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trans-trans-trans-Bis(benzyl isocyanide-*κC*)-dichlorobis(triphenylphosphine-*κP*)-ruthenium(II) dichloromethane solvate

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Key indicators

Single-crystal X-ray study
T = 293 K
Mean σ(C–C) = 0.007 Å
R factor = 0.050
wR factor = 0.132
Data-to-parameter ratio = 20.7

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

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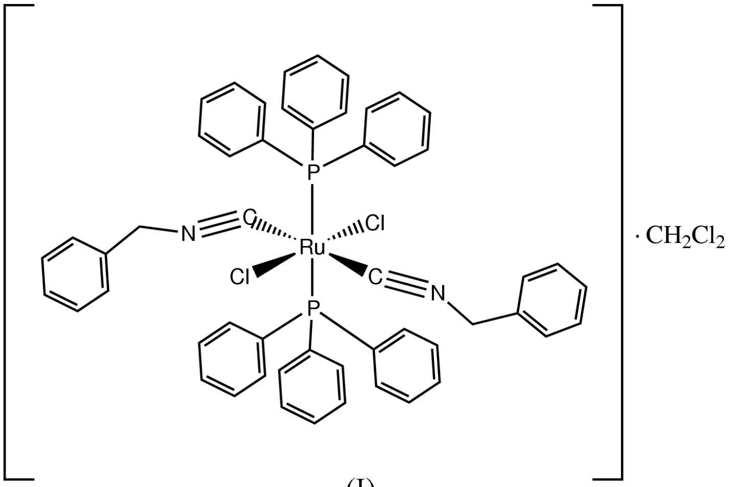
The title compound, $[RuCl_2(C_8H_7N)_2(C_{18}H_{15}P)_2]\cdot CH_2Cl_2$, exhibits the regular octahedral geometry expected for a six-coordinate ruthenium(II) complex. There are two crystallographically independent halves of organometallic molecules in the asymmetric unit, with each metal atom residing on an inversion centre.

Comment

In the course of our studies of the solid-state reactions of organometallic complexes (Nareetsile *et al.*, 2003), we have structurally characterized *ttt*-$RuCl_2(CNBz)_2(PPh_3)_2$ (*t* is *trans*, Bz is benzyl and Ph is phenyl), (I). This complex has previously been characterized in a solvent-free powdered form obtained by rapid solvent evaporation. Solid-state isomerization of the solvent-free material from the *ttt* configuration to the *cct* form (*c* is *cis*) has been examined by differential scanning calorimetry, and IR and NMR studies (Katsuki *et al.*, 1994; Nareetsile *et al.*, 2003). Single crystals suitable for diffraction have been obtained, but they contain dichloromethane. These crystals disintegrate upon heating to 318–328 K, consistent with loss of solvent (Horwood *et al.*, 2003). In this report, the structural characterization of the solvated material is presented.



(I)

Complex (I) crystallizes in space group $P\overline{1}$ with two independent halves of organometallic molecules and one solvent molecule in the asymmetric unit (Fig. 1). No higher symmetry was identified with *PLATON* (Spek, 2003). Each metal atom lies on a centre of inversion. The two molecules show slight differences, with an r.m.s. deviation in the atomic positions of 0.18 Å. As shown in Fig. 2, the structural differences can be

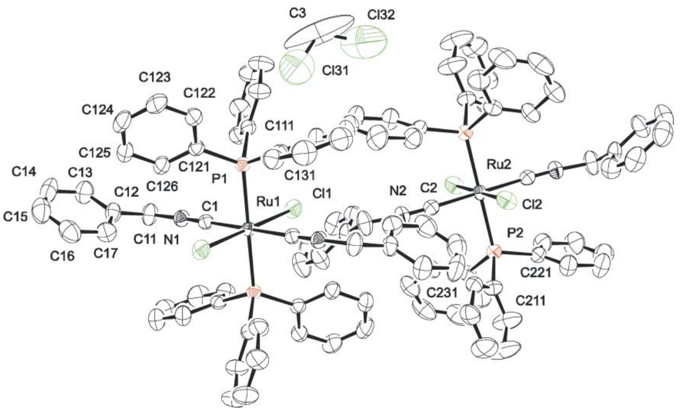


Figure 1
ORTEP-3 diagram (Farrugia, 1997) of (I) showing the atom-labelling scheme for the independent half-molecules and the solvent molecule. Unlabelled atoms are related to labelled atoms by inversion centres located at each metal atom. Displacement ellipsoids are drawn at the 50% probability level. H atoms have been omitted for clarity.

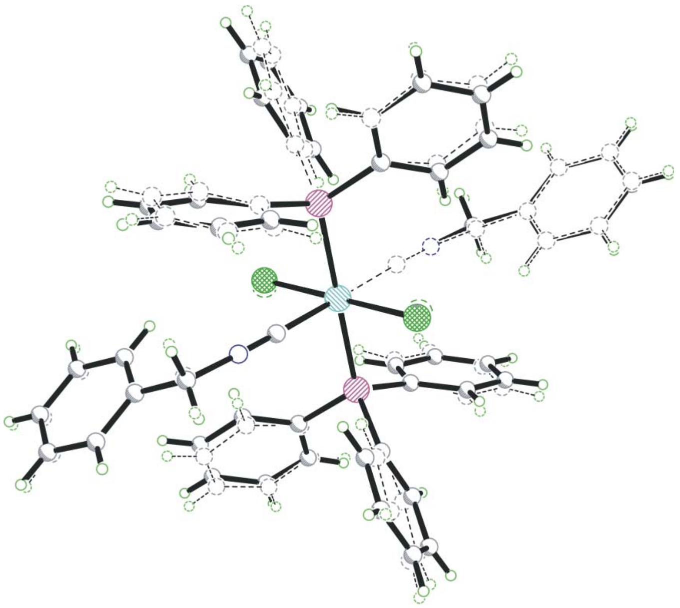


Figure 2
Overlay showing the structural differences for the two crystallographically independent molecules.

attributed to different orientations of the phenyl rings in the triphenylphosphine ligands. The packing diagram (Fig. 3) indicates that the solvent molecules are located within cavities defined by the triphenylphosphine ligands, and are closer to molecules of type 1 (blue) than type 2 (red). The bond lengths and angles for both molecules are normal in comparison with literature values (Tables 1 and 2).

Experimental

The title complex was prepared according to literature methods (Katsuki *et al.*, 1994) and crystallized from a dichloromethane/diethyl ether solution to yield irregular yellow crystals. A spectroscopic analysis has been reported previously (Horwood *et al.*, 2003).

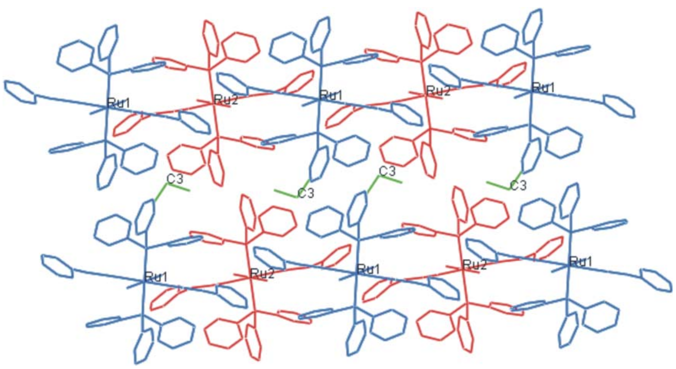


Figure 3
Packing diagram showing the solvent molecules (green) situated between the PPh_3 ligand of the two independent molecules (shown in red and blue). H atoms have been omitted for clarity.

Crystal data

$[RuCl_2(C_8H_7N)_2(C_{18}H_{15}P)_2]\cdot CH_2Cl_2$
$M_r = 1015.73$
Triclinic, $P\overline{1}$
$a = 13.119\ (2)$ Å
$b = 13.314\ (2)$ Å
$c = 14.918\ (3)$ Å
$\alpha = 98.917\ (4)^\circ$
$\beta = 90.842\ (4)^\circ$
$\gamma = 110.201\ (4)^\circ$
$V = 2409.2\ (7)$ Å^3
$Z = 2$
$D_x = 1.400$ Mg m^{-3}
Mo $K\alpha$ radiation
Cell parameters from 983 reflections
$\theta = 2.4$–25.6°
$\mu = 0.65$ mm^{-1}
$T = 293\ (2)$ K
Irregular block, yellow
$0.40 \times 0.26 \times 0.14$ mm

Data collection

Bruker SMART CCD area-detector diffractometer
φ and ω scans
Absorption correction: multi-scan (*SADABS*; Bruker, 1999)
$T_{min} = 0.781$, $T_{max} = 0.914$
16 926 measured reflections
11 613 independent reflections
7270 reflections with $I > 2\sigma(I)$
$R_{int} = 0.032$
$\theta_{max} = 28.3^\circ$
$h = -13 \rightarrow 17$
$k = -17 \rightarrow 16$
$l = -19 \rightarrow 19$

Refinement

Refinement on F^2
$R[F^2 > 2\sigma(F^2)] = 0.050$
$wR(F^2) = 0.132$
$S = 1.01$
11 613 reflections
562 parameters
H-atom parameters constrained
$w = 1/[\sigma^2(F_o^2) + (0.0596P)^2]$ where $P = (F_o^2 + 2F_c^2)/3$
$(\Delta/\sigma)_{max} = 0.001$
$\Delta\rho_{max} = 0.82$ e Å^{-3}
$\Delta\rho_{min} = -0.90$ e Å^{-3}

Table 1
Selected bond lengths and angles (Å, °).

Molecule 1		Molecule 2	
Ru1—C1	1.997 (3)	Ru2—C2	1.985 (4)
Ru1—P1	2.3842 (10)	Ru2—P2	2.3956 (11)
Ru1—Cl1	2.4215 (10)	Ru2—Cl2	2.4264 (10)
N1—C1	1.146 (4)	N2—C2	1.156 (5)
C1—Ru1—P1	90.79 (10)	C2—Ru2—P2	90.68 (11)
C1—Ru1—Cl1	91.10 (11)	C2—Ru2—Cl2	92.08 (11)
P1—Ru1—Cl1	88.79 (3)	P2—Ru2—Cl2	90.66 (3)
Ru1—C1≡N1	178.4 (3)	Ru2—C2≡N2	176.7 (3)
C1≡N1—C11	175.3 (4)	C2≡N2—C21	173.6 (4)

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Table 2
Range of bond lengths (Å) for 22 related complexes from the Cambridge Structural Database.

Ru—P	2.317–2.453	Ru—CN	1.978–1.990
Ru—Cl	2.355–2.452	RuC≡N	1.136–1.155

Notes: a search of the Cambridge Structural Database (Version 5.26; Allen, 2002) for fragments $\mathrm{Ru}(\mathrm{P}R_3)_2X_2L_2$ (X = halide, L = CNR or CO, and R = any alkyl or aryl) resulted in one isocyanide complex (refcode BAGKAW) and 19 carbonyl complexes (BEYGUH, BEYHAO, BOTJAV, CAGPUW, GACQAD, FOGROI, HELREV, HERHER, HOKCOF, JISXOY, NOWQOF, PABZOI, WOXZAK, WOZYEP, YIPMEP, YIPMIT, YUKYEI, ZIGWUH and ZIGXAO). We have recently reported two other isocyanide complexes (Look *et al.*, 2005).

H atoms were positioned geometrically (C—H = 0.93–0,97 Å) and allowed to ride on the parent atoms, with $U_{\rm iso}(\mathrm{H}) = 1.2U_{\rm eq}(\mathrm{C})$. The large residual electron-density peaks are due to the unresolved disorder of the solvent molecules.

Data collection: *SMART* (Bruker, 1998); cell refinement: *SAINT-Plus* (Bruker, 1999); data reduction: *SAINT-Plus*; program(s) used to solve structure: *SHELXS97* (Sheldrick, 1997); program(s) used to refine structure: *SHELXL97* (Sheldrick, 1997); molecular graphics: *ORTEP-3* (Farrugia, 1997); software used to prepare material for publication: *SHELXL97*.

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