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BENZOTRIAZOLE AND BENZOTRIAZOLATE COMPLEXES OF RHODIUM(I)

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Summary

The preparation and properties of mono- and di-nuclear rhodium(I) complexes of general formulae $RhClL_2(btzH)$, $[Rh(\mu\text{-}btz)L_2]_2$, $Rh_2(\mu\text{-}btz)(\mu\text{-}N_3)(L_2)_2$ and $Rh_2(\mu\text{-}btz)(\mu\text{-}SCN)(COD)_2$ [L_2 = diolefin, $(CO)_2$ or $(CO)(PPh_3)$], and of the related tetranuclear derivatives are reported. The latter compounds have benzotriazolate ligands simultaneously bonded to three rhodium atoms.

Introduction

Structure and bonding in rhodium(I) azolate complexes have attracted much interest in recent years [1–6]. In our earlier papers a series of complexes of formulae $[Rh(\mu\text{-}az)L_2]_x$ were described, where L_2 = diolefin, $(CO)_2$, $(CO)(PR_3)$ or $(CS)(PR_3)$, and az is an exobidentate azolate ligand [7–9]. In particular, the versatile pyrazolate type ligands are able to hold two rhodium(I) centers in a chemically stable configuration with a wide range of intermetallic separations (i.e. Rh–Rh distance of 3.220 Å in $[Rh(\mu\text{-}Me_2Pz)(CS)(PPh_3)]_2$ against 3.568 Å in $[Rh(\mu\text{-}Pz)(CO)\{P(OPh)_3\}]_2$ [8]). Higher nuclearity is found for related imidazolate bridged rhodium(I) complexes, as confirmed by crystallographic characterization of $[Rh(\mu\text{-}MeIm)(CO)_2]_4$ [9]. Following our interest in rhodium(I) azolate chemistry we decided to investigate the coordination ability of benzotriazolate type ligands *, where three nitrogen atoms are potentially available as donor sites.

Results and discussion

The addition of benzotriazole type ligands to $[Rh(\mu\text{-}Cl)(COD)]_2$ [10] suspended in acetone results in the formation of RhCl(COD)(btzH) complexes (Ia–Ic). These

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* A generic benzotriazole is indicated by btzH, unsubstituted benzotriazole by BtzH, and the substituted benzotriazole ligands as $5\text{-}NO_2BtzH$ and $5,6\text{-}Me_2BtzH$.

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compounds, which are stable yellow solids, react with carbon monoxide with formation of the orange RhCl$(CO)_2$(btzH) compound. In these complexes the rhodium atoms are probably in a square planar arrangement, where the btzH is coordinated through a pyridinic nitrogen atom.

The addition of triethylamine to a methanol solution of RhCl(COD)(btzH) leads to elimination of the acidic proton of the benzotriazole ligand with formation of $[Rh(\mu\text{-btz})(COD)]_2$. A direct route to prepare yellow $[Rh(\mu\text{-btz})(\text{diolefin})]_2$ complexes, in high yield involves the reaction of $[Rh(\mu\text{-Cl})(\text{diolefin})]_2$ (diolefin = 1,5-

TABLE 1

ANALYTICAL DATA FOR THE COMPOUNDS

Complex		Found(calcd.)(%)			Mol.wt.($CHCl_3$) (found(calcd.))	Yield (%)
		C	H	N		
Ia	RhCl(COD)(BtzH)	46.1	4.5	11.3	474	88
		(45.9)	(4.6)	(11.4)	(365)	
Ib	RhCl(COD)(5-NO_2BtzH)	40.9	3.8	13.4	475	72
		(40.9)	(3.9)	(13.6)	(410)	
Ic	RhCl(COD)(5,6-Me_2BtzH)	47.6	5.2	10.1	410	63
		(48.7)	(5.3)	(10.6)	(393)	
Id	RhCl$(CO)_2$(BtzH)[a]	30.5	1.6	13.3	–	92
		(30.6)	(1.6)	(13.4)		
IIa	$[Rh(\mu\text{-Btz})(COD)]_2$	51.9	4.8	12.1	722	94
		(51.1)	(4.9)	(12.7)	(658)	
IIb	$[Rh(\mu\text{-Btz})(NBD)]_2$	49.5	3.9	13.1	660	87
		(49.8)	(3.7)	(13.4)	(626)	
IIc	$[Rh(\mu\text{-Btz})(TFB)]_2$	47.9	2.5	9.0	–	81
		(48.3)	(2.2)	(9.4)		
IId	$[Rh(\mu\text{-5-}NO_2Btz)(COD)]_2$	45.4	4.0	14.1	751	74
		(44.9)	(4.0)	(14.9)	(750)	
IIg	$[Rh(\mu\text{-Btz})(CO)(PPh_3)]_2$[b]	58.2	3.9	7.8	1111	64
		(58.7)	(3.7)	(8.2)	(1022)	
IIh	$[Rh(\mu\text{-5-}NO_2Btz)(CO)(PPh_3)]_2$[c]	54.7	3.8	9.1	1129	60
		(53.8)	(3.2)	(10.0)	(1114)	
IIIa	(COD)ClRh(IIa)RhCl(COD)	45.7	4.8	6.9	750	88
		(45.9)	(4.9)	(7.2)	(1151)	
IIIb	(TFB)ClRh(IIa)RhCl(TFB)	45.5	3.2	5.6	856	56
		(45.0)	(3.2)	(6.0)	(1387)	
IIIc	(COD)ClRh(IIc)RhCl(COD)	45.1	3.4	5.9	935	62
		(45.0)	(3.2)	(6.0)	(1387)	
IIId	$(CO)_2$ClRh(IIa)RhCl$(CO)_2$[d]	35.8	3.3	7.9	950	40
		(36.7)	(3.0)	(8.0)	(1047)	
IIIe	$(CO)_2$ClRh(IIe)RhCl$(CO)_2$[e]	25.6	0.9	8.3	968	86
		(25.5)	(0.8)	(8.9)	(943)	
IVa	$Rh_2(\mu\text{-Btz})(\mu\text{-}N_3)(COD)_2$	44.9	4.7	14.2	613	84
		(45.3)	(4.8)	(14.4)	(582)	
IVb	$Rh_2(\mu\text{-Btz})(\mu\text{-SCN})(COD)_2$	45.8	4.5	9.1	622	78
		(46.1)	(4.7)	(9.3)	(598)	
IVc	$[Rh(\mu\text{-Btz})(\mu\text{-}N_3)(CO)_4]_x$[f]	25.4	0.8	17.1	–	68
		(25.1)	(0.8)	(17.5)		

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[a] ν(CO): 2090, 2075, 2015, 1985 cm^{-1} (Nujol). [b] ν(CO): 1985 cm^{-1} (CH_2Cl_2). [c] ν(CO): 1990 cm^{-1} (CH_2Cl_2). [d] ν(CO): 2086, 2020 cm^{-1} (CH_2Cl_2). [e] ν(CO): 2100, 2080, 2030, 2015 cm^{-1} (CH_2Cl_2). [f] The dichroism and complex IR spectrum in the ν(CO) region suggest the presence of intermolecular rhodium–rhodium interactions.

cyclooctadiene (COD) [10], 2,5-norbornadiene (NBD) [11] or tetrafluorobenzobarrelene (TFB) [12]) (IIa–IId) with benzotriazole and potassium hydroxide (or triethylamine).

Bubbling carbon monoxide through a dichloromethane solution of the [Rh(μ-btz)(diolefin)]$_2$ complexes probably gave [Rh(μ-btz)(CO)$_2$]$_2$ [ν(CO)(CH$_2$Cl$_2$): 2098,2090, 2036 cm^{-1}, Btz (IIe); 2105,2095,2040 cm^{-1}, 5-NO$_2$-Btz (IIf)], but these were not isolated as solids. Addition of triphenylphosphine to the solution gives [Rh(μ-btz)(CO)(PPh$_3$)]$_2$ complexes, which were isolated as stable yellow microcrystalline solids. The molecular weight measurements on [Rh(μ-btz)(diolefin)]$_2$ or [Rh(μ-btz)(CO)(PPh$_3$)]$_2$ complexes show that they are dinuclear (Table 1), suggesting that the nitrogen atoms of the benzotriazolate ligand involved in coordination are adjacent, as found for pyrazolate bridged rhodium(I) complexes [8,13]. The proposed dinuclear structure (Fig. 1) for these complexes has been confirmed by an X-ray study of [Rh(μ-Btz)(COD)]$_2$ (Rh–Rh 3.27 Å) [14]. However, the related [M(μ-Btz)(CO)(PPh$_3$)]$_x$ complexes, previously prepared by treating [Ir(CO)(Me$_2$CO)(PPh$_3$)$_2$]PF$_6$ or RhCl(CO)(PPh$_3$)$_2$ with Tl(Btz), have been described as tetramers (M = Ir) or hexamers (M = Rh), on the basis of osmometric measurements [15].

An interesting family of complexes III can be prepared by treating [Rh(μ-Btz)(diolefin)]$_2$complexes with [Rh(μ-Cl)L$_2$]$_2$ (L$_2$ = COD, TFB; L = CO) in acetone, or by adding Rh(acac)L$_2$ to a methanol suspension of RhClL$_2$(btzH). The isolated yellow materials analyze as Rh$_2$(μ-btz)(μ-Cl)L$_4$ or L$_2$ClRh(II)RhClL$_2$ *, where II = [Rh(μ-btz)L$_2$]$_2$. The molecular weight measurements on (CO)$_2$ClRh(II)RhCl(CO)$_2$ complexes (IIId–IIIe) support the tetranuclear formation, probably with the structure indicated in Fig. 2. This structure is also probably present for the related (diolefin)ClRh(II)RhCl(diolefin) complexes (IIIa–IIIc), although there may be some dissociation in solution. Complex IIIe, (CO)$_2$ClRh(IIe)RhCl(CO)$_2$ (IIe = [Rh(μ-Btz)(CO)$_2$]$_2$) can be prepared according to eq. 1, or, by bubbling carbon monoxide

$$2\ \mathrm{RhCl(CO)_2(BtzH)} + 2\ \mathrm{Rh(acac)(CO)_2} \rightarrow \mathrm{IIIe} + 2\ \mathrm{acacH} \qquad (1)$$

through a methanol suspension of compound IIIa; this results in displacement of all

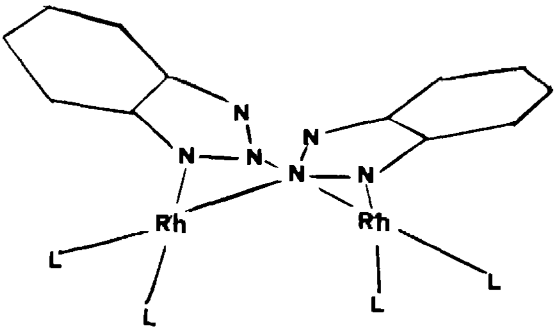

Fig. 1. Proposed structure for complexes II.

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* The same product is obtained when excess of [Rh(μ-Cl)L$_2$]$_2$ is used.

cyclooctadiene groups and the precipitation of complex IIIe. However, when carbon monoxide is bubbled through a dichloromethane solution of complex IIIa, and hexane is added in order to precipitate the rhodium complex, compound IIId is obtained, suggesting the following equilibrium (eq. 2)

$$\mathrm{IIIa} \xrightarrow{8\,\mathrm{CO}} \mathrm{IIIe} \underset{4\,\mathrm{CO}}{\overset{2\,\mathrm{COD}}{\rightleftharpoons}} \mathrm{IIId} \qquad (2)$$

Related equilibria have been previously observed for rhodium(I) indazolate complexes [16]. The possible formation of complexes III can be considered to be consequence of the stability of the "$Rh(\mu\text{-btz})_2Rh$" unit, as well as the presence of a nitrogen atom per bridging benzotriazolate ligand, available as donor site for coordination with a "$RhClL_2$" unit. On the other hand, several homodinuclear platinum(II) complexes having both μ-pyrazolate and μ-chloride as bridging groups, as well as the heterodinuclear compound, $(C_2H_4)ClPt(\mu\text{-pz})(\mu\text{-Cl})Rh(COD)$, have been reported [17–19].

No reaction was observed upon addition of NaN_3 to $[Rh(\mu\text{-Btz})(COD)]_2$, but rhodium(I) complexes having both μ-pyrazolate and μ-azide as bridging ligands, of formula $Rh_2(\mu\text{-Btz})(\mu\text{-N}_3)(COD)_2$, or the related $Rh_2(\mu\text{-Btz})(\mu\text{-SCN})(COD)_2$, were obtained from the reaction of the complex IIIa, [(COD)ClRh(IIa)RhCl(COD)], with NaN_3 or NaSCN. Table 1 lists the analytical data and molecular weight measurements confirming the proposed formulation. The $Rh_2(\mu\text{-Btz})(\mu\text{-N}_3)(COD)_2$ complex react with carbon monoxide with displacement of the coordinated cyclooctadiene, and formation of the red $[Rh_2(\mu\text{-Btz})(\mu\text{-N}_3)(CO)_4]_x$ derivative. The latter complex can also be prepared by adding NaN_3 to a methanol suspension of $(CO)_2ClRh(IIe)RhCl(CO)_2$. No tetranuclear compounds similar to complexes III were observed when the azide or the thiocyanate groups were used. This different behaviour could be related to the favourable formation of square-planar $RhCl(L_2)$(N-donor) * derivatives [1] by cleavage of $[Rh(\mu\text{-Cl})L_2]_2$ compounds with

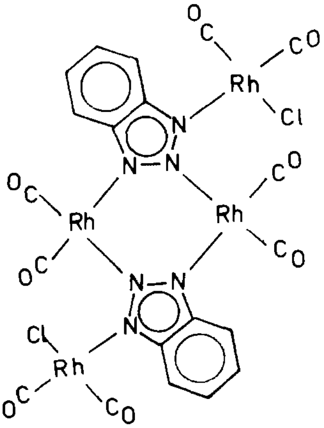

Fig. 2. Proposed structure for complex IIIe.

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[*] Attempts to prepare the complex $Rh(N_3)(COD)(BtzH)$ by treatment $[Rh(\mu\text{-N}_3)(COD)]_2$ [10] with BtzH, were unsuccessful; $[Rh(\mu\text{-Btz})(COD)]_2$ was the main product isolated.

nitrogen donors (in this case, complexes II). However, a dinuclear rhodium(III) complex of formula $Rh_2(\mu\text{-}N_3C_6F_4)(\mu\text{-}N_3)(N_3C_6F_4)_2(C_5Me_5)_2$, containing one bridging azide and one bridging and two terminally bonded bis(trifluoromethyl)triazolate ligands, has been reported by Maitlis and coworkers [20].

Experimental

Reactions were carried out at room temperature, unless otherwise stated. Solvents were distilled before use. C, H, N analysis were performed with a Perkin–Elmer 240 microanalyzer. IR spectra were recorded on a Perkin–Elmer 599 spectrophotometer using Nujol mulls between NaCl plates. Abcissa expansion was used in the carbonyl region. Molecular weights were measured on a Perkin–Elmer 115 osmometer.

Preparation of RhCl(COD)(btzH) complexes (Ia–Ic)

The general method used for making the compounds in which btzH = BtzH, 5-NO_2BtzH or 5,6-Me_2BtzH is described for RhCl(COD)(BtzH). The others were prepared similarly.

To a suspension of $[Rh(\mu\text{-}Cl)(COD)]_2$ (0.05 g, 0.1 mmol) in acetone (10 ml), benzotriazole (0.024 g, 0.2 mmol) was added. The starting material dissolved when the benzotriazole was added. After stirring for 30 min, the solution was evaporated under vacuum, and hexane was added. The solid which separated was filtered off, washed with hexane, and air-dried.

Preparation of $RhCl(CO)_2(BtzH)$ (Id)

Carbon monoxide was bubbled through a solution of RhCl(COD)(BtzH) (0.050 g, 0.13 mmol) in dichloromethane (20 ml) for 15 min. $RhCl(CO)_2(BtzH)$ separated immediately almost quantitatively. It was filtered off, washed with hexane and air-dried.

Preparation of $[Rh(\mu\text{-}btz)(diolefin)]_2$ complexes (IIa–IId)

The compounds in which the diolefin was 1,5-cyclooctadiene, 2,5-norbornadiene, or tetrafluorobenzobarrelene, were prepared by the three general routes described below for the case where the diolefin was 1,5-cyclooctadiene and btz was benzotriazole.

(i) To a suspension of $[Rh(\mu\text{-}Cl)(COD)]_2$ (0.05 g, 0.1 mmol) in methanol (15 ml), a solution of BtzH (0.024 g, 0.2 mmol) and KOH/MeOH 0.091 *N* (2.23 ml, 0.2 mmol) in methanol (5 ml) was added dropwise. The starting material dissolved when a half of the potassium benzotriazolate had been added, and when the addition was complete the product $[Rh(\mu\text{-}Btz)(COD)]_2$ separated almost quantitatively. It was filtered off, washed with methanol/water (2/1 ml) and air-dried.

(ii) The reaction was carried out as above but the solution of potassium benzotriazolate was replaced by a mixture of benzotriazole (0.024 g, 0.2 mmol) and triethylamine (28.3 μl, 0.2 mmol) in methanol (15 ml).

(iii) To a suspension of RhCl(COD)(BtzH) (0.05 g, 0.13 mmol) in methanol (15 ml), triethylamine (19 μl, 0.13 mmol) was added. The starting material dissolved immediately, and $[Rh(\mu\text{-}Btz)(COD)]_2$ separated as a yellow solid. The solid was filtered off, washed with methanol/water (2/1 ml), and air-dried.

Preparation of the complexes [Rh(μ-Btz)(CO)(PPh$_3$)]$_2$ (IIg–IIh)

The general method used making these compounds in which btz = 5-NO$_2$Btz or Btz, is described for [Rh(μ-Btz)(CO)(PPh$_3$)]$_2$. The other was prepared similarly.

Carbon monoxide was bubbled through a solution of [Rh(μ-Btz)(COD)]$_2$ (0.05 g, 0.076 mmol) in dichloromethane (15 ml) for 15 min, to give a pale yellow solution of the dicarbonyl complex [Rh(μ-Btz)(CO)$_2$]$_2$. Addition of solid triphenylphosphine (0.040 g, 0.15 mmol) caused evolution of carbon monoxide and the colour intensified. The solution was evaporated under vacuum and hexane was added. The solid which separated out was filtered off washed with hexane, and air-dried.

Preparation of complexes L$_2$ClRh(II)RhClL$_2$ (IIa–IId)

The compounds in which II was IIa or IIc and L$_2$ = (COD), (TFB) or (CO)$_2$ were prepared by the two general routes described below for the case where II = IIa and L$_2$ = COD.

(i) To a suspension of [Rh(μ-Btz)(COD)]$_2$ (0.033 g, 0.05 mmol) in acetone, [Rh(μ-Cl)(COD)]$_2$ (0.024 g, 0.05 mmol) is added. The starting material dissolved and (COD)ClRh(IIa)RhCl(COD) separated as a yellow solid. To complete the precipitation, the suspension was concentrated under vacuum and hexane was added. The solid was filtered off, washed with hexane and air-dried.

(ii) To a suspension of RhCl(COD)(BtzH) (0.030 g, 0.08 mmol) in methanol, Rh(acac)(COD) (0.025 g, 0.08 mmol) was added. Solid separated immediately and was filtered off, washed with methanol/water (2/1 ml), and air dried.

Preparation of complex (CO)$_2$ClRh(IIe)RhCl(CO)$_2$ (IIIe)

To a suspension of RhCl(CO)$_2$BtzH (0.040 g, 0.14 mmol) in methanol (15 ml), Rh(acac)(CO)$_2$ (0.036 g, 0.14 mmol) was added. Solid separated immediately and was filtered off, washed methanol/water (2/1 ml), and air-dried.

The complex can also be prepared by reaction of IIIa with carbon monoxide in methanol.

Reaction of (COD)ClRh(IIa)RhCl(COD) with CO

(i) Carbon monoxide was bubbled through a suspension of (COD)ClRh(IIa)-RhCl(COD) (0.040 g, 0.03 mmol) in methanol (15 ml) for 30 min. The expected product, (CO)$_2$ClRh(IIe)RhCl(CO)$_2$ separated almost quantitatively. It was filtered off, washed with methanol and hexane, and air-dried.

(ii) Carbon monoxide was bubbled through a solution of (COD)ClRh(IIa)-RhCl(COD) (0.040 g, 0.03 mmol) in dichloromethane (15 ml) for 30 min to give a pale yellow solution (ν(CO)(CH$_2$Cl$_2$): 2100, 2080, 2030, 2015 cm^{-1}). The solution was evaporated under vacuum, and the colour intensified. Hexane was added to complete the precipitation. The precipitated solid, (CO)$_2$ClRh(IIa)RhCl(CO)$_2$ was filtered off, washed with hexane, and air-dried (ν(CO)(CH$_2$Cl$_2$): 2086, 2020 cm^{-1}).

Preparation of the complexes Rh$_2$(μ-Btz)(μ-Y)(COD)$_2$ (IVa–IVb)

The general method used making there compounds in which Y = N$_3$ or SCN is illustrated by the case of Rh$_2$(μ-Btz)(μ-N$_3$)(COD)$_2$.

To a suspension of (COD)ClRh(IIa)RhCl(COD) (0.040 g, 0.07 mmol) in methanol (15 ml), NaN$_3$ (0.0045 g, 0.07 mmol) was added. The mixture was heated for 2 h at 50°C. The suspension was filtered off, washed with methanol/water (2/1 ml), and air dried.

Preparation of the $[Rh_2(\mu\text{-}Btz)(\mu\text{-}N_3)(CO)_4]_x$ *(IVc)*

Two routes are available for the preparation of this compound.

(i) To a suspension of $(CO)_2ClRh(IIe)RhCl(CO)_2$ (0.050 g, 0.1 mmol) in methanol (15 ml), NaN_3 (0.0069 g, 0.1 mmol) was added. Solid $[Rh_2(\mu\text{-Btz})(\mu\text{-}N_3)(CO)_4]_x$ separated immediately, and was filtered off, washed with methanol/water (2/1 ml), and air dried.

(ii) Carbon monoxide was bubbled through a suspension of $Rh_2(\mu\text{-Btz})(\mu\text{-}N_3)(COD)_2$ (0.040 g, 0.068 mmol) in dichloromethane (15 ml), for 30 min to give a pale yellow solution of the complex $[Rh_2(\mu\text{-Btz})(\mu\text{-}N_3)(CO)_4]_x$. The solution was evaporated under vacuum and hexane was added. The solid which separated out was filtered off, washed with hexane, and air dried.

The low solubility of this complex precluded molecular weight measurements.

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