metal-organic compounds

Morpholinium tetraoxidorhenate(VII)

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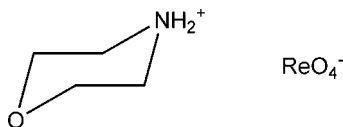Correspondence e-mail: grigoriev@ipc.rssi.ru

The title salt, $(C_4H_{10}NO)[ReO_4]$, contains slightly distorted tetrahedral $[ReO_4]^-$ anions [Re—O = 1.7224 (11)–1.7361 (11) Å]. The structure consists of alternating cationic and anionic layers parallel to (100). The cations and anions are linked by N—H···O hydrogen bonds into chains along [100].

Related literature

See Grigoriev *et al.* (2007) for the structure of guanidinium tetraoxidorhenate.

Experimental

Crystal data

$(C_4H_{10}NO)[ReO_4]$
$M_r = 338.33$
Orthorhombic, $Pca2_1$
$a = 12.4401$ (3) Å
$b = 5.7480$ (2) Å
$c = 11.0420$ (3) Å

$V = 789.57$ (4) Å^3
$Z = 4$
Mo $K\alpha$ radiation
$\mu = 15.37$ mm^{-1}
$T = 100$ (2) K
$0.16 \times 0.12 \times 0.10$ mm

Data collection

Bruker Kappa APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2004)

$T_{min} = 0.239$, $T_{max} = 0.309$
(expected range = 0.167–0.215)
21183 measured reflections
3460 independent reflections
3315 reflections with $I > 2\sigma(I)$
$R_{int} = 0.021$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.010$
$wR(F^2) = 0.020$
$S = 0.94$
3460 reflections
101 parameters
1 restraint

H-atom parameters constrained
$\Delta\rho_{max} = 0.70$ e Å^{-3}
$\Delta\rho_{min} = -0.60$ e Å^{-3}
Absolute structure: Flack (1983), 1650 Friedel pairs
Flack parameter: 0.004 (4)

Table 1
Selected geometric parameters (Å, °).

Re1—O1	1.7361 (11)	Re1—O3	1.7224 (11)
Re1—O2	1.7349 (13)	Re1—O4	1.7256 (13)
O1—Re1—O2	108.44 (6)	O2—Re1—O3	110.14 (6)
O1—Re1—O3	110.39 (6)	O2—Re1—O4	109.39 (6)
O1—Re1—O4	109.41 (6)	O3—Re1—O4	109.06 (6)

Table 2
Hydrogen-bond geometry (Å, °).

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O2^i	0.92	1.99	2.8529 (17)	155
N1—H1B···O1	0.92	1.98	2.8855 (18)	168

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997a); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997a); molecular graphics: *SHELXTL* (Sheldrick, 1997b); software used to prepare material for publication: *SHELXTL*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2306).

References

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supplementary materials

Morpholinium tetraoxidorhenate(VII)

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Comment

The title compound, (I) (Fig. 1), contains slightly distorted tetrahedral ReO_4^- anions with Re—O distances from 1.7224 (11) to 1.7361 (11) Å (Table 1). Morpholinium cations have chair conformation.

The structure of (I) can be described as alternating cationic and anionic layers parallel to the (100) plane (Fig. 2). Morpholinium cations act as proton donors in two hydrogen bonds with O atoms of two ReO_4^- anions from different anionic layers (Table 2). The hydrogen bonds link cations and anions into chains in the [100] direction (Fig. 2).

Experimental

Synthesis of (I) was carried out as a neutralization reaction by dissolution of morpholine under intensive stirring in 0.2 *M* water solution of $HReO_4$ at room temperature to achieve pH = 7, followed by evaporation of the resulting solution over concentrated H_2SO_4. The compound was recrystallized from ethanol.

Refinement

The H atoms of NH_2 and CH_2 groups were refined in idealized geometrical positions with displacement parameters being equal to 1.2 times U_{eq} of the attached N or C atoms.
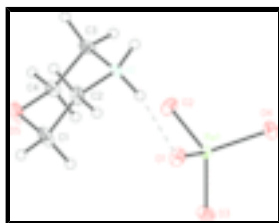
Figures

Fig. 1. A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. Dashed line indicates the hydrogen-bonding interaction.
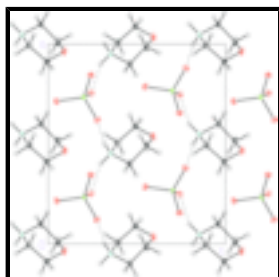

Fig. 2. The packing of (I) showing chains of hydrogen bonds.

supplementary materials

Morpholinium tetraoxidorhenate(VII)

Crystal data

$(C_4H_{10}NO)[Re_1O_4]$	$F_{000} = 624$
$M_r = 338.33$	$D_x = 2.838$ Mg m^{-3}
Orthorhombic, $Pca2_1$	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
Hall symbol: P 2c -2ac	Cell parameters from 7854 reflections
$a = 12.4401$ (3) Å	$\theta = 3.3–35.0°$
$b = 5.7480$ (2) Å	$\mu = 15.37$ mm^{-1}
$c = 11.0420$ (3) Å	$T = 100$ (2) K
$V = 789.57$ (4) Å^3	Fragment, colourless
$Z = 4$	$0.16 \times 0.12 \times 0.10$ mm

Data collection

Bruker Kappa APEX II area-detector diffractometer	3460 independent reflections
Radiation source: fine-focus sealed tube	3315 reflections with $I > 2\alpha(I)$
Monochromator: graphite	$R_{int} = 0.021$
$T = 100(2)$ K	$\theta_{max} = 35.0°$
ω and φ scans	$\theta_{min} = 3.3°$
Absorption correction: multi-scan (SADABS; Sheldrick, 2004)	$h = -20 \rightarrow 19$
$T_{min} = 0.239$, $T_{max} = 0.309$	$k = -9 \rightarrow 9$
21183 measured reflections	$l = -17 \rightarrow 17$

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)] = 0.010$	$w = 1/[\sigma^2(F_o^2)]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2) = 0.020$	$(\Delta/\sigma)_{max} = 0.001$
$S = 0.94$	$\Delta\rho_{max} = 0.70$ e Å^{-3}
3460 reflections	$\Delta\rho_{min} = -0.60$ e Å^{-3}
101 parameters	Extinction correction: SHELXL97, $Fc^* = kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
1 restraint	Extinction coefficient: 0.00092 (6)
Primary atom site location: structure-invariant direct methods	Absolute structure: Flack (1983), with 1650 Friedel pairs
Secondary atom site location: difference Fourier map	Flack parameter: 0.004 (4)

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 $F^2 > 2\text{sigma}(F^2)$ 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	0.733624 (3)	0.804729 (7)	0.201310 (9)	0.00950 (2)
O1	0.62050 (9)	0.9089 (2)	0.27573 (10)	0.0162 (2)
O2	0.75192 (9)	0.5155 (2)	0.24128 (12)	0.0166 (2)
O3	0.84512 (9)	0.9655 (2)	0.24066 (10)	0.0169 (2)
O4	0.71429 (11)	0.8243 (2)	0.04689 (12)	0.0174 (2)
O5	0.53574 (10)	0.5438 (2)	0.59191 (10)	0.0182 (2)
N1	0.45216 (10)	0.5969 (2)	0.35282 (12)	0.0115 (2)
H1A	0.4004	0.5615	0.2963	0.014*
H1B	0.5006	0.6972	0.3177	0.014*
C1	0.48749 (16)	0.7571 (3)	0.55316 (16)	0.0192 (3)
H1C	0.5433	0.8596	0.5180	0.023*
H1D	0.4557	0.8378	0.6238	0.023*
C2	0.40096 (14)	0.7119 (3)	0.45982 (14)	0.0151 (3)
H2A	0.3446	0.6099	0.4943	0.018*
H2B	0.3671	0.8603	0.4350	0.018*
C3	0.50859 (13)	0.3794 (3)	0.39090 (15)	0.0141 (3)
H3A	0.5476	0.3123	0.3210	0.017*
H3B	0.4553	0.2635	0.4191	0.017*
C4	0.58711 (12)	0.4327 (3)	0.49177 (14)	0.0138 (3)
H4A	0.6208	0.2861	0.5196	0.017*
H4B	0.6447	0.5350	0.4603	0.017*

Atomic displacement parameters ($Å^2$)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.00842 (2)	0.01046 (2)	0.00962 (2)	0.00157 (1)	−0.00002 (7)	0.00069 (7)
O1	0.0140 (5)	0.0163 (5)	0.0182 (5)	0.0030 (4)	0.0045 (4)	0.0027 (4)
O2	0.0157 (5)	0.0128 (5)	0.0213 (5)	0.0040 (4)	0.0013 (4)	0.0046 (4)
O3	0.0130 (5)	0.0193 (6)	0.0183 (5)	−0.0017 (4)	0.0005 (4)	−0.0049 (4)
O4	0.0184 (6)	0.0208 (6)	0.0129 (5)	0.0022 (4)	−0.0027 (4)	0.0009 (4)
O5	0.0269 (6)	0.0186 (6)	0.0092 (5)	0.0108 (5)	−0.0016 (4)	0.0001 (4)
N1	0.0126 (5)	0.0139 (6)	0.0080 (5)	−0.0006 (5)	−0.0014 (4)	−0.0015 (5)

C1	0.0298 (9)	0.0169 (7)	0.0109 (7)	0.0106 (6)	−0.0049 (6)	−0.0034 (6)
C2	0.0170 (7)	0.0175 (7)	0.0109 (7)	0.0061 (6)	0.0017 (5)	0.0021 (6)
C3	0.0157 (7)	0.0118 (7)	0.0148 (6)	0.0014 (5)	−0.0021 (5)	−0.0046 (6)
C4	0.0131 (6)	0.0152 (7)	0.0130 (6)	0.0031 (5)	−0.0019 (5)	−0.0025 (5)

Geometric parameters (Å, °)

Re1—O1	1.7361 (11)	C1—C2	1.513 (2)
Re1—O2	1.7349 (13)	C1—H1C	0.9900
Re1—O3	1.7224 (11)	C1—H1D	0.9900
Re1—O4	1.7256 (13)	C2—H2A	0.9900
O5—C4	1.4279 (19)	C2—H2B	0.9900
O5—C1	1.430 (2)	C3—C4	1.513 (2)
N1—C3	1.494 (2)	C3—H3A	0.9900
N1—C2	1.496 (2)	C3—H3B	0.9900
N1—H1A	0.9200	C4—H4A	0.9900
N1—H1B	0.9200	C4—H4B	0.9900
O1—Re1—O2	108.44 (6)	N1—C2—C1	108.11 (14)
O1—Re1—O3	110.39 (6)	N1—C2—H2A	110.1
O1—Re1—O4	109.41 (6)	C1—C2—H2A	110.1
O2—Re1—O3	110.14 (6)	N1—C2—H2B	110.1
O2—Re1—O4	109.39 (6)	C1—C2—H2B	110.1
O3—Re1—O4	109.06 (6)	H2A—C2—H2B	108.4
C4—O5—C1	109.84 (12)	N1—C3—C4	109.94 (13)
C3—N1—C2	110.34 (12)	N1—C3—H3A	109.7
C3—N1—H1A	109.6	C4—C3—H3A	109.7
C2—N1—H1A	109.6	N1—C3—H3B	109.7
C3—N1—H1B	109.6	C4—C3—H3B	109.7
C2—N1—H1B	109.6	H3A—C3—H3B	108.2
H1A—N1—H1B	108.1	O5—C4—C3	111.83 (12)
O5—C1—C2	110.81 (15)	O5—C4—H4A	109.3
O5—C1—H1C	109.5	C3—C4—H4A	109.3
C2—C1—H1C	109.5	O5—C4—H4B	109.3
O5—C1—H1D	109.5	C3—C4—H4B	109.3
C2—C1—H1D	109.5	H4A—C4—H4B	107.9
H1C—C1—H1D	108.1		
C4—O5—C1—C2	62.46 (19)	C2—N1—C3—C4	−54.28 (17)
C3—N1—C2—C1	56.60 (17)	C1—O5—C4—C3	−59.46 (19)
O5—C1—C2—N1	−60.92 (18)	N1—C3—C4—O5	55.57 (17)

Hydrogen-bond geometry (Å, °)

D—H⋯A	D—H	H⋯A	D⋯A	D—H⋯A
N1—H1A⋯O2[i]	0.92	1.99	2.8529 (17)	155
N1—H1B⋯O1	0.92	1.98	2.8855 (18)	168

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

Fig. 1

Fig. 2