Fourier map $= 0.217$ e Å^{-3}. Atomic scattering factors of all atoms and f' and f'' values for non-hydrogen atoms taken from *International Tables for X-ray Crystallography* (1974).

Discussion. Table 1 gives the final atomic positional parameters, and Table 2 lists the main bond lengths, bond angles and torsion angles.* A view of the title compound is shown in Fig. 1, which explains the atom-numbering scheme.

The sandwich fragments about the cobalt are nearly parallel with a dihedral angle between the plane of the diene carbons and the plane of the cyclopentadienyl of $9.2°$. The diene carbons are neither staggered nor eclipsed with respect to the Cp carbons; the torsion angles vary from 12.1 to $26.0°$. The hydrogens on C(10) and C(13) are found to be *syn* to the cobalt and *cis* to each other. All other bond lengths and angles are within normal values.

We thank Dr F. J. Hollander, Professor D. H. Templeton, and Dr Brandon Borgias. We also thank Mr Doug Grotjan who carried out the cycloaddition and prepared the crystals. The equipment used was

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* Lists of structure factors, anisotropic thermal parameters, H-atom parameters, least-squares-planes calculations, intra-molecular distances and angles, and torsion angles have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43126 (24 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.
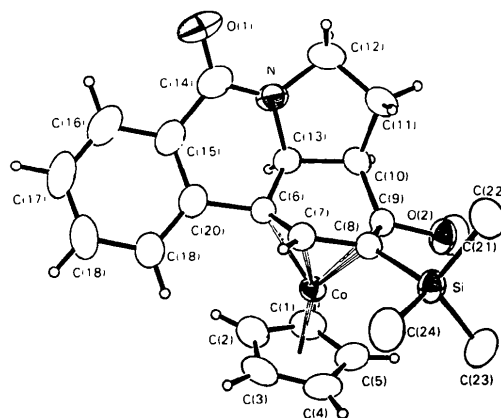

Fig. 1. Molecular structure with crystallographic numbering scheme. The ellipsoids are scaled to represent the 60% probability surface. Hydrogen atoms are given arbitrarily small spheres for clarity.

References

FRENZ, B. A. (1982). *Structure Determination Package*. College Station, Texas, and Enraf–Nonius, Delft; as revised locally by Dr F. J. HOLLANDER.
GROTJAHN, D. (1985). Personal communication.
International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
VOLLHARDT, K. P. C. (1984). *Angew. Chem. Int. Ed. Engl.* **23**(8), 539–556.
ZACHARIASEN, W. H. (1963). *Acta Cryst.* **18**, 1139–1144.

Structure of an Acetonitrile Complex of Platinum(II): Tetraethylammonium Acetonitriletribromoplatinate(II)

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Abstract. [N(C$_2$H$_5$)$_4$][PtBr$_3$(CH$_3$CN)], $M_r = 606.1$, triclinic, $P\bar{1}$, $a = 8.890$ (1), $b = 13.042$ (3), $c = 7.695$ (1) Å, $\alpha = 96.17$ (1), $\beta = 98.25$ (1), $\gamma = 103.62$ (2)°, $V = 848.81$ Å^3, $Z = 2$, $D_x = 2.37$ g cm^{-3}, λ(Mo $K\alpha$) $= 0.71073$ Å, $\mu = 148.59$ cm^{-1}, $F(000) = 560$, $T = 296$ K, final $R = 0.040$ for 2212 observed reflections. The [PtBr$_3$(CH$_3$CN)]$^-$ unit has square-planar geometry about the Pt, with the acetonitrile coordinated to the Pt in a nearly linear manner through the N. The Br *trans* to the N is about 0.04 Å closer to the Pt than the other two Br atoms, which clearly shows the lower *trans* influence of acetonitrile compared to Br.

Introduction. Platinum(II) amine complexes have been widely studied since the discovery in 1965 of the biological activity of *cis*-diamminedichloroplatinum(II) (Rosenberg, Van Camp & Krigas, 1965). The reaction of halogen-bridged complexes of platinum(II) and

palladium(II) with amines or other potential ligands is a well known route to complexes of the type MLX_3^- (Chatt & Venanzi, 1955; Livingstone & Whitley, 1962), which we have used in the preparation of new mixed-ligand complexes $MLL'X_2$, several of which have shown antitumor properties (Cadiz, 1985). Studies of these cleavage reactions in acetonitrile suggested that the acetonitrile can compete with other potential ligands for coordination. We confirm this coordinating ability in this report of the crystal and molecular structure of an acetonitrile complex of platinum(II).

Experimental. 5 ml of an acetonitrile solution of 0·056 g (0·178 mmol) of 2-(2-chloro-5-nitrostyryl)benzo-thiazole were added slowly to 5 ml of an acetonitrile solution of 0·100 g (0·088 mmol) of [NEt$_4$]$_2$[Pt$_2$Br$_6$]. The reaction mixture was heated and stirred for 30 min and left to evaporate at room temperature. A yellow powder was obtained which yielded 72% of product which was recrystallized from acetone (Cadiz, 1985).

Diffraction data were collected by the Molecular Structure Corp., and refined in this laboratory. Orange prismatic crystal, D_m not determined, 0·25 × 0·15 × 0·10 mm, Enraf-Nonius CAD-4 diffractometer, mono-chromated Mo $K\alpha$, ω–θ scan, scan speed 2–20° min^{-1} in ω, scan width 0·7° + 0·35° tanθ, $2\theta_{max} = 50°$, lattice parameters from least squares of 25 reflections in the range of $3 < \theta < 20°$. No systematic absences, $\pm h \pm k + l$ with $|h| \le 10$, $|k| \le 15$, $l \le 9$; three standard reflections, intensities constant within experimental error; total reflections 2964, 2536 unique reflections, $R_{int} = 0·078$, 2212 with $F > 4\sigma(F)$. Absorption corrections from ψ scans, transmission factors 0·409 to 0·996. Structure was solved with $SHELXTL$ (Sheldrick, 1978), heavy-atom and difference-Fourier methods; isotropic then anisotropic refinement on F of all non-H atoms, H atoms in a rigid-group model (C—H 1·08 Å); 147 parameters refined, $R = 0·040$, $wR = 0·042$, $w^{-1} = \sigma^2(F)$, $S = 1·5$; difference syntheses showed a max. density of 0·64 e Å^{-3} (min. $-1·30$ e Å^{-3}); max. $\Delta/\sigma = 0·16$. Neutral-atom (except for Pt^{II}) scattering factors and anomalous-dispersion terms taken from *International Tables for X-ray Crystallography* (1974). No correction for secondary extinction.

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.

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* Lists of structure factors, anisotropic thermal factors, H-atom parameters and least-squares-plane information have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43153 (14 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystal-lography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. *Atom coordinates and equivalent isotropic temperature factors* (Å^2) *for* [NEt$_4$][PtBr$_3$(CH$_3$CN)]

	x	y	z	U_{eq}*
Pt	0·1895 (1)	0·2402 (1)	0·3198 (1)	0·038 (1)
Br(1)	0·0615 (2)	0·3756 (1)	0·4177 (2)	0·064 (1)
Br(2)	0·3319 (2)	0·1092 (1)	0·2401 (2)	0·065 (1)
Br(3)	0·2193 (2)	0·3151 (1)	0·0524 (2)	0·065 (1)
N(1)	0·1566 (11)	0·1729 (8)	0·5319 (13)	0·049 (6)
C(1)	0·1283 (14)	0·1225 (10)	0·6409 (15)	0·047 (7)
C(2)	0·0949 (17)	0·0561 (10)	0·7794 (14)	0·057 (8)
N(2)	0·3172 (10)	0·7046 (7)	0·1555 (10)	0·038 (5)
C(3)	0·3768 (14)	0·8014 (9)	0·0701 (15)	0·052 (7)
C(4)	0·4634 (14)	0·6830 (10)	0·2655 (15)	0·050 (7)
C(5)	0·2284 (13)	0·6094 (10)	0·0194 (14)	0·046 (7)
C(6)	0·2022 (13)	0·7240 (10)	0·2751 (14)	0·038 (5)
C(7)	0·2595 (17)	0·8355 (11)	−0·0535 (15)	0·063 (8)
C(8)	0·4274 (17)	0·5920 (11)	0·3694 (16)	0·062 (8)
C(9)	0·3242 (17)	0·5795 (11)	−0·1211 (16)	0·066 (9)
C(10)	0·2692 (18)	0·8236 (12)	0·4210 (17)	0·072 (10)

* Equivalent isotropic U calculated from anisotropic U. $U_{eq} = (U_{11} + U_{22} + U_{33})/3$.

Table 2. *Bond lengths* (Å) *and angles* (°) *in* [NEt$_4$]-[PtBr$_3$(CH$_3$CN)]

[PtBr$_3$(CH$_3$CN)]$^-$

Pt–Br(1)	2·426 (1)	Pt–N(1)	1·961 (10)
Pt–Br(2)	2·432 (1)	N(1)–C(1)	1·140 (14)
Pt–Br(3)	2·393 (1)	C(1)–C(2)	1·463 (16)

Br(1)–Pt–Br(3)	90·3 (0·1)	Br(1)–Pt–Br(2)	176·0 (0·1)
Br(2)–Pt–Br(3)	91·7 (0·1)	Br(3)–Pt–N(1)	177·1 (0·3)
Br(1)–Pt–N(1)	90·5 (0·3)	Pt–N(1)–C(1)	171·5 (1·0)
Br(2)–Pt–N(1)	87·7 (0·3)	N(1)–C(1)–C(2)	178·4 (1·4)

[NEt$_4$]$^+$

N(2)–C(3)	1·507 (14)	C(3)–C(7)	1·488 (16)
N(2)–C(4)	1·547 (14)	C(4)–C(8)	1·499 (17)
N(2)–C(5)	1·511 (13)	C(5)–C(9)	1·542 (16)
N(2)–C(6)	1·516 (13)	C(6)–C(10)	1·559 (17)

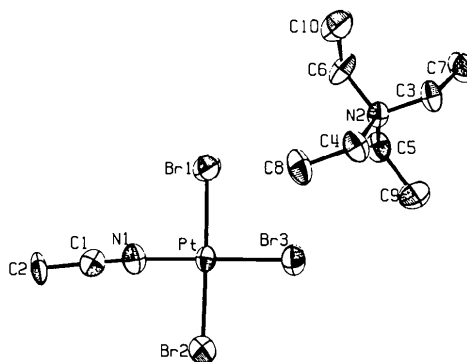| C(3)–N(2)–C(4)	106·2 (0·9)	C(5)–N(2)–C(6)	105·7 (0·8)
C(3)–N(2)–C(5)	111·8 (0·8)	N(2)–C(3)–C(7)	117·1 (1·0)
C(3)–N(2)–C(6)	111·3 (0·9)	N(2)–C(4)–C(8)	114·4 (1·0)
C(4)–N(2)–C(5)	111·7 (0·9)	N(2)–C(5)–C(9)	113·7 (1·0)
C(4)–N(2)–C(6)	110·2 (0·8)	N(2)–C(6)–C(10)	113·9 (0·9)

Fig. 1. Labeling of atoms in [NEt$_4$][PtBr$_3$(CH$_3$CN)] (*ORTEP* diagram, 50% probability ellipsoids).

The [PtBr$_3$(CH$_3$CN)]$^-$ unit has square-planar geometry about the Pt, with no atom deviating from the least-squares plane by more than 0·069 (10) Å [χ^2 = 5·0]. The acetonitrile is coordinated to the Pt in a nearly linear manner, with a Pt–N bond length of 1·961 (10) Å and Pt–N(1)–C(1) angle of 171·5 (1·0)°. The C(1) and C(2) atoms deviate from the PtNBr$_3^-$ plane by 0·24 (1) and 0·46 (1) Å respectively. The closest intramolecular contact involving the non-H atoms of acetonitrile is 2·60 (2) Å [N(1)···C(2)]. Bond lengths and angles for coordinated acetonitrile are similar to those reported for complexes of Zn^{II} and Cu^{II} (⟨N–C⟩ 1·15, ⟨C–C⟩ 1·48 Å and ⟨N–C–C⟩ 173°) (*Molecular Structures and Dimensions*, 1972). The Pt–Br(3) bond length is about 0·04 Å shorter than the other two Pt–Br lengths. This may be explained in terms of the higher *trans* influence of Br compared to acetonitrile. This effect increases the bond distances of the two Br *trans* to the Pt compared to the distance of the Br *trans* to the N.

This complex resulted from our attempt to prepare a platinum(II) complex with an ambidentate styrylbenzothiazole derivative, using acetonitrile as solvent. Acetonitrile, as well as dimethyl sulfoxide (Price, Williamson, Schramm & Wayland, 1972), has a high coordinating ability toward platinum(II) and competes with other potential ligands for coordination. It is evident from this study that acetonitrile competes with the ligand during the reaction. On the other hand, the styrylbenzothiazole derivative provides more steric hindrance than the acetonitrile.

Distances and angles within the tetraethylammonium cation are all normal. The closest intramolecular contact involving the non-H atoms is 2·41 (2) Å [C(6)···C(3)].

References

CADIZ, M. (1985). PhD Thesis, Univ. of Puerto Rico, Rio Piedras.
CHATT, J. & VENANZI, M. (1955). *J. Chem. Soc.* pp. 3858–3864.
International Tables for X-ray Crystallography (1974). Vol. IV, Tables 2.2B and 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)
JOHNSON, C. K. (1976). *ORTEPII.* Report ORNL-5138. Oak Ridge National Laboratory, Tennessee.
LIVINGSTONE, S. E. & WHITLEY, A. (1962). *Aust. J. Chem.* 15, 175–180.
Molecular Structures and Dimensions (1972). Vol. A1, pp. 529–530. International Union of Crystallography.
PRICE, J. H., WILLIAMSON, A. N., SCHRAMM, R. F. & WAYLAND, B. B. (1972). *Inorg. Chem.* 11, 1280–1284.
ROSENBERG, B., VAN CAMP, L. & KRIGAS, T. (1965). *Nature (London)*, 205, 698–699.
SHELDRICK, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen, Federal Republic of Germany.

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Monoclinic Anhydrous Zinc(II) Acetate

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Abstract. *catena*-Bis-μ-acetato-(O,O')-dizinc(II), C$_4$H$_6$O$_4$Zn, M_r = 183·47, C2/c, a = 30·237 (2), b = 4·799 (1), c = 9·260 (1) Å, β = 99·49 (1)°, V = 1325·3 (3) Å^3, Z = 8, D_x = 1·839 Mg m^{-3}, λ(Mo Kα) = 0·71073 Å, μ = 3·74 mm^{-1}, $F(000)$ = 736, T = 293 K, R = 0·025 for 1066 unique observed reflections. The structure consists of polymeric two-dimensional sheets in which tetrahedrally coordinated Zn atoms are connected by acetate bridges in a *syn–anti* arrangement. The geometry of coordination is similar to that in the orthorhombic form of zinc acetate, which, however, forms a three-dimensional network. The methyl-group H atoms are disordered.

Introduction. Capilla & Aranda (1979) reported the crystal structure of anhydrous zinc(II) acetate, in which Zn is tetrahedrally coordinated and the acetate ligands act as *syn–anti* bridges to form a three-dimensional polymeric network. A spectroscopic study of anhydrous zinc(II) acetate, however, led to the suggestion of a mononuclear structure, with chelating ligands, on the basis of the vibrational spectrum (Johnson, Powell &