

C₉H₁₈N₃O⁻ and by Podlahová, Knížek, Loub & Hašek (1988) for C₁₁H₁₆N₃O⁻. The assumed hydrogen bonds are in agreement with infrared spectra, in which N—H vibrations are shifted to lower frequencies compared with [Ni(C₁₁H₁₆N₃O)]ClO₄ (Podlahová *et al.*, 1988), where no hydrogen bonds occur (3303→3253, 3254→3225, 3207→3121 cm⁻¹). O—H vibrations are shifted to lower frequencies when compared with H₂O in the matrix (Tursi & Nixon, 1970) (3725→3494, 3632→3430 cm⁻¹).

Acta Cryst. (1989). **C45**, 1218–1219

Structure of *trans*-Di- μ -chloro-dichlorobis(triethylphosphine)diplatinum

BY ALEXANDER J. BLAKE,* ROBERT O. GOULD, ALASTAIR M. MARR, DAVID W. H. RANKIN
AND MARTIN SCHRÖDER

Department of Chemistry, University of Edinburgh, West Mains Road, Edinburgh EH9 3JJ, Scotland

(Received 3 October 1988; accepted 5 January 1989)

Abstract. [Pt₂Cl₄{P(C₂H₅)₃}₂], *M_r* = 768.31, monoclinic, *P*2₁/*c*, *a* = 7.6461 (6), *b* = 12.1981 (12), *c* = 12.9280 (20) Å, β = 113.226 (6)°, *V* = 1108.0 Å³, *Z* = 2 (implying that each dimer lies on a crystallographic inversion centre), *D_x* = 2.302 Mg m⁻³, λ (Mo *K* α) = 0.71073 Å, μ = 13.37 mm⁻¹, *F*(000) = 712, *T* = 293 K, *R* = 0.0353 for 1275 unique observed reflections. The chloro-bridged dimer is centrosymmetric, with Pt—Cl distances of 2.282 (3) Å (terminal) and 2.318 (3), 2.431 (3) Å (bridging). Angles around Pt range from 83.52 (9)° for Cl(1)—Pt—Cl(1') to 95.93 (9)° for P—Pt—Cl(1), while the angle subtended at the bridging Cl is 96.48 (10)°.

Experimental. Lamellar yellow crystal, 0.38 × 0.35 × 0.08 mm, Stoe STADI-4 diffractometer, graphite-monochromated Mo*K* α X-radiation, cell parameters from 40 reflections measured at $\pm\omega$ ($2\theta = 25$ – 30°). For data collection, ω – 2θ scans, $2\theta_{\max} = 45^\circ$, *h* –8→7, *k* 0→13, *l* 0→13, no significant crystal movement or decay, semi-empirical absorption correction applied using ψ scans, 1453 unique reflections giving 1275 with $F \geq 6\sigma(F)$ for structure solution (the Pt position was deduced from a Patterson synthesis and subsequent iterative cycles of least-squares refinement and difference Fourier synthesis located the remaining non-H atoms) and refinement [using full-matrix least squares on *F* (Sheldrick, 1976)]. Anisotropic thermal parameters for Pt, Cl and P, isotropic for C, H atoms refined in fixed, calculated positions. At convergence, *R*, *wR* = 0.0353, 0.0512, *S* = 0.658 for 61 parameters,

* Author for correspondence.

References

- HABER, V., LOUB, J., PODLAHOVÁ, J., KOPF, J. & WEISS, E. (1988). *Acta Cryst.* **C44**, 1905–1907.
International Tables for X-ray Crystallography (1974). Vol. IV. Birmingham: Kynoch Press. (Present distributor Kluwer Academic Publishers, Dordrecht.)
 PODLAHOVÁ, J., KNÍŽEK, K., LOUB, J. & HAŠEK, J. (1988). *Acta Cryst.* **C44**, 631–633.
 SHELDRIK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 TURSI, A. J. & NIXON, E. R. (1970). *J. Chem. Phys.* **52**, 1521–1528.

(Δ/σ)_{max} in final cycle 0.021, max. and min. residues in final difference Fourier synthesis 1.20, –1.77 e Å⁻³ respectively. The weighting scheme $w^{-1} = \sigma^2(F) + 0.00545F^2$ gave satisfactory agreement analyses. Scattering factors were inlaid (Sheldrick, 1976) except for Pt (Cromer & Mann, 1968). Atom coordinates and equivalent isotropic thermal parameters are given in Table 1 while selected molecular geometry parameters appear in Table 2.† The atom-numbering scheme for the dimer is shown in Fig. 1, which was generated using *ORTEP* (Mallinson & Muir, 1985). Molecular geometry calculations were performed using *CALC* (Gould & Taylor, 1985).

† Lists of structure factors, anisotropic thermal parameters and H-atom coordinates have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 51831 (10 pp.). Copies may be obtained through The Executive Secretary, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

Table 1. Atomic coordinates and isotropic thermal parameters with e.s.d.'s

$$U_{eq} = \frac{1}{3} \sum_i \sum_j U_{ij} a_i^* a_j^* a_i \cdot a_j$$

	<i>x</i>	<i>y</i>	<i>z</i>	<i>U_{eq}/U_{iso}</i> (Å ²)
Pt	0.48880 (4)	0.10145 (3)	0.095240 (20)	0.0386 (4)
P	0.3322 (3)	0.13401 (22)	0.20394 (19)	0.0459 (15)
Cl(1)	0.3440 (4)	–0.06527 (21)	0.02682 (20)	0.0599 (17)
Cl(2)	0.6502 (4)	0.26218 (20)	0.15425 (22)	0.0658 (17)
C(1A)	0.1605 (11)	0.0277 (7)	0.1937 (7)	0.0435 (20)
C(1B)	0.4880 (14)	0.1476 (10)	0.3485 (9)	0.062 (3)
C(1C)	0.1981 (15)	0.2602 (10)	0.1678 (9)	0.067 (3)
C(2A)	0.0543 (15)	0.0409 (10)	0.2718 (8)	0.069 (3)
C(2B)	0.6149 (16)	0.0485 (10)	0.4002 (9)	0.071 (3)
C(2C)	0.0840 (17)	0.2732 (10)	0.0470 (10)	0.081 (3)

Table 2. Bond lengths (Å), angles (°) and torsion angles (°) with *e.s.d.*'s

Pt—P	2.212 (3)	P—C(1B)	1.784 (12)
Pt—Cl(1)	2.318 (3)	P—C(1C)	1.806 (12)
Pt—Cl(2)	2.282 (3)	C(1A)—C(2A)	1.534 (15)
Pt—Cl(1')	2.431 (3)	C(1B)—C(2B)	1.530 (17)
P—C(1A)	1.813 (9)	C(1C)—C(2C)	1.467 (17)
P—Pt—Cl(1)	95.93 (9)	Pt—P—C(1C)	112.5 (4)
P—Pt—Cl(2)	89.43 (10)	C(1A)—P—C(1B)	108.0 (5)
P—Pt—Cl(1')	179.07 (9)	C(1A)—P—C(1C)	105.7 (5)
Cl(1)—Pt—Cl(2)	174.59 (9)	C(1B)—P—C(1C)	105.5 (5)
Cl(1)—Pt—Cl(1')	83.52 (9)	Pt—Cl(1)—Pt'	96.48 (10)
Cl(2)—Pt—Cl(1')	91.12 (9)	P—C(1A)—C(2A)	115.7 (7)
Pt—P—C(1A)	112.5 (3)	P—C(1B)—C(2B)	115.5 (8)
Pt—P—C(1B)	112.2 (4)	P—C(1C)—C(2C)	113.9 (9)
Cl(1)—Pt—P—C(1A)	-1.7 (3)	Pt—P—C(1A)—C(2A)	176.6 (6)
Cl(1)—Pt—P—C(1B)	120.2 (4)	C(1B)—P—C(1A)—C(2A)	52.2 (8)
Cl(1)—Pt—P—C(1C)	-121.0 (4)	C(1C)—P—C(1A)—C(2A)	-60.3 (8)
Cl(2)—Pt—P—C(1A)	179.0 (3)	Pt—P—C(1B)—C(2B)	-58.6 (9)
Cl(2)—Pt—P—C(1B)	-59.1 (4)	C(1A)—P—C(1B)—C(2B)	66.0 (9)
Cl(2)—Pt—P—C(1C)	59.8 (4)	C(1C)—P—C(1B)—C(2B)	178.6 (8)
P—Pt—Cl(1)—Pt'	179.24 (9)	Pt—P—C(1C)—C(2C)	47.6 (10)
Cl(2)—Pt—Cl(1)—Pt'	-8.5 (10)	C(1A)—P—C(1C)—C(2C)	-75.5 (9)
Cl(2)—Pt—Cl(1')—Pt'	179.21 (10)	C(1B)—P—C(1C)—C(2C)	170.3 (9)

Related literature. The heavy-atom (Pt,Cl,P) skeletons of the corresponding complexes with tri-*n*-propylphosphine (Black, Mais & Owston, 1969) and di-*tert*-butylcyclopropylphosphine (Simms, Shang, Jiayi Lu, Youngs & Ibers, 1987) exhibit similar geometries.

We thank the University of Edinburgh for a Studentship (to AMM) and the SERC for funding towards the purchase of a four-circle diffractometer.

Acta Cryst. (1989). **C45**, 1219–1221

Bis[η^5 -bis(trimethylsilyl)cyclopentadienyl]chlorouranium(III) Bis(trimethylsilyl cyanide)

BY ALLAN ZALKIN AND SHARON M. BESHOURI

Materials and Chemical Sciences Division, Lawrence Berkeley Laboratory, 1 Cyclotron Rd, Berkeley, CA 94720, USA

(Received 9 November 1988; accepted 31 January 1989)

Abstract. [UCl{C₅H₃[Si(CH₃)₃]₂}]₂[NCSi(CH₃)₃]₂, [UCl(C₁₁H₂₁Si₂)(C₄H₉NSi)₂], *M_r* = 890.83, monoclinic, *P*2₁/*n*, *a* = 11.602 (2), *b* = 36.947 (5), *c* = 10.999 (2) Å, β = 99.31 (2)°, *V* = 4652.7 Å³, *Z* = 4, *D_x* = 1.272 g cm⁻³, λ (Cu *K*α) = 1.54180 Å, μ = 127.6 cm⁻¹, *F*(000) = 1788, *T* = 296 K, *R* = 0.041 for 4138 *F*² > 2σ(*F*²) of 6929 total unique data. The U atom is coordinated to five ligands with distances: U—Cl 2.698 (3); U—Cp 2.510, 2.520; U—N(cyanide) 2.592 (11), 2.619 (10); ⟨U—C(cyclopentadiene)⟩ 2.789 (22) Å.

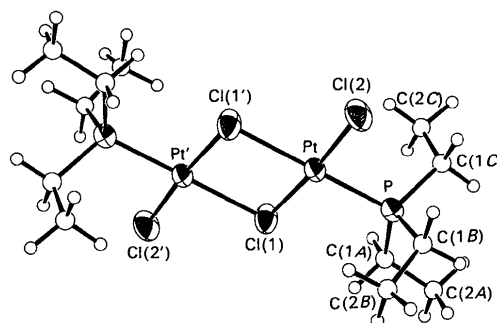
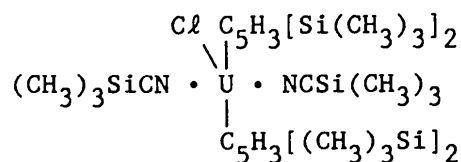


Fig. 1. View of the molecule showing atom numbering scheme. Thermal ellipsoids are drawn at the 30% probability level, excepting those of C and H which have artificial radii of 0.15 and 0.10 Å, respectively, for clarity.

References

- BLACK, M., MAIS, R. H. B. & OWSTON, P. G. (1969). *Acta Cryst.* **25**, 1760–1766.
 CROMER, D. T. & MANN, J. L. (1968). *Acta Cryst.* **A24**, 321–324.
 GOULD, R. O. & TAYLOR, P. (1985). *CALC*. Program for molecular geometry calculations, Fortran77 version. Univ. of Edinburgh, Scotland.
 MALLINSON, P. D. & MUIR, K. W. (1985). *ORTEPII*, interactive version, *J. Appl. Cryst.* **18**, 51–53.
 SHELDRICK, G. M. (1976). *SHELX76*. Program for crystal structure determination. Univ. of Cambridge, England.
 SIMMS, B. L., SHANG, M., JIAXI LU, YOUNGS, W. J. & IBERS, J. A. (1987). *Organometallics*, **6**, 1118–1126.

Experimental. The title compound, shown below, was



prepared by reaction of one equivalent of [UCl{C₅H₃[Si(CH₃)₃]₂}]₂ with four equivalents of NCSi(CH₃)₃ in diethyl ether solution. The product was