

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

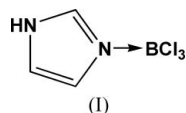
Single-crystal X-ray study
T = 173 K
Mean σ (C–C) = 0.003 Å
R factor = 0.030
wR factor = 0.070
Data-to-parameter ratio = 19.5

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

The crystal structure of the title compound [alternatively called trichloro(*1H*-imidazole- κ N³)boron], C₃H₄N₂–BCl₃ or C₃H₄BCl₃N₂, consists of a weakly hydrogen-bonded network of BCl₃–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 B₂S₃ from the reaction of BCl₃ with (Me₃Si)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.



A variety of nitrogen adducts of BCl₃ 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–H2A 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–H2A donor and Cl2 of another neighbouring molecule cross-links the ribbons to give the overall structure.

Experimental

BCl₃ (1.0 *M* solution in heptane, 0.2 ml, 0.2 mmol) was added to a solution of (Me₃Si)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

Received 18 January 2005

Accepted 24 January 2005

Online 5 February 2005

and the resultant colourless solid was washed with hexane (3 × 10 ml) and dried. The solid was then redissolved in CH₂Cl₂ (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 (CDCl₃): ¹¹B δ 3.1. Analysis calculated for C₃H₄BCl₃N₂: C 19.45, H 2.20, N 15.10%; found: C 19.60, H 1.65, N 14.85%.

Crystal data

C₃H₄BCl₃N₂ Z = 2
M_r = 185.24 D_x = 1.764 Mg m⁻³
Triclinic, P $\bar{1}$ Mo K α radiation
Cell parameters from 1476 reflections
a = 6.0390 (12) Å θ = 3.0–27.4°
b = 7.2210 (14) Å μ = 1.21 mm⁻¹
c = 8.5610 (17) Å T = 173 (2) K
 α = 84.48 (3)° Plate, colourless
 β = 81.33 (3)° 0.15 × 0.15 × 0.05 mm
 γ = 71.08 (3)°
V = 348.67 (14) Å³

Data collection

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

Refinement

Refinement on F² w = 1/[$\sigma^2(F_o^2) + (0.037P)^2 + 0.0819P$]
R[F² > 2 σ (F²)] = 0.030 where P = (F_o² + 2F_c²)/3
wR(F²) = 0.070 (Δ/σ)_{max} < 0.001
S = 1.06 $\Delta\rho_{max}$ = 0.39 e Å⁻³
1597 reflections $\Delta\rho_{min}$ = -0.30 e Å⁻³
82 parameters
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.

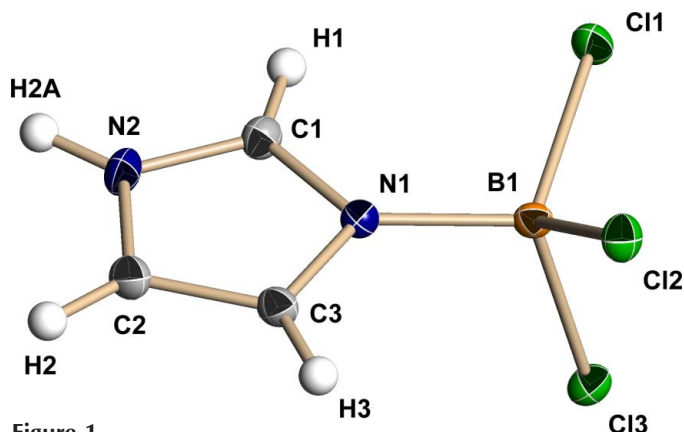


Figure 1 The molecular structure of the title compound, showing the atom labelling scheme. Displacement ellipsoids are drawn at the 50% probability level.

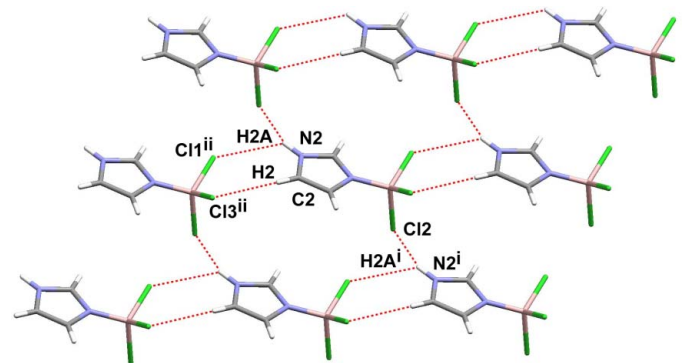


Figure 2 The crystal structure of the title compound, viewed as a series of cross-linked hydrogen-bonded ribbon polymers. [Symmetry codes: (i) x, y – 1, z; (ii) 1 + x, y – 1, z.]

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.

We thank the EPSRC for financial support.

References

Abram, V., Lang, E. S., Abram, S., Wegmann, J., Dilworth, J. R., Kirmse, R. & Woollins, J. D. (1997). *J. Chem. Soc. Dalton Trans.* pp. 623–630.
Anton, K., Noth, H. & Pommering, H. (1984). *Chem. Ber.* **117**, 2479–2494.
Avent, A. G., Hitchcock, P. B., Lappert, M. F., Liu, D., Mignoni, G., Richard, C. & Roche, E. (1995). *Chem. Commun.* pp. 855–856.
Bruker (2002). SMART, SAINT and SHELXTL. Bruker AXS Inc., Madison, Wisconsin, USA.
Dantona, R., Schreda, E. & Stralite, J. (1984). *Z. Naturforsch. Teil B*, **39**, 733–735.
Hess, H. (1969). *Acta Cryst.* **B25**, 2338–2341.
Minkwitz, R., Nass, R. & Preest, H. (1987). *Z. Anorg. Allg. Chem.* **546**, 99–106.
Minkwitz, R., Nass, R., Rieland, M. & Preest, H. (1987). *Z. Anorg. Allg. Chem.* **550**, 133–139.
Ritter, S. & Abram, U. (1996). *Z. Anorg. Allg. Chem.* **622**, 965–973.
Sheldrick, G. M. (2003). SADABS. University of Göttingen, Germany.
Swanson, R., Shriver, D. F. & Ibers, J. A. (1969). *Inorg. Chem.* **8**, 2182–2189.
Töpel, K. H., Hansen, K. & Tromel, M. (1981). *Acta Cryst.* **B37**, 969–971.
Voigt, F., Jacob, K., Seidel, N., Fischer, A., Pietzsch, C. & Zanello, P. (2000). *J. Prakt. Chem.* **342**, 666–674.

supporting information

Acta Cryst. (2005). E61, o553–o554 [https://doi.org/10.1107/S1600536805002631]

N-Imidazole–boron trichloride adduct

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

(I)

Crystal data

$C_3H_4BCl_3N_2$

$M_r = 185.24$

Triclinic, $P\bar{1}$

Hall symbol: -P 1

$a = 6.0390$ (12) Å

$b = 7.2210$ (14) Å

$c = 8.5610$ (17) Å

$\alpha = 84.48$ (3)°

$\beta = 81.33$ (3)°

$\gamma = 71.08$ (3)°

$V = 348.67$ (14) Å³

$Z = 2$

$F(000) = 184$

$D_x = 1.764$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 1476 reflections

$\theta = 3.0$ – 27.4 °

$\mu = 1.21$ mm⁻¹

$T = 173$ K

Plate, colourless

$0.15 \times 0.15 \times 0.05$ mm

Data collection

Bruker SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

Detector resolution: 8.366 pixels mm⁻¹
frames, each covering 0.3° in ω scans

Absorption correction: multi-scan
(SADABS; Sheldrick, 2003)

$T_{\min} = 0.853$, $T_{\max} = 0.940$

4070 measured reflections

1597 independent reflections

1444 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.023$

$\theta_{\max} = 27.5$ °, $\theta_{\min} = 2.4$ °

$h = -7 \rightarrow 7$

$k = -9 \rightarrow 9$

$l = -11 \rightarrow 11$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.030$

$wR(F^2) = 0.070$

$S = 1.06$

1597 reflections

82 parameters

0 restraints

Primary atom site location: structure-invariant
direct methods

Secondary atom site location: difference Fourier
map

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.037P)^2 + 0.0819P]$
where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} < 0.001$

$\Delta\rho_{\max} = 0.39$ e Å⁻³

$\Delta\rho_{\min} = -0.30$ e Å⁻³

Special details

Geometry. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

Refinement. Refinement of F^2 against ALL reflections. The weighted R -factor wR and goodness of fit S are based on F^2 , conventional R -factors R are based on F , with F set to zero for negative F^2 . The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating R -factors(gt) etc. and is not relevant to the choice of reflections for refinement. R -factors based on F^2 are statistically about twice as large as those based on F , and R -factors based on ALL data will be even larger.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2)

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
C1	0.6954 (4)	0.2644 (3)	0.2045 (2)	0.0152 (4)
H1	0.5650	0.2734	0.1512	0.018*
C2	1.0222 (4)	0.1432 (3)	0.3170 (2)	0.0162 (4)
H2	1.1585	0.0529	0.3554	0.019*
C3	0.9453 (3)	0.3391 (3)	0.3283 (2)	0.0142 (4)
H3	1.0189	0.4129	0.3765	0.017*
B1	0.5971 (4)	0.6339 (3)	0.2389 (3)	0.0125 (4)
C11	0.29565 (8)	0.65479 (7)	0.20312 (6)	0.01703 (13)
C12	0.74130 (8)	0.74987 (6)	0.06673 (5)	0.01543 (13)
C13	0.59006 (8)	0.75682 (6)	0.42002 (5)	0.01600 (13)
N1	0.7401 (3)	0.4147 (2)	0.25726 (18)	0.0123 (3)
N2	0.8631 (3)	0.1002 (2)	0.23866 (19)	0.0167 (4)
H2A	0.8713	-0.0180	0.2149	0.020*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
C1	0.0168 (10)	0.0137 (9)	0.0155 (10)	-0.0052 (8)	-0.0020 (8)	-0.0020 (7)
C2	0.0149 (10)	0.0153 (9)	0.0174 (10)	-0.0036 (8)	-0.0036 (8)	0.0018 (7)
C3	0.0124 (9)	0.0151 (9)	0.0150 (9)	-0.0039 (7)	-0.0036 (7)	0.0006 (7)
B1	0.0111 (10)	0.0138 (10)	0.0137 (10)	-0.0050 (8)	-0.0024 (8)	-0.0013 (8)
C11	0.0119 (2)	0.0155 (2)	0.0243 (3)	-0.00375 (17)	-0.00505 (18)	-0.00161 (18)
C12	0.0175 (3)	0.0151 (2)	0.0151 (2)	-0.00724 (18)	-0.00290 (18)	0.00153 (17)
C13	0.0188 (3)	0.0140 (2)	0.0154 (2)	-0.00417 (18)	-0.00315 (18)	-0.00375 (17)
N1	0.0129 (8)	0.0125 (7)	0.0118 (8)	-0.0042 (6)	-0.0023 (6)	-0.0003 (6)
N2	0.0203 (9)	0.0106 (8)	0.0194 (9)	-0.0050 (6)	-0.0022 (7)	-0.0024 (6)

Geometric parameters (\AA , $^\circ$)

C1—N2	1.327 (3)	C3—H3	0.9500
C1—N1	1.332 (2)	B1—N1	1.543 (3)
C1—H1	0.9500	B1—C11	1.847 (2)
C2—C3	1.346 (3)	B1—C13	1.848 (2)
C2—N2	1.378 (3)	B1—C12	1.865 (2)
C2—H2	0.9500	N2—H2A	0.8800
C3—N1	1.389 (2)		

N2—C1—N1	108.82 (18)	C11—B1—C13	110.88 (11)
N2—C1—H1	125.6	N1—B1—C12	109.32 (14)
N1—C1—H1	125.6	C11—B1—C12	109.35 (11)
C3—C2—N2	106.08 (17)	C13—B1—C12	109.11 (11)
C3—C2—H2	127.0	C1—N1—C3	107.41 (16)
N2—C2—H2	127.0	C1—N1—B1	126.94 (16)
C2—C3—N1	108.22 (17)	C3—N1—B1	125.62 (16)
C2—C3—H3	125.9	C1—N2—C2	109.46 (16)
N1—C3—H3	125.9	C1—N2—H2A	125.3
N1—B1—C11	108.73 (13)	C2—N2—H2A	125.3
N1—B1—C13	109.43 (13)		
N2—C2—C3—N1	0.1 (2)	C12—B1—N1—C1	-97.5 (2)
N2—C1—N1—C3	-0.3 (2)	C11—B1—N1—C3	-160.49 (15)
N2—C1—N1—B1	177.77 (17)	C13—B1—N1—C3	-39.2 (2)
C2—C3—N1—C1	0.1 (2)	C12—B1—N1—C3	80.2 (2)
C2—C3—N1—B1	-177.98 (17)	N1—C1—N2—C2	0.4 (2)
C11—B1—N1—C1	21.8 (2)	C3—C2—N2—C1	-0.3 (2)
C13—B1—N1—C1	143.04 (17)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
N2—H2A \cdots C12 ⁱ	0.88	2.57	3.3696 (19)	152
N2—H2A \cdots C11 ⁱⁱ	0.88	2.86	3.429 (2)	124
C2—H2 \cdots C13 ⁱⁱ	0.95	2.87	3.815 (2)	171

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