Supplementary Material (ESI) for Chemical Communications

Supporting Information

Platinum-Catalyzed Cycloisomerization of 1,4-Enynes *via* Activation of a sp^3-Hybridized C-H Bond

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(I) Representative Synthetic procedures:

(a) General procedures:

Unless otherwise noted, all the reactions for the preparation of the substrate were performed in oven-dried glassware under nitrogen atmosphere with freshly distilled solvents. The catalytic reaction were performed under carbon monoxide atmosphere. Toluene, *p*-Xylene, DMF and dichloromethane were distilled from CaH_2 under nitrogen. *N*,*N*-dimethyl formamide (DMF) and triethylamine (Et_3N) were stored over 4Å molecular sieves prior to use. All other commercial reagents were used without further purification, unless otherwise indicated. ^1H NMR and ^{13}C NMR spectra were recorded on a Varian 400 MHz, Bruker 400 and a Bruker 600 MHz spectrometers using chloroform-*d* ($CDCl_3$) as the internal standard.

(b) Typical procedure for the synthesis of ((1-(cyclopentylidenemethyl)cyclopropyl)ethynyl)benzene (1a).

The compound (**s-1)** was prepared from cyclopropylacetylene according to the reported procedure.[1]

(c) Synthesis of tert-butyl((1-(cyclopentylidenemethyl)cyclopropyl)ethynyl) dimethylsilane (s-2).

A THF solution (30mL) of cylopentyltriphenylphosphinebromide (4g, 9.72 mmol) was cooled to 0^oC, and to this solution was added n-BuLi (2.85mL, 2.5 M in hexane, 7.13 mmol). The solution was stirred for 0.5 h at 0^oC, and to this mixture was added compound **s-1** (1.35g, 6.48 mmol) at the same temperature and continued to stir for 2 h at RT. The reaction was quenched with water, extracted with diethyl ether (3x15 ml) and washed with brine solution, dried over $MgSO_4$, and concentrated under reduced pressure. The residue was eluted through a silica column to afford compound **s-2** (1.36 g, 80%) as colorless oil.

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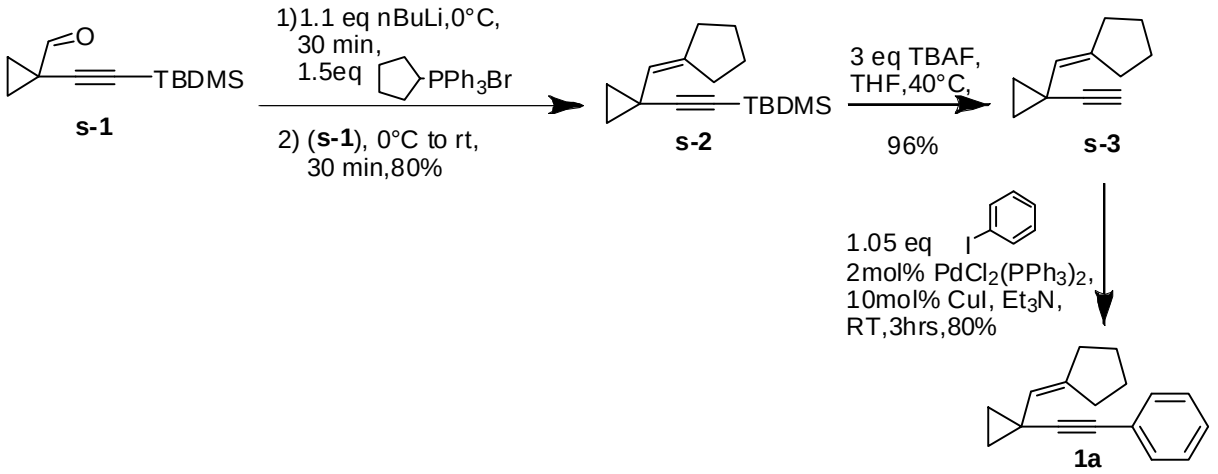
[1] Guotao Li, Xiaogen Huang, and Liming Zhang, *J. Am. Chem. Soc.* **2008,** *130,* 6944.

Scheme-S1



(d) Synthesis of ((1-ethynylcyclopropyl)methylene)cyclopentane (s-3).

To a stirred THF solution (30mL) of compound **s-2** (1.1g, 4.22mmol) was added TBAF(12.7mL, 1.0M in THF) and the resulting solution was heated at 40^{o}C for 5h. After completion of the reaction, the mixture was quenched with aq. NH_4Cl, and extracted with Et_2O (3x10 ml). The combined organic layer was dried over $MgSO_4$, and concentrated under reduced pressure. The residue was eluted through a short bed of silica column to obtain compound **s-3** (0.595 g, 96%) as colorless oil.

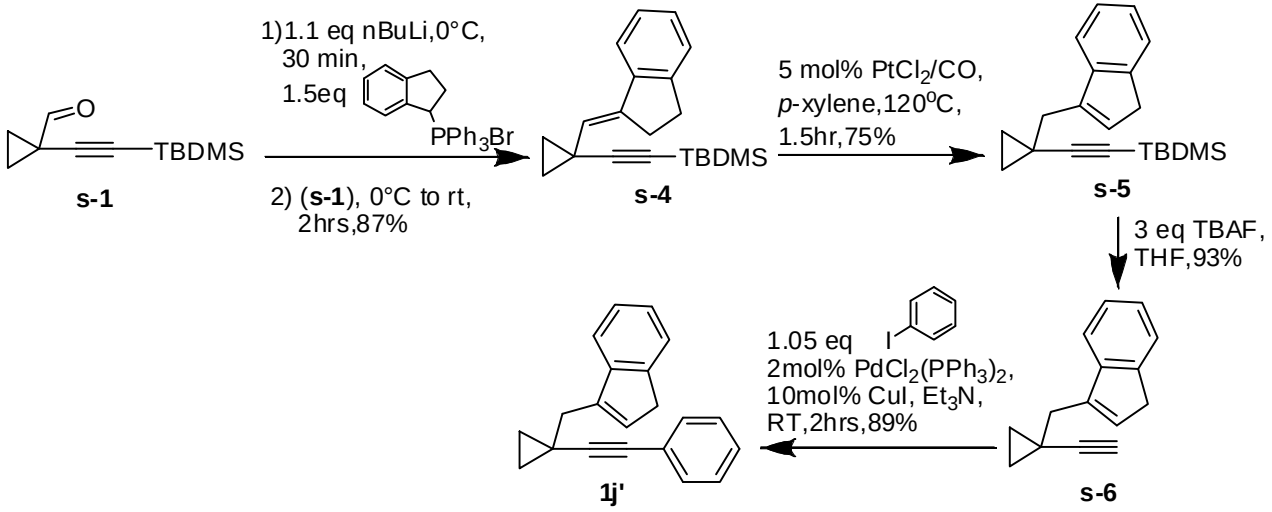
(e) Synthesis of ((1-(cyclopentylidenemethyl)cyclopropyl)ethynyl)benzene (1a).

To a triethylamine solution (10 mL) of $Pd(PPh_3)_2Cl_2$ (19.2 mg, 0.027 mmol) and CuI (26.0 mg, 0.137 mmol) was added iodobenzene (0.293 g, 1.44 mmol) and stir for 5 min, then add compound **s-3** (0.2 g, 1.37 mmol), dropwise and allowed to stir for 3hrs at RT. After completion of the reaction, the mixture 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 **1a** (0.242 g, 80%) as a pale yellow oil.

(f) Synthesis of 3-((1-(phenylethynyl)cyclopropyl)methyl)-1*H*-indene (1j').

The Synthesis of compound (**1j'**) was followed the same procedure as described in scheme-S1.

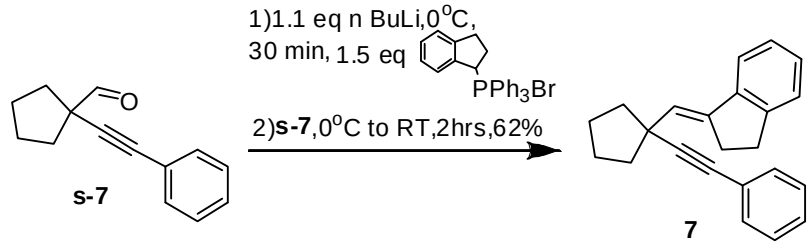
Scheme-S2



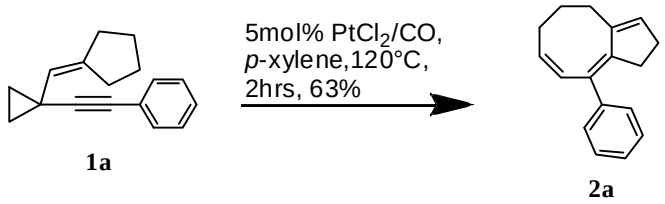
(g) Synthesis of (*E*)-1-((1-(phenylethynyl)cyclopentyl)methylene)-2,3-dihydro-1*H*-indene (7).

The compound (**s-7)** was prepared according to the known literature procedure.[2] The transformation s-7 to 7 was performed according to Scheme-S1.

Scheme-3



(II) Standard procedure for catalytic operation by $PtCl_2$.



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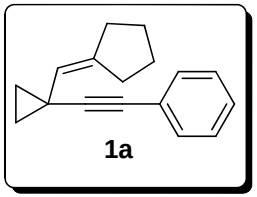
[2] Chun-Yao Yang, Guan-You Lin, Hsin-Yi Liao, Swarup Datta, Rai-Shung Liu, *J. Org. Chem.* **2008**, *73*, 4907.

A long tube containing $PtCl_2$ (5.98 mg, 0.023 mmol) was dried in vacuum for 1h and vacuum was released with CO gas using CO balloon before it was charged with substrate(**1a**) (100 mg, 0.45 mmol) and *p*-xylene (0.9 ml). The mixture was heated 120^{o}C for 2h. The solution was concentrated and eluted through a silica column (hexane) to afford compound (**2a)** (62 mg, 62%) as a pale yellow oil.

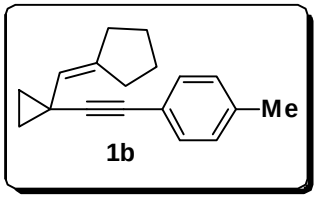
(III) Spectral data of Compounds 1a to 8

Spectral data for ((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)benzene (1a).



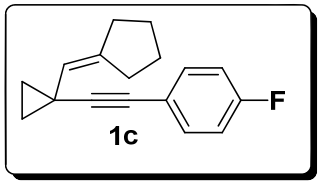
Pale yellow oil, IR (neat, cm^{-1}): 3089 (s), 2935 (w), 2240 (w), 1490 (w), 1441 (s), 889 (s), 749 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.42 ~7.38 (m, 2 H), 7.29 ~ 7.22 (m, 3 H), 5.15 (t, *J* = 2.1 Hz, 1 H), 2.65 ~ 2.61 (m, 2H), 2.31 ~ 2.27 (m, 2 H), 1.78 ~ 1.71 (m, 2H), 1.66 ~ 1.59 (m, 2 H), 1.25 ~ 1.22 (m, 2 H), 0.93 ~ 0.90 (m, 2 H); ^{13}C NMR (100MHz, $CDCl_3$): δ 147.6, 131.5, 128.1, 127.2, 124.1, 120.5, 94.9, 75.9, 34.3, 29.5, 26.7, 26.1, 18.7, 11.9; HRMS calcd for $C_{17}H_{18}$: 222.1409, found: 222.1401.

Spectral data for 1-((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)-4-methylbenzene (1b).



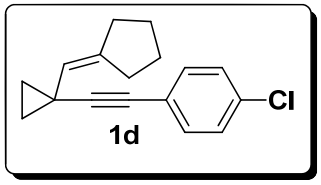
Colorless oil, IR (neat, cm^{-1}): 3092 (s), 2985 (w), 2232 (w), 1432 (s), 872 (s), 732 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.25 (d, J = 8.0 Hz, 2 H), 7.05 (d, J = 8.0 Hz, 2 H), 5.10 (s, 1 H), 2.58 (t, J = 7.2 Hz, 2 H), 2.30 (s, 3 H), 2.24 (t, J = 6.6 Hz, 2 H), 1.71 ~ 1.54 (m, 4 H), 1.19 ~ 1.16 (m, 2 H), 0.88 ~ 0.85 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.5, 137.2, 131.4, 128.8, 121.1, 120.6, 94.0, 75.9, 34.3, 29.4, 26.8, 26.1, 21.3, 18.6, 11.9; HRMS calcd for $C_{18}H_{20}$: 236.1565, found: 236.1561.

Spectral data for 1-((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)-4-fluorobenzene (1c).



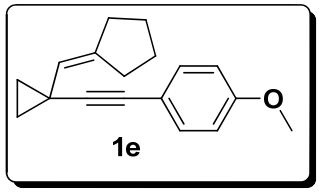
Colorless oil, IR (neat, cm^{-1}): 3102 (s), 2978 (w), 2260 (w), 1438 (s), 904 (s), 793 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.34 ~ 7.31 (m, 2 H), 6.95 ~ 6.91 (m, 2 H), 5.11 (s, 1 H), 2.56 (t, J = 6.8 Hz, 2 H), 2.24 (t, J = 6.6 Hz, 2 H), 1.73 ~ 1.66 (m, 2 H), 1.61 ~ 1.54 (m, 2 H), 1.24 ~ 1.12 (m, 2 H), 0.92 ~ 0.78 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 161.9 (d, J = 246.0 Hz), 147.9, 133.3 (d, 8.0 Hz), 120.4, 120.2 (d, J = 2.9 Hz), 115.3 (d, J = 21.9 Hz), 94.5, 74.7, 34.3, 29.5, 26.8, 26.1, 18.6, 11.8; HRMS calcd for $C_{17}H_{17}F$: 240.1314, found: 240.1311.

Spectral data for 1-chloro-4-((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)benzene (1d).



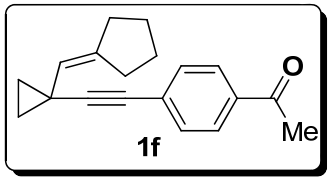
Colorless solid, IR (neat, cm^{-1}): 3092 (s), 2971 (w), 2249 (w), 1426 (s), 884 (s), 783 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.27 (d, *J* = 8.4 Hz, 2 H), 7.20 (d, *J* = 8.4 Hz, 2 H), 5.10 (s, 1 H), 2.54 (t, *J* = 7.0 Hz, 2 H), 2.23 (t, *J* = 6.6 Hz, 2 H), 1.71 ~ 1.66 (m, 2 H), 1.61 ~ 1.54 (m, 2 H), 1.19 ~ 1.16 (m, 2 H), 0.89 ~ 0.86 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.0, 133.2, 132.7, 128.4, 122.7, 120.2, 96.1, 74.8, 34.3, 29.5, 26.7, 26.1, 18.7, 11.9; HRMS calcd for $C_{17}H_{17}Cl$: 256.1019, found: 256.1014.

Spectral data for 1-((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)-4-methoxybenzene (1e).



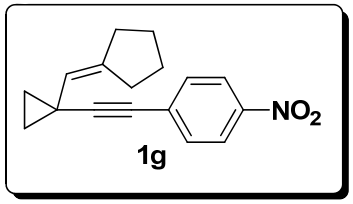
Yellow oil, IR (neat, cm^{-1}): 3072 (s), 2968 (w), 2224 (w), 1455 (w), 1410 (s), 854 (s), 763 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.29 (d, *J* = 8.8 Hz, 2 H), 6.77 (d, *J* = 8.8 Hz, 2 H), 5.10 (s, 1 H), 3.80 (s, 3 H), 2.57 (t, *J* = 7.2 Hz, 2 H), 2.23 (t, *J* = 6.4 Hz, 2 H), 1.72 ~ 1.65 (m, 2 H), 1.60 ~ 1.54 (m, 2 H), 1.17 ~ 1.15 (m, 2 H), 0.86 ~ 0.84 (m, 2H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 158.8, 147.6, 132.9, 120.7, 116.3, 113.7, 93.2, 75.6, 55.2, 34.4, 29.4, 26.8, 26.1, 18.6, 11.9; HRMS calcd for $C_{18}H_{20}O$: 252.1514, found: 252.1510.

Spectral data for 1-(4-((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)phenyl)ethanone (1f).



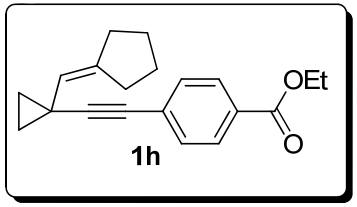
Pale yellow oil, IR (neat, cm^{-1}): 3089 (s), 2978 (w), 2244 (w),1685 (s), 1432 (s), 902 (s), 783 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.81 (d, *J* = 7.6 Hz, 2 H), 7.39 (d, *J* = 7.6 Hz, 2 H), 5.10 (s, 1 H), 2.54 ~ 2.49 (m, 5 H), 2.22 (t, *J* = 7.0 Hz, 2 H), 1.70 ~ 1.66 (m, 2 H), 1.58 ~ 1.54 (m, 2 H), 1.21 ~ 1.18 (m, 2 H), 0.91 ~ 0.89 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.2, 148.1, 135.3, 131.4, 129.2, 128.0, 119.9, 99.0, 75.3, 34.2, 29.5, 26.6, 26.4, 26.0, 18.8, 11.9; HRMS calcd for $C_{19}H_{20}O$: 264.1514, found: 264.1511.

Spectral data for 1-((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)-4-nitrobenzene (1f).



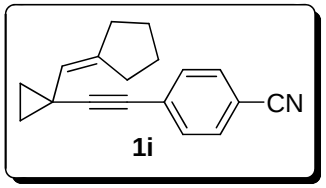
Brown oil, IR (neat, cm^{-1}): 3093 (s), 2984 (w), 2254 (w), 1651 (s), 1438 (s), 902 (s), 796 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 8.10 (d, *J* = 8.8 Hz, 2 H), 7.45 (d, *J* = 8.8 Hz, 2 H), 5.13 (d, *J* = 2.0 Hz, 1 H), 2.52 (t, *J* = 7.2 Hz, 2 H), 2.24 (t, *J* = 6.4 Hz, 2 H), 1.74 ~ 1.67 (m, 2 H), 1.62 ~ 1.55 (m, 2 H), 1.24 ~ 1.22 (m, 2 H), 0.95 ~ 0.93 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.9, 146.6, 132.3, 131.6, 123.7, 119.9, 101.9, 74.9, 34.5, 29.9, 27.0, 26.3, 19.3, 12.3; HRMS calcd for $C_{17}H_{17}NO_2$: 267.1259, found: 267.1252.

Spectral data for ethyl 4-((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)benzoate (1h).



Yellow viscous oil, IR (neat, cm^{-1}): 3098 (s), 2975 (w), 2238 (w), 1726 (s), 1429 (s), 898 (s), 793 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.4 Hz, 2 H), 7.38 (d, *J* = 8.4 Hz, 2 H), 5.12 (s, 1 H), 4.33 (q, *J* = 7.0 Hz, 2 H), 2.55 (t, *J* = 7.2 Hz, 2 H), 2.24 (t, *J* = 7.2 Hz, 2 H), 1.73 ~ 1.66 (m, 2 H), 1.61 ~ 1.54 (m, 2 H), 1.36 (t, *J* = 7.2 Hz, 3 H), 1.26 ~ 1.15 (m, 2 H), 0.95 ~ 0.84 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.5, 148.5, 131.6, 129.5, 129.2, 129.1, 120.3, 98.8, 75.7, 61.2, 34.5, 29.8, 27.0, 26.3, 19.1, 14.5, 12.2; HRMS calcd for $C_{20}H_{22}O_2$: 294.1620, found: 294.1618.

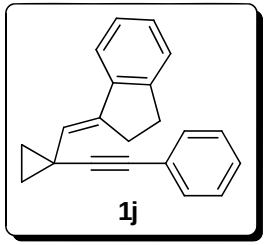
Spectral data for 4-((1-cyclopentylidenemethyl)cyclopropyl)ethynyl)benzonitrile (1i).



1i

Pale yellow oil, IR (neat, cm^{-1}): 3102 (s), 2985 (w), 2258 (m), 2231 (w), 1429 (s), 898 (s), 793 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.51 (d, *J* = 8.0 Hz, 2 H), 7.40 (d, *J* = 8.0 Hz, 2 H), 5.11 (s, 1 H), 2.51 (t, *J* = 6.4 Hz, 2 H), 2.23 (t, *J* = 6.6 Hz, 2 H), 1.72 ~ 1.66 (m, 2 H), 1.62 ~ 1.54 (m, 2 H), 1.21 ~ 1.19 (m, 2 H), 0.93 ~ 0.92 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 148.5, 131.9, 131.8, 129.2, 119.7, 118.6, 110.4, 100.4, 74.6, 34.2, 29.5, 26.7, 26.0, 18.9, 11.9; HRMS calcd for $C_{18}H_{17}N$: 247.1361, found: 247.1358.

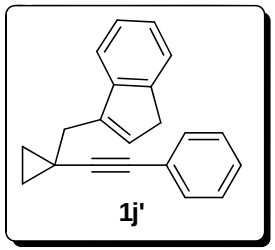
Spectral data for (*E*)-1-((1-(phenylethynyl)cyclopropyl)methylene)-2,3-dihydro-1*H*-indene (1j):



1j

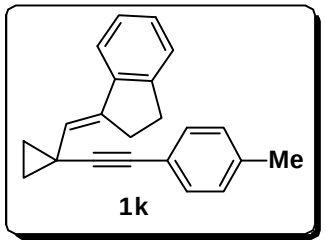
Yellow liquid ; IR (neat, cm^{-1}): 3035 (s), 2985 (w), 2105 (w), 1421 (s), 890 (s), 753 (s); ^1H NMR (400 MHz , $CDCl_3$): δ 7.46 ~ 7.44 (m, 3 H), 7.32 ~ 7.28 (m, 4 H), 7.23 ~ 7.21 (m, 2 H), 5.77 (t, *J* = 2.5 Hz, 1 H), 3.24 ~ 3.20 (m, 2 H), 3.10 ~ 3.07 (m, 2 H), 1.43 ~ 1.40 (m, 2 H), 1.12 ~ 1.10 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 145.5, 141.6, 131.5, 128.1, 127.8, 127.4, 126.3, 125.2, 123.9, 119.8, 119.6, 94.2, 76.5, 30.5, 28.5, 19.3, 12.1; HRMS calcd for $C_{21}H_{18}$: 270.1409; found: 270.1412.

Spectral data for 3-((1-(phenylethynyl)cyclopropyl)methyl)-1*H*-indene (1j').



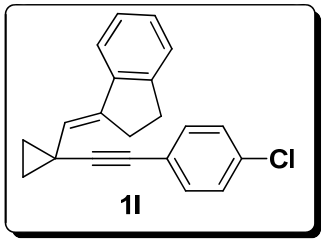
Yellow oil, IR (neat, cm^{-1}): 3055 (s), 2981(w), 2125 (w), 1432 (s), 870 (s), 683 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.52 (d, *J* = 7.2 Hz, 1 H), 7.46 (d, *J* = 7.2 Hz, 1 H), 7.38 ~ 7.34 (m, 3 H), 7.31 ~ 7.26 (m, 4 H), 6.65 (s, 1 H), 3.45 (s, 2 H), 2.82 (s, 2 H), 1.19 ~ 1.17 (m, 2 H), 0.93 ~ 0.90 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.8, 144.4, 141.6, 131.6, 130.3, 128.2, 127.5, 126.1, 124.6, 124.1, 123.8, 119.1, 95.6, 77.5, 38.1, 35.7, 16.0, 11.5; HRMS calcd for $C_{21}H_{18}$: 270.1409, found: 270.1404.

Spectral data for (*E*)-1-((1-(p-tolylethynyl)cyclopropyl)methylene)-2,3-dihydro-1*H*-indene (1k):



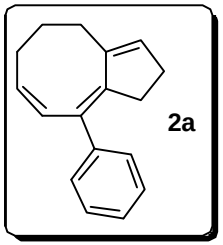
Yellow liquid ; IR (neat, cm^{-1}): 3035 (s), 2105 (w), 2122 (w), 1422 (s), 778 (s), 676 (s); ^1H NMR (400 MHz , $CDCl_3$): δ 7.37 (d, *J* = 7.1 Hz, 1 H), 7.28 ~ 7.20 (m, 3 H), 7.16 ~ 7.14 (m, 2 H), 7.06 (d, *J* = 7.8 Hz, 2 H), 5.69 (s, 1 H), 3.14 ~ 3.12 (m, 2 H), 3.03 ~ 3.00 (m, 2 H), 2.31 (s, 3 H), 1.34 ~ 1.32 (m, 2 H), 1.05 ~ 1.02 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 145.4, 141.7, 137.4, 131.4, 128.9, 127.7, 126.3, 125.2, 120.9, 119.8, 119.7, 93.3, 76.5, 30.5, 28.5, 21.3, 19.3, 12.2; HRMS calcd for $C_{22}H_{20}$: 284.1565; found: 284.1568.

Spectral data for (*E*)-1-((1-((4-chlorophenyl)ethynyl)cyclopropyl)methylene)-2,3-dihydro-1*H*-indene (1l):



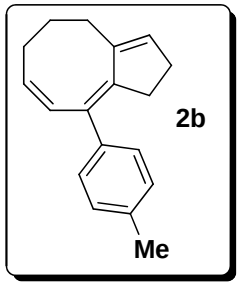
Yellow liquid ; IR (neat, cm^{-1}): 3102 (s), 2982 (w), 2232 (w), 1424 (s), 998 (s), 756 (s); ^1H NMR (400 MHz , $CDCl_3$): δ 7.41 ~ 7.40 (m, 1 H), 7.34 ~ 7.31 (m, 2 H), 7.27 ~ 7.24 (m, 3 H), 7.21 ~ 7.18 (m, 2 H), 5.74 (d, *J* = 2.5 Hz, 1 H), 3.17 ~ 3.13 (m, 2 H), 3.07 ~ 3.03 (m, 2 H), 1.37 (t, *J* = 3.1 Hz, 2 H), 1.08 (t, *J* = 3.1 Hz, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.7, 141.5, 133.3, 132.7, 128.4, 127.9, 126.3, 125.2, 122.4, 119.8, 119.3, 95.3, 75.4, 30.5, 28.5, 19.3, 12.1; HRMS calcd for $C_{21}H_{17}Cl$: 304.1019; found: 304.1017.

Spectral data for (7*Z*,9*E*)-9-phenyl-2,4,5,6-tetrahydro-1*H*-cyclopenta[8]annulene (2a).



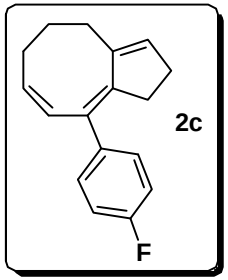
Pale yellow oil, IR (neat, cm^{-1}): 2932 (s), 2838 (w), 1012 (s), 889 (s), 749 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.28 ~ 7.25 (m, 2 H), 7.24 ~ 7.18 (m, 3 H), 6.15 (t, *J* = 8.0 Hz, 1 H), 5.78 ~ 5.69 (m, 2 H), 2.57 ~ 2.53 (m, 4 H), 2.41 ~ 2.39 (m, 2 H), 2.34~ 2.32 (m, 2 H), 1.92 ~ 1.87 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 141.0, 140.6, 138.3, 134.4, 133.1, 131.4, 128.1, 127.0, 126.8, 124.8, 38.4, 36.1, 31.2, 26.9, 23.1; HRMS calcd for $C_{17}H_{18}$: 222.1409, found: 222.1408.

Spectral data for (7*Z*,9*E*)-9-p-tolyl-2,4,5,6-tetrahydro-1*H*-cyclopenta[8]annulene (2b).



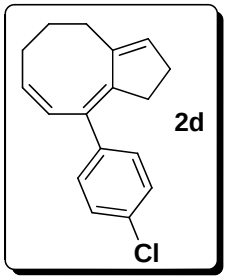
Yellow oil, IR (neat, cm^{-1}): 2938 (s), 2843 (w), 1012 (s), 869 (s), 731 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.12 ~ 7.07 (m, 4 H), 6.11 (t, *J* = 8.0 Hz, 1 H), 5.76 ~ 5.69 (m, 2 H), 2.55 ~ 2.53 (m, 4 H), 2.41 ~ 2.38 (m, 2 H), 2.35 ~ 2.33 (m, 2 H), 2.31 (s, 3 H), 1.92 ~ 1.88 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.9, 138.1, 137.8, 136.4, 134.5, 132.4, 131.4, 128.8, 126.9, 124.8, 38.4, 36.2, 31.3, 26.8, 23.1, 21.1; HRMS calcd for $C_{18}H_{20}$: 236.1565, found: 236.1575.

Spectral data for (7*Z*,9*E*)-9-(4-fluorophenyl)-2,4,5,6-tetrahydro-1*H*-cyclopenta[8]annulene (2c).



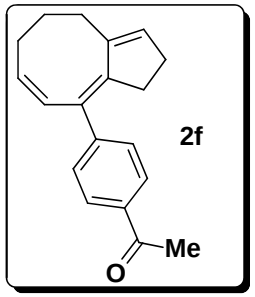
Colorless oil, IR (neat, cm^{-1}): 3098 (s), 2878 (w), 1438 (s), 958 (s), 779 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.17 ~ 7.14 (m, 2 H), 6.97 ~ 6.92 (m, 2 H), 6.09 (t, *J* = 7.9 Hz, 1 H), 5.79 ~ 5.68 (m, 2 H), 2.57 ~ 2.52 (m, 4 H), 2.41 ~ 2.37 (m, 2 H), 2.32 ~ 2.28 (m, 2 H), 1.93 ~ 1.85 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 162.0 (d, *J* = 243.8 Hz), 140.1, 138.5, 136.7 (d, *J* = 3.0 Hz), 134.1, 133.0, 131.5, 128.5 (d, *J* = 7.3 Hz), 124.8, 114.9 (d, *J* = 20.5 Hz), 38.4, 36.1, 31.2, 26.9, 23.1; HRMS calcd for $C_{17}H_{17}F$: 240.1314, found: 240.1315.

Spectral data for (7*Z*,9*E*)-9-(4-chlorophenyl)-2,4,5,6-tetrahydro-1*H*-cyclopenta[8]annulene (2d).



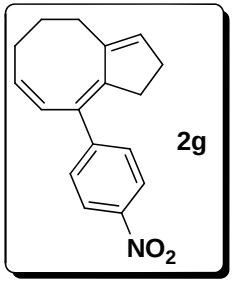
Colorless oil, IR (neat, cm^{-1}): 3035 (s), 2873 (w), 1421(s), 943 (s), 769 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.23 (d, *J* = 8.4 Hz, 2 H), 7.13 (d, *J* = 8.4 Hz, 2 H), 6.12 (t, *J* = 8.1 Hz, 1 H), 5.77 ~ 5.68 (m, 2 H), 2.56 ~ 2.52 (m, 4 H), 2.41 ~ 2.38 (m, 2 H), 2.31 ~ 2.28 (m, 2 H), 1.92 ~ 1.87 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 140.0, 139.1, 138.7, 133.9, 133.6, 132.5, 131.5, 128.3, 128.2, 124.7, 38.4, 36.0, 31.1, 26.9, 23.1; HRMS calcd for $C_{17}H_{17}Cl$: 256.1019, found: 256.1022.

Spectral data for 1-(4-((7*Z*,9*E*)-2,4,5,6-tetrahydro-1*H*-cyclopenta[8]annulen-9-yl)phenyl)ethanone (2f).



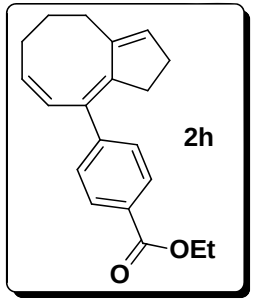
Pale yellow oil, IR (neat, cm^{-1}): 3012 (s), 2852 (w), 1647 (s), 903 (s), 785 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.86 (d, *J* = 8.3 Hz, 2 H), 7.29 (d, *J* = 8.3 Hz, 2 H), 6.25 (t, *J* = 8.0 Hz, 1 H), 5.78 ~ 5.69 (m, 2 H), 2.60 ~ 2.54 (m, 7 H), 2.42 ~ 2.39 (m, 2 H), 2.32 ~ 2.29 (m, 2 H), 1.93 ~ 1.88 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.8, 145.5, 140.3, 139.1, 135.5, 135.2, 133.6, 131.5, 128.4, 127.1, 124.7, 38.4, 36.0, 31.0, 27.0, 26.6, 23.1; HRMS calcd for $C_{19}H_{20}O$: 264.1514, found: 264.1507.

Spectral data for (7*Z*,9*E*)-9-(4-nitrophenyl)-2,4,5,6-tetrahydro-1*H*-cyclopenta[8]annulene (2g).



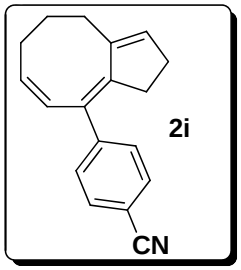
Deep yellow oil, IR (neat, cm^{-1}): 3072 (s), 2972 (w), 1653 (s), 1238 (s), 932 (s), 791 (s) ; ^{1}H NMR (600 MHz, $CDCl_3$): δ 8.13 (d, *J* = 9.0 Hz, 2 H), 7.34 (d, *J* = 9.0 Hz, 2 H), 6.30 (t, *J* = 8.1 Hz, 1 H), 5.79 ~ 5.70 (m, 2 H), 2.62 ~ 2.55 (m, 4 H), 2.44 ~ 2.40 (m, 2 H), 2.31 ~ 2.28 (m, 2 H), 1.95 ~ 1.90 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.3, 139.8, 139.6, 136.7, 132.9, 131.7, 127.6, 124.6, 123.6, 123.4, 38.4, 36.0, 30.9, 27.1, 23.1; HRMS calcd for $C_{17}H_{17}NO_2$: 267.1259, found: 267.1252.

Spectral data for ethyl 4-((7*Z*,9*E*)-2,4,5,6-tetrahydro-1*H*-cyclopenta[8]annulen-9-yl)benzoate (2h).



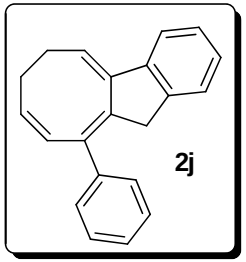
Yellow oil, IR (neat, cm^{-1}): 3056 (s), 2971 (w), 1711 (s), 912 (s), 786 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.94 (d, *J* = 8.2 Hz, 2 H), 7.26 (d, *J* = 8.2 Hz, 2 H), 6.24 (t, *J* = 8.0 Hz, 1 H), 5.79 ~ 5.69 (m, 2 H), 4.34 (q, *J* = 7.1 Hz, 2 H), 2.59 ~ 2.54 (m, 4 H), 2.42 ~ 2.39 (m, 2 H), 2.31 ~ 2.29 (m, 2 H), 1.93 ~ 1.88 (m, 2 H), 1.37 (t, *J* = 7.1 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.6, 145.2, 140.5, 139.0, 134.9, 133.8, 131.5, 129.5, 128.7, 126.9, 124.8, 60.8, 38.4, 36.1, 31.0, 27.0, 23.1, 14.3; HRMS calcd for $C_{20}H_{22}O_2$: 294.1620, found: 294.1619.

Spectral data for 4-((7*Z*,9*E*)-2,4,5,6-tetrahydro-1*H*-cyclopenta[8]annulen-9-yl)benzonitrile (2i).



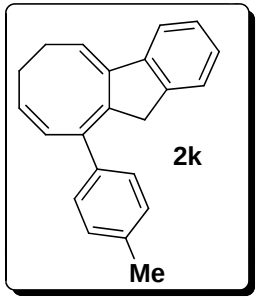
Pale yellow oil, IR (neat, cm^{-1}): 3097 (s), 2983 (w), 2258 (m), 998 (s), 793 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.55 (d, *J* = 8.4 Hz, 2 H), 7.29 (d, *J* = 8.4 Hz, 2 H), 6.24 (t, *J* = 8.0 Hz, 1 H), 5.77 ~ 5.69 (m, 2 H), 2.59 ~ 2.53 (m, 4 H), 2.42 ~ 2.39 (m, 2 H), 2.29 ~ 2.27 (m, 2 H), 1.93 ~ 1.88 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.5, 140.1, 140.0, 136.3, 133.3, 132.3, 131.9, 127.8, 124.9, 119.4, 110.4, 38.6, 36.2, 31.2, 27.3, 23.3; HRMS calcd for $C_{18}H_{17}N$: 247.1361, found: 247.1365.

Spectral data for (4b*E*,8*Z*,10*E*)-10-phenyl-7,11-dihydro-6*H*-cycloocta[*a*]indene (2j):



Yellow liquid ; IR (neat, cm^{-1}): 2935 (s), 2853 (w), 2821(w), 870 (s), 687 (s); ^1H NMR (400 MHz , $CDCl_3$): δ 7.44 ~ 7.32 (m, 4 H), 7.30 ~ 7.20 (m, 5 H), 6.40 ~ 6.32 (m, 2 H), 6.14 ~ 6.08 (m, 1H), 3.44 (s, 2H), 2.67 ~ 2.62 (m, 2 H), 2.56 ~ 2.54 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 143.5, 141.8, 140.7, 139.4, 137.8, 134.7, 128.2, 127.5, 127.1, 126.4, 124.8, 123.5, 120.8, 119.4, 40.5, 31.3, 27.0; HRMS calcd for $C_{21}H_{18}$: 270.1409; found: 270.1411.

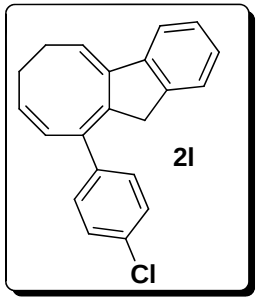
Spectral data for (4b*E*,8*Z*,10*E*)-10-p-tolyl-7,11-dihydro-6*H*-cycloocta[*a*]indene (2k):



Yellow liquid ; IR (neat, cm^{-1}): 2937 (s), 2848 (w), 2810 (w), 865 (s), 683 (s); ^1H NMR (400 MHz , $CDCl_3$): δ 7.42 ~ 7.34 (m, 3 H), 7.24 ~ 7.20 (m, 1 H), 7.15 ~ 7.09 (m, 4 H), 6.37 ~ 6.28 (m, 2 H), 6.11 ~ 6.07 (m, 1 H), 3.43 (s, 2 H), 2.63 ~ 2.60 (m, 2 H), 2.59 ~ 2.52 (m, 2 H), 2.33 (s, 3H);

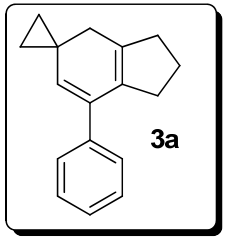
^{13}C NMR (100 MHz, $CDCl_3$): δ 146.1, 143.5, 140.4, 139.6, 139.0, 137.6, 136.8, 134.7, 134.1, 128.9, 127.4, 126.3, 124.8, 123.5, 120.8, 119.4, 40.5, 31.4, 26.9, 21.1; HRMS calcd for $C_{22}H_{20}$: 284.1565; found: 284.1569.

Spectral data for (4b*E*,8*Z*,10*E*)-10-(4-chlorophenyl)-7,11-dihydro-6*H*-cycloocta[*a*]indene (2l):



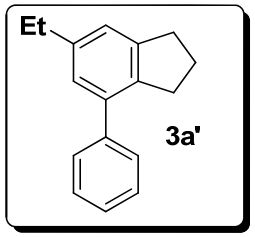
Yellow liquid ; IR (neat, cm^{-1}): 3092 (s), 2987 (w), 1431(w) 981 (s), 772 (s); ^{1}H NMR (400 MHz , $CDCl_3$): δ 7.42 ~ 7.34 (m, 4 H), 7.26 ~ 7.15 (m, 4 H), 6.37 ~ 6.28 (m, 2 H), 6.12 ~ 6.07 (m, 1 H), 3.38 (s, 2 H), 2.63 ~ 2.60 (m, 2 H), 2.59 ~ 2.52 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.9, 143.4, 140.2, 139.6, 138.7, 135.2, 134.9, 132.9, 128.8, 128.4, 126.4, 125.0, 123.5, 120.8, 119.5, 40.4, 31.2, 27.0; HRMS calcd for $C_{21}H_{17}Cl$: 304.1019; found: 304.1021.

Spectral data for 7'-phenyl-1',2',3',4'-tetrahydrospiro[cyclopropane-1,5'-indene] (3a).



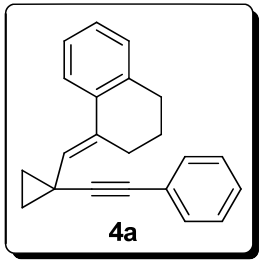
Colorless oil, IR (neat, cm^{-1}): 3017 (s), 2897 (w), 1641 (w), 1442 (s), 886 (s), 679 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.33 ~ 7.31 (m, 2 H), 7.25 ~ 7.22 (m, 3 H), 5.56 (s, 1 H), 2.41 ~ 2.31 (m, 4 H), 2.21 (s, 2 H), 1.92 ~ 1.84 (m, 2 H), 1.02 ~ 0.99 (m, 2 H), 0.72 ~ 0.70 (m, 2 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 142.3, 131.5, 128.1, 127.3, 125.8, 124.2, 95.5, 39.8, 35.4, 32.4, 23.6, 15.7, 11.6; HRMS calcd for $C_{17}H_{18}$: 222.1409, found: 222.1408.

Spectral data for 6-ethyl-4-phenyl-2,3-dihydro-1*H*-indene (3a').



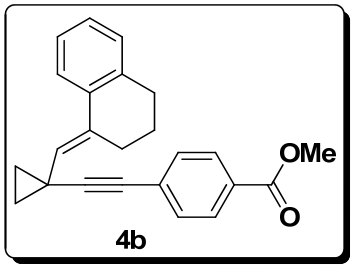
Colorless oil, IR (neat, cm^{-1}): 3011 (s), 2963(s), 2863 (w), 1632 (w), 1210 (w), 856 (s), 669 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.46 ~ 7.38 (m, 4 H), 7.33 ~ 7.28 (m, 1 H), 7.09 (s, 1 H), 7.03 (s, 1 H), 2.96 ~ 2.91 (m, 4 H), 2.66 (q, J = 7.6 Hz, 2 H), 2.07 ~ 1.99 (m, 2 H), 1.26 (t, J = 7.6 Hz, 3 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.2, 142.8, 141.7, 139.2, 138.0, 128.5, 128.2, 126.7, 126.3, 123.0, 33.2, 32.4, 28.7, 25.9, 15.9; HRMS calcd for $C_{17}H_{18}$: 222.1409, found: 222.1402.

Spectral data for (*E*)-1-((1-(phenylethynyl)cyclopropyl)methylene)-1,2,3,4-tetrahydronaphthalene (4a).



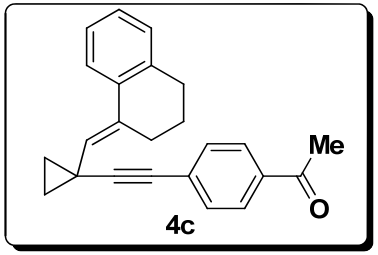
Pale yellow oil, IR (neat, cm^{-1}): 3034 (s), 2986 (w), 2125 (w), 1425 (s), 895 (s), 755 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.54 ~ 7.52 (m, 1 H), 7.37 ~ 7.35 (m, 2 H), 7.27 ~ 7.22 (m, 3 H), 7.14 ~ 7.12 (m, 2 H), 7.09 ~ 7.07 (m, 1 H), 5.99 (s, 1 H), 2.95 ~ 2.91 (m, 2 H), 2.80 (t, *J* = 6.2 Hz, 2 H), 1.93 ~ 1.86 (m, 2 H), 1.36 ~ 1.33 (m, 2 H), 1.03 ~ 1.00 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.2, 137.6, 135.6, 131.5, 128.9, 128.4, 128.1, 127.4, 127.1, 126.0, 124.0, 123.7, 94.5, 75.8, 30.4, 27.7, 23.0, 19.2, 10.9; HRMS calcd for $C_{22}H_{20}$: 284.1565, found: 284.1561.

Spectral data for (*E*)-methyl 4-((1-((3,4-dihydronaphthalen-1(2*H*)-ylidene)methyl)cyclopropyl)ethynyl)benzoate (4b).



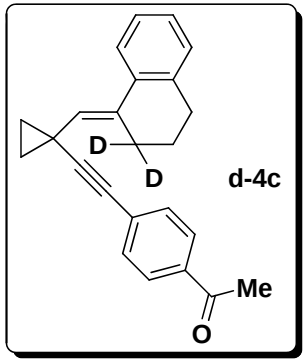
Pale yellow viscous oil, IR (neat, cm^{-1}): 3098 (s), 2991 (w), 2205 (w), 1713 (s), 1432 (s), 915 (s), 768 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.91 (d, *J* = 8.4 Hz, 2 H), 7.52 ~ 7.51 (m, 1 H), 7.39 (d, *J* = 8.4 Hz, 2 H), 7.13 ~ 7.08 (m, 3 H), 5.98 (s, 1 H), 3.88 (s, 3 H), 2.89 (t, *J* = 5.4 Hz, 2 H), 2.79 (t, *J* = 6.0 Hz, 2 H), 1.90 ~ 1.87 (m, 2 H), 1.37 ~ 1.36 (m, 2 H), 1.05 ~ 1.03 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 166.7, 140.6, 137.6, 135.4, 131.4, 129.3, 129.0, 128.9, 128.6, 127.2, 126.0, 123.7, 123.4, 98.2, 75.3, 52.1, 30.4, 27.7, 23.0, 19.3, 11.0; HRMS calcd for $C_{24}H_{22}O_2$: 342.1620, found: 342.1617.

Spectral data for (*E*)-1-(4-((1-((3,4-dihydronaphthalen-1(2*H*)-ylidene)methyl)cyclopropyl)ethynyl)phenyl)ethanone (4c).



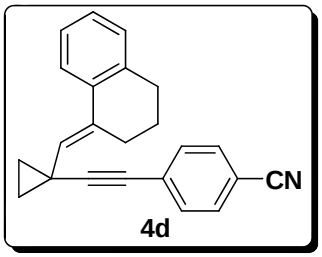
Pale yellow oil, IR (neat, cm^{-1}): 3087 (s), 2988 (w), 2243 (w), 1681 (s), 1422 (s), 892 (s), 763 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 7.83 (d, *J* = 8.4 Hz, 2 H), 7.52 ~ 7.51 (m, 1 H), 7.41 (d, *J* = 8.4 Hz, 2 H), 7.13 ~ 7.09 (m, 3 H), 5.98 (s, 1 H), 2.88 (t, *J* = 6.4 Hz, 2 H), 2.79 (t, *J* = 6.0 Hz, 2 H), 2.56 (s, 3 H), 1.90 ~ 1.85 (m, 2 H), 1.38 ~ 1.36 (m, 2 H), 1.05 ~ 1.04 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.3, 140.6, 137.6, 135.4, 135.3, 131.5, 129.1, 128.9, 128.1, 127.2, 126.0, 123.6, 123.3, 98.6, 75.3, 30.4, 27.7, 26.5, 22.9, 19.3, 11.0; HRMS calcd for $C_{24}H_{22}O$: 326.1671, found: 326.1669.

Spectral data for compound (d-4c).



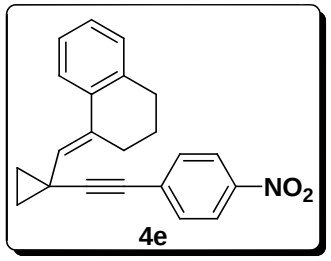
Brown oil, IR (neat, cm^{-1}): 3089 (s), 2982 (w), 2239 (w), 1678 (s), 1421 (s), 890 (s), 759 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.84 (d, *J* = 8.4 Hz, 2 H), 7.53 ~ 7.51 (m, 1 H), 7.41 (d, *J* = 8.4 Hz, 2 H), 7.14 ~ 7.07 (m, 3 H), 5.99 (s, 1 H), 2.80 (t, *J* = 6.4 Hz, 2 H), 2.56 (s, 3 H), 1.88 (t, *J* = 6.0 Hz, 2 H), 1.38 ~ 1.36 (m, 2 H), 1.06 ~ 1.03 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.4, 140.5, 137.6, 135.5, 135.4, 131.6, 129.1, 129.0, 128.1, 127.2, 126.0, 123.7, 123.4, 98.6, 75.3, 30.3, 26.6, 22.8, 19.3, 11.0; HRMS calcd for $C_{24}H_{20}D_2O$: 328.1796, found: 328.1791.

Spectral data for (*E*)-(4-((1-((3,4-dihydronaphthalen-1(2*H*)-ylidene)methyl)cyclopropyl)ethynyl)benzonitrile (4d).



Yellow viscous oil, IR (neat, cm^{-1}): 3106 (s), 2981 (w), 2248 (m), 2231 (w), 1429 (s), 895 (s), 783 (s); ^1H NMR (400 MHz, $CDCl_3$): δ7.54 ~ 7.52 (m, 3 H), 7.41 (d, *J* = 8.0 Hz, 2 H), 7.17 ~ 7.08 (m, 3 H), 5.99 (s, 1 H), 2.85 (t, *J* = 5.8 Hz, 2 H), 2.80 (t, *J* = 6.2 Hz, 2 H), 1.92 ~ 1.86 (m, 2 H), 1.39 ~ 1.36 (m, 2 H), 1.08 ~ 1.05 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 140.8, 137.6, 135.2, 132.0, 131.8, 129.0, 128.9, 127.3, 126.0, 123.6, 123.0, 118.6, 110.5, 99.8, 74.6, 30.3, 27.7, 22.9, 19.4, 10.9; HRMS calcd for $C_{23}H_{19}N$: 309.1517, found: 309.1512.

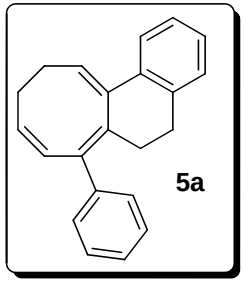
Spectral data for (*E*)-1-((1-((4-nitrophenyl)ethynyl)cyclopropyl)methylene)-1,2,3,4-tetrahydronaphthalene (4e).



4e

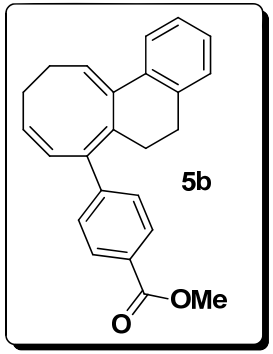
Yellow oil, IR (neat, cm^{-1}): 3095 (s), 2989 (w), 2249 (w), 1651 (s), 1433 (s), 912 (s), 789 (s); ^1H NMR (400 MHz, $CDCl_3$): δ 8.11 (d, *J* = 8.4 Hz, 2 H), 7.54 ~ 7.52 (m, 1 H), 7.46 (d, *J* = 8.4 Hz, 2 H), 7.17 ~ 7.08 (m, 3 H), 6.00 (s, 1 H), 2.88 (t, *J* = 5.8 Hz, 2 H), 2.81 (t, *J* = 6.0 Hz, 2 H), 1.93 ~ 1.87 (m, 2 H), 1.41 ~ 1.39 (m, 2 H), 1.10 ~ 1.07 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 146.4, 140.9, 137.6, 135.2, 132.1, 131.2, 129.0, 127.4, 126.0, 123.7, 123.4, 122.8, 101.0, 74.5, 30.3, 27.8, 22.9, 19.5, 11.0; HRMS calcd for $C_{22}H_{19}NO_2$: 329.1416, found: 329.1412.

Spectral data for (6a*E*,8*Z*,12*E*)-7-phenyl-5,6,10,11-tetrahydrocycloocta[*a*]naphthalene (5a).



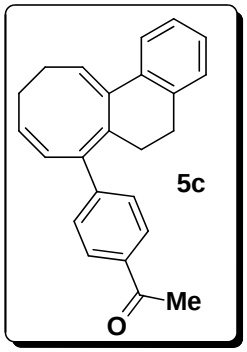
Pale yellow oil, IR (neat, cm^{-1}): 2936 (s), 2855 (w), 2818 (w), 890 (s), 697 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.39 ~ 7.35 (m, 3 H), 7.32 ~ 7.29 (m, 2 H), 7.25 ~ 7.19 (m, 2 H), 7.18 ~ 7.16 (m, 2 H), 6.24 (dd, *J* = 9.3, 6.4 Hz, 1 H), 6.05 (d, *J* = 12.7 Hz, 1 H), 5.94 ~ 5.92 (m, 1 H), 2.85 ~ 2.79 (m, 2 H), 2.75 ~ 2.68 (m, 1 H), 2.50 ~ 2.49 (m, 1 H), 2.29 ~ 2.16 (m, 4 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 144.5, 140.9, 136.2, 135.9, 133.4, 132.4, 132.0, 130.4, 128.4, 127.2, 127.0, 126.9, 126.8 (2xCH), 126.4, 125.4, 29.1, 28.7, 27.7, 27.2; HRMS calcd for $C_{22}H_{20}$: 284.1565, found: 284.1563.

Spectral data for methyl 4-((6a*E*,8*Z*,12*E*)-5,6,10,11-tetrahydrocycloocta[*a*]naphthalene-7-yl)benzoate (5b).



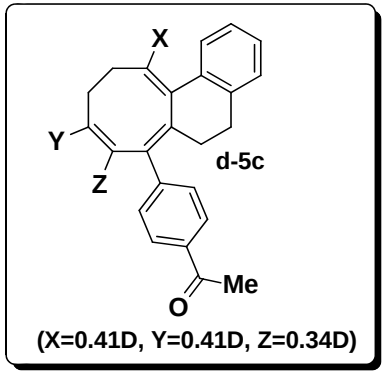
Pale yellow oil, IR (neat, cm^{-1}): 3054 (s), 2979 (w), 1716 (s), 912 (s), 816 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.98 (d, *J* = 8.4 Hz, 2 H), 7.45 (d, *J* = 8.4 Hz, 2 H), 7.36 (dd, *J* = 7.2, 0.6 Hz, 1 H), 7.23 ~ 7.15 (m, 3 H), 6.35 (dd, *J* = 9.6, 6.6 Hz, 1 H), 6.06 (d, *J* = 13.2 Hz, 1 H), 5.95 ~ 5.91 (m, 1 H), 3.90 (s, 3H), 2.87 ~ 2.81 (m, 2 H), 2.73 ~ 2.69 (m, 1 H), 2.55 ~ 2.51 (m, 1 H), 2.29 ~ 2.21 (m, 3 H), 2.14 ~ 2.10 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 167.0, 145.4, 143.8, 136.1, 135.6, 132.9, 132.5, 132.4, 132.0, 129.8, 128.6, 127.3, 127.1, 126.9, 126.7, 126.4, 125.5, 52.0, 28.9, 28.6, 27.7, 27.3; HRMS calcd for $C_{24}H_{22}O_2$: 342.1620, found: 342.1622.

Spectral data for 1-(4-((6a*E*,8*Z*,12*E*)-5,6,10,11-tetrahydrocycloocta[*a*]naphthalen-7-yl)phenyl)ethanone (5c).



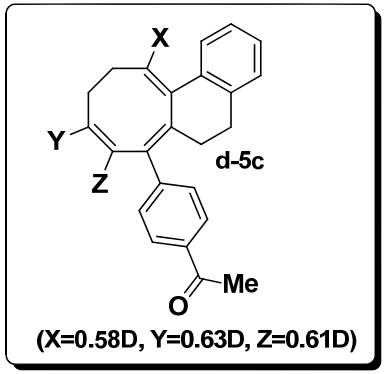
Yellow viscous oil, IR (neat, cm^{-1}): 3016 (s), 2865 (w), 1640 (s), 923 (s), 795 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.92 (d, *J* = 8.3 Hz, 2 H), 7.48 (d, *J* = 8.3 Hz, 2 H), 7.38 (d, *J* = 7.4 Hz, 1 H), 7.25 ~ 7.16 (m, 3 H), 6.38 (dd, *J* = 9.4, 6.4 Hz, 1 H), 6.07 (d, *J* = 12.8 Hz, 1 H), 5.96 ~ 5.93 (m, 1 H), 2.87 ~ 2.83 (m, 2 H), 2.75 ~ 2.71 (m, 1 H), 2.59 (s, 3 H), 2.58 ~ 2.53 (m, 1 H), 2.30 ~ 2.23 (m, 3 H), 2.15 ~ 2.12 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.7, 145.6, 143.7, 136.0, 135.7, 135.5, 132.9, 132.7, 132.4, 132.0, 128.6, 127.3, 127.1, 126.9, 126.8, 126.4, 125.4, 28.9, 28.5, 27.7, 27.2, 26.6; HRMS calcd for $C_{24}H_{22}O$: 326.1671, found: 326.1669.

Spectral data for compound (d-5c).



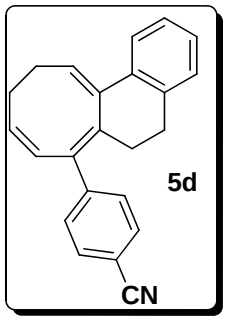
Pale yellow oil, IR (neat, cm^{-1}): 3022 (s), 2871 (w), 1632 (s), 914 (s), 782 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.91 ~ 7.90 (m, 2 H), 7.47 ~ 7.46 (m, 2 H), 7.36 (d, *J* = 7.4 Hz, 1 H), 7.24 ~ 7.16 (m, 3 H), 6.37 ~ 6.34 (m, 1 H), 6.06 ~ 6.05 (m, 1 H), 5.94 ~ 5.91 (m, 1 H), 2.85 ~ 2.81 (m, 2 H), 2.73 ~ 2.64 (m, 1 H), 2.58 ~ 2.52 (m, 4 H), 2.25 ~ 2.21 (m, 3 H), 2.12 ~ 2.10 (m, 1 H); ^{13}C NMR (150 MHz, $CDCl_3$): δ 197.7, 145.6, 143.8, 136.1, 135.7, 135.6, 133.0 ~ 131.9 (m), 128.6, 127.3, 127.1, 126.9, 126.8 ~ 126.7 (m), 126.4, 125.5, 28.9 ~ 28.8 (m), 28.6, 27.7, 27.3 ~ 27.2 (m), 26.6; HRMS calcd for $C_{24}H_{19}D_3O$: 329.1859, found: 329.1855.

Spectral data for compound (d-5c).



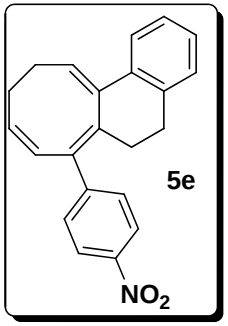
Pale yellow oil, IR (neat, cm^{-1}): 3022 (s), 2871 (w), 1632 (s), 914 (s), 782 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.92 ~ 7.90 (m, 2 H), 7.48 ~ 7.47 (m, 2 H), 7.37 (d, *J* = 7.4 Hz, 1 H), 7.23 ~ 7.17 (m, 3 H), 6.37 (dd, *J* = 9.4, 6.4 Hz, 1 H), 6.08 ~ 6.06 (m, 1 H), 5.95 ~ 5.91 (m, 1 H), 2.86 ~ 2.82 (m, 2 H), 2.73 ~ 2.70 (m, 1 H), 2.59 ~ 2.57 (m, 4 H), 2.26 ~ 2.22 (m, 3 H), 2.13 ~ 2.11 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 197.7, 145.6, 143.7, 136.0, 135.7, 135.6, 132.7 ~ 131.9 (m), 128.6, 127.3, 127.1, 126.9, 126.8 ~ 126.7 (m), 126.4, 125.5, 28.9 ~ 28.8 (m), 28.6, 27.7, 27.3 ~ 27.2 (m), 26.6; HRMS calcd for $C_{24}H_{19}D_3O$: 329.1859, found: 329.1856.

Spectral data for 4-((6a*E*,8*Z*,12*E*)-5,6,10,11-tetrahydrocycloocta[*a*]naphthalen-7-yl)benzonitrile (5d).



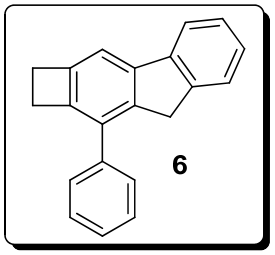
Orange viscous oil, IR (neat, cm^{-1}): 3091 (s), 2988 (w), 2248 (m), 985 (s), 783 (s); ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.60 (d, *J* = 8.5 Hz, 2 H), 7.46 (d, *J* = 8.5 Hz, 2 H), 7.36 (dd, *J* = 7.6, 1.1 Hz, 1 H), 7.25 ~ 7.16 (m, 3 H), 6.35 (dd, *J* = 9.4, 6.4 Hz, 1 H), 6.05 (d, *J* = 12.8 Hz, 1 H), 5.95 ~ 5.94 (m, 1 H), 2.86 ~ 2.80 (m, 2 H), 2.75 ~ 2.72 (m, 1 H), 2.56 ~ 2.53 (m, 1 H), 2.28 ~ 2.22 (m, 3 H), 2.10 ~ 2.08 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.4, 143.3, 135.9, 135.4, 133.5, 133.4, 132.3, 132.1, 131.8, 127.4, 127.3, 127.2, 126.7, 126.5, 125.5, 119.1, 110.4, 28.8, 28.5, 27.6, 27.3; HRMS calcd for $C_{23}H_{19}N$: 309.1517, found: 309.1521.

Spectral data for (6a*E*,8*Z*,12*E*)-7-(4-nitrophenyl)-5,6,10,11-tetrahydrocycloocta[*a*]naphthalene (5e).

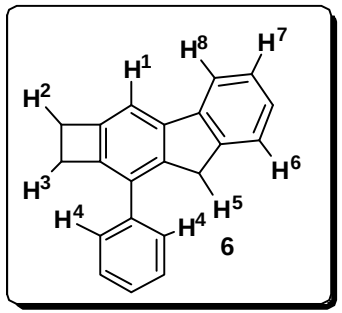


Yellow oil, IR (neat, cm^{-1}): 3079 (s), 2975 (w), 1649 (s), 1338 (s), 932 (s), 823 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 8.17 (d, *J* = 8.9 Hz, 2 H), 7.53 (d, *J* = 8.9 Hz, 2 H), 7.37 (d, *J* = 7.4 Hz, 1 H), 7.25 ~ 7.17 (m, 3 H), 6.41 (dd, *J* = 9.5, 6.4 Hz, 1 H), 6.07 (d, *J* = 12.8 Hz, 1 H), 5.96 ~ 5.95 (m, 1 H), 2.86 ~ 2.81 (m, 2 H), 2.76 ~ 2.73 (m, 1 H), 2.58 ~ 2.54 (m, 1 H), 2.30 ~ 2.24 (m, 3 H), 2.10 ~ 2.07 (m, 1 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 147.4, 146.7, 143.0, 135.9, 135.3, 134.3, 133.5, 132.1, 131.6, 127.4, 127.3, 127.2, 126.7, 126.5, 125.5, 123.8, 28.7, 28.5, 27.6, 27.4; HRMS calcd for $C_{22}H_{19}NO_2$: 329.1416, found: 329.1416.

Spectral data for 9-phenyl-2,8-dihydro-1*H*-cyclobuta[*b*]fluorene (6).

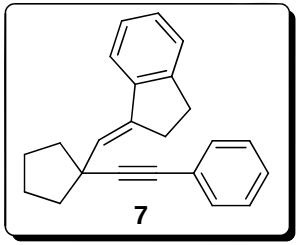


White solid, IR (neat, cm^{-1}): 3023 (s), 2970 (w), 2857(w), 1453 (s), 1365 (s), 906 (s), 788 (s); ^1H NMR (600 MHz, $CDCl_3$): δ 7.76 (d, *J* = 7.2 Hz, 1 H), 7.58 (d, *J* = 7.8 Hz, 2 H), 7.50 (s, 1H), 7.48 ~ 7.45 (m, 3 H), 7.36 ~ 7.33 (m, 2 H), 7.25 ~ 7.23 (m, 1 H), 3.94 (s, 2 H), 3.27 ~ 3.25 (m, 2 H), 3.21 ~ 3.19 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 144.6, 142.7, 142.5, 142.3, 141.5, 139.2, 137.7, 133.5, 128.5, 128.4, 127.2, 126.6, 126.2, 124.7, 119.4, 113.6, 36.9, 29.0, 28.0; HRMS calcd for $C_{21}H_{16}$: 268.1252, found: 268.1255.



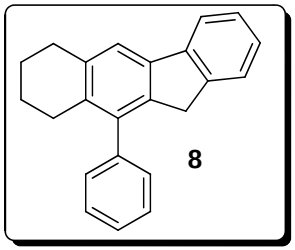
Irradiation	Intensity increase
H^8 (δ 7.76)	H^1 (δ 7.50, 10.61 %), H^7 (δ 7.36 ~ 7.33, 8.22 %),
H^2 (δ 3.21 ~ 3.19)	H^1 (δ 7.50, 1.21 %),
H^3 (δ 3.27 ~ 3.25)	H^4 (δ 7.58, 3.36 %),
H^5 (δ 3.94)	H^4 (δ 7.58, 6.98 %, H^6 (δ 7.48 ~ 7.45, 2.73 %),

Spectral data for (*E*)-1-((1-(phenylethynyl)cyclopentyl)methylene)-2,3-dihydro-1*H*-indene (7).



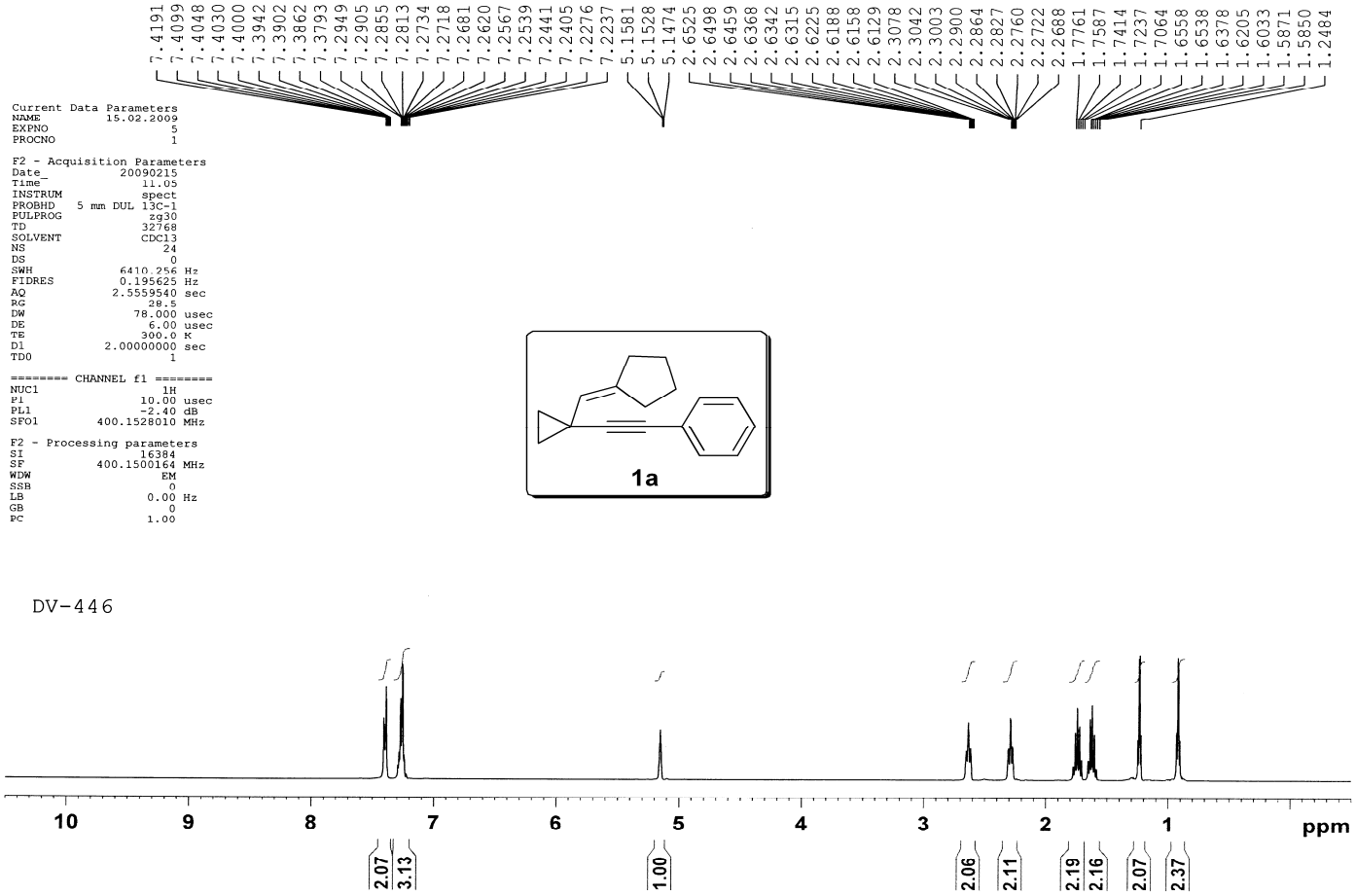
Yellow oil, IR (neat, cm^{-1}): 3033 (s), 2981 (w), 2135 (w), 1451 (s), 1432 (s), 890 (s), 763 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.44 ~ 7.42 (m, 1 H), 7.39 ~ 7.36 (m, 2 H), 7.26 ~ 7.22 (m, 4 H), 7.17 ~ 7.14 (m, 2 H), 6.05 (t, *J* = 2.6 Hz, 1 H), 3.11 ~ 3.08 (m, 2 H), 3.03 ~ 2.99 (m, 2 H), 2.23 ~ 2.18 (m, 2 H), 1.92 ~ 1.85 (m, 4 H), 1.78 ~ 1.75 (m, 2 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 145.6, 143.9, 142.3, 131.4, 128.1, 127.7, 127.4, 126.3, 125.2, 124.3, 123.7, 119.9, 95.4, 81.2, 42.9, 41.9, 30.8, 28.6, 23.9; HRMS calcd for $C_{23}H_{22}$: 298.1722, found: 298.1720.

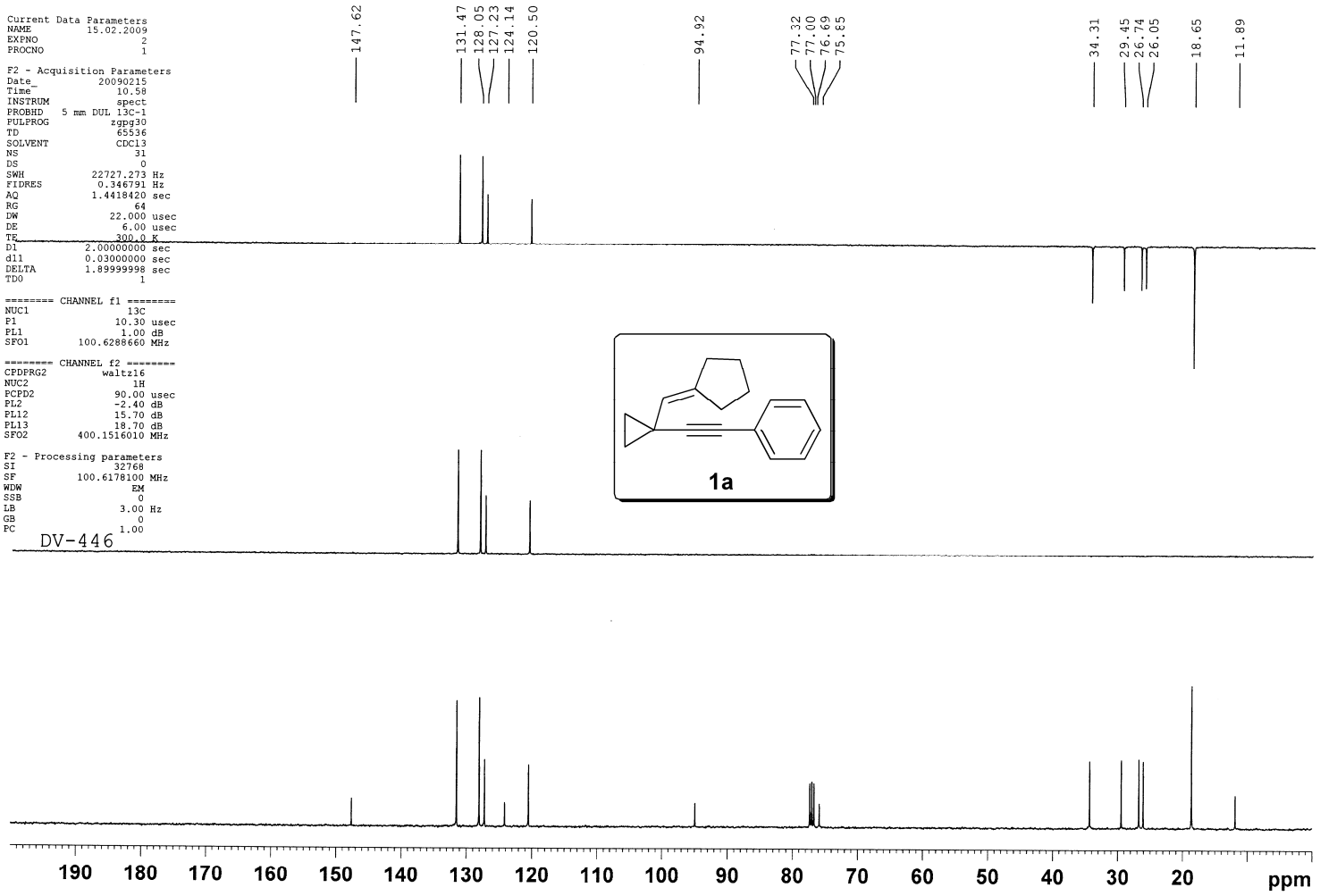
Spectral data for 10-phenyl-7,8,9,11-tetrahydro-6*H*-benzo[b]fluorene (8).

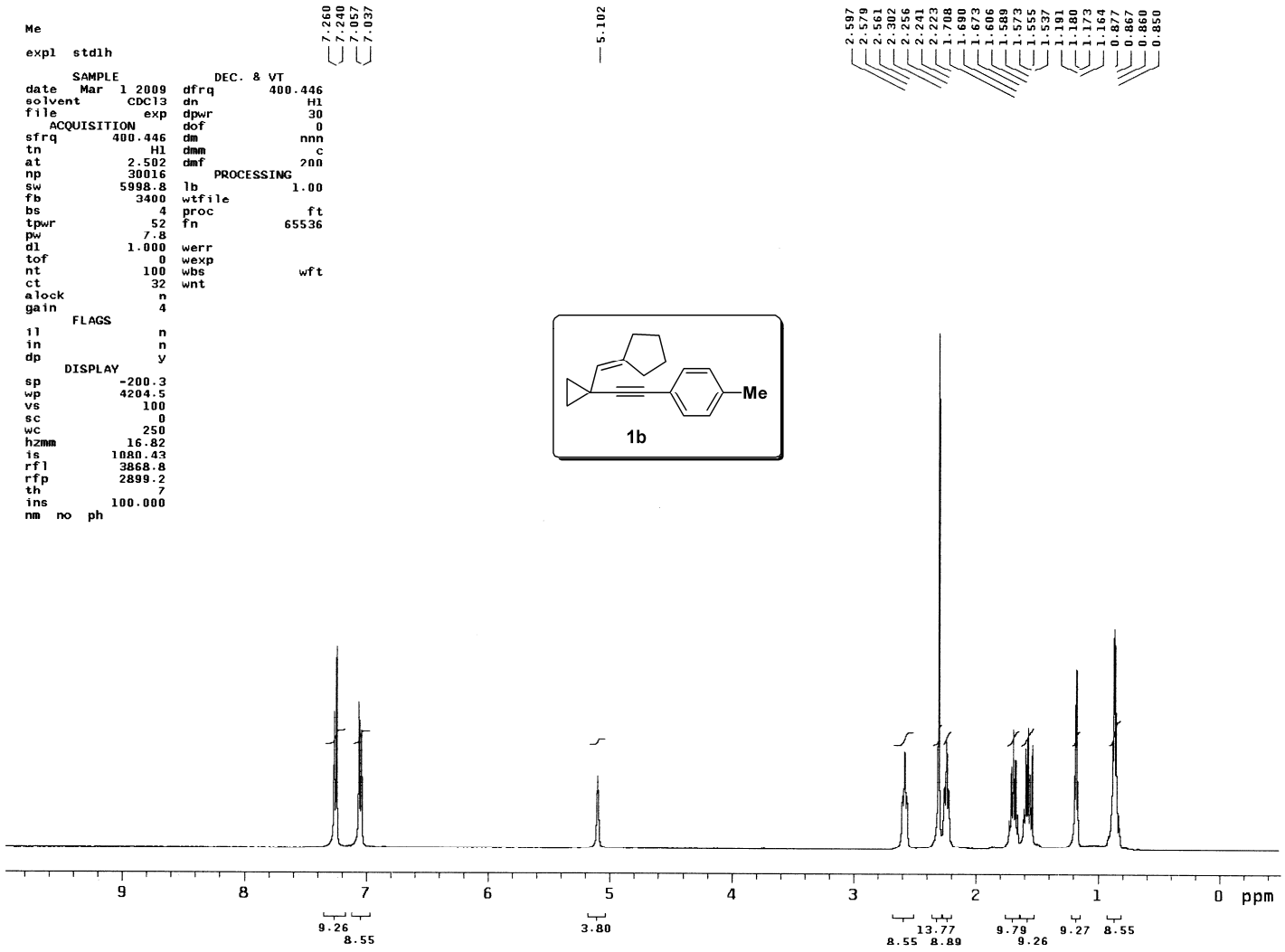


Pale yellow oil, IR (neat, cm^{-1}): 3029 (s), 2870 (w), 1453 (s), 1412 (s), 905 (s), 768 (s); ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.75 (d, *J* = 7.5 Hz, 1 H), 7.53 (s, 1 H), 7.47 ~ 7.43 (m, 2 H), 7.39 ~ 7.31 (m, 3 H), 7.27 ~ 7.19 (m, 3 H), 3.54 (s, 2 H), 2.95 (t, *J* = 6.2 Hz, 2 H), 2.50 (t, *J* = 6.3 Hz, 2 H), 1.84 ~ 1.70 (m, 4 H); ^{13}C NMR (100 MHz, $CDCl_3$): δ 143.5, 141.8, 140.4, 139.7, 138.9, 138.8, 136.3, 133.6, 128.9, 128.5, 126.9, 126.6, 126.2, 124.8, 119.6, 36.5, 30.5, 28.2, 23.5, 23.1; HRMS calcd for $C_{23}H_{20}$: 296.1565, found: 296.1564.

(IV) ^{1}H and ^{13}C spectra of compounds (1a to 8).



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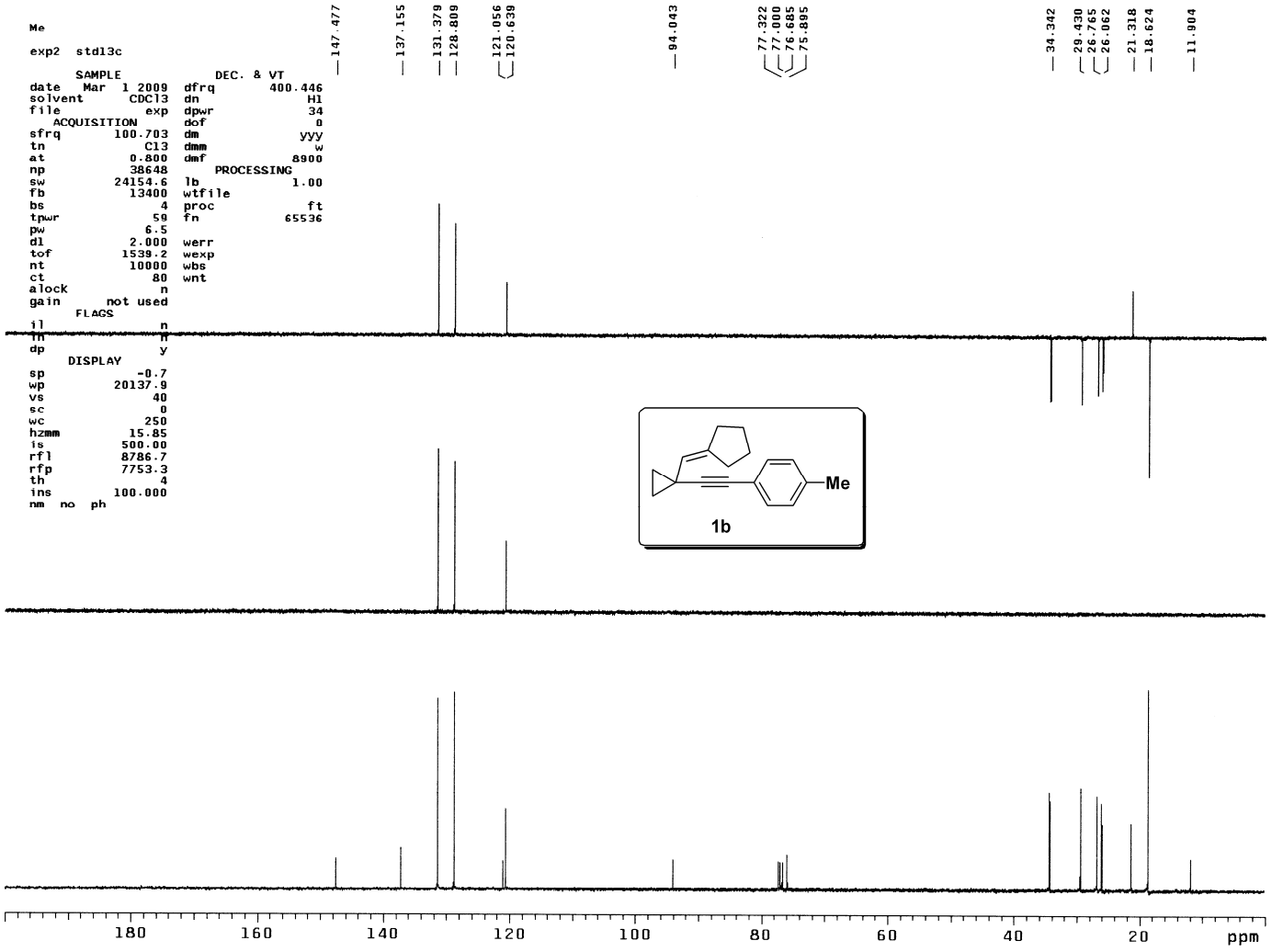

DV-502

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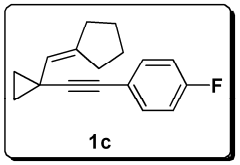
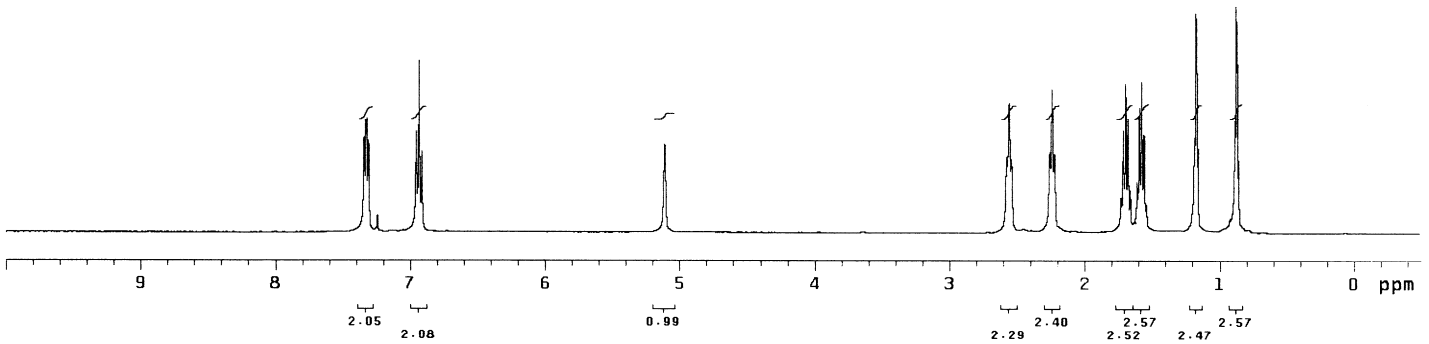
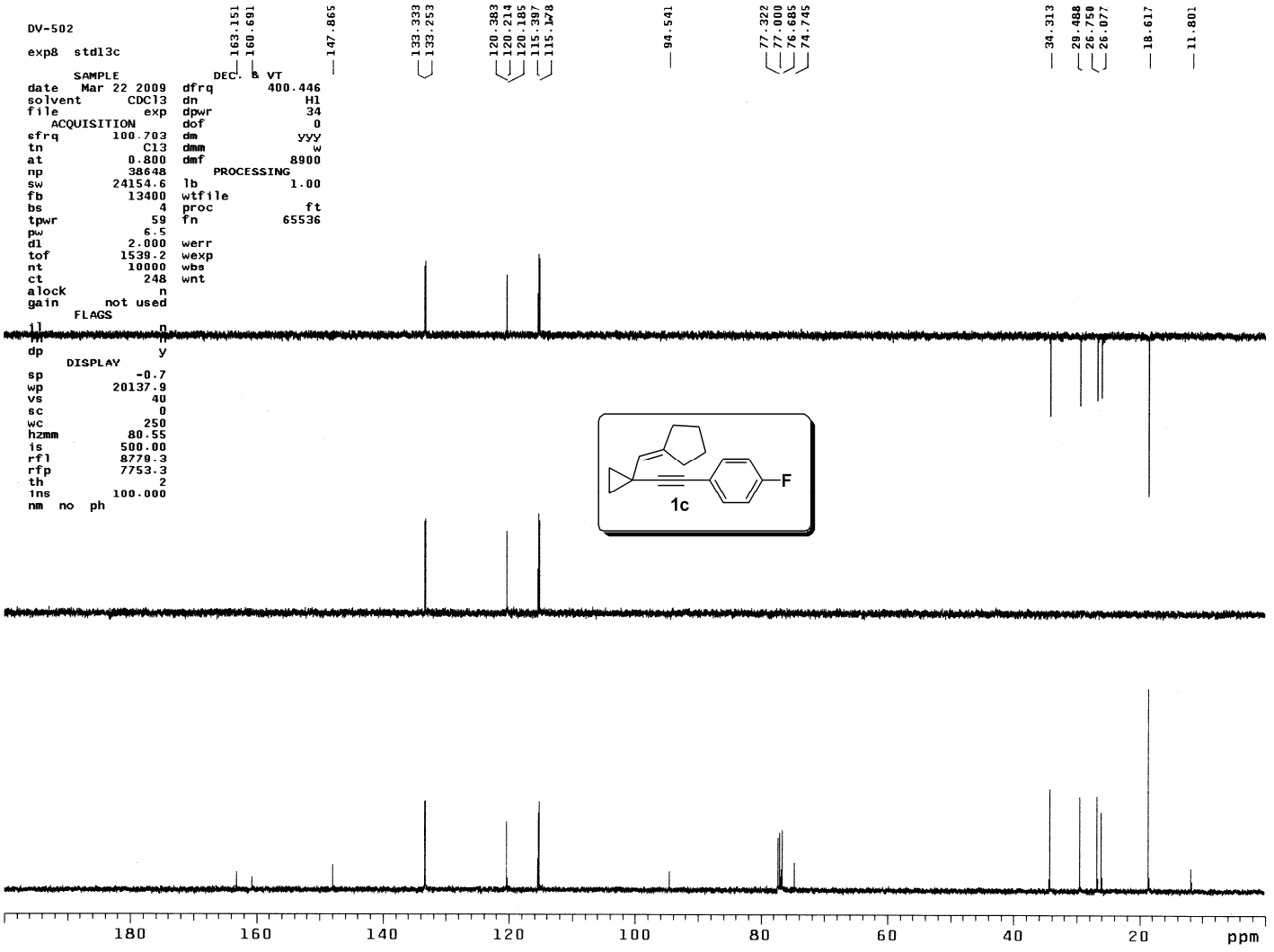
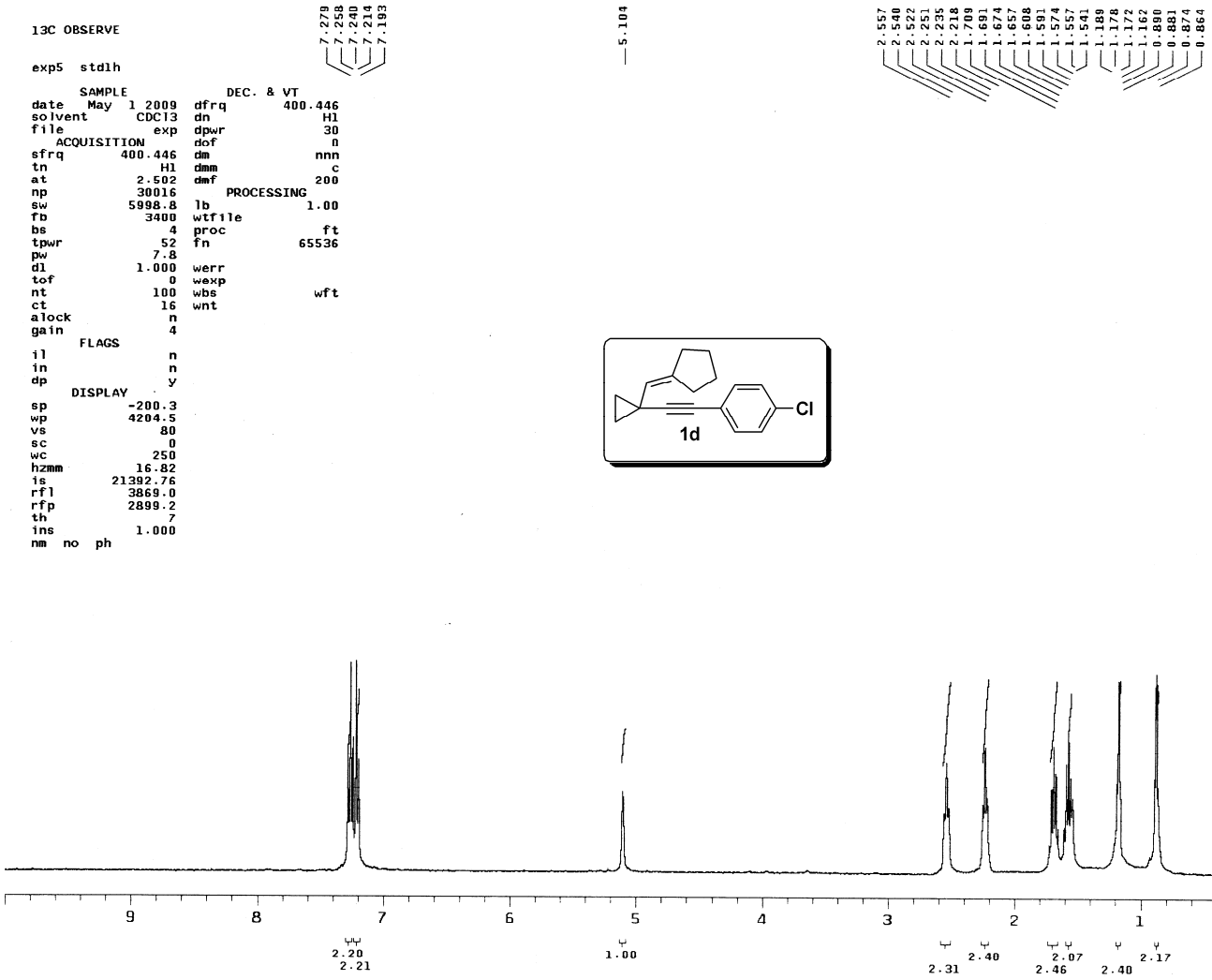
SAMPLE		DEC. & VT	
date	Mar 22 2009	dfrq	400.446
solvent	CDCl3	dn	H1
file	exp	dpwr	30
ACQUISITION		dof	0
sfrq	400.446	dm	nnn
tn	H1	dmm	c
at	2.502	dmf	200
np	30016	PROCESSING	
sw	5998.8	lb	1.00
fb	3400	wtfile	
bs	4	proc	ft
tpwr	52	fn	65536
pw	7.8		
dl	1.000	werr	
tof	0	wexp	
nt	100	wbs	wft
ct	52	wnt	
alock	n		
gain	4		
FLAGS			
il	n		
in	n		
dp	y		
DISPLAY			
sp	-200.3		
wp	4204.5		
vs	40		
sc	0		
wc	250		
hzmm	4.08		
is	500.00		
rfl	3869.2		
rfp	2899.2		
th	1		
ins	1.000		
nm no ph			

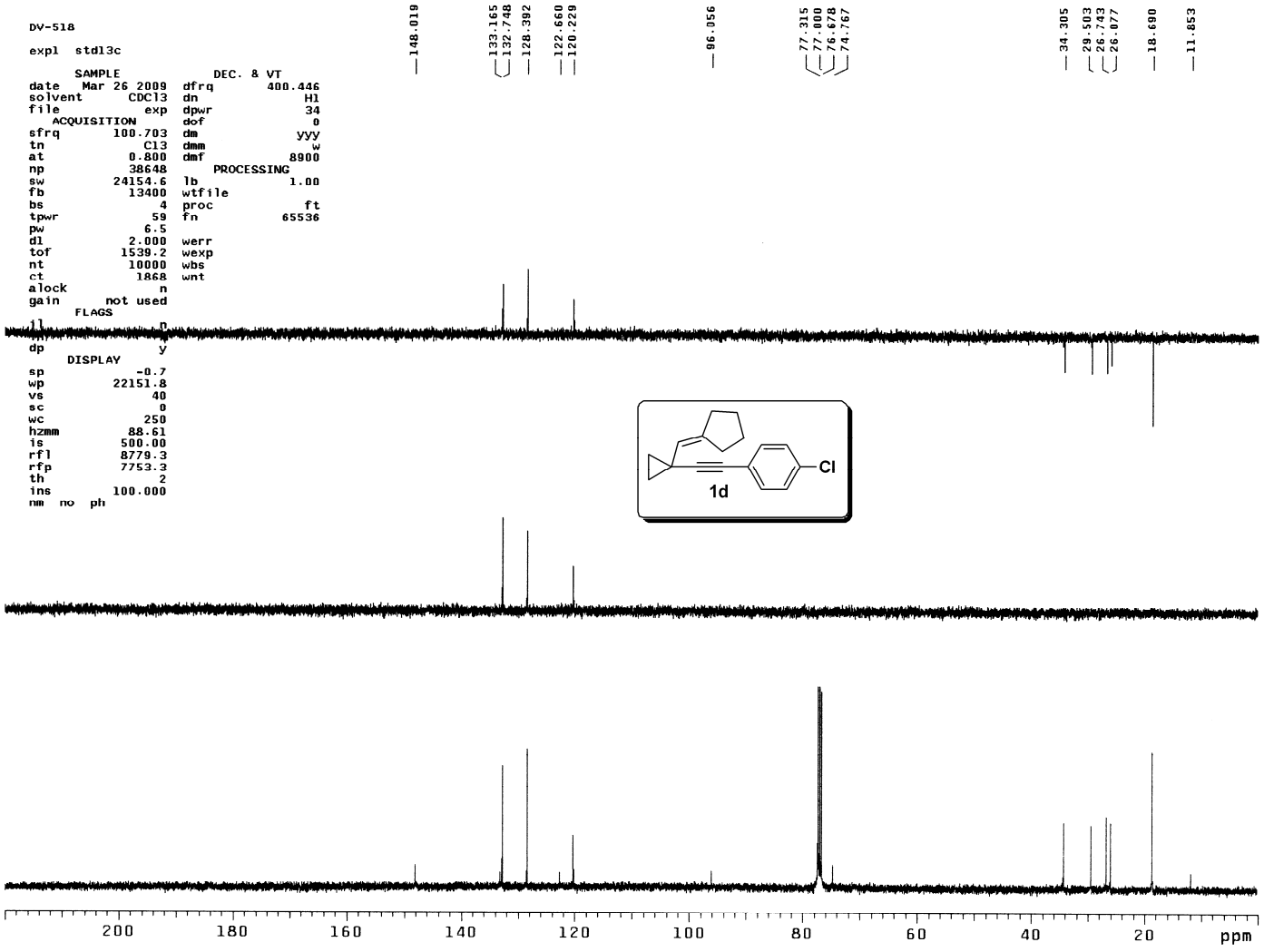


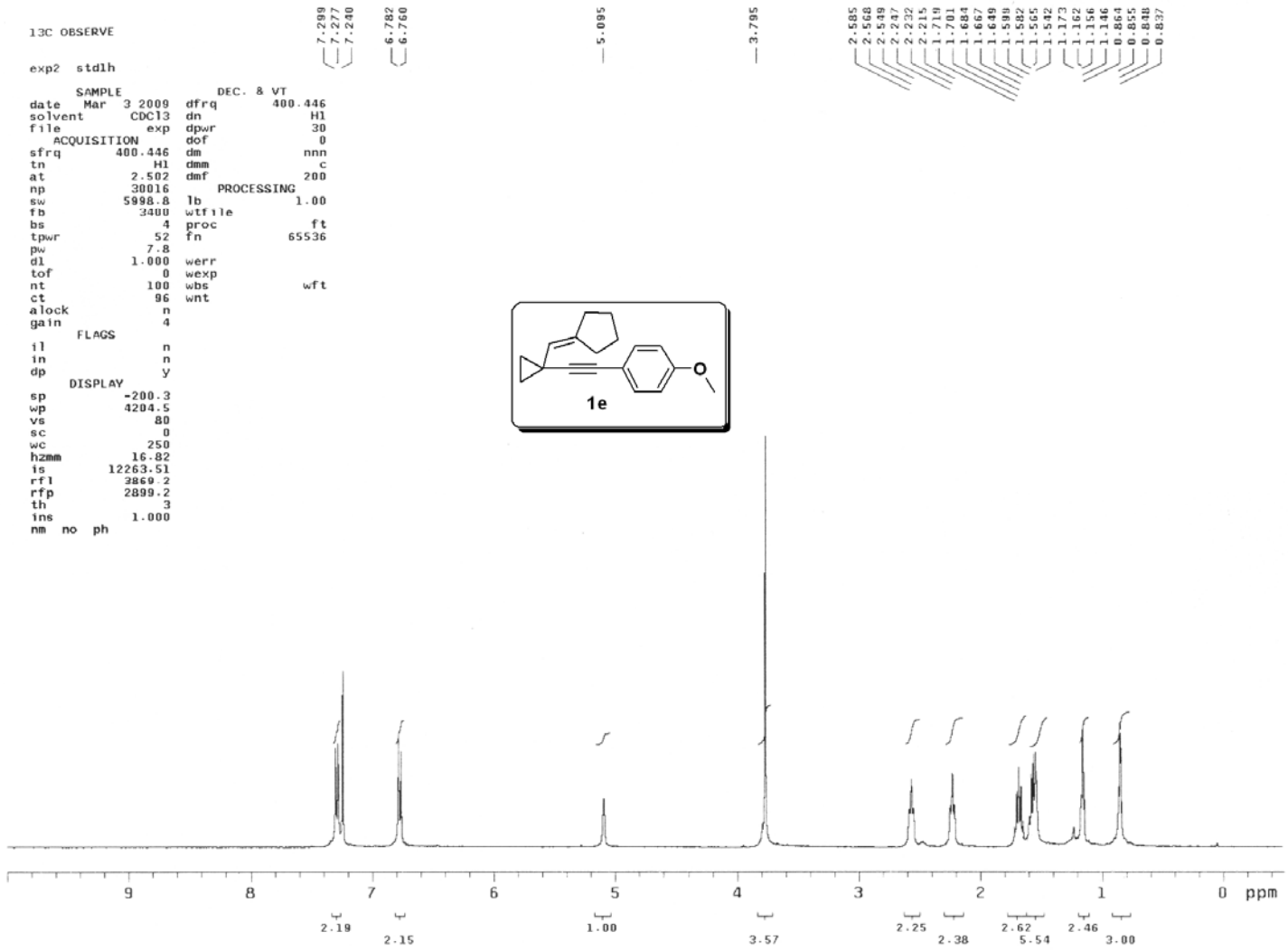
1c

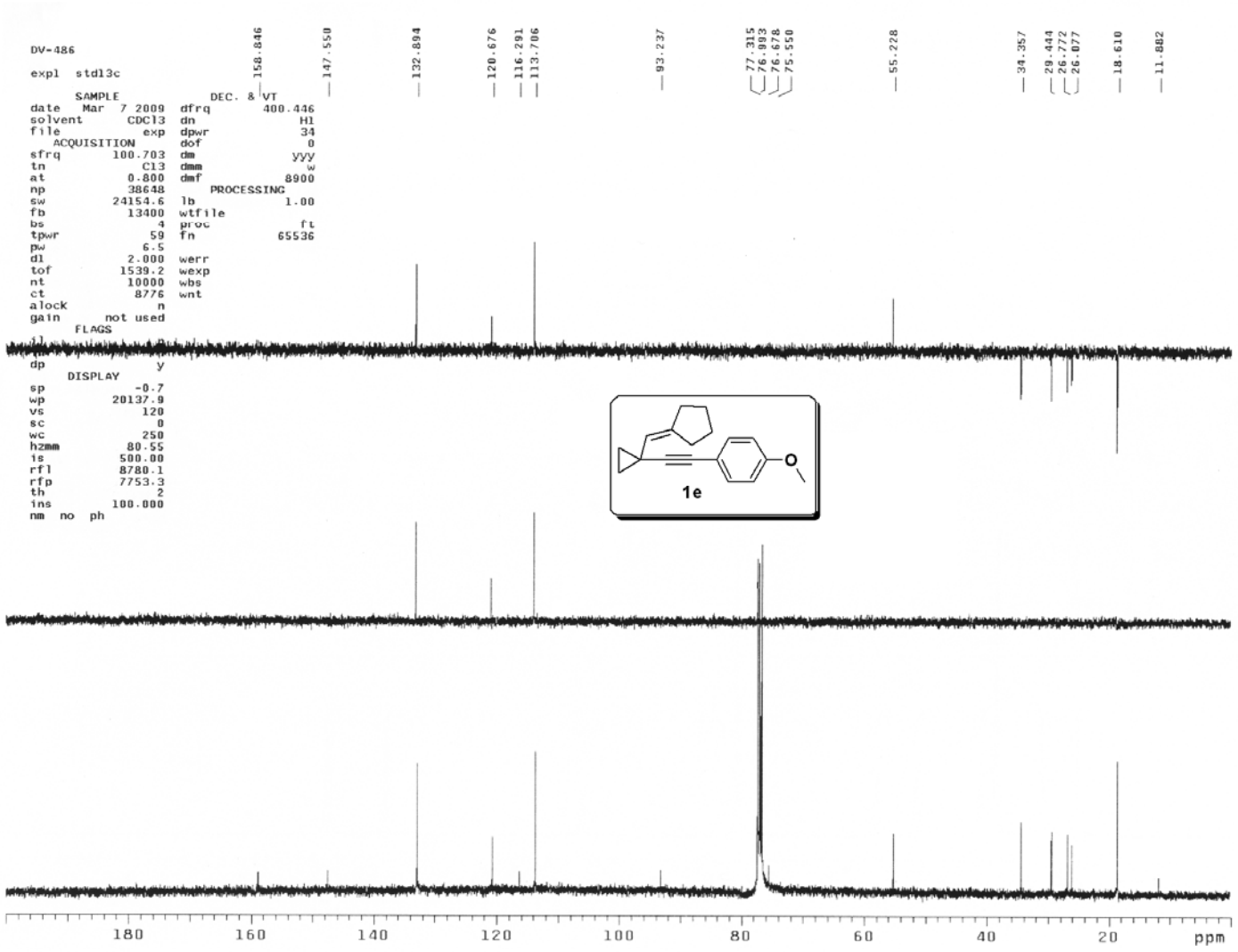


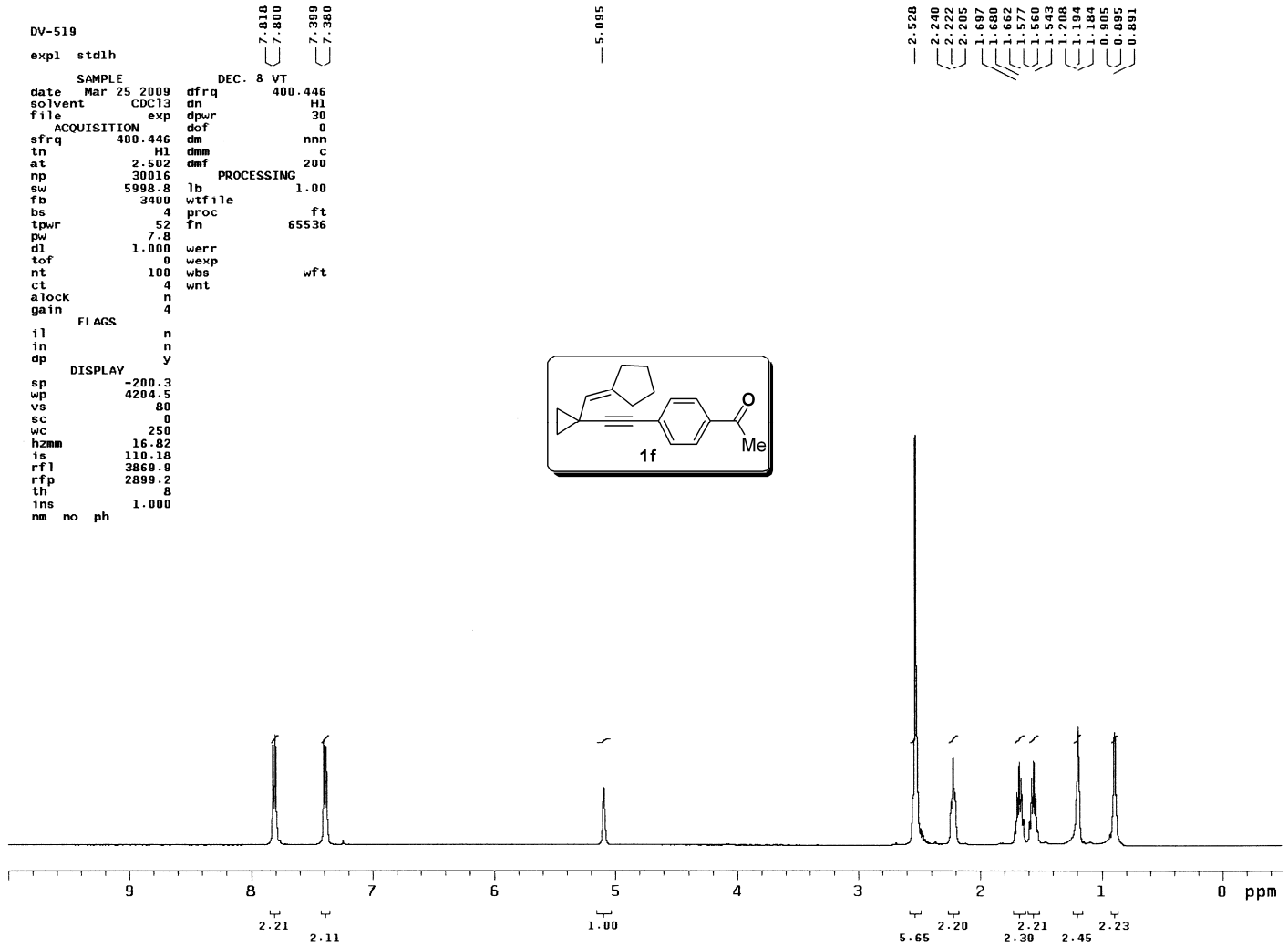

DV-502
exp8 stdl3c
SAMPLE
date Mar 22 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
dl 2.000
tof 1539.2
nt 10000
ct 248
alock n
gain not used
FLAGS
il n
dp y
DISPLAY
sp -0.7
wp 20137.9
vs 40
sc 0
wc 250
hzmm 80.55
is 500.00
rfl 8779.3
rfp 7753.3
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs
wnt
163.151
160.691
147.865
133.333
133.253
120.383
120.214
120.185
115.397
115.178
94.541
77.322
77.000
76.685
74.745
34.313
29.488
26.750
26.077
18.617
11.801
F
1c
180
160
140
120
100
80
60
40
20
ppm

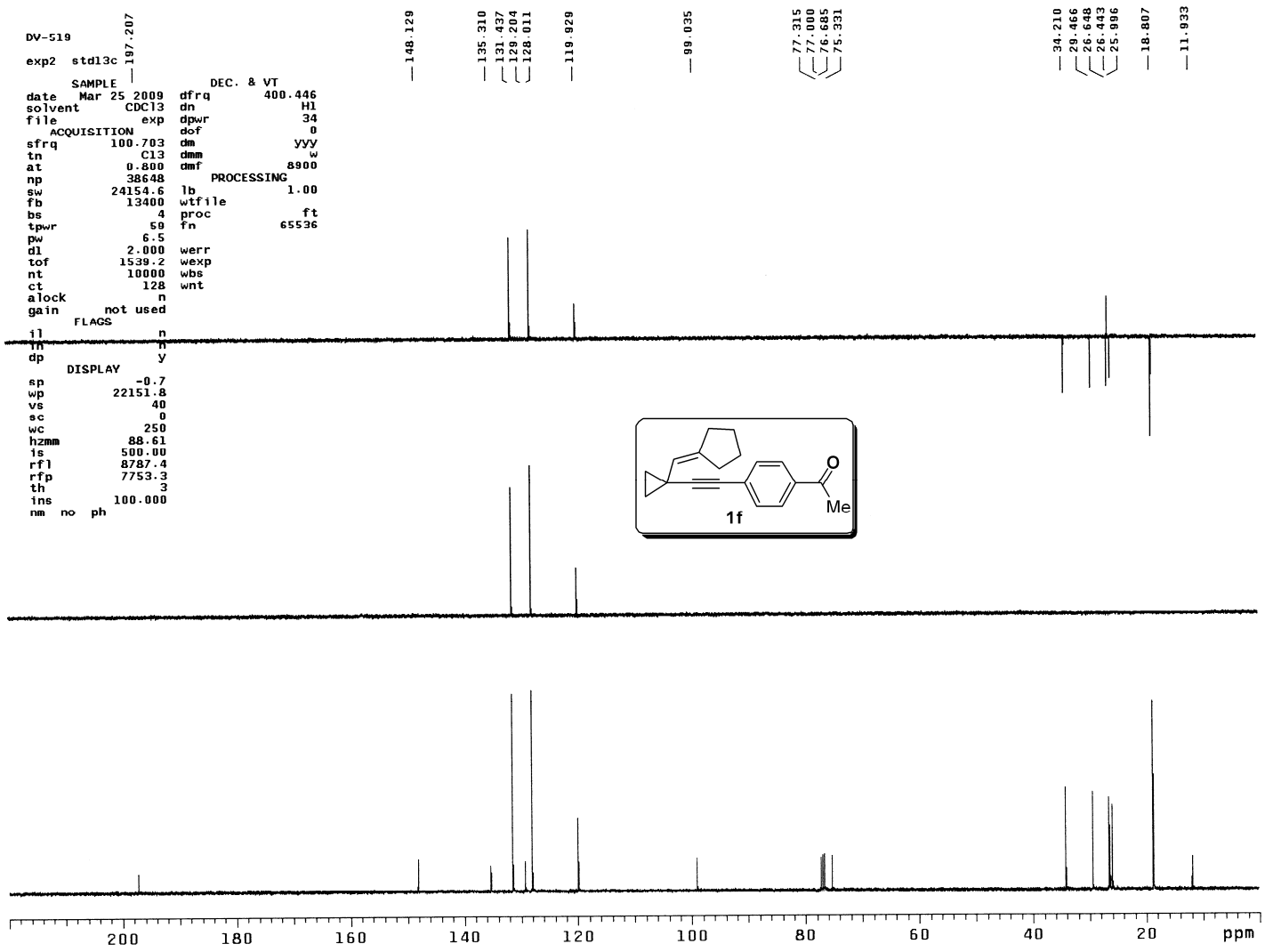

13C OBSERVE
exp5 stdlh
SAMPLE
date May 1 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
dl 1.000
tof 0
nt 100
ct 16
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -200.3
wp 4204.5
vs 80
sc 0
wc 250
hzmm 16.82
is 21392.76
rfl 3869.0
rfp 2899.2
th 7
ins 1.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs wft
wnt
7.279
7.258
7.240
7.214
7.193
5.104
2.557
2.540
2.522
2.251
2.235
2.218
1.709
1.691
1.674
1.657
1.608
1.591
1.574
1.557
1.541
1.189
1.178
1.172
1.162
0.890
0.881
0.874
0.864
Cl
1d
9
8
7
6
5
4
3
2
1
0
ppm
2.20
2.21
1.00
2.31
2.40
2.07
2.46
2.40
2.17

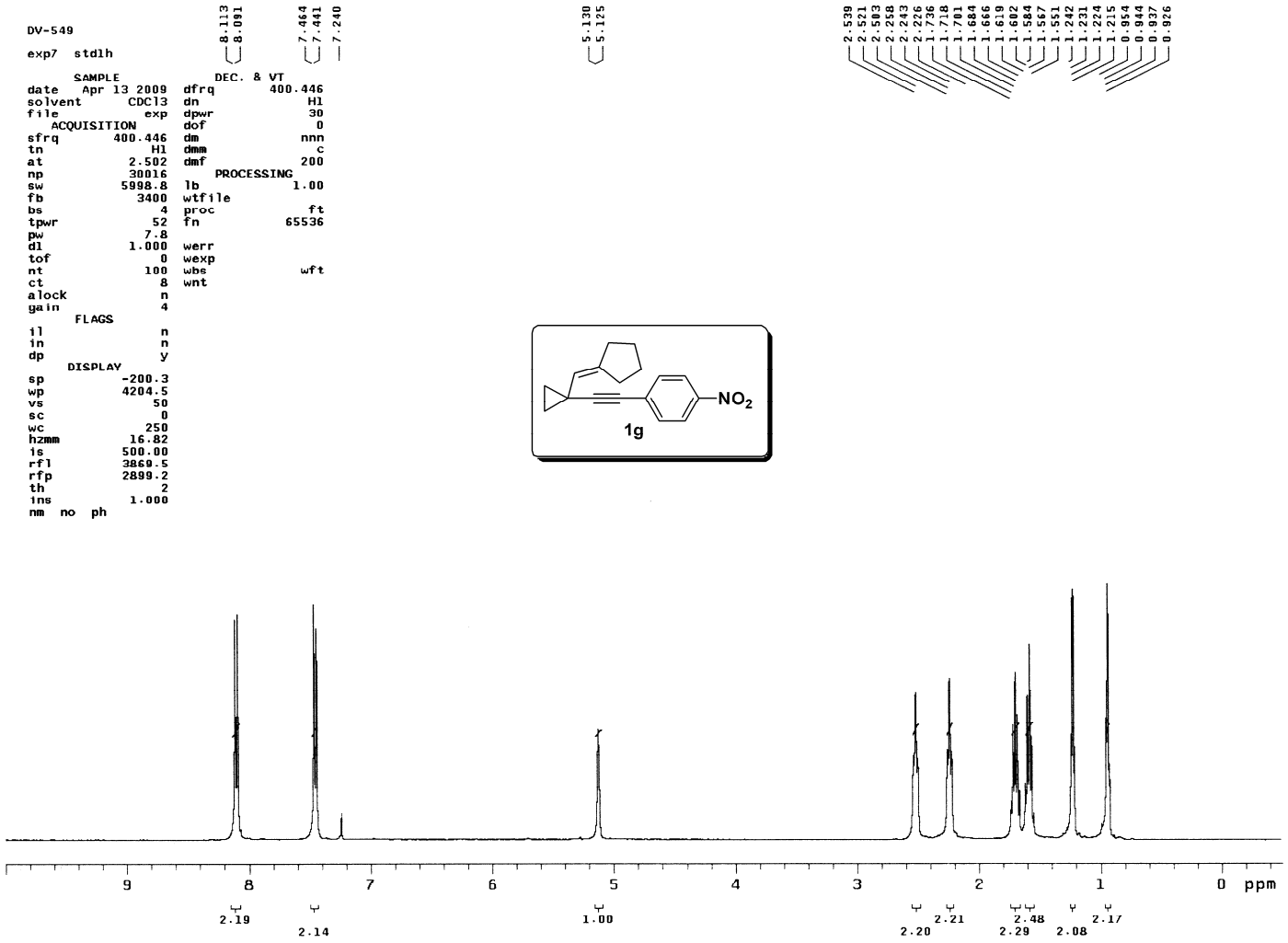


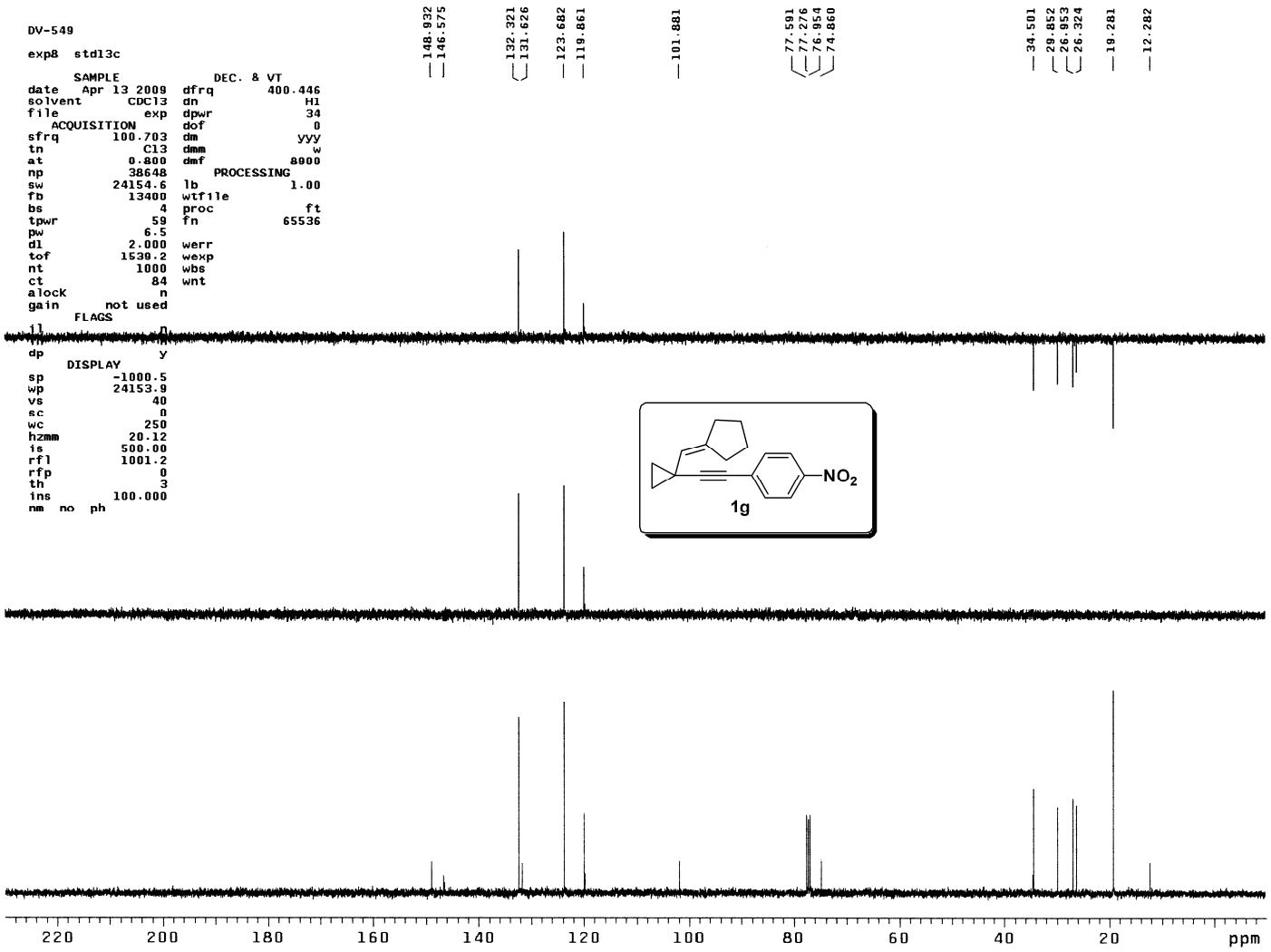


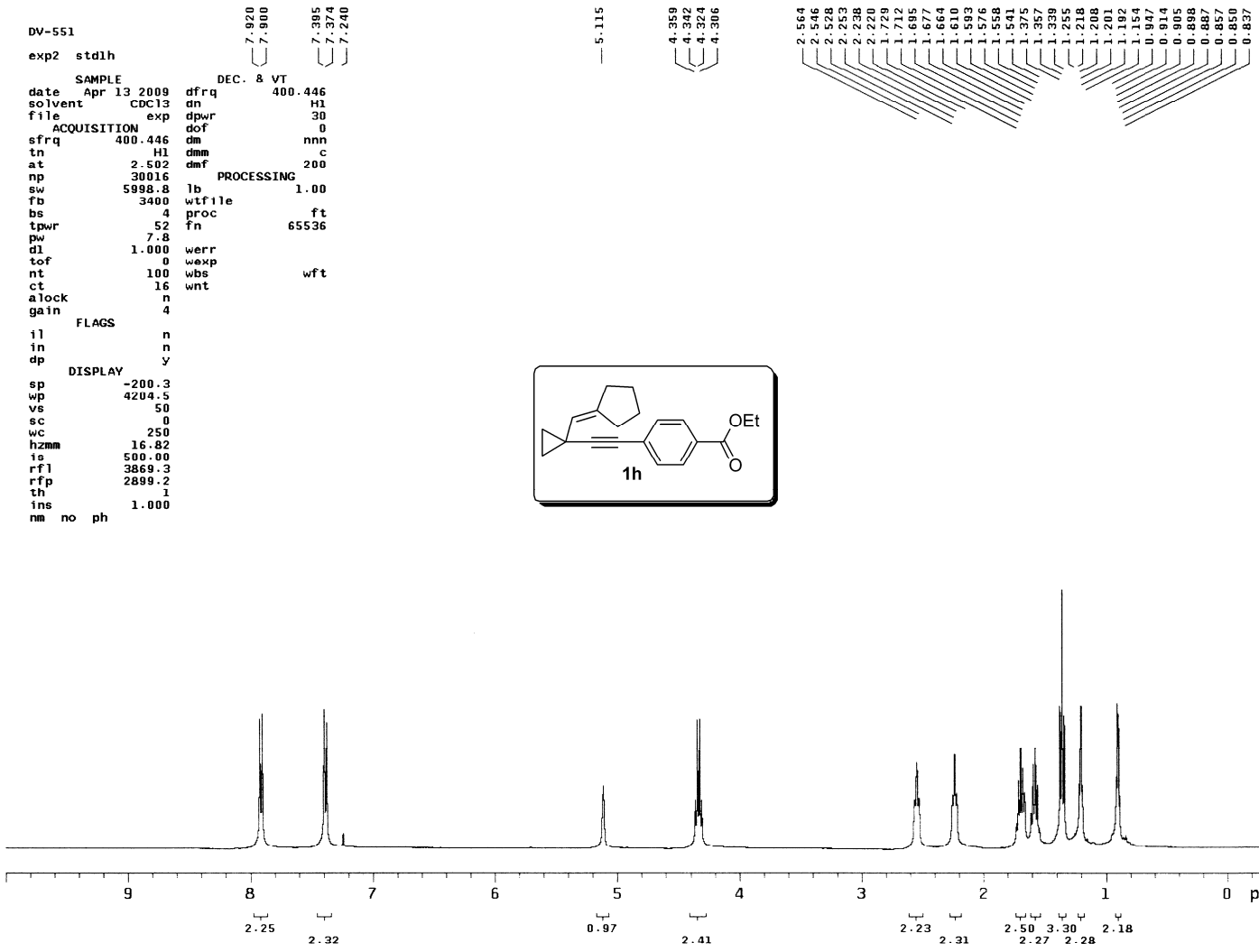

DV-486
expl stdl3c
SAMPLE
date Mar 7 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
dl 2.000
tof 1539.2
nt 10000
ct 8776
alock n
gain not used
FLAGS
il n
dp y
DISPLAY
sp -0.7
wp 20137.9
vs 120
sc 0
wc 250
hzmm 80.55
is 500.00
rfl 8780.1
rfp 7753.3
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs
wnt
158.846
147.550
132.894
120.676
116.291
113.706
93.237
77.315
76.993
76.678
75.550
55.228
34.357
29.444
26.772
26.077
18.610
11.882
O
1e
180
160
140
120
100
80
60
40
20
ppm

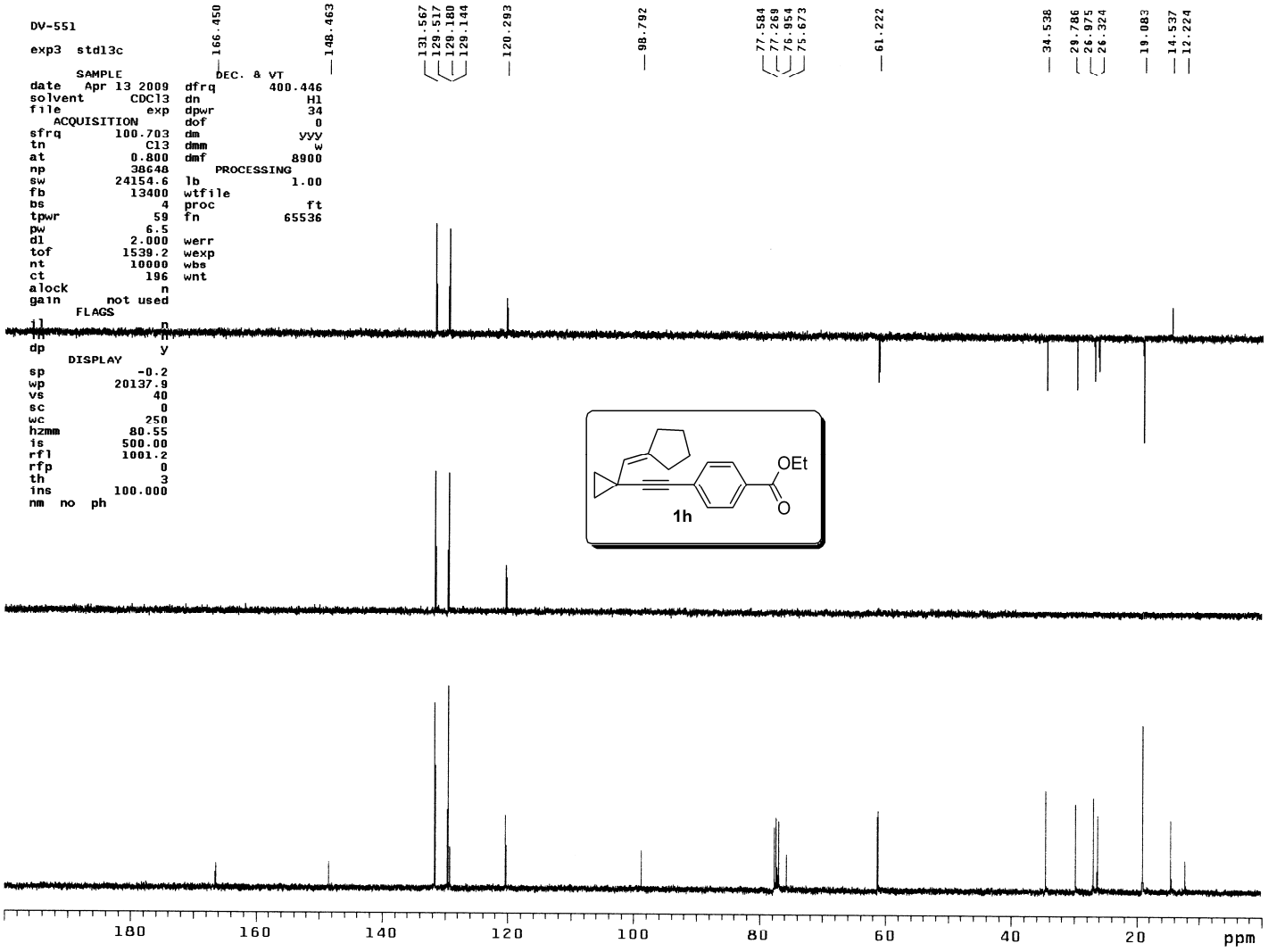


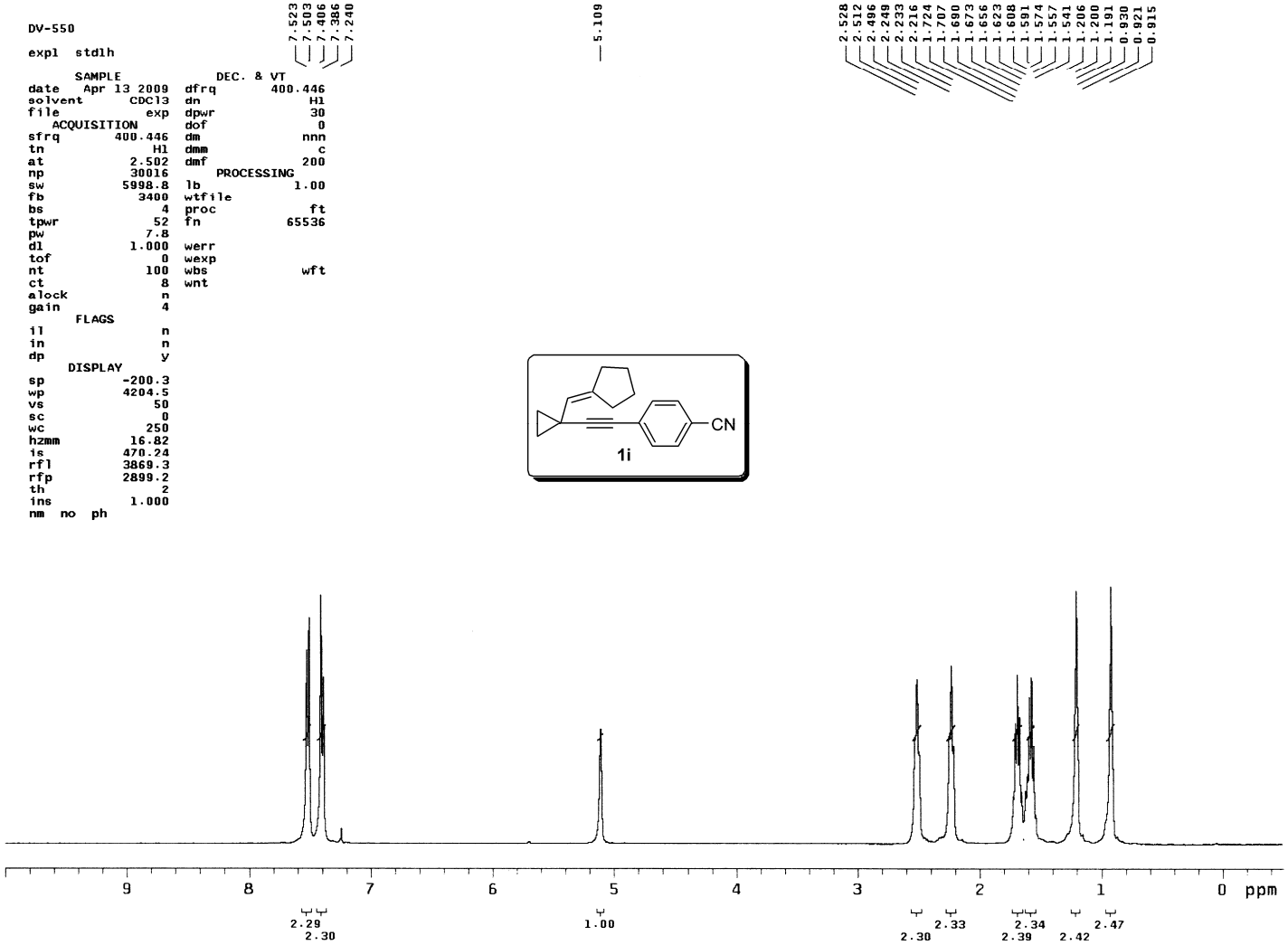


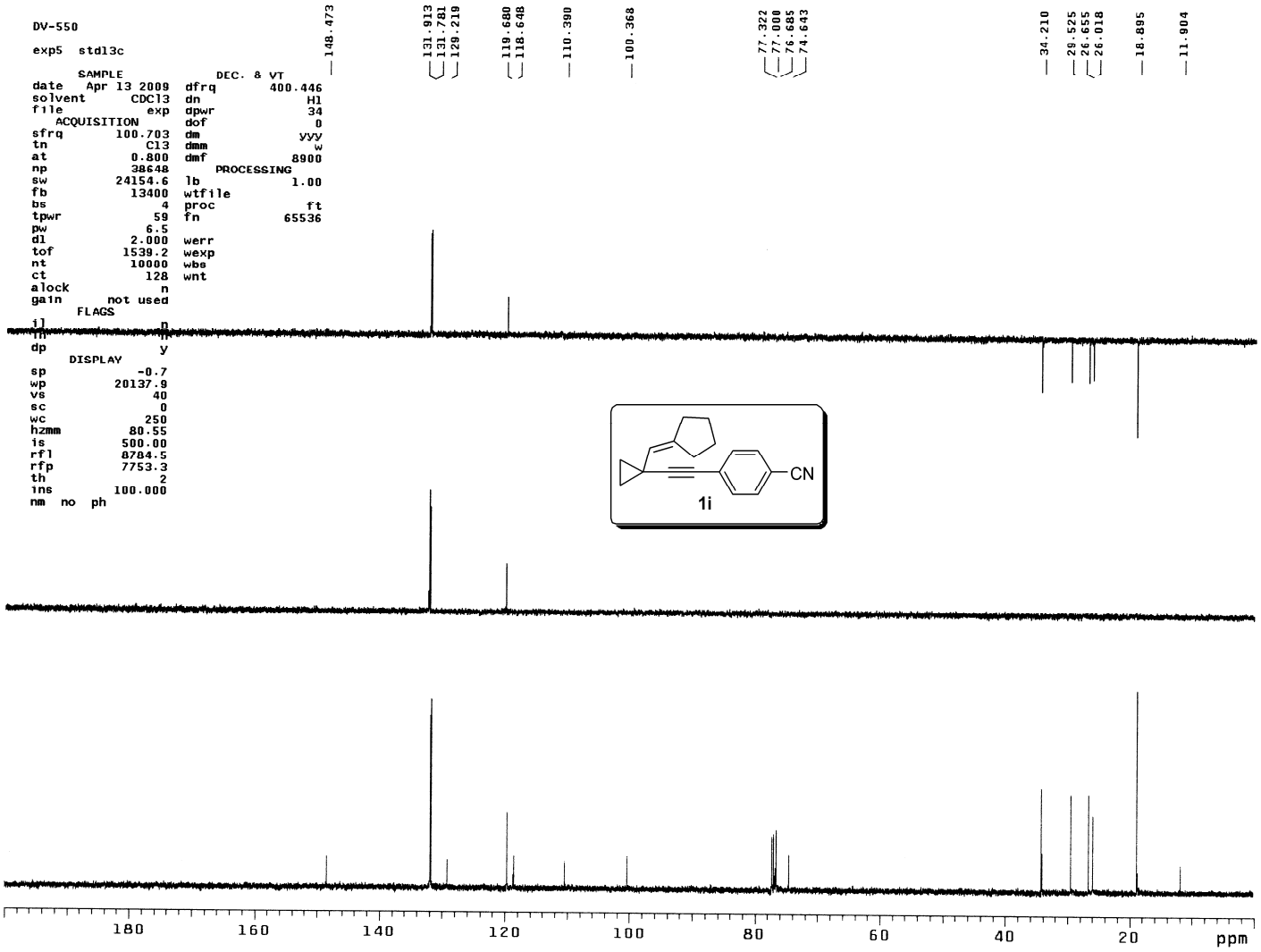

DV-549
exp7 stdlh
SAMPLE
date Apr 13 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
dl 1.000
tof 0
nt 100
ct 8
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -200.3
wp 4204.5
vs 50
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3869.5
rfp 2899.2
th 2
ins 1.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs wft
wnt
8.113
8.091
7.464
7.441
7.240
5.130
5.125
2.539
2.521
2.503
2.258
2.243
2.226
1.736
1.718
1.701
1.684
1.666
1.619
1.602
1.584
1.567
1.551
1.242
1.231
1.224
1.215
0.954
0.944
0.937
0.926
NO2
1g
9
8
7
6
5
4
3
2
1
0 ppm
2.19
2.14
1.00
2.20
2.21
2.48
2.29
2.08
2.17

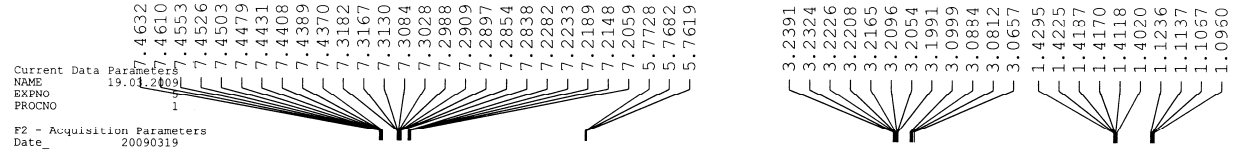



DV-551
exp2 stdlh
SAMPLE
date Apr 13 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
d1 1.000
tof 0
nt 100
ct 16
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -200.3
wp 4204.5
vs 50
sc 0
wc 250
hzmm 16.82
is 500.00
rfl 3869.3
rfp 2899.2
th 1
ins 1.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs wft
wnt
7.920
7.900
7.395
7.374
7.240
5.115
4.359
4.342
4.324
4.306
2.564
2.546
2.528
2.253
2.238
2.220
1.729
1.712
1.695
1.677
1.664
1.610
1.593
1.576
1.558
1.541
1.375
1.357
1.339
1.255
1.218
1.208
1.201
1.192
1.154
0.947
0.914
0.905
0.898
0.887
0.857
0.850
0.837
OEt
O
1h
9
8
7
6
5
4
3
2
1
0
ppm
2.25
2.32
0.97
2.41
2.23
2.31
2.50
2.27
3.30
2.28
2.18

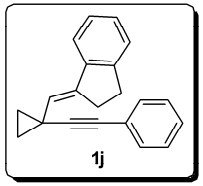


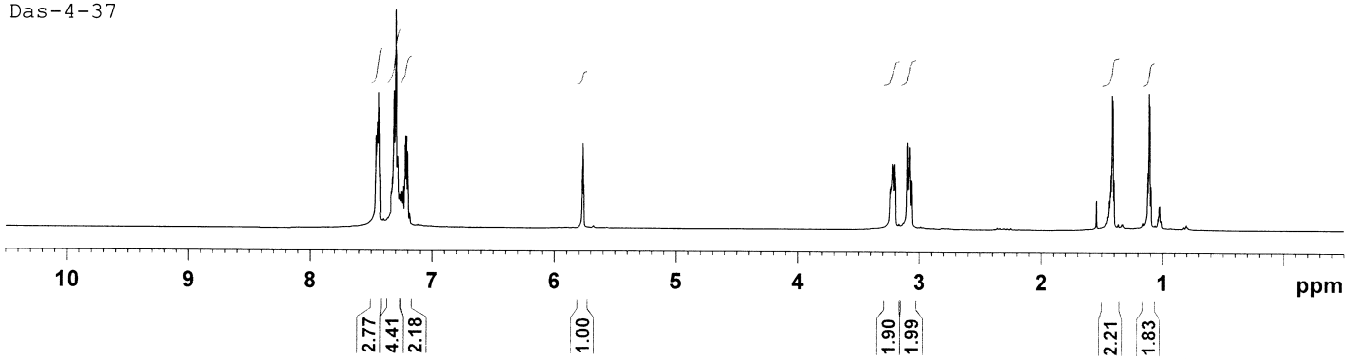


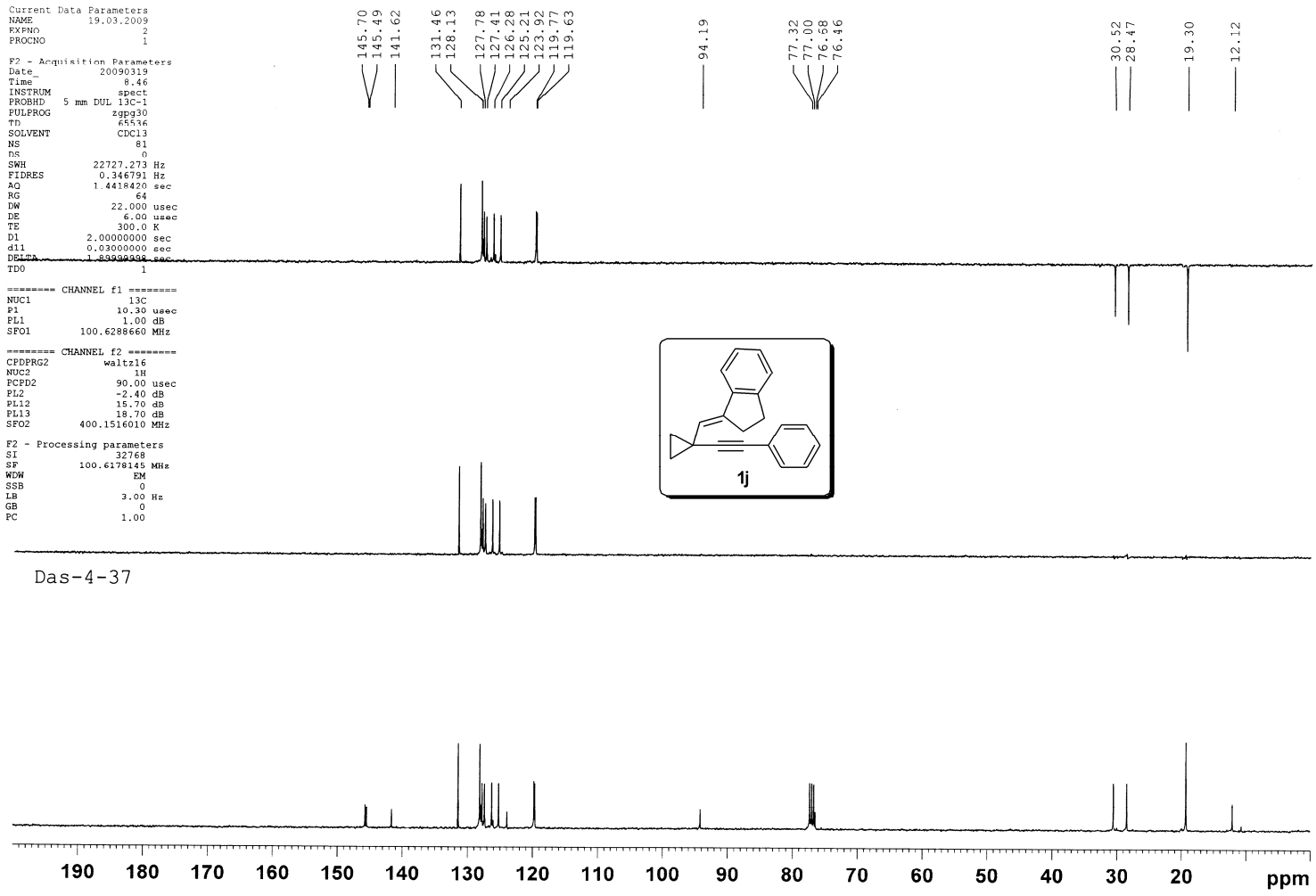

148.473
131.913
131.781
129.219
119.680
118.648
110.390
100.368
77.322
77.000
76.685
74.643
34.210
29.525
26.655
26.018
18.895
11.904
DV-550
exp5 std13c
SAMPLE
date Apr 13 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
d1 2.000
tof 1539.2
nt 10000
ct 128
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.7
wp 20137.9
vs 40
sc 0
wc 250
hzmm 80.55
is 500.00
rfl 8784.5
rfp 7753.3
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs
wnt
CN
1i
180
160
140
120
100
80
60
40
20
ppm

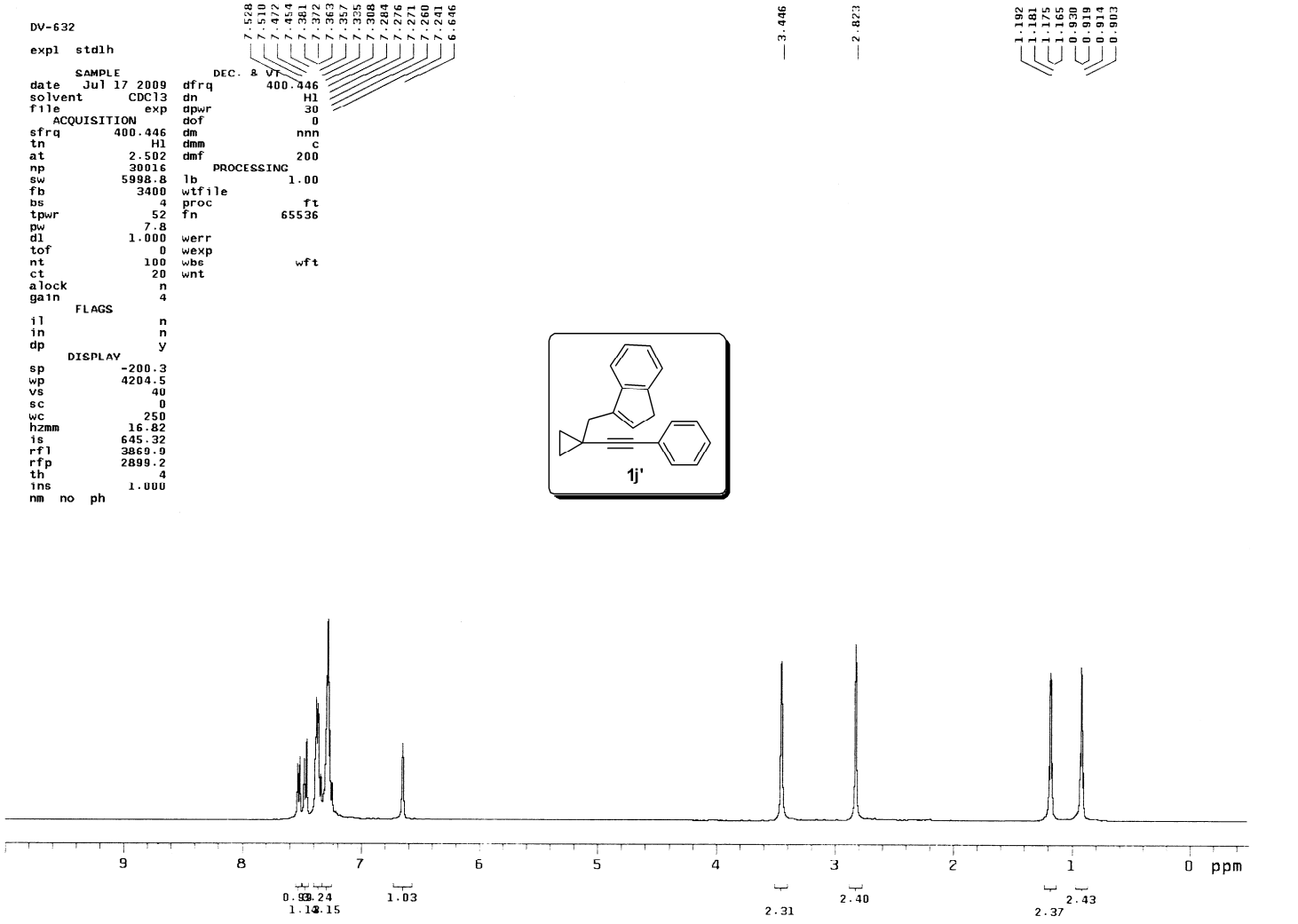


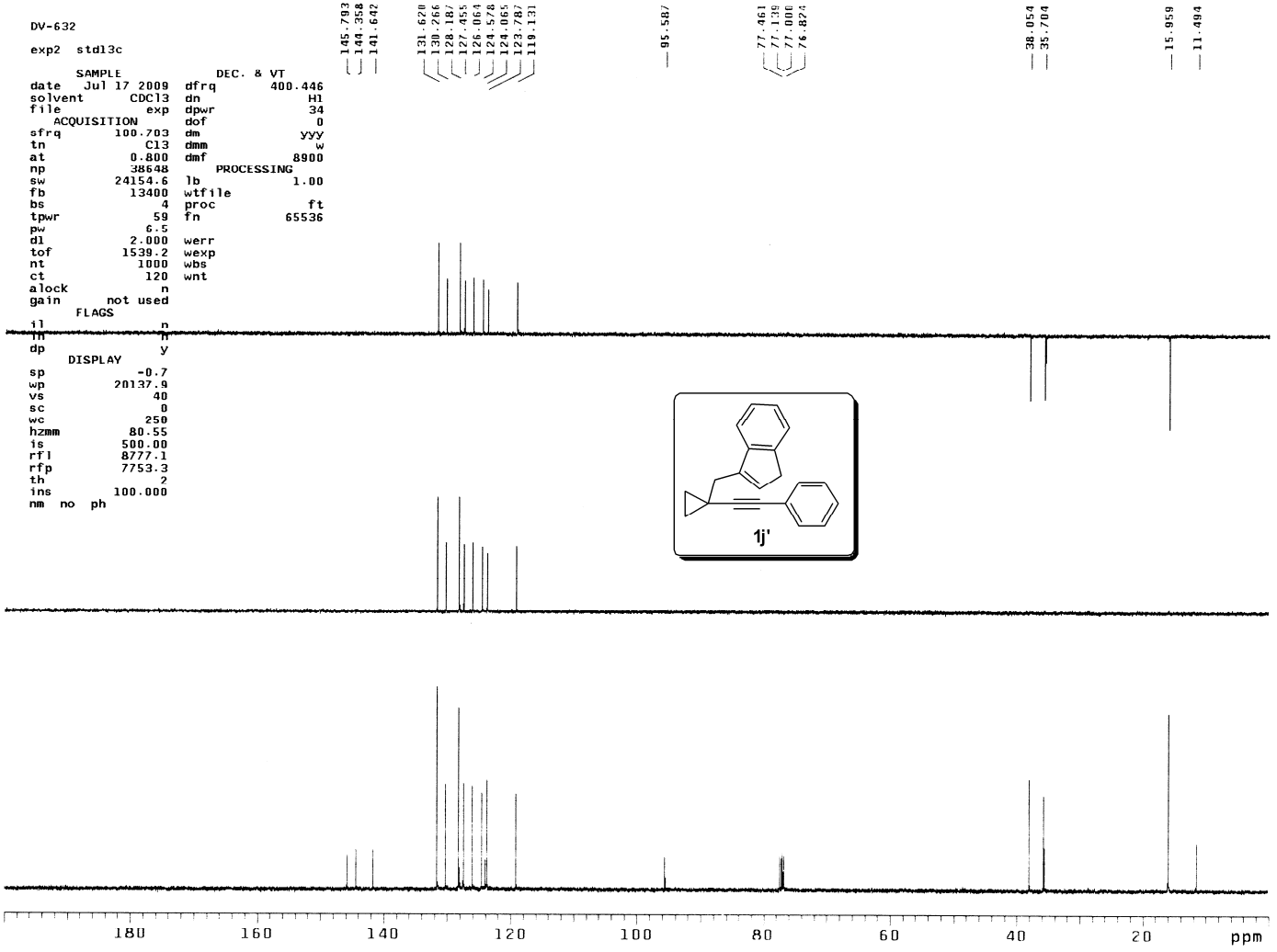

```
Current Data Parameters
NAME          19.01.2009
EXPNO                  5
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090319
Time               8.57
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   24
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ            2.5559540 sec
RG                 45.2
DW               78.000 usec
DE                6.00 usec
TE               300.0 K
D1            2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500165 MHz
WDW                  EM
SSB                   0
LB                0.00 Hz
GB                    0
PC                1.00
```

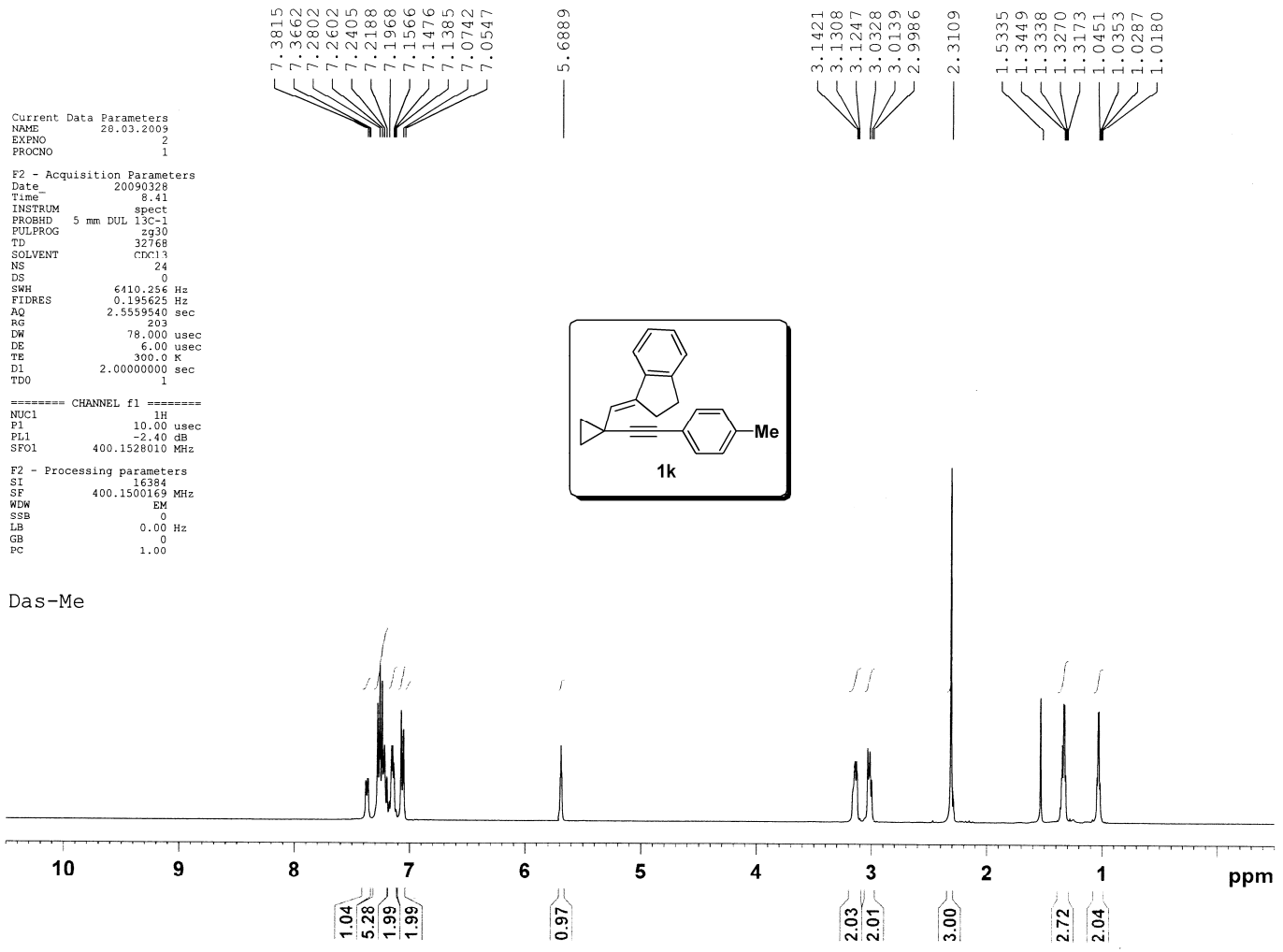


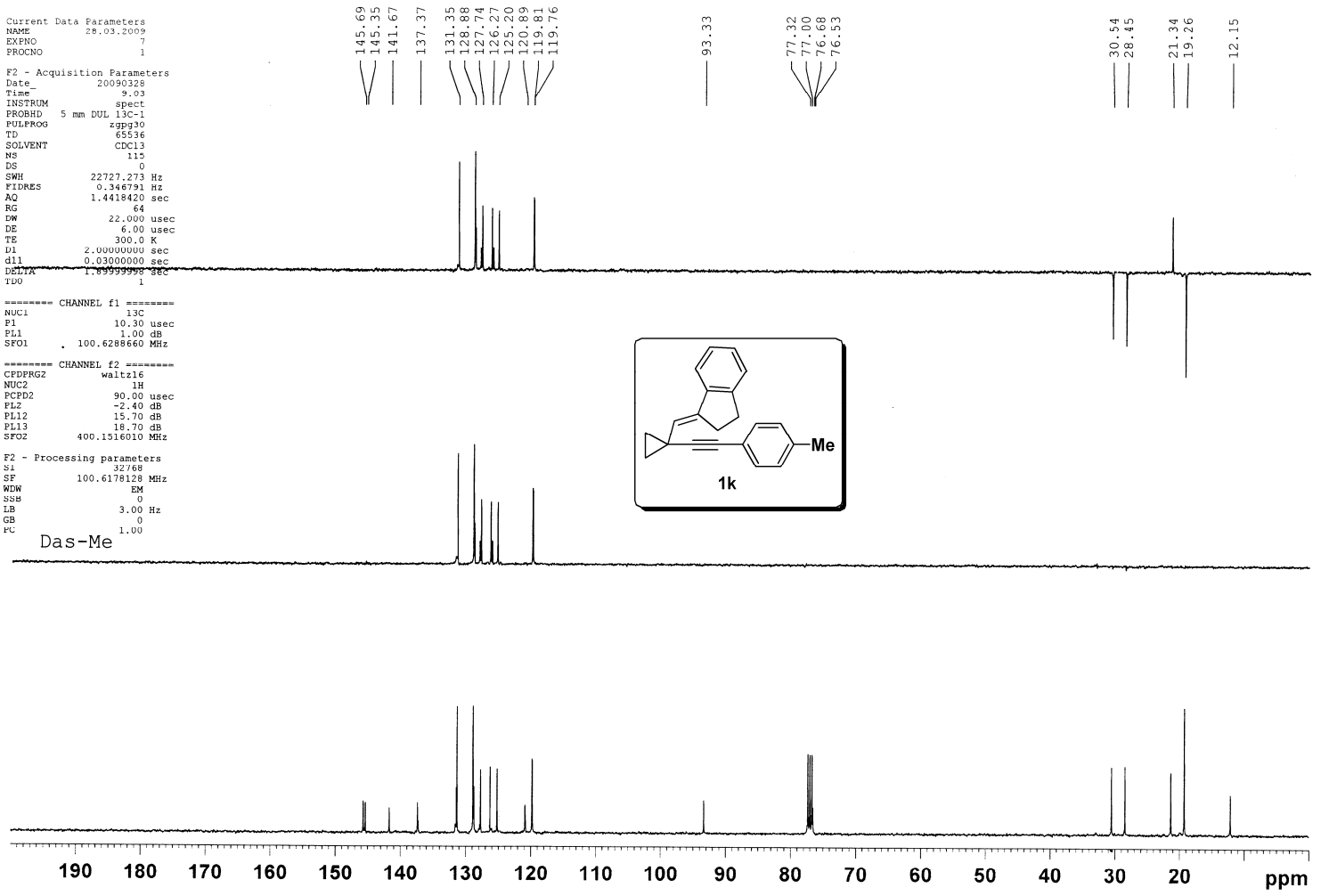


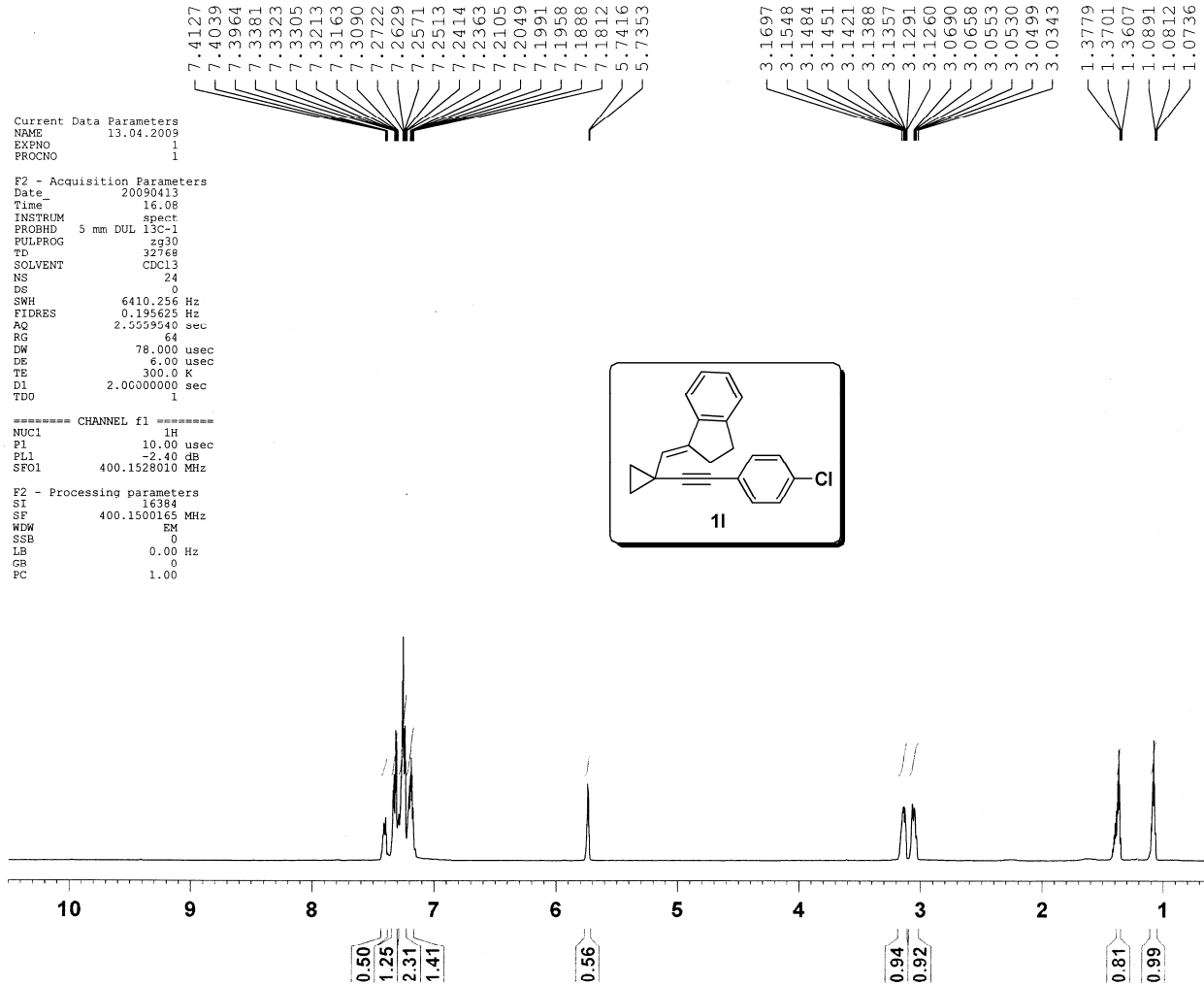


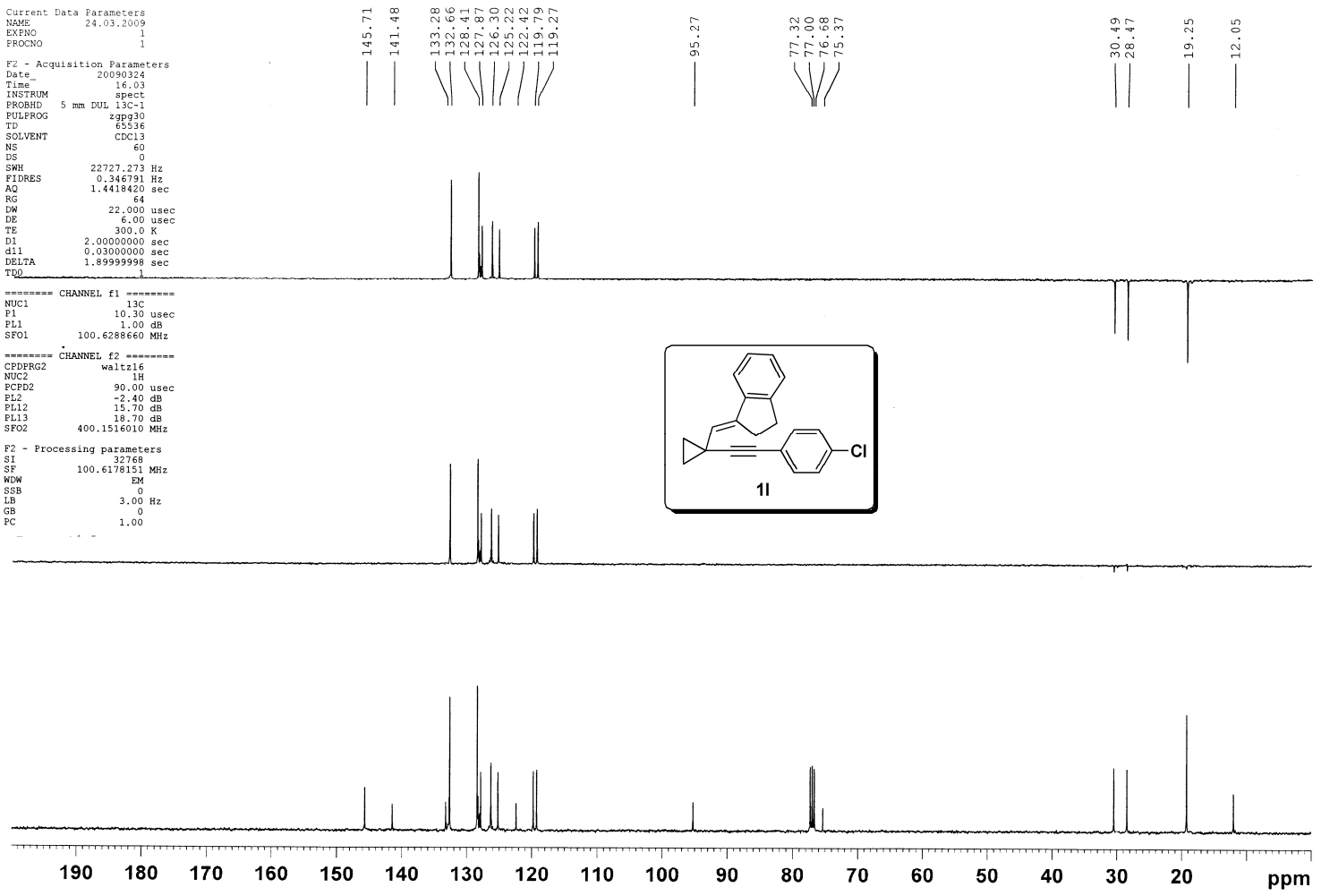

DV-632
expl stdlh
SAMPLE
date Jul 17 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 400.446
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
dl 1.000
tof 0
nt 100
ct 20
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -200.3
wp 4204.5
vs 40
sc 0
wc 250
hzmm 16.82
is 645.32
rfl 3869.9
rfp 2899.2
th 4
ins 1.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs wft
wnt
7.528
7.510
7.472
7.454
7.381
7.372
7.363
7.357
7.335
7.308
7.284
7.276
7.271
7.260
7.241
6.646
3.446
2.823
1.192
1.181
1.175
1.165
0.930
0.919
0.914
0.903
1j'
9
8
7
6
5
4
3
2
1
0 ppm
0.93
1.14
1.24
1.15
1.03
2.31
2.40
2.37
2.43




7.3815
7.3662
7.2802
7.2602
7.2405
7.2188
7.1968
7.1566
7.1476
7.1385
7.0742
7.0547
5.6889
3.1421
3.1308
3.1247
3.0328
3.0139
2.9986
2.3109
1.5335
1.3449
1.3338
1.3270
1.3173
1.0451
1.0353
1.0287
1.0180
Current Data Parameters
NAME 28.03.2009
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090328
Time 8.41
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 24
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 203
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500169 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
Me
1k
Das-Me
10
9
8
7
6
5
4
3
2
1
ppm
1.04
5.28
1.99
1.99
0.97
2.03
2.01
3.00
2.72
2.04







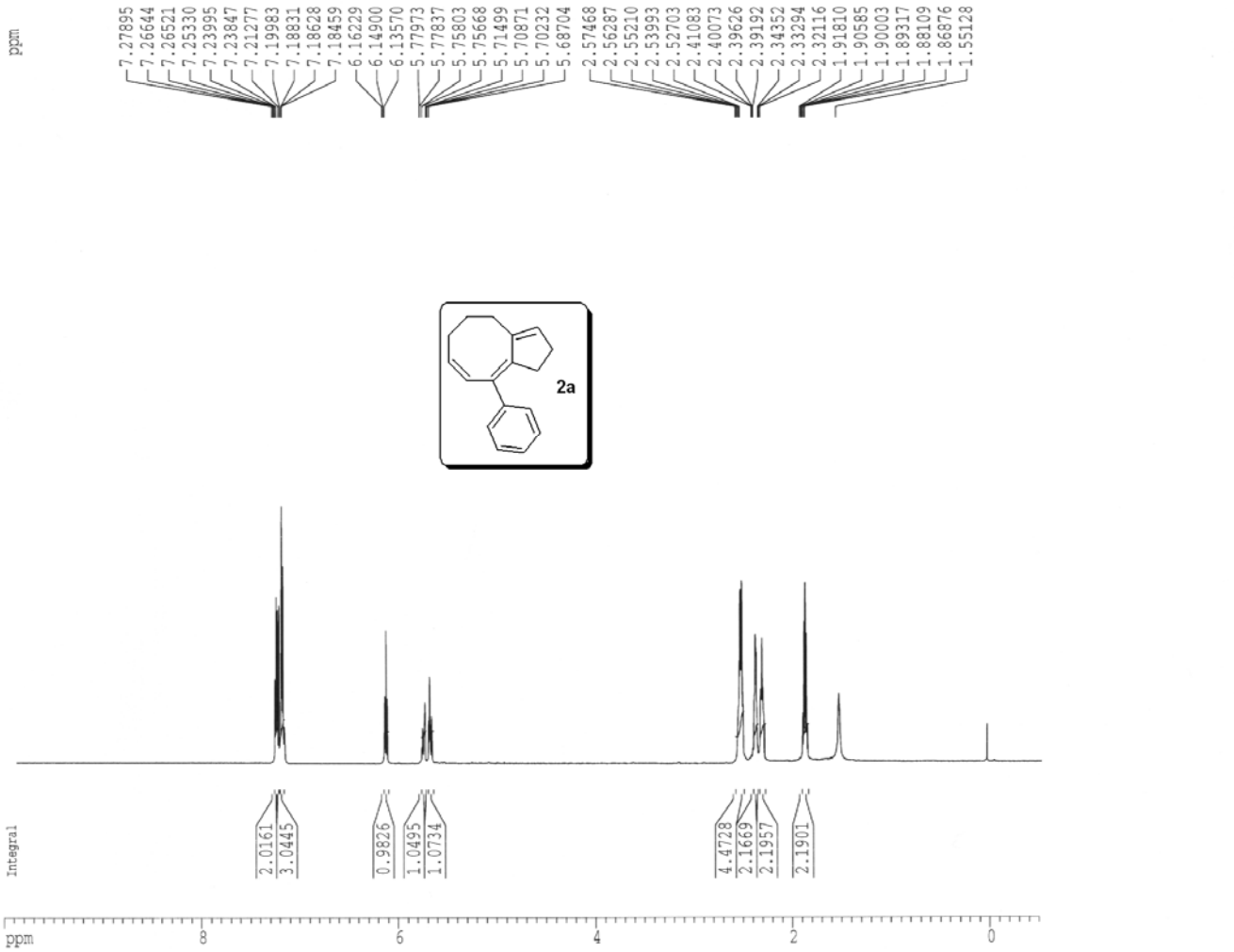
Current Data Parameters
NAME DV-451
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20090303
Time 18.40
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256023 Hz
AQ 1.9530228 sec
RG 256
DW 59.600 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

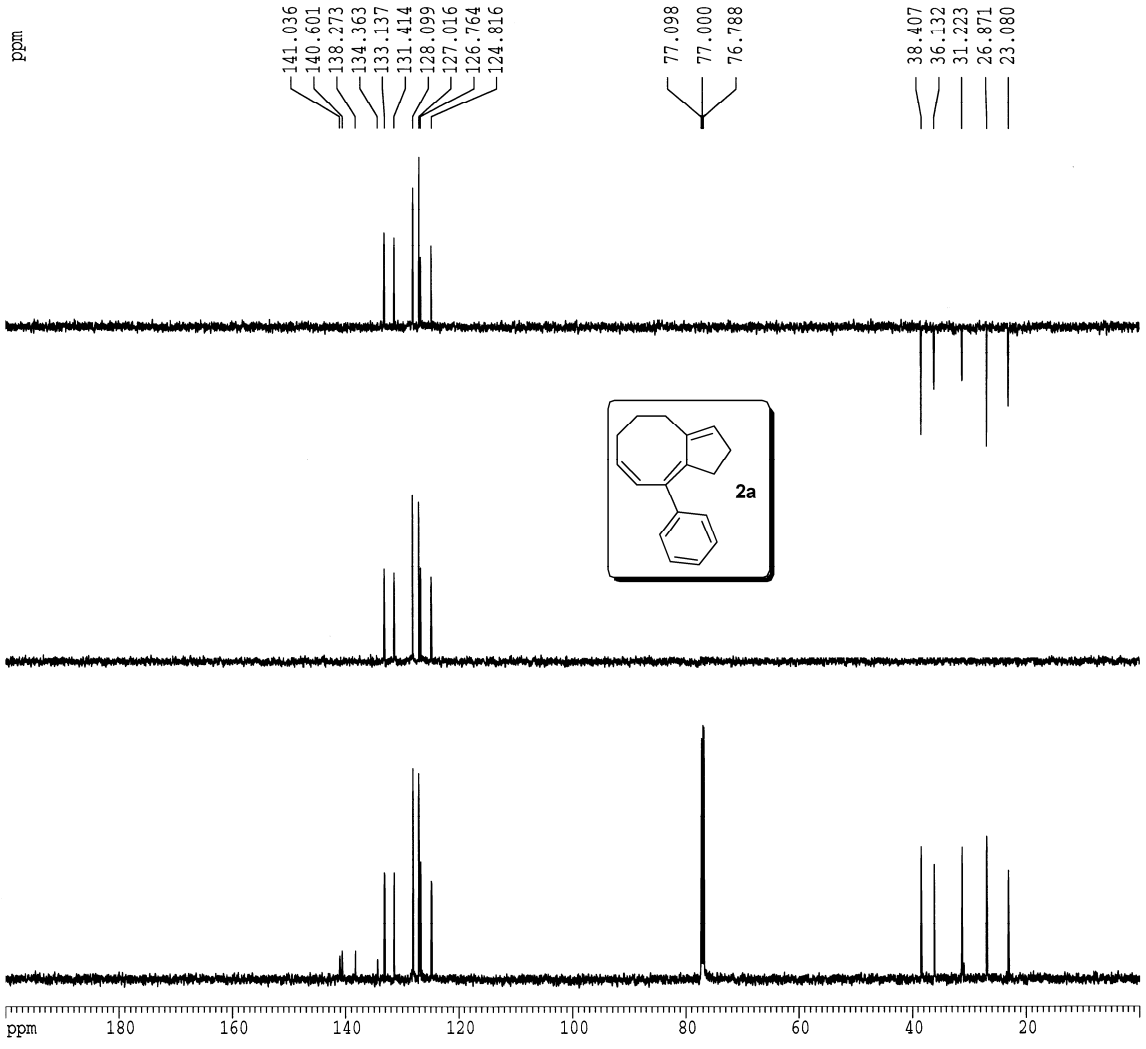
======== CHANNEL f1 ========
NUC1 1H
P1 12.50 usec
PL1 -5.00 dB
SFO1 595.2029960 MHz

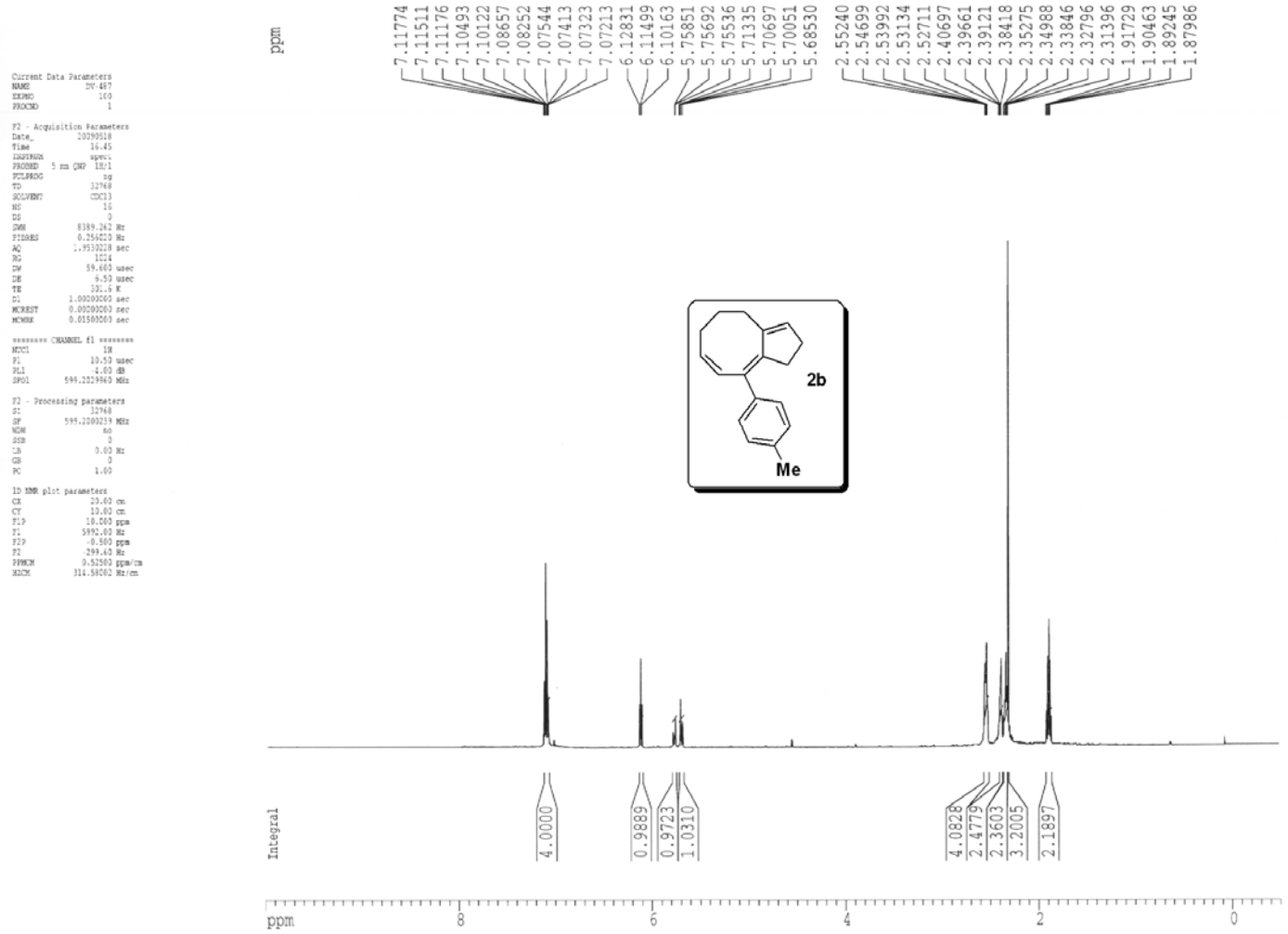
F2 - Processing parameters
SI 32768
SF 595.2000260 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00

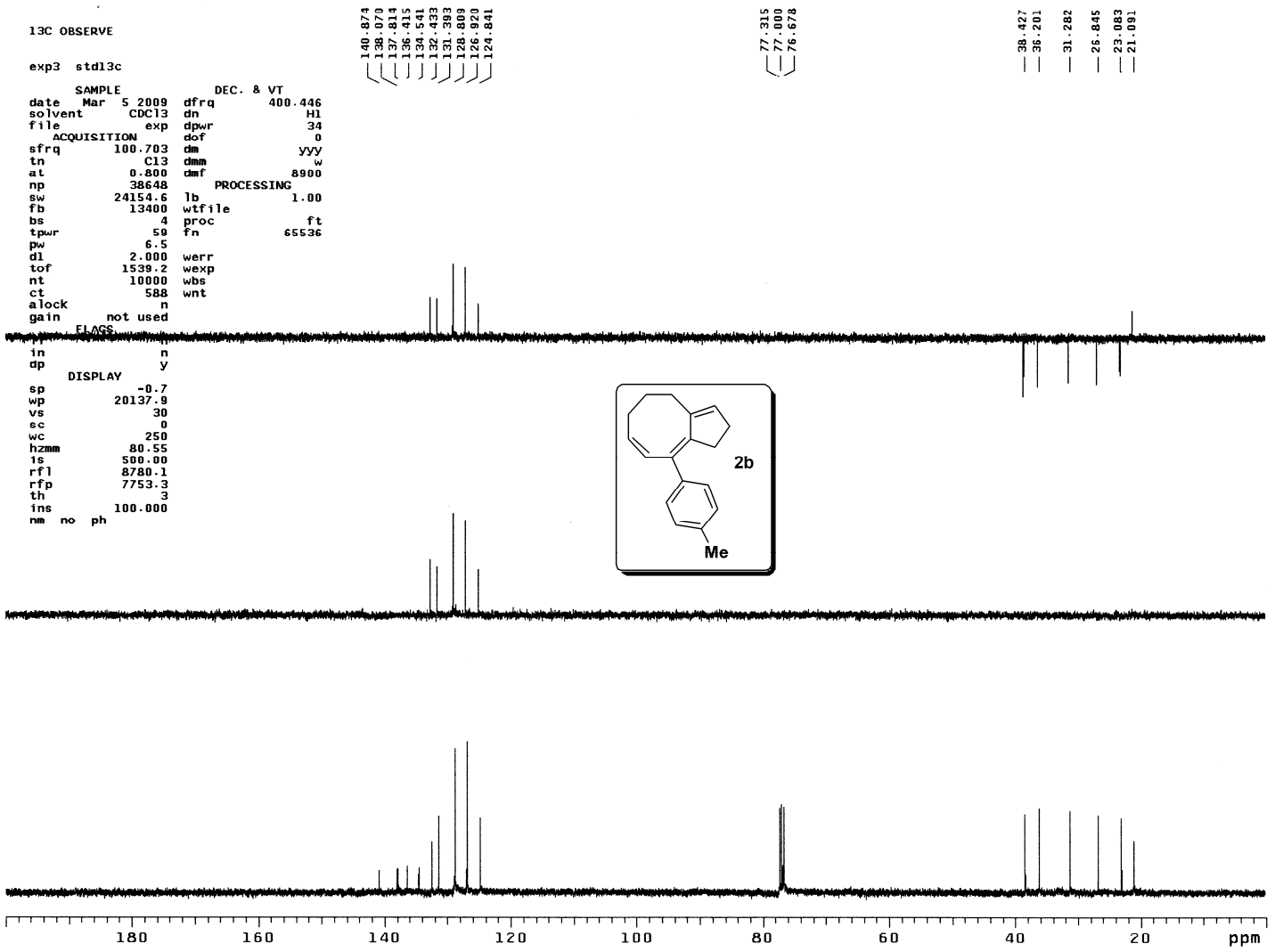
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 10.000 ppm
F1 5952.00 Hz
F2P -0.500 ppm
F2 -299.60 Hz
PPMCM 0.52500 ppm/cm
HZCM 314.58000 Hz/cm

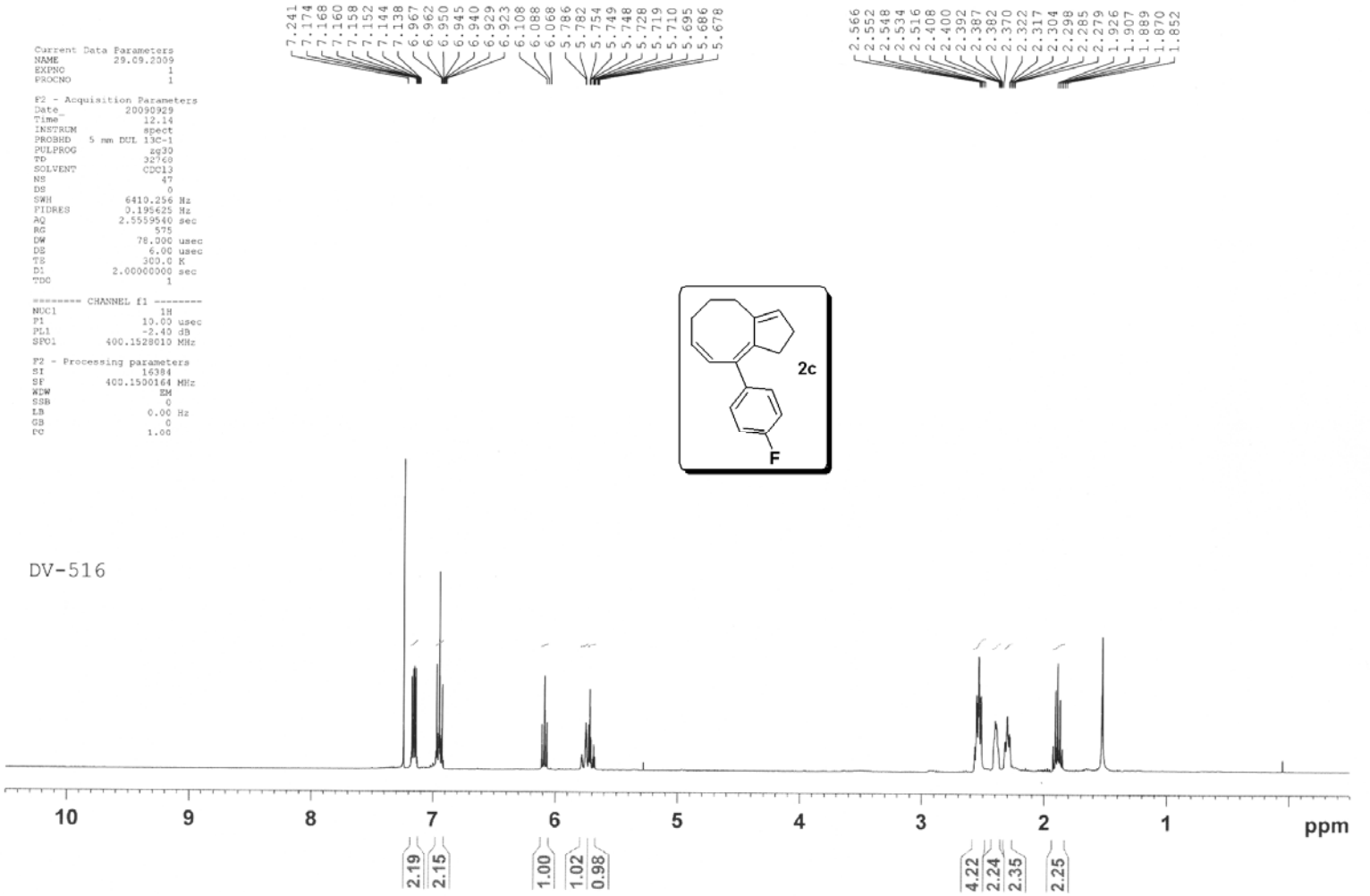


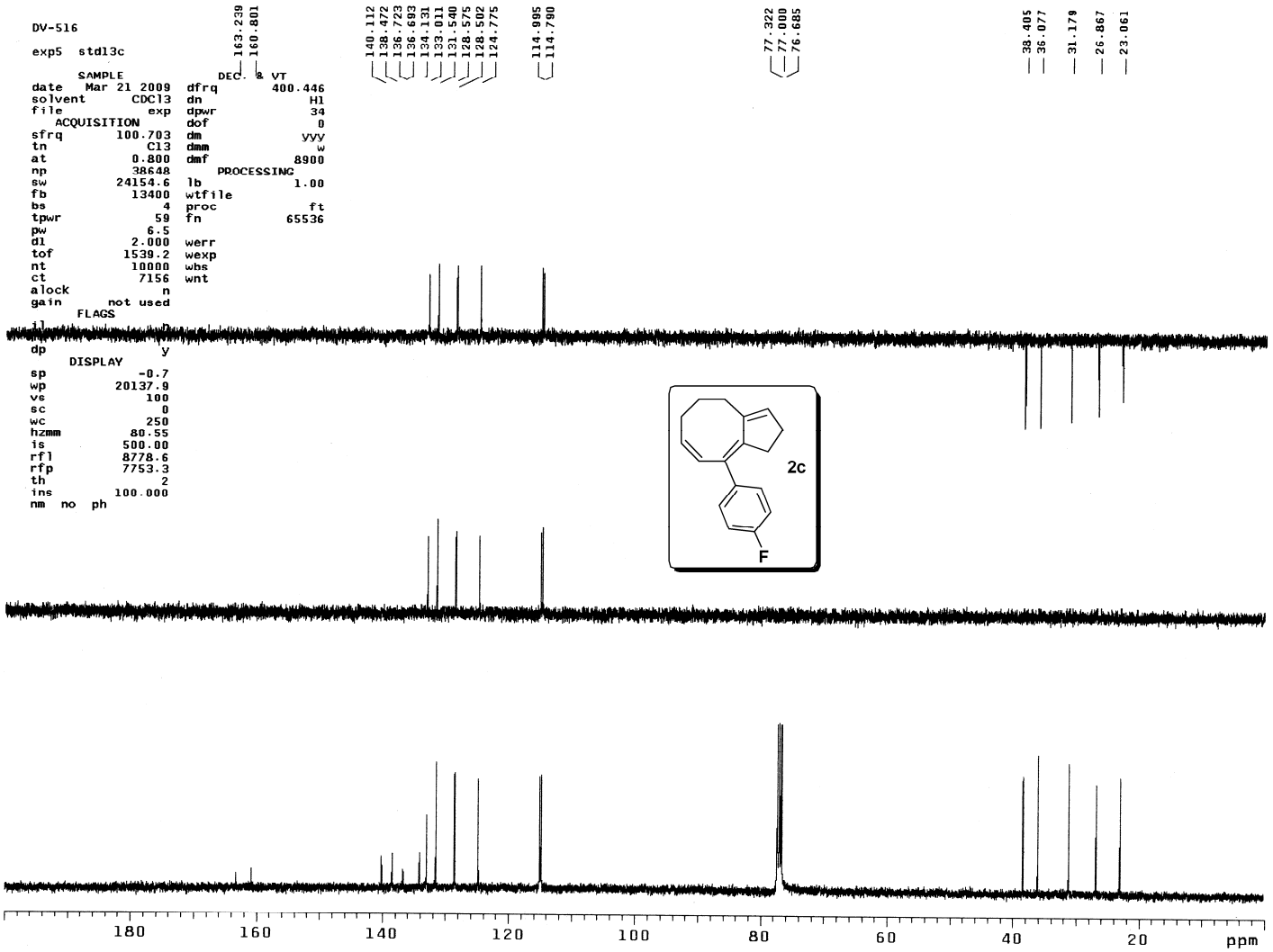
```
Current Data Parameters
NAME            DV-451-2
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090219
Time               17.25
INSTRUM            spect
PROBHD   5 mm QNP  1H/1
PULPROG             zgpg
TD                 32768
SOLVENT          H2O+D2O
NS                   615
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             3.00000000 sec
d11            0.03000000 sec
DELTA          2.90000010 sec
MCREST         0.00000000 sec
MCWRK          0.01500000 sec

======== CHANNEL f1 ========
NUC1                 13C
P1                  4.80 usec
PL1                 0.00 dB
SFO1         150.6855336 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                 1H
PCPD2              92.00 usec
PL2               120.00 dB
PL12               11.30 dB
PL13               14.00 dB
SFO2         599.2029960 MHz

F2 - Processing parameters
SI                  65536
SF           150.6689675 MHz
WDW                    EM
SSB                    0
LB                  3.00 Hz
GB                     0
PC                  0.50

1D NMR plot parameters
CX                 20.00 cm
CY                  4.50 cm
F1P               200.000 ppm
F1              30133.79 Hz
F2P                0.000 ppm
F2                 0.00 Hz
PPMCM          10.00000 ppm/cm
HZCM         1506.68958 Hz/cm
```

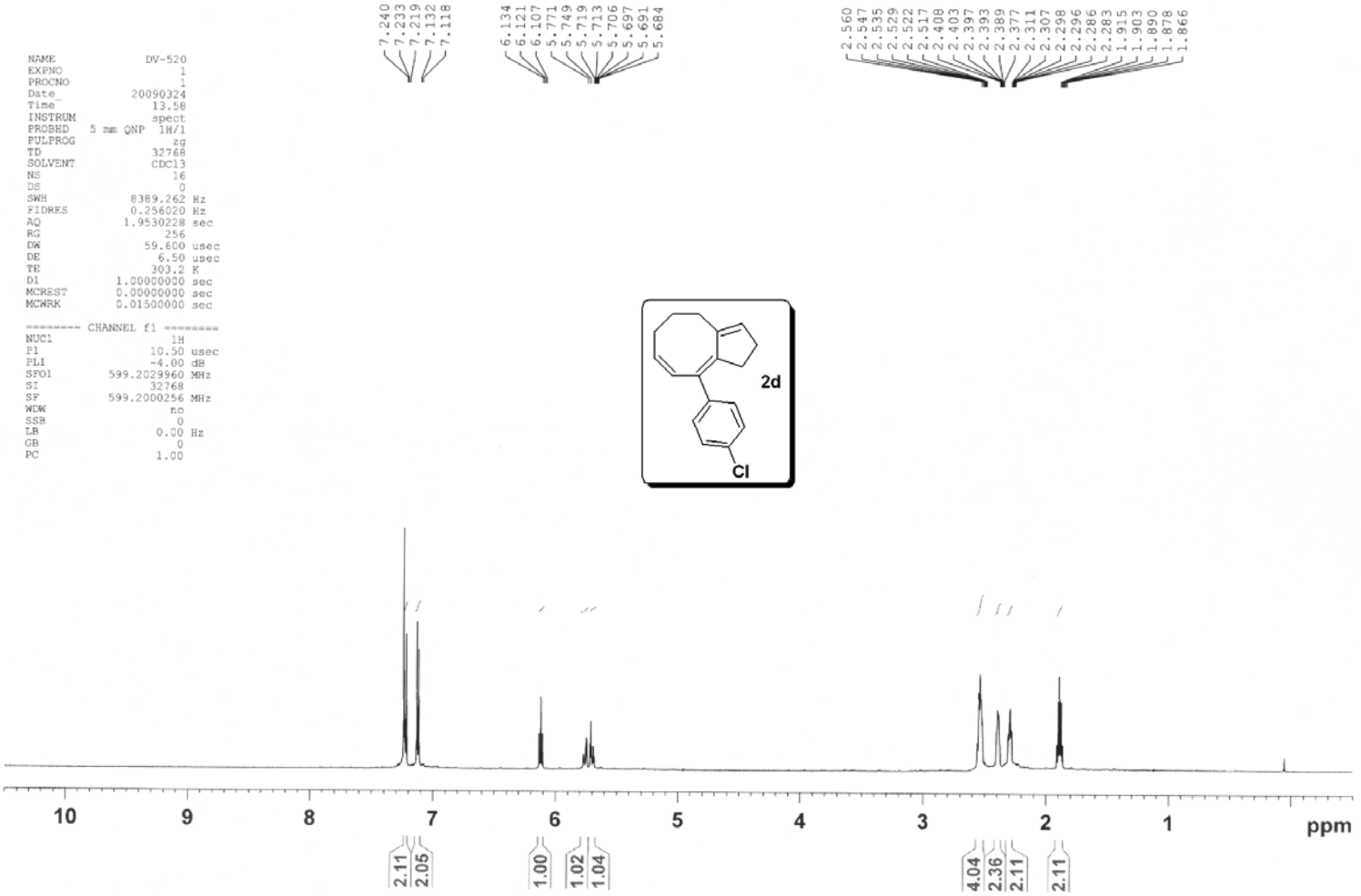




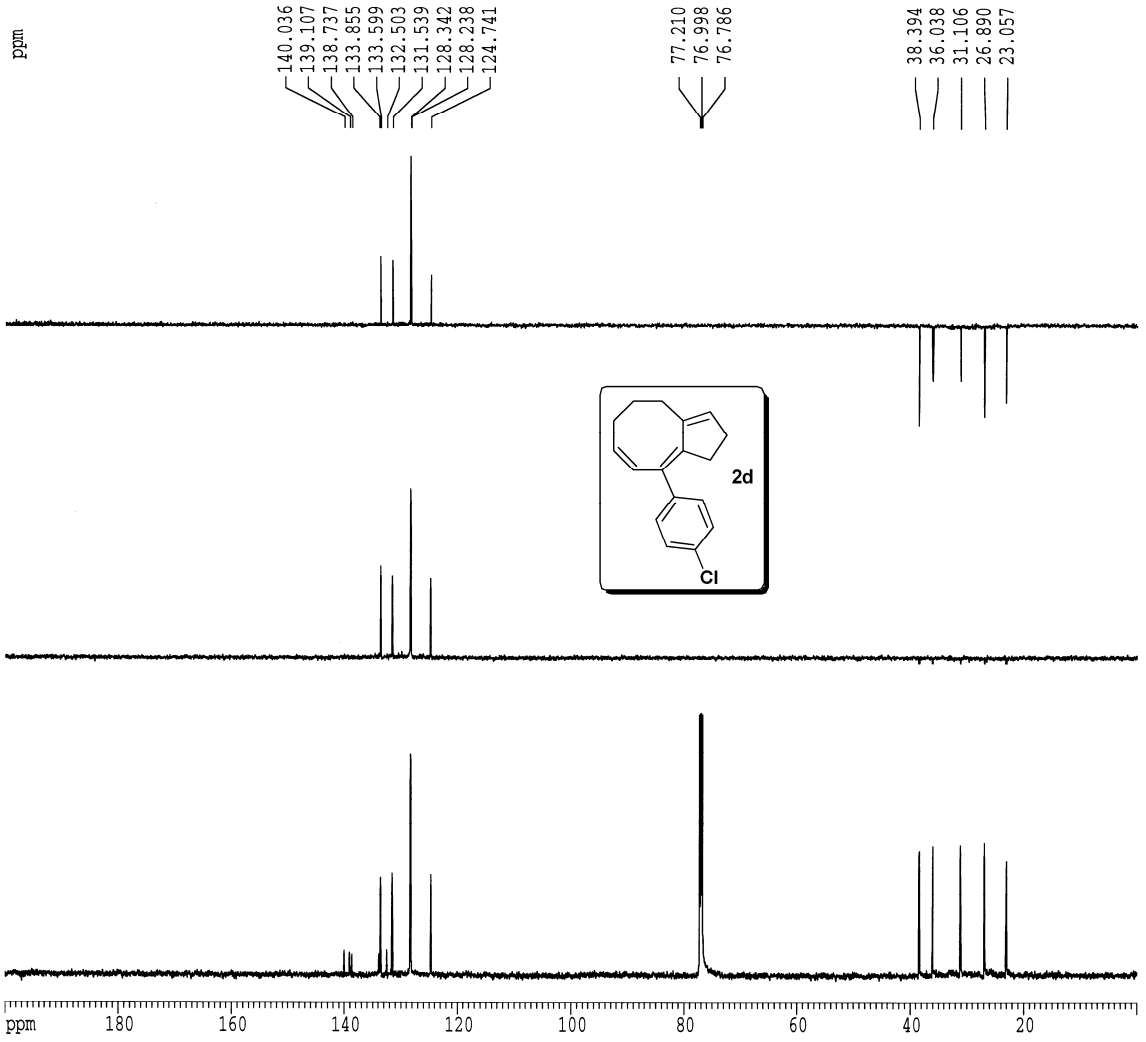

140.874
138.070
137.814
136.415
134.541
132.433
131.393
128.809
126.920
124.841
77.315
77.000
76.678
38.427
36.201
31.282
26.845
23.083
21.091
13C OBSERVE
exp3 std13c
SAMPLE
date Mar 5 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 50
pw 6.5
d1 2.000
tof 1539.2
nt 10000
ct 588
alock n
gain not used
FLAGS
in n
dp y
DISPLAY
sp -0.7
wp 20137.9
vs 30
sc 0
wc 250
hzmm 80.55
is 500.00
rfl 8780.1
rfp 7753.3
th 3
ins 100.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs
wnt
2b
Me
180
160
140
120
100
80
60
40
20
ppm

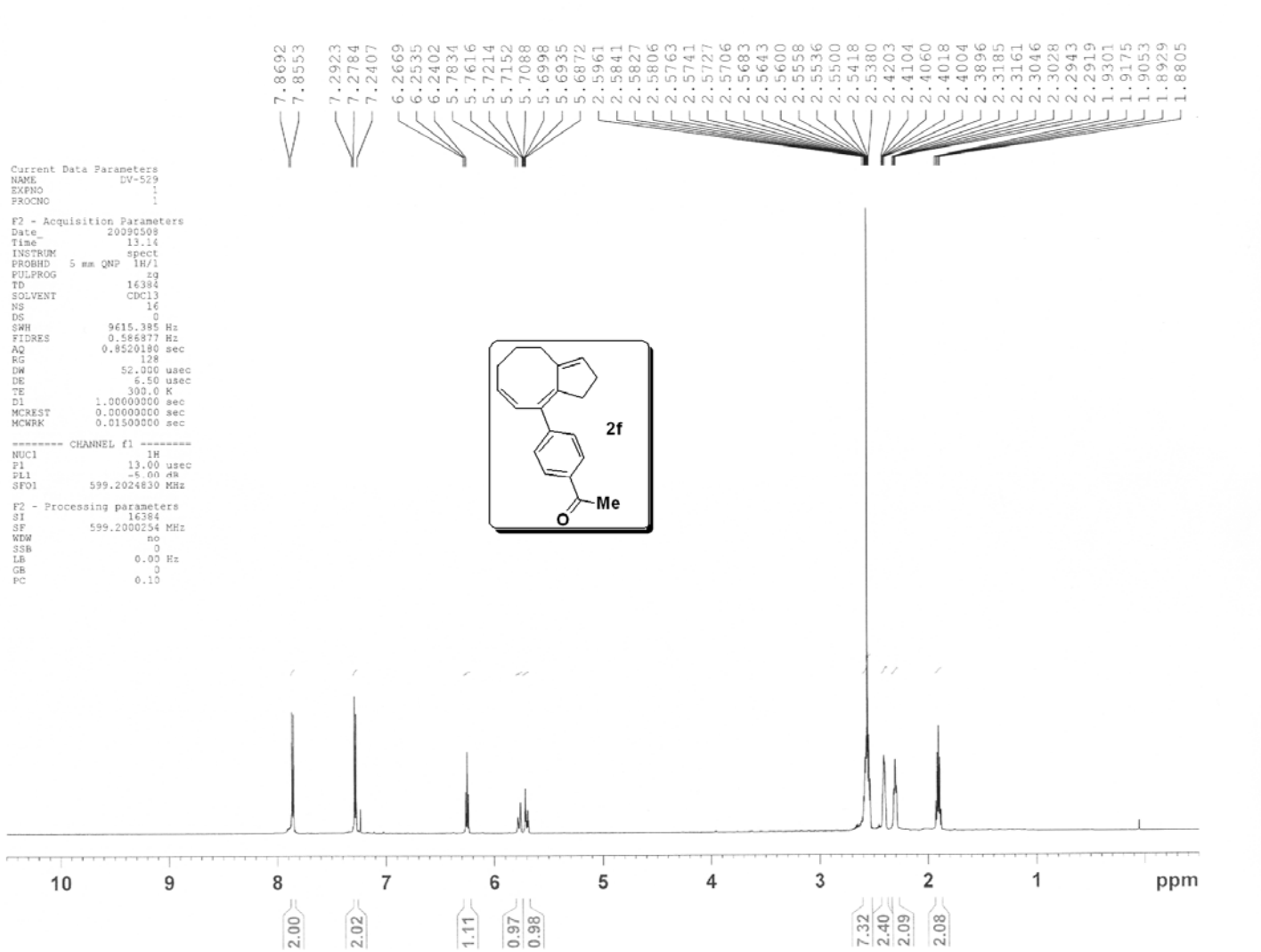


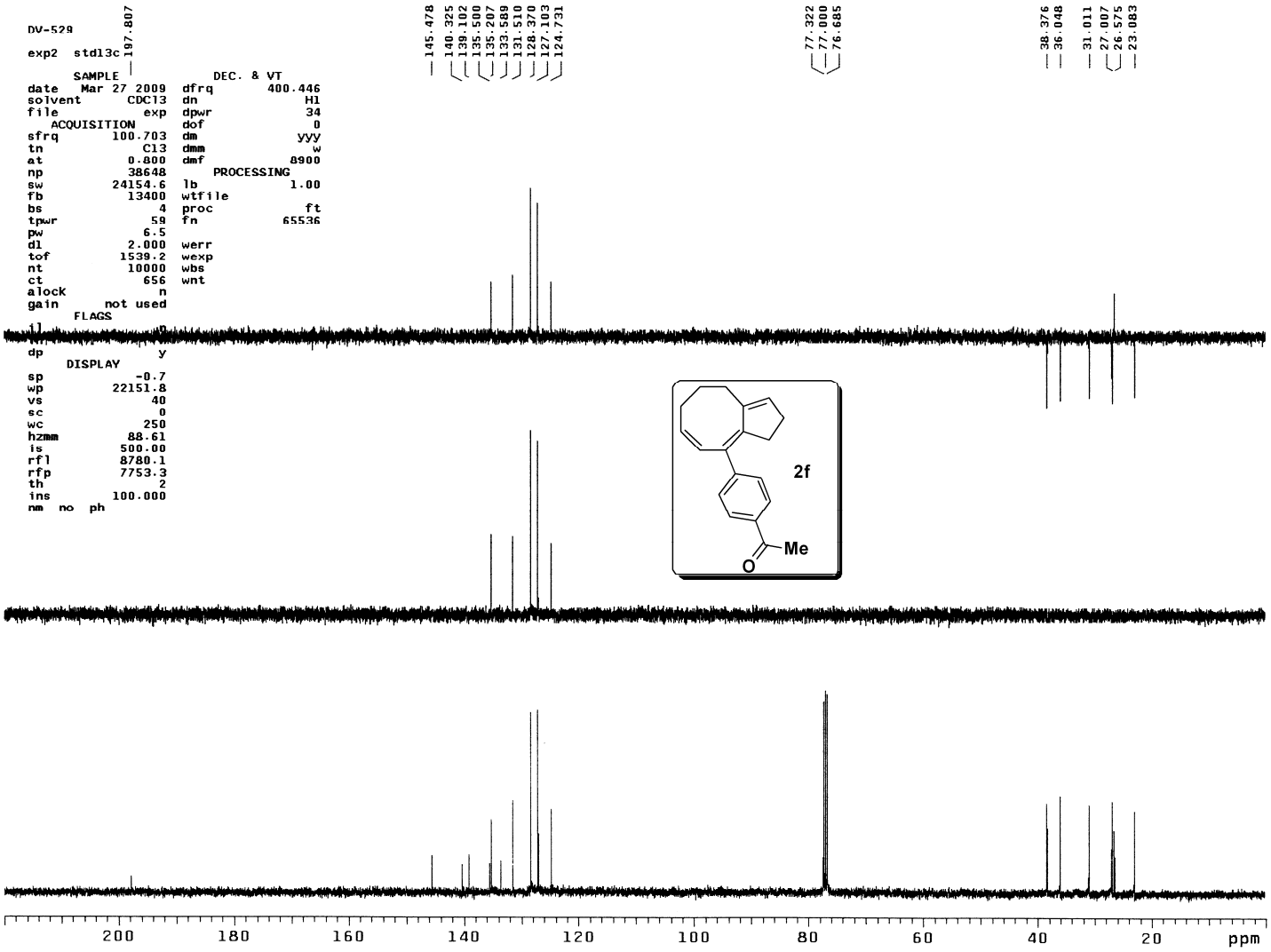


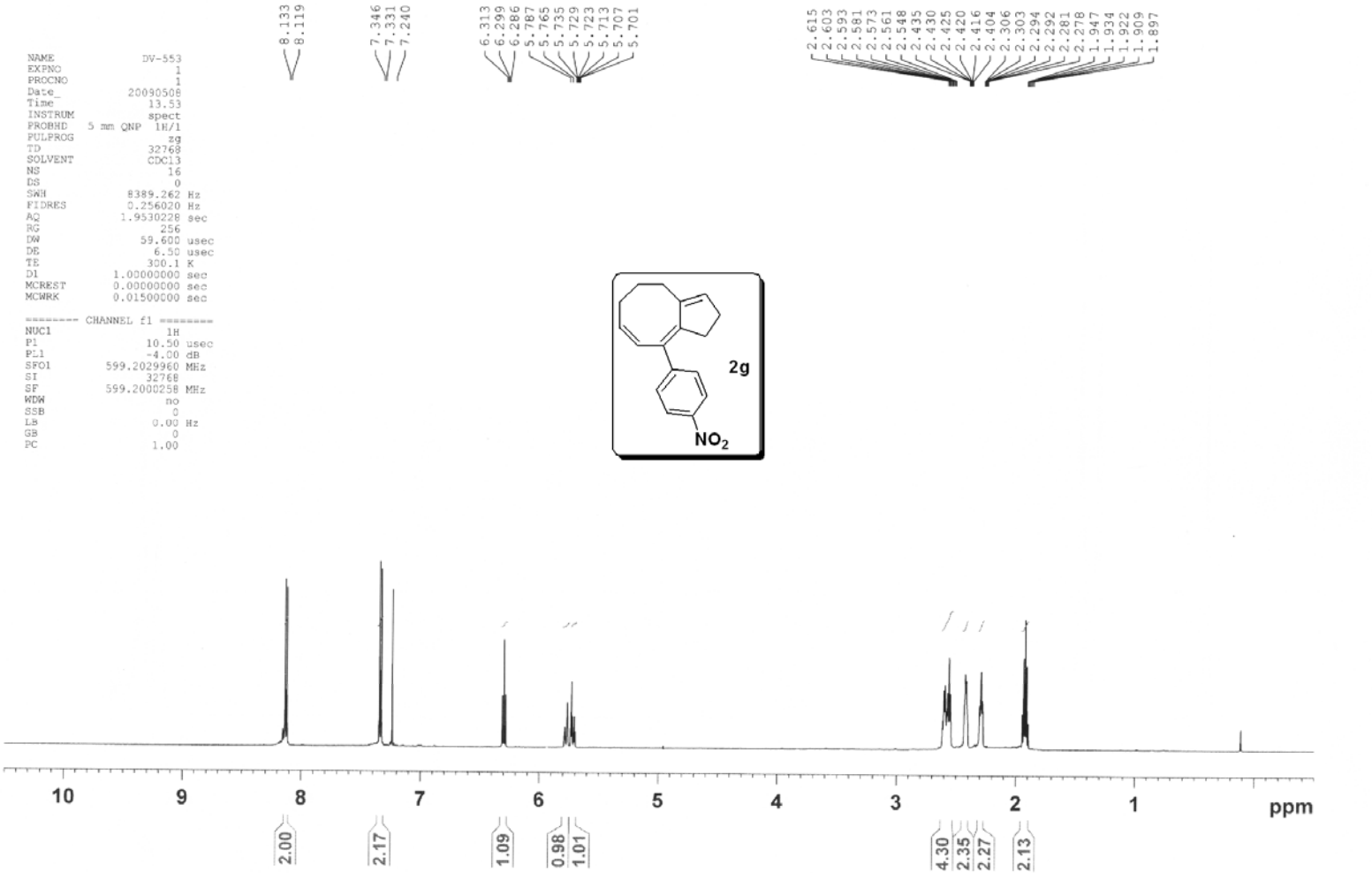

7.240
7.233
7.219
7.132
7.118
6.134
6.121
6.107
5.771
5.749
5.719
5.713
5.706
5.697
5.691
5.684
2.560
2.547
2.535
2.529
2.522
2.517
2.408
2.403
2.397
2.393
2.389
2.377
2.311
2.307
2.298
2.296
2.286
2.283
1.915
1.903
1.890
1.878
1.866
NAME DV-520
EXPNO 1
PROCNO 1
Date_ 20090324
Time 13.58
INSTRUM spect
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 303.2 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -4.00 dB
SFO1 599.2029960 MHz
SI 32768
SF 599.2000256 MHz
WDW no
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
2d
Cl
10
9
8
7
6
5
4
3
2
1
ppm
2.11
2.05
1.00
1.02
1.04
4.04
2.36
2.11
2.11

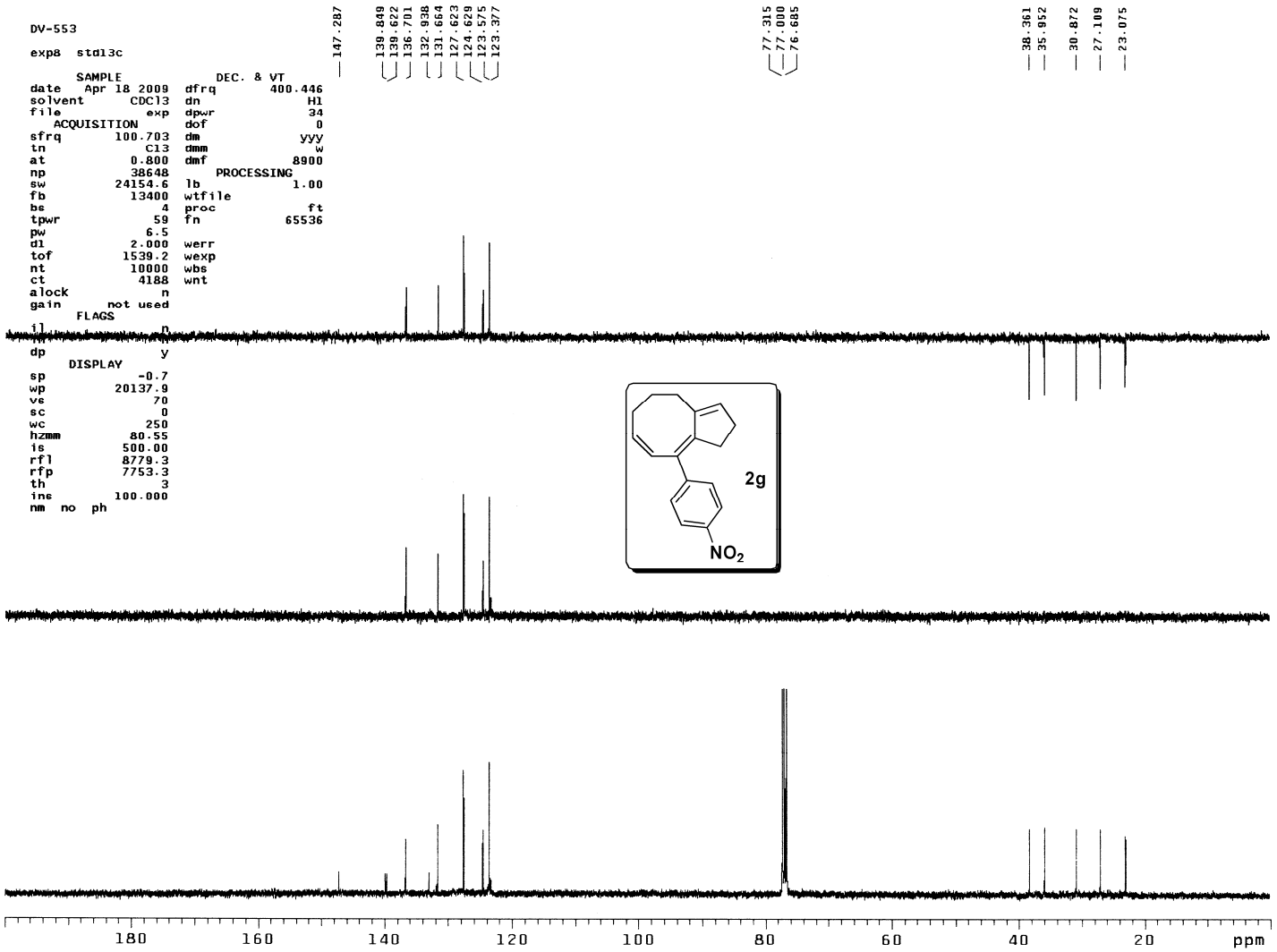
```
Current Data Parameters
NAME                 DV-520
EXPNO                     2
PROCNO                    1

F2 - Acquisition Parameters
Date_              20090323
Time                  22.03
INSTRUM               spect
PROBHD   5 mm QNP  1H/1
PULPROG                zgpg
TD                    32768
SOLVENT           H2O+D2O
NS                     6144
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                   302.3 K
D1              3.00000000 sec
d11             0.03000000 sec
DELTA           2.90000010 sec
MCREST          0.00000000 sec
MCWRK           0.01500000 sec

======== CHANNEL f1 ========
NUC1                    13C
P1                    4.80 usec
PL1                   0.00 dB
SFO1           150.6855336 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                    1H
PCPD2                92.00 usec
PL2                 120.00 dB
PL12                  9.00 dB
PL13                 14.00 dB
SFO2           599.2029960 MHz

F2 - Processing parameters
SI                    65536
SF             150.6689647 MHz
WDW                      EM
SSB                       0
LB                    3.00 Hz
GB                        0
PC                    0.50

1D NMR plot parameters
CX                   20.00 cm
CY                   15.00 cm
F1P                200.000 ppm
F1                30133.79 Hz
F2P                  0.000 ppm
F2                   0.00 Hz
PPMCM             10.00000 ppm/cm
HZCM           1506.68958 Hz/cm
```

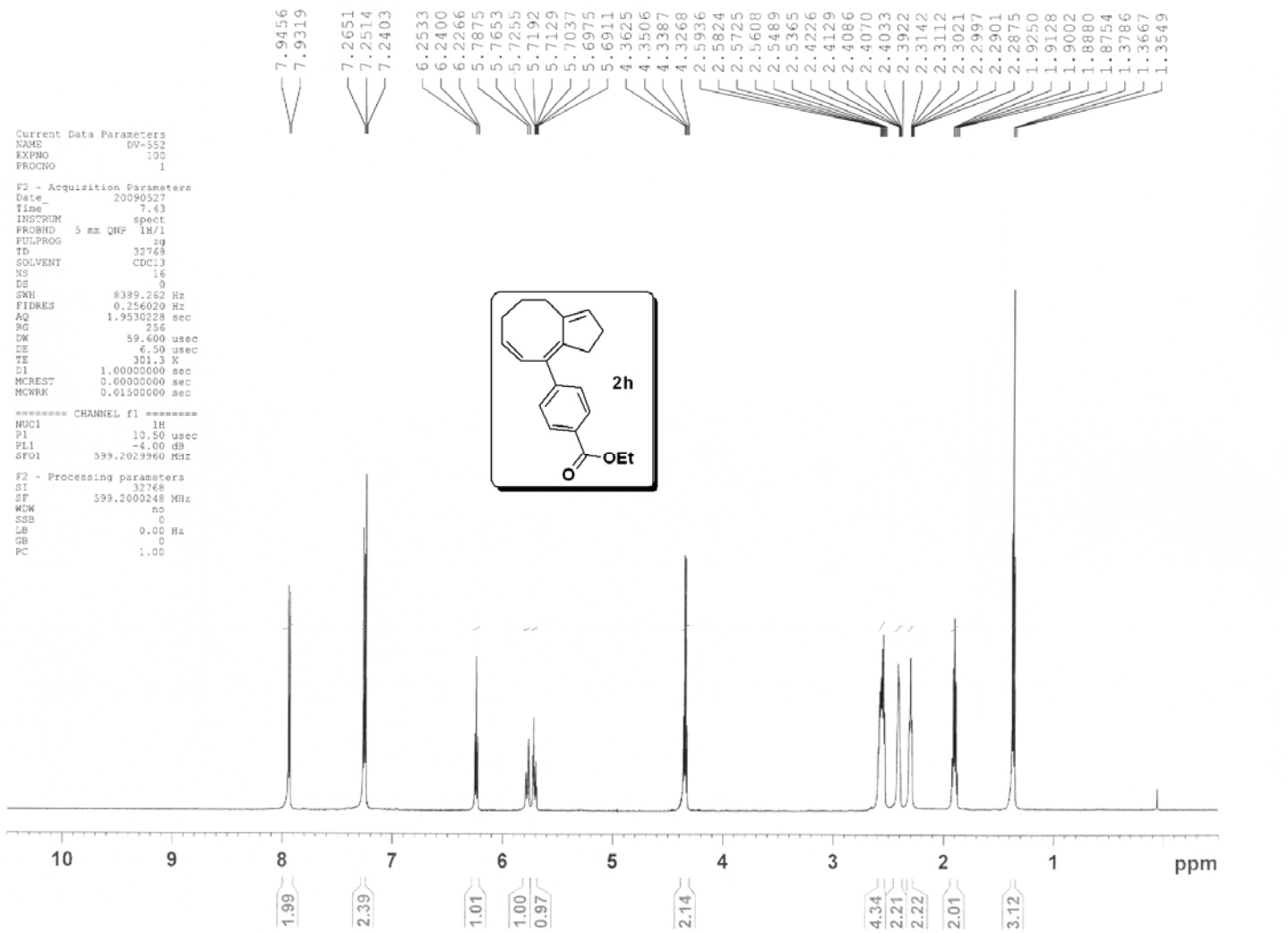


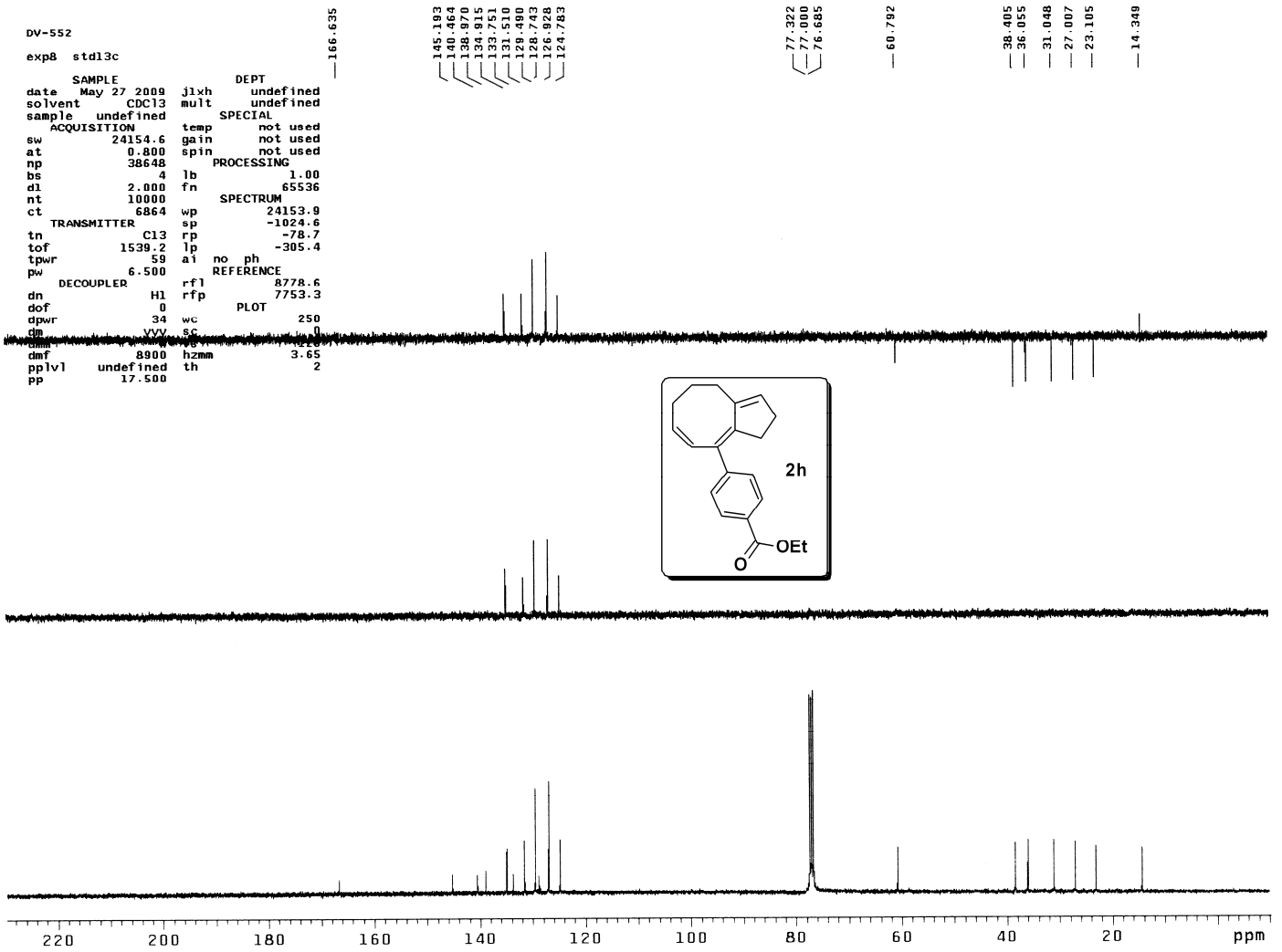


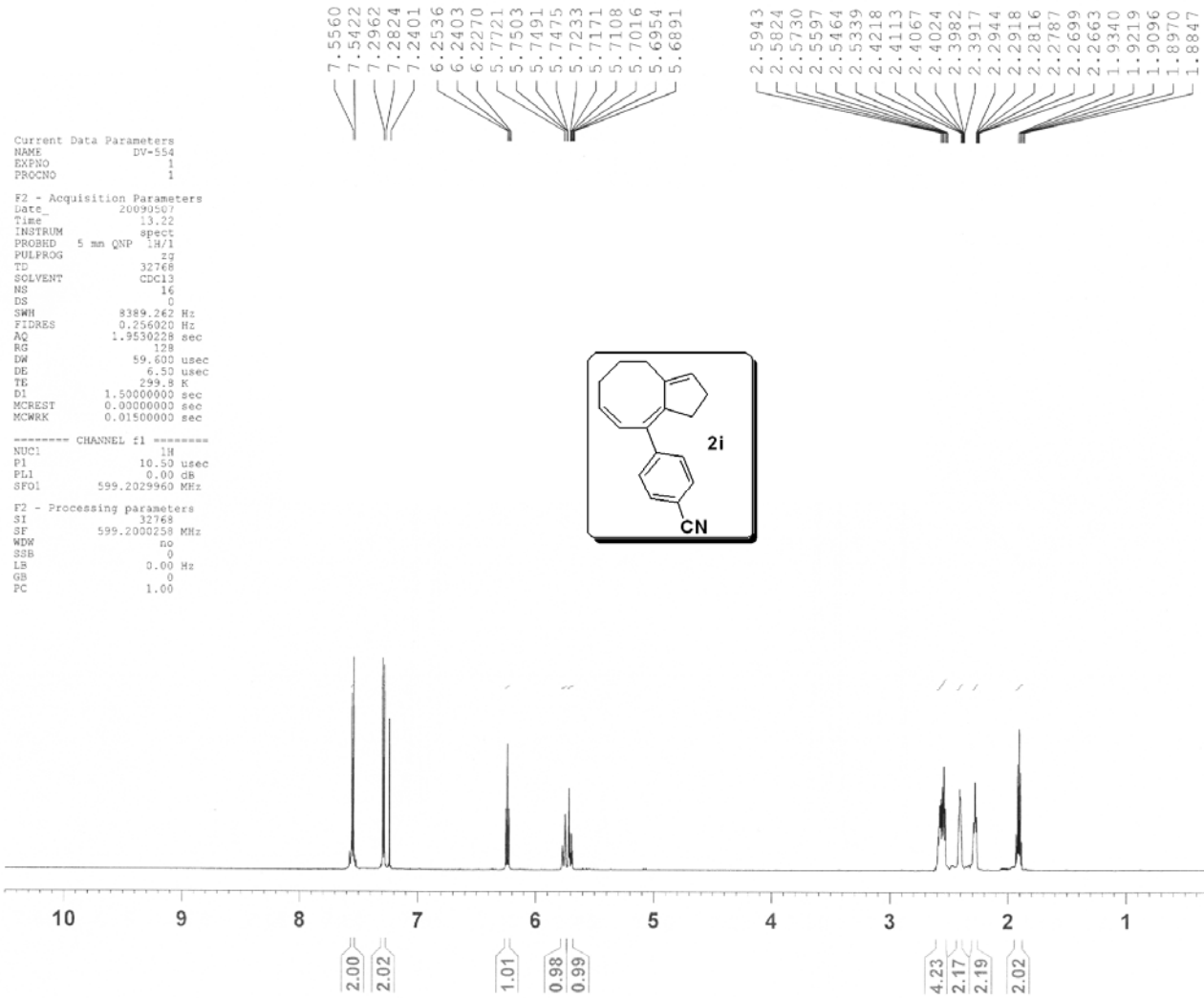

DV-529
exp2 stdl3c
SAMPLE
date Mar 27 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
d1 2.000
tof 1539.2
nt 10000
ct 656
alock n
gain not used
FLAGS
dp y
DISPLAY
sp -0.7
wp 22151.8
vs 40
sc 0
wc 250
hzmm 88.61
is 500.00
rfl 8780.1
rfp 7753.3
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs
wnt
197.807
145.478
140.325
139.102
135.500
135.207
133.589
131.510
128.370
127.103
124.731
77.322
77.000
76.685
38.376
36.048
31.011
27.007
26.575
23.083
2f
Me
O
200
180
160
140
120
100
80
60
40
20
ppm

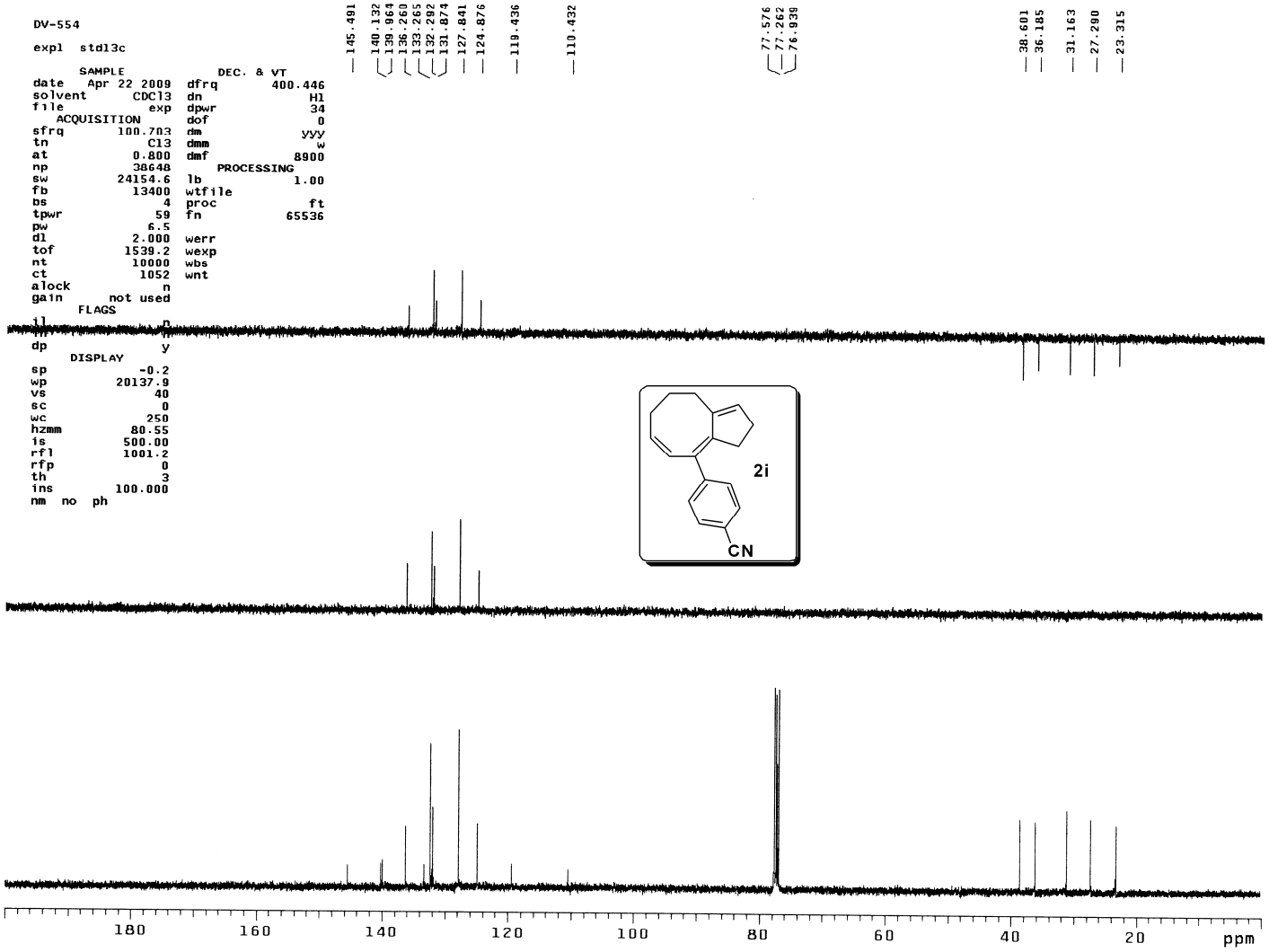


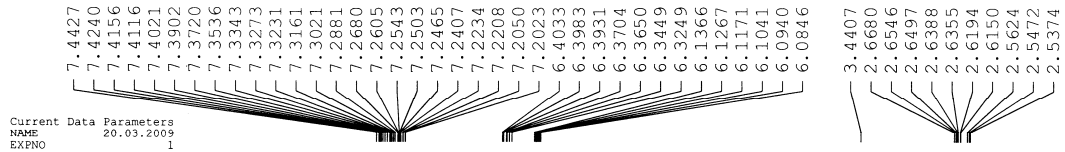




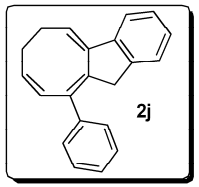


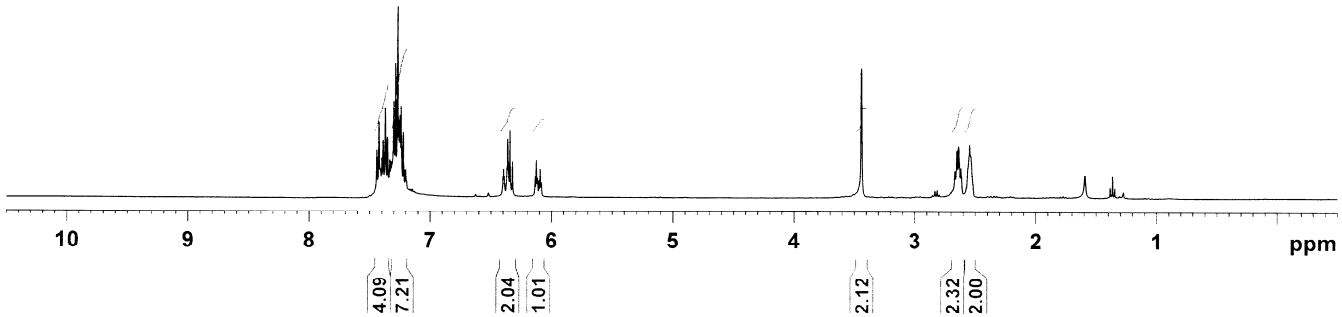


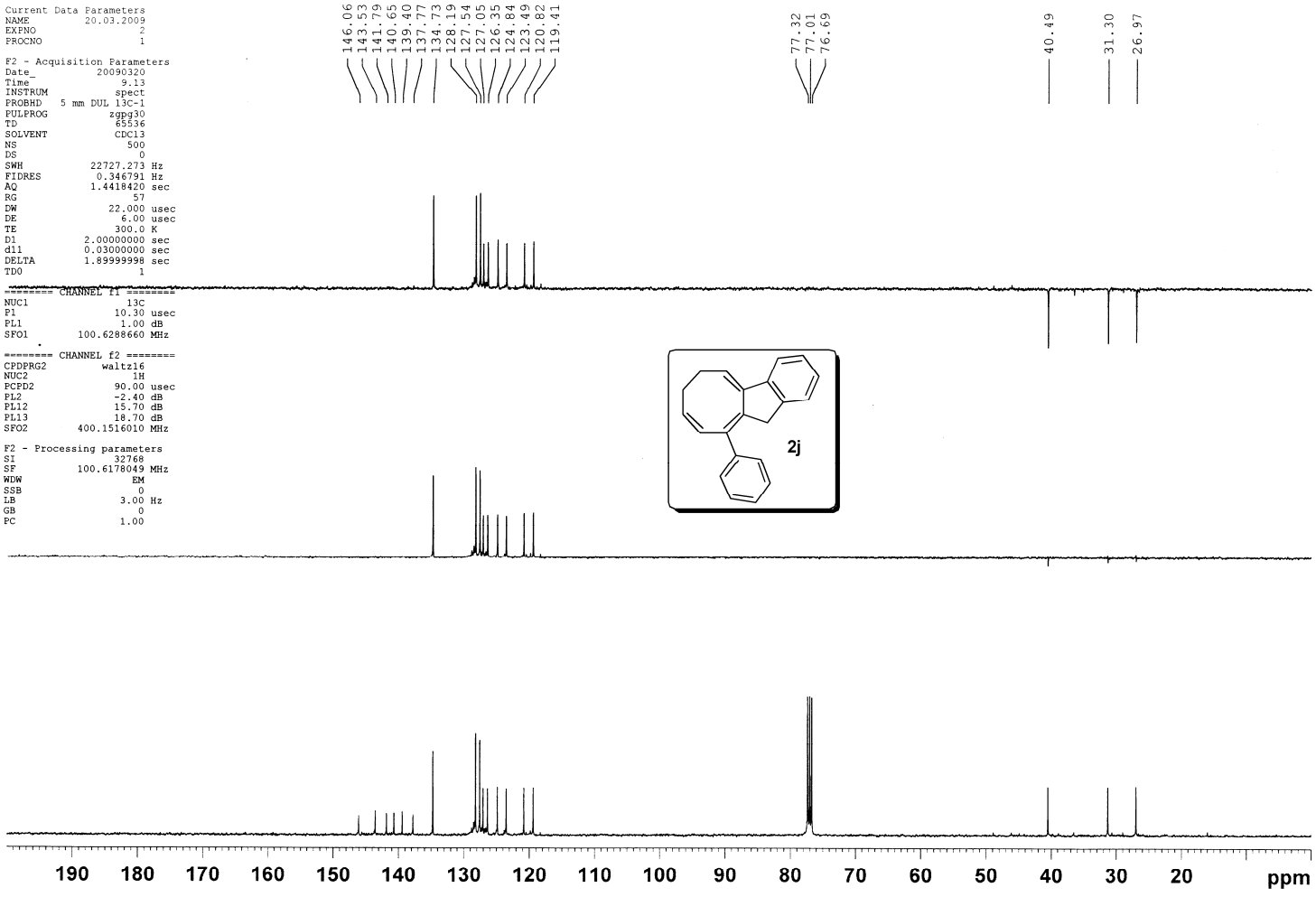


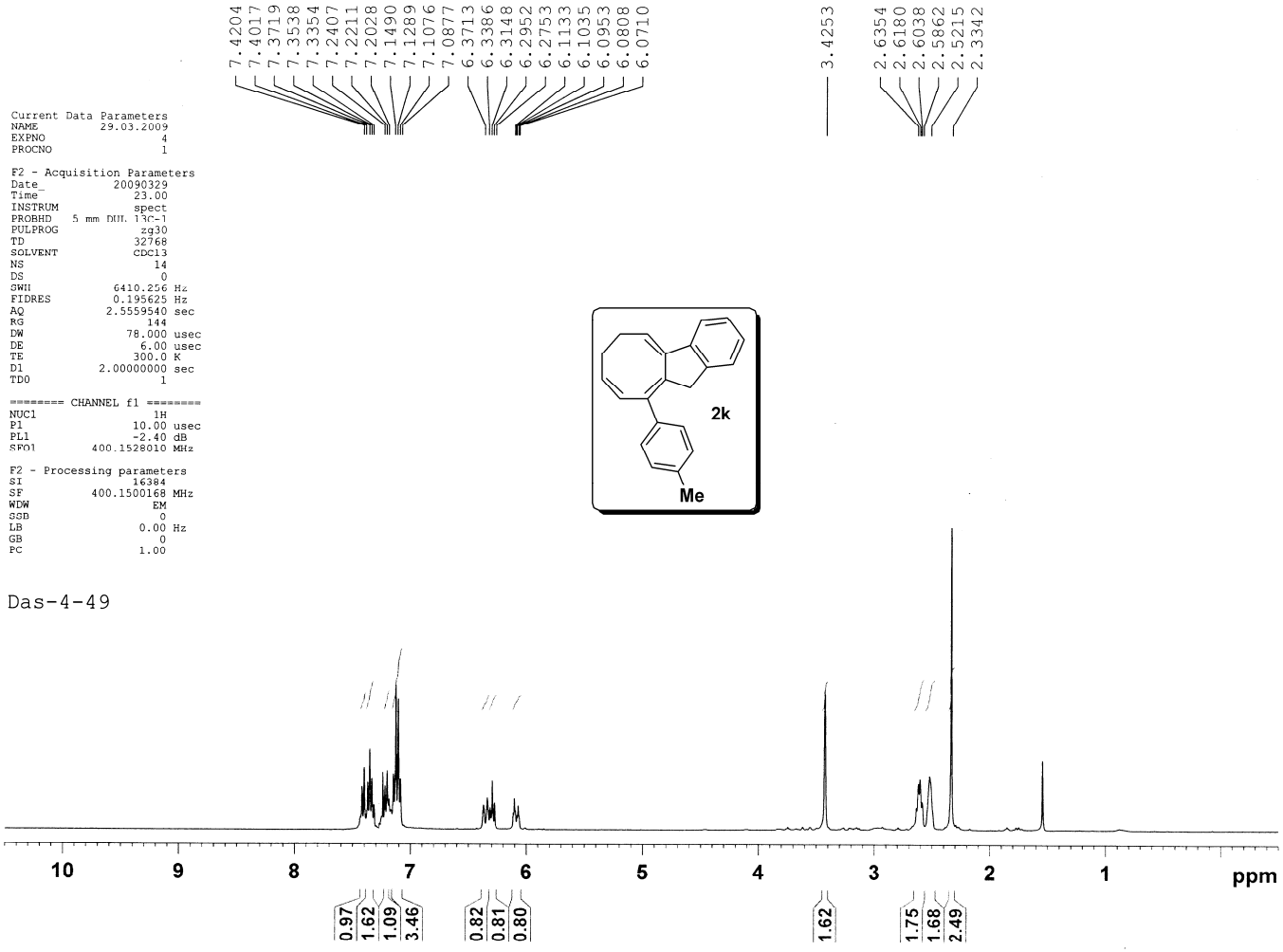


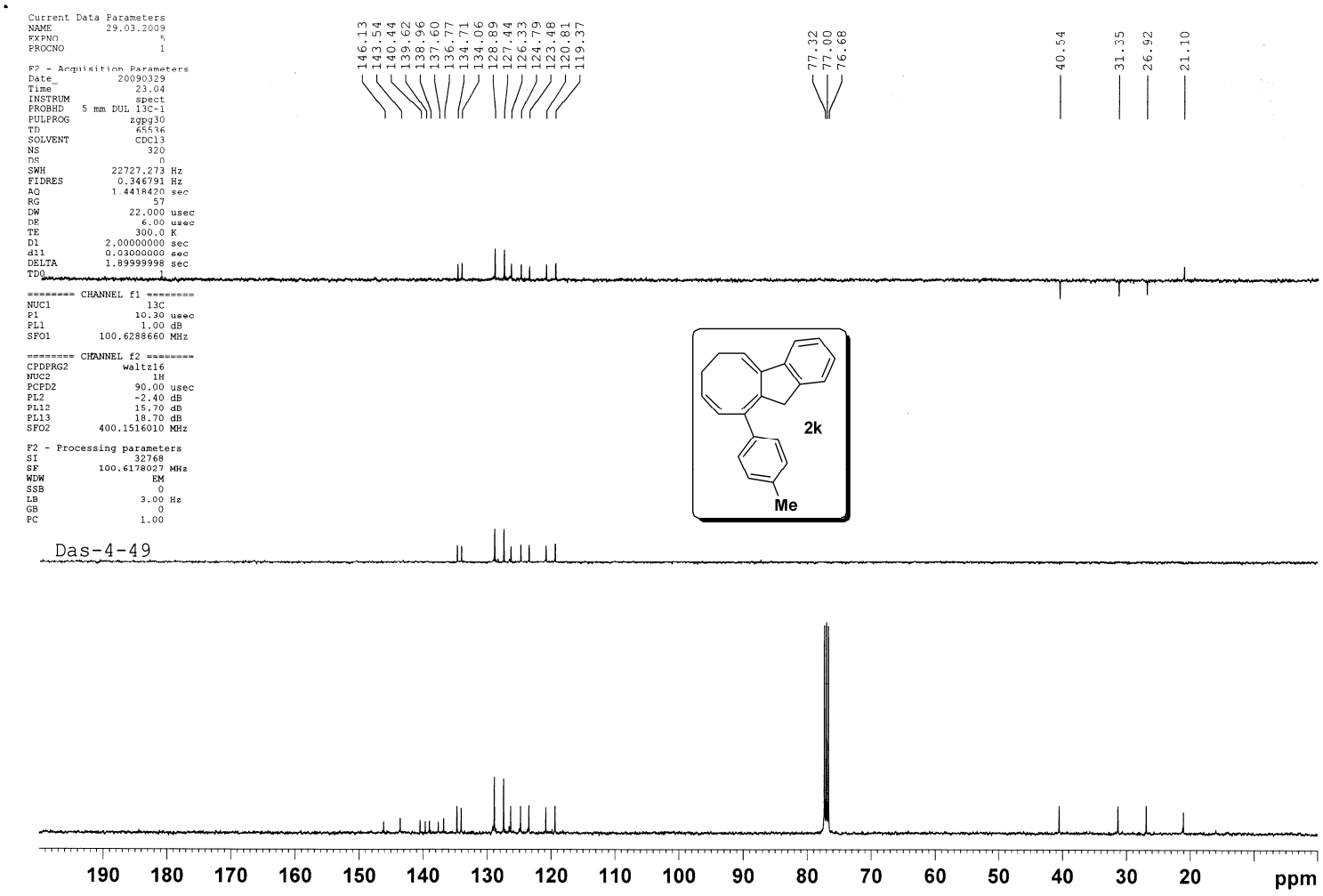

```
Current Data Parameters
NAME          20.03.2009
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090320
Time               9.07
INSTRUM            spect
PROBHD   5 mm DUL 13C-1
PULPROG             zg30
TD                 32768
SOLVENT            CDCl3
NS                    24
DS                     0
SWH             6410.256 Hz
FIDRES          0.195625 Hz
AQ             2.5559540 sec
RG                   114
DW                78.000 usec
DE                 6.00 usec
TE                300.0 K
D1             2.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PL1               -2.40 dB
SFO1         400.1528010 MHz

F2 - Processing parameters
SI                 16384
SF           400.1500165 MHz
WDW                   EM
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.00
```

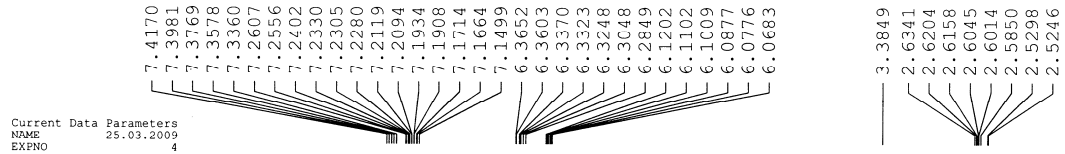




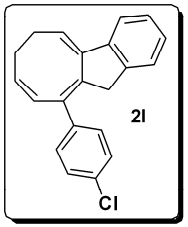




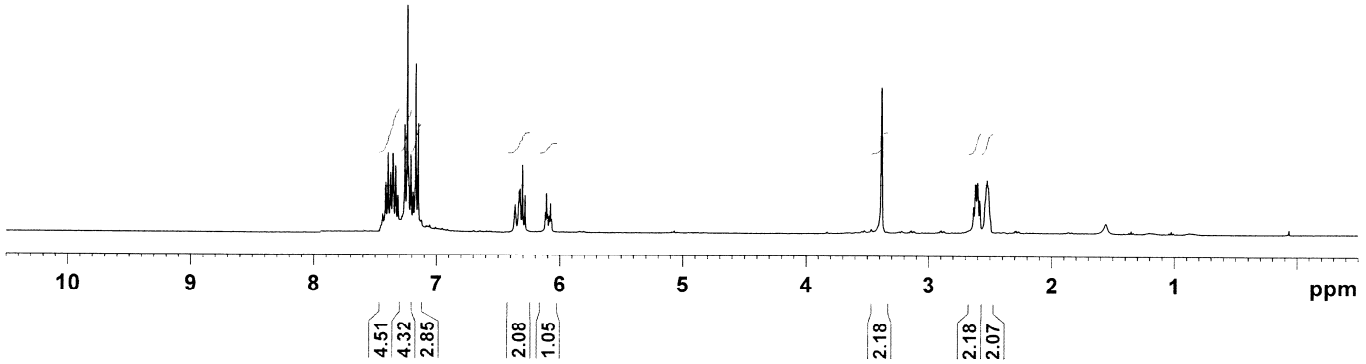


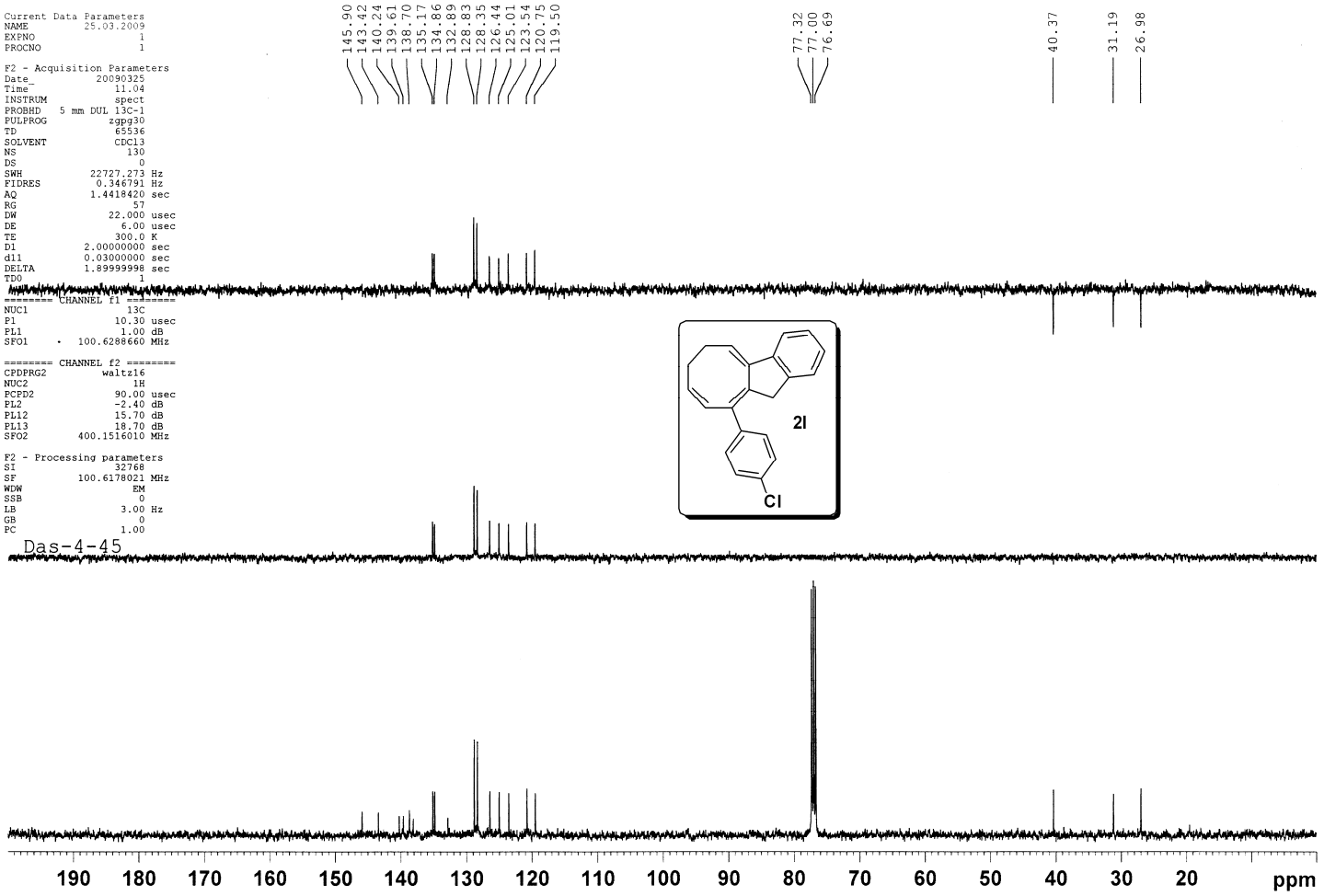


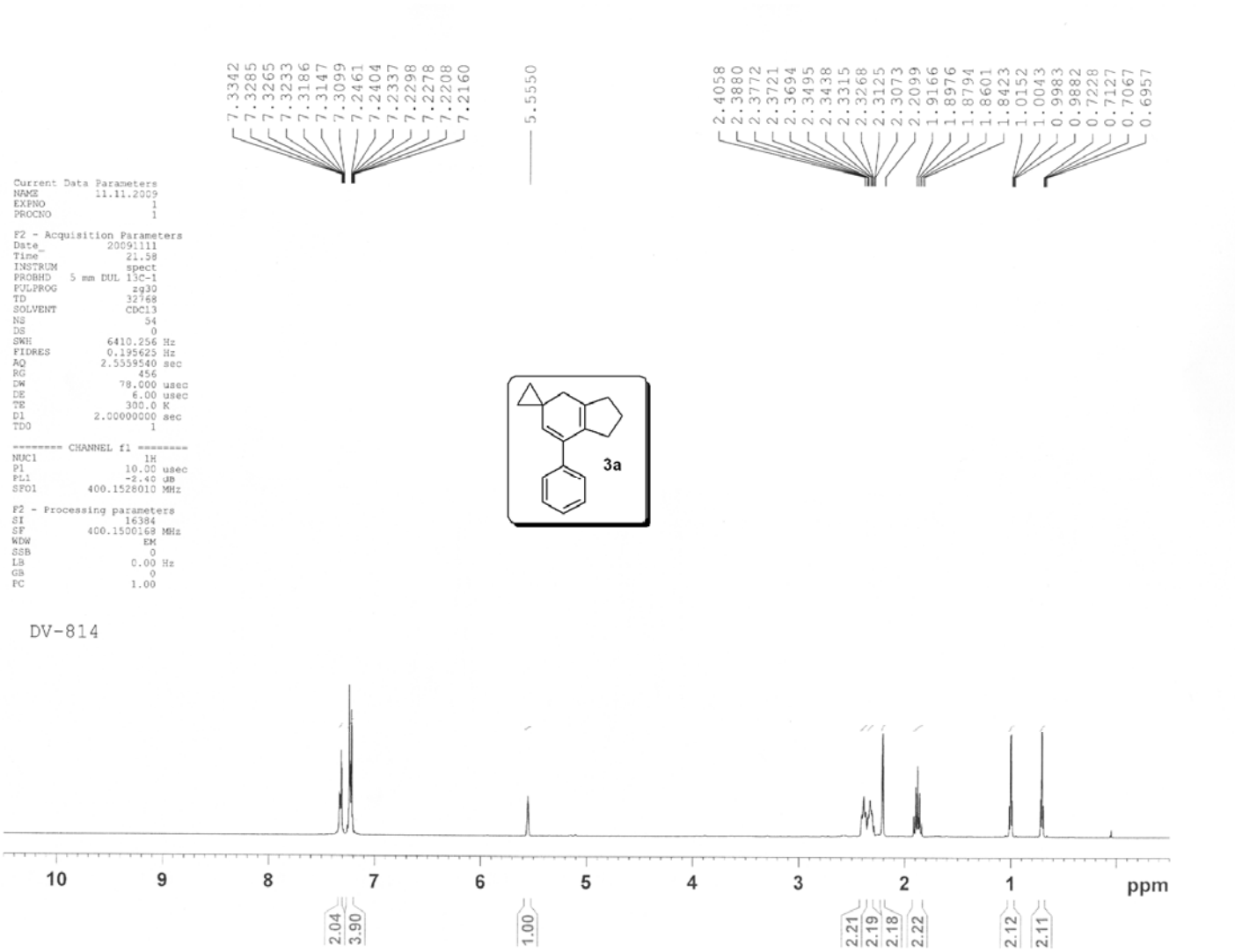

```
Current Data Parameters
NAME          25.03.2009
EXPNO                  4
PROCNO                 1

F2 - Acquisition Parameters
Date_           20090325
Time              11.19
INSTRUM           spect
PROBHD   5 mm DUL 13C-1
PULPROG            zg30
TD                32768
SOLVENT           CDCl3
NS                   24
DS                    0
SWH            6410.256 Hz
FIDRES        0.195625 Hz
AQ           2.5559540 sec
RG                  287
DW               78.000 usec
DE                6.00 usec
TE               300.0 K
D1           2.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 1H
P1                10.00 usec
PL1               -2.40 dB
SFO1        400.1528010 MHz

F2 - Processing parameters
SI                16384
SF          400.1500164 MHz
WDW                  EM
SSB                   0
LB                 0.00 Hz
GB                    0
PC                 1.00
```

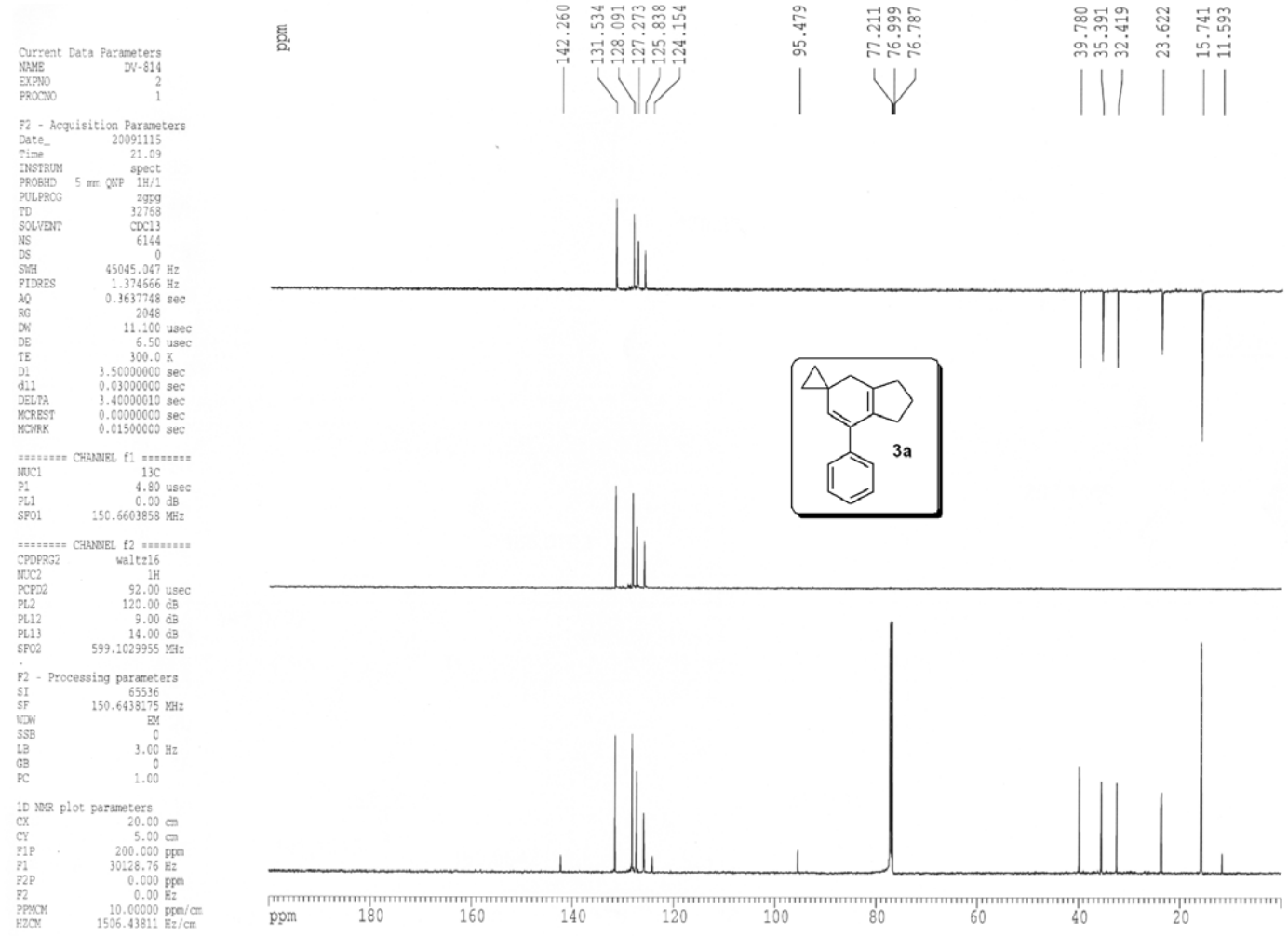


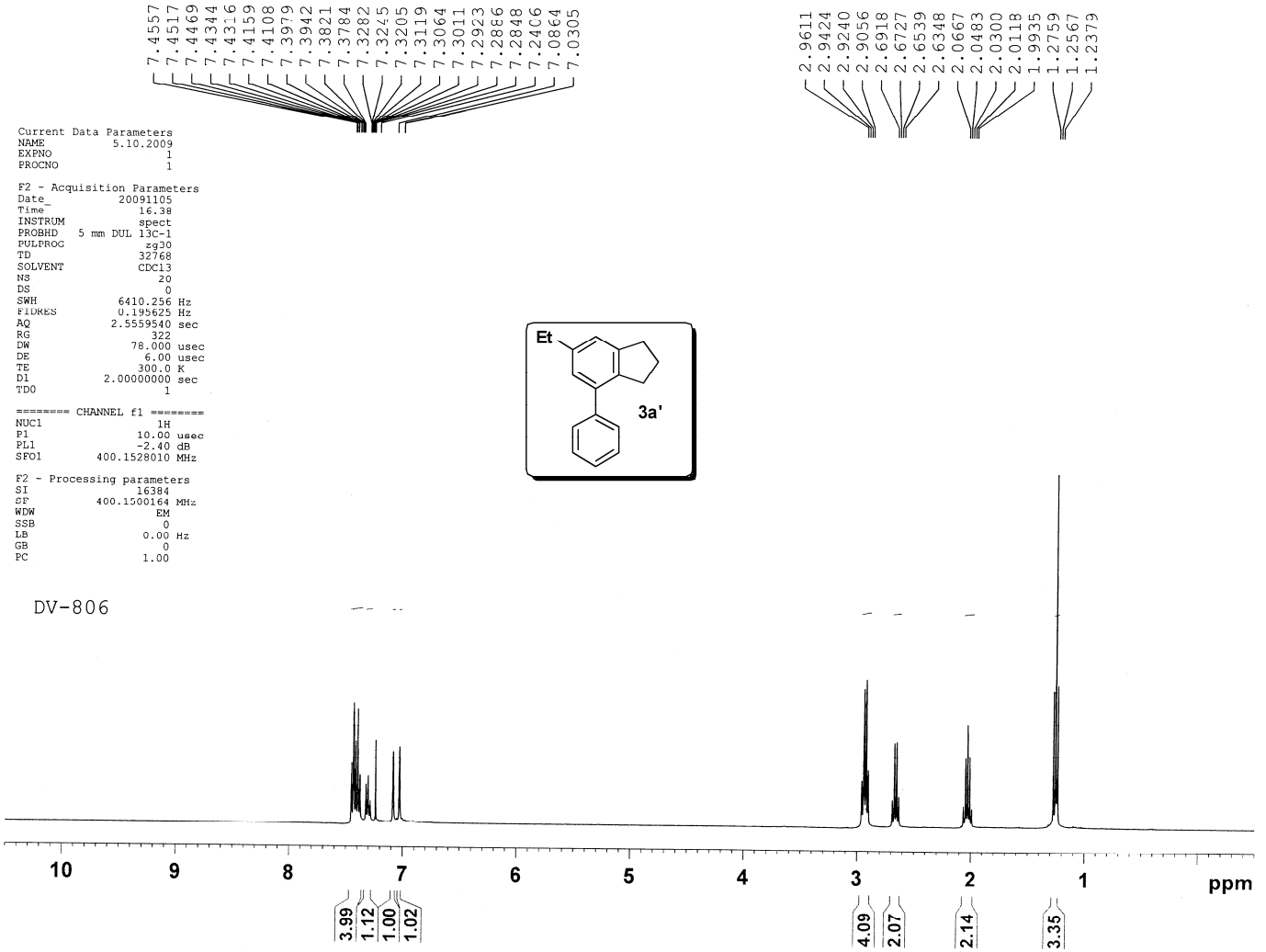
Das-4-45

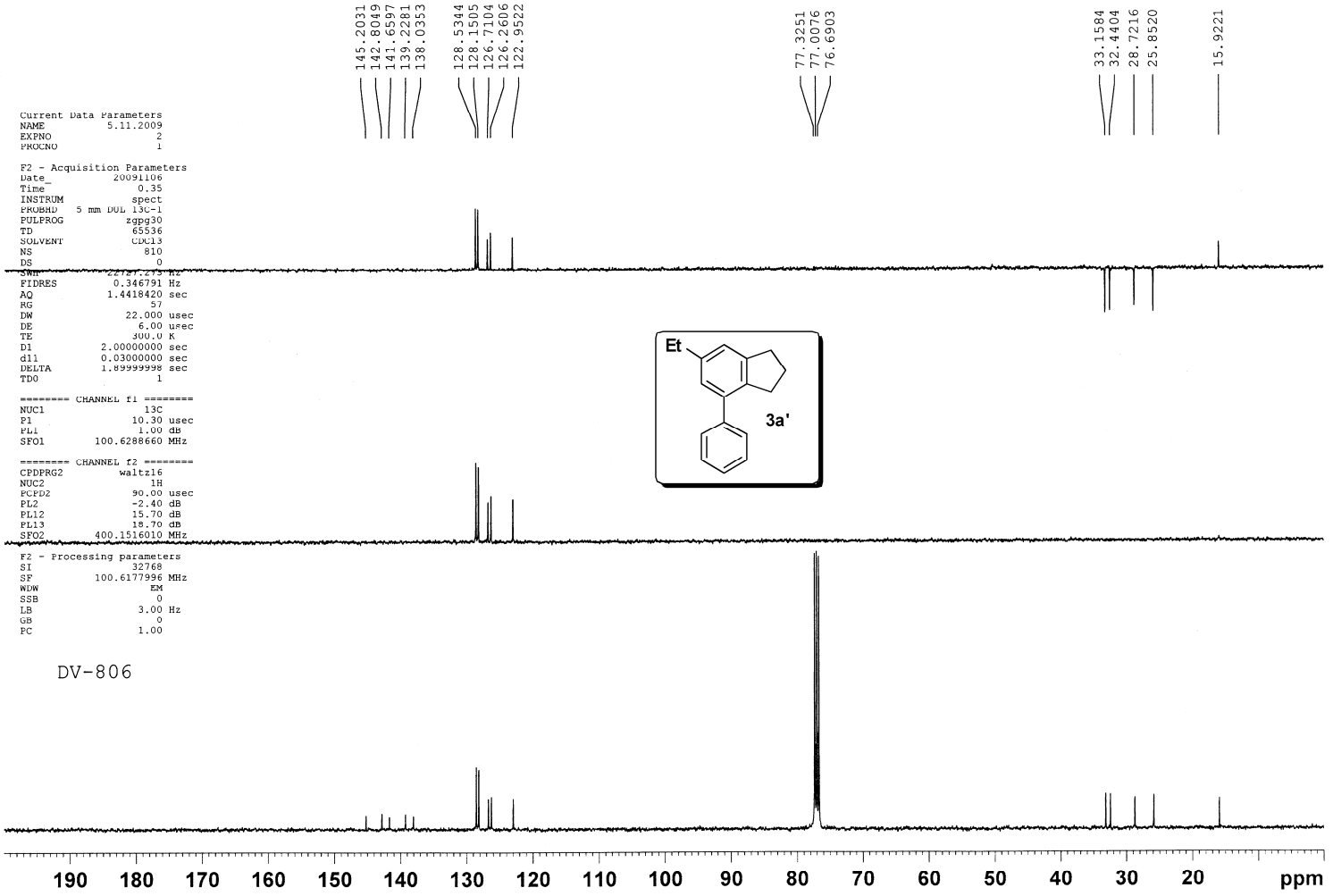



Current Data Parameters
NAME 25.03.2009
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20090325
Time 11.04
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 130
DS 0
SWH 22727.273 Hz
FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 57
DW 22.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178021 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
Das-4-45
145.90
143.42
140.24
139.61
138.70
135.17
134.86
132.89
128.83
128.35
126.44
125.01
123.54
120.75
119.50
77.32
77.00
76.69
40.37
31.19
26.98
2l
Cl
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


7.3342
7.3285
7.3265
7.3233
7.3186
7.3147
7.3099
7.2461
7.2404
7.2337
7.2298
7.2278
7.2208
7.2160
5.5550
2.4058
2.3880
2.3772
2.3721
2.3694
2.3495
2.3438
2.3315
2.3268
2.3125
2.3073
2.2099
1.9166
1.8976
1.8794
1.8601
1.8423
1.0152
1.0043
0.9983
0.9882
0.7228
0.7127
0.7067
0.6957
Current Data Parameters
NAME 11.11.2009
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091111
Time 21.58
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 54
DS 0
SWH 6410.256 Hz
FIDRES 0.195625 Hz
AQ 2.5559540 sec
RG 456
DW 78.000 usec
DE 6.00 usec
TE 300.0 K
D1 2.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PL1 -2.40 dB
SFO1 400.1528010 MHz
F2 - Processing parameters
SI 16384
SF 400.1500168 MHz
WDW EM
SSB 0
LB 0.00 Hz
GB 0
PC 1.00
3a
DV-814
10
9
8
7
6
5
4
3
2
1
ppm
2.04
3.90
1.00
2.21
2.19
2.18
2.22
2.12
2.11


Current Data Parameters
NAME DV-814
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091115
Time 21.09
INSTRUM spect
PROBHD 5 mm QNP 1H/1
PULPROG zgpg
TD 32768
SOLVENT CDCl3
NS 6144
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 300.0 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 150.6603858 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 92.00 usec
PL2 120.00 dB
PL12 9.00 dB
PL13 14.00 dB
SFO2 599.1029955 MHz
F2 - Processing parameters
SI 65536
SF 150.6438175 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
1D NMR plot parameters
CX 20.00 cm
CY 5.00 cm
F1P 200.000 ppm
F1 30128.76 Hz
F2P 0.000 ppm
F2 0.00 Hz
PPMCM 10.00000 ppm/cm
HZCM 1506.43811 Hz/cm
ppm
142.260
131.534
128.091
127.273
125.838
124.154
95.479
77.211
76.999
76.787
39.780
35.391
32.419
23.622
15.741
11.593
3a
ppm
180
160
140
120
100
80
60
40
20

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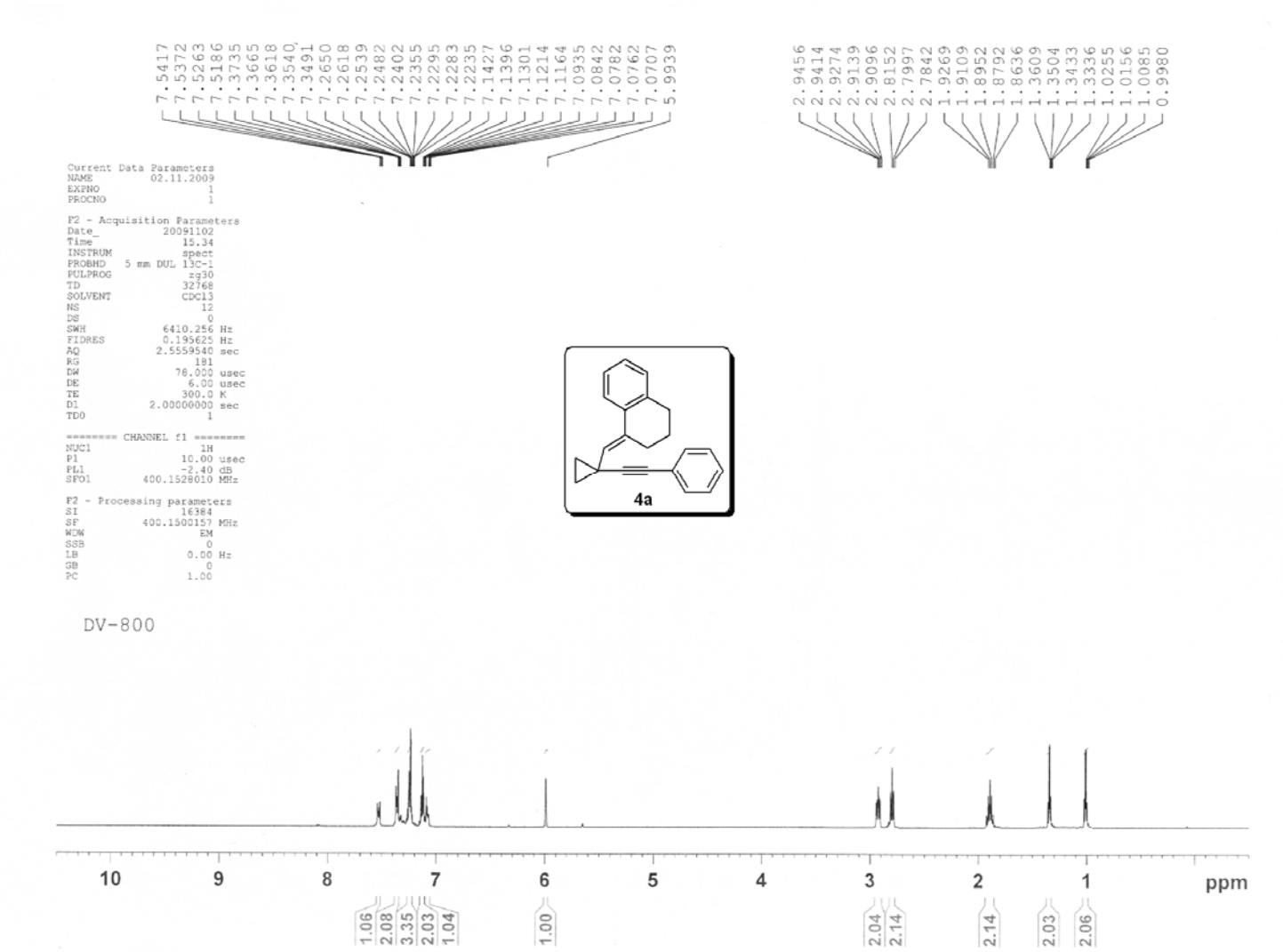
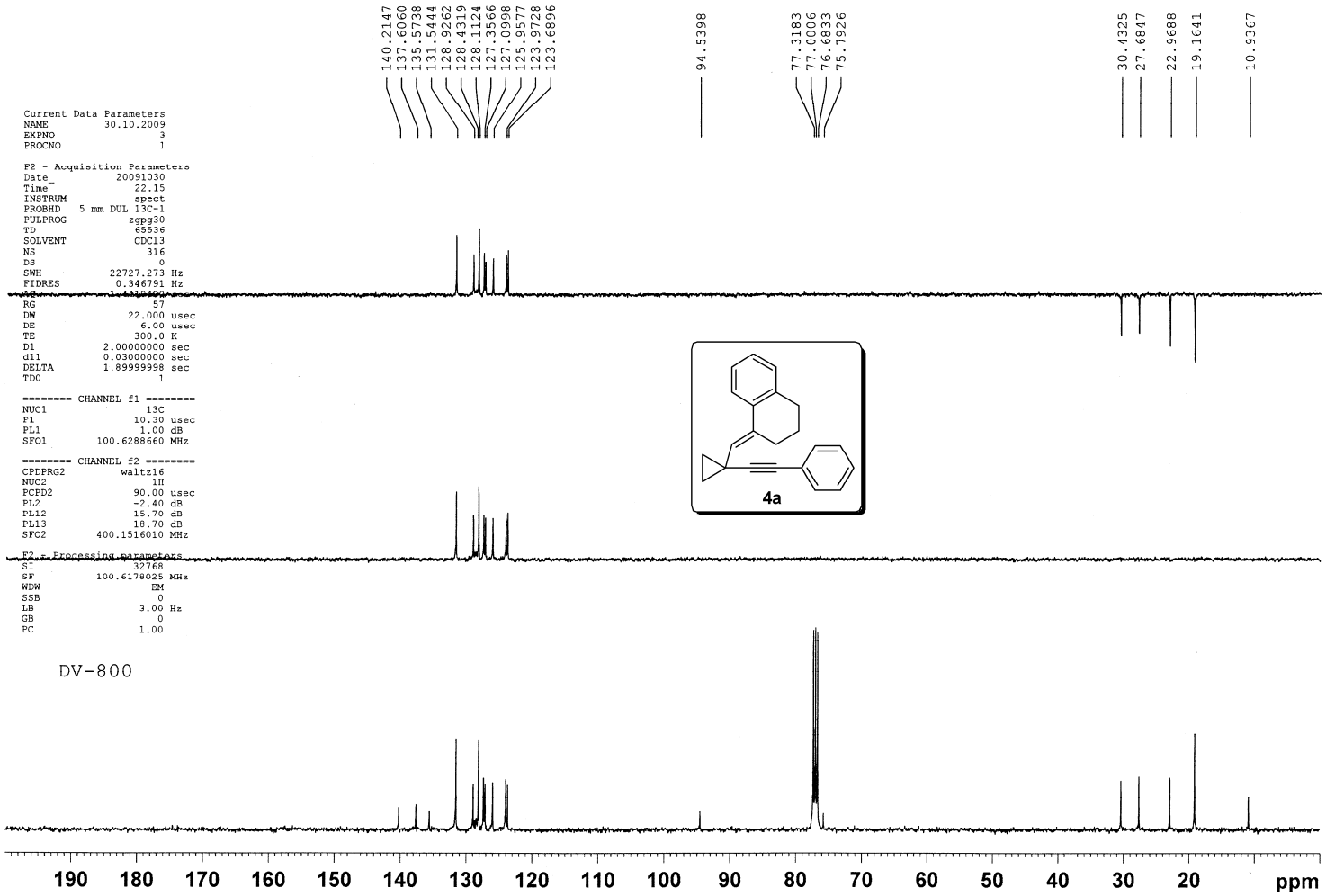
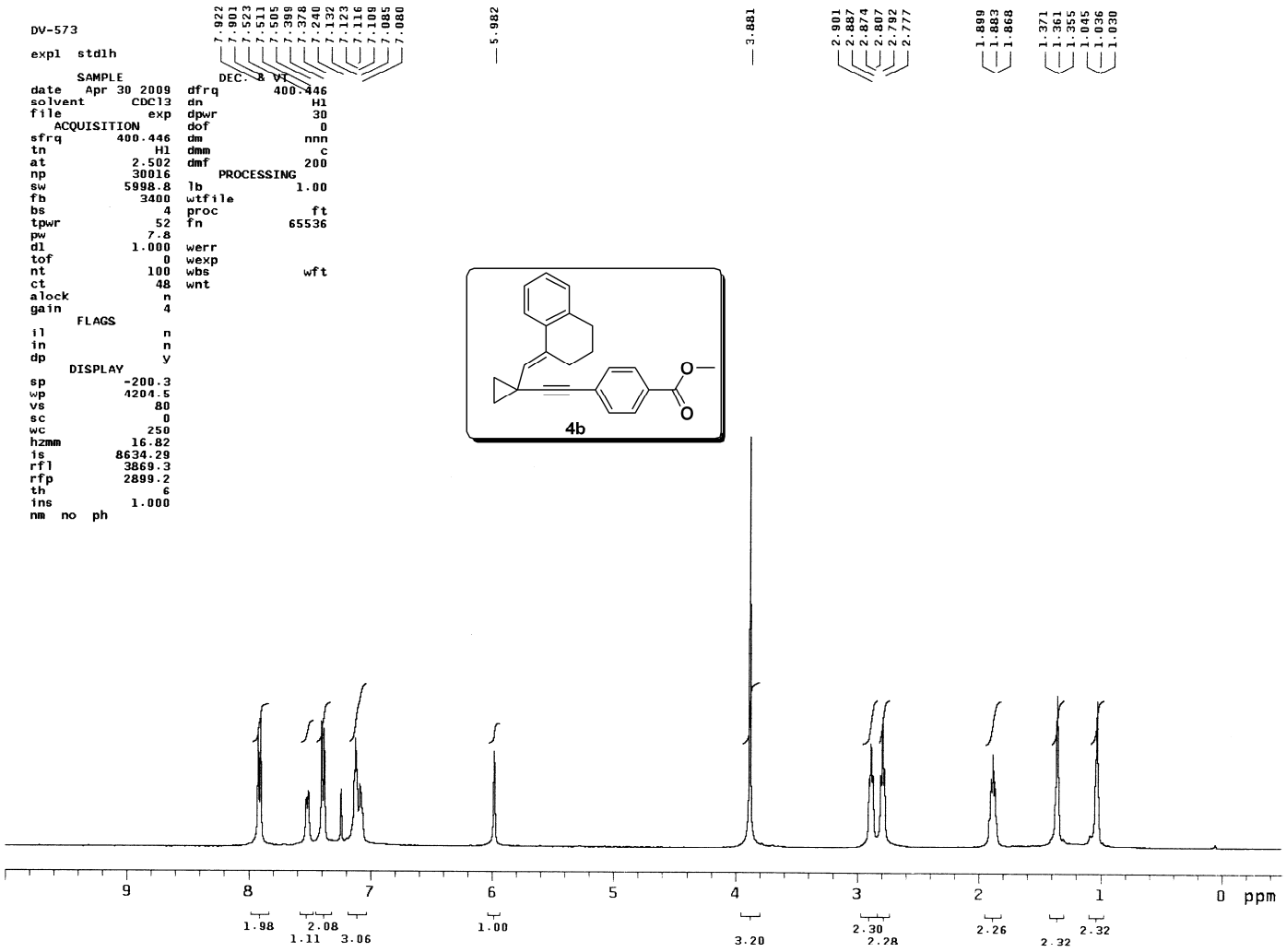
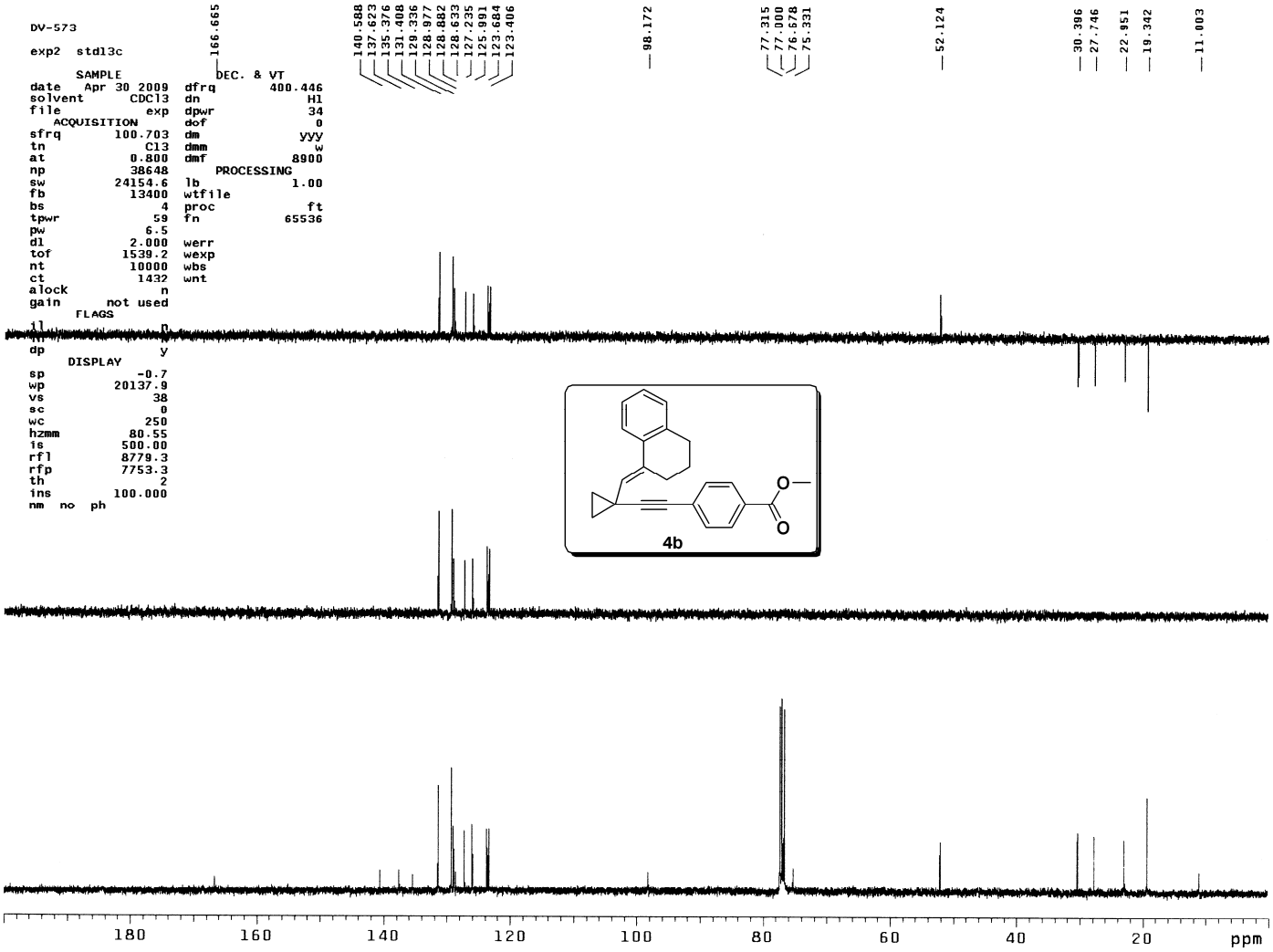
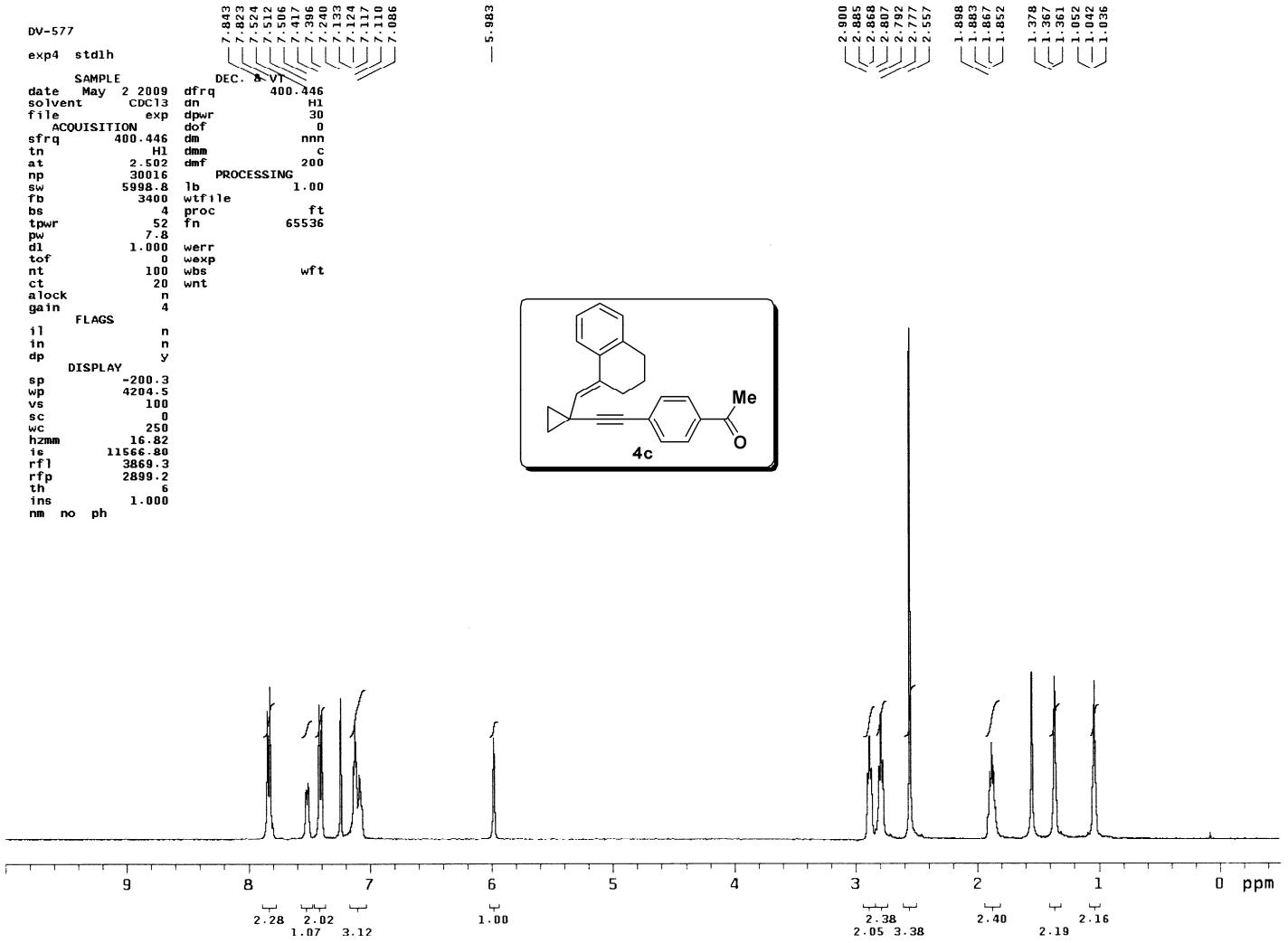
141.6597
139.2281
138.0353
128.5344
128.1505
126.7104
126.2606
122.9522
77.3251
77.0076
76.6903
33.1584
32.4404
28.7216
25.8520
15.9221
Current Data Parameters
NAME 5.11.2009
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091106
Time 0.35
INSTRUM spect
PROBHD 5 mm DUL 13C-1
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 810
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 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
PL13 18.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6177996 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
DV-806
Et
3a'
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

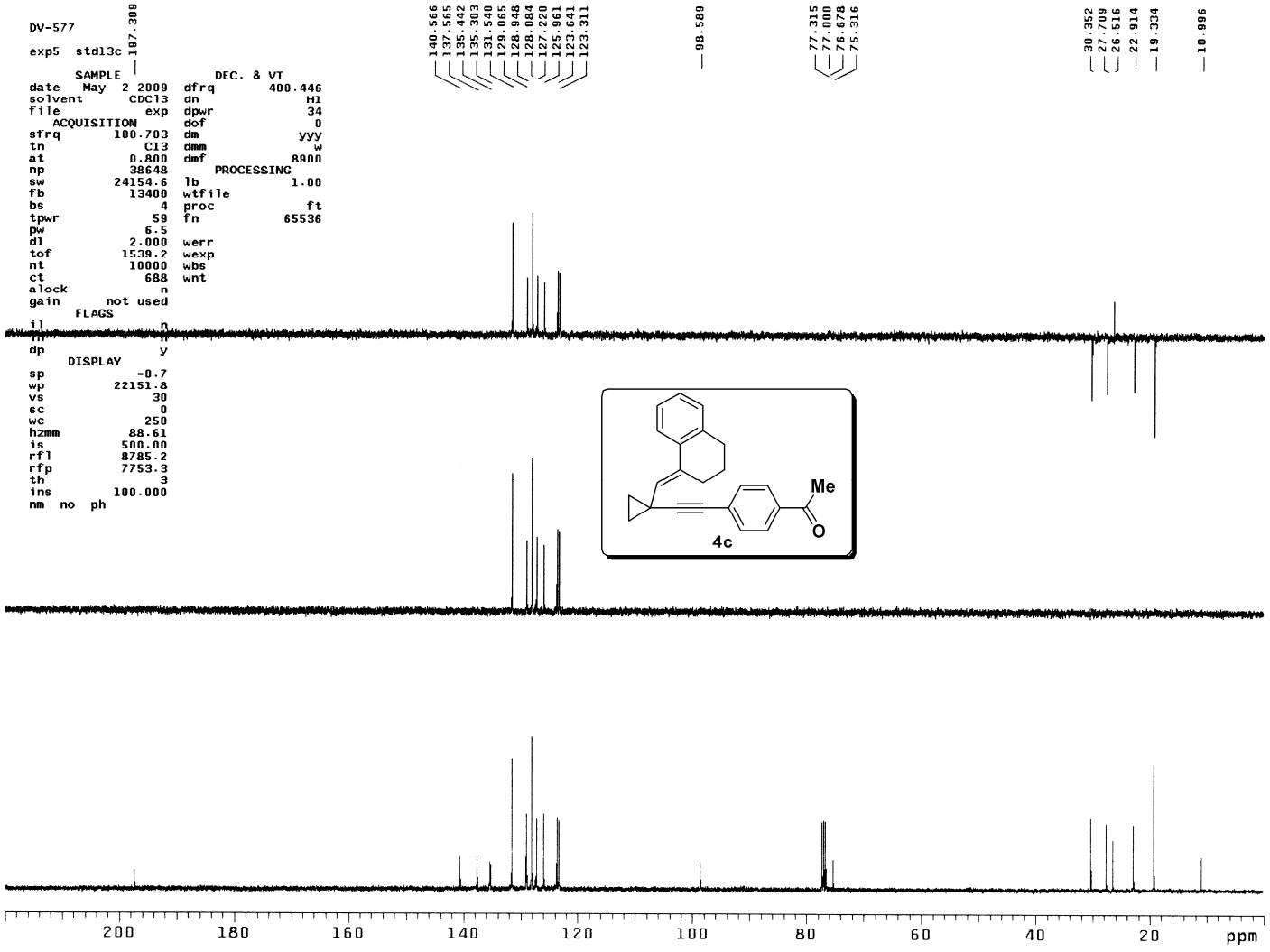


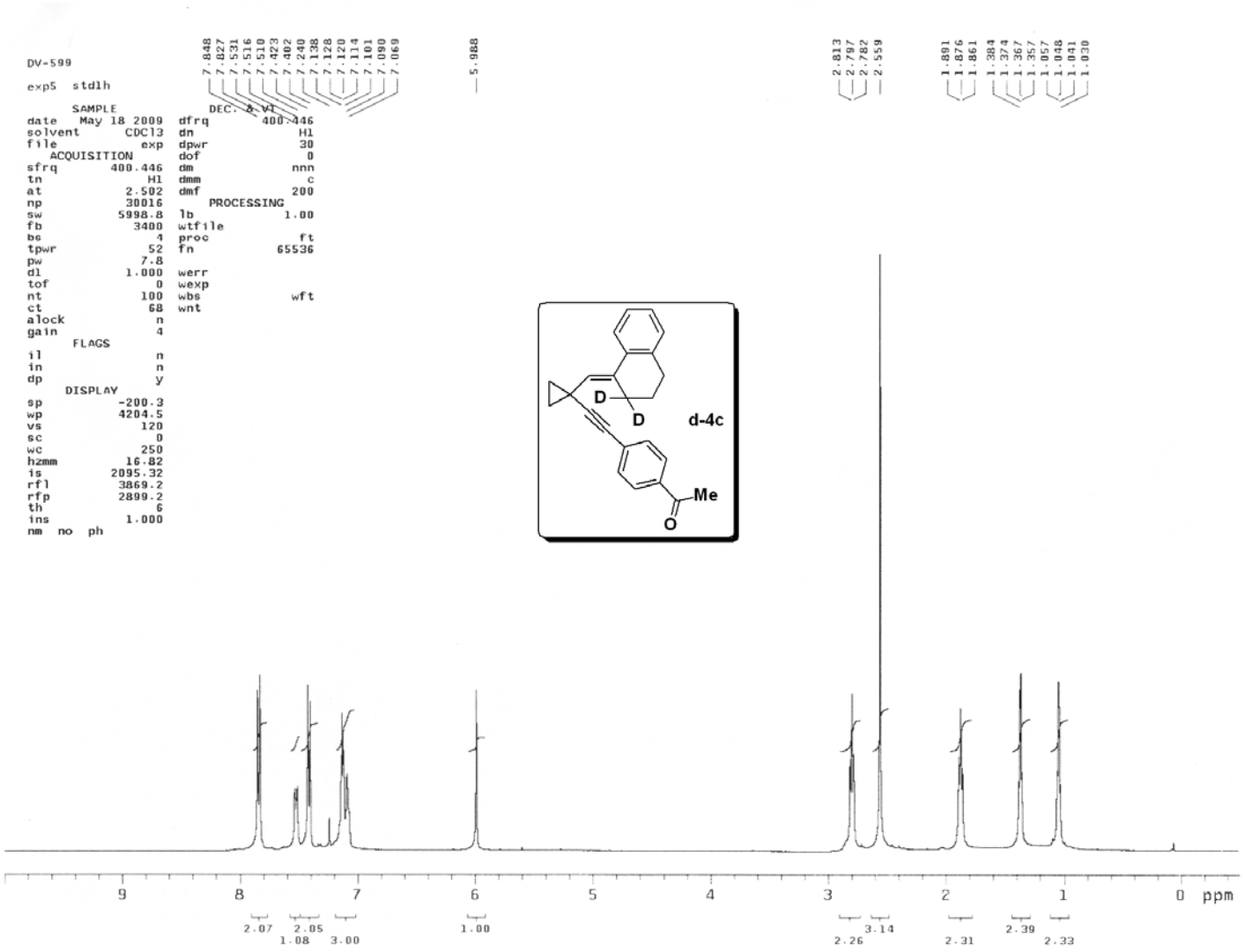


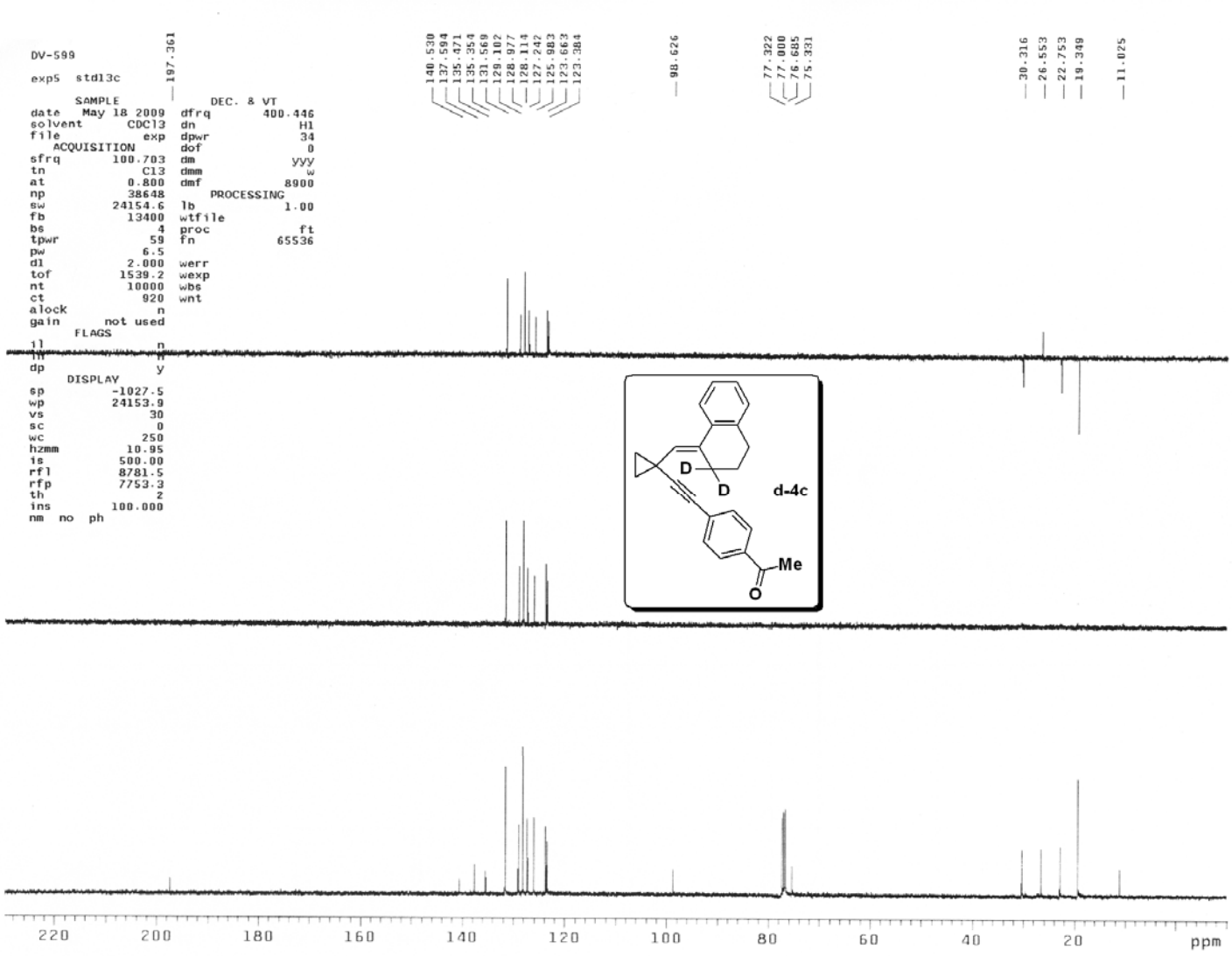


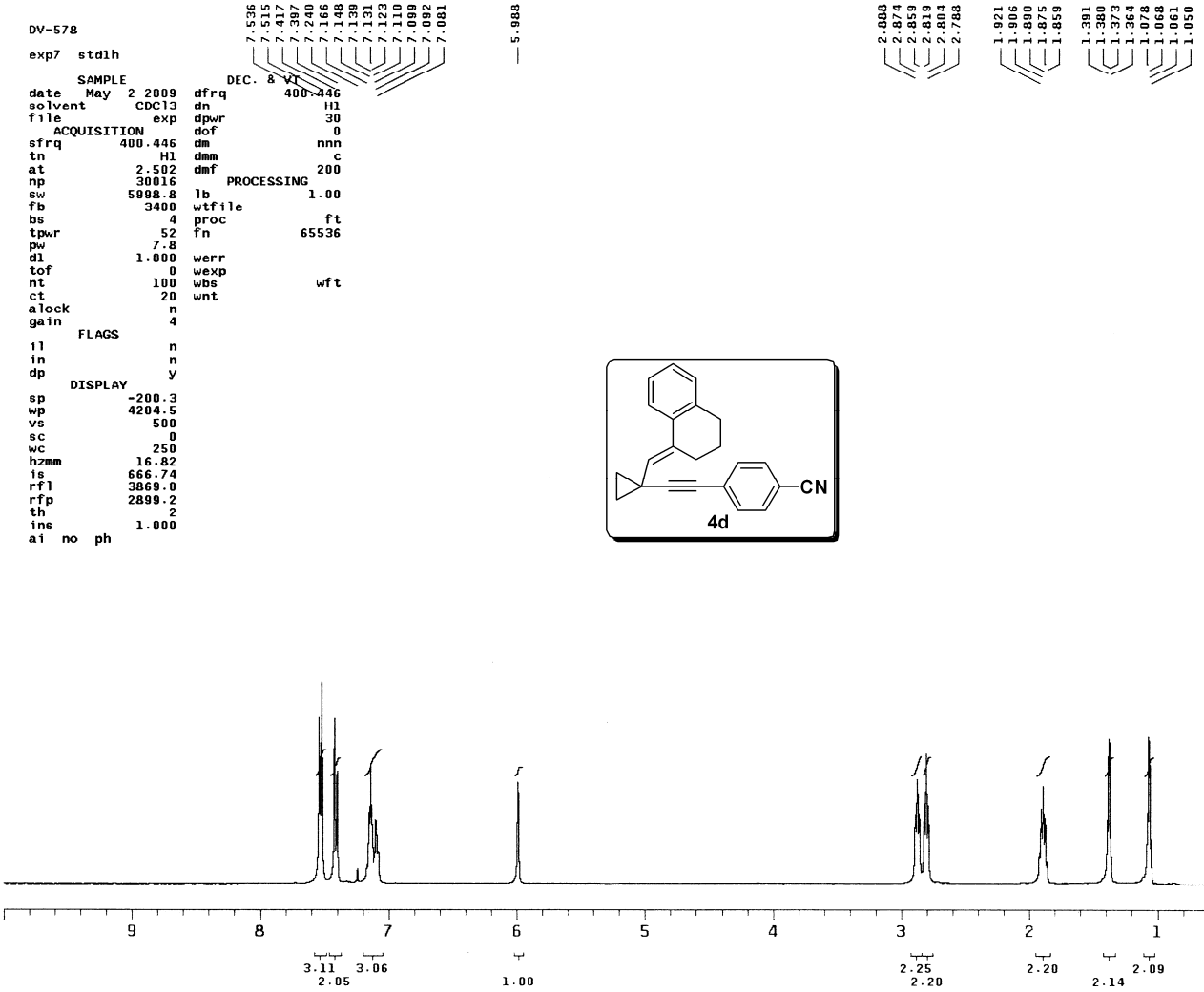











DV-578
exp7 stdlh
SAMPLE
date May 2 2009
solvent CDCl3
file exp
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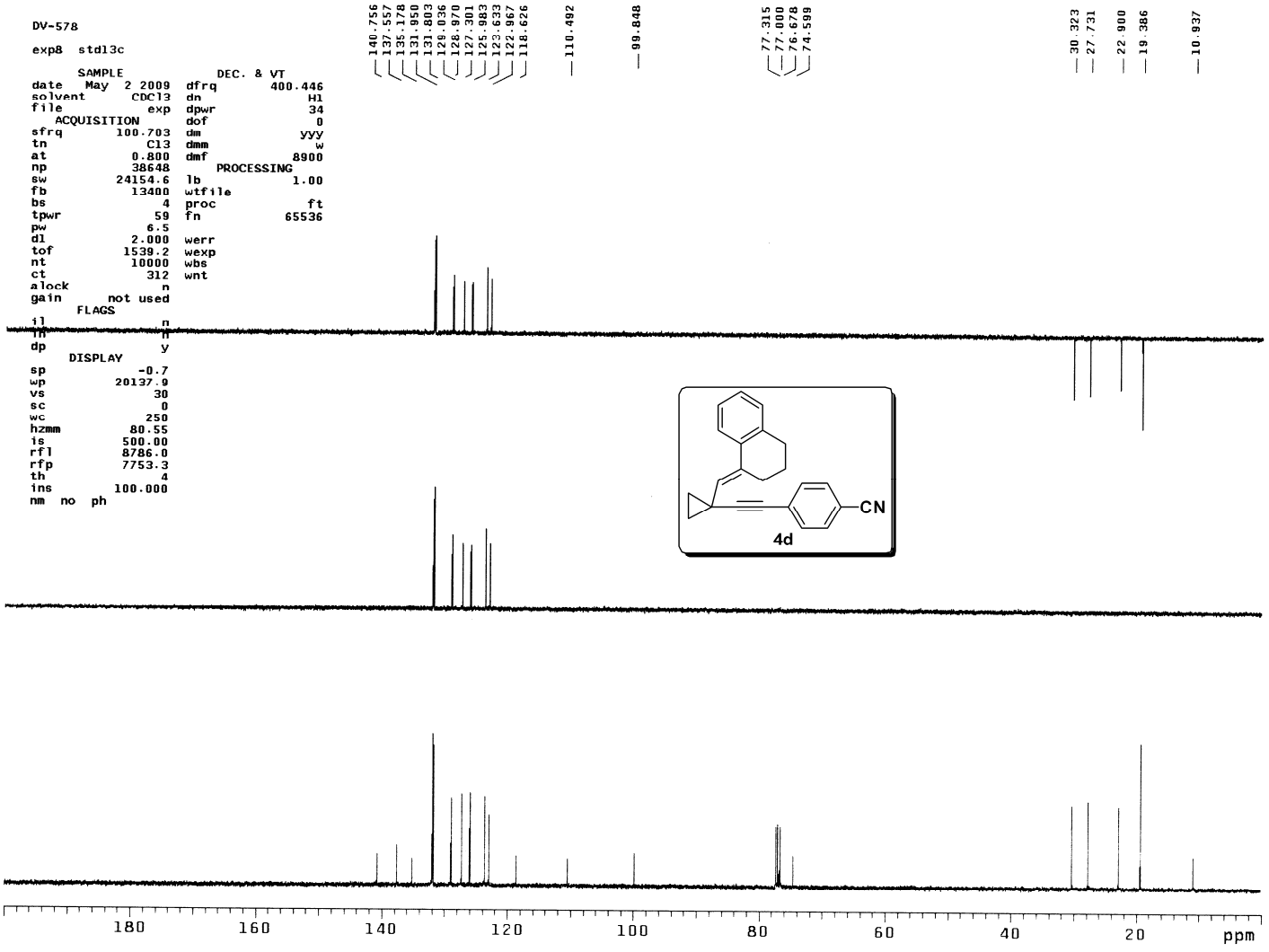
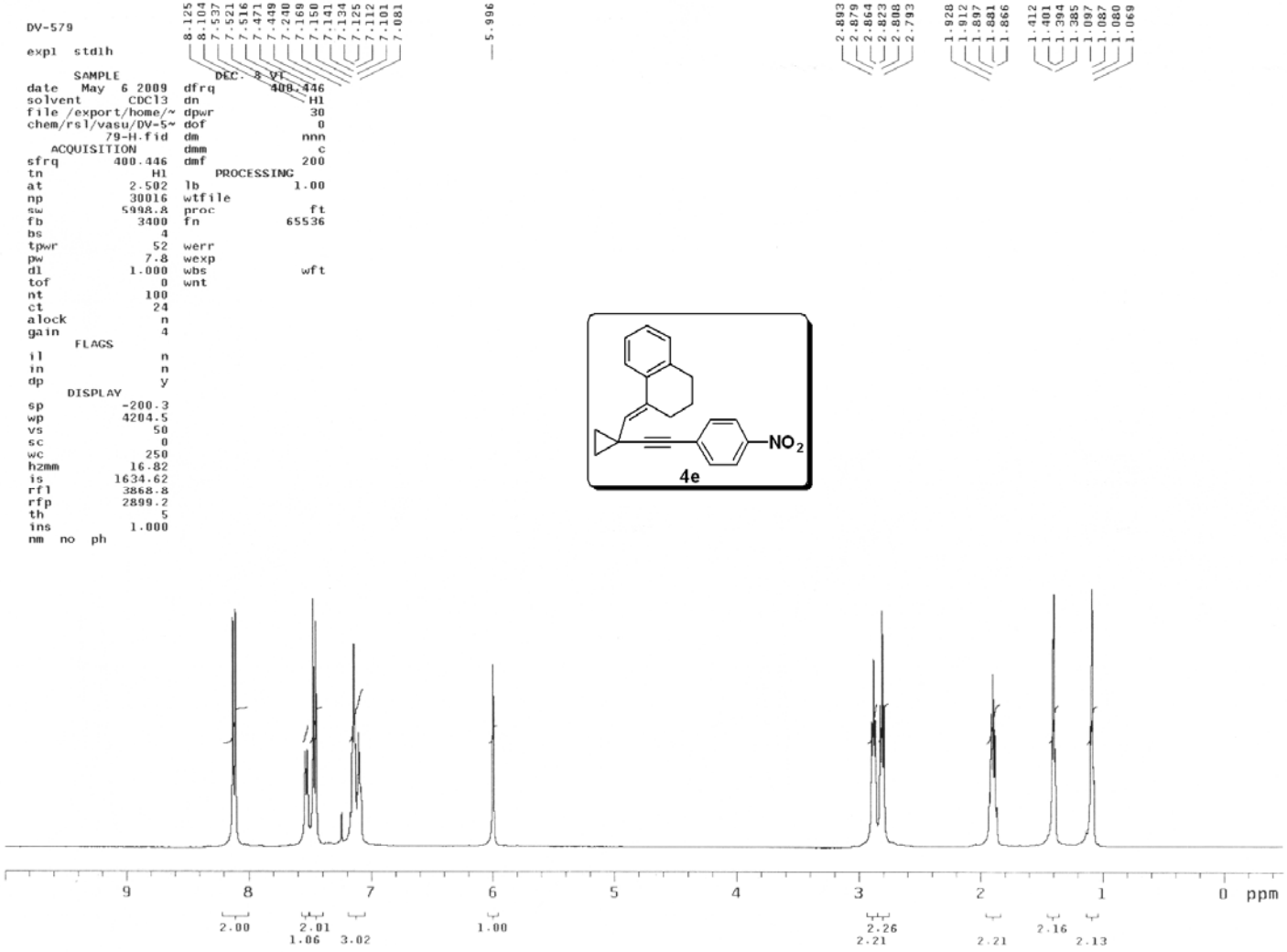
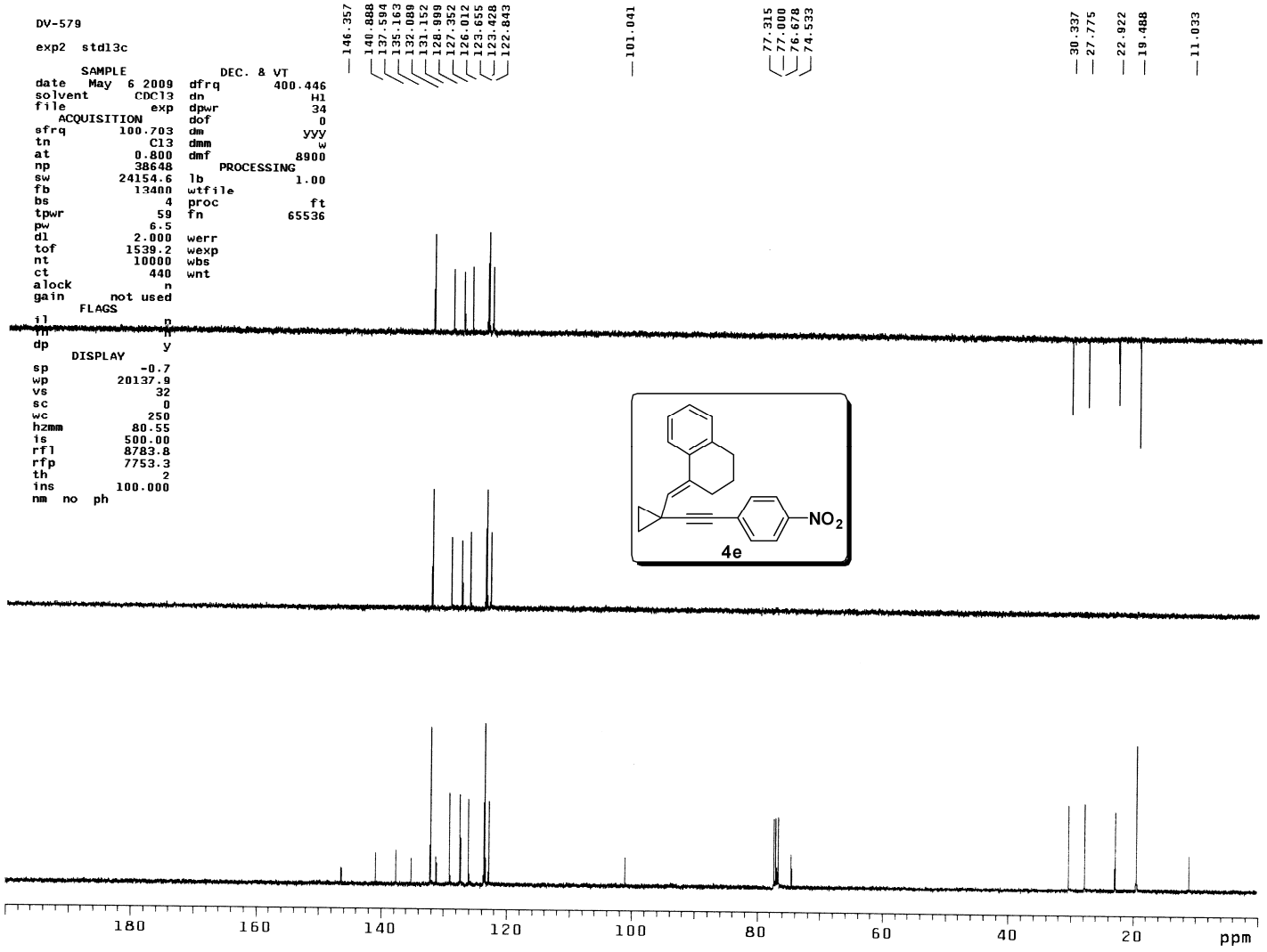
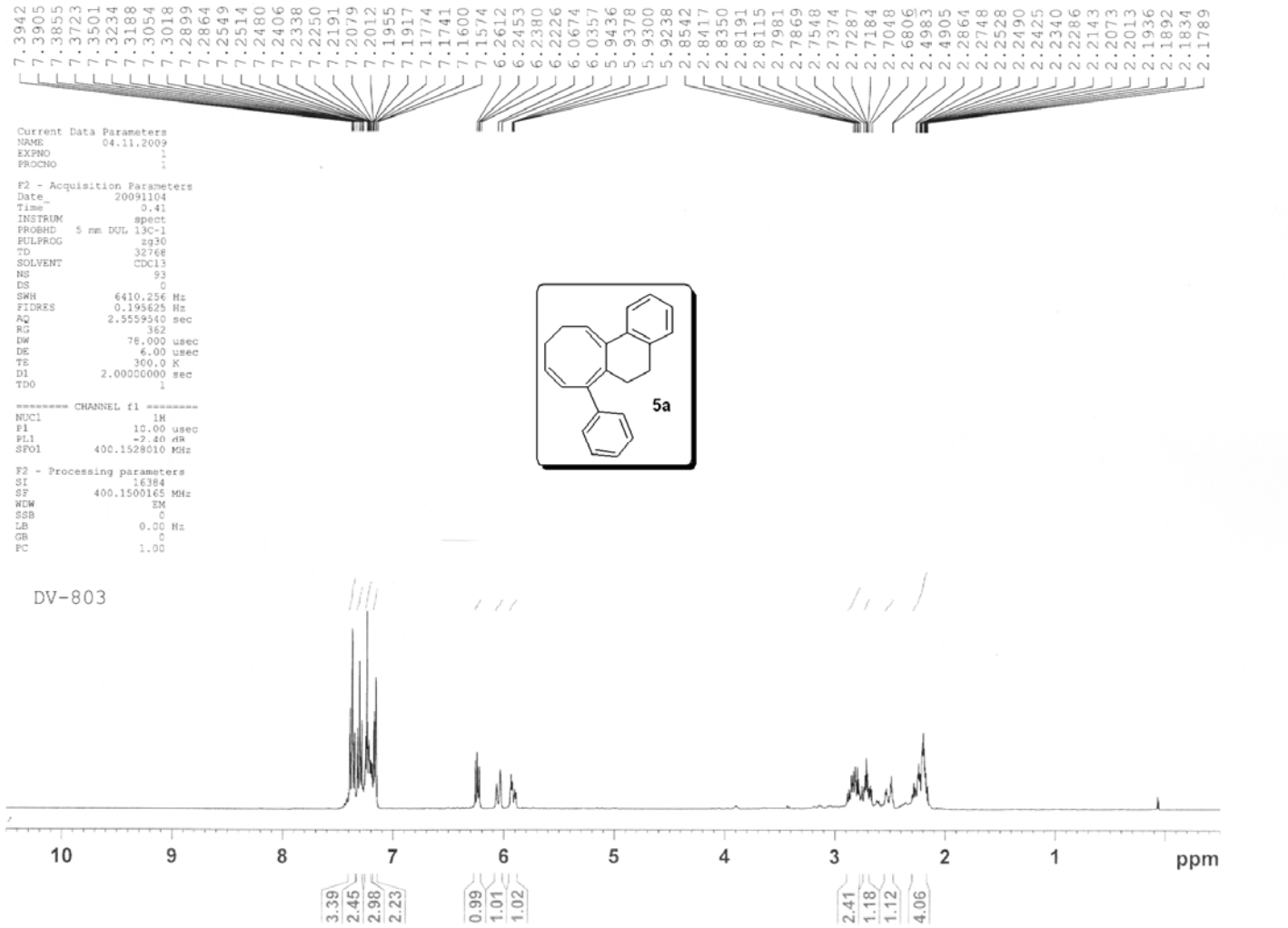
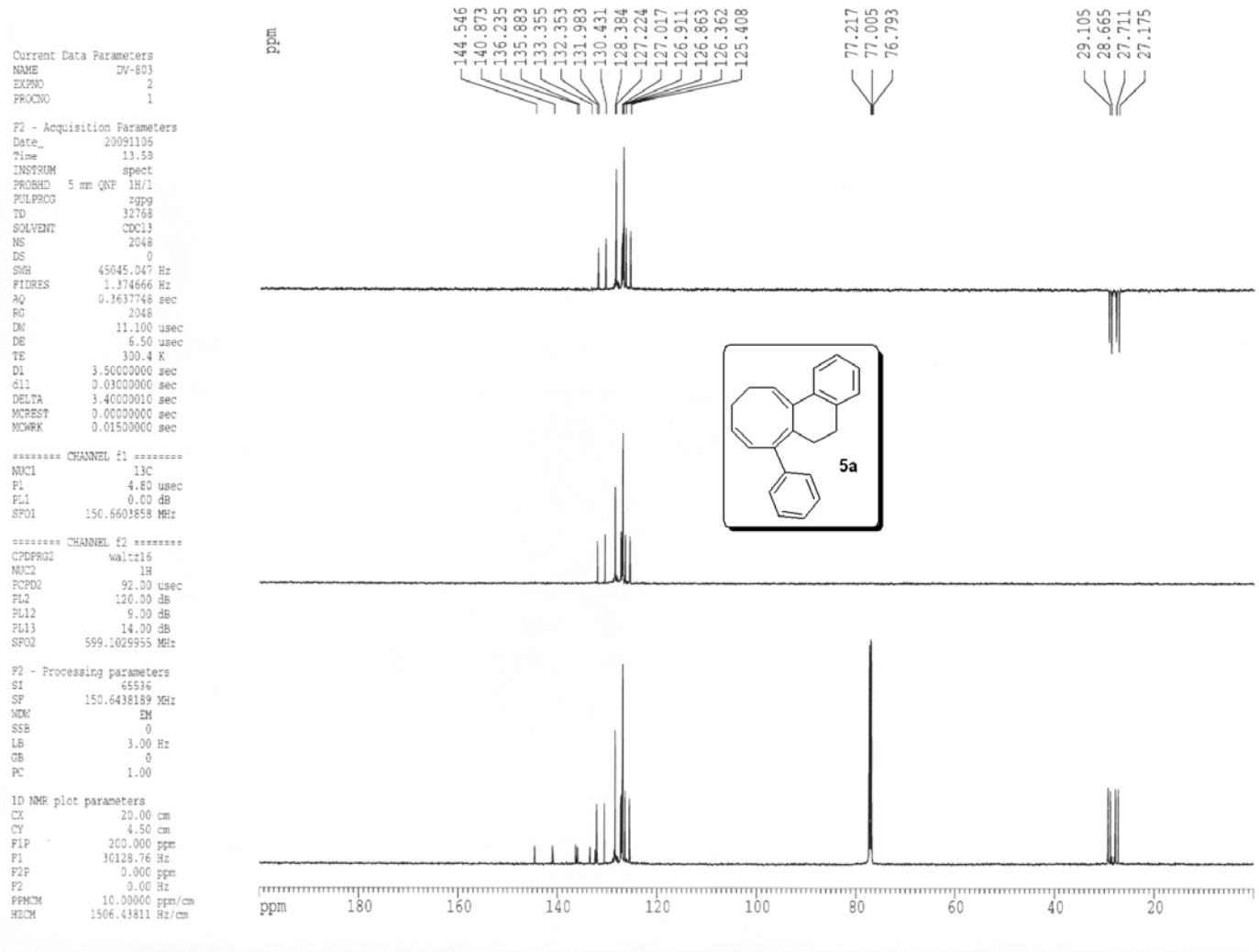
tn H1
at 2.502
np 30016
sw 5998.8
fb 3400
bs 4
tpwr 52
pw 7.8
dl 1.000
tof 0
nt 100
ct 20
alock n
gain 4
FLAGS
il n
in n
dp y
DISPLAY
sp -200.3
wp 4204.5
vs 500
sc 0
wc 250
hzmm 16.82
is 666.74
rfl 3869.0
rfp 2899.2
th 2
ins 1.000
ai no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs wft
wnt
7.536
7.515
7.417
7.397
7.240
7.166
7.148
7.139
7.131
7.123
7.110
7.099
7.092
7.081
5.988
2.888
2.874
2.859
2.819
2.804
2.788
1.921
1.906
1.890
1.875
1.859
1.391
1.380
1.373
1.364
1.078
1.068
1.061
1.050
CN
4d
9
8
7
6
5
4
3
2
1
0 ppm
3.11
2.05
3.06
1.00
2.25
2.20
2.20
2.14
2.09

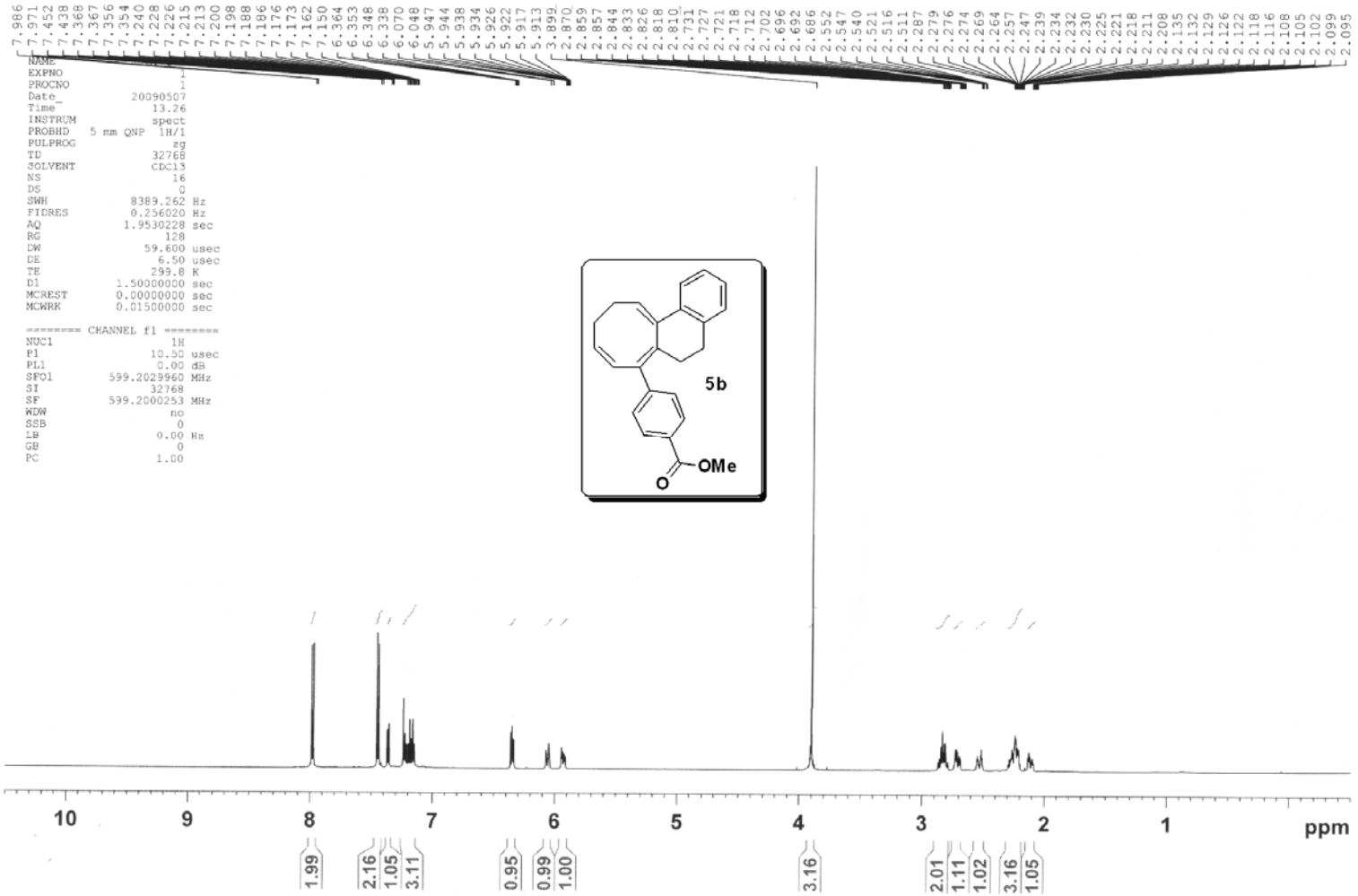


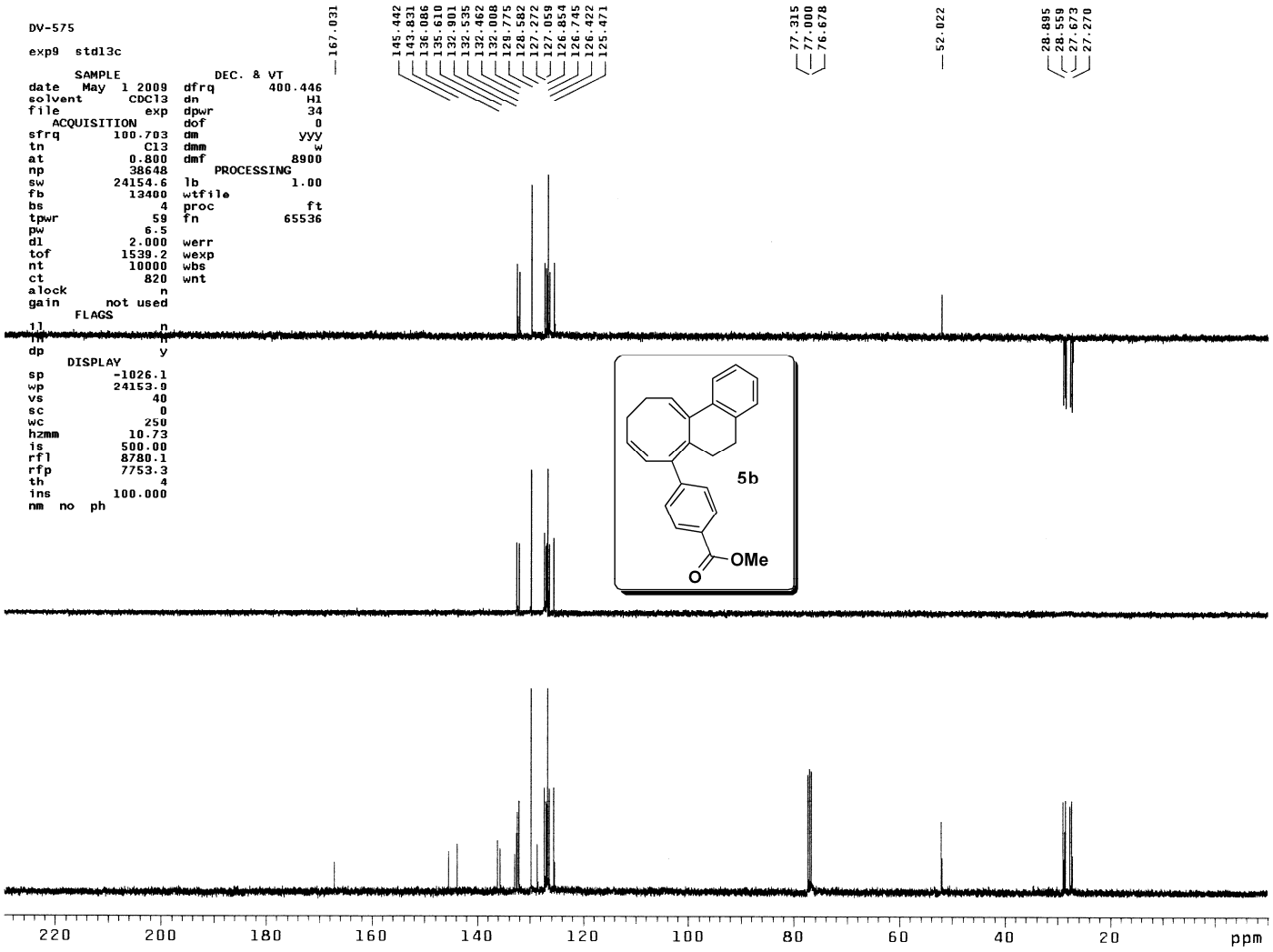


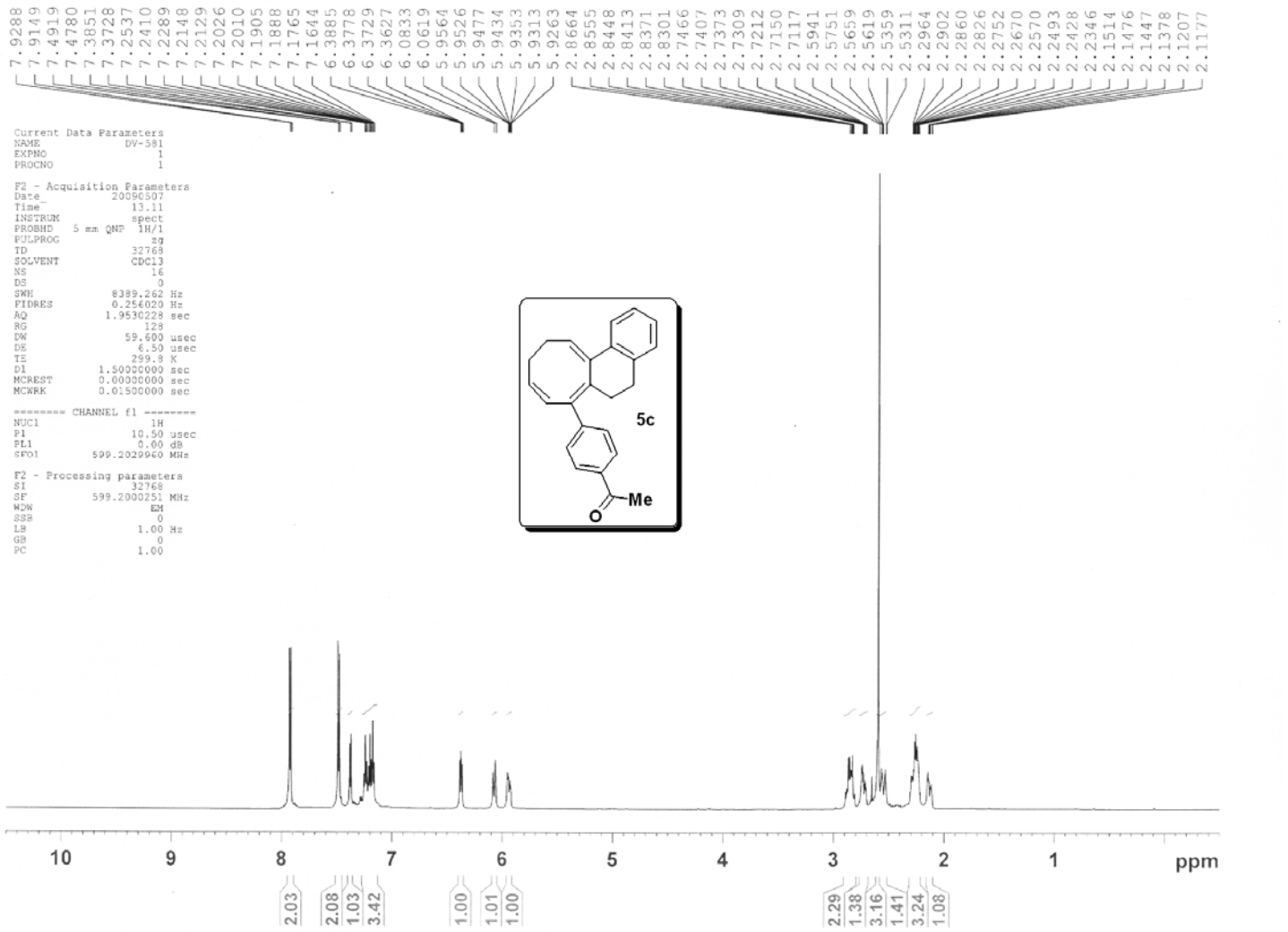


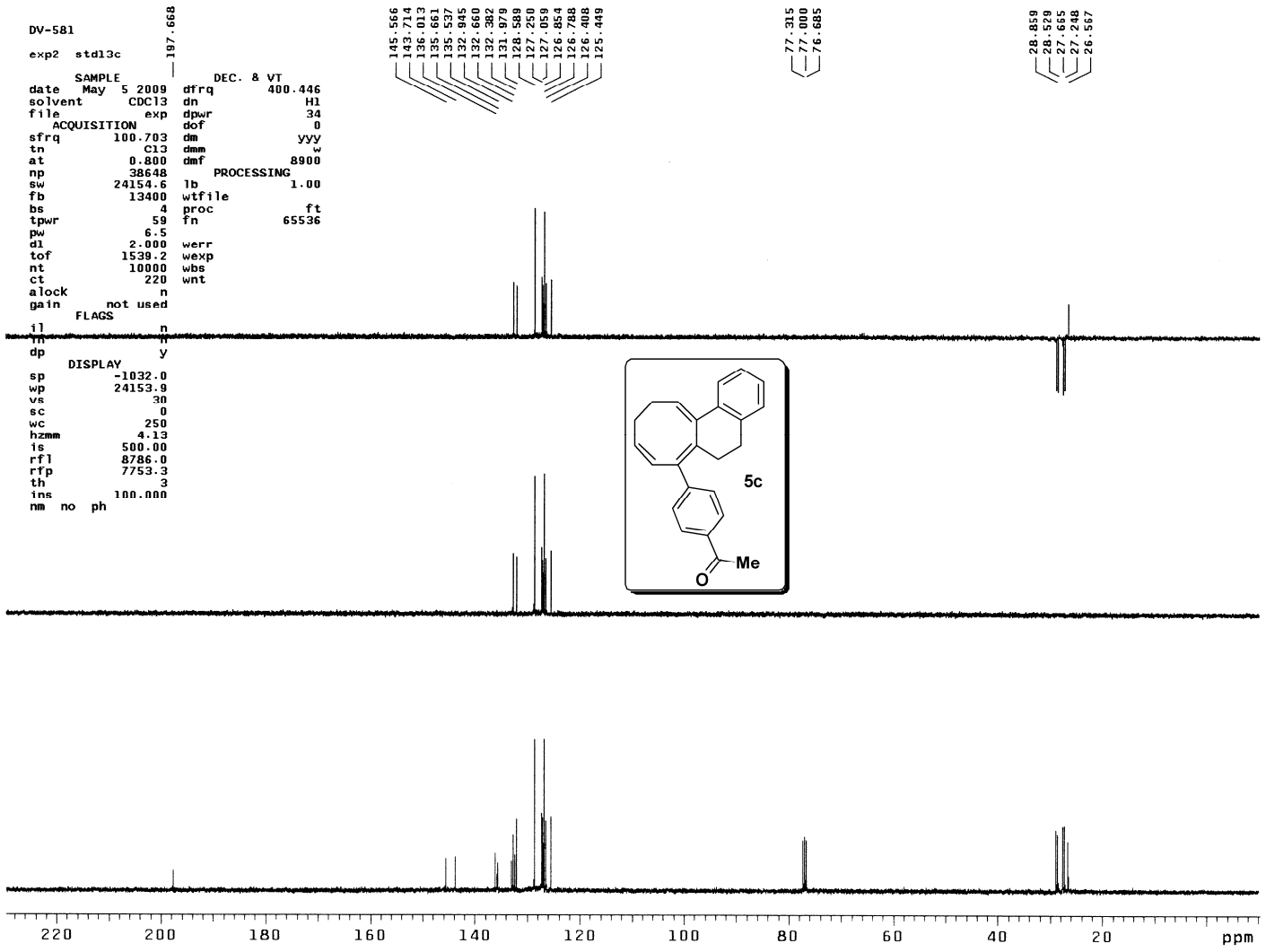











DV-581
exp2 std13c
SAMPLE
date May 5 2009
solvent CDCl3
file exp
ACQUISITION
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tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
d1 2.000
tof 1539.2
nt 10000
ct 220
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -1032.0
wp 24153.9
vs 30
sc 0
wc 250
hzmm 4.13
is 500.00
rfl 8786.0
rfp 7753.3
th 3
ins 100.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs
wnt
197.668
145.566
143.714
136.013
135.661
135.537
132.945
132.660
132.382
131.979
128.589
127.250
127.059
126.854
126.788
126.408
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77.000
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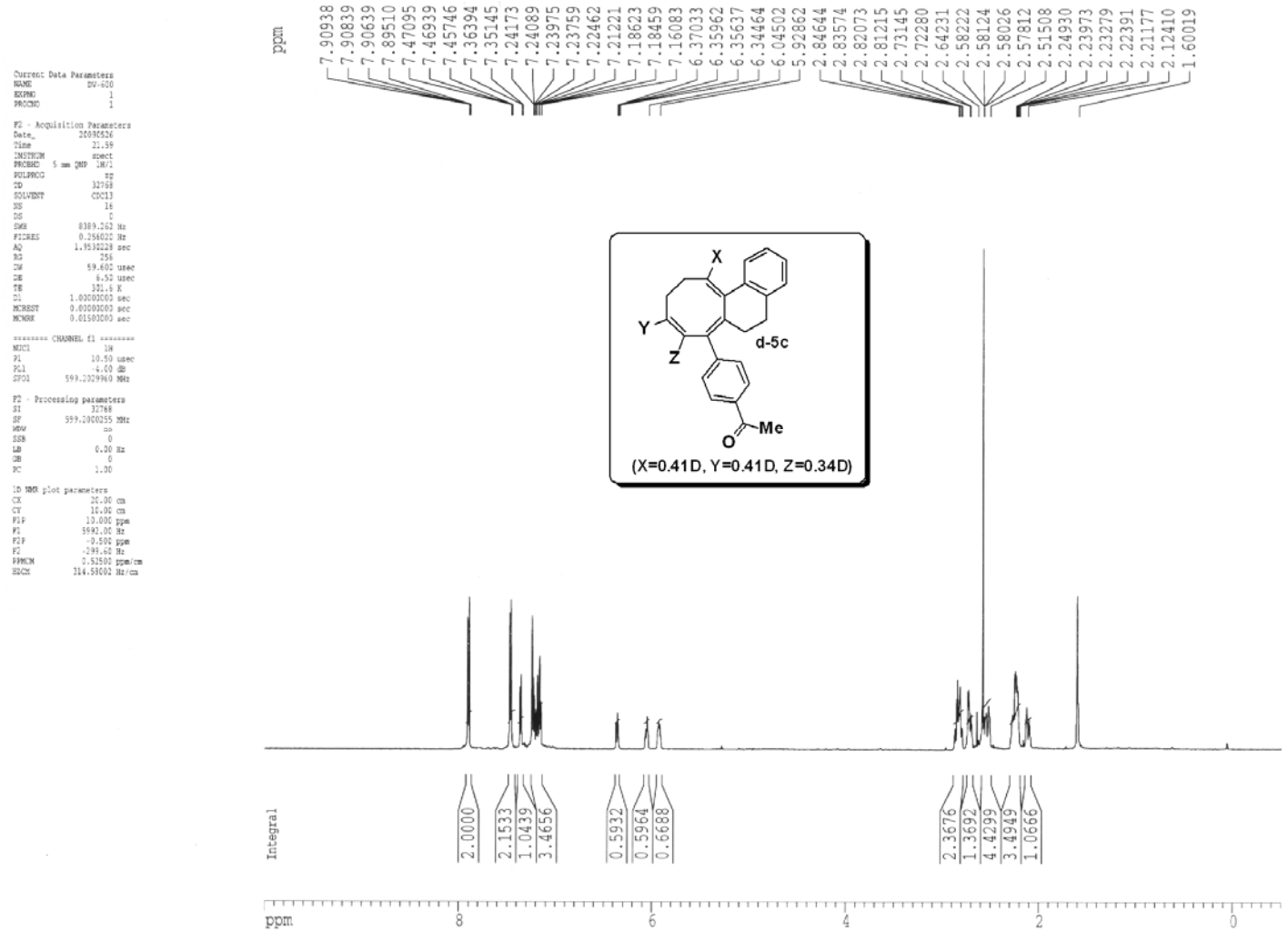
28.529
27.665
27.248
26.567
5c
Me
O
220
200
180
160
140
120
100
80
60
40
20
ppm


Current Data Parameters
NAME DV-620
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
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Time 21.59
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PROBHD 5 mm QNP 1H/1
PULPROG zg
TD 32768
SOLVENT CDCl3
NS 16
DS 0
SWH 8389.262 Hz
FIDRES 0.256022 Hz
AQ 1.9535228 sec
RG 256
DW 59.601 usec
DE 6.50 usec
TE 321.1 K
D1 1.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 ========
NUC1 1H
P1 10.50 usec
PL1 -4.00 dB
SFO1 599.2029960 MHz
F2 - Processing parameters
SI 32768
SF 599.2000255 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 5992.00 Hz
F2P -0.500 ppm
F2 -299.60 Hz
PPMCM 0.52500 ppm/cm
HZCM 314.59003 Hz/cm
ppm
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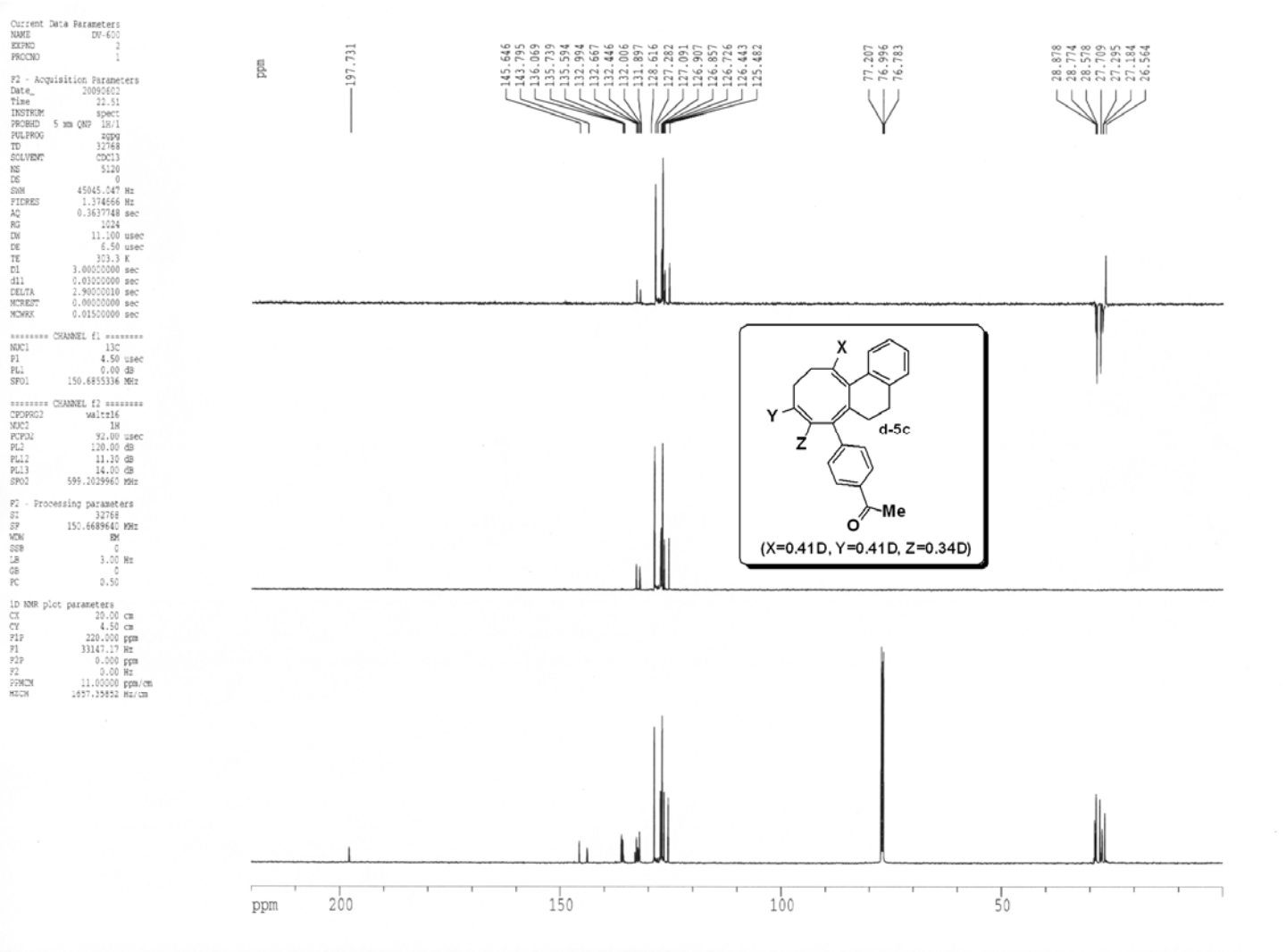
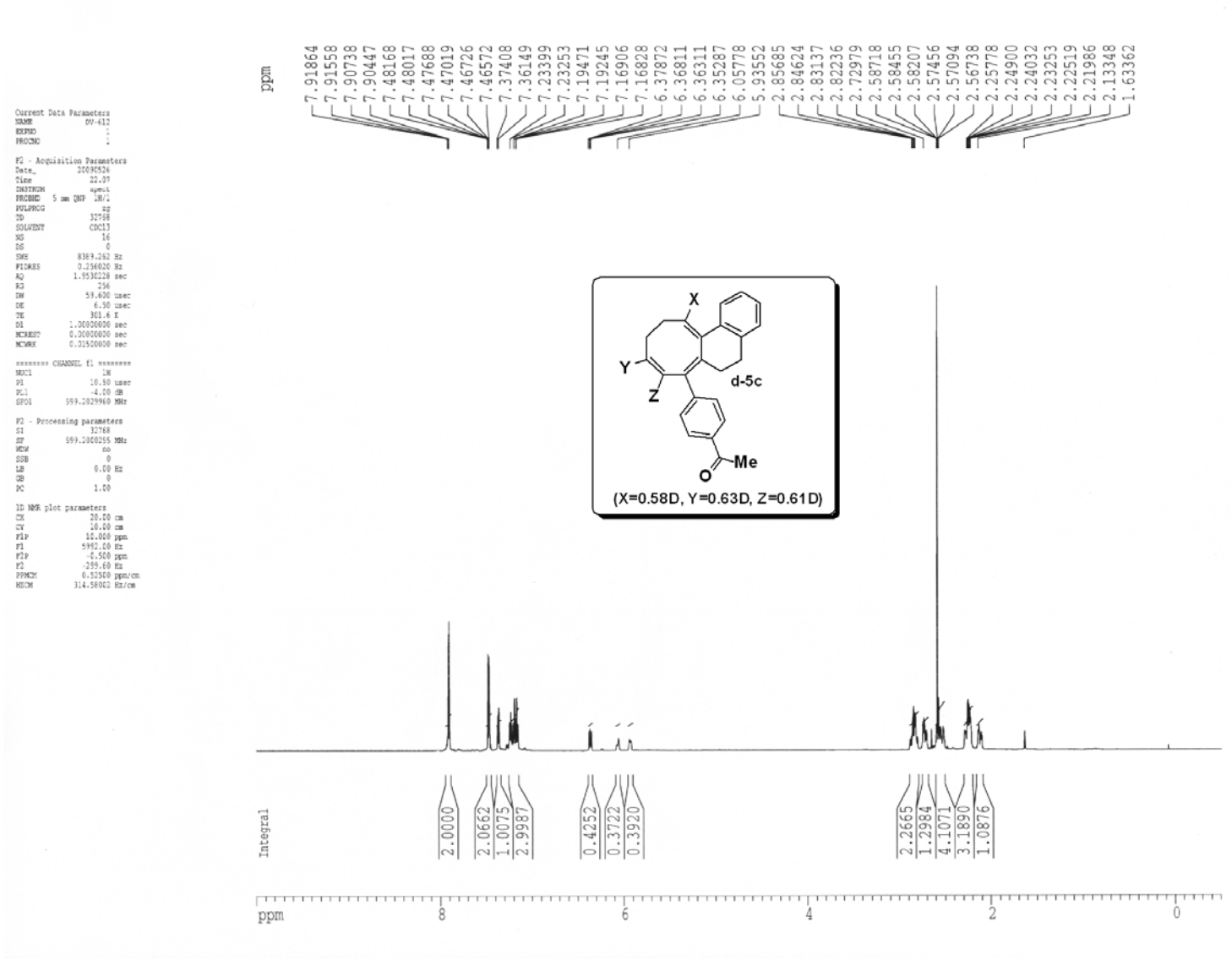
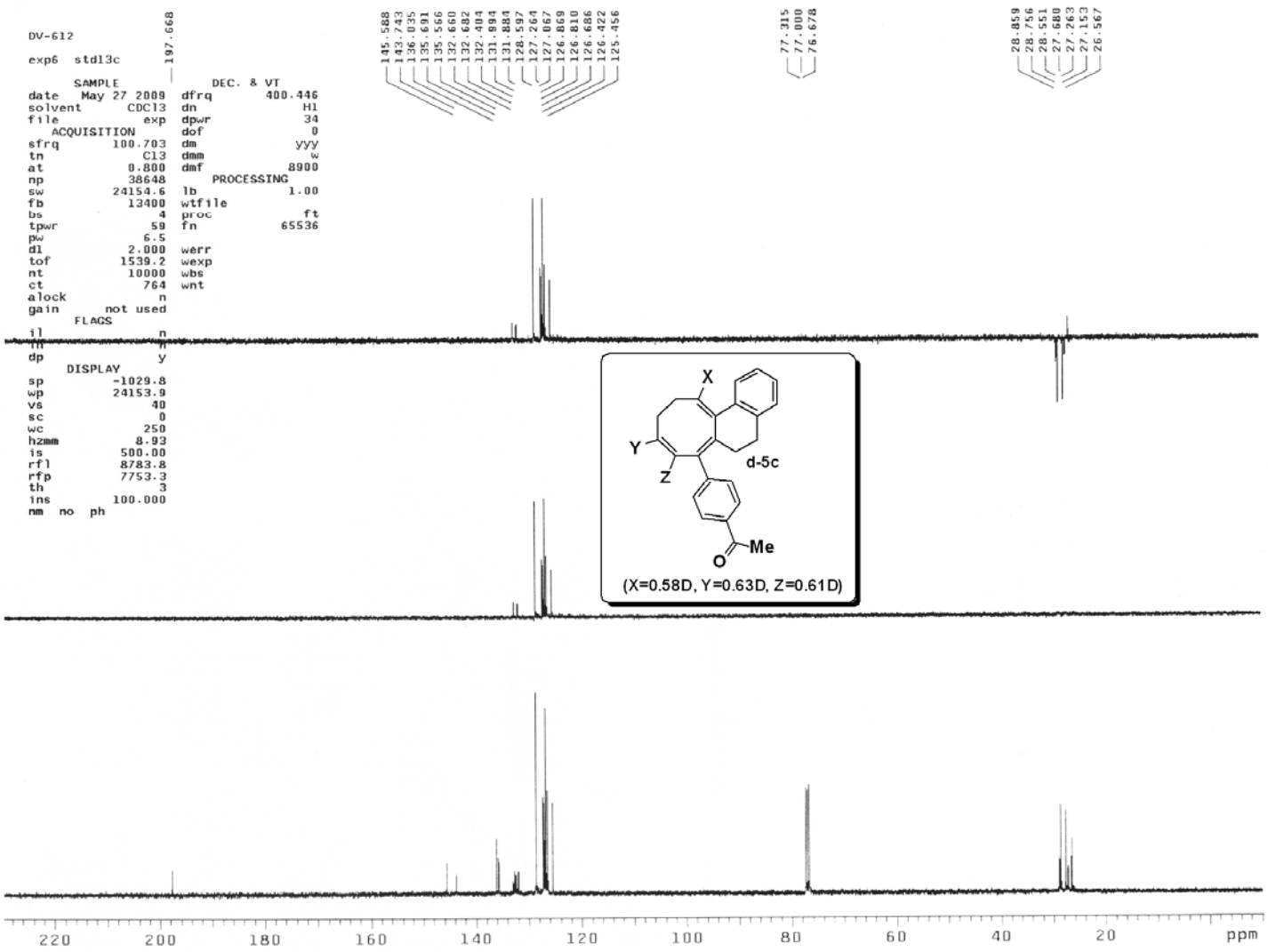
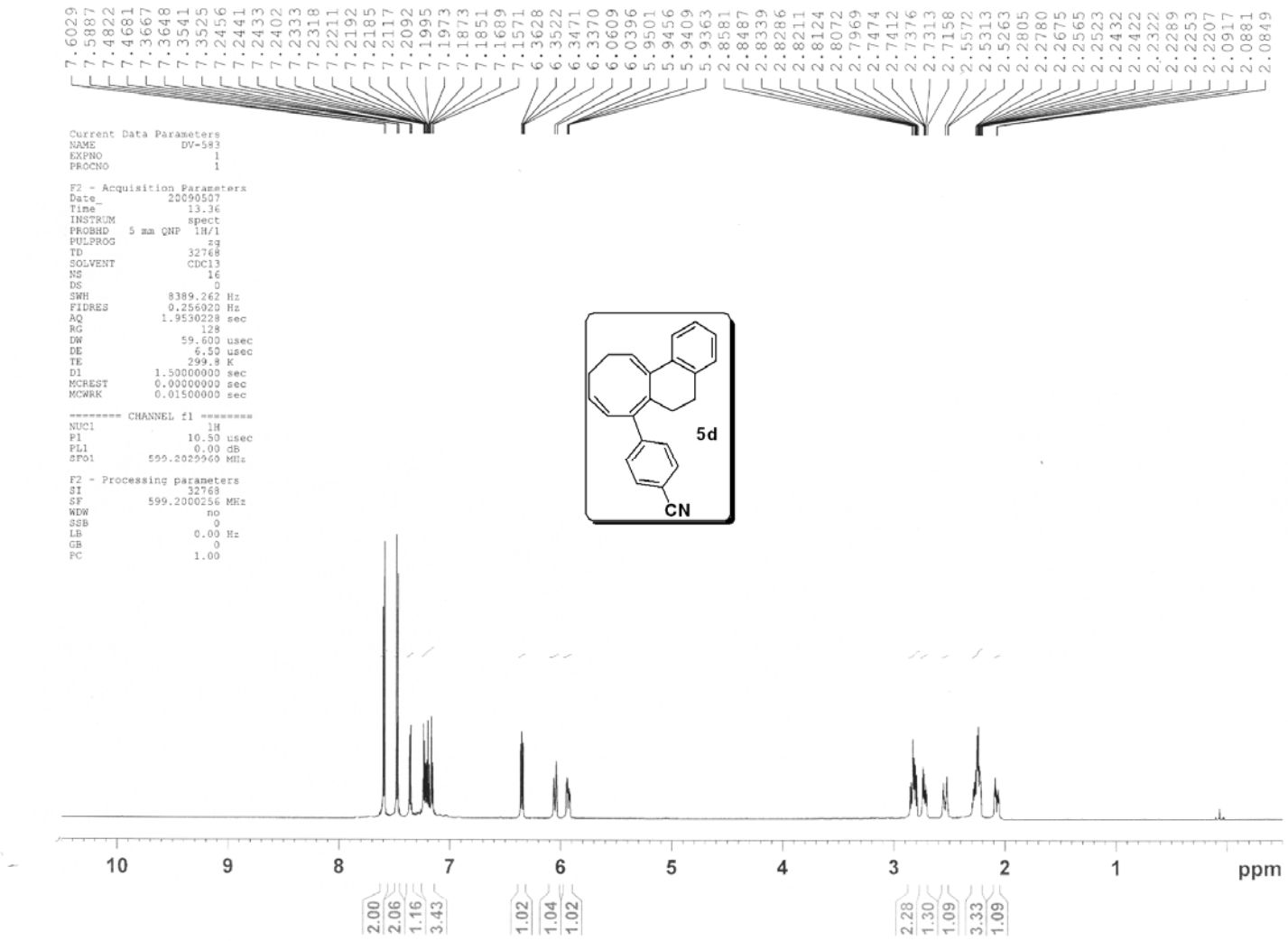
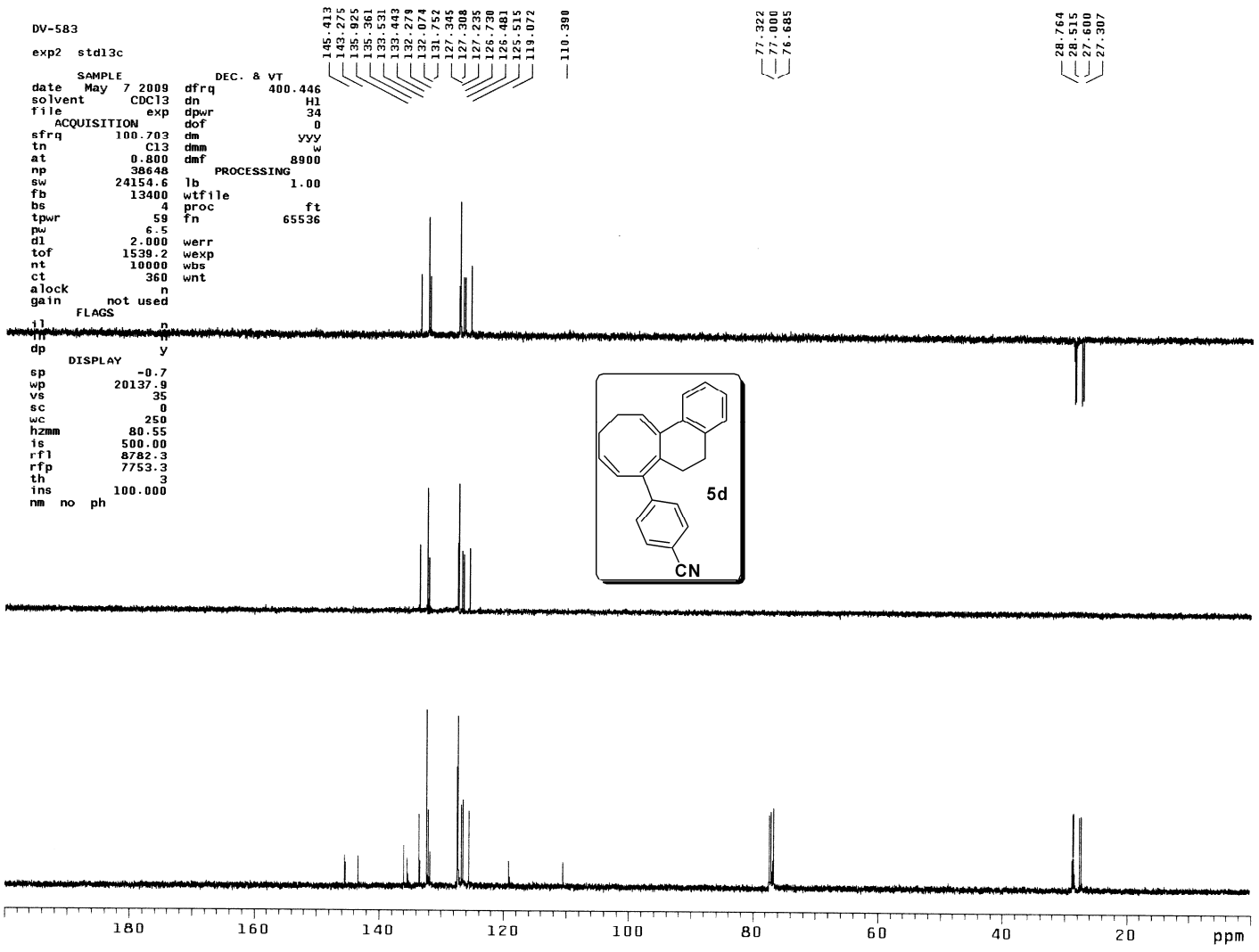
7.90839
7.90639
7.89510
7.47095
7.46939
7.45746
7.36394
7.35145
7.24173
7.24089
7.23975
7.23759
7.22462
7.21221
7.18623
7.18459
7.16083
6.37033
6.35962
6.35637
6.34464
6.04502
5.92862
2.84644
2.83574
2.82073
2.81215
2.73145
2.72280
2.64231
2.58222
2.58124
2.58026
2.57812
2.51508
2.24930
2.23973
2.23279
2.22391
2.21177
2.12410
1.60019
X
Y
Z
d-5c
Me
O
(X=0.41D, Y=0.41D, Z=0.34D)
Integral
2.0000
2.1533
1.0439
3.4656
0.5932
0.5964
0.6688
2.3676
1.3692
4.4299
3.4949
1.0666
ppm
8
6
4
2
0

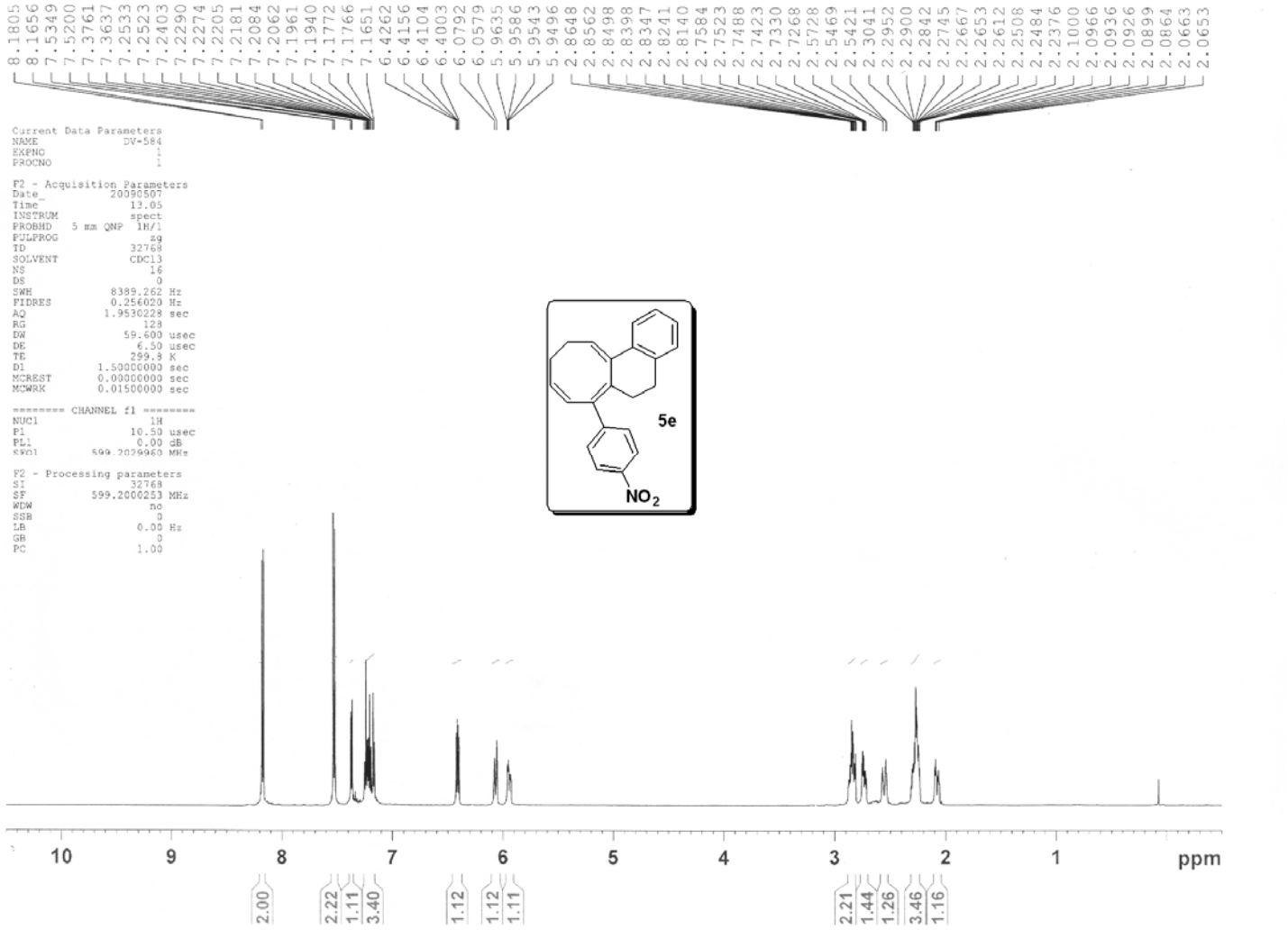


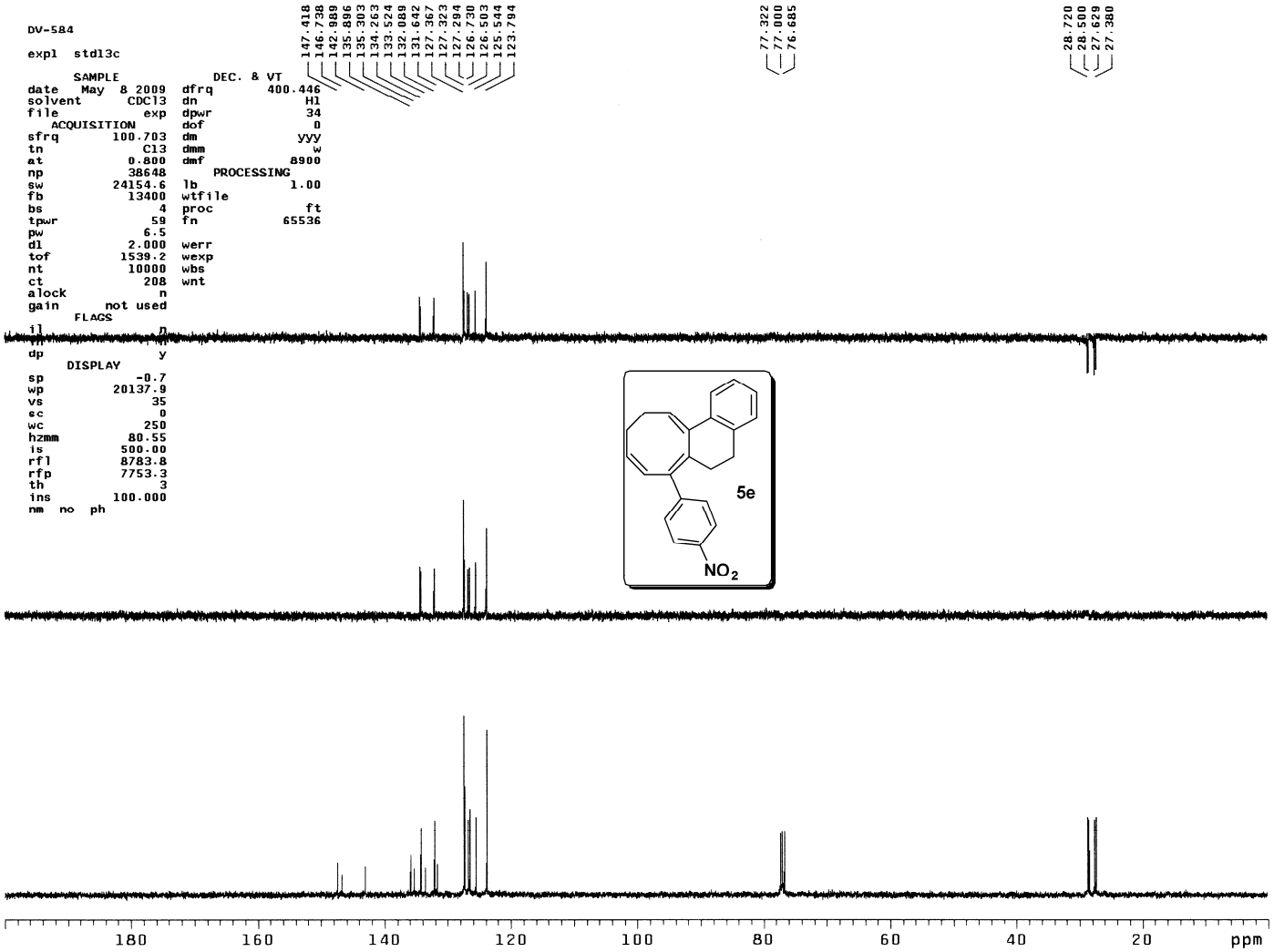











DV-584
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SAMPLE
date May 8 2009
solvent CDCl3
file exp
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tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
d1 2.000
tof 1539.2
nt 10000
ct 208
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.7
wp 20137.9
vs 35
sc 0
wc 250
hzmm 80.55
is 500.00
rfl 8783.8
rfp 7753.3
th 3
ins 100.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs
wnt
147.418
146.738
142.989
135.896
135.303
134.263
133.524
132.089
131.642
127.367
127.323
127.294
126.730
126.503
125.544
123.794
77.322
77.000
76.685
28.720
28.500
27.629
27.380
5e
NO2
180
160
140
120
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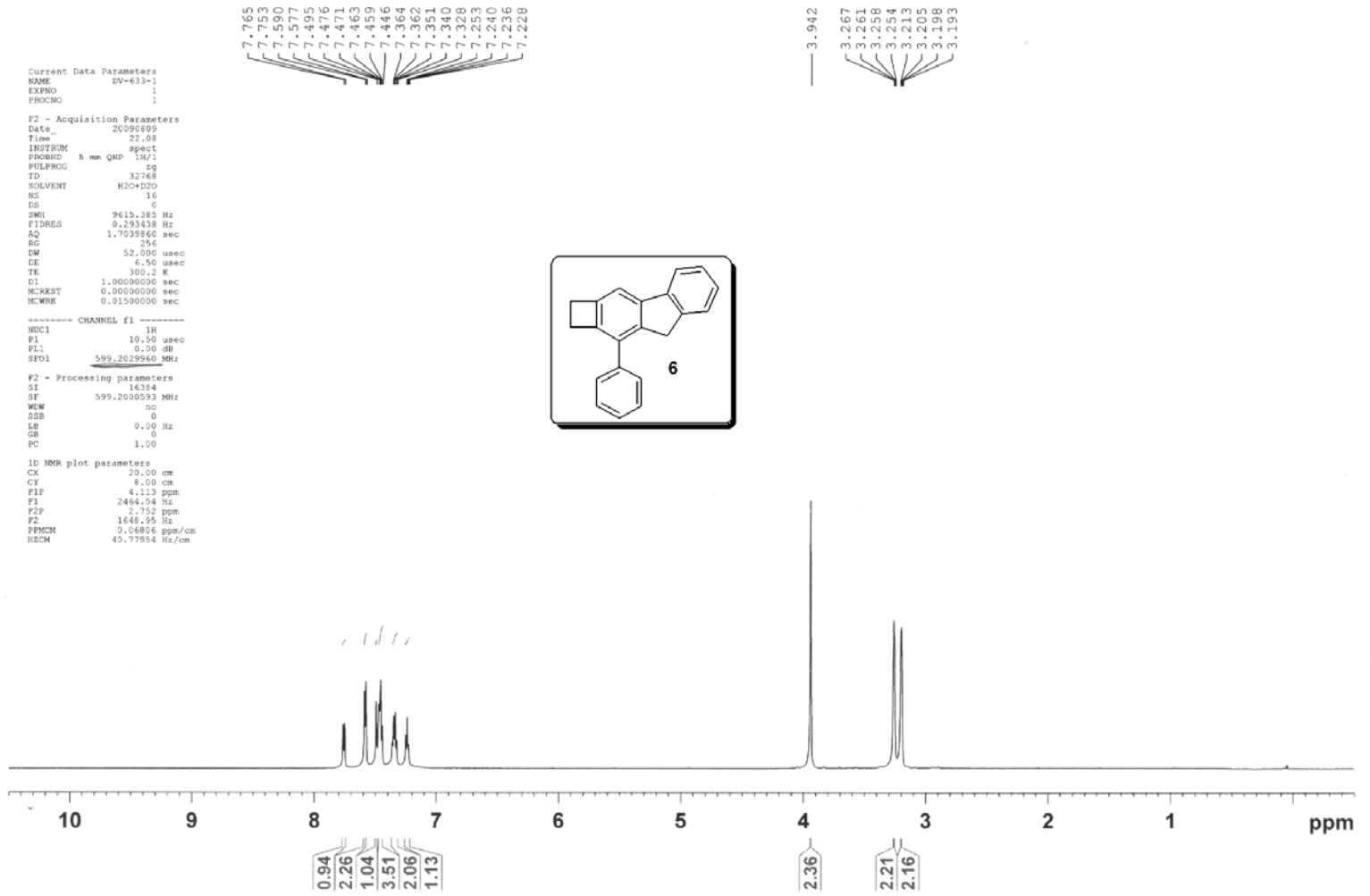
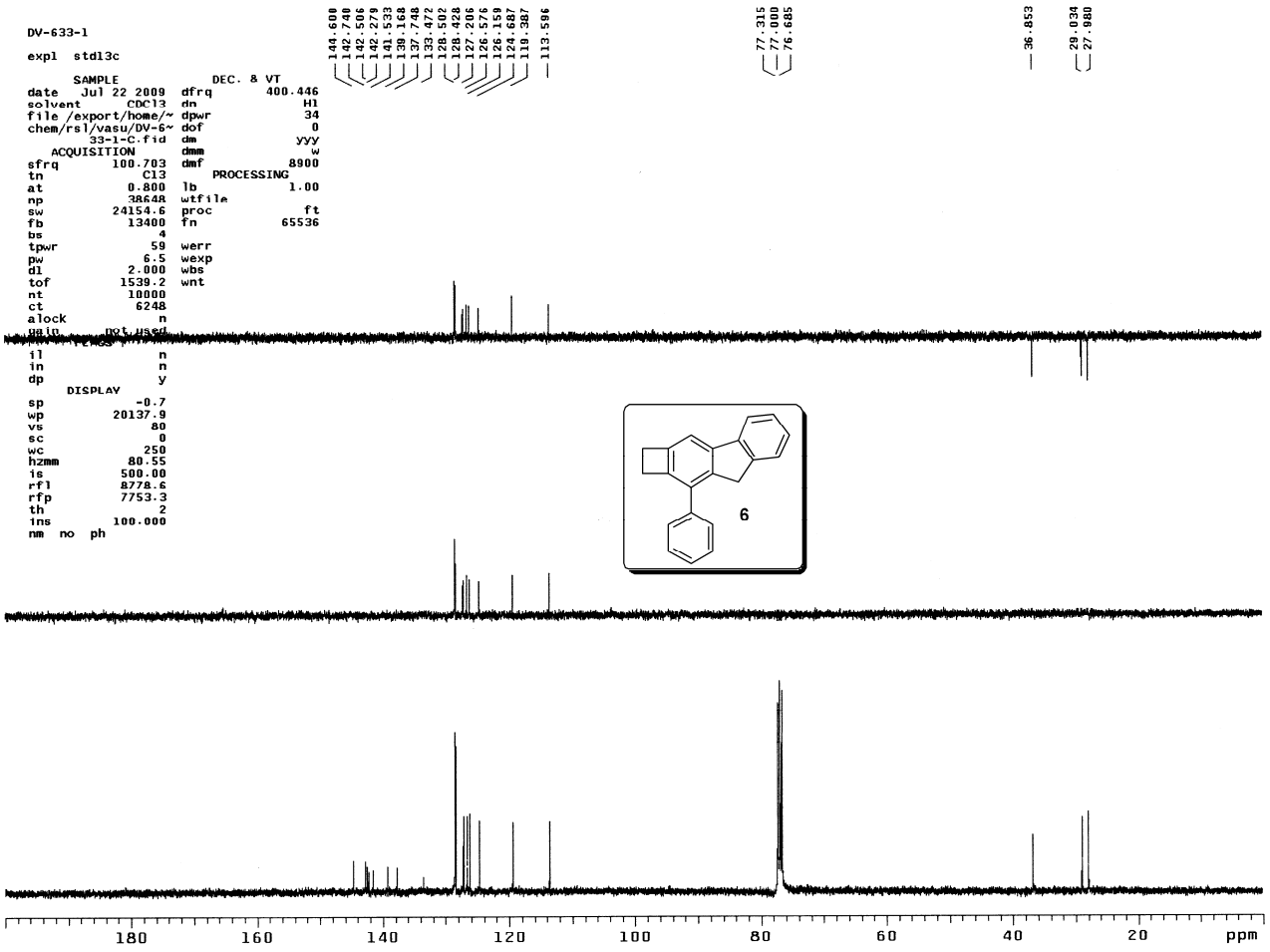
80
60
40
20
ppm

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142.506
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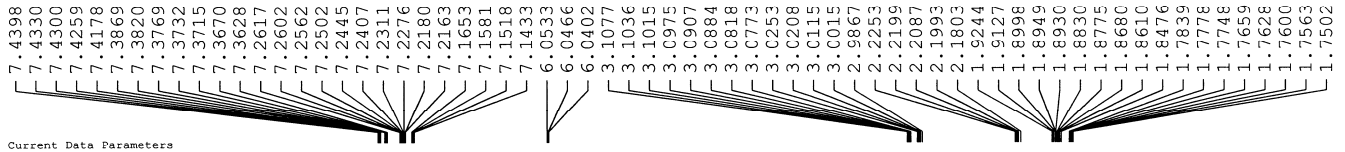
141.533
139.168
137.748
133.472
128.428
127.206
126.576
126.159
124.687
119.387
113.596
77.315
77.000
76.685
36.853
29.034
27.980
DV-633-1
expl stdl3c
SAMPLE
date Jul 22 2009
solvent CDCl3
file /export/home/~
chem/rsl/vasu/DV-6~
33-1-C.fid
ACQUISITION
sfrq 100.703
tn C13
at 0.800
np 38648
sw 24154.6
fb 13400
bs 4
tpwr 59
pw 6.5
d1 2.000
tof 1539.2
nt 10000
ct 6248
alock n
gain Not Used
FLAGS
il n
in n
dp y
DISPLAY
sp -0.7
wp 20137.9
vs 80
sc 0
wc 250
hzmm 80.55
is 500.00
rfl 8778.6
rfp 7753.3
th 2
ins 100.000
nm no ph
DEC. & VT
dfrq 400.446
dn H1
dpwr 34
dof 0
dm yyy
dmm w
dmf 8900
PROCESSING
lb 1.00
wtfile
proc ft
fn 65536
werr
wexp
wbs
wnt
6
180
160
140
120
100
80
60
40
20
ppm

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Current Data Parameters
NAME              1.9.2009
EXPNO                    7
PROCNO                   1

F2 - Acquisition Parameters
Date_              20090901
Time                 23.52
INSTRUM              spect
PROBHD   5 mm DUL 13C-1
PULPROG               zg30
TD                   32768
SOLVENT              CDCl3
NS                      24
DS                       0
SWH               6410.256 Hz
FIDRES           0.195625 Hz
AQ               2.5559540 sec
RG                     456
DW                  78.000 usec
DE                    6.00 usec
TE                   300.0 K
D1             2.00000000 sec
TD0                      1

======== CHANNEL f1 ========
NUC1                    1H
P1                   10.00 usec
PL1                  -2.40 dB
SFO1          400.1528010 MHz

F2 - Processing parameters
SI                   16384
SF            400.1500164 MHz
WDW                     EM
SSB                      0
LB                    0.00 Hz
GB                       0
PC                    1.00
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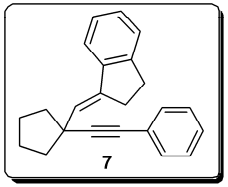

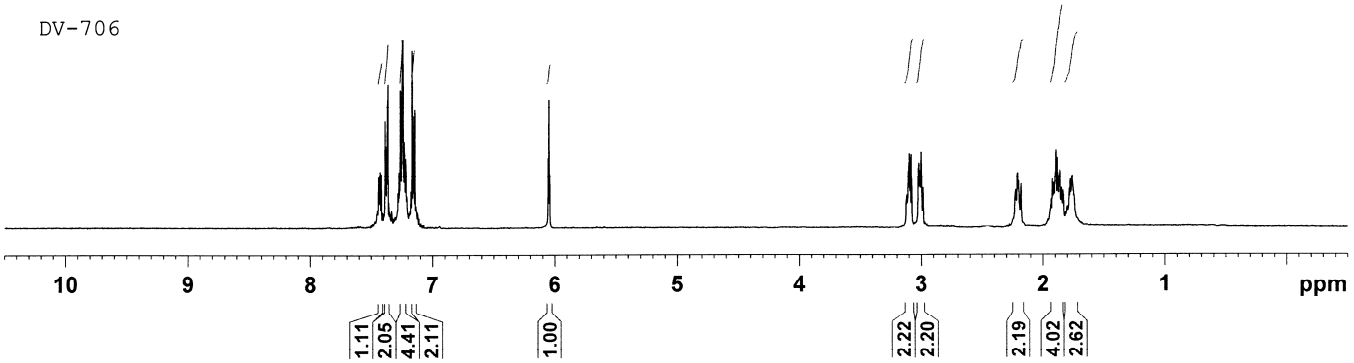

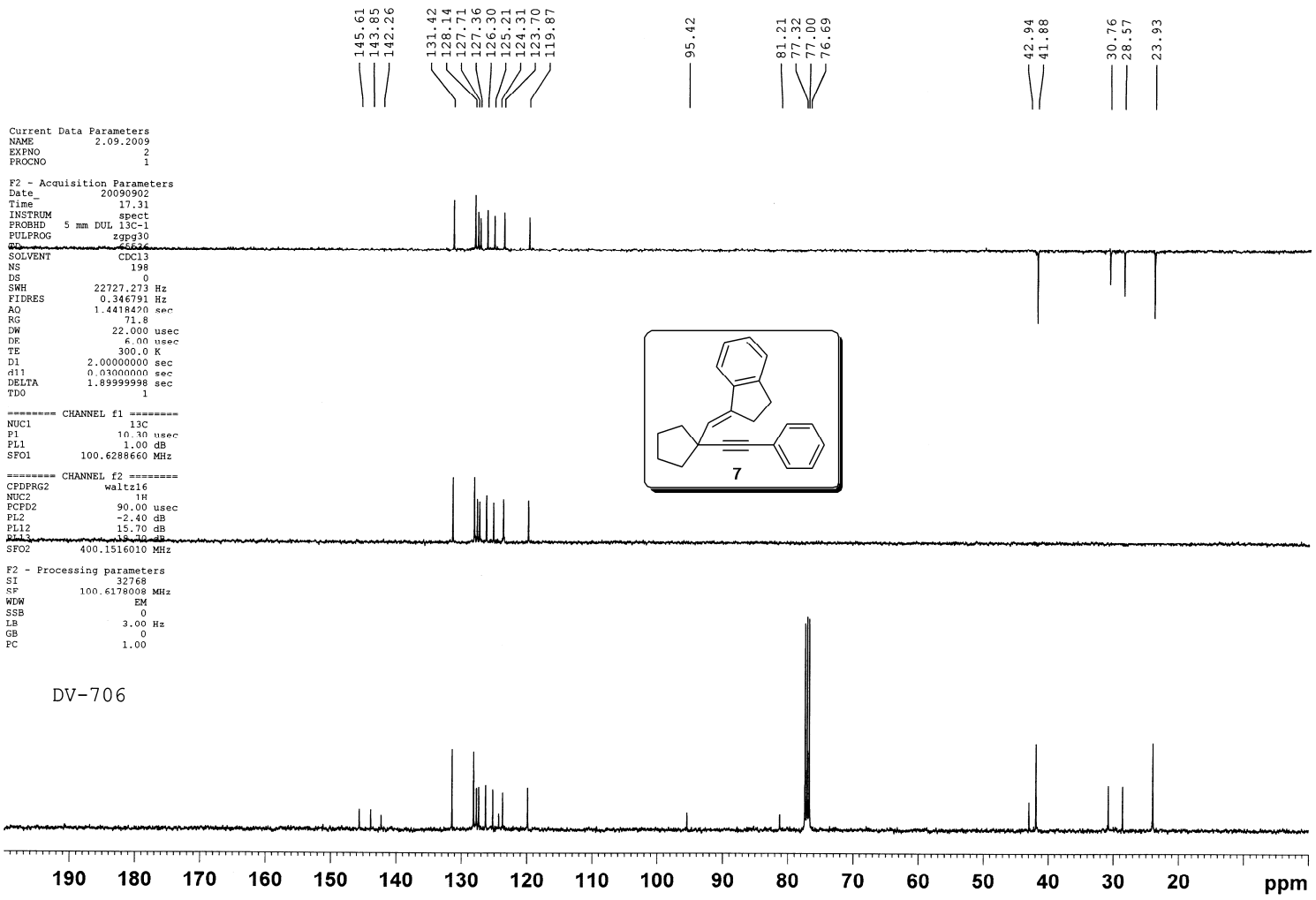
145.61
143.85
142.26
131.42
128.14
127.71
127.36
126.30
125.21
124.31
123.70
119.87
95.42
81.21
77.32
77.00
76.69
42.94
41.88
30.76
28.57
23.93
Current Data Parameters
NAME 2.09.2009
EXPNO 2
PROCNO 1
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Time 17.31
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PULPROG zgpg30
SOLVENT CDCl3
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FIDRES 0.346791 Hz
AQ 1.4418420 sec
RG 71.8
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
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NUC1 13C
P1 10.30 usec
PL1 1.00 dB
SFO1 100.6288660 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PL2 -2.40 dB
PL12 15.70 dB
SFO2 400.1516010 MHz
F2 - Processing parameters
SI 32768
SF 100.6178008 MHz
WDW EM
SSB 0
LB 3.00 Hz
GB 0
PC 1.00
7
DV-706
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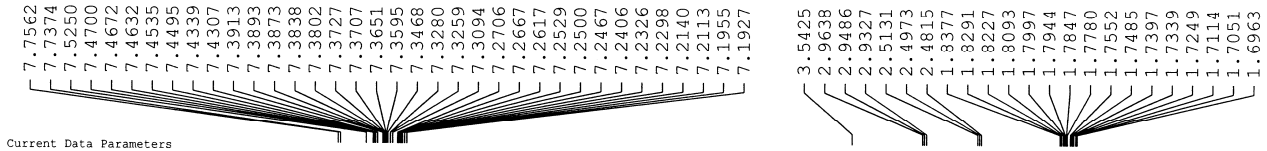


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PROCNO                       1

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TD                    32768
SOLVENT               CDCl3
NS                       63
DS                        0
SWH                6410.256 Hz
FIDRES            0.195625 Hz
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TE                   300.0 K
D1                2.00000000 sec
TD0                       1

======== CHANNEL f1 ========
NUC1                      1H
P1                    10.00 usec
PL1                   -2.40 dB
SFO1           400.1528010 MHz

F2 - Processing parameters
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SF             400.1500165 MHz
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LB                     0.00 Hz
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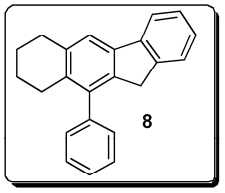
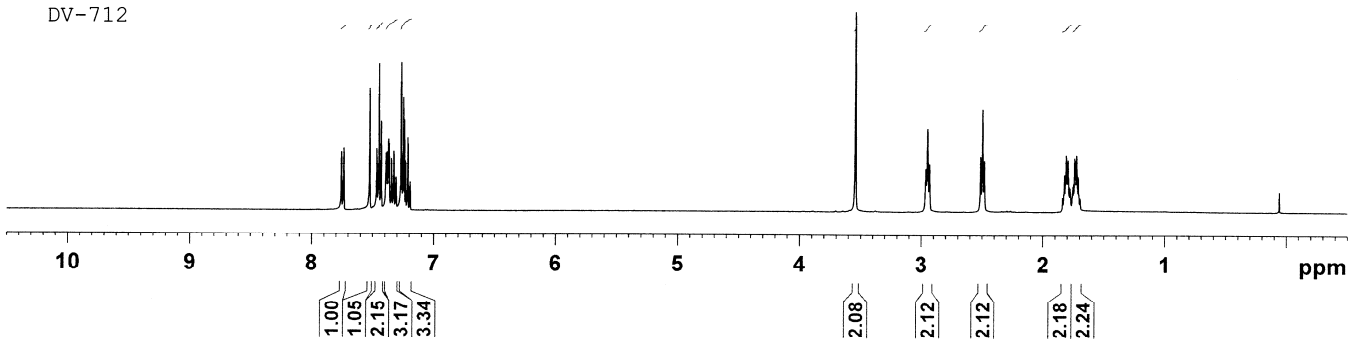
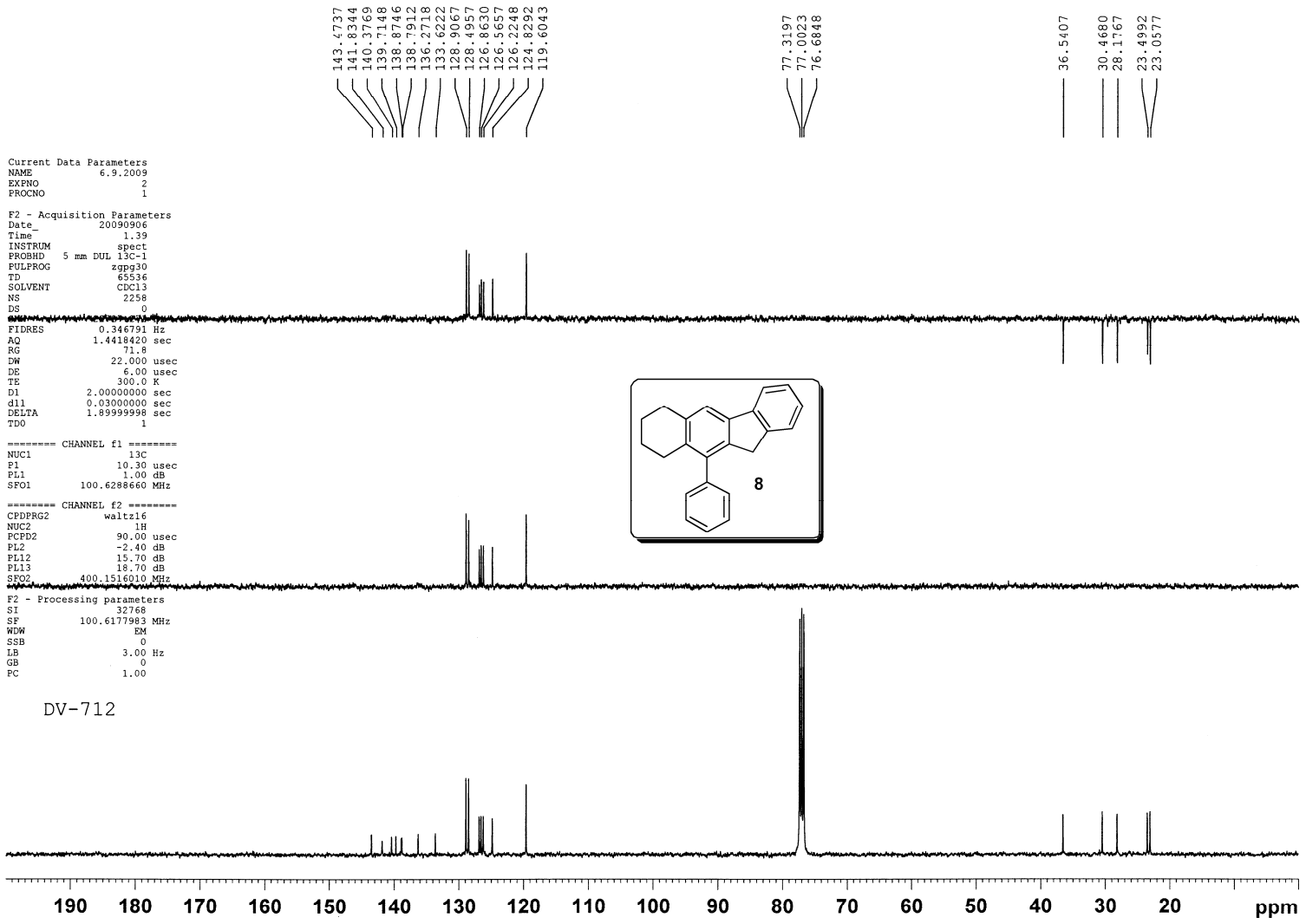
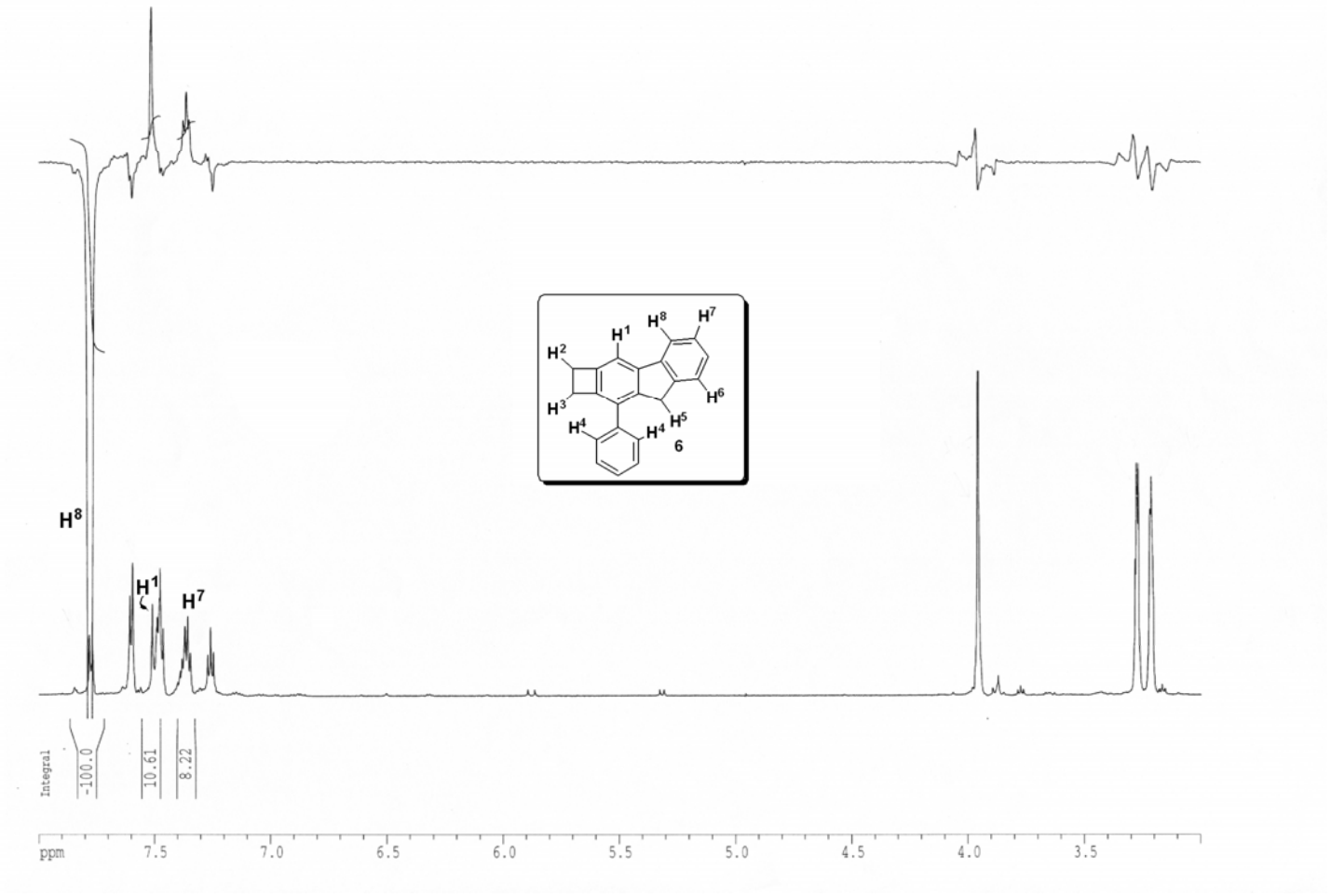
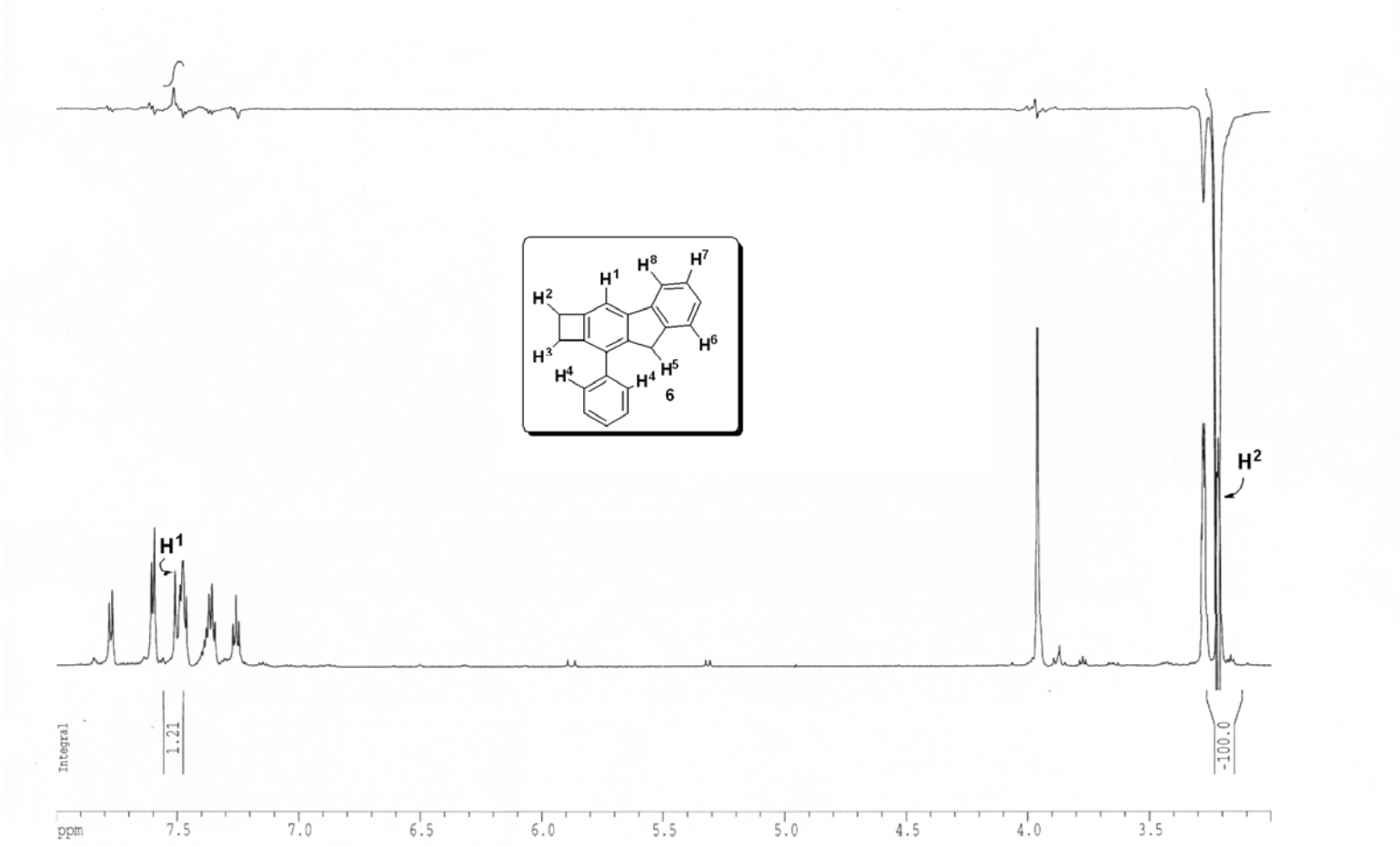


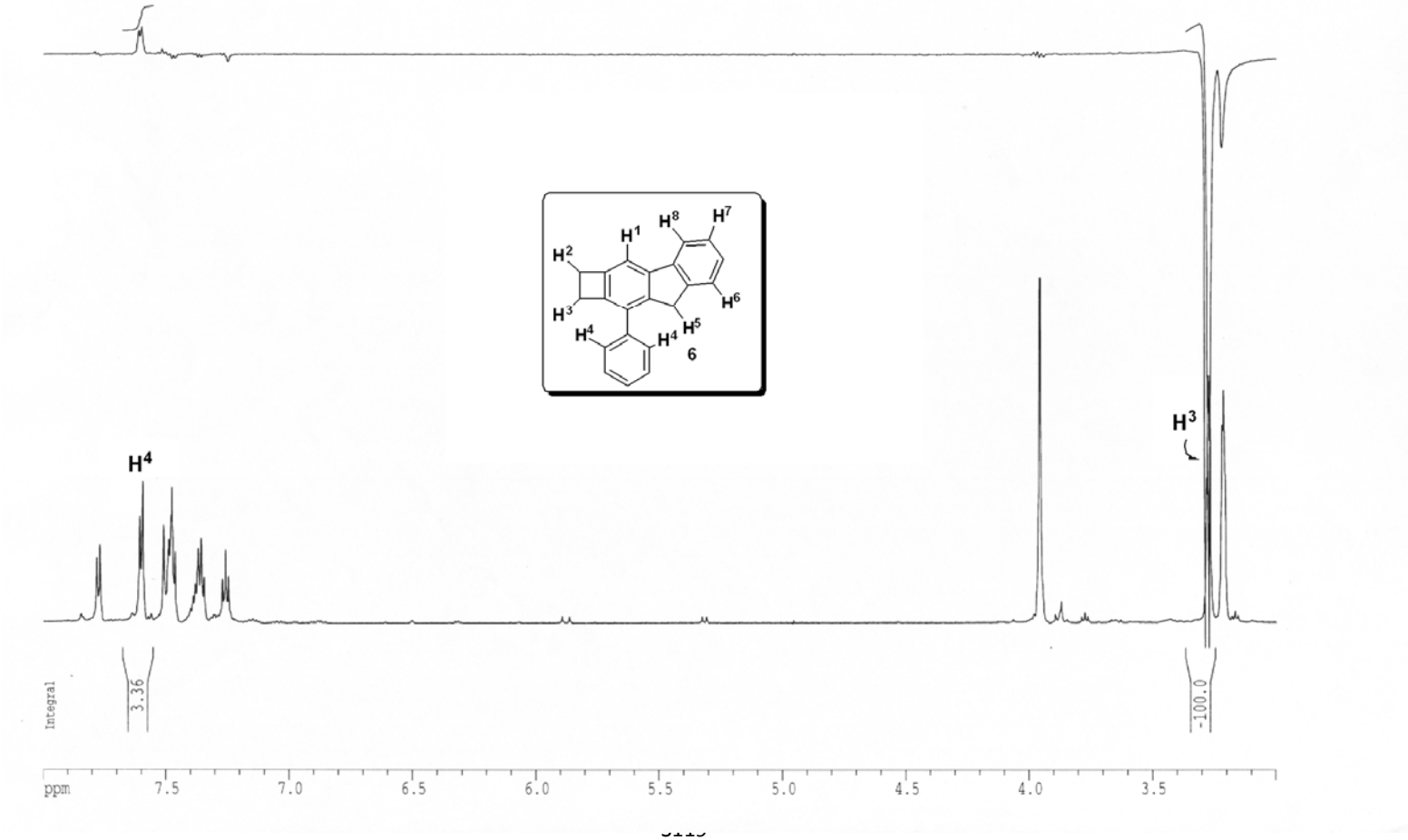


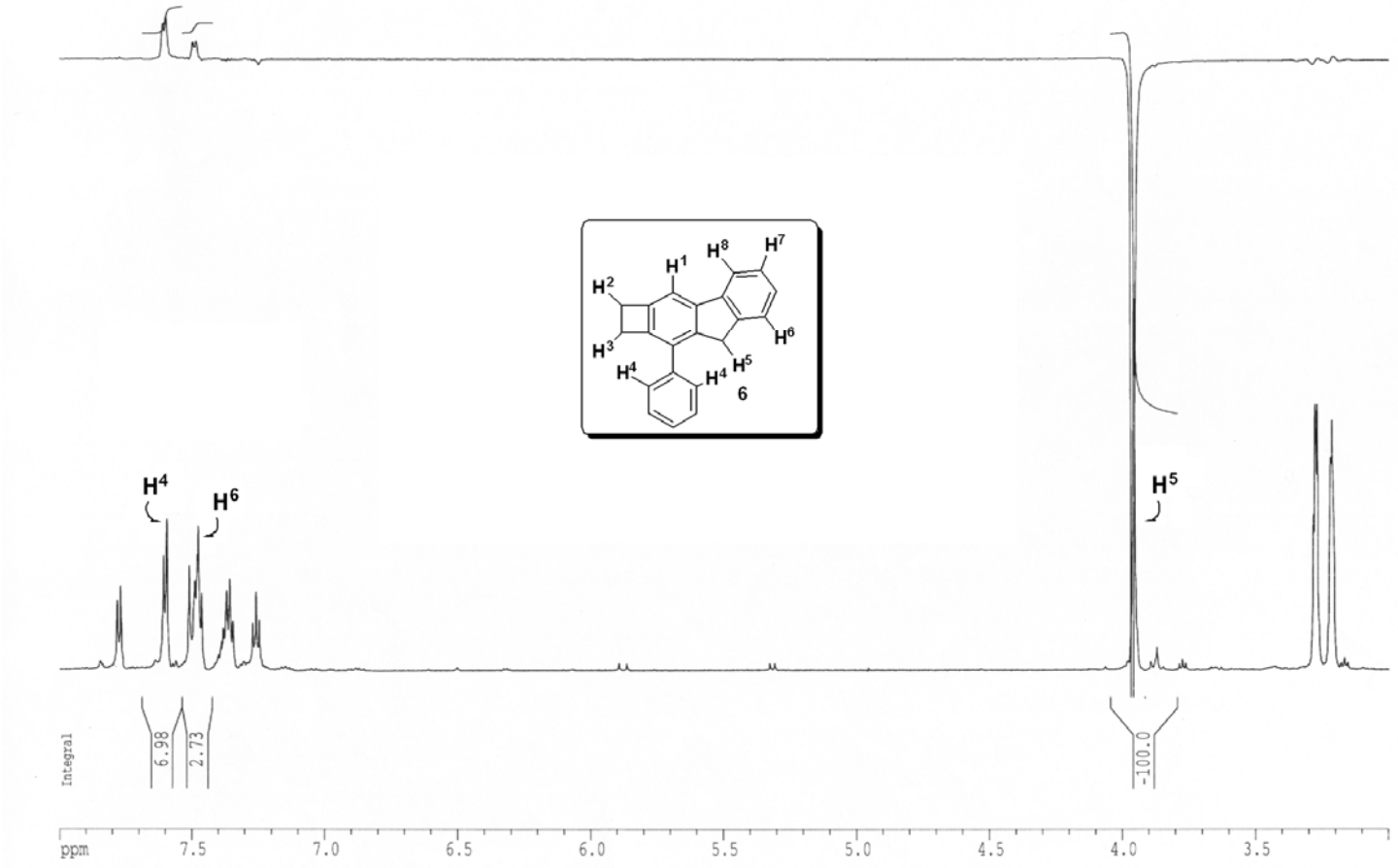


(V) NOE Spectra of compound (6).









(VI) X-ray structure and data for compound (6).

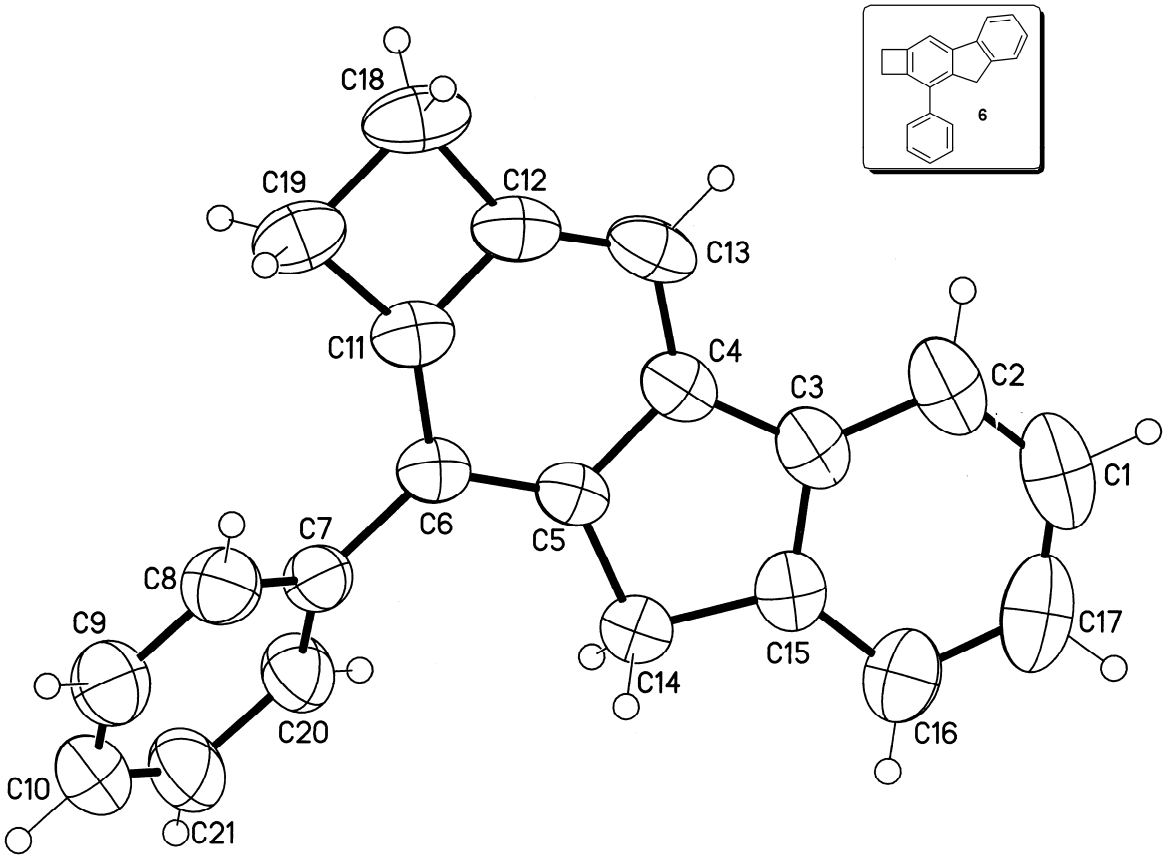


Table 1. Crystal data and structure refinement for 090809_0m.

Identification code	090809_0m	
Empirical formula	C21 H16	
Formula weight	268.34	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	*P*2(1)/*n*	
Unit cell dimensions	a = 7.2738(2) Å	α= 90°.
	b = 22.9796(6) Å	β= 101.426(2)°.
	c = 8.8198(2) Å	γ = 90°.
Volume	1445.00(6) $Å^3$	
Z	4	
Density (calculated)	1.233 Mg/m^3	
Absorption coefficient	0.070 mm^{-1}	

F(000)	568
Crystal size	0.18 x 0.15 x 0.15 mm^3
Theta range for data collection	1.77 to 26.42°.
Index ranges	$-9 \leq h \leq 8$, $-28 \leq k \leq 28$, $-11 \leq l \leq 11$
Reflections collected	12682
Independent reflections	2942 [R(int) = 0.0208]
Completeness to theta = 26.42°	99.3 %
Absorption correction	Empirical
Max. and min. transmission	0.7454 and 0.7131
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2942 / 0 / 190
Goodness-of-fit on F^2	1.094
Final R indices [I>2sigma(I)]	R1 = 0.0421, wR2 = 0.1202
R indices (all data)	R1 = 0.0607, wR2 = 0.1420
Largest diff. peak and hole	0.188 and -0.262 $e.Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for 090809_0m. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	9461(3)	546(1)	1660(3)	85(1)
C(2)	9309(3)	948(1)	491(2)	69(1)
C(3)	9388(2)	1534(1)	877(2)	51(1)
C(4)	9342(2)	2051(1)	-94(2)	45(1)
C(5)	9458(2)	2548(1)	865(2)	41(1)
C(6)	9566(2)	3114(1)	284(2)	42(1)
C(7)	9688(2)	3649(1)	1229(2)	44(1)
C(8)	10786(2)	4115(1)	929(2)	57(1)
C(9)	10870(3)	4624(1)	1767(2)	71(1)
C(10)	9880(3)	4682(1)	2923(2)	76(1)

C(11)	9563(2)	3133(1)	-1298(2)	47(1)
C(12)	9420(2)	2649(1)	-2238(2)	51(1)
C(13)	9301(2)	2094(1)	-1691(2)	54(1)
C(14)	9552(2)	2355(1)	2519(2)	44(1)
C(15)	9586(2)	1706(1)	2418(2)	48(1)
C(16)	9752(2)	1298(1)	3587(2)	63(1)
C(17)	9693(3)	712(1)	3182(3)	83(1)
C(18)	9402(2)	3002(1)	-3684(2)	65(1)
C(19)	9537(2)	3550(1)	-2635(2)	58(1)
C(20)	8691(2)	3719(1)	2405(2)	56(1)
C(21)	8796(3)	4230(1)	3246(2)	72(1)

Table 3. Bond lengths [Å] and angles [°] for 090809_0m.

C(1)-C(2)	1.372(3)
C(1)-C(17)	1.373(3)
C(1)-H(1)	0.9300
C(2)-C(3)	1.387(2)
C(2)-H(2)	0.9300
C(3)-C(15)	1.396(2)
C(3)-C(4)	1.461(2)
C(4)-C(13)	1.407(2)
C(4)-C(5)	1.4127(18)
C(5)-C(6)	1.4054(19)
C(5)-C(14)	1.5134(18)
C(6)-C(11)	1.3959(18)
C(6)-C(7)	1.4786(19)

C(7)-C(20)	1.388(2)
C(7)-C(8)	1.393(2)
C(8)-C(9)	1.378(2)
C(8)-H(8)	0.9300
C(9)-C(10)	1.367(3)
C(9)-H(9)	0.9300
C(10)-C(21)	1.368(3)
C(10)-H(10)	0.9300
C(11)-C(12)	1.379(2)
C(11)-C(19)	1.517(2)
C(12)-C(13)	1.371(2)
C(12)-C(18)	1.510(2)
C(13)-H(13)	0.9300
C(14)-C(15)	1.495(2)
C(14)-H(14A)	0.9700

C(14)-H(14B)	0.9700
C(15)-C(16)	1.380(2)
C(16)-C(17)	1.391(3)
C(16)-H(16)	0.9300
C(17)-H(17)	0.9300
C(18)-C(19)	1.553(2)
C(18)-H(18A)	0.9700
C(18)-H(18B)	0.9700
C(19)-H(19A)	0.9700
C(19)-H(19B)	0.9700
C(20)-C(21)	1.382(2)
C(20)-H(20)	0.9300
C(21)-H(21)	0.9300

C(2)-C(1)-C(17)	121.53(18)

C(2)-C(1)-H(1)	119.2
C(17)-C(1)-H(1)	119.2
C(1)-C(2)-C(3)	118.43(19)
C(1)-C(2)-H(2)	120.8
C(3)-C(2)-H(2)	120.8
C(2)-C(3)-C(15)	120.29(16)
C(2)-C(3)-C(4)	130.62(16)
C(15)-C(3)-C(4)	109.08(13)
C(13)-C(4)-C(5)	121.93(14)
C(13)-C(4)-C(3)	129.62(13)
C(5)-C(4)-C(3)	108.36(13)
C(6)-C(5)-C(4)	122.03(13)
C(6)-C(5)-C(14)	128.84(12)
C(4)-C(5)-C(14)	109.05(12)
C(11)-C(6)-C(5)	113.86(12)

C(11)-C(6)-C(7)	121.70(13)
C(5)-C(6)-C(7)	124.44(12)
C(20)-C(7)-C(8)	117.14(14)
C(20)-C(7)-C(6)	122.45(13)
C(8)-C(7)-C(6)	120.38(13)
C(9)-C(8)-C(7)	121.14(16)
C(9)-C(8)-H(8)	119.4
C(7)-C(8)-H(8)	119.4
C(10)-C(9)-C(8)	120.66(17)
C(10)-C(9)-H(9)	119.7
C(8)-C(9)-H(9)	119.7
C(9)-C(10)-C(21)	119.38(16)
C(9)-C(10)-H(10)	120.3
C(21)-C(10)-H(10)	120.3
C(12)-C(11)-C(6)	124.11(14)

C(12)-C(11)-C(19)	93.15(12)
C(6)-C(11)-C(19)	142.57(14)
C(13)-C(12)-C(11)	122.68(13)
C(13)-C(12)-C(18)	143.82(14)
C(11)-C(12)-C(18)	93.46(13)
C(12)-C(13)-C(4)	115.36(13)
C(12)-C(13)-H(13)	122.3
C(4)-C(13)-H(13)	122.3
C(15)-C(14)-C(5)	103.47(11)
C(15)-C(14)-H(14A)	111.1
C(5)-C(14)-H(14A)	111.1
C(15)-C(14)-H(14B)	111.1
C(5)-C(14)-H(14B)	111.1
H(14A)-C(14)-H(14B)	109.0
C(16)-C(15)-C(3)	120.85(15)

C(16)-C(15)-C(14)	129.26(14)
C(3)-C(15)-C(14)	109.88(13)
C(15)-C(16)-C(17)	118.12(18)
C(15)-C(16)-H(16)	120.9
C(17)-C(16)-H(16)	120.9
C(1)-C(17)-C(16)	120.77(19)
C(1)-C(17)-H(17)	119.6
C(16)-C(17)-H(17)	119.6
C(12)-C(18)-C(19)	86.82(11)
C(12)-C(18)-H(18A)	114.2
C(19)-C(18)-H(18A)	114.2
C(12)-C(18)-H(18B)	114.2
C(19)-C(18)-H(18B)	114.2
H(18A)-C(18)-H(18B)	111.3
C(11)-C(19)-C(18)	86.56(12)

C(11)-C(19)-H(19A)	114.2
C(18)-C(19)-H(19A)	114.2
C(11)-C(19)-H(19B)	114.2
C(18)-C(19)-H(19B)	114.2
H(19A)-C(19)-H(19B)	111.4
C(21)-C(20)-C(7)	121.24(16)
C(21)-C(20)-H(20)	119.4
C(7)-C(20)-H(20)	119.4
C(10)-C(21)-C(20)	120.45(17)
C(10)-C(21)-H(21)	119.8
C(20)-C(21)-H(21)	119.8

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for 090809_0m. The anisotropic displacement factor exponent takes the form: $-2\ ^2[\ h^2\ a^{*2} U^{11} + ... + 2\ h\ k\ a^*\ b^*\ U^{12}\ ]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	80(1)	46(1)	133(2)	-8(1)	32(1)	-1(1)
C(2)	62(1)	50(1)	97(1)	-17(1)	18(1)	-1(1)
C(3)	36(1)	48(1)	69(1)	-11(1)	10(1)	-1(1)
C(4)	33(1)	52(1)	50(1)	-12(1)	7(1)	0(1)
C(5)	34(1)	49(1)	40(1)	-7(1)	8(1)	0(1)
C(6)	36(1)	51(1)	39(1)	-2(1)	10(1)	1(1)
C(7)	45(1)	46(1)	41(1)	1(1)	7(1)	2(1)
C(8)	60(1)	58(1)	55(1)	1(1)	14(1)	-6(1)
C(9)	85(1)	51(1)	72(1)	2(1)	5(1)	-16(1)
C(10)	114(2)	49(1)	63(1)	-10(1)	11(1)	3(1)

C(11)	38(1)	65(1)	40(1)	-1(1)	10(1)	0(1)
C(12)	37(1)	75(1)	41(1)	-9(1)	10(1)	1(1)
C(13)	39(1)	71(1)	50(1)	-25(1)	8(1)	2(1)
C(14)	41(1)	47(1)	44(1)	-3(1)	10(1)	-3(1)
C(15)	37(1)	47(1)	61(1)	0(1)	11(1)	-1(1)
C(16)	56(1)	59(1)	77(1)	12(1)	18(1)	3(1)
C(17)	77(1)	56(1)	121(2)	27(1)	32(1)	7(1)
C(18)	44(1)	111(2)	40(1)	-2(1)	10(1)	-1(1)
C(19)	48(1)	84(1)	44(1)	9(1)	12(1)	-2(1)
C(20)	70(1)	48(1)	55(1)	-2(1)	24(1)	2(1)
C(21)	107(2)	54(1)	62(1)	-6(1)	32(1)	11(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters (Å^{2}x 10^3) for 090809_0m.

	x	y	z	U(eq)
H(1)	9406	152	1416	102
H(2)	9156	830	-535	83
H(8)	11474	4082	149	69
H(9)	11608	4930	1543	85
H(10)	9942	5027	3485	91
H(13)	9200	1769	-2331	64
H(14A)	10677	2501	3191	52
H(14B)	8464	2488	2902	52
H(16)	9900	1412	4616	76

H(17)	9812	430	3951	99
H(18A)	8243	2974	-4445	78
H(18B)	10486	2941	-4152	78
H(19A)	10684	3771	-2574	70
H(19B)	8441	3800	-2851	70
H(20)	7938	3417	2632	68
H(21)	8126	4266	4036	86