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Crystal structure of *trans*-tetrakis(acetonitrile- κ N)bis(triphenylphosphine- κ P)ruthenium(II) dihexafluorophosphate - dichloroform (1/2), $C_{46}H_{44}Cl_6F_{12}N_4P_4Ru$

Table 1: Data collection and handling.

Crystal:	Colourless block
Size:	0.19 × 0.08 × 0.03 mm
Wavelength:	Cu $K\alpha$ radiation (1.54178 Å)
μ :	6.86 mm ⁻¹
Diffractometer, scan mode:	Bruker APEX-II CCD, φ and ω
$\theta_{\max}$, completeness:	63.7°, 88%
$N(hkl)_{\text{measured}}$, $N(hkl)_{\text{unique}}$, R_{int} :	22410, 4348, 0.119
Criterion for I_{obs} , $N(hkl)_{\text{gt}}$:	$I_{\text{obs}} > 2 \sigma(I_{\text{obs}})$, 2515
$N(\text{param})_{\text{refined}}$:	369
Programs:	Bruker [1], SHELX [2, 3], Olex2 [4]

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Abstract

$C_{46}H_{44}Cl_6F_{12}N_4P_4Ru$, orthorhombic, *Pbca* (no. 61), $a = 14.319(2)$ Å, $b = 16.7358(5)$ Å, $c = 22.6972(16)$ Å, $Z = 4$, $V = 5439.1(9)$ Å³, $R_{\text{gt}}(F) = 0.0727$, $wR_{\text{ref}}(F^2) = 0.1673$, $T = 235.38$ K.

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The crystal structure is shown in the figure. Tables 1 and 2 contain details on crystal structure and measurement conditions and a list of the atoms including atomic coordinates and displacement parameters.

Source of material

Na_2CO_3 (0.0053 g, 0.05 mmol) in methanol (10 mL) was added to a solution of 6-mercaptopyridine (0.0155 g, 0.1 mmol), followed by addition of $Ru(PPh_3)_3Cl_2$ (0.0958 g, 0.1 mmol) in acetonitrile (10 mL). The mixture was stirred and refluxed for 6 h to give a yellow solution, which was concentrated to 3 mL under reduced pressure. A solution of NH_4PF_6

(0.0300 g, 0.18 mmol) in distilled water (10 mL) was added, and stirring continued for 0.5 h at room temperature. The resultant suspension was filtered to obtain a yellow precipitate, which was washed with distilled water, diethyl ether. Diffraction-quality crystals were obtained by slow evaporation of a solution in $CHCl_3$ layered with hexane at room temperature.

Experimental details

All hydrogen atoms were placed in idealized positions and refined in riding models. The U_{iso} values of the hydrogen atoms of methyl groups were set to $1.5U_{\text{eq}}(C)$, whereas the U_{iso} values of all other hydrogen atoms were set to $1.2U_{\text{eq}}(C)$. The PF_6^- anion shows a positional disorder.

Comment

Ruthenium complexes have been attracted a lot of interest because of their catalytic, biological and pharmaceutical properties [5–7]. Ruthenium complexes containing CH_3CN ligands have been considered as good precursors to synthesize a large amount of Ru complexes with other donor ligands [8–10].

The asymmetric unit of the crystal structure (cf. the figure) is comprised of one half cationic $[Ru(PPh_3)_2(CH_3CN)_4]^{2+}$, one PF_6^- anion and one $CHCl_3$ molecule. The Ru atom is six-coordinated by two P atoms from PPh_3 ligands and four N atoms from acetonitrile. Two Ru–P bond lengths are 2.397 Å and the Ru–N bond lengths are 2.006 Å. The angles of P–Ru–N are near 90 and 180°. Respectively, all geometric parameters are in the typical ranges [8, 11–13].

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Table 2: Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²).

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
Ru1	0.5000	0.5000	0.5000	0.0219(2)
P1	0.39018(13)	0.51697(13)	0.57874(9)	0.0255(5)
P2	0.24915(17)	0.73262(15)	0.39451(13)	0.0443(6)
Cl1	0.4267(3)	0.8810(2)	0.2932(2)	0.1148(15)
Cl2	0.6086(2)	0.8982(3)	0.25043(17)	0.1116(15)
Cl3	0.5454(3)	0.9976(3)	0.34567(19)	0.1347(17)
N1	0.4882(4)	0.3820(4)	0.5143(3)	0.0262(15)
N2	0.3949(4)	0.4900(4)	0.4420(3)	0.0260(15)
C1	0.2951(6)	0.6442(6)	0.6323(4)	0.042(2)
H1	0.2412	0.6124	0.6291	0.050*
C2	0.2890(7)	0.7180(6)	0.6584(5)	0.051(3)
H2	0.2316	0.7364	0.6732	0.062*
C3	0.3675(7)	0.7648(6)	0.6627(5)	0.053(3)
H3	0.3636	0.8155	0.6804	0.064*
C4	0.4516(6)	0.7385(5)	0.6413(4)	0.038(2)
H4	0.5050	0.7710	0.6438	0.046*
C5	0.4567(6)	0.6638(5)	0.6161(4)	0.036(2)
H5	0.5145	0.6450	0.6021	0.043*
C6	0.3785(5)	0.6159(5)	0.6109(4)	0.0297(18)
C7	0.4023(5)	0.4523(5)	0.6435(3)	0.0274(17)
C8	0.3308(6)	0.4495(6)	0.6847(4)	0.035(2)
H8	0.2789	0.4833	0.6803	0.042*
C9	0.3346(7)	0.3986(6)	0.7314(4)	0.044(2)
H9	0.2846	0.3968	0.7582	0.053*
C10	0.4110(7)	0.3498(6)	0.7398(4)	0.050(2)
H10	0.4134	0.3147	0.7722	0.060*
C11	0.4833(7)	0.3533(5)	0.7003(4)	0.045(2)
H11	0.5359	0.3205	0.7057	0.054*
C12	0.4796(6)	0.4043(5)	0.6528(4)	0.0337(19)
H12	0.5301	0.4066	0.6264	0.040*
C13	0.2746(5)	0.4898(5)	0.5523(3)	0.0268(17)
C14	0.2430(6)	0.4131(5)	0.5577(4)	0.0331(19)
H14	0.2766	0.3757	0.5801	0.040*
C15	0.1608(6)	0.3910(6)	0.5296(4)	0.044(2)
H15	0.1401	0.3378	0.5316	0.052*
C16	0.1098(6)	0.4468(5)	0.4991(4)	0.042(2)
H16	0.0533	0.4322	0.4810	0.050*
C17	0.1417(5)	0.5241(5)	0.4950(4)	0.039(2)
H17	0.1070	0.5622	0.4738	0.047*
C18	0.2240(5)	0.5458(5)	0.5216(4)	0.033(2)
H18	0.2456	0.5987	0.5189	0.039*
C19	0.4761(5)	0.3162(6)	0.5233(4)	0.034(2)
C20	0.4595(7)	0.2325(6)	0.5368(6)	0.064(3)
H20A	0.4533	0.2025	0.5004	0.096*
H20B	0.4026	0.2274	0.5597	0.096*
H20C	0.5117	0.2115	0.5593	0.096*
C21	0.2573(6)	0.4781(6)	0.3690(4)	0.044(2)
H21A	0.2809	0.4745	0.3290	0.066*
H21B	0.2178	0.5250	0.3727	0.066*
H21C	0.2212	0.4307	0.3781	0.066*
C22	0.3346(5)	0.4844(5)	0.4095(3)	0.0253(18)
C23	0.5134(8)	0.9474(7)	0.2811(5)	0.069(3)
H23	0.4909	0.9874	0.2523	0.083*
F1 ^a	0.2285(13)	0.7346(11)	0.4629(7)	0.098(4)
F2 ^a	0.2918(11)	0.6996(10)	0.4544(7)	0.086(4)

Table 2 (continued)

Atom	x	y	z	<i>U</i> _{iso} [*] / <i>U</i> _{eq}
F3	0.1673(4)	0.6699(4)	0.3915(4)	0.095(2)
F4 ^a	0.3189(9)	0.6624(8)	0.4064(8)	0.073(3)
F5 ^a	0.3085(10)	0.6715(10)	0.3590(9)	0.086(4)
F6 ^a	0.2708(17)	0.7287(14)	0.3270(7)	0.123(6)
F7 ^a	0.2099(12)	0.7680(11)	0.3365(7)	0.077(4)
F8 ^a	0.1775(10)	0.8012(9)	0.3846(13)	0.113(6)
F9 ^a	0.1928(11)	0.7948(11)	0.4306(8)	0.078(4)
F10	0.3312(4)	0.7945(4)	0.3976(3)	0.0721(18)

^aOccupancy: 0.5.

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