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Tethering strategies in alkynylamino carbene complexes

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Dedicated to Professor Jose Barluenga on the occasion of his 60th birthday

Abstract

During the synthesis of complex cyclic macromolecular structures, the reaction of different mono- di- and triamino alkynyl metal carbene systems with primary and secondary amines was studied. The results showed the low stability of the metal carbene complexes obtained from the conjugate addition of primary amines in contrast to the fairly good stability of the corresponding complexes obtained from the addition of secondary amines.

Keywords: Aminocarbene complexes; Nucleophilic additions; Macromolecules

1. Introduction

Our current interest in polar acetylenic systems has focused, for some time, on alkynyl alkoxy carbene complexes, and different studies related to cycloadditions using these carbene complexes have been undertaken by us [1]. In another line, we have studied the reactivity of these complexes towards different nucleophiles [2] such as, alcohols, phenols, thiols and diphenyl phosphine [3]. Also, Fischer and Aumann reported the reactivity of the same systems towards carbanions [4] and phosphines [5].

The presence of the metal carbonyl moiety enhances the inherent reactivity of the triple bond and, therefore, the addition of nucleophiles occurs under very mild reaction conditions. The early work by Fischer on the reaction of primary and secondary amines with alkynyl alkoxy metal carbenes [6] revealed the importance of the kinetic versus thermodynamic control in this reaction and the influence brought about by the steric and electronic effects in the regio (1-substitution versus 3-addition) and stereochemistry of the resulting product mixture. A very extensive and complete work related to this subject has been reported by Aumann [7], pointing out the importance of the amine used either in the regio and stereochemistry of the resulting adducts. He also reported that when the reaction takes place at room temperature the only product obtained corresponds to that from 3-addition giving, usually, the *E* isomer for secondary and the *Z* isomer for primary amines. In a related work, de Meijere [8] studied the influence of the substitution of the triple bond on the stereochemistry of the final adducts.

Recently, we have studied the reaction of bidentate nucleophiles (amidines [9] and diamines [10]) with the alkynyl alkoxy metal carbene complexes. The results obtained in these studies, prompted us to explore more deeply the possibilities of these systems towards the synthesis of macrocyclic rings or macromolecular assemblies having metal carbene moieties. One of the possibilities consists of the reaction of amines with the aminocarbene alkynyl complexes. However, in a literature search, very few reports appear related to this subject. In fact, to our knowledge, after the report of Fischer on the substitution/addition of dimethylamine on the alkynyl alkoxy carbenes, under different temperature conditions, scarce information appears related to these compounds. Dötz et al. [11] and Fischer [12] reported the synthesis of related systems by using different synthetic pathways (alkynyl insertion).

We decided then to undertake a study related to the addition of primary and secondary amines to different mono, di and tri amino alkynyl metal carbene com-

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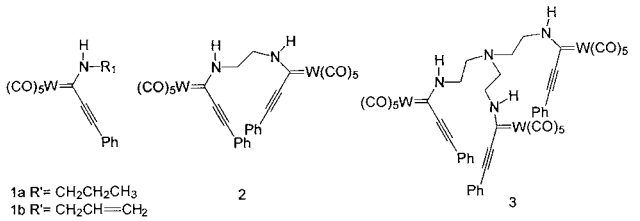


Fig. 1. Amino carbene complexes **1**–**3**.

plexes (Fig. 1), with the purpose of obtaining compounds by bearing, either, two nitrogen atoms with a different reactivity pattern (possible access to hemi labile ligands), or more complex structures with possible further application to the synthesis of macromolecules with metal carbene moieties.

While complexes **1** and **2** have been previously described [13] complex **3** was obtained by reaction of the pentacarbonyl[(ethoxy)phenylethynyl]tungsten carbene complex with the tris (2-aminoethyl)amine at -78°C in a 0.3 M solution in dry THF. The reaction proceeded in a stereoselective way affording as the major product (more than 95% on the crude mixture) the one having the *E*,*E*,*E* stereochemistry for the three carbene carbon nitrogen bonds. This result is in close agreement with that previously described from the reaction of primary amines with alkynyl alkoxy carbene complexes [14].

2. Reaction of complexes 1, 2 and 3 with secondary amines

The results obtained in the reaction of secondary amines with complexes **1**, **2** and **3** are described in Table 1.

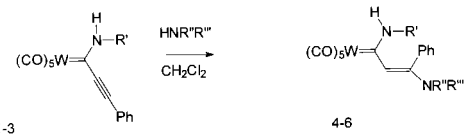
In all the cases, the addition was regio and stereoselective affording only the *E* isomer in accordance with the results reported by Aumann for the addition of similar amines on alkynyl alkoxy systems. To confirm this assumption, we have performed the nOe experiment of the complex **4a** and the results obtained are shown in the Fig. 2.

In the present cases, the reaction time is longer than that described for the addition of the same amines on the corresponding alkynyl alkoxy metal carbene complexes. This is in good agreement with the decrease in reactivity expected for the amino carbene systems, related to the oxy analogs.

We have also studied the reaction of the carbene complex **1b** with *N*,*N*′-dimethylethylenediamine. The reaction is shown in Scheme 1 and the corresponding dicarbene complex **7** was obtained in moderate yield.

The study of the ring closing metathesis (RCM) to afford the expected 15-member macrocycle is now in progress [15].

Table 1
Reaction of secondary amines with amino carbene complexes **1**–**3**



		Amine	Product (% yield) [a]
1	**1a**	R″=R‴=allyl	**4a** (67)
2	**1a**	R″,R‴=$(CH_2)_5$	**4b** (72)
3	**1b**	R″=R‴=allyl	**4c** (70)
4	**1b**	R″=Me, R‴=allyl	**4d** (65)
5	**2**	R″=R‴=allyl	**5a** (75)
6	**2**	R″=Me, R‴=allyl	**5b** (80)
7	**3**	R″=R‴=allyl	**6a** (60)
8	**3**	R″,R‴=$(CH_2)_5$	**6b** (50)

[a] Reaction conditions: 0.1 M solution of the corresponding carbene in CH_2Cl_2, one equivalent of the corresponding amine; room temperature; time: 18 h.

3. Reaction of complexes 1 and 2 with primary amines

The reaction of the corresponding alkynyl amino mono and dicarbenes with primary amines took a completely different path. In all the cases studied we were not able to obtain the expected addition product, but a mixture of compounds. This mixture changed according to the elapsed reaction time, but remained unaffected by changes in solvents (from THF to CH_2Cl_2). We tried the reaction with the amino carbenes **1a**, **1b** and **2**. In all cases the results were found the to be same: decomposition of the original carbene moiety to give instead a isocyanide complex of type **8**.

As an illustrative example, we show the case of addition of allylamine to complex **1b**. Although the expected addition product was detected in the reaction mixture after 1 h of reaction (a vinylic signal at ~ 6.0

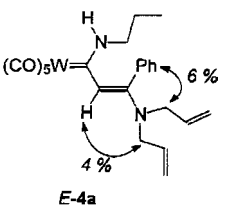
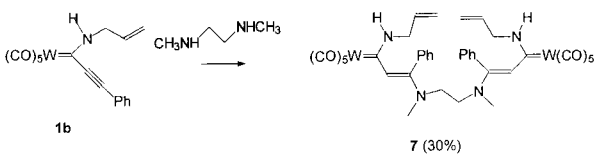


Fig. 2. Results of the nuclear overhauser experiment with E-**ua**.



Scheme 1. Reaction of *N*,*N*′-dimethylethylenediamine with complex **1b**.

ppm was observed in the ^{1}H-NMR spectrum), longer reaction times afforded, as major products, the ones corresponding to the structures **8** and **9**. The formation of these products could be accounted for by a 1,3-proton shift from the amines as shown in Scheme 2.

All attempts to isolate the addition product in the early stages of the reaction failed and, from the chromatographic separation, we were able to obtain only the isonitrile pentacarbonyl metal moiety **8** and acetophenone **9**, albeit in very low yields.

This pattern presents some similarities with the ones proposed by Aumann [16] and de Meijere [17] in the case of addition of hydrazines and primary amines on alkynyl alkoxy carbene systems.

4. Conclusions

We present here a piece of work related to the reactivity and possibilities of the alkynyl amino carbene systems, on the way to the construction of more elaborate molecules and macromolecules. We have shown that the reactivity of these complexes towards amines depends mainly on the use of primary or secondary amines and, in the second case, new fairly stable and easy to handle complexes were obtained. We also show that primary amines did not give the expected compounds. A mechanistic proposal is shown to explain this abnormal behaviour. Further work related to this subject is in progress in our laboratory.

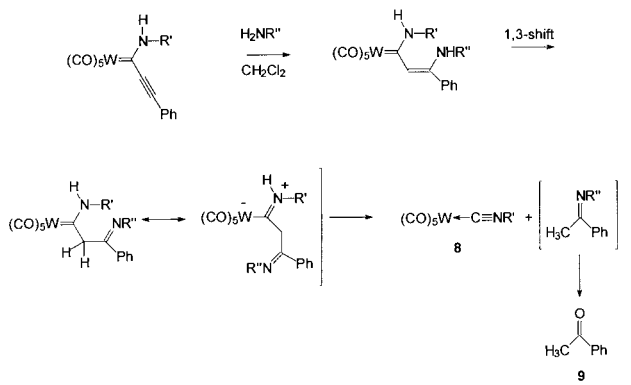
5. Experimental

Unless otherwise stated all common reagents and solvents were used as obtained from commercial suppliers without further purification.

NMR spectra were recorded on a Varian Gemini-200 (200 MHz for ^{1}H-NMR and 50 MHz for ^{13}C-NMR) or a Varian XL-300 apparatus (300 MHz for ^{1}H-NMR and 75.4 MHz for ^{13}C-NMR). All samples of carbene complexes were filtered through a pad of Celite and EDTA prior to recording the spectra. IR spectra were recorded on a Bomem FTIR M-120 spectrophotometer. Mass spectra were obtained on an AutoSpec-Q mass spectrometer. Elemental analyses were performed using a Carlo Erba 1106 apparatus.

Flash column chromatography was performed with 'flash grade' silica (SDS 230–400 mesh).

Unless otherwise indicated all the reactions were performed under Ar atmosphere. Carbene complexes **1a**,**b** [13], and **2** [10] were prepared according to literature procedures.



Scheme 2. Reaction of allylamine with complex **1b**.

5.1. Synthesis of triamino carbene complex *3*

To a stirred solution of 0.482 g of pentacarbonyl[(ethoxy)phenylethynyl]tungsten carbene complex (1 mmol) in 4 ml of dry THF at -78°C, 0.438 g of the tris (2-aminoethyl)amine in (3 mmol) and 2 ml of THF were added dropwise. The disappearance of the starting compound was complete after 5 min of reaction (TLC). The resulting mixture was evaporated and purification by flash chromatography (hexane–CH_2Cl_2, 1:1) afforded 0.872 g of the compound **3** as an orange solid (60% yield).

5.1.1. Compound *3*

IR ($CHCl_3$) : 2167, 2063, 1978, 1942 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 3.05 (t, $J = 6.6$ Hz, 6H, CH_2); 3.84 (q, $J = 6.6$Hz, 6H, CH_2); 7.35–7.55 (m, 15H, Ph); 8.89 (bs, 3H, N*H*). ^{13}C-NMR ($CDCl_3$): δ 50.1 (t); 53.2 (t); 91.4 (s); 120.9 (s); 128.9 (d); 129.1 (s); 131.2 (d); 132.2 (d); 198.3 (s); 203.5 (s); 234.4 (C=W). MS (FAB$^+$, matrix NBA) 1454 (M^+), 1342, 1314, 1286, 1258, 1034, 850. Anal. Calc. for $C_{48}H_{30}N_4O_{15}W_3$: C, 39.61; H, 2.06; N, 3.85. Found: C, 39.39; H, 2.02; N, 3.87%.

5.2. General procedure for the reaction with secondary amines

To a stirred 0.1 M solution of the carbene in CH_2Cl_2 at room temperature (r.t.), one equivalent of the amine was added. The reaction course was monitored by TLC. After the starting carbene complex had completely disappeared, the solvent was removed and the residue passed through a flash chromatography column.

5.3. Synthesis of compound *4a*

The reaction was performed using 0.247 g (0.5 mmol) of **1a** and 0.049 g (0.5 mmol) of the diallyl amine in 4 ml of CH_2Cl_2. After purification by flash chromatography (hexane–CH_2Cl_2, 1:1), 0.198 g of complex **4a** was obtained as a yellow solid (67% yield).

*5.3.1. Compound **4a***

IR ($CHCl_3$): 2063, 1978, 1942 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 0.65 (t, $J = 10.8$ Hz, 3H, CH_3); 0.94 (dq, $J = 10.2$, 10.8 Hz, 2H, CH_2); 3.27 (dt, $J = 10.2$, 10.8 Hz, 2H, CH_2); 3.67 (d, $J = 7.8$ Hz, 2H, CH_2); 5.10–5.27 (m, 2H, CH_2); 5.64–5.89 (m, 1H, C*H*); 6.24 (s, 1H, C*H*); 7.01 (bs, 1H, N*H*); 7.20–7.50 (m, 5H, Ph. ^{13}C-NMR ($CDCl_3$): δ 11.0 (q); 22.0 (t); 51.6 (t); 56.3 (t); 117.9 (t); 118.5 (d); 129.3 (d); 129.4 (d); 129.5 (d); 132.5 (d); 135.2 (s); 147.4 (s); 199.5 (s); 204.3 (s); 237.1 (C=W).

*5.4. Synthesis of compound **4b***

The reaction was performed using 0.250 g (0.5 mmol) of **1a** and pyperidine 0.043 g (0.5 mmol) in 4 ml of CH_2Cl_2. After purification by flash chromatography, 0.208 g of complex **4b** was obtained as a yellow solid. (72%).

*5.4.1. Compound **4b***

IR ($CHCl_3$): 2069, 1984, 1942 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 0.60 (t, $J = 10.8$ Hz, 3H, CH_3); 0.90–1.05 (m, 2H, CH_2); 1.60 (bs, 6H, CH_2); 2.95 (bs, 4H, CH_2); 3.2 (m, 2H, CH_2); 6.12 (s, 1H, C*H*); 7.11 (bs, 1H, NH); 7.20–7.50 (m, 5H, Ph).

*5.5. Synthesis of compound **4c***

The reaction was performed using 0.250 g (0.5 mmol) of **1b** and 0.050 g (0.5 mmol) of diallyl amine in 4 ml of CH_2Cl_2. After purification by flash chromatography (CH_2Cl_2), 0.206 g of complex **4c** was obtained as a yellow solid (70% yield).

*5.5.1. Compound **4c***

IR ($CHCl_3$): 2055, 1932, 1912 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 3.69 (d, $J = 4.5$ Hz, 4H, CH_2); 3.92 (d, $J = 6$ Hz, 2H, CH_2); 5.00–5.27 (m, 6H, CH_2); 5.70–5.80 (m, 3H, C*H*); 6.27 (s, 1H, C*H*); 6.92 (bs, 1H, NH); 7.20–7.50 (m, 5H, Ph). ^{13}C-NMR ($CDCl_3$): δ 51.7 (t); 57.4 (t); 117.9 (t); 118.3 (d); 118.9 (t); 129.2 (d); 129.3 (d); 129.5 (d); 131.9 (d); 132.5 (d); 135.0 (s); 148.0 (s); 199.4 (s); 204.2 (s); 238.0 (C=W).

*5.6. Synthesis of compound **4d***

The reaction was performed using 0.250 g (0.5 mmol) of **1b** and 0.040 g (0.5 mmol) of methyl allyl amine in 4 ml of CH_2Cl_2. After purification by flash chromatography (hexane–CH_2Cl_2, 1:1), 0.183 g of complex **4d** was obtained as a yellow solid (65%).

*5.6.1. Compound **4d***

IR ($CHCl_3$): 2055, 1978, 1905 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 3.54 (d, $J = 5.4$ Hz, 2H, CH_2); 3.92 (t, $J = 5.7$ Hz, 2H, CH_2); 4.80–5.35 (m, 5H, C*H*, CH_2); 5.69–5.75 (m, 1H, C*H*); 6.16 (s, 1H, C*H*); 6.90 (bs, 1H, N*H*); 7.20–7.50 (m, 5H, Ph). ^{13}C-NMR ($CDCl_3$): δ 37.8 (q); 54.4 (t); 57.3 (t); 117.9 (t); 118.3 (t); 118.9 (d); 129.2 (d); 129.3 (d); 129.4 (d); 131.9 (d); 132.6 (d); 135.0 (s); 148.8 (s); 199.4 (s); 204.2 (s); 238.2 (C=W).

*5.7. Synthesis of compound **5a***

The reaction was performed using 0.466 g (0.5 mmol) of **2** and 0.097 g (1 mmol) of the diallyl amine in 4 ml of CH_2Cl_2. After purification by flash chromatography (hexane–CH_2Cl_2, 1:2), 0.422 g of complex **5a** was obtained as a yellow solid (75%).

*5.7.1. Compound **5a***

IR ($CHCl_3$): 2055, 1953, 1897 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.85 (bs, 4H, CH_2); 3.68 (bs, 8H, CH_2); 5.10 (d, $J = 10.8$ Hz, 4H, CH_2); 5.30 (d, $J = 17$ Hz, 4H, CH_2); 5.70 (ddd, $J = 6.9$, 10.8, 17 Hz, 4H, C*H*); 6.23 (s, 2H, C*H*); 6.90 (bs, 2H, N*H*); 7.10–7.20 (m, 4H, Ph); 7.40–7.60 (m, 6H, Ph). ^{13}C-NMR ($CDCl_3$): δ 51.6 (t); 52.7 (t); 117.8 (d); 118.0 (t); 128.8 (d); 129.6 (d); 130.2 (d); 132.2 (d); 134.5 (s); 148.9 (s); 199.4 (s); 203.8 (s); 238.8 (C=W).

*5.8. Synthesis of compound **5b***

The reaction was performed using 0.466 g (0.5 mmol) of **2** and 0.071 g (1 mmol) of allyl methyl amine in 4 ml of CH_2Cl_2. After purification by flash chromatography (hexane–CH_2Cl_2, 1:2), 0.430 g of complex **5b** was obtained as a yellow solid (80%).

*5.8.1. Compound **5b***

IR ($CHCl_3$): 2053, 1955, 1907 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 2.61 (s, 6H, CH_3); 3.00 (bs, 4H, CH_2); 3.91 (t, $J = 6$ Hz, 4H CH_2); 4.95 (dd, $J = 10.6$, 17 Hz, 4H, CH_2); 5.21 (ddd, $J = 6$, 10.6, 17 Hz, 4H, C*H*); 6.04 (s, 2H, C*H*); 6.29 (bs, 2H, N*H*); 7.10–7.20 (m, 4H, Ph); 7.40–7.60 (m, 6H, Ph). ^{13}C-NMR ($CDCl_3$): δ 38.4 (t); 48.9 (t); 67.3 (t); 118.7 (d); 119.1 (t); 129.2 (d); 129.5 (d); 131.6 (d); 134.5 (d); 147.6 (s); 199.3 (s); 203.9 (s); 238.8 (C=W).

*5.9. Synthesis of compound **6a***

The reaction was performed using 0.485 g (0.3 mmol) of **3** and 0.088 g (0.9 mmol) of the diallyl amine in 4 ml of CH_2Cl_2. After purification by flash chromatography (CH_2Cl_2), 0.307 g of complex **6a** was obtained as a yellow solid (60%).

*5.9.1. Compound **6a***

IR ($CHCl_3$): 2055, 1955, 1907 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.72 (bt, 6H, CH_2); 3.13 (bq, 6H, CH_2); 3.70 (bs, 12H, CH_2); 5.25 (d, $J = 17.4$Hz, 6H, CH_2); 5.27 (d,

$J = 10.2$ Hz, 6H, CH_2); 5.70 (ddd, $J = 6$, 10.2, 17.4 Hz, 6H, C*H*); 6.25 (s, 3H, C*H*); 6.82 (bs, 3H, N*H*); 7.10–7.30 (m, 6H, Ph); 7.40–7.60 (m, 9H, Ph). ^{13}C-NMR ($CDCl_3$): δ 51.0 (t); 51.2 (t); 51.7 (t); 118.0 (s); 118.4 (t); 129.3 (d); 129.4 (d); 129.5 (d); 132.3 (d); 135.1 (s); 148.2 (s); 199.6 (s); 203.9 (s); 238.1 (C=W).

5.10. Synthesis of compound **6b**

The reaction was performed using 0.485 g (0.3 mmol) of **3** and 0.064 g (0.9 mmol) of pyperidine in 4 ml of CH_2Cl_2. After purification by flash chromatography (CH_2Cl_2), 0.252 g of complex **6b** was obtained as a yellow solid (50%).

5.10.1. Compound **6b**

IR ($CHCl_3$): 2067, 1947, 1935 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 1.62 (bs, 24h, CH_2); 1.72 (bt, 6H, CH_2); 3.02 (bs, 12H, CH_2); 3.13 (bq, 6H, CH_2); 6.13 (s, 3H, C*H*); 6.87 (bs, 3H, N*H*); 7.10–7.30 (m, 6H, Ph); 7.40–7.60 (m, 9H, Ph). ^{13}C-NMR ($CDCl_3$): δ 24.2 (t) 25.4 (t), 49.4 (t); 50.9 (t); 119.74 (d); 129.2 (d); 129.6 (d); 135.6 (s); 149.5 (s); 199.5 (s); 203.9 (s); 238.9 (C=W).

5.11. Synthesis of compound **7**

The reaction was performed using 0.250 g (0.5 mmol) of **1b** and 0.022 g (0.25 mmol) of *N*,*N*′-dimethylethylenediamine in 4 ml of CH_2Cl_2. After purification by flash chromatography (hexane–CH_2Cl_2, 1:1), 0.175 g of complex **7** was obtained as an orange solid (30%).

5.11.1. Compound **7**

IR ($CHCl_3$): 2051, 1955, 1887 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 3.02 (s, 6H, CH_3); 3.00 (bs, 4H, CH_2); 3.70 (bs, 4H, CH_2); 4.88 (d, $J = 18$Hz, 2H, CH_2); 4.97 (d, $J = 10.2$Hz, 2H, CH_2); 5.21 (ddd, $J = 6$, 10.2, 18 Hz, 2H, C*H*); 6.04 (s, 2H, C*H*); 6.87 (bs, 2H, N*H*); 7.10–7.30 (m, 4H, Ph); 7.40–7.60 (m, 6H, Ph). ^{13}C-NMR ($CDCl_3$): δ 38.4 (q) 48.9 (t); 57.3 (t); 118.7 (d); 119.1 (t); 129.2 (d); 129.5 (d); 131.6 (d); 134.5 (s); 147.6 (s); 199.3 (s); 203.9 (s); 238.8 (C=W).

5.12. Reaction with primary amines. Reaction of **1b** with allyl amine

The reaction was performed using 0.350 g (0.7 mmol) of **1b** and 0.040 g (0.7 mmol) of allylamine in 2 ml of CH_2Cl_2 at r.t. After purification by flash chromatography (hexane–CH_2Cl_2, 1:1), 0.040 g of complex **8a** (15% yield) and 0.020 g of acetophenone **9** (24% yield) were obtained.

5.12.1. Compound **8a**

IR ($CHCl_3$): 2178, 2069, 1950 cm^{-1}. ^{1}H-NMR ($CDCl_3$): δ 4.33 (bs, 2H, CH_2); 5.30–5.50 (m, 2H, CH_2); 5.70–6.00 (m, 1H, C*H*). ^{13}C-NMR ($CDCl_3$): δ 46.5 (t); 118.5 (t); 128.0 (d); 194.2 (s); 196.2 (s); (NC) not found.

Acknowledgements

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