

trans-(2,2'-Bipyrimidine)diiodido(iso-propoxido)oxidorhenium(V)

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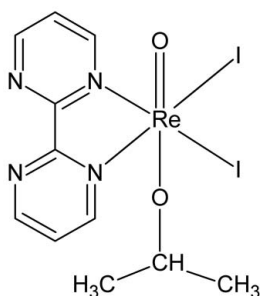
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 Key indicators: single-crystal X-ray study; $T = 100$ K; mean $\sigma(\text{C}-\text{C}) = 0.010$ Å; R factor = 0.029; wR factor = 0.056; data-to-parameter ratio = 15.2.

In the title compound, $[\text{Re}(\text{C}_3\text{H}_7\text{O})\text{I}_2\text{O}(\text{C}_8\text{H}_6\text{N}_4)]$, the Re^V atom adopts a distorted octahedral $\text{ReI}_2\text{O}_2\text{N}_2$ geometry, with the O atoms in a *trans* conformation and the I atoms in a *cis* conformation. Two intramolecular C—H···I contacts occur. The crystal structure is stabilized by intermolecular C—H···O, C—H···N and C—H···I hydrogen bonds.

Related literature

For related structures and for further discussion of rhenium structural chemistry, see: Abrahams *et al.* (2005, 2007); Abram *et al.* (1995); Ciani *et al.* (1983); Gerber *et al.* (2004). Graziani *et al.* (1985); Herrman *et al.* (1990); Irmiler *et al.* (1991); Lebuis *et al.* (1993); Mrozinski *et al.* (2002); Quintal *et al.* (2000); Schmidt-Brucken & Abram (2000). For further synthetic details, see: Watt & Thompson (1963).



Experimental

Crystal data

 $[\text{Re}(\text{C}_3\text{H}_7\text{O})\text{I}_2\text{O}(\text{C}_8\text{H}_6\text{N}_4)]$
 $M_r = 673.26$

 Monoclinic, $P2_1/c$
 $a = 10.3973$ (3) Å

 $b = 10.9046$ (3) Å

 $c = 15.6341$ (6) Å

 $\beta = 117.616$ (2)°

 $V = 1570.63$ (9) Å³
 $Z = 4$

 Mo $K\alpha$ radiation

 $\mu = 11.67$ mm⁻¹
 $T = 100$ K

 $0.12 \times 0.11 \times 0.03$ mm

Data collection

Oxford Diffraction KM-4-CCD diffractometer

 Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2007)

 $T_{\min} = 0.456$, $T_{\max} = 0.611$

15982 measured reflections

2776 independent reflections

 2288 reflections with $I > 2\sigma(I)$
 $R_{\text{int}} = 0.050$

Refinement

 $R[F^2 > 2\sigma(F^2)] = 0.029$
 $wR(F^2) = 0.056$
 $S = 1.03$

2776 reflections

183 parameters

H-atom parameters constrained

 $\Delta\rho_{\max} = 1.85$ e Å⁻³
 $\Delta\rho_{\min} = -1.34$ e Å⁻³
Table 1

Selected geometric parameters (Å, °).

Re1—O1	1.698 (4)	Re1—N2	2.183 (6)
Re1—O2	1.861 (5)	Re1—I1	2.7176 (5)
Re1—N1	2.175 (5)	Re1—I2	2.7235 (5)
N1—Re1—N2	76.5 (2)		

Table 2

Hydrogen-bond geometry (Å, °).

<i>D</i> —H··· <i>A</i>	<i>D</i> —H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> —H··· <i>A</i>
C1—H1···I1	0.95	3.04	3.697 (7)	127
C6—H6···I2	0.95	3.05	3.709 (7)	128
C5—H5···N4 ⁱ	0.95	2.58	3.504 (9)	163
C6—H6···O1 ⁱⁱ	0.95	2.53	3.193 (9)	127
C9—H9···I1 ⁱⁱⁱ	1.00	3.02	3.834 (8)	139

 Symmetry codes: (i) $-x + 1, y - \frac{1}{2}, -z + \frac{1}{2}$; (ii) $-x + 1, -y - 1, -z$; (iii) $x, -y - \frac{1}{2}, z + \frac{1}{2}$.

Data collection: *CrysAlis CCD* (Oxford Diffraction, 2007); cell refinement: *CrysAlis RED* (Oxford Diffraction, 2007); data reduction: *CrysAlis RED*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg & Putz, 2005); software used to prepare material for publication: *SHELXL97*.

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: HB5231).

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supplementary materials

Acta Cryst. (2010). E66, m42-m43 [doi:10.1107/S1600536809052556]

***trans*-(2,2'-Bipyrimidine)diiodido(isopropoxido)oxidorhenium(V)**

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Comment

Re^V complexes have been research object for many authors during the last years. Complexes of general formula [ReOX₂(C₅H₄N)CH(O)CH₂(C₅H₄N)] (*X* = Cl, I) were obtained by a reaction of *trans*-[ReOCl₃(PPh₃)₂] and *trans*-[ReOI₂(OEt)(PPh₃)₂] with *cis*-1,2-di-(2-pyridyl)ethylene (DPE) in ethanol and in benzene on air. The coordinated DPE ligand undergoes addition of water at the ethylene carbon atoms, and the (C₅H₄N)CH(O)CH₂(C₅H₄N) moiety acts as terdentate *N,O,N*-donor ligand. X-ray crystal structures of both complexes have been determined and show distorted octahedral geometry around the rhenium(V) centre (Abrahams *et al.*, 2005). Complexes *cis*-[ReOX₂(msa)(PPh₃)₂] [*X* = Cl(1), I(2)] were prepared from *trans*-[ReOCl₃(PPh₃)₂] or *trans*-[ReOI₂(OEt)(PPh₃)₂] in reaction with 2-(1-iminoethyl)phenol (Hmsa) in acetonitrile. X-ray crystal structure shows, that the bonding distances and angles in 1 and 2 are nearly identical, and that the two halide ligands in each complex are coordinated *cis* to each other in the equatorial plane *cis* to the oxido group. Rhenium(V) complexes with two iodido ligands *cis* to each other are rare (Abrahams *et al.*, 2007). Rhenium(V) complexes with *trans*-O—Re-(2-propoxido) core are known. Rhenium(V) complex with 2-propoxido ligand of formula [ReOCl₂(C₃H₇O)(Ph₃P)₂] has been synthesized (Abram *et al.* 1995), and the other one, [ReClO(C₃H₇O)PPh₃L] (where *L* = pyridine-2-thiolato ligand), was obtained (Schmidt-Brucken & Abram, 2000). This paper contains a report on the synthesis and structure of the first characterized [ReOI₂(C₃H₇O)(2,2'-bipyrimidine)] 1 complex with ReI₂ core and 2-propoxido ligand. Fig. 1 presents the view of molecular structure of compound 1, the compound crystallizes in monoclinic crystal system in *P*2₁/*c* space group. The compound was obtained in the synthesis with (NH₄)₂ReI₆ and 2,2'-bipyrimidine as substrates in a mixture of 2-propanol and acetone. The environment around the metal center is a distorted octahedron, with two iodido ligands, 2-propoxido ligand and 2,2'-bipyrimidine ligand coordinated *via* nitrogen atoms. The Re—I bond lengths are Re—I1 2.7176 (5) Å, Re—I2 2.7235 (5) Å, respectively, similar Re—I distances were observed in 2-(2-aminophenyl)ethanolato-*N,O*-bis(iodido)-oxido-triphenylphosphine-rhenium(V) and diiodido-(3-hydroxidopicolinato)-oxido-triphenylphosphine-rhenium(II) (Gerber *et al.*, 2004, Quintal *et al.*, 2000). On the other hand, the Re—I—O1 1.698 (4) Å bond length is characteristic for monooxido-rhenium(V) complexes (on average 1.69%Å), which is in agreement with the situation in the comparable complexes (Graziani *et al.*, 1985, Lebuis *et al.*, 1993). The O atom from 2-propoxido ligand is coordinated to the rhenium atom with Re—O2 1.861 (5) Å; this bond is remarkably short with the same multiple bond character as discussed (Ciani *et al.* 1983). The O—Re—O unit is nearly linear with an angle of 169.69 (3)°. Moreover, a molecule of 2,2'-bipyrimidine is coordinated with Re—N bond length of Re—I—N1 2.175 (5) Å, Re—I—N2 2.183 (6) Å, which is comparable to the previously investigated *trans*-[ReCl₄(py)₂] (Mrozinski *et al.*, 2002, Herrman *et al.*, 1990). Re atom lies within the plane of I1, I2, N1, N2 atoms, the N1—Re—I—O1 and N2—Re—I—O1 angles are 88.63 (19), 86.93 (14) and N1—Re—I—O2, N2—Re—I—O2 83.37 (14) and 84.87 (15) and are directed in side to 2,2'-bipyrimidine. The rings of the 4,4'-bpy ligand are coplanar with the plane defined by the I ligands, one of the Re—N bond is shorter (Re—I—N1 2.175 (5) Å, Re—I—N2 2.183 (6) Å), which may be a result of iodido ligand presence. The molecule conformation is stabilized by intramolecular hydrogen bonds C(1)—H(1)⋯I(1) and C(6)—H(6)⋯I(2), as well as by the intermolecular hydrogen bonds of C—H⋯O and C—H⋯N type. All hydrogen bonds are summarized in Table 2. In the crystal structure packing along [100] and [010] a layered arrangement of the molecules could be observed. The crystal structure packing viewed along [100] direction is illustrated in Fig. 2. In the structure the

stacking interactions are observed. pi-pi stacking interactions between the 2,2'-bipyrimidine rings contribute to forming a supramolecular network structure (Fig. 2). The centroid-centroid distance of the adjacent aromatic rings is about 3.58 Å, indicating a normal pi-pi interaction. The corresponding TG-DTA curves (the measurement was carried out under nitrogen atmosphere) for the title compound (Fig. 3) show a three-step decomposition process and that the compound is very stable during heating. In the first step of the thermal decomposition process weight loss in the temperature range 242–247°C of 1.39% (calc. 1.43%) is observed. In DTA curve, this decomposition is visible as an endothermic peak at temperature 247°C, indicating the presence of coordinated 4,4'-bipyrimidine (corresponding to the weight loss of 1.39% in thermal decomposition, which is in agreement with the calculated value of 1.43%). Further weight losses correspond to decomposition of further compound parts, they are continuous and are difficult for unambiguous interpretation (decomposition of all compound constituents except for rhenium part). Another peak at 800°C is also observed, however, it is difficult to interpret. It is possible, that the decomposition process leads to rhenium oxides. Similar decomposition process through many intermediate stages has been observed for $(\text{NH}_4)_2[\text{Re}_3\text{Cl}_{12}]$ (Irmiler *et al.*, 1991). In conclusion, an interesting rhenium(V) *trans*-2,2'-bipyrimidine-diiodido-oxido-(2-propoxido)-rhenium(V) complex with 2-propoxido ligand was obtained. The crystal structure is stabilized by hydrogen bonding interactions. This is the first structural report confirming the existence of a iodido rhenium complex with an aromatic amine and 2-propoxido ligand in coordination sphere.

Experimental

A mixture of $(\text{NH}_4)_2[\text{ReI}_6]$ (prepared according to the literature procedure (Watt & Thompson, 1963) (0.12 g) and 0.30 g of 2,2'-(bipyrimidine) (from Aldrich) was added to a mixture of 2-propanol/acetone (1:1) (50 cm³) and then was stirred at 40° for about 4 h, the color of the solution was dark green. The solution was left for slow crystallization by evaporation under parafilm. The mixture was kept at room temperature for crystallization. After five days the pale green plate-shaped crystals were obtained. Anal. Calc. For: $[\text{ReOI}_2(\text{C}_3\text{H}_7\text{O})(2,2\text{-bipyrimidine})]$ C 19.62, H 2.08, N 8.32, I 37.69%; found C 19.01, H 1.89, N 8.15, I 37.50%. Selected IR data (KBr): 2925 (m), 2854 (m), 1717 (m), 1631 (m), 1610 (w), 1558 (s), 1464 (m), 1406 (s), 1378 (m), 1271 (w), 1193 (w), 1191 (w), 978 (w), 908(w), 807 (w), 645 (m), 561 (w), 535 (w), 452 (s), 390 (w), 358(w), 315 (s), 162 (*versus*), 131 (s), 74 (w).

TG–DTA Thermogravimetric measurements were carried out using a TG–DTA SETSYS 16Y18 device under nitrogen atmosphere for a sample placed in Al₂O₃ crucible. The investigated temperature range was from room temperature to 1400°C at 10°C/min. temperature changes rate. IR spectra The room temperature FT–IR spectra of polycrystalline samples were measured by means of the Bruker IFS-66 instrument.

Refinement

The H atoms were generated geometrically and refined using a riding model, with C—H = 0.95 Å and $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$. The highest peak of 0.82 electrons at the difference Fourier map was situated near the Re atom.

Figures

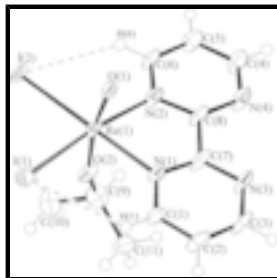


Fig. 1. View of (I) showing 50% displacement ellipsoids (H atoms as spheres of arbitrary radius).

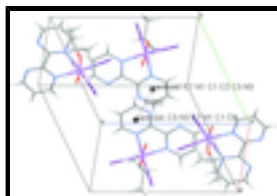


Fig. 2. The crystal packing in (I) viewed down [100].

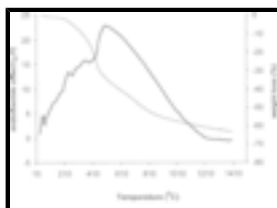


Fig. 3. Corresponding TG/DTA curves for 1. The thicker line denotes the thermal effect, the thinner line denotes the weight loss curve.

***trans*-(2,2'-Bipyrimidine)diiodido(isopropoxido)oxidorhenium(V)**

Crystal data

[Re(C₃H₇O)I₂O(C₈H₆N₄)]

$M_r = 673.26$

Monoclinic, $P2_1/c$

Hall symbol: -P 2ybc

$a = 10.3973$ (3) Å

$b = 10.9046$ (3) Å

$c = 15.6341$ (6) Å

$\beta = 117.616$ (2)°

$V = 1570.63$ (9) Å³

$Z = 4$

$F(000) = 1216$

$D_x = 2.847$ Mg m⁻³

Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å

Cell parameters from 2115 reflections

$\theta = 2.9$ – 25.1 °

$\mu = 11.67$ mm⁻¹

$T = 100$ K

Plate, pale green

$0.12 \times 0.11 \times 0.03$ mm

Data collection

Oxford Diffraction KM-4-CCD diffractometer

Radiation source: fine-focus sealed tube graphite

/w scans

Absorption correction: analytical (*CrysAlis RED*; Oxford Diffraction, 2007)

2776 independent reflections

2288 reflections with $I > 2\sigma(I)$

$R_{int} = 0.050$

$\theta_{max} = 25.1$ °, $\theta_{min} = 2.9$ °

$h = -12 \rightarrow 12$

supplementary materials

$T_{\min} = 0.456$, $T_{\max} = 0.611$
15982 measured reflections

$k = -11 \rightarrow 12$
 $l = -18 \rightarrow 18$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.029$

$wR(F^2) = 0.056$

$S = 1.02$

2776 reflections

183 parameters

0 restraints

Primary atom site location: structure-invariant direct methods

Secondary atom site location: difference Fourier map

Hydrogen site location: inferred from neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0251P)^2 + 3.8665P]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.85 \text{ e } \text{\AA}^{-3}$

$\Delta\rho_{\min} = -1.34 \text{ e } \text{\AA}^{-3}$

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 (\AA^2)

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Re1	0.26746 (3)	-0.33250 (2)	-0.10973 (2)	0.02512 (9)
I1	0.13432 (6)	-0.31460 (4)	-0.30538 (3)	0.03659 (14)
I2	0.22482 (5)	-0.57955 (4)	-0.12777 (3)	0.03021 (13)
O1	0.4380 (5)	-0.3317 (4)	-0.0984 (3)	0.0228 (10)
O2	0.0941 (5)	-0.3139 (4)	-0.1035 (3)	0.0317 (11)
N1	0.2890 (6)	-0.1369 (5)	-0.0790 (4)	0.0228 (12)
N2	0.3689 (7)	-0.3247 (5)	0.0476 (4)	0.0281 (13)
N3	0.3837 (7)	0.0063 (5)	0.0504 (4)	0.0293 (14)
N4	0.4828 (7)	-0.1874 (5)	0.1792 (4)	0.0316 (15)
C1	0.2343 (8)	-0.0453 (6)	-0.1436 (5)	0.0324 (17)
H1	0.1832	-0.0636	-0.2105	0.039*
C2	0.2519 (9)	0.0735 (6)	-0.1134 (5)	0.0365 (19)
H2	0.2114	0.1388	-0.1583	0.044*
C3	0.3303 (8)	0.0959 (6)	-0.0157 (5)	0.0328 (18)
H3	0.3473	0.1786	0.0057	0.039*
C4	0.5150 (9)	-0.2833 (6)	0.2389 (5)	0.0342 (18)

H4	0.5655	-0.2690	0.3065	0.041*
C5	0.4775 (8)	-0.4035 (6)	0.2062 (5)	0.0320 (17)
H5	0.5002	-0.4704	0.2498	0.038*
C6	0.4065 (8)	-0.4201 (6)	0.1084 (5)	0.0273 (16)
H6	0.3834	-0.5009	0.0830	0.033*
C7	0.3584 (8)	-0.1071 (6)	0.0158 (5)	0.0291 (17)
C8	0.4078 (8)	-0.2118 (6)	0.0862 (5)	0.0261 (16)
C9	-0.0043 (9)	-0.2989 (8)	-0.0654 (6)	0.045 (2)
H9	0.0494	-0.3159	0.0055	0.054*
C10	-0.1237 (9)	-0.3889 (8)	-0.1089 (6)	0.043 (2)
H10A	-0.1895	-0.3648	-0.1754	0.065*
H10B	-0.1773	-0.3915	-0.0713	0.065*
H10C	-0.0834	-0.4703	-0.1087	0.065*
C11	-0.0531 (8)	-0.1652 (6)	-0.0783 (6)	0.0382 (18)
H11A	0.0309	-0.1123	-0.0416	0.057*
H11B	-0.1249	-0.1540	-0.0549	0.057*
H11C	-0.0966	-0.1434	-0.1469	0.057*

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.03734 (18)	0.00899 (14)	0.03104 (16)	0.00073 (12)	0.01756 (13)	0.00019 (11)
I1	0.0430 (3)	0.0213 (3)	0.0286 (2)	0.0025 (2)	0.0023 (2)	0.0026 (2)
I2	0.0453 (3)	0.0095 (2)	0.0337 (3)	-0.00078 (19)	0.0165 (2)	-0.00140 (18)
O1	0.031 (3)	0.015 (2)	0.022 (2)	0.0001 (19)	0.011 (2)	-0.0019 (19)
O2	0.041 (3)	0.016 (2)	0.041 (3)	0.002 (2)	0.022 (3)	-0.001 (2)
N1	0.030 (3)	0.013 (3)	0.028 (3)	0.001 (2)	0.016 (3)	0.000 (2)
N2	0.045 (4)	0.012 (3)	0.034 (3)	0.001 (3)	0.025 (3)	0.001 (3)
N3	0.056 (4)	0.012 (3)	0.036 (3)	-0.005 (3)	0.035 (3)	-0.004 (3)
N4	0.065 (4)	0.018 (3)	0.027 (3)	-0.003 (3)	0.034 (3)	-0.001 (2)
C1	0.049 (5)	0.016 (4)	0.029 (4)	0.008 (3)	0.015 (4)	0.002 (3)
C2	0.062 (5)	0.011 (4)	0.045 (5)	0.005 (4)	0.033 (4)	0.009 (3)
C3	0.061 (5)	0.006 (3)	0.050 (5)	-0.002 (3)	0.042 (4)	-0.002 (3)
C4	0.065 (6)	0.021 (4)	0.025 (4)	-0.001 (4)	0.028 (4)	0.000 (3)
C5	0.055 (5)	0.024 (4)	0.030 (4)	0.004 (3)	0.030 (4)	0.001 (3)
C6	0.043 (5)	0.010 (3)	0.038 (4)	-0.002 (3)	0.026 (4)	-0.003 (3)
C7	0.048 (5)	0.011 (4)	0.044 (4)	-0.002 (3)	0.034 (4)	-0.004 (3)
C8	0.045 (5)	0.010 (3)	0.037 (4)	-0.001 (3)	0.030 (4)	-0.001 (3)
C9	0.042 (5)	0.045 (5)	0.039 (4)	0.004 (4)	0.013 (4)	-0.004 (4)
C10	0.041 (5)	0.050 (5)	0.039 (4)	-0.010 (4)	0.019 (4)	-0.009 (4)
C11	0.034 (4)	0.026 (4)	0.051 (5)	0.007 (3)	0.016 (4)	0.001 (4)

Geometric parameters (\AA , $^\circ$)

Re1—O1	1.698 (4)	C2—H2	0.9500
Re1—O2	1.861 (5)	C3—H3	0.9500
Re1—N1	2.175 (5)	C4—C5	1.395 (10)
Re1—N2	2.183 (6)	C4—H4	0.9500
Re1—I1	2.7176 (5)	C5—C6	1.368 (10)

supplementary materials

Re1—I2	2.7235 (5)	C5—H5	0.9500
O2—C9	1.411 (10)	C6—H6	0.9500
N1—C1	1.345 (8)	C7—C8	1.502 (10)
N1—C7	1.353 (9)	C9—C10	1.478 (11)
N2—C6	1.339 (8)	C9—C11	1.526 (10)
N2—C8	1.348 (8)	C9—H9	1.0000
N3—C7	1.326 (8)	C10—H10A	0.9800
N3—C3	1.341 (9)	C10—H10B	0.9800
N4—C8	1.320 (9)	C10—H10C	0.9800
N4—C4	1.337 (9)	C11—H11A	0.9800
C1—C2	1.361 (10)	C11—H11B	0.9800
C1—H1	0.9500	C11—H11C	0.9800
C2—C3	1.380 (10)		
O1—Re1—O2	169.68 (19)	N4—C4—C5	122.8 (6)
O1—Re1—N1	88.66 (19)	N4—C4—H4	118.6
O2—Re1—N1	83.34 (19)	C5—C4—H4	118.6
O1—Re1—N2	86.9 (2)	C6—C5—C4	116.6 (6)
O2—Re1—N2	84.9 (2)	C6—C5—H5	121.7
N1—Re1—N2	76.5 (2)	C4—C5—H5	121.7
O1—Re1—I1	94.56 (13)	N2—C6—C5	121.3 (6)
O2—Re1—I1	92.91 (15)	N2—C6—H6	119.4
N1—Re1—I1	97.18 (14)	C5—C6—H6	119.4
N2—Re1—I1	173.47 (14)	N3—C7—N1	125.1 (6)
O1—Re1—I2	97.35 (14)	N3—C7—C8	118.3 (6)
O2—Re1—I2	89.91 (13)	N1—C7—C8	116.6 (6)
N1—Re1—I2	171.11 (14)	N4—C8—N2	125.3 (6)
N2—Re1—I2	97.22 (14)	N4—C8—C7	118.7 (6)
I1—Re1—I2	88.908 (16)	N2—C8—C7	115.9 (6)
C9—O2—Re1	160.7 (5)	O2—C9—C10	110.3 (6)
C1—N1—C7	118.0 (5)	O2—C9—C11	108.4 (7)
C1—N1—Re1	126.7 (4)	C10—C9—C11	114.6 (7)
C7—N1—Re1	115.2 (4)	O2—C9—H9	107.7
C6—N2—C8	117.7 (6)	C10—C9—H9	107.7
C6—N2—Re1	126.8 (4)	C11—C9—H9	107.7
C8—N2—Re1	115.3 (4)	C9—C10—H10A	109.5
C7—N3—C3	115.6 (6)	C9—C10—H10B	109.5
C8—N4—C4	116.2 (6)	H10A—C10—H10B	109.5
N1—C1—C2	120.3 (7)	C9—C10—H10C	109.5
N1—C1—H1	119.8	H10A—C10—H10C	109.5
C2—C1—H1	119.8	H10B—C10—H10C	109.5
C1—C2—C3	117.9 (7)	C9—C11—H11A	109.5
C1—C2—H2	121.1	C9—C11—H11B	109.5
C3—C2—H2	121.1	H11A—C11—H11B	109.5
N3—C3—C2	123.1 (6)	C9—C11—H11C	109.5
N3—C3—H3	118.5	H11A—C11—H11C	109.5
C2—C3—H3	118.5	H11B—C11—H11C	109.5
O1—Re1—O2—C9	-37 (2)	C1—C2—C3—N3	-3.1 (11)
N1—Re1—O2—C9	-76.5 (14)	C8—N4—C4—C5	2.4 (11)

N2—Re1—O2—C9	0.5 (14)	N4—C4—C5—C6	0.8 (11)
I1—Re1—O2—C9	-173.4 (14)	C8—N2—C6—C5	2.2 (10)
I2—Re1—O2—C9	97.7 (14)	Re1—N2—C6—C5	176.0 (5)
O1—Re1—N1—C1	100.5 (6)	C4—C5—C6—N2	-3.2 (10)
O2—Re1—N1—C1	-86.0 (6)	C3—N3—C7—N1	2.4 (10)
N2—Re1—N1—C1	-172.3 (6)	C3—N3—C7—C8	-176.6 (6)
I1—Re1—N1—C1	6.1 (6)	C1—N1—C7—N3	-3.6 (10)
O1—Re1—N1—C7	-83.2 (5)	Re1—N1—C7—N3	179.7 (5)
O2—Re1—N1—C7	90.2 (5)	C1—N1—C7—C8	175.4 (6)
N2—Re1—N1—C7	3.9 (4)	Re1—N1—C7—C8	-1.3 (7)
I1—Re1—N1—C7	-177.7 (4)	C4—N4—C8—N2	-3.7 (11)
O1—Re1—N2—C6	-91.0 (6)	C4—N4—C8—C7	175.4 (6)
O2—Re1—N2—C6	95.2 (6)	C6—N2—C8—N4	1.5 (11)
N1—Re1—N2—C6	179.6 (6)	Re1—N2—C8—N4	-173.1 (6)
I2—Re1—N2—C6	6.0 (6)	C6—N2—C8—C7	-177.6 (6)
O1—Re1—N2—C8	83.0 (5)	Re1—N2—C8—C7	7.8 (8)
O2—Re1—N2—C8	-90.8 (5)	N3—C7—C8—N4	-4.5 (10)
N1—Re1—N2—C8	-6.4 (5)	N1—C7—C8—N4	176.4 (6)
I2—Re1—N2—C8	180.0 (5)	N3—C7—C8—N2	174.7 (6)
C7—N1—C1—C2	1.3 (11)	N1—C7—C8—N2	-4.4 (9)
Re1—N1—C1—C2	177.5 (5)	Re1—O2—C9—C10	-130.8 (12)
N1—C1—C2—C3	1.8 (11)	Re1—O2—C9—C11	102.9 (14)
C7—N3—C3—C2	1.1 (10)		

Hydrogen-bond geometry (Å, °)

<i>D</i> —H \cdots <i>A</i>	<i>D</i> —H	H \cdots <i>A</i>	<i>D</i> \cdots <i>A</i>	<i>D</i> —H \cdots <i>A</i>
C1—H1 \cdots I1	0.95	3.04	3.697 (7)	127
C6—H6 \cdots I2	0.95	3.05	3.709 (7)	128
C5—H5 \cdots N4 ⁱ	0.95	2.58	3.504 (9)	163
C6—H6 \cdots O1 ⁱⁱ	0.95	2.53	3.193 (9)	127
C9—H9 \cdots I1 ⁱⁱⁱ	1.00	3.02	3.834 (8)	139

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

Fig. 1

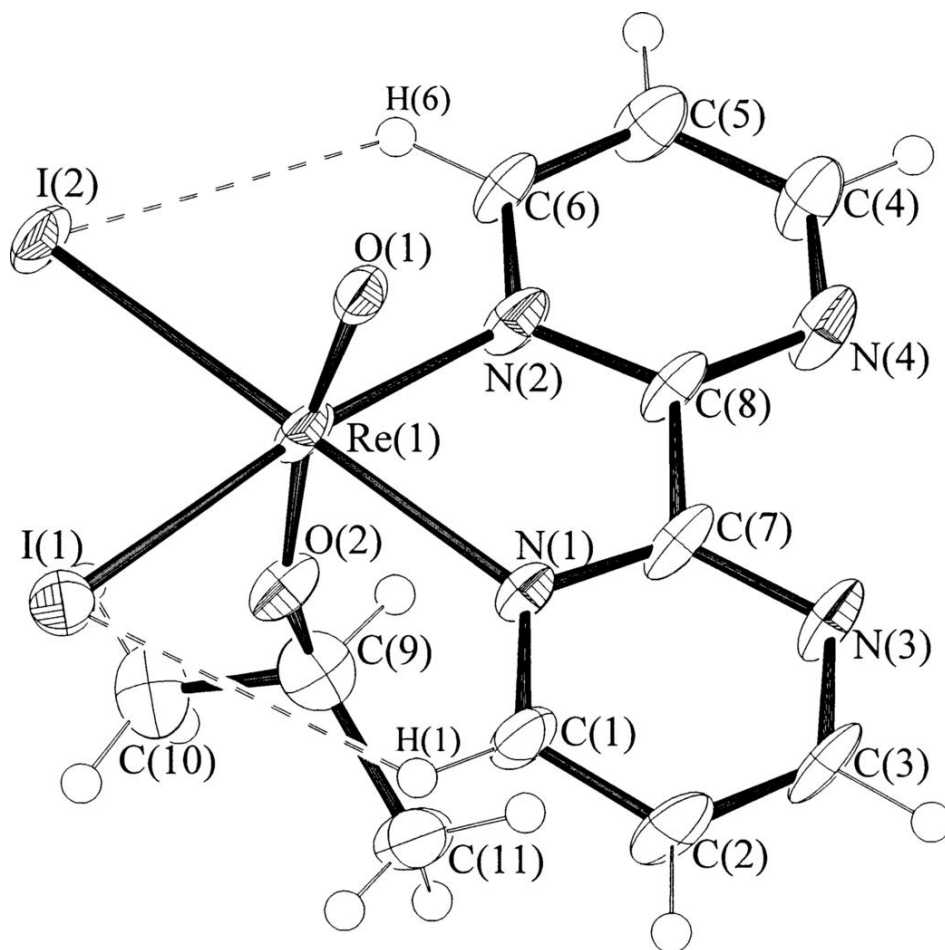


Fig. 2

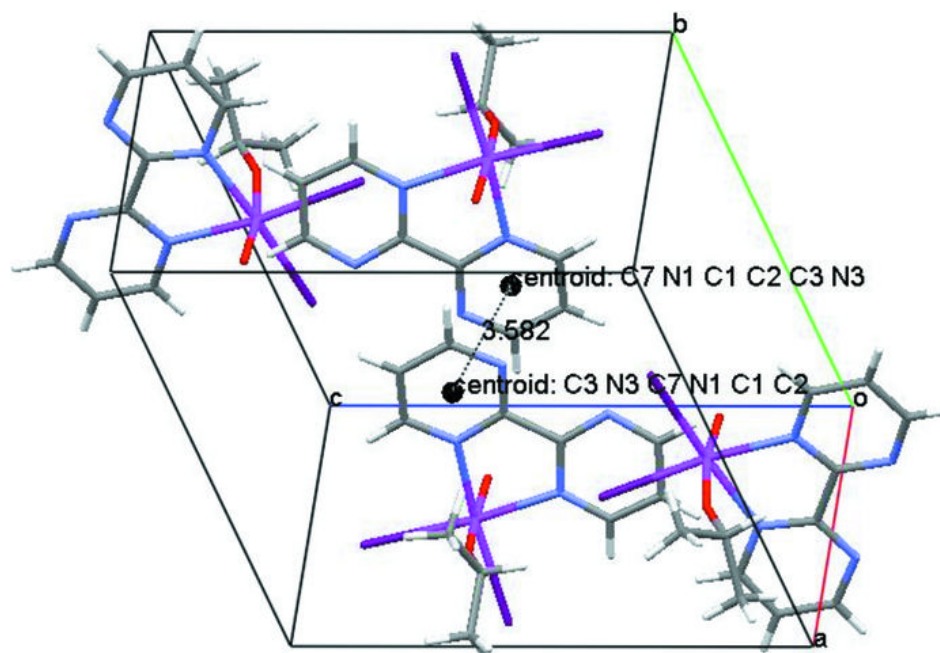


Fig. 3

