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Supporting Information for Base-Promoted Dehydrogenative Coupling of Benzene Derivatives with Amides or Ethers

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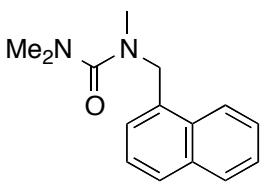
General Remarks. All manipulations of oxygen- and moisture-sensitive materials were conducted using a standard Schlenk technique or a glove box under a nitrogen atmosphere. Nuclear magnetic resonance spectra were taken on a JEOL JNM LA600 spectrometer (^{1}H, 600 MHz and ^{13}C, 150 MHz) or a JEOL JNM LA500 spectrometer (^{1}H, 500 MHz and ^{13}C, 125 MHz). High-resolution mass spectra were obtained with a Bruker Daltonics microTOF-Q spectrometer (ESI). GC spectra were taken on Shimadzu GC-2014. GC-MS spectra were taken on Shimadzu GCMS-QP5050A. GC and GC-MS were equipped with capillary column SGE BPX5. Preparative recycling gel permeation chromatography (GPC) was performed with JAI LC-908 equipped with JAIGEL-1H and -2H using chloroform as an eluent. High performance liquid chromatography (HPLC) was performed with JAI LC-9204 equipped with Inertsil SIL 100A (GL Science Inc.) using hexane and ethyl acetate as eluents. Unless otherwise noted, reagents were commercially available and used without further purification. *N*,*N*,*N*',*N*'-Tetramethylurea (TMU, **1a**), *N*,*N*-dimethylacetamide (DMA, **1b**), *N*-methylacetamide (**1c**) and 1,3-dimethyl-2-imidazolidinone (DMI, **1f**) were purified by distillation from CaH_2. Tetrahydrofuran (THF, **1d**) was purified by passing through an alumina/catalyst column system (GlassContour Co.). Dibutyl ether (**1e**) was purified by distillation from sodium/benzophenone ketyl. Di-*tert*-butyl hyponitrite was prepared according to a literature procedure.[1]

Dehydrogenative Coupling of Arenes with Amides or Ethers: General Procedure. To a 3 mL vial equipped with a stir bar in a glove box were added successively NaO*t*-Bu, an amide/ether (**1**: 17.8 mmol), an arene (**2**: 0.669 mmol), and *t*-BuOO*t*-Bu (0.223 mmol). The vial was taken out of the glove box and stirred under 120 °C for 24 h. The reaction mixture was poured into water (10 mL), extracted with ethyl acetate (3 x 10 mL), and dried over $MgSO_4$. After filtration and concentration, the crude mixture was subjected to silica gel chromatography on preparative TLC (hexane and ethyl acetate) to give the corresponding product (**3**). The product was further purified with GPC and/or HPLC (silica gel) if necessary.

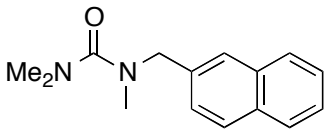
The dehydrogenative coupling can be conducted also in a Schlenk tube. To a 20 mL Schlenk tube equipped with a stir bar under a nitrogen atmosphere were added successively NaO*t*-Bu, **1**, **2** and *t*-BuOO*t*-Bu, and the resulting mixture was stirred at 120 °C for 24 h. The reaction mixture was treated in the same manner as described above. For example, the dehydrogenative coupling of **1a** with **2m** in a 20 mL Schlenk tube gave **3am** and **3'am** in 68% combined yield (**3am**:**3'am** = 94:6).

Refinement of the Methyl Esters Contaminated with tert-Butyl Esters: a Representative Example (Footnote 15). A crude product obtained in the coupling of methyl benzoate (**2q**) with THF using *t*-BuOO*t*-Bu (32.4 mg, 0.222 mmol) under the conditions of entry 5 of Scheme 7 was dissolved in methanol (5.4 mL) and treated with NaOMe (40.0 mg, 0.740 mmol) under reflux for 6 days. After methanol was removed in vacuo, the reaction mixture was poured into a saturated NH_4Cl aqueous solution (20 mL), extracted with ethyl acetate (3 x 20 mL), and dried over $MgSO_4$. After filtration and concentration, the crude mixture was subjected to silica gel chromatography on preparative TLC (hexane/ethyl acetate = 80/20) to give the corresponding methyl esters (**3dq** and **3'dq**) in 56% yield (85:15).

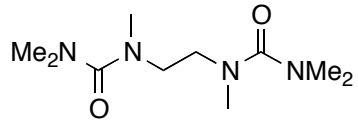
The coupling products in Table 1



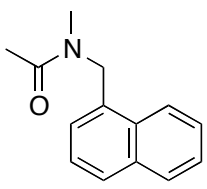
***N,N,N'*-Trimethyl-*N'*-(1-naphthylmethyl)urea (3am).** A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.74 (s, 3 H), 2.82 (s, 6 H), 4.84 (s, 2 H), 7.42–7.47 (m, 2 H), 7.47–7.54 (m, 2 H) 7.77–7.82 (m, 1 H), 7.85–7.90 (m, 1 H), 7.92–7.97 (m, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.1, 38.8, 52.6, 123.4, 125.5, 125.95, 126.01, 126.3, 128.2, 128.9, 131.8, 133.5, 134.0, 165.7. HRMS (ESI) Calcd for $C_{15}H_{18}N_2O$: $[M+Na]^+$, 265.1311. Found: m/z 265.1309.



***N,N,N'*-Trimethyl-*N'*-(2-naphthylmethyl)urea (3'am).** A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.77 (s, 3 H), 2.87 (s, 6 H), 4.53 (s, 2 H), 7.41 (dd, *J* = 8.6, 1.6 Hz, 1 H), 7.46 (td, *J* = 7.0, 1.6 Hz, 1 H), 7.48 (td, *J* = 6.7, 1.6 Hz, 1 H), 7.71 (s, 1 H), 7.79–7.85 (m, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 36.8, 38.9, 54.5, 125.9, 126.0, 126.3, 126.5, 127.8, 127.9, 128.5, 132.9, 133.6, 135.8, 165.8. HRMS (ESI) Calcd for $C_{15}H_{18}N_2O$: $[M+Na]^+$, 265.1311. Found: m/z 265.1318.

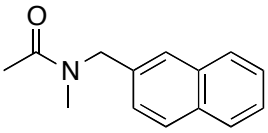


***N,N'*-Dimethyl-*N,N'*-bis(dimethylaminocarbonyl)ethyl enediamine (5a).**[2] A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.77 (s, 12 H), 2.86 (s, 6 H), 3.37 (s, 4 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.5, 38.8, 47.7, 165.3. HRMS (ESI) Calcd for $C_{10}H_{22}N_4O_2$: $[M+Na]^+$, 253.1635 Found: m/z 253.1632.

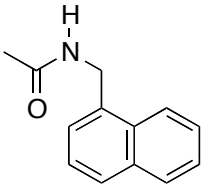


***N*-Methyl-*N*-(1-naphthylmethyl)acetamide (3bm).** A colorless oil. Observed as two rotamers of 63/37 ratio in ^{1}H NMR. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.18/2.13 (s, 3 H), 2.85/3.07 (s, 3 H), 5.07/5.01 (s, 2 H), 7.34/7.24 (d, *J* = 6.9/7.1 Hz, 1 H), 8.10/7.92 (d, *J* = 8.2/7.4 Hz, 1 H). The other peaks were not separated. δ 7.39–7.60 (m, 3 H) [7.43 (t, *J* = 7.4 Hz, 1 H) was distinguished to belong to the major rotamer.], 7.77–7.90 (m, 2 H).

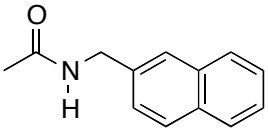
^{13}C NMR (125 MHz, $CDCl_3$) δ 21.4, 22.1, 34.4, 35.0, 48.5, 52.2, 122.1, 122.6, 124.1, 125.2, 125.7, 126.1, 126.2, 126.6, 126.7, 127.2, 128.2, 128.6, 128.7, 129.2, 130.8, 131.6, 131.9, 132.9, 133.9, 134.0, 170.6, 171.7. HRMS (ESI) Calcd for $C_{14}H_{15}NO$: $[M+Na]^+$, 236.1046. Found: m/z 236.1046.



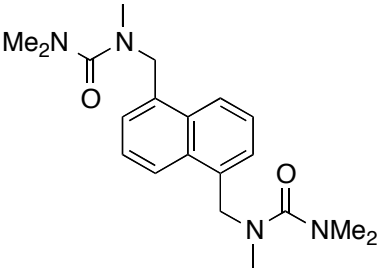
***N*-Methyl-*N*-(2-naphthylmethyl)acetamide (3'bm).** A colorless oil. Observed as two rotamers of 57/43 ratio in ^{1}H NMR. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.19/2.20 (s, 3 H), 2.94/3.01 (s, 3 H), 4.75/4.68 (s, 2 H), 7.38/7.28 (dd, *J* = 8.4, 1.5/8.5, 1.6 Hz, 1 H), 7.67/7.59 (s, 1 H). The other peaks were not separated. δ 7.43–7.54 (m, 2 H), 7.77–7.89 (m, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.6, 22.0, 33.9, 35.6, 50.8, 54.6, 124.5, 124.9, 125.9, 126.2, 126.3, 126.7, 126.9, 127.79, 127.80, 127.83, 127.9, 128.6, 129.0, 132.9, 133.0, 133.5, 133.6, 134.1, 135.1, 170.9, 171.2. HRMS (ESI) Calcd for $C_{14}H_{15}NO$: $[M+Na]^+$, 236.1046. Found: m/z 236.1049.



***N*-(1-Naphthylmethyl)acetamide (3cm).**[3] A colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.01 (s, 3 H), 4.90 (d, *J* = 5.4 Hz, 2 H), 5.64 (bs, 1 H), 7.40–7.48 (m, 2 H), 7.52 (td, *J* = 6.9, 1.2 Hz, 1 H), 7.56 (td, *J* = 6.9, 1.5 Hz, 1 H), 7.83 (dd, *J* = 6.9, 2.6 Hz, 1 H), 7.89 (d, *J* = 8.0 Hz, 1 H), 8.03 (d, *J* = 8.7 Hz, 1 H).



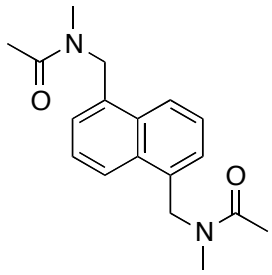
***N*-(2-Naphthylmethyl)acetamide (3'cm).**[3] A colorless solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.06 (s, 3 H), 4.60 (d, *J* = 5.5 Hz, 2 H), 5.78 (bs, 1 H), 7.40 (dd, *J* = 8.5, 2.0 Hz, 1 H), 7.44–7.51 (m, 2 H), 7.72 (s, 1 H), 7.78–7.85 (m, 3 H).



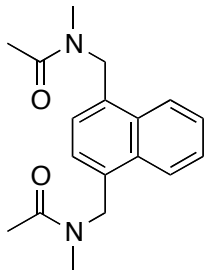
1,5-Bis(*N*-dimethylaminocarbonyl-*N*-methylaminomethyl)naphthalene (4am-a). A yellow soild. ^{1}H NMR (500 MHz, $CDCl_3$), δ 2.75 (s, 6 H), 2.82 (s, 12 H), 4.84 (s, 4 H), 7.42–7.51 (m, 4 H), 7.91 (d, *J* = 7.6 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.2, 38.8, 52.8, 123.3, 125.8, 126.0, 132.2, 134.2, 165.7. HRMS (ESI) Calcd for $C_{20}H_{28}N_4O_2$: $[M+Na]^+$, 379.2104. Found: m/z 379.2112. GC–MS (appears earlier than regioisomer **4am-b** on GC) m/z (% relative intensity, ion) 356 (0.3, M), 254 (21), 197 (28), 182 (22), 72 (100).

The other isomer (**4am-b**) could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **4am-a** (**4am-a**:**4am-b** = 79:21). ^{1}H NMR (500 MHz, $CDCl_3$), δ 2.74 (s, 3 H), 2.78 (s, 3 H), 2.82 (s, 6 H: overlapped with a peak of **4am-a**.), 2.88 (s, 6 H), 4.53 (s, 2 H), 4.82 (s, 2 H), 7.40–7.50 (m, 3 H: overlapped with peaks of **4am-a**), 7.74 (s, 1 H), 7.76 (d, *J* = 8.1 Hz, 1 H), 7.92 (d, *J* = 8.4 Hz, 1 H).

Although the structure of **4am-b** could not be determined due to overlap with peaks of **4am-a** at 7.40–7.50 ppm, the existence of two different TMU units and a singlet in the aromatic region (δ 7.74) imply that these peaks belong to 1,3- or 1,6- or 1,7-bis(*N*-dimethylaminocarbonyl-*N*-methylaminomethyl)naphthalene. GC–MS (appears later than regioisomer **4am-a** on GC) m/z (% relative intensity, ion) 356 (10, M), 254 (13), 182 (15), 72 (100).

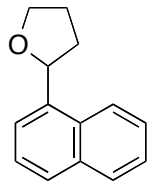


1,5-Bis(*N*-acetyl-*N*-methylaminomethyl)naphthalene (4bm-a). A colorless oil. Each component was observed as a set of 2–4 peaks in ^{1}H NMR due to existence of three rotamers. ^{1}H NMR (500 MHz, $CDCl_3$), δ 2.12/2.18/2.19 (s, 6 H), 2.85/2.88/3.06/3.07 (s, 6 H), 5.01/5.04/5.07/5.09 (s, 4 H), 7.27/7.31/7.36/7.40 (d, *J* = 7.6/6.9/7.2 Hz, the coupling constant of a rotamer (7.27 ppm) could not be read because the peak was overlapped with the solvent residual peak, 2 H), 7.48/7.52/7.54/7.58 (t, *J* = 8.6/8.0/8.1/8.5 Hz, 2 H), 7.85/7.87/8.09/8.10 (d, *J* = 8.6/8.8/8.5/8.6 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.4, 22.1, 22.2, 31.1, 34.5, 35.1, 35.2, 48.8, 48.9, 52.4, 52.5, 122.0, 122.3, 122.9, 124.1, 124.5, 126.0, 126.4, 126.5, 127.1, 127.2, 127.29, 127.34, 131.2, 131.3, 132.2, 132.3, 132.4, 132.9, 133.5, 134.1, 170.6, 170.7, 171.7. HRMS (ESI) Calcd for $C_{18}H_{22}N_2O_2$: $[M+Na]^+$, 321.1573. Found: m/z 321.1566.



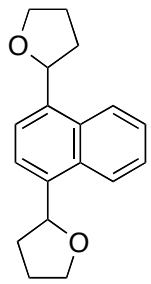
1,4-Bis(*N*-acetyl-*N*-methylaminomethyl)naphthalene (4bm-b). A colorless oil. Each component was observed as a set of 2–4 peaks in ^{1}H NMR due to existence of three rotamers. ^{1}H NMR (500 MHz, $CDCl_3$), δ 2.12/2.14/2.188/2.194 (s, 6 H), 2.86/2.89/3.05/3.07 (s, 6 H), 5.00/5.01/5.06/5.07 (s, 4 H), The peaks of protons (2 H) on C-2 and C-3 appear as a set of two doublets or a singlet [7.18 (d, *J* = 7.1 Hz) and 7.32 (d, *J* = 7.1 Hz); 7.24 (s); 7.27 (s)], 7.56 (dd, *J* = 6.6, 3.3 Hz, 2 H), 7.55–7.63 (m, 2 H), 7.87–7.92 (m, 1 H), 7.95 (dd, *J* = 6.6, 3.1 Hz, 2 H), 8.12 (dd, *J* = 6.6, 3.5 Hz, 2 H), 8.17–8.22 (m, 1 H). Because the amount (0.9 mg) of a sample of this compound was small, only 22 peaks were given in ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.4, 22.2, 34.6, 35.2, 35.3, 48.6, 52.3, 121.9, 122.7, 123.2, 124.7, 125.3, 126.3, 126.6, 126.8, 126.9, 131.3, 131.9, 132.3, 132.9, 133.1, 170.7. HRMS (ESI) Calcd for $C_{18}H_{22}N_2O_2$: $[M+Na]^+$, 321.1573. Found: m/z 321.1569.

The coupling products in Scheme 3

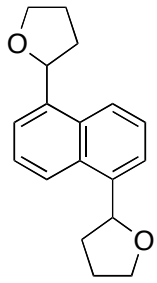


1-(2-Tetrahydrofuranyl)naphthalene (3dm). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.92 (dt, *J* = 18.7, 6.8 Hz, 1 H), 1.98–2.13 (m, 2 H), 2.57 (dq, *J* = 12.3, 7.6 Hz, 1 H), 4.04 (q, *J* = 8.1 Hz, 1 H), 4.24 (td, *J* = 7.9, 5.6 Hz, 1 H), 5.65 (t, *J* = 7.0 Hz, 1 H), 7.43–7.54 (m, 3 H), 7.64 (d, *J* = 7.2

Hz, 1 H), 7.76 (d, *J* = 8.2 Hz, 1 H), 7.87 (d, *J* = 8.5 Hz, 1 H), 7.98 (d, *J* = 8.1 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 33.9, 68.9, 78.1, 122.0, 123.6, 125.5, 125.6, 125.9, 127.6, 128.9, 130.5, 133.9, 139.5. HRMS (ESI) Calcd for $C_{14}H_{14}O$: $[M+Na]^+$, 221.0937. Found: m/z 221.0940.

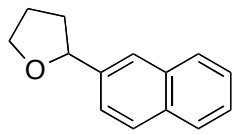


1,4-Bis(2-tetrahydrofuranyl)naphthalene (4dm-a). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.87–1.97 (m, 2 H), 1.97–2.13 (m, 4 H), 2.55 (dq, *J* = 12.1, 7.8 Hz, 2 H), 4.03 (q, *J* = 7.3 Hz, 2 H), 4.23 (td, *J* = 8.0, 5.7 Hz, 2 H), 5.62 (t, *J* = 7.0 Hz, 2 H), 7.48–7.53 (m, 2 H), 7.61 (s, 2 H), 7.98–8.04 (m, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 33.9, 68.8, 78.2, 121.7, 124.3, 125.4, 130.9, 138.5. HRMS (ESI) Calcd for $C_{18}H_{20}O_2$: $[M+Na]^+$, 291.1356. Found: m/z 291.1352.



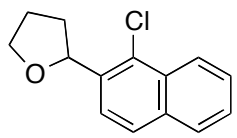
1,5-Bis(2-tetrahydrofuranyl)naphthalene (4dm-b). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.85–1.95 (m, 2 H), 1.96–2.12 (m, 4 H), 2.57 (dq, *J* = 12.3, 7.6 Hz, 2 H), 4.03 (q, *J* = 7.1 Hz, 2 H), 4.24 (td, *J* = 7.8, 5.4 Hz, 2 H), 5.66 (t, *J* = 7.0 Hz, 2 H), 7.49 (dd, *J* = 9.3, 8.1 Hz, 2 H), 7.65 (d, *J* = 7.1 Hz, 2 H), 7.89 (d, *J* = 8.4 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 34.1, 68.9, 78.2, 121.5, 122.8, 125.5, 130.7, 140.3. HRMS (ESI) Calcd for $C_{18}H_{20}O_2$: $[M+Na]^+$, 291.1356. Found: m/z 291.1352.

The coupling products in Scheme 5

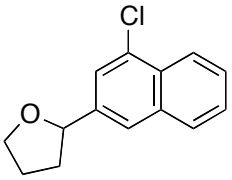


2-(2-Tetrahydrofuranyl)naphthalene (3'dm).[4] A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.89 (dq, *J* = 12.3, 7.6 Hz, 1 H), 2.00–2.12 (m, 2 H), 2.36–2.44 (m, 1 H), 4.00 (q, *J* = 7.5 Hz, 1 H), 4.18 (q, *J* = 7.0 Hz, 1 H), 5.07 (t, *J* = 7.3 Hz, 1 H), 7.41–7.49 (m, 3 H), 7.78–7.85 (m, 4 H).

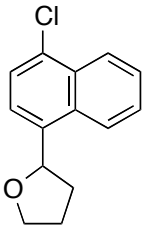
(2-Tetrahydrofuranyl)-substituted 1-chloronaphthalene (7dm). This product was obtained as a mixture (2:6:48:35:7:2) of 2-, 3-, 4-, 5-, 6- and 7-(2-tetrahydrofuranyl)-1-chloronaphthalenes.



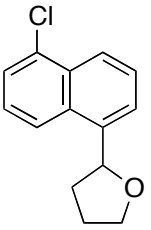
1-Chloro-2-(2-tetrahydrofuranyl)naphthalene (7dm-a). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.10–2.40 (m, 4 H), 4.00 (dt, *J* = 8.1, 6.4 Hz, 1 H), 4.39 (dt, *J* = 7.6, 5.9 Hz, 1 H), 5.78 (dd, *J* = 10.1, 6.9 Hz, 1 H), 7.41 (d, *J* = 8.8 Hz, 1 H), 7.44–7.52 (m, 2 H), 7.68 (d, *J* = 8.8 Hz, 1 H), 7.81 (d, *J* = 8.6 Hz, 1 H), 8.44 (dd, *J* = 8.2, 1.1 Hz, 1 H). GC–MS (the first peak on GC among the isomers) m/z (% relative intensity, ion) 234 (10, M+2), 232 (27, M), 197 (100, M–Cl), 189 (25), 155 (56).



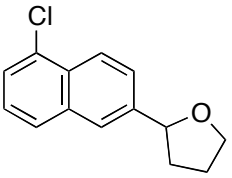
1-Chloro-3-(2-tetrahydrofuranyl)naphthalene (7dm-b). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.83–1.92 (m, 1 H), 1.97–2.13 (m, 2 H), 2.49–2.61 (m, 1 H), 4.03 (q, *J* = 7.2 Hz, 1 H), 4.22 (dt, *J* = 8.0, 5.5 Hz, 1 H), 5.61 (t, *J* = 7.0 Hz, 1 H), 7.53–7.63 (m, 2 H), 7.56 (s, 2 H), 7.98 (d, *J* = 7.6 Hz, 1 H), 8.32 (dd, *J* = 8.3, 1.6 Hz, 1 H). GC–MS (the fifth peak on GC among the isomers) m/z (% relative intensity, ion) 234 (6, M+2), 232 (20, M), 197 (100, M–Cl), 189 (26) 155 (24).



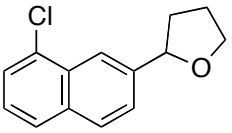
1-Chloro-4-(2-tetrahydrofuranyl)naphthalene (7dm-c). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.75 (dq, *J* = 12.6, 7.7 Hz, 1 H), 1.98–2.21 (m, 2 H), 2.60 (sext, *J* = 7.2 Hz, 1 H), 4.03 (q, *J* = 7.0 Hz, 1 H), 4.22 (q, *J* = 6.7 Hz, 1 H), 5.48 (t, *J* = 7.1 Hz, 1 H), 7.50 (td, *J* = 6.9, 1.3 Hz, 1 H), 7.58 (td, *J* = 6.9, 1.4 Hz, 1 H), 7.66 (d, *J* = 8.6 Hz, 1 H), 7.78 (d, *J* = 8.6 Hz, 1 H), 7.83 (d, *J* = 8.2 Hz, 1 H), 8.30 (d, *J* = 8.6 Hz, 1 H). GC–MS (the second peak on GC among the isomers) m/z (% relative intensity, ion) 234 (10, M+2), 232 (29, M), 197 (100, M–Cl), 189 (35), 155 (38).



1-Chloro-5-(2-tetrahydrofuranyl)naphthalene (7dm-d). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.89 (dt, *J* = 12.3, 6.7 Hz, 1 H), 1.98–2.13 (m, 2 H), 2.56 (dq, *J* = 12.4, 7.6 Hz, 1 H), 4.04 (q, *J* = 7.2 Hz, 1 H), 4.24 (td, *J* = 8.0, 5.5 Hz, 1 H), 5.62 (t, *J* = 7.0 Hz, 1 H), 7.41 (dd, *J* = 8.6, 7.6 Hz, 1 H), 7.55–7.61 (m, 2 H), 7.71 (d, *J* = 7.3 Hz, 1 H), 7.91 (d, *J* = 8.6 Hz, 1 H), 8.23 (d, *J* = 8.5 Hz, 1 H). GC–MS (the fourth peak on GC among the isomers) m/z (% relative intensity, ion) 234 (32, M+2), 232 (100, M), 197 (25, M–Cl), 189 (60), 155 (30).



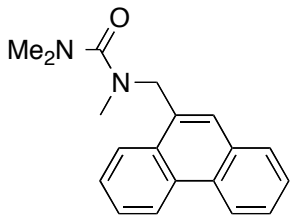
1-Chloro-6-(2-tetrahydrofuranyl)naphthalene (7dm-e). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.88 (dq, *J* = 12.3, 7.5 Hz, 1 H), 2.00–2.10 (m, 2 H), 2.40 (sext, *J* = 7.0 Hz, 1 H), 4.00 (q, *J* = 6.9 Hz, 1 H), 4.17 (dt, *J* = 8.3, 6.9 Hz, 1 H), 5.03 (t, *J* = 7.3 Hz, 1 H), 7.50–7.60 (m, 3 H), 7.71 (s, 1 H), 7.83 (d, *J* = 7.9 Hz, 1 H), 8.23 (d, *J* = 8.1 Hz, 1 H). GC–MS (the sixth peak on GC among the isomers) m/z (% relative intensity, ion) 234 (10, M+2), 232 (29, M), 197 (100, M–Cl), 189 (40), 155 (20).



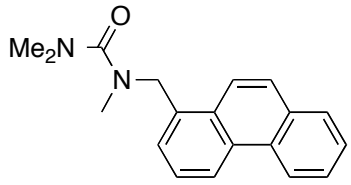
1-Chloro-7-(2-tetrahydrofuranyl)naphthalene (7dm-f). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.90 (dq, *J* = 12.4, 7.6 Hz, 1 H), 2.00–2.13 (m, 2 H), 2.43 (sext, *J* = 7.1 Hz, 1 H), 4.02 (dt, *J* = 7.7, 6.5 Hz, 1 H), 4.19 (q, *J* = 6.8 Hz, 1 H), 5.11 (t, *J* = 7.2 Hz, 1 H), 7.35 (dd, *J* = 8.2, 7.6 Hz, 1 H), 7.53 (dd, *J* = 8.6, 1.7 Hz, 1 H), 7.56 (dd, *J* = 7.5, 1.1 Hz, 1 H), 7.74 (d, *J* = 8.2 Hz, 1 H), 7.84 (d, *J* = 8.5 Hz, 1 H), 8.20 (d, *J* = 0.9 Hz, 1

H). GC–MS (the third peak on GC among the isomers) m/z (% relative intensity, ion) 234 (23, M+2), 232 (65, M), 197 (15, M–Cl), 189 (100), 155 (10).

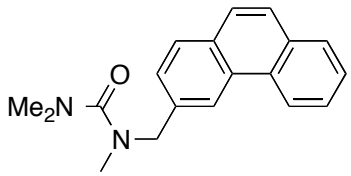
The coupling products in Scheme 6



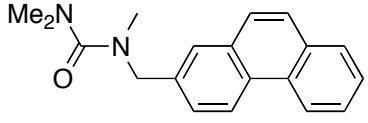
***N,N,N'*-Trimethyl-*N'*-(9-phenanthylmethyl)urea (3an).** A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.81 (s, 3 H), 2.84 (s, 6 H), 4.88 (s, 2 H), 7.57–7.74 (m, 4 H), 7.71 (s, 1 H), 7.88 (d, *J* = 7.7 Hz, 1 H), 7.99 (d, *J* = 8.2 Hz, 1 H), 8.68 (d, *J* = 8.2 Hz, 1 H), 8.75 (d, *J* = 8.2 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.1, 38.8, 53.2, 122.7, 123.4, 124.0, 126.7, 126.816, 126.824, 126.9, 127.0, 128.6, 130.4, 130.7, 130.9, 131.6, 131.7, 165.7. HRMS (ESI) Calcd for $C_{19}H_{20}N_2O$: $[M+Na]^+$, 315.1468. Found: m/z 315.1466.



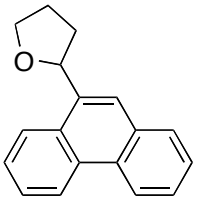
***N,N,N'*-Trimethyl-*N'*-(1-phenanthylmethyl)urea (3'an).** A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.74 (s, 3 H), 2.83 (s, 6 H), 4.90 (s, 2 H), 7.57 (d, *J* = 7.3 Hz, 1 H), 7.58–7.64 (m, 2 H), 7.67 (t, *J* = 6.7 Hz, 1 H), 7.79 (d, *J* = 9.2 Hz, 1 H), 7.85–7.93 (m, 2 H), 8.69 (d, *J* = 8.1 Hz, 1 H), 8.71 (d, *J* = 8.1 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.1, 38.8, 52.9, 122.1, 122.6, 123.1, 126.2, 126.8, 126.9, 127.2, 127.4, 128.7, 130.4, 130.8, 130.9, 131.8, 134.3, 165.7. HRMS (ESI) Calcd for $C_{19}H_{20}N_2O$: $[M+Na]^+$, 315.1468. Found: m/z 315.1470.



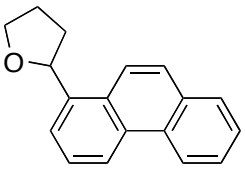
***N,N,N'*-Trimethyl-*N'*-(3-phenanthylmethyl)urea (3''an).** A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.81 (s, 3 H), 2.90 (s, 6 H), 4.64 (s, 2 H), 7.53 (d, *J* = 8.2 Hz, 1 H), 7.60 (t, *J* = 7.7 Hz, 1 H), 7.66 (t, *J* = 7.1 Hz, 1 H), 7.73 (s, 2 H), 7.87 (d, *J* = 8.2 Hz, 1 H), 7.89 (d, *J* = 8.2 Hz, 1 H), 8.58 (s, 1 H), 8.68 (d, *J* = 8.2 Hz, 1 H). The amount (0.2 mg) of a pure sample of this compound was too small to give any peaks in ^{13}C NMR. HRMS (ESI) Calcd for $C_{19}H_{20}N_2O$: $[M+Na]^+$, 315.1468. Found: m/z 315.1472.



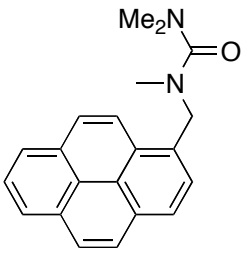
***N,N,N'*-Trimethyl-*N'*-(2-phenanthylmethyl)urea (3'''an).** A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.80 (s, 3 H), 2.89 (s, 6 H), 4.59 (s, 2 H), 7.59 (d, *J* = 7.0 Hz, 1 H), 7.59 (t, *J* = 8.0 Hz, 1 H), 7.66 (td, *J* = 7.1, 1.5 Hz, 1 H) 7.72 (d, *J* = 8.8 Hz, 1 H), 7.75 (d, *J* = 9.1 Hz, 1 H), 7.77 (s, 1 H), 7.89 (d, *J* = 7.6 Hz, 1 H), 8.66 (d, *J* = 8.6 Hz, 1 H), 8.67 (d, *J* = 8.3 Hz, 1 H). The amount (0.6 mg) of a pure sample of this compound was too small to give any peaks in ^{13}C NMR. HRMS (ESI) Calcd for $C_{19}H_{20}N_2O$: $[M+Na]^+$, 315.1468. Found: m/z 315.1466.



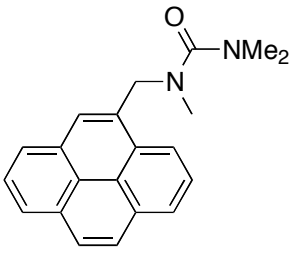
9-(2-Tetrahydrofuranyl)phenanthrene (3dn). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.88–2.20 (m, 3 H), 2.62 (sext, *J* = 7.3 Hz, 1 H), 4.09 (q, *J* = 7.9 Hz, 1 H), 4.32 (q, *J* = 7.7 Hz, 1 H), 5.68 (t, *J* = 6.9 Hz, 1 H), 7.55–7.70 (m, 4 H), 7.91 (d, *J* = 6.5 Hz, 1 H), 7.92 (s, 1 H), 8.01 (d, *J* = 8.0 Hz, 1 H), 8.67 (d, *J* = 8.1 Hz, 1 H), 8.75 (d, *J* = 8.1 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.0, 33.7, 69.0, 78.1, 122.51, 122.54, 123.4, 124.2, 126.3, 126.4, 126.6, 126.8, 128.9, 129.9, 130.0, 130.9, 131.8, 137.6. HRMS (ESI) Calcd for $C_{18}H_{16}O$: $[M+Na]^+$, 271.1093. Found: m/z 271.1086.



1-(2-Tetrahydrofuranyl)phenanthrene (3'dn). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.94 (dt, *J* = 19.1, 6.9 Hz, 1 H), 2.00–2.16 (m, 2 H), 2.61 (sext, *J* = 7.3 Hz, 1 H), 4.07 (q, *J* = 7.8 Hz, 1 H), 4.27 (q, *J* = 7.6 Hz, 1 H), 5.72 (t, *J* = 7.0 Hz, 1 H), 7.60 (t, *J* = 7.1 Hz, 1 H), 7.65 (t, *J* = 7.7 Hz, 1 H), 7.66 (t, *J* = 7.0 Hz, 1 H), 7.75–7.84 (m, 2 H), 7.90 (d, *J* = 7.9 Hz, 1 H), 7.93 (d, *J* = 9.2 Hz, 1 H), 8.65 (d, *J* = 8.3 Hz, 1 H), 8.71 (d, *J* = 8.4 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 34.2, 69.0, 78.3, 121.9, 122.2, 122.9, 123.1, 126.4, 126.7, 126.8, 127.0, 128.6, 128.8, 130.7, 130.8, 131.6, 140.2. HRMS (ESI) Calcd for $C_{18}H_{16}O$: $[M+Na]^+$, 271.1093. Found: m/z 271.1090.

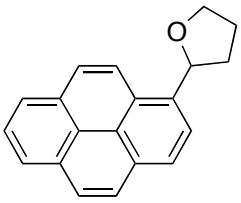


***N*,*N*,*N*'-Trimethyl-*N*'-(1-pyrenylmethyl)urea (3ao).** This product could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **3'ao** (**3ao**:**3'ao** = 93:7). ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.77 (s, 3 H), 2.86 (s, 6 H), 5.12 (s, 2 H), 7.99 (d, *J* = 7.6 Hz, 1 H), 8.01 (t, *J* = 8.2 Hz, 1 H), 8.06 (s, 2 H), 8.13 (d, *J* = 9.7 Hz, 1 H), 8.16 (d, *J* = 8.3 Hz, 1 H), 8.18–8.23 (m, 3 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.2, 38.9, 52.6, 123.0, 124.8, 124.9, 125.2, 125.3, 125.36, 125.40, 126.2, 126.9, 127.4, 127.6, 127.9, 129.3, 130.97, 131.03, 131.5, 165.7. GC-MS (appears later than regioisomer **3'ao** on GC) m/z (% relative intensity, ion) 316 (34, M), 244 (16), 242 (38), 215 (100), 72 (35).

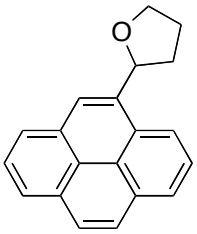


***N*,*N*,*N*'-Trimethyl-*N*'-(9-pyrenylmethyl)urea (3'ao).** This product could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **3ao** (**3'ao**:**3ao** = 91:9). ^{1}H NMR (600 MHz, $CDCl_3$) δ 2.86 (s, 6 H), 2.87 (s, 3 H), 5.04 (s, 2 H), 8.02 (t, *J* = 8.2 Hz, 1 H), 8.03 (t, *J* = 8.2 Hz, 1 H), 8.05 (s, 1 H), 8.09 (s, 2 H), 8.20 (d, *J* = 7.6 Hz, 2 H), 8.22 (d, *J* = 6.9 Hz, 1 H), 8.26 (d, *J* = 8.2 Hz, 1 H). ^{13}C

NMR (125 MHz, $CDCl_3$) δ 37.2, 38.9, 53.4, 121.0, 124.4, 125.31, 125.32, 125.5, 126.1, 126.2, 127.0, 127.4, 127.8, 130.0, 130.7, 131.2, 131.7, 132.7, 165.8. GC-MS (appears earlier than regioisomer **3ao** on GC) m/z (% relative intensity, ion) 316 (57, M), 244 (94), 242 (47), 215 (100), 72 (63).

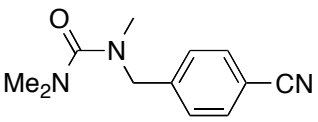


1-(2-Tetrahydrofuranyl)pyrene (3do). This product could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **3'do** (**3do**:**3'do** = 93:7). ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.96–2.22 (m, 3 H), 2.65–2.76 (m, 1 H), 4.14 (q, *J* = 6.9 Hz, 1 H), 4.35 (q, *J* = 7.6 Hz, 1 H), 5.95 (t, *J* = 6.8 Hz, 1 H), 8.00 (t, *J* = 7.6 Hz, 1 H), 8.04 (d, *J* = 8.9 Hz, 1 H), 8.06 (d, *J* = 8.9 Hz, 1 H), 8.11 (d, *J* = 9.6 Hz, 1 H), 8.16–8.23 (m, 4 H), 8.24 (d, *J* = 9.6 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.4, 34.9, 69.1, 78.5, 122.7, 123.0, 125.0, 125.08, 125.09, 125.3, 126.0, 127.1, 127.46, 127.50, 127.7, 130.6, 130.9, 131.6, 137.5. GC-MS (appears later than regioisomer **3'do** on GC) m/z (% relative intensity, ion) 272 (83, M), 229 (43, M–C_3H_7), 215 (25), 202 (100).

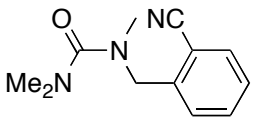


9-(2-Tetrahydrofuranyl)pyrene (3'do). This product could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **3do** (**3'do**:**3do** = 89:11). ^{1}H NMR (600 MHz, $CDCl_3$) δ 1.95–2.21 (m, 3 H), 2.66–2.79 (m, 1 H), 4.15 (q, *J* = 6.9 Hz, 1 H), 4.35–4.44 (m, 1 H), 5.85 (t, *J* = 6.2 Hz, 1 H), 8.01 (t, *J* = 7.6 Hz, 1 H), 8.03 (t, *J* = 7.6 Hz, 1 H), 8.08 (s, 2 H), 8.17 (dd, *J* = 7.6, 1.4 Hz, 1 H), 8.20 (d, *J* = 7.5 Hz, 1 H), 8.21 (d, *J* = 7.5 Hz, 1 H), 8.25 (s, 1 H), 8.26 (d, *J* = 8.9 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 33.9, 69.1, 78.3, 121.1, 122.7, 124.0, 125.0, 125.1, 125.6, 125.7, 126.2, 127.5, 127.7, 129.2, 130.9, 131.1, 131.8, 138.6. GC-MS (appears earlier than regioisomer **3do** on GC) m/z (% relative intensity, ion) 272 (91, M), 229 (35, M–C_3H_7), 215 (33), 202 (100).

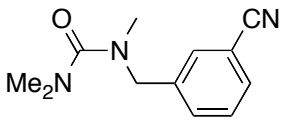
The coupling products in Scheme 7



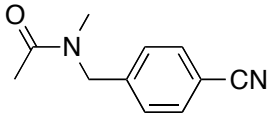
***N*-(4-Cyanophenylmethyl)-*N*,*N*',*N*'-trimethylurea (3ap).** A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.77 (s, 3 H), 2.86 (s, 6 H), 4.42 (s, 2 H), 7.40 (d, *J* = 8.1 Hz, 2 H), 7.63 (d, *J* = 8.1 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 37.3, 38.8, 53.9, 111.2, 118.9, 128.5, 132.5, 144.1, 165.4. HRMS (ESI) Calcd for $C_{12}H_{15}N_3O$: $[M+Na]^+$, 240.1107. Found: m/z 240.1108. GC–MS (the third peak on GC among the isomers) m/z (% relative intensity, ion) 217 (75, M), 145 (99), 116 (100), 72 (95).



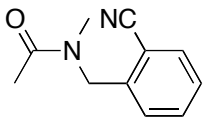
***N*-(2-Cyanophenylmethyl)-*N*,*N*',*N*'-trimethylurea (3'ap).** A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.84 (s, 3 H), 2.85 (s, 6 H), 4.53 (s, 2 H), 7.36 (td, *J* = 7.6, 1.0 Hz, 1 H), 7.51 (d, *J* = 7.6 Hz, 1 H), 7.56 (td, *J* = 7.9, 1.4 Hz, 1 H), 7.65 (dd, *J* = 7.7, 1.1 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 38.0, 38.7, 52.7, 112.2, 117.8, 127.8, 129.3, 133.1, 133.2, 142.6, 165.2. HRMS (ESI) Calcd for $C_{12}H_{15}N_3O$: $[M+Na]^+$, 240.1107. Found: m/z 240.1105. GC–MS (the first peak on GC among the isomers) m/z (% relative intensity, ion) 217 (10, M), 145 (84), 116 (97), 72 (100).



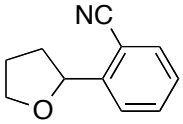
***N*-(3-Cyanophenylmethyl)-*N*,*N*',*N*'-trimethylurea (3''ap).** This product could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **3ap** (**3a''p**:**3ap** = 26:74). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.76 (s, 3 H), 2.86 (s, 6 H), 4.39 (s, 2 H), 7.44 (t, *J* = 7.6 Hz, 1 H), 7.54 (d, *J* = 8.4 Hz, 1 H), 7.56 (d, *J* = 7.8 Hz, 1 H), 7.58 (s, 1 H). The amount (1.4 mg) of a sample of this compound was too small to give any peaks belonging to **3''ap** in ^{13}C NMR. GC-MS (the second peak on GC among the isomers) m/z (% relative intensity, ion) 217 (20, M), 145 (37), 116 (60), 72 (100).



***N*-(4-Cyanophenylmethyl)-*N*-methylacetamide (3bp).** A yellow oil. Observed as two rotamers of 71/29 ratio in ^{1}H NMR. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.17/2.12 (s, 3 H), 2.96/2.95 (s, 3 H), 4.62/4.59 (s, 2 H), 7.34/7.29 (d, *J* = 8.2/8.2 Hz, 2 H), 7.61/7.68 (d, *J* = 8.2/8.2 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.5, 21.8, 34.1, 36.1, 50.7, 54.1, 111.5, 112.0, 118.5, 118.8, 127.1, 128.6, 132.6, 133.0, 142.3, 143.1 171.0, 171.1. HRMS (ESI) Calcd for $C_{11}H_{12}N_2O$: $[M+Na]^+$, 211.0842. Found: m/z 211.0846.

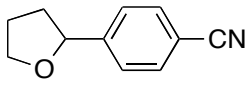


***N*-(2-Cyanophenylmethyl)-*N*-methylacetamide (3'bp).** A yellow oil. Observed as two rotamers of 72/28 ratio in ^{1}H NMR. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.19/2.14 (s, 3 H), 3.03/2.98 (s, 3 H), 4.82/4.76 (s, 2 H), 7.43/7.29 (d, *J* = 7.7/8.1 Hz, 1 H), 7.37/7.43 (t, *J* = 8.0 Hz for the major isomer; the coupling constant of the minor isomer could not be accurately read, 1 H), 7.56/7.63 (t, *J* = 7.7/6.1 Hz, 1 H), 7.65/7.72 (d, *J* = 7.7/7.7 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.5, 21.8, 34.1, 36.4, 49.1, 52.7, 111.3, 112.1, 117.0, 117.7, 126.5, 128.1, 128.4, 129.1, 133.0, 133.4, 133.6, 133.7, 140.6, 141.5, 171.3. HRMS (ESI) Calcd for $C_{11}H_{12}N_2O$: $[M+Na]^+$, 211.0842. Found: m/z 211.0841.

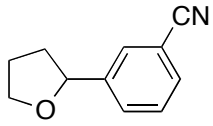


2-(2-Tetrahydrofuranyl)benzonitrile (3dp). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.76 (dq, *J* = 12.5, 8.0 Hz, 1 H), 2.06 (quint, *J* = 6.4 Hz, 2 H), 2.56 (sext, *J* = 6.7 Hz, 1 H), 4.00 (q, *J* = 6.9 Hz, 1 H), 4.18 (q, *J* = 6.9 Hz, 1 H), 5.20 (t, *J* = 7.5 Hz, 1 H), 7.34 (td, *J* = 7.7, 1.9

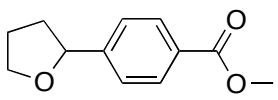
Hz, 1 H), 7.57 (td, *J* = 8.0, 1.3 Hz, 1 H), 7.59 (dd, *J* = 8.0, 1.8 Hz, 1 H), 7.63 (d, *J* = 7.9 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.4, 34.6, 69.4, 79.0, 109.9. 117.8, 126.1, 127.6, 133.06, 133.10, 148.1. HRMS (ESI) Calcd for $C_{11}H_{11}NO$: $[M+Na]^+$, 196.0733. Found: m/z 196.0731. GC-MS (the first peak on GC among the isomers) m/z (% relative intensity, ion) 173 (5, M), 172 (16, M–1), 145 (33), 130 (100).



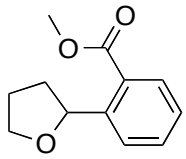
4-(2-Tetrahydrofuranyl)benzonitrile (3'dp). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.74 (dq, *J* = 12.4, 7.9 Hz, 1 H), 1.95–2.08 (m, 2 H), 2.38 (sext, *J* = 7.1 Hz, 1 H), 3.96 (q, *J* = 7.2 Hz, 1 H), 4.10 (dt, *J* = 8.3, 6.8 Hz, 1 H), 4.94 (t, *J* = 7.2 Hz, 1 H), 7.43 (d, *J* = 8.1 Hz, 2 H), 7.62 (d, *J* = 8.1 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 34.8, 69.1, 80.0, 111.0, 119.1, 126.3, 132.3, 149.4. HRMS (ESI) Calcd for $C_{11}H_{11}NO$: $[M+Na]^+$, 196.0733. Found: m/z 196.0731. GC-MS (the third peak on GC among the isomers) m/z (% relative intensity, ion) 173 (11, M), 172 (52, M–1), 145 (34), 130 (61), 42 (100).



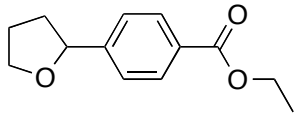
3-(2-Tetrahydrofuranyl)benzonitrile (3''dp). This product could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **3'dp** (**3''dp**:**3'dp** = 19:81). ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.70–1.80 (m, 1 H), 1.95–2.07 (m, 2 H), 2.34–2.43 (m, 1 H), 3.93–4.00 (m, 1 H), 4.06–4.15 (m, 1 H), 4.91 (t, *J* = 6.7 Hz, 1 H), 7.40–7.45 (m, 1 H), 7.54 (d, *J* = 7.5 Hz, 1 H), 7.55 (d, *J* = 7.6 Hz, 1 H), 7.65 (s, 1 H). The amount (1.0 mg) of a sample of this compound was too small to give any peaks belonging to **3''dp** in ^{13}C NMR. GC-MS (the second peak on GC among the isomers) m/z (% relative intensity, ion) 173 (8, M), 172 (42, M–1), 145 (25), 130 (100).



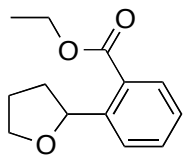
Methyl 4-(2-tetrahydrofuranyl)benzoate (3dq). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.78 (dq, *J* = 12.4, 7.7 Hz, 1 H), 2.01 (quint, *J* = 6.9 Hz, 2 H), 2.36 (sext, *J* = 6.7 Hz, 1 H), 3.91 (s, 3 H), 3.96 (q, *J* = 7.1 Hz, 1 H), 4.11, (q, *J* = 7.3 Hz, 1 H), 4.94 (t, *J* = 7.2 Hz, 1 H), 7.40 (d, *J* = 8.5 Hz, 2 H), 8.00 (d, *J* = 8.5 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 34.8, 52.1, 69.0, 80.3, 125.6, 129.1, 129.8, 149.1, 167.2. HRMS (ESI) Calcd for $C_{12}H_{14}O_3$: $[M+Na]^+$, 229.0835. Found: m/z 229.0840.



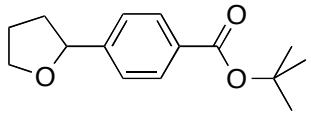
Methyl 2-(2-tetrahydrofuranyl)benzoate (3'dq). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.66 (sext, *J* = 6.8 Hz, 1 H), 1.88–2.05 (m, 2 H), 2.56 (sext, *J* = 7.3 Hz, 1 H), 3.89 (s, 3 H), 3.95 (q, *J* = 7.3 Hz, 1 H), 4.15, (q, *J* = 7.7 Hz, 1 H), 5.61 (t, *J* = 7.0 Hz, 1 H), 7.29 (t, *J* = 7.4 Hz, 1 H), 7.51 (t, *J* = 7.2 Hz, 1 H), 7.69 (d, *J* = 8.0 Hz, 1 H), 7.89 (d, *J* = 7.9 Hz, 1 H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 26.0, 34.9, 52.1, 69.1, 78.4, 125.9, 126.7, 127.7, 130.5, 132.5, 146.6 167.8. HRMS (ESI) Calcd for $C_{12}H_{14}O_3$: $[M+Na]^+$, 229.0835. Found: m/z 229.0830.



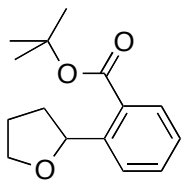
Ethyl 4-(2-tetrahydrofuranyl)benzoate (3dr). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.39 (t, *J* = 7.1 Hz, 3 H), 1.77 (dq, *J* = 12.4, 7.7 Hz, 1 H), 2.01 (quint, *J* = 7.0 Hz, 2 H), 2.36 (sext, *J* = 6.6 Hz, 1 H), 3.96 (q, *J* = 7.1 Hz, 1 H), 4.11 (q, *J* = 7.0 Hz, 1 H), 4.37 (q, *J* = 7.1 Hz, 2 H), 4.95 (t, *J* = 7.1 Hz, 1 H), 7.39 (d, *J* = 8.1 Hz, 2 H), 8.00 (d, *J* = 8.1 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.5, 26.1, 34.9, 61.0, 69.0, 80.3, 125.5, 129.4, 129.8, 149.0, 166.7. HRMS (ESI) Calcd for $C_{13}H_{16}O_3$: $[M+Na]^+$, 243.0992. Found: m/z 243.0995.



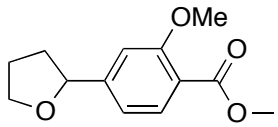
Ethyl 2-(2-tetrahydrofuranyl)benzoate (3'dr). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.39 (t, *J* = 7.1 Hz, 3 H), 1.67 (sext, *J* = 6.9 Hz, 1 H), 1.87–2.03 (m, 2 H), 2.56 (sext, *J* = 7.3 Hz, 1 H), 3.95 (q, *J* = 7.4 Hz, 1 H), 4.15 (q, *J* = 7.7 Hz, 1 H), 4.35 (q, *J* = 7.1 Hz, 2 H), 5.62 (t, *J* = 7.0 Hz, 1 H), 7.29 (t, *J* = 7.4 Hz, 1 H), 7.50 (t, *J* = 7.2 Hz, 1 H), 7.68 (d, *J* = 7.8 Hz, 1 H), 7.90 (d, *J* = 7.8 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 14.4, 26.0, 35.0, 61.1, 69.1, 78.4, 125.9, 126.7, 128.2, 130.4, 132.3, 146.4, 167.4. HRMS (ESI) Calcd for $C_{13}H_{16}O_3$: $[M+Na]^+$, 243.0992. Found: m/z 243.0985.



***tert*-Butyl 4-(2-tetrahydrofuranyl)benzoate (3ds).** A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.59 (s, 9 H), 1.76 (dq, *J* = 12.4, 7.7 Hz, 1 H), 2.00 (quint, *J* = 7.0 Hz, 2 H), 2.35 (sext, *J* = 6.7 Hz, 1 H), 3.96 (q, *J* = 7.1 Hz, 1 H), 4.11 (q, *J* = 7.0 Hz, 1 H), 4.94 (t, *J* = 7.2 Hz, 1 H), 7.37 (d, *J* = 8.2 Hz, 2 H), 7.95 (d, *J* = 8.2 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.1, 28.4, 34.9, 69.0, 80.4, 81.0, 125.4, 129.6, 131.1, 148.5, 165.8. HRMS (ESI) Calcd for $C_{15}H_{18}O_3$: $[M+Na]^+$, 271.1305. Found: m/z 271.1300.

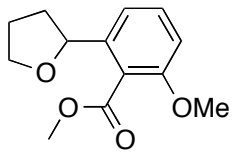


***tert*-Butyl 2-(2-tetrahydrofuranyl)benzoate (3'ds).** A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.59 (s, 9 H), 1.68 (sext, *J* = 7.0 Hz, 1 H), 1.85–2.03 (m, 2 H), 2.54 (sext, *J* = 7.2 Hz, 1 H), 3.95 (q, *J* = 7.4 Hz, 1 H), 4.15 (q, *J* = 7.7 Hz, 1 H), 5.60 (t, *J* = 6.9 Hz, 1 H), 7.26 (t, *J* = 8.0 Hz, 1 H), 7.46 (t, *J* = 7.6 Hz, 1 H), 7.64 (d, *J* = 8.0 Hz, 1 H), 7.80 (d, *J* = 7.7 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.0, 28.4, 35.1, 69.1, 78.3, 81.5, 125.8, 126.6, 130.1, 130.3, 131.7, 145.6, 167.0. HRMS (ESI) Calcd for $C_{15}H_{20}O_3$: $[M+Na]^+$, 271.1305. Found: m/z 243.1300.

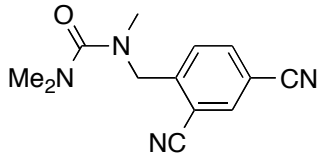


Methyl 4-(2-tetrahydrofuranyl)-2-methoxybenzoate (3dt). A yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.77 (dq, *J* = 12.3, 7.7 Hz, 1 H), 2.00 (quint, *J* = 7.0 Hz, 2 H), 2.36 (sext, *J* = 6.8 Hz, 1 H), 3.88 (s, 3 H), 3.92 (s, 3 H), 3.95 (q, *J* = 7.1 Hz, 1 H), 4.10 (q, *J* = 7.0 Hz, 1 H), 4.91 (t, *J* = 7.3 Hz, 1 H), 6.90 (d, *J* = 8.0 Hz, 1 H), 6.99 (s, 1 H), 7.77

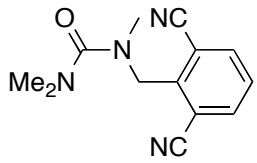
(d, J = 8.0 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.0, 34.7, 52.0, 56.1, 68.9, 80.2, 109.1, 117.3, 118.6, 132.0, 150.2, 159.6, 166.7. HRMS (ESI) Calcd for $C_{13}H_{16}O_4$: $[M+Na]^+$, 259.0941. Found: m/z 259.0937.



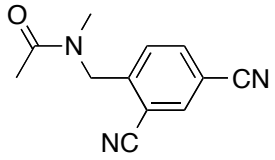
Methyl 2-methoxy-6-(2-tetrahydrofuranyl)benzoate (3'dt). A yellow oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.81 (dq, J = 12.2, 8.0 Hz, 1 H), 1.90–2.06 (m, 2 H), 2.29 (sext, J = 7.1 Hz, 1 H), 3.83 (s, 3 H), 3.89 (s, 3 H), 3.90 (q, J = 7.9 Hz, 1 H), 4.03 (q, J = 7.1 Hz, 1 H), 4.91 (t, J = 7.4 Hz, 1 H), 6.82 (d, J = 8.3 Hz, 1 H), 7.02 (d, J = 7.8 Hz, 1 H), 7.32 (t, J = 8.1 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.2, 35.0, 52.3, 56.2, 69.1, 79.0, 109.8, 117.9, 121.6, 130.6, 142.8, 156.5, 168.8. HRMS (ESI) Calcd for $C_{13}H_{16}O_4$: $[M+Na]^+$, 259.0941. Found: m/z 259.0942.



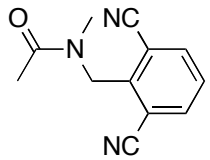
***N*-[(2,4-Dicyanophenyl)methyl]-*N*,*N*',*N*'-trimethylurea (3au).** A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.86 (s, 6 H), 2.92 (s, 3 H), 4.53 (s, 2 H), 7.68 (d, J = 8.1 Hz, 1 H), 7.82 (dd, J = 8.1, 1.7 Hz, 1 H), 7.92 (d, J = 1.7 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 38.5, 38.8, 52.8, 112.4, 113.6, 115.7, 116.8, 130.6, 135.9, 136.3, 148.0, 164.7. HRMS (ESI) Calcd for $C_{13}H_{14}N_4O$: $[M+Na]^+$, 265.1060. Found: m/z 265.1059.



***N*-[(2,6-Dicyanophenyl)methyl]-*N*,*N*',*N*'-trimethylurea (3'au).** A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.85 (s, 6 H), 3.00 (s, 3 H), 4.58 (s, 2 H), 7.48 (t, J = 7.9 Hz, 1 H), 7.84 (d, J = 7.9 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 38.3, 40.1, 52.0, 115.0, 116.4, 128.4, 137.0, 146.9, 164.5. HRMS (ESI) Calcd for $C_{13}H_{14}N_4O$: $[M+Na]^+$, 265.1060. Found: m/z 265.1067.

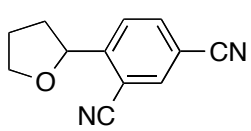


***N*-[(2,4-Dicyanophenyl)methyl]-*N*-methylacetamide (3bu).** A yellow oil. Observed as two rotamers of 87/13 ratio in ^{1}H NMR. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.19/2.11 (s, 3 H), 3.09/2.98 (s, 3 H), 4.82/4.80 (s, 2 H), 7.57/7.44 (d, J = 8.1/8.1 Hz, 1 H), 7.82/7.92 (d, J = 8.1/8.1 Hz, 1 H), 7.93/8.00 (s, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 21.4, 21.7, 34.3, 37.0, 49.7, 52.7, 112.7, 112.9, 113.3, 113.4, 114.8, 115.6, 116.3, 116.7, 127.5, 130.0, 136.2, 136.7, 136.8, 145.8, 146.7, 171.0, 171.5. HRMS (ESI) Calcd for $C_{12}H_{11}N_3O$: $[M+Na]^+$, 236.0794. Found: m/z 236.0792.

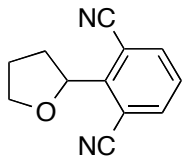


***N*-[(2,6-Dicyanophenyl)methyl]-*N*-methylacetamide (3'bu).** A yellow oil. Observed as two rotamers of 91/9 ratio in ^{1}H NMR. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.16/2.36 (s, 3 H), 3.19/2.79 (s, 3 H), 4.88/4.93 (s, 2 H), 7.49/7.63 (t, J = 8.0/7.7 Hz, 1 H), 7.87/7.96 (d, J =

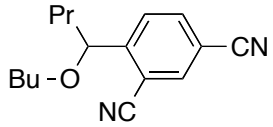
8.0/7.7 Hz, 2 H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 21.6, 22.0, 29.8, 38.3, 50.3, 51.3, 114.0, 115.0, 115.7, 116.2, 128.4, 129.8, 137.4, 137.7, 144.1, 146.0, 172.1. HRMS (ESI) Calcd for $C_{12}H_{11}N_3O$: $[M+Na]^+$, 236.0794. Found: m/z 236.0791.



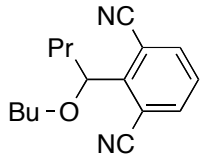
2,4-Dicyano-1-(2-tetrahydrofuranyl)benzene (3du). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.73 (dq, *J* = 12.6, 8.0 Hz, 1 H), 2.00–2.15 (m, 2 H), 2.59–2.69 (m, 1 H), 4.03 (q, *J* = 8.0 Hz, 1 H), 4.18 (q, *J* = 8.2 Hz, 1 H), 5.22 (t, *J* = 7.3 Hz, 1 H), 7.77 (d, *J* = 8.3 Hz, 1 H), 7.84 (dd, *J* = 8.3, 1.7 Hz, 1 H), 7.91 (d, *J* = 1.7 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.3, 34.5, 69.7, 78.5, 111.4, 112.3, 115.6, 116.9, 127.3, 136.2, 136.3, 153.3. HRMS (ESI) Calcd for $C_{12}H_{10}N_2O$: $[M+Na]^+$, 221.0685. Found: m/z 221.0690.



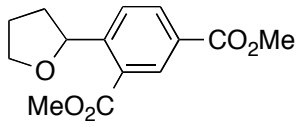
1,3-Dicyano-2-(2-tetrahydrofuranyl)benzene (3'du). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.94 (dq, *J* = 12.5, 9.1 Hz, 1 H), 2.10–2.21 (m, 1 H), 2.22–2.31 (m, 1 H), 2.50–2.58 (m, 1 H), 4.08 (td, *J* = 8.2, 4.8 Hz, 1 H), 4.37 (q, *J* = 8.1 Hz, 1 H), 5.39 (dd, *J* = 8.9, 6.7 Hz, 1 H), 7.49 (t, *J* = 7.8 Hz, 1 H), 7.88 (d, *J* = 7.9 Hz, 2 H). ^{13}C NMR (150 MHz, $CDCl_3$) δ 27.3, 34.9, 70.0, 78.9, 112.5, 116.2, 128.6, 137.7, 151.3. HRMS (ESI) Calcd for $C_{12}H_{10}N_2O$: $[M+Na]^+$, 221.0685. Found: m/z 221.0689.



1-(1-Butoxybutyl)-2,4-dicyanobenzene (3eu). ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.90 (t, *J* = 7.3 Hz, 3 H), 0.94 (t, *J* = 7.4 Hz, 3 H), 1.32–1.44 (m, 3 H), 1.45–1.67 (m, 4 H), 1.69–1.79 (m, 1 H), 3.28 (dt, *J* = 9.2, 6.5 Hz, 1 H), 3.34 (dt, *J* = 9.2, 6.5 Hz, 1 H), 4.66 (dd, *J* = 8.3, 4.7 Hz, 1 H), 7.73 (d, *J* = 8.2 Hz, 1 H), 7.86 (dd, *J* = 8.2, 1.7 Hz, 1 H), 7.91 (d, *J* = 1.8 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.8, 13.9, 19.0, 19.5, 32.0, 40.0, 70.0, 79.4, 112.5, 112.6, 115.5, 116.9, 128.1, 136.1, 136.2, 153.3. HRMS (ESI) Calcd for $C_{16}H_{20}N_2O$: $[M+Na]^+$, 279.1468. Found: m/z 279.1463.



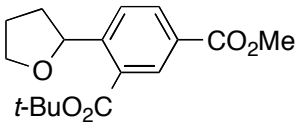
2-(1-Butoxybutyl)-1,3-dicyanobenzene (3'eu). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.90 (t, *J* = 7.5 Hz, 3 H), 0.97 (t, *J* = 7.5 Hz, 3 H), 1.30–1.45 (m, 3 H), 1.55–1.67 (m, 3 H), 1.68–1.78 (m, 1 H), 2.03–2.12 (m, 1 H), 3.32 (dt, *J* = 9.3, 6.6 Hz, 1 H), 3.37 (dt, *J* = 9.1, 6.8 Hz, 1 H), 4.81 (dd, *J* = 8.3, 5.4 Hz, 1 H), 7.50 (t, *J* = 7.8 Hz, 1 H), 7.88 (d, *J* = 7.8 Hz, 2 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.9, 19.0, 19.3, 19.5, 31.8, 38.6, 70.3, 80.0, 113.1, 116.3, 128.6, 137.8, 151.4. HRMS (ESI) Calcd for $C_{16}H_{20}N_2O$: $[M+Na]^+$, 279.1468. Found: m/z 279.1464.

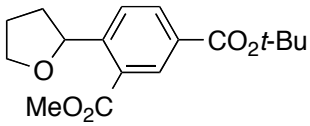


Dimethyl 4-(2-tetrahydrofuranyl)isophthalate (3dv). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.55–1.75 (m, 1 H), 1.85–2.16 (m, 2 H), 2.59 (sext, *J* = 7.6 Hz, 1 H), 3.91 (s, 3

H), 3.93 (s, 3 H), 3.96 (q, J = 7.2 Hz, 1 H), 4.15 (q, J = 7.6 Hz, 1 H), 5.65 (t, J = 7.0 Hz, 1 H), 7.78 (d, J = 8.4 Hz, 1 H), 8.15 (dd, J = 8.4, 1.6 Hz, 1 H), 8.56 (d, J = 1.6 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.0, 34.9, 52.3, 52.4, 69.3, 78.3, 126.3, 127.9, 128.8, 131.9, 133.2, 151.7, 166.4, 166.9. HRMS (ESI) Calcd for $C_{14}H_{16}O_5$: $[M+Na]^+$, 287.0890. Found: m/z 287.0892.

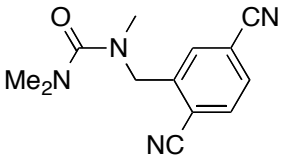
Two isomers of *tert*-butyl methyl 4-(2-tetrahydrofuranyl)isophthalates were also produced and were separated each other. However, they could not be determined to be which isomers.



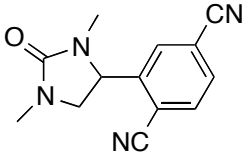


The major isomer: A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.61 (s, 9 H), 1.62–1.70 (m, 1 H), 1.87–2.05 (m, 2 H), 2.57 (sext, J = 7.4 Hz, 1 H), 3.93 (s, 3 H), 3.97 (q, J = 7.4 Hz, 1 H), 4.15 (q, J = 7.7 Hz, 1 H), 5.62 (t, J = 7.0 Hz, 1 H), 7.74 (d, J = 8.2 Hz, 1 H), 8.10 (dd, J = 8.2, 1.6 Hz, 1 H), 8.43 (d, J = 1.6 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.0, 28.3, 35.0, 52.3, 69.2, 78.3, 82.1, 126.1, 128.7, 130.4, 131.6, 132.5, 150.7, 166.2, 166.6. HRMS (ESI) Calcd for $C_{17}H_{22}O_5$: $[M+Na]^+$, 329.1359. Found: m/z 329.1362.

The minor isomer: A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 1.56–1.67 (m, 1 H), 1.60 (s, 9 H), 1.86–2.05 (m, 2 H), 2.58 (sext, J = 7.6 Hz, 1 H), 3.91 (s, 3 H), 3.96 (q, J = 7.4 Hz, 1 H), 4.16 (q, J = 7.9 Hz, 1 H), 5.64 (t, J = 7.0 Hz, 1 H), 7.75 (d, J = 8.2 Hz, 1 H), 8.10 (dd, J = 8.2, 1.6 Hz, 1 H), 8.49 (d, J = 1.6 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.0, 28.3, 34.9, 52.3, 69.3, 78.3, 81.6, 126.1, 127.7, 130.7, 131.7, 133.1, 151.1, 165.1, 167.1. HRMS (ESI) Calcd for $C_{17}H_{22}O_5$: $[M+Na]^+$, 329.1359. Found: m/z 329.1358.

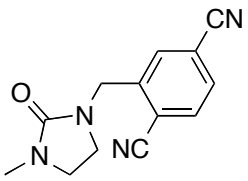


***N*-[(2,5-Dicyanophenyl)methyl]-*N*,*N*',*N*'-trimethylurea (3aw).** A yellow solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.87 (s, 6 H), 2.92 (s, 3 H), 4.51 (s, 2 H), 7.64 (dd, J = 8.0, 1.6 Hz, 1 H), 7.75 (d, J = 8.0 Hz, 1 H), 7.82 (d, J = 1.6 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 38.5, 38.8, 52.3, 116.1, 116.3, 116.8, 117.3, 131.1, 133.0, 133.6, 144.4, 164.7. HRMS (ESI) Calcd for $C_{13}H_{14}N_4O$: $[M+Na]^+$, 265.1060. Found: m/z 265.1059.

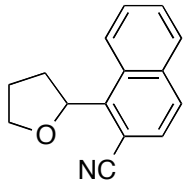


1,3-Dimethyl-4-(2,5-dicyanophenyl)-2-imidazolidinone (3fw). This product could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **3'fw** (**3fw**:**3'fw** = 96:4). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.73 (s, 3 H), 2.85 (s, 3 H), 3.04 (dd, J = 9.1, 7.3 Hz, 1 H), 3.89 (t, J = 9.2 Hz, 1 H), 4.87 (dd, J = 9.3 Hz, 7.4 Hz, 1 H), 7.74 (dd, J = 8.0, 1.6 Hz, 1 H), 7.80 (d, J = 1.6 Hz, 1 H), 7.83 (d, J =

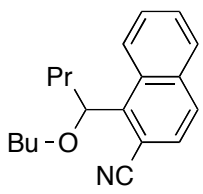
8.0 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.6, 31.2, 52.6, 58.0, 115.4, 115.7, 116.8, 117.8, 130.6, 132.2, 134.1, 145.8, 161.2. GC-MS (the first peak on GC among the isomers) m/z (% relative intensity, ion) 240 (43, M), 168 (42), 113 (100), 42 (80).



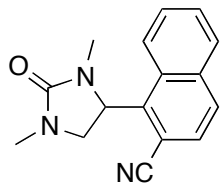
1-[(2,5-Dicyanophenyl)methyl]-3-methyl-2-imidazolidinone (3'fw). This product could not be isolated in a pure form. The following data were obtained from a spectrum of a mixture with **3fw** (**3'fw**:**3fw** = 80:20). ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.86 (s, 3 H), 3.32–3.37 (m, 2 H), 3.38–3.43 (m, 2 H), 4.60 (s, 2 H), 7.66 (dd, *J* = 8.0, 1.6 Hz, 1 H), 7.76 (d, *J* = 8.0 Hz, 1 H), 7.83 (d, *J* = 1.6 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 31.5, 43.4, 45.1, 46.7, 115.95, 116.04, 117.2, 117.3, 131.3, 132.7, 133.5, 143.6, 161.2. GC-MS (the second peak on GC among the isomers) m/z (% relative intensity, ion) 240 (70, M), 182 (39), 141 (40), 114 (48), 99 (100), 42 (93).



2-Cyano-1-(2-tetrahydrofuranyl)naphthalene (3dx). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.06 (dq, *J* = 12.4, 8.5 Hz, 1 H), 2.55 (dtd, *J* = 12.5, 7.6, 5.1 Hz, 1 H), 4.08 (td, *J* = 7.9, 6.3 Hz, 1 H), 4.47 (q, *J* = 6.8 Hz, 1 H), 5.82 (t, *J* = 8.1 Hz, 1 H), 7.57–7.66 (m, 3 H), 7.81 (d, *J* = 8.6 Hz, 1 H), 7.89 (d, *J* = 8.6 Hz, 1 H), 8.20 (d, *J* = 8.5 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 26.9, 34.7, 69.2, 78.4, 107.7, 119.2, 124.5, 127.5, 128.5, 128.6, 128.7, 129.1, 130.2, 135.1, 145.7. HRMS (ESI) Calcd for $C_{15}H_{13}NO$: $[M+Na]^+$, 246.0889. Found: m/z 246.0889.

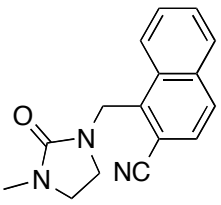


1-(1-Butoxybutyl)-2-cyanonaphthalene (3ex). A colorless oil. ^{1}H NMR (500 MHz, $CDCl_3$) δ 0.84 (t, *J* = 7.5 Hz, 3 H), 0.94 (t, *J* = 7.4 Hz, 3 H), 1.24–1.40 (m, 3 H), 1.50–1.68 (m, 3 H), 1.83–1.93 (m, 1 H), 2.17–2.27 (m, 1 H), 3.26 (q, *J* = 6.7 Hz, 1 H), 3.40 (dt, *J* = 9.1, 6.5 Hz, 1 H), 5.26 (dd, *J* = 8.1, 6.3 Hz, 1 H), 7.52–7.65 (m, 3 H), 7.81 (d, *J* = 8.6 Hz, 1 H), 8.88 (d, *J* = 8.8 Hz, 1 H), 8.84 (bs, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) d 13.9, 14.0, 19.5, 19.8, 32.1, 39.4, 69.7, 82.1, 109.7, 118.7, 126.7, 127.0, 127.1, 128.7, 129.0, 129.1, 130.9, 135.8, 145.9. HRMS (ESI) Calcd for $C_{19}H_{23}NO$: $[M+Na]^+$, 304.1672. Found: m/z 304.1669.

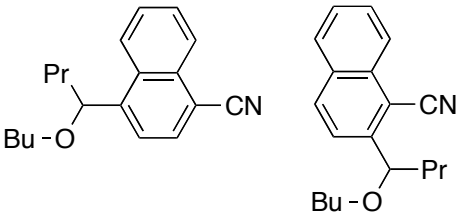


1,3-Dimethyl-4-(2-cyanonaphthyl)methyl-2-imidazolidinone (3fx). A white solid. Exists as two diastereomers (83/17), which could not be separated. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.65/2.72 (s, 3 H), 3.00/2.94 (s, 3 H), 3.60/3.40 (t, *J* = 9.3/9.2 Hz, 1 H), 3.87/3.94 (t, *J* = 9.7/9.5 Hz, 1 H), 5.54/5.53 (t, *J* = 9.7/8.6 Hz, 1 H), 7.55–7.75 (m, 3 H), 7.88–8.00 (m, 2 H) 8.41/8.18 (d, *J* = 8.7/9.1 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 30.0, 30.5, 31.2, 31.3, 50.9, 52.8, 54.5, 60.1, 108.6, 111.7, 117.7, 118.2, 122.5, 124.6,

126.8, 128.3, 128.5, 129.1, 129.2, 129.42, 129.44, 129.5, 129.7, 130.5, 130.8, 131.4, 135.0, 135.9, 139.9, 142.1, 160.6, 161.3. HRMS (ESI) Calcd for $C_{16}H_{15}N_3O$: $[M+Na]^+$, 288.1107. Found: m/z 288.1106.

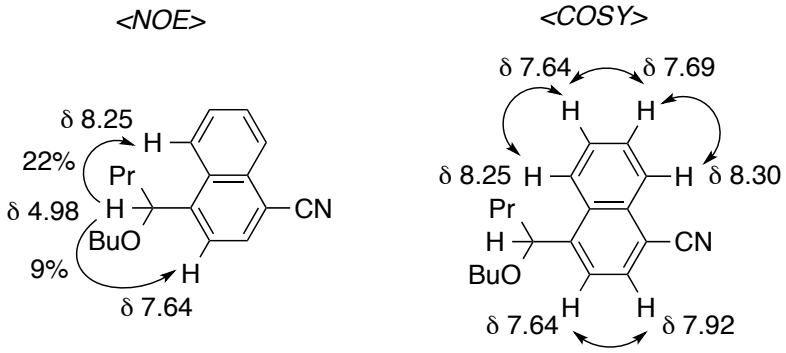


1-(2-Cyanonaphthyl)methyl-3-methyl-2-imidazolidinone (3'fx). A white solid. ^{1}H NMR (500 MHz, $CDCl_3$) δ 2.84 (s, 3 H), 3.20–3.28 (m, 4 H), 5.08 (s, 2 H), 7.59 (d, *J* = 8.6 Hz, 1 H), 7.65 (td, *J* = 7.0, 1.3 Hz, 1 H), 7.69 (td, *J* = 7.0, 1.5 Hz, 1 H), 7.89 (d, *J* = 7.1 Hz, 2 H), 8.54 (d, *J* = 8.4 Hz, 1 H). ^{13}C NMR (125 MHz, $CDCl_3$) δ 31.5, 42.6, 44.9, 45.0, 111.1, 118.7, 126.0, 126.3, 128.6, 128.7, 129.2, 129.8, 131.6, 135.3, 139.8, 160.8. HRMS (ESI) Calcd for $C_{16}H_{15}N_3O$: $[M+Na]^+$, 288.1107. Found: m/z 288.1101.



1-(1-Butoxybutyl)-4-cyanonaphthalene (3ey), 2-(1-Butoxybutyl)-1-cyanonaphthalene (3'ey). These products could not be isolated in pure forms. The following data were obtained from a spectrum of a mixture of **3ey** and **3'ey** (**3 ey**:**3'ey** = 84:16). ^{1}H NMR (500 MHz, $CDCl_3$) **3ey**: δ 4.98 (dd, *J* = 8.4, 3.1 Hz, 1 H), 7.64 (td *J* = 8.6, 1.4 Hz, 1 H), 7.64 (d, *J* = 8.0 Hz, 1 H), 7.69 (td, *J* = 7.0, 1.1 Hz, 1 H), 7.92 (d, *J* = 7.5 Hz, 1 H), 8.25 (d, *J* = 8.5 Hz, 1 H), 8.30 (d, *J* = 8.5 Hz, 1 H). **3'ey**: δ 4.90 (dd, *J* = 8.0, 5.4 Hz, 1 H), 7.60 (td, *J* = 6.9, 1.0 Hz, 1 H), 7.91 (d, *J* = 8.2 Hz, 1 H), 8.07 (d, *J* = 8.7 Hz, 1 H). The other peaks were could not be read because they are not sufficiently separated from those of another isomer. δ 0.85–1.00 (m, 6 H), 1.32–1.49 (m, 3 H), 1.51–1.65 (m, 3 H), 1.67–1.94 (m, 2 H), 3.26–3.40 (m, 2 H), 7.57–7.72 (m, 2 H, **3'ey**), 8.21–8.25 (m, 1H, **3'ey**). ^{13}C NMR (125 MHz, $CDCl_3$) δ 13.92, 13.94, 13.98, 13.99, 19.1, 19.53, 19.58, 19.59, 32.1, 32.3, 40.12, 40.17, 69.56, 69.61, 79.7, 80.2, 108.6, 109.9, 116.3, 123.2, 123.7, 124.3, 125.5, 126.3, 127.3, 128.2, 128.6, 128.7, 131.0, 132.4, 132.55, 132.63, 133.1, 133.3, 136.1, 148.7. HRMS (ESI) Calcd for $C_{19}H_{23}NO$: $[M+Na]^+$, 304.1672. Found: m/z 304.1668. GC-MS m/z (% relative intensity, ion) forward peak (**3'ey**): 281 (4, M), 238 (35, M–C_3H_7), 182 (100), 166 (19); back peak (**3ey**): 281 (6, M), 238 (62, M–C_3H_7), 182 (100), 166 (28).

Further Analysis of 3ey by NMR (NOE and ^{1}H–^{1}H COSY). Upon irradiation at a methyne proton (4.98 ppm), NOE was observed with protons at 7.64 and 8.25 ppm. In a ^{1}H–^{1}H COSY spectrum, the former (7.64 ppm) and the latter (8.25 ppm) belong to correlation groups consisting of two and four protons, respectively. These results shows that the 1-butoxybutyl group is introduced at 4-position in the major regioisomer (**3ey**).



In contrast, no valuable information on structure of the minor isomer (**3'ey**) was obtained from NOE and ^{1}H–^{1}H COSY due to overlaps of their peaks with those of the major isomer (**3ey**). Thus, its structure is unclear. However, comparing the chemical shifts of the peaks in ^{1}H NMR with those of 1-cyanonaphthalene, the 1-butoxybutyl group is likely to be introduced at 2-position in the minor regioisomer (**3'ey**).

References

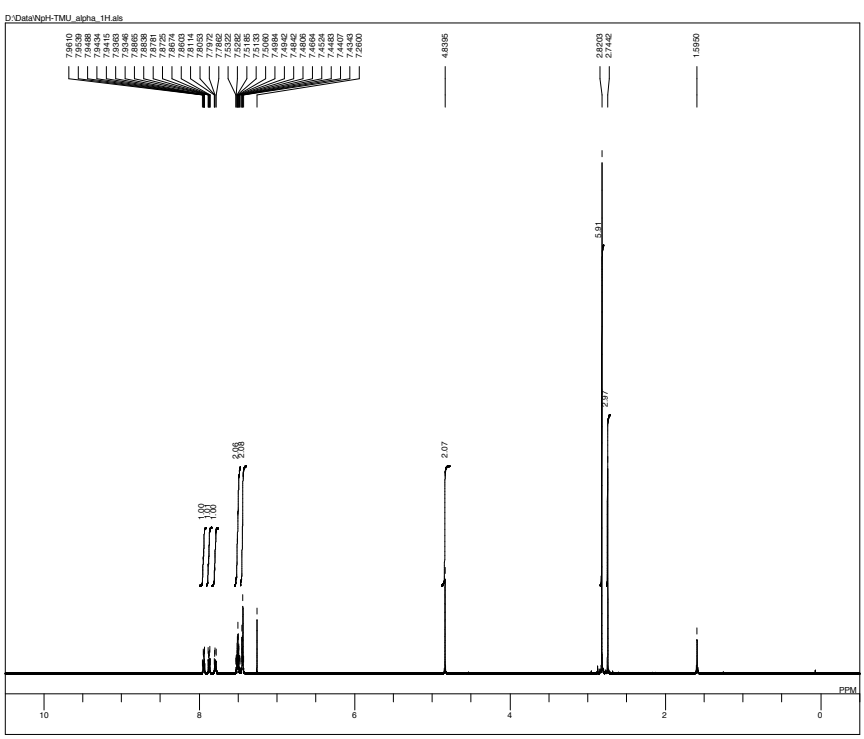
1 A.-P. Schaffner and P. Renaud, *Angew. Chem., Int. Ed.*, 2003, **42**, 2658.

2 H. Naarmann, M. Beaujean, R. Merényi and H. G. Viehe, *Polym. Bull.*, 1980, **2**, 417.

3 N.-M. Bi, M.-G. Ren and Q.-H. Song, *Synth. Commun.*, 2010, **40**, 2617.

4 D. Liu, C. Liu, H. Li and A. Lei, *Angew. Chem., Int. Ed.*, 2013, **52**, 4453.

NMR Spectra of the Products



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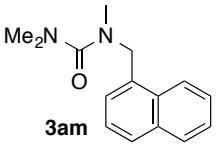
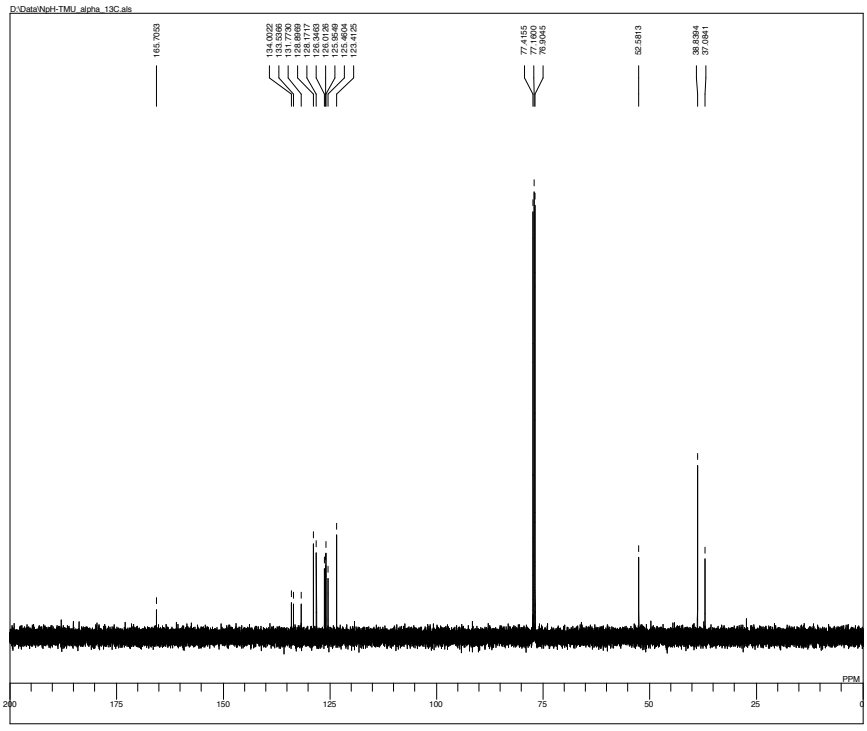


3am



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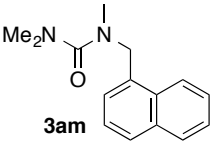

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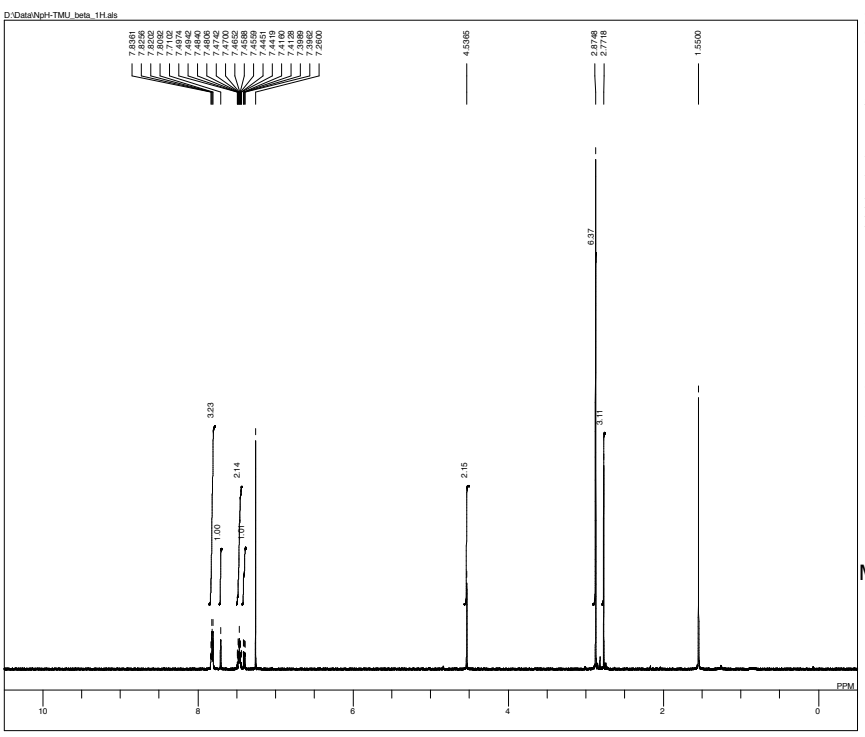


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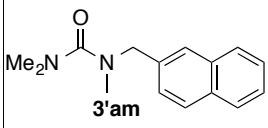

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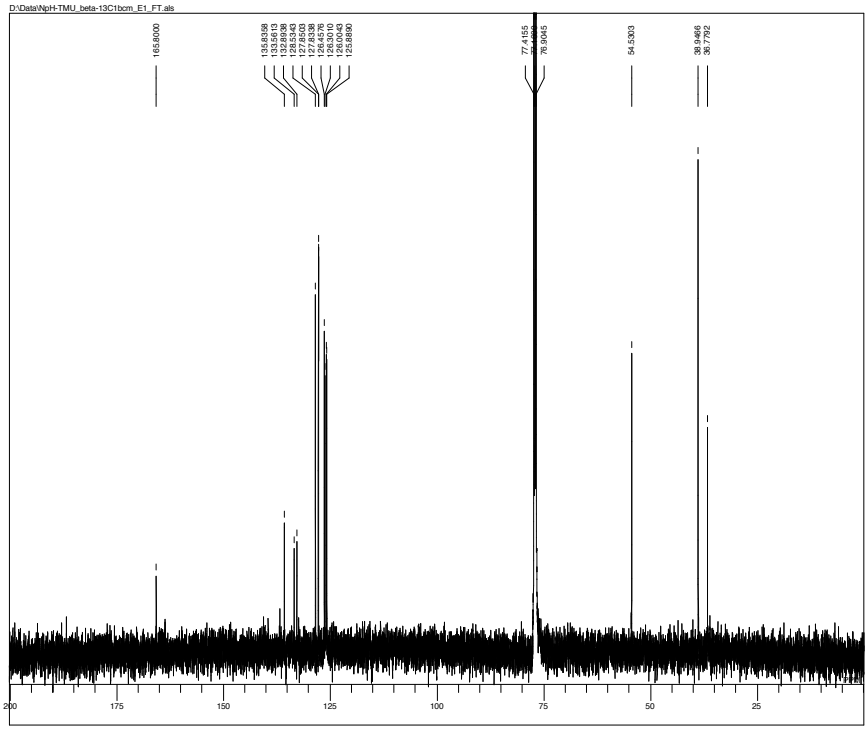


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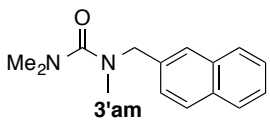

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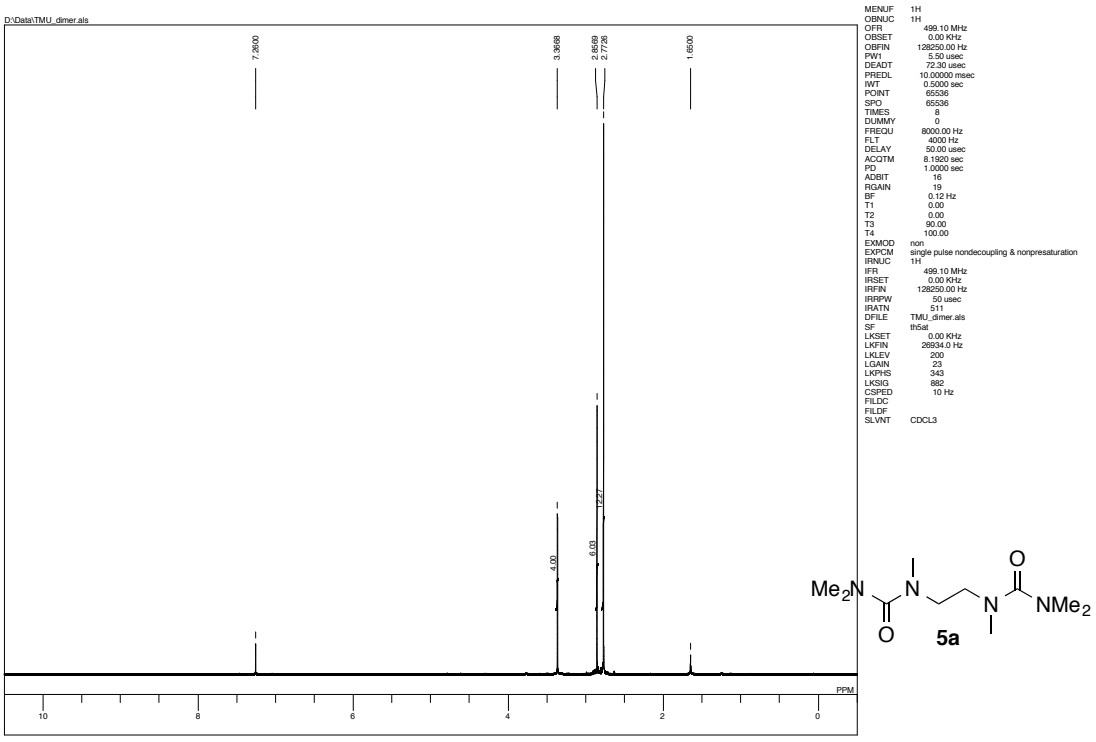
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4
2
0
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OBFIN 128250.00 Hz
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Me2N
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N
O
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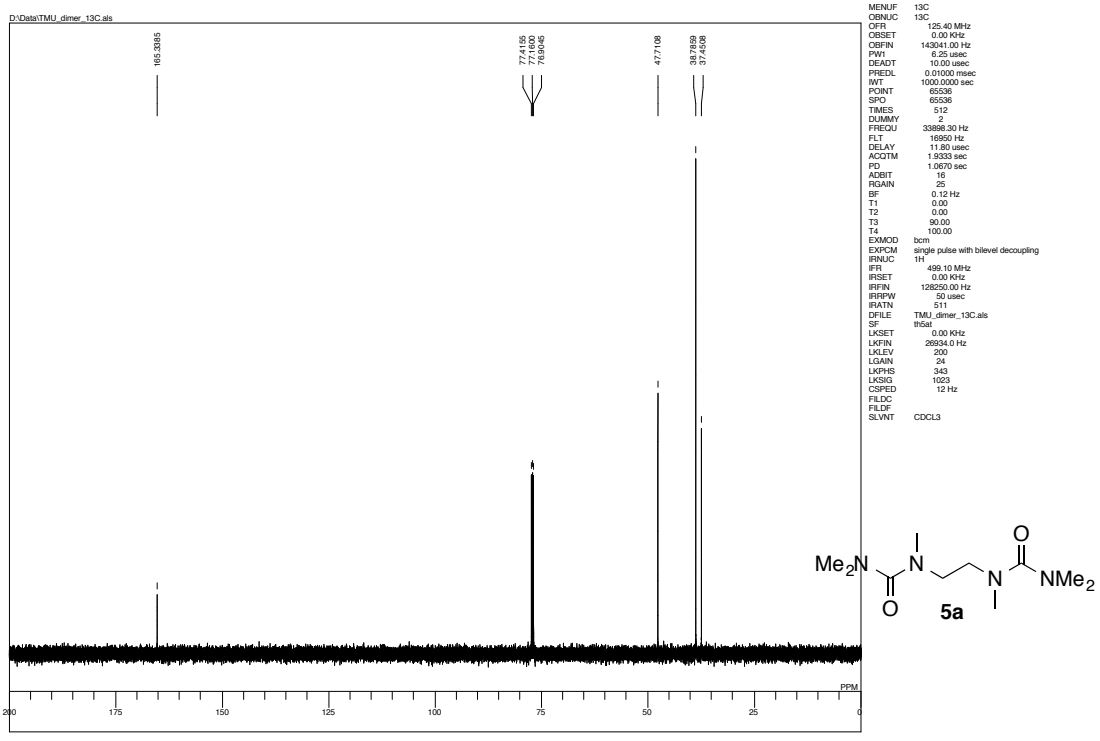
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100
75
50
25
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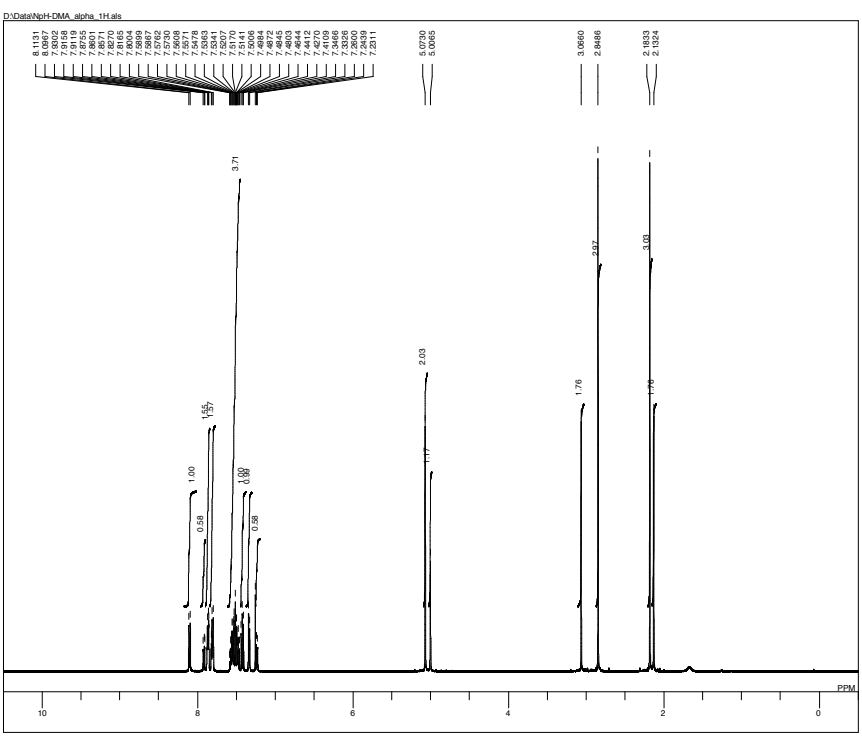

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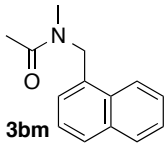

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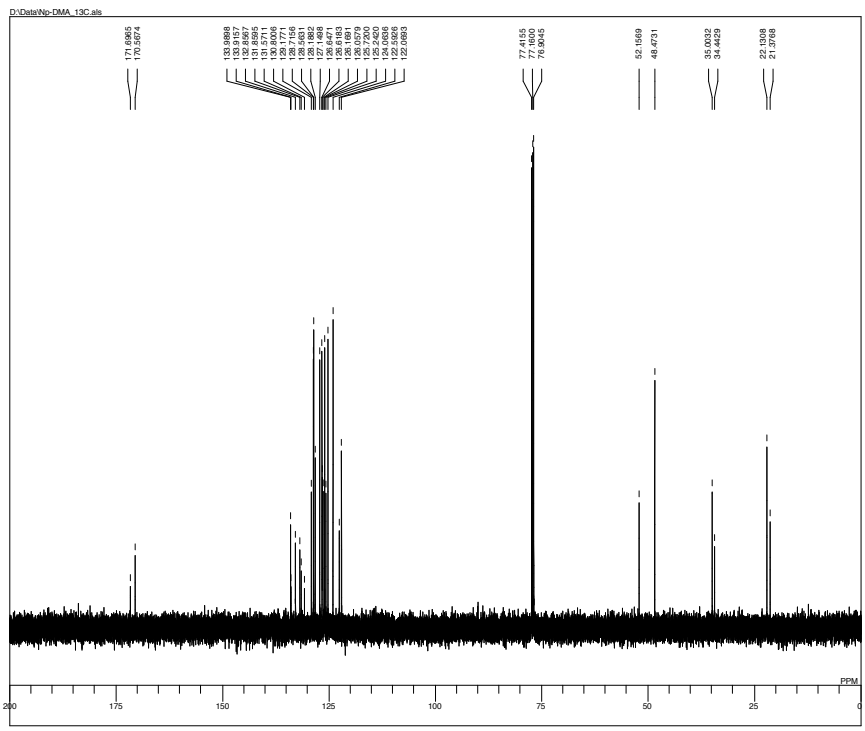

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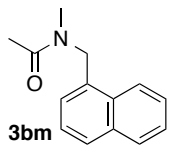

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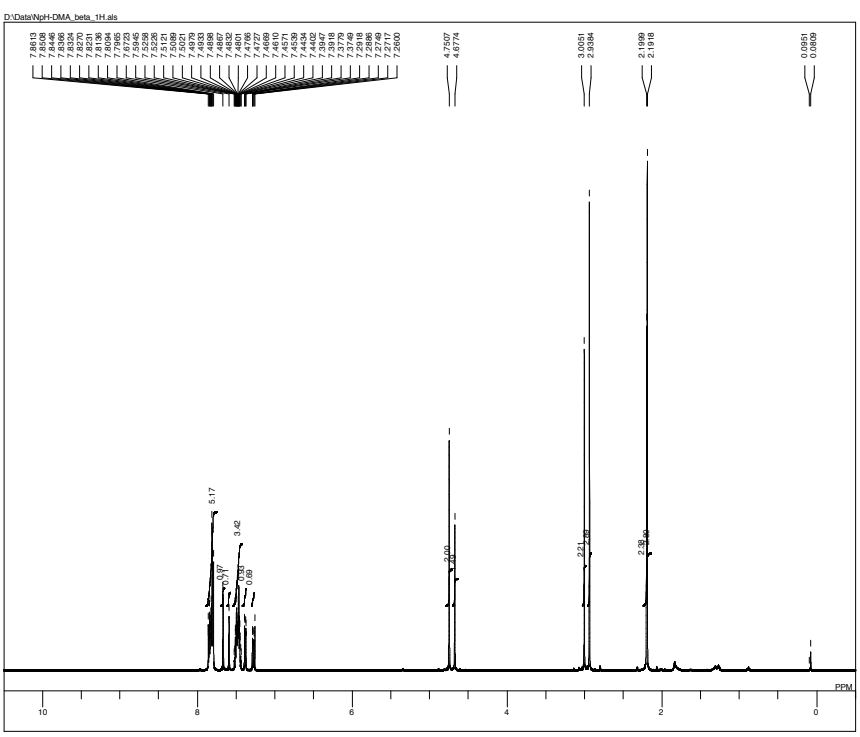


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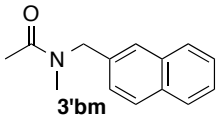

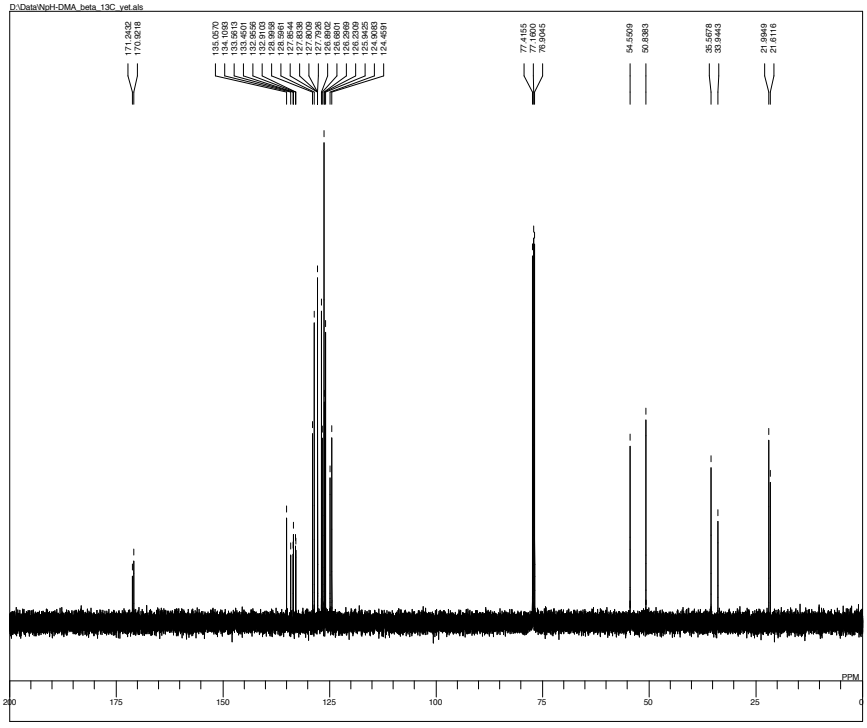


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T4          100.00
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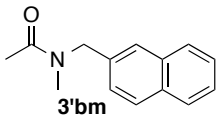

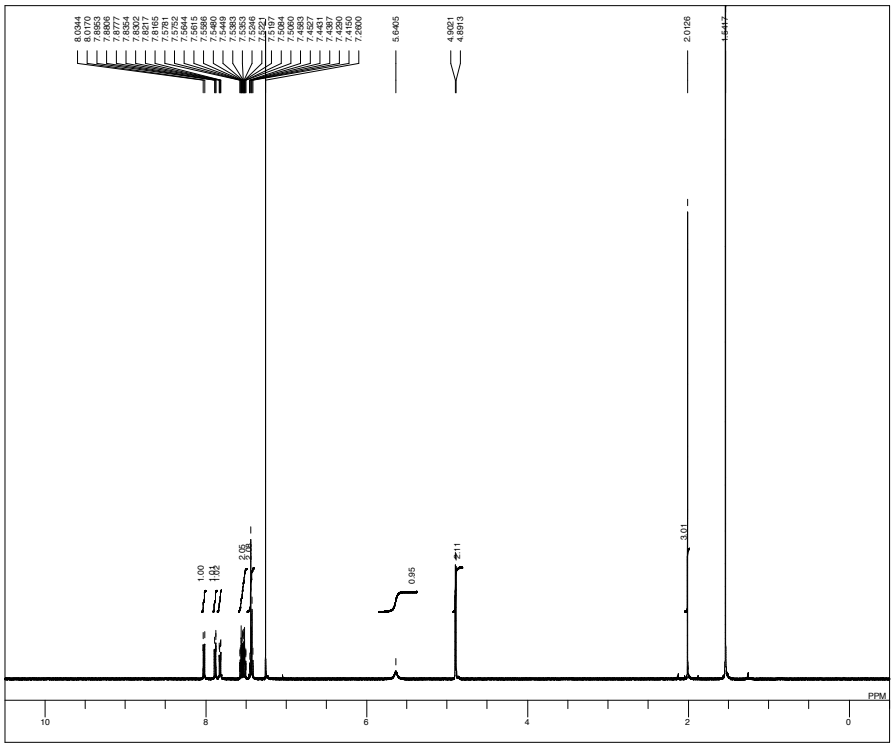


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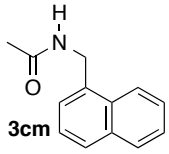
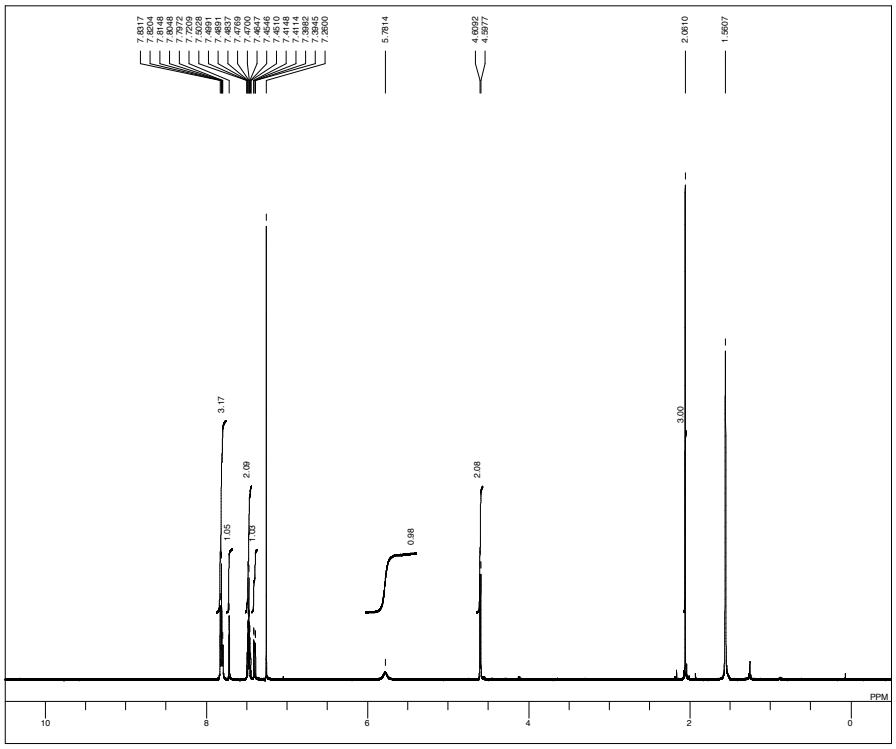
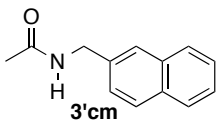
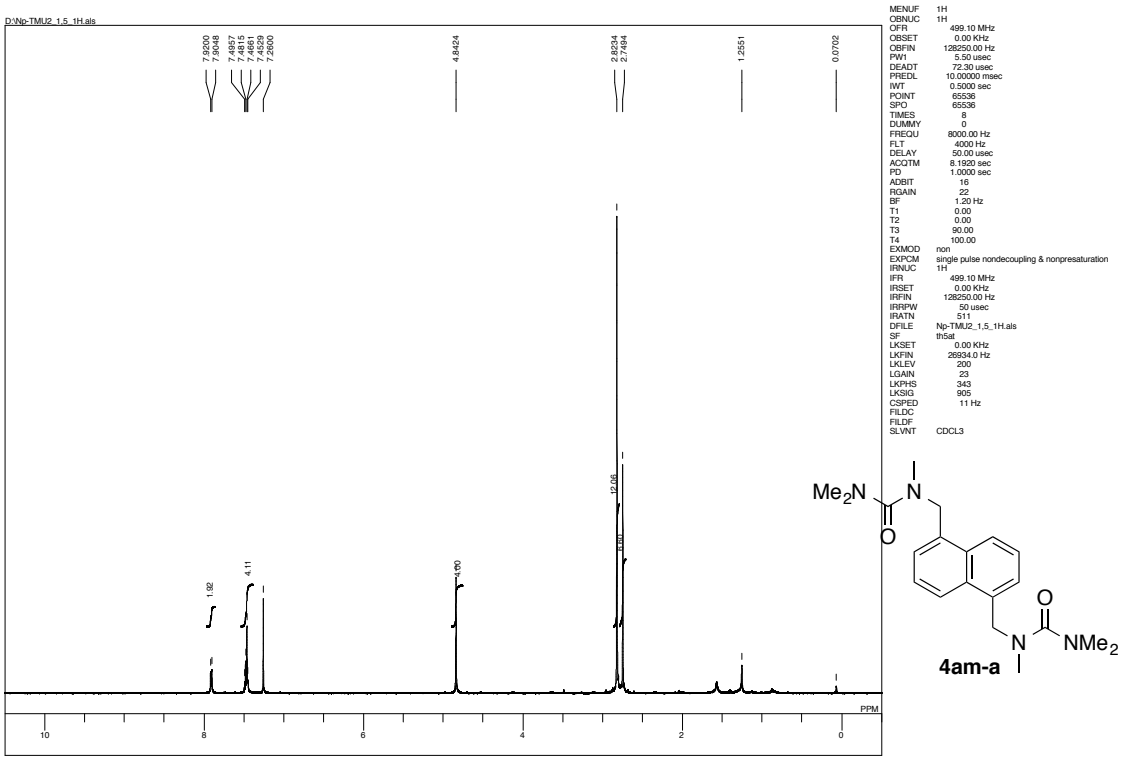
SLVNT      CDCL3
```

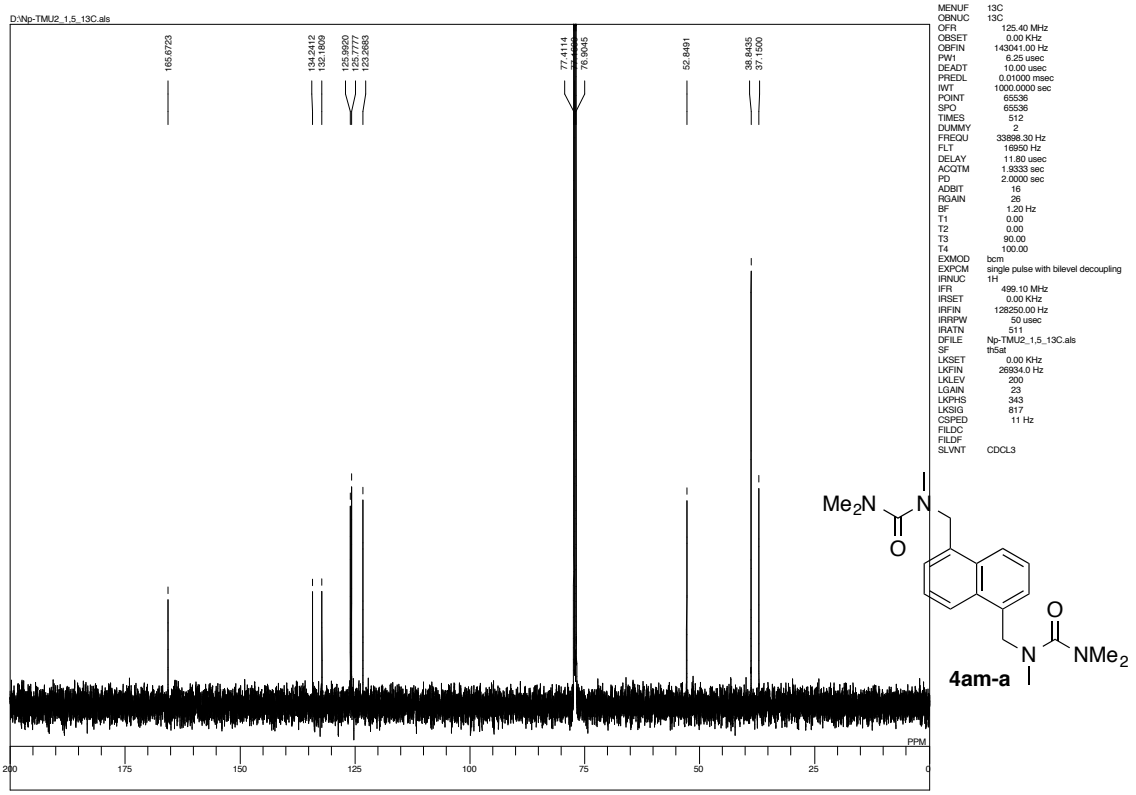


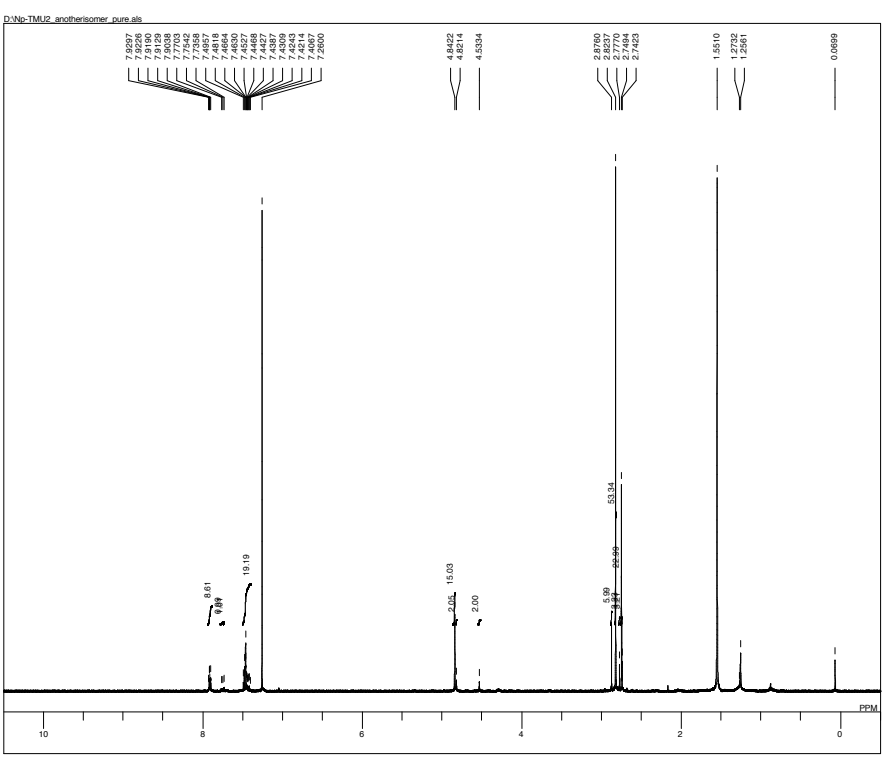



```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES           16
DUMMY            0
FREQU      8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM      8.1920 sec
PD           1.0000 sec
ADBIT           16
RGAIN           24
BF           0.12 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
EXMOD      non
EXPCM      single pulse nondecoupling & nonpresaturation
IRNUC      1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE      Np-NMA_beta_clean.als
SF         th5at
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          803
CSPED         14 Hz
FILDC
FILDF
SLVNT      CDCL3
```



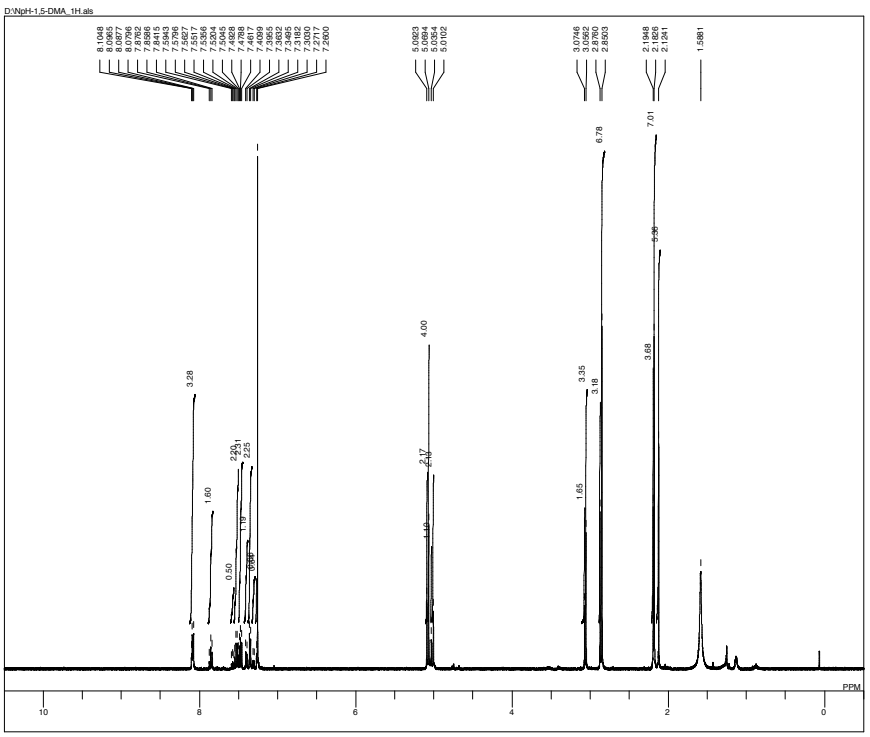

D:\Np-TMU2_1_5_1H.als
7.9200
7.9048
7.4857
7.4715
7.4681
7.4529
7.2600
4.8424
2.8234
2.7494
1.2551
0.0702
1.92
4.11
4.00
12.06
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 22
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Np-TMU2_1_5_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 905
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
Me2N
N
O
O
N
NMe2
4am-a


D:\Np-TMU2_1_5_13C.als
165.6723
134.2412
132.1809
125.9920
125.7777
123.2683
77.4114
77.0000
76.5945
52.8491
38.8435
37.1500
PPM
200
175
150
125
100
75
50
25
0
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 512
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 2.0000 sec
ADBIT 16
RGAIN 26
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Np-TMU2_1_5_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 817
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
Me2N
N
O
O
N
NMe2
4am-a

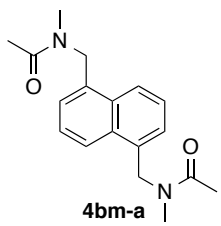



```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES           80
DUMMY            0
FREQU     8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM     8.1920 sec
PD           1.0000 sec
ADBIT           16
RGAIN           25
BF           0.12 Hz
T1              0.00
T2              0.00
T3             90.00
T4            100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW        50 usec
IRATN           511
DFILE     Np-TMU2_anotherisomer_pure.als
SF        th5at
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           947
CSPED          12 Hz
FILDC
FILDF
SLVNT     CDCL3
```

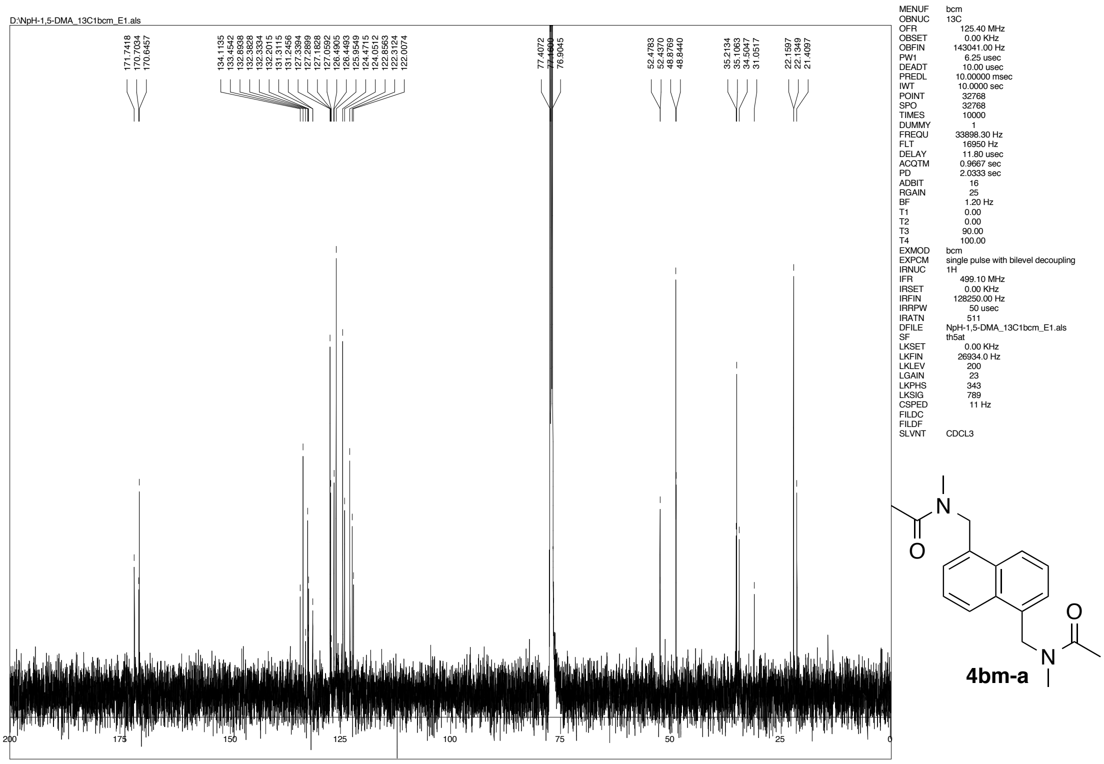
4am-b
a mixture with **4am-a**
(**4am-a**:**4am-b** = 79:21)



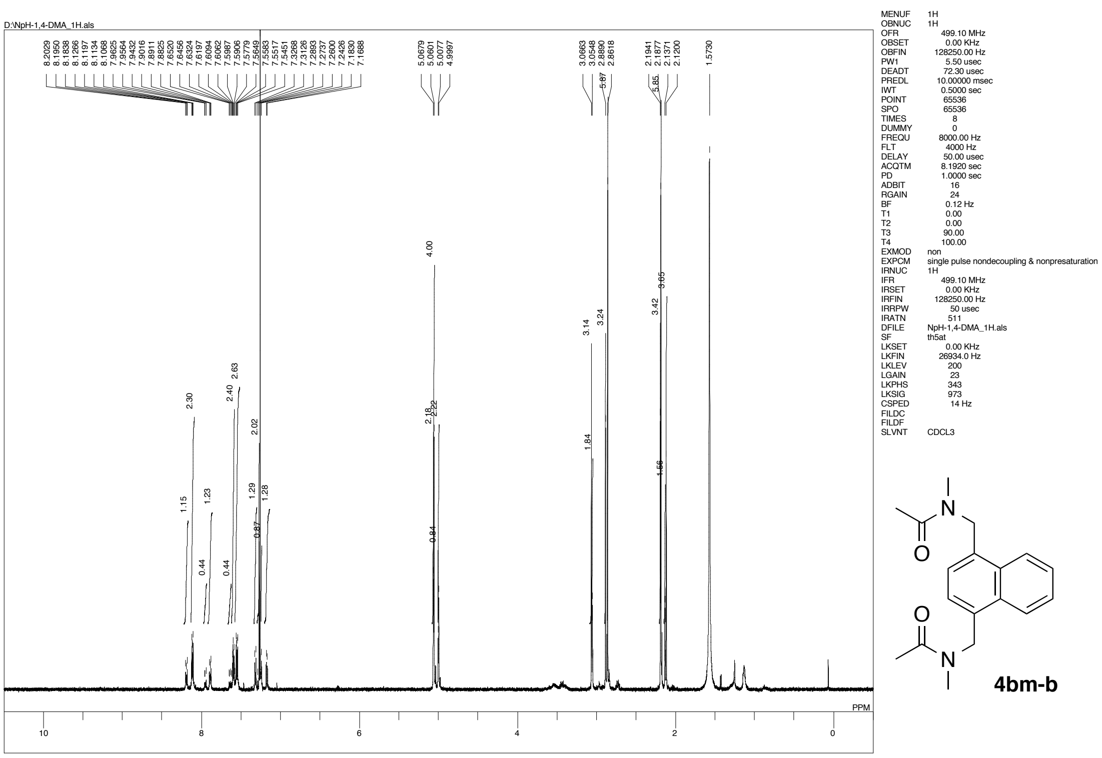

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU     8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM     8.1920 sec
PD           1.0000 sec
ADBIT           16
RGAIN           24
BF           0.12 Hz
T1              0.00
T2              0.00
T3             90.00
T4            100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW        50 usec
IRATN           511
DFILE     NpH-1,5-DMA_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           944
CSPED           5 Hz
FILDC
FILDF
SLVNT     CDCL3
```



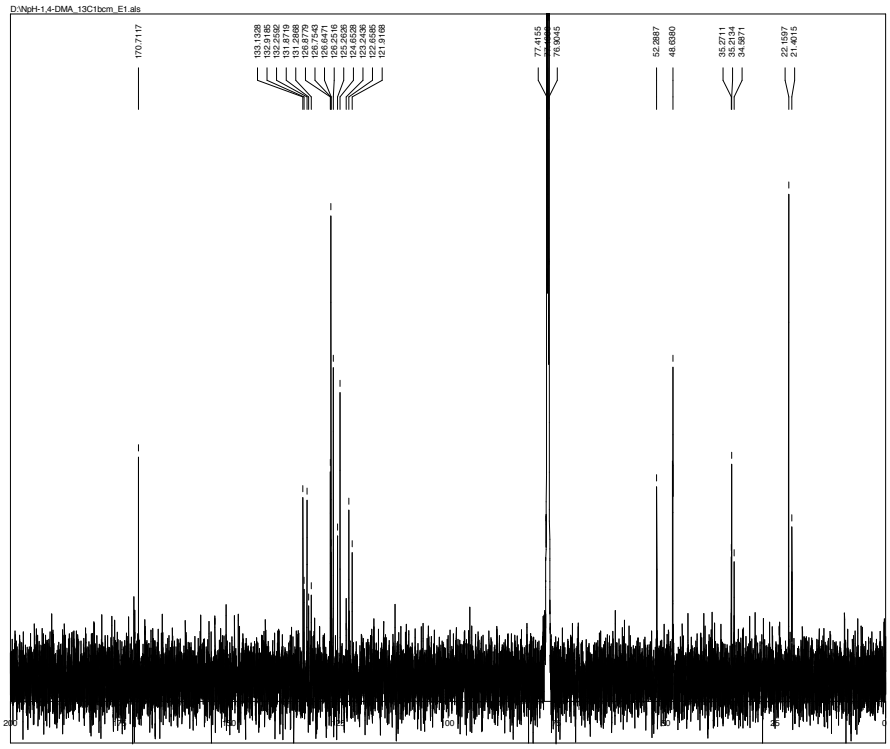
4bm-a


D:\NpH-1,5-DMA_13C1bcm_E1.als
171.7418
170.7034
170.6457
134.1135
133.4542
132.8938
132.3828
132.3334
132.2015
131.3115
131.2456
127.3394
127.2899
127.1828
127.0592
126.4905
126.4493
123.9549
124.4715
124.0512
122.8563
122.3124
122.0074
77.4072
77.1500
76.9045
52.4783
52.4370
48.8769
48.8440
35.2134
35.1063
34.5047
31.0517
22.1597
22.1349
21.4097
200
175
150
125
100
75
50
25
0
MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 10.00000 msec
IWT 10.0000 sec
POINT 32768
SPO 32768
TIMES 10000
DUMMY 1
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 2.0333 sec
ADBIT 16
RGAIN 25
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE NpH-1,5-DMA_13C1bcm_E1.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 789
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
N
O
N
O

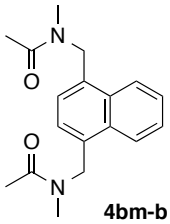
4bm-a


D:\NpH-1,4-DMA_1H.als
8.2029
8.1950
8.1838
8.1266
8.1197
8.1134
8.1068
7.9625
7.9564
7.9432
7.9016
7.8911
7.8825
7.6520
7.6456
7.6324
7.6197
7.6094
7.6062
7.5987
7.5906
7.5779
7.5649
7.5583
7.5517
7.5451
7.3268
7.3126
7.2893
7.2737
7.2600
7.2426
7.1830
7.1688
5.0679
5.0601
5.0077
4.9997
3.0663
3.0548
2.8890
2.8618
2.1941
2.1877
2.1371
2.1200
1.5730
1.15
0.44
2.30
1.23
0.44
2.40
2.63
1.29
0.87
2.02
1.28
2.18
0.84
2.22
4.00
3.14
1.84
3.24
5.87
3.42
1.56
5.85
3.65
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE NpH-1,4-DMA_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 973
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
N
O
O
N

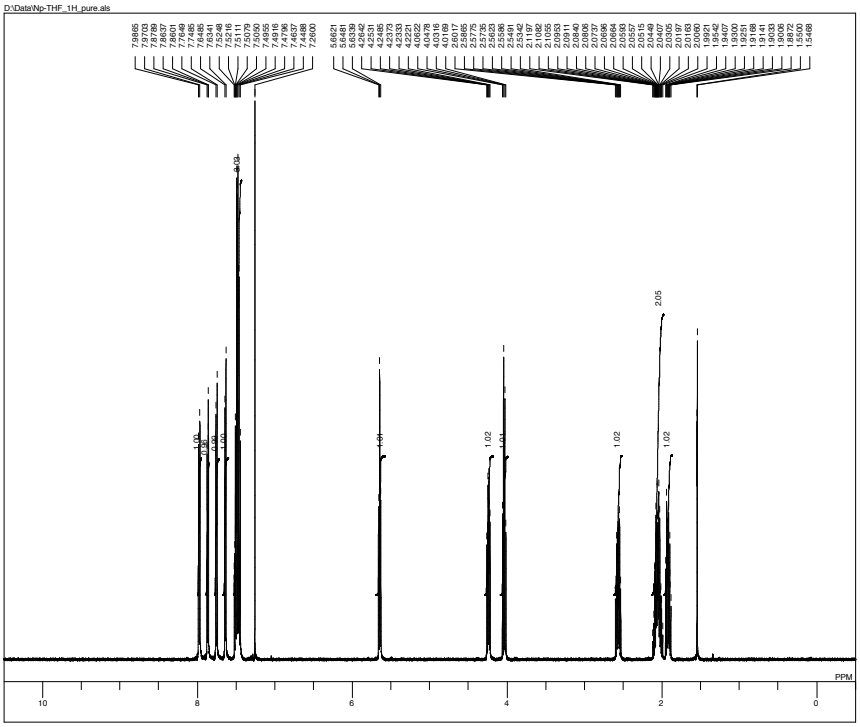
4bm-b



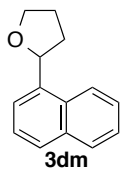
MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 10.00000 msec
IWT 10.0000 sec
POINT 32768
SPO 32768
TIMES 10000
DUMMY 1
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 2.0333 sec
ADBIT 16
RGAIN 27
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRPW 50 usec
IRATN 511
DFILE NpH-1,4-DMA_13C1bcm_E1.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 802
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3



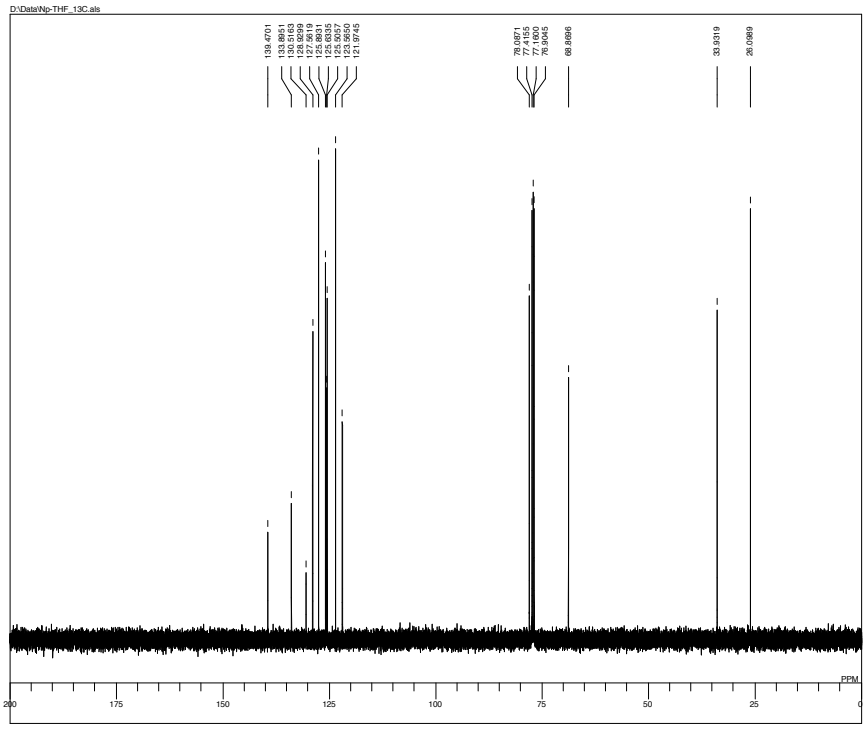
4bm-b



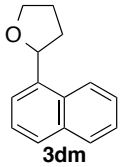
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 21
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRPW 50 usec
IRATN 511
DFILE Np-THF_1H_pure.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 981
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3



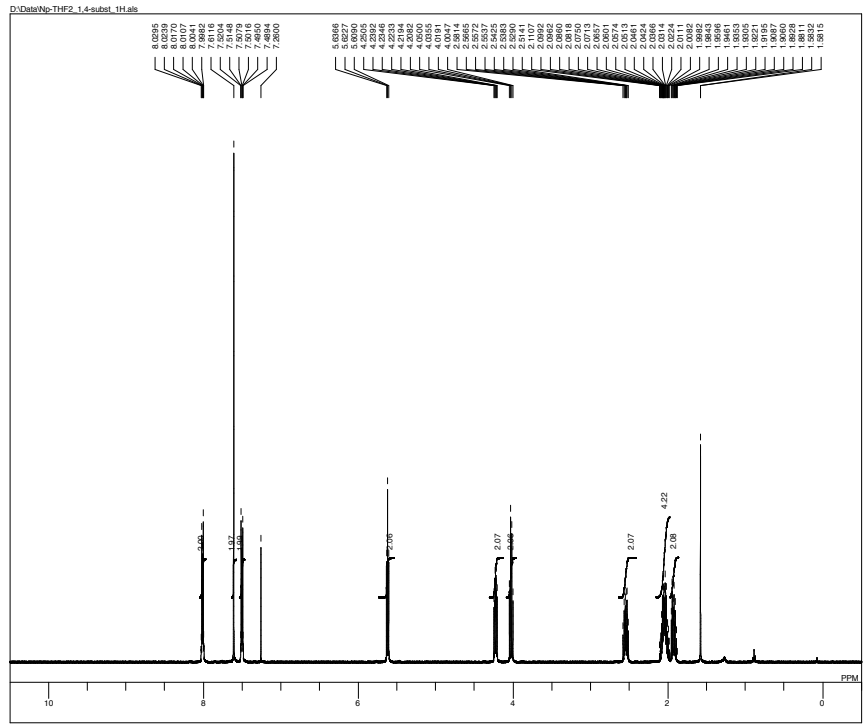
3dm



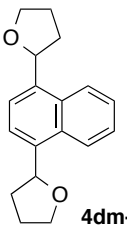

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL         0.01000 msec
IWT        1000.0000 sec
POINT        65536
SPO          65536
TIMES          256
DUMMY            2
FREQU        33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM         1.9333 sec
PD            1.0670 sec
ADBIT            16
RGAIN            24
BF            0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     Np-THF_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            994
CSPED          11 Hz
FILDC
FILDF
SLVNT     CDCL3
```



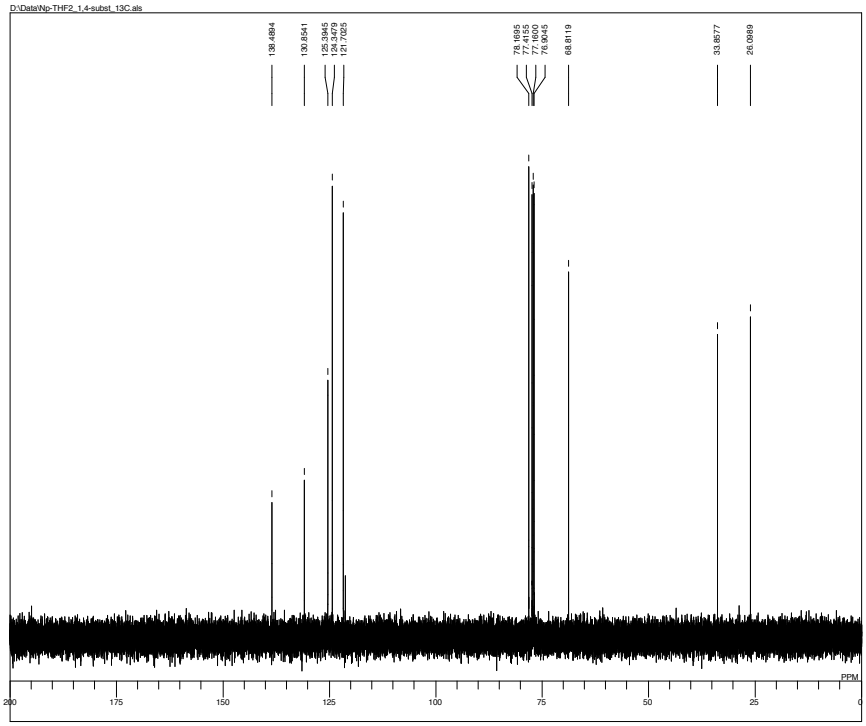
3dm



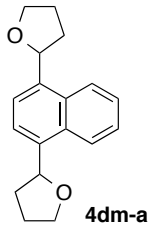

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL         10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU         8000.00 Hz
FLT           4000 Hz
DELAY         50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT            16
RGAIN            19
BF            0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     Np-THF2_1,4-subst_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             22
LKPHS            343
LKSIG            812
CSPED          10 Hz
FILDC
FILDF
SLVNT     CDCL3
```



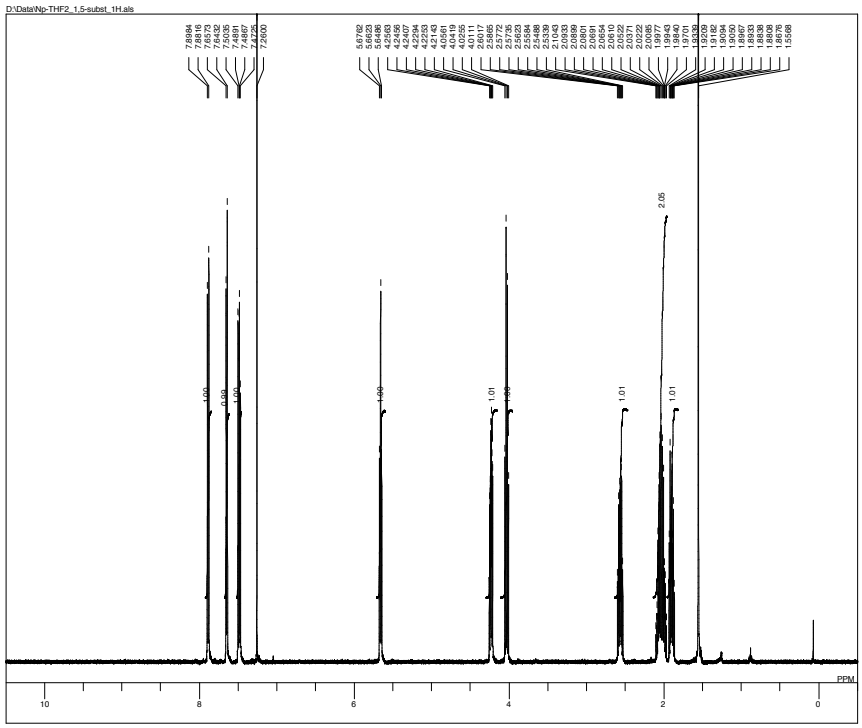
4dm-a



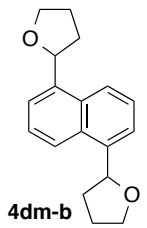

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL         0.01000 msec
IWT        1000.0000 sec
POINT        65536
SPO          65536
TIMES          256
DUMMY            2
FREQU     33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM         1.9333 sec
PD            1.0670 sec
ADBIT            16
RGAIN            27
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRPPW          50 usec
IRATN          511
DFILE     Np-THF2_1,4-subst_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          959
CSPED         14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



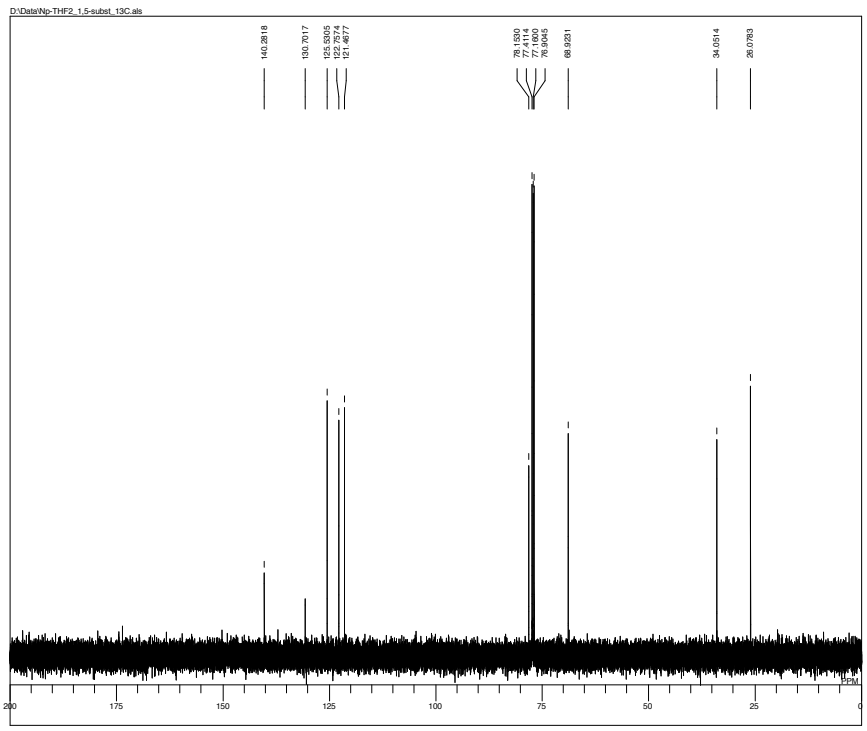
4dm-a



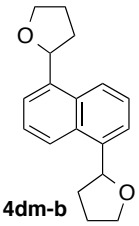

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL         10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU      8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT            16
RGAIN            22
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRPPW          50 usec
IRATN          511
DFILE     Np-THF2_1,5-subst_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           22
LKPHS          343
LKSIG          851
CSPED         10 Hz
FILDC
FILDF
SLVNT     CDCL3
```



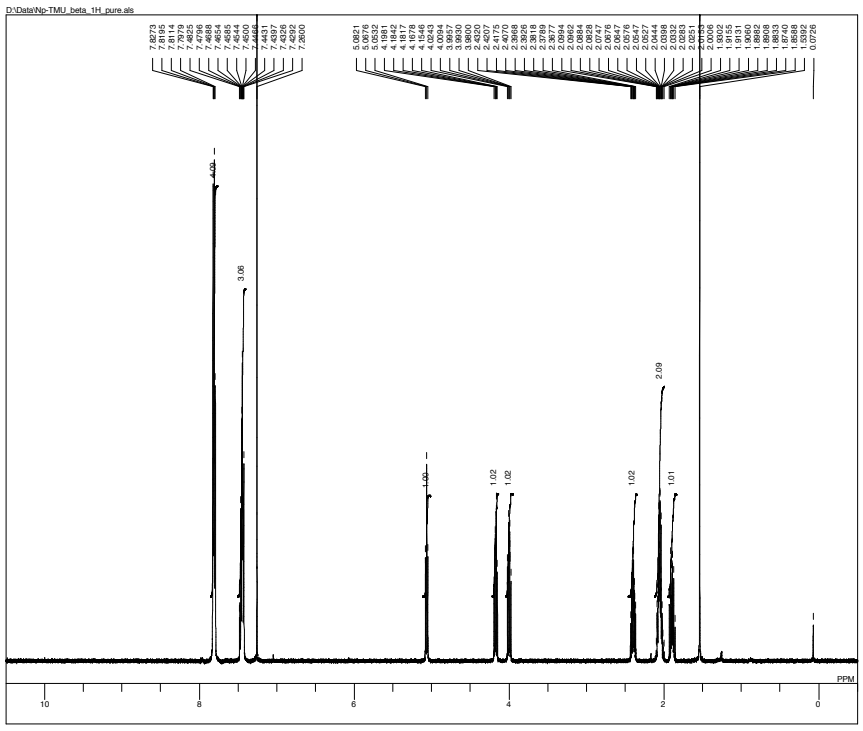
4dm-b



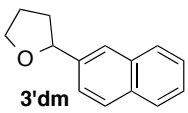

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL         0.01000 msec
IWT        1000.0000 sec
POINT        65536
SPO          65536
TIMES          256
DUMMY            2
FREQU     33898.30 Hz
FLT         16950 Hz
DELAY        11.80 usec
ACQTM         1.9333 sec
PD            1.0670 sec
AOBIT           16
RGAIN           27
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN          511
DFILE     Np-THF2_1,5-subst_13C.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV         200
LGAIN          23
LKPHS         343
LKSIG         924
CSPED          14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



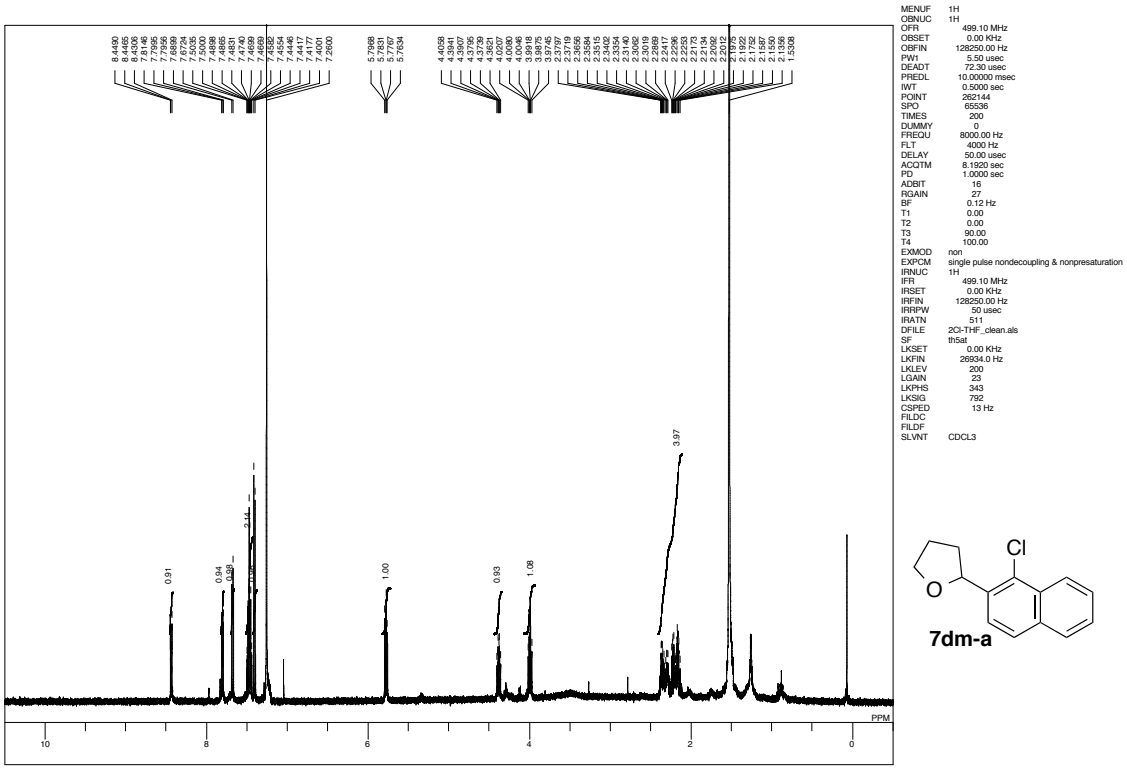
4dm-b




```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL         10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU      8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
AOBIT           16
RGAIN           23
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN          511
DFILE     Np-TMU_beta_1H_pure.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV         200
LGAIN          23
LKPHS         343
LKSIG         901
CSPED          14 Hz
FILDC
FILDF
SLVNT     CDCL3
```

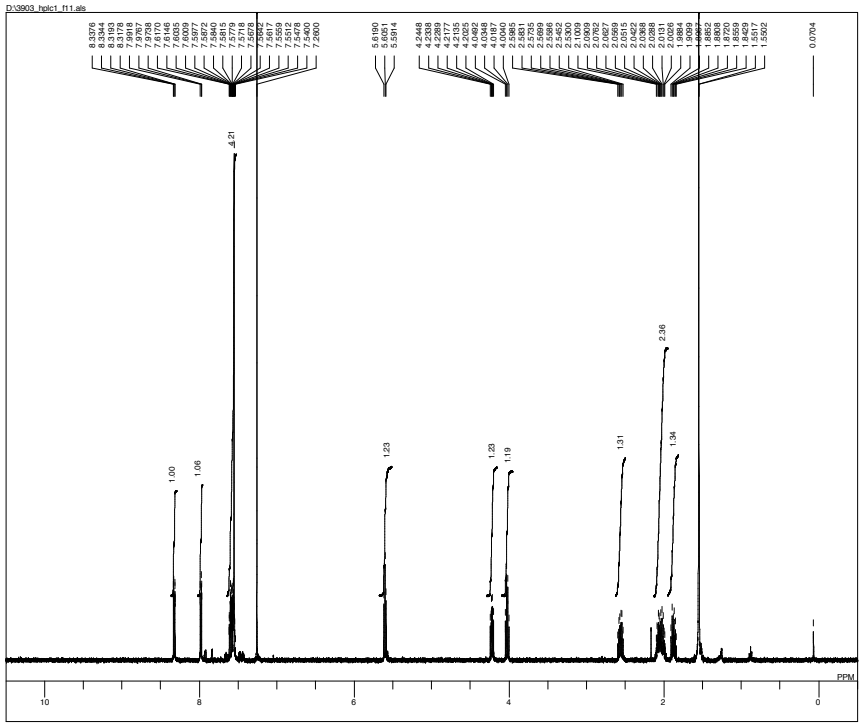


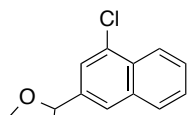
3'dm


MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 262144
SPO 65536
TIMES 200
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 27
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 2Cl-THF_clean.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 792
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
PPM

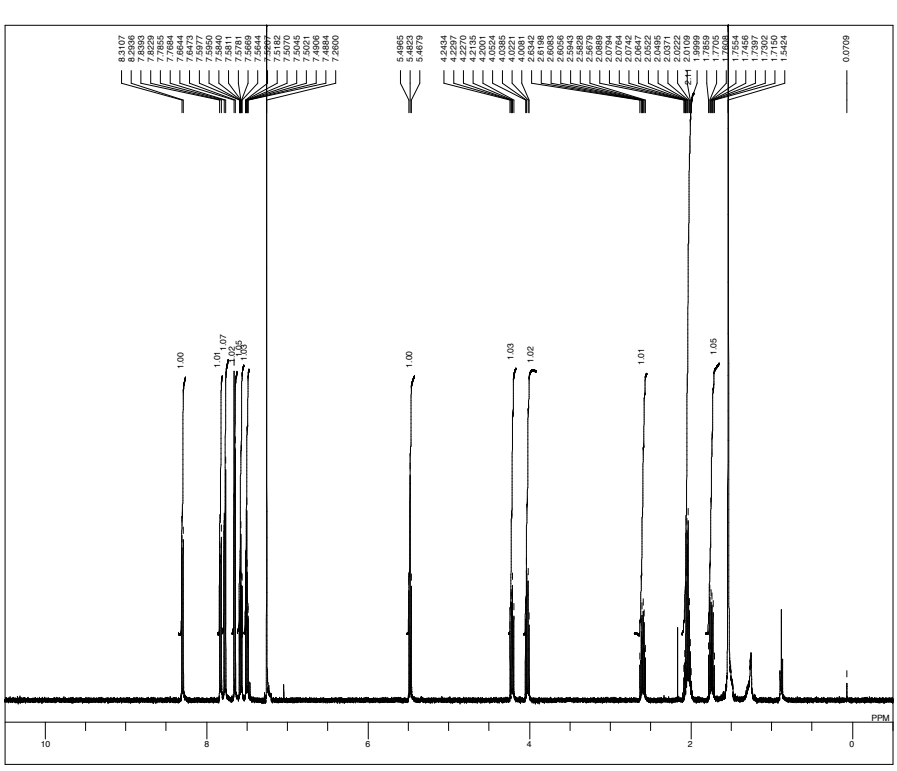
Cl
O

7dm-a

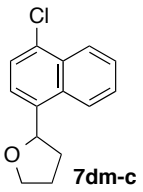

D:\3903_hplc1_f11.als
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE 3903_hplc1_f11.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 1000
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
PPM


Cl
O

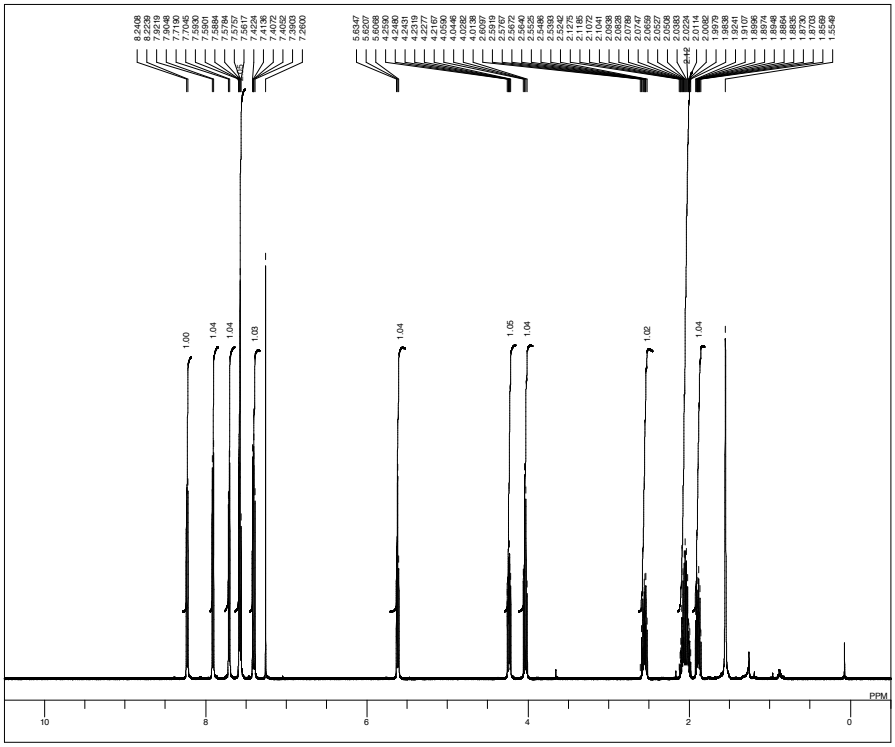
7dm-b



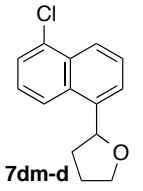
```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN      128250.00 Hz
PW1            5.50 usec
DEADT         72.30 usec
PREDL      10.00000 msec
IWT          0.5000 sec
POINT         65536
SPO           65536
TIMES            32
DUMMY             0
FREQU       8000.00 Hz
FLT           4000 Hz
DELAY        50.00 usec
ACQTM      8.1920 sec
PD           1.0000 sec
ADBIT            16
RGAIN            25
BF            0.12 Hz
T1              0.00
T2              0.00
T3             90.00
T4            100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRRPW         50 usec
IRATN            511
DFILE     4Cl-THF_clean.als
SF        th5at
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           895
CSPED         13 Hz
FILDC
FILDF
SLVNT     CDCL3
```



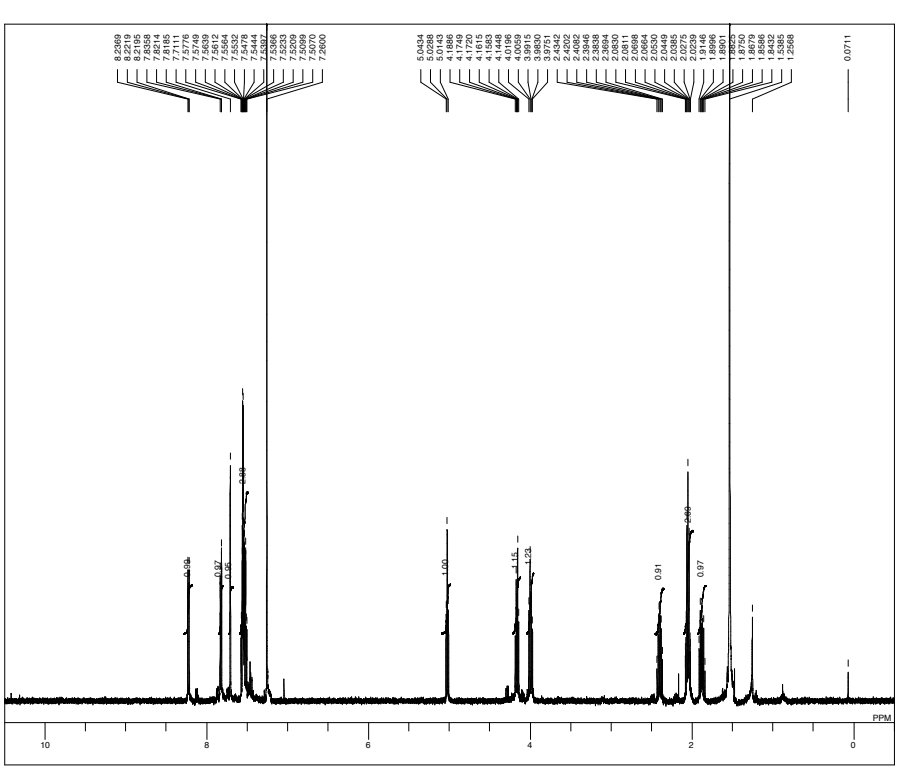
7dm-c



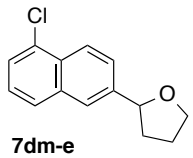
```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN      128250.00 Hz
PW1            5.50 usec
DEADT         72.30 usec
PREDL      10.00000 msec
IWT          0.5000 sec
POINT         65536
SPO           65536
TIMES            80
DUMMY             0
FREQU       8000.00 Hz
FLT           4000 Hz
DELAY        50.00 usec
ACQTM      8.1920 sec
PD           1.0000 sec
ADBIT            16
RGAIN            22
BF            0.12 Hz
T1              0.00
T2              0.00
T3             90.00
T4            100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRRPW         50 usec
IRATN            511
DFILE     5Cl-THF_clean.als
SF        th5at
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV           200
LGAIN            24
LKPHS           343
LKSIG          1034
CSPED         13 Hz
FILDC
FILDF
SLVNT     CDCL3
```



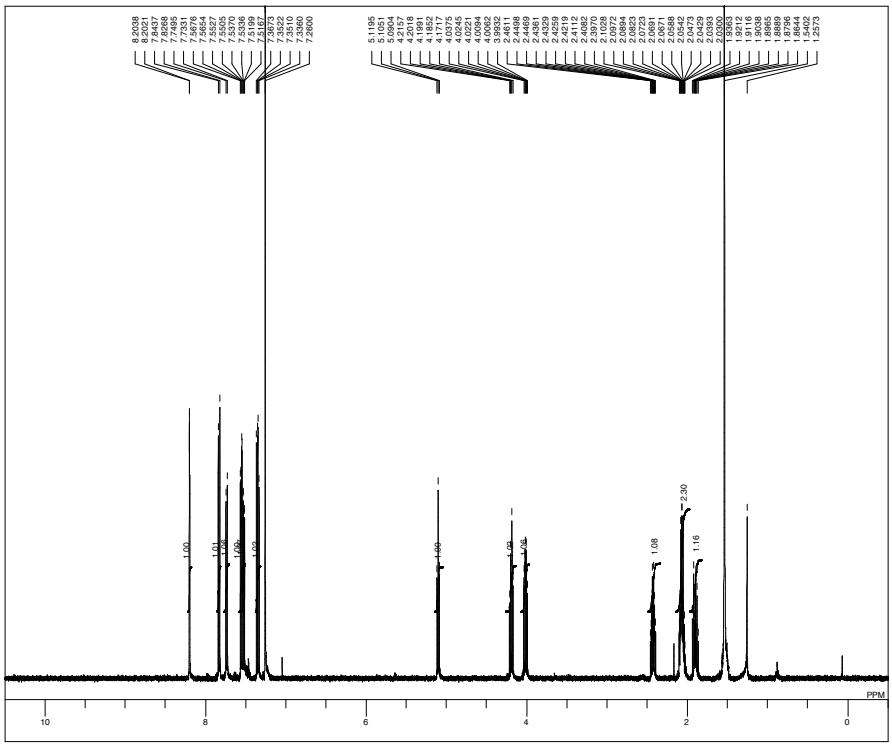
7dm-d



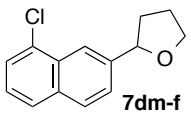
```
MENUF     1H
OBNUC     1H
OFR       499.10 MHz
OBSET     0.00 KHz
OBFIN     128250.00 Hz
PW1       5.50 usec
DEADT     72.30 usec
PREDL     10.00000 msec
IWT       0.5000 sec
POINT     65536
SPO       65536
TIMES     32
DUMMY     0
FREQU     8000.00 Hz
FLT       4000 Hz
DELAY     50.00 usec
ACQTM     8.1920 sec
PD        1.0000 sec
ADBIT     16
RGAIN     26
BF        0.12 Hz
T1        0.00
T2        0.00
T3        90.00
T4        100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR       499.10 MHz
IRSET     0.00 KHz
IRFIN     128250.00 Hz
IRPW      50 usec
IRATN     511
DFILE     6Cl-THF_clean.als
SF        thSat
LKSET     0.00 KHz
LKFIN     26934.0 Hz
LKLEV     200
LGAIN     23
LKPHS     343
LKSIG     836
CSPED     14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



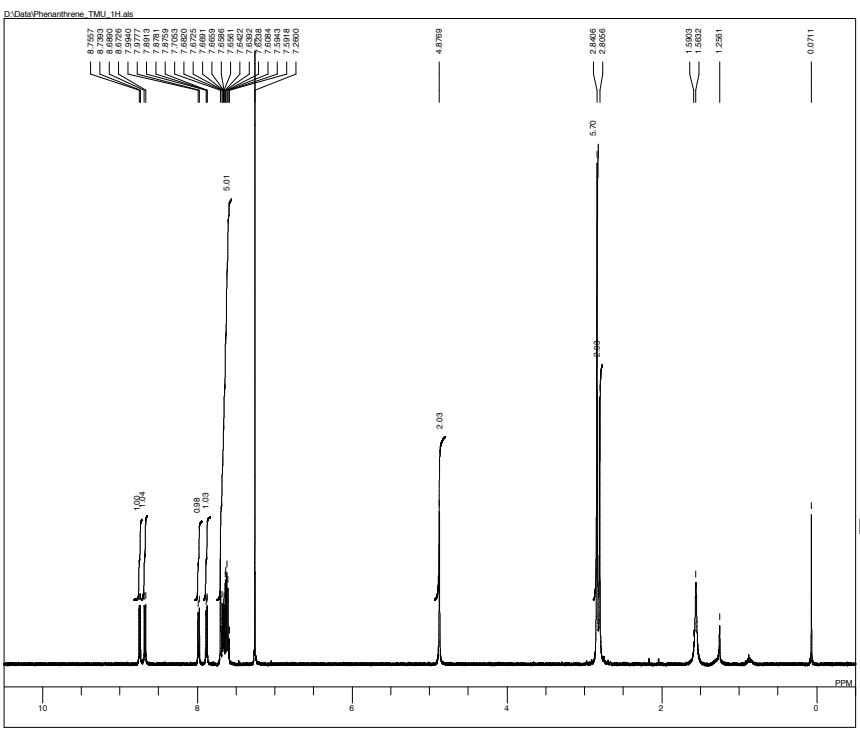
7dm-e



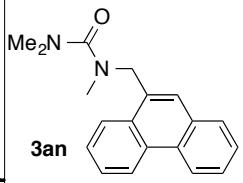
```
MENUF     1H
OBNUC     1H
OFR       499.10 MHz
OBSET     0.00 KHz
OBFIN     128250.00 Hz
PW1       5.50 usec
DEADT     72.30 usec
PREDL     10.00000 msec
IWT       0.5000 sec
POINT     65536
SPO       65536
TIMES     32
DUMMY     0
FREQU     8000.00 Hz
FLT       4000 Hz
DELAY     50.00 usec
ACQTM     8.1920 sec
PD        1.0000 sec
ADBIT     16
RGAIN     26
BF        0.12 Hz
T1        0.00
T2        0.00
T3        90.00
T4        100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR       499.10 MHz
IRSET     0.00 KHz
IRFIN     128250.00 Hz
IRPW      50 usec
IRATN     511
DFILE     7Cl-THF_clean_maybe.als
SF        thSat
LKSET     0.00 KHz
LKFIN     26934.0 Hz
LKLEV     200
LGAIN     23
LKPHS     343
LKSIG     878
CSPED     14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



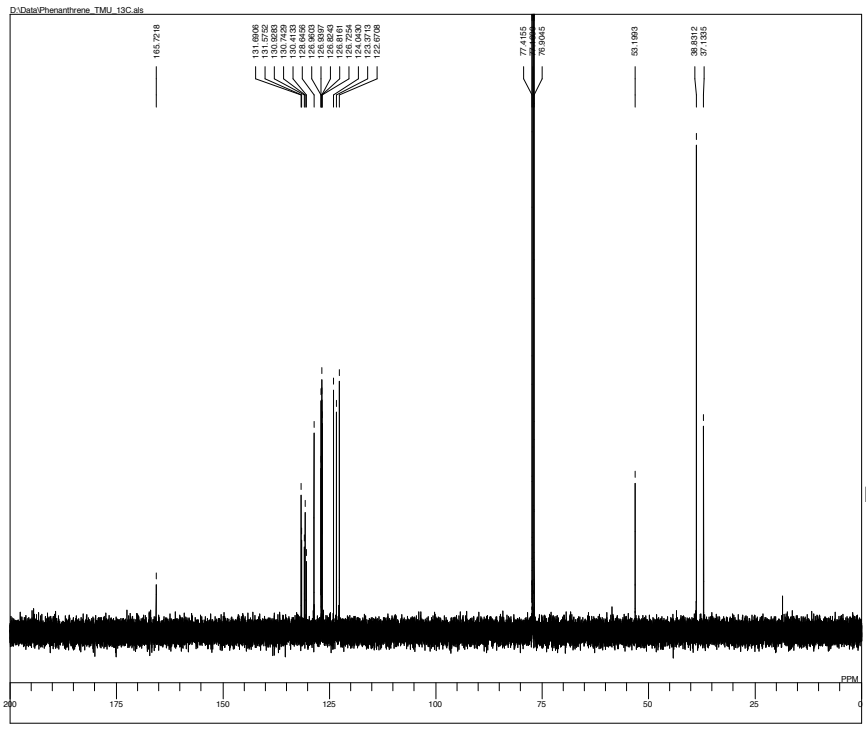
7dm-f



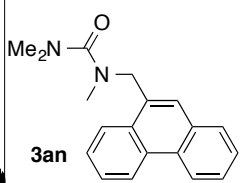

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN      128250.00 Hz
PW1            5.50 usec
DEADT         72.30 usec
PREDL      10.00000 msec
IWT            0.5000 sec
POINT         65536
SPO           65536
TIMES            16
DUMMY             0
FREQU          8000.00 Hz
FLT            4000 Hz
DELAY         50.00 usec
ACQTM          8.1920 sec
PD             1.0000 sec
ADBIT            16
RGAIN            24
BF             0.12 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     Phenanthrene_TMU_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           24
LKPHS          343
LKSIG          980
CSPED           7 Hz
FILDC
FILDF
SLVNT     CDCL3
```



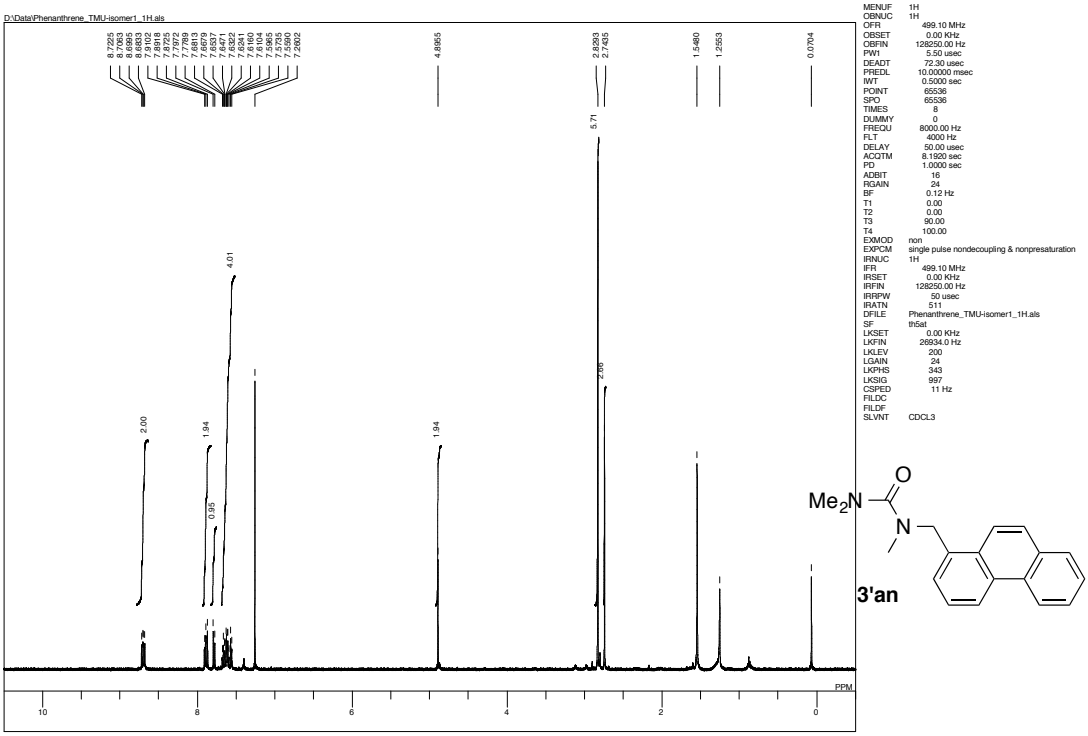
3an

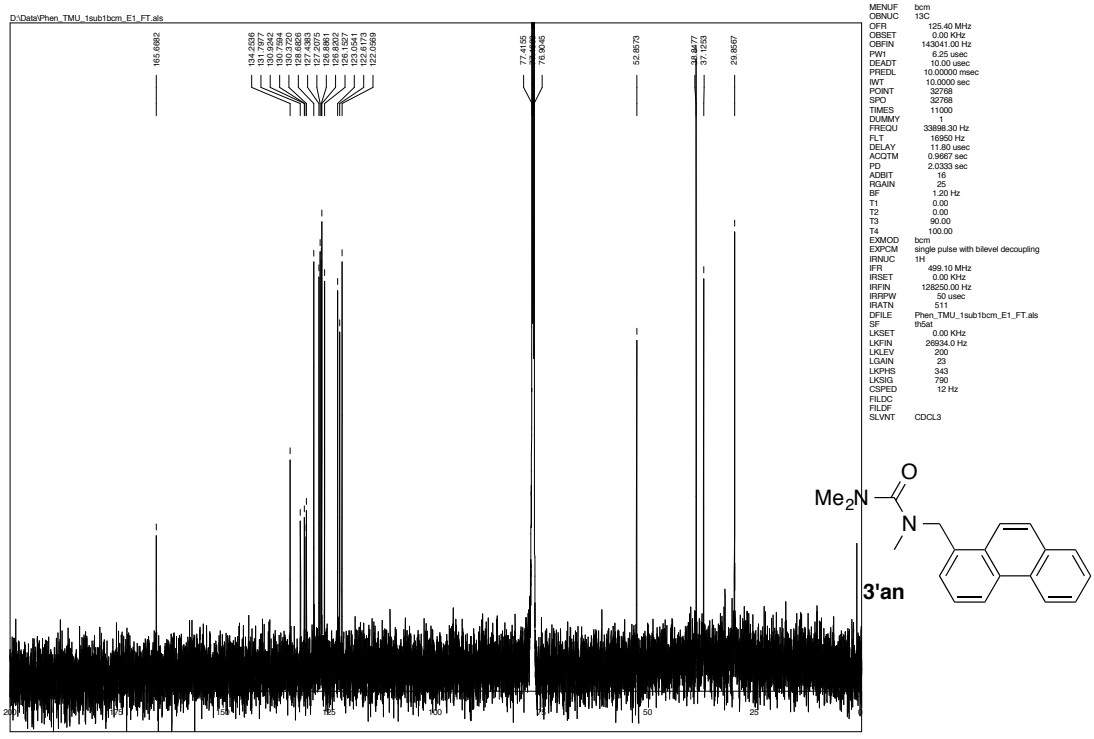


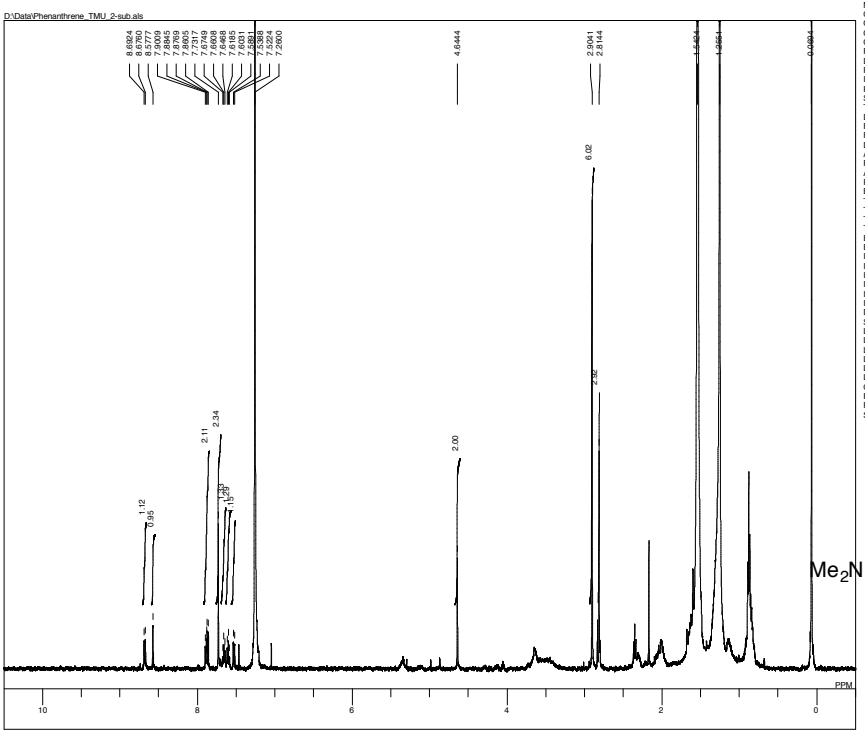

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN      143041.00 Hz
PW1            6.25 usec
DEADT         10.00 usec
PREDL      0.01000 msec
IWT        1000.0000 sec
POINT         65536
SPO           65536
TIMES          1000
DUMMY             2
FREQU       33898.30 Hz
FLT           16950 Hz
DELAY         11.80 usec
ACQTM          1.9333 sec
PD             1.0670 sec
ADBIT            16
RGAIN            25
BF             0.12 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     Phenanthrene_TMU_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          867
CSPED          11 Hz
FILDC
FILDF
SLVNT     CDCL3
```



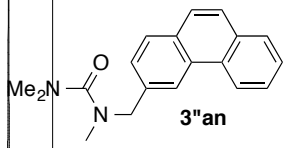
3an


D:\Data\Phenanthrene_TMU-isomer1_1H.als
8.7225
8.7063
8.6965
8.6833
7.9102
7.8914
7.8725
7.7972
7.7789
7.6813
7.6679
7.6597
7.6471
7.6322
7.6161
7.6160
7.6104
7.5995
7.5755
7.5590
7.2602
4.8965
2.8393
2.7435
1.5480
1.2553
0.0704
5.71
4.01
2.00
1.94
0.95
1.94
2.66
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.90 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Phenanthrene_TMU-isomer1_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 24
LKPHS 343
LKSIG 997
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
Me2N
O
N
3'an

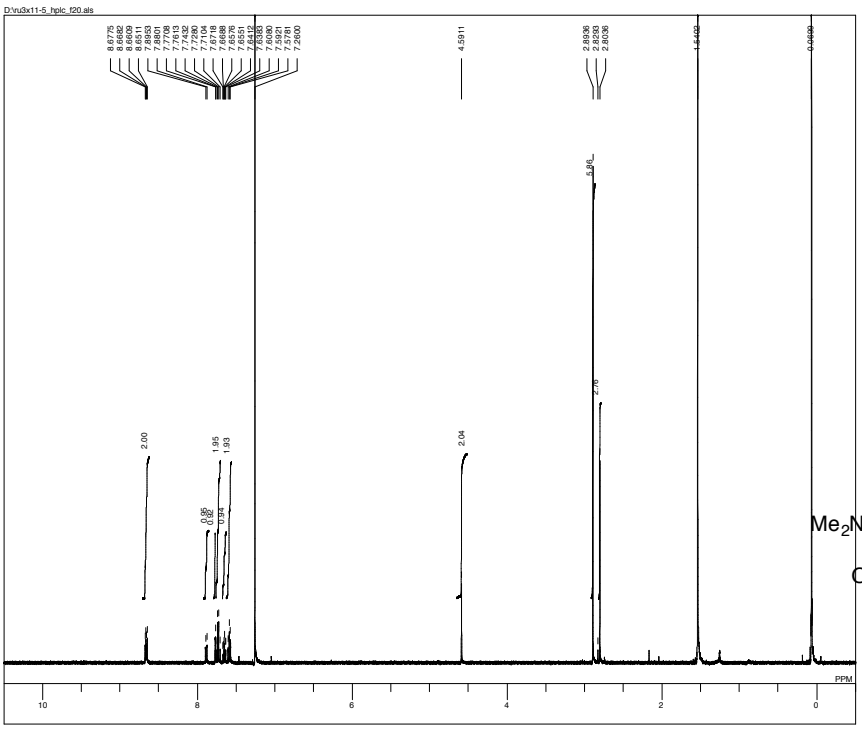

D:\Data\Phen_TMU_1sub1bcm_E1_FT.als
165.6682
134.2939
131.7077
130.9342
130.7994
130.5620
128.8826
127.4383
127.2035
126.8861
126.8202
126.6147
123.0541
122.8173
122.0569
77.4155
76.9045
52.8573
38.9477
37.1253
29.8567
MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 10.00000 msec
IWT 10.0000 sec
POINT 32768
SPO 32768
TIMES 11000
DUMMY 1
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 2.0333 sec
ADBIT 16
RGAIN 25
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Phen_TMU_1sub1bcm_E1_FT.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 790
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
Me2N
O
N
3'an



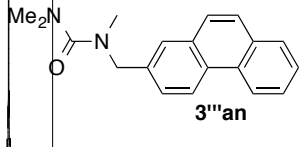
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 100
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 26
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Phenanthrene_TMU_2-sub.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 343
LKSIG 769
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3



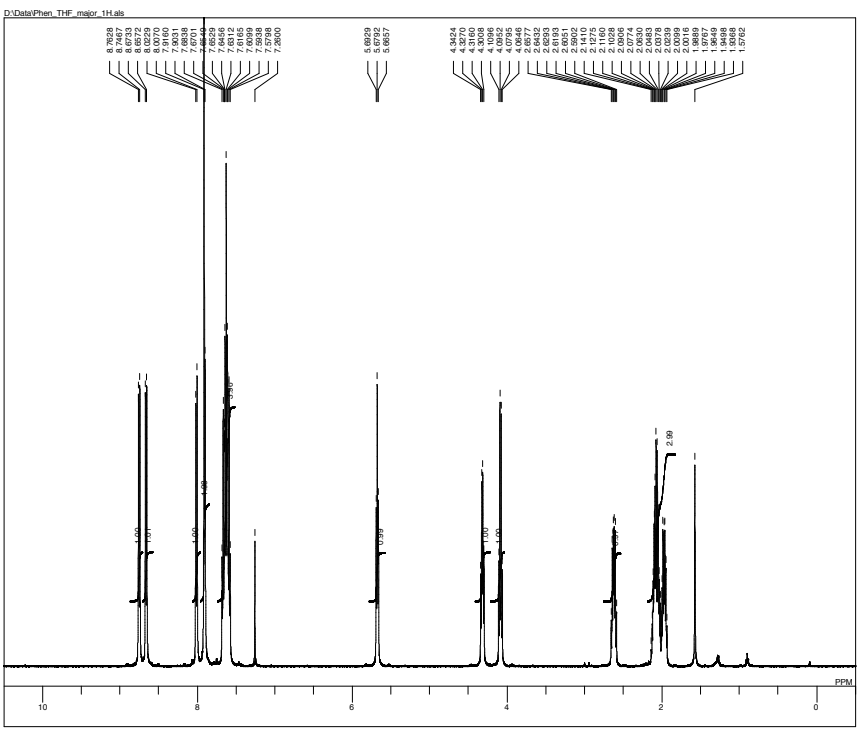
3"an



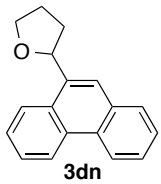
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 16
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE ru3x11-5_hplc_f20.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 343
LKSIG 817
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3



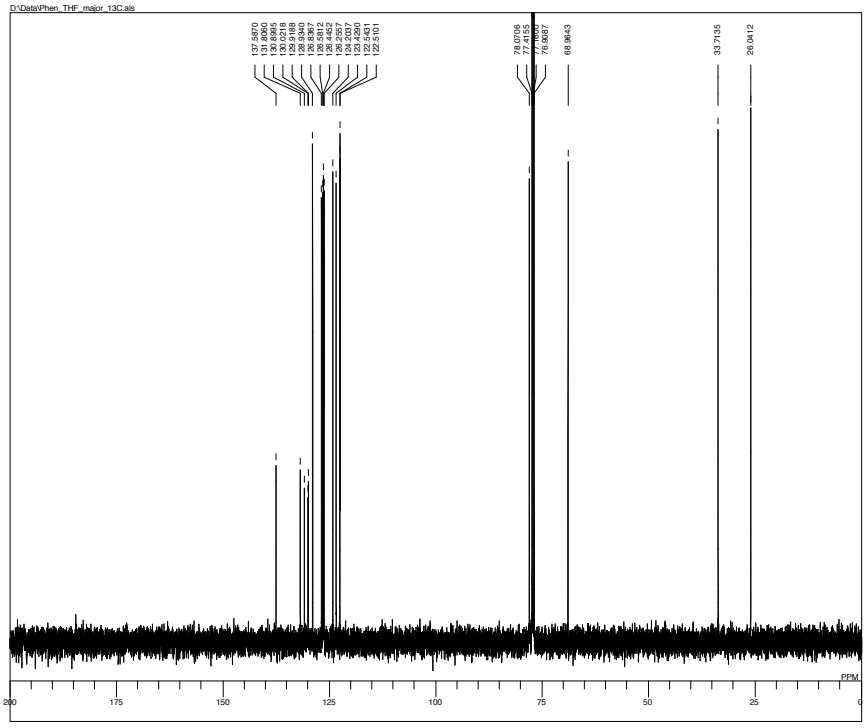
3'''an



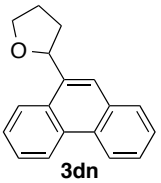

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU        8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT            16
RGAIN            18
BF             1.20 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     Phen_THF_major_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          857
CSPED         12 Hz
FILDC
FILDF
SLVNT     CDCL3
```



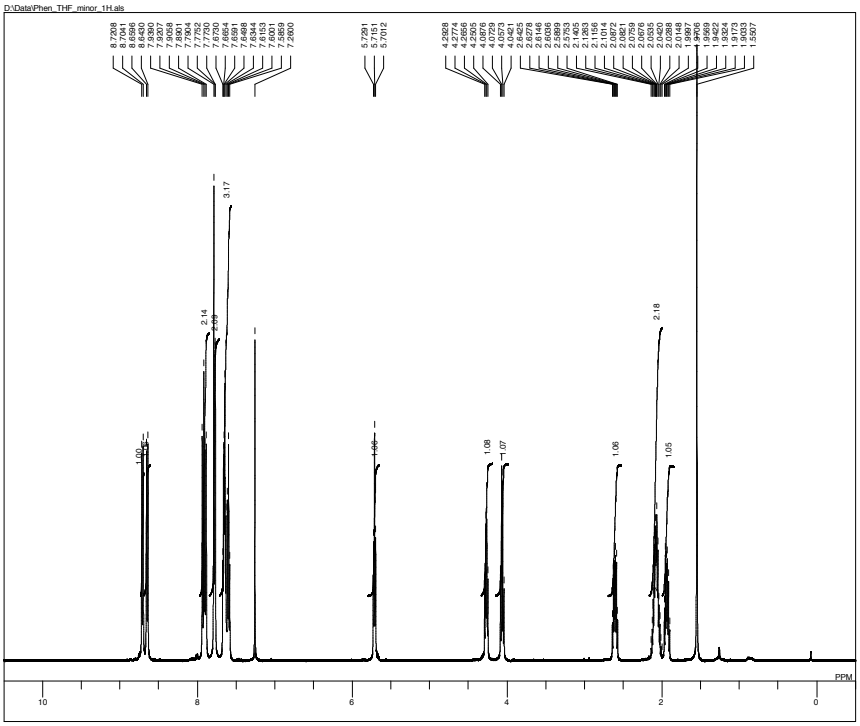
3dn



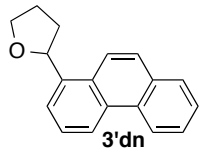

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     0.01000 msec
IWT       1000.0000 sec
POINT        65536
SPO          65536
TIMES          256
DUMMY            2
FREQU       33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM         1.9333 sec
PD            1.0670 sec
ADBIT            16
RGAIN            25
BF             1.20 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     Phen_THF_major_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          904
CSPED         12 Hz
FILDC
FILDF
SLVNT     CDCL3
```



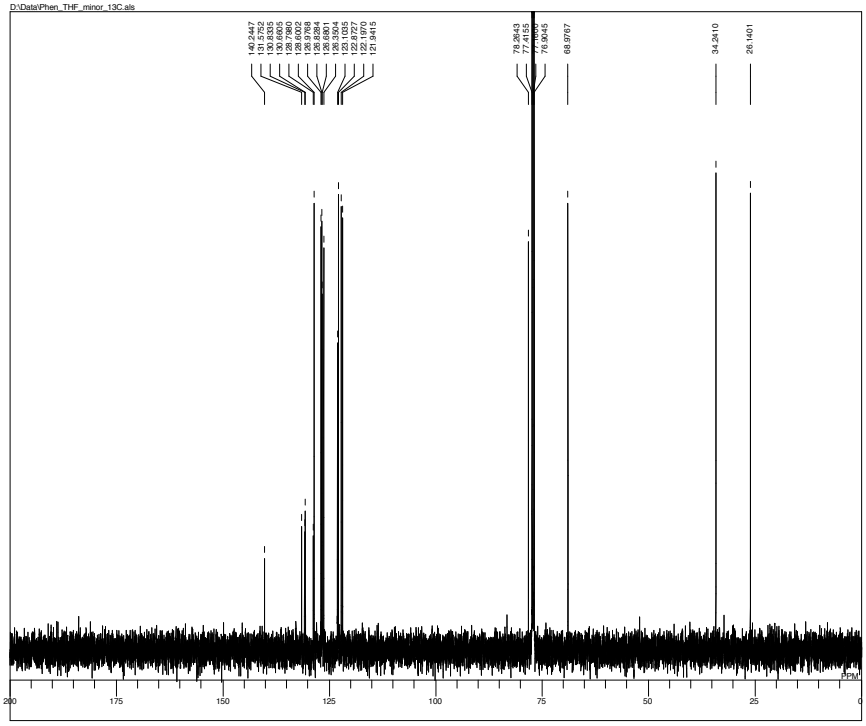
3dn




```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN      128250.00 Hz
PW1            5.50 usec
DEADT         72.30 usec
PREDL      10.00000 msec
IWT            0.5000 sec
POINT         65536
SPO           65536
TIMES             8
DUMMY             0
FREQU      8000.00 Hz
FLT            4000 Hz
DELAY         50.00 usec
ACQTM       8.1920 sec
PD             1.0000 sec
ADBIT            16
RGAIN            21
BF             1.20 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRRPW         50 usec
IRATN            511
DFILE     Phen_THF_minor_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            967
CSPED          14 Hz
FILDC
FILDF
SLVNT     CDCL3
```

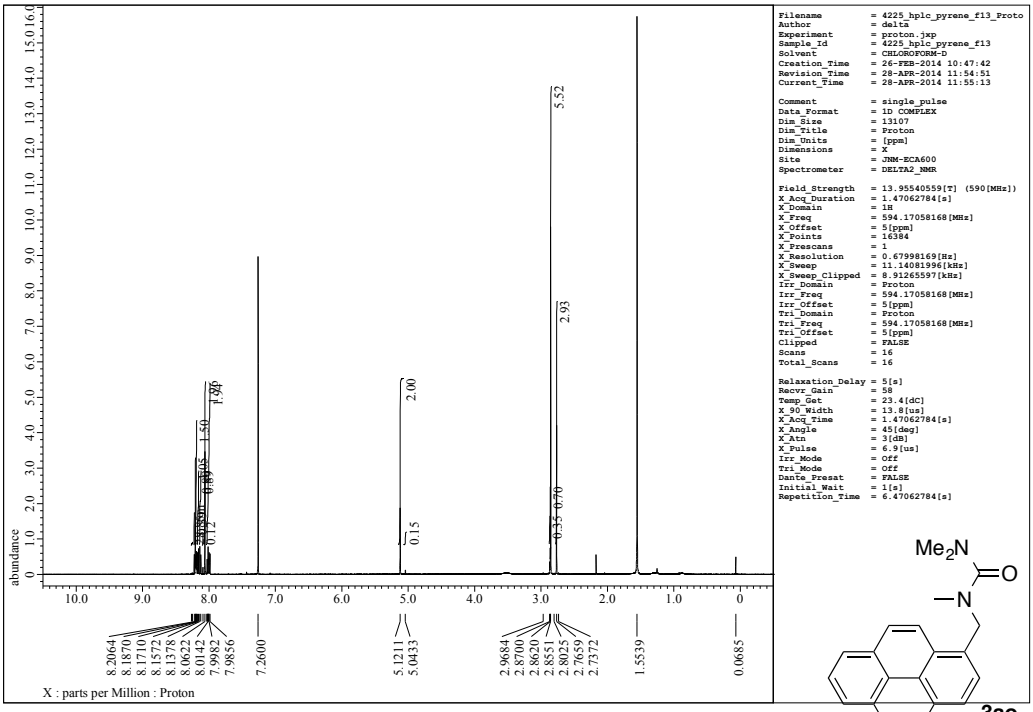


3'dn

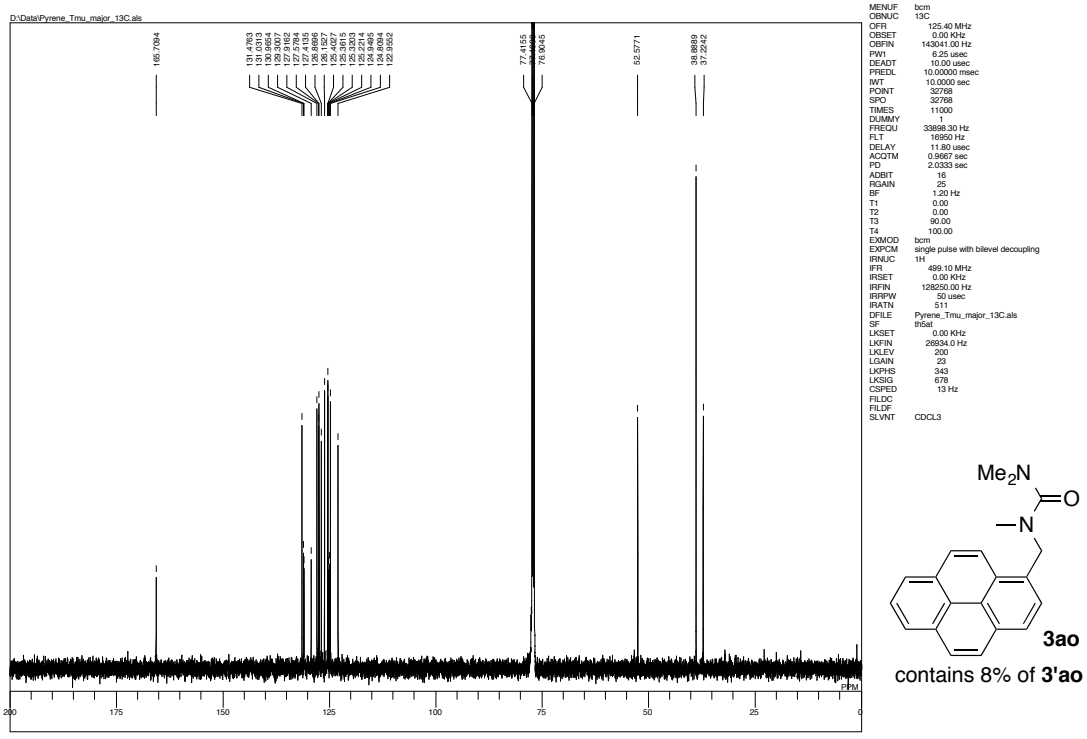



```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN      143041.00 Hz
PW1            6.25 usec
DEADT         10.00 usec
PREDL      0.01000 msec
IWT        1000.0000 sec
POINT         32768
SPO           32768
TIMES           800
DUMMY             2
FREQU      33898.30 Hz
FLT           16950 Hz
DELAY         11.80 usec
ACQTM       0.9667 sec
PD             1.0670 sec
ADBIT            16
RGAIN            25
BF             1.20 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRRPW         50 usec
IRATN            511
DFILE     Phen_THF_minor_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            938
CSPED          11 Hz
FILDC
FILDF
SLVNT     CDCL3
```

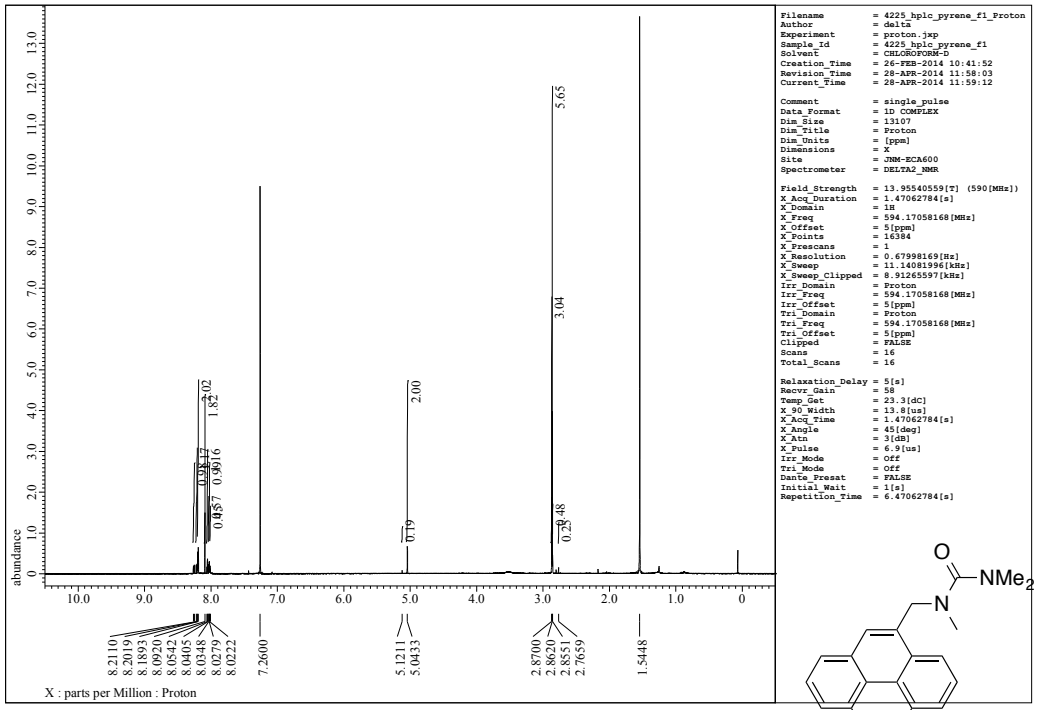
3'dn



contains 8% of **3'ao**

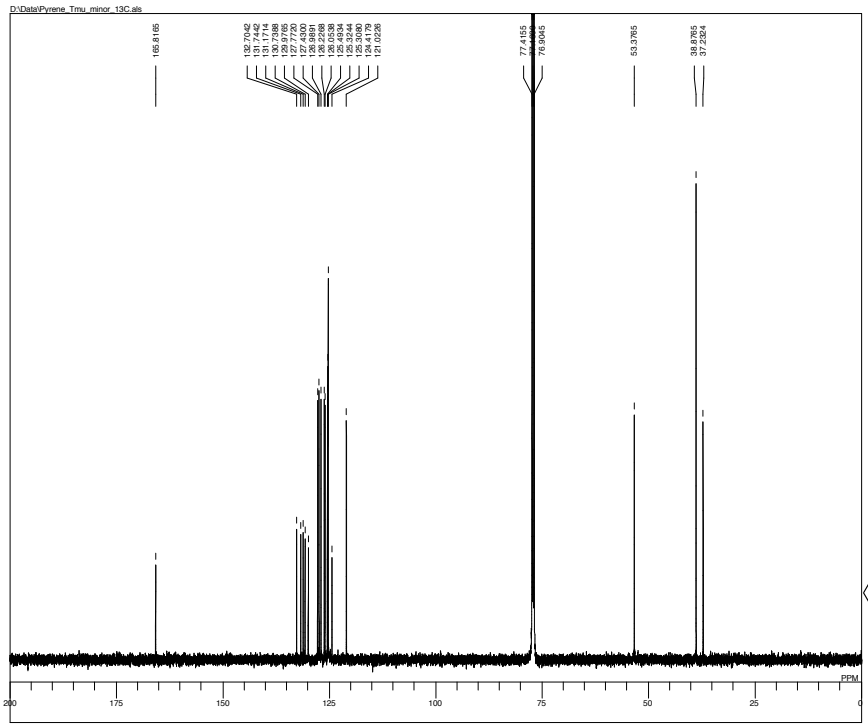


contains 8% of **3'ao**



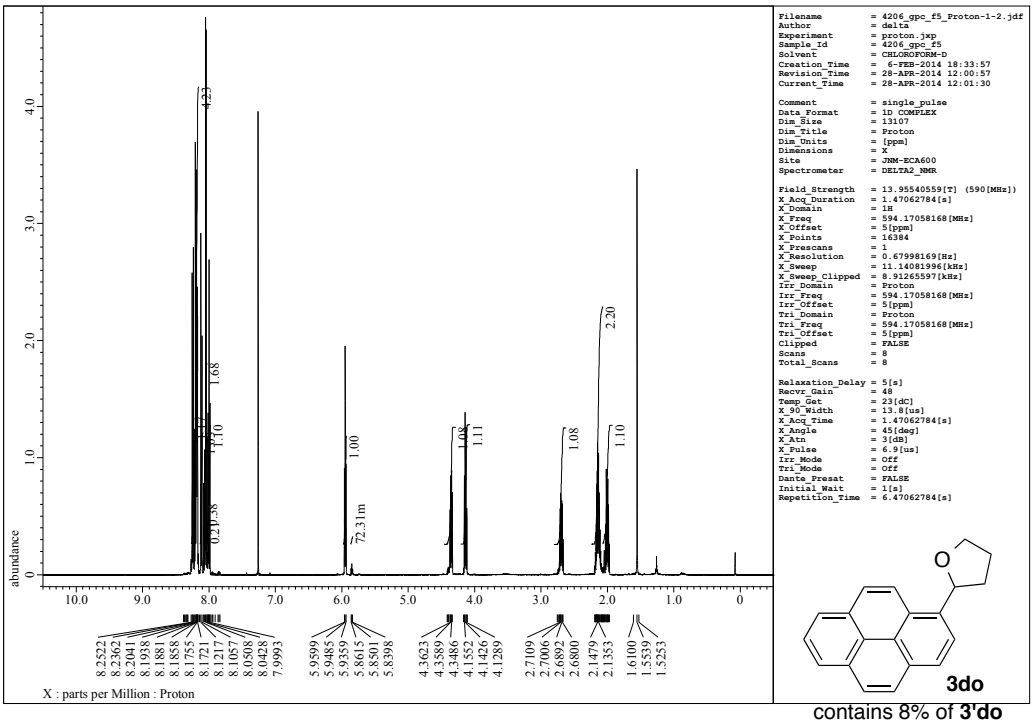
3'ao

contains 10% of **3ao**

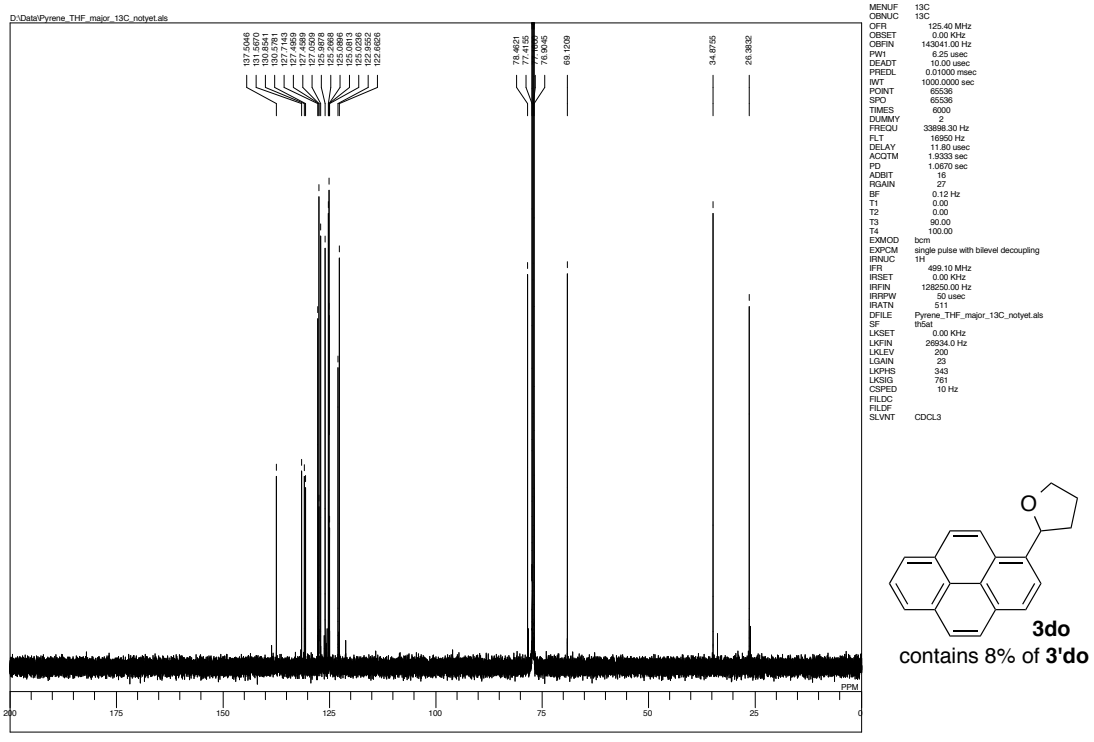


3'ao

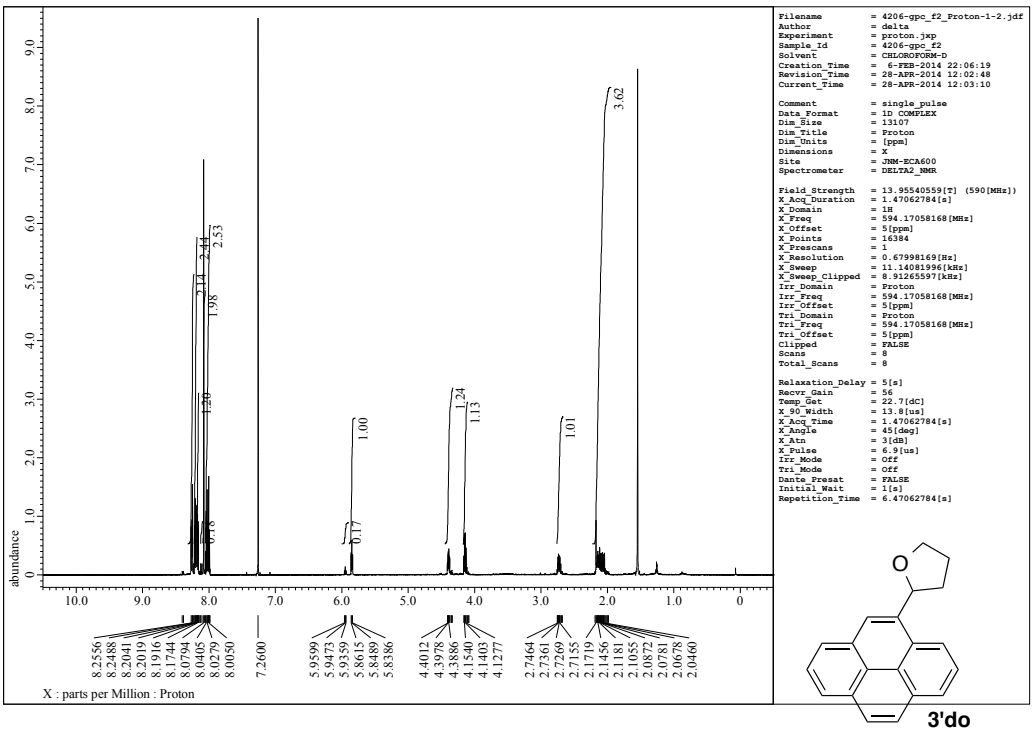
contains 10% of **3ao**



contains 8% of **3'do**

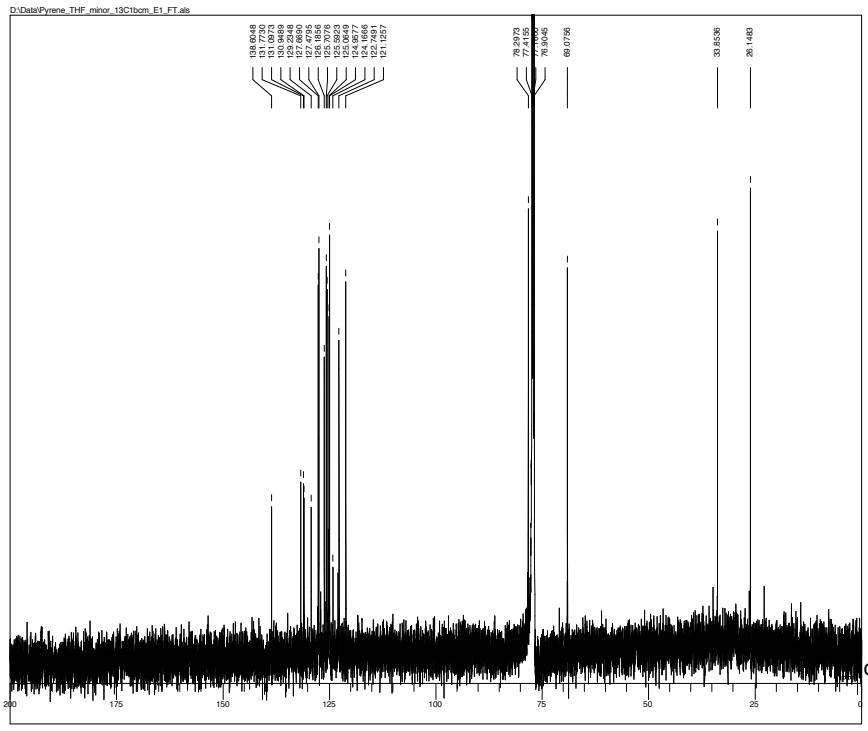


contains 8% of **3'do**



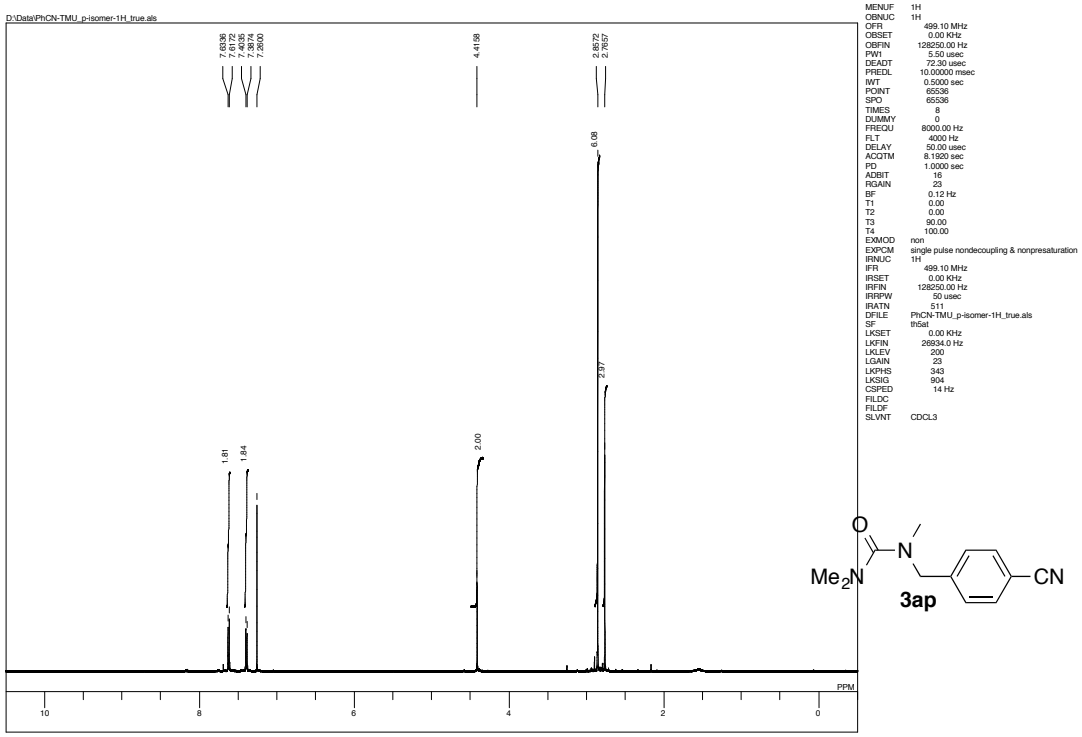
3'do

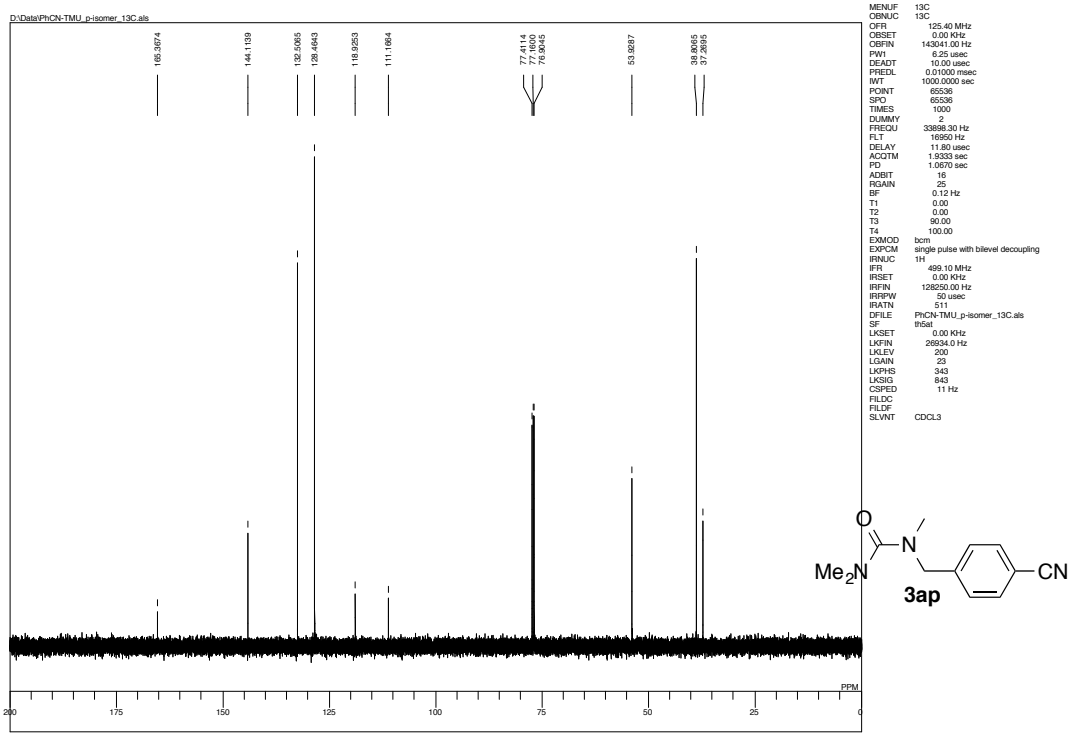
contains 12% of **3do**

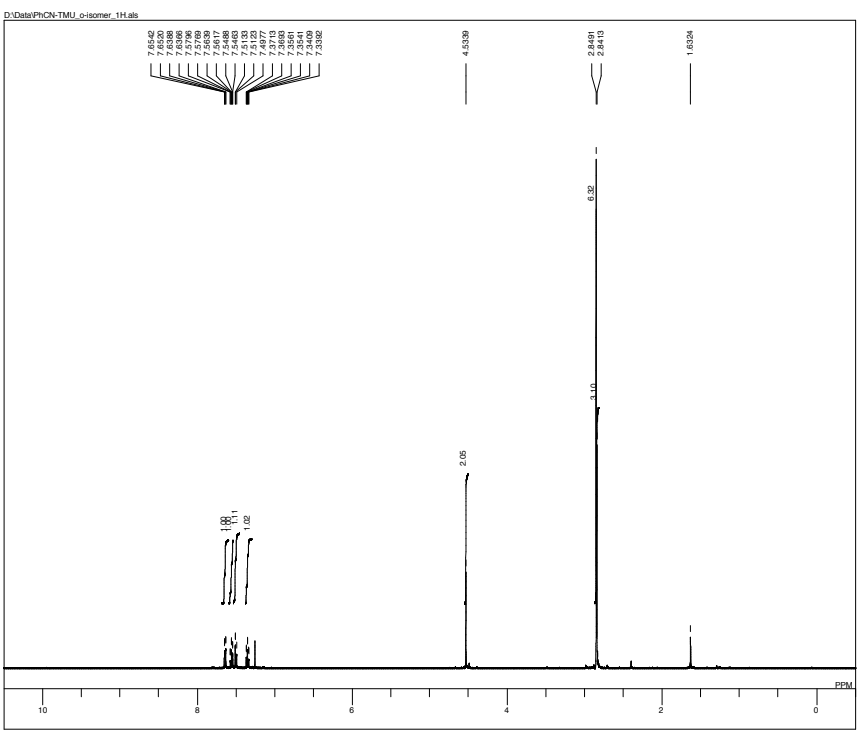


3'do

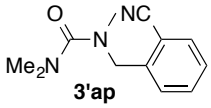
contains 12% of **3do**


D:\Data\PhCN-TMU_p-isomer-1H_true.als
7.6338
7.6172
7.4065
7.3874
7.3600
4.4159
2.8572
2.7657
6.08
2.97
2.00
1.81
1.84
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 23
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE PhCN-TMU_p-isomer-1H_true.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 904
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
O
N
Me2N
CN
3ap

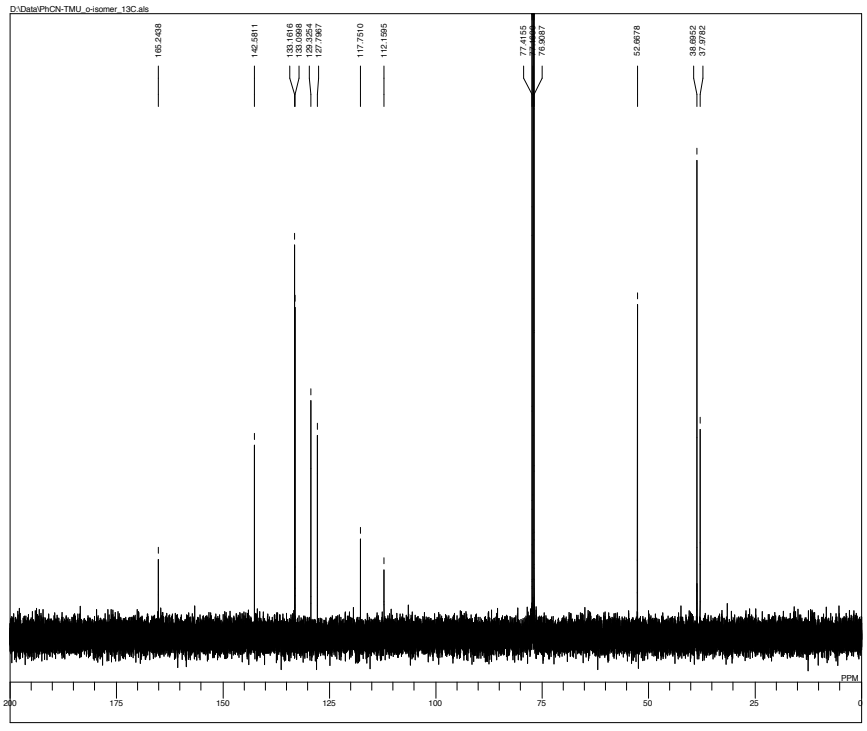

D:\Data\PhCN-TMU_p-isomer_13C.als
165.3674
144.1159
132.5065
128.4643
118.9253
111.1664
77.4114
77.1600
76.8945
53.9287
38.8065
37.2695
PPM
200
175
150
125
100
75
50
25
0
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 1000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE PhCN-TMU_p-isomer_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 843
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
O
N
Me2N
CN
3ap



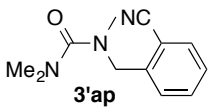

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT          0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU        8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM        8.1920 sec
PD           1.0000 sec
ADBIT            16
RGAIN            19
BF           0.12 Hz
T1           0.00
T2           0.00
T3           90.00
T4           100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     PhCN-TMU_o-isomer_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            907
CSPED          13 Hz
FILDC
FILDF
SLVNT     CDCL3
```



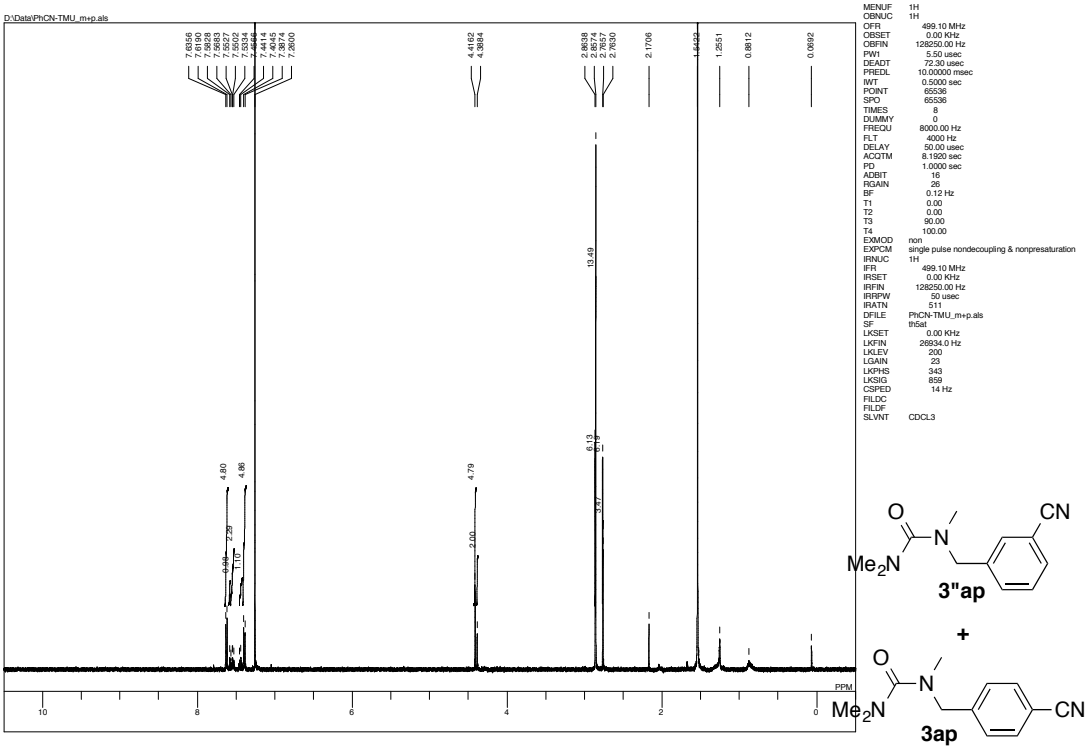
3'ap

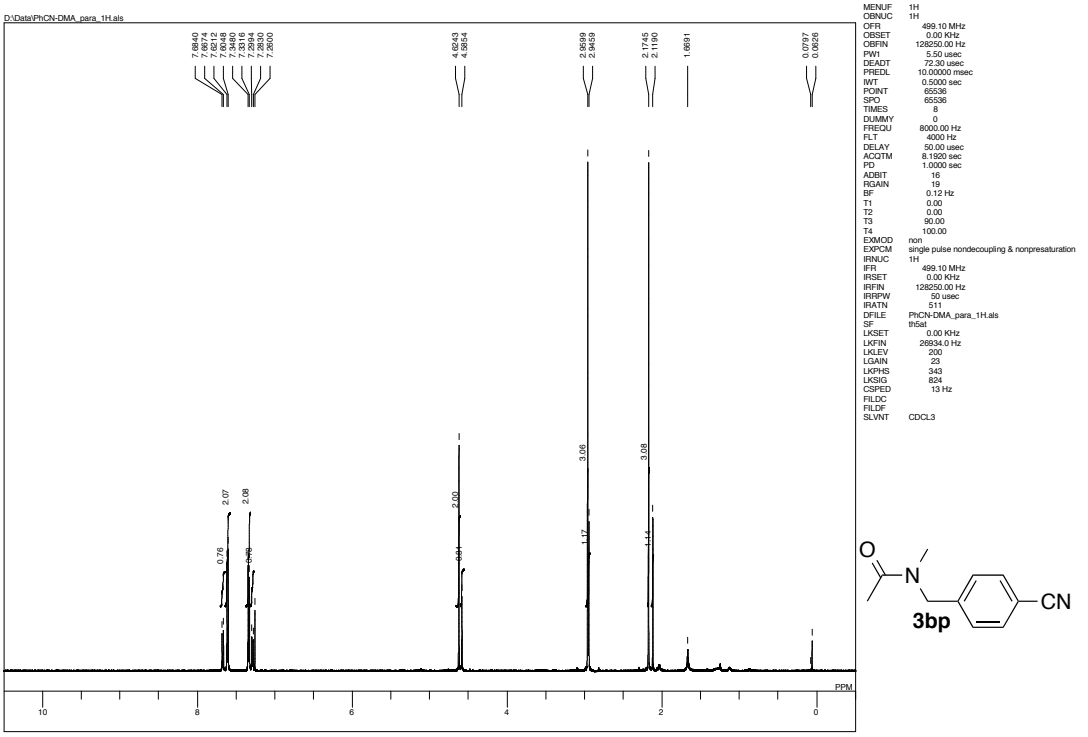


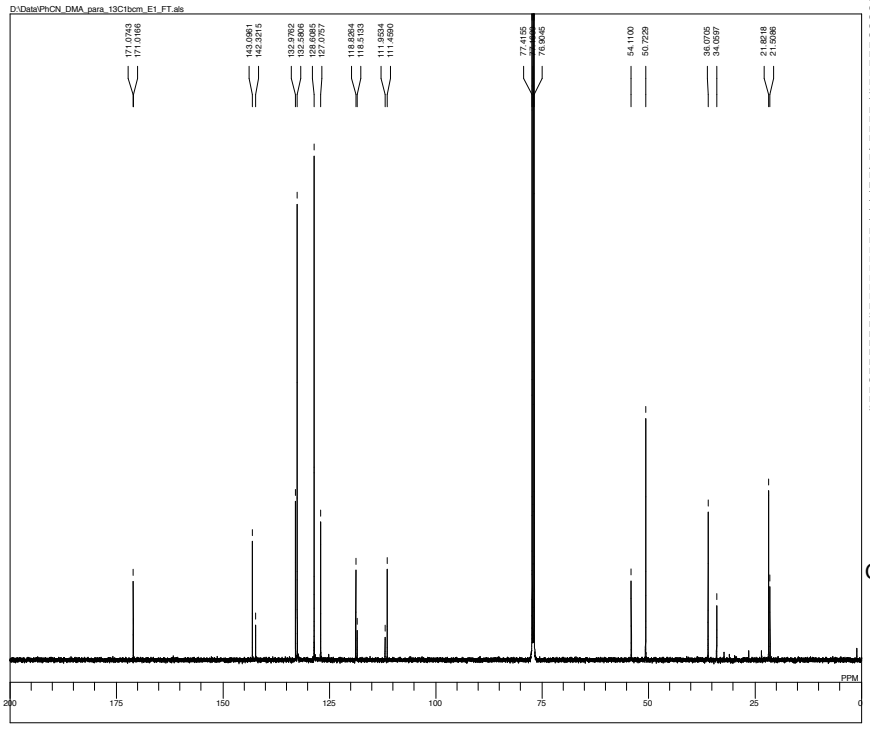

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     0.01000 msec
IWT       1000.0000 sec
POINT        65536
SPO          65536
TIMES         1000
DUMMY            2
FREQU       33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM        1.9333 sec
PD           1.0670 sec
ADBIT            16
RGAIN            25
BF           0.12 Hz
T1           0.00
T2           0.00
T3           90.00
T4           100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     PhCN-TMU_o-isomer_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            839
CSPED          10 Hz
FILDC
FILDF
SLVNT     CDCL3
```



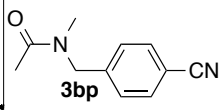
3'ap


D:\Data\PhCN-TMU_m+p.als
7.6356
7.6190
7.5928
7.5883
7.5527
7.5002
7.3334
7.4414
7.4045
7.3674
7.2800
4.4162
4.3884
2.8638
2.8574
2.8510
2.7630
2.1706
1.4422
1.2551
0.8812
0.0992
13.49
6.13
6.19
4.80
4.86
4.79
2.99
2.00
0.99
1.01
3.47
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 26
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE PhCN-TMU_m+p.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 859
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
O
N
CN
Me2N
3"ap
+
O
N
Me2N
CN
3ap

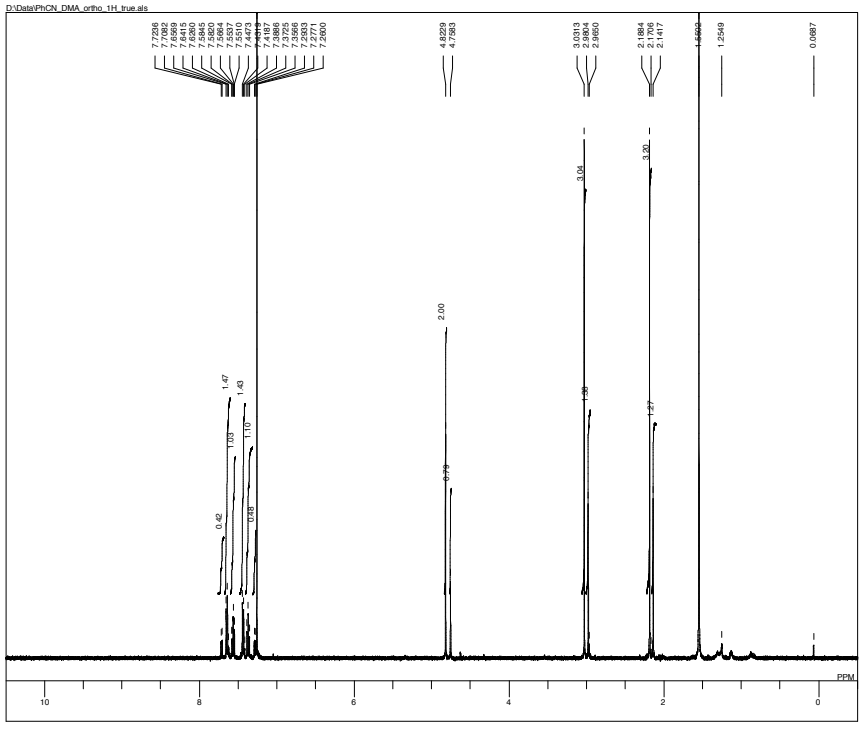

D:\Data\PhCN-DMA_para_1H.als
7.6840
7.6674
7.6513
7.6048
7.3480
7.3315
7.2994
7.2830
7.2600
4.6243
4.5954
2.9599
2.9459
2.1745
2.1190
1.6691
0.0797
0.0628
2.07
2.08
0.76
0.80
2.00
3.06
3.08
1.17
1.14
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 19
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE PhCN-DMA_para_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 824
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
O
N
CN
3bp



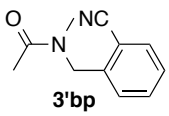

```
MENUF     bcm
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     10.00000 msec
IWT       10.0000 sec
POINT        32768
SPO          32768
TIMES        10000
DUMMY            1
FREQU     33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM     0.9667 sec
PD        2.0333 sec
ADBIT           16
RGAIN           25
BF            1.20 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN          511
DFILE     PhCN_DMA_para_13C1bcm_E1_FT.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            24
LKPHS           343
LKSIG          1100
CSPED         14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



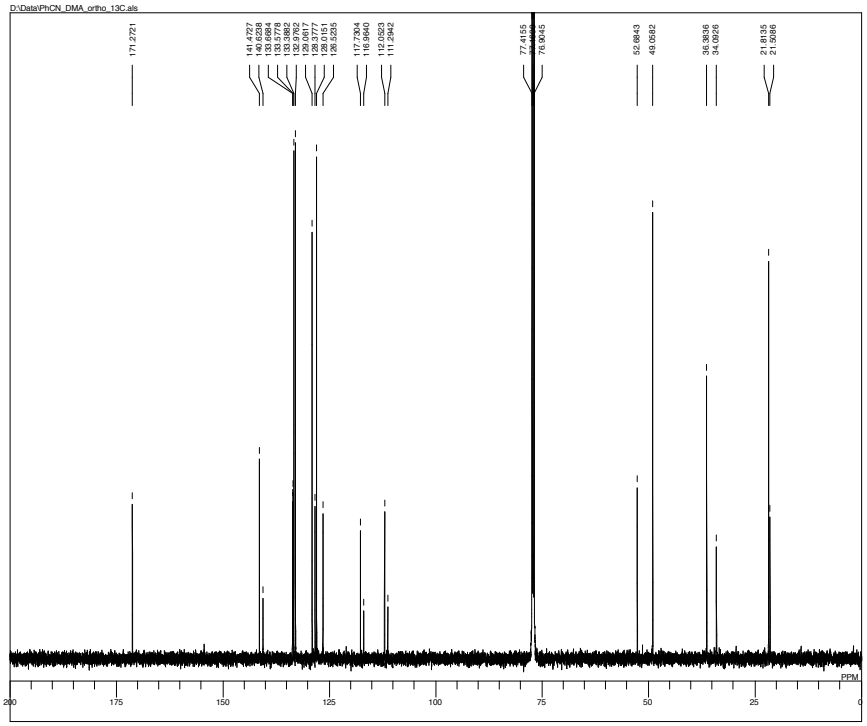
3bp



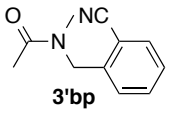

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT       0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU      8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM     8.1920 sec
PD        1.0000 sec
ADBIT           16
RGAIN           25
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN          511
DFILE     PhCN_DMA_ortho_1H_true.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG          913
CSPED         13 Hz
FILDC
FILDF
SLVNT     CDCL3
```



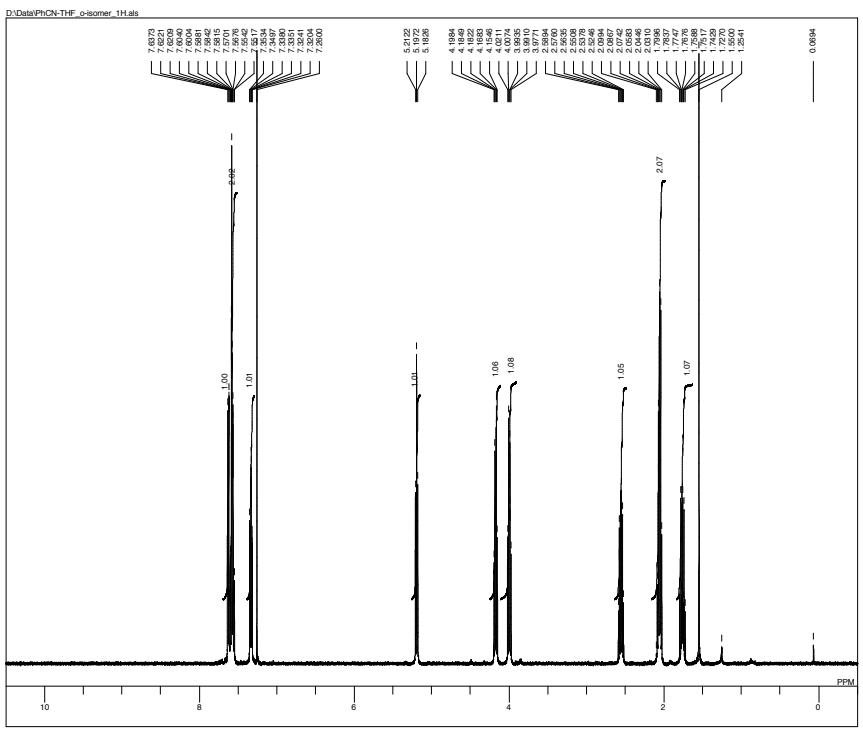
3'bp



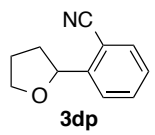

```
MENUF     bcm
OBNUC     13C
OFR       125.40 MHz
OBSET       0.00 KHz
OBFIN     143041.00 Hz
PW1         6.25 usec
DEADT      10.00 usec
PREDL    10.00000 msec
IWT       10.0000 sec
POINT      32768
SPO        32768
TIMES      10000
DUMMY          1
FREQU    33898.30 Hz
FILT       16950 Hz
DELAY      11.80 usec
ACQTM     0.9667 sec
PD        2.0333 sec
ADBIT         16
RGAIN         27
BF          1.20 Hz
T1          0.00
T2          0.00
T3         90.00
T4        100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR       499.10 MHz
IRSET       0.00 KHz
IRFIN     128250.00 Hz
IRRPW       50 usec
IRATN        511
DFILE     PhCN_DMA_ortho_13C.als
SF        th5at
LKSET       0.00 KHz
LKFIN      26934.0 Hz
LKLEV         200
LGAIN          23
LKPHS         343
LKSIG         807
CSPED       11 Hz
FILDC
FILDF
SLVNT     CDCL3
```



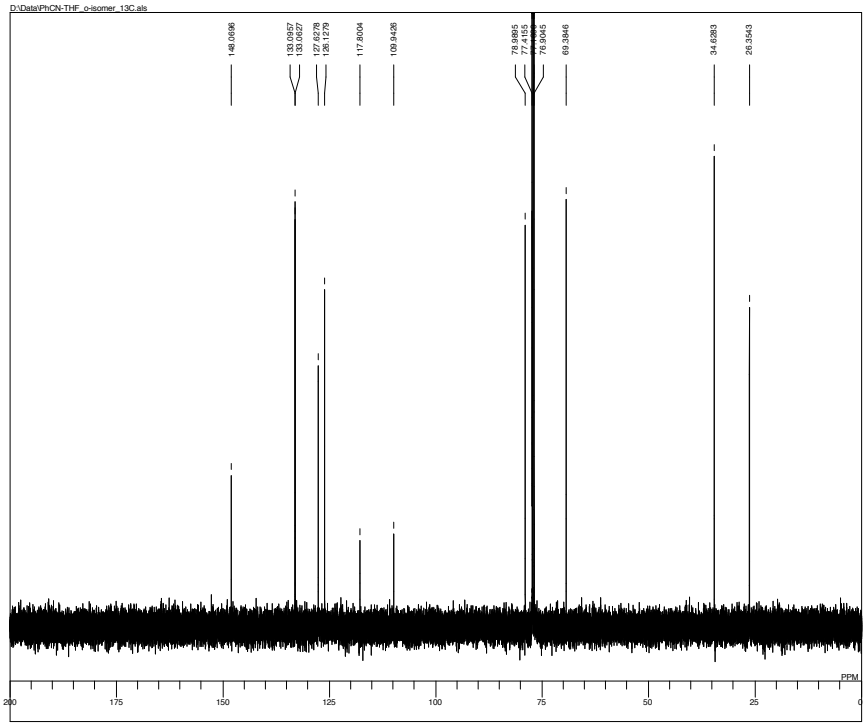
3'bp



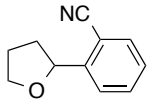

```
MENUF     1H
OBNUC     1H
OFR       499.10 MHz
OBSET       0.00 KHz
OBFIN     128250.00 Hz
PW1         5.50 usec
DEADT      72.30 usec
PREDL    10.00000 msec
IWT       0.5000 sec
POINT      65536
SPO        65536
TIMES          8
DUMMY          0
FREQU     8000.00 Hz
FILT       4000 Hz
DELAY      50.00 usec
ACQTM     8.1920 sec
PD        1.0000 sec
ADBIT         16
RGAIN         23
BF          0.12 Hz
T1          0.00
T2          0.00
T3         90.00
T4        100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR       499.10 MHz
IRSET       0.00 KHz
IRFIN     128250.00 Hz
IRRPW       50 usec
IRATN        511
DFILE     PhCN-THF_o-isomer_1H.als
SF        th5at
LKSET       0.00 KHz
LKFIN      26934.0 Hz
LKLEV         200
LGAIN          23
LKPHS         343
LKSIG         895
CSPED       11 Hz
FILDC
FILDF
SLVNT     CDCL3
```



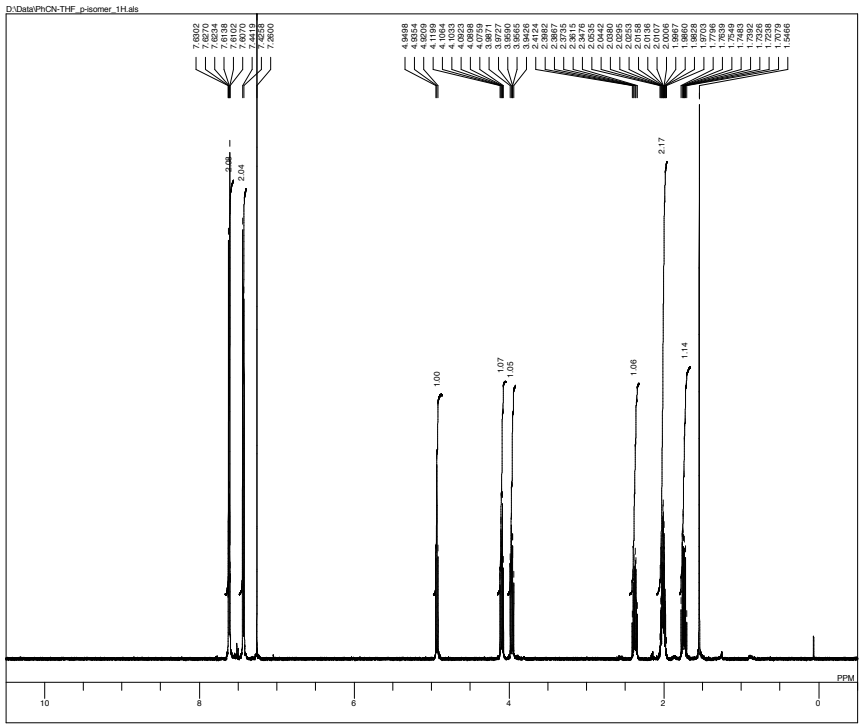
3dp



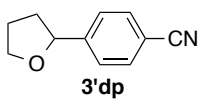

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL         0.01000 msec
IWT        1000.0000 sec
POINT        65536
SPO          65536
TIMES         1000
DUMMY            2
FREQU      33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM         1.9333 sec
PD            1.0670 sec
ADBIT           16
RGAIN           26
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN           511
DFILE     PhCN-THF_o-isomer_13C.als
SF        thSat
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           802
CSPED         13 Hz
FILDC
FILDF
SLVNT     CDCL3
```



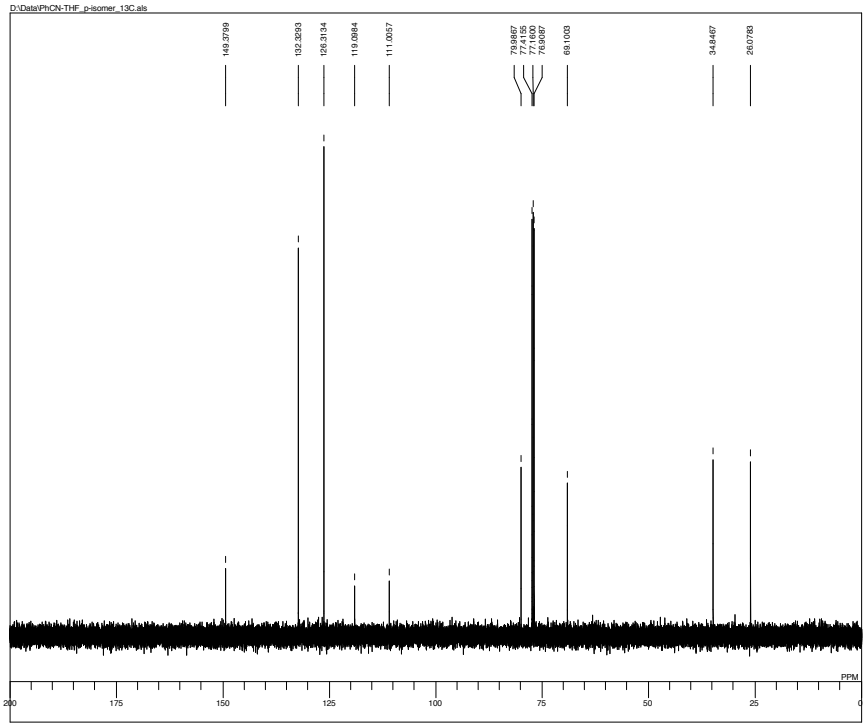
3dp



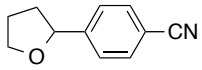

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL         10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU      8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT           16
RGAIN           23
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN           511
DFILE     PhCN-THF_p-isomer_1H.als
SF        thSat
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           956
CSPED         10 Hz
FILDC
FILDF
SLVNT     CDCL3
```



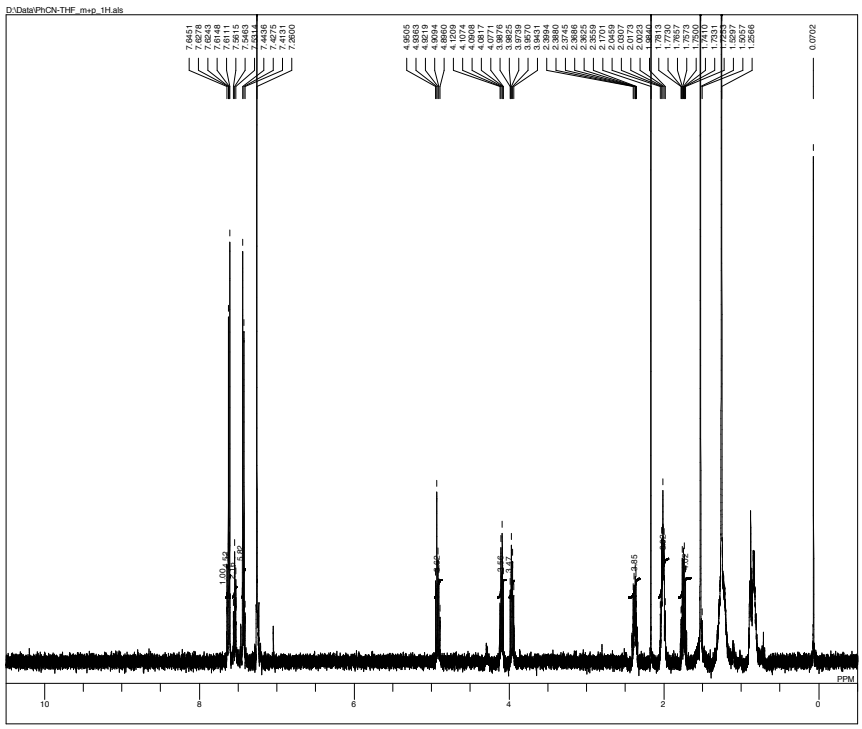
3'dp



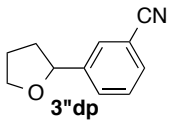

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN      143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL         0.01000 msec
IWT        1000.0000 sec
POINT        65536
SPO          65536
TIMES          256
DUMMY            2
FREQU      33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM         1.9333 sec
PD            1.0670 sec
ADBIT           16
RGAIN           25
BF            0.12 Hz
T1              0.00
T2              0.00
T3             90.00
T4            100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRPW          50 usec
IRATN          511
DFILE     PhCN-THF_p-isomer_13C.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           878
CSPED         12 Hz
FILDC
FILDF
SLVNT     CDCL3
```



3'dp

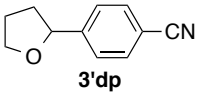



```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN      128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL         10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES           16
DUMMY            0
FREQU      8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT           16
RGAIN           27
BF            0.12 Hz
T1              0.00
T2              0.00
T3             90.00
T4            100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRPW          50 usec
IRATN          511
DFILE     PhCN-THF_m+p_1H.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           914
CSPED         11 Hz
FILDC
FILDF
SLVNT     CDCL3
```

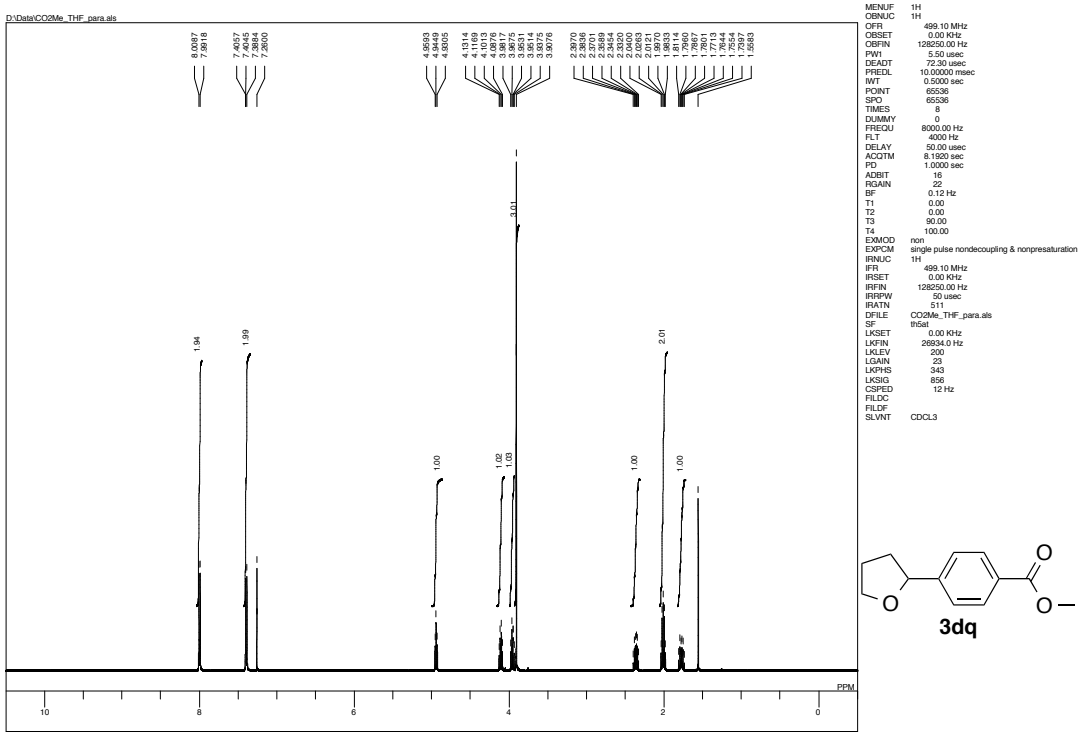


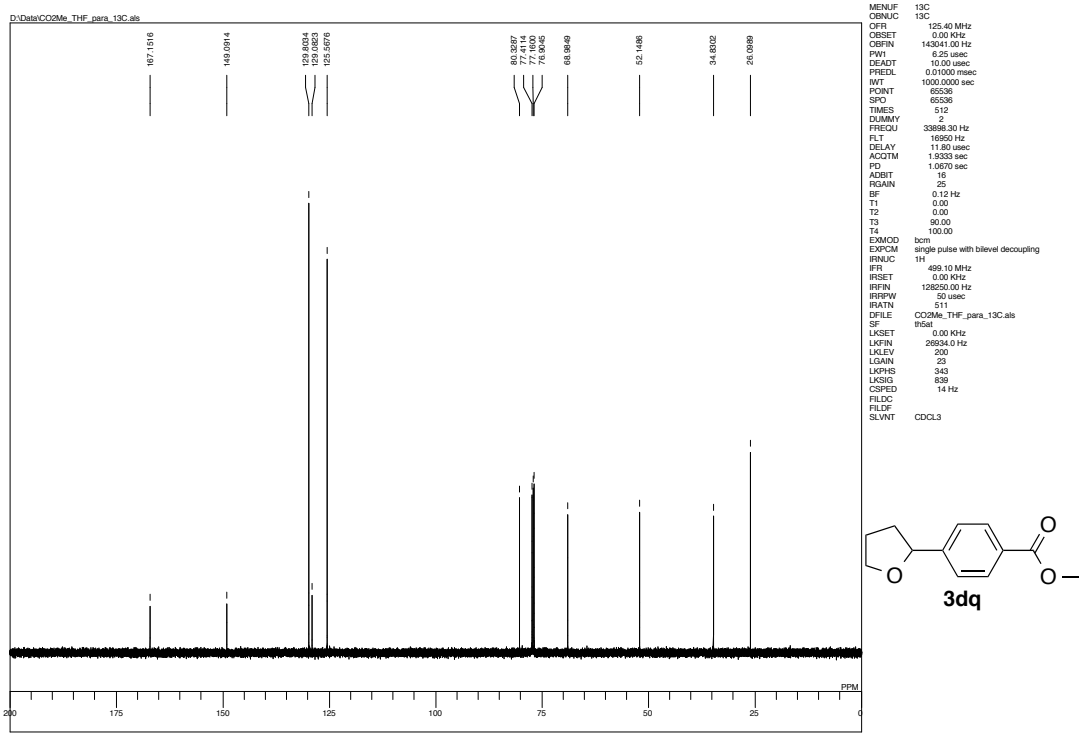
3"dp

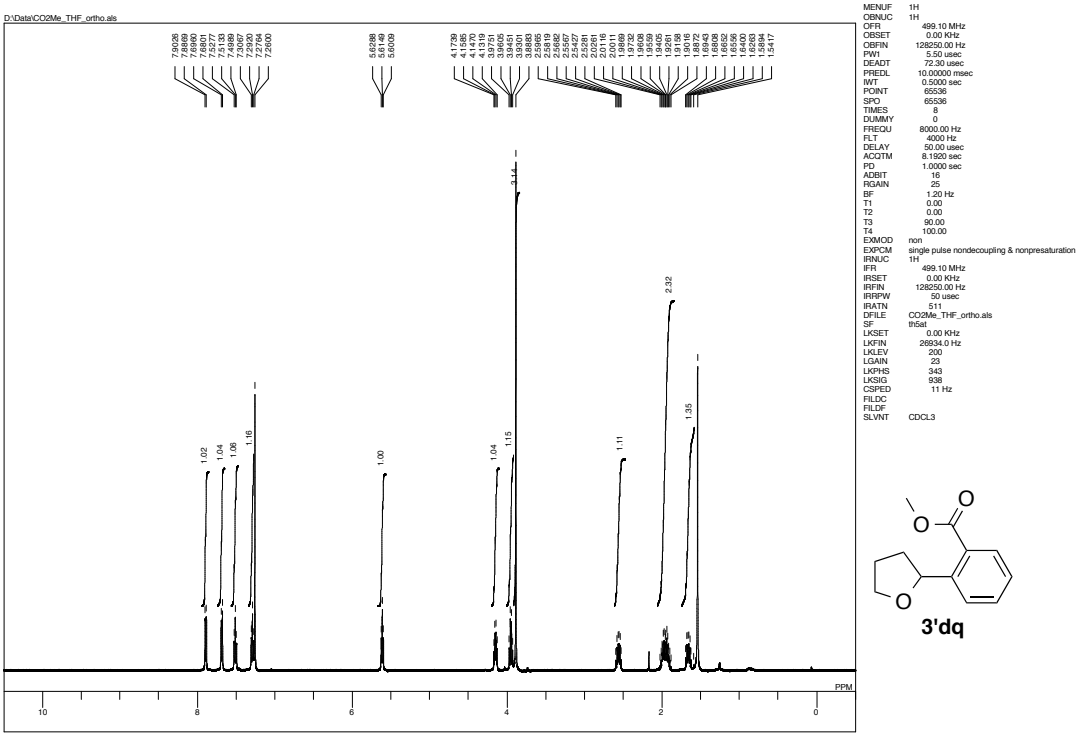
+

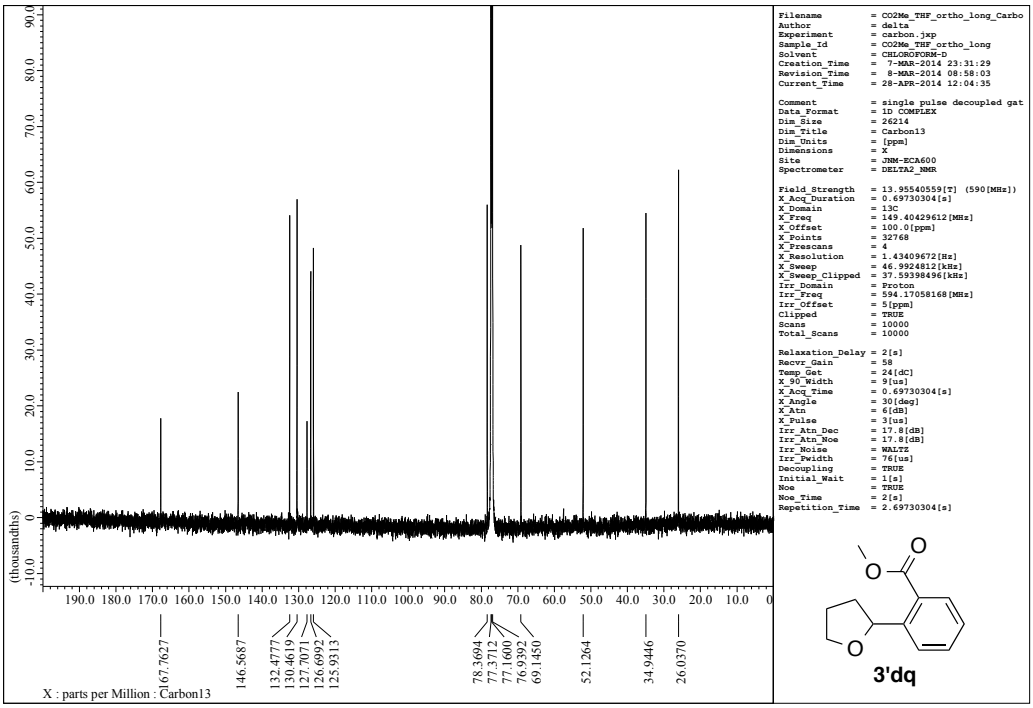


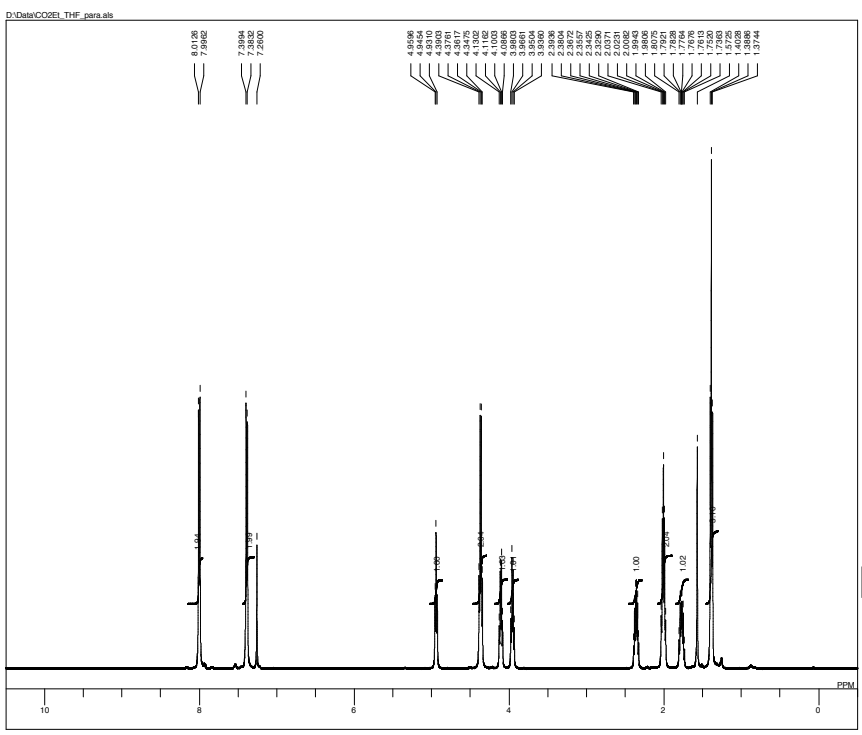
3'dp


D:\Data\CO2Me_THF_para.als
8.0087
7.9918
7.4057
7.4045
7.3864
7.3800
4.9503
4.9449
4.8905
4.1314
4.1169
4.1013
4.0857
3.9817
3.9675
3.9531
3.9514
3.9375
3.9070
2.3970
2.3836
2.3701
2.3589
2.3454
2.3320
2.2400
2.0083
2.0031
1.9970
1.9833
1.8514
1.7980
1.7867
1.7800
1.7713
1.7644
1.7554
1.7397
1.5583
1.94
1.99
3.01
2.01
1.00
1.02
1.03
1.00
1.00
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 22
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE CO2Me_THF_para.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 856
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
O
O
O
3dq

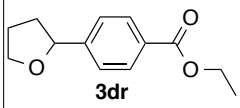

D:\Data\CO2Me_THF_para_13C.als
167.1516
149.0914
129.8834
129.0953
125.5676
80.3207
77.4114
77.1600
76.9045
68.9849
52.1486
34.8302
26.0989
PPM
200
175
150
125
100
75
50
25
0
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 512
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE CO2Me_THF_para_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 839
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3
O
O
O
3dq


D:\Data\CO2Me_THF_ortho.als
7.9026
7.8880
7.8690
7.8491
7.7877
7.5133
7.4989
7.3805
7.2920
7.2764
7.2600
5.6288
5.6149
5.6009
4.1739
4.1585
4.1470
4.1319
4.0170
3.9605
3.9451
3.9300
3.8883
2.5985
2.5819
2.5582
2.5387
2.5257
2.5281
2.0181
2.0011
1.9889
1.9702
1.9606
1.9459
1.9345
1.9261
1.9138
1.8915
1.8872
1.6943
1.6852
1.6652
1.6556
1.6440
1.6283
1.5894
1.5417
1.02
1.04
1.06
1.16
1.00
1.04
1.15
3.14
1.11
2.32
1.35
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
AOBIT 16
RGAIN 25
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRPPW 50 usec
IRATN 511
DFILE CO2Me_THF_ortho.als
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 938
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
O
O
O
3'dq

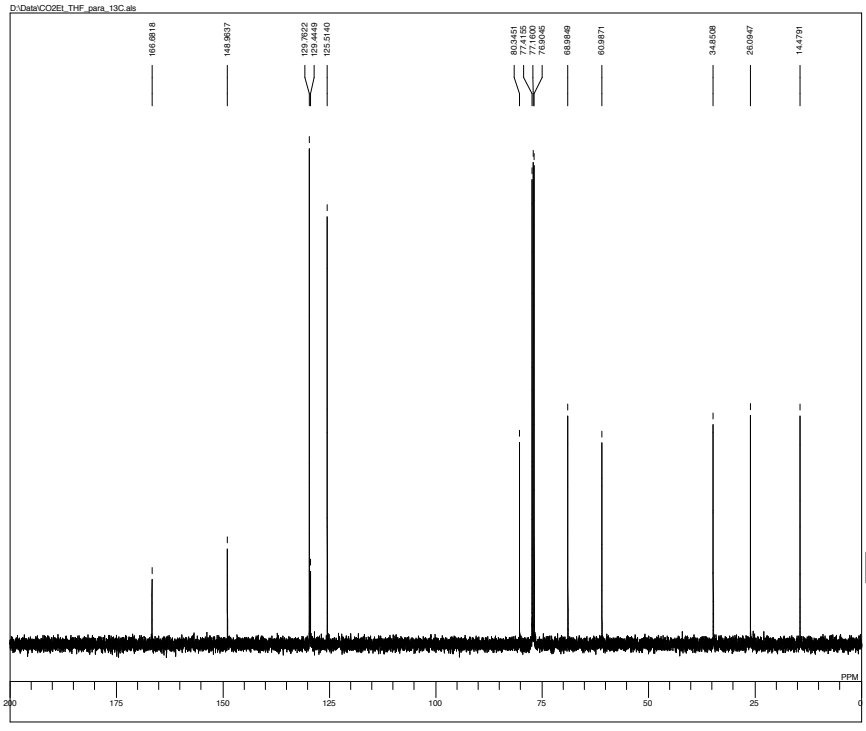

(thousandths)
90.0
80.0
70.0
60.0
50.0
40.0
30.0
20.0
10.0
0
-10.0
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
167.7627
146.5687
132.4777
130.4619
127.7071
126.6992
125.9313
78.3694
77.3712
77.1600
76.9392
69.1450
52.1264
34.9446
26.0370
X : parts per Million : Carbon13
Filename = CO2Me_THF_ortho_long_Carbo
Author = delta
Experiment = carbon.jxp
Sample_Id = CO2Me_THF_ortho_long
Solvent = CHLOROFORM-D
Creation_Time = 7-MAR-2014 23:31:29
Revision_Time = 8-MAR-2014 08:58:03
Current_Time = 28-APR-2014 12:04:35
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 10000
Total_Scans = 10000
Relaxation_Delay = 2[s]
Recvr_Gain = 58
Temp_Get = 24[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
O
O
O
3'dq



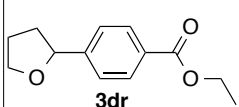

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU        8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM        8.1920 sec
PD           1.0000 sec
ADBIT           16
RGAIN           21
BF           1.20 Hz
T1           0.00
T2           0.00
T3           90.00
T4           100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN          511
DFILE     CO2Et_THF_para.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          488
CSPED         18 Hz
FILDC
FILDF
SLVNT     CDCL3
```



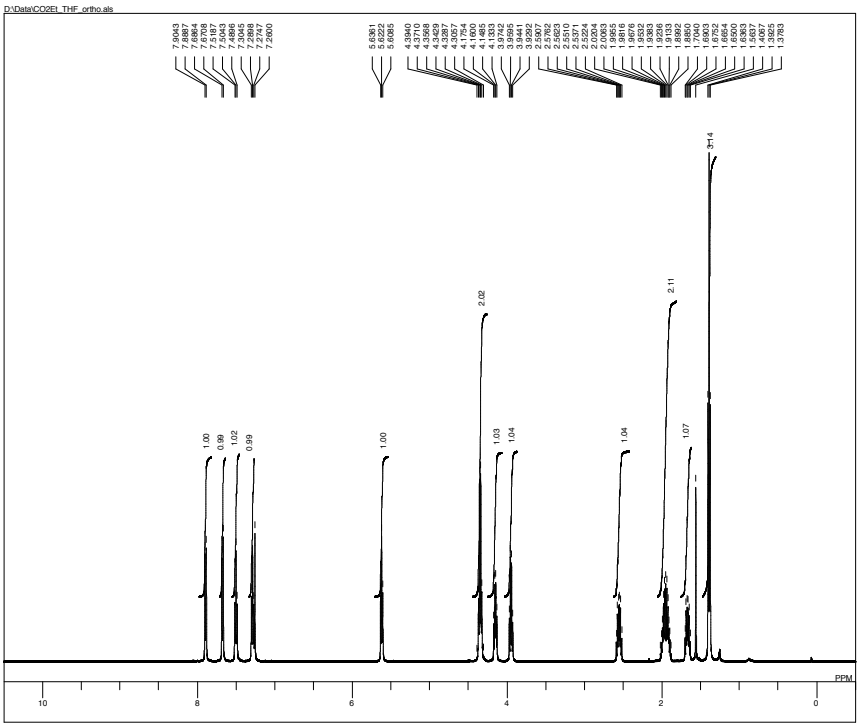
3dr



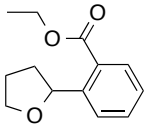

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL      0.01000 msec
IWT        1000.0000 sec
POINT        65536
SPO          65536
TIMES          256
DUMMY            2
FREQU        33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM        1.9333 sec
PD           1.0670 sec
ADBIT           16
RGAIN           25
BF           1.20 Hz
T1           0.00
T2           0.00
T3           90.00
T4           100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW          50 usec
IRATN          511
DFILE     CO2Et_THF_para_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          895
CSPED         11 Hz
FILDC
FILDF
SLVNT     CDCL3
```



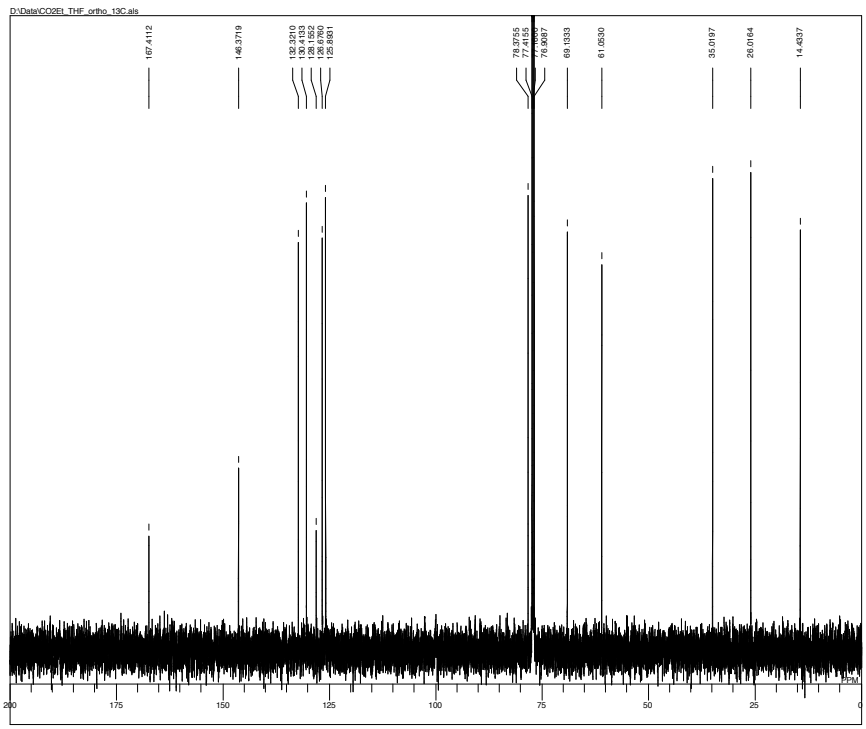
3dr



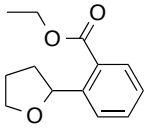

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN      128250.00 Hz
PW1            5.50 usec
DEADT         72.30 usec
PREDL      10.00000 msec
IWT            0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU       8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT           16
RGAIN           21
BF            1.20 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRRPW          50 usec
IRATN          511
DFILE     CO2Et_THF_ortho.als
SF        th5at
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          846
CSPED         14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



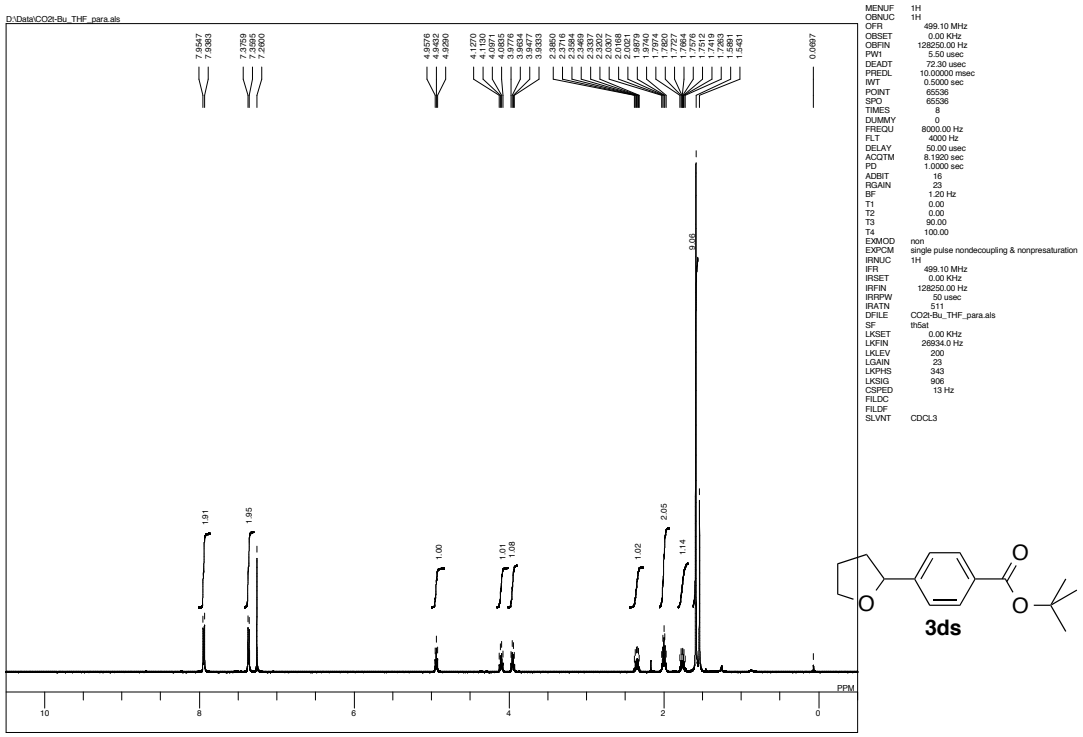
3'dr

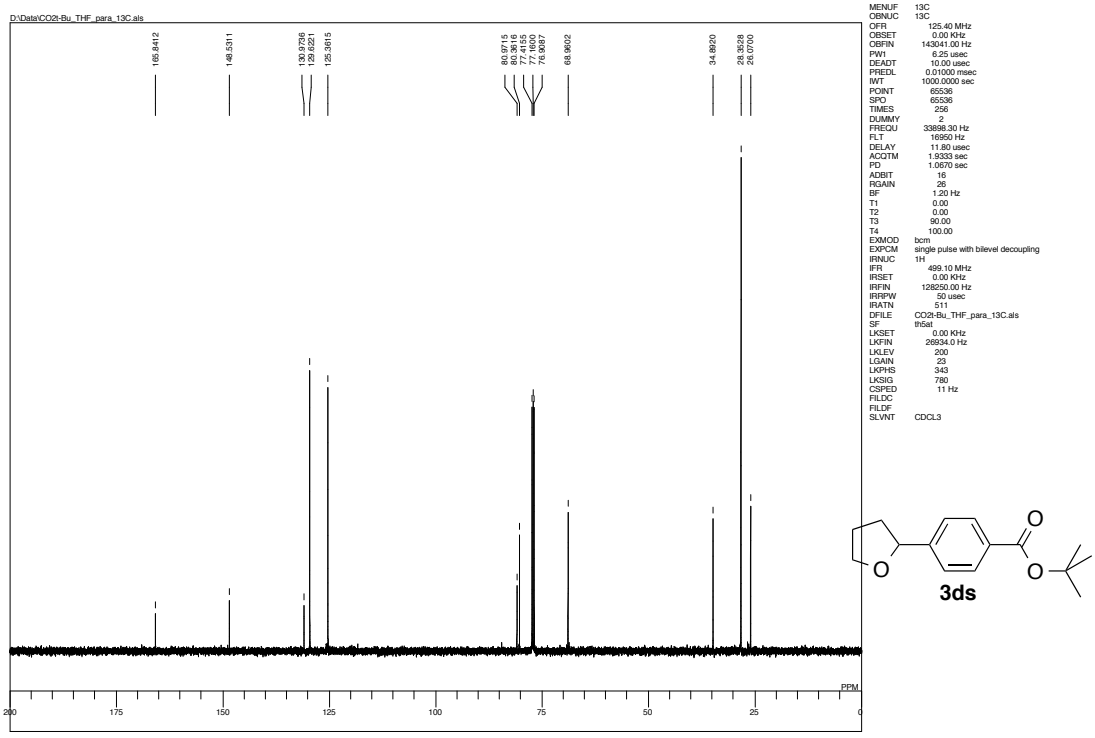


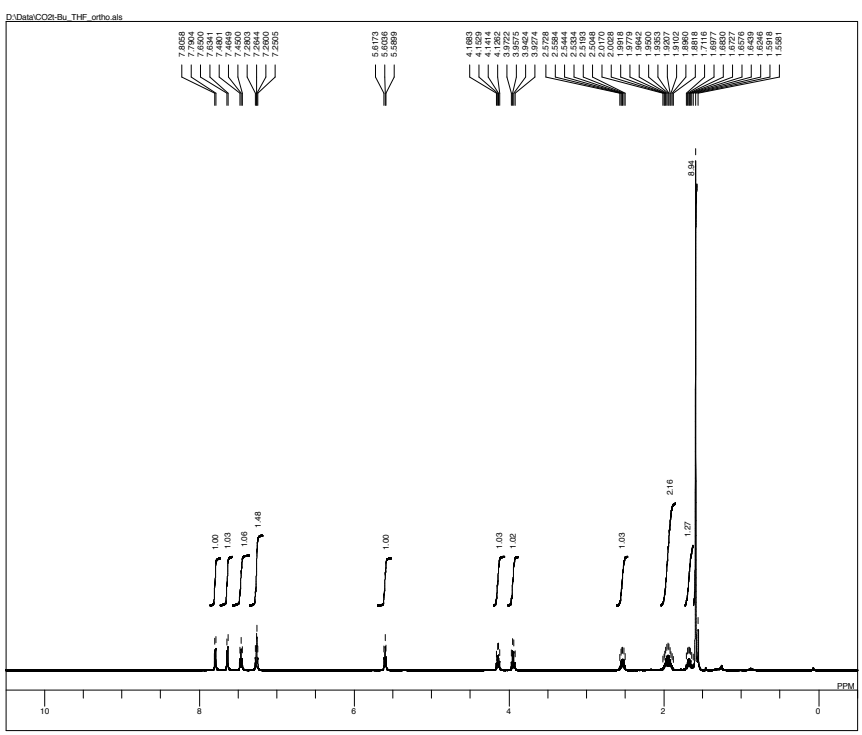

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN      143041.00 Hz
PW1            6.25 usec
DEADT         10.00 usec
PREDL      0.01000 msec
IWT        1000.0000 sec
POINT        65536
SPO          65536
TIMES          512
DUMMY            2
FREQU       33898.30 Hz
FLT          16950 Hz
DELAY         11.80 usec
ACQTM         1.9333 sec
PD            1.0670 sec
ADBIT           16
RGAIN           25
BF            1.20 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128250.00 Hz
IRRPW          50 usec
IRATN          511
DFILE     CO2Et_THF_ortho_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          800
CSPED          10 Hz
FILDC
FILDF
SLVNT     CDCL3
```



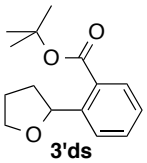
3'dr


D:\Data\CO2t-Bu_THF_para.als
7.9547
7.9383
7.3759
7.3595
7.2800
4.9578
4.9425
4.9290
4.1270
4.1130
4.0977
4.0835
3.9726
3.9574
3.9477
3.9333
2.3950
2.3710
2.3584
2.3489
2.3337
2.3202
2.3057
2.0187
2.0021
1.9879
1.9749
1.7974
1.7820
1.7772
1.7684
1.7518
1.7367
1.7219
1.5383
1.5288
1.5431
0.0997
9.06
1.91
1.95
1.00
1.01
1.08
1.02
2.05
1.14
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 23
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE CO2t-Bu_THF_para.als
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 906
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
O
O
O
3ds

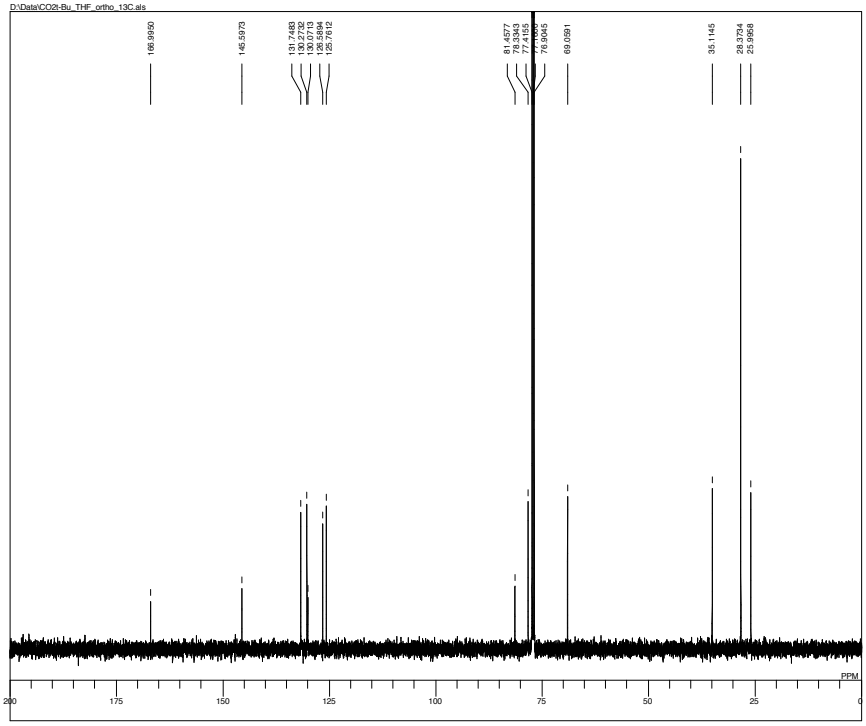

D:\Data\CO2t-Bu_THF_para_13C.als
165.8412
148.5311
130.9736
129.6221
125.3915
80.9715
80.6516
77.4155
77.1000
76.8097
68.9002
34.8920
28.8228
28.6700
PPM
200
175
150
125
100
75
50
25
0
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 26
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE CO2t-Bu_THF_para_13C.als
SF thSat
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 780
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
O
O
O
3ds



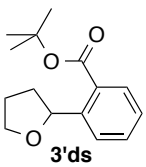
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 20
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE CO2t-Bu_THF_ortho.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 829
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3



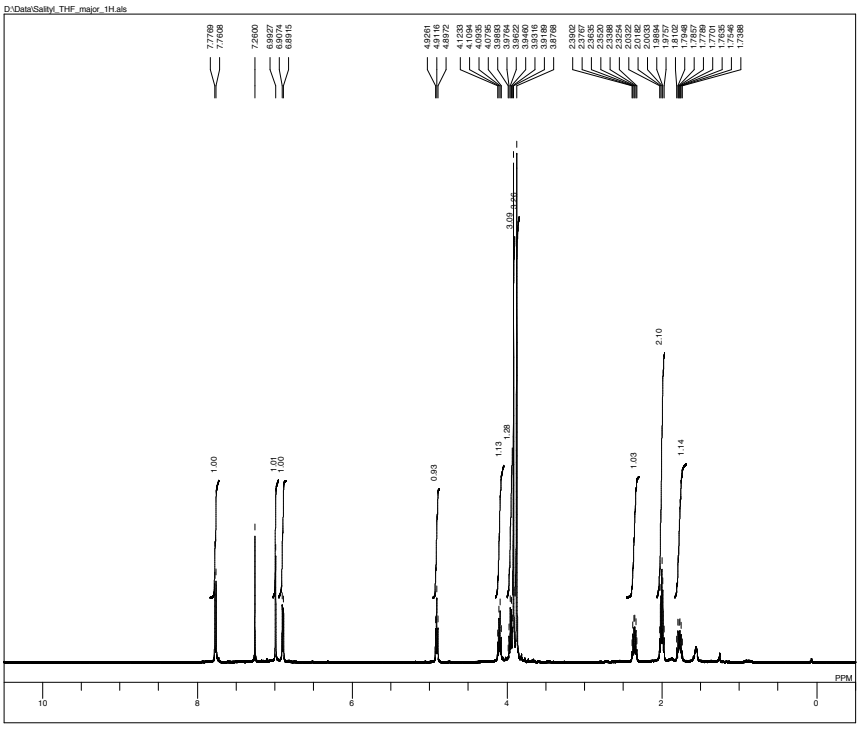
3'ds



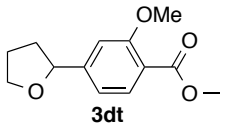
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 512
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 25
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE CO2t-Bu_THF_ortho_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 838
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3



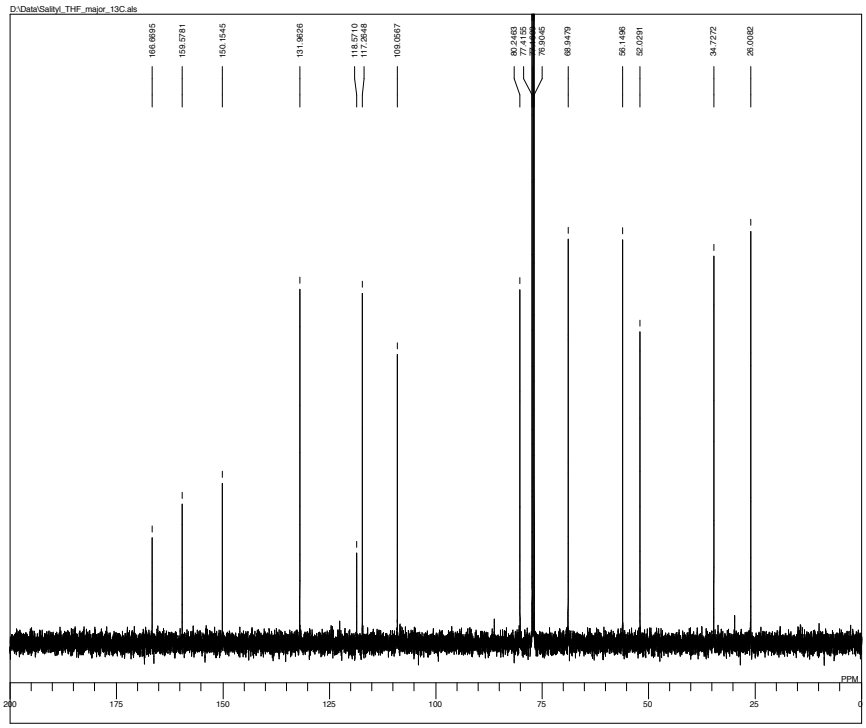
3'ds



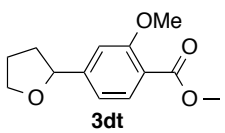

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU        8000.00 Hz
FLT          4000 Hz
DELAY         50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT            16
RGAIN            21
BF            1.20 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN           511
DFILE     Salityl_THF_major_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          859
CSPED         12 Hz
FILDC
FILDF
SLVNT     CDCL3
```



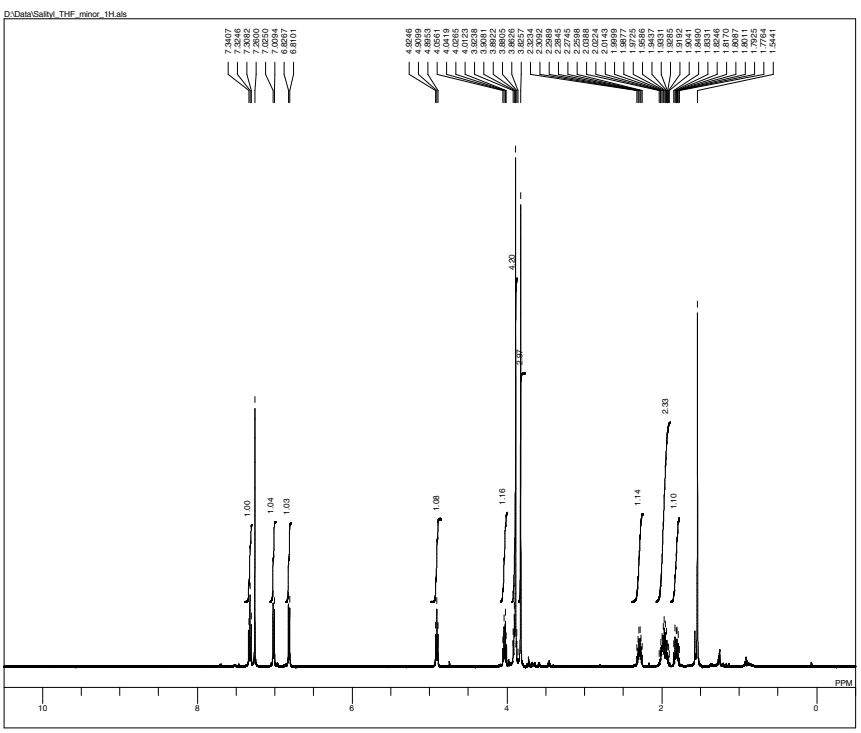
3dt



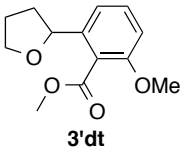

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     0.01000 msec
IWT       1000.0000 sec
POINT        65536
SPO          65536
TIMES          256
DUMMY            2
FREQU        33898.30 Hz
FLT          16950 Hz
DELAY         11.80 usec
ACQTM         1.9333 sec
PD            2.0000 sec
ADBIT            16
RGAIN            25
BF            1.20 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN           511
DFILE     Salityl_THF_major_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          854
CSPED         11 Hz
FILDC
FILDF
SLVNT     CDCL3
```



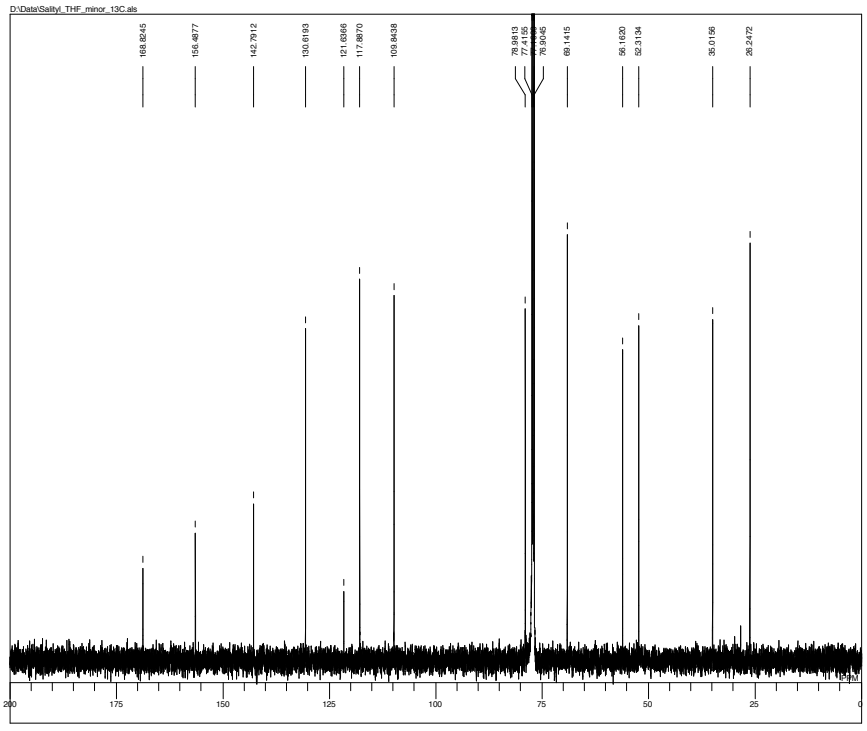
3dt



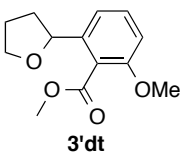

```
MENUIF      1H
OBNUC       1H
OFR           499.10 MHz
OBSET           0.00 KHz
OBFIN      128250.00 Hz
PW1             5.50 usec
DEADT          72.30 usec
PREDL       10.00000 msec
IWT             0.5000 sec
POINT          65536
SPO            65536
TIMES              8
DUMMY              0
FREQU        8000.00 Hz
FLT            4000 Hz
DELAY          50.00 usec
ACQTM          8.1920 sec
PD             1.0000 sec
ADBIT             16
RGAIN             23
BF              1.20 Hz
T1              0.00
T2              0.00
T3             90.00
T4            100.00
EXMOD       non
EXPCM       single pulse nondecoupling & nonpresaturation
IRNUC       1H
IFR           499.10 MHz
IRSET           0.00 KHz
IRFIN      128250.00 Hz
IRRPW          50 usec
IRATN             511
DFILE       Salityl_THF_minor_1H.als
SF          th5at
LKSET           0.00 KHz
LKFIN       26934.0 Hz
LKLEV             200
LGAIN              23
LKPHS             343
LKSIG             938
CSPED           13 Hz
FILDC
FILDF
SLVNT       CDCL3
```



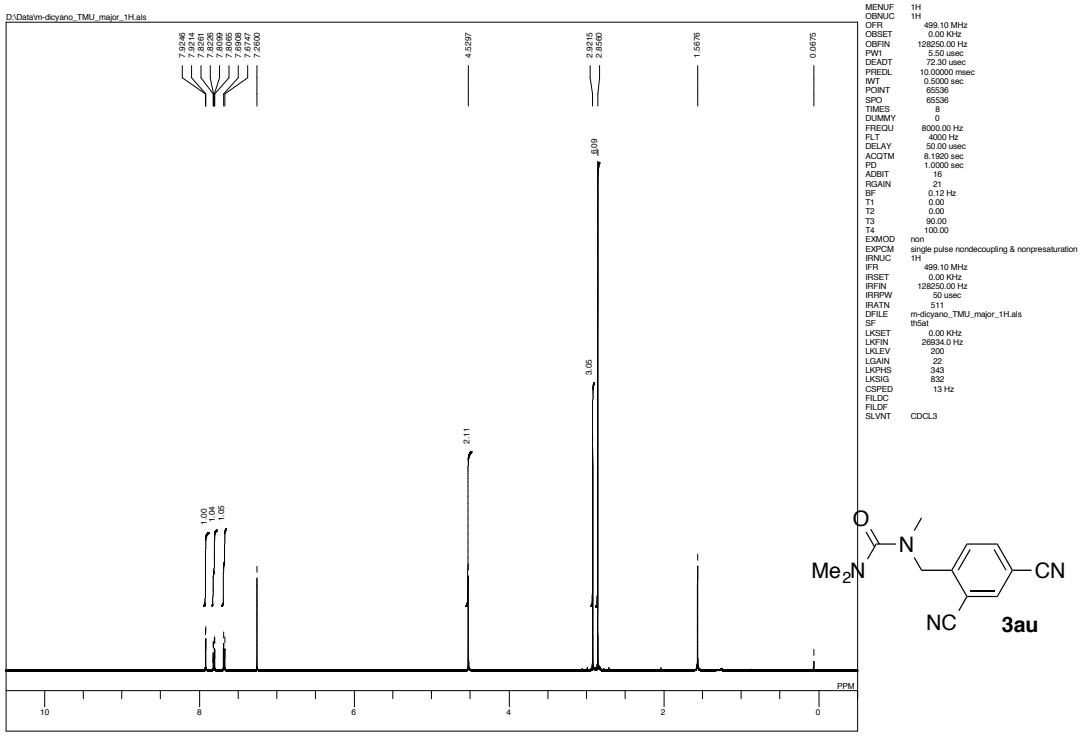
3'dt

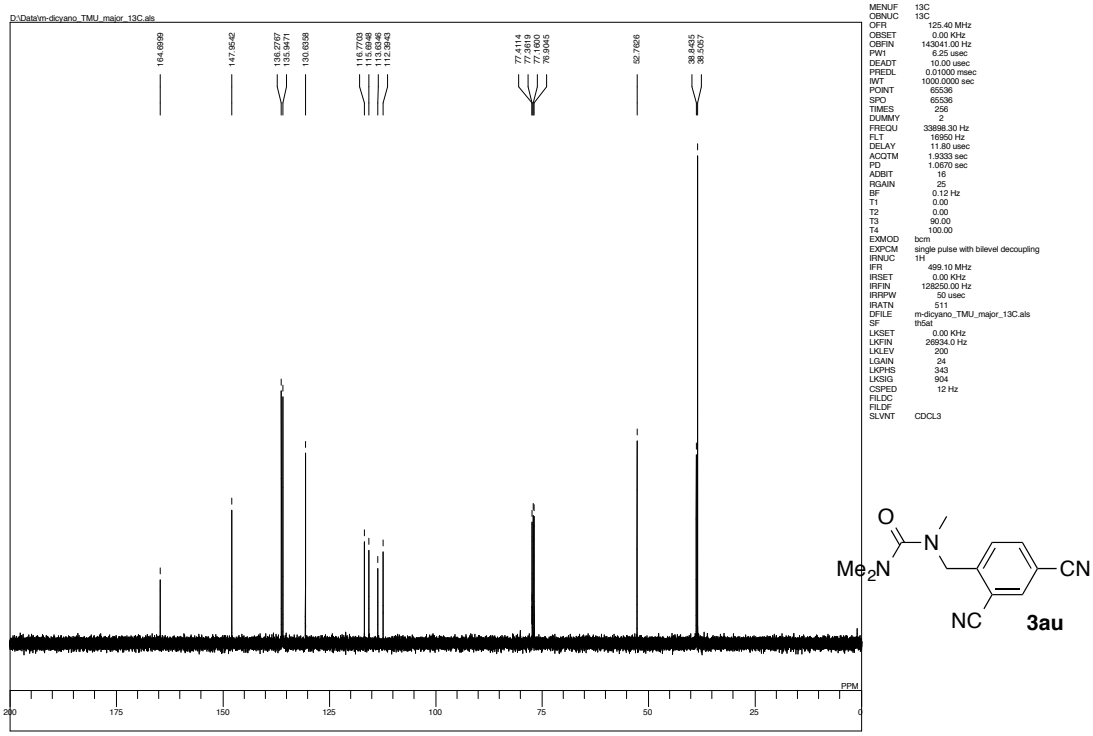


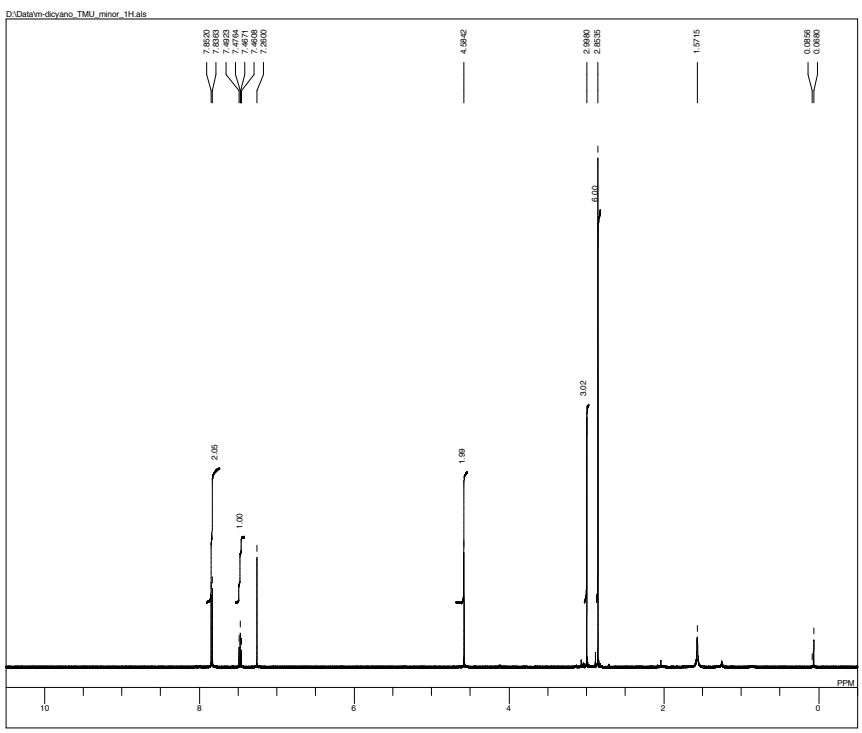

```
MENUIF      bcm
OBNUC       13C
OFR           125.40 MHz
OBSET           0.00 KHz
OBFIN      143041.00 Hz
PW1             6.25 usec
DEADT          10.00 usec
PREDL       10.00000 msec
IWT             10.0000 sec
POINT          32768
SPO            32768
TIMES          10000
DUMMY              1
FREQU       33898.30 Hz
FLT            16950 Hz
DELAY          11.80 usec
ACQTM          0.9667 sec
PD             2.0333 sec
ADBIT             16
RGAIN             27
BF              1.20 Hz
T1              0.00
T2              0.00
T3             90.00
T4            100.00
EXMOD       bcm
EXPCM       single pulse with bilevel decoupling
IRNUC       1H
IFR           499.10 MHz
IRSET           0.00 KHz
IRFIN      128250.00 Hz
IRRPW          50 usec
IRATN             511
DFILE       Salityl_THF_minor_13C.als
SF          th5at
LKSET           0.00 KHz
LKFIN       26934.0 Hz
LKLEV             200
LGAIN              23
LKPHS             343
LKSIG             845
CSPED           12 Hz
FILDC
FILDF
SLVNT       CDCL3
```



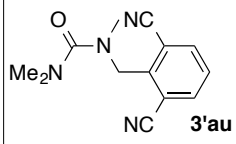
3'dt


D:\Data\m-dicyano_TMU_major_1H.als
7.9246
7.9214
7.8261
7.8228
7.8099
7.8065
7.6908
7.6747
7.2500
4.5297
2.9215
2.8560
1.5676
0.0675
6.09
3.05
2.11
1.00
1.04
1.05
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 21
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE m-dicyano_TMU_major_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 22
LKPHS 343
LKSIG 832
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
O
N
Me2N
CN
NC
3au

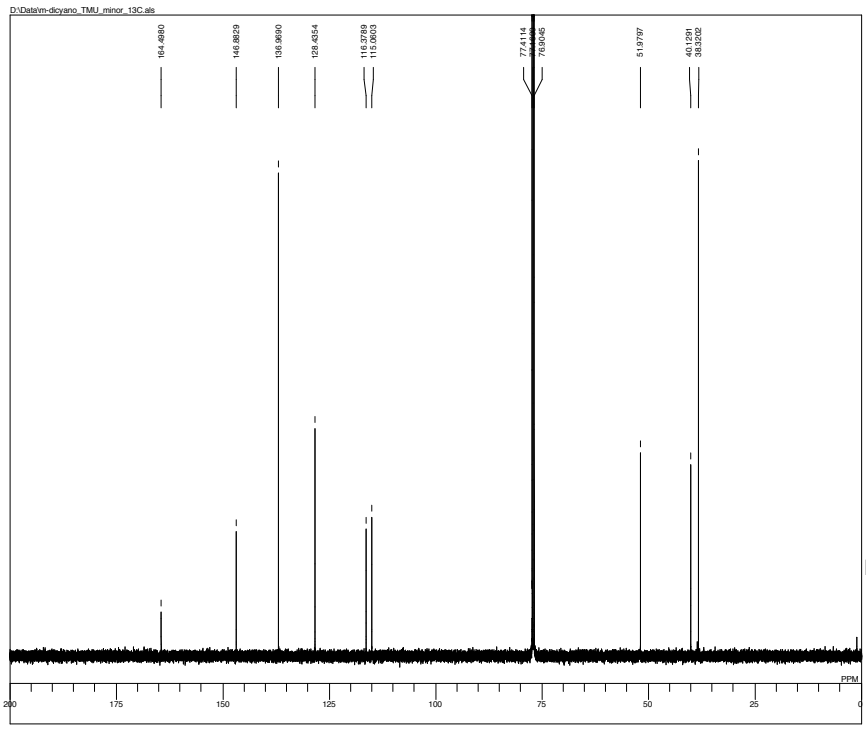

D:\Data\m-dicyano_TMU_major_13C.als
164.6999
147.9542
135.2797
133.9471
130.6338
116.7703
115.6948
113.8346
112.3943
77.4114
77.1600
76.9045
52.7626
38.8435
38.5057
PPM
200
175
150
125
100
75
50
25
0
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE m-dicyano_TMU_major_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 24
LKPHS 343
LKSIG 904
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
O
N
Me2N
CN
NC
3au



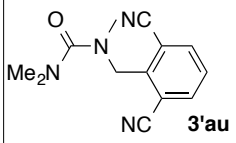

```
MENUF    1H
OBNUC    1H
OFR        499.10 MHz
OBSET        0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT          0.5000 sec
POINT       65536
SPO         65536
TIMES           8
DUMMY           0
FREQU       8000.00 Hz
FLT         4000 Hz
DELAY        50.00 usec
ACQTM        8.1920 sec
PD           1.0000 sec
ADBIT          16
RGAIN          22
BF           0.12 Hz
T1           0.00
T2           0.00
T3          90.00
T4         100.00
EXMOD    non
EXPCM    single pulse nondecoupling & nonpresaturation
IRNUC    1H
IFR        499.10 MHz
IRSET        0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN         511
DFILE    m-dicyano_TMU_minor_1H.als
SF       th5at
LKSET        0.00 KHz
LKFIN       26934.0 Hz
LKLEV         200
LGAIN          22
LKPHS         343
LKSIG         836
CSPED          14 Hz
FILDC
FILDF
SLVNT    CDCL3
```



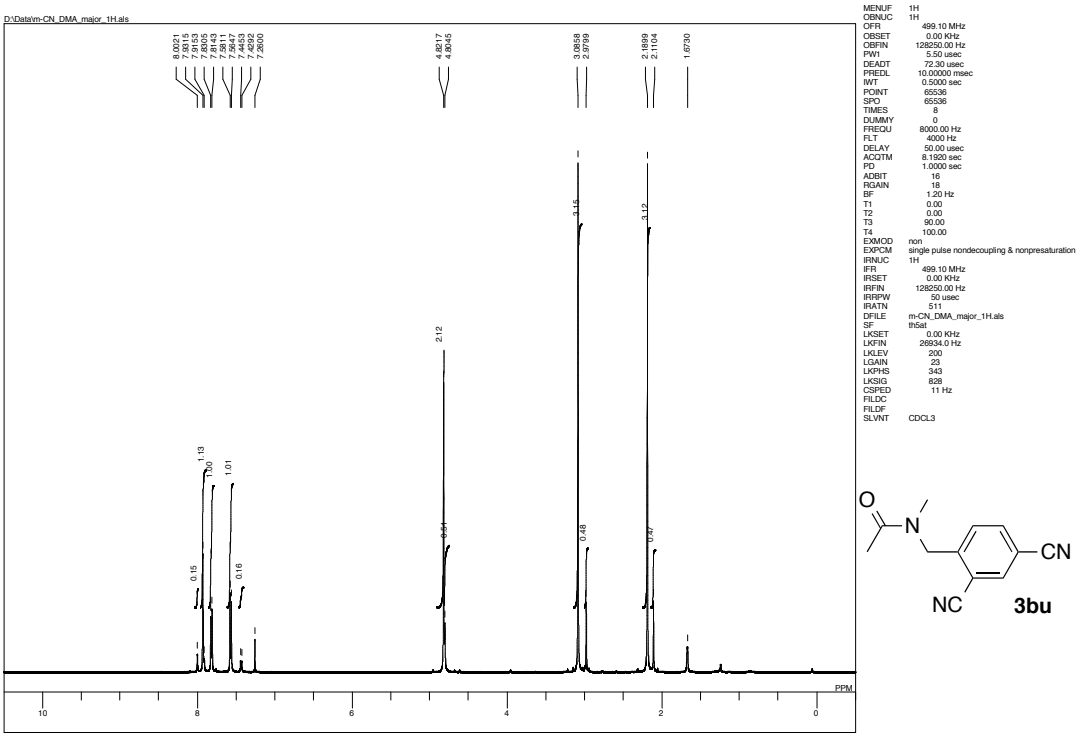
3'au

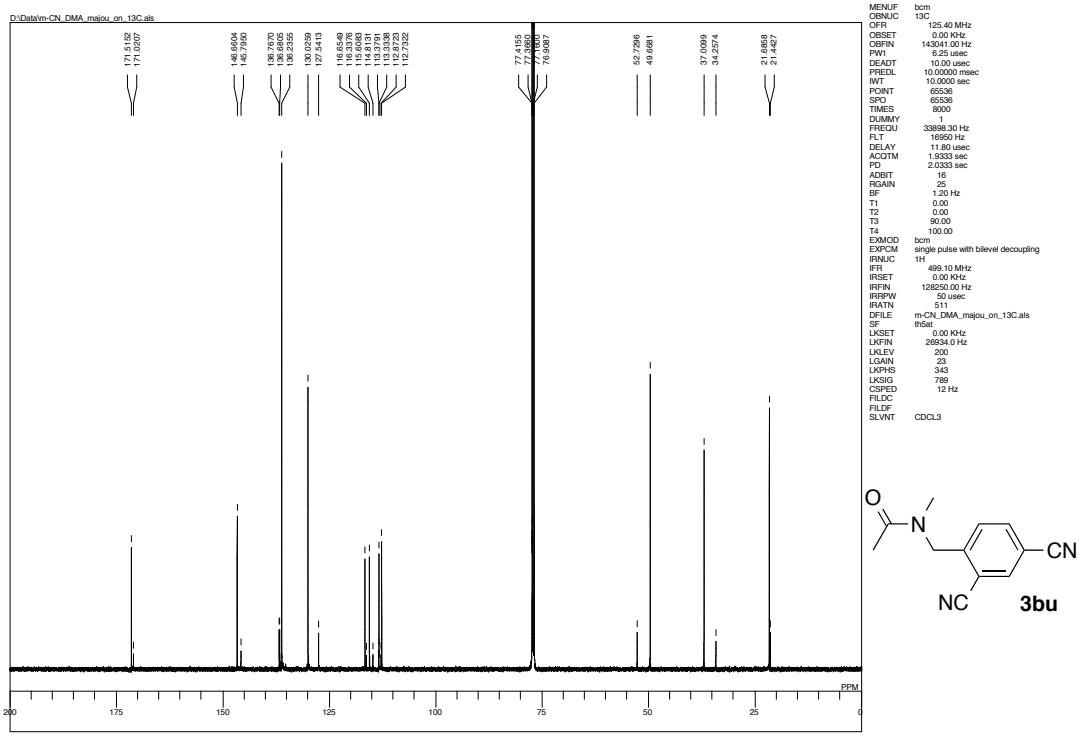


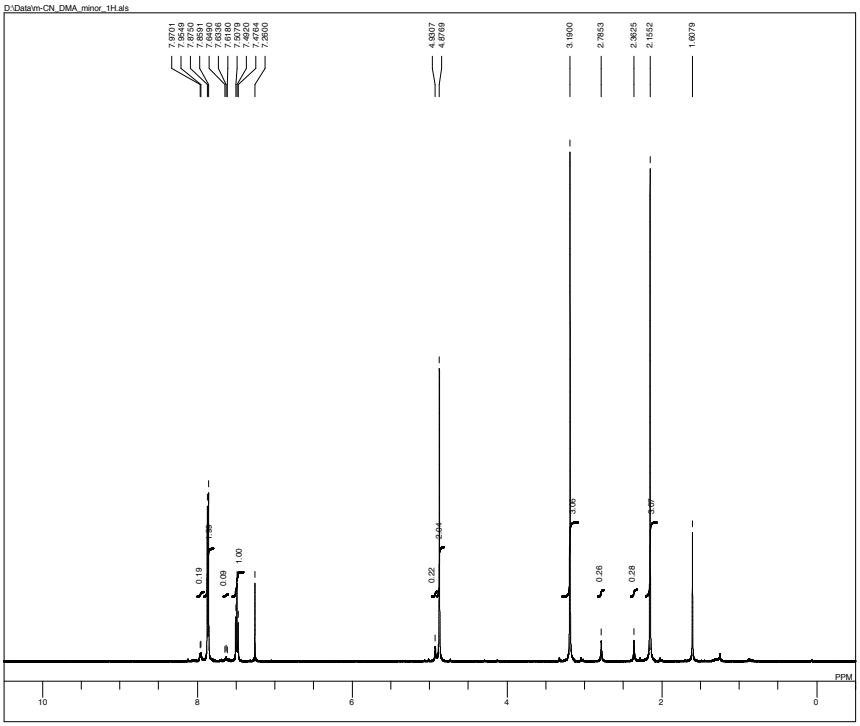

```
MENUF    13C
OBNUC    13C
OFR        125.40 MHz
OBSET        0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL      0.01000 msec
IWT       1000.0000 sec
POINT       65536
SPO         65536
TIMES        9000
DUMMY           2
FREQU       33898.30 Hz
FLT         16950 Hz
DELAY        11.80 usec
ACQTM        1.9333 sec
PD           1.0670 sec
ADBIT          16
RGAIN          25
BF           0.12 Hz
T1           0.00
T2           0.00
T3          90.00
T4         100.00
EXMOD    bcm
EXPCM    single pulse with bilevel decoupling
IRNUC    1H
IFR        499.10 MHz
IRSET        0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN         511
DFILE    m-dicyano_TMU_minor_13C.als
SF       th5at
LKSET        0.00 KHz
LKFIN       26934.0 Hz
LKLEV         200
LGAIN          23
LKPHS         343
LKSIG         651
CSPED          12 Hz
FILDC
FILDF
SLVNT    CDCL3
```



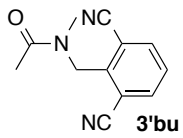
3'au


D:\Data\m-CN_DMA_major_1H.als
8.0221
8.0113
7.9153
7.8005
7.7840
7.5811
7.5647
7.4655
7.4493
7.2800
4.8517
4.8415
3.0858
2.9799
2.1899
2.1104
1.6730
3.15
3.12
2.12
1.13
1.00
1.01
0.15
0.16
0.51
0.48
0.47
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 18
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRPPW 50 usec
IRATN 511
DFILE m-CN_DMA_major_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 828
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3
O
N
CN
NC
3bu

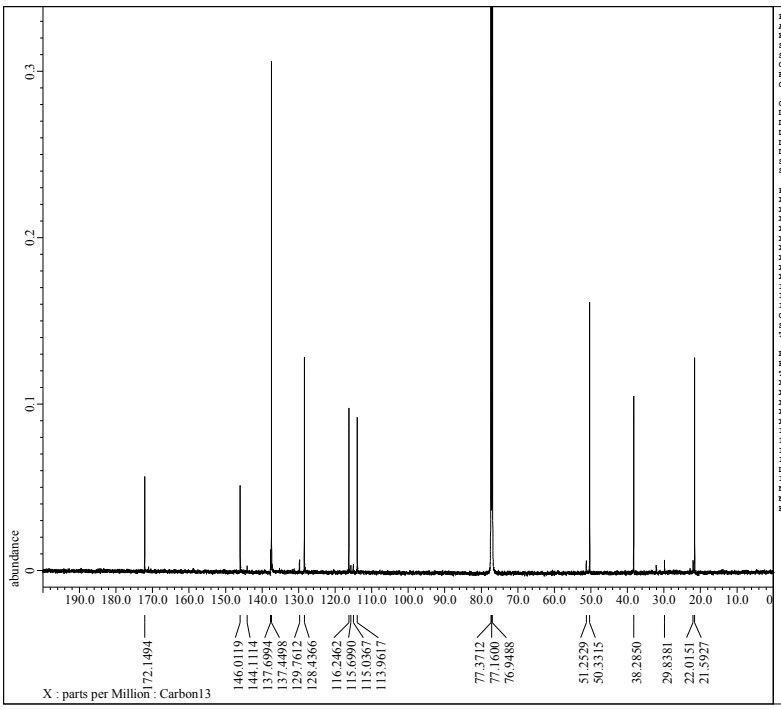

D:\Data\m-CN_DMA_majou_on_13C.als
171.5192
171.0207
146.6604
145.7990
136.7670
136.6805
136.2355
130.0259
127.5413
116.6549
116.3376
115.6668
114.8131
113.3791
113.2158
112.8723
112.7322
77.4155
77.1980
77.1650
76.9087
52.7296
49.6681
37.0099
34.2574
21.6858
21.4427
PPM
200
175
150
125
100
75
50
25
0
MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 10.00000 msec
IWT 10.0000 sec
POINT 65536
SPO 65536
TIMES 8000
DUMMY 1
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 2.0333 sec
ADBIT 16
RGAIN 25
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRPPW 50 usec
IRATN 511
DFILE m-CN_DMA_majou_on_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 789
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
O
N
CN
NC
3bu




```
MENUF     1H
GBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN       128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL        10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU        8000.00 Hz
FLT          4000 Hz
DELAY         50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT           16
RGAIN           21
BF            1.20 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN       128250.00 Hz
IRPW          50 usec
IRATN          511
DFILE     m-CN_DMA_minor_1H.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           908
CSPED         14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



3'bu

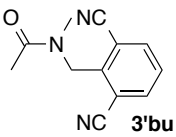



```
Filename          = m-CN_DMA_minor_13C_Carbon-
Author            = delta
Experiment        = carbon.jxp
Sample_Id         = m-CN_DMA_minor_13C
Solvent           = CHLOROFORM-D
Creation_Time     = 25-MAR-2014 23:59:59
Revision_Time     = 26-MAR-2014 18:32:16
Current_Time      = 28-APR-2014 12:06:55

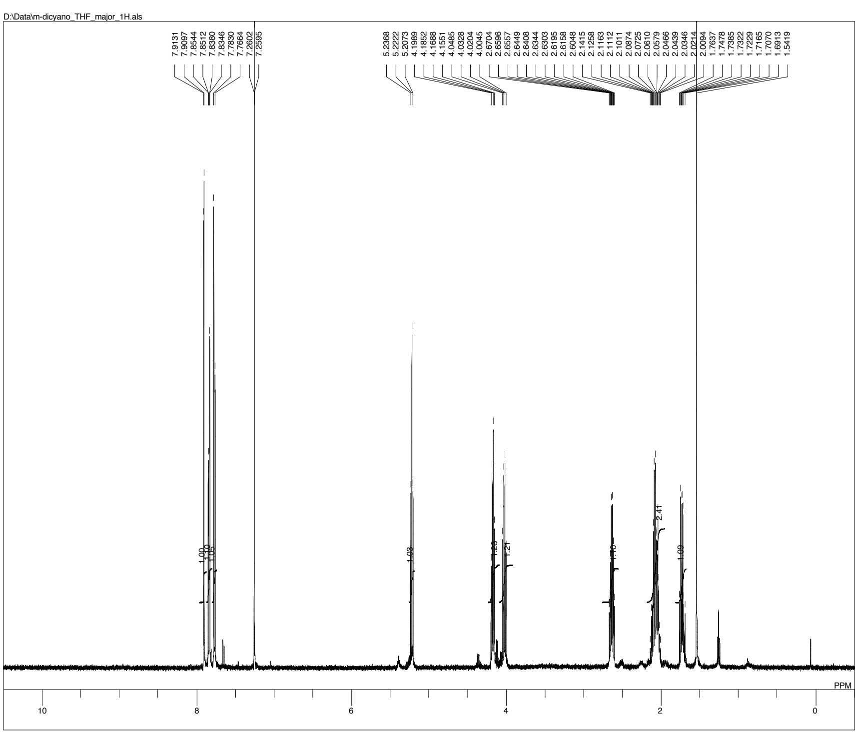
Comment           = single pulse decoupled gat
Data_Format       = 1D COMPLEX
Dim_Size          = 26214
Dim_Title         = Carbon13
Dim_Units         = [ppm]
Dimensions        = X
Site              = JNM-ECA600
Spectrometer      = DELTA2_NMR

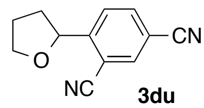
Field_Strength    = 13.95540559[T] (590[MHz])
X_Acq_Duration    = 0.69730304[s]
X_Domain          = 13C
X_Freq            = 149.40429612[MHz]
X_Offset          = 100.0[ppm]
X_Points          = 32768
X_Prescans        = 4
X_Resolution      = 1.43409672[Hz]
X_Sweep           = 46.9924812[kHz]
X_Sweep_Clipped   = 37.59398496[kHz]
Irr_Domain        = Proton
Irr_Freq          = 594.17058168[MHz]
Irr_Offset        = 5[ppm]
Clipped           = TRUE
Scans             = 11500
Total_Scans       = 11500

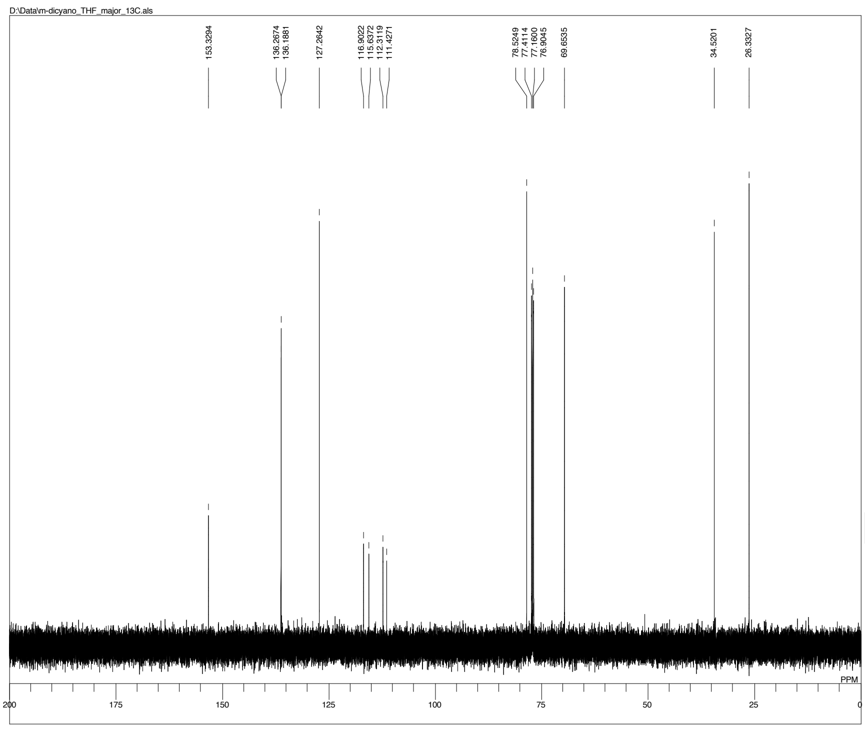
Relaxation_Delay  = 2[s]
Recvr_Gain        = 56
Temp_Get          = 24.9[dC]
X_90_Width        = 9[us]
X_Acq_Time        = 0.69730304[s]
X_Angle           = 30[deg]
X_Atn             = 6[dB]
X_Pulse           = 3[us]
Irr_Atn_Dec       = 17.8[dB]
Irr_Atn_Noe       = 17.8[dB]
Irr_Noise         = WALTZ
Irr_Pwidth        = 76[us]
Decoupling        = TRUE
Initial_Wait      = 1[s]
Noe               = TRUE
Noe_Time          = 2[s]
Repetition_Time   = 2.69730304[s]
```

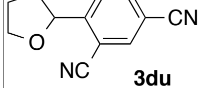


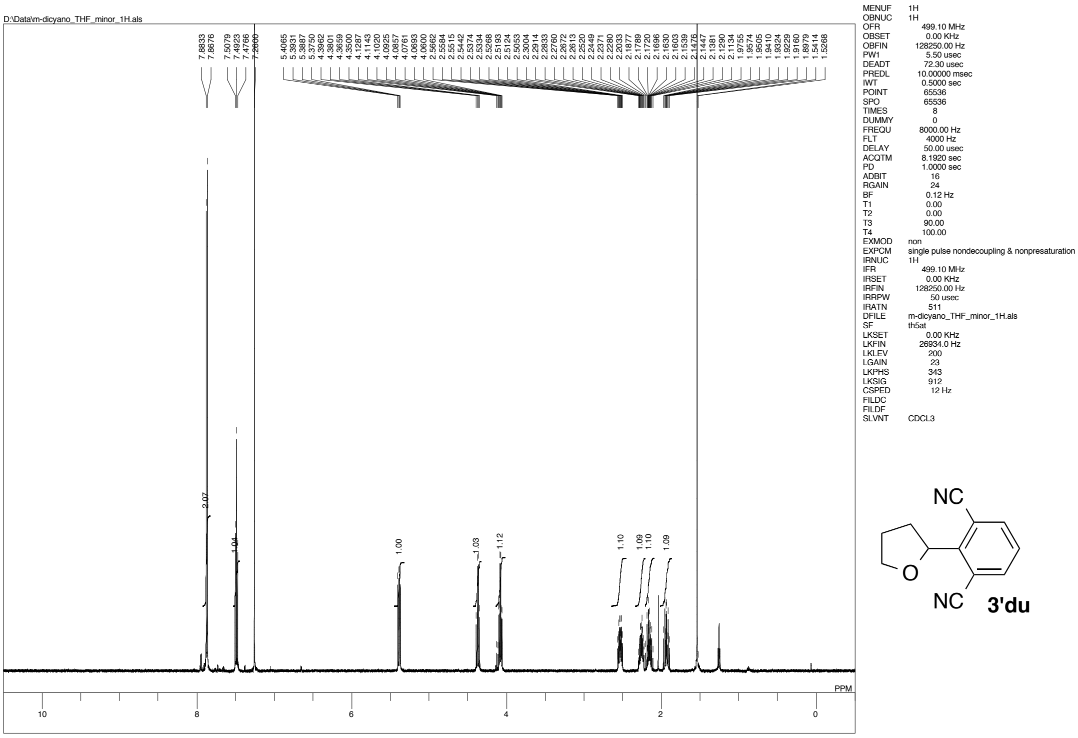
3'bu


D:\Data\m-dicyano_THF_major_1H.als
7.9131 7.9097 7.8544 7.8512 7.8380 7.8346 7.7820 7.7664 7.2602 7.2595
5.2368 5.2222 5.2073 4.1989 4.1852 4.1698 4.1551 4.0485 4.0328 4.0204 4.0045 2.6704 2.6596 2.6557 2.6449 2.6408 2.6344 2.6303 2.6195 2.6158 2.6048 2.1415 2.1258 2.1163 2.1112 2.1011 2.0874 2.0725 2.0610 2.0579 2.0466 2.0439 2.0346 2.0214 2.0094 1.7837 1.7478 1.7385 1.7322 1.7229 1.7165 1.7070 1.6913 1.5419
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE m-dicyano_THF_major_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 927
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3

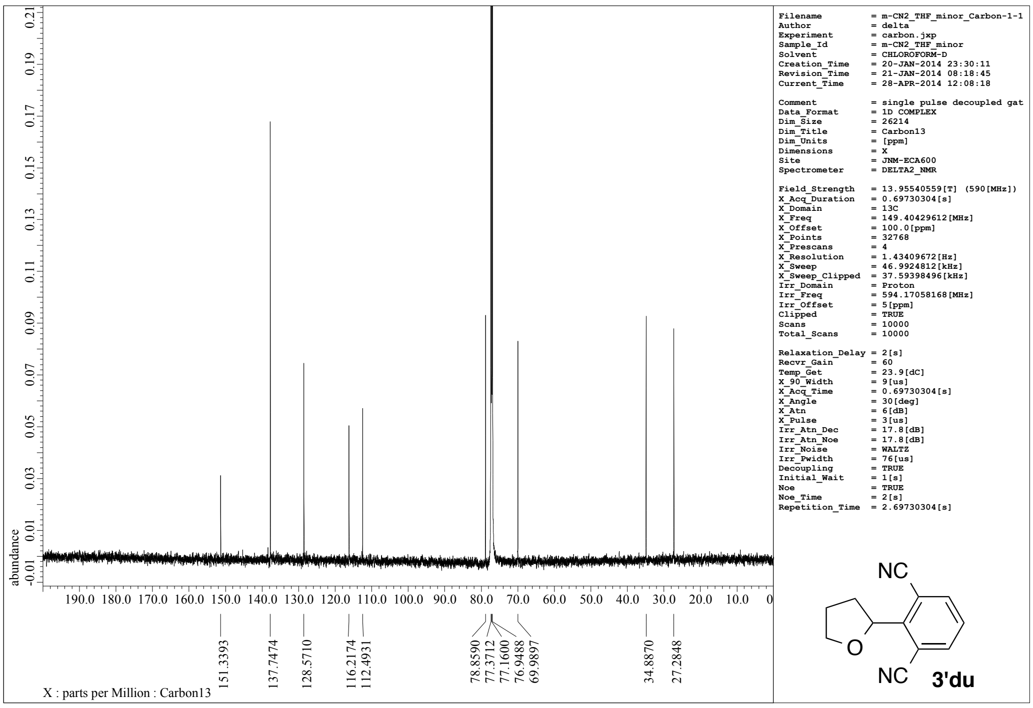

CN
NC
O
3du


D:\Data\m-dicyano_THF_major_13C.als
153.3294
136.2674 136.1881
127.2642
116.9022 115.6372 112.3119 111.4271
78.5249 77.4114 77.1600 76.9045
69.6535
34.5201
26.3327
PPM
200
175
150
125
100
75
50
25
0
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 262144
SPO 65536
TIMES 512
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE m-dicyano_THF_major_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 876
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3

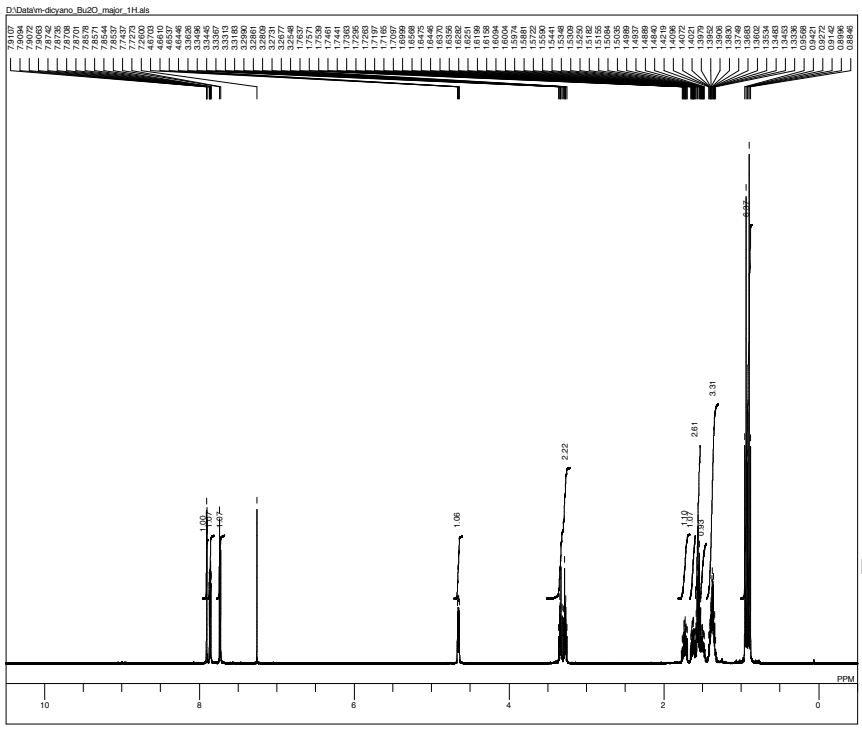

CN
NC
O
3du



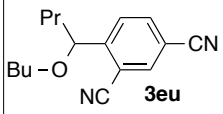
D:\Data\m-dicyano_THF_minor_1H.als
7.8833 7.8676 7.5079 7.4923 7.4766 7.2600
5.4065 5.3931 5.3887 5.3750 4.3962 4.3801 4.3659 4.3500 4.1287 4.1143 4.1020 4.0925 4.0857 4.0781 4.0693 4.0600 2.5662 2.5584 2.5515 2.5442 2.5374 2.5334 2.5268 2.5193 2.5124 2.5053 2.3004 2.2914 2.2833 2.2760 2.2672 2.2613 2.2520 2.2449 2.2371 2.2280 2.2033 2.1977 2.1789 2.1720 2.1696 2.1630 2.1603 2.1539 2.1476 2.1447 2.1381 2.1290 2.1134 1.9755 1.9574 1.9505 1.9410 1.9324 1.9229 1.9160 1.8979 1.5414 1.5268
2.07 1.04 1.00 1.03 1.12 1.10 1.09 1.10 1.09
PPM
10 8 6 4 2 0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 24
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE m-dicyano_THF_minor_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 912
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
NC
O
NC
3'du

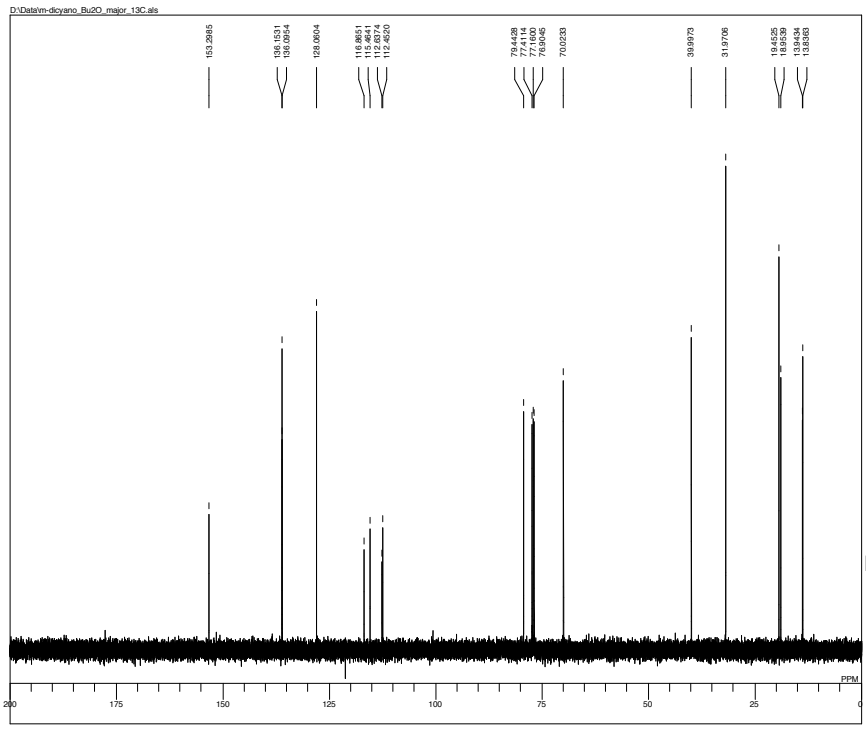


abundance
0.21 0.19 0.17 0.15 0.13 0.11 0.09 0.07 0.05 0.03 0.01 -0.01
190.0 180.0 170.0 160.0 150.0 140.0 130.0 120.0 110.0 100.0 90.0 80.0 70.0 60.0 50.0 40.0 30.0 20.0 10.0 0
151.3393
137.7474
128.5710
116.2174
112.4931
78.8590
77.3712
77.1600
76.9488
69.9897
34.8870
27.2848
X : parts per Million : Carbon13
Filename = m-CN2_THF_minor_Carbon-1-1
Author = delta
Experiment = carbon.jxp
Sample_Id = m-CN2_THF_minor
Solvent = CHLOROFORM-D
Creation_Time = 20-JAN-2014 23:30:11
Revision_Time = 21-JAN-2014 08:18:45
Current_Time = 28-APR-2014 12:08:18
Comment = single pulse decoupled gat
Data_Format = 1D COMPLEX
Dim_Size = 26214
Dim_Title = Carbon13
Dim_Units = [ppm]
Dimensions = X
Site = JNM-ECA600
Spectrometer = DELTA2_NMR
Field_Strength = 13.95540559[T] (590[MHz])
X_Acq_Duration = 0.69730304[s]
X_Domain = 13C
X_Freq = 149.40429612[MHz]
X_Offset = 100.0[ppm]
X_Points = 32768
X_Prescans = 4
X_Resolution = 1.43409672[Hz]
X_Sweep = 46.9924812[kHz]
X_Sweep_Clipped = 37.59398496[kHz]
Irr_Domain = Proton
Irr_Freq = 594.17058168[MHz]
Irr_Offset = 5[ppm]
Clipped = TRUE
Scans = 10000
Total_Scans = 10000
Relaxation_Delay = 2[s]
Recvr_Gain = 60
Temp_Get = 23.9[dC]
X_90_Width = 9[us]
X_Acq_Time = 0.69730304[s]
X_Angle = 30[deg]
X_Atn = 6[dB]
X_Pulse = 3[us]
Irr_Atn_Dec = 17.8[dB]
Irr_Atn_Noe = 17.8[dB]
Irr_Noise = WALTZ
Irr_Pwidth = 76[us]
Decoupling = TRUE
Initial_Wait = 1[s]
Noe = TRUE
Noe_Time = 2[s]
Repetition_Time = 2.69730304[s]
NC
O
NC
3'du

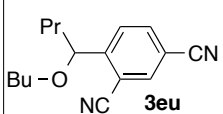


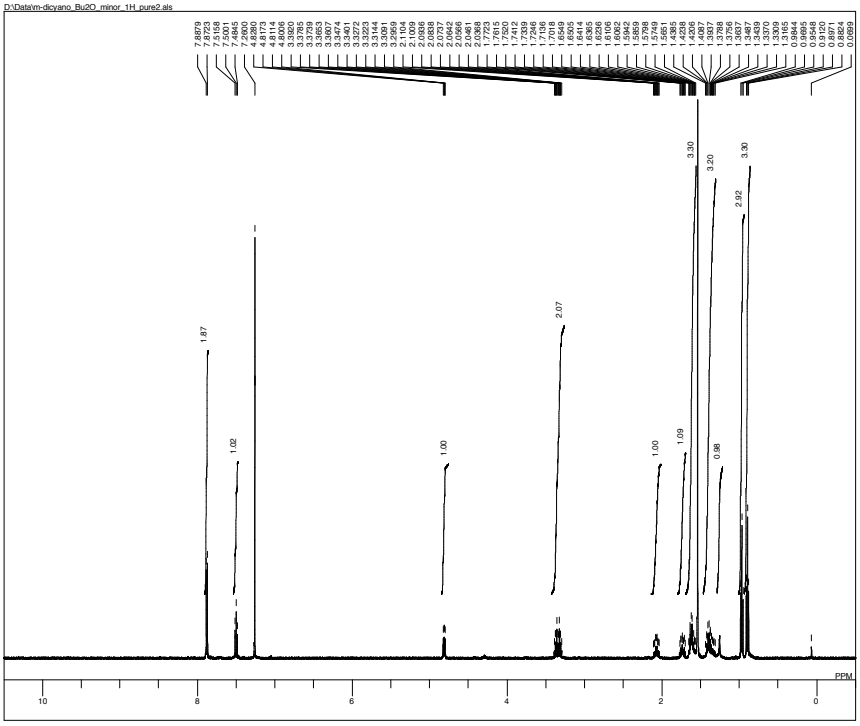

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU      8000.00 Hz
FLT          4000 Hz
DELAY         50.00 usec
ACQTM      8.1920 sec
PD            1.0000 sec
ADBIT            16
RGAIN            14
BF            0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN            511
DFILE     m-dicyano_Bu2O_major_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG           1015
CSPED          13 Hz
FILDC
FILDF
SLVNT     CDCL3
```



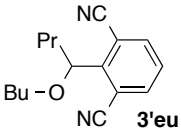



```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     0.01000 msec
IWT       1000.0000 sec
POINT        65536
SPO          65536
TIMES          256
DUMMY            2
FREQU      33898.30 Hz
FLT          16950 Hz
DELAY         11.80 usec
ACQTM      1.9333 sec
PD            1.0670 sec
ADBIT            16
RGAIN            25
BF            0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN            511
DFILE     m-dicyano_Bu2O_major_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV            200
LGAIN             25
LKPHS            343
LKSIG           1437
CSPED          12 Hz
FILDC
FILDF
SLVNT     CDCL3
```

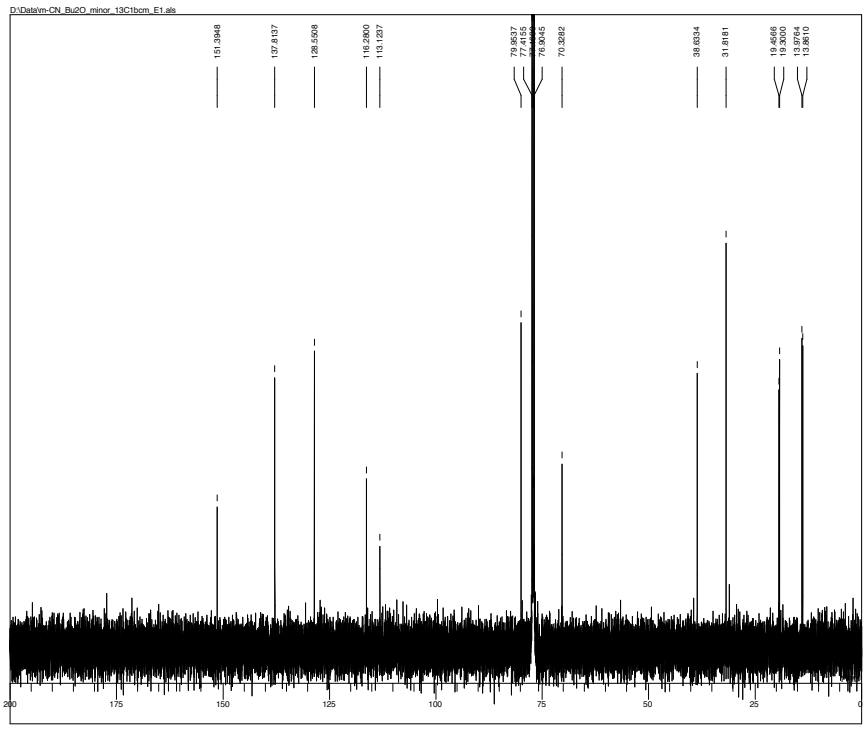




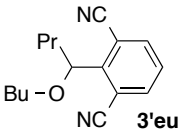

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN    128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT         0.5000 sec
POINT        65536
SPO          65536
TIMES           24
DUMMY            0
FREQU         8000.00 Hz
FLT           4000 Hz
DELAY          50.00 usec
ACQTM        8.1920 sec
PD           1.0000 sec
ADBIT            16
RGAIN            25
BF             0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN    128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     m-dicyano_Bu2O_minor_1H_pure2.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            837
CSPED          12 Hz
FILDC
FILDF
SLVNT     CDCL3
```



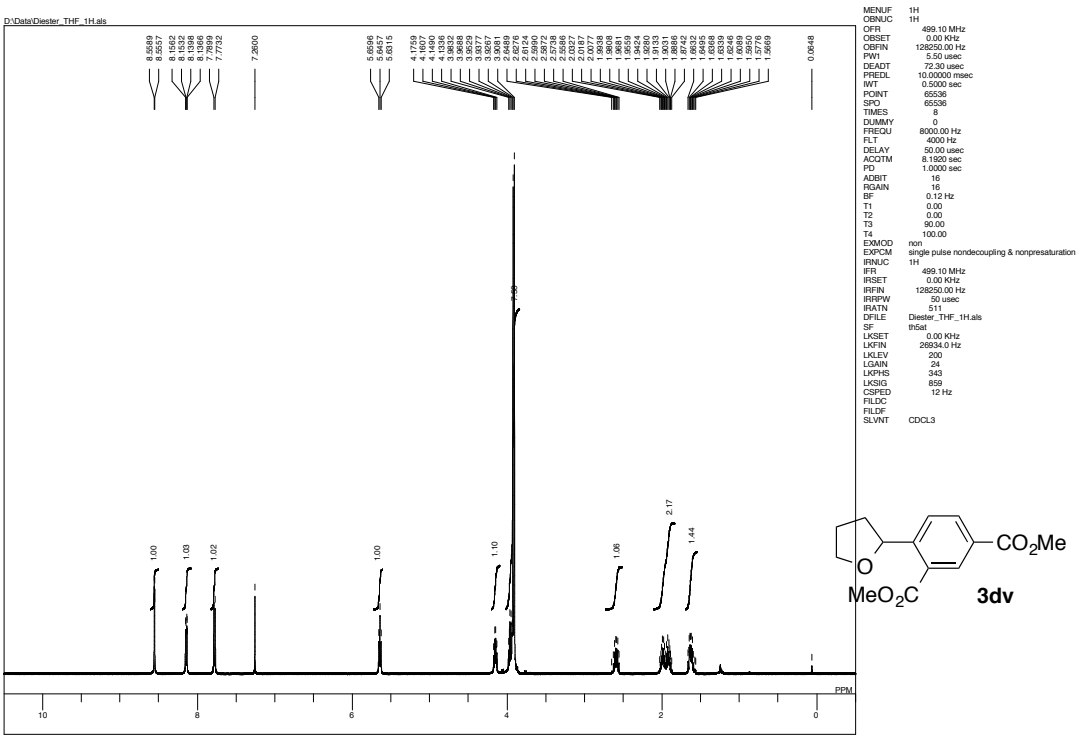
3'eu

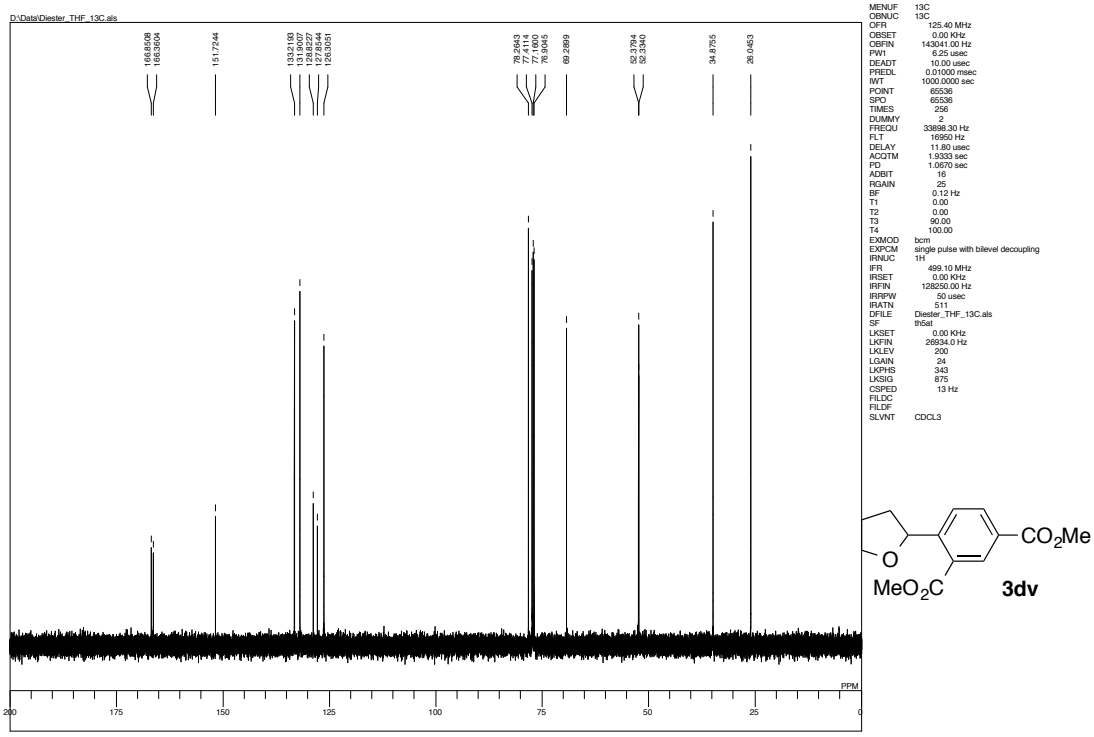


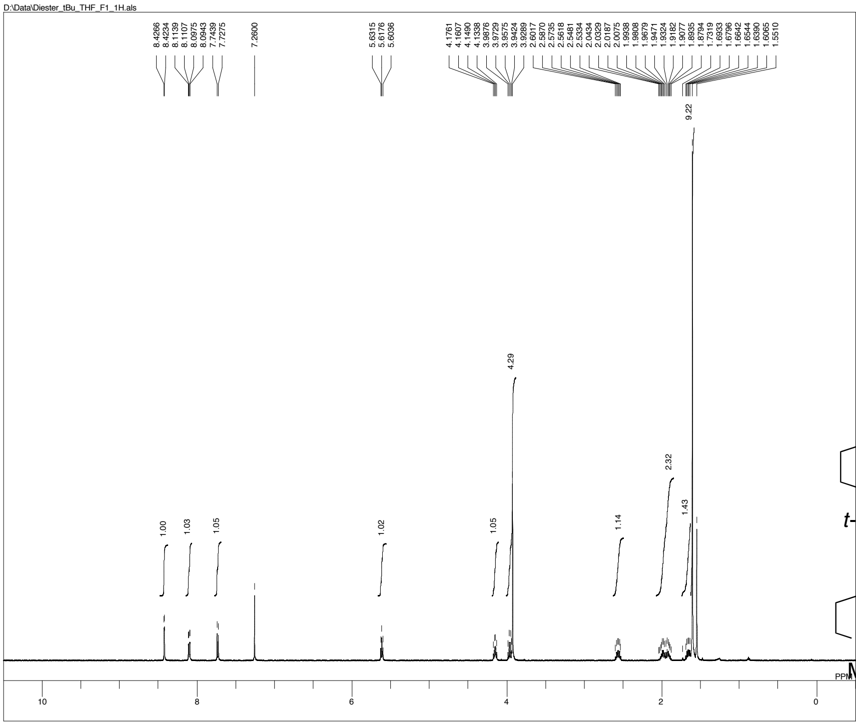

```
MENUF     bcm
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN    143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     10.00000 msec
IWT         10.0000 sec
POINT        32768
SPO          32768
TIMES        10000
DUMMY            1
FREQU        33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM        0.9667 sec
PD           2.0333 sec
ADBIT            16
RGAIN            26
BF             0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN    128250.00 Hz
IRRPW          50 usec
IRATN           511
DFILE     m-CN_Bu2O_minor_13C1bcm_E1.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            710
CSPED          14 Hz
FILDC
FILDF
SLVNT     CDCL3
```

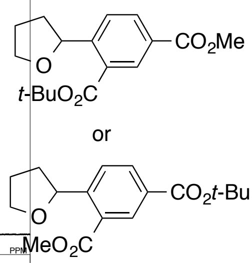


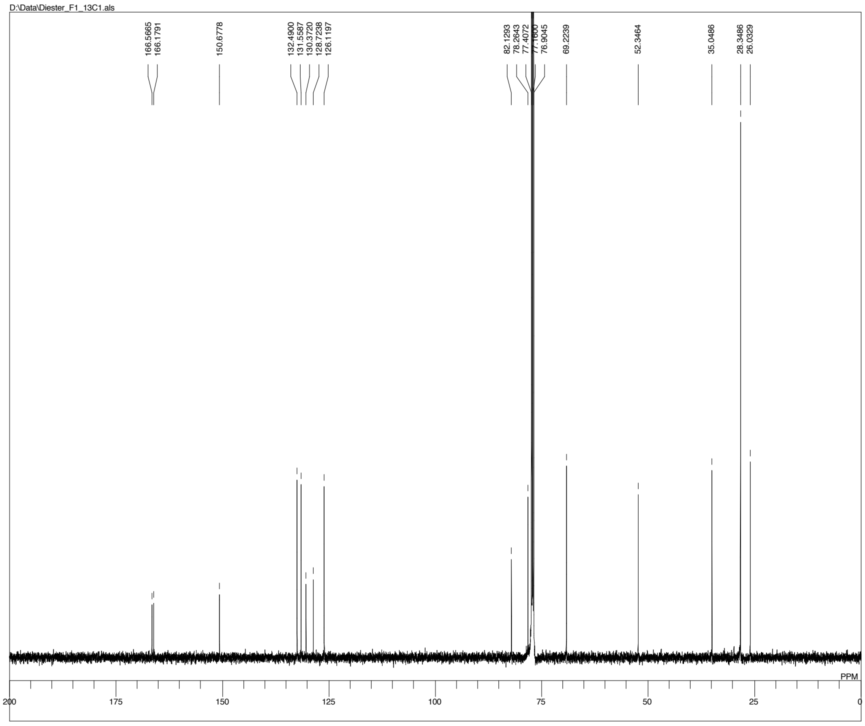
3'eu

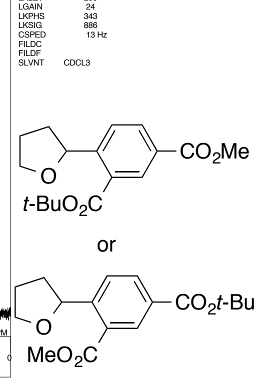

D:\Data\Diester_THF_1H.als
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 16
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Diester_THF_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 24
LKPHS 343
LKSIG 859
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3
PPM
CO2Me
O
MeO2C
3dv

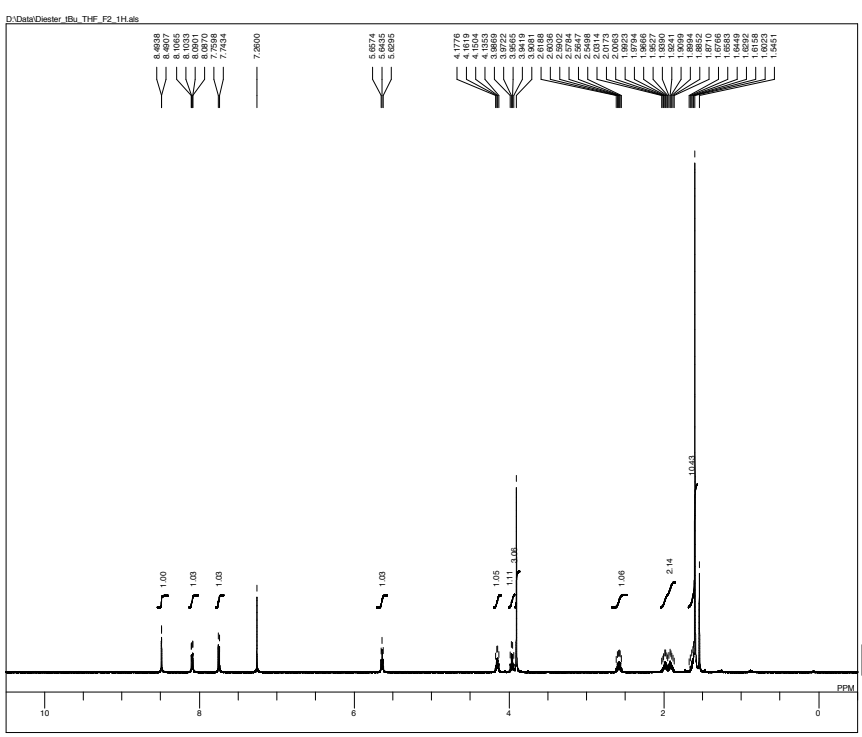

D:\Data\Diester_THF_13C.als
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Diester_THF_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 24
LKPHS 343
LKSIG 875
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3
PPM
CO2Me
O
MeO2C
3dv


D:\Data\Diester_tBu_THF_F1_1H.als
8.4266 8.4234 8.1139 8.1107 8.0975 8.0943 7.7439 7.7275 7.2600 5.6315 5.6176 5.6036 4.1761 4.1607 4.1490 4.1338 3.9876 3.9729 3.9575 3.9424 3.9289 2.6017 2.5870 2.5735 2.5618 2.5481 2.5334 2.0434 2.0329 2.0187 2.0075 1.9938 1.9808 1.9679 1.9471 1.9324 1.9182 1.9077 1.8935 1.8794 1.7319 1.6933 1.6796 1.6642 1.6544 1.6390 1.6065 1.5510
1.00
1.03
1.05
1.02
1.05
4.29
1.14
2.32
1.43
9.22
PPM
10
8
6
4
2
0
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 22
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Diester_tBu_THF_F1_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 942
CSPED 14 Hz
FILDC
FILDF
SLVNT CDCL3

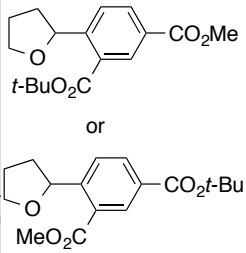

O
CO2Me
t-BuO2C
or
O
CO2t-Bu
MeO2C

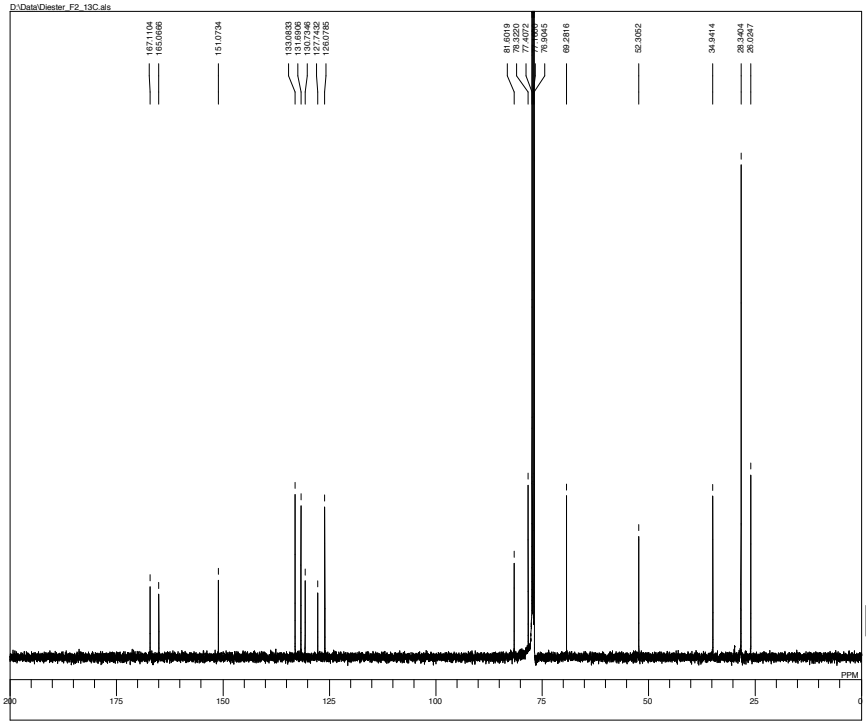

D:\Data\Diester_F1_13C1.als
166.5665 166.1791 150.6778 132.4900 131.5587 130.3720 128.7238 126.1197 82.1293 78.2643 77.4072 76.9045 69.2239 52.3464 35.0486 28.3486 26.0329
PPM
200
175
150
125
100
75
50
25
0
MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 10.00000 msec
IWT 10.0000 sec
POINT 32768
SPO 32768
TIMES 10000
DUMMY 1
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 2.0333 sec
ADBIT 16
RGAIN 25
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Diester_F1_13C1.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 24
LKPHS 343
LKSIG 886
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3


O
CO2Me
t-BuO2C
or
O
CO2t-Bu
MeO2C

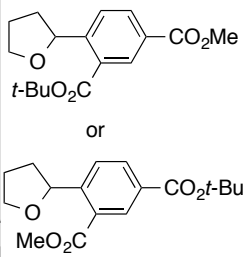


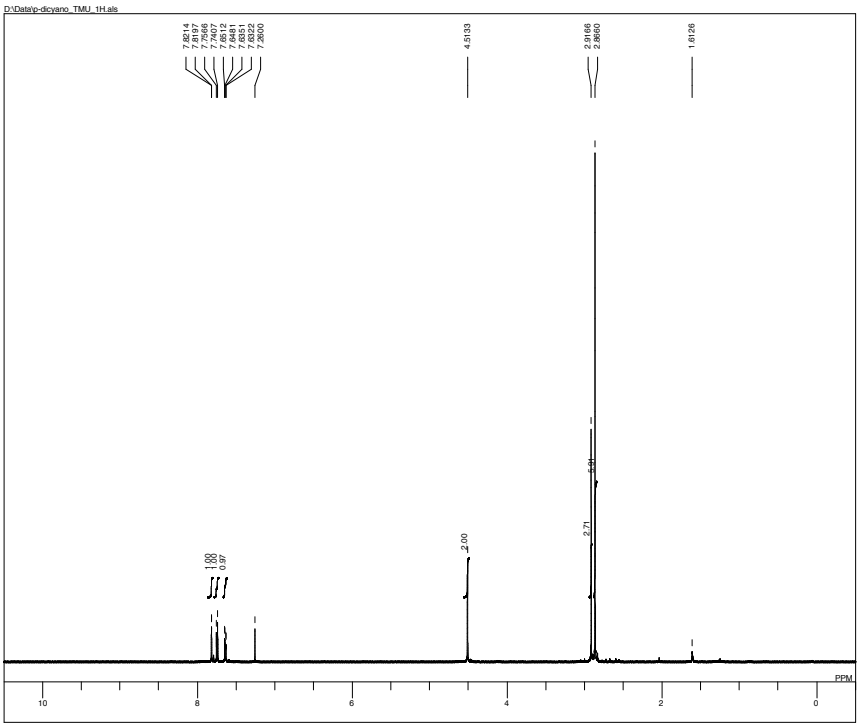
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 22
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Diester_tBu_THF_F2_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 973
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3



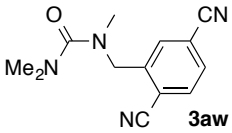


MENUF bcm
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 10.00000 msec
IWT 10.0000 sec
POINT 32768
SPO 32768
TIMES 9000
DUMMY 1
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 0.9667 sec
PD 2.0333 sec
ADBIT 16
RGAIN 25
BF 1.20 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE Diester_F2_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 345
LKSIG 867
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3

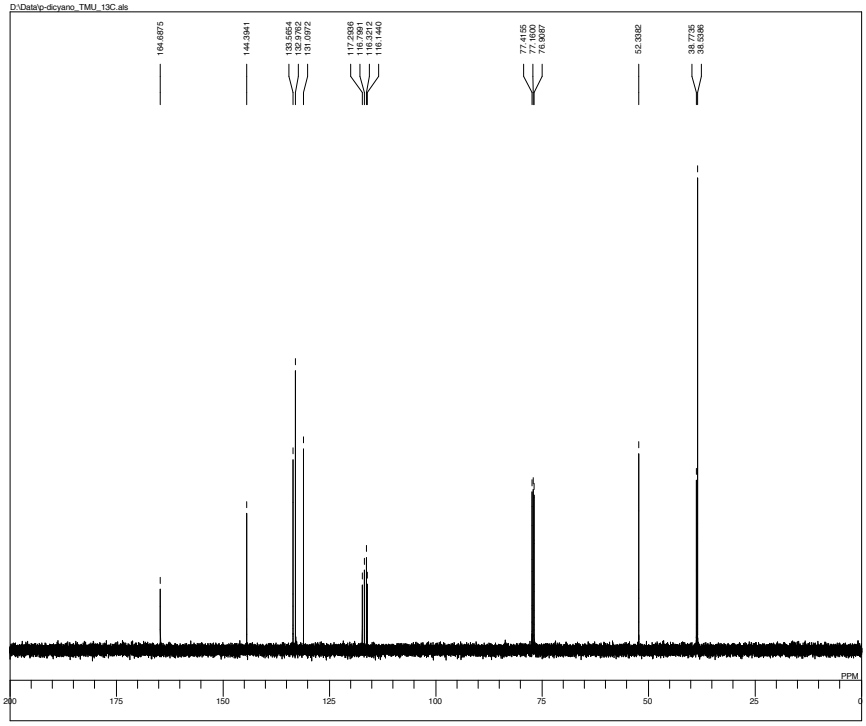




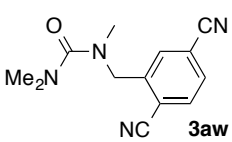
MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 18
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE p-dicyano_TMU_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 942
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3



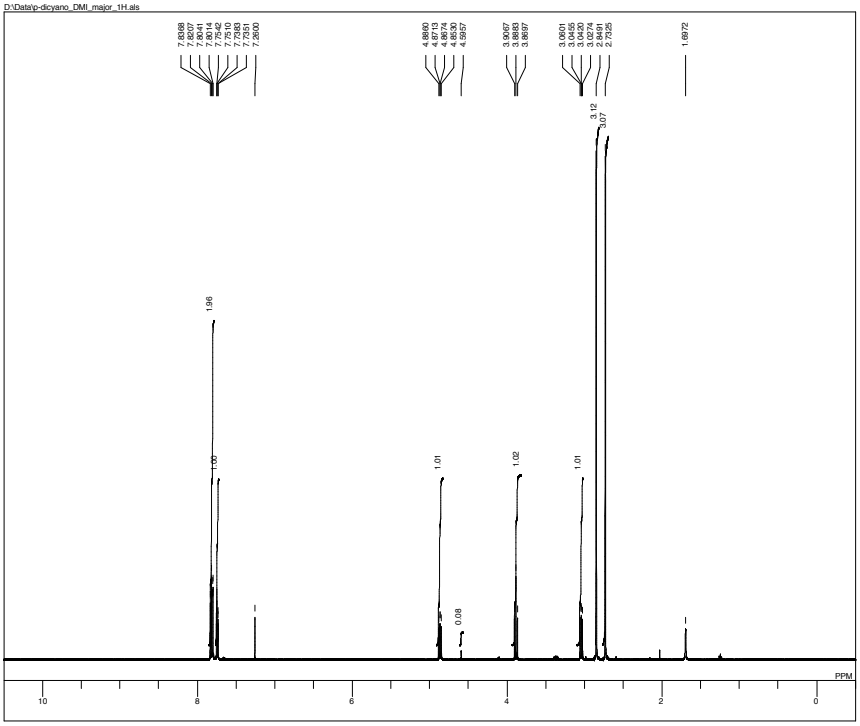
3aw



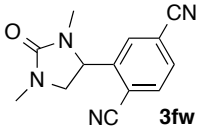
MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 256
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 1.0670 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE p-dicyano_TMU_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 24
LKPHS 343
LKSIG 842
CSPED 11 Hz
FILDC
FILDF
SLVNT CDCL3



3aw

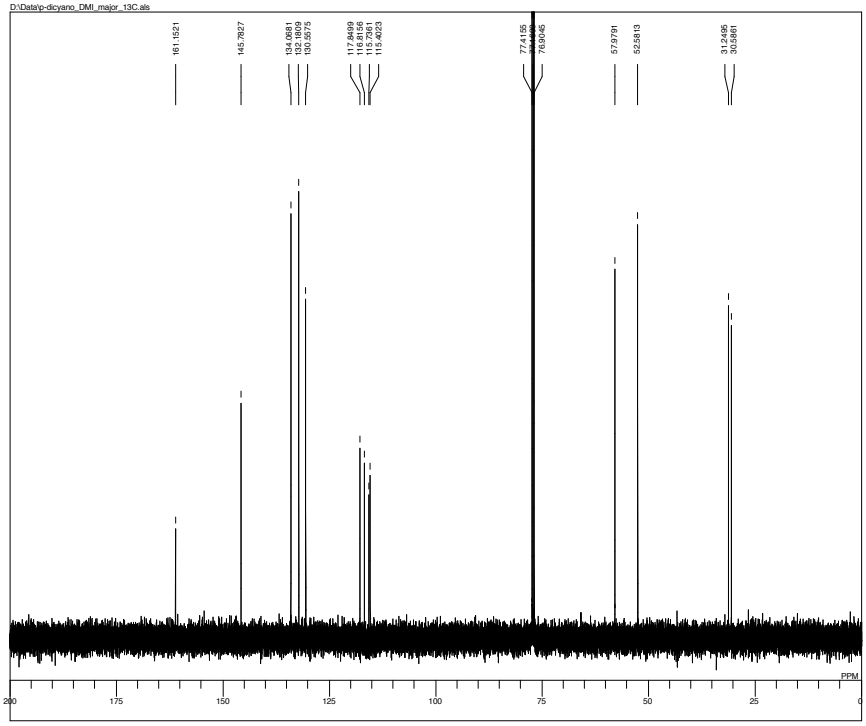


MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 18
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE p-dicyano_DMI_major_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 867
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3

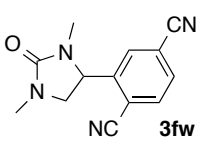


3fw

contains 4% of **3'fw**

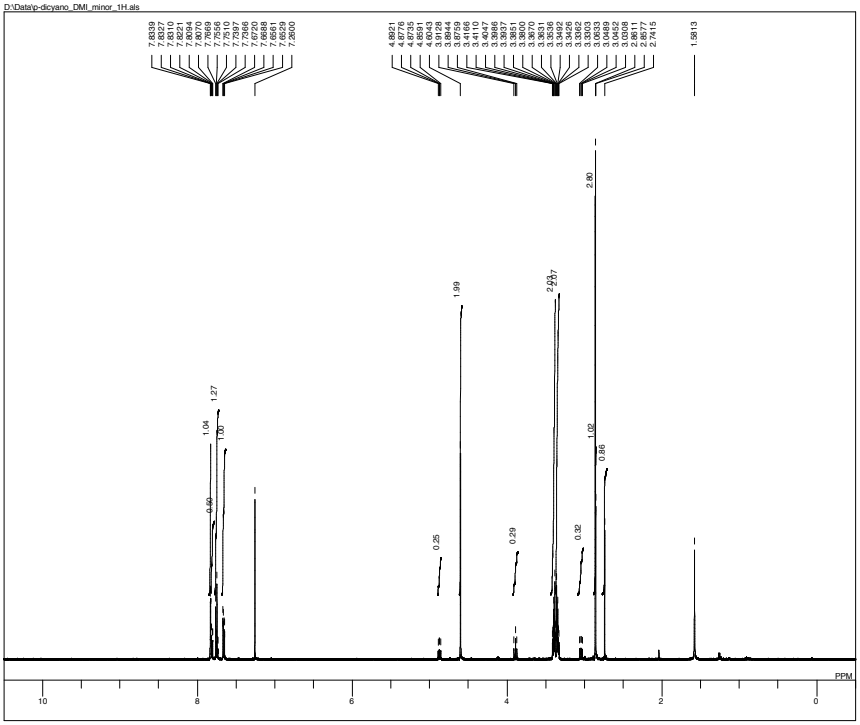


MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 512
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 2.0000 sec
ADBIT 16
RGAIN 25
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE p-dicyano_DMI_major_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 885
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3

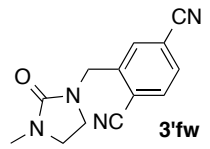


3fw

contains 4% of **3'fw**

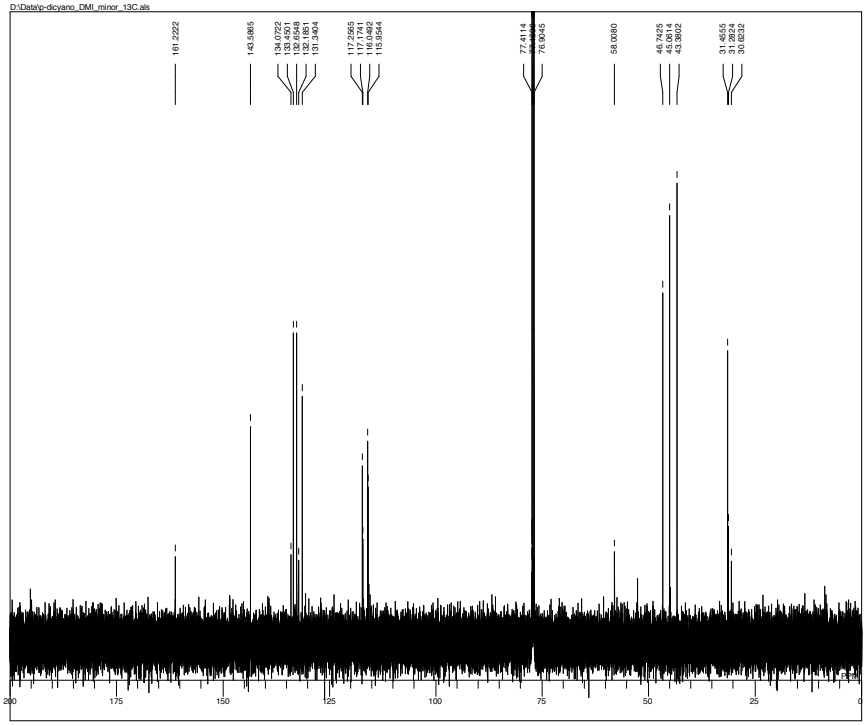


MENUF 1H
OBNUC 1H
OFR 499.10 MHz
OBSET 0.00 KHz
OBFIN 128250.00 Hz
PW1 5.50 usec
DEADT 72.30 usec
PREDL 10.00000 msec
IWT 0.5000 sec
POINT 65536
SPO 65536
TIMES 8
DUMMY 0
FREQU 8000.00 Hz
FLT 4000 Hz
DELAY 50.00 usec
ACQTM 8.1920 sec
PD 1.0000 sec
ADBIT 16
RGAIN 22
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD non
EXPCM single pulse nondecoupling & nonpresaturation
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE p-dicyano_DMI_minor_1H.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 851
CSPED 13 Hz
FILDC
FILDF
SLVNT CDCL3

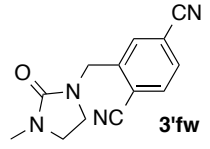


3'fw

contains 25% of **3fw**

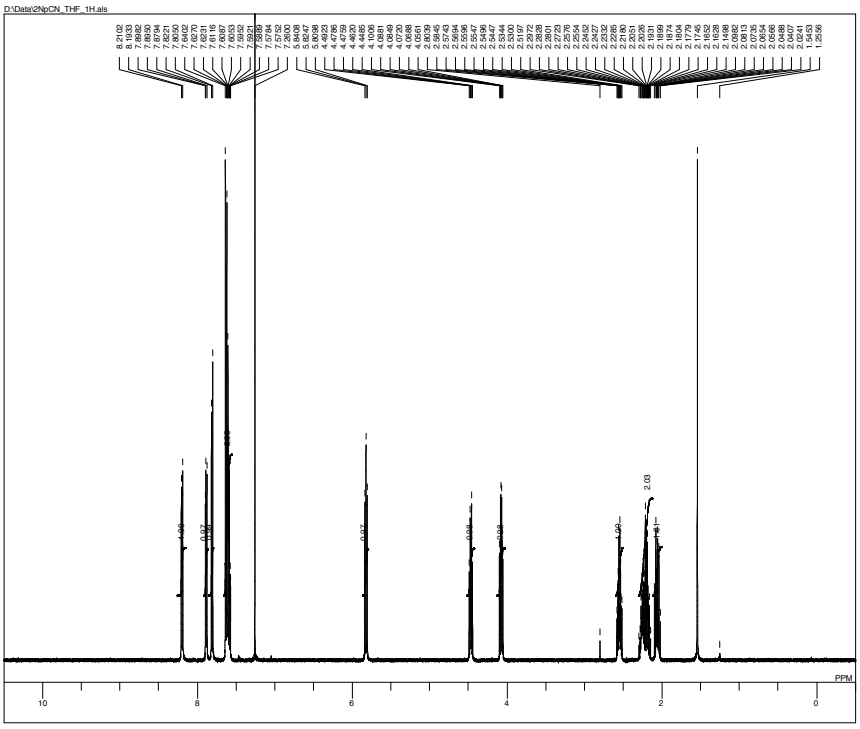


MENUF 13C
OBNUC 13C
OFR 125.40 MHz
OBSET 0.00 KHz
OBFIN 143041.00 Hz
PW1 6.25 usec
DEADT 10.00 usec
PREDL 0.01000 msec
IWT 1000.0000 sec
POINT 65536
SPO 65536
TIMES 1000
DUMMY 2
FREQU 33898.30 Hz
FLT 16950 Hz
DELAY 11.80 usec
ACQTM 1.9333 sec
PD 2.0000 sec
ADBIT 16
RGAIN 27
BF 0.12 Hz
T1 0.00
T2 0.00
T3 90.00
T4 100.00
EXMOD bcm
EXPCM single pulse with bilevel decoupling
IRNUC 1H
IFR 499.10 MHz
IRSET 0.00 KHz
IRFIN 128250.00 Hz
IRRPW 50 usec
IRATN 511
DFILE p-dicyano_DMI_minor_13C.als
SF th5at
LKSET 0.00 KHz
LKFIN 26934.0 Hz
LKLEV 200
LGAIN 23
LKPHS 343
LKSIG 802
CSPED 12 Hz
FILDC
FILDF
SLVNT CDCL3

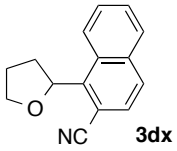


3'fw

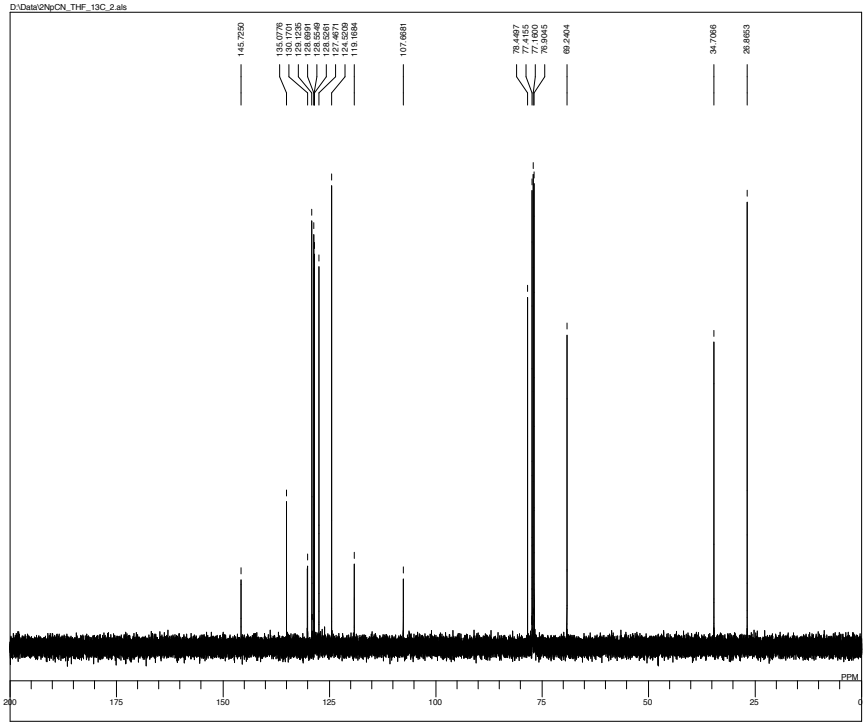
contains 25% of **3fw**



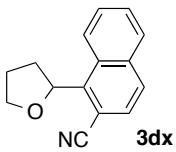

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU        8000.00 Hz
FLT          4000 Hz
DELAY         50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT            16
RGAIN            23
BF            0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRPPW         50 usec
IRATN            511
DFILE     2NpCN_THF_1H.als
SF        thSat
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            941
CSPED         13 Hz
FILDC
FILDF
SLVNT     CDCL3
```



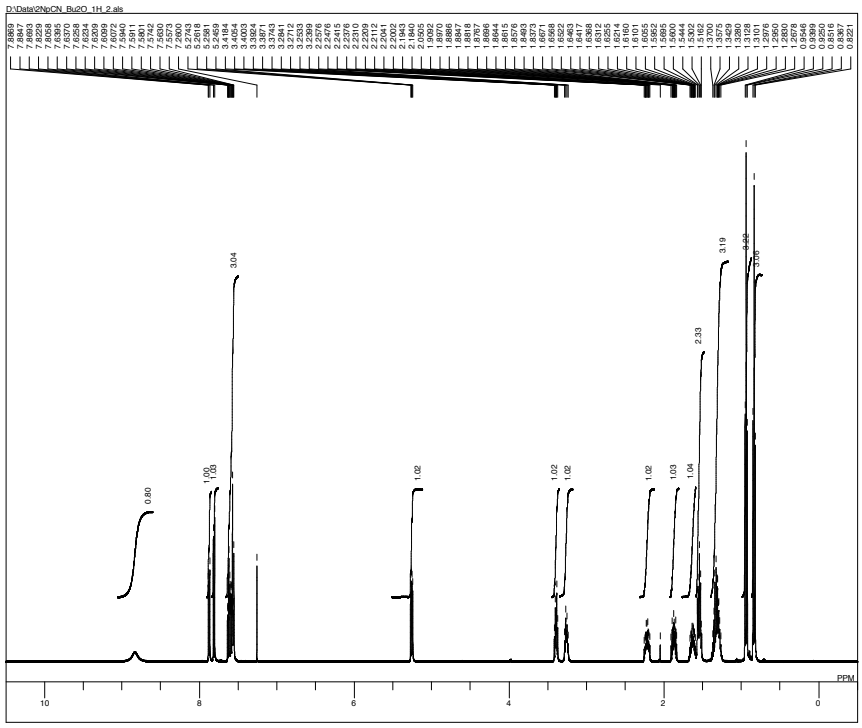
3dx



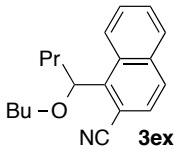

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     0.01000 msec
IWT       1000.0000 sec
POINT        65536
SPO          65536
TIMES          512
DUMMY            2
FREQU     33898.30 Hz
FLT          16950 Hz
DELAY         11.80 usec
ACQTM         1.9333 sec
PD            1.0670 sec
ADBIT            16
RGAIN            25
BF            0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRPPW         50 usec
IRATN            511
DFILE     2NpCN_THF_13C_2.als
SF        thSat
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV            200
LGAIN             23
LKPHS            343
LKSIG            890
CSPED         14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



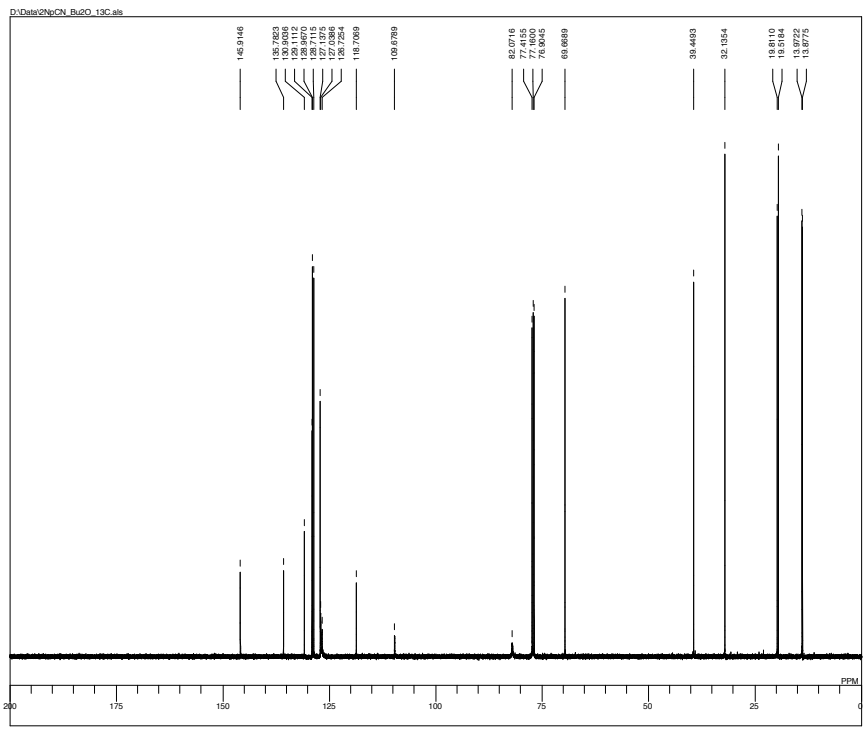
3dx



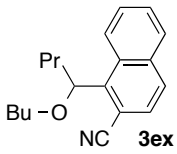

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN    128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU     8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM        8.1920 sec
PD           1.0000 sec
ADBIT           16
RGAIN           16
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN    128250.00 Hz
IRRPW         50 usec
IRATN          511
DFILE     2NpCN_Bu2O_1H_2.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          916
CSPED         11 Hz
FILDC
FILDF
SLVNT     CDCL3
```



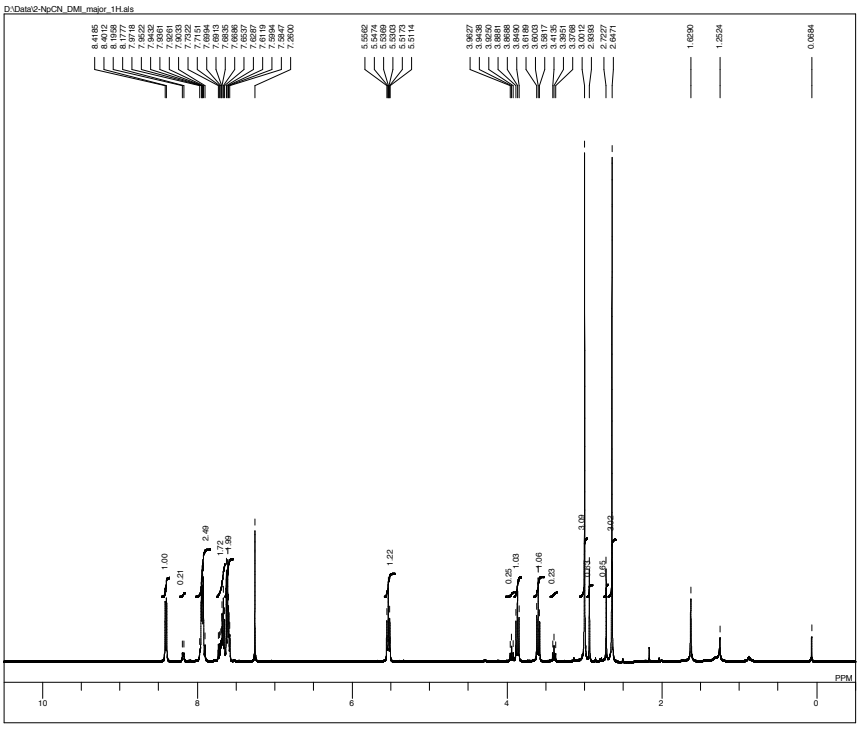
3ex



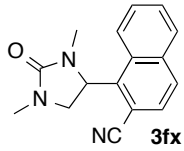

```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN    143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     0.01000 msec
IWT       1000.0000 sec
POINT        65536
SPO          65536
TIMES         6500
DUMMY            2
FREQU     33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM        1.9333 sec
PD           1.0670 sec
ADBIT           16
RGAIN           25
BF            0.12 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN    128250.00 Hz
IRRPW         50 usec
IRATN          511
DFILE     2NpCN_Bu2O_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          606
CSPED          5 Hz
FILDC
FILDF
SLVNT     CDCL3
```



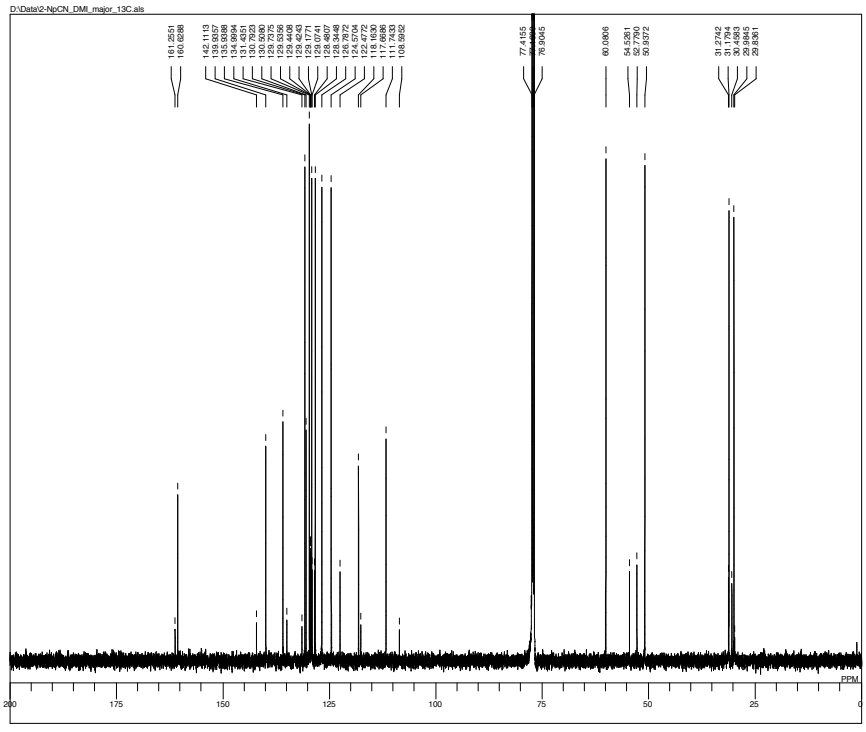
3ex



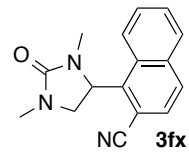

```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT          0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU        8000.00 Hz
FLT          4000 Hz
DELAY         50.00 usec
ACQTM         8.1920 sec
PD           1.0000 sec
ADBIT            16
RGAIN            20
BF            1.20 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN           511
DFILE     2-NpCN_DMI_major_1H.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           806
CSPED         14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



3fx




```
MENUF     bcm
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     10.00000 msec
IWT          10.0000 sec
POINT        65536
SPO          65536
TIMES         7500
DUMMY            1
FREQU        33898.30 Hz
FLT          16950 Hz
DELAY         11.80 usec
ACQTM         1.9333 sec
PD            2.0333 sec
ADBIT            16
RGAIN            27
BF            1.20 Hz
T1            0.00
T2            0.00
T3           90.00
T4          100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW         50 usec
IRATN           511
DFILE     2-NpCN_DMI_major_13C.als
SF        thSat
LKSET         0.00 KHz
LKFIN       26934.0 Hz
LKLEV           200
LGAIN            23
LKPHS           343
LKSIG           720
CSPED         10 Hz
FILDC
FILDF
SLVNT     CDCL3
```



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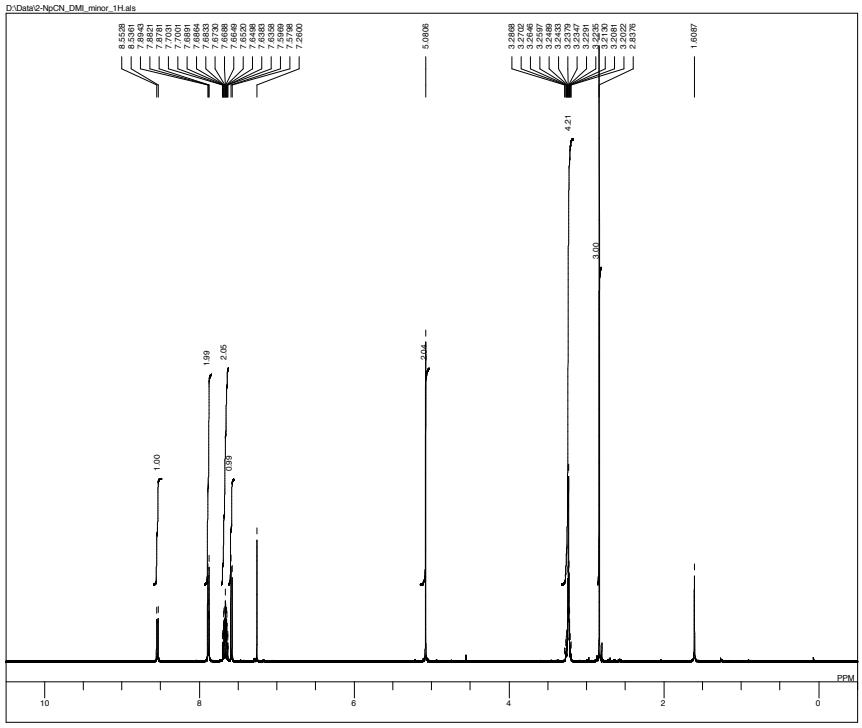
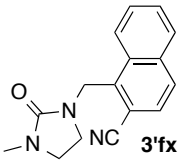



```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT           0.5000 sec
POINT        65536
SPO          65536
TIMES            8
DUMMY            0
FREQU         8000.00 Hz
FLT           4000 Hz
DELAY        50.00 usec
ACQTM         8.1920 sec
PD            1.0000 sec
ADBIT            16
RGAIN            20
BF             0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     non
EXPCM     single pulse nondecoupling & nonpresaturation
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW        50 usec
IRATN           511
DFILE     2-NpCN_DMI_minor_1H.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          924
CSPED           14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



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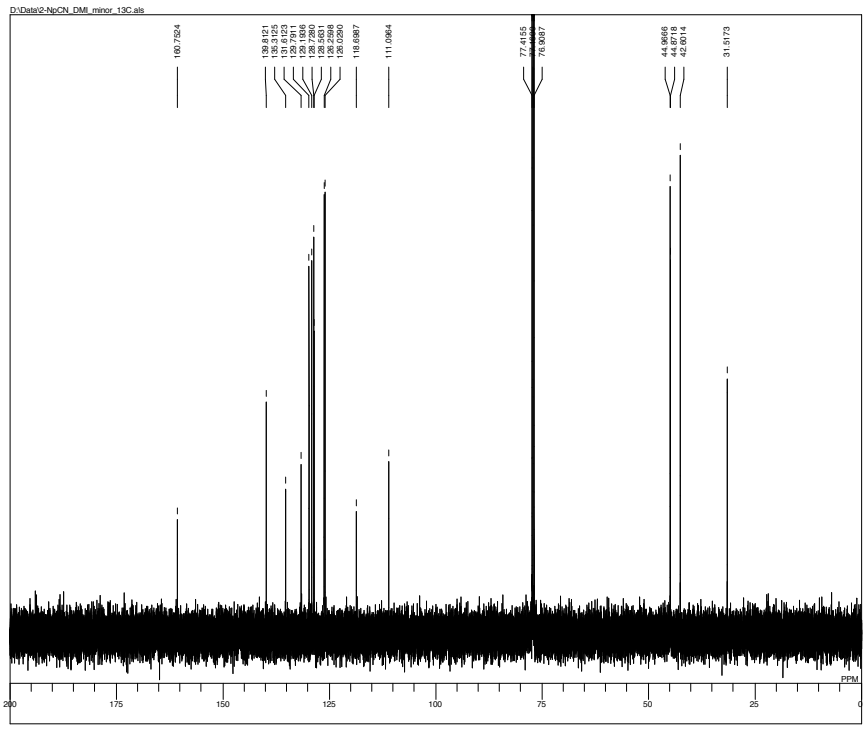
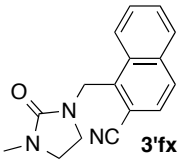



```
MENUF     13C
OBNUC     13C
OFR         125.40 MHz
OBSET         0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     0.01000 msec
IWT       1000.0000 sec
POINT        65536
SPO          65536
TIMES          350
DUMMY            2
FREQU         33898.30 Hz
FLT           16950 Hz
DELAY        11.80 usec
ACQTM         1.9333 sec
PD            2.0000 sec
ADBIT            16
RGAIN            25
BF             0.12 Hz
T1               0.00
T2               0.00
T3              90.00
T4             100.00
EXMOD     bcm
EXPCM     single pulse with bilevel decoupling
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN     128250.00 Hz
IRRPW        50 usec
IRATN           511
DFILE     2-NpCN_DMI_minor_13C.als
SF        th5at
LKSET         0.00 KHz
LKFIN        26934.0 Hz
LKLEV          200
LGAIN           23
LKPHS          343
LKSIG          775
CSPED           14 Hz
FILDC
FILDF
SLVNT     CDCL3
```



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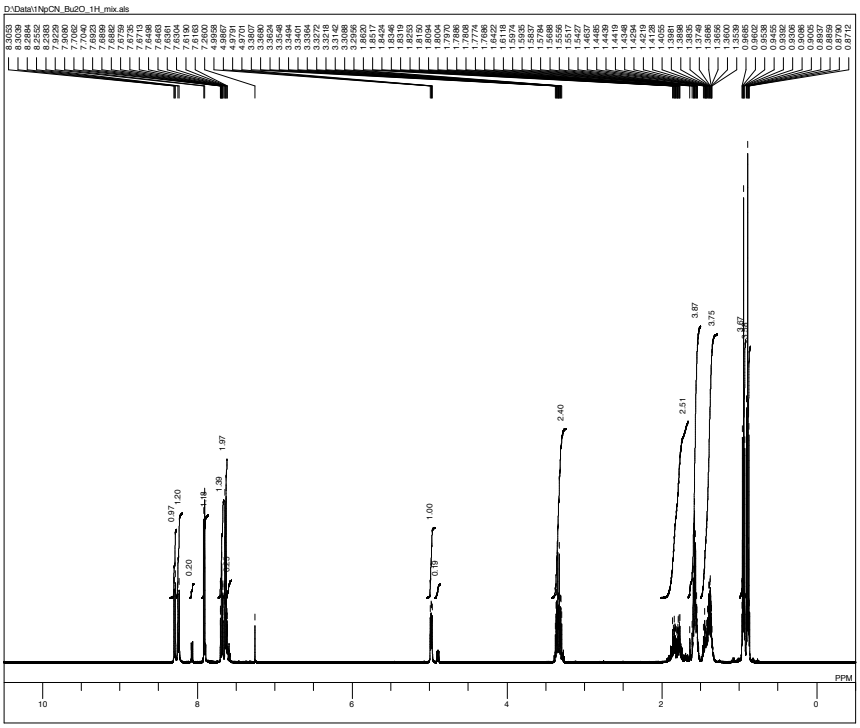
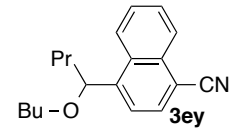



```
MENUF    1H
OBNUC    1H
OFR        499.10 MHz
OBSET        0.00 KHz
OBFIN     128250.00 Hz
PW1           5.50 usec
DEADT        72.30 usec
PREDL     10.00000 msec
IWT          0.5000 sec
POINT       65536
SPO         65536
TIMES           8
DUMMY           0
FREQU     8000.00 Hz
FLT          4000 Hz
DELAY        50.00 usec
ACQTM      8.1920 sec
PD           1.0000 sec
ADBIT           16
RGAIN           13
BF           0.12 Hz
T1           0.00
T2           0.00
T3          90.00
T4         100.00
EXMOD    non
EXPCM    single pulse nondecoupling & nonpresaturation
IRNUC    1H
IFR        499.10 MHz
IRSET        0.00 KHz
IRFIN     128250.00 Hz
IRRPW        50 usec
IRATN          511
DFILE    1NpCN_Bu2O_1H_mix.als
SF       th5at
LKSET        0.00 KHz
LKFIN     26934.0 Hz
LKLEV          200
LGAIN           22
LKPHS          343
LKSIG          777
CSPED       13 Hz
FILDC
FILDF
SLVNT    CDCL3
```



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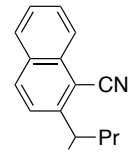

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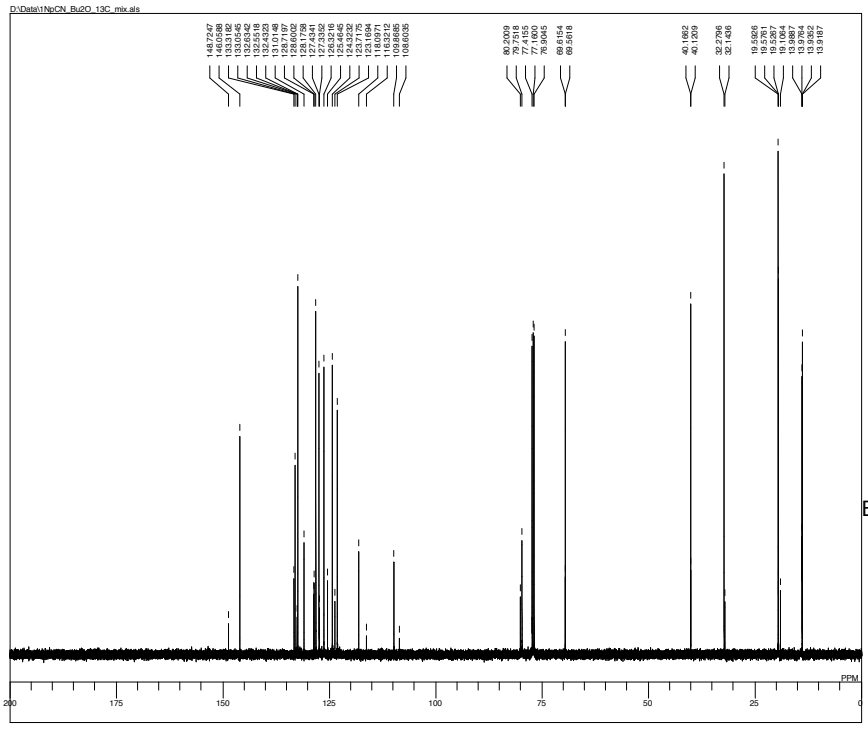
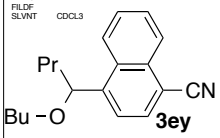



```
MENUF    13C
OBNUC    13C
OFR        125.40 MHz
OBSET        0.00 KHz
OBFIN     143041.00 Hz
PW1           6.25 usec
DEADT        10.00 usec
PREDL     0.01000 msec
IWT       1000.0000 sec
POINT       65536
SPO         65536
TIMES        1000
DUMMY           2
FREQU     33898.30 Hz
FLT          16950 Hz
DELAY        11.80 usec
ACQTM      1.9333 sec
PD           1.0670 sec
ADBIT           16
RGAIN           27
BF           0.12 Hz
T1           0.00
T2           0.00
T3          90.00
T4         100.00
EXMOD    bcm
EXPCM    single pulse with bilevel decoupling
IRNUC    1H
IFR        499.10 MHz
IRSET        0.00 KHz
IRFIN     128250.00 Hz
IRRPW        50 usec
IRATN          511
DFILE    1NpCN_Bu2O_13C_mix.als
SF       th5at
LKSET        0.00 KHz
LKFIN     26934.0 Hz
LKLEV          200
LGAIN           24
LKPHS          343
LKSIG          892
CSPED        7 Hz
FILDC
FILDF
SLVNT    CDCL3
```



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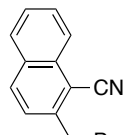

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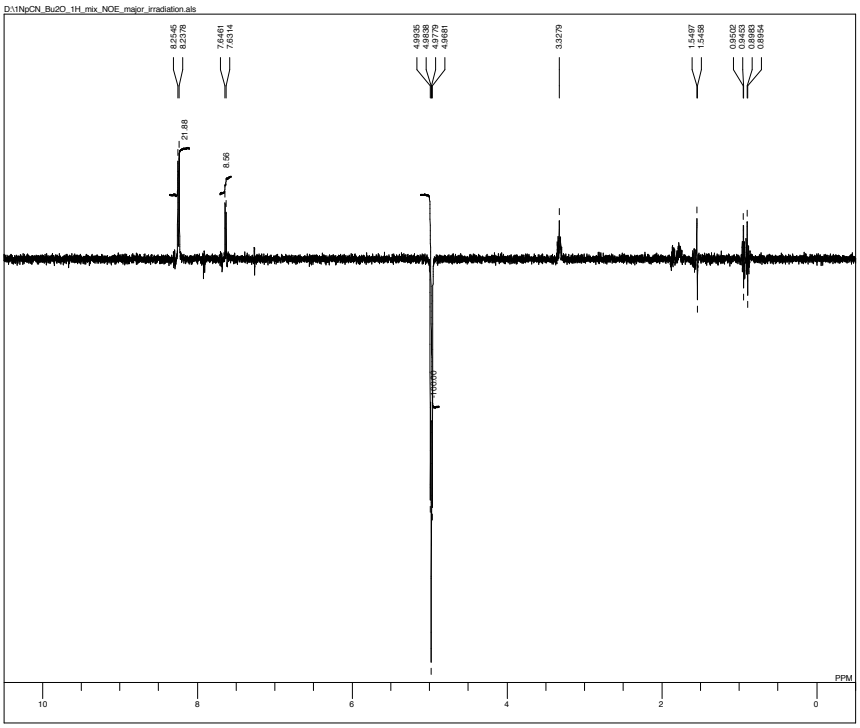
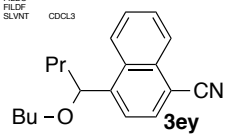



```
MENUF     1H
OBNUC     1H
OFR         499.10 MHz
OBSET         0.00 KHz
OBFIN      128250.00 Hz
PW1           11.00 usec
DEADT         69.50 usec
PREDL      10.00000 msec
IWT           0.5000 sec
POINT        16384
SPO          16384
TIMES          200
DUMMY            4
FREQU      8000.00 Hz
FLT        4000 Hz
DELAY         50.00 usec
ACQTM      2.0480 sec
PD            10.0000 sec
ADBIT            16
RGAIN            21
BF             0.12 Hz
T1             0.00
T2             0.00
T3            90.00
T4           100.00
EXMOD     noe_dif
EXPCM     steady-state NOE(Nuclear Overhauser Effect)_differ
IRNUC     1H
IFR         499.10 MHz
IRSET         0.00 KHz
IRFIN      128244.80 Hz
IRPW          50 usec
IRATN           350
DFILE     1NpCN_Bu2O_1H_mix_NOE_major_irradiation.als
SF        th5at
LKSET         0.00 KHz
LKFIN      26934.0 Hz
LKLEV          200
LGAIN           21
LKPHS          343
LKSIG          460
CSPED        0 Hz
FILDC
FILDF
SLVNT     CDCL3
```



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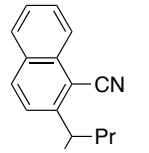

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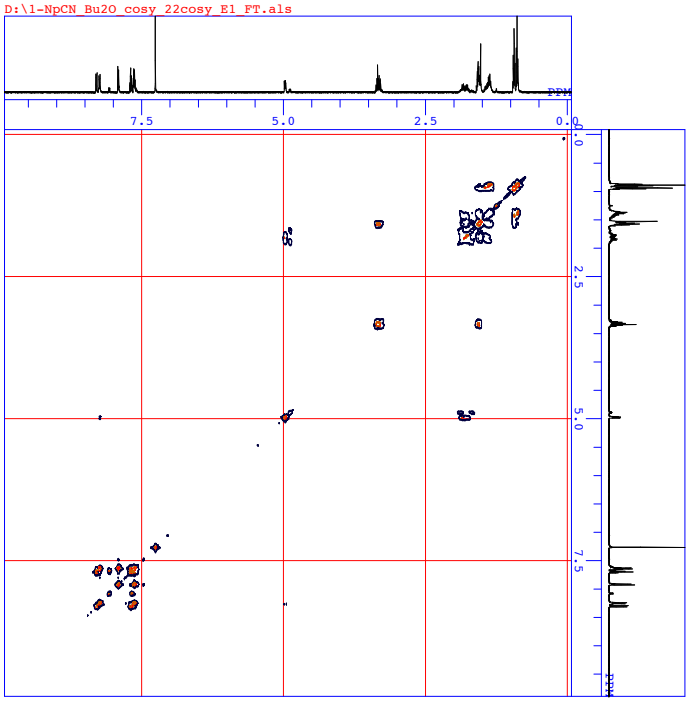
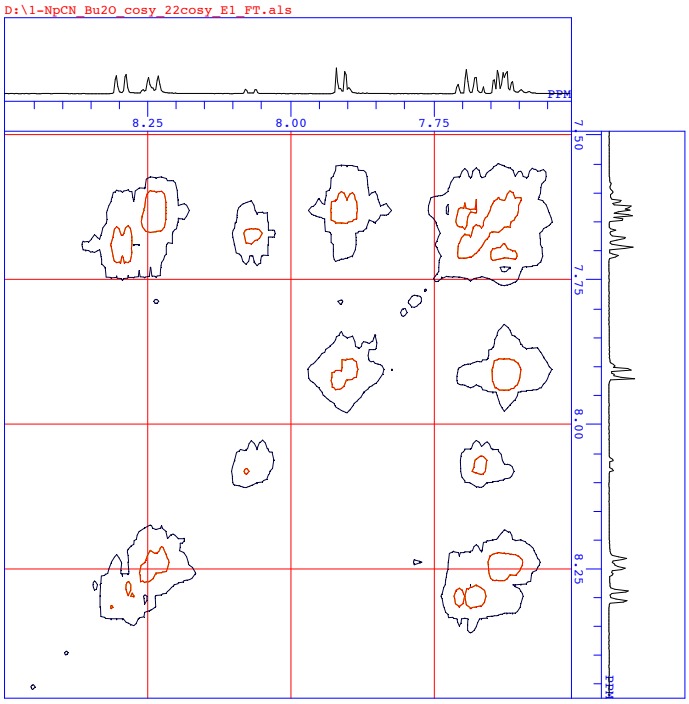



```
DATIM  Tue Apr 29 10:03:58 2014
DFILE  1-NpCN_Bu2O_cosy_22cosy_E1_FT.
OBNUC  1H
EXMOD  cosy
OFR          499.10 MHz
OBSET          0.00 KHz
OBFIN     128215.90 Hz
POINT          1024
FREQU        4992.51 Hz
SCANS            64
ACQTM          0.2051 sec
PD             0.9106 sec
PW1             11.00 usec
IRN
CTEMP            26.5 c
SLVNT  CDCL3
EXREF            7.26 ppm
BF               4.20 Hz
RGAIN              22
```




```
DATIM  Tue Apr 29 10:03:58 2014
DFILE  1-NpCN_Bu2O_cosy_22cosy_E1_FT.
OBNUC  1H
EXMOD  cosy
OFR          499.10 MHz
OBSET          0.00 KHz
OBFIN     128215.90 Hz
POINT          1024
FREQU        4992.51 Hz
SCANS            64
ACQTM         0.2051 sec
PD            0.9106 sec
PW1           11.00 usec
IRN
CTEMP           26.5 c
SLVNT  CDCL3
EXREF           7.26 ppm
BF              4.20 Hz
RGAIN             22
```