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Key indicators

Single-crystal X-ray study
 $T = 100$ K
Mean $\sigma(\text{C}-\text{C}) = 0.003$ Å
 R factor = 0.033
 wR factor = 0.077
Data-to-parameter ratio = 18.6For details of how these key indicators were
automatically derived from the article, see
<http://journals.iucr.org/e>.Diethyl ether–trichloroborane, $(\text{C}_2\text{H}_5)_2\text{O}-\text{BCl}_3$ Received 1 September 2006
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The title compound, $\text{C}_4\text{H}_{10}\text{BCl}_3\text{O}$, is the first crystallographically characterized example of a trihaloborane complex with an ether as electron-pair donor. The B atom is coordinated tetrahedrally, whereas oxygen has a strongly distorted coordination environment. The central B–O bond length is 1.543 (2) Å and is thus longer than those in tetraalkoxyborates, $\text{B}(\text{OR})_4^-$.

Comment

Halogenoborane–Lewis base complexes $L-\text{BX}_3$ (L = amine, phosphane, ether, nitrile or other electron-pair donor; $X = \text{F}, \text{Cl}, \text{Br}, \text{I}$) are versatile reactants in organic and inorganic chemistry. Though investigated spectroscopically in much detail (Hunt & Ault, 1982; Fratiello *et al.*, 1968), no publications dealing with single-crystal structure X-ray analysis of ether complexes of BX_3 have appeared so far. Colorless crystals of $(\text{C}_2\text{H}_5)_2\text{O}-\text{BCl}_3$, (I), were obtained from the reaction of boron trichloride and diethyl ether at 243 K and subsequent crystallization from diethyl ether/*n*-hexane (10:1) at 243 K. The central B–O bond (Fig. 1 and Table 1) is *ca* 5% longer than the average B–O bond lengths in organooxyborate anions, $\text{B}(\text{OR})_4^-$ (Alcock *et al.*, 1982; Abrahams *et al.*, 2005) or the B–O bond lengths of tetrahedrally coordinated B atoms in potassium diborate (Krogh-Moe, 1972). Boron has a slightly distorted tetrahedral coordination environment. The deviation from ideal tetrahedral angles is *ca* 2° for Cl–B–Cl (Table 1). The B–Cl distance in a Lewis base complex of BCl_3 is an indicator of the donor strength of the coordinating base. The average B–Cl bond length in $(\text{C}_2\text{H}_5)_2\text{O}-\text{BCl}_3$ of 1.830 Å (Table 1) reflects the low donor capability of diethyl ether. It is similar to, for example, triphenyl phosphate, $(\text{PhO})_3\text{PO}$ (Levin *et al.*, 1982), and

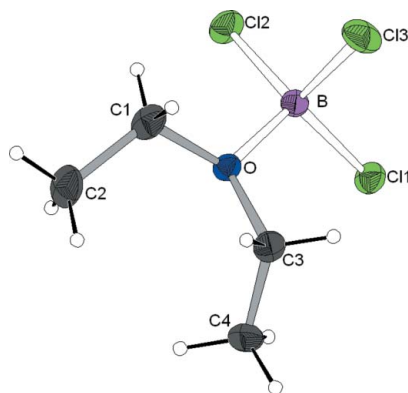
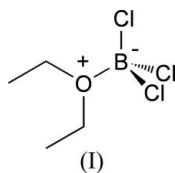


Figure 1

The molecular structure of the title compound, showing the atom-labeling scheme. Displacement ellipsoids are drawn at the 50% probability level.

tetrahydrothiophene, C₄H₈S (Krebs *et al.*, 1989) BCl₃ complexes [average B–Cl distances in (PhO)₃PO–BCl₃ and C₄H₈S–BCl₃ are 1.824 (5) and 1.827 (3) Å, respectively]. In contrast, the average B–Cl bond distances in BCl₃ complexes with stronger donors, such as trimethylphosphine, Me₃P (Black & Taylor, 1975), and Et₂(ArCH₂)N [Ar = 2-BCl₂-3-(CH₂NEt₂)C₆H₃; Schlengermann *et al.*, 1997], are 1.855 (5) and 1.849 (5) Å, respectively. The coordination environment of oxygen deviates significantly from an ideal pseudo-tetrahedral (the fourth position is occupied by the non-coordinating electron lone pair). The sum of bond angles at O is 346.7°.



Experimental

All manipulations were performed in a purified Ar atmosphere using standard Schlenk techniques. Solvents were dried prior to their use by distillation from sodium. BCl₃ (8 g, 68 mmol) was condensed into a 250 ml flask which was cooled to 243 K. With vigorous stirring, diethyl ether (50 ml) was added slowly. Precipitation of a colorless solid was observed. The reaction mixture was allowed to warm to room temperature overnight. *n*-Hexane was added to the clear reaction mixture (the precipitate completely dissolved upon warming) until it turned slightly cloudy. Slow cooling to 243 K afforded crystals of (C₂H₅)₂O–BCl₃ as colorless blocks.

Crystal data

C ₄ H ₁₀ BCl ₃ O	$Z = 8$
$M_r = 191.28$	$D_x = 1.489 \text{ Mg m}^{-3}$
Orthorhombic, <i>Pbcn</i>	Mo $K\alpha$ radiation
$a = 11.4235 (6) \text{ \AA}$	$\mu = 1.00 \text{ mm}^{-1}$
$b = 11.8071 (6) \text{ \AA}$	$T = 100 (2) \text{ K}$
$c = 12.6502 (6) \text{ \AA}$	Block, colorless
$V = 1706.24 (15) \text{ \AA}^3$	$0.45 \times 0.35 \times 0.15 \text{ mm}$

Data collection

Bruker SMART APEX diffractometer	24274 measured reflections
ω scans	2274 independent reflections
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	2196 reflections with $I > 2\sigma(I)$
$T_{\min} = 0.663$, $T_{\max} = 0.865$	$R_{\text{int}} = 0.028$
	$\theta_{\text{max}} = 29.0^\circ$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.0258P)^2 + 1.1215P]$
$R[F^2 > 2\sigma(F^2)] = 0.033$	where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.077$	$(\Delta/\sigma)_{\text{max}} = 0.001$
$S = 1.33$	$\Delta\rho_{\text{max}} = 0.48 \text{ e \AA}^{-3}$
2274 reflections	$\Delta\rho_{\text{min}} = -0.22 \text{ e \AA}^{-3}$
122 parameters	
All H-atom parameters refined	

Table 1

Selected geometric parameters (Å, °).

Cl1–B	1.8292 (18)	O–C1	1.4945 (19)
Cl2–B	1.8250 (17)	O–C3	1.5006 (18)
Cl3–B	1.8361 (18)	O–B	1.5430 (19)
O–B–Cl2	108.29 (10)	O–B–Cl3	108.10 (10)
O–B–Cl1	107.24 (10)	Cl2–B–Cl3	111.40 (9)
Cl2–B–Cl1	110.10 (9)	Cl1–B–Cl3	111.55 (9)

H atoms were observed in a difference Fourier map and refined freely [C–H = 0.93 (2)–0.98 (3) Å].

Data collection: *SMART* (Bruker, 1997); cell refinement: *SAINTE* (Bruker, 1997); data reduction: *SAINTE*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *CIFTAB* in *SHELXL97*.

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