

C(32)	-0.0218 (12)	0.4371 (18)	0.1349 (17)	0.071 (6)
C(33)	0.0679 (12)	0.4047 (19)	0.3034 (17)	0.075 (7)
C(1p)	0.1855 (12)	0.1464 (18)	0.2451 (18)	0.076 (6)
O(11)	0.2938 (9)	0.4291 (14)	0.2571 (13)	0.094 (5)
O(12)	0.2299 (10)	0.5268 (14)	-0.0338 (13)	0.094 (5)
O(13)	0.3335 (10)	0.2404 (14)	0.0169 (13)	0.095 (6)
O(21)	-0.0521 (9)	0.0903 (14)	0.0874 (12)	0.085 (5)
O(22)	-0.0411 (9)	0.3444 (12)	-0.0931 (12)	0.080 (5)
O(23)	0.1494 (9)	0.1025 (13)	-0.1042 (12)	0.084 (5)
O(31)	-0.0902 (9)	0.2345 (14)	0.2526 (13)	0.095 (5)
O(32)	-0.0628 (10)	0.4918 (15)	0.0946 (14)	0.102 (6)
O(33)	0.0874 (10)	0.4390 (14)	0.3750 (14)	0.097 (5)

Table 6. Geometric parameters (Å, °) for (3)

Os(1)—Os(2)	2.9776 (11)	Os(3)—C(31)	1.83 (2)
Os(1)—Os(3)	2.9741 (11)	Os(3)—C(32)	1.87 (2)
Os(2)—Os(3)	2.8547 (11)	Os(3)—C(33)	1.86 (3)
Os(1)—P(1)	2.350 (5)	C(11)—O(11)	1.18 (3)
Os(2)—P(1)	2.318 (5)	C(12)—O(12)	1.14 (3)
Os(3)—P(1)	2.317 (5)	C(13)—O(13)	1.16 (3)
P(1)—C(1p)	1.83 (3)	C(21)—O(21)	1.10 (3)
Os(1)—C(11)	1.87 (2)	C(22)—O(22)	1.16 (3)
Os(1)—C(12)	1.92 (3)	C(23)—O(23)	1.16 (3)
Os(1)—C(13)	1.88 (2)	C(31)—O(31)	1.19 (3)
Os(2)—C(21)	1.92 (2)	C(32)—O(32)	1.20 (3)
Os(2)—C(22)	1.92 (2)	C(33)—O(33)	1.19 (3)
Os(2)—C(23)	1.86 (2)		
Os(2)—Os(1)—Os(3)	57.33 (3)	Os(1)—Os(2)—C(21)	146.5 (6)
Os(1)—Os(2)—Os(3)	61.28 (3)	Os(1)—Os(2)—C(22)	109.3 (7)
Os(1)—Os(3)—Os(2)	61.40 (3)	Os(1)—Os(2)—C(23)	102.9 (6)
Os(2)—Os(1)—P(1)	49.89 (13)	Os(3)—Os(2)—C(21)	94.7 (6)
Os(3)—Os(1)—P(1)	49.92 (13)	Os(3)—Os(2)—C(22)	96.0 (7)
Os(1)—Os(2)—P(1)	50.84 (13)	Os(3)—Os(2)—C(23)	160.7 (6)
Os(3)—Os(2)—P(1)	51.96 (13)	Os(1)—Os(3)—C(31)	147.8 (6)
Os(1)—Os(3)—P(1)	50.90 (13)	Os(1)—Os(3)—C(32)	109.5 (7)
Os(2)—Os(3)—P(1)	52.00 (13)	Os(1)—Os(3)—C(33)	103.70 (8)
Os(1)—P(1)—Os(2)	79.27 (17)	Os(2)—Os(3)—C(31)	92.3 (6)
Os(1)—P(1)—Os(3)	79.18 (17)	Os(2)—Os(3)—C(32)	98.3 (7)
Os(1)—P(1)—C(1p)	127.5 (8)	Os(2)—Os(3)—C(33)	157.3 (8)
Os(2)—P(1)—Os(3)	76.04 (16)	C(11)—Os(1)—C(12)	97.1 (10)
Os(2)—P(1)—C(1p)	136.7 (8)	C(11)—Os(1)—C(13)	91.5 (10)
Os(3)—P(1)—C(1p)	135.6 (8)	C(12)—Os(1)—C(13)	98.0 (10)
P(1)—Os(1)—C(11)	95.3 (7)	C(21)—Os(2)—C(22)	95.4 (9)
P(1)—Os(1)—C(12)	162.4 (8)	C(21)—Os(2)—C(23)	94.5 (9)
P(1)—Os(1)—C(13)	94.0 (7)	C(22)—Os(2)—C(23)	100.0 (9)
P(1)—Os(2)—C(22)	146.7 (7)	C(31)—Os(3)—C(32)	91.4 (10)
P(1)—Os(2)—C(23)	110.1 (7)	C(31)—Os(3)—C(33)	94.7 (10)
P(1)—Os(2)—C(21)	96.3 (6)	C(32)—Os(3)—C(33)	103.0 (11)
P(1)—Os(3)—C(31)	98.9 (6)	Os(1)—C(11)—O(11)	175 (2)
P(1)—Os(3)—C(32)	148.6 (7)	Os(1)—C(12)—O(12)	175 (2)
P(1)—Os(3)—C(33)	105.5 (8)	Os(1)—C(13)—O(13)	177 (2)
Os(2)—Os(1)—C(11)	144.6 (7)	Os(2)—C(21)—O(21)	177.0 (19)
Os(2)—Os(1)—C(12)	115.6 (8)	Os(2)—C(22)—O(22)	175 (2)
Os(2)—Os(1)—C(13)	96.8 (7)	Os(2)—C(23)—O(23)	178.8 (19)
Os(3)—Os(1)—C(11)	97.2 (7)	Os(3)—C(31)—O(31)	175.9 (18)
Os(3)—Os(1)—C(12)	115.9 (8)	Os(3)—C(32)—O(32)	175 (2)
Os(3)—Os(1)—C(13)	143.4 (7)	Os(3)—C(33)—O(33)	175 (2)

The structure of (1) was solved by direct methods using *SHELXS84* (Sheldrick, 1984). The methyl groups of the phosphine were each found to be disordered over two sites, such that the occupancy of C(11) and C(12) refined to 0.606 (9) and that of the alternative positions represented by C(11') and C(12') to 0.394 (9). Only the Os atoms were allowed anisotropic thermal motion. The absolute structure was determined and is presented here; the other polarity gave higher residuals *R* and *wR* of 0.0450 and 0.0493, respectively, and higher errors in atomic positional and displacement parameters. The structures of compounds (2) and (3) were solved similarly. The atoms allowed anisotropic thermal motion were Os, P and O for (2) and Os and P for (3). The programs used were *SHELX76* (Sheldrick, 1976) and *SHELXS86* (Sheldrick, 1985). *CALC* (Gould & Taylor, 1985) was used for molecular geometry calculations.

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Lists of structure factors and anisotropic displacement parameters have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71506 (36 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1044]

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## Tetra-*n*-butylammonium Tris(perfluorophenyl)(triphenylphosphine)platinate(II)

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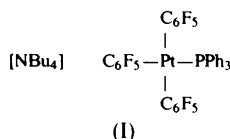
## Abstract

The coordination geometry of the ligands around the Pt atom is approximately square planar in  $[\text{N}(\text{C}_4\text{H}_9)_4][\text{Pt}(\text{C}_6\text{F}_5)_3(\text{C}_6\text{H}_5)_3\text{P}]$ , with Pt—C dis-

tances in the range 2.038 (11)–2.066 (10) Å and the Pt—P distance 2.279 (3) Å. Bond angles between adjacent ligands range from 86.2 (4) to 94.6 (3)°.

### Comment

Many cationic or neutral mononuclear Pt derivatives containing triphenylphosphine (PPh<sub>3</sub>) as a ligand have been reported in the literature; however, anionic complexes are practically unknown. The structure determination of the title compound (I) was



undertaken in order to establish structural differences, if any, with known cationic or neutral complexes. Since the Pt—P bond has a certain degree of  $\pi$  back-donation it would be expected that an anionic complex could allow more electron density to drift towards the P atom, thus resulting in shorter distances. Details of synthetic work have been reported previously (Usón, Forniés, Martínez & Tomás, 1980; Usón, Forniés, Tomás & Fandos, 1984).

The Pt atom is located in an approximately square-planar environment formed by three C atoms [C(1), C(7) and C(13)] and one P atom. Bond lengths are Pt—C(1) = 2.066 (10), Pt—C(7) = 2.057 (10), Pt—C(13) = 2.038 (11) and Pt—P = 2.279 (3) Å. All Pt—C distances can be considered identical within experimental error. Bond angles between the Pt and two adjacent atoms range from 86.2 (4) to 94.6 (3)°, while the *trans* bond angles are 173.6 (4) and 176.7 (3)°. Bond distances and angles within the pentafluorophenyl and phenyl rings are consistent with the corresponding values usually shown by these groups when coordinated to Pt centres (Usón & Forniés, 1988). As the space group is non-centrosymmetric, the enantiomorph was chosen on the basis of a lower weighted *R* factor.

A search of the Cambridge Structural Database showed a large number of mononuclear complexes in which the Pt—P distances are comparable with the one in the present structure, even though they correspond to neutral or cationic complexes. However, a tendency to longer distances in cationic complexes is observed, with an average value larger than 2.300 Å. As far as we know, the only structurally characterized triphenylphosphineplatinum(II) anionic complex shows a Pt—P distance of 2.236 Å (de Renzi, di Blasio, Panunzi, Pedone & Vitagliano, 1976), which is shorter than the distance found in the present study. This may be due to the different *trans* influ-

ence of the ligands, which, together with the charge of the complex, leads to the distance found in the structure.

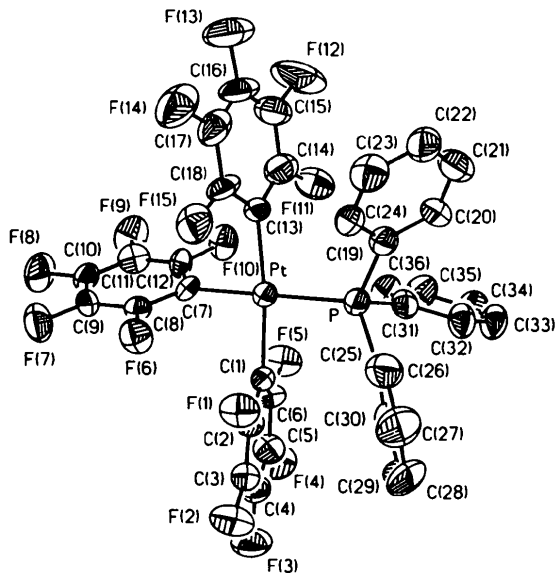


Fig. 1. Structure and labelling scheme of the complex anion [Pt(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>(PPh<sub>3</sub>)]<sup>-</sup>. Atoms are represented by their 40% probability ellipsoids.

### Experimental

#### Crystal data

[N(C<sub>4</sub>H<sub>9</sub>)<sub>4</sub>][Pt(C<sub>6</sub>F<sub>5</sub>)<sub>3</sub>-(C<sub>18</sub>H<sub>15</sub>P)]  
*M<sub>r</sub>* = 1201.023  
 Orthorhombic  
*P*2<sub>1</sub>2<sub>1</sub>  
*a* = 11.903 (2) Å  
*b* = 20.019 (4) Å  
*c* = 21.640 (3) Å  
*V* = 5156.3 (15) Å<sup>3</sup>  
*Z* = 4  
*D<sub>x</sub>* = 1.539 Mg m<sup>-3</sup>

Mo *K*α radiation  
 $\lambda$  = 0.71069 Å  
 Cell parameters from 48 reflections  
 $\theta$  = 11.5–15°  
 $\mu$  = 2.841 mm<sup>-1</sup>  
*T* = 291 K  
 Prismatic  
 0.27 × 0.06 × 0.06 mm  
 Colourless  
 Crystal source:  
 dichloromethane/hexane

#### Data collection

Stoe Siemens AED-2 diffractometer  
 $\omega/\theta$  scans  
 Absorption correction: none  
 10 322 measured reflections  
 8474 independent reflections  
 5025 observed reflections  
 [*F* > 6.0 $\sigma$ (*F*)]

*R*<sub>int</sub> = 2.82  
 $\theta_{\text{max}}$  = 25°  
*h* = -14 → 14  
*k* = -23 → 23  
*l* = -25 → 25  
 3 standard reflections  
 frequency: 45 min  
 intensity variation: none

#### Refinement

Refinement on *F*  
*R* = 0.0391

$\Delta\rho_{\text{max}}$  = 0.99 e Å<sup>-3</sup>  
 $\Delta\rho_{\text{min}}$  = -0.61 e Å<sup>-3</sup>

$wR = 0.0411$   
 $S = 1.0387$   
 5025 reflections  
 636 parameters  
 Only H-atom  $U$ 's refined  
 $w = 1/[\sigma^2(F) + 0.0004F^2]$   
 $(\Delta/\sigma)_{\max} = -0.115$

Extinction correction: none  
 Atomic scattering factors  
 from *International Tables*  
 for *X-ray Crystallography*  
 (1974, Vol. IV, pp. 55, 99,  
 149)

C(43)	-0.2513 (18)	0.0996 (13)	0.4470 (10)	0.188 (14)
C(44)	-0.3441 (31)	0.0607 (16)	0.4431 (14)	0.280 (22)
C(45)	-0.0006 (12)	0.1071 (8)	0.6024 (8)	0.091 (7)
C(46)	-0.0414 (12)	0.1766 (8)	0.6193 (8)	0.107 (8)
C(47)	0.0483 (14)	0.2287 (8)	0.6240 (8)	0.116 (8)
C(48)	0.0036 (17)	0.2975 (8)	0.6398 (9)	0.138 (9)
C(49)	-0.1397 (12)	0.0440 (7)	0.6610 (6)	0.092 (6)
C(50)	-0.0639 (11)	0.0208 (9)	0.7127 (6)	0.108 (7)
C(51)	-0.1252 (17)	0.0258 (9)	0.7753 (7)	0.136 (9)
C(52)	-0.1307 (22)	0.0876 (10)	0.8034 (10)	0.211 (16)

Table 1. Fractional atomic coordinates and equivalent isotropic displacement parameters ( $\text{\AA}^2$ )

$$U_{eq} = (1/3)\sum_i \sum_j U_{ij} a_i^* a_j^* \mathbf{a}_i \cdot \mathbf{a}_j$$

	$x$	$y$	$z$	$U_{eq}$
Pt	0.1002 (1)	-0.0113 (1)	0.1820 (1)	0.047 (1)
P	0.1293 (3)	0.0848 (1)	0.2361 (2)	0.053 (1)
F(1)	0.2802 (6)	0.0469 (4)	0.0869 (3)	0.078 (3)
F(2)	0.2643 (8)	0.1088 (4)	-0.0210 (4)	0.107 (4)
F(3)	0.0586 (8)	0.1380 (4)	-0.0701 (3)	0.114 (4)
F(4)	-0.1286 (7)	0.1029 (5)	-0.0068 (4)	0.108 (4)
F(5)	-0.1138 (6)	0.0401 (4)	0.1011 (3)	0.084 (3)
F(6)	0.2400 (6)	-0.1018 (4)	0.0825 (4)	0.076 (3)
F(7)	0.1863 (8)	-0.2141 (4)	0.0230 (4)	0.101 (4)
F(8)	-0.0176 (7)	-0.2711 (3)	0.0377 (4)	0.104 (4)
F(9)	-0.1690 (7)	-0.2145 (4)	0.1150 (4)	0.099 (4)
F(10)	-0.1181 (5)	-0.1043 (3)	0.1774 (4)	0.077 (3)
F(11)	-0.0594 (8)	-0.0438 (5)	0.3004 (4)	0.112 (4)
F(12)	-0.0474 (12)	-0.1300 (6)	0.3916 (5)	0.186 (7)
F(13)	0.1398 (12)	-0.2098 (4)	0.4016 (4)	0.178 (7)
F(14)	0.3135 (10)	-0.1944 (4)	0.3209 (5)	0.147 (5)
F(15)	0.2973 (7)	-0.1090 (4)	0.2270 (4)	0.095 (3)
C(1)	0.0861 (10)	0.0409 (4)	0.0999 (5)	0.045 (4)
C(2)	0.1776 (11)	0.0594 (7)	0.0654 (6)	0.056 (5)
C(3)	0.1685 (13)	0.0906 (7)	0.0103 (6)	0.066 (6)
C(4)	0.0656 (15)	0.1067 (6)	-0.0137 (6)	0.077 (7)
C(5)	-0.0278 (13)	0.0901 (7)	0.0182 (7)	0.073 (6)
C(6)	-0.0141 (11)	0.0566 (6)	0.0733 (6)	0.057 (5)
C(7)	0.0643 (9)	-0.0967 (5)	0.1330 (5)	0.044 (4)
C(8)	0.1361 (10)	-0.1274 (5)	0.0930 (5)	0.050 (4)
C(9)	0.1103 (13)	-0.1849 (5)	0.0609 (5)	0.067 (5)
C(10)	0.0058 (13)	-0.2150 (6)	0.0684 (6)	0.067 (5)
C(11)	-0.0679 (10)	-0.1848 (7)	0.1077 (6)	0.066 (5)
C(12)	-0.0371 (11)	-0.1290 (5)	0.1389 (6)	0.056 (5)
C(13)	0.1164 (12)	-0.0721 (5)	0.2570 (5)	0.053 (5)
C(14)	0.0359 (15)	-0.0806 (7)	0.3016 (7)	0.084 (7)
C(15)	0.0389 (21)	-0.1259 (9)	0.3507 (8)	0.110 (9)
C(16)	0.1332 (25)	-0.1640 (9)	0.3560 (8)	0.120 (12)
C(17)	0.2171 (18)	-0.1562 (7)	0.3151 (9)	0.104 (8)
C(18)	0.2095 (14)	-0.1114 (6)	0.2663 (7)	0.069 (6)
C(19)	0.1926 (10)	0.0743 (5)	0.3130 (7)	0.059 (4)
C(20)	0.1503 (12)	0.1046 (6)	0.3651 (7)	0.071 (6)
C(21)	0.2042 (14)	0.0951 (7)	0.4209 (6)	0.086 (6)
C(22)	0.2989 (14)	0.0567 (7)	0.4265 (7)	0.085 (6)
C(23)	0.3394 (11)	0.0265 (6)	0.3754 (7)	0.082 (6)
C(24)	0.2878 (10)	0.0341 (5)	0.3198 (7)	0.065 (4)
C(25)	0.2174 (12)	0.1488 (6)	0.2003 (5)	0.062 (5)
C(26)	0.3284 (16)	0.1583 (7)	0.2156 (7)	0.094 (7)
C(27)	0.3942 (17)	0.2046 (8)	0.1848 (9)	0.127 (8)
C(28)	0.3495 (21)	0.2428 (8)	0.1396 (10)	0.131 (11)
C(29)	0.2382 (20)	0.2354 (8)	0.1234 (8)	0.117 (9)
C(30)	0.1708 (15)	0.1877 (6)	0.1525 (6)	0.088 (6)
C(31)	-0.0003 (13)	0.1298 (7)	0.2494 (5)	0.0064 (5)
C(32)	0.0006 (13)	0.1972 (7)	0.2702 (6)	0.076 (6)
C(33)	-0.0966 (16)	0.2300 (6)	0.2800 (6)	0.083 (6)
C(34)	-0.1962 (16)	0.1998 (8)	0.2703 (7)	0.091 (7)
C(35)	-0.2011 (13)	0.1345 (8)	0.2502 (7)	0.089 (7)
C(36)	-0.1009 (14)	0.0995 (6)	0.2400 (6)	0.074 (5)
N	-0.0863 (10)	0.0527 (5)	0.5989 (5)	0.072 (4)
C(37)	-0.0255 (11)	-0.0105 (8)	0.5809 (7)	0.092 (6)
C(38)	-0.0989 (16)	-0.0718 (7)	0.5732 (7)	0.100 (7)
C(39)	-0.0382 (15)	-0.1303 (8)	0.5471 (9)	0.116 (8)
C(40)	-0.1022 (18)	-0.1922 (8)	0.5434 (9)	0.147 (10)
C(41)	-0.1815 (11)	0.0699 (7)	0.5546 (6)	0.084 (6)
C(42)	-0.1502 (14)	0.0785 (9)	0.4867 (7)	0.111 (8)

Table 2. Selected geometric parameters ( $\text{\AA}$ ,  $^\circ$ )

Pt—P	2.279 (3)	Pt—C(1)	2.066 (10)
Pt—C(7)	2.057 (10)	Pt—C(13)	2.038 (11)
P—C(19)	1.838 (14)	P—C(25)	1.828 (13)
P—C(31)	1.809 (15)	F(1)—C(2)	1.330 (15)
F(2)—C(3)	1.375 (17)	F(3)—C(4)	1.375 (15)
F(4)—C(5)	1.343 (18)	F(5)—C(6)	1.371 (15)
F(6)—C(8)	1.359 (14)	F(7)—C(9)	1.354 (16)
F(8)—C(10)	1.335 (14)	F(9)—C(11)	1.352 (15)
F(10)—C(12)	1.366 (14)	F(11)—C(14)	1.352 (20)
F(12)—C(15)	1.359 (25)	F(13)—C(16)	1.351 (20)
F(14)—C(17)	1.385 (22)	F(15)—C(18)	1.348 (18)
P—Pt—C(1)	91.6 (3)	P—Pt—C(7)	176.7 (3)
C(1)—Pt—C(7)	87.7 (4)	P—Pt—C(13)	94.6 (3)
C(1)—Pt—C(13)	173.6 (4)	C(7)—Pt—C(13)	86.2 (4)
Pt—P—C(19)	115.5 (4)	Pt—P—C(25)	117.5 (4)
C(19)—P—C(25)	103.2 (5)	Pt—P—C(31)	111.9 (5)
C(19)—P—C(31)	105.2 (6)	C(25)—P—C(31)	102.0 (6)
Pt—C(1)—C(2)	122.7 (9)	Pt—C(1)—C(6)	123.5 (9)
Pt—C(7)—C(8)	124.9 (8)	Pt—C(7)—C(12)	121.7 (8)
Pt—C(13)—C(14)	124.7 (11)	Pt—C(13)—C(18)	122.3 (10)

Two octants (+ + + and - - -) were measured. All methylene and phenyl H atoms were included in calculated positions; a common isotropic displacement parameter refined to  $0.11 (1) \text{\AA}^2$ . Only the H atoms of two methyl groups [C(40) and C(52)] could be located [ $U = 0.24 (7)$  and  $0.14 (4) \text{\AA}^2$ , respectively] by combining the information of the difference Fourier map and calculated positions. The correct enantiomorph was chosen on the basis of a lower weighted  $R$  factor ( $0.0411$  versus  $0.0749$ ). All calculations were performed using *SHELXTL-Plus* (Sheldrick, 1987).

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Lists of structure factors, anisotropic displacement parameters, H-atom coordinates and complete geometry have been deposited with the British Library Document Supply Centre as Supplementary Publication No. SUP 71566 (37 pp.). Copies may be obtained through The Technical Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England. [CIF reference: HU1049]

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