Acta Cryst. (1996). C**52**, 2571–2573

Triphenyl(tetrahydrofuranyl)boron

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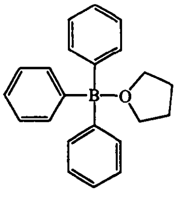
(*Received 4 September 1995; accepted 20 May 1996*)

Abstract

The title compound, $(C_6H_5)_3B(C_4H_8O)$, is monomeric and contains a B atom in a distorted tetrahedral coordination environment bonded to three phenyl groups and one thf moiety. The phenyl groups and thf are canted and the (ligand)—B—(ligand) angles fall within the range 104.2 (2)–116.3 (3)°.

Comment

In the course of studying the ring-opening polymerization reaction of L,L-lactide ($C_6H_8O_4$) with the yttrium alkoxides (McLain, Ford & Drysdale, 1992), crystals of $(C_6H_5)_3B$(thf), (I), were isolated from the reaction of $C_6H_8O_4$ with $[Y(OCMe_3)(Cl)(thf)_5][B(C_6H_5)_4]$ (Evans, Olofson & Ziller, 1990) in THF. The formation of $B(C_6H_5)_3$ from $[B(C_6H_5)_4]^-$ has considerable precedence (Kliegel, Lubkowitz, Rettig & Trotter, 1993; Bakshi *et al.*, 1994). A literature search (Allen *et al.*, 1979) indicates that $(C_6H_5)_3B$(thf) is only the second $(C_6H_5)_3B$(solvent) structure known. The closest related structure, $(C_6H_5)_3B(H_2O)$, is found in the complex *trans*-$[Re(O)_2(cyclam)]Cl.2[(C_6H_5)_3B(H_2O)]$, presumably from the acid-catalysed hydrolysis of $B(C_6H_5)_4^-$ (Blake, Grieg & Schröder, 1988). Recently, triphenylboron has been used in chemical vapour deposition preparations of highly conductive boron-doped graphite films (Kouvetakis, McElfresh & Beach, 1994).



(I)

The title compound is monomeric and the B(1) atom has distorted tetrahedral coordination generated by three phenyl ligands and one tetrahydrofuran ligand, as shown in Fig. 1. The planes of the phenyl rings are canted in a propeller-like fashion. The range of (ligand)—B—(ligand) angles defining this tetrahedron is 104.2 (2)–116.3 (3)°. The B—O(thf) bond distance [1.660 (4) Å] is longer those of 1.524 (4), 1.526 (3) and 1.506 (6) Å in $[2,3-(C_2H_5)_2C_2B_4H_4]$-5-Co$[B_9H_{12}$-1-(thf)], $[2,3-(CH_3)_2C_2B_3H_5]$-5-Co$[B_9H_{12}$-1-(thf)] and $[1,2-(C_2H_5)_2C_2B_7H_7]$-6-Co$[B_9H_{12}$-2-(thf)], respectively (Borodinsky, Sinn & Grimes, 1982). The average B—C bond distance of 1.622 (4) Å in (I) is similar to that of 1.647 (9) Å found in $(C_6H_5)_3B(H_2O)$ (Blake *et al.*, 1988). The range of C—H distances in (I) is 0.92 (3)–1.05 (4) Å and U_{iso} for the H atoms varied from 0.013 (7)–0.069 (14). Fig. 2 shows a plot of two molecules oriented to show the closest contacts. The distance of H(21*A*), which is bonded to C21, to the C13–C18 ring ranges from 2.90–3.15 Å.

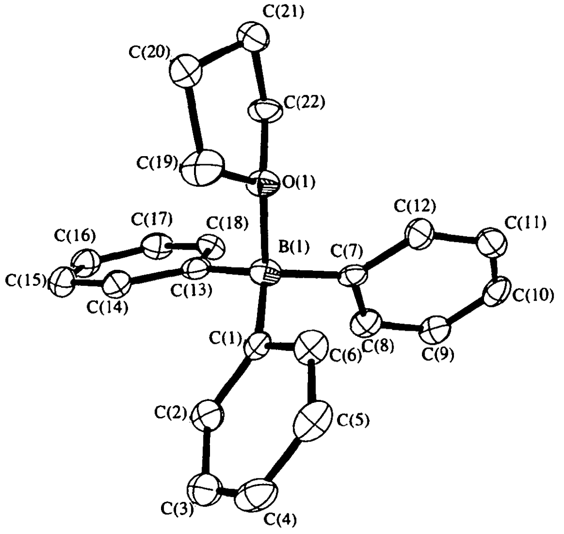


Fig. 1. Molecular structure of (I) with the probability ellipsoids drawn at the 50% level. H atoms have been omitted for clarity.

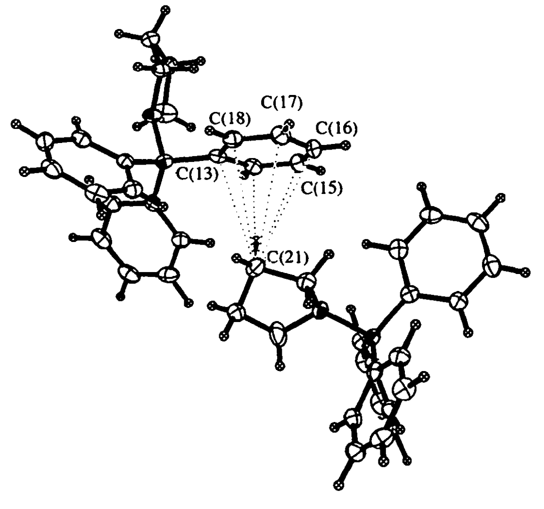


Fig. 2. Plot of two molecules oriented to show closest contacts.

Experimental

$(C_6H_5)_3B(thf)$ was formed in the reaction of $C_6H_8O_4$ with $[Y(OCMe_3)(Cl)(thf)_5][BPh_4]$ in THF, and recrystallized from THF at 238 K.

Crystal data

$C_{22}H_{23}BO$
$M_r = 314.2$
Monoclinic
$P2_1/c$
$a = 9.0882\,(8)$ Å
$b = 12.7583\,(14)$ Å
$c = 15.5461\,(13)$ Å
$\beta = 104.725\,(7)°$
$V = 1743.4\,(3)$ Å^3
$Z = 4$
$D_x = 1.197$ Mg m^{-3}
D_m not measured

Mo $K\alpha$ radiation
$\lambda = 0.71073$ Å
Cell parameters from 36 reflections
$\theta = 10$–$14.5°$
$\mu = 0.070$ mm^{-1}
$T = 163$ K
Prism
$0.43 \times 0.23 \times 0.20$ mm
Colourless

Data collection

Syntex $P2_1$ diffractometer
$\theta/2\theta$ scans
Absorption correction: none
3461 measured reflections
2749 independent reflections
2228 observed reflections $[F > 3\sigma(F)]$
$R_{int} = 0.0109$

$\theta_{max} = 25.0°$
$h = 0 \rightarrow 13$
$k = 0 \rightarrow 17$
$l = -21 \rightarrow 21$
2 standard reflections monitored every 98 reflections
intensity decay: <1%

Refinement

Refinement on F
$R = 0.063$
$wR = 0.067$
$S = 1.31$
2228 reflections
310 parameters
H atoms refined isotropically
$w = 1/[\sigma^2(F) + 0.0010F^2]$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.26$ e Å^{-3}
$\Delta\rho_{min} = -0.29$ e Å^{-3}

Extinction correction: $F^* = F[1+0.002\chi F^2/\sin(2\theta)]^{-1/4}$
Extinction coefficient: $\chi = 0.0010\,(4)$
Atomic scattering factors from *International Tables for Crystallography* (1992, Vol. C)

Table 1. *Fractional atomic coordinates and equivalent isotropic displacement parameters (Å^2)*

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
B(1)	0.8742 (4)	0.3085 (3)	0.1958 (2)	0.211 (11)
O(1)	0.8768 (2)	0.2520 (2)	0.29229 (12)	0.228 (7)
C(1)	1.0511 (3)	0.3352 (2)	0.20086 (18)	0.0214 (9)
C(2)	1.1156 (4)	0.3185 (2)	0.1297 (2)	0.254 (10)
C(3)	1.2636 (4)	0.3498 (3)	0.1325 (2)	0.320 (12)
C(4)	1.3519 (4)	0.3996 (3)	0.2066 (2)	0.337 (12)
C(5)	1.2903 (4)	0.4184 (3)	0.2781 (2)	0.320 (11)
C(6)	1.1426 (3)	0.3871 (2)	0.2745 (2)	0.271 (11)
C(7)	0.7830 (3)	0.4173 (2)	0.19778 (18)	0.220 (9)
C(8)	0.7181 (3)	0.4717 (2)	0.1186 (2)	0.255 (10)
C(9)	0.6472 (4)	0.5682 (3)	0.1172 (2)	0.311 (11)
C(10)	0.6413 (3)	0.6162 (3)	0.1967 (2)	0.314 (12)
C(11)	0.7060 (4)	0.5658 (3)	0.2761 (2)	0.300 (11)
C(12)	0.7748 (3)	0.4687 (2)	0.2763 (2)	0.266 (10)
C(13)	0.7919 (3)	0.2246 (2)	0.12080 (19)	0.220 (9)
C(14)	0.8699 (4)	0.1391 (2)	0.0964 (2)	0.254 (10)
C(15)	0.8004 (4)	0.0682 (3)	0.0306 (2)	0.292 (11)
C(16)	0.6484 (4)	0.0803 (3)	−0.0137 (2)	0.302 (11)
C(17)	0.5660 (4)	0.1612 (3)	0.0106 (2)	0.302 (11)
C(18)	0.6362 (4)	0.2310 (3)	0.0767 (2)	0.277 (11)
C(19)	0.9969 (4)	0.1760 (3)	0.3333 (3)	0.412 (14)
C(20)	0.9292 (4)	0.1060 (3)	0.3905 (2)	0.287 (11)
C(21)	0.7816 (4)	0.1597 (3)	0.3957 (2)	0.283 (11)
C(22)	0.7318 (3)	0.2142 (3)	0.3071 (2)	0.249 (10)

Table 2. *Selected geometric parameters (Å, °)*

B(1)—O(1)	1.660 (4)	C(8)—C(9)	1.388 (4)
B(1)—C(1)	1.626 (4)	C(9)—C(10)	1.392 (5)
B(1)—C(7)	1.620 (4)	C(10)—C(11)	1.383 (5)
B(1)—C(13)	1.619 (4)	C(11)—C(12)	1.387 (4)
O(1)—C(19)	1.477 (4)	C(13)—C(14)	1.405 (4)
O(1)—C(22)	1.476 (4)	C(13)—C(18)	1.409 (4)
C(1)—C(2)	1.394 (5)	C(14)—C(15)	1.390 (4)
C(1)—C(6)	1.399 (4)	C(15)—C(16)	1.387 (4)
C(2)—C(3)	1.393 (5)	C(16)—C(17)	1.383 (5)
C(3)—C(4)	1.380 (4)	C(17)—C(18)	1.386 (4)
C(4)—C(5)	1.387 (5)	C(19)—C(20)	1.499 (6)
C(5)—C(6)	1.388 (4)	C(20)—C(21)	1.527 (5)
C(7)—C(8)	1.406 (4)	C(21)—C(22)	1.505 (4)
C(7)—C(12)	1.404 (4)		
O(1)—B(1)—C(1)	105.1 (2)	O(1)—B(1)—C(13)	105.1 (2)
O(1)—B(1)—C(7)	104.2 (2)	C(1)—B(1)—C(13)	116.3 (3)
C(1)—B(1)—C(7)	109.0 (2)	C(7)—B(1)—C(13)	115.8 (2)

Background counts were estimated from a 96-step profile using the program *CARESS* as implemented in the *UCLA Crystallographic Computing Package* (Strouse, 1981). All subsequent crystallographic calculations (structure solution, refinement *etc.*) were performed using *SHELXTL/PC* (Sheldrick, 1990).

For support of this research, we thank the Division of Chemical Sciences of the Office of Basic Energy Sciences of the Department of Energy.

Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the IUCr (Reference: BK1195). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Acta Cryst. (1996). C**52**, 2573–2575

A Reinvestigation of the Quinoidal Effect in *N*-*n*-Propyl-2-oxo-1-naphthylidenemethylamine

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(Received 22 January 1996; accepted 7 May 1996)

Abstract

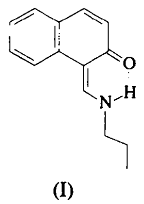
In the title compound $C_{14}H_{15}NO$ (systematic name: 1-*n*-propylaminomethylene-2-naphthalenone), an intramolecular N—H···O hydrogen bond [N···O 2.578 (2), N—H 0.775, H···O 1.936 Å] arises when the hydroxyl-H atom of the Schiff base prepared from 2-hydroxy-1-naphthaldehyde and *n*-propylamine shifts to the N atom. This is the consequence of the pronounced quinoidal effect in the 2-oxo-1-naphthaldimine moiety which has a very short C3=C4 bond of 1.343 (3) Å. The spatial orientation of the *N*-substituent with respect to the rest of the molecule (which is planar) depends on the surroundings of the molecules defined by the crystal packing.

Comment

In a recently published paper on the structure of 2-hydroxy-*N*-*n*-propyl-1-naphthaldimine (Hökelek, Gündüz, Hayvali & Kiliç, 1995), the H atom involved in the intramolecular O—H···N bond was positioned geometrically at 1.0 Å from the donor atom. This proton assignment was obviously inferred from NMR spectroscopy data. This conclusion is in contrast with the results obtained from the study of the dominant tautomeric form of 2-oxo-1-naphthaldimines (Gavranić, Kaitner & Meštrović, 1996).

An investigation of the structures of the ligands themselves was undertaken in order to reveal the presence of either the benzenoid or quinoid form (or the predominant presence of one of them) in the crystalline state. The synthesis and structure of Schiff bases derived from 2-hydroxy-1-naphthaldehyde with various alkyl and aryl *N*-substituents, and their metal complexes have been of interest recently in our laboratory. Bidentate Schiff base ligands derived from aryl aldehydes with a hydroxyl group in position 2 of the aryl ring to which the C=N imino group is bonded have been studied extensively for years. Unfortunately, almost all investigations of the crystal structures of the above-mentioned systems were performed on the aldimine compounds or their metal complexes based on salicylaldehyde (2-hydroxy-1-benzaldehyde). There is a lack of information about similar Schiff base compounds derived from 2-hydroxy-1-naphthaldehyde. The dissimilarity between these two aldehydes arises from the difference in the ring moiety, a single ring compared to a fused double-ring system. The discrepancy appears in the arrangement of the endocyclic bond distances. The characteristic D_{2h} pattern of the naphthalene bond lengths (Allen, Kennard, Watson, Brammer & Orpen, 1987) is preserved in the imino compounds of 2-hydroxy-1-naphthaldehyde and this is the most important feature distinguishing the naphthaldimines from the salicylaldimines. The existence of the enol (or predominantly enol) tautomer has been established in all crystal structures of *N*-substituted salicylaldimines listed so far in the Cambridge Structural Database (October 1995 release). In contrast, no structural data on the tautomeric form assumed in the naphthaldimine derivatives could be found. The predominance of the keto-imine form of 2-hydroxy-1-naphthaldimines in solution was confirmed by NMR spectroscopy (Costamagna, Vargas, Latorre, Alvarado & Mena, 1992).

Our crystallographic studies showed an intramolecular N—H···O hydrogen bond to be present in naphthaldimines in the solid state (Pavlović, Doležal, Gabud & Kaitner, 1995; Gavranić, Kaitner & Meštrović, 1996) regardless of the kind of *N*-substituent, aryl or alkyl. We established the presence in the title compound (I) of an N—H···O hydrogen bond with very similar parameters to those reported by Hökelek *et al.* (1995). In (I), the O···N 'bite' distance is 2.578 (2) Å with N—H1N and O···H1N distances of 0.775 and 1.936 Å, respectively. A view of the title compound is presented in Fig. 1. For clarity, the same atomic numbering scheme as used by Hökelek *et al.* (1995) was adopted. The atom H1N was located from a difference Fourier map as a well defined small electron density maximum of 0.53 e Å^{-3} (Fig. 2).



(I)