and the supernatant decanted. Water (10 mL) was added to resuspend the precipitate, which was shaken vigorously and again centrifuged. After decanting, acetone was added to the precipitate which was again shaken vigorously and centrifuged. This was repeated once more, after which the precipitate was dried in vacuum. The polymer was obtained as an orange-brown solid (15 mg, 76%).

Received: August 20, 2002 [Z50010]

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[8] $\frac{T}{D}=\frac{n+1}{n-1}$ where T is the number of terminal units, D the number of dendritic units, and n the degree of polymerization. This equation is readily derived by observing the obvious facts that $T=D+1$ and $n=T+D$.

[(arene)Rh(cod)]$^+$ Complexes as Catalysts for [5+2] Cycloaddition Reactions**

Paul A. Wender* and Travis J. Williams

As part of our studies on the design and development of new metal-catalyzed [*m*+*n*] cycloaddition reactions, we previously reported the first examples of metal-catalyzed [5+2] cycloadditions of vinylcyclopropanes and tethered π systems.[1] In our initial and ongoing screening of catalysts for these reactions, rhodium(I) complexes have proven to be the most general. These complexes are active in both inter- and intramolecular cycloadditions. The latter reactions involve alkynes, alkenes, and allenes as the 2-π component and provide cycloadducts in high yields often at 25–40 °C.[2] This finding is consistent with earlier observations by our group[3] and others[4] on the utility of rhodium(I) in metal-catalyzed [4+2] cycloadditions of dienes and π systems. More recently, the groups of Gilbertson,[4e] Trost,[5] and Zhang[6] have reported other catalysts that are also effective in certain types of [5+2] cycloadditions.

Recently, Chung and co-workers have reported a readily prepared, air-stable naphthalene complex of rhodium ($[(C_{10}H_8)Rh(cod)]^+BF_4^-$) which is an efficient catalyst for [4+2] cycloaddition reactions of dienes and alkynes.[7] We have now examined the effectiveness of this and several related complexes in [5+2] cycloaddition reactions and have found them to be exceptionally effective in all cases studied, often providing cycloadducts in excellent yields in minutes at room temperature. The details of this study and X-ray crystallographic data on selected complexes are described herein.

Complex $[(C_{10}H_8)Rh(cod)]^+SbF_6^-$ (**1**) is prepared in a single step from commercially available $[\{RhCl(cod)\}_2]$[8] by treatment with $AgSbF_6$, followed by introduction of the naphthalene ligand.[9] The complex is air-stable at room temperature and retains its catalytic activity even after several months of storage.

Although complexes differing from **1** in only the counter ion have been reported,[7,9] characterization data available for these complexes do not directly establish the hapticity of the naphthalene ligand. Fortunately, with some effort, we were able to crystallize complex **1** from a concentrated dichloromethane solution under an anhydrous atmosphere. X-ray crystallographic analysis of these crystals led to the ORTEP representation of this complex given in Figure 1. This is apparently the first X-ray structure of a simple η^6 naphthalene–rhodium complex,[10] and is comparable to crystal structures that have been recorded for some similar ruthenium– and rhodium–naphthalene complexes.[11]

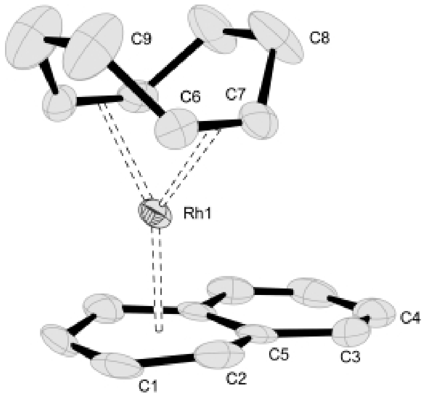

Figure 1. ORTEP diagram of $[(C_{10}H_8)Rh(cod)]^+SbF_6^-$ (**1**). Ellipsoids drawn at 50% probability level.

Complex **1** was tested in a variety of [5+2] cycloaddition reactions and compared, where relevant, with some other effective catalysts (Table 1). The initial series was selected to explore the utility of the catalyst with a diverse variety of substitution patterns. Excellent results were obtained with vinylcyclopropanes tethered to terminal and internal alkynes, alkynoates, and alkenes. Substitution of the vinylcyclopropane is tolerated both on and adjacent to the cyclopropane

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Supporting information for this article is available on the WWW under http://www.angewandte.org or from the author.

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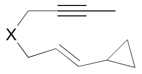
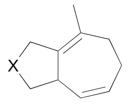
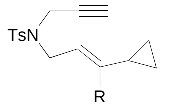
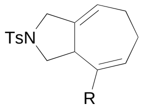
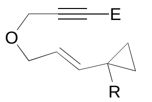
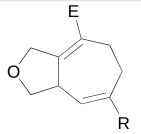
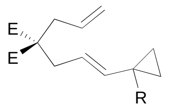
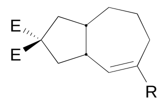
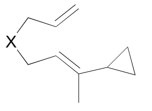
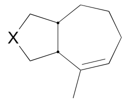
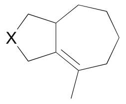
Table 1. Performance of complex **1** with a variety of alkyne vinylcyclopropanes and alkene vinylcyclopropanes.

Entry	Substrate, Catalyst	Cycloadduct, Yield	Conditions[a]
			
	2 $X = C(CO_2Me)_2$	**3**	
1	2 mol% **1**	> 99%[b]	15 min, RT, 0.15 M
2	1 mol% $[\{RhCl(CO)_2\}_2]$	89%	PhMe, 3 h, 110 °C, 2.0 M
3	10 mol% $[RhCl(PPh_3)_3]$	90–95%	TFE,[c] 19 h, 55 °C, 0.01 M
	4 X = NTs	**5**	
4	5 mol% **1**[d]	90%	65 min, RT, 0.20 M
			
	6 R = H	**7**	
5	5 mol% **1**[d]	90%[e]	65 min, RT, 0.20 M
	8 R = Me	**9**	
6	5 mol% **1**	93%	60 min, 60 °C, 0.03 M
			
	10 R = H	**11**	
7	2 mol% **1**	96%	16 min, RT, 0.15 M
8	10 mol% $[RhCl(PPh_3)_3]$, AgOTf	81%	PhMe, 1 h, 110 °C, 5.0 M
	12 R = Me	**13**	
9	2 mol% **1**	98%	10 min, RT, 0.11 M
10	10 mol% $[RhCl(PPh_3)_3]$, AgOTf	83%	PhMe, 1 h, 110 °C, 5.0 M
			
	14 R = H	**15**	
11	5 mol% **1**	96%	6 h, 60 °C, 0.05 M
12	0.1 mol% $[RhCl(PPh_3)_3]$, AgOTf	90%	PhMe, 15 h, 110 °C, 0.01 M
	16 R = Me	**17**	
13	5 mol% **1**	90%	6.5 h, 60 °C, 0.01 M
14	1 mol% $[RhCl(PPh_3)_3]$, AgOTf	90%	PhMe, 10 h, 110 °C, 0.01 M
		 	
	18 X = NTs	**19**	
15	5 mol% **1**	76%	19 h, 60 °C, 0.03 M
	20 $X = C(CO_2Me)_2$	**21a**, **21b**	
16	10 mol% **1**	75% (**a**)	10 h, 70 °C, 0.03 M
17	5 mol% $[RhCl(PPh_3)_3]$, AgOTf	78% (**b**)	PhMe, 15 h, 110 °C, 0.01 M
18	5 mol% $[RhCl(PPh_3)_3]$, AgOTf	67%, 2.3:1 (**a**:**b**)	PhMe, 66 h, 85 °C, 0.01 M

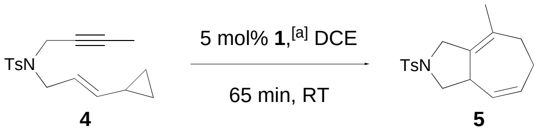
[a] Unless otherwise indicated, each reaction was run in a tightly capped vial at the indicated temperature, catalyst load, and concentration in 1,2-dichloroethane. [b] Found 85% with BF_4^- anion. [c] TFE = 2,2,2-trifluoroethanol. [d] Catalyst added in four aliquots. [e] 1-g scale, found 96% with BF_4^- anion.

ring. Diester-substituted and heteroatom (O, NTs) tethers are well tolerated. Reactions were conducted with 2 to 10 mol% catalyst at up to 0.20 M. It is noteworthy that the yields obtained with this catalyst exceed those recorded with other previously explored catalysts of the [5+2] reaction.[1,2] Furthermore, the reactions with the naphthalene catalyst (**1**) were found to be more rapid than those with other catalysts. For example, substrate **2** is readily converted in > 99% yield to cycloadduct **3** in only 15 min at room temperature (entry 1). By contrast, this same transformation requires 3 h in refluxing toluene when $[RhCl(PPh_3)_3]$ (Wilkinson's catalyst) is used (89%, entry 2) and 19 h at 55 °C in 2,2,2-trifluoroethanol when $[\{RhCl(CO)_2\}_2]$ is employed (90–95%, entry 3). Importantly, in a test preparative experiment, the yield for the conversion of **6** to **7** was found to change by < 1% as the scale was increased over two orders of magnitude (from milligrams to grams).

Complex **1** efficiently catalyzes the reactions of both alkynes and alkenes with vinylcyclopropanes (Table 1, entries 11, 13, 15, and 16) and offers greater generality than non-rhodium catalysts reported thus far.[5] This is particularly advantageous in the case of substrate **18**, for which a complex mixture of side products is observed with Wilkinson's catalyst. In the case of substrate **20**, product alkene isomerization is observed with Wilkinson's catalyst, but not with complex **1**. Isomerization is also not seen in unconjugated cycloheptadiene products of the reactions of tethered alkyne vinylcyclopropanes,[12] as is sometimes encountered when such compounds are treated with Wilkinson's catalyst.[2] Diester- and ether-tethered substrates afforded optimum performance with 2 mol% catalyst at 0.20 M substrate concentration. Sulfonamide-tethered substrates were found to be less reactive at lower catalyst loading, yet **4** was found to react efficiently at high concentrations (Table 2) when catalyst was added in portions.

Given the excellent performance of complex **1** in the above study, we next examined several related complexes that differ in the arene ligand. Three additional complexes based on 1,4-dimethylnaphthalene, benzene, and hexamethylbenzene were prepared (Table 3). X-ray crystallographic data (obtained for **1**, **22**, and **24**) and solution NMR data suggest that these complexes have a similar η^6-arene-coordination state.[13] The complexes were compared with each other and with the catalyst generated in situ by the treatment of $[\{RhCl(cod)\}_2]$ with $AgSbF_6$ (Table 3, entry 1) by using the conditions found to be optimal for conversion of substrate **2** with catalyst **1**.

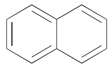
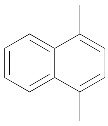
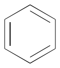
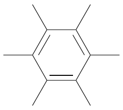
Table 2. Effect of concentration on the conversion of **4**.



Initial Conc.	Yield
0.20 M	90 %
0.80 M	88 %
1.60 M	84 %

[a] Catalyst added as four aliquots delivered every 15 min from a suspension in 1,2-dichloroethane (DCE).

Table 3. Synthesis and reaction of some $[(\text{arene})\text{Rh(cod)}]^+\text{SbF}_6^-$ complexes.

Entry[a]	Rhodium complex (yield)	Yield[b] of **3**
1	$[\{\text{RhCl(cod)}\}_2]$ + 2 AgSbF_6	90 %
	Arene Complexes ($[(\text{arene})\text{Rh(cod)}]^+\text{SbF}_6^-$)	
2	**1** (70 %) arene = 	> 99 %
3	**22** (67 %) arene = 	88 %
4	**23** (65 %) arene = 	91 %
5	**24** (87 %) arene = 	Trace

[a] Arene complexes prepared as with **1**, see Supporting Information for preparative details. [b] Yield of the conversion of **2** to **3** with 2 mol % Rh catalyst, each run in 1,2-dichloroethane (0.15 M) at room temperature in 15 min.

The trend is noticed that increasing arene substitution with methyl groups results in lower yields.[14] For example, benzene-ligated complex **23** was found to be quite effective in mediating the intramolecular [5+2] reaction, while the hexamethylbenzene-ligated complex **24** was not. These findings are consistent with the results of Chung et al. according to which $[(\text{C}_6\text{Me}_6)\text{Rh(cod)}]^+\text{BF}_4^-$ has no catalytic activity in the [4+2] cycloaddition. We observed similar results when complexes **1**, **23**, and **24** were examined in the intermolecular [4+2] cycloaddition of 2,3-dimethyl-1,3-butadiene and propargyl methyl ether; [4+2] cycloadduct yields of > 99 %, 88 %, and ≈0 % were obtained, respectively.[15]

In conclusion, we have shown that complex **1** is an exceptionally effective and, in cases where comparisons have been made, preferred catalyst for the [5+2] cycloaddition reaction of tethered alkyne vinylcyclopropanes and alkene vinylcyclopropanes. The alkyne substrates are converted to cycloadducts in high yield (> 90 %) and in only minutes at room temperature. Alkene vinylcyclopropanes, which do not undergo cycloaddition with ruthenium catalysts, are readily converted to cycloadducts with the new catalysts, often in higher yields and under milder conditions than are found with other rhodium(I) catalysts reported thus far. Tether variations as well as substitution of the C_5 and C_2 components are well tolerated. Further studies on these and new catalysts for this reaction are continuing.

Experimental Section

Full procedures and characterization data are given in the Supporting Information.

Sample procedure: Freshly prepared and crystallized substrate **6** (1.00 g, 3.46 mmol) was weighed out in an oven-dried 25 mL flask equipped with a stir bar. 1,2-Dichloroethane (DCE) (17.3 mL, 0.20 M) and catalyst complex **1** (99.3 mg, 173 μmol, added in four aliquots dispersed in 1.25 mL DCE and spaced 15 min apart) were then added as the reaction was stirred at room temperature for a total 55 minutes. The crude product was concentrated and purified by flash column chromatography (elution with 10 % diethyl ether in pentane). Product-containing fractions were concentrated and recrystallized to yield product **7**, 900.3 mg (90 %) as a white solid.

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[10] Crystal structure analysis of complex **1** (also see Supporting Information): Crystal dimensions are $0.06 \times 0.11 \times 0.18$ mm^3, orthorhombic, spacegroup *Pnma*, $a = 13.199(1)$ Å, $b = 9.605(1)$ Å, $c = 15.007(1)$ Å, $V = 1902.4(0)$ Å^3, $Z = 4$, $\rho_{\text{calcd}} = 2.01\ \text{g cm}^{-3}$, $\text{Mo}_{K\alpha}$ radiation, $\lambda = 0.71069$ Å, $\mu(\text{Mo}_{K\alpha}) = 23.38\ \text{cm}^{-1}$, $T = -133\,°\text{C}$. Data were collected on a Bruker-Siemens SMART CCD diffractometer, 8469 measured

reflections, 2495 unique reflections, 1700 used ($4^\circ < 2\Theta < 49.36^\circ$), $R_1 = 0.030$ (3344 reflections, $I > 10\sigma(I)$), $wR_2 = 7.4\,\%$ (all data), GOF = 0.97. Minimum and maximum peaks in the final difference map are −1.25 and 1.47 e Å^{-3}. CCDC-191383 (**1**), CCDC-191384 (**7**), CCDC-191385 (**22**), and CCDC-191386 (**24**) contain the supplementary crystallographic data for this paper. These data can be obtained free of charge via www.ccdc.cam.ac.uk/conts/retrieving.html (or from the Cambridge Crystallographic Data Centre, 12, Union Road, Cambridge CB2 1EZ, UK; fax: (+44) 1223-336-033; or deposit@ccdc.cam.ac.uk)

[11] The nearest examples are $[(\eta^6\text{-}C_{10}H_8)Ru(cod)]$, M. Crocker, M. Green, J. A. K. Howard, N. C. Norman, D. M. Thomas, *J. Chem. Soc. Dalton Trans.* **1990**, 2299; and $[(\eta^4\text{-}C_{10}H_8)Rh(Cp)]$, G. Müller, P. E. Gaede, C. Hirsch, K. Qaio, *J. Organomet. Chem.* **1994**, *472*, 329–335.

[12] As determined by X-ray crystallography (**7**) and NMR, see Supporting Information.

[13] No other hapticity was noticed for the naphthalene ligand. For more structural and NMR information on related ruthenium–naphthalene complexes, see a) M. A. Bennett, H. Neumann, M. Thomas, X.-Q. Wang, P. Pertici, P. Salvadori, G. Vituilli, *Organometallics* **1991**, *10*, 3237–3245; b) M. A. Bennett, Z. Lu, X. Wang, M. Brown, D. C. R. Hockless, *J. Am. Chem. Soc.* **1998**, *120*, 10409–10415.

[14] For more information on the effect of arene methylation in $\{(\eta^6\text{-arene})Rh^I\}$ complexes, see: E. T. Singewald, C. S. Slone, C. L. Stern, C. A. Mirkin, G. P. A. Yap, L. M. Liable-Sands, A. L. Rheingold, *J. Am. Chem. Soc.* **1997**, *119*, 3048–3056.

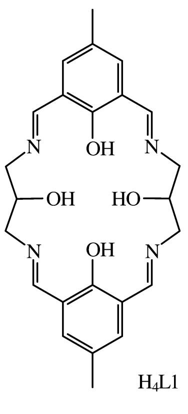
[15] See supporting information for details; yields given for the conversion of 2,3-dimethylbutadiene and propargyl methyl ether to the corresponding cyclohexadiene.

Di-, Tri-, and Tetracopper(II) Complexes of a Pseudocalixarene Macrocycle**

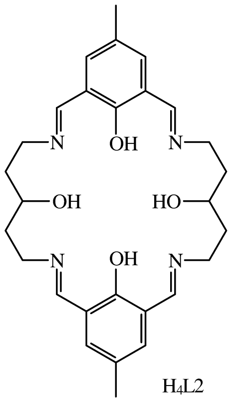
Julia Barreira Fontecha, Sandrine Goetz, and Vickie McKee*

Schiff base Robson-type macrocycles containing two bridging phenol groups have been widely used to synthesize homo- and heterodinuclear complexes for many years. Detailed investigation of the chemistry of these systems has enabled insights in a number of areas, such as magnetochemistry, site selection, and bioinorganic model chemistry.[1] Expansion to larger macrocycles accommodating polynuclear assemblies has been achieved in the last ten years.[2,3] Interest in such complexes is centered on their ability to hold a number of metal ions in a fixed geometric relationship and has potential relevance in bioinorganic and catalytic chemistry as well as nanotechnological applications.

The macrocycle H_4L1 can form mono- and dinuclear complexes, but is unable to use the pendant alcohol groups to bind more than two metal ions within the macrocycle because of geometric constraints imposed by the overall cavity size.[4] The longer saturated chain in H_4L2 permits formation of homo- and heterotetranuclear complexes, as well as complexes in which some sites are vacant.[3]

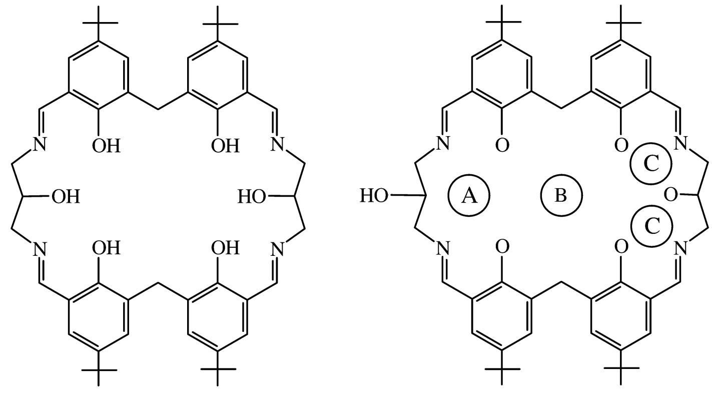


H_4L1



H_4L2

We have now synthesized the new dialdehyde dihydroxy-5,5′-di-*tert*-butyl-3,3′-methanediyldibenzaldehyde (dhtmb)[5] and used this group to expand the H_4L1 macrocycle. Schiff base condensation of dhtmb with 1,3-diaminopropan-2-ol in the presence of metal template ions yields complexes of the new macrocycle H_6L3 (Scheme 1). This ligand can be considered as an expanded calix[4]arene containing two $=NCH_2CH(OH)CH_2N=$ inserts. Unlike most aza- or oxo-calixarenes (where inserts are placed between each pair of phenol rings),[6] H_6L3 preserves two methylene linkages while introducing imine and pendant alcohol donors.



H_6L3 Binding sites

Scheme 1. Pseudocalixarene ligand and binding sites.

Approximately planar conformations are sterically prohibited in genuine calix[n]arenes (where $n = 4$–6) and metal ions cannot take advantage of the bridging potential of the phenol groups, which generally leads to mononuclear complexes.[6,7] In contrast, H_6L3 can use some or all of the phenolic oxygen donors in the bridging mode and can be di- or polynucleating. The presence of relatively soft imine donors in addition to the phenolic oxygen atoms also widens the range of metal ions that might be expected to bind with high stability constants.

Three types of metal binding site are potentially available within H_6L3 (Scheme 1): site A is analogous to the conventional "Robson" site and is similar to that observed for complexes of H_4L1;[4] there are two of these in the macrocycle.

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