

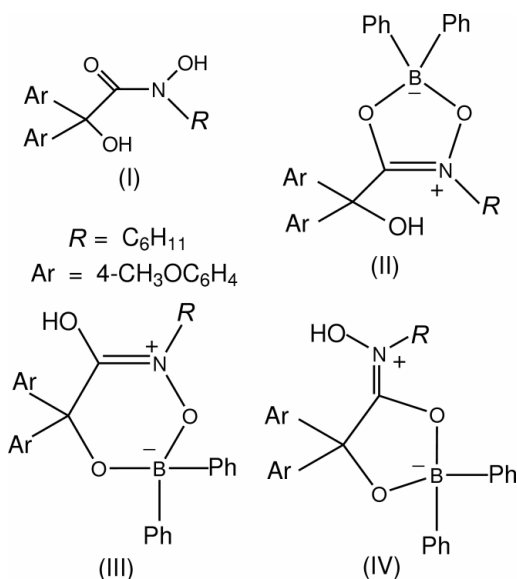
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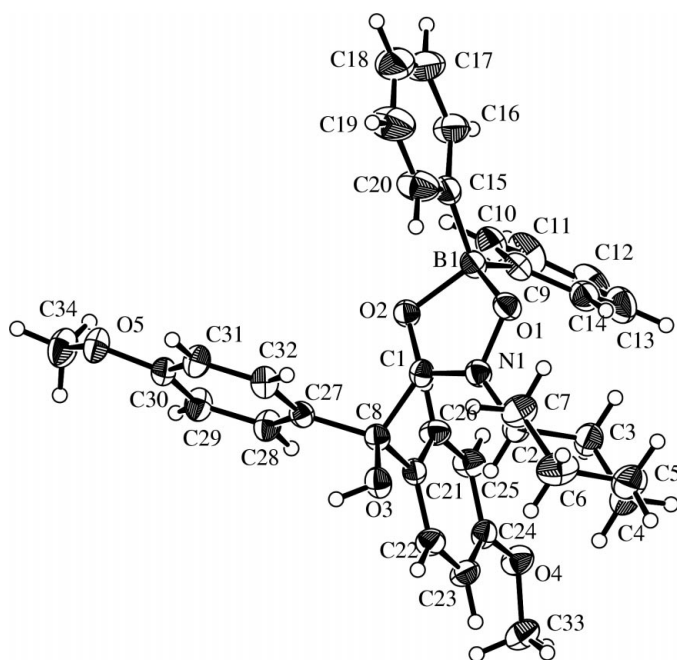
## Key indicators

Single-crystal X-ray study  
 $T = 294\text{ K}$   
Mean  $\sigma(\text{C}-\text{C}) = 0.004\text{ \AA}$   
 $R$  factor = 0.040  
 $wR$  factor = 0.038  
Data-to-parameter ratio = 10.0For details of how these key indicators were automatically derived from the article, see <http://journals.iucr.org/e>.A diphenylboron chelate of an *N*-alkyl-2,2-diaryl-glycolohydroxamic acid4-Cyclohexyl-5-[hydroxy-bis(4-methoxyphenyl)methyl]-2,2-diphenyl-1,3-dioxo-4-azonia-2-borata-4-cyclopentene,  $\text{C}_{34}\text{H}_{36}\text{BNO}_5$ , contains a five-membered heterocyclic ring with a *B*-envelope conformation. Bond lengths and angles are similar to those in related hydroxamatoboron chelates.Received 29 August 2002  
Accepted 5 September 2002  
Online 20 September 2002

## Comment

In the course of our investigations of hydroxamic acids as bi- or polydentate ligands in the formation of boron complexes (Kliegel *et al.*, 1996, and references therein; Kliegel *et al.*, 2001), we chose the 2,2-diaryl derivative of an *N*-alkyl glycolohydroxamic acid, (I), for the chelation of the diphenylboronium ( $\text{Ph}_2\text{B}^+$ ) ion.

The crystalline compound obtained from the reaction of (I) with diphenylboronic acid (anhydride) shows an elemental composition and spectroscopic data corresponding with any of the structural alternatives (II)–(IV). In the  $^1\text{H}$  NMR spectrum of the colorless substance, however, the chemical shift of the OH-proton signal at relatively high field ( $\delta = 3.15$  p.p.m.) points to an alcoholic hydroxy group [as in (II)], rather than to hydroxamic acid moieties [as in (III) or (IV)], which normally give OH signals at a lower field. The molecular structure (II) would harmonize with the five-membered BONCO chelate ring systems established for the diphenylboron chelate of an *N*-unsubstituted 2,2-dialkylglycolohydroxamic acid and other



**Figure 1**  
View of the molecule (33% displacement ellipsoids).

hydroxamatoboron complexes (Kliegel *et al.*, 1996, and references therein; Kliegel *et al.*, 2001). A six-membered BONCCO ring, as in the possible structure (III), had been found in the difluoroboron chelate of an *N,O*-disubstituted 2,2-diphenylglycolohydroxamic acid (Kliegel, Schumacher, Rettig & Trotter, 1991), and the five-membered BOCCO ring system, as in the alternative structure (IV), is realized in the difluoroboron chelates of other derivatives of 2,2-diarylglycolohydroxamic acids (Kliegel *et al.*, 1992). The X-ray analysis confirms the molecular structure (II), a typical hydroxamatoboron complex, the general geometry of which is very similar to that of related compounds (Kliegel *et al.*, 1996, and references therein; Kliegel *et al.*, 2001). The preference of the hydroxamate group as the ligand for the  $\text{Ph}_2\text{B}^+$  ion is in agreement with the findings for the diphenylboron chelates of hydroxamic acids containing additional 2-hydroxy groups as possible nucleophilic ligands in the carbonic acid part (Kliegel *et al.*, 1996) or in the hydroxylamine part (Kliegel, Schumacher, Tajerbashi *et al.*, 1991).

The five-membered BONCO ring has a pronounced *B*-envelope conformation with approximate planarity of the ONCO ring portion [dihedral angle  $4.2(2)^\circ$ ]. A considerable double-bond character of the C1–N1 bond [1.299 (3) Å], with an estimated (Häfelinger, 1970) and calculated (Paolini, 1990)  $\pi$ -bond order of 0.77 and 0.82, respectively, justifies the depicted chemical formula (II), with a predominant C=N double bond, representing the most important canonical structure. However, the C1–O2 bond [1.292 (2) Å] retains about 55% of its double-bond character. The two O–B bonds show an abnormally large difference of 0.083 Å [B–O(N) =

1.515 (3), B–O(C) = 1.598 (3) Å], compared to other hydroxamatoboron complexes, where differences are between 0.001 and 0.035 Å (Kliegel *et al.*, 1996, and references therein; Kliegel *et al.*, 2001). The considerably longer (C)O–B bond [1.598 (3) Å] points to the weaker donor property of the carbonyl group in this *O,O*-bidentate hydroxamate ligand.

Molecules of the chelate (II) are linked along a glide plane by O3–H1...O5 hydrogen bonds (Table 2).

## Experimental

*N*-Cyclohexyl-2,2-bis(4-methoxyphenyl)glycolohydroxamic acid (Geffken & Lauterbach, 1985) (1.16 g, 3 mmol) and diphenylboronic acid anhydride [oxybis(diphenylborane)] (0.52 g, 1.5 mmol) were dissolved in a small amount of absolute ethanol by slight heating. During cooling of the solution crystallization commenced spontaneously or after addition of petroleum ether. Yield of (II): 1.18 g (72%) of colorless crystals (with nacrous lustre), m.p. 417–419 K (from ethanol). IR (KBr): 3450 (O–H), 1610, 1595, 1585 (C=N/C=O, C=C).  $^1\text{H}$  NMR (90 MHz,  $\text{CDCl}_3$ -TMS),  $\delta$  (p.p.m.): 0.75–2.25 (*m*,  $(\text{CH}_2)_5$ ), 3.15 (*s*, exchangeable, OH), 3.78 (*s*, 2 OCH<sub>3</sub>), 3.75–4.30 (*m*, N–CH), 6.73–6.97 (*m*, 4 aromatic H), 7.12–7.60 (*m*, 14 aromatic H). Analysis calculated for  $\text{C}_{34}\text{H}_{36}\text{BNO}_5$ : C 74.32, H 6.60, N 2.55%; found: C 74.25, H 6.65, N 2.65%. The compound gives a blue color reaction with diphenylcarbazone in methanolic solution, indicating the presence of a diphenylboron moiety (Neu, 1960; Friese & Umland, 1978). Crystals suitable for X-ray crystallography were obtained by slow crystallization from ethanol.

### Crystal data

$\text{C}_{34}\text{H}_{36}\text{BNO}_5$   
 $M_r = 549.45$   
Orthorhombic, *Pbca*  
 $a = 24.106(2)$  Å  
 $b = 20.763(2)$  Å  
 $c = 11.8193(7)$  Å  
 $V = 5915.7(8)$  Å<sup>3</sup>  
 $Z = 8$   
 $D_x = 1.234$  Mg m<sup>-3</sup>

Mo  $K\alpha$  radiation  
Cell parameters from 25 reflections  
 $\theta = 18.0$ – $21.4^\circ$   
 $\mu = 0.08$  mm<sup>-1</sup>  
 $T = 294$  K  
Prism, colorless  
 $0.30 \times 0.30 \times 0.20$  mm

### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $\omega$ - $2\theta$  scans  
Absorption correction:  $\psi$  scan (Rettig, 1978)  
 $T_{\min} = 0.97$ ,  $T_{\max} = 0.98$   
6740 measured reflections  
6740 independent reflections

3752 reflections with  $I > 3\sigma(I)$   
 $\theta_{\max} = 27.5^\circ$   
 $h = 0 \rightarrow 31$   
 $k = 0 \rightarrow 27$   
 $l = 0 \rightarrow 15$   
3 standard reflections every 150 reflections  
intensity decay: 1.4%

### Refinement

Refinement on  $F$   
 $R = 0.040$   
 $wR = 0.038$   
 $S = 1.96$   
3752 reflections  
375 parameters

H atoms treated by a mixture of independent and constrained refinement  
 $w = 1/[\sigma^2(F_o) + 0.0004F_o^2]$   
 $(\Delta/\sigma)_{\max} = 0.03$   
 $\Delta\rho_{\max} = 0.16$  e Å<sup>-3</sup>  
 $\Delta\rho_{\min} = -0.22$  e Å<sup>-3</sup>

**Table 1**

Selected geometric parameters (Å, °).

O1—N1	1.393 (2)	C10—C11	1.387 (4)
O1—B1	1.515 (3)	C11—C12	1.364 (5)
O2—C1	1.292 (2)	C12—C13	1.364 (5)
O2—B1	1.598 (3)	C13—C14	1.386 (4)
O3—C8	1.429 (2)	C15—C16	1.382 (3)
O4—C24	1.368 (2)	C15—C20	1.388 (3)
O4—C33	1.427 (3)	C15—B1	1.598 (3)
O5—C30	1.376 (2)	C16—C17	1.391 (4)
O5—C34	1.429 (3)	C17—C18	1.363 (5)
N1—C1	1.299 (2)	C18—C19	1.367 (5)
N1—C2	1.469 (2)	C19—C20	1.378 (4)
C1—C8	1.529 (3)	C21—C22	1.379 (3)
C2—C3	1.518 (3)	C21—C26	1.394 (3)
C2—C7	1.508 (4)	C22—C23	1.390 (3)
C3—C4	1.525 (4)	C23—C24	1.373 (3)
C4—C5	1.500 (4)	C24—C25	1.385 (3)
C5—C6	1.514 (4)	C25—C26	1.371 (3)
C6—C7	1.528 (4)	C27—C28	1.376 (3)
C8—C21	1.537 (3)	C27—C32	1.397 (3)
C8—C27	1.529 (3)	C28—C29	1.388 (3)
C9—C10	1.390 (4)	C29—C30	1.374 (3)
C9—C14	1.386 (3)	C30—C31	1.386 (3)
C9—B1	1.605 (3)	C31—C32	1.368 (3)
N1—O1—B1	105.6 (1)	C16—C15—B1	123.8 (2)
C1—O2—B1	106.7 (1)	C20—C15—B1	120.0 (2)
C24—O4—C33	117.3 (2)	C15—C16—C17	122.1 (2)
C30—O5—C34	117.7 (2)	C16—C17—C18	120.0 (3)
O1—N1—C1	111.6 (1)	C17—C18—C19	119.1 (3)
O1—N1—C2	115.5 (1)	C18—C19—C20	120.7 (3)
C1—N1—C2	133.0 (2)	C15—C20—C19	121.8 (3)
O2—C1—N1	114.1 (2)	C8—C21—C22	121.0 (2)
O2—C1—C8	119.8 (2)	C8—C21—C26	121.2 (2)
N1—C1—C8	125.9 (2)	C22—C21—C26	117.8 (2)
N1—C2—C3	111.0 (2)	C21—C22—C23	121.1 (2)
N1—C2—C7	109.9 (2)	C22—C23—C24	120.3 (2)
C3—C2—C7	111.9 (2)	O4—C24—C23	124.4 (2)
C2—C3—C4	109.0 (2)	O4—C24—C25	116.6 (2)
C3—C4—C5	110.9 (2)	C23—C24—C25	119.0 (2)
C4—C5—C6	111.6 (3)	C24—C25—C26	120.6 (2)
C5—C6—C7	111.0 (2)	C21—C26—C25	121.2 (2)
C2—C7—C6	110.1 (2)	C8—C27—C28	123.9 (2)
O3—C8—C1	107.4 (1)	C8—C27—C32	118.4 (2)
O3—C8—C21	110.7 (1)	C28—C27—C32	117.6 (2)
O3—C8—C27	109.0 (1)	C27—C28—C29	121.6 (2)
C1—C8—C21	105.2 (1)	C28—C29—C30	119.7 (2)
C1—C8—C27	109.3 (1)	O5—C30—C29	124.7 (2)
C21—C8—C27	114.9 (1)	O5—C30—C31	115.7 (2)
C10—C9—C14	115.8 (2)	C29—C30—C31	119.6 (2)
C10—C9—B1	122.4 (2)	C30—C31—C32	120.2 (2)
C14—C9—B1	121.6 (2)	C27—C32—C31	121.2 (2)
C9—C10—C11	122.2 (3)	O1—B1—O2	97.6 (1)
C10—C11—C12	119.9 (3)	O1—B1—C9	112.1 (2)
C11—C12—C13	119.7 (3)	O1—B1—C15	110.1 (2)
C12—C13—C14	120.1 (3)	O2—B1—C9	106.4 (2)
C9—C14—C13	122.2 (3)	O2—B1—C15	109.1 (2)
C16—C15—C20	116.2 (2)	C9—B1—C15	119.2 (2)

**Table 2**

Hydrogen-bonding geometry (Å, °).

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
O3—H1 $\cdots$ O5 <sup>i</sup>	0.87 (3)	2.08 (3)	2.892 (2)	155 (2)

Symmetry code: (i)  $x, \frac{3}{2} - y, \frac{1}{2} + z$ .

The O-bound H atom was refined freely; other H atoms were riding on their parent atoms (C—H 0.95 Å).

Data collection: *CAD-4 Diffractometer Control Software* (Enraf-Nonius, 1978); cell refinement: *CAD-4 Diffractometer Control Software*; data reduction: local programs (Rettig, 1978); program(s) used to solve structure: *MULTAN80* (Main *et al.*, 1980); program(s) used to refine structure: *ORFLS* (Busing *et al.*, 1962); software used to prepare material for publication: *teXsan* (Molecular Structure Corporation, 1989).

We thank the Natural Sciences and Engineering Research Council of Canada and the Fonds der Chemische Industrie, Frankfurt am Main, for financial support.

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