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Guanidinium tetraoxidorhenate(VII)

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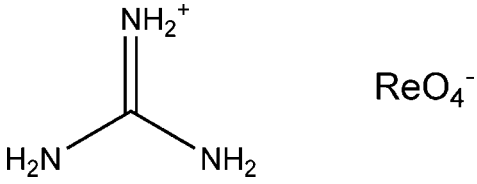
Key indicators: single-crystal X-ray study; T = 100 K; mean σ(e–O) = 0.002 Å; R factor = 0.018; wR factor = 0.040; data-to-parameter ratio = 32.9.

The coordination geometry of the Re atom in the title compound, $(CH_6N_3)[ReO_4]$, is tetrahedral. The structure consists of alternating cationic and anionic layers parallel to the $(1\bar{2}0)$ plane; the layers are held in a three-dimensional structure by N—H···O hydrogen bonds.

Related literature

The structures of tetraoxidorhenates of several cyclic derivatives of guanidinium were described by Leibnitz *et al.* (2001) and Tamm *et al.* (2004). Guanidinium perchlorate was reported by Koziol (1984) and guaninidium tetrafluoridoborate by Kozak *et al.* (1987).



Experimental

Crystal data

$(CH_6N_3)[ReO_4]$
M_r = 310.29
Triclinic, $P\bar{1}$
a = 4.9657 (4) Å
b = 7.7187 (7) Å
c = 8.4423 (7) Å
α = 75.314 (4)°
β = 88.707 (5)°
γ = 80.985 (5)°
V = 309.09 (5) Å^3
Z = 2
Mo $K\alpha$ radiation
μ = 19.61 mm^{-1}
T = 100 (2) K
0.12 × 0.10 × 0.06 mm

Data collection

Bruker Kappa APEXII area-detector diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 1996)
T_{min} = 0.192, T_{max} = 0.346
(expected range = 0.171–0.308)
11709 measured reflections
2698 independent reflections
2506 reflections with $I > 2\sigma(I)$
R_{int} = 0.027

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.018
$wR(F^2)$ = 0.040
S = 1.11
2698 reflections
82 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 1.59 e Å^{-3}
$\Delta\rho_{min}$ = −2.90 e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Re1—O1	1.727 (2)	Re1—O3	1.720 (2)
Re1—O2	1.728 (2)	Re1—O4	1.733 (2)
O1—Re1—O2	109.53 (12)	O2—Re1—O3	108.35 (11)
O1—Re1—O3	109.35 (11)	O2—Re1—O4	109.43 (11)
O1—Re1—O4	111.43 (11)	O3—Re1—O4	108.69 (11)

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

D—H···A	D—H	H···A	D···A	D—H···A
N1—H1A···O3^{i}	0.88	2.41	3.101 (3)	136
N1—H1A···O4^{ii}	0.88	2.45	3.177 (3)	140
N1—H1B···O2^{iii}	0.88	2.10	2.911 (3)	153
N2—H2A···O1^{iv}	0.88	2.22	2.966 (3)	142
N2—H2A···O3	0.88	2.49	3.164 (3)	134
N2—H2B···O2^{iii}	0.88	2.27	3.037 (3)	145
N3—H3A···O4^{ii}	0.88	2.08	2.901 (3)	155
N3—H3B···O1^{iv}	0.88	2.14	2.907 (3)	146
N3—H3B···O4^{v}	0.88	2.50	3.080 (3)	124

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

Data collection: *APEX2* (Bruker, 2006); cell refinement: *SAINT-Plus* (Bruker, 1998); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997*a*); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997*a*); molecular graphics: *SHELXTL97* (Sheldrick, 1997*b*); software used to prepare material for publication: *SHELXTL97*.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: NG2292).

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supplementary materials

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Comment

The title compound, (I) (Fig. 1), contains slightly distorted tetrahedral ReO_4^- anions with Re—O distances from 1.720 (2) to 1.733 (2) Å (Table 1).

Guanidinium cations act as proton donors in a number of weak hydrogen bonds (Fig. 2, Table 2). In contrast to the structure of 2,2,8,8-tetraallyl-3,4,6,7,8,9-hexahydro-2*H*-pyrimidino(1,2 − a)pyrimidinium tetraoxorhenate(VII) (Leibnitz *et al.*, 2001), only one O atom of each tetraoxoanion is bonded to the same cation.

The structure of (I) can be described as alternating cationic and anionic layers parallel to the $(1\bar{2}0)$ plane (Fig. 3). Similar alternating layers are present in the structures of $C(NH_2)_3ClO_4$ (Koziol, 1984) and $C(NH_2)_3BF_4$ (Kozak *et al.*, 1987), but there is an essential difference in the orientation of tetrahedral anions between cationic layers. In (I), two O atoms of each anion participate in hydrogen bonding with one cationic layer and two - with another cationic layer. In $C(NH_2)_3ClO_4$ and isostructural $C(NH_2)_3BF_4$, three O or F atoms of tetrahedral anion are connected with one cationic layer and one atom - with another layer. The resulting hydrogen bond net in (I) is three-dimensional.

Experimental

Synthesis of (I) was carried out as a neutralization reaction by dissolution of stoichiometric quantity of guanidine under intensive stirring in 0.2 *M* water solution of $HReO_4$ at room temperature, followed by evaporation of the resulting solution over P_2O_5. The compound was recrystallized from ethanol.

Refinement

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

Largest electron density peak on the final difference Fourier-synthesis is 1.593 e $Å^{-3}$ (0.94 Å from Re1), the deepest hole is −2.902 e $Å^{-3}$ (0.69 Å from Re1).

Figures

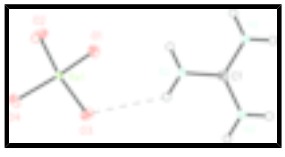

Fig. 1. A view of (I), showing the atom-labelling scheme. Displacement ellipsoids are drawn at the 50% probability level and H atoms are represented by circles of arbitrary size. Dashed line indicates the hydrogen-bonding interaction.

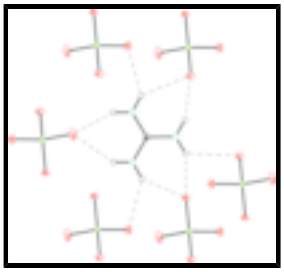

Fig. 2. A pattern of the hydrogen-bonding of one guanidinium cation in (I).

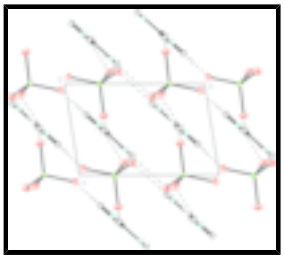

Fig. 3. The packing of (I) showing three-dimensional net of hydrogen bonds.

Guanidinium tetraoxidorhenate(VII) $[C(NH_2)_3][ReO_4]$

Crystal data

$(CH_6N_3)[ReO_4]$	$Z = 2$
$M_r = 310.29$	$F_{000} = 280$
Triclinic, $P\overline{1}$	$D_x = 3.334$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation $\lambda = 0.71073$ Å
$a = 4.9657$ (4) Å	Cell parameters from 8692 reflections
$b = 7.7187$ (7) Å	$\theta = 2.8$–35.0^o
$c = 8.4423$ (7) Å	$\mu = 19.61$ mm^{-1}
$\alpha = 75.314$ (4)°	$T = 100$ (2) K
$\beta = 88.707$ (5)°	Plate, colourless
$\gamma = 80.985$ (5)°	0.12 × 0.10 × 0.06 mm
$V = 309.09$ (5) Å^3	

Data collection

Bruker KappaAPEXII area-detector diffractometer	2698 independent reflections
Radiation source: fine-focus sealed tube	2506 reflections with $I > 2\sigma(I)$
Monochromator: graphite	$R_{int} = 0.027$
$T = 100(2)$ K	$\theta_{max} = 35.0^o$
ω and φ scans	$\theta_{min} = 2.8^o$
Absorption correction: multi-scan (SADABS; Sheldrick, 1996)	$h = -7 \rightarrow 8$
$T_{min} = 0.192$, $T_{max} = 0.346$	$k = -12 \rightarrow 12$
11709 measured reflections	$l = -13 \rightarrow 13$

Refinement

Refinement on F^2	Secondary atom site location: difference Fourier map
Least-squares matrix: full	Hydrogen site location: inferred from neighbouring sites
$R[F^2 > 2\sigma(F^2)] = 0.018$	H-atom parameters constrained
$wR(F^2) = 0.040$	$w = 1/[\sigma^2(F_o^2) + (0.0177P)^2 + 0.58P]$ where $P = (F_o^2 + 2F_c^2)/3$
$S = 1.11$	$(\Delta/\sigma)_{max} = 0.002$
2698 reflections	$\Delta\rho_{max} = 1.59$ e Å^{-3}
82 parameters	$\Delta\rho_{min} = -2.90$ e Å^{-3}
Primary atom site location: structure-invariant direct methods	Extinction correction: none

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 $F^2 > 2$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	0.00024 (2)	0.252412 (14)	0.685859 (11)	0.00754 (3)
O1	−0.1246 (5)	0.3316 (3)	0.4871 (2)	0.0164 (4)
O2	−0.0731 (5)	0.0363 (3)	0.7671 (3)	0.0185 (4)
O3	0.3487 (5)	0.2424 (3)	0.6862 (3)	0.0150 (4)
O4	−0.1391 (5)	0.3927 (3)	0.8075 (3)	0.0139 (4)
N1	0.4480 (5)	0.1966 (4)	0.0569 (3)	0.0129 (4)
H1A	0.5021	0.2371	−0.0438	0.015*
H1B	0.3283	0.1212	0.0769	0.015*
N2	0.4610 (5)	0.1881 (4)	0.3302 (3)	0.0125 (4)
H2A	0.5238	0.2229	0.4119	0.015*
H2B	0.3413	0.1127	0.3486	0.015*
N3	0.7270 (5)	0.3627 (3)	0.1487 (3)	0.0117 (4)
H3A	0.7809	0.4031	0.0479	0.014*
H3B	0.7927	0.3977	0.2294	0.014*
C1	0.5471 (5)	0.2493 (3)	0.1790 (3)	0.0087 (4)

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.00736 (5)	0.00912 (5)	0.00673 (4)	−0.00250 (3)	0.00081 (3)	−0.00245 (3)
O1	0.0142 (10)	0.0288 (12)	0.0075 (8)	−0.0076 (9)	−0.0017 (7)	−0.0042 (8)
O2	0.0139 (10)	0.0106 (9)	0.0292 (12)	−0.0040 (8)	0.0018 (8)	−0.0004 (8)
O3	0.0097 (9)	0.0179 (10)	0.0179 (9)	−0.0032 (8)	0.0006 (7)	−0.0046 (8)
O4	0.0161 (10)	0.0155 (9)	0.0112 (8)	−0.0009 (8)	0.0026 (7)	−0.0066 (7)
N1	0.0165 (11)	0.0169 (11)	0.0075 (8)	−0.0088 (9)	−0.0008 (8)	−0.0033 (8)
N2	0.0154 (11)	0.0157 (11)	0.0075 (8)	−0.0073 (9)	0.0022 (7)	−0.0021 (8)
N3	0.0148 (11)	0.0119 (10)	0.0100 (9)	−0.0064 (8)	0.0005 (7)	−0.0028 (7)
C1	0.0092 (11)	0.0081 (10)	0.0083 (9)	−0.0006 (8)	−0.0008 (8)	−0.0016 (8)

Geometric parameters (Å, °)

Re1—O1	1.727 (2)	C1—N2	1.330 (3)
Re1—O2	1.728 (2)	N2—H2A	0.8800
Re1—O3	1.720 (2)	N2—H2B	0.8800
Re1—O4	1.733 (2)	C1—N3	1.323 (3)
C1—N1	1.330 (3)	N3—H3A	0.8800
N1—H1A	0.8800	N3—H3B	0.8800
N1—H1B	0.8800		
O1—Re1—O2	109.53 (12)	C1—N2—H2A	120.0
O1—Re1—O3	109.35 (11)	C1—N2—H2B	120.0
O1—Re1—O4	111.43 (11)	H2A—N2—H2B	120.0
O2—Re1—O3	108.35 (11)	C1—N3—H3A	120.0
O2—Re1—O4	109.43 (11)	C1—N3—H3B	120.0
O3—Re1—O4	108.69 (11)	H3A—N3—H3B	120.0
C1—N1—H1A	120.0	N1—C1—N2	119.1 (2)
C1—N1—H1B	120.0	N1—C1—N3	119.9 (2)
H1A—N1—H1B	120.0	N2—C1—N3	120.9 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
N1—H1A···O3^{i}	0.88	2.41	3.101 (3)	136
N1—H1A···O4^{ii}	0.88	2.45	3.177 (3)	140
N1—H1B···O2^{iii}	0.88	2.10	2.911 (3)	153
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N3—H3B···O1^{iv}	0.88	2.14	2.907 (3)	146
N3—H3B···O4^{v}	0.88	2.50	3.080 (3)	124

Symmetry codes: (i) *x*, *y*, *z*−1; (ii) *x*+1, *y*, *z*−1; (iii) −*x*, −*y*, −*z*+1; (iv) *x*+1, *y*, *z*; (v) −*x*+1, −*y*+1, −*z*+1.

Fig. 1

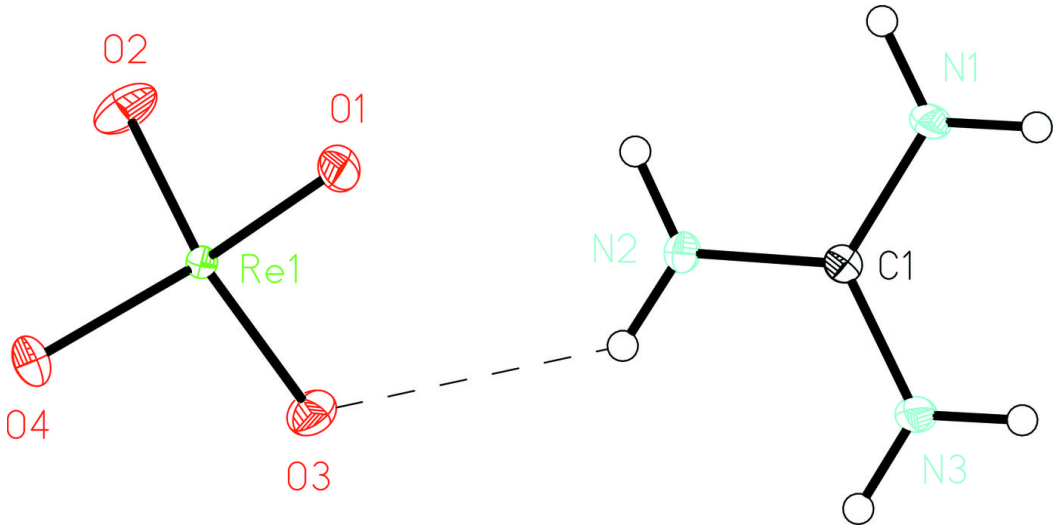

Fig. 2

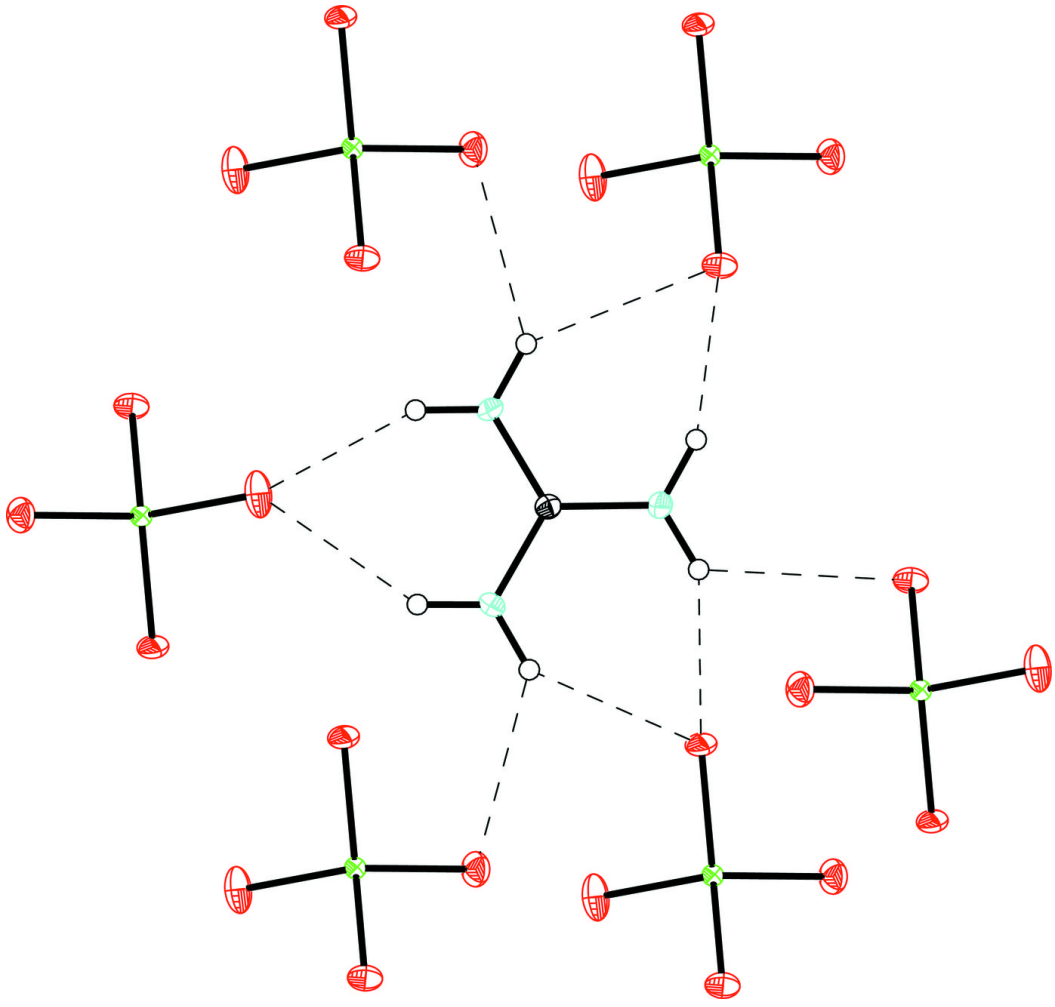

Fig. 3