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(*tert*-Butyl isocyanide)tetracarbonyl[*N*-(1,2,3,4-tetraphenylbutadienyl)benzophenone imine]diruthenium(I)

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The reaction of [$Ru_2\{\mu$-PhC=CPh-CPh=CPh-N=CPh-$(C_6H_4)\}(\mu$-CO$)(CO)_4]$, (1), with ^{t}BuNC leads to the carbonyl substitution product [$Ru_2\{\mu$-PhC=CPh-CPh=CPh-N=C-Ph$(C_6H_4)\}(\mu$-CO$)(CO)_3(^t$BuNC)] or [$Ru_2(C_5H_9N)(C_{41}H_{29}N)(CO)_4$], (2). The X-ray molecular structure of (2) is similar to its parent compound (1) and shows that the isonitrile ligand is in a *cis* arrangement with respect to both the iminic N atom and the C atom of the orthometalated phenyl ring.

Key indicators

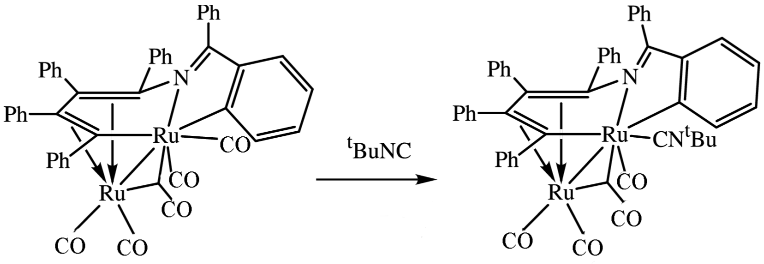
Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.012 Å
R factor = 0.048
wR factor = 0.142
Data-to-parameter ratio = 13.3

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

Comment

This work is part of a project dealing with insertion reactions of unsaturated organic ligands into the C—Ru bonds of polynuclear ruthenium–carbonyl complexes (Cabeza *et al.*, 1998).

The reaction of (1) with *tert*-butylisonitrile does not lead to insertion of the isonitrile into any of the C—Ru bonds of (1) but to the carbonyl substitution product (2). The crystal structure of (2) was determined by X-ray diffraction in order to ascertain the position of the isonitrile ligand in the complex.



(1)

(2)

The molecular structure of (2) (Fig. 1) is similar to that of its parent compound (1) (Cabeza *et al.*, 1998). The *tert*-butylisonitrile ligand is terminally bound to Ru2 through its C5 atom [Ru2—C5 = 2.015 (9) Å and Ru2—C5—N1 = 174.8 (9)°], being *cis* to both the N atom of the imine ligand [C5—Ru2—N2 = 94.5 (3)°] and the C atom of the orthometalated phenyl ring [C5—Ru2—C12 = 80.4 (3)°].

Experimental

Synthesis of (2): a solution of (1) (100 mg, 0.114 mmol) and ^{t}BuNC (26 µl, 0.228 mmol) in dry and deoxygenated tetrahydrofuran (THF; 20 ml) was stirred under nitrogen at reflux temperature for 30 min. The solution was evaporated to dryness under reduced pressure and the residue was separated by column chromatography on neutral alumina (activity IV). Hexane eluted the excess isonitrile. Dichloromethane eluted an orange band which afforded compound (2) upon removal of the solvent. The orange solid was recrystallized at 253 K by liquid diffusion of pentane into a concentrated solution of the complex in toluene. Yield: 82 mg (77%). Analysis (%), found: C 64.52, H 4.33, N 2.89%; calculated for $C_{50}H_{38}N_2O_4Ru_2$: C 64.37, H 4.11, N 3.00%. IR (THF, cm^{-1}): ν(CN) 2166 (*m*); ν(CO) 2008 (*vs*), 1974 (*s*), 1943 (*m*), 1840 (*m*, *br*). ^{1}H NMR (CDCl$_3$, 300 MHz, p.p.m.): δ 7.7–6.5 (*m*, 27H), 5.86 (*d*, 7.4 Hz, 1H), 5.09 (*d*, 7.4 Hz, 1H), 1.56 (*s*, 9H). Compound (1) was prepared as described previously (Cabeza *et al.*, 1998).

Crystal data

$[Ru_2(C_5H_9N)(C_{41}H_{29}N)(CO)_4]$
M_r = 932.96
Monoclinic, $P2_1/c$
a = 20.212 (10) Å
b = 11.111 (8) Å
c = 19.362 (14) Å
β = 96.53 (13)°
V = 4320 (5) Å^3
Z = 4
D_x = 1.434 Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 25 reflections
θ = 10–15°
μ = 0.75 mm^{-1}
T = 293 (2) K
Prismatic, red
0.23 × 0.20 × 0.16 mm

Data collection

Nonius CAD-4 diffractometer
ω–2θ scans
Absorption correction: empirical (*SHELXA*; Sheldrick, 1997*a*)
$T_{\min}$ = 0.840, $T_{\max}$ = 0.880
7792 measured reflections
7562 independent reflections
3795 reflections with $I > 2\sigma(I)$
$R_{\rm int}$ = 0.072
$\theta_{\max}$ = 25.0°
$h = -23 \rightarrow 23$
$k = 0 \rightarrow 13$
$l = 0 \rightarrow 22$
3 standard reflections every 200 reflections
frequency: 60 min
intensity decay: 7.3%

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)]$ = 0.048
$wR(F^2)$ = 0.142
S = 1.02
7562 reflections
570 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0676P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{\max} < 0.001$
$\Delta\rho_{\max}$ = 0.71 e Å^{-3}
$\Delta\rho_{\min}$ = −0.87 e Å^{-3}

Table 1
Selected bond lengths (Å).

Ru1—C3	1.870 (10)	Ru2—C2	2.129 (8)
Ru1—C4	1.890 (8)	O2—C2	1.171 (8)
Ru1—C2	2.054 (7)	O3—C3	1.150 (9)
Ru1—C31	2.215 (7)	O4—C4	1.140 (9)
Ru1—C17	2.260 (6)	O1—C1	1.130 (9)
Ru1—C24	2.313 (7)	N2—C10	1.297 (8)
Ru1—C38	2.317 (7)	N2—C38	1.456 (8)
Ru1—Ru2	2.736 (3)	N1—C5	1.125 (10)
Ru2—C1	1.869 (9)	N1—C6	1.466 (11)
Ru2—N2	2.046 (6)	C17—C24	1.431 (8)
Ru2—C12	2.077 (7)	C24—C31	1.454 (9)
Ru2—C17	2.103 (6)	C31—C38	1.426 (9)

All the H atoms were placed geometrically and refined riding on their parent atoms. At the end of the refinement, the highest peak in the electron density was 1.45 Å from C3 and the deepest hole was 0.03 Å from Ru1.

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*b*); molecular graphics: *PLATON* (Spek, 1982); software used to prepare material for publication: *SHELXL*97.

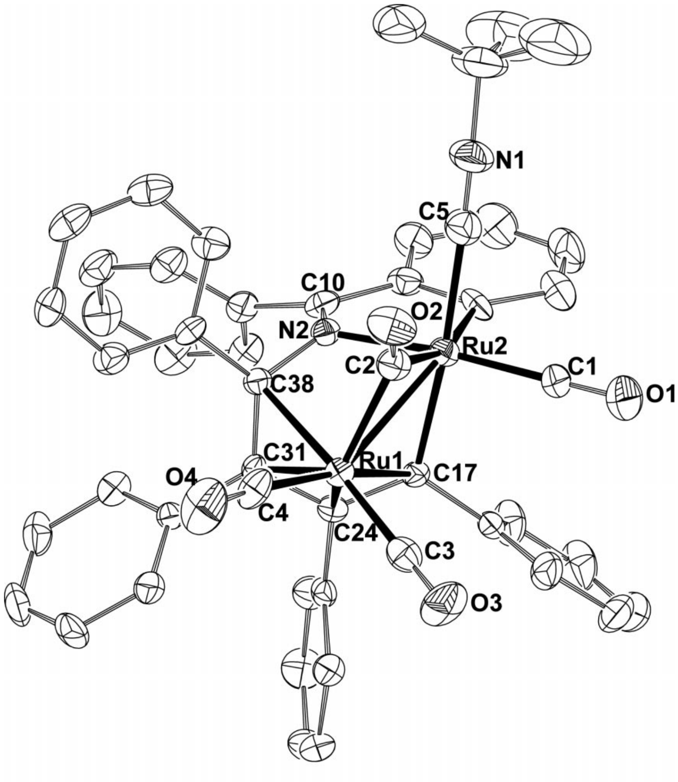


Figure 1
View of a molecule of compound (2). Displacement ellipsoids are shown at the 30% probability level.

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References

Beurskens, P. T., Admiraal, G., Beurskens, G., Bosman, W. P., Garcia-Granda, S., Gould, R. O., Smits, J. M. M. & Smykalla, C. (1992). *The DIRDIF Program System*. Technical Report. Crystallography Laboratory, University of Nijmegen, The Netherlands.

Cabeza, J. A., del Río, I., Moreno, M., Riera, V. & Grepioni, F. (1998). *Organometallics*, **17**, 3027–3033.

Enraf–Nonius (1994). *CAD*-4 *EXPRESS*. Version 5.1/1.2. Enraf–Nonius, Delft, The Netherlands.

García-Granda, S., Aguirre-Pérez, A. & Gutiérrez-Rodríguez, A. (1999). *REFLEX*. Internal Report. X-ray Laboratory, University of Oviedo, Spain.

Sheldrick, G. M. (1997*a*). *SHELXA*. University of Göttingen, Germany.

Sheldrick, G. M. (1997*b*). *SHELXL*97. University of Göttingen, Germany.

Spek, A. L. (1982). *The EUCLID Package. Computational Crystallography*, edited by D. Sayre, p. 528. Oxford: Clarendon Press.

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