Journal of Organometallic Chemistry, 288 (1985) 261–268
Elsevier Sequoia S.A., Lausanne – Printed in The Netherlands

CARBONYLATIVE CROSS-COUPLING REACTION OF ARYL IODIDES WITH ALKYLALUMINUMS BY PALLADIUM COMPLEX CATALYSIS

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(Received December 12th, 1984)

Summary

Secondary and/or tertiary alcohols and unsymmetrical ketones have been obtained in moderate to good yields by the palladium-catalyzed (5 mol%) carbonylative coupling of aryl iodides with alkylaluminum compounds under very mild conditions (20–50°C, 1 atm of carbon monoxide). The type of the reaction product depended on the aluminum reagent employed. While the selective formation of secondary alcohols was observed in the reaction with $i\text{-}Bu_3Al$, the use of Et_3Al led to a mixture of a ketone and two alcoholic products. With Et_2AlCl predominantly unsymmetrical ketones were produced. In all cases, formation of directly cross-coupled products was not observed. DME and benzene can be used as solvents, but THF is unsuitable. Nickel catalysts were found to be ineffective for this reaction.

Introduction

Considerable attention has been increasingly devoted to the study of palladium-catalyzed carbonylative coupling reactions of organic halides with organometallic compounds. For example, bimetallic systems such as Pd-Sn [1–4] and Pd-Zn(Cu) [5] have been recently employed in the synthesis of unsymmetrical ketones under catalytic carbonylation conditions. Although many bimetallic systems used in transition-metal-catalyzed cross-coupling reactions [6,7] may be applied in the carbonylation reaction, it is essential that carbon monoxide insertion and successive reductive elimination of carbonyl-containing products occur prior to reductive elimination of directly cross-coupled products. Therefore, an investigation of such bimetallic systems is of primary interest and importance for the formation of C–C–C structures.

We are currently exploring the scope of palladium-catalyzed carbonylative cross-coupling reactions with a variety of organometallic compounds. During the course of the investigations, we have found that the Pd-Al system is effective for the one-step synthesis of alcohols and ketones. In this paper, the results of the palladium-catalyzed carbonylative cross-coupling of aryl iodides with alkylaluminum compounds are reported.

0022-328X/85/$03.30

Results and discussion

Alkylaluminum compounds have two fundamental characteristics [8,9]: alkylation and reduction. Each characteristic is usually used for a distinct synthetic purpose, although these characteristics are often responsible for competitive side reactions and interfere with the main purpose of the reaction. We considered that it would be possible to produce secondary alcohols from the carbonylative coupling of organic halides and alkylaluminum compounds if the above mentioned fundamental characteristics of the aluminum reagents took part in the desired steps of the reaction. In this study, alkylaluminum compounds containing ethyl or isobutyl groups bonded to the aluminum atom, were chosen as the essential reagents because of their easy availability and relevance to our purpose.

Our initial attempts to force the reaction between aryl iodides and i-Bu_2AlH at 50°C in 1,2-dimethoxyethane (DME) in the presence of $PdCl_2(PPh_3)_2$ and carbon monoxide gave unsatisfactory results, with a significant amount of reduced products of the starting halides and low yields (up to 40%) of the desired alcoholic products. Therefore, i-Bu_3Al was examined. When equimolar amounts of i-Bu_3Al and the halide were used, the reaction was not completed, it stopped after approximately 70% conversion of the halide. The optimum molar ratio of i-Bu_3Al/halide for this reaction in DME usually was found to be 1.5, and the reaction temperature was 50°C. Thus, the desired secondary alcohols were selectively obtained (eq. 1):

$$\underset{(1)}{ArI} + CO + i\text{-}Bu_3Al \xrightarrow[DME,\,50°C]{PdCl_2(PPh_3)_2,\,PPh_3} \underset{(2)}{ArCHCH_2CH(CH_3)_2}\ (OH) \qquad (1)$$

(**a**: Ar = C_6H_5; **b**: Ar = p-$CH_3C_6H_4$; **c**: Ar = p-$CH_3OC_6H_4$; **d**: Ar = p-$CH_3O_2CC_6H_4$; **e**: Ar = p-BrC_6H_4; **f**: Ar = p-$HO_2CC_6H_4$)

TABLE 1

PALLADIUM-CATALYZED CARBONYLATION OF ARYL IODIDES IN THE PRESENCE OF TRIISOBUTYLALUMINUM [a]

Run	Halide	Molar ratio (Al/halide)	Temperature (°C)	Time (h)	Product	Yield [b] (%)
1	**1a**	1.5	50	19	2a	95
2	**1a**	1.5	40	23 [c]	2a	40
3	**1b**	1.5	50	20	2b	82
4	**1c**	1.5	50	42	2c	(73)
5	**1c**	2.0	50	10	2c	73
6	**1d**	1.5	50	30	2d	(22) [d]
7	**1e**	1.5	50	56	2e	83
8	**1f**	2.5	50	7	no reaction	

[a] The reactions were carried out in dry DME using 1 mol of the halide, 5 mol% of $PdCl_2(PPh_3)_2$ and 10 mol% of PPh_3 under a balloon of carbon monoxide. [b] Yields are based on the starting iodides and were determined by GLC; isolated yields in parentheses. [c] During this reaction time, 50% of iodobenzene was consumed. [d] About 60% of the starting halide was recovered.

TABLE 2

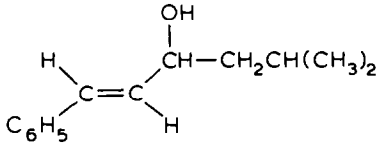
CARBONYLATIONS IN DIFFERENT SOLVENTS

Run	Halide	Solvent [a]	Molar ratio (Al/halide)	Temperature (°C) [b]	Time (h)	Product	Yield (%) [c]
1	**1a**	Benzene	1.5	50	0.5	**2a**	76
2	**1a**	Benzene	1.5	rt	20	**2a**	90
3	**1a**	n-Hexane	1.5	50	16	**2a**	91
4	**1a**	THF	1.5	50	24	**2a**	8
5	**1c**	Benzene	1.0	50	2	**2c** [d]	<10
6	**1c**	Benzene	1.5	rt	2.5 [e]	**2c**	61
7	**1c**	THF/Benzene (1/20)	1.5	rt	6	no reaction	
8	**1c**	THF/Benzene (1/20)	2.5 [f]	50	24	**2c**	79
9	**1c**	n-Hexane	1.5	50	27	**2c**	28
10	**1c**	THF	1.5	50	24	**2c**	trace
11	**1d**	Benzene	1.5	50	24	**2d**	(68)
12	**1e**	Benzene	1.5	50	4	**2e** [d]	<10
13	**1e**	Benzene	1.5	rt	3.5	**2e**	60
14	**1f**	Benzene	2.5	50	15	**2f**	(55) [g]

[a] In all cases, n-hexane was added as a solvent of i-Bu_3Al; see experimental. [b] rt = room temperature (20–25°C). [c] Yields determined by GLC; isolated yields in parentheses. [d] The halide was consumed completely, and some other undetermined peaks were observed in GLC analysis. [e] Although the presence of the unreacted material was still observed by GLC analysis, the yield of the product decreased gradually on increased reaction time. [f] When 1.5 molar equiv. of i-Bu_3Al (based on the starting halide) was used, the reaction was not completed within 48 h at 50°C. [g] The product was isolated as a methyl ester derivative of **1f** by treating the reaction mixture with diazomethane, and identified by comparison with **2d**.

The results and reaction conditions are summarized in Table 1. Although i-Bu_3Al contains a small amount (~ 10%) of i-Bu_2AlH [9], reduction of starting halides practically did not occur under the reaction conditions.

In contrast to the smooth consumption of aryl iodides in the reaction system, aryl bromides remained unaffected. Thus, *p*-bromoiodobenzene (**1e**) was readily converted to the corresponding alcohol **2e** with the *p*-bromo moiety remaining with 83% yield (run 7). On the other hand, the reaction with (*E*)-β-bromostyrene proceeded even under 40°C, being accompanied by competitive formation of styrene and undetermined side reactions. The corresponding alcoholic product **3** was obtained with 50% yield.



(**3**)

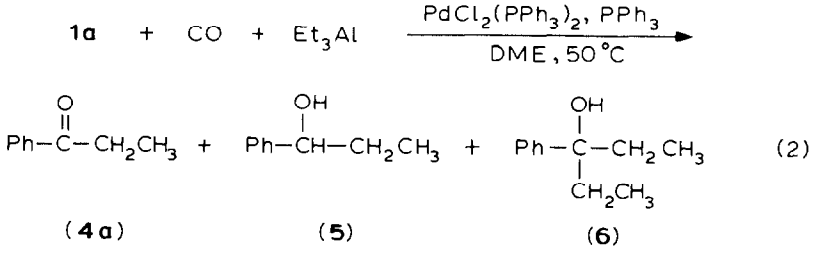
Although this reaction could not tolerate aryl iodides containing reducible groups such as ketones, an ester group was readily accepted * in DME. For example, **1d**

* It is known that an ester function is slowly alkylated or reduced by alkylaluminum reagents [10,11].

underwent the carbonylative coupling reaction almost unaffected at the ester moiety. However, this reaction was slow and only a low yield (22%) was obtained after 30 h at 50°C (run 6). The electron-withdrawing property of the ester group, which retards carbon monoxide insertion [12,19,5], must be responsible for the slowness of the reaction. In the reaction with *p*-nitroiodobenzene and *p*-aminoiodobenzene, no perceptible amount of the desired product was isolated even with a large excess of the aluminum reagent.

The effects of several different solvents on the reaction rates and the yields of alcohols were examined. As can be seen in Table 2, the reaction in THF was extremely slow. This effect agrees with the rate-retarding effect of donor solvents reported for carboalumination reactions [13]. This finding suggests that to proceed successfully the present carbonylative coupling reaction requires an electrophilic attack by the trivalent aluminum center on electron donors, such as the palladium atom, terminal CO, and acyl groups [14] bonded to the palladium atom. Such behavior of the aluminum reagent seems to be closely related to the ready transmetalations mentioned below. On the other hand, the reaction in benzene proceeded relatively fast, and better results were obtained in certain cases (run 11, 14) compared to those of the reaction in DME (Table 1). The low basicity of benzene and increased solubility * of the palladium complexes formed may be responsible for the fast reaction in this medium. Unfortunately, the reaction in benzene was usually accompanied by some tedious side reactions ** which decreased the yield of the product (run 5, 6, 12). This problem was solved by the addition of a small amount of THF to the reaction mixture although in this case the reaction required more aluminum reagent and a longer reaction time (run 8); the presence of THF in amounts equivalent to the amount of i-Bu_3Al used greatly suppressed the side reactions. This effect indicates that the moderate donating ability of DME makes it possible to control the reaction. Hexane did not appear to be a suitable solvent for this reaction because of its low solubilizing properties, although a good yield was obtained in the reaction with **1a** (run 3). In conclusion, the present reaction should be preferentially carried out in DME or benzene.

The use of Et_3Al instead of i-Bu_3Al gave different reaction products. When Et_3Al was allowed to react in DME with **1a** in the presence of the palladium catalyst and carbon monoxide, three products were obtained (eq. 2), and their yields varied with the molar ratio of Et_3Al to **1a** (run 1–3 in Table 3). While propiophenone (**4a**)



was predominantly produced in the equimolar reaction of Et_3Al with **1a**, the yield of

* In most cases, the reactions in benzene gave clear solutions.

** These reactions may involve secondary reactions of the aluminum reagent with the alcoholic products [15].

4a diminished on increasing the ratio to 3/1 and the main product was 1-ethyl-1-phenyl-1-propanol (**6**), formed by the second addition of an ethyl group to the initially produced **4a**. These contrasting differences in the reaction pathway, between the reaction with i-Bu_3Al and with Et_3Al, can be ascribed to the substantial differences in both alkylating and reducing character of these aluminum reagents [9]. The second alkylation of the initial product is due to the ability of Et_3Al to function as a strong alkylating agent, unlike i-Bu_3Al which has higher reducing abilities. Therefore, we have examined a mixture of Et_3Al with i-Bu_3Al in the reaction with **1a** to suppress the second alkylation by Et_3Al. When a 3/1 mixture of Et_3Al with i-Bu_3Al was used (Al/halide 2.0), the desired secondary alcohol **5** was obtained as

$$\mathbf{1a} + CO + Et_3Al/i\text{-}Bu_3Al \xrightarrow[DME,\,50^\circ C]{PdCl_2(PPh_3)_2,PPh_3} \mathbf{2a} + \mathbf{5} \qquad (3)$$

1	/ 2	59%	36%
2	/ 1	23%	70%
3	/ 1	9%	86%

the main product (eq. 3). Furthermore, the predictable use of less reactive Et_2AlCl, as an alternative to Et_3Al in the reaction with **1a**, produced **4a** with 79% yield (run 4, Table 3). The reaction can be performed even with the halide having a carboxyl group (run 8). Further results and the reaction conditions are summarized in Table 3.

GLC analysis of the above reaction mixtures did not show the direct formation of cross-coupled products. Interestingly, the direct cross-coupling reaction did not occur even under conditions without carbon monoxide. This result contradicts the results reported for a Pd-Sn system [1], and suggests that carbon monoxide plays an important role in facilitating the transmetalation with aluminum reagents. Furthermore, a much used β-hydride elimination [16,17], which easily occurs when organic halides or organometallics containing β-hydrogens are employed in palladium-mediated reaction systems, did not interfere in the above reactions under our reaction

TABLE 3

CARBONYLATIONS WITH TRIETHYLALUMINUM OR DIETHYLALUMINUM CHLORIDE

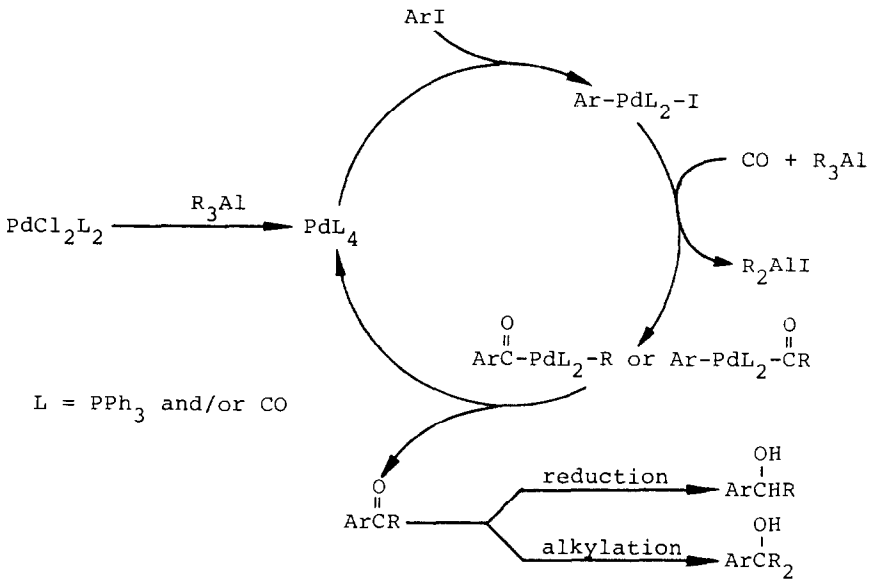
Run	Halide	Aluminum reagent [a]	Solvent [b]	Temperature (°C)	Time (h)	Product(s) (% yield) [c]
1	**1a**	Et_3Al(1.0)	DME	50	20	**4a**(61),**5**(10),**6**(17)
2	**1a**	Et_3Al(2.0)	DME	50	25	**4a**(30),**5**(25),**6**(45)
3	**1a**	Et_3Al(3.0)	DME	50	7	**4a**(trace),**5**(25),**6**(64)
4	**1a**	Et_2AlCl(2.0)	DME	50	15	**4a**(79)
5	**1d**	Et_2AlCl(2.0)	DME	50	24	**4d**(61*)
6	**1d**	Et_2AlCl(2.0)	Benzene	rt	24	**4d**(35) [d]
7	**1e**	Et_2AlCl(2.5)	DME	50	24	**4e**(63*)
8	**1f**	Et_2AlCl(2.5)	Benzene	50	10	**4f**(72*) [e]

[a] The values in parentheses refer to the molar ratios of aluminum reagents to the halides used. [b] In all cases, n-hexane or toluene was added as a solvent of the aluminum reagent; see experimental. [c] Yields determined by GLC; asterisks refer to isolated yields. [d] The starting halide was consumed completely, and some other undetermined peaks were observed in GLC analysis. [e] The product was isolated as a methyl ester of **4f** by treating the reaction mixture with diazomethane, and identified by comparison with **4d**.

conditions. Negishi et al. [17] proposed that reductive elimination could proceed much faster than β-elimination in the palladium-catalyzed cross-coupling reaction of alkylzinc compounds with alkenyl halides. Considering the higher tendency of the acetyl group compared with that of the methyl group reductive elimination [18a], the most likely pathway seems to involve fast reductive elimination of ketones from acylalkylpalladium intermediates prior to β-elimination. At present, however, it can not be neglected that other preferential factors or pathways [5,18b] to avoid the β-elimination may exist in this reaction system, since the mechanism of the palladium-mediated carbonylation reaction is very complex [12] and reductive elimination has been less studied [18a,19] than other key reactions.

In Scheme 1, a possible pathway is shown, involving oxidative addition of aryl

SCHEME 1



iodides to the palladium(0) catalyst, insertion of carbon monoxide and transmetalation with the aluminum reagents, reductive elimination of ketones, and reduction or second alkylation of the ketones by the aluminum reagents.

Finally, we examined the possibility of carbonylative cross-coupling between aryl iodides and i-Bu_3Al by nickel catalysts *. In all cases the reaction failed to yield the desired product and the starting materials were recovered only under similar conditions to those for the palladium catalyst.

Although the inability to tolerate many reactions limits the scope of this reaction, the present study is important for the development of alkylaluminum compounds as reagents for organic synthesis. The study also shows how the use of palladium or nickel catalysts affect the reaction mechanism producing very different results.

* $NiBr_2(PPh_3)_2$ and $Ni(acac)_2$ were examined.

Experimental

1,2-Dimethoxyethane (DME) was distilled from $LiAlH_4$ and stored over sodium wire. Benzene was distilled at atmospheric pressure and also stored over sodium wire. Both tetrahydrofuran (THF) and n-hexane were freshly distilled from $LiAlH_4$ just before use in the carbonylation reactions. Melting points are uncorrected. Infrared spectra were recorded on a JASCO IRA-1 spectrophotometer. ^1H NMR spectra were obtained with a JEOL PS-100 spectrometer using Me_4Si as internal standard. Mass spectra were determined on a JEOL D-300 mass spectrometer. Elemental analyses were performed by the Service Center of the Elementary Analysis of Organic Compounds, Kyushu University. GLC analyses were carried out with a Hitachi 063 chromatograph equipped with a flame ionization detector using (a) a 2 m × 3 mm stainless steel column packed with 5% Carbowax 20M on Chromosorb WAW (60–80 mesh) or (b) a 1 m × 3 mm stainless steel column packed with 15% Polyethylene Glycol 6000 on Chromosorb WAW (60–80 mesh). Column chromatography was carried out on silica gel (70–230 mesh, Merck).

Aluminum reagents. The following aluminum reagents were purchased from commercial sources and were used directly: diisobutylaluminum hydride (15 wt.% solution in n-hexane), triisobutylaluminum (15 wt.% solution in n-hexane), triethylaluminum (15 wt.% solution in toluene), diethylaluminum chloride (15 wt.% solution in n-hexane).

General procedure for the palladium-catalyzed carbonylative cross-coupling reaction of aryl iodides with alkylaluminum compounds. A mixture of 1.92 mmol of an aryl iodide, 0.096 mmol of dichlorobis(triphenylphosphine)palladium(II) and 0.192 mmol of triphenylphosphine (and an appropriate internal standard when the products were analyzed by gas chromatography) in 4 ml of solvent was placed in a 20-ml side-arm flask attached to a balloon via a reflux condenser. The flask was flushed several times with carbon monoxide and then brought to the desired temperature in a constant temperature bath while being stirred. A solution of the aluminum reagent in an appropriate solvent was added using a gas-tight syringe over a 15–20 min period. The reaction was monitored by gas and/or thin-layer chromatography. After completion of the reaction, the mixture was hydrolyzed with water and filtered through a Celite-pad. The filtrate was completely extracted with ether and the extract was dried (Na_2SO_4) and evaporated under reduced pressure. The residue was chromatographed on silica gel.

Carbonylation products. The following products were separated and purified by column chromatography and/or distillation, and then identified by comparison with a commercial sample (propiophenone (**4a**), *p*-bromopropiophenone (**4e**)) or an independently synthesized sample: 1-phenyl-1-propanol (**5**) from propiophenone and BH_3/THF, 3-phenyl-3-pentanol (**6**) from propiophenone and ethyllithium.

*3-Methyl-1-phenyl-1-butanol (**2a**).* (Found: C, 80.19; H, 9.84. $C_{11}H_{16}O$ calcd.: C, 80.44; H, 9.83%.) IR (neat): 3320 cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.95 (d, 6H, 2Me), 1.40–1.85 (m, 3H, CH_2–CH), 1.90 (s, 1H, OH), 4.70 (br t, 1H, C*H*(OH)), 7.20–7.40 (m, 5H, aromatic). MS: *m/e* 164 (M^+).

*3-Methyl-1-(p-methylphenyl)-1-butanol (**2b**).* (Found: C, 80.81; H, 10.20. $C_{12}H_{18}O$ calcd.: C, 80.85; H, 10.18%.) IR (neat): 3320 cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.90 (d, 6H, 2Me), 1.40–1.80 (m, 3H, CH_2–CH), 2.15 (s, 1H, OH), 2.30 (s, 3H, Me), 4.60 (br t, 1H, C*H*(OH)), 7.10 (m, 3H, aromatic). MS: *m/e* 178 (M^+), 121.

1-(p-Methoxyphenyl)-1-butanol (2c). (Found: C, 73.95; H, 9.32. $C_{12}H_{18}O_2$ calcd.: C, 74.19; H, 9.34%.) IR (neat): 3350 cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.90 (d, 6H, 2Me), 1.40–1.80 (m, 3H, CH_2–CH), 2.00 (s, 1H, OH), 3.75 (s, 3H, OMe), 4.60 (br t, 1H, C*H*(OH)), 6.80 (d, 2H, aromatic), 7.20 (d, 2H, aromatic). MS: *m/e* 194 (M^+), 176, 137.

1-(p-Methoxycarbonylphenyl)-3-methyl-1-butanol (2d). (Found: C, 69.99; H, 8.28. $C_{13}H_{18}O_3$ calcd.: C, 70.24; H, 8.16%.) IR (neat): 3400, 1730, 1710 cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.95 (d, 6H, 2Me), 1.40–1.80 (m, 3H, CH_2–CH), 2.30 (s, 1H, OH), 3.90 (s, 3H, COOMe), 4.80 (br t, 1H, C*H*(OH)), 7.30–8.00 (m, 4H, aromatic). MS: *m/e* 222 (M^+), 204, 165.

1-(p-Bromophenyl)-3-methyl-1-butanol (2e). (Found: C, 54.37; H, 6.27. $C_{11}H_{15}BrO$ calcd.: C, 54.33; H, 6.22%.) IR (neat): 3350 cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.90 (d, 6H, 2Me), 1.35–1.80 (m, 3H, CH_2–CH), 2.10 (s, 1H, OH), 4.70 (br t, 1H, C*H*(OH)), 7.20 (d, 2H, aromatic), 7.45 (d, 2H, aromatic). MS: *m/e* 244 ($M^+ + 2$), 242 (M^+), 187, 185.

(E)-3-Hydroxy-5-methyl-1-phenyl-1-hexene (3). m.p. 33–35°C. (Found: C, 81.88; H, 9.62. $C_{13}H_{18}O$ calcd.: C, 82.06; H, 9.45%.) IR (neat): 3320 cm^{-1}. ^1H NMR ($CDCl_3$): δ 0.95 (d, 6H, 2Me), 1.25–1.95 (m, 3H, CH_2–CH), 1.60 (s, 1H, OH), 4.35 (br q, 1H, C*H*(OH)), 6.05–6.30 (dd, 1H), 6.60 (d, 1H), 7.20–7.40 (m, 5H, aromatic). MS: *m/e* 190 (M^+), 133.

p-Methoxycarbonylpropiophenone (4d). m.p. 47–49°C. (Found: C, 68.60; H, 6.53. $C_{11}H_{12}O_3$ calcd.: C, 68.73; H, 6.29%.) IR ($CHCl_3$): 1720, 1690 cm^{-1}. ^1H NMR ($CDCl_3$): δ 1.20 (t, 3H, Me), 2.95–3.15 (q, 2H, CH_2), 3.90 (s, 3H, COOMe), 7.50 (t, 1H, aromatic), 8.05–8.25 (m, 2H, aromatic), 8.60 (m, 1H, aromatic). MS: *m/e* 192 (M^+), 163.

Acknowledgment

The authors thank the Ministry of Education, Japan, for financial support.

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