N—H···O hydrogen bonds involving the nitro O atom are observed in *p*-nitrobenzamide (Di Rienzo, Domenicano & Serantoni, 1977).

References

AOYAMA, T., OHBA, S. & SAITO, Y. (1988). *Acta Cryst.* **C44**, 1703–1707.

COPPENS, P. & LEHMANN, M. S. (1976). *Acta Cryst.* **B32**, 1777–1784.

DI RIENZO, F., DOMENICANO, A. & SERANTONI, E. F. (1977). *Acta Cryst.* **B33**, 3854–3858.

GLAZER, A. M. & STADNICKA, K. (1989). *Acta Cryst.* **A45**, 234–238.

HAMILTON, W. C. (1959). *Acta Cryst.* **12**, 609–610.

HILLER, W., GARCIA-FERNANDEZ, M. E., BERMEJO, M. R. & CASTANO, M. V. (1986). *Acta Cryst.* **C42**, 60–62.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

KATRITZKY, A. R., RANDALL, E. W. & SUTTON, L. E. (1957). *J. Chem. Soc.* pp. 1769–1775.

MCCONNELL, N. M., DAY, R. O. & WOOD, J. S. (1986). *Acta Cryst.* **C42**, 1094–1095.

O'CONNOR, C. J., SINN, E. & CARLIN, R. L. (1977). *Inorg. Chem.* **16**, 3314–3320.

SAKURAI, T. & KOBAYASHI, K. (1979). *Rikagaku Kenkyusho Hokoku*, **55**, 69–77.

TAYLOR, D. (1978). *Aust. J. Chem.* **31**, 713–721.

TWIEG, R. J. & JAIN, K. (1983). In *Non-linear Optical Properties of Organic, Polymeric Materials*, edited by D. J. WILLIAMS, pp. 57–80. *Am. Chem. Soc. Ser.* No. 233.

Acta Cryst. (1990). **C46**, 765–767

Structure of [Pt(C_6H_5)$_2$(btz-*N*,*N′*)].$CHCl_3$, btz = 2,2′-Bi-5,6-dihydro-4*H*-1,3-thiazine

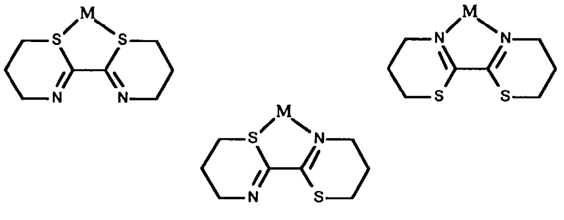
BY G. BRUNO, S. LANZA AND F. NICOLÒ

Dipartimento di Chimica Inorganica e Struttura Molecolare dell'Università, Vill. S. Agata, Salita Sperone 31, 98100 *Messina, Italy*

(*Received* 1 *February* 1989; *accepted* 27 *June* 1989)

Abstract. (2,2′-Bi-5,6-dihydro-4*H*-1,3-thiazine-*N*,*N′*)-diphenylplatinum trichloromethane solvate, [Pt(C_6H_5)$_2$($C_8H_{12}N_2S_2$)].$CHCl_3$, $M_r = 669{\cdot}0$, monoclinic, $P2_1/n$, $a = 14{\cdot}945\,(2)$, $b = 9{\cdot}841\,(1)$, $c = 16{\cdot}873\,(3)$ Å, $\beta = 109{\cdot}8\,(1)°$, $V = 2334{\cdot}7\,(4)$ Å^3, $Z = 4$, $D_x = 1{\cdot}90$ Mg m^{-3}, λ(Mo $K\alpha$) = 0·71069 Å, $\mu = 6{\cdot}260$ mm^{-1}, $F(000) = 1296$, $T = 298$ K, final $R = 0{\cdot}036$ and $wR = 0{\cdot}038$ for 3123 independent reflections [$I > 3\sigma(I)$]. The [Pt(C_6H_5)$_2$(btz-*N*,*N′*)] complex has square-planar geometry about the Pt atom, with the btz coordinated to the Pt atom through the N atoms of the dihydrothiazine. The average Pt—N and Pt—C bond distances are 2·089 (6) and 2·008 (7) Å. In the solid state there is an interaction between the H atom of the chloroform molecule and the metal atom [Pt···H = 2·48 (1) Å].

Introduction. 2,2′-Bi-5,6-dihydro-4*H*-1,3-thiazine (btz) is a heterocyclic ligand with three possible coordination modes:



Its coordinating properties and those of the parent ligand 2,2′-bi-4,5-dihydrothiazole have been widely investigated (Nelson, Nelson & Perry, 1976). It has been shown that the chelation occurs *via* the α-di-imine group, except in a copper(I) derivative where the ligand is bound to both S and N atoms (Drew, Pearson, Murphy & Nelson, 1983).

We have prepared the title compound in order to achieve a mononuclear precursor for bimetallic systems. The X-ray analysis was undertaken to clarify the coordination mode of the 2,2′-bi-4,5-dihydrothiazine.

Experimental. The title compound was obtained by mixing, in chloroform, equimolecular quantities of *cis*-[Pt(C_6H_5)$_2$(Me_2SO)$_2$] and btz. Red prismatic crystals suitable for X-ray investigation were obtained by slow evaporation from a chloroform–hexane solution.

A quasi-spherical single crystal with approximate dimensions 0·15 × 0·15 × 0·16 mm was used for intensity-data collection. Accurate unit-cell dimensions were obtained from least-squares refinement of the angular setting of 20 strong reflections in the range $14 < 2\theta < 30°$. Siemens–Stoe four-circle diffractometer using graphite-monochromated Mo $K\alpha$ radiation, ω–2θ mode, $2\theta_{\max} = 50°$ ($0 \le h \le 13$, $-14 \le k \le 14$, $-16 \le l \le 16$). Four standard reflections

0108-2701/90/050765-03$03.00

$(\overline{22}3$, $33\bar{7}$, $\overline{662}$ and $\bar{6}38)$ monitored every 5200 s showed no significant intensity variation. Lorentz and polarization corrections applied, no correction for absorption or secondary extinction.

The structure was solved by standard Patterson methods, least-squares refinements, and difference Fourier maps. All non-H atoms were refined anisotropically. All H atoms were included at calculated positions with a common thermal parameter. Of 4558 measured independent reflections, 3123 having $I > 3\sigma(I)$ were used to refine 26 parameters to final residuals of $R = 0{\cdot}036$ and $wR = 0{\cdot}038$, $w = [\sigma^2(F_o) + 0{\cdot}00189F_o^2]^{-1}$, $S = 2{\cdot}146$, $(\Delta/\sigma)_{\max} < 0{\cdot}1$. Max. peak height in final difference Fourier map 0·65 e Å^{-3}.

Scattering factors were taken from *International Tables for X-ray Crysdtallography* (1974). All calculations were performed with *SHELX*76 (Sheldrick, 1976) and *PARST* (Nardelli, 1983) programs on the IBM 4341 computer at the Centro di Calcolo dell'Università di Messina. *ORTEP* (Johnson, 1976) was used to produce Figs. 1 and 2.

Table 1. *Atomic coordinates* ($\times 10^4$) *and equivalent isotropic displacement parameters* (Å$^2 \times 10^3$)

	x	*y*	*z*	U_{eq}*
Pt	6204 (1)	4331 (1)	8177 (1)	41 (1)
S(2)	2966 (1)	4936 (3)	7041 (2)	60 (1)
S(1)	3595 (1)	3043 (3)	5934 (1)	73 (1)
Cl(1)	4781 (3)	587 (3)	8217 (2)	108 (2)
Cl(2)	6072 (2)	971 (3)	9907 (2)	104 (1)
Cl(3)	4333 (2)	2478 (3)	9281 (2)	93 (1)
C	5257 (6)	1746 (10)	9021 (6)	70 (4)
N(1)	4843 (4)	5164 (7)	7945 (4)	51 (2)
N(2)	5369 (4)	3337 (6)	7094 (4)	44 (2)
C(1)	4633 (6)	6197 (10)	8477 (6)	66 (3)
C(2)	3852 (6)	7108 (11)	8017 (7)	81 (4)
C(3)	2922 (6)	6419 (11)	7638 (7)	80 (4)
C(4)	4246 (6)	1827 (11)	5549 (6)	75 (4)
C(5)	5785 (5)	2405 (9)	6639 (5)	56 (3)
C(6)	5252 (6)	2241 (12)	5715 (5)	75 (4)
C(7)	4188 (5)	4625 (7)	7346 (5)	46 (3)
C(8)	4488 (5)	3635 (8)	6822 (4)	45 (2)
C(12)	7203 (6)	5065 (10)	9970 (5)	62 (3)
C(13)	7706 (8)	5959 (13)	10624 (6)	87 (5)
C(14)	7885 (7)	7257 (12)	10462 (7)	77 (4)
C(15)	7587 (7)	7695 (10)	9657 (7)	78 (5)
C(16)	7096 (6)	6847 (9)	9000 (5)	63 (3)
C(11)	6909 (5)	5500 (8)	9163 (5)	52 (3)
C(22)	7565 (5)	2010 (9)	8522 (5)	59 (3)
C(23)	8408 (6)	1354 (9)	8584 (6)	63 (4)
C(24)	9130 (6)	2046 (10)	8444 (6)	64 (3)
C(25)	9018 (6)	3372 (11)	8246 (7)	72 (4)
C(26)	8188 (6)	4039 (8)	8194 (6)	61 (4)
C(21)	7435 (5)	3361 (7)	8323 (4)	43 (2)
H	5614	2429	8829	90†

* U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

† Calculated position.

Table 2. *Selected bond lengths* (Å) *and angles* (°)

Pt—N(1)	2·103 (6)	Pt—N(2)	2·075 (5)
Pt—C(11)	2·003 (8)	Pt—C(21)	2·013 (7)
S(2)—C(3)	1·79 (1)	S(2)—C(7)	1·748 (7)
S(1)—C(4)	1·80 (1)	S(1)—C(8)	1·735 (7)
N(1)—C(7)	1·262 (9)	N(1)—C(1)	1·46 (1)
N(2)—C(5)	1·463 (9)	N(2)—C(8)	1·273 (8)

N(2)—Pt—N(1)	76·4 (2)	C(11)—Pt—N(1)	97·7 (3)
C(11)—Pt—N(2)	172·7 (3)	C(21)—Pt—N(1)	173·8 (3)
C(21)—Pt—N(2)	97·8 (2)	C(21)—Pt—C(11)	88·2 (3)
C(7)—S(2)—C(3)	102·3 (4)	C(8)—S(1)—C(4)	100·6 (4)
C(1)—N(1)—Pt	123·6 (5)	C(7)—N(1)—Pt	115·1 (5)
C(5)—N(2)—Pt	121·4 (4)	C(8)—N(2)—Pt	116·7 (5)
C(7)—N(1)—C(1)	121·0 (6)	C(8)—N(2)—C(5)	121·8 (6)
C(2)—C(1)—N(1)	113·5 (7)	C(3)—C(2)—C(1)	114·2 (9)
C(2)—C(3)—S(2)	114·1 (7)	C(6)—C(4)—S(1)	112·7 (7)
C(6)—C(5)—N(2)	116·2 (7)	C(5)—C(6)—C(4)	111·8 (7)
N(1)—C(7)—S(2)	127·7 (6)	C(8)—C(7)—S(2)	115·9 (5)
C(8)—C(7)—N(1)	116·4 (6)	N(2)—C(8)—S(1)	129·3 (6)
C(7)—C(8)—S(1)	115·8 (5)	C(7)—C(8)—N(2)	114·9 (6)
C(12)—C(11)—Pt	123·7 (7)	C(16)—C(11)—Pt	117·7 (6)
C(22)—C(21)—Pt	122·6 (5)	C(26)—C(21)—Pt	120·6 (5)

Discussion. The crystal consists of $[Pt(C_6H_5)_2$(btz-*N*,*N'*)] molecules and solvent chloroform in the molar ratio 1:1. Final atomic coordinates and equivalent isotropic temperature factors are listed in Table 1,* selected bond lengths and angles are listed in Table 2. A view of the molecule is shown in Fig. 1 and Fig. 2 shows the packing diagram. The $[Pt(C_6H_5)_2$(btz-*N*,*N'*)] molecule displays a *cis*-square-planar coordination around the Pt atom and approximate C_s symmetry. Nevertheless, the least-squares plane through the four atoms of the coordination sphere indicates that this portion of the molecule deviates slightly from planarity [N(1) −0·053 (7), N(2) 0·046 (6), C(11) 0·070 (8) and C(21) −0·054 (7) Å]; the Pt atom is 0·016 (1) Å out of this plane. The observed Pt—C bond distances of 2·002 (7) and 2·013 (7) Å are shorter than 2·043 (5) and 2·049 (6) Å observed in *cis*-$[Pt(C_6H_5)_2(Me_2SO)_2]$ (Bardi, Del Prà, Piazzesi & Trozzi, 1981); the bond angle C(11)—Pt—C(21) is 88·2 (3)°. Such a difference could be due to the lower *trans* influence of the N donor atoms of the btz ligand. The C—C bond lengths fall within the range 1·35 (1)–1·41 (1) Å, the mean being 1·38 (1) Å. The phenyl rings are tilted from the coordination plane by 67·1 (2) and 74·9 (3)°, being thus rotated by 83·7 (3)° to each other. The Pt atom completes its coordination with the N atoms of the bidentate btz ligand. The Pt—N bond lengths 2·103 (6) and 2·076 (6) Å are in good agreement with the values found in related compounds. The N(1)—Pt—N(2) bond angle is 76·4 (2)°. The small 'bite' of the btz allows the two coordination angles N(1)—Pt—C(11) and N(2)—Pt—C(21) to be 97·7 (3) and 97·8 (3)° for minimizing the steric interaction between the phenyl groups and the corresponding rings of the btz. The two six-membered heterocyclic rings are stereo-

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 52377 (23 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

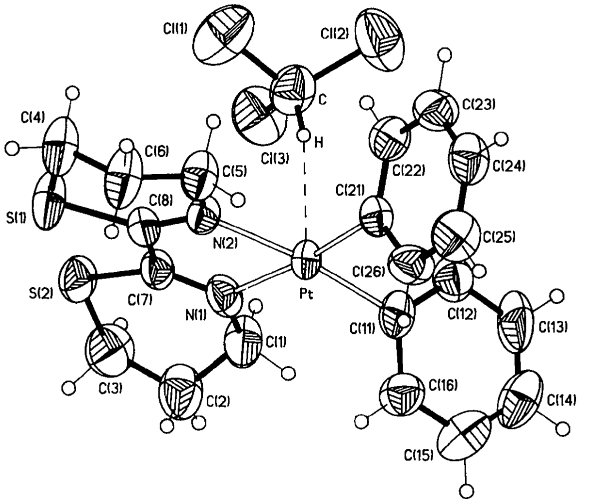


Fig. 1. Molecular structure with the atom-numbering scheme.

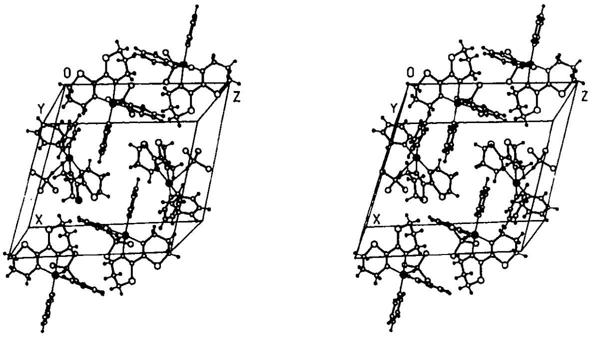


Fig. 2. A stereoscopic view of the packing.

chemically different. In ring *A*, five atoms, N(2), C(8), S(1), C(4) and C(5), are almost coplanar, while C(6) lies 0·63 Å out of this plane; in ring *B*, C(2) lies 0·62 Å out of the best plane through N(1), C(7), S(2), C(1) and C(3).

Bond distances within the rings are as expected, though it is noticeable that S(2)—C(7) and S(1)—C(8) [1·748 (7) and 1·735 (7) Å] are significantly shorter than S(2)—C(3) and S(1)—C(4) [1·79 (1) and 1·80 (1) Å]. The values, together with the value of 1·48 (1) Å for the C(7)—C(8) single-bond distance suggest a small degree of multiple-bond delocalization over the S—C—N system. There is a small twist around the C(7)—C(8) bond connecting the two six-membered rings [N(1)—C(7)—C(8)—N(2) torsion angle = 7·1 (9)°].

The two rings are not coplanar because of a strong S(1)···S(2) (3·005 Å) intramolecular contact which justifies the *trans* configuration observed in the free ligand (Huffman & Sattelberger, 1981). An intermolecular interaction between the H atom of chloroform and the metal atom may be present. The carbon to platinum separation is 3·44 Å. The Pt···H separation (for the H atom in calculated position) of 2·48 Å is in the range found for weak intramolecular *M*···H interactions (Crabtree, Holt, Lavin & Morehouse, 1985).

References

Bardi, R., Del Prà, A., Piazzesi, A. M. & Trozzi, M. (1981). *Cryst Struct. Commun.* **10**, 301–311.

Crabtree, R. H., Holt, E. M., Lavin, M. & Morehouse, S. M. (1985). *Inorg. Chem.* **24**, 1986–1992.

Drew, M. G. B., Pearson, T. R., Murphy, B. P. & Nelson, S. M. (1983). *Polyhedron*, **2**, 269–274.

Huffman, J. C. & Sattelberger, A. D. (1981). *Cryst. Struct. Commun.* **10**, 1535–1538.

International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)

Johnson, C. K. (1976). *ORTEP*II. Report ORNL-5138. Oak Ridge National Laboratory, Tennessee, USA.

Nardelli, M. (1983). *Comput. Chem.* **7**, 95–98.

Nelson, J., Nelson, S. M. & Perry, W. D. (1976). *J. Chem. Soc. Dalton Trans.* pp. 1282–1289.

Sheldrick, G. M. (1976). *SHELX*76. Program for crystal structure determination. Univ. of Cambridge, England.

Acta Cryst. (1990). **C46**, 767–770

Structure of Di(4-picolinium) Pentachloro(phenyl)stannate(IV)

Peter Storck and Alarich Weiss

Institut für Physikalische Chemie, Physikalische Chemie III, *Technische Hochschule Darmstadt, Petersenstrasse* 20, *D*-6100 *Darmstadt, Federal Republic of Germany*

(*Received* 4 *March* 1989; *accepted* 9 *August* 1989)

Abstract. $2C_6H_8N^+.[Sn(C_6H_5)Cl_5]^{2-}$, $M_r = 561·34$, monoclinic, $P2_1/c$, $a = 14·527$ (3), $b = 8·454$ (2), $c = 18·367$ (4) Å, $\beta = 94·04$ (1)°, $V = 2250·06$ Å^3, $Z = 4$, $D_m = 1·67$ Mg m^{-3}, $D_x = 1·66$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0·71073$ Å, $\mu = 1602$ m^{-1}, $F(000) = 1112$, $T = 296$ K, final $R = 0·027$ for 2724 unique reflexions. The title