Acta Crystallographica Section E
Structure Reports
Online
ISSN 1600-5368

1,3-Bis(2,4,6-trimethylphenyl)-3*H*-imidazol-1-ium tetraoxidorhenate(VII)

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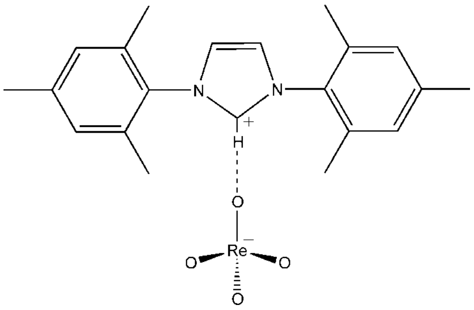
Received 10 November 2011; accepted 25 November 2011

Key indicators: single-crystal X-ray study; T = 293 K; mean σ(C–C) = 0.006 Å; R factor = 0.033; wR factor = 0.083; data-to-parameter ratio = 15.1.

The title compound, $(C_{21}H_{25}N_2)[ReO_4]$, was formed as the unexpected product in an attempted synthesis of a rhenium(I)–*N*-heterocyclic carbene (NHC) complex. The compound has crystallographic mirror symmetry with both the cation and the tetrahedral anion located across a mirror plane. The cation and anion are linked by a C—H···O hydrogen bond.

Related literature

For related structures of some halide salts, see: Arduengo *et al.* (1995); Cole *et al.* (2002); Cole & Junk (2004); Lorber & Vendier (2009).



Experimental

Crystal data

$(C_{21}H_{25}N_2)[ReO_4]$
M_r = 555.63
Monoclinic, $P2_1/m$
a = 8.2989 (12) Å
b = 16.373 (2) Å
c = 8.3168 (12) Å
β = 111.948 (2)°
V = 1048.2 (3) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 5.83 mm^{-1}
T = 293 K
0.39 × 0.10 × 0.09 mm

Data collection

Bruker (Siemens) P4 diffractometer with a Bruker SMART 1000 CCD detector
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
T_{min} = 0.489, T_{max} = 0.592
5688 measured reflections
2056 independent reflections
1895 reflections with $I > 2\sigma(I)$
R_{int} = 0.029

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.033
$wR(F^2)$ = 0.083
S = 1.13
2056 reflections
136 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.32 e Å^{-3}
$\Delta\rho_{min}$ = −0.86 e Å^{-3}

Table 1
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
C1—H1···O2^{i}	0.93	2.21	3.12 (1)	167 (1)

Symmetry code: (i) $x - 1, y, z$.

Data collection: *SMART* (Bruker, 2001); cell refinement: *SAINT* (Bruker, 2001); data reduction: *SAINT*; program(s) used to solve structure: *SHELXTL* (Sheldrick, 2008); program(s) used to refine structure: *SHELXTL*; molecular graphics: *ORTEP-3 for Windows* (Farrugia, 1997) and *POV-RAY* (Cason, 2004); software used to prepare material for publication: *SHELXTL* and *PLATON* (Spek, 2009).

Funding received for this work from the University of Pretoria and the National Research Foundation is acknowledged.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: EZ2273).

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supporting information

Acta Cryst. (2011). E**67**, m1897 [https://doi.org/10.1107/S1600536811050677]

1,3-Bis(2,4,6-trimethylphenyl)-3*H*-imidazol-1-ium tetraoxidorhenate(VII)

Marilé Landman, Belinda van der Westhuizen, Daniela I. Bezuidenhout and David C. Liles

S1. Comment

1,3-Bis(2,4,6-trimethylphenyl)-3*H*-imidazolium tetraoxorhenate(VII) (IMesH[ReO_4], **1**) was formed as the unexpected product in an attempted synthesis of a rhenium(I)-*N*-heterocyclic carbene (NHC) complex. The reaction pathway is unexplained. The main feature in the formation of this complex is the oxidation of the rhenium metal from the +1 to the +7 state resulting in a $[ReO_4]^-$ anion. This anion replaces the Cl^- anion in the IMesHCl salt since it is larger and the co-crystallization of more similar sized ions is favoured..

The compound has crystallographic mirror symmetry with the Re1, O1, O2 and the imidazolium C1 and H1 atoms all lying in a mirror plane. The geometry of the imidazolium cation is similar to those observed for previously reported structures of 1,3-Bis(2,4,6-trimethylphenyl)-3*H*-imidazolium salts, for example halide (Cl^-, Br^-) salts (Arduengo *et al.*, 1995; Cole *et al.*, 2002; Cole & Junk, 2004; Lorber &Vendier, 2009). The imidazolium C1—H1 is hydrogen bonded to O2 of the $[ReO_4]^-$ anion (at x - 1, y, z) with H1···O2 = 2.21 (1) Å and C1—H1···O2 = 167 (1)°. Similar hydrogen bonds were observed between the cation and the anion in the halide salts (Cole & Junk, 2004). The Re—O2 bond is somewhat longer (1.714 (5) Å) than the other (nominally double) Re—O bonds (mean 1.690 (6) Å) and may indicate some localization of the anion negative charge on O2.

S2. Experimental

N,*N*′-Dimesitylimidazolium chloride (IMesHCl, 2 mmol, 0.66 g) was deprotonated with KO^{t}Bu in tetrahydrofuran (thf) solution. This mixture was added to a thf solution of $[Re_2(CO)_{10}]$ (1.06 mmol, 0.68 g) and stirred. Thin layer chromatography (TLC) was used to monitor the reaction and, due to the initial lack of product formation, the reaction mixture was heated in an oilbath at 70°C for an hour. The thf solvent was removed leaving a brown residue. The product was extracted with hexane and the hexane was then removed under reduced pressure. The residue was purified by column chromatography. A dark yellow fraction was eluted with dichloromethane (dcm). Crystallization from a 1:1 dcm:hexane solution gave light brown crystals of IMesH[ReO_4] (**1**). 1H NMR (δ, p.p.m.), C_6D_6: 1.91 (br,12*H*), 2.19 (br, 6H), 6.70 (br, 4H), 7.14 (s, 2H); 13 C NMR (δ, p.p.m.), C_6D_6: 17.8, 20.9, 121.8, 126.9, 128.9, 129.6, 141.2

S3. Refinement

All hydrogen atoms were added in calculated positions. Each was allowed to ride on the atom to which it is bonded with an isotropic adp set to 1.2 x (1.5 x for methyl H atoms) the equivalent isotropic adp of that atom.

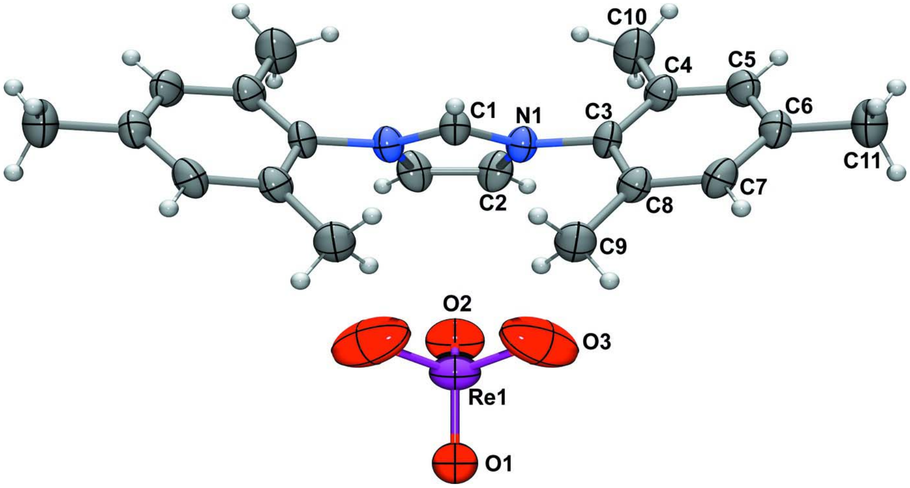

Figure 1

The molecular structure of 1 showing the atomic numbering scheme. Displacement elipsoids are shown at the 50% probability level and H atoms are shown as small spheres of arbitary radii.

1,3-Bis(2,4,6-trimethylphenyl)-3*H*-imidazol-1-ium tetraoxidorhenate(VII)

Crystal data

$(C_{21}H_{25}N_2)[ReO_4]$
$M_r = 555.63$
Monoclinic, $P2_1/m$
Hall symbol: -P 2yb
$a = 8.2989$ (12) Å
$b = 16.373$ (2) Å
$c = 8.3168$ (12) Å
$\beta = 111.948$ (2)°
$V = 1048.2$ (3) Å^3
$Z = 2$

$F(000) = 544$
$D_x = 1.760$ Mg m^{-3}
Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
Cell parameters from 4658 reflections
$\theta = 2.6$–26.5°
$\mu = 5.83$ mm^{-1}
$T = 293$ K
Needle, light brown
0.39 × 0.10 × 0.09 mm

Data collection

Bruker (Siemens) P4 diffractometer
Radiation source: fine-focus sealed tube
Graphite monochromator
Detector resolution: 8.3 pixels mm^{-1}
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 2001)
$T_{min} = 0.489$, $T_{max} = 0.592$

5688 measured reflections
2056 independent reflections
1895 reflections with $I > 2\sigma(I)$
$R_{int} = 0.029$
$\theta_{max} = 26.5°$, $\theta_{min} = 2.5°$
$h = -10 \rightarrow 9$
$k = -19 \rightarrow 20$
$l = -3 \rightarrow 10$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)] = 0.033$
$wR(F^2) = 0.083$
$S = 1.13$
2056 reflections
136 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

$w = 1/[\sigma^2(F_o^2) + (0.0381P)^2 + 1.6122P]$
where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$

$\Delta\rho_{max} = 1.32$ e Å^{-3}
$\Delta\rho_{min} = -0.86$ e Å^{-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.
All hydrogen atoms were added in calculated positions. Each was allowed to ride on the atom to which it is bonded with an isotropic adp set to 1.2 *x* (1.5 *x* for methyl H atoms) the equivalent isotropic adp of that atom.

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

	x	y	z	U_{iso}*/U_{eq}
N1	0.3511 (4)	0.3161 (2)	0.0558 (4)	0.0350 (7)
C1	0.2687 (8)	0.2500	0.0779 (7)	0.0332 (11)
H1	0.1694	0.2500	0.1046	0.040*
C2	0.4899 (6)	0.2909 (3)	0.0166 (6)	0.0441 (10)
H2	0.5694	0.3245	−0.0058	0.053*
C3	0.3080 (5)	0.3994 (2)	0.0842 (5)	0.0340 (8)
C4	0.2575 (6)	0.4545 (2)	−0.0539 (5)	0.0392 (9)
C5	0.2133 (6)	0.5328 (3)	−0.0207 (6)	0.0425 (9)
H5	0.1779	0.5705	−0.1109	0.051*
C6	0.2198 (6)	0.5569 (2)	0.1404 (6)	0.0402 (9)
C7	0.2735 (6)	0.5004 (2)	0.2743 (6)	0.0408 (9)
H7	0.2783	0.5160	0.3835	0.049*
C8	0.3202 (6)	0.4215 (2)	0.2506 (5)	0.0370 (9)
C9	0.3843 (7)	0.3635 (3)	0.4026 (6)	0.0476 (11)
H9A	0.4806	0.3326	0.3976	0.071*
H9B	0.2923	0.3270	0.3982	0.071*
H9C	0.4209	0.3941	0.5088	0.071*
C10	0.2518 (9)	0.4325 (3)	−0.2318 (6)	0.0603 (14)
H10A	0.1737	0.4687	−0.3159	0.091*
H10B	0.2119	0.3772	−0.2584	0.091*
H10C	0.3660	0.4375	−0.2345	0.091*
C11	0.1718 (8)	0.6422 (3)	0.1711 (8)	0.0561 (13)
H11A	0.0786	0.6404	0.2131	0.084*
H11B	0.1352	0.6724	0.0644	0.084*
H11C	0.2707	0.6686	0.2555	0.084*
Re1	0.91490 (4)	0.2500	0.40489 (3)	0.05388 (13)
O1	1.0911 (8)	0.2500	0.5917 (7)	0.0756 (18)
O2	0.9768 (8)	0.2500	0.2300 (8)	0.0769 (17)
O3	0.7942 (8)	0.3344 (5)	0.3947 (7)	0.127 (2)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
N1	0.0437 (19)	0.0286 (16)	0.0382 (17)	−0.0031 (14)	0.0216 (15)	−0.0005 (13)
C1	0.038 (3)	0.026 (3)	0.041 (3)	0.000	0.021 (2)	0.000
C2	0.050 (2)	0.039 (2)	0.056 (3)	−0.009 (2)	0.034 (2)	−0.002 (2)
C3	0.043 (2)	0.0231 (17)	0.042 (2)	−0.0053 (16)	0.0221 (18)	−0.0030 (15)
C4	0.047 (2)	0.032 (2)	0.041 (2)	−0.0073 (18)	0.0204 (19)	−0.0007 (17)
C5	0.051 (3)	0.031 (2)	0.045 (2)	−0.0054 (18)	0.017 (2)	0.0048 (17)
C6	0.043 (2)	0.029 (2)	0.051 (2)	−0.0043 (17)	0.0203 (19)	−0.0030 (17)
C7	0.051 (3)	0.035 (2)	0.042 (2)	−0.0067 (18)	0.024 (2)	−0.0105 (17)
C8	0.042 (2)	0.033 (2)	0.040 (2)	−0.0068 (17)	0.0191 (18)	−0.0027 (16)
C9	0.062 (3)	0.044 (2)	0.039 (2)	−0.003 (2)	0.021 (2)	0.0002 (18)
C10	0.096 (4)	0.049 (3)	0.043 (2)	−0.006 (3)	0.035 (3)	−0.002 (2)
C11	0.071 (3)	0.035 (2)	0.068 (3)	0.003 (2)	0.032 (3)	−0.005 (2)
Re1	0.04629 (19)	0.0774 (2)	0.04186 (17)	0.000	0.02096 (13)	0.000
O1	0.077 (4)	0.060 (3)	0.072 (4)	0.000	0.008 (3)	0.000
O2	0.077 (4)	0.107 (5)	0.060 (3)	0.000	0.041 (3)	0.000
O3	0.116 (5)	0.179 (6)	0.081 (3)	0.073 (5)	0.032 (3)	0.004 (4)

Geometric parameters (Å, º)

N1—C1	1.330 (5)	C7—H7	0.9300
N1—C2	1.372 (5)	C8—C9	1.510 (6)
N1—C3	1.452 (5)	C9—H9A	0.9600
C1—N1^{i}	1.330 (5)	C9—H9B	0.9600
C1—H1	0.9300	C9—H9C	0.9600
C2—C2^{i}	1.341 (9)	C10—H10A	0.9600
C2—H2	0.9300	C10—H10B	0.9600
C3—C4	1.395 (6)	C10—H10C	0.9600
C3—C8	1.397 (6)	C11—H11A	0.9600
C4—C5	1.390 (6)	C11—H11B	0.9600
C4—C10	1.506 (6)	C11—H11C	0.9600
C5—C6	1.379 (6)	Re1—O1	1.689 (5)
C5—H5	0.9300	Re1—O3	1.691 (6)
C6—C7	1.388 (6)	Re1—O3^{i}	1.691 (6)
C6—C11	1.500 (6)	Re1—O2	1.714 (5)
C7—C8	1.385 (6)		
C1—N1—C2	108.0 (4)	C3—C8—C9	122.7 (4)
C1—N1—C3	124.8 (3)	C8—C9—H9A	109.5
C2—N1—C3	126.9 (3)	C8—C9—H9B	109.5
N1—C1—N1^{i}	109.0 (5)	H9A—C9—H9B	109.5
N1—C1—H1	125.5	C8—C9—H9C	109.5
N1^{i}—C1—H1	125.5	H9A—C9—H9C	109.5
C2^{i}—C2—N1	107.5 (2)	H9B—C9—H9C	109.5
C2^{i}—C2—H2	126.3	C4—C10—H10A	109.5
N1—C2—H2	126.3	C4—C10—H10B	109.5

C4—C3—C8	122.4 (4)	H10A—C10—H10B	109.5
C4—C3—N1	119.3 (3)	C4—C10—H10C	109.5
C8—C3—N1	118.2 (3)	H10A—C10—H10C	109.5
C5—C4—C3	117.0 (4)	H10B—C10—H10C	109.5
C5—C4—C10	120.1 (4)	C6—C11—H11A	109.5
C3—C4—C10	122.9 (4)	C6—C11—H11B	109.5
C6—C5—C4	122.7 (4)	H11A—C11—H11B	109.5
C6—C5—H5	118.7	C6—C11—H11C	109.5
C4—C5—H5	118.7	H11A—C11—H11C	109.5
C5—C6—C7	118.2 (4)	H11B—C11—H11C	109.5
C5—C6—C11	121.1 (4)	O1—Re1—O3	109.8 (2)
C7—C6—C11	120.7 (4)	O1—Re1—O3^{i}	109.8 (2)
C8—C7—C6	122.1 (4)	O3—Re1—O3^{i}	109.7 (6)
C8—C7—H7	118.9	O1—Re1—O2	110.5 (3)
C6—C7—H7	118.9	O3—Re1—O2	108.5 (2)
C7—C8—C3	117.5 (4)	O3^{i}—Re1—O2	108.5 (2)
C7—C8—C9	119.8 (4)		

C2—N1—C1—N1^{i}	0.6 (6)	C3—C4—C5—C6	0.7 (7)
C3—N1—C1—N1^{i}	−174.5 (3)	C10—C4—C5—C6	−178.5 (5)
C1—N1—C2—C2^{i}	−0.3 (4)	C4—C5—C6—C7	0.3 (7)
C3—N1—C2—C2^{i}	174.6 (3)	C4—C5—C6—C11	179.7 (4)
C1—N1—C3—C4	−119.1 (5)	C5—C6—C7—C8	0.0 (7)
C2—N1—C3—C4	66.8 (6)	C11—C6—C7—C8	−179.4 (4)
C1—N1—C3—C8	61.3 (6)	C6—C7—C8—C3	−1.4 (6)
C2—N1—C3—C8	−112.7 (5)	C6—C7—C8—C9	177.6 (4)
C8—C3—C4—C5	−2.1 (6)	C4—C3—C8—C7	2.4 (6)
N1—C3—C4—C5	178.3 (4)	N1—C3—C8—C7	−178.0 (4)
C8—C3—C4—C10	177.1 (5)	C4—C3—C8—C9	−176.5 (4)
N1—C3—C4—C10	−2.4 (6)	N1—C3—C8—C9	3.0 (6)

Symmetry code: (i) *x*, −*y*+1/2, *z*.

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C1—H1···O2^{ii}	0.93	2.21	3.12 (1)	167 (1)

Symmetry code: (ii) *x*−1, *y*, *z*.