

Supporting information

Vilsmeier Cyclization of α -Acetyl- α -aroyl Ketene-*N*, *S*-Acetals: Direct and Efficient Synthesis of Halogenated Pyridin-2(1*H*)-ones

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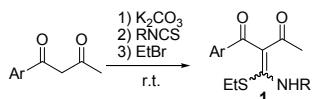
1. General Considerations

All the manipulations of air-and/or moisture-sensitive compounds were carried out under a nitrogen atmosphere using standard Schlenk techniques. Reaction solvents were dried and distilled prior to use by the literature methods. ^1H and $^{13}\text{C}\{1\text{H}\}$ NMR spectra were recorded on a Bruker DRX-600 spectrometer and all chemical shift values refer to δ TMS = 0.00 ppm or CDCl_3 ($\delta(^1\text{H})$, 7.26 ppm; $\delta(^{13}\text{C})$, 77.16 ppm). The HRMS analysis was achieved on Bruck microTof by using ESI method. All the melting points were uncorrected. Analytical TLC plates, Sigma-Aldrich silica gel 60F200 were viewed by UV light (254 nm). Chromatographic purifications were performed on SDZF silica gel 160.

2. Experimental procedures

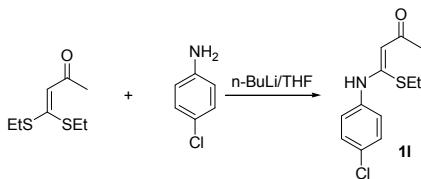
(1) Typical procedure for the preparation of α -acetyl- α -aryloyl ketene- N, S -acetals

1a-k



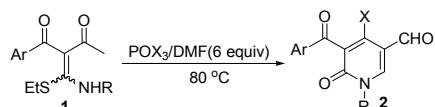
K_2CO_3 (5.0 mmol) was added to a stirred solution of 1-arylbutan-1,3-dione (5.0 mmol) in DMF (10 mL), and the mixture was stirred for 1 h at room temperature. The isothiocyanate (5.0 mmol) was then added dropwise, and the mixture was stirred for 2 h at room temperature. The bromoethane (5.0 mmol) and K_2CO_3 (5.0 mmol) was added. The reaction was quenched with 100 mL of H_2O after the mixture was further stirred 4 h at room temperature. The resulting mixture was extracted with dichloromethane (3×30 mL), and the combined organic phase was washed with water, dried over anhydrous MgSO_4 , filtered, and evaporated in vacuo. The crude product was purified by silica gel chromatography (petroleum ether ($30-60$ °C) /diethyl ether=30:1, v/v) to give α -acetyl- α -aryloyl ketene- N, S -acetals **1a-k**.

(2) Typical procedure for the preparation of α -acetyl- N, S -acetals **1l**



To a stirred solution of 4-chlorobenzenamine (635 mg, 5 mmol) in dry THF (20 mL) was added *n*-BuLi (2.6 mL, 6 mmol) under nitrogen atmosphere over a period of 10 min at -78 °C. The reaction mixture was brought to room temperature and further stirred for 45 min. A solution of 4, 4-bis(ethylthio)but-3-en-2-one (900 mg, 5 mmol) in dry THF (10 mL) was added at 0 °C, and the reaction mixture was further stirred at room temperature for 2 h. It was refluxed for 18-20 h to complete the reaction, cooled, poured into saturated NH₄Cl solution (50 mL), and extracted with CHCl₃ (3×15 mL). The combined extracts were washed with water and brine, dried (Na₂SO₄), and evaporated to give crude product which was purified by silica gel chromatography (petroleum ether/ethyl acetate=6:1, v/v) to give **1l** in 77% yield as a white solid.

(3) Typical Procedure for the Synthesis of 5-aryloyl-4-halo-3-formyl pyridin-2(1H)-ones **2**



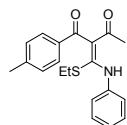
The Vilsmeier reagent was prepared by adding POX₃/DMF (1.5 mmol) into icecold dry DMF (1 mL) under stirring. The mixture was then stirred for 15 min at 0 °C. To the above Vilsmeier reagent was added **1** (0.25 mmol) as a solution in DMF (1 mL). The mixture was heated to 80 °C and stirred for 4.0 h. After cooling to room temperature, the resulting mixture was poured into saturated aqueous NaCl (100 mL), which was extracted with dichloromethane (3×30 mL). The combined organic phase was washed with water, dried over anhydrous MgSO₄, filtered, and evaporated in vacuo. The crude product was purified by silica gel chromatography (petroleum ether/ethyl acetate=4:1, v/v) to give **2** as a white solid.

(4) X-ray crystallographic studies

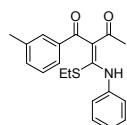
Single crystal X-ray diffraction data of compounds (*Z*)-**1a** were recorded on a Bruker SMART APEX II diffractometer with graphite-monochromated Mo K α radiation ($\lambda = 0.71073 \text{ \AA}$) at 293K. Cell parameters were obtained by global refinement of the positions of all collected reflections. Intensities were corrected for Lorentz and polarization effects and empirical absorption. The structures were solved by direct methods and refined by full-matrix least squares on *F*². All non-hydrogen

atoms were refined with anisotropic temperature parameters. All hydrogen atoms on carbon atoms were generated geometrically. Structure solution and refinement were performed by using the SHELXL-97 program. The X-ray crystallographic files, in CIF format, are available from the Cambridge Crystallographic Data Centre on quoting the deposition numbers CCDC 974611 for (*Z*)-**1a**. Copies of this information may be obtained free of charge from The Director, CCDC, 12 Union Road, Cambridge CB2IEZ, UK (Fax: +44-1223-336033; e-mail: deposit@ccdc.cam.ac.uk or www: <http://www.ccdc.cam.ac.uk>).

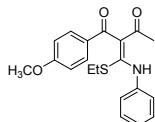
3 Analytical data



(Z)-2-(ethylthio(phenylamino)methylene)-1-p-tolylbutane-1,3-dione ((Z)-1a): yield 85%. Yellowish crystalline solid. M.p.: 58-59 °C. ^1H NMR (600 MHz, CDCl_3) δ : 13.06 (s, 1H), 7.88 (d, $J = 7.9$ Hz, 2 H), 7.40 (d, $J = 8.0$ Hz, 2 H), 7.35 (t, 2 H), 7.27 (d, $J = 7.9$ Hz, 2 H), 7.21 (t, 1 H), 2.43 (s, 3 H), 2.21 (q, 2 H), 2.07 (s, 3 H), 0.88 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ : 195.9, 194.1, 158.6, 143.9, 138.9, 137.1, 129.5 (2C), 129.4 (2 C), 129.2 (2 C), 125.9, 124.3 (2 C), 116.3, 29.0, 28.0, 21.7, 14.0. HRMS Cacl for $\text{C}_{20}\text{H}_{21}\text{NO}_2\text{S}$: $[\text{M}+\text{H}]^+$ 340.1366; Found: 340.1366.

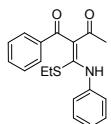


(Z)-2-(ethylthio(phenylamino)methylene)-1-m-tolylbutane-1,3-dione ((Z)-1b): yield 86%. Yellowish liquid. ^1H NMR (600 MHz, CDCl_3) δ 13.07 (s, 1 H), 7.80 (s, 1 H), 7.76 (d, $J = 7.4$ Hz, 1 H), 7.40 (d, $J = 8.1$ Hz, 2 H), 7.37-7.33 (m, 4 H), 7.20 (t, 1 H), 2.42 (s, 3 H), 2.20 (q, 2 H), 2.08 (s, 3 H), 0.86 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 196.3, 194.2, 158.9, 139.8, 138.9, 138.5, 133.8, 129.7, 129.3 (2C), 128.5, 126.7, 126.0, 124.3 (2C), 116.4, 29.0, 28.1, 21.4, 14.0. HRMS Cacl for $\text{C}_{20}\text{H}_{21}\text{NO}_2\text{S}$: $[\text{M}+\text{H}]^+$ 340.1366; Found: 340.1380.



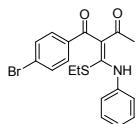
(Z)-2-(ethylthio(phenylamino)methylene)-1-(4-methoxyphenyl)butane-1, 3-dione

((Z)-1c): yield 84%. Yellowish crystalline solid. M.p.: 68-69 °C. ^1H NMR (600 MHz, CDCl_3) δ 13.03 (s, 1 H), 7.97 (d, $J = 8.6$ Hz, 2 H), 7.40 (d, $J = 8.0$ Hz, 2 H), 7.35 (t, 2 H), 7.20 (t, 1 H), 6.96 (d, $J = 8.4$ Hz, 2 H), 3.88 (s, 3 H), 2.23 (q, 2 H), 2.07 (s, 3 H), 0.90 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 195.0, 194.0, 163.7, 158.4, 139.1, 132.6, 131.8 (2 C), 129.3 (2 C), 126.0, 124.3 (2 C), 116.3, 114.0 (2 C), 55.6, 29.0, 28.0, 14.2. HRMS Cacl for $\text{C}_{20}\text{H}_{21}\text{NO}_3\text{S}$: $[\text{M}+\text{H}]^+$ 356.1315; Found: 356.1320.



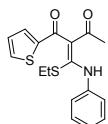
(Z)-2-(ethylthio(phenylamino)methylene)-1-phenylbutane-1, 3-dione ((Z)-1d):

yield 88%. Yellowish crystalline solid. M.p.: 66-67 °C. ^1H NMR (600 MHz, CDCl_3) δ 13.08 (s, 1 H), 7.97 (d, $J = 7.3$ Hz, 2 H), 7.55 (t, 1 H), 7.46 (t, 2 H), 7.39 (d, $J = 7.8$ Hz, 2 H), 7.34 (t, 2 H), 7.20 (t, 1 H), 2.20 (q, 2 H), 2.09 (s, 3 H), 0.84 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 196.1, 194.2, 159.1, 140.0, 139.0, 132.9, 129.3 (4 C), 128.7 (2 C), 126.1, 124.4 (2 C), 116.2, 29.1, 28.1, 14.0. HRMS Cacl for $\text{C}_{19}\text{H}_{19}\text{NO}_2\text{S}$: $[\text{M}+\text{H}]^+$ 326.1210; Found: 326.1214.



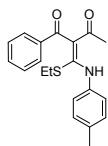
(Z)-1-(4-bromophenyl)-2-(ethylthio(phenylamino)methylene)butane-1, 3-dione

((Z)-1e): yield 90%. Yellowish crystalline solid. M.p.: 37-38 °C. ^1H NMR (600 MHz, CDCl_3) δ 13.11 (s, 1 H), 7.83 (d, $J = 8.5$ Hz, 2 H), 7.60 (d, $J = 8.5$ Hz, 2 H), 7.39-7.34 (m, 4 H), 7.21 (t, 1 H), 2.20 (q, 2 H), 2.09 (s, 3 H), 0.86 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 195.0, 194.0, 159.4, 138.8, 138.7, 132.0 (2 C), 130.8 (2 C), 129.3 (2 C), 128.0, 126.3, 124.4 (2 C), 115.6, 29.0, 28.2, 14.0. HRMS Cacl for $\text{C}_{19}\text{H}_{18}\text{BrNO}_2\text{S}$: $[\text{M}+\text{H}]^+$ 404.0315; Found: 404.0311.



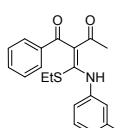
(Z)-2-(ethylthio(phenylamino)methylene)-1-(thiophen-2-yl)butane-1,3-dione

((Z)-1f): yield 86%. Yellowish liquid. ^1H NMR (600 MHz, CDCl_3) δ 13.02 (s, 1 H), 7.68 (t, 2 H), 7.39 (d, $J = 7.5$ Hz, 2 H), 7.35 (t, 2 H), 7.21 (t, 1 H), 7.14 (t, 1 H), 2.50 (q, 2 H), 2.14 (s, 3 H), 0.92 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 193.5, 188.2, 158.8, 147.2, 138.7, 134.4, 133.4, 129.2 (2 C), 128.2, 126.1, 124.3 (2 C), 120.1, 28.7, 28.2, 14.0. HRMS Cacl for $\text{C}_{17}\text{H}_{17}\text{NO}_2\text{S}_2$: $[\text{M}+\text{H}]^+$ 332.0774; Found: 332.0793.



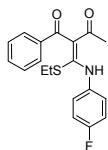
(Z)-2-((p-toluidino)(ethylthio)methylene)-1-phenylbutane-1,3-dione ((Z)-1g)

yield 83%. Yellowish crystalline solid. M.p.: 61-62 °C. ^1H NMR (600 MHz, CDCl_3) δ (Z)-1g: 13.09 (s, 1 H), 7.96 (d, $J = 7.6$ Hz, 2 H), 7.56 (t, 1 H), 7.47 (t, 2 H), 7.27 (d, $J = 7.4$ Hz, 2 H), 7.15 (d, $J = 7.9$ Hz, 2 H), 2.34 (s, 3 H), 2.20 (q, 2 H), 2.08 (s, 3 H), 0.84 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 196.3, 194.0, 159.6, 140.0, 136.2, 136.0, 132.9, 129.9 (2 C), 129.3 (2 C), 128.6 (2 C), 124.3 (2 C), 115.8, 29.0, 28.1, 21.1, 14.0. (HRMS Cacl for $\text{C}_{20}\text{H}_{21}\text{NO}_2\text{S}$: $[\text{M}+\text{H}]^+$ 340.1366; Found: 340.1370.



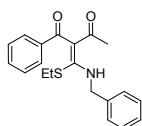
(Z)-2-((m-toluidino)(ethylthio)methylene)-1-phenylbutane-1,3-dione ((Z)-1h)

yield 85%. Yellowish liquid. ^1H NMR (600 MHz, CDCl_3) δ 13.07 (s, 1 H), 7.97 (d, $J = 7.4$ Hz, 2 H), 7.55 (t, 1 H), 7.46 (t, 2 H), 7.21 (t, 2 H), 7.19 (s, 1 H), 7.01 (d, $J = 7.5$ Hz, 1 H), 2.34 (s, 3 H), 2.22 (q, 2 H), 2.08 (s, 3 H), 0.85 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 196.2, 194.1, 159.3, 139.9, 139.3, 138.8, 132.9, 129.3 (2 C), 129.1, 128.6 (2 C), 126.9, 124.9, 121.3, 116.0, 29.0, 28.1, 21.4, 14.0. HRMS Cacl for $\text{C}_{20}\text{H}_{21}\text{NO}_2\text{S}$: $[\text{M}+\text{H}]^+$ 340.1366; Found: 340.1385.



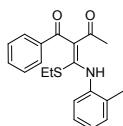
(Z)-2-(ethylthio(4-fluorophenylamino)methylene)-1-phenylbutane-1, 3-dione ((Z)-1i)

((Z)-1i): yield 75%. Yellowish liquid. ^1H NMR (600 MHz, CDCl_3) δ 13.01 (s, 1 H), 7.96 (d, $J = 8.3$ Hz, 2 H), 7.56 (t, 1 H), 7.47 (t, 2 H), 7.36-7.34 (m, 2 H), 7.05 (t, 2 H), 2.20 (q, 2 H), 2.09 (s, 3 H), 0.85 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 196.1, 194.4, 161.6, 160.0, 159.2, 139.9, 133.1, 129.3 (2C), 128.7 (2 C), 126.4, 126.3, 116.4, 116.3, 116.1, 29.1, 28.2, 14.0. HRMS Cacl for $\text{C}_{19}\text{H}_{18}\text{FNO}_2\text{S}$: ([M+H] $^+$) 344.1115; Found: 344.1119.



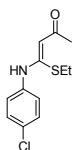
(Z)-2-((benzylamino)(ethylthio)methylene)-1-phenylbutane-1, 3-dione ((Z)-1j)

((Z)-1j): yield 87%. Yellowish crystalline solid. M.p.: 61-62 °C. ^1H NMR (600 MHz, CDCl_3) δ 12.16 (s, 1 H), 7.90 (d, $J = 7.9$ Hz, 2 H), 7.54 (t, 1 H), 7.44 (t, 2 H), 7.37 (t, 2 H), 7.30 (d, $J = 7.2$ Hz, 3 H), 4.77 (d, $J = 5.8$ Hz, 2 H), 2.62 (q, 2 H), 2.09 (s, 3 H), 1.00 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 196.2, 193.8, 162.6, 140.3, 137.3, 132.5, 129.1 (2 C), 128.8 (2 C), 128.4 (2 C), 127.6, 126.9 (2 C), 114.35, 48.9, 30.4, 28.6, 14.2. HRMS Cacl for $\text{C}_{20}\text{H}_{21}\text{NO}_2\text{S}$: ([M+H] $^+$) 340.1366; Found: 340.1378.

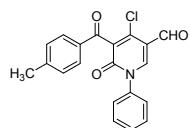


(Z)-2-((o-toluidino)(ethylthio)methylene)-1-phenylbutane-1, 3-dione ((Z)-1k)

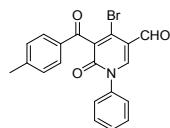
((Z)-1k): yield 87%. Yellowish liquid. ^1H NMR (600 MHz, CDCl_3) δ 13.08 (s, 1 H), 7.97 (d, $J = 7.4$ Hz, 2 H), 7.55 (t, 1 H), 7.46 (t, 3 H), 7.22 (d, $J = 7.3$ Hz, 1 H), 7.19-7.13 (m, 2 H), 2.37 (s, 3 H), 2.17 (q, 2 H), 2.11 (s, 3 H), 0.83 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 196.2, 194.1, 160.7, 140.1, 137.6, 132.9, 132.8, 130.9, 129.3 (2 C), 128.6 (2 C), 126.6, 126.5, 125.5, 115.8, 28.9, 28.1, 18.2, 14.0. HRMS Cacl for $\text{C}_{20}\text{H}_{21}\text{NO}_2\text{S}$: ([M+H] $^+$) 340.1366; Found: 340.1382.



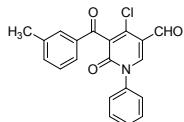
(Z)-4-(4-chlorophenylamino)-4-(ethylthio)but-3-en-2-one 1l: yield 76%. White crystalline solid, M.p.: 68-69 °C. ^1H NMR (600 MHz, CDCl_3) δ 12.93 (s, 1 H), 7.30 (d, J = 8.8 Hz, 2 H), 7.19 (d, J = 8.6 Hz, 2 H), 5.26 (s, 1 H), 2.84 (q, 2 H), 2.13 (s, 3 H), 1.32 (t, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 193.4, 164.5, 137.1, 131.7, 129.2 (2 C), 126.6 (2 C), 93.1, 29.4, 26.1, 13.4. HRMS Cacl for $\text{C}_{12}\text{H}_{14}\text{ClNO}_2$: $[\text{M}+\text{H}]^+$ 256.0557; Found: 256.0553.



4-chloro-5-(4-methylbenzoyl)-6-oxo-1-phenyl-1,6-dihydropyridine-3-carbaldehyde 2a: yield 85%. White solid. M.p.: 160-161 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.14 (s, 1 H), 8.30 (s, 1 H), 7.85 (d, J = 8.2 Hz, 2 H), 7.53-7.48 (m, 3 H), 7.40 (d, J = 8.5 Hz, 2 H), 7.29 (d, J = 8.0 Hz, 2 H), 2.43 (s, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 190.3, 185.6, 158.6, 145.8, 143.8, 143.2, 138.6, 133.1, 130.0, 129.9 (3 C), 129.8 (2 C), 129.7 (2 C), 126.3 (2 C), 114.5, 22.0. HRMS Cacl for $\text{C}_{20}\text{H}_{14}\text{ClNO}_2$: $[\text{M}+\text{H}]^+$ 352.0735; Found: 352.0739.

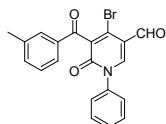


4-bromo-5-(4-methylbenzoyl)-6-oxo-1-phenyl-1,6-dihydropyridine-3-carbaldehyde 2b: yield 81%. White solid. M.p.: 173-174 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.10 (s, 1 H), 8.26 (s, 1 H), 7.85 (d, J = 8.2 Hz, 2 H), 7.53-7.48 (m, 3 H), 7.40 (d, J = 8.5 Hz, 2 H), 7.29 (d, J = 8.0 Hz, 2 H), 2.43 (s, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 191.2, 187.5, 158.2, 145.7, 143.3 (2 C), 138.6, 133.0, 132.8, 132.6, 129.8 (2 C), 129.7 (2 C), 129.6 (2 C), 126.1 (2 C), 115.0, 22.0. HRMS Cacl for $\text{C}_{20}\text{H}_{14}\text{BrNO}_2$: $[\text{M}+\text{H}]^+$ 396.0230; Found: 396.0218.



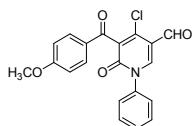
**4-chloro-5-(3-methylbenzoyl)-6-oxo-1-phenyl-1,6-dihdropyridine-3-carbaldehyde
e 2c:**

yield 82%. Yellowish solid. M.p.: 171-172 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.18 (s, 1 H), 8.34 (s, 1H), 7.80 (s, 1 H), 7.55 (d, *J* = 7.7 Hz, 1 H), 7.57-7.52 (m, 3 H), 7.47 (d, *J* = 7.4 Hz, 1 H), 7.45-7.40 (m, 3 H), 2.45 (s, 3 H). ¹³C NMR (150 MHz, CDCl₃) δ 190.9, 185.5, 158.5, 143.7, 143.2, 139.0, 138.6, 135.5, 135.4, 129.9, 129.7 (4 C), 128.9, 126.8, 126.2 (2 C), 114.5, 21.4. HRMS Cacl for C₂₀H₁₄ClNO₃: ([M+H]⁺) 352.0735; Found: 352.0730.



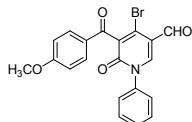
**4-bromo-5-(3-methylbenzoyl)-6-oxo-1-phenyl-1,6-dihdropyridine-3-carbaldehyde
yde 2d:**

yield 80%. Yellowish solid. M.p.: 163-164 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.11 (s, 1 H), 8.27 (s, 1H), 7.78 (s, 1 H), 7.74 (d, *J* = 7.7 Hz, 1 H), 7.53-7.49 (m, 3 H), 7.44 (d, *J* = 7.5 Hz, 1 H), 7.42-7.38 (m, 3 H), 2.42 (s, 3 H). ¹³C NMR (150 MHz, CDCl₃) δ 191.8, 187.5, 158.2, 143.4, 139.0, 138.6, 135.4, 135.0, 133.1, 132.8, 129.8, 129.7 (3 C), 129.0, 126.9, 126.1 (2 C), 115.1, 21.4. HRMS Cacl for C₂₀H₁₄BrNO₃: ([M+H]⁺) 396.0230; Found: 396.0231.

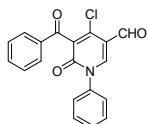


**4-chloro-5-(4-methoxybenzoyl)-6-oxo-1-phenyl-1,6-dihdropyridine-3-carbaldehyde
yde 2e:**

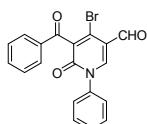
yield 85%. Yellowish solid. M.p.: 140-141 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.13 (s, 1 H), 8.29 (s, 1 H), 7.92 (d, *J* = 8.8 Hz, 2 H), 7.53-7.48 (m, 3 H), 7.39 (d, *J* = 7.3 Hz, 2 H), 6.96 (d, *J* = 8.8 Hz, 2 H), 3.87 (s, 3 H). ¹³C NMR (150 MHz, CDCl₃) δ 189.3, 185.8, 165.0, 158.8, 143.9, 143.3, 138.9, 132.2 (2 C), 130.3, 130.1, 130.0 (2 C), 128.8, 126.5 (2 C), 114.8, 114.7 (2 C), 56.0. HRMS Cacl for C₂₀H₁₄ClNO₄: ([M+H]⁺) 368.0685; Found: 368.0680.



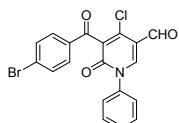
4-bromo-5-(4-methoxybenzoyl)-6-oxo-1-phenyl-1, 6-dihydropyridine-3-carbaldehyde 2f: yield 83%. Yellowish solid. M.p.: 96-97 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.10 (s, 1 H), 8.26 (s, 1 H), 7.92 (d, *J* = 8.8 Hz, 2 H), 7.53-7.48 (m, 3 H), 7.39 (d, *J* = 7.4 Hz, 2 H), 6.96 (d, *J* = 8.8 Hz, 2 H), 3.88 (s, 3 H). ¹³C NMR (150 MHz, CDCl₃) δ 190.0, 187.5, 164.7, 158.2, 143.3, 138.6, 133.0, 132.8, 132.0 (2 C), 129.8, 129.7 (2 C), 128.1, 126.1 (2 C), 115.0, 114.8 (2 C), 55.6. HRMS Cacl for C₂₀H₁₄BrNO₄: ([M+H]⁺) 412.0180; Found: 412.0182.



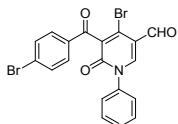
5-benzoyl-4-chloro-6-oxo-1-phenyl-1, 6-dihydropyridine-3-carbaldehyde 2g: yield 84%. Yellowish solid. M.p.: 138-139 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.14 (s, 1 H), 8.31 (s, 1 H), 7.95 (d, *J* = 7.5 Hz, 2 H), 7.63 (t, 1 H), 7.52-7.49 (m, 5 H), 7.39 (d, *J* = 7.3 Hz, 2 H). ¹³C NMR (150 MHz, CDCl₃) δ 190.7, 185.4, 158.5, 143.8, 143.3, 138.5, 135.4, 134.5, 129.9, 129.8 (2 C), 129.7, 129.4 (2 C), 129.1 (2 C), 126.2 (2 C), 114.5. HRMS Cacl for C₁₉H₁₂ClNO₃: ([M+H]⁺) 338.0579; Found: 338.0582.



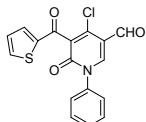
5-benzoyl-4-bromo-6-oxo-1-phenyl-1, 6-dihydropyridine-3-carbaldehyde 2h: yield 84%. Yellowish solid. M.p.: 148-149 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.10 (s, 1 H), 8.27 (s, 1 H), 7.96 (d, *J* = 7.4 Hz, 2 H), 7.63 (t, 1 H), 7.53-7.49 (m, 5 H), 7.39 (d, *J* = 7.4 Hz, 2 H). ¹³C NMR (150 MHz, CDCl₃) δ 191.6, 187.4, 158.2, 143.5, 138.5, 135.0, 134.5, 133.2, 132.6, 129.9, 129.7 (2 C), 129.5 (2 C), 129.1 (2 C), 126.1 (2 C), 115.0. HRMS Cacl for C₁₉H₁₂BrNO₃: ([M+H]⁺) 382.0074; Found: 382.0081.



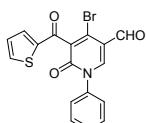
5-(4-bromobenzoyl)-4-chloro-6-oxo-1-phenyl-1,6-dihdropyridine-3-carbaldehyd e 2i: yield 79%. Yellowish solid. M.p.: 106-107 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.13 (s, 1 H), 8.31 (s, 1 H), 7.80 (d, *J* = 8.5 Hz, 2 H), 7.64 (d, *J* = 8.5 Hz, 2 H), 7.53-7.49 (m, 3 H), 7.39 (d, *J* = 8.5 Hz, 2 H). ¹³C NMR (150 MHz, CDCl₃) δ 189.5, 185.0, 158.2, 143.8, 143.4, 138.2, 134.0, 132.2 (2 C), 130.6 (2 C), 129.8, 129.7 (2 C), 129.5, 128.8, 125.9 (2 C), 114.2. HRMS Cacl for C₁₉H₁₁BrClNO₃: ([M+H]⁺) 415.9684; Found: 415.9681.



4-bromo-5-(4-bromobenzoyl)-6-oxo-1-phenyl-1,6-dihdropyridine-3-carbaldehyd -e 2j: yield 77%. Yellowish solid. M.p.: 92-93 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.10 (s, 1 H), 8.28 (s, 1 H), 7.81 (d, *J* = 8.5 Hz, 2 H), 7.65 (d, *J* = 8.5 Hz, 2 H), 7.53-7.50 (m, 3 H), 7.39 (d, *J* = 8.3 Hz, 2 H). ¹³C NMR (150 MHz, CDCl₃) δ 190.7, 187.2, 158.2, 143.7, 138.6 133.8, 133.5, 132.5 (2 C), 130.8 (2 C), 130.0, 129.9, 129.8 (2 C), 126.1 (2 C), 115.1. HRMS Cacl for C₁₉H₁₁Br₂NO₃: ([M+H]⁺) 459.9179; Found: 459.9185.

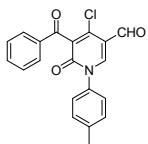


4-chloro-6-oxo-1-phenyl-5-(thiophene-2-carbonyl)-1,6-dihdropyridine-3-carbaldehyde 2k: yield 80%. White solid. M.p.: 115-117 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.15 (s, 1 H), 8.31 (s, 1 H), 7.78 (d, *J* = 4.9 Hz, 1 H), 7.69 (d, *J* = 3.8 Hz, 1 H), 7.54-7.50 (m, 3 H), 7.40 (d, *J* = 8.4 Hz, 2 H), 7.16 (d, *J* = 8.7 Hz, 1 H). ¹³C NMR (150 MHz, CDCl₃) δ 185.4, 182.3, 158.4, 144.0, 143.6, 142.5, 138.5, 136.1, 135.0, 129.9, 129.7 (2 C), 129.5, 128.6, 126.2 (2 C), 114.4. HRMS Cacl for C₁₇H₁₀ClNO₃S: ([M+H]⁺) 344.0143; Found: 344.0143.

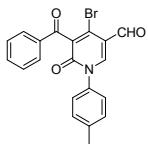


4-bromo-6-oxo-1-phenyl-5-(thiophene-2-carbonyl)-1,6-dihdropyridine-3-carbal

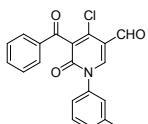
dehyde 2l: yield 79%. White solid. M.p.: 136-137 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.13 (s, 1 H), 8.30 (s, 1 H), 7.80 (d, J = 4.8 Hz, 1 H), 7.69 (d, J = 3.8 Hz, 1 H), 7.56-7.53 (m, 3 H), 7.44 (d, J = 8.3 Hz, 2 H), 7.18 (d, J = 8.7 Hz, 1 H). ^{13}C NMR (150 MHz, CDCl_3) δ 187.4, 183.4, 158.1, 143.6, 142.1, 138.5, 136.1, 135.0, 133.5, 132.4, 129.9, 129.7 (2 C), 128.6, 126.1 (2 C), 115.0. HRMS Cacl for $\text{C}_{17}\text{H}_{10}\text{BrNO}_3\text{S}$: ([M+H] $^+$) 387.9638; Found: 387.9640.



5-benzoyl-4-chloro-6-oxo-1-p-tolyl-1,6-dihdropyridine-3-carbaldehyde 2m: yield 85%. White solid. M.p.: 72-73 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.14 (s, 1 H), 8.30 (s, 1 H), 7.94 (d, J = 7.2 Hz, 2 H), 7.63 (t, 1 H), 7.50 (t, 2 H), 7.30 (d, J = 8.4 Hz, 2 H), 7.27 (d, J = 8.4 Hz, 2 H), 2.41 (s, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 190.7, 185.5, 158.6, 143.9, 143.2, 140.1, 136.0, 135.4, 134.5, 130.2 (2 C), 129.4 (2 C), 129.0 (2 C), 125.8 (2 C), 114.3, 21.2. HRMS Cacl for $\text{C}_{20}\text{H}_{14}\text{ClNO}_3$: ([M+H] $^+$) 352.0735; Found: 352.0736.

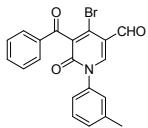


5-benzoyl-4-bromo-6-oxo-1-p-tolyl-1,6-dihdropyridine-3-carbaldehyde 2n: yield 81%. White solid. M.p.: 87-88 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.12 (s, 1 H), 8.29 (s, 1 H), 7.98 (d, J = 7.8 Hz, 2 H), 7.65 (t, 1 H), 7.52 (t, 2 H), 7.34-7.29 (m, 4 H), 2.44 (s, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 191.6, 187.4, 158.2, 143.5, 140.1, 135.9, 134.9, 134.4, 133.0, 132.3, 130.2 (2 C), 129.4 (2 C), 129.0 (2 C), 125.7 (2 C), 114.9, 21.7. HRMS Cacl for $\text{C}_{20}\text{H}_{14}\text{BrNO}_3$: ([M+H] $^+$) 396.0230; Found: 396.0240.



5-benzoyl-4-chloro-6-oxo-1-m-tolyl-1,6-dihdropyridine-3-carbaldehyde 2o: yield 82%. White solid. M.p.: 124-125 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.14 (s, 1 H), 8.30 (s, 1 H), 7.94 (d, J = 8.4 Hz, 2 H), 7.62 (t, 1 H), 7.50 (t, 2 H), 7.39 (t, 1 H), 7.28

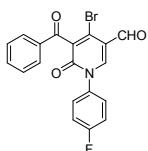
(d, $J = 7.7$ Hz, 1 H), 7.19 (t, 2 H), 2.41 (s, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 190.7, 185.5, 158.6, 143.9, 143.3, 140.0, 138.4, 135.4, 134.5, 130.6, 129.6, 129.5, 129.4 (2 C), 129.1 (2 C), 126.7, 123.1, 114.3, 21.3. HRMS Cacl for $\text{C}_{20}\text{H}_{14}\text{ClNO}_3$: ($[\text{M}+\text{H}]^+$) 352.0735; Found: 352.0737.



5-benzoyl-4-bromo-6-oxo-1-m-tolyl-1,6-dihdropyridine-3-carbaldehyde 2p:
yield 83%. White solid. M.p.: 138-139 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.10 (s, 1 H), 8.26 (s, 1 H), 7.97 (d, $J = 8.1$ Hz, 2 H), 7.63 (t, 1 H), 7.50 (t, 2 H), 7.40 (t, 1 H), 7.28 (d, $J = 7.6$ Hz, 1 H), 7.20 (t, 2 H), 2.41 (s, 3 H). ^{13}C NMR (150 MHz, CDCl_3) δ 191.6, 187.4, 158.3, 143.5, 140.0, 138.4, 135.0, 134.4, 133.1, 132.5, 130.6, 129.5, 129.4 (2 C), 129.0 (2 C), 126.2, 123.1, 114.9, 21.3. HRMS Cacl for $\text{C}_{20}\text{H}_{14}\text{BrNO}_3$: ($[\text{M}+\text{H}]^+$) 396.0230; Found: 396.0231.

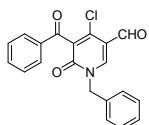


5-benzoyl-4-chloro-1-(4-fluorophenyl)-6-oxo-1,6-dihdropyridine-3-carbaldehyde 2q: yield 79%. White solid. M.p.: 148-149 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.14 (s, 1 H), 8.27 (s, 1 H), 7.94 (d, $J = 8.0$ Hz, 2 H), 7.63 (t, 1 H), 7.50 (t, 2 H), 7.41-7.38 (m, 2 H), 7.20 (t, 2 H). ^{13}C NMR (150 MHz, CDCl_3) δ 190.5, 185.4, 163.8, 162.1, 158.5, 143.6, 143.4, 135.3, 134.6, 134.4, 129.4, 129.1 (2 C), 128.2 (2 C), 128.1, 116.9, 116.7, 114.6. HRMS Cacl for $\text{C}_{19}\text{H}_{11}\text{ClFNO}_3$: ($[\text{M}+\text{H}]^+$) 356.0485; Found: 356.0484.

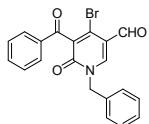


5-benzoyl-4-bromo-1-(4-fluorophenyl)-6-oxo-1,6-dihdropyridine-3-carbaldehyde 2r: yield 78%. White solid. M.p.: 128-129 °C. ^1H NMR (600 MHz, CDCl_3) δ 10.10 (s, 1 H), 8.23 (s, 1 H), 7.95 (d, $J = 8.0$ Hz, 2 H), 7.63 (t, 1 H), 7.50 (t, 2 H), 7.42-7.38 (m, 2 H), 7.20 (t, 2 H). ^{13}C NMR (150 MHz, CDCl_3) δ 191.4, 187.3, 164.2, 161.2, 158.2, 143.2, 134.9, 134.5, 133.3, 132.8, 129.5 (2 C), 129.1 (2 C), 128.2, 128.1,

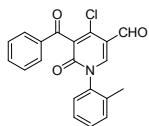
116.9, 116.7, 115.2. HRMS Cacl for $C_{19}H_{11}BrFNO_3$: ($[M+H]^+$) 399.9979; Found: 399.9981.



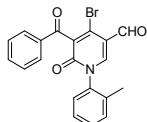
5-benzoyl-1-benzyl-4-chloro-6-oxo-1,6-dihdropyridine-3-carbaldehyde 2s: yield 85%. White solid. M.p.: 129-130 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.05 (s, 1 H), 8.23 (s, 1 H), 7.90 (d, *J* = 8.0 Hz, 2 H), 7.63 (t, 1 H), 7.49 (t, 2 H), 7.39-7.37 (m, 3 H), 7.36-7.34 (m, 2 H), 5.18 (s, 2 H). ¹³C NMR (150 MHz, CDCl₃) δ 190.7, 185.3, 159.0, 143.1, 143.0, 135.4, 134.5, 134.1, 129.4 (4 C), 129.2, 129.1 (2 C), 129.0, 128.9 (2 C), 114.5. 53.2. HRMS Cacl for $C_{20}H_{14}ClNO_3$: ($[M+H]^+$) 352.0735; Found: 352.0742.



5-benzoyl-1-benzyl-4-bromo-6-oxo-1,6-dihdropyridine-3-carbaldehyde 2t: yield 85%. White solid. M.p.: 137-138 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.00 (s, 1 H), 8.20 (s, 1 H), 7.90 (d, *J* = 7.3 Hz, 2 H), 7.63 (t, 1 H), 7.49 (t, 2 H), 7.39-7.35 (m, 5 H), 5.16 (s, 2 H). ¹³C NMR (150 MHz, CDCl₃) δ 191.7, 187.3, 158.7, 142.7, 135.0, 134.5, 134.1, 132.9, 131.8, 129.5 (2 C), 129.4 (2 C), 129.2, 129.1 (2 C), 128.8 (2 C), 115.1. 53.3. HRMS Cacl for $C_{20}H_{14}BrNO_3$: ($[M+H]^+$) 396.0230; Found: 396.0237.

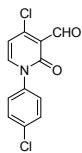


5-benzoyl-4-chloro-6-oxo-1-o-tolyl-1,6-dihdropyridine-3-carbaldehyde 2u: yield 77%. White solid. M.p.: 66-67 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.14 (s, 1 H), 8.19 (s, 1 H), 7.93 (d, *J* = 8.3 Hz, 2 H), 7.63 (t, 1 H), 7.50 (t, 2 H), 7.39 (t, 1 H), 7.35 (d, *J* = 7.5 Hz, 2 H), 7.20 (d, *J* = 7.9 Hz, 1 H), 2.19 (s, 3 H). ¹³C NMR (150 MHz, CDCl₃) δ 190.5, 185.4, 158.2, 144.1, 143.5, 137.9, 135.5, 134.7, 134.5, 131.5, 130.3, 129.7, 129.3 (2 C), 129.1 (2 C), 127.6, 126.8, 114.4. 17.7. HRMS Cacl for $C_{20}H_{14}ClNO_3$: ($[M+H]^+$) 352.0735; Found: 352.0741.



5-benzoyl-4-bromo-6-oxo-1-o-tolyl-1,6-dihdropyridine-3-carbaldehyde 2v:

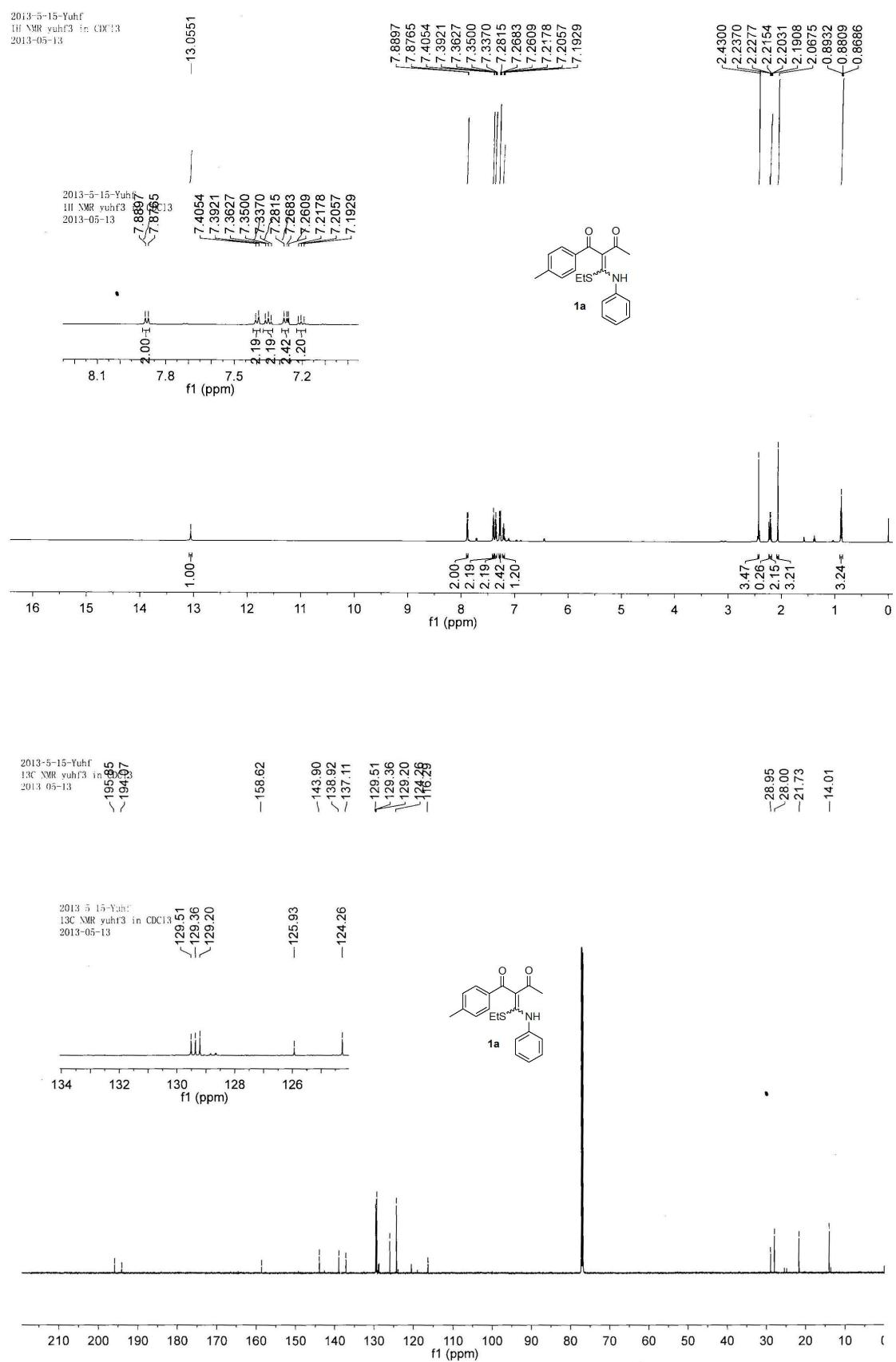
White solid. M.p.: 73-754 °C. ¹H NMR (600 MHz, CDCl₃) δ 10.10 (s, 1 H), 8.16 (s, 1 H), 7.93 (d, *J* = 8.3 Hz, 2 H), 7.63 (t, 1 H), 7.50 (t, 2 H), 7.39 (t, 1 H), 7.35 (d, *J* = 7.5 Hz, 2 H), 7.20 (d, *J* = 7.8 Hz, 1 H), 2.19 (s, 3 H). ¹³C NMR (150 MHz, CDCl₃) δ 191.5, 187.4, 158.8, 143.8, 137.9, 135.1, 134.7, 134.4, 133.3, 132.5, 131.5, 130.3, 129.4 (2 C), 129.1 (2 C), 127.6, 126.8, 115.0. HRMS Cacl for C₂₀H₁₄BrNO₃: ([M+H]⁺) 396.0230; Found: 396.0235.



4-chloro-1-(4-chlorophenyl)-2-oxo-1,2-dihdropyridine-3-carbaldehyde 2w:

yellowish solid. M.p.: 166-167 °C. ¹H NMR (600 MHz, CDCl₃) δ: 10.27 (s, 1 H), 7.59 (d, *J* = 7.1 Hz, 1 H), 7.53 (d, *J* = 8.5 Hz, 2 H), 7.27 (d, *J* = 8.4 Hz, 2 H), 6.76 (d, *J* = 7.1 Hz, 1 H). ¹³C NMR (150 MHz, CDCl₃) δ 187.4, 181.9, 144.4, 141.9, 140.7, 136.3, 135.9, 130.2 (2 C), 128.1 (2 C), 115.2. HRMS Cacl for C₁₂H₇Cl₂NO₂: ([M+H]⁺) 267.9927; Found: 267.9928.

4. Copies of NMR spectra for new compounds



yul-20
1H NMR yul in CDCl₃
2013-07-29

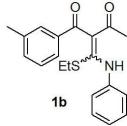
-13.0649

yul-20
1H NMR yul in

CDCl₃
2013-07-29

8.1 7.9 7.7 7.5 7.3 7.1

f1 (ppm)



2.4179
2.3407
2.2144
2.2021
2.1898
2.1775
2.0841
2.0865
0.8562
0.8439

14.5 13.5 12.5 11.5 10.5 9.5 8.5 7.5 6.5 5.5

f1 (ppm)

yul-20
13C NMR yul in CDCl₃
2013-07-29

~196.32

~194.17

-158.86

~133.81

139.76

~138.92

~138.07

~138.33

139.46

~129.71

~129.27

~128.53

~126.73

~126.04

~124.28

128.55

~126.73

~126.04

~124.28

116.40

77.37
77.16
76.95

3.78
0.38
2.37
3.48

29.02
28.05
~21.41
~13.99

3.52

yul-20
13C NMR yul in CDCl₃
2013-07-29

~133.81

~132.71

~129.71

~128.53

~126.73

~126.04

~124.28

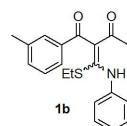
128.55

~126.73

~126.04

~124.28

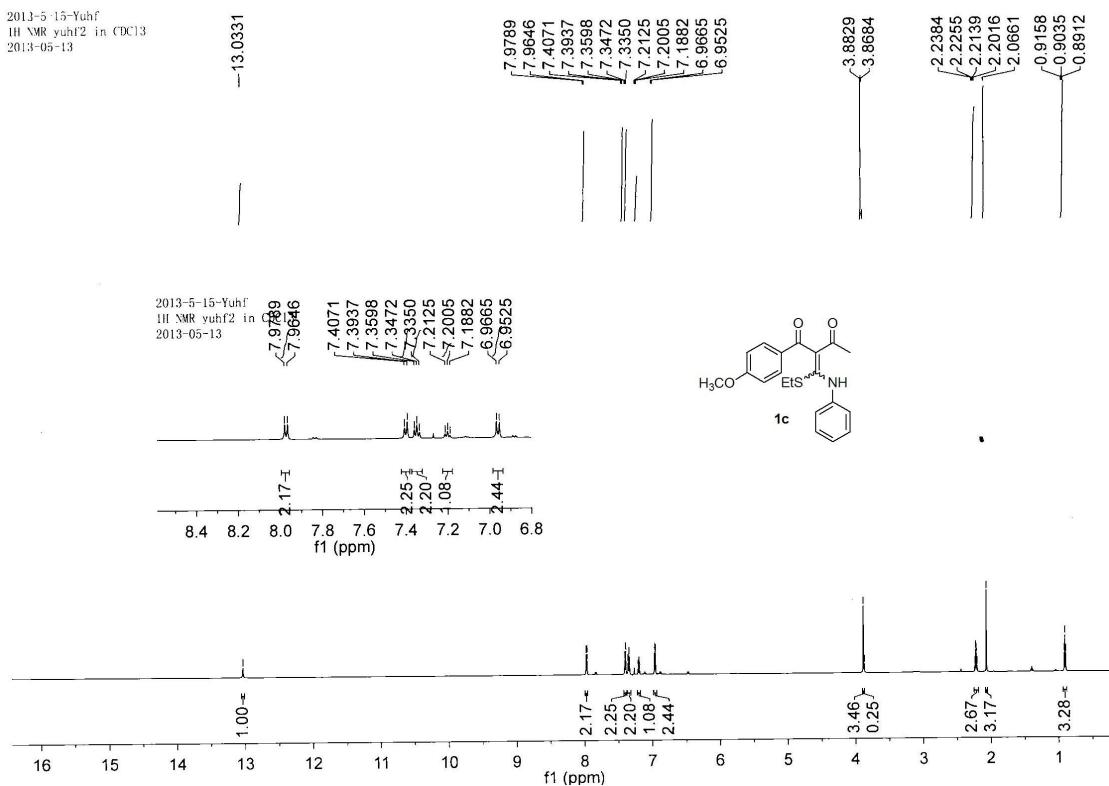
116.40



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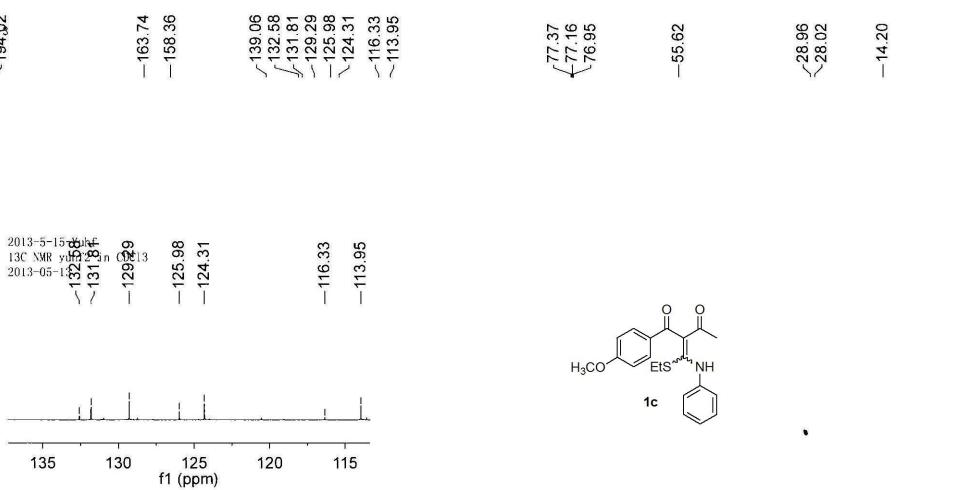
2013-5-15-Yuhf
1H NMR yuhf2 in CDCl3
2013-05-13

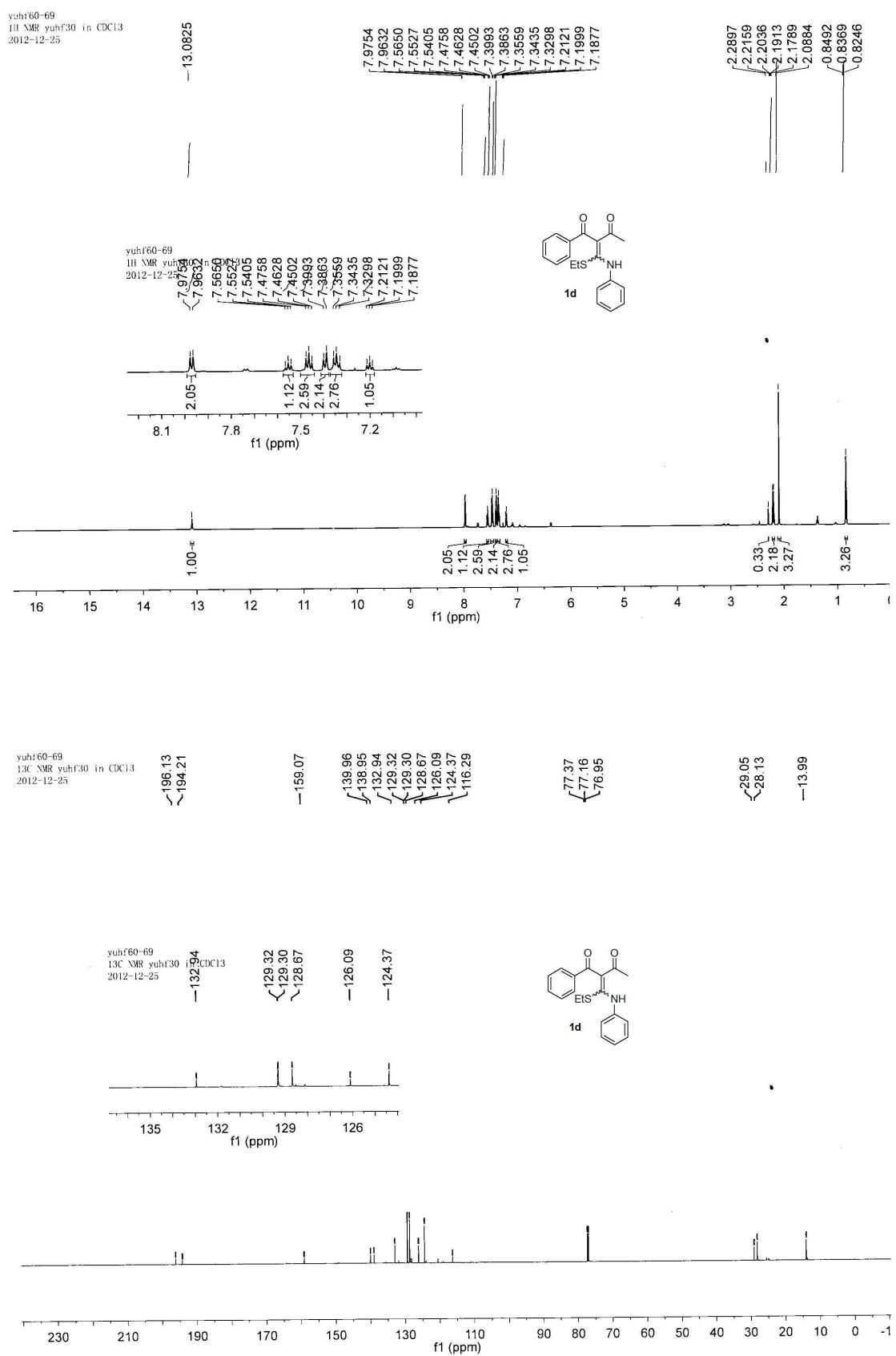
-13.0331

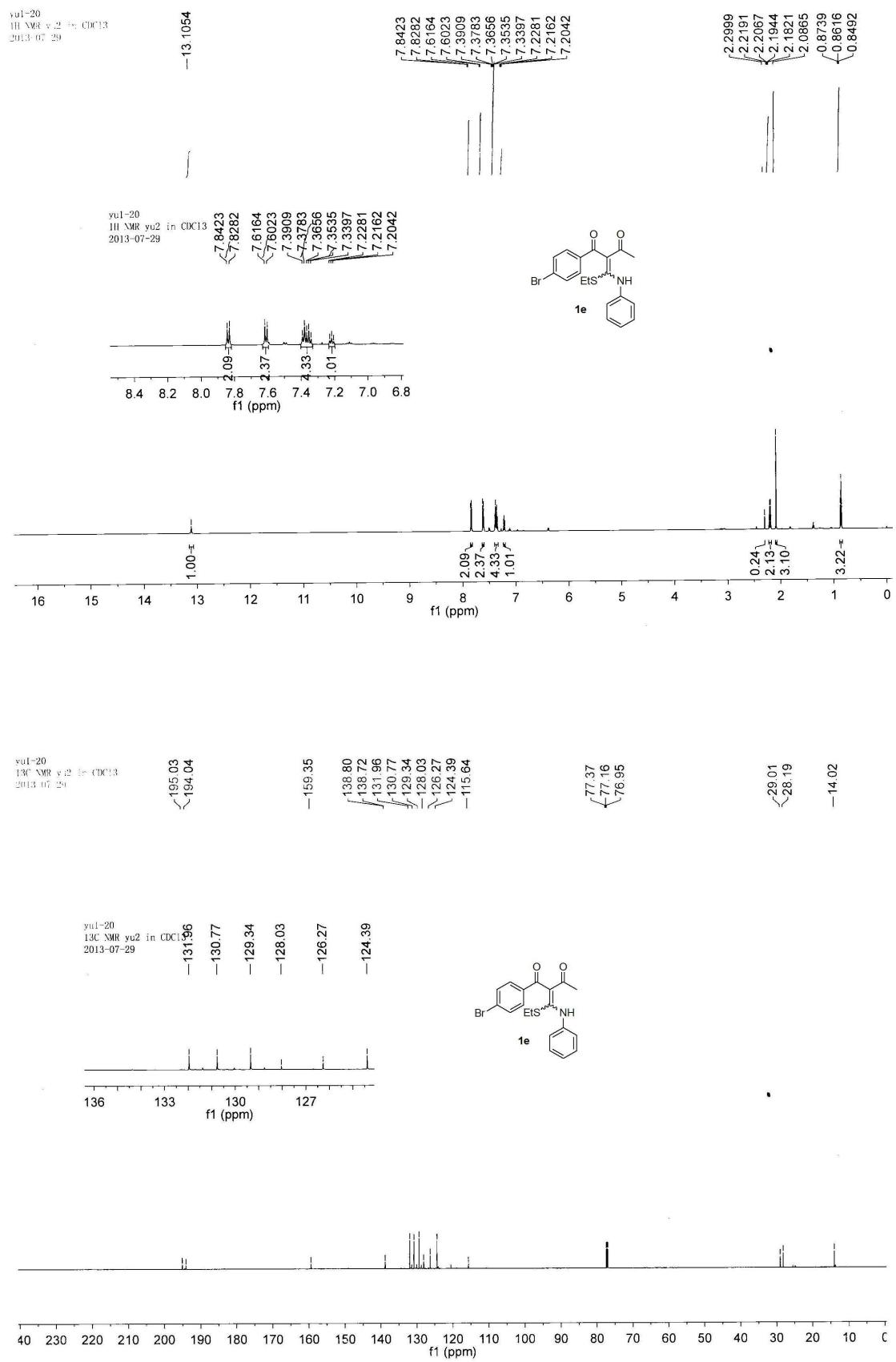


2013-5-15-Yuhf
13C NMR yuhf2 in CDCl3
2013-05-13

194.97
194.92
163.74
158.36
139.06
132.58
131.81
129.29
125.98
124.31
116.33
113.95

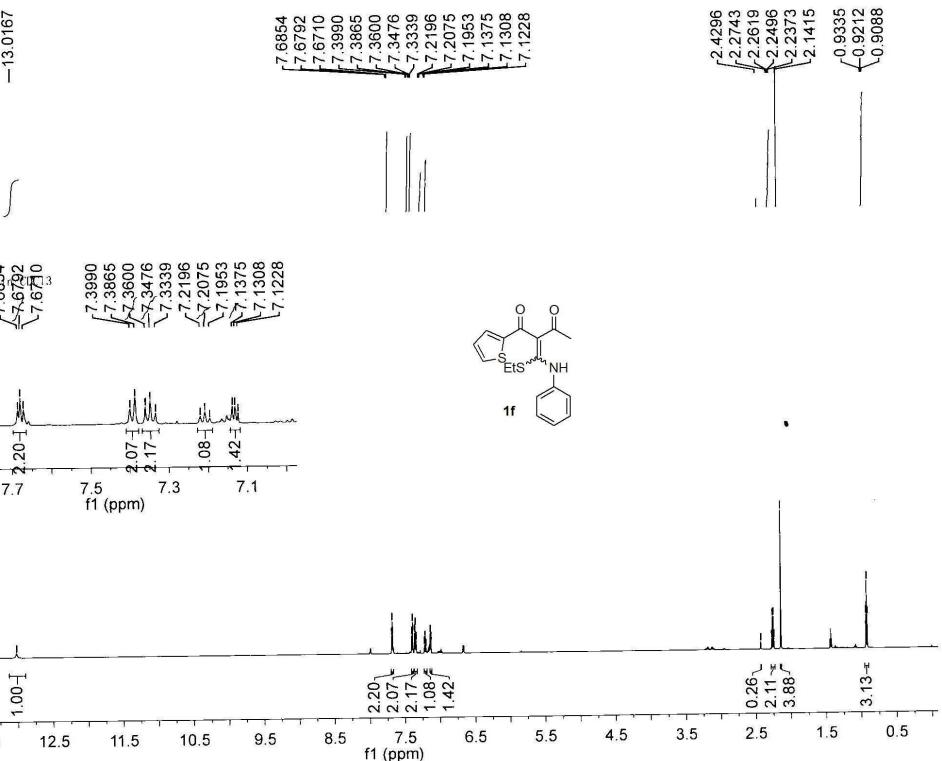






yu1-20
1H NMR yu3 in CDCl₃
2013-07-29

-13.0167



yu1-20
13C NMR yu3 in CDCl₃
2013-07-29

-193.46

-188.22

-158.75

-147.15

-138.72

-134.44

-133.41

-129.23

-128.20

-126.10

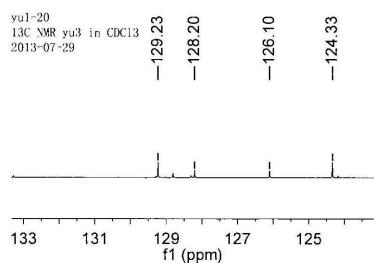
-124.33

-120.13

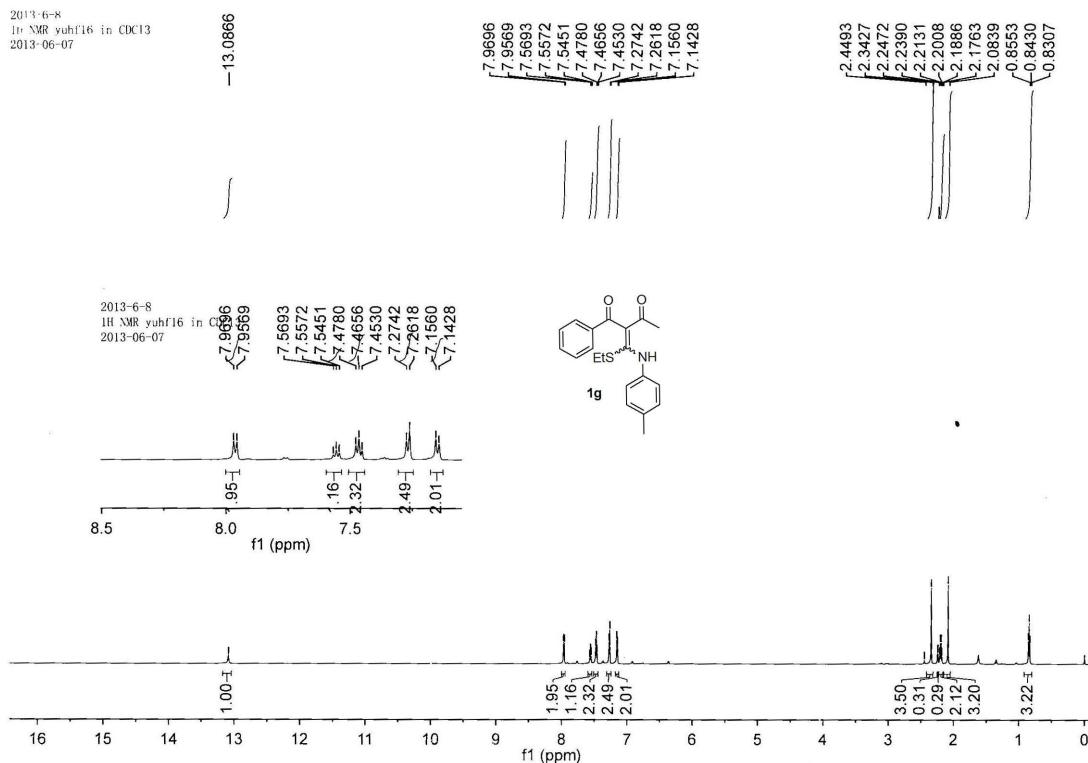
77.37
77.16
76.95

28.67
28.17

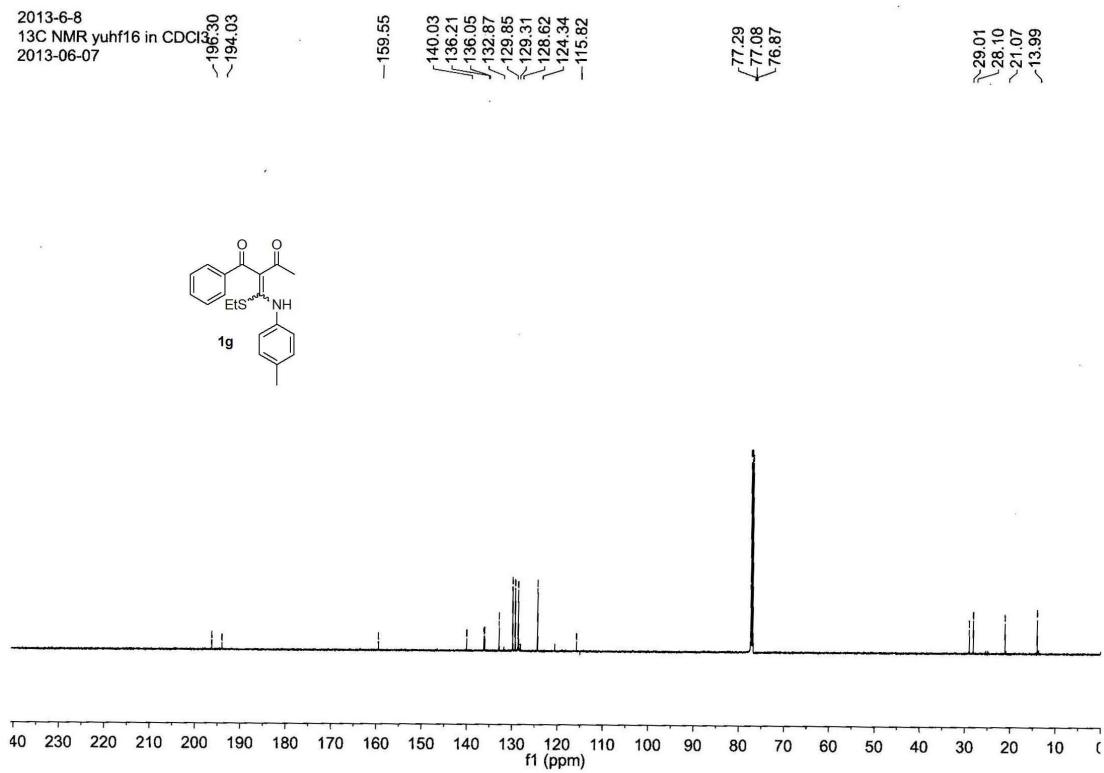
14.00

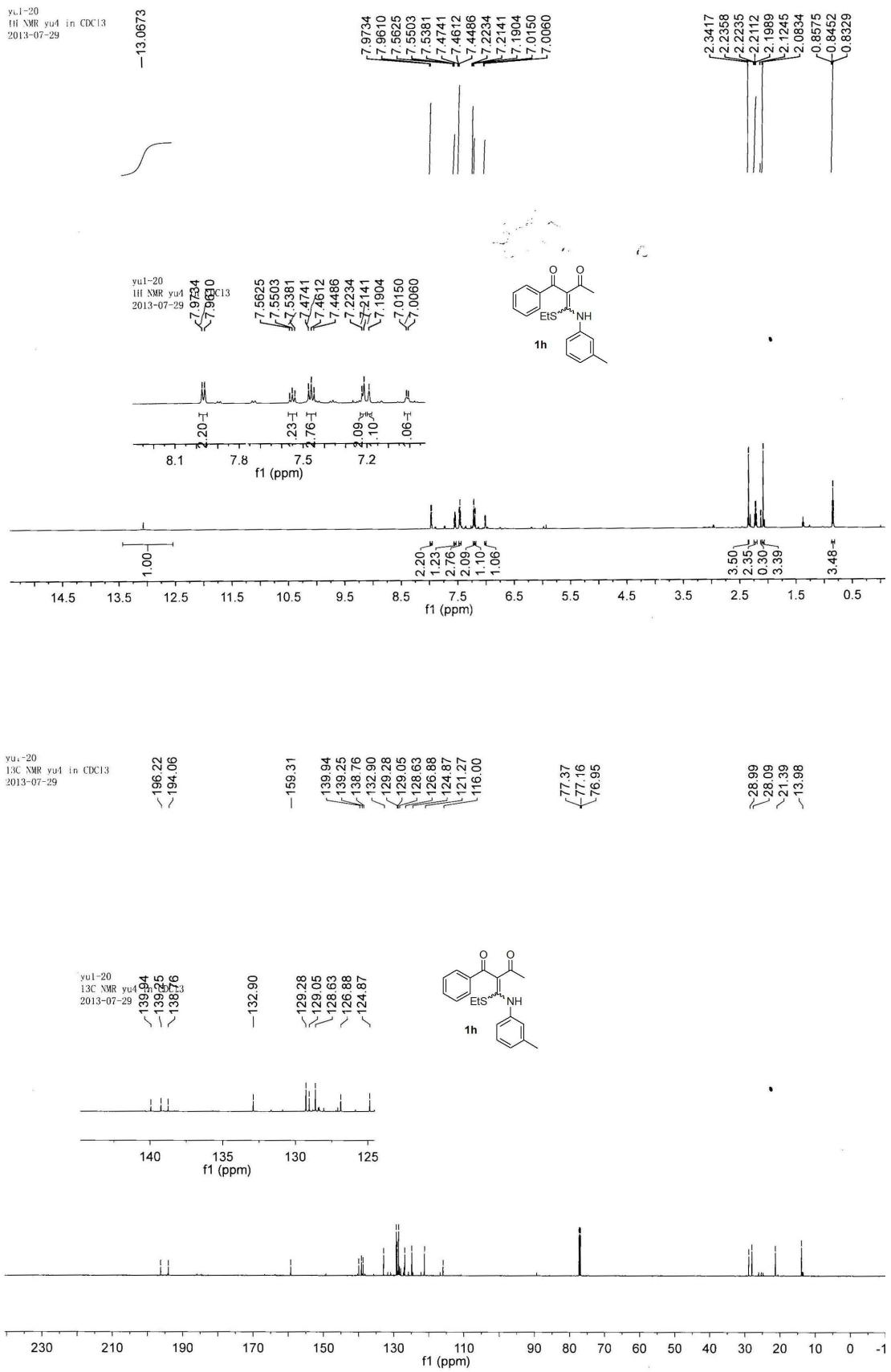


2013-6-8
¹H NMR yuhf16 in CDCl₃
2013-06-07

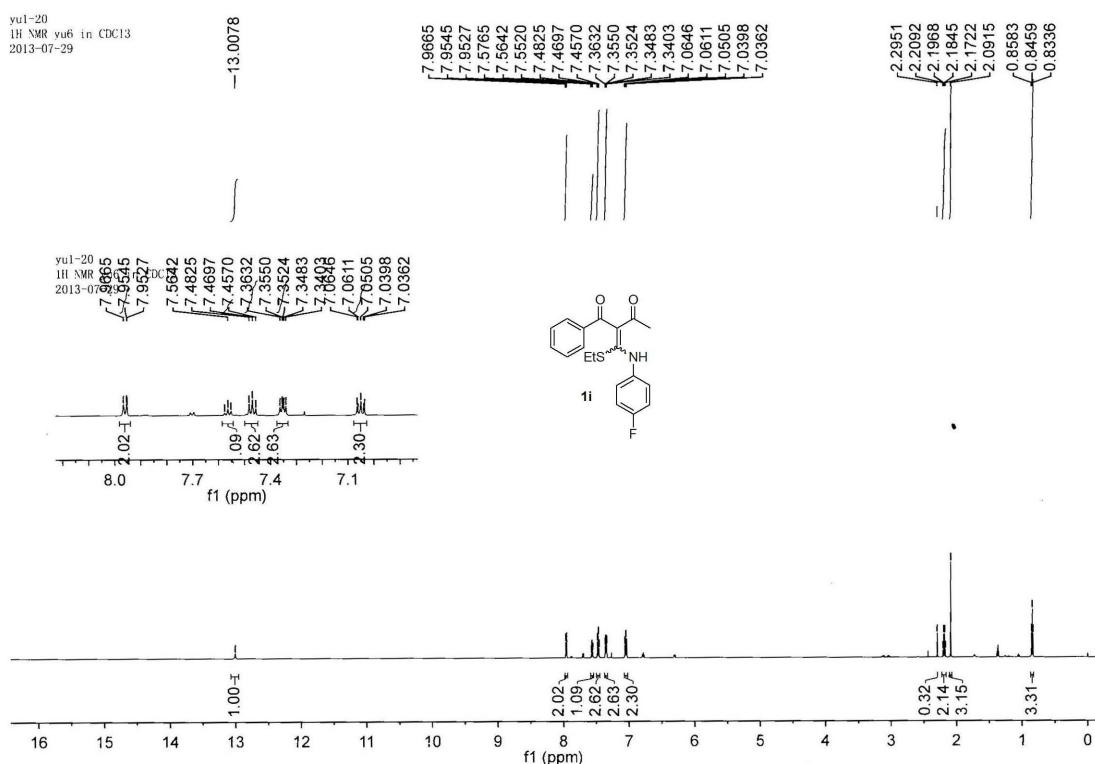


2013-6-8
¹³C NMR yuhf16 in CDCl₃
2013-06-07

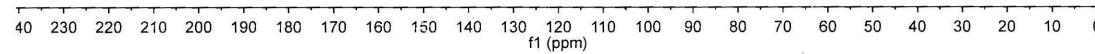
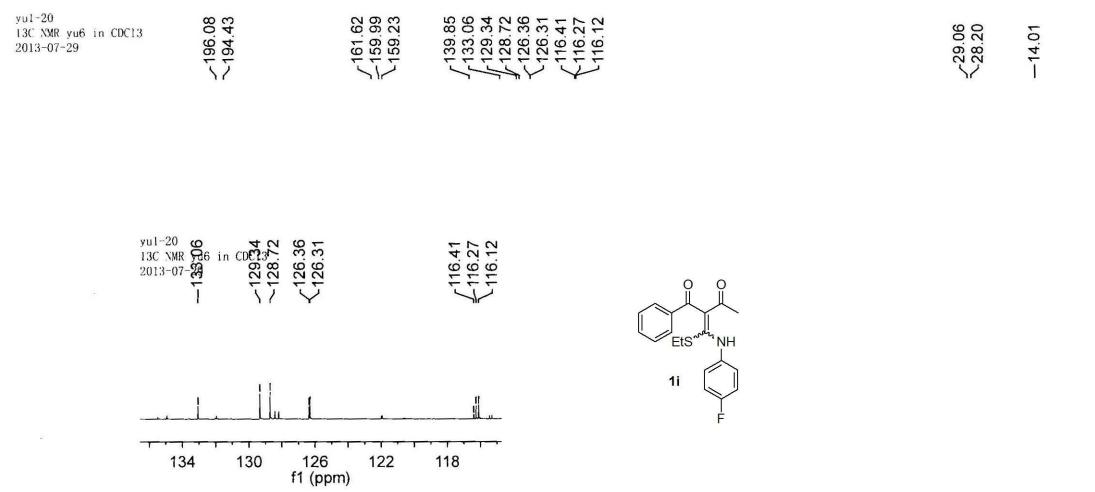




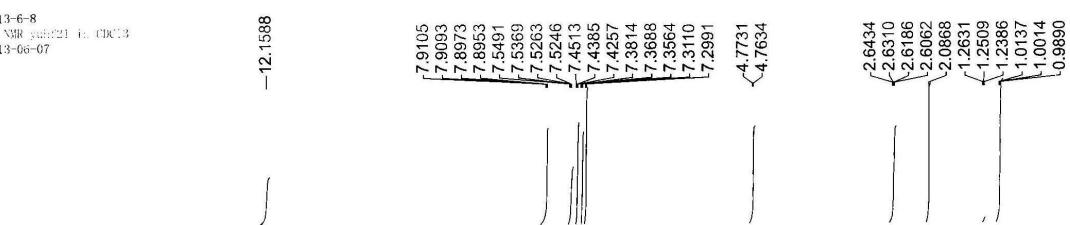
yul-20
1H NMR yu6 in CDCl₃
2013-07-29



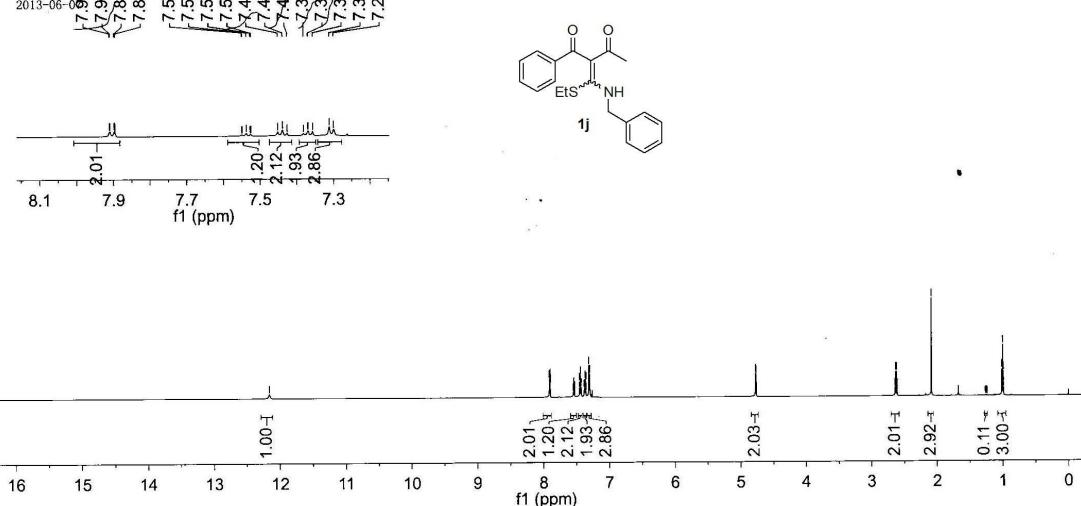
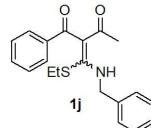
yul-20
13C NMR yu6 in CDCl₃
2013-07-29



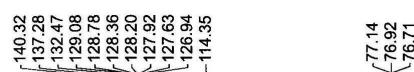
2013-6-8
1H NMR δ (ppm) in CDCl₃
2013-06-07



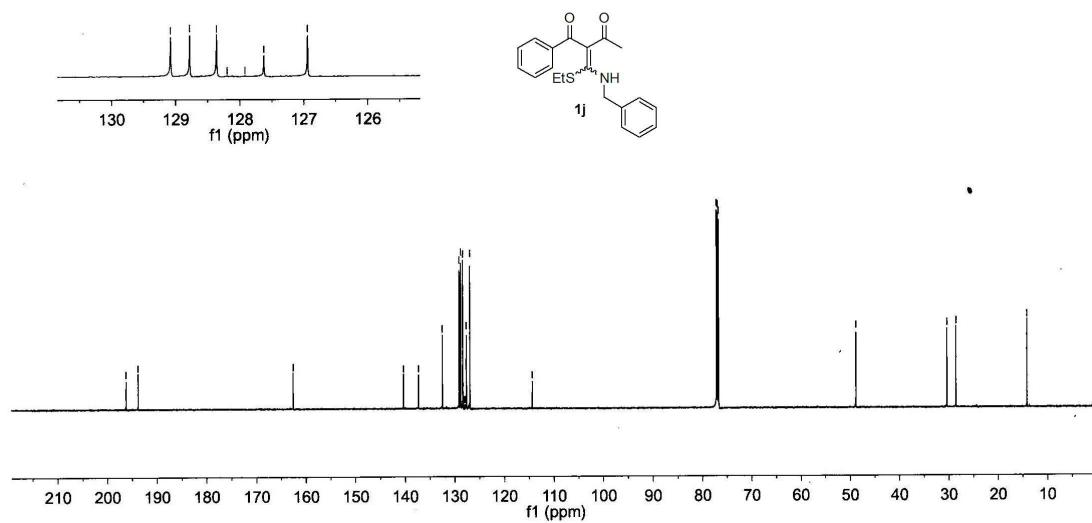
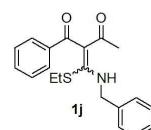
2013-6-8
 1H NMR δ (ppm): 1.05, 2.03, 2.97, 3.95, 5.31, 6.49, 7.36, 7.63, 7.66, 7.73, 7.76, 7.81, 7.84, 7.86, 7.91, 7.93, 7.95, 8.01, 8.03, 8.05, 8.07, 8.10, 8.12, 8.14, 8.16, 8.18, 8.20, 8.22, 8.24, 8.26, 8.28, 8.30, 8.32, 8.34, 8.36, 8.38, 8.40, 8.42, 8.44, 8.46, 8.48, 8.50, 8.52, 8.54, 8.56, 8.58, 8.60, 8.62, 8.64, 8.66, 8.68, 8.70, 8.72, 8.74, 8.76, 8.78, 8.80, 8.82, 8.84, 8.86, 8.88, 8.90, 8.92, 8.94, 8.96, 8.98, 9.00, 9.02, 9.04, 9.06, 9.08, 9.10, 9.12, 9.14, 9.16, 9.18, 9.20, 9.22, 9.24, 9.26, 9.28, 9.30, 9.32, 9.34, 9.36, 9.38, 9.40, 9.42, 9.44, 9.46, 9.48, 9.50, 9.52, 9.54, 9.56, 9.58, 9.60, 9.62, 9.64, 9.66, 9.68, 9.70, 9.72, 9.74, 9.76, 9.78, 9.80, 9.82, 9.84, 9.86, 9.88, 9.90, 9.92, 9.94, 9.96, 9.98, 9.99, 10.00.

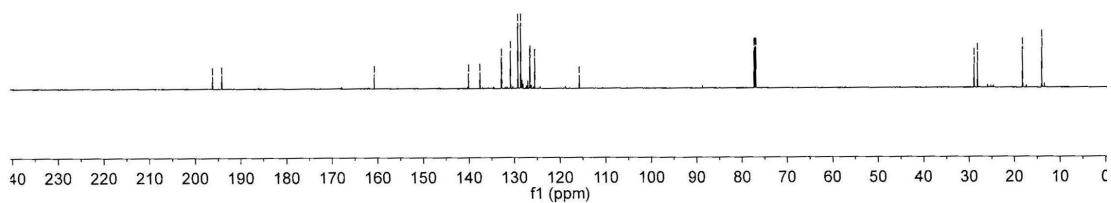
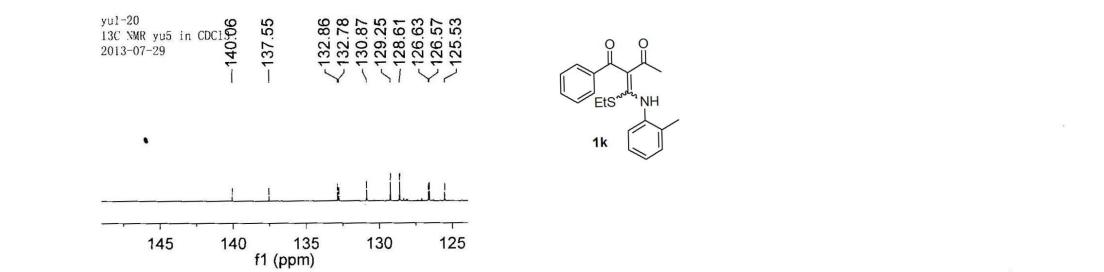
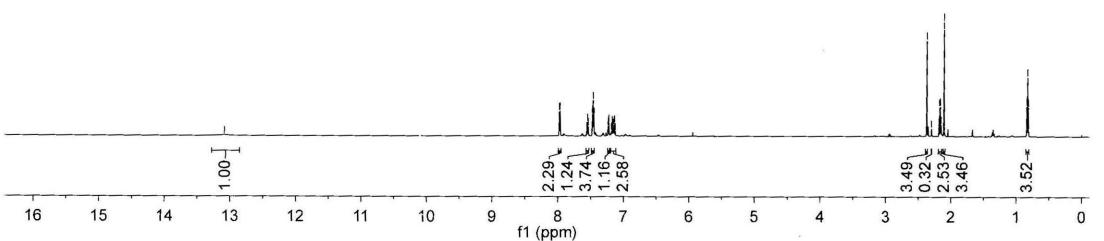
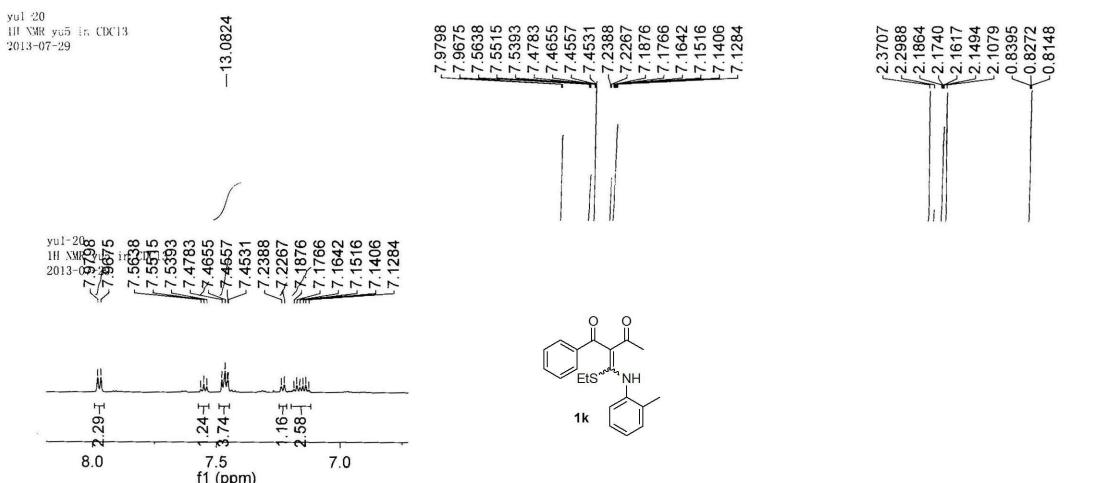


liuzs538-551
13C NMR yuhf21 in CDCl3
2013-06-07 96.20 93.78

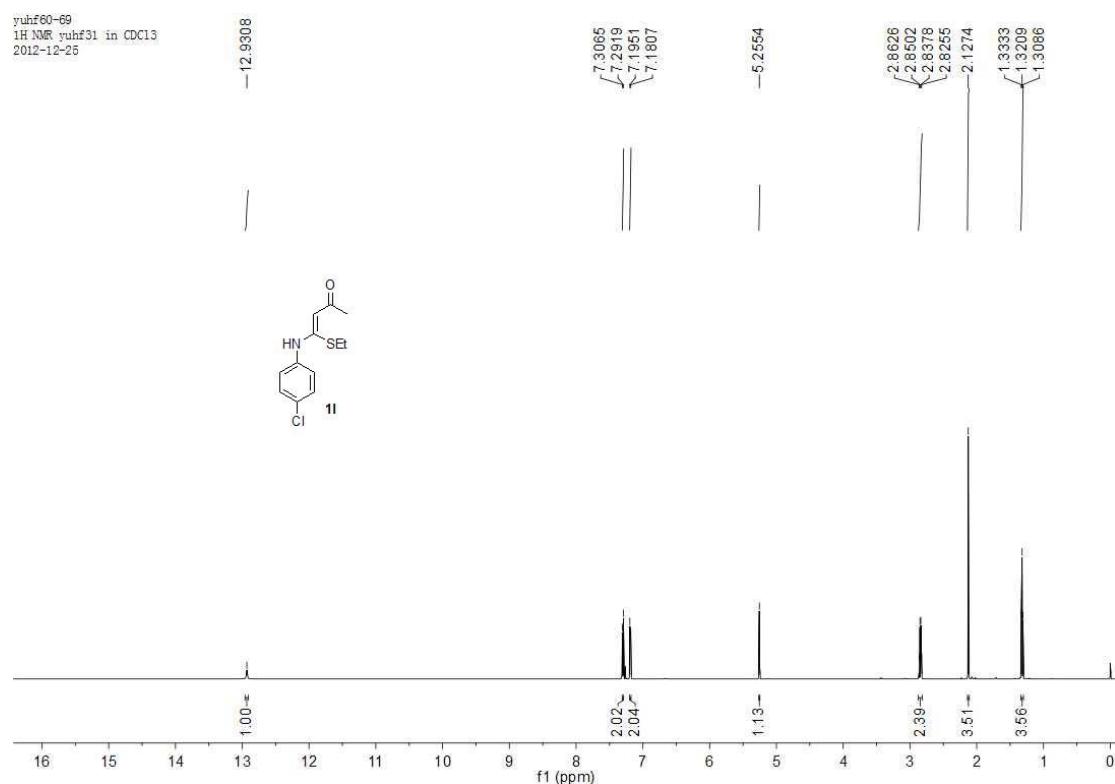


liuzs538-551
13C NMR yuhf21 in CDCl3
2013-06-07

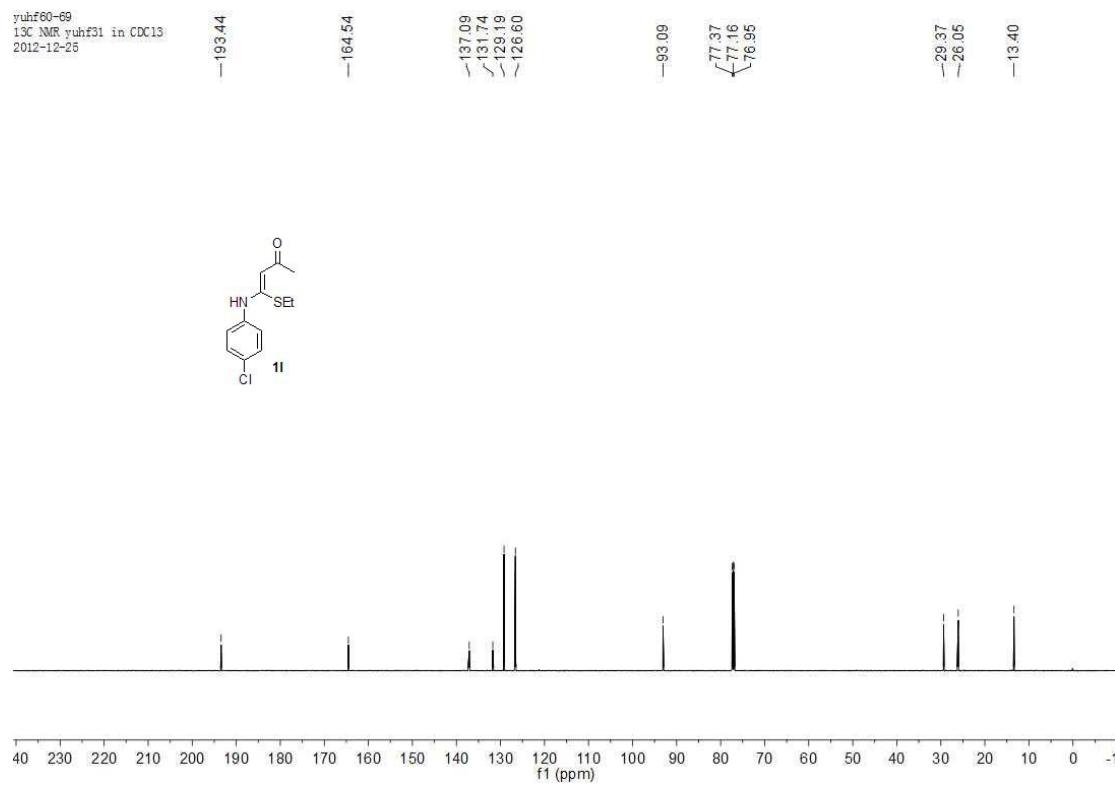




yuhf60-69
1H NMR yuhf31 in CDCl₃
2012-12-25



yuhf60-69
13C NMR yuhf31 in CDCl₃
2012-12-25

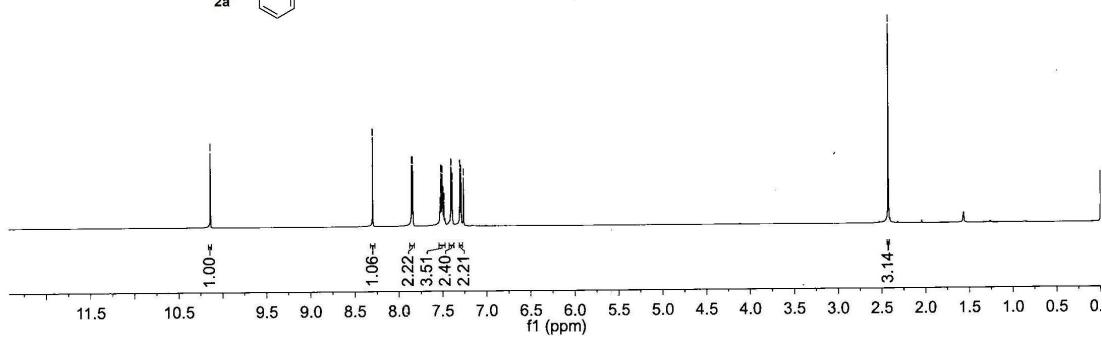
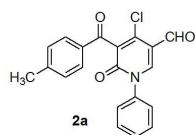


2013-5-15-Yuhf
III NMR yuhf9 in CDCl₃
2013-05-14

-10.1384

-8.2971
7.8536
7.8400
7.5276
7.5162
7.5033
7.4947
7.4830
7.4035
7.4010
7.3894
7.3011
7.2877
7.2603

-2.4245



2013-5-15-Yuhf
13C NMR yuhf9 in CDCl₃
2013-05-14

-190.27
-185.56

-158.57
145.77
143.76
138.63
133.08
129.96
129.86
129.76
129.66
126.25
126.51

77.35
77.14
76.93

-21.99

2013-5-15-Yuhf
13C NMR yuhf9 in CDCl₃
2013-05-14

-145.77

143.76

143.20

143.08

129.96

129.86

129.76

129.66

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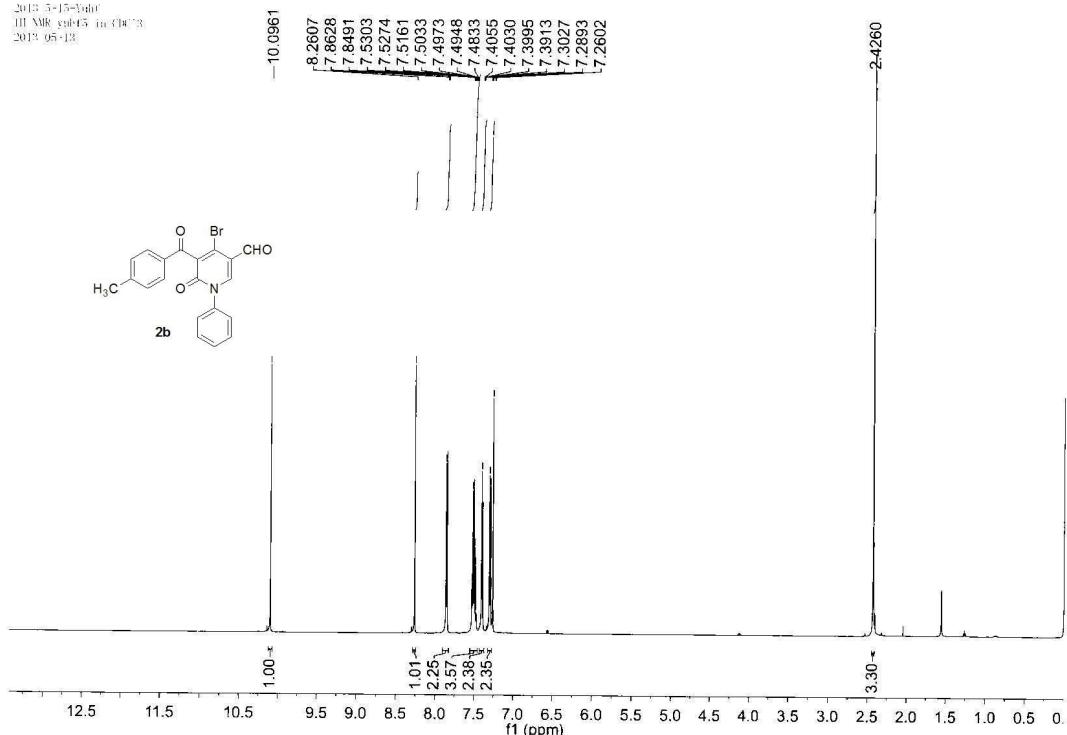
126.51

126.51

126.51

126.51

2013-5-15-Yuif
1H NMR yuhf5 in CDCl3
2013-05-13



2013-5-15-Yuif
13C NMR yuhf5 in CDCl3
2013-05-13

191.5
187.48

133.03
132.76
132.57

129.81
129.68
129.64
129.60
129.57
129.53
129.50
129.46
129.43

126.02

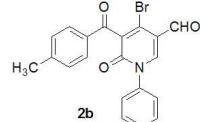
115.67
143.34
138.57
133.03
132.57
129.81
129.68
129.64
129.60
129.57
129.53
129.50
129.46
129.43

108.20

77.27
77.86
76.84
21.92

137 136 135 134 133 132 131 130 129

f1 (ppm)



2013-5-15-Yuif

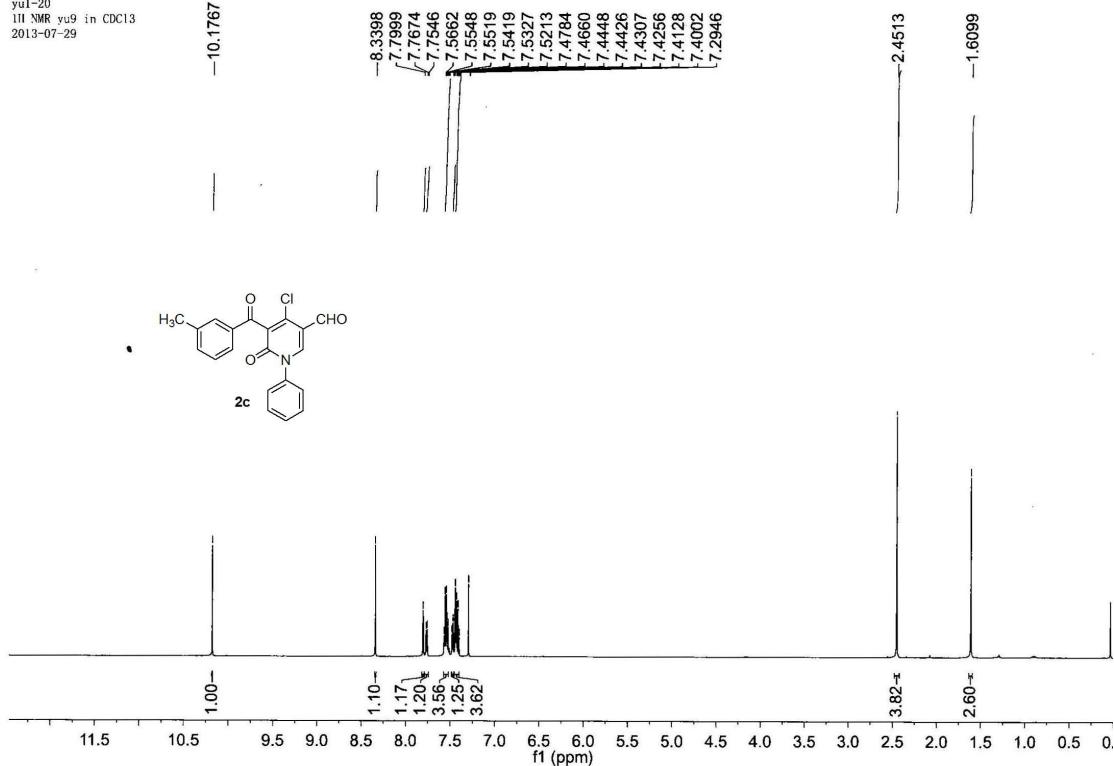
13C NMR yuhf5 in CDCl3

2013-05-13

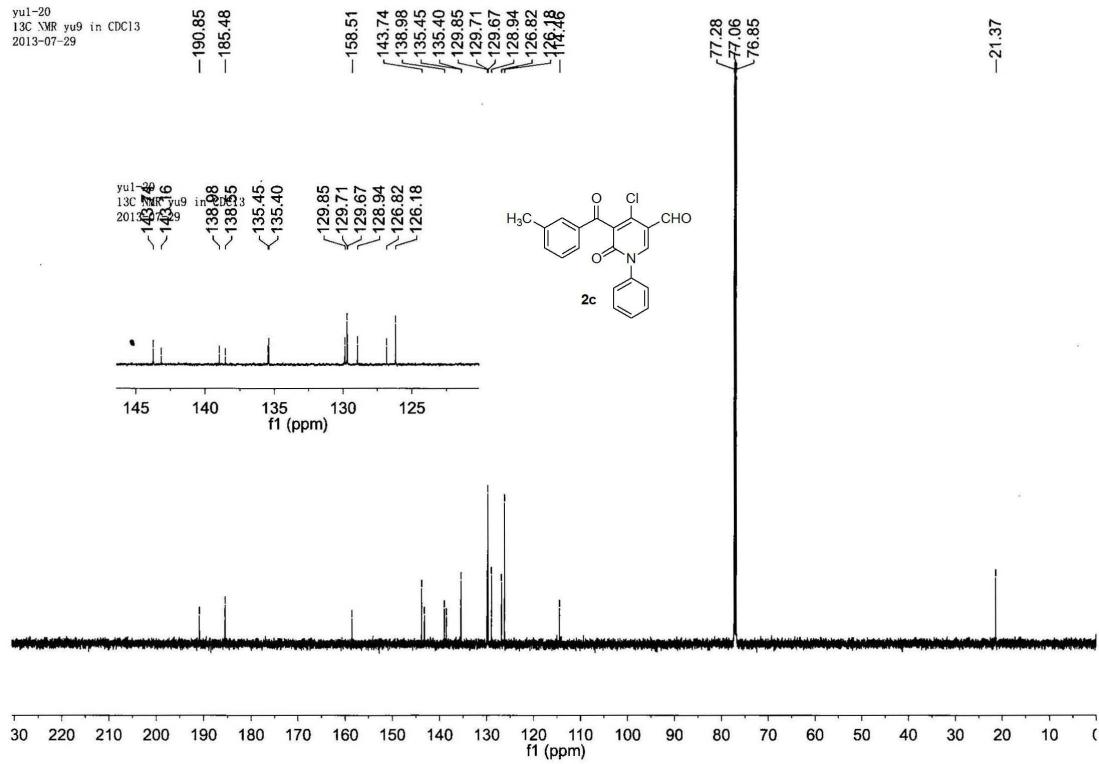
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 -10

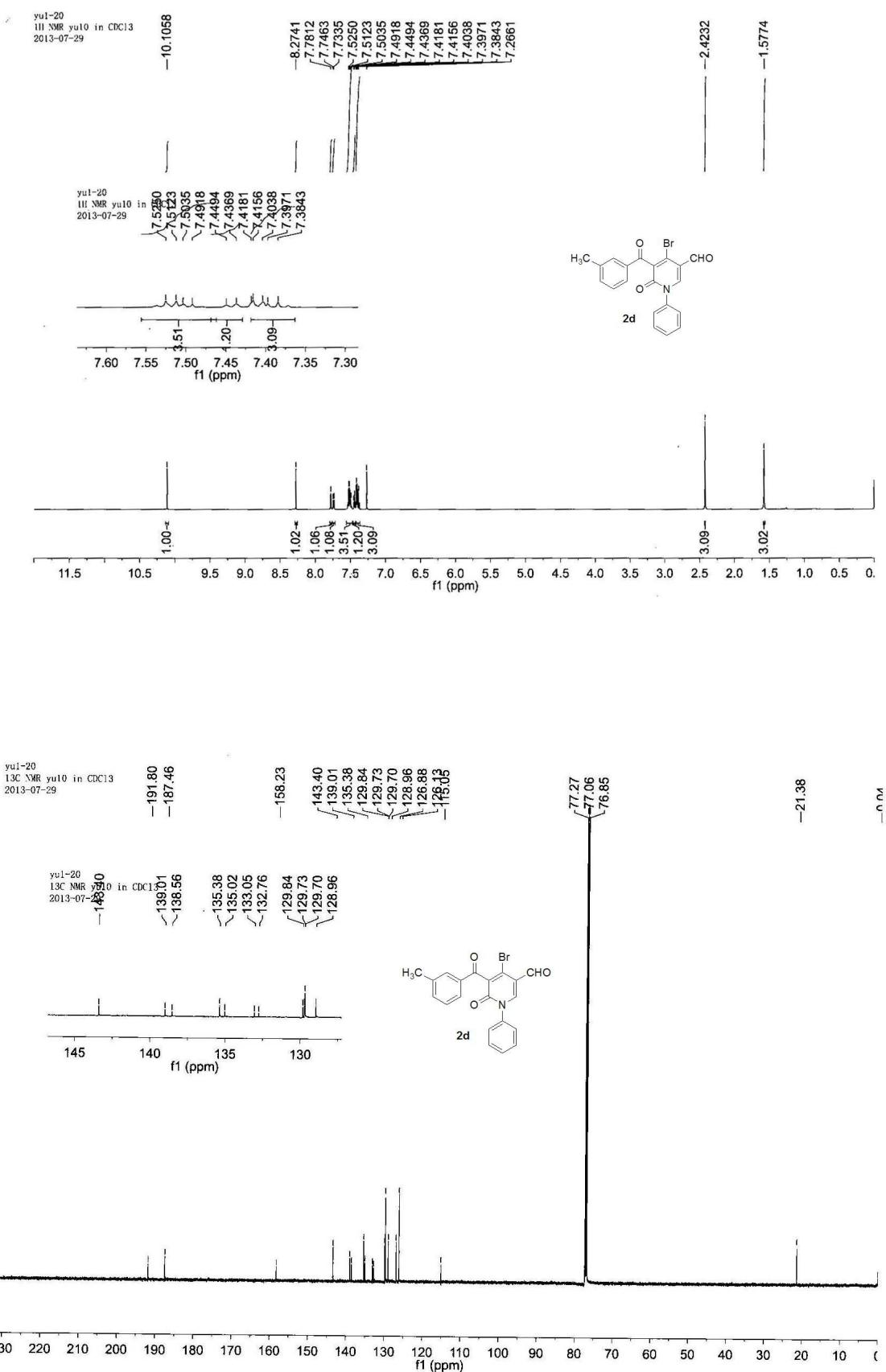
f1 (ppm)

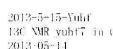
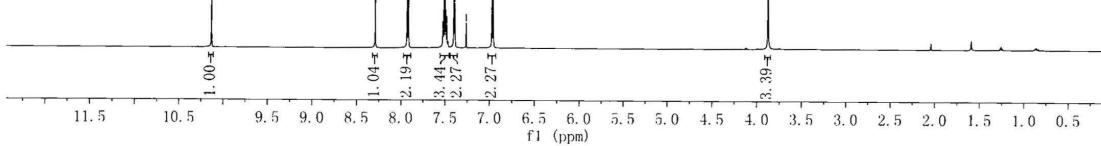
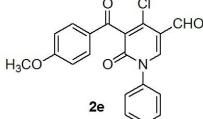
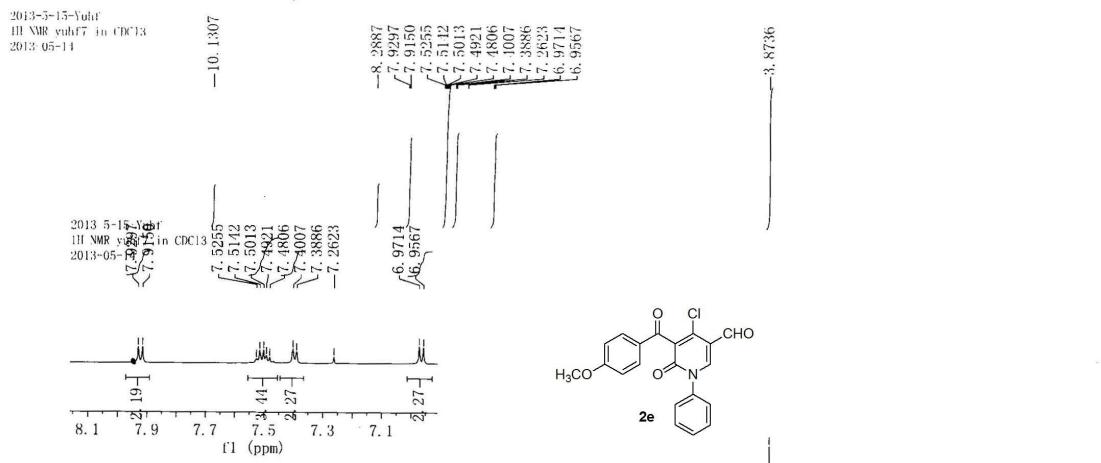
yul-20
1H NMR yu9 in CDCl₃
2013-07-29



yul-20
13C NMR yu9 in CDCl₃
2013-07-29







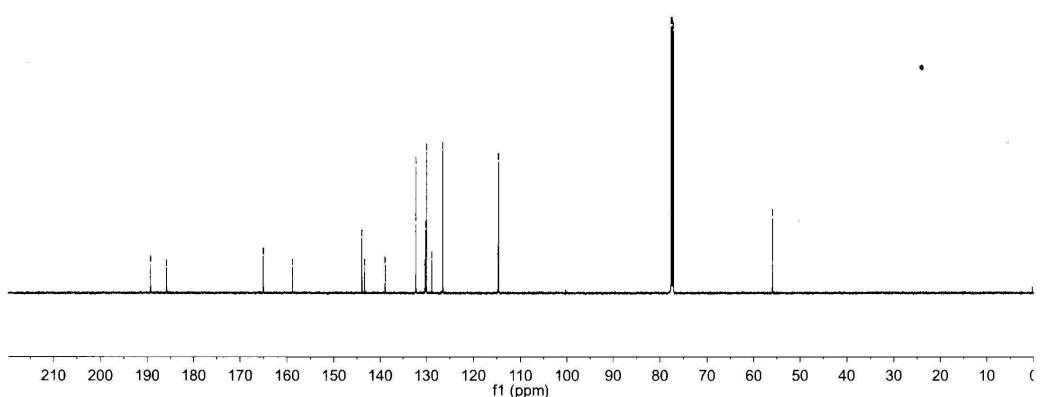
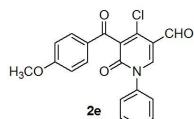
2013-5-15-Yuhf
13C NMR yuhf7 in C
2013-05-14

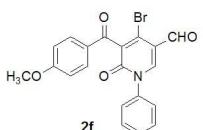
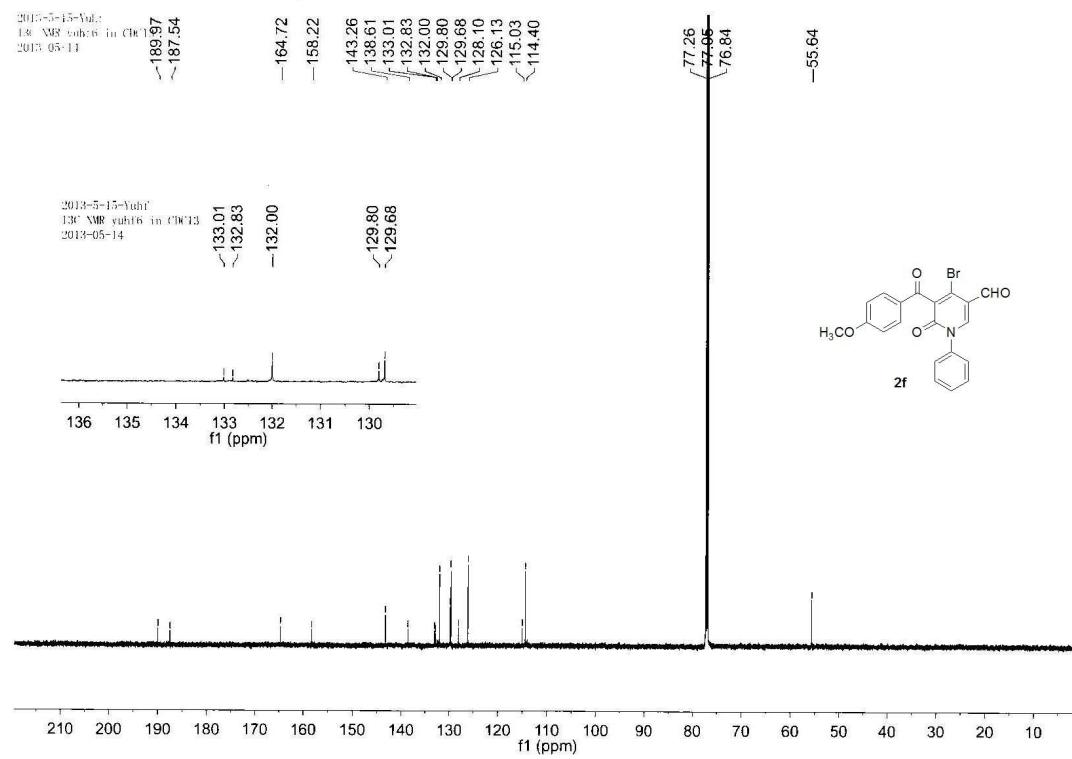
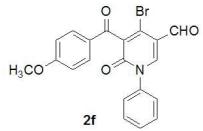
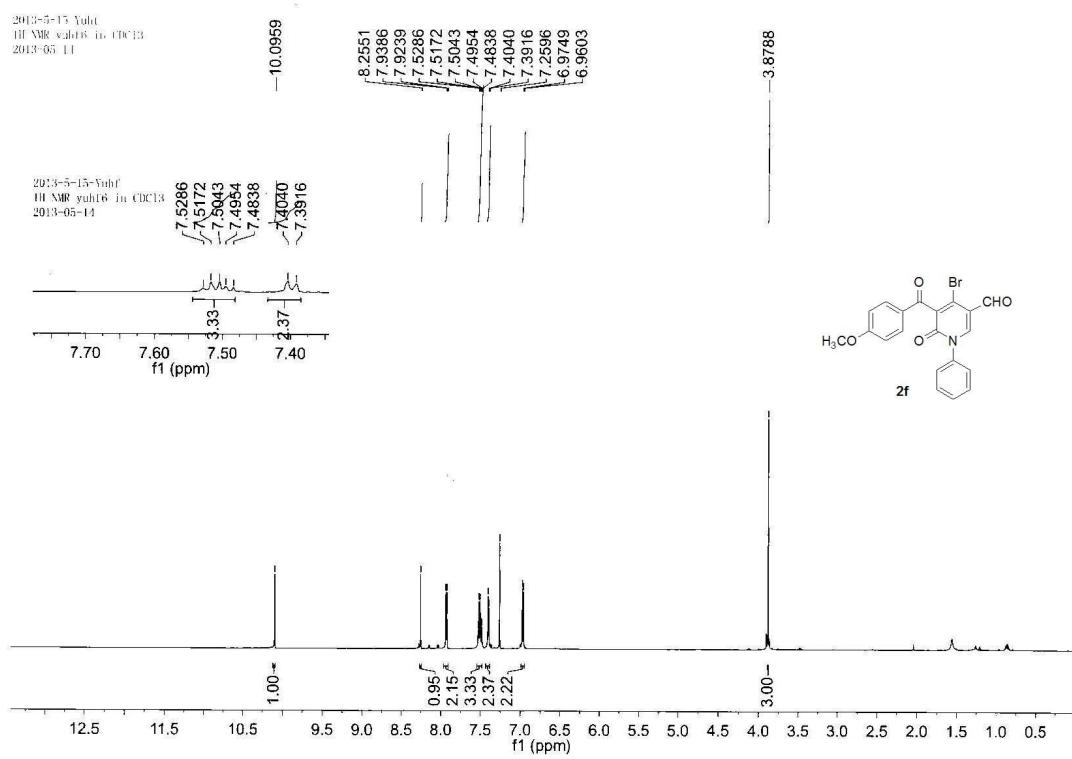
65.03

13.94
13.33
88.90
32.24
80.26
80.10
99.98
28.83
26.48
4.74
4.66

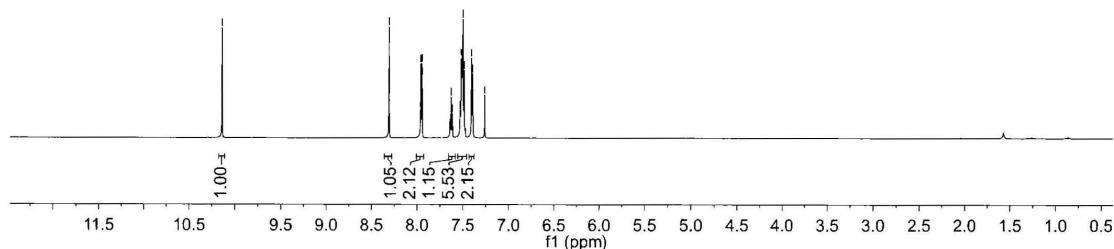
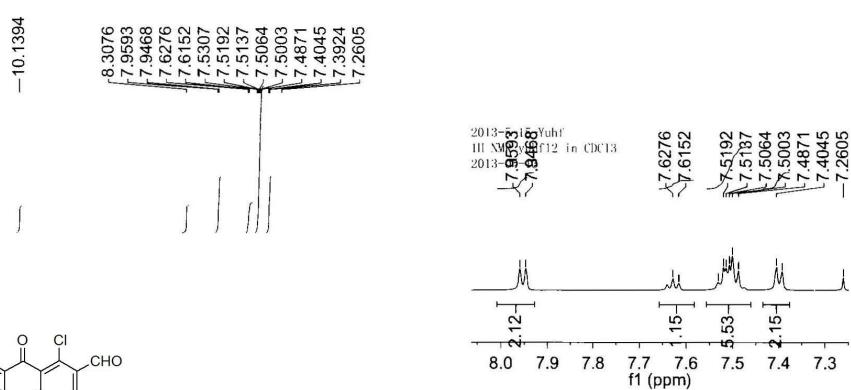
58
37
16

5.95



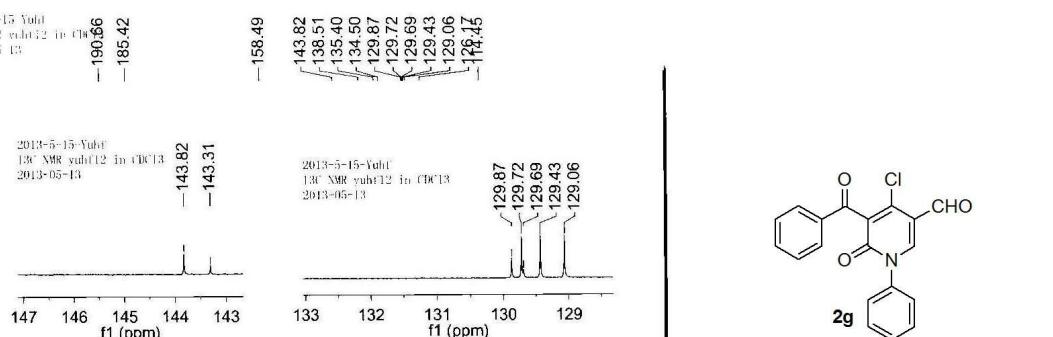


2013-5-15-Yuhf^t
1H NMR vuhf12 in CDCl₃
2013-05-13

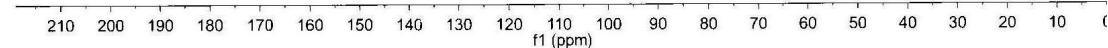


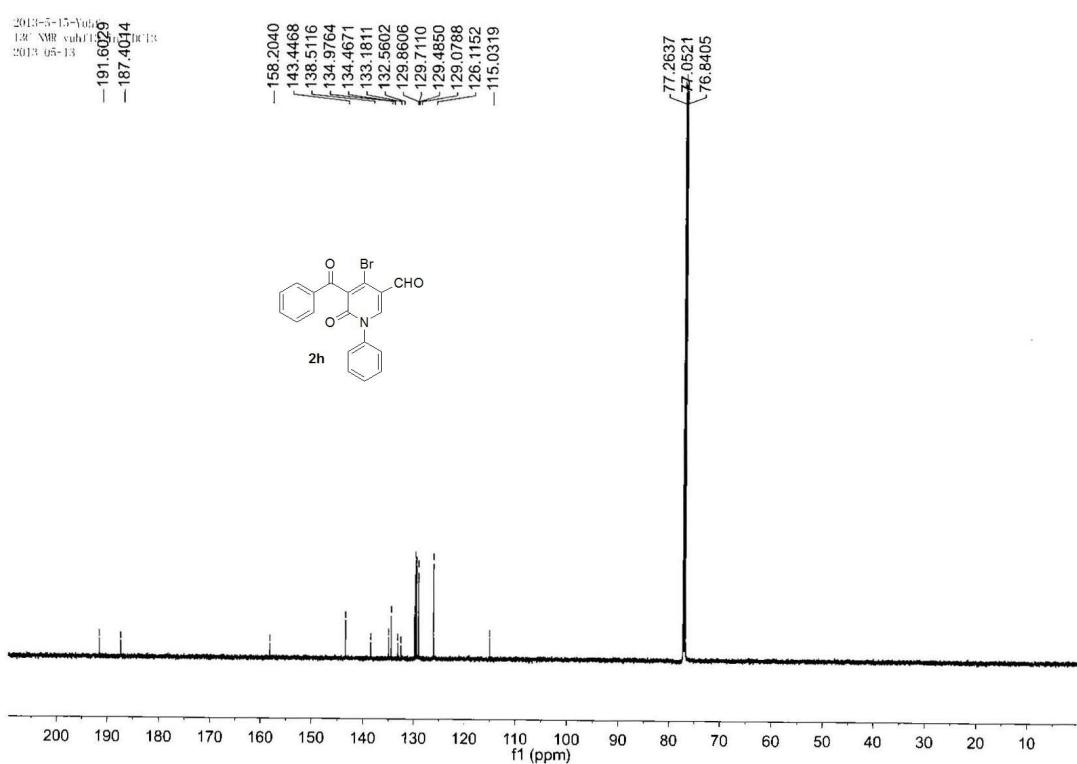
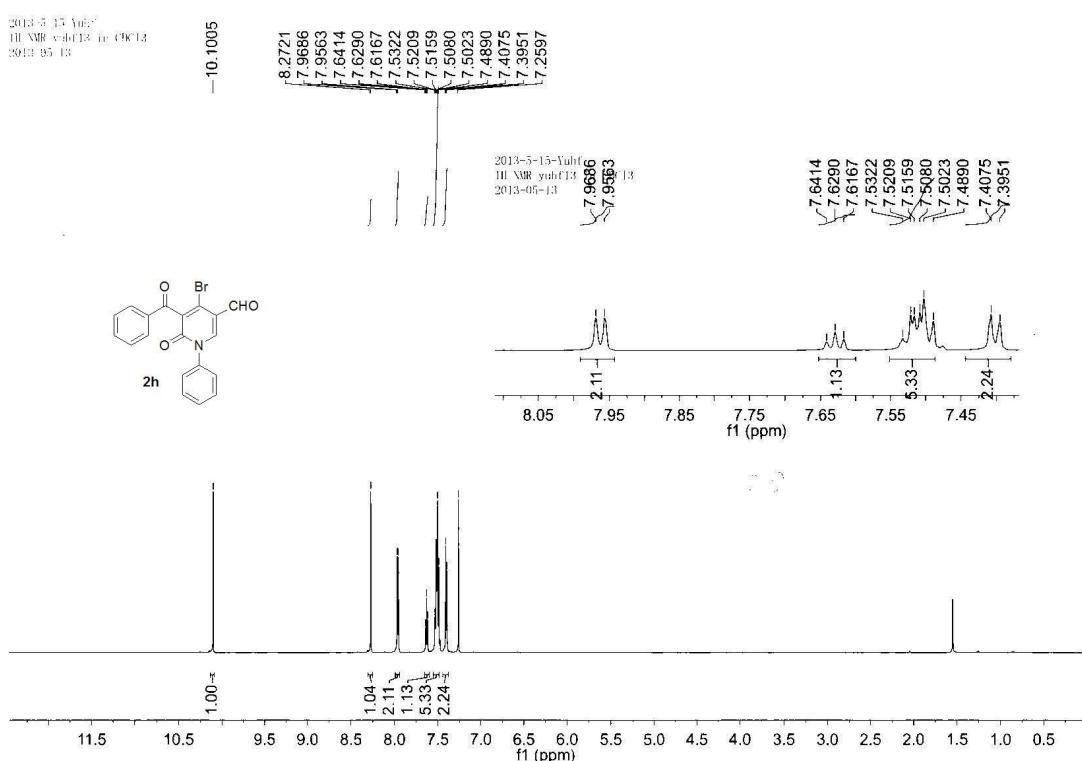
2013-5-15-Yuhf^t
13C NMR vuhf12 in CDCl₃
2013-05-13

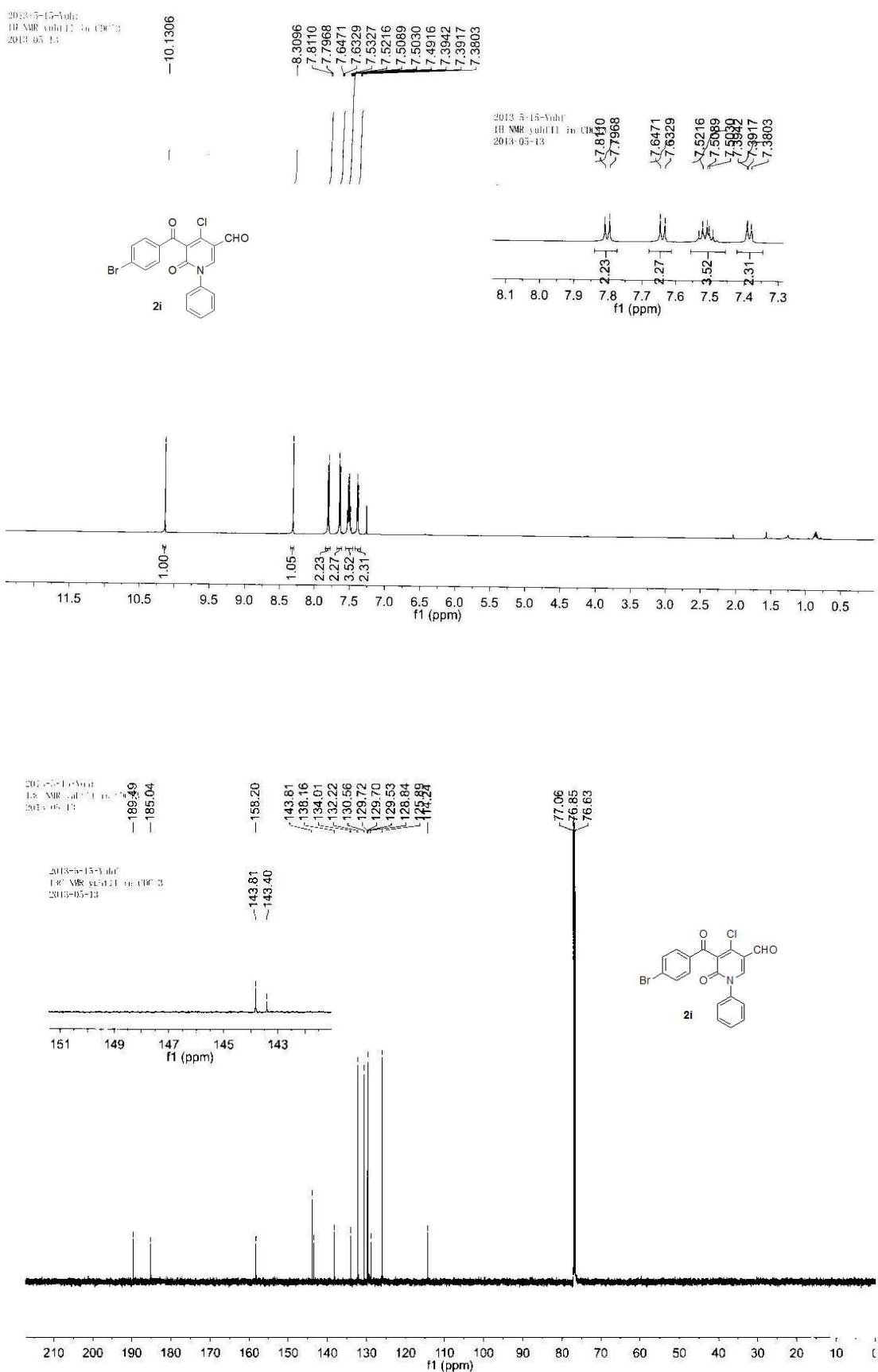
—190.66
—185.42

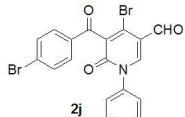
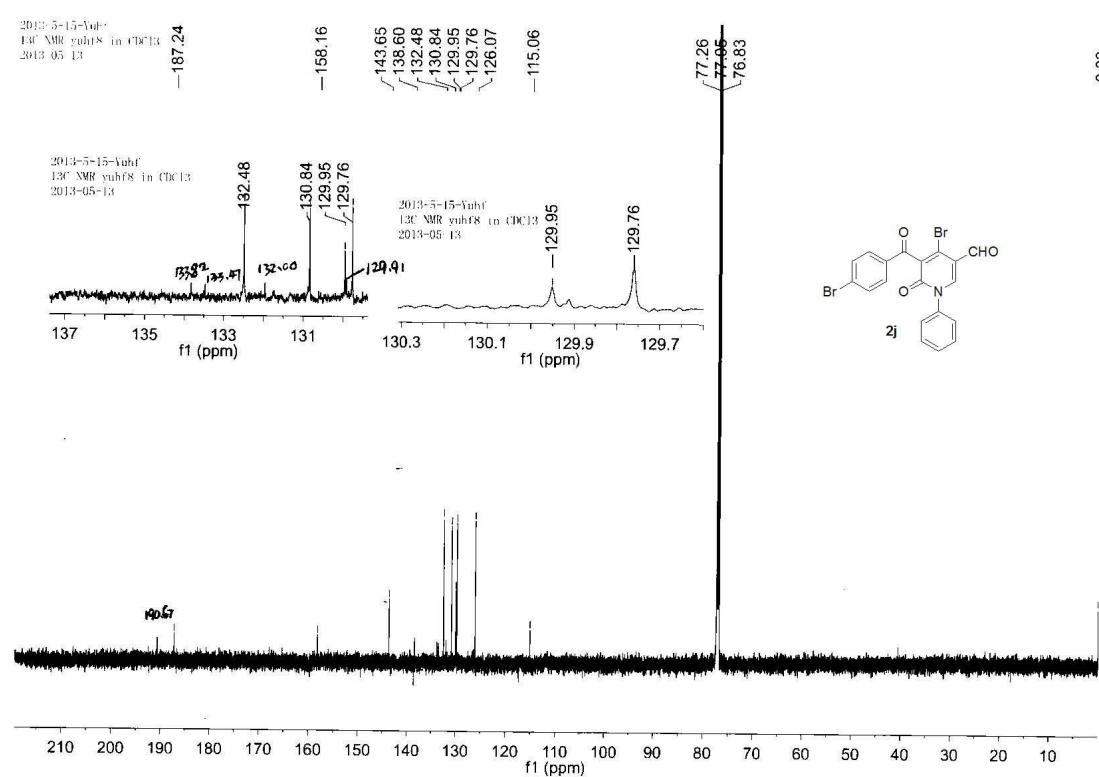
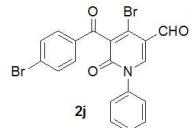
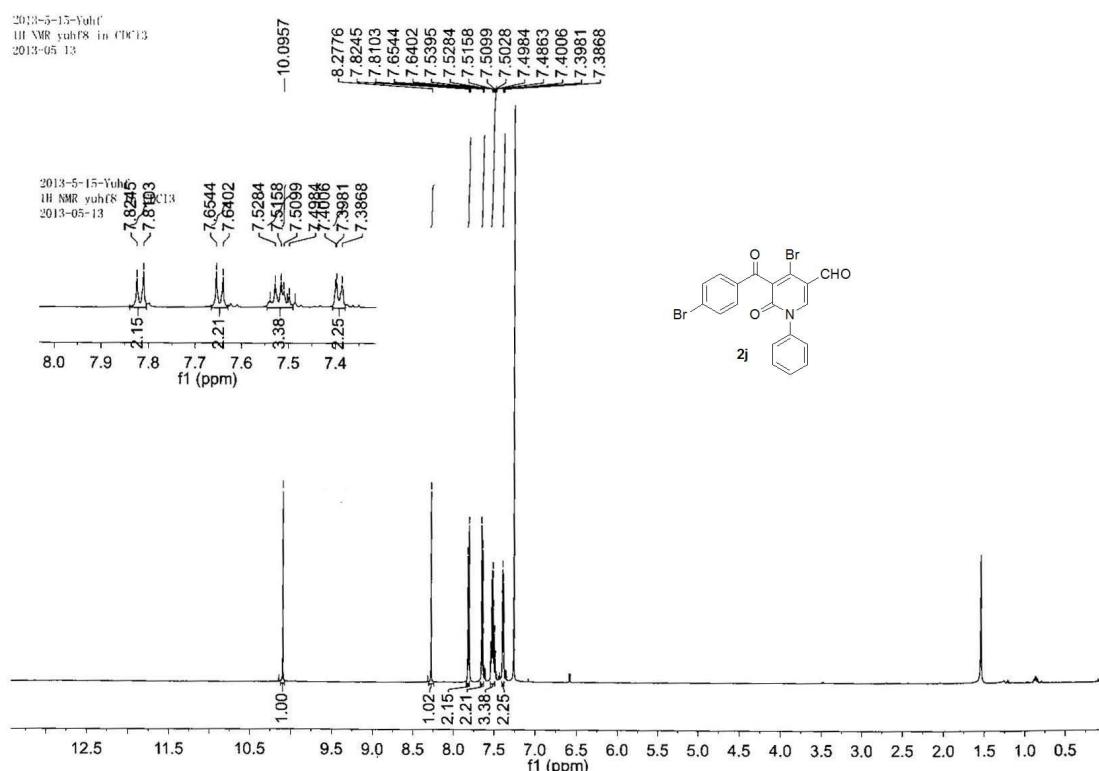


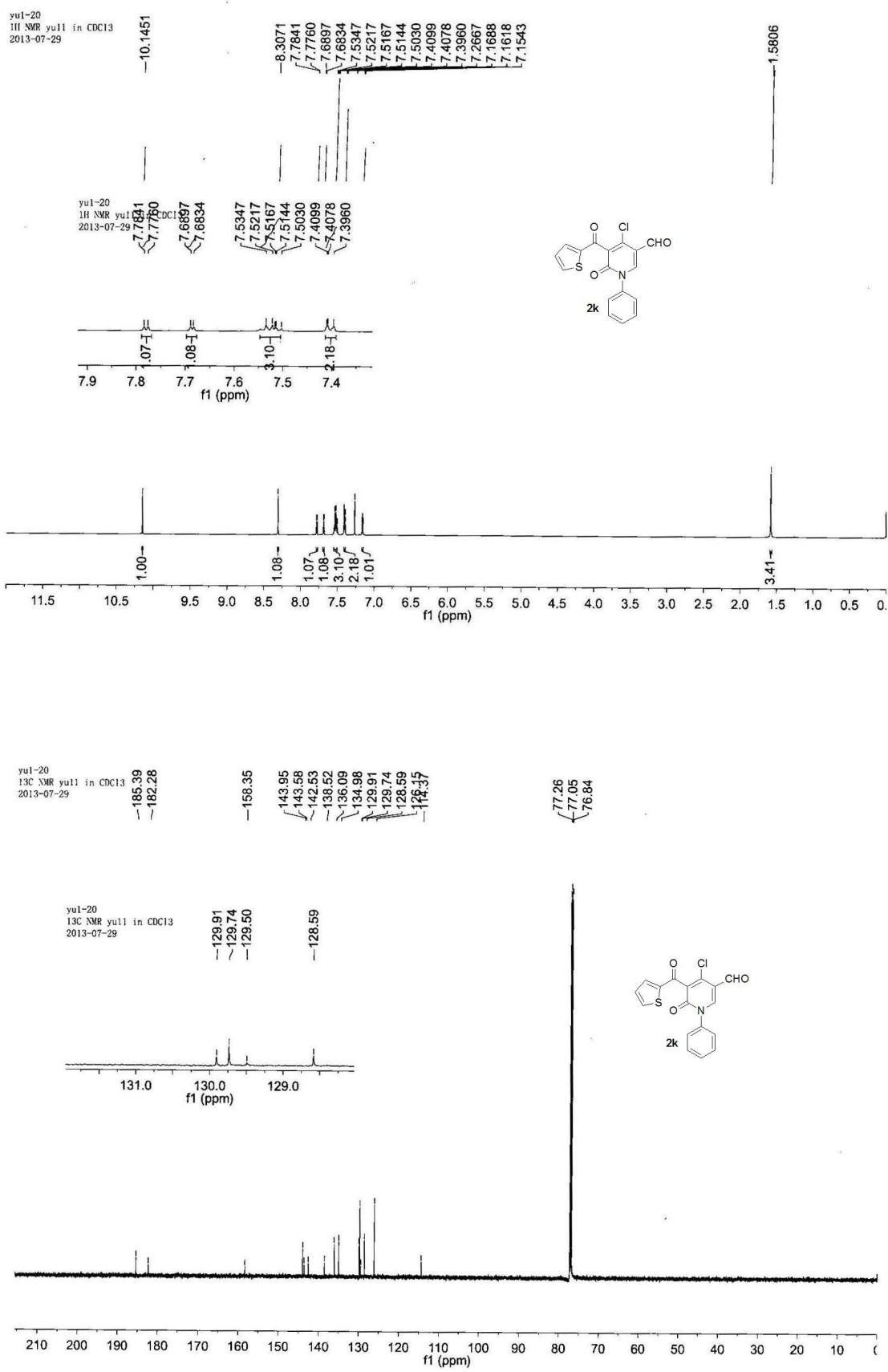
2013-5-15-Yuhf^t
13C NMR vuhf12 in CDCl₃
2013-05-13

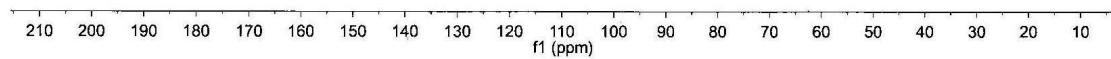
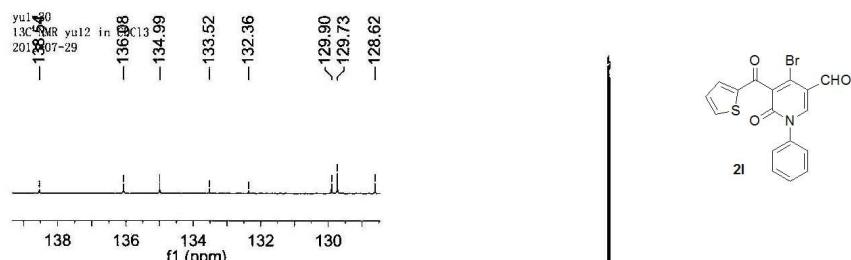
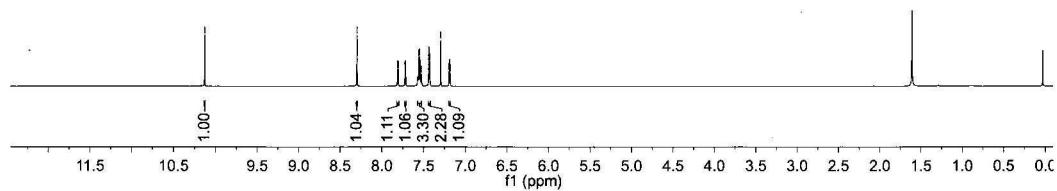
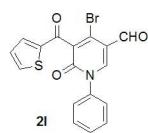
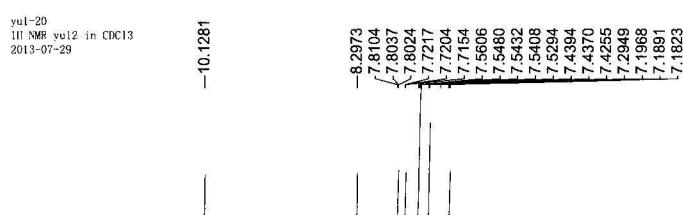




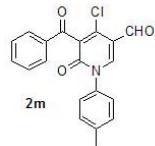
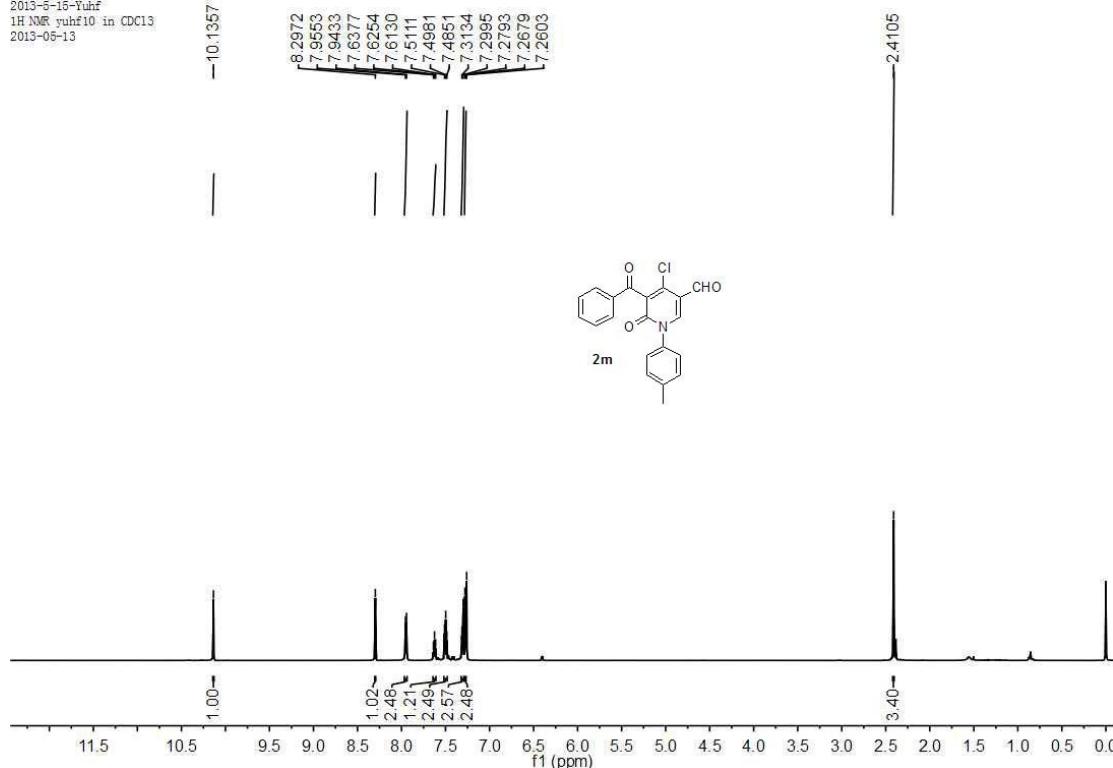




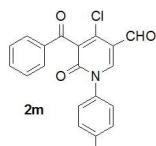
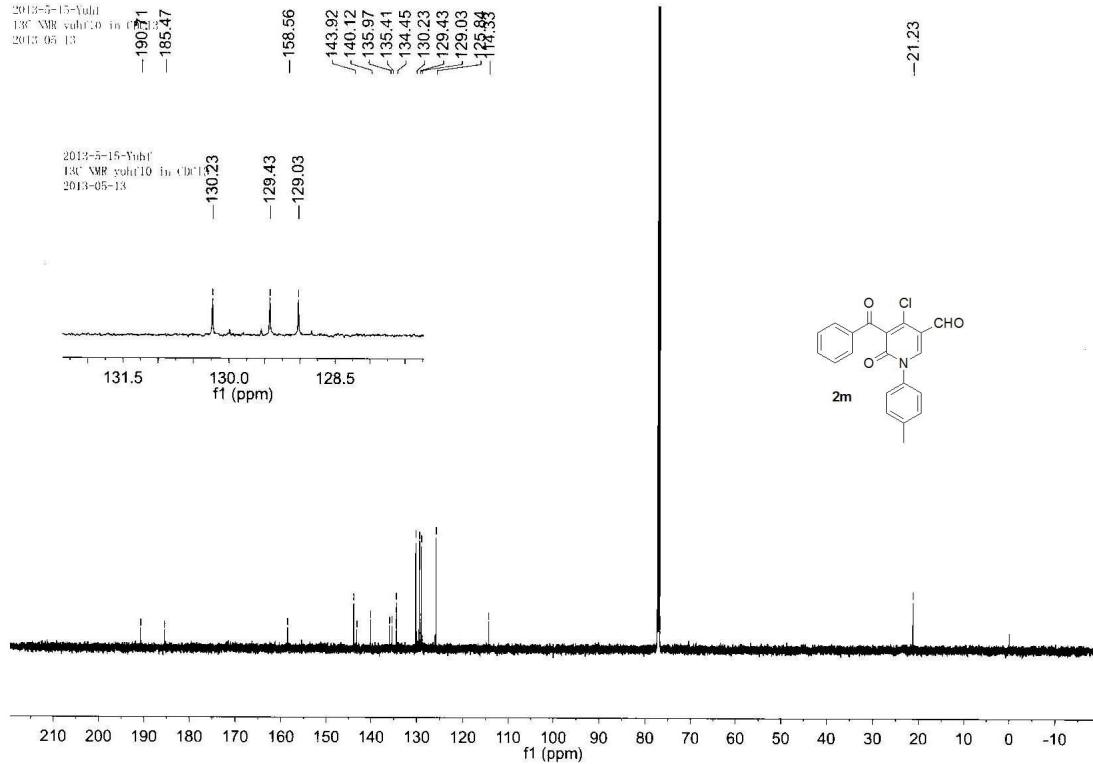


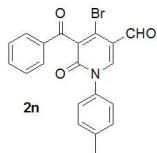
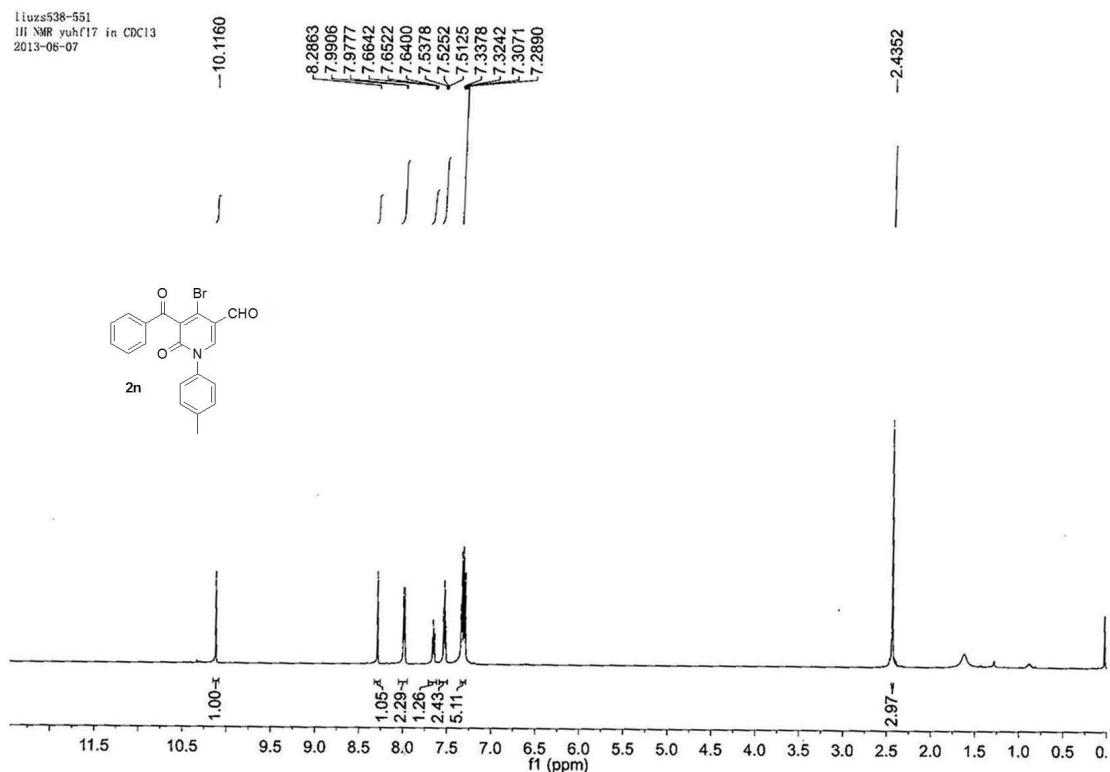


2013-5-15-Yuhf
1H NMR yuhf10 in CDCl3
2013-05-13

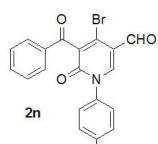
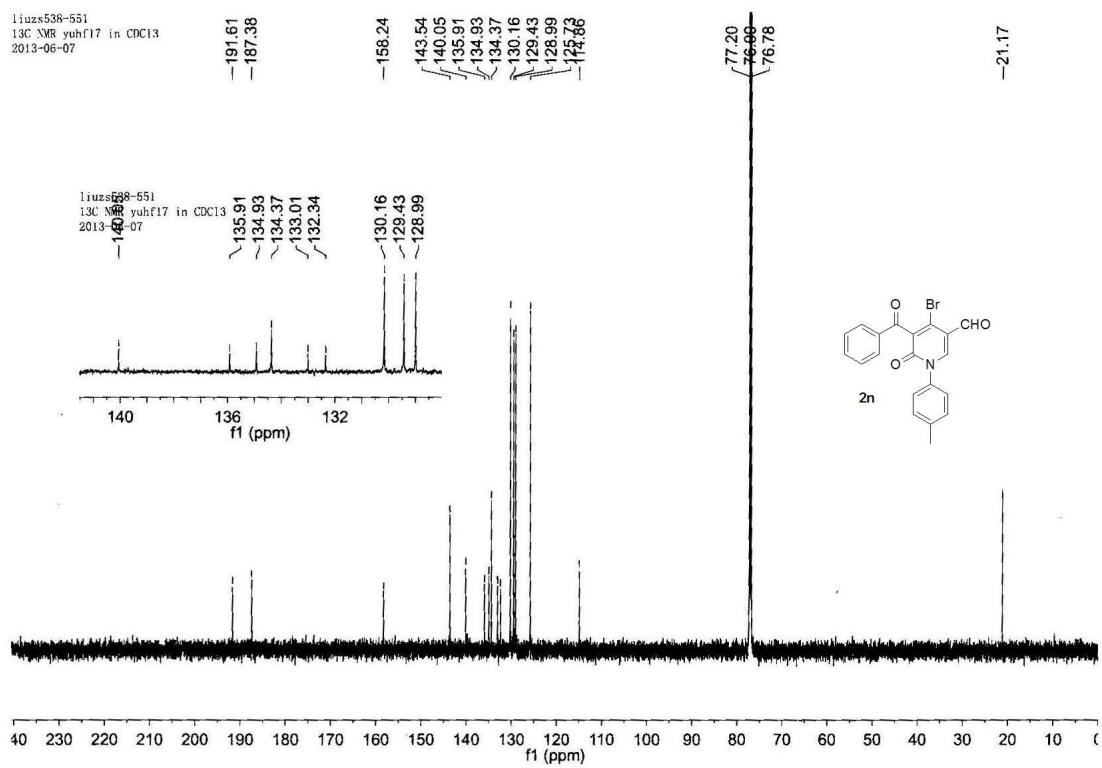


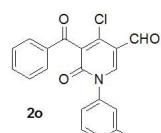
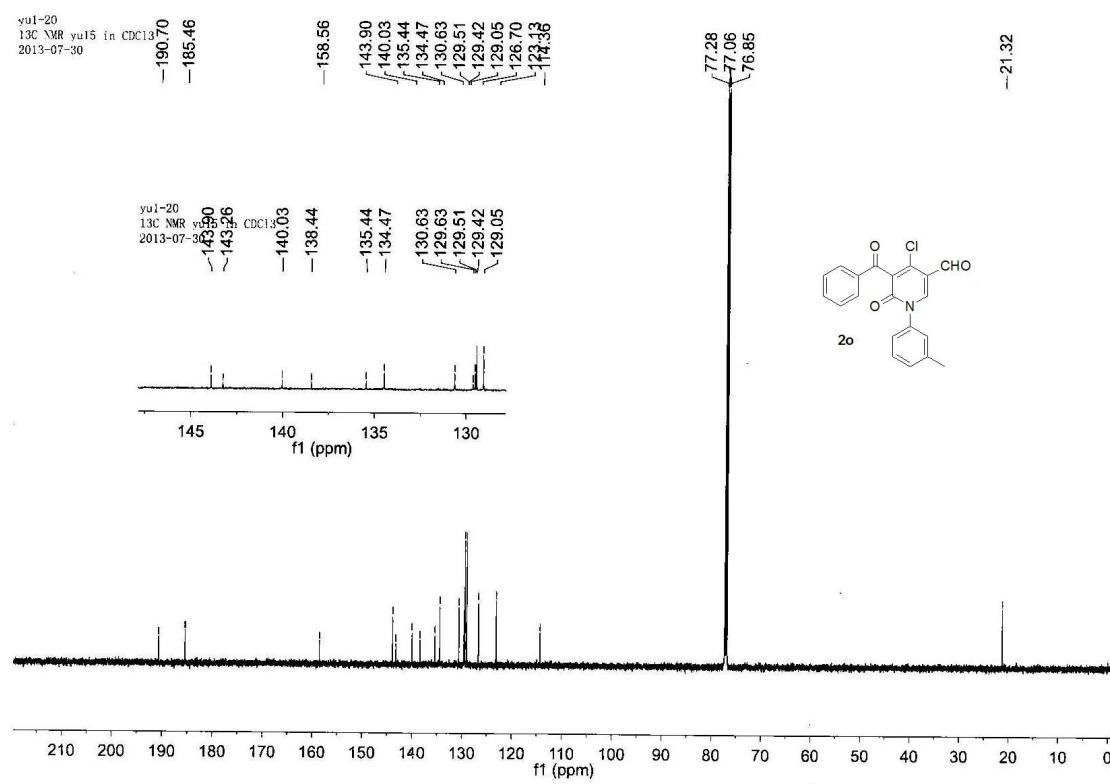
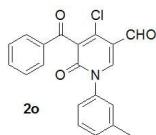
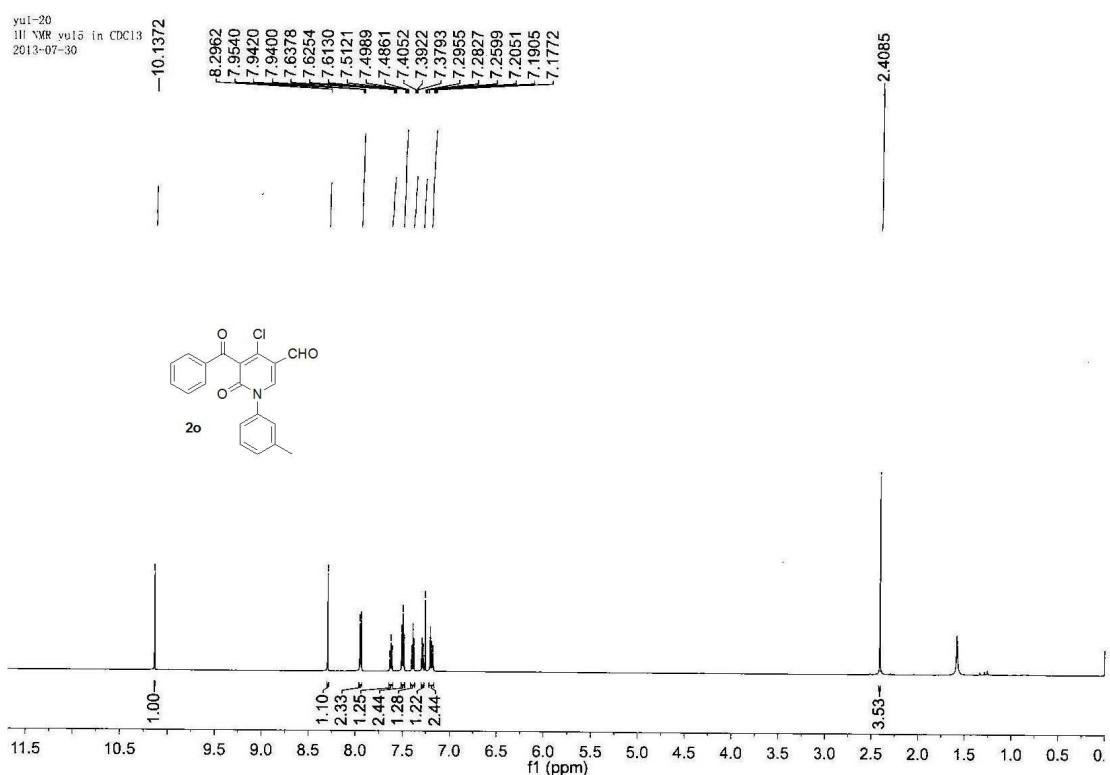
19071
185.47



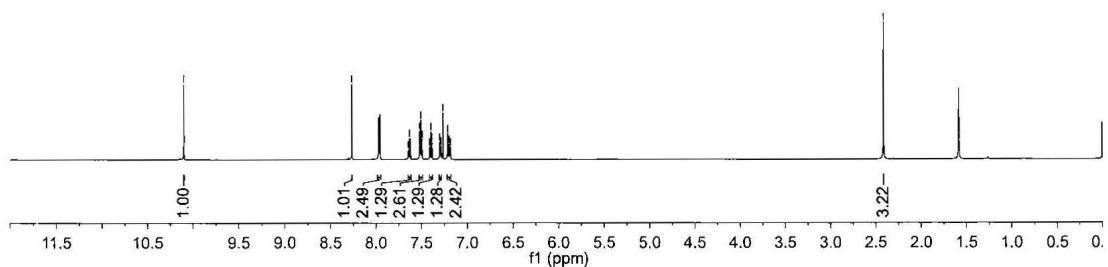
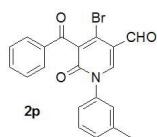
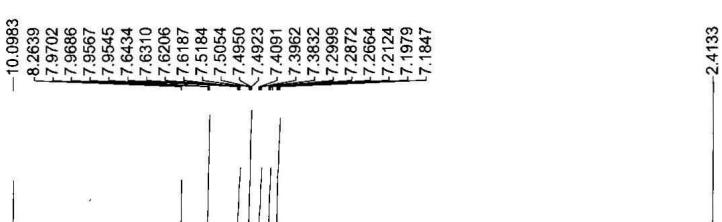


liuzs538-551
13C NMR yuhf17 in CDCl3
2013-06-07

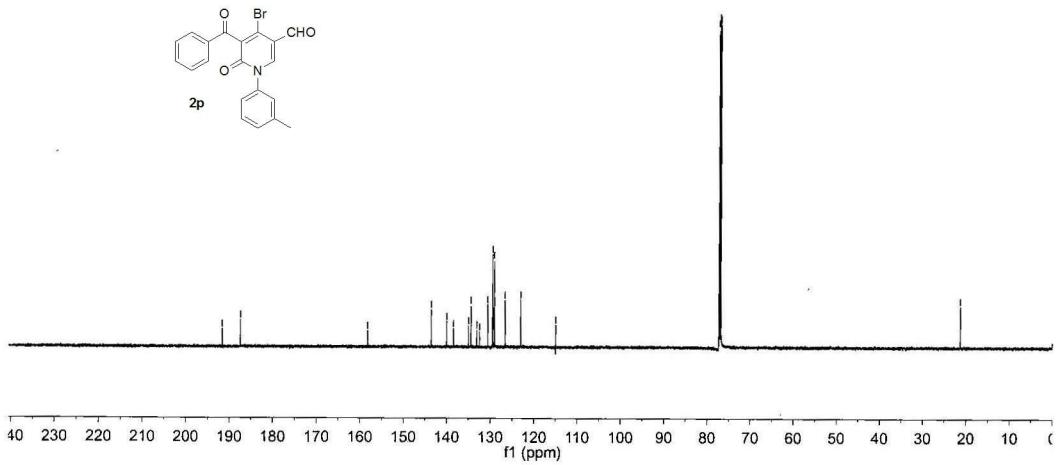
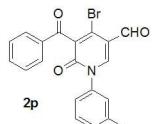


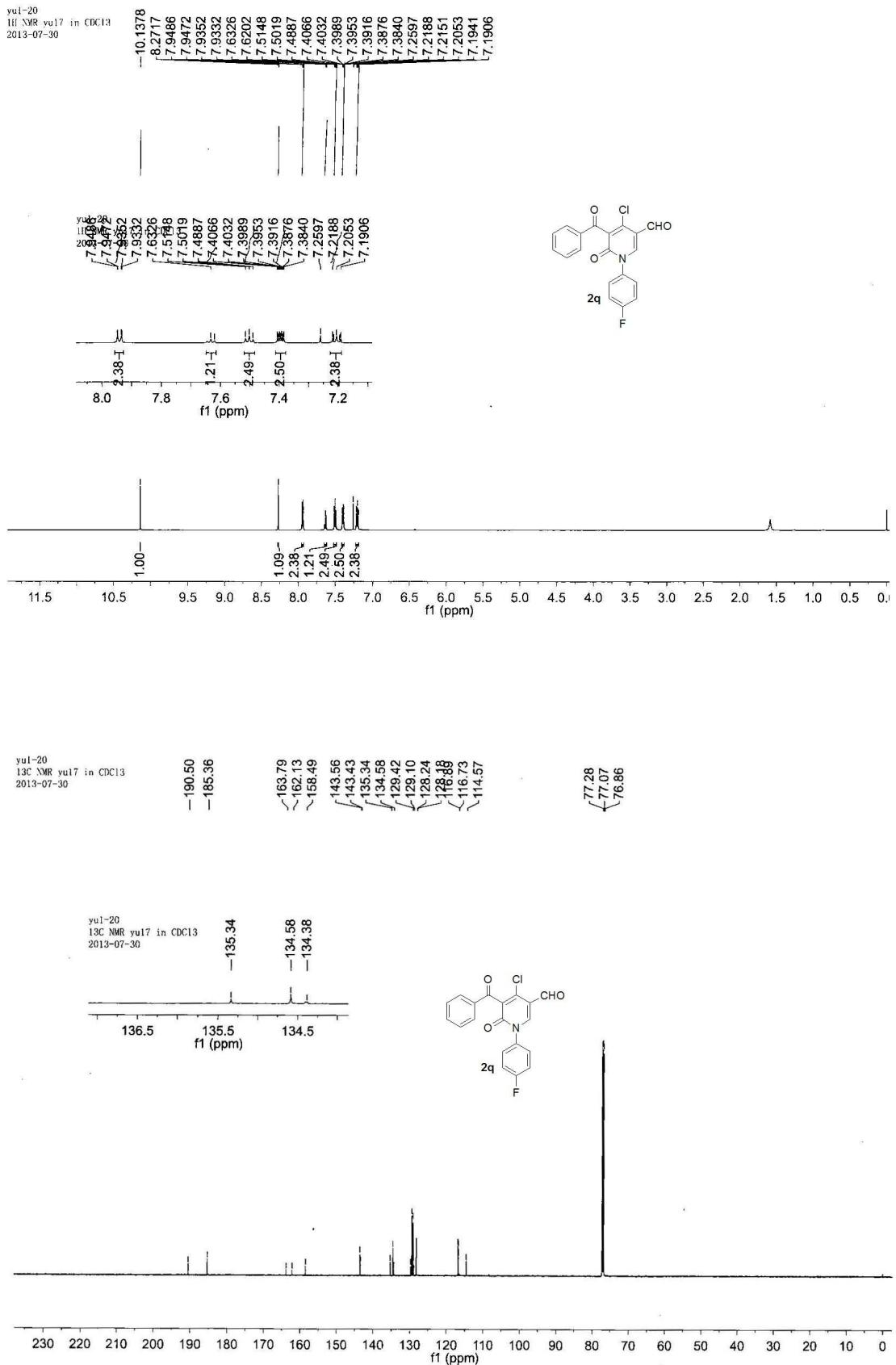


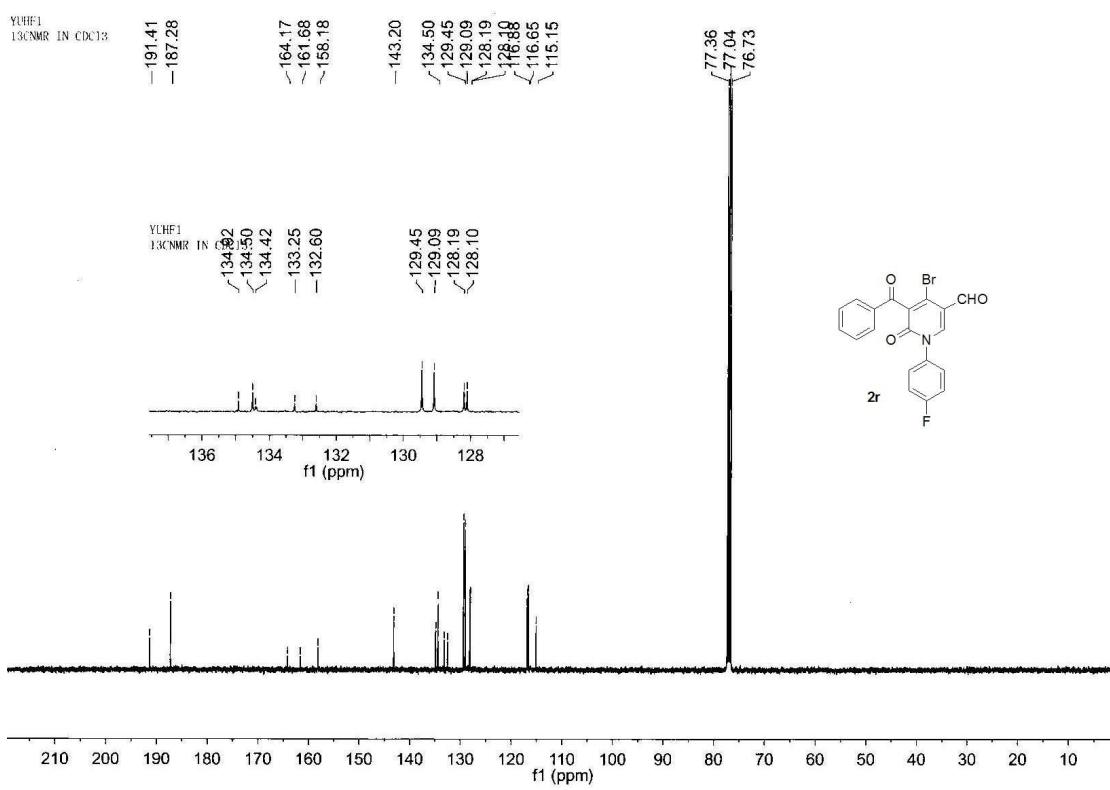
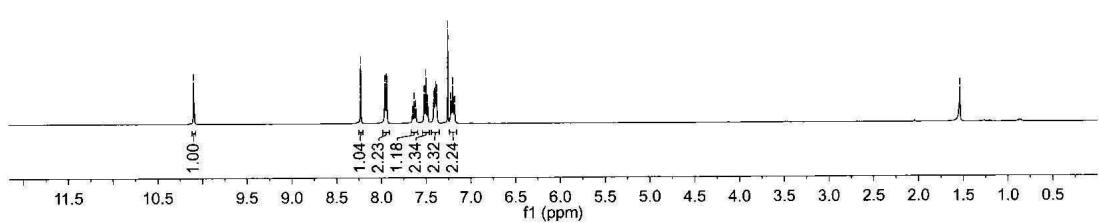
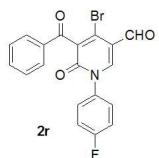
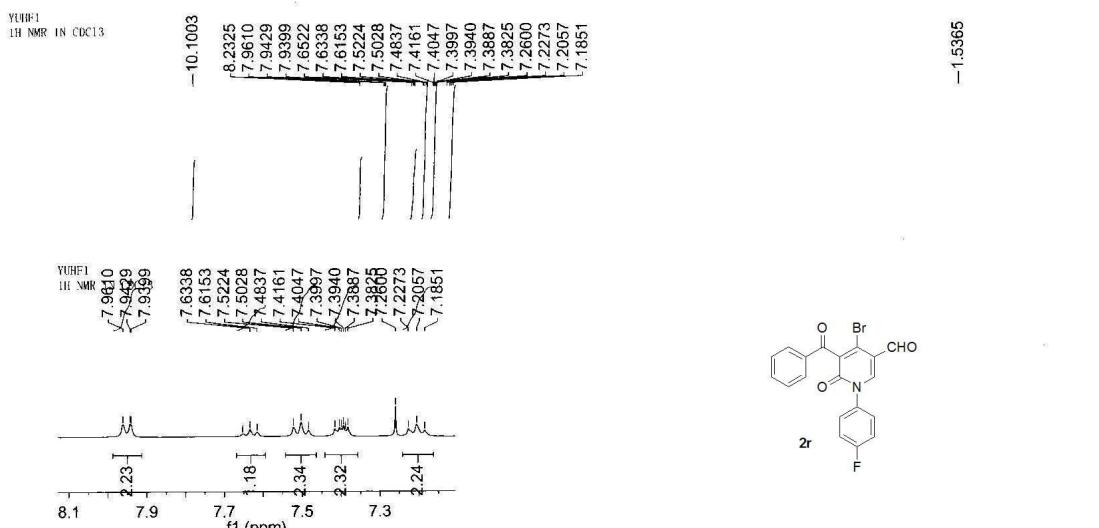
yul-20
III NMR yu16 in CDCl₃
2013-07-30

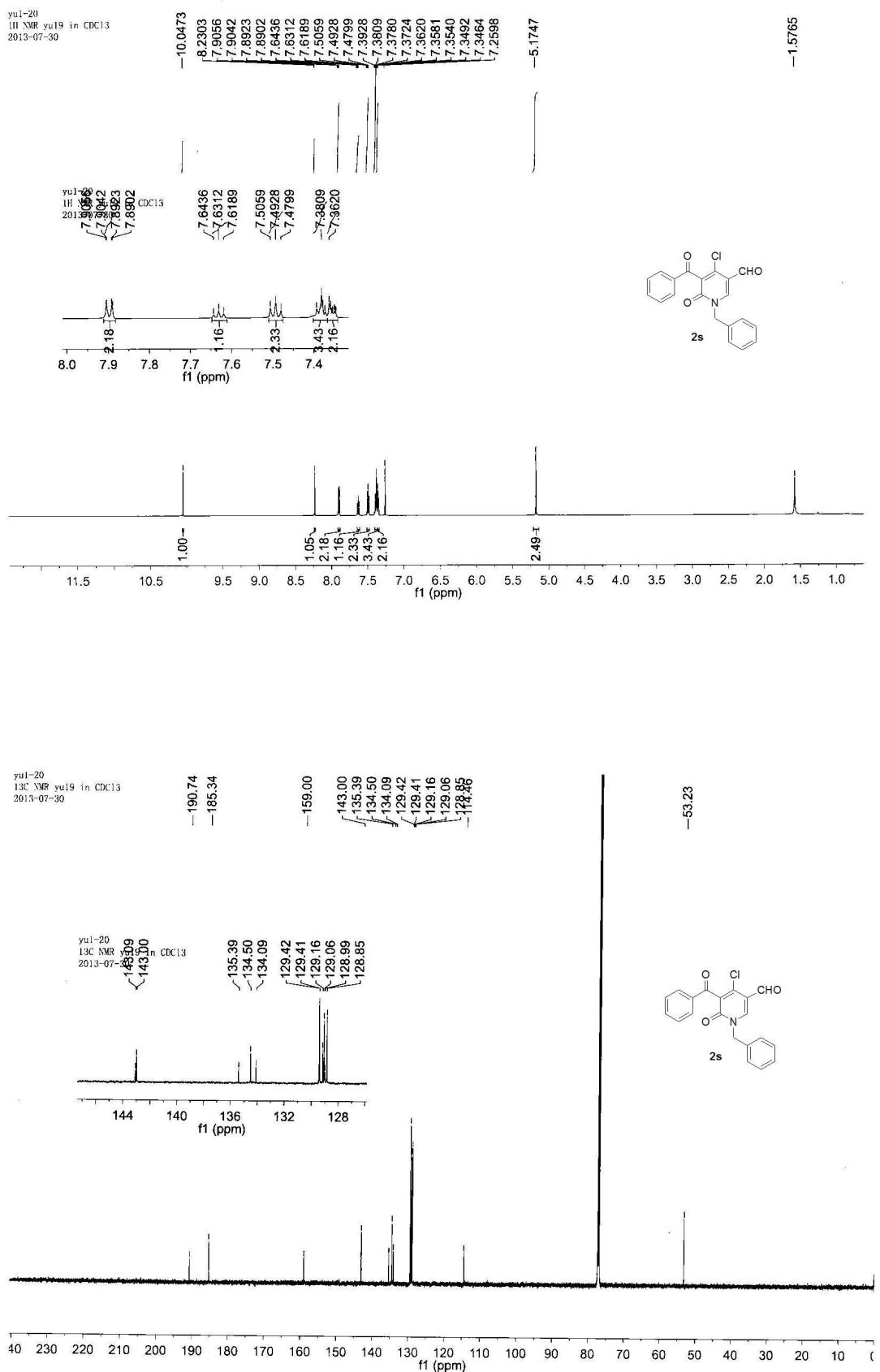


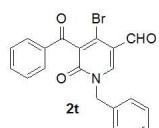
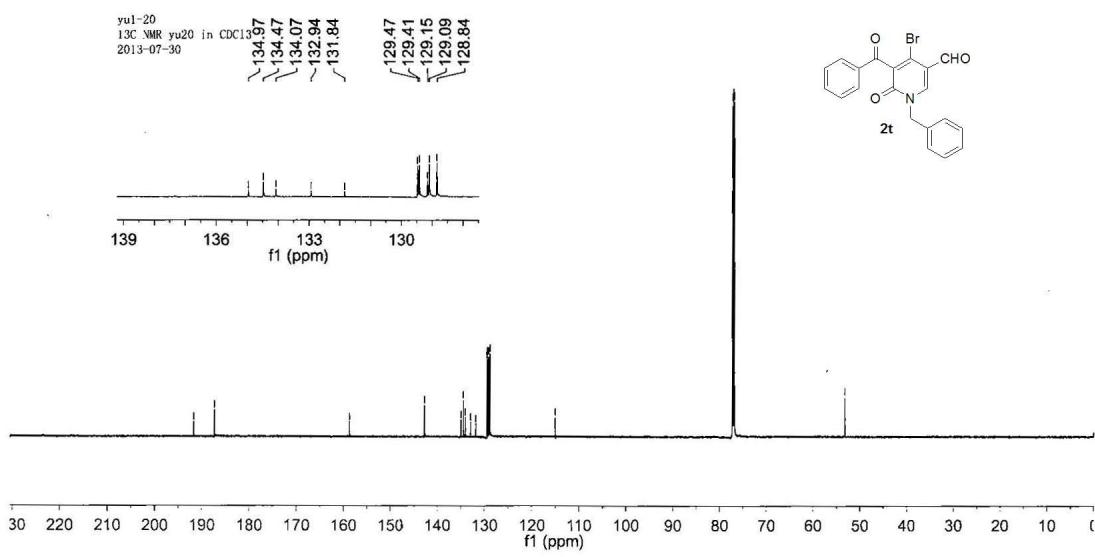
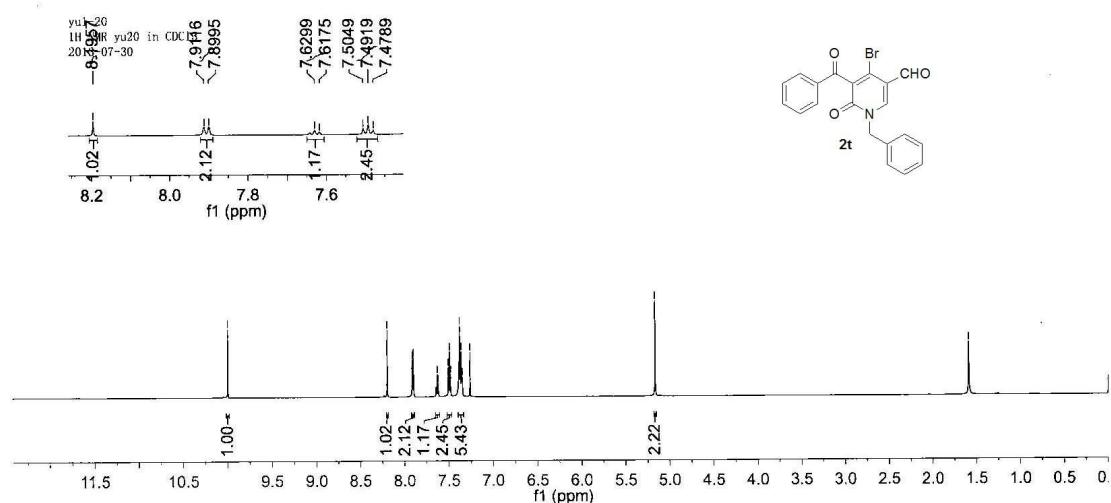
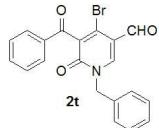
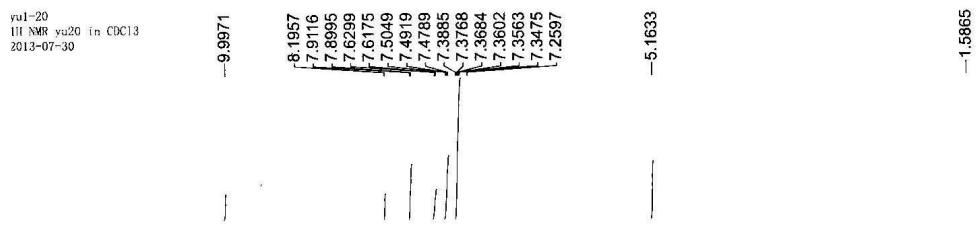
yul-20
13C NMR yu16 in CDCl₃
2013-07-30



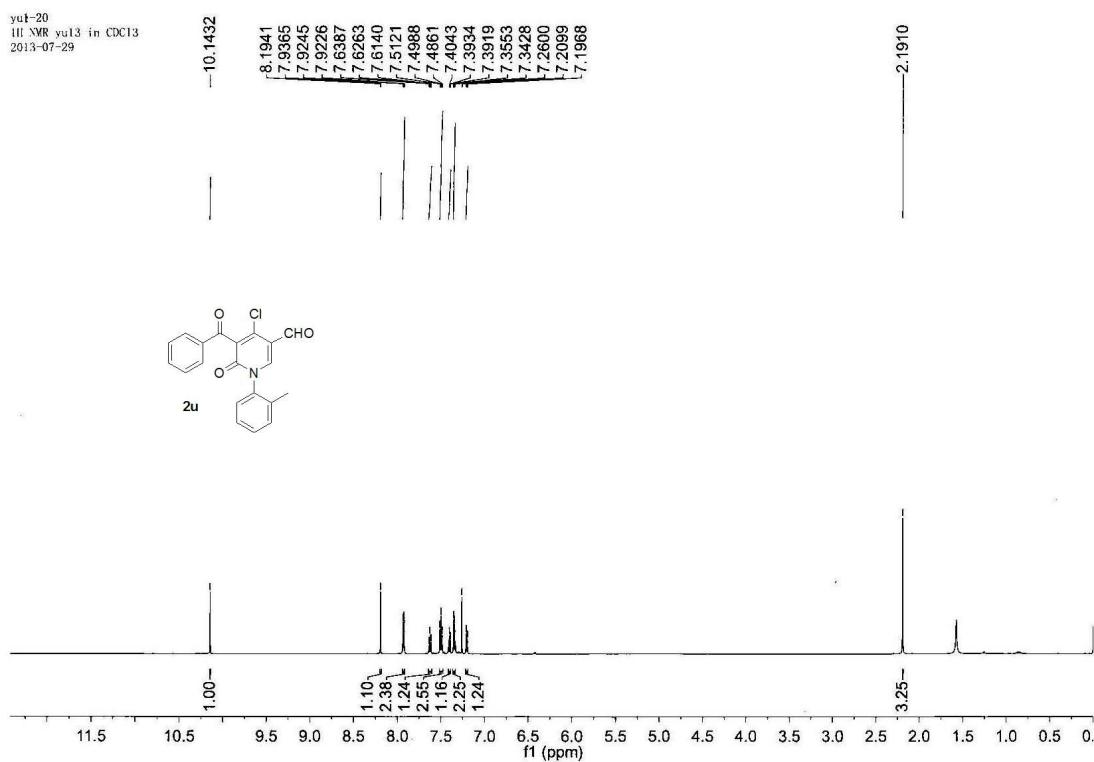




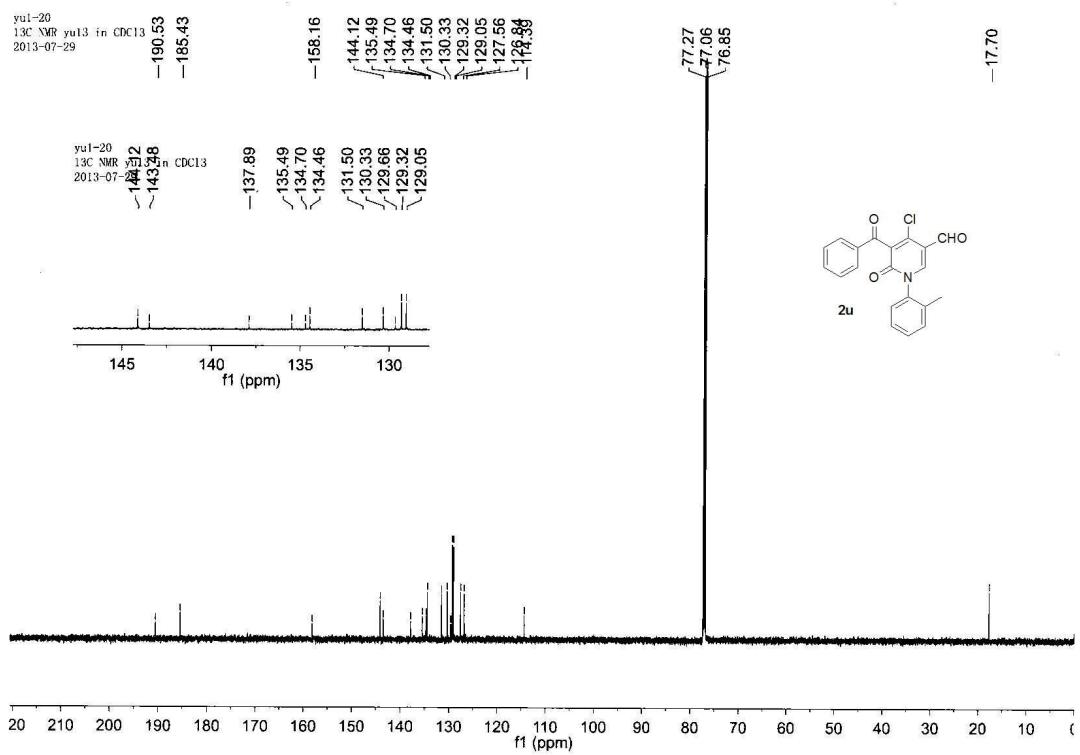




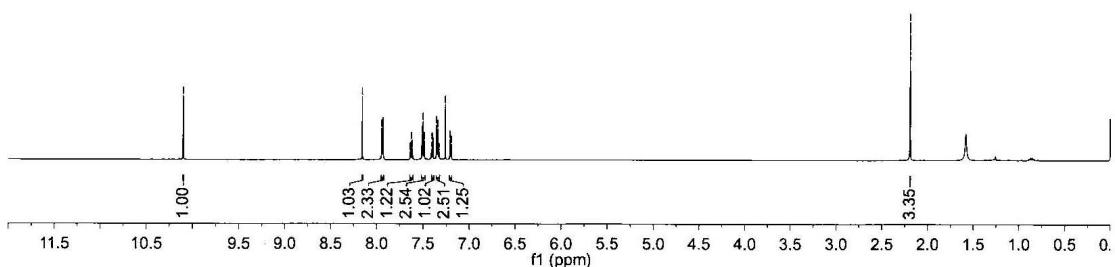
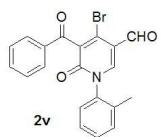
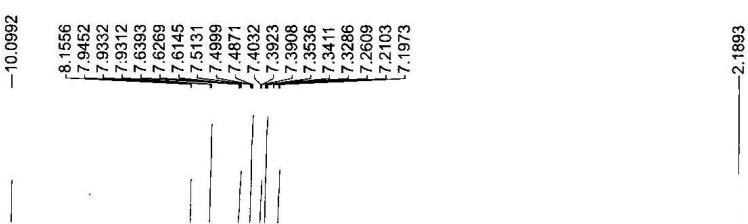
yul-20
1H NMR yul13 in CDCl₃
2013-07-29



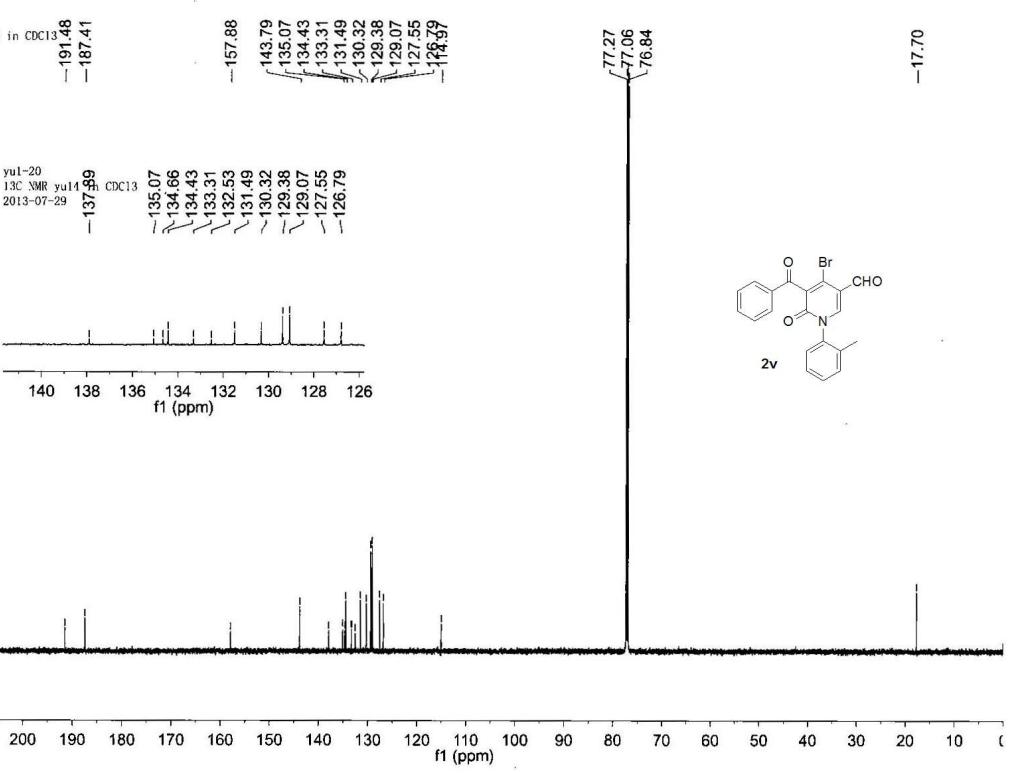
yul-20
13C NMR yul13 in CDCl₃
2013-07-29



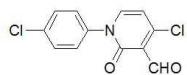
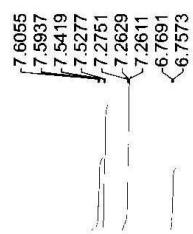
yu1-20
1H NMR yu14 in CDCl₃
2013-07-29



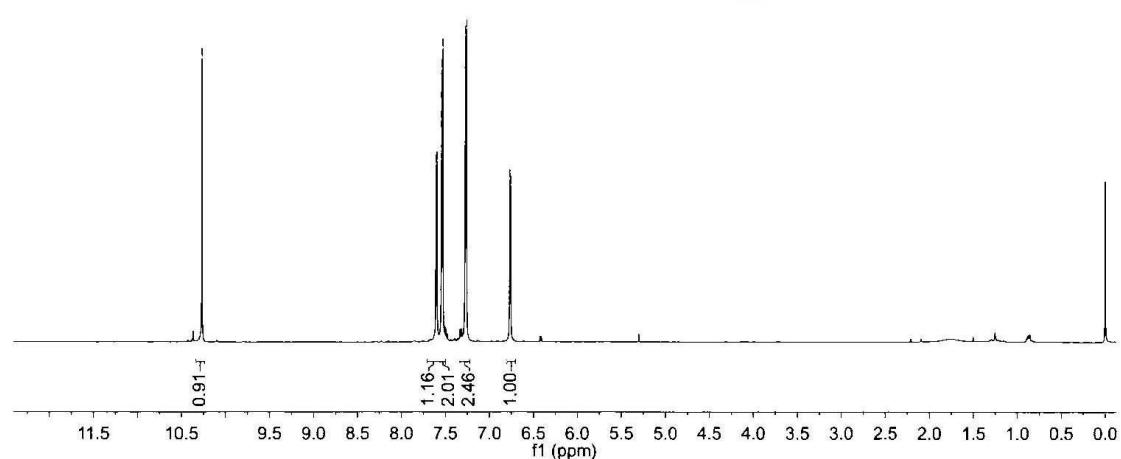
yu1-20
13C NMR yu14 in CDCl₃
2013-07-29



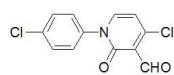
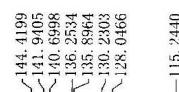
vhf56
1H NMR yuhf56 in CDCl₃
-10.26δ4



2w



vhf56
13C NMR yuhf56 in CDCl₃
-187.40δ00
-181.93δ33



2w

