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*Acta Cryst.* (1997). **C53**, 301–302

### (Acetonitrile-*N*)( $\eta^5$ -cyclopentadienyl)bis(tri-phenylphosphine-*P*)ruthenium(II) Tetrafluoroborate

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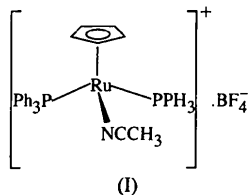
(Received 15 July 1996; accepted 4 November 1996)

#### Abstract

The title compound, [Ru(C<sub>5</sub>H<sub>5</sub>)(CH<sub>3</sub>CN){C(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>P<sub>2</sub>}]<sup>+</sup>BF<sub>4</sub><sup>-</sup>, crystallizes with C<sub>1</sub> local point group symmetry. The Ru—P distances are 2.343 (1) and 2.365 (1) Å, and the Ru—N—C angle is 169.8 (5)°.

#### Comment

While investigating the reactivity of [Ru(Cp){C(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>P<sub>2</sub>}(CH<sub>3</sub>CN)]<sup>+</sup> (Cp is cyclopentadienyl), we obtained single crystals of its tetrafluoroborate salt, (I). A search of the literature yielded few structurally characterized acetonitrile–Ru derivatives (Crocker, Green, Orpen & Thomas, 1984; Crocker *et al.*, 1987; Luginbühl *et al.*, 1991; McCormick, Cox & Gleason, 1993; Kirchner, Taube, Scot & Willett, 1993), which prompted us to carry out the present crystal structure determination.



The title Ru complex does not exhibit mirror symmetry. The angles around the Ru atom are P1—Ru—N1 89.8 (1), P2—Ru—N1 92.2 (1), P1—Ru—P2

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101.16 (3), Cp—Ru—P1 124.0, Cp—Ru—P2 120.4 and Cp—Ru—N1 121.3° (where Cp is the Cp centroid). The average distance from the Ru atom to the C atoms of the Cp ligand is 2.206 Å, while the Ru—Cp distance is 1.848 Å and the average C—C bond length in the Cp ligand is 1.416 Å. The Ru—N—C angle is 169.8 (5)°, showing a significant deviation from perfect linear coordination. The Ru—P distances are significantly different, albeit <sup>31</sup>P NMR shows one signal only at 42.48 p.p.m. (CD<sub>2</sub>Cl<sub>2</sub> solution). The overall slight dissymmetry of the complex is reminiscent of the situation found in [Ru(Cp){C(C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>C<sub>4</sub>}(CH<sub>3</sub>CN)]<sup>+</sup> (Crocker *et al.*, 1984), who ascribe the asymmetric coordination of the (C<sub>6</sub>H<sub>5</sub>)<sub>4</sub>C<sub>4</sub> ligand to the metal centre to the nature of the frontier orbitals of the Cp—Ru(MeCN)<sup>+</sup> fragment. Also noteworthy are the high displacement parameters of the acetonitrile group likening it to a weakly bound pendulum. This, in fact, is an ‘X-ray photograph’ of the leaving group character of the acetonitrile ligand.

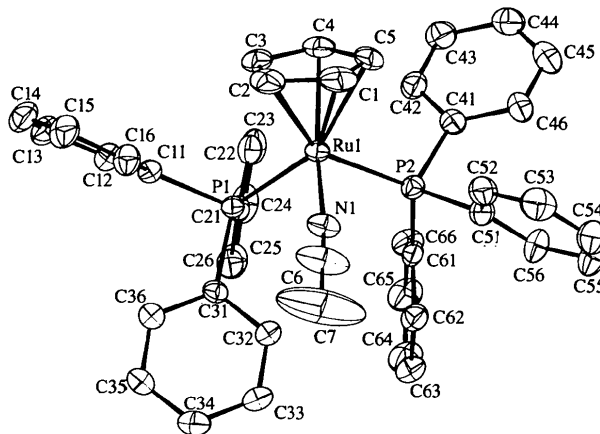


Fig. 1. The molecular structure of (I) showing 30% probability displacement ellipsoids. H atoms have been omitted for clarity.

#### Experimental

The synthesis of (I) was carried out by stirring stoichiometric amounts of [Ru(Cp){C(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>P<sub>2</sub>Cl}] (Bruce, Hamester, Swincer & Wallis, 1982) with AgBF<sub>4</sub> in acetonitrile at room temperature overnight. After filtering out AgCl, the solution was evaporated to dryness and the yellow solid recrystallized from CH<sub>2</sub>Cl<sub>2</sub>–heptane (1:5). Single crystals were obtained by slow evaporation of a degassed toluene–CH<sub>2</sub>Cl<sub>2</sub> (1:1) solution under an inert atmosphere over a period of 15 d.

#### Crystal data

[Ru(C<sub>5</sub>H<sub>5</sub>)(C<sub>2</sub>H<sub>3</sub>N)-  
(C<sub>18</sub>H<sub>15</sub>P<sub>2</sub>)<sub>2</sub>]BF<sub>4</sub>  
*M*<sub>r</sub> = 818.60  
Monoclinic  
P2<sub>1</sub>/c

Mo *K*α radiation  
λ = 0.71069 Å  
Cell parameters from 24  
reflections  
θ = 11–12°

$a = 10.320(4) \text{ \AA}$   
 $b = 17.062(8) \text{ \AA}$   
 $c = 21.803(1) \text{ \AA}$   
 $\beta = 90.28(2)^\circ$   
 $V = 3839(2) \text{ \AA}^3$   
 $Z = 4$   
 $D_x = 1.42 \text{ Mg m}^{-3}$   
 $D_m$  not measured

$\mu = 0.53 \text{ mm}^{-1}$   
 $T = 293 \text{ K}$   
 Parallelepiped  
 $0.2 \times 0.1 \times 0.1 \text{ mm}$   
 Yellow

#### Data collection

Enraf–Nonius CAD-4 diffractometer  
 $2\theta/\omega$  scans  
 Absorption correction:  $\psi$  scans (North, Phillips & Mathews, 1968)  
 $T_{\min} = 0.71$ ,  $T_{\max} = 0.95$   
 6454 measured reflections  
 6164 independent reflections

4722 reflections with  $I > 3\sigma(I)$   
 $R_{\text{int}} = 0.03$   
 $\theta_{\text{max}} = 25^\circ$   
 $h = -12 \rightarrow 12$   
 $k = 0 \rightarrow 20$   
 $l = -25 \rightarrow 0$   
 3 standard reflections  
 frequency: 60 min  
 intensity decay: 1.9%

#### Refinement

Refinement on  $F^2$   
 $R = 0.040$   
 $wR = 0.049$   
 $S = 1.065$   
 4722 reflections  
 470 parameters  
 H atoms placed geometrically after each cycle  
 Weighting scheme: Chebychev polynomial (Carruthers & Watkin, 1979)

$(\Delta/\sigma)_{\text{max}} = 0.318$   
 $\Delta\rho_{\text{max}} = 0.94 \text{ e \AA}^{-3}$   
 $\Delta\rho_{\text{min}} = -0.52 \text{ e \AA}^{-3}$   
 Extinction correction: Larson (1970)  
 Extinction coefficient: 13.749  
 Scattering factors from *International Tables for X-ray Crystallography* (Vol. IV)

Table 1. Selected geometric parameters ( $\text{\AA}$ )

Ru1—P1	2.3434 (9)	N1—C6	1.129 (6)
Ru1—P2	2.365 (1)	C1—C2	1.436 (7)
Ru1—N1	2.040 (3)	C1—C5	1.399 (7)
Ru1—C1	2.210 (4)	C2—C3	1.416 (7)
Ru1—C2	2.205 (4)	C3—C4	1.409 (7)
Ru1—C3	2.208 (4)	C4—C5	1.419 (7)
Ru1—C4	2.202 (4)	C6—C7	1.450 (9)
Ru1—C5	2.204 (4)		

All non-H atoms refined anisotropically.

Data collection: *CAD-4 Software* (Enraf–Nonius, 1989). Cell refinement: *CAD-4 Software*. Data reduction: *RC93* (Watkin, Prout & Lilley, 1994). Program(s) used to solve structure: *SHELXS86* (Sheldrick, 1985). Program(s) used to refine structure: *CRYSTALS* (Watkin, Prout, Carruthers & Betteridge, 1996). Molecular graphics: *CAMERON* (Watkin, Prout & Pearce, 1996). Software used to prepare material for publication: *CRYSTALS*.

AP thanks the CNRS and Laboratoire de Physique des Solides for allowing his stay in Mexico.

Lists of atomic coordinates, displacement parameters, structure factors and complete geometry have been deposited with the IUCr (Reference: FR1009). Copies may be obtained through The Managing Editor, International Union of Crystallography, 5 Abbey Square, Chester CH1 2HU, England.

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*Acta Cryst.* (1997). **C53**, 302–305

### Nonacarbonyl[ $\mu_3$ -(1,2-diphenylethyl)-phenylphosphinito]triosmium(2 Os—Os)

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(Received 10 April 1996; accepted 20 June 1996)

#### Abstract

The reaction of 1,2,3-triphenylphosphirene with [H<sub>2</sub>Os<sub>3</sub>-(CO)<sub>10</sub>] leads to the title compound, [Os<sub>3</sub>(CO)<sub>9</sub>{(C<sub>6</sub>H<sub>5</sub>)<sub>3</sub>-C<sub>2</sub>PO}], which displays an open Os<sub>3</sub>(CO)<sub>9</sub> cluster [Os—Os 2.703 (1) and 2.867 (2) Å, and Os—Os 124.24 (4)°]. This moiety is  $\mu_3, \eta^3$ -bonded to a phosphirene ligand with a cleaved P—C bond, PhC—C(Ph)—P. An O atom was incorporated into the molecule resulting in an unusual five-membered metallacycle,