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REGIOSELECTIVITY IN DIRECT ARYLATION OF BENZANILIDE POSSESSING OXYGEN SUBSTITUENT IN THE BENZOYL PART USING PALLADIUM WITHOUT PHOSPHINE LIGAND

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Abstract – The direct arylation of benzanilide (**1**) possessing ether oxygen(s) in benzoyl part was examined under phosphine ligand-free conditions, which afforded *ortho*-product (**2**) and *para*-product (**3**) in very similar ratio to presence of P(*o*-Tol)₃ conditions. This results suggest that bulkiness of palladium under phosphine-free conditions would be comparable to that of P(*o*-Tol)₃ conditions.

Direct arylation (aryl-aryl coupling reaction)^{1c} of a nonactivated aryl C-H bond with an activated arene by palladium-phosphine reagent has been used to synthesize many condensed aromatic compounds.¹ We reported that an intramolecular direct arylation of 2-halo-*N*-arylbenzamides was a convenient method for synthesizing polycyclic aromatic lactams.² Moreover, we successfully synthesized benzonaphthazepine,³ pyrrolophenanthridine alkaloids,³ quinazoline alkaloids⁴ and benzpyranones^{4,5} utilizing palladium reagent. Recently, we reported that the oxygen substituent(s) of *N*-(2-iodophenyl)benzamide (**1**) and 2-iodo-*N*-methylbenzanilide (**A**) affected the regioselectivity of coupling position in the direct arylation using Pd-phosphine reagent and the ratio of *ortho*-product (**2**) to *para*-product (**3**) was influenced by the coordinating ability of oxygen substituent(s) to the Pd^{II} complex and by the steric relationship between the substituent(s) and the phosphine ligand in the complex (**B**).^{6,7}

It is well known that aryl iodide produces a coupling product without phosphine ligand.⁸ Then, we investigated whether the ratio of *ortho*-product (**2**) to *para*-product (**3**) is influenced by presence or absence of phosphine ligand in the aryl-aryl coupling reaction of benzanilide (**1**) possessing ether oxygen(s) in the benzoyl part.

The results of the coupling reactions using phosphine ligand-free Method A [$\text{Pd}(\text{OAc})_2$ (10 mol%), and K_2CO_3 (200 mol%)] in DMF under reflux are summarized in Table 1.

The phosphine-free conditions afforded *ortho*-product (**2**) and *para*-product (**3**) in very similar ratio to the $\text{P}(o\text{-Tol})_3$ conditions (Method C in Table 1). We proposed tentatively the δ -bond metathesis mechanism for the direct arylation.^{6b,6c,9} According to this hypothesis, this results indicate that bulkiness of palladium complex under phosphine-free conditions would be comparable to that of $\text{P}(o\text{-Tol})_3$ conditions by solvation as shown in **B** (L=solvent) in Scheme 1.

Direct arylation of benzanilide possessing other substituents under phosphine ligand-free is now in progress..

EXPERIMENTAL

Melting points were measured on a micro-melting point hot-stage apparatus (Yanagimoto) and are uncorrected. IR spectra were recorded on a JASCO FT/IR 350 spectrophotometer and ^1H -NMR spectra in deuteriochloroform on Varian Mercury 300 or VXR-500 spectrometers. NMR spectral data are reported in parts per million downfield from tetramethylsilane as the internal standard (δ 0.0), and the coupling constants are given in Hertz. MS spectra were obtained on a VG-70SE. Analytical HPLC was performed with a Shimadzu SPD-6A on a silica gel column (Chemcosorb 5Si-U). Column chromatography was carried out on a Merck silica gel (230–400 mesh). All the extracts were washed with brine, dried over anhydrous MgSO_4 , and filtered; the filtrate was concentrated to dryness under reduced pressure.

N-(2-Iodophenyl)-*N*-methyl-3,4-methylenedioxybenzamide (**1a**)

Methyl iodide (0.55 mL, 8.16 mmol) was added to a suspension of *N*-(2-iodophenyl)-3,4-methylenedioxybenzamide^{8b} (2.0 g, 5.44 mmol) and NaH (60%, 0.49 g, 16.2 mmol) in dry DMF (100 mL). After stirring for 15 min at rt, the excess NaH was decomposed with ice water, and extracted with Et_2O . The residue in Et_2O was subjected to column chromatography on a silica gel. Elution with hexane:AcOEt (4:1) gave **1a** (1.82 g, 89%) as colorless needles (from hexane), mp 95–96 °C (lit.,¹⁰ mp 97–97.5 °C).

3,4-Ethylenedioxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (**1b**)

A mixture of 3,4-ethylenedioxybenzoic acid¹¹ (0.50 g, 2.8 mmol) in five drops of dry DMF and thionyl chloride (0.3 mL, 4.2 mmol) was first refluxed for 15 min and then concentrated to dryness under reduced pressure. A solution of 2-iodo-*N*-methylaniline¹² (0.65 g, 3.36 mmol) in dry CH_2Cl_2 (3.5 mL) and dry NEt_3 (0.3 mL, 3.36 mmol) was added to this residue and the whole was stirred for 1.5 h at rt. The reaction mixture was concentrated to dryness and diluted with AcOEt, and then washed with 10% HCl and brine. The residue in CHCl_3 was subjected to column chromatography on silica gel. The elution with

hexane:AcOEt (1:1) gave **1b** (0.67 g, 61%) as colorless needles (from CHCl₃), mp 162–163 °C. IR (KBr) cm⁻¹: 1630. ¹H-NMR (300 MHz) δ : 3.34 (3H, s), 4.18 (4H, s), 6.59 (1H, br d, *J* = 7.8 Hz), 6.80 (1H, br d, *J* = 7.2 Hz), 6.93 (1H, br t, *J* = 7.5 Hz), 6.97 (1H, br s), 7.10 (1H, br d, *J* = 7.2 Hz), 7.25 (1H, br t, *J* = 7.5 Hz), 7.83 (1H, br d, *J* = 7.5 Hz). *Anal.* Calcd for C₁₆H₁₄NO₃I: C, 48.63; H, 3.57; N, 3.54. Found: C, 48.35; H, 3.60; N, 3.65.

3,4-Dimethoxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (**1c**)

A few drops of dry DMF and oxalyl chloride (2.0 mL, 21.6 mmol) were added to a solution of 3,4-dimethoxybenzoic acid (2.0g, 10.8 mmol) in dry CH₂Cl₂ (100 mL) and the mixture was stirred for 3 h under ice cooling. Then, the reaction mixture was concentrated to dryness under reduced pressure. A solution of 2-iodoaniline (2.40 g, 10.8 mmol) in dry CH₂Cl₂ (50 mL) and dry NEt₃ (1.8 mL, 12.9 mmol) was added to this residue and the whole was stirred for 3 h at rt. The reaction mixture was concentrated to dryness, diluted with CHCl₃, and then washed with 10% HCl, 5% aqueous NaOH solution, and brine. The residue in CHCl₃ was subjected to column chromatography on silica gel. Elution with hexane:AcOEt (2:1) gave 3,4-dimethoxy-*N*-(2-iodophenyl)benzamide (3.04 g, 72%) as colorless needles (from Et₂O), mp 154–156 °C. IR (KBr) cm⁻¹: 3250, 1640, 1515. ¹H-NMR (500 MHz) δ: 3.96 (3H, s), 3.98 (3H, s), 6.87 (1H, ddd, *J* = 8.0, 8.0, 1.3 Hz), 6.97 (1H, d, *J* = 8.3 Hz), 7.40 (1H, ddd, *J* = 8.0, 8.0, 1.1 Hz), 7.54 (1H, dd, *J* = 8.0, 1.3 Hz), 7.55 (1H, d, *J* = 1.3 Hz), 7.82 (1H, dd, *J* = 8.0, 1.1 Hz), 8.26 (1H, brs), 8.45 (1H, dd, *J* = 8.3, 1.3 Hz). *Anal.* Calcd for C₁₅H₁₄NO₃I: C, 47.02; H, 3.68; N, 3.66. Found: C, 47.00; H, 3.96; N, 3.53.

Methyl iodide (0.55 mL, 8.0 mmol) was added to a suspension of 3,4-dimethoxy-*N*-(2-iodophenyl)benzamide (2.00g, 5.48 mmol) and NaH (60%, 0.49 g, 16.2 mmol) in dry DMF (100 mL). After stirring for 15 min under ice cooling, the excess NaH was decomposed with ice water, and extracted with Et₂O. The residue in CHCl₃ was subjected to column chromatography on a silica gel. Elution with hexane:AcOEt (1:1) gave **1c** (1.82 g, 89%) as colorless needles (from CHCl₃-hexane), mp 125–126.5 °C. IR (KBr) cm⁻¹: 1630. ¹H-NMR (500 MHz) δ: 3.36 (3H, s), 3.70 (3H, s), 3.81 (3H, s), 6.65 (1H, d, *J* = 7.5 Hz), 6.90–6.92 (2H, m), 7.01 (1H, d, *J* = 7.5 Hz), 7.06 (1H, d, *J* = 7.5 Hz), 7.22 (1H, dd, *J* = 7.5, 7.5 Hz), 7.85 (1H, d, *J* = 7.5 Hz). *Anal.* Calcd for C₁₆H₁₆NO₃I: C, 48.38; H, 4.06; N, 3.53. Found : C, 48.34; H, 4.11; N, 3.45.

3-Methoxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (**1d**)

The compound (**1d**) was prepared according to reference 9.

3-Isopropoxy-4-methoxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (**1e**)

A mixture of 3-isopropoxy-4-methoxybenzoic acid (4.2 g, 20 mmol) in five drops of dry DMF and thionyl chloride (2.17 mL, 30 mmol) was first refluxed for 20 min and then concentrated to dryness under reduced pressure. A solution of 2-iodo-*N*-methylaniline (5.25 g, 26 mmol) in dry CH₂Cl₂ (30 mL) and dry

NEt₃ (3.62 mL, 26 mmol) was added to this residue and the whole was stirred for 40 min at rt. The reaction mixture was concentrated to dryness and diluted with AcOEt, and then washed with 10% HCl, 5% aqueous NaHCO₃ solution, and brine. The residue was recrystallized from Et₂O to afford 3-isopropoxy-4-methoxy-*N*-(2-iodophenyl)benzamide (6.32 g, 77 %) as colorless needles, mp 131-132 °C. IR (KBr) cm⁻¹: 1645, 1506. ¹H-NMR (300 MHz) δ: 1.42 (6H, d, *J* = 6.0 Hz), 3.93 (3H, s), 4.67 (1H, septet, *J* = 6.0 Hz), 6.87 (1H, ddd, *J* = 8.1, 7.8, 2.1 Hz), 6.97 (1H, d, *J* = 8.4 Hz), 7.40 (1H, ddd, *J* = 8.1, 8.1, 1.5 Hz), 7.53 (1H, dd, *J* = 8.1, 2.1 Hz), 7.56 (1H, d, *J* = 1.5 Hz), 7.81 (1H, dd, *J* = 7.8, 1.5 Hz), 8.24 (1H, br s), 8.45 (1H, dd, *J* = 8.4, 1.5 Hz). *Anal.* Calcd for C₁₇H₁₈NO₃I: C, 49.65; H, 4.41; N, 3.41. Found: C, 49.50; H, 4.33; N, 3.37.

Methyl iodide (1.0 mL, 15.0 mmol) was added to a suspension of 3-isopropoxy-4-methoxy-*N*-(2-iodophenyl)benzamide (4.11g, 10.0 mmol) and NaH (60%, 1.16 g, 30 mmol) in dry DMF (50 mL). After stirring for 45 min at rt, the excess NaH was decomposed with ice water, and extracted with AcOEt. The residue in CHCl₃ was subjected to column chromatography on a silica gel. Elution with hexane:AcOEt (4:1) gave **1e** (4.09 g, 96%) as a pale yellow oil. IR (KBr) cm⁻¹: 1645. ¹H-NMR (300 MHz) δ: 1.18 (3H, d, *J* = 6.0 Hz), 1.28 (3H, d, *J* = 6.0 Hz), 3.35 (3H, s), 3.78 (3H, s), 4.30 (1H, septet, *J* = 6.0 Hz), 6.66 (1H, d, *J* = 8.4 Hz), 6.87-7.25 (5H, m), 7.83 (1H, d, *J* = 6.9 Hz). High resolution MS (FAB) *m/z*: Calcd for C₁₈H₂₁NO₃I [M+1]⁺: 426.0566. Found: 426.0654.

3-*tert*-Butoxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (**1f**)

A solution of methyl 3-*tert*-butoxybenzoate (2.86 g, 13.7 mmol) in dry DMF (100 mL) was added to a suspension of NaH (60%, 1.22 g, 31.7 mmol) and 2-iodoaniline in dry DMF (30 mL) and the reaction mixture was stirred at rt for 72 h under an argone atmosphere. The excess NaH was decomposed with ice water, and extracted with AcOEt. The residue in CHCl₃ was subjected to column chromatography on a silica gel. Elution with hexane:AcOEt (5:1) gave 3-*tert*-butoxy-*N*-(2-iodophenyl)benzamide (4.50 g, 83%) as colorless needles (from Et₂O-hexane), mp 61–63 °C. IR (KBr) cm⁻¹: 3390, 1680, 1515. *Anal.* Calcd for C₁₇H₁₈NO₂I: C, 51.66; H, 4.59; N, 3.54. Found: C, 51.49; H, 4.61; N, 3.38.

Methyl iodide (1.74 mL, 26.0 mmol) was added to a suspension of 3-*tert*-butoxy-*N*-(2-iodophenyl)benzamide (4.11g, 10.4 mmol) and NaH (60%, 1.2 g, 31.2 mmol) in dry DMF (100 mL). After stirring for 45 min under ice cooling, the excess NaH was decomposed with ice water, and extracted with AcOEt. The residue in CHCl₃ was subjected to column chromatography on a silica gel. Elution with hexane:AcOEt (3:1) gave **1f** (3.29 g, 77%) as colorless prisms (from Et₂O-hexane), mp 183–185 °C. IR (KBr) cm⁻¹: 1630. ¹H-NMR (300 MHz) δ: 1.18 (9H, s), 3.37 (3H, s), 6.84-7.22 (6H, m), 7.80 (1H, d, *J* = 7.8 Hz). *Anal.* Calcd for C₁₈H₂₀NO₃I: C, 52.83; H, 4.93; N, 3.42. Found: C, 52.81; H, 5.08; N, 3.52.

General Procedure for the Direct Arylation of Benzamides (1)

Coupling reaction was carried out under the reaction conditions indicated in Table 1. Then, the reaction mixture was diluted with AcOEt, and the precipitates were removed by filtration. The filtrate was washed with brine.

Direct arylation of *N*-(2-iodophenyl)-*N*-methyl-3,4-methylenedioxybenzamide (1a)

The residue was dissolved in CHCl_3 and was subjected to column chromatography on silica gel. Elution with hexane:AcOEt (8:1) gave 5-methyl-9,10-methylenedioxyphenanthridin-6(5*H*)-one (2a) and successive elution with the same solvent gave 5-methyl-8,9-methylenedioxyphenanthridin-6(5*H*)-one (3a).

5-Methyl-9,10-methylenedioxyphenanthridin-6(5*H*)-one (2a): colorless needles (from CHCl_3), mp 179-181 °C. IR (KBr) cm^{-1} : 1600. $^1\text{H-NMR}$ (500 MHz) δ : 3.78 (3H, s), 6.28 (2H, s), 7.10 (1H, d, $J = 8.5$ Hz), 7.29 (1H, dd, $J = 7.8, 7.8$ Hz), 7.37 (1H, d, $J = 8.0$ Hz), 7.53 (1H, dd, $J = 7.8, 7.8$ Hz), 8.21 (1H, d, $J = 8.5$ Hz), 8.65 (1H, d, $J = 7.8$ Hz). *Anal.* Calcd for $\text{C}_{15}\text{H}_{11}\text{NO}_3$: C, 71.14; H, 4.38; N, 5.53. Found: C, 71.27; H, 4.29; N, 5.51.

5-Methyl-8,9-methylenedioxyphenanthridin-6(5*H*)-one (3a): colorless needles (from CHCl_3 -hexane), mp 245-247 °C (lit.,¹³ 238 °C). *Anal.* Calcd for $\text{C}_{15}\text{H}_{11}\text{NO}_3$: C, 71.14; H, 4.38; N, 5.53. Found: C, 71.07; H, 4.60; N, 5.47.

Direct arylation of 3,4-ethylenedioxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (1b)

The residue was dissolved in CHCl_3 and subjected to column chromatography on silica gel. Elution with hexane:AcOEt (2:1) gave 9,10-ethylenedioxy-5-methylphenanthridin-6(5*H*)-one (2b) and successive elution with the same solvent gave 8,9-methylenedioxy-5-methylphenanthridin-6(5*H*)-one (3b).

9,10-Ethylenedioxy-5-methylphenanthridin-6(5*H*)-one (2b): colorless needles (from CHCl_3 -hexane), mp 219-221 °C. IR (KBr) cm^{-1} : 1650. $^1\text{H-NMR}$ (300 MHz) δ : 3.81 (3H, s), 4.43-4.53 (4H, m), 7.13 (1H, d, $J = 8.7$ Hz), 7.28 (1H, ddd, $J = 8.4, 8.4, 1.2$ Hz), 7.41 (1H, dd, $J = 8.4, 1.2$ Hz), 7.53 (1H, ddd, $J = 8.4, 8.4, 1.5$ Hz), 8.19 (1H, d, $J = 8.7$ Hz), 9.20 (1H, dd, $J = 8.4, 1.5$ Hz). *Anal.* Calcd for $\text{C}_{16}\text{H}_{13}\text{NO}_3$: C, 71.90; H, 4.90; N, 5.24. Found: C, 71.66; H, 4.99; N, 5.36.

8,9-Ethylenedioxy-5-methylphenanthridin-6(5*H*)-one (3b): colorless needles (from CHCl_3 -hexane), mp 183-184 °C. IR (KBr) cm^{-1} : 1650. $^1\text{H-NMR}$ (300 MHz) δ : 3.80 (3H, s), 4.36-4.41 (4H, m), 7.28 (1H, br t, $J = 7.8, 6.9$ Hz), 7.38 (1H, br d, $J = 7.8$ Hz), 7.50 (1H, br t, $J = 7.8, 6.9$ Hz), 7.69 (1H, s), 8.02 (1H, s), 8.09 (1H, br d, $J = 7.8$ Hz). High resolution MS (FAB) m/z : Calcd for $\text{C}_{16}\text{H}_{14}\text{NO}_3$ $[\text{M}+1]^+$: 268.0974. Found: 268.1007.

Direct arylation of 3,4-dimethoxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (1c)

The residue was dissolved in CHCl_3 and subjected to column chromatography on silica gel. Elution with hexane:AcOEt (2:1) gave 9,10-dimethoxy-5-methylphenanthridin-6(5*H*)-one (2c) and successive elution

with the same solvent gave 8,9-dimethoxy-5-methylphenanthridin-6(5*H*)-one (**3c**).

9,10-Dimethoxy-5-methylphenanthridin-6(5*H*)-one (2c): colorless needles (from CHCl₃-hexane), mp 129-130 °C. IR (KBr) cm⁻¹: 1595. ¹H-NMR (500 MHz) δ: 3.79 (3H, s), 3.90 (3H, s), 4.03 (3H, s), 7.22 (1H, d, *J* = 8.6 Hz), 7.32 (1H, ddd, *J* = 7.9, 7.9, 1.3 Hz), 7.40 (1H, dd, *J* = 7.9, 1.3 Hz), 7.55 (1H, ddd, *J* = 7.9, 7.9, 1.4 Hz), 8.42 (1H, d, *J* = 8.6 Hz), 9.28 (1H, dd, *J* = 7.9, 1.4 Hz). High resolution MS (FAB) *m/z*: Calcd for C₁₆H₁₆NO₃ [M+1]⁺: 270.1130. Found: 270.1117.

8,9-Dimethoxy-5-methylphenanthridin-6(5*H*)-one (3c): colorless needles (from CHCl₃-hexane), mp 221.5-222.5 °C (lit.,¹⁴ 219-220 °C).

Direct arylation of 3-Isopropoxy-4-methoxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (1d)

The residue was dissolved in CHCl₃ and was subjected to column chromatography on silica gel. Elution with hexane:AcOEt:CHCl₃ (3:1:5) gave 8-isopropoxy-9-methoxy-5-methylphenanthridin-6(5*H*)-one (**3d**) and successive elution with the same solvent gave 10-isopropoxy-9-methoxy-5-methylphenanthridin-6(5*H*)-one (**2d**).

10-Isopropoxy-9-methoxy-5-methylphenanthridin-6(5*H*)-one (2d): yellow oil. IR (KBr) cm⁻¹: 1645. ¹H-NMR (300 MHz) δ: 1.29 (6H, d, *J* = 6.3 Hz), 3.79 (3H, s), 4.00 (3H, s), 4.55 (1H, sep, *J* = 6.3 Hz), 7.20 (1H, br d, *J* = 7.5 Hz), 7.27 (1H, br t, *J* = 7.5 Hz), 7.38 (1H, br d, *J* = 8.1 Hz), 7.52 (1H, br t, *J* = 7.2 Hz), 8.39 (1H, d, *J* = 8.1 Hz), 9.48 (1H, br d, *J* = 7.8 Hz). High resolution MS (FAB) *m/z*: Calcd for C₁₈H₂₁NO₃ [M+1]⁺: 298.1443. Found: 298.1446.

8-Isopropoxy-9-methoxy-5-methylphenanthridin-6(5*H*)-one (3d): colorless prisms (from CHCl₃-hexane), mp 160 °C. IR (KBr) cm⁻¹: 1644. ¹H-NMR (300 MHz) δ: 1.54 (6H, d, *J* = 6.0 Hz), 3.83 (3H, s), 4.07 (3H, s), 4.83 (1H, sep, *J* = 6.0 Hz), 7.32 (1H, br d, *J* = 7.2 Hz), 7.42 (1H, br d, *J* = 7.8 Hz), 7.52 (1H, br t, *J* = 7.4 Hz), 7.62 (1H, s), 7.98 (1H, s), 8.17 (1H, br d, *J* = 7.2 Hz). *Anal.* Calcd for C₁₈H₁₉NO₃: C, 72.71; H, 6.44; N, 4.71. Found: C, 72.71; H, 6.49; N, 4.75.

Direct arylation of 3-methoxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (1e)

The residue was dissolved in CHCl₃ and was subjected to column chromatography on silica gel. Elution with hexane:isopropyl ether (2:1) gave 10-methoxy-5-methylphenanthridin-6(5*H*)-one (**2e**)⁹ and successive elution with the same solvent gave 8-methoxy-5-methylphenanthridin-6(5*H*)-one (**3e**).⁹

Direct arylation of 3-*tert*-butoxy-*N*-(2-iodophenyl)-*N*-methylbenzamide (1f)

The residue was dissolved in CHCl₃ and subjected to column chromatography on silica gel. Elution with hexane:AcOEt (5:1) gave a mixture of coupling products, which was separated by preparative thin layer chromatography using CHCl₃:CH₂Cl₂:hexane (3:3:2). The upper zone gave 10-*tert*-butoxy-5-methylphenanthridin-6(5*H*)-one (**2f**) and the lower zone gave 8-*tert*-butoxy-5-methylphenanthridin-6(5*H*)-one (**3f**).

10-*tert*-Butoxy-5-methylphenanthridin-6(5*H*)-one (2f): colorless needles (from CHCl₃-hexane), mp 89-91 °C. IR (KBr) cm⁻¹: 1650. ¹H-NMR (300 MHz) δ: 1.43 (9H, s), 3.81 (3H, s), 7.25-7.54 (4H, m), 8.36 (1H, br d, *J* = 8.5 Hz), 9.52 (1H, dd, *J* = 8.3, 1.8 Hz). *Anal.* Calcd for C₁₈H₁₉NO₂·1/2H₂O: C, 74.46; H, 6.94; N, 4.82. Found: C, 74.58; H, 6.70; N, 4.88. FAB-MS (positive ion mode) *m/z*: 282 [M+1]⁺.

10-*tert*-Butoxy-5-methylphenanthridin-6(5*H*)-one (3f): colorless oil. IR (KBr) cm⁻¹: 1650. ¹H-NMR (300 MHz) δ: 1.45 (9H, s), 3.82 (3H, s), 7.31 (1H, dd, *J* = 7.5, 7.5 Hz), 7.37-7.54 (3H, m), 8.14-8.23 (2H, m). FAB-MS (positive ion mode) *m/z*: 282 [M+1]⁺.

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