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Palladium-catalyzed synthesis of aromatic acids from carbon monoxide and aromatic compounds via the aromatic C-H bond activation

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Abstract

Various aromatic acids can be prepared from aromatic compounds and CO under mild conditions by Pd-based catalysts via direct aromatic C-H bond activation. With Pd^{II} catalysts, the reaction is electrophilic and proceeds with *ortho-para* orientation when an electron-donating group is attached to the benzene ring; addition of t-BuOOH and allyl halides makes the reaction catalytic with respect to palladium(II). β-Naphthoic acid can be prepared from naphthalene and CO by the Pd/phenanthroline catalyst system in 421% yield based on Pd and in 92% selectivity.

At present the new reactions via C-H bond activation by transition metal complexes are of great interest [1]. However, little is known on the reactions involving C-H bond activation, especially activation of aromatic C-H bonds promoted by transition metals [2]. We have found that the Pd-catalyzed substitution reactions of olefins by aromatic compounds give aryl olefins in one step, which proceeds via an ArPdL σ complex intermediate (**1**) derived from direct activation of an aromatic C-H bond by Pd^{II} [3]. This is the first example of arylation of olefins involving direct activation of an aromatic C-H bond promoted by Pd complexes, to give **1**, and was followed by recent examples of the isolation of ArML complexes from ArH via an aromatic C-H bond activation [4]. After our initial findings, it was found that intermediate **1** could be prepared in situ from various aryl derivatives

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such as mercurials [5], halides [6], amines [7], phosphines [8], arsines [8], bismuthines [8], stibines [8] and diazonium salts [9]. However, we have been especially interested in exploring new reactions via the in situ formation of **1** from the direct aromatic C–H bond activation, and have found that in addition to the olefins, the intermediate **1** thus formed reacts with CO, CO_2 [10] and O_2 [11] to give carboxylic acids and phenol, respectively. Here we describe the palladium-catalyzed reaction of aromatic compounds with carbon monoxide via **1** which derives from direct C–H bond activation, to give aromatic acids in one step, and its application to the selective synthesis of β-naphthoic acid from naphthalene and carbon monoxide. Preliminary communications of this work have appeared [12].

Results and discussion

Reaction of benzene with CO with palladium(II) salts

First the stoichiometric reaction with respect to the palladium(II) salts, $Pd(OAc)_2$ and $Pd(NO_3)_2$, was examined. Reaction of benzene with CO at 15 bar in the presence of $Pd(OAc)_2$ with or without AcOH at 100 °C for 6 h in an autoclave gave benzoic acid in 30 and 44% yields based on Pd, respectively. A CO pressure of 1 bar gave benzoic acid in 27% yield. $Pd(NO_3)_2$ gave a lower yield.

Reaction of various aromatic compounds with CO with $Pd(OAc)_2$

The reactions of CO with anisole, toluene, chlorobenzene, furan, thiophene, and naphthalene are listed in Table 1; the data show that the reactivity of monosubstituted benzenes decreases in the order: OMe > Me > H > Cl, and that the reaction proceeds with *ortho–para* orientation when an electron-releasing group is attached to the benzene ring, which indicates that the reaction is electrophilic.

Effects of additives

a. Allyl halides. The reaction of benzene with CO (1 bar) with $Pd(OAc)_2$ at 75 °C was monitored by GLC. The yield of benzoic acid becomes constant ca. 12 h after initiation of the reaction, and a palladium mirror was formed inside the reaction flask. The reaction is stoichiometric with respect to palladium(II) and ca. 70% of the Pd^{II} species is reduced to Pd^0 by CO because no products other than benzoic acid were formed. In order to enhance the yield of benzoic acid, allyl

Table 1

Reactions of aromatic compounds with CO [a]

Run	Aromatic compound	Product	Yield (%) [b]	*o-/m-/p-*
1	benzene	benzoic acid	26	
2	anisole	methoxybenzoic acid	48	10/0/90
3	toluene	toluic acid	33	36/9/55
4	chlorobenzene	chlorobenzoic acid	14	29/14/57
5	furan	furan-2-carboxylic acid	35	
6	thiophene	thiophen-2-carboxylic acid	18	
7	naphthalene [c]	naphthoic acid	25	55/45 [d]

[a] Aromatic compound: 20 mmol, pCO: 15 bar, $[Pd(OAc)_2]$: 1 mmol, AcOH: 3 ml, 100 °C, 20 h. [b] Isolated yields based on [Pd]. [c] Naphthalene: 20 g. [d] Ratio of α/β-naphthoic acid.

Table 2

Carboxylation of benzene with CO by the $Pd(OAc)_2$/t-BuOOH/CH_2=$CHCH_2Cl$ system[a]

Run	$Pd(OAc)_2$ (mmol)	t-BuOOH[b] (mmol)	CH_2=$CHCH_2Cl$ (mmol)	Time (h)	Temp. (°C)	pCO (bar)	Product yield (%)[c]			
							PhCOOH	PhOH	PhPh	total
1	0.1	35	0.6	24	75	1	448	153	580	1181
2	0.6	30 [d]	0.3 [d]	24	80	bubbling	355	51	388	794
3	0.6	25 [e]	0.3	20	75	1	268	39	266	573
4	0.1	40 [f]	0.35 [g]	24	75	bubbling	1240	195	1927	3362
5	0.1	50 [h]	1 [i]	72	75	bubbling	1318	219	1138	2675

[a] Benzene: 10 ml, AcOH: 2.5 ml. [b] 80% aq. t-BuOOH. [c] Yields based on [Pd]. [d] t-BuOOH and CH_2=$CHCH_2Cl$ both were added during the 24 h period. [e] Initially 15 mmol were added and then 10 mmol were added after 12 h. [f] Initially 10 mmol were added and then 5 mmol each were added every 2 h. [g] Initially 0.05 mmol was added and then 0.05 mmol each added every 2 h. [h] Initially 15 mmol were added and then 5 mmol each added 2, 4, 6, 8, 20, 25, and 30 h after the reaction. [i] Initially 0.3 mmol was added and then 0.1 mmol each added as in *h*.

halides were added to the reaction mixture. The addition of allyl bromide increased the yield to 66% which suggests that the Pd^0 formed is reoxidized to Pd^{II} by oxidative addition of Pd^0 to the halide *. Allyl chloride gave a similar result but allyl iodide and acetate gave lower yields.

b. Hydroperoxides. In order to make the reaction catalytic with respect to palladium(II), we investigated effects of various oxidants. Although Wacker-type additives such as $CuCl_2$ were ineffectual in the reaction, hydroperoxides, especially t-BuOOH, causes the reaction to progress catalytically. For example, reaction of benzene with CO (1 bar) in the presence of t-BuOOH at 75 °C for 24 h gave benzoic acid in 114% yield with a small amount of biphenyl.

A plot of the t-BuOOH concentration ** against reaction time in the reaction of benzene (60 ml) with CO (1 bar) in the presence of $Pd(OAc)_2$ (0.5 mmol), t-BuOOH (50 mmol), and Ac_2O (15 ml) at 80 °C is depicted in Fig. 1, and shows that ca. 75% of the t-BuOOH has been consumed in the first 2 h of the reaction ***. Thus, to keep the t-BuOOH concentration high, repeated additions of peroxide during the reaction were necessary. The results are listed in Table 2. The data in Table 2 show that the repeated additions of t-BuOOH every 2 h to replace what was consumed during the reaction significantly increased the yield of benzoic acid – up to 1300% (run 5). Fig. 2 shows that repeated additions of t-BuOOH every 2 h keep the average t-BuOOH concentration at ca. 0.4 mol/1, indicating that a high concentration of the peroxide is needed to ensure the catalytic carboxylation of benzene with CO.

The probable catalyst cycle is shown in Scheme 1, and involves electrophilic attack of Pd^{II} on the benzene ring to give **1**, insertion of CO into **1** to give **2**, reductive elimination of **2** to the acid anhydride plus Pd^0, and reoxidation of Pd^0 to

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* Oxidative addition of allyl halides to Pd^0 to form π-allyl palladium(II) complexes is well known: see for example, J.P. Collman, L.S. Hegedus, Principles and Application of Organotransition Metal Chemistry, University Science Books, Mill Valley CA, 1980.

** Concentration of t-BuOOH was measured by iodometry.

*** It was found that t-BuOOH was transformed in the presence of AcOH or Ac_2O, into t-$BuOOC(O)CH_3$ which then undergoes decomposition to give O_2, CH_4 and CO_2; the CO_2 thus formed reacts with benzene to give carboxylic acid in low yield [12b].

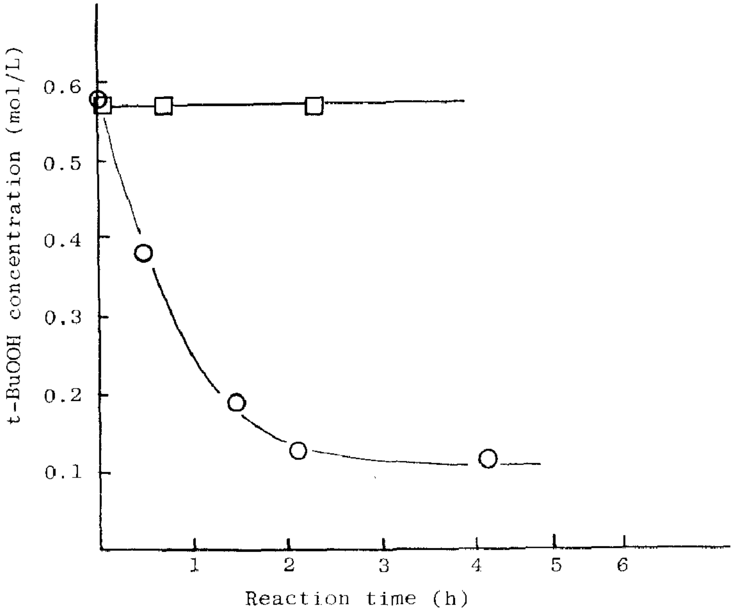


Fig. 1. Concentration of t-BuOOH vs. reaction time in the reaction of benzene (60 ml) with CO (1 bar) in the presence of t-BuOOH (70% aq. solution, 50 mmol) at 80 °C. ○: with $Pd(OAc)_2$ (0.5 mmol) and Ac_2O (15 ml); □: without $Pd(OAc)_2$ and with AcOH (15 ml).

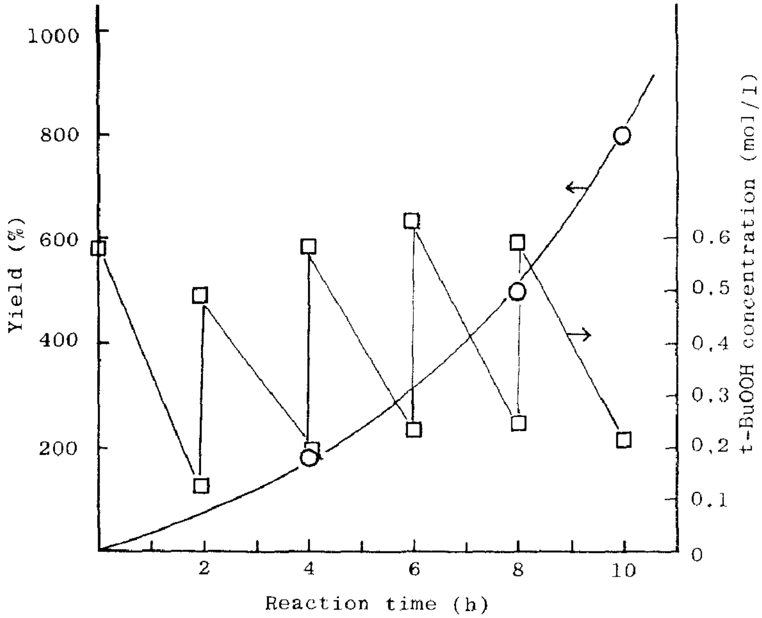
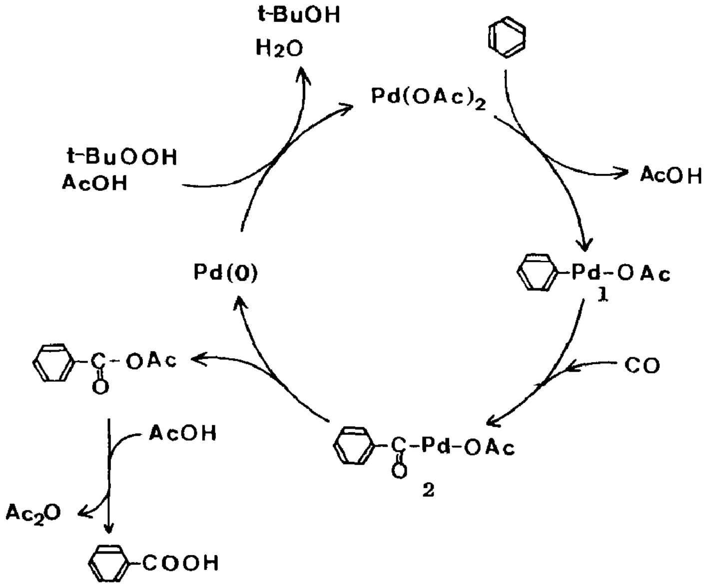


Fig. 2. Yield of benzoic acid (based on Pd) and concentration of t-buOOH vs. reaction time. Reaction conditions: $Pd(OAc)_2$, 1.0 mmol; benzene, 50 ml; AcOH, 12.5 ml; t-BuOOH, initially 28 mmol added and then 28 mmol each added 2, 4, 6, and 8 h after the starting of the reaction. ○: yield of benzoic acid; □: concentration of t-BuOOH.



Scheme 1

Pd^{II} by t-BuOOH. The formation of Ac_2O was confirmed by GLC. Oxidation of Pd^0 to Pd^{II} by t-BuOOH was checked by UV spectroscopy using $Pd(PPh_3)_4$ as Pd^0 species. A suspension of $Pd(PPh_3)_4$ (0.2 mmol) and t-BuOOH (10 mmol) in benzene (12 ml)/AcOH (3 ml) was heated under reflux for 2 h and the UV spectrum of the resulting solution was recorded (Fig. 3). As can be seen from Fig. 3, the spectrum of the resulting solution is similar to that of $Pd(OAc)_2$ in benzene/AcOH solution, and different from that of $Pd(PPh_3)_4$. Calculations from the peak at 505 nm characteristic of Pd^{II} [13] revealed that ca. 85% of the Pd^0 species was oxidized to Pd^{II}.

Selective synthesis of β-naphthoic acid from naphthalene and CO

The repeated addition of t-BuOOH was used in the carboxylation of naphthalene with CO with various palladium catalysts in $CHCl_3$ solution. The repeated addition of t-BuOOH every 2 h gave high yields (1300%) of naphthoic acids in which the α-isomer was a main product and the β-isomer a byproduct *. Interestingly enough, not only Pd^{II} species such as $Pd(OAc)_2$, $Pd(acac)_2$ and $PdCl_2$ but also Pd^0 species such as Pd black and Pd-C are reactive catalysts in this reaction. This indicates that the Pd^0 species is oxidized in situ to Pd^{II} by t-BuOOH.

β-Naphthoic acid is an important raw material since it easily undergoes disproportionation to give naphthalene and a product that can be transformed into high grade polyester resins [14] naphthalene-2,6-dicarboxylic acid. Therefore, we have investigated the reaction conditions where the β-isomer is formed predominantly from CO and naphthalene. Table 3 summarizes the effect of additives on the selective formation of β-naphthoic acid from CO and naphthalene. The data in

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* The usual electrophilic substitution of naphthalene also gives α-isomers as main products; J.D. Roberts, R. Stewart, and M.C. Caserio, Organic Chemistry, W.A. Benjamin Inc., Menlo Park CA, 1973, p. 575.

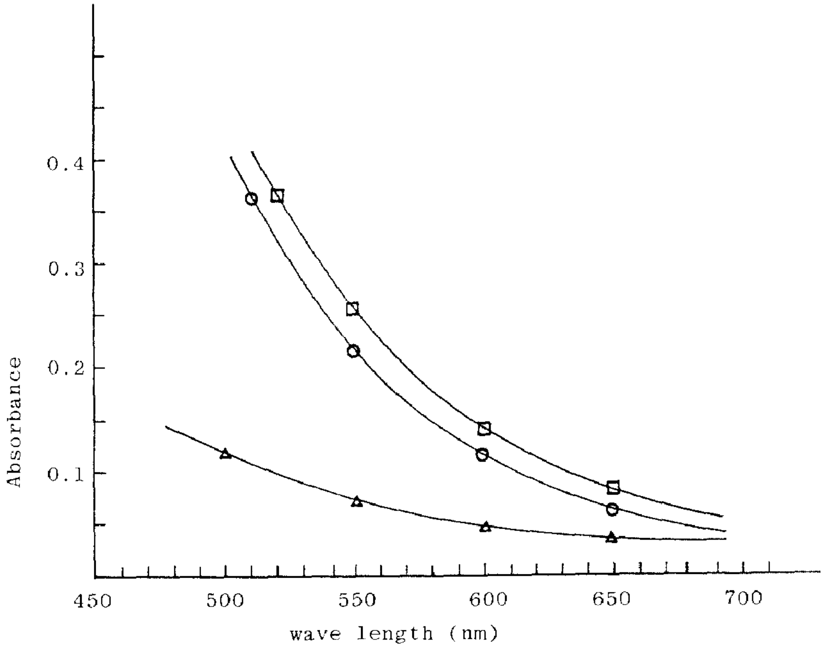

Fig. 3. UV absorbance of $Pd(OAc)_2$ (0.2 mmol) (□) in benzene (4 ml)/AcOH (1 ml), the resulting mixture (○) obtained from the reaction of $Pd(PPh_3)_4$ (0.2 mmol) with t-BuOOH (10 mmol) in benzene (12 ml)/AcOH (3 ml) at reflux for 2 h, and $Pd9PPh_3)_4$ (0.2 mmol) (△) in benzene (4 ml)/AcOH (1 ml). Reference solution is benzene (4 ml)/AcOH (1 ml).

Table 3 show that addition of 1,10-phenanthroline (phen) greatly increases the yield (306%) and selectivity (91%) (α/β-isomer ratio) of β-naphthoic acid (run 1 compared with 2 and 3) and that even with phen, addition of $CHCl_3$ as solvent and a smaller amount of naphthalene (36 mmol compared with 78 mmol) reduce the yield and the selectivity of the β-isomer (run 4).

Table 3

Effect of additives on the selective synthesis of β-naphthoic acid [a]

Run	Additive	Time (h)	Yield of naphthoic acid (%) [b]	α/β
1	–	24	27	55/45
2	1,10-phenanthroline	24	48	19/81
3	1,10-phenanthroline	72	306	9/91
4	1,10-phenanthroline [c]	24	1.2	87/13
5	bipyridyl	24	16	51/49
6	aniline	24	0 [d]	–
7	quinoline	24	10	48/52
8	triphenylphosphine	24	30	60/40
9	anthranilic acid	24	8	55/45
10	tetraphenylporphine	24	0	–
11	α,α,α-tripyridyl	24	6	30/70
12	dimethylphosphinoethane	24	8	55/45

[a] $[Pd(OAc)_2]$: 0.2 mmol, pCO: 1 bar, [naphthalene]: 78 mmol, [additive]: 1.0 mmol, AcOH: 2.5 g, cyclohexane: 1.5 g, 115°C. [b] Yields based on [Pd]. [c] [Naphthalene]: 36 mmol, $CHCl_3$: 6 ml. [d] No naphthoamide formed.

Table 4

β-Naphthoic acid synthesis from CO and naphthalene by various catalysts [a]

Run	Catalyst	pCO (bar)	pO$_2$ (bar)	Temp. (°C)	Time (h)	Yield of naphthoic acid (%) [b]	α/β
1	Pd(OAc)$_2$	1	–	115	24	48	19/81
2	Pd(OAc)$_2$	1 [c]	–	115	12	0.2	–
3	Pd(OAc)$_2$	0.66	0.34	115	20	421	8/92
4	Pd black	1	–	115	12	12	18/82
5	Pd black	1	–	115	24	32	6/94
6	Pd black	1	–	115	32	143	16/84
7	Pd black	1	–	115	70	250	16/84
8	Pd black [d]	0.66	0.34	135	24	65	7/93
9	Pd(acac)$_2$	1	–	115	24	14	11/89
10	5%Pd-C	1	–	115	24	9	8/92
11	PdCl$_2$	1	–	115	24	0	–
12	Pd(NO$_3$)$_2$	1	–	115	24	32	10/90

[a] [Catalyst]: 0.2 mmol, [phen]: 1.0 mmol, [naphthalene]: 78 mmol, AcOH: 2.5 g, cyclohexane: 1.5 g. [b] Yields based on [Pd]. [c] CO$_2$ was used instead of CO. [d] No cyclohexane used.

The results of the β-naphthoic acid synthesis by the various palladium catalysts are listed in Table 4; it shows that not only Pd(OAc)$_2$ but also Pd-C and Pd black, the zerovalent species, are effective for the direct conversion of naphthalene into β-naphthoic acid. Thus Pd black catalyzes the reaction to afford the acid in 250% yield based on Pd and in 84% selectivity to β-isomer (run 7). This finding is notable because these zerovalent Pd species have been regarded as being nonreactive towards direct activation of an aromatic C–H bond under usual reaction conditions [3].

The formation of naphthoic acid from naphthalene and CO requires one extra oxygen atom for stoichiometry. Thus the origin of the oxygen atom in the naphthoic acid formed was investigated. Table 5 shows that the presence of either AcOH (run 3) or molecular O$_2$ (run 2) alone gives the acid * and the presence of both results in higher yields (run 4), whereas the absence of both AcOH and O$_2$ gives only a small amount of the acid (run 1), suggesting that one oxygen atom in the product is

Table 5

Effect of acetic acid and O$_2$ on the formation of naphthoic acid [a]

Run	AcOH (g)	pCO (bar)	pO$_2$ (bar)	Time (h)	Yield of naphthoic acid (%) [b]
1	0	1.0	0	48	0.1
2	0	0.66	0.34	48	17.5
3	2.5	1.0	0	24	32
4	2.5	0.66	0.34	24	143

[a] [Pd black]: 0.2 mmol, [phen]: 1.0 mmol, [naphthalene]: 78 mmol, cyclohexane: 1.5 g, 115 °C. [b] Yields based on [Pd].

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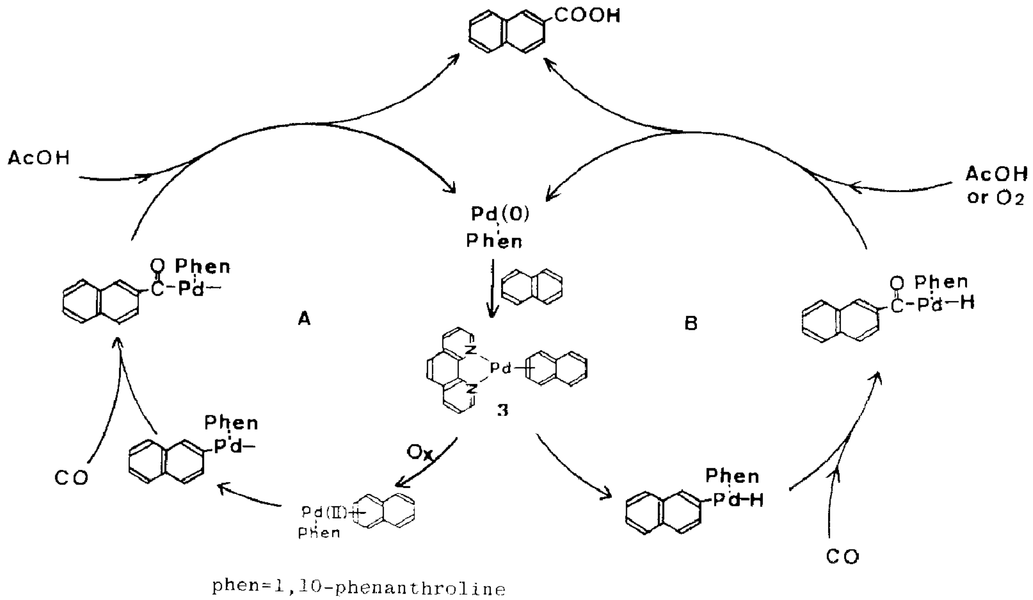
* Hydroxylation of naphthalene does not occur under these conditions: higher CO/O$_2$ pressures are necessary to bring about hydroxylation [11].



Scheme 2

derived from AcOH and/or O_2. The acceleration of the reaction by O_2 (run 3 vs. 4) suggests that it might also reoxidize Pd^0 to Pd^{II}, because $Pd(OAc)_2$, the divalent species, is more active than Pd black or Pd-C under similar conditions (run 3 vs. 5 and 10, Table 4).

The selective formation of β-naphthoic acid can be explained in terms of the reaction paths A and B shown in Scheme 2. Path A involves the usual electrophilic substitution of Pd^{II} with naphthalene and path B involves oxidative addition of a naphthalene C–H bond to Pd^0 which is activated by the coordination of phen. That both phen and a high concentration of naphthalene are needed for selective β-isomer formation, and that the absence of CO results in the formation of binaphthyls indicates that these two catalytic cycles proceed via a very bulky intermediate such as **3** in which the naphthalene ligand is attacked by Pd^{II} (path A) or Pd^0 (path B) at the sterically less-hindered β-position, to give predominantly the β isomer.

Conclusions

Various aromatic acids can be prepared from aromatic compounds and CO under mild conditions by using Pd-based catalysts. With Pd^{II} catalysts, the reaction is electrophilic and proceeds with *ortho–para* orientation when an electron-donating group such as CH_3 is attached to the benzene ring. Addition of t-BuOOH and allyl halides makes the reaction catalytic with respect to palladium(II). β-Naphthoic acid can be prepared from naphthalene and CO by the Pd/phenanthoroline catalyst system in 421% yield based on Pd and in 92% selectivity. Our method for the synthesis of aromatic acids is convenient and useful since the reaction proceeds under very mild conditions and produces the acids in a single step by direct C–H bond activation as compared with the usual methods such as oxidation of alkylarenes.

Experimental

Materials and methods

Palladium black was made from palladium plate dissolved in aqua regia and subsequently reduced by sodium formate. $PdCl_2$ was prepared from the palladium black prepared as above, which was dissolved in aqua regia and the solution was heated to dryness with repeated additions of concentrated HCl. $Pd(OAc)_2$ was prepared from palladium black and glacial acetic acid in the presence of concentrated HNO_3 by the procedure of Wilkinson and coworkers [15]. $Pd(NO_3)_2$ [16] and $Pd(acac)_2$ [17] were prepared by standard procedures. All the other starting materials and solvents were reagent grade and purified before use.

UV spectra were recorded on a Hitachi Model 100-50 spectrophotometer.

General procedure for carboxylation of aromatic compounds with CO.

a. Reaction under 1 bar CO. A 50 ml glass centrifuge tube containing a magnetic stirring bar, appropriate amounts of aromatic compound and AcOH in air, was sealed with a serum cap. CO was bubbled through the mixture by a needle for 15 min, and the Pd catalyst and additives were added to the mixture. Additional CO was bubbled through the mixture for several minutes, and the mixture was stirred at a set temperature for the required length of time (see Tables) under a balloon filled with CO. The mixture was filtered to remove palladium metal, the filtrate was washed with saturated NaCl, and the organic layer was separated off. The organic layer was treated with saturated $NaHCO_3$ and the aqueous layer was separated off. To the aqueous layer was added concentrated HCl and the products were extracted with ether. The combined ethereal solution was dried over Na_2SO_4 and after evaporation of the solvent, the products were isolated by column or gas chromatography. The products were analyzed by use of a Shimazdu GC-6AF or GC-3B gas chromatograph on an OV-17 column. The products were identified from their mixture melting points, IR and NMR spectra, or from their GLC retention times compared with those of commercially available authentic samples.

b. Reaction under CO pressure. In a 200-ml autoclave was placed a 50-ml glass centrifuge tube containing a magnetic stirring bar, appropriate amounts of Pd catalyst, the aromatic compound, and AcOH. The autoclave was closed, flushed with CO and pressurized to 15 bar. The mixture was heated at a set temperature for the required length of time. The resulting mixture was treated as described above.

Analysis of isomer ratio of substituted benzoic acids and naphthoic acids

The product mixture (20 mg) was dissolved in ether (20 ml) and esterified by diazomethane evolved from *p*-toluenesulfonyl-*N*-methyl-*N*-nitrosoamide until the solution turned yellow. The resulting ester was analyzed by gas chromatography on an OV-101 capillary column (60 m). Results are listed in Tables 1, 3, and 4.

UV spectroscopic analysis of Pd^{II} species

A standard solution of $Pd(OAc)_2$ (0.2 mmol) in AcOH (3 ml)/benzene (12 ml), was prepared. For the oxidation test of $Pd(PPh_3)_4$ with t-BuOOH, the standard solution was made by heating a mixture of $Pd(PPh_3)_4$ (0.2 mmol), t-BuOOH (10 mmol) in benzene (12 ml)/AcOH (3 ml) under reflux for 2 h. The sample solutions was prepared by adding 0.2 ml of 1 wt% ethanol solution of 1-nitroso-2-naphthol to

0.2 ml of the standard solution, and the mixture was diluted with benzene/AcOH (4/1) to 10 ml. Then the solution was left to stand for 30 min, and its UV spectrum was recorded at 505 nm [13].

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