y coordinates as presented in this study and those given by Chou *et al.* Consequently, the structure reported by the latter authors is incorrect.

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Structure of *trans*-Dichloro(ethylene)(pyrazole)platinum(II)

BY D. A. JOHNSON, W. C. DEESE AND A. W. CORDES

Department of Chemistry, University of Arkansas, Fayetteville, Arkansas 72701, *USA*

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Abstract. [*trans*-$Pt(C_2H_4)(C_3H_4N_2)Cl_2$], $P2_1/c$, a = 7·086 (1), b = 11·204 (2), c = 11·118 (1) Å, β = 103·08 (1)°, $Z = 4, D_o$ = 2·74 (2), D_c = 2·79 Mg m^{-3}; R_F = 0·051 for 1172 reflections collected with a manual diffractometer and Mo X-rays. A nearly coplanar conformation [dihedral angle = 3·5 (7)°] is observed for the ligation plane and molecular plane of the aromatic ligand, as a result of intramolecular hydrogen bonding between the pyrazole NH proton and a chloro ligand.

Introduction. In an investigation of the chemical properties of a series of pyrazole adducts of Zeise's anion, $[Pt(C_2H_4)Cl_3]^-$, we have noted that deprotonation of the coordinated pyrazole (Hpz) ligand yields an anion which can react as a bridging metallochelator toward a number of class-B metal centers (Johnson & Deese, 1981). In a structural study of the symmetrical binuclear product (Deese, Johnson & Cordes, 1981) $[(C_2H_4)(Cl)Pt(\mu\text{-pz})(\mu\text{-Cl})Pt(Cl)(C_2H_4)]$, it was found that, with the exception of the olefinic moieties, the complex was planar. The possibility of a metal interaction with the ring π system in maintaining the planarity of the central portion of this structure was discussed; however, strong support for such an interaction could not be inferred from one crystallographic determination. In an attempt to obtain further data on the tendency of coplanar coordination by the pyrazole group, we have carried out a crystallographic investigation of the structure of the neutral adduct of Zeise's anion and a monodentate pyrazole, *trans*-$Pt(C_2H_4)Cl_2(Hpz)$. Again, to our surprise, an essentially planar conformation is observed for all non-olefinic atoms. Al-Najjar, Green & Sarhan (1980) have suggested that enhanced platinum-to-aromatic-ligand π bonding may occur when such a coplanar conformation is forced by other molecular interactions. In the present case, an intramolecular hydrogen bond between the ring NH proton of the pyrazole and a coordinated chloride appears to be important in establishing the conformation of this complex; however, structural evidence for metal–ligand π bonding was not observed.

All reagents used were reagent-grade chemicals and were used without further purification. Synthesis of the title compound has been previously reported (Johnson, Deese & Howe, 1980). Yellow parallelepiped-shaped

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crystals of *trans*-$Pt(C_2H_4)Cl_2(Hpz)$ were obtained by the slow evaporation of a CCl_4 solution of this complex. A crystal with dimensions 0·22 × 0·28 × 0·41 mm (with the *a* axis parallel to the long dimension) was selected and mounted with epoxy cement on a glass fiber. The monoclinic space group $P2_1/c$ was assigned on the basis of Weissenberg photographs. Unit-cell parameters were determined by a least-squares fit of 14 reflections which had been carefully centered on the diffractometer ($80 \geq 2\theta \geq 69°$) [$\lambda(\mathrm{Cu}\,K\alpha_1) = 1·5405$ Å].

Data collection was by the θ–2θ scan technique with a GE XRD-5 manual diffractometer (Zr-filtered Mo $K\alpha$ radiation, $\lambda = 0·7107$ Å). A scan rate of 2° min^{-1} was used with 2° scans and 10 s stationary-crystal–stationary-counter background at each end of the scan. The intensities of 1604 independent reflections ($2\theta \leq 50°$) were measured and, of these, 1172 were considered observed ($I > 3\sigma$). Owing to obvious human error in the manual recording of the data 13 reflections were omitted in late stages of the refinement. An additional 15 low-angle reflections of high intensity were given zero weight during the refinement because of extinction effects. Three standard reflections were examined every 50 reflections to monitor crystal degradation; the total intensity decrease was 15% and was compensated by a linear correction. The $\mu(\mathrm{Mo}\,K\alpha)$ value was 17·703 mm^{-1}. An absorption correction was made with summation points every 0·04 mm; the correction factors for I varied from 0·15 to 0·29.

Platinum atom positions were determined by Patterson methods and the remaining non-hydrogen atoms were located with Fourier methods. Refinement of the atom positions was by full-matrix least-squares methods with $w = 1/(\sigma_F)^2$, where σ_F is derived from the σ of the counting statistics. The temperature factors of all non-hydrogen atoms were refined anisotropically. The hydrogen atom positions were not located by difference Fourier maps; therefore, hydrogen atom positions for the pyrazole ring were calculated (C—H = 0·95 Å). The final R value was 0·051 and the weighted R value was 0·067. A final difference Fourier map yielded a number of chemically-uninterpretable peaks with intensities less than 2·0 e Å^{-3}; all of the larger peaks were within 1·2 Å of a heavy-atom position. The final $\Delta F/\sigma$ values showed no dependence on F or θ. In the final cycle of refinement no parameter shifted by more than 0·14σ.

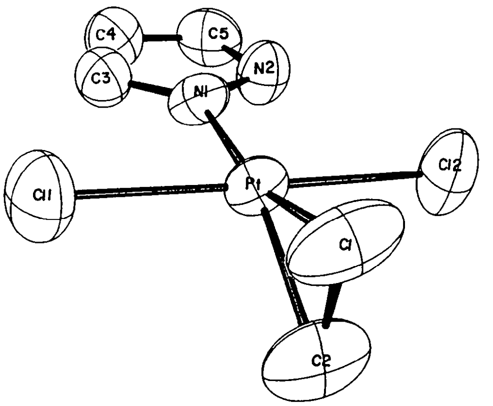

Fig. 1. *ORTEP* (Johnson, 1965) diagram of *trans*-dichloro(ethylene)(pyrazole)platinum(II). Only experimentally located atoms are shown.

Table 1. *Final atomic coordinates with e.s.d.'s in parentheses*

	x	y	z	B_{eq} (Å^2)*
Pt	0·11534 (9)	0·05943 (6)	0·22306 (5)	3·45 (4)
Cl(1)	0·3658 (7)	0·1614 (5)	0·1733 (5)	5·5 (2)
Cl(2)	−0·1321 (8)	−0·0467 (6)	0·2709 (5)	5·9 (3)
C(1)	0·278 (3)	0·037 (2)	0·407 (2)	6 (1)
C(2)	0·317 (4)	−0·060 (2)	0·343 (2)	6 (1)
C(3)	−0·038 (3)	0·212 (2)	−0·004 (2)	4·5 (8)
C(4)	−0·206 (3)	0·242 (2)	−0·087 (2)	5·0 (9)
C(5)	−0·348 (3)	0·180 (2)	−0·057 (2)	5·4 (9)
N(1)	−0·071 (2)	0·134 (1)	0·072 (1)	4·0 (6)
N(2)	−0·261 (2)	0·114 (2)	0·042 (1)	4·6 (7)
H(N2)	−0·328	0·060	0·084	5·0
H(C3)	0·090	0·243	−0·002	5·0
H(C4)	−0·225	0·302	−0·159	5·0
H(C5)	−0·483	0·182	−0·099	5·0

* Equivalent isotropic temperature factors B_{eq} (Å^2) have been calculated from $B_{eq} = \frac{4}{3}(\beta_{11}a^2 + \beta_{22}b^2 + \beta_{33}c^2 + \beta_{12}ab\cos\alpha + \beta_{13}ac\cos\beta + \beta_{23}bc\cos\gamma)$. For hydrogen atoms an isotropic B value of 5 Å^2 was assigned.

Table 2. *Selected interatomic distances* (Å) *and angles* (°) *with e.s.d.'s in parentheses*

Pt—Cl(1)	2·282 (6)	Cl(1)—Pt—Cl(2)	178·6 (2)
Pt—Cl(2)	2·279 (6)	Cl(1)—Pt—N(1)	89·4 (4)
Pt—C(1)	2·13 (2)	Cl(2)—Pt—N(1)	90·9 (4)
Pt—C(2)	2·18 (2)	Cl(1)—Pt—*X**	90·8 (7)
Pt—*X**	2·04 (2)	Cl(2)—Pt—*X**	88·9 (7)
Pt—N(1)	2·06 (1)	N(1)—Pt—*X**	178·7 (8)
N(1)—N(2)	1·33 (2)	C(1)—Pt—C(2)	36·8 (9)
N(1)—C(3)	1·28 (3)	Pt—N(1)—N(2)	124 (1)
C(3)—C(4)	1·37 (3)	Pt—N(1)—C(3)	129 (1)
C(4)—C(5)	1·33 (3)	N(2)—N(1)—C(3)	105 (2)
C(5)—N(2)	1·35 (3)	N(1)—C(3)—C(4)	110 (2)
C(1)—C(2)	1·36 (3)	C(3)—C(4)—C(5)	107 (2)
		C(4)—C(5)—N(2)	105 (2)
		C(5)—N(2)—N(1)	111 (2)

* X = midpoint between C(1) and C(2).

The atom numbering scheme is given in Fig. 1. The final atom positions are recorded in Table 1 and selected intramolecular bond lengths and angles are summarized in Table 2. A best-planes analysis of subunits of the molecular structure along with the

Table 3. *Atom distances* (Å) *from selected best planes*

Asterisks indicate atoms used in the calculation of the best planes.

	Plane 1	Plane 2	Plane 3
Pt	0·006 (7)*	0·0*	−0·1053 (7)
Cl(1)	−0·026 (6)*	2·286 (6)	−0·056 (6)
Cl(2)	−0·028 (6)*	−2·277 (6)	−0·100 (6)
C(1)	0·71 (2)	0·0*	−0·94 (2)
C(2)	−0·65 (2)	0·0*	0·41 (2)
X†	0·03 (2)*	0·00 (3)	−0·26 (2)
C(3)	0·09 (2)	0·96 (2)	−0·01 (2)*
C(4)	0·15 (2)	0·51 (2)	0·01 (2)*
C(5)	0·14 (2)	−0·81 (2)	0·00 (2)*
N(1)	0·01 (2)*	−0·01 (2)	0·01 (2)*
N(2)	0·06 (2)	−1·11 (2)	−0·00 (2)*

Dihedral angles between planes (°)

Plane 1–plane 2	91 (1)
Plane 1–plane 3	3·5 (7)
Plane 2–plane 3	88 (1)

† *X* = midpoint between C(1) and C(2).

Table 4. *Typical dihedral angles* (°) *between ligation planes and aromatic ligand planes in platinum*(II) *complexes*

Complex	Dihedral angle	Reference
trans-[Pt(N-methylimidazole)(NH_3)$_2$]Cl_2.$2H_2O$	49·2	(*a*)
cis-[Pt(N-methylimidazole)$_2$$Cl_2$]	41·7	(*b*)
trans-[Pt(C_2H_4)Cl_2(4-methylpyridine)]	49·7	(*c*)
trans-[Pt(C_2H_4)Cl_2(2,4,6-trimethylpyridine)]	82·8	(*c*)
trans-[Pt(Me_2SO)Cl_2(pyridine)]	59·4	(*d*)
trans-[Pt(pyridine)$_2$$Cl_2$]	56·2	(*e*)
K[Pt(2,6-dimethylpyridine)Cl_3]	81·6	(*f*)
[(C_2H_4)ClPt(μ-Cl)(μ-pyrazolide)Pt(C_2H_4)Cl]	5·7	(*g*)
trans-[Pt(C_2H_4)Cl_2(pyrazole)]	3·5	(*h*)

References: (*a*) Carmichael, Chan, Cordes, Fair & Johnson (1972); (*b*) Graves, Hodgson, van Kralingen & Reedijk (1978); (*c*) Caruso, Spagna & Zambonelli (1978); (*d*) Caruso, Spagna & Zambonelli (1980); (*e*) Colamarino & Orioli (1975); (*f*) Melanson & Rochon (1976); (*g*) Deese, Johnson & Cordes (1981); (*h*) this work.

dihedral angles between these planes is given in Table 3.*

Discussion. The most interesting structural feature of *trans*-Pt(C_2H_4)Cl_2(Hpz) is the planarity of the non-olefinic portion of the molecule. In general, the dihedral angle between the ligation plane of a platinum(II) complex and the molecular plane of a coordinated monodentate azaaromatic ligand is 40–60° (or larger if the aromatic ligand carries bulky substituents on the α ring atoms). The ligand-ring–ligating-plane dihedral angles of a number of platinum(II) complexes of five- and six-membered aromatic bases are given in Table 4. With the exception of the two pyrazole-based complexes, this dihedral angle has been considered to be primarily determined by van der Waals contacts in each of the cases cited. Intermolecular non-bonded contacts involving the pyrazole ring are all greater than the van der Waals values suggesting that intermolecular interactions are not the driving force for the planarity.

A likely origin of the overall planarity resides in the apparent intramolecular hydrogen bonding between the pyrazole NH proton and the Cl(2) chloro ligand. Although this proton was not experimentally located, both the 3·08 (2) Å approach of N(2) to Cl(2) and the distance of the calculated proton position from Cl(2) (2·51 Å) are consistent with NH···Cl hydrogen bonding (Pimentel & McClellan, 1960). Spectroscopic evidence of hydrogen-bond formation is found in the frequency lowering (~150 cm^{-1}) for the ν(NH) in the title complex in both the solid and solution spectra.

All of the other observed intramolecular distances are in good agreement with previously reported values (Berthou, Elguero & Rerat, 1970; Black, Mais & Owston, 1969; Hartley, 1972). Complexes at (x,y,z) and ($\bar{x},\bar{y},\bar{z}$) appear to be weakly dimerized (Fig. 2). The platinum atom of one molecule occupies a position approximately centered over the plane of the aromatic ligand in the adjacent molecule; however, the platinum–ring-plane distance is 3·676 (1) Å, which is too long for an appreciable intermolecular π interaction.

We have examined the structural data in an effort to detect structural evidence for enhanced platinum(II)–pyrazole π bonding; however, no bond distances supporting conformation-dependent π bonding are observed. For example, the Pt—N distances for all the complexes in Table 4, which range from 1·98 to 2·08 Å, show no conformational dependence and bracket the observed Pt—N distances for similar complexes of non-π-bonding amines.

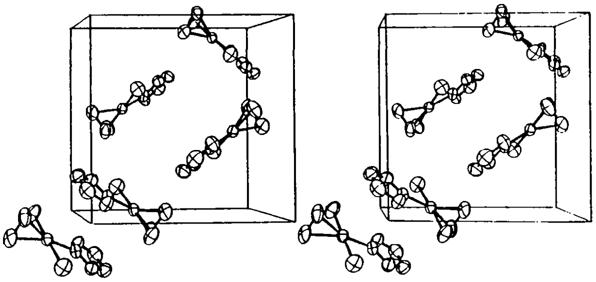

Fig. 2. Stereoscopic view of packing in the unit cell. The *b* axis is vertical, the *c* axis is horizontal and the *a* axis projects out of the page. The origin is in the upper right rear corner.

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* Lists of structure factors and anisotropic thermal parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 36192 (8 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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The Structure of the Trifluoroacetate of the (3-Oxo-2-butyl)(pyridine)mercury(II) Cation

BY JOAN HALFPENNY

*Department of Chemistry, University of Durham, South Road, Durham DH*1 3*LE, England*

AND R. W. H. SMALL

*Department of Chemistry, University of Lancaster, Lancaster LA*1 4*YA, England*

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Abstract. $[Hg(C_5H_5N)(C_4H_7O)](C_2F_3O_2)$, $C_9H_{12}HgNO^+.C_2F_3O_2^-$, triclinic, $P\bar{1}$, $a = 9.81\,(1)$, $b = 9.20\,(1)$, $c = 10.04\,(1)$ Å, $\alpha = 117.7\,(1)$, $\beta = 61.72\,(6)$, $\gamma = 110.7\,(1)$°, $D_m = 2.245$, $D_c = 2.22$ Mg m^{-3} with $Z = 2$; $R = 0.049$ for 1741 diffractometer-measured intensities. Hg forms two covalent bonds: to the 3-oxo-2-butyl group, Hg—C = 2·12 (2) Å, and to pyridine, Hg—N = 2·16 (1) Å, with ∠C—Hg—N = 174 (1)°. In addition there are contacts from Hg to trifluoroacetate O, 2·77 (2), 2·82 (1), 2·85 (2) Å, and to the 3-oxo-2-butyl O, 2·89 (1) Å. The distances indicate an ionic structure.

Introduction. This compound is one of a series of complexes of nitrogen donor ligands with mercury(II) trifluoroacetate and its derivatives. Material supplied by Dr A. J. Bloodworth was recrystallized from dichloromethane to give oblique prisms showing the forms {011}, {100} and {010}. With Cu $K\alpha$ radiation ($\lambda = 1.542$ Å), the Laue group and cell dimensions were obtained from Weissenberg photographs with crystals mounted successively about **a**, **b** and **c**. Intensity data on 1944 unique reflections were collected with graphite-monochromatized Mo $K\alpha$ radiation ($\lambda = 0.7107$ Å) on the Stoe STADI-2 automatic two-circle diffractometer, from a well developed crystal with dimensions 0·16 × 0·06 × 0·20 mm. Data from the layers $l = 0$–9 within the range $20 \leq 40°$ were processed with an initial data reduction program and all further calculations were performed with *SHELX* (Sheldrick, 1976). Absorption corrections ($\mu = 10.67$ mm^{-1}) were applied and reflections with $F < 6\sigma(F)$ were eliminated, leaving 1741 values for the structure determination and refinement. Statistical tests indicated a centric intensity distribution. Assuming the space group $P\bar{1}$, the Patterson map was used to determine the position of Hg. These coordinates were used to phase an $(F_o - F_c)$ map to give the positions of the remaining non-H atoms. Refinement of atomic parameters proceeded with anisotropic U_{ij} for Hg and isotropic U for C, N and O. Scale factors for the data collection layers were also refined at an intermediate stage. When refinement was

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