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# *trans*-Tetrakis(4-methylpyridine-*κN*)-dioxidorhenium(V) hexafluoridophosphate

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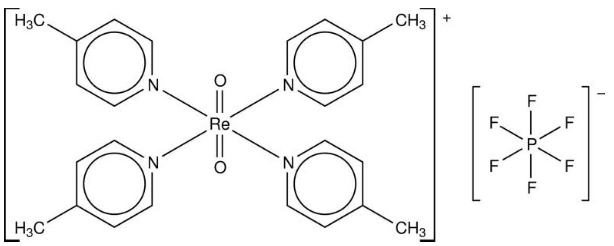
Key indicators: single-crystal X-ray study; *T* = 173 K; mean σ(C–C) = 0.004 Å; *R* factor = 0.022; *wR* factor = 0.044; data-to-parameter ratio = 17.8.

The title compound, $[ReO_2(C_6H_7N)_4]PF_6$, contains octahedral $[ReO_2(4\text{-Mepy})_4]^+$ cations (4-Mepy is 4-methylpyridine) and $PF_6^-$ anions. Both the cation and the anion reside on special positions, the Re atom on a crystallographic center of inversion and the P atom on a $C_2$ axis parallel to the *b* axis. The $Re^V$ atom in the cation exhibits an octahedral coordination geometry with two O atoms in the apical positions and four N atoms of the 4-Mepy ligands in the equatorial plane. The Re═O and Re—N bond lengths fall in the typical ranges of *trans*-dioxidorhenium(V) complexes.

## Related literature

For rhenium(V) complexes as radiopharmaceuticals, see: Dilworth & Parrott (1998); Volkert & Hoffman (1999). *trans*-Dioxidorhenium(V) $ReO_2^+$ complexes exhibit interesting properties as redox- and photo-active catalysts, see: Grey *et al.* (2004); Pipes & Meyer (1985); Thorp *et al.* (1989). For the synthesis of the title compound, see: Brewer & Gray (1989). For the crystal structures of *trans*-dioxidorhenium(V) complexes, see: Bélanger & Beauchamp (1996); Canlier *et al.* (2010); Gancheff *et al.* (2006); Kochel (2006); Kremer *et al.* (1996); Machura *et al.* (2008); Luck & O'Neill (2001); Reddy *et al.* (1999); Siczek *et al.* (2009).



## Experimental

### *Crystal data*

$[ReO_2(C_6H_7N)_4]PF_6$
$M_r$ = 735.68
Monoclinic, $C2/c$
*a* = 10.4914 (4) Å
*b* = 19.5359 (8) Å
*c* = 14.0923 (5) Å
β = 109.5810 (11)°
*V* = 2721.31 (18) Å$^3$
*Z* = 4
Mo *Kα* radiation
μ = 4.60 mm$^{-1}$
*T* = 173 K
0.26 × 0.13 × 0.12 mm

### *Data collection*

Rigaku R-AXIS RAPID diffractometer
Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995)
$T_{min}$ = 0.354, $T_{max}$ = 0.576
12743 measured reflections
3113 independent reflections
2556 reflections with $F^2 > 2\sigma(F^2)$
$R_{int}$ = 0.021

### *Refinement*

$R[F^2 > 2\sigma(F^2)] = 0.022$
$wR(F^2) = 0.044$
*S* = 1.16
3113 reflections
175 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 0.46 e Å$^{-3}$
$\Delta\rho_{min}$ = −0.39 e Å$^{-3}$

**Table 1**
Selected geometric parameters (Å, °).

| | | | |
|---|---|---|---|
| Re1—O1 | 1.7688 (19) | Re1—N2 | 2.146 (2) |
| Re1—N1 | 2.147 (2) | | |
| O1—Re1—O1$^i$ | 180.00 (12) | O1—Re1—N2 | 90.35 (8) |
| O1—Re1—N1 | 90.33 (9) | N1—Re1—N2 | 90.20 (8) |

Symmetry code: (i) $-x+\frac{3}{2}, -y+\frac{1}{2}, -z+1$.

Data collection: *PROCESS-AUTO* (Rigaku, 2006); cell refinement: *PROCESS-AUTO*; data reduction: *CrystalStructure* (Rigaku/MSC, 2006); program(s) used to solve structure: *SIR92* (Altomare *et al.*, 1994); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *CrystalMaker* (CrystalMaker, 2007); software used to prepare material for publication: *CrystalStructure* (Rigaku/MSC, 2006).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: ZQ2045).

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# supplementary materials

*Acta Cryst.* (2010). E**66**, m857-m858    [ **doi:10.1107/S160053681002458X** ]

# ***trans*-Tetrakis(4-methylpyridine-*κN*)dioxidorhenium(V) hexafluoridophosphate**

**T. Kawasaki, A. Canlier, S. Chowdhury and Y. Ikeda**

## Comment

Rhenium(V) complexes as radiopharmaceuticals for therapy and diagnosis continue to attract attention, because rhenium isotopes have suitable radionuclear properties for the applications, *i.e.*, $^{186}$Re: $E_{max}$ = 1.1 MeV for β-emission and $E_{max}$ = 0.137 MeV for γ-emission with $t_{1/2}$ = 90.6 h, $^{188}$Re: $E_{max}$ = 2.1 MeV for β-emission and $E_{max}$ = 0.155 MeV for γ-emission with $t_{1/2}$ = 17 h (Dilworth & Parrott, 1998; Volkert & Hoffman, 1999). On the other hand, *trans*-dioxorhenium(V) $ReO_2^+$ complexes have been known to exhibit interesting properties as redox- and photo-active catalysts (Grey *et al.*, 2004; Pipes & Meyer, 1985; Thorp *et al.*,1989). To our knowledge, the title compound of formula $[ReO_2(4\text{-Mepy})_4]^+.PF_6^-$ (4-Mepy = 4-methylpyridine) (I) was already synthesized by Brewer & Gray (Brewer & Gray, 1989), but a crystallographic characterization has not been yet reported. In this article, we report the X-ray crystal structure of the title compound.

Complex I crystallized in the centrosymmetric space group *C*2/*c*. The crystal structure is constructed by the packing of $[ReO_2(4\text{-Mepy})_4]^+$ cations and octahedral $PF_6^-$ anions as shown Figs. 1 and 2. The Re atom is located on a crystallographic center of inversion and the P atom lies on a $C_2$ axis parallel to the *b* axis. The $Re^V$ atom in the cation exhibits an octahedral coordination geometry with two O atoms in the apical positions and four N atoms of the 4-Mepy ligands in the equatorial plane. The Re═O and Re—N bond lengths fall in the typical ranges of *trans*-dioxorhenium(V) complexes. No classical hydrogen bonds are observed in the crystal structure. The N1—Re1—N2 bond angle and all N—Re1═O angles are almost 90 °. The bond lengths of Re1—O1N, Re1—N1, and Re1—N2 are 1.769 (2), 2.147 (2) and 2.146 (2) Å, respectively. These values are similar to those found for other *trans*-dioxorhenium(V) complexes (Bélanger & Beauchamp, 1996; Canlier *et al.*, 2010; Gancheff *et al.*, 2006; Kochel, 2006; Kremer *et al.*, 1996; Machura *et al.*, 2008; Luck & O'Neill, 2001; Reddy *et al.*, 1999; Siczek *et al.*, 2009).

## Experimental

The title complex was synthesized according to the literature method by Brewer & Gray (Brewer & Gray, 1989). $[ReO_2(PPh_3)_2]I$ was reacted with 4-Mepy in methanol and the resulting $[ReO_2(4\text{-Mepy})_4]I$ was reacted with $NH_4PF_6$ in methanol.

## Refinement

All H atoms were positionated geometrically, with C—H = 0.95 and 0.98 Å for aromatic and methyl H atoms, respectively, and constrained to ride on their parent atoms, with $U_{iso}(H) = 1.2U_{eq}(C)$.

## Figures

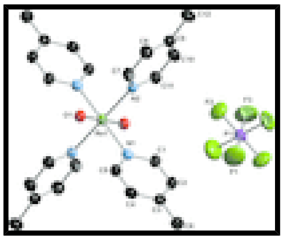

Fig. 1. View of the $[ReO_2(4\text{-}Mepy)_4]^+$ cation and $PF_6^-$ anion with 50% thermal ellipsoids. Hydrogen atoms are omitted clarity.

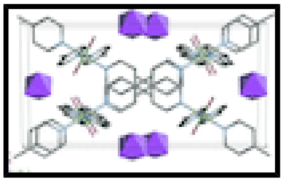

Fig. 2. Packing view of the $[ReO_2(4\text{-}Mepy)_4]^+$ (stick) and $PF_6^-$ (octahedron) along the *c* axis.

## *trans*-Tetrakis(4-methylpyridine-κ*N*)dioxidorhenium(V) hexafluoridophosphate

### *Crystal data*

| | |
|---|---|
| $[ReO_2(C_6H_7N)_4]PF_6$ | $F(000) = 1440.00$ |
| $M_r = 735.68$ | $D_x = 1.796$ Mg m$^{-3}$ |
| Monoclinic, *C*2/*c* | Mo *K*α radiation, λ = 0.71075 Å |
| Hall symbol: -C 2yc | Cell parameters from 13289 reflections |
| *a* = 10.4914 (4) Å | θ = 3.1–27.4° |
| *b* = 19.5359 (8) Å | μ = 4.60 mm$^{-1}$ |
| *c* = 14.0923 (5) Å | *T* = 173 K |
| β = 109.5810 (11)° | Block, orange |
| *V* = 2721.31 (18) Å$^3$ | 0.26 × 0.13 × 0.12 mm |
| *Z* = 4 | |

### *Data collection*

| | |
|---|---|
| Rigaku R-AXIS RAPID diffractometer | 3113 independent reflections |
| Radiation source: fine-focus sealed tube | 2556 reflections with $F^2 > 2\sigma(F^2)$ |
| graphite | $R_{int} = 0.021$ |
| Detector resolution: 10.00 pixels mm$^{-1}$ | $\theta_{max} = 27.4°$ |
| ω scans | *h* = −12→13 |
| Absorption correction: multi-scan (*ABSCOR*; Higashi, 1995) | *k* = −25→25 |
| $T_{min} = 0.354$, $T_{max} = 0.576$ | *l* = −18→18 |
| 12743 measured reflections | |

### *Refinement*

| | |
|---|---|
| Refinement on $F^2$ | Secondary atom site location: difference Fourier map |

| | |
|---|---|
| $R[F^2 > 2\sigma(F^2)] = 0.022$ | Hydrogen site location: inferred from neighbouring sites |
| $wR(F^2) = 0.044$ | H-atom parameters constrained |
| $S = 1.16$ | $w = 1/[\sigma^2(F_o^2) + (0.0157P)^2 + 6.0203P]$<br>where $P = (F_o^2 + 2F_c^2)/3$ |
| 3113 reflections | $(\Delta/\sigma)_{max} < 0.001$ |
| 175 parameters | $\Delta\rho_{max} = 0.46$ e Å$^{-3}$ |
| 0 restraints | $\Delta\rho_{min} = -0.39$ e Å$^{-3}$ |
| Primary atom site location: structure-invariant direct methods | |

### *Special details*

**Geometry**. All e.s.d.'s (except the e.s.d. in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell e.s.d.'s are taken into account individually in the estimation of e.s.d.'s in distances, angles and torsion angles; correlations between e.s.d.'s in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell e.s.d.'s is used for estimating e.s.d.'s involving l.s. planes.

**Refinement**. Refinement of $F^2$ against ALL reflections. The weighted $R$-factor $wR$ and goodness of fit $S$ are based on $F^2$, conventional $R$-factors $R$ are based on $F$, with $F$ set to zero for negative $F^2$. The threshold expression of $F^2 > \sigma(F^2)$ is used only for calculating $R$-factors(gt) *etc*. and is not relevant to the choice of reflections for refinement. $R$-factors based on $F^2$ are statistically about twice as large as those based on $F$, and $R$- factors based on ALL data will be even larger.

### *Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å$^2$)*

| | $x$ | $y$ | $z$ | $U_{iso}*/U_{eq}$ |
|---|---|---|---|---|
| Re1 | 0.7500 | 0.2500 | 0.5000 | 0.02256 (4) |
| P1 | 0.5000 | 0.04791 (6) | 0.7500 | 0.0334 (2) |
| F1 | 0.5776 (2) | 0.10485 (15) | 0.8276 (2) | 0.0838 (8) |
| F2 | 0.6185 (2) | 0.04807 (14) | 0.70289 (19) | 0.0670 (6) |
| F3 | 0.4238 (3) | −0.00954 (16) | 0.6730 (2) | 0.0962 (10) |
| O1 | 0.90491 (19) | 0.29340 (10) | 0.52054 (14) | 0.0268 (4) |
| N1 | 0.7527 (2) | 0.27819 (12) | 0.64789 (17) | 0.0257 (4) |
| N2 | 0.8575 (2) | 0.15747 (12) | 0.55935 (16) | 0.0249 (4) |
| C1 | 0.6844 (3) | 0.24197 (16) | 0.6970 (2) | 0.0311 (6) |
| C2 | 0.6861 (3) | 0.25974 (15) | 0.7926 (2) | 0.0331 (6) |
| C3 | 0.7583 (3) | 0.31654 (15) | 0.8417 (2) | 0.0311 (6) |
| C4 | 0.8272 (3) | 0.35354 (15) | 0.7905 (2) | 0.0319 (6) |
| C5 | 0.8225 (2) | 0.33362 (15) | 0.6953 (2) | 0.0294 (5) |
| C6 | 0.7652 (3) | 0.33635 (18) | 0.9466 (2) | 0.0440 (8) |
| C7 | 0.9942 (3) | 0.15588 (16) | 0.5950 (2) | 0.0331 (6) |
| C8 | 1.0667 (3) | 0.09651 (17) | 0.6268 (2) | 0.0372 (6) |
| C9 | 1.0011 (3) | 0.03451 (16) | 0.6232 (2) | 0.0323 (6) |
| C10 | 0.8602 (3) | 0.03686 (16) | 0.5894 (2) | 0.0365 (6) |
| C11 | 0.7926 (3) | 0.09779 (15) | 0.5586 (2) | 0.0313 (6) |
| C12 | 1.0782 (3) | −0.03129 (17) | 0.6533 (2) | 0.0418 (7) |
| H1 | 0.6337 | 0.2031 | 0.6650 | 0.037* |
| H2 | 0.6373 | 0.2328 | 0.8249 | 0.040* |

| | | | | |
|---|---|---|---|---|
| H4 | 0.8780 | 0.3928 | 0.8209 | 0.038* |
| H5 | 0.8704 | 0.3600 | 0.6617 | 0.035* |
| H6A | 0.7107 | 0.3044 | 0.9706 | 0.053* |
| H6B | 0.7299 | 0.3829 | 0.9458 | 0.053* |
| H6C | 0.8594 | 0.3347 | 0.9916 | 0.053* |
| H7 | 1.0423 | 0.1975 | 0.5982 | 0.040* |
| H8 | 1.1628 | 0.0981 | 0.6514 | 0.045* |
| H10 | 0.8102 | −0.0040 | 0.5876 | 0.044* |
| H11 | 0.6965 | 0.0977 | 0.5360 | 0.038* |
| H12A | 1.0144 | −0.0693 | 0.6453 | 0.050* |
| H12B | 1.1370 | −0.0284 | 0.7238 | 0.050* |
| H12C | 1.1334 | −0.0391 | 0.6103 | 0.050* |

*Atomic displacement parameters (Å$^2$)*

| | $U^{11}$ | $U^{22}$ | $U^{33}$ | $U^{12}$ | $U^{13}$ | $U^{23}$ |
|---|---|---|---|---|---|---|
| Re1 | 0.01850 (7) | 0.02494 (7) | 0.02294 (7) | −0.00157 (8) | 0.00521 (5) | 0.00297 (7) |
| P1 | 0.0302 (5) | 0.0239 (5) | 0.0474 (6) | 0.0000 | 0.0145 (4) | 0.0000 |
| F1 | 0.0809 (19) | 0.0820 (19) | 0.0810 (18) | −0.0331 (15) | 0.0174 (14) | −0.0387 (15) |
| F2 | 0.0484 (13) | 0.0881 (18) | 0.0769 (15) | −0.0047 (12) | 0.0375 (12) | 0.0053 (14) |
| F3 | 0.095 (2) | 0.084 (2) | 0.127 (2) | −0.0475 (17) | 0.0597 (19) | −0.0657 (18) |
| O1 | 0.0204 (9) | 0.0301 (10) | 0.0288 (9) | −0.0027 (7) | 0.0066 (7) | 0.0020 (8) |
| N1 | 0.0213 (11) | 0.0257 (10) | 0.0284 (11) | 0.0004 (9) | 0.0062 (9) | 0.0022 (9) |
| N2 | 0.0233 (11) | 0.0277 (11) | 0.0229 (10) | −0.0007 (9) | 0.0065 (8) | 0.0027 (8) |
| C1 | 0.0310 (13) | 0.0327 (16) | 0.0304 (13) | −0.0047 (12) | 0.0110 (10) | 0.0022 (12) |
| C2 | 0.0357 (15) | 0.0348 (18) | 0.0317 (13) | 0.0025 (12) | 0.0150 (11) | 0.0065 (12) |
| C3 | 0.0330 (15) | 0.0319 (15) | 0.0272 (13) | 0.0116 (12) | 0.0084 (11) | 0.0046 (11) |
| C4 | 0.0310 (15) | 0.0281 (14) | 0.0333 (14) | 0.0021 (12) | 0.0062 (11) | −0.0013 (11) |
| C5 | 0.0275 (14) | 0.0303 (14) | 0.0295 (13) | −0.0015 (11) | 0.0084 (11) | 0.0029 (11) |
| C6 | 0.063 (2) | 0.0379 (17) | 0.0343 (16) | 0.0082 (16) | 0.0204 (15) | 0.0007 (13) |
| C7 | 0.0248 (14) | 0.0338 (15) | 0.0378 (15) | −0.0013 (12) | 0.0066 (11) | 0.0063 (12) |
| C8 | 0.0242 (14) | 0.0424 (17) | 0.0413 (16) | 0.0057 (13) | 0.0062 (12) | 0.0076 (13) |
| C9 | 0.0362 (16) | 0.0335 (15) | 0.0267 (13) | 0.0085 (12) | 0.0099 (11) | 0.0030 (11) |
| C10 | 0.0362 (16) | 0.0288 (15) | 0.0425 (16) | −0.0022 (13) | 0.0106 (13) | 0.0028 (12) |
| C11 | 0.0250 (14) | 0.0326 (15) | 0.0344 (15) | −0.0008 (12) | 0.0073 (11) | 0.0039 (12) |
| C12 | 0.0445 (19) | 0.0348 (17) | 0.0448 (18) | 0.0137 (14) | 0.0135 (14) | 0.0044 (14) |

*Geometric parameters (Å, °)*

| | | | |
|---|---|---|---|
| Re1—O1 | 1.7688 (19) | C4—C5 | 1.382 (4) |
| Re1—O1$^{i}$ | 1.7688 (19) | C7—C8 | 1.377 (4) |
| Re1—N1 | 2.147 (2) | C8—C9 | 1.386 (4) |
| Re1—N1$^{i}$ | 2.147 (2) | C9—C10 | 1.394 (4) |
| Re1—N2 | 2.146 (2) | C9—C12 | 1.502 (4) |
| Re1—N2$^{i}$ | 2.146 (2) | C10—C11 | 1.379 (4) |
| P1—F1 | 1.581 (2) | C1—H1 | 0.950 |
| P1—F1$^{ii}$ | 1.581 (2) | C2—H2 | 0.950 |
| P1—F2 | 1.593 (2) | C4—H4 | 0.950 |

| | | | |
|---|---|---|---|
| P1—F2$^{ii}$ | 1.593 (2) | C5—H5 | 0.950 |
| P1—F3 | 1.579 (3) | C6—H6A | 0.980 |
| P1—F3$^{ii}$ | 1.579 (3) | C6—H6B | 0.980 |
| N1—C1 | 1.351 (4) | C6—H6C | 0.980 |
| N1—C5 | 1.352 (3) | C7—H7 | 0.950 |
| N2—C7 | 1.352 (3) | C8—H8 | 0.950 |
| N2—C11 | 1.348 (3) | C10—H10 | 0.950 |
| C1—C2 | 1.386 (4) | C11—H11 | 0.950 |
| C2—C3 | 1.390 (3) | C12—H12A | 0.980 |
| C3—C4 | 1.384 (4) | C12—H12B | 0.980 |
| C3—C6 | 1.506 (4) | C12—H12C | 0.980 |

| | | | |
|---|---|---|---|
| O1—Re1—O1$^{i}$ | 180.00 (12) | C2—C3—C6 | 122.2 (3) |
| O1—Re1—N1 | 90.33 (9) | C4—C3—C6 | 121.1 (2) |
| O1—Re1—N1$^{i}$ | 89.67 (9) | C3—C4—C5 | 120.3 (2) |
| O1—Re1—N2 | 90.35 (8) | N1—C5—C4 | 122.9 (2) |
| O1—Re1—N2$^{i}$ | 89.65 (8) | N2—C7—C8 | 122.8 (2) |
| O1$^{i}$—Re1—N1 | 89.67 (9) | C7—C8—C9 | 120.7 (2) |
| O1$^{i}$—Re1—N1$^{i}$ | 90.33 (9) | C8—C9—C10 | 116.2 (2) |
| O1$^{i}$—Re1—N2 | 89.65 (8) | C8—C9—C12 | 121.6 (2) |
| O1$^{i}$—Re1—N2$^{i}$ | 90.35 (8) | C10—C9—C12 | 122.2 (2) |
| N1—Re1—N1$^{i}$ | 180.00 (12) | C9—C10—C11 | 120.7 (2) |
| N1—Re1—N2 | 90.20 (8) | N2—C11—C10 | 122.6 (2) |
| N1—Re1—N2$^{i}$ | 89.80 (8) | N1—C1—H1 | 119.0 |
| N1$^{i}$—Re1—N2 | 89.80 (8) | C2—C1—H1 | 119.0 |
| N1$^{i}$—Re1—N2$^{i}$ | 90.20 (8) | C1—C2—H2 | 119.6 |
| N2—Re1—N2$^{i}$ | 180.00 (12) | C3—C2—H2 | 119.6 |
| F1—P1—F1$^{ii}$ | 90.54 (15) | C3—C4—H4 | 119.9 |
| F1—P1—F2 | 89.65 (15) | C5—C4—H4 | 119.9 |
| F1—P1—F2$^{ii}$ | 90.19 (15) | N1—C5—H5 | 118.6 |
| F1—P1—F3 | 179.37 (16) | C4—C5—H5 | 118.6 |
| F1—P1—F3$^{ii}$ | 90.02 (14) | C3—C6—H6A | 109.5 |
| F1$^{ii}$—P1—F2 | 90.19 (15) | C3—C6—H6B | 109.5 |
| F1$^{ii}$—P1—F2$^{ii}$ | 89.65 (15) | C3—C6—H6C | 109.5 |
| F1$^{ii}$—P1—F3 | 90.02 (14) | H6A—C6—H6B | 109.5 |
| FP$^{ii}$—P1—F3$^{ii}$ | 179.37 (14) | H6A—C6—H6C | 109.5 |
| F2—P1—F2$^{ii}$ | 179.78 (16) | H6B—C6—H6C | 109.5 |
| F2—P1—F3 | 90.06 (16) | N2—C7—H7 | 118.6 |
| F2—P1—F3$^{ii}$ | 90.10 (16) | C8—C7—H7 | 118.6 |
| F2$^{ii}$—P1—F3 | 90.10 (16) | C7—C8—H8 | 119.6 |
| F2$^{ii}$—P1—F3$^{ii}$ | 90.06 (16) | C9—C8—H8 | 119.6 |
| F3—P1—F3$^{ii}$ | 89.41 (16) | C9—C10—H10 | 119.6 |
| Re1—N1—C1 | 121.74 (18) | C11—C10—H10 | 119.6 |
| Re1—N1—C5 | 120.9 (2) | N2—C11—H11 | 118.7 |

| | | | |
|---|---|---|---|
| C1—N1—C5 | 117.3 (2) | C10—C11—H11 | 118.7 |
| Re1—N2—C7 | 121.15 (19) | C9—C12—H12A | 109.5 |
| Re1—N2—C11 | 121.84 (18) | C9—C12—H12B | 109.5 |
| C7—N2—C11 | 116.9 (2) | C9—C12—H12C | 109.5 |
| N1—C1—C2 | 122.1 (2) | H12A—C12—H12B | 109.5 |
| C1—C2—C3 | 120.8 (3) | H12A—C12—H12C | 109.5 |
| C2—C3—C4 | 116.7 (2) | H12B—C12—H12C | 109.5 |

Symmetry codes: (i) $-x+3/2$, $-y+1/2$, $-z+1$; (ii) $-x+1$, $y$, $-z+3/2$.

Fig. 1

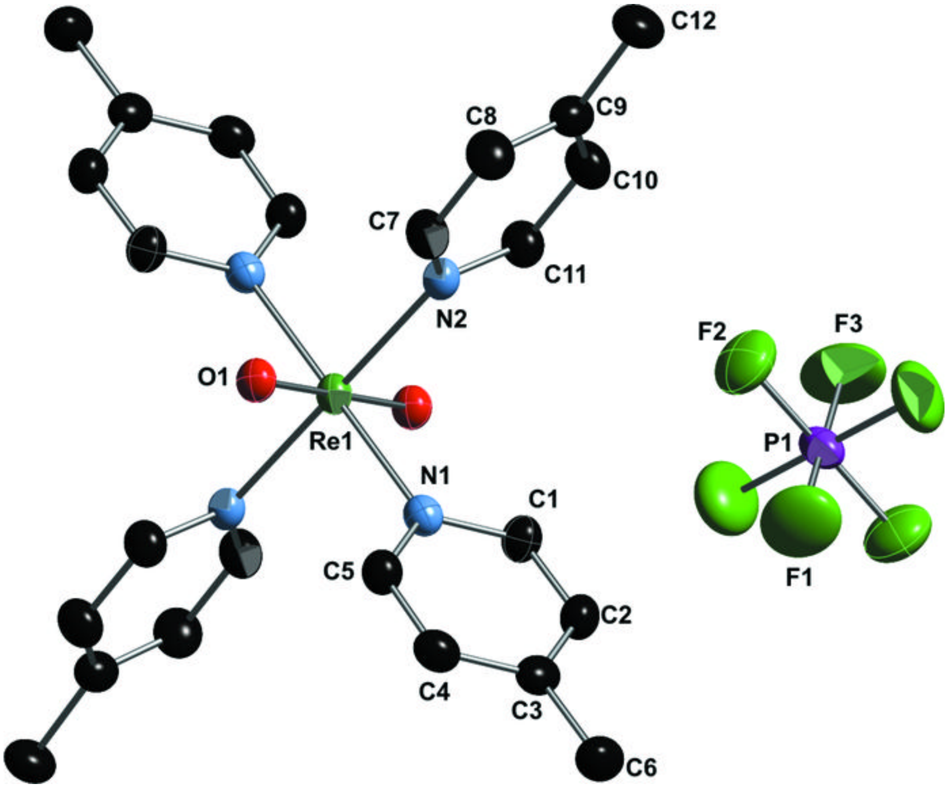

**Fig. 2**