

Direct Synthesis of Oxazolines from Olefins and Amides Using *tert*-BuOI

Supporting Information

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Experimental Procedures

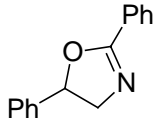
General Methods.

Melting points were determined on a Yanagimoto micro melting point apparatus and are uncorrected. IR spectra were obtained on a Jasco FT/IR-410 infrared spectrophotometer. ¹H and ¹³C NMR spectra were recorded on a JEOL FT-NMR JNM GSX-400 or EX-270 spectrometer (¹H NMR, 400/270 MHz; ¹³C NMR, 100/68 MHz) using tetramethylsilane as an internal standard. Mass spectra were measured with a Shimadzu Model GCMS-QP5000 spectrometer. High-resolution mass spectral data were obtained on a JEOL DX-303 mass spectrometer. Elemental analyses were performed at the Analytical Center, Faculty of Engineering, Osaka University. Flash column chromatography (FCC) was performed using silica gel FL60D (Fuji Silysia Chemical Co.). Preparative gel permeation liquid chromatography (GPLC) was performed on a JAI (Japan Analytical Industry) LC-908 instrument with JAIGEL 1H-2H columns and chloroform as the eluent. Analytical thin layer chromatography was performed using EM reagent 0.25 mm silica gel 60-F plates. Visualization was accomplished with UV light and spraying with an ethanolic phosphomolybdic acid solution followed by heating.

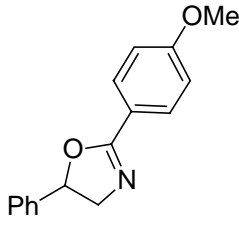
General procedure for the synthesis of oxazolines.

To a mixture of amide (0.5 mmol), olefin (1.0 mmol) and NaI (1.5 mmol) in MeCN (3 mL) was added *t*-BuOCl (1.5 mmol). The mixture was allowed to stir in the dark at room temperature for the indicated time under an atmosphere of nitrogen, quenched with 0.3M aqueous Na₂S₂O₃ (3 mL), the solution extracted with CH₂Cl₂, dried over MgSO₄, and concentrated under vacuum. The residue was purified by flash column chromatography on silica gel (eluent: hexane/ethyl acetate).

2.5-Diphenyl-2-oxazoline (1)

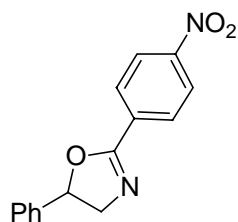
 According to the above general procedure, benzamide (60 mg, 0.50 mmol) and styrene (104 mg, 1.00 mmol) were used, and the title compound isolated (60 mg, 53%). Spectroscopic data were in agreement with previously reported data for the material.¹ Colorless oil; TLC R_f 0.21 (hexane/EtOAc, 4:1); ¹H NMR (CDCl₃, 270 MHz) δ 3.99 (dd, 1H, *J* = 7.9, 14.7 Hz, -NCHHCHO-), 4.48 (dd, 1H, *J* = 10.1, 14.7 Hz, -NCHHCHO-), 5.66 (dd, 1H, *J* = 7.9, 10.1 Hz, -NCH₂CHO-), 7.30-7.53 (m, 8H, ArH), 8.02 (d, 2H, *J* = 8.2 Hz, ArH).

2-(*p*-Methoxyphenyl)-5-phenyl-2-oxazoline (2a)

 According to the above general procedure, *p*-methoxybenzamide (76 mg, 0.50 mmol) and styrene (104 mg, 1.00 mmol) were used, and the title compound isolated (46 mg, 37%). Spectroscopic data were in agreement with previously reported data for the material.² Brown solid; mp 58-60 °C; TLC R_f 0.21 (hexane/EtOAc, 4:1); ¹H NMR (CDCl₃, 400 MHz) δ 3.84 (s, 3H, OMe), 3.96 (dd, 1H, *J* = 8.0,

14.9 Hz, -NCHHCHO-), 4.45 (dd, 1H, $J = 10.0, 14.9$ Hz, -NCHHCHO-), 5.62 (dd, 1H, $J = 8.0, 10.0$ Hz, -NCH₂CHO-), 6.93 (d, 2H, $J = 8.8$ Hz, ArH), 7.30-7.40 (m, 5H, ArH), 7.96 (d, 2H, $J = 8.8$ Hz, ArH).

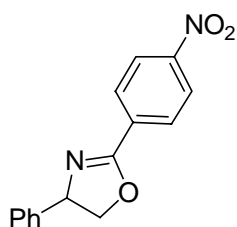
2-(*p*-Nitrophenyl)-5-phenyl-2-oxazoline (2b)



According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and styrene (108 mg, 1.04 mmol) were used, and the title compound isolated (90 mg, 67%). Spectroscopic data were in agreement

with previously reported data for the material.³ Pale orange solid; mp 153-156 °C; TLC R_f 0.19 (hexane/EtOAc, 4:1); ¹H NMR (CDCl₃, 270 MHz) δ 4.06 (dd, 1H, $J = 8.1, 15.5$ Hz, -NCHHCHO-), 4.54 (dd, 1H, $J = 10.2, 15.5$ Hz, -NCHHCHO-), 5.72 (dd, 1H, $J = 8.1, 10.2$ Hz, -NCH₂CHO-), 7.30-7.47 (m, 5H, ArH), 8.18 (d, 2H, $J = 8.6$ Hz, ArH), 8.29 (d, 2H, $J = 8.6$ Hz, ArH).

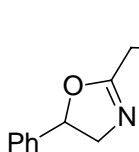
2-(*p*-Nitrophenyl)-4-phenyl-2-oxazoline (3b)



According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and styrene (108 mg, 1.04 mmol) were used, and the title compound isolated (15 mg, 11%). Spectroscopic data were in agreement with previously reported data for the material.⁴ Brown solid; mp 104-107

°C; TLC R_f 0.26 (hexane/EtOAc, 4:1); ¹H NMR (CDCl₃, 400 MHz) δ 4.36 (dd, 1H, $J = 8.4, 8.4$ Hz, -OCHHCHN-), 4.87 (dd, 1H, $J = 8.4, 10.5$ Hz, -OCHHCHN-), 5.45 (dd, 1H, $J = 8.4, 10.5$ Hz, -OCH₂CHN-), 7.29-7.41 (m, 5H, ArH), 8.22 (d, 2H, $J = 8.8$ Hz, ArH), 8.30 (d, 2H, $J = 8.8$ Hz, ArH).

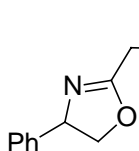
2-(*n*-Butyl)-5-phenyl-2-oxazoline (2c)



According to the above general procedure, *n*-valeramide (50 mg, 0.50 mmol) and styrene (103 mg, 0.99 mmol) were used, and the title compound isolated (24 mg, 24%). Yellow oil; TLC R_f 0.19 (hexane/EtOAc, 4:1); IR (KBr, cm^{-1})

3350, 3070, 2962, 1738, 1647, 1551, 1454, 1340, 1255, 1196, 1057; ^1H NMR (CDCl_3 , 400 MHz) δ 0.94 (t, 3H, $J = 7.6$ Hz, $-\text{CH}_2\text{CH}_3$), 1.42 (tq, 2H, $J = 7.6, 7.6$ Hz, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 1.69 (tt, 2H, $J = 7.6, 7.6$ Hz, $-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 2.37 (t, 2H, $J = 7.6$ Hz, $-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 3.75 (dd, 1H, $J = 8.0, 14.4$ Hz, $-\text{NCHHCHO-}$), 4.23 (dd, 1H, $J = 10.4, 14.4$ Hz, $-\text{NCHHCHO-}$), 5.44 (dd, 1H, $J = 8.0, 10.4$ Hz, $-\text{NCH}_2\text{CHO-}$), 7.27-7.39 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 13.7, 22.3, 27.8, 28.0, 62.5, 80.5, 125.6, 128.1, 128.6, 141.1, 168.1; MS (CI, isobutane) m/z (relative intensity, %) 204 ($\text{M}^+ + \text{H}$, 100); HRMS (CI, isobutane) calcd for $\text{C}_{17}\text{H}_{15}\text{N}_2\text{O}_3$ ($\text{M} + \text{H}$) $^+$ 204.1388, found 204.1383.

2-(*n*-Butyl)-4-phenyl-2-oxazoline (3c)

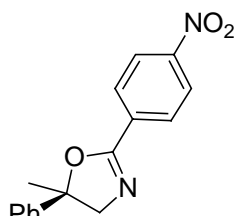


According to the above general procedure, *n*-valeramide (50 mg, 0.50 mmol) and styrene (103 mg, 0.99 mmol) were used, and the title compound isolated (4 mg, 4%). Yellow oil; TLC R_f 0.27 (hexane/EtOAc, 4:1); IR (neat, cm^{-1}) 3292,

3068, 2956, 1734, 1645, 1543, 1456, 1379, 1263, 1178, 1076; ^1H NMR (CDCl_3 , 400 MHz) δ 0.95 (t, 3H, $J = 7.6$ Hz, $-\text{CH}_2\text{CH}_3$), 1.43 (tq, 2H, $J = 7.6, 7.6$ Hz, $-\text{CH}_2\text{CH}_2\text{CH}_3$), 1.69 (tt, 2H, $J = 7.6, 7.6$ Hz, $-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 2.40 (t, 2H, $J = 7.6$ Hz, $-\text{CH}_2\text{CH}_2\text{CH}_2\text{CH}_3$), 4.06 (dd, 1H, $J = 8.4, 8.4$ Hz, $-\text{OCHHCHN-}$), 4.59 (dd, 1H, $J = 8.4, 9.4$ Hz, $-\text{OCHHCHN-}$), 5.16 (dd, 1H, $J = 8.4, 9.4$ Hz, $-\text{OCH}_2\text{CHN-}$), 7.23-7.38 (m, 5H, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 13.7, 22.3, 27.7, 28.2, 69.5,

74.4, 126.5, 127.4, 128.6, 142.5, 169.0; MS (CI, isobutane) m/z (relative intensity, %) 204 ($M^+ + H$, 100); HRMS (CI, isobutane) calcd for $C_{17}H_{15}N_2O_3$ ($M+H$)⁺ 204.1388, found 204.1377.

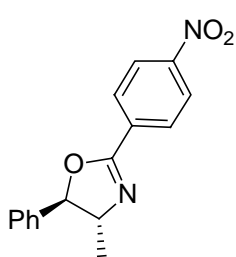
5-Methyl-2-(*p*-nitrophenyl)-5-phenyl-2-oxazoline (3d)



According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and α -methylstyrene (121 mg, 1.02 mmol) were used, and the title compound isolated (112 mg, 79%). White solid; mp 81-82

°C; TLC R_f 0.11 (hexane/EtOAc, 4:1); IR (KBr, cm^{-1}) 3435, 3066, 2976, 1645, 1595, 1520, 1346, 1271, 1066; 1H NMR ($CDCl_3$, 400 MHz) δ 1.82 (s, 3H, $-CH_3$), 4.21 (s, 2H, $-NCH_2CO-$), 7.26-7.40 (m, 5H, *ArH*), 8.20 (d, 2H, $J = 8.8$ Hz, *ArH*), 8.29 (d, 2H, $J = 8.8$ Hz, *ArH*); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 28.3, 69.0, 87.7, 123.5, 124.1, 127.5, 128.5, 129.0, 133.6, 144.7, 149.3, 161.0; MS (EI) m/z (relative intensity, %) 282 (M^+ , 46), 162 (100), 146 (10); HRMS (EI) calcd for $C_{16}H_{14}N_2O_3$ (M)⁺ 283.1004, found 282.1009.

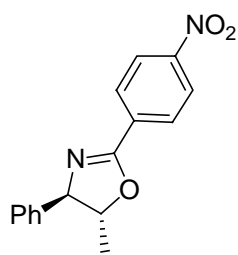
4-Methyl-2-(*p*-nitrophenyl)-5-phenyl-2-oxazoline (2e)



According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and *trans*- β -methylstyrene (118 mg, 1.00 mmol) were used, and the title compound isolated (93 mg, 65%). Spectroscopic data were in agreement with previously reported data for the material.⁵ Orange solid; mp

70-72 °C; TLC R_f 0.29 (hexane/EtOAc, 4:1); 1H NMR ($CDCl_3$, 270 MHz) δ 1.51 (d, 3H, $J = 6.8$ Hz, $-CHCH_3$), 4.28 (dq, 1H, $J = 6.8, 6.8$ Hz, $-OCHCHN-$), 5.16 (d, 1H, $J = 6.8$ Hz, $-OCHCHN-$), 7.30-7.45 (m, 5H, *ArH*), 8.19 (d, 2H, $J = 8.8$ Hz, *ArH*), 8.29 (d, 2H, $J = 8.8$ Hz, *ArH*).

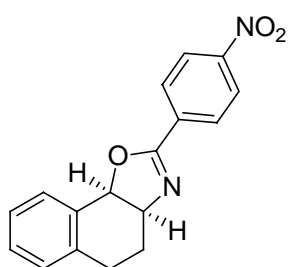
5-Methyl-2-(*p*-nitrophenyl)-4-phenyl-2-oxazoline (3e)



According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and *trans*- β -methylstyrene (118 mg, 1.00 mmol) were used, and the title compound isolated (10 mg, 7%). White solid; mp 69-70 °C; TLC R_f 0.37 (hexane/EtOAc, 4:1); IR (KBr, cm^{-1}) 3438, 2924, 1645, 1599,

1525, 1346, 1064; ^1H NMR (CDCl_3 , 400 MHz) δ 1.60 (d, 3H, $J = 6.4$ Hz, $-\text{CHCH}_3$), 4.67 (dq, 1H, $J = 6.4, 7.6$ Hz, $-\text{OCHCHN}-$), 4.90 (d, 1H, $J = 7.6$ Hz, $-\text{OCHCHN}-$), 7.27-7.40 (m, 5H, ArH), 8.21 (d, 2H, $J = 8.4$ Hz, ArH), 8.29 (d, 2H, $J = 8.4$ Hz, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 20.7, 77.5, 84.9, 123.5, 126.6, 127.9, 128.9, 129.4, 133.7, 141.3, 149.6, 162.2; MS (EI) m/z (relative intensity, %) 282 (M^+ , 3), 267 ($\text{M}^+ - \text{Me}$, 1), 238 (100), 192 (39), 165 (16); HRMS (CI, isobutane) calcd for $\text{C}_{16}\text{H}_{15}\text{N}_2\text{O}_3$ ($\text{M} + \text{H}$) $^+$ 283.1083, found 283.1089.

cis-2-(*p*-Nitrophenyl)-3a,4,5,9b-tetrahydronaphtho[2,1-*d*]oxazole (2f)

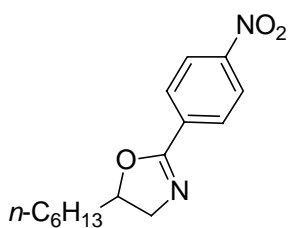


According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and 1,2-dihydronaphthalene (130 mg, 1.00 mmol) were used, and the title compound isolated (71 mg, 55%). Yellow solid; mp 69-70 °C; TLC R_f 0.09 (hexane/EtOAc, 4:1); IR (KBr, cm^{-1}) 3384, 3068, 2939,

1641, 1595, 1520, 1444, 1342, 1072; ^1H NMR (CDCl_3 , 400 MHz) δ 2.03-2.12 (m, 2H, $-\text{NCHCH}_2\text{CH}_2-$), 2.61 (ddd, 1H, $J = 5.0, 5.0, 15.5$ Hz, $-\text{NCHCH}_2\text{CHH}-$), 2.77 (ddd, 1H, $J = 5.0, 9.9, 15.5$ Hz, $-\text{NCHCH}_2\text{CHH}-$), 4.79 (ddd, 1H, $J = 5.0, 5.0, 9.8$ Hz, $-\text{NCHCHO}-$), 5.74 (d, 1H, $J = 9.8$ Hz, $-\text{NCHCHO}-$), 7.16-7.18 (m, 1H, ArH), 7.27-7.32 (m, 2H, ArH), 7.47-7.50 (m, 1H, ArH), 8.10 (d, 2H, $J = 8.4$ Hz, ArH), 8.22 (d, 2H, $J = 8.4$ Hz, ArH); ^{13}C NMR (CDCl_3 , 100 MHz) δ 25.6, 28.3, 65.1, 79.6, 123.4, 126.8, 128.4, 128.8, 129.2, 130.5, 132.0, 133.5, 139.6, 149.4, 162.3; MS

(CI, isobutane) m/z (relative intensity, %) 295 ($M^+ + H$, 100); HRMS (CI, isobutane) calcd for $C_{17}H_{15}N_2O_3$ ($M+H$)⁺ 295.1083, found 295.1088.

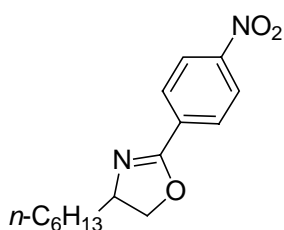
5-(*n*-Hexyl)-2-(*p*-nitrophenyl)-2-oxazoline (2g)



According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and 1-octene (108 mg, 0.96 mmol) were used, and the title compound isolated (49 mg, 35%). White solid; mp 77-79 °C; TLC R_f 0.19 (hexane/EtOAc, 4:1); IR (KBr, cm^{-1}) 3435, 2925, 1647,

1599, 1522, 1348, 1076; 1H NMR ($CDCl_3$, 400 MHz) δ 0.90 (t, 3H, $J = 6.5$ Hz, $-CH_2CH_3$), 1.24-1.56 (m, 8H, $-CH_2CH_2CH_2CH_2CH_2CH_3$), 1.56-1.88 (m, 2H, $-CH_2-(CH_2)_4-CH_3$), 3.71 (dd, 1H, $J = 7.6, 15.1$ Hz, $-NCHHCHO-$), 4.17 (dd, 1H, $J = 9.6, 15.1$ Hz, $-NCHHCHO-$), 4.72-4.83 (m, 1H, $-NCH_2CHO-$), 8.11 (d, 2H, $J = 8.4$ Hz, ArH), 8.27 (d, 2H, $J = 8.4$ Hz, ArH); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 14.1, 22.5, 25.1, 29.0, 31.7, 35.4, 60.3, 80.9, 123.5, 129.1, 133.9, 149.4, 162.2; MS (EI) m/z (relative intensity, %) 276 (M^+ , 5), 205 ($M^+ - n-C_5H_{11}$, 11), 191 ($M^+ - n-C_6H_{13}$, 28), 162 (94), 150 (63); HRMS (CI, isobutane) calcd for $C_{15}H_{21}N_2O_3$ ($M+H$)⁺ 277.1552, found 277.1557. Anal. Calcd for $C_{15}H_{20}N_2O_3$: C, 65.20; H, 7.30; N, 10.14. Found: C, 64.91; H, 7.27; N, 9.91.

4-(*n*-Hexyl)-2-(*p*-nitrophenyl)-2-oxazoline (3g)

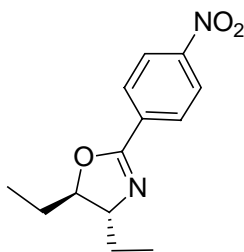


According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and 1-octene (108 mg, 0.96 mmol) were used, and the title compound isolated (21 mg, 15%). White solid; mp 48-50 °C; TLC R_f 0.39 (hexane/EtOAc, 4:1); IR (KBr, cm^{-1}) 3435, 2925, 1649,

1525, 1348, 1070; 1H NMR ($CDCl_3$, 270 MHz) δ 0.89 (t, 3H, $J = 6.6$ Hz, $-CH_2CH_3$), 1.30-1.63 (m,

9H, $-CHHCH_2CH_2CH_2CH_2CH_3$), 1.70-1.77 (m, 1H, $-CHH-(CH_2)_4-CH_3$), 4.09 (dd, 1H, $J = 8.1$, 8.1 Hz, $-OCHHCHN-$), 4.27-4.38 (m, 1H, $-OCH_2CHN-$), 4.55 (dd, 1H, $J = 8.1$, 9.5 Hz, $-OCHHCHN-$), 8.11 (d, 2H, $J = 9.2$ Hz, ArH), 8.26 (d, 2H, $J = 9.2$ Hz, ArH); ^{13}C NMR (CDCl₃, 68 MHz) δ 14.1, 22.6, 25.9, 29.2, 31.7, 35.8, 67.2, 73.1, 123.5, 129.2, 133.8, 149.4, 161.6; MS (EI) m/z (relative intensity, %) 276 (M^+ , 1), 206 (32), 191 ($M^+ - n-C_6H_{13}$, 89), 167 (39), 163 (28), 150 (69); HRMS (CI, isobutane) calcd for C₁₅H₂₁N₂O₃ ($M+H$)⁺ 277.1552, found 277.1550.

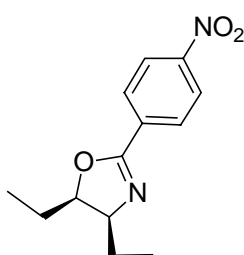
***trans*-4,5-Diethyl-2-(*p*-nitrophenyl)-2-oxazoline (2h)**



According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and *trans*-3-hexene (84 mg, 0.99 mmol) were used, and the title compound isolated (89 mg, 71%). Spectroscopic data were in agreement with previously reported data for the material.⁶ Yellow solid; mp

47-50 °C; TLC R_f 0.31 (hexane/EtOAc, 4:1); 1H NMR (CDCl₃, 270 MHz) δ 1.02 (t, 3H, $J = 7.4$ Hz, $-CH_2CH_3$), 1.04 (t, 3H, $J = 7.3$ Hz, $-CH_2CH_3$), 1.56-1.81 (m, 4H, $CH_3CH_2CHCHCH_2CH_3$), 3.84 (dt, 1H, $J = 6.4$, 6.4 Hz, $-NCHCHO-$), 4.30 (dt, 1H, $J = 6.4$, 6.4 Hz, $-NCHCHO-$), 8.12 (d, 2H, $J = 9.0$ Hz, ArH), 8.26 (d, 2H, $J = 9.0$ Hz, ArH); ^{13}C NMR (CDCl₃, 68 MHz) δ 9.2, 9.9, 28.2, 28.5, 73.0, 86.4, 123.4, 129.1, 134.0, 149.3, 160.9; MS (EI) m/z (relative intensity, %) 248 (M^+ , 1), 219 ($M^+ - Et$, 62), 190 (22), 175 (40), 149 (18).

***cis*-4,5-Diethyl-2-(*p*-nitrophenyl)-2-oxazoline (2i)**



According to the above general procedure, *p*-nitrobenzamide (83 mg, 0.50 mmol) and *cis*-3-hexene (84 mg, 1.00 mmol) were used, and the title compound isolated (85 mg, 68%). Spectroscopic data were in

agreement with previously reported data for the material.⁶ Orange solid; mp 78-81 °C; TLC R_f 0.29 (hexane/EtOAc, 4:1); ^1H NMR (CDCl_3 , 270 MHz) δ 1.12 (t, 3H, $J = 7.3$ Hz, $-\text{CH}_2\text{CH}_3$), 1.13 (t, 3H, $J = 7.3$ Hz, $-\text{CH}_2\text{CH}_3$), 1.47-1.77 (m, 4H, $\text{CH}_3\text{CH}_2\text{CHCHCH}_2\text{CH}_3$), 4.14 (dt, 1H, $J = 5.1$, 9.3 Hz, $-\text{NCHCHO}-$), 4.65 (dt, 1H, $J = 5.1$, 8.9 Hz, $-\text{NCHCHO}-$), 8.13 (d, 2H, $J = 9.0$ Hz, ArH), 8.26 (d, 2H, $J = 9.0$ Hz, ArH); ^{13}C NMR (CDCl_3 , 68 MHz) δ 11.2, 11.8, 22.5, 23.3, 70.2, 85.2, 123.4, 129.1, 134.2, 149.3, 161.0; MS (EI) m/z (relative intensity, %) 248 (M^+ , 2), 219 ($\text{M}^+ - \text{Et}$, 60), 190 (37), 175 (56), 149 (18).

¹H NMR spectra of reaction between benzamide and *t*-BuOI as mentioned in the text (ref. 11)

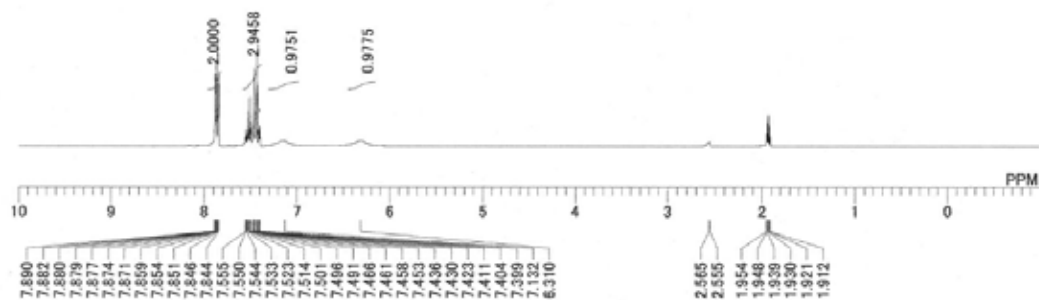
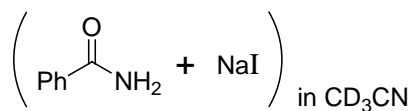
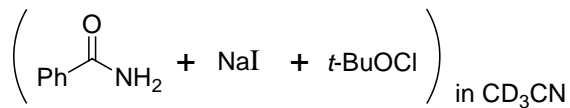


Figure SI-1. ¹H-NMR spectrum of benzamide (0.02 mmol) and NaI (0.02 mmol) in CD₃CN (0.5 mL) at room temperature.



after 30 min

Change of the chemical shift corresponding to the phenyl group

Disappearance of the signals of N-H

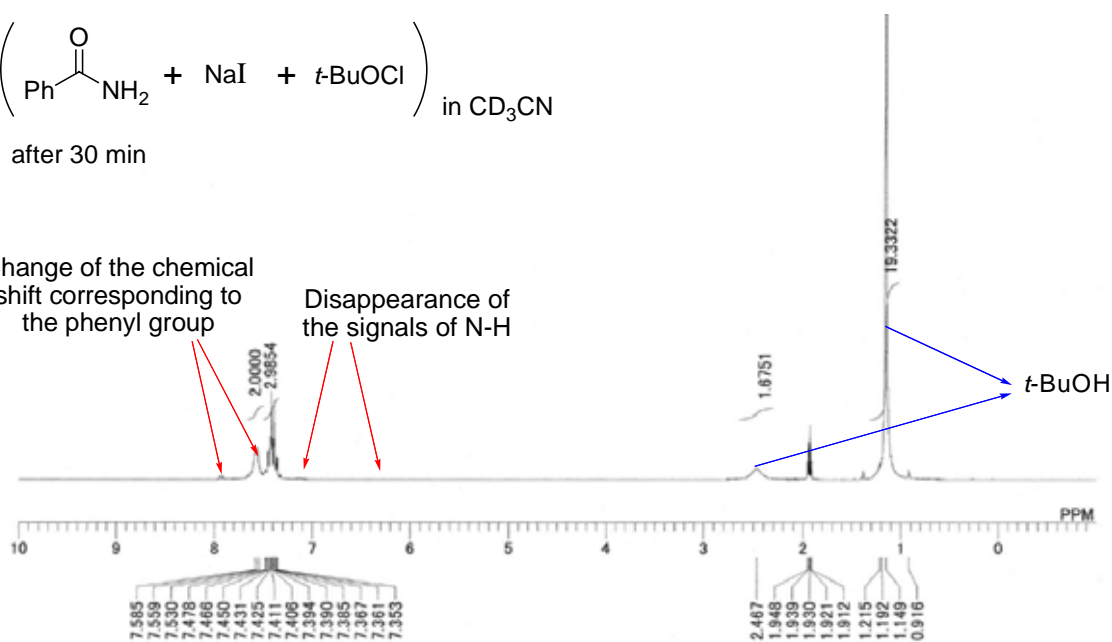


Figure SI-2. ¹H-NMR spectrum after treating the above mixture with *t*-BuOCl (0.02 mmol) in CD₃CN at room temperature.

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