C2	0.7386 (3)	0.0100 (2)	0.1980 (4)	0.0564 (8)
C3	0.8178 (2)	0.0189 (2)	0.3396 (3)	0.0550 (8)
C4	0.9242 (3)	0.2936 (2)	0.4346 (3)	0.0550 (8)
C5	0.8170 (3)	0.2995 (2)	0.4017 (3)	0.0539 (8)

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

Ni1—S1	2.6014 (8)	N1—C1	1.148 (3)
Ni1—N1	2.055 (2)	N2—C2	1.473 (4)
Ni1—N2	2.078 (2)	N3—C3	1.486 (4)
Ni1—N3	2.113 (2)	N4—C4	1.471 (4)
Ni1—N4	2.087 (2)	N5—C5	1.462 (4)
Ni1—N5	2.115 (2)	C2—C3	1.503 (5)
S1—C1'	1.630 (2)	C4—C5	1.506 (5)
S1—Ni1—N1	91.56 (7)	N3—Ni1—N5	92.5 (1)
S1—Ni1—N2	88.44 (8)	N4—Ni1—N5	82.4 (1)
S1—Ni1—N3	86.50 (7)	Ni1—S1—C1'	102.43 (9)
S1—Ni1—N4	91.23 (7)	Ni1—N1—C1	162.5 (2)
S1—Ni1—N5	173.47 (7)	Ni1—N2—C2	109.0 (2)
N1—Ni1—N2	92.5 (1)	Ni1—N3—C3	106.7 (2)
N1—Ni1—N3	174.9 (1)	Ni1—N4—C4	109.4 (2)
N1—Ni1—N4	89.4 (1)	Ni1—N5—C5	107.2 (2)
N1—Ni1—N5	89.97 (9)	S1'—C1—N1	179.1 (2)
N2—Ni1—N3	82.8 (1)	N2—C2—C3	108.5 (3)
N2—Ni1—N4	178.06 (9)	N3—C3—C2	108.7 (3)
N2—Ni1—N5	97.8 (1)	N4—C4—C5	109.3 (3)
N3—Ni1—N4	95.29 (9)	N5—C5—C4	109.4 (3)

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

Table 3. *Hydrogen-bonding distances* (Å)

D—H···A	H···A	D—H···A	H···A
N2—H7···F5	2.45 (4)	N3—H2···F3'''	2.81 (4)
N2—H7···F7	2.65 (3)	N3—H2···F4	2.58 (3)
N2—H8···F6'	2.79 (4)	N4—H10···F6''	2.52 (3)
N2—H8···F7'	2.62 (3)	N5—H15···F1'	2.93 (3)
N3—H1···F2''	2.70 (3)	N5—H16···F3'''	2.32 (3)
H···A	H···A	H···A	H···A
H1···S1''	2.97 (3)	H9···S1''	2.83 (3)

Symmetry codes: (i) $1 - x, -y, -z$; (ii) $x, -y, z + \frac{1}{2}$; (iii) $2 - x, y, \frac{1}{2} - z$; (iv) $x - \frac{1}{2}, y - \frac{1}{2}, z$; (v) $x + \frac{1}{2}, \frac{1}{2} - y, z + \frac{1}{2}$; (vi) $2 - x, y, \frac{1}{2} - z$.

The structure was solved by direct methods and developed by alternating cycles of difference Fourier syntheses and full-matrix least-squares refinements. The positions of the atoms were determined unequivocally. The non-H atoms were refined anisotropically, while the H atoms were refined isotropically.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1994). Cell refinement: *CAD-4 Software*. Data reduction: *TEXSAN* (Molecular Structure Corporation, 1997). Program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1993). Program(s) used to refine structure: *TEXSAN*. Software used to prepare material for publication: *TEXSAN*.

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Bis(dimethylammonium) Hexachlorotellurate(IV)

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Abstract

The title compound, $[(CH_3)_2NH_2]_2[TeCl_6]$, is isostructural with $[(CH_3)_2NH_2]_2[SnCl_6]$ and the high-density form of $[(CH_3)_2NH_2]_2[PtCl_6]$. It consists of a slightly distorted $[TeCl_6]^{2-}$ octahedron and $[(CH_3)_2NH_2]^+$ ions connected by a weak bifurcated hydrogen bond.

Comment

$[(CH_3)_2NH_2]_2[TeCl_6]$, (I), was reported to be isomorphous with $[(CH_3)_2NH_2]_2[SnCl_6]$ (Ben Ghozlen & Daoud, 1981; Knop *et al.*, 1983) from X-ray powder diffraction data taken at room temperature (Ishida *et al.*, 1984). The phase transitions and the molecular motions of the cation and anion were studied by ^{1}H NMR (Ishida *et al.*, 1984), ^{35}Cl NQR (Horiuchi *et al.*, 1985; Ishikawa *et al.*, 1989) and ^{2}H NMR (Ikeda *et al.*, 1989) under the assumption that the Te salt has the same atomic arrangement as the Sn salt. The 180° flip motion of the cation about its pseudo-C_2 axis and the 90° re-orientational jumps of the anion around the Te—Cl bond axis were found to occur at room temperature in the Te salt with smaller activation energies than those determined in the Sn salt. This implies that the ions in the Te salt are bound more loosely than in the Sn salt.

(I)
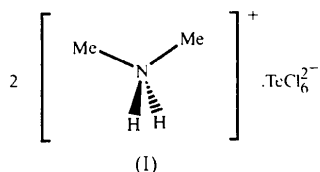
The present X-ray study showed that the Te salt is isostructural with both the Sn salt and the dense form of [(CH₃)₂NH₂]₂[PtCl₆] (Ries, 1902; Khodadad et al., 1984). It consists of parallel sheets of slightly distorted [TeCl₆]²⁻ octahedra which are held together in a sheet by weak bifurcated hydrogen bonds formed between the [(CH₃)₂NH₂]⁺ groups and the Cl atoms (Fig. 1). The shortest contact distances between the cation and the anion are N···Cl(2) [3.449 (8) Å] and N···Cl(2ⁱ) [3.553 (8) Å] which are slightly longer than those reported for the Sn salt, being consistent with the NMR and NQR results: N···Cl(2) 3.449 (8), H···Cl(2) 2.69 Å, N—H···Cl(2) 137°; N···Cl(2ⁱ) 3.553 (8), H···Cl(2ⁱ) 2.84 Å, N—H···Cl(2ⁱ) 133° [symmetry code: (i) −x, −y, z].
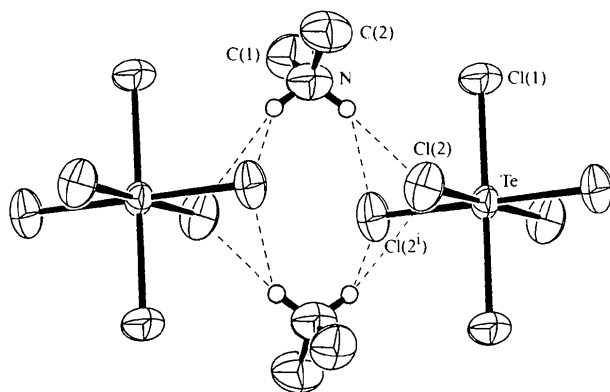

Fig. 1. An ORTEPII (Johnson, 1976) perspective view and atomic numbering. Displacement ellipsoids are drawn at the 50% probability level. The N—H···Cl-type hydrogen bonds are indicated by broken lines. [Symmetry code: (i) −x, −y, z.]

Experimental

Crystals of [(CH₃)₂NH₂]₂[TeCl₆] were grown by slow evaporation from a concentrated HCl solution containing stoichiometric amounts of (CH₃)₂NH₂Cl and TeO₂.

Crystal data

(C₂H₈N)₂[TeCl₆]
$M_r = 432.50$
Orthorhombic
Pnnm
$a = 7.410 (4)$ Å
$b = 14.392 (1)$ Å
$c = 7.368 (4)$ Å
$V = 785.7 (8)$ Å³
$Z = 2$
$D_x = 1.828$ Mg m⁻³
D_m not measured

Mo Kα radiation
$\lambda = 0.71073$ Å
Cell parameters from 25 reflections
$\theta = 11.9–12.5°$
$\mu = 2.883$ mm⁻¹
$T = 291$ K
Prismatic
$0.23 \times 0.23 \times 0.20$ mm
Yellow

Data collection

Rigaku AFC-5R diffractometer
$\omega–2\theta$ scans
Absorption correction:
  ψ scan (North et al., 1968)
  $T_{min} = 0.45$, $T_{max} = 0.56$
1089 measured reflections
1089 independent reflections

605 reflections with $I > 3\sigma(I)$
$\theta_{max} = 27.5°$
$h = 0 \rightarrow 9$
$k = 0 \rightarrow 18$
$l = 0 \rightarrow 9$
3 standard reflections
  every 97 reflections
  intensity decay: 1%

Refinement

Refinement on F
$R = 0.033$
$wR = 0.032$
$S = 2.65$
605 reflections
39 parameters
H atoms not refined
$w = 1/[\sigma^2(F_o) + 0.00002|F_o|^2]$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 0.62$ e Å⁻³
$\Delta\rho_{min} = -0.41$ e Å⁻³
Extinction correction:
  Zachariasen (1967)
Extinction coefficient:
  6.5 (8) × 10⁻⁷
Scattering factors from
  International Tables for
  Crystallography (Vol. C)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters (Å²)

$$U_{eq} = (1/3)\Sigma_i\Sigma_j U^{ij}a^i a^j \mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	U_{eq}
Te	0	0	0	0.0422 (1)
Cl(1)	0.1841 (3)	0.1481 (1)	0	0.0782 (6)
Cl(2)	0.1957 (1)	−0.06638 (8)	0.2486 (2)	0.0725 (4)
N	0.1906 (8)	0.1358 (4)	1/2	0.075 (2)
C(1)	0.103 (1)	0.2271 (4)	1/2	0.081 (2)
C(2)	0.389 (1)	0.1357 (5)	1/2	0.090 (3)

Table 2. Selected geometric parameters (Å, °)

Te—Cl(1)	2.531 (3)	N—C(1)	1.46 (1)
Te—Cl(2)	2.524 (2)	N—C(2)	1.47 (1)
Cl(1)—Te—Cl(2)	90.52 (7)	Cl(2)—Te—Cl(2ⁱⁱ)	93.06 (9)
Cl(1)—Te—Cl(2ⁱ)	89.48 (7)	C(1)—N—C(2)	116 (1)
Cl(2)—Te—Cl(2ⁱ)	86.94 (9)		

Symmetry codes: (i) −x, −y, z; (ii) x, y, −z.

H atoms could not be located from a difference Fourier map. H atoms attached to N were placed in geometrically ideal positions with N—H = 0.95 Å, but were not refined. The methyl H atoms were not included because these may be disordered around the C—N bond.

Data collection: MSC/AFC Diffractometer Control Software (Molecular Structure Corporation, 1988). Cell refinement: MSC/AFC Diffractometer Control Software. Data reduction: TEXSAN (Molecular Structure Corporation, 1997). Program(s) used to solve structure: SIR92 (Altomare et al., 1993). Program(s) used to refine structure: TEXSAN. Software used to prepare material for publication: TEXSAN.

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Comment

In metalloborane chemistry, ligand-exchange reactions at the metal centre of a compound present a reasonably viable way to form new metalloborane compounds (Siedle & Todd, 1976). An analysis of the literature shows that, in such processes, the leaving groups are often phosphine ligands (PPh_3 or PMe_2Ph), while the substituents could be such ligands as PMe_3 (Bown et al., 1987), PMe_2Ph (Kennedy, 1986), $Ph_2PCH_2PPh_2$ (Elrington et al., 1984), $C_2S_2(CN)_2^{2-}$, CN^- (Siedle & Todd, 1976) and $SCSNEt_2$ (Beckett et al., 1985). Siedle & Todd (1976) published the synthesis of $[(phen)PdB_{10}H_{12}]$ via a ligand-displacement reaction, which was characterized by spectroscopic methods (Siedle & Todd, 1976). No ruthenium analogue has so far been reported. We report here the first phen-ligated closo-ruthenaborane compound, (I), the structure of which was established by X-ray diffraction.

(I)
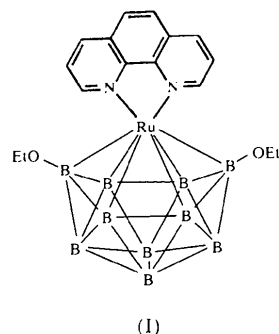
Compound (I) (Fig. 1) contains an $\{RuB_{10}H_8(OC_2H_5)_2\}$ 11-vertex metalloborane cluster, with the Ru atom lying above the boat-type face of six B atoms. Such a cluster is very similar to $[(PPh_3)_2RuB_{10}H_8(OC_2H_5)_2]$, (II) (Crook et al., 1984, 1985), and $[(PMe_3)(PPh_3)RuB_{10}H_8(OC_2H_5)_2]$, (III) (Bown et al., 1987). However, the Ru—B bond lengths in (I) [2.014 (4)–2.326 (3) Å] are shorter than those reported for both (II) [2.041 (8)–2.402 (8) Å] and (III) [2.032 (5)–2.374 (5) Å]. This indicates that the coordination of the phen ligand strengthens slightly the metal–boron bonding within the present cluster compared with the phosphines.

The bidentate phen ligand coordinates to the Ru atom with Ru—N distances of 2.117 (2) and 2.128 (2) Å. These bonds are significantly longer than the corresponding Ru—N_{phen} bond lengths in the literature, for example, 2.056 (8) and 2.082 (9) Å for [Ru(bipy)$_2$(phen)]$^{2+}$ (Ye et al., 1995), where bipy is 2,2′-bipyridine, 2.096 (5)–2.100 (5) Å for [Ru(phen)$_2$(py)$_2$]$^{2+}$ (Bonneson et al., 1983), where py is pyridine, and an average of 2.063 (4) Å for [Ru(phen)$_3$]$^{2+}$ (Breu & Stoll, 1996). The relative elongation of the Ru—N bond lengths can be attributed to the influence of the $\{RuB_{10}\}$ cage. In compound (I), both the N—Ru—N bite angle [78.26 (8)°]

Acta Cryst. (1998). C54, 1813–1815

A 1,10-Phenanthroline-Ligated closo-Ruthenaundecaborane: [(C12H8N2)RuB10H8(OC2H5)2]

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Abstract

In the title compound, 2,3-diethoxy-1-(1,10-phenanthroline-N,N′)-1-ruthena-closo-undecaborane, [Ru(C4H18B10O2)(C12H8N2)], the Ru atom is bound to two N atoms of the 1,10-phenanthroline (phen) ligand and six B atoms of the closo 11-vertex $\{RuB_{10}\}$ cage. The Ru—N bond distances are 2.117 (2) and 2.128 (2) Å, and the N1—Ru1—N10 bite angle is 78.26 (8)°.