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Structural features of the mixed $\eta^2:\eta^4$ coordination of two transition metal atoms in binuclear and trinuclear π-complexes with semiquinoid ligands. Crystal and molecular structure of (η^2-ethylene)(4-methyl-4-*exo*-n-butyl-1-*anti*-η^2-methylene-[(2,5-η^4-cyclohexadiene)rhodium-2,4-*O*,*O*′-pentanedionate])-rhodium-2,4-*O*,*O*′-pentanedionate *

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Abstract

The crystal and molecular structure of (η^2-ethylene)(4-methyl-4-*exo*-n-butyl-1-*anti*-η^2-methylene-[(2,5-η^4-cyclohexadiene)rhodium-2,4-*O*,*O*′-pentanedionate]-rhodium-2,4-*O*,*O*-pentanedionate has been determined by an X-ray diffraction study. Comparison of the data obtained with those of a previous study of a mononuclear η^4-coordinated rhodium(I) complex with the 4-methyl-4-trichloromethyl-2,5-cyclohexadiene-1-one ligand reveals that the most prominent feature of the mixed η^2 : η^4 coordination in this series consists in the greater deviation (up to 30 and 40°, respectively) of the exo-unsaturated and the saturated geminal fragments of the semiquinoid ligand from the central plane of its cyclohexadiene ring. The shielding of the H^A proton of the n-butyl group (-$CH^AH^BC_3H_7$) by the η^2-coordinated rhodium atom (as well as by other fragments of the molecule) is different from that of the H^B proton of the same group, which accounts for the unusual features in the ^1H NMR spectra of this complex. There is a pronounced transannular contact between the rhodium atom and the hydrogen atom of the peripheral C–H^A bond, practically at the face of the fragment being coordinated (the Rh...H^A and Rh...CH^A distances are 2.57 and 3.50 Å, respectively, and the

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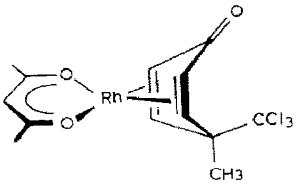
* Dedicated to Professor Ernst Otto Fischer on the occasion of his 70th birthday on November 10th, 1988.

Rh...H^A–CH^B angle is 160°). Use of molecular modelling based on the results of the X-ray diffraction study shows that for the recently synthesized trinuclear rhodium(I) complexes with two *exo*-methylene-2,5-cyclohexadiene ligands, the *anti-trans* configuration is the most likely.

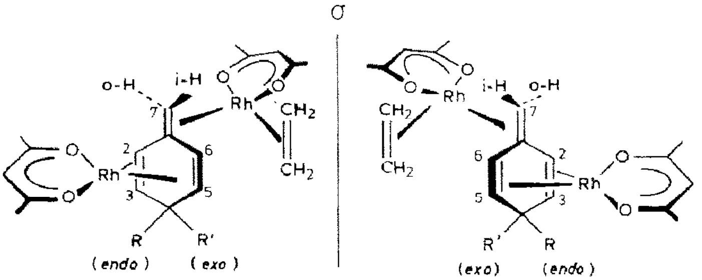
Introduction

The main structural features of the η^4-type of π-coordination of 4,4-disubstituted 2,5-cyclohexadienones, by transition metals (as exemplified by 4-methyl-4-trichloromethyl derivative I of acetylacetonatorhodium) have been studied previously [1]. These are manifested in the coordinational fixation of a non-symmetric boat conformation for the organometallic molecule and results, firstly in a non-equivalent orientation of the geminal substituents (*exo-* and *endo*-positions) relative to the metal atom, and secondly, if a trichloromethyl group is present, in the formation of a stereospecific π-complex:

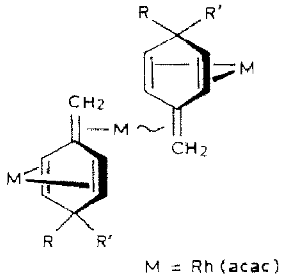


(η^4-I)

In the course of the subsequent investigations of organoelement transformations in semiquinoid systems (cyclohexadienones, alkylidenecyclohexadienes, their analogues and derivatives) a series of new bi- (II) and trinuclear (III) acetylacetonatorhodium(I) complexes with 4,4-disubstituted methylidene cyclohexadienes was recently obtained [2]. These complexes are characterized by a mixed $\eta^2:\eta^4$ transition metal coordination with the cross-π, π-conjugated triene ligand.



($\eta^2:\eta^4$-II)
(*anti* isomers)



($\eta^2:\eta^4$-III, R = R' = CH_3)
(*trans-anti* isomers)

R,R' = CH_3 (a); R = CH_3, R' = $CHCl_2$ (b, *exo*); R = $CHCl_2$, R' = CH_3 (b, *endo*); R = CH_3, R' = n-C_4H_9 (c, *exo*); R = n-C_4H_9, R' = CH_3 (c, *endo*).

Compounds with such a structure are undoubtedly of interest as new models in organometallic stereochemistry [3]. The ^1H NMR data and the molecular modelling indicate that the two transition metal atoms are most probably in the *anti*-position with respect to the semiquinoid ligand central plane in the case of complexes II and III *, and the two coordinated semiquinoid fragments (in complex III) are most probably in a *trans*-position relative to the central plane of the chelated metal atom. Furthermore, for the chiral racemic IIc *exo*-complex which was studied by double homonuclear resonance on the ^1H nuclei the unusual effect of a considerable difference in the degree of shielding of the H^A and H^B protons adjacent to the ring methylene group CH^AH^B of the n-butyl substituent ($\Delta\delta$ 0.66 ppm, J_{AB} 12.5 Hz) was observed. This effect is probably due to the *exo*-orientation of the n-butyl group, which brings the methylene group closer to the η^2-coordinated metal atom of the Rh(acac)(C_2H_4) group [2].

In order to obtain direct proof of the above stereochemical assumptions, and to verify the main structural features of the $\eta^2 : \eta^4$-coordination (and to compare them with those of the η^4-coordination [1]) in a series of semiquinoid π-complexes an X-ray diffraction study of binuclear complex IIc was carried out and is described herein.

Experimental

The single crystal used for X-ray measurements was obtained by slow crystallization of compound IIc (*exo*-isomer XVIIIa, m.p. 168 °C (decomp.), of ref. 2) from hexane during 7 days. Crystals of IIc are triclinic; at $-120\,^\circ$C a 8.375(3), b 11.488(4), c 13.790(6) Å, α 112.81(3), β 92.72(3), γ 97.21(3)°, V 1206.6(8) Å^3, $d_{calc.}$ 1.636 g/cm^3, $Z = 2$, space group $P\bar{1}$. The unit cell parameters and the intensities of 3680 independent reflections with $F^2 \geqslant 3\sigma$ were measured with a 4-circle automatic "Syntex P2$_1$" diffractometer (-120°, Mo-K_α radiation, graphite monochromator, $\theta/2\theta$ scan, $\theta \leqslant 28°$).

The structure was solved by direct methods. Both Rh atoms were located in the E-map; all other non-hydrogen atoms were found by successive approximations of the electron density syntheses. The structure was refined, first isotropically and then anisotropically, by least squares. In the final cycles of refinement the contribution by H atoms to F_{calc} (all of them were located in the difference Fourier synthesis) was taken into account with a fixed B_{iso} value of 3 Å^2, but their positional and thermal parameters were not refined. All calculations were carried out with an "Eclipse S/200" computer using the INEXTL program [4]. The final R-factor was 0.045, the weighted R_w-factor 0.055. Atomic coordinates and their temperature factors are presented in Table 1, bond lengths and angles in Tables 2 and 3, respectively.

Results and discussion

The X-ray diffraction study has confirmed that compound IIc is a binuclear acetylacetonatorhodium complex with 1-methylidenecyclohexa-2,5-diene ligand coordinated by two rhodium atoms (Fig. 1). One of them, (Rh(1)), is coordinated by

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* In the formulae of the type II compounds the numeration of C and H atoms specifies the position of the exo methylene hydrogen atoms directed towards the ethylene fragment (inner-H) and contrary-wise (outer-H) (see NMR spectra parameters in Table 1 of ref. 2.

Table 1

Atomic coordinates ($\times 10^4$, for Rh $\times 10^5$) and their equivalent isotropic temperature factors B (Å^2)

Atom	*x*	*y*	*z*	*B*
Rh(1)	−15761(5)	44172(4)	16079(4)	1.76(1)
Rh(2)	−129(5)	11234(4)	24608(4)	1.72(1)
O(1)	−2790(5)	4317(4)	262(4)	2.1(1)
O(2)	−240(5)	6122(4)	1833(4)	2.2(1)
O(3)	−506(5)	134(4)	3404(4)	2.0(1)
O(4)	−1940(5)	41(4)	1389(4)	2.1(1)
C(1)	−2511(7)	5140(6)	−153(5)	2.1(2)
C(2)	−317(7)	6658(6)	1184(5)	2.0(2)
C(3)	−1350(8)	6255(6)	261(6)	2.3(2)
C(4)	−3499(8)	4850(7)	−1186(6)	2.6(2)
C(5)	919(8)	7858(6)	1458(6)	2.7(2)
C(6)	−399(7)	2610(5)	1887(5)	1.8(2)
C(7)	−48(7)	3962(5)	2638(5)	1.7(2)
C(8)	−1238(6)	4580(5)	3181(5)	1.8(2)
C(9)	−2913(6)	3823(5)	3139(5)	1.8(2)
C(10)	−3227(7)	3102(6)	1958(5)	2.0(2)
C(11)	−2068(7)	2448(5)	1378(5)	1.8(2)
C(12)	−4135(7)	4752(6)	3534(6)	2.5(2)
C(13)	−2938(7)	2931(6)	3743(5)	2.0(2)
C(14)	−2347(7)	3580(6)	4909(5)	2.3(2)
C(15)	−2166(8)	2661(6)	5452(6)	2.5(2)
C(16)	−3756(9)	1899(8)	5513(8)	4.0(3)
C(17)	766(7)	1873(6)	1366(5)	2.1(2)
C(18)	1632(8)	2422(6)	3770(6)	2.7(2)
C(19)	2431(7)	1444(7)	3106(6)	2.7(2)
C(20)	−1748(7)	−734(6)	3231(5)	2.1(2)
C(21)	−3018(7)	−761(6)	1546(6)	2.3(2)
C(22)	−2963(7)	−1143(6)	2382(6)	2.5(2)
C(23)	−1792(8)	−1372(7)	3992(6)	3.0(2)
C(24)	−4415(9)	−1312(8)	695(7)	3.7(3)

Table 2

Bond lengths *d* (Å)

Bond	*d*	Bond	*d*
Rh(1)–O(1)	2.029(5)	C(2)–C(5)	1.521(9)
Rh(1)–O(2)	2.032(5)	C(6)–C(7)	1.477(9)
Rh(1)–C(7)	2.122(6)	C(6)–C(11)	1.490(8)
Rh(1)–C(8)	2.106(7)	C(6)–C(17)	1.410(9)
Rh(1)–C(10)	2.129(7)	C(7)–C(8)	1.376(8)
Rh(1)–C(11)	2.142(7)	C(8)–C(9)	1.540(8)
Rh(2)–O(3)	2.058(5)	C(9)–C(10)	1.505(9)
Rh(2)–O(4)	2.045(4)	C(9)–C(12)	1.536(9)
Rh(2)–C(6)	2.193(7)	C(9)–C(13)	1.550(9)
Rh(2)–C(17)	2.099(7)	C(10)–C(11)	1.392(9)
Rh(2)–C(18)	2.131(8)	C(13)–C(14)	1.513(9)
Rh(2)–C(19)	2.121(6)	C(14)–C(15)	1.530(9)
O(1)–C(1)	1.286(9)	C(15)–C(16)	1.523(9)
O(2)–C(2)	1.272(8)	C(18)–C(19)	1.413(9)
O(3)–C(20)	1.290(8)	C(20)–C(22)	1.403(9)
O(4)–C(21)	1.295(8)	C(20)–C(23)	1.493(9)
C(1)–C(3)	1.407(9)	C(21)–C(22)	1.384(9)
C(1)–C(4)	1.509(9)	C(21)–C(24)	1.501(9)
C(2)–C(3)	1.386(9)		

Table 3

Bond angles ω (deg.)

Angle	ω	Angle	ω
O(1)Rh(1)O(2)	91.9(2)	C(7)C(6)C(11)	105.1(5)
O(1)Rh(1)A(7,8) [a]	170.7	C(7)C(6)C(17)	125.1(6)
O(1)Rh(1)A(10,11) [a]	99.9	C(11)C(6)C(17)	122.3(6)
O(2)Rh(1)A(7,8) [a]	97.3	C(6)C(7)C(8)	121.3(6)
O(2)Rh(1)A(10,11) [a]	168.1	C(7)C(8)C(9)	120.3(6)
A(7,8)Rh(1)A(10,11) [a]	70.8	C(8)C(9)C(10)	98.2(5)
O(3)Rh(2)O(4)	91.2(2)	C(8)C(9)C(12)	109.6(5)
O(3)Rh(2)A(6,17) [a]	171.1	C(8)C(9)C(13)	114.8(5)
O(3)Rh(2)A(18,19) [a]	85.5	C(10)C(9)C(12)	110.2(5)
O(4)Rh(2)A(6,17) [a]	86.8	C(10)C(9)C(13)	112.9(5)
O(4)Rh(2)A(18,19) [a]	171.0	C(12)C(9)C(13)	110.5(5)
A(6,17)Rh(2)A(18,19)	97.7	C(9)C(10)C(11)	121.0(6)
Rh(1)O(1)C(1)	124.7(4)	C(6)C(11)C(10)	120.3(6)
Rh(1)O(2)C(2)	123.8(4)	C(9)C(13)C(14)	115.5(5)
Rh(2)O(3)C(20)	124.7(4)	C(13)C(14)C(15)	114.4(6)
Rh(2)O(4)C(21)	124.1(4)	C(14)C(15)C(16)	114.3(6)
O(1)C(1)C(3)	125.6(6)	O(3)C(20)C(22)	125.5(6)
O(1)C(1)C(4)	116.2(6)	O(3)C(20)C(23)	114.8(6)
C(3)C(1)C(4)	118.1(6)	C(22)C(20)C(23)	119.6(6)
O(2)C(2)C(3)	127.6(6)	O(4)C(21)C(22)	126.5(6)
O(2)C(2)C(5)	114.7(6)	O(4)C(21)C(24)	113.4(6)
C(3)C(2)C(5)	117.7(6)	C(22)C(21)C(24)	120.1(6)
C(1)C(3)C(2)	126.1(6)	C(20)C(22)C(21)	127.3(6)

[a] A(6,17), A(7,8), A(10,11), A(18,19) are the midpoints of the C(6)=C(17), C(7)=C(8), C(10)=C(11), C(18)=C(19) bonds, respectively.

the double bonds of the cyclohexadiene ring, and the second, (Rh(2)), is coordinated by the exocyclic double bond and the ethylene molecule. The metal atoms in complex IIc are on opposite sides of the methylidenecyclohexadiene ligand, and the n-butyl substituent is on the same side as the Rh(2) atom. Though molecule IIc is chiral [2], the crystal is centrosymmetric and hence represents a racemate.

The Rh(1) atom in molecule IIc has essentially the same coordination environment as that in complex I studied by us previously [1]. Thus, the geometric

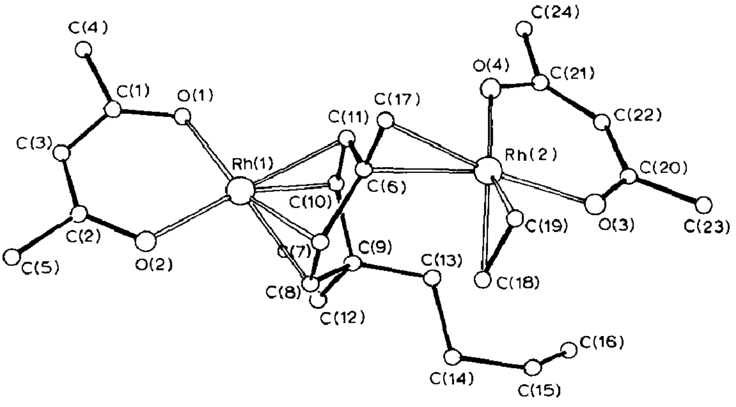
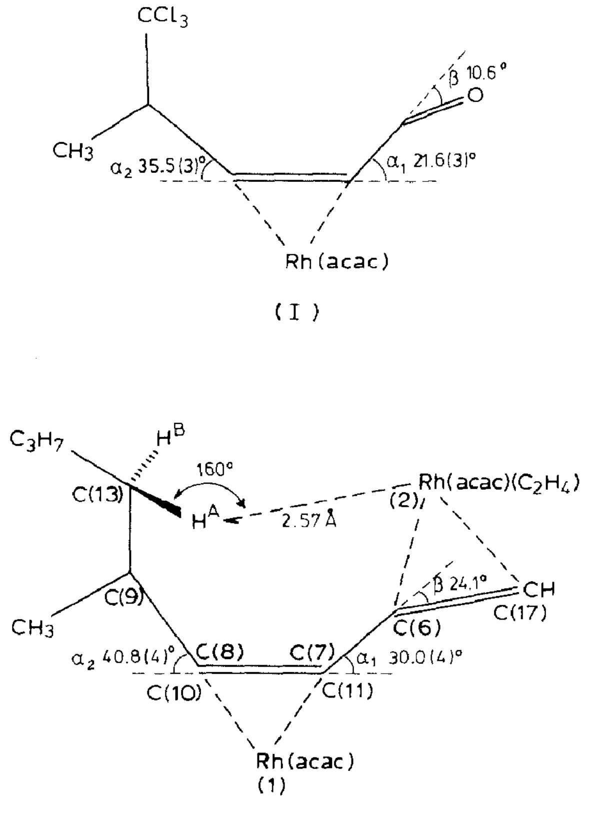


Fig. 1. Structure of molecule IIc.



(I)

(IIc)

Fig. 2. Comparison of molecular conformations of I and IIc.

parameters characterizing Rh(1) atom coordination in IIc are very close to those found in I. As in the case of complex I, the plane of the chelate acetylacetonate ring in IIc is approximately perpendicular (dihedral angle 91.7(3)° to the mean plane C(7)C(8)C(10)C(11)(A) of the double bonds (the maximum atomic deviation from this plane being 0.002 Å). The Rh(1) atom is displaced from its coordination plane (defined by the O(1), O(2) atoms and the midpoints of the C(7)=C(8) and C(10)=C(11) double bonds) only by 0.015 Å.

The 6-membered cycle of methylidenecyclohexadiene ligand in IIc has a non-symmetric boat conformation with the n-butyl substituent in the axial position; the vinylidene C(6) atoms is displaced from the plane A by a somewhat smaller distance than the sp^3-hybridized C(9) atom. Nevertheless, the C(6) atom displacement from the plane A in IIc, equal to 0.451(6) Å (the folding angle of the ring along the C(7)...C(11) line α_1 30.0(4)°; see Fig. 2), is substantially greater than that of the carbonyl carbon atoms in complex I (0.315 Å) and in the series of *p*-benzoquinone rhodium derivatives [5,6]. This difference seems to be a consequence of the C(6)=C(17) double bond participation in the π-coordination with the Rh(2) atom. However, in order to confirm this assumption one should compare the geometric parameters of molecule IIc with the geometry of mononuclear methylidenecyclohexadiene rhodium complexes, which as far as we know, have not yet been synthesized.

The sp^2-hybridized key carbon atom C(6) in IIc, in *p*-benzoquinonerhodium complexes, and in molecule I, has an essentially non-planar environment: the C(17) atom is displaced from the C(6)C(7)C(11) plane by 0.577(7) Å, so that the C(6)=C(17) bond turns out to be almost parallel to the C(7)=C(8) and C(10)=C(11) double bonds (the angles between the vectors of the C(6)=C(17)/C(7)=C(8) and C(6)=C(17)/C(10)=C(11) bonds are 6.1 and 6.0°, respectively). It should be emphasized that, whereas in molecule I the analogous geometry of the exocyclic carbonyl group does not exclude the possibility of its conjugation with endocyclic double bonds (see discussion in ref. 1) analogous conjugation in IIc appears to be extremely unlikely, since the π-systems of endo- and exo-cyclic double bonds in the methylidenecyclohexadienone ligand take part in the coordination with two different Rh atoms. Distortion from planarity of the C(6) atom environment in IIc and a considerable increase in the β angle, as compared with that found in I [1] (see Fig. 2), can clearly be explained in terms of a partial rehybridization ($sp^2 \rightarrow sp^3$) of the carbon atoms, which is quite usual for π-coordinated olefin ligands.

The saturated C(9) carbon atom, which is on the opposite side of the ring, is displaced from plane A to a greater degree (0.650(6) Å) than C(6). The folding angle of the ring along the line C(8)...C(10) (α_2 40.7(4)°) is close to the corresponding dihedral angles (42.3 and 44.7°) found in a cyclohexadienerhodium complex (cyclopentadienyl)(3-carbmethoxycyclohexa-1,4-diene)rhodium [7]. A somewhat smaller folding (α_2 36.5°) of the cyclohexadiene ring in I, noted previously [1] is probably because of steric repulsion between one of the chlorine atoms of the CCl_3 group and the carbonyl atom C(6). The replacement of the bulky trichloromethyl substituent in I by the n-Bu group in IIc allows these steric hindrances to be removed by a favourable rotation around the C(9)–C(13) single bond. It is also possible that this conformational difference is brought about by the transannular (attractive) interaction of the Rh(2) atom with one of the H atoms of the methylene $C(13)H_2$ group. The conformation about the C(9)–C(13) bond in molecule IIc is close to staggered; the C(10)C(9)C(13)C(14) torsion angle is 169.8(7)°. The same conformation has also been found for the other bonds in the n-butyl substituent, which, however, does not have the planar zigzag-like structure characteristic of n-alkyl groups; the C(9)C(13)C(14)C(15) and C(13)C(14)C(15)C(16) torsion angles are −172.1(7) and −66.3(7)°, respectively.

Whereas the Rh(1) atom in IIc has an almost exactly planar coordination as noted above, the environment of the Rh(2) atom is characterized by noticeable deviations from planarity: the plane passing through the midpoints of C(6)=C(17), C(18)=C(19) double bonds and the Rh(2) atom forms a dihedral angle of 11.3° with the Rh(2)O(3)O(4) plane, the angle between the vectors of the double bonds C(6)=C(17) and C(18)=C(19) is 15.2°. The lengths of the Rh–C bonds involving the Rh(1) and Rh(2) atoms (2.10–2.19 Å), as well as the lengths of the coordinated C=C bonds (1.38–1.41 Å) are consistent with the values found both in our and in other rhodium π-olefin complexes having cyclic and acyclic ligands [7,8–10]. The different nature of the substituents at carbon atoms C(6) and C(17) results in a markedly asymmetric π-coordination of this bond (Rh(2)–C(6) 2.099(7), Rh(2)–C(17) 2.193(7) Å).

Chelate rings of both the acac ligands in IIc are essentially planar: the Rh(1) and Rh(2) atoms are displaced from the planes O(1)O(2)C(1)C(2)C(3) and O(3)O(4)C(20)C(21)C(22) by 0.071(1) and 0.149(1) Å, respectively. The Me groups in IIc and in molecule I are not quite in the planes of the corresponding metallocycles: the maximum deviation (0.214(8) Å) was found for the C(23) atom.

We have pointed out previously [1] that the Rh–O(acac) bond lengths in acetylacetonatorhodium complexes are strongly dependent on the electronic effects of other ligands in the complex. In particular, in duroquinone and cyclohexadiene complexes, where the ligand has rather strong electron-accepting properties, the Rh–O(acac) bonds were found to be very short; 1.97–1.98 Å [5] and 2.014 Å [1]. The ligands in the *trans*-position with respect to the acac-groups in complex IIc, methylidenecyclohexadiene (for Rh(1)) and the olefin bonds (for Rh(2)), are characterized by a lower electron-accepting ability, as compared with similar carbonyl-containing systems, which results in the somewhat longer Rh–O(acac) distances in IIc (2.029–2.058 Å). However, at a given level of accuracy such fine differences between the bond lengths in individual structures can only be regarded as a basis for further, more detailed investigations. Sufficiently well-substantiated conclusions

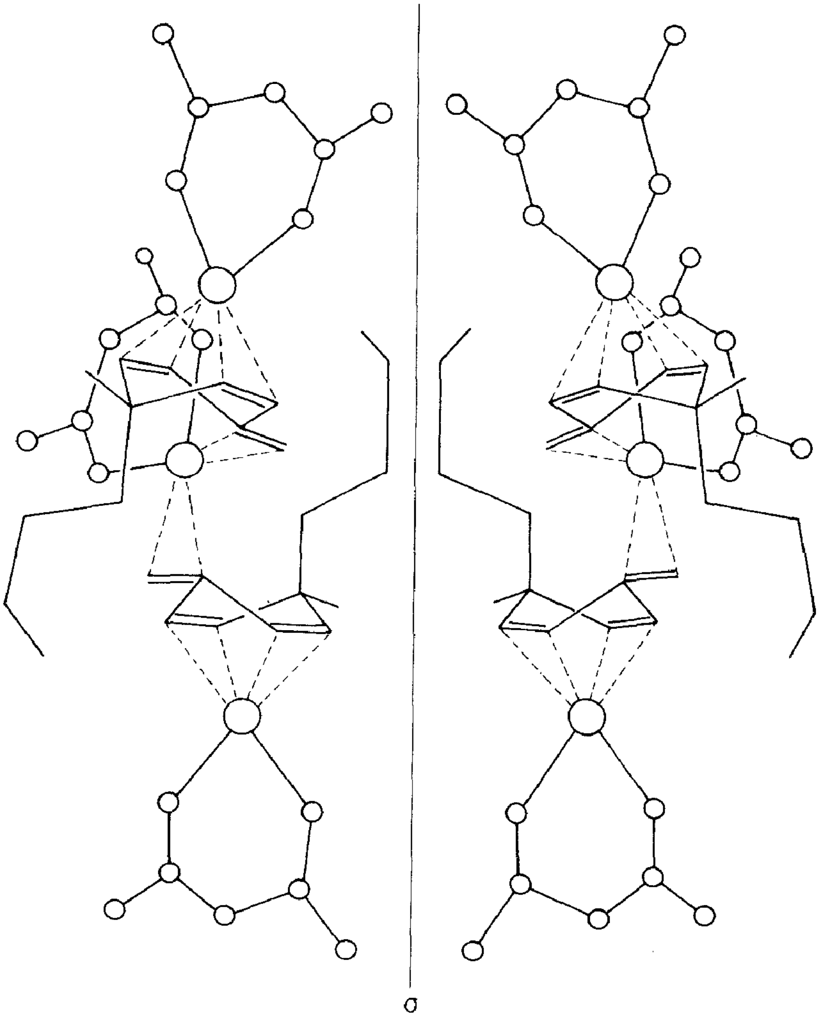


Fig. 3. *Anti-trans* structure proposed for complex IIc.

regarding the correlation between the Rh–O(acac) bond lengths and the nature of the *trans*-ligand should be based on a statistical analysis of the profusion of data in the literature.

A significant difference in the shielding of the two methylene protons (C(13)H^AH^B), which was evident in the ^1H NMR spectrum of IIc [2], should reflect the different dispositions of these protons with respect to the Rh(2) atom. Indeed, it was found that one of the methylene group hydrogen atoms (H^B) is as much as 3.93 Å away from the metal atom and, obviously, cannot be influenced by it, whereas the other hydrogen atom (H^A) is much closer to the Rh(2) atom, the Rh...H^A distance being 2.57 Å. This difference is enough for the two protons in question to have a markedly different magnetic environment (to which, apart from the Rh(2) atom, other adjacent magnetically active groups in the molecule can also contribute) which thus is responsible for the considerable difference in their shielding in ^1H NMR spectra.

We have compared, both here and previously [1] a broad range of organometallic complexes with cyclohexadiene ligands containing an exo-unsaturated unit (with η^4- and $\eta^2:\eta^4$-types of rhodium(I) atom coordination). The comparisons showed their structural parameters to be similar, confirming that at a quantitative structural level, semiquinoid organometallics occupy an intermediate position between the well-studied quinoid and the dihydrobenzoid systems [1].

This approach, within broad limits, enables the use of previously available X-ray diffraction data to predict the main features of the structures of other classes of compounds belonging to the type in question. Thus the structural parameters of complex IIc were used here to estimate the most likely stereochemistry of the tri-nuclear semiquinoid rhodium complex III. Taking into account its easy formation via symmetrization of the corresponding binuclear complex IIa and the large similarity in the NMR spectral parameters of the compounds of type II and III [2] one could enivsage a high degree of similarity between their molecular structures. Our theoretical reconstruction of molecule III on the basis of two real $\eta^2:\eta^4$-coordinated semiquinoid structural fragments of compound IIc in the assumption of a usual square-planar geometry for the central rhodium atom shows that of the two possible configurations (*cis* or *trans*) of these bulky fragments relative to one another the most probable is the *anti-trans*-structure shown in Fig. 3. Such a configuration would provide the least possible steric repulsion between the alkyl substituents in the geminal units of the molecule.

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