## Experimental section

Tetrahydrofuran-$^2H_8$ was thoroughly degassed during four freeze, thaw and degass cycles and vacuum transferred to an ampoule containing freshly prepared sodium-potassium alloy. All NMR samples were prepared in either flame-sealed (*ca.* $10^{-6}$ Torr) or Teflon® stoppered NMR tubes. $^1H$ (300.13 or 500.13 MHz), $^{13}C$ (125.70 MHz) and $^{31}P$ (201.46 MHz) NMR spectra were recorded on Bruker or Varian instruments. Chemical shifts ($\delta$) for $^1H$ NMR spectra are reported relative to residual protons in the deuterated solvents and for $^{13}C$ NMR spectra relative to the carbons in the deuterated solvent. $^{31}P$ NMR spectra are reported relative to 85% phosphoric acid ($\delta$ = 0.0 ppm) as the external standard.

**Preparation of $[RuH(\eta^2\text{-}H_2)(P^iPr_3)_2\{C_6H_5py\}]^+[BAr^F]^-$ (2).** A Fischer-Porter bottle was loaded with the solids $[RuH(H_2)(PiPr_3)_2(C_6H_4py)]$ (0.46 g, 0.79 mmol) and HBarf (0.79 g, 0.79 mmol) and cooled to –78°C. After *ca.* 10 mins THF (*ca.* 15 mL) was added in a single aquilot yielding a pale orange solution, the solids remaining essentially insoluble at this temperature. The bottle was then immersed in liquid nitrogen and the heterogenous mixture thoroughly degassed during three freeze and thaw cycles. The bottle was then pressurized with $H_2$ (3 bar) at –78°C and allowed to warm to room temperature over a period of *ca.* 45 mins. After this period all the starting materials had been consumed to afford an orange solution; no effervescence was observed during the reaction even at room temperature. Subsequently, the solution was consecutively decanted, filtered and concentrated *in vacuo* and the resulting deep red solution was repressurized with $H_2$ (3 bar). Orange crystals of **2**, suitable for X-ray diffraction analysis, were obtained at room temperature under $H_2$ (0.45 g, 70 %). The compound is best stored under an atmosphere of $H_2$ at –20 °C ; it rapidly decomposes in the solid state *in vacuo*. A pure sample of **2** for spectroscopic measurements was prepared by loading an NMR tube with an equimolar ratio of $[RuH(H_2)(PiPr_3)_2(C_6H_4py)]$ (20 mg scale) and HBarf.$(Et_2O)_2$. $^2H_8$-thf was then condensed onto the solids and the tube

sealed under dihydrogen (800 mbar). $^1$H NMR at 500.13 MHz in $^2H_8$-thf at 293K: -13.73 (b, 3H, Ru*H2*), 6.18 (br, 2H, *H1b* and *H7*), 7.45 (t, 1H, $^3J_{H-H}$ 6.4 Hz, *H2*), 7.71 (br t, 1H, $^3J_{H-H}$ 8.3 Hz, *H9*), 7.95 (br t, 2H, $^3J_{H-H}$ 6.8 Hz, *H8* and *H10*), 8.03 (t, 1H, $^3J_{H-H}$ 7.7 Hz, *H3*), 8.21 (d, 1H, $^3J_{H-H}$ 7.9 Hz, *H4*), 9.11 (d, 1H, $^3J_{H-H}$ 4.9 Hz, *H1*), 0.97 (dd, 18H, P(CH*Me*$_2$)$_3$), 1.04 (dd, 18H, P(CH*Me*$_2$)$_3$), 1.81 (br, 6H, P(C*H*Me$_2$)$_3$), 7.56 (br, 4H, barf anion, $H_{para}$), 7.78 (br, 8H, barf anion, $H_{ortho}$). $^1$H NMR at 500.13 MHz in $^2H_8$-thf at 173K: -14.12 (t, 1H, $^2J_{P-H}$ 17.8 Hz, Ru*H1a*), -13.02 (br, 2H, Ru($\eta^2$-*H*$_2$)), 4.14 (br, 1H, *H1b*), 7.54 (t, 1H, $^3J_{H-H}$ 6.1 Hz, *H2*), 7.79 (t, 1H, $^3J_{H-H}$ 7.6 Hz, *H9*), 7.87 (t, 1H, $^3J_{H-H}$ 7.6 Hz, *H8*), 8.19 (t, 1H, $^3J_{H-H}$ 7.8 Hz, *H3*), 8.22 (d, 1H, $^3J_{H-H}$ 8.1 Hz, *H7*), 8.30 (t, 1H, $^3J_{H-H}$ 6.5 Hz, *H10*), 8.38 (d, 1H, $^3J_{H-H}$ 8.1 Hz, *H4*), 9.20 (d, 1H, $^3J_{H-H}$ 4.6 Hz, *H1*), 0.96 (br, 18H, P(CH*Me*$_2$)$_3$), 1.02 (br, 18H, P(CH*Me*$_2$)$_3$), 1.78 (v br, 6H, P(C*H*Me$_2$)$_3$), 7.54 (t, 1H, $^3J_{H-H}$ 6.1 Hz, $H_2$), 7.82 (br, 4H, barf anion, $H_{para}$), 7.97 (br, 8H, barf anion, $H_{ortho}$), 9.20 (d, 1H, $^3J_{H-H}$ 4.6 Hz, $H_1$). $^{31}$P{$^1$H} NMR at 201.46 MHz in $^2H_8$-thf at 293K: 52.01 (s). $^{13}$C{$^1$H} NMR at 125.70 MHz in $^2H_8$-thf at 293K: 120.2 (v br, *C7* and *C11*), 125.5 (s, *C2*), 125.5 (s, *C4*), 132.6 (s, *C9*), 136.5 (br, *C8* and *C10*), 139.1 (s, *C3*), 144.1 (s, *C6*), 157.2 (s, *C1*), 161.4 (s, *C5*), 19.5 (s, P(CH*Me*$_2$)$_3$), 20.3 (s, P(CH*Me*$_2$)$_3$), 26.8 (m, P(*C*HMe$_2$)$_3$), 118.2 (septett, $^3J_{C-F}$ 3.9 Hz, barf anion, *p*-C), 125.5 (q, $^1J_{C-F}$ 272.4 Hz, barf anion, *C*F$_3$), 130.0 (qq, $^3J_{B-C}$ 2.8 Hz, $^2J_{C-F}$ 31.6 Hz, *m-C*), 135.6 (s, barf anion, *o-C*), 162.8 (q, $^1J_{B-C}$ 49.9 Hz, barf anion, *i-C*). $T_1$ (min) of 16 ms at 243 K at 300.13 MHz. Agostic proton $^1J_{C-H}$ = 128 Hz (doublet) resolved by 1D-gs-HMQC spectroscopy at 183 K.

**[Ru($\eta^2$-H$_2$)(thf)(P$^i$Pr$_3$)$_2$(C$_6$H$_4$py)]$^+$[BArf]$^-$ (3)**

$^1$H NMR at 500.13 MHz in $^2H_8$-THF at 293K: -7.53 (br, 2H, Ru($\eta^2$-*H*$_2$)), 6.80 (dt, 1H, $^3J_{H-H}$ 7.3 Hz, $^4J_{H-H}$ 1.6 Hz, *H9*), 6.88 (t, 1H, $^3J_{H-H}$ 7.3 Hz, *H8*), 7.32 (d, 1H, $^3J_{H-H}$ 7.8 Hz, *H10*), 7.45 (dt, 1H, $^3J_{H-H}$ 6.5 Hz, $^4J_{H-H}$ 1.2 Hz, *H2*), 7.75 (dd, 1H, $^3J_{H-H}$ 7.8 Hz, $^4J_{H-H}$ 1.7 Hz, *H7*), 8.03 (t, 1H, $^3J_{H-H}$ 8.2 Hz, *H3*), 8.27 (d, 1H, $^3J_{H-H}$ 8.2 Hz, *H4*), 9.03 (br, 1H, $^3J_{H-H}$ not resolved, *H1*), 0.89 (q, 18H, P(CH*Me*$_2$)$_3$), 0.94 (q, 18H, P(CH*Me*$_2$)$_3$), 2.11 (m, 6H, P(C*H*Me$_2$)$_3$), 7.56 (br, 4H, barf anion, $H_{para}$), 7.78 (br, 8H, barf anion, $H_{ortho}$). $^{31}$P{$^1$H} NMR at 201.46 MHz in $^2H_8$-thf at 293K: 37.84

(s). $^{13}C\{^{1}H\}$ NMR at 125.70 MHz in $^{2}H_{8}$-thf at 293K: 121.3 (s, *C4*), 122.1 (s, *C8*), 123.3 (s, *C2*), 125.6 (s, *C7*), 129.9 (s, *C9*), 138.9 (s, *C3*), 142.4 (s, *C6*), 143.1 (s, *C10*), 158.0 (s, *C1*), 165.0 (s, *C5*), 169.0 (br, *C11*), 19.4 (s, $P(CH\mathit{Me}_2)_3$), 19.6 (s, $P(CH\mathit{Me}_2)_3$), 25.6 (m, $P(\mathit{C}HMe_2)_3$), 118.2 (septett, $^{3}J_{C\text{-}F}$ 3.9 Hz, barf anion, *p*-C), 125.5 (q, $^{1}J_{C\text{-}F}$ 272.4 Hz, barf anion, $\mathit{C}F_3$), 130.1 (qq, $^{3}J_{B\text{-}C}$ 2.8 Hz, $^{2}J_{C\text{-}F}$ 31.6 Hz, *m-C*), 135.6 (s, barf anion, *o-C*), 162.8 (q, $^{1}J_{B\text{-}C}$ 49.9 Hz, barf anion, *i-C*). $T_{1\,min}$ 11 ms at 263 K at 300 MHz. $^{1}J_{H\text{-}D} = 27$ Hz.

**Computational details :**

All the calculations were performed with the Gaussian 98 set of programs[1] at the B3PW91 level of theory.[2] Ruthenium was represented with the Hay-Wadt relativistic core potential (ECP) for the 28 innermost electrons and its associated double-ζ basis set[3a] augmented by a polarization f function with an exponent of 1.235. Phosphorus atoms were also represented with Los Alamos ECPs and the associated double-ζ basis set[3b] augmented by a polarization d function with an exponent of 0.387. A 6-31G(d,p) basis set was used for the atoms directly attached to the metal (C, N and all the hydrides) and a 6-31G basis set was used for the remaining atoms.[4] Full geometry optimizations without any symmetry constraint have been carried out and the nature of the located extrema (minima or transition states) was checked through analytical computations of the vibrationnal frequencies.

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2) a) Becke, A. D. *J. Chem. Phys.* **1993**, *98*, 5648. b) Perdew, J. P, Wang, Y. *Phys. Rev. B* **1992**, *45*, 13244.

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**X-ray analysis of Complex 2 :**

Crystal data $C_{65}H_{74}BF_{24}NOPRu$, Formula weight: 1515.07, Crystal size (0.5x0.5x0.4)$mm^3$ Crystal system triclinic, space group, P $2_1/n$

Unit cell dimensions a = 15.232(3)Å, b = 25.028(5)Å, c = 19.142(4)Å,

$\alpha = 90.26(3)°$, $\beta = 110.70(2)°$, $\gamma = 90.05(3)°$, Volume: 6826(2)$Å^3$, Z = 4,

$\rho_{calcd}$ = 1.474$mg/m^3$, $\mu$ = 0.384$mm^{-1}$, F(000) = 3096. Data collection was collected at low temperature (T = 140(2)K) on a Stoe Imaging Plate Diffraction System (IPDS), equipped with an Oxford Cryosystems Cryostream Cooler device and using a graphite-monochromated Mo-K$\alpha$ radiation

($\lambda$ = 0.71073Å), the crystal-to-detector distance was chosen to be 70mm, a final unit cell parameters were obtained by least-squares refinement of a set of 5000 reflections, and a crystal decay was monitored by measuring 200 reflections per image. Any fluctuations of the intensity were observed over the course of the data collection. Numerical corrections absorption [1] were applied on the data. The structure was solved by Direct Methods using SIR92[2], and refined by least-squares procedures on a $F^2$ with the aid of (SHELXL97), [3] by minimizing the function : $\Sigma w(Fo^2-Fc^2)^2$, where Fo and Fc are respectively the observed and calculated structure factors. The Atomic Scattering Factors were taken from International tables for X-Ray crystallography[4].

Largest electron density residue are : ($\Delta\rho_{max}$ = 0.358, $\Delta\rho_{min}$ = -0.257)e.$Å^{-3}$, model reached convergence with R(F) = 0.0357 and wR2 = 0.0916 for [I>2$\sigma$(I)]. All hydrogen atoms were located on a difference Fourier maps, and refined with a idealized model with a fixed isotropic thermal parameter. Concerning hydrure atom H1a, the hydrogen agostic H1b and hydrogen atoms labelled H1c and H2c of ligand dihydrogen, these atoms have been isotropically refined. All non-hydrogens atom were anisotropically refined, and in the last cycles of refinement a weighting scheme was used. Drawing of molecule is performed with the aid of program ZORTEP [5] with 50% probability displacement ellipsoids for non-hydrogen atoms.

Further details on the Crystal Structure Investigation are available on request from the Director of the Cambridge Crystallographic Data centre, 12 Union Road, GB-Cambridge UK, on quoting the full journal citation.

**References, X-ray section :**

1) X-SHAPE (revision 1.01) July 1996. *A Crystal Optimisation For numerical Correction* STOE and Cie. (X-SHAPE is based on the Program "HABITUS" by Dr Wolfang Herrendorf, Institüt für Anorganische Chemie, Universitât GIESSEN).

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Burla, M.C., Polidori, G. and Camalli, M. (1994). *SIR92 program for automatic solution of crystal structures by direct methods*. J. Appl. Cryst. 27, 435.

3) Sheldrick, G. M; (1997). SHELXL97. *Program for the refinement of Crystal Structures*. University of Göttingen, Germany.

4) INTERNATIONAL tables for X-Ray crystallography, 1974, Vol IV, Kynoch Press, Birmingham, England.

5) L.Zolnaï, 1998, ZORTEP, *Graphical Program for X-Ray Structures Analysis* University of Heilderberg, Germany.

## X-Ray Tables

**Table S1 .Crystal data, data collection and structure refinement**

| | |
|---|---|
| Empirical formula | $C_{65}H_{74}BF_{24}NOPRu$ |
| Formula weight | 1515.07 |
| Temperature | 140(2)K |
| Wavelength | 0.71073Å |
| Crystal system, space group | monoclinic, P $2_1/n$ |
| Unit cell dimensions | a = 15.232(3)Å $\alpha$ = 90.26(3)° |
| | b = 25.028(5)Å $\beta$ = 110.70(2)° |
| | c = 19.142(4)Å $\gamma$ = 90.05(3)° |
| Volume | 6826(2)$Å^3$ |
| Z, Calculated density | 4, 1.474$mg/m^3$ |
| Absorption coefficient | 0.384$mm^{-1}$ |
| F(000) | 3096 |
| Crystal size | (0.5x0.5x0.4)mm |
| Crystal form | Parallelepiped |
| Crystal color | Dark brown |
| Tube power | 1.50kW |
| Tube voltage | 50kV |
| Tube current | 30mA |
| Collimator size | 0.8mm |
| Detector distance | 70.0mm |
| 2theta range | (3.3 - 52.1)° |
| d(hkl) range | (12.453 - 0.809)Å |
| Phi movement mode | Rotation |
| Phi start | 0.0° |
| Phi end | 200.4° |
| Phi incr. | 0.6° |
| Number of exposures | 192 |
| Irradiation / exposure | 3.00min |
| Measurement duration | 40h |

| | |
|---|---|
| Reflections collected / unique | 39875 / 9389 [R(int) = 0.0320]Completeness to 2theta = 23.26 95.6%Refinement method |
| Full-matrix least-squares on $F^2$ | |
| Data / restraints / parameters | 9389 / 595 / 1024 |
| Absoption corrections | Numerical* |
| Tmin - Tmax | 0.68 - 1.0Goodness-of-fit on $F^2$ 1.032 |
| Scheme of ponderation where $P = (F_o^2+2F_c^2)/3$ | Weight = $1/[\sigma^2(Fo^2)+(0.0538P)^2+7.60P]$ |
| Final R indices [$I>2\sigma(I)$] | R1 = 0.0357, wR2 = 0.0916 |
| R indices (all data) | R1 = 0.0403, wR2 = 0.0950 |
| Largest diff. peak and hole | (0.849 and -0.563)e.$Å^{-3}$ |

* X-SHAPE Crystal Optimisation for Numerical Absorption Correction Revision 1.01 July 1996 STOE and Cie GMBH 1996.
*(X-SHAPE is based on the program 'HABITUS' by Dr.W.Herrendorf, Institut für Anorganische Chemie, Universität Giessen)*

**Table S2. Atomic coordinates ($x10^4$) and equivalent isotropic displacement parameters ($Å^2x10^3$) for , U(eq) is defined as one third of the trace of the orthogonalized Uij tensor**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| Ru | -2721(1) | 9871(1) | 7702(1) | 19(1) |
| P(1) | -3157(1) | 10651(1) | 8224(1) | 22(1) |
| P(2) | -1969(1) | 9079(1) | 7534(1) | 23(1) |
| N | -4078(2) | 9536(1) | 7265(1) | 28(1) |
| C(1) | -4365(2) | 9153(1) | 7632(2) | 38(1) |
| C(2) | -5242(2) | 8921(1) | 7346(3) | 57(1) |
| C(3) | -5841(2) | 9085(2) | 6662(3) | 65(1) |
| C(4) | -5570(2) | 9478(2) | 6284(2) | 53(1) |
| C(5) | -4680(2) | 9707(1) | 6592(2) | 35(1) |
| C(6) | -4356(2) | 10140(1) | 6230(2) | 35(1) |
| C(7) | -4982(3) | 10505(2) | 5746(2) | 51(1) |
| C(8) | -4647(3) | 10922(2) | 5443(2) | 63(1) |
| C(9) | -3714(3) | 10990(2) | 5591(2) | 59(1) |
| C(10) | -3077(3) | 10638(1) | 6060(2) | 43(1) |
| C(11) | -3404(2) | 10223(1) | 6384(2) | 32(1) |
| C(12) | -2176(2) | 10853(1) | 9088(2) | 41(1) |
| C(13) | -4204(2) | 10640(1) | 8495(2) | 33(1) |
| C(14) | -3367(2) | 11254(1) | 7612(2) | 32(1) |
| C(15) | -2743(2) | 8565(1) | 6908(2) | 30(1) |
| C(16) | -957(2) | 9208(1) | 7227(2) | 31(1) |
| C(17) | -1403(2) | 8703(1) | 8414(2) | 36(1) |
| C(121) | -1762(3) | 10401(1) | 9647(2) | 54(1) |
| C(122) | -2373(3) | 11331(2) | 9507(2) | 67(1) |
| C(131) | -4145(3) | 10227(1) | 9091(2) | 53(1) |
| C(132) | -5109(2) | 10567(1) | 7826(2) | 41(1) |
| C(141) | -3928(2) | 11717(1) | 7770(2) | 44(1) |
| C(142) | -2448(3) | 11462(1) | 7556(2) | 50(1) |
| C(151) | -2398(2) | 7984(1) | 7005(2) | 42(1) |
| C(152) | -3036(2) | 8726(1) | 6088(2) | 40(1) |

| | | | | |
|---|---|---|---|---|
| C(161) | -441(2) | 8710(1) | 7100(2) | 46(1) |
| C(162) | -1163(2) | 9590(1) | 6568(2) | 41(1) |
| C(171) | -612(2) | 9008(1) | 8986(2) | 50(1) |
| C(172) | -2121(3) | 8517(1) | 8756(2) | 51(1) |

**counteranion:**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| B | 2079(2) | 7538(1) | 7053(2) | 21(1) |
| C(101) | 1902(2) | 7249(1) | 7758(1) | 18(1) |
| C(102) | 1358(2) | 7463(1) | 8141(2) | 21(1) |
| C(103) | 1203(2) | 7190(1) | 8718(2) | 23(1) |
| C(104) | 1580(2) | 6687(1) | 8935(2) | 24(1) |
| C(105) | 2107(2) | 6461(1) | 8553(2) | 23(1) |
| C(106) | 2258(2) | 6735(1) | 7978(2) | 21(1) |
| C(107) | 667(2) | 7441(1) | 9151(2) | 33(1) |
| F(1) | 519(2) | 7959(1) | 9020(1) | 71(1) |
| F(2) | -126(2) | 7211(1) | 9062(2) | 108(1) |
| F(3) | 1139(2) | 7424(1) | 9890(1) | 65(1) |
| C(108) | 2551(2) | 5926(1) | 8791(2) | 30(1) |
| F(4) | 3432(3) | 5931(2) | 8893(6) | 76(3) |
| F(5) | 2195(7) | 5556(2) | 8308(5) | 100(5) |
| F(6) | 2499(8) | 5758(3) | 9417(5) | 92(4) |
| F(4') | 2995(13) | 5732(3) | 8384(8) | 109(7) |
| F(5') | 1946(4) | 5560(3) | 8786(8) | 76(4) |
| F(6') | 3134(9) | 5913(3) | 9461(6) | 95(6) |
| C(201) | 3163(2) | 7452(1) | 7094(2) | 21(1) |
| C(202) | 3906(2) | 7393(1) | 7766(2) | 26(1) |
| C(203) | 4838(2) | 7363(1) | 7806(2) | 32(1) |
| C(204) | 5050(2) | 7397(1) | 7160(2) | 37(1) |
| C(205) | 4325(2) | 7464(1) | 6486(2) | 32(1) |
| C(206) | 3400(2) | 7492(1) | 6452(2) | 27(1) |
| C(207) | 5601(2) | 7302(2) | 8537(2) | 55(1) |
| F(7) | 5356(5) | 7155(4) | 9097(4) | 62(2) |
| F(8) | 6239(10) | 6940(8) | 8561(6) | 155(6) |
| F(9) | 6049(10) | 7750(5) | 8813(6) | 148(5) |
| F(7') | 5458(8) | 6917(7) | 8910(9) | 178(7) |

| | | | | |
|---|---|---|---|---|
| F(8') | 6406(4) | 7247(5) | 8463(5) | 82(3) |
| F(9') | 5724(7) | 7740(4) | 8923(6) | 127(5) |
| C(208) | 4556(2) | 7526(1) | 5796(2) | 43(1) |
| F(10) | 5084(3) | 7146(1) | 5703(2) | 133(2) |
| F(11) | 4986(2) | 7975(1) | 5775(1) | 70(1) |
| F(12) | 3813(2) | 7539(2) | 5172(2) | 117(1) |
| C(301) | 1297(2) | 7266(1) | 6320(1) | 21(1) |
| C(302) | 1456(2) | 6799(1) | 5983(2) | 25(1) |
| C(303) | 758(2) | 6556(1) | 5397(2) | 29(1) |
| C(304) | -132(2) | 6773(1) | 5107(2) | 29(1) |
| C(305) | -308(2) | 7236(1) | 5432(2) | 24(1) |
| C(306) | 386(2) | 7469(1) | 6029(2) | 23(1) |
| C(307) | 972(2) | 6058(1) | 5059(2) | 42(1) |
| F(13) | 1386(6) | 5697(3) | 5584(5) | 64(2) |
| F(14) | 1534(7) | 6122(5) | 4697(7) | 70(3) |
| F(15) | 209(4) | 5800(3) | 4642(8) | 81(3) |
| F(13') | 1072(18) | 5630(5) | 5432(11) | 94(6) |
| F(14') | 1748(13) | 6106(9) | 4923(16) | 98(6) |
| F(15') | 389(16) | 6001(9) | 4350(9) | 123(8) |
| C(308) | -1231(2) | 7508(1) | 5109(2) | 33(1) |
| F(16) | -1859(1) | 7232(1) | 4574(1) | 64(1) |
| F(17) | -1155(1) | 7979(1) | 4809(2) | 73(1) |
| F(18) | -1625(1) | 7616(1) | 5612(1) | 51(1) |
| C(401) | 1971(2) | 8193(1) | 7028(2) | 21(1) |
| C(402) | 1649(2) | 8473(1) | 6352(2) | 24(1) |
| C(403) | 1624(2) | 9027(1) | 6319(2) | 27(1) |
| C(404) | 1932(2) | 9329(1) | 6966(2) | 30(1) |
| C(405) | 2271(2) | 9062(1) | 7642(2) | 28(1) |
| C(406) | 2288(2) | 8507(1) | 7670(2) | 24(1) |
| C(407) | 1281(2) | 9296(1) | 5573(2) | 42(1) |
| F(19) | 1834(13) | 9223(9) | 5195(11) | 124(6) |
| F(20) | 477(8) | 9098(6) | 5150(8) | 86(4) |
| F(21) | 1133(11) | 9803(3) | 5606(8) | 93(4) |
| F(19') | 720(14) | 9026(5) | 5030(9) | 94(5) |
| F(20') | 1983(9) | 9446(9) | 5374(10) | 101(5) |
| F(21') | 854(13) | 9748(5) | 5582(11) | 93(4) |
| C(408) | 2593(3) | 9379(1) | 8350(2) | 48(1) |

| | | | | |
|---|---|---|---|---|
| F(22) | 1936(9) | 9604(7) | 8529(7) | 118(5) |
| F(23) | 3180(16) | 9763(7) | 8332(8) | 146(7) |
| F(24) | 3064(10) | 9110(5) | 8951(7) | 67(3) |
| F(22') | 1908(9) | 9384(5) | 8632(7) | 108(4) |
| F(23') | 2733(9) | 9876(2) | 8255(6) | 81(3) |
| F(24') | 3316(11) | 9182(6) | 8841(8) | 121(6) |

**solvent:**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| O(100) | -5199(2) | 14455(2) | 12988(2) | 121(2) |
| C(100) | -3833(3) | 14142(2) | 13879(3) | 74(1) |
| C(200) | -4757(4) | 14335(4) | 13735(4) | 136(3) |
| C(300) | -3802(3) | 14037(1) | 13114(2) | 57(1) |
| C(400) | -4617(3) | 14341(2) | 12604(3) | 82(1) |

**Table S3. Full list of bond lengths [Å] and angles [°] for , e.s.d's in parentheses refers to the last significant digit**

| | | | | | |
|---|---|---|---|---|---|
| Ru | - P(2) | 2.3671(9) | Ru | - P(1) | 2.3889(9) |
| Ru | - N | 2.108(2) | H(1c) | - H(2c) | 0.816(43) |
| Ru | - H(1a) | 1.528(20) | Ru | - H(1c) | 1.564(20) |
| Ru | - H(2c) | 1.547(21) | Ru | - H(1b) | 1.924(33) |
| H(1a) | - H(1c) | 2.379(39) | H(1a) | - H(2c) | 1.793(46) |
| H(1a) | - H(1b) | 3.417(39) | H(1b) | - H(1c) | 2.465(46) |
| H(1b) | - H(2c) | 2.946(46) | Ru | - C(11) | 2.528(3) |
| P(1) | - C(13) | 1.843(3) | P(1) | - C(12) | 1.862(3) |
| P(1) | - C(14) | 1.873(3) | P(2) | - C(17) | 1.858(3) |
| P(2) | - C(15) | 1.861(3) | P(2) | - C(16) | 1.863(3) |
| N | - C(1) | 1.350(4) | N | - C(5) | 1.362(4) |
| C(1) | - C(2) | 1.379(5) | C(2) | - C(3) | 1.370(6) |
| C(3) | - C(4) | 1.370(6) | C(4) | - C(5) | 1.394(4) |
| C(5) | - C(6) | 1.466(5) | C(6) | - C(11) | 1.388(4) |
| C(6) | - C(7) | 1.409(5) | C(7) | - C(8) | 1.377(6) |
| C(8) | - C(9) | 1.358(6) | C(9) | - C(10) | 1.386(5) |
| C(10) | - C(11) | 1.391(4) | C(12) | - C(122) | 1.527(5) |
| C(12) | - C(121) | 1.535(5) | C(13) | - C(131) | 1.523(4) |
| C(13) | - C(132) | 1.524(5) | C(14) | - C(141) | 1.531(4) |
| C(14) | - C(142) | 1.532(4) | C(15) | - C(152) | 1.527(5) |
| C(15) | - C(151) | 1.537(4) | C(16) | - C(162) | 1.528(4) |
| C(16) | - C(161) | 1.536(4) | C(17) | - C(171) | 1.515(5) |
| C(17) | - C(172) | 1.533(5) | | | |

**counteranion:**

| | | | | | |
|---|---|---|---|---|---|
| B | - C(301) | 1.630(4) | B | - C(201) | 1.639(4) |
| B | - C(101) | 1.640(4) | B | - C(401) | 1.647(4) |
| C(101) | - C(102) | 1.391(3) | C(101) | - C(106) | 1.403(4) |
| C(102) | - C(103) | 1.390(4) | C(103) | - C(104) | 1.387(4) |
| C(103) | - C(107) | 1.491(4) | C(104) | - C(105) | 1.384(4) |
| C(105) | - C(106) | 1.386(4) | C(105) | - C(108) | 1.498(4) |

| | | | |
|---|---|---|---|
| C(107) - F(2) | 1.293(4) | C(107) - F(1) | 1.325(3) |
| C(107) - F(3) | 1.342(4) | C(108) - F(4) | 1.286(5) |
| C(108) - F(5) | 1.281(5) | C(108) - F(6) | 1.301(5) |
| C(108) - F(4') | 1.293(6) | C(108) - F(5') | 1.297(6) |
| C(108) - F(6') | 1.276(7) | C(201) - C(202) | 1.389(4) |
| C(201) - C(206) | 1.402(4) | C(202) - C(203) | 1.397(4) |
| C(203) - C(204) | 1.387(4) | C(203) - C(207) | 1.479(5) |
| C(204) - C(205) | 1.381(5) | C(205) - C(206) | 1.388(4) |
| C(205) - C(208) | 1.492(4) | C(207) - F(7) | 1.306(7) |
| C(207) - F(8) | 1.318(8) | C(207) - F(9) | 1.320(8) |
| C(207) - F(7') | 1.264(8) | C(207) - F(8') | 1.291(7) |
| C(207) - F(9') | 1.294(8) | C(208) - F(10) | 1.296(4) |
| C(208) - F(11) | 1.308(4) | C(208) - F(12) | 1.325(5) |
| C(301) - C(302) | 1.396(4) | C(301) - C(306) | 1.397(4) |
| C(302) - C(303) | 1.381(4) | C(303) - C(304) | 1.382(4) |
| C(303) - C(307) | 1.491(4) | C(304) - C(305) | 1.385(4) |
| C(305) - C(306) | 1.379(4) | C(305) - C(308) | 1.487(4) |
| C(307) - F(13) | 1.334(7) | C(307) - F(14) | 1.288(8) |
| C(307) - F(15) | 1.321(7) | C(307) - F(13') | 1.268(10) |
| C(307) - F(14') | 1.302(11) | C(307) - F(15') | 1.341(9) |
| C(308) - F(16) | 1.319(4) | C(308) - F(18) | 1.329(3) |
| C(308) - F(17) | 1.336(4) | C(401) - C(406) | 1.389(4) |
| C(401) - C(402) | 1.401(4) | C(402) - C(403) | 1.389(4) |
| C(403) - C(404) | 1.380(4) | C(403) - C(407) | 1.499(4) |
| C(404) - C(405) | 1.388(4) | C(405) - C(406) | 1.388(4) |
| C(405) - C(408) | 1.492(4) | C(407) - F(19) | 1.302(9) |
| C(407) - F(20) | 1.301(8) | C(407) - F(21) | 1.295(8) |
| C(407) - F(19') | 1.276(10) | C(407) - F(20') | 1.312(9) |
| C(407) - F(21') | 1.309(9) | C(408) - F(22) | 1.295(9) |
| C(408) - F(23) | 1.321(9) | C(408) - F(24) | 1.311(9) |
| C(408) - F(22') | 1.334(8) | C(408) - F(23') | 1.286(8) |
| C(408) - F(24') | 1.270(9) | | |

**solvent:**

| | | | |
|---|---|---|---|
| O(100) - C(400) | 1.366(6) | O(100) - C(200) | 1.382(7) |
| C(100) - C(200) | 1.422(7) | C(100) - C(300) | 1.502(6) |

| | |
|---|---|
| C(300) - C(400) | 1.491(6) |

| | | | | | | | |
|---|---|---|---|---|---|---|---|
| H(1a) | - Ru - | H(1c) | 100.6(17) | H(1a) | - Ru - | H(2c) | 71.32(19) |
| H(1b) | - Ru - | H(2c) | 115.7(18) | H(1b) | - Ru - | H(1c) | 89.32(16) |
| H(1a) | - Ru - | H(1b) | 163.54(14) | H(1b) | - Ru - | N | 79.39(9) |
| P(1) | - Ru - | H(1a) | 82.52(11) | P(1) | - Ru - | H(1c) | 83.90(11) |
| P(1) | - Ru - | H(2c) | 88.02(14) | P(1) | - Ru - | H(1b) | 111.80(9) |
| P(2) | - Ru - | H(1a) | 84.42(11) | P(2) | - Ru - | H(1c) | 88.77(12) |
| P(2) | - Ru - | H(2c) | 78.35(15) | P(2) | - Ru - | H(1b) | 82.71(9) |
| N | - Ru - | P(2) | 93.99(6) | N | - Ru - | P(1) | 96.24(6) |
| P(2) | - Ru - | P(1) | 163.56(3) | N | - Ru - | C(11) | 75.14(10) |
| P(2) | - Ru - | C(11) | 102.06(7) | P(1) | - Ru - | C(11) | 92.99(7) |
| C(13) | - P(1) - | C(12) | 104.88(15) | C(13) | - P(1) - | C(14) | 102.40(13) |
| C(12) | - P(1) - | C(14) | 104.23(14) | C(13) | - P(1) - | Ru | 120.09(10) |
| C(12) | - P(1) - | Ru | 109.54(10) | C(14) | - P(1) - | Ru | 114.19(9) |
| C(17) | - P(2) - | C(15) | 102.61(14) | C(17) | - P(2) - | C(16) | 101.81(14) |
| C(15) | - P(2) - | C(16) | 108.85(13) | C(17) | - P(2) - | Ru | 113.37(10) |
| C(15) | - P(2) - | Ru | 115.83(9) | C(16) | - P(2) - | Ru | 113.00(9) |
| C(1) | - N - | C(5) | 119.0(3) | C(1) | - N - | Ru | 121.8(2) |
| C(5) | - N - | Ru | 119.18(19) | N | - C(1) - | C(2) | 122.6(4) |
| C(3) | - C(2) - | C(1) | 118.4(4) | C(2) | - C(3) - | C(4) | 120.0(3) |
| C(3) | - C(4) - | C(5) | 120.0(4) | N | - C(5) - | C(4) | 120.0(3) |
| N | - C(5) - | C(6) | 117.2(2) | C(4) | - C(5) - | C(6) | 122.8(3) |
| C(11) | - C(6) - | C(7) | 117.2(3) | C(11) | - C(6) - | C(5) | 120.6(3) |
| C(7) | - C(6) - | C(5) | 122.1(3) | C(8) | - C(7) - | C(6) | 120.4(3) |
| C(9) | - C(8) - | C(7) | 121.6(3) | C(8) | - C(9) - | C(10) | 119.7(4) |
| C(9) | - C(10) - | C(11) | 119.3(4) | C(6) | - C(11) - | C(10) | 121.8(3) |
| C(6) | - C(11) - | Ru | 101.0(2) | C(10) | - C(11) - | Ru | 128.5(2) |
| C(122) | - C(12) - | C(121) | 108.7(3) | C(122) | - C(12) - | P(1) | 115.5(2) |
| C(121) | - C(12) - | P(1) | 115.2(2) | C(131) | - C(13) - | C(132) | 110.3(3) |
| C(131) | - C(13) - | P(1) | 113.2(2) | C(132) | - C(13) - | P(1) | 112.4(2) |
| C(141) | - C(14) - | C(142) | 110.2(2) | C(141) | - C(14) - | P(1) | 118.2(2) |
| C(142) | - C(14) - | P(1) | 111.1(2) | C(152) | - C(15) - | C(151) | 110.0(2) |
| C(152) | - C(15) - | P(2) | 111.47(19) | C(151) | - C(15) - | P(2) | 117.4(2) |
| C(162) | - C(16) - | C(161) | 110.3(3) | C(162) | - C(16) - | P(2) | 114.75(19) |
| C(161) | - C(16) - | P(2) | 115.8(2) | C(171) | - C(17) - | C(172) | 110.4(3) |

| | | | |
|---|---|---|---|
| C(171) - C(17) - P(2) | 113.3(2) | C(172) - C(17) - P(2) | 111.8(2) |

**counteranion:**

| | | | |
|---|---|---|---|
| C(301) - B - C(201) | 113.8(2) | C(301) - B - C(101) | 104.0(2) |
| C(201) - B - C(101) | 110.9(2) | C(301) - B - C(401) | 110.6(2) |
| C(201) - B - C(401) | 102.84(19) | C(101) - B - C(401) | 115.0(2) |
| C(102) - C(101) - C(106) | 115.7(2) | C(102) - C(101) - B | 124.4(2) |
| C(106) - C(101) - B | 119.8(2) | C(103) - C(102) - C(101) | 121.9(2) |
| C(104) - C(103) - C(102) | 121.3(2) | C(104) - C(103) - C(107) | 117.7(2) |
| C(102) - C(103) - C(107) | 120.9(2) | C(105) - C(104) - C(103) | 117.9(2) |
| C(104) - C(105) - C(106) | 120.4(2) | C(104) - C(105) - C(108) | 119.1(2) |
| C(106) - C(105) - C(108) | 120.4(2) | C(105) - C(106) - C(101) | 122.8(2) |
| F(2) - C(107) - F(1) | 108.7(3) | F(2) - C(107) - F(3) | 104.4(3) |
| F(1) - C(107) - F(3) | 103.1(3) | F(2) - C(107) - C(103) | 114.3(3) |
| F(1) - C(107) - C(103) | 113.3(2) | F(3) - C(107) - C(103) | 112.1(2) |
| F(4) - C(108) - F(6) | 105.3(5) | F(5) - C(108) - F(4) | 105.6(5) |
| F(5) - C(108) - F(6) | 106.3(5) | F(4') - C(108) - F(5') | 104.7(7) |
| F(6') - C(108) - F(4') | 105.6(7) | F(6') - C(108) - F(5') | 103.2(7) |
| F(4) - C(108) - C(105) | 111.9(3) | F(5) - C(108) - C(105) | 113.1(3) |
| F(6) - C(108) - C(105) | 114.0(3) | F(4') - C(108) - C(105) | 115.2(4) |
| F(5') - C(108) - C(105) | 112.7(3) | F(6') - C(108) - C(105) | 114.3(4) |
| C(202) - C(201) - C(206) | 116.2(2) | C(202) - C(201) - B | 122.5(2) |
| C(206) - C(201) - B | 121.0(2) | C(201) - C(202) - C(203) | 122.4(3) |
| C(204) - C(203) - C(202) | 120.1(3) | C(204) - C(203) - C(207) | 120.0(3) |
| C(202) - C(203) - C(207) | 120.0(3) | C(205) - C(204) - C(203) | 118.6(3) |
| C(204) - C(205) - C(206) | 120.9(3) | C(204) - C(205) - C(208) | 118.6(3) |
| C(206) - C(205) - C(208) | 120.5(3) | C(205) - C(206) - C(201) | 121.8(3) |
| F(7) - C(207) - F(8) | 101.3(8) | F(7) - C(207) - F(9) | 99.9(6) |
| F(8) - C(207) - F(9) | 107.3(8) | F(7') - C(207) - F(8') | 109.8(8) |
| F(7') - C(207) - F(9') | 110.0(10) | F(8') - C(207) - F(9') | 101.6(6) |
| F(7) - C(207) - C(203) | 116.9(4) | F(8) - C(207) - C(203) | 115.5(6) |
| F(9) - C(207) - C(203) | 114.2(6) | F(7') - C(207) - C(203) | 112.5(6) |
| F(8') - C(207) - C(203) | 111.4(5) | F(9') - C(207) - C(203) | 110.9(5) |
| F(10) - C(208) - F(11) | 106.5(3) | F(10) - C(208) - F(12) | 105.8(3) |
| F(11) - C(208) - F(12) | 102.9(3) | F(10) - C(208) - C(205) | 113.3(3) |

| | | | |
|---|---|---|---|
| F(11) - C(208) - C(205) | 113.4(3) | F(12) - C(208) - C(205) | 114.0(3) |
| C(302) - C(301) - C(306) | 115.5(2) | C(302) - C(301) - B | 123.4(2) |
| C(306) - C(301) - B | 120.8(2) | C(303) - C(302) - C(301) | 122.0(2) |
| C(302) - C(303) - C(304) | 121.2(2) | C(302) - C(303) - C(307) | 119.5(3) |
| C(304) - C(303) - C(307) | 119.3(3) | C(303) - C(304) - C(305) | 117.9(3) |
| C(306) - C(305) - C(304) | 120.5(3) | C(306) - C(305) - C(308) | 119.3(2) |
| C(304) - C(305) - C(308) | 120.1(3) | C(305) - C(306) - C(301) | 122.8(2) |
| F(13') - C(307) - F(14') | 104.8(11) | F(13') - C(307) - F(15') | 113.0(9) |
| F(14') - C(307) - F(15') | 97.5(11) | F(14) - C(307) - F(15) | 110.2(7) |
| F(14) - C(307) - F(13) | 105.3(7) | F(15) - C(307) - F(13) | 101.9(6) |
| F(13) - C(307) - C(303) | 111.0(5) | F(14) - C(307) - C(303) | 114.6(6) |
| F(15) - C(307) - C(303) | 112.8(4) | F(13') - C(307) - C(303) | 117.6(7) |
| F(14') - C(307) - C(303) | 111.2(10) | F(15') - C(307) - C(303) | 110.7(5) |
| F(16) - C(308) - F(18) | 106.2(2) | F(16) - C(308) - F(17) | 106.0(3) |
| F(18) - C(308) - F(17) | 105.2(2) | F(16) - C(308) - C(305) | 113.8(2) |
| F(18) - C(308) - C(305) | 113.1(3) | F(17) - C(308) - C(305) | 111.9(2) |
| C(406) - C(401) - C(402) | 115.6(2) | C(406) - C(401) - B | 122.3(2) |
| C(402) - C(401) - B | 121.8(2) | C(403) - C(402) - C(401) | 122.7(3) |
| C(404) - C(403) - C(402) | 120.4(3) | C(404) - C(403) - C(407) | 120.2(2) |
| C(402) - C(403) - C(407) | 119.4(3) | C(403) - C(404) - C(405) | 118.0(2) |
| C(406) - C(405) - C(404) | 121.2(3) | C(406) - C(405) - C(408) | 119.8(3) |
| C(404) - C(405) - C(408) | 119.0(3) | C(405) - C(406) - C(401) | 122.1(2) |
| F(20) - C(407) - F(19) | 104.7(10) | F(21) - C(407) - F(19) | 108.7(9) |
| F(21) - C(407) - F(20) | 105.0(9) | F(19') - C(407) - F(20') | 107.1(10) |
| F(19') - C(407) - F(21') | 105.8(10) | F(21') - C(407) - F(20') | 102.4(9) |
| F(19) - C(407) - C(403) | 113.0(8) | F(20) - C(407) - C(403) | 110.9(8) |
| F(21) - C(407) - C(403) | 113.9(7) | F(19') - C(407) - C(403) | 116.5(8) |
| F(20') - C(407) - C(403) | 111.1(8) | F(21') - C(407) - C(403) | 112.8(9) |
| F(22) - C(408) - F(23) | 106.4(8) | F(22) - C(408) - F(24) | 103.1(8) |
| F(24) - C(408) - F(23) | 103.5(10) | F(23') - C(408) - F(22') | 104.0(6) |
| F(24') - C(408) - F(22') | 107.5(9) | F(24') - C(408) - F(23') | 109.9(9) |
| F(22) - C(408) - C(405) | 115.7(7) | F(23) - C(408) - C(405) | 111.7(6) |
| F(24) - C(408) - C(405) | 115.2(7) | F(22') - C(408) - C(405) | 108.6(6) |
| F(23') - C(408) - C(405) | 113.6(6) | F(24') - C(408) - C(405) | 112.8(8) |

**solvent:**

| | | | |
|---|---|---|---|
| O(100) - C(200) - C(100) | 111.3(5) | O(100) - C(400) - C(300) | 107.5(4) |
| C(400) - O(100) - C(200) | 109.9(4) | C(200) - C(100) - C(300) | 103.8(4) |
| C(400) - C(300) - C(100) | 104.2(3) | | |

**Table S4. Anisotropic displacement parameters ($Å^2x10^3$) the anisotropic displacement factor exponent takes the form:**

$$\exp[-2p^2(U_{11}h^2a^{*2} + U_{22}k_2b^{*2} + U_{33}l^2c^{*2} + 2U_{12}hka^*b^* + 2U_{13}hla^*c^* + 2U_{23}klb^*c^*)]$$

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| Ru | 17(1) | 18(1) | 21(1) | -1(1) | 5(1) | 1(1) |
| P(1) | 25(1) | 19(1) | 22(1) | -2(1) | 7(1) | 1(1) |
| P(2) | 20(1) | 22(1) | 27(1) | -2(1) | 7(1) | 4(1) |
| N | 21(1) | 23(1) | 40(2) | -10(1) | 13(1) | 1(1) |
| C(1) | 33(2) | 25(1) | 64(2) | -8(1) | 27(2) | -1(1) |
| C(2) | 40(2) | 35(2) | 111(3) | -22(2) | 45(2) | -12(2) |
| C(3) | 26(2) | 56(2) | 109(4) | -43(2) | 22(2) | -11(2) |
| C(4) | 21(2) | 62(2) | 65(3) | -34(2) | 3(2) | 2(2) |
| C(5) | 21(1) | 41(2) | 39(2) | -21(1) | 5(1) | 7(1) |
| C(6) | 35(2) | 41(2) | 21(2) | -9(1) | 2(1) | 14(1) |
| C(7) | 44(2) | 71(2) | 28(2) | -7(2) | 1(2) | 27(2) |
| C(8) | 84(3) | 71(3) | 30(2) | 18(2) | 15(2) | 44(2) |
| C(9) | 89(3) | 61(2) | 35(2) | 20(2) | 31(2) | 31(2) |
| C(10) | 60(2) | 45(2) | 31(2) | 11(1) | 24(2) | 16(2) |
| C(11) | 38(2) | 34(2) | 20(2) | 3(1) | 7(1) | 15(1) |
| C(12) | 42(2) | 36(2) | 33(2) | -12(1) | 0(2) | 3(1) |
| C(13) | 41(2) | 27(1) | 40(2) | -1(1) | 24(2) | 4(1) |
| C(14) | 43(2) | 21(1) | 30(2) | 1(1) | 10(1) | 1(1) |
| C(15) | 25(1) | 24(1) | 42(2) | -8(1) | 14(1) | -1(1) |
| C(16) | 19(1) | 33(2) | 42(2) | -8(1) | 12(1) | 0(1) |
| C(17) | 41(2) | 31(2) | 34(2) | 4(1) | 10(1) | 19(1) |
| C(121) | 60(2) | 54(2) | 30(2) | -10(2) | -6(2) | 17(2) |
| C(122) | 75(3) | 51(2) | 48(3) | -25(2) | -10(2) | 13(2) |
| C(131) | 73(3) | 39(2) | 64(3) | 8(2) | 46(2) | 7(2) |
| C(132) | 32(2) | 40(2) | 57(2) | -11(2) | 22(2) | 0(1) |
| C(141) | 57(2) | 25(2) | 48(2) | 4(1) | 14(2) | 7(1) |
| C(142) | 61(2) | 37(2) | 60(2) | 5(2) | 33(2) | -9(2) |
| C(151) | 42(2) | 26(2) | 60(2) | -9(1) | 22(2) | -1(1) |
| C(152) | 35(2) | 37(2) | 39(2) | -12(1) | 3(2) | -2(1) |

| | | | | | | |
|---|---|---|---|---|---|---|
| C(161) | 32(2) | 45(2) | 67(3) | -7(2) | 26(2) | 6(1) |
| C(162) | 35(2) | 46(2) | 50(2) | -1(2) | 24(2) | -4(1) |
| C(171) | 53(2) | 51(2) | 34(2) | -2(2) | -1(2) | 24(2) |
| C(172) | 70(2) | 44(2) | 47(2) | 21(2) | 30(2) | 23(2) |

**counteranion:**

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| B | 23(2) | 20(1) | 22(2) | 0(1) | 10(1) | 1(1) |
| C(101) | 16(1) | 21(1) | 18(2) | -3(1) | 4(1) | -2(1) |
| C(102) | 18(1) | 21(1) | 22(2) | 0(1) | 5(1) | 2(1) |
| C(103) | 20(1) | 28(1) | 24(2) | 0(1) | 11(1) | 2(1) |
| C(104) | 24(1) | 28(1) | 22(2) | 4(1) | 10(1) | -3(1) |
| C(105) | 21(1) | 20(1) | 26(2) | 0(1) | 7(1) | -1(1) |
| C(106) | 20(1) | 21(1) | 24(2) | -3(1) | 9(1) | 0(1) |
| C(107) | 38(2) | 36(2) | 33(2) | 7(1) | 21(2) | 9(1) |
| F(1) | 110(2) | 60(1) | 72(2) | 22(1) | 67(2) | 48(1) |
| F(2) | 67(2) | 148(2) | 147(3) | -92(2) | 86(2) | -52(2) |
| F(3) | 83(2) | 87(2) | 35(1) | 5(1) | 32(1) | 36(1) |
| C(108) | 33(2) | 26(2) | 34(2) | 5(1) | 15(2) | 3(1) |
| F(4) | 34(2) | 40(3) | 154(8) | 35(4) | 32(3) | 21(2) |
| F(5) | 117(7) | 27(3) | 90(6) | -28(3) | -45(5) | 28(3) |
| F(6) | 157(9) | 71(5) | 87(6) | 61(4) | 90(7) | 71(5) |
| F(4') | 224(16) | 41(5) | 139(11) | 48(6) | 161(12) | 70(8) |
| F(5') | 46(3) | 26(3) | 149(12) | 35(5) | 25(5) | -5(2) |
| F(6') | 108(9) | 43(4) | 66(7) | -14(4) | -52(6) | 33(5) |
| C(201) | 26(1) | 17(1) | 24(2) | -1(1) | 12(1) | 0(1) |
| C(202) | 24(1) | 27(1) | 29(2) | -3(1) | 13(1) | -2(1) |
| C(203) | 24(1) | 35(2) | 40(2) | -5(1) | 14(1) | -2(1) |
| C(204) | 28(2) | 41(2) | 51(2) | -11(1) | 25(2) | -7(1) |
| C(205) | 38(2) | 29(2) | 41(2) | -7(1) | 28(2) | -7(1) |
| C(206) | 33(2) | 22(1) | 30(2) | -1(1) | 16(1) | -2(1) |
| C(207) | 24(2) | 82(3) | 56(3) | -4(2) | 13(2) | 1(2) |
| F(7) | 25(3) | 126(5) | 31(3) | 11(3) | 7(2) | 14(3) |

| | | | | | | |
|---|---|---|---|---|---|---|
| F(8) | 111(9) | 287(14) | 59(5) | 3(8) | 18(6) | 147(9) |
| F(9) | 145(9) | 167(8) | 87(6) | 18(6) | -17(6) | -122(8) |
| F(7') | 78(7) | 240(12) | 144(11) | 142(10) | -54(6) | -60(8) |
| F(8') | 9(2) | 166(8) | 65(5) | -18(5) | 4(2) | 11(3) |
| F(9') | 88(6) | 186(8) | 66(5) | -84(6) | -24(4) | 64(6) |
| C(208) | 45(2) | 51(2) | 44(2) | -13(2) | 32(2) | -15(2) |
| F(10) | 250(4) | 79(2) | 164(3) | 44(2) | 189(3) | 67(2) |
| F(11) | 107(2) | 63(1) | 63(2) | -4(1) | 57(2) | -31(1) |
| F(12) | 91(2) | 233(4) | 45(2) | -36(2) | 47(2) | -73(2) |
| C(301) | 25(1) | 22(1) | 19(2) | 2(1) | 11(1) | 0(1) |
| C(302) | 27(1) | 24(1) | 25(2) | 1(1) | 9(1) | 3(1) |
| C(303) | 35(2) | 25(1) | 28(2) | -5(1) | 13(1) | -2(1) |
| C(304) | 31(2) | 32(2) | 22(2) | -3(1) | 8(1) | -5(1) |
| C(305) | 25(1) | 28(1) | 20(2) | 5(1) | 9(1) | 0(1) |
| C(306) | 28(1) | 22(1) | 21(2) | 1(1) | 11(1) | 2(1) |
| C(307) | 39(2) | 39(2) | 45(2) | -17(2) | 14(2) | -1(1) |
| F(13) | 82(4) | 27(3) | 71(3) | -8(2) | 12(3) | 17(3) |
| F(14) | 120(6) | 47(4) | 75(5) | -5(4) | 76(5) | 8(4) |
| F(15) | 36(3) | 63(4) | 126(7) | -67(4) | 8(3) | -7(2) |
| F(13') | 193(16) | 24(4) | 122(10) | -12(5) | 126(11) | -11(7) |
| F(14') | 93(7) | 84(10) | 152(16) | -79(9) | 88(9) | -26(6) |
| F(15') | 129(10) | 108(11) | 72(7) | -74(7) | -38(7) | 68(8) |
| C(308) | 27(2) | 37(2) | 31(2) | 3(1) | 7(1) | 1(1) |
| F(16) | 36(1) | 77(1) | 56(2) | -24(1) | -10(1) | 12(1) |
| F(17) | 43(1) | 64(1) | 105(2) | 54(1) | 17(1) | 15(1) |
| F(18) | 36(1) | 70(1) | 45(1) | -2(1) | 13(1) | 18(1) |
| C(401) | 17(1) | 22(1) | 25(2) | 2(1) | 10(1) | 1(1) |
| C(402) | 25(1) | 24(1) | 26(2) | 0(1) | 11(1) | -2(1) |
| C(403) | 25(1) | 24(1) | 30(2) | 4(1) | 8(1) | -2(1) |
| C(404) | 28(1) | 19(1) | 42(2) | 1(1) | 12(1) | -1(1) |
| C(405) | 26(1) | 24(1) | 32(2) | -4(1) | 9(1) | -1(1) |
| C(406) | 23(1) | 25(1) | 24(2) | 0(1) | 8(1) | 2(1) |
| C(407) | 47(2) | 30(2) | 40(2) | 9(1) | 4(2) | -4(1) |
| F(19) | 129(10) | 199(14) | 72(8) | 79(8) | 70(9) | 76(8) |
| F(20) | 77(5) | 89(7) | 50(7) | 32(4) | -30(4) | -35(4) |
| F(21) | 159(10) | 19(3) | 53(5) | 12(3) | -21(5) | -19(4) |

| | | | | | | |
|---|---|---|---|---|---|---|
| F(19') | 173(12) | 39(4) | 32(4) | 10(3) | -10(7) | -37(6) |
| F(20') | 62(5) | 168(12) | 71(9) | 80(7) | 21(4) | -11(6) |
| F(21') | 115(8) | 76(7) | 76(7) | 30(6) | 20(6) | 63(7) |
| C(408) | 66(2) | 30(2) | 41(2) | -8(1) | 11(2) | 0(2) |
| F(22) | 115(8) | 134(11) | 72(6) | -53(6) | -6(5) | 86(7) |
| F(23) | 251(15) | 123(10) | 67(7) | -49(7) | 59(10) | -148(10) |
| F(24) | 111(7) | 41(4) | 28(3) | -11(3) | 0(4) | 16(4) |
| F(22') | 149(7) | 119(8) | 86(6) | -69(5) | 81(6) | -41(6) |
| F(23') | 143(7) | 21(3) | 59(4) | -18(2) | 9(5) | -5(3) |
| F(24') | 121(8) | 86(8) | 77(8) | -48(5) | -63(6) | 41(6) |

**solvent:**

| | U11 | U22 | U33 | U23 | U13 | U12 |
|---|---|---|---|---|---|---|
| O(100) | 71(2) | 193(4) | 108(3) | 49(3) | 42(2) | 79(3) |
| C(100) | 70(3) | 73(3) | 80(3) | 9(2) | 27(2) | 21(2) |
| C(200) | 93(4) | 229(8) | 101(5) | 38(5) | 53(4) | 84(5) |
| C(300) | 53(2) | 43(2) | 74(3) | -4(2) | 23(2) | 16(2) |
| C(400) | 68(3) | 94(3) | 77(3) | 2(3) | 18(2) | 34(2) |

**Table 5. Hydrogen coordinates ($x10^4$) and isotropic displacement parameters ($Å^2 x10^3$)**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(1a) | -2570(20) | 9644(11) | 8474(13) | 35(8) |
| H(1b) | -2980(20) | 9945(12) | 6648(18) | 31(8) |
| H(1c) | -1774(17) | 10177(12) | 7860(20) | 37(9) |
| H(2c) | -1693(16) | 9978(16) | 8210(20) | 59(11) |
| H(1) | -3946 | 9039 | 8105 | 46 |
| H(2) | -5426 | 8653 | 7617 | 68 |
| H(3) | -6444 | 8926 | 6449 | 77 |
| H(4) | -5989 | 9595 | 5813 | 63 |
| H(7) | -5639 | 10463 | 5627 | 61 |
| H(8) | -5078 | 11167 | 5124 | 75 |
| H(9) | -3499 | 11279 | 5373 | 71 |
| H(10) | -2423 | 10678 | 6159 | 52 |
| H(12) | -1657 | 10970 | 8917 | 49 |
| H(13) | -4241 | 10998 | 8716 | 40 |
| H(14) | -3747 | 11124 | 7099 | 38 |
| H(15) | -3333 | 8566 | 7025 | 36 |
| H(16) | -486 | 9400 | 7656 | 37 |
| H(17) | -1122 | 8374 | 8281 | 43 |
| H(12A) | -1588 | 10102 | 9392 | 81 |
| H(12B) | -2230 | 10282 | 9856 | 81 |
| H(12C) | -1204 | 10533 | 10051 | 81 |
| H(12D) | -2546 | 11640 | 9173 | 100 |
| H(12E) | -1810 | 11417 | 9938 | 100 |
| H(12F) | -2891 | 11244 | 9679 | 100 |
| H(13A) | -3655 | 10332 | 9562 | 79 |
| H(13B) | -3993 | 9877 | 8932 | 79 |
| H(13C) | -4750 | 10206 | 9163 | 79 |
| H(13D) | -5074 | 10776 | 7405 | 62 |
| H(13E) | -5642 | 10689 | 7957 | 62 |

| | | | | |
|---|---|---|---|---|
| H(13F) | -5192 | 10188 | 7685 | 62 |
| H(14A) | -4539 | 11586 | 7758 | 67 |
| H(14B) | -4021 | 11993 | 7389 | 67 |
| H(14C) | -3582 | 11869 | 8264 | 67 |
| H(14D) | -2082 | 11161 | 7478 | 75 |
| H(14E) | -2086 | 11649 | 8019 | 75 |
| H(14F) | -2588 | 11709 | 7135 | 75 |
| H(15A) | -2274 | 7872 | 7520 | 62 |
| H(15B) | -1819 | 7958 | 6892 | 62 |
| H(15C) | -2880 | 7752 | 6663 | 62 |
| H(15D) | -3251 | 9098 | 6032 | 59 |
| H(15E) | -3547 | 8493 | 5784 | 59 |
| H(15F) | -2499 | 8691 | 5923 | 59 |
| H(16A) | -329 | 8464 | 7520 | 69 |
| H(16B) | 161 | 8817 | 7065 | 69 |
| H(16C) | -825 | 8532 | 6636 | 69 |
| H(16D) | -1402 | 9928 | 6689 | 62 |
| H(16E) | -1635 | 9429 | 6125 | 62 |
| H(16F) | -586 | 9656 | 6466 | 62 |
| H(17A) | -135 | 9101 | 8772 | 75 |
| H(17B) | -329 | 8786 | 9429 | 75 |
| H(17C) | -860 | 9336 | 9128 | 75 |
| H(17D) | -2655 | 8348 | 8369 | 77 |
| H(17E) | -2341 | 8825 | 8965 | 77 |
| H(17F) | -1826 | 8259 | 9153 | 77 |

**counteranion:**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(102) | 1084 | 7805 | 8004 | 25 |
| H(104) | 1480 | 6504 | 9334 | 29 |
| H(106) | 2619 | 6569 | 7722 | 25 |
| H(202) | 3776 | 7373 | 8215 | 31 |
| H(204) | 5681 | 7374 | 7180 | 45 |
| H(206) | 2915 | 7538 | 5981 | 32 |

| | | | | |
|---|---|---|---|---|
| H(302) | 2063 | 6643 | 6161 | 30 |
| H(304) | -607 | 6609 | 4697 | 35 |
| H(306) | 239 | 7780 | 6251 | 27 |
| H(402) | 1439 | 8275 | 5898 | 29 |
| H(404) | 1912 | 9708 | 6949 | 36 |
| H(406) | 2524 | 8337 | 8142 | 29 |

**solvent:**

| | x | y | z | U(eq) |
|---|---|---|---|---|
| H(10A) | -3720 | 13810 | 14177 | 89 |
| H(10B) | -3358 | 14413 | 14149 | 89 |
| H(20A) | -5127 | 14062 | 13883 | 163 |
| H(20B) | -4729 | 14660 | 14040 | 163 |
| H(30A) | -3206 | 14167 | 13078 | 68 |
| H(30B) | -3868 | 13650 | 12995 | 68 |
| H(40A) | -4399 | 14675 | 12442 | 98 |
| H(40B) | -4954 | 14124 | 12155 | 98 |

(') correspond fluor atoms of $CF_3$ groups statistically disordered on two sites.