Electronic Supplementary Material (ESI) for Chemical Communications

Supporting Information

Gold-Catalyzed Synthesis of Substituted 2- Aminofurans *via* a Formal [4+1]-Cycloadditions on 3-En-1-Ynamides

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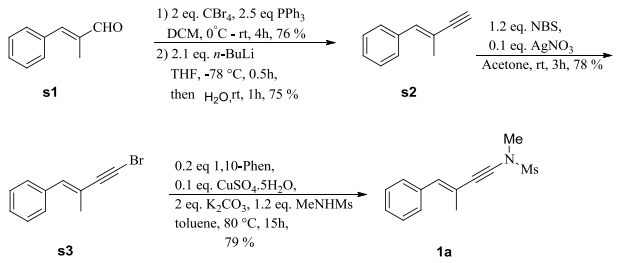
Contents:

(I) Experimental Procedure for the synthesis of ynamides.

(a) General Information.

Unless otherwise noted, all the reactions for the preparation of the substrates were performed in oven-dried glassware under nitrogen atmosphere with freshly distilled solvents. The catalytic reactions were performed under nitrogen atmosphere. Toluene, DCE and Methanol were distilled from CaH_2 under nitrogen. Methanol and triethylamine (Et_3N) were stored over 4Å molecular sieves prior to use. All other commercial reagents were used without further purification, unless otherwise noted. ^1H NMR and ^{13}C NMR spectra were recorded on varian 400 MHz, Bruker 400 and 600 MHz spectrometers using $CDCl_3$ and CD_2Cl_2 as the internal standards.

(b) Experimental Procedure for the synthesis of (*E*)-*N*-methyl-*N*-(3-methyl-4-phenylbut-3-en-1-yn-1-yl)methanesulfonamide (1a).



(1) Synthesis of (*E*)-(4,4-dibromo-2-methylbuta-1,3-dien-1-yl)benzene (s2).

To a dichloromethane (DCM) solution of carbon tetrabromide (18.14 g, 54.70 mmol) was added triphenylphosphine (21.48 g, 81.90 mmol) in DCM (100 mL) at 0 °C; the reaction mixture was stirred at 0 °C for 10 min, before a solution of (*E*)-2-methyl-3-phenylacrylaldehyde **(s1)** (4.00 g, 27.30 mmol) in anhydrous CH_2Cl_2 (10 mL) was added. The resulting mixture was stirred for 1 h at 0 °C before an addition of H_2O (100 mL) to partition the organic layer. The resulting mixture was extracted with DCM (3 x 20 mL); the combined organic layer was washed with brine, dried over Na_2SO_4, and concentrated under reduced pressure. To this residue was added 100 mL of diethyl ether, and the resulting suspension is filtered to remove triphenylphosphine oxide. The ethereal filtrate is concentrated in vacuo, and chromatographed through a silica gel column (hexane/ether, 10:1) to afford (*E*)-(4,4-dibromo-2-methylbuta-1,3-dien-1-yl)benzene (6.20 g, 76 %) as yellow oil.

To a THF solution of (*E*)-(4,4-dibromo-2-methylbuta-1,3-dien-1-yl)benzene (5.80 g,

20.14 mmol) was added *n*-BuLi (17.72 mL, 2.5 M in hexane, 44.31 mmol) dropwise at -78 °C for 30 min. The resulting solution was stirred for 30 min at -78 °C, before it was added with water (5 mL) at -78 °C. The resulting solution was stirred at -30 °C for 30 min, and warmed to RT before stirring for additional one hour. A saturated aqueous NH_4Cl (100 mL) was added, and the aqueous layer was separated and extracted with (3 x 20 mL) of ether. The organic layer is washed with brine (50 mL), dried over Na_2SO_4, and concentrated under reduced pressure. The residue was eluted through a silica column to afford compound **s2** (2.10 g, 75 %) as colourless liquid.

(2) Synthesis of (*E*)-(4-bromo-2-methylbut-1-en-3-yn-1-yl)benzene (s3)

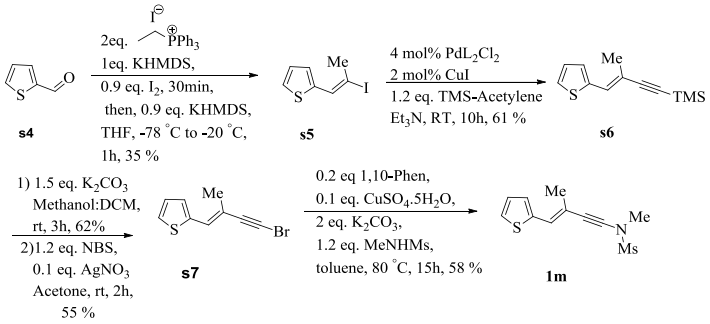
To a acetone (50 mL) solution of triisopropylsilyl acetylene (**s2)** (3.40 g, 23.90 mmol) was added NBS (5.10 g, 28.60 mmol) and $AgNO_3$ (0.4 g, 23.90 mmol); the resulting mixture was stirred for 3 h at room temperature. The solution was concentrated in vacuo before an addition of water. The organic layer was extracted with pentane (30 mL×3), dried over $MgSO_4$, and concentrated under reduced pressure to obtain pure colorless oil of (*E*)-(4-bromo-2-methylbut-1-en-3-yn-1-yl)benzene (**s3**) (4.11 g, 78 %) as colourless liquid.

(3) Synthesis of (*E*)-N-methyl-*N*-(3-methyl-4-phenylbut-3-en-1-ynyl)methanesulfon-amide (1a)

To a dried flask was added *N*-methylmethanesulfonamide[s1] (552 mg, 5.04 mmol), $CuSO_4 \cdot 5H_2O$ (104.8 mg, 0.420 mmol), 1,10-phenanthroline (162 mg, 0.904 mmol) and K_2CO_3 (1.16 g, 8.40 mmol), and this mixture was added with a toluene (5 mL) solution of (*E*)-(4-bromo-2-methylbut-1-en-3-yn-1-yl)benzene (**s3**) (1.00 g, 4.20 mmol). The flask was charged with nitrogen, and the resulting solution was heated at 80 ^oC overnight. The solution was cooled to room temperatures, filtered through Celite^{TM}, and concentrated *in vacuo*. The crude residue was chromatographed on a short silica column, giving ynamide **1a** as a white solid (880 mg, 79 %).[S1]

[S1]. J. S, Reddy, E. V. Bharathi, D. Dastagiri and A. Kamal, *Tetrahedron Lett.*, 2008,**49**, 348.

(c) Experimental Procedure for the synthesis of (*E*)-*N*-methyl-*N*-(3-methyl-4-(thiophen-2-yl)but-3-en-1-yn-1-yl)methanesulfonamide (1m)



(1) Synthesis of (*E*)-2-(2-iodoprop-1-enyl)thiophene (s5)

To a suspension of (ethyl)triphenylphosphonium iodide (11.2 g, 26.70 mmol) in THF (50 mL) was added *n*-BuLi (10.68 mL, 2.5 M in hexane, 26.70 mmol) at room temperature. After disappearance of solid material, the red solution was transferred into a vigorously stirred solution of iodine (2.25 g, 8.90 mmol) in THF (50 mL) at -78 °C. The resulting dark brown suspension was stirred for 5 min, and warmed gradually to -30 °C. To this mixture was added a solution of NaHMDS (26.70 mL, 1.0 M in THF) to afford a dark red solution. A THF (3 mL) solution of thiophene-2-carbaldehyde (1.0 g, 8.90 mmol) was slowly added, and the mixture was stirred for 30 min at -30 °C. The resulting mixture was diluted with pentane (40 mL), filtered through a Celite pad, and concentrated in vacuo. The crude residue was chromatographed through a silica column (hexane/ethyl acetate, 15:1) to afford (*Z*)-2-(2-iodoprop-1-enyl)thiophene **s5** (0.77 g, 35 %) as a colourless liquid[(S2)].

[S2]C.B. Lee, T.-C. Chou, X.-G. Zhang, Z.-G. Wang, S.D. Kuduk, M.D. Chappell, S.J Stachel, and S.J. Danishefsky, *J. Org. Chem.* 2000, **65**, 6525.

(2) Synthesis of (*E*)-trimethyl(3-methyl-4-(thiophen-2-yl)but-3-en-1-ynyl)silane(s6)

To a triethylamine solution (10 mL) of $Pd(PPh_3)_2Cl_2$ (56.21 mg, 0.07 mmol) and CuI (4.11 mg, 0.03 mmol) was added (*Z*)-2-(2-iodoprop-1-enyl)thiophene (**s1**) (500 mg, 1.99 mmol), the mixture was stirred for 10 min. To this mixture was added trimethylsilylacetylene (392 mg, 3.99 mmol) dropwise over 30 min. The resulting solution was stirred for 8 h at room temperature. The resulting solution was filtered through a short celite bed, and concentrated under reduced pressure. The residue was eluted through a silica column (hexane/ethyl acetate = 10:1) to afford compound (**s2**), (270 mg, 61 %) as a pale yellow oil.

(3) Synthesis of (*E*)-2-(4-bromo-2-methylbut-1-en-3-ynyl)thiophene (s7).

To a methanol/dichloromethane (20 mL, 2:1) mixing solvent was added (*E*)-trimethyl(3-methyl-4-(thiophen-2-yl)but-3-en-1-ynyl)silane (**s2**) (500 mg, 2.26 mmol) and K_2CO_3 (627 mg, 4.53 mmol); the resulting mixture was stirred for 30 min at room temperature, The solution was concentrated in vacuo before treatment with water. The organic layer was extracted with hexane (25 mL×3), and the organic layer was dried over $MgSO_4$. The crude product was purified by a vacuum distillation to afford compound (*E*)-2-(2-methylbut-1-en-3-ynyl)thiophene (210 mg, 62 %) as colorless oil.

The experimental procedure for the preparation of **s7** and **1m** is similar to **s3** and **1a** resp.

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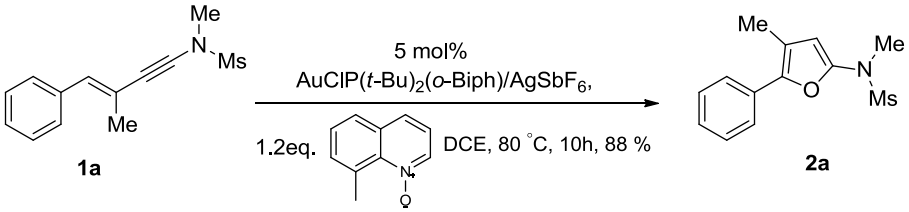
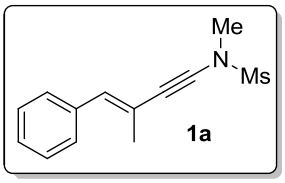
(IV) Standard procedure for gold(I) catalyzed [4+1]-cycloaddition reactions.



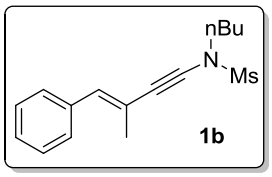
A two-necked flask was charged with P(*t*-Bu)$_2$(*o*-biphenyl)AuCl(I) (10.60 mg, 0.02 mmol) and silver hexafluoroantimonate (6.87 mg, 0.02 mmol), and to this mixture was added dry DCE (1.0 mL). The resulting solution was stirred at room temperature for 5 min. To this solution was added a dichloroethane solution (2 mL) of compound **1a** (100 mg, 0.40 mmol) and 8-methylquinoline 1-oxide (76.70 mg, 0.48 mmol). The mixture was kept stirring at 80 °C for 10 h before it was filtered over a short silica bed. The solvent was evaporated under reduced pressure, and the crude product was eluted through a silica gel column to afford compound **1a** as yellow oil (93 mg, 88 %).

Spectral data for (*E*)-*N*-methyl-*N*-(3-methyl-4-phenylbut-3-en-1-yn-1-yl)methanesulfonamide (1a)



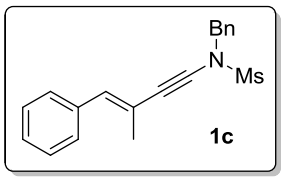
White solid; melting point: 73 °C; IR (KBr, cm^{-1}): 2229 (m), 1624 (m), 1621 (m), 1335 (s), 716 (w); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.31 (t, *J* = 7.6 Hz, 2 H), 7.24 ~ 7.21 (m, 3 H), 6.74 (s, 1 H), 3.24 (s, 3 H); 3.01 (s, 3 H) 2.03 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): 136.6, 134.9, 128.8, 128.2, 127.0, 118.8, 82.2, 73.1, 39.1, 36.5, 19.3. HRMS calcd. for $C_{13}H_{15}NO_2S$: 249.0823; found: 249.0820.

Spectral data for (*E*)-*N*-butyl-*N*-(3-methyl-4-phenylbut-3-en-1-yn-1-yl)methanesulfonamide (1b).



Brown oil; IR (neat, cm^{-1}): 2239 (m), 1621 (m), 1604 (m), 1321 (b), 701 (w); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.30 (t, *J* = 8.0 Hz, 2 H), 7.22 ~ 7.19 (m, 3 H), 6.73 (s, 1 H), 3.45 (t, *J* = 4.0 Hz, 2 H), 3.05 (s, 3 H); 2.04 (s, 3 H), 1.75 ~ 1.68 (m, 2 H), 1.43 ~ 1.38 (m, 2 H), 1.00 ~ 0.94 (m, 3 H) ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.6, 134.4, 128.7, 128.1, 127.0, 118.9, 80.8, 74.6, 51.3, 37.9. 30.2, 19.3, 19.2, 13.5; HRMS calcd. for $C_{16}H_{21}NO_2S$: 291.1293; found: 291.1297.

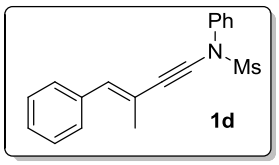
Spectral data for (*E*)-*N*-benzyl-*N*-(3-methyl-4-phenylbut-3-en-1-yn-1-yl)methanesulfonamide (1c).



Pale yellow oil; IR (neat, cm^{-1}): 2241 (m), 1619 (m), 1611 (s), 1318 (b), 703 (w); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.46 (dd, *J* = 6.4, 1.6 Hz, 2 H), 7.41 ~ 7.36 (m, 3 H), 7.32 (t, *J* = 7.2 Hz, 3 H), 7.22 (t, *J* = 4.4 Hz, 2 H), 6.69 (s, 1 H), 4.67 (s, 2 H), 2.91 (s, 3 H), 2.00 (s, 3 H); ^{13}C

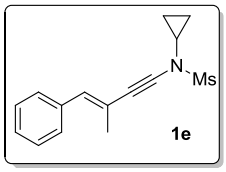
NMR (100 MHz, $CDCl_3$): δ 136.5, 134.4, 128.6, 128.5, 128.1, 127.6, 126.9, 118.8, 81.2, 75.3, 55.5, 38.7, 19.1; HRMS calcd. for $C_{19}H_{19}NO_2S$: 325.1136; found: 325.1139.

Spectral data for (*E*)-*N*-(3-methyl-4-phenylbut-3-en-1-yn-1-yl)-*N*-phenylmethanesulfonamide (1d).



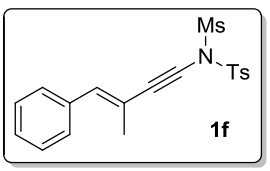
Brown oil; IR (neat, cm^{-1}): 2246 (m), 1618 (m), 1613 (s), 1367 (b), 711 (w); ^1H NMR (400 MHz, $CDCl_3$): δ 7.56 ~ 7.52 (m, 2 H), 7.44 ~ 7.40 (m, 2 H), 7.36 ~ 7.31 (m, 3 H), 7.27 ~ 7.22 (m, 3 H), 6.90 (s, 1 H), 3.11 (s, 3 H), 2.08 (s, 3 H) ^{13}C NMR (100 MHz, $CDCl_3$): δ 138.7, 136.5, 135.2, 129.4, 128.8, 128.2, 127.1, 125.4, 118.7, 81.1, 74.6, 36.2, 19.2; HRMS calcd. for $C_{18}H_{17}NO_2S$: 311.0980; found: 309.0976.

Spectral data for(*E*)-*N*-cyclopropyl-*N*-(3-methyl-4-phenylbut-3-en-1-yn-1-yl)methanesulfonamide (1e).



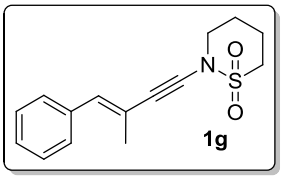
Pale yellow oil; IR (neat, cm^{-1}): 2256 (m), 1610 (m), 1607 (s), 1319 (b), 711 (w); ^1H NMR (400 MHz, $CDCl_3$): δ 7.32 ~ 7.29 (m, 3 H), 7.23 ~ 7.20 (m, 2 H), 6.72 (s, 1H), 3.09 (s, 3 H), 3.09 ~ 3.03 (m, 1 H), 2.06 (s, 3 H), 0.99 ~ 1.00 (m, 2 H), 0.90 ~ 0.86 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.6, 134.6, 128.7, 128.1, 127.7, 118.8, 79.8, 74.8, 37.5, 32.7; 19.3, 6.5; HRMS calcd. for $C_{15}H_{17}NO_2S$: 275.0980; found: 275.0979.

Spectral data for((*E*)-4-methyl-*N*-(3-methyl-4-phenylbut-3-en-1-yn-1-yl)-*N*-(methylsulfonyl)benzenesulfonamide (1f).



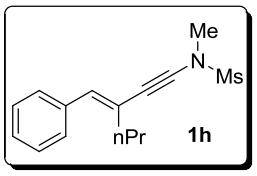
Pale yellow solid; melting point 81 °C; IR (KBr, cm^{-1}): 2231 (m), 1617 (m), 1611 (s), 1355 (b), 713 (s); ^{1}H.NMR (600 MHz, CD_2Cl_2): δ 7.81 (d, *J* = 8.4 Hz, 2 H), 7.42 (dd, *J* = 6.4 Hz, 0.8 Hz, 2 H), 7.37 ~ 7.19 (m, 5 H), 6.72 (s, 1 H), 3.10 (s, 3 H), 2.47 (s, 3 H), 2.05 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 144.7, 136.8, 134.4, 133.2, 129.8, 128.8, 128.2, 127.8, 127.0, 119.1, 83.2, 72.8, 39.3, 21.6, 19.3; HRMS calcd. for $C_{19}H_{19}NO_4S_2$: 389.0755; found: 389.0759.

Spectral data for (*E*)-2-(3-methyl-4-phenylbut-3-en-1-yn-1-yl)-1,2-thiazinane 1,1-dioxide (1g).



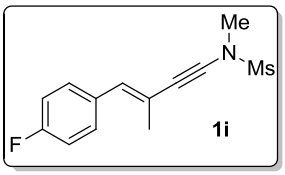
Pale yellow solid; melting point 128 °C; IR (KBr, cm^{-1}): 2251 (m), 1613 (m), 1619 (s), 1355 (b), 705 (w); ^{1}H NMR (400 MHz, CD_2Cl_2): δ 7.34 ~ 7.30 (m, 2 H), 7.25 ~ 7.20 (m, 3 H), 6.76 (s, 1 H), 3.80 (t, *J* = 5.6 Hz, 2 H), 3.26 ~ 3.22 (m, 2 H); 2.26 ~ 2.20 (m, 2 H), 2.04 (s, 3 H), 1.86 ~ 1.81(m, 2 H) ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.7, 134.7, 128.8, 128.2, 126.9, 119.0, 82.1, 72.7, 55.4, 47.9; 23.7, 21.1, 19.4; HRMS calcd. for $C_{15}H_{17}NO_2S$: 275.0980; found: 275.00983.

Spectral data for (*E*)-*N*-methyl-*N*-(3-methyl-4-phenyloct-3-en-1-yn-1-yl)methanesulfonamide (1h).



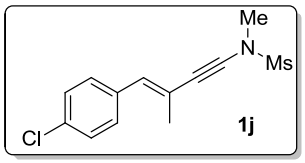
Brown oil; IR (neat, cm^{-1}): 2265 (m), 1621 (m), 1611 (s), 1321 (b), 715 (w); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.71 (d, *J* = 4.0 Hz, 2 H), 7.32 (q, *J* = 8.8 Hz, 2 H), 7.23 (t, *J* =8.8 1 H), 6.48 (s, 1 H), 3.27 (s, 3 H), 3.09 (s, 3 H), 2.29 (t, *J* = 7.6 Hz, 2 H); 1.65 ~ 1.57 (m, 2 H), 0.95 (t, *J* = 7.6 Hz, 3 H) ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.7, 131.9, 128.2, 128.1, 127.8, 121.2, 89.1, 70.5, 41.0, 39.0, 36.6, 21.7, 13.7; HRMS calcd. for $C_{15}H_{19}NO_2S$: 277.1136; found: 277.113.

Spectral data for (*E*)-*N*-(4-(4-fluorophenyl)-3-methylbut-3-en-1-yn-1-yl)-*N*-methylmethanesulfonamide (1i).



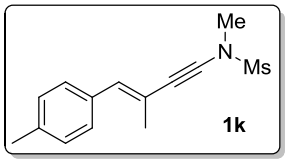
Brown oil; IR (neat, cm^{-1}): 2234 (m), 1599 (m), 1619 (s), 1349 (b), 1210 (m), 710 (w); ^1H NMR (400 MHz, $CDCl_3$): δ 7.68 ~ 7.64 (m, 2 H), 7.01 ~ 7.00 (m, 2 H), 6.40 (s, 1 H), 3.30 (s, 3 H), 3.08 (s, 3 H), 2.04 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.8 (*J* = 306.8 Hz), 132.9 (d, *J* = 3.87), 131.0, 129.4 (d, *J* = 9.3 Hz), 115.9, 114.9 (d, *J* = 26.5 Hz), 88.7, 71.0, 39.0, 36.8, 25.0; HRMS calcd. for $C_{13}H_{14}FNO_2S$: 267.0729; found: 267.0732.

Spectral data for(*E*)-*N*-(4-(4-chlorophenyl)-3-methylbut-3-en-1-yn-1-yl)-*N*-methylmethanesulfonamide (1j).



Pale white solid; melting point; 76 °C; IR (KBr, cm^{-1}): 2250 (m), 1612 (m), 1611 (w), 1352 (b), 703 (w), 315 (m); ^1H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 8.4 Hz, 2 H), 7.22 (d, *J* = 8.4 Hz, 2 H), 6.33 (s, 1 H), 3.21 (s, 3 H), 2.98 (s, 3 H), 2.02 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 135.1, 133.5, 132.8, 130.1, 128.5, 119.6, 82.7, 73.1, 39.2, 36.7, 19.3; HRMS calcd. for $C_{13}H_{14}ClNO_2S$: 283.0434; found: 283.0430.

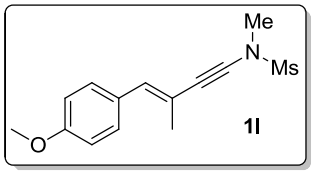
Spectral data for(*E*)-*N*-methyl-*N*-(3-methyl-4-(*p*-tolyl)but-3-en-1-yn-1-yl)methanesulfonamide (1k).



Brown oil; IR (neat, cm^{-1}): 2234 (m), 1612 (m), 1620 (s), 1351 (b), 713 (w); ^1H NMR (400 MHz, $CDCl_3$): δ 7.16 ~ 7.11 (m, 4 H), 6.72 (s, 1 H), 3.23 (s, 3 H), 3.10 (s, 3 H), 2.32 (s, 3

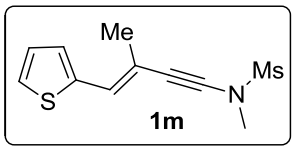
H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 136.9, 135.1, 133.8, 128.9, 128.8, 117.9, 81.9, 73.3, 39.2, 36.7, 21.2, 19.3; HRMS calcd. for $C_{14}H_{17}NO_2S$: 263.0980; found: 263.0985.

Spectral data for (*E*)-*N*-(4-(4-methoxyphenyl)-3-methylbut-3-en-1-yn-1-yl)-*N*-methylmethanesulfonamide (1l).



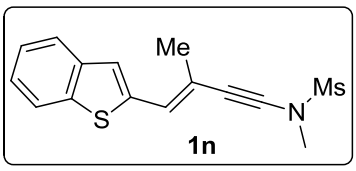
Pale yellow oil; IR (neat, cm^{-1}): 2235 (m), 1621 (m), 1603 (w), 1355 (b), 736 (w); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.65 (d, *J* = 8.8 Hz, 2 H), 6.84 (d, *J* = 8.8 Hz, 2 H), 6.85 (s, 1 H), 3.79 (s, 3 H), 3.25 (s, 3 H), 3.30 (s, 3 H), 2.02 (s, 3 H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 158.9, 132.2, 129.7, 129.2, 113.8, 113.7, 88.2, 71.3, 55.3, 39.1, 36.8, 25.1; HRMS calcd. for $C_{14}H_{17}NO_3S$: 279.0929; found: 279.0933.

Spectral data for (*E*)-*N*-methyl-*N*-(3-methyl-4-(thiophen-2-yl)but-3-en-1-yn-1-yl)methanesulfonamide (1m):



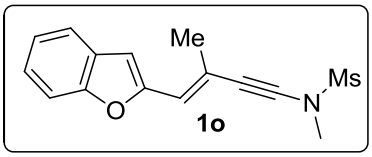
Pale yellow oil; IR (neat, cm^{-1}): 3120 (m), 2261 (m), 1621 (m), 1619 (s), 1510 (v), 1350 (b), 710 (w); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.19 (d, *J* = 5.2 Hz, 1 H), 7.10 (d, *J* = 3.2 Hz, 1 H), 6.97 (dd, *J* = 5.2 Hz, 3.2 Hz, 1 H), 6.69 (s, 1 H), 3.34 (s, 3 H), 3.12 (s, 3 H), 2.04 (s, 3 H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.8, 127.5, 127.0, 126.4, 124.7, 114.3, 91.4, 70.5, 38.8, 39.6, 24.4; HRMS calcd. for $C_{11}H_{13}NO_2S_2$: 255.0388; found: 255.0385.

Spectral data for (*E*)-*N*-(4-(benzo[*b*]thiophen-2-yl)-3-methylbut-3-en-1-yn-1-yl)-*N*-methylmethanesulfonamide (1n).



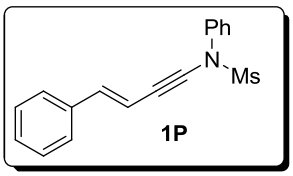
Brown oil; IR (neat, cm^{-1}): 3039 (m), 2209 (m), 1602 (m), 1611 (w), 1551 (v), 1355 (b), ^1H NMR (400 MHz, $CDCl_3$): δ 7.74 (t, *J* = 7.6 Hz, 1 H), 7.70 (t, *J* = 7.6 Hz, 1 H), 7.37 (s, 1 H), 7.32 ~ 7.25 (m, 2 H), 6.73 (s, 1 H), 3.39 (s, 3 H), 3.15 (s, 3 H), 2.10 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.6, 139.2, 138.9, 126.8, 124.4, 124.3, 123.3, 121.9, 117.3, 91.8, 70.6, 38.7, 37.2, 24.7; HRMS calcd. For $C_{15}H_{15}NO_2S_2$: 305.0544; found: 305.0547.

Spectral data for (*E*)-*N*-(4-(benzofuran-2-yl)-3-methylbut-3-en-1-yn-1-yl)-*N*-methylmethanesulfonamide (1o).



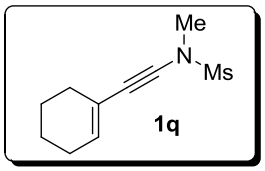
Brown oil; IR (neat, cm^{-1}): 3048 (m), 2223 (m),1612 (m), 1616 (m) (s), 1619 (s), 1316 (b); ^1H NMR (400 MHz, $CDCl_3$ 7.54 (d, *J* = 7.6, Hz, 1 H), 7.43 (d, *J* = 7.6 Hz, 1 H), 7.22 ~ 7.15 (m, 3 H), 6.45 (s, 1 H), 3.40 (s, 3 H), 3.14 (s, 3 H), 2.10 (s, 3 H), ^{13}C NMR (100 MHz, $CDCl_3$): δ 154.1, 129.0, 124.2, 122.9, 122.7, 120.9, 120.4, 118.3, 110.9, 104.4, 91.4, 71.5, 39.0, 36.9, 24.5; HRMS calcd. for $C_{15}H_{15}NO_3S$: 289.0773; found: 289.0777.

Spectral data for (*E*)-*N*-phenyl-*N*-(4-phenylbut-3-en-1-yn-1-yl)methanesulfonamide



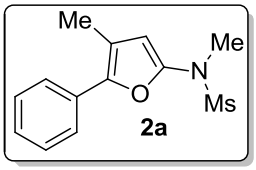
Brown oil; IR (neat, cm^{-1}): 2253 (m), 1612 (m), 1615 (s), 1351 (b), 1115 (s), 706 (w); ^1H NMR (600 MHz, $CDCl_3$ 7.54 (d, *J* = 8.4 Hz, 2 H), 7.43 (t, *J* = 7.8 Hz, 2 H), 7.37 ~ 7.35 (m, 3 H), 7.31 (t, *J* = 7.8 Hz, 2 H), 7.26 (d, *J* = 8.4 Hz, 1 H), 6.92 (d, *J* =16.0 Hz, 1 H), 6.28 (d, *J* =16.0 Hz 1 H), 3.13 (s, 3 H) ^{13}C NMR (150 MHz, $CDCl_3$): δ 140.5, 138.6, 136.2, 129.5, 128.7, 128.5, 128.4, 126.1, 125.6, 107.0, 83.8, 70.5, 36.9; HRMS calcd. for $C_{17}H_{15}NO_2S$: 297.0823; found: 297.0827

Spectral data of *N*-(cyclohex-1-en-1-ylethynyl)-*N*-methylmethanesulfonamide(1q)



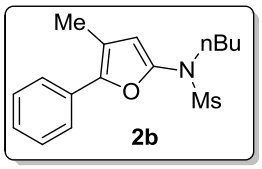
Brown oil; IR (neat, cm^{-1}): 2139 (s), 1612 (m), 1351 (b), 706 (w); ^1H NMR (400 MHz, $CDCl_3$ 6.03 (d, *J* = 2.0 Hz, 1 H), 3.17 (s, 3 H), 3.00 (s, 3 H), 2.07 ~ 2.05 (m, 4 H), 1.61 ~ 1.52 (m, 4 H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 134.8, 119.6, 80.6, 70.9, 39.2, 36.3, 29.4, 25.6, 22.3, 21.4; HRMS calcd. for $C_{10}H_{15}NO_2S$: 213.0823; found: 213.0826.

Spectral data for *N*-methyl-*N*-(4-methyl-5-phenylfuran-2-yl)methanesulfonamide (2a).



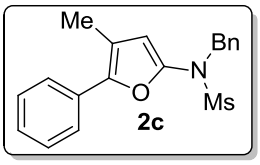
Colourless liquid; IR (neat, cm^{-1}): 3090 (m), 1601 (m), 1610 (m), 1355 (b), 705 (w), 550 (s); ^1H NMR (400 MHz, $CDCl_3$): 7.51 (d, *J* = 7.2 Hz, 2 H), 7.36 (t, *J* = 6.8 Hz, 2 H), 7.24 (t, *J* = 7.2 Hz, 1 H), 6.15 (s, 1 H), 3.29 (s, 3 H), 3.06 (s, 3 H), 2.21 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.2, 144.8, 131.0, 128.5, 127.1, 125.3, 117.7, 109.7, 37.3, 37.2, 11.1; HRMS calcd. for $C_{13}H_{15}NO_3S$: 265.0773; found: 265.0770.

Spectral data for *N*-butyl-*N*-(4-methyl-5-phenylfuran-2-yl)methanesulfonamide (2b).



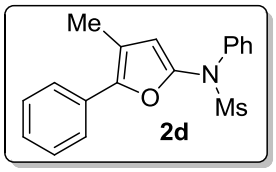
Brown oil; IR (neat, cm^{-1}): 3031 (m), 1611 (m), 1595 (m), 1315 (b), 706 (w), 559 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.54 ~ 7.52 (m, 2 H), 7.40 ~ 7.37 (m, 2 H), 7.26 (t, *J* = 5.2 Hz, 1 H), 6.20 (s, 1 H), 3.61 (t, *J* = 4.2 Hz, 2 H), 3.01 (s, 3 H), 2.24 (s, 3 H), 1.57~1.53 (m, 2 H), 1.39 ~ 1.35 (m, 2 H), 0.90 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.8, 143.0, 131.1, 128.6, 127.1, 125.4, 117.8, 112.4, 50.0, 38.8, 30.7, 19.5, 13.6, 12.0; HRMS calcd. for $C_{16}H_{21}NO_3S$: 307.1242; found: 307.1243.

Spectral data for *N*-benzyl-*N*-(4-methyl-5-phenylfuran-2-yl)methanesulfonamide (2c).



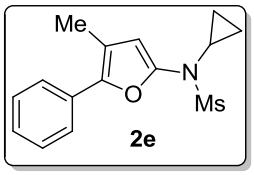
Brown oil; IR (neat, cm^{-1}): 3044 (m), 1621 (m), 1599 (m), 1339 (b), 700 (w), 590 (s); ^1H NMR (400 MHz, $CDCl_3$: δ 7.50 (d, *J* = 8.0 Hz, 2 H), 7.41 ~ 7.23 (m, 8 H), 6.02 (s, 1 H), 4.80 (s, 2 H), 3.00 (s, 3 H), 2.16 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.6, 143.0, 135.6, 131.0, 128.8, 128.7, 128.0, 127.1, 125.3, 117.8, 112.2, 54.3, 39.7, 11.9; HRMS calcd. for $C_{19}H_{19}NO_3S$: 341.1086; found: 341.1084.

Spectral data for *N*-(4-methyl-5-phenylfuran-2-yl)-*N*-phenylmethanesulfonamide (2d).



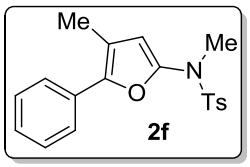
Brown oil; IR (neat, cm^{-1}): 3134 (m), 1609 (m), 1592 (m), 1341 (b), 709 (w), 590 (s). ^1H NMR (400 MHz, $CDCl_3$): δ 7.58 (d, *J* = 7.2 Hz, 2 H), 7.43 ~ 7.37 (m, 4 H), 7.31 (q, *J* = 9.0 Hz, 2 H), 7.30 ~ 7.29 (m, 2 H), 6.29 (s, 1 H), 3.17 (s, 3 H), 2.23 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.3, 143.8, 139.9, 131.0, 129.5, 128.6, 128.0, 127.3, 127.1, 125.6, 118.0, 112.3, 39.4, 12.0; HRMS calcd. for $C_{18}H_{17}NO_3S$: 327.0929; found: 327.0914.

Spectral data for *N*-cyclopropyl-*N*-(4-methyl-5-phenylfuran-2-yl)methanesulfonamide (2e).



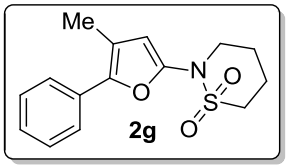
Colourless liquid; IR (neat, cm^{-1}): 3034 (m), 1609 (m), 1595 (m), 1350 (b), 700 (w), 589 (s). ^1H NMR (400 MHz, $CDCl_3$): δ 7.54 (d, *J* = 7.6 Hz, 2 H), 7.38 (t, *J* = 7.6 Hz, 2 H), 7.27 (d, *J* = 7.2 Hz, 1 H), 6.12(s, 1H), 3.10 (s, 3 H), 3.00 ~ 2.99 (m, 1 H), 2.23 (s, 3 H), 0.87 ~ 0.82 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.8, 144.1, 131.1, 128.5, 127.1, 125.4, 117.6, 111.7, 38.4, 31.7, 11.9, 7.7; HRMS calcd. for $C_{15}H_{17}NO_3S$: 291.0929; found: 291.0932.

Spectral data for *N*,4-dimethyl-*N*-(4-methyl-5-phenylfuran-2-yl)benzenesulfonamide (2f).



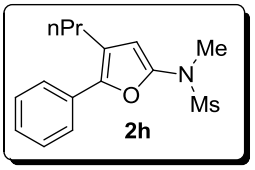
Brown oil; IR (neat, cm^{-1}): 3104 (m), 1604 (m), 1355 (b), 714 (w), 590 (s); ^{1}H NMR (600 MHz, $CDCl_3$: δ 7.65 ~ 7.63 (m, 2 H), 7.39 (dd, *J* = 8.4, 1.2 Hz, 2 H), 7.35 ~ 7.32 (m, 2 H), 7.28 (d, *J* = 8.4 Hz, 2 H), 7.22 (dd, *J* = 7.2, 1.2 Hz, 1 H), 6.13 (s, 1 H), 3.17 (s, 3 H), 2.40 (s, 3 H), 2.23 (s, 3 H), ^{13}C NMR (150 MHz, $CDCl_3$): δ 145.9, 145.1, 144.0, 134.6, 131.2, 129.6, 128.4, 127.9, 126.8, 125.2, 117.6, 109.7, 37.1, 21.6, 12.0; HRMS calcd. for $C_{19}H_{19}NO_3S$: 341.1086; found: 341.1088.

Spectral data for 2-(4-methyl-5-phenylfuran-2-yl)-1,2-thiazinane 1,1-dioxide (2g).



Yellow oil; IR (neat, cm^{-1}): 3036 (m), 1620 (m), 1609 (m),1353 (b), 702 (w), 591 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 (dd, *J* = 8.4 Hz, 2 H), 7.40 ~ 7.37 (m, 2 H), 7.27 ~ 7.23 (m, 1 H), 6.14 (s, 1 H), 3.83 (t, *J* = 5.6 Hz, 2 H), 3.23 (t, *J* = 5.6 Hz 2 H), 2.36 ~ 2.15 (m, 2 H), 2.14 (s, 3 H), 1.94 ~ 1.89 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.6, 145.2, 131.4, 128.9, 126.7, 125.6, 117.9, 110.9, 52.9, 49.6, 24.4, 23.9, 12.2; HRMS calcd. for $C_{15}H_{17}NO_3S$: 291.0929; found: 291.0933.

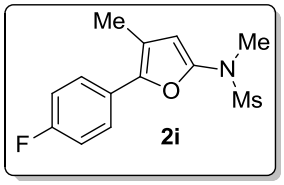
Spectral data for *N*-(4-butyl-5-phenylfuran-2-yl)-*N*-methylmethanesulfonamide (2h).



Pale yellow oil; IR (neat, cm^{-1}): 3033 (m), 1607 (m), 1599 (m), 1354 (b), 1231 (s), 706 (w), 590 (s); ^{1}H NMR (600 MHz, $CDCl_3$: δ 7.52 ~ 7.50 (m, 2 H), 7.40 ~ 7.37 (m, 2 H), 7.27 (dd, *J* = 7.2, 6.0 Hz, 1 H), 6.21 (s, 1 H), 3.31 (s, 3 H), 2.99 (s, 3 H), 2.57 (t, *J* = 7.8 Hz, 2 H), 1.66 ~

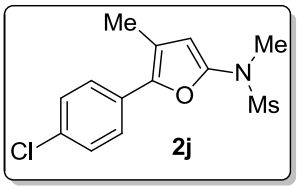
1.62 (m, 2 H), 0.96 (t, *J* = 7.2 Hz, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 146.0, 145.0, 131.1, 128.6, 127.2, 125.7, 123.0, 108.1, 37.4, 37.3, 28.0, 22.9, 14.0; HRMS calcd. for $C_{15}H_{19}NO_3S$: 293.1086, found : 293.1083.

Spectral data for *N*-(5-(4-fluorophenyl)-4-methylfuran-2-yl)-*N*-methylmethanesulfonamide (2i).



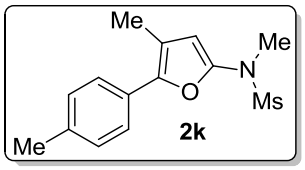
Pale yellow oil; IR (neat, cm^{-1}): 3033 (m), 1607 (m), 1590 (m), 1209 (m), 1352 (b), 592 (s); ^{1}H NMR (400 MHz, $CDCl_3$: δ 7.51 ~ 7.48 (m, 2 H), 7.11 ~ 7.06 (m, 2 H), 6.16 (s, 1 H), 3.34 (s, 3 H), 3.00 (s, 3 H), 2.20 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.7(d, *J* = 306.8 Hz), 145.5, 144.8, 132.3, 127.2 (dd, *J* = 12.3, 3.7 Hz), 117.4, 115.8, 115.7 (d, *J* = 33.2), 109.7, 37.4, 37.3, 11.8; HRMS calcd. for $C_{13}H_{14}FNO_3S$: 283.0678 ; found: 283.0678

Spectral data *N*-(5-(4-chlorophenyl)-4-methylfuran-2-yl)-*N*-methylmethanesulfonamide (2j).



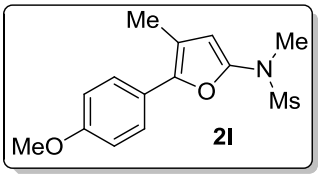
Colourless liquid; IR (neat, cm^{-1}): 3041 (m), 1614 (m), 1596 (m), 1344 (b), 703 (w), 5799 (s), 299 (m); ^{1}H NMR (600 MHz, $CDCl_3$: δ 7.45 (d, *J* = 8.8 Hz, 2 H), 7.35 (d, *J* = 8.8 Hz, 2 H), 6.16 (s, 1 H), 3.31 (s, 3 H), 3.00 (s, 3 H), 2.11 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 145.1, 144.8, 132.8, 129.5, 129.3, 126.5, 118.3, 109.8 40.5, 37.4, 12.0; HRMS calcd. for $C_{13}H_{14}ClNO_3S$: 299.0383; found: 299.0341.

Spectral data for*N*-methyl-*N*-(4-methyl-5-(p-tolyl)furan-2-yl)methanesulfonamide (2k).



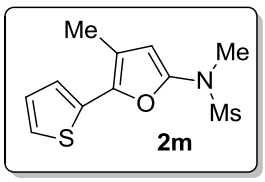
Colourless oil; IR (neat, cm^{-1}): 3122 (m), 1603 (m), 1597(m), 1344 (b), 703 (w), 591 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.42 (d, *J* = 8.4 Hz, 2 H), 7.21 (d, *J* = 8.4 Hz, 2 H), 6.16 (s, 1 H), 3.30 (s, 3 H), 2.99 (s, 3 H), 2.35 (s, 3 H), 2.20 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 146.6, 144.4, 137.0, 128.9, 128.3, 125.4, 117.1, 109.9, 37.4, 37.3, 21.2, 11.9; HRMS calcd. for $C_{14}H_{17}NO_3S$: 279.0929; found: 279.0927.

Spectral data for *N*-(5-(4-methoxyphenyl)-4-methylfuran-2-yl)-*N*-methylmethanesulfonamide (2l).



Colourless oil; IR (neat, cm^{-1}): 3042 (m), 1605 (m), 1596 (w), 1340 (b), 701 (w), 590 (s); ^1H NMR (600 MHz, $CDCl_3$: δ 7.45 (d, *J* = 9.0 Hz, 2 H), 6.92 (d, *J* = 9.0 Hz, 2 H), 6.14 (s, 1 H), 3.82 (s, 3 H), 3.30 (s, 3 H), 2.99 (s, 3 H), 2.19 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 158.8, 146.5, 144.2, 126.9, 124.0, 116.2, 114.0, 109.9, 55.3, 37.3, 30.9, 11.8; HRMS calcd. for $C_{14}H_{17}NO_4S$: 295.0878; found: 295.0872.

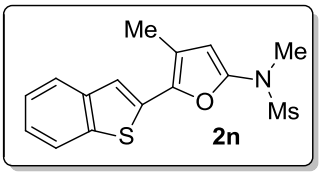
Spectral data for *N*-methyl-*N*-(4-methyl-5-(thiophen-2-yl)furan-2-yl)methanesulfonamide (2m).



Brown oil; IR (neat, cm^{-1}): 3043 (m), 1601 (m), 1559 (v), 1344 (b), 704 (w), 595(s); ^1H NMR (600 MHz, CD_2Cl_2): δ 7.29 (d, *J* = 5.4 Hz, 1 H), 7.20 (d, *J* = 3.6 Hz, 1 H), 7.08 (t, *J* = 5.4 Hz, 1 H), 6.17(s, 1 H), 3.28 (s, 3 H), 3.00 (s, 3 H), 2.20 (s, 3 H); ^{13}C NMR (150 MHz,

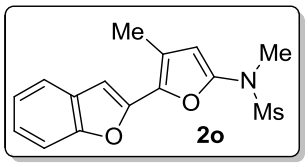
CD_2Cl_2): δ 145.2, 143.0, 133.4, 127.8, 124.7, 123.5, 117.7, 109.7, 37.9, 37.6, 11.6; HRMS calcd. for $C_{11}H_{13}NO_3S_2$: 271.0337; found: 271.0341.

Spectral data for *N*-(5-(benzo[*b*]thiophen-2-yl)-4-methylfuran-2-yl)-*N*-methylmethanesulfonamide (2n).



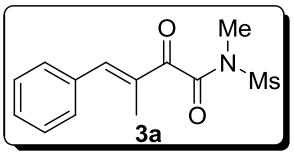
Yellow oil IR (neat, cm^{-1}): 3078 (m), 1601 (m), 1502 (v), 1335 (b), 703 (w), 555 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.88 (d, *J* = 7.8 Hz, 1 H), 7.74 (d, *J* = 7.8 Hz, 1 H), 7.36 (s, 1 H), 7.33 (dd, *J* = 7.8 Hz, 1.2 Hz, 1 H), 7.31 ~ 7.38 (m, 2 H), 3.33 (s, 3 H), 3.04 (s, 3 H), 2.29 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 145.3, 142.4, 139.8, 138.8, 132.6, 124.7, 124.4, 123.5, 122.0, 119.5, 119.0, 109.6, 37.4, 37.3, 11.6; HRMS calcd. for $C_{15}H_{15}NO_3S_2$: 321.0493; found: 321.0497.

Spectral data for *N*-(5-(benzofuran-2-yl)-4-methylfuran-2-yl)-*N*-methylmethanesulfonamide (2o).



Yellow oil; IR (neat, cm^{-1}): 3078 (m), 1602 (m), 1529 (v), 1335 (b), 707 (w), 597 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.54 (d, *J* = 3.6 Hz, 1 H), 7.48 (d, *J* = 3.6 Hz, 1 H), 7.26 ~ 7.22(m, 2 H), 6.77 (s, 1 H), 6.21 (s, 1 H), 3.34 (s, 3 H), 3.03 (s, 3 H), 2.33 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 154.4, 147.9, 146.0, 139.3, 128.4, 124.3,123.2, 120.9, 120.8, 111.1, 109.2, 101.8, 37.5, 37.2, 11.1; HRMS calcd. for $C_{15}H_{15}NO_4S$: 305.0722; found: 305.0722.

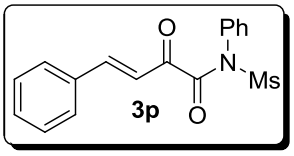
Spectral data for (*E*)-*N*,3-dimethyl-*N*-(methylsulfonyl)-2-oxo-4-phenylbut-3-enamide(3a)



Yellow oil; IR (neat, cm^{-1}): 1701 (s), 1669 (s), 1621 (m), 1610 (m) 1335 (b), 707 (w);

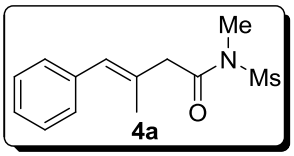
^{1}HNMR (600 MHz, $CDCl_3$): δ 7.47 (t, *J* = 4.2 Hz, 2 H), 7.41 ~ 7.39 (m, 2 H), 7.37 (t, *J* = 1.2 Hz, 1 H), 7.36 (s, 1 H), 3.34 (s, 3 H), 3.26 (s, 3 H), 2.15 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 191.6, 167.4, 146.5, 134.8, 132.8, 130.3, 129.6, 128.7, 40.5, 31.3, 12.4; HRMS calcd. for $C_{13}H_{15}NO_4S$: 281.0722; found: 281.0715.

Spectral data for (*E*)-*N*-(methylsulfonyl)-2-oxo-*N*,4-diphenylbut-3-enamide (3p).



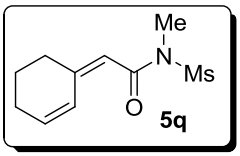
Pale yellow oil; IR (neat, cm^{-1}): 1700 (s), 1669 (s), 1621 (m), 1610 (w),; ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.69 (d, *J* = 16.2 Hz, 1 H), 7.57 (t, *J* = 4.2 Hz, 2 H), 7.49 ~ 7.48 (m, 3 H), 7.44 ~ 7.40 (m, 3 H), 7.36 ~ 7.34 (m, 2 H), 6.87 (d, *J* =16.2 Hz 1 H), 3.42 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 187.1, 167.2, 149.3, 133.8, 133.7, 131.7, 130.4, 129.9, 129.8, 129.1, 129.0, 121.7, 41.2.

Spectral data for (*E*)-*N*,3-dimethyl-*N*-(methylsulfonyl)-2-oxo-4-phenylbut-3-enamide(4a)



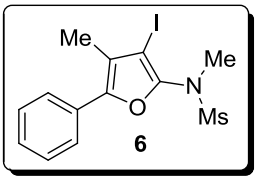
Brown oil; IR (neat, cm^{-1}): 1667 (s), 1621 (m), 1613 (s), 1335 (b), 709 (w); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.33 ~ 7.30 (m, 2 H), 7.23 ~ 7.21 (m, 3 H), 6.34 (s, 1 H), 3.48 (s, 2 H), 3.32 (s, 3 H), 3.27 (s, 3 H),1.93 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 171.8, 137.1, 130.9, 129.6, 128.8, 128.2, 126.8, 47.5, 41.5, 32.7, 18.2; HRMS calcd. for $C_{13}H_{17}NO_3S$: 267.0929; found: 267.0924

Spectral data for (*Z*)-2-(cyclohex-2-en-1-ylidene)-*N*-methyl-*N*(methylsulfonyl)acetamide



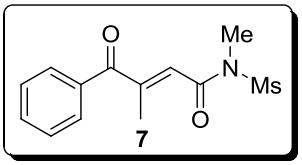
Yellow oil; IR (neat, cm^{-1}): 1637 (m), 1621 (m), 1671 (s), 709 (w); ^1H NMR (400 MHz, $CDCl_3$): δ 6.29 ~ 6.25 (m, 1 H), 6.14 (d, *J* = 8.8 Hz 1 H), 6.05 (s, 1 H), 3.26 (s, 3 H), 3.22 (s, 3 H), 2.88 ~ 2.85 (m, 2 H), 2.21 ~ 2.17 (m, 2 H), 1.74 ~ 1.67 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.0, 155.7, 139.4, 130.1, 114.4, 41.6, 32.9, 27.1, 25.6, 21.8; HRMS calcd. for $C_{10}H_{15}NO_3S$: 229.0773; found: 229.0773.

Spectral data for *N*-(3-iodo-4-methyl-5-phenylfuran-2-yl)-*N*-methylmethanesulfonamide(6)



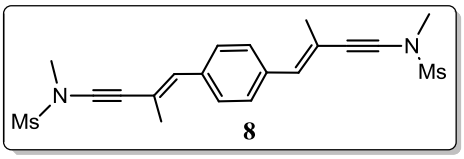
Brown oil; IR (neat, cm^{-1}): 1621 (m), 1596 (m), 1335 (b), 707 (w), 559 (s), 280 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.53 (d, *J* = 7.8 Hz, 2 H), 7.40 (t, *J* = 7.8 Hz, 2 H), 7.32 ~ 7.23 (m, 1 H), 3.30 (s, 3 H), 3.12 (s, 3 H), 2.20 (s, 3 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 147.5, 145.8, 130.3, 128.8, 128.0, 126.0, 120.4, 75.0, 39.1, 37.7, 13.4; HRMS calcd. for $C_{13}H_{14}INO_3S$: 390.9739; found: 390.9739.

Spectral data for (*E*)-*N*,3-dimethyl-*N*-(methylsulfonyl)-4-oxo-4-phenylbut-2-enamide



Brown oil; IR (neat, cm^{-1}): 1700(s), 1668 (s), 1620 (w), 1335 (b), 704 (w); ^1H NMR (600 MHz, $CDCl_3$): δ 7.87 ~ 7.85 (m, 2 H), 7.57 ~ 7.56 (m, 1 H), 7.48 ~ 7.45 (m, 2 H), 6.80 (s, 1 H), 3.19 (s, 3 H), 3.14 (s, 3 H), 2.18 (s, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 198.4, 165.0, 156.4, 134.4, 133.6, 128.8, 128.7, 120.1, 41.4, 32.6, 21.9.

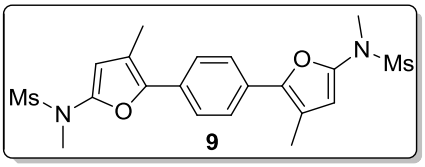
Spectral data for (*E*)-*N*-methyl-*N*-(3-methyl-4-(4-(2-methylprop-1-en-1-yl)phenyl)but-3-en-1-yn-1-yl)methanesulfonamide compound with *N*-ethynyl-*N*-methylmethanesulfonamide (7)



8

Pale yellow solid; melting point 158 °C; IR (KBr, cm^{-1}): 2275 (m), 2150 (m), 1617 (m), 1610 (s), 1575 (b), 1340 (b), 710 (s); ^{1}H NMR (600 MHz, CD_2Cl_2): δ 7.23 (s, 4 H), 6.72 (s, 2 H), 3.25 (s, 6 H), 3.09 (s, 6 H), 2.07 (s, 6 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 135.2, 133.5, 128.9, 119.0, 84.2, 72.5, 38.9, 36.4, 19.6; HRMS calcd. for $C_{20}H_{24}N_2O_4S_2$: 420.5507; found: 420.5509.

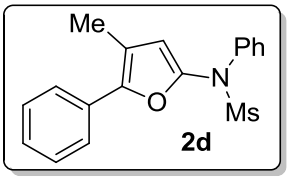
Spectral Data for *N*,*N*'-(5,5'-(1,4-phenylene)bis(4-methylfuran-5,2-diyl))bis(*N*-methylmethanesulfonamide)(9)



9

Pale yellow solid; melting point: 184 °C; IR (KBr, cm^{-1}): 3070 (m), 1610 (m), 1601 (m), 1365 (b), 705 (w); ^{1}H NMR (400 MHz, $CDCl_3$): 7.58 (s, 4 H), 6.18 (s, 2 H), 3.32 (s, 6 H), 3.01 (s, 6 H), 2.25 (s, 6 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.8, 144.9, 129.5, 125.3, 118.3, 109.8, 37.4, 37.3, 12.1; HRMS calcd. for $C_{20}H_{24}N_2O_6S_2$: 452.1073; found: 452.1079.

(V) X –ray crystal structure and data of compound 2d:



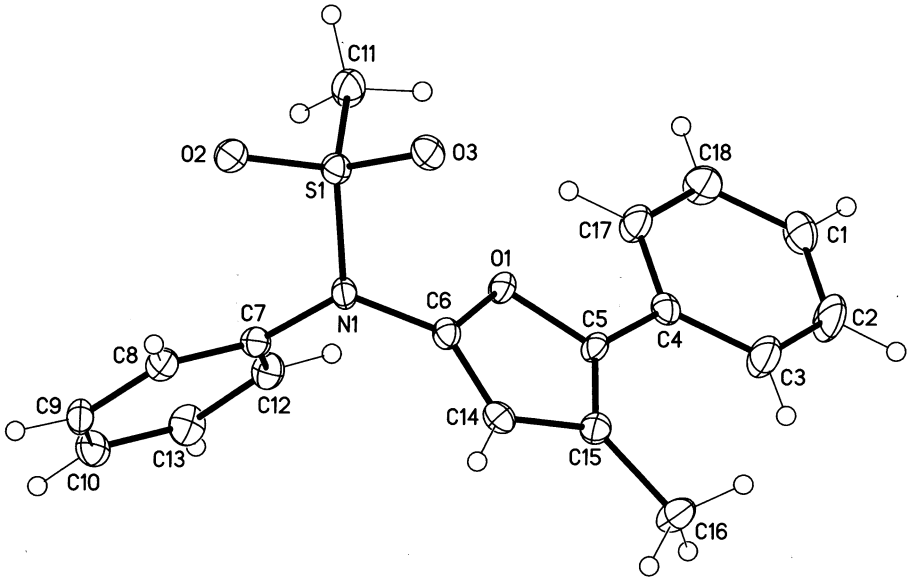


Table 1. Crystal data and structure refinement for mo_111104lt_0m.

Identification code	mo_111104lt_0m
Empirical formula	C18 H17 N O3 S
Formula weight	327.39
Temperature	100(2) K
Wavelength	0.71073 Å

Crystal system	Monoclinic	
Space group	P 1 n 1	
Unit cell dimensions	a = 9.0128(18) Å	α= 90°.
	b = 9.1833(18) Å	β= 110.846(4)°.
	c = 10.1798(19) Å	γ = 90°.
Volume	787.4(3) $Å^3$	
Z	2	
Density (calculated)	1.381 Mg/m^3	
Absorption coefficient	0.220 mm^{-1}	
F(000)	344	
Crystal size	0.22 x 0.18 x 0.15 mm^3	
Theta range for data collection	2.22 to 26.30°.	
Index ranges	-11<=h<=11, -11<=k<=11, -12<=l<=12	
Reflections collected	6296	
Independent reflections	2990 [R(int) = 0.0226]	
Completeness to theta = 26.30°	98.9 %	
Absorption correction	Semi-empirical from equivalents	
Max. and min. transmission	0.9486 and 0.8213	
Refinement method	Full-matrix least-squares on F^2	
Data / restraints / parameters	2990 / 2 / 210	
Goodness-of-fit on F^2	1.030	
Final R indices [I>2sigma(I)]	R1 = 0.0274, wR2 = 0.0615	
R indices (all data)	R1 = 0.0318, wR2 = 0.0634	
Absolute structure parameter	0.09(5)	

Largest diff. peak and hole	0.137 and -0.243 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for mo_111104lt_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
S(1)	3321(1)	11313(1)	3036(1)	16(1)
O(1)	3741(1)	7934(1)	2662(1)	16(1)
O(2)	2407(2)	12532(1)	3189(1)	22(1)
O(3)	4992(2)	11250(1)	3818(1)	20(1)
N(1)	2548(2)	9837(2)	3501(2)	15(1)
C(1)	5738(2)	4274(2)	66(2)	24(1)
C(2)	6154(3)	3751(2)	1411(2)	32(1)
C(3)	5771(3)	4496(2)	2432(2)	28(1)
C(4)	4933(2)	5803(2)	2097(2)	16(1)
C(5)	4514(2)	6619(2)	3151(2)	15(1)
C(6)	3458(2)	8555(2)	3775(2)	15(1)
C(7)	836(2)	9659(2)	3071(2)	16(1)
C(8)	-21(2)	10570(2)	3627(2)	18(1)
C(9)	-1640(2)	10341(2)	3270(2)	19(1)
C(10)	-2390(2)	9197(2)	2399(2)	22(1)
C(11)	3005(2)	11088(2)	1237(2)	23(1)
C(12)	87(2)	8535(2)	2166(2)	19(1)
C(13)	-1518(2)	8295(2)	1845(2)	22(1)
C(14)	4011(2)	7710(2)	4922(2)	16(1)

C(15)	4689(2)	6437(2)	4532(2)	16(1)
C(16)	5390(2)	5180(2)	5493(2)	23(1)
C(17)	4493(2)	6313(2)	728(2)	22(1)
C(18)	4898(2)	5565(2)	-278(2)	24(1)

Table 3. Bond lengths [Å] and angles [°] for mo_111104lt_0m.

S(1)-O(2)	1.4311(13)
S(1)-O(3)	1.4322(14)
S(1)-N(1)	1.6681(16)
S(1)-C(11)	1.762(2)
O(1)-C(6)	1.372(2)
O(1)-C(5)	1.394(2)
N(1)-C(6)	1.405(2)
N(1)-C(7)	1.455(2)
C(1)-C(2)	1.371(3)
C(1)-C(18)	1.383(3)
C(1)-H(1)	0.9500
C(2)-C(3)	1.388(3)
C(2)-H(2)	0.9500
C(3)-C(4)	1.394(3)
C(3)-H(3)	0.9500
C(4)-C(17)	1.388(3)
C(4)-C(5)	1.464(3)
C(5)-C(15)	1.368(3)
C(6)-C(14)	1.341(3)
C(7)-C(12)	1.388(3)
C(7)-C(8)	1.389(3)
C(8)-C(9)	1.389(3)

C(8)-H(8)	0.9500
C(9)-C(10)	1.386(3)
C(9)-H(9)	0.9500
C(10)-C(13)	1.392(3)
C(10)-H(10)	0.9500
C(11)-H(11A)	0.9800
C(11)-H(11B)	0.9800
C(11)-H(11C)	0.9800
C(12)-C(13)	1.382(3)
C(12)-H(12)	0.9500
C(13)-H(13)	0.9500
C(14)-C(15)	1.439(3)
C(14)-H(14)	0.9500
C(15)-C(16)	1.500(3)
C(16)-H(16A)	0.9800
C(16)-H(16B)	0.9800
C(16)-H(16C)	0.9800
C(17)-C(18)	1.386(3)
C(17)-H(17)	0.9500
C(18)-H(18)	0.9500

O(2)-S(1)-O(3)	120.22(8)
O(2)-S(1)-N(1)	106.85(8)
O(3)-S(1)-N(1)	105.80(7)

O(2)-S(1)-C(11)	108.59(9)
O(3)-S(1)-C(11)	108.78(9)
N(1)-S(1)-C(11)	105.66(9)
C(6)-O(1)-C(5)	106.36(14)
C(6)-N(1)-C(7)	116.57(14)
C(6)-N(1)-S(1)	117.64(12)
C(7)-N(1)-S(1)	120.74(11)
C(2)-C(1)-C(18)	118.94(19)
C(2)-C(1)-H(1)	120.5
C(18)-C(1)-H(1)	120.5
C(1)-C(2)-C(3)	121.52(19)
C(1)-C(2)-H(2)	119.2
C(3)-C(2)-H(2)	119.2
C(2)-C(3)-C(4)	119.9(2)
C(2)-C(3)-H(3)	120.0
C(4)-C(3)-H(3)	120.0
C(17)-C(4)-C(3)	118.20(18)
C(17)-C(4)-C(5)	120.46(16)
C(3)-C(4)-C(5)	121.34(18)
C(15)-C(5)-O(1)	109.47(15)
C(15)-C(5)-C(4)	136.43(16)
O(1)-C(5)-C(4)	114.10(16)
C(14)-C(6)-O(1)	110.93(16)
C(14)-C(6)-N(1)	131.90(18)

O(1)-C(6)-N(1)	116.85(16)
C(12)-C(7)-C(8)	120.58(17)
C(12)-C(7)-N(1)	119.77(16)
C(8)-C(7)-N(1)	119.56(16)
C(9)-C(8)-C(7)	119.16(17)
C(9)-C(8)-H(8)	120.4
C(7)-C(8)-H(8)	120.4
C(10)-C(9)-C(8)	120.66(18)
C(10)-C(9)-H(9)	119.7
C(8)-C(9)-H(9)	119.7
C(9)-C(10)-C(13)	119.57(18)
C(9)-C(10)-H(10)	120.2
C(13)-C(10)-H(10)	120.2
S(1)-C(11)-H(11A)	109.5
S(1)-C(11)-H(11B)	109.5
H(11A)-C(11)-H(11B)	109.5
S(1)-C(11)-H(11C)	109.5
H(11A)-C(11)-H(11C)	109.5
H(11B)-C(11)-H(11C)	109.5
C(13)-C(12)-C(7)	119.77(17)
C(13)-C(12)-H(12)	120.1
C(7)-C(12)-H(12)	120.1
C(12)-C(13)-C(10)	120.22(18)
C(12)-C(13)-H(13)	119.9

C(10)-C(13)-H(13)	119.9
C(6)-C(14)-C(15)	107.03(17)
C(6)-C(14)-H(14)	126.5
C(15)-C(14)-H(14)	126.5
C(5)-C(15)-C(14)	106.21(16)
C(5)-C(15)-C(16)	129.27(17)
C(14)-C(15)-C(16)	124.51(18)
C(15)-C(16)-H(16A)	109.5
C(15)-C(16)-H(16B)	109.5
H(16A)-C(16)-H(16B)	109.5
C(15)-C(16)-H(16C)	109.5
H(16A)-C(16)-H(16C)	109.5
H(16B)-C(16)-H(16C)	109.5
C(18)-C(17)-C(4)	121.27(18)
C(18)-C(17)-H(17)	119.4
C(4)-C(17)-H(17)	119.4
C(1)-C(18)-C(17)	120.12(19)
C(1)-C(18)-H(18)	119.9
C(17)-C(18)-H(18)	119.9

Symmetry transformations used to generate equivalent atoms:

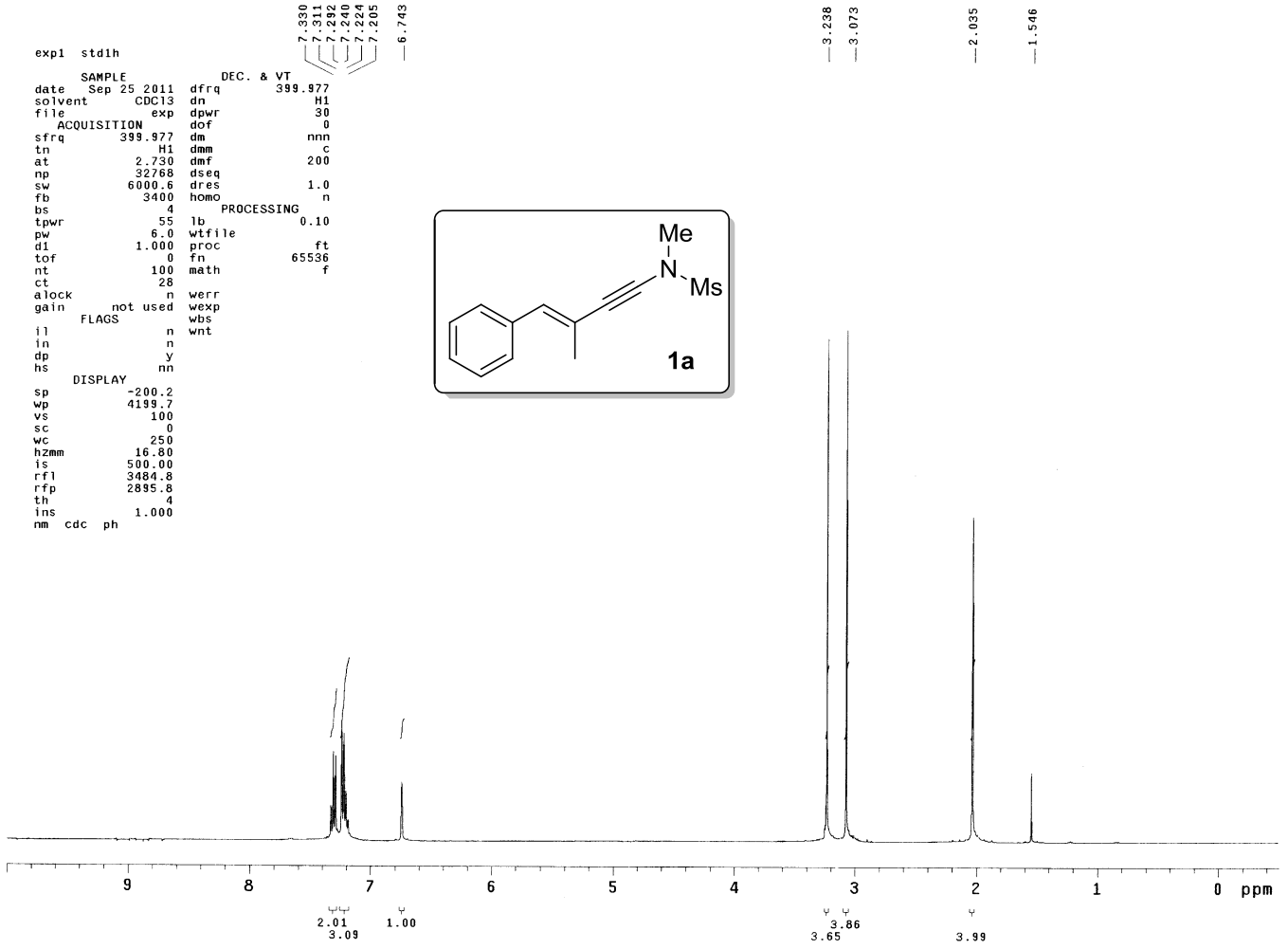
Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for mo_111104lt_0m. The anisotropic displacement factor exponent takes the form: $-2\pi^2[\ h^2\ a^{*2}U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
S(1)	16(1)	14(1)	19(1)	-1(1)	8(1)	0(1)
O(1)	18(1)	12(1)	18(1)	1(1)	7(1)	4(1)
O(2)	24(1)	15(1)	30(1)	2(1)	14(1)	2(1)
O(3)	15(1)	21(1)	24(1)	0(1)	7(1)	-2(1)
N(1)	11(1)	13(1)	20(1)	-1(1)	6(1)	1(1)
C(1)	25(1)	22(1)	28(1)	-6(1)	13(1)	3(1)
C(2)	41(1)	21(1)	37(1)	2(1)	18(1)	16(1)
C(3)	36(1)	23(1)	28(1)	5(1)	15(1)	13(1)
C(4)	13(1)	13(1)	22(1)	-2(1)	7(1)	-2(1)
C(5)	13(1)	9(1)	22(1)	2(1)	5(1)	0(1)
C(6)	14(1)	12(1)	18(1)	-2(1)	6(1)	1(1)
C(7)	13(1)	18(1)	16(1)	2(1)	4(1)	0(1)
C(8)	21(1)	17(1)	15(1)	0(1)	7(1)	2(1)
C(9)	16(1)	20(1)	20(1)	3(1)	7(1)	6(1)
C(10)	14(1)	29(1)	21(1)	4(1)	4(1)	1(1)
C(11)	22(1)	27(1)	19(1)	3(1)	8(1)	6(1)
C(12)	20(1)	18(1)	18(1)	-2(1)	6(1)	2(1)
C(13)	18(1)	21(1)	22(1)	-4(1)	2(1)	-2(1)
C(14)	14(1)	18(1)	17(1)	-2(1)	6(1)	-3(1)

C(15)	15(1)	14(1)	19(1)	0(1)	5(1)	-1(1)
C(16)	32(1)	17(1)	21(1)	4(1)	10(1)	4(1)
C(17)	26(1)	16(1)	24(1)	1(1)	10(1)	6(1)
C(18)	30(1)	26(1)	18(1)	0(1)	10(1)	4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for mo_111104lt_0m.

	x	y	z	U(eq)
H(1)	6022	3758	-618	29
H(2)	6718	2857	1648	38
H(3)	6078	4116	3359	33
H(8)	494	11341	4244	21
H(9)	-2240	10975	3628	23
H(10)	-3492	9030	2181	26
H(11A)	1892	10831	729	34
H(11B)	3692	10309	1124	34
H(11C)	3254	11999	859	34
H(12)	675	7933	1769	22
H(13)	-2027	7512	1244	27
H(14)	3964	7913	5821	19
H(16A)	4881	4273	5050	35
H(16B)	5216	5319	6382	35
H(16C)	6533	5126	5677	35
H(17)	3902	7191	476	26
H(18)	4598	5940	-1207	29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7.292
7.240
7.224
7.205
6.743
3.238
3.073
2.035
1.546
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date Sep 25 2011
solvent CDCl3
file exp
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tn H1
at 2.730
np 32768
sw 6000.6
fb 3400
bs 4
tpwr 55
pw 6.0
d1 1.000
tof 0
nt 100
ct 28
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -200.2
wp 4199.7
vs 100
sc 0
wc 250
hzmm 16.80
is 500.00
rfl 3484.8
rfp 2895.8
th 4
ins 1.000
nm cdc ph
DEC. & VT
dfrq 399.977
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb 0.10
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
Me
N
Ms
1a
9
8
7
6
5
4
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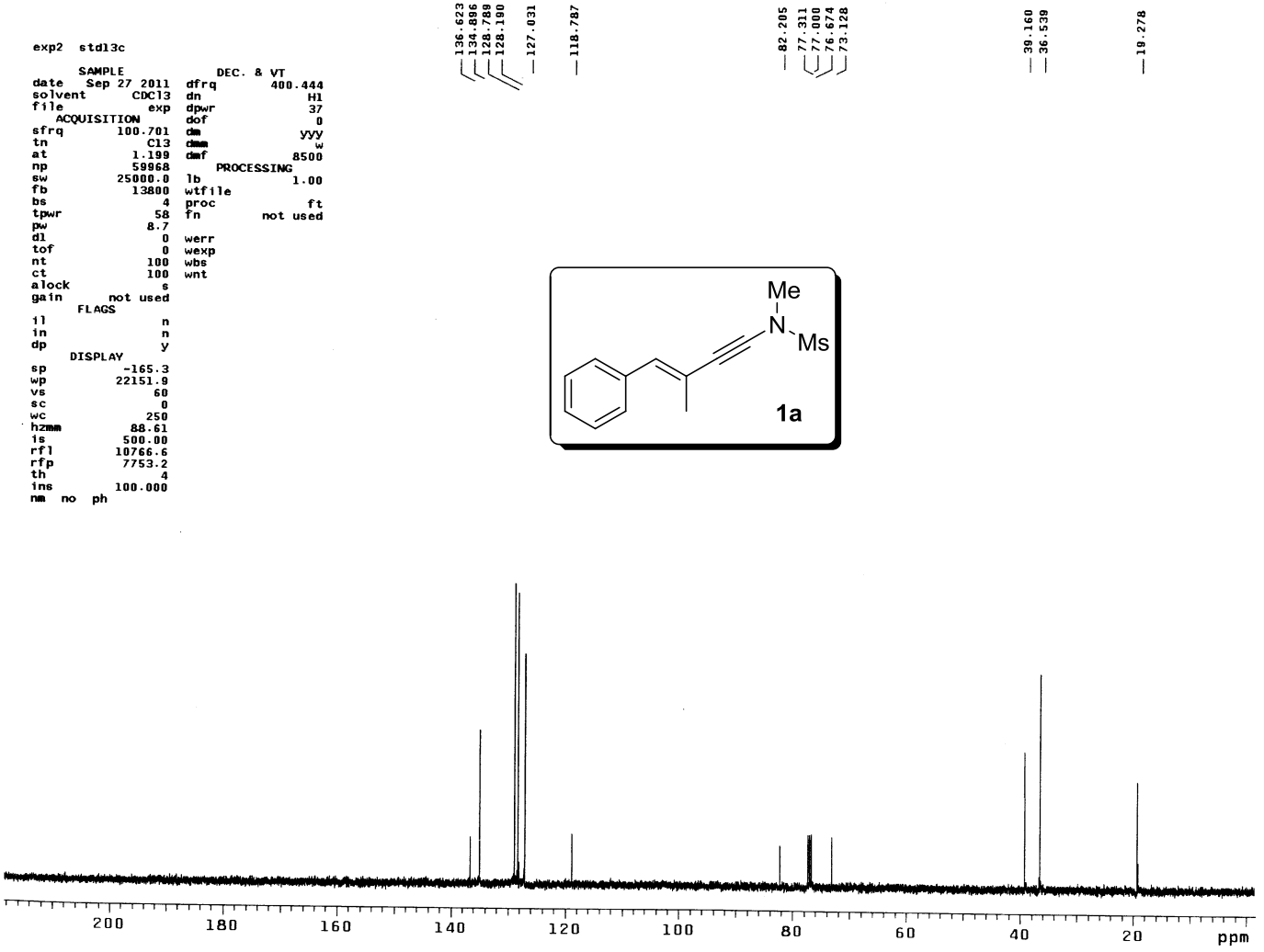
2.01
3.09
1.00
3.86
3.65
3.99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date Sep 27 2011
solvent CDCl3
file exp
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tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 100
ct 100
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -165.3
wp 22151.9
vs 60
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10766.6
rfp 7753.2
th 4
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
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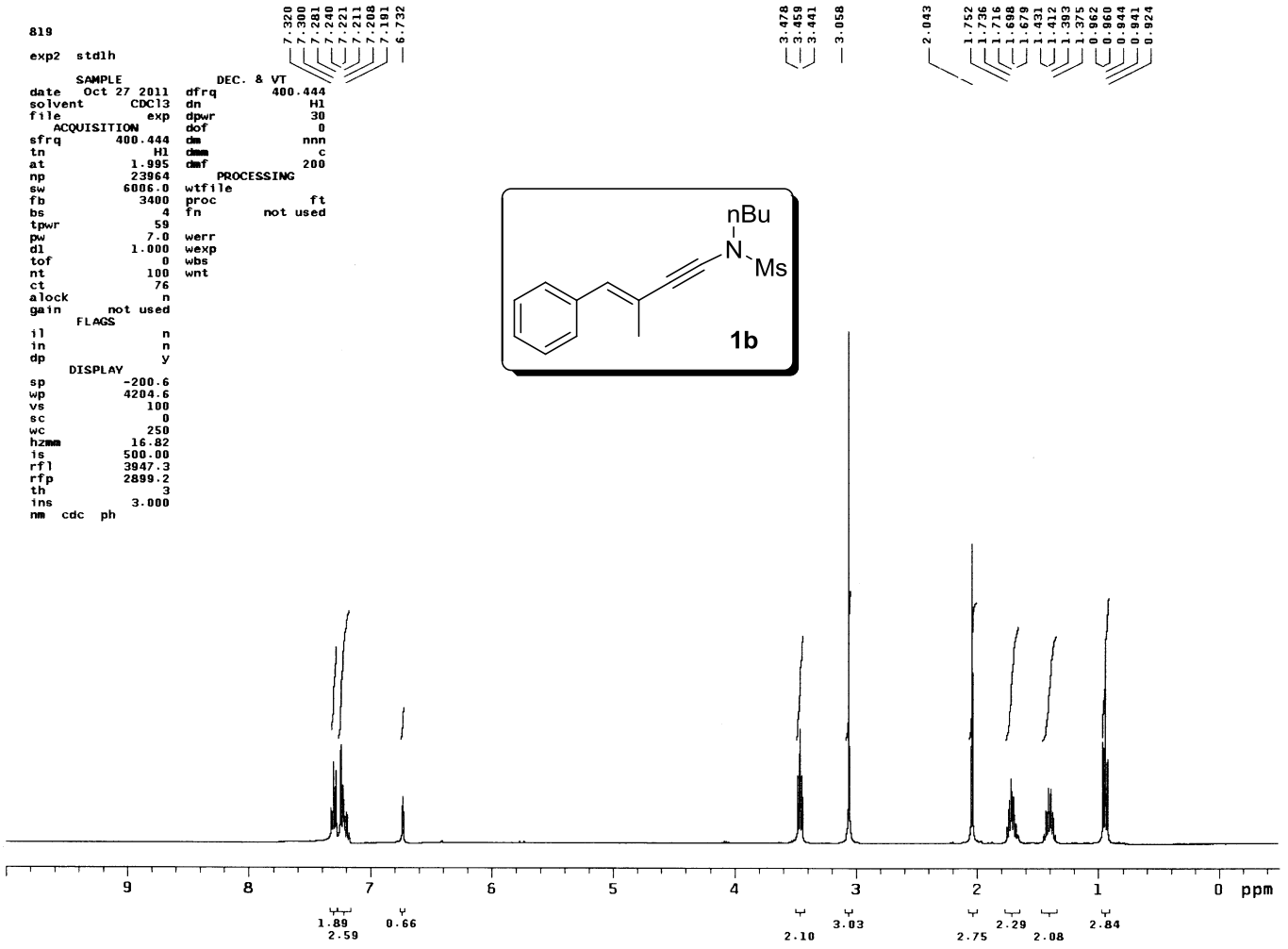
134.896
128.789
128.190
127.031
118.787
82.205
77.311
77.000
76.674
73.128
39.160
36.539
19.278
Me
N
Ms
1a
200
180
160
140
120
100
80
60
40
20
ppm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date Oct 27 2011
solvent CDCl3
file exp
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at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
d1 1.000
tof 0
nt 100
ct 76
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 100
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3947.3
rfp 2899.2
th 3
ins 3.000
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
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7.300
7.281
7.240
7.221
7.211
7.208
7.191
6.732
3.478
3.459
3.441
3.058
2.043
1.752
1.736
1.716
1.698
1.679
1.431
1.412
1.393
1.375
0.962
0.960
0.944
0.941
0.924
nBu
N
Ms
1b
9
8
7
6
5
4
3
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0 ppm
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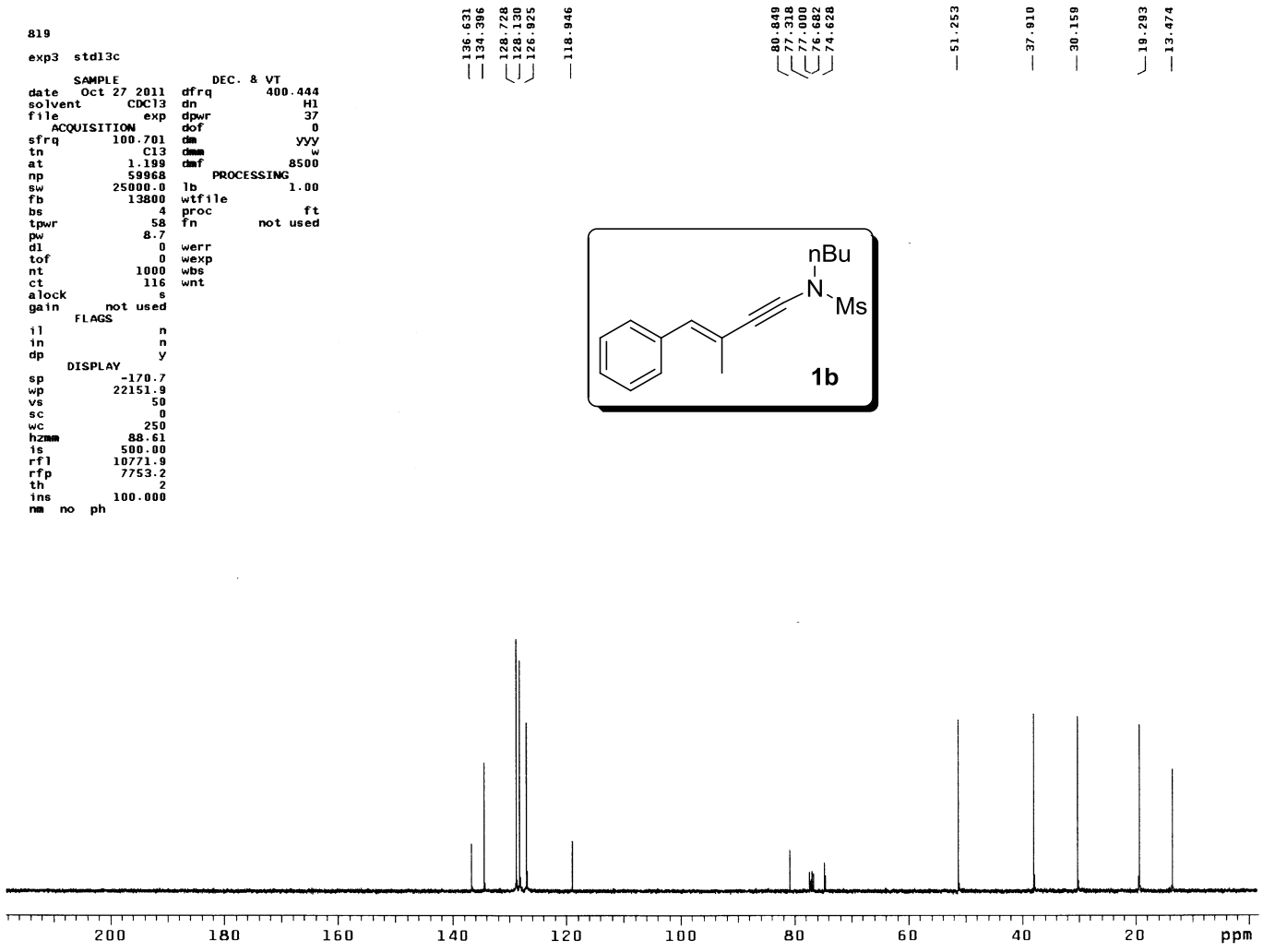
3.03
2.75
2.29
2.08
2.84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solvent CDCl3
file exp
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tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 1000
ct 116
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -170.7
wp 22151.9
vs 50
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10771.9
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
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proc ft
fn not used
werr
wexp
wbs
wnt
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128.130
126.925
118.946
80.849
77.318
77.000
76.682
74.628
51.253
37.910
30.159
19.293
13.474
nBu
N
Ms
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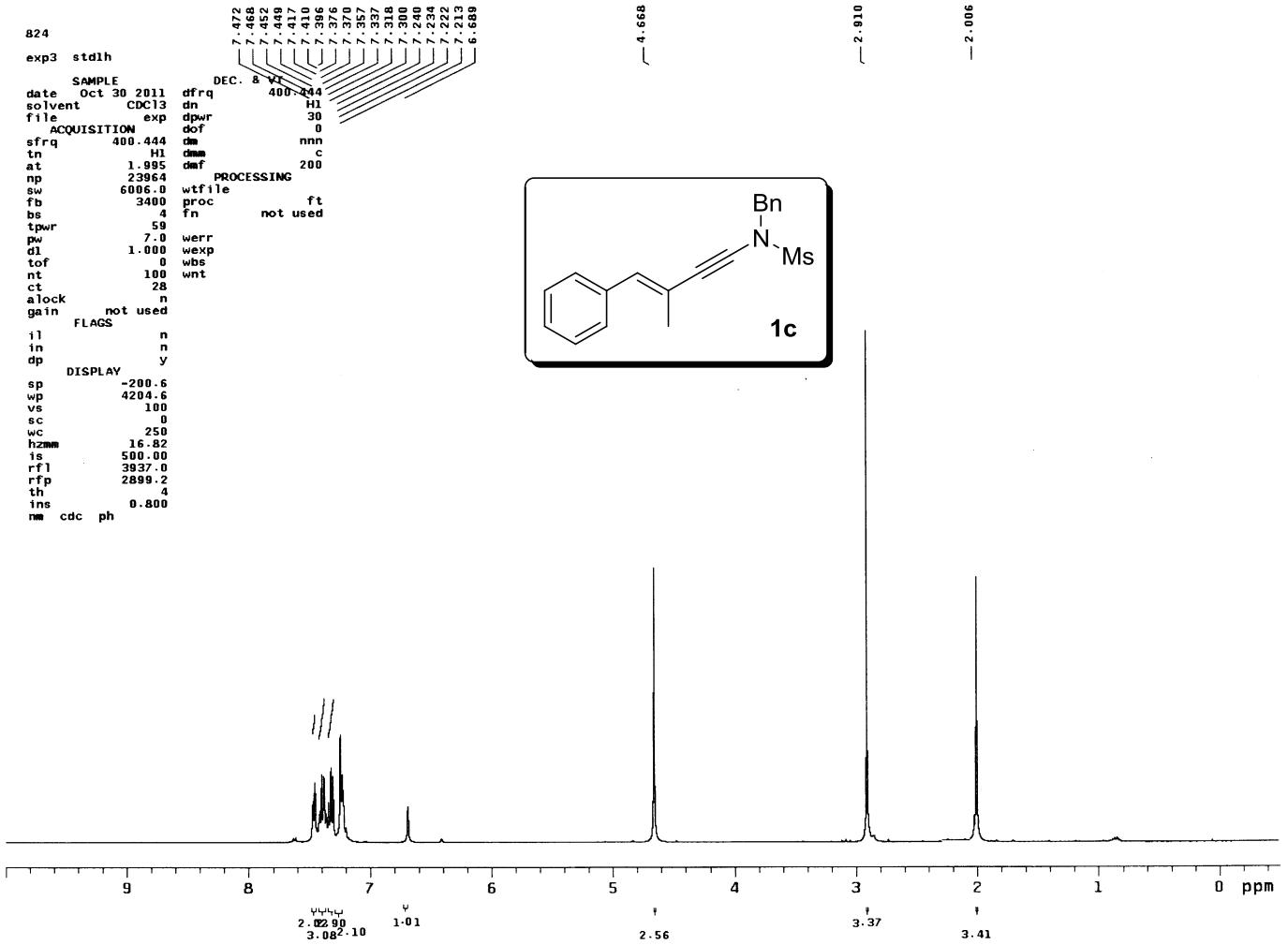
160
140
120
100
80
60
40
20
ppm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solvent CDCl3
file exp
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tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
dl 1.000
tof 0
nt 100
ct 28
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 100
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3937.0
rfp 2899.2
th 4
ins 0.800
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
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7.468
7.452
7.449
7.417
7.410
7.396
7.376
7.370
7.357
7.337
7.318
7.300
7.240
7.234
7.222
7.213
6.689
4.668
2.910
2.006
Bn
N
Ms
1c
9
8
7
6
5
4
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2
1
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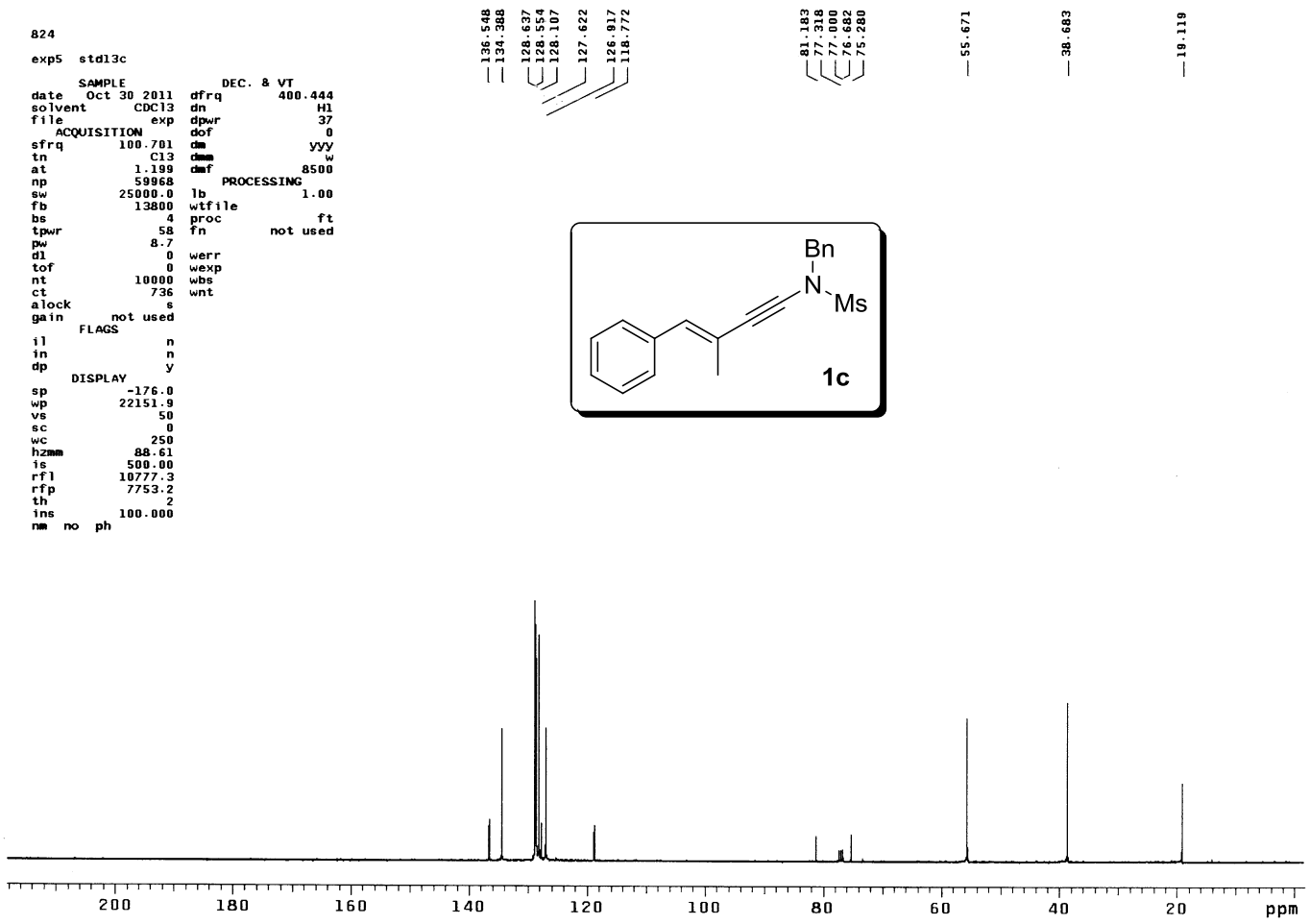
2.10
1.01
2.56
3.37
3.41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solvent CDCl3
file exp
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tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 10000
ct 736
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -176.0
wp 22151.9
vs 50
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10777.3
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
136.548
134.388
128.637
128.554
128.107
127.622
126.917
118.772
81.183
77.318
77.000
76.682
75.280
55.671
38.683
19.119
Bn
N
Ms
1c
200
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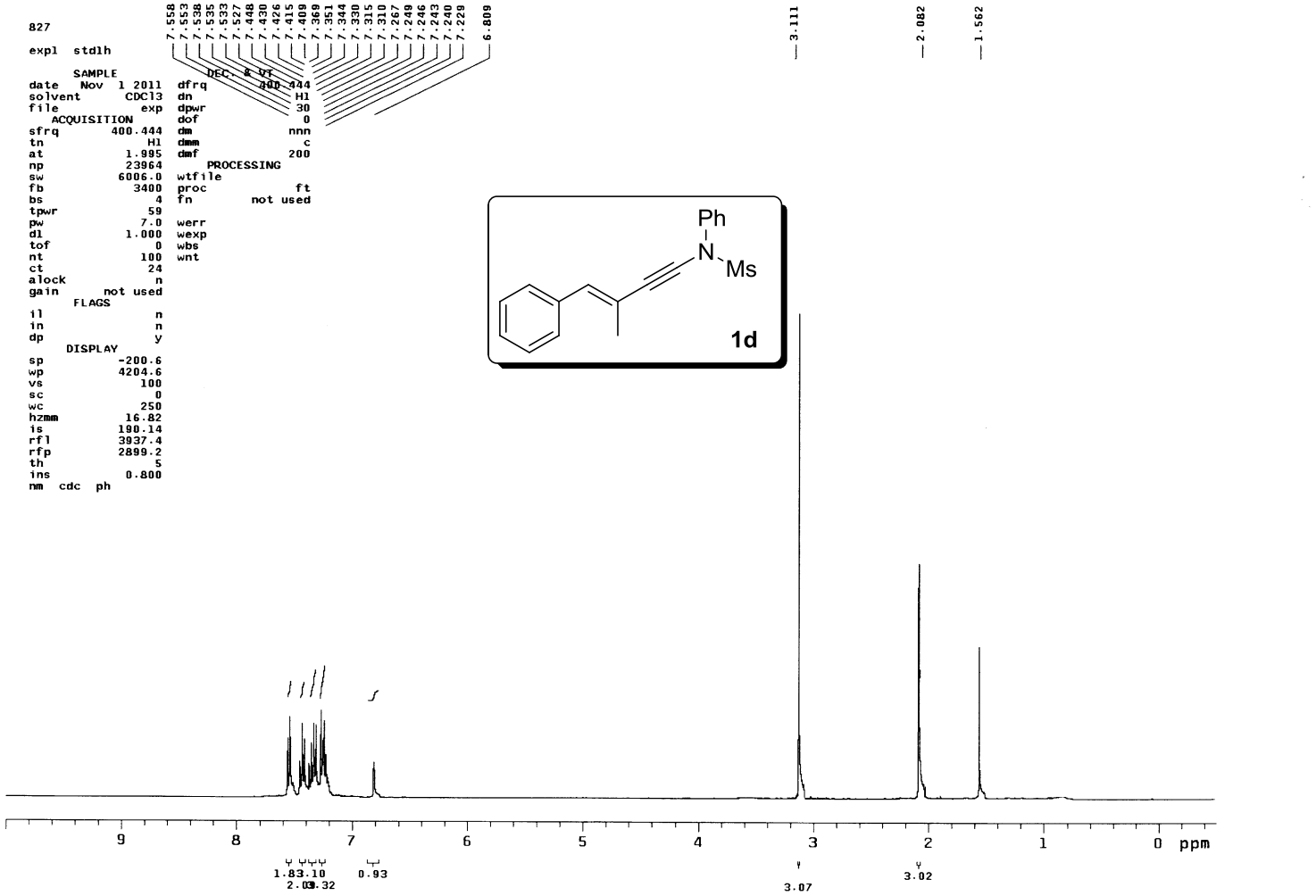
160
140
120
100
80
60
40
20
ppm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solvent CDCl3
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fb 3400
bs 4
tpwr 59
pw 7.0
d1 1.000
tof 0
nt 100
ct 24
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 100
sc 0
wc 250
hzmm 16.82
is 190.14
rfl 3937.4
rfp 2899.2
th 5
ins 0.800
nm cdc ph
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
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fn not used
werr
wexp
wbs
wnt
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7.538
7.535
7.533
7.527
7.448
7.430
7.426
7.415
7.409
7.369
7.351
7.344
7.330
7.315
7.310
7.267
7.249
7.246
7.243
7.240
7.229
6.809
3.111
2.082
1.562
Ph
N
Ms
1d
9
8
7
6
5
4
3
2
1
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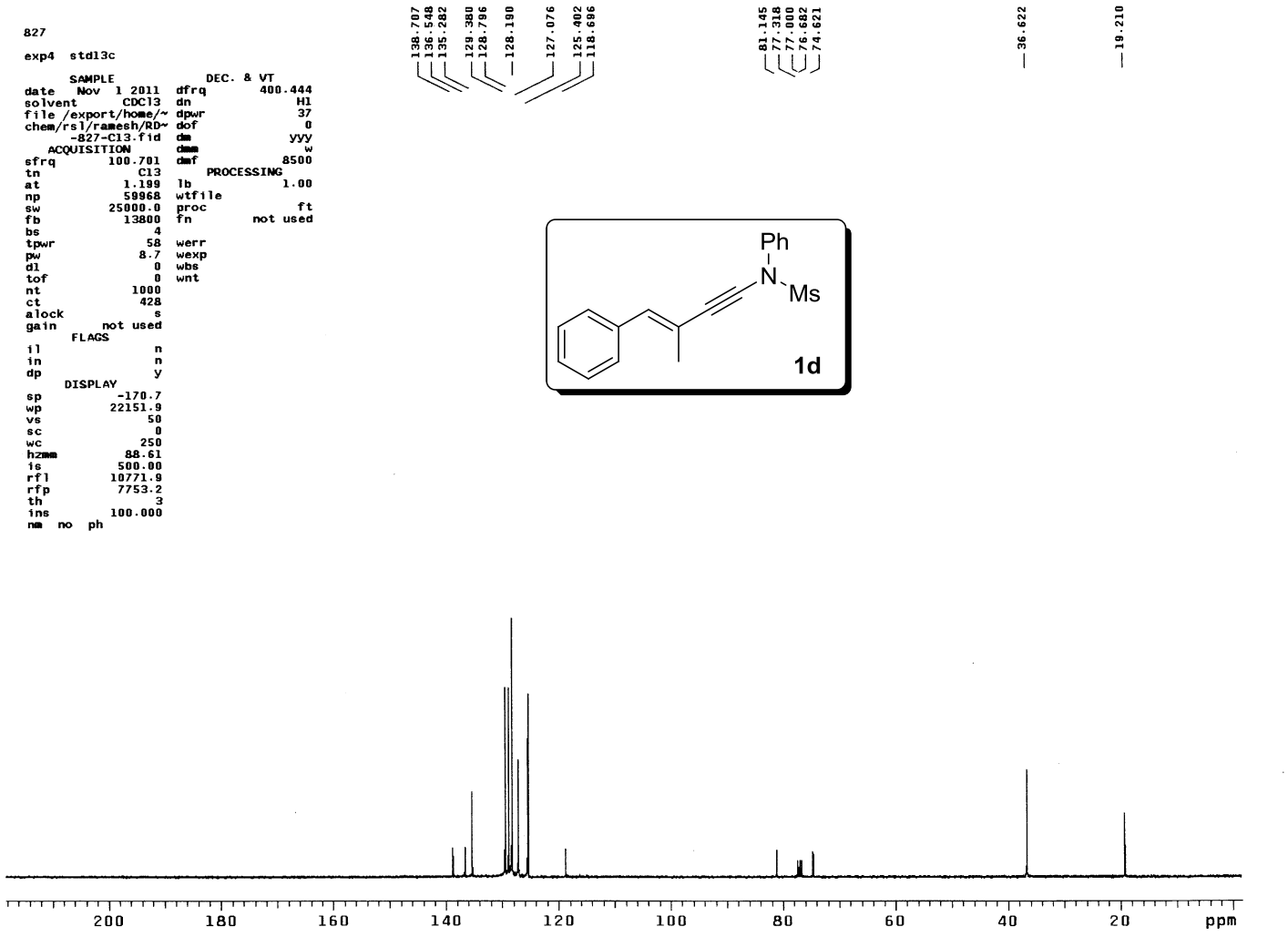
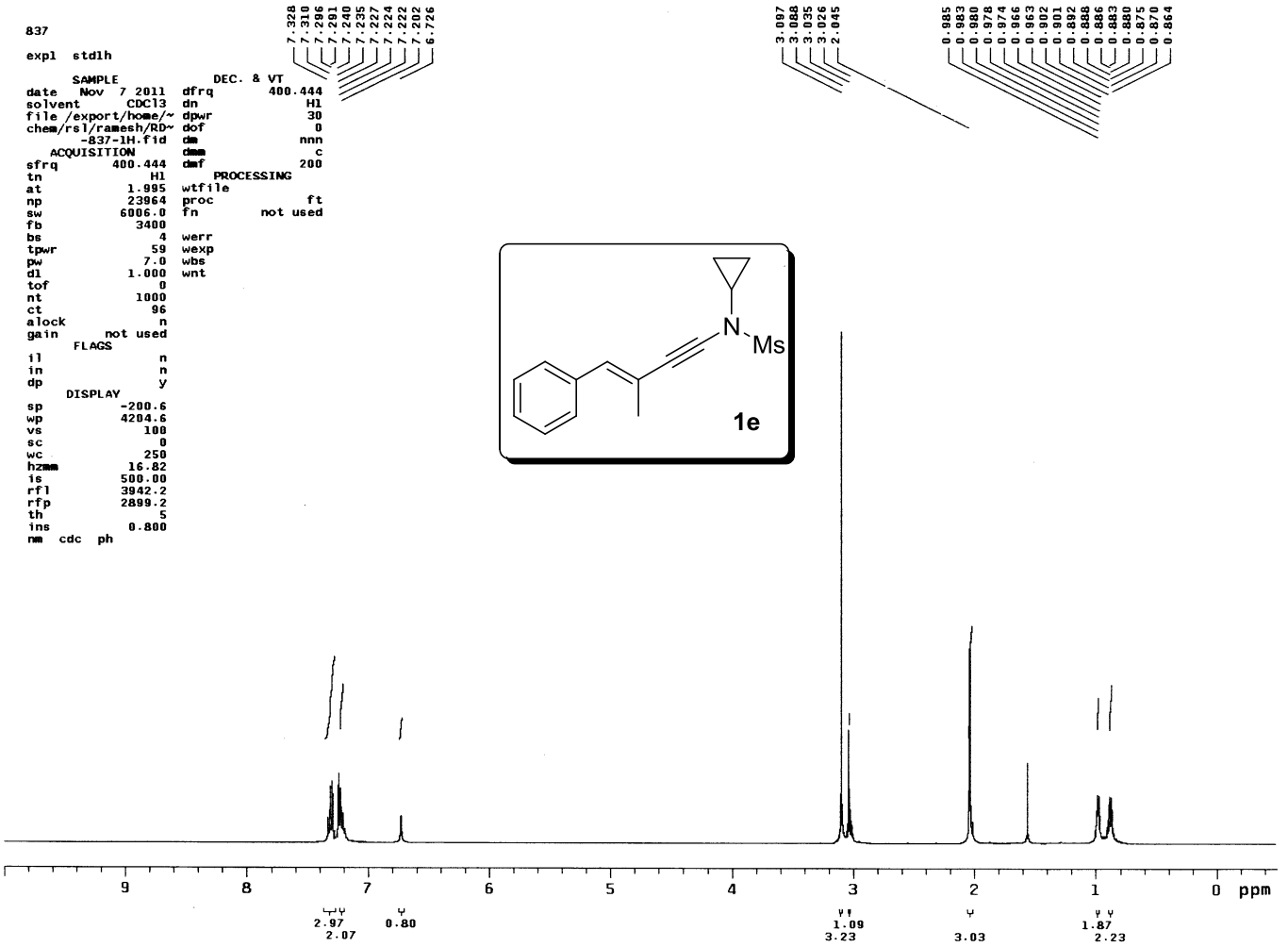
2.03
3.32
0.93
3.07
3.02

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solvent CDCl3
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tn H1
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np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
dl 1.000
tof 0
nt 1000
ct 96
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 100
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3942.2
rfp 2899.2
th 5
ins 0.800
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.328
7.310
7.296
7.291
7.240
7.235
7.227
7.224
7.222
7.202
6.726
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3.088
3.035
3.026
2.045
0.985
0.983
0.980
0.978
0.974
0.966
0.963
0.902
0.901
0.892
0.888
0.886
0.883
0.880
0.875
0.870
0.864
N
Ms
1e
9
8
7
6
5
4
3
2
1
0 ppm
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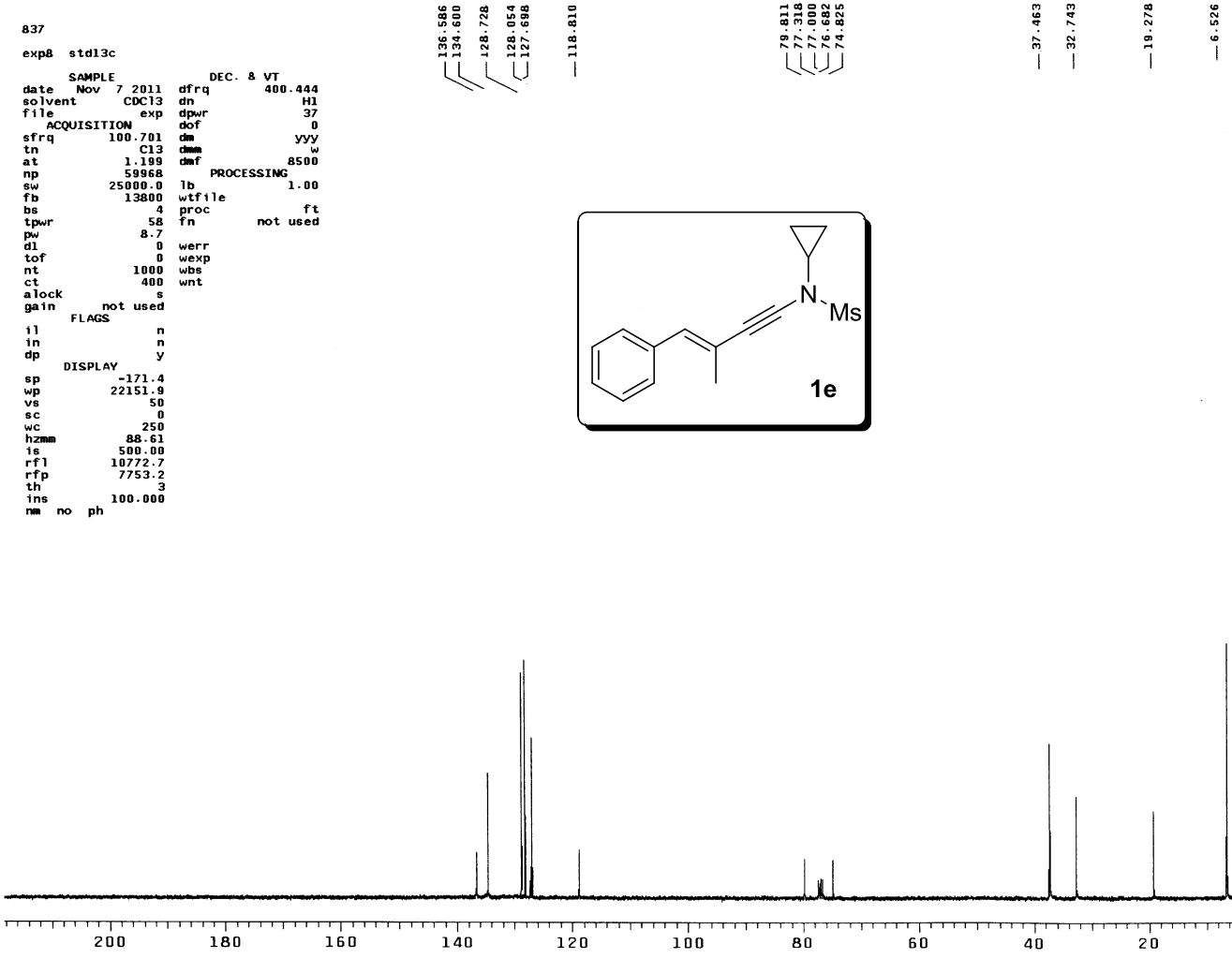
3.23
3.03
1.87
2.23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solvent CDCl3
file exp
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sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 1000
ct 400
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -171.4
wp 22151.9
vs 50
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10772.7
rfp 7753.2
th 3
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
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wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
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134.600
128.728
128.054
127.698
118.810
79.811
77.318
77.000
76.682
74.825
37.463
32.743
19.278
6.526
N
Ms
1e
200
180
160
140
120
100
80
60
40
20
ppm

```
Current Data Parameters
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EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20120322
Time                  18.29
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG                  zg
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8389.262 Hz
FIDRES                0.256020 Hz
AQ                 1.9530228 sec
RG                      128
DW                    59.600 usec
DE                     6.50 usec
TE                    296.5 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     8.00 usec
PL1                    0.00 dB
SFO1            598.9029945 MHz

F2 - Processing parameters
SI                     16384
SF                598.9000794 MHz
WDW                       EM
SSB                       0
LB                     2.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                    10.00 cm
F1P                  10.000 ppm
F1                 5989.00 Hz
F2P                  -0.500 ppm
F2                 -299.45 Hz
PPMCM                 0.52500 ppm/cm
HZCM               314.42255 Hz/cm
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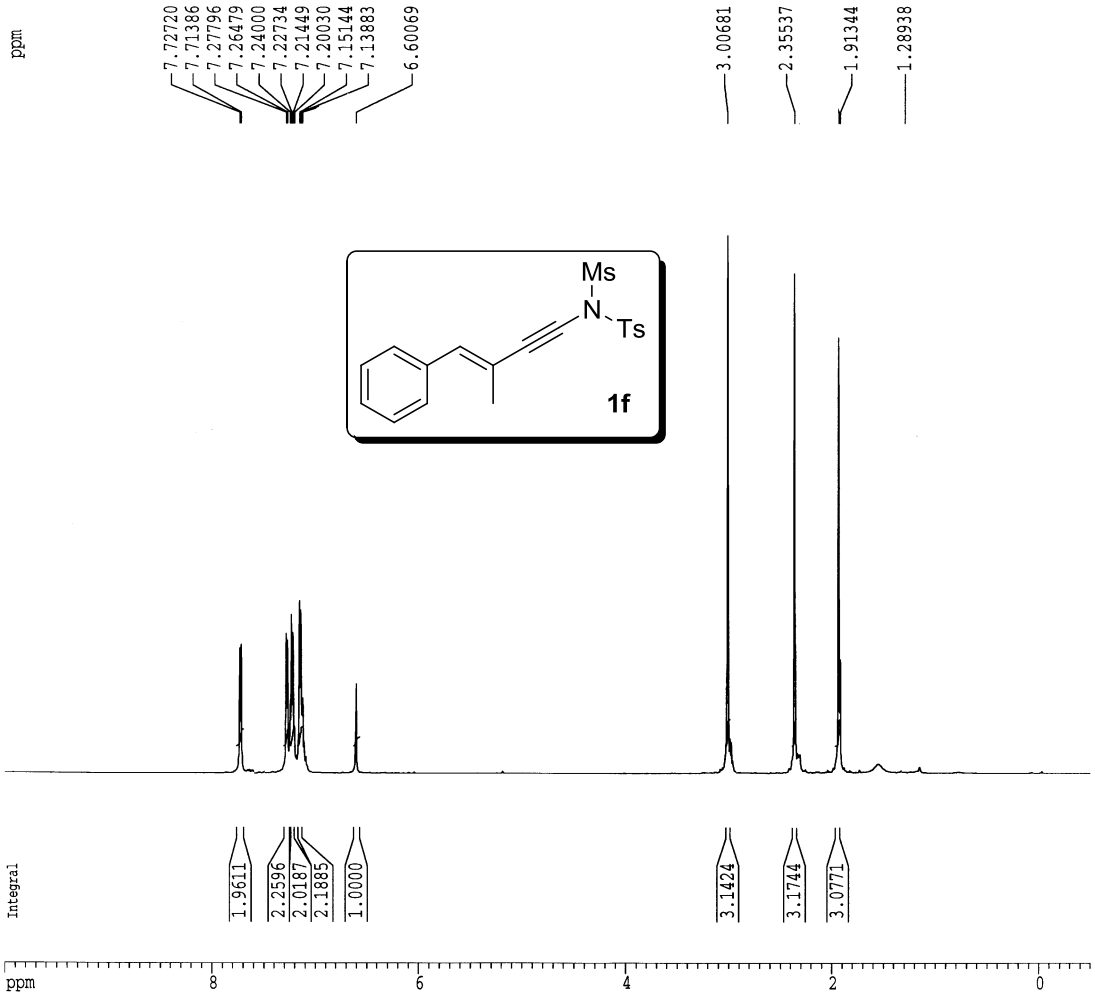

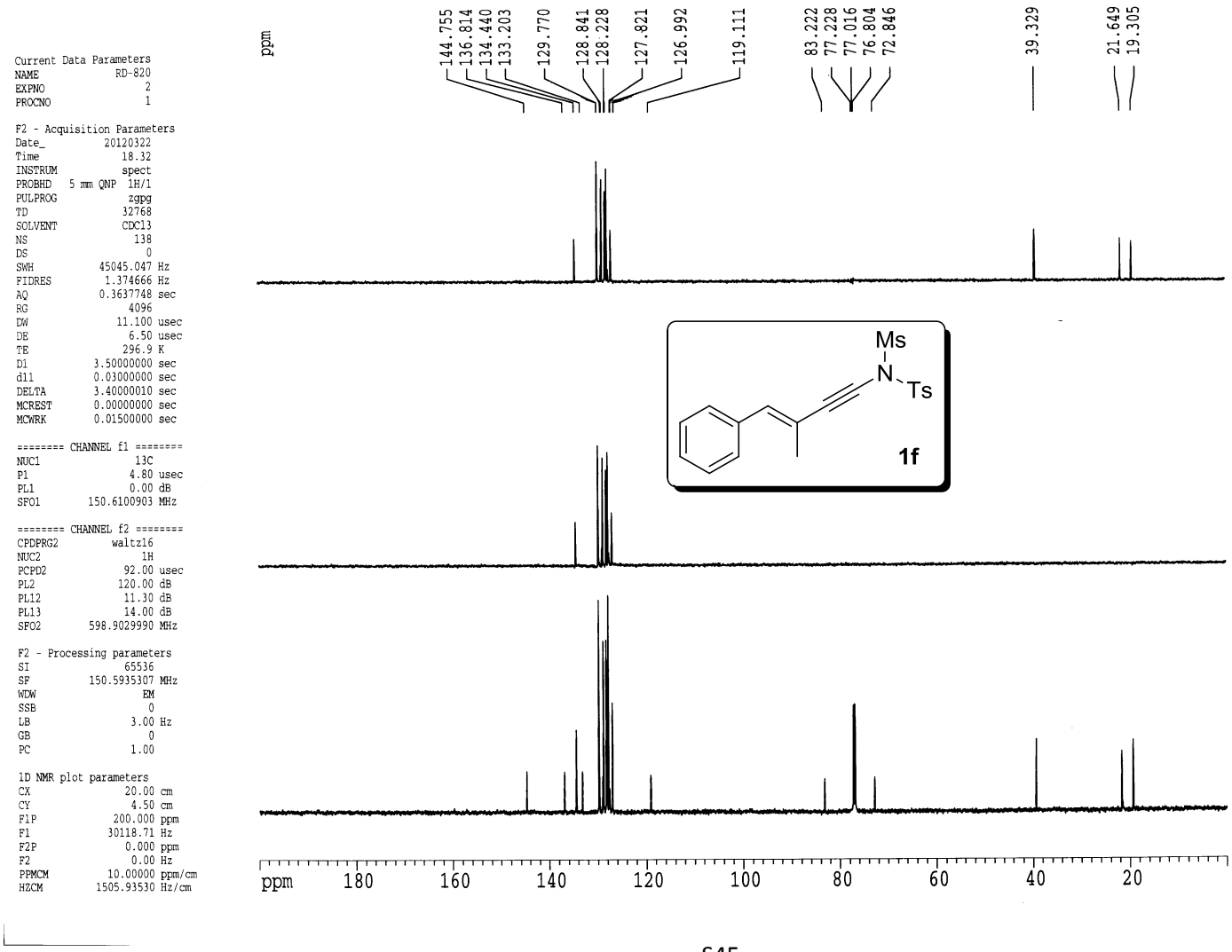


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EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20120322
Time                  18.32
INSTRUM               spect
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PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                      138
DS                        0
SWH               45045.047 Hz
FIDRES             1.374666 Hz
AQ                0.3637748 sec
RG                     4096
DW                   11.100 usec
DE                    6.50 usec
TE                   296.9 K
D1                3.50000000 sec
d11               0.03000000 sec
DELTA             3.40000010 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     13C
P1                    4.80 usec
PL1                   0.00 dB
SFO1            150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                     1H
PCPD2                92.00 usec
PL2                 120.00 dB
PL12                 11.30 dB
PL13                 14.00 dB
SFO2            598.9029990 MHz

F2 - Processing parameters
SI                    65536
SF              150.5935307 MHz
WDW                      EM
SSB                       0
LB                    3.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                   20.00 cm
CY                    4.50 cm
F1P                200.000 ppm
F1                30118.71 Hz
F2P                  0.000 ppm
F2                   0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM            1505.93530 Hz/cm
```
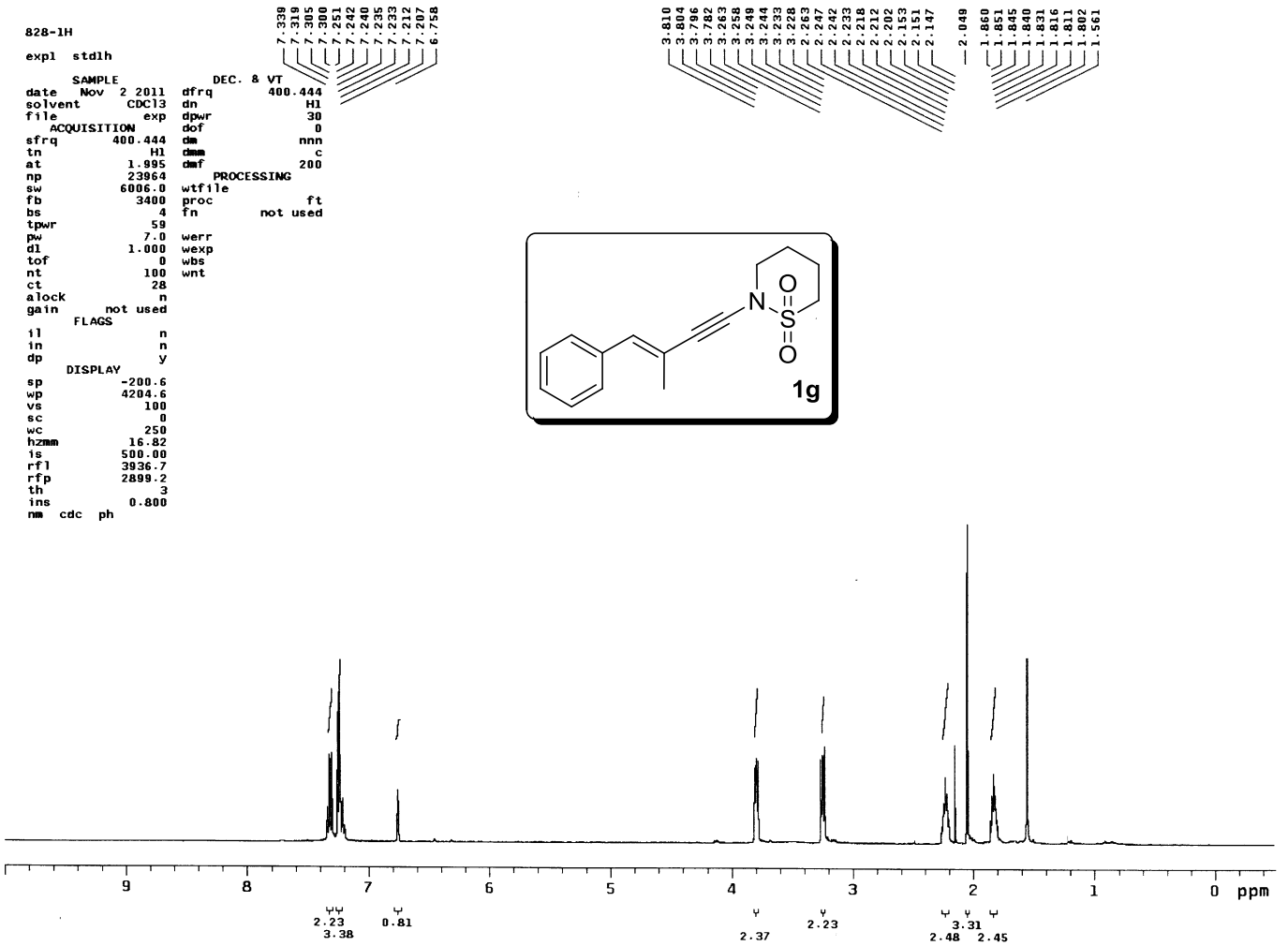
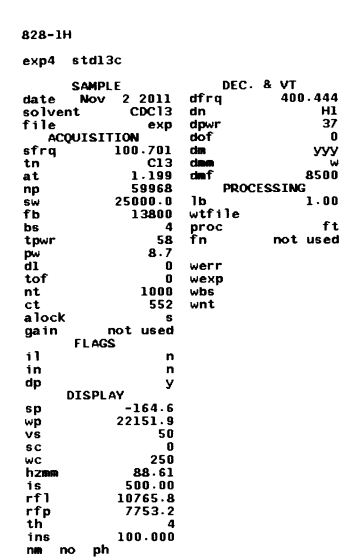
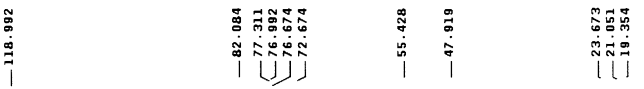
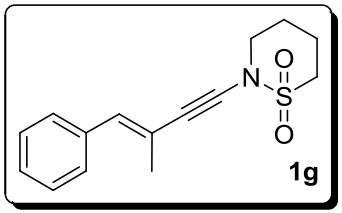
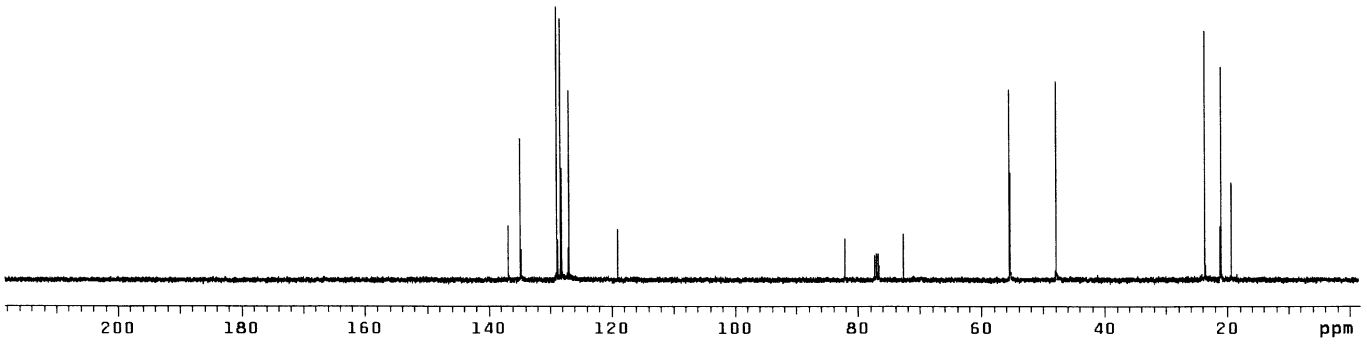
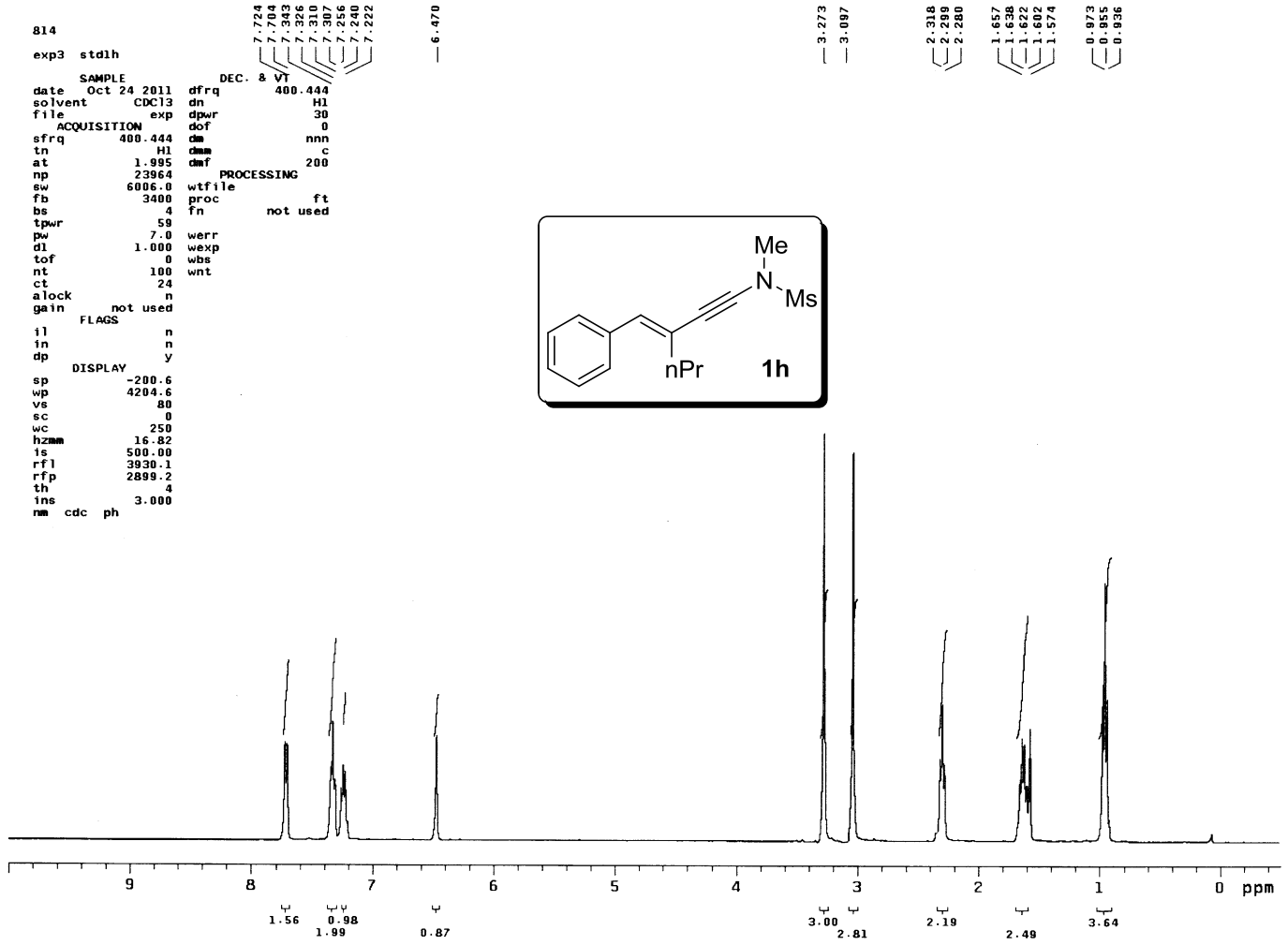



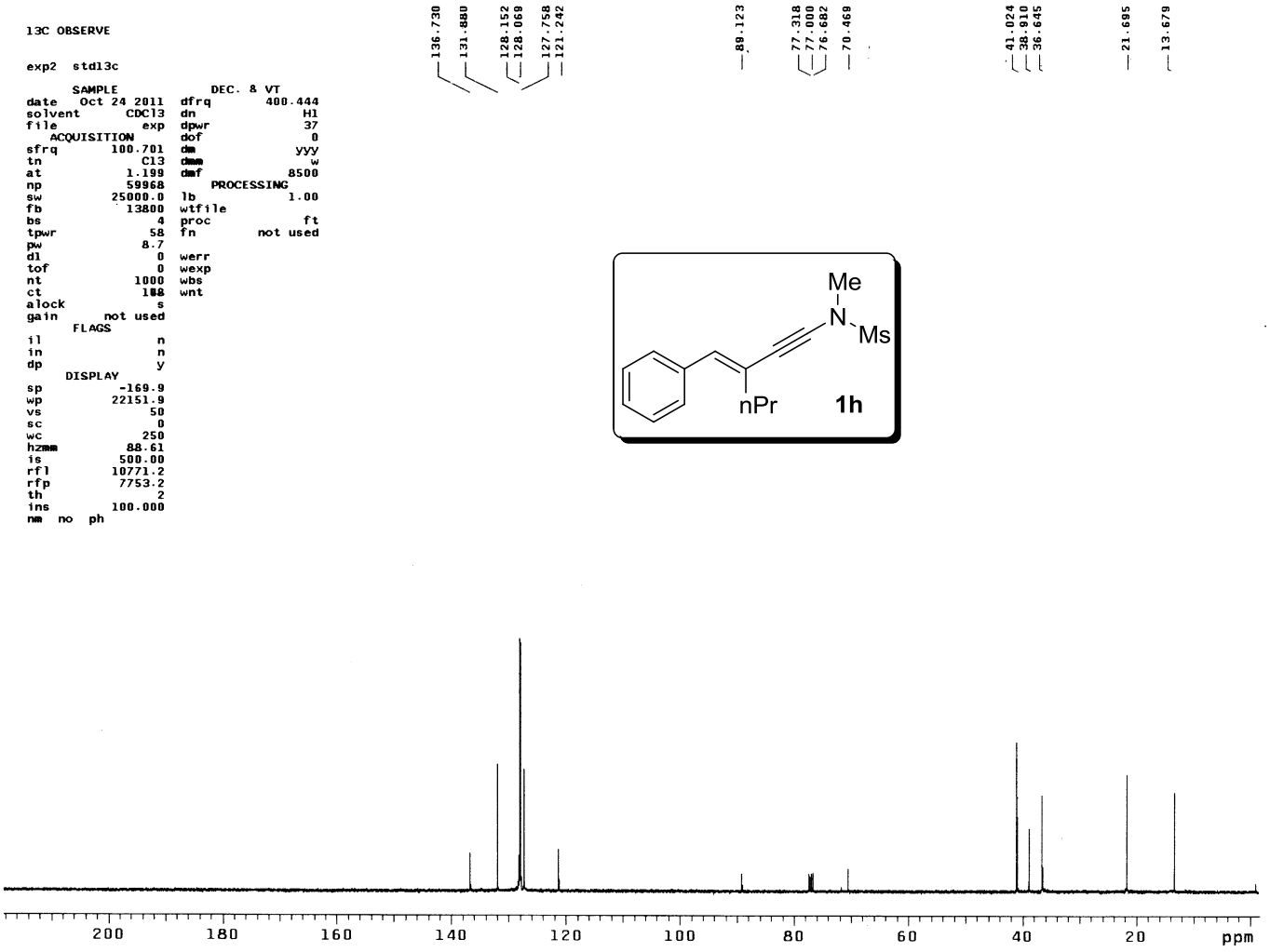

828-1H
exp4 std13c
SAMPLE
date Nov 2 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 1000
ct 552
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -164.6
wp 22151.9
vs 50
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10765.8
rfp 7753.2
th 4
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt

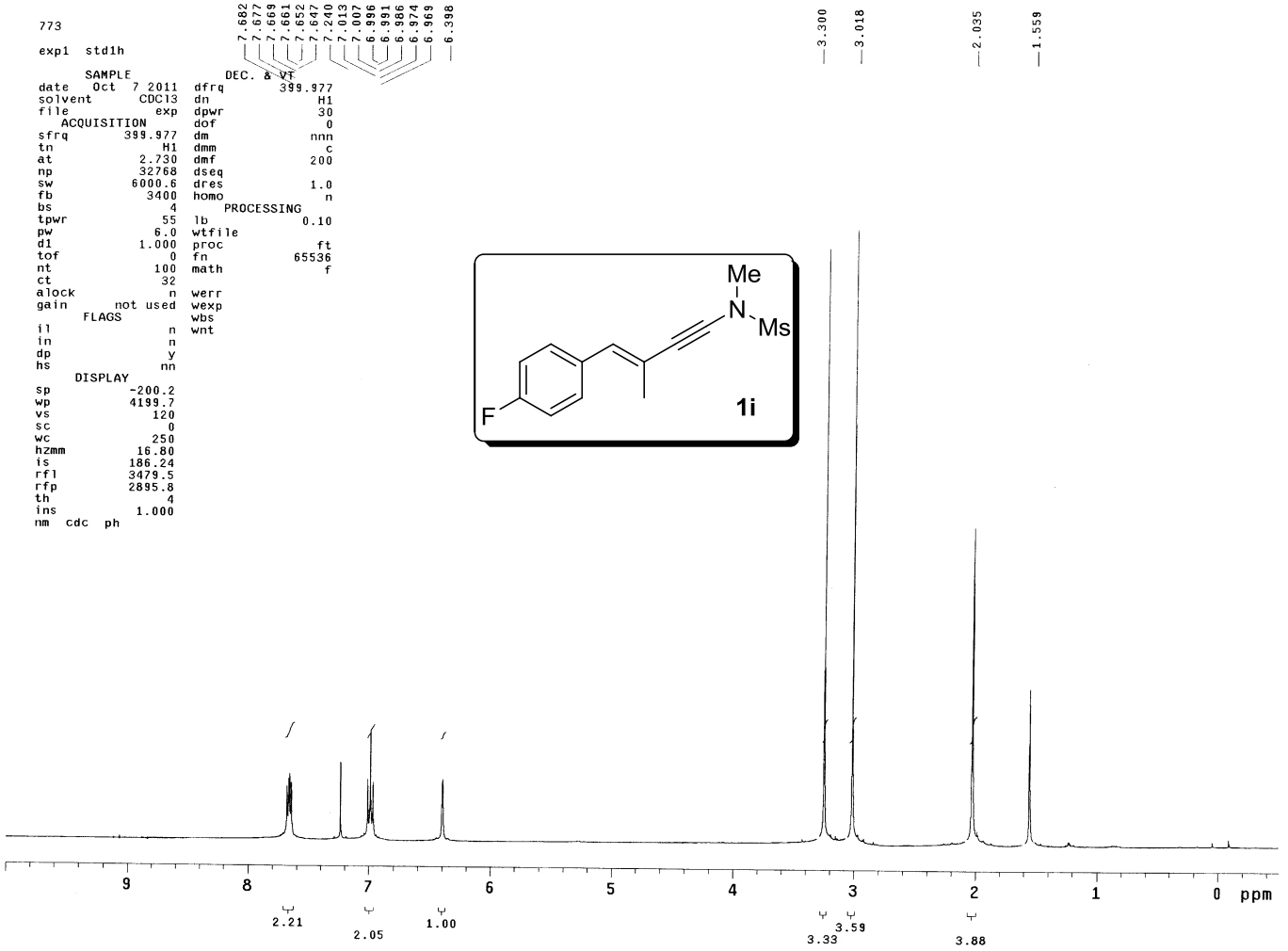

136.730
134.744
128.774
128.160
126.940
118.992
82.084
77.311
76.992
76.674
72.674
55.428
47.919
23.673
21.051
19.354

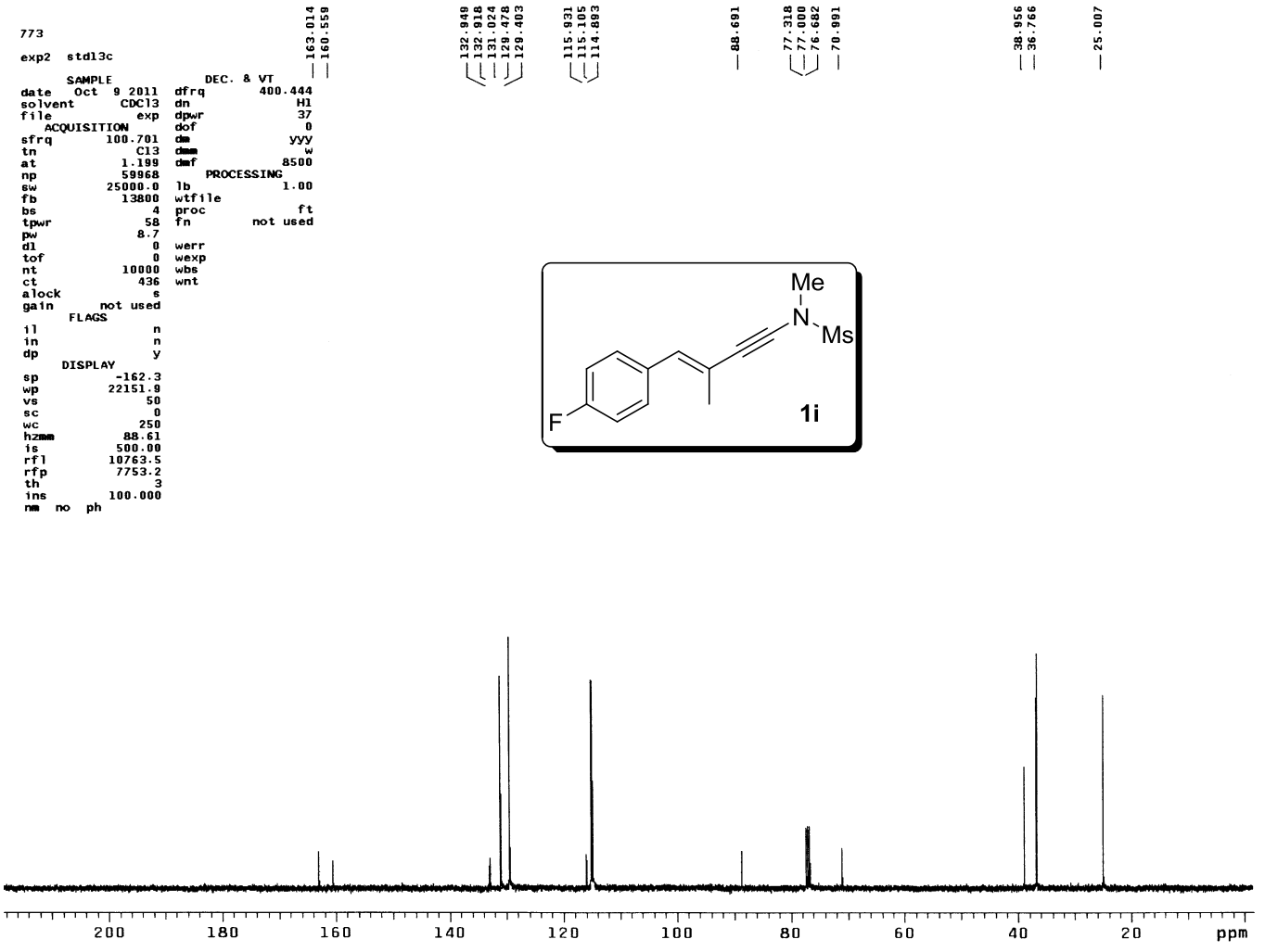

N
O
S
O
O
1g

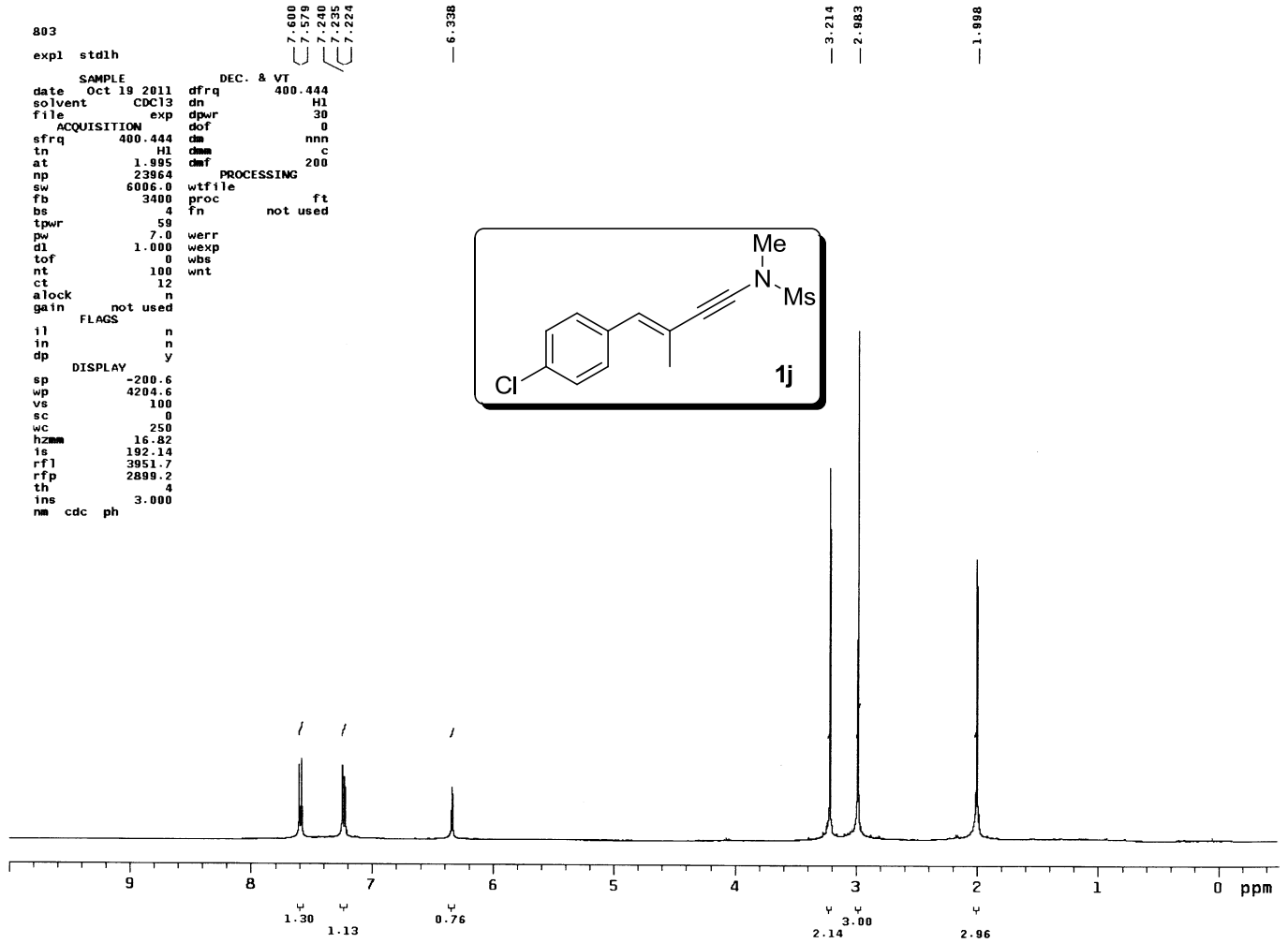

200
180
160
140
120
100
80
60
40
20
ppm


814
exp3 stdlh
SAMPLE
date Oct 24 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
dl 1.000
tof 0
nt 100
ct 24
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 80
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3930.1
rfp 2899.2
th 4
ins 3.000
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.724
7.704
7.343
7.326
7.310
7.307
7.256
7.240
7.222
6.470
3.273
3.097
2.318
2.299
2.280
1.657
1.638
1.622
1.602
1.574
0.973
0.955
0.936
Me
N
Ms
nPr
1h
9
8
7
6
5
4
3
2
1
0 ppm
1.56
0.98
1.99
0.87
3.00
2.81
2.19
2.49
3.64




773
exp1 std1h
SAMPLE
date Oct 7 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 399.977
tn H1
at 2.730
np 32768
sw 6000.6
fb 3400
bs 4
tpwr 55
pw 6.0
d1 1.000
tof 0
nt 100
ct 32
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp -200.2
wp 4199.7
vs 120
sc 0
wc 250
hzmm 16.80
is 186.24
rfl 3479.5
rfp 2895.8
th 4
ins 1.000
nm cdc ph
DEC. & VT
dfrq 399.977
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
lb 0.10
wtfile
proc ft
fn 65536
math f
werr
wexp
wbs
wnt
7.682
7.677
7.669
7.661
7.652
7.647
7.240
7.013
7.007
6.996
6.991
6.986
6.974
6.969
6.398
3.300
3.018
2.035
1.559
Me
N
Ms
F
1i
9
8
7
6
5
4
3
2
1
0 ppm
2.21
2.05
1.00
3.33
3.59
3.88



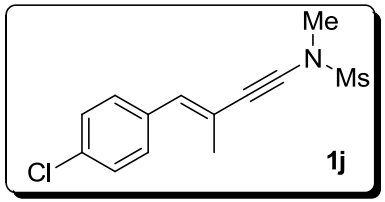


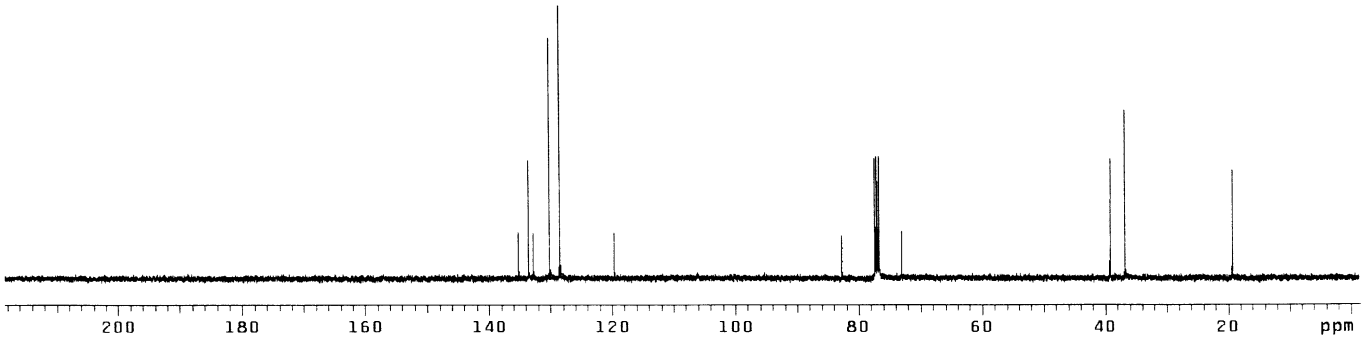
879

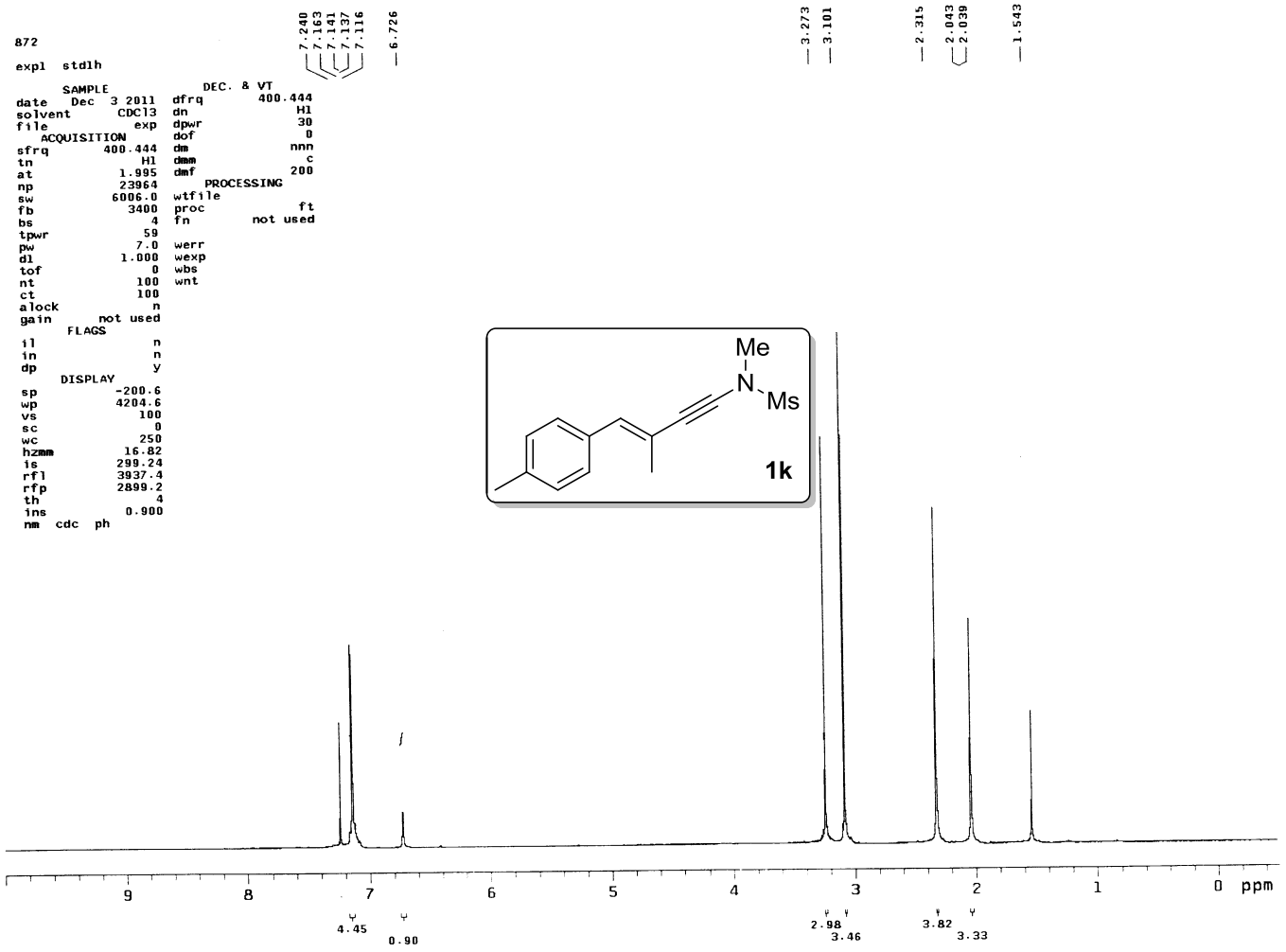
exp3 std13c

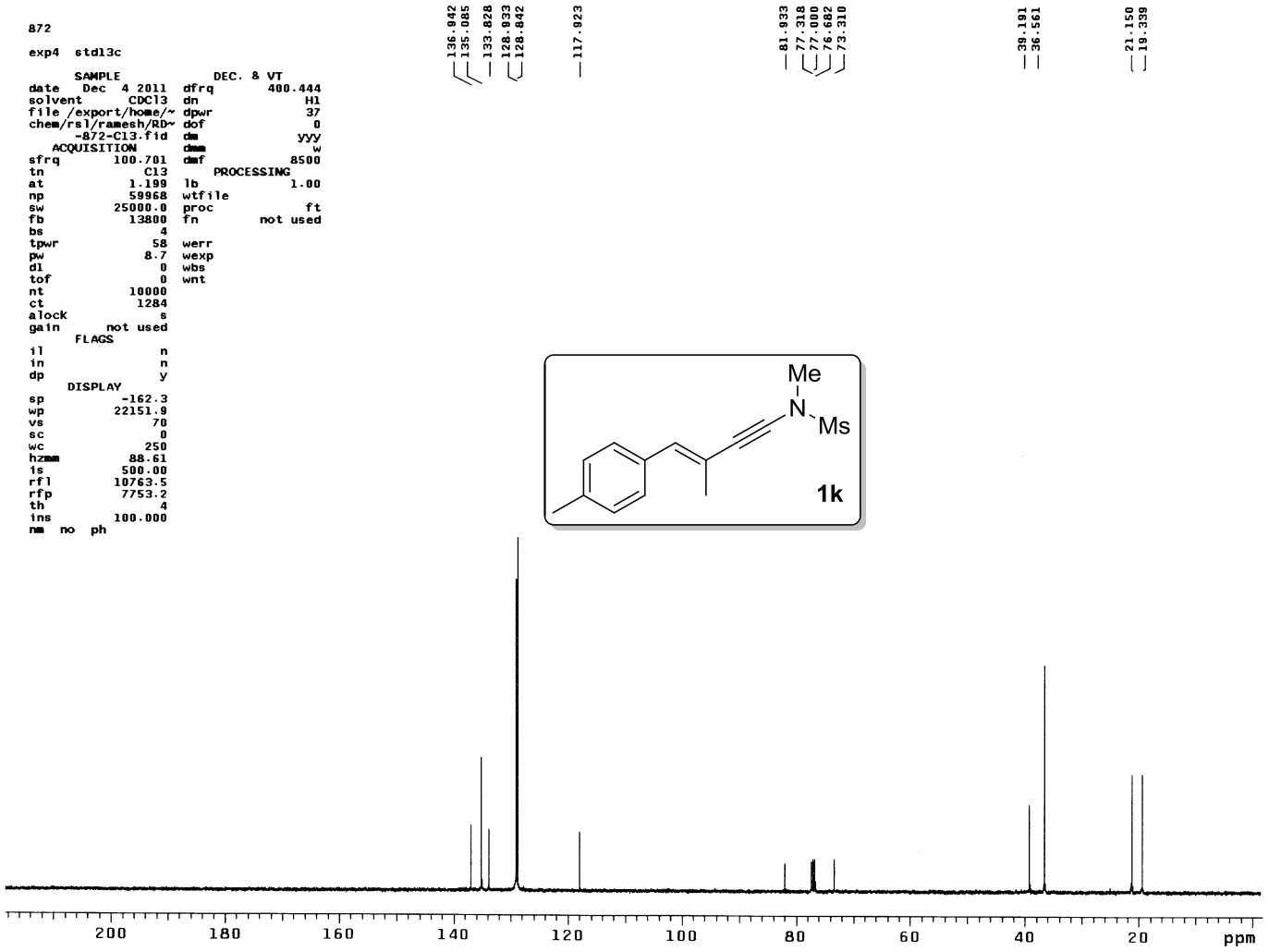
SAMPLE		DEC. & VT	
date	Dec 7 2011	dfrq	400.444
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	100.701	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	8500
np	59968	PROCESSING	
sw	25000.0	lb	1.00
fb	13800	wtfile	
bs	4	proc	ft
tpwr	58	fn	not used
pw	8.7		
dl	0	werr	
tof	0	wexp	
nt	10000	wbs	
ct	2428	wnt	
alock	s		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-156.9		
wp	22151.9		
vs	50		
sc	0		
wc	250		
hzmm	88.61		
is	500.00		
rfl	10758.2		
rfp	7753.2		
th	3		
ins	100.000		
nm no ph			

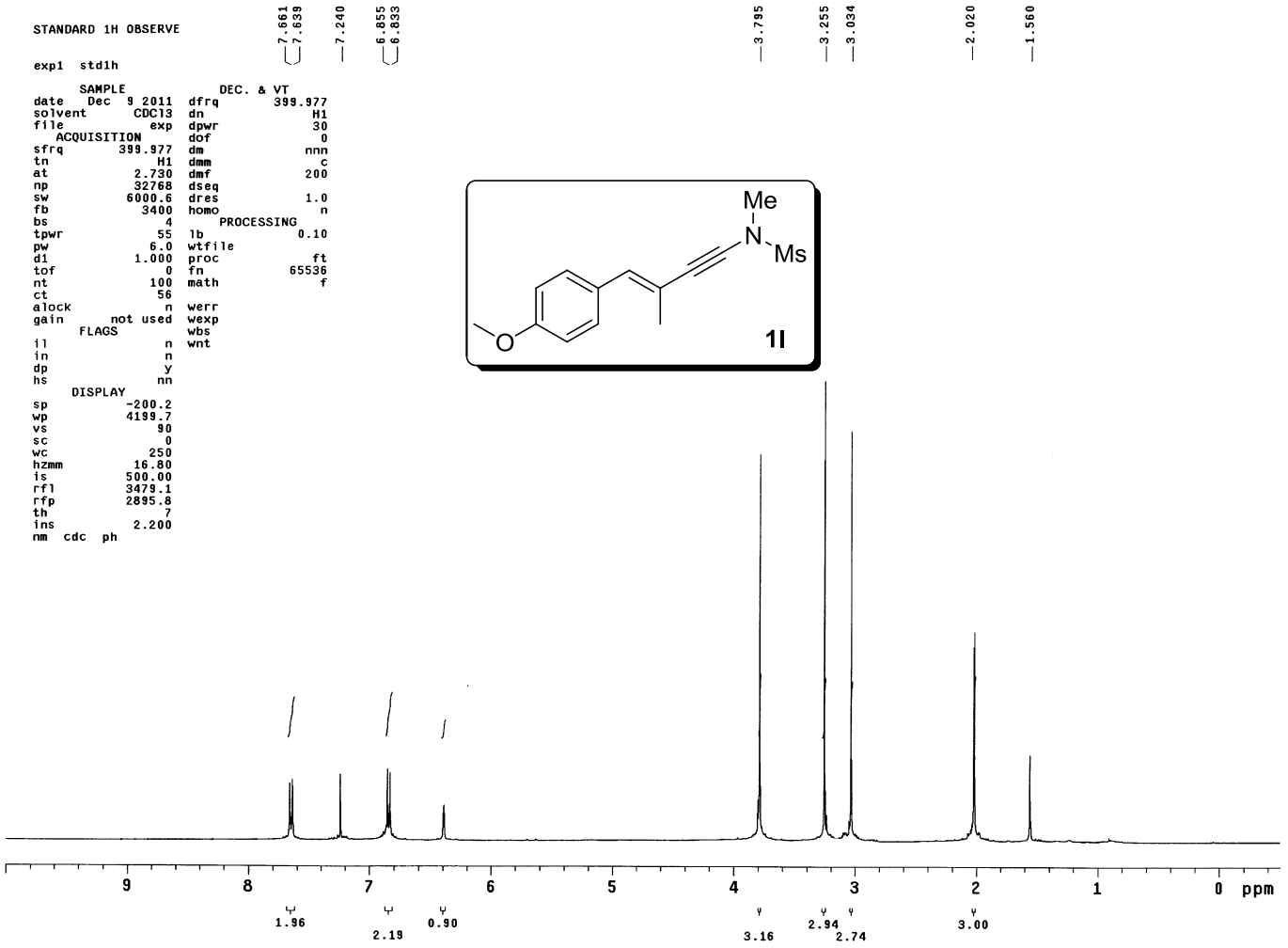
135.138
133.532
132.835
130.085
128.463
119.590
82.728
77.318
77.000
76.689
73.083
39.198
36.743
19.324

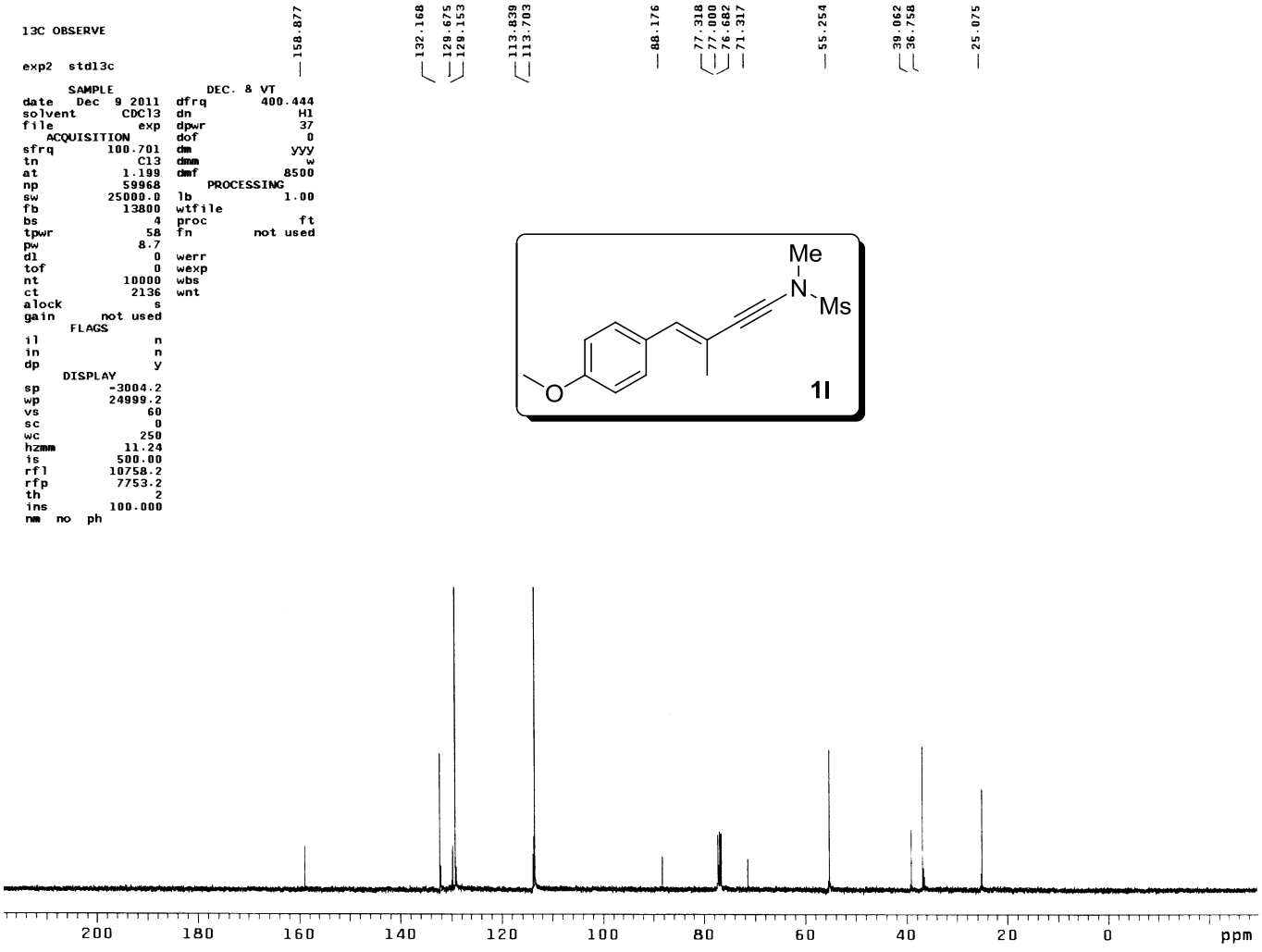


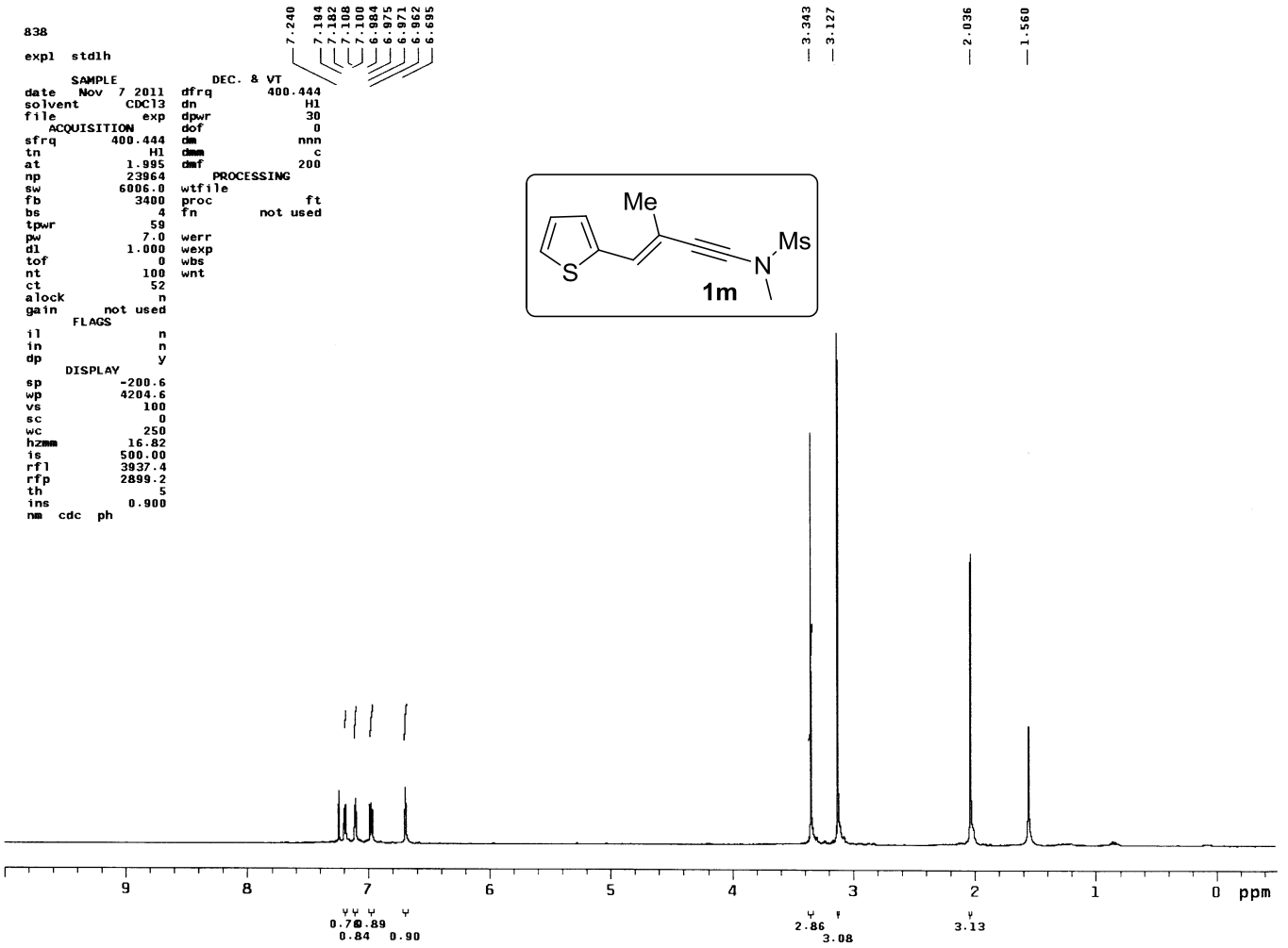


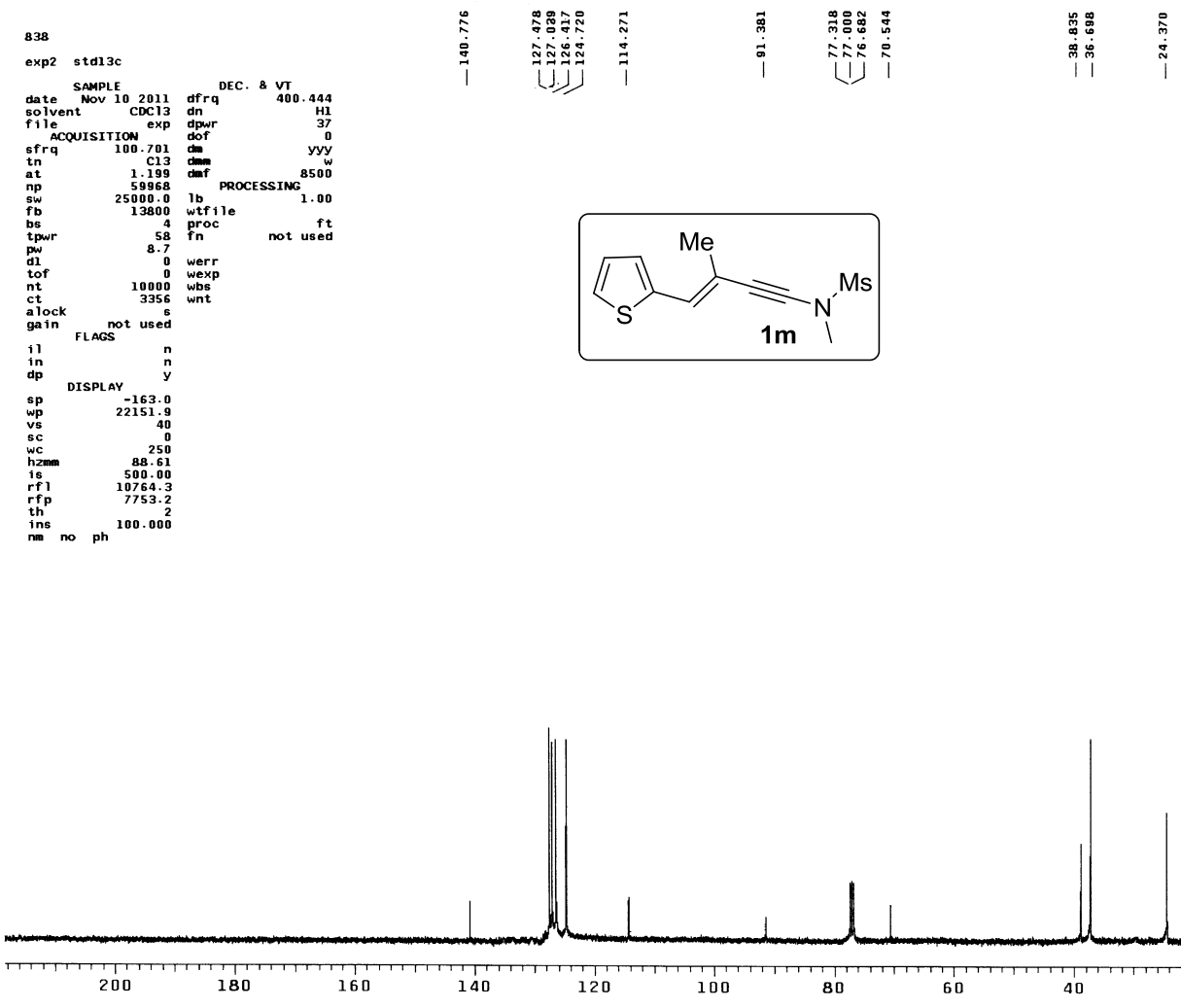

872
expl stdlh
SAMPLE
date Dec 3 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
dl 1.000
tof 0
nt 100
ct 100
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 100
sc 0
wc 250
hzmm 16.82
is 299.24
rfl 3937.4
rfp 2899.2
th 4
ins 0.900
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.240
7.163
7.141
7.137
7.116
6.726
3.273
3.101
2.315
2.043
2.039
1.543
Me
N
Ms
1k
9
8
7
6
5
4
3
2
1
0 ppm
4.45
0.90
2.98
3.46
3.82
3.33

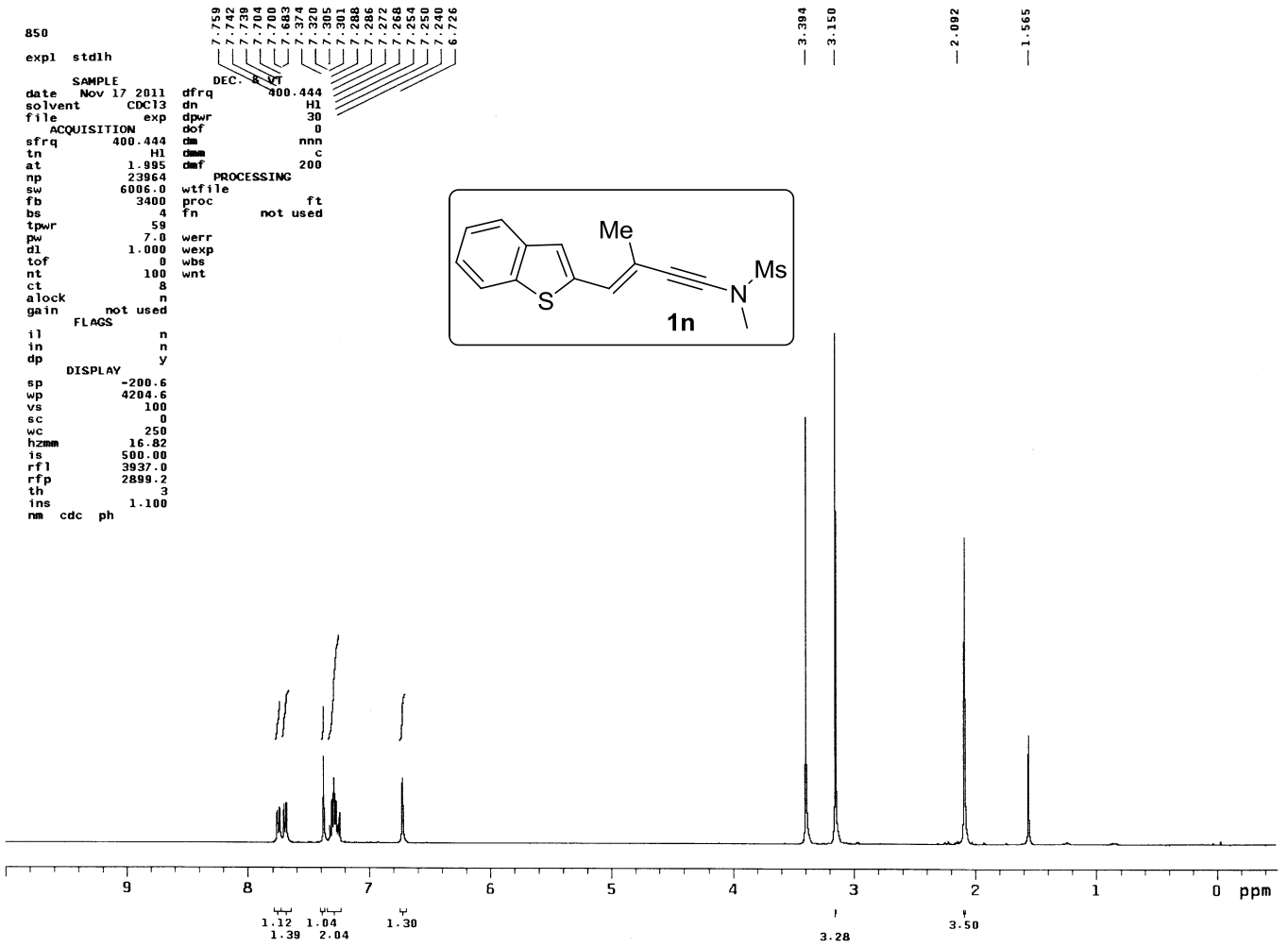

872
exp4 stdl3c
SAMPLE
date Dec 4 2011
solvent CDCl3
file /export/home/~
chem/rsl/ramesh/RD~
-872-C13.fid
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 10000
ct 1284
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -162.3
wp 22151.9
vs 70
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10763.5
rfp 7753.2
th 4
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
136.942
135.085
133.828
128.933
128.842
117.923
81.933
77.318
77.000
76.682
73.310
39.191
36.561
21.150
19.339
Me
N
Ms
1k
200
180
160
140
120
100
80
60
40
20
ppm




158.877
132.168
129.675
129.153
113.839
113.703
88.176
77.318
77.000
76.682
71.317
55.254
39.062
36.758
25.075
13C OBSERVE
exp2 std13c
SAMPLE
date Dec 9 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 10000
ct 2136
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -3004.2
wp 24999.2
vs 60
sc 0
wc 250
hzmm 11.24
is 500.00
rfl 10758.2
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Me
N
Ms
O
1l
200
180
160
140
120
100
80
60
40
20
0
ppm






850
expl stdlh
SAMPLE
date Nov 17 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
d1 1.000
tof 0
nt 100
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 100
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3937.0
rfp 2899.2
th 3
ins 1.100
nm cdc ph
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
DEC. & FT
7.759
7.742
7.739
7.704
7.700
7.683
7.374
7.320
7.305
7.301
7.288
7.286
7.272
7.268
7.254
7.250
7.240
6.726
3.394
3.150
2.092
1.565
Me
Ms
N
S
1n
9
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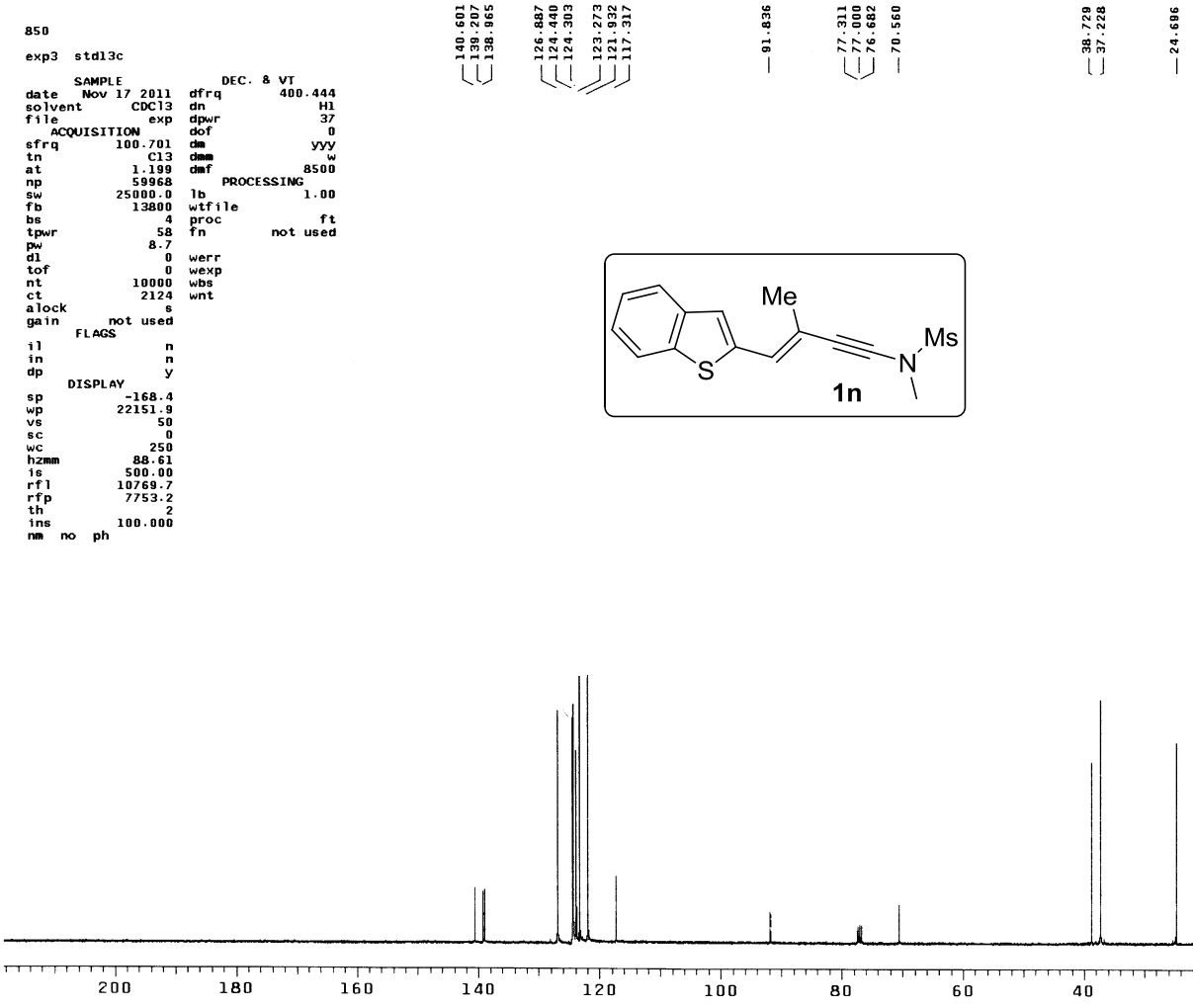
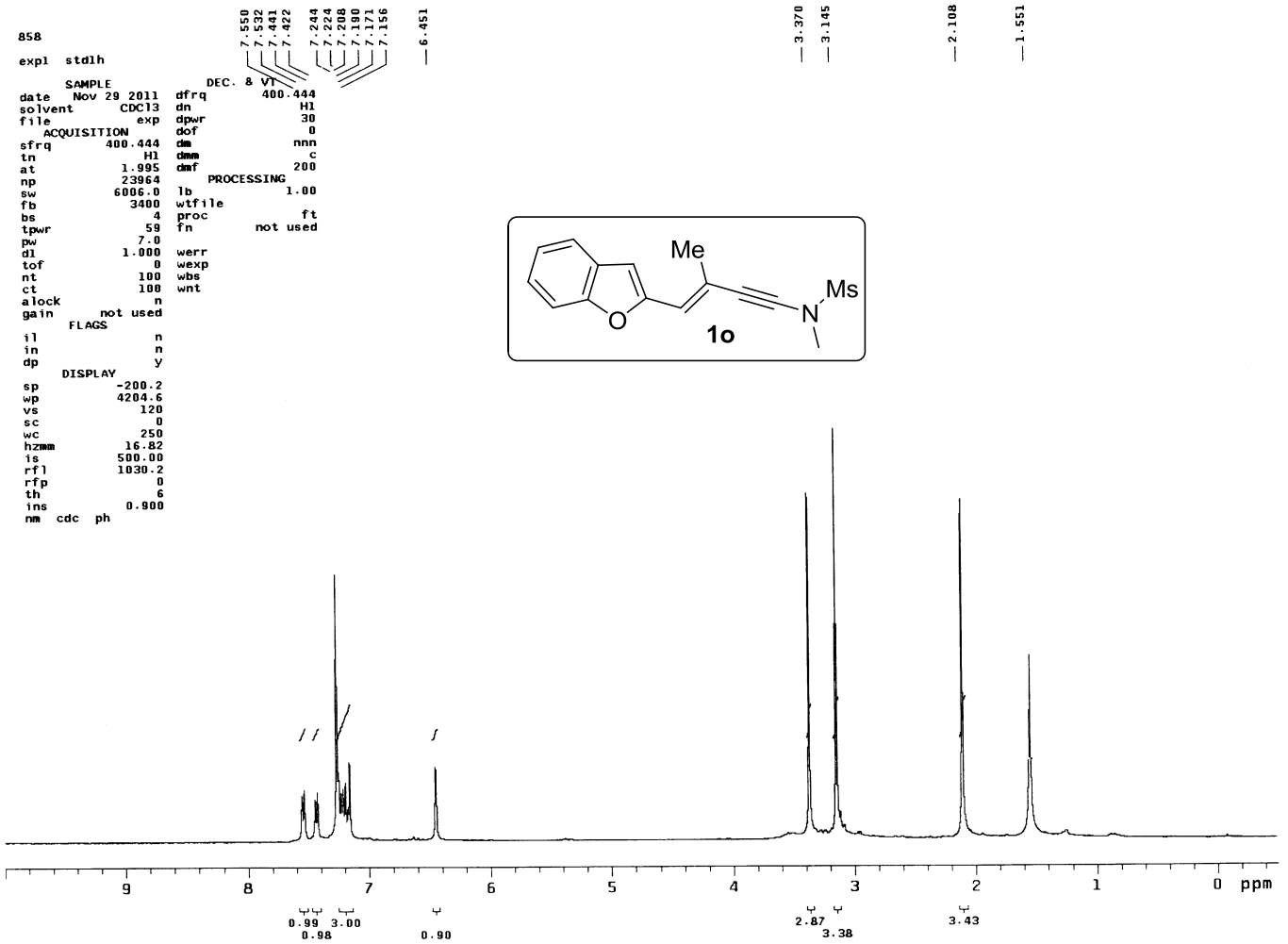
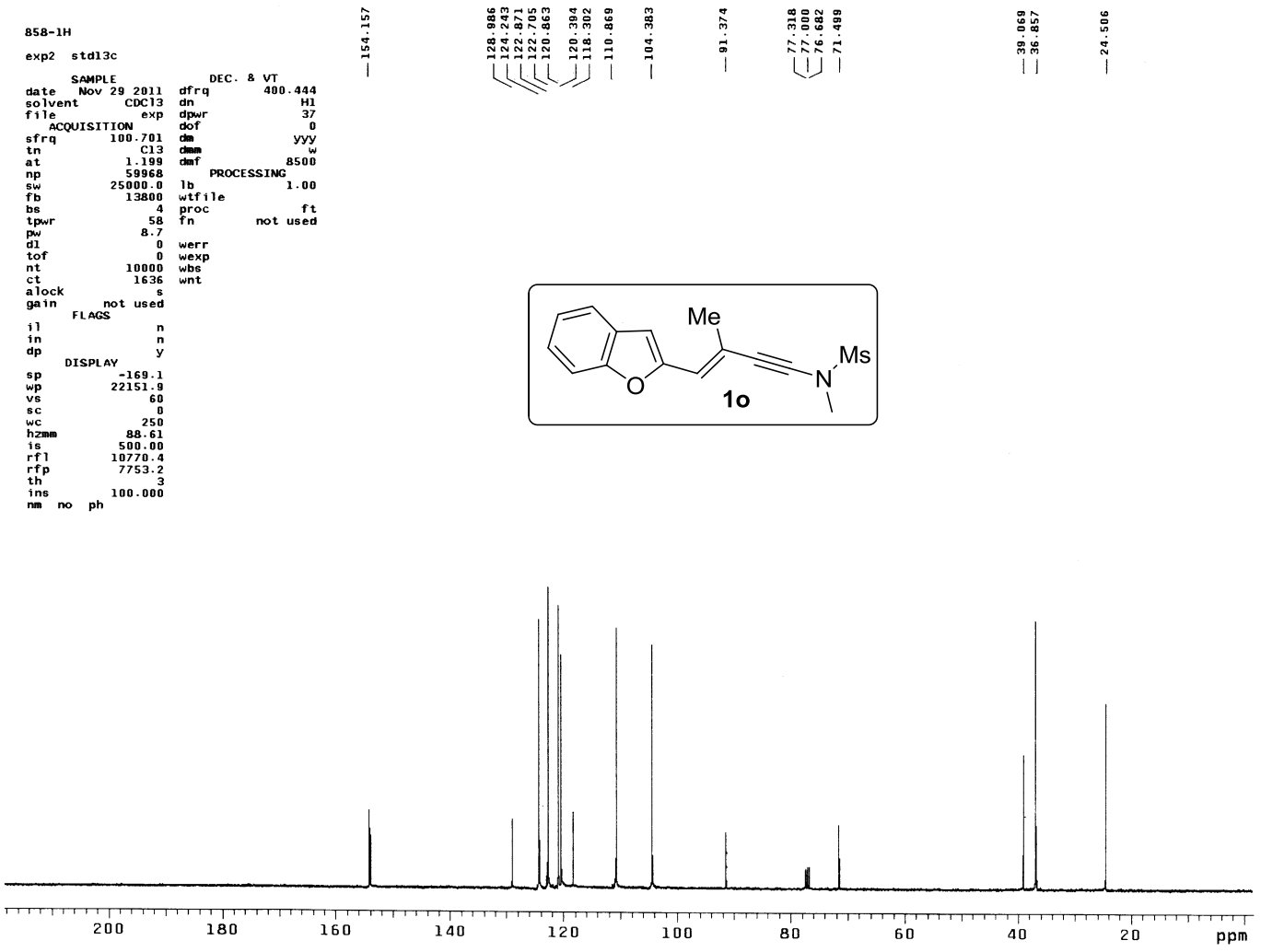
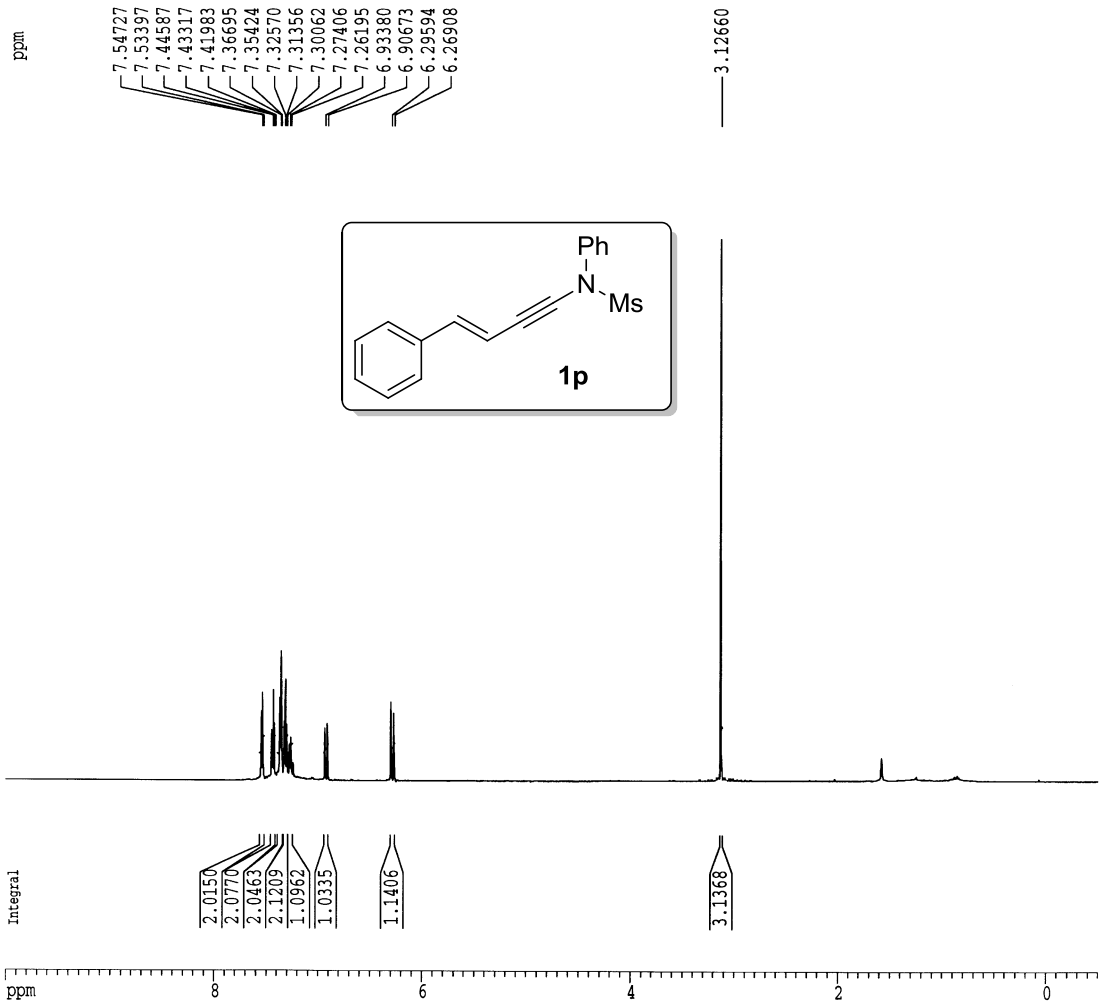
1.39
1.04
2.04
1.30
3.28
3.50



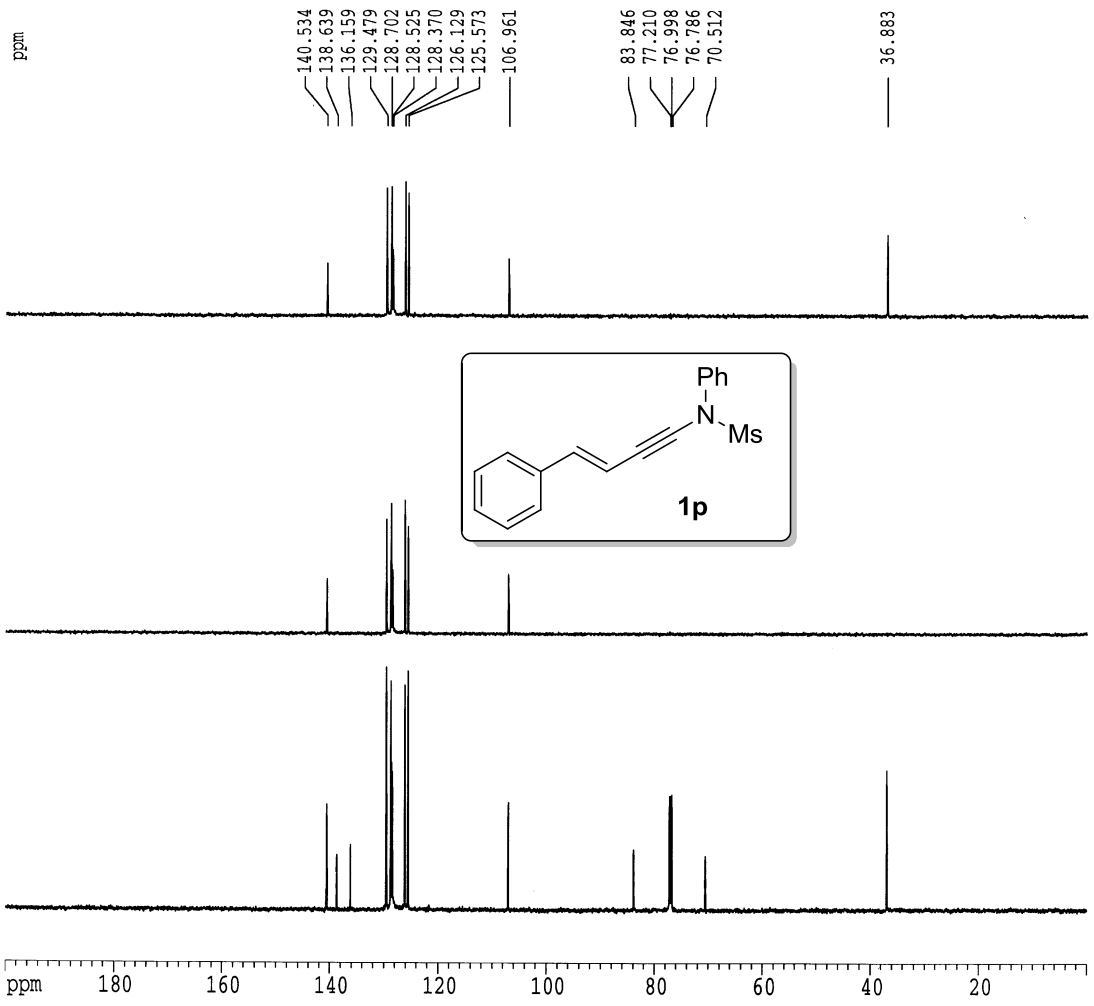

858
expl stdlh
SAMPLE
date Nov 29 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
dl 1.000
tof 0
nt 100
ct 100
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.2
wp 4204.6
vs 120
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 1030.2
rfp 0
th 6
ins 0.900
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.550
7.532
7.441
7.422
7.244
7.224
7.208
7.190
7.171
7.156
6.451
3.370
3.145
2.108
1.551
Me
Ms
N
O
1o
9
8
7
6
5
4
3
2
1
0 ppm
0.99
3.00
0.98
0.90
2.87
3.38
3.43

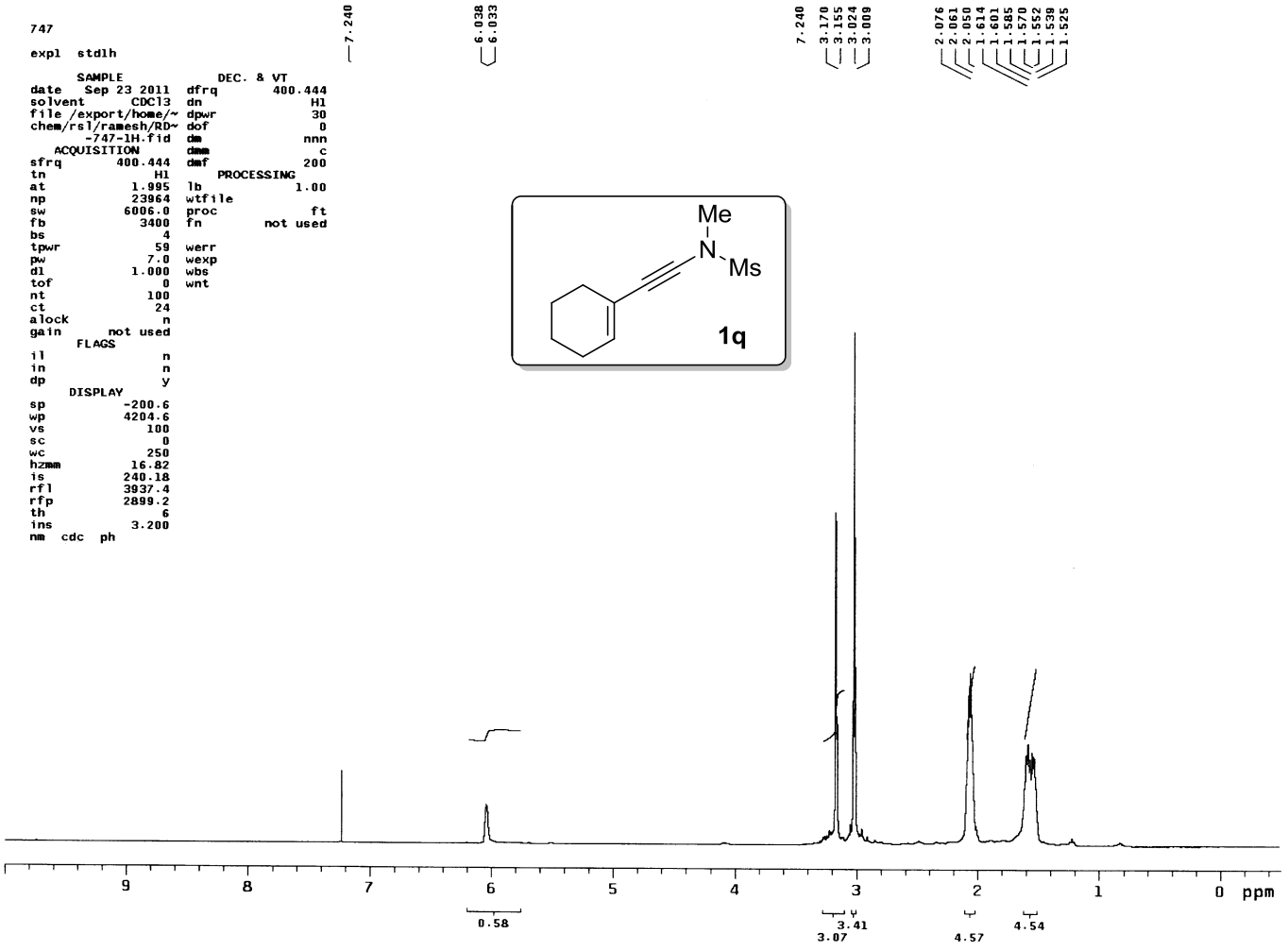

858-1H
exp2 stdl3c
SAMPLE
date Nov 29 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 10000
ct 1636
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -169.1
wp 22151.9
vs 60
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10770.4
rfp 7753.2
th 3
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
154.157
128.986
124.243
122.871
122.705
120.863
120.394
118.302
110.869
104.383
91.374
77.318
77.000
76.682
71.499
39.069
36.857
24.506
Me
Ms
N
O
1o
200
180
160
140
120
100
80
60
40
20
ppm

```
Current Data Parameters
NAME                 RD-935
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20120321
Time                  15.15
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG                 zg
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8389.262 Hz
FIDRES             0.256020 Hz
AQ                 1.9530228 sec
RG                      512
DW                   59.600 usec
DE                    6.50 usec
TE                   296.3 K
D1                 1.00000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    8.00 usec
PL1                   0.00 dB
SFO1           598.9029945 MHz

F2 - Processing parameters
SI                       16384
SF              598.9000256 MHz
WDW                         no
SSB                          0
LB                    0.00 Hz
GB                           0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   10.00 cm
F1P                 10.000 ppm
F1                 5989.00 Hz
F2P                  -0.500 ppm
F2                 -299.45 Hz
PPMCM               0.52500 ppm/cm
HZCM              314.42252 Hz/cm
```



```
Current Data Parameters
NAME                  RD-935
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20120321
Time                  15.26
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                      148
DS                        0
SWH               45045.047 Hz
FIDRES             1.374666 Hz
AQ                 0.3637748 sec
RG                     4096
DW                   11.100 usec
DE                    6.50 usec
TE                   296.7 K
D1                3.50000000 sec
d11               0.03000000 sec
DELTA             3.40000010 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     4.80 usec
PL1                    0.00 dB
SFO1            150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                92.00 usec
PL2                 120.00 dB
PL12                 11.30 dB
PL13                 14.00 dB
SFO2            598.9029990 MHz

F2 - Processing parameters
SI                     65536
SF              150.5935348 MHz
WDW                       EM
SSB                        0
LB                      3.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                      4.50 cm
F1P                  200.000 ppm
F1                 30118.71 Hz
F2P                    0.000 ppm
F2                     0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM              1505.93530 Hz/cm
```




747
expl stdlh
SAMPLE
date Sep 23 2011
solvent CDCl3
file /export/home/~
chem/rsl/ramesh/RD~
-747-1H.fid
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
dl 1.000
tof 0
nt 100
ct 24
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 100
sc 0
wc 250
hzmm 16.82
is 240.18
rfl 3937.4
rfp 2899.2
th 6
ins 3.200
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.240
6.038
6.033
7.240
3.170
3.155
3.024
3.009
2.076
2.061
2.050
1.614
1.601
1.585
1.570
1.552
1.539
1.525
Me
N
Ms
1q
9
8
7
6
5
4
3
2
1
0 ppm
0.58
3.07
3.41
4.57
4.54

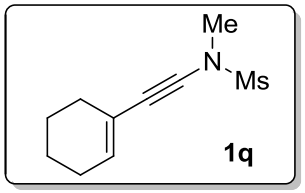
13C OBSERVE

exp5 std13c

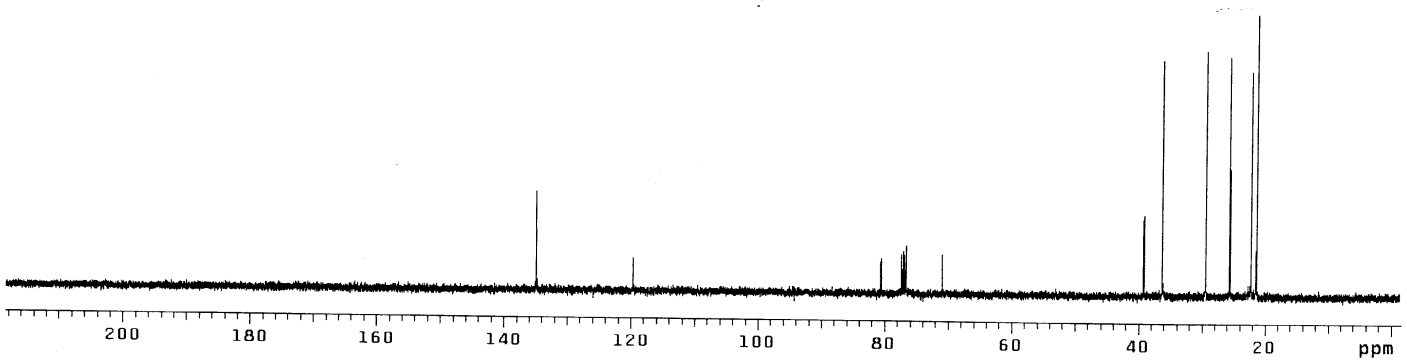
SAMPLE		DEC. & VT	
date	Sep 23 2011	dfrq	400.444
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	100.701	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	8500
np	59968	PROCESSING	
sw	25000.0	lb	1.00
fb	13800	wtfile	
bs	4	proc	ft
tpwr	58	fn	not used
pw	8.7		
dl	0	werr	
tof	0	wexp	
nt	1000	wbs	
ct	768	wnt	
alock	s		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-157.7		
wp	22151.9		
vs	50		
sc	0		
wc	250		
hzmm	88.61		
is	500.00		
rfl	10759.0		
rfp	7753.2		
th	3		
ins	100.000		

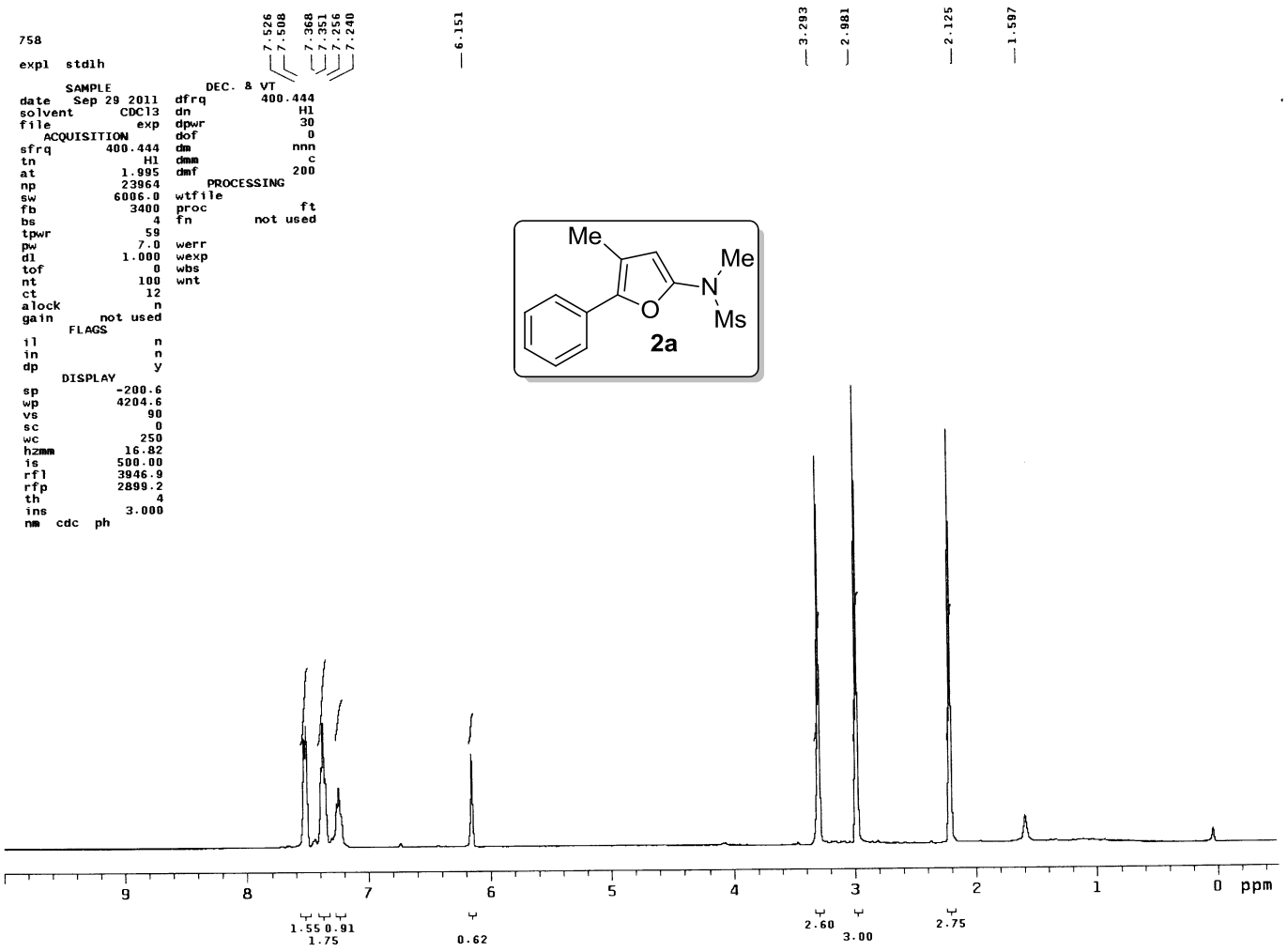
nm no ph

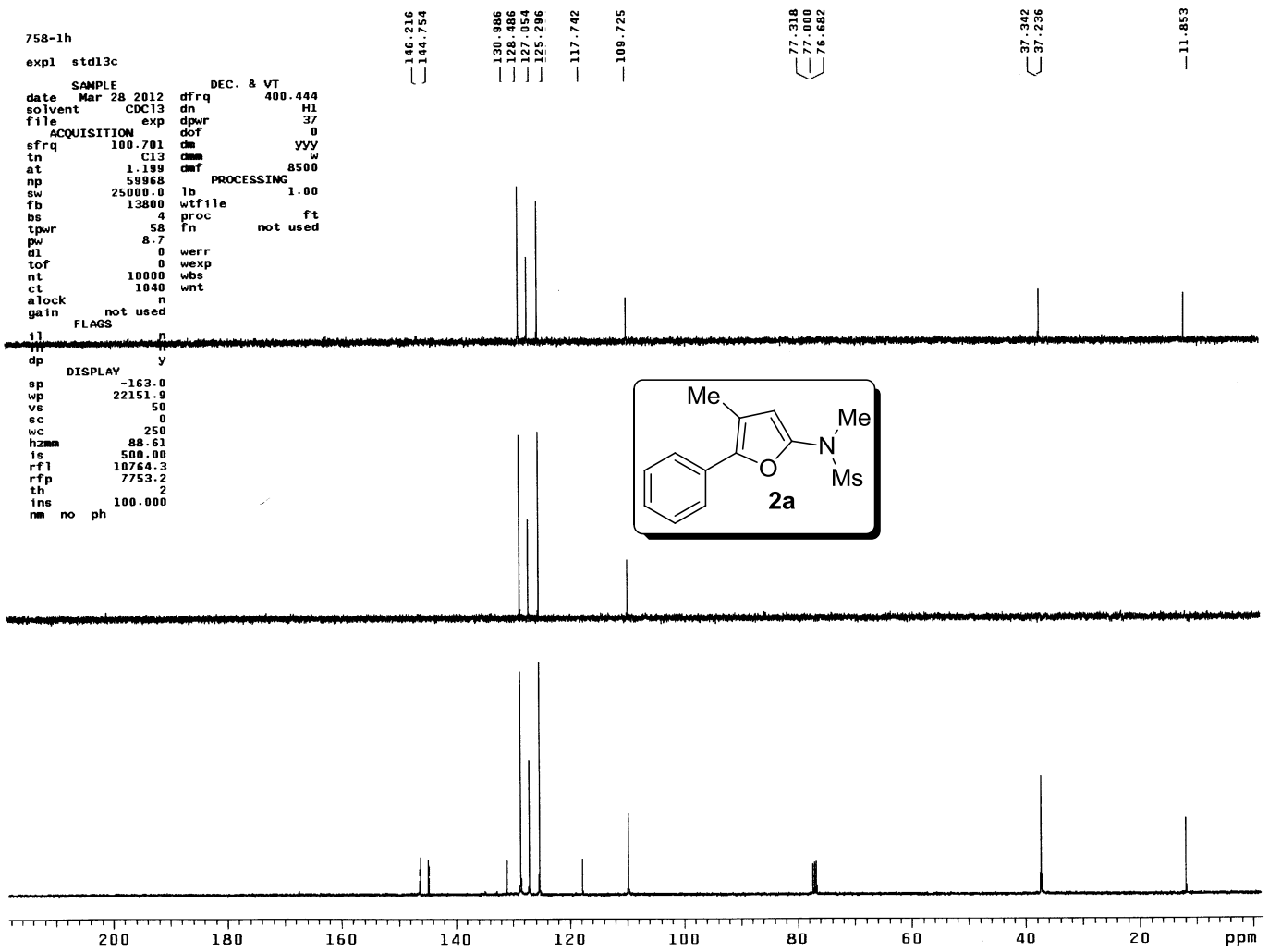
134.843
119.590
80.561
77.311
76.992
76.682
70.931
39.198
36.274
29.447
25.620
22.264
21.407



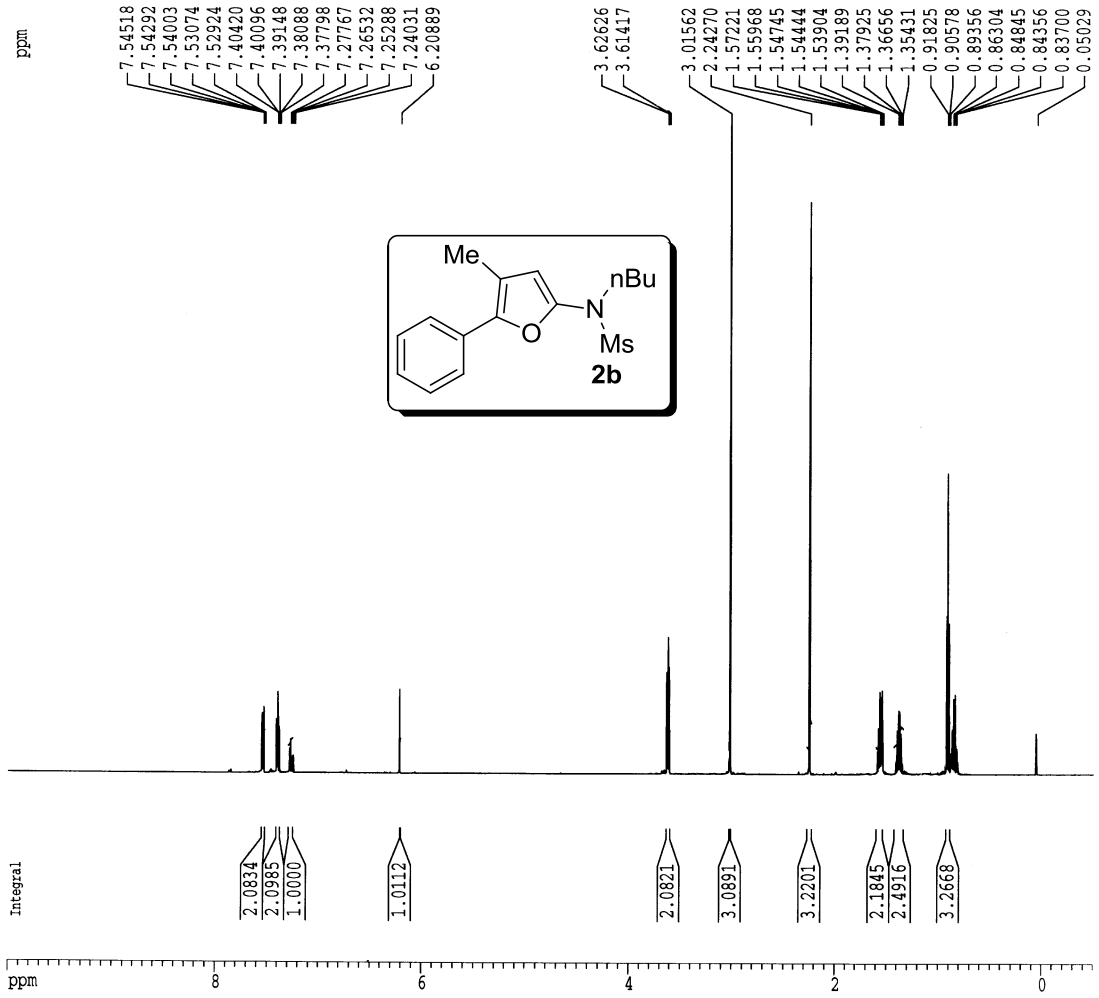
1q

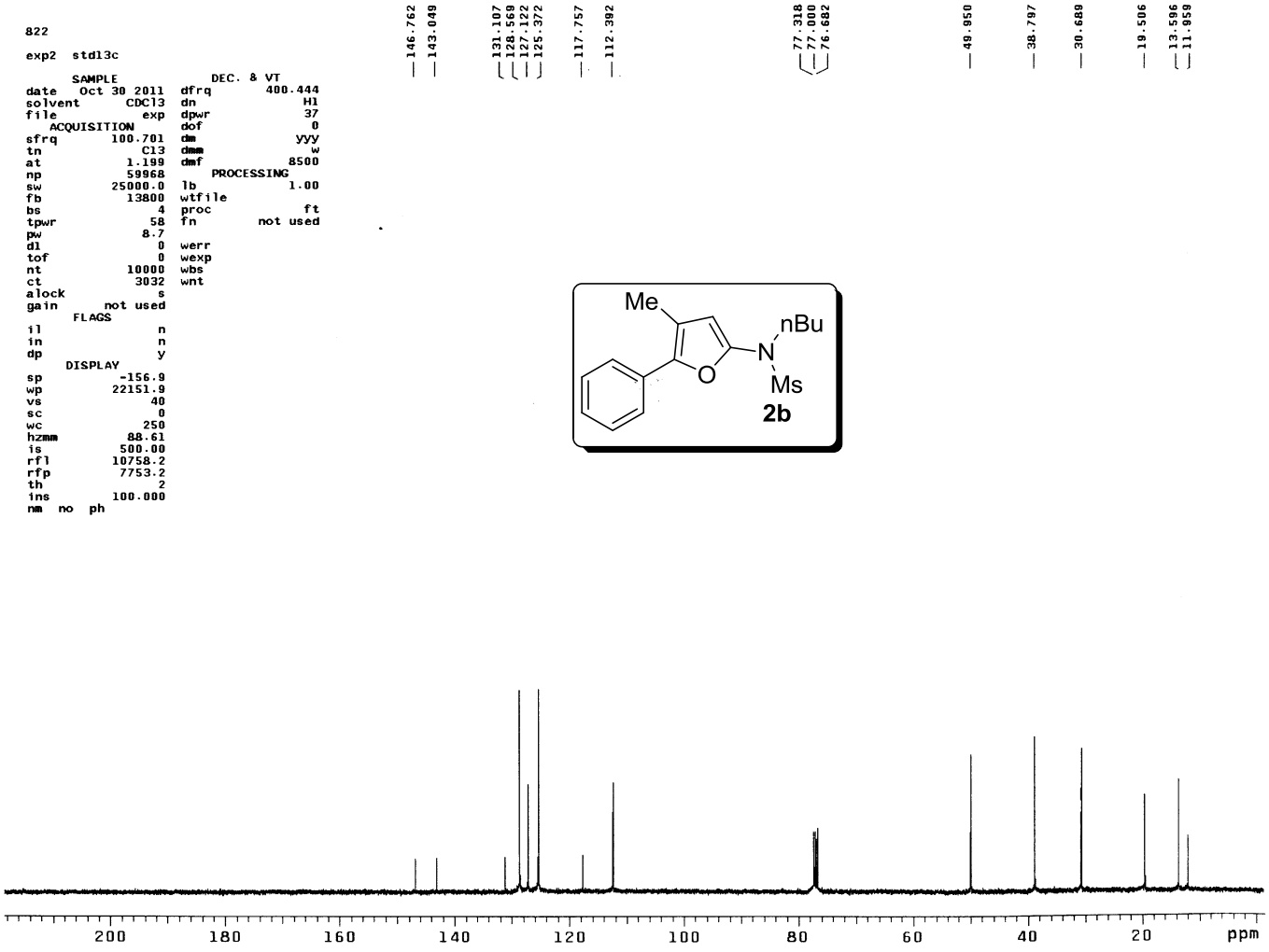


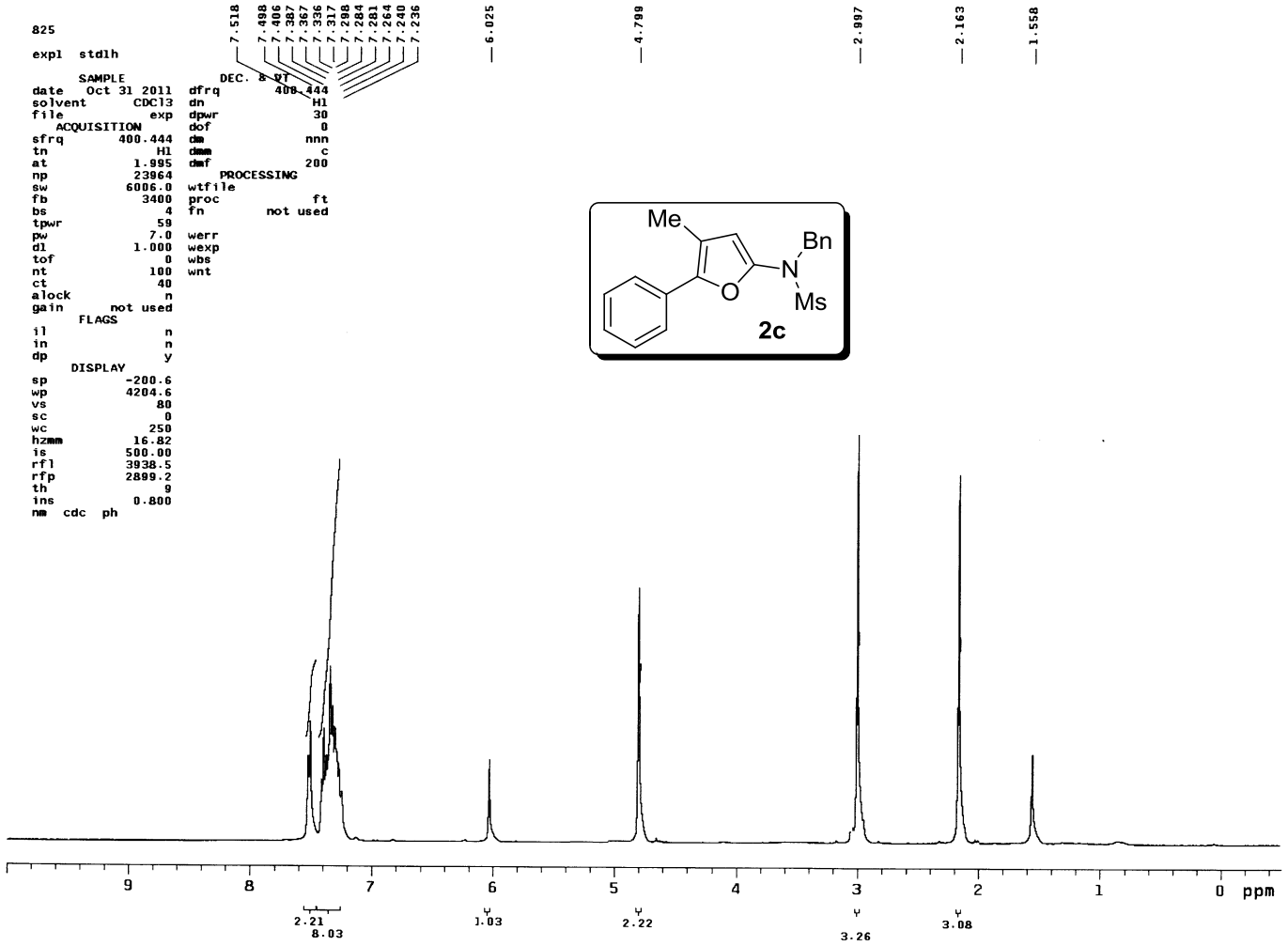


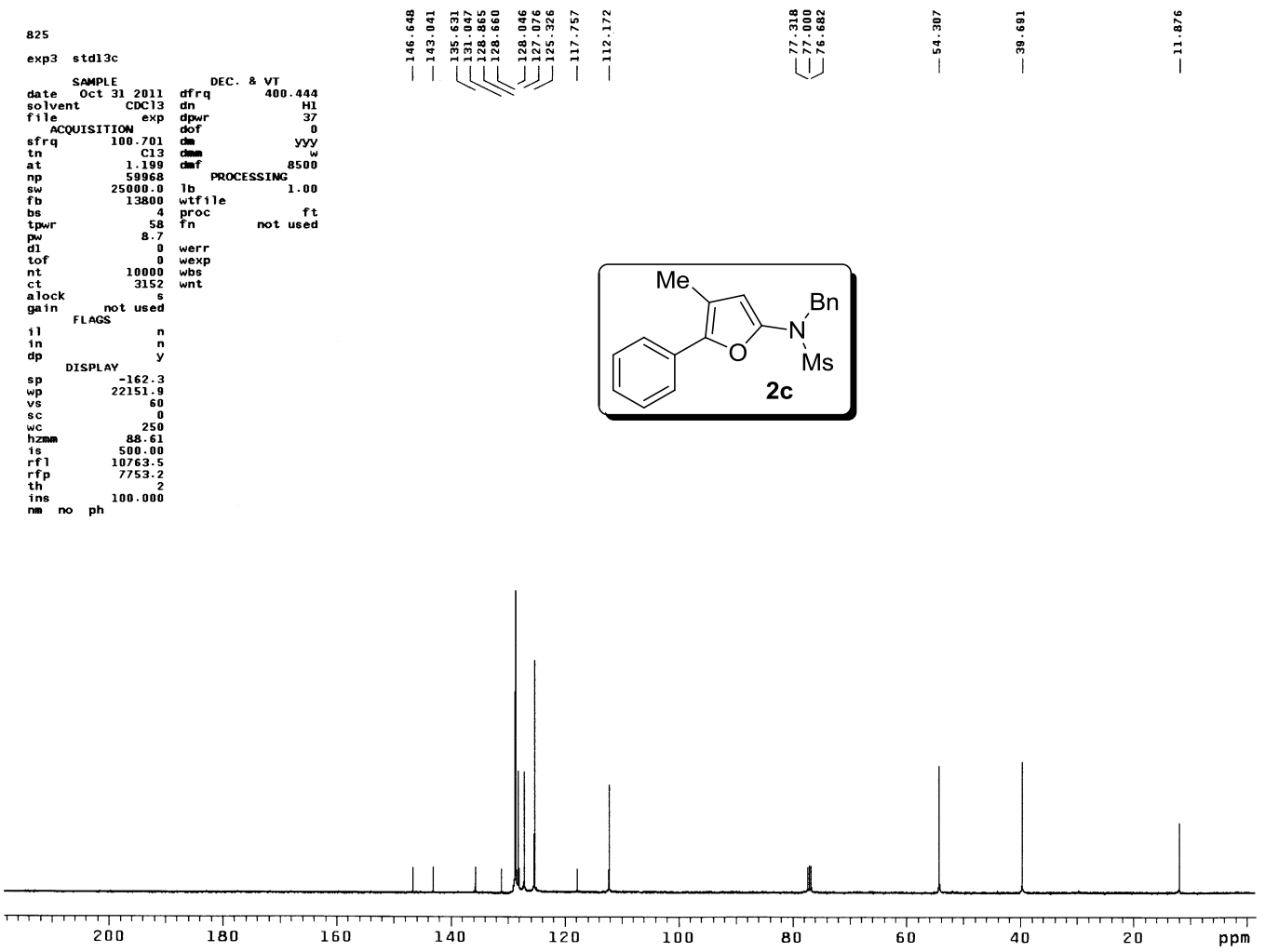

758-1h
expl std13c
SAMPLE
date Mar 28 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 10000
ct 1040
alock n
gain not used
FLAGS
dp y
DISPLAY
sp -163.0
wp 22151.9
vs 50
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10764.3
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
146.216
144.754
130.986
128.486
127.054
125.296
117.742
109.725
77.318
77.000
76.682
37.342
37.236
11.853
Me
Me
N
O
Ms
2a
200
180
160
140
120
100
80
60
40
20
ppm

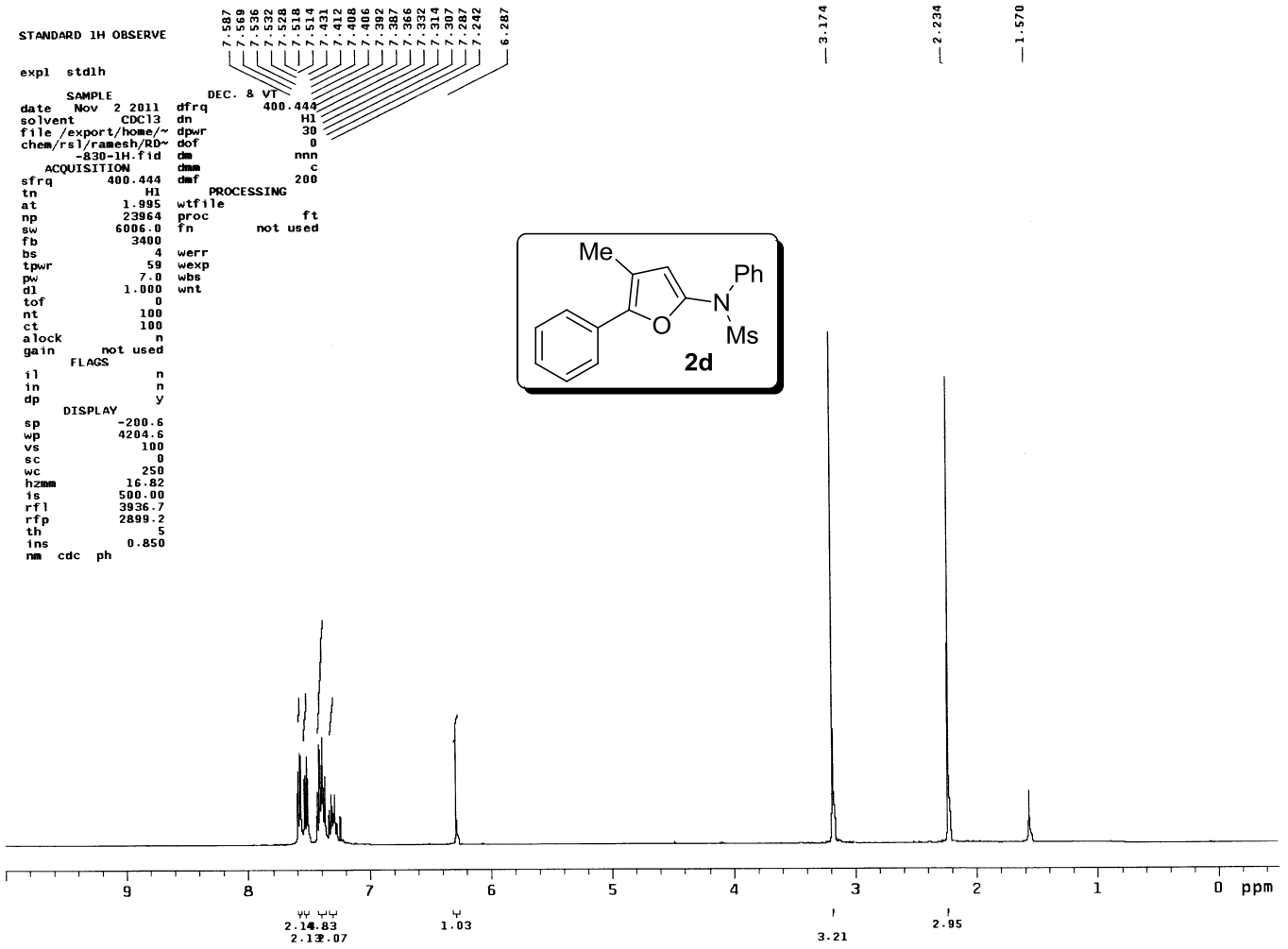
```
Current Data Parameters
NAME                  RD-822
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20111106
Time                  14.41
INSTRUM                spect
PROBHD    5 mm QNP  1H/1
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                8389.262 Hz
FIDRES             0.256020 Hz
AQ                 1.9530228 sec
RG                       256
DW                    59.600 usec
DE                     6.50 usec
TE                     0.0 K
D1                 1.50000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     10.50 usec
PL1                     0.00 dB
SFO1            598.9028131 MHz

F2 - Processing parameters
SI                        16384
SF              598.9000240 MHz
WDW                          no
SSB                           0
LB                      0.00 Hz
GB                            0
PC                      1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     15.00 cm
F1P                   10.000 ppm
F1                   5989.00 Hz
F2P                   -0.500 ppm
F2                   -299.45 Hz
PPMCM                0.52500 ppm/cm
HZCM               314.42252 Hz/cm
```

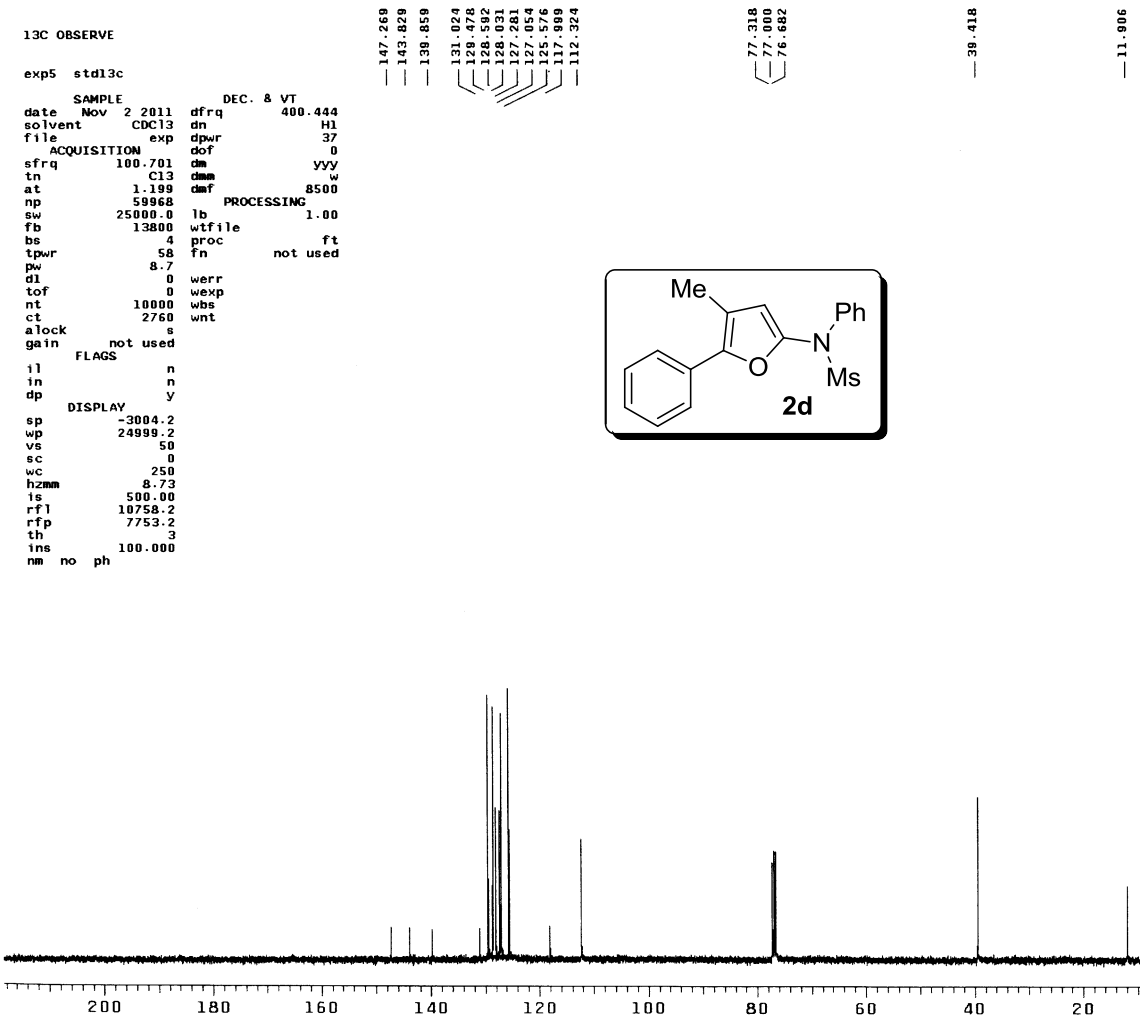


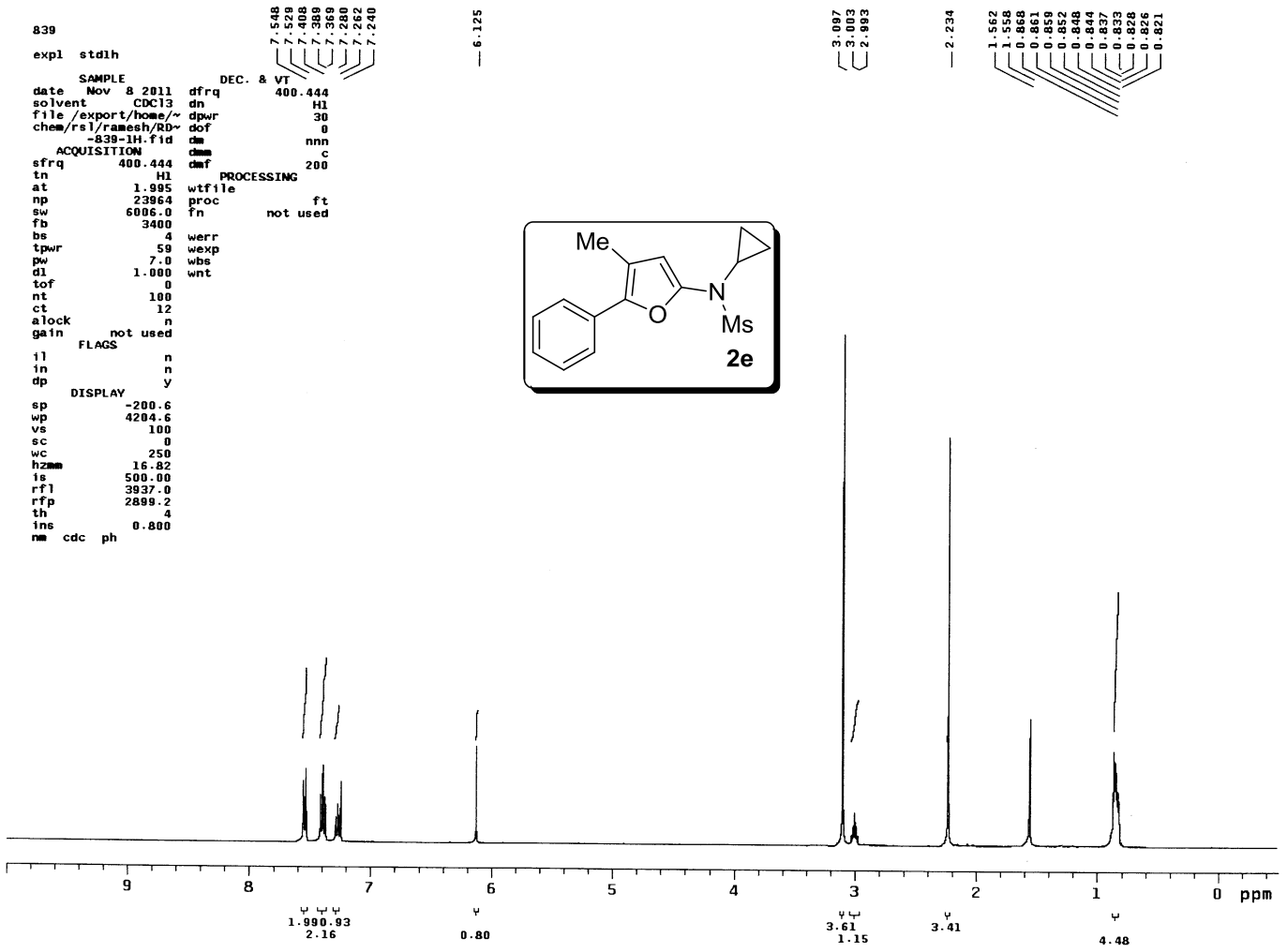

822
exp2 stdl3c
SAMPLE
date Oct 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 10000
ct 3032
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -156.9
wp 22151.9
vs 40
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10758.2
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
146.762
143.049
131.107
128.569
127.122
125.372
117.757
112.392
77.318
77.000
76.682
49.950
38.797
30.689
19.506
13.596
11.959
Me
nBu
N
O
Ms
2b
200
180
160
140
120
100
80
60
40
20
ppm


825
expl stdlh
SAMPLE
date Oct 31 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
dl 1.000
tof 0
nt 100
ct 40
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 80
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3938.5
rfp 2899.2
th 9
ins 0.800
nm cdc ph
DEC. & PT
dfrq 400.444
dn H1
dpwr 30
dof 0
nnn
c
200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.518
7.498
7.406
7.387
7.367
7.336
7.317
7.298
7.284
7.281
7.264
7.240
7.236
6.025
4.799
2.997
2.163
1.558
Me
Bn
N
O
Ms
2c
9
8
7
6
5
4
3
2
1
0 ppm
2.21
8.03
1.03
2.22
3.26
3.08


146.648
143.041
135.631
131.047
128.865
128.660
128.046
127.076
125.326
117.757
112.172
77.318
77.000
76.682
54.307
30.691
11.876
825
exp3 stdl3c
SAMPLE
date Oct 31 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 10000
ct 3152
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -162.3
wp 22151.9
vs 60
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10763.5
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Me
Bn
N
O
Ms
2c
200
180
160
140
120
100
80
60
40
20
ppm




13C OBSERVE
exp5 std13c
SAMPLE
date Nov 2 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 10000
ct 2760
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -3004.2
wp 24999.2
vs 50
sc 0
wc 250
hzmm 8.73
is 500.00
rfl 10758.2
rfp 7753.2
th 3
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
147.269
143.829
139.859
131.024
129.478
128.592
128.031
127.281
127.054
125.576
117.999
112.324
77.318
77.000
76.682
38.418
11.906
Me
Ph
N
O
Ms
2d
200
180
160
140
120
100
80
60
40
20
0
ppm

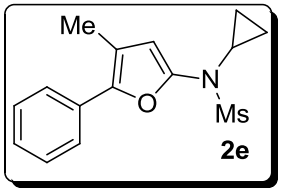

839
expl stdlh
SAMPLE
date Nov 8 2011
solvent CDCl3
file /export/home/~
chem/rsl/ramesh/RD~
-839-1H.fid
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
d1 1.000
tof 0
nt 100
ct 12
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 100
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3937.0
rfp 2899.2
th 4
ins 0.800
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.548
7.529
7.408
7.389
7.369
7.280
7.262
7.240
6.125
3.097
3.003
2.993
2.234
1.562
1.558
0.868
0.861
0.859
0.852
0.848
0.844
0.837
0.833
0.828
0.826
0.821
Me
N
O
Ms
2e
9
8
7
6
5
4
3
2
1
0 ppm
1.99 0.93
2.16
0.80
3.61
1.15
3.41
4.48

839

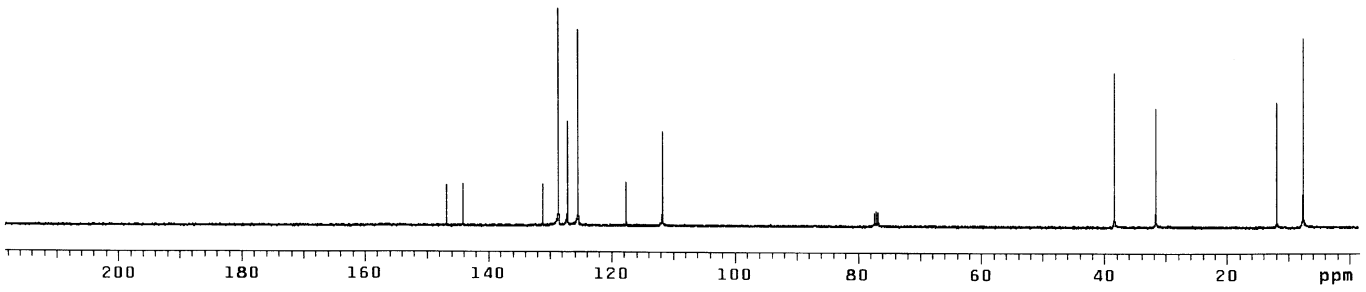
exp6 std13c

SAMPLE		DEC. & VT	
date	Nov 8 2011	dfrq	400.444
solvent	CDCl3	dn	H1
file	exp	dpwr	37
ACQUISITION		dof	0
sfrq	100.701	dm	yyy
tn	C13	dmm	w
at	1.199	dmf	8500
np	59968	PROCESSING	
sw	25000.0	lb	1.00
fb	13800	wtfile	
bs	4	proc	ft
tpwr	58	fn	not used
pw	8.7		
dl	0	werr	
tof	0	wexp	
nt	10000	wbs	
ct	1496	wnt	
alock	s		
gain	not used		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-162.3		
wp	22151.9		
vs	40		
sc	0		
wc	250		
hzmm	88.61		
is	500.00		
rfl	10763.5		
rfp	7753.2		
th	1		
ins	100.000		
nm nо ph			

146.769, 144.064, 131.062, 128.509, 127.076, 125.379, 117.582, 111.688, 77.311, 76.992, 76.674, 38.357, 31.659, 11.891, 7.610



2e



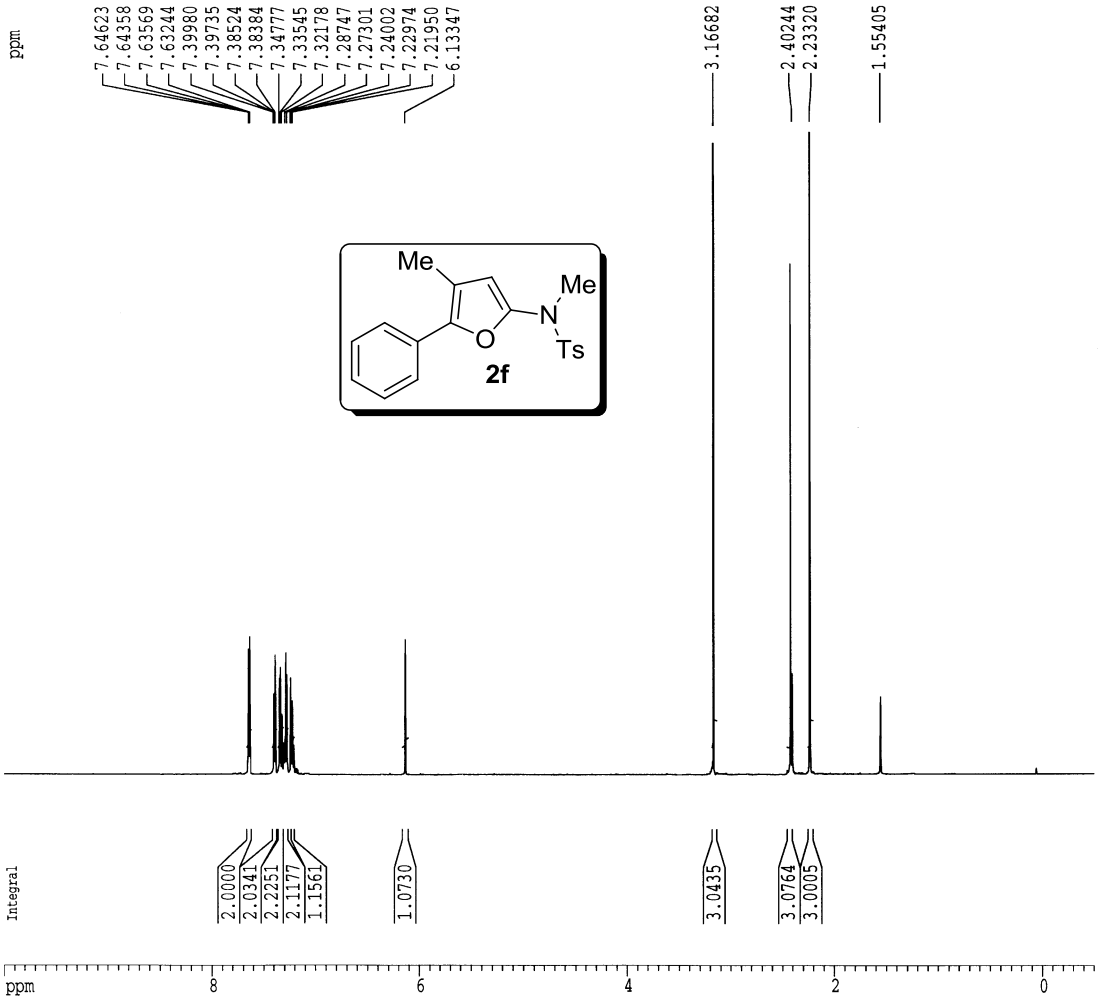
Current Data Parameters
NAME RD-821-P
EXPNO 1
PROCNO 1

F1 - Acquisition Parameters
Date_ 20121127
Time 13.34
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256020 Hz
AQ 1.9530228 sec
RG 128
DW 59.600 usec
DE 6.50 usec
TE 297.1 K
D1 1.50000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

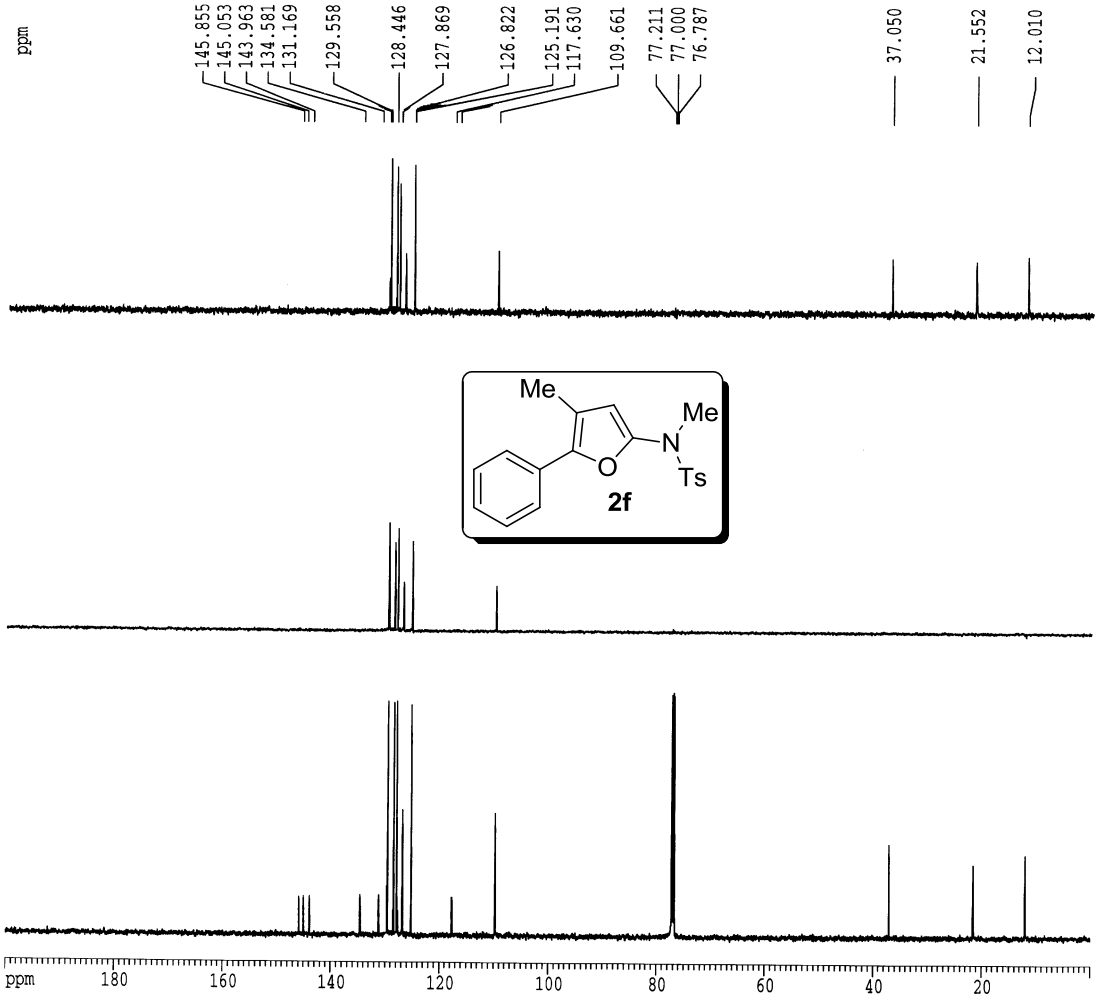
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 0.00 dB
SFO1 598.9028101 MHz

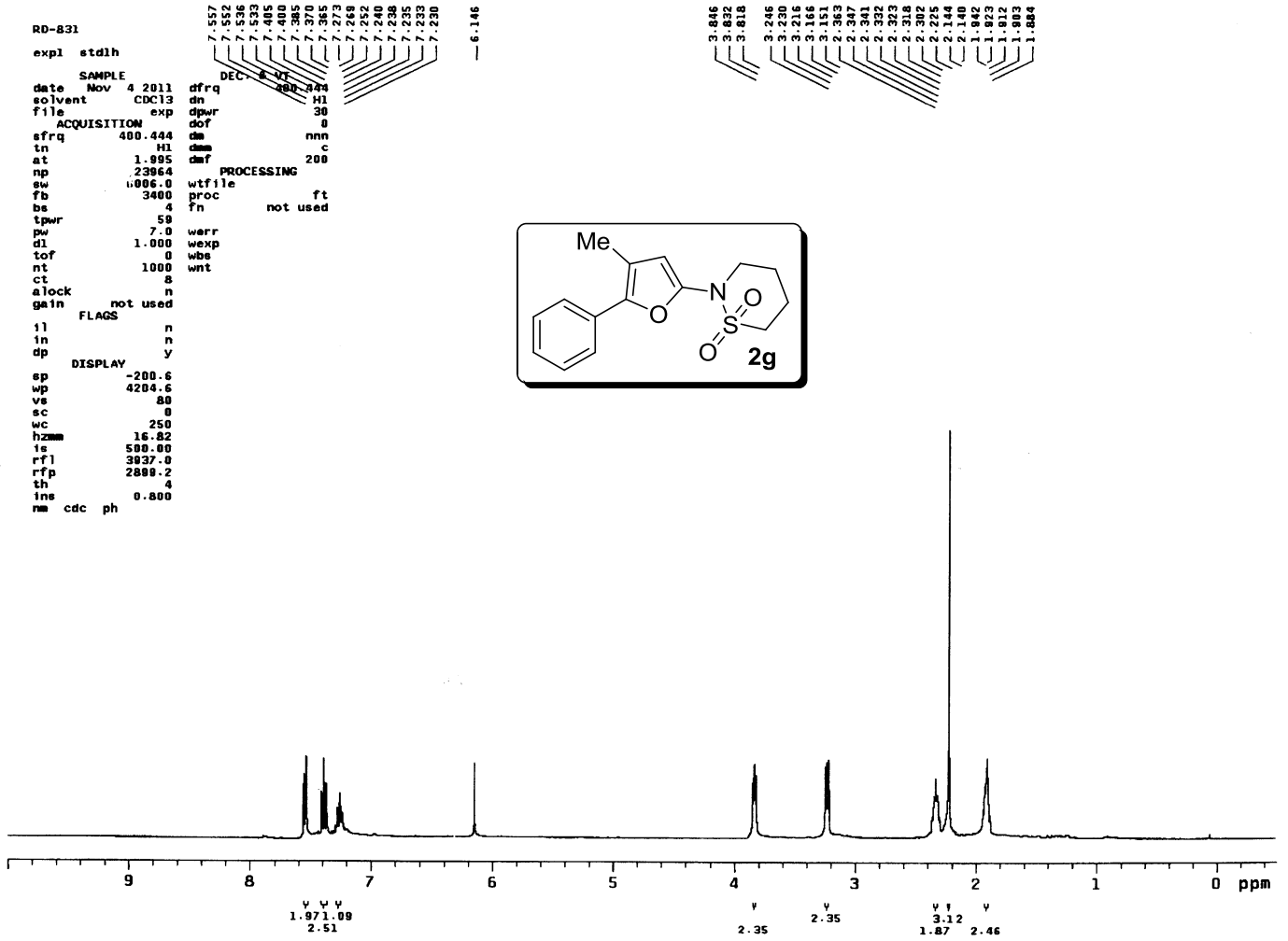
F2 - Processing parameters
SI 16384
SF 598.9000250 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

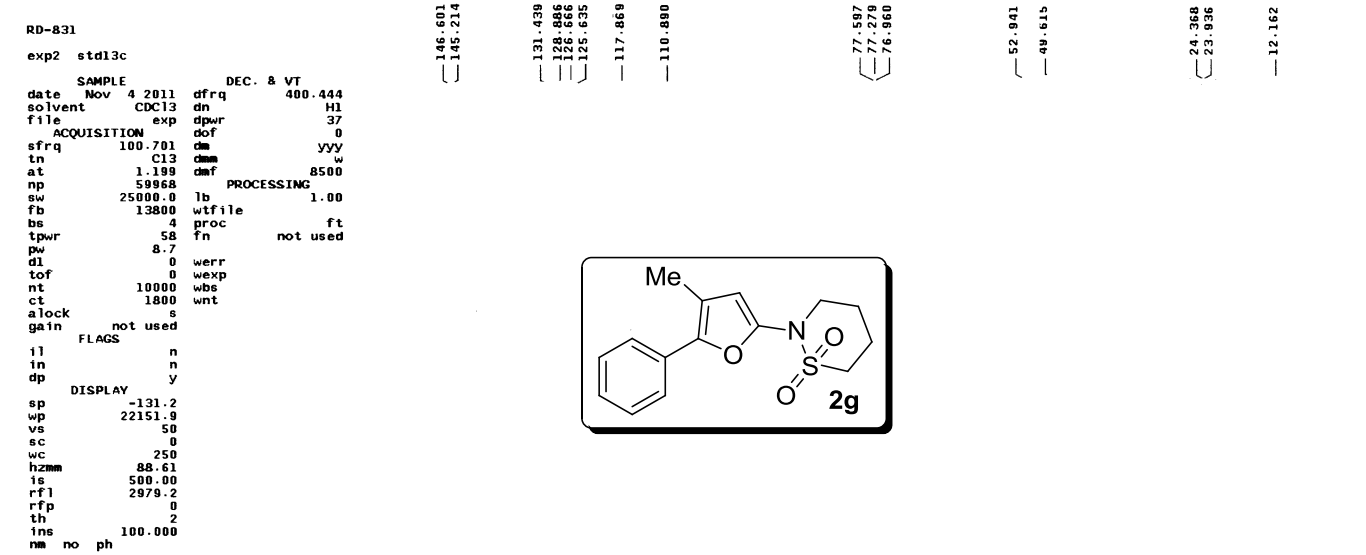
1D NMR plot parameters
CX 20.00 cm
CY 15.00 cm
F1P 10.000 ppm
F1 5989.00 Hz
F2P -0.500 ppm
F2 -299.45 Hz
PPMCM 0.52500 ppm/cm
HZCM 314.42252 Hz/cm



```
Current Data Parameters
NAME                  RD-821-P
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20111127
Time                     13.35
INSTRUM                  spect
PROBHD    5 mm QNP  1H/1
PULPROG                  zgpg
TD                       32768
SOLVENT                  CD2Cl2
NS                         414
DS                           0
SWH                45045.047 Hz
FIDRES                1.374666 Hz
AQ                   0.3637748 sec
RG                        4096
DW                      11.100 usec
DE                       6.50 usec
TE                      297.2 K
D1                   3.50000000 sec
d11                  0.03000000 sec
DELTA                3.40000010 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                        4.80 usec
PL1                       0.00 dB
SFO1               150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    92.00 usec
PL2                     120.00 dB
PL12                     11.30 dB
PL13                     14.00 dB
SFO2               598.9029990 MHz

F2 - Processing parameters
SI                        65536
SF                 150.5935293 MHz
WDW                          EM
SSB                           0
LB                        3.00 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                       20.00 cm
CY                        4.50 cm
F1P                     200.000 ppm
F1                    30118.71 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                 1505.93530 Hz/cm
```




RD-831
expl stdlh
SAMPLE
date Nov 4 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 400.444
tn H1
at 1.995
np 23964
sw 6006.0
fb 3400
bs 4
tpwr 59
pw 7.0
dl 1.000
tof 0
nt 1000
ct 8
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -200.6
wp 4204.6
vs 80
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3937.0
rfp 2899.2
th 4
ins 0.800
nm cdc ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
7.557
7.552
7.536
7.533
7.405
7.400
7.385
7.370
7.365
7.273
7.269
7.252
7.240
7.238
7.235
7.233
7.230
6.146
3.846
3.832
3.818
3.246
3.230
3.216
3.166
3.151
2.363
2.347
2.341
2.332
2.323
2.318
2.302
2.225
2.144
2.140
1.942
1.923
1.912
1.903
1.884
Me
N
O
O
S
O
O
2g
9
8
7
6
5
4
3
2
1
0
ppm
1.97
1.09
2.51
2.35
2.35
3.12
1.87
2.46


RD-831
exp2 std13c
SAMPLE
date Nov 4 2011
solvent CDCl3
file exp
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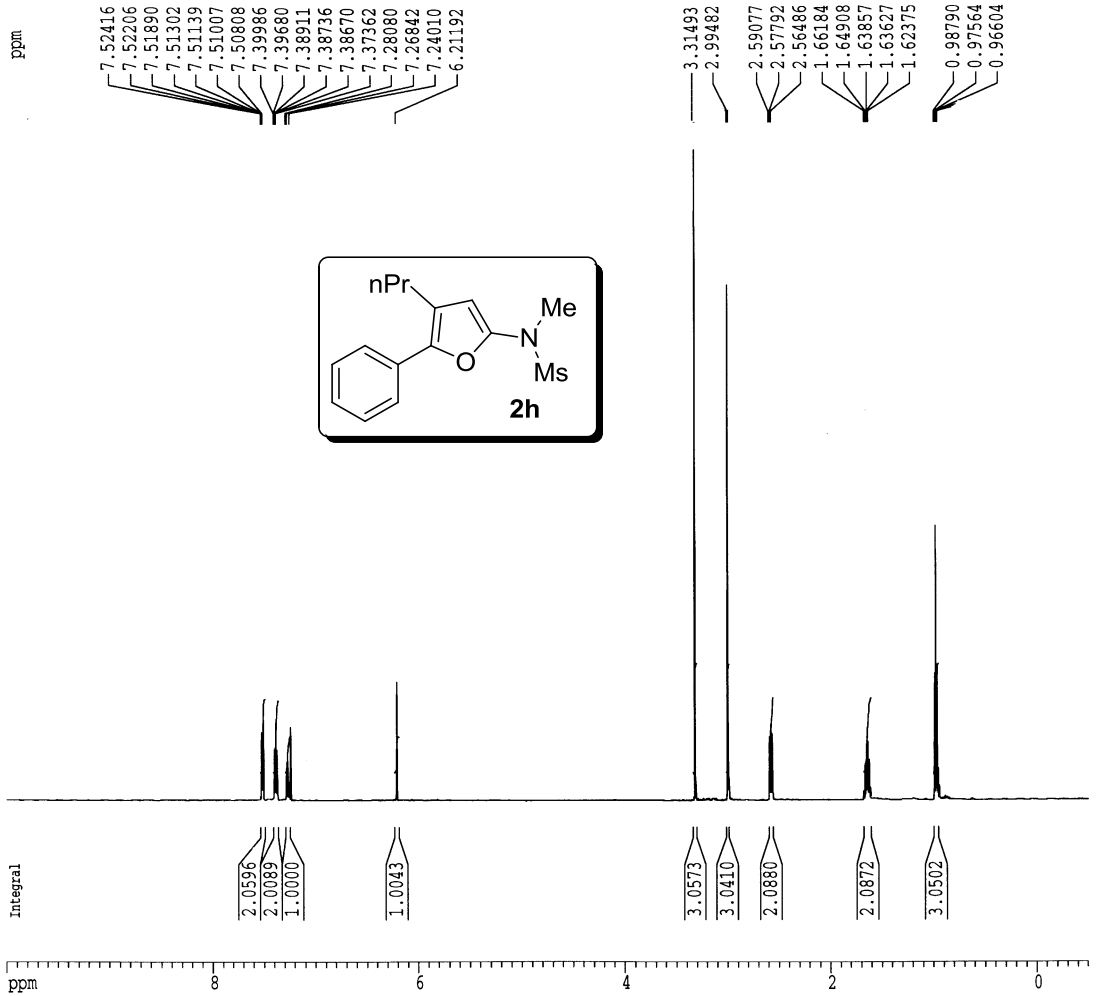
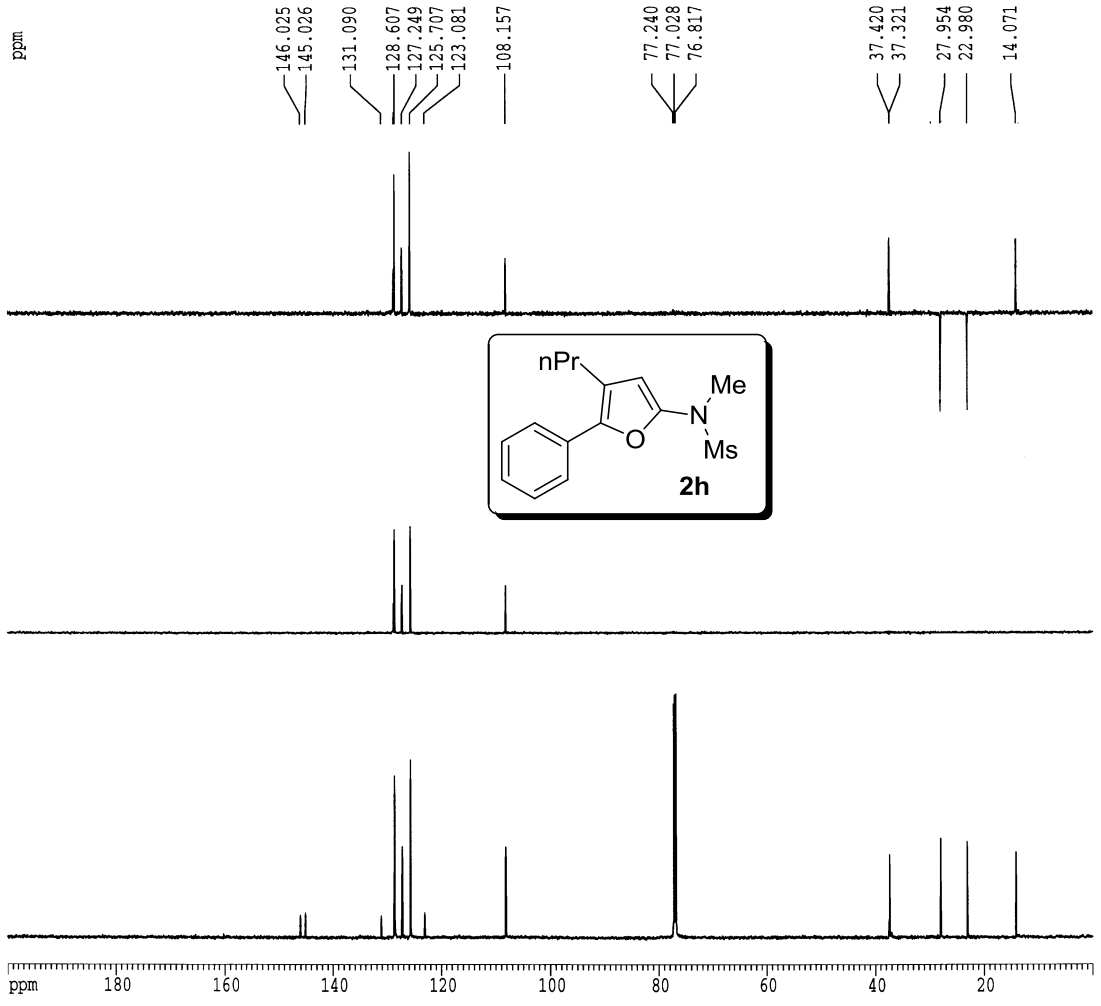
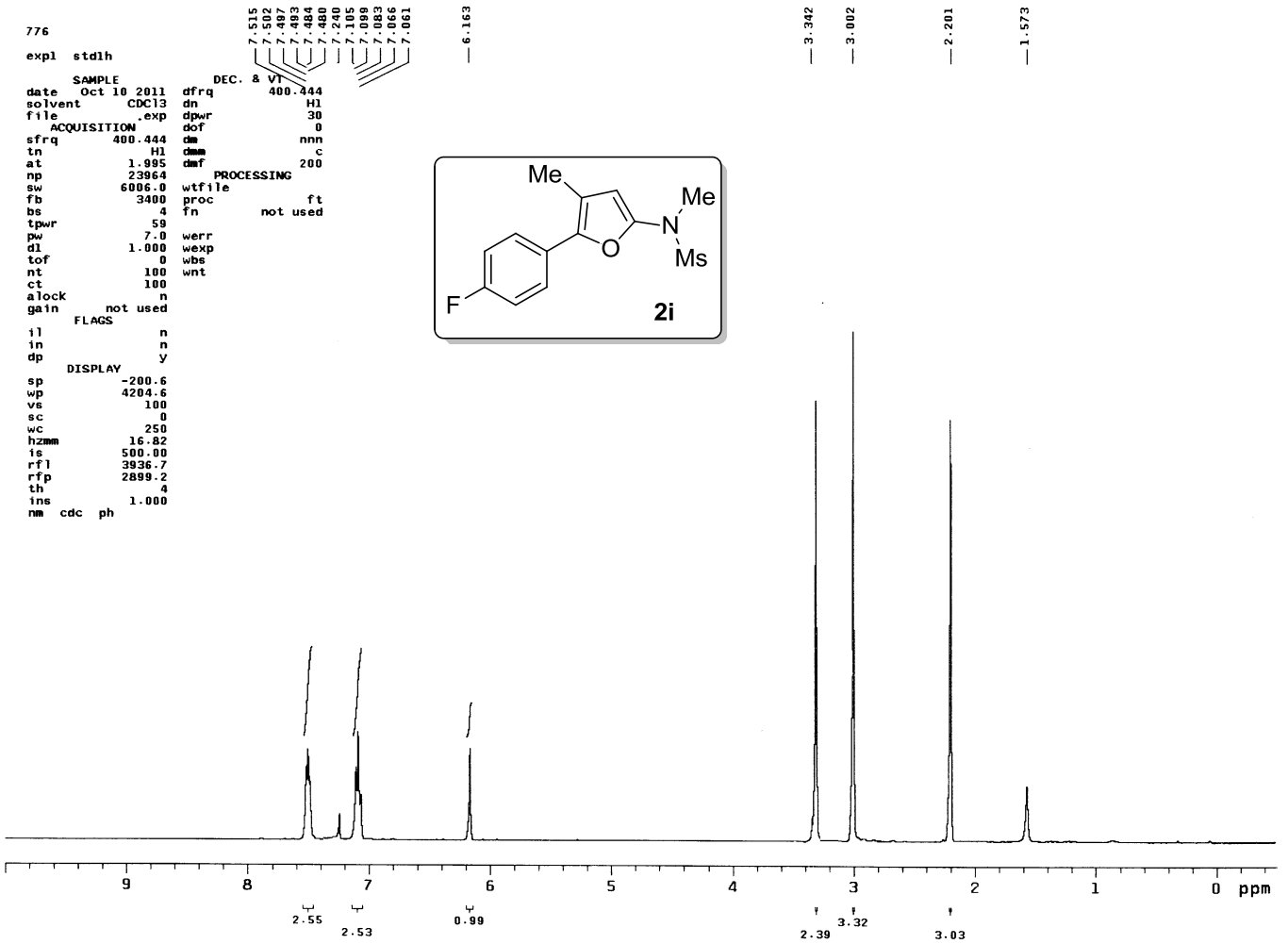
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 10000
ct 1800
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -131.2
wp 22151.9
vs 50
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 2979.2
rfp 0
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
146.601
145.214
131.439
128.886
126.666
125.635
117.869
110.890
77.597
77.279
76.960
52.941
49.615
24.368
23.936
12.162
Me
N
O
O
S
O
2g
200
180
160
140
120
100
80
60
40
20
ppm

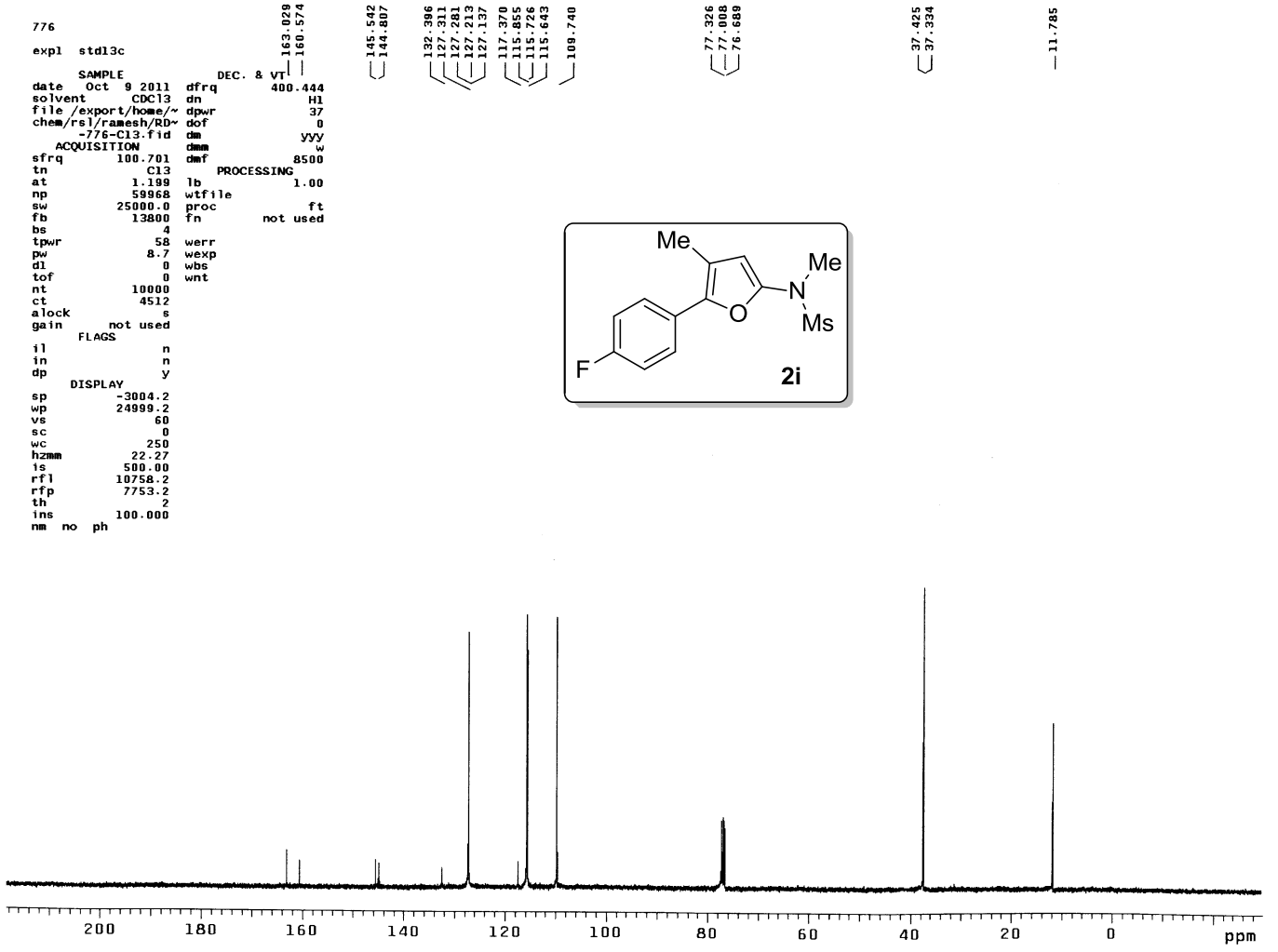
```
Current Data Parameters
NAME                  RD-815
EXPNO                      1
PROCNO                     1

F2 - Acquisition Parameters
Date_              20111222
Time                  18.34
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG                  zg
TD                    32768
SOLVENT               CDCl3
NS                       16
DS                        0
SWH                8389.262 Hz
FIDRES             0.256020 Hz
AQ                 1.9530228 sec
RG                      512
DW                   59.600 usec
DE                    6.50 usec
TE                   295.9 K
D1                1.00000000 sec
MCREST            0.00000000 sec
MCWRK             0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    8.20 usec
PL1                   0.00 dB
SFO1           598.9029945 MHz

F2 - Processing parameters
SI                    32768
SF              598.9000246 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   12.00 cm
F1P                 10.000 ppm
F1                 5989.00 Hz
F2P                 -0.500 ppm
F2                 -299.45 Hz
PPMCM               0.52500 ppm/cm
HZCM              314.42252 Hz/cm
```



```
Current Data Parameters
NAME                  RD-815
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_              20111222
Time                  18.58
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG                zgpg
TD                    32768
SOLVENT               CDCl3
NS                     1024
DS                        0
SWH               45045.047 Hz
FIDRES             1.374666 Hz
AQ                0.3637748 sec
RG                     4096
DW                   11.100 usec
DE                    6.50 usec
TE                   296.7 K
D1              3.50000000 sec
d11             0.03000000 sec
DELTA           3.40000010 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                     4.80 usec
PL1                    0.00 dB
SFO1            150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                 92.00 usec
PL2                  120.00 dB
PL12                  11.30 dB
PL13                  14.00 dB
SFO2            598.9029945 MHz

F2 - Processing parameters
SI                    32768
SF              150.5935252 MHz
WDW                      EM
SSB                       0
LB                     3.00 Hz
GB                        0
PC                     1.00

1D NMR plot parameters
CX                    20.00 cm
CY                     4.50 cm
F1P                 200.000 ppm
F1                30118.71 Hz
F2P                   0.000 ppm
F2                    0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM            1505.93518 Hz/cm
```



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145.542
144.807
132.396
127.311
127.281
127.213
127.137
117.370
115.855
115.726
115.643
109.740
77.326
77.008
76.689
37.425
37.334
11.785
776
expl stdl3c
SAMPLE
date Oct 9 2011
solvent CDCl3
file /export/home/~
chem/rsl/ramesh/RD~
-776-C13.fid
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tn C13
at 1.189
np 59968
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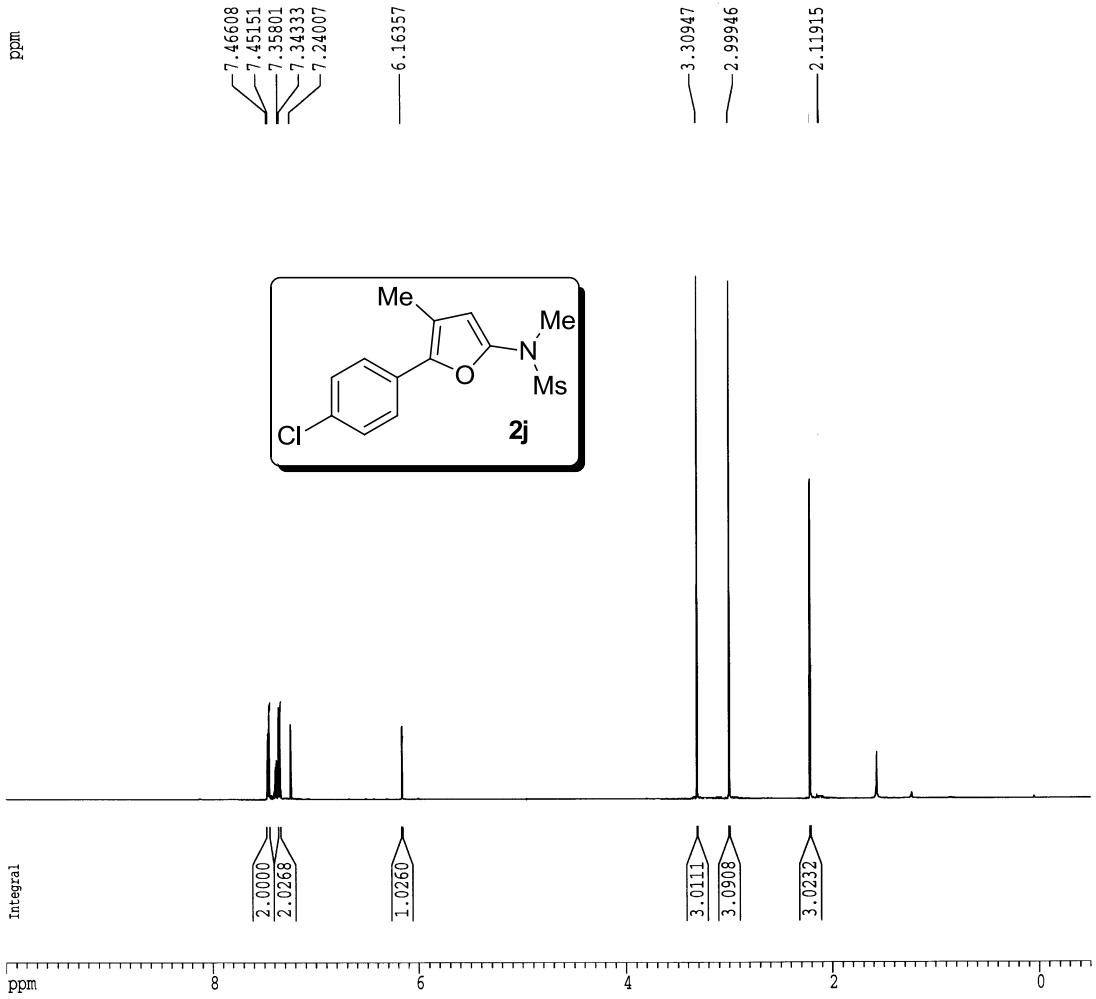
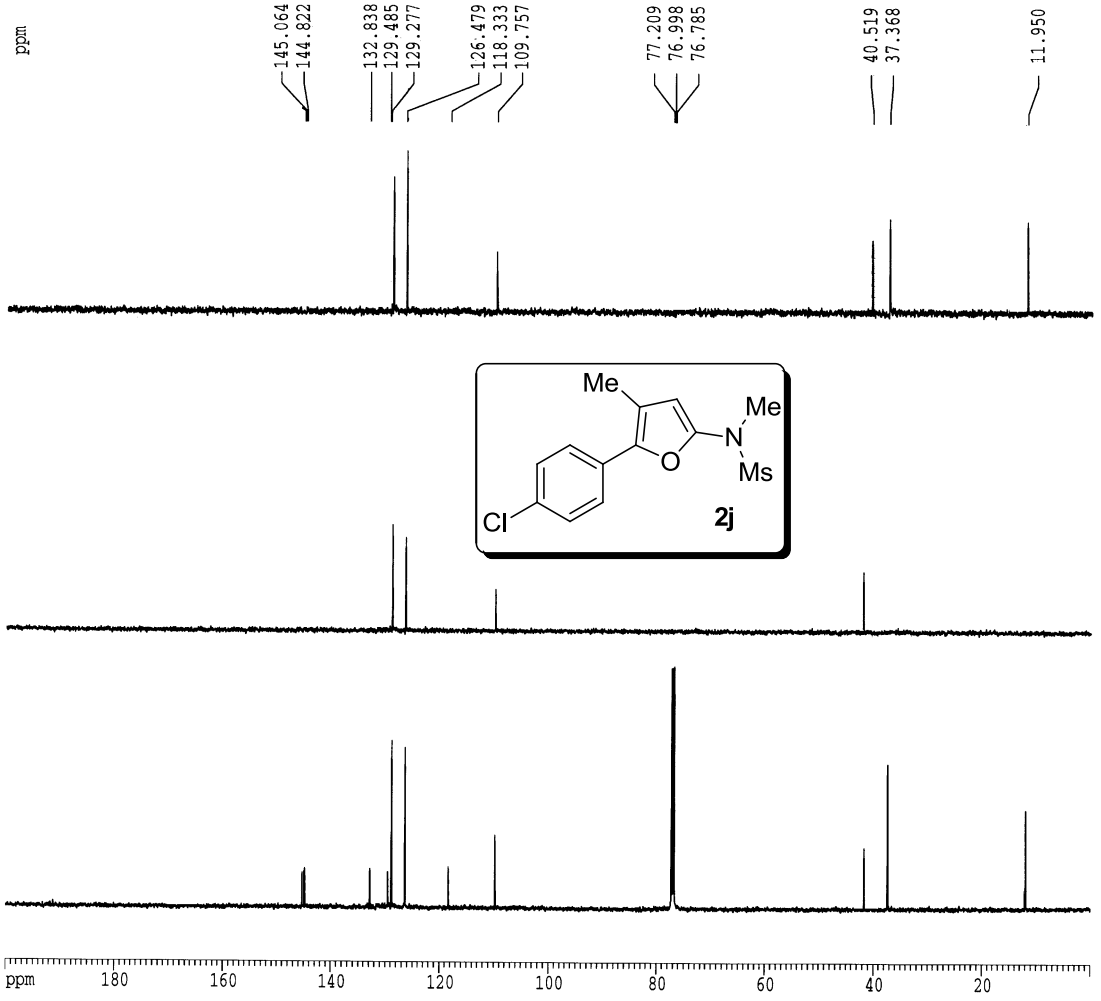
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 10000
ct 4512
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -3004.2
wp 24999.2
vs 60
sc 0
wc 250
hzmm 22.27
is 500.00
rfl 10758.2
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
Me
Me
N
Ms
O
F
2i
200
180
160
140
120
100
80
60
40
20
0
ppm

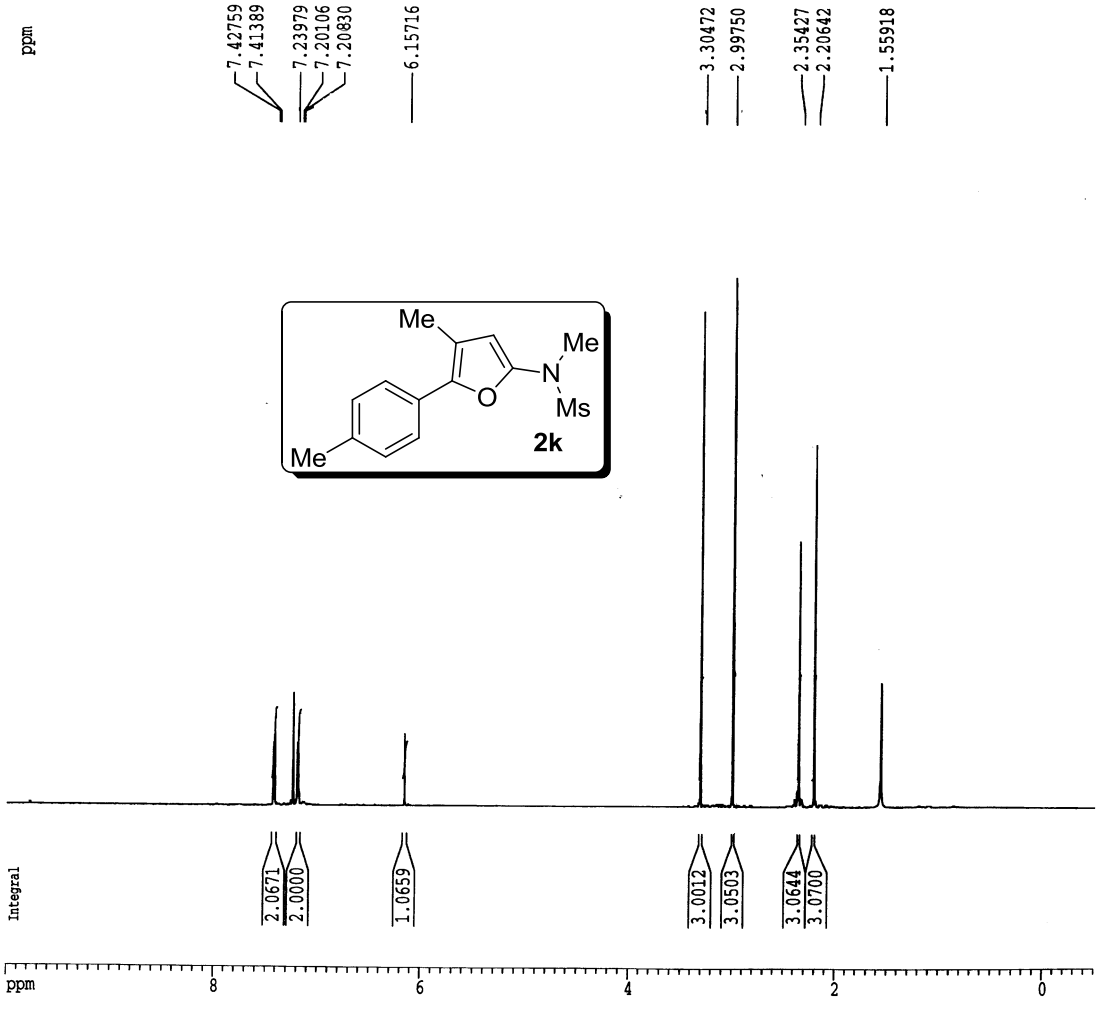
```
Current Data Parameters
NAME                  RD-880P
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_              20111213
Time                  12.54
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG                   zg
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                7183.908 Hz
FIDRES             0.219235 Hz
AQ                 2.2807028 sec
RG                        32
DW                   69.600 usec
DE                    6.50 usec
TE                   296.9 K
D1                 3.50000000 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                   10.00 usec
PL1                   0.00 dB
SFO1            598.9029945 MHz

F2 - Processing parameters
SI                     32768
SF              598.9000247 MHz
WDW                       no
SSB                        0
LB                    0.00 Hz
GB                         0
PC                      1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   10.00 cm
F1P                 10.000 ppm
F1                 5989.00 Hz
F2P                 -0.500 ppm
F2                 -299.45 Hz
PPMCM               0.52500 ppm/cm
HZCM              314.42252 Hz/cm
```



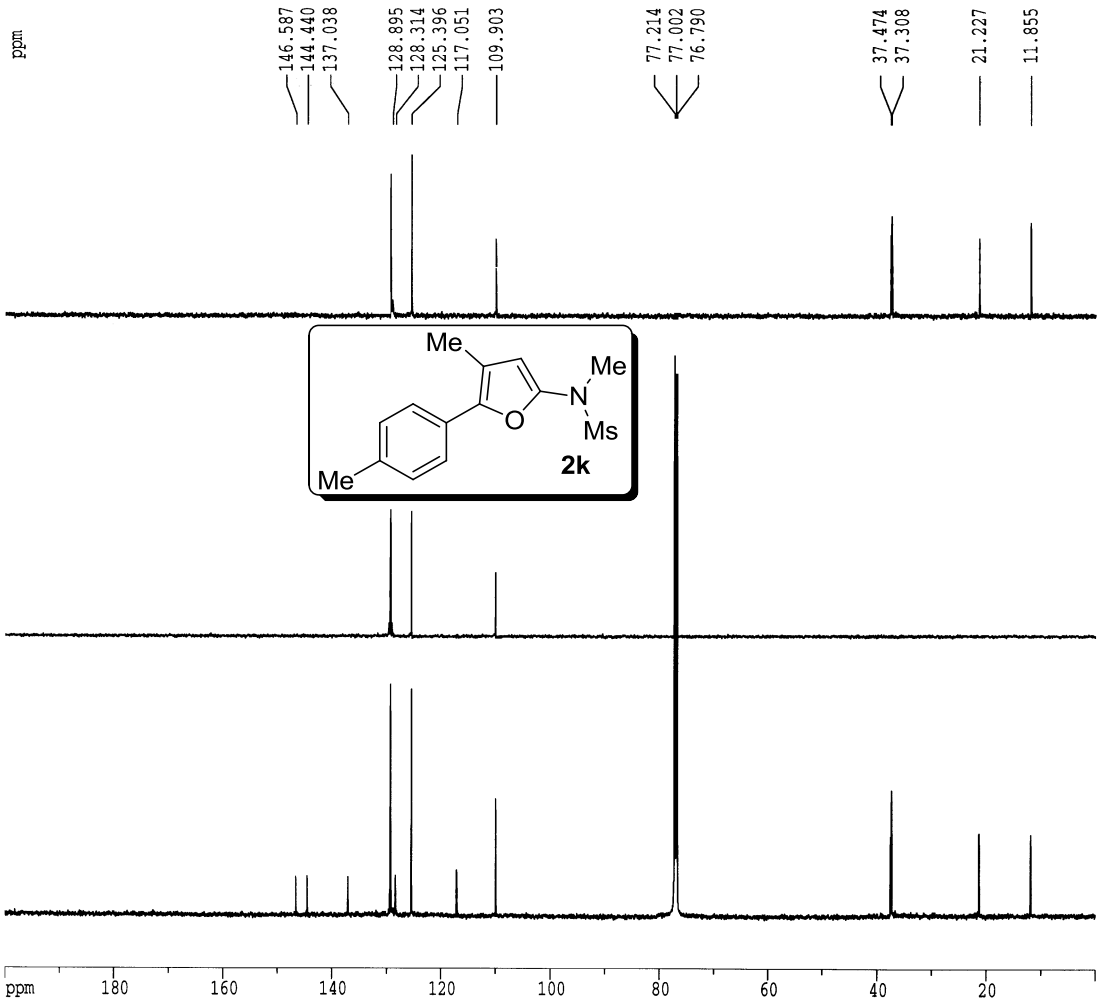
```
Current Data Parameters
NAME                 RD-880P
EXPNO                      2
PROCNO                     1

F2 - Acquisition Parameters
Date_               20111213
Time                   13.08
INSTRUM                spect
PROBHD   5 mm QNP  1H/1
PULPROG                zgpg
TD                     32768
SOLVENT                CDCl3
NS                       500
DS                         0
SWH                45045.047 Hz
FIDRES                 1.374666 Hz
AQ                 0.3637748 sec
RG                      4096
DW                     11.100 usec
DE                      6.50 usec
TE                     297.7 K
D1                 3.50000000 sec
d11                0.03000000 sec
DELTA              3.40000010 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                       4.80 usec
PL1                      0.00 dB
SFO1             150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   92.00 usec
PL2                    120.00 dB
PL12                    11.30 dB
PL13                    14.00 dB
SFO2             598.9029990 MHz

F2 - Processing parameters
SI                       65536
SF               150.5935293 MHz
WDW                         EM
SSB                          0
LB                        3.00 Hz
GB                           0
PC                        1.00

1D NMR plot parameters
CX                       20.00 cm
CY                        4.50 cm
F1P                     200.000 ppm
F1                    30118.71 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM              1505.93530 Hz/cm
```



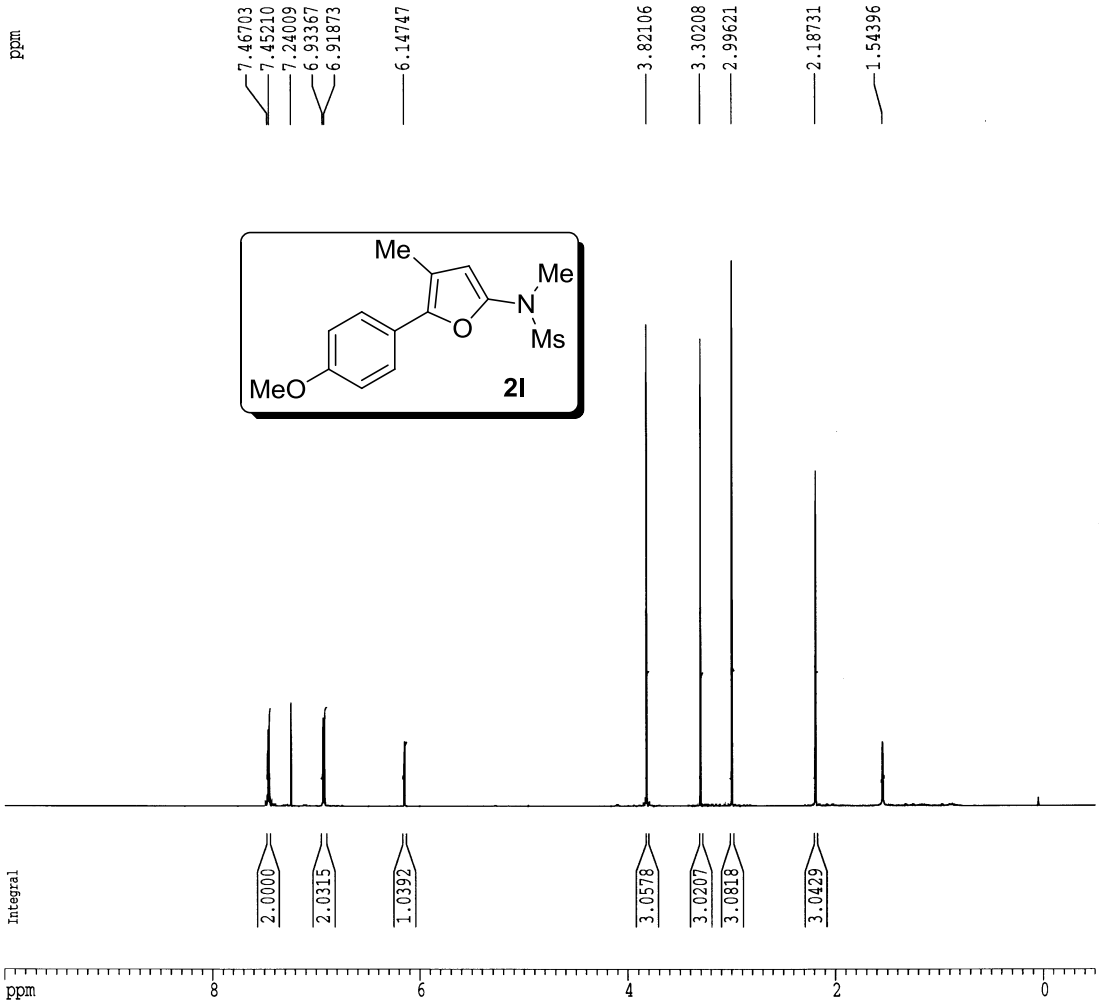
```
Current Data Parameters
NAME                  RD-873-2
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_               20111107
Time                     21.48
INSTRUM                  spect
PROBHD    5 mm QNP  1H/1
PULPROG                    zg
TD                       32768
SOLVENT                  C6D6
NS                          16
DS                           0
SWH                 8389.262 Hz
FIDRES              0.256020 Hz
AQ                  1.9530228 sec
RG                         256
DW                    59.600 usec
DE                     6.50 usec
TE                     297.3 K
D1                  1.00000000 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                    10.00 usec
PL1                    0.00 dB
SFO1              598.9035934 MHz

F2 - Processing parameters
SI                       32768
SF                598.9000487 MHz
WDW                         no
SSB                          0
LB                     0.00 Hz
GB                           0
PC                     1.00

1D NMR plot parameters
CX                     20.00 cm
CY                     10.00 cm
F1P                   10.000 ppm
F1                  5989.00 Hz
F2P                   -0.500 ppm
F2                  -299.45 Hz
PPMCM                 0.52500 ppm/cm
HZCM                314.42252 Hz/cm
```



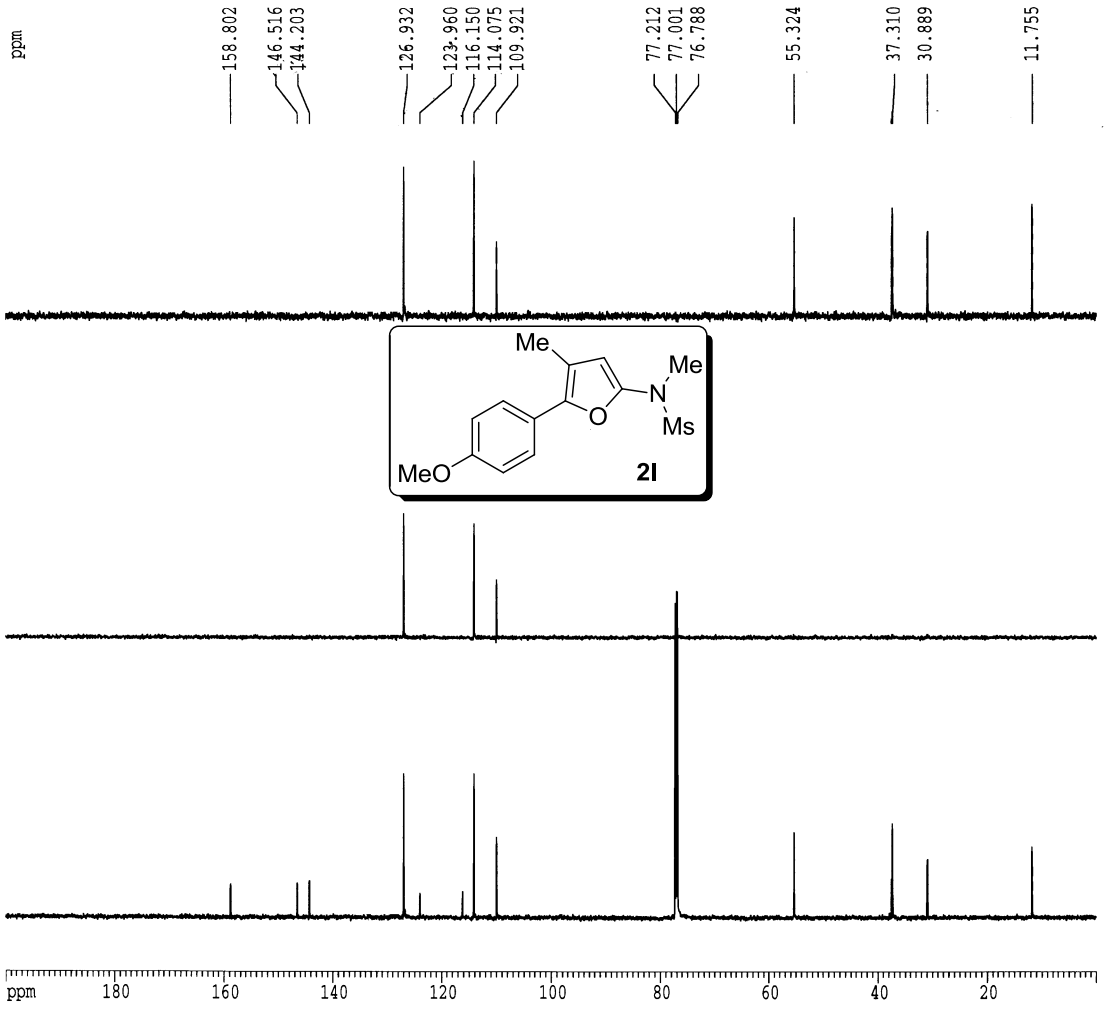
```
Current Data Parameters
NAME           RD-873-2
EXPNO                 2
PROCNO                1

F2 - Acquisition Parameters
Date_          20111207
Time              21.49
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG           zgpg
TD               32768
SOLVENT           DMSO
NS                6144
DS                   0
SWH          45045.047 Hz
FIDRES        1.374666 Hz
AQ           0.3637748 sec
RG                4096
DW              11.100 usec
DE               6.50 usec
TE              297.3 K
D1           3.50000000 sec
d11          0.03000000 sec
DELTA        3.40000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1               13C
P1                4.80 usec
PL1               0.00 dB
SFO1       150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2        waltz16
NUC2               1H
PCPD2            92.00 usec
PL2             120.00 dB
PL12             11.30 dB
PL13             14.00 dB
SFO2       598.9029990 MHz

F2 - Processing parameters
SI                65536
SF          150.5935327 MHz
WDW                  EM
SSB                   0
LB                 3.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                20.00 cm
F1P              200.000 ppm
F1             30118.71 Hz
F2P              0.000 ppm
F2               0.00 Hz
PPMCM         10.00000 ppm/cm
HZCM        1505.93530 Hz/cm
```

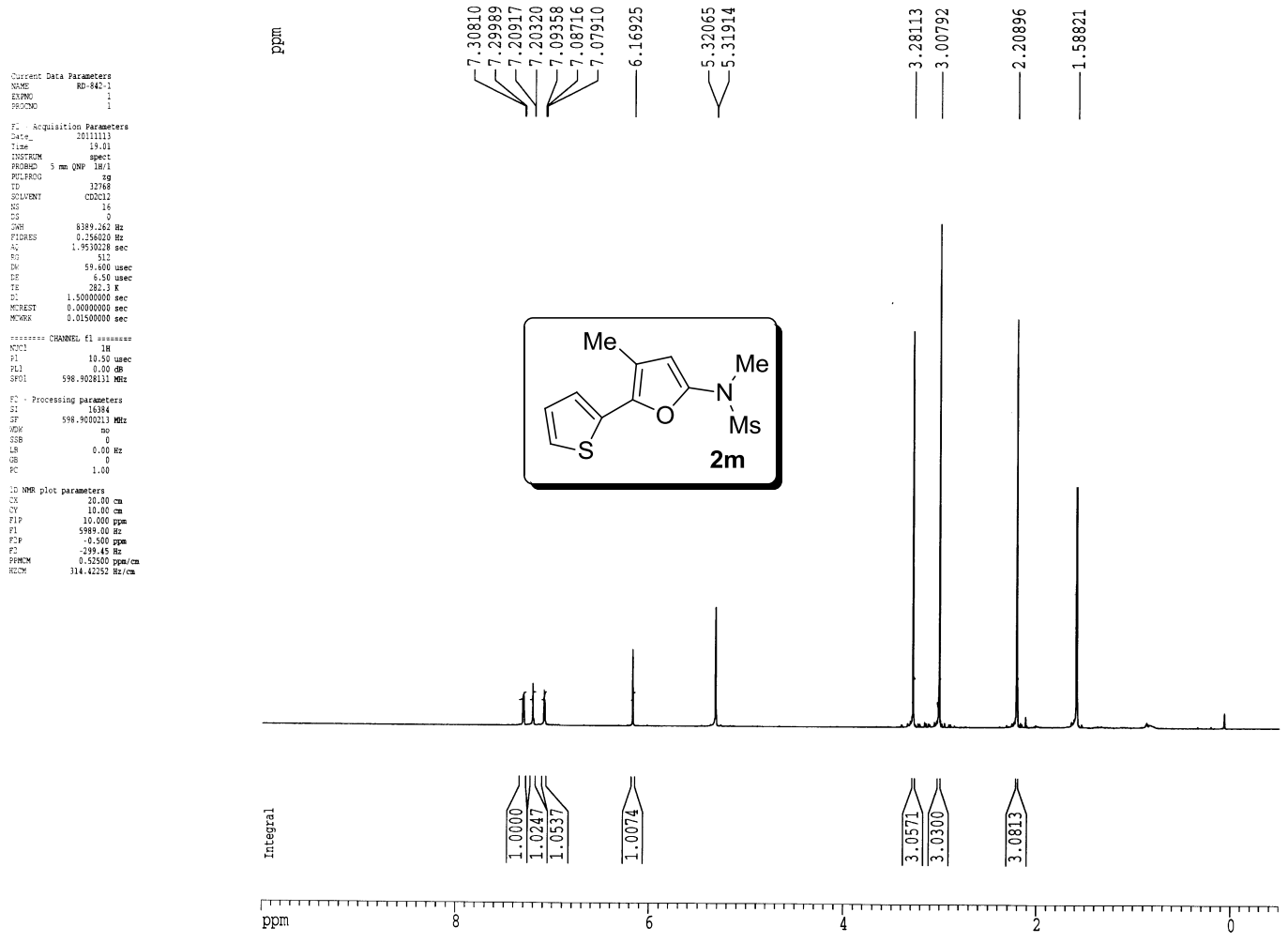


```
Current Data Parameters
NAME              RD-870
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111214
Time               18.30
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            7183.908 Hz
FIDRES        0.219235 Hz
AQ            2.2807028 sec
RG                   32
DW               69.600 usec
DE                6.50 usec
TE                300.1 K
D1           1.00000000 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1               10.00 usec
PL1               0.00 dB
SFO1        598.9029945 MHz

F2 - Processing parameters
SI                32768
SF          598.9000234 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                10.00 cm
F1P               10.000 ppm
F1              5989.00 Hz
F2P               -0.500 ppm
F2              -299.45 Hz
PPMCM           0.52500 ppm/cm
HZCM          314.42252 Hz/cm
```

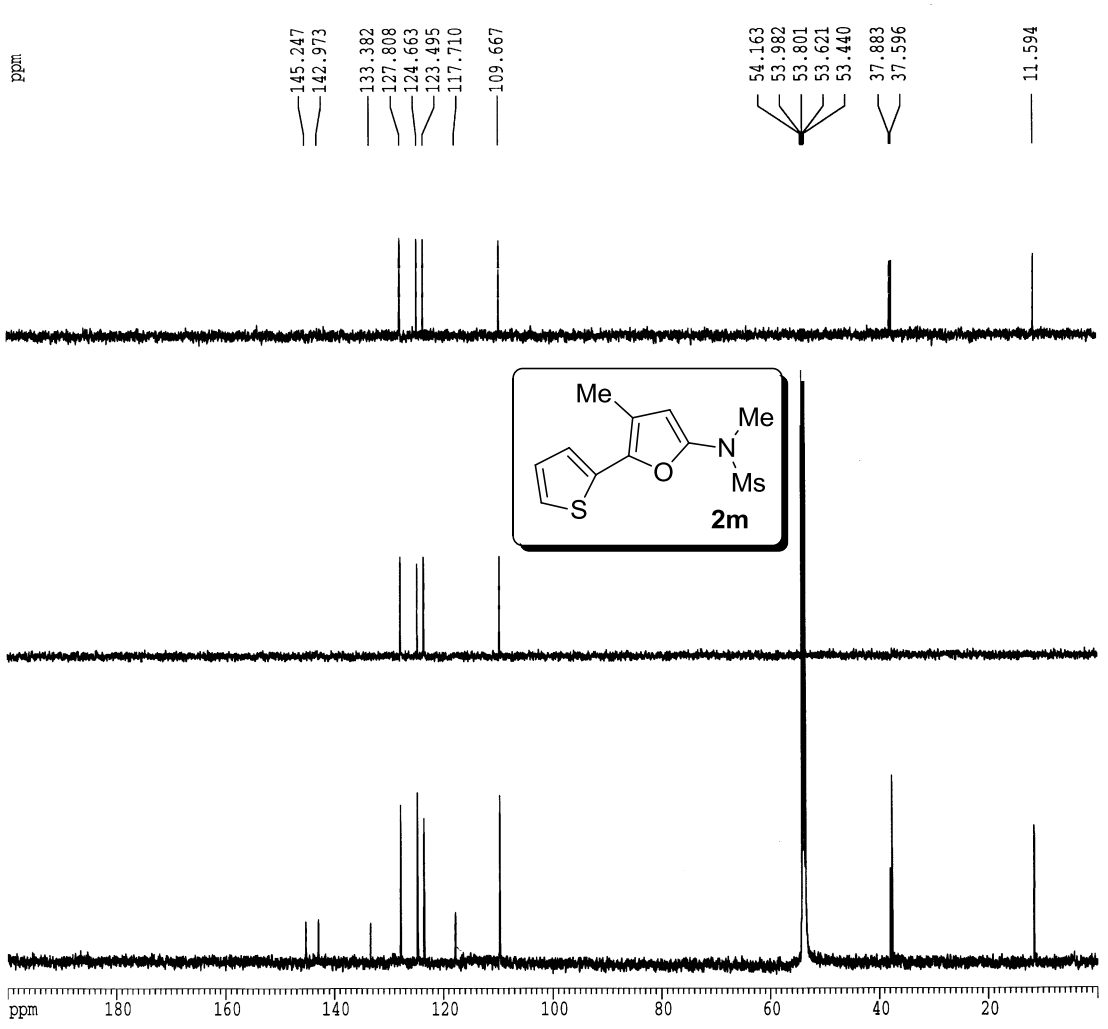


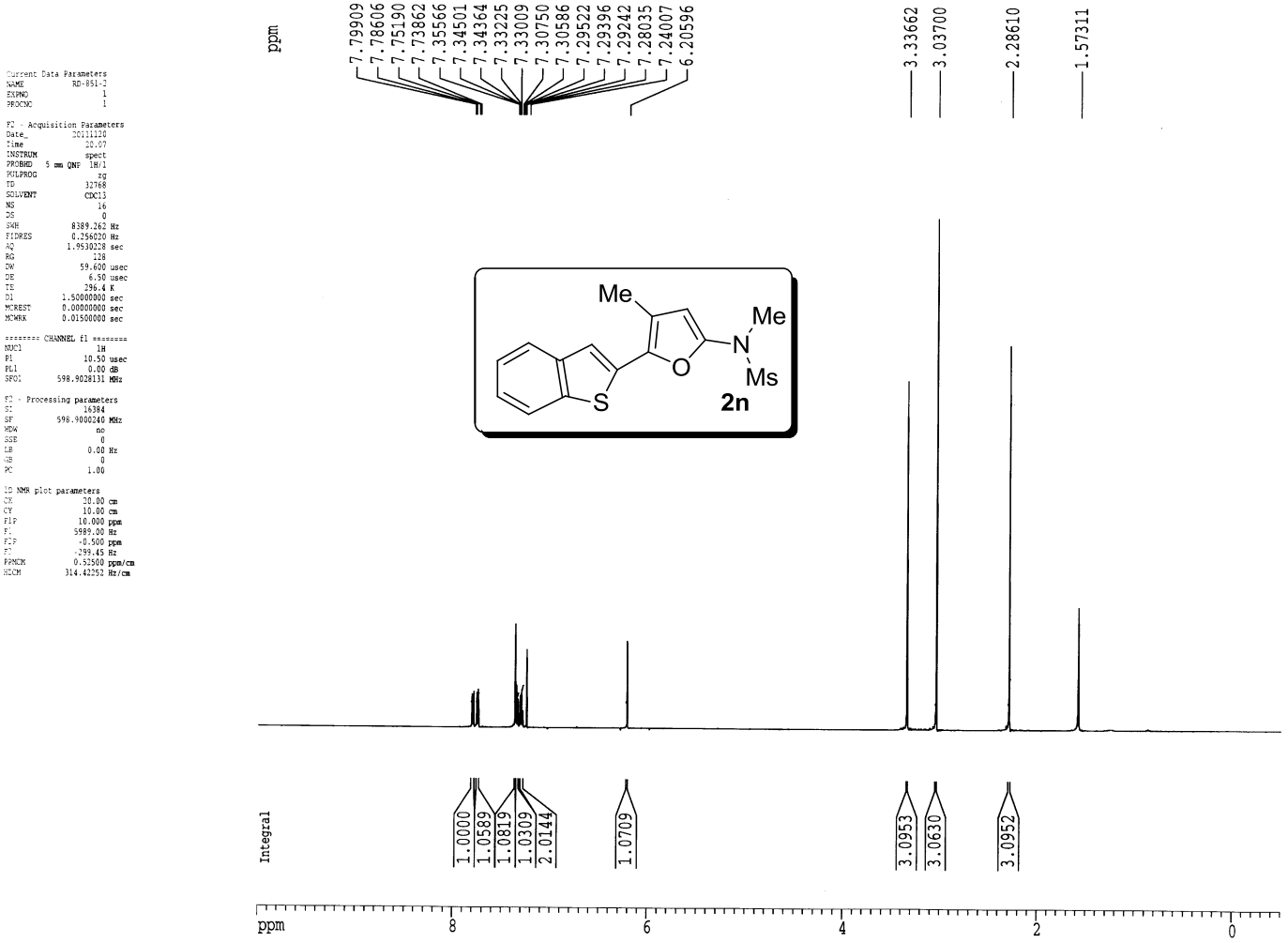
```
Current Data Parameters
NAME              RD-870
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111214
Time               18.54
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG            zgpg
TD                 32768
SOLVENT            CDCl3
NS                   968
DS                     0
SWH            45045.047 Hz
FIDRES          1.374666 Hz
AQ              0.3637748 sec
RG                  4096
DW                11.100 usec
DE                 6.50 usec
TE                300.1 K
D1            3.50000000 sec
d11           0.03000000 sec
DELTA         3.40000010 sec
MCREST        0.00000000 sec
MCWRK         0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  4.80 usec
PL1                 0.00 dB
SFO1         150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              92.00 usec
PL2               120.00 dB
PL12               11.30 dB
PL13               14.00 dB
SFO2         598.9029990 MHz

F2 - Processing parameters
SI                  65536
SF           150.5935265 MHz
WDW                    EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                  6.00 cm
F1P              200.000 ppm
F1             30118.71 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1505.93518 Hz/cm
```



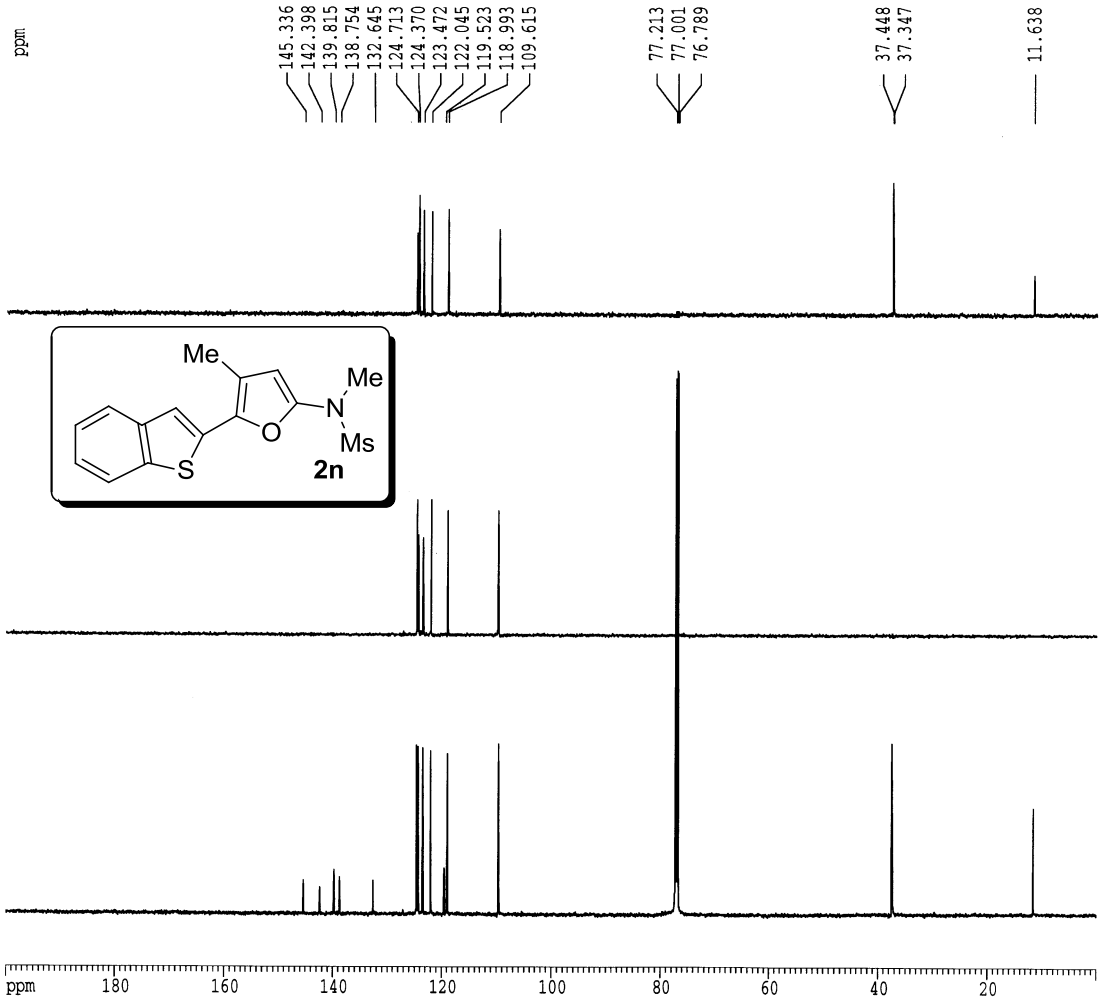


```
Current Data Parameters
NAME            RD-842-1
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111114
Time               13.47
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zgpg
TD                 32768
SOLVENT           CD2Cl2
NS                  2576
DS                     0
SWH          45045.047 Hz
FIDRES        1.374666 Hz
AQ            0.3637748 sec
RG                  4096
DW             11.100 usec
DE              6.50 usec
TE              296.4 K
D1          3.50000000 sec
d11         0.03000000 sec
DELTA       3.40000010 sec
MCREST      0.00000000 sec
MCWRK       0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1               4.80 usec
PL1              0.00 dB
SFO1       150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2           92.00 usec
PL2             120.00 dB
PL12             11.30 dB
PL13             14.00 dB
SFO2       598.9029990 MHz

F2 - Processing parameters
SI                 65536
SF          150.5934675 MHz
WDW                   EM
SSB                    0
LB               3.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX              20.00 cm
CY              30.00 cm
F1P            200.000 ppm
F1            30118.69 Hz
F2P             0.000 ppm
F2              0.00 Hz
PPMCM        10.00000 ppm/cm
HZCM       1505.93457 Hz/cm
```

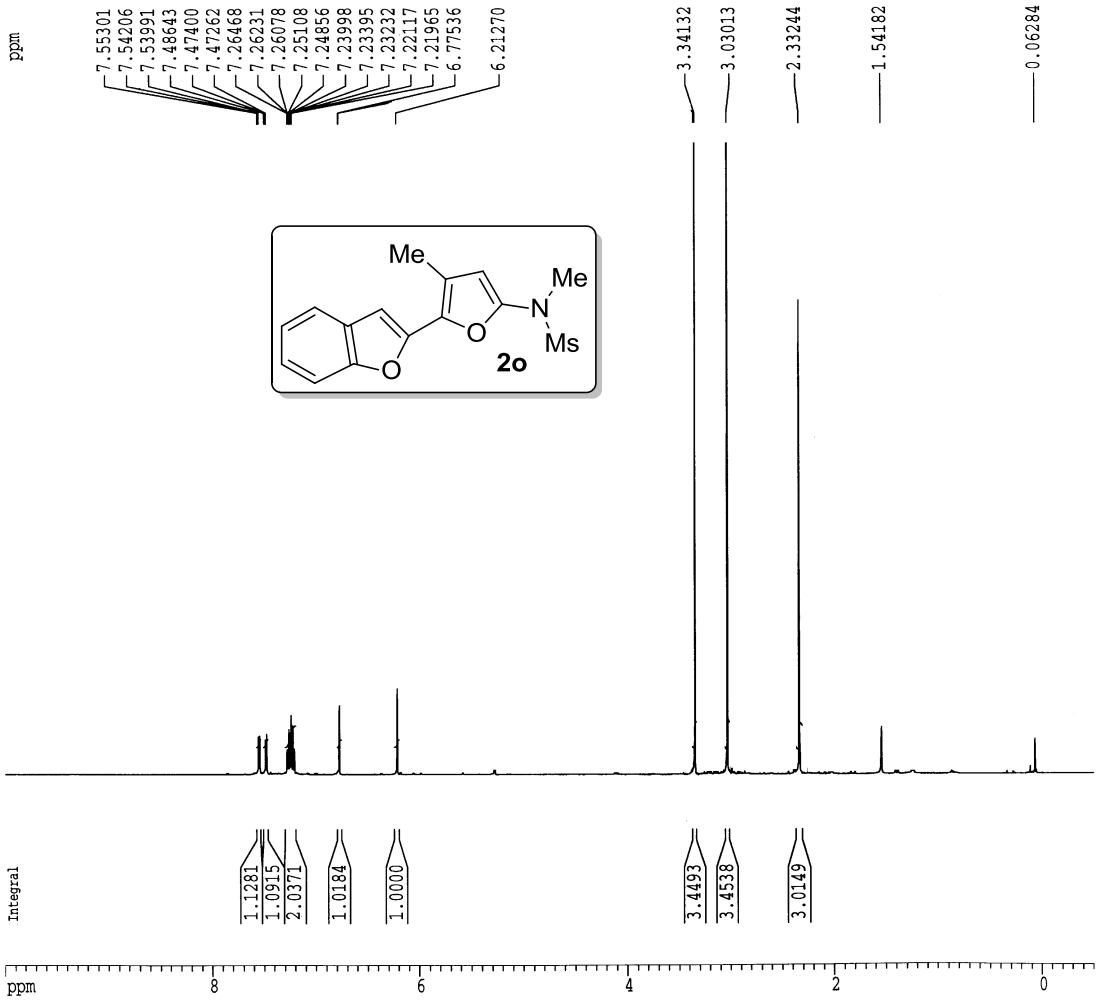




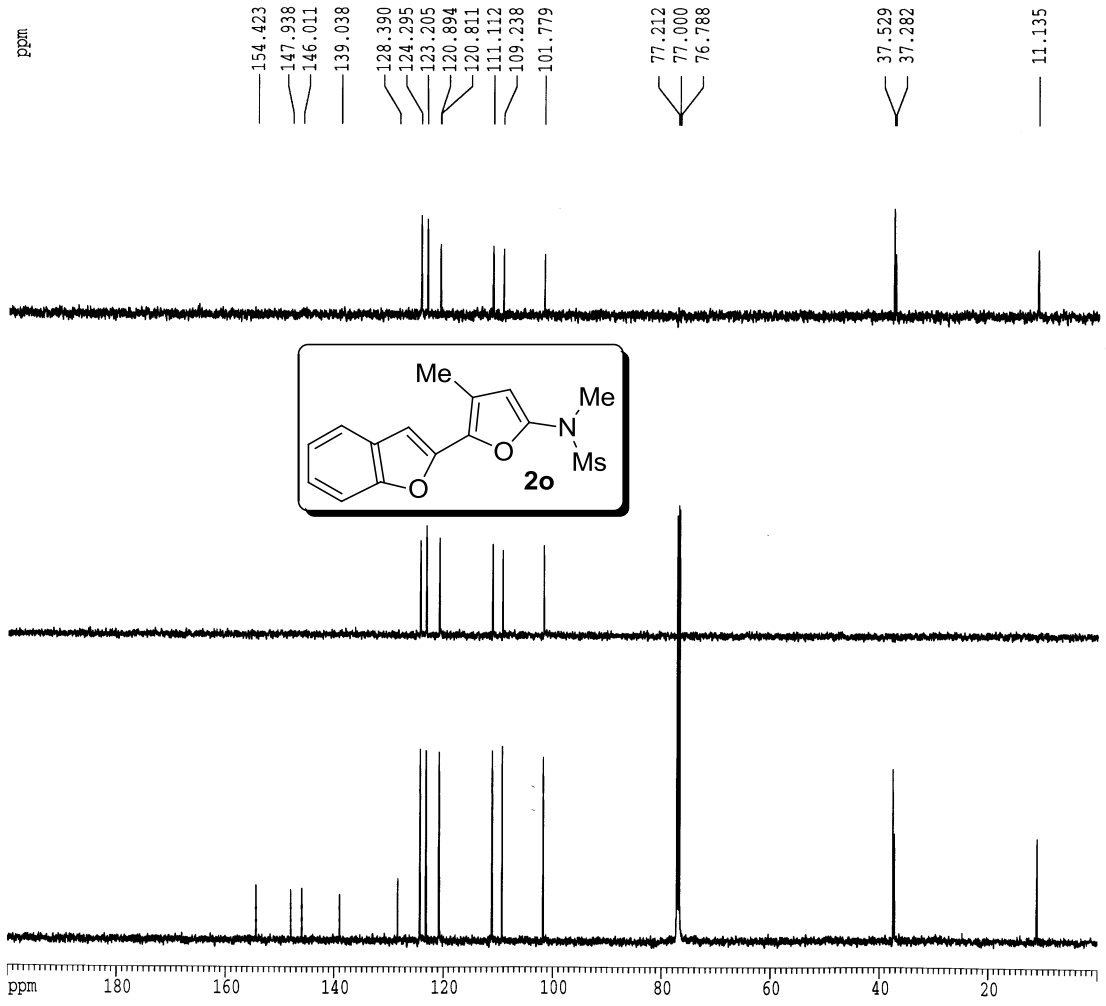
```
Current Data Parameters
NAME            RD-851-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20111120
Time               20.16
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG            zgpg
TD                 32768
SOLVENT            CD2Cl2
NS                  3072
DS                     0
SWH           45045.047 Hz
FIDRES         1.374666 Hz
AQ            0.3637748 sec
RG                  4096
DW                11.100 usec
DE                 6.50 usec
TE                297.0 K
D1           3.50000000 sec
d11          0.03000000 sec
DELTA        3.40000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 4.80 usec
PL1                0.00 dB
SFO1        150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                1H
PCPD2              92.00 usec
PL2               120.00 dB
PL12               11.30 dB
PL13               14.00 dB
SFO2        598.9029990 MHz

F2 - Processing parameters
SI                 65536
SF          150.5935293 MHz
WDW                   EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                 10.00 cm
F1P               200.000 ppm
F1              30118.71 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1505.93530 Hz/cm
```



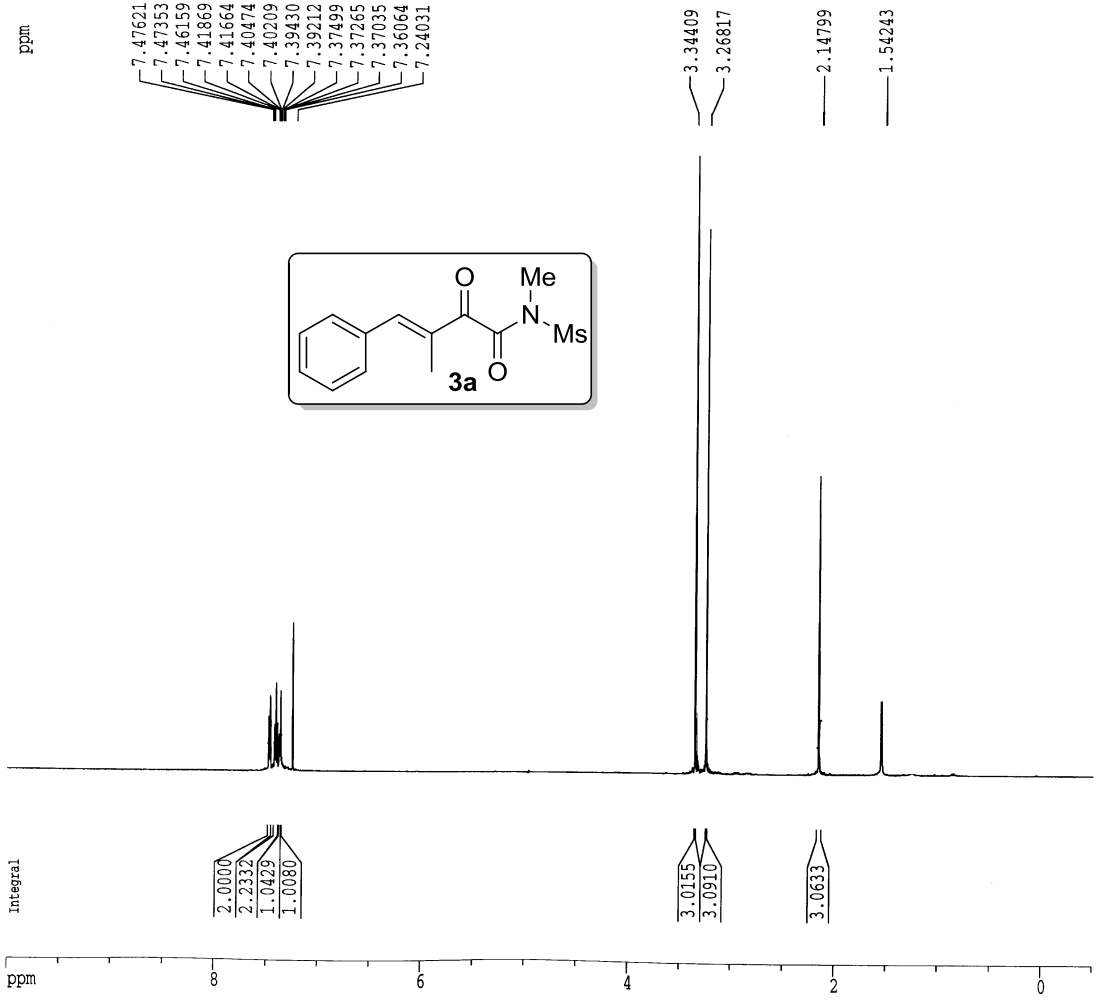
```
Current Data Parameters
NAME                 RD-859-1
EXPNO                       1
PROCNO                      1

F2 - Acquisition Parameters
Date_               20111127
Time                   16.43
INSTRUM                spect
PROBHD     5 mm QNP  1H/1
PULPROG                 zg
TD                     32768
SOLVENT                CDCl3
NS                        16
DS                         0
SWH                 8389.262 Hz
FIDRES                 0.256020 Hz
AQ                  1.9530228 sec
RG                       128
DW                    59.600 usec
DE                     6.50 usec
TE                     298.1 K
D1                  1.50000000 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                      10.50 usec
PL1                      0.00 dB
SFO1             598.9028101 MHz

F2 - Processing parameters
SI                        16384
SF                598.9000255 MHz
WDW                          no
SSB                           0
LB                       0.00 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                      20.00 cm
CY                      15.00 cm
F1P                   10.000 ppm
F1                    5989.00 Hz
F2P                    -0.500 ppm
F2                    -299.45 Hz
PPMCM                 0.52500 ppm/cm
HZCM                314.42252 Hz/cm
```



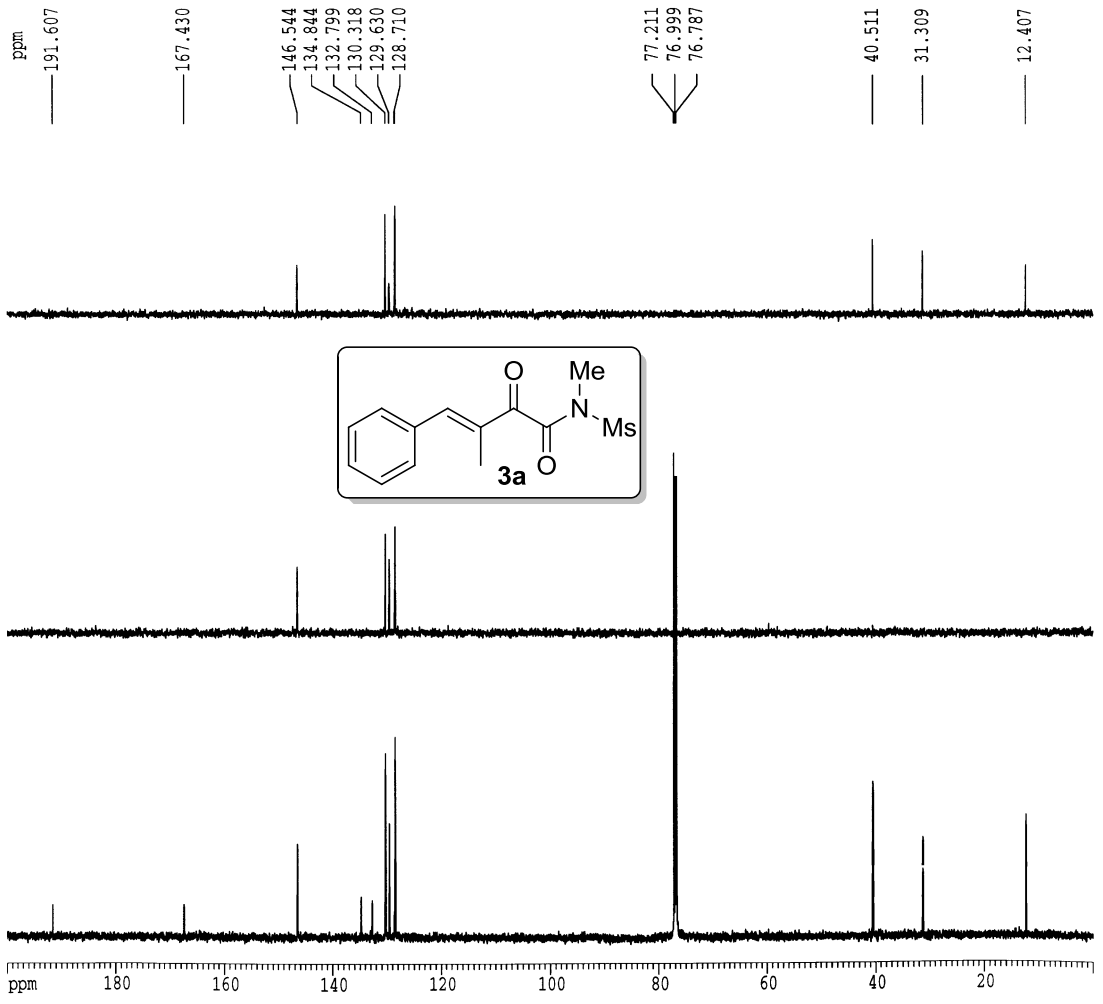
```
Current Data Parameters
NAME                  RD-859-1
EXPNO                        2
PROCNO                       1

F1 - Acquisition Parameters
Date_               20111127
Time                   14.46
INSTRUM                spect
PROBHD   5 mm QNP  1H/1
PULPROG                 zgpg
TD                     32768
SOLVENT             CD2Cl2
NS                      1015
DS                         0
SWH                45045.047 Hz
FIDRES                1.374666 Hz
AQ                    0.3637748 sec
RG                        4096
DW                      11.100 usec
DE                       6.50 usec
TE                      297.7 K
D1                 3.50000000 sec
d11                0.03000000 sec
DELTA              3.40000010 sec
MCREST             0.00000000 sec
MCWRK              0.01500000 sec

======== CHANNEL f1 ========
NUC1                      13C
P1                       4.80 usec
PL1                      0.00 dB
SFO1               150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   92.00 usec
PL2                    120.00 dB
PL12                    11.30 dB
PL13                    14.00 dB
SFO2               598.9029990 MHz

F2 - Processing parameters
SI                        65536
SF                 150.5935279 MHz
WDW                        EM
SSB                         0
LB                       3.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      20.00 cm
CY                       8.00 cm
F1P                    200.000 ppm
F1                   30118.71 Hz
F2P                      0.000 ppm
F2                       0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                1505.93518 Hz/cm
```



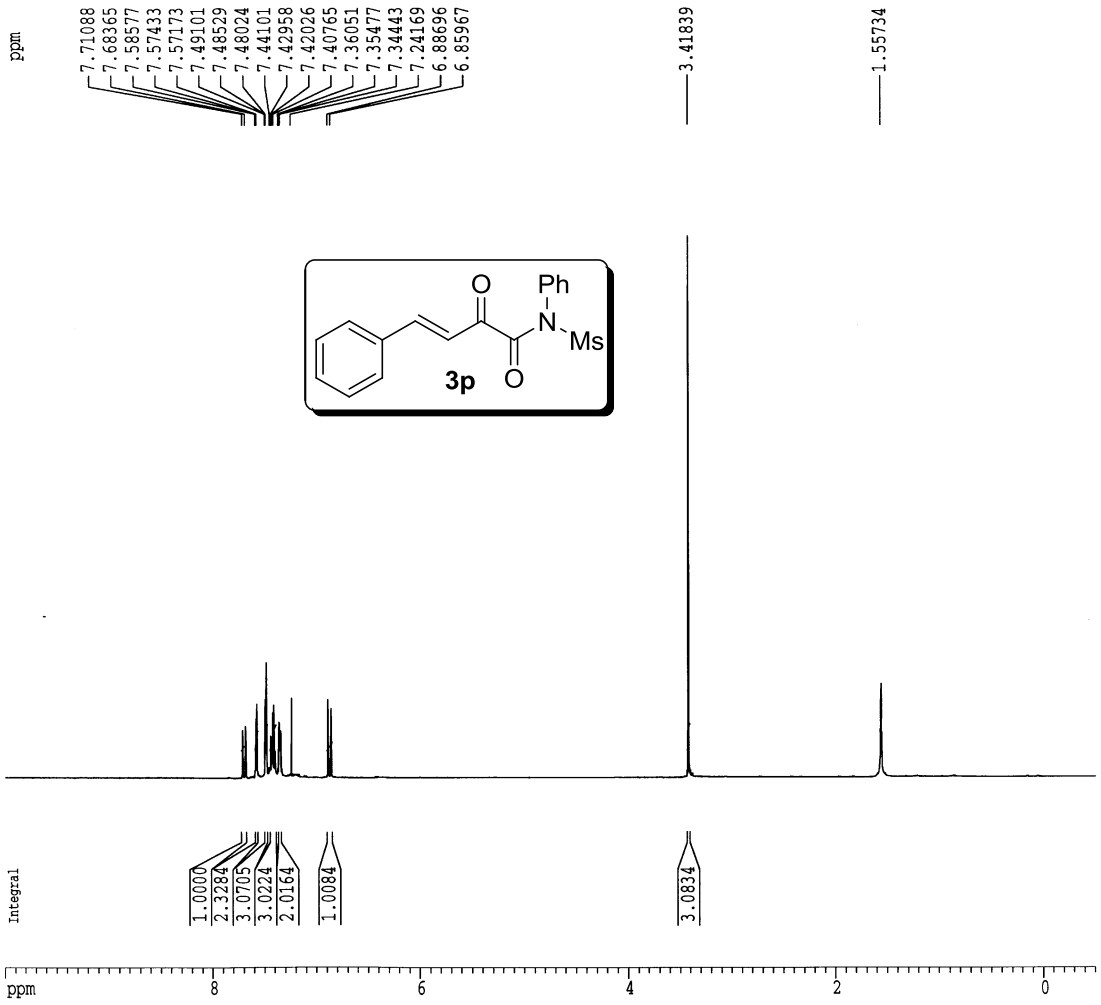
```
Current Data Parameters
NAME              RD-946-P
EXPNO                    1
PROCNO                   1

F2 - Acquisition Parameters
Date_           20120405
Time              14.48
INSTRUM           spect
PROBHD   5 mm QNP  1H/1
PULPROG             zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ             1.9530228 sec
RG                  512
DW               59.600 usec
DE                6.50 usec
TE                296.4 K
D1             2.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                9.80 usec
PL1               0.00 dB
SFO1        598.9029945 MHz

F2 - Processing parameters
SI                32768
SF          598.9000248 MHz
WDW                  no
SSB                   0
LB                0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX               20.00 cm
CY               12.00 cm
F1P             10.000 ppm
F1              5989.00 Hz
F2P             -0.500 ppm
F2             -299.45 Hz
PPMCM          0.52500 ppm/cm
HZCM         314.42252 Hz/cm
```

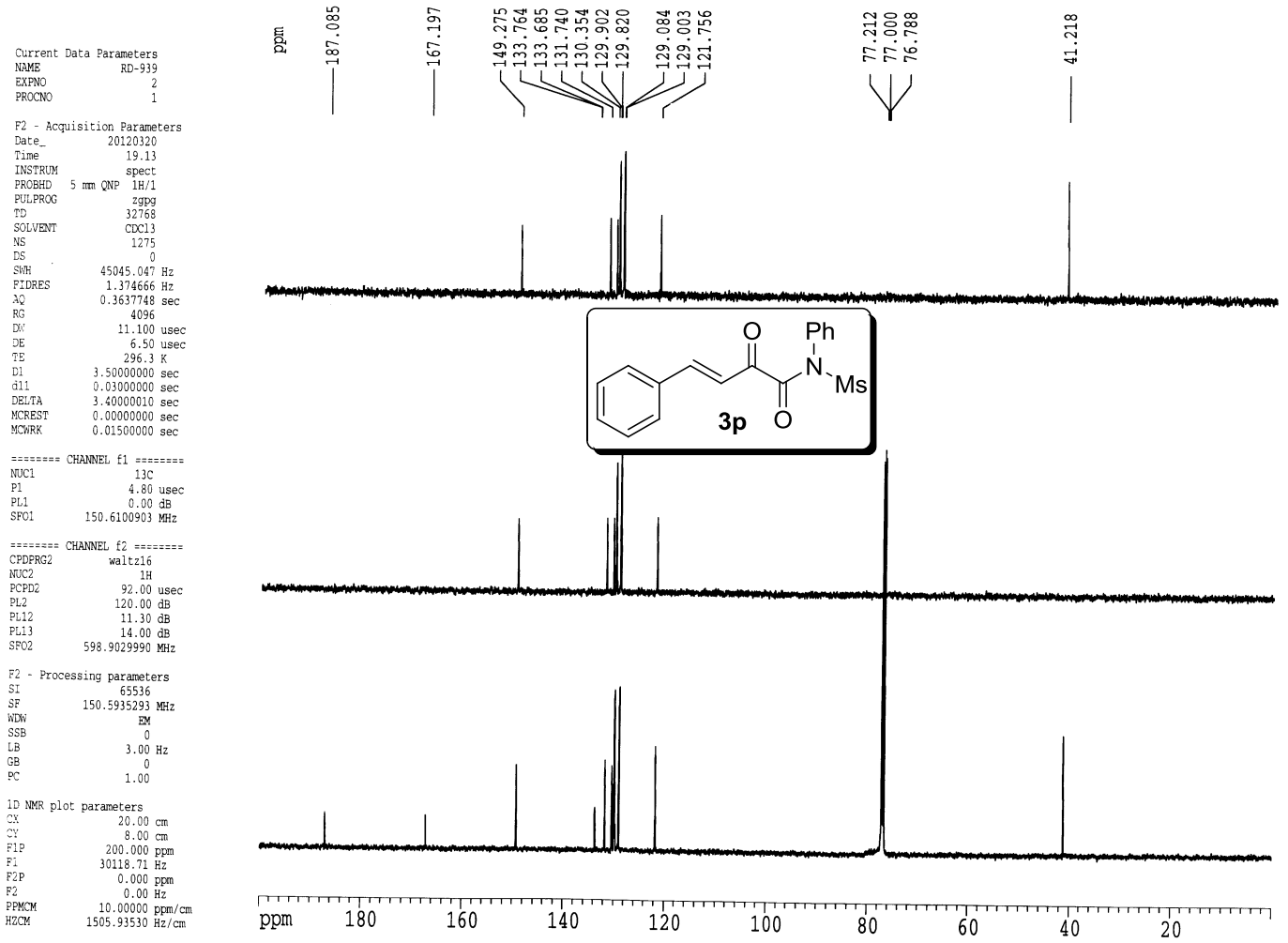


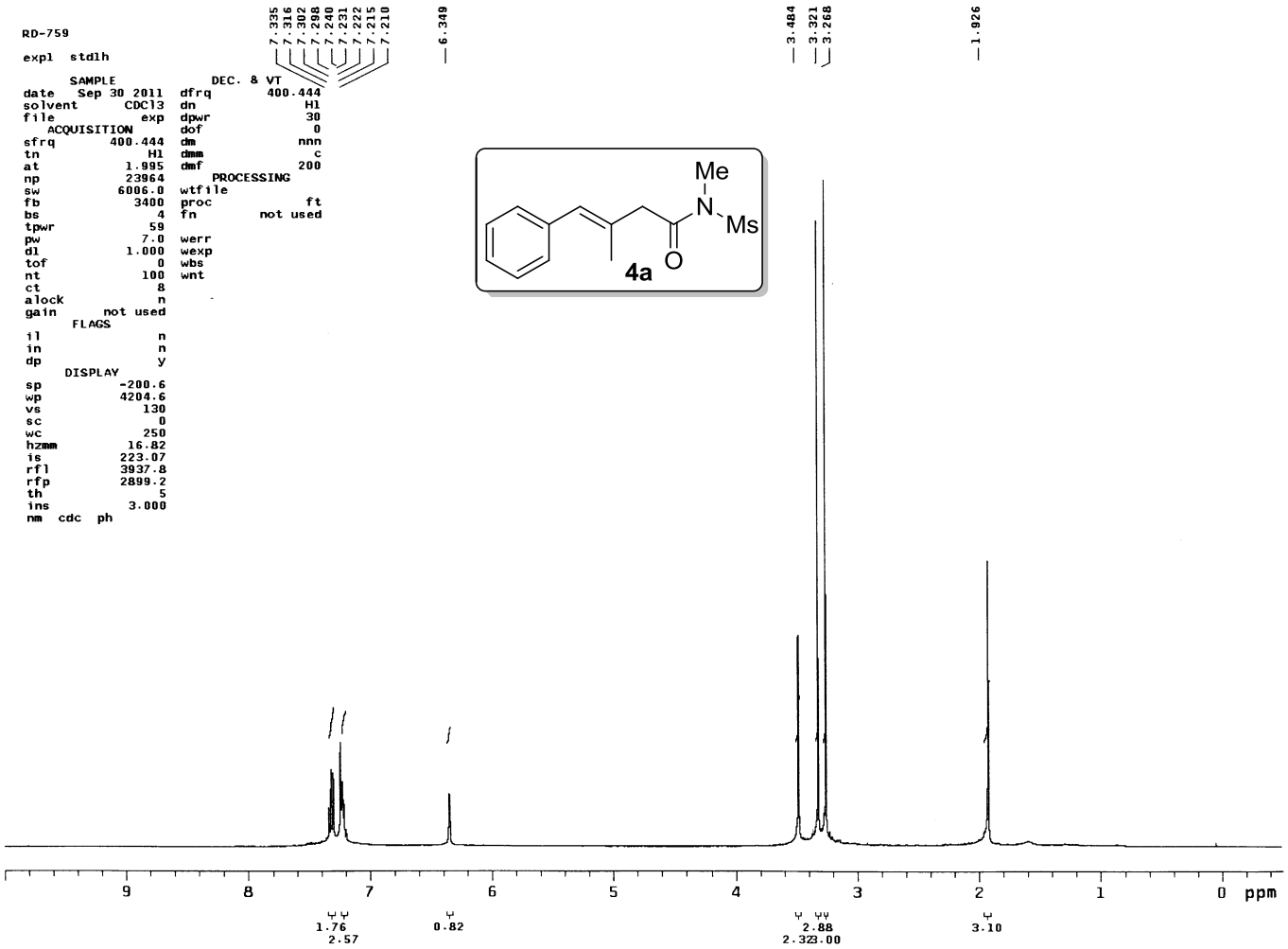
```
Current Data Parameters
NAME            RD-946-P
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120405
Time               14.49
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG            zgpg
TD                32768
SOLVENT          CD2Cl2
NS                  1156
DS                     0
SWH           45045.047 Hz
FIDRES         1.374666 Hz
AQ            0.3637748 sec
RG                  2048
DW                11.100 usec
DE                 6.50 usec
TE                296.4 K
D1           3.50000000 sec
d11          0.03000000 sec
DELTA        3.40000010 sec
MCREST       0.00000000 sec
MCWRK        0.01500000 sec

======== CHANNEL f1 ========
NUC1                13C
P1                 4.80 usec
PL1                0.00 dB
SFO1        150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2         waltz16
NUC2                1H
PCPD2             92.00 usec
PL2              120.00 dB
PL12               9.00 dB
PL13              14.00 dB
SFO2        598.9029945 MHz

F2 - Processing parameters
SI                 65536
SF          150.5935279 MHz
WDW                   EM
SSB                    0
LB                  2.00 Hz
GB                     0
PC                  1.00

1D NMR plot parameters
CX                 20.00 cm
CY                 12.00 cm
F1P               200.000 ppm
F1              30118.71 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1505.93518 Hz/cm
```

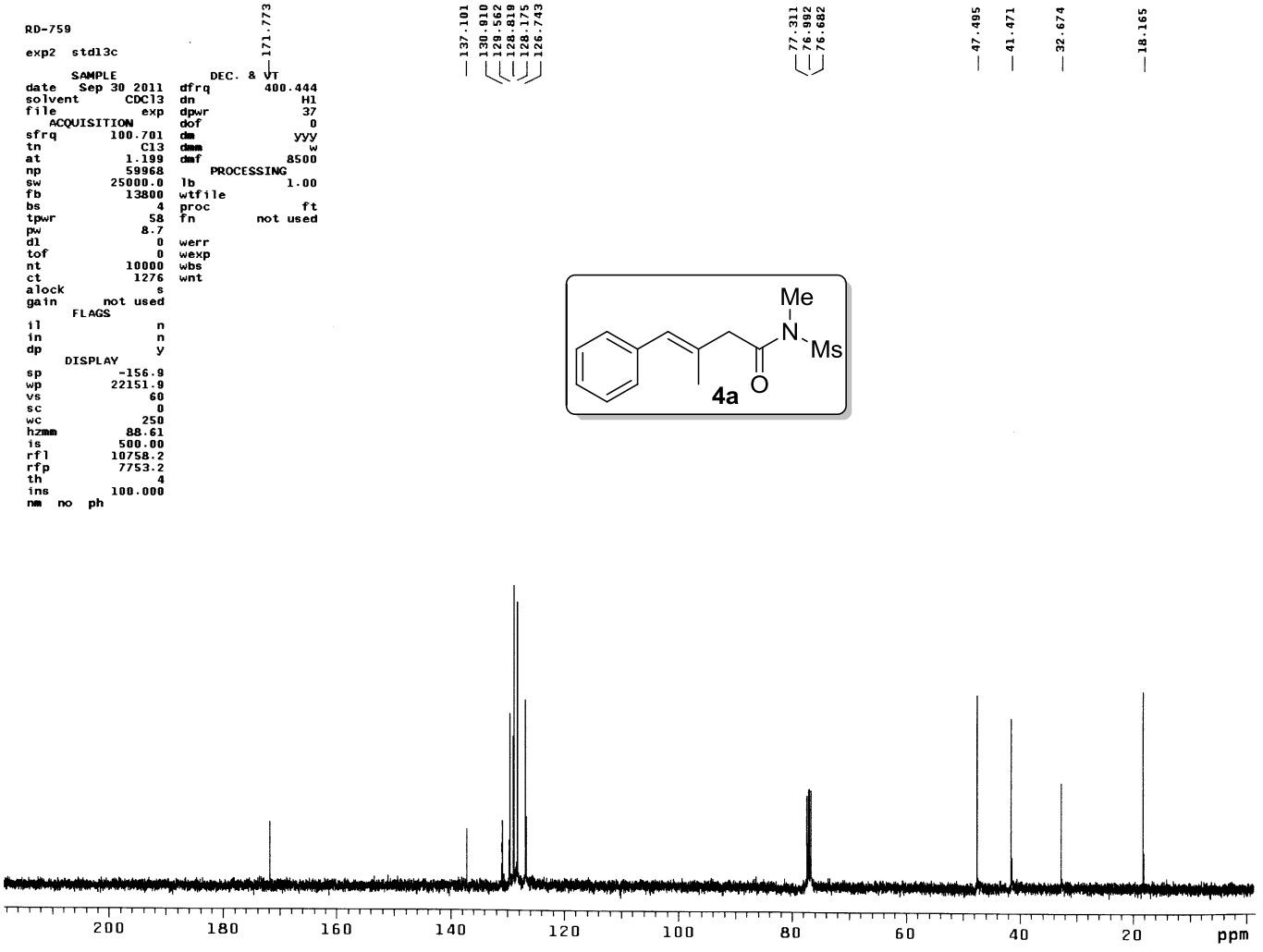


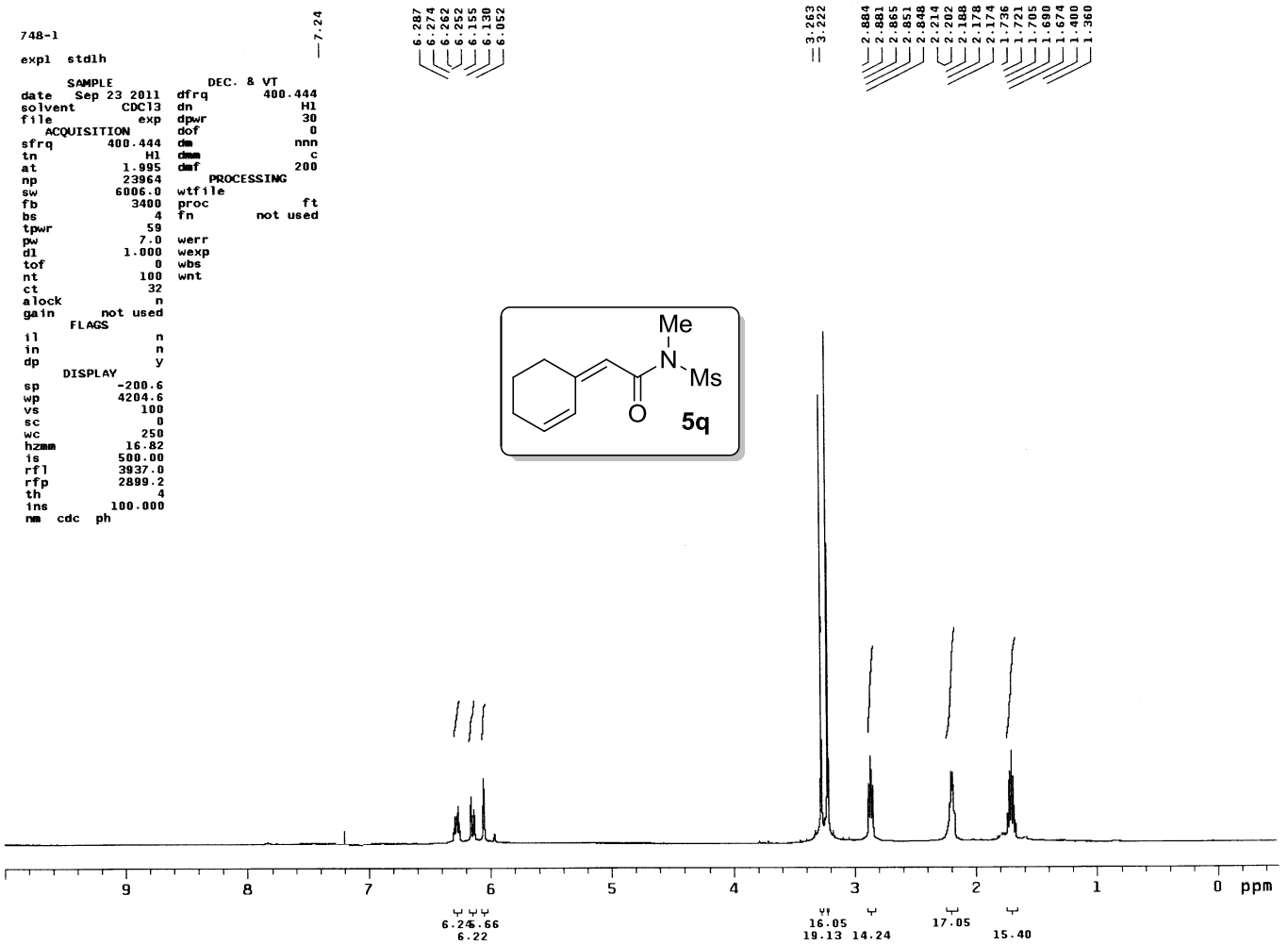
```
Current Data Parameters
NAME                 RD-939
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_             20120320
Time                 18.59
INSTRUM              spect
PROBHD   5 mm QNP  1H/1
PULPROG                 zg
TD                   32768
SOLVENT              CD2Cl2
NS                       16
DS                        0
SWH               8389.262 Hz
FIDRES            0.256020 Hz
AQ                1.9530228 sec
RG                      512
DW                 59.600 usec
DE                  6.50 usec
TE                  295.7 K
D1              1.00000000 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    8.00 usec
PL1                   0.00 dB
SFO1          598.9029945 MHz

F2 - Processing parameters
SI                    16384
SF            598.9000246 MHz
WDW                      no
SSB                       0
LB                    0.00 Hz
GB                        0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
CY                   10.00 cm
F1P                  10.000 ppm
F1                 5989.00 Hz
F2P                 -0.500 ppm
F2                -299.45 Hz
PPMCM              0.52500 ppm/cm
HZCM             314.42252 Hz/cm
```

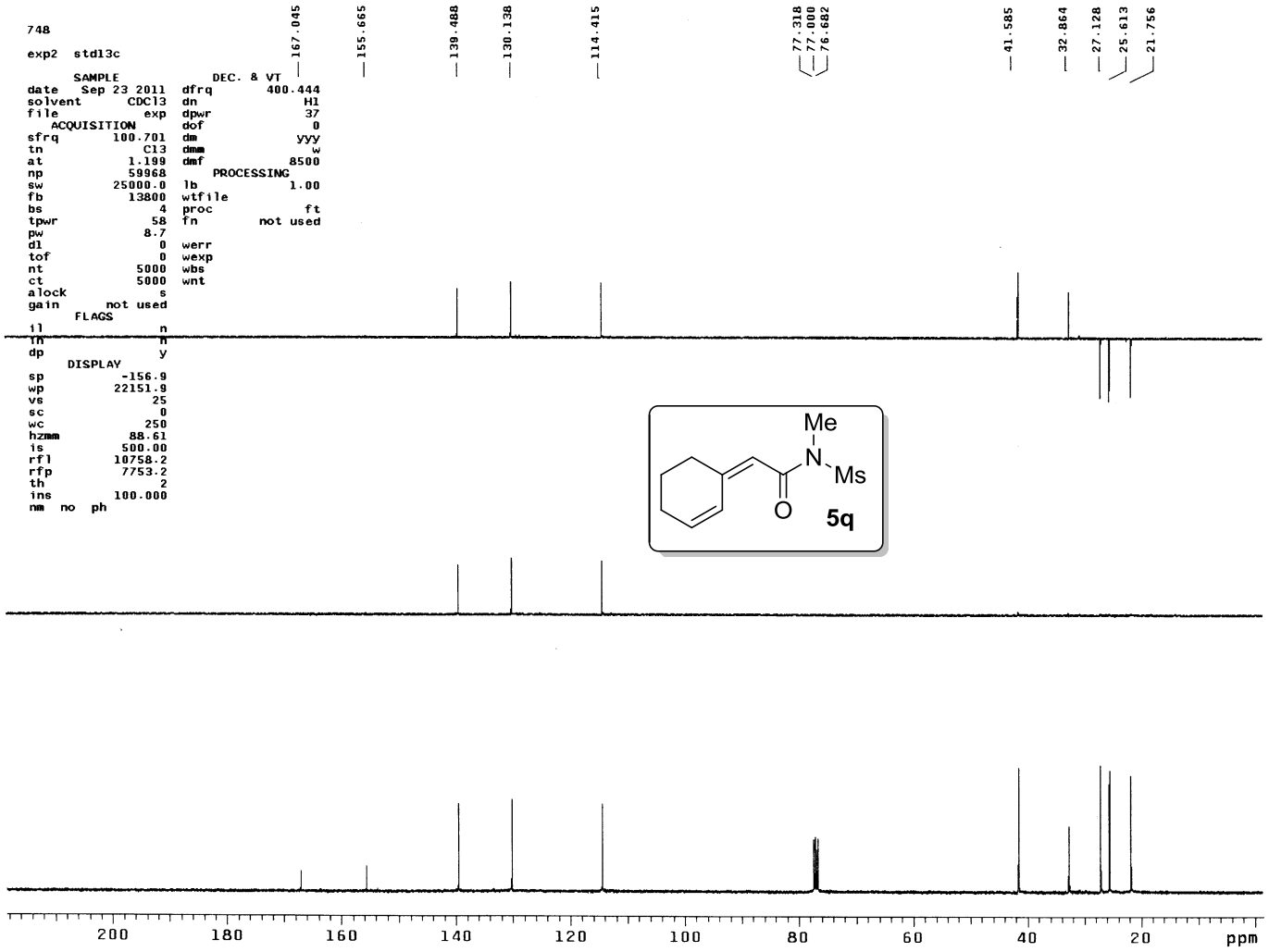




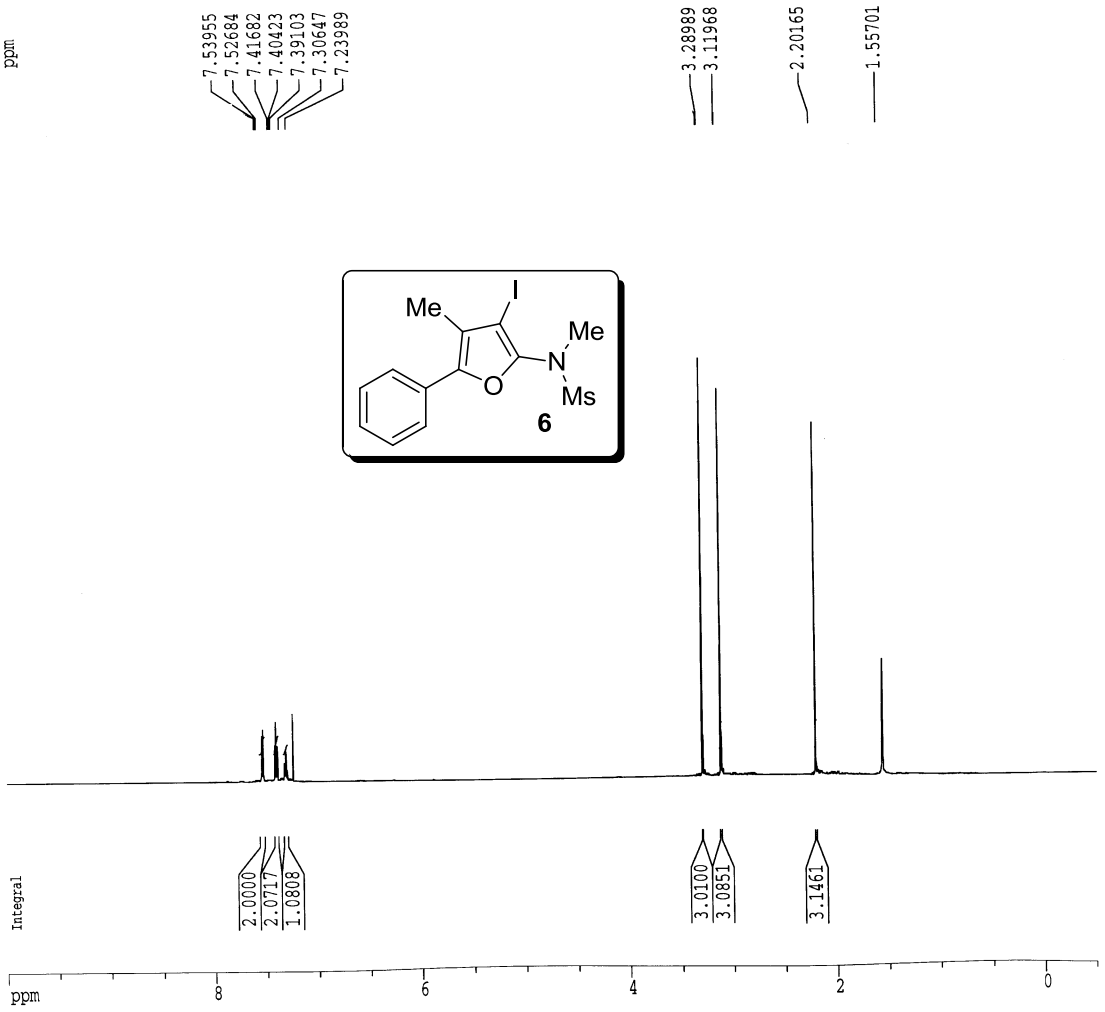



RD-759
exp2 std13c
SAMPLE
date Sep 30 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 10000
ct 1276
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -156.9
wp 22151.9
vs 60
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10758.2
rfp 7753.2
th 4
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
171.773
137.101
130.910
129.562
128.819
128.175
126.743
77.311
76.992
76.682
47.495
41.471
32.674
18.165
Me
N
Ms
O
4a
200
180
160
140
120
100
80
60
40
20
ppm

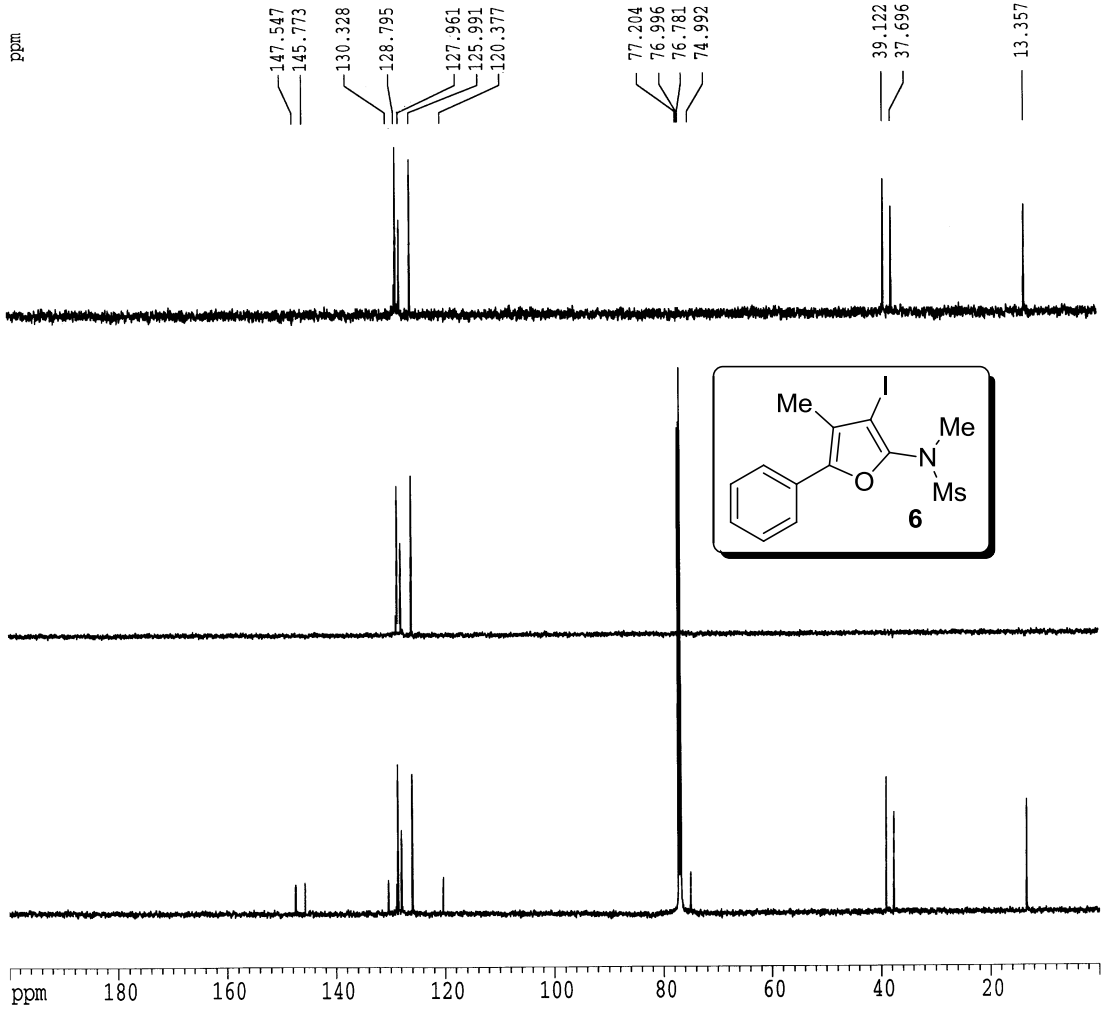



748
exp2 stdl3c
SAMPLE
date Sep 23 2011
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 4
tpwr 58
pw 8.7
dl 0
tof 0
nt 5000
ct 5000
alock s
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -156.9
wp 22151.9
vs 25
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 10758.2
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
167.045
155.665
139.488
130.138
114.415
77.318
77.000
76.682
41.585
32.864
27.128
25.613
21.756
Me
N
Ms
O
5q
200
180
160
140
120
100
80
60
40
20
ppm

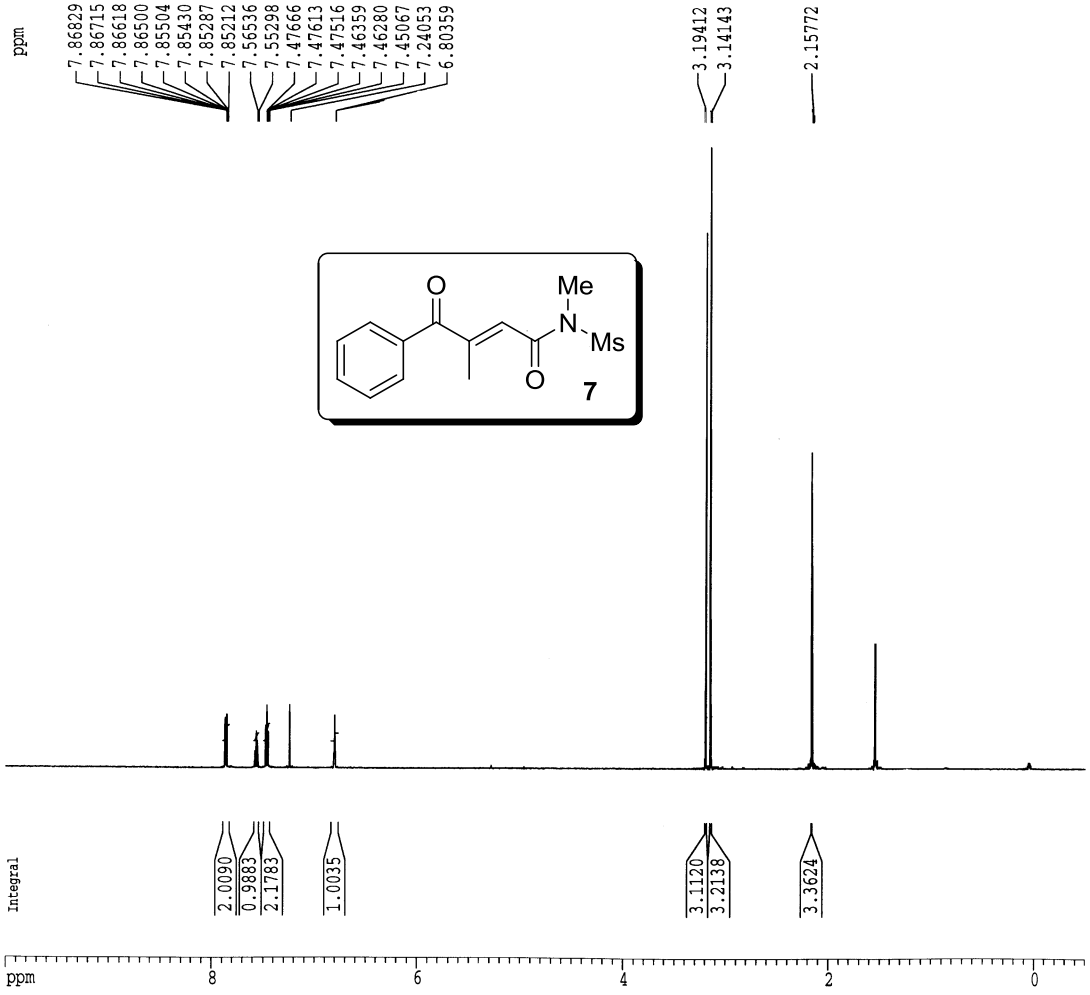
```
Current Data Parameters
NAME            RD-942-A
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20120404
Time               14.47
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG              zg
TD                32768
SOLVENT           CDCl3
NS                   16
DS                    0
SWH            8389.262 Hz
FIDRES         0.256020 Hz
AQ             1.9530228 sec
RG                  512
DW                59.600 usec
DE                 6.50 usec
TE                296.5 K
D1             1.00000000 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 1H
P1                 8.00 usec
PL1                0.00 dB
SFO1         598.9029945 MHz

F2 - Processing parameters
SI                16384
SF          598.9000246 MHz
WDW                  no
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00

1D NMR plot parameters
CX                20.00 cm
CY                 8.00 cm
F1P               10.000 ppm
F1              5989.00 Hz
F2P               -0.500 ppm
F2              -299.45 Hz
PPMCM          0.52500 ppm/cm
HZCM         314.42252 Hz/cm
```

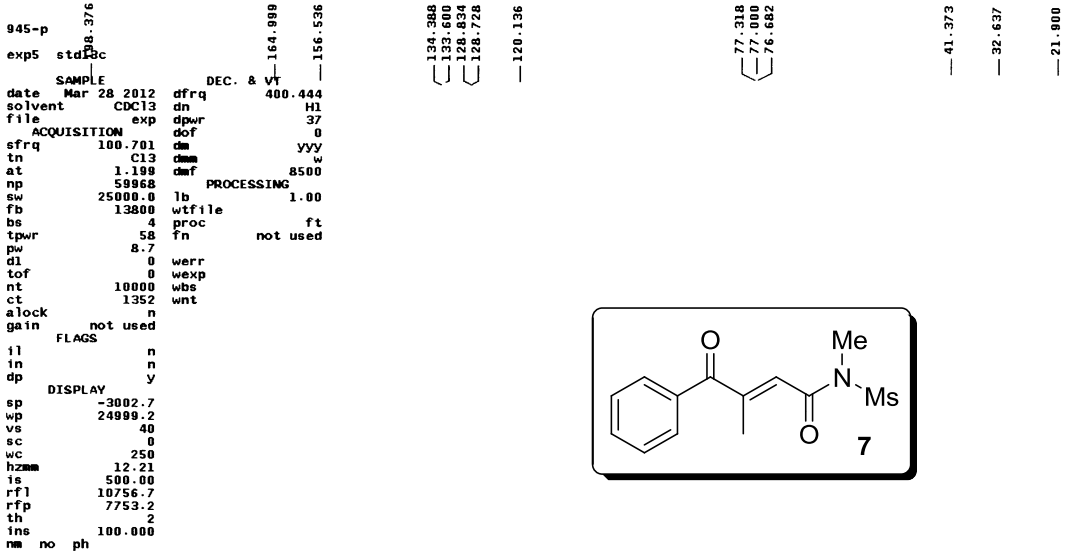


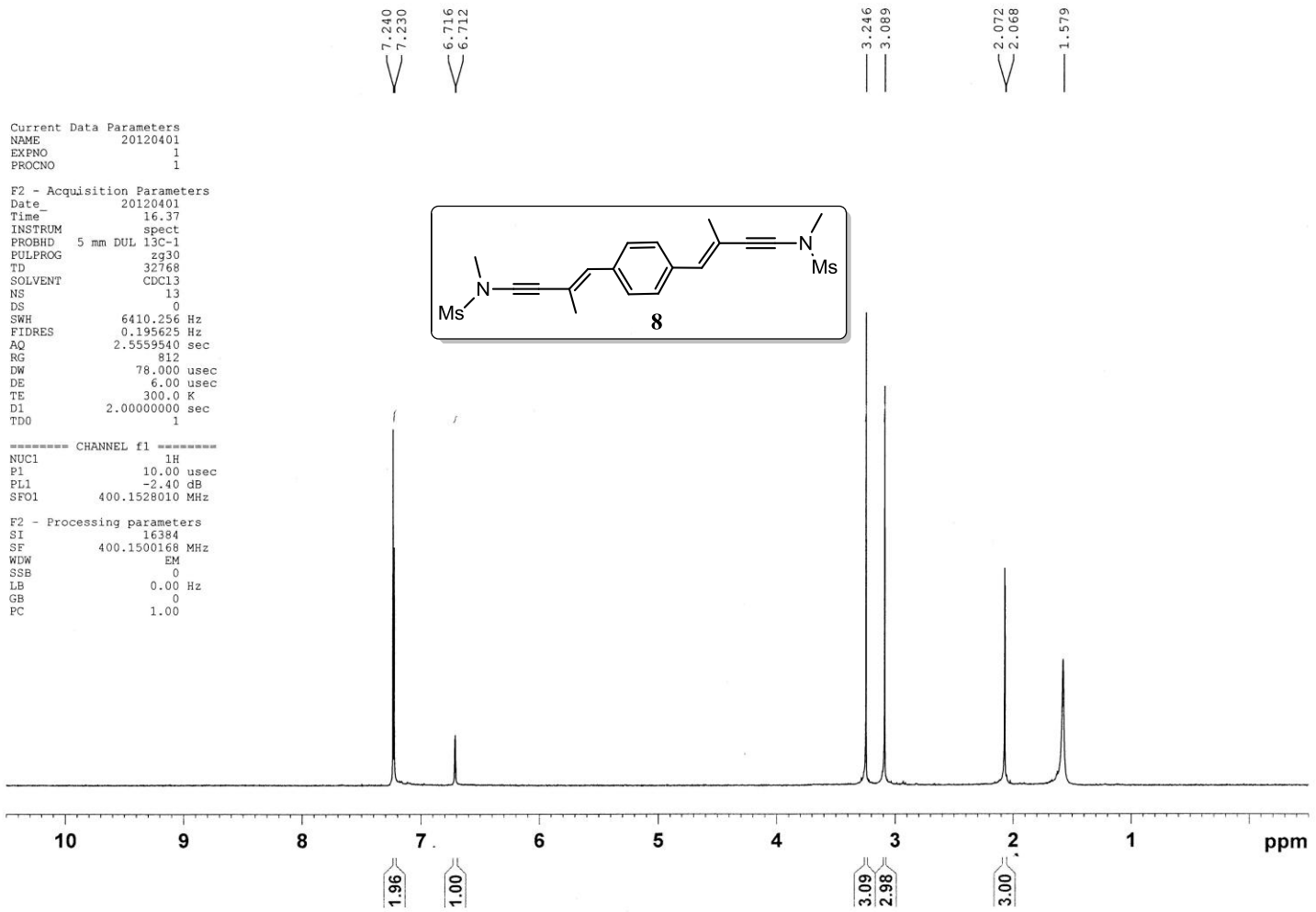
```
Current Data Parameters
NAME                  RD-942-A
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20120404
Time                     14.55
INSTRUM                  spect
PROBHD   5 mm QNP  1H/1
PULPROG                  zgpg
TD                      32768
SOLVENT                 CDCl3
NS                       1645
DS                          0
SWH                 45045.047 Hz
FIDRES               1.374666 Hz
AQ                   0.3637748 sec
RG                        4096
DW                      11.100 usec
DE                       6.50 usec
TE                      296.9 K
D1                  3.50000000 sec
d11                 0.03000000 sec
DELTA               3.40000010 sec
MCREST              0.00000000 sec
MCWRK               0.01500000 sec

======== CHANNEL f1 ========
NUC1                       13C
P1                        4.80 usec
PL1                       0.00 dB
SFO1                150.6100903 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                    92.00 usec
PL2                     120.00 dB
PL12                     11.30 dB
PL13                     14.00 dB
SFO2                598.9029990 MHz

F2 - Processing parameters
SI                        16384
SF                  150.5935293 MHz
WDW                          EM
SSB                           0
LB                        3.00 Hz
GB                            0
PC                         1.00

1D NMR plot parameters
CX                       20.00 cm
CY                       10.00 cm
F1P                     200.000 ppm
F1                    30118.71 Hz
F2P                       0.000 ppm
F2                        0.00 Hz
PPMCM                10.00000 ppm/cm
HZCM                 1505.93530 Hz/cm
```

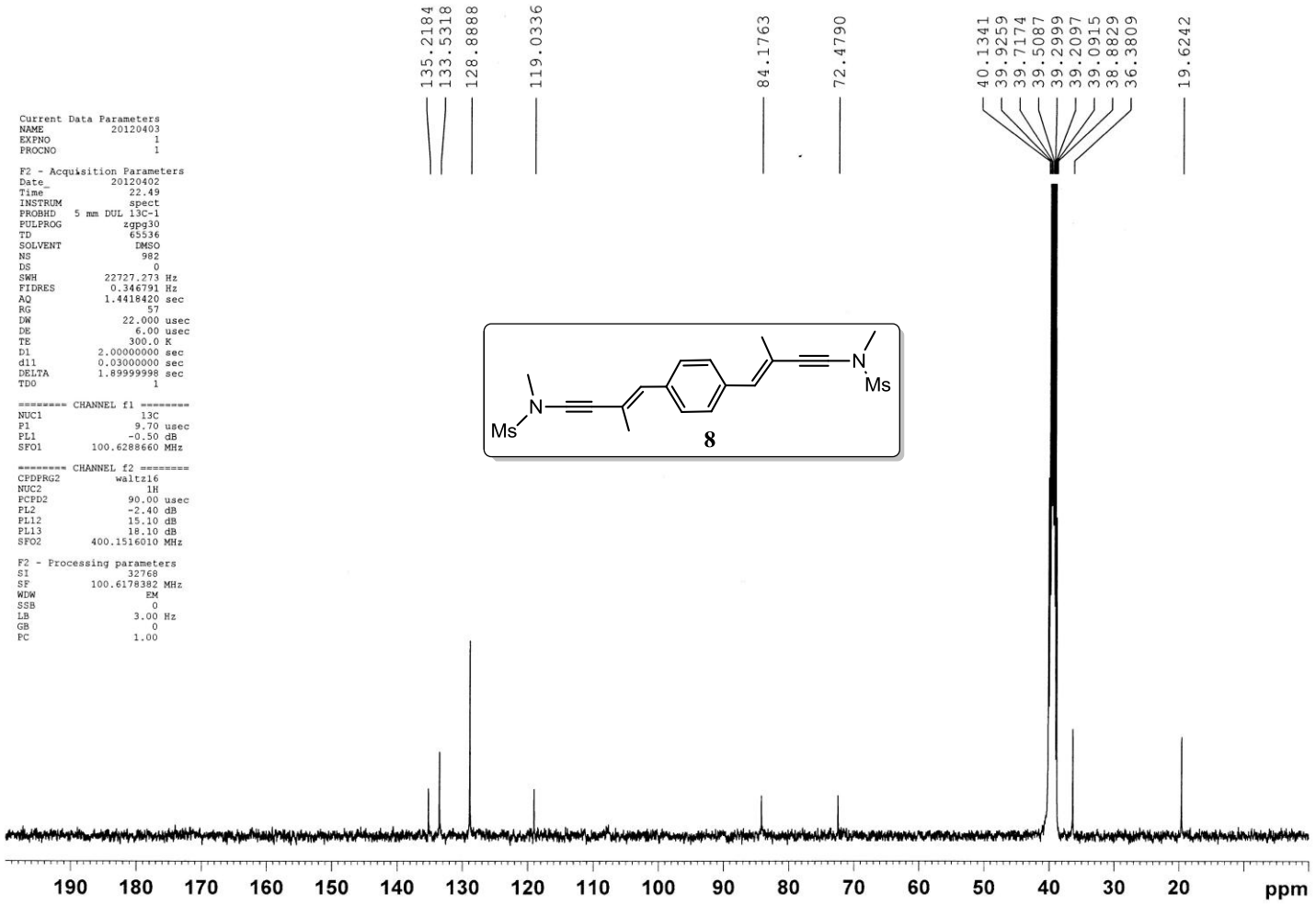


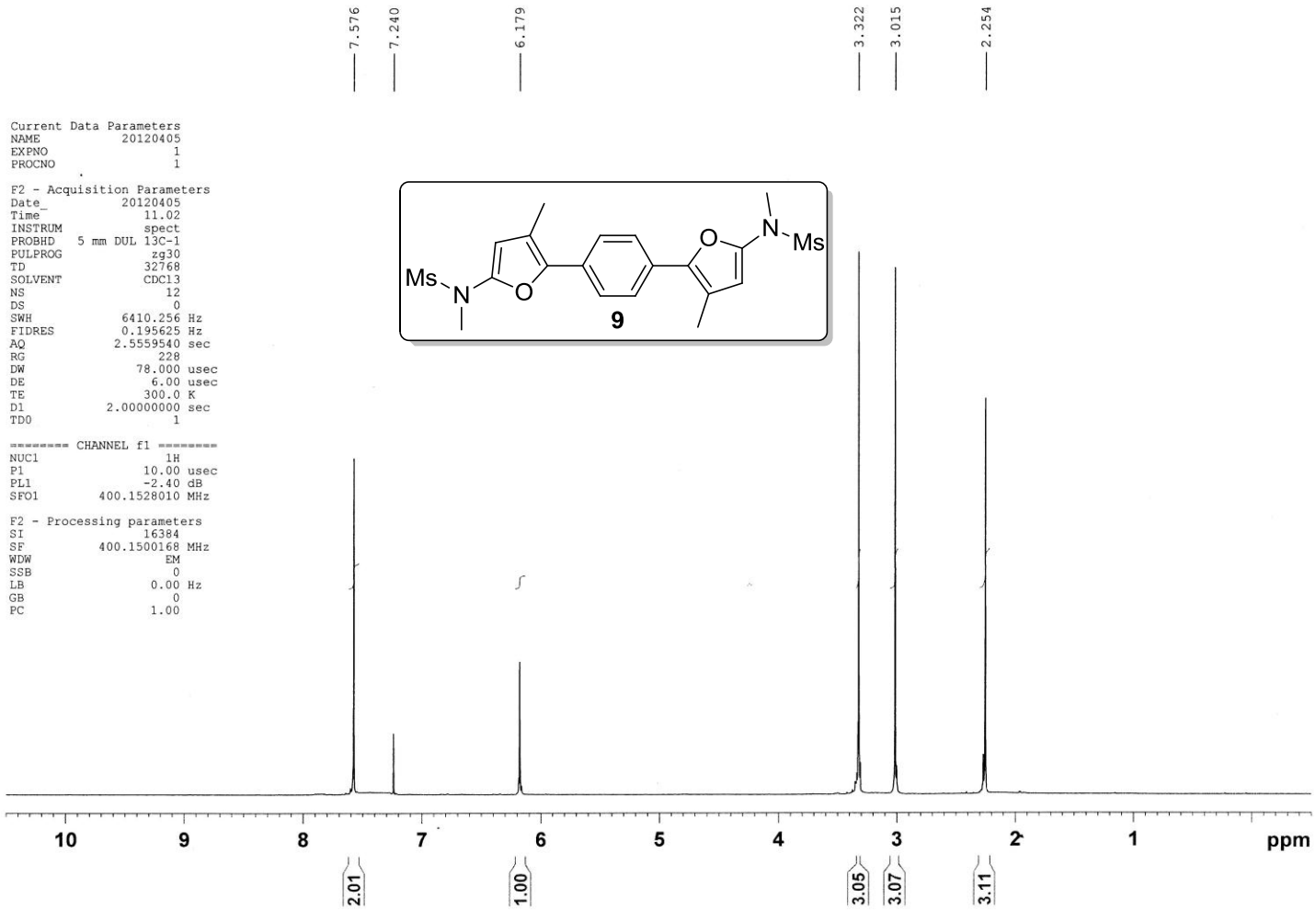
```
Current Data Parameters
NAME                  RD-945-P
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_              20120405
Time                    18.54
INSTRUM                 spect
PROBHD    5 mm QNP  1H/1
PULPROG                    zg
TD                      32768
SOLVENT                 CDCl3
NS                         16
DS                          0
SWH                  8389.262 Hz
FIDRES               0.256020 Hz
AQ                   1.9530228 sec
RG                        128
DW                     59.600 usec
DE                      6.50 usec
TE                     296.3 K
D1                   2.00000000 sec
MCREST               0.00000000 sec
MCWRK                0.01500000 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       9.80 usec
PL1                      0.00 dB
SFO1               598.9029945 MHz

F2 - Processing parameters
SI                      32768
SF                 598.9000248 MHz
WDW                        no
SSB                         0
LB                       0.00 Hz
GB                          0
PC                       1.00

1D NMR plot parameters
CX                      20.00 cm
CY                      12.00 cm
F1P                     10.000 ppm
F1                    5989.00 Hz
F2P                     -0.500 ppm
F2                    -299.45 Hz
PPMCM                  0.52500 ppm/cm
HZCM                 314.42252 Hz/cm
```

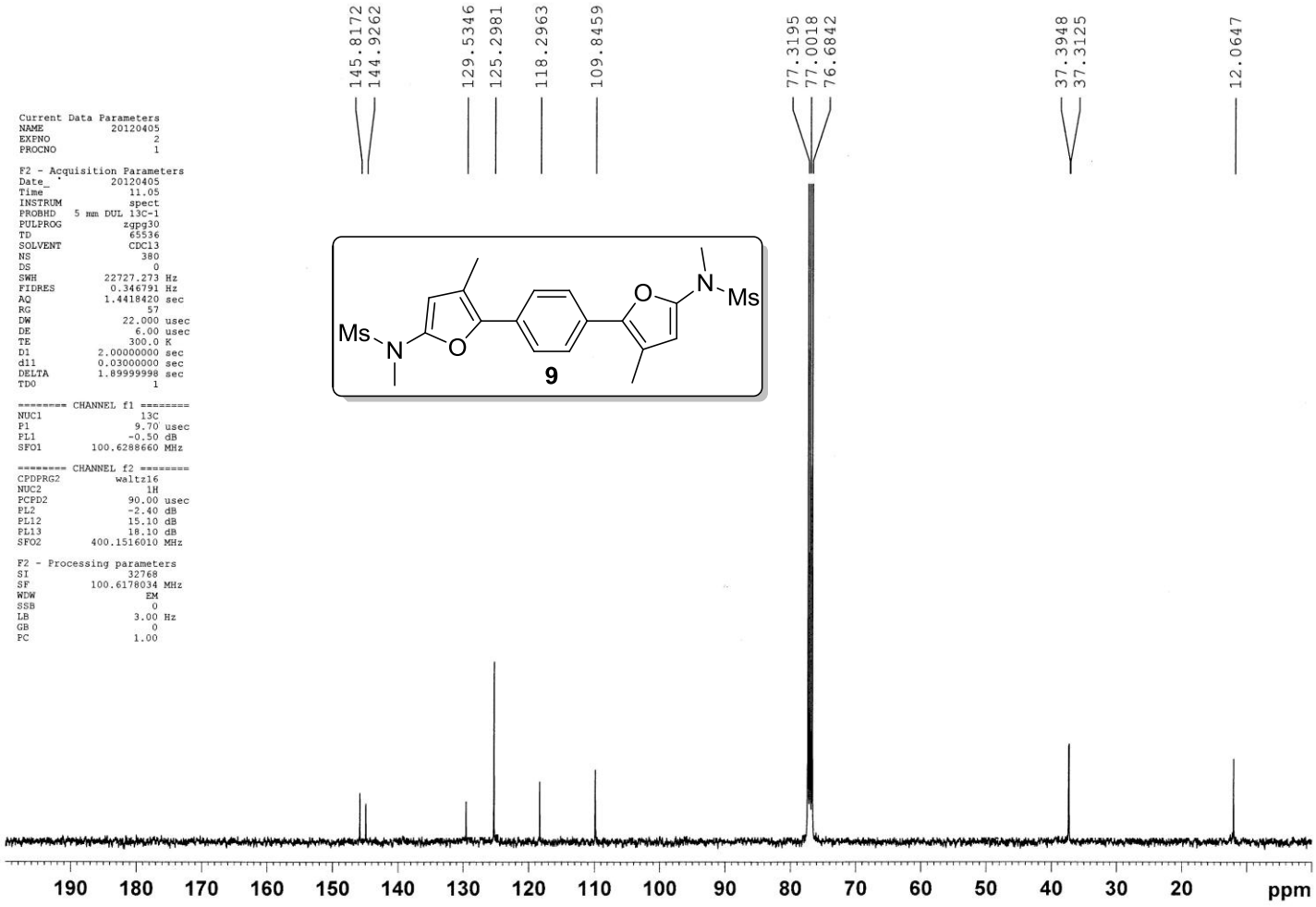



168.376
164.999
156.536
134.388
133.600
128.834
128.728
120.136
77.318
77.000
76.682
41.373
32.637
21.900
945-p
exp5 std13c
SAMPLE
date Mar 28 2012
solvent CDCl3
file exp
ACQUISITION
sfrq 100.701
tn C13
at 1.199
np 59968
sw 25008.0
fb 13800
bs 4
tpwr 58
pw 8.7
d1 0
tof 0
nt 10000
ct 1352
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -3002.7
wp 24999.2
vs 40
sc 0
wc 250
hzmm 12.21
is 500.00
rfl 10756.7
rfp 7753.2
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.444
dn H1
dpwr 37
dof 0
dm yyy
dmm w
dmf 8500
PROCESSING
lb 1.00
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
O
Me
N
Ms
O
7
200
180
160
140
120
100
80
60
40
20
0
ppm










```
Current Data Parameters
NAME                  20120405
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_               20120405
Time                   11.05
INSTRUM                spect
PROBHD    5 mm DUL 13C-1
PULPROG              zgpg30
TD                    65536
SOLVENT               CDCl3
NS                      380
DS                        0
SWH                22727.273 Hz
FIDRES             0.346791 Hz
AQ                 1.4418420 sec
RG                       57
DW                    22.000 usec
DE                     6.00 usec
TE                    300.0 K
D1                 2.00000000 sec
d11                0.03000000 sec
DELTA              1.89999998 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                      13C
P1                       9.70 usec
PL1                     -0.50 dB
SFO1            100.6288660 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                   90.00 usec
PL2                     -2.40 dB
PL12                    15.10 dB
PL13                    18.10 dB
SFO2            400.1516010 MHz

F2 - Processing parameters
SI                        32768
SF              100.6178034 MHz
WDW                          EM
SSB                           0
LB                       3.00 Hz
GB                            0
PC                       1.00
```