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Bis[glycinium(0.5+)] perrhenate

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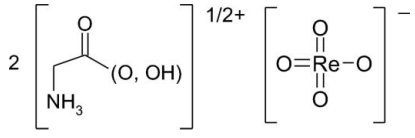
Key indicators: single-crystal X-ray study; T = 291 K; mean σ(C–C) = 0.003 Å; disorder in solvent or counterion; R factor = 0.030; wR factor = 0.055; data-to-parameter ratio = 60.9.

All the residues of the title compound, $(C_2H_{5.5}NO_2)_2[ReO_4]$, are located in general crystallographic positions. The glycine molecules have usual conformations [Rodrigues Matos Beja *et al.* (2006). *Acta Cryst.* C**62**, o71–o72] with the H atom of the carboxylate group half-occupied, thus bearing a formal half-positive charge per molecule. The perrhenate anion has nearly ideal tetrahedral geometry. A large number of strong hydrogen bonds give rise to the overall three-dimensional network. A two-dimensional network, parallel to (100), is made up of strong O—H···O hydrogen bonds with a donor acceptor distance of 2.445 (2) Å. A large number of weaker O—H···O and N—H···O hydrogen bonds consolidates the structure into an overall three-dimensional network.

Related literature

For a related structure, see: Rodrigues *et al.* (2006).



Experimental

Crystal data

$(C_2H_{5.5}NO_2)_2[ReO_4]$
M_r = 401.35
Monoclinic, $P2_1/c$
a = 15.7095 (5) Å
b = 8.1826 (3) Å
c = 8.2909 (3) Å
β = 103.7152 (16)°
V = 1035.36 (6) Å^3
Z = 4
Mo $K\alpha$ radiation
μ = 11.77 mm^{-1}
T = 291 (2) K
0.15 × 0.13 × 0.10 mm

Data collection

Bruker APEXII diffractometer
Absorption correction: multi-scan (*SADABS*; Sheldrick, 2003)
T_{min} = 0.18, T_{max} = 0.31
78826 measured reflections
8587 independent reflections
6232 reflections with $I > 2\sigma(I)$
R_{int} = 0.038

Refinement

$R[F^2 > 2\sigma(F^2)]$ = 0.030
$wR(F^2)$ = 0.055
S = 1.06
8587 reflections
141 parameters
H-atom parameters constrained
$\Delta\rho_{max}$ = 2.33 e Å^{-3}
$\Delta\rho_{min}$ = −2.87 e Å^{-3}

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

D—H···A	D—H	H···A	D···A	D—H···A
O12—H12···O22^{i}	0.82	1.64	2.445 (2)	167
N11—H11A···O11^{ii}	0.89	2.02	2.869 (2)	158
N11—H11B···O21	0.89	2.18	3.003 (2)	153
N11—H11B···O2	0.89	2.47	3.000 (3)	119
N11—H11C···O11^{iii}	0.89	1.94	2.830 (3)	175
O22—H22···O12^{i}	0.82	1.64	2.445 (2)	165
N21—H21A···O21	0.89	2.27	2.738 (3)	113
N21—H21A···O1	0.89	2.29	3.136 (4)	158
N21—H21B···O3^{iv}	0.89	2.10	2.896 (3)	149
N21—H21B···O1^{v}	0.89	2.60	3.274 (4)	133
N21—H21C···O4^{vi}	0.89	2.14	2.794 (3)	130
N21—H21C···O1^{vii}	0.89	2.37	3.012 (3)	130

Symmetry codes: (i) $-x, -y+1, -z$; (ii) $-x, y-\frac{1}{2}, -z+\frac{1}{2}$; (iii) $x, -y+\frac{3}{2}, z-\frac{1}{2}$; (iv) $-x+1, -y+1, -z$; (v) $x, -y+\frac{1}{2}, z-\frac{1}{2}$; (vi) $x, -y+\frac{1}{2}, z+\frac{1}{2}$; (vii) $-x+1, y-\frac{1}{2}, -z+\frac{1}{2}$.

Data collection: *APEX2* (Bruker–Nonius, 2004); cell refinement: *SAINT* (Bruker, 2003); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *ORTEPII* (Johnson, 1976); software used to prepare material for publication: *SHELXL97* and *PLATON* (Spek, 2003).

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Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: RN2049).

References

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supplementary materials

Acta Cryst. (2009). E**65**, m19 [**doi:10.1107/S160053680803849X**]

Bis[glycinium(0.5+)] perrhenate

V. H. Rodrigues, M. M. R. R. Costa, T. Dekola and E. de Matos Gomes

Experimental

Crystals of the monoclinic polymorph of glycinium glycine perrhenate were obtained from a water solution of analytical grade reagents glycine(99.5%) and perrhenic acid solution (65–75% water, 99.5%), purchased from Aldrich, in a 2:1 molar ratio.

Refinement

The structure was solved by direct methods using *SHELXS97*. All H atoms were first located on a difference Fourier map; those bonded to C atoms and carboxyl O atoms were placed at idealized positions and refined as riding [C—H=0.97 and 0.98 Å, O—H=0.82 Å, U_{iso}(H)=1.2U_{eq}(C) and U_{iso}(H)=1.5U_{eq}(O)].

Examination of the crystal structure with *PLATON* (Spek, 2003) showed that there are no solvent-accessible voids in the crystal lattice.

Figures

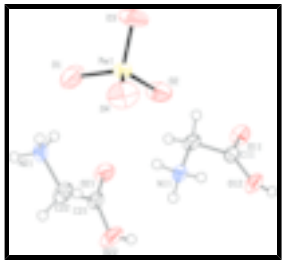

Fig. 1. *ORTEPII* (Johnson, 1976) plot of the title compound. Displacement ellipsoids are drawn at the 50% level.

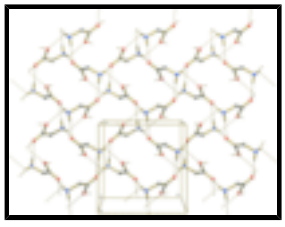

Fig. 2. Packing of glycine molecules showing the (100) network.

Bis[glycinium(0.5+)] perrhenate

Crystal data

$2(C_2H_{5.5}NO_2)[ReO_4]$	F_{000} = 752
M_r = 401.35	D_x = 2.575 Mg m^{-3}
Monoclinic, $P2_1/c$	Mo $K\alpha$ radiation λ = 0.71073 Å

Hall symbol: -P 2ybc	Cell parameters from 7839 reflections
a = 15.7095 (5) Å	θ = 4.0–40.1º
b = 8.1826 (3) Å	μ = 11.77 mm^{-1}
c = 8.2909 (3) Å	T = 291 (2) K
β = 103.7152 (16)º	Block, translucent colourless
V = 1035.36 (6) Å^3	0.15 × 0.13 × 0.10 mm
Z = 4	

Data collection

Bruker APEXII diffractometer	8587 independent reflections
Radiation source: fine-focus sealed tube	6232 reflections with $I > 2\sigma(I)$
Monochromator: graphite	R_{int} = 0.038
T = 291(2) K	θ_{max} = 45.3º
φ and ω scans	θ_{min} = 2.7º
Absorption correction: multi-scan (SADABS; Sheldrick, 2003)	h = −30→31
T_{min} = 0.18, T_{max} = 0.31	k = −16→15
78826 measured reflections	l = −16→16

Refinement

Refinement on F^2	Hydrogen site location: inferred from neighbouring sites
Least-squares matrix: full	H-atom parameters constrained
$R[F^2 > 2\sigma(F^2)]$ = 0.030	$w = 1/[\sigma^2(F_o^2) + (0.001P)^2 + 2.2865P]$ where $P = (F_o^2 + 2F_c^2)/3$
$wR(F^2)$ = 0.055	$(\Delta/\sigma)_{max}$ = 0.002
S = 1.06	$\Delta\rho_{max}$ = 2.33 e Å^{-3}
8587 reflections	$\Delta\rho_{min}$ = −2.87 e Å^{-3}
141 parameters	Extinction correction: SHELXL97 (Sheldrick, 2008), $Fc^*=kFc[1+0.001xFc^2\lambda^3/\sin(2\theta)]^{-1/4}$
Primary atom site location: structure-invariant direct methods	Extinction coefficient: 0.00614 (15)
Secondary atom site location: difference Fourier map	

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}	Occ. (<1)
Re1	0.384633 (5)	0.604894 (11)	0.053166 (13)	0.02896 (3)	
O1	0.43021 (16)	0.4923 (4)	0.2269 (3)	0.0636 (7)	
O2	0.28327 (14)	0.6733 (3)	0.0625 (4)	0.0633 (8)	
O3	0.44936 (16)	0.7699 (3)	0.0397 (5)	0.0685 (9)	
O4	0.3784 (2)	0.4775 (4)	−0.1121 (3)	0.0698 (8)	
O11	0.02851 (12)	0.9119 (2)	0.2663 (2)	0.0343 (4)	
O12	−0.02823 (11)	0.7477 (2)	0.0538 (2)	0.0366 (4)	
H12	−0.0747	0.7890	0.0614	0.055*	0.50
C11	0.03285 (13)	0.7965 (3)	0.1730 (3)	0.0242 (3)	
C12	0.11921 (13)	0.7067 (3)	0.1971 (3)	0.0291 (4)	
H12A	0.1608	0.7754	0.1588	0.035*	
H12B	0.1424	0.6864	0.3145	0.035*	
N11	0.11042 (11)	0.5501 (2)	0.1070 (2)	0.0253 (3)	
H11A	0.0761	0.4833	0.1479	0.038*	
H11B	0.1631	0.5048	0.1189	0.038*	
H11C	0.0868	0.5676	−0.0002	0.038*	
O21	0.24920 (12)	0.3008 (2)	0.0961 (3)	0.0409 (4)	
O22	0.16419 (12)	0.1024 (3)	−0.0423 (3)	0.0504 (6)	
H22	0.1241	0.1641	−0.0353	0.076*	0.50
C21	0.23754 (14)	0.1710 (3)	0.0210 (3)	0.0281 (4)	
C22	0.31325 (16)	0.0716 (3)	−0.0074 (4)	0.0373 (5)	
H22A	0.3076	−0.0399	0.0286	0.045*	
H22B	0.3109	0.0690	−0.1254	0.045*	
N21	0.39933 (12)	0.1372 (3)	0.0823 (3)	0.0337 (4)	
H21A	0.3923	0.2372	0.1194	0.051*	
H21B	0.4351	0.1417	0.0136	0.051*	
H21C	0.4224	0.0724	0.1676	0.051*	

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.02220 (4)	0.02717 (4)	0.04083 (5)	−0.00013 (3)	0.01407 (3)	−0.00404 (3)
O1	0.0401 (12)	0.093 (2)	0.0524 (14)	−0.0127 (13)	−0.0001 (10)	0.0177 (14)
O2	0.0333 (10)	0.0379 (10)	0.130 (2)	0.0040 (8)	0.0416 (13)	−0.0007 (13)
O3	0.0467 (12)	0.0383 (11)	0.136 (3)	−0.0090 (9)	0.0521 (16)	−0.0019 (14)
O4	0.080 (2)	0.083 (2)	0.0523 (15)	−0.0094 (16)	0.0276 (14)	−0.0268 (14)
O11	0.0309 (7)	0.0398 (9)	0.0320 (8)	0.0054 (7)	0.0069 (6)	−0.0106 (7)
O12	0.0226 (7)	0.0378 (9)	0.0440 (10)	0.0077 (6)	−0.0031 (7)	−0.0144 (7)
C11	0.0206 (7)	0.0270 (8)	0.0257 (9)	0.0011 (6)	0.0068 (6)	−0.0006 (7)
C12	0.0194 (7)	0.0314 (9)	0.0348 (11)	0.0015 (7)	0.0029 (7)	−0.0069 (8)
N11	0.0187 (6)	0.0262 (7)	0.0300 (9)	0.0035 (5)	0.0043 (6)	0.0002 (6)
O21	0.0259 (7)	0.0310 (8)	0.0635 (13)	0.0041 (6)	0.0063 (8)	−0.0144 (8)
O22	0.0219 (7)	0.0475 (11)	0.0747 (15)	0.0057 (7)	−0.0029 (8)	−0.0284 (11)
C21	0.0213 (8)	0.0288 (9)	0.0323 (10)	0.0054 (7)	0.0027 (7)	−0.0034 (8)

C22	0.0254 (9)	0.0429 (13)	0.0417 (13)	0.0092 (9)	0.0043 (9)	−0.0133 (10)
N21	0.0217 (7)	0.0309 (9)	0.0490 (12)	0.0096 (6)	0.0094 (8)	0.0134 (8)

Geometric parameters (Å, °)

Re1—O4	1.706 (3)	N11—H11B	0.8900
Re1—O2	1.707 (2)	N11—H11C	0.8900
Re1—O3	1.710 (2)	O21—C21	1.223 (3)
Re1—O1	1.717 (3)	O22—C21	1.277 (3)
O11—C11	1.233 (3)	O22—H22	0.8200
O12—C11	1.267 (3)	C21—C22	1.505 (3)
O12—H12	0.8200	C22—N21	1.481 (3)
C11—C12	1.514 (3)	C22—H22A	0.9700
C12—N11	1.473 (3)	C22—H22B	0.9700
C12—H12A	0.9700	N21—H21A	0.8900
C12—H12B	0.9700	N21—H21B	0.8900
N11—H11A	0.8900	N21—H21C	0.8900
O4—Re1—O2	111.10 (15)	C12—N11—H11C	109.5
O4—Re1—O3	110.60 (15)	H11A—N11—H11C	109.5
O2—Re1—O3	108.64 (11)	H11B—N11—H11C	109.5
O4—Re1—O1	106.21 (17)	C21—O22—H22	109.5
O2—Re1—O1	110.24 (14)	O21—C21—O22	127.1 (2)
O3—Re1—O1	110.03 (15)	O21—C21—C22	121.4 (2)
C11—O12—H12	109.5	O22—C21—C22	111.5 (2)
O11—C11—O12	125.87 (19)	N21—C22—C21	112.7 (2)
O11—C11—C12	118.03 (19)	N21—C22—H22A	109.0
O12—C11—C12	116.08 (18)	C21—C22—H22A	109.0
N11—C12—C11	112.44 (17)	N21—C22—H22B	109.0
N11—C12—H12A	109.1	C21—C22—H22B	109.0
C11—C12—H12A	109.1	H22A—C22—H22B	107.8
N11—C12—H12B	109.1	C22—N21—H21A	109.5
C11—C12—H12B	109.1	C22—N21—H21B	109.5
H12A—C12—H12B	107.8	H21A—N21—H21B	109.5
C12—N11—H11A	109.5	C22—N21—H21C	109.5
C12—N11—H11B	109.5	H21A—N21—H21C	109.5
H11A—N11—H11B	109.5	H21B—N21—H21C	109.5
O11—C11—C12—N11	166.3 (2)	O21—C21—C22—N21	7.3 (4)
O12—C11—C12—N11	−15.7 (3)	O22—C21—C22—N21	−172.9 (2)

Hydrogen-bond geometry (Å, °)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
O12—H12···O22^{i}	0.82	1.64	2.445 (2)	167
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Fig. 1

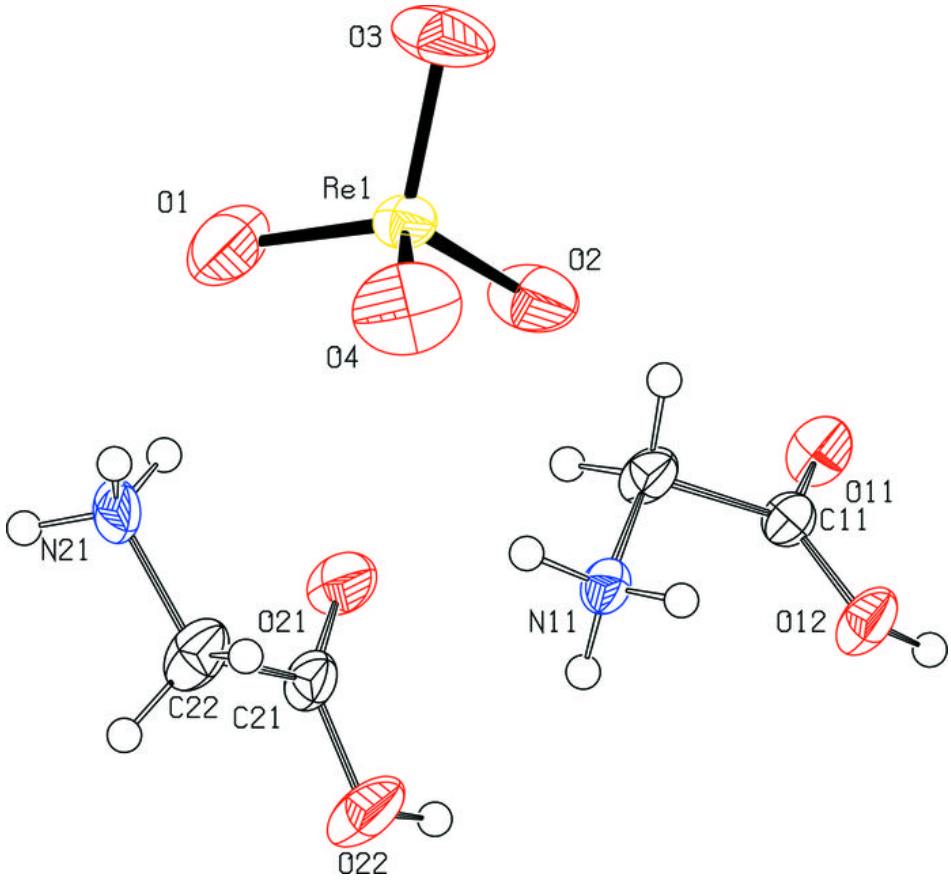

Fig. 2