

unique (including 1194, $F^2 > 3\sigma$), $R_{int} = 0.057$; structure solved by Patterson and Fourier methods; refined on F , 99 parameters, H atoms not included, anisotropic thermal parameters; $R = 0.072$ (2045 data), $R = 0.027$ [$F^2 > 3\sigma(F^2)$], $wR = 0.035$, $S = 1.24$, $w = 4F^2[\sigma(F^2) + (0.04F^2)^2]^{-1}$; max. (shift/ σ) = 0.01; empirical extinction correction, $F_{corr} = (1 + 9.5 \times 10^{-7})^{-1}$; max. and min. of ΔF synthesis 0.9 and $-1.2 e \text{ \AA}^{-3}$. Atomic scattering factors and anomalous-dispersion terms were taken from *International Tables for X-ray Crystallography* (1974). Local unpublished programs and ORTEP (Johnson, 1965) used. Atomic parameters are listed in Table 1. A list of selected bond distances and angles is given in Table 2. An ORTEP view of the molecule is shown in Fig. 1.*

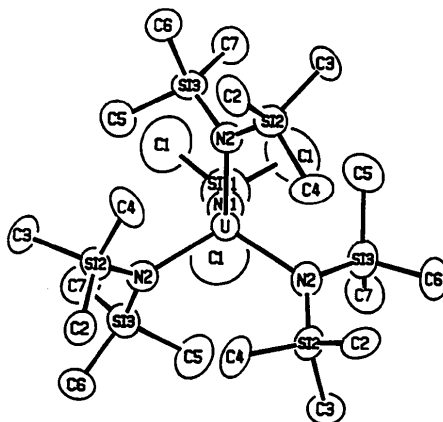


Fig. 1. ORTEP drawing (Johnson, 1965) of the molecule looking down and slightly off the threefold axis.

Discussion. The U–N(1)–Si(1) angle is required by the space group to be linear. The U–N(1) distance is 1.91 (2) Å and is substantially shorter than that found in $(\text{MeC}_5\text{H}_4)_3\text{U}(\text{NC}_6\text{H}_5)$ of 2.019 (6) Å (Brennan & Andersen, 1985), where the U–N–C angle is slightly non-linear [167.4 (6)°]. Clearly there is a profound change in the geometrical parameters upon replacing the methylcyclopentadienyl groups with the bis(trimethylsilyl)amido groups in molecules of the same general stereochemistry, $L_3\text{UL}'$. The U–N(2) bond length is ca 0.4 Å longer than U–N(1), while the Si–N distances in the imido and amido ligands are nearly

equal, as are the Si–C distances. The N(2) atom is 0.07 Å from the plane consisting of U, Si(3) and Si(4).

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* Lists of anisotropic thermal parameters and structure factors have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 44989 (12 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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Structure of a Styrylbenzothiazole Platinum(II) Complex: $[\text{Net}_4][\text{PtBr}_3(\text{asb})]$

BY GRISELL M. GOMEZ, MARIEL M. MUIR, JAMES A. MUIR AND OSVALDO COX

Departments of Chemistry and Physics, University of Puerto Rico, Rio Piedras, PR 00931, USA

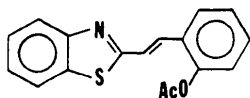
(Received 22 December 1987; accepted 6 May 1988)

Abstract. Tetraethylammonium tribromo[2-(2-acetoxystyryl)benzothiazole]platinate(II), $[(\text{C}_2\text{H}_5)_4\text{N}][\text{PtBr}_3(\text{C}_{17}\text{H}_{13}\text{NO}_2\text{S})]$, $M_r = 860.4$, monoclinic, $P2_1/c$, $a = 11.230$ (9), $b = 19.333$ (4), $c = 13.685$ (6) Å, $\beta = 101.06$ (4)°, $V = 2916$ (4) Å³, $Z = 4$, $D_x = 1.96$ g cm⁻³, $\lambda(\text{Mo K}\alpha) = 0.71073$ Å, $\mu = 90.3$ cm⁻¹, $F(000) = 1648$, $T = 296$ K, final $R = 0.047$ for 3384

unique observed reflections. The $[\text{PtBr}_3(\text{asb})]^-$ unit has square-planar geometry about the Pt, with the asb coordinated to the Pt through the N of the thiazole ring and a Pt–N bond distance of 2.010 (8) Å. The average Pt–Br distance is 2.426 (7) Å. The ligand is non-planar with a dihedral angle of 22.4 (7)° between the benzothiazole and the acetoxystyrene rings. The

dihedral angles between the platinum coordination plane and the benzothiazole and benzene rings are 85.7 (1) and 71.3 (3)° respectively.

Introduction. *cis*-Diamminedichloroplatinum(II), *cis*-Pt(NH₃)₂Cl₂, has become one of the most widely used antitumor compounds in cancer treatment. Use of *cis*-platin has been limited, however, by side effects such as nephrotoxicity. Many complexes have been synthesized in an effort to obtain improved anticancer activity, less toxicity and higher water solubility (Hacker, Douple & Krakoff, 1984). Among the ligands which have been used for the synthesis of new complexes are *N*-heterocycles such as imidazole, thiazole and benzoxazole (Ponticelli, Biddau, Zakharova & Tatjanenko, 1987; Massacesi, Pinna, Biddau, Ponticelli & Zakharova, 1983; Van Kralingen, De Ridder & Reedijk, 1979). Some Schiff bases derived from thiazoles and benzothiazoles (Dash, Patra & Praharaj, 1980) and several derivatives of styryl-benzothiazole (Cox, Jackson, Vargas, Baez, Colon, Gonzalez & de Leon, 1982) have shown biological activity. The asb ligand [where asb is 2-(2-acetoxystyryl)benzothiazole, shown below] is potentially ambidentate with either N or S available for coordination. The complex [PtBr₃(asb)]⁻ has been synthesized and its crystal and molecular structures have been determined. Structures of several analogous platinum complexes have been determined in an effort to understand better the structure-activity relationship of platinum complexes with antitumor activity. This complex was the least active in preliminary screening against CHO-K tumor cells.



Experimental. An acetone solution (10 mL) of 0.053 g (0.178 mmol) of 2-(2-acetoxystyryl)benzothiazole was added slowly to 10 mL of an acetone solution of 0.100 g (0.088 mmol) of [NEt₄]₂[Pt₂Br₆] (Livingstone & Whitley, 1962). The mixture was stirred and heated for 30 min and left to evaporate at room temperature. Orange crystals were obtained from the mother liquor in 88% yield.

Orange prismatic crystal, D_m not determined, 0.40 × 0.40 × 0.30 mm, Enraf-Nonius CAD-4 diffractometer, graphite-monochromated Mo K α , ω - θ scan, scan width 0.9 + 0.34tan θ °, 2 θ_{max} = 53°, lattice parameters from least-squares fit of setting angles for 24 reflections in the range 12 < θ < 14°. Systematic absences 0k0 ($k = 2n + 1$), and $h0l$ ($l = 2n + 1$); $\pm h - k + l$ with $|h| \leq 14$, $-k \leq 24$, $l \leq 17$; three standard reflections, intensities constant within 2.3% after 76.2 h, an anisotropic correction for decay was applied;

total unique reflections measured 6217, 3384 with $F^2 > 2\sigma(F_o)^2$, $\sigma(F_o) = \{[\sigma(I_{raw})^2 + 0.04F_o^2]\}^{1/2}/2F$. Absorption corrections from ψ scans of high χ -value reflections, relative transmission factors 0.607 to 0.995. Structure was solved with the Enraf-Nonius *Structure Determination Package* (Frenz, 1986), heavy-atom and difference-Fourier methods; isotropic then anisotropic refinement on F of all non-hydrogen atoms, H atoms at calculated positions [C-H 0.95 Å, $U(H) = 1.3U(C)$]; 308 parameters refined, $wR = 0.056$, $w = 1/[\sigma(F_o)]^2$, $S = 1.2$; difference syntheses showed no densities above 0.83 (min. -0.55 e Å⁻³); max $\Delta/\sigma = 0.01$.

Neutral-atom scattering factors were used, except for Pt^{II} [all from *International Tables for X-ray Crystallography* (1974)], and were corrected for anomalous dispersion (Cromer & Liberman, 1970). An extinction parameter which refined to $x = 1.65 \times 10^{-7}$ was applied and the calculated structure factor became $F_c = 1/(1 + xI_o)$.

Discussion. The atomic coordinates and equivalent isotropic temperature factors are listed in Table 1, and bond lengths and angles in Table 2.* An ORTEP plot (Johnson, 1976) of the title compound with the atom numbering is shown in Fig. 1, and the packing of the molecules (Motherwell & Clegg, 1976) in Fig. 2.

The [PtBr₃(asb)]⁻ unit has square-planar geometry about the Pt with no atom deviating from the least-squares coordination plane by more than 0.02 (1) Å. The asb molecule, a potentially ambidentate ligand, with either the N or S available for coordination, bonds to the Pt through the N of the benzothiazole ring. The Pt-N and average Pt-Br bond distances are 2.010 (8) and 2.426 (7) Å respectively, which are typical for Pt^{II} complexes. No clear evidence for a *trans* influence could be noted in the Pt-Br distances. In previous similar structures the Pt-N bond distance which is *trans* to the Br atom varies from 1.96 (1) Å, for an acetonitrile complex (Muir, Gomez & Muir, 1986), to 2.01 (1) Å for another styrylbenzothiazole derivative (Muir, Gomez, Muir, Cox & Cadiz, 1987). However, in a diethylenetriamine Pt^{II} complex (Melanson, Rochon, & Hubert, 1979) the Pt-N bond distance ranges from 1.95 (5) to 2.08 (5) Å, with the Pt-N bond distance which is *trans* to the Br atom being the shortest.

The asb ligand is non-planar with a dihedral angle of 22.4 (7)° between the benzothiazole and the acetoxybenzene rings. This non-planarity is considerably smaller than that observed [60.1 (4)°] between the benzothiazole and nitrobenzene rings in the related

* Lists of structure factors, anisotropic thermal factors, H-atom parameters and least-squares planes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51012 (40 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and equivalent isotropic temperature factors (Å²) for [NET₄][Pt(asb)Br₃]

	x	y	z	B _{eq} (Å ²)
Pt	0.16973 (4)	0.39646 (2)	0.36587 (3)	3.034 (8)
Br1	0.0008 (1)	0.36582 (7)	0.2350 (1)	5.53 (3)
Br2	0.2616 (1)	0.46319 (7)	0.24889 (9)	4.68 (3)
Br3	0.0895 (1)	0.33266 (7)	0.4914 (1)	5.07 (3)
S	0.5025 (3)	0.4245 (2)	0.6089 (2)	3.46 (6)
O1	0.4005 (7)	0.2004 (4)	0.3360 (5)	4.0 (2)
O2	0.5459 (7)	0.2388 (5)	0.2607 (6)	5.1 (2)
N1	0.3063 (7)	0.4238 (5)	0.4766 (6)	3.1 (2)
N2	0.0134 (8)	0.3731 (5)	0.8342 (7)	4.0 (2)
C1	0.3054 (8)	0.4843 (5)	0.5306 (7)	2.5 (2)
C2	0.211 (1)	0.5324 (6)	0.5138 (9)	4.1 (3)
C3	0.224 (1)	0.5918 (6)	0.5736 (9)	4.3 (3)
C4	0.326 (1)	0.6006 (7)	0.6487 (9)	4.6 (3)
C5	0.419 (1)	0.5528 (6)	0.6638 (9)	3.9 (3)
C6	0.4058 (9)	0.4931 (5)	0.6052 (8)	3.1 (2)
C7	0.4043 (9)	0.3866 (5)	0.5121 (7)	2.8 (2)
C8	0.4233 (9)	0.3175 (6)	0.4754 (7)	2.9 (2)
C9	0.515 (1)	0.2777 (6)	0.5181 (8)	3.2 (2)
C10	0.5396 (9)	0.2053 (6)	0.4967 (7)	3.2 (2)
C11	0.4897 (9)	0.1690 (6)	0.4080 (9)	3.5 (2)
C12	0.519 (1)	0.0998 (7)	0.397 (1)	5.6 (3)
C13	0.600 (1)	0.0648 (7)	0.469 (1)	5.9 (4)
C14	0.654 (1)	0.0990 (7)	0.5566 (9)	5.4 (3)
C15	0.622 (1)	0.1686 (7)	0.5690 (9)	4.6 (3)
C16	0.442 (1)	0.2374 (7)	0.2651 (8)	4.2 (3)
C17	0.339 (1)	0.2682 (7)	0.1954 (9)	5.3 (3)
C18	0.132 (1)	0.3497 (9)	0.901 (1)	7.2 (4)
C19	0.181 (2)	0.397 (1)	0.987 (1)	12.7 (8)
C20	0.035 (1)	0.4441 (7)	0.795 (1)	6.3 (4)
C21	0.140 (1)	0.4492 (8)	0.739 (1)	7.0 (4)
C22	-0.018 (1)	0.3219 (7)	0.754 (1)	7.1 (4)
C23	-0.138 (2)	0.330 (1)	0.685 (2)	12.3 (7)
C24	-0.086 (1)	0.3798 (8)	0.894 (1)	7.1 (4)
C25	-0.125 (1)	0.3123 (9)	0.934 (1)	8.1 (4)

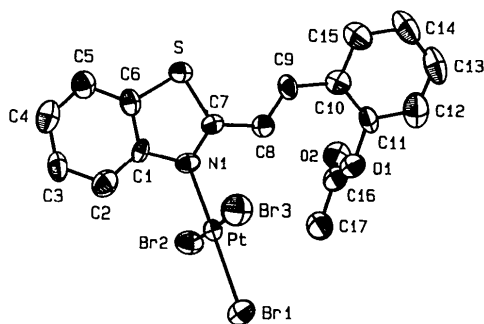
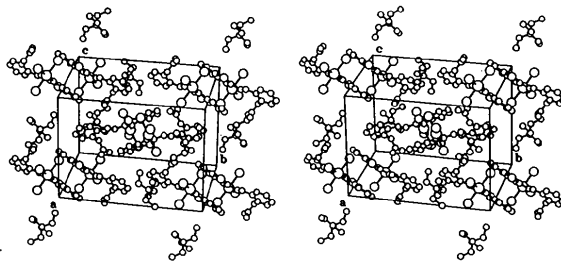
Anisotropically refined atoms are given in the form of the isotropic equivalent displacement parameter defined as $\frac{1}{3}[a^2B(1,1) + b^2B(2,2) + c^2B(3,3) + ab(\cos\gamma)B(1,2) + ac(\cos\beta)B(1,3) + bc(\cos\alpha)B(2,3)]$.

[NET₄][PtBr₃(nsb)] complex, where nsb is 2-(2-chloro-5-nitrostyryl)benzothiazole (Muir, Gomez, Muir, Cox & Cadiz, 1987). In the nsb complex the nitrobenzene rings are stacked in a parallel but staggered form, at a distance which permits π interactions [3.53 (1) Å]. From the packing diagram of the present complex it can be seen that the benzothiazole rings are stacked in a similar fashion, although the closest distance between these rings [7.02 (1) Å] is greater than in the nsb analogue. In the related *cis*-Pt(asb)₂Cl₂ complex (Muir, Gomez, Muir, Cadiz, Cox & Barnes, 1988), however, where no such stacking interactions existed, a dihedral angle of 36(3)° was found. The lack of such interactions was presumably due to the overall geometry of the *cis* complex. It seems, then, that the deviation from planarity in these complexes can be attributed principally to packing forces.

The average C-N, C-S and C-C distances [1.36 (4), 1.714 (6), 1.39 (1) Å] and angles [120 (2), 108 (10)°] for the coordinated asb ligand are similar to those found in a Pd^{II} benzothiazole complex (Churchill, Wasserman & Young, 1980), and in the nsb and *cis*-Pt(asb)₂Cl₂ analogues. The average C-N, C-S and C-C distances found in two uncoordinated styryl-benzothiazole derivatives are 1.34 (5), 1.74 (1) and 1.39 (2) Å (Bernard, 1988). It is evident that upon coordination the \langle C-N \rangle distance increases by 0.02 Å and the \langle C-S \rangle distance decreases by 0.03 Å. These

Table 2. Bond lengths (Å) and angles (°) for [NET₄][Pt(asb)Br₃]

Pt-Br1	2.421 (1)	C3-C4	1.40 (2)
Pt-Br2	2.434 (1)	C4-C5	1.38 (2)
Pt-Br3	2.424 (2)	C5-C6	1.40 (2)
Pt-N1	2.010 (8)	C7-C8	1.46 (2)
S-C6	1.71 (1)	C8-C9	1.33 (1)
S-C7	1.718 (9)	C9-C10	1.47 (2)
O1-C11	1.40 (1)	C10-C11	1.42 (1)
O1-C16	1.36 (1)	C10-C15	1.41 (2)
O2-C16	1.18 (2)	C11-C12	1.39 (2)
N1-C1	1.39 (1)	C12-C13	1.38 (2)
N1-C7	1.33 (1)	C13-C14	1.39 (2)
N2-C18	1.54 (2)	C14-C15	1.40 (2)
N2-C20	1.51 (2)	C16-C17	1.47 (2)
N2-C22	1.47 (2)	C18-C19	1.49 (3)
N2-C24	1.52 (2)	C20-C21	1.52 (2)
C1-C2	1.39 (2)	C22-C23	1.50 (2)
C1-C6	1.38 (1)	C24-C25	1.51 (2)
C2-C3	1.40 (2)		
Br1-Pt-Br2	90.80 (5)	S-C6-C5	128.9 (8)
Br1-Pt-Br3	93.14 (5)	C1-C6-C5	120.6 (9)
Br1-Pt-N1	178.1 (3)	S-C7-N1	114.6 (8)
Br2-Pt-Br3	176.05 (5)	S-C7-C8	122.9 (7)
Br2-Pt-N1	89.9 (3)	N1-C7-C8	122.4 (8)
Br3-Pt-N1	86.1 (3)	C7-C8-C9	122.1 (9)
C6-S-C7	89.9 (5)	C8-C9-C10	128.8 (9)
C11-O1-C16	115.7 (9)	C9-C10-C11	125.6 (9)
Pt-N1-C1	122.8 (6)	C9-C10-C15	117.7 (9)
Pt-N1-C7	126.4 (7)	C11-C10-C15	117 (1)
C1-N1-C7	110.7 (7)	O1-C11-C10	120.0 (9)
C18-N2-C20	107 (1)	O1-C11-C12	119.5 (9)
C18-N2-C22	108 (1)	C10-C11-C12	120 (1)
C18-N2-C24	110.3 (9)	C11-C12-C13	122 (1)
C20-N2-C22	112 (1)	C12-C13-C14	119 (1)
C20-N2-C24	108 (1)	C13-C14-C15	120 (1)
C22-N2-C24	112 (1)	C10-C15-C14	122 (1)
N1-C1-C2	124.0 (8)	O1-C16-O2	122 (1)
N1-C1-C6	114.2 (9)	O1-C16-C17	110 (1)
C2-C1-C6	122 (1)	O2-C16-C17	128 (1)
C1-C2-C3	117.6 (9)	N2-C18-C19	116 (1)
C2-C3-C4	120 (1)	N2-C20-C21	116 (1)
C3-C4-C5	121 (1)	N2-C22-C23	117 (1)
C4-C5-C6	118 (1)	N2-C24-C25	114 (1)
S-C6-C1	110.6 (7)		

Fig. 1. Labeling of atoms in the [PtBr₃(asb)]⁻ anion (50% probability ellipsoids).Fig. 2. Packing diagram of [NET₄][PtBr₃(asb)] molecules in the unit cell.

changes can be attributed to a strong σ Pt—N interaction.

The benzothiazole and acetoxybenzene rings are essentially planar with no atom deviating from the least-squares planes by more than 0.02 (1) Å. The dihedral angles between the PtNBr₃ plane and the benzothiazole and acetoxybenzene planes are 85.7 (1) and 71.3 (3)° respectively. The distances and angles within the tetraethylammonium cation are all normal.

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Structure of Tris(tri-*p*-methoxyphenylphosphine)mercury(II) Diperchlorate

BY TIM ALLMAN AND RAM G. GOEL

Department of Chemistry, University of Guelph, Guelph, Ontario, Canada N1G 2W1

AND ANDRÉ L. BEAUCHAMP

Département de Chimie, Université de Montréal, CP 6128, Succ. A, Montréal, Québec, Canada H3C 3J7

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Abstract. [Hg{(C₇H₇O)₃P₃}(ClO₄)₂], $M_r = 1456.6$, triclinic, $P\bar{1}$, $a = 10.569$ (4), $b = 16.697$ (4), $c = 19.631$ (7) Å, $\alpha = 74.91$ (2), $\beta = 84.34$ (3), $\gamma = 71.80$ (2)°, $V = 3176.9$ Å³, $Z = 2$, $D_m = 1.51$, $D_x = 1.523$ Mg m⁻³, $\lambda(\text{Mo } K\alpha) = 0.71069$ Å, $\mu = 2.645$ mm⁻¹, $F(000) = 1472$, $T = 296$ K, final $R = 0.041$ for 4943 observed reflections. The unit cell contains ClO₄⁻ anions and [Hg(PR₃)₃]²⁺ cations. Hg has a trigonal planar coordination [P—Hg—P = 126.7 (1), 113.1 (1), 115.5 (1)°] with a small pyramidal distortion.

Introduction. Crystal structures of HgX₂ adducts have been determined for various substituted phosphines, but

X is generally a soft ligand (halide, SCN, CN) occupying a site in the metal coordination sphere (Podlahová, Kratochvil, Loub & Paulus, 1986; Allen, Bell, Fong, March & Nowell, 1985; Buergi, Fischer, Kunz, Parvez & Pregosin, 1982; Alyea, Dias, Ferguson & Khan, 1979a; Makhija, Rivest & Beauchamp, 1979; Aurivillius & Wendel, 1976). Even harder donors like nitrate (Whitlow, 1974; Alyea, Dias, Ferguson & Restivo, 1977; Alyea, Dias, Ferguson & Parvez, 1979; Buergi *et al.*, 1982; Alyea, Dias, Ferguson & Siew, 1983) and carboxylates (Roberts, Ferguson, Goel, Ogin & Restivo, 1978; Alyea, Dias, Ferguson & Khan, 1979b; Alyea, Dias, Ferguson & Siew, Khan & Roberts, 1979; Alyea, Dias, Ferguson & Siew,