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# SYNTHESIS AND EMISSION PROPERTIES OF 1,6-METHANO[10]ANNULENE-3,4-DICARBOXIMIDES

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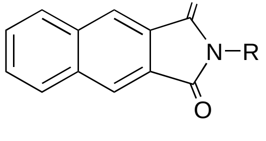
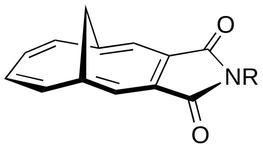
**Abstract** – A mixture of 1,6-methano[10]annulene-3,4-dicarboximide (**1e**) and 1,6-methano[10]annulene-3,4-dicarboxylic anhydride (**3**) was obtained through a two-step sequence involving cyanation and subsequent hydrolysis from ethyl 4-bromo-1,6-methano[10]annulene-3-carboxylate (**5**). Alkylations of **1e** provided **1f** and **1g**, and the nucleophilic substitution of *p*-fluoronitrobenzene with **1e** yielded **1i**. Copper-catalyzed arylations of **1e** with arylhalides gave **1a** and **1h**. Reactions of **3** with anilines afforded **1a** and **1h**. Emission properties of the imides obtained are also reported.

## INTRODUCTION

Spectroscopic properties and photochemistry of 2,3-naphtalimides have long received much attention, especially their luminescent behavior,[1] photocycloadditions[2] and photoreductions.[3] Quite recently Heagy *et al*. reported that dual fluorescent 2,3-naphtalimides could be used in ratiometric DNA detection and white organic light-emitting devices.[4] During the course of our continuing research on 1,6- methano[10]-annulenes,[5] we have been interested in physical and chemical properties of

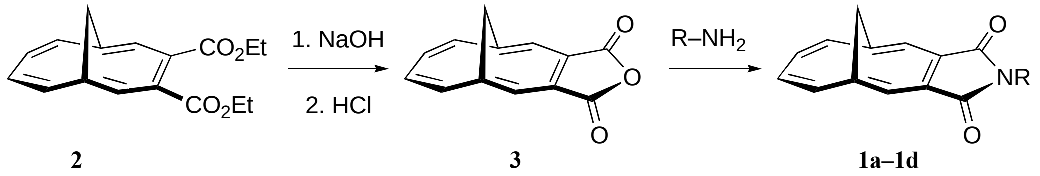


2,3-naphtalimides



**1a** R = $C_6H_5$
**1b** R = *p*-Me-$C_6H_4$
**1c** R = *p*-Et-$C_6H_4$
**1d** R = *n*-Bu
**1e** R = H
**1f** R = Me
**1g** R = Et
**1h** R = *p*-MeO-$C_6H_4$
**1i** R = *p*-$NO_2$-$C_6H_4$

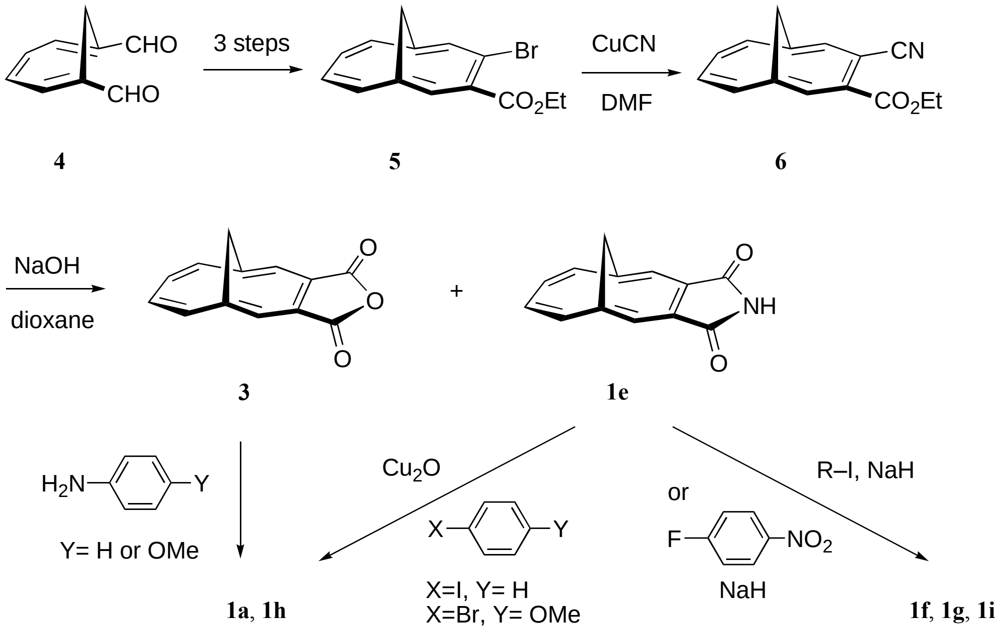
1,6-methano[10]annulene-3,4-dicarboximides **1**, which are structurally related to 2,3-naphtalimides. Recently Zuo *et al*. reported synthesis of **1a–d** from diethyl 1,6-methano[10]annulene-3,4-dicarboxylate (**2**) as shown in Scheme 1.[6] Their papers prompt us to report our independent synthetic efforts for the imides[7] and also emission properties thereof.[8]



**Scheme 1**

## RESULTS AND DISCUSSION

Our synthesis started with ethyl 4-bromo-1,6-methano[10]annulene-3-carboxylate (**5**), which was prepared in three steps from 1,3,5-cycloheptatriene-1,6-dicarbaldehyde (**4**)[9] by the Neidlein's method.[10] Cyanation of **5** with CuCN in DMF[11] gave **6** in 91% yield. Hydrolysis of **6** with NaOH in refluxing dioxane and subsequent acidification provided a mixture of **3** and **1e** in 51 and 29% yields, respectively (Scheme 2).



**Scheme 2**

Reactions of **3** with aniline and *p*-anisidine in refluxing xylene afforded **1a** and **1h** in 32 and 49% yields, respectively. These results confirm the data reported by Zuo *et al*.[6] On the other hand, alkylations of **1e** with methyl- and ethyliodides under basic conditions gave **1f** and **1g** in 90 and 88% yields, respectively, and the nucleophilic substitution of *p*-fluoronitrobenzene with **1e** in DMF provided **1i** in 94% yield.

Copper-catalyzed couplings[12] of **1e** with arylhalides were also examined. Iodobenzene and *p*-bromoanisole react with **1e** in the presence of copper(I) oxide in refluxing quinoline to give **1a** and **1h** in 63 and 91% yields, respectively, which are better than those from **3**.

Structures of all new compounds were characterized by spectroscopic and elemental or high-resolution mass spectrometric analyses. Hydrogens at the methylene bridge of the carboximides in their $^1$H NMR spectra are deshielded by the [10]annulene ring to appear at a range between $\delta$ 0.2 and –0.2. Their IR spectra show typical carbonyl stretching bands of a carboximide group at 1750–1760 and 1680-1690 cm$^{-1}$. The carboximides exhibited four absorption bands in UV-Vis spectra (Figure 1). Among them, the bands around 290 nm are very strong. All carboximides except **1h** exhibited emission upon excitation at the longest-wavelength absorptions. Figure 2 shows emission spectra of **1e** in methanol for example. Their photophysical data are shown in Table 1. Kossanyi *et al.* reported that the quantum yield of *N*-phenyl-2,3-

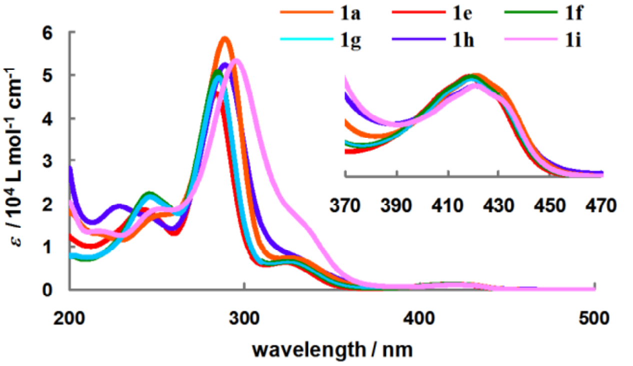


**Figure 1.** UV-Vis absorption spectra of **1a**,**1e**, **1f**, **1g**, **1h**, and **1i**. Inset is expanded spectra at a region of 370–470 nm. Methanol was used as a solvent for **1a**,**1e**, **1f**, **1g**, and **1h**, and acetonitrile for **1i**.

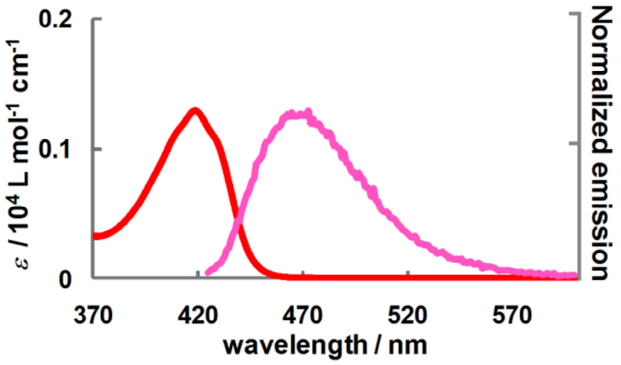


**Figure 2.** UV-Vis absorption and nomalized emission spectra of **1e**.

**Table 1.** Photophysical data of **1a**, **1e**, **1f**, **1g**, **1h**, and **1i**

| | $\lambda_{abs}$ (nm) | $\varepsilon^a$ | $\lambda_{emi}$ (nm) | Stokes shift (nm) | $\Phi^{emi}$ (%) |
|---|---|---|---|---|---|
| **1a**[b] | 421 | 0.13 | 472 | 51 | 8.5 |
| **1e**[b] | 418 | 0.13 | 468 | 50 | 0.13 |
| **1f**[b] | 419 | 0.13 | 470 | 51 | 9.7 |
| **1g**[b] | 419 | 0.13 | 470 | 51 | 9.6 |
| **1h**[b] | 421 | 0.12 | – | – | – |
| **1i**[c] | 421 | 0.12 | 467 | 46 | 5.5 |

[a] in 10$^4$ Lmol$^{-1}$cm$^{-1}$, [b] measured in MeOH, [c] measured in MeCN

naphtalimide (2 x $10^{-2}$%) was far smaller than those of the corresponding *N*-methyl derivative (24%) and the unsubstituted compound (26%).[1] On the other hand, the quantum yield of **1a** (8.5%) is comparable to those of **1f** (9.7%) and **1g** (9.6%) as shown in Table 1. Besides, it should be noted that the emission spectra of the carboximides **1** do not show dual emission as seen in 2,3-naphtalimides. These observations clearly reveal different emission properties of 1,6-methano[10]annulene-3,4-dicarboximides from those of 2,3-naphtalimides. Further studies on photochemical reactivity of the carboximides are now in progress.

## CONCLUSION

The title 1,6-methano[10]annulene-3,4-dicarboximides **1** were synthesized by the independent synthetic method involving cyanation and subsequent functional transformations starting from ethyl 4-bromo-1,6-methano[10]annulene-3-carboxylate (**5**). Their emission spectra showed different properties from those of structurally related 2,3-naphtalimides.

## EXPERIMENTAL

Melting points were measured on a Yanaco MP-3. IR spectra were recorded on a Perkin-Elmer Spectrum RX I spectrometer. UV-Vis spectra were measured on a Shimadzu UV-1600 spectrometer. Emission spectra were recorded in a degassed solution at rt on a Shimadzu RF-5300PC spectrometer. $^1$H and $^{13}$C-NMR were recorded with tetramethylsilane as internal standard on a JEOL α400 NMR instrument. Mass spectra were measured on a JMS-700 mass spectrometer. Column chromatography was done with Kieselgel 60. DMF and quinoline were purified by distillation from $CaH_2$. Ethyl 4-bromo-1,6-methano[10]annulene-3-carboxylate was prepared from 1,3,5-cycloheptatriene-1,6-dicarbaldehyde[9] according to a literature procedure.[10] Emission quantum yields were determined by comparison of a total emission area with that of anthracene ($\Phi$ = 0.27, upon excitation at 356 nm in ethanol). The refractive indices, $n$ = 1.33 (methanol), $n$ = 1.37 (ethanol), and $n$ = 1.35 (acetonitrile), were used in correction.

### Synthesis of ethyl 4-cyano-1,6-methano[10]annulene-3-carboxylate (6)

A suspension of 4.15 g (14.2 mM) of **5** and 5.08 g (56.7 mM) of cuprous cyanide in 40 mL of DMF was refluxed on an oil bath for 3 h under nitrogen atmosphere. After the reaction mixture was cooled to room temperature, solids formed were removed by filtration and the filtrate was evacuated under reduced pressure. The residue was purified by column chromatography. Elution with 20%AcOEt-hexane gave 3.15 g of **6** as a yellow oil (93% yield). $^1$H NMR ($CDCl_3$) $\delta$ = 8.33 (s, 1H), 7.90 (s, 1H), 7.40–7.65 (m, 4H), 4.49 (q, $J$ = 7.2 Hz, 2H), 1.47 (t, $J$ = 7.2 Hz, 3H), 0.04 (dt, $J$ = 9.4, 0.1, 1H), –0.18 (dt, $J$ = 9.4, 0.1 Hz, 1H) ppm; $^{13}$C NMR ($CDCl_3$) $\delta$ = 166.9, 137.2, 134.2, 129.8, 129.3, 129.2, 128.5, 128.5, 120.1, 115.1, 114.2, 107.8, 62.3, 34.3, 14.1 ppm; IR (KBr) $\nu_{max}$ = 3035w, 2983w, 2217m, 1719s, 1100s, 1050s, 870w, 830s, 773s cm$^{-1}$; MS *m/z* (rel int) 239 (M$^+$, 67), 211 (10), 194 (78), 167 (49), 166 (100), 140 (48), 139 (34); UV-Vis ($CH_3OH$) $\lambda_{max}$ = 271 (log$\varepsilon$ = 4.88), 403 (3.04) nm. HRMS *m/z* Calcd for $C_{15}H_{13}NO_2$ (M$^+$) 239.0946, found 239.0924.

### Hydrolysis of 6; Synthesis of 1,6-methano[10]annulene-3,4-dicarboximide (1e) and 1,6-methano[10]annulene-3,4-dicarboxylic anhydride (3)

A 15% NaOH aqueous solution (30 mL) was added nto a solution of 3.15 g (13.2 mM) of **6** in 25 mL of dioxane. This mixture was refluxed on an oil bath for 2 h. The resulted reaction mixture was poured in 100 mL of a saturated NaCl solution and was extracted with AcOEt (50 mL x 3). The combined organic layer was dried over $MgSO_4$. The solvent was evaporated to give yellow solids, which were collected and washed with ether to give 0.81 g (29% yield) of **1e**. The water layer was acidified with 3M HCl and the crystals formed were collected by filtration to give 1.41 g (51% yield) of **3**.

**1e**: Light yellow microcrystals, mp 248–250 ºC. $^1$H NMR ($CDCl_3$) $\delta$ = 8.27 (s, 2H), 7.74 (brt, 1H), 7.62 (m, 2H), 7.35 (m, 2H), 0.10 (d, *J* = 10.0 Hz, 1H), –0.20 (d, *J* = 10.0 Hz, 1H) ppm; $^{13}$C NMR ($CDCl_3$) $\delta$ = 169.7, 130.7, 130.2, 129.3, 128.9, 119.4, 35.3 ppm; IR (KBr) $\nu_{max}$ = 3528m, 3047m, 2955m, 1757vs, 1690vs, 1521s, 1331s, 1165s, 781s, 745s, 634m, 610w, 585m cm$^{-1}$; UV-Vis ($CH_3OH$) $\lambda_{max}$ = 242 (log$\varepsilon$ = 4.39), 285 (4.77), 324 (3.93), 419 (3.23) nm; MS *m/z* (rel int) 212 (M$^+$+1, 10), 211 (M$^+$, 67), 210 (10), 168 (28), 167 (17), 165 (12), 141 (12), 140 (100), 139 (70). *Anal.* Calcd for $C_{13}H_9NO_2$: C, 73.92; H, 4.29; N, 6.56%. Found C, 73.58; H, 4.44; N, 6.56%.

**3**: Creamy white prisms, mp 166–168 ˚C (lit.,[6] 165–166 ˚C). $^1$H-NMR ($CDCl_3$) $\delta$ = 8.41 (s, 2H), 7.73 (m, 2H), 7.42 (m, 2H), –0.08 (d, *J* = 10.0 Hz, 1H), –0.14 (d, *J* = 10.0 Hz, 1H) ppm; $^{13}$C-NMR($CDCl_3$) $\delta$ = 165.5, 132.4, 131.1, 130.0, 126.5, 120.9, 34.5 ppm; MS *m/z* (rel int) 212 (M$^+$, 35), 168 (32), 140 (100), 113 (12), 89 (15), 70 (19), 63 (23).

### Synthesis of *N*-methyl-, *N*-ethyl-, and *N*-*p*-nitrophenyl-1,6-methano[10]annulene-3,4-dicarboximides (1f, 1g, and 1i)

To a solution of 70.1 mg (0.332 mM) of **1e** in DMF was added 15.9 mg (0.390 mM) of 60% NaH in mineral oil. This mixture was stirred at rt under nitrogen atmosphere for 1h. Then, to this solution was added 31.0 μL (0.498 mM) of iodomethane. The resulted mixture was stirred at rt for further 2h, and then was poured into water. The solids formed were collected by filtration, washed with aqueous ethanol, and dried to give 67.0 mg (90% yield) of **1f**. An analytical sample was obtained by recrystallization from hexane-dichloromehane. By the similar method with iodoethane (rt, 3 h) and *p*-fluoronitrobenzene (120 ˚C, 2 h), **1g** and **1i** were obtained in 88 and 94% yields, respectively.

**1f**: Yellow microcrystals, mp 168–170 ºC. $^1$H NMR (CDCl$_3$) $\delta$ = 8.25 (s, 2H), 7.57 (m, 2H), 7.33 (m, 2H), 3.20 (s, 3H), 0.18 (d, $J$ = 9.6 Hz, 1H), –0.22 (d, $J$ = 9.6 Hz, 1H) ppm; $^{13}$C NMR (CDCl$_3$) $\delta$ = 170.2, 130.3, 129.8, 129.1, 128.5, 119.3, 35.6, 24.3 ppm. IR (KBr) $\nu_{max}$ = 3037w, 2956w, 2927w, 1750s, 1687vs, 1436s, 1384m, 1256w, 757s, 745s, 648m, 610w, 582m cm$^{-1}$; UV-Vis (CH$_3$OH) $\lambda_{max}$ = 246 (log$\varepsilon$ = 4.35), 286 (4.69), 325 (3.81), 420 (3.11) nm; MS *m/z* (rel int) 225 (M$^+$, 100), 224 (50), 197 (25), 181 (69), 168 (100), 166 (30), 141 (72). HRMS *m/z* Calcd for $C_{14}H_{11}NO_2$ (M$^+$) 225.0790, found 225.0770.

**1g**: Yellow microcrystals, mp 163–165 ºC. $^1$H NMR (CDCl$_3$) $\delta$ = 8.25 (s, 2H), 7.58 (m, 2H), 7.32 (m, 2H), 3.77 (q, $J$ = 7.2 Hz, 2H), 1.28 (t, $J$ = 7.2 Hz, 3H), 0.19 (d, $J$ = 10.0 Hz, 1H), –0.22 (d, $J$ = 10.0 Hz, 1H) ppm; $^{13}$C NMR (CDCl$_3$) $\delta$ = 170.0, 130.2, 129.8, 129.0, 128.6, 119.3, 35.5, 33.2, 13.8 ppm; IR (KBr) $\nu_{max}$ = 3036w, 2981w, 2947w, 1752s, 1689vs, 1402s, 1380m, 761m cm$^{-1}$; UV-Vis (CH$_3$OH) $\lambda_{max}$ = 247 (log$\varepsilon$ = 4.42), 286 (4.77), 326 (3.87), 420 (3.14) nm; MS *m/z* (rel int) 239 (M$^+$, 36), 224 (82), 168 (40), 167 (9), 140 (100), 139 (65), 115 (13). *Anal.* Calcd for $C_{15}H_{13}NO_2$: C, 75.30; H, 5.48; N, 5.85%, found C, 75.43; H, 5.45; N, 5.87%.

**1i**: Yellow solids, mp 268–270 ºC. $^1$H NMR (CDCl$_3$) $\delta$ = 8.41 (s, 2H), 8.37 (d, $J$ = 8.6 Hz, 2H), 7.82 (d, $J$ = 8.6 Hz, 2H), 7.67 (m, 2H), 7.39 (m, 2H), 0.19 (d, $J$ = 9.8 Hz, 1H), –0.10 (d, $J$ = 9.8 Hz, 1H) ppm; $^{13}$C NMR (CDCl$_3$) $\delta$ = 168.3, 146.3, 137.9, 131.4, 130.3, 129.6, 127.5, 126.3, 124.3, 120.0, 35.4 ppm; IR (KBr) $\nu_{max}$ = 3079w, 1759s, 1703vs, 1518m 1493m, 1371s, 1337s, 847m, 756m, 745m cm$^{-1}$; UV-Vis (CH$_3$CN) $\lambda_{max}$ = 251 (log$\varepsilon$ = 4.27), 295 (4.73), 332sh (4.21), 421 (3.07) nm; MS *m/z* (rel int) 332 (M$^+$, 66), 302 (10), 288 (17), 286 (10), 242 (23), 168 (43), 140 (100), 139 (72). HRMS *m/z* Calcd for $C_{19}H_{12}N_2O_4$ (M$^+$) 332.0797, found, 332.0773.

## Synthesis of *N*-phenyl- and *N*-*p*-methoxyphenyl-1,6-methano[10]annulene-3,4-dicarboximides (1a and 1h) from 3

A solution of 85.0 mg (0.401 mM) of **3** and 41.1 mg (0.441 mM) of aniline in 5 mL of xylene was refluxed on an oil bath for 5 h under nitrogen atmosphere. The solvent was removed under reduced pressure and the residue was purified by column chromatography (CHCl$_3$/hexane = 1/4) to give 37.1 mg (32% yield) of **1a**. Compound **1h** was obtained similarly with **3** and *p*-anisidine (49% yield).

**1a**: Yellow microcrystals, mp 158–159 ºC (lit.,[6] 170–171 ˚C).[13] $^1$H NMR (CDCl$_3$) $\delta$ = 8.37 (s, 2H), 7.64 (m, 2H), 7.50 (m, 3H), 7.47 (m, 2H), 7.36 (m, 2H), 0.21 (d, $J$ = 9.8 Hz, 1H), –0.14 (d, $J$ = 9.8 Hz, 1H) ppm; $^{13}$C NMR (CDCl$_3$) $\delta$ = 169.1, 130.9, 130.1, 129.2, 129.1, 129.0, 128.1, 128.0, 126.5, 119.6, 35.5 ppm; UV-Vis (CH$_3$OH) $\lambda_{max}$ = 253 (log$\varepsilon$ = 3.27), 290 (4.71), 327 (2.91), 422 (3.12) nm.

**1h**: Yellow microcrystals, mp 217–218 ºC. $^1$H NMR (CDCl$_3$) $\delta$ = 8.35 (s, 2H), 7.62 (m, 2H), 7.37 (d, $J$ = 8.8 Hz, 2H), 7.35 (m, 2H), 7.02 (d, $J$ = 8.8 Hz, 2H), 3.85 (s, 3H), 0.21 (d, $J$ = 9.8 Hz, 1H), –0.16 (d, $J$ = 9.8 Hz, 1H) ppm; $^{13}$C NMR (CDCl$_3$) $\delta$ = 169.3, 159.1, 130.8, 130.0, 129.2, 128.2, 127.8, 124.7, 119.6, 114.4, 55.5, 35.5 ppm; IR (KBr) $\nu_{max}$ = 3034w, 1756s, 1705vs, 1508s, 1387s, 1246s, 1154m, 1028m, 827m, 774m, 751m cm$^{-1}$; UV-Vis ($CH_3OH$) $\lambda_{max}$ = 229 (log$\varepsilon$ = 4.33), 289 (4.77), 338 (3.83), 421 (3.11) nm; MS *m/z* (rel int) 317 ($M^+$, 100), 302 (31), 168 (22), 140 (56), 139 (33). HRMS *m/z* Calcd for $C_{20}H_{15}NO_3$ ($M^+$) 317.1052, found 317.1049.

### Synthesis of *N*-phenyl- and *N*-*p*-methoxyphenyl-1,6-methano[10]annulene-3,4-dicarboximides (1a and 1h) from 1e

A suspension of 106 mg (0.500 mM) of **1e**, 112 mg (0.550 mM) of iodobenzene, and 36 mg (0.25 mM) of copper(I) oxide in 5 mL of quinoline was refluxed on an oil bath for 5 h under nitrogen atmosphere. Solids were removed by filtration. The solvent was removed under reduced pressure and the residue was purified by column chromatography ($CHCl_3$/hexane = 1/4) to give 90.4 mg (63% yield) of **1a**. By the similar way with *p*-bromoanisole and **1e**, **1h** was obtained in 91% yield.

## ACKNOWLEDGEMENTS

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