only are the main structural feature of this compound. This polyanion is isostructural to the chains in the SiS_2 structure (Büssem, Fischer & Gruner, 1935). The mean Ag—I bond distance of 2·781 Å within these tetrahedra is shorter than that of 2·856 Å in $Ag_2I_4C_8H_{22}N_2$ (Thackeray & Coetzer, 1975) and that of 2·88 Å in $(CH_3)_4NAg_2I_3$ (Meyer, 1963). The $N(CH_3)_4$ tetrahedra are arranged between the infinite anions to form a zigzag chain of N and I(2) and to cause a connection of the anions parallel to **a**. The N atoms and the C atoms of the tetrahedral cations form planes parallel to *ab* as well as planes parallel to *bc*.

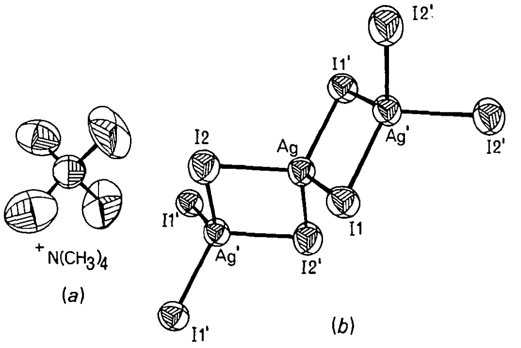

Fig. 1. Perspective drawings of (*a*) the cation $(CH_3)_4N^+$ and (*b*) a section of the anion $^1_\infty[AgI_2]^-$, with the labelling of the atoms.

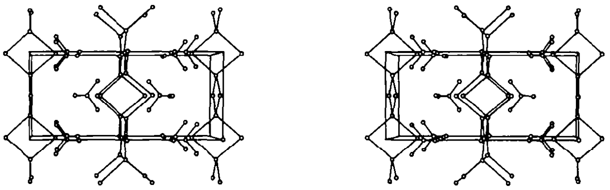

Fig. 2. Stereoscopic view of the unit cell.

References

Brink, C., Binnendijk, N. F. & van de Linde, J. (1954). *Acta Cryst.* **7**, 176–180.

Büssem, W., Fischer, H. & Gruner, E. (1935). *Naturwissenschaften*, **23**, 740.

International Tables for X-ray Crystallography (1974). Vol. IV, pp. 55, 99, 149. Birmingham: Kynoch Press.

Meyer, H. J. (1963). *Acta Cryst.* **16**, 788–791.

Peters, K., Ott, W. & von Schnering, H.-G. (1982). *Angew. Chem. Int. Ed. Engl.* **21**, 697–698; *Angew. Chem. Suppl.* pp. 1479–1490.

Sheldrick, G. M. (1978). *SHELXTL. An Integrated System for Solving, Refining and Displaying Crystal Structures from Diffraction Data.* Univ. of Göttingen.

Thackeray, M. M. & Coetzer, J. (1975). *Acta Cryst.* B**31**, 2341–2342.

Acta Cryst. (1984). C**40**, 790–793

The Structure of 1,2;3,4-Bis{μ-[bis(diphenylphosphino)methane]-*P*,*P*′}-1,2;2,4;4,1-tri-μ-carbonyl-1,2,3,3,4-pentacarbonyl-*tetrahedro*-tetrairidium(0), $C_{58}H_{44}Ir_4O_8P_4$

By Marjorie M. Harding, Barry S. Nicholls and Anthony K. Smith*

Department of Inorganic, Physical and Industrial Chemistry, University of Liverpool, PO Box 147, Grove Street, Liverpool L69 3BX, England

(*Received* 4 *November* 1983; *accepted* 10 *January* 1984)

Abstract. $M_r = 1762$, monoclinic, $I2/a$, $a = 28{\cdot}833$ (2), $b = 17{\cdot}264$ (4), $c = 23{\cdot}074$ (2) Å, $\beta = 97{\cdot}00$ (1)°, $V = 11400$ Å^3, $Z = 8$, $D_m = 2{\cdot}2$ (1), $D_x = 2{\cdot}05$ Mg m^{-3}, Mo $K\alpha$, $\lambda = 0{\cdot}7107$ Å, $\mu = 9{\cdot}11$ mm^{-1}, $F(000) = 6592$, $T = 298$ K, $R = 0{\cdot}042$ for 4779 observed reflections. The Ir atoms form a nearly regular tetrahedron with Ir—Ir bonds in the range 2·692–2·746 (1) Å. Each dppm [dppm = $(C_6H_5)_2PCH_2P(C_6H_5)_2$] ligand bridges two Ir atoms, with mean Ir—P = 2·297 (5) Å; the $Ir_4(dppm)_2$ portion has approximate, and non-crystallographic, symmetry $\bar{4}(S_4)$, but the carbonyl groups, three bridging and five terminal, do not conform to this.

Introduction. The crystal structures of several transition-metal cluster complexes containing dppm [bis(diphenylphosphino)methane] as a ligand have been determined by X-ray methods. Thus, for example, the structures of $Rh_4(CO)_8(dppm)_2$ (Carré, Cotton & Frenz, 1976), $Ru_3(CO)_8(dppm)_2$ (Lavigne, Lugan & Bonnet, 1982), and $Rh_6(CO)_{10}(dppm)_3$ (Ceriotti, Ciani,

---

* To whom all correspondence should be addressed.

0108-2701/84/050790-04$01.50

Garlaschelli, Sartorelli & Sironi, 1982) have been reported. In all cases the dppm ligands adopt a bridging configuration, although the chelating configuration for dppm is known for some mononuclear transition-metal complexes (see, for example, Steffen & Palenik, 1976).

Chelation of a bidentate phosphine ligand to a single metal atom in a cluster complex is known, for example in the complex $H_4Ru_4(CO)_{10}$(dppe) [dppe = bis(diphenylphosphino)ethane] (Churchill & Lashewycz, 1978), and for a diarsine ligand in the complex $Ir_4(CO)_{10}$(diars) [diars = 1,2-bis(dimethylarsino)benzene] (Shapley, Stuntz, Churchill & Hutchinson, 1979). We recently reported the synthesis of $Ir_4(CO)_8(dppm)_2$ (Foster, Nicholls & Smith, 1982), but an unambiguous structural characterization was not possible by spectroscopic methods. In particular, the chemical shifts obtained in the ^{31}P NMR spectrum suggested that the phosphorus atoms are involved in four-membered rings and therefore that the dppm ligands are coordinated in a chelating mode to one Ir atom, whereas infrared spectroscopic data indicated the dppm ligands to be coordinated in a bridging mode. We therefore undertook an X-ray structure determination of $Ir_4(CO)_8(dppm)_2$.

Experimental. Orange-red crystals from benzene/heptane solution, D_m by flotation in CCl_4/CH_3I; crystal 0·4 × 0·1 × 0·08 mm; preliminary photographic data showed systematic absences consistent with space groups *Ia* or *I*2/*a*, analysis confirmed the latter; intensity data recorded on a CAD-4 diffractometer by Dr M. Hursthouse (Queen Mary College, London), 25 reflections used to determine lattice parameters; θ–2θ scans, $2\theta_{max} = 60°$ (h 0→32; k 0→19; l 25→–25); empirical absorption correction applied, transmission factors 1·000–0·594; no intensity variation for 3 standard reflections throughout data collection; 7196 unique reflections recorded, 4779 [$F > 2{\cdot}5\sigma(F)$] used in structure refinement; Ir atom positions found by direct methods, remaining atoms other than H by Fourier methods; least-squares refinement (based on F) converged to R = 0·042, wR = 0·063, w = 0·7521/[$\sigma^2(F) + 0{\cdot}002677F^2$], using *SHELX* (Sheldrick, 1976); phenyl groups constrained to be regular hexagons, with individual isotropic vibration parameters assigned to each C atom; H atoms allowed to ride on the C atoms of phenyl groups at stereochemically expected positions, remaining H atoms as members of rigid methylene groups constrained so that C—H = 1·08 Å; one isotropic temperature factor used for all H atoms; anisotropic vibration parameters used for all other atoms; final difference Fourier map showed max. and min. heights of 1·8 and –0·7 e $Å^{-3}$, highest peaks located well out of bonding range of all molecules in unit cell; max. $\Delta/\sigma = 0{\cdot}6$. Atomic scattering factors from *International Tables for X-ray Crystallography* (1974) and Cromer & Liberman (1970); diagram was produced using *PLUTO* (Motherwell, 1976).

Table 1. *Atomic coordinates* (×10^4) *and thermal parameters* ($Å^2$ ×10^2)

B_{eq} is given for Ir, P, O and C(1)–C(10).
$B_{eq} = \frac{1}{3}(B_{11}+B_{22}+B_{33})$.

	x	y	z	B_{eq}/B
Ir(1)	3697·4 (3)	1479·7 (5)	952·3 (3)	4·47 (4)
Ir(2)	2902·6 (2)	2078·7 (5)	396·8 (3)	4·49 (4)
Ir(3)	3730·3 (3)	2754·1 (5)	246·4 (3)	4·58 (4)
Ir(4)	3518·1 (2)	1344·4 (4)	–236·6 (3)	4·22 (4)
P(1)	3317 (2)	390 (3)	1252 (2)	4·12 (27)
P(2)	2425 (2)	1069 (3)	567 (2)	3·93 (26)
P(3)	4448 (2)	2792 (3)	–108 (2)	4·16 (26)
P(4)	3939 (2)	1616 (3)	–976 (2)	3·81 (25)
C(1)	4152 (6)	1605 (11)	1561 (8)	5·0 (11)
O(1)	4435 (6)	1749 (10)	1966 (7)	8·8 (11)
C(2)	2442 (7)	2830 (11)	305 (10)	6·0 (13)
O(2)	2180 (6)	3299 (10)	229 (10)	11·8 (15)
C(3)	3869 (7)	3413 (12)	934 (10)	5·7 (13)
O(3)	3980 (6)	3759 (10)	1324 (6)	8·3 (11)
C(4)	3396 (7)	331 (11)	–453 (7)	4·5 (11)
O(4)	3338 (5)	–320 (9)	–583 (6)	7·0 (10)
C(5)	3399 (7)	3367 (12)	–304 (9)	5·5 (12)
O(5)	3153 (5)	3756 (9)	–641 (7)	6·8 (10)
C(6)	2887 (6)	1853 (11)	–548 (8)	4·7 (11)
O(6)	2628 (5)	1974 (9)	–958 (5)	6·0 (9)
C(7)	3204 (7)	2252 (12)	1247 (9)	5·6 (13)
O(7)	3160 (5)	2638 (8)	1654 (6)	6·3 (9)
C(8)	4129 (7)	1061 (11)	371 (8)	4·9 (12)
O(8)	4495 (4)	823 (9)	344 (5)	5·7 (9)
C(9)	2769 (6)	210 (10)	759 (7)	4·0 (10)
C(10)	4490 (7)	2112 (12)	–715 (8)	5·8 (12)
C(11)	3617 (4)	–537 (8)	1252 (6)	4·4 (4)
C(12)	3381 (4)	–1242 (8)	1244 (6)	7·2 (6)
C(13)	3631 (4)	–1936 (8)	1277 (6)	8·9 (8)
C(14)	4117 (4)	–1924 (8)	1319 (6)	8·0 (7)
C(15)	4354 (4)	–1218 (8)	1327 (6)	7·1 (6)
C(16)	4104 (4)	–525 (8)	1293 (6)	6·5 (6)
C(17)	3114 (5)	404 (8)	1985 (5)	5·3 (5)
C(18)	2729 (5)	–31 (8)	2108 (5)	6·5 (6)
C(19)	2585 (5)	–3 (8)	2663 (5)	6·9 (6)
C(20)	2825 (5)	459 (8)	3096 (5)	6·4 (6)
C(21)	3210 (5)	894 (8)	2974 (5)	8·1 (7)
C(22)	3354 (5)	866 (8)	2418 (5)	6·8 (6)
C(23)	2022 (4)	780 (7)	–63 (6)	4·8 (5)
C(24)	2118 (4)	170 (7)	–427 (6)	5·5 (5)
C(25)	1816 (4)	16 (7)	–933 (6)	6·9 (6)
C(26)	1418 (4)	470 (7)	–1075 (6)	8·6 (7)
C(27)	1323 (4)	1080 (7)	–711 (6)	9·8 (8)
C(28)	1625 (4)	1234 (7)	–204 (6)	6·2 (5)
C(29)	2046 (5)	1165 (7)	1142 (5)	5·2 (5)
C(30)	1714 (5)	597 (7)	1213 (5)	6·2 (6)
C(31)	1457 (5)	630 (7)	1687 (5)	7·4 (6)
C(32)	1533 (5)	1232 (7)	2091 (5)	6·5 (6)
C(33)	1865 (5)	1801 (7)	2020 (5)	6·7 (6)
C(34)	2122 (5)	1767 (7)	1546 (5)	6·0 (5)
C(35)	4592 (4)	3743 (7)	–405 (5)	3·9 (4)
C(36)	4353 (4)	4403 (7)	–258 (5)	5·7 (5)
C(37)	4460 (4)	5124 (7)	–482 (5)	6·2 (6)
C(38)	4805 (4)	5184 (7)	–853 (5)	6·9 (6)
C(39)	5045 (4)	4525 (7)	–1000 (5)	7·5 (7)
C(40)	4938 (4)	3804 (7)	–777 (5)	6·3 (6)
C(41)	4996 (4)	2572 (8)	350 (6)	4·6 (5)
C(42)	5014 (4)	2719 (8)	947 (6)	6·9 (6)
C(43)	5433 (4)	2627 (8)	1312 (6)	8·6 (7)
C(44)	5834 (4)	2388 (8)	1080 (6)	8·5 (7)
C(45)	5816 (4)	2242 (8)	483 (6)	8·0 (7)
C(46)	5397 (4)	2334 (8)	118 (6)	7·3 (7)
C(47)	4192 (5)	807 (7)	–1342 (4)	4·4 (4)
C(48)	4364 (5)	184 (7)	–996 (4)	5·6 (5)
C(49)	4575 (5)	–439 (7)	–1247 (4)	5·0 (5)
C(50)	4614 (5)	–438 (7)	–1844 (4)	6·4 (6)
C(51)	4442 (5)	185 (7)	–2190 (4)	6·8 (6)
C(52)	4231 (5)	808 (7)	–1939 (4)	5·3 (5)
C(53)	3654 (5)	2190 (6)	–1570 (6)	4·1 (4)
C(54)	3310 (5)	1820 (6)	–1950 (6)	5·5 (5)
C(55)	3070 (5)	2228 (6)	–2415 (6)	8·1 (7)
C(56)	3174 (5)	3006 (6)	–2500 (6)	9·2 (8)
C(57)	3517 (5)	3376 (6)	–2120 (6)	8·2 (7)
C(58)	3757 (5)	2968 (6)	–1655 (6)	5·7 (5)

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

Ir(1)—Ir(2)	2·692 (1)	C(1) O(1)	1·190 (24)
Ir(1)—Ir(3)	2·746 (1)	C(2)—O(2)	1·107 (27)
Ir(1)—Ir(4)	2·739 (1)	C(3)—O(3)	1·094 (26)
Ir(2)—Ir(3)	2·716 (1)	C(4)—O(4)	1·170 (25)
Ir(2)—Ir(4)	2·743 (1)	C(5)—O(5)	1·194 (24)
Ir(3)—Ir(4)	2·715 (1)	C(6)—O(6)	1·151 (21)
Ir(1)—P(1)	2·326 (5)	C(7)—O(7)	1·169 (25)
Ir(2)—P(2)	2·286 (5)	C(8)—O(8)	1·142 (25)
Ir(3)—P(3)	2·316 (5)	P(1)—C(11)	1·818 (14)
Ir(4)—P(4)	2·262 (5)	P(1)—C(17)	1·857 (13)
Ir(1)—C(1)	1·813 (18)	P(1)—C(9)	1·859 (16)
Ir(2)—C(2)	1·850 (20)	P(2)—C(9)	1·809 (17)
Ir(3)—C(3)	1·954 (22)	P(2)—C(23)	1·816 (12)
Ir(3)—C(5)	1·829 (20)	P(2)—C(29)	1·826 (15)
Ir(4)—C(4)	1·841 (19)	P(3)—C(35)	1·847 (13)
Ir(1)—C(7)	2·123 (21)	P(3)—C(41)	1·830 (13)
Ir(2)—C(7)	2·069 (19)	P(3)—C(10)	1·843 (21)
Ir(2)—C(6)	2·210 (20)	P(4)—C(10)	1·839 (19)
Ir(4)—C(6)	2·068 (17)	P(4)—C(47)	1·829 (13)
Ir(1)—C(8)	2·067 (20)	P(4)—C(53)	1·804 (13)
Ir(4)—C(8)	2·169 (19)		
Ir(2)—Ir(1)—Ir(3)	59·92 (2)	Ir(1)—Ir(4)—P(4)	133·3 (1)
Ir(2)—Ir(1)—Ir(4)	60·67 (2)	Ir(2)—Ir(4)—P(4)	138·4 (1)
Ir(3)—Ir(1)—Ir(4)	59·35 (2)	Ir(3)—Ir(4)—P(4)	90·5 (1)
Ir(1)—Ir(2)—Ir(3)	61·02 (2)	P(1)—Ir(1)—C(1)	100·9 (6)
Ir(1)—Ir(2)—Ir(4)	60·51 (2)	P(1)—Ir(1)—C(7)	93·1 (6)
Ir(3)—Ir(2)—Ir(4)	59·65 (2)	P(1)—Ir(1)—C(8)	104·6 (6)
Ir(1)—Ir(3)—Ir(2)	59·06 (2)	P(2)—Ir(2)—C(2)	96·6 (7)
Ir(1)—Ir(3)—Ir(4)	60·20 (2)	P(2)—Ir(2)—C(6)	95·4 (5)
Ir(2)—Ir(3)—Ir(4)	60·67 (2)	P(2)—Ir(2)—C(7)	98·0 (6)
Ir(1)—Ir(4)—Ir(2)	58·83 (2)	P(3)—Ir(3)—C(3)	99·5 (7)
Ir(1)—Ir(4)—Ir(3)	60·45 (2)	P(3)—Ir(3)—C(5)	98·7 (7)
Ir(2)—Ir(4)—Ir(3)	59·68 (2)	P(4)—Ir(4)—C(4)	95·5 (6)
Ir(2)—Ir(1)—P(1)	92·7 (1)	P(4)—Ir(4)—C(6)	100·4 (6)
Ir(3)—Ir(1)—P(1)	151·2 (1)	P(4)—Ir(4)—C(8)	93·8 (5)
Ir(4)—Ir(1)—P(1)	101·0 (1)	Ir(3)—Ir(4)—C(6)	84·7 (5)
Ir(1)—Ir(2)—P(2)	97·0 (1)	Ir(3)—Ir(1)—C(7)	76·1 (6)
Ir(3)—Ir(2)—P(2)	155·1 (1)	Ir(3)—Ir(1)—C(8)	80·1 (5)
Ir(4)—Ir(2)—P(2)	100·5 (1)	Ir(3)—Ir(1)—C(1)	106·9 (6)
Ir(1)—Ir(3)—P(3)	109·2 (1)	Ir(3)—Ir(2)—C(2)	108·3 (6)
Ir(2)—Ir(3)—P(3)	153·4 (1)	Ir(3)—Ir(4)—C(4)	171·4 (5)
Ir(4)—Ir(3)—P(3)	92·7 (1)	P(1)—C(9)—P(2)	114·4 (9)
P(3)—C(10)—P(4)	114·4 (11)		

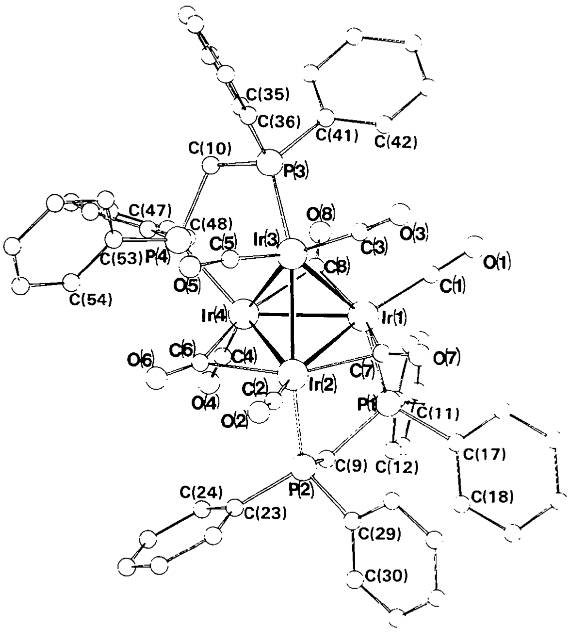

Fig. 1. Geometry and labelling of the complex $Ir_4(CO)_8(dppm)_2$; hydrogen atoms have been omitted.

Discussion. Atom parameters are given in Table 1* and selected bond lengths and angles in Table 2. As shown in Fig. 1, the molecule consists of a tetrahedron of Ir atoms, with the two dppm ligands coordinated in a bridging mode such that one carbonyl group on each Ir atom has been replaced by a phosphine. Also, three edges of the Ir_4 tetrahedron are symmetrically bridged by CO groups. Thus, Ir(1), Ir(2) and Ir(4) define a carbonyl-bridged basal plane, in which the bridging carbonyl groups are significantly tilted towards the apical Ir(3). The Ir—Ir bond lengths range from 2·692 (1) to 2·746 (1) Å, typical values for phosphine-substituted tetrahedral iridium carbonyl clusters (for example, Darensbourg & Baldwin-Zuschke, 1981), and slightly longer than the average value determined in the parent $Ir_4(CO)_{12}$ cluster (2·693 Å) (Churchill & Hutchinson, 1978), in which all the carbonyl ligands are terminally bound. It may be significant that the shortest Ir—Ir bond, Ir(1)—Ir(2), is between the Ir atoms that are bridged by both a carbonyl group and a dppm ligand. This feature was also observed in the analogous rhodium complex, $Rh_4(CO)_8(dppm)_2$ (Carré *et al.*, 1976), where the doubly bridged Rh atoms are separated by 2·671 (1) Å compared to a mean of 2·719 Å [range 2·696 (1)–2·740 (1) Å] for the other five Rh—Rh bond lengths.

The dppm ligands span the edges Ir(1)—Ir(2) and Ir(3)—Ir(4). P(1) and P(2) both occupy axial sites in the basal Ir_3 plane, while P(4) occupies an equatorial site in that plane. This stereochemistry of two axial and one equatorial phosphine groups is the same as that observed in monodentate phosphine-substituted Ir_4 carbonyl clusters (Darensbourg & Baldwin-Zuschke, 1981).

The molecular geometry does not appear to reveal any features which might account for the anomalous ^{31}P NMR chemical shifts of the phosphine groups in this complex.

We thank the SERC for an allocation of data sets on the X-ray crystallographic Service, and Dr M. B. Hursthouse for the data collection.

---

* Lists of structure factors, anisotropic thermal parameters and H-atom parameters have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39169 (31 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

References

Carré, F. H., Cotton, F. A. & Frenz, B. A. (1976). *Inorg. Chem.* **15**, 380–387.

Ceriotti, A., Ciani, G., Garlaschelli, L., Sartorelli, U. & Sironi, A. (1982). *J. Organomet. Chem.* **229**, C9–C12.

Churchill, M. R. & Hutchinson, J. P. (1978). *Inorg. Chem.* **17**, 3528–3535.

CHURCHILL, M. R. & LASHEWYCZ, R. A. (1978). *Inorg. Chem.* **17**, 1950–1957.
CROMER, D. T. & LIBERMAN, D. (1970). *J. Chem. Phys.* **53**, 1891–1898.
DARENSBOURG, D. J. & BALDWIN-ZUSCHKE, B. J. (1981). *Inorg. Chem.* **20**, 3846–3850.
FOSTER, D. F., NICHOLLS, B. S. & SMITH, A. K. (1982). *J. Organomet. Chem.* **236**, 395–402.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press.
LAVIGNE, G., LUGAN, N. & BONNET, J.-J. (1982). *Acta Cryst.* B**38**, 1911–1916.
MOTHERWELL, W. D. S. (1976). *PLUTO*. A program for plotting molecular and crystal structures. Univ. of Cambridge, England.
SHAPLEY, J. R., STUNTZ, G. F., CHURCHILL, M. R. & HUTCHINSON, J. P. (1979). *J. Am. Chem. Soc.* **101**, 7425–7428.
SHELDRICK, G. M. (1976). *SHELX*. Program for crystal structure determination. Univ. of Cambridge, England.
STEFFEN, W. L. & PALENIK, G. S. (1976). *Inorg. Chem.* **15**, 2432–2439.

Acta Cryst. (1984). C**40**, 793–795

trans-Dichloro(dimethyl sulfoxide)(isopropylamine)platinum(II), $C_5H_{15}Cl_2NOPtS$

BY ROBERT MELANSON AND F. D. ROCHON

Département de Chimie, Université du Québec à Montréal, CP 8888, *Succ. A, Montréal, Canada H3C* 3*P*8

(*Received* 17 *May* 1983; *accepted* 11 *January* 1984)

Abstract. $M_r = 403{\cdot}24$, triclinic, $P\bar{1}$, $a = 7{\cdot}789\,(4)$, $b = 8{\cdot}781\,(5)$, $c = 8{\cdot}874\,(7)$ Å, $\alpha = 108{\cdot}23\,(5)$, $\beta = 101{\cdot}67\,(5)$, $\gamma = 93{\cdot}05\,(4)^\circ$, $V = 560{\cdot}1\,(6)$ Å^3, $Z = 2$, $D_x = 2{\cdot}390$, $D_m = 2{\cdot}38\,(2)$ Mg m^{-3}, $\lambda(\text{Mo } K\alpha) = 0{\cdot}71069$ Å, $\mu = 13{\cdot}778$ mm^{-1}, $F(000) = 376$, $T = 295$ K. Final $R = 0{\cdot}035$ for 2829 unique observed reflections. The coordination around the Pt atom is square-planar. The compound which was synthesized in our laboratory is the *trans*-isomer. The Pt—Cl bond distances are 2·290 (2) and 2·315 (2) Å, the Pt—N bond is 2·067 (6) Å while the Pt—S bond is 2·220 (2) Å. The crystal is stabilized by intermolecular hydrogen bonds.

Introduction. Continuing our studies on (Me_2SO)(amine)platinum compounds, we have synthesized *trans*-[Pt(Me_2SO)(isopropylamine)Cl_2] and determined its molecular and crystal structure. It was prepared from the aqueous reaction of K[Pt(Me_2SO)Cl_3] with isopropylamine. In these conditions, only the *trans*-isomer is produced.

Experimental. Crystals recrystallized from water; density by flotation in a thallous malonate aqueous solution; hexagonal plate 0·6 mm by 0·08 mm thick; precession photographs indicated triclinic system; Syntex $P\bar{1}$ diffractometer; graphite-monochromatized Mo $K\alpha$ radiation; cell parameters from setting angles of 15 centered reflections ($10 < 2\theta < 25^\circ$); 3626 independent reflections measured up to $2\theta < 60^\circ$ by θ–2θ scan technique; range of hkl: h $-10\rightarrow10$, k $-12\rightarrow12$, l $-12\rightarrow11$; standard reflections 130, 410 and $0\bar{1}4$: variations <3%; reflections with $I_{\text{net}} < 2{\cdot}5\sigma(I)$ unobserved, $\sigma(I)$ calculated as in Melanson & Rochon (1975); absorption correction based on equations of crystal faces, transmission factors: 0·031 to 0·309; data corrected for Lorentz and polarization effects; 2829 unique observed reflections. Patterson map indicated space group $P\bar{1}$ and position of Pt; positions of other atoms (except H) obtained by structure-factor and Fourier-map calculations; H, except those in CH_3, fixed at calculated positions (C—H = 0·95 and N—H = 0·85 Å) with isotropic $\beta = 6{\cdot}0$ Å^2. $R = 0{\cdot}035$, $R_w = 0{\cdot}049$; $w = 1/\sigma^2(F)$; $(\Delta/\sigma)_{\max}$ in final refinement cycle <0·008; $\rho_{\max} = 1{\cdot}6$ (close to Pt), $\rho_{\min} = -0{\cdot}5$ e Å^{-3} in final difference Fourier synthesis. Isotropic secondary-extinction correction (Coppens & Hamilton, 1970). Atomic scattering factors of Cromer & Waber (1965) for Pt, Cl, S, O, N and of Stewart, Davidson & Simpson (1965) for H; anomalous-dispersion terms of Pt, Cl and S from Cromer (1965). Calculations on a Cyber 171 with programs of Melanson & Rochon (1975).

Discussion. The refined atomic parameters are listed in Table 1.* A labeled diagram of the molecule is shown in Fig. 1. The coordination around the Pt atom is square-planar. The deviations from the weighted coordination plane are: Pt, 0·0007 (3); Cl(1), −0·017 (2); Cl(2), −0·013 (2); S, −0·007 (2); and N, −0·076 (6) Å. The bond lengths and angles are given in Table 2. The angles around the Pt atom are close to the expected values of 90 and 180°.

---

* Lists of structure factors, anisotropic thermal parameters and coordinates of the H atoms have been deposited with the British Library Lending Division as Supplementary Publication No. SUP 39166 (15 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

0108-2701/84/050793-03\$01.50