The Synthesis, Structural Characterization and Conformational Analysis of (1,3-Bis(2-methyl-4-diethylaminophenyl)imidazolidin-2-ylidene)chloro(1,5-cyclooctadiene)rhodium(I)

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A four-coordinated Rh(I) complex with a new heterocyclic carbene ligand, functionalized by amino donor pendants, **4**, was synthesized and characterized by elemental analyses, NMR and IR spectroscopy, and the molecular structure of the title compound has been determined by X-ray crystallography. Crystallographic data: monoclinic, $P2_1/m$, a = 7.9307(5), b = 25.0061(12), c = 8.0780(6) Å, $\beta = 101.366(6)^{\circ}$, V = 1570.58(17) Å³, $\rho_{calc} = 1.3515(1)$ g cm⁻³, Z = 2. The experimentally obtained structural parameters for compound **4** compare reasonably well with those calculated at the semi-empirical ZINDO/1 level of theory carried out to elucidate conformational flexibility and steric hindrances.

Key words: N-Heterocyclic Carbenes, Rhodium Complexes, Carbene Ligand, Crystal Structure, ZINDO/1

Introduction

The renaissance of metal complexes of *N*-heterocyclic carbenes began after their use as alternatives for phosphine complexes used in homogeneous catalysis [1-5]. The use of CC-saturated imidazolidin-2ylidene ligands results in even higher catalytic activity. Examples of such reactions include the Heck olefination of haloarenes [6], hydroformylation [6], the asymmetric hydrosilylation of acetophenone [7, 8], hydrogenation [9] and cyclopropanation of alkenes [10].

The nature of the *N*-substituents of the carbene ligand has a pronounced effect upon the catalytic activity of the complexes [10, 11]. On the other hand, biphasic catalysis consisting of a water-phase containing catalytic species and a non-miscible organic phase continues to attract interest in view of industrial applications [12]. For this purpose a number of attempts have been made to introduce hydrophilic functional groups on convenient ligands, mainly phosphines [12–15]. Despite their similar catalytic behaviour, examples of hydrophilic carbene complexes are rare [16, 17]. The employment of highly reactive nucleophiles as well as electrophiles at different stages of the imidazolidin-2ylidene metal complex synthesis precludes the presence of many types of functional groups [17, 18]. As a possible system for this research, we have chosen 4-diethylamino-2-methylphenyl as the *N*-substituent. The presence of peripheral $-NEt_2$ group is expected to improve water solubility through quaternization of this group.

Experimental Section

Materials and methods

All manipulations were performed by using Schlenktype flasks under dry argon and standard high vacuumline techniques. The solvents were analytical grade and distilled after drying. NMR spectra were recorded at 297 K on a Varian instrument at 400 MHz (¹H), 100,56 MHz (¹³C). Elemental analyses were carried out by the analytical service of TUBITAK with a Carlo Erba Strumentazione Model 1106 apparatus. Crystallographic data from single crystal X-ray diffraction have been collected with the STOE IPDS2 diffractometer.

Preparation of the title compound

A mixture of 1,3-bis(2-methyl-4-diethylaminophenyl) imidazolinium chloride (0.37 g, 0.86 mmol) and [Rh($\mu\text{-}$

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 $\frac{1}{1}$ 2 3 4 Fig. 1. Synthesis of **4**. (i) Glyoxal, EtOH, RT; (ii) NaBH₄, EtOH, 78 °C; (iii) CH(OEt)₃, NH₄Cl, 130 °C; (iv) [Rh(μ -OMe)(1,5-COD)]₂, 110 °C, PhMe.



Fig. 2. ORTEP3 drawing of the title compound showing the atomic numbering scheme for non-hydrogen atoms. Displacement ellipsoids of non-H atoms are shown at the 30% probability level. [(i): x, y+3/2, z)

OMe)(1,5-COD)]₂ (0.208 g, 0.43 mmol) in toluene (5 cm³) was heated under reflux for 2 h. The solution was cooled to room temperature and hexane (15 ml) was added. The solid was filtered off and recrystallized from CH₂Cl₂/Et₂O (1 cm³/3 cm³), filtered and dried. Yield: 0.360 g. (68%, m. p.: 248–250 °C. Analysis for RhClC₃₃H₄₈N₄: calcd. C 62.19, H 7.59, N 8.79) (found: C 62.23, H 7.76, N 8.83). ¹H NMR (400 MHz, CDCl₃): δ = 7.95 (d, *J* = 8.58 Hz, 2H, C₆H₃); 6.68 (dd, *J* = 8.77 Hz, 2H, C₆H₃); 6.53 (d, *J* = 2.73, 2H, C₆H₃); 4.57 (s, 2H, COD_{vinyl}); 3.96 (t, *J* = 18.33 Hz, 2H, NCH₂CH₂); 3.84 (t, *J* = 18.33, 2H, NCH₂CH₂); 3.42-3.37 (m, 8H, NCH₂CH₃); 1.75–1.52 (m, 8H, COD_{vinyl}); 1.2 (t, *J* = 14.04, 12H, NCH₂CH₃). ¹³C NMR

(d, CDCl₃): d 214.80 (d, J = 47.26 Hz, C_{carb}=Rh); 147.53, 135.26, 131.87, 128.92, 113.25, 110.16 (C_{aren}); 97.16 (d, J = 6.83 Hz, COD(CH=CH); 66.99 (d, J = 15.28 Hz, COD(CH=CH); 52.8 (NCH₂CH₂N); 44.76 (NCH₂CH₃); 32.49 (COD(CH₂)); 28.40 (COD(CH₂)); 19.18 (2-CH₃-C₆H₃); 12.8 (NCH₂CH₃).

X-ray crystallography

Suitable crystals were obtained directly from the synthesis as yellow needles. The structure was solved by the Patterson method (SHELXS-97) [19] and refined by fullmatrix least squares techniques (SHELXL-97) [20]. Details

Crystal data		Data collection		Refinement	
Chemical formula	RhClC33H48N4	Diffractometer	STOE IPDS2	Unique reflections	3162
Formula weight	639.11	Diff. meth.	rotation	Refined parameters	197
Cryst. system	monoclinic	Abs. corr. type	integration	R_1 (for all data)	0.0481
Space group	$P2_1/m$ (No:11)	Intensity decay	< 1%	wR_2 (for all data)	0.0826
Wavelength [Å]	0.71073 (Mo-K _α)	Temperature [K]	293(2)	$R_1(F_{\rm o} > 4\sigma(F_{\rm o}))$	0.0367
a [Å]	7.9307(5)	θ_{\min} [°]	1.63	$wR_2(F_{\rm o}>4\sigma(F_{\rm o}))$	0.082
b [Å]	25.0061(12)	$\theta_{\rm max}$ [°]	28.30	GoF	0.859
c [Å]	8.0780(6)	T_{\min}	0.6713	Refines. with $(F_{\rm o} > 4\sigma(F_{\rm o}))$	2261
β [°]	101.366(6)	$T_{\rm max}$	0.8675	Deepest hole (e Å ⁻³)	-0.62
V [Å ³]	1570.58(17)	h, k, l ranges	$-10 \rightarrow 10, 0 \rightarrow 33, 0 \rightarrow 10$	Highest peak (eÅ ⁻³)	0.81
Z/F(000)	2/672	Meas. frac.theta max.	0.996	Mean shift / esd	0.0020
$\rho_{\rm calc} [{ m g}{ m cm}^{-3}]$	1.352	Meas. frac.theta full	0.993	Max. shift / esd	0.0140
μ (Mo-K _{α}) [cm ⁻¹]	6.57	Method of abs. corr.	Psi-scan	Max. shift	0.0010
Crystal size (mm)	$0.60 \times 0.53 \times 0.22$	Monochromator	plane graphite		

Table 1. Crystal data and structure refinement details for the title compound.

Table 2. Atomic fractional coordinates and equivalent isotropic displacement parameters of the atoms in the asymmetric unit (in $Å^2$ with ESDs in parentheses). U_{eq} is defined as one third of the trace of the orthogonalized U_{ij} tensor.

Atom	x	У	z	$U_{\rm eq}$ [Å]
Rh1	0.34683(5)	1/4	0.35422(4)	0.0365(1)
Cl1	0.52785(14)	1/4	0.14913(14)	0.0502(4)
N1	0.2493(4)	-0.00732(10)	0.2432(4)	0.0766(13)
N2	0.0635(3)	0.20680(8)	0.0884(3)	0.0404(8)
C1	0.1098(3)	0.15220(10)	0.1321(4)	0.0391(10)
C2	-0.0008(4)	0.11912(12)	0.1985(4)	0.0496(11)
C3	0.0483(5)	0.06641(12)	0.2336(5)	0.0588(13)
C4	0.2030(4)	0.04564(11)	0.2072(4)	0.0530(11)
C5	0.3120(4)	0.08011(11)	0.1428(4)	0.0488(11)
C6	0.2642(4)	0.13261(11)	0.1057(4)	0.0430(10)
C7	-0.1718(5)	0.13802(14)	0.2298(6)	0.0762(14)
C8	0.1675(6)	-0.03953(17)	0.3596(6)	0.0905
C9	0.0369(7)	-0.0705(2)	0.2673(8)	0.140(3)
C10	0.3833(6)	-0.03279(15)	0.1747(6)	0.0674(15)
C11	0.5562(6)	-0.02982(18)	0.2867(6)	0.095(2)
C12	0.2021(4)	0.22236(14)	0.5260(4)	0.0630(12)
C13B	0.306(2)	0.1772(9)	0.636(3)	0.071(5)
C13A	0.307(2)	0.1997(10)	0.685(3)	0.072(5)
C14	0.4875(6)	0.18904(17)	0.6766(5)	0.0880(17)
C15	0.5604(4)	0.22284(12)	0.5537(4)	0.0553(11)
C16	0.1422(5)	1/4	0.1658(5)	0.0342(11)
C17	-0.0707(5)	0.21965(13)	-0.0596(5)	0.0556(11)

of the crystal data^{*}, parameters for data collection, the solution and refinement procedures are given in Table 1. All non-hydrogen atoms were anisotropically refined and an extinction correction (extinct. coeff. = 0.00746) was applied in the refinement process. Scattering factors were taken from International Tables for X-ray Crystallography [21]. There is half an independent molecule in the asymmetric unit.

Computational details

The initial molecular structure obtained from the Xray crystallographic study was optimized by ZINDO/1 [22] semi-empirical quantum mechanical calculations at the spin-Restricted Hartree-Fock (RHF) level [23, 24] without any symmetry constraints. ZINDO/1 has been parameterized to generate geometries of molecules including transition metals. The optimized geometry was obtained by the application of the sequential conjugate gradient method, called the Polak-Ribiere [25] and Eigenvector Following [26] optimization algorithm with a convergence limit of 0.01 kcal/mol and an RMS gradient of 0.05 kcal/Å mol. The semi-empirical calculations were carried out using the HyperChem 6.0 program package [27]. After geometry optimization, to obtain the molecular energy profile versus the selected torsion angle [T(C17-N2-C1-C6)], this torsion angle was varied from -180° to $+180^{\circ}$ in steps of 10° , and the energy profile of the molecule was obtained by single point calculations on the computed potential energy surface. Probability-weighted coordinates of C13, which is disordered in the crystal, were used in the computations.

Results and Discussion

The key intermediates 1-3 were obtained readily by the condensation of 2-methyl-4-diethyla-minoaniline with glyoxal. The resulting Schiff base 1 underwent a smooth reduction providing the ethylenediamine 2 which was converted to the corresponding imidazolinium chloride by treatment with CH(OEt)₃ in the presence of NH₄Cl. Next, the salt 3 was deprotonated in situ with [Rh(μ -OMe)(1,5-COD)]₂ to afford the desired complex 4.

^{*}CCDC 243199 contains the supplementary crystallographic data for this paper. These data can be obtained free of charge *via* www.ccdc.cam.ac.uk/data_request/cif, by emailing data_request@ccdc.cam.ac.uk, or by contacting The Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: +44 1223 336033.

Elemental analyses and NMR spectra were in agreement with the proposed structure. The clearest spectroscopic evidence identifying **4** as a carbene complex is the appearance of a highly deshielded ¹³C NMR doublet for C_{carb} at 214.80 ppm ($J_{RhC} = 47.3$ Hz). The IR spectrum of **4** shows a strong band at 1520 cm⁻¹ attributable to $v(CN_2)$. The absorption bands in the 1400–1600 cm⁻¹ regions arise from the skeletal vibrations of the aromatic rings of the ligand.

A thermal ellipsoid drawing of compound 4 is shown in Fig. 2 [28]. Fractional atomic coordinates and equivalent isotropic displacement parameters for the non-hydrogen atoms are listed in Table 2. Single crystal X-ray diffraction analysis confirmed the monomeric structure of 4. Several features of this structure, including the planar geometry around the Rh atom and the short N-C_{carb} distances (~ 1.34 Å), are consistent with a carbene complex structure. Rh1, Cl1 and C16 atoms have special positions (y = 1/4). No chiral centers are found in the molecule, which has two-fold symmetry on the mid points of the C12-C12ⁱ and C15-C15ⁱ bonds. Rh1, Cl1 and C16 are located on the mirror plane and the whole molecule has mirror plane through the Rh1-C16 bond. Weighted average ring bond distances in the cyclooctadiene and carbene rings are 1.4395 and 1.3908 Å, respectively. The COD ring exhibits a boat conformation with the Rh-C_{COD} distances ranging from 1.994(4) to 2.203(4) Å. The bond distances Rh1-C12 and Rh1-C12ⁱ are shorter than Rh1-C15 and Rh1-C15¹. These results agree with the literature values [29, 30]. Bond distances between C and N atoms in the N-heterocyclic carbene ring have considerable differences. Although both C16-N2 and C17-N2 are single bonds, C16-N2 [1.339(3) Å] is shorter than the others. Similar arguments can be proposed for the C17ⁱ-N2ⁱ and C16-N2ⁱ bonds. The theoretical studies indicate that the stability of these carbenes is due to electron donation from the nitrogen lone pairs into the formally vacant $p(\pi)$ orbital of the carbene carbon [31]. Indeed, excess charge located on the carbene carbon atom linked to rhodium is relatively higher than on other carbon atoms in the molecule, whereas excess charges located on the nitrogen atoms in the carbene ring are lower than on other nitrogen atoms in the molecule (see Fig. 5). The Cl-Rh-C_{carbene} angle of 88.89(12)° is in consistence with this type of compounds in the literatures [1, 2, 6, 16, 32-34]. The dihedral angle between planes defined by Rh-C12-C12ⁱ and Rh-C15-C15ⁱ is $87.3(3)^{\circ}$. The deviation of the chlo-



Fig. 3. Molecular packing diagram of the unit cell.



Fig. 4. Calculated energy profile from ZINDO/1 of the title molecule *versus* the torsion angle T(C17-N2-C1-C6).

rine atom from the plane Rh-C12-C12ⁱ is 0.045(1) Å, and the deviation of C_{carbene} from the plane Rh-C15-C15ⁱ is 0.005(3) Å. When midpoints of C12-C12ⁱ and C15-C15ⁱ are considered, it can be stated that the title compound is a cis isomer. These midpoints, the chlorine atom and Ccarbene exhibit a slight distortion from exact planarity at the rhodium atom. The coordination geometry about the rhodium center is approximately square-planar, as expected for a 16 \bar{e} Rh(I) complex. Throughout the refinement process, C13 was treated as disordered. C13 is located in C13A and C13B positions with probabilities of 48.2% and 51.8%. There is weak π - π stacking [symm. code: -x, -y, -z] among 2-methyl-4-diethylaminophenylrings (centroid, Cg16) in the crystal. The distance between the ring centroids is 5.9731(18) Å, and two C-H... π interactions are also found in the structure. For the C9---H23b...Cg16 interaction [sym. code: -x, -y, -z], the H23b...Cg16,

1.406

1.391

study and the ZINDO/1 senii-empirical method.								
-	 Bond distances — 		— B	ond angles –	-	— Torsi	on angles —	
	X-ray	ZINDO/1		X-ray	ZINDO/1		X-ray	ZINDO/1
C17-C17 ⁱ	1.518(5)	1.473	Cl1-Rh1-C16	88.89(12)	98.15	C15-Rh1-C12-C13B	-27.2(9)	-24.05
N2-C17	1.471(5)	1.414	Cl1-Rh1-C15	91.53(9)	85.65	C15-Rh1-C12-C12	106.2(2)	109.09
N2-C16	1.339(3)	1.337	Cl1-Rh1-C12	160.61(10)	162.77	C16-Rh1-C12-C13B	135.8(9)	137.93
Rh1-C16	1.994(4)	1.887	C12-Rh1-C15	81.70(12)	88.20	C16-Rh1-C12-C12	-90.73(18)	-88.93
Rh1-C12	2.085(3)	2.047	C12-Rh1-C16	92.06(14)	83.15	C12i-Rh1-C12-C13B	-133.4(9)	-133.14
Rh1-C15	2.202(3)	2.068	C15-Rh1-C16	162.03(8)	162.24	C15 ⁱ -Rh1-C12-C13	-61.3(9)	-59.86
Rh1-Cl1	2.3965(12)	2.284	N2-C16-N2 ⁱ	107.5(3)	110.17	Cl1-Rh1-C15-C14	-148.5(2)	-150.05
C12-C12 ⁱ	1.382(5)	1.345	C16-N2-C17	113.5(2)	110.23	Cl1-Rh1-C15-C15	90.50(18)	89.59
C15-C15 ⁱ	1.358(4)	1.341	N2-C17-C17 ⁱ	102.6(3)	104.63	C12-Rh1-C15-C14	13.2(2)	10.08
C12-C13A/B	1.50(2)/1.57(2)	1.450	C1-C2-C7	122.5(3)	122.38	C12-Rh1-C15-C15	-107.7(2)	-110.28
C13A/B-C14	1.471(18)/1.443(18)	1.464	N2-C1-C2	120.7(2)	122.79	C12 ⁱ -Rh1-C15-C14	50.2(2)	46.64
C14-C15	1.503(5)	1.452	N1-C8-C9	109.7(4)	116.18	C15 ⁱ -Rh1-C15-C14	121.0(3)	120.36

116.9(3)

113.9(4)

117.35

115.98

Cl1-Rh1-C16-N2

C12-Rh1-C16-N2

C8-N1-C10

N1-C10-C11

Table 3. A comparison of selected bond distances [Å], bond angles and torsion angles $[^{\circ}]$ from the X-ray crystallographic study and the ZINDO/1 semi-empirical method.



Fig. 5. Calculated excess charges on the non-hydrogen atoms by ZINDO/1 method.

Table 4. Calculated parameters of the optimized title compound.

1.440(3)

1.390(4)

N2-C1

N1-C4

Total energy [kcal/mol]	-208320.373
Total energy (a.u.)	-331.973
Binding energy [kcal/mol]	-27666.488
Isolated atomic energy [kcal/mol]	-180653.885
Electronic energy [kcal/mol]	-1944804.692
Core-core interaction [kcal/mol]	1736484.318
Heat of formation [kcal/mol]	-18912.232
Molecular point group	C_1
Point charge dipole (debye)	6.922
sp hybrid dipole (debye)	2.752
pd hybrid dipole (debye)	0.069
Total dipole (debye)	9.486

C9...Cg16 distances and the C9---H23b...Cg angle are 3.1718, 3.634(6) Å and 111.46° , respectively. In the related interaction for the ring 2, which has also

the same C9...Cg distance, C9---H23c...Cg16 [sym. code: -x, -y, -z], the H23c...Cg16 distances and the C9---H23c...Cg16 angle are 3.2244 Å and 107.78° respectively.

Although the optimized geometry of the molecule is in C_1 molecular point symmetry group, the crystallographic structure is in C_S point symmetry group. Except for this difference, no considerable difference is found in the study. Some geometrical parameters obtained by the different methods are compared in Table 3, and energy parameters are given in Table 4.The calculated energy profile from ZINDO/1 versus the torsion angle T(C17-N2-C1-C6) is given in Fig. 4. In this profile, the prominent peak near to 0° is due to steric hindrance between the methyl group linked to C2 and the COD ring, especially the rhodium atom. The other

92.10

106.48

88.3(3)

111.1(3)

peaks are also primarily due to the rhodium atom and COD, not due to the position or orientation of the chlorine atom.

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