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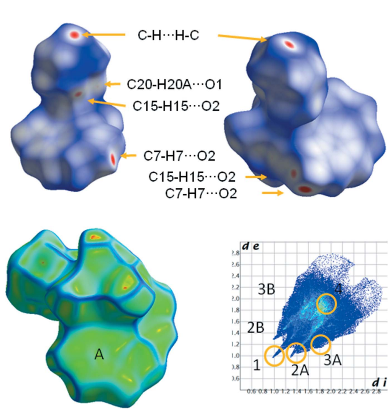

Crystal structure of *fac*-tricarbonyl(cyclohexyl isocyanide-κC)(quinoline-2-carboxylato-$\kappa^2 N,O$)-rhenium(I)

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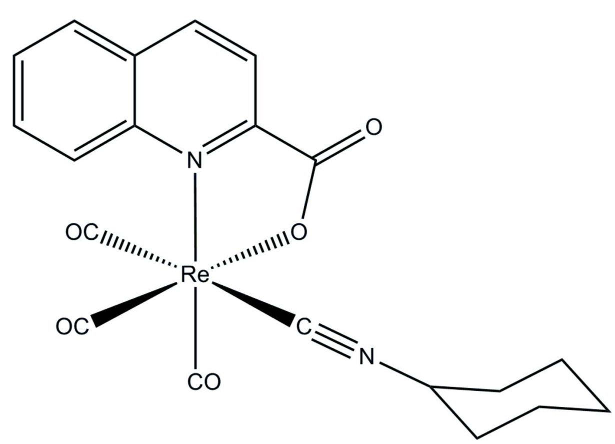
In the title compound, $[Re(C_{10}H_6NO_2)(C_7H_{11}N)(CO)_3]$, the Re^I atom is coordinated by three carbonyl ligands in a facial arrangement and by the N, O and C atoms from a chelating quinaldate anion and a monodentate isocyanide ligand, respectively. The resultant C_4NO coordination sphere is distorted octahedral. A lengthening of the axial Re—CO bond *trans* to the isocyanide ligand is indicative of the *trans* effect. Individual complexes are stacked into rods parallel to [001] through displaced π–π interactions. Weak C—H···O hydrogen-bonding interactions between the rods lead to the formation of layers parallel to (010). These layers are stacked along [010] by C—H···H—C van der Waals contacts.

1. Chemical context

Tricarbonylrhenium(I) compounds are being explored as luminescent probes for cell imaging, photosensitizers in photocatalysis (Lyczko *et al.*, 2015; Bertrand *et al.*, 2014), and as potential radiopharmaceuticals based on the already extensive use of radioactive $^{186/188}$Re compounds in nuclear medicine for pain palliation and radiosynovectomy (Schneider *et al.*, 2005; Bodei *et al.*, 2008). Recent studies have also revealed the potential of cold tricarbonylrhenium(I) complexes as anticancer agents (Leodinova & Gasser, 2014).



As part of our ongoing research in the field of Re/Tc coordination compounds, the crystal structure of a new '2 + 1' tricarbonyl rhenium complex, *fac*-[M(CO)$_3$(L)(QA-NO)],

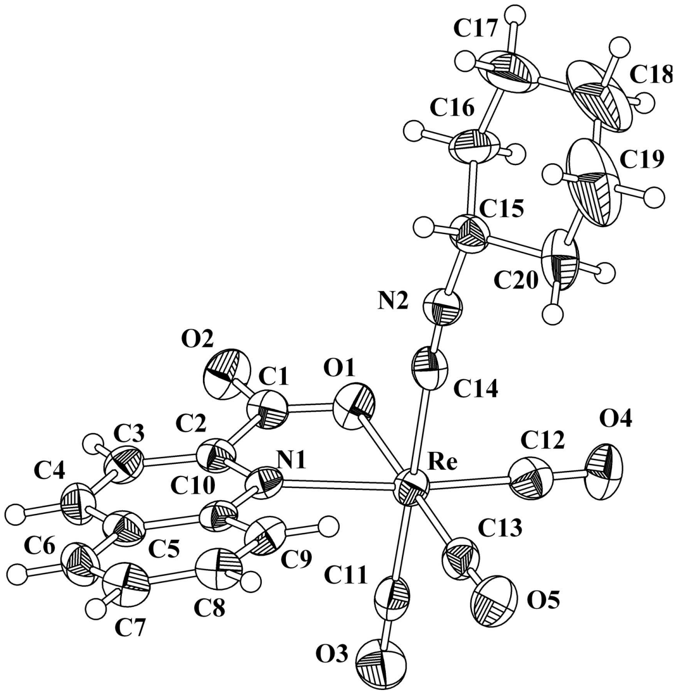


Figure 1
The molecular structure and atom-labelling scheme of the title compound. Displacement ellipsoids are drawn at the 50% probability level.

where *M* is Re,Tc, *L* is the monodentate ligand cyclohexylisocyanide, and QA-NO is deprotonated quinaldic acid, is presented. As a result of of the versatility of the '2 + 1' system, *fac*-[$M(CO)_3(L)$(QA-NO)] complexes can be used as model compounds in the development of targeted radiopharmaceuticals or anticancer agents by suitable replacement of either the bidentate or monodentate ligand. For example, the monodentate ligand may be the isocyanide derivative of a pharmacophore with affinity for a certain receptor. Alternatively, the bidentate ligand may be a more extensive conjugated system to act as a DNA intercalator. Both quinaldate- and isocyanide-based ligands have been used as possible DNA intercalators (Li *et al.*, 2009; Agorastos *et al.*, 2007).

2. Structural commentary

The molecular structure of the title compound, $[Re(C_{10}H_6NO_2)(C_7H_{11}N)(CO)_3]$, is shown in Fig. 1. The Re^I atom is six-coordinated by four C, one N and one O atoms in a distorted octahedral coordination sphere. The carbonyl C atoms are in a facial arrangement, with distances in the range 1.903 (8)–1.960 (8) Å, resulting in a *cis* arrangement of the bi- and monodentate ligands. The longest distance involving the carbonyl ligands [1.960 (8) Å; Re—C11] corresponds to the ligand *trans* to the isocyanide cyclohexyl ligand, defining the axial direction of the octahedral complex. The Re^I atom almost lies in the equatorial plane (deviation, 0.006 Å) defined by the C12, C13, O1 and N1 atoms. The bite angle (N1—Re—O1) of the chelating ligand, corresponding to a five-membered ring, has a typical value of 75.2 (2)° (Lyczko *et al.*, 2015). The Re—N1 and Re—O1 bond lengths are 2.273 (5) and 2.149 (5) Å, respectively. The isocyanide carbon atom, C14, is at a distance of 2.107 (8) Å from the metal site. All these values are close to those of a complex with the same core (Agorastos *et al.*, 2007). The isocyanide group is oriented within the equatorial plane of the cyclohexyl ring which exhibits a chair conformation.

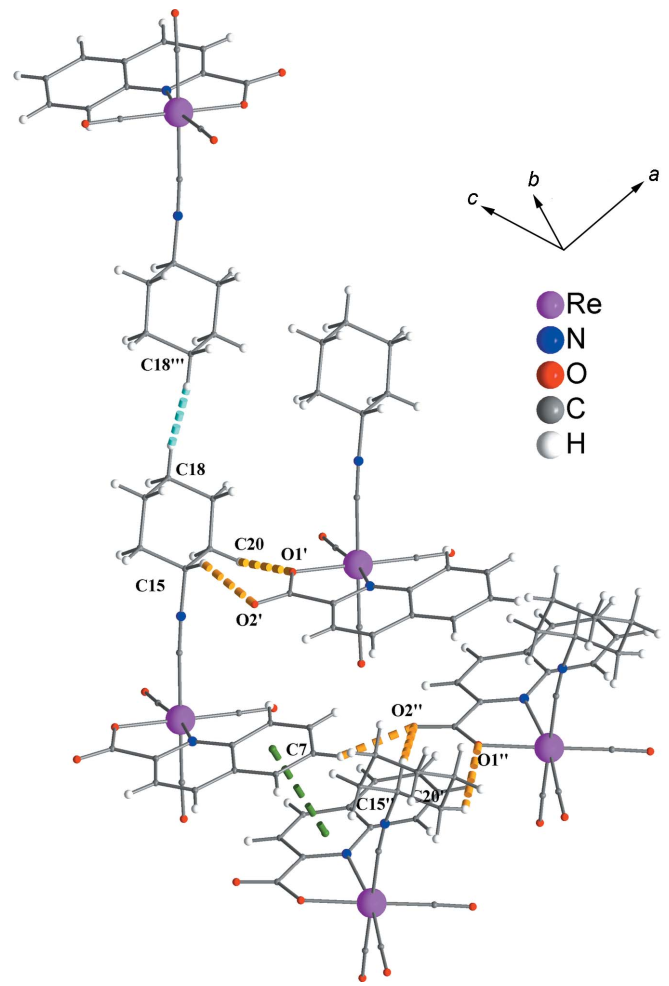


Figure 2
Intermolecular interactions of the title complex with its neighbours. π–π interactions, weak C—H···O hydrogen bonds and short van der Waals contacts are shown with green, orange and turquoise dashed lines, respectively. [Symmetry codes: (′) $x + 1, y, z$; (″) $1 + x, \frac{1}{2} - y, -\frac{1}{2} + z$; (‴) $4 - x, 1 - y, 2 - z$.]

3. Supramolecular features

Figs. 2 and 3 show the supramolecular interactions of each complex molecule with its neighbours. Displaced π–π interactions between the phenyl and pyridine rings of quinaldate ligands of neighbouring complexes are present, with a $Cg1\cdots Cg2^i$ distance of 3.650 Å [$Cg1$ and $Cg2$ are the centroids of the (C5–C10) and (N1,C2,C3,C4,C5,C10) rings, respectively; symmetry code: (i): $4 - x, 1 - y, 2 - z$]. These interactions help to consolidate the stacking of the molecules into

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

D—H$\cdots$$A$	D—H	H$\cdots$$A$	$D\cdots A$	D—H$\cdots$$A$
C20—H20$A$$\cdots$O1^{i}	0.99	2.61	3.260 (10)	123
C15—H15$\cdots$O2^{i}	0.99	2.52	3.365	142
C7—H7$\cdots$O2^{ii}	0.99	2.37	3.133	137

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

rods parallel to [001] (Figs. 3 and 4). Weak intermolecular C—H$\cdots$O hydrogen-bonding interactions (Table 1), including supramolecular $R_2^2(7)$ loops (C20—H20$A$$\cdots$O1 and C15—H15$\cdots$O2) join neighbouring rods into sheets parallel to (010) (Fig. 4). An additional type of interactions, *viz.* short van der Waals forces of the C—H$\cdots$H—C type (Sankolli *et al.*, 2015), is realized through C18—H18$\cdots$H18—C18 contacts. The cyclohexyl end of the isocyanide ligands is hanging above and below the sheets of molecules (Figs. 3 and 4), creating a perhydrogenated outer wall (Sankolli *et al.*, 2015) at both sides of the layers. Such layers are stacked along [010] (through centres of symmetry located at b/2) and interact through the aforementioned C—H$\cdots$H—C contacts (Fig. 5).

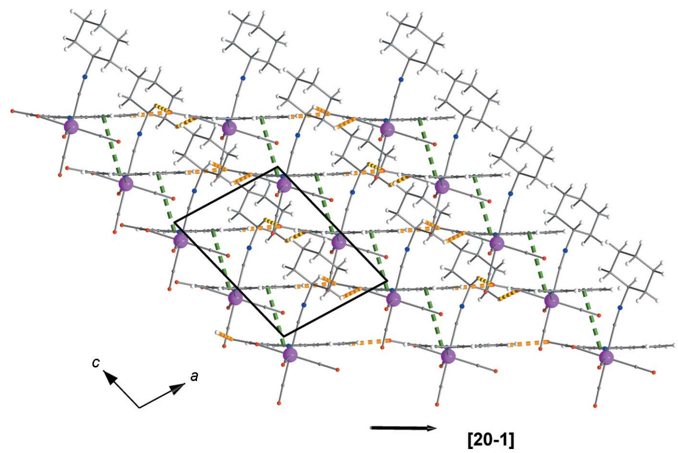


Figure 4
Sheet of complexes arranged parallel to (010) showing π–π and weak C—H$\cdots$O interactions. The colour code is as in Fig. 2.

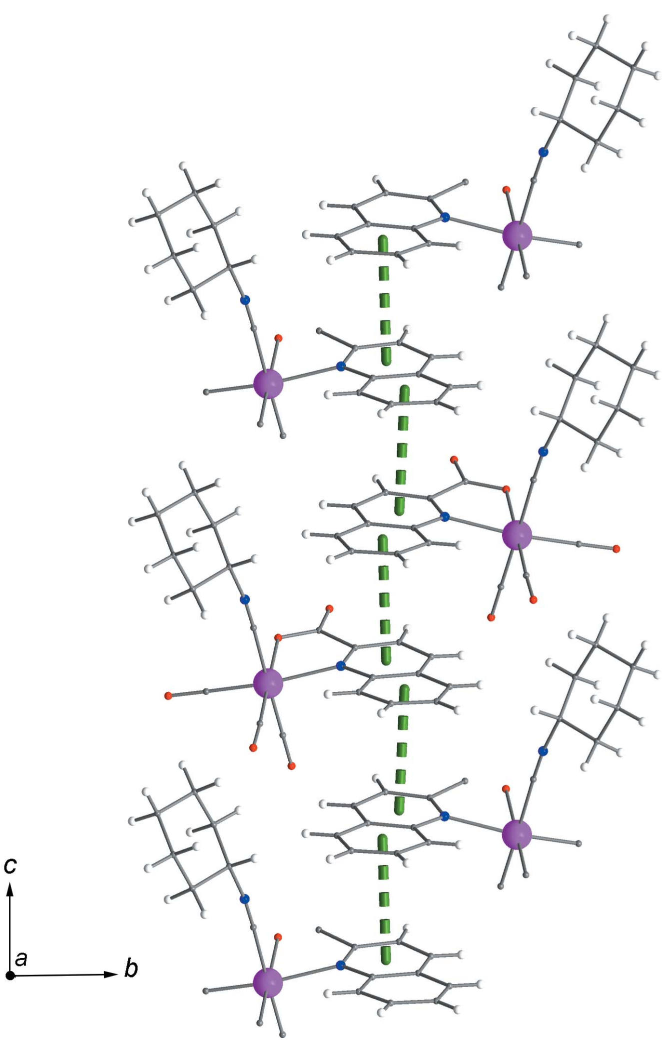


Figure 3
A rod of complexes extending parallel to [001] through π–π interactions. The colour code is as in Fig. 2.

4. Hirshfeld surface analysis

The packing of the complexes in the structure was further investigated with Hirshfeld surface analysis using the *Crystal Explorer* package (Wolff *et al.*, 2012). The d_{norm} and curvedness (Spackman & Jayatilaka, 2009) surface mappings are presented in Fig. 6a, 6b and 6c, respectively. All C—H$\cdots$O and C—H$\cdots$H—C contacts are recognized on the d_{norm} mapped surface as deep-red depression areas in Fig. 6a and 6b, which represent two different upper views of the complex. Arrows at these figures indicate the specific type of contacts at each red

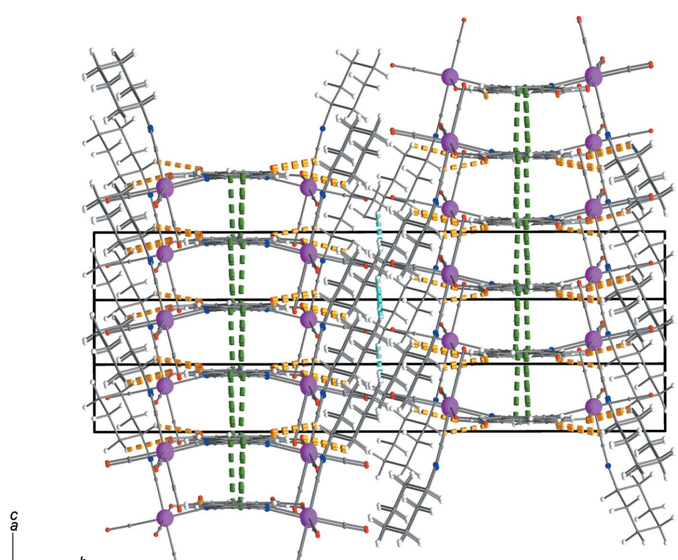


Figure 5
Stack of layers along [010] with C—H$\cdots$H—C van der Waals contacts (light-blue dashed lines) developed among them, shown along the opposite [20$\overline{1}$] direction (see Fig. 4).

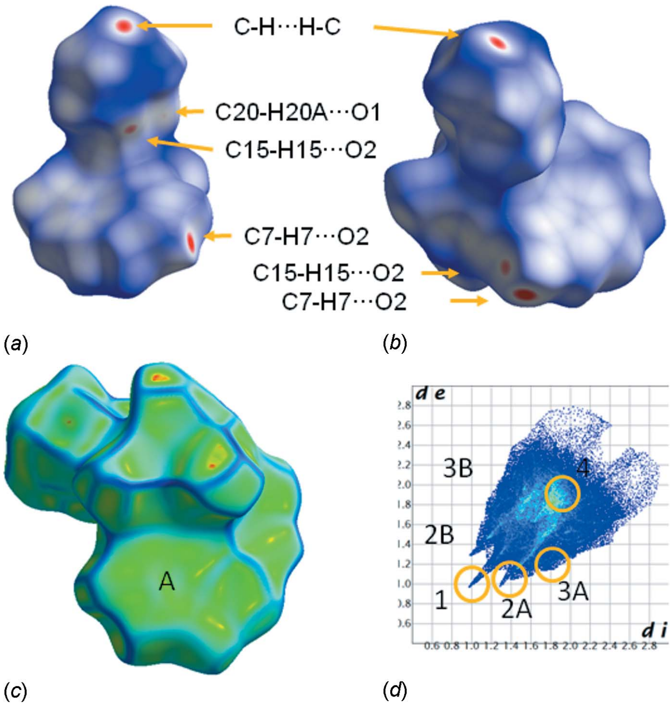


Figure 6
Views of Hirshfeld surfaces mapped with d_{norm} (*a*)/(*b*), curvedness (*c*) properties and (*d*) fingerprint plots for the title complex. d_e and d_i are the distances to the nearest atom centre exterior and interior to the surface. **1**, **2**, **3** and **4** indicate H···H, H···O, C···H and C···C interactions, and **A** and **B** stand for acceptor and donor atoms, respectively.

point. A bottom view of the surface mapped with curvedness (Fig. 6*c*) shows broad, relatively flat regions (indicated by letter A) characteristic of planar stacking of complexes (Spackman & Jayatilaka, 2009), corresponding to the π–π interactions. In the fingerprint plot (Rohl *et al.*, 2008), shown in Fig. 6*d*, the points indicated by 1, 2A & 2B, 3A & 3B and 4 correspond to H···H, H···O, C···H and C···C interactions with relative contributions of 25.1, 44.2, 18.1 and 4.3%, respectively. These types of interactions add to 91.7% of the intermolecular contacts of the Hirshfeld surface area. The remaining contributions (8.3%) correspond to N···H (2.1%), O···C (2.8%) and other less-important interactions (<1%).

5. NMR investigation

In the solution NMR spectra of the complex, both the quinaldate and isocyanocyclohexane moieties are distinguishable. Coordination by the quinaldate is evident from the downfield shifts of all its protons ranging from 0.10 to 0.44 p.p.m. compared to free quinaldic acid under the same conditions (our data). Downfield shifts are also recorded for most of the C atoms of quinaldic acid, the most notable one (4.8 p.p.m.) being the one of the carboxylate carbonyl carbon. For the isocyanocyclohexane moiety, downfield shifts are recorded for the C atom (2.7 p.p.m.) bearing the isocyanide group and for its H atom (0.31 p.p.m.) compared to the free ligand. The most characteristic sign of coordination of the isocyanocyclohexane moiety is the sizable upfield shift of the isocyanide C atom of 15.5 p.p.m., attributed to an increased carbene character upon coordination (Stephany *et al.*, 1974; Sagnou *et al.*, 2010, 2011). In the ^{13}C NMR spectrum of the complex, one of the carbonyl ligands of the $Re(CO)_3^+$ core appears shielded (by 2.8 p.p.m. on average) compared to the other two, an observation that may also be attributed to the *trans* effect of the isocyanide ligand.

6. Database survey

A search of the Cambridge Structural Database (Groom & Allen, 2014) has revealed eight tricarbonyl complexes in facial arrangement and different *N,O*-bidentate ligands with a pyridine carboxylato-2 group at the binding side of the corresponding ligand. The *N,O*-binding sites together with the two carbonyl groups *trans* to N and O atoms define an equatorial plane, and the third together with the monodentate ligand define the axial position. The Re—C bond lengths of axial carbonyl ligands (1.883–1.922 Å) *trans* to the monodentate ligand have values equal or smaller than the equatorial ones (1.892–1.945 Å) when the ligand is an aqua ligand (Schutte & Visser, 2008; Mundwiler *et al.*, 2004). The carbonyl Re—C bond lengths are intermediate (1.914–1.917 Å) between the values of the Re—C bonds *trans* to the equatorial O (1.886–1.916 Å) and N (1.921–1.926 Å) atoms, if the *trans* ligand is bonded to Re through an N atom (Benny *et al.*, 2009; Mundwiler *et al.*, 2004). Finally, the respective bond length, 1.947 Å, is longer than both Re—C bonds *trans* to equatorial O (1.912 Å) and N (1.914 Å) atoms if the Re atom is bonded to a P atom of a phosphine ligand (Hayes *et al.*, 2014). In the case of the isocyanide group *trans* to the axial Re—C bond (Agorastos *et al.*, 2007), the results are indistinct. In one case (XIDPUW), the axial bond length (1.756 Å) is shorter than the equatorial one (1.849 Å *trans* to O and 1.901 Å *trans* to N) whereas in the other case (XIDQAD), the corresponding length (1.914 Å) is longer than the equatorial one (1.495 Å *trans* to O and 1.885 Å *trans* to N). In the present structure, the Re—C11 bond (1.960 Å), is longer than the Re—C13 (1.903 Å, *trans* to O) and Re—C12 (1.912 Å, *trans* to N) bonds. This result is supported by the NMR analysis and is indicative of the structural *trans* effect (Coe & Glenwright, 2000).

7. Synthesis and crystallization

To a stirred solution of quinaldic acid (17.3 mg, 0.1 mmol) in 5 ml methanol, a solution of $[NEt_4]_2[ReBr_3(CO)_3]$ (77 mg, 0.1 mmol) in 5 ml methanol was added. The mixture was heated at 333 K, and after 30 min a solution of cyclohexyl isocyanide (0.1 mmol) in 3 ml methanol was added. The mixture was stirred at room temperature for 2 h and the reaction progress was monitored by HPLC. The solvent was removed under reduced pressure and the solid residue was recrystallized from dichloromethane/hexane. The resulting solid was redissolved in a minimum volume of dichloromethane, layered with hexane and left to stand at room temperature. After a few days crystals suitable for X-ray analysis were isolated (yield: 44 mg, 80%). ^{1}H NMR (DMSO-d_6, p.p.m.): 8.93 (1H), 8.58 (1H), 8.32 (1H), 8.28 (1H), 8.18 (1H), 7.94 (1H), 4.08 (1H), 1.50 (2H), 1.40 (2H), 1.13(2H), 1.08 (2H), 0.88 (2H); ^{13}C NMR (DMSO-d_6, p.p.m.): 193.65, 193.12, 190.54, 172.06, 152.63, 146.23, 142.09, 138.85, 133.04, 130.47, 129.67, 129.61, 127.78, 122.78, 53.72, 30.48, 23.91, 20.70.

8. Refinement

Crystal data, data collection and structure refinement details are summarized in Table 2. C-bound H atoms were placed in idealized positions and refined using a riding model with C—H = 0.95 Å (aromatic H atoms), C—H = 0.99 Å (methylene H atoms), and with $U_{iso}(H) = 1.2U_{eq}(C)$.

Acknowledgements

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Table 2
Experimental details.

Crystal data	
Chemical formula	$[Re(C_{10}H_6NO_2)(C_7H_{11}N)(CO)_3]$
M_r	551.55
Crystal system, space group	Monoclinic, $P2_1/c$
Temperature (K)	170
a, b, c (Å)	7.1529 (1), 29.5703 (5), 9.6309 (2)
β (°)	105.572 (1)
V (Å^3)	1962.29 (6)
Z	4
Radiation type	Cu $K\alpha$
μ (mm^{-1})	12.41
Crystal size (mm)	0.49 × 0.12 × 0.04
Data collection	
Diffractometer	Rigaku R-AXIS SPIDER *IPDS*
Absorption correction	Multi-scan (*CrystalClear*; Rigaku 2005)
T_{min}, T_{max}	0.374, 1.000
No. of measured, independent and observed [$I > 2\sigma(I)$] reflections	21436, 3283, 2723
R_{int}	0.069
$(\sin \theta/\lambda)_{max}$ (Å^{-1})	0.588
Refinement	
$R[F^2 > 2\sigma(F^2)]$, $wR(F^2)$, S	0.038, 0.102, 1.17
No. of reflections	3283
No. of parameters	253
H-atom treatment	H-atom parameters constrained
$\Delta\rho_{max}$, $\Delta\rho_{min}$ (e Å^{-3})	1.30, −1.43

Computer programs: *CrystalClear* (Rigaku, 2005), *SHELXS97* and *SHELXTL* (Sheldrick, 2008), *SHELXL2014* (Sheldrick, 2015), *DIAMOND* (Crystal Impact, 2012) and *PLATON* (Spek, 2009).

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supporting information

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Crystal structure of *fac*-tricarbonyl(cyclohexyl isocyanide-κC)(quinoline-2-carboxylato-$\kappa^2 N,O$)rhenium(I)

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Computing details

Data collection: *CrystalClear* (Rigaku, 2005); cell refinement: *CrystalClear* (Rigaku, 2005); data reduction: *CrystalClear* (Rigaku, 2005); program(s) used to solve structure: *SHELXS97* (Sheldrick, 2008); program(s) used to refine structure: *SHELXL2014* (Sheldrick, 2015); molecular graphics: *DIAMOND* (Crystal Impact, 2012); software used to prepare material for publication: *SHELXTL* (Sheldrick, 2008) and *PLATON* (Spek, 2009).

fac-Tricarbonyl(cyclohexyl isocyanide-κC)(quinoline-2-carboxylato-$\kappa^2 N,O$)rhenium(I)

Crystal data

$[Re(C_{10}H_6NO_2)(C_7H_{11}N)(CO)_3]$
M_r = 551.55
Monoclinic, $P2_1/c$
a = 7.1529 (1) Å
b = 29.5703 (5) Å
c = 9.6309 (2) Å
β = 105.572 (1)°
V = 1962.29 (6) Å^3
Z = 4
$F(000)$ = 1064
D_x = 1.867 Mg m^{-3}
Cu $K\alpha$ radiation, λ = 1.54178 Å
Cell parameters from 17036 reflections
θ = 6.6–71.9°
μ = 12.41 mm^{-1}
T = 170 K
Parallelepiped, colorless
0.49 × 0.12 × 0.04 mm

Data collection

Rigaku R-AXIS SPIDER IPDS diffractometer
Radiation source: fine-focus sealed tube
θ scans
Absorption correction: multi-scan (*CrystalClear*; Rigaku 2005)
T_{min} = 0.374, T_{max} = 1.000
21436 measured reflections
3283 independent reflections
2723 reflections with $I > 2\sigma(I)$
R_{int} = 0.069
θ_{max} = 65.0°, θ_{min} = 6.6°
$h = -8 \rightarrow 8$
$k = -33 \rightarrow 27$
$l = -11 \rightarrow 11$

Refinement

Refinement on F^2
Least-squares matrix: full
$R[F^2 > 2\sigma(F^2)]$ = 0.038
$wR(F^2)$ = 0.102
S = 1.17
3283 reflections
253 parameters
0 restraints
Hydrogen site location: inferred from neighbouring sites
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0314P)^2 + 5.6979P]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max}$ = 0.001
$\Delta\rho_{max}$ = 1.30 e Å^{-3}
$\Delta\rho_{min}$ = −1.43 e Å^{-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.

Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (Å²)

	x	y	z	U_{iso}*/U_{eq}
Re	0.92831 (4)	0.37585 (2)	0.37372 (3)	0.03094 (14)
N1	0.9723 (7)	0.30250 (18)	0.4300 (5)	0.0284 (12)
O1	0.7817 (7)	0.36522 (17)	0.5387 (5)	0.0416 (12)
O2	0.6499 (7)	0.31296 (18)	0.6478 (5)	0.0484 (13)
C1	0.7486 (9)	0.3239 (2)	0.5673 (7)	0.0355 (16)
C2	0.8488 (9)	0.2879 (2)	0.5021 (6)	0.0301 (14)
C3	0.8153 (10)	0.2429 (2)	0.5234 (7)	0.0346 (15)
H3	0.7263	0.2344	0.5762	0.042*
C4	0.9110 (9)	0.2111 (2)	0.4680 (7)	0.0364 (16)
H4	0.8825	0.1799	0.4757	0.044*
C5	1.0531 (9)	0.2242 (2)	0.3988 (7)	0.0350 (16)
C6	1.1665 (10)	0.1931 (2)	0.3467 (7)	0.0413 (17)
H6	1.1444	0.1616	0.3549	0.050*
C7	1.3075 (10)	0.2069 (3)	0.2848 (7)	0.0421 (18)
H7	1.3827	0.1855	0.2496	0.051*
C8	1.3385 (10)	0.2532 (2)	0.2742 (7)	0.0395 (17)
H8	1.4385	0.2629	0.2332	0.047*
C9	1.2302 (9)	0.2851 (2)	0.3207 (6)	0.0344 (15)
H9	1.2542	0.3164	0.3112	0.041*
C10	1.0834 (8)	0.2709 (2)	0.3826 (6)	0.0279 (14)
C11	0.6948 (11)	0.3602 (2)	0.2213 (8)	0.0386 (16)
O3	0.5615 (7)	0.3509 (2)	0.1301 (6)	0.0588 (16)
C12	0.8592 (10)	0.4384 (3)	0.3514 (8)	0.0419 (17)
O4	0.8223 (8)	0.47647 (18)	0.3391 (6)	0.0549 (14)
C13	1.0602 (10)	0.3846 (2)	0.2285 (8)	0.0381 (17)
O5	1.1396 (8)	0.39159 (19)	0.1404 (6)	0.0529 (13)
C14	1.1806 (11)	0.3915 (2)	0.5378 (8)	0.0381 (16)
N2	1.3213 (8)	0.40084 (19)	0.6200 (6)	0.0353 (13)
C15	1.5026 (9)	0.4170 (2)	0.7131 (7)	0.0365 (16)
H15	1.5983	0.3916	0.7321	0.044*
C16	1.4738 (12)	0.4329 (3)	0.8551 (7)	0.057 (2)
H16A	1.4335	0.4071	0.9061	0.068*
H16B	1.3704	0.4561	0.8376	0.068*
C17	1.6631 (17)	0.4527 (4)	0.9473 (10)	0.098 (4)
H17A	1.6427	0.4645	1.0384	0.117*
H17B	1.7629	0.4287	0.9717	0.117*
C18	1.7339 (18)	0.4906 (4)	0.8689 (17)	0.133 (6)
H18A	1.6382	0.5155	0.8505	0.160*
H18B	1.8582	0.5024	0.9302	0.160*

C19	1.7628 (14)	0.4744 (4)	0.7288 (17)	0.111 (5)
H19A	1.8662	0.4512	0.7479	0.133*
H19B	1.8056	0.5001	0.6788	0.133*
C20	1.5778 (10)	0.4546 (3)	0.6321 (9)	0.055 (2)
H20A	1.6038	0.4421	0.5437	0.066*
H20B	1.4784	0.4786	0.6032	0.066*

Atomic displacement parameters (Å^2)

	U^{11}	U^{22}	U^{33}	U^{12}	U^{13}	U^{23}
Re	0.0284 (2)	0.0315 (2)	0.0323 (2)	0.00254 (11)	0.00704 (15)	0.00250 (11)
N1	0.025 (3)	0.034 (3)	0.024 (3)	0.003 (2)	0.003 (2)	0.002 (2)
O1	0.044 (3)	0.041 (3)	0.046 (3)	0.011 (2)	0.022 (2)	0.004 (2)
O2	0.050 (3)	0.055 (3)	0.051 (3)	0.007 (3)	0.032 (3)	0.007 (3)
C1	0.031 (4)	0.037 (4)	0.036 (4)	0.007 (3)	0.006 (3)	−0.002 (3)
C2	0.023 (3)	0.035 (4)	0.029 (3)	0.002 (3)	0.001 (3)	−0.001 (3)
C3	0.038 (4)	0.040 (4)	0.029 (3)	0.001 (3)	0.015 (3)	0.003 (3)
C4	0.037 (4)	0.028 (4)	0.041 (4)	−0.001 (3)	0.007 (3)	0.002 (3)
C5	0.027 (3)	0.040 (4)	0.034 (4)	0.002 (3)	−0.001 (3)	−0.005 (3)
C6	0.044 (4)	0.029 (4)	0.048 (4)	0.007 (3)	0.007 (3)	−0.005 (3)
C7	0.033 (4)	0.049 (5)	0.046 (4)	0.007 (3)	0.014 (3)	−0.005 (4)
C8	0.031 (4)	0.049 (5)	0.040 (4)	0.005 (3)	0.012 (3)	−0.004 (3)
C9	0.027 (3)	0.044 (4)	0.032 (3)	0.002 (3)	0.008 (3)	0.006 (3)
C10	0.023 (3)	0.034 (4)	0.023 (3)	0.005 (3)	−0.001 (3)	0.001 (3)
C11	0.047 (4)	0.031 (4)	0.042 (4)	0.002 (3)	0.019 (4)	0.006 (3)
O3	0.038 (3)	0.059 (4)	0.066 (4)	−0.003 (3)	−0.010 (3)	0.001 (3)
C12	0.032 (4)	0.049 (5)	0.044 (4)	−0.006 (4)	0.008 (3)	−0.001 (4)
O4	0.063 (4)	0.036 (3)	0.067 (4)	0.010 (3)	0.021 (3)	0.013 (3)
C13	0.036 (4)	0.033 (4)	0.042 (4)	0.001 (3)	0.004 (4)	0.004 (3)
O5	0.052 (3)	0.057 (4)	0.055 (3)	−0.004 (3)	0.024 (3)	0.008 (3)
C14	0.042 (4)	0.028 (4)	0.044 (4)	0.004 (3)	0.012 (4)	0.001 (3)
N2	0.039 (3)	0.036 (3)	0.032 (3)	−0.003 (3)	0.009 (3)	−0.002 (2)
C15	0.034 (4)	0.035 (4)	0.038 (4)	0.003 (3)	0.005 (3)	−0.004 (3)
C16	0.077 (6)	0.055 (6)	0.029 (4)	0.013 (5)	−0.002 (4)	−0.005 (4)
C17	0.123 (9)	0.068 (7)	0.064 (6)	0.021 (7)	−0.042 (6)	−0.025 (5)
C18	0.093 (9)	0.073 (9)	0.172 (14)	−0.017 (7)	−0.072 (9)	−0.035 (9)
C19	0.051 (6)	0.066 (8)	0.203 (15)	−0.020 (5)	0.009 (8)	0.001 (9)
C20	0.047 (5)	0.037 (5)	0.085 (7)	−0.005 (3)	0.023 (5)	0.012 (4)

Geometric parameters (Å, º)

Re—C13	1.903 (8)	C9—C10	1.404 (8)
Re—C12	1.912 (8)	C9—H9	0.9500
Re—C11	1.960 (8)	C11—O3	1.144 (8)
Re—C14	2.107 (8)	C12—O4	1.155 (8)
Re—O1	2.149 (5)	C13—O5	1.159 (8)
Re—N1	2.237 (5)	C14—N2	1.135 (8)
N1—C2	1.333 (8)	N2—C15	1.446 (8)

N1—C10	1.380 (7)	C15—C16	1.512 (9)
O1—C1	1.289 (8)	C15—C20	1.537 (9)
O2—C1	1.224 (8)	C15—H15	1.0000
C1—C2	1.510 (9)	C16—C17	1.523 (12)
C2—C3	1.376 (9)	C16—H16A	0.9900
C3—C4	1.356 (9)	C16—H16B	0.9900
C3—H3	0.9500	C17—C18	1.512 (17)
C4—C5	1.411 (9)	C17—H17A	0.9900
C4—H4	0.9500	C17—H17B	0.9900
C5—C6	1.405 (9)	C18—C19	1.496 (17)
C5—C10	1.413 (9)	C18—H18A	0.9900
C6—C7	1.365 (9)	C18—H18B	0.9900
C6—H6	0.9500	C19—C20	1.516 (13)
C7—C8	1.394 (10)	C19—H19A	0.9900
C7—H7	0.9500	C19—H19B	0.9900
C8—C9	1.371 (9)	C20—H20A	0.9900
C8—H8	0.9500	C20—H20B	0.9900

C13—Re—C12	87.2 (3)	C10—C9—H9	120.5
C13—Re—C11	88.4 (3)	N1—C10—C9	120.1 (6)
C12—Re—C11	90.1 (3)	N1—C10—C5	120.4 (6)
C13—Re—C14	91.6 (3)	C9—C10—C5	119.6 (6)
C12—Re—C14	90.9 (3)	O3—C11—Re	178.3 (6)
C11—Re—C14	179.1 (3)	O4—C12—Re	178.3 (7)
C13—Re—O1	179.2 (2)	O5—C13—Re	177.4 (6)
C12—Re—O1	93.5 (2)	N2—C14—Re	175.9 (6)
C11—Re—O1	91.8 (2)	C14—N2—C15	173.2 (7)
C14—Re—O1	88.1 (2)	N2—C15—C16	110.3 (6)
C13—Re—N1	104.1 (2)	N2—C15—C20	107.6 (5)
C12—Re—N1	168.7 (2)	C16—C15—C20	112.7 (6)
C11—Re—N1	89.4 (2)	N2—C15—H15	108.7
C14—Re—N1	89.7 (2)	C16—C15—H15	108.7
O1—Re—N1	75.16 (18)	C20—C15—H15	108.7
C2—N1—C10	118.3 (5)	C15—C16—C17	109.4 (8)
C2—N1—Re	111.7 (4)	C15—C16—H16A	109.8
C10—N1—Re	129.2 (4)	C17—C16—H16A	109.8
C1—O1—Re	116.8 (4)	C15—C16—H16B	109.8
O2—C1—O1	123.8 (6)	C17—C16—H16B	109.8
O2—C1—C2	119.8 (6)	H16A—C16—H16B	108.2
O1—C1—C2	116.4 (6)	C18—C17—C16	111.0 (8)
N1—C2—C3	123.8 (6)	C18—C17—H17A	109.4
N1—C2—C1	116.4 (6)	C16—C17—H17A	109.4
C3—C2—C1	119.8 (6)	C18—C17—H17B	109.4
C4—C3—C2	119.1 (6)	C16—C17—H17B	109.4
C4—C3—H3	120.4	H17A—C17—H17B	108.0
C2—C3—H3	120.4	C19—C18—C17	111.2 (10)
C3—C4—C5	119.8 (6)	C19—C18—H18A	109.4
C3—C4—H4	120.1	C17—C18—H18A	109.4

C5—C4—H4	120.1	C19—C18—H18B	109.4
C6—C5—C4	123.1 (7)	C17—C18—H18B	109.4
C6—C5—C10	118.7 (6)	H18A—C18—H18B	108.0
C4—C5—C10	118.2 (6)	C18—C19—C20	111.6 (9)
C7—C6—C5	121.7 (7)	C18—C19—H19A	109.3
C7—C6—H6	119.2	C20—C19—H19A	109.3
C5—C6—H6	119.2	C18—C19—H19B	109.3
C6—C7—C8	118.4 (7)	C20—C19—H19B	109.3
C6—C7—H7	120.8	H19A—C19—H19B	108.0
C8—C7—H7	120.8	C19—C20—C15	109.5 (8)
C9—C8—C7	122.6 (7)	C19—C20—H20A	109.8
C9—C8—H8	118.7	C15—C20—H20A	109.8
C7—C8—H8	118.7	C19—C20—H20B	109.8
C8—C9—C10	119.0 (7)	C15—C20—H20B	109.8
C8—C9—H9	120.5	H20A—C20—H20B	108.2

Hydrogen-bond geometry (Å, º)

D—H···*A*	*D*—H	H···*A*	*D*···*A*	*D*—H···*A*
C20—H20*A*···O1^{i}	0.99	2.61	3.260 (10)	123
C15—H15···O2^{i}	0.99	2.52	3.365	142
C7—H7···O2^{ii}	0.99	2.37	3.133	137

Symmetry codes: (i) *x*+1, *y*, *z*; (ii) *x*+1, −*y*+1/2, *z*−1/2.