractometer	2300 reflections with $I > 2.5\sigma(I)$
$\omega/2\theta$ scans	$R_{int} = 0.028$
Absorption correction: empirical ψ scan (North, Phillips & Mathews, 1968)	$\theta_{max} = 24.97°$
	$h = 0 \rightarrow 12$
	$k = 0 \rightarrow 17$
$T_{min} = 0.046$, $T_{max} = 0.069$	$l = -13 \rightarrow 12$
3202 measured reflections	3 standard reflections
3187 independent reflections	every 200 reflections
	intensity decay: none

Refinement

Refinement on F	$(\Delta/\sigma)_{max} = 0.0034$
$R = 0.053$	$\Delta\rho_{max} = 1.41$ e Å^{-3}
$wR = 0.061$	$\Delta\rho_{min} = -0.69$ e Å^{-3}
$S = 1.905$	Extinction correction: none
2300 reflections	Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)
208 parameters	
H atoms not refined	
Weighting scheme based on measured s.u.'s; $w = 1/[\sigma_c^2(F_o) + (p^2/4F_o^2)]$, with $p = 0.03$	

Table 1. *Selected geometric parameters (Å, °)*

Br(1)—C(8)	1.972 (5)	N(1)—C(1)	1.306 (7)
Br(2)—C(9)	2.007 (6)	N(1)—C(7)	1.385 (7)
Cl—C(11)	1.736 (5)	C(1)—C(8)	1.492 (7)
S—C(1)	1.729 (5)	C(8)—C(9)	1.476 (8)
S—C(2)	1.711 (5)	C(9)—C(10)	1.504 (7)
C(1)—S—C(2)	89.5 (3)	Br(1)—C(8)—C(1)	110.4 (4)
C(1)—N(1)—C(7)	109.9 (4)	Br(1)—C(8)—C(9)	106.2 (4)
S—C(1)—N(1)	116.2 (4)	C(1)—C(8)—C(9)	113.2 (5)
S—C(1)—C(8)	120.4 (4)	Br(2)—C(9)—C(8)	107.0 (4)
N(1)—C(1)—C(8)	123.3 (5)	Br(2)—C(9)—C(10)	106.7 (4)
S—C(2)—C(3)	129.2 (5)	C(8)—C(9)—C(10)	117.0 (5)
S—C(2)—C(7)	109.4 (4)	C(9)—C(10)—C(11)	122.2 (5)
N(1)—C(7)—C(2)	114.9 (5)	C(9)—C(10)—C(15)	120.5 (4)
N(1)—C(7)—C(6)	125.2 (5)		

The largest peak in the final difference Fourier map (1.41 e Å^{-3}) is 0.39 Å from the Br(2) atom.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *CAD-4-PC Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

This work was supported by the National Science Foundation (OSR-9452893 and RII-8610677) and the National Institutes of Health (MBRS5S06-GM08224).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1024). Services for accessing these data are described at the back of the journal.

References

Alegría, A. E., Cox, O., Santiago, V., Colón, M., Reyes, Z., Rivera, L. A. & Dumas, J. A. (1993). *Free Rad. Biol. Med.* **15**, 49–56.

Altomare, A., Cascarano, M., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.

Cox, O., Jackson, H., Vargas, V. A., Báez, A., Colón, J. I., González, B. C. & De Leon, M. (1982). *J. Med. Chem.* **25**, 1378–1380.

Enraf–Nonius (1992). *CAD-4-PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.

Gòmez, G. M., Muir, M. M., Muir, J. A. & Cox, O. (1988). *Acta Cryst.* C**44**, 1554–1557.

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Molecular Structure Corporation (1985, 1992). *TEXSAN. Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX, 77381, USA.

Muir, J. A., Cox, O., Bernard, L. & Muir, M. M. (1992*a*). *Acta Cryst.* C**48**, 1677–1679.

Muir, J. A., Cox, O., Bernard, L. & Muir, M. M. (1992*b*). *J. Crystallogr. Spectrosc. Res.* **22**, 695–697.

Muir, M. M., Cox, O., Bernard, L. & Muir, J. A. (1992*c*). *Acta Cryst.* C**48**, 583–585.

Muir, M. M., Cox, O., Bernard, L. & Muir, J. A. (1992*d*). *J. Crystallogr. Spectrosc. Res.* **22**, 271–273.

Muir, J. A., Cox, O., Bernard, L. & Muir, M. M. (1993). *J. Crystallogr. Spectrosc. Res.* **23**, 499–502.

Muir, M. M., Cox, O., Rivera, L. A., Càdiz, M. E. & Medina, E. (1992). *Inorg. Chim. Acta*, **191**, 131–139.

Muir, M. M., Gòmez, G. M., Muir, J. A., Càdiz, M. E., Cox, O. & Barnes, C. L. (1988). *Acta Cryst.* C**44**, 803–806.

Muir, J. A., Gòmez, G. M., Muir, M. M., Cox, O. & Càdiz, M. E. (1987). *Acta Cryst.* C**43**, 1258–1261.

North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

Acta Cryst. (1998). C**54**, 69–71

N-(*o*-Tolyl)-2-bromo-4,5-dimethoxybenzamide†

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Abstract

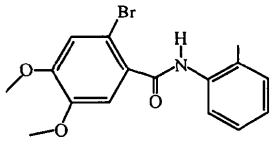
The molecular structure of the title compound, $C_{16}H_{16}BrNO_3$, consists of 2-bromo-4,5-dimethoxybenzene and *o*-methylphenyl rings linked by an amide group. The two methoxy groups are almost coplanar with the phenyl ring.

Comment

A number of antipsychotic agents of the *o*-methoxybenzamide type show interesting selectivity for dopamine–D2 receptors (Hogberg, 1991). Benzamides with an *N*-(*cis*-2-phenylcycloheptyl) or an *N*-ethyl-2-(pyrrolidine)methyl side chain and a series of

† Alternative name: 2-bromo-4,5-dimethoxy-2′-methylbenzanilide.

5-substituted dimethoxybenzamides also display a high stereoselective affinity for dopamine–D2 receptors (Hogberg *et al.*, 1987; Hogberg, 1991). During our studies on the organic synthesis of new antipsychotic drugs, we obtained the title compound, (I), and in order to evaluate its bioactivity on the basis of structure–property relationships, we determined its X-ray crystal structure.



(I)

An *ORTEP*II (Johnson, 1976) representation of the title compound is presented in Fig. 1. The structure consists of 2-bromo-4,5-dimethoxybenzene and *o*-methylphenyl rings linked by an amide group. The dihedral angle between the two aromatic rings is 74.3 (8)°. The two methoxy groups are almost coplanar with the phenyl ring [torsion angles C(3)—C(4)—O(1)—C(7) −6.6 (6) and C(6)—C(5)—O(2)—C(8) 6.8 (5)°]. All the other bond distances and angles are in the normal ranges.

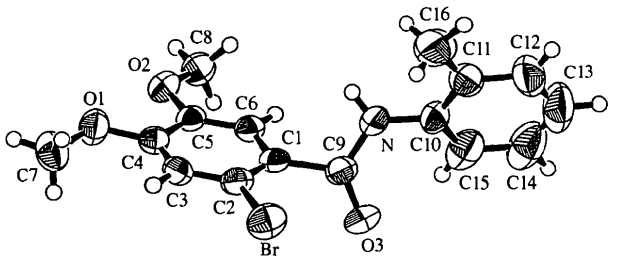


Fig. 1. An *ORTEP*II (Johnson, 1976) representation of the title compound showing 50% probability displacement ellipsoids.

Experimental

The title compound was synthesized by the reaction of bromoveratric acid chloride with *o*-toluidine. The crude product was recrystallized from acetone to give white single crystals (m.p. 462–464 K) of X-ray quality.

Crystal data

$C_{16}H_{16}BrNO_3$
M_r = 350.21
Monoclinic
$P2_1/n$
a = 9.022 (3) Å
b = 11.615 (4) Å
c = 14.787 (6) Å
β = 99.44 (4)°
V = 1528.6 (9) Å^3
Z = 4
D_x = 1.522 Mg m^{-3}
D_m not measured
Mo $K\alpha$ radiation
λ = 0.7107 Å
Cell parameters from 25 reflections
θ = 14–16°
μ = 2.706 mm^{-1}
T = 295 (1) K
Prism
0.40 × 0.26 × 0.22 mm
Colorless

Data collection

Enraf–Nonius CAD-4 diffractometer
$\omega/2\theta$ scans
Absorption correction: empirical ψ scan (North, Phillips & Mathews, 1968)
T_{min} = 0.379, T_{max} = 0.551
3048 measured reflections
3031 independent reflections
2146 reflections with $I > 2.5\sigma(I)$
R_{int} = 0.021
θ_{max} = 24.97°
h = 0 → 10
k = 0 → 13
l = −17 → 17
3 standard reflections
every 200 reflections
intensity decay: none

Refinement

Refinement on F
R = 0.069
wR = 0.072
S = 2.425
2146 reflections
190 parameters
H atoms not refined
Weighting scheme based on measured s.u.'s; $w = 1/[\sigma^2(F_{obs} + 0.0026|F_{obs}|^2]$
$(\Delta/\sigma)_{max}$ = 0.0212
$\Delta\rho_{max}$ = 1.28 e Å^{-3}
$\Delta\rho_{min}$ = −0.54 e Å^{-3}
Extinction correction: none
Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. *Selected geometric parameters (Å, °)*

Br—C(2)	1.900 (4)	O(2)—C(8)	1.410 (6)
O(1)—C(4)	1.358 (5)	O(3)—C(9)	1.209 (5)
O(1)—C(7)	1.413 (6)	N—C(9)	1.347 (5)
O(2)—C(5)	1.370 (5)	N—C(10)	1.410 (6)
C(4)—O(1)—C(7)	117.9 (4)	O(2)—C(5)—C(4)	115.4 (4)
C(5)—O(2)—C(8)	116.9 (3)	O(2)—C(5)—C(6)	124.8 (4)
C(9)—N—C(10)	125.2 (4)	O(3)—C(9)—N	124.9 (4)
Br—C(2)—C(1)	121.0 (3)	O(3)—C(9)—C(1)	122.2 (4)
Br—C(2)—C(3)	117.1 (3)	N—C(9)—C(1)	113.0 (3)
O(1)—C(4)—C(3)	125.8 (4)	N—C(10)—C(11)	117.9 (4)
O(1)—C(4)—C(5)	114.6 (4)	N—C(10)—C(15)	120.4 (5)
C(3)—C(4)—C(5)	119.6 (4)		

The largest peak in the final difference Fourier map (1.28 e Å^{-3}) is 0.47 Å from the Br atom.

Data collection: *CAD-4-PC Software* (Enraf–Nonius, 1992). Cell refinement: *CAD-4-PC Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1985, 1992). Program(s) used to solve structure: *SIR*92 (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

This work was supported by the National Science Foundation (OSR-9452893 and RII-8610677) and the National Institutes of Health (MBRS5S06-GM08224).

Supplementary data for this paper are available from the IUCr electronic archives (Reference: SX1023). Services for accessing these data are described at the back of the journal.

References

Altomare, A., Cascarano, M., Giacovazzo, C. & Guagliardi, A. (1993). *J. Appl. Cryst.* **26**, 343–350.
Enraf–Nonius (1992). *CAD-4-PC Software*. Version 1.1. Enraf–Nonius, Delft, The Netherlands.
Hogberg, T. (1991). *Drugs Future*, **16**, 333–357.

Hogberg, T., Ramsby, S., Ogren, S.-O. & Norinder, U. (1987). *Acta Pharm. Suec.* **24**, 289–328.
Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5183. Oak Ridge National Laboratory, Tennessee, USA.
Molecular Structure Corporation (1985, 1992). *TEXSAN. Crystal Structure Analysis Package*. MSC, 3200 Research Forest Drive, The Woodlands, TX, 77381, USA.
North, A. C. T., Phillips, D. C. & Mathews, F. S. (1968). *Acta Cryst.* A**24**, 351–359.

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A 2-Alkyl Substituted 2,3,1-Benzodiazaborine

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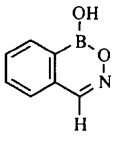
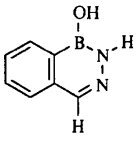
Abstract

The structure of the fourth member of a family of structurally related 2,3,1-benzodiheteroborines has been solved by crystallographic means, providing data for proper direct comparison with the others. The title compound, 1,2-dihydro-1-hydroxy-2-methyl-2,3,1-benzodiazaborine ($C_8H_9BN_2O$) is obtained *via* condensation of 2-formylbenzeneboronic acid and methylhydrazine, and is similar in most respects to its 2-unsubstituted parent, in internal geometry and intramolecular association topography, but has some subtle oxazaborine-like characteristics.

Comment

Although it has been known for quite some time that condensation of 2-formylbenzeneboronic acid with hydroxylamines or hydrazines affords 2,3,1-benzoxaza- or benzodiazaborines, respectively (Tschampel & Snyder, 1964; Dewar & Dougherty, 1964), these and related boron heterocycles have until recently received little attention. We are interested in them as they are quite robust and potentially useful as platforms for the construction of new biologically active compounds (see, for example, Groziak *et al.*, 1994). To develop the 2,3,1-benzodiheteroborines further, we are undertaking a systematic physicochemical study of heterocycles like compounds (1)–(5) by crystallographic and other means.


(1)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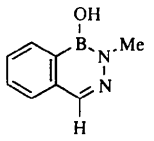
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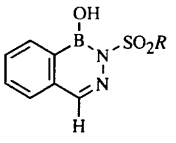
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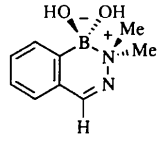
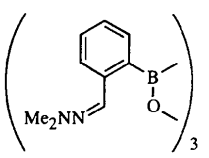

(5a)


(5b)

The parent oxaza- and diazaborines, (1) and (2), respectively, were the subjects of our most recent report (Groziak *et al.*, 1997), which among other determinations reported their crystal structures. The structure of the title compound (3), which is the 2-methyl-substituted version of (2), is presented here, while that of a representative 2-alkyl- or arylsulfonylated version, (4), is currently being sought. Although a crystal structure determination of a thiophene analog of (3), 7-hydroxy-6-methyl-7,6-borazarothieno[3,2-*c*]-pyridine, has been reported (Aurivillius & Löefving, 1974), we solved the structure of (3) in order to compare it directly with (1) and (2) without the need to allow for differences in ring-fusion identity. Interestingly, the thiophene analog reportedly decomposed in air of normal humidity so rapidly that the crystal for analysis had to be kept over a saturated aqueous solution of the substance. No such unusual lability was noted for our crystalline (3).

The 2,3,1-benzodiazaborines in particular can exhibit a wide diversity of structures. Some of the known biocidal (Grassberger *et al.*, 1984) heterocycles, (4), have been found by crystallography to form a tetrahedral borate complex in the active site of enoyl reductase (Baldock *et al.*, 1996) and the 2-dimethyl-substituted 2,3,1-benzodiazaborine, (5*a*), exists as a zwitterion, but only in water. An unusual double intramolecular chelate form of triarylboroxine (5*b*) was found to be its structure in the solid state (Robinson *et al.*, 1996).

An *ORTEP* (Johnson, 1965) view of the molecule of crystalline (3), together with its atom numbering, is given in Fig. 1. This molecule is a true B—N for C=C replacement analog of the enol form of 3-methyl-3*H*-isoquinolin-4-one, but to our knowledge no simple 3-alkyl-4-hydroxyisoquinoline has yet been studied crystallographically. Instead, the closest nitrogen heterocycle reference compounds for (3) appear to be several 3,4-dihydro-4-oxo-3-(arylmethyl)-1-phthalazineacetic acids (Mylari *et al.*, 1991) and a 4-(*p*-chlorobenzyl)-2-(alkyl)-1(2*H*)-phthalazinone monohydrate (Scheffler *et al.*, 1988). Features associated with the benzene ring and methyl substituent of (3) are

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