



Crystal structure of the *cis* and *trans* polymorphs of bis[μ -2-(1,3-benzothiazol-2-yl)phenolato]- $\kappa^3N,O:O;\kappa^3O:N,O$ -bis[*fac*-tricarbonylrhenium(I)]¹

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¹This paper is dedicated to the memory of Professor S. K. Mohanlal.

Keywords: crystal structure; rhenium tricarbonyl complexes; polymorphism.

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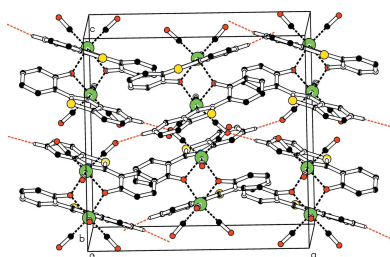
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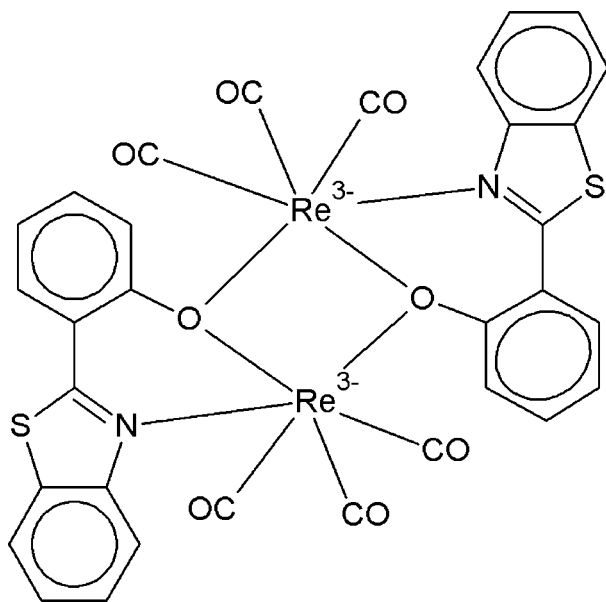
The title dinuclear complex, [Re₂(C₁₃H₈NOS)₂(CO)₆], crystallizes in two polymorphs where the 2-(1,3-benzothiazol-2-yl)phenolate ligands and two carbonyl groups are *trans*- (**I**) or *cis*-arranged (**II**) with respect to the [Re₂O₂(CO)₄] core. Polymorphs **I** and **II** exhibit a crystallographically imposed centre of symmetry and a twofold rotation axis, respectively. The structures may be described as being formed by two octahedrally distorted metal-coordinating units fused through μ -oxido bridges, leading to edge-sharing dimers. The crystal packing is governed by C—H \cdots O hydrogen-bonding interactions, forming chains parallel to the *c* axis in **I** and a three-dimensional network in **II**.

1. Chemical context

Organometallic complexes are regarded as interesting and important compounds owing to their versatile photophysical, photochemical and biological properties. In particular, the importance of the use of metal complexes in medicine began with the discovery of the anti-cancer activity of *cis*-platin (Rosenberg *et al.*, 1965). Since then, attempts to synthesize and characterize novel organometallics with potential pharmaceutical applications remains the main focus of anticancer drug discovery.

While it has been discovered recently that some rhenium-indolato complexes exhibit light-induced anti-cancer activity (Kastl *et al.*, 2013), a number of tricarbonyl-rhenium complexes are well known agents in the field of biomedical imaging (Lo *et al.*, 2010, 2011). Several rhenium(I) tricarbonyl heterocyclic complexes are known to exhibit intense luminescence in the visible region and, owing to their stability to photodecomposition, are promising candidates for solar energy conversion applications (Wallace & Rillema, 1993). In the context of earlier works (Shi *et al.*, 1996; Bradshaw & Westwell, 2004; Potgieter *et al.*, 2012) suggesting benzothiazole derivatives to be promising ligands for rhenium which possess potential usefulness in radiotherapy, the intra- and intermolecular features of the crystal structures of the title compound may well be regarded as relevant. More recently, a host of rhenium-tricarbonyl complexes containing heterocyclic derivatives have been shown to exhibit antimicrobial properties (Kumar *et al.*, 2016). In a recent review, a systematic evaluation of neutral Re^I tricarbonyl complexes was undertaken for their suitability as organic light-emitting diodes (Zhao *et al.*, 2016).





2. Structural commentary

The title compound, $[\text{Re}(\text{CO})_3(\text{L})]_2$ where $\text{L} = 2\text{-(1,3-benzothiazol-2-yl)phenolate}$, crystallizes in two different forms, *viz.* the *trans* form (**I**, Fig. 1) in the triclinic space group $P\bar{1}$ and the

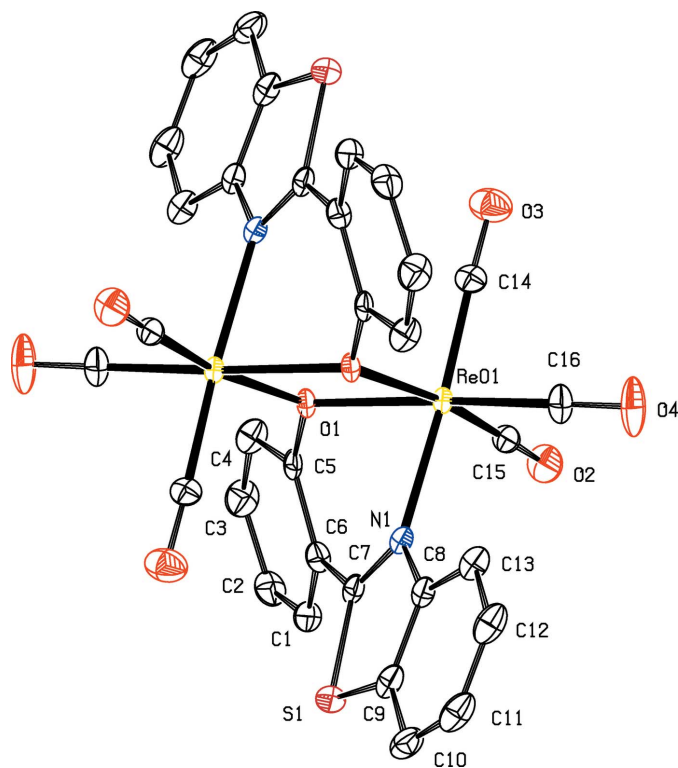


Figure 1

The molecular structure of **I**, with displacement ellipsoids drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by $(1 - x, 1 - y, 1 - z)$. H atoms have been omitted for clarity.

cis form (**II**, Fig. 2) in the orthorhombic space group $Pbca$. The structure of the compound may be described as being composed of two octahedral metal-coordinating units fused through μ -oxido bridges leading to edge-sharing dimers. The presence of the inversion centre in **I** leads to Re–O-bridged centrosymmetric dimeric molecular units. In **II**, dimerization through Re–O bridging is achieved through a twofold rotation axis. In both **I** and **II**, coordination around the rhenium atom is similar, the metal exhibiting a distorted octahedral environment with atoms C16 and N1 occupying the apical sites and atoms C14, C15, O1 and O1ⁱ/O1ⁱⁱ at the equatorial plane [symmetry codes: (i) $1 - x, 1 - y, 1 - z$; (ii) $-x, y, \frac{1}{2} - z$]. The N1–ReO1–O1ⁱ–C9ⁱ torsion angle associated with the Re–O bridging of symmetry-related molecules in *trans* polymorph **I** [$137.1(5)^\circ$] is distinctly different from the corresponding value in the *cis* polymorph **II** [$-59.4(3)^\circ$]. The Re \cdots Re and O1 \cdots O1 separations in the Re₂O₂ core are 3.4799(5) and 2.581(8) Å, and 3.4332(5) and 2.535(4) Å in **I** and **II**, respectively.

The conformation of the ligand in **I** and **II** is significantly different. The dihedral angles between the planar benzothiazole unit and the benzene rings in **I** and **II** are $32.23(18)$ and $22.78(8)^\circ$, respectively. The value observed in **II** closely agrees with that observed in the crystal structure of 2-(4-hydroxyphenyl)benzothiazole [$18.49(6)^\circ$; Teo *et al.*, 1995], which interestingly crystallizes in the same space group. The larger value observed in **I** may be attributed to the ‘flipping’ of the twofold symmetry into an inversion centre.

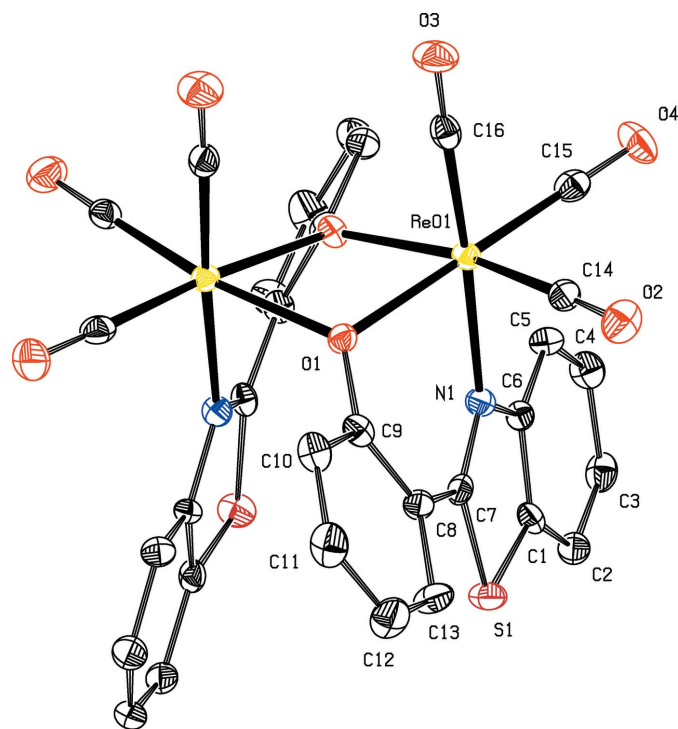


Figure 2

The molecular structure of **II**, with displacement ellipsoids drawn at the 50% probability level. Unlabelled atoms are related to labelled atoms by $(-x, y, \frac{1}{2} - z)$. H atoms have been omitted for clarity.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C13-H13\cdots O3^i$	0.95	2.52	3.276 (8)	137

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

3. Supramolecular features

The crystal structures of **I** and **II** are governed by $C-H\cdots O$ hydrogen bonds which significantly differ in their strengths and the mode of participation of the carbonyl O atoms. In **I**, the O3 atom of the apical carbonyl group $C16=O3$ plays a role in connecting the molecules across inversion centres into a chain along the c axis (Fig. 3, Table 1). In addition, a short $O4\cdots O4^{iii}$ contact [symmetry code: (iii) $-x + 1, -y + 2, -z + 1$] involving centrosymmetrically related carbonyl groups $C15=O4$ [2.792 (10) Å] is present, linking the chains along the b axis to form layers parallel to the bc plane.

In **II**, the oxygen atom of the equatorial carbonyl group $C14=O2$ links the molecules across the glide planes into a three-dimensional network (Fig. 4, Table 2). Similarly to that observed in **I**, a $C-H\cdots O$ hydrogen bond involving the O3 atom of the apical carbonyl group $C16=O3$ is present, which extends along the b axis through translation. Therefore it may be concluded that in both the *trans* and *cis* polymorphs, the mode of participation to the hydrogen-bonding network of the O atom of the apical carbonyl group is through simple translation, while there is a significant ‘switching’ in the choice of the O atoms of the equatorial carbonyl groups. A common feature between the two structures is that one of the three

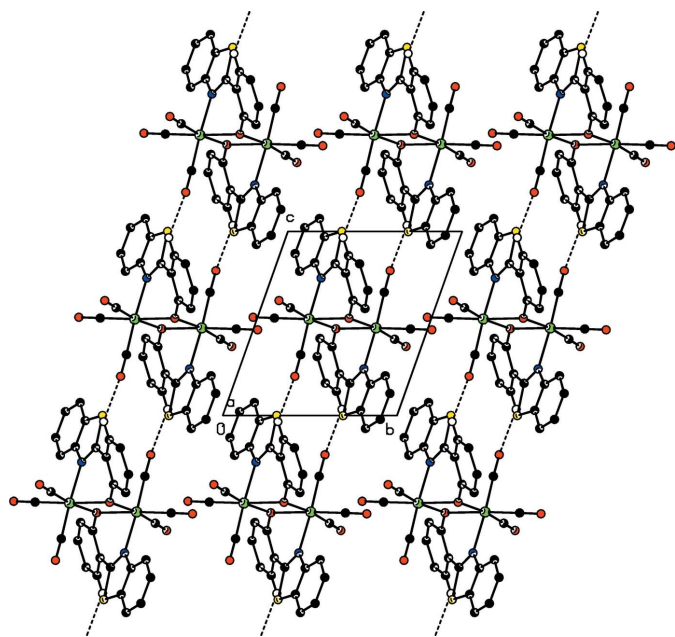


Figure 3
Crystal packing of **I**, showing the formation of molecular chains parallel to the c axis via $C-H\cdots O$ hydrogen bonds (dashed lines). H atoms not involved in hydrogen bonding are omitted.

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

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
$C4-H4\cdots O2^i$	0.93	2.49	3.387 (4)	163
$C2-H2\cdots O3^{ii}$	0.93	2.64	3.467 (5)	149

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

carbonyl groups, namely $C14=O2$ in **I** and $C15=O4$ in **II**, forbids its O atom from participating in the intermolecular interactions.

4. Database survey

A search in the Cambridge structural Database (Version 5.35, November 2014 update; Groom *et al.*, 2016) for μ -oxido bridging dinuclear complexes of rhenium having an octahedral coordination environment similar to that observed in the title compounds (*i.e.* involving three carbonyl C atoms, two oxygens and a nitrogen) was made. The search returned 45 crystal structures with three-dimensional coordinates determined, excluding duplicate structure determinations and having an R factor less than 0.075. Out of these 45 crystal structures, 25 crystallize in the monoclinic, nine in the triclinic, eight in the orthorhombic and three in the trigonal systems. In these compounds, the $Re\cdots Re$ distance ranges from 3.330 to 3.501 Å, the $O\cdots O$ separation within the Re_2O_2 core ranges from 2.485 to 2.701 Å, and $Re-O$ bond lengths from 2.065 to 2.215 Å.

5. Synthesis and crystallization

For **I**:

A mixture of $Re_2(CO)_{10}$ (101.3 mg, 0.1552 mmol), 2-(1,3-benzothiazol-2-yl)phenol (69.7 mg, 0.307 mmol) and 2-phenyl-2-imidazoline (45.8 mg, 0.323 mmol) in toluene (10 ml) in a

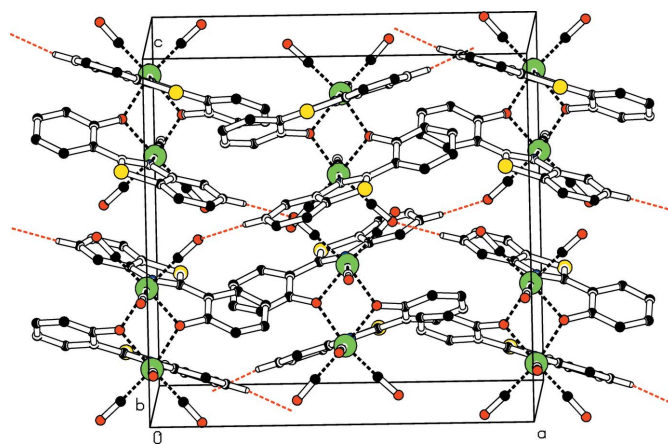


Figure 4
Crystal packing of **II** approximately, viewed along the b axis, showing molecules linked into a three-dimensional network through $C-H\cdots O$ hydrogen bonds (red dashed lines). H atoms not involved in hydrogen bonding are omitted.

Table 3
Experimental details.

	I	II
Crystal data		
Chemical formula	[Re ₂ (C ₁₃ H ₈ NOS) ₂ (CO) ₆]	[Re ₂ (C ₁₃ H ₈ NOS) ₂ (CO) ₆]
<i>M_r</i>	992.99	992.99
Crystal system, space group	Triclinic, <i>P</i> $\bar{1}$	Orthorhombic, <i>Pbcn</i>
Temperature (K)	100	296
<i>a</i> , <i>b</i> , <i>c</i> (Å)	8.9250 (11), 9.7342 (12), 10.0844 (12)	16.1480 (7), 11.6519 (5), 15.6329 (8)
α , β , γ (°)	66.438 (5), 75.636 (5), 63.585 (5)	90, 90, 90
<i>V</i> (Å ³)	716.59 (16)	2941.4 (2)
<i>Z</i>	1	4
Radiation type	Mo <i>K</i> α	Mo <i>K</i> α
μ (mm ⁻¹)	8.64	8.42
Crystal size (mm)	0.28 × 0.18 × 0.15	0.25 × 0.18 × 0.12
Data collection		
Diffractometer	Bruker SMART APEX CCD	Bruker SMART APEX CCD
Absorption correction	Multi-scan (<i>SADABS</i> ; Bruker, 2009)	Multi-scan (<i>SADABS</i> ; Bruker, 2009)
<i>T</i> _{min} , <i>T</i> _{max}	0.168, 0.357	0.18, 0.38
No. of measured, independent and observed [<i>I</i> > 2 σ (<i>I</i>)] reflections	23724, 3325, 3113	11735, 3510, 2943
<i>R</i> _{int}	0.105	0.027
(<i>sin</i> θ / λ) _{max} (Å ⁻¹)	0.654	0.688
Refinement		
<i>R</i> [<i>F</i> ² > 2 σ (<i>F</i> ²)], <i>wR</i> (<i>F</i> ²), <i>S</i>	0.036, 0.088, 1.08	0.027, 0.060, 1.07
No. of reflections	3325	3510
No. of parameters	208	208
H-atom treatment	H-atom parameters constrained	H-atom parameters constrained
$\Delta\rho_{\max}$, $\Delta\rho_{\min}$ (e Å ⁻³)	2.91, -2.74	1.09, -0.95

Computer programs: *APEX2* and *SAINT* (Bruker, 2009), *SHELXS2013* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *PLUTON* (Spek, 2009) and *pubCIF* (Westrip, 2010).

Teflon flask was placed in a steel bomb. The bomb was placed in an oven maintained at 433 K for 48 h and then cooled to 298 K. Pale-yellow crystals were obtained and separated by filtration.

For II:

A mixture of Re₂(CO)₁₀ (101.8 mg, 0.156 mmol), 2-(1,3-benzothiazol-2-yl)phenol (69.9 mg, 0.308 mmol) and 2-(pyridin-4-yl)-1-(2,4,6-trimethylbenzyl)-1*H*-benzo[*d*]imidazole (101.1 mg, 0.309 mmol) in toluene (10 ml) in a Teflon flask was placed in a steel bomb. The bomb was placed in an oven maintained at 433 K for 48 h and then cooled to 298 K. Pale-yellow crystals were obtained and separated by filtration.

6. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 3. In both **I** and **II**, the H atoms were placed in calculated positions (C–H = 0.93–0.97 Å) and were included in the refinement in the riding-model approximation, with *U*_{iso}(H) set at 1.2–1.5*U*_{eq}(C). In **I**, two outliers (9 11 2, 2 2 4) were omitted in the last cycles of refinement.

Acknowledgements

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Crystal structure of the *cis* and *trans* polymorphs of bis[μ -2-(1,3-benzothiazol-2-yl)phenolato]- κ^3 N,O:O; κ^3 O:N,O-bis[*fac*-tricarbonylrhenium(I)]

Maruthupandiyan Priyatharsini, Bhaskaran Shankar, Malaichamy Sathiyendiran, Navaneethakrishnan Srinivasan and Rajaputi Venkatraman Krishnakumar

Computing details

For both compounds, data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINTE* (Bruker, 2009); data reduction: *SAINTE* (Bruker, 2009); program(s) used to solve structure: *SHELXS2013* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *PLUTON* (Spek, 2009); software used to prepare material for publication: *publCIF* (Westrip, 2010).

(I) *trans*-Bis[μ -2-(1,3-benzothiazol-2-yl)phenolato]- κ^3 N,O:O; κ^3 O:N,O-bis[*fac*-tricarbonylrhenium(I)]

Crystal data

[Re₂(C₁₃H₈NOS)₂(CO)₆]

$M_r = 992.99$

Triclinic, *P* $\bar{1}$

$a = 8.9250$ (11) Å

$b = 9.7342$ (12) Å

$c = 10.0844$ (12) Å

$\alpha = 66.438$ (5)°

$\beta = 75.636$ (5)°

$\gamma = 63.585$ (5)°

$V = 716.59$ (16) Å³

$Z = 1$

$F(000) = 468$

$D_x = 2.301$ Mg m⁻³

Mo *K* α radiation, $\lambda = 0.71073$ Å

Cell parameters from 3113 reflections

$\theta = 2.5$ – 27.7 °

$\mu = 8.64$ mm⁻¹

$T = 100$ K

Needle, yellow

$0.28 \times 0.18 \times 0.15$ mm

Data collection

Bruker SMART APEX CCD
diffractometer

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2009)

$T_{\min} = 0.168$, $T_{\max} = 0.357$

23724 measured reflections

3325 independent reflections

3113 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.105$

$\theta_{\max} = 27.7$ °, $\theta_{\min} = 2.5$ °

$h = -11 \rightarrow 11$

$k = -12 \rightarrow 12$

$l = -13 \rightarrow 13$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.036$

$wR(F^2) = 0.088$

$S = 1.08$

3325 reflections

208 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0558P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 2.91$ e Å⁻³

$\Delta\rho_{\min} = -2.74$ e Å⁻³

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

Refinement. Refined as a 2-component perfect inversion twin.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Re01	0.36347 (2)	0.70142 (2)	0.47571 (2)	0.01179 (10)
S1	0.5151 (2)	0.69074 (18)	−0.00066 (15)	0.0204 (3)
O1	0.3841 (5)	0.4733 (5)	0.4716 (4)	0.0136 (7)
O2	0.0115 (6)	0.9108 (6)	0.3751 (5)	0.0285 (11)
O3	0.2004 (7)	0.6662 (7)	0.7869 (5)	0.0329 (12)
O4	0.3450 (7)	1.0258 (6)	0.4663 (8)	0.0413 (14)
N1	0.4733 (6)	0.7154 (5)	0.2528 (5)	0.0143 (9)
C1	0.6280 (7)	0.7795 (7)	0.0280 (6)	0.0206 (12)
C2	0.7395 (9)	0.8452 (8)	−0.0704 (7)	0.0258 (14)
H2	0.7625	0.8448	−0.1674	0.031*
C3	0.8153 (8)	0.9111 (8)	−0.0215 (8)	0.0283 (15)
H3	0.8930	0.9550	−0.0856	0.034*
C4	0.7797 (8)	0.9142 (8)	0.1200 (8)	0.0267 (14)
H4	0.8337	0.9599	0.1507	0.032*
C5	0.6676 (8)	0.8521 (7)	0.2161 (7)	0.0201 (12)
H5	0.6427	0.8563	0.3120	0.024*
C6	0.5912 (7)	0.7828 (6)	0.1703 (6)	0.0164 (11)
C7	0.4244 (7)	0.6635 (6)	0.1754 (6)	0.0158 (10)
C8	0.3038 (7)	0.5852 (7)	0.2262 (6)	0.0173 (11)
C9	0.2952 (7)	0.4856 (6)	0.3742 (6)	0.0144 (10)
C10	0.1877 (8)	0.4040 (7)	0.4184 (7)	0.0214 (12)
H10	0.1813	0.3356	0.5164	0.026*
C11	0.0890 (9)	0.4215 (8)	0.3198 (7)	0.0255 (13)
H11	0.0175	0.3636	0.3513	0.031*
C12	0.0942 (8)	0.5229 (8)	0.1760 (7)	0.0225 (12)
H12	0.0248	0.5369	0.1101	0.027*
C13	0.2023 (8)	0.6028 (7)	0.1310 (6)	0.0208 (12)
H13	0.2073	0.6714	0.0329	0.025*
C14	0.1446 (8)	0.8311 (7)	0.4146 (6)	0.0203 (12)
C15	0.3586 (8)	0.9012 (7)	0.4703 (7)	0.0244 (13)
C16	0.2666 (8)	0.6763 (7)	0.6697 (6)	0.0176 (11)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re01	0.01088 (13)	0.00927 (12)	0.01551 (13)	−0.00340 (8)	−0.00144 (7)	−0.00480 (8)
S1	0.0232 (7)	0.0199 (7)	0.0161 (6)	−0.0080 (6)	0.0009 (5)	−0.0060 (5)
O1	0.0121 (18)	0.0103 (17)	0.0185 (17)	−0.0032 (14)	0.0007 (14)	−0.0074 (14)

O2	0.016 (2)	0.029 (2)	0.032 (2)	0.0011 (18)	-0.0068 (18)	-0.0100 (19)
O3	0.027 (3)	0.042 (3)	0.025 (2)	-0.008 (2)	-0.0042 (19)	-0.012 (2)
O4	0.030 (3)	0.021 (2)	0.083 (4)	-0.010 (2)	-0.001 (3)	-0.029 (3)
N1	0.014 (2)	0.010 (2)	0.017 (2)	-0.0043 (17)	-0.0020 (17)	-0.0023 (16)
C1	0.016 (3)	0.013 (2)	0.024 (3)	-0.004 (2)	-0.001 (2)	-0.001 (2)
C2	0.024 (3)	0.020 (3)	0.022 (3)	-0.007 (2)	0.002 (2)	0.000 (2)
C3	0.016 (3)	0.021 (3)	0.035 (3)	-0.009 (2)	0.002 (2)	0.003 (2)
C4	0.020 (3)	0.020 (3)	0.036 (3)	-0.010 (2)	-0.005 (3)	-0.001 (2)
C5	0.015 (3)	0.017 (3)	0.025 (3)	-0.007 (2)	-0.003 (2)	-0.003 (2)
C6	0.014 (3)	0.011 (2)	0.021 (2)	-0.004 (2)	0.0010 (19)	-0.0040 (19)
C7	0.013 (3)	0.010 (2)	0.019 (2)	-0.0011 (19)	-0.0009 (19)	-0.0040 (19)
C8	0.015 (3)	0.016 (2)	0.020 (2)	-0.005 (2)	0.001 (2)	-0.009 (2)
C9	0.012 (2)	0.009 (2)	0.021 (2)	-0.0012 (19)	-0.0004 (19)	-0.0073 (19)
C10	0.018 (3)	0.019 (3)	0.027 (3)	-0.010 (2)	-0.004 (2)	-0.004 (2)
C11	0.026 (3)	0.025 (3)	0.032 (3)	-0.014 (3)	-0.008 (3)	-0.009 (2)
C12	0.022 (3)	0.024 (3)	0.028 (3)	-0.007 (2)	-0.009 (2)	-0.013 (2)
C13	0.020 (3)	0.018 (3)	0.020 (2)	-0.003 (2)	-0.003 (2)	-0.007 (2)
C14	0.028 (3)	0.017 (3)	0.017 (2)	-0.011 (2)	0.002 (2)	-0.006 (2)
C15	0.021 (3)	0.015 (3)	0.036 (3)	-0.004 (2)	0.000 (2)	-0.012 (2)
C16	0.014 (3)	0.022 (3)	0.018 (2)	-0.005 (2)	-0.006 (2)	-0.006 (2)

Geometric parameters (Å, °)

Re01—C14	1.890 (7)	C2—H2	0.9500
Re01—C15	1.905 (7)	C3—C4	1.392 (11)
Re01—C16	1.898 (6)	C3—H3	0.9500
Re01—O1	2.162 (4)	C4—C5	1.376 (8)
Re01—O1 ⁱ	2.171 (4)	C4—H4	0.9500
Re01—N1	2.194 (5)	C5—C6	1.396 (9)
S1—C1	1.722 (7)	C5—H5	0.9500
S1—C7	1.726 (6)	C7—C8	1.465 (8)
O1—C9	1.348 (7)	C8—C13	1.390 (9)
O1—Re01 ⁱ	2.171 (4)	C8—C9	1.424 (7)
O2—C14	1.157 (8)	C9—C10	1.390 (8)
O3—C16	1.170 (8)	C10—C11	1.400 (9)
O4—C15	1.148 (9)	C10—H10	0.9500
N1—C7	1.317 (8)	C11—C12	1.394 (9)
N1—C6	1.405 (7)	C11—H11	0.9500
C1—C2	1.398 (8)	C12—C13	1.382 (9)
C1—C6	1.400 (8)	C12—H12	0.9500
C2—C3	1.384 (11)	C13—H13	0.9500
C14—Re01—C15	83.9 (3)	C5—C4—H4	119.4
C14—Re01—C16	87.9 (2)	C3—C4—H4	119.4
C15—Re01—C16	86.4 (3)	C4—C5—C6	118.8 (6)
C14—Re01—O1	98.2 (2)	C4—C5—H5	120.6
C15—Re01—O1	176.1 (2)	C6—C5—H5	120.6
C16—Re01—O1	97.0 (2)	C5—C6—C1	119.8 (5)

C14—Re01—O1 ⁱ	170.6 (2)	C5—C6—N1	126.5 (5)
C15—Re01—O1 ⁱ	104.5 (2)	C1—C6—N1	113.7 (5)
C16—Re01—O1 ⁱ	96.8 (2)	N1—C7—C8	125.9 (5)
O1—Re01—O1 ⁱ	73.12 (18)	N1—C7—S1	115.5 (4)
C14—Re01—N1	92.1 (2)	C8—C7—S1	118.6 (5)
C15—Re01—N1	97.5 (2)	C13—C8—C9	119.9 (5)
C16—Re01—N1	176.0 (2)	C13—C8—C7	120.9 (5)
O1—Re01—N1	79.11 (16)	C9—C8—C7	119.1 (5)
O1 ⁱ —Re01—N1	82.68 (16)	O1—C9—C10	120.0 (5)
C1—S1—C7	89.8 (3)	O1—C9—C8	121.6 (5)
C9—O1—Re01	115.5 (3)	C10—C9—C8	118.3 (6)
C9—O1—Re01 ⁱ	126.8 (3)	C9—C10—C11	120.6 (5)
Re01—O1—Re01 ⁱ	106.88 (18)	C9—C10—H10	119.7
C7—N1—C6	110.7 (5)	C11—C10—H10	119.7
C7—N1—Re01	120.7 (4)	C12—C11—C10	120.8 (6)
C6—N1—Re01	128.5 (4)	C12—C11—H11	119.6
C2—C1—C6	121.4 (6)	C10—C11—H11	119.6
C2—C1—S1	128.4 (5)	C13—C12—C11	118.8 (6)
C6—C1—S1	110.2 (4)	C13—C12—H12	120.6
C3—C2—C1	117.7 (6)	C11—C12—H12	120.6
C3—C2—H2	121.2	C12—C13—C8	121.4 (5)
C1—C2—H2	121.2	C12—C13—H13	119.3
C2—C3—C4	121.2 (6)	C8—C13—H13	119.3
C2—C3—H3	119.4	O2—C14—Re01	179.0 (5)
C4—C3—H3	119.4	O4—C15—Re01	175.7 (6)
C5—C4—C3	121.2 (7)	O3—C16—Re01	177.0 (5)
C7—S1—C1—C2	-178.2 (6)	C1—S1—C7—N1	-0.3 (5)
C7—S1—C1—C6	0.3 (4)	C1—S1—C7—C8	-179.6 (4)
C6—C1—C2—C3	1.3 (9)	N1—C7—C8—C13	149.4 (6)
S1—C1—C2—C3	179.7 (5)	S1—C7—C8—C13	-31.3 (7)
C1—C2—C3—C4	-1.0 (9)	N1—C7—C8—C9	-32.5 (8)
C2—C3—C4—C5	-0.1 (10)	S1—C7—C8—C9	146.7 (4)
C3—C4—C5—C6	1.0 (9)	Re01—O1—C9—C10	-128.2 (5)
C4—C5—C6—C1	-0.8 (9)	Re01 ⁱ —O1—C9—C10	92.4 (6)
C4—C5—C6—N1	-179.4 (5)	Re01—O1—C9—C8	49.8 (6)
C2—C1—C6—C5	-0.4 (8)	Re01 ⁱ —O1—C9—C8	-89.7 (6)
S1—C1—C6—C5	-179.1 (4)	C13—C8—C9—O1	-175.9 (5)
C2—C1—C6—N1	178.4 (5)	C7—C8—C9—O1	6.0 (8)
S1—C1—C6—N1	-0.3 (6)	C13—C8—C9—C10	2.0 (9)
C7—N1—C6—C5	178.8 (6)	C7—C8—C9—C10	-176.0 (5)
Re01—N1—C6—C5	2.0 (8)	O1—C9—C10—C11	177.1 (6)
C7—N1—C6—C1	0.1 (7)	C8—C9—C10—C11	-0.9 (9)
Re01—N1—C6—C1	-176.7 (4)	C9—C10—C11—C12	-1.0 (10)
C6—N1—C7—C8	179.4 (5)	C10—C11—C12—C13	1.7 (10)
Re01—N1—C7—C8	-3.5 (8)	C11—C12—C13—C8	-0.6 (10)

C6—N1—C7—S1	0.1 (6)	C9—C8—C13—C12	−1.3 (9)
Re01—N1—C7—S1	177.2 (2)	C7—C8—C13—C12	176.7 (6)

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

Hydrogen-bond geometry (\AA , $^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C13—H13 \cdots O3 ⁱⁱ	0.95	2.52	3.276 (8)	137

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

(II) *cis*-Bis[μ -2-(1,3-benzothiazol-2-yl)phenolato]- $\kappa^3N,O;\kappa^3O:N,O$ -bis[*fac*-tricarbonylrhenium(I)]

Crystal data

$[\text{Re}_2(\text{C}_{13}\text{H}_8\text{NOS})_2(\text{CO})_6]$

$M_r = 992.99$

Orthorhombic, *Pbcn*

$a = 16.1480$ (7) \AA

$b = 11.6519$ (5) \AA

$c = 15.6329$ (8) \AA

$V = 2941.4$ (2) \AA^3

$Z = 4$

$F(000) = 1872$

$D_x = 2.242$ Mg m^{-3}

Mo $K\alpha$ radiation, $\lambda = 0.71073$ \AA

Cell parameters from 2943 reflections

$\theta = 2.9\text{--}28.0^\circ$

$\mu = 8.42$ mm^{-1}

$T = 296$ K

Needle, yellow

$0.25 \times 0.18 \times 0.12$ mm

Data collection

Bruker SMART APEX CCD
diffractometer

ω scans

Absorption correction: multi-scan
(SADABS; Bruker, 2009)

$T_{\min} = 0.18$, $T_{\max} = 0.38$

11735 measured reflections

3510 independent reflections

2943 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.027$

$\theta_{\max} = 29.3^\circ$, $\theta_{\min} = 2.9^\circ$

$h = -12 \rightarrow 22$

$k = -15 \rightarrow 14$

$l = -21 \rightarrow 19$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.027$

$wR(F^2) = 0.060$

$S = 1.07$

3510 reflections

208 parameters

0 restraints

Hydrogen site location: inferred from
neighbouring sites

H-atom parameters constrained

$w = 1/[\sigma^2(F_o^2) + (0.0263P)^2]$

where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.001$

$\Delta\rho_{\max} = 1.09$ e \AA^{-3}

$\Delta\rho_{\min} = -0.95$ e \AA^{-3}

Special details

Geometry. All esds (except the esd in the dihedral angle between two l.s. planes) are estimated using the full covariance matrix. The cell esds are taken into account individually in the estimation of esds in distances, angles and torsion angles; correlations between esds in cell parameters are only used when they are defined by crystal symmetry. An approximate (isotropic) treatment of cell esds is used for estimating esds involving l.s. planes.

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

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Re01	−0.00490 (2)	0.15435 (2)	0.35969 (2)	0.01406 (6)

S1	0.07316 (6)	0.54284 (8)	0.35632 (6)	0.0210 (2)
O1	0.07833 (17)	0.1789 (2)	0.25483 (14)	0.0159 (6)
O2	0.13167 (17)	0.1285 (2)	0.49308 (18)	0.0287 (7)
O3	-0.01421 (17)	-0.1089 (3)	0.34887 (19)	0.0293 (7)
O4	-0.13228 (17)	0.1328 (2)	0.50382 (18)	0.0302 (7)
N1	0.00453 (16)	0.3439 (3)	0.36089 (19)	0.0158 (8)
C1	-0.0277 (2)	0.5344 (3)	0.3923 (2)	0.0164 (8)
C2	-0.0769 (2)	0.6243 (3)	0.4226 (2)	0.0198 (8)
H2	-0.0566	0.6988	0.4265	0.024*
C3	-0.1569 (2)	0.5978 (3)	0.4467 (2)	0.0212 (8)
H3	-0.1914	0.6555	0.4672	0.025*
C4	-0.1866 (2)	0.4861 (3)	0.4407 (3)	0.0219 (9)
H4	-0.2410	0.4705	0.4562	0.026*
C5	-0.1369 (2)	0.3980 (3)	0.4121 (2)	0.0173 (8)
H5	-0.1576	0.3237	0.4083	0.021*
C6	-0.0555 (2)	0.4210 (3)	0.3892 (2)	0.0142 (7)
C7	0.0748 (2)	0.3953 (3)	0.3416 (2)	0.0159 (8)
C8	0.1503 (2)	0.3426 (3)	0.3103 (2)	0.0167 (8)
C9	0.1499 (2)	0.2380 (3)	0.2657 (2)	0.0165 (8)
C10	0.2230 (2)	0.1963 (3)	0.2318 (2)	0.0213 (9)
H10	0.2227	0.1273	0.2019	0.026*
C11	0.2959 (2)	0.2549 (4)	0.2414 (2)	0.0271 (10)
H11	0.3443	0.2262	0.2173	0.033*
C12	0.2980 (2)	0.3564 (3)	0.2867 (3)	0.0294 (10)
H12	0.3478	0.3950	0.2945	0.035*
C13	0.2258 (2)	0.4003 (3)	0.3203 (3)	0.0237 (9)
H13	0.2272	0.4694	0.3501	0.028*
C14	0.0788 (2)	0.1374 (3)	0.4440 (3)	0.0183 (8)
C15	-0.0848 (2)	0.1418 (3)	0.4488 (3)	0.0197 (8)
C16	-0.0108 (2)	-0.0097 (4)	0.3516 (2)	0.0189 (9)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re01	0.01466 (9)	0.01247 (9)	0.01505 (10)	0.00037 (6)	0.00097 (6)	0.00113 (6)
S1	0.0214 (5)	0.0153 (4)	0.0264 (6)	-0.0040 (4)	0.0031 (4)	-0.0005 (4)
O1	0.0149 (13)	0.0178 (12)	0.0151 (14)	-0.0016 (10)	0.0013 (10)	-0.0023 (11)
O2	0.0307 (16)	0.0265 (14)	0.0288 (18)	0.0057 (12)	-0.0083 (14)	-0.0005 (13)
O3	0.0357 (17)	0.0147 (14)	0.038 (2)	-0.0035 (12)	0.0049 (14)	0.0005 (14)
O4	0.0323 (16)	0.0318 (15)	0.0265 (18)	0.0006 (13)	0.0133 (14)	0.0065 (13)
N1	0.0161 (17)	0.0151 (18)	0.0162 (19)	-0.0012 (12)	0.0013 (13)	0.0003 (12)
C1	0.0161 (17)	0.0199 (19)	0.0132 (19)	-0.0010 (15)	0.0031 (15)	0.0030 (17)
C2	0.028 (2)	0.0132 (17)	0.018 (2)	0.0005 (15)	-0.0005 (18)	0.0004 (16)
C3	0.027 (2)	0.0184 (19)	0.018 (2)	0.0071 (16)	-0.0001 (17)	-0.0029 (17)
C4	0.0179 (18)	0.025 (2)	0.022 (2)	0.0023 (16)	0.0024 (17)	0.0004 (17)
C5	0.0166 (18)	0.0140 (17)	0.021 (2)	-0.0012 (14)	-0.0008 (16)	-0.0004 (16)
C6	0.0168 (18)	0.0117 (16)	0.0142 (18)	0.0025 (14)	0.0014 (15)	-0.0007 (15)
C7	0.0168 (18)	0.0191 (19)	0.0120 (19)	-0.0018 (15)	-0.0010 (15)	-0.0024 (16)

C8	0.0138 (17)	0.0193 (19)	0.017 (2)	-0.0018 (15)	0.0004 (15)	-0.0007 (16)
C9	0.0137 (17)	0.0189 (18)	0.017 (2)	0.0020 (15)	-0.0012 (15)	0.0046 (16)
C10	0.0186 (19)	0.025 (2)	0.020 (2)	0.0063 (16)	0.0023 (16)	0.0014 (17)
C11	0.0165 (19)	0.039 (3)	0.026 (2)	0.0049 (18)	0.0050 (17)	-0.004 (2)
C12	0.0152 (19)	0.044 (3)	0.029 (3)	-0.0067 (18)	0.0014 (18)	-0.008 (2)
C13	0.0205 (19)	0.020 (2)	0.030 (2)	-0.0053 (17)	0.0003 (18)	-0.0063 (17)
C14	0.0218 (19)	0.0146 (18)	0.019 (2)	0.0011 (15)	0.0018 (17)	-0.0015 (16)
C15	0.0224 (19)	0.0132 (18)	0.023 (2)	0.0021 (15)	-0.0029 (17)	-0.0010 (16)
C16	0.0155 (18)	0.024 (2)	0.017 (2)	0.0015 (15)	0.0018 (15)	0.0019 (16)

Geometric parameters (Å, °)

Re01—C14	1.898 (4)	C2—H2	0.9300
Re01—C15	1.904 (4)	C3—C4	1.390 (4)
Re01—C16	1.918 (4)	C3—H3	0.9300
Re01—O1	2.139 (2)	C4—C5	1.377 (4)
Re01—O1 ⁱ	2.166 (2)	C4—H4	0.9300
Re01—N1	2.214 (3)	C5—C6	1.389 (5)
S1—C1	1.726 (4)	C5—H5	0.9300
S1—C7	1.735 (4)	C7—C8	1.450 (5)
O1—C9	1.355 (4)	C8—C13	1.401 (5)
O1—Re01 ⁱ	2.166 (2)	C8—C9	1.404 (5)
O2—C14	1.153 (4)	C9—C10	1.382 (5)
O3—C16	1.158 (5)	C10—C11	1.370 (5)
O4—C15	1.157 (5)	C10—H10	0.9300
N1—C7	1.318 (4)	C11—C12	1.378 (6)
N1—C6	1.393 (4)	C11—H11	0.9300
C1—C6	1.396 (5)	C12—C13	1.377 (5)
C1—C2	1.398 (5)	C12—H12	0.9300
C2—C3	1.380 (5)	C13—H13	0.9300
C14—Re01—C15	88.10 (18)	C5—C4—H4	119.4
C14—Re01—C16	88.70 (15)	C3—C4—H4	119.4
C15—Re01—C16	86.45 (14)	C4—C5—C6	119.4 (3)
C14—Re01—O1	95.63 (14)	C4—C5—H5	120.3
C15—Re01—O1	175.25 (12)	C6—C5—H5	120.3
C16—Re01—O1	96.54 (13)	C5—C6—N1	128.0 (3)
C14—Re01—O1 ⁱ	167.74 (13)	C5—C6—C1	118.5 (3)
C15—Re01—O1 ⁱ	104.12 (14)	N1—C6—C1	113.5 (3)
C16—Re01—O1 ⁱ	92.87 (13)	N1—C7—C8	127.5 (3)
O1—Re01—O1 ⁱ	72.12 (12)	N1—C7—S1	114.0 (3)
C14—Re01—N1	92.80 (12)	C8—C7—S1	118.5 (3)
C15—Re01—N1	96.72 (12)	C13—C8—C9	118.4 (3)
C16—Re01—N1	176.53 (14)	C13—C8—C7	119.4 (3)
O1—Re01—N1	80.20 (10)	C9—C8—C7	122.1 (3)
O1 ⁱ —Re01—N1	85.00 (10)	O1—C9—C10	120.1 (3)
C1—S1—C7	90.05 (17)	O1—C9—C8	120.5 (3)
C9—O1—Re01	120.5 (2)	C10—C9—C8	119.4 (3)

C9—O1—Re01 ⁱ	129.6 (2)	C11—C10—C9	121.1 (4)
Re01—O1—Re01 ⁱ	105.77 (11)	C11—C10—H10	119.4
C7—N1—C6	112.3 (3)	C9—C10—H10	119.4
C7—N1—Re01	120.7 (2)	C10—C11—C12	120.3 (4)
C6—N1—Re01	126.7 (2)	C10—C11—H11	119.8
C6—C1—C2	122.6 (3)	C12—C11—H11	119.8
C6—C1—S1	110.2 (3)	C13—C12—C11	119.6 (4)
C2—C1—S1	127.2 (3)	C13—C12—H12	120.2
C3—C2—C1	117.2 (3)	C11—C12—H12	120.2
C3—C2—H2	121.4	C12—C13—C8	121.0 (3)
C1—C2—H2	121.4	C12—C13—H13	119.5
C2—C3—C4	120.9 (3)	C8—C13—H13	119.5
C2—C3—H3	119.6	O2—C14—Re01	177.6 (3)
C4—C3—H3	119.6	O4—C15—Re01	178.7 (3)
C5—C4—C3	121.3 (3)	O3—C16—Re01	178.3 (3)
C7—S1—C1—C6	-1.5 (3)	C1—S1—C7—N1	1.0 (3)
C7—S1—C1—C2	177.1 (4)	C1—S1—C7—C8	-179.2 (3)
C6—C1—C2—C3	-2.7 (6)	N1—C7—C8—C13	-158.5 (4)
S1—C1—C2—C3	178.8 (3)	S1—C7—C8—C13	21.7 (5)
C1—C2—C3—C4	0.0 (6)	N1—C7—C8—C9	25.2 (6)
C2—C3—C4—C5	1.2 (6)	S1—C7—C8—C9	-154.6 (3)
C3—C4—C5—C6	0.2 (6)	Re01—O1—C9—C10	135.2 (3)
C4—C5—C6—N1	178.4 (4)	Re01 ⁱ —O1—C9—C10	-71.1 (4)
C4—C5—C6—C1	-2.8 (5)	Re01—O1—C9—C8	-45.7 (4)
C7—N1—C6—C5	177.8 (4)	Re01 ⁱ —O1—C9—C8	108.0 (3)
Re01—N1—C6—C5	-9.0 (5)	C13—C8—C9—O1	179.7 (3)
C7—N1—C6—C1	-1.0 (4)	C7—C8—C9—O1	-4.0 (5)
Re01—N1—C6—C1	172.2 (2)	C13—C8—C9—C10	-1.2 (5)
C2—C1—C6—C5	4.2 (6)	C7—C8—C9—C10	175.1 (3)
S1—C1—C6—C5	-177.2 (3)	O1—C9—C10—C11	179.5 (3)
C2—C1—C6—N1	-176.9 (3)	C8—C9—C10—C11	0.4 (6)
S1—C1—C6—N1	1.8 (4)	C9—C10—C11—C12	1.1 (6)
C6—N1—C7—C8	-180.0 (4)	C10—C11—C12—C13	-1.8 (6)
Re01—N1—C7—C8	6.3 (5)	C11—C12—C13—C8	0.9 (6)
C6—N1—C7—S1	-0.2 (4)	C9—C8—C13—C12	0.5 (6)
Re01—N1—C7—S1	-173.91 (16)	C7—C8—C13—C12	-175.9 (4)

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

Hydrogen-bond geometry ($\text{\AA}, ^\circ$)

$D-H\cdots A$	$D-H$	$H\cdots A$	$D\cdots A$	$D-H\cdots A$
C4—H4 \cdots O2 ⁱⁱ	0.93	2.49	3.387 (4)	163
C2—H2 \cdots O3 ⁱⁱⁱ	0.93	2.64	3.467 (5)	149

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