

Keywords: crystal structure; rhenium; cluster; β -alanine; zwitterionic ammoniacarboxylato complex; quadruple metal–metal bond

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Crystal structure of *cis*-bis(μ - β -alanine- κ^2 O:O')bis-[trichloridorhenium(III)](*Re–Re*) sesquihydrate

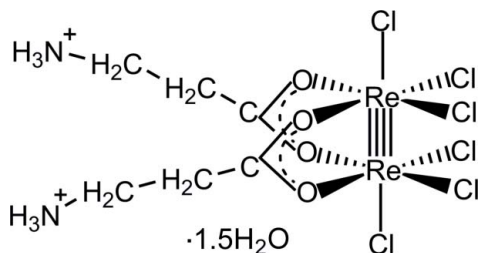
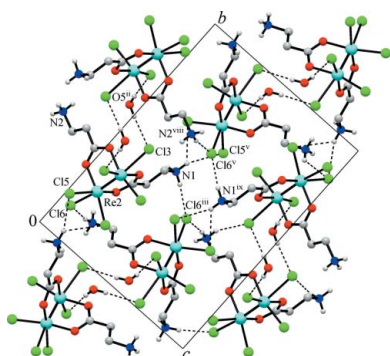
Alexander A. Golichenko,^{a*} Konstantin V. Domasevitch,^b Dina E. Kytova^a and Alexander V. Shtemenko^a

^aDepartment of Inorganic Chemistry, Ukrainian State University of Chemical Technology, Gagarin Ave. 8, Dnipropetrovsk 49005, Ukraine, and ^bDepartment of Inorganic Chemistry, National Taras Shevchenko University of Kyiv, Volodymirska Str. 64, Kyiv 01033, Ukraine. *Correspondence e-mail: golichenko_alex@i.ua

The structure of the title compound, $[\text{Re}_2\text{Cl}_6(\text{C}_3\text{H}_7\text{NO}_2)_2] \cdot 1.5\text{H}_2\text{O}$, comprises a dinuclear complex cation [$\text{Re}–\text{Re} = 2.2494(3) \text{ \AA}$] involving *cis*-oriented double carboxylate bridges, four equatorial chloride ions and two weakly bonded chloride ligands in the axial positions at the two rhenium(III) atoms. In the crystal, two complex molecules and two water molecules constitute hydrogen-bonded dimers, while an extensive hydrogen-bonding network involving the groups of the zwitterionic ligand is important for generation of the framework. An additional partially occupied water molecule is disordered over two sets of sites about a symmetry centre with a site-occupancy ratio of 0.3:0.2.

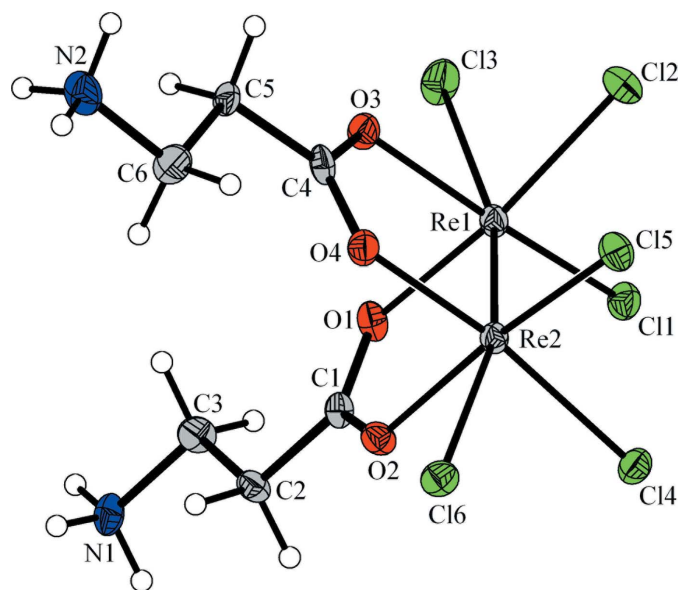
1. Chemical context

Investigations of complex compounds with multiple metal–metal bonds, which exhibit biological activity, generate great interest at the present stage of development of coordination chemistry (Jung & Lippard, 2007; Shtemenko *et al.*, 2013). Binuclear clusters of rhenium(III) are the classical complexes with a unique quadruple metal–metal bond (Cotton *et al.*, 2005; Golichenko & Shtemenko, 2006). In our previous studies, we have shown that these compounds can be used in medical practice as antitumor, antiradical, and hepato- and nephro-protective substances with low toxicity (Dimitrov & Eastland, 1978; Shtemenko *et al.*, 2007, 2008, 2009, 2013). Labile axial ligands and equatorial chloride groups are the reactive centres not only for other substances *in vitro*, but also in interactions with biological macromolecules, such as proteins, DNA, and others *in vivo* (Shtemenko *et al.*, 2013). In this context, we present the synthesis and crystal structure of a new complex compound of dirhenium(III) with β -alanine as biologically active substance, which can exhibit antitumor activity (Shtemenko *et al.*, 2009).



2. Structural commentary

It is well known that β -alanine and other amino acids are able to coordinate to a variety of transition metals (Korp *et al.*,


Figure 1

The molecular structure of the title complex, with displacement ellipsoids drawn at the 40% probability level. Solvent water molecules have been omitted for clarity.

1981; Shtemenko *et al.*, 2009). The quadruple Re–Re bond [2.2494 (3) Å] is typical of related dicarboxylato clusters (Cotton *et al.*, 2005; Shtemenko *et al.*, 2009). The octahedral coordination environment of each rhenium ion in the title compound (Fig. 1) also comprises two chloride anions and two oxygen atoms of zwitterionic alanine ligands. The distorted octahedral coordination of the metals is completed by weakly bonded chloride ions [Re1–Cl3 = 2.6766 (16) and Re2–Cl6 = 2.7501 (14) Å], in a *trans*-position to the Re–Re bond. This may be compared with the similar weak binding of N- or O-donors, which is characteristic of dicarboxylatodirhenium compounds (Bera *et al.*, 2003; Shtemenko *et al.*, 2009) and is even more appreciable for cationic tetracarboxylatodirhenium species commonly accommodating a pair of chloride anions at both axial sites (Re–Cl = 2.48–2.52 Å; Shtemenko *et al.*, 2001).

3. Supramolecular features

The title compound displays a three-dimensional structure dominated by weak hydrogen bonds of the O–H···Cl, N–H···Cl, C–H···O and C–H···Cl types (Table 1). The primary supramolecular motif consists of centrosymmetric dimers (symmetry code: $-x, -y + 1, -z$) incorporating two complex moieties and two water molecules (Fig. 2), with a typical hydrogen-bonding geometry [O···Cl = 3.342 (6) and 3.360 (6) Å], while an extensive hydrogen-bonding network involving the ammonium groups and chloride acceptors assembles the dimers into a three-dimensional framework. One of these N–H···Cl bonds is bifurcated and one is trifurcated (Table 1). It is worth noting that most of the N–H···Cl interactions are observed for the Cl3 and Cl6 acceptors. Such selectivity is likely predetermined by the steric

Table 1

Hydrogen-bond geometry (Å, °).

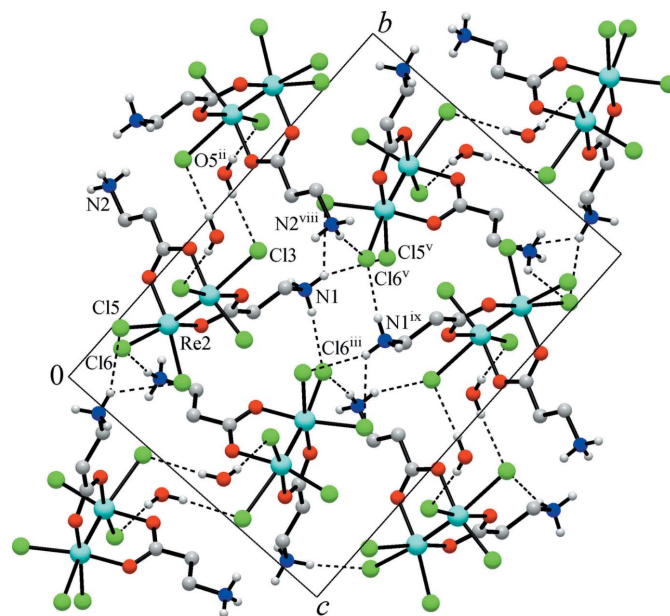
<i>D</i> –H··· <i>A</i>	<i>D</i> –H	H··· <i>A</i>	<i>D</i> ··· <i>A</i>	<i>D</i> –H··· <i>A</i>
O5–H1W···Cl2 ⁱ	0.85	2.51	3.360 (6)	174
O5–H2W···Cl3 ⁱⁱ	0.85	2.50	3.342 (6)	174
N1–H1N···Cl6 ⁱⁱⁱ	0.90	2.32	3.202 (5)	167
N1–H2N···Cl4 ^{iv}	0.90	2.78	3.396 (6)	127
N1–H2N···Cl5 ^{iv}	0.90	2.78	3.557 (5)	145
N1–H2N···Cl6 ^{iv}	0.90	2.75	3.410 (6)	131
N1–H3N···Cl3 ⁱ	0.90	2.34	3.223 (5)	167
N2–H4N···Cl6 ^v	0.90	2.30	3.188 (6)	172
N2–H5N···Cl2 ^{vi}	0.90	2.84	3.575 (6)	140
N2–H5N···Cl5 ^{vi}	0.90	2.66	3.373 (5)	137
N2–H6N···Cl3 ⁱⁱ	0.90	2.40	3.238 (7)	156
C3–H3A···O5	0.98	2.54	3.253 (9)	129
C2–H2A···Cl2 ⁱ	0.98	2.78	3.717 (6)	160

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

accessibility and relative negative charge located at the Cl atoms, since these distal ‘axial’ chloride ligands Cl3 and Cl6 are the most underbonded and highly nucleophilic. The disordered water molecules reside in the framework cages and adopt a series of short contacts, which may be attributed to weak hydrogen bonding [O···Cl = 3.07 (2)–3.42 (4) Å].

4. Synthesis and crystallization

1.00 g (1.25 mmol) of $[\beta\text{-AlaH}]_2\text{Re}_2\text{Cl}_8$ was dissolved in 20 ml of acetonitrile and the solution was concentrated to half of the


Figure 2

The crystal structure of the title complex viewed down the *a* axis, with the C–H hydrogens and disordered water molecules omitted for clarity. Dotted lines indicate hydrogen bonds involving the OH and NH groups. Note the assembly of the hydrogen-bonded dimers constituted by two complex molecules and two water molecules. [Symmetry codes: (ii) $-x + 1, -y + 1, -z$; (iii) $x + \frac{1}{2}, -y + \frac{1}{2}, z + \frac{1}{2}$; (v) $-x + \frac{3}{2}, y + \frac{1}{2}, -z + \frac{1}{2}$; (viii) $-x, -y + 1, -z$; (ix) $-x + 1, -y + 1, -z + 1$.]

Table 2
Experimental details.

Crystal data	
Chemical formula	[Re ₂ Cl ₆ (C ₃ H ₇ NO ₂) ₂].1.5H ₂ O
<i>M_r</i>	790.32
Crystal system, space group	Monoclinic, <i>P</i> 2 ₁ / <i>n</i>
Temperature (K)	223
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.2884 (9), 17.4526 (14), 13.2715 (14)
β (°)	107.838 (3)
<i>V</i> (Å ³)	1827.5 (3)
<i>Z</i>	4
Radiation type	Mo <i>K</i> α
μ (mm ⁻¹)	14.13
Crystal size (mm)	0.25 × 0.22 × 0.14
Data collection	
Diffraction	Siemens SMART CCD area detector
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2008)
<i>T</i> _{min} , <i>T</i> _{max}	0.139, 0.267
No. of measured, independent and observed [<i>I</i> > 2σ(<i>I</i>)] reflections	11029, 4413, 4235
<i>R</i> _{int}	0.027
(sin θ/λ) _{max} (Å ⁻¹)	0.665
Refinement	
<i>R</i> [<i>F</i> ² > 2σ(<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.031, 0.075, 1.26
No. of reflections	4413
No. of parameters	210
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{\text{max}}$, $\Delta\rho_{\text{min}}$ (e Å ⁻³)	1.70, -1.62

Computer programs: *SMART* and *SAINT* (Bruker, 2008), *SHELXS97* and *SHELXL97* (Sheldrick, 2008), *DIAMOND* (Brandenburg, 1999) and *WinGX* (Farrugia, 2012).

initial volume using a rotary evaporator. A new portion (10 ml) of the solvent was added and the solution was evaporated to half of the initial volume. This procedure was repeated five times. The dark-green crystals obtained were filtered, washed with two 5 ml portions of cold acetonitrile and diethyl ether and dried under vacuum at 353 K. The product (0.77 g) was recrystallized from acetone, yielding the title complex in 81% yield.

5. Refinement details

Crystal data, data collection and structure refinement details are summarized in Table 2. H atoms were refined using a riding model, with O–H = 0.85, N–H = 0.90, C–H = 0.98 Å,

and with $U_{\text{iso}}(\text{H}) = 1.2U_{\text{eq}}(\text{C})$ or $1.5U_{\text{eq}}(\text{N},\text{O})$. One of the solvate water molecules is disordered over two unequal contributions, which are further disordered about an inversion centre. The refined partial occupancies for this oxygen atom (O6A and O6B) are 0.3 and 0.2, respectively. Both sites were refined anisotropically. The H atoms of the partially occupied water molecule could not be located and were omitted from the final refinement.

Acknowledgements

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Crystal structure of *cis*-bis(μ - β -alanine- κ^2 O:O')bis[trichloridorhenium(III)](*Re*–*Re*) sesquihydrate

Alexander A. Golichenko, Konstantin V. Domasevitch, Dina E. Kytova and Alexander V. Shtemenko

Computing details

Data collection: *SMART* (Bruker, 2008); cell refinement: *SAINTE* (Bruker, 2008); data reduction: *SAINTE* (Bruker, 2008); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *DIAMOND* (Brandenburg, 1999); software used to prepare material for publication: *WinGX* (Farrugia, 2012).

cis-Bis(μ - β -alanine- κ^2 O:O')bis[trichloridorhenium(III)](*Re*–*Re*) sesquihydrate

Crystal data

[Re₂Cl₆(C₃H₇NO₂)₂]·1.5H₂O

M_r = 790.32

Monoclinic, *P*2₁/*n*

a = 8.2884 (9) Å

b = 17.4526 (14) Å

c = 13.2715 (14) Å

β = 107.838 (3)°

V = 1827.5 (3) Å³

Z = 4

F(000) = 1452

D_x = 2.872 Mg m⁻³

Mo *K* α radiation, λ = 0.71073 Å

Cell parameters from 11029 reflections

θ = 2.6–28.2°

μ = 14.13 mm⁻¹

T = 223 K

Prism, green

0.25 × 0.22 × 0.14 mm

Data collection

Siemens SMART CCD area-detector
diffractometer

Radiation source: fine-focus sealed tube

Graphite monochromator

ω scans

Absorption correction: multi-scan
(*SADABS*; Bruker, 2008)

T_{min} = 0.139, *T_{max}* = 0.267

11029 measured reflections

4413 independent reflections

4235 reflections with *I* > 2 σ (*I*)

R_{int} = 0.027

θ_{\max} = 28.2°, θ_{\min} = 2.6°

h = -10→11

k = -23→14

l = -17→17

Refinement

Refinement on *F*²

Least-squares matrix: full

R[*F*² > 2 σ (*F*²)] = 0.031

wR(*F*²) = 0.075

S = 1.26

4413 reflections

210 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

$$w = 1/[\sigma^2(F_o^2) + (0.0324P)^2 + 7.030P]$$

where $P = (F_o^2 + 2F_c^2)/3$
 $(\Delta/\sigma)_{\max} = 0.001$

$$\Delta\rho_{\max} = 1.70 \text{ e } \text{\AA}^{-3}$$

$$\Delta\rho_{\min} = -1.62 \text{ e } \text{\AA}^{-3}$$

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.

One of the solvate water molecules is disordered over center of inversion. Moreover, judging by the high anisotropy of thermal motion for this oxygen atom, two contributions of the disorder were considered and the refined partial occupancy factors were 0.20 and 0.30. Both of this contributions were refined anisotropically. However, the hydrogen atoms were not added for this disordered molecule.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$	Occ. (<1)
Re1	0.33803 (3)	0.336279 (13)	0.155287 (17)	0.01817 (7)	
Re2	0.46372 (3)	0.229582 (13)	0.120550 (17)	0.01690 (7)	
Cl1	0.31703 (18)	0.31409 (9)	0.32193 (11)	0.0244 (3)	
Cl2	0.05470 (17)	0.30638 (10)	0.07595 (12)	0.0279 (3)	
Cl3	0.2389 (2)	0.47984 (9)	0.17579 (14)	0.0334 (3)	
Cl4	0.48355 (18)	0.15473 (8)	0.26858 (11)	0.0234 (3)	
Cl5	0.23913 (18)	0.15472 (9)	0.02031 (11)	0.0245 (3)	
Cl6	0.66636 (18)	0.12368 (9)	0.06428 (11)	0.0241 (3)	
O1	0.5745 (5)	0.3823 (2)	0.2274 (3)	0.0223 (8)	
O2	0.6952 (5)	0.2750 (2)	0.1966 (3)	0.0218 (8)	
O3	0.3516 (5)	0.3841 (2)	0.0171 (3)	0.0235 (8)	
O4	0.4770 (5)	0.2789 (2)	-0.0169 (3)	0.0223 (8)	
N1	1.0580 (6)	0.4837 (3)	0.3589 (4)	0.0244 (10)	
H1N	1.1055	0.4574	0.4190	0.037*	
H2N	1.0596	0.5341	0.3737	0.037*	
H3N	1.1168	0.4753	0.3131	0.037*	
N2	0.5515 (7)	0.3709 (4)	-0.2897 (4)	0.0314 (12)	
H4N	0.4435	0.3778	-0.3302	0.047*	
H5N	0.6038	0.3386	-0.3225	0.047*	
H6N	0.6058	0.4162	-0.2791	0.047*	
C1	0.7059 (7)	0.3423 (3)	0.2338 (4)	0.0176 (10)	
C2	0.8792 (7)	0.3729 (3)	0.2871 (5)	0.0202 (11)	
H2A	0.9507	0.3637	0.2416	0.024*	
H2B	0.9287	0.3451	0.3535	0.024*	
C3	0.8795 (7)	0.4581 (3)	0.3108 (5)	0.0245 (12)	
H3A	0.8269	0.4866	0.2454	0.029*	
H3B	0.8143	0.4678	0.3599	0.029*	
C4	0.4226 (7)	0.3459 (4)	-0.0408 (5)	0.0209 (11)	
C5	0.4335 (7)	0.3820 (4)	-0.1409 (4)	0.0204 (11)	

H5A	0.4735	0.4349	-0.1266	0.025*	
H5B	0.3206	0.3832	-0.1932	0.025*	
C6	0.5537 (8)	0.3375 (4)	-0.1853 (5)	0.0281 (13)	
H6A	0.6688	0.3399	-0.1357	0.034*	
H6B	0.5191	0.2836	-0.1945	0.034*	
O5	0.7596 (7)	0.4427 (4)	0.0542 (4)	0.0549 (16)	
H1W	0.8330	0.4072	0.0644	0.082*	
H2W	0.7539	0.4649	-0.0038	0.082*	
O6B	0.464 (7)	0.497 (3)	0.458 (3)	0.072 (15)	0.20
O6A	0.472 (3)	0.4887 (13)	0.4049 (19)	0.051 (6)	0.30

Atomic displacement parameters (Å²)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.01700 (11)	0.01642 (12)	0.02398 (12)	0.00109 (8)	0.01056 (8)	0.00113 (8)
Re2	0.01678 (11)	0.01375 (12)	0.02137 (11)	-0.00057 (8)	0.00761 (8)	0.00023 (8)
Cl1	0.0256 (6)	0.0265 (7)	0.0241 (6)	-0.0002 (6)	0.0121 (5)	0.0022 (6)
Cl2	0.0179 (6)	0.0335 (8)	0.0326 (7)	0.0021 (6)	0.0080 (5)	-0.0022 (6)
Cl3	0.0406 (8)	0.0206 (7)	0.0484 (9)	0.0069 (7)	0.0273 (7)	0.0041 (7)
Cl4	0.0268 (7)	0.0198 (7)	0.0249 (6)	0.0005 (5)	0.0097 (5)	0.0040 (5)
Cl5	0.0212 (6)	0.0259 (7)	0.0257 (6)	-0.0053 (5)	0.0059 (5)	-0.0044 (6)
Cl6	0.0268 (6)	0.0206 (7)	0.0254 (6)	0.0031 (6)	0.0089 (5)	-0.0005 (5)
O1	0.0208 (18)	0.020 (2)	0.029 (2)	-0.0041 (16)	0.0123 (16)	-0.0047 (17)
O2	0.0180 (18)	0.018 (2)	0.028 (2)	0.0003 (16)	0.0055 (16)	-0.0019 (16)
O3	0.0242 (19)	0.021 (2)	0.030 (2)	0.0042 (17)	0.0158 (17)	0.0039 (17)
O4	0.0220 (19)	0.021 (2)	0.026 (2)	-0.0003 (17)	0.0108 (16)	0.0018 (17)
N1	0.029 (2)	0.020 (3)	0.026 (2)	-0.009 (2)	0.010 (2)	-0.001 (2)
N2	0.028 (3)	0.044 (4)	0.028 (3)	0.001 (3)	0.017 (2)	0.000 (2)
C1	0.020 (2)	0.016 (3)	0.019 (2)	-0.003 (2)	0.009 (2)	-0.003 (2)
C2	0.015 (2)	0.019 (3)	0.026 (3)	0.001 (2)	0.006 (2)	-0.002 (2)
C3	0.024 (3)	0.013 (3)	0.035 (3)	0.001 (2)	0.007 (2)	-0.004 (2)
C4	0.019 (2)	0.023 (3)	0.026 (3)	-0.004 (2)	0.015 (2)	-0.002 (2)
C5	0.021 (2)	0.023 (3)	0.022 (3)	0.002 (2)	0.013 (2)	0.007 (2)
C6	0.033 (3)	0.026 (3)	0.030 (3)	0.006 (3)	0.016 (3)	0.004 (3)
O5	0.051 (3)	0.074 (5)	0.044 (3)	0.020 (3)	0.023 (3)	0.025 (3)
O6B	0.10 (4)	0.05 (2)	0.08 (3)	0.03 (2)	0.05 (4)	-0.01 (3)
O6A	0.064 (15)	0.023 (10)	0.057 (14)	0.003 (10)	0.003 (12)	0.003 (11)

Geometric parameters (Å, °)

Re1—O3	2.049 (4)	N2—C6	1.498 (8)
Re1—O1	2.063 (4)	N2—H4N	0.9000
Re1—Re2	2.2494 (3)	N2—H5N	0.9000
Re1—Cl1	2.3037 (14)	N2—H6N	0.9000
Re1—Cl2	2.3197 (14)	C1—C2	1.492 (7)
Re1—Cl3	2.6766 (16)	C2—C3	1.519 (8)
Re2—O2	2.035 (4)	C2—H2A	0.9800
Re2—O4	2.050 (4)	C2—H2B	0.9800

Re2—C14	2.3227 (14)	C3—H3A	0.9800
Re2—C15	2.3291 (14)	C3—H3B	0.9800
Re2—C16	2.7501 (14)	C4—C5	1.498 (7)
O1—C1	1.273 (7)	C5—C6	1.518 (8)
O2—C1	1.267 (7)	C5—H5A	0.9800
O3—C4	1.288 (7)	C5—H5B	0.9800
O4—C4	1.258 (7)	C6—H6A	0.9800
N1—C3	1.490 (7)	C6—H6B	0.9800
N1—H1N	0.9000	O5—H1W	0.8500
N1—H2N	0.9000	O5—H2W	0.8500
N1—H3N	0.9000		
O3—Re1—O1	87.24 (17)	H2N—N1—H3N	109.5
O3—Re1—Re2	89.88 (12)	C6—N2—H4N	109.5
O1—Re1—Re2	89.05 (12)	C6—N2—H5N	109.5
O3—Re1—C11	165.62 (13)	H4N—N2—H5N	109.5
O1—Re1—C11	87.73 (12)	C6—N2—H6N	109.5
Re2—Re1—C11	103.50 (4)	H4N—N2—H6N	109.5
O3—Re1—C12	90.63 (13)	H5N—N2—H6N	109.5
O1—Re1—C12	169.84 (13)	O2—C1—O1	121.7 (5)
Re2—Re1—C12	100.88 (4)	O2—C1—C2	117.3 (5)
C11—Re1—C12	91.96 (5)	O1—C1—C2	121.0 (5)
O3—Re1—C13	79.19 (12)	C1—C2—C3	112.9 (5)
O1—Re1—C13	82.31 (12)	C1—C2—H2A	109.0
Re2—Re1—C13	166.33 (4)	C3—C2—H2A	109.0
C11—Re1—C13	86.79 (5)	C1—C2—H2B	109.0
C12—Re1—C13	87.54 (6)	C3—C2—H2B	109.0
O2—Re2—O4	88.74 (17)	H2A—C2—H2B	107.8
O2—Re2—Re1	90.07 (11)	N1—C3—C2	108.8 (5)
O4—Re2—Re1	89.38 (12)	N1—C3—H3A	109.9
O2—Re2—C14	89.33 (12)	C2—C3—H3A	109.9
O4—Re2—C14	168.37 (12)	N1—C3—H3B	109.9
Re1—Re2—C14	102.09 (4)	C2—C3—H3B	109.9
O2—Re2—C15	165.67 (12)	H3A—C3—H3B	108.3
O4—Re2—C15	88.78 (12)	O4—C4—O3	121.6 (5)
Re1—Re2—C15	104.01 (4)	O4—C4—C5	120.0 (5)
C14—Re2—C15	90.27 (5)	O3—C4—C5	118.3 (5)
O2—Re2—C16	80.51 (12)	C4—C5—C6	110.8 (5)
O4—Re2—C16	80.62 (12)	C4—C5—H5A	109.5
Re1—Re2—C16	166.35 (3)	C6—C5—H5A	109.5
C14—Re2—C16	87.75 (5)	C4—C5—H5B	109.5
C15—Re2—C16	85.16 (5)	C6—C5—H5B	109.5
C1—O1—Re1	119.3 (4)	H5A—C5—H5B	108.1
C1—O2—Re2	119.8 (4)	N2—C6—C5	109.7 (5)
C4—O3—Re1	118.9 (4)	N2—C6—H6A	109.7
C4—O4—Re2	120.1 (4)	C5—C6—H6A	109.7
C3—N1—H1N	109.5	N2—C6—H6B	109.7
C3—N1—H2N	109.5	C5—C6—H6B	109.7

H1N—N1—H2N	109.5	H6A—C6—H6B	108.2
C3—N1—H3N	109.5	H1W—O5—H2W	108.4
H1N—N1—H3N	109.5		
O3—Re1—Re2—O2	89.01 (17)	O4—Re2—O2—C1	86.9 (4)
O1—Re1—Re2—O2	1.77 (16)	Re1—Re2—O2—C1	-2.5 (4)
Cl1—Re1—Re2—O2	-85.66 (12)	Cl4—Re2—O2—C1	-104.6 (4)
Cl2—Re1—Re2—O2	179.63 (13)	Cl5—Re2—O2—C1	166.9 (4)
Cl3—Re1—Re2—O2	52.4 (2)	Cl6—Re2—O2—C1	167.6 (4)
O3—Re1—Re2—O4	0.28 (16)	O1—Re1—O3—C4	87.3 (4)
O1—Re1—Re2—O4	-86.96 (16)	Re2—Re1—O3—C4	-1.7 (4)
Cl1—Re1—Re2—O4	-174.40 (12)	Cl1—Re1—O3—C4	157.0 (4)
Cl2—Re1—Re2—O4	90.90 (12)	Cl2—Re1—O3—C4	-102.6 (4)
Cl3—Re1—Re2—O4	-36.4 (2)	Cl3—Re1—O3—C4	170.0 (4)
O3—Re1—Re2—Cl4	178.34 (13)	O2—Re2—O4—C4	-89.0 (4)
O1—Re1—Re2—Cl4	91.10 (12)	Re1—Re2—O4—C4	1.1 (4)
Cl1—Re1—Re2—Cl4	3.67 (5)	Cl4—Re2—O4—C4	-169.5 (5)
Cl2—Re1—Re2—Cl4	-91.04 (6)	Cl5—Re2—O4—C4	105.2 (4)
Cl3—Re1—Re2—Cl4	141.70 (18)	Cl6—Re2—O4—C4	-169.6 (4)
O3—Re1—Re2—Cl5	-88.31 (13)	Re2—O2—C1—O1	1.8 (7)
O1—Re1—Re2—Cl5	-175.54 (12)	Re2—O2—C1—C2	-179.0 (4)
Cl1—Re1—Re2—Cl5	97.02 (5)	Re1—O1—C1—O2	0.3 (7)
Cl2—Re1—Re2—Cl5	2.31 (6)	Re1—O1—C1—C2	-178.9 (4)
Cl3—Re1—Re2—Cl5	-124.95 (18)	O2—C1—C2—C3	169.6 (5)
O3—Re1—Re2—Cl6	42.93 (18)	O1—C1—C2—C3	-11.2 (8)
O1—Re1—Re2—Cl6	-44.31 (18)	C1—C2—C3—N1	-177.3 (5)
Cl1—Re1—Re2—Cl6	-131.75 (14)	Re2—O4—C4—O3	-2.8 (7)
Cl2—Re1—Re2—Cl6	133.55 (14)	Re2—O4—C4—C5	179.6 (4)
Cl3—Re1—Re2—Cl6	6.3 (2)	Re1—O3—C4—O4	3.1 (7)
O3—Re1—O1—C1	-91.7 (4)	Re1—O3—C4—C5	-179.3 (4)
Re2—Re1—O1—C1	-1.8 (4)	O4—C4—C5—C6	-15.0 (8)
Cl1—Re1—O1—C1	101.8 (4)	O3—C4—C5—C6	167.2 (5)
Cl2—Re1—O1—C1	-169.8 (5)	C4—C5—C6—N2	175.4 (5)
Cl3—Re1—O1—C1	-171.2 (4)		

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>
O5—H1 <i>W</i> ...Cl2 ⁱ	0.85	2.51	3.360 (6)	174
O5—H2 <i>W</i> ...Cl3 ⁱⁱ	0.85	2.50	3.342 (6)	174
N1—H1 <i>N</i> ...Cl6 ⁱⁱⁱ	0.90	2.32	3.202 (5)	167
N1—H2 <i>N</i> ...Cl4 ^{iv}	0.90	2.78	3.396 (6)	127
N1—H2 <i>N</i> ...Cl5 ^{iv}	0.90	2.78	3.557 (5)	145
N1—H2 <i>N</i> ...Cl6 ^{iv}	0.90	2.75	3.410 (6)	131
N1—H3 <i>N</i> ...Cl3 ⁱ	0.90	2.34	3.223 (5)	167
N2—H4 <i>N</i> ...Cl6 ^v	0.90	2.30	3.188 (6)	172
N2—H5 <i>N</i> ...Cl2 ^{vi}	0.90	2.84	3.575 (6)	140
N2—H5 <i>N</i> ...Cl5 ^{vi}	0.90	2.66	3.373 (5)	137

N2—H6 <i>N</i> ···C13 ⁱⁱ	0.90	2.40	3.238 (7)	156
C3—H3 <i>A</i> ···O5	0.98	2.54	3.253 (9)	129
C2—H2 <i>A</i> ···C12 ⁱ	0.98	2.78	3.717 (6)	160

Symmetry codes: (i) $x+1, y, z$; (ii) $-x+1, -y+1, -z$; (iii) $x+1/2, -y+1/2, z+1/2$; (iv) $-x+3/2, y+1/2, -z+1/2$; (v) $x-1/2, -y+1/2, z-1/2$; (vi) $x+1/2, -y+1/2, z-1/2$.