trans-(Acetonitrile)bis[1,2-bis(diphenylphosphino)ethane-κ^2P,P'](phenylethynyl)ruthenium(II) hexafluorophosphate ethanol hemisolvate

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Key indicators

Single-crystal X-ray study

T = 200 K

Mean σ(C–C) = 0.007 Å

H-atom completeness 95%

Disorder in solvent or counterion

R factor = 0.051

wR factor = 0.054

Data-to-parameter ratio = 12.8

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

The title compound, [Ru(C$_8$H$_5$)(C$_2$H$_3$N)(C$_{26}$H$_{24}$P$_2$)$_2$]PF$_6$·0.5C$_2$H$_5$OH, comprises a pseudo-octahedral ruthenium complex cation with two bidentate diphosphine ligands and *trans*-disposed acetonitrile and phenylethynyl ligands, together with a disordered hexafluorophosphate anion and a disordered ethanol solvent molecule.

Comment

The title complex, (I), is the first (alkynyl)bis(bidentate diphosphine)(nitrile)ruthenium complex, although both (chloro)(nitrile)- and (alkynyl)(chloro)-analogues exist. The former are obtained with *cis* stereochemistry (Basallote *et al.*, 1998; Winter & Scheiring, 2000), whereas the latter are isolated with *trans*-disposed ligands (McDonagh *et al.*, 1996; Naulty *et al.*, 1998).

(I)
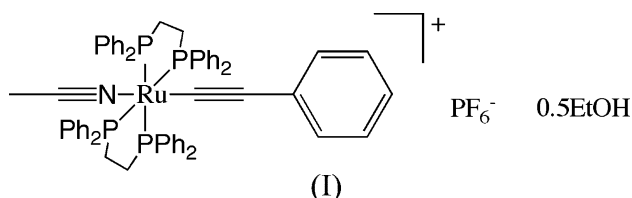
For the most part, the bond distances and angles of (I) are unremarkable and similar to those of the above-mentioned related complexes. Deviations from octahedral geometry at Ru result from the presence of two chelating 1,2-bis-(diphenylphosphino)ethane ligands [P1—Ru1—P2 = 81.46 (3)° and P3—Ru1—P4 = 81.56 (3)°].

Experimental

A solution of *trans*-[Ru(C$_2$Ph)Cl(dppe)$_2$] (200 mg, 0.193 mmol) and sodium hexafluorophosphate (65 mg, 0.40 mmol) in acetonitrile (15 ml) was refluxed for 30 min and then allowed to cool. The solution was filtered and taken to dryness under reduced pressure. The residue was then extracted with CH$_2$Cl$_2$ and taken to dryness to give a very-light yellow solid (228 mg, 99%). Analysis, calculated for C$_{62}$H$_{56}$F$_6$NP$_5$Ru: C 62.84, H 4.76, N 1.18%; found: C 62.89, H 4.67, N 1.15%. IR (CH$_2$Cl$_2$): (νNC) 1967 cm^{-1}, (νCC) 2086 cm^{-1}; FAB MS, m/z (fragment, relative intensity): 1040 ([M]$^+$, 10), 999 ([M − NC$_2$H$_3$]$^+$, 45), 878 ([Ru(dppe)$_2$]$^+$, 80). Yellow crystals suitable for structural study were obtained by liquid diffusion of successively ethanol, methanol and then acetonitrile into a dichloromethane solution.

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Crystal data

$[Ru(C_8H_5)(C_2H_3N)(C_{26}H_{24}P_2)_2]$-
$F_6P \cdot 0.5C_2H_6O$
$M_r = 1208.9$
Monoclinic, $P2_1/n$
$a = 18.4312 (2)$ Å
$b = 17.0281 (2)$ Å
$c = 18.5444 (2)$ Å
$\beta = 101.1934 (4)°$

$V = 5709.42 (11)$ Å^3
$Z = 4$
$D_x = 1.405$ Mg m^{-3}
Mo $K\alpha$ radiation
$\mu = 0.48$ mm^{-1}
$T = 200$ K
Plate, yellow
$0.24 \times 0.20 \times 0.13$ mm

Data collection

Enraf–Nonius KappaCCD area-
detector diffractometer
φ and ω scans
Absorption correction: integration
via Gaussian method (Coppens,
1970) implemented in *MAXUS*
(MacKay *et al.*, 2000)
$T_{min} = 0.907$, $T_{max} = 0.958$

110542 measured reflections
13121 independent reflections
8703 reflections with $I > 3\sigma(I)$
$R_{int} = 0.04$
$\theta_{max} = 27.5°$

Refinement

Refinement on F
$R[F^2 > 3\sigma(F^2)] = 0.051$
$wR[F^2 > 3\sigma(F^2)] = 0.055$
$S = 1.00$
8703 reflections
681 parameters
H-atom parameters constrained

Chebychev polynomial with 3 para-
meters (Carruthers & Watkin,
1979), 1.46 0.729 1.01
$(\Delta/\sigma)_{max} = 0.018$
$\Delta\rho_{max} = 1.20$ e Å^{-3}
$\Delta\rho_{min} = -2.27$ e Å^{-3}
Extinction correction: Larson
(1970), eq. 22
Extinction coefficient: 6.4 (7) $\times 10^2$

Table 1
Selected geometric parameters (Å, °).

Ru1—P1	2.3407 (8)	Ru1—P4	2.3980 (8)
Ru1—P2	2.3724 (8)	Ru1—N1	2.072 (3)
Ru1—P3	2.3894 (8)	Ru1—C1	2.022 (3)
P1—Ru1—P2	81.46 (3)	P4—Ru1—N1	89.54 (8)
P1—Ru1—P3	97.30 (3)	P1—Ru1—C1	82.91 (9)
P2—Ru1—P3	176.24 (3)	P2—Ru1—C1	83.29 (8)
P1—Ru1—P4	178.35 (3)	P3—Ru1—C1	100.11 (8)
P2—Ru1—P4	99.75 (3)	P4—Ru1—C1	96.11 (9)
P3—Ru1—P4	81.56 (3)	N1—Ru1—C1	172.93 (11)
P1—Ru1—N1	91.54 (8)	Ru1—N1—C13	174.5 (3)
P2—Ru1—N1	91.58 (7)	Ru1—C1—C2	171.3 (3)
P3—Ru1—N1	84.89 (7)		

The crystallographic asymmetric unit of (I) consists of one $[Ru(C_8H_5)(C_2H_3N)(C_{26}H_{24}P_2)_2]^+$ cation, one PF_6^- anion and a solvent molecule disordered across a crystallographic inversion centre. Within the cation, the alkynyl ligand vibrates largely as a whole, so the atoms furthest removed from the metal (C5–C7) have large anisotropy of their displacement parameters. The anion appears to be disordered over two locations. Each atom was split over two sites and refined with isotropic displacement parameters, which were constrained to be equal for each pair; the refined site occupancy factors are 0.539 (5) and 0.461 (5). Restraints were imposed on bond distances and angles for each component of the anion. Several peaks were observed in a subsequent electron-density difference map near the crystallographic inversion centre at ($\frac{1}{2}$, $\frac{1}{2}$, $\frac{1}{2}$), indicating the presence of solvent. Various solvents had been used during preparation and recrystallization, but the best match appears to be with disordered ethanol; the O atom was modelled as disordered over two sites, each with site occupancy factor 0.25.
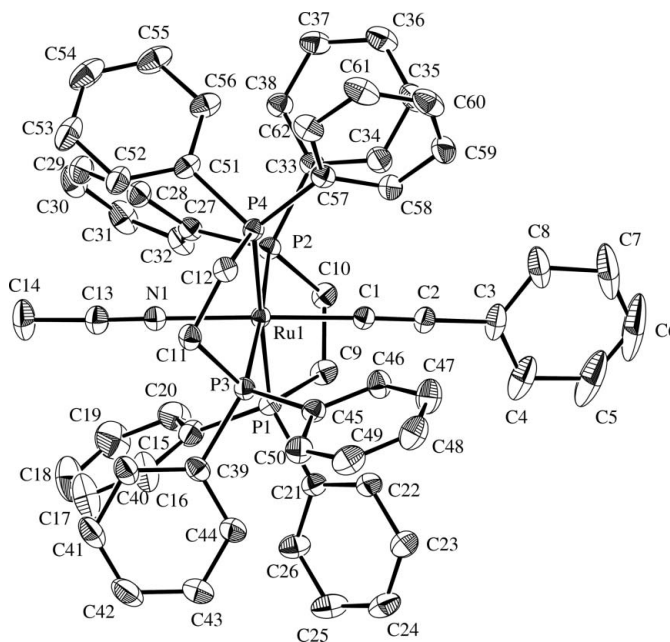

Figure 1
A view of the cation of (I), with the atom-numbering scheme. Displacement ellipsoids are drawn at the 30% probability level. H atoms have been omitted.

The H atoms of the cation were included in idealized positions and treated as riding on the atoms to which they are bonded, with C—H = 1.0 Å and $U_{iso}(H) = 1.2U_{eq}(C)$. The methyl group was oriented to give the best fit to peaks in a difference electron-density map. H atoms of the solvent molecule were not included.

The largest peaks in the final difference electron-density map were located close to the atoms of the disordered anion. The highest peak is located 1.28 Å from atom F61 and the deepest hole 0.54 Å from P50.

Data collection: *COLLECT* (Enraf–Nonius, 1997); cell refine-ment: *DENZO/SCALEPACK*; data reduction: *DENZO/SCALE-PACK* (Otwinowski & Minor, 1997); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *CRYSTALS* (Betteridge *et al.*, 2003); molecular graphics: *ORTEPII* (Johnson, 1976) in *TEXSAN* (Molecular Structure Corporation, 1997); software used to prepare material for publica-tion: *CRYSTALS*.

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