Copper-catalyzed Tandem Annulation/Arylation for the synthesis of Diindolylmethanes from Propargylic Alcohols

Hui Li,[†,‡] Xiaoxun Li,[†] Hao-Yuan Wang,[†] Gabrielle N. Winston-McPherson,[†] Hao-miao Julie Geng,[†] Ilia A. Guzei,[§] Weiping Tang[†,§,*]

[†] School of Pharmacy, University of Wisconsin, Madison, WI, 53705, USA; [‡] School of Pharmaceutical Science and Technology, Tianjin University, Tianjin, 300072, P. R. China; [§] Department of Chemistry, University of Wisconsin, Madison, WI, 53706, USA

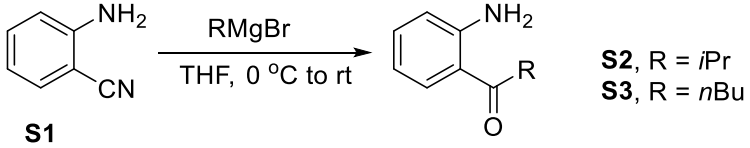
General Remarks

All reactions in non-aqueous media were conducted under a positive pressure of dry argon in glassware that had been oven dried prior to use unless noted otherwise. Anhydrous solutions were transferred via an oven dried syringe or cannula. All solvents were dried prior to use unless noted otherwise. Thin layer chromatography was performed using precoated silica gel plates (EMD Chemical Inc. 60, F254). Flash column chromatography was performed with silica gel (Silicycle, 40-63 μm). Infrared spectra (IR) were obtained on a Bruker Equinox 55 Spectrophotometer. ^{1}H and ^{13}C nuclear magnetic resonance spectra (NMR) were obtained on a Varian Unity-Inova 400 MHz or 500 MHz recorded in ppm (δ) downfield of TMS (δ=0) in $CDCl_3$ unless noted otherwise. Signal splitting patterns were described as singlet (s), doublet (d), triplet(t), quartet (q), quintet (quint), or multiplet (m), with coupling constants (*J*) in hertz. High resolution mass spectra (HRMS) were performed by Analytical Instrument Center at the School of Pharmacy on an Electron Spray Injection (ESI) mass spectrometer. The optical rotation was determined using a Perkin-Elmer 241 Polarimeter.

Experimental procedure for the preparation of propargylic alcohol substrates:

1) Preparation of substrates 7 and 11a-11c[1-4]

Ketones **S2** and **S3** were prepared from **S1**.[1]

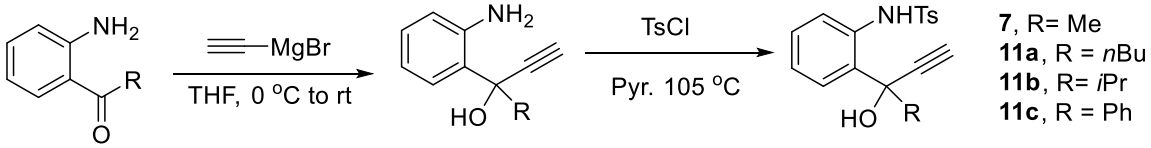


S1

S2, R = *i*Pr
S3, R = *n*Bu

To a solution of 2-aminobenzonitrile **S1** (0.50 g, 4.2 mmol) in THF (4 mL) was added isopropylmagnesium bromide (16.8 mL, 0.5 M in THF, 8.4 mmol) at 0 °C over 5 min. The reaction was then allowed to warm to ambient temperature and stir at this temperature for 1h. The reaction was quenched by slow addition of 1 M HCl. The mixture was extracted by EtOAc. The combined organic layers was washed with $NaHCO_3$ and brine, dried over Na_2SO_4, and concentrated under vacuum. The residue was purified by flash column chromatography (10-30% EtOAc in hexanes) to give **S2** (0.60 g, 88%) as colorless oil.

Following the same procedure, **S3** was prepare from **S1** in 44.3 mmol scale (4.61 g, 59%) as colorless oil.



S4, R = Me

S5, R = Me

7, R= Me
11a, R = *n*Bu
11b, R= *i*Pr
11c, R = Ph

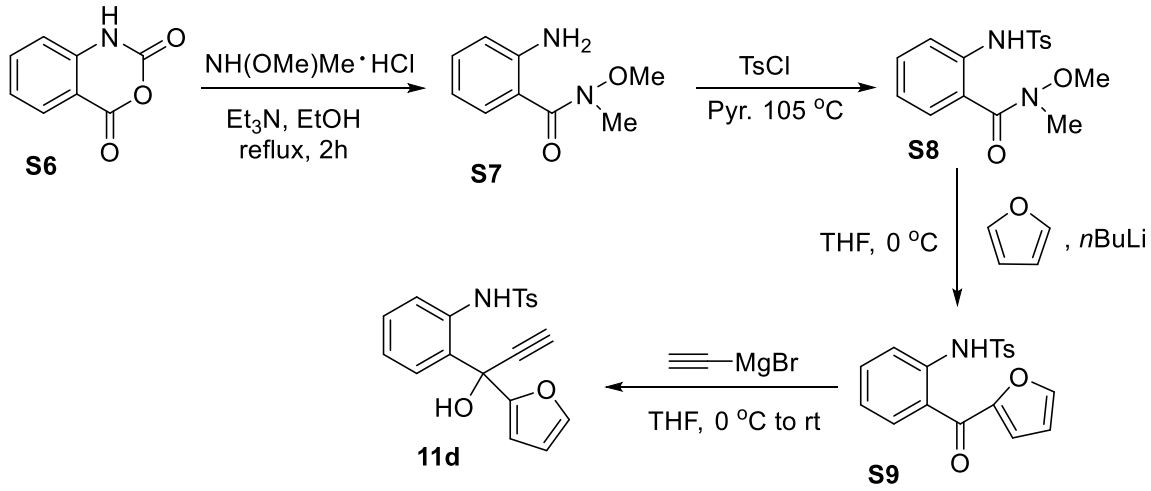
Commercially available 1-(2-aminophenyl)ethanone **S4** (2.12 g, 15.7 mmol) was dissolved in dry THF (100 mL). To the above solution was added ethynylmagnesium bromide (157 mL, 0.5 M in THF, 78.5 mmol) at 0 °C over 30 min with stirring. After additional stirring at 0 °C for 15 min, the mixture was allowed to warm up to room temperature, and stirred over night. The reaction was quenched by saturated aqueous NH_4Cl. The mixture was extracted with EtOAc and the combined organic layers were washed with brine and dried over Na_2SO_4. The solvent was removed under vacuum and the crude product **S5** was directly used for the next step.

To a solution of crude **S5** was added tosyl chloride (1.05 equiv) in pyridine (0.8 M) and the resulting solution was stirred at 105 °C for 30 min under argon. The reaction mixture was cooled to room temperature and diluted with EtOAc. The mixture was washed with 3 M HCl and brine, dried over Na_2SO_4 and concentrated under vacuum. The resulting residue was purified with flash column chromatography (10-30% EtOAc in hexanes) to give the pure product **7**[4] (2.56 g, 62% for two steps) as white solid.

Other substrates were prepared following the same procedure.

11a (0.69 g, 48%); **11b** (0.94 g, 69%); **11c**[5] (1.87 g, 56%).

2) Preparation of substrate 11d:



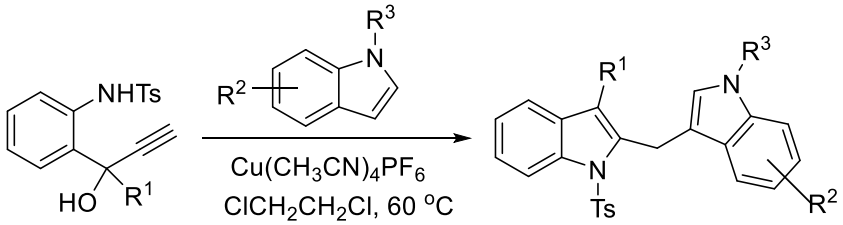
To a solution of *N*,*O*-dimethylhydroxylamine hydrochloride (14.6 g, 0.15 mol) in 90% aqueous ethanol (57 mL) was added triethlyamine (20.8 mL, 0.15 mol) and after 10 min of stirring at room temperature. To the above solution was added isatonic anhydride **S6** (16.3 g, 0.1 mol) in several portions. The reaction was then heated at reflux for 2h and poured onto an equal volume of ice and saturated aqueous Na_2CO_3. The ethanol was then removed under vacuum and the resulting aqueous mixture was extracted with EtOAc. The combined organic layers were washed with water and brine, dried over Na_2SO_4 and concentrated. The resulting residue was purified by column chromatography on silica gel (10-30% EtOAc in hexanes) to give **S7** as a yellow oil (14.4 g, 80%).

Following the same procedure[3] for the preparation of **7** from **S5**, we prepared **S8** from **S7** in 50 mmol scale to obtain (8.16 g, 49%) of the product.

We adapted literature procedure for the preparation of **S9**.[6]
To a solution of furan (0.48 mL, 6.0 mmol) in THF (20 mL) was added *n*BuLi (1.2 mL, 2.5 M in hexanes, 3.0 mmol) dropwise at -78 °C. The solution was then allowed to warm to rt and stirred over night. The solution was then cooled to 0 °C. To this solution was added **S8** (0.67 g, 2.0 mmol) in THF (2 mL). After the addition was complete, the reaction was stirred at 0 °C for 4h. Reaction was diluted with EtOAc and quenched with saturated aqueous NH_4Cl. The aqueous phase was extracted with EtOAc and the combined organic layers were washed with brine and dried over Na_2SO_4. The solvent was removed under vacuum and the resulting residue was purified by flash column chromatography (10-30% EtOAc in hexanes) to give product **S9** (0.61 g, 89%) as colorless oil.

Following the same procedure[2] for the preparation of **S5** from **S4**, we prepared **11d** from **S9** in 0.59 mmol scale to obtain (0.18 g, 67%) of the product.

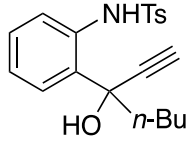
General experimental procedure for the preparation of diindolylmethanes:



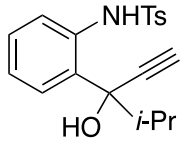
Propargylic alcohol (1.0 equiv) and tetrakis(acetonitrile) copper (I) hexafluorophosphate (10 mol%) were dissolved in 1,2-dichloroethane (0.1 M). To the above solution was added indole substrate (2.0 equiv). The mixture was stirred under argon at 60 °C for overnight. After the reaction was completed, the solvent was removed under reduced pressure. The crude product was purified by flash column chromatography (5% to 20% EtOAc in hexanes) to give the corresponding diindolylmethane.

Characterization data:

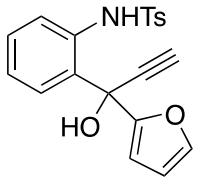
Substrates **7**,[4] **11c**,[5] and **11e**[7] are known compounds.



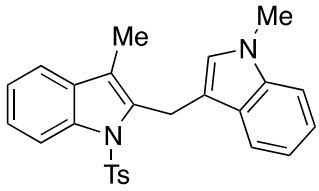
Compound **11a** was isolated as white solid (1.86 g, 61%). mp = 85-86 °C. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 9.08 (s, 1H), 7.73 (d, *J* = 8.0 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.56 (dd, *J* = 8.0, 1.6 Hz, 1H), 7.26-7.21 (m, 3H), 7.01 (t, *J* = 8.0 Hz, 1H), 3.02 (s, 1H), 2.78 (s, 1H), 2.36 (s, 3H), 1.79-1.71 (m, 1H), 1.67-1.60 (m, 1H), 1.38-1.32 (m, 1H), 1.17-0.98 (m, 3H), 0.80 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.9, 137.1, 135.7, 130.2, 129.8, 129.1, 128.7, 127.2, 123.5, 120.2, 84.8, 76.2, 76.0, 42.6, 27.0, 22.5, 21.6, 14.1. IR (neat) ν 3675, 2949, 2877, 1734, 1717, 1692, 1641, 1513. HRMS (ESI) *m/z* calcd for $C_{20}H_{23}NO_3S$ $(M+Na)^+$ 380.1291, found 380.1293.



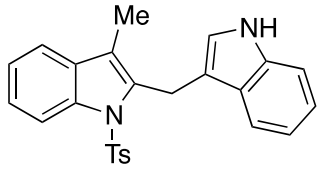
Compound **11b** was isolated as colorless solid (2.15 g, 76%). mp = 111-113 °C. ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.97 (s, 1H), 7.70 (d, *J* = 8.0 Hz, 2H), 7.65 (d, *J* = 8.0 Hz, 1H), 7.56 (d, *J* = 8.0 Hz, 1H), 7.26-7.21 (m, 3H), 7.00 (t, *J* = 8.0 Hz, 1H), 2.99 (s, 1H), 2.78 (s, 1H), 2.37 (s, 3H), 1.79 (m, 1H), 1.04 (d, *J* = 8.0 Hz, 3H), 0.44 (d, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.0, 137.4, 135.8, 130.0, 129.9, 129.8, 129.3, 127.3, 123.4, 120.9, 82.6, 80.4, 77.6, 37.2, 21.7, 18.5, 17.2. IR (neat) ν 3676, 2947, 1734, 1717, 1678, 1641, 1530. HRMS (ESI) *m/z* calcd for $C_{19}H_{21}NO_3S$ $(M+Na)^+$ 366.1134, found 366.1137.



Compound **11d** was isolated as colorless solid (0.15 g, 67%). mp = 123-124 ºC. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.69 (s, 1H), 7.69 (d, *J* = 5.0 Hz, 2H), 7.62 (d, *J* = 10 Hz, 1H), 7.40-7.34 (m, 2H), 7.28-7.24 (m, 1H), 7.20 (d, *J* = 10 Hz, 2H), 7.01-6.98 (m, 1H), 6.30-6.29 (m, 1H), 6.17 (d, *J* = 5.0 Hz, 1H), 3.43 (s, 1H), 2.84 (s, 1H), 2.37 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 152.9, 143.9, 143.8, 137.1, 136.5, 130.1, 129.8, 129.0, 127.8, 127.7, 123.5, 119.6, 110.9, 109.2, 82.4, 76.9, 71.3, 21.8. IR (neat) ν 3681, 2944, 1734, 1652, 1537. HRMS (ESI) *m/z* calcd for $C_{20}H_{17}NO_4S$ $(M+Na)^+$ 390.0771, found 390.0790.

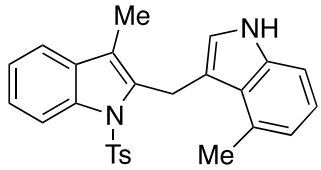


Compound **9a** was isolated as brown oil (16 mg, 76%, [0.05 mmol]; 62 mg, 73% [0.2 mmol]). ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.23 (d, *J* = 5.0 Hz, 1H), 7.60 (d, *J* = 10.0 Hz, 1H), 7.44 (d, *J* = 10.0 Hz, 1H), 7.30-7.24 (m, 4H), 7.22-7.19 (m, 2H), 7.11-7.08 (m, 1H), 6.86 (d, *J* = 10.0 Hz, 2H), 6.39 (s, 1H), 4.51 (s, 2H), 3.53 (s, 3H), 2.24 (s, 3H), 2.23 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.1, 137.1, 137.0, 136.5, 135.7, 131.4, 129.3, 127.74, 127.69, 126.5, 124.5, 123.5, 121.7, 119.2, 119.1, 118.8, 117.3, 115.4, 112.1, 109.2, 32.7, 22.1, 21.7, 9.5. IR (neat) ν 3681, 3639, 2928, 1652, 1537. HRMS (ESI) *m/z* calcd for $C_{26}H_{24}N_2O_2S$ $(M+Na)^+$ 451.1451, found 451.1460.

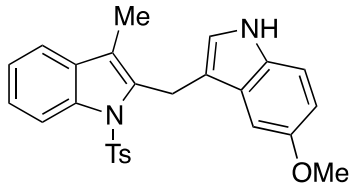


Compound **9b** was isolated as white solid (15 mg, 75%). mp=176-178 ºC. ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.20 (d, *J* = 10.0 Hz, 1H), 7.80 (s, 1H), 7.58 (d, *J* = 10.0, 1H), 7.42 (d, *J* = 5.0 Hz, 1H), 7.33 (d, *J* = 10.0 Hz, 2H), 7.30-7.22 (m, 3H), 7.14 (t, *J* = 10.0 Hz, 1H), 7.07 (t, *J* = 10.0 Hz, 1H), 6.87 (d, *J* = 5.0 Hz, 2H), 6.60 (s, 1H), 4.51 (s, 2H), 2.21 (s, 3H), 2.19 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.2, 136.9, 136.4, 136.2, 135.5, 131.5, 129.4, 127.3, 126.5, 124.5, 123.5, 123.0, 122.1, 119.6, 119.0, 118.8, 117.6, 115.4, 113.7, 111.3, 22.3, 21.7, 9.5. IR (neat) ν 3676, 2948, 1734, 1692, 1660, 1530.

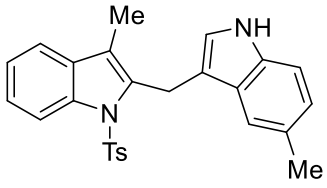
HRMS (ESI) *m/z* calcd for $C_{25}H_{22}N_2O_2S$ $(M+Na)^+$ 437.1294, found 437.1297. X-ray structure (CCDC 1016687).



Compound **9c** was isolated as pink oil (33 mg, 77%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.28 (d, *J* = 8.0 Hz, 1H), 7.64 (s, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.40 (d, *J* = 8.0 Hz, 2H), 7.36-7.26 (m, 2H), 7.14-7.13 (m, 1H), 7.06 (d, *J* = 8.0 Hz, 1H), 6.93 (d, *J* = 8.0 Hz, 2H), 6.88 (d, *J* = 8.0 Hz, 1H), 6.13 (s, 1H), 4.78 (s, 2H), 2.84 (s, 3H), 2.24 (s, 3H), 2.15 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.3, 137.1, 136.8, 136.5, 135.5, 131.4, 131.3, 129.5, 126.8, 126.0, 124.5, 123.4, 122.44, 122.38, 121.3, 118.7, 117.4, 115.3, 115.0, 109.2, 24.4, 21.8, 21.0, 9.1. IR (neat) ν 3361, 2925, 1717, 1685, 1647, 1521. HRMS (ESI) *m/z* calcd for $C_{26}H_{24}N_2O_2S$ $(M+Na)^+$ 451.1451, found 451.1454.



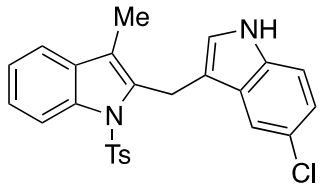
Compound **9d** was isolated as yellow oil (21 mg, 95%). ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.20 (d, *J* = 5.0 Hz, 1H), 7.69 (s, 1H), 7.43 (d, *J* = 10.0 Hz, 1H), 7.34 (d, *J* = 5.0 Hz, 2H), 7.31-7.28 (m, 1H), 7.18 (d, *J* = 5.0 Hz, 1H), 7.03 (d, *J* = 2.5 Hz, 1H), 6.91 (d, *J* = 10.0 Hz, 2H), 6.82 (dd, *J* = 10.0, 2.5 Hz, 1H), 6.63 (s, 1H), 4.50 (s, 2H), 3.82 (s, 3H), 2.25 (s, 3H), 2.23 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 154.3, 144.2, 136.9, 136.3, 135.5, 131.6, 131.5, 129.4, 127.7, 126.6, 124.5, 123.7, 123.5, 118.7, 117.6, 115.4, 113.6, 112.4, 111.9, 101.0, 56.1, 22.4, 21.7, 9.5. IR (neat) ν 3662, 3447, 2943, 1717, 1652, 1537, 1521. HRMS (ESI) *m/z* calcd for $C_{26}H_{24}N_2O_3S$ $(M+Na)^+$ 467.1400, found 467.1403.



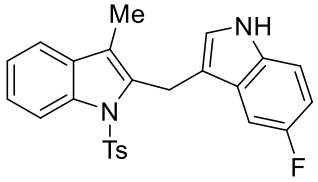
Compound **9e** was isolated as a yellow oil (29 mg, 68% yield).

^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.21 (d, *J* = 8.0 Hz, 1H), 7.69 (s, 1H), 7.45 – 7.34 (m, 4H), 7.33 – 7.26 (m, 2H), 7.20 (d, *J* = 8.4 Hz, 1H), 7.00 (d, *J* = 8.0 Hz, 1H), 6.93 (d, *J* = 8.4 Hz, 2H), 6.56 (d, *J* = 1.2 Hz, 1H), 4.50 (s, 2H), 2.46 (s, 3H), 2.24 (s, 3H), 2.20 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$): δ 144.2, 136.9, 136.3, 135.6, 134.8, 131.5, 129.5, 128.9,

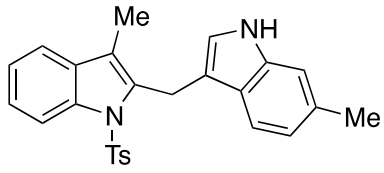
127.6, 126.6, 124.4, 123.9, 123.5, 123.0, 118.8, 118.7, 117.6, 115.4, 113.4, 110.9, 22.2, 21.9, 21.7, 9.5. IR (neat) ν 3360, 2944, 2925, 1652, 1521 cm^{-1}. HRMS (ESI) for $C_{26}H_{24}N_2O_2S$ (M+Na), 451.1451 (Calc.), found 451.1448



Compound **9f** was isolated as yellow oil (14 mg, 63%). ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.20 (d, *J* = 5.0 Hz, 1H), 7.86 (s, 1H), 7.54 (d, *J* = 2.0 Hz, 1H), 7.44 (d, *J* = 5.0 Hz, 1H), 7.38 (d, *J* =10.0 Hz, 2H), 7.32-7.27 (m, 2H), 7.20 (d, *J* = 10.0 Hz, 1H), 7.11 (dd, *J* = 10.0, 2.0 Hz, 1H), 6.96 (d, *J* = 10.0 Hz, 2H), 6.71 (s, 1H), 4.46 (s, 2H), 2.25 (s, 3H), 2.23 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.4, 137.0, 136.3, 135.0, 134.7, 131.5, 129.6, 128.4, 126.5, 125.4, 124.6, 124.4, 123.6, 122.5, 118.9, 118.6, 117.8, 115.4, 113.8, 112.2, 22.3, 21.8, 9.5. IR (neat) ν 3399, 2926, 2253, 1726, 1598. HRMS (ESI) *m/z* calcd for $C_{25}H_{21}ClN_2O_2S$ $(M+Na)^+$ 471.0904, found 471.0905.

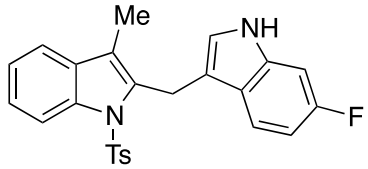


Compound **9g** was isolated as a brown solid (43 mg, 70% yield). mp = 55-60 ºC ^{1}H NMR (500 MHz, $CDCl_3$): δ 2.158 (s, 3H), 2.161 (s, 3H), 4.38 (s, 2H), 6.67 (d, *J* = 0.5 Hz, 1H), 6.81 (td, *J* = 7.5, 2.5 Hz, 1H), 6.86 (d, *J* = 8.5 Hz, 2H), 7.09-7.12 (m, 2H), 7.18-7.23 (m, 2H), 7.28 (d, *J* = 8.5, 2H), 7.36 (d, *J* = 8.5, 1H), 7.78 (broad s, 1H), 8.12 (d, *J* = 7.5, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 9.5, 21.7, 22.4, 104.0 (d, *J* = 22.9 Hz), 110.5 (d, *J* = 26.7 Hz), 111.8 (d, *J* = 9.1 Hz), 114.1, 115.4, 117.8, 118.9, 123.6, 124.6, 124.9, 126.5, 129.5, 131.5, 132.9, 135.1, 136.3, 137.0, 144.3, 158.0 (d, *J* = 232.9 Hz). δ IR (neat) 1358.92, 1486.49, 1581.04, 1597.66, 1710.37, 2853.40, 2923.35, 3422.05 ν cm^{-1}. HRMS (ESI) M/Z calcd. for $C_{25}H_{21}FN_2O_2S$ $(M + Na)^+$ 455.1200, found 455.1200.

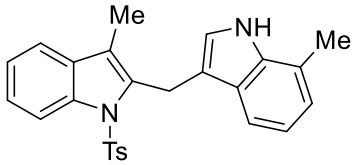


Compound **9h** was isolated as yellow oil (33 mg, 77%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.20 (d, *J* = 4.0 Hz, 1H), 7.67 (s, 1H), 7.46 (d, *J* = 8.0 Hz, 1H), 7.43-7.41 (m, 1H), 7.34 (d, *J* = 8.0 Hz, 2H), 7.31-7.25 (m, 1H), 7.23-7.19 (m, 1H), 7.08 (s, 1H), 6.93-

6.90 (m, 3H), 6.58 (s, 1H), 4.50 (s, 2H), 2.44 (s, 3H), 2.23 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.2, 136.93, 136.91, 136.3, 135.7, 132.0, 131.5, 129.5, 126.6, 125.3, 124.4, 123.5, 122.3, 121.5, 118.8, 118.7, 117.5, 115.4, 113.7, 111.2, 22.3, 22.0, 21.7, 9.5. IR (neat) ν 3663, 3446, 2949, 2882, 1734, 1717, 1652, 1537. HRMS (ESI) *m/z* calcd for $C_{26}H_{24}N_2O_2S$ $(M+Na)^+$ 451.1451, found 451.1445.

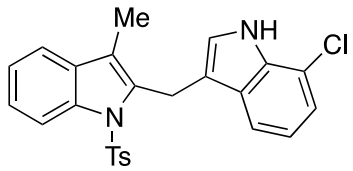


Compound **9i** was isolated as colorless oil (30 mg, 70%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.20 (d, *J* = 8.0 Hz, 1H), 7.80 (s, 1H), 7.49-7.43 (m, 2H), 7.33 (d, *J* = 8.0 Hz, 2H), 7.30-7.25 (m, 2H), 6.98 (dd, *J* = 9.6, 2.4 Hz, 1H), 6.93 (d, *J* = 8.0 Hz, 2H), 6.88-6.83 (m, 1H), 6.66-6.65 (m, 1H), 4.50 (s, 2H), 2.25 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 160.3 (d, *J* = 236.8 Hz), 144.3, 137.0, 136.3, 135.2, 131.4, 129.5, 126.5, 124.6, 124.0, 123.6, 123.2, 119.9 (d, *J* = 10 Hz), 118.8, 117.7, 115.4, 114.1, 108.5 (d, *J* = 24 Hz), 97.5 (d, *J* = 26 Hz), 22.3, 21.7, 9.5. IR (neat) ν 3649, 2948, 1734, 1692, 1641, 1513. HRMS (ESI) *m/z* calcd for $C_{25}H_{22}N_2O_2SF$ $(M+Na)^+$ 455.1200, found 455.1198.



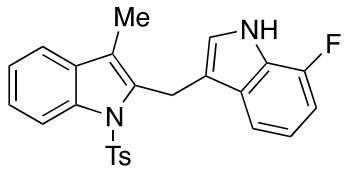
Compound **9j** was isolated as a yellow oil (28 mg, 67% yield).

^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.20 (d, *J* = 8.0 Hz, 1H), 7.69 (s, 1H), 7.43 (d, *J* = 7.6 Hz, 2H), 7.37 – 7.22 (m, 4H), 6.98 (m, 2H), 6.88 (d, *J* = 8.4 Hz, 2H), 6.63 (d, *J* = 1.2 Hz, 1H), 4.51 (s, 2H), 2.42 (s, 3H), 2.22 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.1, 137.0, 136.4, 135.6, 131.5, 129.4, 126.9, 126.5, 124.5, 123.5, 122.73, 122.69, 120.3, 119.9, 118.8, 117.6, 116.9, 115.4, 114.4, 22.5, 21.7, 16.8, 9.5. IR (neat) ν 3388, 2922, 2854, 1652, 1457 cm^{-1}. HRMS (ESI) for $C_{26}H_{24}N_2O_2S$ (M+Na), 451.1451 (Calc.), found 451.1449.

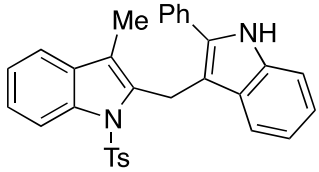


Compound **9k** was isolated as colorless oil (26 mg, 59%). ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.21 (d, *J* = 5.0 Hz, 1H), 7.95 (s, 1H), 7.47-7.43 (m, 2H), 7.33-7.24 (m, 3H), 7.14

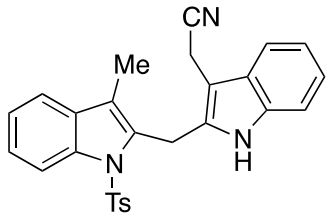
(d, *J* = 5.0 Hz, 1H), 7.01-6.98 (m, 1H), 6.85 (d, *J* = 10.0 Hz, 2H), 6.67 (s, 1H), 4.50 (s, 2H), 2.26 (s, 3H), 2.22 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.2, 137.1, 136.4, 135.0, 133.6, 131.2, 129.3, 128.8, 126.2, 124.7, 123.7, 123.5, 121.5, 120.5, 118.8, 117.8, 117.6, 116.7, 115.3, 114.9, 22.4, 21.7, 9.5. IR (neat) ν 3674, 3384, 2949, 1734, 1717, 1678, 1641, 1631, 1513. HRMS (ESI) *m/z* calcd for $C_{25}H_{21}ClN_2O_2S$ $(M+Na)^+$ 471.0904, found 471.0897.



Compound **9l** was isolated as brown oil (35 mg, 81%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.13 (d, *J* =8.0 Hz, 1H), 7.87 (s, 1H), 7.39-7.37 (m, 1H), 7.25-7.18 (m, 6H), 6.91 (ddd, *J* = 20.0, 12.0, 4.0 Hz, 1H), 6.83 (d, *J* = 8.0 Hz, 2H), 6.62 (s, 1H), 4.44 (s, 2H), 2.18 (s, 3H), 2.16 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 149.7 (d, *J* = 242.8 Hz), 144.3, 137.1, 136.4, 135.1, 131.2 (d, *J* = 29.8 Hz), 129.9, 129.2 (d, *J* = 40 Hz), 126.4, 124.6, 123.7, 123.6, 120.0 (d, *J* = 6.1 Hz), 118.8, 117.7, 115.4, 115.0, 114.9, 114.8, 107.0 (d, *J* =15 Hz), 22.4, 21.7, 9.5. IR (neat) ν 3801, 3344, 1749, 1734, 1652, 1530. HRMS (ESI) *m/z* calcd for $C_{25}H_{21}FN_2O_2S$ $(M+Na)^+$ 455.1200, found 455.1202.

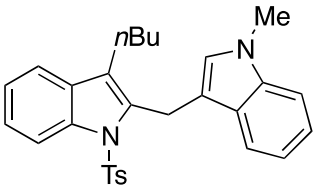


Compound **9m** was isolated as colorless oil (35 mg, 71%). ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.25 (d, *J* = 5.0 Hz, 1H), 8.04 (s, 1H), 7.51 (d, *J* = 10.0 Hz, 2H), 7.41-7.22 (m, 9H), 7.08 (t, *J* = 10.0 Hz, 1H), 7.00-6.94 (m, 3H), 6.84-6.81 (m, 1H), 4.66, (s, 2H), 2.26 (s, 3H), 1.81 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.3, 137.0, 136.6, 135.9, 135.2, 134.3, 133.2, 131.7, 129.6, 129.3, 129.1, 128.4, 128.0, 126.3, 124.5, 123.4, 122.4, 120.0, 119.6, 118.6, 118.0, 115.2, 110.9, 109.4, 24.0, 21.8, 9.0. IR (neat) ν 3675, 2948, 1749, 1734, 1661, 1513. HRMS (ESI) *m/z* calcd for $C_{31}H_{26}N_2O_2S$ $(M+Na)^+$ 513.1607, found 513.1617.

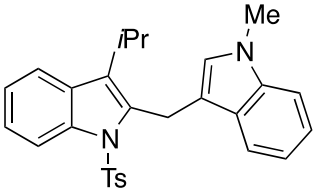


Compound **10** was isolated as colorless oil (35 mg, 78%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.20 (d, *J* = 8.0 Hz, 1H), 8.17 (s, 1H), 7.58-7.56 (m, 1H), 7.47 (d, *J* = 8.0 Hz, 1H),

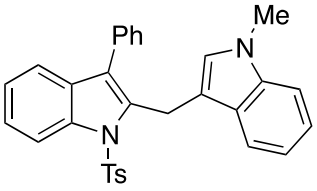
7.38-7.25 (m, 4H), 7.17-7.14 (m, 3H), 6.84 (d, *J* = 8.0 Hz, 2H), 4.56 (s, 2H), 3.89 (s, 2H), 2.33 (s, 3H), 2.19 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.1, 137.1, 135.6, 135.2, 132.9, 131.6, 131.0, 129.8, 127.4, 126.2, 125.5, 124.1, 122.6, 120.5, 119.3, 119.2, 118.4, 118.0, 115.5, 111.3, 100.6, 23.3, 21.7, 13.3, 9.6. IR (neat) ν 3337, 1734, 1717, 1652, 1513. HRMS (ESI) *m/z* calcd for $C_{27}H_{23}N_3O_2S$ $(M+Na)^+$ 476.1403, found 476.1407.



Compound **12a** was isolated as yellow oil (16 mg, 65%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.24 (d, *J* = 8.0 Hz, 1H), 7.61 (d, *J* = 8.0 Hz, 1H), 7.48 (d, *J* = 8.0 Hz, 1H), 7.32-7.24 (m, 4H), 7.20 (d, *J* = 4.0 Hz, 2H), 7.10 (ddd, *J* = 20.0, 12.0, 4.0 Hz, 1H), 6.86 (d, *J* = 8.0 Hz, 2H), 6.34 (s, 1H), 4.51 (s, 2H), 3.51 (s, 3H), 2.69 (t, *J* = 8.0 Hz, 2H), 2.24 (s, 3H), 1.51 (m, 2H), 1.28 (m, 2H), 0.84 (t, *J* = 8.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.0, 137.3, 137.1, 136.4, 135.6, 130.9, 129.2, 127.74, 127.68, 126.5, 124.3, 123.4, 122.3, 121.7, 119.2, 119.1, 119.0, 115.6, 112.4, 109.2, 32.7, 32.4, 24.4, 22.9, 22.1, 21.7, 14.2. IR (neat) ν 3675, 2949, 1734, 1678, 1513. HRMS (ESI) *m/z* calcd for $C_{29}H_{30}N_2O_2S$ $(M+Na)^+$ 493.1920, found 493.1932.

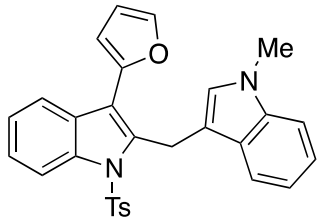


Compound **12b** was isolated as brown oil (38 mg, 83%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.32 (d, *J* = 8.0 Hz, 1H), 7.67 (q, *J* = 8.0 Hz, 2H), 7.32-7.21 (m, 6H), 7.15-7.11 (m, 1H), 6.86 (d, *J* = 8.0 Hz, 2H), 6.18 (s, 1H), 4.54 (s, 2H), 3.49 (s, 3H), 3.22 (m, 1H), 2.25 (s, 3H), 1.36 (s, 3H), 1.34 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.0, 137.7, 137.2, 136.5, 134.1, 129.23, 129.17, 127.6, 127.4, 126.9, 126.6, 124.0, 123.0, 121.8, 120.6, 119.2, 119.1, 115.7, 112.8, 109.2, 32.7, 26.3, 22.5, 21.9, 21.7. IR (neat) ν 2969, 2936, 2875, 1722, 1679, 1612. HRMS (ESI) *m/z* calcd for $C_{28}H_{28}N_2O_2S$ $(M+Na)^+$ 479.1764, found 479.1770.

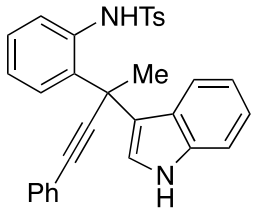


Compound **12c** was isolated as colorless oil (21 mg, 42%). ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.29 (d, *J* = 10.0 Hz, 1H), 7.48 (d, *J* = 5.0 Hz, 2H), 7.44-7.17 (m, 11H), 7.02 (m, 1H), 6.91 (d, *J* = 5.0 Hz, 2H), 6.46 (s, 1H), 4.55 (s, 2H), 3.52 (s, 3H), 2.28 (s, 3H). ^{13}C

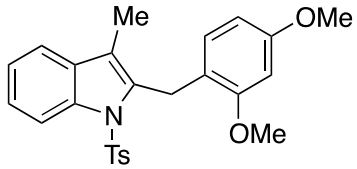
NMR (125 MHz, $CDCl_3$): δ 144.2, 137.2, 137.0, 136.4, 133.4, 130.3, 130.0, 129.3, 128.9, 127.9, 127.8, 127.6, 127.4, 126.5, 124.8, 123.8, 123.7, 121.7, 119.9, 119.3, 119.1, 115.4, 113.0, 109.1, 32.7, 22.7, 21.8. IR (neat) ν 3764, 2923, 1738, 1711, 1530, 1513. HRMS (ESI) *m/z* calcd for $C_{31}H_{26}N_2O_2S$ $(M+Na)^+$ 513.1607, found 513.1609.



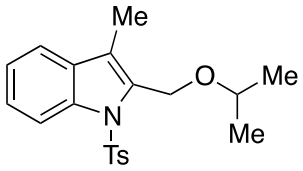
Compound **12d** was isolated as brown oil (29 mg, 60%). ^1H NMR (500 MHz, $CDCl_3$, TMS): δ 8.31 (d, *J* = 10.0 Hz, 1H), 7.94 (d, *J* = 10.0 Hz, 1H), 7.56-7.53 (m, 2H), 7.38-7.29 (m, 4H), 7.23-7.21 (m, 2H), 7.11-7.08 (m, 1H), 6.87 (d, *J* = 10.0 Hz, 2H), 6.48-6.43 (m, 2H), 6.38 (s, 1H), 4.74 (s, 2H), 3.51 (s, 3H), 2.24 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 148.4, 144.4, 142.3, 137.1, 137.0, 136.9, 136.3, 129.4, 128.3, 127.72, 127.70, 126.6, 125.0, 124.1, 121.8, 120.9, 119.3, 119.2, 115.3, 114.0, 111.7, 111.5, 109.2, 108.8, 32.7, 23.2, 21.7. IR (neat) ν 3763, 2947, 1739, 1692, 1642, 1550. HRMS (ESI) *m/z* calcd for $C_{31}H_{24}N_2O_2S$ $(M+Na)^+$ 503.1400, found 503.1400.



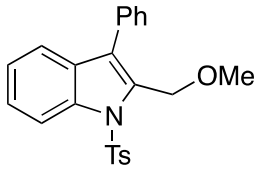
Compound **13** was isolated as white solid (30 mg, 63%). mp=161-163 ºC. ^1H NMR (500 MHz, $CDCl_3$, TMS): δ 8.27 (s, 1H), 7.98 (s, 1H), 7.73 (d, *J* = 10.0 Hz, 1H), 7.50-7.48 (m, 2H), 7.44 (d, *J* = 2.5 Hz, 1H), 7.38-7.36 (m, 2H), 7.32-7.30 (m, 3H), 7.19-7.14 (m, 2H), 7.10-7.06 (m, 3H), 7.03 (d, *J* = 5.0 Hz, 1H), 6.88-6.85 (m, 1H), 6.80 (d, *J* = 10.0 Hz, 2H), 2.23 (s, 3H), 2.12 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 143.5, 137.7, 136.4, 132.1, 131.3, 129.5, 128.62, 128.60, 128.55, 127.5, 127.3, 125.0, 123.5, 123.1, 122.8, 122.2, 120.6, 120.1, 120.0, 118.4, 111.9, 92.8, 85.0, 38.1, 29.6, 21.7. IR (neat) ν 2929, 2820, 1726, 1550. HRMS (ESI) *m/z* calcd for $C_{31}H_{26}N_2O_2S$ $(M+Na)^+$ 513.1607, found 513.1609. X-ray structure (CCDC 1016688).



Compound **14** was isolated as colorless oil (14 mg, 64%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.23 (d, *J* = 8.0 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.33-7.24 (m, 2H), 7.04 (d, *J* = 8.0 Hz, 2H), 6.51 (d, *J* = 8.0 Hz, 1H), 6.46 (d, *J* = 4.0 Hz, 1H), 6.18 (dd, *J* = 8.0, 4.0 Hz, 1H), 4.32 (s, 2H), 3.88 (s, 3H), 3.75 (s, 3H), 2.28 (s, 3H), 2.11 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 159.5, 158.0, 144.4, 137.0, 136.4, 134.7, 131.5, 129.7, 129.0, 126.7, 124.4, 123.5, 119.9, 118.7, 115.4, 110.0, 104.0, 98.6, 55.8, 55.6, 25.1, 21.8, 9.2. IR (neat) ν 3763, 2941, 2839, 1613, 1589, 1505. HRMS (ESI) *m/z* calcd for $C_{25}H_{25}NO_4S$ $(M+Na)^+$ 458.1397, found 458.1396.



Compound **15** was isolated as colorless oil (17 mg, 48%). ^{1}H NMR (400 MHz, $CDCl_3$, TMS): δ 8.09 (d, *J* = 8.0 Hz, 1H), 7.88 (d, *J* = 8.0 Hz, 2H), 7.43 (d, *J* = 8.0 Hz, 1H), 7.31-7.27 (m, 1H), 7.24-7.20 (m, 1H), 7.15 (d, *J* = 8.0 Hz, 2H), 4.86 (s, 2H), 3.82 (m, 1H), 2.31 (s, 3H), 2.27 (s, 3H), 1.24 (s, 3H), 1.22 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.5, 136.5, 133.4, 130.8, 129.7, 127.4, 125.3, 123.4, 120.3, 119.4, 115.1, 71.8, 60.0, 22.3, 21.8, 9.3. IR (neat) ν 2941, 2839, 1613, 1589, 1505. HRMS (ESI) *m/z* calcd for $C_{20}H_{23}NO_3S$ $(M+Na)^+$ 380.1291, found 380.1292.



Compound **16** was isolated as colorless oil (24 mg, 62%). ^{1}H NMR (500 MHz, $CDCl_3$, TMS): δ 8.20 (d, *J* = 10.0 Hz, 1H), 7.96 (d, *J* = 10.0 Hz, 2H), 7.50-7.45 (m, 5H), 7.42-7.39 (m, 1H), 7.37-7.33 (m, 1H), 7.23-7.19 (m, 3H), 4.73 (s, 2H), 3.36 (s, 3H), 2.33 (s, 3H). ^{13}C NMR (125 MHz, $CDCl_3$): δ 144.8, 136.5, 136.4, 133.1, 132.7, 130.4, 129.8, 129.3, 128.9, 128.1, 127.5, 126.7, 125.7, 123.8, 120.6, 115.1, 64.6, 58.1, 21.8. The spectra of compound **16** are in accordance with literature.[5]

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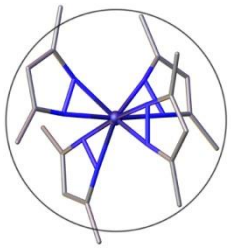


MOLECULAR STRUCTURE LABORATORY

ILIA A. GUZEI, PH.D.

University of Wisconsin-Madison
2124 Chemistry Department
1101 University Ave
Madison, WI 53706

☎ 608-263-4694
Fax 608-262-0381
E-mail: iguzei@chem.wisc.edu

Structural report on **9b**

(CCDC 1016687)

MAY 22, 2014

Crystallographic Experimental Section

Data Collection

A colorless crystal with approximate dimensions 0.56 × 0.48 × 0.41 mm^3 was selected under oil under ambient conditions and attached to the tip of a MiTeGen MicroMount©. The crystal was mounted in a stream of cold nitrogen at 100(1) K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker Quazar SMART APEXII diffractometer with Mo K_α (λ = 0.71073 Å) radiation and the diffractometer to crystal distance of 4.96 cm.

The initial cell constants were obtained from three series of ω scans at different starting angles. Each series consisted of 12 frames collected at intervals of 0.5º in a 6º range about ω with the exposure time of 2 seconds per frame. The reflections were successfully indexed by an automated indexing routine built in the APEXII program suite. The final cell constants were calculated from a set of 9673 strong reflections from the actual data collection.

The data were collected by using the full sphere data collection routine to survey the reciprocal space to the extent of a full sphere to a resolution of 0.70 Å. A total of 48251 data were harvested by collecting 6 sets of frames with 0.5º scans in ω and φ with exposure times of 20 sec per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements. [1]

Structure Solution and Refinement

The systematic absences in the diffraction data were uniquely consistent for

the space group $P2_1/n$ that yielded chemically reasonable and computationally stable results of refinement [2-4].

A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms except H2(N2) were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. The H2 atom was located in the difference Fourier map and refined independently

The final least-squares refinement of 277 parameters against 4971 data resulted in residuals *R* (based on F^2 for $I \geq 2\sigma$) and *wR* (based on F^2 for all data) of 0.0370 and 0.1014, respectively. The final difference Fourier map was featureless.

Summary

Crystal Data for $C_{25}H_{22}N_2O_2S$ (M =414.50): monoclinic, space group $P2_1/n$ (no. 14), a = 13.247(3) Å, b = 11.312(3) Å, c = 14.218(4) Å, β = 109.780(16)°, V = 2005.0(9) Å^3, Z = 4, T = 100.01 K, μ(MoKα) = 0.187 mm^{-1},*Dcalc* = 1.373 g/mm^3, 48251 reflections measured (3.634 ≤ 2Θ ≤ 56.666), 4971 unique (R_{int} = 0.0212, R_{sigma} = 0.0096) which were used in all calculations. The final R_1 was 0.0370 (I > 2σ(I)) and wR_2 was 0.1014 (all data).

References

[1] Bruker-AXS. (**2007-2014**) APEX2 (Ver. 2014.1-1), SADABS (2012-1), and SAINT+ (Ver. 8.32A) Software Reference Manuals. Bruker-AXS, Madison, Wisconsin, USA.

[2] Sheldrick, G. M. (**2008**) SHELXL. *Acta Cryst.* **A64**, 112-122.

[3] Dolomanov, O.V.; Bourhis, L.J.; Gildea, R.J.; Howard, J.A.K.; Puschmann, H. "OLEX2: a complete structure solution, refinement and analysis program". *J. Appl. Cryst.* (**2009**) **42**, 339-341.

[4] Guzei, I.A. (**2013**). Internal laboratory computer programs Gn.

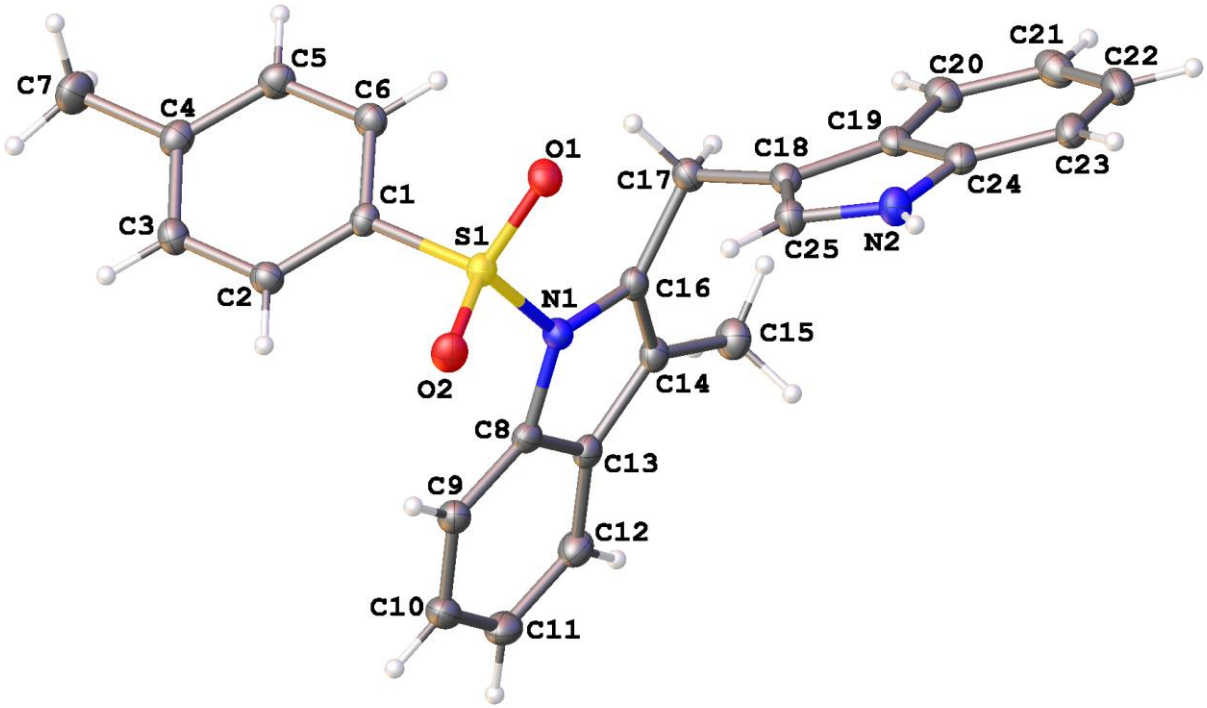


Figure 1. A molecular drawing of **9b** shown with 50% probability ellipsoids.

Table 1 Crystal data and structure refinement for 9b.

Identification code	**9b**
Empirical formula	$C_{25}H_{22}N_2O_2S$
Formula weight	414.50
Temperature/K	100.01
Crystal system	monoclinic
Space group	$P2_1/n$
a/Å	13.247(3)
b/Å	11.312(3)
c/Å	14.218(4)
α/°	90
β/°	109.780(16)
γ/°	90
Volume/Å^3	2005.0(9)
Z	4
ρ_{calc}mg/mm^3	1.373
m/mm^{-1}	0.187
F(000)	872.0
Crystal size/mm^3	0.56 × 0.48 × 0.41
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection	3.634 to 56.666°
Index ranges	-17 ≤ h ≤ 17, -15 ≤ k ≤ 15, -18 ≤ l ≤ 18
Reflections collected	48251
Independent reflections	4971 [R_{int} = 0.0212, R_{sigma} = 0.0096]
Data/restraints/parameters	4971/0/277
Goodness-of-fit on F^2	1.046
Final R indexes [I>=2σ (I)]	R_1 = 0.0370, wR_2 = 0.1000
Final R indexes [all data]	R_1 = 0.0384, wR_2 = 0.1014
Largest diff. peak/hole / e Å^{-3}	0.42/-0.37

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 9b. U_{eq} is defined as 1/3 of of the trace

of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	5203.5(2)	2906.9(2)	7821.4(2)	16.03(9)
O1	4426.5(7)	3399.6(8)	8208.3(6)	20.49(18)
O2	6123.7(7)	2323.2(8)	8489.8(6)	21.52(19)
N1	5672.5(8)	4022.4(9)	7323.6(7)	17.15(19)
N2	4941.6(9)	6834.2(10)	9582.1(8)	21.0(2)
C1	4542.4(9)	1943.3(10)	6846.3(8)	15.6(2)
C2	5069.0(9)	930.1(11)	6711.4(9)	18.5(2)
C3	4529(1)	153.3(11)	5951.7(9)	20.7(2)
C4	3481(1)	380.1(11)	5329.1(9)	20.3(2)
C5	2970.8(10)	1407.1(11)	5486.5(9)	20.8(2)
C6	3488.6(9)	2187.9(10)	6246.2(9)	18.7(2)
C7	2898.2(11)	-481.8(13)	4521.2(11)	29.8(3)
C8	6562.0(9)	3887.2(10)	6989.1(9)	17.8(2)
C9	7359.5(10)	3023.6(11)	7206.9(10)	21.9(2)
C10	8149.9(10)	3154.1(12)	6770.9(11)	25.6(3)
C11	8145.1(10)	4106.2(13)	6143.7(11)	26.7(3)
C12	7347.8(10)	4959.2(12)	5930.9(10)	23.5(3)
C13	6544.0(9)	4846.2(10)	6358.4(9)	18.4(2)
C14	5618.7(9)	5564.8(10)	6283.4(8)	18.0(2)
C15	5332.3(11)	6691.0(11)	5700.7(10)	23.5(2)
C16	5092.3(9)	5054.7(10)	6847.5(8)	16.9(2)
C17	4114.4(9)	5513.7(11)	7031.2(9)	18.4(2)
C18	4345.8(9)	6235.3(10)	7974.0(9)	17.8(2)
C19	3989.4(9)	7426.2(11)	8024.3(9)	18.0(2)
C20	3392.2(10)	8242.7(11)	7312(1)	21.7(2)
C21	3204.6(11)	9355.7(12)	7622.9(10)	24.8(3)
C22	3585.4(10)	9663.8(11)	8640.2(10)	25.0(3)
C23	4162.8(10)	8875.9(11)	9365(1)	21.8(2)
C24	4368.4(9)	7764.8(11)	9043.6(9)	18.7(2)
C25	4920.2(10)	5917.3(11)	8936.3(9)	20.1(2)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 9b. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}

S1	18.20(15)	15.47(14)	13.91(14)	-0.78(9)	4.77(10)	-0.36(9)
O1	24.3(4)	20.4(4)	19.2(4)	-1.8(3)	10.5(3)	-0.3(3)
O2	22.6(4)	21.6(4)	16.4(4)	0.7(3)	1.3(3)	0.9(3)
N1	17.3(4)	15.0(4)	20.1(5)	-0.2(4)	7.6(4)	0.5(3)
N2	24.8(5)	19.3(5)	18.9(5)	-2.3(4)	7.4(4)	0.0(4)
C1	18.3(5)	14.8(5)	13.5(5)	-0.5(4)	5.1(4)	-1.8(4)
C2	17.5(5)	18.9(5)	18.6(5)	0.1(4)	5.6(4)	1.1(4)
C3	22.6(6)	18.6(5)	21.9(6)	-3.3(4)	8.8(5)	0.9(4)
C4	22.5(6)	21.2(6)	17.4(5)	-2.4(4)	6.8(4)	-3.5(4)
C5	18.9(5)	22.2(6)	18.5(5)	1.0(4)	2.6(4)	-0.1(4)
C6	19.2(5)	16.6(5)	19.3(5)	1.1(4)	5.4(4)	1.4(4)
C7	27.3(6)	31.0(7)	27.3(6)	-11.4(5)	4.5(5)	-4.2(5)
C8	16.0(5)	18.1(5)	19.1(5)	-4.9(4)	5.7(4)	-2.5(4)
C9	18.1(5)	19.4(5)	27.3(6)	-3.0(5)	6.7(5)	-0.2(4)
C10	17.8(5)	24.7(6)	34.5(7)	-7.0(5)	9.0(5)	0.4(5)
C11	21.7(6)	28.8(7)	33.9(7)	-8.2(5)	15.0(5)	-5.0(5)
C12	24.7(6)	22.7(6)	26.2(6)	-4.7(5)	12.5(5)	-5.6(5)
C13	19.2(5)	17.0(5)	19.0(5)	-5.5(4)	6.5(4)	-2.9(4)
C14	20.5(5)	16.1(5)	17.3(5)	-3.3(4)	6.5(4)	-1.1(4)
C15	28.2(6)	19.3(6)	23.4(6)	2.0(5)	9.5(5)	-0.1(5)
C16	18.3(5)	14.8(5)	16.6(5)	-2.4(4)	4.6(4)	0.1(4)
C17	18.6(5)	17.5(5)	19.1(5)	-0.9(4)	6.5(4)	1.4(4)
C18	17.6(5)	17.2(5)	20.6(5)	-1.3(4)	8.9(4)	0.0(4)
C19	17.0(5)	17.9(5)	22.1(5)	-0.5(4)	10.6(4)	-0.7(4)
C20	22.3(5)	22.7(6)	23.5(6)	2.5(5)	12.2(5)	2.5(5)
C21	26.2(6)	21.5(6)	31.1(6)	6.3(5)	15.2(5)	5.0(5)
C22	26.0(6)	18.1(6)	35.8(7)	-2.3(5)	17.0(5)	0.1(5)
C23	22.6(5)	19.7(6)	26.8(6)	-5.0(5)	12.9(5)	-3.1(4)
C24	18.1(5)	18.2(5)	22.3(6)	-1.1(4)	10.2(4)	-1.3(4)
C25	21.9(5)	17.2(5)	21.3(6)	-2.4(4)	7.5(4)	0.5(4)

Table 4 Bond Lengths for 9b.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	O1	1.4339(9)	C9	C10	1.3935(18)
S1	O2	1.4287(9)	C10	C11	1.397(2)
S1	N1	1.6664(11)	C11	C12	1.3863(19)

S1	C1	1.7480(12)	C12	C13	1.3992(17)
N1	C8	1.4204(15)	C13	C14	1.4444(16)
N1	C16	1.4354(15)	C14	C15	1.4969(17)
N2	C24	1.3703(16)	C14	C16	1.3569(16)
N2	C25	1.3793(16)	C16	C17	1.4984(16)
C1	C2	1.3885(16)	C17	C18	1.5096(16)
C1	C6	1.3954(16)	C18	C19	1.4373(16)
C2	C3	1.3884(17)	C18	C25	1.3696(17)
C3	C4	1.3947(17)	C19	C20	1.4009(17)
C4	C5	1.3997(18)	C19	C24	1.4165(17)
C4	C7	1.5054(17)	C20	C21	1.3846(18)
C5	C6	1.3842(17)	C21	C22	1.405(2)
C8	C9	1.3947(17)	C22	C23	1.3814(19)
C8	C13	1.4026(17)	C23	C24	1.3954(17)

Table 5 Bond Angles for 9b.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	S1	N1	106.72(5)	C11	C12	C13	118.50(12)
O1	S1	C1	108.34(6)	C8	C13	C14	108.24(10)
O2	S1	O1	119.08(6)	C12	C13	C8	119.84(11)
O2	S1	N1	106.01(6)	C12	C13	C14	131.92(12)
O2	S1	C1	108.97(6)	C13	C14	C15	124.73(11)
N1	S1	C1	107.11(5)	C16	C14	C13	108.06(11)
C8	N1	S1	122.10(8)	C16	C14	C15	127.18(11)
C8	N1	C16	107.59(9)	N1	C16	C17	123.79(10)
C16	N1	S1	126.82(8)	C14	C16	N1	108.94(10)
C24	N2	C25	108.85(11)	C14	C16	C17	127.10(11)
C2	C1	S1	118.95(9)	C16	C17	C18	114.48(10)
C2	C1	C6	121.65(11)	C19	C18	C17	125.42(11)
C6	C1	S1	119.36(9)	C25	C18	C17	128.48(11)
C3	C2	C1	118.64(11)	C25	C18	C19	106.10(10)
C2	C3	C4	121.16(11)	C20	C19	C18	134.16(12)
C3	C4	C5	118.82(11)	C20	C19	C24	118.64(11)
C3	C4	C7	120.56(12)	C24	C19	C18	107.20(10)
C5	C4	C7	120.59(11)	C21	C20	C19	119.16(12)
C6	C5	C4	121.05(11)	C20	C21	C22	120.91(12)

C5	C6	C1	118.66(11)	C23	C22	C21	121.54(12)
C9	C8	N1	130.77(11)	C22	C23	C24	117.20(12)
C9	C8	C13	122.11(11)	N2	C24	C19	107.55(10)
C13	C8	N1	107.11(10)	N2	C24	C23	129.91(12)
C10	C9	C8	116.99(12)	C23	C24	C19	122.53(11)
C9	C10	C11	121.59(12)	C18	C25	N2	110.29(11)
C12	C11	C10	120.97(12)				

Table 6 Hydrogen Bonds for 9b.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N2	H2	O1[1]	0.877(18)	2.123(19)	2.9766(16)	164.2(16)

[1]1-X,1-Y,2-Z

Table 7 Torsion Angles for 9b.

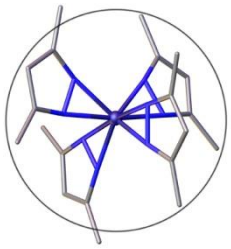
A	B	C	D	Angle/°	A	B	C	D	Angle/°
S1	N1	C8	C9	-18.46(17)	C9	C10	C11	C12	-0.1(2)
S1	N1	C8	C13	162.66(8)	C10	C11	C12	C13	-0.05(19)
S1	N1	C16	C14	-161.64(9)	C11	C12	C13	C8	0.25(18)
S1	N1	C16	C17	22.74(16)	C11	C12	C13	C14	-179.65(12)
S1	C1	C2	C3	178.36(9)	C12	C13	C14	C15	-1.9(2)
S1	C1	C6	C5	-179.06(9)	C12	C13	C14	C16	179.71(12)
O1	S1	N1	C8	171.33(9)	C13	C8	C9	C10	0.21(18)
O1	S1	N1	C16	-32.44(11)	C13	C14	C16	N1	1.73(13)
O1	S1	C1	C2	-145.08(9)	C13	C14	C16	C17	177.17(11)
O1	S1	C1	C6	32.69(11)	C14	C16	C17	C18	-94.30(14)
O2	S1	N1	C8	43.43(10)	C15	C14	C16	N1	-176.62(11)
O2	S1	N1	C16	-160.33(9)	C15	C14	C16	C17	-1.2(2)
O2	S1	C1	C2	-14.13(11)	C16	N1	C8	C9	-178.67(12)
O2	S1	C1	C6	163.64(9)	C16	N1	C8	C13	2.44(12)
N1	S1	C1	C2	100.13(10)	C16	C17	C18	C19	123.67(12)
N1	S1	C1	C6	-82.10(10)	C16	C17	C18	C25	-56.27(17)

N1	C8	C9	C10	−178.53(12)	C17	C18	C19	C20	-0.9(2)
N1	C8	C13	C12	178.66(10)	C17	C18	C19	C24	179.35(11)
N1	C8	C13	C14	-1.42(12)	C17	C18	C25	N2	−179.92(11)
N1	C16	C17	C18	80.51(14)	C18	C19	C20	C21	−178.79(12)
C1	S1	N1	C8	-72.81(10)	C18	C19	C24	N2	1.01(13)
C1	S1	N1	C16	83.42(11)	C18	C19	C24	C23	−179.74(11)
C1	C2	C3	C4	0.32(18)	C19	C18	C25	N2	0.13(14)
C2	C1	C6	C5	-1.35(18)	C19	C20	C21	C22	-1.32(19)
C2	C3	C4	C5	-0.55(18)	C20	C19	C24	N2	−178.77(10)
C2	C3	C4	C7	−178.82(12)	C20	C19	C24	C23	0.49(17)
C3	C4	C5	C6	-0.19(19)	C20	C21	C22	C23	0.3(2)
C4	C5	C6	C1	1.11(18)	C21	C22	C23	C24	1.02(18)
C6	C1	C2	C3	0.64(18)	C22	C23	C24	N2	177.63(12)
C7	C4	C5	C6	178.08(12)	C22	C23	C24	C19	-1.44(18)
C8	N1	C16	C14	-2.63(13)	C24	N2	C25	C18	0.51(14)
C8	N1	C16	C17	−178.24(10)	C24	C19	C20	C21	0.91(17)
C8	C9	C10	C11	-0.01(19)	C25	N2	C24	C19	-0.94(13)
C8	C13	C14	C15	178.20(11)	C25	N2	C24	C23	179.88(12)
C8	C13	C14	C16	-0.20(13)	C25	C18	C19	C20	179.03(13)
C9	C8	C13	C12	-0.34(18)	C25	C18	C19	C24	-0.69(13)
C9	C8	C13	C14	179.58(11)					

Table 8 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for 9b.

Atom	*x*	*y*	*z*	U(eq)
H2	5253(14)	6801(16)	10233(14)	30(4)
H2A	5784	772	7130	22
H3	4880	−545	5854	25
H5	2258	1571	5065	25
H6	3134	2876	6357	22

H7A	3389	−1116	4488	45
H7B	2636	−70	3877	45
H7C	2290	−821	4673	45
H9	7364	2376	7633	26
H10	8706	2582	6903	31
H11	8696	4170	5859	32
H12	7347	5606	5504	28
H15A	5893	7281	5990	35
H15B	4647	6986	5727	35
H15C	5269	6543	5004	35
H17A	3657	4835	7065	22
H17B	3701	6007	6455	22
H20	3119	8035	6624	26
H21	2813	9919	7142	30
H22	3442	10432	8834	30
H23	4410	9082	10053	26
H25	5256	5172	9131	24



MOLECULAR STRUCTURE LABORATORY

ILIA A. GUZEI, PH.D.

University of Wisconsin-Madison
2124 Chemistry Department
1101 University Ave
Madison, WI 53706

☎ 608-263-4694
Fax 608-262-0381
E-mail: iguzei@chem.wisc.edu

Structural report on **13**

(CCDC 1016688)

MAY 22, 2014

Crystallographic Experimental Section

Data Collection

A colorless crystal with approximate dimensions 0.56 × 0.47 × 0.44 mm^3 was selected under oil under ambient conditions and attached to the tip of a MiTeGen MicroMount©. The crystal was mounted in a stream of cold nitrogen at 100(1) K and centered in the X-ray beam by using a video camera.

The crystal evaluation and data collection were performed on a Bruker SMART APEXII diffractometer with Cu K_α (λ = 1.54178 Å) radiation and the diffractometer to crystal distance of 4.03 cm.

The initial cell constants were obtained from three series of ω scans at different starting angles. Each series consisted of 35 frames collected at intervals of 0.6º in a 25º range about ω with the exposure time of 2 seconds per frame. The reflections were successfully indexed by an automated indexing routine built in the APEXII program. The final cell constants were calculated from a set of 9384 strong reflections from the actual data collection.

The data were collected by using the full sphere data collection routine to survey the reciprocal space to the extent of a full sphere to a resolution of 0.82 Å. A total of 46397 data were harvested by collecting 22 sets of frames with 0.7º scans in ω and φ with an exposure time 5-10 sec per frame. These highly redundant datasets were corrected for Lorentz and polarization effects. The absorption correction was based on fitting a function to the empirical transmission surface as sampled by multiple equivalent measurements. [1]

Structure Solution and Refinement

The systematic absences in the diffraction data were uniquely consistent for the space group $P2_1/c$ that yielded chemically reasonable and computationally stable

results of refinement [2-4].

A successful solution by the direct methods provided most non-hydrogen atoms from the *E*-map. The remaining non-hydrogen atoms were located in an alternating series of least-squares cycles and difference Fourier maps. All non-hydrogen atoms were refined with anisotropic displacement coefficients. All hydrogen atoms except H1(N1) and H2(N2) were included in the structure factor calculation at idealized positions and were allowed to ride on the neighboring atoms with relative isotropic displacement coefficients. Atoms H1 and H2 were located in the difference Fourier map and refined independently

The final least-squares refinement of 333 parameters against 4996 data resulted in residuals *R* (based on F^2 for $I \geq 2\sigma$) and *wR* (based on F^2 for all data) of 0.0348 and 0.0893, respectively. The final difference Fourier map was featureless.

Summary

Crystal Data for $C_{31}H_{26}N_2O_2S$ (M =490.60): monoclinic, space group $P2_1/c$ (no. 14), a = 14.1262(11) Å, b = 9.3818(5) Å, c = 20.2526(8) Å, β = 108.247(5)°, V = 2549.1(3) Å^3, Z = 4, T = 99.98 K, μ(CuKα) = 1.370 mm^{-1},*Dcalc* = 1.278 g/mm^3, 46397 reflections measured (6.588 ≤ 2Θ ≤ 145.99), 4996 unique (R_{int} = 0.0173, R_{sigma} = 0.0083) which were used in all calculations. The final R_1 was 0.0348 (I > 2σ(I)) and wR_2 was 0.0893 (all data).

References

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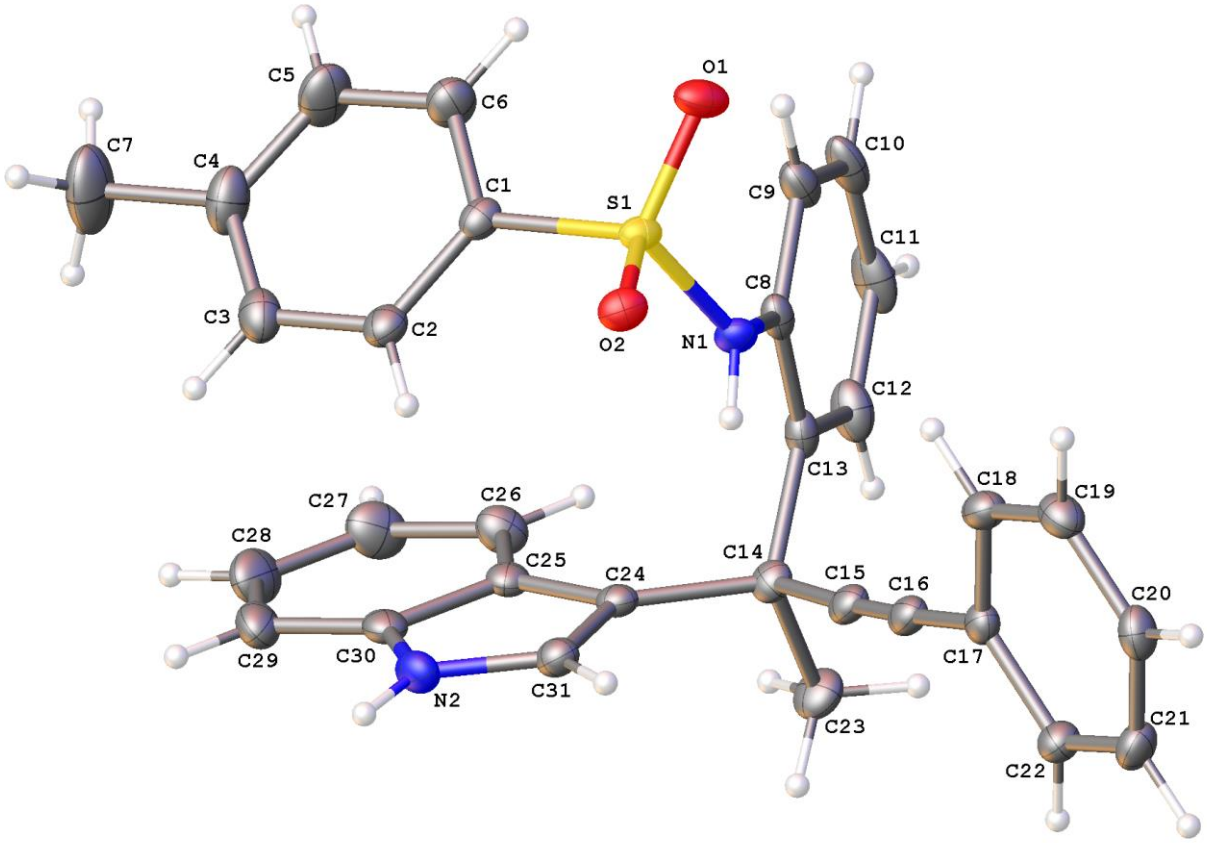


Figure 1. A molecular drawing of **13** shown with 50% probability ellipsoids.

Table 1 Crystal data and structure refinement for 13.

Identification code	**13**
Empirical formula	$C_{31}H_{26}N_2O_2S$
Formula weight	490.60
Temperature/K	99.98
Crystal system	monoclinic
Space group	$P2_1/c$
a/Å	14.1262(11)
b/Å	9.3818(5)
c/Å	20.2526(8)
α/°	90
β/°	108.247(5)
γ/°	90
Volume/Å^3	2549.1(3)
Z	4
ρ_{calc}mg/mm^3	1.278
m/mm^{-1}	1.370
F(000)	1032.0
Crystal size/mm^3	0.56 × 0.47 × 0.44
Radiation	CuKα (λ = 1.54178)
2Θ range for data collection	6.588 to 145.99°
Index ranges	-17 ≤ h ≤ 16, -11 ≤ k ≤ 11, -25 ≤ l ≤ 25
Reflections collected	46397
Independent reflections	4996 [R_{int} = 0.0173, R_{sigma} = 0.0083]
Data/restraints/parameters	4996/0/333
Goodness-of-fit on F^2	1.055
Final R indexes [I>=2σ (I)]	R_1 = 0.0348, wR_2 = 0.0892
Final R indexes [all data]	R_1 = 0.0349, wR_2 = 0.0893
Largest diff. peak/hole / e Å^{-3}	0.32/-0.49

Table 2 Fractional Atomic Coordinates (×10^4) and Equivalent Isotropic Displacement Parameters (Å^2×10^3) for 13. U_{eq} is defined as 1/3 of of the trace

of the orthogonalised U_{IJ} tensor.

Atom	*x*	*y*	*z*	U(eq)
S1	7366.3(2)	5399.3(3)	6318.8(2)	15.13(9)
O1	8059.2(7)	5873.2(10)	6961.2(5)	23.2(2)
O2	6353.3(6)	5887.7(9)	6118.7(5)	19.66(19)
N1	7256.0(8)	3670.0(11)	6335.8(5)	16.1(2)
N2	5753.8(8)	3114.4(12)	4033.4(6)	21.1(2)
C1	7877.8(9)	5798.4(13)	5654.3(6)	17.4(2)
C2	7258.7(10)	5775.3(13)	4967.3(7)	20.7(3)
C3	7660.2(11)	6070.6(14)	4442.0(7)	25.6(3)
C4	8668.6(12)	6382.9(17)	4590.4(8)	31.5(3)
C5	9271.7(11)	6388.6(19)	5279.9(9)	34.9(4)
C6	8884.6(10)	6106.6(16)	5817.5(7)	26.4(3)
C7	9097.1(14)	6719(2)	4013.2(10)	50.8(5)
C8	8022.9(9)	2633.1(13)	6456.8(6)	16.4(2)
C9	8976.6(9)	2941.7(15)	6897.2(6)	21.8(3)
C10	9717(1)	1908.2(17)	7044.8(7)	27.1(3)
C11	9501.1(11)	563.0(17)	6760.4(7)	29.5(3)
C12	8550.8(11)	252.4(15)	6324.1(7)	25.6(3)
C13	7794.3(9)	1274.7(14)	6152.0(6)	18.1(3)
C14	6755.2(9)	930.3(13)	5636.0(6)	17.6(2)
C15	5990.4(9)	1242.1(13)	5976.5(6)	18.5(3)
C16	5396.4(9)	1571.5(13)	6262.7(6)	18.8(3)
C17	4721.5(9)	1972.1(13)	6634.6(6)	17.4(2)
C18	4892.6(10)	3213.0(14)	7038.8(7)	20.9(3)
C19	4268(1)	3557.7(14)	7424.1(7)	23.5(3)
C20	3473.1(10)	2679.0(15)	7413.3(7)	23.4(3)
C21	3297(1)	1447.5(15)	7012.1(7)	23.4(3)
C22	3913(1)	1094.2(14)	6622.3(7)	20.5(3)
C23	6645.8(11)	-658.8(14)	5420.5(8)	25.6(3)
C24	6556.5(9)	1821.6(12)	4976.2(6)	16.0(2)
C25	7166.8(9)	1884.9(13)	4524.8(6)	17.7(2)
C26	8093.4(10)	1300.1(15)	4544.5(7)	24.8(3)
C27	8440.3(12)	1529.9(18)	3986.6(8)	32.8(3)
C28	7897.5(12)	2345.5(18)	3411.2(8)	33.5(3)
C29	6991.2(11)	2937.3(15)	3378.2(7)	27.1(3)
C30	6632.9(10)	2692.8(13)	3936.8(6)	19.9(3)

C31	5710.5(9)	2575.0(13)	4654.1(6)	18.7(2)

Table 3 Anisotropic Displacement Parameters ($Å^2×10^3$) for 13. The Anisotropic displacement factor exponent takes the form: $-2π^2[h^2a^{*2}U_{11}+2hka^*b^*U_{12}+...]$.

Atom	U_{11}	U_{22}	U_{33}	U_{23}	U_{13}	U_{12}
S1	13.59(16)	15.99(15)	16.54(15)	-4.14(10)	5.76(12)	-1.95(10)
O1	19.1(5)	30.0(5)	21.0(5)	-10.2(4)	6.8(4)	-5.5(4)
O2	17.1(4)	18.1(4)	25.0(4)	-3.0(3)	8.2(4)	0.8(3)
N1	11.1(5)	16.9(5)	18.9(5)	0.0(4)	2.5(4)	-1.3(4)
N2	19.6(6)	20.3(5)	19.2(5)	3.0(4)	0.1(4)	0.6(4)
C1	19.3(6)	14.0(5)	21.5(6)	-1.3(5)	9.8(5)	-0.8(4)
C2	23.8(7)	16.5(6)	22.5(6)	-1.1(5)	8.1(5)	-3.1(5)
C3	34.7(8)	21.4(6)	22.1(6)	2.6(5)	11.1(6)	1.5(5)
C4	35.4(8)	33.2(8)	33.1(8)	10.3(6)	21.2(7)	6.7(6)
C5	22.3(7)	47.2(9)	39.5(8)	9.4(7)	15.9(6)	-0.6(6)
C6	20.4(7)	32.9(7)	26.9(7)	1.6(6)	9.1(5)	-1.8(5)
C7	45.7(10)	74.7(13)	43.1(10)	23.9(9)	30.2(9)	12.8(9)
C8	14.3(6)	22.4(6)	13.4(5)	4.9(5)	5.3(4)	3.3(5)
C9	16.8(6)	33.3(7)	14.9(6)	2.9(5)	4.3(5)	0.9(5)
C10	15.8(6)	48.4(9)	15.9(6)	8.9(6)	3.4(5)	7.5(6)
C11	24.0(7)	43.5(9)	21.9(7)	12.9(6)	8.4(6)	19.7(6)
C12	30.0(7)	26.1(7)	22.4(6)	6.9(5)	10.7(6)	12.4(6)
C13	18.7(6)	21.4(6)	15.7(6)	5.6(5)	7.6(5)	4.4(5)
C14	19.3(6)	12.8(6)	20.7(6)	1.4(5)	6.4(5)	1.2(4)
C15	18.7(6)	14.3(5)	21.4(6)	3.0(5)	4.9(5)	-2.7(5)
C16	18.5(6)	16.0(6)	20.4(6)	3.6(5)	3.8(5)	-2.9(5)
C17	18.0(6)	17.7(6)	15.4(5)	4.1(4)	3.7(5)	-0.1(5)
C18	19.5(6)	19.1(6)	21.3(6)	1.4(5)	2.4(5)	-3.9(5)
C19	29.3(7)	20.4(6)	18.4(6)	-1.7(5)	3.9(5)	0.2(5)
C20	27.5(7)	26.6(7)	18.5(6)	4.5(5)	10.8(5)	3.7(5)
C21	23.4(7)	23.8(7)	25.0(6)	4.5(5)	10.3(5)	-4.2(5)
C22	22.9(6)	18.0(6)	20.4(6)	0.1(5)	6.5(5)	-3.7(5)
C23	32.3(8)	13.7(6)	31.7(7)	0.8(5)	11.3(6)	-0.2(5)
C24	15.6(6)	13.7(5)	17.1(6)	-2.4(4)	3.1(5)	-3.0(4)
C25	20.1(6)	15.6(6)	16.4(6)	-4.5(4)	4.2(5)	-3.8(5)
C26	22.8(7)	29.1(7)	22.6(6)	-3.2(5)	7.2(5)	2.1(5)

C27	28.8(8)	43.6(9)	30.4(7)	-7.8(6)	15.4(6)	0.9(6)
C28	41.3(9)	41.7(9)	23.0(7)	-6.4(6)	17.7(6)	-8.1(7)
C29	36.7(8)	26.7(7)	17.2(6)	-1.9(5)	7.4(6)	-6.4(6)
C30	22.7(6)	16.7(6)	17.7(6)	-4.1(5)	2.7(5)	-5.3(5)
C31	17.4(6)	16.7(6)	21.0(6)	0.5(5)	4.7(5)	-2.1(5)

Table 4 Bond Lengths for 13.

Atom	Atom	Length/Å	Atom	Atom	Length/Å
S1	O1	1.4321(9)	C13	C14	1.5461(17)
S1	O2	1.4347(9)	C14	C15	1.4815(17)
S1	N1	1.6313(11)	C14	C23	1.5475(17)
S1	C1	1.7557(12)	C14	C24	1.5260(17)
N1	C8	1.4191(15)	C15	C16	1.2000(19)
N2	C30	1.3743(18)	C16	C17	1.4377(17)
N2	C31	1.3743(17)	C17	C18	1.4000(18)
C1	C2	1.3927(18)	C17	C22	1.4021(17)
C1	C6	1.3861(18)	C18	C19	1.3867(19)
C2	C3	1.3813(19)	C19	C20	1.3876(19)
C3	C4	1.392(2)	C20	C21	1.389(2)
C4	C5	1.390(2)	C21	C22	1.3858(19)
C4	C7	1.509(2)	C24	C25	1.4409(17)
C5	C6	1.388(2)	C24	C31	1.3656(17)
C8	C9	1.3935(18)	C25	C26	1.4082(18)
C8	C13	1.4088(18)	C25	C30	1.4140(18)
C9	C10	1.3884(19)	C26	C27	1.382(2)
C10	C11	1.381(2)	C27	C28	1.404(2)
C11	C12	1.388(2)	C28	C29	1.378(2)
C12	C13	1.3965(18)	C29	C30	1.3953(19)

Table 5 Bond Angles for 13.

Atom	Atom	Atom	Angle/°	Atom	Atom	Atom	Angle/°
O1	S1	O2	119.67(5)	C15	C14	C13	108.69(10)
O1	S1	N1	109.40(6)	C15	C14	C23	107.37(10)
O1	S1	C1	107.57(6)	C15	C14	C24	109.78(10)

O2	S1	N1	103.31(5)	C24	C14	C13	110.65(10)
O2	S1	C1	109.20(6)	C24	C14	C23	107.83(10)
N1	S1	C1	107.05(6)	C16	C15	C14	176.17(13)
C8	N1	S1	127.78(9)	C15	C16	C17	177.40(13)
C31	N2	C30	108.98(11)	C18	C17	C16	120.16(11)
C2	C1	S1	118.82(10)	C18	C17	C22	119.30(12)
C6	C1	S1	119.97(10)	C22	C17	C16	120.48(11)
C6	C1	C2	121.19(12)	C19	C18	C17	119.95(12)
C3	C2	C1	119.05(12)	C18	C19	C20	120.46(12)
C2	C3	C4	121.01(13)	C19	C20	C21	119.94(12)
C3	C4	C7	120.57(15)	C22	C21	C20	120.18(12)
C5	C4	C3	118.80(13)	C21	C22	C17	120.17(12)
C5	C4	C7	120.62(15)	C25	C24	C14	126.55(11)
C6	C5	C4	121.29(14)	C31	C24	C14	126.54(11)
C1	C6	C5	118.65(13)	C31	C24	C25	106.47(11)
C9	C8	N1	120.06(12)	C26	C25	C24	134.55(12)
C9	C8	C13	120.76(12)	C26	C25	C30	118.59(12)
C13	C8	N1	119.12(11)	C30	C25	C24	106.81(11)
C10	C9	C8	120.32(13)	C27	C26	C25	118.71(13)
C11	C10	C9	119.69(13)	C26	C27	C28	121.49(14)
C10	C11	C12	120.05(13)	C29	C28	C27	121.15(13)
C11	C12	C13	121.80(14)	C28	C29	C30	117.51(13)
C8	C13	C14	121.61(11)	N2	C30	C25	107.56(11)
C12	C13	C8	117.35(12)	N2	C30	C29	129.89(13)
C12	C13	C14	121.03(12)	C29	C30	C25	122.53(13)
C13	C14	C23	112.45(10)	C24	C31	N2	110.16(11)

Table 6 Hydrogen Bonds for 13.

D	H	A	d(D-H)/Å	d(H-A)/Å	d(D-A)/Å	D-H-A/°
N2	H2	O2[1]	0.859(18)	2.453(17)	3.0426(15)	126.4(14)

[1]1-X,1-Y,1-Z

Table 7 Torsion Angles for 13.

A	B	C	D	Angle/°	A	B	C	D	Angle/°

S1	N1	C8	C9	32.08(16)	C12	C13	C14	C24	−114.28(13)
S1	N1	C8	C13	−150.78(10)	C13	C8	C9	C10	-0.28(18)
S1	C1	C2	C3	−179.18(10)	C13	C14	C24	C25	56.04(15)
S1	C1	C6	C5	178.66(12)	C13	C14	C24	C31	−132.53(13)
O1	S1	N1	C8	-56.14(11)	C14	C24	C25	C26	-4.7(2)
O1	S1	C1	C2	−164.22(10)	C14	C24	C25	C30	172.53(11)
O1	S1	C1	C6	16.80(13)	C14	C24	C31	N2	−173.26(11)
O2	S1	N1	C8	175.35(10)	C15	C14	C24	C25	176.00(11)
O2	S1	C1	C2	-32.91(12)	C15	C14	C24	C31	-12.57(17)
O2	S1	C1	C6	148.10(11)	C16	C17	C18	C19	−176.78(12)
N1	S1	C1	C2	78.29(11)	C16	C17	C22	C21	176.49(12)
N1	S1	C1	C6	−100.69(11)	C17	C18	C19	C20	0.1(2)
N1	C8	C9	C10	176.81(11)	C18	C17	C22	C21	-0.61(19)
N1	C8	C13	C12	−175.46(11)	C18	C19	C20	C21	-0.3(2)
N1	C8	C13	C14	5.70(17)	C19	C20	C21	C22	0.0(2)
C1	S1	N1	C8	60.14(11)	C20	C21	C22	C17	0.5(2)
C1	C2	C3	C4	0.2(2)	C22	C17	C18	C19	0.32(19)
C2	C1	C6	C5	-0.3(2)	C23	C14	C24	C25	-67.32(15)
C2	C3	C4	C5	0.2(2)	C23	C14	C24	C31	104.11(14)
C2	C3	C4	C7	−179.34(15)	C24	C25	C26	C27	176.52(14)
C3	C4	C5	C6	-0.8(2)	C24	C25	C30	N2	0.91(13)
C4	C5	C6	C1	0.8(2)	C24	C25	C30	C29	−178.07(12)
C6	C1	C2	C3	-0.21(19)	C25	C24	C31	N2	-0.43(14)
C7	C4	C5	C6	178.82(16)	C25	C26	C27	C28	0.9(2)
C8	C9	C10	C11	-0.89(19)	C26	C25	C30	N2	178.67(11)
C8	C13	C14	C15	-56.09(14)	C26	C25	C30	C29	-0.31(19)
C8	C13	C14	C23	−	C26	C27	C28	C29	-0.6(2)

				174.83(11)			
C8	C13	C14	C24	64.52(14)	C27	C28	C29	C30	-0.2(2)
C9	C8	C13	C12	1.65(17)	C28	C29	C30	N2	-178.11(13)
C9	C8	C13	C14	-177.19(11)	C28	C29	C30	C25	0.6(2)
C9	C10	C11	C12	0.6(2)	C30	N2	C31	C24	1.03(14)
C10	C11	C12	C13	0.8(2)	C30	C25	C26	C27	-0.47(19)
C11	C12	C13	C8	-1.95(19)	C31	N2	C30	C25	-1.19(14)
C11	C12	C13	C14	176.91(12)	C31	N2	C30	C29	177.69(13)
C12	C13	C14	C15	125.11(12)	C31	C24	C25	C26	-177.54(14)
C12	C13	C14	C23	6.37(16)	C31	C24	C25	C30	-0.30(13)

Table 8 Hydrogen Atom Coordinates (Å×10⁴) and Isotropic Displacement Parameters (Å²×10³) for 13.

Atom	*x*	*y*	*z*	U(eq)
H1	6676(12)	3411(17)	6097(8)	19
H2	5264(13)	3537(18)	3736(9)	25
H2A	6570	5560	4861	25
H3	7242	6060	3972	31
H5	9962	6589	5385	42
H6	9301	6124	6288	32
H7A	8901	5976	3656	76
H7B	9826	6757	4202	76
H7C	8843	7643	3807	76
H9	9121	3864	7098	26
H10	10369	2125	7340	33
H11	10004	-150	6864	35
H12	8411	-681	6137	31
H18	5436	3818	7049	25
H19	4385	4401	7697	28
H20	3050	2919	7680	28
H21	2753	846	7005	28
H22	3787	255	6346	25
H23A	6746	-1255	5834	38

H23B	7145	-902	5195	38
H23C	5977	-826	5096	38
H26	8473	758	4934	30
H27	9060	1127	3993	39
H28	8160	2492	3038	40
H29	6624	3491	2990	33
H31	5170	2707	4833	22

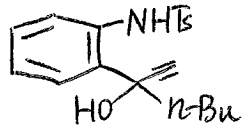
20140820.LiH-n-Bu

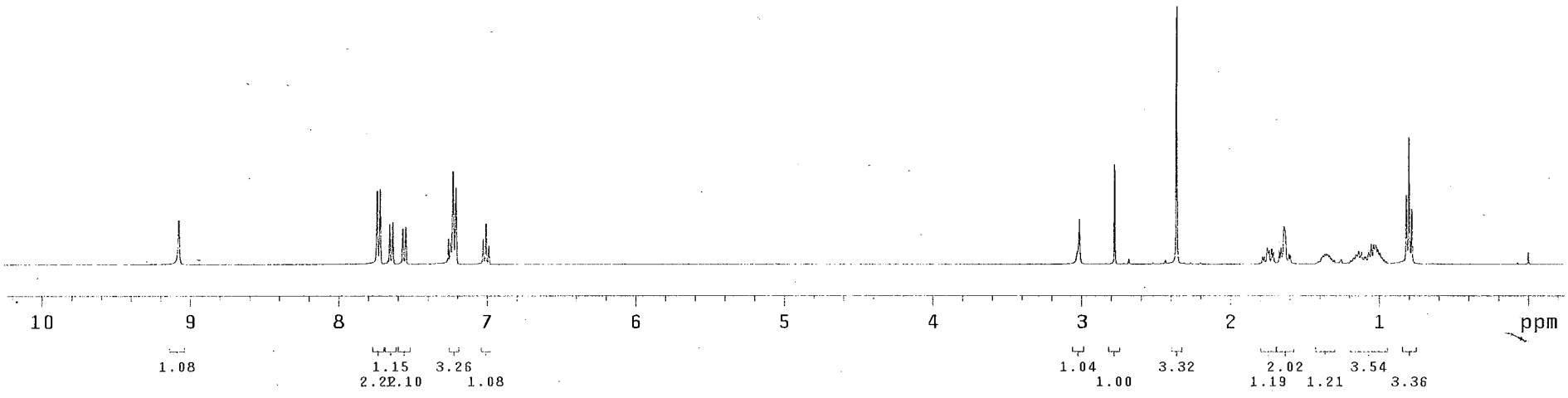
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: 20140820.LiH-n-Bu

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
32 repetitions
OBSERVE H1, 399.7865233 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 51 sec





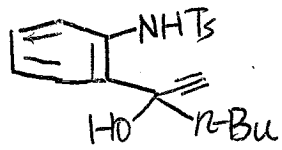
LiH-I-45_24Oct2013-19:01:21

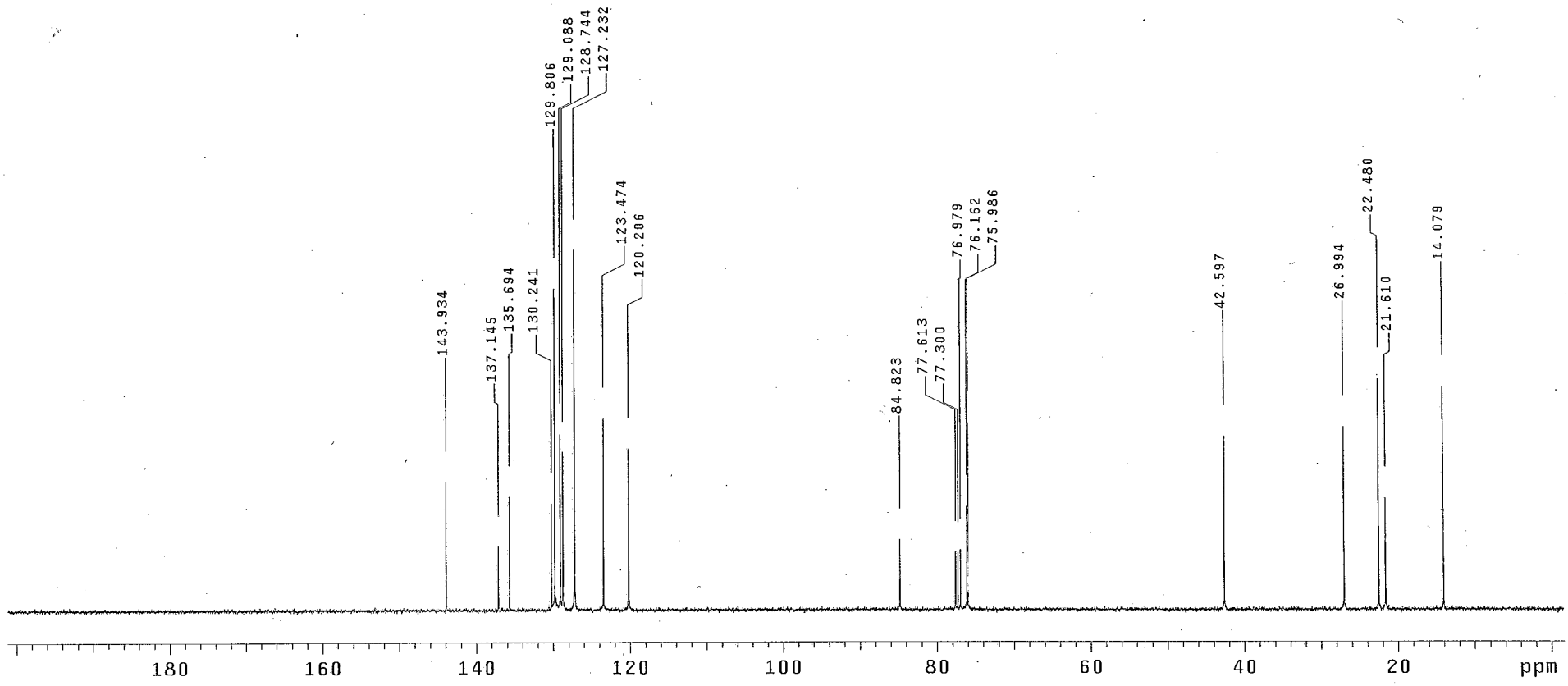
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-45_24Oct2013-19:01:21

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
128 repetitions
OBSERVE C13, 100.5263859 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 4 min, 42 sec





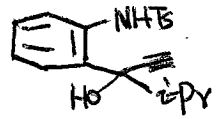
LiH-I-44_29Oct2013

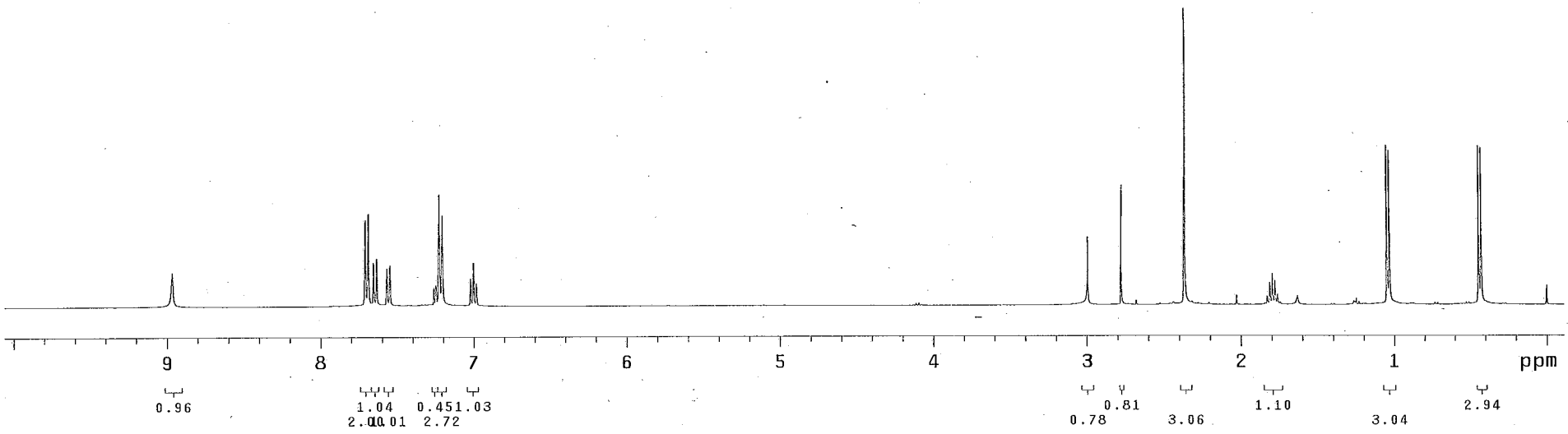
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-44_29Oct2013

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
8 repetitions
OBSERVE H1, 399.7865227 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 57 sec





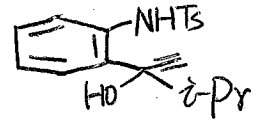
LiH-I-44_29Oct2013-16:58:01

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-44_29Oct2013-16:58:01

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
256 repetitions
OBSERVE C13, 100.5263736 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 9 min, 24 sec



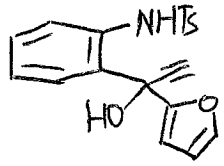
20140820.LiH-furan

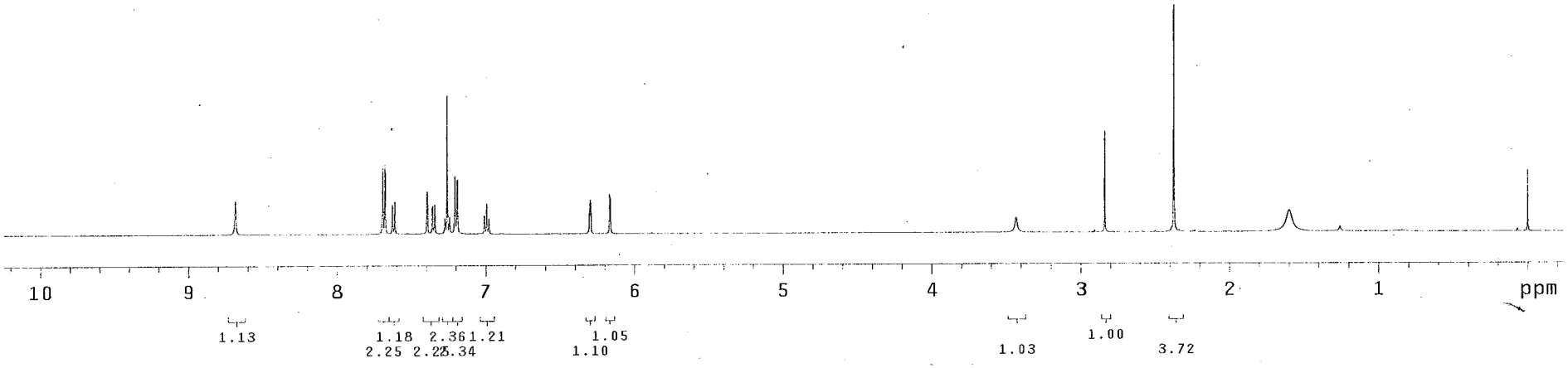
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: 20140820.LiH-furan

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7288173 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 32 sec





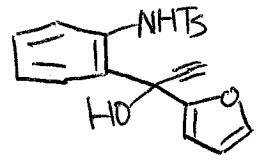
LiH-I-59_29Oct2013-16:31:29

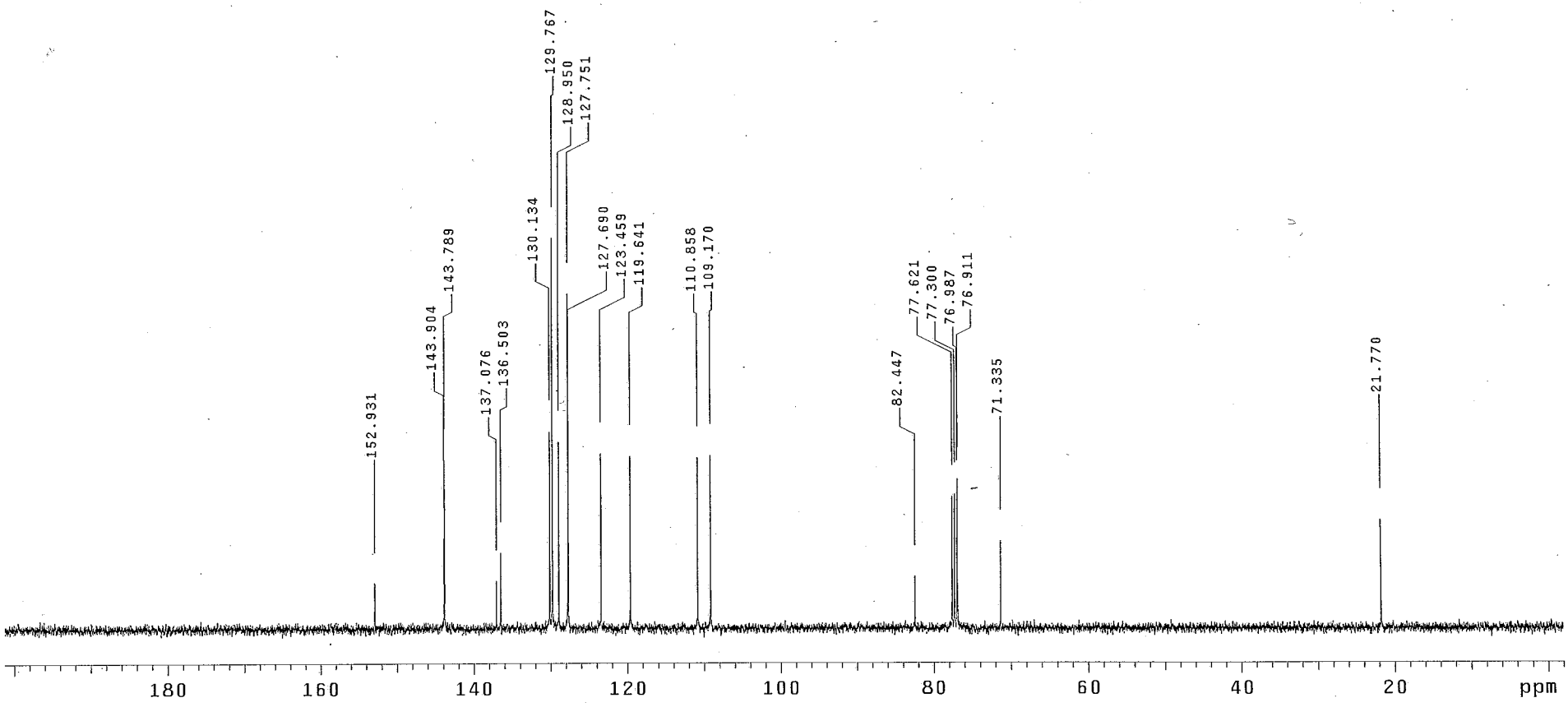
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-59_29Oct2013-16:31:29

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
256 repetitions
OBSERVE C13, 100.5263752 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 9 min, 24 sec





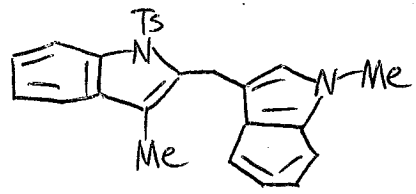
LiH-II-33_20May2014

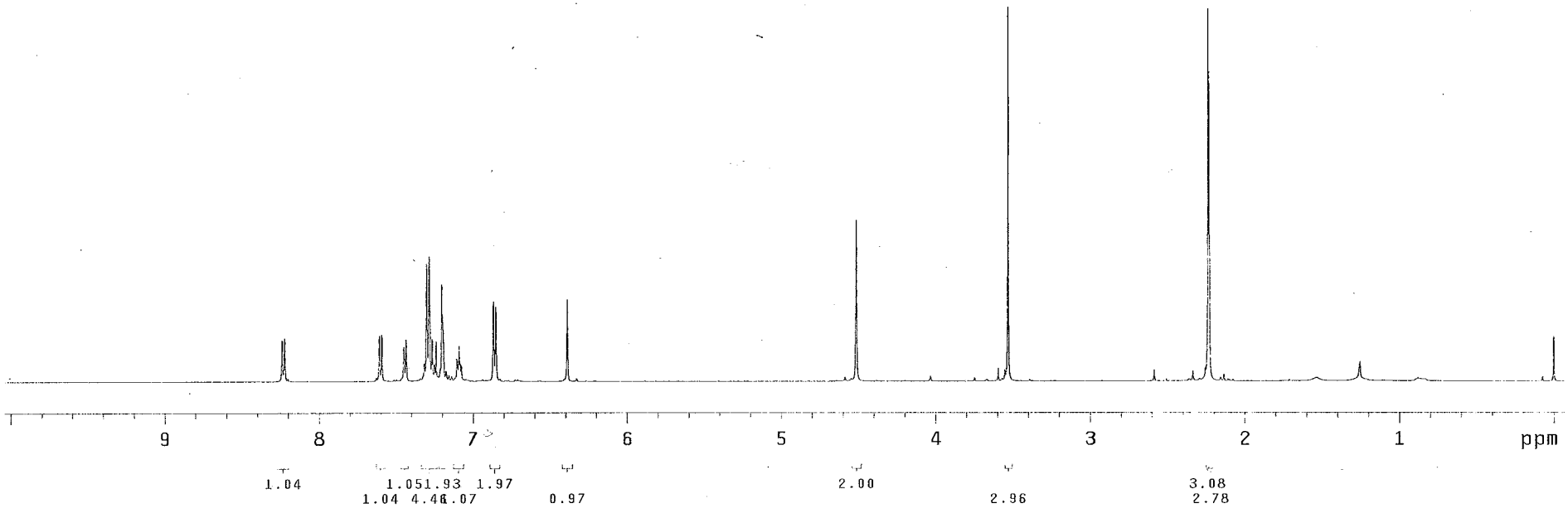
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-33_20May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7288268 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min, 38 sec





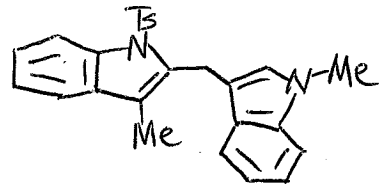
LiH-II-33_21May2014

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-33_21May2014
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "ui500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
467 repetitions
OBSERVE C13, 125.6568765 MHz
DECOUPLE H1, 499.7312897 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 19 min, 46 sec



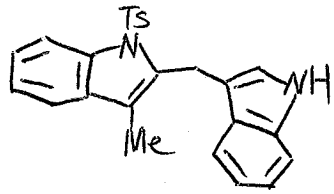
LiH-II-32_01Jun2014

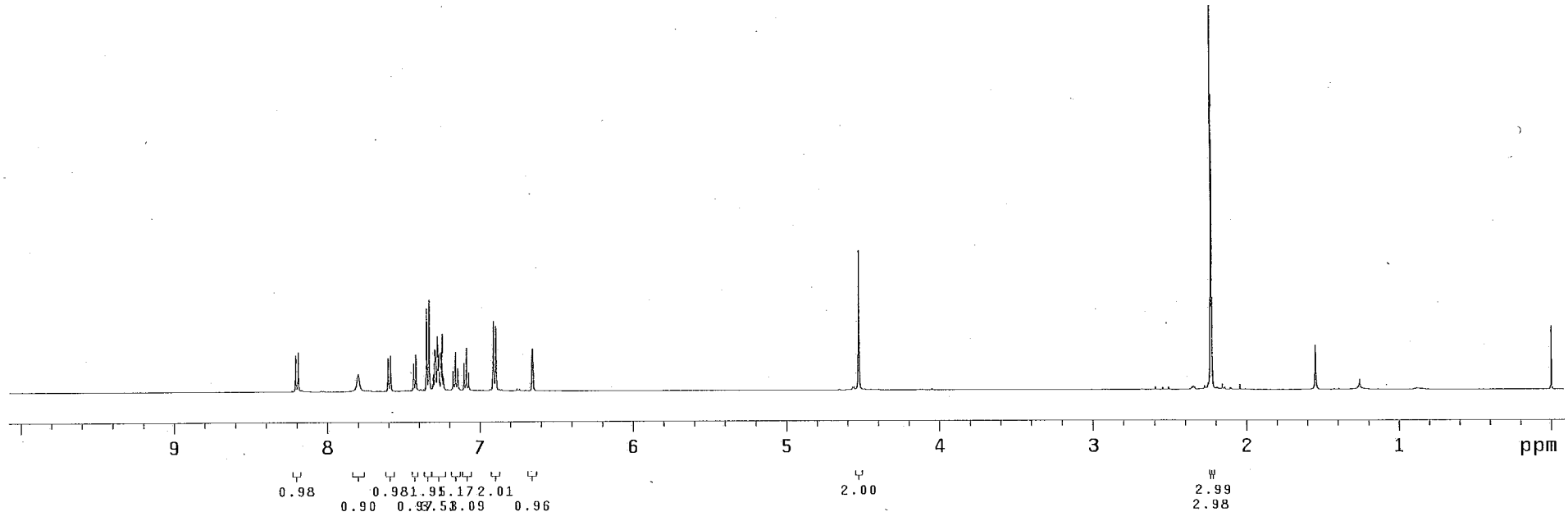
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-32_01Jun2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7288239 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 32 sec





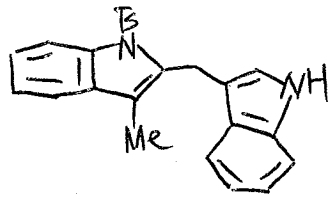
LiH-II-32_01Jun2014-17:12:16

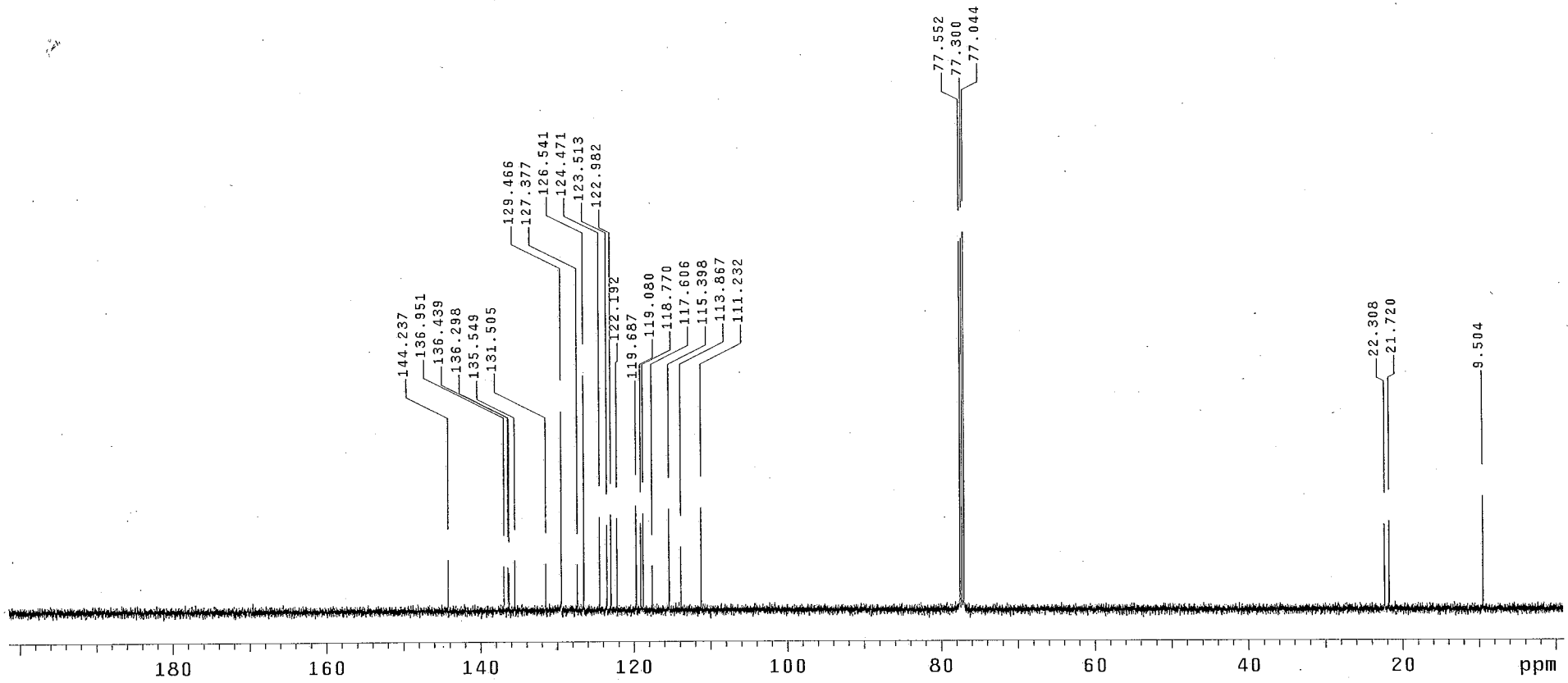
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-32_01Jun2014-17:12:16

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
248 repetitions
OBSERVE C13, 125.6568760 MHz
DECOUPLE H1, 499.7312897 MHz
Power 46 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 19 min, 46 sec





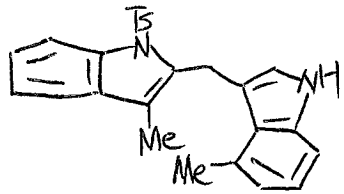
LiH-II-40p_05Jun2014

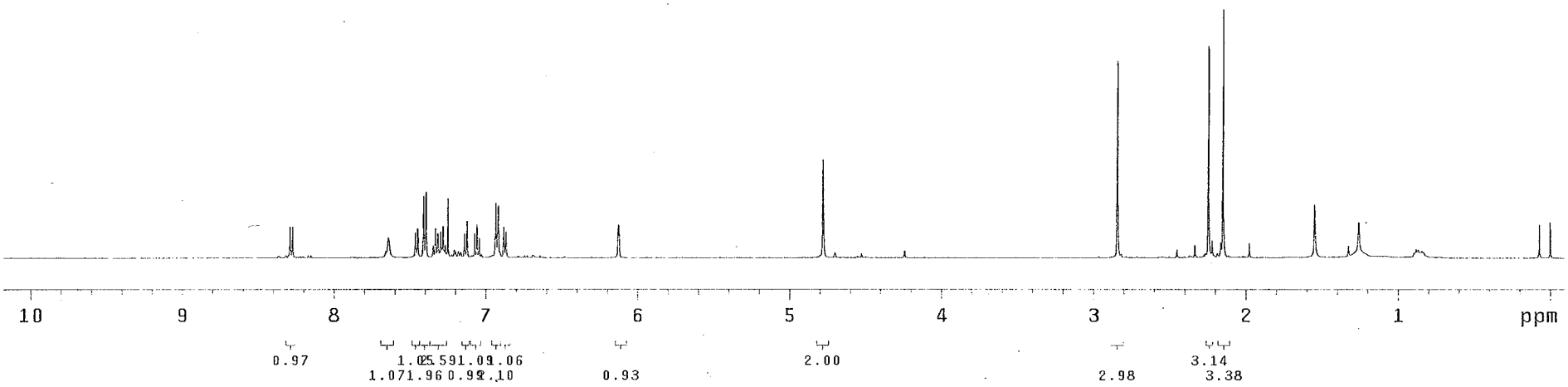
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-40p_05Jun2014
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "ui500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7288217 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 32 sec





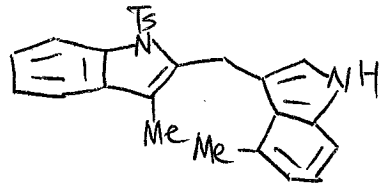
LiH-II-40p_05Jun2014-16:15:37

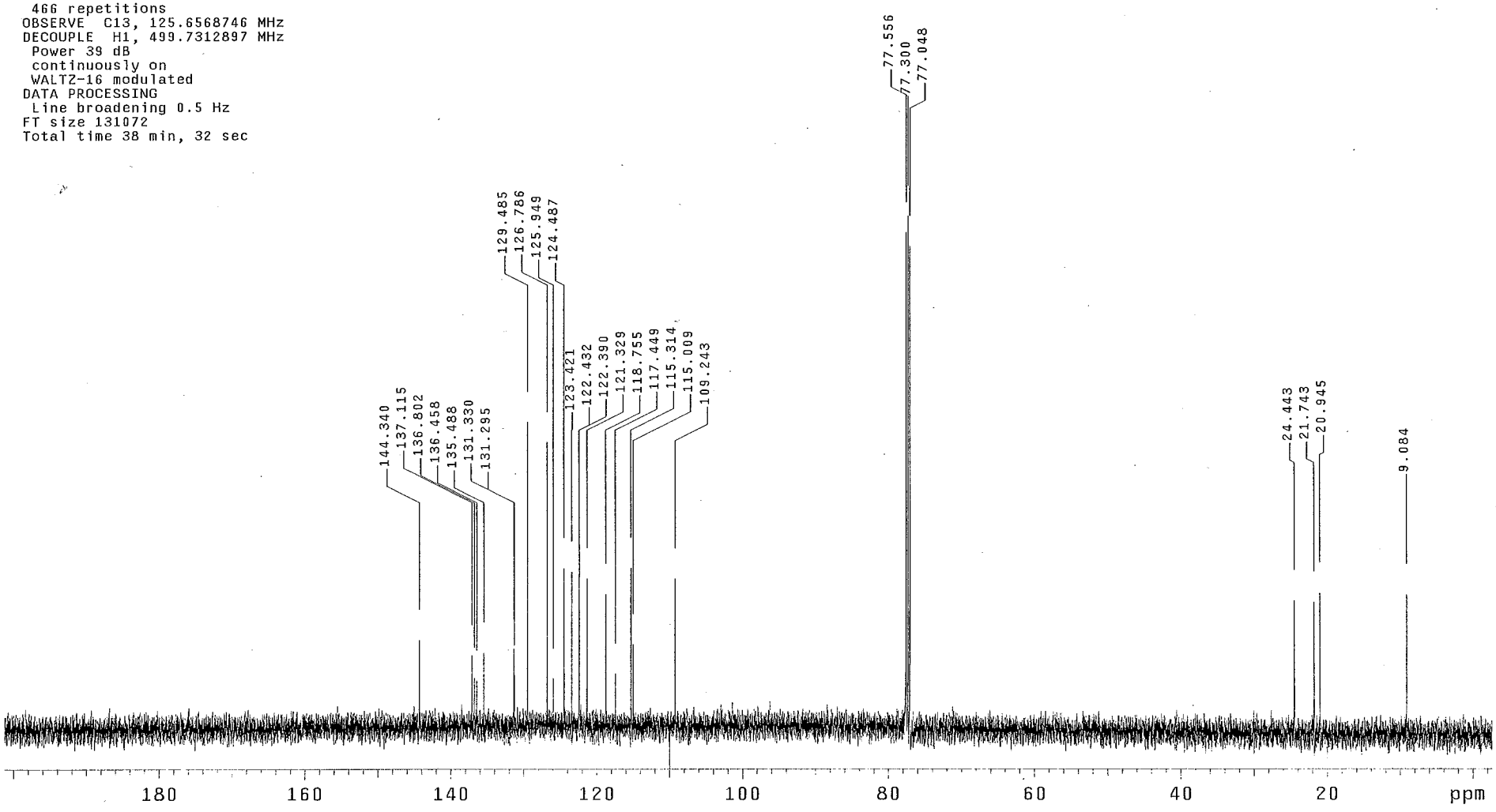
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-40p_05Jun2014-16:15:37
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "ui500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
466 repetitions
OBSERVE C13, 125.6568746 MHz
DECOUPLE H1, 499.7312897 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 32 sec





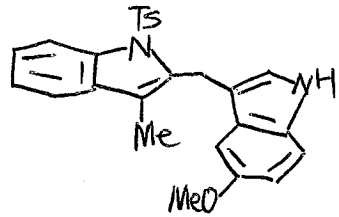
LiH-II-13p_05Jun2014

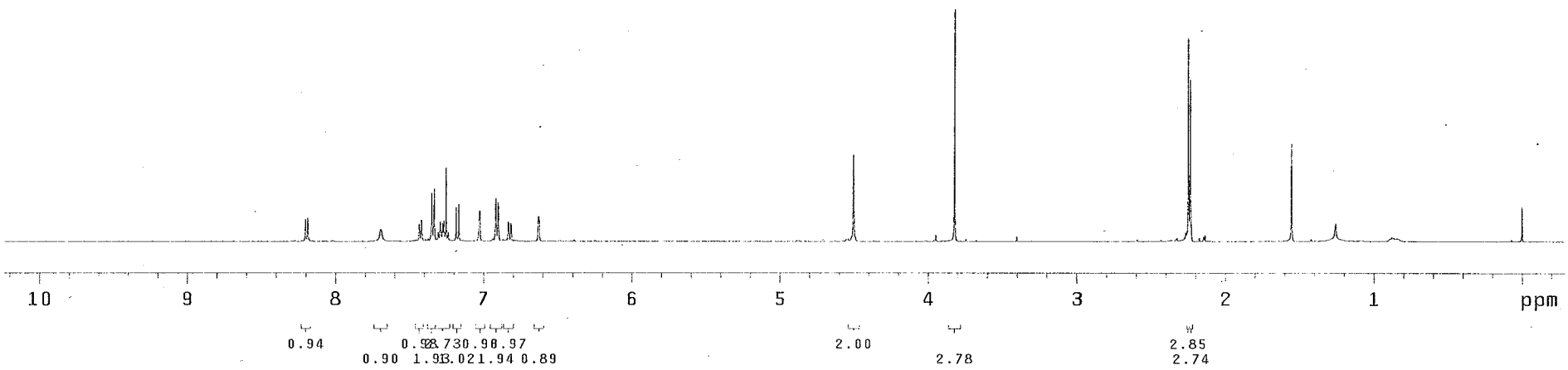
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-13p_05Jun2014
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "ui500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7288202 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 32 sec





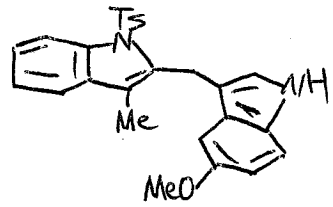
LiH-I-269-2_24Apr2014

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-269-2_24Apr2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
965 repetitions
OBSERVE C13, 100.5263721 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 36 min, 45 sec



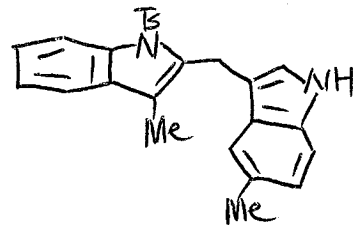
why-2-250-p-proton-2_09Jul2014

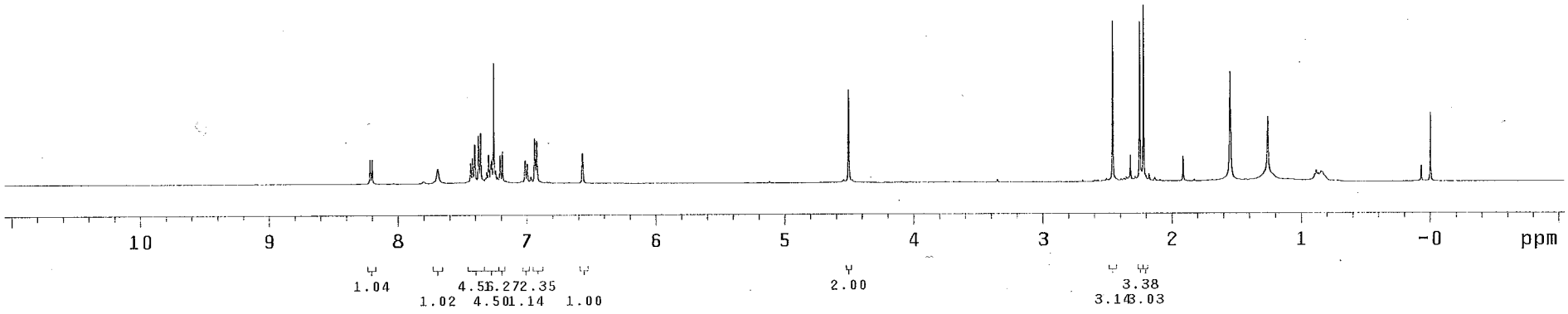
Archive directory: /export/home/haoyuan/vnmrsys/data
Sample directory: why-2-250-p-proton-2_09Jul2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7288205 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 32 sec





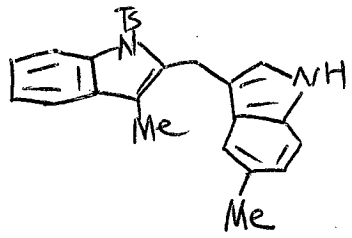
why-2-250-p-carbon_06Jun2014

Archive directory: /export/home/haoyuan/vnmrsys/data
Sample directory: why-2-250-p-carbon_06Jun2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
900 repetitions
OBSERVE C13, 125.6568744 MHz
DECOUPLE H1, 499.7312897 MHz
Power 46 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 3 hr, 12 min, 23 sec



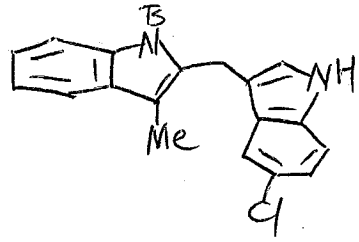
LiH-I-269-3_23Apr2014

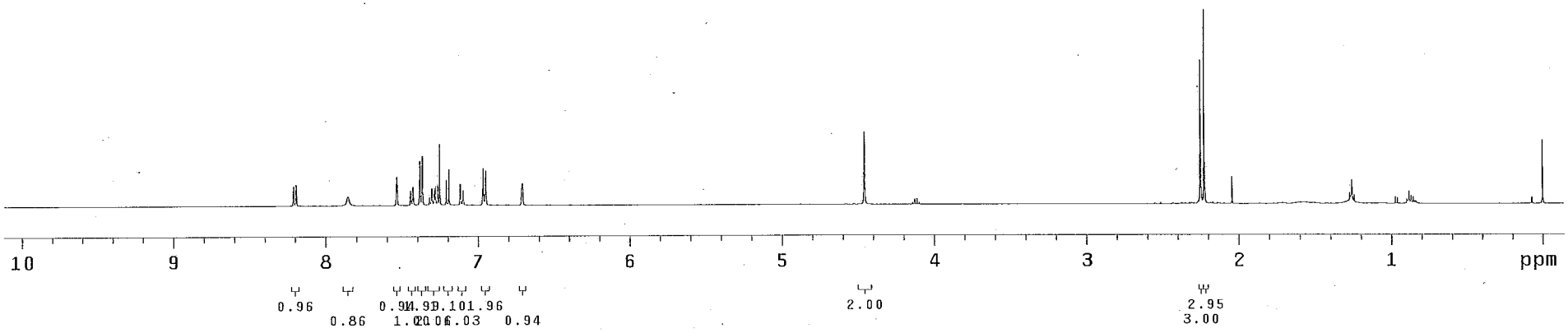
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-269-3_23Apr2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7288200 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min, 38 sec





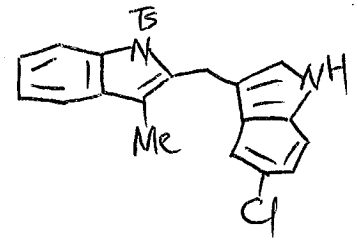
LiH-I-269-3_23Apr2014

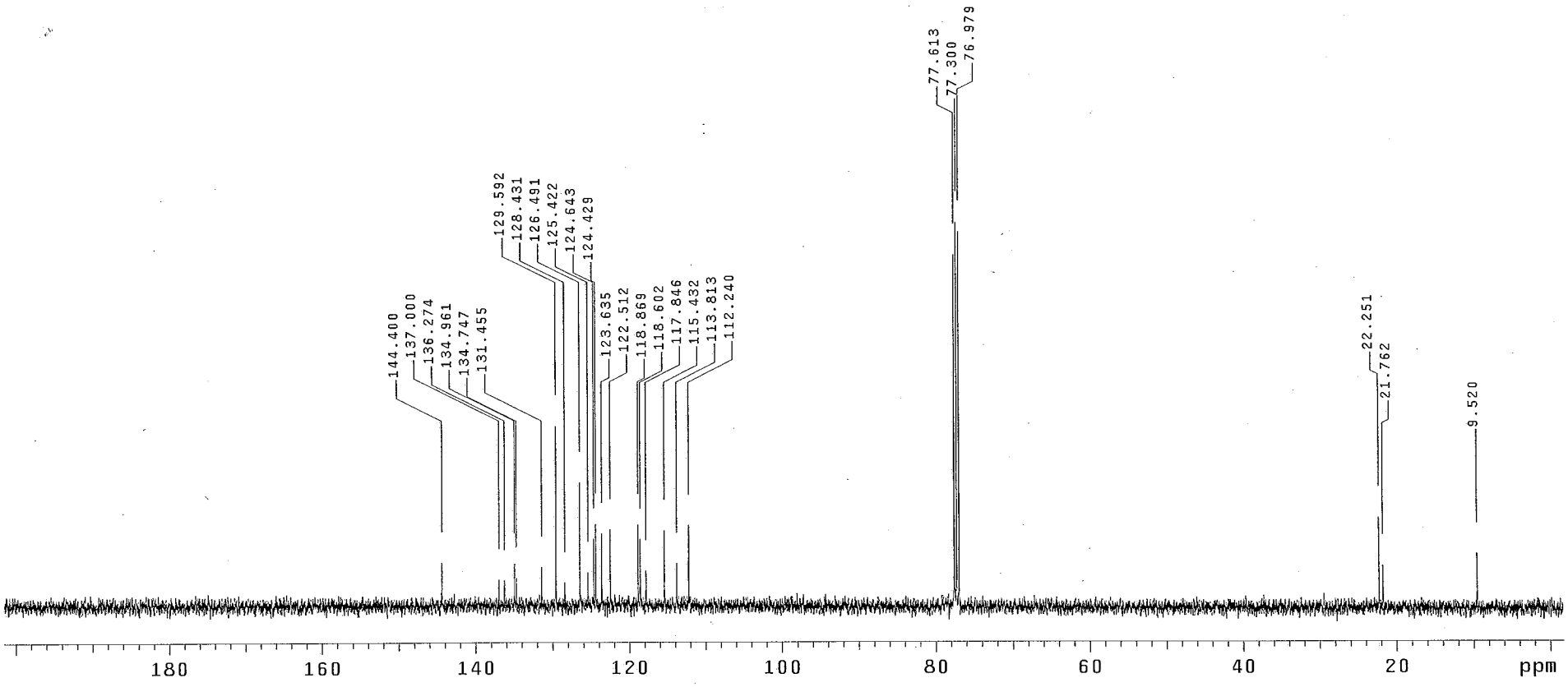
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-269-3_23Apr2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
472 repetitions
OBSERVE C13, 100.5263721 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 18 min, 49 sec





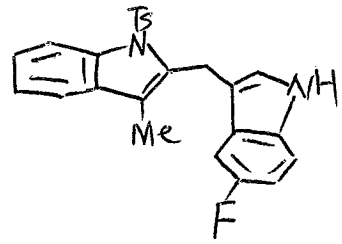
GH-48-1-2_09Jun2014

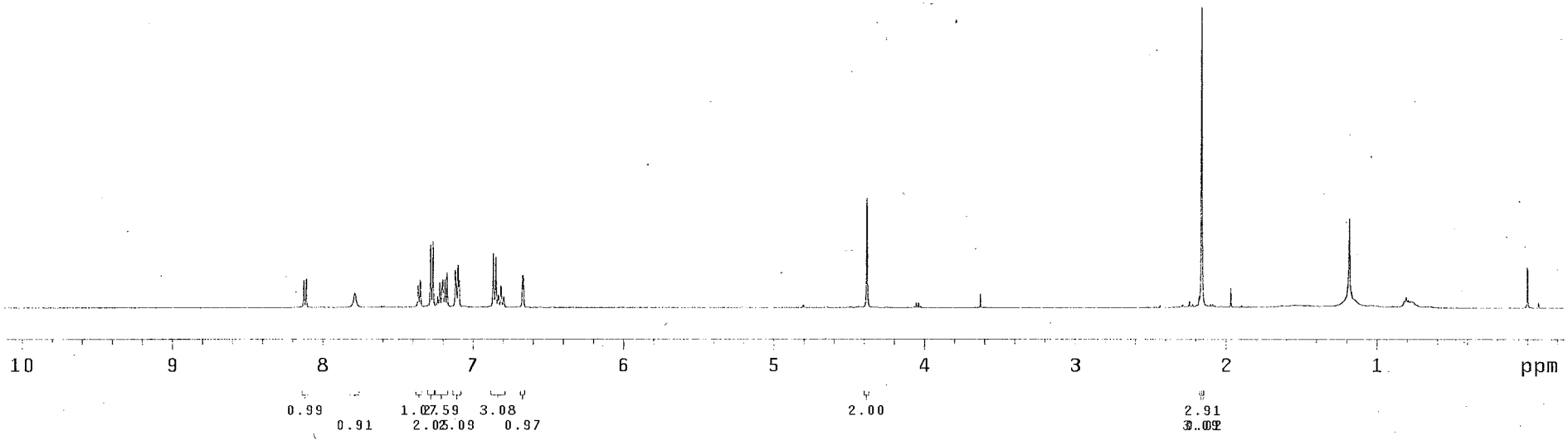
Archive directory: /export/home/gabby/vnmrsys/data
Sample directory: GH-48-1-2_09Jun2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7288598 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 16 sec





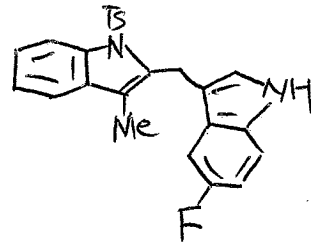
JG-I-48-3C_10Jul2014

Archive directory: /export/home/gabby/vnmrsys/data
Sample directory: JG-I-48-3C_10Jul2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
512 repetitions
OBSERVE C13, 100.5263721 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 18 min, 49 sec



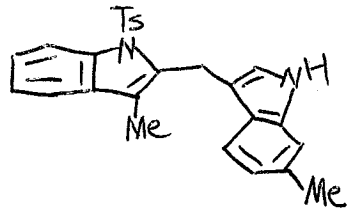
LiH-II-39p_05Jun2014

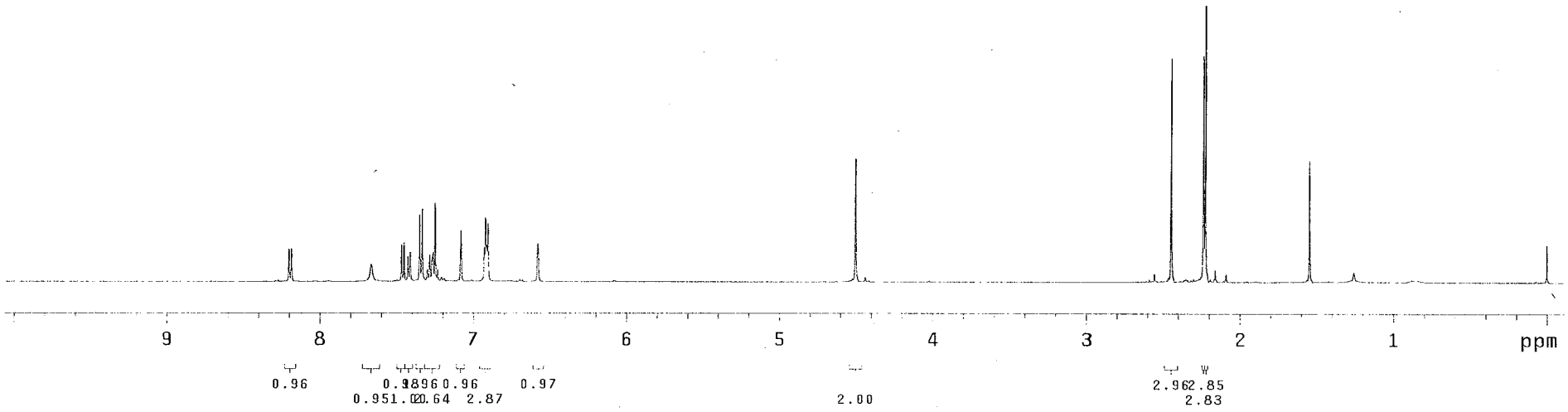
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-39p_05Jun2014
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-500 "ui500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7288217 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 32 sec





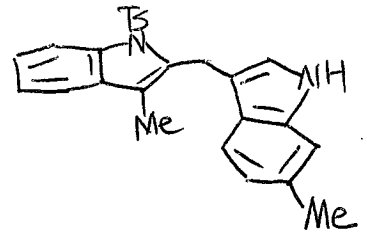
LiH-II-39p_05Jun2014-15:36:42

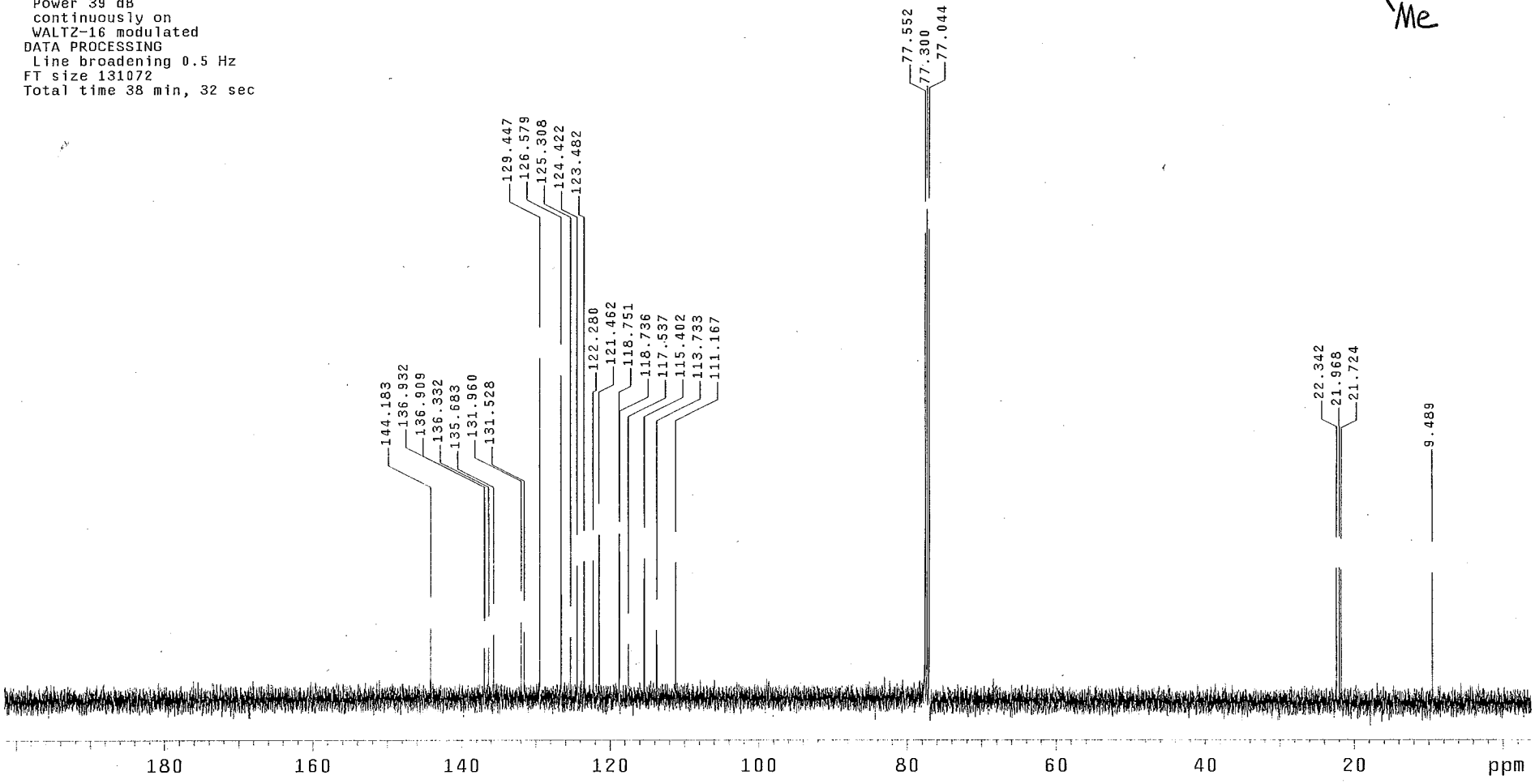
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-39p_05Jun2014-15:36:42
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
INOVA-500 "ui500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
535 repetitions
OBSERVE C13, 125.6568746 MHz
DECOUPLE H1, 499.7312897 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 38 min, 32 sec





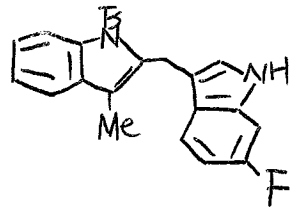
LiH-II-47p_27Jun2014

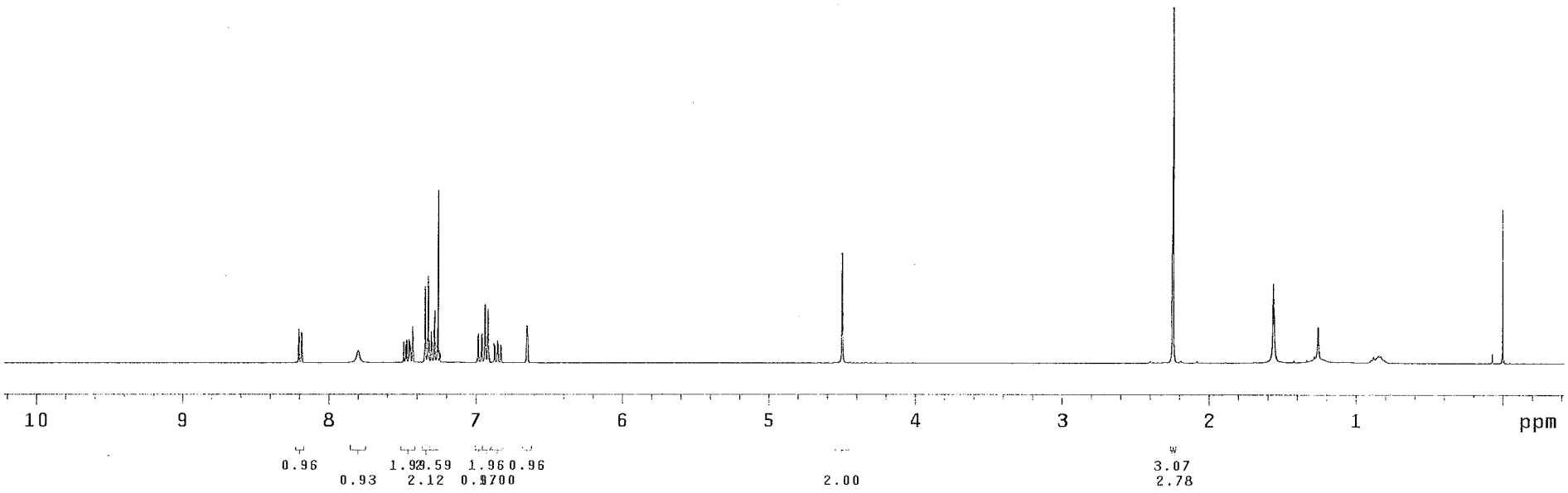
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-47p_27Jun2014
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "ui400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
16 repetitions
OBSERVE H1, 399.7865249 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 35 sec





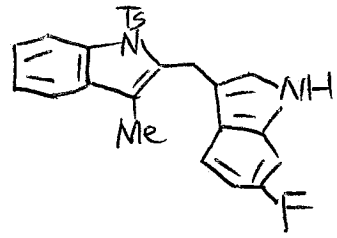
LiH-II-47p_25Jun2014-13:42:22

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-47p_25Jun2014-13:42:22

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
1679 repetitions
OBSERVE C13, 100.5263713 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 13 min, 31 sec



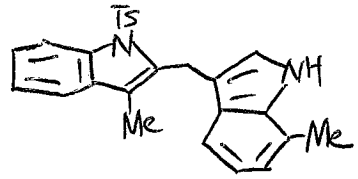
why-2-249-p-proton_09Jul2014

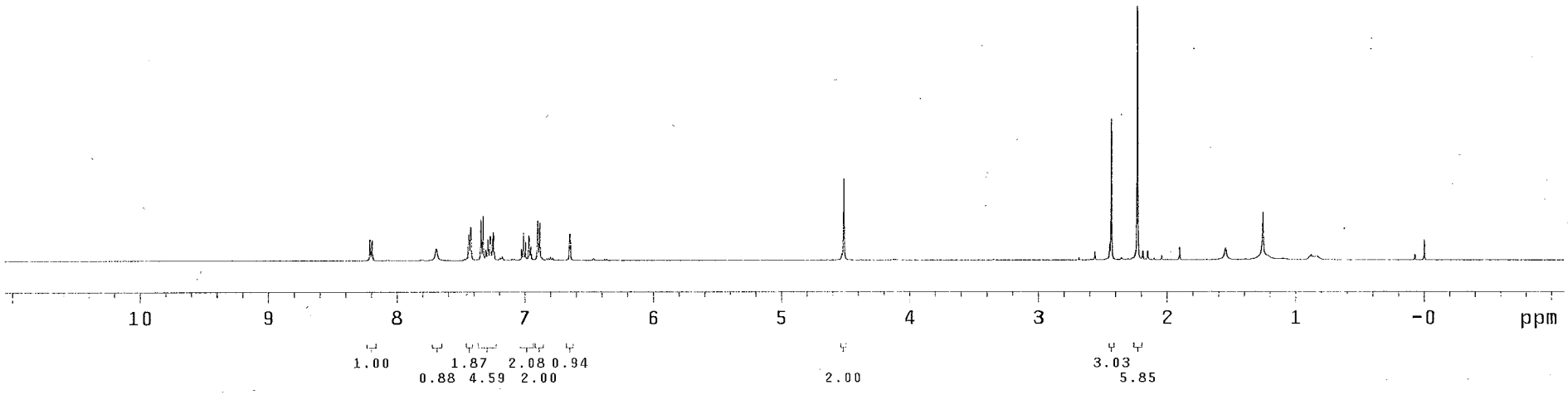
Archive directory: /export/home/haoyuan/vnmrsys/data
Sample directory: why-2-249-p-proton_09Jul2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
32 repetitions
OBSERVE H1, 499.7288256 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 32 sec





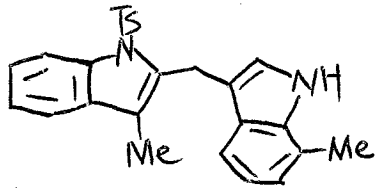
LiH-WHY-249_10Jul2014

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-WHY-249_10Jul2014
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "ui400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
1474 repetitions
OBSERVE C13, 100.5263713 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 13 min, 31 sec



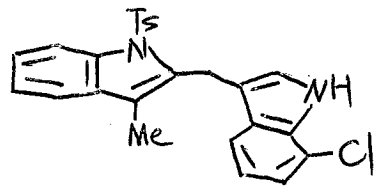
LiH-II-41p_06Jun2014

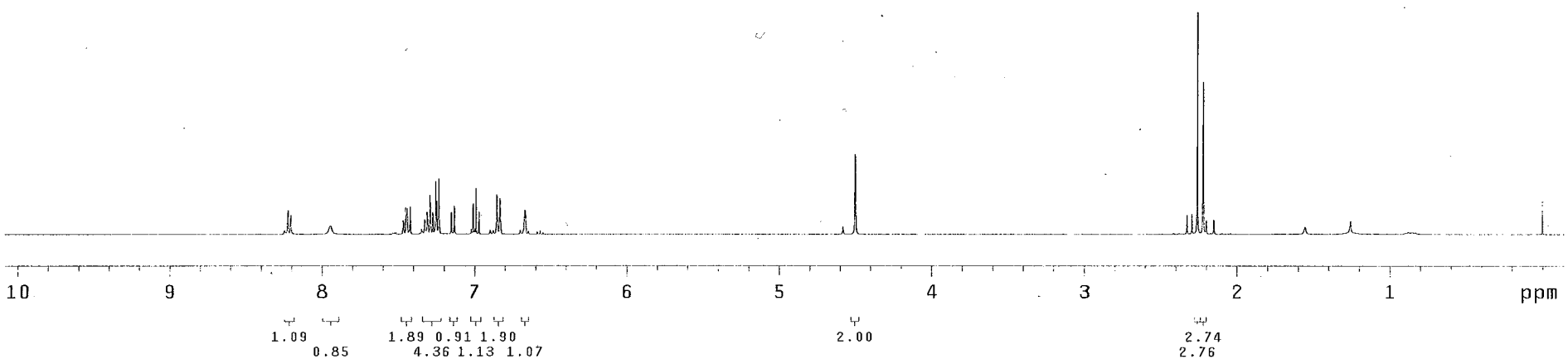
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-41p_06Jun2014
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "ui400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
32 repetitions
OBSERVE H1, 399.7865296 MHz
DATA PROCESSING
FT size 65536
Total time 2 min, 51 sec





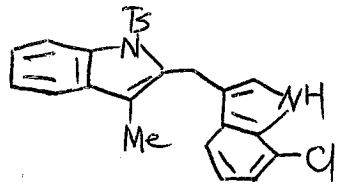
LiH-II-41p_06Jun2014-14:30:54

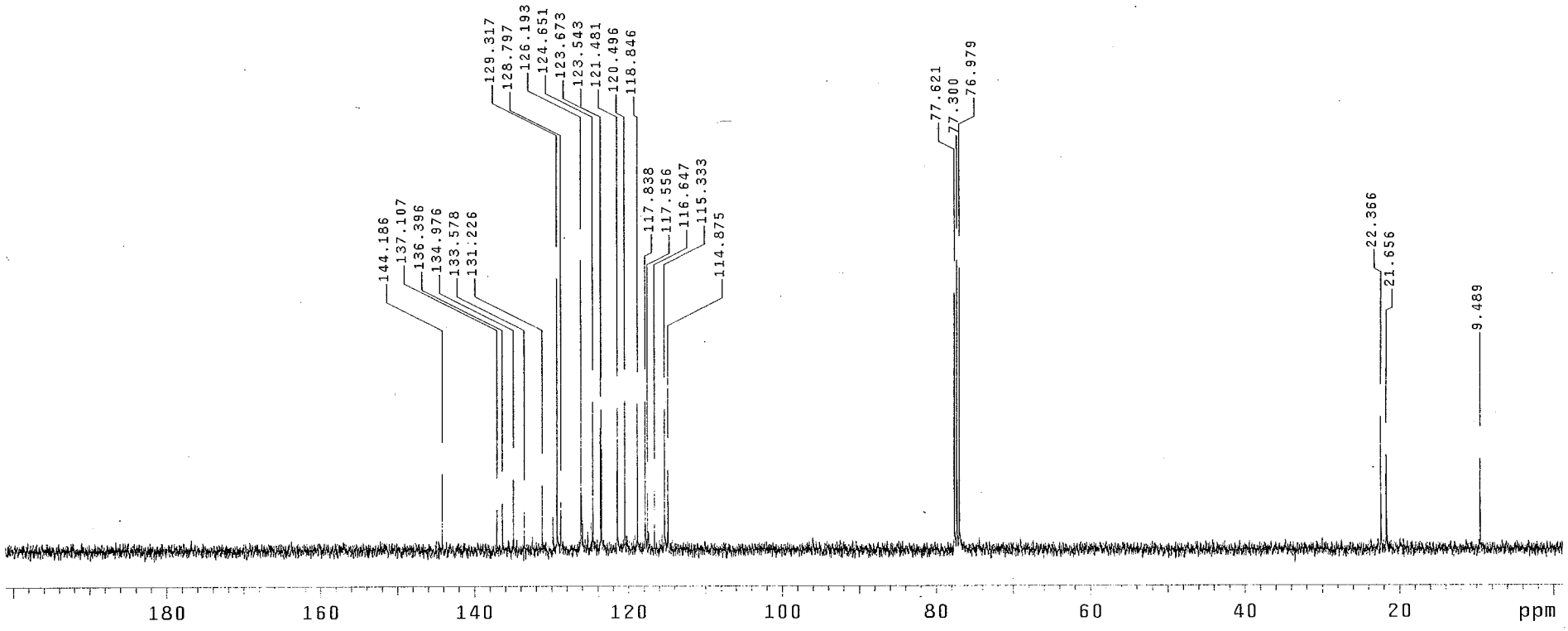
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-41p_06Jun2014-14:30:54

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
341 repetitions
OBSERVE C13, 100.5263736 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 18 min, 49 sec





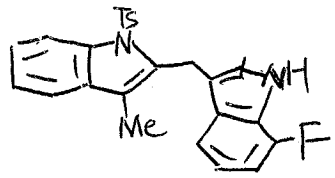
LiH-II-25_15May2014

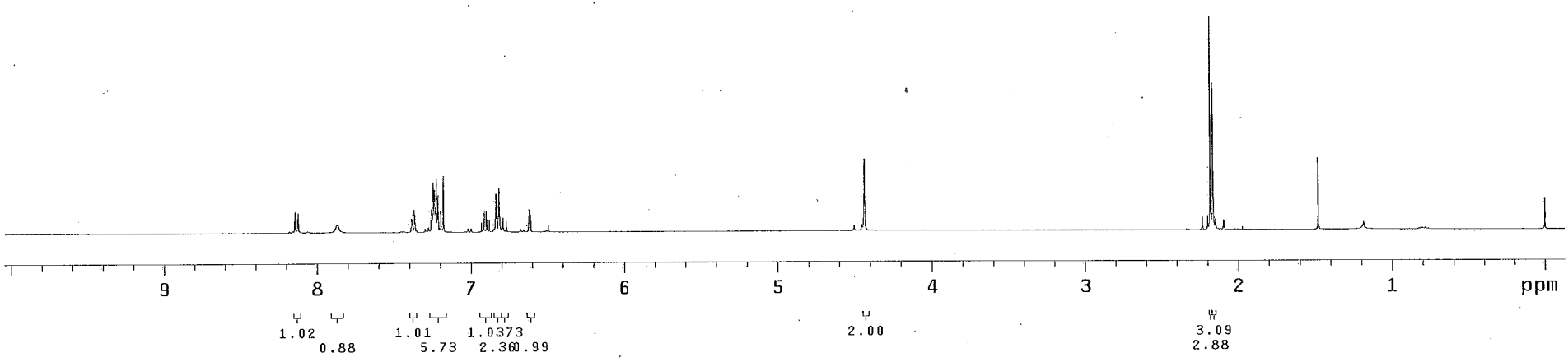
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-25_15May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
8 repetitions
OBSERVE H1, 399.7865555 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 57 sec





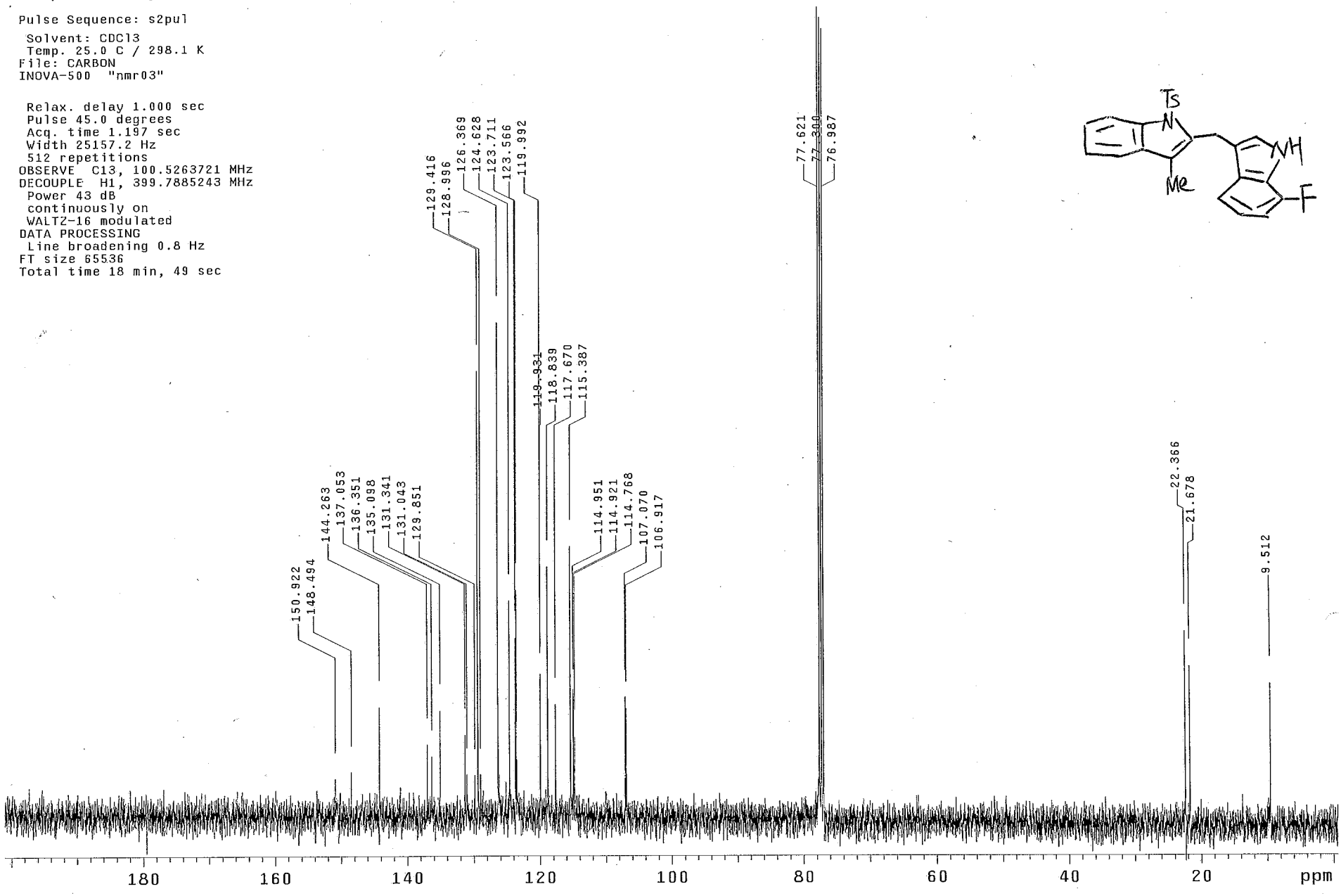
LiH-II-25_15May2014-12:38:26

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-25_15May2014-12:38:26

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
512 repetitions
OBSERVE C13, 100.5263721 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 18 min, 49 sec



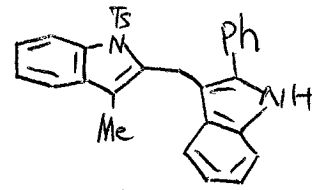
LiH-II-44_20Jun2014

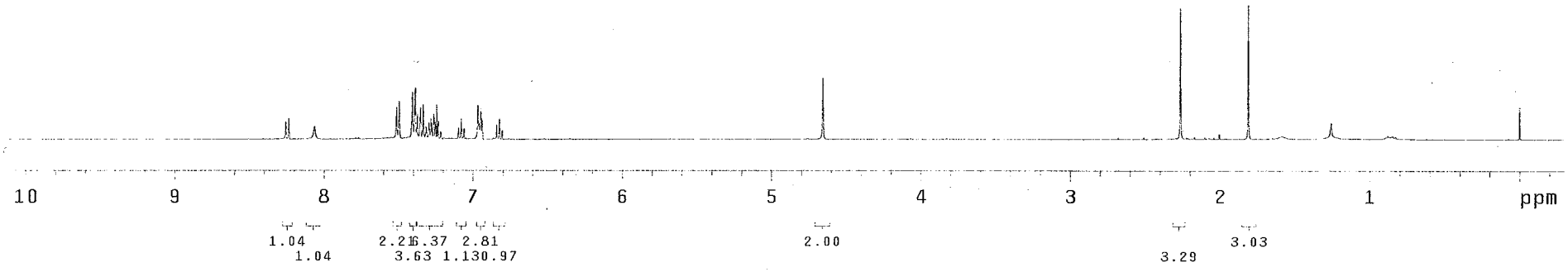
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-44_20Jun2014
File: PROTON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "ui400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
16 repetitions
OBSERVE H1, 399.7865268 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 35 sec





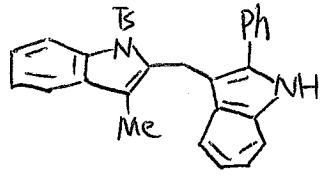
LiH-II-44_20Jun2014-11:39:07

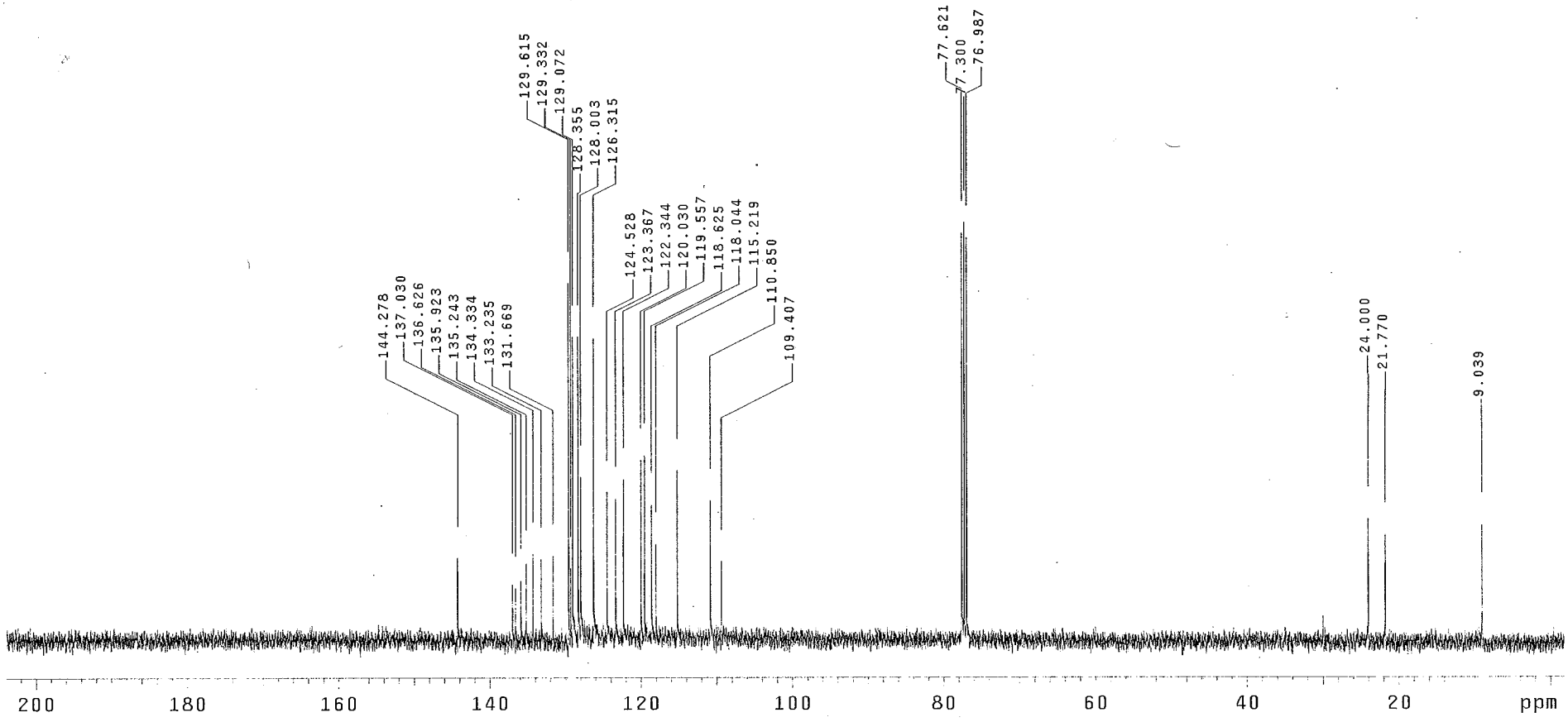
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-44_20Jun2014-11:39:07
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "ui400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
356 repetitions
OBSERVE C13, 100.5263729 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 36 min, 45 sec





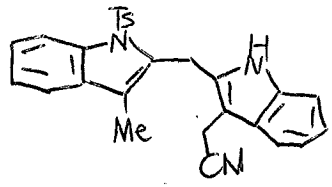
LiH-II-48_20Jun2014

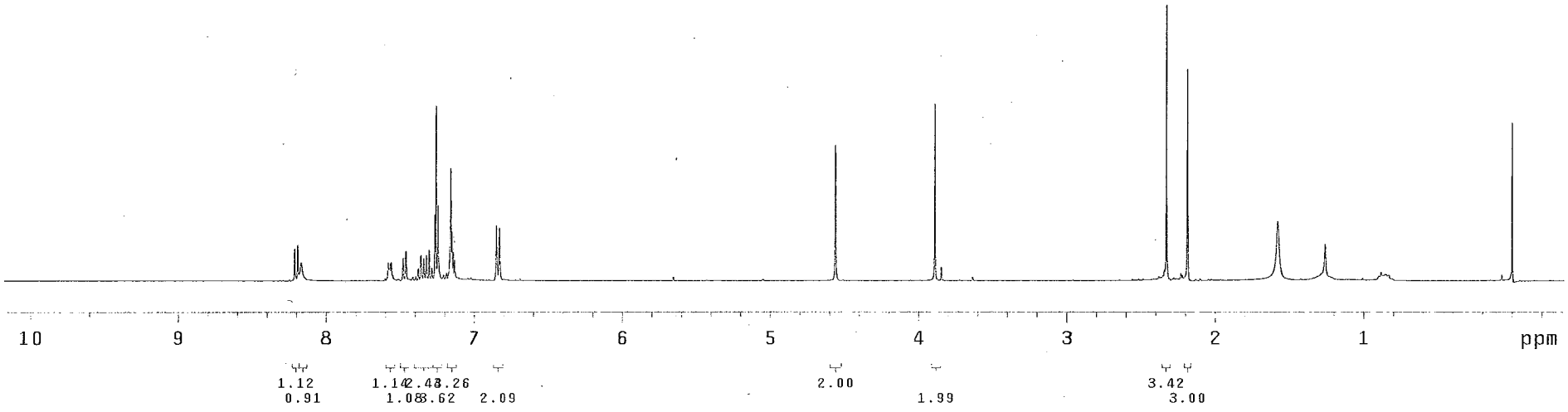
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-48_20Jun2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
16 repetitions
OBSERVE H1, 399.7865214 MHz
DATA PROCESSING
FT size 65536
Total time 1 min, 35 sec





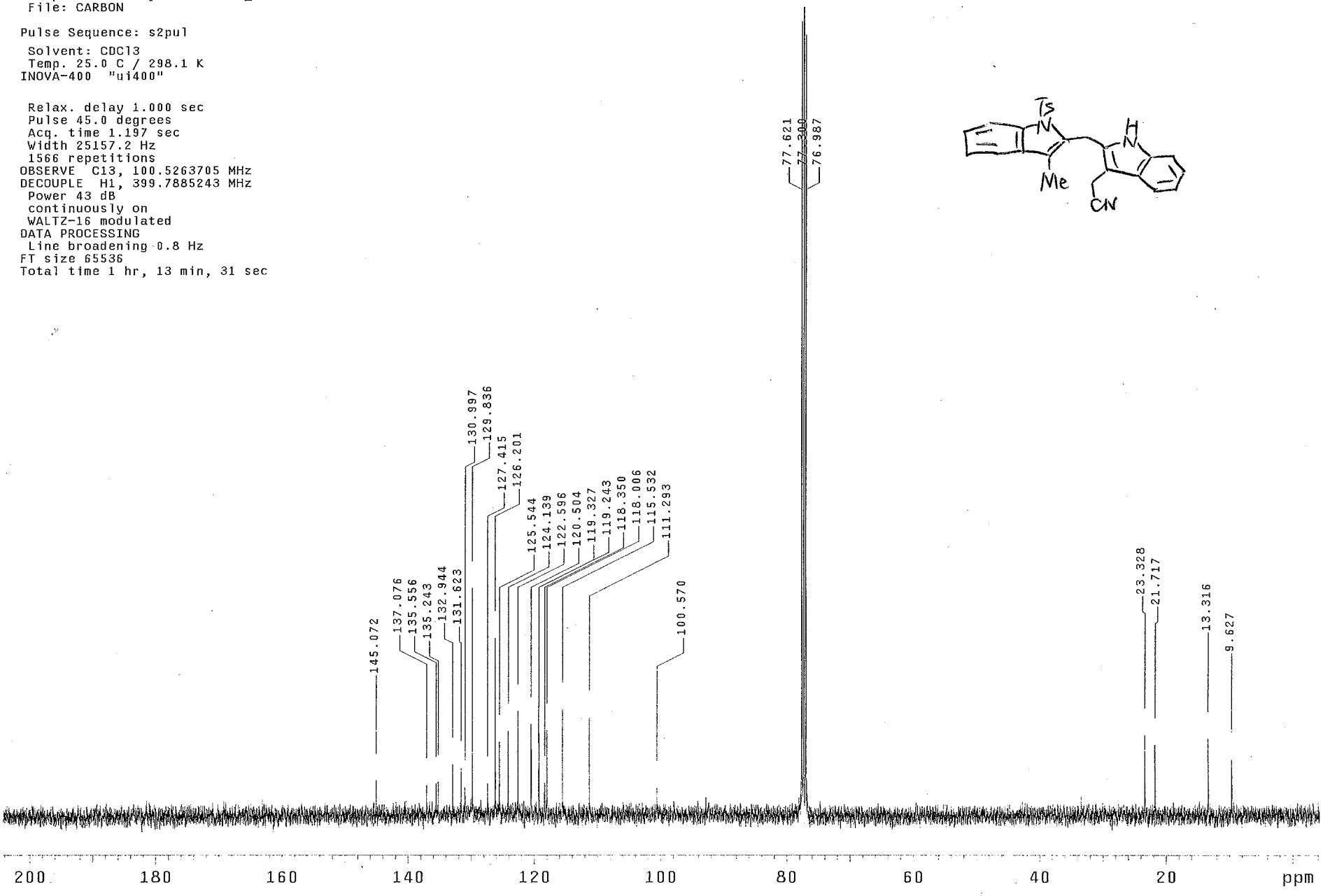
LiH-II-48_20Jun2014-13:56:16

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-48_20Jun2014-13:56:16
File: CARBON

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
INOVA-400 "ui400"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
1566 repetitions
OBSERVE C13, 100.5263705 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 1 hr, 13 min, 31 sec



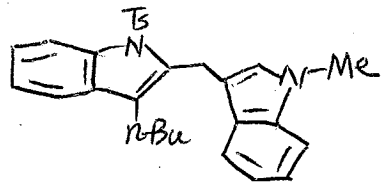
LiH-I-296-1_07May2014

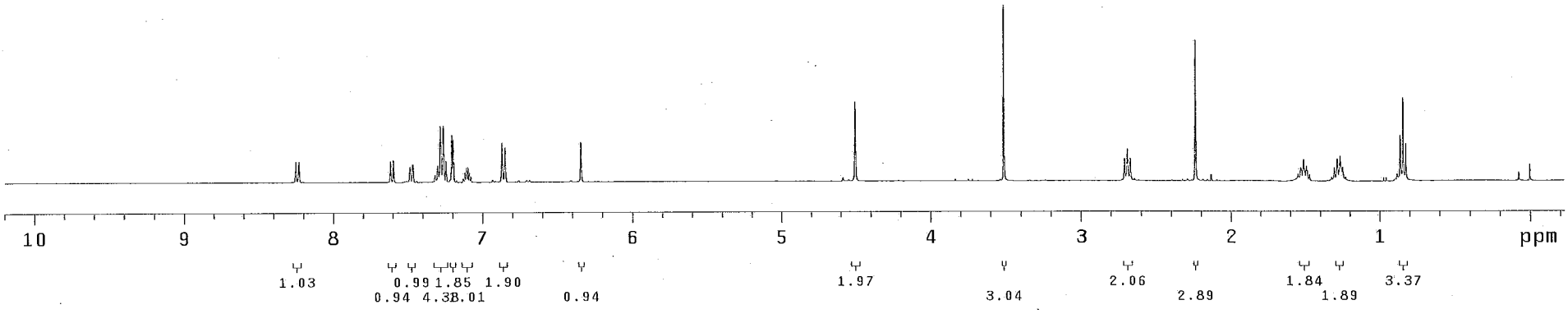
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-296-1_07May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
8 repetitions
OBSERVE H1, 399.7865300 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 57 sec





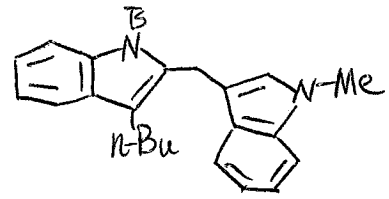
LiH-I-296-1_07May2014-16:14:37

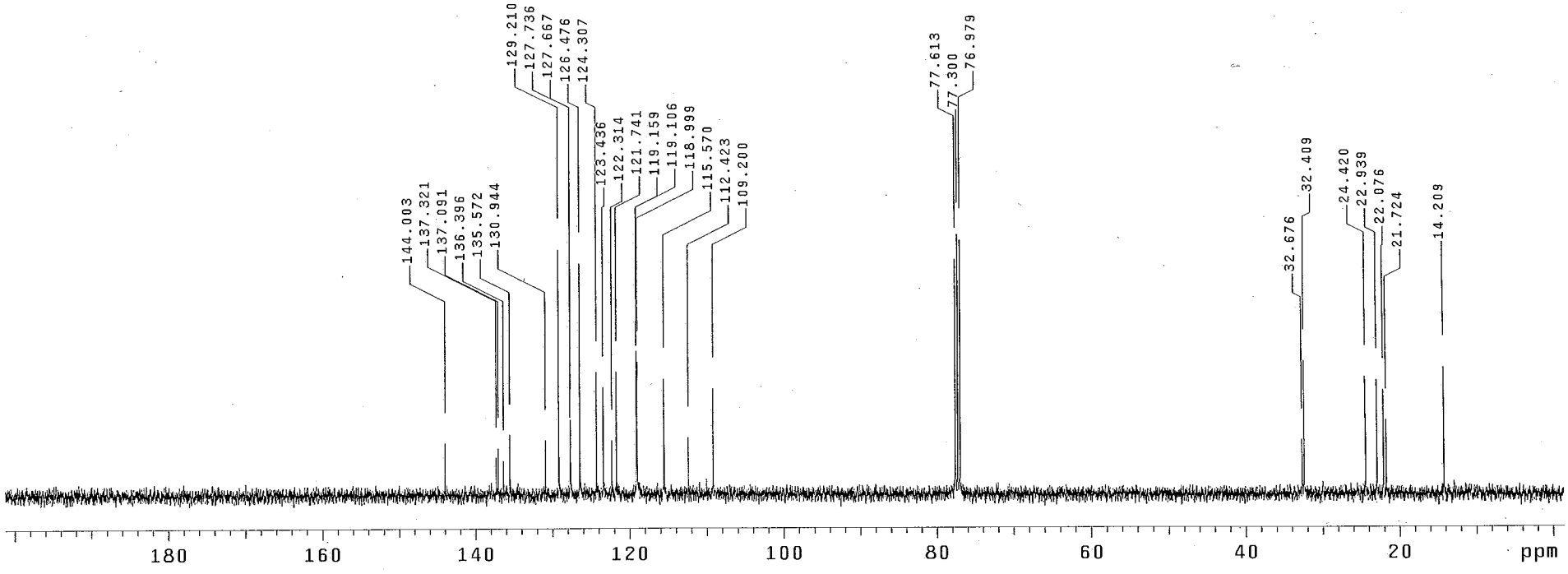
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-296-1_07May2014-16:14:37

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
256 repetitions
OBSERVE C13, 100.5263736 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 9 min, 24 sec





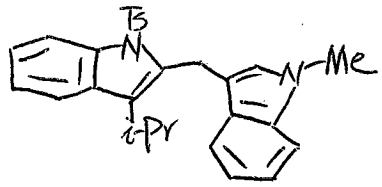
LiH-I-297_08May2014

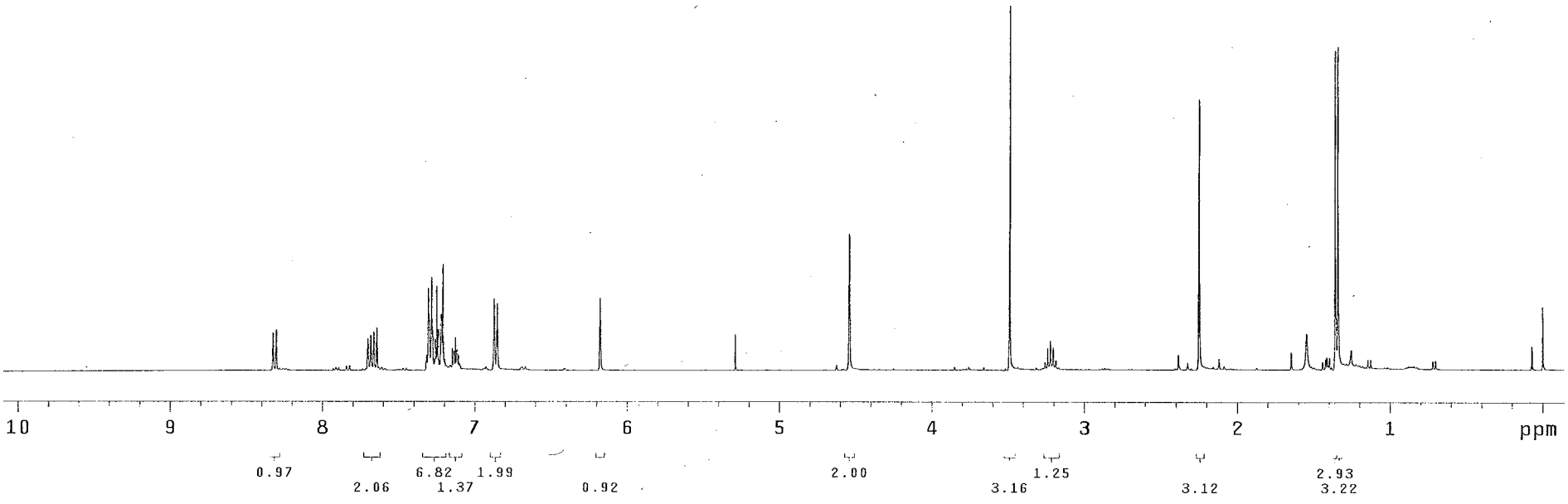
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-297_08May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
8 repetitions
OBSERVE H1, 399.7865274 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 57 sec





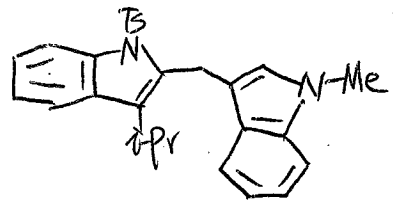
LiH-I-297_07May2014-17:30:06

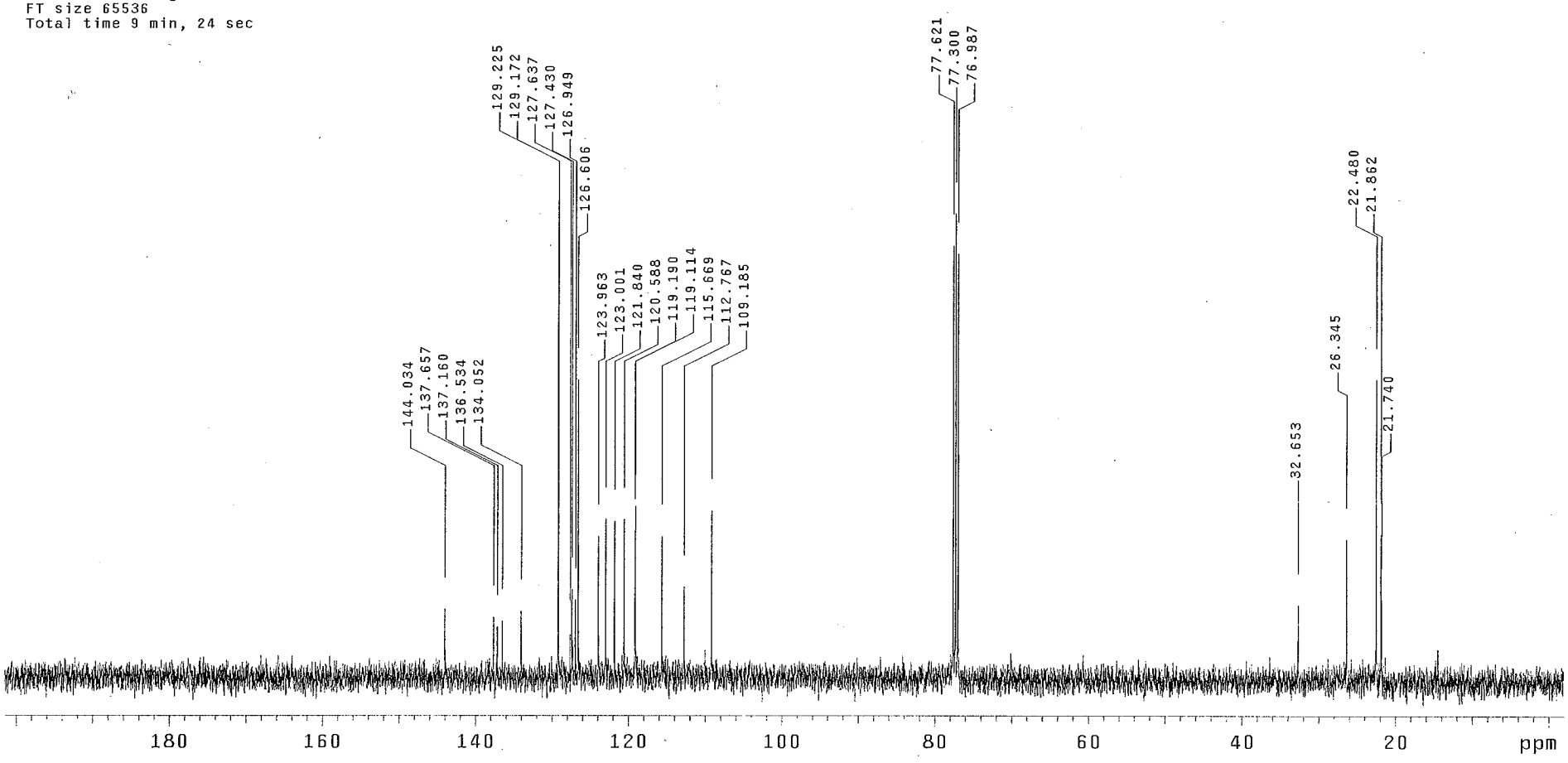
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-297_07May2014-17:30:06

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
256 repetitions
OBSERVE C13, 100.5263729 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 9 min, 24 sec





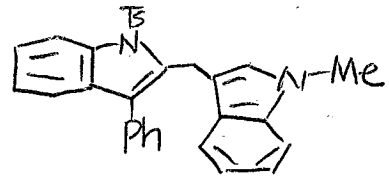
LiH-I-299-1_07May2014

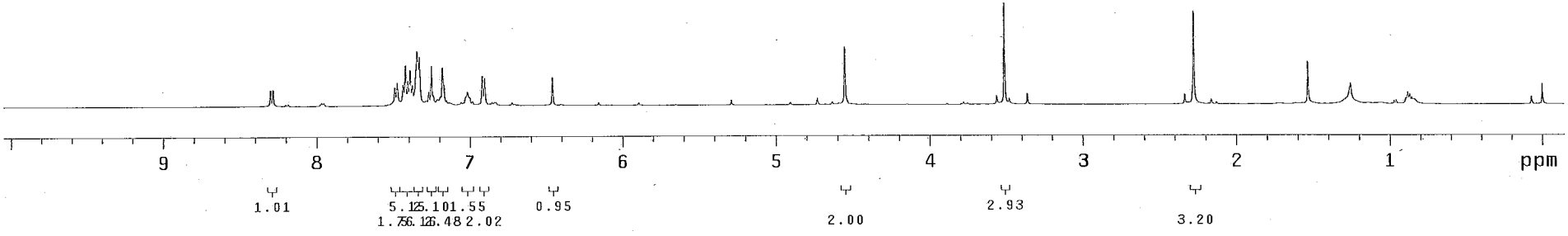
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-299-1_07May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7288222 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min, 38 sec





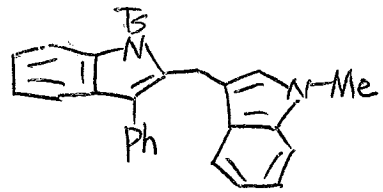
LiH-I-299-1_07May2014

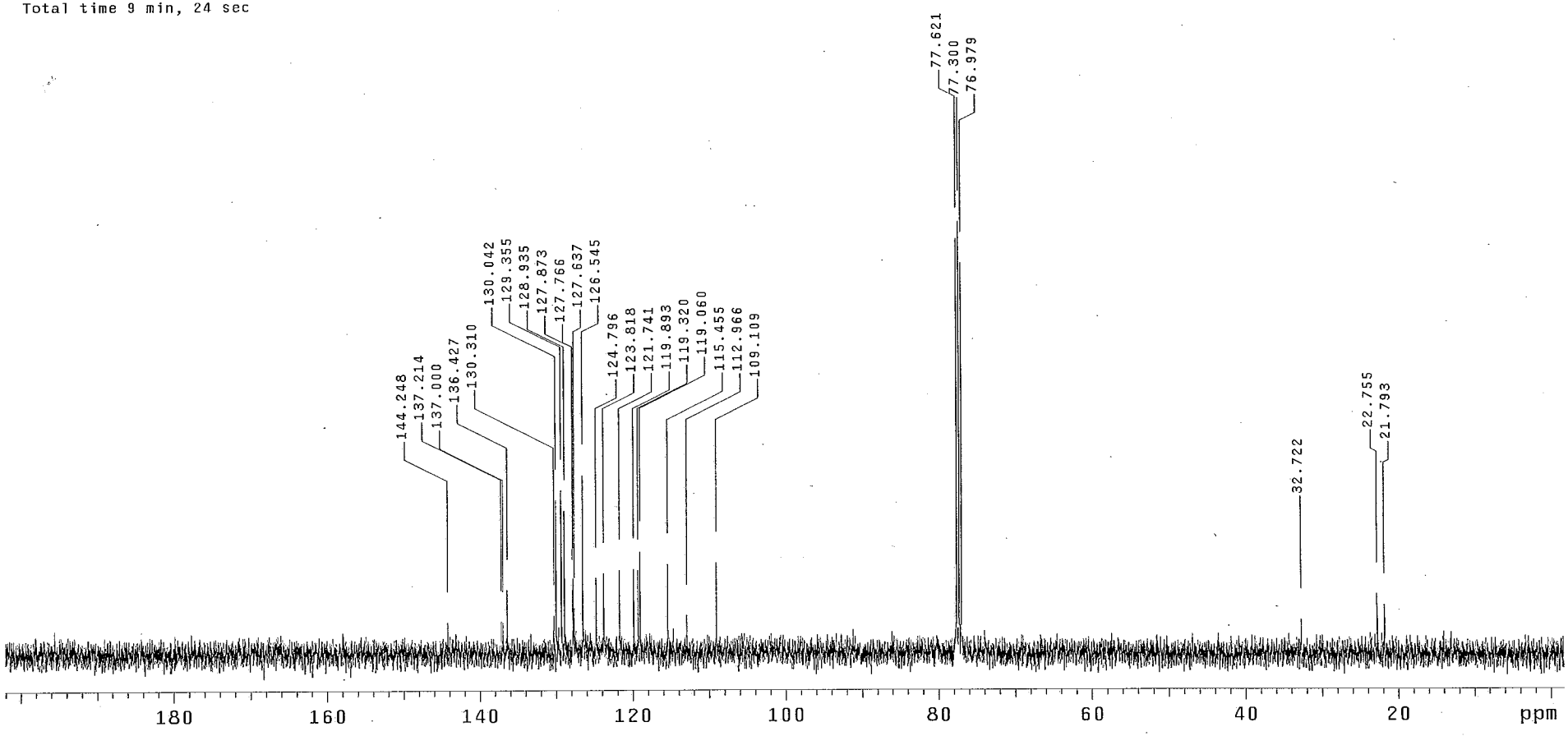
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-299-1_07May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
256 repetitions
OBSERVE C13, 100.5263721 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 9 min, 24 sec





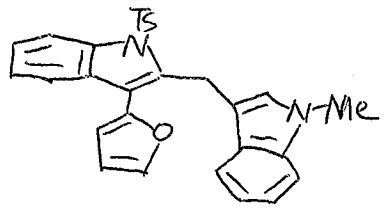
20140821.LiH-furanproduct

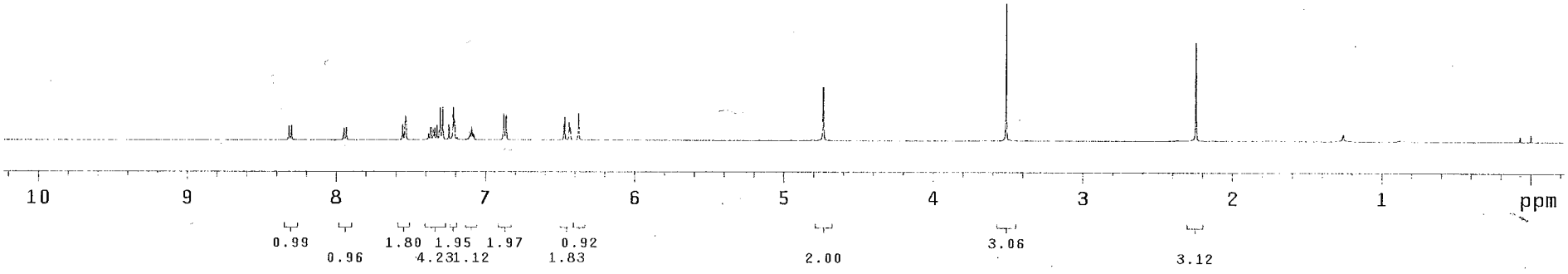
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: 20140821.LiH-furanproduct

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
18 repetitions
OBSERVE H1, 499.7288244 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 2 min, 32 sec





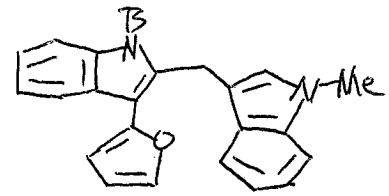
LiH-I-298_07May2014-17:26:09

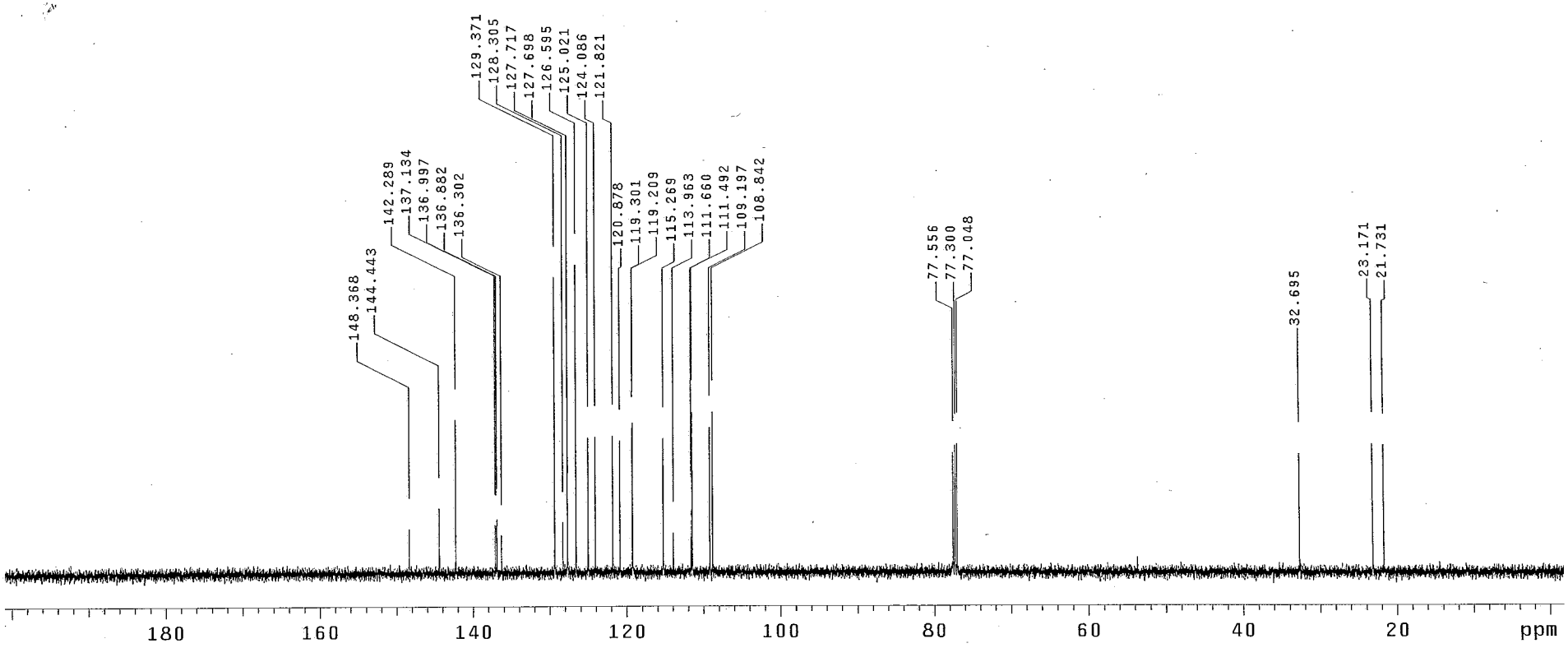
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-298_07May2014-17:26:09

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
244 repetitions
OBSERVE C13, 125.6568789 MHz
DECOUPLE H1, 499.7312897 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 55 sec





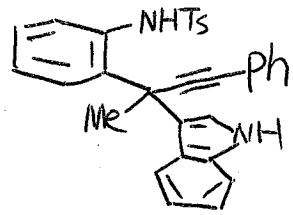
LiH-II-32-2_22May2014

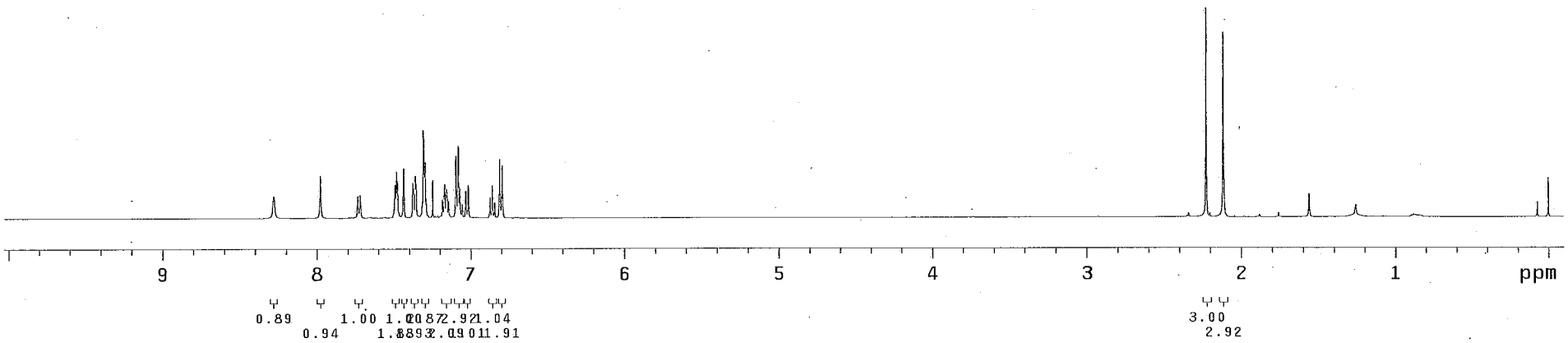
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-32-2_22May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
16 repetitions
OBSERVE H1, 499.7288234 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 1 min, 16 sec





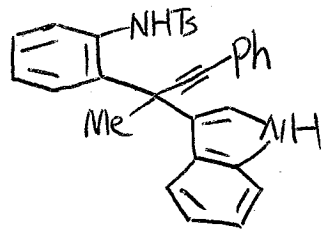
LiH-II-32-2_22May2014-11:52:15

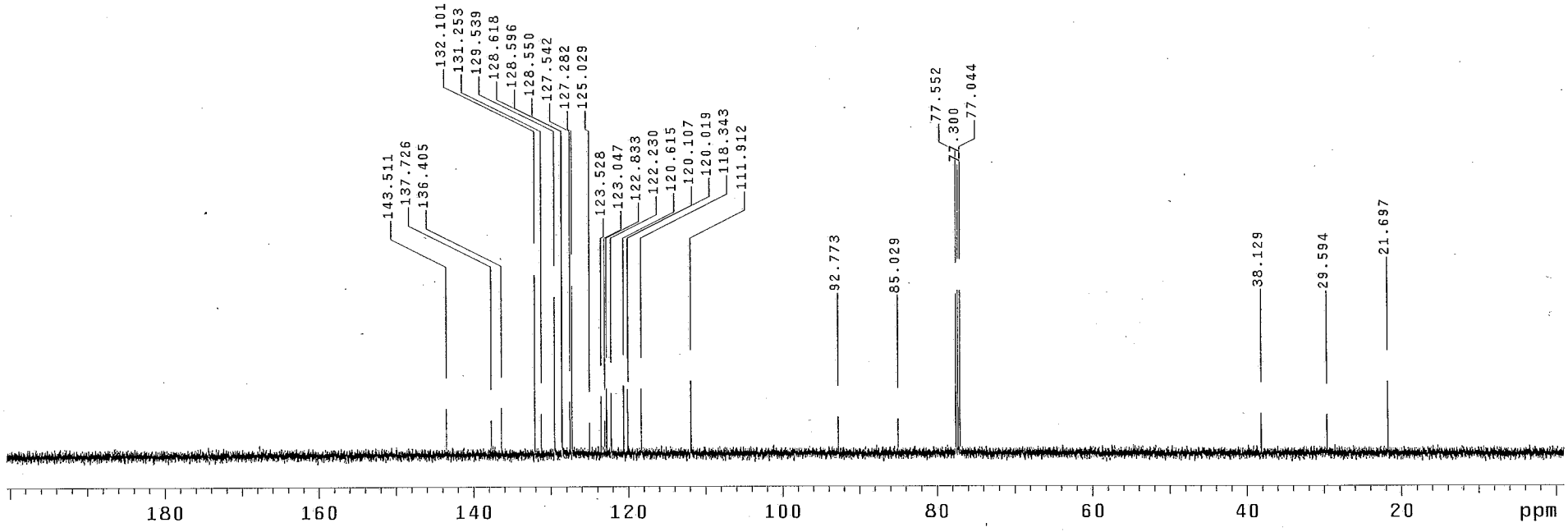
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-32-2_22May2014-11:52:15

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
256 repetitions
OBSERVE C13, 125.6568760 MHz
DECOUPLE H1, 499.7312897 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 55 sec





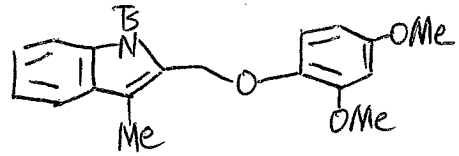
LiH-I-289_05May2014

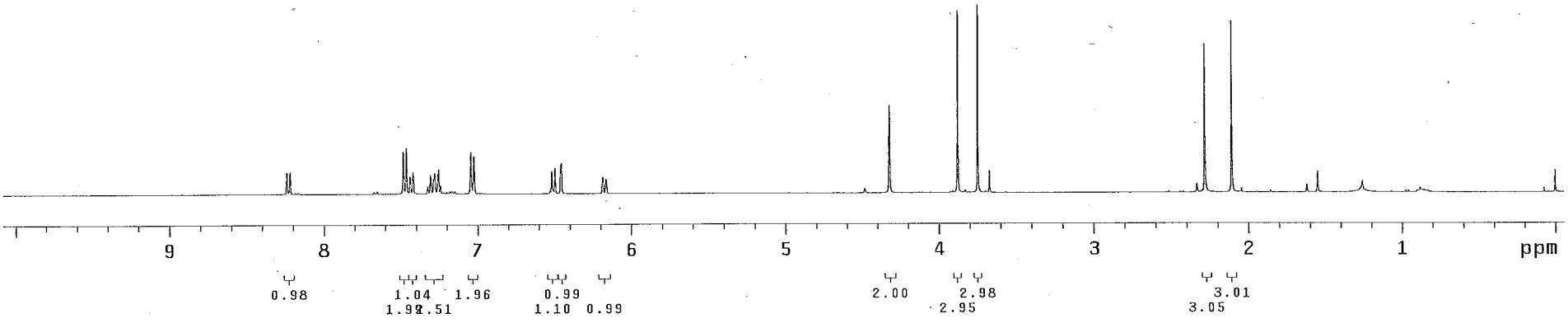
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-289_05May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
8 repetitions
OBSERVE H1, 399.7865266 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 57 sec





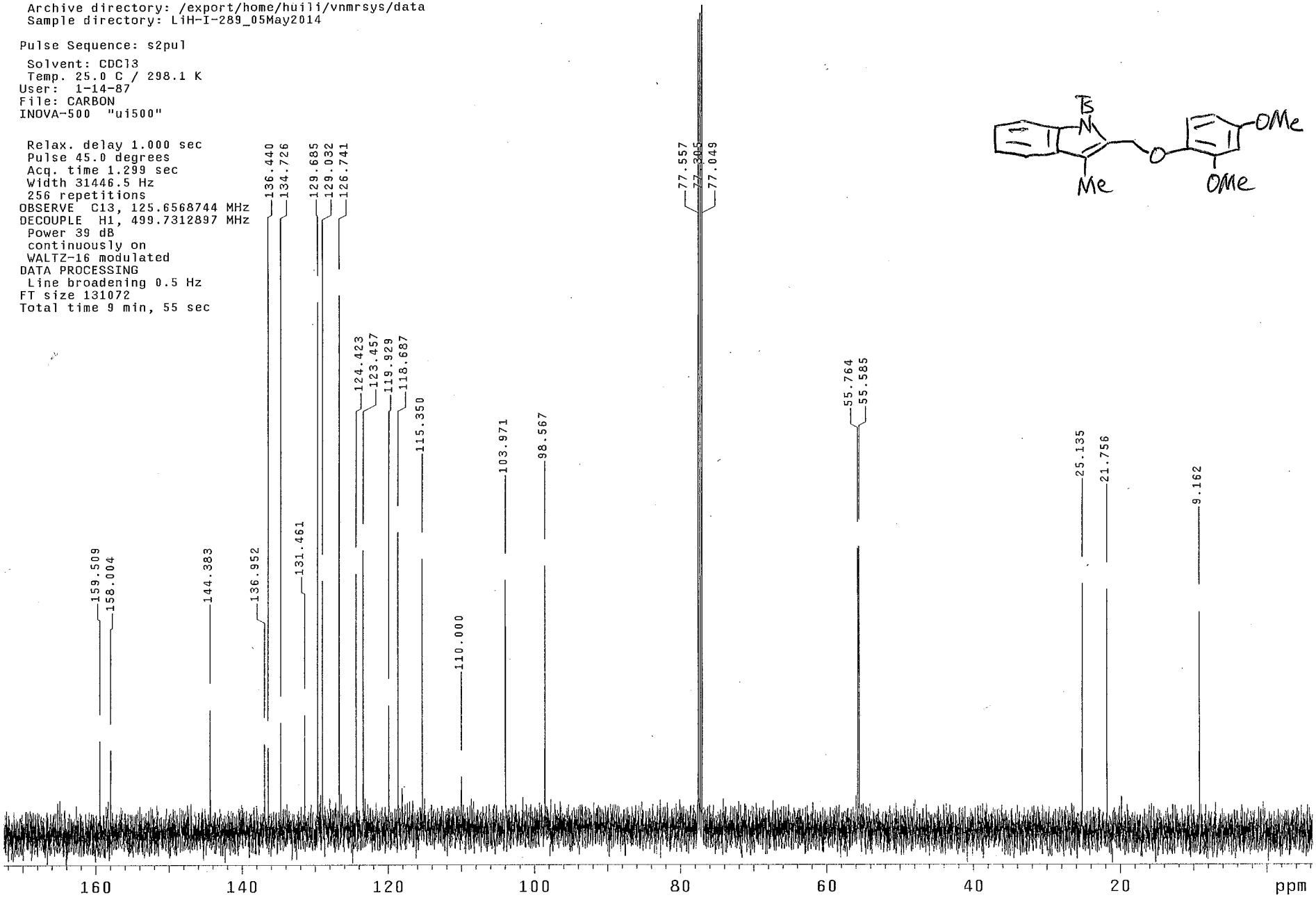
LiH-I-289_05May2014

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-289_05May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: CARBON
INOVA-500 "ui500"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
256 repetitions
OBSERVE C13, 125.6568744 MHz
DECOUPLE H1, 499.7312897 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 55 sec



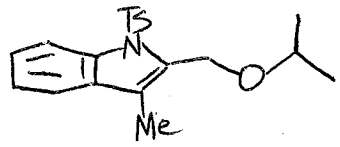
LiH-II-38_06Jun2014

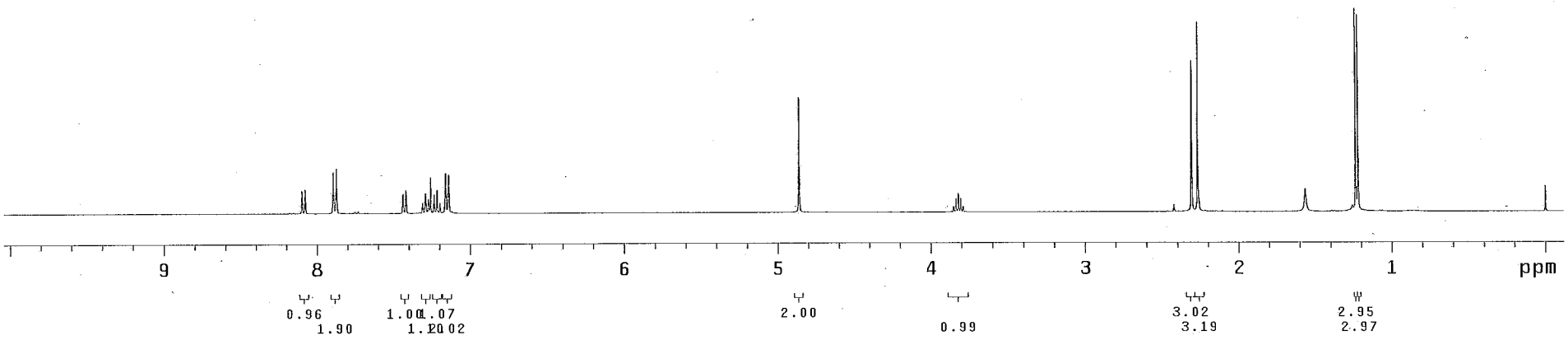
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-38_06Jun2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.754 sec
Width 6387.7 Hz
8 repetitions
OBSERVE H1, 399.7865245 MHz
DATA PROCESSING
FT size 65536
Total time 0 min, 57 sec





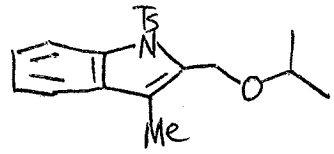
LiH-II-38_06Jun2014-15:21:52

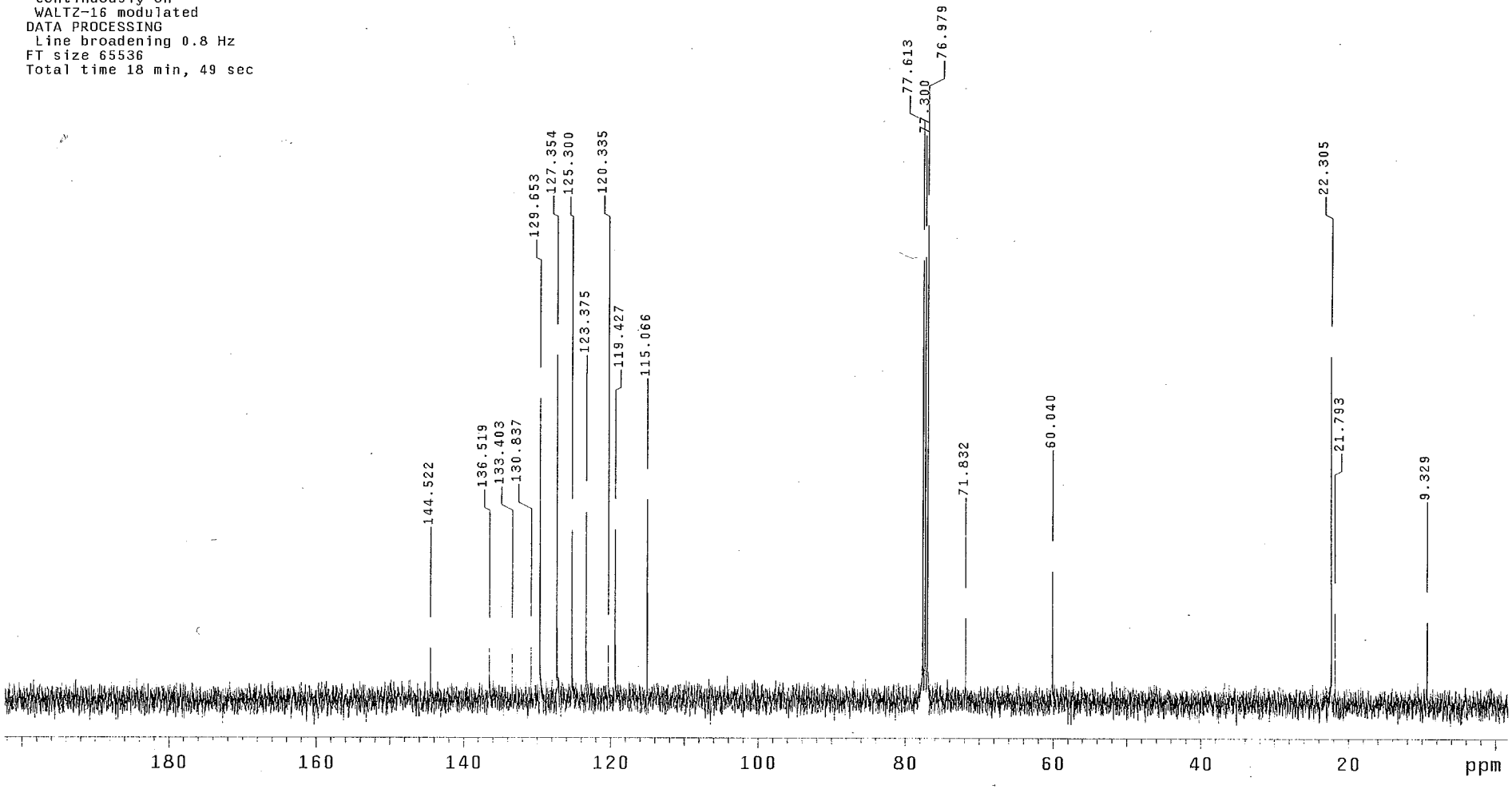
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-II-38_06Jun2014-15:21:52

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.197 sec
Width 25157.2 Hz
365 repetitions
OBSERVE C13, 100.5263721 MHz
DECOUPLE H1, 399.7885243 MHz
Power 43 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.8 Hz
FT size 65536
Total time 18 min, 49 sec





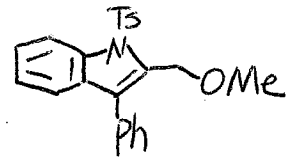
LiH-I-303_07May2014

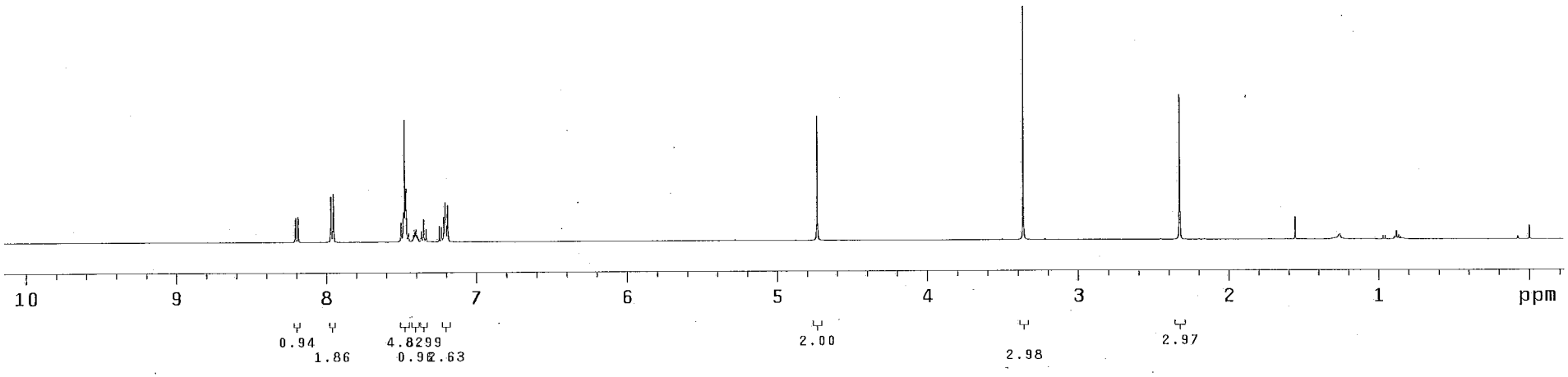
Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-303_07May2014

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
File: PROTON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 3.747 sec
Width 8000.0 Hz
8 repetitions
OBSERVE H1, 499.7288241 MHz
DATA PROCESSING
Line broadening 0.5 Hz
FT size 65536
Total time 0 min, 38 sec





LiH-I-303_07May2014-17:02:21

Archive directory: /export/home/huili/vnmrsys/data
Sample directory: LiH-I-303_07May2014-17:02:21

Pulse Sequence: s2pul

Solvent: CDCl3
Temp. 25.0 C / 298.1 K
User: 1-14-87
File: CARBON
INOVA-500 "nmr03"

Relax. delay 1.000 sec
Pulse 45.0 degrees
Acq. time 1.299 sec
Width 31446.5 Hz
187 repetitions
OBSERVE C13, 125.6568760 MHz
DECOUPLE H1, 499.7312897 MHz
Power 39 dB
continuously on
WALTZ-16 modulated
DATA PROCESSING
Line broadening 0.5 Hz
FT size 131072
Total time 9 min, 55 sec