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Journal of Molecular Catalysis A: Chemical 110 (1996) 227–233

JOURNAL OF MOLECULAR CATALYSIS A: CHEMICAL

# Effect of a proximal substituent on the triruthenium dodecacarbonyl-catalyzed reductive carbonylation of nitroarenes

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Received 26 February 1996; accepted 17 May 1996

**Abstract**

Three nitroarenes were submitted to $Ru_3(CO)_{12}$-catalyzed reductive carbonylation in acetonitrile and in *cis*-cyclooctene. The main reaction products were the corresponding amines, ureas and six- or five-membered cyclization products. Optimization of the reaction varying the temperature, the CO pressure, the catalyst/substrate ratio and the reaction time and a statistical analysis of conversion and selectivity data allow to suggest a reaction mechanism in some reaction conditions.

*Keywords:* Carbon monoxide; Carbonylation; Nitroarenes; Ruthenium

## 1. Introduction

The reduction of aromatic nitroderivatives with triethylphosphite and the thermal or photochemical decomposition of aromatic azides are methods of choice for the preparation of a wide variety of heterocyclic compounds [1]. These reactions occur through the intermediate formation of aryl nitrenes [2]. The organometallic analogue of this behavior is the reactivity of metal arylnitrene complexes [3].

The ruthenium(0)-catalyzed reductive carbonylation of *para*-nitroarenes was used by us and others [4,5] for the synthesis of symmetrical ureas. Carbamates were obtained performing the reaction in alcohols. In low conversion conditions, i.e., in the reductive carbonylation of nitrobenzene, the bis-nitrene complex $Ru_3(CO)_8(NPh)_2$ was detected in solution and was suggested to be the intermediate in the formation of diphenylurea [5]. However, recent results seem to exclude the presence of nitrene-cluster species as intermediates in this reaction [6]. Cyclization to indoles [7], imidazoles [8], triazoles [9], carbazole [10], and quinolones [11] was achieved by Ru(0)-catalyzed reductive carbonylation of 2-nitrostilbenes, 2-nitrobenzylideneanilines, 2-nitrophenylazo compounds, 2-nitrobiphenyl, and 2-nitrochalcones, respectively.

Co-catalysts may be used in this reaction. The use of alkali halides allowed to have higher cyclization yields of *o*-nitrobiphenyl to carbazole [12]. The formation of carbamates was obtained by the reductive carbonylation in the presence of montmorillonite-bipyridinyl-palladium(II) acetate [13].

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*PII* S1381-1169(96)00198-7

These reactions are generally more efficient than the deoxygenation with triethylphosphite [14]. The intermediacy of a ruthenium–nitrene complex in the insertion into the aromatic carbon–hydrogen bond was suggested by the isolation of $Ru_3(\mu_3NC_6H_4\text{-}o\text{-}C_6H_5)_2(CO)_9$ in the reductive carbonylation of *ortho*-nitrobiphenyl. This organoruthenium compound was shown to give carbazole on heating [10]. Also force field calculations suggested that cyclization occurred if the nitrenic nitrogen and the aromatic carbon–hydrogen bond were in spatial proximity [15].

In the aim of testing the general validity of the cyclization reaction [16] and to study the interactions between the nitro group to be reduced and one adjacent group, we report here the results of the $Ru_3(CO)_{12}$-catalyzed reductive carbonylation of *ortho*-nitrobiarenes and aromatic carbonyl compounds in two different solvents: acetonitrile and *cis*-cyclooctene.

## 2. Results

The three compounds we submitted to reaction were two *ortho*-substituted nitroderivatives: 1,2-dinitrobenzene (**1**) and 2-nitrobenzaldehyde (**11**) and one dinitrobiarene: 2,2'-dinitrobiphenyl (**16**).

The interaction of the reductive carbonylation of a nitro group with an adjacent nitro group was tested using 1,2-dinitrobenzene (**1**) as the substrate. The reaction was performed at 220°C for 5 h using a substrate to catalyst molar ratio of 50:1 and a partial pressure of CO of 60 bar. Compound (**1**) gave in acetonitrile (reaction a) (Table 1) the diamine (**2**) as the main reaction product, deriving from the reduction of the two nitro groups. Several other reaction products derived from the insertion of acetonitrile and eventually of carbon monoxide to give 5- and 6-membered cyclization products. They were benzimidazole (**3**), 2-methylbenzimidazole (**4**), 2-ethylbenzimidazole (**5**) and 2,3-dimethylquinoxaline (**6**) (Table 1). In *cis*-cyclooctene (reaction b) the reductive carbonylation of compound (**1**) gave essentially the diamine (**2**), and a small amount of the nitrosamine (**7**).

Since some of these cyclization products could derive from acetamides formed by insertion of an intermediate into the carbon–nitrogen triple bond of acetonitrile [14], also 2-aminoacetanilide (**8**) and diacetyl-*ortho*-phenyl-

Table 1
Reaction yields in the Ru(0)-catalyzed reductive carbonylation of aromatic nitroderivatives

| Reaction | Substrate | Solvent | Reduction | Insertion | | Cyclization | | Other |
|---|---|---|---|---|---|---|---|---|
| | | | Amine | Acetamide | Carboxamide | 5-Membered | 6-Membered | |
| a | **1** | ACN | **2**:33 | | | **3**:7 | **6**:8 | |
| | | | | | | **4**:20 | | |
| | | | | | | **5**:tr | | |
| b | **1** | CYC | **2**:53 | | | | | **7**:3 |
| c | **8** | ACN | | | | **4**:65 | | **9**:3 |
| | | | | | | **10**:3 | | |
| d | **9** | ACN | | | | **4**:70 | | |
| e | **11** | ACN | **12**:74 | | | **13**:1 | | |
| | | | | | | **14**:1 | | |
| f | **11** | CYC | **12**:60 | | **15**:30 | | | |
| g | **16** | ACN | **17**:64 | **18**:7 | **19**:7 | **20**:13 | | |
| | | | | | | **21**:8 | | |
| h | **16** | CYC | **17**:89 | | | **20**:9 | | |
| | | | | | | **22**:1 | | |

tr = traces.
Reaction conditions: $p_{CO}$ = 60 bar; $T$ = 220°C; $r$ = 50; $t$ = 5 h.

enediamine (**9**) were submitted to the Ru-catalyzed reductive carbonylation. That this was the case resulted from the fact that compound (**8**) gave in acetonitrile (reaction c) 2-methylbenzimidazole (**4**) in high yields, and small amounts of benzimidazol-2-one (**10**) and the diacetylderivative (**9**). Compound (**9**) gave (reaction d) only 2-methylbenzimidazole (**4**) in high yields.

The interaction of the intermediates in the reductive carbonylation of a nitro group with an adjacent carbonyl group was tested using 2-nitrobenzaldehyde (**11**) as the substrate. This compound gave in acetonitrile (reaction e) essentially 2-aminobenzaldehyde (**12**), deriving from the reduction of the nitro group. Also small amounts of compounds deriving from insertion into the triple bond of the acetonitrile molecule were formed. They were quinoline (**13**) and quinoxaline (**14**). In *cis*-cyclooctene compound (**11**) gave (reaction f) 2-aminobenzaldehyde (**12**) and the cyclooctanecarboxamide (**15**). The latter derived from the insertion of an intermediate into the carbon–carbon double bond of *cis*-cyclooctene.

The interaction of the reductive carbonylation of a nitro group with a proximal nitro group was tested using 2,2′-dinitrobiphenyl (**16**) as the substrate. This compound had been also studied in preceding papers [11,14,16]. In acetonitrile (reaction g) the diamine (**17**) was the main reaction product. Carbon monoxide insertion gave the formamide (**18**) and insertion into acetonitrile gave the acetamide **19**. Minor amounts of benzocinnoline (**20**) and carbazole (**21**) were also found. In *cis*-cyclooctene (reaction h) the diamine (**17**) was again formed, together with two cyclization products: benzocinnoline (**20**) and the cyclic urea (**22**).

In order to have a better insight into the reaction mechanism of the Ru(0)-catalyzed reductive carbonylation, the reaction with 2,2′-dinitrobiphenyl (**16**) in *cis*-cyclooctene was optimized changing the reaction temperature ($T$, °C), the pressure of carbon monoxide ($p_{CO}$, bar), the molar catalytic ratio ($r$), the reaction time ($t$, s). The influence of changing these parameters on the conversion of compound (**16**) is shown in Table 2.

Table 2
The influence of reaction temperature ($T$, °C), the pressure of carbon monoxide ($p_{CO}$, bar), the catalytic ratio ($r$), the reaction time ($t$, s) on the conversion of 2,2′-dinitrobiphenyl (**16**) in the Ru(0)-catalyzed reductive carbonylation

| Run | $T$ (°C) | $p_{CO}$ (bar) | $t$ (s) | $r$ | Conversion (%) |
|---|---|---|---|---|---|
| 1 | 200 | 30 | 1877 | 1.25 | 82 |
| 2 | 190 | 15 | 1878 | 1.40 | 72 |
| 3 | 200 | 15 | 1880 | 1.25 | 97 |
| 4 | 190 | 15 | 1893 | 1.15 | 87 |
| 5 | 190 | 15 | 1883 | 1.25 | 55 |
| 6 | 210 | 15 | 1882 | 1.25 | 55 |
| 7 | 200 | 15 | 1881 | 1.40 | 53 |
| 8 | 210 | 15 | 1890 | 1.15 | 61 |
| 9 | 210 | 15 | 1892 | 1.40 | 54 |
| 10 | 200 | 15 | 1879 | 1.15 | 53 |
| 11 | 190 | 30 | 1888 | 1.25 | 49 |
| 12 | 190 | 30 | 1878 | 1.40 | 34 |
| 13 | 190 | 50 | 1888 | 1.25 | 50 |
| 14 | 210 | 50 | 1887 | 1.25 | 75 |

Also the selectivity in the main reaction products was determined, and is shown in Table 3.

Conversion ranged from 34% to 97%. Selectivity in the diamine (**17**) was in the range 8% to 81%, in the formamide (**18**) was 1–28%; in

Table 3
The influence of reaction temperature ($T$, °C), the pressure of carbon monoxide ($p_{CO}$, bar), the catalytic ratio ($r$), the reaction time ($t$, s) on the selectivity in product formation from 2,2′-dinitrobiphenyl (**16**) in the Ru(0)-catalyzed reductive carbonylation

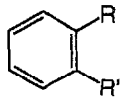
| Run | Diamine (**17**) | Formamide (**18**) | Benzocinnoline (**20**) | Carbazole (**21**) | Urea (**22**) |
|---|---|---|---|---|---|
| 1 | 52 | 10 | 10 | | 4 |
| 2 | 49 | 28 | 10 | | 5 |
| 3 | 81 | 5 | 6 | 5 | 1 |
| 4 | 40 | 14 | 7 | 1 | 24 |
| 5 | 19 | 5 | 16 | 1 | 9 |
| 6 | 8 | 10 | 15 | | 17 |
| 7 | 25 | 8 | 15 | | |
| 8 | 18 | 16 | 17 | 1 | 10 |
| 9 | 32 | 6 | 13 | 1 | 1 |
| 10 | 12 | 4 | 9 | 1 | 16 |
| 11 | 27 | 1 | 15 | 1 | 3 |
| 12 | 10 | 1 | 21 | | |
| 13 | 16 | 6 | 28 | | |
| 14 | 46 | 7 | 21 | | |

benzocinnoline (**20**) was 6–28%. Carbazole (**21**) was only occasionally present, whereas the urea (**22**) was an important reaction product only in some cases. Thus, the fourteen runs were a representative panel of the influence of the variables involved in the reaction mechanism.

The compounds are shown in Schemes 1 and 2.

## 3. Discussion

Nitroarenes react with $Ru_3(CO)_{12}$ to give nitrene complexes [10,16], presumably via the intermediate formation of the corresponding nitroso compounds [3]. Literature data support the view that the catalytic deoxygenation of nitrosoarenes by carbon monoxide is a faster process [17]. The ruthenium nitrene complexes are



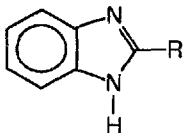
(1): R = R' = $NO_2$

(2): R = R' = $NH_2$

(7): R = $NH_2$ ; R' = $NO_2$

(8): R = $NH_2$ ; R' = $NHCOCH_3$

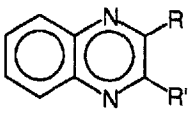
(9): R = R' = $NHCOCH_3$



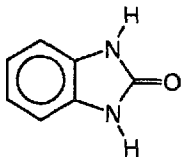
(3): R = H

(4): R = Me

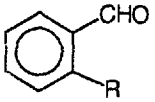
(5): R = Et



(6): R = R' = Me



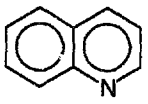
(10)



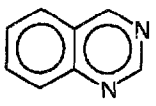
(11): R = $NO_2$

(12): R = $NH_2$

(15): $NHCOC_8H_{15}$

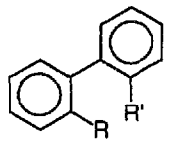


(13)



(14)

Scheme 1.

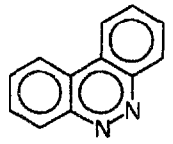


(16): R = R' = $NO_2$

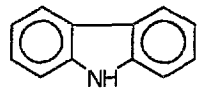
(17): R = R' = $NH_2$

(18): R= $NH_2$; R' = NHCHO

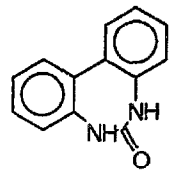
(19): R = $NH_2$; R' = $NHCOCH_3$



(20)



(21)



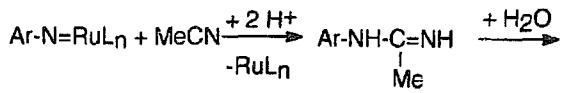
(22)

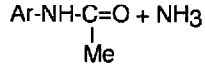
Scheme 2.

key intermediates in the insertion into the aromatic carbon–hydrogen bond [10]. However, in the reaction conditions used in this paper the most important pathways are:

(a) The reduction to an amine and carbonylation and reduction to a formamide. Protonolysis of a ruthenium–nitrene intermediate could be the origin of the amine [18].

(b) The formation of acetanilides deriving from the insertion of the ruthenium-bound nitrene into the carbon–nitrogen triple bond of acetonitrile to give an imine and subsequent hydrolysis to an acetamide:




Acetanilides such as (**8**) and (**9**) cyclize to benzimidazole (**3**), 2-methylbenzimidazole (**4**), 2-ethylbenzimidazole (**5**) and 2,3-dimethylquinoxaline (**6**) in a non-Ru-catalyzed reaction.

(c) Insertion into the aromatic carbon–hydrogen bond to give cyclization to five- and to six-membered heterocycles.

Concerning the effect of substituents *ortho* to the nitro group, the pathway deriving from the reduction of only one nitro group is not important. This suggests that the reduction of the second nitro group is faster than that of the first nitro group. Moreover, the aldehydic group is involved in the reaction only in the formation of very low amounts of quinoline (**13**) and quinoxaline (**14**).

In the reductive carbonylation of 2,2'-dinitrobiphenyl (**16**), together with reduction to the diamine (**17**) and carbonylation and reduction to the formamide (**18**), also intramolecular cyclization to benzocinnoline (**20**), to carbazole (**21**) and to the cyclic urea (**22**) occurred. Yields in the urea ranged from 12% of the converted substrate (run 3) to 63% of the converted substrate (run 6).

The optimization of the reductive carbonylation of compound (**16**) was performed varying the temperature in the range 190–220°C, carbon monoxide pressure in the range 15–50 bar, the catalyst to substrate ratio in the range 1:15–1:40 and observing the influence of these variables on the conversion and the selectivity in compounds (**17**), (**18**) and (**20**). Table 4 shows the correlation matrix thus obtained. The numbers shown in Table 4 indicate the degree of correlation between the evolution of a parameter (e.g. the conversion) and a given set of reaction conditions. The correlation per cent obtained may give information about some mechanistic details.

The conversion of compound (**16**) has 87.4% correlation with the selectivity in the most important reaction product: the diamine (**17**). This suggests the homogeneity of the data (no casual errors).

The influence of the reaction conditions on the conversion of compound (**16**) showed correlation for runs 1, 2, 3, 11, 13 with a $r^2\% = 82.3$ and $r^2_{CV}\% = 55.7$ (CV = crossvalidated). Four of these runs (1, 3, 11, 13) have in common the catalytic ratio $r = 1.25$. The maximum range of $p_{CO}$ (15–50 bar) and $T$ (190–210°C) is represented in these runs. This suggests that these runs have a common mechanism where the catalytic ratio ($r$) plays an important role.

Concerning the selectivity in the reaction products, the selectivity in the amine (**17**) correlates with the parameters of the reaction with $r^2_{CV}\% = 44.9$ only for runs 1, 2, 3, 11, 13. This value was not modified ($r^2_{CV}\% = 43.5$) if the selectivity in compounds (**18**) and (**20**) was added to the correlation. This suggests that compounds (**17**), (**18**) and (**20**) derive from a common intermediate.

The selectivity in the formamide (**18**) gave $r^2_{CV}\% = 64.1$ by correlation with the parameters of the reaction in runs 1, 2, 3, 11, 13. This suggested that the selectivity in compound (**18**) was essentially determined by the catalytic ratio ($r$).

No information could be obtained from the selectivity in benzocinnoline (**20**).

These data allow to suggest a possible com-

Table 4
Correlation matrix for the reductive carbonylation of compound **16**

| | Conv. | $T$ | $p_{CO}$ | $r$ | $t$ | Sel. **17** | Sel. **20** | Sel. **18** |
|---|---|---|---|---|---|---|---|---|
| Conversion | 1 | 0.121 | −0.12 | 0.264 | 0.012 | 0.874 | 0.419 | −0.611 |
| $T$ | 0.121 | 1 | −0.093 | 0.104 | 0.177 | 0.024 | 0.011 | −0.036 |
| $p_{CO}$ | −0.12 | −0.093 | 1 | −0.154 | 0.118 | −0.021 | −0.312 | 0.725 |
| $r$ | 0.264 | 0.104 | −0.154 | 1 | 0.34 | −0.123 | 0.082 | −0.252 |
| $t$ | 0.012 | 0.177 | 0.118 | 0.34 | 1 | −0.121 | −0.016 | 0.201 |
| Sel. **17** | 0.874 | 0.024 | −0.021 | −0.123 | −0.121 | 1 | 0.268 | −0.536 |
| Sel. **20** | 0.419 | 0.011 | −0.312 | 0.082 | −0.016 | 0.268 | 1 | −0.311 |
| Sel. **18** | −0.611 | −0.036 | 0.725 | −0.252 | 0.201 | −0.563 | −0.311 | 1 |

Conversion: conversion of 2,2'-dinitrobiphenyl (**16**); $T$: temperature, °C; $p_{CO}$: CO pressure, bar; $r$: catalyst: substrate ratio; $t$: time (s). Sel.: selectivity.

mon reaction mechanism for runs 1, 2, 3, 11, 13.

The reductive carbonylation consists in several steps:

(a) A coordinatively unsaturated ruthenium cluster is formed:

$$Ru_3(CO)_{12} \rightleftharpoons Ru_3(CO)_{11} + CO$$

This reaction is favored by the temperature, disfavored by $p_{CO}$, depends from the concentration of the catalyst, but not from the concentration of substrate.

(b) The decomposition of the ruthenium cluster:

$$Ru_3(CO)_{12} + 3CO \rightleftharpoons 3Ru(CO)_5$$

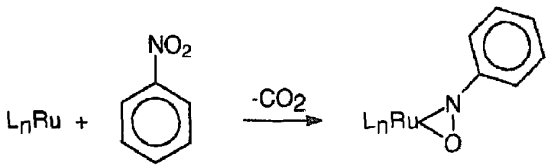
This reaction is disfavored by the temperature, favored by $p_{CO}$ depends on the concentration of the catalyst, but not on the concentration of the substrate.

(c) The formation of a coordinatively unsaturated mononuclear ruthenium species:

$$Ru(CO)_5 \rightleftharpoons Ru(CO)_4 + CO$$

This reaction is favored by the temperature, disfavored by $p_{CO}$, depends from the concentration of the catalyst, but not from the concentration of substrate.

(d) The oxidative addition of the nitroderivative on one of these coordinatively unsaturated species [19] to give a five-membered metallacycle which loses $CO_2$ and forms a coordinated nitroso compound:




This reaction depends from the concentration of both the cluster and the substrate.

Conversion and selectivity in compounds (17) and (18) correlate for different values of $T$, $p_{CO}$ and $t$ for a given value of $r$. This suggests that $r$ is not important in the correlation. Hence, the concentration of reactants (which constitutes $r$) is not important in the correlation. Hence, the generation of a reactive species could be rate determining for runs 1, 2, 3, 11, 13. The conclusion is that these reaction could occur in a region of reactivity dominated by the equilibria which generate the reactive species $Ru_3(CO)_{11}$ and $Ru(CO)_4$. This point have been shown to occur also in the reductive carbonylation of 2-nitrostilbene [20].

## 4. Experimental

*Cis*-cyclooctene, acetonitrile, and $Ru_3(CO)_{12}$ were Merck reagents. Carbon monoxide was a SIO product high purity grade. GLC–MS was performed with a Hewlett Packard Mass Selective Detector 5890 instrument equipped with a SPB-5 30 m column (0.32 mm ID). The samples were injected in split–splitless mode. After 2 min at 40°C a 10°C/min linear gradient was programmed to reach 250°C. The column was kept at 250°C for 23 min. Mass spectra were measured in positive ions electron impact and in positive ions chemical ionization (reactant gas isobutane) mode. Reverse Phase HPLC analyses were performed using a RP-C18 Lichrosorb Merck column (250 × 4 mm), 5 μm particle size and eluting with a 20 min linear gradient from 50% aqueous acetonitrile to 100% acetonitrile (flow rate 0.8 ml/min). The reactions were performed putting the solutions $10^{-5}$ M in the substrate and containing the appropriate amount of catalyst in a 100 ml glass liner. This was placed inside a 250 ml stainless steel autoclave. The air in the autoclave was replaced with dinitrogen by three freeze–pump–thaw cycles before introducing the appropriate amount of carbon monoxide. The autoclave was heated at the required temperature with a thermoregulated oil bath and magnetic stirring was applied. After 5 h, the autoclave was rapidly cooled in an ice bath and blown off. Silica gel chromatography was performed using silica gel Merck

0.05–0.2 mm ($R = 100$) eluting with $CH_2Cl_2$ and $CH_2Cl_2$–ethyl acetate mixtures.

## Acknowledgements

This work was supported by a CNR grant (Piano Finalizzato Chimica Fine II). We thank Dr. Paolo Bossini, Mr. Marco Nardini and Mr. Andrea Cirani for their technical help.

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