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THE FORMATION AND MOLECULAR STRUCTURE OF $(\eta^5\text{-}C_5H_5)Rh(CO)[\overline{C(NMe)CH_2CH_2(NMe)}]$

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Summary

A reaction between $(\eta^5\text{-}C_5H_5)Rh(CO)_2$ and bis(1,3-dimethylimidazolindin-2-ylidene) in methylcyclohexane produces $(\eta^5\text{-}C_5H_5)Rh(CO)[\overline{C(NMe)CH_2CH_2(NMe)}]$ in 53% yield. This mononuclear cyclopentadienylrhodium carbene complex was characterized by elemental analysis, ^1H NMR, $^{13}C\{^1H\}$ NMR, IR, mass spectral, and single-crystal X-ray diffraction methods. The compound $(\eta^5\text{-}C_5H_5)Rh(CO)$-$[\overline{C(NMe)CH_2CH_2(NMe)}]$ crystallizes in the monoclinic space group $P2_1/n$ with a 9.053(3), b 10.256(4), c 12.696(5) Å, β 91.18(3)° and D_{calc} 1.66 g cm^{-3} for $Z = 4$. The structure was refined by least-squares techniques to a final conventional R value of 0.045 using 1739 independent observed ($I \geq 3\sigma(I)$) reflections. The overall structure is that of a two-legged piano stool. The Rh–C(η^5) separations average 2.29(3) Å. The Rh=C and Rh–CO bond lengths are 1.994(7) and 1.795(9) Å, respectively.

Introduction

Rhodium carbene complexes have widespread current interest as potential intermediates in several important processes including organic synthesis [1–3], Fischer–Tropsch chemistry [4], and α-hydrogen migration reactions [5]. Recently, the formation, structure, thermodynamics, and reactivity of $RhCH_2^+$ in the gas phase have also been described [6]. Several mononuclear [7–13] and dinuclear [14–21] carbene complexes of rhodium in different oxidation states and containing various ligands have been reported in the literature. In contrast, there are relatively few mononuclear rhodium carbene complexes containing a cyclopentadienyl ligand [22,23]. Carbenoid cyclopentadienylrhodium(III) complexes have also recently appeared in the literature [24]. To our knowledge there is no reported X-ray crystallographic information for mononuclear cyclopentadienylrhodium carbene complexes [25] nor any carbene complexes derived directly from $(\eta^5\text{-}C_5H_5)Rh(CO)_2$.

Experimental

A. Synthetic procedure

All synthetic operations were conducted using Schlenk tube techniques under an atmosphere of dry, oxygen-free nitrogen. $(\eta^5\text{-}C_5H_5)Rh(CO)_2$ [26] and bis(1,3-dimethylimidazolin-2-ylidene) [27] were prepared according to literature methods. Methylcyclohexane was distilled from CaH_2 under nitrogen.

Reaction of bis(1,3-dimethylimidazolin-2-ylidene) with $(\eta^5\text{-}C_5H_5)Rh(CO)_2$. $(\eta^5\text{-}C_5H_5)Rh(CO)_2$ (0.340 g, 1.52 mmol) and bis(1,3-dimethylimidazolin-2-ylidene) (0.150 g, 0.76 mmol) were heated to 90°C for 15 h in 10 ml of methylcyclohexane. The solution was allowed to cool to 25°C, filtered, and the solvent removed under vacuum (25°C, 10^{-3} mmHg). The resulting residue was taken up in 10 ml of pentane/toluene (4/1), filtered, and cooled to -30°C for 12 h. The resulting yellow-orange crystals were freed from solvent and dried under vacuum (25°C, 10^{-3} mmHg) to afford 0.226 g (53%) of complex I. (Found: C, 44.92; H, 5.14; N, 9.55. $C_{11}H_{15}N_2ORh$ calcd.: C, 44.91; H, 5.14; N, 9.53%). ^1H NMR (C_6D_6): δ 2.46 (s, 4H), 2.98 (s, 6H), 5.40 (s, 5H) ppm. $^{13}C\{^1H\}$ NMR (C_6D_6): δ 39.25, 50.38, 85.60, 196.84, (d, $J(^{103}Rh\text{-}^{13}C)$ 92.9 Hz), 207.72 ppm (d, $J(^{103}Rh\text{-}^{13}C)$ 62.9 Hz).

B. X-ray data collection, structure determination, and refinement for $(\eta^5\text{-}C_5H_5)Rh(CO)[\overline{C(NMe)CH_2CH_2(N}Me)]$

Single crystals of the title compound were sealed under N_2 in thin-walled glass

TABLE 1

CRYSTAL DATA AND SUMMARY OF INTENSITY DATA COLLECTION AND STRUCTURE REFINEMENT

Compound	$(\eta^5\text{-}C_5H_5)Rh(CO)[\overline{C(NMe)CH_2CH_2(N}Me)]$
Mol wt.	294.2
Space group	$P2_1/n$
Cell constants	
a, Å	9.053(3)
b, Å	10.256(4)
c, Å	12.696(5)
β, deg	91.18(3)
Cell vol, Å	1178.5
Molecules/unit cell	4
ρ(calc), g cm^{-3}	1.66
μ(calc), cm^{-1}	13.9
Radiation	Mo-K_α
Max crystal dimensions, mm	$0.23 \times 0.93 \times 1.13$
Scan width	$0.80 + 0.20 \tan \theta$
Standard reflections	(0,8,0)(0,0,12)(8,0,0)
Decay of standards	±2%
Reflections measured	2401
2θ range	$2° \leq 2\theta \leq 50°$
Reflections collected	1739
No. of parameters varied	136
GOF	1.96
R	0.045
R_w	0.053

TABLE 2

FINAL FRACTIONAL COORDINATES FOR $(\eta^5\text{-}C_5H_5)Rh(CO)[\overline{C(NMe)CH_2CH_2(N}Me)]$

Atom	x/a	y/b	z/c
Rh	0.95145(6)	0.07846(6)	0.75621(4)
O	0.7699(9)	0.1949(7)	0.5847(5)
N(1)	0.7023(7)	0.1384(7)	0.8996(5)
N(2)	0.8656(8)	0.2913(7)	0.9027(5)
C(1)	0.8296(8)	0.1785(8)	0.8574(5)
C(2)	0.650(1)	0.225(1)	0.9800(7)
C(3)	0.763(1)	0.334(1)	0.9803(7)
C(4)	0.6274(9)	0.018(1)	0.8771(8)
C(5)	0.997(1)	0.365(1)	0.8805(8)
C(6)	1.040(1)	−0.134(1)	0.7637(8)
C(7)	1.051(1)	−0.078(1)	0.8635(6)
C(8)	1.150(1)	0.027(1)	0.8601(7)
C(9)	1.2036(9)	0.038(1)	0.7582(8)
C(10)	1.127(1)	−0.057(1)	0.6971(6)
C(11)	0.8416(9)	0.1498(9)	0.6520(6)
H(1)[C(2)]	0.551	0.256	0.962
H(2)[C(2)]	0.644	0.182	1.047
H(3)[C(3)]	0.808	0.345	1.049
H(4)[C(3)]	0.718	0.416	0.961
H(5)[C(4)]	0.537	0.023	0.847
H(6)[C(4)]	0.680	−0.057	0.932
H(7)[C(4)]	0.674	−0.036	0.823
H(8)[C(5)]	1.084	0.362	0.922
H(9)[C(5)]	1.061	0.343	0.816
H(10)[C(5)]	0.973	0.458	0.859
H(11)[C(6)]	0.985	−0.212	0.744
H(12)[C(7)]	0.998	−0.107	0.926
H(13)[C(8)]	1.177	0.083	0.920
H(14)[C(9)]	1.278	0.099	0.733
H(15)[C(10)]	1.134	−0.068	0.621

capillaries. Final lattice parameters as determined from a least-squares refinement of $((\sin\theta)/\lambda)^2$ values for 25 reflections ($\theta > 10°$) accurately centered on the diffractometer are given in Table 1. The space group was determined to be centric $P2_1/n$ from the systematic absences.

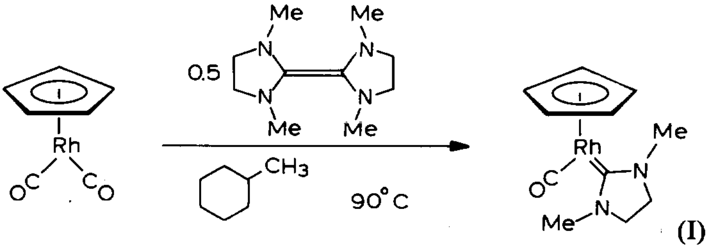
Data were collected on an Enraf–Nonius CAD-4 diffractometer by the θ–2θ scan technique. A summary of the data collection parameters is given in Table 1. The intensities were corrected for Lorentz, polarization effects, and for absorption.

Calculations were carried out with the SHELX system of computer programs [28]. Neutral atom scattering factors for Rh, O, C, and H were taken from ref. 29 and the scattering was corrected for the real and imaginary components of anomalous dispersion [29].

The position of the rhodium atom was found from a Patterson map. A difference Fourier map phased on the rhodium atom readily revealed the positions of the non-hydrogen atoms. Least-squares refinement with isotropic thermal parameters led to $R = \Sigma \| F_o | - | F_c \| / \Sigma | F_o | = 0.087$. The hydrogen atoms bonded to C(2), C(3), and C(6)–C(10) were placed in calculated positions 0.95 Å from the bonded carbons. The methyl hydrogens were located with the aid of a difference Fourier map. The hydrogen atom parameters were not refined. Refinement of the non-hydrogen atoms with anisotropic temperature factors led to final values of $R = 0.045$ and $R_w = 0.053$. A final difference Fourier showed no feature greater than 0.2 e$^-$/Å^3. The weighting scheme was based on unit weights; no systematic variation of $w(|F_o| - |F_c|)$ vs. (F_o) or $(\sin \theta)/\lambda$ was noted. Four reflections (110, $\bar{2}02$, 002, $\bar{1}12$) were omitted from the final cycles of refinement due to secondary extinction. The final values of the positional parameters are given in Table 2 [30].

Results and discussion

The reaction between $(\eta^5\text{-}C_5H_5)Rh(CO)_2$ and bis(1,3-dimethylimidazolin-2-ylidene) in methylcyclohexane at 90°C produced $(\eta^5\text{-}C_5H_5)Rh(CO)[C(NMe)CH_2CH_2(NMe)]$ (I) in 53% yield. Compound I is air-stable in common organic solvents and in the solid state. The IR spectrum (C_6D_6) of I exhibits a strong intensity band at 1920 cm^{-1} characteristic of a low-energy metal carbonyl frequency. Complexes with similar values include $(\eta^5\text{-}C_5H_5)Rh(CO)(PMe_2Ph)$ (1933 cm^{-1}, cyclohexane) [31] and $(\eta^5\text{-}C_5Me_5)Rh(CO)(PMe_3)$ (1910 cm^{-1}, C_6H_6) [32]. This suggests the carbene fragment in I is a strong σ-donor ligand [7,33,34]. The mass spectrum of I exhibits peaks at m/e 294 [54, M^+], 266 [57, $(M - CO)^+$], 223 [26, $(M - CO - C_2H_5N)^+$], 209 [16], 182 [22, $(CpRhCH_2)^+$], 168 [100, $(CpRh)^+$], 144 [30], 103 [19, Rh^+].



(I)

The molecular structure and atom labelling scheme for complex I are presented in Fig. 1. The two-legged piano stool structure consists of a π-bonded cyclopentadienyl seat with the metal coordination completed by the carbene and carbonyl ligands. The C(carbene)–Rh–C(carbonyl) angle, 87.7(3)°, is near the 88.5(4)° found for the C–Rh–C angle in $(\eta^5\text{-}C_5H_4NO_2)Rh(CO)_2$ [35]. The centroid–Rh–C angles are not equivalent (127.8°, centroid–Rh–C(1) and 144.5°, centroid–Rh–C(11)) and apparently reflect a tilt of the cyclopentadienyl ligand toward the carbene. Other bond distances and angles are given in Table 3.

The Rh–C(1)(carbene) bond distance of 1.994(7) Å is similar to the 2.004(4) Å rhodium–carbene separation found for *cis*-[RhCl(COD){(*S*)-CN(CH$_2$CH$_2$CH$_2$)-CHCH$_2$(NMe)}] [12]. Other Rh–C(carbene) bond distances which contain similar carbene fragments include 2.01(2) Å for $[(CF_3)_2CN](Ph_3P)_2Rh[C(NMe)CH_2CH_2(NMe)]$ [36], 1.97(1) Å for $I_3(CO)RhC(Ph)N(Me)C(Ph)=NMe$ [37], and 1.96(1) Å for $Cl_3(Et_3P)_2Rh[C(H)NMe_2]$ [38]. The carbene ligand is planar to within 0.01 Å, with the Rh atom 0.19 Å, and the methyl groups 0.05 Å out of the mean plane defined by C(1), N(1), C(2), C(3) and N(2).

The Rh–C(11) bond distance of 1.795(9) A is considerably shorter than the 1.85(1) Å rhodium–carbonyl separation found for $(\eta^5\text{-}C_5H_4NO_2)Rh(CO)_2$ [35]. The

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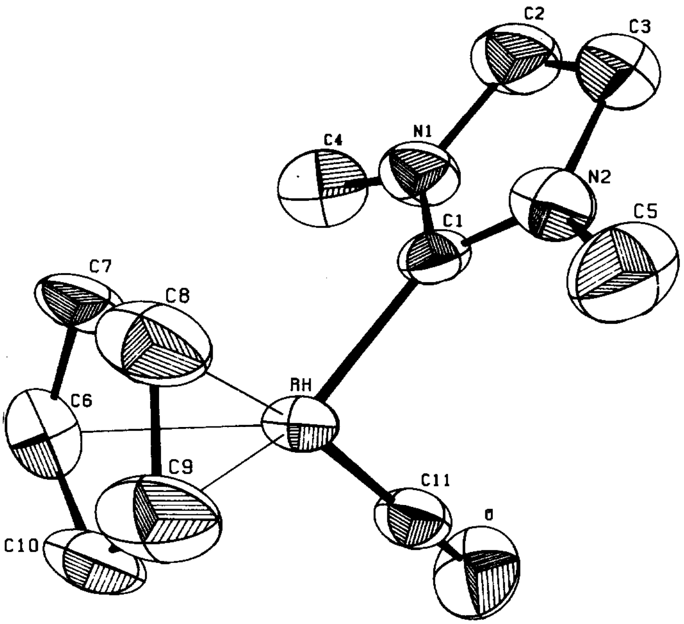

Fig. 1. Molecular structure and atom labelling scheme for $(\eta^5\text{-}C_5H_5)Rh(CO)[\overline{C(NMe)CH_2CH_2(N}Me)]$. The atoms are represented by their 50% probability ellipsoids for thermal motion. Hydrogen atoms have been omitted.

TABLE 3

BOND DISTANCES (Å) AND ANGLES (°) FOR $(\eta^5\text{-}C_5H_5)Rh(CO)[\overline{C(NMe)CH_2CH_2(N}Me)]$

Atoms	Distance	Atoms	Distance
Rh–C(1)	1.994(7)	Rh–C(6)	2.324(9)
Rh–C(7)	2.279(9)	Rh–C(8)	2.276(9)
Rh–C(9)	2.321(8)	Rh–C(10)	2.261(9)
Rh–C(11)	1.795(9)	O–C(11)	1.16(1)
N(1)–C(1)	1.35(1)	N(1)–C(2)	1.44(1)
N(1)–C(4)	1.43(1)	N(2)–C(1)	1.33(1)
N(2)–C(3)	1.44(1)	N(2)–C(5)	1.45(1)
C(2)–C(3)	1.51(2)	C(6)–C(7)	1.39(1)
C(6)–C(10)	1.41(1)	C(7)–C(8)	1.40(1)
C(8)–C(9)	1.39(1)	C(9)–C(10)	1.41(1)
Cent [a]–Rh	1.96		
Atoms	**Angle**	**Atoms**	**Angle**
C(1)–Rh–C(11)	87.7(3)	C(1)–N(1)–C(2)	113.1(8)
C(1)–N(1)–C(4)	126.1(7)	C(2)–N(1)–C(4)	120.6(7)
C(1)–N(2)–C(3)	113.8(8)	C(1)–N(2)–C(5)	124.5(7)
C(3)–N(2)–C(5)	121.6(8)	Rh–C(1)–N(1)	126.1(6)
Rh–C(1)–N(2)	126.3(6)	N(1)–C(1)–N(2)	107.3(7)
N(1)–C(2)–C(3)	102.8(7)	N(2)–C(3)–C(2)	102.9(8)
C(7)–C(6)–C(10)	107(1)	C(6)–C(7)–C(8)	108.7(8)
C(7)–C(8)–C(9)	108.8(9)	C(8)–C(9)–C(10)	106.4(9)
C(6)–C(10)–C(9)	109.1(8)	Rh–C(11)–O	179.4(8)
Cent–Rh–C(1)	127.8	Cent–Rh–C(11)	144.5

[a] Cent refers to the centroid of the plane defined by atoms C(6)–C(10).

TABLE 4

DISTANCES (Å) IN Rh MOLECULES CONTAINING CYCLOPENTADIENYL, CARBENE, AND CARBONYL LIGANDS

Compound	Rh–C(carbene) [a]	Rh–C(π) [b]	Rh–Cp [c]	Rh–C(=O) [a]	C=O [a,d]	Reference
$(\eta^5\text{-}C_5H_5)Rh(CO)[C(NMe)CH_2CH_2(NMe)]$ (I)	1.994(7)	2.29(3)	1.96	1.795(9)	1.16(1)	This study
$[RhCl(COD)\{(S)\text{-}CN(CH_2CH_2CH_2)CHCH_2(NMe)\}]$	2.004(4)	–	–	–	–	12
$[(CF_3)_2CN](Ph_3P)_2Rh[C(NMe)CH_2CH_2(NMe)]$	2.01(2)	–	–	–	–	36
$I_3(CO)RhC(Ph)N(Me)C(Ph)=NMe$	1.97(1)	–	–	1.89(1)	1.11(2)	37
$Cl_3(Et_3P)Rh[C(H)NMe_2]$	1.96(1)	–	–	–	–	38
$(\eta^5\text{-}C_5Me_5)Rh[C(C_6H_4Me)NHCH_2CMe_3]Cl_2\cdot 0.5[(\eta^5\text{-}C_5Me_5)RhCl_2]_2$	2.03(1)	–	1.81	–	–	42
$(\eta^5\text{-}C_5H_4NO_2)Rh(CO)_2$	–	–	–	1.85(1) 1.86(1)	1.13(1) 1.15(1)	35
$(Ph_3P)(acac)Rh(CO)$	–	–	–	1.801(8)	1.15(1)	40
$\mu\text{-}CH_2[(\eta^5\text{-}C_5H_5)Rh(CO)_2]_2$	2.029(4) [e] 2.055(4) [e]	2.26(3) 2.26(2)	1.91 1.92	1.831(4) 1.833(4)	1.144(5) 1.135(6)	14
$(\eta^5\text{-}C_5H_5)Rh(\eta^4\text{-}C_4Ph_4)$	–	2.214(4)	1.87	–	–	41

[a] Distances and deviations. [b] Average distances. [c] Distance to ring plane. [d] Terminal carbonyl. [e] To bridging CH_2 group.

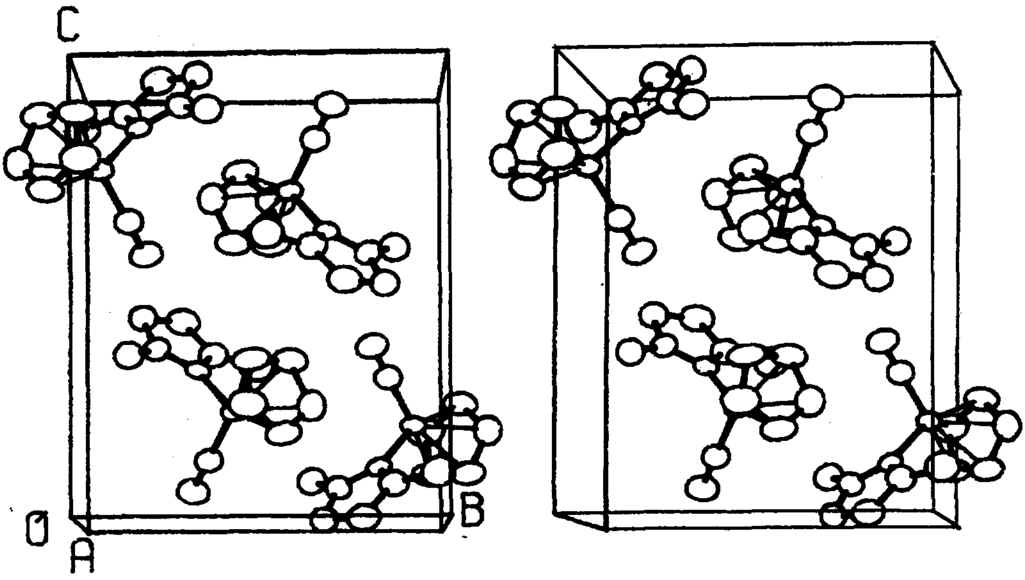

Fig. 2. Stereoscopic view of the unit cell contents.

cyclopentadienyl ligand is a better π-donor than the nitrocyclopentadienyl ligand [35,39]. Likewise, the carbene ligand $\overline{C(NMe)CH_2CH_2(N}Me)$ is a better σ-donor ligand than carbon monoxide [7,33,34]. These two factors contribute to the shorter Rh–C(carbonyl) bond distance in I. A similar Rh–C(carbonyl) bond distance of 1.801(8) Å was found in $(Ph_3P)(acac)Rh(CO)$ [40] and Rh–C(carbonyl) separations of 1.831(4) and 1.833(4) Å were observed in μ-$CH_2[(\eta^5$-$C_5H_5)Rh(CO)_2]_2$ [14].

The cyclopentadienyl ligand is planar to within 0.03 Å. The Rh–C(π) separations average 2.29(3) Å. This is about the same as the 2.26(3), 2.26(2) Å values observed for the Rh–C(π) distances in μ-$CH_2[(\eta^5$-$C_5H_5)Rh(CO)_2]_2$ [14] and slightly longer than the 2.214(4) Å found for $(\eta^5$-$C_5H_5)Rh(\eta^4$-$C_4Ph_4)$ [41]. A comparison of bond distances in various rhodium molecules containing cyclopentadienyl, carbene, and carbonyl ligands is presented in Table 4. A stereoscopic view of the unit cell contents is given in Fig. 2. No unusually short intermolecular contacts were noted *.

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* Supplementary material. Tables of thermal parameters, least-squares plane results, and observed and calculated structure factors for I are available.

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