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Rhodium(III)-mediated cycloaddition of alkynes: reactivity of [Cp*Rh(η^2-NO_3)(OTf)] bearing two labile ligands

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Abstract

[Cp*Rh(η^2-NO_3)(OTf)] (**1**) mediated cyclodimerization or cyclotrimerization of alkynes and alkynyl esters. In addition, compound **1** reacted with propargyl halides to give triply halide-bridged dinuclear compounds, [Cp*Rh(μ_2-X)$_3$RhCp*](OTf) (X = Cl or Br).

Keywords: Cyclodimerization; Cyclotrimerization; Alkynes; Alkynyl esters

1. Introduction

Metal-mediated cycloaddition of alkynes has received widespread attractions [1], and cyclobutadiene–metal compounds are particularly interesting due to their applications as reagents or intermediates in organic synthesis [2]. Many synthetic strategies employ a designed metal precursor to modify and control its reactivity toward alkynes [3]. In this context, extensive studies on alkyne–cobalt reactions, catalytic or stoichiometric, have demonstrated a high chemo-, regio-, or stereoselectivity [1–4]. For example, Caffyn et al. recently reported the reaction of (η^5-phospholyl)cobalt dicarbonyl with alkynes to give an (η^4-cyclobutadiene)–cobalt compound and free arenes [4a]. However, corresponding studies on rhodium counterparts have been relatively unexplored, and most reactions have been carried out with rhodium(I) compounds although a few rhodium(III) compounds have been investigated [1–3,5].

Very recently, we reported the synthesis and structure of [Cp*Rh(η^2-NO_3)(OTf)] (**1**), which contains two labile ligands, nitrato (NO_3^-) and trifluoromethanesulfonato (OTf^-) [6]. As a continuation of our work, we examined the reactivity of **1** toward alkynes, which are internal or terminal with various substituents. In this paper, we report the results of the reactivity of **1** toward several alkynes.

2. Experimental

Unless otherwise stated, all reactions have been performed with standard Schlenk line and cannula techniques under argon. Air-sensitive solids were manipulated in a glove box filled with argon. Glassware was soaked in KOH-saturated 2-propanol for about 24 h and washed with distilled water and acetone before use. Glassware was either flame- or oven-dried. Hydrocarbon solvents were stirred over concentrated H_2SO_4 for about 48 h, neutralized with K_2CO_3, stirred over sodium metal, and distilled by vacuum transfer. Diethyl ether was stirred over sodium metal and dichloromethane over CaH_2. Acetone, methyl alcohol, and ethyl alcohol were distilled and stored under argon. NMR solvent ($CDCl_3$) was degassed by freeze-pump-thaw cycles before use and stored over molecular sieves under argon. Rhodium(III) chloride hydrate, 1,2,3,4,5-pentamethylcyclopentadiene (Cp*), silver nitrate ($AgNO_3$), silver trifluoromethanesulfonate (AgOTf), diphenylacetylene (PhC≡CPh), phenylacetylene (HC≡CPh), 4-ethynyltoluene (HC≡C$C_6H_4CH_3$), 3-chloro-1-propyne (HC≡CCH_2Cl), 3-bromo-1-propyne (HC≡CCH_2Br), di-

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methyl acetylenedicarboxylate ($MeO_2CC{\equiv}CCO_2Me$), and diethyl acetylenedicarboxylate ($EtO_2CC{\equiv}CCO_2Et$) were purchased. [Cp*Rh(η^2-NO_3)(OTf)] (**1**) was prepared by the literature method [6].

^{1}H- and $^{13}C\{^1H\}$-NMR spectra were recorded with a Bruker AMX 500 MHz spectrometer. IR spectra were recorded with a Nicolet Avatar 320 FTIR spectrophotometer. Elemental analyses were performed by the Korea Basic Science Institute.

2.1. Preparation of [Cp*Rh(η^4-C_4Ph_4)] (2)

A solution of **1** (100 mg, 0.22 mmol) and diphenylacetylene (79 mg, 0.44 mmol) in EtOH (30 ml) was refluxed for 3 h, and then the solvent was removed under vacuum. The resulting solids were washed with EtOH (20 ml) and diethyl ether (20 ml × 2), and then dried under vacuum to give a yellow solid of [Cp*Rh(η^4-C_4Ph_4)] (**2**) (98 mg, 0.16 mmol, 74%). This product was recrystallized from CH_2Cl_2–MeOH. ^{1}H-NMR ($CDCl_3$): δ 1.55 (s, 15H, $C_5(CH_3)_5$), 7.26–7.12 (m, 20H, C_6H_5). $^{13}C\{^1H\}$-NMR ($CDCl_3$): δ 9.30 (s, $C_5(CH_3)_5$), 93.9 (d, $J_{Rh-C} = 6.7$ Hz, $C_5(CH_3)_5$). Anal. Calc. for $C_{38}H_{35}Rh$: C, 76.76; H, 5.93%. Found: C, 76.59; H, 6.07%. M.p. (dec.): 273–275 °C. IR (KBr): 3058, 2965, 1635, 1601 (C=C), 1499, 1097, 1070, 1027, 804, 743, 701, 648 cm^{-1}.

2.2. Preparation of [Cp*Rh{(η^4-C_4)HAr_2(C≡CAr)}] (**3a**: Ar = Ph; **3b**: Ar = p-tolyl)

A solution of **1** (100 mg, 0.22 mmol) and phenylacetylene (0.024 ml, 0.22 mmol) in acetone (30 ml) was stirred for 3 h, and then the solvent was removed under vacuum. The resulting solids were washed with MeOH (20 ml), and then dried under vacuum to give a yellow solid of **3a** (28 mg, 0.052 mmol, 23%).

Compound **3b** was similarly prepared (29 mg, 0.050 mmol, 22%). Compounds **3a** and **3b** were identified by the comparison with literature data [7].

2.3. Reactions of 1 with dimethyl acetylenedicarboxylate and diethyl acetylenedicarboxylate

A solution of **1** (100 mg, 0.22 mmol) and dimethyl acetylenedicarboxylate (0.059 ml, 0.66 mmol) in acetone (30 ml) was stirred for 3 h, and then the solvent was removed under vacuum. The resulting solids were extracted with diethyl ether (20 ml × 2), and then dried under vacuum to give a mixture of 1,2,4- and 1,3,5-$C_6H_3(CO_2Me)_3$ (38 mg, 0.15 mmol, 68%).

The reaction of **1** with diethyl acetylenedicarboxylate similarly proceeded (41 mg, 0.14 mmol, 62%). The spectral data for these trimers are exactly the same as those for the genuine compounds in the literature [8].

2.4. Preparation of [Cp*Rh(μ-X)$_3$RhCp*](OTf) (**4a**: X = Cl; **4b**: X = Br)

A solution of **1** (100 mg, 0.22 mmol) and 3-chloro-1-propyne (0.051 ml, 0.66 mmol) in acetone (30 ml) was stirred for 3 h, and then the solvent was removed under vacuum. The resulting solids were washed with diethyl ether (20 ml × 2) and hexane (20 ml × 2), and then dried under vacuum to give an orange solid of [Cp*Rh(μ-Cl)$_3$RhCp*](OTf) (**4a**) (58 mg, 0.080 mmol, 72%). This product was recrystallized from CH_2Cl_2–hexane. ^{1}H-NMR ($CDCl_3$): δ 1.69 (s, 15H, $C_5(CH_3)_5$). $^{13}C\{^1H\}$-NMR ($CDCl_3$): δ 9.9 (s, $C_5(CH_3)_5$), 96.5 (d, $J_{Rh-C} = 16.3$ Hz, $C_5(CH_3)_5$). Anal. Calc. for $C_{21}H_{30}F_3O_3SCl_3Rh_2$: C, 34.47; H, 4.13; S, 4.38%. Found: C, 33.95; H, 4.02; S, 4.46%. M.p. (dec.) > 300 °C. IR (KBr): 2915, 1632, 1470, 1376, 1265, 1173, 1034, 646 cm^{-1}.

Compound **4b** was similarly prepared (76 mg, 0.088 mmol, 79%). ^{1}H-NMR ($CDCl_3$): δ 1.76 (s, 15H, $C_5(CH_3)_5$). $^{13}C\{^1H\}$-NMR ($CDCl_3$): δ 10.4 (s, $C_5(CH_3)_5$), 97.3 (d, $J_{Rh-C} = 15.2$ Hz, $C_5(CH_3)_5$). Anal. Calc. for $C_{21}H_{30}F_3O_3SBr_3Rh_2$: C, 29.16; H, 3.50; S, 3.71%. Found: C, 30.02; H, 3.46; S, 3.67%. M.p. (dec.) > 300 °C. IR (KBr): 2920, 1629, 1470, 1376, 1271, 1146, 1026, 640 cm^{-1}.

2.5. X-ray structure determination

All X-ray data were collected with the use of a Siemens P4 diffractometer equipped with a Mo X-ray tube. Details on crystal and intensity data are given in Table 1. The orientation matrix and unit-cell parameters were determined by the least-squares analyses of the setting angles of 23 reflections for both **2** and **4a** in the range of $15.0° < 2\theta < 25.0°$. Intensity data were empirically corrected for absorption with ψ-scan data. All calculations were carried out with the use of SHELXTL programs [9].

All crystal structures were solved by direct methods. All non-hydrogen atoms were refined anisotropically. All hydrogen atoms were generated in ideal positions and refined in a riding model. Details on crystal data and refinement details are given in Table 1. Selected bond lengths and bond angles are given in Table 2.

3. Results and discussion

3.1. Reactivity toward alkynes

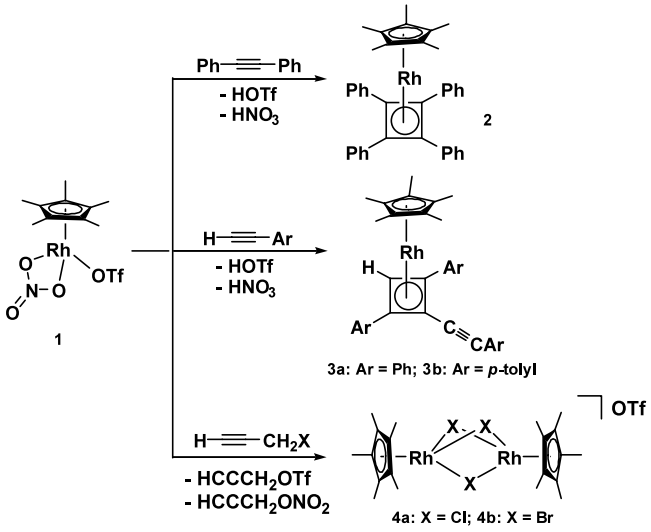
Refluxing **1** with a stoichiometric amount of an internal alkyne PhC≡CPh in ethanol for 3 h gives an (η^4-cyclobutadiene)–rhodium(I) compound (**2**) in 74% yield (Scheme 1). The same reaction in methanol in place of ethanol also proceeds, although with a lower yield

Table 1
X-ray data collection and structure refinement

	2	4a
Empirical formula	$C_{38}H_{35}Rh$	$C_{21}H_{30}F_3O_3SCl_3Rh_2$
F_w	594.57	731.68
Temperature (K)	293(2)	293(2)
Crystal system	monoclinic	triclinic
Space group	$P2_1/n$	$P\bar{1}$
Unit cell dimensions		
a (Å)	13.720(2)	14.913(3)
b (Å)	14.046(2)	15.401(4)
c (Å)	16.556(3)	15.630(3)
α (°)		96.27(1)
β (°)	112.38(1)	113.25(1)
γ (°)		115.86(2)
V (Å^3)	2950.2(7)	2791(1)
Z	4	4
D_{calc} (g cm^{-3})	1.339	1.741
μ (mm^{-1})	0.603	1.585
F(0 0 0)	1232	1456
T_{min}	0.3714	0.4143
T_{max}	0.7807	0.8023
2θ Range (°)	3.5–50	3.5–50
Scan type	ω	ω
Scan speed	variable	variable
Number of reflections measured	5358	9686
Number of reflections unique	5136	9263
Number of reflections with $I >$ $2\sigma(I)$	4709	4452
Number of parameters refined	353	595
Max in $\Delta\rho$ (e Å^{-3})	0.261	1.629
Min in $\Delta\rho$ (e Å^{-3})	−0.370	−0.678
Goodness-of-fit on F^2	1.052	1.010
R	0.0307	0.0698
wR_2 [a]	0.0760	0.1462

[a] $wR_2 = \Sigma[w(F_o^2 - F_c^2)^2]/\Sigma[w(F_o^2)^2]^{1/2}$.

(21%). However, the reaction in acetone does not occur at all. These results suggest that the solvent ethanol might act as a proton donor to the two labile ligands (NO_3^- and OTf^-) to facilitate their leaving from the rhodium coordination sphere. In addition, the reduction



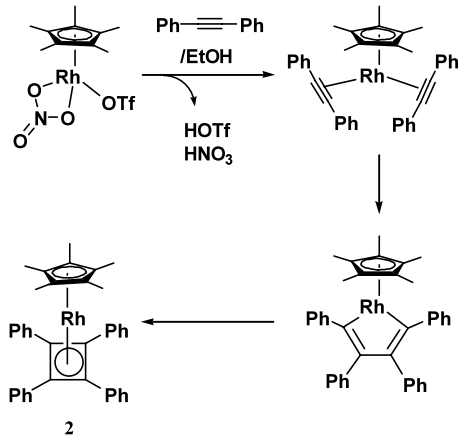
Scheme 1.

of rhodium from +3 to +1 during the reaction suggests that ethanol might also act as a reducing agent although we did not isolate acetaldehyde, the oxidation product of ethanol. The reducing ability of ethanol in this type was also observed in the Pd-mediated cyclotrimerization of phenylethyne to fulvene [10]. However, solvent effects cannot be fully understood only in terms of the acidity of alcohols, because methanol is a little bit more acidic than ethanol. Some other factors such as solubility seem to operate in our reactions.

One possible mechanism for the formation of compound **2** is proposed in Scheme 2. This mechanism adopts the most common process that goes via a metallacyclopentadiene intermediate [2]. The first step is the formation of the intermediate bis(alkyne)–rhodium, which accompanies the removal of the two labile ligands in their protonated forms. In this step, the solvent ethanol is likely to have acted as a proton donor. The next step is the oxidative coupling of the two

Table 2
Selected bond lengths (Å) and bond angles (°)

Compound **2**					
Bond lengths					
Rh1–C35	2.120(3)	Rh1–C36	2.113(3)	Rh1–C37	2.120(2)
Rh1–C38	2.117(3)	C11–C35	1.461(4)	C17–C36	1.471(4)
C23–C37	1.462(4)	C29–C38	1.468(4)	C35–C36	1.467(4)
C35–C38	1.474(4)	C36–C37	1.464(4)	C37–C38	1.465(4)
Bond angles					
C36–C35–C38	89.2(2)	C36–C37–C38	89.7(2)	C37–C36–C35	90.7(2)
C37–C38–C35	90.4(2)				
Compound **4a**					
Bond lengths					
Rh1–Cl1	2.477(4)	Rh1–Cl2	2.451(4)	Rh1–Cl3	2.456(4)
Rh2–Cl1	2.475(4)	Rh2–Cl2	2.451(4)	Rh2–Cl3	2.444(4)
Bond angles					
Rh1–Cl1–Rh2	81.3(1)	Rh1–Cl2–Rh2	82.3(1)	Rh1–Cl3–Rh2	82.3(1)



Scheme 2.

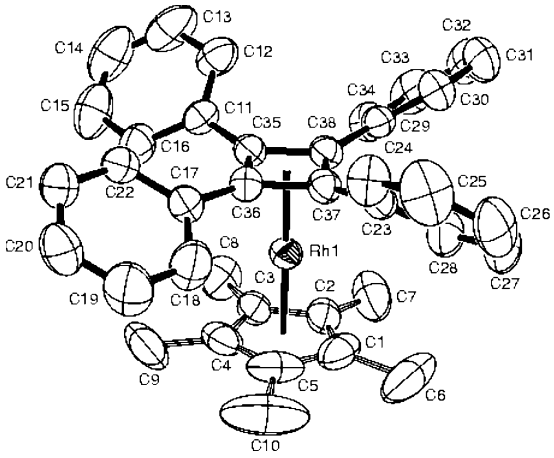


Fig. 1. ORTEP drawing of **2**, showing the atom-labeling scheme and 50% probability thermal ellipsoids.

coordinated alkyne ligands to give the intermediate metallacyclopentadiene, which undergoes reductive elimination to form the cyclobutadiene compound **2**.

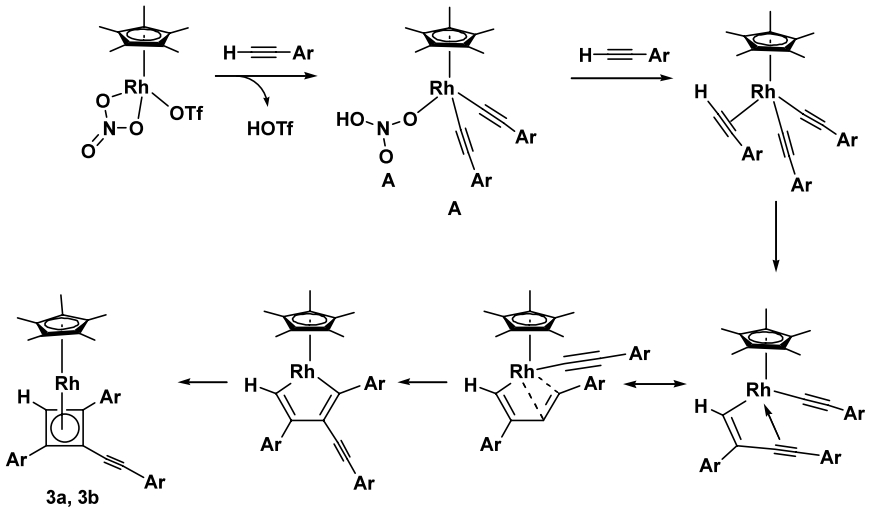
A related cycloaddition reaction of dimethyl acetylenedicarboxylate ($MeO_2CC{\equiv}CCO_2Me$) in the presence of H_2 was reported for the tetrahaptobenzene–rhodium compound [Cp*Rh{η^4-$C_6(CO_2Me)_6$}] [11]. Unfortunately, the reactions of **1** with $MeO_2CC{\equiv}CCO_2Me$, $PhC{\equiv}C{-}C{\equiv}C{-}Ph$, or $Me_3Si{-}C{\equiv}C{-}C{\equiv}C{-}SiMe_3$ in EtOH gave a mixture of several species, and we failed to separate them. In acetone, however, we do not observe any sign of reactions of **1** with internal alkynes or alkynyl esters ($PhC{\equiv}CPh$, $PhC{\equiv}CMe$, $PhC{\equiv}CEt$, $EtC{\equiv}CEt$, $EtC{\equiv}CMe$, $MeC{\equiv}C(CH_2)_2CH_3$, $Ph{-}C{\equiv}C{-}C{\equiv}C{-}Ph$, $Me_3Si{-}C{\equiv}C{-}C{\equiv}C{-}SiMe_3$, $MeO_2CC{\equiv}CCO_2Me$, and $EtO_2CC{\equiv}CCO_2Et$). To our best knowledge, cyclodimerization reactions of internal alkynes have not been investigated for Cp*Rh(III) compounds, and most of those reactions have been carried out for Cp*Rh(I) compounds [12].

Compound **2** was fully characterized by spectroscopy, elemental analysis, and X-ray diffraction. It has a two-legged pseudo-piano-stool structure, with the tetraphenylcyclobutadiene ring being regarded as the two legs (Fig. 1). The Rh–Ct1 (Ct1: a centroid of C35–C38; 1.846 Å) and Rh–Ct2 (Ct2: a centroid of C1–C5; 1.866 Å) distances and the Ct1–Rh–Ct2 angle (178.56°) are very close to those of the cyclopentadienyl (Cp) analogue [CpRh(η^4-C_4Ph_4)] [12a].

Compound **1** undergoes cyclotrimerization with terminal aryl alkynes. It reacts with a stoichiometric amount of HC≡CAr in acetone at room temperature for 3 h to give (η^4-cyclobutadiene)–rhodium(I) compounds [Cp*Rh{(η^4-C_4)HAr_2(C≡CAr)}] {Ar = Ph (**3a**), 23%; Ar = *p*-tolyl (**3b**), 22%} (Scheme 1). Lamata et al. recently prepared the identical compounds (**3a** and **3b**) by treating [Cp*Rh(L-alaninate)Cl] with phenylethyne in the presence of a base (NEt_3) for 20 h [7]. We could prepare them relatively fast even in the absence of a base, and therefore the reactivity of **1** seems to be comparable to that of [Cp*Rh(L-alaninate)Cl]. The structures of **3a** and **3b** were confirmed by comparing their spectral and crystallographic data with those for the genuine compounds [7]. Compound **1** also readily reacts with other terminal alkynes ($HC{\equiv}C{-}CMe_3$, $HC{\equiv}C{-}SiMe_3$, $HC{\equiv}C{-}(CH_2)_3CH_3$, $HC{\equiv}C{-}CH_2OH$, $HC{\equiv}C{-}(CH_2)_2OH$, $HC{\equiv}C{-}CH(OH){-}CH_3$, and $HC{\equiv}C{-}CH_2OCH_3$), but these reactions give intractable mixtures and we failed to separate them.

Although the present results do not give any detailed information about how compound **3** has been formed, one of the possible mechanisms is shown in Scheme 3, on the basis of the previous reports on alkyne cyclotrimerization. We speculate that the aryl alkyne protonates the triflate and nitrato ligands to give an intermediate **A**. Because we have used acetone as a solvent in place of ethanol, the acidic terminal alkyne hydrogen is likely to act as a proton source. In this reaction, the variable haptacity of the nitrato ligand is expected to play an important role to retain coordinative saturation of the intermediates. Compounds related to the intermediate **A** were previously reported by Maitlis's group ([Cp*Rh(C_2Ph)$_2$(NCMe)], [5]), Lamata's group ([Cp*Rh(L-prolinate)(C_2Ph)], [7]), and Ara's group ([Cp*Rh(C_2Ph)$_2$(PEt_3)], [13]). The intermediate **A** binds one alkyne, which inserts into the Rh–alkynyl bond, followed by the C–C coupling to give the metallacyclopentadiene. This metallacycle intermediate undergoes reductive elimination to give the ultimate product, compound **3**.

We examined the reactivity of **1** toward propargyl halides, terminal alkynes with a halide at the propargyl position. Treatment of **1** with three equivalents of $HC{\equiv}CCH_2X$ in acetone at room temperature for 12 h gives triply halide-bridged dinuclear compounds [Cp*Rh(μ-$X)_3$RhCp*](OTf) {X = Cl (**4a**), 72%; X = Br (**4b**), 79%} (Scheme 1). This reaction can be interpreted as the

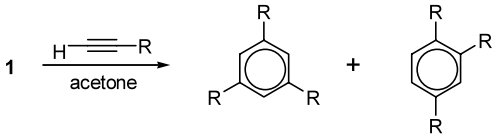


Scheme 3.

replacement of weakly coordinating ligands (NO_3^- and OTf^-) by strong donor ligands (Cl^- or Br^-). Similar triply halide-bridged rhodium salts are known: [Cp*Rh(μ-Cl)$_3$RhCp*](BPh$_4$), [Cp*Rh(μ-Cl)$_3$RhCp*](PF$_6$), and [Cp*Rh(μ-I)$_3$RhCp*](BF$_4$) [14]. The molecular structure of **4a** is given in Fig. 2. There are two crystallographically independent molecules in an asymmetric unit, which are chemically equal and have either eclipsed or staggered Cp* rings.

3.2. Reactivity toward alkynyl esters

Treating terminal alkynyl esters with **1** produces free arenes instead of (η^4-cyclobutadiene)–rhodium compounds. The reaction of **1** (100 mg) with three equivalents of HC≡CCO$_2$R in acetone at room temperature for 24 h gives a mixture of 1,2,4- and 1,3,5-C$_6$H$_3$(CO$_2$R)$_3$ (R = Me, 68%; R = Et, 62%), together with **1** (63 mg) (Eq. (1)). On the contrary to the cases for the formation of **3a** and **3b**, the terminal C–H bond cleavage does not occur. We examined the possibility of a catalytic cyclotrimerization by treating a catalytic amount of **1** with HC≡CCO$_2$Me (mole ratio: [Rh]:[ester] = 1:100), and we obtained a mixture of 1,2,4- and 1,3,5-C$_6$H$_3$(CO$_2$Me)$_3$ in 42% yield with the mole ratio of 1:6 (^{1}H-NMR). The spectral data for these trimers are exactly the same as those for the genuine compounds in the literature [8].



(1)

In summary, [Cp*Rh(η^2-NO$_3$)(OTf)] (**1**) mediated the cycloaddition of alkynes and alkynyl esters. On the other hand, the reaction of **1** with propargyl halides gave triply halide-bridged dinuclear compounds. These results indicate that the reactivity of **1** depends basically on the substituent on the alkyne. We observed a possibility of **1** as a catalyst for the cyclotrimerization of alkynyl esters to benzene derivatives. We do believe that the interesting reactivity of **1** is attributed to the two labile ligands, nitrato and trifluoromethanesulfonato.

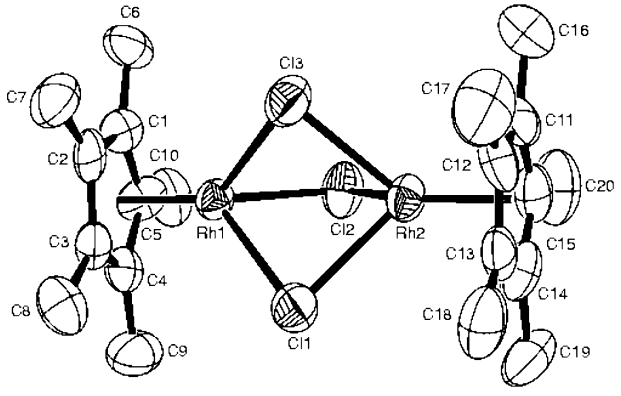


Fig. 2. ORTEP drawing of **4a**.

4. Supplementary material

Crystallographic data for the structural analysis have been deposited at the Cambridge Crystallographic Data Center: CCDC No. 201654 for compound **2** and 201655 for compound **4a**. Copies of this information may be obtained free of charge from The director, CCDC, 12 Union Road, Cambridge, CB2 1EZ, UK (Fax: +44-1223-336033; email: deposit@ccdc.cam.ac.uk or www: http://www.ccdc.cam.ac.uk).

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