

β -DIKETONE CHELATES OF BIS(ETHYLENE)RHODIUM AND PRODUCTS OF EXCHANGE REACTIONS WITH CHLORO-SUBSTITUTED DIENES

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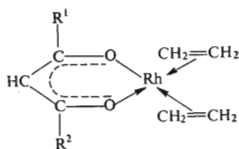
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Complexes of the type $(R^1COCHCOR^2)Rh(C_2H_4)_2$ were synthesized and subsequently transformed into complexes of the type $(R^1COCHCOR^2)Rh(Y)$ via exchange reactions with 1,6-dichloro-1,5-cyclooctadiene, 1-chloro-1,3-butadiene, 2-chloro-1,3-butadiene and 2,3-dichloro-1,3-butadiene (Y). The IR and NMR spectra of the complexes are used to discuss the coordination of chloro-substituted dienes with Rh(I).

An intensive study of the coordination compounds of Rh(I) with olefins and dienes brought about a number of fundamental findings concerning the character and reactions of complex compounds of the transition metals, and also led to the use of rhodium compounds as catalysts of important industrial reactions, such as isomerization, dimerization, and polymerization¹. It can be expected, using conceptions of the character of the bond existing between unspecified donors and the central metal atom, that this bond will be affected both by substitution on the multiple bonds of unsaturated ligands and by the other ligands coordinated with the central metal atom, as well as by the nature of substituents in these ligands^{2,3}. The influence of the induction effect of the substituent in an olefin upon the π -bond component of the coordination bond olefin-Rh(I) was studied by Cramer⁴, who demonstrated that the electronegative substituents in the molecule of an unspecified donor, which reduce the electron density of the multiple bonds, weaken the σ -bond component of the coordination bond, at the same time facilitating the formation of the π -bond component. So far, however, no rhodium complexes of chloro-substituted dienes have been described in the literature available to us.

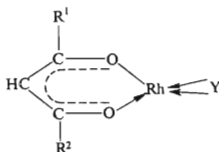
In the present paper, the synthesis of a number of β -diketone complexes of the type $(R^1COCHCOR^2)Rh(Cl\text{-diene})$ have been described, and the effect of the structure of chloro diene and β -diketone ligand upon the coordination of dienes with Rh(I) has been evaluated. The reaction of dichlorotetraethylenedirhodium (I) with the selected β -diketones yielded complexes of the type A(II–VIII), whose properties and analyses are summarized in Table I. They are microcrystalline, yellow to brown-red in colour. The majority of these compounds did not melt even at 350°C; temperatures above 170°C brought about a gradual decomposition. A partial decomposition of the complexes took place also *in vacuo*; it made impossible the determination of the mass spectra of the complexes and their purification by sublimation. The complexes are soluble in organic solvents to a limited extent and monomeric in acetone solutions,

according to the determined values of molecular weights (Table I). Attempts to prepare solutions having the necessary concentration in solvents suitable for measuring the NMR spectra have been unsuccessful.



Type A (II–VIII)

- II, IX, XVI–XVIII: $R^1 = R^2 = \text{CH}_3$
 III, X: $R^1 = \text{CH}_3$, $R^2 = \text{C}_6\text{H}_5$
 IV, XI: $R^1 = R^2 = \text{C}_6\text{H}_5$
 V, XII: $R^1 = R^2 = \text{C}_6\text{H}_4\text{CH}_3$
 VI, XIII: $R^1 = R^2 = \text{C}_6\text{H}_4\text{OCH}_3$
 VII, XIV: $R^1 = R^2 = \text{C}_6\text{H}_4\text{Cl}$
 VIII, XV: $R^1 = R^2 = \text{C}_6\text{H}_4\text{NO}_2$



Type B (IX–XVIII)

- IX–XV: $Y = 1,6\text{-dichloro-1,5-cyclooctadiene}$
 XVI: $Y = 1\text{-chloro-1,3-butadiene}$
 XVII: $Y = 2\text{-chloro-1,3-butadiene}$
 XVIII: $Y = 2,3\text{-dichloro-1,3-butadiene}$

TABLE I

Properties and Analyses of the Type A Complexes

Complex	Colour (Yield, %)	Solubility	Mol. weight calc./found	Calc./Found	
				% C	% H
II ^a	orange (90)	EtOH, Et ₂ O, Me ₂ CO CHCl ₃ , C ₆ H ₆	258.1 —	41.9 41.81	5.87 5.85
III	orange (57)	EtOH, Et ₂ O, Me ₂ CO, CHCl ₃ , C ₆ H ₆	320.2 —	52.3 52.68	5.33 5.47
IV	yellow (79)	CHCl ₃ , Me ₂ CO, —	382.3 —	59.98 60.02	5.02 5.11
V	yellow (66)	Et ₂ O, MeCO, CHCl ₃ , C ₆ H ₆	410.3 430 ^b	61.5 61.60	5.66 5.74
VI	yellow (77)	CHCl ₃ , Me ₂ CO, C ₆ H ₆	442.3 460 ^b	57.1 56.45	5.25 5.25
VII	orange (89)	Et ₂ O, Me ₂ CO, CHCl ₃ , C ₆ H ₆	451.2 480 ^b	50.7 50.95	3.83 ^c 3.91
VIII	red (94)	CHCl ₃	472.3 —	48.3 48.06	3.64 ^d 3.94

^a Ref. 5. ^b Determined in an acetone solution using osmometry in the vapour phase. ^c Chlorine content: calc.: 16.29%; found: 16.23%. ^d Nitrogen content: calc.: 5.93%; found: 5.93%.

Bis(ethylene)-2,4-pentanedionerhodium (*II*) is a thermodynamically stable complex which from the kinetic point of view is very reactive, since the number of electrons in the sphere of Rh(I) is lower by two electrons compared to the electron configuration of a corresponding inert gas⁵. This circumstance allowed to carry out reactions of the complex *II* with 1-chloro-1,3-butadiene, 2-chloro-1,3-butadiene and 2,3-dichloro-1,3-butadiene, and similarly reactions of the individual complexes *II*–*VIII* with 1,6-dichloro-1,5-cyclooctadiene, accompanied by formation of the type B complexes (Table II), under conditions where the side reactions of chlorosubstituted dienes are considerably limited⁶. Complexes *IX*–*XV* containing a diene ligand 1,6-dichloro-1,5-cyclooctadiene are yellow crystalline compounds with a sharp melting point (the crystalline structure of these complexes was proved by X-ray analysis); complex *XV* was an exception, decomposing slowly at a temperature above 350°C and being soluble in common solvents to a limited extent only. By analogy with complex *IX* it can be assumed that also the other complexes are monomeric in solutions (Table II). Complex *XVI* is a brown microcrystalline compound, decomposing at 225°C and partly soluble in common organic solvents. Neither complex *XVII* (brown) nor complex *XVIII* (deep yellow) could be obtained in a sufficiently pure form, which probably is due to the considerable unstability of these complexes. In the case of a reaction between complex *II* and 2-chloro-1,3-butadiene, isolated under the conditions of isolation of complexes *IX*–*XV*, the product exhibits a lower ratio of the diene ligand with respect to rhodium than it would correspond to the assumed structure; on the other hand, after 24 hours of the reaction this ratio is higher, probably as a consequence of dimerization or polymerization of 2-chloro-1,3-butadiene catalyzed with Rh(I) complexes⁶.

The chemical reactivity of the type A complexes is determined by the electron configuration of the central Rh(I) atom, which analogously to bis(ethylene)-2,4-pentanedionerhodium⁵ allows the formation of a transition complex having the structure where rhodium has the coordination number five. It is probable that the increase in the electron density in the rhodium sphere due to the introduction of

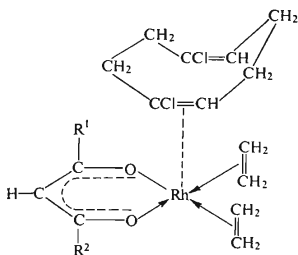


TABLE II
 Properties and Analyses of the Type B Complexes

Complex	M.p., °C (Yield, %)	Solubility	Mol. ions, <i>m/e</i> (<i>I</i> , %)	Mol. weight calc./found ^a	Calc./Found		
					% C	% H	% Cl
<i>IX</i>	133.5–134.5 (66)	Me ₂ CO, CHCl ₃ , C ₆ H ₆	378; 380; 382 (100.0; 66.6; 11.1)	379.1 379; 377 ^b	41.20 41.31	4.52 4.56	18.70 18.72
<i>X</i>	153–154 (46)	Me ₂ CO, CHCl ₃ , C ₆ H ₆	440; 442; 444 (100.0; 65.0; 11.7)	441.2 441	49.10 49.07	4.34 4.37	16.70 16.70
<i>XI</i>	216–218 (94)	CHCl ₃ , C ₆ H ₆	502; 504; 506 (100.0; 65.2; 13.5)	503.2 503	54.84 55.15	4.22 4.23	14.60 14.74
<i>XII</i>	240–242 (66)	Et ₂ O, Me ₂ CO, CHCl ₃ , C ₆ H ₆	530; 532; 534 (40.9; 26.4; 4.5)	531.1 531	56.60 56.59	4.72 4.69	13.38 13.61
<i>XIII</i>	226–228 (76)	Me ₂ CO, CHCl ₃ C ₆ H ₆	562; 564; 566 (83.3; 55.5; 9.2)	563.3 563	53.30 53.28	4.48 4.49	12.59 12.72
<i>XIV</i>	218–220 (89)	Et ₂ O, Me ₂ CO, CHCl ₃ , C ₆ H ₆	570; 572; 574; 576; 578 (70.0; 100.0; 46.7; 11.7; 1.5)	572.1 572	48.2 48.10	3.35 3.37	24.75 24.78
<i>XV</i>	— ^c (94)	Me ₂ CO, CHCl ₃	592; 594; 596 (46.8; 31.3; 5.3)	594.2 593	46.45 45.94	3.39 3.43	11.92 12.06
<i>XVI</i>	225 ^e (52)	—	—	290.6 —	37.11 37.12	4.20 4.19	12.20 11.98
<i>XVII</i>	90 ^e	CHCl ₃	—	290.6 —	37.11 32.07 ^f	4.20 3.81 ^f	12.20 11.09 ^f
<i>XVIII</i>	129 ^e (46)	—	—	325.0 —	33.3 32.85	3.41 3.59	21.55 21.58

^a Determined by mass spectrography. ^b Determined in toluene solution using osmometry in the vapour phase. ^c Not melting up to 350°C. ^d Nitrogen content: calc.: 4.72%, found: 4.76%. ^e Decomposition temperature. ^f Analysis of the product isolated after two hours of reaction. The product after 24 hrs of reaction: 39.60% C, 4.34% H, 20.76% Cl.

aromatic substituents into the chelate ring will have as its consequence a decrease in the probability of formation of the transition complex.

The infrared spectra of the type A and B complexes were recorded in the region from 1400 to 1700 cm^{-1} where the absorption bands appear characterizing the stretching vibrations of the C=O and C=C bonds of the chelate ring⁷⁻¹⁰ and the stretching vibrations of the double bonds coordinated with the central metal atom³. Since the vibrations of the C=O and C=C bonds in the chelate ring undergo a strong mutual interaction, it is not possible to assign unambiguously the bands in this region to the C=O or C=C bonds; this is also the reason why the literature data on the assignment of these bonds differ from each other^{7,8}. In spite of this, the bands in this region can be regarded as characteristic of the coordination of the β -diketone ligand with the central metal atom. The wave numbers of these bands in the spectra of the 2,4-pentanedionate chelates of transition metals are c. 1580 and 1530 cm^{-1} (ref.¹¹). Analogous bands appear also in the spectra of complexes *II*, *IX*, *XIV* and *XVIII* (Table III), whose position is independent of the character of chlorosubstituted diene coordinated with Rh(I) within the limits of experimental accuracy.

Substitution of the methyl group in the diketone ligand with an aromatic substituent (complex *III* and *X*) is reflected in a shift of the band at 1580 cm^{-1} . Both complexes exhibit bands at 1555 cm^{-1} and 1590 cm^{-1} . A symmetrical aromatic substitution in the chelate ring (complexes *IV* and *XI*) decreases the wave number of the band at 1555 cm^{-1} by approximately 20 cm^{-1} . The skeletal vibrations of the aromatic rings can also be seen in this region, particularly at 1590 and 1500 cm^{-1} . The substituents in the aromatic rings do not essentially affect the IR spectra of complexes *V*–*VIII* and *XII*–*XV* compared to complexes *IV* and *IX*. The IR spectra cannot

TABLE III
Infrared Spectra of Complexes of Series A and B in the Region 1450–1700 cm^{-1}

Complex	$\tilde{\nu}$, cm^{-1}			
<i>II</i>	1 530 (s)	1 580 (s)		
<i>IX</i>	1 523 (s)	1 578 (s)		
<i>III</i>	1 491 (m)	1 518 (s)	1 555 (s)	1 591 (m)
<i>X</i>	1 488 (m)	1 517 (s)	1 555 (s)	1 590 (m)
<i>IV</i>	1 485 (s)	1 537 (s)	1 594 (m)	
<i>XI</i>	1 481 (s)	1 538 (s)	1 592 (m)	
<i>V</i>	1 497 (s)	1 536 (s)	1 588 (m)	1 614 (w)
<i>XII</i>	1 494 (s)	1 533 (vs)	1 586 (m)	1 609 (w)
<i>VI</i>	1 496 (s)	1 533 (s)	1 590 (m)	1 605 (s)
<i>XIII</i>	1 494 (vs)	1 534 (vs)	1 589 (vs)	1 604 (vs)
<i>VII</i>	1 481 (vs)	1 535 (vs)	1 587 (s)	
<i>XIV</i>	1 479 (vs)	1 535 (vs)	1 587 (vs)	
<i>VIII</i>	1 486 (s)	1 530 (vs)	1 554 (vs)	1 605 (w)
<i>XV</i>	1 485 (m)	1 525 (s)	1 554 (s)	1 606 (w)
<i>XVI</i> ^a	1 522	1 580	1 630	1 687
<i>XVIII</i> ^a	1 522	1 580	1 610	

^a Measured using the KBr-technique.

be used as a basis for the study of the coordination of the ethylene ligands in the type A complexes. An effort to determine the Raman spectra of these complexes failed, owing to the intense colouration of the sample.

In the given wave number region the series of the type B complexes ought to exhibit an absorption maximum corresponding to the coordination of the multiple bonds of 1,6-dichloro-1,5-cyclooctadiene with Rh(I). In the spectrum of an uncoordinated 1,6-dichloro-1,5-cyclooctadiene⁶ the double bond has a corresponding maximum at 1665 cm^{-1} . The spectra of complexes IX–XV have no maxima in this region. As follows from Table III, in the case of complex IX the absorption band of the coordinated double bond is shifted into the region of wave numbers lower than 1580 cm^{-1} , where the weak absorption bands of dichlorocyclooctadiene ring are obviously overlapped by the absorptions of the chelate ring. The shift of the wave number $\Delta\tilde{\nu}$ is therefore at least 80 cm^{-1} . It can also be deduced from the spectra that a change in the character of the ligand during the transition from complexes of the A series to those of the B series is virtually not reflected in the vibrations of the chelate ring, which suggests that the character of the chelate ring remains the same in both cases. The character of the coordination of chlorosubstituted conjugated dienes in complexes XVI and XVIII was estimated using the published spectra of these dienes^{12,13}. Their comparison with the spectra of corresponding complexes reveals the absence of a band at 1640 cm^{-1} in the case of coordination of 1-chloro-1,3-butadiene with Rh(I). Owing to the rather complex character of the infrared spectrum of complex XVI in comparison with complex IX, it is not possible to make an estimate of the shift of the wave number of the multiple bond due to coordination with Rh(I).

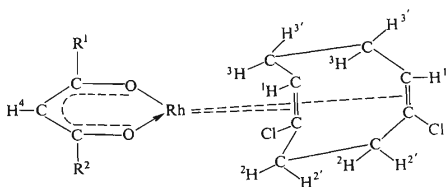
A survey of the τ values of the NMR spectra of complexes IX–XV is given in Table IV. The formation of a complex is in the first place reflected in a change in the chemical shift of H^1 toward higher τ values with respect to the spectrum of an uncoordinated 1,6-dichloro-1,5-cyclooctadiene (H^1 4.22(8), $\text{H}^{2,2'}$ 7.67(4), $\text{H}^{3,3'}$ 7.24(4) p.p.m.; cf. Table IV). This change is also accompanied by a change in the shape of the H^1 band, which is in accordance with the data of Bennett and Saxby¹⁴. It also follows from the data in Table IV that the substituents in the chelate ring affect the chemical shift, both that of the proton of H^1 of the diene ligand and that of the proton of H^4 of the β -diketone ring. Introduction of the aromatic rings is reflected in a shift of the H^4 bands in the spectra of the complexes toward lower τ values. In the case of complex X with one substituted CH_3 -group this shift is 0.75 p.p.m. with respect to τH^4 in the spectrum of complex IX; the same shift in the case of complex XI with two aromatic rings is 1.43 p.p.m. Analogous shifts of the H^4 bands were observed in the spectra of the β -diketone carbonyl complexes¹⁵. The shift can be explained in terms of delocalization of the electrons of the double bonds of the chelate ring, owing to conjugation with the aromatic substituents.

The shifts of the H^1 bands can be interpreted on the basis of changes of the shielding of protons bonded to the double bonds, owing to coordination of these bonds with the metal atom¹⁶. It can be assumed that the magnetic dipole representing the effect of the electrons of the double bond will be shifted in the direction of the double bond — metal connecting line. It is probable that owing to an increase in the π -bond component of the coordination bond between diene and rhodium this shift is smaller; taking into account that H^1 lies in a plane perpendicular to this dipole, it can be expected that an approach of the magnetic dipole will lead to a shift of the H^1 band to the region of lower τ values. Table IV shows that the H^1 bands are shifted by 0.1 p.p.m.

(complex *X*), owing to the asymmetrical aromatic substitution of the β-diketone ring; the symmetrically substituted complex *XI* exhibits a shift of 0.17 p.p.m. compared to *IX*. Introduction of the phenyl group into the chelate ring leads to a strengthening of the bond of the diketone ligand with the central metal atom¹⁷. The stronger ligand field of substituted β-diketone ligands probably contributes to the strengthening of the π-bond component of the coordination bond between 1,6-dichloro-1,5-cyclooctadiene and Rh(I).

TABLE IV

Chemical Shifts of Protons in Complexes of the Type



Complex	R ¹	R ²	τ, p.p.m. ^a		
			H ¹	H ^{2,2'} , H ^{3,3'}	H ⁴
<i>IX</i>	CH ₃	CH ₃	5.52 (2)	8.36–7.00 (8)	4.70 (1)
<i>X</i>	CH ₃	C ₆ H ₅	5.42 (2)	8.33–6.85 (8)	3.95 (1)
<i>XI</i>	C ₆ H ₅	C ₆ H ₅	5.35 (2)	8.33–6.82 (8)	3.27 (1)
<i>XII</i>	CH ₃ C ₆ H ₄	CH ₃ C ₆ H ₄	5.34 (2)	8.38–6.86 (8)	3.29 (1)
<i>XIII</i>	CH ₃ OC ₆ H ₄	CH ₃ OC ₆ H ₄	5.36 (2)	8.31–6.88 (8)	3.35 (1)
<i>XIV</i>	ClC ₆ H ₄	ClC ₆ H ₄	5.39 (2)	8.31–6.85 (8)	3.39 (1)
<i>XV</i>	NO ₂ C ₆ H ₄	NO ₂ C ₆ H ₄	5.29 (2)	8.42–6.76 (8)	3.27 (1)

^a Numbers in brackets designate the relative band intensities.

EXPERIMENTAL

The IR spectra were recorded in chloroform solution with a Zeiss UR-10 spectrometer. The NMR spectra (0.5M solutions in CDCl₃, HMDS as external standard) were recorded with a JEOL-PS-100 apparatus. Molecular weights were determined using a Vapour-Pressure-Osmometer Hitachi Perkin-Elmer 115. An AEI Mass Spectrometer MS 902 was used for recording the mass spectra.

Materials

The original dichlorotetraethylenedirrhodium(*I*) complex was synthesized after Cramer¹⁸, b.p. 116°C and transformed into bis(ethylene)-2,4-pentanedionerhodium(*II*), b.p. 144–146°C, via reaction with acetylacetone⁵. Benzoylacetone, dibenzoylmethane, bis(*p*-toluyl)methane, bis(*p*-

methoxybenzoyl)methane, bis(*p*-chlorobenzoyl)methane and bis(*p*-nitrobenzoyl)methane were prepared after Sieglitz and Horn¹⁹. 1,6-Dichloro-1,5-cyclooctadiene, b.p. 67.5°C/0.5 Torr, n_D^{25} 1.5335, prepared by thermal dimerization²⁰ of 2-chloro-1,3-butadiene (J. Dimitrov's Chemical Work, Šala), b.p. 59.4°C/760 Torr, n_D^{25} 1.4853, 1-chloro-1,3-butadiene, b.p. 65.5°C/760 Torr, n_D^{25} 1.4712, was synthesized after ref.²¹. From commercial 2,3-dichloro-1,3-butadiene (Kaučuk, Kralupy), the fraction 70°C/300 Torr, n_D^{20} 1.4898 was employed.

Preparation of Complexes

Bis(ethylene)-1-phenyl-1,3-butanedionerhodium(III). To a mixture of 1.8 g (4.5 mmol) of complex I, 25 ml ether and 1.5 g (9 mmol) benzoylacetone, cooled to -80°C, a solution of 1.5 g KOH in 5 ml water was added dropwise. The mixture was heated to 0°C while stirring, another 25 ml of ether was added and the stirring was continued for another half an hour at 0°C. The ether layer was then separated, filtered, concentrated and cooled to -80°C. Orange-coloured small crystals separated out, yield 1.98 g (57%), m.p. 90.5–91.5°C (ether). The same method was used to prepare the ethylene complexes IV–VIII.

(1,6-Dichloro-1,5-cyclooctadiene)-2,4-pentanedionerhodium (IX). To a suspension of 0.26 g (1 mmol) of complex II in 2 ml of toluene, 0.3 ml (1.7 mmol) of 1,6-dichloro-1,5-cyclooctadiene was added. After the evolution of ethylene had stopped, the solution was filtered, concentrated, and the yellow crystals washed with light petroleum; the yield was 0.35 g (94%), m.p. 133.5–134.5°C (benzene). Complexes X–XV were obtained in the same way as complex IX.

(1-Chloro-1,3-butadiene)-2,4-pentanedionerhodium (XVI). To a suspension of 0.26 g (1 mmol) of complex II in 2 ml of toluene, 1.0 ml (10.8 mmol) of 1-chloro-1,3-butadiene was added. After the evolution of ethylene had stopped, the solution was poured while stirring into 50 ml of light petroleum. The brown precipitate, insoluble in common solvents, decomposes at 225°C without melting. The yield is 0.17 g (60%). The procedure employed to prepare complexes XVII and XVIII is analogous to that of the synthesis of complex XVI.

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