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Articles

A New Indirect Application of Aggregative Activation: Synthesis of Esters by Cobalt-Catalyzed Carbonylation of Aryl, Heterocyclic, and Vinyl Halides under Atmospheric Pressure

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Sun lamp illuminated alkoxycarbonylation of aryl, heteroaryl, and vinyl halides was performed under atmospheric pressure of CO in the presence of a cobalt catalyst *in situ* generated from $Co(OAc)_2$. Illumination through a Pyrex flask was sufficient to catalyze the reaction. This process avoids the use of $Co_2(CO)_8$ and excess CH_3I, which were required in the earlier procedure. A $S_{RN}1$ mechanism is proposed.

Introduction

Metal-catalyzed carbonylation of aryl and vinyl halides is a very interesting process in preparative organic chemistry.[1] Among the possible catalysts, cobalt derivatives are particularly attractive. However, while the carbonylation of aryl halides in the presence of cobalt species has been presented in numerous publications,[2] the carbonylations of vinyl halides with such catalysts are rather scarce.[3] Moreover, most reactions performed with aryl halides under atmospheric pressure are carried out in the presence of $Co_2(CO)_8$, an expensive and not very easily handled reagent which, in addition, necessitates the presence of CH_3I in excess,[2-4] an alkylating agent which must be handled with great care on a large scale.

The investigations previously performed in our laboratory on the aggregative activation of NaH[5] led to discovering carbonyl cobalt complex reducing agents NaH−RONa−$Co(OAc)_2$−CO (R = *t*-Am, *neo*-Pent, *t*-Bu, and noted CoCRACO)[6] easily prepared from cobalt acetate and very efficient in the atmospheric pressure catalytic carbonylation of aryl halides. We showed that CoCRACO's contained the catalytic carbonylating reagent $NaCo(CO)_4$ accompanied by a large amount of unknown but necessary cobalt species. In THF or DME and in the presence of the appropriate amount of NaH and RONa, aryl halides were carbonylated into the corresponding aryl esters ArCOOR accompanied by a small amount of the corresponding acid. The mechanism of these carbonylations was found to be an unprecedented $S_{RN}1$ reaction between aryl halides and $[Co(CO)_4]^-$ whose initiating steps were further proposed.[5]

These results led us to next discover the first PTC carbonylations of aryl and vinyl halides in the presence of a catalytic amount of $[Co(CO)_4]^-$ *in situ* generated from $Co_2(CO)_8$.[7a] Of course, NaH could not be used under such conditions and was replaced by light. It is very important to note that such carbonylations do not necessitate special and expensive devices. Indeed, an ordinary nonfluorescent sun lamp (in fact, a simple "tan lamp" found in the supermarket) illuminating the reaction medium and a common Pyrex flask was sufficient. A number of applications of such reactions were then published.[7,8] The main drawbacks of these PTC carbonylations are the use of $Co_2(CO)_8$ as starting material and the formation of acids as the only possible products.

We wondered if a judicious association of the two kinds of reactions could not be a good solution to the preparation of esters without the use of $Co_2(CO)_8$ and CH_3I. In fact, the carbonylation of aryl and vinyl halides into the corresponding esters necessitated the following requirements: (i) CoCRACO usually used in THF should catalyze the carbonylations in the presence of an excess of alcohol. (ii) The activating alkoxide included in the CoCRACO must not compete with the alcohol whose ester is expected. (iii) Since NaH, the radical initiator usually included in CoCRACO, will be destroyed the $S_{RN}1$ alkoxycarbonylation should be photostimulated with a sun lamp illumination through a Pyrex flask. Exploratory experiments showed that the above requirements might be fulfilled.[9] In the present publication we want to report more details on the scope and limitations of these new alkoxycarbonylations.

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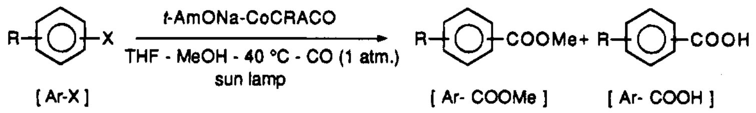
Table 1. Methoxycarbonylation of Various Aromatic Bromides and Chlorides Catalyzed by CoCRACO[a]



run	R	X	reactn time (h)	conversn[b] (%)	isolated yields[c] (%)		overall carbonylation with respect to Co (%)
					ArCOOMe	ArCOOH	
1	H	Br	6	97	88	9	970
2	*o*-Me	Br	18	75	53	9.5	625
3	*m*-Me	Br	16	95	78.5	9.5	880
4	*p*-Me	Br	14	93.5	76	13.5	895
5	*o*-MeO	Br	20	95	84.5	8	925
6	*m*-MeO	Br	17	92	80	8	880
7	*p*-MeO	Br	16	93.5	79	12.5	915
8	*p*-MeS	Br	71	85	70.5	11.5	820
9	*o*-F	Br	8	92	81	3	840
10	*m*-F	Br	7	97	85	8	930
11	*p*-F	Br	7	97.5	81.5	8	895
12	*o*-Cl	Br	10	95	76[d]	3	1030
13	*m*-Cl	Br	8	99	86[e]	9.5	1005
14	*p*-Cl	Br	8	99	86[f]	3	1030
15	*o*-Br	Br	18	93.5	85[g]	3[g]	1810
16	*m*-Br	Br	14	99	86[h]	5[h]	1930
17	*p*-Br	Br	14	99	80.5[i]	6[i]	1850
18	*o*-Cl	Cl	71	88	61	17	780
19	*m*-Cl	Cl	63	91.5	66	17	830
20	*p*-Cl	Cl	63	90.5	65	15	800
21	*o*-CF_3	Br	18	95.5	83.5	9	925
22	*m*-CF_3	Br	16	99	76	19	950
23	*p*-CF_3	Br	15	95	72	16	880
24	*o*-CN	Br	12	82	*o*-$C_6H_4(COOH)_2$	78	780
25	*m*-CN	Br	36	86	71.5	9	805
26	*p*-CN	Br	35	85	68.5	12.5	810
27	*o*-NO_2	Br	42				
28	*m*-NO_2	Br	42				
29	*p*-NO_2	Br	42				
30	1-Br-naphtyl		76	83	63	15	780
31	2-Br-naphtyl		43	85	75.5	5.5	810

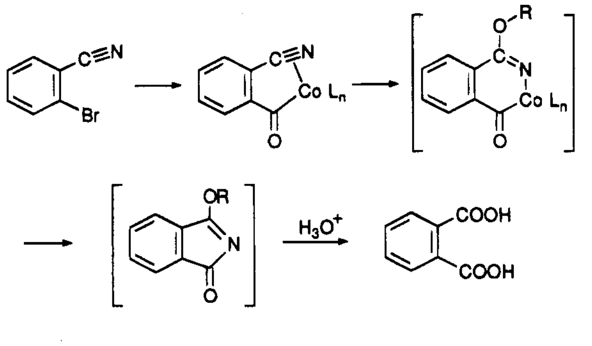
[a] Reactions performed at 40 °C in 10 mL of THF and 10 mL of MeOH under CO (1 atm) with the following reactant ratio: ArX/NaH/*t*-AmONa/$Co(OAc)_2$ = 10/26/4/1. [b] Calculated after isolation of unreacted ArX. [c] Isolated yields based on starting aryl halide. [d] 12% of *o*-$C_6H_4(COOMe)_2$ were isolated. [e] 2.5% of *m*-$C_6H_4(COOMe)_2$ was isolated. [f] 7% of *p*-$C_6H_4(COOMe)_2$ were isolated. [g] Yields of diester and diacid respectively. 4% of *o*-Br-C_6H_4COOMe was also isolated. [h] Yields of diester and diacid respectively. 8% of *m*-Br-C_6H_4COOMe was also isolated. [i] Yields of diester and diacid respectively. 12% of *p*-Br-C_6H_4COOMe was also isolated.

Results and Discussion

Methoxycarbonylation of Aryl and Heteroaryl Halides. From a systematic study performed in MeOH, the following useful information emerged: (i) In THF, use of a sun lamp through the Pyrex flask photostimulated carbonylation of PhBr in the presence of a catalytic amount of CoCRACO's. (ii) The main product formed was methylbenzoate. (iii) No reaction took place without a sun lamp. (iv) Among the possible activating agents of CoCRACO,[6] the best was t-$C_5H_{11}ONa$. (v) The best reagents ratio for the t-$C_5H_{11}ONa$–CoCRACO was NaH/t-$C_5H_{11}ONa$/$Co(OAc)_2$ equal to 2.6/0.4/0.1 relative to the substrate. Thus, to perform the further carbonylations we used catalytic amounts of the above defined *t*-AmONa–CoCRACO prepared under atmospheric pressure of CO.

Methyl Esters from Aryl Halides. The results obtained with a number of representative aryl halides have been reported in the Table 1. These data merit a number of comments. In every case, the carbonylations were catalytic relative to the cobalt of CoCRACO. The expected methyl esters were obtained in very good yields accompanied with one exception (run 24) by a small amount of the corresponding acid. The formation of such acids has already been observed.[6-9] It is essentially due to the presence of some NaOH in NaH and some residual water in the reagents. A notable exception was found with 2-bromobenzonitrile (run 24) which appeared as much more reactive than the 3- and 4-bromo isomers and led only, whatever the experimental conditions used, to orthophtalic acid. This result may be compared to the recently published formation of 3-alkoxyphthalide during the Pd(0)-catalyzed carbonylation of 2-bromobenzaldehyde.[10] According to these authors, run 24 may be interpreted as given in Scheme 1.

Scheme 1



Most of our results support the existence of a S_{RN}^{1} mechanism. Thus, no reaction took place without photostimulation. As expected for radical reactions, inductive electronic effects did not substantially intervene

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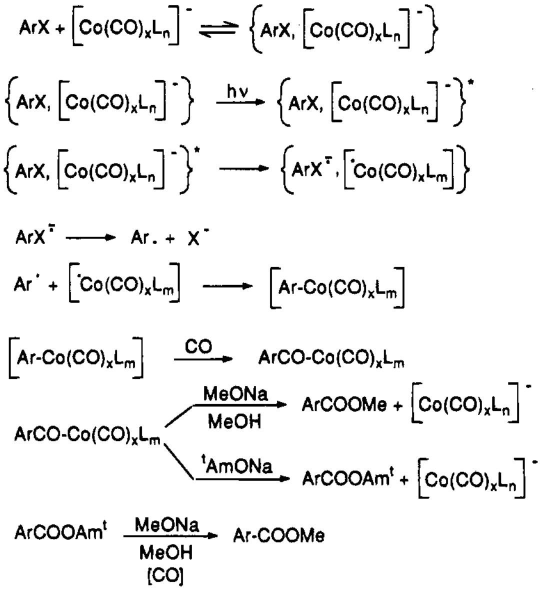
Table 2. Methoxycarbonylation of Various Heteroaromatic Bromides and Chlorides Catalyzed by CoCRACO[a]

$$[\text{HetAr-X}] \xrightarrow[\text{THF - MeOH - 40 °C - CO (1 atm.)} \atop \text{sun lamp}]{t\text{-AmONa-CoCRACO}} [\text{HetAr-COOMe}] + [\text{HetAr-COOH}]$$

run	HetAr-X	reactn time (h.)	conversn[b] (%)	isolated yield[c] (%)		overall carbonylation with respect to Co (%)
				HetArCOOMe	HetArCOOH	
1	2-Cl-thiophene	15	91.5	81	7	880
2	3-Cl-thiophene	17	89	77.5	8.5	860
3	2-Br-thiophene	13	99	83	2	850
4	3-Br-thiophene	14	99	80	12.5	925
5	2-Br-5-Me-thiophene	19	98	86.5	7	935
6	2-Cl-benzothiophene	48	85	71	9.5	805
7	3-Br-benzothiophene	48	85	65.5	12.5	780
8	2,5-di-Br-thiophene	54	81	55[d]	17[d]	1440
9	3-Br-pyridine	61	91.5	70	*e*	700
10	2-Br-furan	42	99	72	2	740
11	3-Br-furan	36	89.5	74	5	790

[a] Reactions performed at 40 °C in 10 mL of THF and 10 mL of MeOH under CO (1 atm) with the following reactant ratio: ArX/NaH/*t*-AmONa/Co(OAc)$_2$ = 10/26/4/1. [b] Calculated after isolation of unreacted HetArX. [c] Isolated yields based on starting heteroaryl halide. [d] Yields of diester and diacid, respectively. [e] Nonisolated.

Scheme 2



during these carbonylations (compare, for example, runs 1–7 to runs 21–23). On the contrary, substrates with stabilizing radical structures (runs 25, 26, and 27–31) were carbonylated very slowly in such a way that nitroaryl halides did not react under our conditions (runs 27–29). We guess that the presence of sulfur as a stabilizing atom must also be responsible for the low reactivity observed with *p*-MeSC$_6$H$_4$Br (run 8).

The study of dihalogenobenzenes (runs 12–20) also led to interesting observations. With dibromobenzenes (runs 15–17), the diesters were the main products of the reaction. Monitoring the reaction by GC showed that the carbonylation of the bromo ester intermediates was faster than the carbonylation of the starting dibromo substrate.

The usual difference of reactivity between chlorine and bromine was well emphasized in runs 12–14 where chloroesters were easily obtained from the corresponding chlorobromobenzenes. Interestingly, while chlorobenzene was unreactive, dichlorobenzenes were slowly carbonylated into the corresponding chloroesters (runs 18–20). We noted that under 4 bars of CO the reactions took place in 24 h. Such an observation is of some interest from a further practical point of view. Finally, it appeared that our carbonylating reagents were insensitive to steric effects since no reactivity difference was observed between *ortho*-substituted halides and their corresponding *meta* and *para* isomers.

Methyl Esters from Heterocyclic Halides. It is well known that transition metal complex catalysts may be deactivated by substrates containing heteroatoms such as nitrogen and especially sulfur. Thus, it was of interest to know if our carbonylation reagents tolerated such poisoning derivatives and if the carbonylation of heteroaryl halide could be performed. On the other hand, since sulfur is the most efficient poisoning heteroatom, we focused most of our study on thiophene derivatives. The results obtained are gathered in Table 2 where a few oxygen- and nitrogen-containing heterocycles are also mentioned. From these data, it appears that heteroaryl halides may be easily carbonylated in good to excellent yields. Even in the presence of a large excess of sulfur, since cobalt was used in catalytic amount, the carbonylation took place as easily as in the aromatic series. The intriguing point was that chlorothiophenes (runs 1 and 2) were carbonylated as easily as corresponding bromo derivatives (runs 3 and 4) while chlorobenzene never reacted under our conditions. For the present time we have no clear interpretation for this apparent contradiction.

Mechanism of the Methyl Ester Formation. According to Bunnett's hypothesis[11] dealing with photostimulated S$_{RN}$1 reactions, the mechanism reported in Scheme 2 may be proposed. The carbonylating species is just symbolized since in such a complex reaction medium we cannot ascertain that [Co(CO)$_4$]$^-$ is the actual reagent. The first step ought to be the formation of a complex between the aryl halide and the cobalt species when then transforms into an exciplex by photostimulation. Single electron transfer (SET) must then take place in the exciplex leading finally to the radical anion of the aryl halide which is the starting point of the usual S$_{RN}$1 mechanism. Two pathways may lead to methyl esters. The first is the usual condensation of sodium methoxide with the acyl cobalt species. However the *t*-AmONa included in CoCRACO may also interact to give the corresponding ester, transesterification of which should lead to the methyl ester. In fact, it is well known

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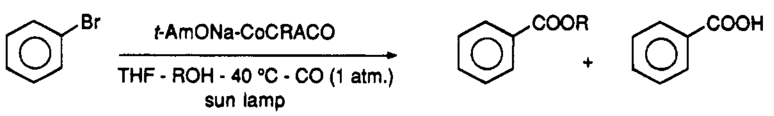
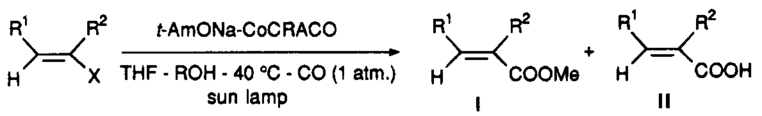
Table 3. Alkoxycarbonylation of Bromobenzene Catalyzed by CoCRACO[a]



run	R	reactn time (h.)	conversn[b] (%)	isolated yield[c] (%) PhCOOR	PhCOOH	overall carbonylation with respect to Co (%)
1	Me	14	89	76	3	1580
2	Et	24	95	83	6	1780
3	$C_{10}H_{21}$	79	88	66.5	11.5	1560
4	$MeO(CH_2)_2$	20	87	69	16	1700
5	$EtO(CH_2)_2$	15	88	81	5	1720
6	$BuO(CH_2)_2$	22	89	74.5	12	1730
7	$PhCH_2$	86	93	69.5	16	1710
8	*i*-C_3H_7	32	93.5	82	1.5	1670
9	*i*-C_5H_{11}	32	95	73	2.5	1510
10	*c*-C_5H_9	18	97	78.5	12.5	1820
11	*t*-C_4H_9	72	84.5	72	5.5	1550
12	*t*-C_5H_{11}	40	3	tr.	tr.	

[a] Reactions performed at 40 °C in 10 mL of THF and 10 mL of ROH under CO (1 atm) with the following reactant ratio: ArX/NaH/*t*-AmONa/$Co(OAc)_2$ = 20/26/4/1. [b] Calculated after isolation of unreacted PhBr. [c] Isolated yields based on starting PhBr.

Table 4. Methoxycarbonylation of Various Vinylic Bromides and Chlorides Catalyzed by CoCRACO[a]



run	R^1	R^2	X	reactn time (h.)	conversn[b] (%)	isolated yield[c] (%) I	II	overall carbonylation with respect to Co (%)
1	H	C_3H_7	Br	62	86.5	62.5	10.5	730
2		$-(CH_2)_4-$	Cl	38	92	78	5	830
3		$-(CH_2)_4-$	Br	22	72.5	59.5	8	675
4		$-(CH_2)_6-$	Cl	62	80.5	67	10.5	775
5		$-(CH_2)_6-$	Br	38	83	65.5	11.5	770
6	H	$C_6H_{11}CH_2$	Cl	120	71	54.5	13	675
7	H	$C_6H_{11}CH_2$	Br	91	81	65	5	700
8	Ph	H	Br	41	85	65[d]	4[d]	690
9		$-O(CH_2)_3-$	Br	57	87	71	13	840

[a] Reactions performed at 40 °C in 10 mL of THF and 10 mL of MeOH under CO (1 atm) with the following reactant ratio: RX/NaH/*t*-AmONa/$Co(OAc)_2$ = 10/26/4/1. [b] Calculated after isolation of unreacted vinyl halide. [c] Isolated yields based on starting vinyl halide. [d] *trans*-Cinnamic ester or acid was exclusively obtained.

that transesterifications take place in the presence of alkoxides.[12] Moreover, we verified that *tert*-amyl benzoate was easily transformed into methyl benzoate in our reaction medium. This result agrees with our previous work dealing with the CRA-catalyzed transesterifications.[13] So, it must be reasonably accepted that the two pathways took place simultaneously during the carbonylations. According to these hypotheses, alkoxycarbonylation with alcohols other than methanol had to be expected as possible. This point has been verified.

Alkoxycarbonylation of Bromobenzene. In order to verify our above conclusions, we performed a number of carbonylations of bromobenzene in the presence of some representative alcohols. The results obtained are reported in Table 3, where methanol is mentioned for comparison. From these data it appears that alkoxycarbonylations catalyzed by *t*-AmONa−CoCRACO may be easily performed with a number of alcohols. Yields were generally good and the reactions highly catalytic in cobalt. If the molecular weights are not too different the trend of reactivity of the alcohols was, as expected: primary > secondary > tertiary. Unexpectedly, carbonylation in *t*-AmOH was extremely slow while *t*-AmONa−CoCRACO appeared as a very good carbonylating reagent. However, some limitations were observed. Thus, we never succeeded in the carbonylation of amino-, dialkylamino-, or (alkylthio)ethanols.

Methyl Esters from Vinyl Halides. With the above results in hand, we wondered if *t*-AmONa−CoCRACO would not be efficient in the catalytic alkoxy carbonylation of vinyl halides. To the best of our knowledge, the cobalt carbonylation of vinyl halides under atmospheric pressure has rarely been published.[3,7a] Under our conditions, we found that a number of vinyl halides could be alkoxy carbonylated in fair to good yields as displayed in Table 4. The results obtained underline the special properties of CoCRACO's which have already been found able to carbonylate some vinyl halides in THF.[7] One unexpected, but interesting point, was the easy alkoxycarbonylation of vinyl chlorides. Our interest in the synthesis of oxygen-containing heterocycles starting from 3-bromodehydropyran[14] led us to study its carbonylation (run 9). It appeared that the presence of the heteroatom did not impede the reaction.

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Conclusion

The present work shows that RONa–CoCRACO's easily prepared from $Co(OAc)_2$ are good catalysts in the alkoxycarbonylation of aryl, heterocyclic, and vinyl halides. They avoid the use of CH_3I and compare favorably with $Co_2(CO)_8$. The new procedure requires only inexpensive lamps and common Pyrex glassware. It can therefore be conveniently applied to larger scale reactions. Current investigations aim at improving and extending the fields of application of this new carbonylating process.

Experimental Section

Materials. N20 Carbon monoxide (Airgaz) was used. Fluka sodium hydride (60–65% in oil) was used after three washings with THF under nitrogen. Each batch of sodium hydride was titrated by standard techniques. Cobalt acetate (Fluka) was dried in vacuo for 12 h at 100–120 °C. The activating and esterifying alcohols were distilled from sodium. THF was distilled from a sodium–benzophenone adduct and stored over sodium wires. The absence of peroxides was checked before each run. Aromatic and heteroaromatic halogenated substrates were purchased from commercial sources and were used without further purifications. 2-Bromofuran or 3-bromobenzothiophene was prepared from furan or benzothiophene and *N*-bromosuccinimide according to the Prugh procedure.[15] 2-Bromobenzothiophene was prepared as described by Coderc.[16] 2-Bromopentene, 3-cyclohexyl-2-bromopropene, and 3-cyclooctyl-2-bromopropene were prepared according to Lespiau and Bourguel's procedure.[17] Chlorocyclohexene and chlorocyclooctene were prepared by the Mousseron and Jacquier procedure.[18] Bromocyclohexene and bromocyclooctene were prepared, respectively, from dibromocyclohexane and dibromocyclooctane.[19]

General Procedure for Methoxycarbonylation. *t*-AmOH (4 mmol) in 3 mL of THF was added to a suspension of degreased NaH (26 mmol) in a gently refluxing THF (3 mL) under a N_2 atmosphere. After 1 h, the reaction medium was cooled to 15 °C and N_2 was replaced by CO. Then, cobalt acetate (1 mmol) in 3 mL of THF was added. A deep blue color rapidly developed. Note that the development of a black color indicates a noncarbonylating reagent. The mixture was stirred for 1 h under a slow stream of CO at 15 °C. Methanol (10 mL) was then slowly added at −5 °C, and the photolysis by nonfluorescent sun lamp was begun. The obtained reaction mixture was heated to 40 °C (heating was effected by means of the IR output of the sun lamp), and the halogenated compound (10 mmol) was then added to 1 mL of THF. The reaction was monitored by GC analysis of small aliquots. Upon completion of the reaction, the medium was acidified with dilute HCl and extracted with diethyl ether. Esters and acids were then obtained after classical acid–base extraction, drying over $MgSO_4$, evaporation of the solvents, and usual flash chromatography. Isolated acids and esters gave spectroscopic data and physical constants in accord with the literature[20] or those of authentic samples.[3,4,6-10]

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