

**Cyanation of α,β -unsaturated enones by malononitrile in open air
with metal-catalyst-free**

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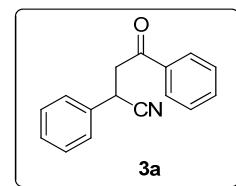
I. General

All reagents were purchased from commercial sources and used without treatment, unless otherwise indicated. The products were purified by column chromatography over silica gel. ^1H NMR and ^{13}C NMR spectra were recorded at 25 °C on a Varian 500 MHz and 125 MHz, respectively, and TMS as internal standard. IR spectra (KBr) were recorded on a Magna-560 FTIR spectrophotometer in the range of 400~4000 cm⁻¹. Elemental analyses were measured on an E-2400 analyzer (Perkin-Elmer). Mass spectra were recorded on Agilent 1100 LCMsD mass spectrometer.

II. Synthesis and analytical data of 3.

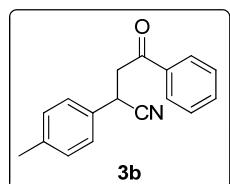
General procedure for the preparation of **3** (**3a** as an example): To a solution of (*E*)-chalcone **1a** (208 mg, 1.0 mmol) in DMF (4 mL) was added malononitrile (73 mg, 1.1 mmol) and K₂CO₃ (152 mg, 1.1 mmol). The reaction mixture was stirred at room temperature for 12 h. After completion of the reaction, the solution poured into water and then extracted with CH₂Cl₂ (3 × 10 mL). The combined organic phase was washed with water (3 × 10 mL), dried over anhydrous MgSO₄, filtered and concentrated under reduced pressure. The crude product was purified by flash chromatography (silica gel, petroleum ether: diethyl ether = 9: 1) to give **3a** (223 mg, 95%) as a white solid.

4-oxo-2,4-diphenylbutanenitrile (**3a**)



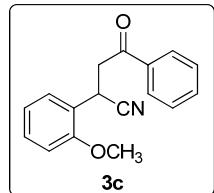
White solid. m.p. 120-122 °C. ^1H NMR (500 MHz, CDCl₃): δ = 3.49-3.54 (m, 1H), 3.71-3.77 (m, 1H), 4.56-4.59 (m, 1H), 7.34-7.36 (d, J = 7.0 Hz, 1H), 7.38-7.44 (m, 2H), 7.45-7.49 (m, 4H), 7.59-7.60 (d, J = 7.5 Hz, 1H), 7.92-7.94 (t, J = 7.0 Hz, 2H); ^{13}C NMR (CDCl₃, 125 MHz): δ = 31.8, 44.5, 120.6, 127.5, 128.1, 128.4, 128.8, 129.3, 133.9, 135.2, 135.6, 194.6. MS calcd m/z 235.1, found 236.1 [(M + 1)]⁺. Anal. Calcd for C₁₆H₁₃NO: C, 81.68; H, 5.57; N, 5.95; Found: C, 81.75; H, 5.59; N, 6.01.

4-oxo-4-phenyl-2-(*p*-tolyl)butanenitrile (3b)



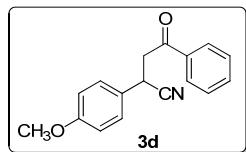
White solid. m.p. 65-67 °C. ^1H NMR (500 MHz, CDCl_3): δ = 2.35 (s, 3H), 3.47-3.52 (m, 1H), 3.69-3.74 (m, 1H), 4.52-4.55 (m, 1H), 7.19-7.20 (d, J = 8.0 Hz, 2H), 7.31-7.33 (d, J = 8.5 Hz, 2H), 7.46-7.49 (t, J = 7.5 Hz, 2H), 7.58-7.61 (t, J = 7.5 Hz, 1H), 7.92-7.93 (d, J = 7.5 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 21.1, 31.5, 44.5, 120.8, 127.3, 128.1, 128.8, 129.9, 132.2, 133.9, 135.6, 138.2, 194.7. MS calcd m/z 249.1, found 250.1 [(M + 1)] $^+$. Anal. Calcd for $\text{C}_{17}\text{H}_{15}\text{NO}$: C, 81.90; H, 6.06; N, 5.62; Found: C, 81.78; H, 6.01; N, 5.57.

2-(2-methoxyphenyl)-4-oxo-4-phenylbutanenitrile (3c)



White solid. m.p. 72-74 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.48-3.53 (m, 1H), 3.63-3.68 (m, 1H), 3.82 (s, 3H), 4.77-4.79 (m, 1H), 6.91-6.93 (d, J = 8.5 Hz, 1H), 6.99-7.02 (m, 1H), 7.32-7.35 (m, 1H), 7.45-7.47 (d, J = 7.5 Hz, 2H), 7.48-7.52 (m, 1H), 7.57-7.60 (m, 1H), 7.93-7.95 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 27.4, 42.1, 55.5, 110.9, 120.6, 121.0, 123.0, 128.0, 128.7, 128.9, 129.7, 133.7, 135.8, 156.2, 195.3. MS calcd m/z 265.1, found 266.1 [(M + 1)] $^+$. Anal. Calcd for $\text{C}_{17}\text{H}_{15}\text{NO}_2$: C, 76.96; H, 5.70; N, 5.28; Found: C, 77.04; H, 5.68; N, 5.21.

2-(4-methoxyphenyl)-4-oxo-4-phenylbutanenitrile (3d)

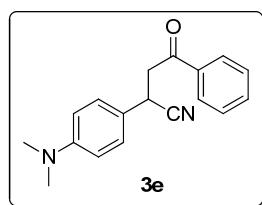


White solid. m.p. 107-109 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.47-3.52 (m, 1H), 3.67-3.72 (m, 1H), 3.80 (s, 3H), 4.51-4.54 (t, J = 7.0 Hz, 1H), 6.90-6.91 (d, J = 7.0 Hz,

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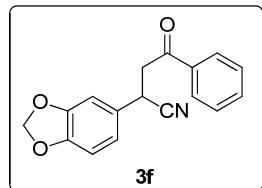
2H), 7.34-7.36 (d, J = 9.0 Hz, 2H), 7.45-7.49 (t, J = 8.0 Hz, 2H), 7.58-7.61 (t, J = 7.0 Hz, 1H), 7.92-7.93 (d, J = 8.0 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 31.1, 44.6, 55.4, 114.6, 120.9, 127.2, 128.1, 128.2, 128.9, 132.9, 135.8, 159.6, 194.8. MS calcd m/z 265.1, found 266.1 [(M + 1)] $^+$. Anal. Calcd for $\text{C}_{17}\text{H}_{15}\text{NO}_2$: C, 76.96; H, 5.70; N, 5.28; Found: C, 77.08; H, 5.67; N, 5.20.

2-(4-(dimethylamino)phenyl)-4-oxo-4-phenylbutanenitrile (3e)



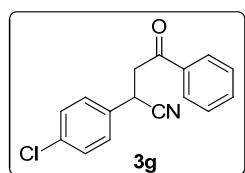
Yellow solid. m.p. 87-89 °C. ^1H NMR (500 MHz, CDCl_3): δ = 2.95 (s, 6H), 3.45-3.49 (m, 1H), 3.65-3.71 (m, 1H), 4.45-4.48 (m, 1H), 6.69-6.71 (d, J = 8.0 Hz, 2H), 7.26-7.28 (t, J = 9.0 Hz, 2H), 7.45-7.48 (t, J = 8.0 Hz, 2H), 7.57-7.59 (d, J = 7.5 Hz, 1H), 7.91-7.93 (t, J = 7.05 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 31.1, 40.5, 44.7, 112.8, 121.3, 128.1, 128.3, 128.8, 133.8, 135.9, 150.3, 195.1. MS calcd m/z 278.1, found 279.1 [(M + 1)] $^+$. Anal. Calcd for $\text{C}_{18}\text{H}_{18}\text{N}_2\text{O}$: C, 77.67; H, 6.52; N, 10.06; Found: C, 77.76; H, 6.55; N, 10.13.

2-(benzo[d][1,3]dioxol-5-yl)-4-oxo-4-phenylbutanenitrile (3f)



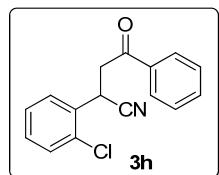
White solid. m.p. 126-128 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.48-3.52 (m, 1H), 3.66-3.72 (m, 1H), 4.48-4.50 (t, J = 7.5 Hz, 1H), 5.99 (s, 2H), 6.79-6.81 (d, J = 8.0 Hz, 1H), 6.89-6.91 (t, J = 7.5 Hz, 2H), 7.47-7.50 (t, J = 8.0 Hz, 2H), 7.59-7.62 (t, J = 7.5 Hz, 1H), 7.92-7.94 (d, J = 7.5 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 31.5, 44.5, 101.5, 107.9, 108.7, 120.7, 121.0, 128.1, 128.7, 128.8, 133.9, 135.6, 147.6, 148.3, 194.6. MS calcd m/z 279.1, found 280.1 [(M + 1)] $^+$. Anal. Calcd for $\text{C}_{17}\text{H}_{13}\text{NO}_3$: C, 73.11; H, 4.69; N, 5.02; Found: C, 73.02; H, 4.66; N, 4.95.

2-(4-chlorophenyl)-4-oxo-4-phenylbutanenitrile (3g)



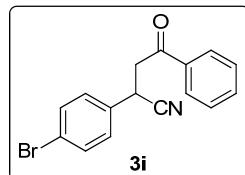
White solid. m.p. 112-114 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.49-3.54 (m, 1H), 3.69-3.74 (m, 1H), 4.55-4.58 (t, J = 7.0 Hz, 1H), 7.36-7.40 (m, 4H), 7.47-7.50 (m, 2H), 7.59-7.63 (m, 1H), 7.91-7.93 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 31.2, 44.3, 120.3, 128.5, 128.9, 128.9, 129.4, 133.7, 134.0, 134.4, 135.4, 194.3. MS calcd m/z 269.0, found 270.0 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{ClNO}$: C, 71.25; H, 4.48; N, 5.19; Found: C, 71.35; H, 4.50; N, 5.24.

2-(2-chlorophenyl)-4-oxo-4-phenylbutanenitrile (3h)



White solid. m.p. 91-93 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.52-3.56 (m, 1H), 3.65-3.71 (m, 1H), 4.92-4.95 (m, 1H), 7.31-7.38 (m, 2H), 7.43-7.45 (m, 1H), 7.47-7.50 (m, 2H), 7.59-7.63 (m, 1H), 7.68-7.70 (m, 1H), 7.95-7.97 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 30.1, 42.4, 119.7, 127.8, 128.1, 128.9, 129.5, 129.9, 130.3, 132.7, 133.9, 135.6, 194.4. MS calcd m/z 269.0, found 270.0 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{ClNO}$: C, 71.25; H, 4.48; N, 5.19; Found: C, 71.16; H, 4.45; N, 5.13.

2-(4-bromophenyl)-4-oxo-4-phenylbutanenitrile (3i)

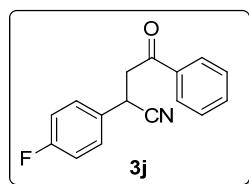


White solid. m.p. 126-128 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.49-3.53 (m, 1H),

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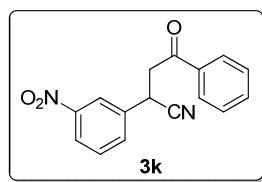
3.69-3.74 (m, 1H), 4.53-4.56 (t, $J = 7.0$ Hz, 1H), 7.31-7.32 (d, $J = 8.5$ Hz, 2H), 7.46-7.53 (m, 4H), 7.59-7.62 (t, $J = 7.5$ Hz, 1H), 7.91-7.92 (t, $J = 7.5$ Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): $\delta = 31.4, 44.2, 120.2, 122.5, 128.1, 128.9, 129.3, 132.4, 134.1, 134.3, 135.5, 194.3$. MS calcd m/z 313.0, found 314.0 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{BrNO}$: C, 61.17; H, 3.85; N, 4.46; Found: C, 61.28; H, 3.87; N, 4.51.

2-(4-fluorophenyl)-4-oxo-4-phenylbutanenitrile (3j)



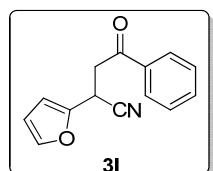
White solid. m.p. 105-107 °C. ^1H NMR (500 MHz, CDCl_3): $\delta = 3.49\text{-}3.54$ (m, 1H), 3.69-3.74 (m, 1H), 4.56-4.59 (t, $J = 7.0$ Hz, 1H), 7.07-7.10 (t, $J = 8.5$ Hz, 2H), 7.41-7.44 (m, 2H), 7.46-7.50 (t, $J = 7.5$ Hz, 2H), 7.59-7.62 (t, $J = 7.0$ Hz, 1H), 7.91-7.92 (d, $J = 8.0$ Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): $\delta = 31.2, 44.4, 116.2, 116.4, 120.5, 128.1, 128.9, 129.3, 129.4, 134.0, 135.6, 161.6, 163.5, 194.4$. MS calcd m/z 253.1, found 254.1 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{FNO}$: C, 75.88; H, 4.78; N, 5.53; Found: C, 75.99; H, 4.81; N, 5.58.

2-(3-nitrophenyl)-4-oxo-4-phenylbutanenitrile (3k)



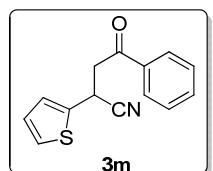
White solid. m.p. 128-130 °C. ^1H NMR (500 MHz, CDCl_3): $\delta = 3.59\text{-}3.64$ (m, 1H), 3.78-3.83 (m, 1H), 4.71-4.74 (t, $J = 7.0$ Hz, 1H), 7.47-7.50 (t, $J = 7.5$ Hz, 2H), 7.59-7.63 (m, 2H), 7.84-7.85 (d, $J = 8.0$ Hz, 1H), 7.92-7.94 (d, $J = 7.5$ Hz, 2H), 8.21-8.23 (d, $J = 8.5$ Hz, 1H), 8.34 (s, 1H); ^{13}C NMR (CDCl_3 , 125 MHz): $\delta = 31.6, 43.9, 119.6, 122.8, 123.6, 128.1, 130.0, 130.4, 133.9, 134.3, 135.3, 137.3, 193.8$. MS calcd m/z 280.1, found 281.1 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{N}_2\text{O}_3$: C, 68.56; H, 4.32; N, 9.99; Found: C, 68.66; H, 4.36; N, 10.06.

2-(furan-2-yl)-4-oxo-4-phenylbutanenitrile (3l)



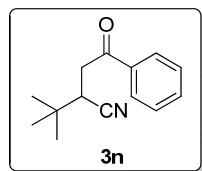
White solid. m.p. 66-68 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.66-3.74 (m, 2H), 4.67-4.70 (t, J = 6.5 Hz, 1H), 6.36-6.40 (m, 2H), 7.39-7.40 (d, J = 1.0 Hz, 1H), 7.48-7.52 (t, J = 8.0 Hz, 2H), 7.61-7.64 (t, J = 7.0 Hz, 1H), 7.96-7.98 (d, J = 8.0 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 25.9, 40.6, 108.2, 110.8, 118.3, 128.1, 128.8, 134.0, 135.5, 143.1, 146.8, 194.2. MS calcd m/z 225.0, found 226.0 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{14}\text{H}_{11}\text{NO}_2$: C, 74.65; H, 4.92; N, 6.22; Found: C, 74.54; H, 4.89; N, 6.16.

4-oxo-4-phenyl-2-(thiophen-2-yl)butanenitrile (3m)



White solid. m.p. 111-113 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.61-3.66 (m, 1H), 3.75-3.80 (m, 1H), 4.85-4.88 (t, J = 6.5 Hz, 1H), 6.98-6.99 (m, 1H), 7.16-7.17 (m, 1H), 7.27-7.29 (m, 1H), 7.48-7.51 (m, 2H), 7.60-7.64 (m, 1H), 7.95-7.96 (m, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 27.3, 44.6, 119.8, 125.9, 126.7, 127.2, 127.6, 128.2, 128.9, 134.1, 135.6, 137.0, 194.3. MS calcd m/z 241.0, found 242.0 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{14}\text{H}_{11}\text{NOS}$: C, 69.68; H, 4.59; N, 5.80; Found: C, 69.59; H, 4.56; N, 5.74.

2-(tert-butyl)-4-oxo-4-phenylbutanenitrile (3n)

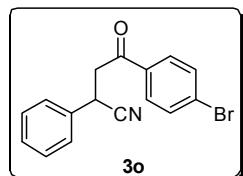


White solid. m.p. 106-108 °C. ^1H NMR (500 MHz, CDCl_3): δ = 1.13 (s, 9H), 3.16-3.21 (m, 2H), 3.34-3.40 (m, 1H), 7.49-7.52 (t, J = 8.0 Hz, 2H), 7.60-7.61 (d, J =

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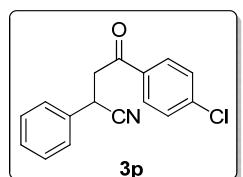
7.0 Hz, 1H), 7.97-7.98 (t, J = 7.0 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 27.4, 32.8, 36.9, 37.8, 121.1, 128.1, 128.8, 133.8, 136.1, 195.8. MS calcd m/z 215.1, found 216.1 [(M + 1)]⁺. Anal. Calcd for $\text{C}_{14}\text{H}_{17}\text{NO}$: C, 78.10; H, 7.96; N, 6.51; Found: C, 78.18; H, 7.98; N, 6.56.

4-(4-bromophenyl)-4-oxo-2-phenylbutanenitrile (3o)



White solid. m.p. 120-122 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.44-3.49 (m, 1H), 3.67-3.72 (m, 1H), 4.53-4.56 (m, 1H), 7.33-7.36 (t, J = 6.5 Hz, 1H), 7.39-7.44 (m, 4H), 7.61-7.63 (d, J = 8.5 Hz, 2H), 7.78-7.80 (d, J = 8.5 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 31.8, 44.4, 120.4, 127.4, 128.3, 128.4, 129.0, 129.3, 129.5, 132.5, 134.2, 134.9, 193.7. MS calcd m/z 313.0, found 314.0 [(M + 1)]⁺. Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{BrNO}$: C, 61.17; H, 3.85; N, 4.46; Found: C, 61.28; H, 3.87; N, 4.52.

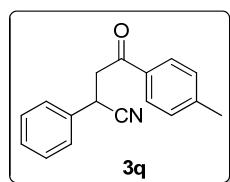
4-(4-chlorophenyl)-4-oxo-2-phenylbutanenitrile (3p)



White solid. m.p. 111-113 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.44-3.49 (m, 1H), 3.67-3.72 (m, 1H), 4.53-4.55 (m, 1H), 7.32-7.22 (d, J = 4.5 Hz, 1H), 7.34-7.36 (m, 2H), 7.38-7.43 (m, 4H), 7.85-7.87 (d, J = 8.5 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 31.9, 44.5, 120.5, 127.5, 128.5, 129.2, 134.0, 135.1, 140.5, 193.5. MS calcd m/z 269.0, found 270.0 [(M + 1)]⁺. Anal. Calcd for $\text{C}_{16}\text{H}_{12}\text{ClNO}$: C, 71.25; H, 4.48; N, 5.19; Found: C, 71.36; H, 4.51; N, 5.24.

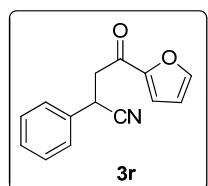
4-oxo-2-phenyl-4-(*p*-tolyl)butanenitrile (3q)

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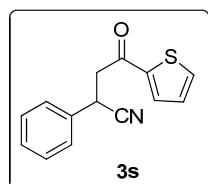
White solid. m.p. 64-66 °C. ^1H NMR (500 MHz, CDCl_3): δ = 2.41 (s, 3H), 3.46-3.51 (m, 1H), 3.68-4.703 (m, 1H), 4.55-4.58 (m, 1H), 7.25-7.27 (t, J = 8.0 Hz, 2H), 7.33-7.35 (d, J = 7.5 Hz, 1H), 7.38-7.41 (t, J = 7.0 Hz, 2H), 7.43-7.44 (d, J = 7.0 Hz, 2H), 7.82-7.83 (d, J = 8.5 Hz, 2H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 21.7, 31.9, 44.4, 120.7, 127.5, 128.2, 129.5, 133.3, 135.4, 144.9, 194.2. MS calcd m/z 249.10, found 250.1 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{17}\text{H}_{15}\text{NO}$: C, 81.90; H, 6.06; N, 5.62; Found: C, 81.99; H, 6.09; N, 5.68.

4-(furan-2-yl)-4-oxo-2-phenylbutanenitrile (3r)



White solid. m.p. 78-80 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.35-3.40 (m, 1H), 3.56-3.61 (m, 1H), 4.52-4.54 (t, J = 7.5 Hz, 1H), 6.55-6.56 (m, 1H), 7.23-7.26 (d, J = 3.5 Hz, 1H), 7.31-7.34 (t, J = 7.5 Hz, 1H), 7.36-7.42 (m, 4H), 7.58 (s, 1H); ^{13}C NMR (CDCl_3 , 125 MHz): δ = 31.5, 43.9, 112.7, 118.1, 120.4, 127.5, 128.4, 129.3, 135.0, 147.0, 151.8, 183.7. MS calcd m/z 225.1 found 226.1 $[(\text{M} + 1)]^+$. Anal. Calcd for $\text{C}_{14}\text{H}_{11}\text{NO}_2$: C, 74.65; H, 4.92; N, 6.22; Found: C, 74.76; H, 4.94; N, 6.27.

4-oxo-2-phenyl-4-(thiophen-2-yl)butanenitrile (3s)

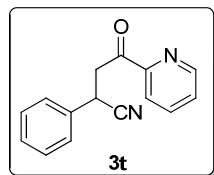


White solid. m.p. 91-93 °C. ^1H NMR (500 MHz, CDCl_3): δ = 3.42-3.47 (m, 1H), 3.63-3.68 (m, 1H), 4.55-4.58 (m, 1H), 7.13-7.14 (t, J = 4.0 Hz, 1H), 7.32-7.35 (m,

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1H), 7.35-7.41 (m, 2H), 7.42-7.44 (m, 2H), 7.68-7.69 (d, $J = 4.0$ Hz, 2H); ^{13}C NMR (CDCl₃, 125 MHz): $\delta = 32.0, 44.9, 120.4, 127.5, 127.5, 129.3, 132.6, 134.8, 135.0, 142.7, 187.4$. MS calcd m/z 241.0, found 242.0 [(M + 1)⁺]. Anal. Calcd for C₁₄H₁₁NOS: C, 69.68; H, 4.59; N, 5.80; Found: C, 69.58; H, 4.57; N, 5.72.

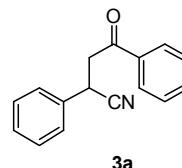
4-oxo-2-phenyl-4-(pyridin-2-yl)butanenitrile (3t)



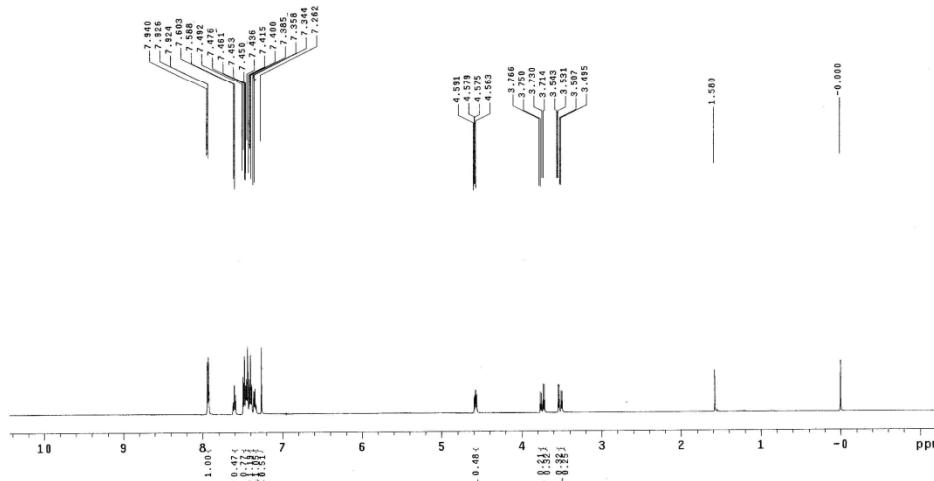
White solid. m.p. 88-90 °C. ^1H NMR (500 MHz, CDCl₃): $\delta = 3.78\text{-}3.83$ (m, 1H), 3.98-4.03 (m, 1H), 4.51-4.54 (m, 1H), 7.30-7.34 (m, 1H), 7.37-7.40 (m, 2H), 7.44-7.45 (m, 2H), 7.49-7.51 (m, 1H), 7.84-7.87 (m, 1H), 8.06-8.08 (m, 1H), 8.64-8.66 (m, 1H); ^{13}C NMR (CDCl₃, 125 MHz): $\delta = 31.9, 44.5, 120.5, 127.5, 128.5, 129.2, 134.0, 135.1, 140.5, 193.5$. MS calcd m/z 236.0, found 237.0 [(M + 1)⁺]. Anal. Calcd for C₁₅H₁₂N₂O: C, 76.25; H, 5.12; N, 11.86; Found: C, 76.36; H, 5.15; N, 11.94.

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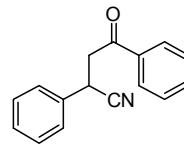
STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File size: 1000000
INNOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.092 sec
W1 10000 Hz
8 repetitions
OBSERVE: H1 499.8025913 MHz
DATA PROCESSING:
FT size 65536
Total time 6 min, 23 sec



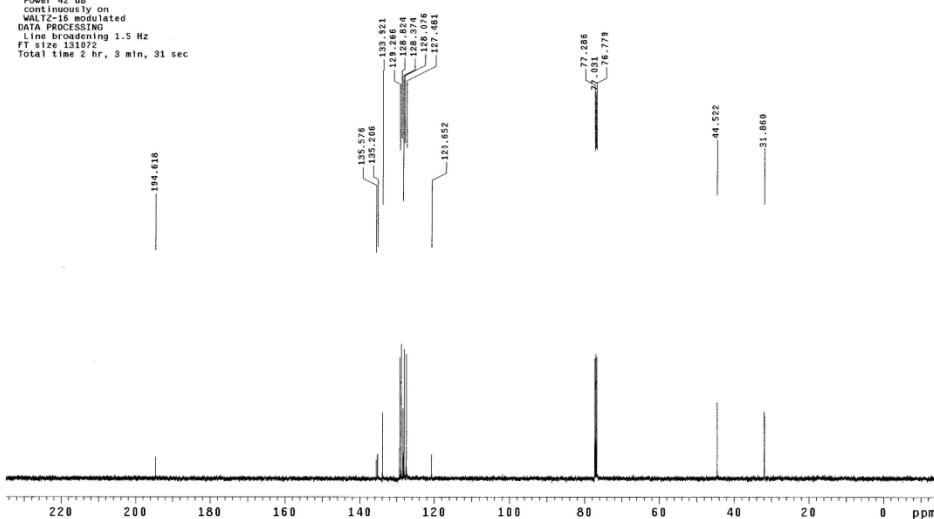
3a



STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
User: 1-14-87
File size: 1000000
INNOVA-500 "NENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.302 sec
W1 10000 Hz
64 repetitions
OBSERVE: C13 133.6756858 MHz
DECOUPLE: H1 499.8025913 MHz
Power 42 dB
Gated decoupling on
WALTZ-16 modulated
DATA PROCESSING:
L1 133.6756858 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec



3a



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```

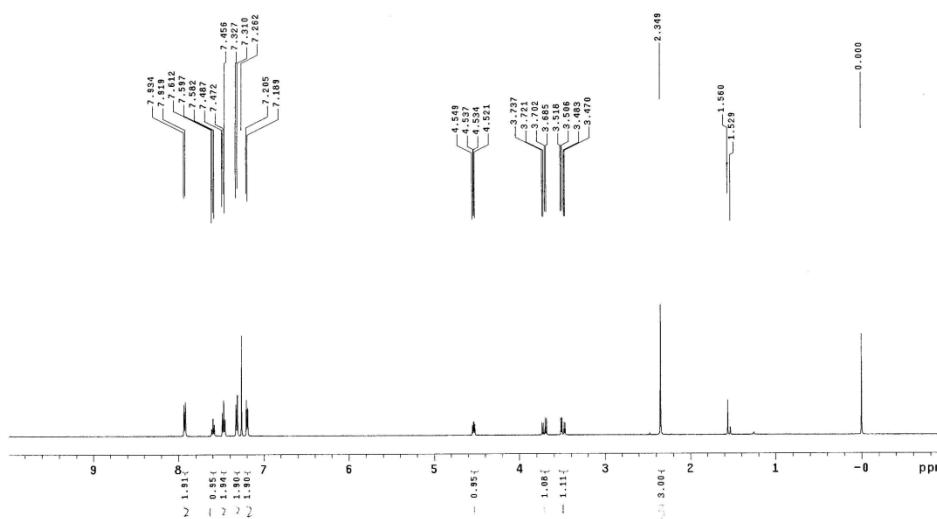
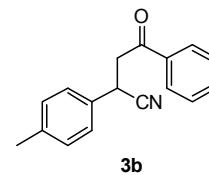
STANDARD PROTON PARAMETERS

Archive directory: /export/home/ouyy/vmrssys/data
Sample directory: 

Pulse Sequence: zgpu1
Solvent: CDCl3
Ambient temperature
File: c0932
INOVA-500 "NENU500"

Relax, delay 1.000 sec
Acq. time 0.000 sec
Width 8578.2 Hz
Sweep width 1000.0 Hz
OBSERVE: H1, 498.8025914 MHz
DATA PROCESSING
Total time 0 min, 23 sec

```



```

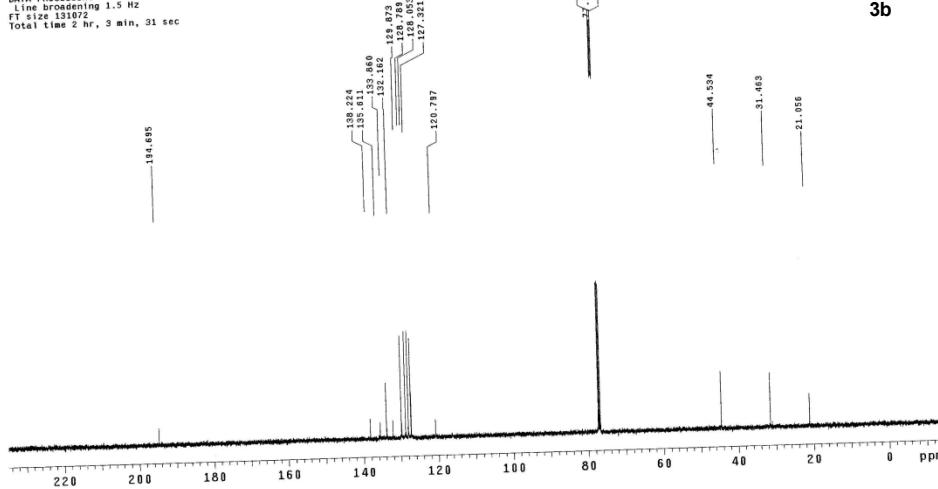
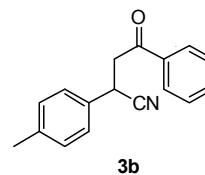
STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory: 

Put pulse sequence: z2pul
Pulse sequence: z2pul
Ambient temperature
User: 1-14-87
Instrument: INOVA-500 "NEMUSO" 500 MHz

Relax. delay 0.500 sec
Pulse 4.0 degrees
Acq. time 1.300 sec
Width 14250 Hz
128 repetitions
DOSSEERVE C13 1.6754873 MHz
DECIMATOR 489.035155 MHz
Power 42 dB
continuously
WIDENING 1600 Hz
DATA PROCESSING
line broadening 1.5 Hz
FT T1 1000000
Total time 2 hr, 3 min, 31 sec

```



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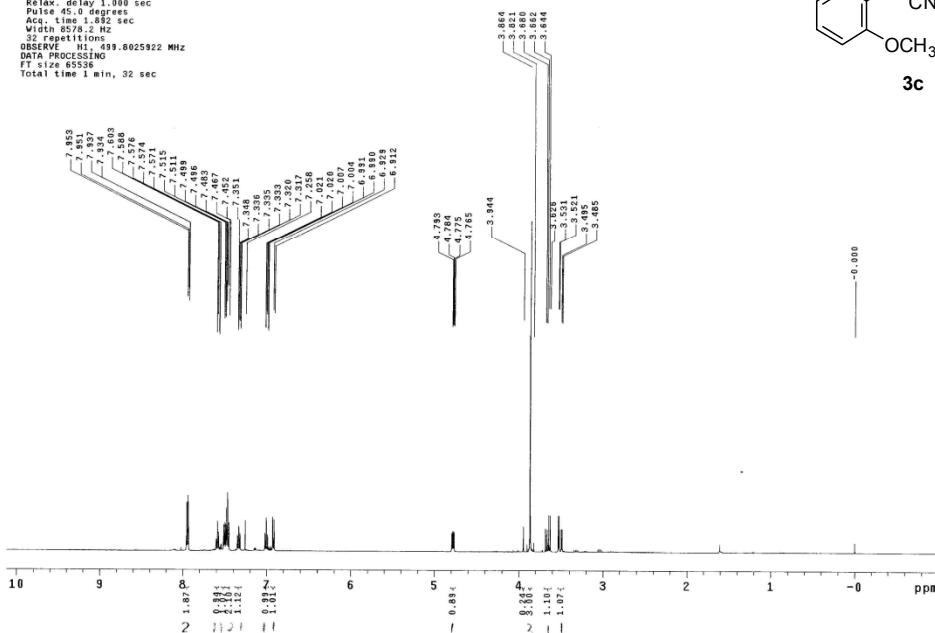
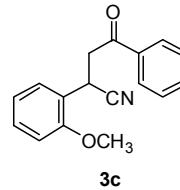
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STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vmrssys/datal
Sample directory: /data

Pulse Sequence: s2pul
Solvent: CDCl3
Ambient temperature
File ID: d6885
INIVNA-399 "NENUS99"

```

Max. delay 1.000 sec
 Pulse: 45.000 deg
 Acq. time 1.082 sec
 Water presat 1
 32 repetitions
 QBRWV1.1, 493.8025922 MHz
 DATA PROCESSING
 FT size 65536
 Total time 1 min, 32 sec



```

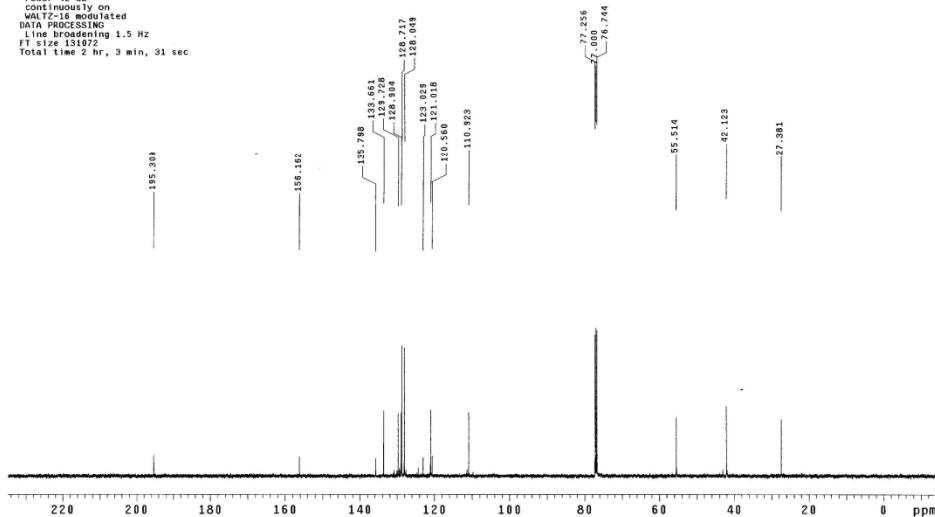
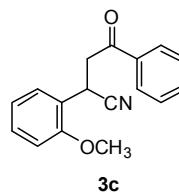
STANDARD CARBON PARAMETERS

Archive directory: /export/home/ouyy/vnmsys/data
Public directory: /vnmsys/public

Pulse Sequence: z2pul
Solvent: dcd13
Ambient temperature
User: i-14-87
Date: 1990-08-08
INVOA=500 "NENUOS"

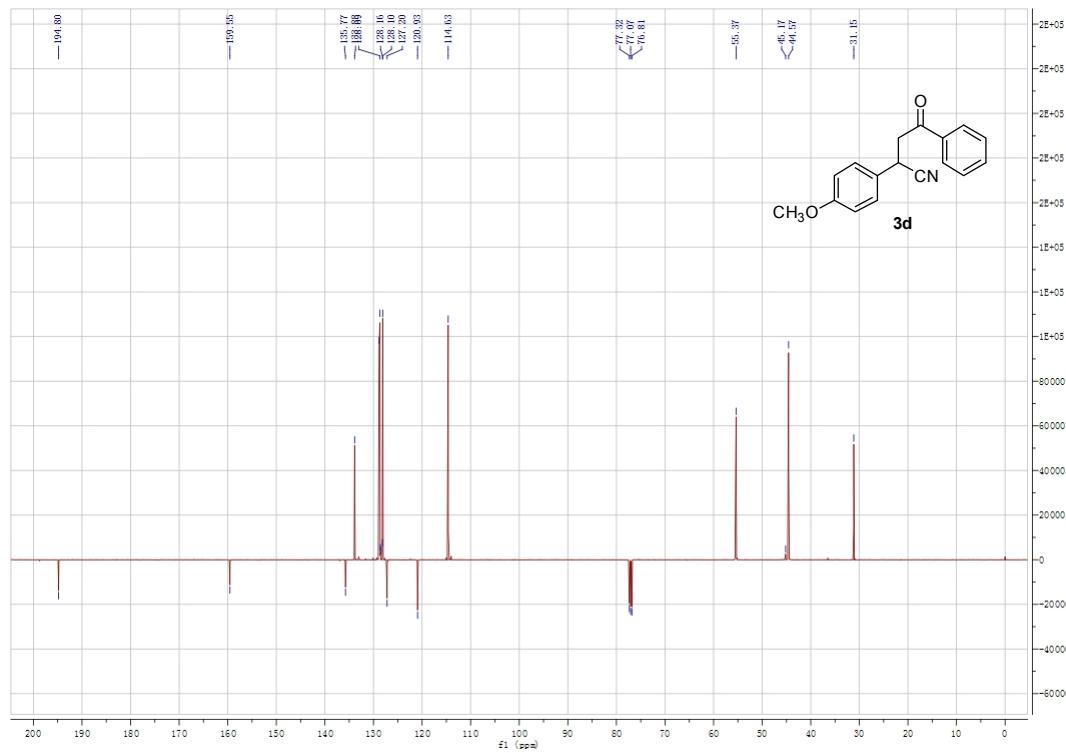
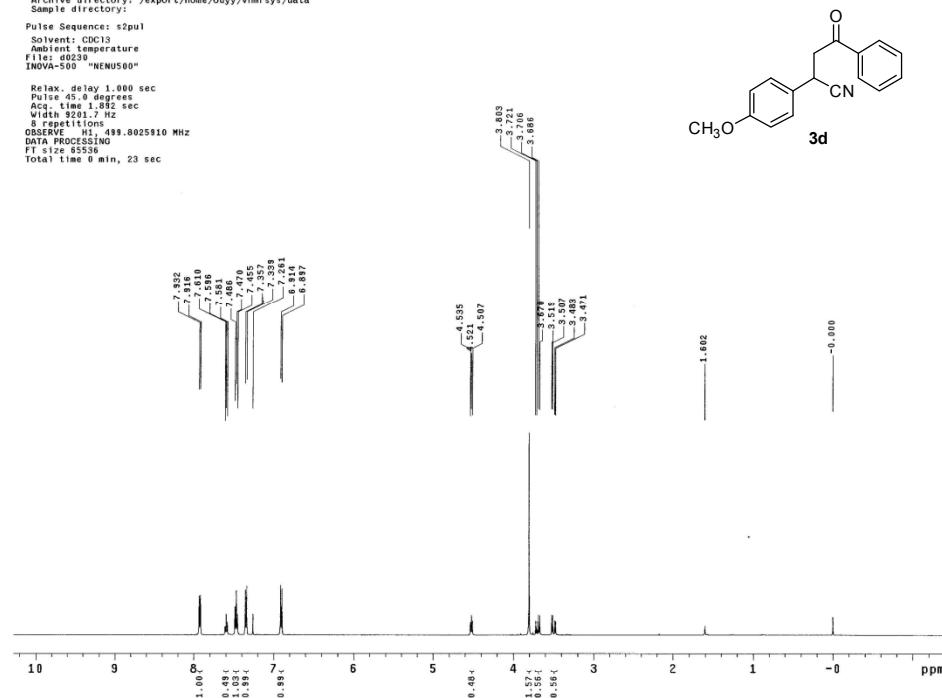
```

Pulse, delay 0.500 sec
Pulse 45.0 degrees
Aca. time 1.300 sec
With 3142.8 Hz
183.000000 MHz
OBSERVE C13, 125.674575 MHz
DECODE H1, 499.005095 MHz
Power 42
continuously on
unmodulated
DATA PROCESSING
Line broadening 1.5 Hz
(Fourier transform)
Total_time 2 hr, 3 min, 31 sec

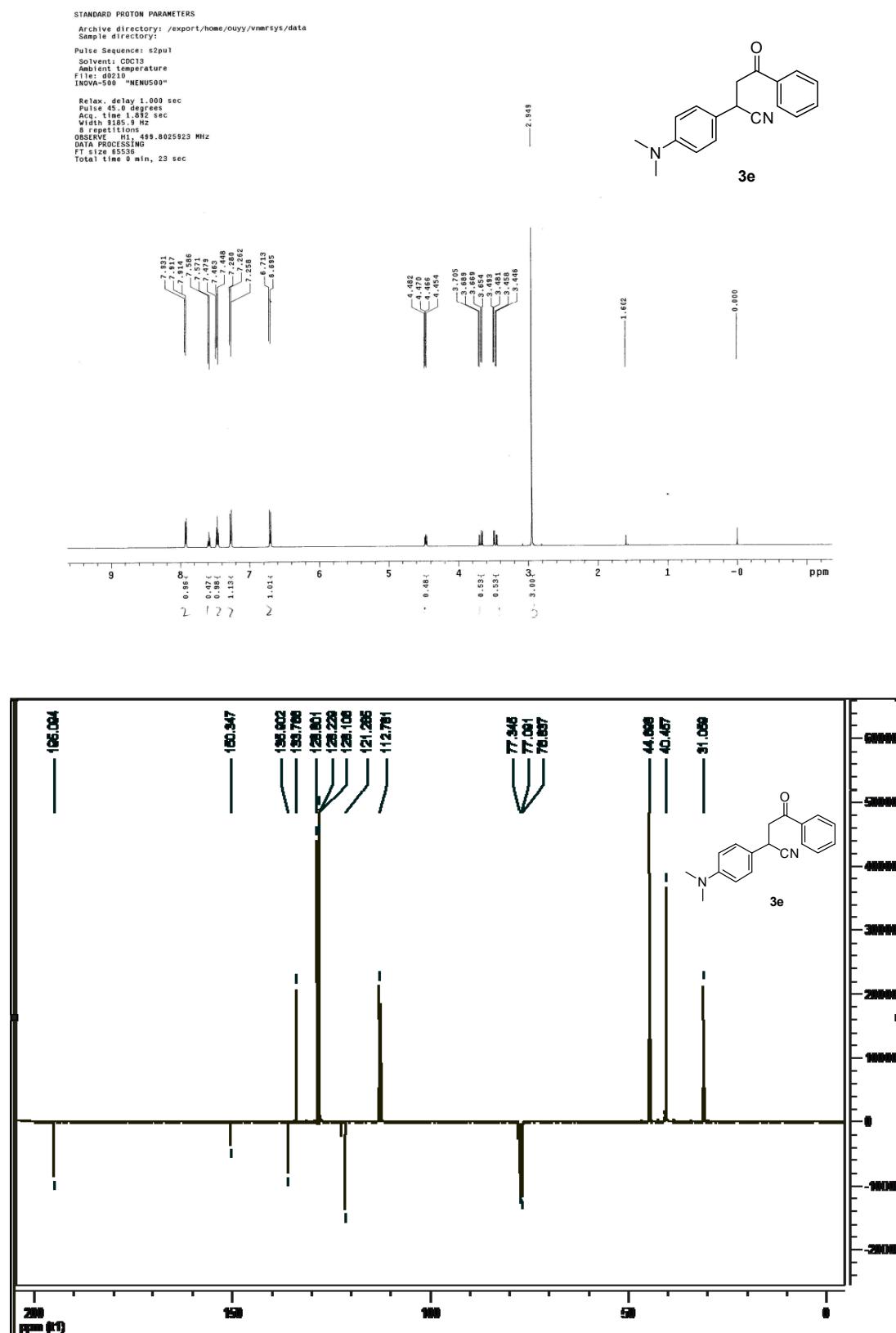


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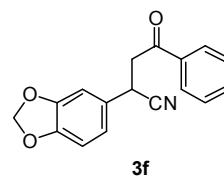
STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl₃
Acq. temperature
File: d023
INNOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.812 sec
W1 10000 Hz
8 repetitions
DSB 10000 Hz, 488.8025810 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



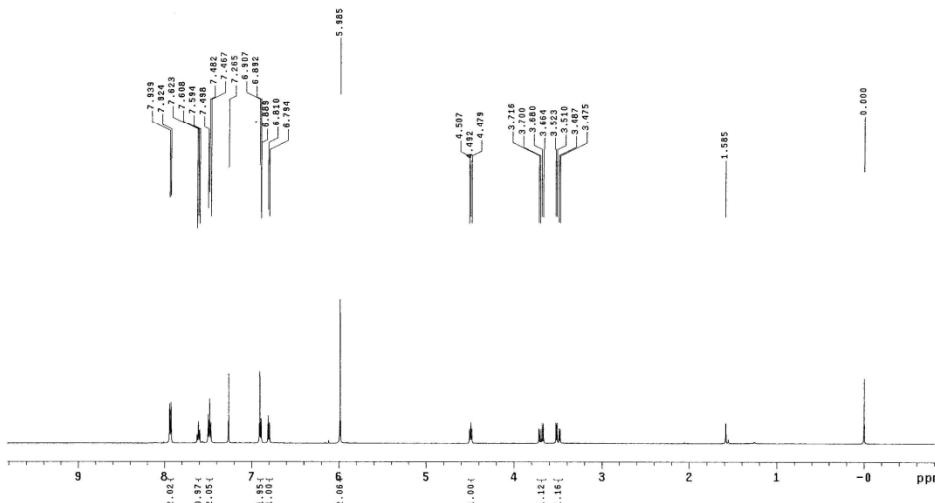
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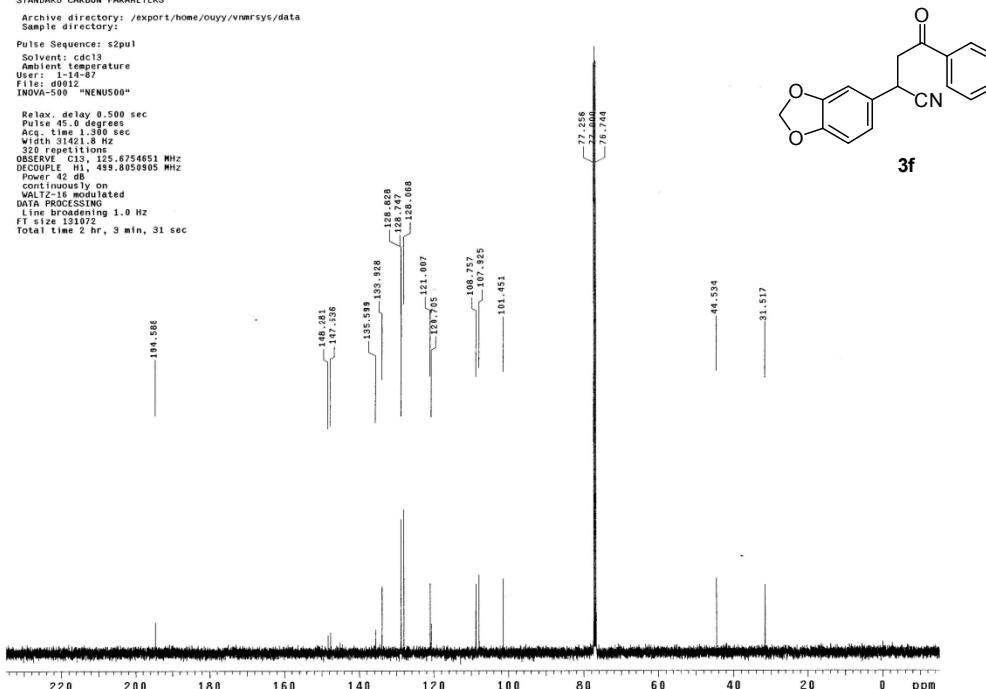
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STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: d9012 "NEMUS00"
INNOVA-500 "NEMUS00"
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 1.052 sec
Width 31421.8 Hz
8 repetitions
OBSERVE: H1 499.8025889 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
File: d9012 "NEMUS00"
INNOVA-500 "NEMUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.052 sec
Width 31421.8 Hz
32 repetitions
OBSERVE: C13 102.6754651 MHz
DECOUPLE: H1 499.8050905 MHz
Power: 100.00000000000000
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec



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STANDARD PROTON PARAMETERS

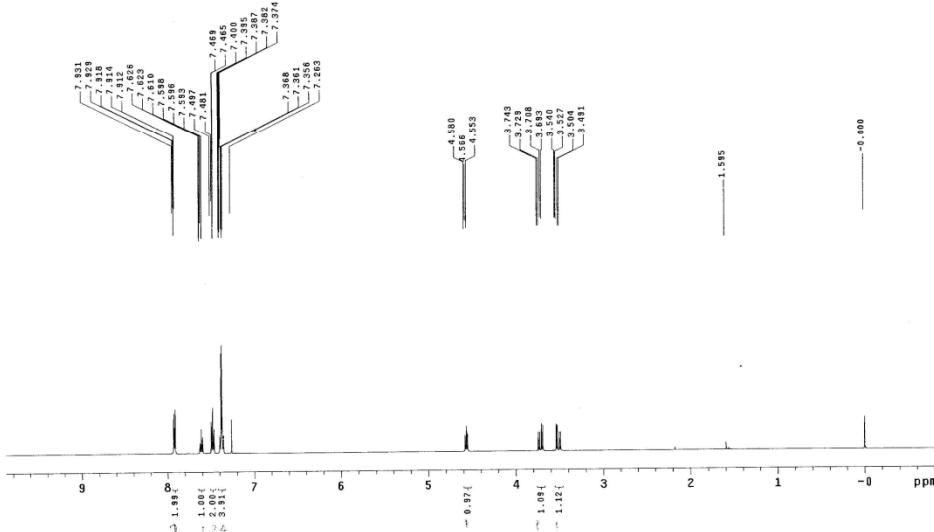
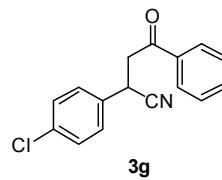
Archive directory: /export/home/ouyu/vnmrsys/data
Sample directory:

Pulse Sequence: spzpl
Solvvent: CDCl3
Acquisition temperature:
File: cb1000
INDVA-500 "NENUS00"

Relaxation delay 1.000 sec
Pulse 45.000 degrees
Accumulation time 1.888 sec
Width 8578.2 Hz
& 8 scans

OBSERVE HI .495 0.825906 MHz
DATA PROCESSING
T1 = 1.000 sec
Total time 0 min, 23 sec

```



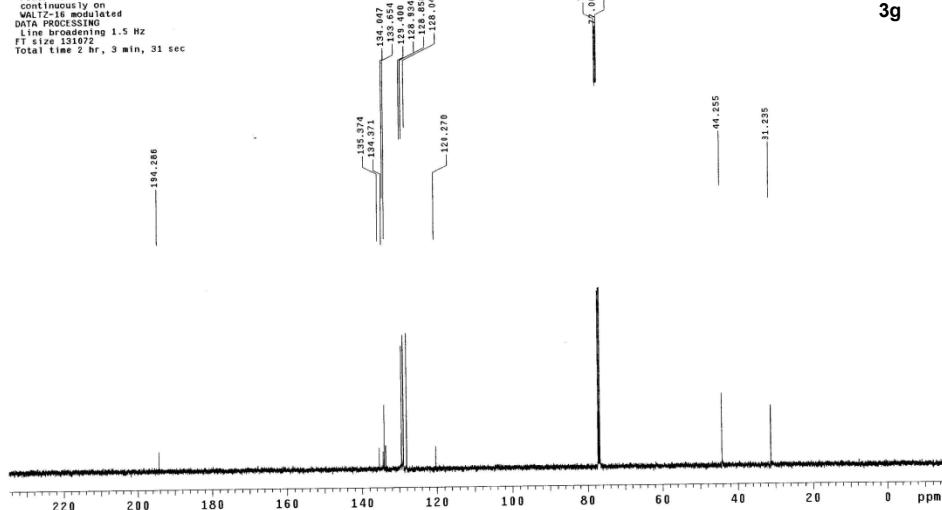
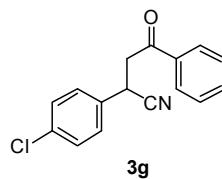
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STANDARD CARBON PARAMETERS

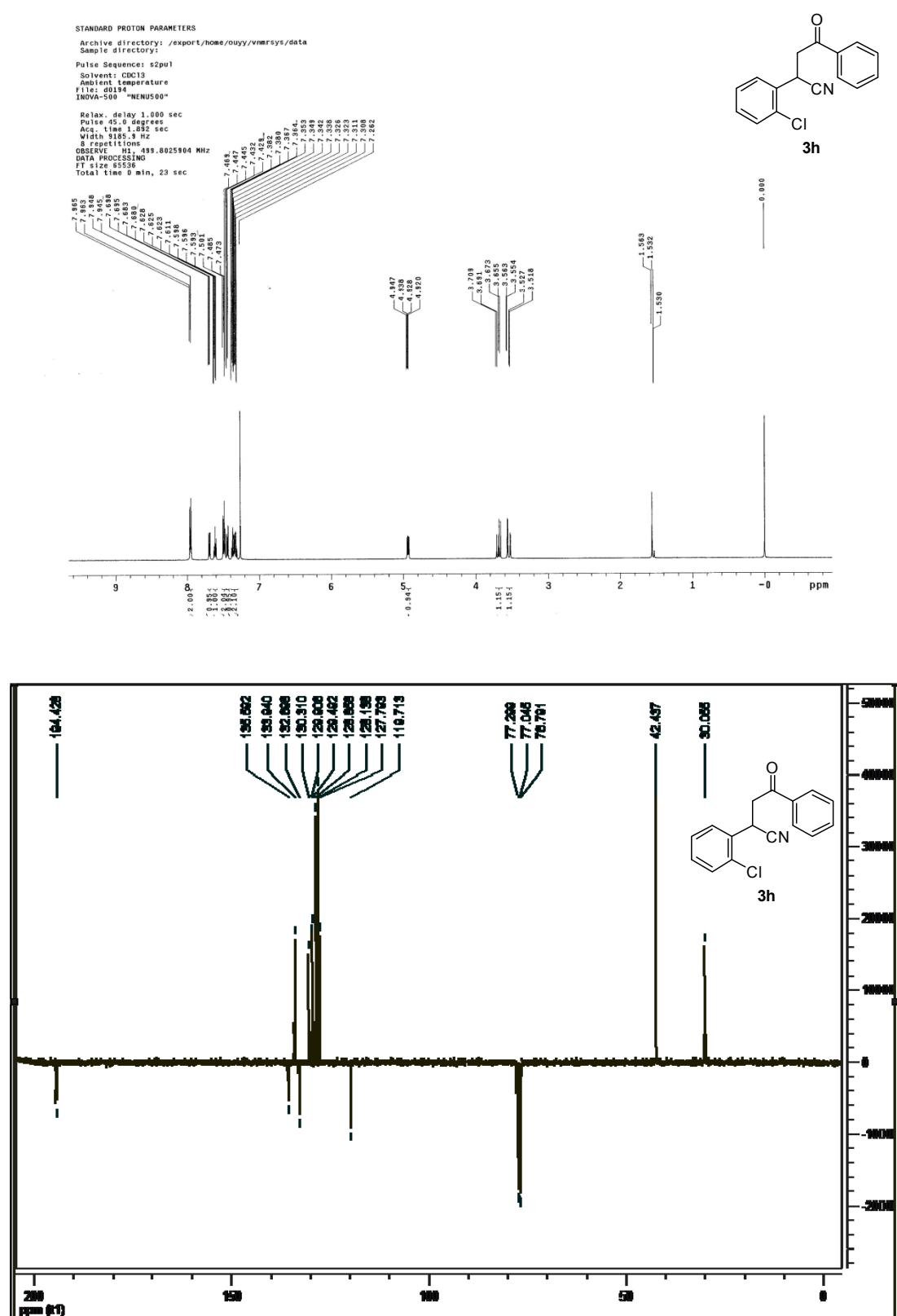
Archive directory: /export/home/ouyy/vnmrsys/date
Sample directory:
Pulse Sequence: 32pul
Solvent editing: circ1
Ambient temperature
User: 1-14-97
File: C13_1
INVOA-500 "NENUS90"

Relax. delay 0.500 sec
Pulse 45.0 degrees
Acc. time 1.300 sec
VRAM 1024
128 repetitions
OBSERVE C13, 123.675400 MHz
DECODE 123.6750900 MHz
Power 42 dB
Continuous
WIDENING modulated
Data PROCESSING
FID size 1.5 Hz
FT size 131024
Total time 2 hr, 3 min, 31 sec

```

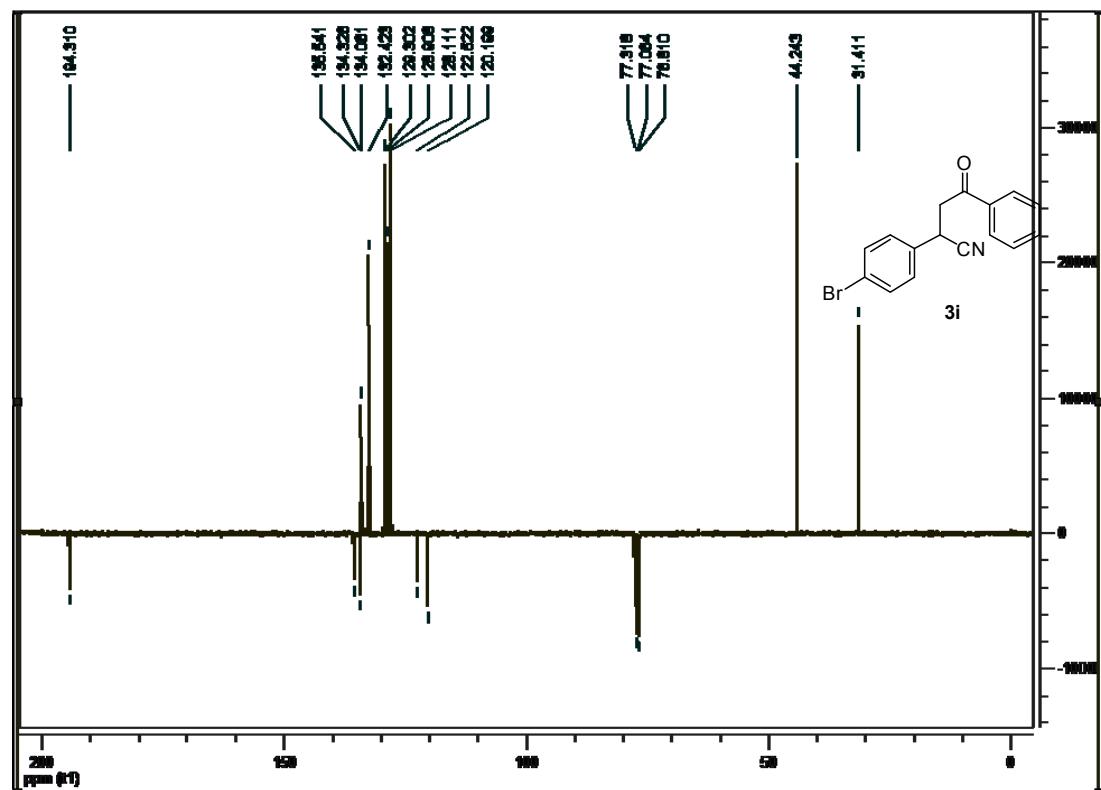
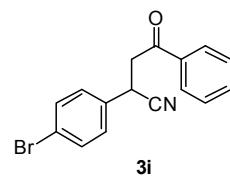
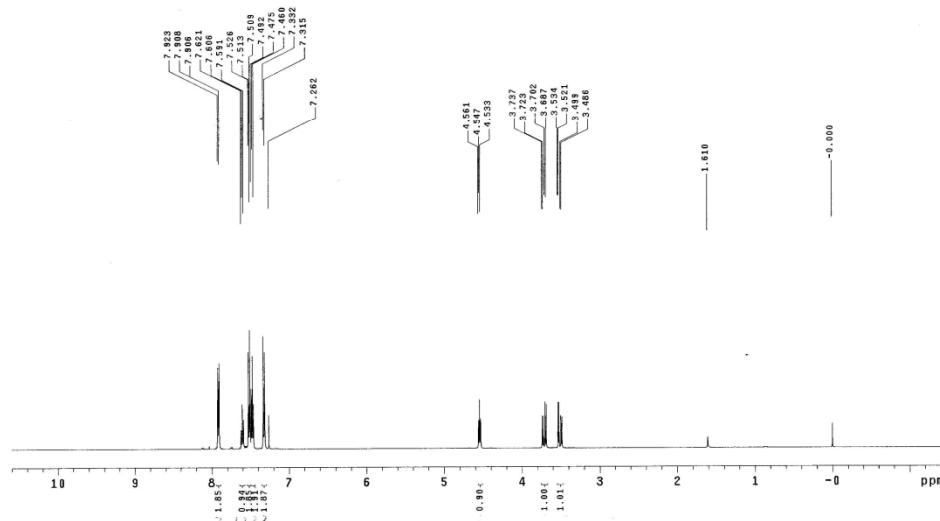


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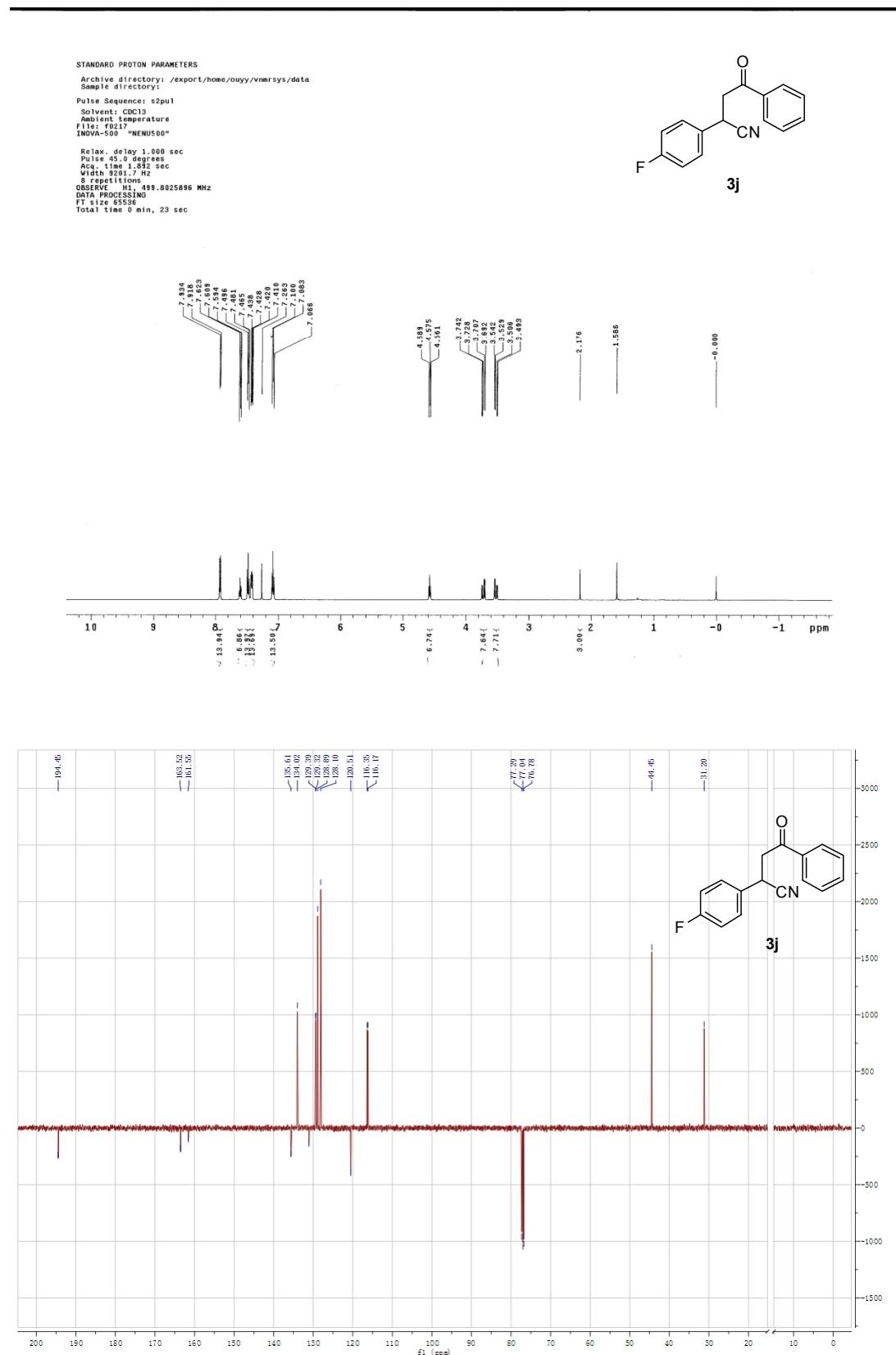


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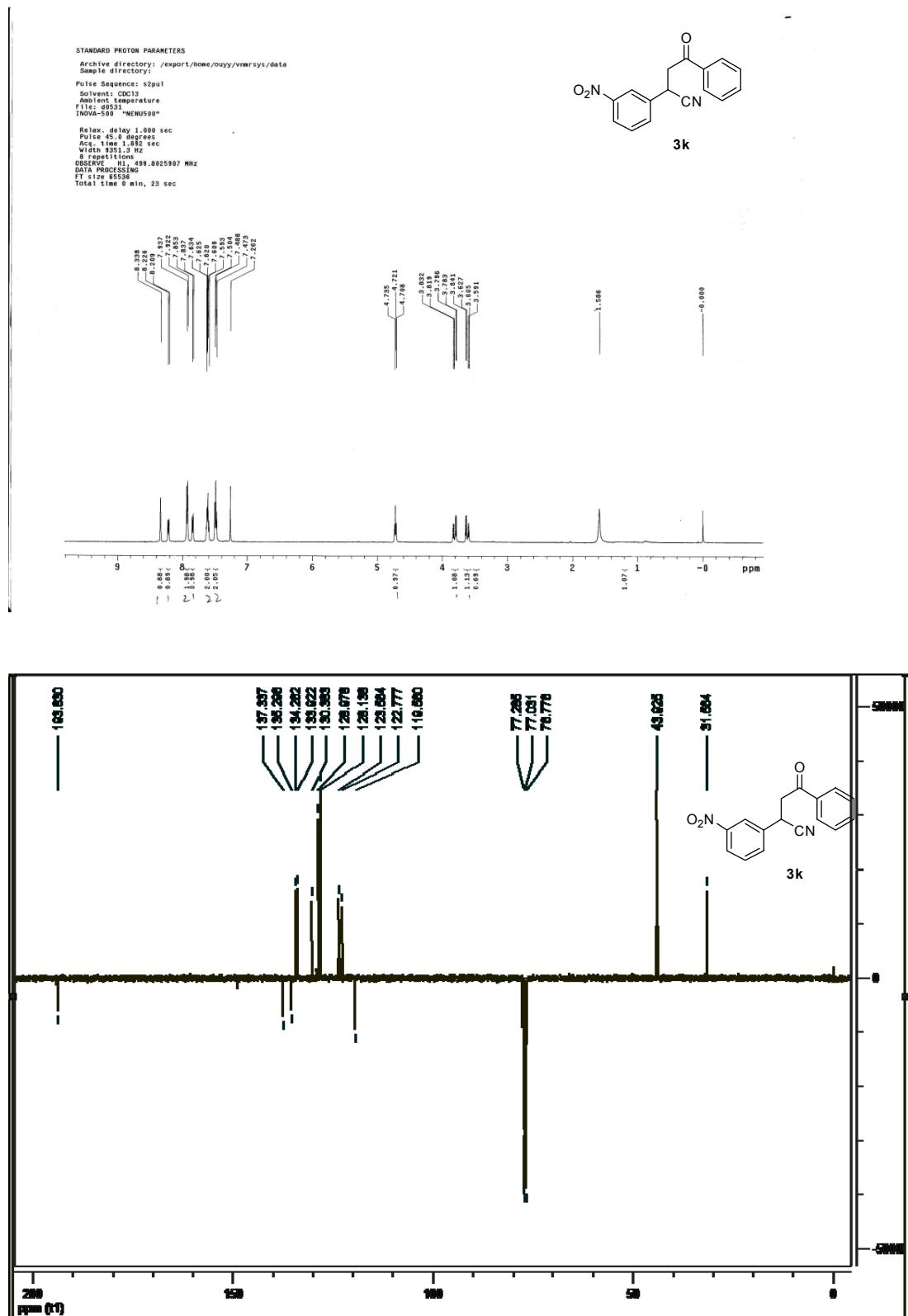
STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
File: d9191 "NEWUS00"
INDIVIDUALS
Relaxation delay 1.000 sec
Pulse 90 degrees
Acq. time 1.892 sec
W1 10.000 sec
8 repetitions
OBSERVE H1 499.8025809 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 23 sec



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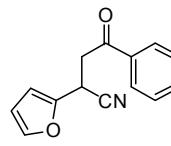


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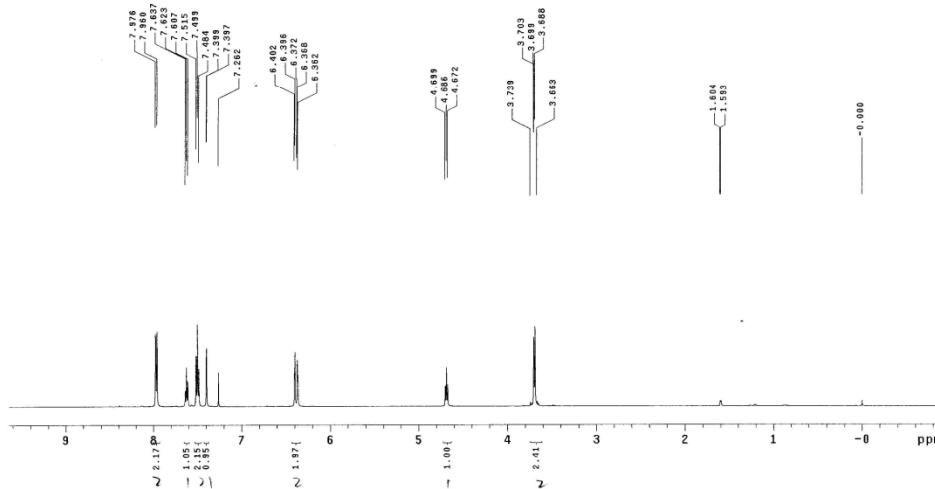


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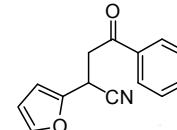
STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Field: 400.00 MHz
INNOVA-500 "NENUS00"
Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.002 sec
W1 14.000 Hz
8 repetitions
OBSERVE: H1, 499.8025909 MHz
DATA PROCESSING
FT size 65536
total time 0 min, 23 sec



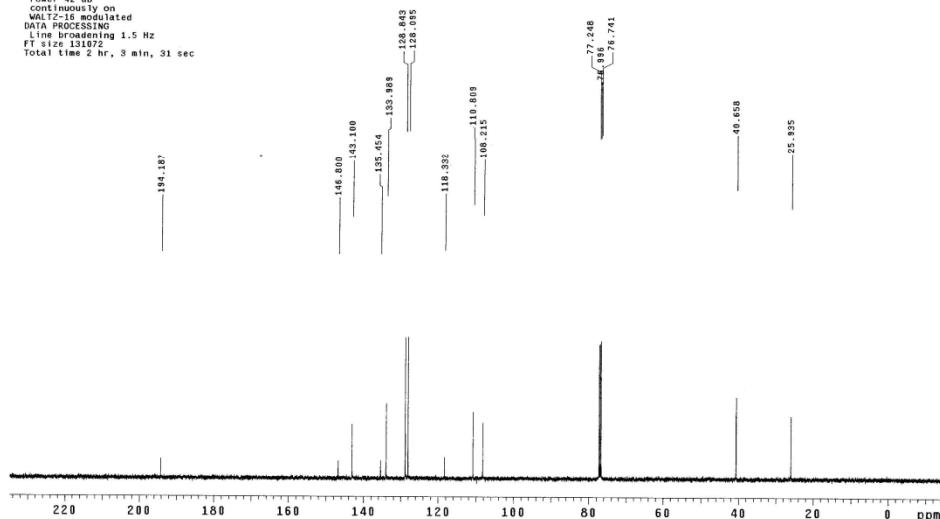
3l



STANDARD CARBON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: cdcl3
Ambient temperature
User: 1-14-07
File: 14-07-04
INNOVA-500 "NENUS00"
Relax. delay 0.500 sec
Pulse 45.0 degrees
Acq. time 1.300 sec
W1 31421.8 Hz
163 repetitions
OBSERVE: C13, 125.6754675 MHz
DECODED TO: 499.8050905 MHz
Power 42 dB
Continuously on
W1 31421.8 Hz
DATA PROCESSING
Line broadening 1.5 Hz
FT size 131072
Total time 2 hr, 3 min, 31 sec

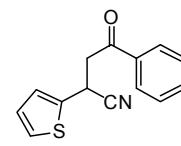
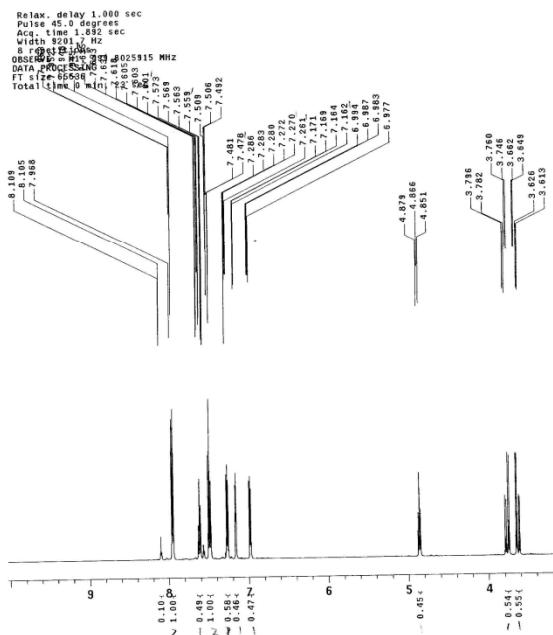


3l

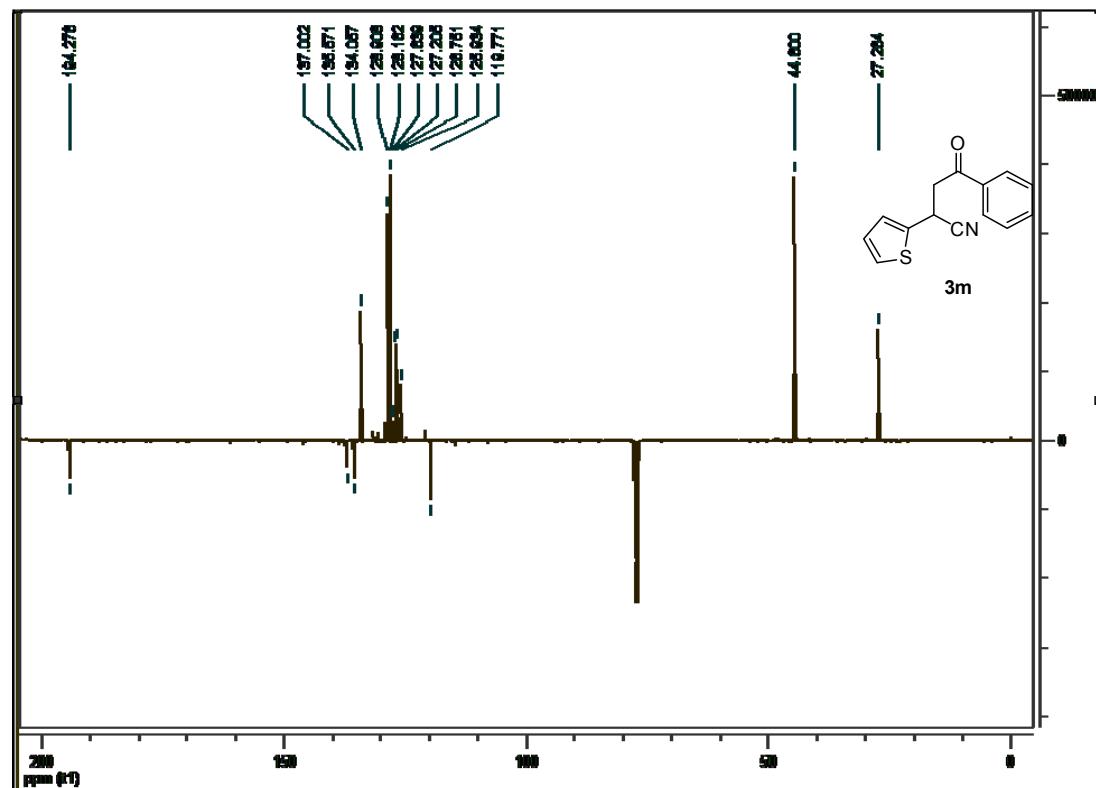


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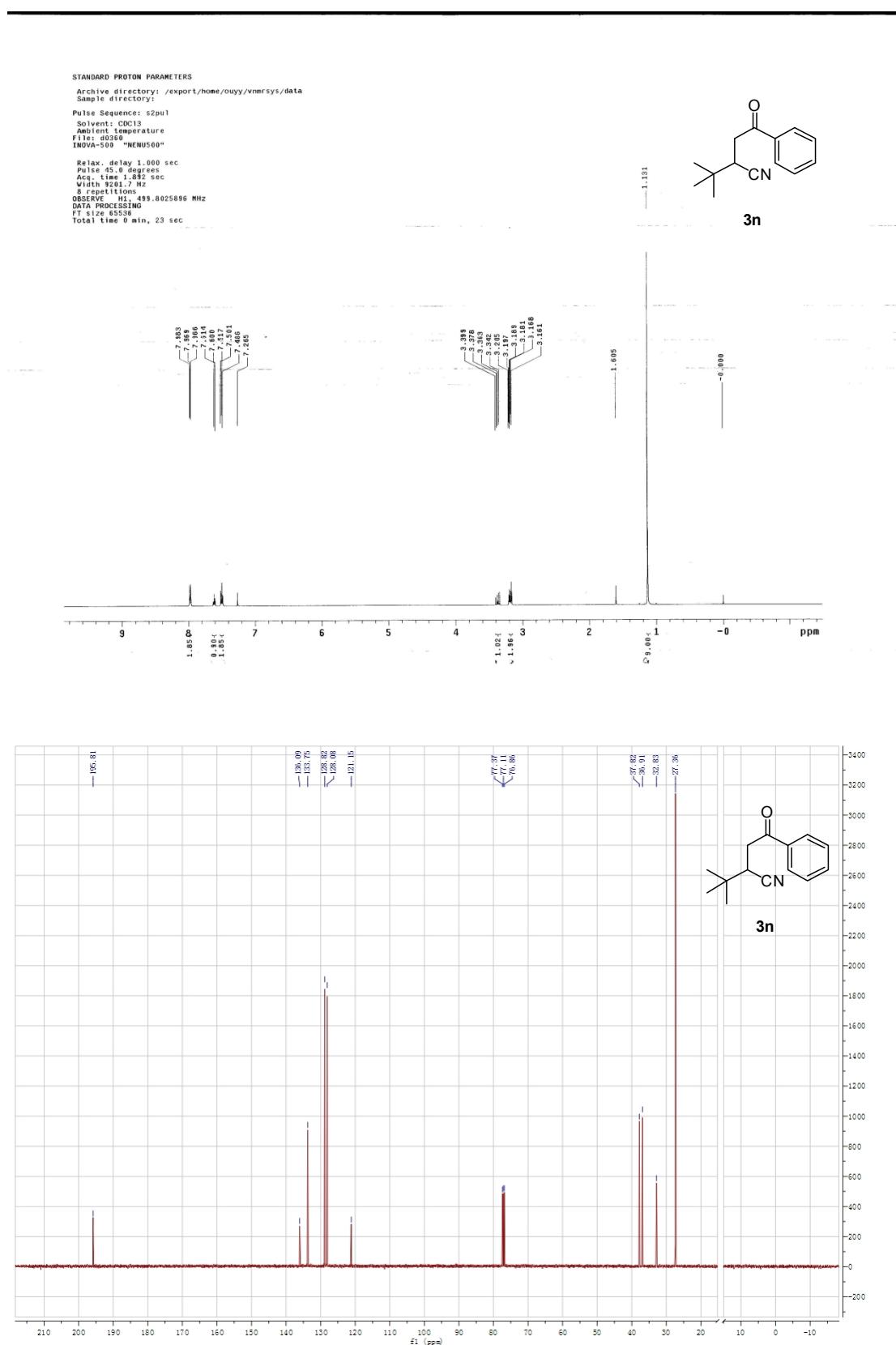
STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnversys/data
Sample directory:
Pulse Sequence: s2pul1
Solvent: CDCl3
Ambient temperature
FID抑制时间: 100 sec
INNOVA-500 "NENU500"
Relax. delay 1.000 sec
Pulse 90.0 degrees
Acq. time 1.682 sec
Width 1.000 sec
8 FID's stacked
OBSERVE FID NO. 1, T2=655815 MHz
DATA FID NO. 1, T2=655815 MHz
FT FID NO. 1, T2=655815 MHz
Total time [0 min]: 1.682 sec



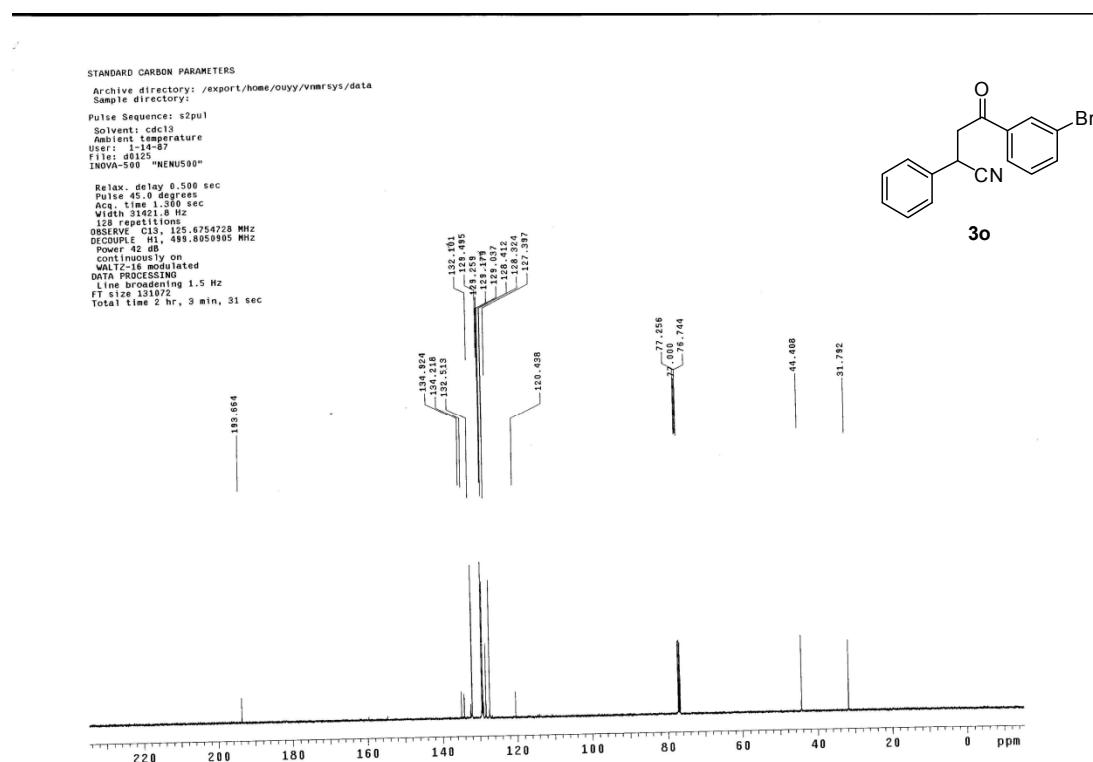
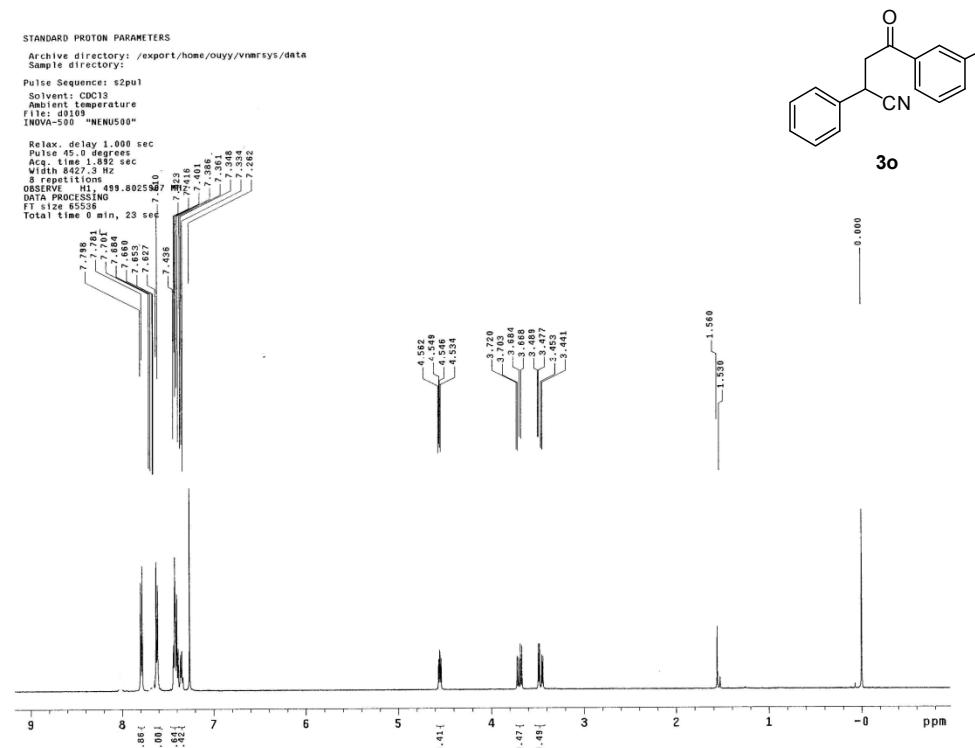
3m



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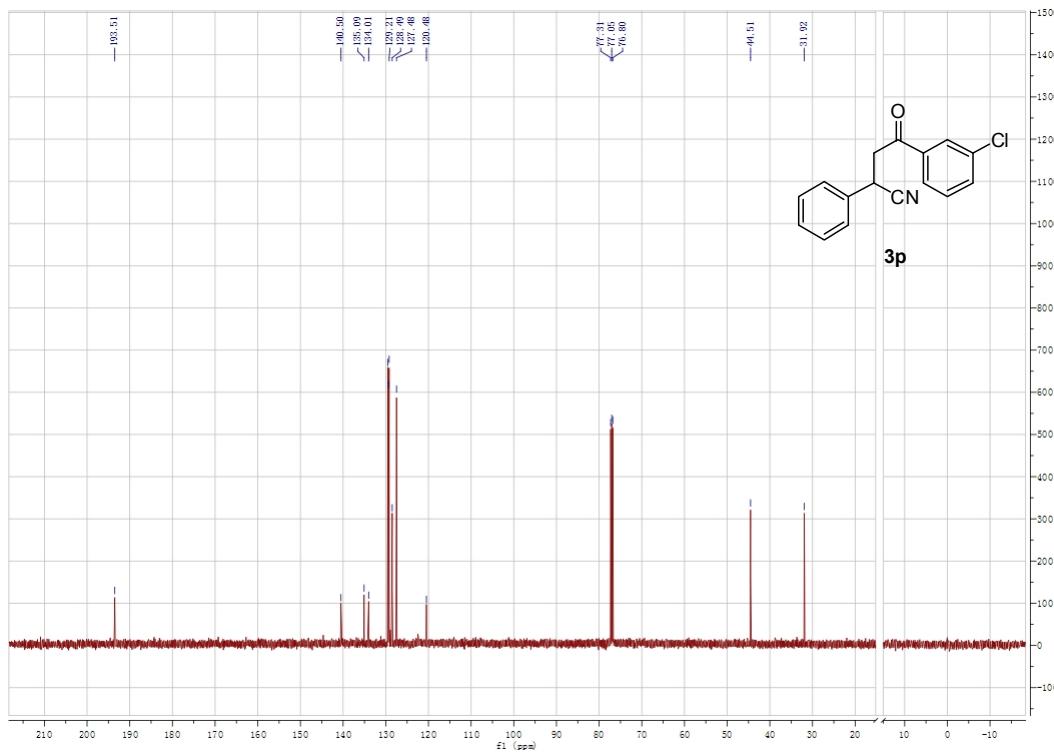
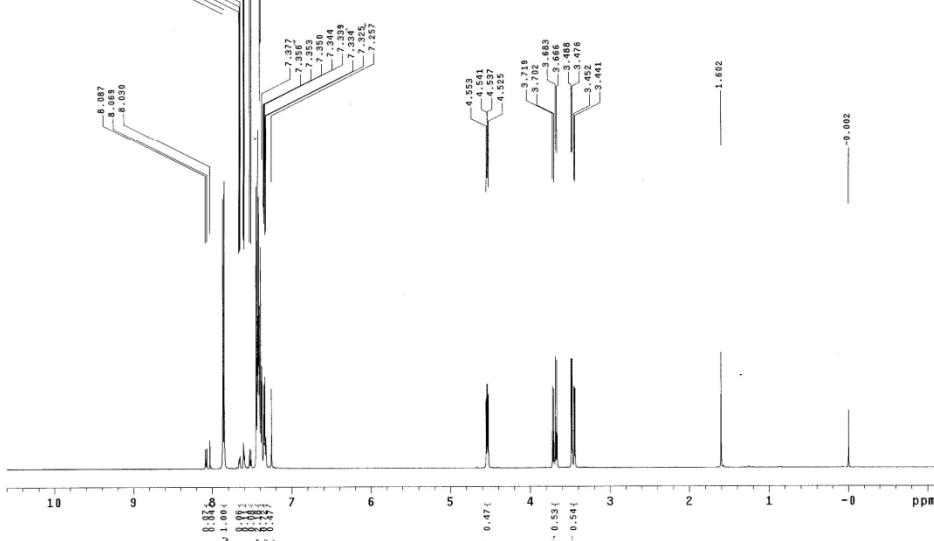


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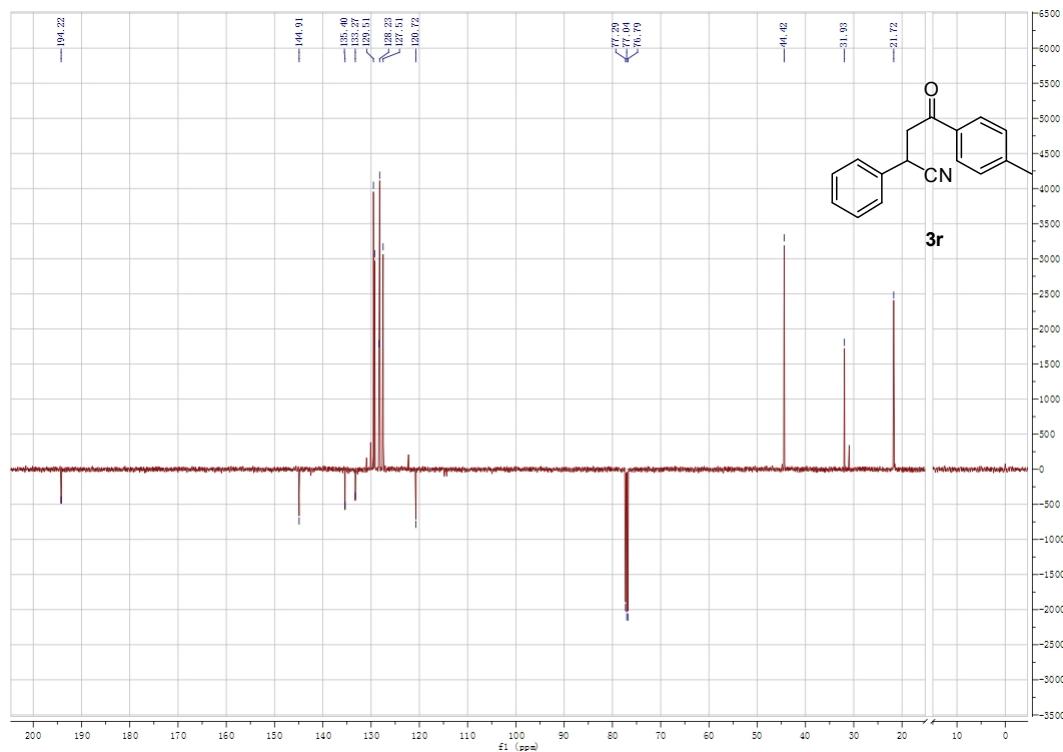
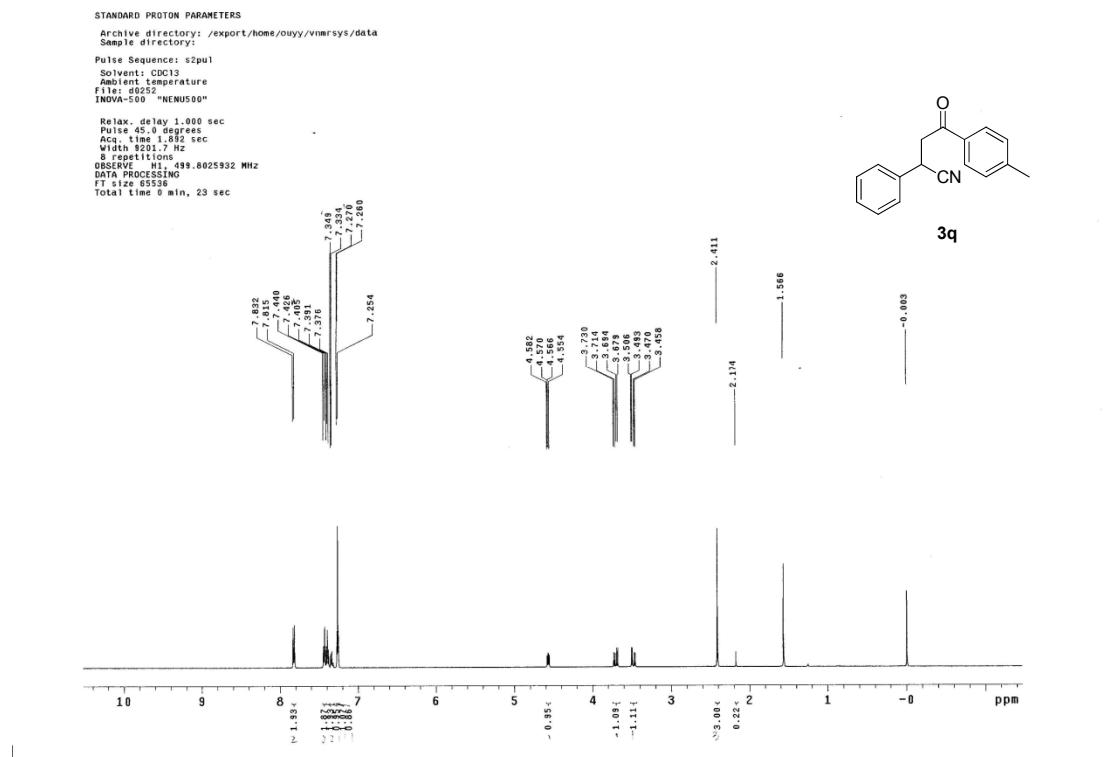


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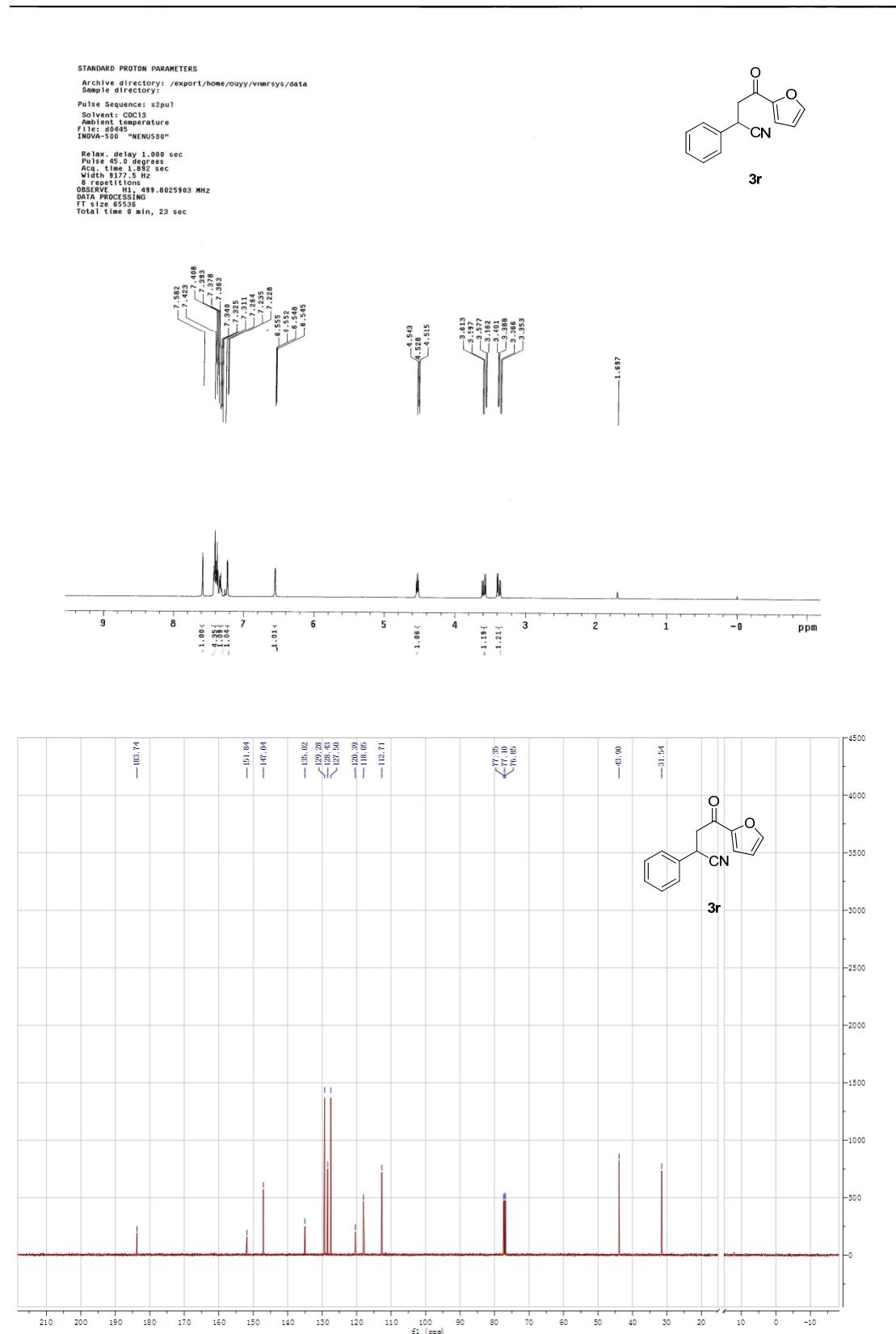
STANDARD PROTON PARAMETERS
Archive directory: /export/home/ouyy/vnmrsys/data
Sample directory:
Pulse Sequence: s2pul
Solvent: CDCl₃
Ambient temperature
Filter: d9411
IN1DVA=500 "NENUS00"
Relaxation delay: 1.000 sec
Pulse width: 90 degrees
Acq. time: 1.882 sec
W1: 10.000 Hz
8 repetitions
OBSERVE: H1, 499.8025839 MHz
DATA PROCESSING
FT size: 65536
Total time: 0 min, 23 sec



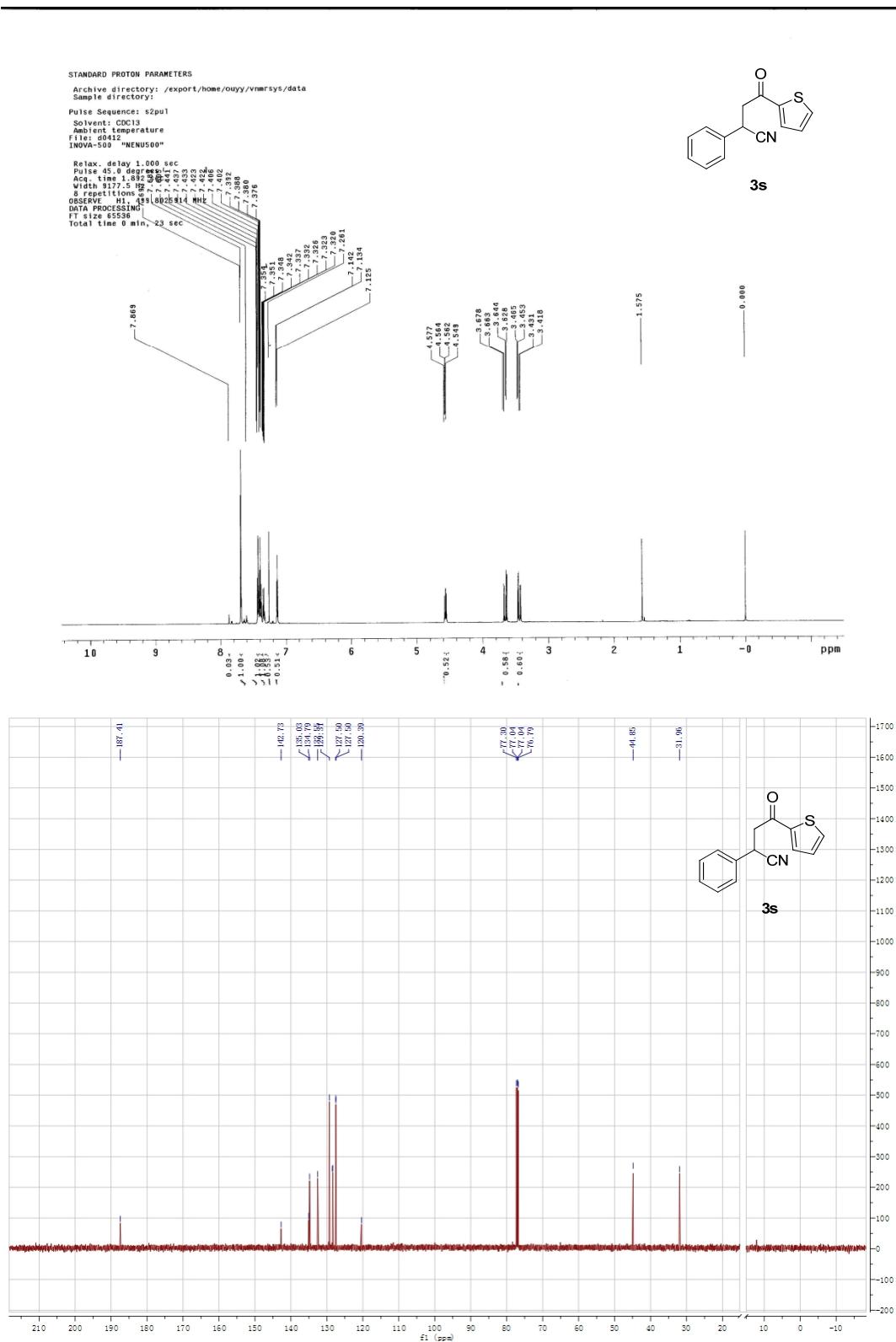
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