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N-Imidazole–boron trichloride adduct

R. Angharad Baber, Andrew E. A. Bull, Jonathan P. H. Charmant,* Nicholas C. Norman and A. Guy Orpen

School of Chemistry, University of Bristol, Bristol BS8 1TS, England

Correspondence e-mail: jon.charmant@bris.ac.uk

Key indicators

Single-crystal X-ray study
T = 173 K
Mean σ(C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.070
Data-to-parameter ratio = 19.5

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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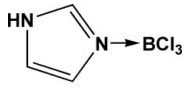
The crystal structure of the title compound [alternatively called trichloro(1*H*-imidazole-κN^3)boron], $C_3H_4N_2$–BCl_3 or $C_3H_4BCl_3N_2$, consists of a weakly hydrogen-bonded network of BCl_3–imidazole adducts. The network formed may be viewed as a cross-linked hydrogen-bonded ribbon polymer.

Comment

The title compound, (I), was obtained as a colourless powder during an attempt to synthesize a product of formula B_2S_3 from the reaction of BCl_3 with $(Me_3Si)_2S$ (containing trace amounts of imidazole as a stabiliser). Recrystallization yielded crystals suitable for a diffraction study. The molecular structure of (I) is shown in Fig. 1, and selected bond lengths and angles are presented in Table 1.



(I)

A variety of nitrogen adducts of BCl_3 have previously been characterized crystallographically. These include amine (Minkwitz, Nass & Preest, 1987; Minkwitz, Nass, Rieland & Preest, 1987; Avent *et al.*, 1995, Hess, 1969; Anton *et al.*, 1984; Abram *et al.*, 1997; Voigt *et al.*, 2000), pyridine (Töpel *et al.*, 1981) and acetonitrile (Swanson *et al.*, 1969) adducts. The B—N bond length in (I) is shorter than any previously reported, with the exception of adducts with rhenium nitride complexes (Dantona *et al.*, 1984; Abram *et al.*, 1997; Ritter & Abram, 1996).

The crystal structure of (I) may be viewed as a cross-linked hydrogen-bonded ribbon polymer (see Fig. 2). The N2—H2*A* donor of the imidazole makes a weak hydrogen bond with atom Cl1 in a neighbouring molecule. This interaction is supplemented by a weak interaction between C2—H2 and Cl3 of the same molecule. Although such an interaction might seem dubious, it is possible that C2 and N2 are disordered with respect to each other, leading to a disordered hydrogen bond between Cl1 or Cl3 and the two chemically feasible NH positions on the imidazole. Attempts to model this disorder were unsuccessful. A slightly stronger interaction between the N2—H2*A* donor and Cl2 of another neighbouring molecule cross-links the ribbons to give the overall structure.

Experimental

BCl_3 (1.0 *M* solution in heptane, 0.2 ml, 0.2 mmol) was added to a solution of $(Me_3Si)_2S$ (0.57 ml, 0.3 mmol) in hexane (10 ml), resulting in the immediate formation of a colourless precipitate. The solution was stirred for 24 h, whereupon the solvent was removed by syringe

and the resultant colourless solid was washed with hexane (3 × 10 ml) and dried. The solid was then redissolved in CH_2Cl_2 (10 ml), placed in a fresh Schlenk tube, layered with hexane (7 ml) and refrigerated at 243 K overnight, resulting in the formation of large colourless crystals (yield: 0.0056 g, 6%). NMR ($CDCl_3$): ^{11}B δ 3.1. Analysis calculated for $C_3H_4BCl_3N_2$: C 19.45, H 2.20, N 15.10%; found: C 19.60, H 1.65, N 14.85%.

Crystal data

$C_3H_4BCl_3N_2$	$Z = 2$
$M_r = 185.24$	$D_x = 1.764$ Mg m^{-3}
Triclinic, $P\overline{1}$	Mo $K\alpha$ radiation
$a = 6.0390$ (12) Å	Cell parameters from 1476 reflections
$b = 7.2210$ (14) Å	$\theta = 3.0$–$27.4°$
$c = 8.5610$ (17) Å	$\mu = 1.21$ mm^{-1}
$\alpha = 84.48$ (3)°	$T = 173$ (2) K
$\beta = 81.33$ (3)°	Plate, colourless
$\gamma = 71.08$ (3)°	0.15 × 0.15 × 0.05 mm
$V = 348.67$ (14) Å^3	

Data collection

Bruker SMART CCD area-detector diffractometer	1597 independent reflections
	1444 reflections with $I > 2\sigma(I)$
ω scans	$R_{int} = 0.023$
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)	$\theta_{max} = 27.5°$
	$h = -7 \rightarrow 7$
$T_{min} = 0.853$, $T_{max} = 0.940$	$k = -9 \rightarrow 9$
4070 measured reflections	$l = -11 \rightarrow 11$

Refinement

Refinement on F^2	$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 0.0819P]$ where $P = (F_o^2 + 2F_c^2)/3$
$R[F^2 > 2\sigma(F^2)] = 0.030$	
$wR(F^2) = 0.070$	
$S = 1.06$	$(\Delta/\sigma)_{max} < 0.001$
1597 reflections	$\Delta\rho_{max} = 0.39$ e Å^{-3}
82 parameters	$\Delta\rho_{min} = -0.30$ e Å^{-3}
H-atom parameters constrained	

Table 1
Selected geometric parameters (Å, °).

C1—N2	1.327 (3)	B1—N1	1.543 (3)
C1—N1	1.332 (2)	B1—Cl1	1.847 (2)
C2—C3	1.346 (3)	B1—Cl3	1.848 (2)
C2—N2	1.378 (3)	B1—Cl2	1.865 (2)
C3—N1	1.389 (2)		
N2—C1—N1	108.82 (18)	Cl1—B1—Cl2	109.35 (11)
C3—C2—N2	106.08 (17)	Cl3—B1—Cl2	109.11 (11)
C2—C3—N1	108.22 (17)	C1—N1—C3	107.41 (16)
N1—B1—Cl1	108.73 (13)	C1—N1—B1	126.94 (16)
N1—B1—Cl3	109.43 (13)	C3—N1—B1	125.62 (16)
Cl1—B1—Cl3	110.88 (11)	C1—N2—C2	109.46 (16)
N1—B1—Cl2	109.32 (14)		

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

D—H···A	D—H	H···A	D···A	D—H···A
N2—H2A···Cl2^{i}	0.88	2.57	3.3696 (19)	152
N2—H2A···Cl1^{ii}	0.88	2.86	3.429 (2)	124
C2—H2···Cl3^{ii}	0.95	2.87	3.815 (2)	171

Symmetry codes: (i) $x, y-1, z$; (ii) $1+x, y-1, z$.

H atoms were constrained to ideal geometries (C—H = 0.95 Å) and refined with displacement parameters equal to 1.2 times U_{eq} of their parent atom.

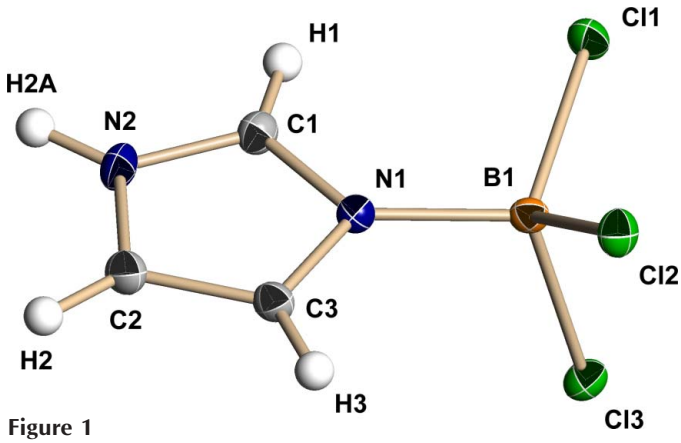


Figure 1
The molecular structure of the title compound, showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

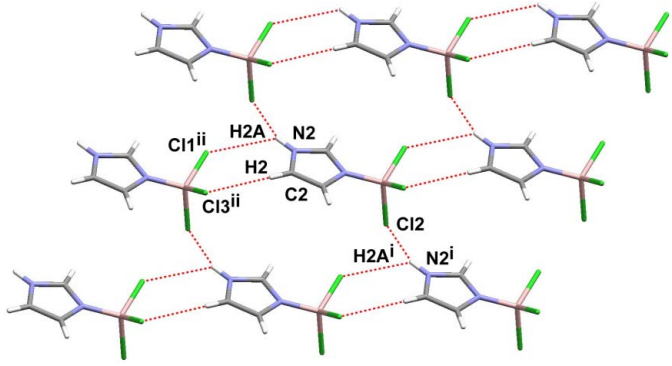


Figure 2
The crystal structure of the title compound, viewed as a series of cross-linked hydrogen-bonded ribbon polymers. [Symmetry codes: (i) $x, y-1, z$; (ii) $1+x, y-1, z$.]

Data collection: *SMART* (Bruker, 2002); cell refinement: *SAINT* (Bruker, 2002); data reduction: *SAINT* and *SHELXTL* (Bruker, 2002); program(s) used to solve structure: *SHELXTL*; program(s) used to refine structure: *SHELXTL*; molecular graphics: *SHELXTL*; software used to prepare material for publication: *SHELXTL*.

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