Supporting Information

Palladium-Catalyzed Direct C−H Arylation of Pyridine *N*-oxides with Potassium Aryl- and Heteroaryltrifluoroborates

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CONTENTS:

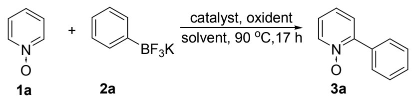
I. General

Unless otherwise noted, all reagents were obtained from commercial suppliers and used without further purification. Deuterated solvents were purchased from Aldrich. Refinement of the mixted system through Column chromatography which was performed on silica gel (200-300 mesh) with ethyl acetate (solvent A)/alcohol (solvent B) gradients as elution. In addition, all yields were referred to isolated yields (average of two runs) of compounds unless otherwise specified. On the one hand, the known compounds were partly characterized by melting points (for solid samples), ^{1}H NMR, and compared to authentic samples or the literature data. Melting points were determined with a RD-II digital melting point apparatus and were uncorrected. ^{1}H NMR data were obtained at 300 K on a Bruker AMX-600 spectrometer. The ^{1}H NMR (600 MHz) chemical shifts were measured relative to $CDCl_3$ as the internal reference ($CDCl_3$: δ = 7.26 ppm). Spectra are reported as follows: chemical shift (δ = ppm), multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet), coupling constants (Hz), integration, and assignment. On the other hand, the unknown compounds were partly characterized by ^{13}C NMR and HR-MS as well. The ^{13}C NMR (100 MHz) chemical shifts were given using $CDCl_3$ as the internal standard ($CDCl_3$: δ = 77.16 ppm). High-resolution mass spectra (HR-MS) were obtained with a Waters-Q-TOF-Premier (ESI).

II. Optimization of the other reaction parameters

An extensive survey of catalysts was conducted and the results are listed in Table *S1*. No reaction was detected in the absence of a catalyst (Table *S1*, entry 1). Using $Pd(OAc)_2$ as the catalyst resulted in 60% yield of **3a** (Table *S1*, entry 2). Inferior yields were obtained with $Cu(OAc)_2$, $Ni(OAc)_2$**.**$4H_2O$ and $FeCl_3$ (Table *S1*, entries 3-5). Unfortunately, other Pd catalysts including $PdCl_2$, Pd (dppf) Cl_2, Pd (PhCN) Cl_2, $Pd(PPh_3)_2Cl_2$, Pd(dppe)Cl_2, $Pd(PPh_3)_4$ and $Pd(OAc)_2(PPh_3)$ all gave lower yields (Table *S1*, entries 6-12 *vs.* 2). The examination of oxidants demonstrated Oxone, TBHP, I_2, MCPBA, $KMnO_4$, and $Cu(OAc)_2$ provided inferior results (Table *S1*, entries 14-19). When 1,4-dioxane was replaced with DMF, DMSO and NMP, the yield of **3a** was dramatically decreased (Table *S1*, entries 20-22 *vs.* 2).

Table *S1* Optimization of the catalysts, oxidants and solvents for the reaction[a]



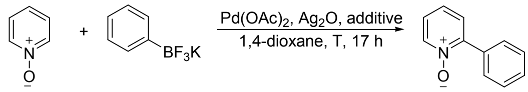
Entry	Catalyst	Oxidant	Solvent	**3a** (%)[b]
1	-	Ag_2O	1,4-dioxane	n.r.[c]
2	**$Pd(OAc)_2$**	**Ag_2O**	**1,4-dioxane**	**60**
3	$Cu(OAc)_2$	Ag_2O	1,4-dioxane	9

4	$Ni(OAc)_2.4H_2O$	Ag_2O	1,4-dioxane	7
5	$FeCl_3$	Ag_2O	1,4-dioxane	trace
6	$PdCl_2$	Ag_2O	1,4-dioxane	34
7	Pd (dppf)Cl_2	Ag_2O	1,4-dioxane	19
8	Pd (PhCN)Cl_2	Ag_2O	1,4-dioxane	46
9	$Pd(PPh_3)_2Cl_2$	Ag_2O	1,4-dioxane	44
10	Pd(dppe)Cl_2	Ag_2O	1,4-dioxane	39
11	$Pd(PPh_3)_4$	Ag_2O	1,4-dioxane	29
12	$Pd(OAc)_2(PPh_3)$	Ag_2O	1,4-dioxane	51
13	$Pd(OAc)_2$	-	1,4-dioxane	trace
14	$Pd(OAc)_2$	Oxone	1,4-dioxane	n.r.
15	$Pd(OAc)_2$	TBHP	1,4-dioxane	16
16	$Pd(OAc)_2$	I_2	1,4-dioxane	n.r.
17	$Pd(OAc)_2$	MCPBA	1,4-dioxane	n.r.
18	$Pd(OAc)_2$	$KMnO_4$	1,4-dioxane	7
19	$Pd(OAc)_2$	$Cu(OAc)_2$	1,4-dioxane	trace
20	$Pd(OAc)_2$	Ag_2O	DMF	27
21	$Pd(OAc)_2$	Ag_2O	DMSO	trace
22	$Pd(OAc)_2$	Ag_2O	NMP	29

[a] Reactions were carried out with pyridine *N*-oxide **1a** (0.45 mmol), potassium phenyltrifluoroborate **2a** (0.15 mmol), oxidant (0.3 mmol, 2 equiv.) and catalyst (10 mol%) in solvent (0.5 mL) at 90 °C for 17 h. [b] Isolated yields. [c] n.r. = no reaction.

The examination of catalyst loading indicated that 10 mol % was the most suitable (Table *S2*, entry 1 *vs.* 2 and 3). 2.0 equiv. of Ag_2O exhibited the better results (Table *S2*, entry 5 *vs.* 4 and 6). It was found that up to 95% yield was obtained when 3.3 equiv of pyridine *N*-oxide **1a** was used at 90 °C (Table *S2*, entry 9 *vs.*7 and 8). Lower yields were offered when using other temperature (Table *S2*, entry 9 *vs.* 10 and 11).

Table *S2* Optimization of other reaction conditions



1a **2a** **3a**

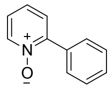
Entry	$Pd(OAc)_2$ (mol%)	Ag_2O (equiv.)	**1a** (equiv.)	T (°C)	Yield (%)[a]
1[b]	10	2.0	3	90	60
2[b]	5	2.0	3	90	56
3[b]	15	2.0	3	90	57
4[c]	10	1.5	3	90	50
5[c]	10	2.0	3	90	60
6[c]	10	2.5	3	90	55
7[d]	10	2.0	3	90	88
8[d]	10	2.0	2.5	90	78
9[d]	***10***	2.0	***3.3***	***90***	***95***
10[e]	10	2.0	3.3	80	92
11[e]	10	2.0	3.3	100	80

[a] Isolated yields. [b] Reactions were carried out with pyridine *N*-oxide **1a** (0.45 mmol, 3 equiv), potassium

phenyltrifluoroborate **2a** (0.15 mmol), Ag_2O (0.3 mmol, 2 equiv) and $Pd(OAc)_2$ in 1,4-dioxane (0.5 mL) at 90 °C for 17 h. [c] Reactions were carried out with pyridine *N*-oxide **1a** (0.45 mmol, 3 equiv), potassium phenyltrifluoroborate **2a** (0.15 mmol), Ag_2O and $Pd(OAc)_2$ (10 mol %) in 1,4-dioxane (0.5 mL) at 90 °C for 17 h. [d] Reactions were carried out with pyridine *N*-oxide **1a**, potassium phenyltrifluoroborate **2a** (0.15 mmol), Ag_2O (0.3 mmol, 2 equiv.) $Pd(OAc)_2$ (10 mol %) and TBAI (20 mol %) in 1,4-dioxane (0.5 mL) at 90 °C for 17 h. [e] Reactions were carried out with pyridine *N*-oxide **1a** (0.5 mmol, 3.3 equiv), potassium phenyltrifluoroborate **2a** (0.15 mmol), Ag_2O (0.3 mmol, 2 equiv) $Pd(OAc)_2$ (10 mol %) and TBAI (20 mol %) in 1,4-dioxane (0.5 mL) for 17 h.

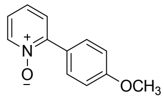
III. Characterization data for coupling products

2-phenylpyridine N-oxide (3a). [1, 2]



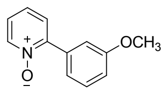
R_f = 0.5 (AcOEt/Alcohol = 10/1 v/v); light yellow solid; M.p. = 141-142 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.20-7.24 (m, 1H), 7.28 (td, *J* = 7.6, 1.1 Hz, 1H), 7.41 (dd, *J* = 8.0, 2.2 Hz, 1H), 7.44-7.47 (m, 1H), 7.46-7.50 (m, 2H), 7.80-7.83 (m, 2H), 8.32 (dd, *J* = 6.6, 0.7 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3063, 3044, 1477, 1418, 1240, 841, 759, 724, 697.

2-(4-methoxyphenyl)-pyridine N-oxide (3b). [3, 4, 5, 6]



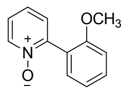
R_f = 0.6 (AcOEt/Alcohol = 6/1 v/v); Yellowish solid; M.p. = 121-123 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 3.87 (s, 3H), 6.99 (dt, *J* = 8.9, 2.9 Hz, 2H), 7.17-7.21 (m, 1H), 7.27 (td, *J* = 7.7, 1.2 Hz, 1H), 7.41 (dd, *J* = 7.9, 2.0 Hz, 1H), 7.81 (dt, *J* = 8.9, 2.9 Hz, 2H), 8.31 (dd, *J* = 6.6, 0.5 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3064, 2984, 1584, 1497, 1446, 1332, 1251, 1204, 1179, 833, 766.

2-(3-methoxyphenyl)-pyridine N-oxide (3c). [4]



R_f = 0.6 (AcOEt/Alcohol = 6/1 v/v); White solid; M.p. = 120-122 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 3.85 (s, 3H), 6.99 (ddd, *J* = 8.2, 2.6, 1.0 Hz, 1H), 7.22-7.26 (m, 1H), 7.29-7.34 (m, 2H), 7.38 (t, *J* = 7.7 Hz, 1H), 7.42-7.44 (m, 2H), 8.32 (dd, *J* = 6.5, 0.8 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3102, 3057, 2935, 2841, 1608, 1531, 1435, 1243, 830, 761.

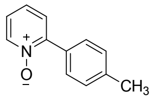
2-(2-methoxyphenyl)-pyridine N-oxide (3d). [5]



R_f = 0.5 (AcOEt/Alcohol = 6/1 v/v); White solid; M.p. = 169-171 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ =

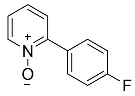
3.82 (s, 3H), 7.01 (d, *J* = 8.3 Hz, 1H), 7.05 (td, *J* = 7.5, 1.0 Hz, 1H), 7.23-7.27 (m, 1H), 7.27 (dd, *J* = 7.6, 1.4 Hz, 1H), 7.34-7.37 (m, 1H), 7.38 (dd, *J* = 7.5, 1.7Hz, 1H), 7.43-7.46 (m, 1H), 8.33-8.35 (m,1H) ppm. IR (cm^{-1}, KBr): 3094, 3044, 2961, 2843, 1598, 1487, 1435, 1367, 1243, 961, 827, 733.

2-(4-methylphenyl)-pyridine N-oxide (3e). [3, 4, 5, 6]



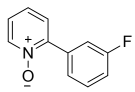
R_f = 0.6 (Petroleum ether/AcOEt = 6/1 v/v); Yellowish solid; M.p. = 129-131 °C; ^1H NMR (600 MHz, $CDCl_3$): δ = 2.41 (s, 3H), 7.19-7.23 (m, 1H), 7.28-7.32 (m, 3H), 7.60-7.64 (m, 3H), 7.4 (dd, *J* = 7.9, 2.3 Hz, 1H), 7.71 (dt, *J* = 8.1, 2.0 Hz, 2H), 8.32 (dt, *J* = 6.1, 1.0 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3066, 3043, 2915, 1614, 1430, 1240, 1010, 816, 760.

2-(4-fluorophenyl)-pyridine N-oxide (3f). [7]



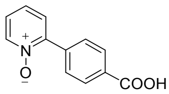
R_f = 0.6 (AcOEt/Alcohol = 10/1 v/v); Yellowish solid; M.p. = 161-163 °C; ^1H NMR (600 MHz, $CDCl_3$): δ = 7.15-7.20 (m, 2H), 7.23-7.26 (m, 1H), 7.30 (td, *J* = 7.8, 1.2 Hz, 1H), 7.41 (dd, *J* = 7.8, 2.0 Hz, 1H), 7.83-7.86 (m, 2H), 8.33 (dd, *J* = 6.4, 0.9Hz, 1H) ppm. IR (cm^{-1}, KBr): 3062, 3040, 2463, 1916, 1597, 1246, 1018, 760, 572.

2-(3-fluorophenyl)-pyridine N-oxide (3g).



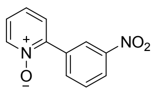
R_f = 0.6 (AcOEt /Alcohol = 6/1 v/v); Yellowish solid; M.p. = 106-107 °C; ^1H NMR (600 MHz, $CDCl_3$): δ = 7.14-7.18 (m, 1H), 7.25-7.29 (m, 1H), 7.31 (td, *J* = 7.7, 1.2 Hz, 1H), 7.43-7.48 (m, 2H), 7.55-7.58(m, 1H), 7.61 (dt, *J* = 9.9, 1.7 Hz, 1H) , 8.34 (dd, *J* = 6.4, 0.7 Hz, 1H) ppm; ^{13}C NMR (150 MHz, $CDCl_3$): δ = 119.4, 119.5, 119.7, 127.9, 128.8, 130.2, 132.7 (d, *J* = 8.6 Hz), 132.8, 143.5, 164.5 (d, *J* = 244.2 Hz) ppm; HRMS (ESI, *m/z*): Calcd for $C_{11}H_8FNO$ $[M + H]^+$ 190.0668, found 190.0662. IR (cm^{-1}, KBr): 3074, 3051, 2421, 1603, 1497, 1332, 1263, 1007, 801, 596.

4-(N-Oxopyridin-2-yl)benzoic acid (3h). **CAS:281234-68-2**



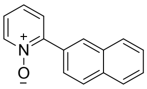
R_f = 0.2 (AcOEt); Yellow solid; M.p. = 162-164 °C; ^1H NMR (400 MHz, $CDCl_3$): δ = 7.28-7.33 (m, 1H), 7.33 (td, *J* = 7.6, 1.3 Hz, 1H), 7.46 (dd, *J* = 7.8, 2.0 Hz, 1H), 8.00 (s, 4H), 8.35 (dd, *J* = 6.2, 0.8 Hz, 1H) , 10.8 (s, 1H) ppm. IR (cm^{-1}, KBr): 3632, 3103, 3051, 2443, 1597, 1497, 1348, 1203, 819, 796.

2-(3-nitrophenyl)-pyridine N-oxide (3i). **CAS:103985-13-3**



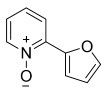
R_f = 0.4 (AcOEt/Alcohol = 10/1 v/v); pale yellow solid; M.p. = 175-177 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 7.31 (td, J = 6.4, 2.2 Hz, 1H), 7.36 (td, J = 7.6, 1.4 Hz, 1H), 7.50 (dd, J = 7.8, 2.2 Hz, 1H), 7.66 (t, J = 8.0 Hz, 1H), 8.25 (dt, J = 8.1, 1.3 Hz, 1H), 8.31 (dq, J = 8.3, 1.0 Hz, 1H), 8.36 (dd, J = 6.2, 0.9 Hz, 1H), 8.66 (t, J = 1.9 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3076, 3041, 1580, 1440, 1368, 1237, 1108, 928, 743.

***2-(2-naphthalenyl)-pyridine N-oxide (3j)*. CAS: 1622867-80-4**



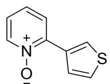
R_f = 0.4 (AcOEt/Alcohol = 6/1 v/v); pale solid; M.p. = 139-141 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.25-7.28 (m, 1H), 7.33 (td, J = 7.6, 1.1 Hz, 1H), 7.50-7.53 (m, 1H), 7.52-7.57 (m, 2H), 7.87 (d, J = 7.9 Hz, 1H), 7.91 (t, J = 5.9 Hz, 1H), 7.93-7.97 (m, 2H), 8.26 (s, 1H), 8.37-8.39 (m, 1H) ppm. IR (cm^{-1}, KBr): 3097, 3056, 1607, 1529, 1430, 1368, 1137, 892, 698.

***2-(2-furanyl)-pyridine N-oxide (3k)*. CAS: 55484-25-8**



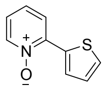
R_f = 0.4 (AcOEt); pale yellow solid; M.p. = 94-96 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 6.61 (q, J = 1.7 Hz, 1H), 7.11 (td, J = 7.1, 1.9 Hz, 1H), 7.29-7.33 (m, 1H), 7.59 (d, J = 1.1 Hz, 1H), 7.93 (dd, J = 8.2, 1.9 Hz, 1H), 8.02 (d, J = 3.4 Hz, 1H), 8.27 (d, J = 6.4 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3075, 1594, 1498, 1423, 1276, 1258, 898, 841, 591.

***2-(3-thienyl)-pyridine N-oxide (3l)*. CAS: 92928-98-8**



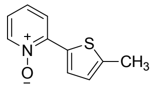
R_f = 0.4 (AcOEt); pale yellow solid; M.p. = 118-119 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.15-7.18 (m, 1H), 7.28-7.32 (m, 1H), 7.40 (dd, J = 5.2, 3.1 Hz, 1H), 7.64 (dd, J = 5.2, 1.3 Hz, 1H), 7.68 (dd, J = 8.1, 1.9 Hz, 1H), 8.33 (dd, J = 6.2, 0.8 Hz, 1H), 8.86 (dd, J = 3.2, 1.3 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3044, 1563, 1477, 1450, 1291, 1197, 898, 872, 633.

***2-(2-thienyl)-pyridine N-oxide (3m)*.** [8]



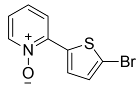
R_f = 0.4 (AcOEt); Pale solid; M.p. = 143-146 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.11 (td, J = 6.9, 1.9 Hz, 1H), 7.21-7.23 (m, 1H), 7.30-7.34 (m, 1H) ,7.57 (dd, J = 5.2, 1.1 Hz, 1H), 7.86 (dd, J = 4.0, 1.1 Hz, 1H), 7.93 (td, J = 8.3, 1.8 Hz, 1H) , 8.31 (dd, J = 6.8, 0.8 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3059, 1544, 1498, 1423, 1276, 1258, 898, 841, 591.

2-(5-methyl-2-thienyl)-pyridine N-oxide (3n). [2, 9]



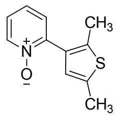
R_f = 0.4 (AcOEt); Yellow solid; M.p. = 140-142 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.55 (s, 3H), 6.88 (dd, J = 4.0, 1.0 Hz, 1H), 7.06 (td, J = 7.1, 1.9 Hz, 1H), 7.28 (dd, J = 7.6, 1.3 Hz, 1H), 7.67 (d, J = 4.0 Hz, 1H), 7.85 (dd, J = 8.3, 1.7 Hz, 1H), 8.27-8.30 (m, 1H) ppm. IR (cm^{-1}, KBr): 3034, 3017, 2986, 1599, 1509, 1479, 1329, 1276, 1128, 935, 698.

2-(5-bromo-2-thienyl)-pyridine N-oxide (3o).



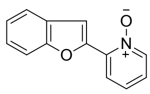
R_f = 0.4 (AcOEt); pale yellow solid; M.p. = 164-166 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.14 (dd, J = 7.2, 1.8 Hz, 1H), 7.18 (d, J = 4.3 Hz, 1H), 7.33-7.36 (m, 1H), 7.59 (d, J = 4.3 Hz, 1H), 7.87 (dd, J = 8.3, 1.7 Hz, 1H), 8.30 (d, J = 6.9 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 119.9, 121.3, 122.3, 126.1, 126.5, 128.8, 132.4, 139.1, 142.5 ppm; HRMS (ESI, m/z): Calcd for C_9H_6BrNOS [M + H]$^+$ 255.9432, found 255.9442. IR (cm^{-1}, KBr): 3071, 1603, 1508, 1329, 1307, 1258, 1124, 1085, 876, 691.

2-(2,5-dimethyl-3-thienyl)-pyridine N-oxide (3p).



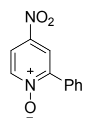
R_f = 0.4 (AcOEt/Alcohol = 6/1 v/v); pale yellow liquid; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.38 (s, 3H), 2.42 (s, 3H), 6.89 (d, J = 0.6 Hz, 1H), 7.18 (td, J = 6.7, 2.5 Hz, 1H), 7.23 (d, J = 8.1 Hz, 1H), 7.26-7.29 (m, 1H), 8.31 (d, J = 6.4 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 14.4, 15.2, 124.2, 124.9, 126.4, 128.0, 129.1, 136.1, 138.0, 140.5, 146.0 ppm; HRMS (ESI, m/z): Calcd for $C_{11}H_{12}NOS$ [M + H]$^+$ 206.0640, found 206.0643. IR (cm^{-1}, KBr): 3077, 3049, 2989, 2908, 1583, 1494, 1371, 1267, 1209, 1107, 877, 806.

2-(2-benzofuranyl)-pyridine N-oxide (3q). [2]



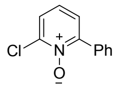
R_f = 0.5 (AcOEt); Pale yellow solid; M.p. = 139-141 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.19-7.22 (m, 1H), 7.28-7.31 (m, 1H), 7.35 (dd, J = 8.2, 0.9 Hz, 1H), 7.37-7.42 (m, 1H), 7.52 (dd, J = 8.3, 0.7 Hz, 1H), 7.71-7.74 (m, 1H), 8.15 (dd, J = 8.2, 2.0 Hz, 1H), 8.34-8.36 (m, 1H), 8.46 (d, J = 0.9, Hz, 1H) ppm. IR (cm^{-1}, KBr): 3098, 3059, 1558, 1494, 1307, 1298, 1109, 1047, 897, 791.

4-nitro-2-phenylpyridine N-oxide (3r). [10]



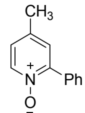
R_f = 0.5 (AcOEt); Pale yellow solid; M.p. = 135-136 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.52-7.54 (m, 3H), 7.80-7.82 (m, 2H), 8.02 (dd, J = 7.1, 3.2 Hz, 1H), 8.28 (d, J = 3.2 Hz, 1H), 8.36 (d, J = 7.2 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3041, 1597, 1508, 1339, 1284, 1231, 1114, 895, 724.

***2-chloro-6-phenylpyridine N-oxide (3s)*. CAS: 119492-88-5**



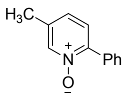
R_f = 0.3 (Petroleum ether/AcOEt = 3/1 v/v); Pale yellow solid; M.p. = 141-142 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 7.20 (t, J = 8.0 Hz, 1H), 7.35 (dd, J = 7.9, 2.0 Hz, 1H), 7.45-7.50 (m, 4H), 7.78-7.81 (m, 2H) ppm. IR (cm^{-1}, KBr): 3048, 1610, 1508, 1497, 1328, 1263, 1209, 1095, 747, 678.

***4-methyl-2-phenylpyridine N-oxide (3t)*. CAS: 80635-42-3**



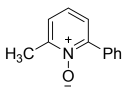
R_f = 0.2 (AcOEt /Alcohol = 6/1 v/v); Pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.39 (s, 3H), 7.04 (dd, J = 6.5, 2.3 Hz, 1H), 7.31 (t, J = 8.1 Hz, 1H), 7.45-7.50 (m, 3H), 7.78-7.84 (m, 2H), 8.27 (d, J = 6.7 Hz, 1H) ppm. IR (cm^{-1}, KBr): 3072, 2978, 1598, 1541, 1469, 1382, 1294, 1098, 814,716.

***5-methyl-2-phenylpyridine N-oxide (3u)*.** [6]



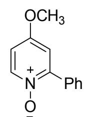
R_f = 0.3 (AcOEt /Alcohol = 10/1 v/v); Pale solid; M.p. = 168-169 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.33 (s, 3H), 7.12-7.15 (m, 1H), 7.30 (d, J = 8.0 Hz, 1H), 7.42-7.50 (m, 3H), 7.78 (dd, J = 8.3, 1.7 Hz, 2H), 8.20 (s, 1H) ppm. IR (cm^{-1}, KBr): 3048, 2967, 1603, 1523, 1481, 1378, 1321, 1245, 1103, 829, 732, 699.

***6-methyl-2-phenylpyridine N-oxide (3v)*.** [3, 6, 8]



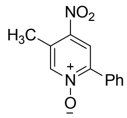
R_f = 0.5 (Petroleum ether/AcOEt = 2/1 v/v); Pale solid; M.p. = 118-119 °C; ^{1}H NMR (600 MHz, $CDCl_3$): δ = 2.58 (s, 3H), 7.18 (t, J = 7.7 Hz, 1H), 7.24 (d, J = 2.2 Hz, 1H), 7.30 (dd, J = 8.0, 2.2 Hz, 1H), 7.42-7.48 (m, 3H), 7.77 (dd, J = 8.3, 1.6 Hz, 2H) ppm. IR (cm^{-1}, KBr): 3059, 3015, 2946, 2889, 1597, 1523, 1470, 1369, 1315, 1287, 1103, 799, 693.

***4-methoxy-2-phenylpyridine N-oxide (3w)*.**



R_f = 0.5 (AcOEt/Alcohol = 4/1 v/v); Pale yellow liquid; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 3.87 (s, 3H), 6.78 (q, J = 3.5 Hz, 1H), 6.92 (d, J = 3.5 Hz, 1H), 7.44-7.50 (m, 3H), 7.80 (dd, J = 9.7, 1.9 Hz, 2H), 8.23 (d, J = 7.3 Hz, 1H) ppm; ^{13}C NMR (100 MHz, $CDCl_3$): δ = 56.1, 110.9, 112.4, 128.3, 129.3, 129.7, 132.8, 141.2, 149.8, 157.6 ppm; HRMS (ESI, *m/z*): Calcd for $C_{12}H_{11}NO_2[M + H]^+$ 202.0868, found 202.0867. IR (cm^{-1}, KBr): 3074, 2954, 2883, 1590, 1514, 1473, 1382, 1342, 1291, 1249, 1073, 832, 719.

***3-methyl-4-nitro-6-phenylpyridine N-oxide (3x)*. CAS:1344663-62-2**



R_f = 0.3 (Petroleum ether/AcOEt = 3/1 v/v); Yellow solid; M.p. = 169-170 °C; ^{1}H NMR (400 MHz, $CDCl_3$): δ = 2.63 (d, J = 0.5 Hz, 3H), 7.50-7.53 (m, 3H), 7.78-7.81 (m, 2H), 8.20 (s, 1H), 8.26 (s, 1H) ppm. IR (cm^{-1}, KBr): 3082, 1590, 1514, 1346, 1295, 1249, 1073, 827, 743.

V. References

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[2] Liu, W.; Yu, X.; Li, Y.; Kuang, C. *Chem. Commun.,* **2014***, 50,* 9291–9294.

[3] Do, H. Q. R.; Khan, M. K.; Daugulis, O. *J. Am. Chem. Soc.* **2008**, *130*, 15185–15192.

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[5] Campeau, L.-C.; Rousseaux, S.; Fagnou, K. *J. Am. Chem. Soc.* **2005**, *127*, 18020–18021.

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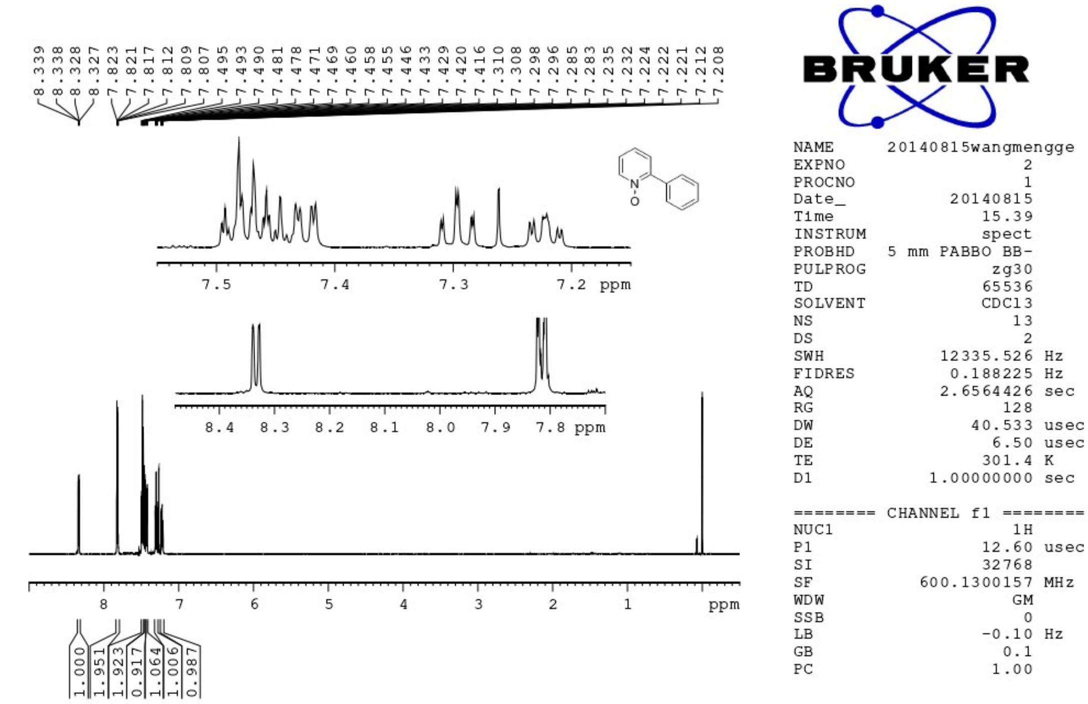
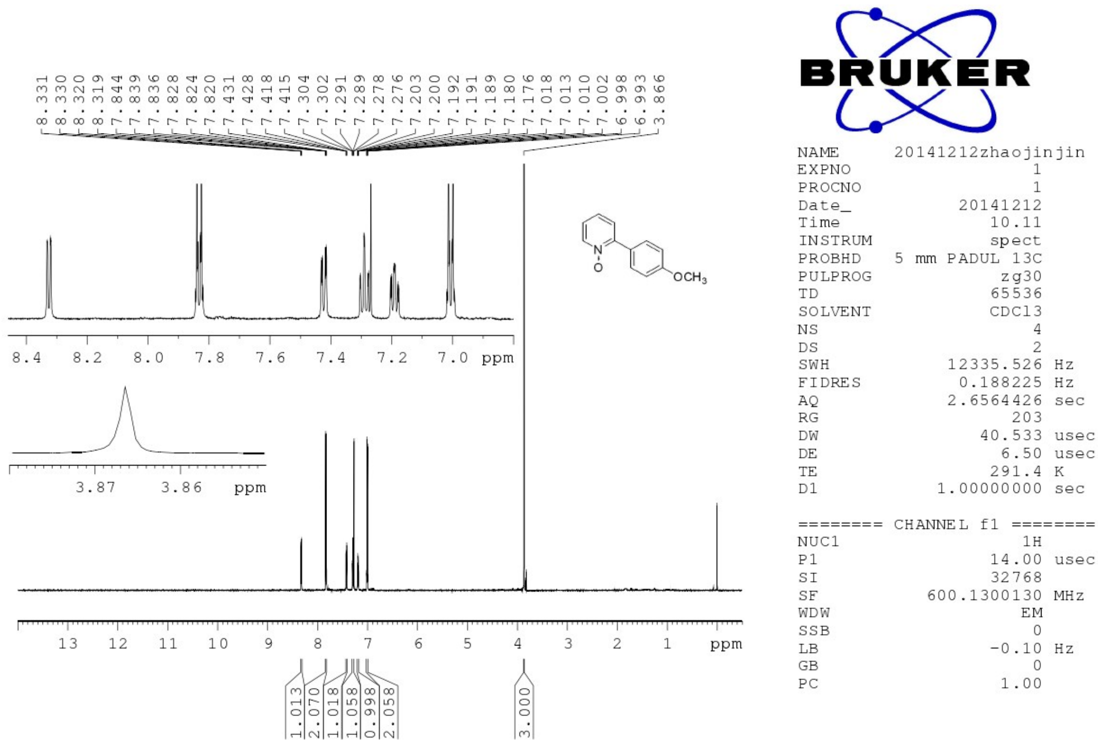
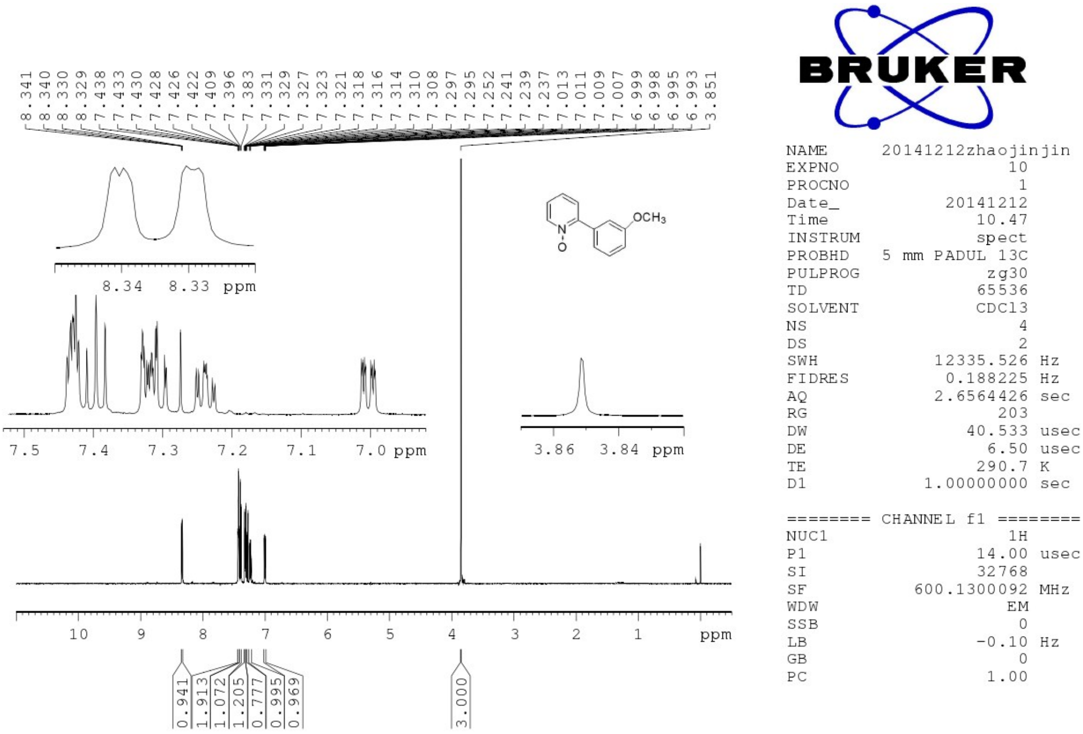
IV. ^{1}HNMR and ^{13}CNMR spectra



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PROCNO 1
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TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 290.7 K
D1 1.00000000 sec
======== CHANNEL f1 ========
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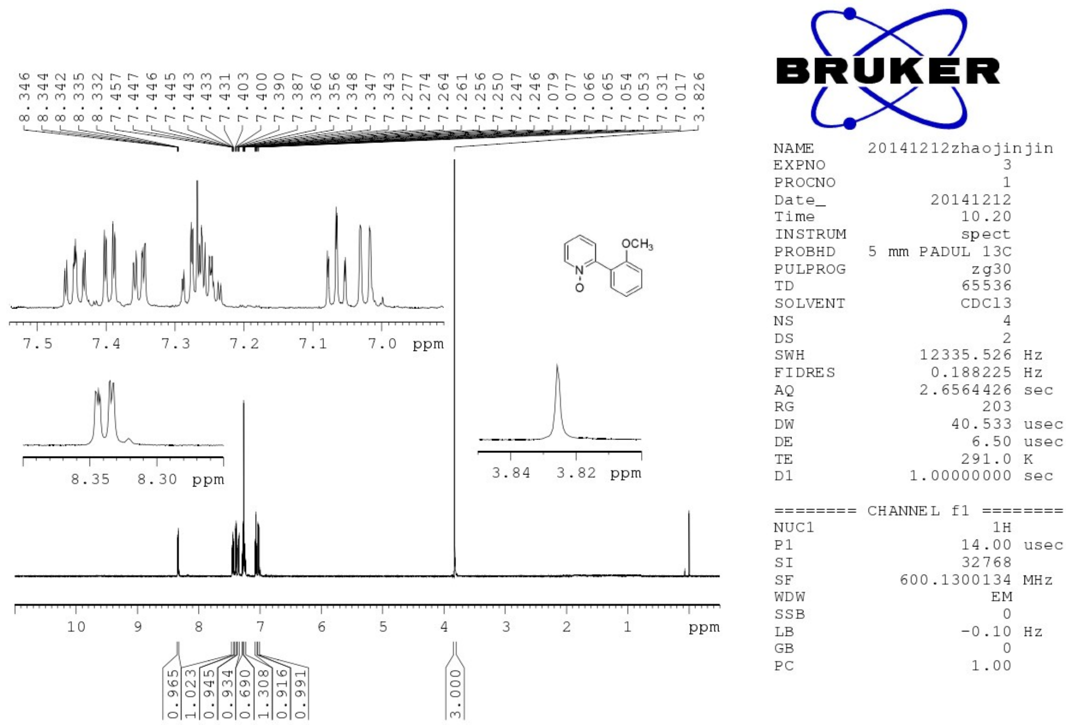
P1 14.00 usec
SI 32768
SF 600.1300092 MHz
WDW EM
SSB 0
LB -0.10 Hz
GB 0
PC 1.00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EXPNO 3
PROCNO 1
Date_ 20141212
Time 10.20
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 291.0 K
D1 1.00000000 sec
======== CHANNEL f1 ========
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P1 14.00 usec
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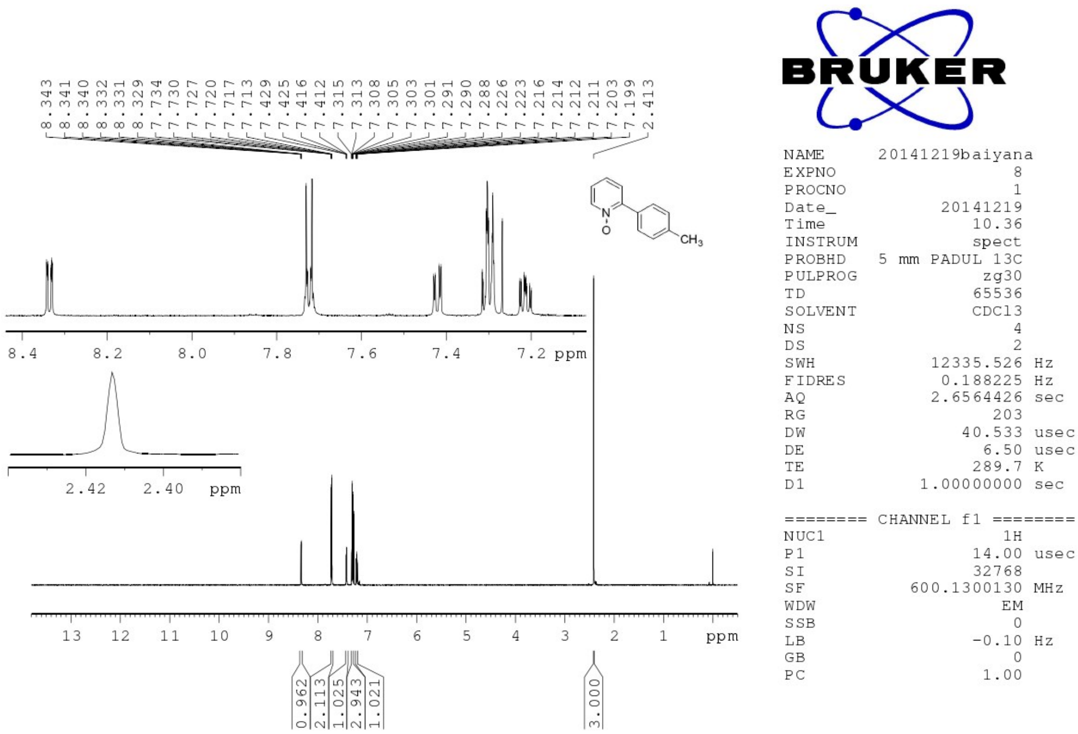
SSB 0
LB -0.10 Hz
GB 0
PC 1.00



8.343 8.341 8.340 8.332 8.331 8.329 7.734 7.730 7.727 7.720 7.717 7.713 7.429 7.425 7.416 7.412 7.315 7.313 7.308 7.305 7.303 7.301 7.291 7.290 7.288 7.226 7.223 7.216 7.214 7.212 7.211 7.203 7.199 2.413
N
O
CH3
8.4 8.2 8.0 7.8 7.6 7.4 7.2 ppm
2.42 2.40 ppm
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.962 2.113 1.025 2.943 1.021 3.000
BRUKER
NAME 20141219baiyana
EXPNO 8
PROCNO 1
Date_ 20141219
Time 10.36
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PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 289.7 K
D1 1.00000000 sec
======== CHANNEL f1 ========
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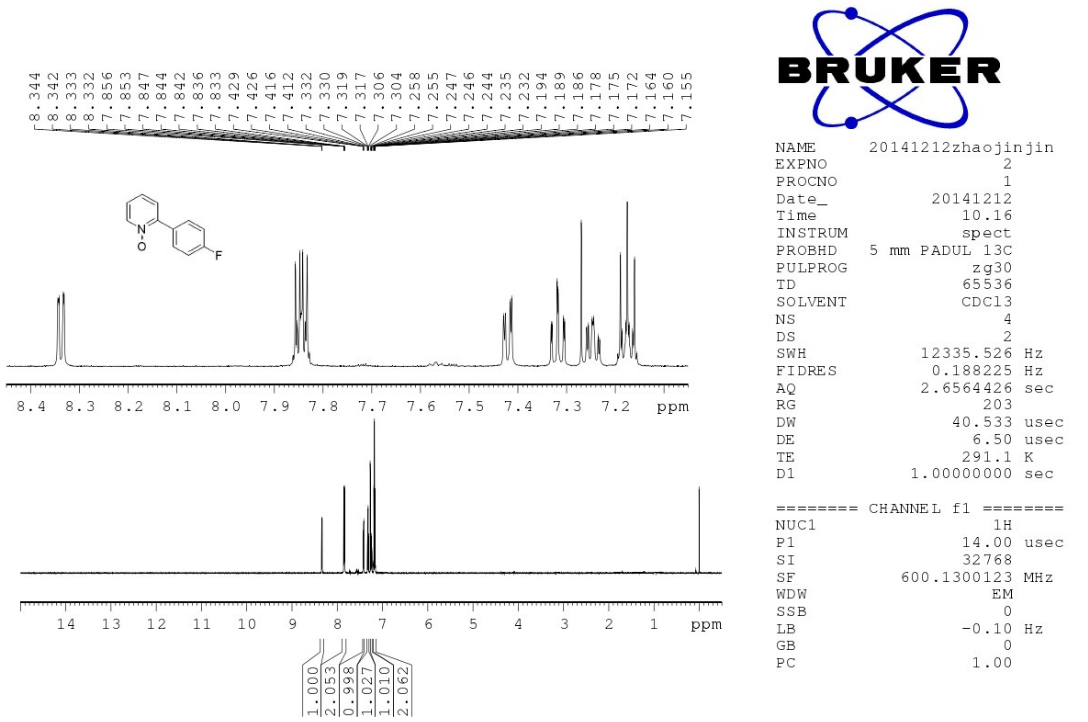
SI 32768
SF 600.1300130 MHz
WDW EM
SSB 0
LB -0.10 Hz
GB 0
PC 1.00



8.344 8.342 8.333 8.332 7.856 7.853 7.847 7.844 7.842 7.836 7.833 7.429 7.426 7.416 7.412 7.332 7.330 7.319 7.317 7.306 7.304 7.258 7.255 7.247 7.246 7.244 7.235 7.232 7.194 7.189 7.186 7.178 7.175 7.172 7.164 7.160 7.155
N
O
F
8.4 8.3 8.2 8.1 8.0 7.9 7.8 7.7 7.6 7.5 7.4 7.3 7.2 ppm
14 13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.000 2.053 0.998 1.027 1.010 2.062
BRUKER
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EXPNO 2
PROCNO 1
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PULPROG zg30
TD 65536
SOLVENT CDCl3
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DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 291.1 K
D1 1.00000000 sec
======== CHANNEL f1 ========
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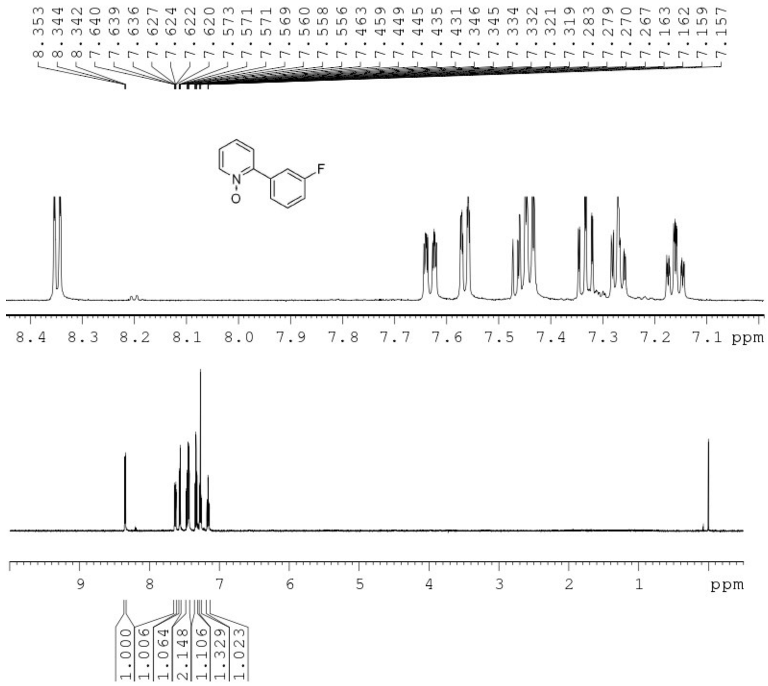

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TD 65536
SOLVENT CDCl3
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DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec

======== CHANNEL f1 ========
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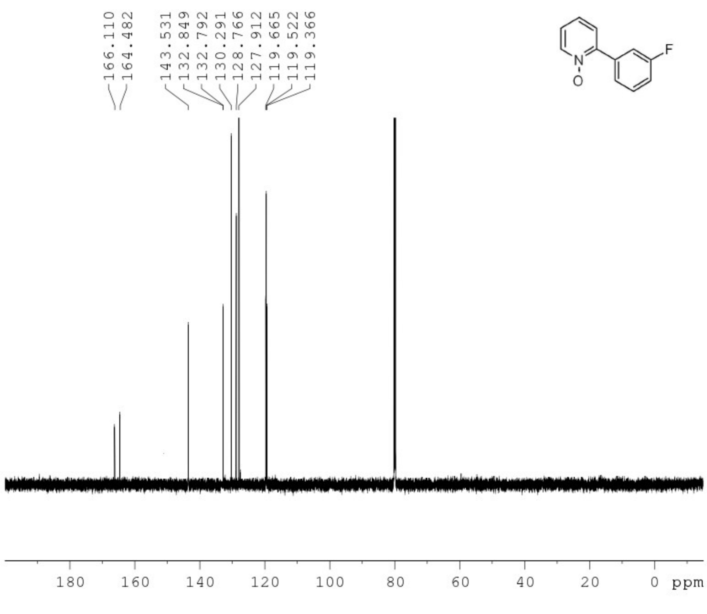
GB 0
PC 1.00



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PROCNO 1
Date_ 20150408
Time 15.01
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PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 765
DS 2
SWH 36057.691 Hz
FIDRES 0.550197 Hz
AQ 0.9088159 sec
RG 203
DW 13.867 usec
DE 6.50 usec
TE 293.8 K
D1 1.00000000 sec
D11 0.03000000 sec

======== CHANNEL f1 ========
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P1 9.80 usec
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SF 150.9023724 MHz
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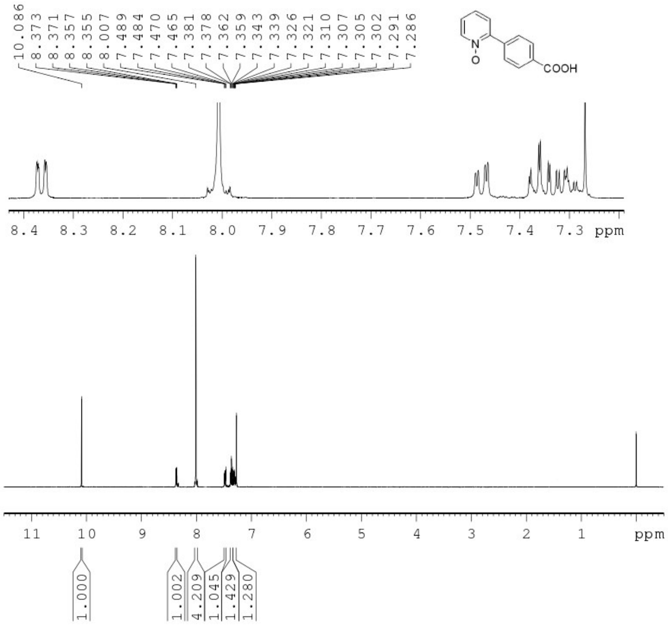
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



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Time	10.43
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PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	87.46
DW	62.400 usec
DE	6.50 usec
TE	296.5 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1324710 MHz
NUC1	1H
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SSB	0
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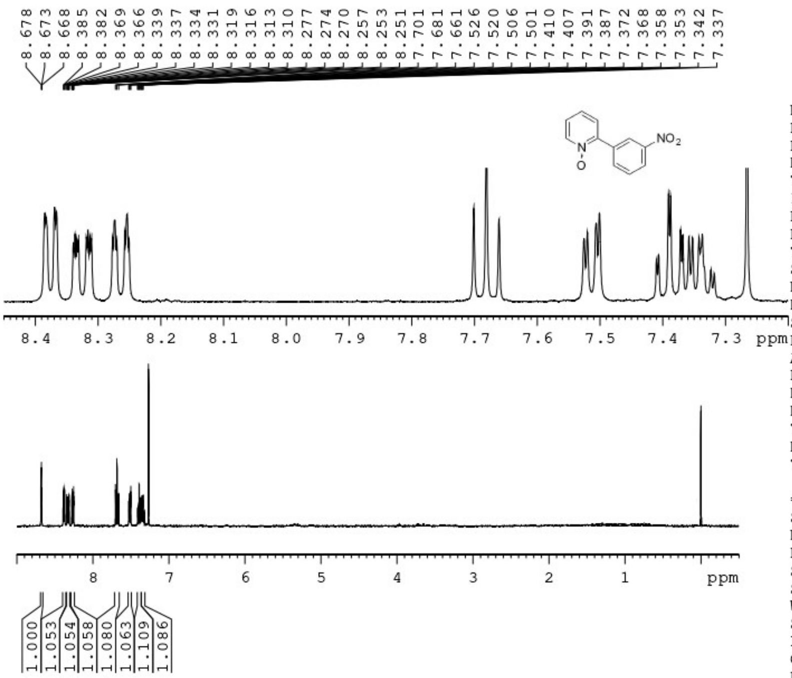
PC	1.00



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PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	87.46
DW	62.400 usec
DE	6.50 usec
TE	294.3 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1324710 MHz
NUC1	1H
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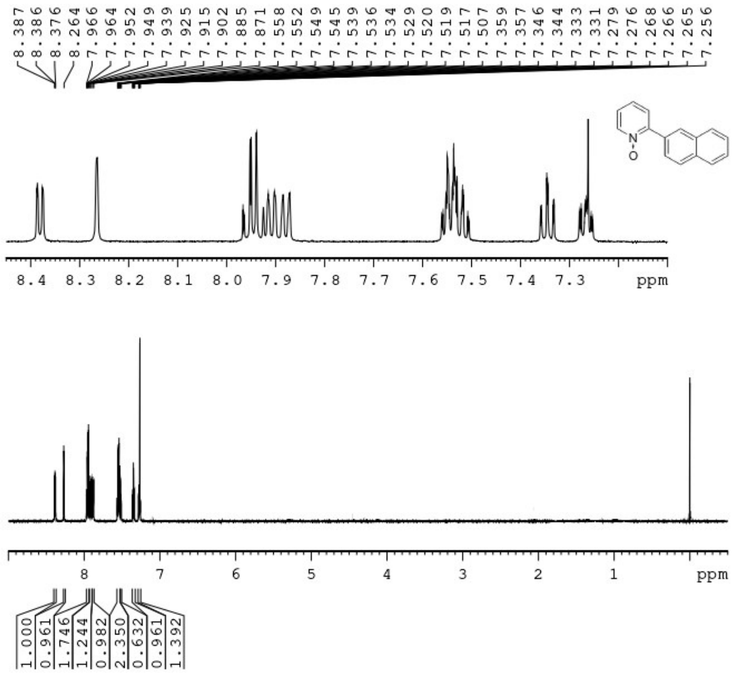
PC	1.00



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SOLVENT	CDCl3
NS	4
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	203
DW	40.533 usec
DE	6.50 usec
TE	293.4 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
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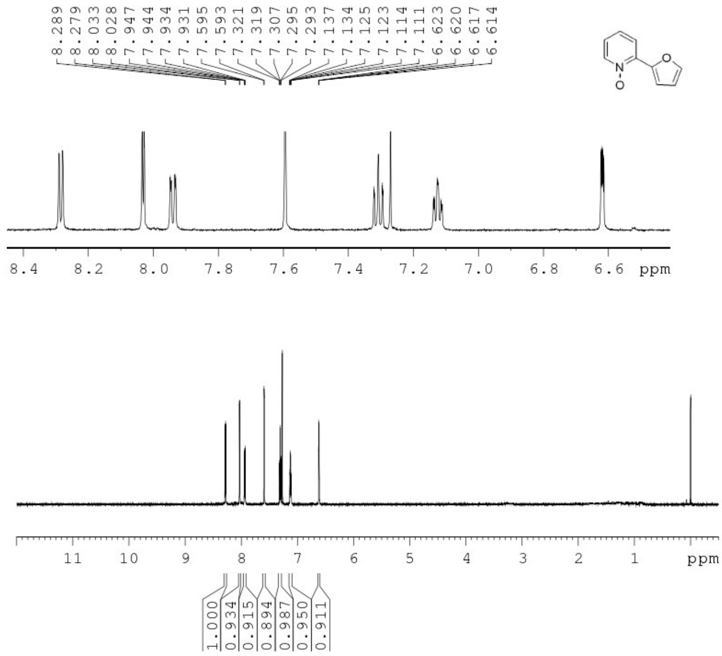
LB	-0.10 Hz
GB	0
PC	1.00



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PROCNO	1
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FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	181
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DE	6.50 usec
TE	293.4 K
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======== CHANNEL f1 ========	
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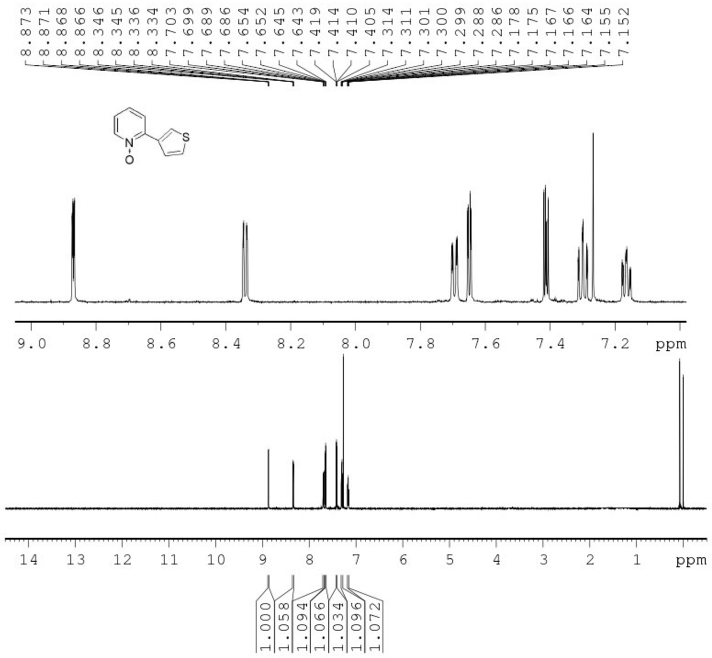


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PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           4
DS                           2
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FIDRES                0.188225 Hz
AQ                   2.6564426 sec
RG                         203
DW                      40.533 usec
DE                        6.50 usec
TE                       287.6 K
D1                  1.00000000 sec

======== CHANNEL f1 ========
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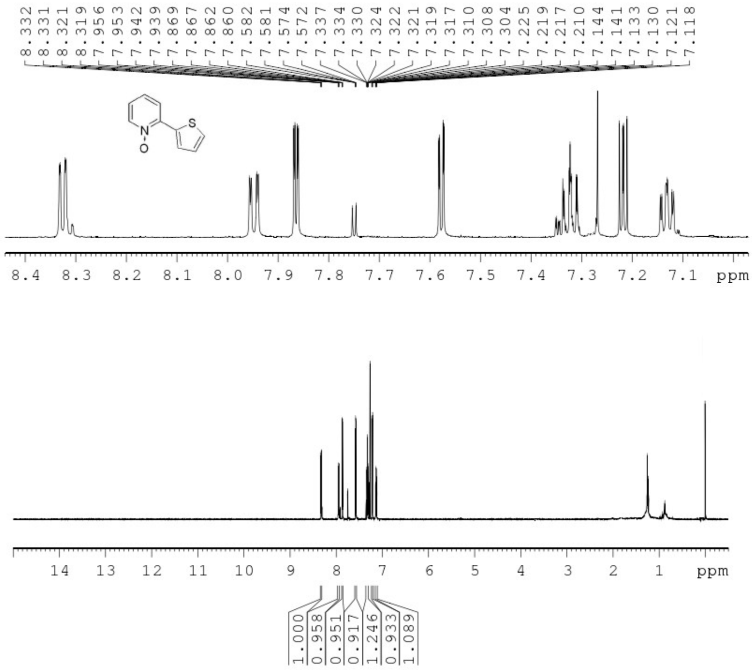


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Time                     15.44
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TD                       65536
SOLVENT                  CDCl3
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DS                           2
SWH                  12335.526 Hz
FIDRES                0.188225 Hz
AQ                   2.6564426 sec
RG                         144
DW                      40.533 usec
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D1                  1.00000000 sec

======== CHANNEL f1 ========
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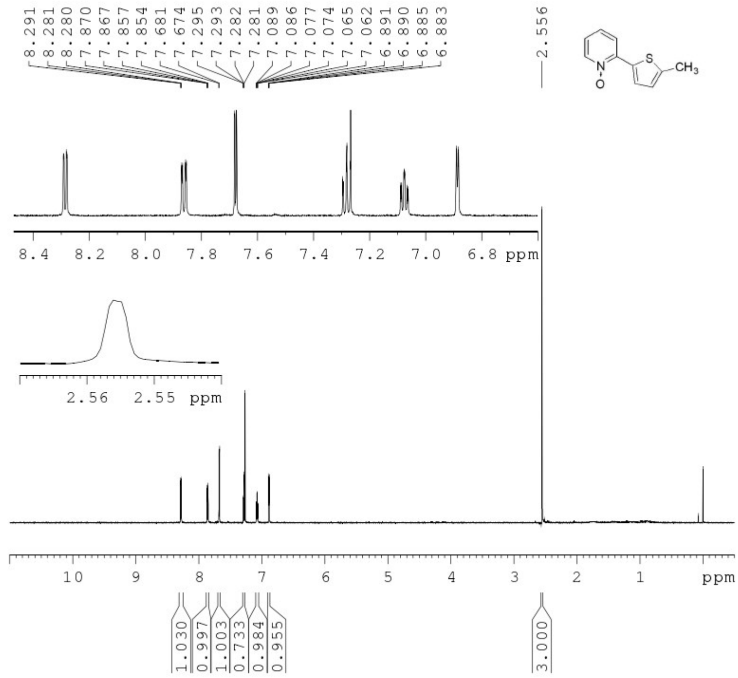
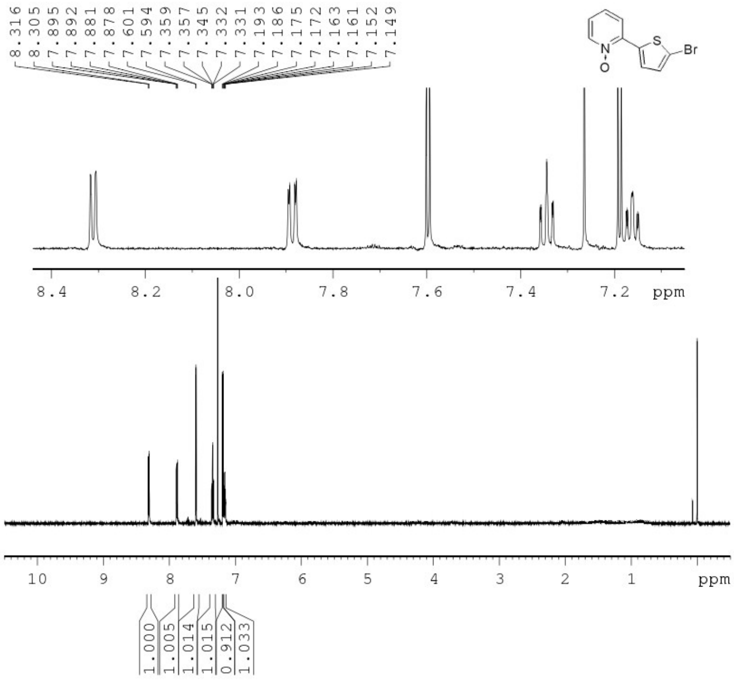
WDW                         EM
SSB                          0
LB                       -0.10 Hz
GB                           0
PC                        1.00
```



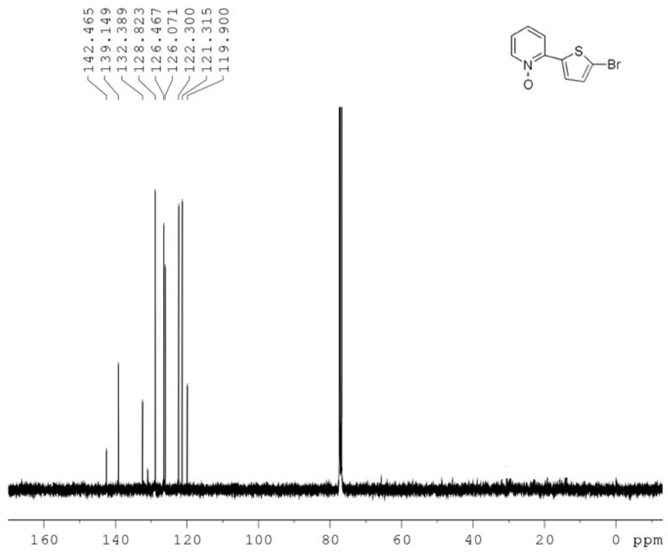
NAME 20150113zhaojinjin
EXPNO 5
PROCNO 1
Date_ 20150113
Time 15.25
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 181
DW 40.533 usec
DE 6.50 usec
TE 293.2 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300134 MHz
WDW EM
SSB 0
LB -0.10 Hz
GB 0
PC 1.00



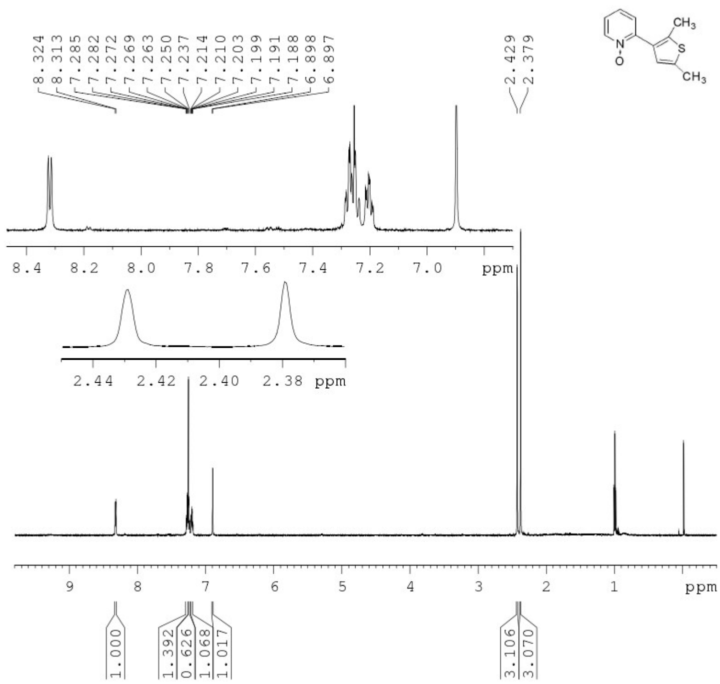
NAME 20150113zhaojinjin
EXPNO 9
PROCNO 1
Date_ 20150113
Time 15.40
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 293.0 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300150 MHz
WDW EM
SSB 0
LB -0.10 Hz
GB 0
PC 1.00



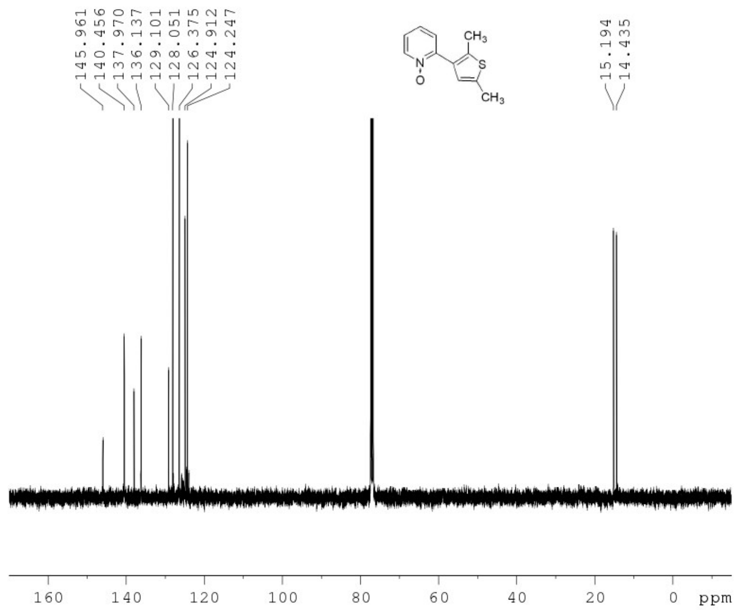
NAME 2015-04-23 tylg-huf
EXPNO 10
PROCNO 1
Date_ 20150423
Time 16.28
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 1024
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 123.61
DW 20.800 usec
DE 6.50 usec
TE 297.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

======== CHANNEL f1 ========
SFO1 100.6228293 MHz
NUC1 13C
P1 9.50 usec
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

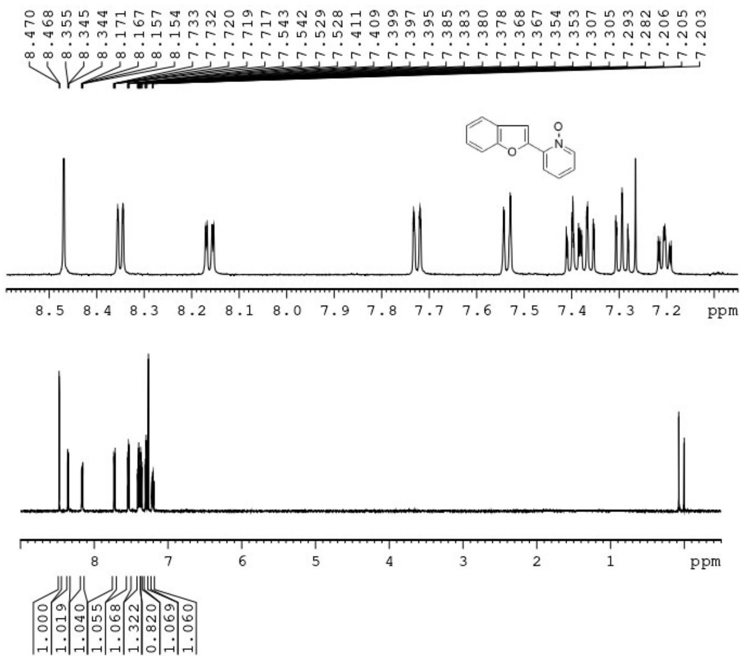


NAME 20150119zhaojinjin
EXPNO 6
PROCNO 1
Date_ 20150119
Time 15.19
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 293.4 K
D1 1.00000000 sec

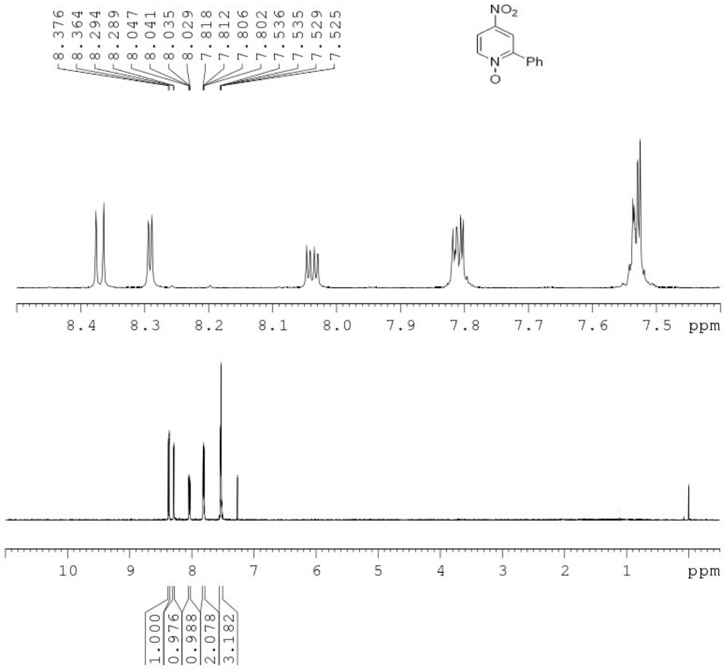
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300213 MHz
WDW EM
SSB 0
LB -0.10 Hz
GB 0
PC 1.00



NAME	2015-04-23 tylg-huf
EXPNO	10
PROCNO	1
Date_	20150423
Time	17.30
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	137.42
DW	20.800 usec
DE	6.50 usec
TE	297.7 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	9.50 usec
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

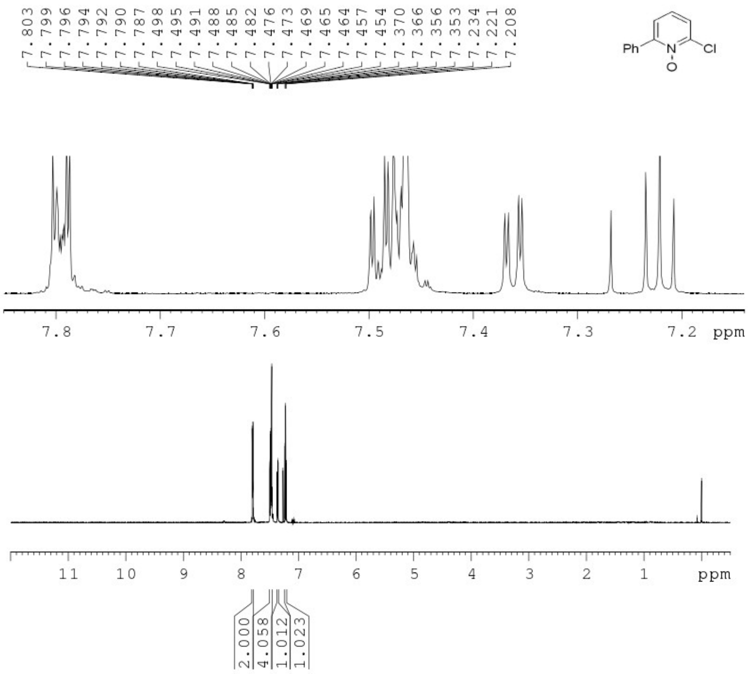


NAME	20150121wangmengge
EXPNO	4
PROCNO	1
Date_	20150121
Time	10.34
INSTRUM	spect
PROBHD	5 mm PADUL 13C
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	4
DS	2
SWH	12335.526 Hz
FIDRES	0.188225 Hz
AQ	2.6564426 sec
RG	203
DW	40.533 usec
DE	6.50 usec
TE	291.1 K
D1	1.00000000 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	14.00 usec
SI	32768
SF	600.1300144 MHz
WDW	EM
SSB	0
LB	-0.10 Hz
GB	0
PC	1.00



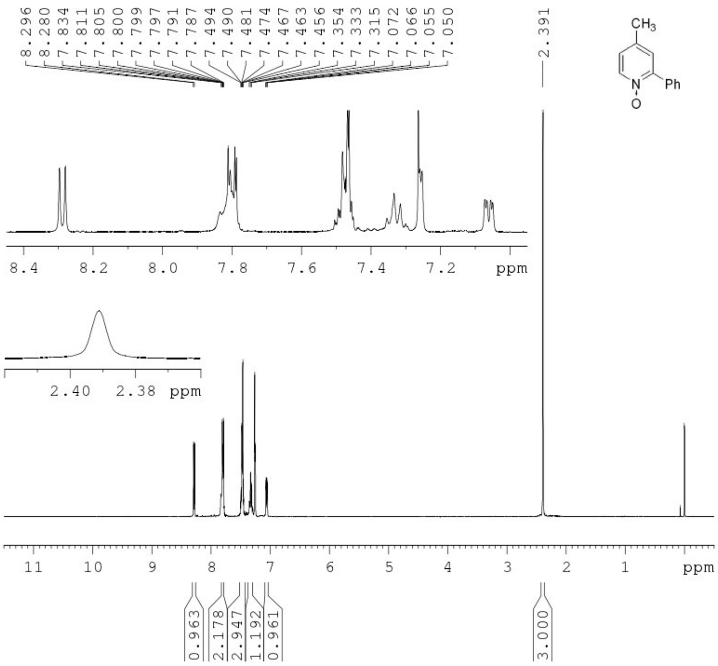
NAME 20150320zhouxin
EXPNO 3
PROCNO 1
Date_ 20150320
Time 14.53
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 181
DW 40.533 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300154 MHz
WDW EM
SSB 0
LB -0.10 Hz
GB 0
PC 1.00



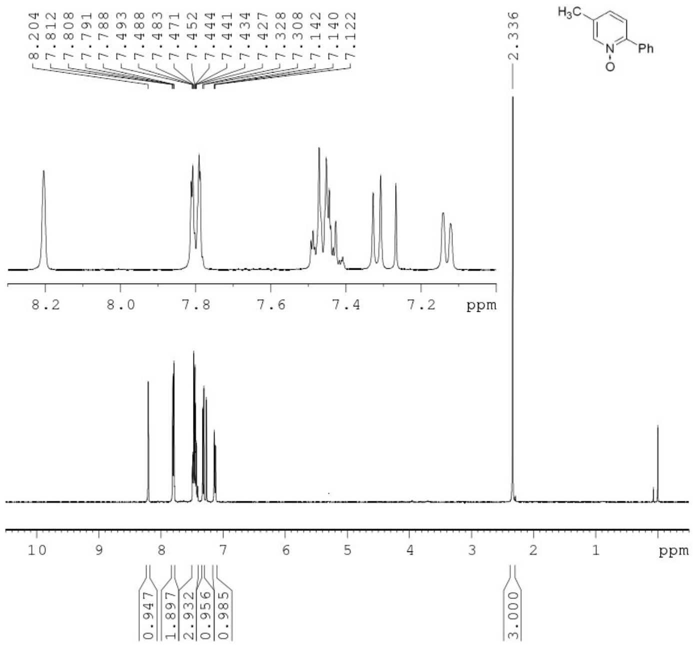
NAME 20150403zhourui
EXPNO 1
PROCNO 1
Date_ 20150403
Time 9.53
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 294.2 K
D1 1.00000000 sec

======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300121 MHz
WDW EM
SSB 0
LB -0.10 Hz
GB 0
PC 1.00



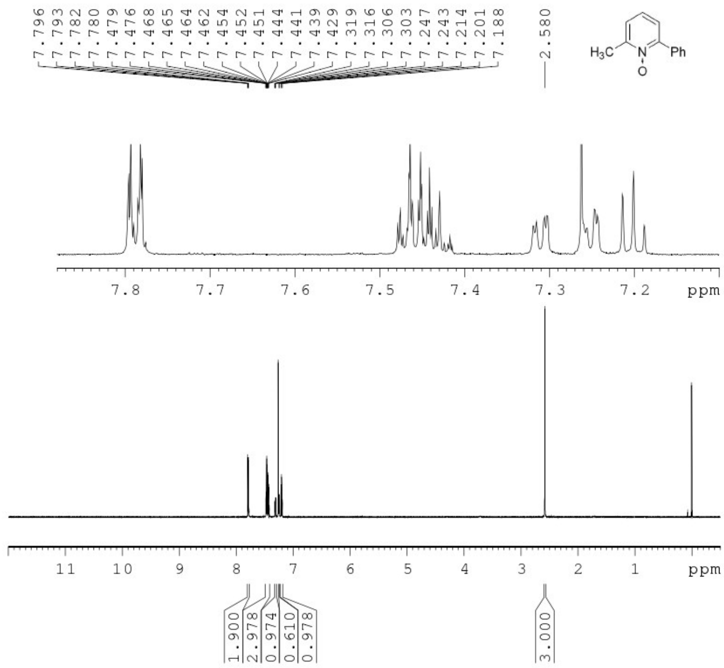

```
NAME        2015-04-20 tylg-huf
EXPNO                   10
PROCNO                   1
Date_             20150420
Time                 10.31
INSTRUM              spect
PROBHD      5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ               4.0894966 sec
RG                   74.25
DW                  62.400 usec
DE                    6.50 usec
TE                   294.3 K
D1              1.00000000 sec
TD0                      1

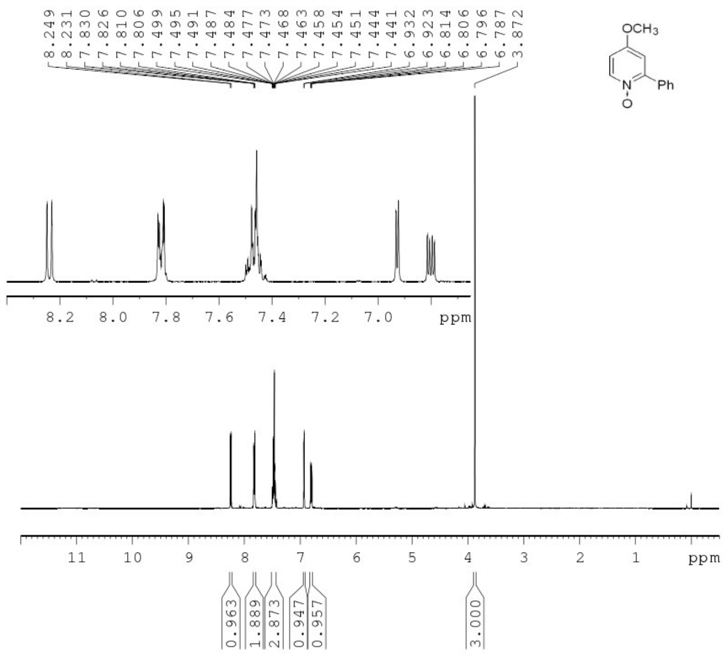
======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                    1H
P1                    9.70 usec
SI                   65536
SF             400.1300085 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```

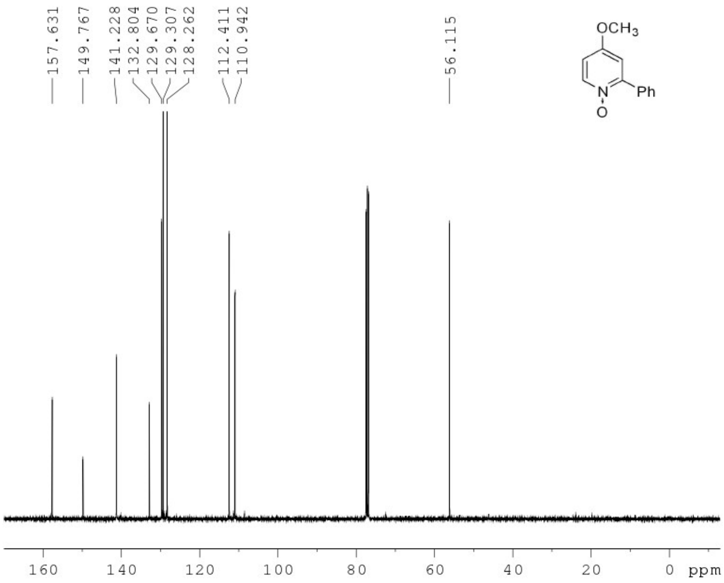



```
NAME        2015-04-16 tylg-huf
EXPNO                   10
PROCNO                   1
Date_             20150416
Time                  9.47
INSTRUM              spect
PROBHD      5 mm PABBO BB/
PULPROG               zg30
TD                   65536
SOLVENT              CDCl3
NS                      16
DS                       2
SWH               8012.820 Hz
FIDRES            0.122266 Hz
AQ               4.0894966 sec
RG                   74.25
DW                  62.400 usec
DE                    6.50 usec
TE                   293.7 K
D1              1.00000000 sec
TD0                      1

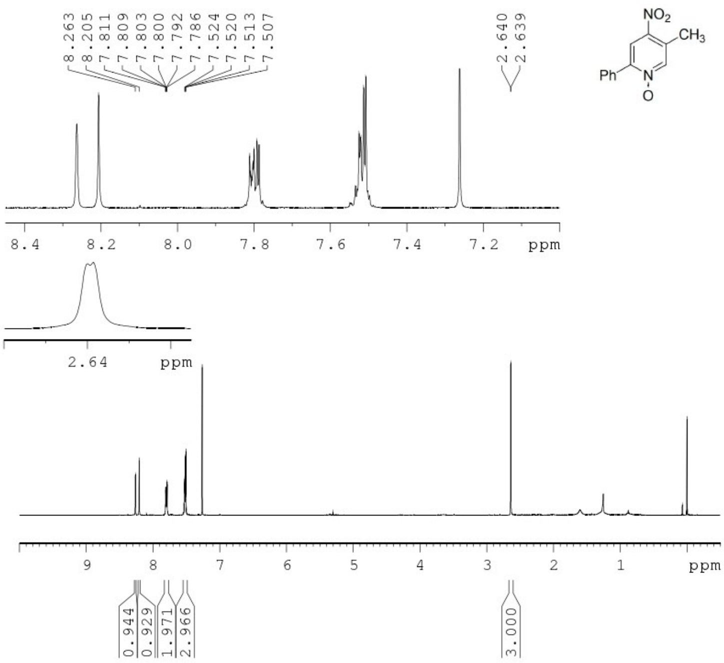
======== CHANNEL f1 ========
SFO1           400.1324710 MHz
NUC1                    1H
P1                    9.70 usec
SI                   65536
SF             400.1300070 MHz
WDW                     EM
SSB                      0
LB                    0.30 Hz
GB                       0
PC                    1.00
```


NAME 20150320zhouxin
EXPNO 2
PROCNO 1
Date_ 20150320
Time 14.44
INSTRUM spect
PROBHD 5 mm PADUL 13C
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 12335.526 Hz
FIDRES 0.188225 Hz
AQ 2.6564426 sec
RG 203
DW 40.533 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 14.00 usec
SI 32768
SF 600.1300162 MHz
WDW EM
SSB 0
LB -0.10 Hz
GB 0
PC 1.00


NAME 2015-04-16 tylg-huf
EXPNO 10
PROCNO 1
Date_ 20150416
Time 9.51
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 34.32
DW 62.400 usec
DE 6.50 usec
TE 293.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1324710 MHz
NUC1 1H
P1 9.70 usec
SI 65536
SF 400.1299960 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



NAME	2015-04-16 tylg-huf
EXPNO	20
PROCNO	1
Date_	20150416
Time	10.40
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	1024
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	185.43
DW	20.800 usec
DE	6.50 usec
TE	294.4 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	100.6228293 MHz
NUC1	13C
P1	9.50 usec
SI	32768
SF	100.6127690 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40



NAME	2015-05-07 tylg-huf
EXPNO	10
PROCNO	1
Date_	20150507
Time	12.06
INSTRUM	spect
PROBHD	5 mm PABBO BB/
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	2
SWH	8012.820 Hz
FIDRES	0.122266 Hz
AQ	4.0894966 sec
RG	107.24
DW	62.400 usec
DE	6.50 usec
TE	295.6 K
D1	1.00000000 sec
TD0	1
======== CHANNEL f1 ========	
SFO1	400.1324710 MHz
NUC1	1H
P1	9.70 usec
SI	65536
SF	400.1300093 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00