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NICKEL-CATALYZED AMIDATION OF BROMO- AND IODOBENZENE

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Summary

The carbonylation of aryl halides in the presence of p-$RC_6H_4NH_2$ (R = H, CH_3, Cl) or C_6H_5NHR (R = CH_3, C_2H_5) and a catalytic amount of a nickel(II) or nickel(0) tertiary phosphine complex at 150°C or above under carbon monoxide pressures is reported. Amides were obtained in high yields in reactions with p-$RC_6H_4NH_2$, but with C_6H_5NHR compounds the expected *N*-alkyl benzanilides were not formed, benzanilide in low yield being formed instead. A possible catalytic cycle based on an active Ni^0-carbonyl complex is suggested, and the observed deactivation of the catalytic system when the carbon monoxide pressure falls to 6 atm or below, is accounted for in terms of a side reaction which produces an inactive Ni^{II} compound.

Introduction

The nickel-catalyzed carbonylation of organic halides to give carboxylic acids or ester derivatives has been extensively studied under mild [1] and drastic [2] conditions. For both catalytic and stoichiometric reactions $Ni(CO)_4$ is usually, and nickel-phosphine complexes have been little studied [2a,3], but it was recently reported that nickel-phosphine complexes catalyze the reductive carbonylation of aromatic nitro compounds [4] and that some amide formation is observed when p-bromonitrobenzene is used. Investigation showed that the amides are produced by carbonylation of the C–Br bonds. The catalyzed amidation of aryl halides in the presence of aniline or its derivatives is reported in this paper.

Results and discussion

$NiL_2(CO)_2$ (L = PPh_3) was found to react unselectively with iodobenzene (I/Ni = 2) and aniline in a toluene or benzene solution at 100°C under nitrogen to give various organic products such as benzanilide, diphenyl, and benzene. Variable amounts of carbon monoxide (from 1.2 to 1.5 mole per mole of starting complex) were found in the gas phase, and nickel was recovered as $NiI_2(PhNH_2)_4$. No reaction was observed under one atmosphere of carbon monoxide unless temperatures above 140°C were used. The conditions have been established under which iodo- and bromo-benzene react with CO ($P_{CO} \geqslant 8$ atm) and aniline or its derivatives p-$RC_6H_4NH_2$ ($R = CH_3$, Cl) in the presence of catalytic amounts of $NiL_2(CO)_2$ to give amides selectively (Reaction 1, $Ar = p$-RC_6H_4).

$$2ArNH_2 + PhX + CO \rightarrow PhCONHAr + ArNH_3X \qquad (1)$$

The reaction can be carried out at 150–180°C with iodobenzene, but higher temperatures (180–200°C) are required for bromobenzene (Table 1, run 7, 8), while no reaction occurred with chlorobenzene up to 220°C (Table 1, run 9). Such a reactivity sequence was previously observed in the alkoxy-carbonylation of aryl halides in the presence of $Ni(CO)_4$ [1c]. The catalysis was performed in benzene or toluene solution and also, in the case of the benzanilide synthesis, in pure aniline.

Ni^{II}-phosphine complexes of formula NiX_2L_2 or their carbonyl derivatives NiX_2L_2CO (X = Cl, Br, I; L = trialkyl or alkyl-aryl phosphine) and $NiX_2(PhNH_2)_y$ (X = Cl, Br; $y = 2$; X = I, $y = 4$) were also found to be effective catalysts for the reaction. N-alkylanilines C_6H_5NHR ($R = CH_3$, C_2H_5) were found to react under the same conditions with PhI and CO, but in these cases the expected N-alkylamides were not formed and the reaction proceeded as in eq. 2.

$$PhI + CO + C_6H_5NHR \rightarrow PhCONHC_6H_5 + [C_6H_5NHR_2]^+I^- \qquad (2)$$

The ammonium salts were characterized by elemental analyses and comparison of their IR spectra with those of authentic samples. Alkyl halides could not be used as substrates owing to formation of alkyl-ammonium salts with anilines. Finally amidation of the aryl halides did not occur in the presence of alkyl and dialkyl-amines RNH_2 and R_2NH ($R = C_3H_7$, C_4H_9); in these cases the outcome was as observed by Hoberg et al. [5]. The results of the most significant aryl halide amidation experiments are listed in Table 1, which shows the yields and reaction conditions. Only a small change in catalytic activity was observed when the catalyst system was varied (Table 1, run 1–6). There was a clear dependence on the temperature and the CO pressure, the activity increasing over the range 150–180°C (Table 1, run 10–12) and the range 8–40 atm (run 13–17).

Independent of the catalytic system used, the IR spectra of the reaction solutions show a set of the bands, at 2070w, 1990vs, and 1935s cm^{-1}, characteristic of the presence of a mixture of $NiL_2(CO)_2$ and $NiL(CO)_3$ [6]. In the case of catalytic systems involving the more basic phosphines, an additional band at 2040 cm^{-1} is also observed. This band, which disappears when a stream of nitrogen is bubbled through can be assigned to $Ni(CO)_4$. Its formation could be due to the oxidation of the phosphine to phosphine-oxide (eq. 3), such as was observed with 1,2-bis(diphenylphosphino)ethane in a basic medium [6].

$$Ni^{2+} + H_2O + L \rightarrow Ni^0 + LO + 2H^+ \qquad (3)$$

However, when the reaction was carried out at lower pressures, no carbonyl band was detected in the reaction solution, and the systems were inactive (see below). Under these conditions, the nickel starting complex was recovered as the Ni^{II}-amino complex, e.g. $NiI_2(PhNH_2)_4$ in the case of the reaction with $PhNH_2$ and PhI.

Control experiments showed that under the catalysis conditions $NiI_2(PPh_3)_2$ and $C_6H_5NH_2$ reacted in benzene to form $NiI_2(PhNH_2)_4$.

TABLE 1

AMIDATION OF ARYL HALIDE

Run	Catalyst [a]	Halide [b]	Amine [b]	Time (h)	Temp. (°C)	P_{CO} (atm)	Halide conversion [c] (%)	Amide selectivity [d] (%)
1	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	180	40	95	90
2	$NiBr_2(PMe_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	180	40	93	88
3	$NiCl_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$(22.0)	6	180	40	95	90
4	$NiI_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	180	40	90	89
5	$NiI_2(PhNH_2)_4$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	180	40	92	90
6	$NiI_2(CO)(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	180	40	90	91
7	$Ni(CO)_2(PPh_3)_2$	C_6H_5Br (4.8)	$C_6H_5NH_2$ (22.0)	10	180	40	40	85
8	$NiCl_2(PPh_3)_2$	C_6H_5Br (4.8)	$C_6H_5NH_2$ (22.0)	6	200	40	85	86
9	$Ni(CO)_2(PPh_3)_2$	C_6H_5Cl (4.9)	$C_6H_5NH_2$ (22.0)	10	220	40	4	–
10	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	170	40	65	89
11	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	150	40	36	90
12	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	10	140	40	4	–
13	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	180	30	75	85
14	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	180	20	65	80
15	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	6	180	10	40	78
16	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	15	180	7	25	70
17	$NiI_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NH_2$ (22.0)	15	180	7	5	–
18	$NiCl_2(PPh_3)_2$	C_6H_5I (4.5)	4-$ClC_6H_4NH_2$ (11.8)	6	170	40	92	80
19	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	4-$ClC_6H_4NH_2$ (11.8)	6	180	40	98	81
20	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	4-$CH_3C_6H_4NH_2$ (14.0)	6	180	40	92	85
21	$NiI_2(PPh_3)_2$	C_6H_5I (4.5)	4-$CH_3C_6H_4NH_2$ (14.0)	6	180	40	90	87
22	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NHCH_3$ (12.1)	6	180	40	90	80
23	$Ni(CO)_2(PPh_3)_2$	C_6H_5I (4.5)	$C_6H_5NHC_2H_5$ (12.1)	6	180	40	75	65

[a] 0.1 mmol of complex were used. [b] mmol in parentheses. [c] By gas-chromatography. [d] Based on the weight of product after crystallization from water/ethanol.

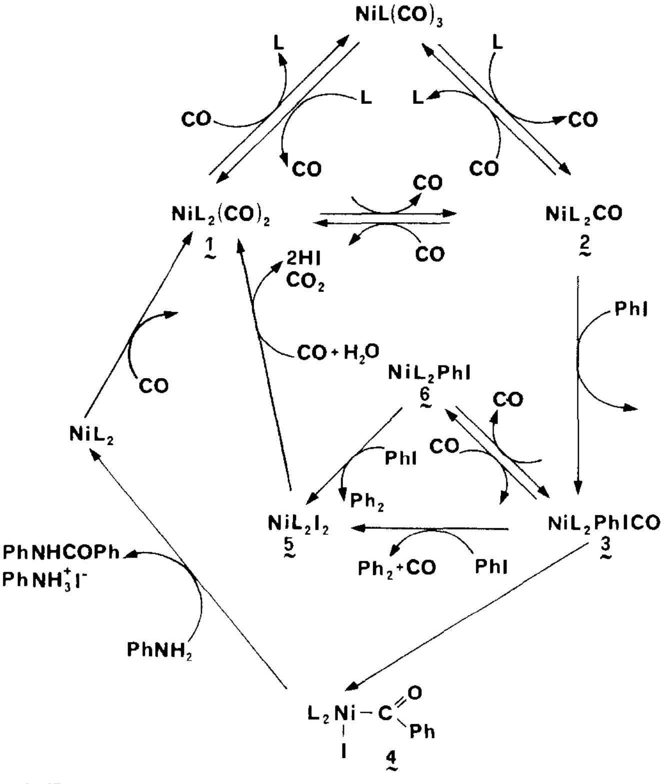
Ni^0 [1a,c] or Ni^{II} [5,7] compounds have been found to be active species in carbonylations and catalytic cycles based on Ni^0-Ni^{II} mechanism have been proposed [1b]. An Ni^0 complex may be assumed to be the active species in our reactions in the light of the following observations

(i) Ni^{II} complexes were ineffective in the catalysis when the carbon monoxide pressure was 7 atm or below. Conversely $NiL_2(CO)_2$ was effective under the same conditions even at low conversions of PhI into amide (Table 1, run 13–15).

(ii) All the investigated Ni^{II} complexes became active when reduced to Ni^0. The reductive carbonylation of Ni^{II} to Ni^0 (eq. 4), which occurs under mild conditions in aqueous-alcoholic media [6], requires higher temperatures and pressures in benzene solution [8].

$$Ni^{2+} + CO + H_2O \rightarrow Ni^0 + CO_2 + 2H^+ \qquad (4)$$

Further support for the view that an Ni^0 species is the active catalyst comes either from the observation that in carbonylation at 10 atm the extent of conversion is independent of the reaction time (Table 1, run 16,17) and from the absence of Ni^0 carbonyl complex bands in the IR spectra of the solution under these conditions. Thus, a catalytic cycle based on the oxidative addition of aryl halide to an unsaturated Ni^0 carbonyl complex **2** (Scheme 1) seems very likely, at least in the aniline reaction.



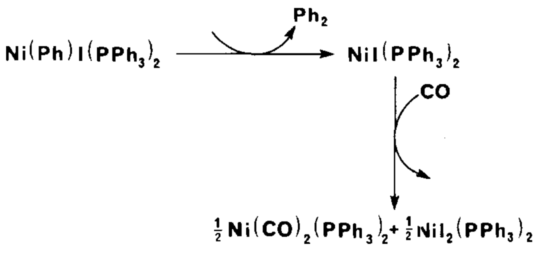
SCHEME 1

The formation of **2**, by thermal dissociation from **1**, is consistent with the difference in reactivity of $NiL_2(CO)_2$ at 100°C when used under nitrogen or carbon monoxide. Higher temperatures must be used to produce **2** under CO, in agreement with the early investigation of carbon monoxide exchange [9]. Oxidative addition of PhI on **2** produces the Ni^{II} complex **3**, which is known [10] to decompose under CO into PhCOI and a Ni^0 carbonyl complex. Thus, the amide is formed through a subsequent reaction between the acyl halide and $PhNH_2$. When the reaction catalysed by $NiL_2(CO)_2$ was performed in absence of aniline, some PhCOI, was produced, as evidenced by the appearance in the IR spectrum of the solution of bands at 1760s, 1710w cm^{-1}, which disappeared when aniline was added (An identical spectrum was obtained when a benzene solution of $Ni(Ph)I(PPh_3)_2$ was exposed to CO).

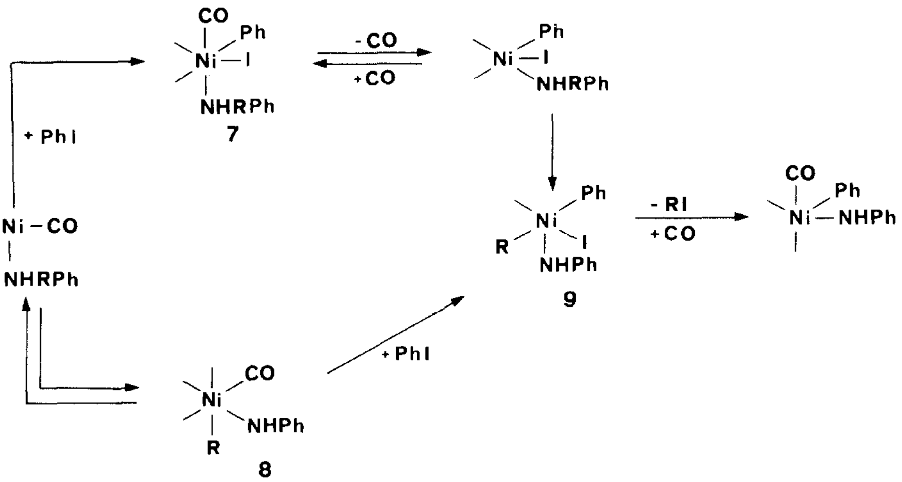
However, the conversion of PhI into PhCOI was low owing to the deactivation of catalytic system, which was recovered as $NiI_2(PPh_3)_2$.

The influence of the carbon monoxide pressure on the catalytic activity, as well as the deactivation of the system when the pressure falls to lower values (6–7 atm), have to be accounted for. A key intermediate appears to be the complex **3**, which under some conditions may go to the amide, and regenerate the active Ni^0 complex, and under other conditions give an inactive Ni^{II} complex. The increase in catalytic activity with increasing pressure (Table 1, run 13–15) suggests that the decomposition of **3**, via the acyl complex **4**, is favoured under these conditions. Furthermore, the high selectivity towards amide when the pressure of CO is above 10 atm suggests that the rate of conversion of **3** into **5** remains fairly low. The effects of this reaction will be more marked with decreasing CO pressure, and the catalytic system will become inactive when the carbon monoxide pressure is insufficient to reduce the Ni^{II} to Ni^0 carbonyl complex via reaction 3. Support for this view comes from the fact that when a pure sample of $NiI(Ph)(PPh_3)_2$, in a benzene-aniline mixture was exposed to one atmosphere of CO in presence of PhI, $NiI_2(PhNH_2)_4$ was formed along with small amounts of diphenyl, benzanilide, and $Ni(PPh_3)_2(CO)_2$. The same products were obtained in absence of PhI, and this suggests that **3** can be transformed into a Ni^{II} compound in the reaction sequence shown in Scheme 2 [11].

In contrast, when $NiI(Ph)(PPh_3)_2$, PhI, and $PhNH_2$ were allowed to react under 40 atm of CO the products were benzanilide and $Ni(PPh_3)_2(CO)_2$. Thus the carbon monoxide pressure may have the dual effect of suppressing Ni^{II} formation and regenerating the Ni^0 catalyst. However, the mechanism illustrated in the Scheme 1 is not consistent with the reaction shown in eq. 2, and the formation of benzanilide and $[NH(CH_3)_2Ph]^+I^-$ in the carbonylation of PhI in presence of $PhNHCH_3$



SCHEME 2



SCHEME 3

suggests the formation of an intermediate species in which coordination of amine to the central metal may be a key step. The migration of the alkyl group from nitrogen to nickel with subsequent reductive elimination of CH_3I may occur, followed by reaction with $NHCH_3Ph$ to form an alkyl ammonium salt. We have no experimental evidence to decide whether the oxidative addition of the PhI occurs before or after the alkyl migration (Scheme 3), but in either case the Ni^{IV} intermediate **9** will be formed. Control experiments showed that the $NHCH_3Ph$ was not carbonylated in the absence of PhI under the conditions used for amide formation, and this favours a reaction sequence involving the intermediate **7**.

Experimental

All reactions and other operations were carried out under oxygen-free nitrogen or carbon monoxide by use of conventional Schlenk techniques. The solvents were purified by standard methods, and distilled and degased before use. The amines were distilled and stored over molecular sieves. Iodo- and bromo-benzene were reagent-grade and used without further purification. Published methods were employed for preparations of $Ni(CO)_2(PPh_3)_2$ [12], $Ni(PPh_3)_4$ [12], NiX_2L_2 [13], $Ni(Ph)I(PPh_3)_2$ [14], NiX_2COL_2 [15], $NiI_2(PhNH_2)_4$ [16]. Gas chromatographic determinations were carried out on Porapack Q 3.5 m column using a Carlo Erba Fractovap C gas chromatograph connected to a Hewlett Packard 3380 A Integrator. GLC analyses were carried out with a GP 20% SP 2100 0.1% Carbowax 1.8 m column in a Varian Vista 6000 gas chromatograph connected to a Varian 4270 Integrator. Infrared spectra were recorded on a Perkin–Elmer 577 spectrophotometer with CaF_2 and KBr cells. The catalysis was carried out in a 55.6 ml stainless steel autoclave containing a Teflon coated stirrer bar. The autoclave was charged, placed in an electric oven equipped with a magnetic stirrer, and reaction was carried out at the temperatures and for the times indicated in Table 1. Amides and anilinium salts were identified by elemental analyses and by comparison of their IR spectra with those of authentic samples prepared by reaction between C_6H_5COCl and *p*-

$RC_6H_4NH_2$ (R = H, CH_3, Cl) or between p-$RC_6H_4NH_2$ and RX (R = H, CH_3; X = I) respectively. The anilinium salts were quantitatively analyzed either by titration with NaOH or by extraction of the amine with ether after neutralization of the ammonium salt with NaOH.

Carbonylation of iodobenzene

In the presence of p-$RC_6H_4NH_2$. Iodobenzene (4.5 mmol) was placed in the autoclave along with $Ni(CO)_2(PPh_3)_2$ (0.10 mmol), aniline (22.0 mmol), benzene (8 ml), and carbon monoxide (40 atm). After the reaction (6 h at 180°C) the autoclave was cooled and the solid contents were filtered off and dried, to give 1.60 g of a white-cream product whose IR spectrum appeared to be a simple superimposition of the spectra of $C_6H_5CONHC_6H_5$ and $[C_6H_5NH_3]^+I^-$. The filtered solution was analyzed by gas chromatography, and iodobenzene (0.046 g, 5% of the starting amount), aniline (1.20 g) and trace amounts of diphenyl and diphenyl ketone were detected. The solid mixture was dissolved in warm methanol (15 ml) and H_2O (15 ml) was added to produce a white microcrystalline product, which was filtered off, dried, weighed, and shown to be pure benzanilide (0.75 g, 90% based on the iodobenzene converted). Elem. Anal. Found (%) (calcd. for $C_{13}H_{11}NO$) C, 79.0 (79.19); H, 5.4 (5.58); N, 6.9 (7.11).

The aqueous-alcoholic solution which contained the anilinium salt was evaporated to dryness and the residue was crystallized from alcohol/benzene. Elem. Anal. Found (%) (calcd. for C_6H_8IN) C, 32.3 (32.6); H, 3.5 (3.62); I, 56.9 (57.45).

In run 2 (Table 1) the aqueous-alcoholic solution obtained by the above procedure was found to require 7.9 ml (4.0 equiv) of a 0.51 *N* NaOH solution for neutralization.

The reactions of PhI with other anilines and catalysts under the same or different conditions of pressure and temperature were carried out by the same procedure. The yields, of amides and the extent of halide conversion are shown in Table 1 (run 18–21).

In the presence of N-alkylanilines. Iodobenzene (4.5 mmol), $Ni(CO)_2(PPh_3)_2$ (0.10 mmol), *N*-methylaniline (12.1 mmol), C_6H_6 (8 ml), and carbon monoxide, were placed in the autoclave and allowed to react for 6 h at 180°C. The mixture was cooled to room temperature, to give two differently coloured layers. The upper layer consisted of a pale-orange crystalline product and the other was a red semi-solid oil. The mixture was filtered and then washed with two 10 ml portions of benzene/ethanol solution (5/1), and the solid residue was identified from its elemental analysis and IR spectrum as $C_6H_5CONHC_6H_5$ (0.64 g, 80% of the reacted C_6H_5I). Elem. Anal. Found (%) C, 79.0; H, 5.4; N, 6.9. Filtration of the product mixture gave a filtrate which consisted of an insoluble red oil and a benzene solution, which were separated by decantation. The benzene solution was analyzed by GLC, and found to contain iodobenzene (0.091 g, 10% of the amount initially taken), $NHCH_3C_6H_5$ (3.8 mmol), and traces of toluene and diphenyl and of an unidentified product. The red oil was washed with cold toluene and dissolved in 5 ml of a methanol/diethyl ether mixture (1/4). Cooling to −10°C gave a pale-yellow microcrystalline product, which after being filtered off become reddish. It was identified as $[C_6H_5NH(CH_3)_2]^+I^-$ from IR spectrum and elemental analysis, although the latter was fully exact. Found (%) (calcd. for $C_8H_{12}IN$) C, 37.4 (38.57); H, 4.6 (4.82); I, 49.1 (50.98).

The reaction with $NHC_2H_5C_6H_5$ (run 23) was carried out by the same procedure. Of the reacted iodobenzene, 65% was converted into benzanilide, but some unidentified products were detected in the solution by GLC.

Reaction between $Ni(CO)_2(PPh_3)_2$ and PhI

A mixture of $Ni(CO)_2(PPh_3)_2$ (1.72 mmol), aniline (15 mmol), iodobenzene (0.5 ml, 4.5 mmol), and benzene (10 ml) was introduced under nitrogen into a 120 ml glass-vessel reactor fitted with a magnetic stirrer bar. The resulting pale-yellow solution was raised to 100°C and allowed to react until the nickel carbonyl bands in the IR spectrum had disappeared (about 1 h). Upon cooling the red-brown reaction solution turned green, and when it was set aside at room temperature overnight, a mixture of green and white crystals was deposited, and this was filtered off and dried (1.03 g). The IR spectrum of the solid mixture contained the bands from both $NiI_2(PhNH_2)_4$ and $C_6H_5CONHC_6H_5$. Green crystals of pure $NiI_2(PhNH_2)_4$ (0.81 g) were obtained by washing the solid mixture with a hot aniline/benzene (1/1) solution. Elem. Anal., Found (%) (calcd. for $C_{24}H_{28}I_2N_4Ni$) C, 41.9 (40.7); H, 4.2 (4.09); I, 36.9 (37.08); Ni, 8.4 (8.58). The mother liquor from the reaction was analyzed by GLC, and iodobenzene (0.20 g, 0.98 mmol), aniline and diphenyl (0.7 g) were detected. When the reaction was carried out in toluene, benzene was also detected. The amount of carbon monoxide released in the reaction was determined in separate experiments carried out under argon with hydrogen as internal standard; reactions with 1.12, 0.88, and 0.79 mmol of $Ni(CO)_2(PPh_3)_2$ gave 1.34, 1.32, and 1.06 mmol, respectively, of CO.

Reactions between $Ni(Ph)I(PPh_3)_2$, C_6H_5I and $PhNH_2$

Under one atmosphere of CO. A mixture of $Ni(Ph)I(PPh_3)_2$ (0.81 g, 1.02 mmol), benzene (10 ml) aniline (1 ml) and PhI (0.5 ml) was kept at 100°C under nitrogen. The resulting red-brown solution turned green when the nitrogen was replaced by CO. The solid mixture was filtered off and dried (0.7 g). Its IR spectrum showed the bands of $NiI_2(PhNH_2)_4$ along with bands consistent with benzanilide.

Under carbon monoxide pressures. $Ni(Ph)I(PPh_3)_2$ (0.80 g, 1.02 mmol) was introduced into the autoclave along with a test-tube containing benzene (9 ml), $PhNH_2$ (1 ml) and C_6H_5I (0.5 ml). The autoclave was filled with CO 40 (atm) and mounted in the electric oven and the temperature raised to 180°C then the vessel was turned upside down to initiate the reaction. The vessel was allowed to cool to room temperature (about 2 h) and the solid was filtered off and dried (0.350 g of a mixture of benzanilide and $[C_6H_5NH_3]^+I^-$ was obtained). The IR spectrum of the yellow filtrate displayed bands at 2070s, 1990vs and 1935vs cm^{-1}. GLC analysis revealed the presence of aniline and PhI.

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