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Tricarbonylchlorido(6'7'-dihydro-5'H-spiro[cyclopentane-1,6'-dipyrido-[3,2-d:2',3'-f][1,3]diazepine]- κ^2 N1,N11)-rhenium(I)

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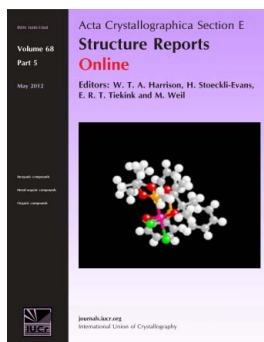
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Tricarbonylchlorido(6',7'-dihydro-5'*H*-spiro[cyclopentane-1,6'-dipyrido[3,2-*d*:2',3'-*f*][1,3]diazepine]- κ^2 N¹,N¹¹)rhenium(I)

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Acta Cryst. (2013). E69, m526

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Tricarbonylchlorido(6',7'-dihydro-5'H-spiro[cyclopentane-1,6'-dipyrido[3,2-d:2',3'-f][1,3]diazepine]- κ^2 N¹,N¹¹)-rhenium(I)

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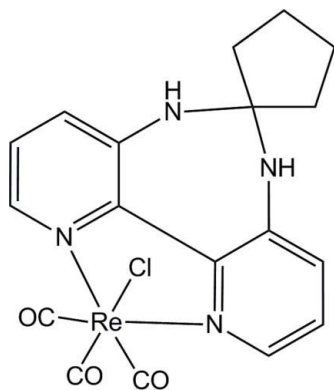
Received 7 August 2013; accepted 16 August 2013

Key indicators: single-crystal X-ray study; $T = 150$ K; mean $\sigma(\text{C}-\text{C}) = 0.004$ Å; R factor = 0.037; wR factor = 0.075; data-to-parameter ratio = 44.1.

In the title compound, $[\text{ReCl}(\text{C}_{15}\text{H}_{16}\text{N}_4)(\text{CO})_3]$, the Re^{I} ion is coordinated in a distorted octahedral geometry by one Cl atom, two N atoms of the bidentate ligand and three carbonyl groups. The cyclopentane group is orientated in a *transoid* fashion with respect to the chloride ligand. The dihedral angle between the pyridine rings is 10.91 (12)°. In the crystal, $\text{N}-\text{H}\cdots\text{Cl}$ hydrogen bonds link complex molecules, forming a two-dimensional network parallel to (001).

Related literature

For a review of the photophysical properties of Re-poly-pyridyl complexes, see: Coleman *et al.* (2008). For the synthesis of $[\text{Re}(3,3'\text{-diamino-2,2'\text{-bipyridine})(\text{CO})_3\text{Cl}]$ and for the preparation of oxo-steroid derivatives of $[\text{Re}(3,3'\text{-diamino-2,2'\text{-bipyridine})(\text{CO})_3\text{Cl}]$, see: Bullock *et al.* (2012). For the reaction of $[\text{Re}(3,3'\text{-diamino-2,2'\text{-bipyridine})(\text{CO})_3\text{Cl}]$ with ketones, see: Clayton *et al.* (2008). For the structure of the cyclohexane analog of the title compound, see: Clegg *et al.* (2013).



Experimental

Crystal data

$[\text{ReCl}(\text{C}_{15}\text{H}_{16}\text{N}_4)(\text{CO})_3]$	$V = 3612.0$ (2) Å ³
$M_r = 558.00$	$Z = 8$
Orthorhombic, $Pbca$	Mo $K\alpha$ radiation
$a = 12.1162$ (5) Å	$\mu = 6.90$ mm ⁻¹
$b = 11.9638$ (5) Å	$T = 150$ K
$c = 24.9181$ (9) Å	$0.50 \times 0.50 \times 0.20$ mm

Data collection

Bruker APEXII CCD diffractometer	45382 measured reflections
Absorption correction: multi-scan (SADABS; Bruker, 2009)	10757 independent reflections
$T_{\text{min}} = 0.16$, $T_{\text{max}} = 0.34$	7941 reflections with $I > 2\sigma(I)$
	$R_{\text{int}} = 0.053$

Refinement

$R[F^2 > 2\sigma(F^2)] = 0.037$	244 parameters
$wR(F^2) = 0.075$	H-atom parameters constrained
$S = 1.04$	$\Delta\rho_{\text{max}} = 2.28$ e Å ⁻³
10757 reflections	$\Delta\rho_{\text{min}} = -5.29$ e Å ⁻³

Table 1

Hydrogen-bond geometry (Å, °).

$D-\text{H}\cdots A$	$D-\text{H}$	$\text{H}\cdots A$	$D\cdots A$	$D-\text{H}\cdots A$
$\text{N3}-\text{H3A}\cdots\text{Cl1}^{\text{i}}$	0.88	2.53	3.363 (3)	158
$\text{N4}-\text{H4}\cdots\text{Cl1}^{\text{ii}}$	0.88	2.65	3.419 (2)	147

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

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for publication: *OLEX2*.

The authors wish to thank the University of Huddersfield for financial support.

Supplementary data and figures for this paper are available from the IUCr electronic archives (Reference: LH5644).

References

- Bruker (2009). *APEX2*, *SAINT* and *SADABS*. Bruker AXS Inc., Madison, Wisconsin, USA.
- Bullock, S., Hallett, A. J., Harding, L. P., Higginson, J. J., Piela, S. A. F., Pope, S. J. A. & Rice, C. R. (2012). *Dalton Trans.* **41**, 14690–14696.
- Clayton, H. J., Harding, L. P., Irvine, J. P., Jeffery, J. C., Riis-Johannessen, T., Laws, A. P., Rice, C. R. & Whitehead, M. (2008). *Chem. Commun.* pp. 108–110.
- Clegg, O. R., Harding, L. P., Miller, J. W. & Rice, C. R. (2013). *Acta Cryst.* **E69**, m527.
- Coleman, A., Brennan, C., Vos, J. G. & Pryce, M. T. (2008). *Coord. Chem. Rev.* **252**, 2585–2595.
- Dolomanov, O. V., Bourhis, L. J., Gildea, R. J., Howard, J. A. K. & Puschmann, H. (2009). *J. Appl. Cryst.* **42**, 339–341.
- Sheldrick, G. M. (2008). *Acta Cryst.* **A64**, 112–122.

supplementary materials

Acta Cryst. (2013). E69, m526 [doi:10.1107/S1600536813023076]

Tricarbonylchlorido(6',7'-dihydro-5'H-spiro[cyclopentane-1,6'-dipyrido[3,2-d':2',3'-f][1,3]diazepine]- κ^2 N¹,N¹¹)rhenium(I)

Oliver R. Clegg, Lindsay P. Harding, John W. Miller and Craig R. Rice

1. Comment

The title complex was prepared as part of a larger study into conjugation of [Re(3,3'-diamino-2,2'-bipyridine)(CO)₃Cl] with oxo-steroids to form luminescent derivatives (Bullock *et al.* 2012). These steroids contain a cyclopentyl ring (ring D) with a ketone group in the 17-position; therefore, cyclopentanone was used as a model compound to examine the potential reactivity of such steroids with the rhenium complex. The photophysical properties of Re-polypyridyl complexes have been studied (Coleman *et al.*, 2008) as well as the reaction of [Re(3,3'-diamino-2,2'-bipyridine)(CO)₃Cl] with ketones (Clayton *et al.*, 2008).

Single-crystal X-ray analysis of the product gave the structure shown in Fig. 1. The rhenium centre adopts a distorted octahedral coordination geometry and is coordinated by two nitrogen atoms from 3,3'-diamino-2,2'-bipyridyl and two carbonyl ligands in the equatorial positions (Re—N distances 2.158 (2) - 2.169 (2) Å, Re—C distances 1.924 (3) - 1.925 (3) Å). Carbonyl and chloride ligands occupy the axial positions (Re—C distance 1.891 (3) Å, Re—Cl distance 2.5046 (6) Å). The cyclopentyl ring is orientated in a *trans*-oid fashion with respect to the chloride ligand on the rhenium centre. In the crystal, N—H...O hydrogen bonds (see, Table 1) link complex molecules to form a two-dimensional network parallel (001).

A similar compound has been prepared using cyclohexanone instead of cyclopentanone. This compound is essentially isostructural with the compound reported here (Clegg *et al.* 2013).

2. Experimental

To a solution of [Re(3,3'-diamino-2,2'-bipyridine)(CO)₃Cl] in dichloromethane was added cyclopentanone (10 μ L, *ca* 2 eq.) and a few grains of camphorsulfonic acid. The solution was stirred at room temperature for 2 h. The resulting precipitate was filtered *in vacuo*, washed with dichloromethane and dried, affording the product as a yellow solid. Slow evaporation of an acetonitrile solution of the complex gave yellow crystals suitable for X-ray analysis.

3. Refinement

All non-hydrogen atoms were refined anisotropically. Hydrogen atoms on *sp*² and *sp*³ carbons were placed in calculated positions (C—H = 0.95 - 0.99 Å) and refined with riding constraints and with isotropic displacement parameters 1.2 *x* their parent carbon atoms. H atoms on the nitrogen atoms were treated similarly with N—H = 0.88 Å.

Computing details

Data collection: *APEX2* (Bruker, 2009); cell refinement: *SAINT* (Bruker, 2009); data reduction: *SAINT* (Bruker, 2009); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL97* (Sheldrick, 2008); molecular graphics: *OLEX2* (Dolomanov *et al.*, 2009); software used to prepare material for

publication: OLEX2 (Dolomanov *et al.*, 2009).

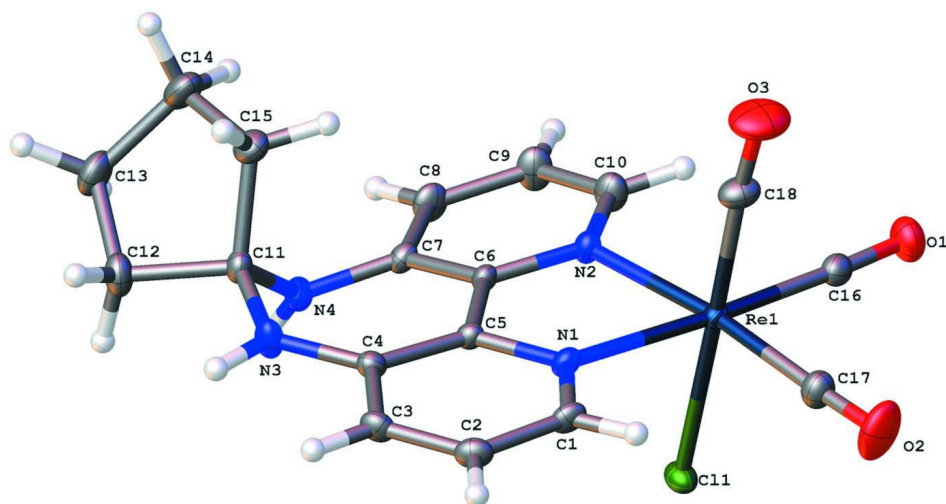


Figure 1

The molecular structure of the title compound with displacement ellipsoids shown for non-H atoms at the 50% probability level.

Tricarbonylchlorido(6',7'-dihydro-5'H-spiro[cyclopentane-1,6'-dipyrido[3,2-d:2',3'-f][1,3]diazepine]- κ^2N^1,N^{11})rhenium(I)

Crystal data

[ReCl(C₁₅H₁₆N₄)(CO)₃]

$M_r = 558.00$

Orthorhombic, *Pbca*

$a = 12.1162 (5) \text{ \AA}$

$b = 11.9638 (5) \text{ \AA}$

$c = 24.9181 (9) \text{ \AA}$

$V = 3612.0 (2) \text{ \AA}^3$

$Z = 8$

$F(000) = 2144$

$D_x = 2.052 \text{ Mg m}^{-3}$

Mo $K\alpha$ radiation, $\lambda = 0.71073 \text{ \AA}$

Cell parameters from 9921 reflections

$\theta = 2.9\text{--}39.3^\circ$

$\mu = 6.90 \text{ mm}^{-1}$

$T = 150 \text{ K}$

Block, yellow

$0.50 \times 0.50 \times 0.20 \text{ mm}$

Data collection

Bruker APEXII CCD

diffractometer

Graphite monochromator

Detector resolution: $8.3333 \text{ pixels mm}^{-1}$

φ and ω scans

Absorption correction: multi-scan

(*SADABS*; Bruker, 2009)

$T_{\min} = 0.16$, $T_{\max} = 0.34$

45382 measured reflections

10757 independent reflections

7941 reflections with $I > 2\sigma(I)$

$R_{\text{int}} = 0.053$

$\theta_{\max} = 39.4^\circ$, $\theta_{\min} = 2.3^\circ$

$h = -21 \rightarrow 20$

$k = -21 \rightarrow 13$

$l = -44 \rightarrow 41$

Refinement

Refinement on F^2

Least-squares matrix: full

$R[F^2 > 2\sigma(F^2)] = 0.037$

$wR(F^2) = 0.075$

$S = 1.04$

10757 reflections

244 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
 $w = 1/[\sigma^2(F_o^2) + (0.0093P)^2 + 8.4209P]$
 where $P = (F_o^2 + 2F_c^2)/3$

$(\Delta/\sigma)_{\max} = 0.002$
 $\Delta\rho_{\max} = 2.28 \text{ e } \text{\AA}^{-3}$
 $\Delta\rho_{\min} = -5.29 \text{ e } \text{\AA}^{-3}$

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.

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

	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Re1	0.454601 (7)	0.268496 (9)	0.14683 (4)	0.01301 (3)
Cl1	0.61326 (5)	0.24039 (6)	0.08383 (3)	0.01986 (12)
N1	0.39870 (16)	0.38723 (19)	0.08695 (8)	0.0143 (4)
N2	0.53943 (17)	0.4231 (2)	0.16399 (9)	0.0156 (4)
N3	0.4418 (2)	0.6806 (2)	0.04288 (10)	0.0216 (5)
H3A	0.4175	0.7169	0.0145	0.026*
N4	0.61142 (17)	0.6801 (2)	0.09197 (9)	0.0170 (4)
H4	0.6735	0.6957	0.0752	0.02*
O1	0.55829 (19)	0.1241 (2)	0.23572 (10)	0.0301 (5)
O2	0.3300 (2)	0.0584 (2)	0.10992 (12)	0.0425 (7)
O3	0.2621 (2)	0.3006 (3)	0.22352 (12)	0.0467 (7)
C1	0.3219 (2)	0.3577 (2)	0.05113 (10)	0.0177 (5)
H1	0.2898	0.2854	0.0533	0.021*
C2	0.2877 (2)	0.4305 (3)	0.01061 (10)	0.0191 (5)
H2	0.2351	0.4073	-0.0154	0.023*
C3	0.3314 (2)	0.5352 (3)	0.00911 (10)	0.0182 (5)
H3	0.3084	0.5854	-0.0182	0.022*
C4	0.41063 (19)	0.5711 (2)	0.04738 (9)	0.0149 (4)
C5	0.44682 (18)	0.4917 (2)	0.08588 (9)	0.0131 (4)
C6	0.53279 (19)	0.5069 (2)	0.12741 (9)	0.0133 (4)
C7	0.60801 (19)	0.5965 (2)	0.13002 (10)	0.0153 (4)
C8	0.6856 (2)	0.5981 (3)	0.17185 (12)	0.0211 (5)
H8	0.737	0.6578	0.1742	0.025*
C9	0.6876 (2)	0.5138 (3)	0.20945 (12)	0.0242 (6)
H9	0.7393	0.515	0.2381	0.029*
C10	0.6127 (2)	0.4277 (3)	0.20445 (11)	0.0219 (5)
H10	0.6129	0.3697	0.2305	0.026*
C11	0.5101 (2)	0.7430 (2)	0.07960 (11)	0.0173 (5)
C12	0.5445 (3)	0.8552 (3)	0.05556 (13)	0.0249 (6)
H12A	0.481	0.8927	0.0382	0.03*
H12B	0.604	0.8453	0.0287	0.03*
C13	0.5857 (3)	0.9228 (3)	0.10410 (14)	0.0303 (7)
H13A	0.5668	1.0029	0.1	0.036*

H13B	0.6667	0.9157	0.108	0.036*
C14	0.5256 (3)	0.8717 (3)	0.15337 (13)	0.0319 (7)
H14A	0.5796	0.84	0.179	0.038*
H14B	0.4815	0.9295	0.1721	0.038*
C15	0.4503 (2)	0.7797 (3)	0.13103 (13)	0.0228 (5)
H15A	0.3758	0.8091	0.1229	0.027*
H15B	0.4435	0.7168	0.1567	0.027*
C16	0.5196 (2)	0.1764 (3)	0.20172 (11)	0.0189 (5)
C17	0.3787 (2)	0.1363 (3)	0.12295 (12)	0.0228 (5)
C18	0.3353 (2)	0.2923 (3)	0.19437 (12)	0.0238 (6)

Atomic displacement parameters (\AA^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re1	0.01383 (4)	0.01096 (5)	0.01425 (4)	0.00013 (3)	0.00075 (3)	-0.00102 (3)
Cl1	0.0199 (2)	0.0217 (3)	0.0180 (2)	0.0056 (2)	0.00425 (19)	0.0036 (2)
N1	0.0139 (7)	0.0123 (10)	0.0166 (8)	0.0004 (7)	-0.0027 (6)	-0.0025 (7)
N2	0.0174 (8)	0.0125 (10)	0.0170 (8)	0.0000 (7)	-0.0035 (7)	0.0010 (7)
N3	0.0285 (11)	0.0178 (13)	0.0183 (9)	-0.0037 (9)	-0.0082 (8)	0.0047 (8)
N4	0.0163 (8)	0.0146 (11)	0.0202 (9)	-0.0006 (7)	0.0012 (7)	0.0025 (8)
O1	0.0363 (12)	0.0265 (13)	0.0275 (11)	0.0024 (9)	-0.0035 (9)	0.0085 (10)
O2	0.0472 (15)	0.0211 (14)	0.0590 (18)	-0.0087 (11)	-0.0202 (13)	-0.0053 (12)
O3	0.0349 (13)	0.058 (2)	0.0469 (16)	0.0043 (13)	0.0198 (12)	-0.0117 (15)
C1	0.0174 (9)	0.0164 (13)	0.0193 (10)	-0.0012 (8)	-0.0041 (8)	-0.0063 (9)
C2	0.0177 (10)	0.0242 (15)	0.0156 (10)	0.0013 (9)	-0.0036 (8)	-0.0039 (9)
C3	0.0194 (10)	0.0227 (15)	0.0126 (9)	-0.0005 (9)	-0.0032 (8)	0.0007 (9)
C4	0.0163 (9)	0.0160 (13)	0.0125 (9)	-0.0004 (8)	-0.0002 (7)	-0.0004 (8)
C5	0.0143 (8)	0.0128 (11)	0.0121 (8)	0.0008 (7)	-0.0019 (7)	-0.0009 (7)
C6	0.0166 (9)	0.0100 (11)	0.0132 (9)	-0.0001 (7)	-0.0015 (7)	-0.0010 (7)
C7	0.0157 (9)	0.0118 (12)	0.0183 (10)	0.0010 (8)	-0.0021 (8)	-0.0019 (8)
C8	0.0201 (10)	0.0182 (14)	0.0250 (12)	-0.0054 (9)	-0.0067 (9)	-0.0023 (10)
C9	0.0241 (12)	0.0233 (16)	0.0252 (13)	-0.0047 (10)	-0.0120 (10)	0.0025 (11)
C10	0.0249 (12)	0.0205 (15)	0.0205 (11)	-0.0004 (10)	-0.0085 (9)	0.0022 (10)
C11	0.0217 (10)	0.0126 (13)	0.0176 (10)	0.0001 (9)	-0.0019 (8)	0.0020 (8)
C12	0.0297 (13)	0.0180 (15)	0.0271 (13)	-0.0026 (11)	-0.0042 (11)	0.0073 (11)
C13	0.0381 (17)	0.0156 (15)	0.0373 (17)	-0.0025 (12)	-0.0048 (13)	-0.0031 (13)
C14	0.0504 (19)	0.0193 (16)	0.0259 (15)	0.0032 (14)	-0.0048 (13)	-0.0067 (12)
C15	0.0252 (12)	0.0195 (16)	0.0237 (12)	0.0053 (10)	0.0018 (9)	-0.0016 (10)
C16	0.0207 (10)	0.0160 (14)	0.0200 (11)	-0.0019 (9)	0.0019 (8)	-0.0006 (9)
C17	0.0267 (12)	0.0186 (15)	0.0232 (12)	-0.0020 (10)	-0.0037 (10)	0.0000 (10)
C18	0.0208 (11)	0.0261 (17)	0.0245 (12)	0.0018 (10)	0.0032 (9)	-0.0062 (11)

Geometric parameters (\AA , $^\circ$)

Re1—C18	1.891 (3)	C3—H3	0.95
Re1—C17	1.924 (3)	C4—C5	1.419 (4)
Re1—C16	1.925 (3)	C5—C6	1.479 (3)
Re1—N2	2.158 (2)	C6—C7	1.409 (4)
Re1—N1	2.169 (2)	C7—C8	1.404 (4)
Re1—Cl1	2.5046 (6)	C8—C9	1.377 (4)

N1—C1	1.337 (3)	C8—H8	0.95
N1—C5	1.380 (3)	C9—C10	1.378 (4)
N2—C10	1.345 (3)	C9—H9	0.95
N2—C6	1.358 (3)	C10—H10	0.95
N3—C4	1.368 (4)	C11—C12	1.528 (4)
N3—C11	1.442 (4)	C11—C15	1.536 (4)
N3—H3A	0.88	C12—C13	1.538 (5)
N4—C7	1.379 (4)	C12—H12A	0.99
N4—C11	1.473 (3)	C12—H12B	0.99
N4—H4	0.88	C13—C14	1.553 (5)
O1—C16	1.153 (4)	C13—H13A	0.99
O2—C17	1.149 (4)	C13—H13B	0.99
O3—C18	1.150 (4)	C14—C15	1.535 (5)
C1—C2	1.396 (4)	C14—H14A	0.99
C1—H1	0.95	C14—H14B	0.99
C2—C3	1.361 (4)	C15—H15A	0.99
C2—H2	0.95	C15—H15B	0.99
C3—C4	1.420 (3)		
C18—Rel—C17	87.23 (13)	N4—C7—C8	118.7 (2)
C18—Rel—C16	87.36 (13)	N4—C7—C6	122.7 (2)
C17—Rel—C16	86.85 (12)	C8—C7—C6	118.6 (2)
C18—Rel—N2	96.39 (12)	C9—C8—C7	120.5 (3)
C17—Rel—N2	173.32 (11)	C9—C8—H8	119.8
C16—Rel—N2	98.89 (10)	C7—C8—H8	119.8
C18—Rel—N1	95.38 (11)	C8—C9—C10	118.3 (2)
C17—Rel—N1	100.15 (11)	C8—C9—H9	120.8
C16—Rel—N1	172.58 (10)	C10—C9—H9	120.8
N2—Rel—N1	73.97 (8)	N2—C10—C9	122.2 (3)
C18—Rel—C11	179.06 (11)	N2—C10—H10	118.9
C17—Rel—C11	93.60 (9)	C9—C10—H10	118.9
C16—Rel—C11	93.12 (8)	N3—C11—N4	110.3 (2)
N2—Rel—C11	82.74 (6)	N3—C11—C12	111.3 (2)
N1—Rel—C11	84.05 (6)	N4—C11—C12	107.7 (2)
C1—N1—C5	121.4 (2)	N3—C11—C15	114.0 (2)
C1—N1—Rel	120.25 (19)	N4—C11—C15	111.4 (2)
C5—N1—Rel	118.36 (15)	C12—C11—C15	101.8 (2)
C10—N2—C6	120.8 (2)	C11—C12—C13	104.0 (2)
C10—N2—Rel	119.9 (2)	C11—C12—H12A	111.0
C6—N2—Rel	118.13 (16)	C13—C12—H12A	111.0
C4—N3—C11	127.0 (2)	C11—C12—H12B	111.0
C4—N3—H3A	116.5	C13—C12—H12B	111.0
C11—N3—H3A	116.5	H12A—C12—H12B	109.0
C7—N4—C11	119.3 (2)	C12—C13—C14	105.2 (3)
C7—N4—H4	120.3	C12—C13—H13A	110.7
C11—N4—H4	120.3	C14—C13—H13A	110.7
N1—C1—C2	121.6 (3)	C12—C13—H13B	110.7
N1—C1—H1	119.2	C14—C13—H13B	110.7
C2—C1—H1	119.2	H13A—C13—H13B	108.8

C3—C2—C1	118.6 (2)	C15—C14—C13	105.9 (3)
C3—C2—H2	120.7	C15—C14—H14A	110.5
C1—C2—H2	120.7	C13—C14—H14A	110.5
C2—C3—C4	121.6 (2)	C15—C14—H14B	110.5
C2—C3—H3	119.2	C13—C14—H14B	110.5
C4—C3—H3	119.2	H14A—C14—H14B	108.7
N3—C4—C5	127.7 (2)	C14—C15—C11	103.1 (2)
N3—C4—C3	114.9 (2)	C14—C15—H15A	111.1
C5—C4—C3	117.4 (2)	C11—C15—H15A	111.1
N1—C5—C4	119.3 (2)	C14—C15—H15B	111.1
N1—C5—C6	113.3 (2)	C11—C15—H15B	111.1
C4—C5—C6	127.5 (2)	H15A—C15—H15B	109.1
N2—C6—C7	119.5 (2)	O1—C16—Re1	177.8 (3)
N2—C6—C5	114.9 (2)	O2—C17—Re1	177.4 (3)
C7—C6—C5	125.5 (2)	O3—C18—Re1	176.3 (3)

Hydrogen-bond geometry (Å, °)

<i>D—H...A</i>	<i>D—H</i>	<i>H...A</i>	<i>D...A</i>	<i>D—H...A</i>
N3—H3A...Cl1 ⁱ	0.88	2.53	3.363 (3)	158
N4—H4...Cl1 ⁱⁱ	0.88	2.65	3.419 (2)	147

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