

2200 reflections  
223 parameters  
All H-atom parameters  
refined  
 $w = 1/[\sigma^2(F_o^2) + (0.0428P)^2 + 0.2855P]$   
where  $P = (F_o^2 + 2F_c^2)/3$

Scattering factors from  
*International Tables for  
X-ray Crystallography*  
(Vol. C, Tables 4.2.6.8 and  
6.1.1.4)

Table 1. Selected geometric parameters (Å, °)

N1—C9	1.362 (6)	C4—C5	1.393 (8)
N1—C10	1.403 (6)	C5—C6	1.385 (6)
O1—C9	1.227 (6)	C6—C7	1.522 (6)
O2—C12	1.323 (6)	C7—C8	1.524 (7)
O2—C13	1.445 (7)	C7—C11	1.522 (6)
O3—C12	1.189 (6)	C8—C9	1.493 (7)
C1—C2	1.380 (8)	C10—C11	1.339 (6)
C1—C6	1.384 (7)	C10—C14	1.491 (7)
C2—C3	1.366 (8)	C11—C12	1.469 (6)
C3—C4	1.34 (1)		
C9—N1—C10	125.5 (4)	C7—C8—C9	114.7 (4)
C12—O2—C13	116.6 (4)	O1—C9—C8	124.1 (4)
C2—C1—C6	121.7 (5)	N1—C9—C8	115.2 (4)
C1—C2—C3	120.0 (5)	N1—C9—O1	120.7 (4)
C2—C3—C4	119.3 (6)	N1—C10—C14	111.9 (4)
C3—C4—C5	121.9 (6)	N1—C10—C11	119.4 (4)
C4—C5—C6	119.7 (4)	C11—C10—C14	128.7 (4)
C1—C6—C5	117.5 (4)	C7—C11—C10	120.4 (4)
C5—C6—C7	120.8 (4)	C10—C11—C12	121.0 (4)
C1—C6—C7	121.7 (4)	C7—C11—C12	118.6 (4)
C6—C7—C11	112.8 (3)	O3—C12—C11	127.0 (4)
C6—C7—C8	111.8 (4)	O2—C12—C11	112.0 (4)
C8—C7—C11	110.0 (4)	O2—C12—O3	121.0 (4)

Table 2. Hydrogen-bonding geometry (Å, °)

D—H...A	D—H	H...A	D...A	D—H...A
N1—H...O1 <sup>i</sup>	0.86 (4)	2.05 (4)	2.903 (5)	179 (4)

Symmetry code: (i)  $-x, 1 - y, 1 - z$ .

The structure was solved by direct methods

Data collection: *CAD-4 Software* (Enraf–Nonius, 1992). Cell refinement: *CRYSDA (DIRDIF; Beurskens et al., 1992)*. Data reduction: *REFLEX* (local program). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1990). Program(s) used to refine structure: *SHELXL93* (Sheldrick, 1993). Molecular graphics: *SHELXTL-Plus* (Sheldrick, 1991). Software used to prepare material for publication: *PARST* (Nardelli, 1983*b*); *PARSTCIF* (Nardelli, 1992).

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: BS1040). Services for accessing these data are described at the back of the journal.

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## *N*-Borane-*N*-(trimethylsilyl)morpholine

RAMON HUERTAS, JESUS R. MEDINA, JOHN A. SODERQUIST AND SONGPING D. HUANG

*Department of Chemistry, University of Puerto Rico, PO Box 23346, San Juan, PR 00931, USA. E-mail: huang@zintl.chem.uprr.pr*

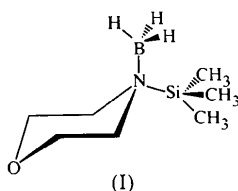
(Received 2 February 1998; accepted 27 May 1998)

## Abstract

The title compound, C<sub>7</sub>H<sub>20</sub>BNOSi, features an *N,N*-disubstituted six-membered morpholine ring in a chair conformation, with the trimethylsilyl group in the equatorial position and the borane group in the axial position. The least-squares plane formed by the four C atoms of the morpholine ring has a mean deviation of 0.013 (2) Å. The O and N atoms are 0.672 (2) and 0.650 (2) Å above and below the plane, respectively.

## Comment

Borane–THF and borane dimethyl sulfide (BMS) are the most frequently used laboratory borane sources for hydroboration and other borane conversions (Brown, 1975; Soderquist, 1994). Both are safe and more convenient to handle than gaseous diborane. However, there are disadvantages of the use of either borane–THF or BMS as a borane source. For instance, concentrated solutions (>2*M*) of borane–THF are unstable and can lose diborane. Moreover, the dimethyl sulfide complex is a foul-smelling liquid and dimethyl sulfide can contaminate the reaction solvents making their recovery difficult. As part of our efforts at developing new hydroborating agents, we carried out a reaction between gaseous diborane and *N*-(trimethylsilyl)morpholine in diethyl ether at 233 K yielding the title compound, (I), as the only product. In principle, two conformational isomers may exist with either one of the two different *N*-substituents occupying the axial or equatorial positions.



The <sup>1</sup>H, <sup>13</sup>C and <sup>11</sup>B NMR studies in solution indicated that the reaction product is a single conformer of borane- and trimethylsilyl-containing morpholine. In order to confirm the identity and to study the conformation of this compound, we carried out a single-crystal X-ray structure analysis. Selected bond distances and bond angles of the title compound are given in Table 1. Fig. 1 is an *ORTEP* (Johnson, 1976) representation of its molecular structure. The conformation of the six-membered morpholine ring is almost a perfect chair with the borane group occupying the axial position and the trimethylsilyl group occupying the equatorial position. This conformer is evidently energetically favoured because vicinal repulsions between the ring and the bulkier —Si(CH<sub>3</sub>)<sub>3</sub> group are reduced when it occupies the equatorial position. The bond distances of C(1)—C(2), C(3)—C(4), O—C(2), O—C(3), N—C(1) and N—C(4) in the morpholine ring all signify single bonds, while the N—B and N—Si bond distances are both in the normal range of single N—B and N—Si bonds. All non-hydrogen elements except the O atom have tetrahedral geometry, with the bond angles ranging from 107.3 (1)° to 112.6 (1)°.

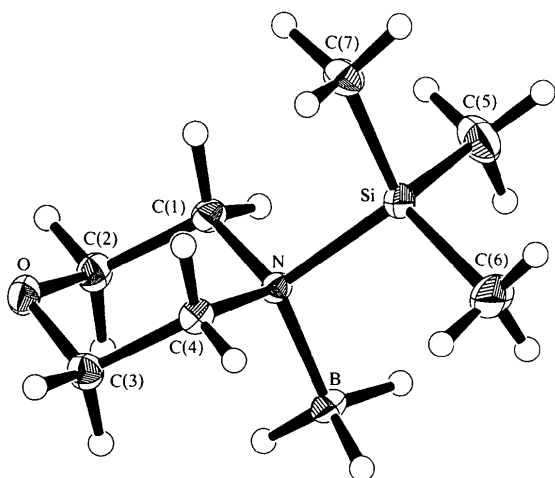


Fig. 1. *ORTEP* representation of the title compound showing 50% probability displacement ellipsoids.

## Experimental

Crystals of X-ray diffraction quality were obtained by vacuum sublimation. Gaseous diborane was reacted with *N*-trimethylsilylmorpholine in diethyl ether at 233 K. The title compound was isolated in ~84% yield.

## Crystal data

C<sub>7</sub>H<sub>20</sub>BNOSi  
*M<sub>r</sub>* = 173.14  
 Monoclinic  
*P*2<sub>1</sub>/*c*  
*a* = 8.4110 (7) Å  
*b* = 11.8372 (9) Å  
*c* = 11.1004 (9) Å  
 $\beta$  = 100.066 (1)°  
*V* = 1088.2 (1) Å<sup>3</sup>  
*Z* = 4  
*D<sub>s</sub>* = 1.057 Mg m<sup>-3</sup>  
*D<sub>m</sub>* not measured

## Data collection

Bruker CCD diffractometer  
 $\omega$  scans  
 Absorption correction: none  
 5749 measured reflections  
 2291 independent reflections  
 1637 reflections with  
 $I > 3.00\sigma(I)$

## Refinement

Refinement on *F*  
*R* = 0.032  
*wR* = 0.050  
*S* = 1.17  
 1637 reflections  
 101 parameters  
 H-atom parameters not refined  
 $w = 1/[\sigma^2(F_o) + 0.00102|F_o|^2]$

Mo *K*α radiation  
 $\lambda$  = 0.7107 Å  
 Cell parameters from 1171 reflections  
 $\theta$  = 1.34–27.06°  
 $\mu$  = 0.170 mm<sup>-1</sup>  
*T* = 173 (1) K  
 Block  
 0.26 × 0.14 × 0.10 mm  
 Colourless

*R*<sub>int</sub> = 0.016  
 $\theta_{\max}$  = 27.06°  
*h* = 0 → 10  
*k* = -15 → 15  
*l* = -14 → 13  
 Intensity decay: none

( $\Delta/\sigma$ )<sub>max</sub> = 0.0028  
 $\Delta\rho_{\max}$  = 0.22 e Å<sup>-3</sup>  
 $\Delta\rho_{\min}$  = -0.24 e Å<sup>-3</sup>  
 Extinction correction:  
 Zachariasen (1967)  
 Extinction coefficient:  
 0.0000017 (5)  
 Scattering factors from  
*International Tables for Crystallography* (Vol. C)

Table 1. Selected geometric parameters (Å, °)

Si—N	1.839 (2)	N—C(1)	1.512 (2)
Si—C(5)	1.852 (2)	N—C(4)	1.507 (2)
Si—C(6)	1.844 (2)	N—B	1.639 (2)
Si—C(7)	1.859 (2)	C(1)—C(2)	1.519 (2)
O—C(2)	1.432 (2)	C(3)—C(4)	1.509 (2)
O—C(3)	1.427 (2)		
N—Si—C(5)	108.73 (8)	C(1)—N—C(4)	107.3 (1)
N—Si—C(6)	108.64 (8)	C(1)—N—B	111.7 (1)
C(5)—Si—C(6)	112.6 (1)	N—C(1)—C(2)	112.5 (1)
C(2)—O—C(3)	109.4 (1)	O—C(2)—C(1)	111.7 (1)
Si—N—C(1)	109.3 (1)	N—C(4)—C(3)	112.2 (1)
Si—N—B	107.4 (1)		

H atoms were either located from the difference Fourier maps or calculated and included in the structure model, but not refined (fixed).

Data collection: *SMART* (Siemens, 1994a). Cell refinement: *SAINT* (Siemens, 1994b). Data reduction: *SAINT*. Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN* (Molecular Structure Corporation, 1995). Software used to prepare material for publication: *TEXSAN*.

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Supplementary data for this paper are available from the IUCr electronic archives (Reference: FR1124). Services for accessing these data are described at the back of the journal.

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## *o*-Phenylenediamine Pyrocatechol Hemihydrate Complex

SHYAMAPROSAD GOSWAMI,<sup>a</sup> RESHMI MUKHERJEE,<sup>b</sup> GUR DAYAL NIGAM,<sup>c</sup> KANDASAMY CHINNAKALI<sup>d†</sup> AND HOONG-KUN FUN<sup>d</sup>

<sup>a</sup>Department of Chemistry, Bengal Engineering College, Botanic Garden, Howrah 711103, India, <sup>b</sup>Department of Chemistry, Indian Institute of Technology, Kharagpur 721302, West Bengal, India, <sup>c</sup>Department of Physics and Meteorology, Indian Institute of Technology, Kharagpur 721302, West Bengal, India, and <sup>d</sup>X-ray Crystallography Unit, School of Physics, Universiti Sains Malaysia, 11800 USM, Penang, Malaysia. E-mail: hkfun@usm.my

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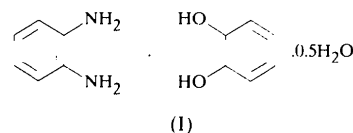
## Abstract

A well defined supramolecular structure, C<sub>6</sub>H<sub>8</sub>N<sub>2</sub>·C<sub>6</sub>H<sub>6</sub>O<sub>2</sub>·0.5H<sub>2</sub>O, is generated by molecular recognition between pyrocatechol and *o*-phenylenediamine in the presence of water. They spontaneously self assemble

through a unique network of hydrogen bonds around a twofold axis.

## Comment

The development of solid state structures which are able to exhibit new physical and chemical properties (Hanesian *et al.*, 1994) is the basis of crystal engineering. The self-assembling complementarity of diamine/diol motifs provides convincing evidence that the engineering of highly organized supramolecular structures based on molecular recognition can now be extended to include 'weak' hydrogen-bonding interactions. The X-ray structure analysis of the title compound, (I), supports this viewpoint.



The asymmetric unit consists of one molecule each of *o*-phenylenediamine, pyrocatechol and water. In the crystal, the water molecules lie on the twofold axis and they are linked to the pyrocatechol molecules through an O2—H···O3<sup>iii</sup> hydrogen bond (Table 3 and Fig. 2). The diamine molecules are connected to the water molecules by O3—H···N2<sup>iv</sup> hydrogen bonds between O3 and N2<sup>iv</sup>. The diol and diamine molecules themselves are involved in N—H···O hydrogen bonds (N2···O1 and N1···O2<sup>1</sup>). This hydrogen-bonded network forms a supramolecular structure around the twofold axis. The neighbouring molecular assemblies are interlinked by O1—H1O1···N1<sup>ii</sup> hydrogen bonds. Due to the hydrogen bonding, both the N atoms and water O atoms exhibit full tetra-coordination. The hydrogen-bonded assembly in this structure is significantly different from the supramolecular helicate structures observed for the 1:1 complex of *trans*-1,2-diaminocyclohexane and (1*S*,2*S*)-*trans*-1,2-cyclohexanediol (Hanesian *et al.*, 1994), which may be due to the presence of water molecules. The C—N, C—O and C—C lengths observed in this structure agree with the reported values (Allen *et al.*, 1987). The dihedral angle between the mean planes through the diamine and diol moieties is 25.87 (3)°. The

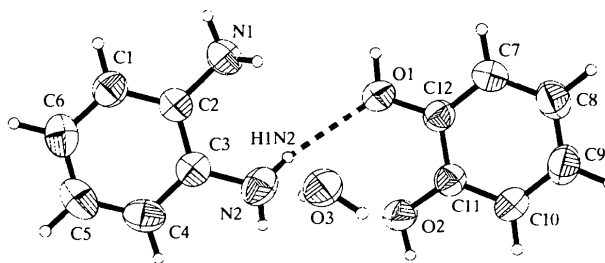


Fig. 1. The structure of the title compound showing 50% probability displacement ellipsoids and the atom-numbering scheme.

† On leave from: Department of Physics, Anna University, Chennai 600 025, India.