Hexagonal
$P\bar{6}2m$
a = 6,878 (1) Å
c = 4,417 (1) Å
V = 180,96 (6) Å^3
Z = 1
D_x = 2,747 Mg m^{-3}

Gitterparameter aus 25 Reflexen
θ = 5-25°
μ = 6,927 mm^{-1}
T = 293 (2) K
0,78 × 0,28 × 0,14 mm
Farblos

Datensammlung

Enraf-Nonius CAD-4 Diffraktometer
ω-Abtastung
Absorptionskorrektur: numerisch, *SHELX*76 (Sheldrick, 1976)
$T_{\min}$ = 0,12, $T_{\max}$ = 0,39
1252 gemessene Reflexe
141 unabhängige Reflexe
141 beobachtete Reflexe [$I > 2\sigma(I)$]

$R_{\rm int}$ = 0,109
$\theta_{\max}$ = 24,90°
$h = -8 \rightarrow 8$
$k = -8 \rightarrow 8$
$l = -5 \rightarrow 5$
2 Kontrollreflexe gemessen nach je 250 Reflexen
Häufigkeit: 120 min

Verfeinerung

Verfeinerung auf F^2
$R(F)$ = 0,0222
$wR(F^2)$ = 0,0677
S = 1,352
141 Reflexe
11 Parameter
Berechnetes Wichtungsschema
$w = 1/[\sigma^2(F_o^2) + (0{,}0388P)^2]$ mit $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max}$ = 0,013

$\Delta\rho_{\max}$ = 0,626 e Å^{-3}
$\Delta\rho_{\min}$ = −0,813 e Å^{-3}
Extinktionskorrektur: nein
Atomformfaktoren aus *International Tables for Crystallography* (1992, Bd. C, Tabelle 4.2.6.8 und 6.1.1.4)

Tabelle 1. *Fraktionelle Atomkoordinaten und isotrope Äquivalente der Verschiebungsfaktoren* (Å^2)

$$U_{\rm äq} = \tfrac{1}{3}\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	$U_{\rm äq}$
La	0	0	0	0,0146 (4)
Cl	0,2910 (5)	x	1/2	0,0326 (9)
O	0,365 (2)	0	0	0,055 (5)

Tabelle 2. *Geometrische Parameter* (Å, °)

La—O	2,51 (1)	La—Cl	2,980 (2)
O$^{\rm i}$—La—O	120,0	Cl—La—Cl$^{\rm iv}$	71,11 (8)
O$^{\rm ii}$—La—Cl	132,18 (5)	Cl—La—Cl$^{\rm v}$	95,64 (10)
O—La—Cl	70,38 (2)	La—Cl—La$^{\rm vi}$	95,64 (10)
Cl—La—Cl$^{\rm iii}$	140,77 (4)		

Symmetriebezeichnunge: (i) y, x, z; (ii) $-x, -x+y, z$; (iii) $-x, -x+y, z-1$; (iv) $-y, x-y, z$; (v) $x, y, z-1$; (vi) $x, y, 1+z$.

Datensammlung: *CAD*-4 *Software* (Enraf-Nonius, 1992). Gitterverfeinerung: *CELDIM* (Enraf-Nonius, 1992). Datenreduktion: *XCAD4PC* (Harms, 1993). Lösung der Struktur mit Programm: *SHELXS*86 (Sheldrick, 1985). Verfeinerung der Struktur mit Programm: *SHELXL*93 (Sheldrick, 1993). Zeichenprogramm: *SCHAKAL*92 (Keller, 1993). Programme zur Berechnung der geometrische Datan und zur Erstellung der Tabellen: *SHELXL*93.

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Die Liste der Strukturfaktoren, anisotropen Verschiebungsfaktoren, und vollständigen geometrischen Daten sind bei dem IUCr (Aktenzeichen: SE1038) hinterlegt. Kopien sind erhältich durch: The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Tetraammineplatinum(II) Hexachlorostannate(IV), $[Pt(NH_3)_4][SnCl_6]$

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Abstract

A crystal of the title complex is built up from square $[Pt(NH_3)_4]^{2+}$ cations and octahedral $SnCl_6^{2-}$ anions. In the pseudo-cubic unit cell, the complex ions occupy the positions of the NaCl structure type. As well as coulombic forces, packing involves weak

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hydrogen bonds of the N—H···Cl type, which determine the orientation of the ammonia ligands.

Comment

The present crystal structure determination forms part of a structural study on new bimetallic precursors of reforming catalysts (Garnier, 1993; Michelot, Ouali, Blais, Guérin & Kappenstein, 1988).

The structure is ionic and is formed by square $[Pt(NH_3)_4]^{2+}$ cations and octahedral $SnCl_6^{2-}$ anions which occupy the ion positions of the NaCl structure type in a pseudo-cubic unit cell. Thus, the coordination of the ions is 6/6, as found in similar bimetallic complexes: $[Ir(NH_3)_5Cl][PtCl_4]$ (Garnier & Bele, 1993) and $[Pt(NH_3)_4][Ir(H_2O)Cl_5].2H_2O$ (Garnier & Bele, 1994).

A search of the Cambridge Structural Database (Allen *et al.*, 1992) showed that the structures of ten complexes containing the $Pt(NH_3)_4^{2+}$ cation and 60 complexes containing the $SnCl_6^{2-}$ anion have been reported. The observed Pt—N and Sn—Cl bond distances (Table 2) are in agreement with those found in related complexes, *e.g.* $[Pt(NH_3)_4][PtCl_3(OSMe_2)]_2$ [Pt—N = 2.05 (1) Å (Khodadad & Rodier, 1987)] and $[H_3N(CH_2)_2NH_3][SnCl_6]$ [Sn—Cl 2.422 (3)–2.433 (3) Å (Abriel, 1986)]. The angles are as expected. Apart from the coulombic interactions, hydrogen bonds of the type N—H···Cl contribute to the packing forces and orientation of the ions. A similar hydrogen-bonding system was found in the $[Ir(NH_3)_5Cl][PtCl_6]Cl_2$ complex, but with shorter N···Cl distances [3.200 (7)–3.436 (5) Å (Garnier, 1993)], showing that in the title complex the hydrogen-bond system is weaker. Only the Cl(1) atoms are involved in hydrogen-bond formation. The Cl(2) atoms remain free from such hydrogen bonding and as a consequence their displacement ellipsoids are greater.

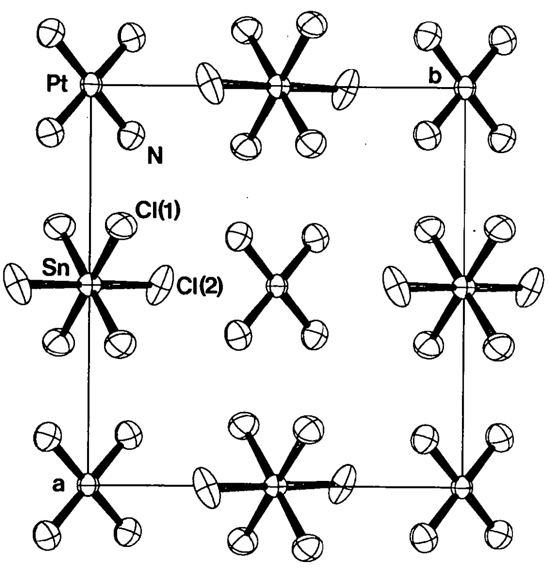

Fig. 1. A *MACORTEP* (Michalowicz & André, 1991) view of the packing of the ions in the *ab* plane. Displacement ellipsoids are represented at the 50% probability level.

Experimental

The title complex was prepared from the reaction between $[Pt(NH_3)_4]Cl_2.H_2O$ and $SnCl_4.5H_2O$ by crystallization in gel formed from sodium metasilicate, Na_2SiO_3.

Crystal data

$[Pt(NH_3)_4][SnCl_6]$
$M_r = 594.58$
Orthorhombic
Cmca
$a = 11.701$ (1) Å
$b = 11.067$ (1) Å
$c = 10.550$ (1) Å
$V = 1366.2$ (2) Å^3
$Z = 4$
$D_x = 2.892$ Mg m^{-3}
$D_m = 2.91$ Mg m^{-3}
D_m measured by flotation

Mo $K\alpha$ radiation
$\lambda = 0.7107$ Å
Cell parameters from 25 reflections
$\theta = 5.63$–$9.96°$
$\mu = 13.102$ mm^{-1}
$T = 295$ K
Bipyramid
0.320 × 0.284 × 0.232 mm
Colourless

Data collection

CAD-4L diffractometer
ω scans
Absorption correction: empirical (*ABSORB*; Ugozzoli, 1987)
$T_{min} = 0.831$ (θ), 0.943 (φ); $T_{max} = 1.174$ (θ), 1.532 (φ)
3769 measured reflections
1930 independent reflections
1147 observed reflections [$F_o \geq 6(F_o)$]

$R_{int} = 0.037$
$\theta_{max} = 38°$
$h = 0 \rightarrow 20$
$k = 0 \rightarrow 19$
$l = -18 \rightarrow 18$
2 standard reflections monitored every 100 reflections intensity variation: −8.4%

Refinement

Refinement on *F*
$R = 0.0361$
$wR = 0.0462$
$S = 2.26$
1147 reflections
40 parameters
Only H-atom *U*'s refined
$w = 1/\sigma^2(F_o)$
$(\Delta/\sigma)_{max} = 0.003$
$\Delta\rho_{max} = 2.9$ e Å^{-3}
$\Delta\rho_{min} = -2.6$ e Å^{-3}

Extinction correction: isotropic empirical (*SHELX76*; Sheldrick, 1976)
Extinction coefficient: 0.30 (3) × 10^{-7}
Atomic scattering factors from *International Tables for X-ray Crystallography* (1974, Vol. IV) (Pt) and *SHELX76* (Sn, Cl, N, H)

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

$$B_{eq} = (8\pi^2/3)\Sigma_i\Sigma_j U_{ij}a_i^*a_j^*\mathbf{a}_i.\mathbf{a}_j.$$

	x	y	z	B_{eq}
Pt	0	0	0	1.84 (1)
Sn	1/2	0	0	1.96 (1)
Cl(1)	0.3537 (1)	0.0875 (2)	0.1355 (2)	3.13 (3)
Cl(2)	1/2	0.1825 (2)	−0.1271 (2)	3.64 (6)
N	0.1227 (5)	0.1032 (5)	−0.0842 (5)	3.07 (2)

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

Pt—N	2.038 (5)	Sn—Cl(2)	2.424 (1)
Sn—Cl(1)	2.431 (1)		
N—Pt—N^i	90.46 (3)	Cl(1)—Sn—Cl(1^{ii})	89.52 (6)
Cl(1)—Sn—Cl(2)	89.62 (5)		

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

Table 3. *Hydrogen-bonding geometry* (Å, °)

D	H	*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N	H(1)	Cl(1^i)	1.010	2.692 (6)	3.644 (6)	157.2 (6)
N	H(2)	Cl(1)	1.010	2.556 (6)	3.565 (6)	177.0 (6)
N	H(3)	Cl(1^{ii})	1.010	2.545 (6)	3.477 (6)	153.2 (6)

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

Data reduction: *LOPOTRI* (Gravereau, 1982). Program(s) used to solve structure: *SHELXS*86 (Sheldrick, 1985). Program(s) used to refine structure: *SHELX*76 (Sheldrick, 1976). Geometric analysis: *PARST* (Nardelli, 1983). At first, the structure was refined in a non-centrosymmetric space group *Aba*2, but during the refinement a strong correlation between the parameters of the centrosymmetrically related atoms was observed; the space group then became centrosymmetric. The absorption correction was performed using the *ABSORB* program (Ugozzoli, 1987) following the method of Walker & Stuart (1983). In the final difference map some high peaks were observed near the heavy atoms as a consequence of the inadequacy of the absorption correction. The H atoms of NH_3 were refined as a rigid group with fixed distances (N—H = 1.01 and H···H = 1.649 Å); the common U_{iso} of the H atoms refined to 9 (2) $Å^2$.

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Lists of structure factors, anisotropic displacement parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71792 (5 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: DU1058]

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