An Unprecedented Approach to 4,5-Disubstituted Pyrimidine Derivatives by a $ZnCl_2$-Catalyzed Three-Component Coupling Reaction

Supporting Information

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Table of Contents

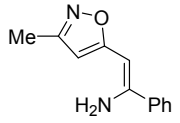
S2	General
S2-S6	Synthesis of Enamines
S6-S12	Synthesis of Pyrimidines
S12-S13	Synthesis of 2-azadiene **10a** and compound **13a**
S13	References
S14-S20	ORTEP of **4a** and crystallographic data
S21-S28	ORTEP of **5** and crystallographic data
S29-S36	ORTEP of **13a** and crystallographic data
S37-S61	Copies of ^{1}H and ^{13}C NMR Spectra of new compounds

Experimental Section

General Methods: Column chromatography was performed using Silica gel 60. THF and Toluene were distilled from sodium-benzophenone and dried over MS5A and MS4A, respectively. MeCN and 1,2-dichloroethane (DCE) were distilled from P_2O_5 and dried over MS4A. EtOH was distilled from Mg/I_2 and dried over MS3A. Prior to use, 3,5-dimethylisoxazol, 2-picoline, and *N*,*N*-dimethylacetoamide were distilled. Other materials were commercially available, and were used without further purification. All reactions were carried out under a nitrogen atmosphere, unless otherwise noted. All melting points are uncorrected. ^{1}H NMR spectra were measured at 500 or 300 MHz using tetramethylsilane as an internal standard. ^{13}C NMR spectra were measured at 125 or 75 MHz using the center peak of chloroform (77.0 ppm) as an internal standard. High resolution mass spectra were measured using NBA (3-nitrobenzyl alcohol) as a matrix. Elemental analyses were performed at the High Technology Research Center of the Tokyo University of Science.

General procedure for the synthesis of enamines 1a-l[1)]: To a THF solution (300 mL) of diisopropylamine (30.3 g, 300 mmol) was added *n*-BuLi (330 mmol, in hexane) at -70 °C, and the mixture was stirred at the same temperature. After 30 min, 3,5-dimethylisoxazole (29.1 g, 300 mmol) was added dropwise and the mixture was stirred for 1 h at -70 °C. Benzonitrile (30.9 g, 300 mmol) was gradually added to the solution, and the reaction mixture was further stirred for 1 h at the same temperature and then for 1 h at room temperature. To quench the reaction, water was added to the mixture. The mixture was extracted several times with AcOEt, and the combined organic extracts were dried over Na_2SO_4, filtered, and then concentrated under reduced pressure. The residue was purified either by recrystallization (hexane / AcOEt) or distillation under reduced pressure to give enamine **1a-l**.

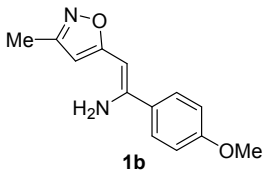
2-(3'-Methylisoxazol-5'-yl)-1-phenyl-1-ethenamine[2)] **(1a)**



1a

300 mmol scale; 90% yield; a yellow solid; mp 84.0-85.7 °C (lit.[2)] 85 °C); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.26 (s, 3H), 5.15 (br, 2H), 5.36 (s, 1H), 5.71 (s, 1H), 7.37-7.39 (m, 3H), 7.54-7.57 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.2, 85.0, 98.8, 126.1, 128.7, 129.3, 138.7, 148.4, 159.5, 170.3; MS (FAB) *m/z* 200 (M^+, 100%).

2-(3'-Methylisoxazol-5'-yl)-1-(4-methoxyphenyl)-1-ethenamine[3)] **(1b)**

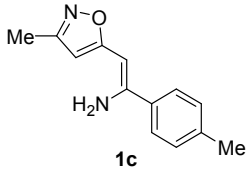


1b

30 mmol scale; 75% yield; a brown solid (AcOEt-hexane); mp 115.2-116.8 °C; ^{1}H NMR ($CDCl_3$, 500

MHz) δ 2.24 (s, 3H), 3.80 (s, 3H), 5.11 (br, 2H), 5.30 (s, 1H), 5.67 (s, 1H), 6.89 (d, 2H, *J* = 8.5 Hz), 7.48 (d, 2H, *J* = 8.5 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.2, 55.3, 84.1, 98.3, 114.0, 127.3, 131.0, 148.1, 159.4, 160.5, 170.4.

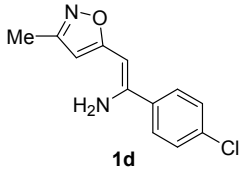
2-(3'-Methylisoxazol-5'-yl)-1-(4-methylphenyl)-1-ethenamine[3] (1c)



1c

30 mmol scale; 51% yield; a brown solid (AcOEt-hexane); mp 102.2-104.5 °C; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.27 (s, 3H), 2.38 (s, 3H), 5.15 (br, 2H), 5.36 (s, 1H), 5.71 (s, 1H), 7.21 (d, 2H, *J* = 8.1 Hz), 7.47 (d, 2H, *J* = 8.1 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 11.1, 21.1, 84.3, 98.4, 125.8, 129.3, 135.7, 139.3, 148.4, 159.4, 170.3.

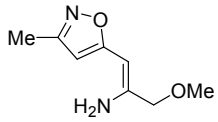
2-(3'-Methylisoxazol-5'-yl)-1-(4-chlorophenyl)-1-ethenamine[1] (1d)



1d

30 mmol scale; 84% yield; a pale yellow solid (AcOEt-hexane); mp 143.1-144.5 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.25 (s, 3H), 5.11 (br, 2H), 5.32 (s, 1H), 5.72 (s, 1H), 7.34 (d, 2H, *J* = 8.0 Hz), 7.48 (d, 2H, *J* = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.2, 85.4, 99.1, 127.4, 128.9, 135.1, 137.1, 147.1, 159.5, 169.9; MS (FAB) *m/z* 234 (M^+, 100%); Anal. Calcd for $C_{12}H_{11}ClN_2O$: C, 61.41; H, 4.72; N, 11.94%, Found: C, 61.57; H, 4.74; N, 11.96%.

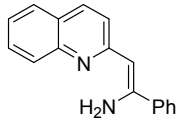
2-(3'-Methylisoxazol-5'-yl)-1-methoxymethyl-1-ethenamine[1] (1e)



1e

30 mmol scale; 60% yield; colorless oil; ^{1}H NMR ($CDCl_3$, 300 MHz) δ 2.25 (s, 3H), 3.36 (s, 3 H), 3.99 (s, 2 H), 5.01 (s, 1H), 5.14 (br, 2H), 5.63 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.0, 57.8, 73.4, 83.1, 97.7, 145.8, 159.3, 169.8; MS (FAB) *m/z* 168 (M^+, 100%); HRMS (FAB): Calcd for $C_8H_{13}N_2O_2$: 169.0977, Found 169.0986 (M+H).

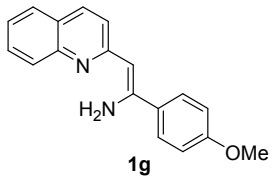
2-(Quinolin-2-yl)-1-phenyl-1-ethenamine[1] (1f)



1f

30 mmol scale; 92% yield; a yellow solid; mp 154.2-156.0 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.60 (s, 3H), 7.11 (d, 1H, *J* = 8.0 Hz), 7.32 (t, 1H, *J* = 8.0 Hz), 7.37-7.42 (m, 3H), 7.57 (t, 1H, *J* = 8.0 Hz), 7.62-7.66 (m, 3H), 7.86 (d, 1H, *J* = 8.0 Hz), 7.88 (d, 1H, *J* = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 95.5, 122.6, 124.1, 125.1, 126.1, 127.3, 127.5, 128.7, 129.0, 135.0, 139.6, 147.4, 152.6, 159.7; MS (FAB) *m/z* 247 (M+H, 100%); Anal. Calcd for $C_{17}H_{14}N_2$: C, 82.90; H, 5.73; N, 11.37%, Found: C, 82.93; H, 5.92; N, 11.32%.

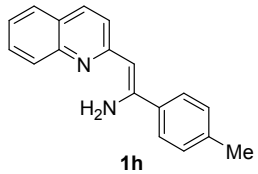
2-(Quinolin-2-yl)-1-(4-methoxyphenyl)-1-ethenamine[1] **(1g)**



1g

30 mmol scale; 90% yield; a yellow crystal (AcOEt-hexane); mp 156.7-158.0 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.84 (s, 3H), 5.56 (s, 1H), 6.94 (d, 2H, *J* = 8.5 Hz), 7.12 (d, 1H, *J* = 8.0 Hz), 7.33 (t, 1H, *J* = 8.0 Hz), 7.58 (t, 1H, *J* = 8.0 Hz), 7.61 (d, 2H, *J* = 8.5 Hz), 7.64 (d, 1H, *J* = 8.0 Hz), 7.88 (d, 1H, *J* = 8.0 Hz), 7.88 (d, 1H, *J* = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 55.3, 94.7, 114.0, 122.6, 124.0, 125.1, 127.3, 127.4, 127.5, 129.0, 132.0, 134.9, 147.4, 152.4, 159.9, 160.4; MS (FAB) *m/z* 276 (M^+, 100%); Anal. Calcd for $C_{18}H_{16}N_2O$: C, 78.24; H, 5.84; N, 10.14%, Found: C, 78.40; H, 5.76; N, 10.06%.

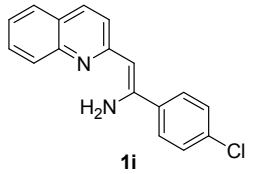
2-(Quinolin-2-yl)-1-(4-methylphenyl)-1-ethenamine[1] **(1h)**



1h

30 mmol scale; 78% yield; a yellow solid (AcOEt-hexane); mp 142.8-144.1 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.41 (s, 3H), 5.60 (s, 1H), 7.14 (d, 1H, *J* = 8.0 Hz), 7.25 (d, 2H, *J* = 8.0 Hz), 7.34 (t, 1H, *J* = 8.0 Hz), 7.57-7.61 (m, 3H), 7.65 (d, 1H, *J* = 8.0 Hz), 7.89 (d, 1H, *J* = 8.0 Hz), 7.90 (d, 1H, *J* = 8.0 Hz); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 21.3, 95.0, 122.7, 124.0, 125.1, 126.0, 127.3, 127.5, 129.0, 129.3, 134.9, 136.7, 139.1, 147.4, 152.6, 159.9; MS (FAB) *m/z* 261 (M+H, 100%); Anal. Calcd for $C_{18}H_{16}N_2$: C, 83.04; H, 6.19; N, 10.76%, Found: C, 83.20; H, 6.37; N, 10.63%.

2-(Quinolin-2-yl)-1-(4-chlorophenyl)-1-ethenamine[1] **(1i)**

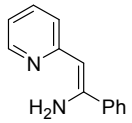


1i

30 mmol scale; 99% yield; a yellow solid (AcOEt-hexane); mp 167.8-169.0 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.55 (s, 1H), 7.10 (d, 1H, *J* = 8.5 Hz), 7.33-7.37 (m, 3H), 7.54-7.60 (m, 3H), 7.64 (d, 1H, *J* = 8.5 Hz), 7.87 (d, 1H, *J* = 8.5 Hz), 7.87 (d, 1H, *J* = 8.5 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 95.9, 122.6, 124.3, 125.2, 127.3, 127.4, 127.6, 128.9, 129.1, 134.8, 135.1, 138.0, 147.3, 151.2, 159.5; MS (FAB) *m/z* 281

(M+H, 100%); Anal. Calcd for $C_{17}H_{13}ClN_2$: C, 72.73; H, 4.67; N, 9.98%, Found: C, 72.69; H, 4.68; N, 9.94%.

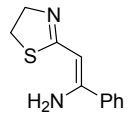
2-(Pyridin-2-yl)-1-phenyl-1-ethenamine[4] **(1j)**



1j

30 mmol scale; 80% yield; a pale yellow solid (AcOEt-hexane); mp 42.2-44.0 °C (lit.[4] 50-52 °C); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.48 (s, 1H), 6.83 (br, 2H), 6.84 (dd, 1H, *J* = 7.5, 5.0 Hz), 7.00 (d, 1H, *J* = 7.5 Hz), 7.33-7.40 (m, 3H), 7.47 (t, 1H, *J* = 7.5 Hz), 7.62 (d, 2H, *J* = 7.5 Hz), 8.44 (d, 1H, *J* = 5.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 95.7, 117.3, 122.2, 126.0, 128.5, 128.7, 135.5, 140.0, 147.4, 150.3, 159.8; MS (FAB) *m/z* 196 (M^+, 100%).

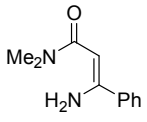
2-(Thiazolin-2-yl)-1-phenyl-1-ethenamine[5] **(1k)**



1k

30 mmol scale; 52% yield; yellow liquid; bp 180 °C / 4 mmHg (lit.[5] mp 26-28 °C); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.23 (t, 2H, *J* = 8.0 Hz), 4.35 (t, 2H, *J* = 8.0 Hz), 5.16 (s, 1H), 6.82 (br, 2H), 7.38-7.41 (m, 3H), 7.54-7.56 (m, 2H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 32.8, 64.4, 88.7, 126.1, 128.7, 129.6, 138.0, 153.7, 167.1; MS (FAB) *m/z* 205 (M+H, 100%).

2-[(Dimethylamino)carbonyl]-1-phenyl-1-ethenamine[6] **(1l)**

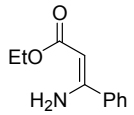


1l

67% yield; a pale yellow solid (AcOEt-hexane); mp 85.4-86.9 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.00 (s, 6H), 5.06 (s, 1H), 7.36-7.39 (m, 3H), 7.51-7.53 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 35.0, 37.3, 83.8, 126.1, 128.6, 129.5, 139.1, 158.5, 170.6; MS (FAB) *m/z* 191 (M+H, 100%).

Procedure for the synthesis of enamine 1m[7]: To an EtOH (80 mL) solution of benzoylacetic acid ethyl ester (9.61 g, 50.0 mmol) was added ammonium acetate (5.78 g, 75.0 mmol) at room temperature, and the mixture was heated at 80 °C for 24 h. The mixture was cooled to room temperature and the solvent was evaporated under reduced pressure. Water was added to the resulting residue. The mixture was extracted several times with AcOEt, and the combined organic extracts were dried over Na_2SO_4, filtered, and then concentrated under reduced pressure. The crude product was purified by distillation under reduced pressure to give enamine **1m** (4.78 g, 50%) as a colorless liquid.

Ethyl 1-phenyl-1-ethenamin-2-calboxylate[7] (1m)

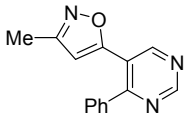


1m

bp 123 °C / 0.5 mmHg (lit.[8] 112 °C / 0.3 mmHg); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.27 (t, 3H, *J* = 7.0 Hz), 4.15 (q, 2H, *J* = 7.0 Hz), 4.94 (s, 1H), 7.37-7.40 (m, 3H), 7.50-7.52 (m, 2H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 14.5, 58.8, 84.5, 126.1, 128.7, 130.1, 137.6, 160.4, 170.3; MS (EI) *m/z* 191 (M^+, 100%).

General procedure for the synthesis of pyrimidine 4 or 9: To a PhMe (1 mL) solution of $ZnCl_2$ (6.8mg, 0.050 mmol) and acetal **2** (220 mg, 1.5 mmol) in a screw-capped vial were added enamine **1** (0.50 mmol) (or ketone **8**) and ammonium acetate (77 mg, 1.0 mmol), and the vial was sealed with a cap containing a PTFE septum. The mixture was heated at 100 °C and monitored by TLC or GC analysis until the enamine had been consumed. To quench the reaction, a saturated aqueous solution of $NaHCO_3$ (5 mL) was added to the mixture. The mixture was extracted several times with $CHCl_3$, and the combined organic extracts were dried over Na_2SO_4, filtered, and then concentrated under reduced pressure. The crude product was purified by silica gel chromatography (AcOEt-hexane) to produce pyrimidine **4** (or **9**).

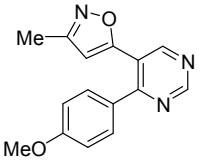
4-Phenyl-5-(3'-methylisoxazol-5'-yl)-pyrimidine (4a)



4a

0.50 mmol scale; 99% yield; a pale yellow solid (CH_2Cl_2-hexane); mp 72.5-73.2 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.27 (s, 3H), 5.81 (s, 1H), 7.45-7.53 (m, 5H), 9.09 (s, 1H), 9.29 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.3, 105.3, 120.6, 128.6, 128.7, 130.3, 136.8, 156.8, 158.7, 160.1, 164.0, 164.7; MS (FAB) *m/z* 238 (M+H, 100%); Anal. Calcd for $C_{14}H_{11}N_3O$: C, 70.87; H, 4.67; N, 17.71%, Found: C, 70.53; H, 4.69; N, 17.80%.

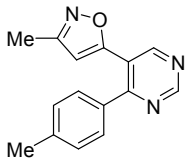
4-(4-Methoxyphenyl)-5-(3'-methylisoxazol-5'-yl)-pyrimidine (4b)



4b

0.50 mmol scale; 80% yield; a pale yellow solid (CH_2Cl_2-hexane); mp 88.4-89.2 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.30 (s, 3H), 3.87 (s, 3H), 5.95 (s, 1H), 6.95 (d, 2H, *J* = 8.5 Hz), 7.51 (d, 2H, *J* = 8.5 Hz), 8.99 (s, 1H), 9.25 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.4, 55.4, 105.0, 114.1, 120.1, 129.0, 130.6, 157.0, 158.8, 160.2, 161.5, 163.5, 165.6; MS (FAB) *m/z* 268 (M+H, 100%); Anal. Calcd for $C_{15}H_{13}N_3O_2$: C, 67.40; H, 4.90; N, 15.72%, Found: C, 67.50; H, 4.85; N, 15.65%.

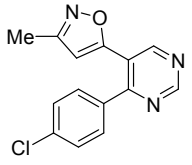
4-(4-Methylphenyl)-5-(3'-methylisoxazol-5'-yl)-pyrimidine (4c)



4c

0.50 mmol scale; 97% yield; a brown solid (CH_2Cl_2-hexane); mp 106.7-107.5 ^oC; ^1H NMR ($CDCl_3$, 300 MHz) δ 2.28 (s, 3H), 2.42 (s, 3H), 5.88 (s, 1H), 7.25 (d, 2H, *J* = 8.4 Hz), 7.43 (d, 2H, *J* = 8.4 Hz), 9.04 (s, 1H), 9.27 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 11.3, 21.3, 105.0, 120.3, 128.6, 129.2, 133.8, 140.6, 156.7, 158.6, 160.0, 163.9, 164.9; MS (FAB) *m/z* 252 (M+H, 100%); Anal. Calcd for $C_{15}H_{13}N_3O$: C, 71.70; H, 5.21; N, 16.72%, Found: C, 71.64; H, 5.24; N, 16.54%.

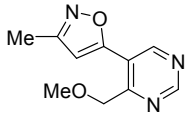
4-(4-Chlorophenyl)-5-(3'-methylisoxazol-5'-yl)-pyrimidine (4d)



4d

0.50 mmol scale; 80% yield; a colorless solid (CH_2Cl_2-hexane); mp 109.8-110.2 ^oC; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.24 (s, 3H), 5.87 (s, 1H), 7.36 (d, 2H, *J* = 8.5 Hz), 7.42 (d, 2H, *J* = 8.5 Hz), 8.98 (s, 1H), 9.22 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.5, 105.3, 120.5, 129.0, 130.1, 135.1, 136.8, 157.2, 158.9, 160.3, 162.8, 164.6; MS (FAB) *m/z* 272 (M+H, 100%); Anal. Calcd for $C_{14}H_{10}ClN_3O$: C, 61.89; H, 3.71; N, 15.47%, Found: C, 61.74; H, 3.79; N, 15.58%.

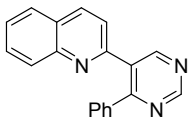
4-Methoxymethyl-5-(3'-methylisoxazol-5'-yl)-pyrimidine (4e)



4e

0.50 mmol scale; 82% yield; pale yellow oil; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.42 (s, 3H), 3.50 (s, 3H), 4.74 (s, 2H), 6.59 (s, 1H), 9.07 (s, 1H), 9.26 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.3, 58.8, 73.1, 105.6, 121.1, 156.0, 158.6, 160.4, 161.9, 163.6; MS (FAB) *m/z* 206 (M+H, 100%); HRMS (FAB): Calcd for $C_{10}H_{12}N_3O_2$: 206.0930, Found 206.0953 (M+H).

4-Phenyl-5-(quinolin-2-yl)-pyrimidine (4f)

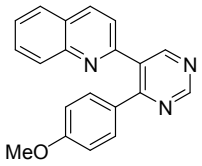


4f

0.50 mmol scale; 91% yield; a pale yellow solid (CH_2Cl_2-hexane); mp 66.4-68.0 ^oC; ^1H NMR ($CDCl_3$, 500 MHz) δ 7.05 (d, 1H, *J* = 8.0 Hz), 7.30 (t, 2H, *J* = 8.0 Hz), 7.38 (t, 1H, *J* = 8.0 Hz), 7.49 (d, 2H, *J* = 8.0 Hz), 7.60 (t, 1H, *J* = 8.0 Hz), 7.78-7.82 (m, 2H), 7.96 (d, 1H, *J* = 8.0 Hz), 8.20 (d, 1H, *J* = 8.0 Hz), 9.17 (s, 1H), 9.36 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 122.6, 127.0, 127.2, 127.6, 128.5, 129.7, 129.8,

129.9, 130.0, 132.5, 135.9, 137.3, 148.5, 155.2, 158.3, 159.2, 163.7; MS (FAB) *m/z* 284 (M+H, 100%); Anal. Calcd for $C_{19}H_{13}N_3$: C, 80.54; H, 4.62; N, 14.83%, Found: C, 80.43; H, 4.64; N, 14.75%.

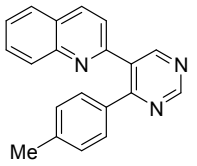
4-(4-Methoxyphenyl)-5-(quinolin-2-yl)-pyrimidine (4g)



4g

0.50 mmol scale; 90% yield; yellow oil; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.78 (s, 3H), 6.79 (d, 2H, *J* = 8.5 Hz), 7.10 (d, 1H, *J* = 8.0 Hz), 7.45 (d, 2H, *J* = 8.5 Hz), 7.59 (t, 1H, *J* = 8.0 Hz), 7.78 (t, 1H, *J* = 8.0 Hz), 7.81 (d, 1H, *J* = 8.0 Hz), 7.98 (d, 1H, *J* = 8.0 Hz), 8.20 (d, 1H, *J* = 8.0 Hz), 9.10 (s, 1H), 9.30 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 55.2, 113.8, 122.6, 126.9, 127.1, 127.6, 129.3, 129.6, 129.9, 131.4, 131.9, 135.9, 148.4, 155.6, 158.1, 158.9, 161.1, 163.0; MS (FAB) *m/z* 314 (M+H, 100%); HRMS (FAB): Calcd for $C_{20}H_{16}N_3O$: 314.1293, Found 314.1292 (M+H).

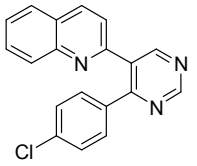
4-(4-Methylphenyl)-5-(quinolin-2-yl)-pyrimidine (4h)



4h

0.50 mmol scale; 94% yield; a colorless solid (CH_2Cl_2-hexane); mp 137.5-138.3 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.21 (s, 3H), 6.95-6.98 (m, 1H), 6.97 (d, 2H, *J* = 8.0 Hz), 7.28 (d, 2H, *J* = 8.0 Hz), 7.47 (t, 1H, *J* = 8.0 Hz), 7.64-7.69 (m, 2H), 7.83 (d, 1H, *J* = 8.0 Hz), 8.09 (d, 1H, *J* = 8.0 Hz), 9.03 (s, 1H), 9.22 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 21.2, 122.5, 126.8, 127.0, 127.5, 129.1, 129.5, 129.6, 129.8, 132.1, 134.2, 135.8, 140.1, 148.3, 155.4, 158.0, 158.9, 163.5; MS (FAB) *m/z* 298 (M+H, 100%); HRMS (FAB): Calcd for $C_{20}H_{16}N_3$: 298.1344, Found 298.1337 (M+H).

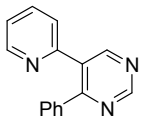
4-(4-Chlorophenyl)-5-(quinolin-2-yl)-pyrimidine (4i)



4i

0.50 mmol scale; 77% yield; a brown solid (CH_2Cl_2-hexane); mp 156.0-158.5 ^{o}C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.08 (d, 1H, *J* = 8.0 Hz), 7.27 (d, 2H, *J* = 8.5 Hz), 7.43 (d, 2H, *J* = 8.5 Hz), 7.62 (t, 1H, *J* = 8.0 Hz), 7.81 (t, 1H, *J* = 8.0 Hz), 7.84 (d, 1H, *J* = 8.0 Hz), 8.02 (d, 1H, *J* = 8.0 Hz), 8.18 (d, 1H, *J* = 8.0 Hz), 9.16 (s, 1H), 9.35 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 122.4, 127.0, 127.4, 127.7, 128.9, 129.7, 130.2, 131.1, 132.5, 135.6, 136.3, 136.3, 148.5, 154.9, 158.3, 159.3, 162.4; MS (FAB) *m/z* 318 (M+H, 100%); HRMS (FAB): Calcd for $C_{19}H_{13}ClN_3$: 318.0798, Found 318.0797 (M+H).

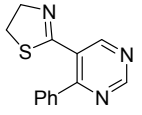
4-Phenyl-5-(pyridin-2-yl)-pyrimidine (4j)



4j

0.50 mmol scale; 46% yield; a pale yellow solid (CH_2Cl_2-hexane); mp 100.9-102.2 ^oC; ^1H NMR ($CDCl_3$, 500 MHz) δ 7.06 (d, 1H, *J* = 8.0 Hz), 7.26 (dd, 1H, *J* = 8.0, 5.0 Hz), 7.30-7.33 (m, 2H), 7.37-7.40 (m, 1H), 7.43-7.45 (m, 2H), 7.56 (t, 1H, *J* = 8.0 Hz), 8.72 (d, 1H, *J* = 5.0 Hz), 9.01 (s, 1H), 9.31 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 122.7, 125.0, 128.4, 129.6, 129.7, 132.2, 136.1, 137.3, 150.3, 154.9, 158.1, 158.7, 163.7; MS (FAB) *m/z* 234 (M+H, 100%); Anal. Calcd for $C_{15}H_{11}N_3$: C, 77.23; H, 4.75; N, 18.01%, Found: C, 76.94; H, 4.87; N, 17.61%.

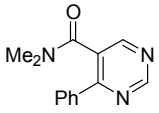
4-Phenyl-5-(thiazolin-2-yl)-pyrimidine (4k)



4k

0.50 mmol scale; 24% yield; a yellow solid (CH_2Cl_2-hexane); mp 73.6-74.4 ^oC; ^1H NMR ($CDCl_3$, 500 MHz) δ 3.40 (t, 2H, *J* = 8.5 Hz), 4.38 (t, 2H, *J* = 8.5 Hz), 7.45-7.50 (m, 3H), 7.70-7.71 (m, 2H), 8.94 (s, 1H), 9.29 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 35.4, 65.4, 126.9, 128.4, 129.3, 130.4, 136.7, 157.4, 159.1, 164.3, 165.2; MS (FAB) *m/z* 242 (M+H, 100%); HRMS (FAB): Calcd for $C_{13}H_{12}N_3S$: 242.0752, Found 242.0758 (M+H).

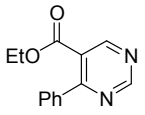
4-Phenyl-5-[(dimethylamino)carbonyl]-pyrimidine (4l)



4l

0.50 mmol scale; 26% yield; a colorless solid (CH_2Cl_2-hexane); mp 76.9-77.4 ^oC; ^1H NMR ($CDCl_3$, 500 MHz) δ 2.45 (s, 3H), 2.99 (s, 3H), 7.47-7.52 (m, 3H), 7.81-7.83 (m, 2H), 8.78 (s, 1H), 9.30 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 34.9, 37.9, 128.5, 128.6, 128.9, 130.9, 136.6, 156.5, 158.9, 161.4, 167.5; MS (FAB) *m/z* 228 (M+H, 100%); Anal. Calcd for $C_{13}H_{13}N_3O$: C, 68.70; H, 5.77; N, 18.49%, Found: C, 68.91; H, 5.57; N, 18.54%.

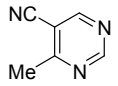
Ethyl 4-phenylpyrimidin-5-calboxylate[9] (4m)



4m

0.50 mmol scale; 71% yield; colorless oil; ^1H NMR ($CDCl_3$, 500 MHz) δ 1.14 (t, 3H, *J* = 7.0 Hz), 4.25 (q, 2H, *J* = 7.0 Hz), 7.46-7.51 (m, 3H), 7.61-7.63 (m, 2H), 9.09 (s, 1H), 9.33 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 13.7, 61.9, 124.8, 128.3, 128.7, 130.3, 137.3, 158.1, 159.5, 165.5, 166.1; MS (EI) *m/z* 228 (M^+, 100%).

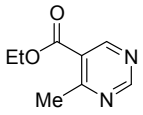
4-Methyl-5-cyano-pyrimidine (4n)



4n

0.50 mmol scale; 65% yield; pale yellow oil; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.70 (s, 3H), 8.84 (s, 1H), 9.16 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 23.2, 109.2, 114.2, 159.5, 159.6, 170.0; MS (EI) *m/z* 119 (M^+, 100%); Anal. Calcd for $C_6H_5N_3$: C, 60.50; H, 4.23; N, 35.27%, Found: C, 60.89; H, 3.91; N, 35.27%.

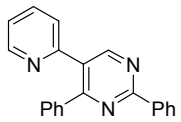
Ethyl 4-methylpyrimidin-5-carboxylate[10] (4o)



4o

0.50 mmol scale; 77% yield; pale yellow oil; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.43 (t, 3H, *J* = 7.0 Hz), 2.84 (s, 3H), 4.43 (q, 2H, *J* = 7.0 Hz), 9.14 (s, 1H), 9.16 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 14.1, 24.2, 61.6, 123.7, 158.4, 159.9, 164.6, 168.4; MS (FAB) *m/z* 167 (M+H, 100%).

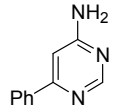
2,4-Diphenyl-5-(pyridin-2-yl)-pyrimidine (5)



5

a colorless crystal (CH_2Cl_2-hexane); mp 136.2-137.5 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.08 (d, 1H, *J* = 7.5 Hz), 7.23 (dd, 1H, *J* = 7.5, 5.0 Hz), 7.32-7.35 (m, 2H), 7.39 (t, 1H, *J* = 7.5 Hz), 7.50-7.56 (m, 6H), 8.59-8.61 (m, 2H), 8.72 (d, 1H, *J* = 5.0 Hz), 9.07 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 122.4, 125.0, 128.3, 128.5, 128.5, 129.6, 129.8, 129.8, 130.8, 136.0, 137.4, 137.9, 150.2, 155.4, 159.2, 163.7, 163.7; MS (FAB) *m/z* 310 (M+H, 100%); Anal. Calcd for $C_{21}H_{15}N_3$: C, 81.53; H, 4.89; N, 13.58%, Found: C, 81.30; H, 4.99; N, 13.42%.

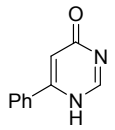
4-Amino-6-phenylpyrimidine[11] (6)



6

a brown solid; mp 225.3-228.3 °C (lit.[11] 226-228 °C); ^{1}H NMR (DMSO, 500 MHz) δ 6.87 (s, 1H), 6.91 (br, 2H), 7.46-7.50 (m, 3H), 7.95-7.97 (m, 2H), 8.43 (s, 1H); ^{13}C NMR (DMSO, 125 MHz) δ 99.7, 126.2, 128.7, 129.9, 137.3, 158.6, 160.8, 164.4; MS (FAB) *m/z* 172 (M+H, 100%).

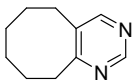
6-Phenylpyrimidin-4(1H)-one[11] (7)



7

a colorless solid; mp 273.3-274.5 °C (lit.[11] 270-272 °C); ^{1}H NMR (DMSO, 500 MHz) δ 6.86 (s, 1H), 7.46-7.47 (m, 3H), 8.01-8.03 (m, 2H), 8.26 (s, 1H); ^{13}C NMR (DMSO, 75 MHz) δ 109.7, 126.9, 128.8, 130.6, 136.1, 149.9, 160.7, 161.8; MS (FAB) *m/z* 173 (M+H, 100%); Anal. Calcd for $C_{10}H_8N_2O$: C, 69.76; H, 4.68; N, 16.27%, Found: C, 70.06; H, 4.79; N, 16.07%.

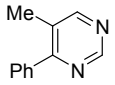
5,6,7,8,9,10-Hexahydrocycloocta[d]pyrimidine[12] (9a)



9a

0.50 mmol scale; 86% yield; pale yellow oil; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.30-1.35 (m, 4H), 1.63-1.67 (m, 2H), 1.73-1.78 (m, 2H), 2.69 (t, 2H, *J* = 6.5 Hz), 2.85 (t, 2H, *J* = 6.5 Hz), 8.32 (s, 1H), 8.90 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 25.6, 25.7, 28.9, 29.9, 31.8, 33.9, 133.9, 156.1, 156.8, 169.3; MS (EI) *m/z* 162 (M^+, 100%).

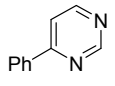
4-Phenyl-5-methylpyrimidine[13] (9b)



9b

0.50 mmol scale; 70% yield; a pale yellow solid; mp 30.2-30.4 °C (lit.[13] 29-31 °C); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 2.32 (s, 3H), 7.39-7.44 (m, 3H), 7.53-7.55 (m, 2H), 8.54 (s, 1H), 9.05 (s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 17.1, 128.2, 128.4, 128.8, 129.4, 137.8, 156.6, 158.7, 164.9; MS (EI) *m/z* 170 (M^+, 100%).

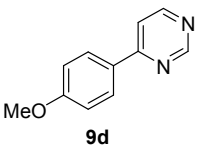
4-Phenylpyrimidine[14] (9c)



9c

0.50 mmol scale; 70% yield; a colorless crystal; mp 57.8-58.5 °C (lit.[14] 60-62 °C); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.39-7.41 (m, 3H), 7.58 (dd, 1H, *J* = 5.5, 1.5 Hz), 7.96-7.99 (m, 2H), 8.63 (d, 1H, *J* = 5.5 Hz), 9.16 (d, 1H, *J* = 1.5 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 116.8, 127.0, 128.9, 130.9, 136.3, 157.3, 158.9, 163.7; MS (EI) *m/z* 156 (M^+, 100%).

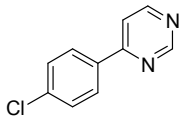
4-(4-Methoxyphenyl)-pyrimidine[14] (9d)



9d

0.50 mmol scale; 54% yield; a pale yellow solid; mp 79.7-80.3 °C (lit.[14] 76-79 °C); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 3.88 (s, 3H), 7.02 (d, 2H, *J* = 8.5 Hz), 7.65 (d, 1H, *J* = 5.0 Hz), 8.08 (d, 2H, *J* = 8.5 Hz), 8.70 (d, 1H, *J* = 5.0 Hz), 9.21 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 55.5, 114.4, 116.1, 128.7, 129.0, 157.2, 159.1, 162.2, 163.4; MS (EI) *m/z* 186 (M^+, 100%).

4-(4-Chlorophenyl)-pyrimidine[15] (9e)

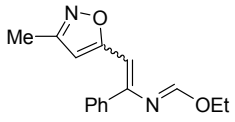


9e

0.50 mmol scale; 61% yield; a pale yellow solid; mp 75.9-76.8 °C (lit.[15] 84-90 °C); ^{1}H NMR ($CDCl_3$, 500 MHz) δ 7.48 (d, 2H, *J* = 8.5 Hz), 7.68 (dd, 1H, *J* = 5.5, 1.5 Hz), 8.04 (d, 2H, *J* = 8.5 Hz), 8.77 (d, 1H, *J* = 5.5 Hz), 9.26 (d, 1H, *J* = 1.5 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 116.7, 128.4, 129.3, 134.9, 137.4, 157.6, 159.1, 162.6; MS (EI) *m/z* 190 (M^+, 100%).

Procedure for the synthesis of 2-azadiene 10a: To a PhMe (1 mL) solution of enamine **1a** (100 mg, 0.50 mmol) in a screw-capped vial was added acetal **2** (74 mg, 0.50 mmol) at room temperature, and the vial was sealed with a cap containing a PTFE septum. The reaction mixture was heated at 100 °C. After 16 h, the mixture was cooled to room temperature and the solvent was evaporated under reduced pressure. The crude product was purified by silica gel chromatography to give 2-azadiene **10a** as pale yellow oil.

1-Ethoxy-4-(3'-methylisoxazol-5'-yl)-3-phenyl-2-aza-1,3-butadiene (10a)

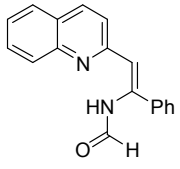


10a

34% NMR yield; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 1.46 (t, 3H, *J* = 7.0 Hz), 2.30 (s, 3H), 4.48 (q, 2H, *J* = 7.0 Hz), 6.12 (s, 1H), 6.37 (s, 1H), 7.35-7.40 (m, 3H), 7.52-7.53 (m, 2H), 7.58 (s, 1H); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 11.5, 14.3, 62.9, 100.3, 102.3, 126.5, 128.6, 129.0, 138.0, 149.4, 156.9, 159.6, 168.0; MS (FAB) *m/z* 257 (M+H, 100%); HRMS (FAB): Calcd for $C_{15}H_{17}N_2O_2$: 257.1290, Found 257.1262 (M+H).

Procedure for the synthesis of compound 13a: To a screw-capped vial were added enamine **1f** (74 mg, 0.30 mmol), acetal **2** (890 mg, 6.0 mmol), ammonium acetate (46 mg, 0.6 mmol), and $ZnCl_2$ (41 mg, 0.30 mmol) at room temperature, and the vial was sealed with a cap containing a PTFE septum. The reaction mixture was heated at 100 °C for 1 h. To quench the reaction, water (5 ml) was added to the mixture. The mixture was extracted several times with $CHCl_3$, and the combined organic extracts were dried over Na_2SO_4, filtered, and then concentrated under reduced pressure. The crude product was purified by silica gel chromatography to give compound **13a** as a yellow crystal (AcOEt-hexane).

***N*-{1-Phenyl-2-(quinolin-2-yl)vinyl}formamide (13a)**



13a

33% NMR yield; mp 83.5-84.0 °C; ^{1}H NMR ($CDCl_3$, 500 MHz) δ 5.75 (s, 1H), 7.23 (d, 1H, *J* = 8.5 Hz), 7.47-7.51 (m, 4H), 7.55-7.57 (m, 2H), 7.72 (t, 1H, *J* = 8.5 Hz), 7.76 (d, 1H, *J* = 8.5 Hz), 8.09 (d, 1H, *J* = 8.5 Hz), 8.13 (d, 1H, *J* = 8.5 Hz), 8.56 (d, 1H, *J* = 10.0 Hz), 13.42 (d, 1H, *J* = 10.0 Hz); ^{13}C NMR ($CDCl_3$, 125 MHz) δ 107.5, 122.7, 126.1, 126.1, 127.4, 128.5, 128.6, 128.9, 129.5, 130.0, 135.5, 136.6, 144.6, 146.8, 156.4, 163.3; MS (FAB) *m/z* 275 (M+H, 100%); HRMS (FAB): Calcd for $C_{18}H_{15}N_2O$: 275.1184, Found 275.1169 (M+H).

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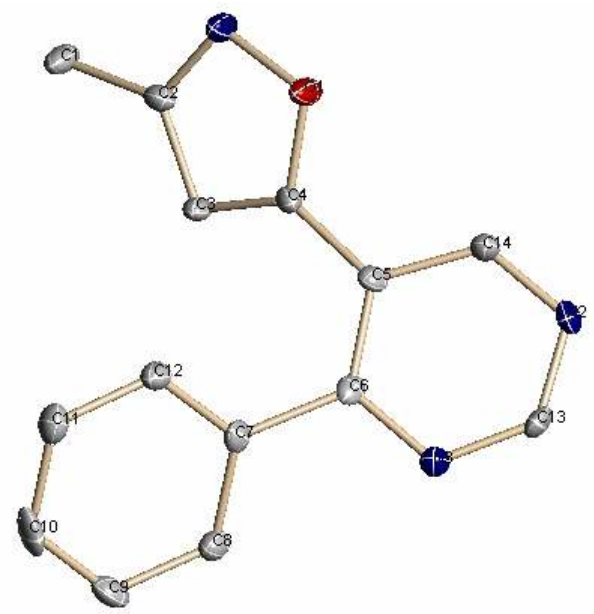
X-ray data for pyrimidine **4a**



ORTEP Drawing of **4a**

Table 1. Crystal data and structure refinement for 4a.

Identification code	4a	
Empirical formula	C14 H11 N3 O	
Formula weight	237.26	
Temperature	273(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 5.613(3) Å	α= 90°.
	b = 9.682(5) Å	β= 92.403(8)°.
	c = 21.629(10) Å	γ = 90°.
Volume	1174.4(9) $Å^3$	
Z	4	
Density (calculated)	1.342 Mg/m^3	
Absorption coefficient	0.088 mm^{-1}	
F(000)	496	
Crystal size	0.56 x 0.19 x 0.10 mm^3	
Theta range for data collection	1.88 to 28.29°.	
Index ranges	-6<=h<=7, -12<=k<=10, -28<=l<=27	
Reflections collected	7004	
Independent reflections	2694 [R(int) = 0.0558]	
Completeness to theta = 28.29°	92.2 %	
Absorption correction	Empirical	
Max. and min. transmission	-183 and -183	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2694 / 0 / 164	
Goodness-of-fit on F^2	1.311	
Final R indices [I>2sigma(I)]	R1 = 0.1099, wR2 = 0.2184	
R indices (all data)	R1 = 0.1219, wR2 = 0.2240	
Largest diff. peak and hole	0.501 and -0.360 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 4a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	-1507(4)	6646(3)	795(1)	21(1)
N(3)	283(5)	11388(3)	1107(1)	17(1)
N(2)	-2841(5)	10783(3)	380(1)	18(1)
C(5)	-423(6)	9022(3)	872(2)	14(1)
N(1)	-485(5)	5322(3)	758(2)	22(1)
C(12)	2299(6)	8747(4)	2153(2)	18(1)
C(9)	6207(6)	10484(4)	2276(2)	23(1)
C(1)	3480(7)	4318(4)	786(2)	23(1)
C(14)	-2279(6)	9467(4)	472(2)	17(1)
C(4)	269(6)	7572(4)	874(2)	15(1)
C(7)	2594(6)	9797(3)	1723(2)	15(1)
C(6)	778(6)	10057(3)	1217(2)	14(1)
C(10)	5897(7)	9448(4)	2699(2)	26(1)
C(2)	1795(6)	5508(4)	813(2)	18(1)
C(8)	4535(6)	10680(4)	1796(2)	17(1)
C(3)	2375(6)	6919(3)	897(2)	17(1)
C(11)	3946(7)	8580(4)	2638(2)	24(1)
C(13)	-1459(6)	11675(3)	692(2)	17(1)

Table 3. Bond lengths [Å] and angles [°] for 4a.

O(1)-C(4)	1.346(4)
O(1)-N(1)	1.408(4)
N(3)-C(13)	1.329(4)
N(3)-C(6)	1.338(4)
N(2)-C(14)	1.325(4)
N(2)-C(13)	1.326(4)
C(5)-C(14)	1.394(5)
C(5)-C(6)	1.404(5)
C(5)-C(4)	1.457(5)
N(1)-C(2)	1.293(5)
C(12)-C(11)	1.379(5)
C(12)-C(7)	1.392(5)
C(9)-C(10)	1.373(6)
C(9)-C(8)	1.383(5)
C(1)-C(2)	1.493(5)
C(4)-C(3)	1.340(5)
C(7)-C(8)	1.389(5)
C(7)-C(6)	1.486(5)
C(10)-C(11)	1.382(6)
C(2)-C(3)	1.414(5)

C(4)-O(1)-N(1)	108.2(3)
C(13)-N(3)-C(6)	117.6(3)
C(14)-N(2)-C(13)	114.8(3)
C(14)-C(5)-C(6)	116.0(3)
C(14)-C(5)-C(4)	119.4(3)
C(6)-C(5)-C(4)	124.4(3)
C(2)-N(1)-O(1)	105.8(3)
C(11)-C(12)-C(7)	119.9(3)
C(10)-C(9)-C(8)	119.9(3)
N(2)-C(14)-C(5)	123.9(3)
C(3)-C(4)-O(1)	109.8(3)
C(3)-C(4)-C(5)	133.6(3)
O(1)-C(4)-C(5)	116.4(3)
C(8)-C(7)-C(12)	119.3(3)
C(8)-C(7)-C(6)	119.3(3)
C(12)-C(7)-C(6)	121.2(3)
N(3)-C(6)-C(5)	120.1(3)

N(3)-C(6)-C(7)	115.2(3)
C(5)-C(6)-C(7)	124.7(3)
C(9)-C(10)-C(11)	120.3(3)
N(1)-C(2)-C(3)	111.6(3)
N(1)-C(2)-C(1)	121.0(3)
C(3)-C(2)-C(1)	127.4(3)
C(9)-C(8)-C(7)	120.3(3)
C(4)-C(3)-C(2)	104.7(3)
C(12)-C(11)-C(10)	120.3(4)
N(2)-C(13)-N(3)	127.2(3)

Symmetry transformations used to generate equivalent atoms:

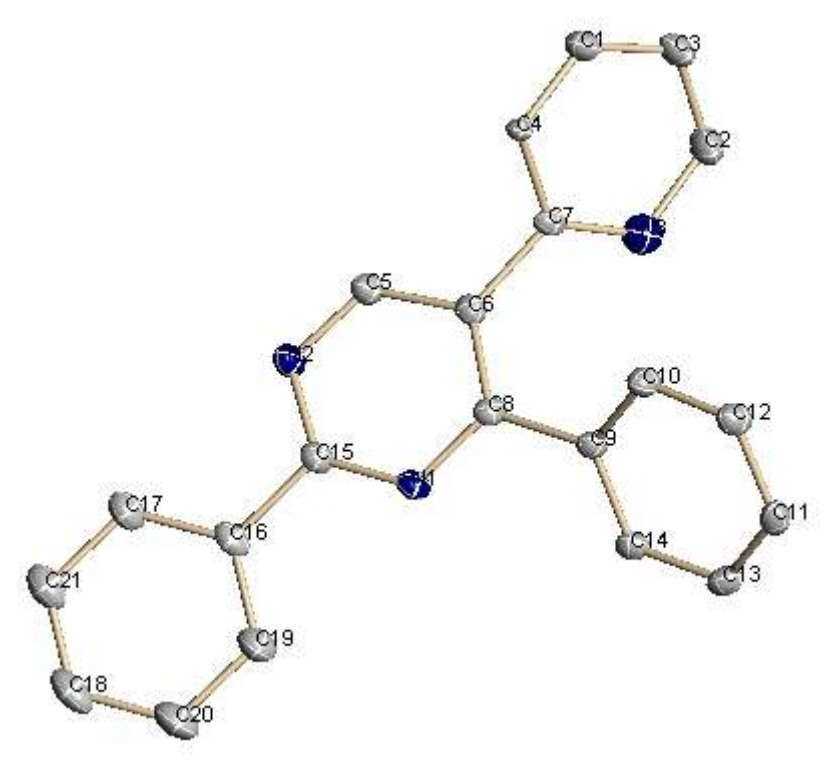
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 4a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	12(1)	12(1)	39(2)	0(1)	-4(1)	0(1)
N(3)	17(2)	14(1)	19(1)	0(1)	-3(1)	0(1)
N(2)	14(2)	19(2)	22(2)	2(1)	-6(1)	1(1)
C(5)	9(2)	14(2)	20(2)	-1(1)	-1(1)	-2(1)
N(1)	17(2)	11(1)	37(2)	-1(1)	-6(1)	1(1)
C(12)	14(2)	13(2)	26(2)	1(1)	-3(1)	-1(1)
C(9)	14(2)	24(2)	30(2)	-5(2)	-5(1)	-3(2)
C(1)	17(2)	12(2)	41(2)	4(2)	-2(2)	-2(1)
C(14)	18(2)	12(2)	19(2)	-1(1)	-5(1)	-1(1)
C(4)	15(2)	13(2)	17(2)	4(1)	-5(1)	-3(1)
C(7)	14(2)	12(2)	19(2)	0(1)	-1(1)	5(1)
C(6)	8(2)	14(2)	20(2)	3(1)	3(1)	1(1)
C(10)	27(2)	35(2)	16(2)	-3(2)	-12(2)	11(2)
C(2)	18(2)	15(2)	19(2)	1(1)	-5(1)	-5(1)
C(8)	16(2)	12(2)	24(2)	1(1)	-1(1)	1(1)
C(3)	13(2)	11(2)	28(2)	2(1)	-5(1)	-3(1)
C(11)	26(2)	21(2)	23(2)	6(2)	-5(2)	3(2)
C(13)	20(2)	8(2)	22(2)	2(1)	-1(1)	3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for 4a.

	x	y	z	U(eq)
H(12)	993	8157	2112	22
H(9)	7540	11054	2313	27
H(1A)	2619	3470	831	35
H(1B)	4677	4397	1114	35
H(1C)	4228	4322	395	35
H(14)	-3175	8801	257	20
H(10)	7003	9330	3027	32
H(8)	4713	11406	1520	21
H(3)	3883	7307	955	21
H(11)	3743	7881	2926	28
H(13)	-1741	12606	611	20

X-ray data for pyrimidine **5**



ORTEP Drawing of **5**

Table 1. Crystal data and structure refinement for 5.

Identification code	5	
Empirical formula	C21 H15 N3	
Formula weight	309.36	
Temperature	273 K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	Pbcn	
Unit cell dimensions	a = 14.874(2) Å	α= 90°.
	b = 8.8246(12) Å	β= 90°.
	c = 24.091(3) Å	γ = 90°.
Volume	3162.2(7) $Å^3$	
Z	8	
Density (calculated)	1.300 Mg/m^3	
Absorption coefficient	0.078 mm^{-1}	
F(000)	1296	
Crystal size	0.64 x 0.37 x 0.23 mm^3	
Theta range for data collection	1.69 to 27.58°.	
Index ranges	-18<=h<=19, -10<=k<=11, -27<=l<=31	
Reflections collected	18181	
Independent reflections	3610 [R(int) = 0.0397]	
Completeness to theta = 27.58°	98.4 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9822 and 0.9516	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3610 / 0 / 217	
Goodness-of-fit on F^2	0.817	
Final R indices [I>2sigma(I)]	R1 = 0.0713, wR2 = 0.2123	
R indices (all data)	R1 = 0.0781, wR2 = 0.2206	
Largest diff. peak and hole	0.822 and -0.818 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 5. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
N(1)	2621(1)	359(2)	5627(1)	17(1)
C(15)	2195(1)	-336(2)	5208(1)	17(1)
C(4)	-133(1)	1157(2)	6523(1)	11(1)
C(5)	815(1)	383(3)	5507(1)	18(1)
N(2)	1295(1)	-376(2)	5136(1)	19(1)
C(6)	1181(1)	1160(2)	5958(1)	17(1)
C(8)	2121(1)	1077(2)	6009(1)	16(1)
C(13)	3957(2)	2980(3)	6835(1)	20(1)
C(16)	2752(2)	-1163(3)	4796(1)	19(1)
C(17)	2346(2)	-2094(3)	4401(1)	24(1)
C(9)	2636(1)	1698(2)	6489(1)	16(1)
C(7)	555(1)	1999(3)	6328(1)	16(1)
C(14)	3446(1)	2446(2)	6392(1)	17(1)
C(18)	3806(2)	-2794(3)	4047(1)	30(1)
C(19)	3691(2)	-1037(3)	4805(1)	25(1)
C(1)	-761(2)	1871(3)	6824(1)	21(1)
C(10)	2360(2)	1450(3)	7033(1)	20(1)
C(11)	3684(2)	2705(3)	7375(1)	20(1)
C(12)	2890(2)	1933(3)	7474(1)	22(1)
C(20)	4210(2)	-1845(3)	4429(1)	31(1)
N(3)	636(2)	3543(3)	6422(1)	32(1)
C(21)	2874(2)	-2917(3)	4030(1)	27(1)
C(2)	-33(2)	4256(3)	6732(1)	25(1)
C(3)	-742(2)	3412(3)	6936(1)	23(1)

Table 3. Bond lengths [Å] and angles [°] for 5.

N(1)-C(15)	1.339(3)
N(1)-C(8)	1.343(3)
C(15)-N(2)	1.350(3)
C(15)-C(16)	1.485(3)
C(4)-C(1)	1.340(3)
C(4)-C(7)	1.348(3)
C(4)-H(4)	0.9300
C(5)-N(2)	1.327(3)
C(5)-C(6)	1.395(3)
C(5)-H(5)	0.9300
C(6)-C(8)	1.404(3)
C(6)-C(7)	1.487(3)
C(8)-C(9)	1.491(3)
C(13)-C(11)	1.387(3)
C(13)-C(14)	1.392(3)
C(13)-H(13)	0.9300
C(16)-C(17)	1.394(3)
C(16)-C(19)	1.401(3)
C(17)-C(21)	1.395(3)
C(17)-H(17)	0.9300
C(9)-C(10)	1.391(3)
C(9)-C(14)	1.394(3)
C(7)-N(3)	1.387(3)
C(14)-H(14)	0.9300
C(18)-C(20)	1.382(4)
C(18)-C(21)	1.391(4)
C(18)-H(18)	0.9300
C(19)-C(20)	1.388(3)
C(19)-H(19)	0.9300
C(1)-C(3)	1.387(3)
C(1)-H(1)	0.9300
C(10)-C(12)	1.390(3)
C(10)-H(10)	0.9300
C(11)-C(12)	1.385(3)
C(11)-H(11)	0.9300
C(12)-H(12)	0.9300
C(20)-H(20)	0.9300
N(3)-C(2)	1.393(3)

C(21)-H(21)	0.9300
C(2)-C(3)	1.382(3)
C(2)-H(2)	0.9300
C(3)-H(3)	0.9300

C(15)-N(1)-C(8)	118.05(18)
N(1)-C(15)-N(2)	125.31(19)
N(1)-C(15)-C(16)	117.68(19)
N(2)-C(15)-C(16)	116.98(19)
C(1)-C(4)-C(7)	117.26(19)
C(1)-C(4)-H(4)	121.4
C(7)-C(4)-H(4)	121.4
N(2)-C(5)-C(6)	124.21(19)
N(2)-C(5)-H(5)	117.9
C(6)-C(5)-H(5)	117.9
C(5)-N(2)-C(15)	115.71(18)
C(5)-C(6)-C(8)	115.57(19)
C(5)-C(6)-C(7)	117.77(18)
C(8)-C(6)-C(7)	126.65(19)
N(1)-C(8)-C(6)	121.03(19)
N(1)-C(8)-C(9)	114.85(18)
C(6)-C(8)-C(9)	124.08(18)
C(11)-C(13)-C(14)	120.1(2)
C(11)-C(13)-H(13)	120.0
C(14)-C(13)-H(13)	120.0
C(17)-C(16)-C(19)	119.3(2)
C(17)-C(16)-C(15)	120.2(2)
C(19)-C(16)-C(15)	120.4(2)
C(21)-C(17)-C(16)	120.0(2)
C(21)-C(17)-H(17)	120.0
C(16)-C(17)-H(17)	120.0
C(10)-C(9)-C(14)	119.18(19)
C(10)-C(9)-C(8)	121.39(19)
C(14)-C(9)-C(8)	119.26(18)
C(4)-C(7)-N(3)	123.4(2)
C(4)-C(7)-C(6)	114.22(19)
N(3)-C(7)-C(6)	122.2(2)
C(13)-C(14)-C(9)	120.3(2)
C(13)-C(14)-H(14)	119.9
C(9)-C(14)-H(14)	119.9

C(20)-C(18)-C(21)	120.1(2)
C(20)-C(18)-H(18)	120.0
C(21)-C(18)-H(18)	120.0
C(20)-C(19)-C(16)	120.2(2)
C(20)-C(19)-H(19)	119.9
C(16)-C(19)-H(19)	119.9
C(4)-C(1)-C(3)	123.6(2)
C(4)-C(1)-H(1)	118.2
C(3)-C(1)-H(1)	118.2
C(12)-C(10)-C(9)	120.3(2)
C(12)-C(10)-H(10)	119.8
C(9)-C(10)-H(10)	119.8
C(12)-C(11)-C(13)	119.9(2)
C(12)-C(11)-H(11)	120.1
C(13)-C(11)-H(11)	120.1
C(11)-C(12)-C(10)	120.2(2)
C(11)-C(12)-H(12)	119.9
C(10)-C(12)-H(12)	119.9
C(18)-C(20)-C(19)	120.2(2)
C(18)-C(20)-H(20)	119.9
C(19)-C(20)-H(20)	119.9
C(7)-N(3)-C(2)	118.0(2)
C(18)-C(21)-C(17)	120.1(2)
C(18)-C(21)-H(21)	119.9
C(17)-C(21)-H(21)	120.0
C(3)-C(2)-N(3)	119.5(2)
C(3)-C(2)-H(2)	120.3
N(3)-C(2)-H(2)	120.3
C(2)-C(3)-C(1)	118.3(2)
C(2)-C(3)-H(3)	120.8
C(1)-C(3)-H(3)	120.8

Symmetry transformations used to generate equivalent atoms:

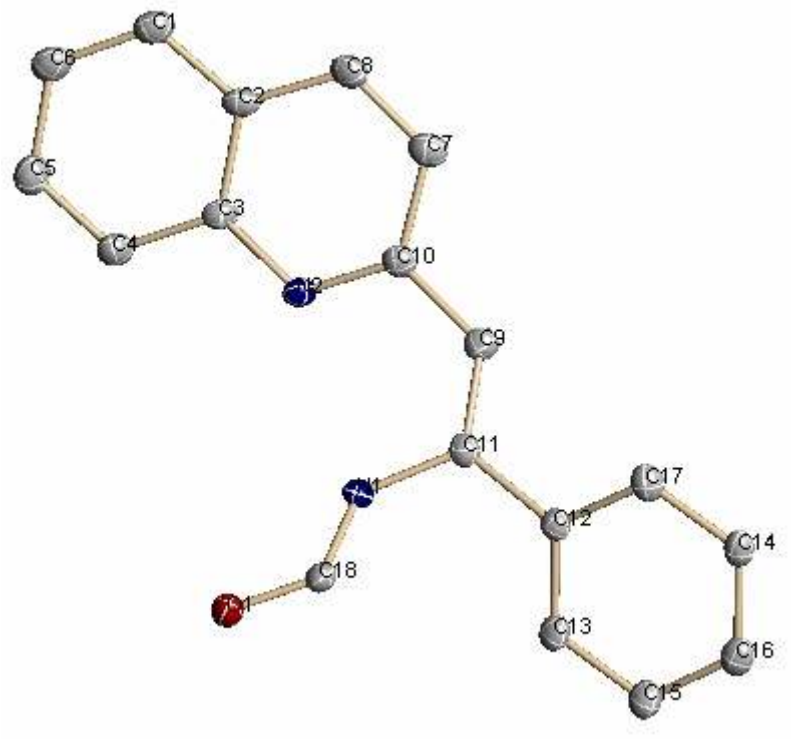
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 5. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
N(1)	16(1)	21(1)	14(1)	1(1)	1(1)	2(1)
C(15)	19(1)	20(1)	13(1)	2(1)	0(1)	2(1)
C(4)	9(1)	12(1)	11(1)	-1(1)	2(1)	0(1)
C(5)	15(1)	22(1)	16(1)	1(1)	-2(1)	1(1)
N(2)	19(1)	23(1)	15(1)	-1(1)	-1(1)	1(1)
C(6)	18(1)	18(1)	14(1)	1(1)	1(1)	0(1)
C(8)	14(1)	19(1)	13(1)	3(1)	1(1)	0(1)
C(13)	13(1)	19(1)	26(1)	2(1)	0(1)	-2(1)
C(16)	24(1)	22(1)	12(1)	3(1)	3(1)	5(1)
C(17)	27(1)	27(1)	17(1)	-1(1)	2(1)	5(1)
C(9)	13(1)	17(1)	16(1)	0(1)	0(1)	2(1)
C(7)	13(1)	23(1)	13(1)	0(1)	-1(1)	2(1)
C(14)	15(1)	19(1)	18(1)	2(1)	2(1)	2(1)
C(18)	39(1)	31(1)	19(1)	1(1)	10(1)	12(1)
C(19)	25(1)	29(1)	20(1)	-2(1)	4(1)	1(1)
C(1)	16(1)	24(1)	23(1)	-1(1)	4(1)	-1(1)
C(10)	16(1)	26(1)	18(1)	-1(1)	2(1)	-4(1)
C(11)	17(1)	22(1)	21(1)	-6(1)	-6(1)	2(1)
C(12)	19(1)	30(1)	16(1)	-1(1)	1(1)	0(1)
C(20)	26(1)	39(1)	28(1)	0(1)	8(1)	6(1)
N(3)	32(1)	32(1)	33(1)	-1(1)	1(1)	-1(1)
C(21)	39(1)	26(1)	18(1)	-1(1)	3(1)	7(1)
C(2)	29(1)	19(1)	28(1)	-4(1)	4(1)	-1(1)
C(3)	21(1)	26(1)	23(1)	-4(1)	4(1)	6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for 5.

	x	y	z	U(eq)
H(4)	-169	123	6452	13
H(5)	193	397	5465	21
H(13)	4482	3523	6767	24
H(17)	1722	-2166	4386	28
H(14)	3646	2589	6030	20
H(18)	4157	-3352	3802	35
H(19)	3967	-410	5064	29
H(1)	-1236	1303	6964	25
H(10)	1818	959	7102	24
H(11)	4034	3038	7671	24
H(12)	2710	1737	7837	26
H(20)	4833	-1746	4434	37
H(21)	2602	-3549	3770	33
H(2)	-2	5292	6801	30
H(3)	-1194	3865	7144	28

X-ray data for pyrimidine **13a**



ORTEP Drawing of **13a**

Table 1. Crystal data and structure refinement for 13a.

Identification code	13a	
Empirical formula	C18 H14 N2 O	
Formula weight	274.31	
Temperature	299 K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 12.380(4) Å	α= 90°.
	b = 7.301(3) Å	β= 98.533(7)°.
	c = 15.108(5) Å	γ = 90°.
Volume	1350.4(8) $Å^3$	
Z	4	
Density (calculated)	1.349 Mg/m^3	
Absorption coefficient	0.085 mm^{-1}	
F(000)	576	
Crystal size	0.39 x 0.38 x 0.37 mm^3	
Theta range for data collection	1.99 to 27.84°.	
Index ranges	-16<=h<=11, -9<=k<=9, -13<=l<=19	
Reflections collected	7657	
Independent reflections	3037 [R(int) = 0.0545]	
Completeness to theta = 27.84°	94.7 %	
Absorption correction	Empirical	
Max. and min. transmission	0.9692 and 0.9676	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	3037 / 0 / 190	
Goodness-of-fit on F^2	0.740	
Final R indices [I>2sigma(I)]	R1 = 0.0633, wR2 = 0.1810	
R indices (all data)	R1 = 0.1049, wR2 = 0.2239	
Largest diff. peak and hole	0.405 and -0.247 $e.Å^{-3}$	

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 13a. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
O(1)	571(2)	1210(3)	7229(1)	23(1)
N(1)	2419(2)	1005(3)	7392(1)	18(1)
N(2)	3391(2)	984(3)	5897(1)	18(1)
C(12)	3627(2)	1437(4)	8835(2)	17(1)
C(1)	3973(2)	2214(4)	3625(2)	24(1)
C(2)	4138(2)	2034(4)	4570(2)	19(1)
C(9)	4316(2)	1579(4)	7381(2)	19(1)
C(10)	4270(2)	1649(4)	6411(2)	19(1)
C(3)	3316(2)	1162(4)	4984(2)	18(1)
C(13)	3017(2)	340(4)	9334(2)	20(1)
C(14)	4689(2)	2487(4)	10218(2)	20(1)
C(11)	3473(2)	1367(4)	7845(2)	18(1)
C(15)	3238(2)	323(4)	10267(2)	22(1)
C(4)	2378(2)	465(4)	4450(2)	21(1)
C(16)	4081(2)	1381(4)	10704(2)	21(1)
C(7)	5151(2)	2458(4)	6044(2)	22(1)
C(18)	1463(2)	1516(4)	7659(2)	18(1)
C(17)	4455(2)	2542(4)	9289(2)	20(1)
C(5)	2246(2)	653(4)	3537(2)	25(1)
C(8)	5078(2)	2654(4)	5137(2)	22(1)
C(6)	3045(2)	1538(4)	3126(2)	26(1)

Table 3. Bond lengths [Å] and angles [°] for 13a.

O(1)-C(18)	1.217(3)
N(1)-C(18)	1.357(3)
N(1)-C(11)	1.406(3)
N(1)-H(1)	0.8600
N(2)-C(10)	1.332(3)
N(2)-C(3)	1.375(3)
C(12)-C(13)	1.397(4)
C(12)-C(17)	1.402(4)
C(12)-C(11)	1.480(4)
C(1)-C(6)	1.369(4)
C(1)-C(2)	1.417(4)
C(1)-H(1A)	0.9300
C(2)-C(3)	1.422(4)
C(2)-C(8)	1.413(4)
C(9)-C(11)	1.350(4)
C(9)-C(10)	1.458(4)
C(9)-H(9)	0.9300
C(10)-C(7)	1.423(4)
C(3)-C(4)	1.407(4)
C(13)-C(15)	1.394(4)
C(13)-H(13)	0.9300
C(14)-C(16)	1.386(4)
C(14)-C(17)	1.392(4)
C(14)-H(14)	0.9300
C(15)-C(16)	1.385(4)
C(15)-H(15)	0.9300
C(4)-C(5)	1.371(4)
C(4)-H(4)	0.9300
C(16)-H(16)	0.9300
C(7)-C(8)	1.368(4)
C(7)-H(7)	0.9300
C(18)-H(18)	0.9300
C(17)-H(17)	0.9300
C(5)-C(6)	1.402(4)
C(5)-H(5)	0.9300
C(8)-H(8)	0.9300
C(6)-H(6)	0.9300

C(18)-N(1)-C(11)	126.2(2)
C(18)-N(1)-H(1)	116.9
C(11)-N(1)-H(1)	116.9
C(10)-N(2)-C(3)	118.6(2)
C(13)-C(12)-C(17)	118.7(2)
C(13)-C(12)-C(11)	121.8(2)
C(17)-C(12)-C(11)	119.3(2)
C(6)-C(1)-C(2)	120.3(3)
C(6)-C(1)-H(1A)	119.8
C(2)-C(1)-H(1A)	119.8
C(3)-C(2)-C(8)	117.1(2)
C(3)-C(2)-C(1)	118.6(3)
C(8)-C(2)-C(1)	124.3(3)
C(11)-C(9)-C(10)	127.4(3)
C(11)-C(9)-H(9)	116.3
C(10)-C(9)-H(9)	116.3
N(2)-C(10)-C(7)	122.1(2)
N(2)-C(10)-C(9)	118.7(2)
C(7)-C(10)-C(9)	119.2(2)
N(2)-C(3)-C(4)	117.9(2)
N(2)-C(3)-C(2)	122.5(2)
C(4)-C(3)-C(2)	119.6(2)
C(12)-C(13)-C(15)	120.7(3)
C(12)-C(13)-H(13)	119.7
C(15)-C(13)-H(13)	119.7
C(16)-C(14)-C(17)	120.2(3)
C(16)-C(14)-H(14)	119.9
C(17)-C(14)-H(14)	119.9
C(9)-C(11)-N(1)	120.2(2)
C(9)-C(11)-C(12)	121.8(2)
N(1)-C(11)-C(12)	118.0(2)
C(16)-C(15)-C(13)	119.9(3)
C(16)-C(15)-H(15)	120.0
C(13)-C(15)-H(15)	120.0
C(5)-C(4)-C(3)	120.4(3)
C(5)-C(4)-H(4)	119.8
C(3)-C(4)-H(4)	119.8
C(14)-C(16)-C(15)	120.1(3)
C(14)-C(16)-H(16)	119.9
C(15)-C(16)-H(16)	120.0

C(8)-C(7)-C(10)	119.6(3)
C(8)-C(7)-H(7)	120.2
C(10)-C(7)-H(7)	120.2
O(1)-C(18)-N(1)	123.6(3)
O(1)-C(18)-H(18)	118.2
N(1)-C(18)-H(18)	118.2
C(14)-C(17)-C(12)	120.3(3)
C(14)-C(17)-H(17)	119.9
C(12)-C(17)-H(17)	119.9
C(4)-C(5)-C(6)	120.4(3)
C(4)-C(5)-H(5)	119.8
C(6)-C(5)-H(5)	119.8
C(7)-C(8)-C(2)	120.0(3)
C(7)-C(8)-H(8)	120.0
C(2)-C(8)-H(8)	120.0
C(1)-C(6)-C(5)	120.8(3)
C(1)-C(6)-H(6)	119.6
C(5)-C(6)-H(6)	119.6

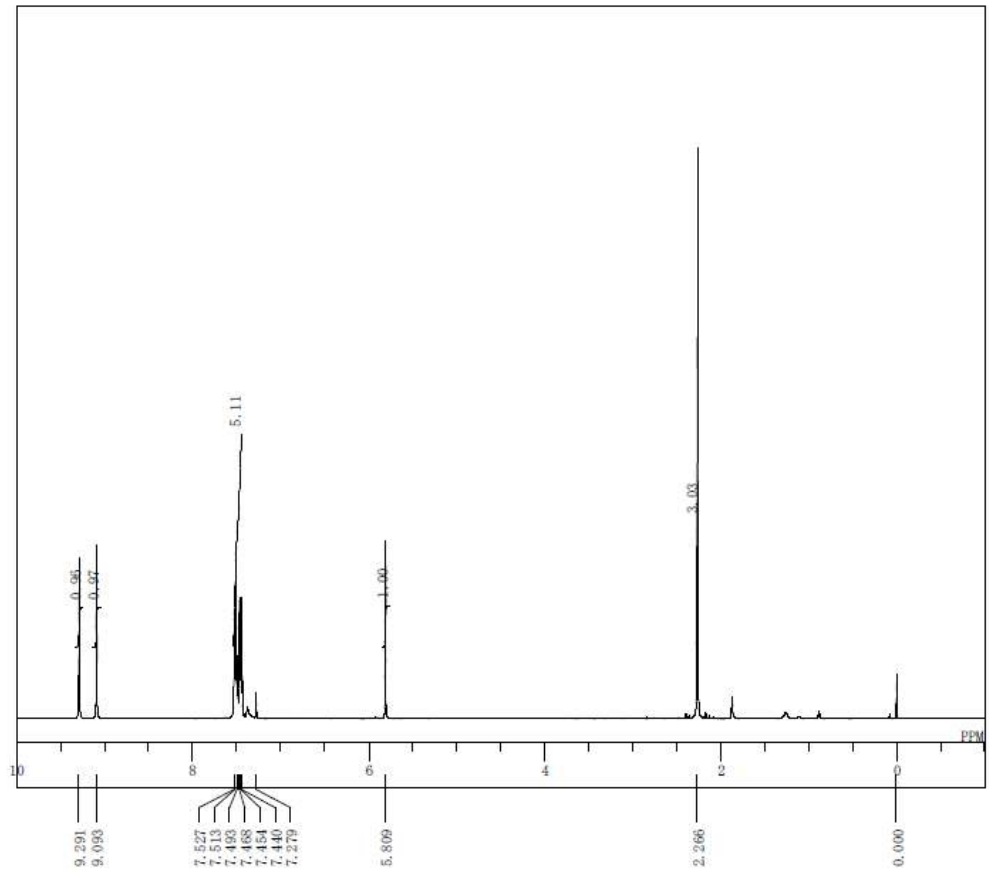
Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 13a. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ...\ + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
O(1)	18(1)	32(1)	19(1)	0(1)	2(1)	1(1)
N(1)	18(1)	22(1)	15(1)	-3(1)	3(1)	-2(1)
N(2)	19(1)	20(1)	18(1)	-1(1)	6(1)	0(1)
C(12)	16(1)	18(1)	17(1)	1(1)	3(1)	3(1)
C(1)	25(2)	27(2)	23(1)	3(1)	12(1)	1(1)
C(2)	19(1)	19(1)	22(1)	2(1)	8(1)	3(1)
C(9)	17(1)	21(1)	20(1)	0(1)	2(1)	2(1)
C(10)	17(1)	21(1)	19(1)	-2(1)	5(1)	4(1)
C(3)	20(1)	16(1)	18(1)	-2(1)	7(1)	2(1)
C(13)	16(1)	21(1)	20(1)	0(1)	-2(1)	2(1)
C(14)	17(1)	23(2)	20(1)	-5(1)	0(1)	2(1)
C(11)	18(1)	16(1)	19(1)	0(1)	1(1)	2(1)
C(15)	23(2)	22(1)	20(1)	3(1)	3(1)	2(1)
C(4)	19(1)	26(2)	20(1)	-1(1)	8(1)	-2(1)
C(16)	23(2)	24(2)	17(1)	-1(1)	2(1)	8(1)
C(7)	17(1)	22(1)	26(2)	-2(1)	4(1)	1(1)
C(18)	20(1)	21(1)	14(1)	0(1)	4(1)	1(1)
C(17)	19(1)	20(1)	20(1)	-1(1)	3(1)	2(1)
C(5)	22(2)	30(2)	21(1)	-5(1)	2(1)	0(1)
C(8)	17(1)	23(2)	28(2)	1(1)	10(1)	0(1)
C(6)	28(2)	33(2)	18(1)	1(1)	9(1)	6(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^{-3}) for 13a.

	x	y	z	U(eq)
H(1)	2375	401	6899	22
H(1A)	4498	2793	3343	29
H(9)	5005	1694	7718	23
H(13)	2457	-386	9043	24
H(14)	5255	3195	10514	24
H(15)	2820	-397	10594	26
H(4)	1844	-127	4717	26
H(16)	4240	1349	11325	26
H(7)	5773	2850	6419	26
H(18)	1493	2135	8200	22
H(17)	4851	3316	8967	24
H(5)	1623	191	3189	29
H(8)	5647	3196	4893	26
H(6)	2944	1668	2507	31

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OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            22.4 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.23 Hz
RGAIN             14
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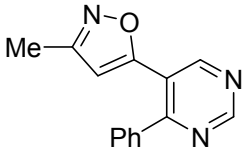
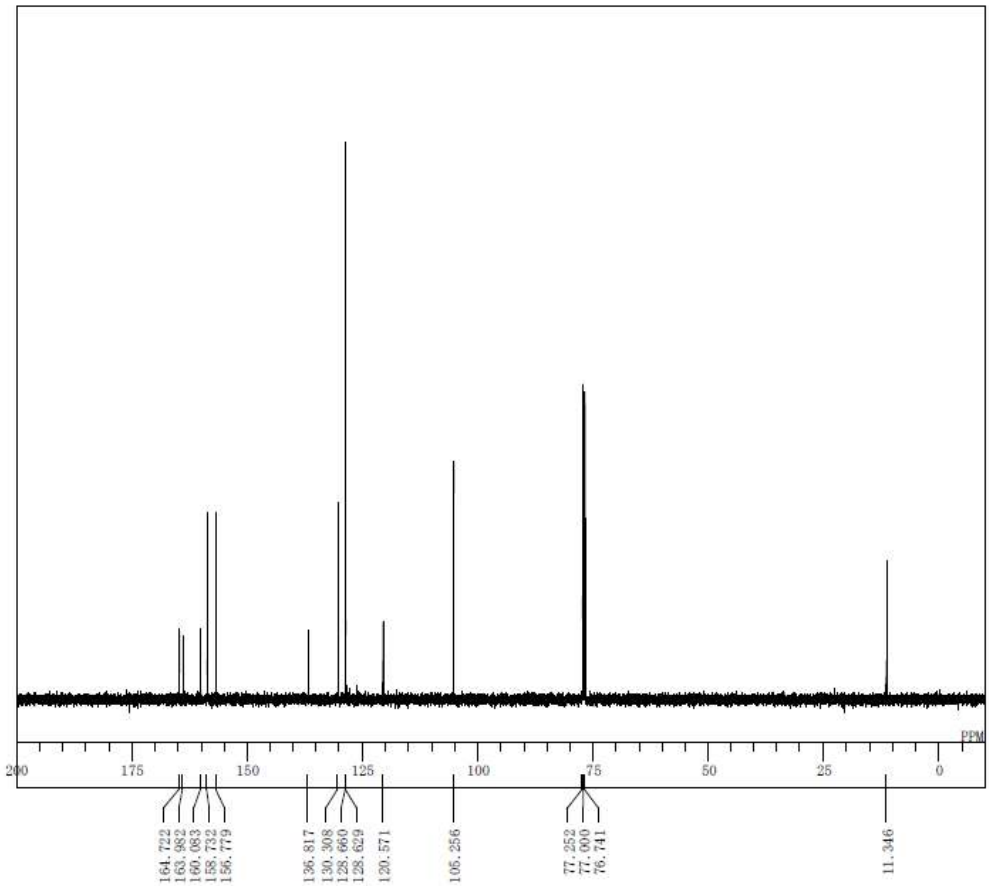


4a

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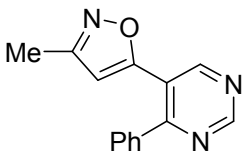
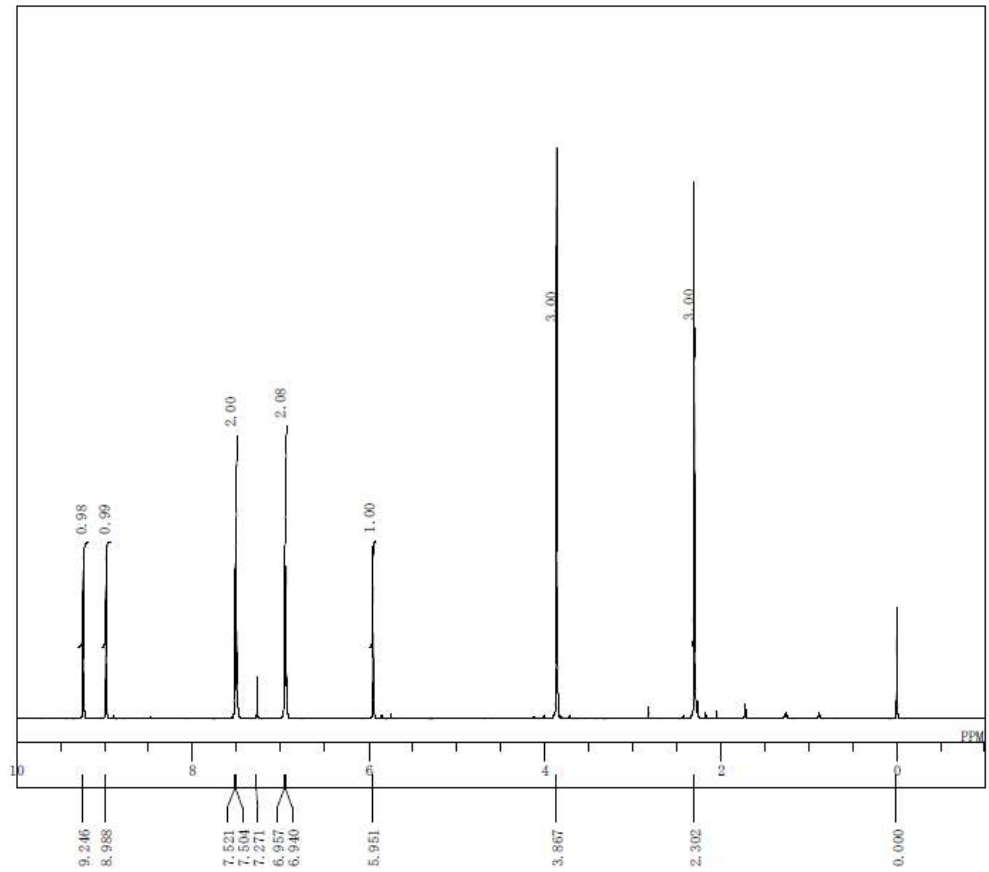
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU        31446.54 Hz
SCANS            305
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            25.8 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.48 Hz
RGAIN             30
```



4a

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EXMOD   single_pulse.exp
OBFRQ         500.00 MHz
OBSET         162.00 KHz
OBFIN         416.02 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM         2.1823 sec
PD            4.0000 sec
PW1             7.50 usec
IRNUC   1H
CTEMP          22.8 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             1.00 Hz
RGAIN            17
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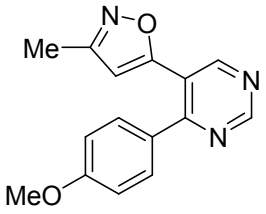
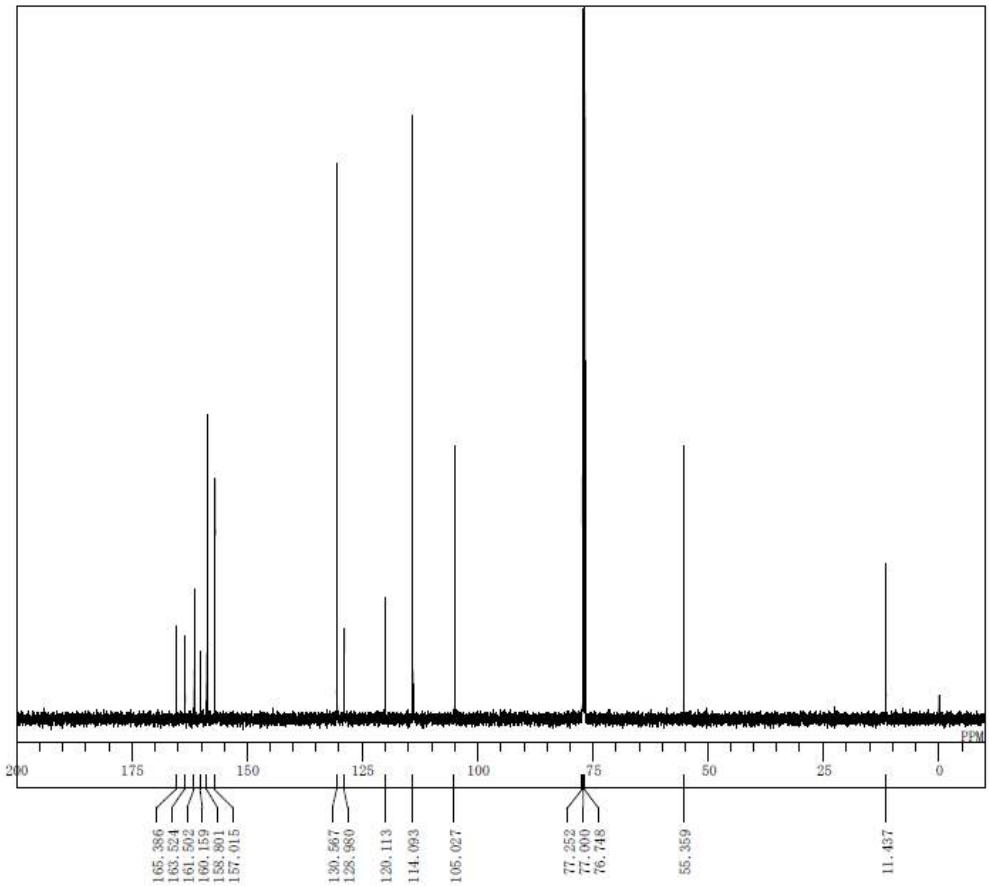


4b

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EXMOD   single_pulse_dec
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OBSET         777.00 KHz
OBFIN         875.47 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS             1298
ACQTM         1.0420 sec
PD            1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP          26.0 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.70 Hz
RGAIN            30
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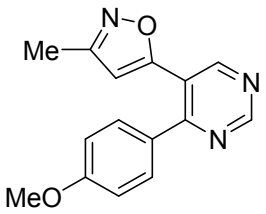
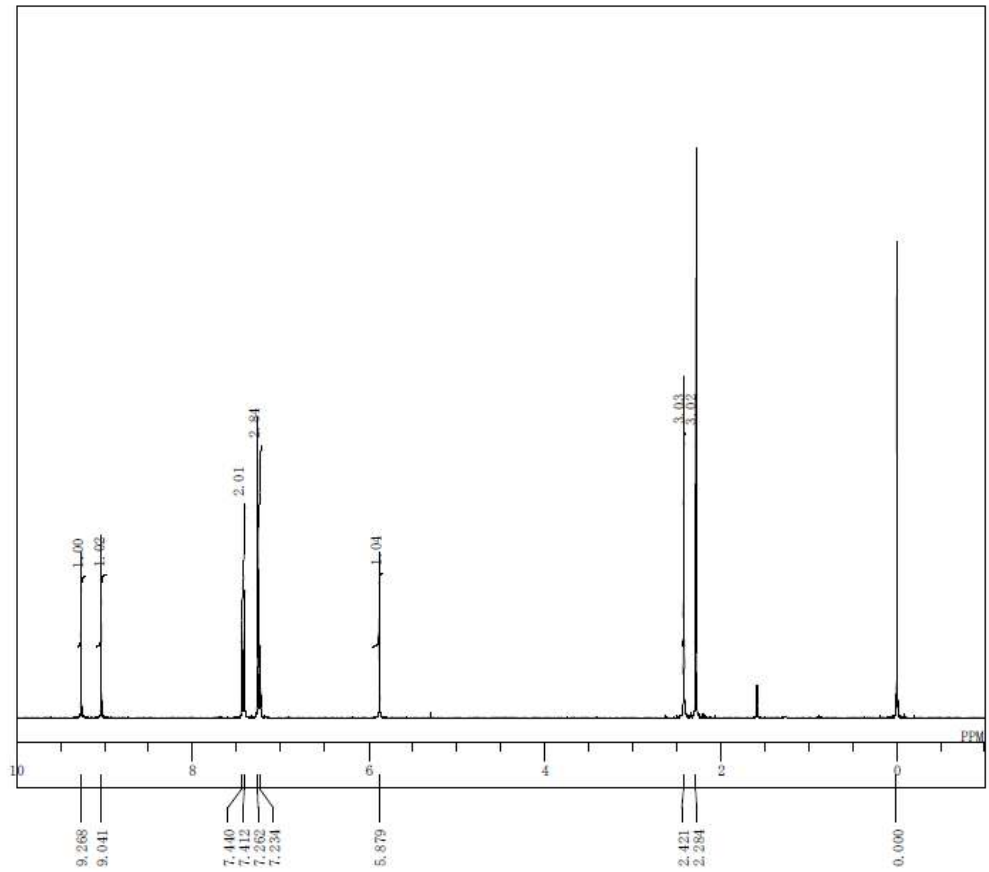


4b

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OBSET           1.15 KHz
OBFIN           8.57 Hz
POINT          16384
FREQU        4508.57 Hz
SCANS             8
ACQTM           3.6340 sec
PD              4.0000 sec
PW1             4.90 usec
IRNUC
CTEMP            25.1 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.14 Hz
RGAIN              22
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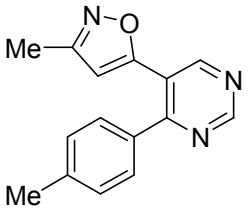
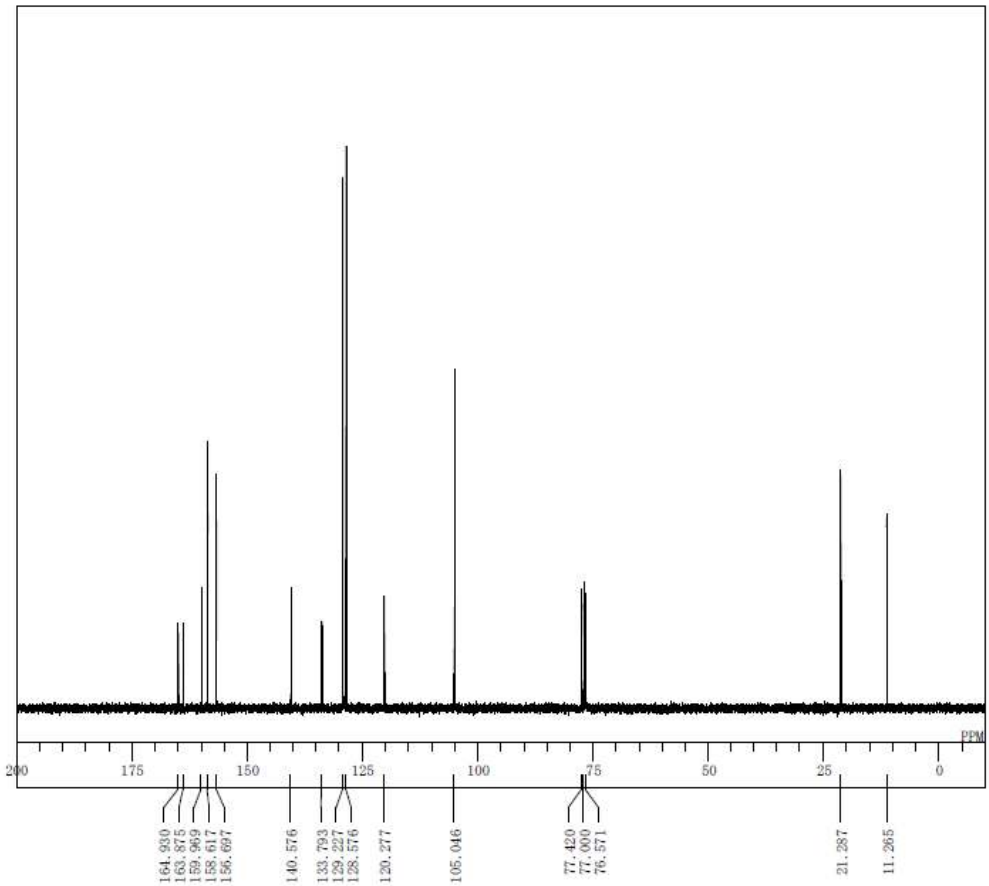
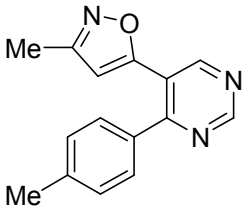


4c




```
COMNT
DATIM   Fri Sep 14 18:16:44 2007
OBNUC   13C
EXMOD   BCM
OBFRQ           75.45 MHz
OBSET          124.00 KHz
OBFIN         1840.00 Hz
POINT           32768
FREQU        20408.10 Hz
SCANS             143
ACQTM           1.6056 sec
PD              1.3940 sec
PW1             4.00 usec
IRNUC   1H
CTEMP            24.6 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF               0.31 Hz
RGAIN              27
```



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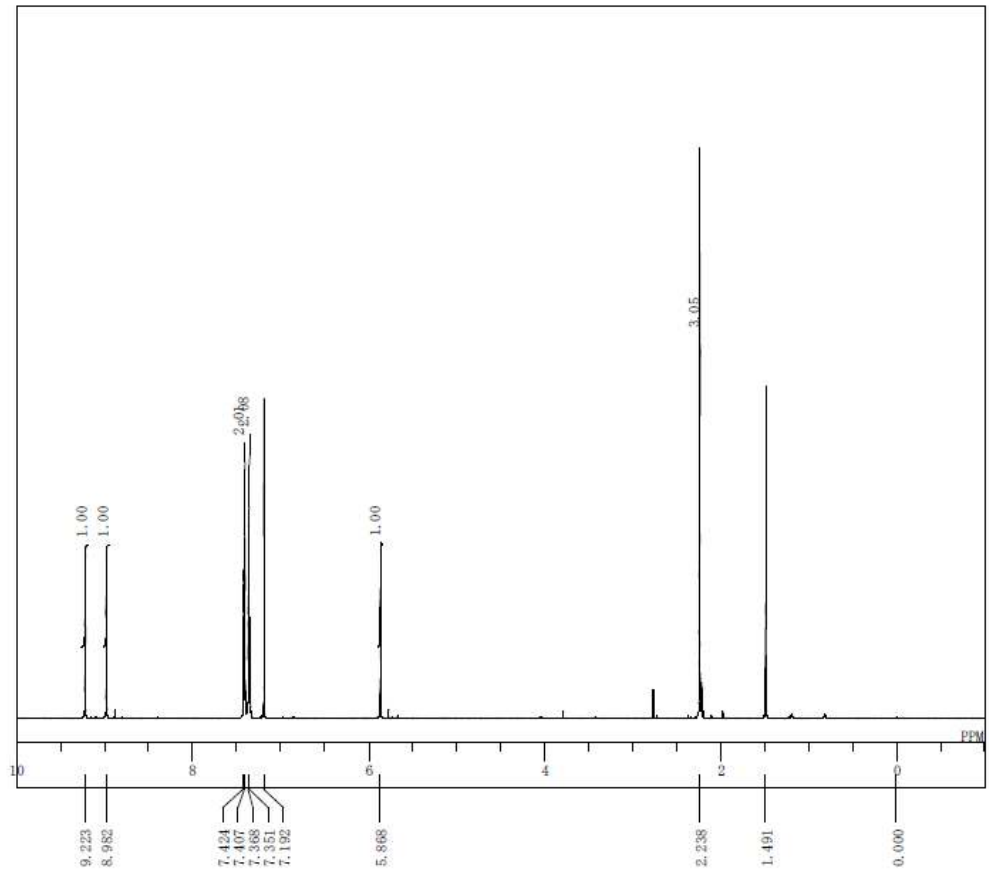
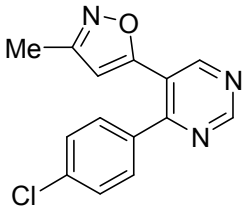
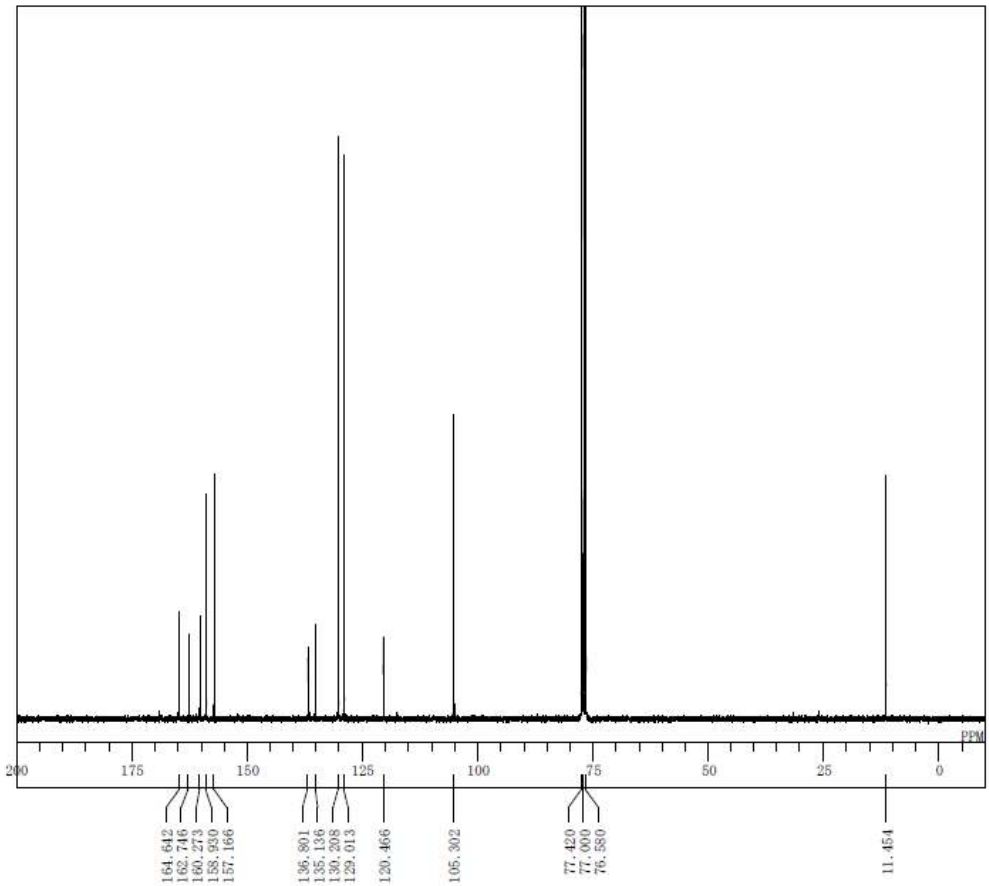


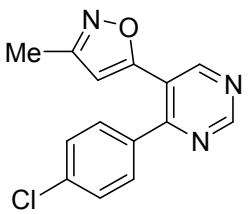

```
COMNT   Single Pulse Experiment
DATIM   17-JAN-2007 15:31:14
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.00 MHz
OBSET         162.00 KHz
OBFIN         416.02 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM         2.1823 sec
PD            4.0000 sec
PW1             7.50 usec
IRNUC   1H
CTEMP          24.2 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.76 Hz
RGAIN            24
```



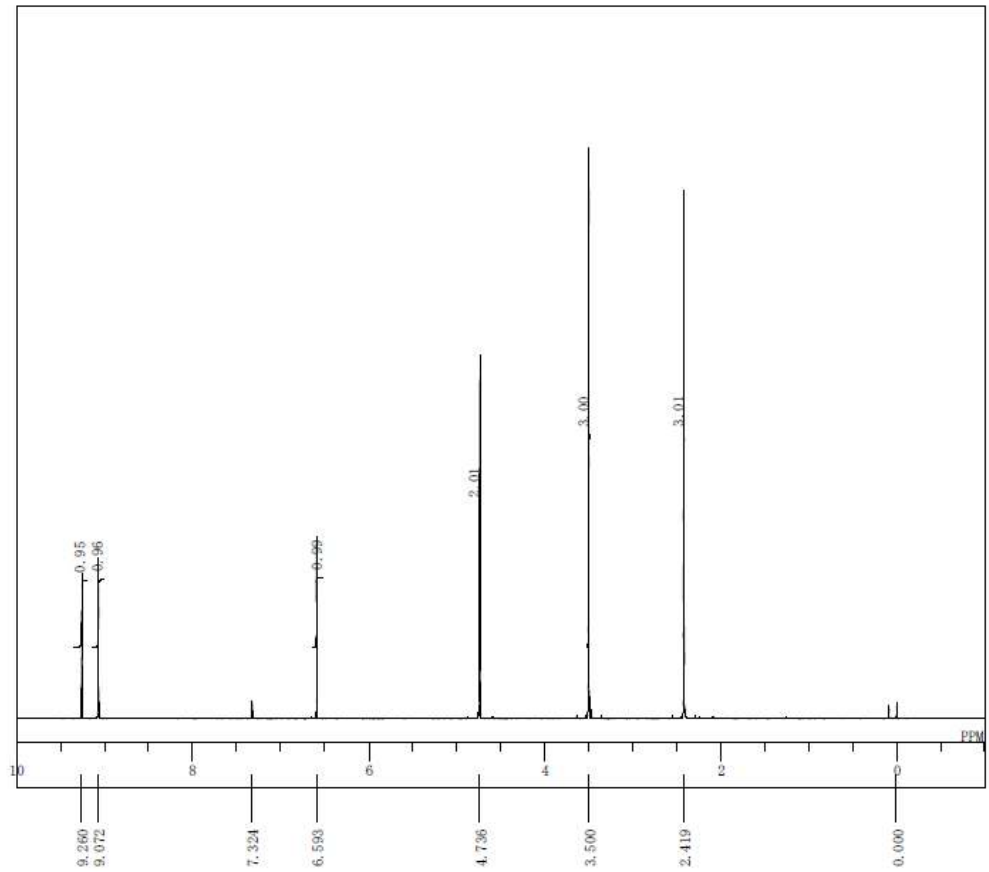
4d



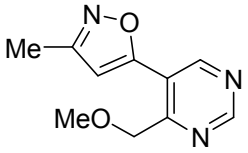

```
COMNT
DATIM   Wed Jan 17 09:29:59 2007
OBNUC   13C
EXMOD   BCM
OBFRQ          75.45 MHz
OBSET         124.00 KHz
OBFIN        1840.00 Hz
POINT          32768
FREQU       20408.10 Hz
SCANS          16901
ACQTM          1.6056 sec
PD             1.3940 sec
PW1              4.00 usec
IRNUC   1H
CTEMP          19.3 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.23 Hz
RGAIN            26
```



4d




```
COMNT   Single Pulse Experiment
DATIM   19-01-2008 14:15:13
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            25.6 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              13
```



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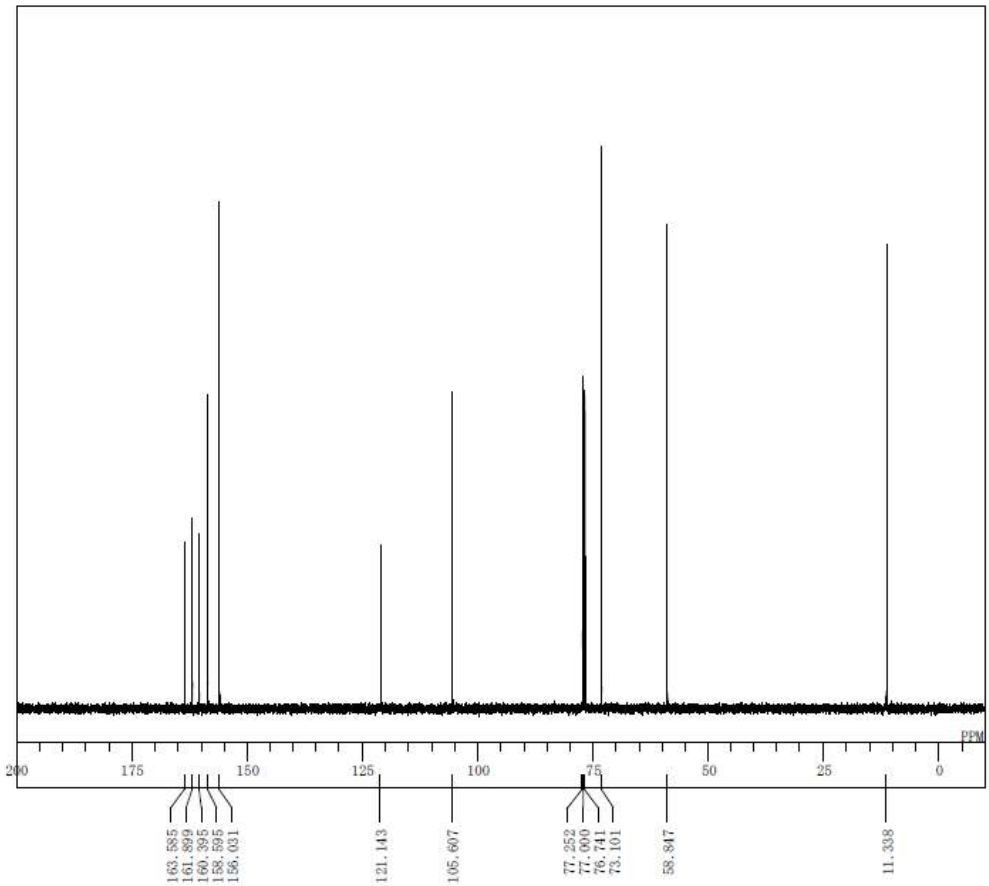
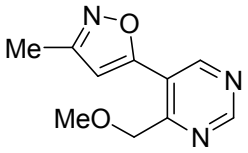
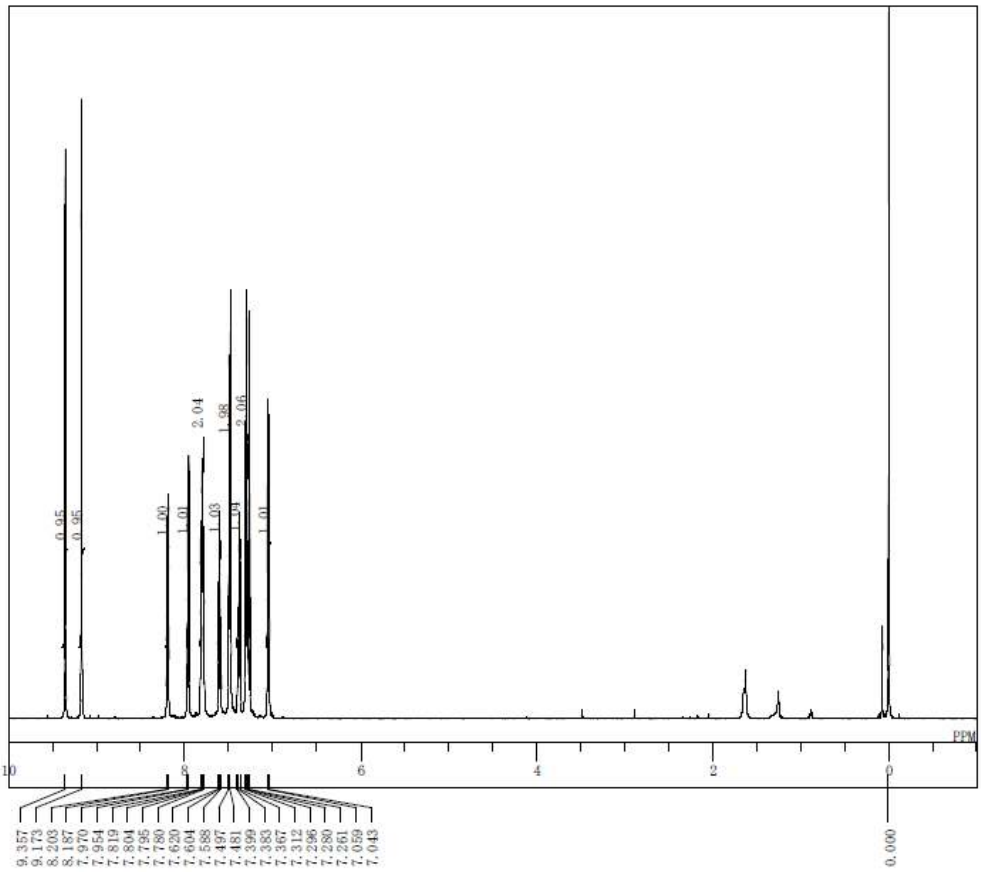


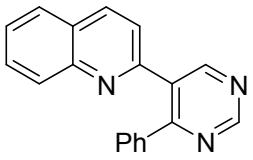

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   19-01-2008 14:31:55
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU        31446.54 Hz
SCANS            393
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            27.0 c
SLVNT   CDCL3
EXREF            77.00 ppm
BF               0.48 Hz
RGAIN              30
```



4e




```
COMNT   Single Pulse Experiment
DATIM   20-JAN-2007 16:31:17
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.00 MHz
OBSET         162.00 KHz
OBFIN         416.02 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM         2.1823 sec
PD            4.0000 sec
PW1             7.50 usec
IRNUC   1H
CTEMP          24.7 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             1.00 Hz
RGAIN            19
```



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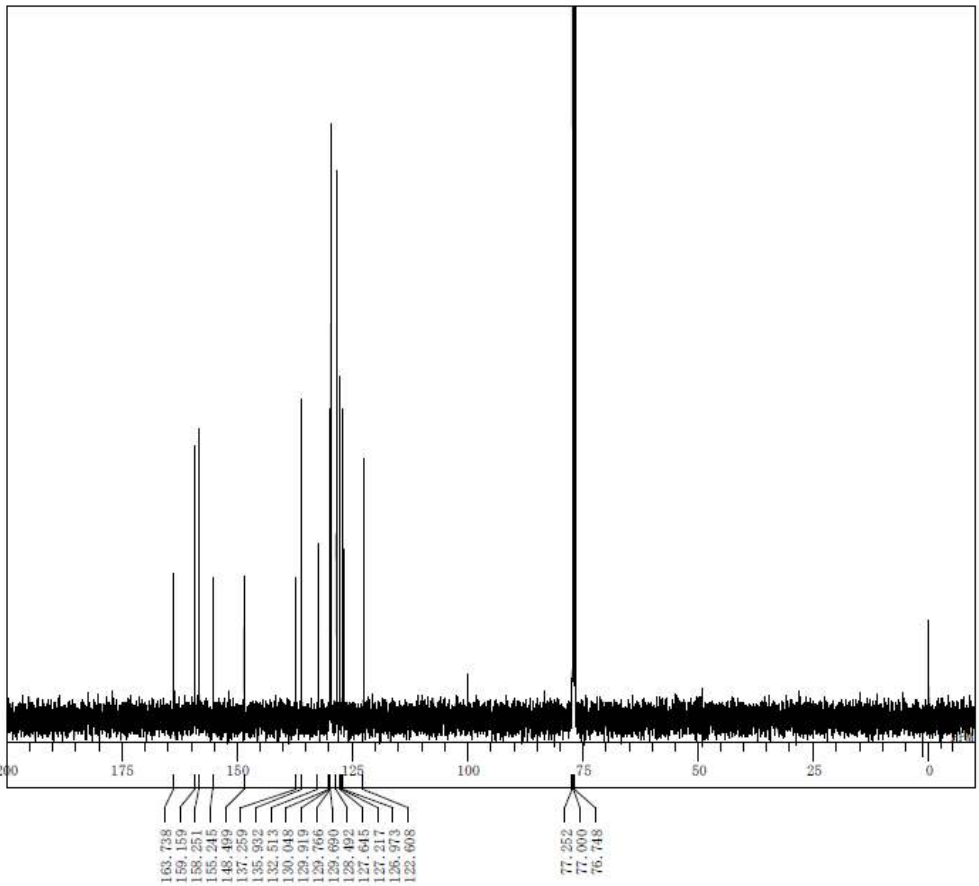
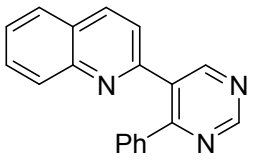
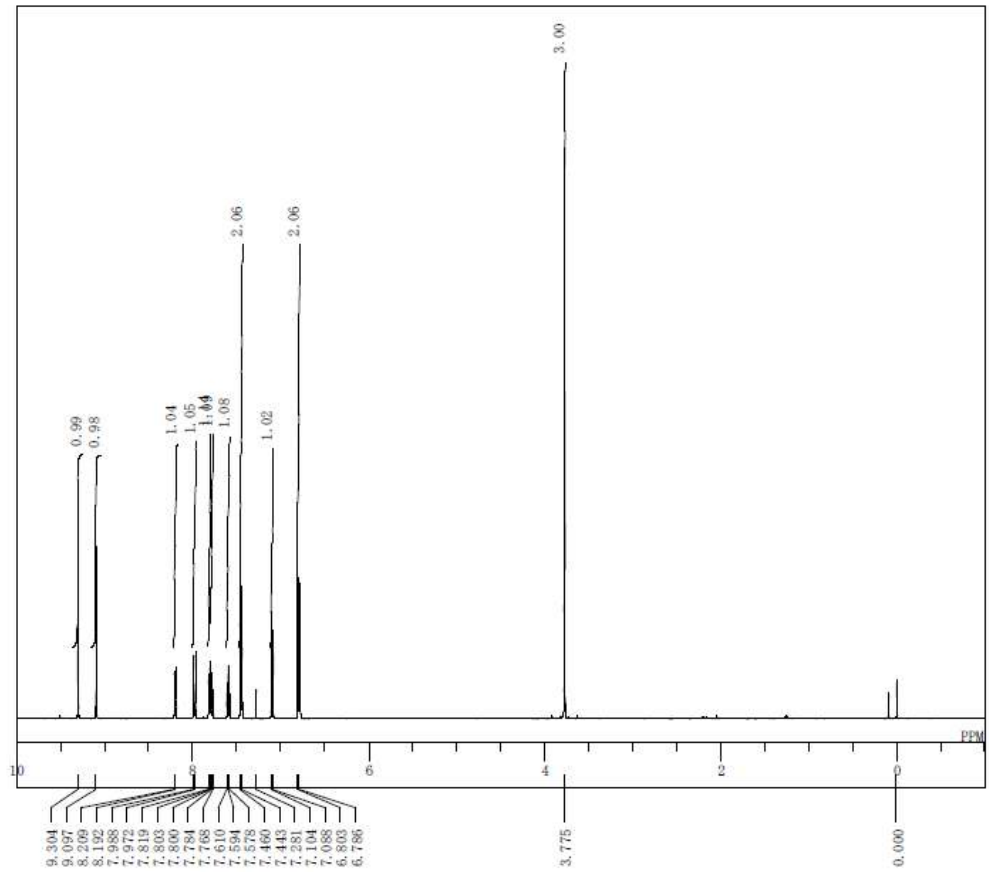


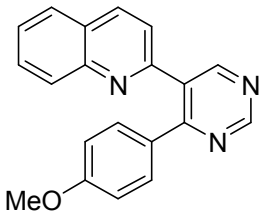

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   20-JAN-2007 17:15:55
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.00 MHz
OBSET         777.00 KHz
OBFIN         875.47 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS          1277
ACQTM         1.0420 sec
PD            1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP          25.8 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF             1.00 Hz
RGAIN            30
```



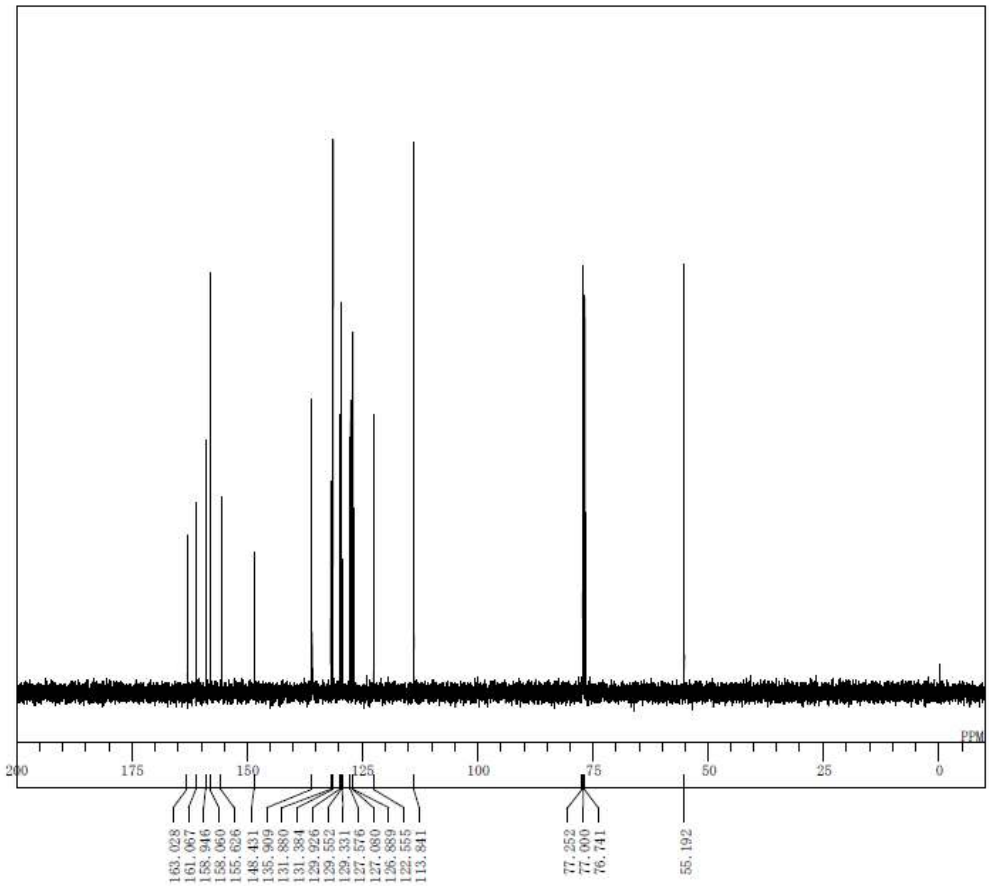
4f



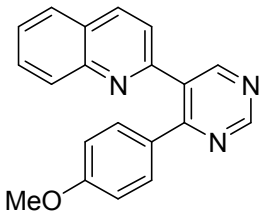

```
COMNT   Single Pulse Experiment
DATIM   05-06-2007 15:36:07
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS              8
ACQTM          2.1823 sec
PD             4.0000 sec
PW1              7.50 usec
IRNUC
CTEMP           21.9 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.23 Hz
RGAIN             13
```



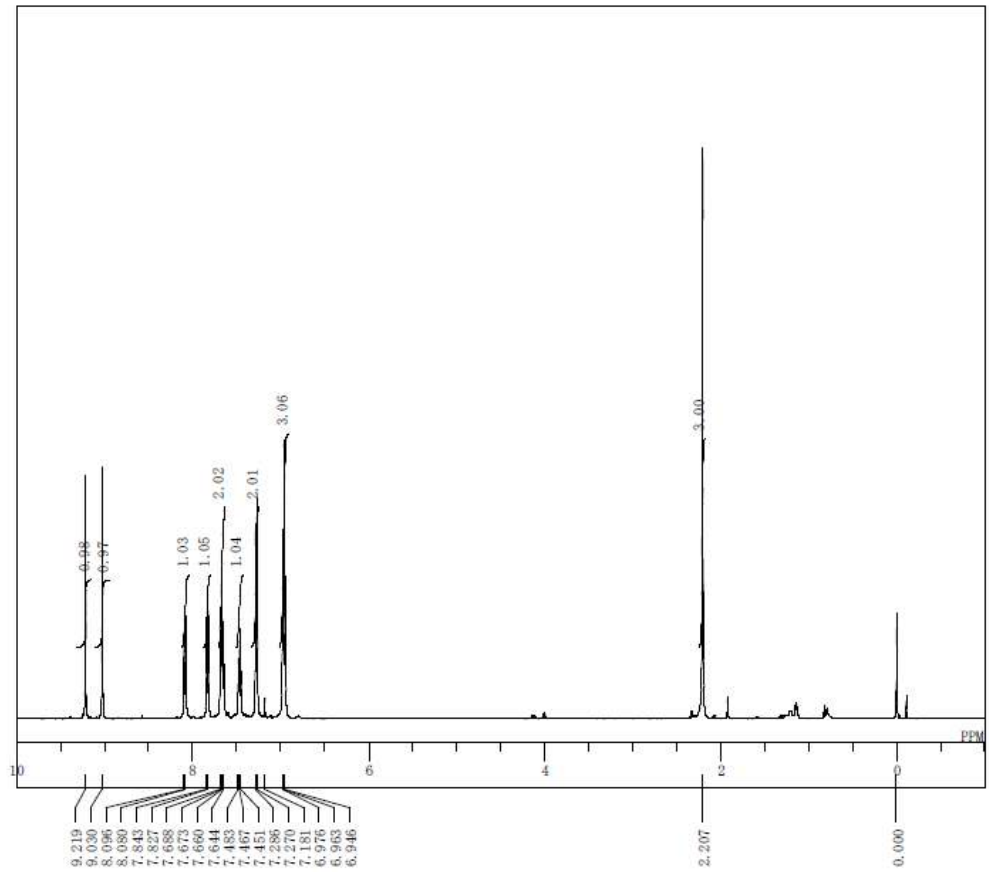
4g



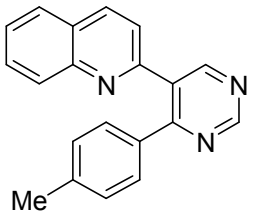

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   05-06-2007 15:41:01
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS            103
ACQTM          1.0420 sec
PD             1.0000 sec
PW1              4.17 usec
IRNUC   1H
CTEMP           23.8 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              0.48 Hz
RGAIN             30
```



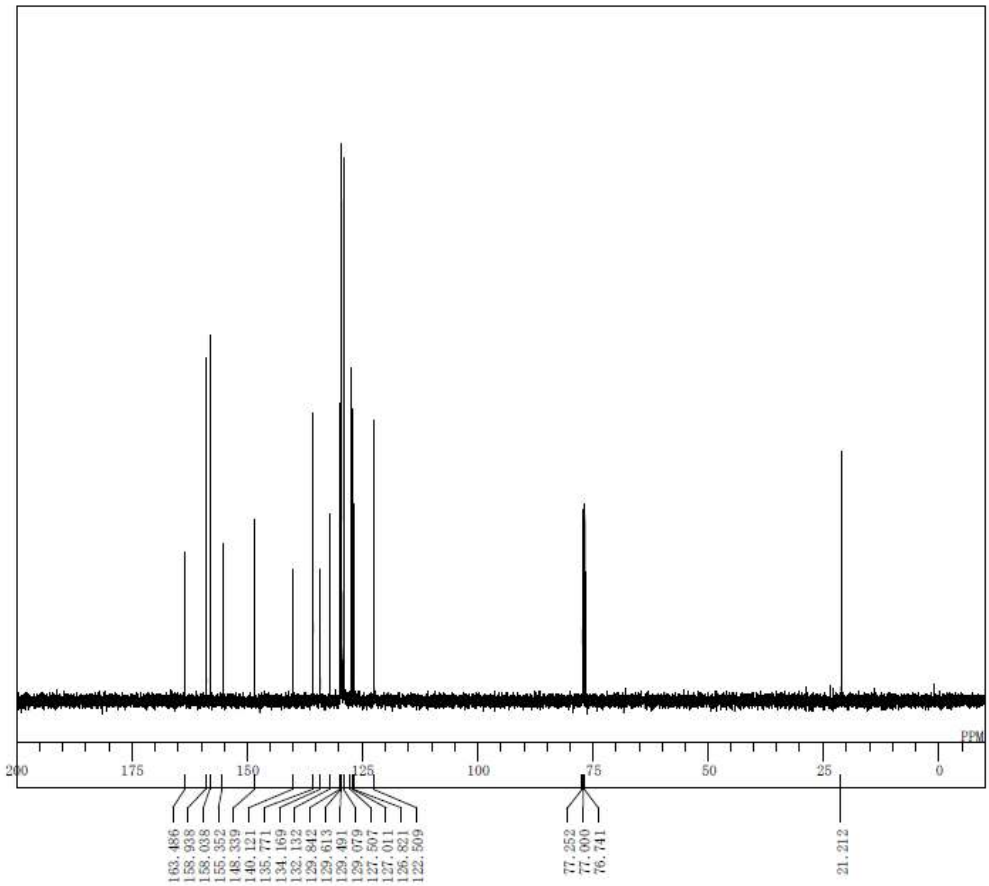
4g



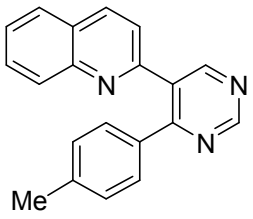

```
COMNT   Single Pulse Experiment
DATIM   28-05-2007 17:40:25
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.50 usec
IRNUC
CTEMP            22.5 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              11
```



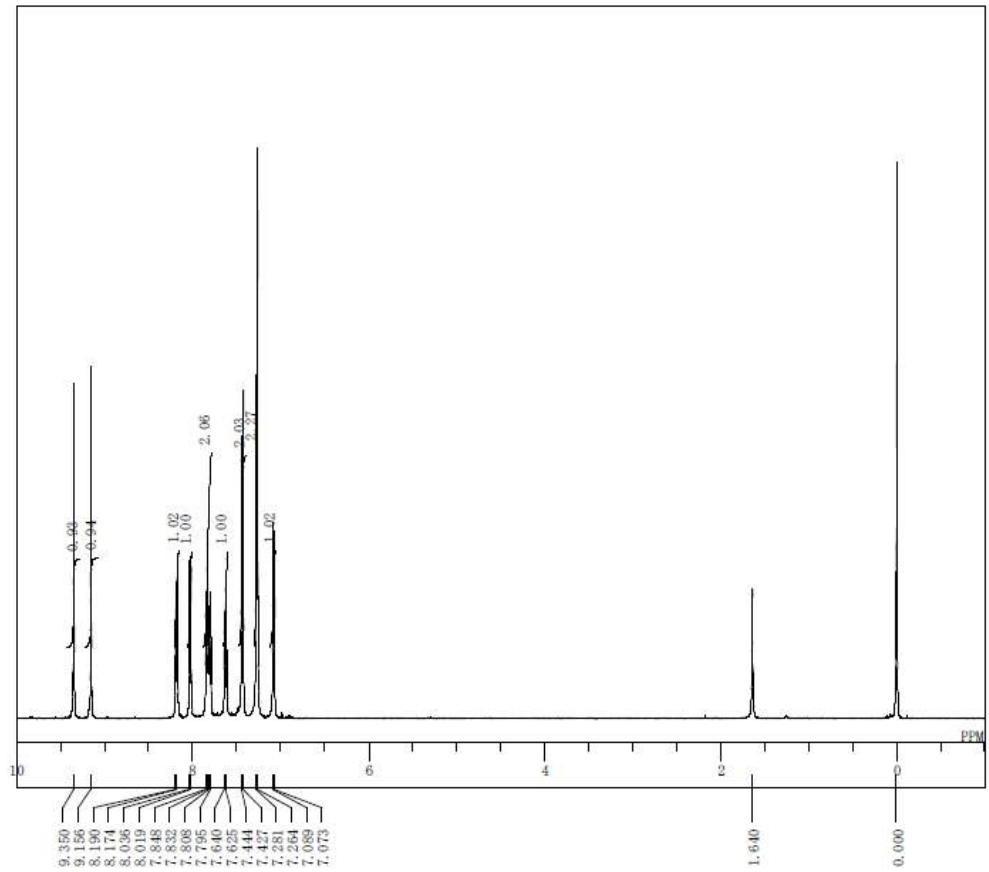
4h



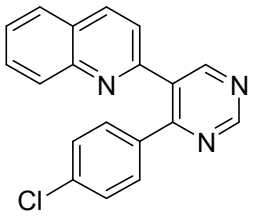

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   28-05-2007 17:44:43
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU        31446.54 Hz
SCANS            101
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            24.8 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF               0.48 Hz
RGAIN              30
```



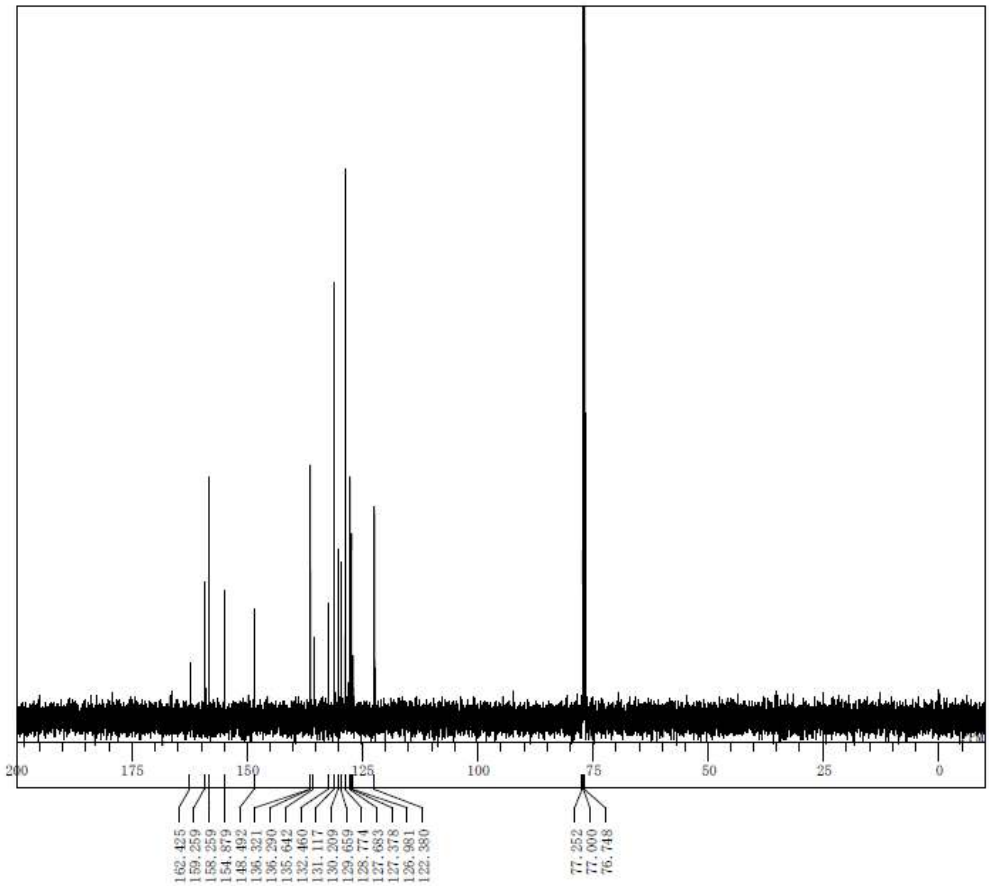
4h



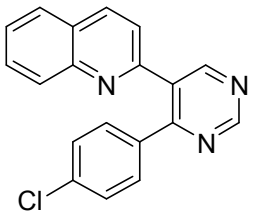

```
COMNT   Single Pulse Experiment
DATIM   05-09-2008 14:20:37
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            20.6 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              19
```



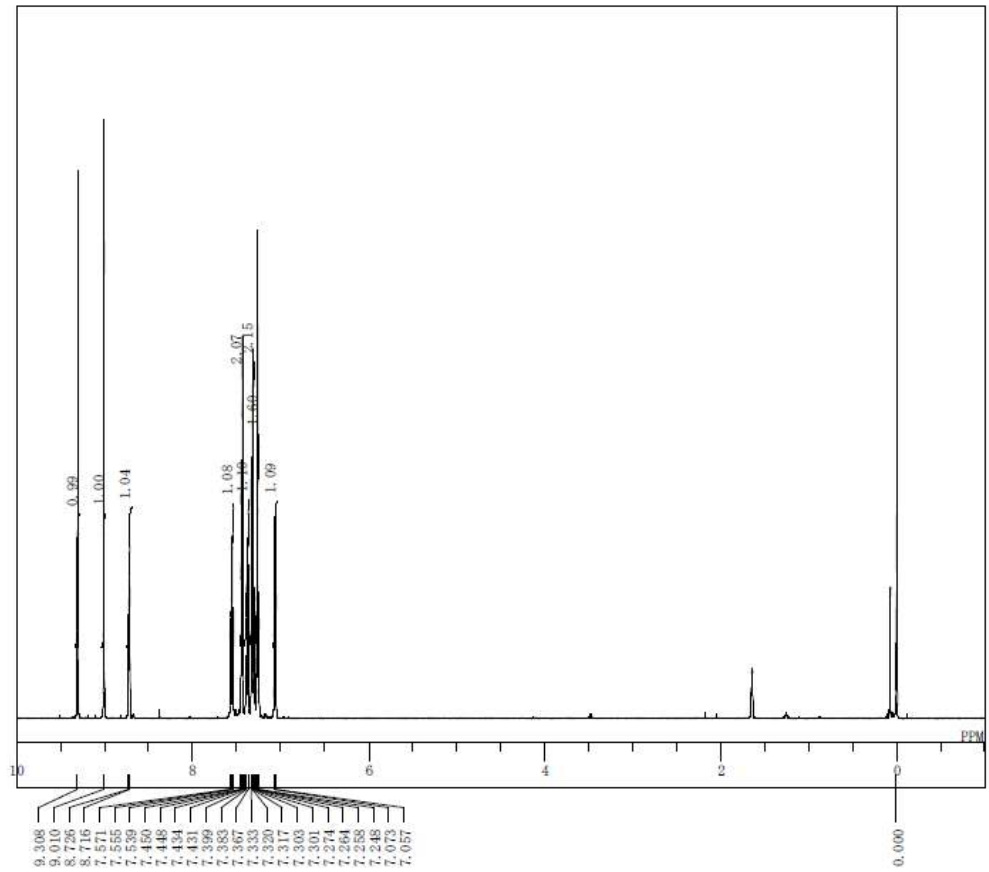
4i



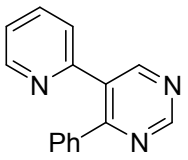

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   10-FEB-2007 11:57:24
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.00 MHz
OBSET         777.00 KHz
OBFIN         875.47 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS             1000
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            25.4 c
SLVNT   CDCL3
EXREF            77.00 ppm
BF                0.00 Hz
RGAIN               30
```



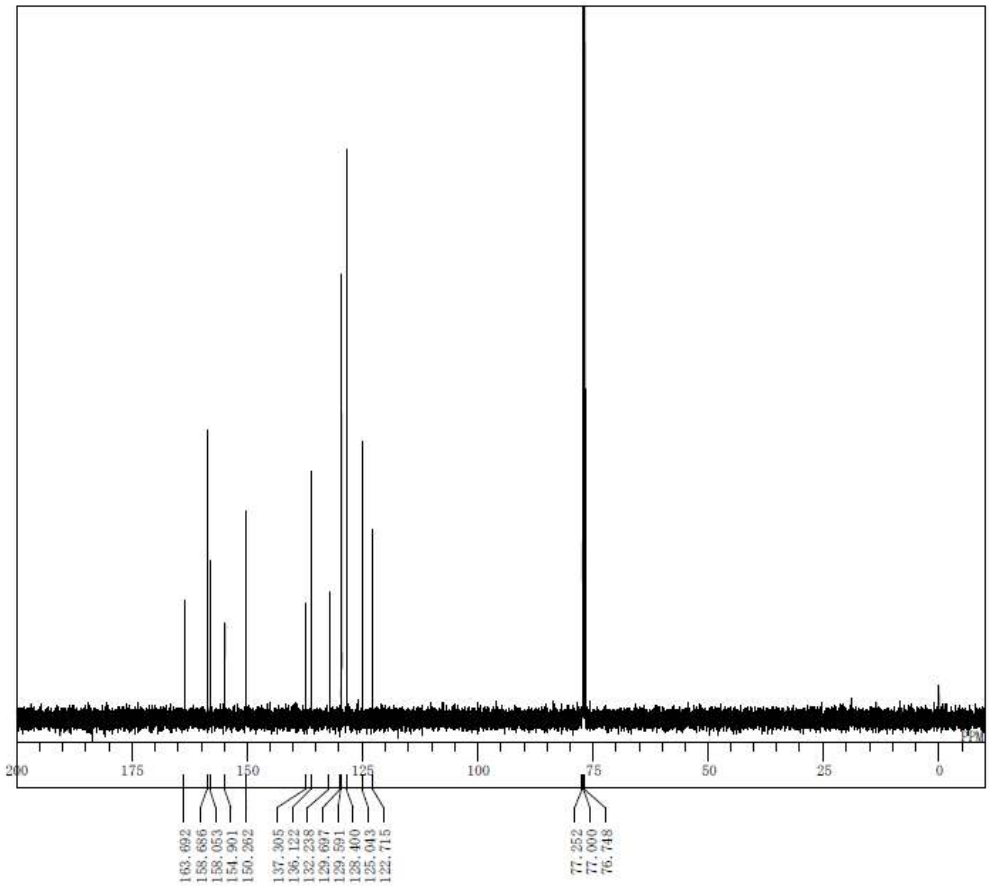
4i



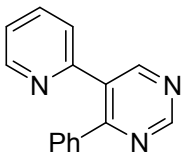

```
COMNT   Single Pulse Experiment
DATIM   23-JAN-2007 13:45:34
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.00 MHz
OBSET         162.00 KHz
OBFIN         416.02 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM         2.1823 sec
PD            4.0000 sec
PW1             7.50 usec
IRNUC   1H
CTEMP          24.5 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.18 Hz
RGAIN            21
```



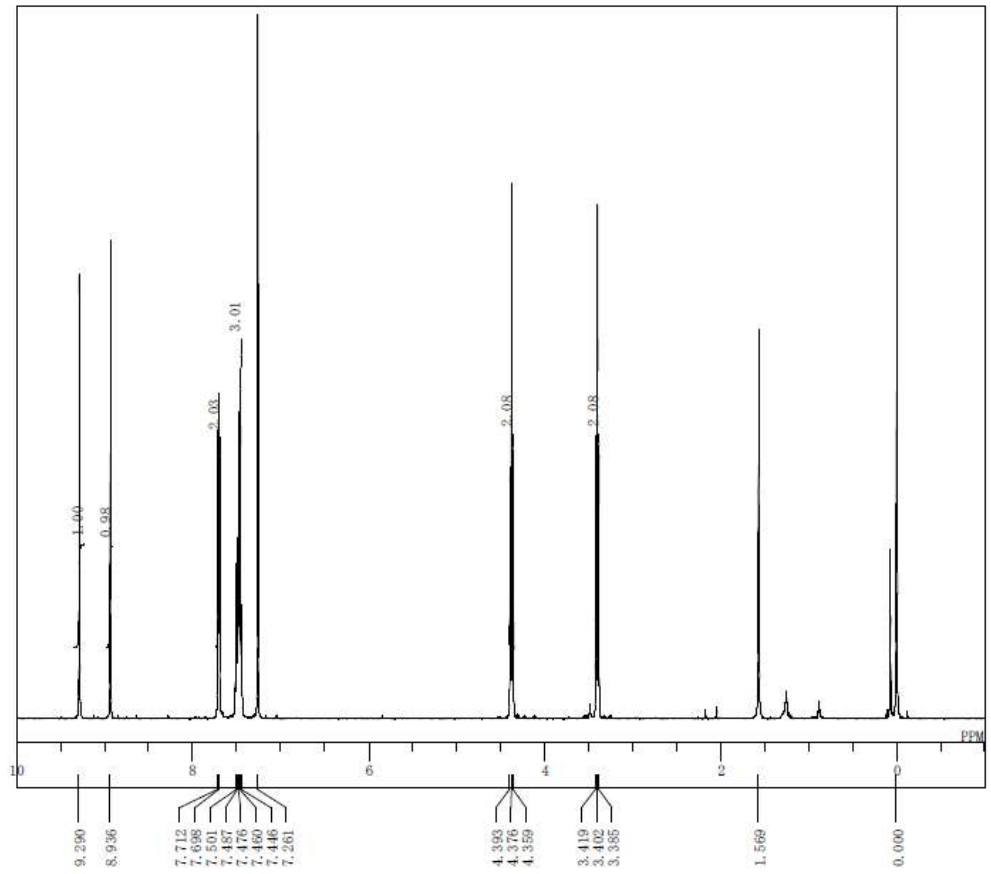
4j



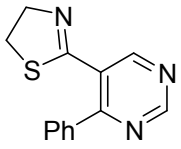

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   25-JAN-2007 17:39:18
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.00 MHz
OBSET         777.00 KHz
OBFIN         875.47 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS             1581
ACQTM         1.0420 sec
PD            1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP          26.8 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              0.05 Hz
RGAIN             30
```



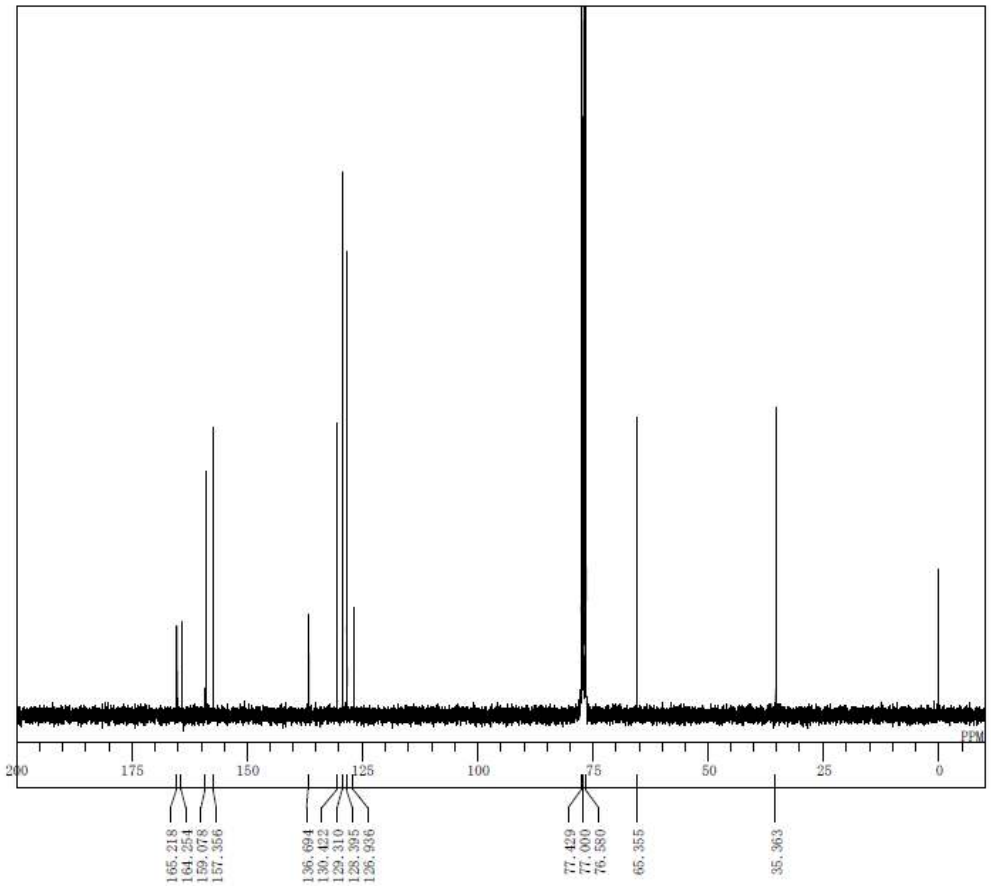
4j



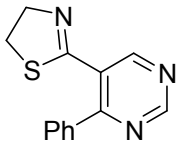

```
COMNT   Single Pulse Experiment
DATIM   20-JAN-2007 16:21:10
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.00 MHz
OBSET         162.00 KHz
OBFIN         416.02 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM         2.1823 sec
PD            4.0000 sec
PW1             7.50 usec
IRNUC   1H
CTEMP          24.7 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             1.00 Hz
RGAIN            24
```



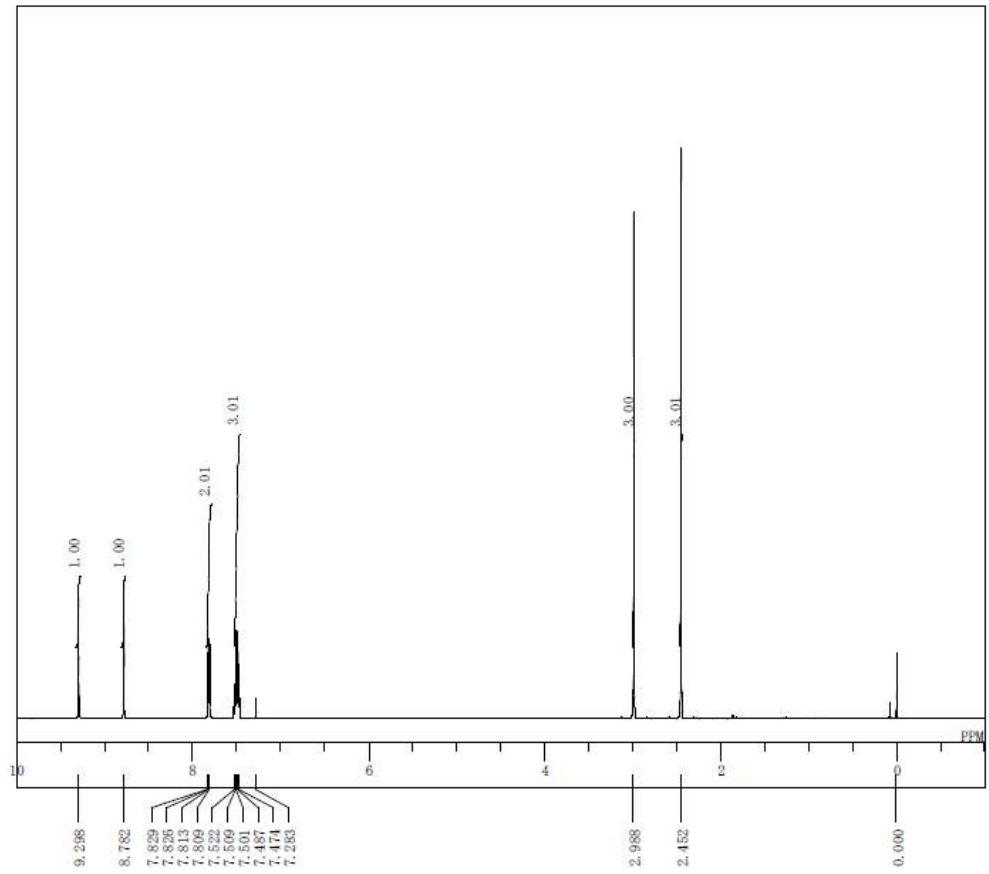
4k



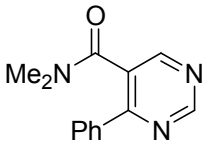

```
COMNT
DATIM   Mon Jan 22 09:42:19 2007
OBNUC   13C
EXMOD   BCM
OBFRQ          75.45 MHz
OBSET         124.00 KHz
OBFIN        1840.00 Hz
POINT          32768
FREQU       20408.10 Hz
SCANS             46481
ACQTM          1.6056 sec
PD             1.3940 sec
PW1              4.00 usec
IRNUC   1H
CTEMP          19.3 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              0.05 Hz
RGAIN             26
```



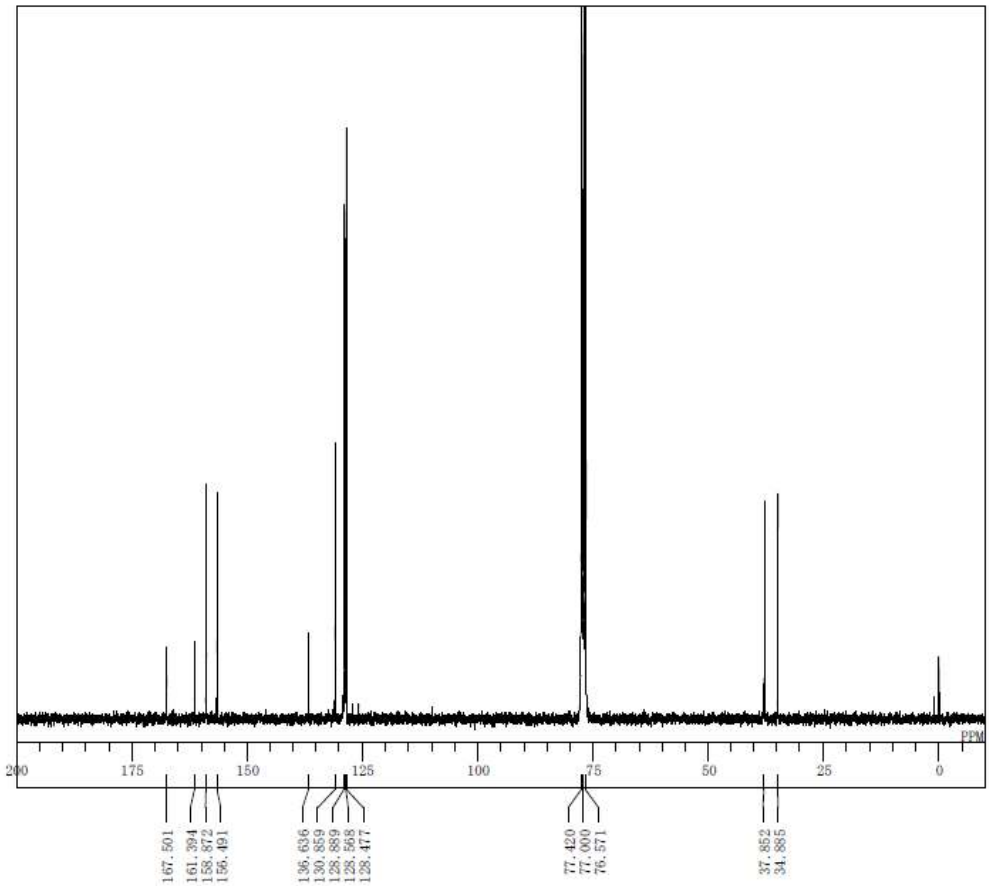
4k



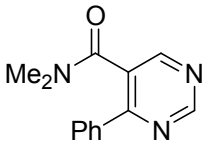

```
COMNT   Single Pulse Experiment
DATIM   27-JAN-2007 13:22:39
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.00 MHz
OBSET         162.00 KHz
OBFIN         416.02 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM         2.1823 sec
PD            4.0000 sec
PW1             7.50 usec
IRNUC   1H
CTEMP          26.4 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.23 Hz
RGAIN            15
```



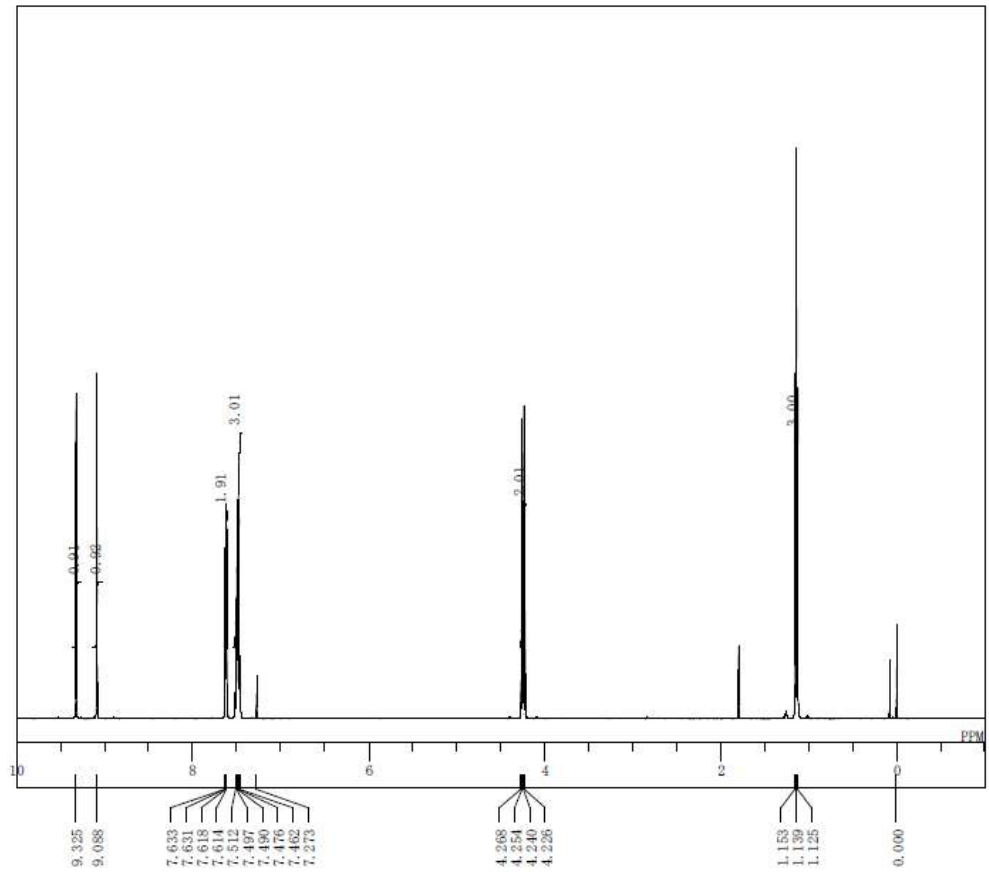
4l



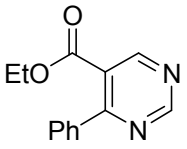

```
COMNT
DATIM   Sat Dec 09 09:28:54 2006
OBNUC   13C
EXMOD   BCM
OBFRQ          75.45 MHz
OBSET         124.00 KHz
OBFIN        1840.00 Hz
POINT          32768
FREQU       20408.10 Hz
SCANS          14295
ACQTM          1.6056 sec
PD             1.3940 sec
PW1              4.00 usec
IRNUC   1H
CTEMP          22.0 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              1.00 Hz
RGAIN             27
```



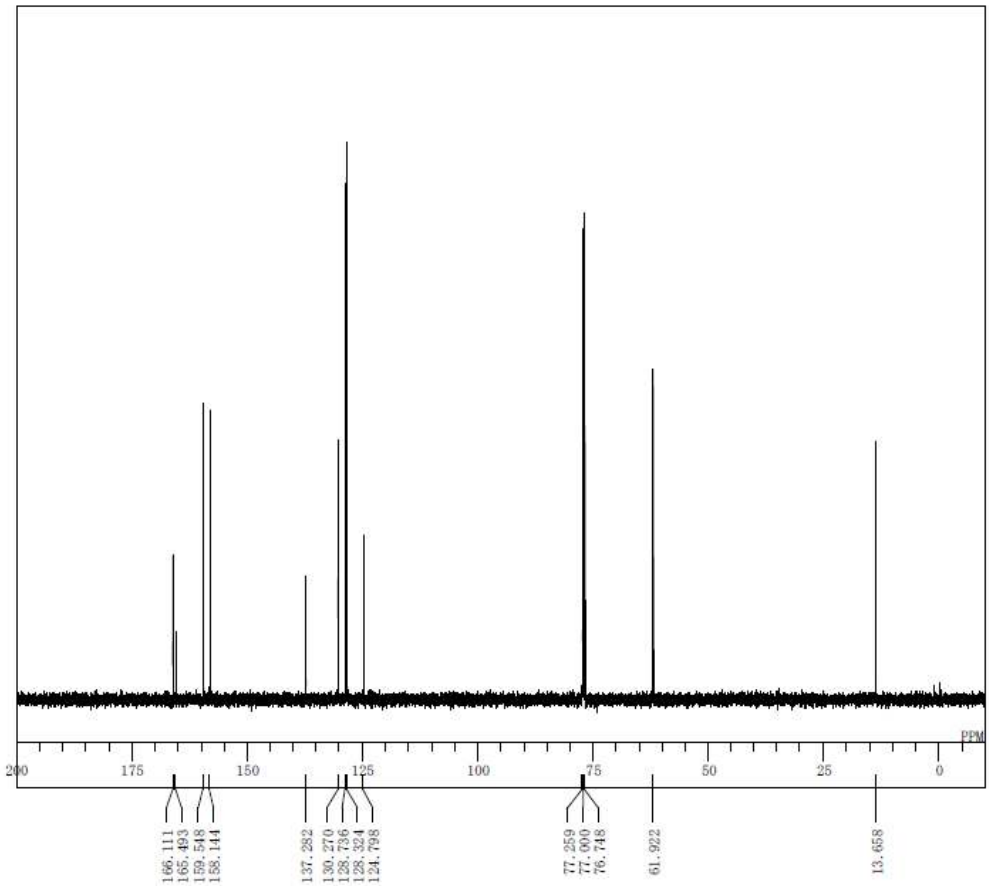
4l



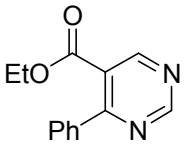
COMNT Single Pulse Experiment
DATIM 14-05-2008 18:12:28
OBNUC 1H
EXMOD single_pulse.exp
OBFRQ 500.16 MHz
OBSET 2.41 KHz
OBFIN 6.01 Hz
POINT 16384
FREQU 7507.51 Hz
SCANS 8
ACQTM 2.1823 sec
PD 4.0000 sec
PW1 7.00 usec
IRNUC
CTEMP 23.2 c
SLVNT CDCL3
EXREF 0.00 ppm
BF 0.23 Hz
RGAIN 15



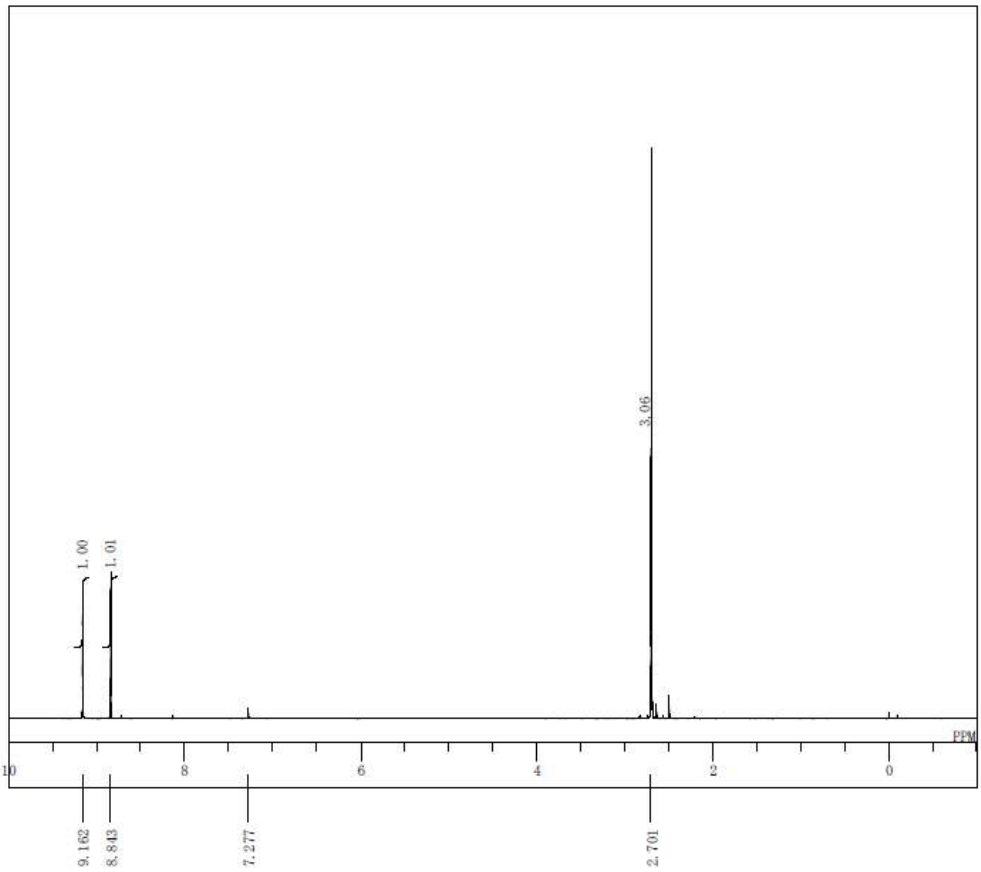
4m



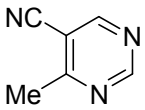
COMNT Single Pulse with Broadband Decoupling
DATIM 14-05-2008 18:29:26
OBNUC 13C
EXMOD single_pulse_dec
OBFRQ 125.77 MHz
OBSET 7.87 KHz
OBFIN 4.21 Hz
POINT 32768
FREQU 31446.54 Hz
SCANS 388
ACQTM 1.0420 sec
PD 1.0000 sec
PW1 4.17 usec
IRNUC 1H
CTEMP 26.5 c
SLVNT CDCL3
EXREF 77.00 ppm
BF 0.48 Hz
RGAIN 30



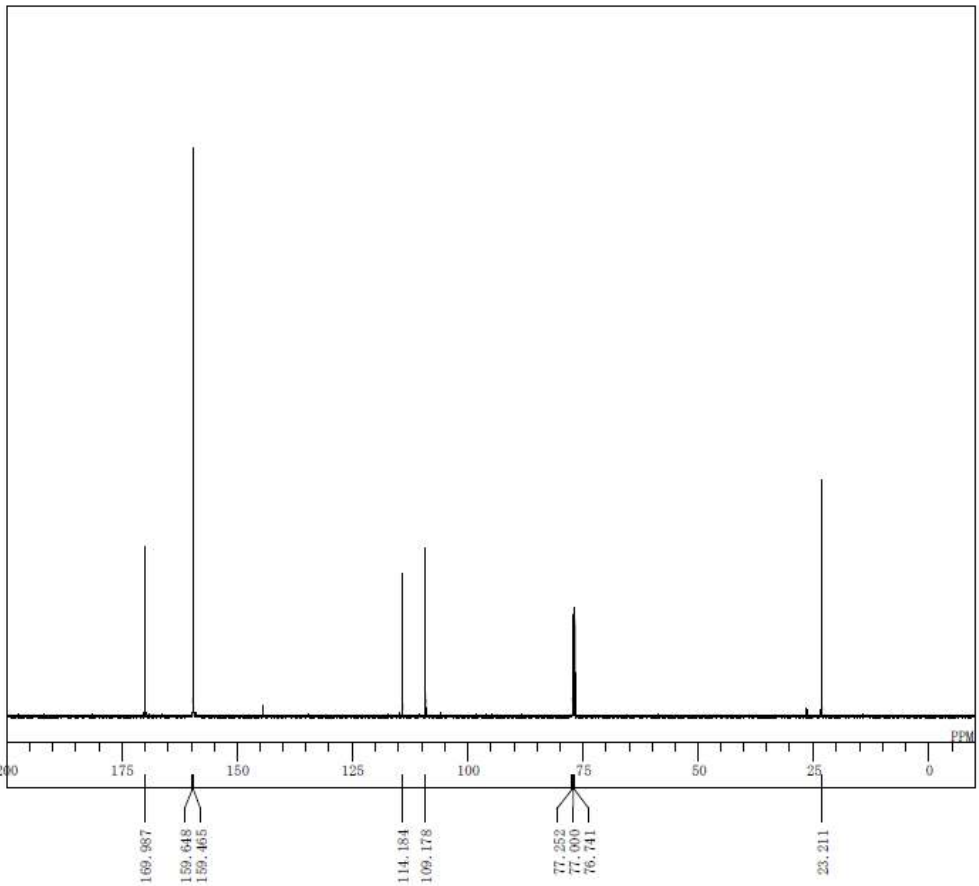
4m



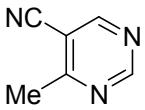

```
COMNT   Single Pulse Experiment
DATIM   24-01-2008 14:39:25
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            25.1 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              12
```



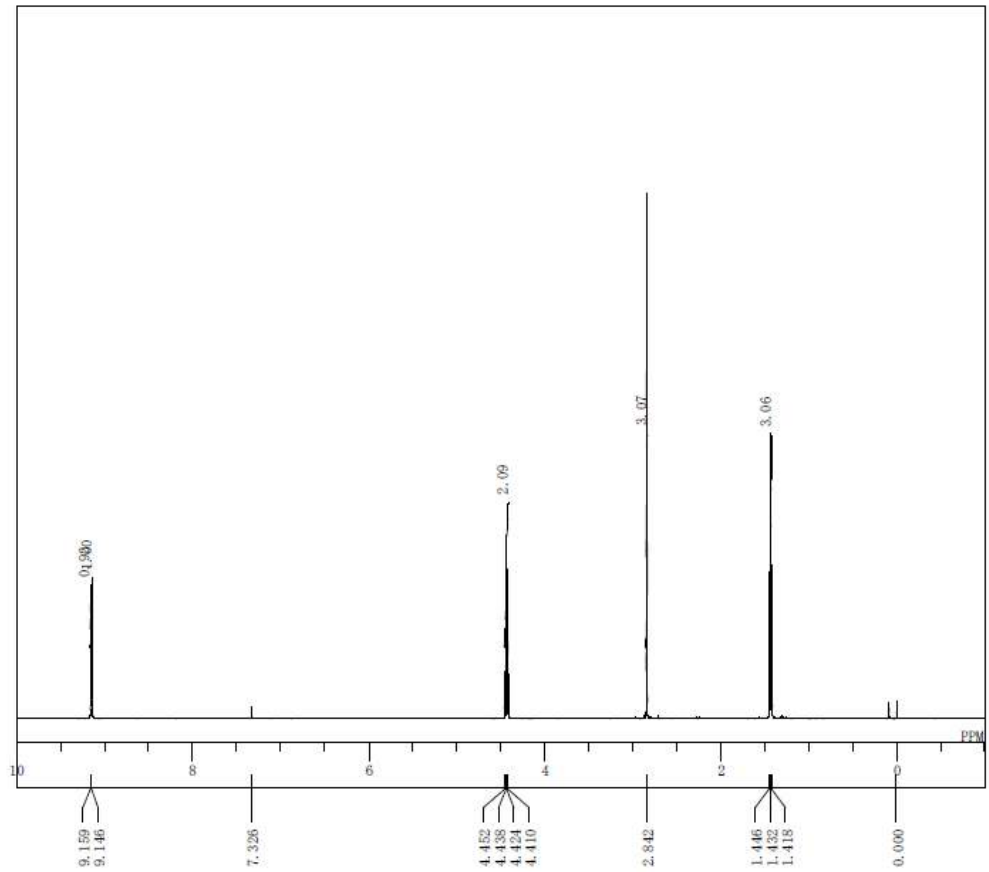
4n



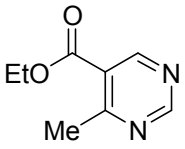

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   24-01-2008 15:01:19
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU        31446.54 Hz
SCANS            617
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            27.2 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF               0.48 Hz
RGAIN              30
```



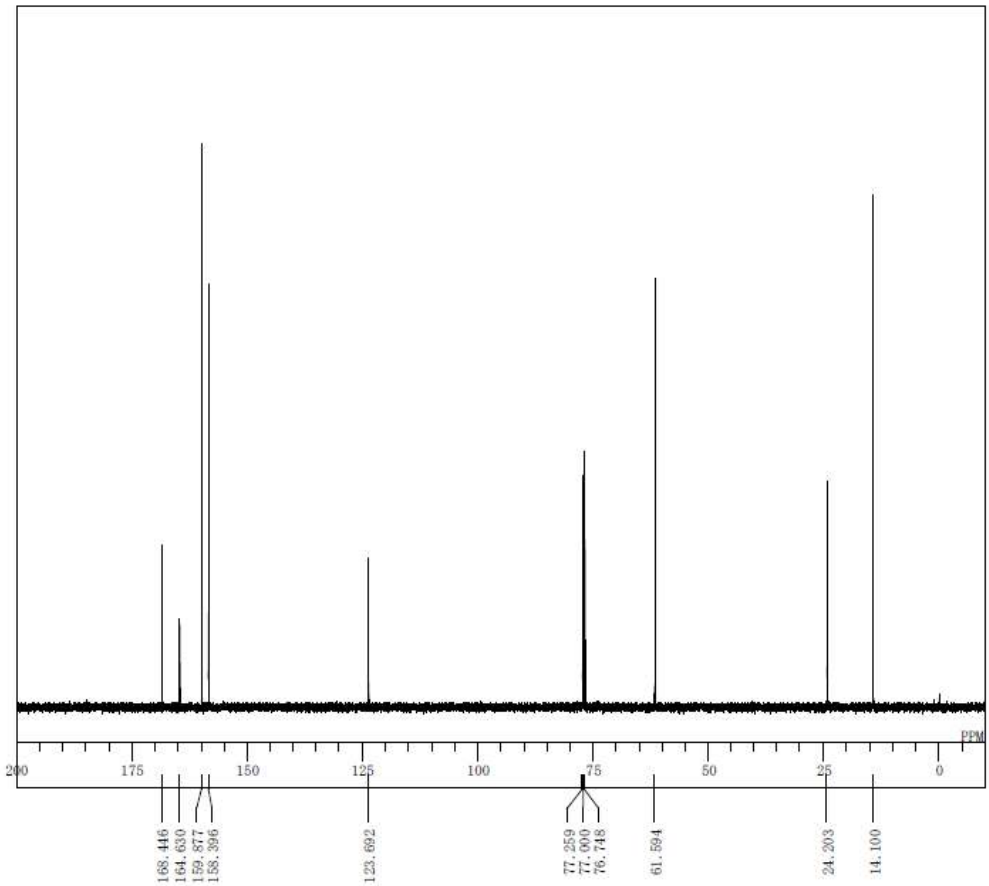
4n



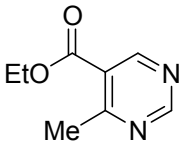

```
COMNT   Single Pulse Experiment
DATIM   21-01-2008 12:24:50
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS              8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP           26.3 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.23 Hz
RGAIN             13
```



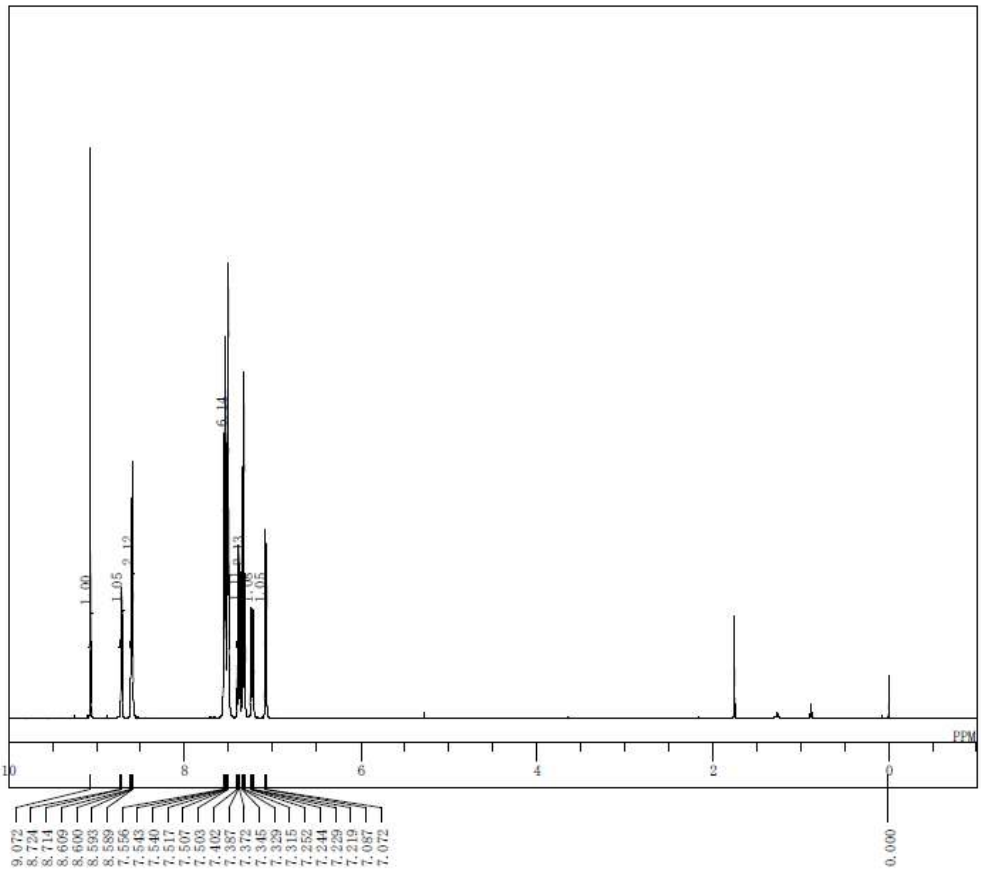
4o



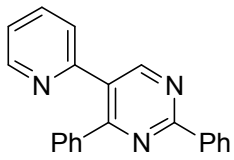

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   21-01-2008 12:38:08
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS            297
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP           28.0 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.48 Hz
RGAIN             30
```



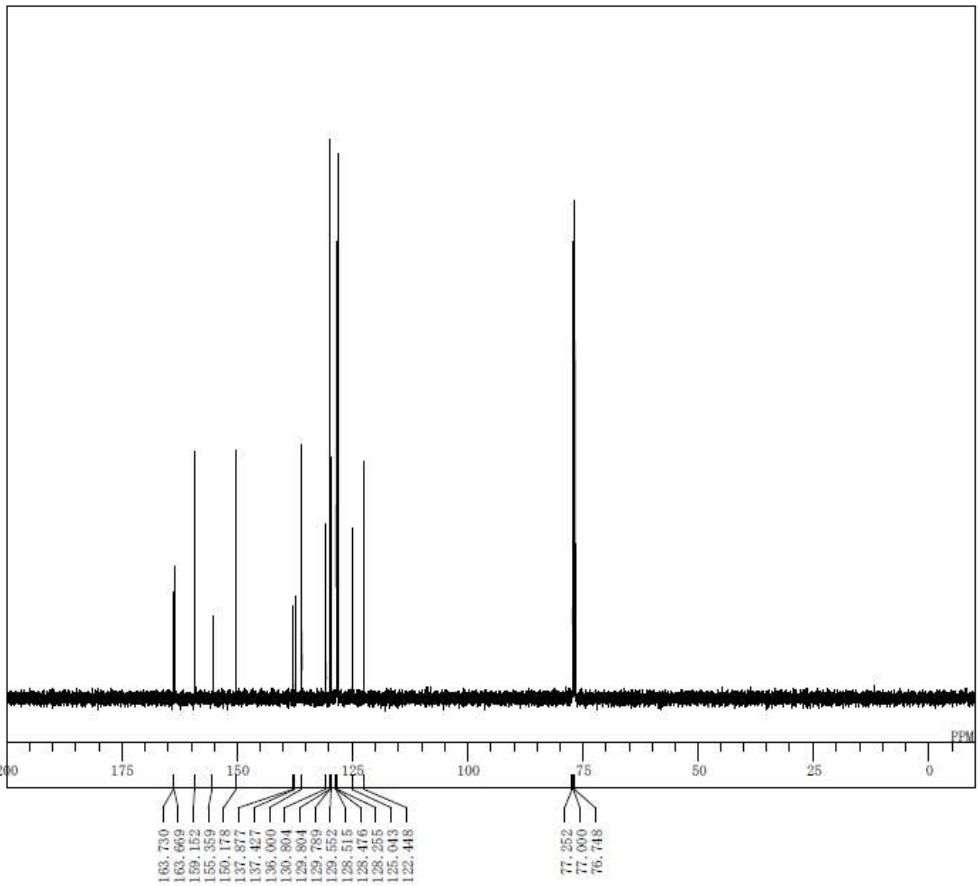
4o



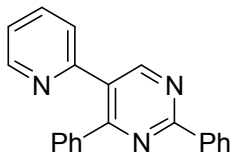

```
COMNT   Single Pulse Experiment
DATIM   15-03-2008 14:49:12
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            26.7 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              15
```



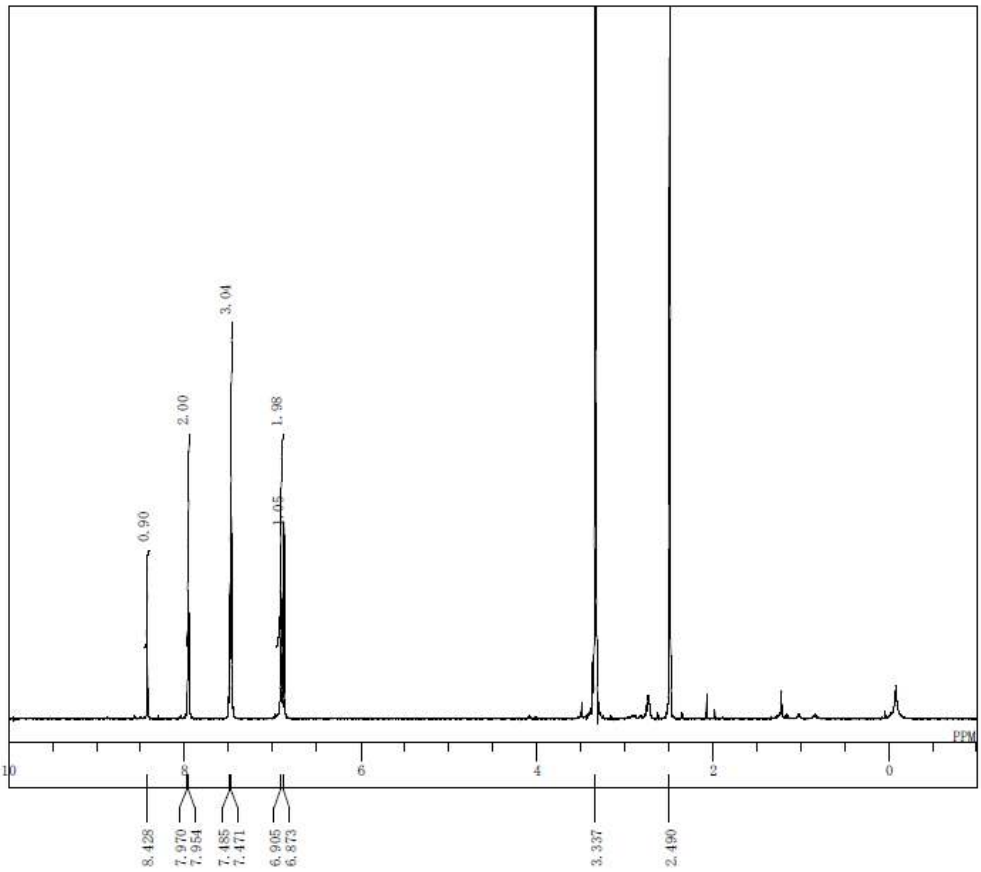
5



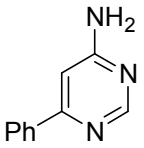

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   15-03-2008 15:05:24
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU        31446.54 Hz
SCANS            413
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            29.1 c
SLVNT   CDCL3
EXREF            77.00 ppm
BF               0.48 Hz
RGAIN              30
```



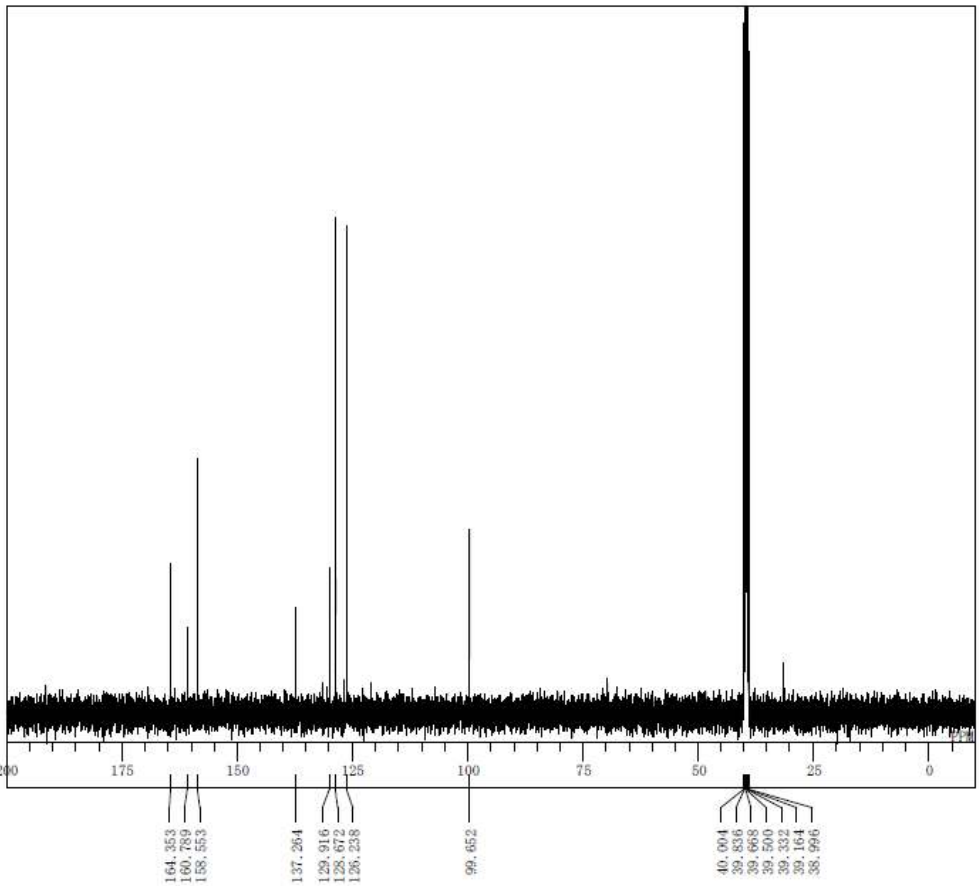
5



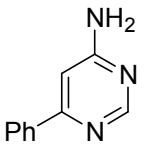

```
COMNT   Single Pulse Experiment
DATIM   05-09-2008 13:02:34
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            21.1 c
SLVNT   DMSO
EXREF           2.49 ppm
BF              0.23 Hz
RGAIN             19
```



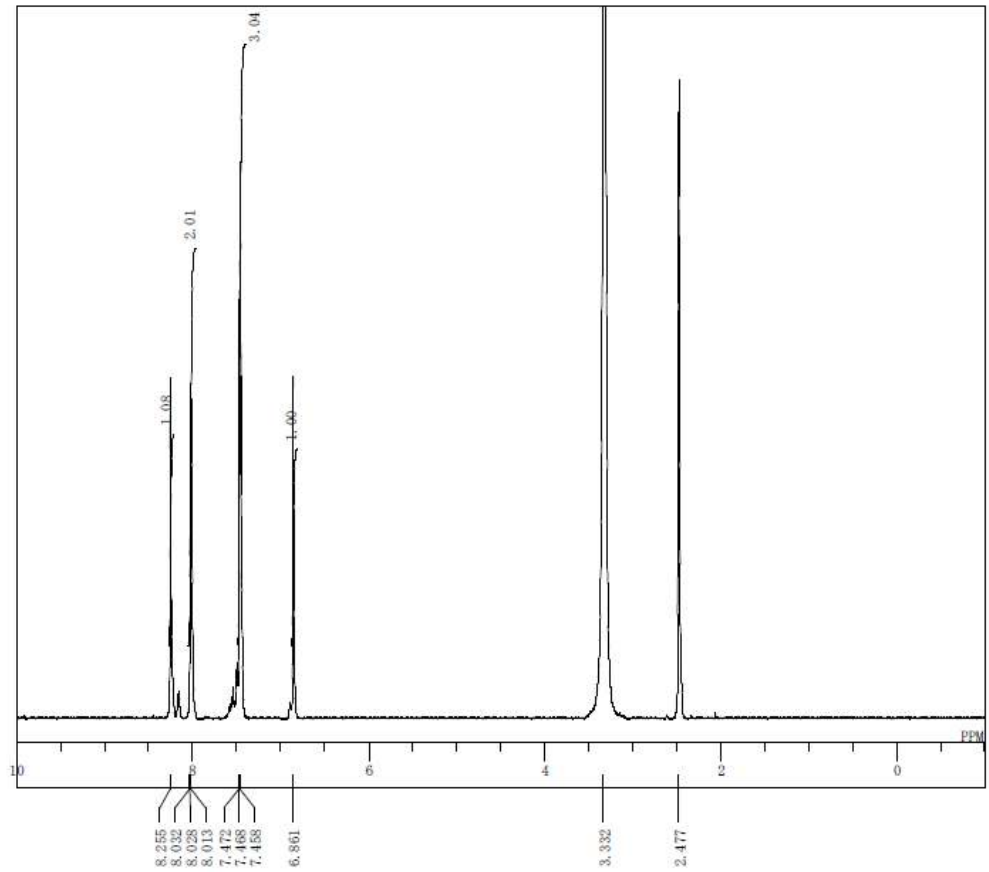
6



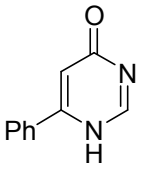

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   05-03-2008 18:54:27
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU        31446.54 Hz
SCANS           2072
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            28.3 c
SLVNT   DMSO
EXREF           39.50 ppm
BF              0.48 Hz
RGAIN             30
```



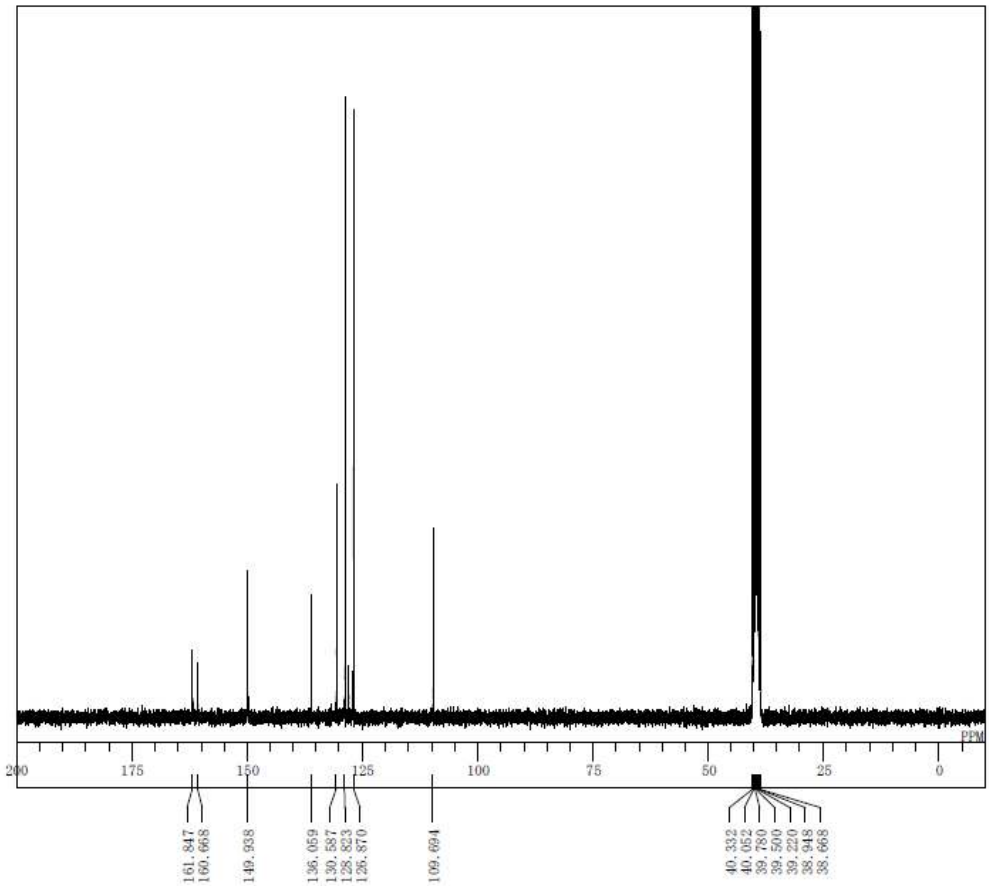
6



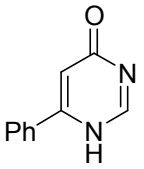

```
COMNT   Single Pulse Experiment
DATIM   25-01-2008 12:52:45
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            24.8 c
SLVNT   DMSO
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              17
```



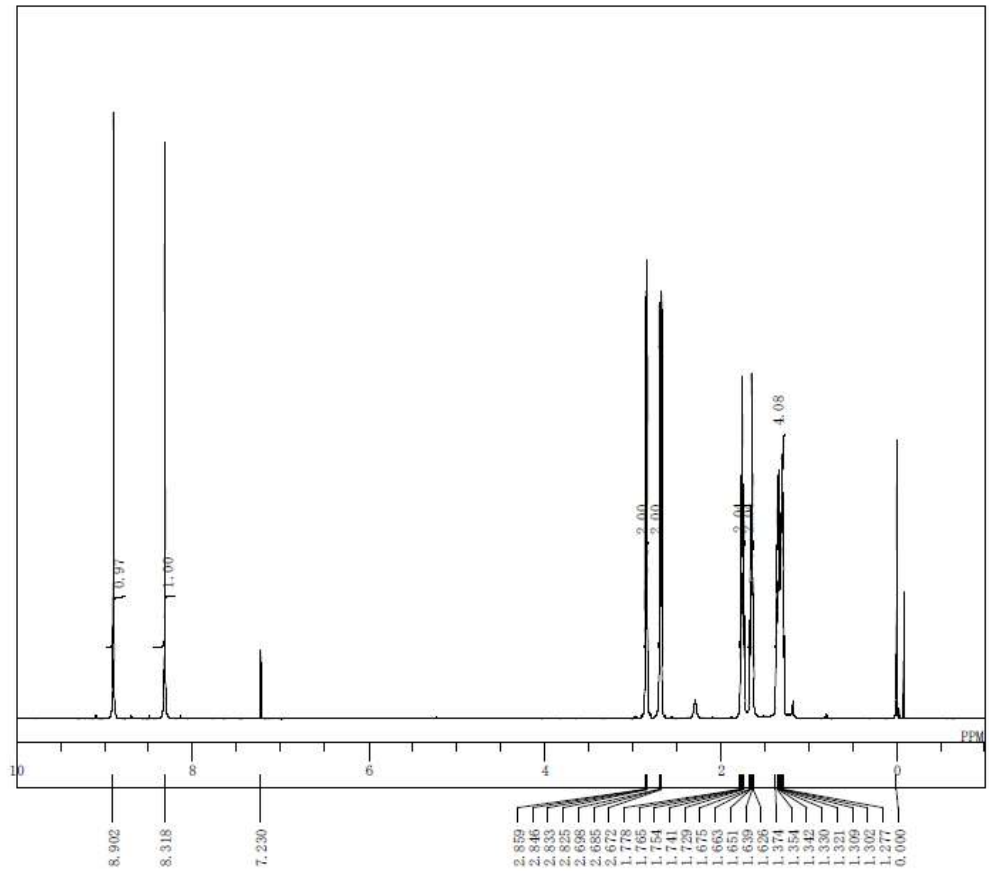
7



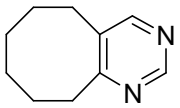

```
COMNT
DATIM   Fri Jan 25 10:13:24 2008
OBNUC   13C
EXMOD   BCM
OBFRQ           75.45 MHz
OBSET          124.00 KHz
OBFIN         1840.00 Hz
POINT           32768
FREQU        20408.10 Hz
SCANS            17061
ACQTM            1.6056 sec
PD               1.3940 sec
PW1              4.00 usec
IRNUC   1H
CTEMP             16.5 c
SLVNT   DMSO
EXREF            39.50 ppm
BF                0.31 Hz
RGAIN               24
```



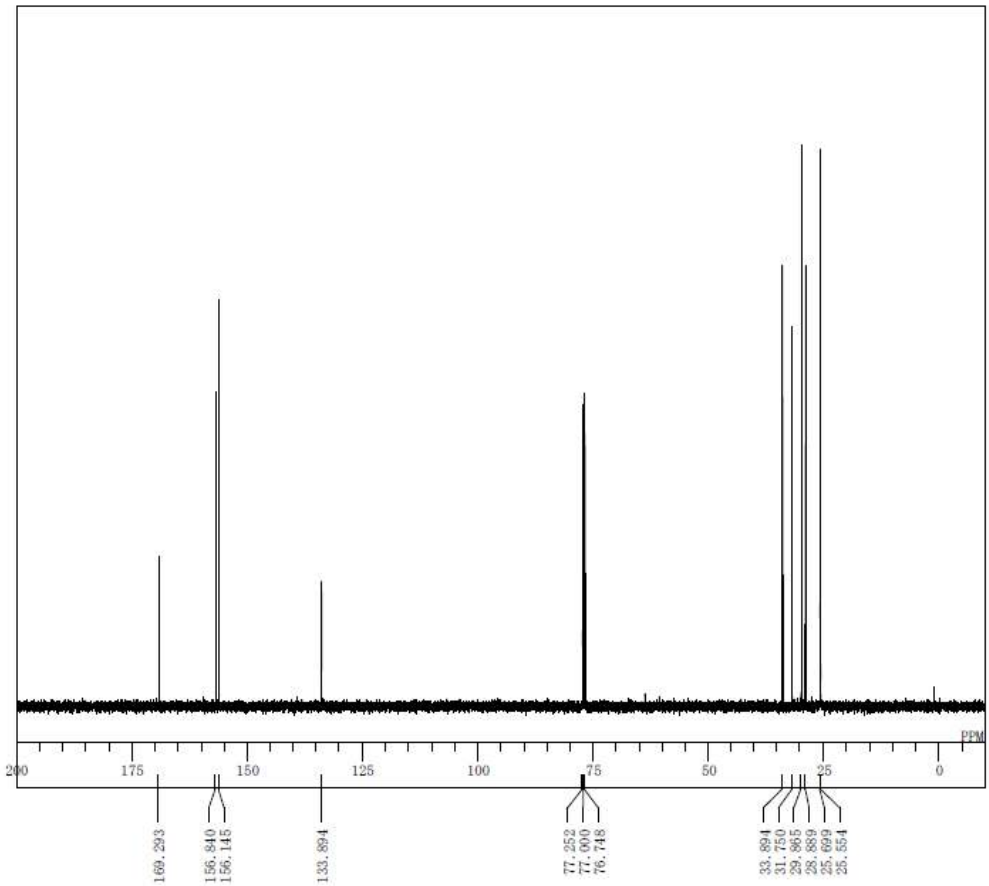
7



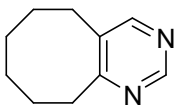

```
COMNT   Single Pulse Experiment
DATIM   03-04-2008 19:12:11
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP          25.8 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.23 Hz
RGAIN             13
```



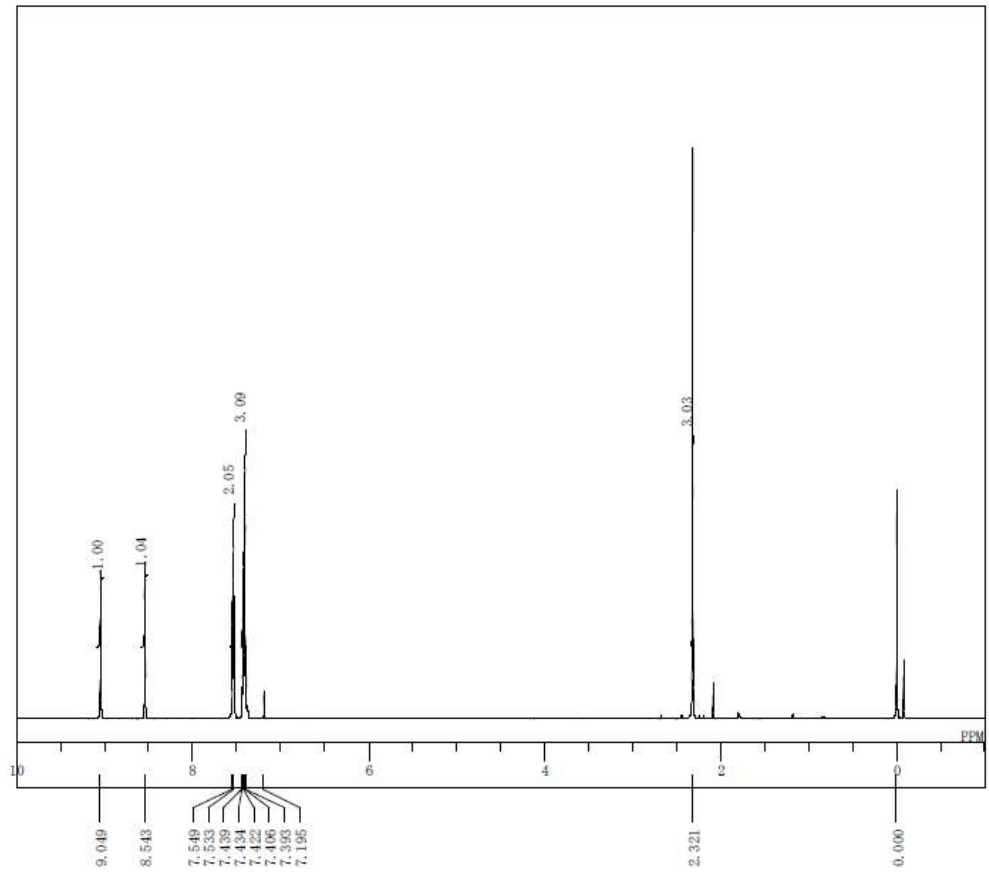
9a



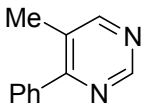

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   03-04-2008 19:23:32
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS            291
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP          28.0 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              0.48 Hz
RGAIN             30
```



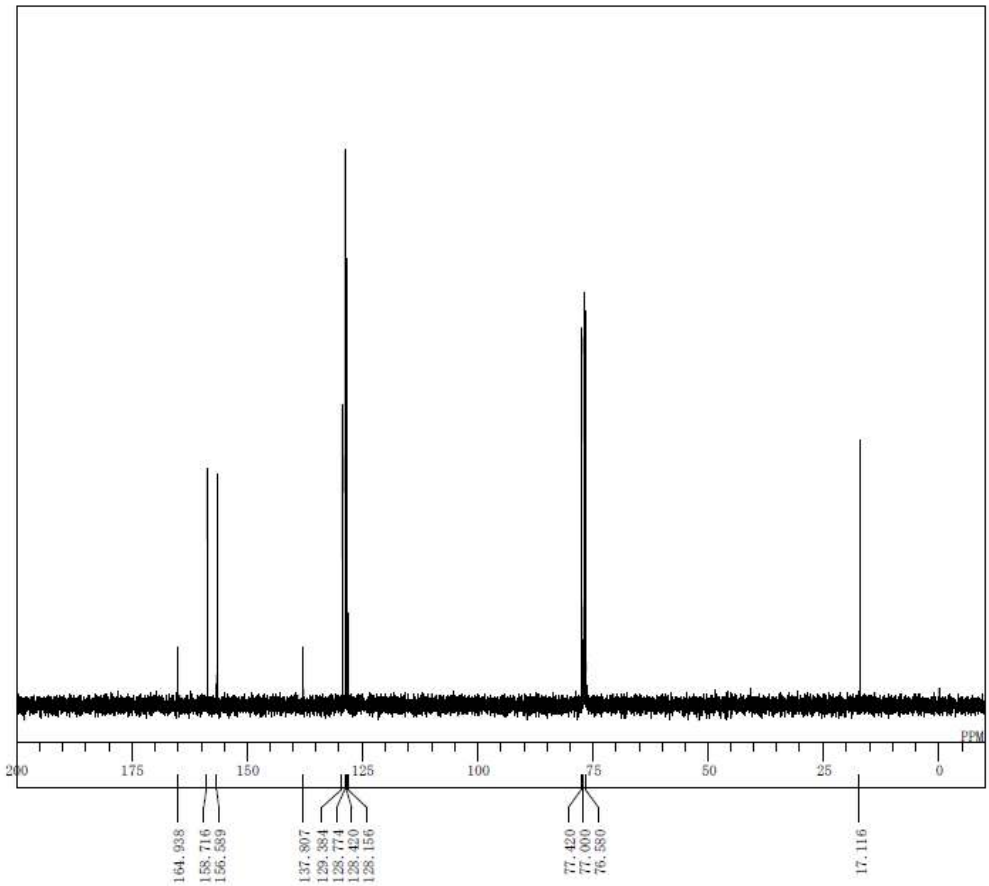
9a



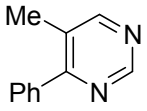

```
COMNT   Single Pulse Experiment
DATIM   26-02-2008 10:03:46
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            26.0 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              15
```



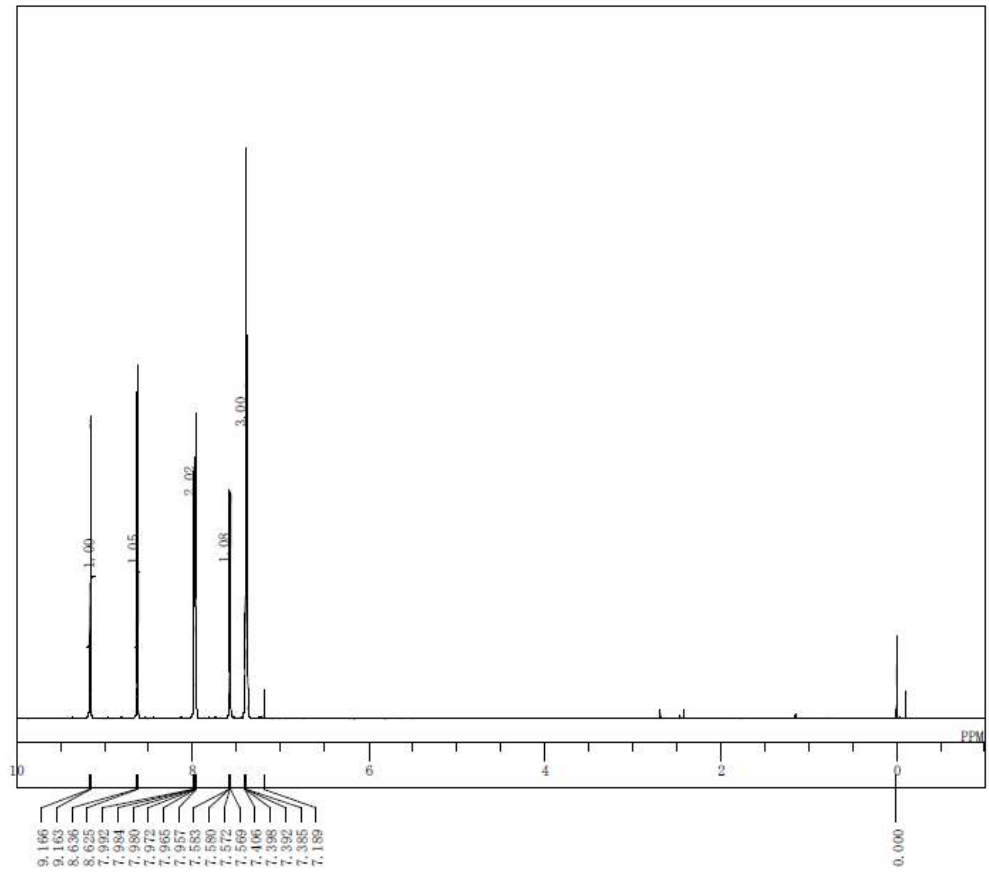
9b



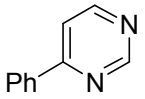

```
COMNT
DATIM   Thu Feb 28 19:53:18 2008
OBNUC   13C
EXMOD   BCM
OBFRQ          75.45 MHz
OBSET         124.00 KHz
OBFIN        1840.00 Hz
POINT          32768
FREQU        20408.10 Hz
SCANS            301
ACQTM           1.6056 sec
PD              1.3940 sec
PW1             4.00 usec
IRNUC   1H
CTEMP            19.0 c
SLVNT   CDCL3
EXREF            77.00 ppm
BF               0.31 Hz
RGAIN              28
```



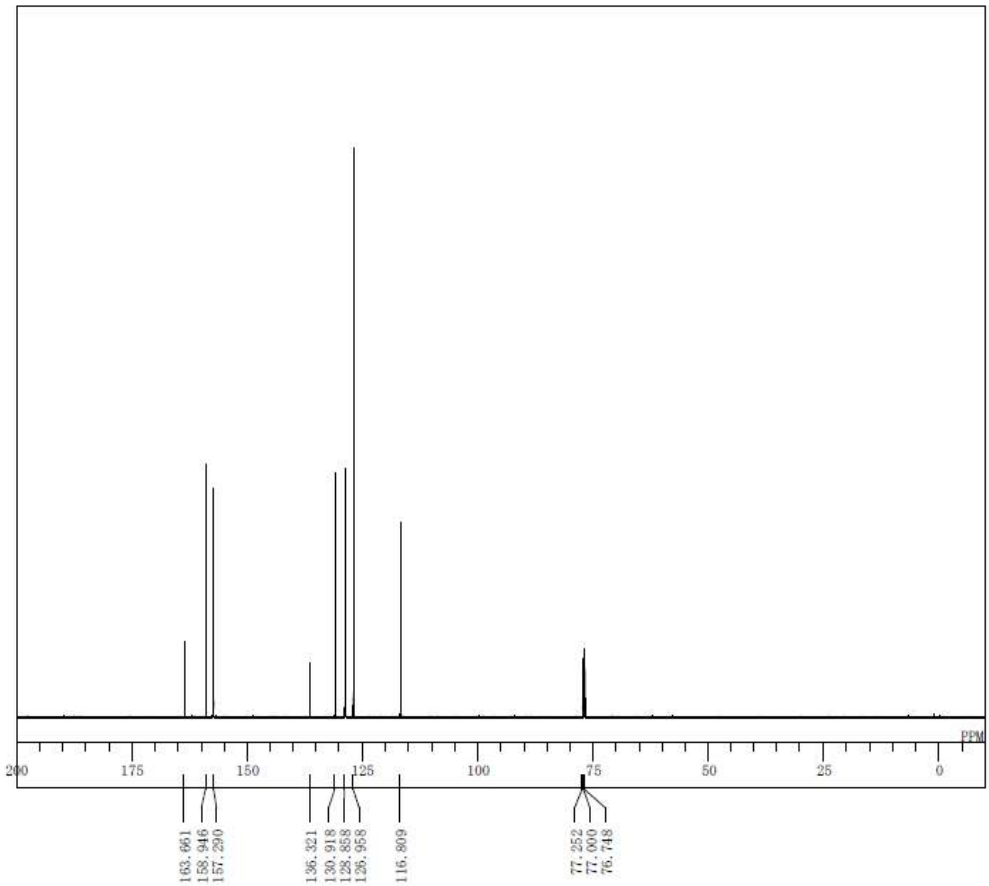
9b



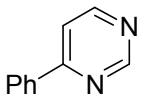

```
COMNT   Single Pulse Experiment
DATIM   24-01-2008 14:14:05
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            24.5 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              11
```



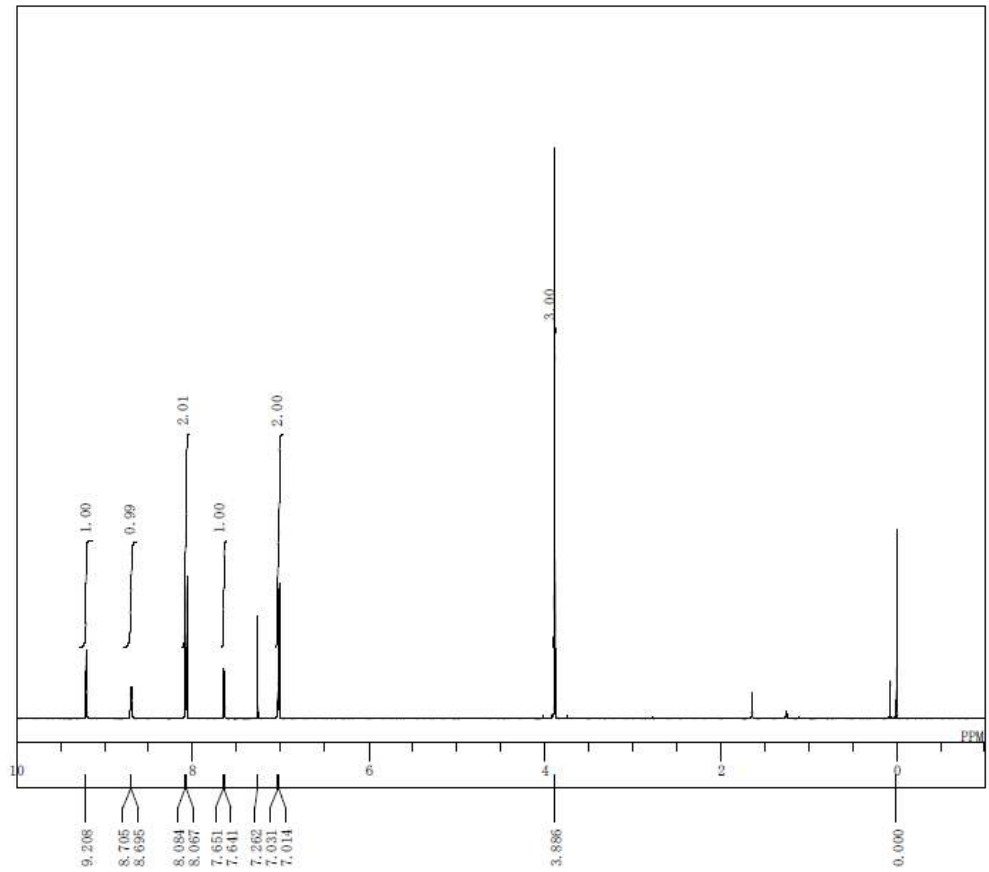
9c



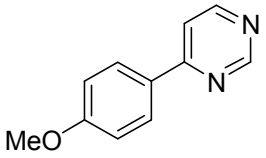

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   24-01-2008 14:33:47
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU        31446.54 Hz
SCANS            553
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            27.1 c
SLVNT   CDCL3
EXREF            77.00 ppm
BF               0.48 Hz
RGAIN              30
```



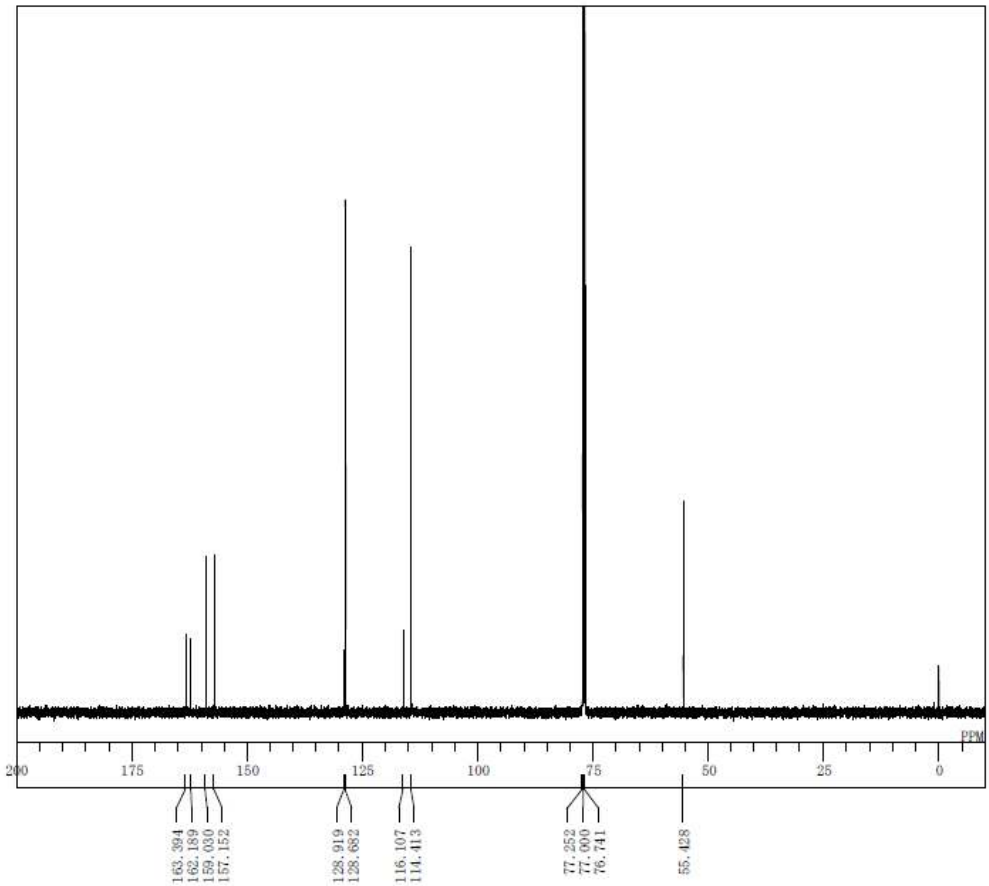
9c



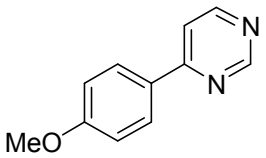

```
COMNT   Single Pulse Experiment
DATIM   21-03-2008 13:10:13
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP            27.5 c
SLVNT   CDCL3
EXREF            0.00 ppm
BF               0.23 Hz
RGAIN              21
```



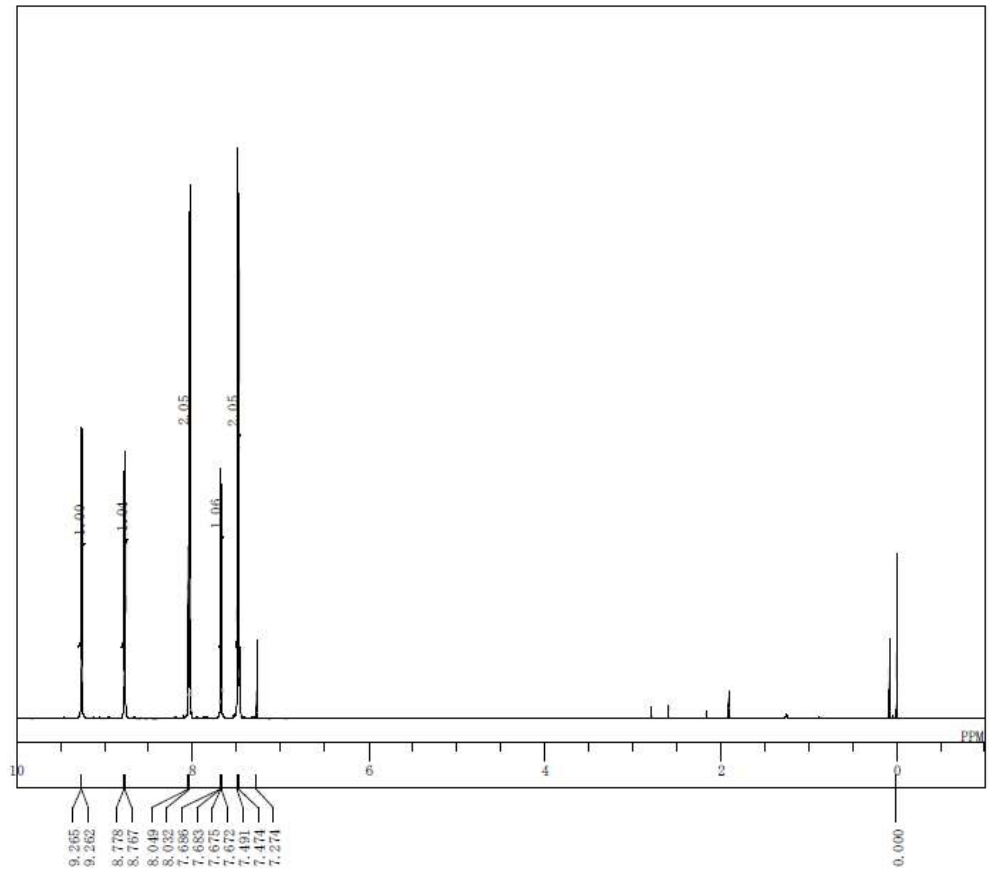
9d



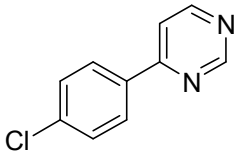

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   21-03-2008 14:45:36
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU        31446.54 Hz
SCANS           2777
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP            29.1 c
SLVNT   CDCL3
EXREF            77.00 ppm
BF               0.48 Hz
RGAIN              30
```



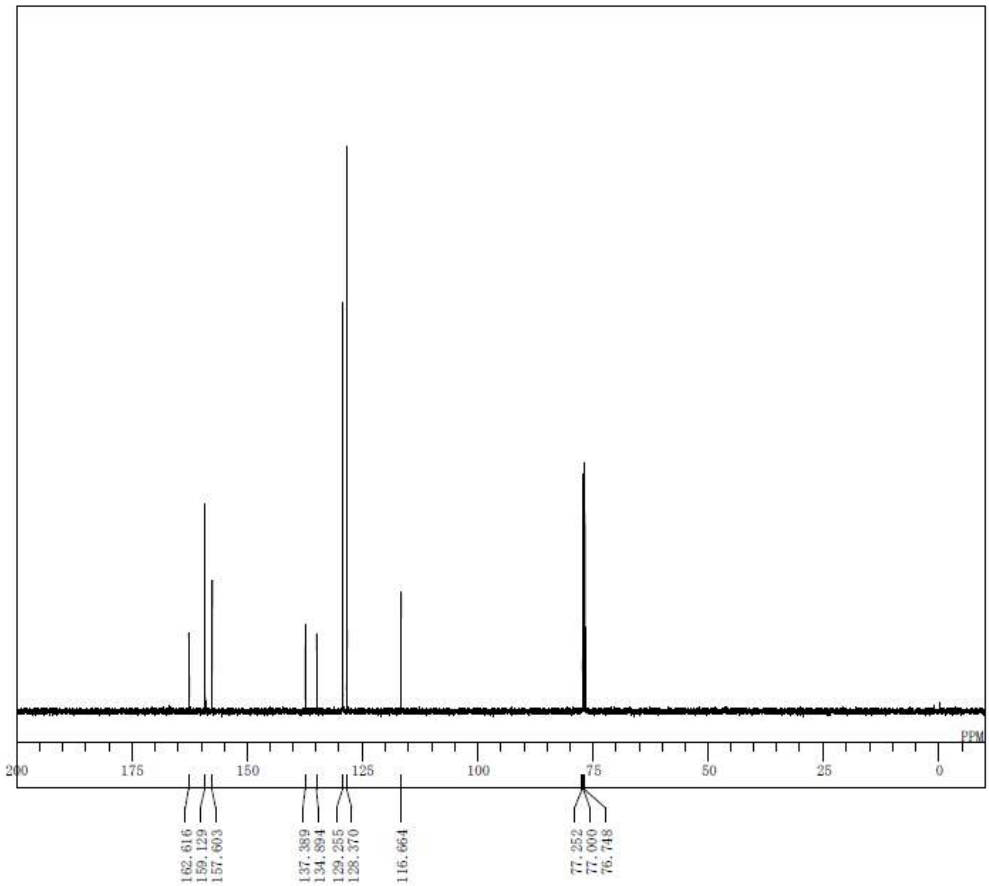
9d



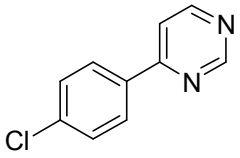

```
COMNT   Single Pulse Experiment
DATIM   21-03-2008 18:24:59
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           2.41 KHz
OBFIN           6.01 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM           2.1823 sec
PD              4.0000 sec
PW1             7.00 usec
IRNUC
CTEMP           28.1 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.23 Hz
RGAIN             17
```



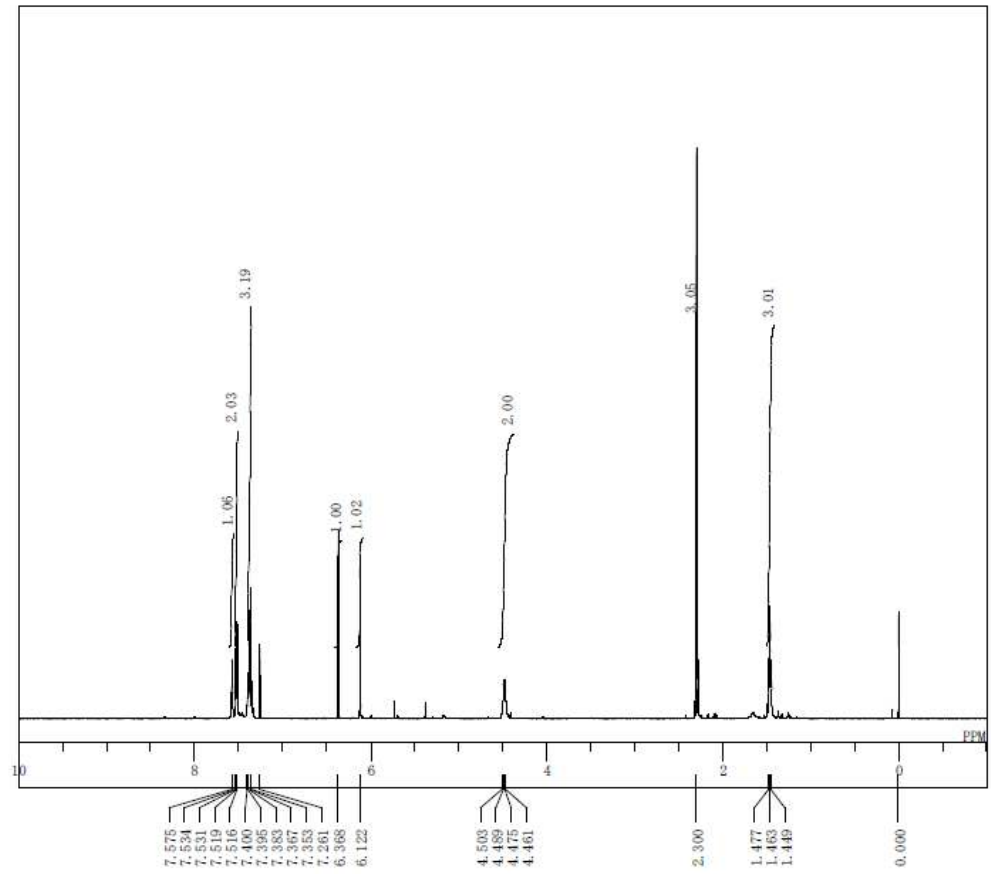
9e



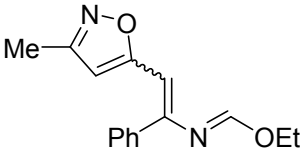

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   21-03-2008 18:43:17
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS            512
ACQTM           1.0420 sec
PD              1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP           29.9 c
SLVNT   CDCL3
EXREF          77.00 ppm
BF              0.48 Hz
RGAIN             30
```



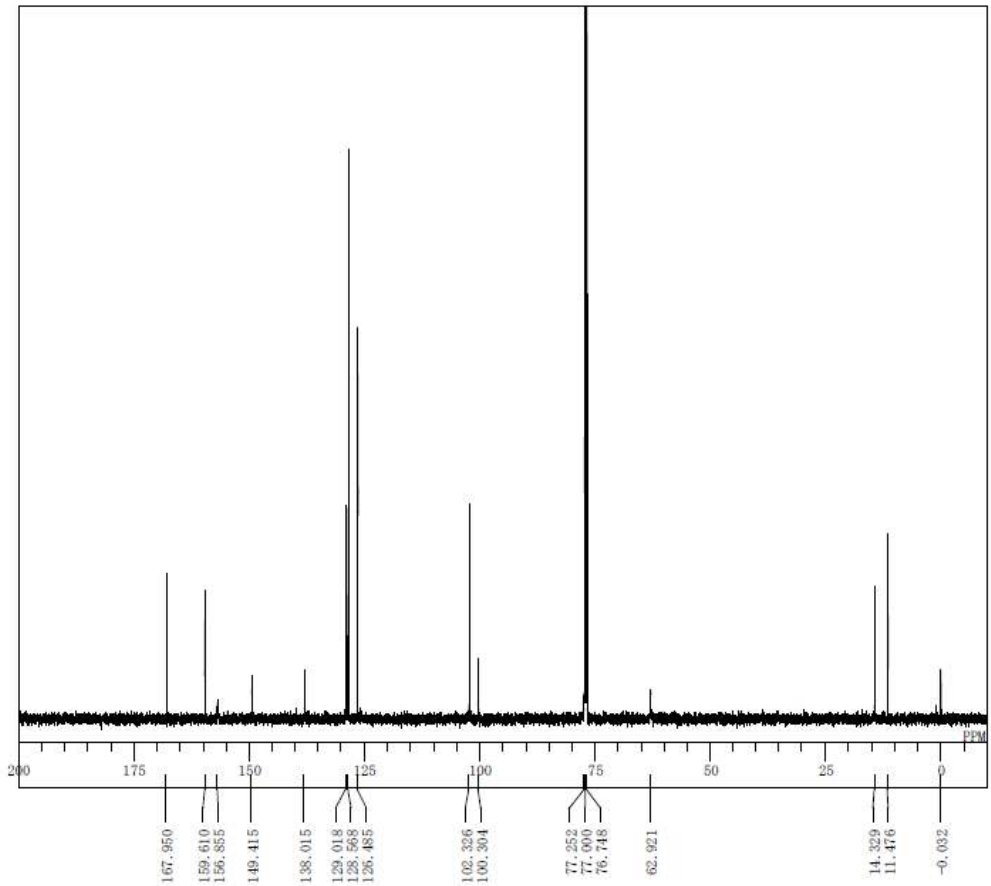
9e



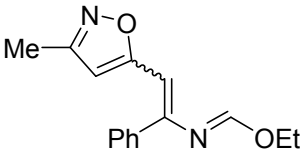

```
COMNT   Single Pulse Experiment
DATIM   28-APR-2006 12:55:56
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.00 MHz
OBSET         162.00 KHz
OBFIN         416.02 Hz
POINT          16384
FREQU        7507.51 Hz
SCANS             8
ACQTM         2.1823 sec
PD            4.0000 sec
PW1             7.50 usec
IRNUC   1H
CTEMP          23.7 c
SLVNT   CDCL3
EXREF          0.00 ppm
BF             0.23 Hz
RGAIN            17
```



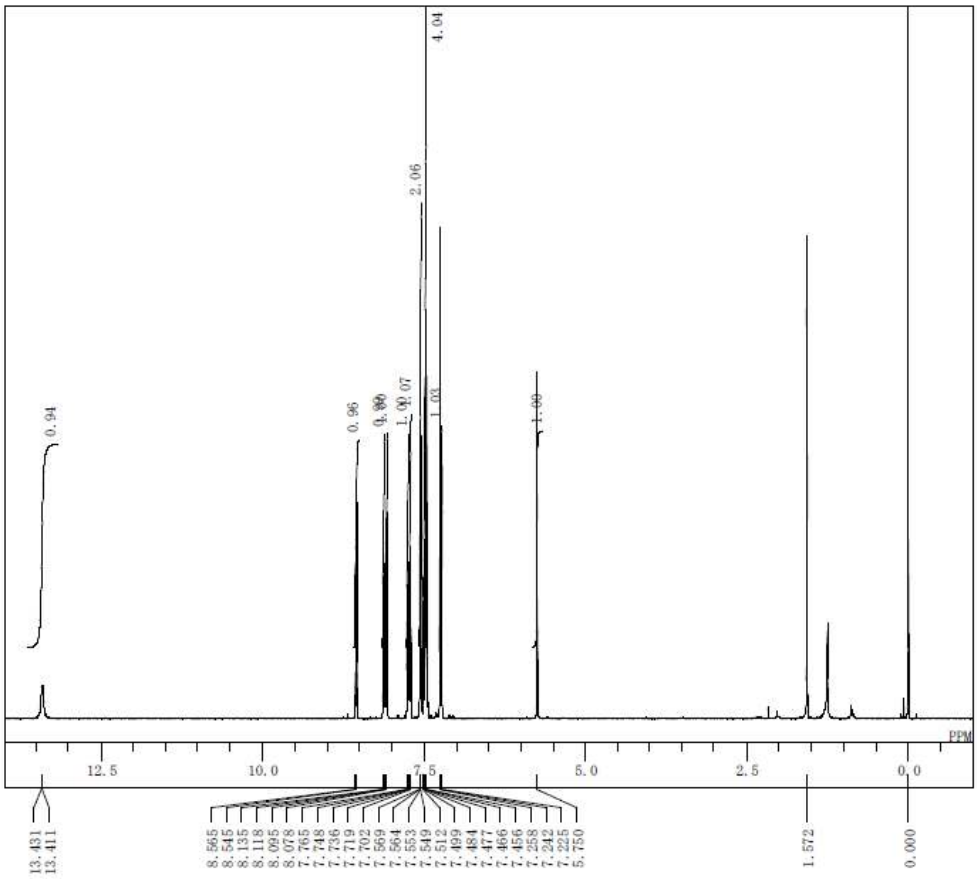
10a



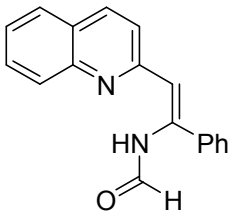

```
COMNT   Single Pulse with Broadband Decoupling
DATIM   18-FEB-2006 14:02:08
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.00 MHz
OBSET         777.00 KHz
OBFIN         875.47 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS             2661
ACQTM         1.0420 sec
PD            1.0000 sec
PW1             4.17 usec
IRNUC   1H
CTEMP          27.9 c
SLVNT   CDCL3
EXREF         77.00 ppm
BF             0.96 Hz
RGAIN            30
```



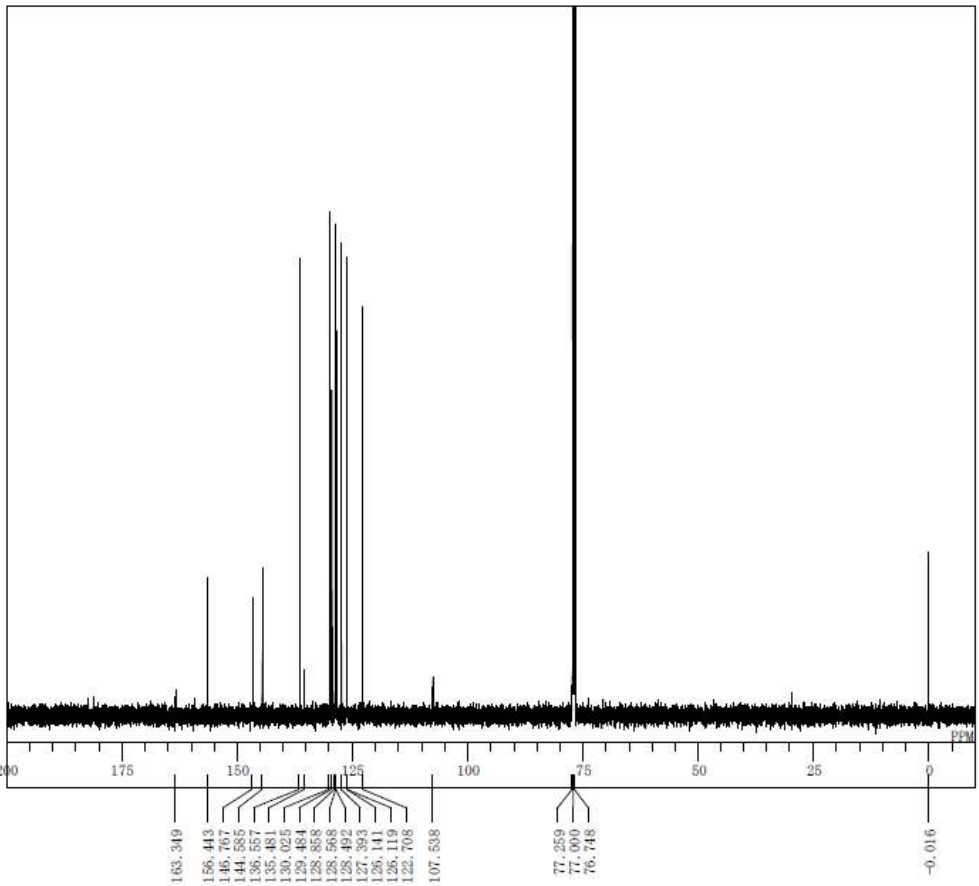
10a



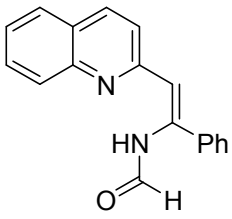

```
COMNT   Single Pulse Experiment
DATIM   01-11-2008 11:37:46
OBNUC   1H
EXMOD   single_pulse.exp
OBFRQ         500.16 MHz
OBSET           3.41 KHz
OBFIN           6.33 Hz
POINT          16384
FREQU       10010.01 Hz
SCANS              8
ACQTM          1.6368 sec
PD             4.0000 sec
PW1              7.00 usec
IRNUC
CTEMP           25.0 c
SLVNT   CDCL3
EXREF           0.00 ppm
BF              0.30 Hz
RGAIN             22
```



13a




```
COMNT   Single Pulse with Broadband Decoupling
DATIM   02-11-2008 08:08:05
OBNUC   13C
EXMOD   single_pulse_dec
OBFRQ         125.77 MHz
OBSET           7.87 KHz
OBFIN           4.21 Hz
POINT          32768
FREQU       31446.54 Hz
SCANS             23489
ACQTM          1.0420 sec
PD             1.0000 sec
PW1              4.17 usec
IRNUC   1H
CTEMP           27.6 c
SLVNT   CDCL3
EXREF           77.00 ppm
BF              0.09 Hz
RGAIN             30
```



13a