SHORT-FORMAT PAPERS

Contributions intended for publication under this heading should follow the format given in the Checklist for Authors [*Acta Cryst.* (1985). C**41**, 1–4].

Acta Cryst. (1986). C**42**, 1643–1644

Structure of *cis*-Bis(acetonitrile)dichloroplatinum(II)

By George R. Newkome,* Vinod K. Gupta and Frank R. Fronczek

Department of Chemistry, Louisiana State University, Baton Rouge, Louisiana 70803-1804, *USA*

(*Received* 20 *March* 1986; *accepted* 3 *June* 1986)

Abstract. $[PtCl_2(C_2H_3N)_2]$, $M_r = 348{\cdot}1$, monoclinic, $P2_1/c$, $a = 6{\cdot}653\ (1)$, $b = 12{\cdot}506\ (3)$, $c = 10{\cdot}290\ (2)$ Å, $\beta = 105{\cdot}39\ (2)°$, $V = 825{\cdot}5\ (6)$ Å^3, $Z = 4$, $D_x = 2{\cdot}801$ g cm^{-3}, $\lambda(\text{Mo}\ K\alpha) = 0{\cdot}71073$ Å, $\mu = 177{\cdot}51$ cm^{-1}, $F(000) = 624$, $T = 297$ K, $R = 0{\cdot}032$ for 1826 observations (of 2395 unique data). The molecule is nearly planar, its nine non-H atoms exhibiting a mean deviation of 0·056 Å from coplanarity, with a Cl atom having maximum deviation 0·140 (2) Å. Coordination geometry is square planar, with the largest deviation from orthogonal bonds being N—Pt—N 91·2 (2)°. Acetonitrile ligands coordinate linearly with Pt—N—C angles 174·6 (6) and 179·0 (6)°. Pt—Cl distances are 2·266 (2) and 2·271 (2) Å; Pt—N distances are 1·969 (5) and 1·978 (5) Å. The closest intermolecular distance between Pt atoms is 5·187 (1) Å.

Experimental. Material obtained as yellow crystals from CH_3CN solution. Crystal size $0{\cdot}16 \times 0{\cdot}28 \times 0{\cdot}40$ mm, space-group determination from systematic absences $h0l$ with l odd, $0k0$ with k odd, cell dimensions from setting angles of 25 reflections having $13 < \theta < 14°$. Data collection on Enraf–Nonius CAD-4 diffractometer, Mo $K\alpha$ radiation, graphite monochromator, ω–2θ scans designed for $I = 50\sigma(I)$, subject to max. scan time 120 s, scan rates varied 0·56–4·0° min^{-1}. Data having $1 < \theta < 30°$, $0 \le h \le 9$, $0 \le k \le 17$, $-14 \le l \le 14$ measured, corrected for Lorentz, polarization, background effects. Absorption correction by ψ scans, min. relative transmission coefficient 35·53%. Standard reflections 200, 020, 002, ±1·9% max. variation. 2395 unique data ($R_{\text{int}} = 0{\cdot}026$), 1826 observed $I > 3\sigma(I)$. Structure solved by heavy-atom methods and refined by full-matrix least squares based on F with weights $w = 4F_o^2[\sigma^2(I) + (0{\cdot}02F_o^2)^2]^{-1}$, using Enraf–Nonius SDP (Frenz & Okaya, 1980), scattering factors of Cromer & Waber (1974), anomalous-dispersion coefficients of Cromer (1974). Non-H atoms anisotropic, H atoms not located. Final $R = 0{\cdot}032$ (0·053 all data), $wR = 0{\cdot}036$, $S = 2{\cdot}296$ for 83 variables. Max. $\Delta/\sigma = 0{\cdot}01$ in final cycle, max. residual density 1·26 (near Pt), min. $-0{\cdot}86$ e Å^{-3}, extinction $g = 1{\cdot}87\ (5) \times 10^{-7}$, where $|F_c| = |F_o|(1 + gI_c)$. Atomic coordinates and equivalent isotropic thermal parameters are given in Table 1.† Fig. 1 shows the atom-numbering scheme and geometry of the molecule.

Table 1. *Coordinates and equivalent isotropic thermal parameters*

$$B_{\text{eq}} = \tfrac{4}{3}(a^2\beta_{11} + b^2\beta_{22} + c^2\beta_{33} + ac\beta_{13}\cos\beta).$$

	x	y	z	B_{eq}(Å^2)
Pt	0·19867 (5)	0·22380 (3)	0·22191 (3)	2·597 (5)
Cl(1)	0·1289 (4)	0·3900 (2)	0·2874 (2)	3·75 (4)
Cl(2)	0·4371 (4)	0·2913 (2)	0·1227 (2)	4·38 (5)
N(1)	0·252 (1)	0·0801 (7)	0·1581 (8)	2·5 (1)
N(2)	−0·002 (1)	0·1646 (6)	0·3128 (8)	2·4 (1)
C(1)	0·272 (1)	0·0012 (8)	0·1127 (9)	3·2 (2)
C(2)	0·297 (2)	−0·1026 (8)	0·0498 (10)	3·8 (2)
C(3)	−0·115 (1)	0·1300 (7)	0·3661 (9)	3·4 (2)
C(4)	−0·260 (1)	0·0862 (9)	0·4393 (10)	4·3 (2)

Related literature. *cis*-Bis(benzonitrile)dichloroplatinum(II): Eysel, Guggolz, Kopp & Ziegler (1983); *trans*-bis(benzonitrile)dichloroplatinum(II): Lauher & Ibers (1975); *cis*-dichloroplatinum(II) complexes: Howard-Lock, Lock, Turner & Zvagulis (1981), Newkome, Evans, Kiefer, Delord, Fronczek, Gupta & Xia (1986), Osborn & Rogers (1974).

This work was supported in part by the Center for Energy Studies, LSU.

---

* To whom correspondence should be addressed.

† Lists of anisotropic thermal parameters and structure-factor amplitudes have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 43135 (13 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/86/111643-02$01.50

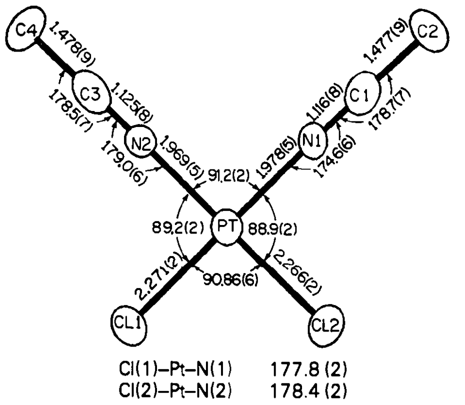

Fig. 1. Numbering scheme and geometry of the title compound. (Distances in Å, angles in °.)

References

CROMER, D. T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.3.1. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

CROMER, D. T. & WABER, J.T. (1974). *International Tables for X-ray Crystallography*, Vol. IV, Table 2.2.B. Birmingham: Kynoch Press. (Present distributor D. Reidel, Dordrecht.)

EYSEL, H. H., GUGGOLZ, E., KOPP, M. & ZIEGLER, M. L. (1983). *Z. Anorg. Allg. Chem.* **449**, 31–43.

FRENZ, B. A. & OKAYA, Y. (1980). *Enraf–Nonius Structure Determination Package*. Enraf–Nonius, Delft.

HOWARD-LOCK, H. E., LOCK, C. J. L., TURNER, G. & ZVAGULIS, M. (1981). *Can. J. Chem.* **59**, 2737–2745.

LAUHER, J. W. & IBERS, J. A. (1975). *Inorg. Chem.* **14**, 640–645.

NEWKOME, G. R., EVANS, D. W., KIEFER, G. E., DELORD, T. J., FRONCZEK, F. R., GUPTA, V. K. & XIA, Y. (1986). *Inorg. Chem.* In preparation.

OSBORN, R. S. & ROGERS, D. (1974). *J. Chem. Soc. Dalton Trans.* pp. 1002–1004.

Acta Cryst. (1986). C**42**, 1644–1646

Tetraphenylphosphonium-hexawolframat-Dibromomethan-Solvat

VON WOLFGANG WILLING, ULRICH MÜLLER* UND ALEXANDER BERG

*Fachbereich Chemie der Universität, Hans-Meerwein-Straße, D-*3550 *Marburg, Bundesrepublik Deutschland*

(*Eingegangen am* 2. *August* 1985; *angenommen am* 12. *Mai* 1986)

Abstract. $[P(C_6H_5)_4]_2[W_6O_{19}].4CH_2Br_2$, $M_r = 2781{\cdot}27$, triclinic, $P\bar{1}$, $a = 12{\cdot}079$ (3), $b = 12{\cdot}469$ (2), $c = 12{\cdot}685$ (3) Å, $\alpha = 84{\cdot}00$ (2), $\beta = 62{\cdot}20$ (3), $\gamma = 89{\cdot}82$ (2)°, $V = 1678{\cdot}6$ Å^3, $Z = 1$, $D_x = 2{\cdot}75$ g cm^{-3}, λ(Mo $K\alpha$) = 0·7017 Å, $\mu = 152{\cdot}9$ cm^{-1}, $F(000) = 1266$, $T = 295$ K, $R = 0{\cdot}062$ for 2135 independent observed reflexions. In the $[W_6O_{19}]^{2-}$ ion six octahedra share a common corner in the center; the symmetry of the ion deviates only marginally from $m3m$. The cations are arranged into $(PPh_4^+)_2$ pairs about inversion centers. Each $[W_6O_{19}]^{2-}$ ion is surrounded by eight of these pairs involving the 12 closest PPh_4^+ ions. P—C bond lengths: 1·78 (2) to 1·79 (2) Å; C—P—C angles: 107 (1) to 110 (1)°. C—Br bond lengths: 1·86 (4) and 1·89 (4) Å.

Experimentelles. 1 g $W(NO)_2Br_2$ (Johnson & Al-Obaidi, 1970) wurde mit einer Lösung von 1,04 g PPh_4Br in 30 ml CH_2Br_2 unter Ausschluß von Feuchtigkeit in Lösung gebracht. Im Verlaufe von zwei Wochen kristallisierten 1,46 g grünes PPh_4-$[W(NO)_2Br_3]$ aus, die abgefrittet wurden. Aus dem Filtrat kristallisierte die farblose Titelverbindung im Verlaufe eines Jahres. Über den Reaktionsablauf wissen wir nichts; möglicherweise ist durch einen undichten Verschluß Feuchtigkeit eindiffundiert und hat zur Bildung des $[W_6O_{19}]^{2-}$-Ions geführt, oder es hat eine Reaktion mit der Glaswand stattgefunden. Der zur Strukturuntersuchung verwendete Kristall maß 0,288 × 0,23 × 0,023 mm.

Messung: Vierkreisdiffraktometer Enraf–Nonius CAD-4, Mo $K\alpha$-Strahlung (Graphit-Monochromator), Gitterparameterberechnung mit 11 Reflexen $14 < \theta < 20°$; ω-scan, $\sin\theta/\lambda < 0{,}48$ Å^{-1} $(0 \le h \le 10, -11 \le k \le 11, -10 \le l \le 10)$; Kontrollreflexe 200 und 222 zeigten gegen Ende einen Intensitätsabfall von *ca* 3%; Lorentz-, Polarisationsfaktor- and Absorptionskorrektur (Transmissionsfaktoren 0,123 bis 0,721); 2674 unabhängige Reflexe, davon 539 mit $F < 2\sigma(F)$ als unbeobachtet gewertet und zur Verfeinerung nicht verwendet. Strukturaufklärung mittels Patterson-Synthese und Differenz-Fourier-Synthese. Verfeinerung durch Minimalisieren von $\sum w(|F_o| - |F_c|)^2$ mit $w = 1/\sigma^2(F)$ bis alle $\Delta/\sigma < 0{,}1$. H-Atome in berechneten Lagen angenommen, Phenylgruppen als starre Einheiten behandelt (C—C 1,395 Å, C—H 1,08 Å); anisotrope Temperaturfaktoren für W, Br und P, isotrope für O und C, je ein gemeinsamer isotroper für die Phenyl-H- bzw. Lösungsmittel-H-Atome. Abschließende Differenz-Fourier-Synthese: $|\Delta\rho| <$

---

* Korrespondenzautor.