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(Benzophenone imine-*N*)nonacarbonyldirhenium(0)(*Re*—*Re*)

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.016 Å
R factor = 0.034
wR factor = 0.094
Data-to-parameter ratio = 14.7

For details of how these key indicators were automatically derived from the article, see http://journals.iucr.org/e.

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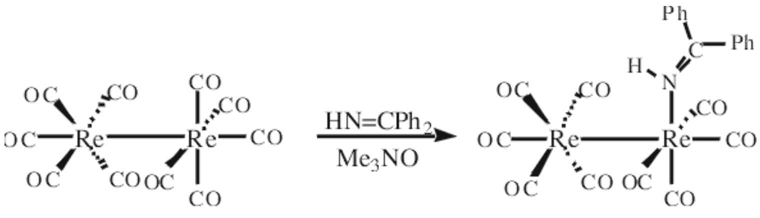
The reaction of [$Re_2(CO)_{10}$] with benzophenone imine in dichloroethane, in the presence of Me_3NO, leads to the binuclear title compound, nonacarbonyl(diphenylmethanimine-*N*)dirhenium(0)(*Re*—*Re*), [$Re_2(HN{=}CPh_2)(CO)_9$]. Both Re atoms are in an octahedral environment. The $HN{=}CPh_2$ ligand is attached to one of the metal atoms through the N atom, occupying an equatorial position. The equatorial carbonyl ligands of each octahedral fragment are staggered by 45°.

Comment

Interest in the synthesis and reactivity of late-transition-metal amido complexes has grown considerably in recent years as a consequence of the relative scarcity of such compounds and of their potential use in carbon–nitrogen bond-forming reactions (Cabeza *et al.*, 1998). In this field, we have recently communicated the first insertion of a non-activated alkyne into a metal–nitrogen bond, achieved on a triruthenium cluster derived from benzophenone imine (Cabeza *et al.*, 1997). In an extension of the interesting reactivity observed for these ruthenium complexes to other transition metals, we studied the reactivity of benzophenone imine with osmium (Cabeza *et al.*, 2000) and rhenium carbonyl derivatives. The crystal structure reported herein of (benzophenone imine-*N*)nonacarbonyldirhenium(0)(*Re*—*Re*), (I), is part of this latter study.



(I)

Experimental

Me_3NO (46 mg, 0.767 mmol) was added to a solution of [$Re_2(CO)_{10}$] (200 mg, 0.307 mmol) and benzophenone imine (103 µl, 0.614 mmol) in 1,2-dichloroethane (20 ml). The color changed immediately to dark orange. The mixture was heated at reflux temperature for 1 h. The solution was concentrated under reduced pressure to *ca* 3 ml and the residue set on the top of a column of neutral alumina (2 × 10 cm, activity I). Elution with hexanes afforded a small amount of unreacted [$Re_2(CO)_{10}$]. Subsequent elution with hexanes/dichloromethane (3:1) afforded an orange band which contained 110 mg (44%) of the title compound, which was crystallized from Et_2O/hexanes at 253 K.

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Crystal data

$[Re_2(C_{13}H_{11}N)(CO)_9]$
$M_r = 805.72$
Monoclinic, $P2_1/c$
$a = 10.8071\ (6)$ Å
$b = 13.6951\ (8)$ Å
$c = 16.4285\ (11)$ Å
$\beta = 102.780\ (4)°$
$V = 2371.3\ (2)$ Å^3
$Z = 4$

$D_x = 2.275$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 2241 reflections
$\theta = 1–25°$
$\mu = 10.25$ mm^{-1}
$T = 293\ (2)$ K
Prismatic, yellow
$0.26 \times 0.10 \times 0.07$ mm

Data collection

Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical (*XABS*2; Parkin *et al.*, 1995)
$T_{min} = 0.113$, $T_{max} = 0.485$
7361 measured reflections
4499 independent reflections
2349 reflections with $I > 2\sigma(I)$
$R_{int} = 0.054$

$\theta_{max} = 26.0°$
$h = -13 \rightarrow 12$
$k = 0 \rightarrow 16$
$l = 0 \rightarrow 20$
3 standard reflections every 200 reflections frequency: 60 min intensity decay: 2.4%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.034$
$wR(F^2) = 0.094$
$S = 1.00$
4499 reflections
307 parameters

H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0426P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} < 0.001$
$\Delta\rho_{max} = 0.94$ e Å^{-3}
$\Delta\rho_{min} = -1.28$ e Å^{-3}

Table 1
Selected geometric parameters (Å, °).

Re1—C15	1.904 (11)	N1—C1	1.285 (11)
Re1—C17	1.929 (11)	O14—C14	1.137 (13)
Re1—C16	1.976 (12)	O15—C15	1.160 (12)
Re1—C14	1.986 (12)	O16—C16	1.136 (12)
Re1—N1	2.209 (7)	O17—C17	1.146 (11)
Re1—Re2	3.0542 (6)	O18—C18	1.169 (15)
Re2—C18	1.918 (14)	O19—C19	1.133 (13)
Re2—C20	1.956 (12)	O20—C20	1.166 (12)
Re2—C21	1.960 (13)	O21—C21	1.145 (13)
Re2—C19	1.968 (14)	O22—C22	1.143 (12)
Re2—C22	1.995 (12)		
C15—Re1—C17	90.5 (4)	C18—Re2—C21	94.2 (6)
C15—Re1—C16	91.3 (4)	C20—Re2—C21	90.5 (5)
C17—Re1—C16	89.6 (5)	C18—Re2—C19	96.2 (6)
C15—Re1—C14	94.4 (5)	C20—Re2—C19	87.9 (5)
C17—Re1—C14	88.6 (4)	C21—Re2—C19	169.5 (5)
C16—Re1—C14	174.0 (5)	C18—Re2—C22	96.4 (5)
C15—Re1—N1	93.6 (3)	C20—Re2—C22	169.0 (5)
C17—Re1—N1	175.9 (4)	C21—Re2—C22	91.2 (5)
C16—Re1—N1	89.9 (4)	C19—Re2—C22	88.5 (5)
C14—Re1—N1	91.5 (4)	C18—Re2—Re1	177.6 (4)
C15—Re1—Re2	177.1 (3)	C20—Re2—Re1	83.9 (3)
C17—Re1—Re2	87.3 (3)	C21—Re2—Re1	84.1 (4)
C16—Re1—Re2	86.9 (3)	C19—Re2—Re1	85.4 (3)
C14—Re1—Re2	87.3 (3)	C22—Re2—Re1	85.4 (3)
N1—Re1—Re2	88.6 (2)	C1—N1—Re1	137.4 (7)
C18—Re2—C20	94.3 (5)		

H atoms were placed in geometrically idealized positions employing appropriate riding models with isotropic displacement parameters constrained to 1.2 times the U_{eq} of their carrier atoms.

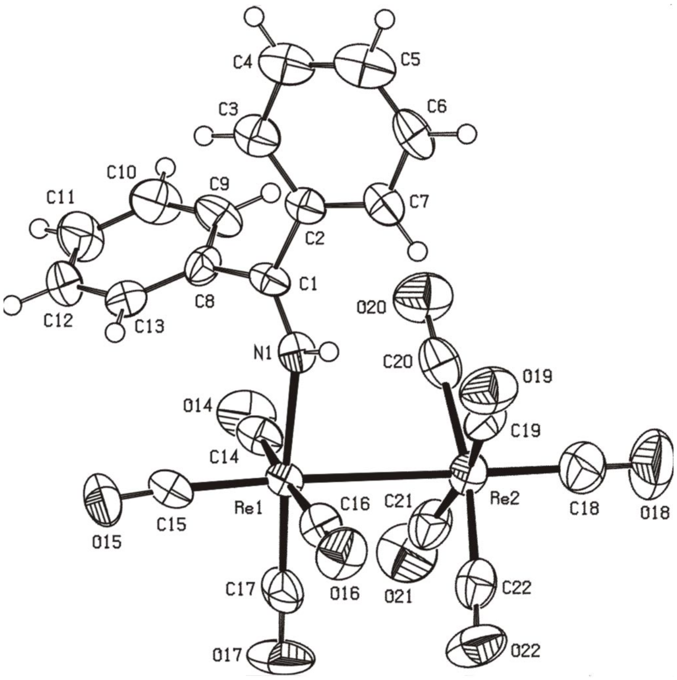

Figure 1
The structure of the title complex showing 50% probability displacement ellipsoids and the atom-numbering scheme.

Data collection: *CAD*-4 *EXPRESS* (Enraf–Nonius, 1994); cell refinement: CRYSDA (Beurskens *et al.*, 1992); data reduction: *REFLEX* (García-Granda *et al.*, 1999); program(s) used to solve structure: *DIRDIF* (Beurskens *et al.*, 1992); program(s) used to refine structure: *SHELXL*97 (Sheldrick, 1997); molecular graphics: *EUCLID* (Spek, 1982); software used to prepare material for publication: *SHELXL*97.

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