Synthesis of 1,2,3,4-tetrasubstituted naphthalenes through cascade reaction triggered by silyl acetal activation

Hikaru Yanai,* Nobuyuki Ishii, and Takashi Matsumoto*

School of Pharmacy, Tokyo University of Pharmacy and Life Sciences

1432-1 Horinouchi, Hachioji, Tokyo 192-0392, Japan.

Tel.: +81 426 76 3265, Fax: +81 426 76 3257

E-mail: yanai@toyaku.ac.jp (HY) and tmatsumo@toyaku.ac.jp (TM)

Electronic Supplementary Information

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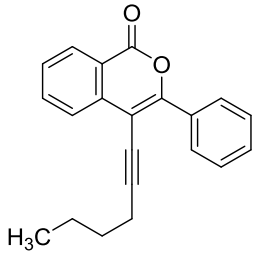
1. General and materials

All reactions were carried out under Ar atmosphere. Melting points were uncorrected. NMR spectra were recorded on a Bruker Avance III Nanobay 400 MHz spectrometer (400 MHz for ^{1}H, 100 MHz for ^{13}C) in $CDCl_3$ or CD_3CN. Data are reported as follows: chemical shifts, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sex = sextet, m = multiplet, br = broad) and coupling constants. Chemical shifts (in ppm) were referenced to the solvent signal ($CDCl_3$, 7.26 ppm for ^{1}H NMR and 77.0 ppm for ^{13}C NMR; CD_3CN, 1.93 ppm for ^{1}H NMR and 117.7 ppm for ^{13}C NMR). Coupling constants (*J*) are given in Hz. Mass spectra were measured on a MICROMASS LCT mass spectrometer using electrospray ionization-time of flight (ESI-TOF). Column chromatography was performed on neutral silica gel (Kanto Chemical, Silica gel 60N, 63-210 μm or 40-100 μm) or basic alumina (ICN Alumina B–Super I). Zwitterion catalyst **1** was prepared by our reported procedure from Tf_2CH_2.[1] Tf_2CH_2 was supplied from Central Glass Co. and this compound can be also prepared by Waller's procedure in the laboratory.[2]

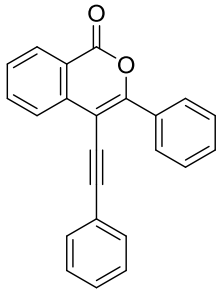
2. Preparation of 1*H*-isochromen-1-ones

4-(Hex-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-one (7a)



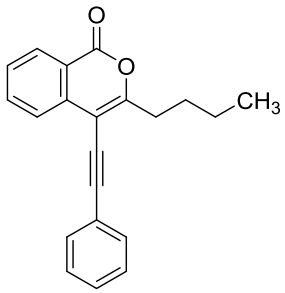
This compound was obtained by simple modification of the Larock's conditions.[3] To a solution of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (1.04 g, 2.99 mmol) and hex-1-yne (0.74 g, 9.0 mmol) in DMF (3.0 mL) and Et_3N (1.2 mL), $(Ph_3P)_2PdCl_2$ (42 mg, 60 μmol) and CuI (38 mg, 200 μmol) were added at room temperature. After being stirred for 3 h at 55 °C, the reaction mixture was diluted with H_2O (100 mL), then it was extracted with Et_2O (50 mL x 3), dried over anhydrous $MgSO_4$, and evaporated. The resulting residue was purified by column chromatography on silica gel (hexane/EtOAc = 20 : 1) to give **7a** in 88% yield (801 mg, 2.65 mmol). Colorless crystals (from hexane/EtOAc); Mp. 32.0-32.5 °C; IR (ATR) ν 2952, 2223, 1722, 1603, 1480, 1076, 758, 690, 552 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.96 (3H, t, J = 7.4 Hz), 1.44-1.54 (2H, m), 1.59-1.69 (2H, m), 2.52 (2H, t, J = 7.1 Hz), 7.43-7.49 (3H, m), 7.52-7.58 (1H, m), 7.78-7.84 (1H, m), 7.92 (1H, d, J = 8.0 Hz), 8.15-8.21 (2H, m), 8.32 (1H, dd, J = 7.8, 0.6 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.6, 19.5, 22.1, 30.5, 73.5, 99.2, 100.2, 119.8, 125.5, 128.0, 128.4, 128.5, 129.3, 130.0, 132.5, 134.9, 137.6, 156.0, 161.2.; MS (ESI-TOF) *m*/*z* 303 $[M+H]^+$; HRMS calcd for $C_{21}H_{19}O_2$ $[M+H]^+$, 303.1385; found, 303.1383. Anal. Calcd for $C_{21}H_{18}O_2$: C, 83.42; H, 6.00. Found: C, 83.44; H, 6.05.

3-Phenyl-4-(phenylethynyl)-1*H*-isochromen-1-one (7b)



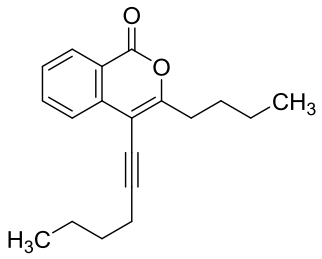
According to the synthetic procedure for **7a**, this compound was obtained in 95% yield (1.84 g, 5.71 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (2.09 g, 6.01 mmol) with ethynylbenzene (0.80 mL, 7.2 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (84 mg, 0.12 mmol) and CuI (69 mg, 0.36 mmol) in a mixed solvent of DMF (6.0 mL) and Et_3N (2.4 mL) for 5 h at 55 °C and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1). Its structure was confirmed by comparison with the reported ^1H and ^{13}C NMR spectra.[3] ^1H NMR (400 MHz, $CDCl_3$) δ 7.35-7.42 (3H, m), 7.45-7.56 (5H, m), 7.60 (1H, t, J = 7.8 Hz), 7.87 (1H, t, J = 7.8 Hz), 8.12 (1H, d, J = 7.8 Hz), 8.20-8.28 (2H, m), 8.37 (1H, d, J = 7.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 82.6, 97.5, 99.6, 119.6, 122.7, 125.3, 128.1, 128.5, 128.58, 128.63, 128.7, 129.4, 130.4, 131.3, 132.3, 135.1, 136.9, 156.6, 160.9.

3-Butyl-4-(phenylethynyl)-1*H*-isochromen-1-one (7c)



To a solution of methyl 2-(hex-1-yn-1-yl)benzoate[4] (435 mg, 2.01 mmol) in CH_2Cl_2 (10 mL), a solution of I_2 (535 mg, 2.21 mmol) in CH_2Cl_2 (10 mL) was added. After being stirred for 1 h at room temperature, the reaction mixture was quenched with saturated $Na_2S_2O_3$ aqueous solution (25 mL) and extracted with Et_2O (25 mL x 3). The combined organic layer was washed with brine (25 mL), dried over anhydrous Na_2SO_4, and evaporated. The resulting residue contained 6-*endo*- and 5-*exo*-iodolactones in a ratio of 93:7. Due to low stability of these lactones, this mixture was used in the following Sonogashira reaction without further purification. This mixture mainly containing 6-*endo*-iodolactone was dissolved in a mixed solvent of DMF (2.0 mL) and Et_3N (0.8 mL). To this solution, ethynylbenzene (0.27 mL, 2.4 mmol), $(Ph_3P)_2PdCl_2$ (29 mg, 40 μmol), and CuI (21 mg, 110 μmol) were added. After being stirred for 5 h at 55 °C, the reaction mixture was filtrated through celite pad. The filtrate was evaporated, diluted with water (25 mL), and extracted with Et_2O (25 mL x 3). The combined organic layer was washed with brine, dried over anhydrous Na_2SO_4, and concentrated under reduced pressure. This compound was isolated in 71% yield (432 mg, 1.43 mmol) over two steps by column chromatography on silica gel (hexane/EtOAc = 10 : 1) followed by recycling HPLC (hexane/EtOAc = 10 : 1). Colorless crystals (from hexane/EtOAc); Mp. 85.5-88.0 °C; IR (ATR) ν 2924, 1728, 1620, 1480, 1016, 750, 684 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.98 (3H, t, J = 7.5 Hz), 1.46 (2H, sex, J = 7.5 Hz), 1.769-1.86 (2H, m), 2.91 (2H, t, J = 7.6 Hz), 7.37-7.43 (3H, m), 7.53 (1H, brt, J = 7.9 Hz), 7.56-7.60 (2H, m), 7.77-7.83 (1H, m), 7.92 (1H, dd, J = 8.0, 0.5 Hz), 8.29 (1H, dd, J = 7.9, 0.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 22.2, 29.3, 32.5, 81.3, 96.8, 100.2, 119.4, 122.8, 124.6, 128.1, 128.5, 128.6, 129.4, 131.4, 135.1, 136.5, 161.5, 162.8; MS (ESI-TOF) *m*/*z* 352 $[M+Na]^+$; HRMS calcd for $C_{21}H_{18}NaO_2$ $[M+Na]^+$, 325.1204; found, 325.1207.

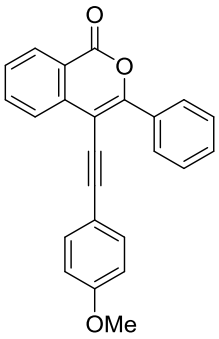
3-Butyl-4-(hex-1-yn-1-yl)-1*H*-isochromen-1-one (7d)



According to the synthetic procedure for **7c**, this compound was obtained in 80% yield (318 mg, 1.13 mmol) as follows. A crude mixture of 3-butyl-4-iodo-1*H*-isochromen-1-one, which was obtained by the iodolactonization of methyl 2-(hex-1-yn-1-yl)benzoate[4] (305 mg, 1.41 mmol) with I_2 (393 mg, 1.55 mmol) in CH_2Cl_2 (15 mL), was dissolved in a mixed solvent of DMF (2.0 mL) and Et_3N (0.8 mL). This solution was treated with hex-1-yne (0.48 mL, 4.2 mmol), $(Ph_3P)_2PdCl_2$ (29 mg, 40 μmol), and CuI (21 mg, 110 μmol) for 5 h at 55 °C.

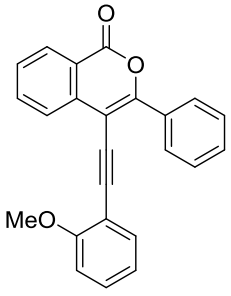
After usual extractive workup, isolation of this compound was achieved by column chromatography on silica gel (hexane/EtOAc = 20 : 1) followed by recycling HPLC (hexane/EtOAc = 10 : 1). Pale yellow oil; IR (neat) ν 2950, 2925, 2870, 2225, 1740, 1620, 1480, 1316, 1100, 1020, 768, 696 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.95 (3H, t, J = 7.4 Hz), 0.98 (3H, t, J = 7.3 Hz), 1.42 (2H, sex, J = 7.4 Hz), 1.47-1.58 (2H, m), 1.59-1.69 (2H, m), 1.69-1.78 (2H, m), 2.52 (2H, t, J = 7.0 Hz), 2.81 (2H, t, J = 7.4 Hz), 7.48 (1H, td, J = 8.0, 1.3 Hz), 7.75 (1H, td, J = 8.0, 1.3 Hz), 7.81 (1H, dd, J = 8.0, 0.7 Hz), 8.24 (1H, dd, J = 8.0, 0.7 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.6, 13.8, 19.3, 22.0, 22.2, 29.3, 30.9, 32.3, 72.3, 98.0, 100.6, 119.4, 124.6, 127.8, 129.3, 134.9, 137.1, 161.8, 162.0; MS (ESI-TOF) m/z 305 $[M+Na]^+$; HRMS calcd for $C_{19}H_{22}NaO_2$ $[M+Na]^+$, 305.1517; found, 305.1517.

4-((4-Methoxyphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-one (7e)



According to the synthetic procedure for **7a**, this compound was obtained in 93% yield (658 mg, 1.87 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (696 mg, 2.00 mmol) with 1-ethynyl-4-methoxybenzene (295 mg, 2.20 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (28 mg, 40 μmol) and CuI (21 mg, 0.11 mmol) in a mixed solvent of DMF (2.0 mL) and Et_3N (0.80 mL) for 4 h at 55 °C and the following flash column chromatography on silica gel (hexane/EtOAc = 5 : 1). Colorless crystals (from hexane/EtOAc); Mp. 115-117 °C; IR (ATR) ν 3033, 2958, 2839, 1731, 1602, 1250, 1223, 1016, 821, 753, 689 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.89 (3H, s), 6.91 (2H, d, J = 8.9 Hz), 7.44-7.54 (5H, m), 7.59 (1H, td, J = 7.6, 1.1 Hz), 7.86 (1H, td, J = 7.6, 1.4 Hz), 8.11 (1H, brd, J = 7.6 Hz), 8.22-8.26 (2H, m), 8.35 (1H, dd, J = 7.6, 1.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 81.3, 97.6, 100.0, 114.2, 114.9, 119.7, 125.5, 128.1, 128.6, 128.7, 129.5, 130.3, 132.4, 132.9, 135.1, 137.2, 156.2, 160.0, 161.1 MS (ESI-TOF) m/z 353 $[M+H]^+$; HRMS calcd for $C_{24}H_{17}O_3$ $[M+H]^+$, 353.1176; found, 353.1178.

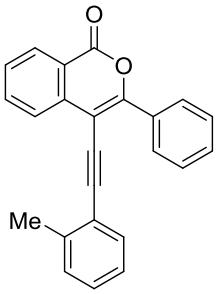
4-((2-Methoxyphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-one (7f)



According to the synthetic procedure for **7a**, this compound was obtained in 79% yield (553 mg, 1.57 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (696 mg, 2.00 mmol) with

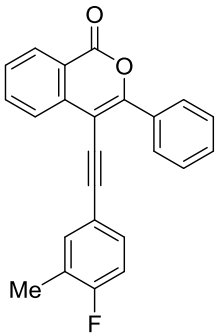
1-ethynyl-2-methoxybenzene (322 mg, 2.40 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (28 mg, 40 μmol) and CuI (20 mg, 105 μmol) in a mixed solvent of DMF (2.0 mL) and Et_3N (0.80 mL) for 4 h at 55 °C and the following flash column chromatography on silica gel (hexane/EtOAc = 5 : 1). Colorless crystals (from hexane/EtOAc); Mp. 144-145 °C; IR (ATR) ν 2971, 2936, 2832, 1736, 1602, 1244, 1015, 743, 692 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.97 (3H, s), 6.92-7.02 (2H, m), 7.31-7.39 (1H, m), 7.43-7.55 (4H, m), 7.57-7.63 (1H, m), 7.83-7.91 (1H, m), 8.38 (1H, d, J = 7.6 Hz), 8.32-8.40 (3H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.8, 86.6, 94.8, 100.1, 110.7, 112.2, 119.7, 120.6, 125.8, 128.1, 128.6, 128.7, 129.4, 130.2, 130.3, 132.3, 132.9, 135.1, 137.4, 156.0, 160.3, 161.1; MS (ESI-TOF) m/z 353 $[M+H]^+$; HRMS calcd for $C_{24}H_{17}O_3$ $[M+H]^+$, 353.1178; found, 353.1176. Anal. Calcd for $C_{24}H_{18}O_3$: C, 81.80; H, 4.58. Found: C, 81.60; H, 4.55.

3-Phenyl-4-(*o*-tolylethynyl)-1*H*-isochromen-1-one (7g)



According to the synthetic procedure for **7a**, this compound was obtained in 95% yield (632 mg, 1.88 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (692 mg, 1.99 mmol) with 2-ethynyltoluene (255 mg, 2.20 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (29 mg, 40 μmol) and CuI (21 mg, 110 μmol) in a mixed solvent of DMF (2.0 mL) and Et_3N (0.8 mL) for 2 h at 55 °C and the following column chromatography on silica gel (hexane/EtOAc = 10 : 1). Yellow crystals (from Et_2O); Mp. 157-159 °C; IR (ATR) ν 1736, 1479, 1080, 1054, 1015, 751, 689 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.50 (3H, s), 7.18-7.32 (3H, m), 7.47-7.54 (4H, m), 7.58-7.64 (1H, m), 7.84-7.90 (1H, m), 8.15 (1H, d, J = 8.0 Hz), 8.20-8.26 (2H, m), 8.37 (1H, d, J = 7.9 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.0, 86.2, 96.7, 100.1, 119.8, 122.6, 125.4, 125.8, 128.2, 128.71, 128.76, 128.82, 129.6, 129.7, 130.4, 132.0, 132.4, 135.2, 137.2, 140.0, 156.6, 161.0; MS (ESI-TOF) m/z 337 $[M+H]^+$; HRMS calcd for $C_{24}H_{17}O_2$ $[M+H]^+$, 337.1229; found, 337.1241.

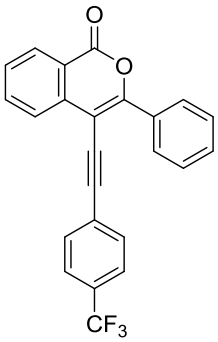
4-((4-Fluoro-3-methylphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-one (7h)



According to the synthetic procedure for **7a**, this compound was obtained in 87% yield (610 mg, 1.72 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (692 mg, 1.99 mmol) with 4-ethynyl-1-fluoro-2-methylbenzene (293 μL, 2.20 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (29 mg, 40 μmol)

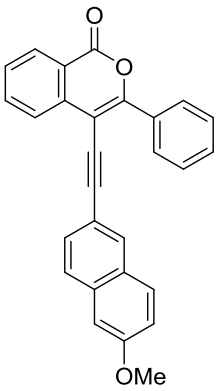
and CuI (23 mg, 120 μmol) in a mixed solvent of DMF (2.0 mL) and Et_3N (0.8 mL) for 3 h at 55 °C and the following column chromatography on silica gel (hexane/EtOAc = 10 : 1). Pale yellow crystals (from EtOAc); Mp. 137-139 °C; IR (ATR) ν 1730, 1481, 1213, 1097, 808, 764, 689 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.30 (3H, d, J_{HF} = 1.9 Hz), 7.02 (1H, t, J_{HF} = 8.5 Hz, J_{HH} = 8.5 Hz), 7.32 (1H, ddd, J_{HF} = 4.9 Hz, J_{HH} = 8.5, 1.5 Hz), 7.36 (1H, dd, J_{HF} = 7.2 Hz, J_{HH} = 1.5 Hz), 7.46-7.54 (3H, m), 7.57-7.63 (1H, m), 7.81-7.92 (1H, m), 8.09 (1H, d, J = 8.0 Hz), 8.19-8.24 (2H, m), 8.36 (1H, dd, J = 7.9, 1.0 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.4 (d, J_{CF} = 3.0 Hz), 82.0, 96.7, 99.7, 115.5 (d, J_{CF} = 23.1 Hz), 118.5 (d, J_{CF} = 3.0 Hz), 119.7, 125.4, 125.5 (d, J_{CF} = 18.1 Hz), 128.2, 128.65 (3C), 128.73, 129.5, 130.4, 130.6 (d, J_{CF} = 9.1 Hz), 132.4, 134.5 (d, $J_{C\text{-}F}$ = 5.0 Hz), 135.2, 137.0, 156.7, 160.7 (d, J_{CF} = 75.5 Hz); MS (ESI-TOF) *m*/*z* 355 $[M+H]^+$; HRMS calcd for $C_{24}H_{16}FO_2$ $[M+H]^+$, 355.1134; found, 355.1139.

3-Phenyl-4-((4-(trifluoromethyl)phenyl)ethynyl)-1*H*-isochromen-1-one (7i)



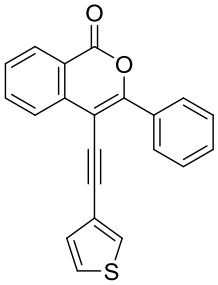
According to the synthetic procedure for **7a**, this compound was obtained in 83% yield (641 mg, 1.66 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (692 mg, 1.99 mmol) with 1-ethynyl-4-(trifluoromethyl)benzene (314 μL, 2.20 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (29 mg, 40 μmol) and CuI (23 mg, 120 μmol) in a mixed solvent of DMF (2.0 mL) and Et_3N (0.8 mL) for 3 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1). Pale yellow crystals (from EtOAc); Mp. 149-152 °C; IR (ATR) ν 1727, 1315, 1065, 1029, 839, 760 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.50-7.55 (3H, m), 7.59-7.68 (5H, m), 7.88 (1H, td, J = 8.0, 1.4 Hz), 8.08 (1H, d, J = 8.0 Hz), 8.17-8.22 (2H, m), 8.37 (1H, dd, J = 8.0, 0.9 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 82.3, 95.8, 99.2, 119.7, 122.5, 125.2, 125.5 (q, J_{CF} = 3.0 Hz), 127.8 (q, J_{CF} = 258 Hz), 128.2, 128.7, 128.9, 129.7, 130.4 (q, J_{CF} = 33.2 Hz), 130.7, 131.5, 132.2, 135.3, 136.7, 157.0, 160.8; ^{19}F NMR (376 MHz, $CDCl_3$) δ –0.3 (3F, s); MS (ESI-TOF) *m*/*z* 391 $[M+H]^+$; HRMS calcd for $C_{24}H_{14}F_3O_2$ $[M+H]^+$, 391.0946; found, 391.0950.

4-((6-Methoxynaphthalen-2-yl)ethynyl)-3-phenyl-1*H*-isochromen-1-one (7j)



According to the synthetic procedure for **7a**, this compound was obtained in 89% yield (723 mg, 1.80 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (700 mg, 2.01 mmol) with 2-ethynyl-6-methoxynaphthalene (399 mg, 2.19 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (29 mg, 40 μmol) and CuI (25 mg, 130 μmol) in a mixed solvent of DMF (2.0 mL) and Et_3N (0.8 mL) for 3 h at 55 °C and the following column chromatography on silica gel (hexane/EtOAc = 10 : 1). Pale yellow crystals (from EtOAc); Mp. 169-171 °C; IR (ATR) ν 1731, 1666, 1643, 1235, 1216, 892, 765 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 3.95 (3H, s), 7.14 (1H, d, J = 2.5 Hz), 7.19 (1H, dd, J = 8.9, 2.5 Hz), 7.48-7.57 (4H, m), 7.61 (1H, td, J = 7.7, 0.9 Hz), 7.74 (2H, d, J = 8.9 Hz), 7.85-7.92 (1H, m), 7.97 (1H, brs), 8.18 (1H, d, J = 7.7 Hz), 8.26-8.31 (2H, m), 8.37 (1H, dd, J = 7.7, 0.9 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 55.4, 82.3, 98.2, 99.9, 105.9, 117.6, 119.70 (2C), 119.74, 125.5, 127.1, 128.2, 128.5, 128.70, 128.72, 129.4, 129.5, 130.4, 131.2, 132.4, 134.4, 135.2, 137.2, 156.5, 158.6, 161.1; MS (ESI-TOF) m/z 403 $[M+H]^+$; HRMS calcd for $C_{28}H_{19}O_3$ $[M+H]^+$, 403.1334; found, 40.31338.

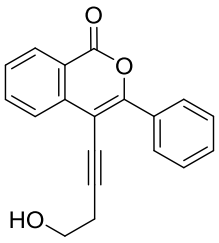
3-Phenyl-4-(thiophen-2-ylethynyl)-1*H*-isochromen-1-one (7k)



According to the synthetic procedure for **7a**, this compound was obtained in 81% yield (538 mg, 1.64 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (701 mg, 2.01 mmol) with 3-ethynylthiophene (354 μL, 3.59 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (56 mg, 80 μmol) and CuI (50 mg, 0.26 mmol) in a mixed solvent of DMF (2.0 mL) and Et_3N (0.8 mL) for 3 h at 55 °C and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1 ~ 10 : 1). Colorless crystals (from Et_2O); Mp. 108-110 °C; IR (ATR) ν 3100, 1733, 1719, 1098, 789, 759, 689 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 7.21 (1H, dd, J = 5.0, 1.0 Hz), 7.35 (1H, dd, J = 5.0, 3.0 Hz), 7.46-7.53 (3H, m), 7.55 (1H, dd, J = 3.0, 1.0 Hz), 7.56-7.62 (1H, m), 7.83-7.88 (1H, m), 8.08 (1H, d, J = 8.0 Hz), 8.19-8.25 (2H, m), 8.35 (1H, d, J = 7.9 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 82.2, 92.8, 99.7, 119.7, 121.8, 125.4, 125.8, 128.2, 128.6, 128.7, 129.0, 129.49, 129.52, 130.4, 132.3, 135.2, 137.0, 156.6, 161.0; MS (ESI-TOF) m/z 329 $[M+H]^+$; HRMS calcd for $C_{21}H_{13}O_2S$ $[M+H]^+$, 329.0636; found,

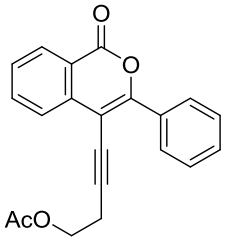
329.0640.

4-(4-Hydroxybut-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-one (7p)



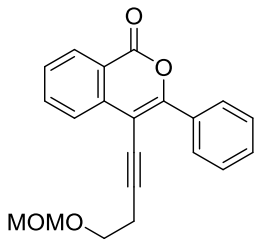
According to the synthetic procedure for **7a**, this compound was obtained in 81% yield (1.41 g, 4.86 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (2.10 g, 6.03 mmol) with but-3-yn-1-ol (1.37 mL, 18.1 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (0.42 g, 0.60 mmol) and CuI (0.12 g, 0.60 mmol) in a mixed solvent of DMF (6.0 mL) and Et_3N (4.0 mL) for 1 h at 55 °C and the following column chromatography on silica gel (hexane/EtOAc = 1 : 1). Colorless crystals (from hexane/EtOAc); Mp. 87.5-90.0 °C; IR (ATR) ν 3281, 3072, 1721, 1605, 1480, 1177, 1041, 759, 692 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.79 (2H, t, J = 6.2 Hz), 3.85 (2H, t, J = 6.2 Hz), 7.45-7.51 (3H, m), 7.56 (1H, dd, J = 7.4, 1.1 Hz), 7.79-7.85 (1H, m), 8.00 (1H, d, J = 8.0 Hz), 8.12-8.16 (2H, m), 8.32 (1H, dd, J = 7.9, 0.8 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 24.3, 60.9, 75.4, 95.3, 99.8, 119.7, 125.4, 128.2, 128.5, 128.7, 129.5, 130.4, 132.4, 135.1, 137.3, 156.7, 161.1; MS (ESI-TOF) m/z 291 $[M+H]^+$; HRMS calcd for $C_{19}H_{15}O_3$ $[M+H]^+$, 291.1021; found, 291.1024.

4-(1-Oxo-3-phenyl-1*H*-isochromen-4-yl)but-3-yn-1-yl acetate (7m)



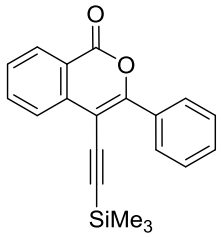
A solution of 4-(4-hydroxybut-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-one **7p** (584 mg, 2.01 mmol) in pyridine (1.0 mL) was treated with Ac_2O (1.0 mL) for 1 h at room temperature. After evaporation of the reaction mixture, the residue was purified by column chromatography on silica gel to give the corresponding acetate **7m** in 87% yield (580 mg, 1.75 mmol). Colorless crystals (from hexane/EtOAc); Mp. 81.0-83.0 °C; IR (ATR) ν 1740, 1729, 1720, 1605, 1481, 1044, 759, 691 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.08 (3H, s), 2.87 (2H, t, J = 6.6 Hz), 4.30 (2H, t, J = 6.6 Hz), 7.43-7.51 (3H, m), 7.54-7.60 (1H, m), 7.79-7.85 (1H, m), 7.99 (1H, dd, J = 8.0, 0.4 Hz), 8.11-8.17 (2H, m), 8.32 (1H, dd, J = 7.7, 0.4 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 20.3, 20.9, 62.0, 75.2, 94.3, 99.6, 119.7, 125.3, 128.1, 128.5, 128.6, 129.4, 130.3, 132.3, 135.0, 137.3, 156.7, 161.0, 170.8; MS (ESI-TOF) m/z 333 $[M+H]^+$; HRMS calcd for $C_{21}H_{17}O_4$ $[M+H]^+$, 333.1127; found, 333.1120. Anal. Calcd for $C_{21}H_{16}O_4$: C, 75.89; H, 4.85. Found: C, 75.68; H, 4.81.

4-(4-(Methoxymethoxy)but-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-one (7n)



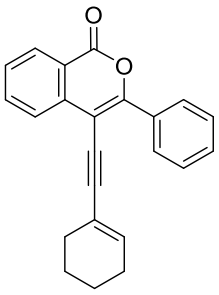
To a solution of 4-(4-hydroxybut-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-one **7p** (880 mg, 3.03 mmol) and *i*-Pr_2NEt (0.62 mL, 3.6 mmol) in CH_2Cl_2 (15 mL), MOMCl (0.28 mL, 3.6 mmol) was added at 0 °C. After being stirred for 10 h at room temperature, the reaction mixture was quenched with saturated aqueous $NaHCO_3$ solution (15 mL), extracted with EtOAc (20 mL x 3), and washed with brine (15 mL). The combined organic layer was dried over anhydrous Na_2SO_4 and evaporated. The resulting residue was purified by flash column chromatography on silica gel (hexane/EtOAc = 5 : 1) to give the MOM ether **7n** in 57% yield (588 mg, 1.76 mmol). Colorless oil; IR (neat) ν 3071, 2945, 2890, 1744, 1732, 1608, 1483, 1113, 1080, 1034, 766, 699 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 2.82 (2H, t, J = 6.7 Hz), 3.37 (3H, s), 3.78 (2H, t, J = 6.7 Hz), 4.69 (2H, s), 7.44-7.49 (3H, m), 7.56 (1H, td, J = 7.9, 0.9 Hz), 7.78-7.84 (1H, m), 8.03 (1H, d, J = 7. 9 Hz), 8.15-8.20 (2H, m), 8.32 (1H, d, J = 7.9 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.4, 55.4, 65.8, 74.6, 95.8, 96.6, 99.8, 119.7, 125.5 (2C), 128.1, 128.5, 129.4, 130.2, 132.4, 135.0, 137.5, 156.4, 161.1; MS (ESI-TOF) m/z 357 $[M+Na]^+$; HRMS calcd for $C_{21}H_{18}NaO_4$ $[M+Na]^+$, 357.1103; found, 357.1116. Anal. Calcd for $C_{21}H_{18}O_4$: C, 75.43; H, 5.43. Found: C, 75.25; H, 5.44.

3-Phenyl-4-((trimethylsilyl)ethynyl)-1*H*-isochromen-1-one (7o)



According to the synthetic procedure for **7a**, this compound was obtained in 83% yield (763 mg, 2.40 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (1.04 g, 2.99 mmol) with ethynyltrimethylsilane (1.25 mL, 9.04 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (0.21 g, 0.30 mmol) and CuI (57 mg, 0.30 mmol) in a mixed solvent of DMF (3.0 mL) and Et_3N (12 mL) for 3 h at 55 °C and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1). Colorless crystals (from hexane); Mp. 121-123 °C; IR (ATR) ν 3067, 2956, 2148, 1720, 1481, 1243, 1153, 1096, 897, 758, 677 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.29 (9H, s), 7.43-7.52 (3H, m), 7.57 (1H, td, J = 7.9, 1.1 Hz), 7.81-7.88 (1H, m), 8.01 (1H, dd, J = 7.9, 0.4 Hz), 8.19-8.27 (2H, m), 8.32 (1H, dd, J = 7.9, 1.1 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ –0.3, 98.0, 99.7, 104.3, 119.6, 125.5, 128.0, 128.66, 128.71, 129.4, 130.5, 132.1, 135.1, 137.0, 157.6, 160.9.; MS (ESI-TOF) m/z 341 $[M+Na]^+$; HRMS calcd for $C_{20}H_{18}NaO_2Si$ $[M+Na]^+$, 341.0974; found, 341.0972. Anal. Calcd for $C_{20}H_{18}O_2Si$: C, 75.43; H, 5.70. Found: C, 74.41; H, 5.69.

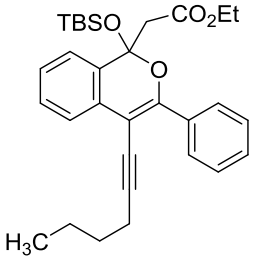
4-(Cyclohex-1-en-1-ylethynyl)-3-phenyl-1*H*-isochromen-1-one (7q)



According to the synthetic procedure for **7a**, this compound was obtained in 94% yield (927 mg, 2.82 mmol) by the reaction of 4-iodo-3-phenyl-1*H*-isochromen-1-one **6** (1.04 g, 2.99 mmol) with 1-ethynylcyclohexene (1.06 mL, 9.00 mmol) in the presence of $(Ph_3P)_2PdCl_2$ (0.21 g, 0.30 mmol) and CuI (57 mg, 0.30 mmol) in a mixed solvent of DMF (3.0 mL) and Et_3N (12 mL) for 3 h at 55 °C and the following column chromatography on silica gel (hexane/CH_2Cl_2 = 3 : 1 ~ 1 : 1). Colorless crystals (from hexane); Mp. 145-148 °C; IR (ATR) ν 3051, 3019, 2931, 1736, 1142, 760, 689, 549 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.61-1.68 (2H, m), 1.68-1.76 (2H, m), 2.14-2.21 (2H, m), 2.24-2.30 (2H, m), 6.23-6.27 (1H, m), 7.43-7.51 (3H, m), 7.56 (1H, td, *J* = 8.0, 0.5 Hz), 7.79-7.85 (1H, m), 8.01 (1H, d, *J* = 8.0 Hz), 8.17-8.23 (2H, m), 8.33 (1H, dd, *J* = 7.9, 0.5 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ 21.5, 22.2, 25.8, 28.8, 80.0, 99.7, 100.1, 119.8, 120.7, 125.5, 128.0, 128.54, 128.56, 129.4, 130.2, 132.5, 135.0, 135.9, 137.3, 157.9, 161.1; MS (ESI-TOF) *m*/*z* 327 $[M+H]^+$; HRMS calcd for $C_{23}H_{19}O_2$ $[M+H]^+$, 327.1385; found, 327.1383. Anal. Calcd for $C_{23}H_{18}O_2$: C, 84.64; H, 5.56. Found: C, 84.69; H, 5.59.

3. Zwitterion-induced addition reaction

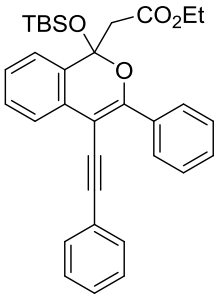
Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(hex-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-yl)acetate (8a)



To a solution of 4-(hex-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-one **7a** (601 mg, 1.99 mmol) and zwitterion **1** (10 mg, 19 μmol) in CH_2Cl_2 (6.0 mL), a solution of *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (483 mg, 2.39 mmol) in CH_2Cl_2 (2.0 mL) was slowly added at 0 °C over 1 h using a syringe pump. After being stirred for additional 1 h at room temperature, the reaction mixture was quenched with Et_3N (1.0 mL). After usual extractive workup, the resulting residue was purified by column chromatography on alumina (hexane/EtOAc = 20 : 1) to give silyl acetal **8a** in 95% yield (949 mg, 1.88 mmol). Colorless oil; IR (neat) ν 3060, 2931, 2852, 1738, 1598, 1483, 1324, 1250, 1178, 1017, 838, 760, 697 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.20 (3H, s), –0.12 (3H, s), 0.85 (9H, s), 0.93 (3H, t, *J* = 7.3 Hz), 1.09 (3H, t, *J* = 7.2 Hz), 1.39-1.51 (2H, m), 1.52-1.62 (2H, m), 2.47 (2H, t, *J* = 7.0 Hz), 3.01 (1H, d, *J* = 13.8 Hz), 3.12 (1H, d, *J* = 13.8 Hz), 3.92-4.09 (2H, m), 7.34 (1H, td, *J* = 7.4, 1.2 Hz), 7.39-7.48 (5H, m), 7.64 (1H, d, *J* = 7.4 Hz), 8.11-8.17 (2H, m); ^{13}C NMR (100 MHz,

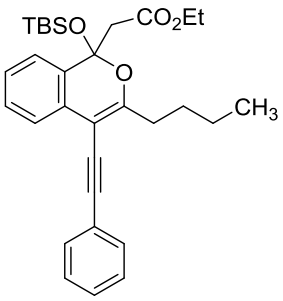
CD_3CN) δ –4.0, –3.5, 13.3, 13.8, 18.1, 19.3, 22.2, 25.5, 30.9, 47.5, 60.8, 75.6, 96.9, 97.6, 101.1, 124.0, 124.9, 127.9, 128.2, 129.2, 129.5, 130.0, 130.4, 131.9, 134.7, 153.7, 168.6; MS (ESI-TOF) *m*/*z* 527 $[M+Na]^+$; HRMS calcd for $C_{31}H_{40}NaO_4Si$ $[M+Na]^+$, 527.2594; found, 527.2604.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-(phenylethynyl)-1*H*-isochromen-1-yl)acetate (8b)



According to the synthetic procedure for **8a**, this compound was obtained in 90% yield (954 mg, 1.82 mmol) by the reaction of 3-phenyl-4-(phenylethynyl)-1*H*-isochromen-1-one **7b** (652 mg, 2.02 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (492 mg, 2.43 mmol) in the presence of zwitterion **1** (11 mg, 21 μmol) in CH_2Cl_2 (2.0 mL) for 1 h at 0 °C and the following column chromatography on alumina (hexane/EtOAc = 20 : 1). Colorless crystals (from hexane); Mp. 83.5-86.5 °C; IR (ATR) ν 3061, 2980, 2952, 2855, 2206, 1729, 1593, 1561, 1330, 1190, 1014, 887, 759, 689 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.17 (3H, s), –0.09 (3H, s), 0.87 (9H, s), 1.10 (3H, t, *J* = 7.1 Hz), 3.06 (1H, d, *J* = 14.0 Hz), 3.18 (1H, d, *J* = 14.0 Hz), 3.95-4.08 (2H, m), 7.35-7.42 (4H, m), 7.45-7.54 (7H, m), 7.76 (1H, d, *J* = 7.6 Hz), 8.18-8.23 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.0, –3.6, 13.8, 18.0, 25.5, 47.6, 60.8, 85.4, 95.2, 96.9, 101.5, 123.8, 123.9, 125.1, 128.1, 128.4, 128.7, 129.1, 129.4, 129.6, 129.7, 130.4, 131.3, 131.7, 134.5, 155.2, 168.5; MS (ESI-TOF) *m*/*z* 547 $[M+Na]^+$; HRMS calcd for $C_{33}H_{36}NaO_4Si$ $[M+Na]^+$, 547.2281; found, 547.2272.

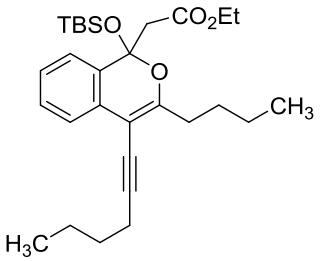
Ethyl 2-(3-butyl-1-((*tert*-butyldimethylsilyl)oxy)-4-(phenylethynyl)-1*H*-isochromen-1-yl)acetate (8c)



According to the synthetic procedure for **8a**, this compound was obtained in 97% yield (980 mg, 1.94 mmol) by the reaction of 3-butyl-4-(phenylethynyl)-1*H*-isochromen-1-one **7c** (605 mg, 2.00 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (486 mg, 2.41 mmol) in the presence of zwitterion **1** (11 mg, 21 μmol) in CH_2Cl_2 (8.0 mL) for 0.5 h at 0 °C and the following column chromatography on alumina (hexane/EtOAc = 20 : 1). Colorless oil; IR (neat) ν 2952, 2851, 2203, 1739, 1619, 1257, 1178, 1019, 839, 757 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.15 (3H, s), 0.03 (3H, s), 0.90 (9H, s), 0.95 (3H, t, *J* = 7.1 Hz), 1.12 (3H, t, *J* = 7.3 Hz), 1.37-1.49 (2H, m), 1.69 (2H, quint, *J* = 7.5 Hz), 2.63 (1H, dt, *J* = 13.7, 7.5 Hz), 2.74

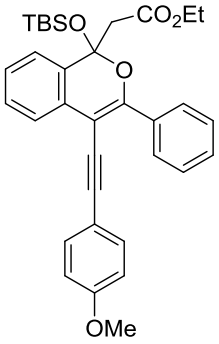
(1H, dt, *J* = 13.7, 7.5 Hz), 2.87 (1H, d, *J* = 13.8 Hz), 3.01 (1H, d, *J* = 13.8 Hz), 3.91-4.07 (2H, m), 7.28 (1H, td, *J* = 7.2, 1.2 Hz), 7.33-7.42 (5H, m), 7.51-7.56 (3H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –3.9, –3.4, 13.6, 13.9, 18.1, 22.4, 25.5, 29.1, 32.8, 48.0, 60.7, 84.3, 95.2, 96.4, 101.6, 122.7, 124.1, 125.3, 127.3, 128.5, 129.01, 129.03, 129.5, 131.2, 131.3, 161.4, 168.3; MS (ESI-TOF) *m*/*z* 527 $[M+Na]^+$; HRMS calcd for $C_{31}H_{40}NaO_4Si$ $[M+Na]^+$, 527.2594; found, 527.2593.

Ethyl 2-(3-butyl-1-((*tert*-butyldimethylsilyl)oxy)-4-(hex-1-yn-1-yl)-1*H*-isochromen-1-yl)acetate (8d)



According to the synthetic procedure for **8a**, this compound was obtained in 99% yield by the reaction of 3-butyl-4-(hex-1-yn-1-yl)-1*H*-isochromen-1-one **7d** (93% purity, 242 mg, 0.858 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (208 mg, 1.03 mmol) in the presence of zwitterion **1** (4.5 mg, 8.6 μmol) in CH_2Cl_2 (3.5 mL) for 3 h at 0 °C and the following column chromatography on alumina (hexane/EtOAc = 100 : 1). Colorless oil; IR (neat) ν 2950, 2927, 2853, 1738, 1619, 1321, 1247, 1177, 1132, 1018, 836, 779, 760 cm^{-1}; ^{1}H NMR (400 MHz, CD_3CN) δ –0.18 (3H, s), 0.00 (3H, s), 0.89 (9H, s), 0.93 (3H, t, *J* = 7.6 Hz), 0.95 (3H, t, *J* = 7.2 Hz), 1.11 (3H, t, *J* = 7.2 Hz), 1.38 (2H, sex, *J* = 7.2 Hz), 1.44-1.55 (2H, m), 1.55-1.67 (4H, m), 2.47 (2H, t, *J* = 6.8 Hz), 2.47-2.55 (1H, m), 2.61 (1H, dt, *J* = 13.6, 7.2 Hz), 2.81 (1H, d, *J* = 13.6 Hz), 2.94 (1H, d, *J* = 13.6 Hz), 3.90-4.03 (2H, m), 7.23 (1H, td, *J* = 7.4, 1.3 Hz), 7.31-7.36 (2H, m), 7.39 (1H, dd, *J* = 7.7, 1.3 Hz); ^{13}C NMR (100 MHz, CD_3CN) δ –4.0, –3.5, 13.3, 13.5, 13.8, 18.0, 19.1, 22.1, 22.4, 25.5, 29.0, 31.2, 32.5, 47.8, 60.6, 74.6, 95.9, 96.8, 101.2, 122.5, 125.1, 127.0, 129.3, 129.6, 131.3, 159.7, 168.3; MS (ESI-TOF) *m*/*z* 507 $[M+Na]^+$; HRMS calcd for $C_{29}H_{44}NaO_4Si$ $[M+Na]^+$, 507.2907; found, 507.2910.

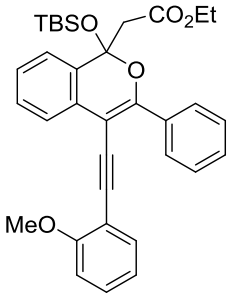
Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-((4-methoxyphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-yl)-acetate (8e)



According to the synthetic procedure for **8a**, this compound was obtained in 95% yield (269 mg, 0.485 mmol) by the reaction of 4-((4-methoxyphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-one **7e** (180 mg, 0.511 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (124 mg, 0.614 mmol) in the presence of zwitterion **1** (2.7 mg, 5.1 μmol) in CH_2Cl_2 (2.4 mL) for 1 h at 0 °C and the following column chromatography on alumina

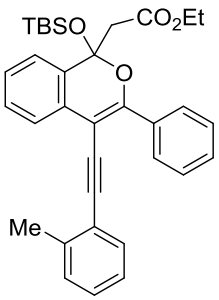
(hexane/EtOAc = 20 : 1). Colorless oil; IR (neat) ν 2952, 2928, 2858, 1739, 1606, 1509, 1253, 1183, 1023, 837, 761 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.17 (3H, s), –0.09 (3H, s), 0.87 (9H, s), 1.10 (3H, t, *J* = 7.2 Hz), 3.04 (1H, d, *J* = 14.1 Hz), 3.18 (1H, d, *J* = 14.1 Hz), 3.80 (3H, s), 3.90-4.09 (2H, m), 6.93 (2H, d, *J* = 8.8 Hz), 7.34-7.41 (1H, m), 7.41-7.53 (7H, m), 7.54 (1H, d, *J* = 8.0 Hz), 8.17-8.26 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.0, –3.5, 13.8, 18.1, 25.5, 47.6, 55.5, 60.8, 83.8, 95.3, 97.2, 101.4, 114.7, 115.8, 124.0, 125.0, 128.0, 128.3, 129.3, 129.7, 129.8, 130.3, 131.8, 132.8, 134.6, 154.5, 160.2, 168.5; MS (ESI-TOF) *m*/*z* 577 $[M+Na]^+$; HRMS calcd for $C_{34}H_{38}NaO_5Si$ $[M+Na]^+$, 577.2386; found, 577.2379. Anal. Calcd for $C_{34}H_{38}O_5$: C, 73.61; H, 6.90. Found: C, 73.50; H, 7.00.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-((2-methoxyphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-yl)-acetate (8f)



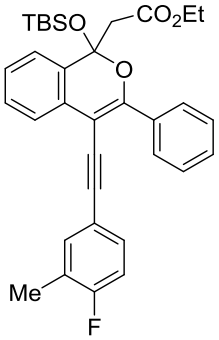
According to the synthetic procedure for **3a**, this compound was obtained in 94% yield (520 mg, 0.937 mmol) by the reaction of 4-((2-methoxyphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-one **7f** (356 mg, 1.01 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (245 mg, 1.21 mmol) in the presence of zwitterion **1** (5.3 mg, 10 μmol) in CH_2Cl_2 (4.0 mL) for 1 h at 0 °C and the following column chromatography on alumina (hexane/CH_2Cl_2/EtOAc = 20 : 20 : 1). Pale yellow crystals (from hexane/EtOAc); Mp. 67.0-69.5 °C; IR (ATR) ν 2953, 2930, 1726, 1192, 1149, 1134, 1013, 745 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.17 (3H, s), –0.08 (3H, s), 0.87 (9H, s), 1.10 (3H, t, *J* = 7.2 Hz), 3.05 (1H, d, *J* = 13.9 Hz), 3.18 (1H, d, *J* = 13.9 Hz), 3.92 (3H, s), 3.95-4.10 (2H, m), 6.96 (1H, td, *J* = 7.5, 1.0 Hz), 7.03 (1H, d, *J* = 8.4 Hz), 7.31-7.44 (3H, m), 7.44-7.54 (5H, m), 7.87 (1H, d, *J* = 7.7 Hz), 8.26-8.31 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.0, –3.5, 13.8, 18.1, 25.5, 47.6, 55.9, 60.8, 89.1, 92.7, 97.3, 101.5, 111.5, 112.9, 120.9, 124.2, 125.0, 128.1, 128.3, 129.3, 129.7, 129.9, 130.2, 130.3, 131.8, 132.8, 134.4, 154.3, 160.4, 168.6; MS (ESI-TOF) *m*/*z* 577 $[M+Na]^+$; HRMS calcd for $C_{34}H_{38}NaO_5Si$ $[M+Na]^+$, 577.2386; found, 577.2385.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-(*o*-tolylethynyl)-1*H*-isochromen-1-yl)acetate (8g)



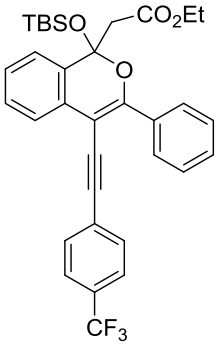
According to the synthetic procedure for **8a**, this compound was obtained in 88% yield (386 mg, 0.716 mmol) by the reaction of 3-phenyl-4-(*o*-tolylethynyl)-1*H*-isochromen-1-one **7g** (274 mg, 0.815 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (230 mg, 1.14 mmol) in the presence of zwitterion **1** (5.4 mg, 10 μmol) in CH_2Cl_2 (6.0 mL) for 2 h at 0 °C and the following column chromatography on silica gel (hexane/ EtOAc = 40 : 1). Yellow oil; IR (neat) ν 3058, 2960, 2941, 2852, 1732, 1483, 1251, 1182, 1118, 760 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.18 (3H, s), –0.09 (3H, s), 0.87 (9H, s), 1.10 (3H, t, J = 7.1 Hz), 2.41 (3H, s), 3.03 (1H, d, J = 13.9 Hz), 3.16 (1H, d, J = 13.9 Hz), 3.93-4.10 (2H, m), 7.13-7.25 (3H, m), 7.34-7.39 (1H, m), 7.41-7.50 (6H, m), 7.78 (1H, d, J = 7.8 Hz), 8.17-8.23 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –3.9, –3.4, 13.9, 18.1, 20.8, 25.6, 47.6, 60.8, 89.2, 94.4, 97.3, 101.5, 123.6, 124.0, 125.0, 126.3, 128.1, 128.4, 128.7, 129.5, 129.7, 129.9, 130.1, 130.4, 131.8, 131.9, 134.5, 139.9, 155.0, 168.5; MS (ESI-TOF) m/z 561.2437 $[M+Na]^+$; HRMS calcd for $C_{34}H_{38}NaO_4Si$ $[M+Na]^+$, 561.2437; found, 561.2442.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-((4-fluoro-3-methylphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-yl)acetate (8h)



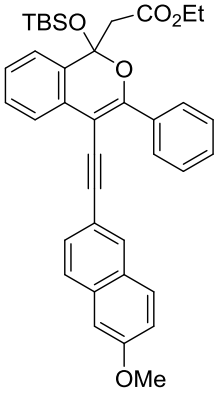
According to the synthetic procedure for **8a**, this compound was obtained in 88% yield (486 mg, 0.873 mmol) by the reaction of 4-((4-fluoro-3-methylphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-one **7h** (352 mg, 0.992 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (245 mg, 1.21 mmol) in the presence of zwitterion **1** (5.6 mg, 11 μmol) in CH_2Cl_2 (7.0 mL) for 1 h at 0 °C and the following column chromatography on silica gel (hexane/ EtOAc = 40 : 1). Yellow oil; IR (neat) ν 3057, 2951, 2932, 2853, 1738, 1498, 1234, 1110, 839, 760 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.18 (3H, s), –0.08 (3H, s), 0.87 (9H, s), 1.11 (3H, t, J = 7.2 Hz), 2.23 (3H, d, J_{HF} = 1.7 Hz), 3.01 (1H, d, J = 13.8 Hz), 3.15 (1H, d, J = 13.8 Hz), 3.95-4.09 (2H, m), 7.01 (1H, d, J_{HF} = 9.6 Hz, J_{HH} = 8.4 Hz), 7.25-7.32 (1H, m), 7.33-7.39 (2H, m), 7.40-7.51 (5H, m), 7.74 (1H, d, J = 7.3 Hz), 8.19-8.23 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –3.8, –3.3, 14.0, 18.2, 25.7, 47.6, 60.9, 84.9, 94.6, 97.0, 101.6, 115.8 (d, J_{CF} = 22.1 Hz), 119.9 (d, J_{CF} = 4.0 Hz), 124.1, 125.0, 126.0 (d, J_{CF} = 18.1 Hz), 128.2, 128.4, 129.4, 129.5, 129.7, 130.5, 130.75, 130.83, 131.8, 134.5, 134.6 (br), 155.1, 161.4 (d, J_{CF} = 246 Hz), 168.5; ^{19}F NMR (376 MHz, CD_3CN) δ –53.9~–53.7 (1F, m); MS (ESI-TOF) m/z 579 $[M+Na]^+$; HRMS calcd for $C_{34}H_{37}FNaO_4Si$ $[M+Na]^+$, 579.2343; found, 579.2353.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-((4-(trifluoromethyl)phenyl)ethynyl)-1*H*-isochromen-1-yl)acetate (8i)



According to the synthetic procedure for **8a**, this compound was obtained in 95% yield (278 mg, 0.469 mmol) by the reaction of 3-phenyl-4-((4-(trifluoromethyl)phenyl)ethynyl)-1*H*-isochromen-1-one **7i** (193 mg, 0.494 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (120 mg, 0.599 mmol) in the presence of zwitterion **1** (2.6 mg, 5.4 μmol) in CH_2Cl_2 (3.5 mL) for 1 h at 0 °C and the following column chromatography on silica gel (hexane/ EtOAc = 40 : 1). Yellow oil; IR (neat) ν 2941, 2857, 2203, 1739, 1601, 1324, 1168, 1130, 1069, 840 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.18 (3H, s), –0.10 (3H, s), 0.86 (9H, s), 1.10 (3H, t, J = 7.1 Hz), 3.04 (1H, d, J = 13.9 Hz), 3.17 (1H, d, J = 13.9 Hz), 3.92-4.09 (2H, m), 7.37-7.40 (1H, m), 7.45-7.54 (5H, m), 7.61 (2H, d, J = 8.2 Hz), 7.67 (2H, d, J = 8.2 Hz), 7.77 (1H, dd, J = 7.2, 1.2 Hz), 8.16-8.21 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.7, –4.2, 13.1, 17.4, 24.8, 47.0, 60.2, 87.7, 93.2, 95.7, 101.0, 123.2, 124.0 (q, J_{CF} = 271 Hz), 124.4, 125.2 (q, J_{CF} = 4.0 Hz), 127.3, 127.5, 127.7, 128.6, 128.7 (q, J_{CF} = 32.2 Hz), 128.8, 129.1, 130.0, 130.9, 131.0, 133.6, 155.7, 167.8; ^{19}F NMR (376 MHz, CD_3CN) δ –0.04 (3F, s); MS (ESI-TOF) m/z 615 $[M+Na]^+$; HRMS calcd for $C_{34}H_{35}F_3NaO_4Si$ $[M+Na]^+$, 615.2154; found, 615.2141.

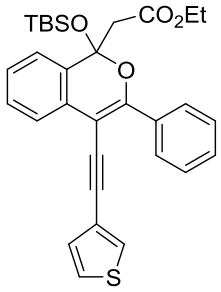
Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-((6-methoxynaphthalen-2-yl)ethynyl)-3-phenyl-1*H*-isochromen-1-yl)acetate (8j)



According to the synthetic procedure for **8a**, this compound was obtained in 92% yield (554 mg, 0.916 mmol) by the reaction of 4-((6-methoxynaphthalen-2-yl)ethynyl)-3-phenyl-1*H*-isochromen-1-one **7j** (400 mg, 0.994 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (245 mg, 1.21 mmol) in the presence of zwitterion **1** (5.3 mg, 11 μmol) in CH_2Cl_2 (8.5 mL) for 1 h at room temperature and the following column chromatography on silica gel (hexane/ EtOAc = 25 : 1). Pale yellow oil; IR (neat) ν 3055, 2953, 2924, 2856, 1738, 1597, 1482, 1246, 1037, 838 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.18 (3H, s), –0.09 (3H, s), 0.86 (9H, s), 1.10

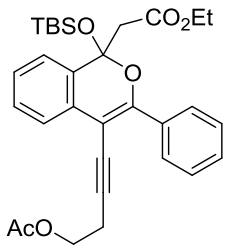
(3H, t, *J* = 7.1 Hz), 3.04 (1H, d, *J* = 13.9 Hz), 3.18 (1H, d, *J* = 13.9 Hz), 3.87 (3H, s), 3.95-4.07 (2H, m), 7.16 (1H, dd, *J* = 9.0, 2.4 Hz), 7.23 (1H, d, *J* = 2.4 Hz), 7.34-7.40 (1H, m), 7.44-7.55 (6H, m), 7.75 (2H, d, *J* = 8.7 Hz), 7.79-7.83 (1H, m), 7.93 (1H, brs), 8.22-8.27 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.6, –4.1, 13.2, 17.5, 24.9, 46.9, 54.9, 60.2, 84.4, 95.3, 96.5, 100.9, 105.8, 118.1, 119.3, 123.3, 124.4, 126.9, 127.5, 127.8, 128.1, 128.3, 128.8, 129.0, 129.10, 129.12, 129.8, 130.2, 131.1, 133.9, 134.0, 154.3, 158.3, 167.9; MS (ESI-TOF) *m*/*z* 627 $[M+Na]^+$; HRMS calcd for $C_{38}H_{40}NaO_5Si$ $[M+Na]^+$, 627.2543; found, 627.2534.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-(thiophen-2-ylethynyl)-1*H*-isochromen-1-yl)acetate (8k)



According to the synthetic procedure for **8a**, this compound was obtained in 95% yield (251 mg, 0.473 mmol) by the reaction of 3-phenyl-4-(thiophen-2-ylethynyl)-1*H*-isochromen-1-one **7k** (164 mg, 0.499 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (248 mg, 1.23 mmol) in the presence of zwitterion **1** (7.8 mg, 15 μmol) in CH_2Cl_2 (3.0 mL) for 4 h at 0 °C and the following column chromatography on silica gel (hexane/EtOAc = 50 : 1). Yellow oil; IR (neat) ν 2935, 2850, 1734, 1598, 1258, 1181, 783, 759 cm^{-1}; ^{1}H NMR (400 MHz, CD_3CN) δ –0.18 (3H, s), –0.09 (3H, s), 1.87 (9H, s), 1.10 (3H, t, *J* = 7.1 Hz), 3.03 (1H, d, *J* = 13.9 Hz), 3.17 (1H, d, *J* = 13.9 Hz), 3.97-4.09 (2H, m), 7.18 (1H, dd, *J* = 5.0, 1.0 Hz), 7.34-7.40 (1H, m), 7.43 (1H, dd, *J* = 5.0, 3.0 Hz), 7.45-7.52 (5H, m), 7.57 (1H, dd, *J* = 3.0, 1.0 Hz), 7.73 (1H, d, *J* = 7.8 Hz), 8.16-8.21 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.0, –3.5, 13.8, 18.0, 25.5, 47.5, 60.8, 84.6, 90.6, 96.9, 101.5, 122.7, 123.9, 125.0, 126.7, 128.1, 128.3, 128.7, 129.3, 129.63, 129.67, 129.72, 130.4, 131.7, 134.4, 154.9, 168.5; MS (ESI-TOF) *m*/*z* 553 $[M+Na]^+$; HRMS calcd for $C_{31}H_{34}O_4NaSSi$ $[M+Na]^+$, 553.1845; found, 553.1851.

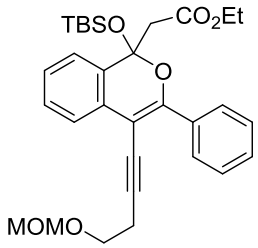
Ethyl 2-(4-(4-acetoxybut-1-yn-1-yl)-1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-1*H*-isochromen-1-yl)acetate (8m)



According to the synthetic procedure for **8a**, this compound was obtained in 94% yield (503 mg, 0.940 mmol) by the reaction of 4-(1-oxo-3-phenyl-1*H*-isochromen-4-yl)but-3-yn-1-yl acetate **7m** (333 mg, 1.00 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (249 mg, 1.23 mmol) in the presence of zwitterion **1** (5.3 mg, 10

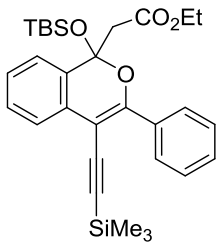
μmol) in CH_2Cl_2 (4.0 mL) for 0.5 h at 0 °C and the following column chromatography on silica gel (hexane/EtOAc = 10 : 1). Colorless oil; IR (neat) ν 2959, 2943, 2856, 1743, 1239, 1181, 1042, 840, 762 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.20 (3H, s), –0.12 (3H, s), 0.86 (9H, s), 1.09 (3H, t, *J* = 7.1 Hz), 2.00 (3H, s), 2.79 (2H, t, *J* = 6.5 Hz), 3.00 (1H, d, *J* = 13.8 Hz), 3.13 (1H, d, *J* = 13.8 Hz), 3.93-4.06 (2H, m), 4.20 (2H, t, *J* = 6.5 Hz), 7.34 (1H, td, *J* = 7.5, 1.3 Hz), 7.40-7.48 (5H, m), 7.61-7.68 (1H, m), 8.10-8.18 (1H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.1, –3.6, 13.8, 18.0, 20.2, 20.5, 25.5, 47.4, 60.8, 62.6, 76.8, 92.9, 97.0, 101.2, 123.9, 124.9, 127.9, 128.2, 129.2, 129.5, 130.10, 130.11, 131.7, 134.5, 154.3, 168.5, 170.9; MS (ESI-TOF) *m*/*z* 557 $[M+Na]^+$; HRMS calcd for $C_{31}H_{38}NaO_6Si$ $[M+Na]^+$, 557.2335; found, 557.2329. Anal. Calcd for $C_{31}H_{38}O_6Si$: C, 69.72; H, 7.16. Found: C, 69.71; H, 7.28.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(4-(methoxymethoxy)but-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-yl)acetate (8n)



According to the synthetic procedure for **8a**, this compound was obtained in 96% yield (531 mg, 0.989 mmol) by the reaction of 4-(4-(methoxymethoxy)but-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-one **7n** (344 mg, 1.03 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (245 mg, 1.21 mmol) in the presence of zwitterion **1** (5.4 mg, 10 μmol) in CH_2Cl_2 (4.0 mL) for 1 h at 0 °C and the following column chromatography on silica gel (hexane/EtOAc = 10 : 1). Colorless oil; IR (neat) ν 2947, 2854, 1739, 1602, 1486, 1181, 1152, 1116, 1035, 840, 763 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.21 (3H, s), –0.12 (3H, s), 0.85 (9H, s), 1.09 (3H, t, *J* = 7.1 Hz), 2.73 (2H, t, *J* = 6.5 Hz), 3.00 (1H, d, *J* = 13.8 Hz), 3.12 (1H, d, *J* = 13.8 Hz), 3.29 (3H, s), 3.69 (2H, t, *J* = 6.5 Hz), 3.91-4.08 (2H, m), 4.63 (2H, s), 7.34 (1H, td, *J* = 7.5, 1.0 Hz), 7.40-7.49 (5H, m), 7.66 (1H, d, *J* = 7.5 Hz), 8.14-8.19 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.1, –3.6, 13.8, 18.0, 21.3, 25.5, 47.4, 54.9, 60.7, 66.2, 76.3, 94.3, 96.6, 97.2, 101.2, 124.0, 124.9, 127.9, 128.2, 129.1, 129.5, 130.0, 130.2, 131.7, 134.5, 154.0, 168.5; MS (ESI-TOF) *m*/*z* 559 $[M+Na]^+$; HRMS calcd for $C_{31}H_{40}NaO_6Si$ $[M+Na]^+$, 559.2492; found, 559.2488. Anal. Calcd for $C_{31}H_{40}O_6Si$: C, 69.37; H, 7.51. Found: C, 69.17; H, 7.39.

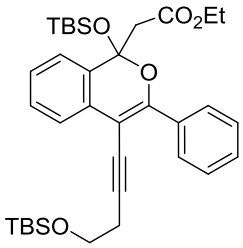
Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-((trimethylsilyl)ethynyl)-1*H*-isochromen-1-yl)acetate (8o)



According to the synthetic procedure for **8a**, this compound was obtained in 84% yield (427 mg, 0.820 mmol)

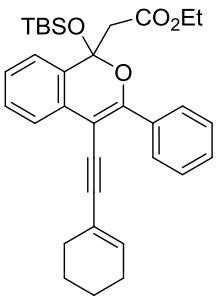
by the reaction of 3-phenyl-4-((trimethylsilyl)ethynyl)-1*H*-isochromen-1-one **7o** (308 mg, 0.969 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (235 mg, 1.16 mmol) in the presence of zwitterion **1** (5.2 mg, 9.9 μmol) in CH_2Cl_2 (4.0 mL) for 1.5 h at 0 °C and the following column chromatography on silica gel (hexane/EtOAc = 30 : 1). Colorless oil; IR (neat) ν 2956, 2146, 1736, 1250, 837, 757 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.19 (3H, s), –0.10 (3H, s), 0.24 (9H, s), 0.87 (9H, s), 1.10 (3H, t, *J* = 6.8 Hz), 3.02 (1H, d, *J* = 13.6 Hz), 3.13 (1H, d, *J* = 13.6 Hz), 3.92-4.10 (2H, m), 7.38 (1H, t, *J* = 7.6 Hz), 7.41-7.49 (5H, m), 7.52 (1H, d, *J* = 7.2 Hz), 8.16-8.22 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –4.1, –3.6, –0.7, 13.8, 18.0, 25.4, 47.7, 60.8, 96.8, 100.9, 101.3, 10.5, 123.8, 125.0, 128.1, 128.2, 129.39, 129.42, 129.6, 130.4, 131.6, 134.21, 155.9, 168.5; MS (ESI-TOF) *m*/*z* 521 $[M+H]^+$; HRMS calcd for $C_{30}H_{41}O_4Si_2$ $[M+H]^+$, 521.2543; found, 521.2553.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(4-((*tert*-butyldimethylsilyl)oxy)but-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-yl)acetate (8p)



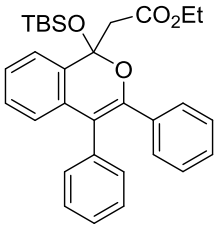
According to the synthetic procedure for 8**a**, this compound was obtained in 96% yield (589 mg, 0.970 mmol) by the reaction of 4-(4-hydroxybut-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-one **7p** (293 mg, 1.01 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (450 mg, 2.23 mmol) in the presence of zwitterion **1** (5.3 mg, 10 μmol) in CH_2Cl_2 (4.0 mL) at 0 °C for 3 h and the following column chromatography on alumina (hexane/EtOAc = 30 : 1). Colorless oil; IR (neat) ν 2944, 2850, 1737, 1601, 1484, 1183, 1025, 841, 761 cm^{-1}; ^1H NMR (400 MHz, CD_3CN) δ –0.21 (3H, s), –0.12 (3H, s), 0.07 (6H, s), 0.85 (9H, s), 0.89 (9H, s), 1.09 (3H, t, *J* = 7.2 Hz), 2.64 (2H, t, *J* = 6.5 Hz), 3.01 (1H, d, *J* = 13.9 Hz), 3.03 (1H, d, *J* = 13.9 Hz), 3.82 (2H, t, *J* = 6.5 Hz), 3.92-4.09 (2H, m), 7.33 (1H, td, *J* = 7.5, 1.3 Hz), 7.39-7.47 (5H, m), 7.67 (1H, brd, *J* = 7.8 Hz), 8.12-8.18 (2H, m); ^{13}C NMR (100 MHz, CD_3CN) δ –5.7, –4.1, –3.6, 13.8, 18.0, 18.3, 24.2, 25.5, 25.7, 47.4, 60.7, 62.1, 76.4, 94.4, 97.3, 101.0, 124.0, 124.8, 127.8, 128.2, 129.1, 129.4, 130.0, 130.2, 131.7, 134.6, 153.9, 168.5; MS (ESI-TOF) *m*/*z* 607 $[M+H]^+$; HRMS calcd for $C_{35}H_{51}O_5Si_2$ $[M+H]^+$, 607.3275; found, 607.3279.

Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(cyclohex-1-en-1-ylethynyl)-3-phenyl-1*H*-isochromen-1-yl)-acetate (8q)



According to the synthetic procedure for **8a**, this compound was obtained in 88% yield (464 mg, 0.878 mmol) by the reaction of 4-(cyclohex-1-en-1-ylethynyl)-3-phenyl-1*H*-isochromen-1-one **7q** (327 mg, 1.00 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (242 mg, 1.20 mmol) in the presence of zwitterion **1** (5.2 mg, 9.9 μmol) in CH_2Cl_2 (4.0 mL) at room temperature for 2 h and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1). Colorless crystals (from EtOAc); Mp. 104-107 °C; IR (ATR) ν 2926, 2855, 1732, 1185, 1150, 1046, 753 cm^{-1}; ^1H NMR (400 MHz, acetone-d_6) δ –0.16 (3H, s), –0.03 (3H, s), 0.90 (9H, s), 1.32 (3H, t, *J* = 7.1 Hz), 1.58-1.66 (2H, m), 1.66-1.73 (2H, m), 2.11-2.18 (2H, m), 2.20-2.28 (2H, m), 3.03 (1H, d, *J* = 13.8 Hz), 3.18 (1H, d, *J* = 13.8 Hz), 3.98-4.10 (2H, m), 6.11-6.18 (1H, m), 7.38 (1H, td, *J* = 7.4, 1.0 Hz), 7.42-7.51 (4H, m), 7.53 (1H, dd, *J* = 7.4, 1.0 Hz). 7.66 (1H, dd, *J* = 7.4, 0.8 Hz), 8.22-8.28 (2H, m); ^{13}C NMR (100 MHz, acetone-d_6) δ –4.0, –3.4, 13.9, 18.1, 22.5, 25.7, 25.8, 28.7, 47.3, 60.4, 97.4, 97.6, 101.5, 121.5, 124.8, 127.8, 128.0, 129.3, 129.4, 129.95, 130.01, 132.0, 134.2, 134.6, 153.9, 168.0; MS (ESI-TOF) *m*/*z* 551 $[M+Na]^+$; HRMS calcd for $C_{33}H_{40}NaO_4Si$ $[M+Na]^+$, 551.2594; found, 551.2597. Anal. Calcd for $C_{33}H_{40}O_4Si$: C, 74.96; H, 7.63. Found: 74.83; H, 7.70.

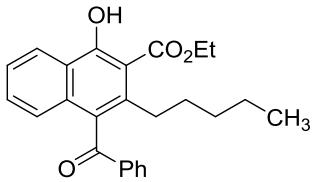
Ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3,4-diphenyl-1H-isochromen-1-yl)acetate



According to the synthetic procedure for **8a**, this compound was obtained in 90% yield (923 mg, 1.85 mmol) by the reaction of 3,4-diphenyl-1*H*-isochromen-1-one[3] (610 mg, 2.05 mmol) with *tert*-butyl((1-ethoxyvinyl)oxy)dimethylsilane (434 mg, 2.15 mmol) in the presence of zwitterion **1** (11 mg, 19 μmol) in CH_2Cl_2 (8.0 mL) at 0 °C for 4 h and the following column chromatography on neutral alumina (hexane/EtOAc = 50 : 1). Colorless crystals (from hexane/EtOAc); Mp. 102-104 °C; IR (ATR) ν 2981, 2952, 2854, 1737, 1332, 1185, 1145, 1018, 8355, 764 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ –0.11 (3H, s), –0.07 (3H, s), 0.89 (9H, s), 1.09 (3H, t, *J* = 7.2 Hz), 3.12 (1H, d, *J* = 13.8 Hz), 3.28 (1H, d, *J* = 13.8 Hz), 3.95-4.09 (2H, m), 6.82 (1H, dd, *J* = 6.9, 1.5 Hz), 7.12-7.39 (12H, m), 7.51 (1H, dd, *J* = 7.4, 1.2 Hz); ^{13}C NMR (100 MHz, $CDCl_3$) δ –3.9, –3.6, 13.8, 18.1, 25.5, 47.3, 60.6, 100.6, 114.9, 123.5, 125.2, 127.2, 127.7, 127.9, 128.6, 129.05, 129.07, 129.6, 131.9, 132.0, 132.2, 135.8, 136.8, 147.7, 168.8; MS (ESI-TOF) *m*/*z* 523 $[M+Na]^+$; HRMS calcd for $C_{31}H_{36}NaO_4Si$ $[M+Na]^+$, 523.2281; found, 523.2211. Anal. Calcd for $C_{31}H_{36}O_4Si$: C, 74.36; H, 7.25. Found: 74.38; H, 7.26.

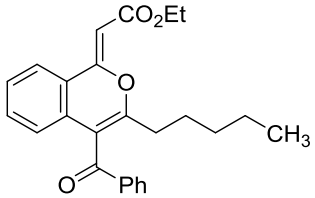
4. Fluoride-triggered ring rearrangement reaction

Ethyl 4-benzoyl-1-hydroxy-3-pentyl-2-naphthoate (9a)



To a mixture of MS4A powder (3.0 g) and TBAF (a 1.0 M solution in THF, 3.0 mL, 3.0 mmol) in THF (3.0 mL), a solution of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(hex-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-yl)-acetate **8a** (506 mg, 1.00 mmol) in THF (1.0 mL) was added at room temperature. After being stirred for 3 h at 70 °C, the obtained reaction mixture was quenched with saturated NH_4Cl aqueous solution (10 mL). This mixture was filtrated through celite pad. The resulting filtrate was diluted with water (25 mL) and extracted with EtOAc (20 mL x 3). The combined organic layer was washed with brine (10 mL), dried over anhydrous Na_2SO_4, and evaporated. Thus obtained residue was purified by column chromatography on silica gel (hexane/EtOAc = 20 : 1) to give naphthalene **9a** in 99.8% yield (390 mg, 1.00 mmol). Colorless crystals (from hexane/EtOAc); Mp. 90.5-92.0 °C; IR (ATR) ν 3065, 2961, 2950, 2870, 1660, 1644, 1578, 1407, 1325, 1239, 1221, 1160, 823, 765, 635 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.78 (3H, t, J = 6.8 Hz), 1.02-1.30 (4H, m), 1.40-1.54 (2H, br), 1.44 (3H, t, J = 7.1 Hz), 2.50-2.68 (1H, m), 2.90-3.16 (1H, m), 4.50 (2H, q, J = 7.1 Hz), 7.30-7.37 (1H, m), 7.42 (2H, t, J = 7.4 Hz), 7.42-7.50 (2H, m), 7.53 (1H, tt, J = 7.4, 1.3 Hz), 7.83 (2H, d, J = 7.4 Hz), 8.40-8.51 (1H, m), 12.86 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 14.1, 22.3, 31.6, 32.2, 33.6, 62.0, 105.6, 123.6, 124.4, 124.7, 125.5, 128.8, 128.9, 129.7, 130.2, 133.3, 133.7, 136.7, 138.1, 162.9, 172.4, 199.8; MS (ESI-TOF) *m*/*z* 391 $[M+H]^+$; HRMS calcd for $C_{25}H_{27}O_4$ $[M+H]^+$, 391.1909; found, 391.1901.

Ethyl (*Z*)-2-(4-benzoyl-3-pentyl-1*H*-isochromen-1-ylidene)acetate (10a)

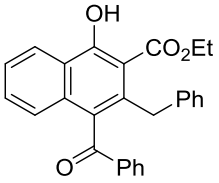


The reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(hex-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-yl)-acetate **8a** (120 mg, 0.238 mmol) with TBAF (a 1.0 M solution in THF, 0.24 mL, 0.24 mmol) in THF (2.0 mL) was conducted in the absence of MS4A. After usual extractive workup, chromatographic purification of the resulting residue gave naphthalene **9a** (53% yield, 49.1 mg, 0.126 mmol), lactone **7a** (24% yield, 17.0 mg, 56.2 μmol), and divinyl ether **10a** (15% yield, 14.3 mg, 36.6 μmol).

For **10a** Yellow oil; IR (neat) ν 2950, 2930, 2860, 1705, 1665, 1640, 1610, 1595, 1260, 1145, 1110, 1050 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.80 (3H, t, J = 6.8 Hz), 1.15-1.25 (4H, m), 1.33 (3H, t, J = 7.2 Hz), 1.69-1.82 (2H, m), 2.33 (2H, t, J = 7.6 Hz), 4.24 (2H, q, J = 7.2 Hz), 5.80 (1H, s), 6.92 (1H, dd, J = 7.2, 1.6 Hz), 7.27-7.35 (2H, m), 7.43-7.51 (2H, m), 7.62 (1H, t, J = 7.2 Hz), 7.73 (1H, dd, J = 7.6, 1.6 Hz), 7.94-7.99 (2H, m); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.9, 14.5, 22.2, 26.4, 31.2, 32.0, 59.5, 90.4, 114.0, 122.5, 124.0, 124.1,

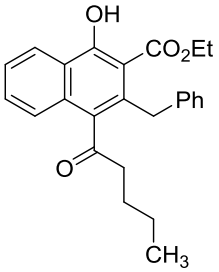
128.0, 129.0, 129.7, 130.2, 132.0, 134.1, 137.6, 155.2, 159.3, 165.4, 195.0; MS (ESI-TOF) *m*/*z* 391 $[M+H]^+$; HRMS calcd for $C_{25}H_{27}O_4$ $[M+H]^+$, 391.1909; found, 391.1920.

Ethyl 4-benzoyl-3-benzyl-1-hydroxy-2-naphthoate (9b)



According to the synthetic procedure for **9a**, this compound was obtained in 92% yield (377 mg, 0.918 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-(phenylethynyl)-1*H*-isochromen-1-yl)-acetate **8b** (524 mg, 0.999 mmol) with TBAF (a 1.0 M solution in THF, 3.0 mL, 3.0 mmol) in the presence of MS4A powder (3.0 g) in THF (4.0 mL) for 1 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 5 : 1). Colorless crystals (from hexane/EtOAc); Mp. 133-134 °C; IR (ATR) ν 2935, 1736, 1670, 1576, 1407, 1325, 1240, 1171, 1103, 835, 740, 631 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.10 (3H, t, *J* = 7.1 Hz), 4.05-4.28 (3H, m), 4.44 (1H, d, *J* = 16.0 Hz), 6.94 (2H, d, *J* = 7.0 Hz), 7.06 (1H, t, *J* = 7.3 Hz), 7.10-7.20 (2H, m), 7.30-7.47 (3H, m), 7.48-7.59 (3H, m), 7.82 (2H, d), 8.45-8.57 (1H, m), 12.81 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 38.8, 61.9, 106.2, 124.1, 124.4, 125.0, 125.6, 126.1, 127.8, 128.0, 128.7, 129.8, 130.3, 130.6, 132.9, 133.2, 133.8, 137.8, 140.7, 162.9, 171.8, 199.4; MS (ESI-TOF) *m*/*z* 411 $[M+H]^+$; HRMS calcd for $C_{27}H_{23}O_7$ $[M+H]^+$, 411.1596; found, 411.1610. Anal. Calcd for $C_{27}H_{22}O_7$: C, 79.01; H, 5.40. Found: C, 78.71; H, 5.58.

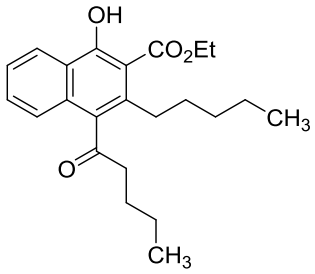
Ethyl 3-benzyl-1-hydroxy-4-pentanoyl-2-naphthoate (9c)



According to the synthetic procedure for **9a**, this compound was obtained in 92% yield (362 mg, 0.928 mmol) by the reaction of ethyl 2-(3-butyl-1-((*tert*-butyldimethylsilyl)oxy)-4-(phenylethynyl)-1*H*-isochromen-1-yl)-acetate **8c** (508 mg, 1.01 mmol) with TBAF (a 1.0 M solution in THF, 1.1 mL, 1.1 mmol) in THF (4.0 mL) for 3 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 5 : 1). Colorless crystals (from hexane/EtOAc); Mp. 68.0-69.5 °C; IR (ATR) ν 2955, 2871, 1692, 1635, 1406, 1328, 1163, 762, 737, 706, 636 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.83 (3H, t, *J* = 7.3 Hz), 1.06 (3H, t, *J* = 7.1 Hz), 1.28 (2H, sex, *J* = 7.3 Hz), 1.66 (2H, quint, *J* = 7.3 Hz), 2.60-2.82 (2H, br), 4.18 (2H, q, *J* = 7.1 Hz), 4.26-4.50 (2H, br), 6.99 (2H, d, *J* = 7.2 Hz), 7.13 (1H, t, *J* = 7.2 Hz), 7.22 (2H, t, *J* = 7.2 Hz), 7.47 (1H, d, *J* = 8.3 Hz), 7.50-7.60 (1H, m), 7.60-7.68 (1H, m), 8.49 (1H, d, *J* = 8.3 Hz), 12.80 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.6, 13.8, 22.1, 25.4, 38.2, 45.8, 61.9, 106.0, 124.0, 124.3, 124.6, 125.8, 126.0, 127.8 (2C), 128.3 (2C), 130.4, 130.6, 132.0, 133.8, 140.9, 162.7, 171.8, 210.2; MS (ESI-TOF) *m*/*z* 391 $[M+H]^+$; HRMS calcd for $C_{25}H_{27}O_4$

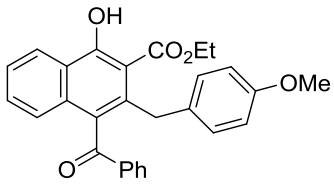
$[M+H]^+$, 391.1909; found, 391.1914.

Ethyl 1-hydroxy-4-pentanoyl-3-pentyl-2-naphthoate (9d)



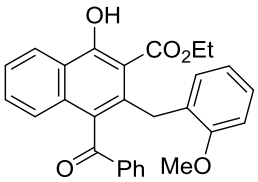
According to the synthetic procedure for **9a**, this compound was obtained in 97% yield (168 mg, 0.455 mmol) by the reaction of ethyl 2-(3-butyl-1-((*tert*-butyldimethylsilyl)oxy)-4-(hex-1-yn-1-yl)-1*H*-isochromen-1-yl)-acetate **8d** (227 mg, 0.467 mmol) with TBAF (a 1.0 M solution in THF, 1.4 mL, 1.4 mmol) in the presence of MS4A powder (1.4 g) in THF (3.5 mL) for 3 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 10 : 1). Colorless crystals (from hexane/EtOAc); Mp. 57.0-58.5 °C; IR (ATR) ν 2868, 1698, 1639, 1405, 1236, 1015, 833, 767 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.90 (3H, t, J = 7.1 Hz), 0.96 (3H, t, J = 7.3 Hz), 1.24-1.38 (4H, m), 1.40-1.60 (4H, m), 1.46 (3H, t, J = 7.1 Hz), 1.78 (2H, quint, J = 7.4 Hz), 2.50-3.24 (2H, br), 2.83 (2H, t, J = 7.4 Hz), 4.50 (2H, q, J = 7.1 Hz), 7.36-7.44 (1H, m), 7.48 (1H, td, J = 6.9, 1.1 Hz), 7.58 (1H, td, J = 6.9, 1.3 Hz), 8.37-8.49 (1H, m), 12.76 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.98, 14.07, 14.12, 22.3, 22.6, 25.6, 32.2, 32.4, 33.5, 46.0, 62.0, 105.3, 123.6, 123.7, 124.6, 125.5, 130.3, 132.0 (2C), 134.7, 162.6, 172.3, 210.2; MS (ESI-TOF) m/z 393 $[M+Na]^+$; HRMS calcd for $C_{23}H_{30}NaO_4$ $[M+Na]^+$, 393.2042; found, 393.2049.

Ethyl 4-benzoyl-1-hydroxy-3-(4-methoxybenzyl)-2-naphthoate (9e)



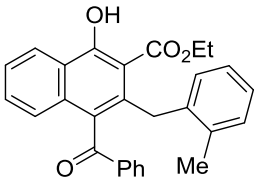
According to the synthetic procedure for **9a**, this compound was obtained in 91% yield (169 mg, 0.384 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-((4-methoxyphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-yl)acetate **8e** (233 mg, 0.420 mmol) with TBAF (a 1.0 M solution in THF, 1.3 mL, 1.3 mmol) in the presence of MS4A powder (1.3 g) in THF (2.5 mL) for 1 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 5 : 1). Colorless crystals (from hexane/EtOAc); Mp. 131-132 °C; IR (ATR) ν 2992, 2832, 1669, 1635, 1511, 1439, 1370, 1237, 820, 766, 630 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.17 (3H, t, J = 7.2 Hz), 3.71 (3H, s), 4.08 (1H, d, J = 14.8 Hz), 4.16-4.30 (2H, m), 4.39 (1H, d, J = 14.8 Hz), 6.69 (2H, d, J = 8.8 Hz), 6.86 (2H, d, J = 8.8 Hz), 7.34-7.44 (3H, m), 7.47-7.57 (3H, m), 7.83 (2H, d), 8.46-8.54 (1H, m), 12.73 (1H, s); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 37.8, 55.2, 61.9, 106.2, 113.5, 124.1, 124.4, 125.0, 126.0, 128.7, 128.8, 129.8, 130.3, 130.4, 132.8, 133.2, 133.5, 133.8, 137.8, 157.6, 162.7, 171.8, 199.4; MS (ESI-TOF) m/z 463 $[M+Na]^+$; HRMS calcd for $C_{28}H_{24}NaO_5$ $[M+Na]^+$, 463.1521; found, 463.1514. Anal. Calcd for $C_{28}H_{24}O_5$: C, 76.35; H, 5.49. Found: C, 76.05; H, 5.61.

Ethyl 4-benzoyl-1-hydroxy-3-(2-methoxybenzyl)-2-naphthoate (9f)



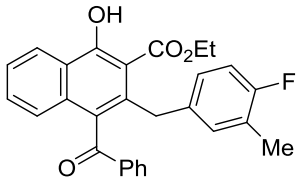
According to the synthetic procedure for **9a**, this compound was obtained in 91% yield (161 mg, 0.366 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-((2-methoxyphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-yl)acetate **8f** (222 mg, 0.400 mmol) with TBAF (a 1.0 M solution in THF, 1.2 mL, 1.2 mmol) in the presence of MS4A powder (1.2 g) in THF (2.5 mL) for 1 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 9 : 1). Colorless crystals (from hexane/EtOAc); Mp. 192-193 °C; IR (ATR) ν 2933, 1666, 1638, 1578, 1404, 1239, 754, 628 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.98 (3H, t, J = 6.8 Hz), 3.75 (3H, s), 3.93 (1H, brd, J = 17.0 Hz), 4.13 (2H, q, J = 6.8 Hz), 4.50 (1H, brd, J = 17.0 Hz), 6.67-6.76 (3H, m), 7.04-7.10 (1H, m), 7.36 (2H, t, J = 7.2 Hz), 7.39-7.42 (1H, m), 7.47-7.57 (3H, m), 7.83 (2H, d, J = 7.2 Hz), 8.50-8.56 (1H, m), 13.0 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.1, 32.9, 54.8, 61.7, 106.3, 109.0, 120.3, 124.1, 124.4, 124.9, 125.9, 126.6, 128.0, 128.6, 129.5, 129.8, 130.2, 130.8, 133.1, 133.2, 133.7, 137.7, 156.2, 162.9, 172.0, 199.2; MS (ESI-TOF) m/z 463 $[M+Na]^+$; HRMS calcd for $C_{28}H_{24}NaO_5$ $[M+Na]^+$, 463.1521; found, 463.1523.

Ethyl 4-benzoyl-1-hydroxy-3-(2-methylbenzyl)-2-naphthoate (9g)



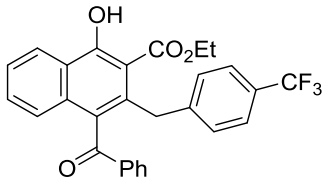
According to the synthetic procedure for **9a**, this compound was obtained in 89% yield (135 mg, 0.318 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-(*o*-tolylethynyl)-1*H*-isochromen-1-yl)-acetate **8g** (193 mg, 0.358 mmol) with TBAF (a 1.0 M solution in THF, 1.1 mL, 1.1 mmol) in the presence of MS4A powder (1.1 g) in THF (2.5 mL) for 1 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1~10 : 1). Colorless crystals (from hexane/EtOAc); Mp. 165-168 °C; IR (ATR) ν 2971, 1671, 1634, 1595, 1463, 1292, 1240, 841, 757, 631 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.87 (3H, t, J = 7.2 Hz), 2.17 (3H, s), 4.04 (1H, brd, J = 16.2 Hz), 4.08-4.20 (2H, m), 4.32 (1H, brd, J = 16.2 Hz), 6.68 (1H, d, J = 7.0 Hz), 6.92-7.00 (2H, m), 7.01-7.08 (1H, m), 7.35 (2H, t, J = 8.2 Hz), 7.42 (1H, dd, J = 7.0, 1.6 Hz), 7.47-7.58 (3H, m), 7.83 (2H, d, J = 7.2 Hz), 8.52-8.59 (1H, m), 13.09 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.2, 19.6, 36.4, 61.8, 106.3, 124.1, 124.4, 124.9, 125.5, 125.8, 126.0, 126.7, 128.6, 129.2, 129.7, 130.3, 130.7, 132.8, 133.3, 133.7, 134.8, 137.6, 139.2, 163.1, 171.9, 199.3; MS (ESI-TOF) m/z 425 $[M+H]^+$; HRMS calcd for $C_{28}H_{25}O_4$ $[M+H]^+$, 425.1753; found, 425.1758.

Ethyl 4-benzoyl-3-(4-fluoro-3-methylbenzyl)-1-hydroxy-2-naphthoate (9h)



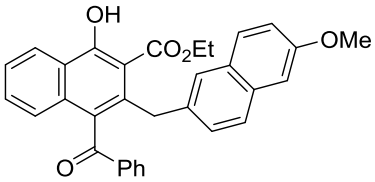
According to the synthetic procedure for **9a**, this compound was obtained in 94% yield (181 mg, 0.409 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-((4-fluoro-3-methylphenyl)ethynyl)-3-phenyl-1*H*-isochromen-1-yl)acetate **8h** (243 mg, 0.436 mmol) with TBAF (a 1.0 M solution in THF, 1.3 mL, 1.3 mmol) in the presence of MS4A powder (1.3 g) in THF (2.5 mL) for 1 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1). Colorless crystals (from hexane/EtOAc); Mp. 134-136 °C; IR (ATR) ν 2927, 1661, 1641, 1496, 1406, 1236, 1162, 767, 627 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.16 (3H, t, J = 7.2 Hz), 2.14 (3H, d, J_{HF} = 0.8 Hz), 4.11 (1H, brd, J = 16.0 Hz), 4.18-4.32 (2H, m), 4.42 (1H, brd, J = 16.0 Hz), 6.72-6.83 (3H, m), 7.37 (2H, t, J = 7.7 Hz), 7.40-7.46 (1H, m), 7.48-7.59 (3H, m), 7.84 (2H, d, J = 7.7 Hz), 8.49-8.56 (1H, m), 12.86 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 14.4 (d, J_{CF} = 4.0 Hz), 37.8, 61.9, 106.0, 114.3 (d, J_{CF} = 22.1 Hz), 123.9, 124.0, 124.4, 124.9, 126.0, 126.5 (d, J_{CF} = 8.0 Hz), 128.6, 129.7, 130.3, 130.4, 130.8, 133.0 (d, J_{CF} = 25.2 Hz), 133.8, 135.85, 135.89, 137.6, 159.4 (d, J_{CF} = 242 Hz), 162.9, 171.7, 199.3; ^{19}F NMR (376 MHz, $CDCl_3$) δ –59.6 (1F, brs); MS (ESI-TOF) *m*/*z* 443 $[M+H]^+$; HRMS calcd for $C_{28}H_{24}FO_4$ $[M+H]^+$, 443.1659; found, 443.1670.

Ethyl 4-benzoyl-1-hydroxy-3-(4-(trifluoromethyl)benzyl)-2-naphthoate (9i)



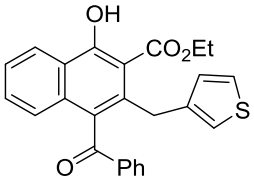
According to the synthetic procedure for **9a**, this compound was obtained in 84% yield (167 mg, 0.348 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-((4-(trifluoromethyl)phenyl)ethynyl)-1*H*-isochromen-1-yl)acetate **8i** (248 mg, 0.418 mmol) with TBAF (a 1.0 M solution in THF, 1.3 mL, 1.3 mmol) in the presence of MS4A powder (1.3 g) in THF (3.0 mL) for 3 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1). Colorless crystals (from EtOAc); Mp. 113-115 °C; IR (ATR) ν 2981, 1667, 1643, 1466, 1320, 1108, 867, 613 cm^{-1}; ^{1}H NMR (400 MHz, $CDCl_3$) δ 1.07 (3H, t, J = 7.1 Hz), 4.13-4.29 (3H, m), 4.53 (1H, d, J = 16.5 Hz), 7.09 (2H, t, J = 7.9 Hz), 7.35-7.44 (5H, m), 7.50-7.59 (3H, m), 7.80 (2H, t, J = 7.9 Hz), 8.50-8.57 (1H, m), 12.96 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 38.6, 62.0, 105.8, 124.23, 124.24 (q, J_{CF} = 267 Hz), 124.5, 124.9 (q, J_{CF} = 4.0 Hz), 125.0, 126.3, 128.0 (q, J_{CF} = 32.2 Hz), 128.1, 128.8, 129.7, 130.6, 130.8, 131.6, 133.2, 134.0, 137.5, 144.9, 163.3, 171.6, 199.2; ^{19}F NMR (376 MHz, $CDCl_3$) δ –0.4 (3F, s); MS (ESI-TOF) *m*/*z* 479 $[M+H]^+$; HRMS calcd for $C_{28}H_{22}F_3O_4$ $[M+H]^+$, 479.1470; found, 479.1460.

Ethyl 4-benzoyl-1-hydroxy-3-((6-methoxynaphthalen-2-yl)methyl)-2-naphthoate (9j)



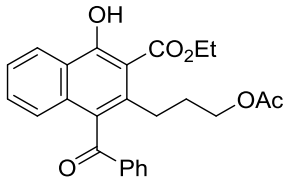
According to the synthetic procedure for **9a**, this compound was obtained in 95% yield (184 mg, 0.375 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-((6-methoxynaphthalen-2-yl)ethynyl)-3-phenyl-1*H*-isochromen-1-yl)acetate **8j** (240 mg, 0.397 mmol) with TBAF (a 1.0 M solution in THF, 1.2 mL, 1.2 mmol) in the presence of MS4A powder (1.2 g) in THF (2.5 mL) for 1 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 20 : 1~10 : 1). Colorless crystals (from hexane/EtOAc); Mp. 189-191 °C; IR (ATR) ν 3044, 2967, 1734, 1602, 1250, 1023, 851, 765, 692 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.07 (3H, t, J = 7.2 Hz), 3.88 (3H, s), 4.09-4.23 (2H, m), 4.29 (1H, d, J = 16.2 Hz), 4.56 (1H, d, J = 16.2 Hz), 6.99-7.06 (2H, m), 7.11 (1H, dd, J = 8.5, 1.7 Hz), 7.22 (1H, brs), 7.34 (2H, t, J = 8.5 Hz), 7.41-7.47 (1H, m), 7.48-7.58 (5H, m), 7.81-7.87 (2H, m), 8.51-8.56 (1H, m), 12.82 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.8, 38.7, 55.2, 61.9, 105.4, 106.2, 118.4, 124.2, 124.4, 125.0, 125.8, 126.1, 126.4, 127.3, 128.7, 128.9, 129.0, 129.7, 130.3, 130.6, 132.8, 133.1, 133.3, 133.8, 136.0, 137.7, 157.1, 162.9, 171.8, 199.4; MS (ESI-TOF) *m*/*z* 491 $[M+H]^+$; HRMS calcd for $C_{32}H_{27}O_5$ $[M+H]^+$, 491.1858; found, 491.1849.

Ethyl 4-benzoyl-1-hydroxy-3-(thiophen-3-ylmethyl)-2-naphthoate (9k)



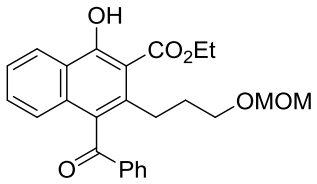
According to the synthetic procedure for **9a**, this compound was obtained in 82% yield (161 mg, 0.387 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-(thiophen-2-ylethynyl)-1*H*-isochromen-1-yl)acetate **8k** (251 mg, 0.473 mmol) with TBAF (a 1.0 M solution in THF, 1.4 mL, 1.4 mmol) in the presence of MS4A powder (1.4 g) in THF (2.5 mL) for 1.5 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 10 : 1). Colorless crystals (from hexane/EtOAc); Mp. 175-177 °C; IR (ATR) ν 2978, 1672, 1635, 1593, 1327, 1240, 766, 628 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.19 (3H, t, J = 7.2 Hz), 4.10 (1H, brd, J = 16.0 Hz), 4.28 (2H, q, J = 7.2 Hz), 4.43 (1H, brd, J = 16.0 Hz), 6.63 (1H, dd, J = 3.0, 0.9 Hz), 6.76 (1H, dd, J = 4.9, 0.9 Hz), 7.10 (1H, dd, J = 4.9, 3.0 Hz), 7.23-7.42 (3H, m), 7.47-7.58 (3H, m), 7.83 (2H, d, J = 7.4 Hz), 8.47-8.53 (1H, m), 12.78 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 13.7, 34.0, 62.0, 105.9, 120.5, 124.0, 124.4, 124.8, 125.0, 126.0, 127.9, 128.7, 129.7, 129.9, 130.3, 133.20, 133.22, 133.8, 137.6, 140.9, 162.7, 171.8, 199.3; MS (ESI-TOF) *m*/*z* 417 $[M+H]^+$; HRMS calcd for $C_{25}H_{21}O_4S$ $[M+H]^+$, 417.1161; found, 417.1159.

Ethyl 3-(3-acetoxypropyl)-4-benzoyl-1-hydroxy-2-naphthoate (9m)



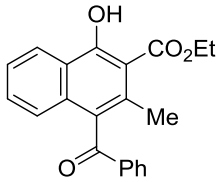
According to the synthetic procedure for **9a**, this compound was obtained in 86% yield (334 mg, 0.795 mmol) by the reaction of ethyl 2-(4-(4-acetoxybut-1-yn-1-yl)-1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-1*H*-isochromen-1-yl)acetate **8m** (492 mg, 0.920 mmol) with TBAF (a 1.0 M solution in THF, 2.8 mL, 2.8 mmol) in the presence of MS4A powder (2.8 g) in THF (5.0 mL) for 2 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 5 : 1). Colorless crystals (from hexane/EtOAc); Mp. 99.0-99.5 °C; IR (ATR) ν 3063, 2967, 1726, 1647, 1410, 1238, 1023, 770 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.46 (3H, t, J = 7.1 Hz), 1.81-1.97 (2H, m), 1.95 (3H, s), 2.61-2.72 (1H, m), 3.06-3.18 (1H, m), 3.90-4.00 (2H, m), 4.52 (2H, q, J = 7.1 Hz), 7.31-7.36 (1H, m), 7.43 (2H, t, J = 7.4 Hz), 7.45-7.51 (2H, m), 7.59 (1H, tt, J = 7.4, 1.2 Hz), 7.82 (2H, d, J = 7.4 Hz), 8.43-8.50 (1H, m), 12.94 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.1, 20.9, 30.2, 30.6, 62.2, 64.2, 105.4, 123.8, 124.4, 124.7, 125.8, 125.9, 129.3, 129.7, 130.4, 133.2, 133.9, 135.0, 137.8, 163.2, 171.0, 172.1, 199.5; MS (ESI-TOF) *m*/*z* 443 $[M+Na]^+$; HRMS calcd for $C_{25}H_{24}NaO_6$ $[M+Na]^+$, 443.1471; found, 443.1474.

Ethyl 4-benzoyl-1-hydroxy-3-(3-(methoxymethoxy)propyl)-2-naphthoate (9n)



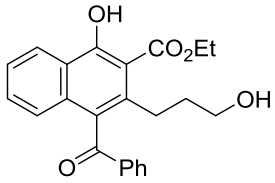
According to the synthetic procedure for **9a**, this compound was obtained in 93% yield (175 mg, 0.415 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(4-(methoxymethoxy)but-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-yl)acetate **8n** (239 mg, 0.445 mmol) with TBAF (a 1.0 M solution in THF, 1.3 mL, 1.3 mmol) in the presence of MS4A powder (1.3 g) in THF (2.5 mL) for 2 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 5 : 1). Pale yellow oil; IR (neat) ν 3060, 2947, 1672, 1651, 1580, 1406, 1327, 1240, 1109, 1040 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.46 (3H, t, J = 7.1 Hz), 1.72-1.90 (2H, m), 2.62-2.80 (1H, m), 3.08-3.21 (1H, m), 3.26 (3H, s), 3.37-3.45 (2H, m), 4.50 (2H, s), 4.51 (1H, d, J = 14.5 Hz), 4.52 (1H, d, J = 14.5 Hz), 7.30-7.37 (1H, m), 7.42 (2H, t, J = 7.8 Hz), 7.44-7.50 (2H, m), 7.57 (1H, t, J = 7.4 Hz), 7.83 (2H, d, J = 7.2 Hz), 8.44-8.49 (1H, m), 12.89 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 30.4, 31.7, 55.0, 62.2, 67.4, 96.2, 105.6, 123.7, 124.4, 124.7, 125.7, 128.8, 129.2, 129.7, 130.3, 133.2, 133.8, 135.8, 138.0, 163.0, 172.3, 199.7; MS (ESI-TOF) *m*/*z* 423 $[M+H]^+$; HRMS calcd for $C_{25}H_{27}O_6$ $[M+H]^+$, 423.1808; found, 423.1815. Anal. Calcd for $C_{25}H_{26}O_6$: C, 71.07; H, 6.20. Found: C, 70.99; H, 6.21.

Ethyl 4-benzoyl-1-hydroxy-3-methyl-2-naphthoate (9o)



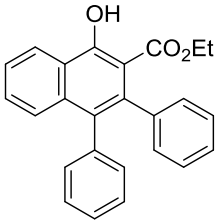
According to the synthetic procedure for **9a**, this compound was obtained in 91% yield (156 mg, 0.467 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3-phenyl-4-((trimethylsilyl)ethynyl)-1*H*-isochromen-1-yl)acetate **8o** (267 mg, 0.512 mmol) with TBAF (a 1.0 M solution in THF, 1.5 mL, 1.5 mmol) in the presence of MS4A powder (1.5 g) in THF (2.5 mL) for 1.5 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 15 : 1~10 : 1). Colorless crystals (from hexane/EtOAc); Mp. 126-127 °C; IR (ATR) ν 3064, 2980, 1665, 1645, 1578, 1408, 1326, 1245, 1221, 1173, 1157, 849, 770, 624 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.43 (3H, t, J = 7.1 Hz), 2.45 (3H, s), 4.49 (2H, q, J = 7.1 Hz), 7.32-7.38 (1H, m), 7.45 (2H, t, J = 8.0 Hz), 7.40-7.50 (2H, m), 7.54-7.62 (1H, m), 7.85 (2H, dd, J = 7.1, 1.2 Hz), 8.42-8.50 (1H, m), 12.9 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.2, 21.1, 62.0, 106.2, 123.5, 124.3, 124.5, 125.5, 128.9, 129.0, 129.7, 130.3, 131.8, 133.2, 133.8, 137.8, 162.7, 172.5, 200.2; MS (ESI-TOF) *m*/*z* 335 $[M+H]^+$; HRMS calcd for $C_{21}H_{19}O_4$ $[M+H]^+$, 335.1283; found, 335.1284.

Ethyl 4-benzoyl-1-hydroxy-3-(3-hydroxypropyl)-2-naphthoate (9p)



According to the synthetic procedure for **9a**, this compound was obtained in 97% yield (102 mg, 0.270 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(4-((*tert*-butyldimethylsilyl)oxy)but-1-yn-1-yl)-3-phenyl-1*H*-isochromen-1-yl)acetate **8p** (169 mg, 0.278 mmol) with TBAF (a 1.0 M solution in THF, 0.83 mL, 0.83 mmol) in the presence of MS4A powder (0.83 g) in THF (2.0 mL) for 1 h at 70 °C and the following column chromatography on silica gel (hexane/EtOAc = 2 : 1). Pale yellow crystals (from hexane/EtOAc); Mp. 92.0-93.5 °C; IR (ATR) ν 3566, 2955, 1657, 1641, 1576, 1407, 1238, 765, 629 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.44 (3H, t, J = 7.1 Hz), 1.70-1.90 (2H, m), 1.93-2.12 (1H, m, O*H*), 2.58-2.73 (1H, m), 3.20-3.36 (1H, m), 3.44-3.55 (2H, m), 4.43-4.59 (2H, m), 7.34-7.38 (1H, m), 7.39-7.50 (4H, m), 7.58 (1H, t, J = 7.4 Hz), 7.84 (2H, d, J = 7.2 Hz), 8.42-8.51 (1H, m), 12.9 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.0, 29.7, 34.6, 61.9, 62.2, 105.4, 123.7, 124.4, 124.7, 125.7, 128.8, 129.1, 129.8, 130.1, 134.0, 135.6, 137.8, 163.0, 172.1, 200.7; MS (ESI-TOF) *m*/*z* 401 $[M+H]^+$; HRMS calcd for $C_{23}H_{22}NaO_5$ $[M+Na]^+$, 401.1365; found, 401.13665.

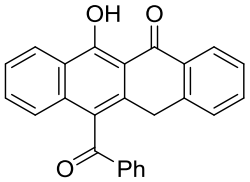
Ethyl 1-hydroxy-3,4-diphenyl-2-naphthoate



According to the synthetic procedure for **9a**, this compound was obtained in 80% yield (148 mg, 0.401 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-3,4-diphenyl-1*H*-isochromen-1-yl)acetate (250 mg, 0.499 mmol) with TBAF (a 1.0 M solution in THF, 0.50 mL, 0.50 mmol) in THF (2.5 mL) for 7 h at room temperature and the following column chromatography on silica gel (hexane/EtOAc = 30 : 1). Colorless crystals (from hexane/EtOAc); Mp. 157-158 °C; IR (ATR) ν 3053, 3022, 2986, 1638, 1398, 1320, 1209, 1097, 745, 697 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 0.66 (3H, t, J = 7.2 Hz), 3.94 (2H, q, J = 7.2 Hz), 6.94-7.11 (7H, m), 7.13-7.22 (3H, m), 7.38 (1H, dd, J = 8.3, 1.2 Hz), 7.49 (1H, td, J = 7.0, 1.2 Hz), 7.51-7.57 (1H, m), 8.53 (1H, brd, J = 7.0 Hz), 12.38 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 12.9, 60.9, 106.5, 123.8, 1241, 125.60, 125.61, 126.3, 126.67, 126.70, 127.5, 1298.6, 129.8, 131.2, 131.5, 135.5, 137.2, 138.7, 142.0, 160.5, 172.1; MS (ESI-TOF) *m*/*z* 369 $[M+H]^+$; HRMS calcd for $C_{25}H_{21}O_3$ $[M+H]^+$, 369.1491; found, 369.1472.

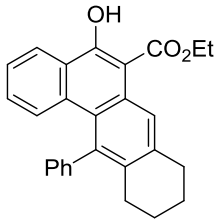
5. Construction of higher polycyclic systems

11-Benzoyl-6-hydroxytetracen-5(12*H*)-one (11)



Naphthol **9b** (103 mg, 0.251 mmol) was dissolved in TfOH (1.0 mL). After being stirred for 2 h at room temperature, the reaction mixture was diluted with CH_2Cl_2 (20 mL). The resulting mixture was poured into water (50 mL), and extracted with EtOAc (20 mL x 3). The combined organic layer was dried over anhydrous Na_2SO_4 and evaporated. The yellow residue was quickly purified by column chromatography on silica gel (hexane/EtOAc = 10 : 1) to give tetracenone **11** in 86% yield (79.0 mg, 0.213 mmol). Yellow crystals (from EtOAc); Mp. 178-181 °C; IR (ATR) ν 3057, 2923, 1734, 1618, 1575, 1247, 739, 614 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 4.26 (2H, brs), 7.28 (1H, d, J = 7.4 Hz), 7.40-7.49 (4H, m), 7.50-7.58 (3H, m), 7.62 (1H, tt, J = 7.4, 0.3 Hz), 7.89 (1H, d, J = 7.4 Hz), 8.35 (2H, dd, J = 7.9, 1.1 Hz), 8.55-8.60 (1H, m), 15.2 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 30.4, 109.7, 123.3, 124.5, 125.8, 125.5, 125.8, 127.1, 127.2, 128.4, 129.1, 129.8, 130.6, 130.9, 132.1, 133.8, 134.17, 134.24, 137.2, 140.3, 164.4, 189.8, 199.3; MS (ESI-TOF) *m*/*z* 365 $[M+H]^+$; HRMS calcd for $C_{25}H_{17}O_3$ $[M+H]^+$, 365.1178; found, 365.1182.

Ethyl 5-hydroxy-12-phenyl-8,9,10,11-tetrahydrotetraphene-6-carboxylate (12)

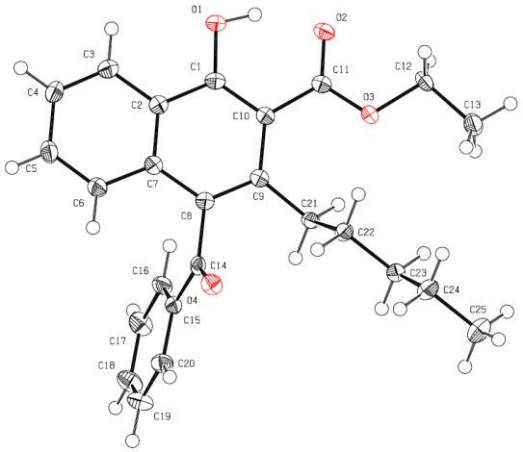


According to the synthetic procedure for **9a**, this compound was obtained in 67% yield (66.6 mg, 0.168 mmol) by the reaction of ethyl 2-(1-((*tert*-butyldimethylsilyl)oxy)-4-(cyclohex-1-en-1-ylethynyl)-3-phenyl-1*H*-isochromen-1-yl)acetate **8q** (132 mg, 0.250 mmol) with TBAF (a 1.0 M solution in THF, 0.75 mL, 0.75 mmol) in the presence of MS4A powder (0.75 g) in THF (2.5 mL) for 12 h at 70 °C and the following column chromatography on silica gel (hexane/CH_2Cl_2 = 4 : 1). Yellow crystals (from Et_2O); Mp. 172-180 °C; IR (ATR) ν 3061, 2923, 2851, 1640, 1308, 1249, 805, 693 cm^{-1}; ^1H NMR (400 MHz, $CDCl_3$) δ 1.56 (3H, t, J = 7.1 Hz), 1.71-1.79 (2H, m), 1.80-1.88 (2H, m), 2.46 (2H, t, J = 6.4 Hz), 3.04 (2H, t, J = 6.4 Hz), 4.61 (2H, q, J = 7.1 Hz), 7.09 (1H, dd, J = 7.2, 1.4 Hz), 7.19-7.24 (2H, m), 7.33-7.40 (2H, m), 7.41-7.52 (3H, m), 8.46 (1H, brd, J = 7.9 Hz), 8.52 (1H, s), 12.81 (1H, s, O*H*); ^{13}C NMR (100 MHz, $CDCl_3$) δ 14.3, 22.8, 23.7, 28.9, 30.9, 60.9, 101.7, 123.6, 124.1, 125.5, 125.7, 126.2, 126.9, 128.1, 128.2, 128.4, 129.3, 129.9, 133.2, 134.1, 136.7, 169.1, 143.7, 161.1, 172.6; MS (ESI-TOF) *m*/*z* 397 $[M+H]^+$; HRMS calcd for $C_{27}H_{25}O_3$ $[M+H]^+$, 397.1804; found, 397.1809.

6. X-ray crystallographic data

X-ray crystallographic data of **9a**, **9b**, **11**, and **12** have been deposited with Cambridge Crystallographic Data Center (CCDC) as supplementary publication Nos. CCDC 1469784 (**9a**), 1469783 (**9b**), 1469782 (**11**), and 1473607 (**12**). These data can be obtained free of charge from the CCDC *via* www.ccdc.cam.ac.uk/data_request/cif.

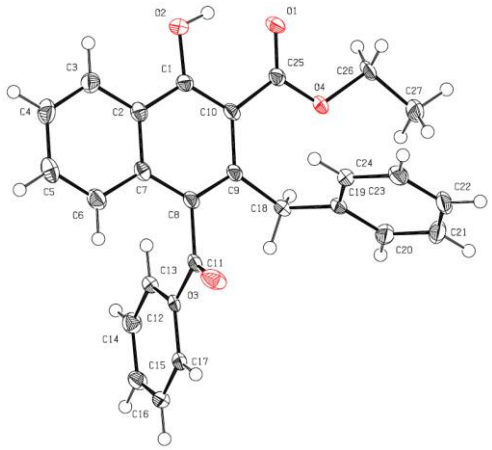
Table S1. Crystallographic data of **9a**



$C_{25}H_{26}O_4$	$F(000)$ = 832
M_r = 390.46	D_x = 1.216 Mg m^{-3}
Monoclinic, $P2_1/n$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -P 2yn	Cell parameters from 4024 reflections
a = 14.1779 (17) Å	θ = 2.8–27.6°
b = 7.8527 (9) Å	μ = 0.08 mm^{-1}
c = 19.280 (2) Å	T = 90 K
β = 96.4640 (16)°	Block, colourless
V = 2132.9 (4) Å^3	0.26 × 0.14 × 0.11 mm
Z = 4	
Bruker APEXII CCD area detector diffractometer	3758 independent reflections
Radiation source: Bruker TXS fine-focus rotating anode	3224 reflections with $I > 2\sigma(I)$
Bruker Helios multilayer confocal mirror	R_{int} = 0.021
Detector resolution: 8.333 pixels mm^{-1}	θ_{max} = 25.0°, θ_{min} = 2.1°
phi and ω scans	h = -12→16
Absorption correction: analytical	k = -9→8
Crystal Faces plugin in Bruker *APEX2* software	
T_{min} = 0.979, T_{max} = 0.991	l = -22→19
10003 measured reflections	
Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map

$R[F^2 > 2\sigma(F^2)] = 0.035$	Hydrogen site location: inferred from neighbouring sites
$wR(F^2) = 0.096$	H atoms treated by a mixture of independent and constrained refinement
$S = 1.04$	$w = 1/[\sigma^2(F_o^2) + (0.0514P)^2 + 0.5033P]$ where $P = (F_o^2 + 2F_c^2)/3$
3758 reflections	$(\Delta/\sigma)_{max} < 0.001$
267 parameters	$\Delta\rangle_{max} = 0.19$ e Å^{-3}
0 restraints	$\Delta\rangle_{min} = -0.21$ e Å^{-3}

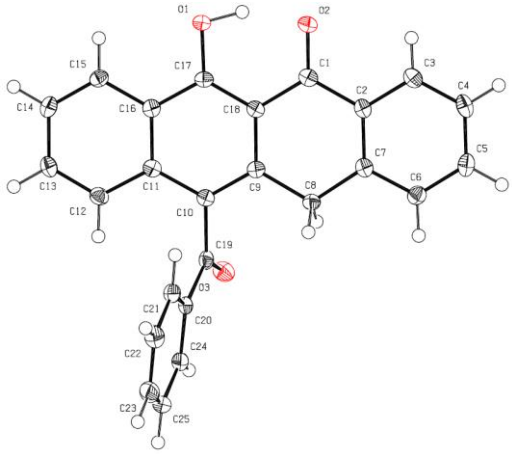
Table S2. Crystallographic data of **9b**



$C_{27}H_{22}O_4$	$Z = 2$
$M_r = 410.45$	$F(000) = 432$
Triclinic, $P\bar{1}$	$D_x = 1.319$ Mg m^{-3}
Hall symbol: -P 1	Mo $K\alpha$ radiation, $\lambda = 0.71073$ Å
$a = 9.173$ (2) Å	Cell parameters from 4575 reflections
$b = 10.382$ (3) Å	$\theta = 2.3–27.5°$
$c = 12.748$ (3) Å	$\mu = 0.09$ mm^{-1}
$\alpha = 104.062$ (3)°	$T = 90$ K
$\beta = 94.660$ (3)°	Block, colorless
$\gamma = 116.023$ (2)°	0.28 × 0.16 × 0.15 mm
$V = 1033.4$ (4) Å^3	
Bruker APEXII CCD area detector diffractometer	3635 independent reflections
Radiation source: Bruker TXS fine-focus rotating anode	3133 reflections with $I > 2\sigma(I)$
Bruker Helios multilayer confocal mirror	$R_{int} = 0.043$
Detector resolution: 8.333 pixels mm^{-1}	$\theta_{max} = 25.0°$, $\theta_{min} = 2.3°$
phi and ω scans	$h = -10\rightarrow10$
Absorption correction: analytical	$k = -12\rightarrow12$
Crystal Faces plugin in Bruker *APEX2* software	

T_{min} = 0.976, T_{max} = 0.987	l = -15→15
10078 measured reflections	
Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.047	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.137	H-atom parameters constrained
S = 1.05	$w = 1/[\sigma^2(F_o^2) + (0.0829P)^2 + 0.2205P]$
	where $P = (F_o^2 + 2F_c^2)/3$
3635 reflections	$(\Delta/\sigma)_{max} < 0.001$
282 parameters	$\Delta\rangle_{max}$ = 0.26 e Å^{-3}
204 restraints	$\Delta\rangle_{min}$ = -0.31 e Å^{-3}

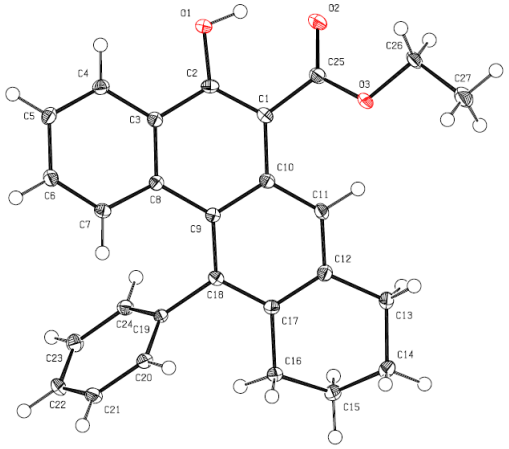
Table S3. Crystallographic data of **11**



$C_{25}H_{16}O_3$	F(000) = 1520
M_r = 364.38	D_x = 1.398 Mg m^{-3}
Monoclinic, $C2/c$	Mo $K\alpha$ radiation, λ = 0.71073 Å
Hall symbol: -C 2yc	Cell parameters from 2632 reflections
a = 25.507 (4) Å	θ = 2.5–27.5°
b = 8.1552 (14) Å	μ = 0.09 mm^{-1}
c = 16.661 (3) Å	T = 90 K
β = 92.869 (2)°	Plate, pale yellow
V = 3461.4 (10) Å^3	0.21 × 0.15 × 0.05 mm
Z = 8	
Bruker APEXII CCD area detector diffractometer	3046 independent reflections
Radiation source: Bruker TXS fine-focus rotating anode	2504 reflections with $I > 2\sigma(I)$
Bruker Helios multilayer confocal mirror	R_{int} = 0.030
Detector resolution: 8.333 pixels mm^{-1}	θ_{max} = 25.0°, θ_{min} = 2.5°
phi and ω scans	h = -28→30

Absorption correction: analytical	k = -9→5
Crystal Faces plugin in Bruker *APEX2* software	
T_{min} = 0.981, T_{max} = 0.996	l = -19→18
8153 measured reflections	
Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.036	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.093	H atoms treated by a mixture of independent and constrained refinement
S = 1.04	$w = 1/[\sigma^2(F_o^2) + (0.0404P)^2 + 1.9447P]$ where $P = (F_o^2 + 2F_c^2)/3$
3046 reflections	$(\Delta/\sigma)_{max} < 0.001$
256 parameters	$\Delta\rangle_{max}$ = 0.20 e Å^{-3}
0 restraints	$\Delta\rangle_{min}$ = -0.18 e Å^{-3}

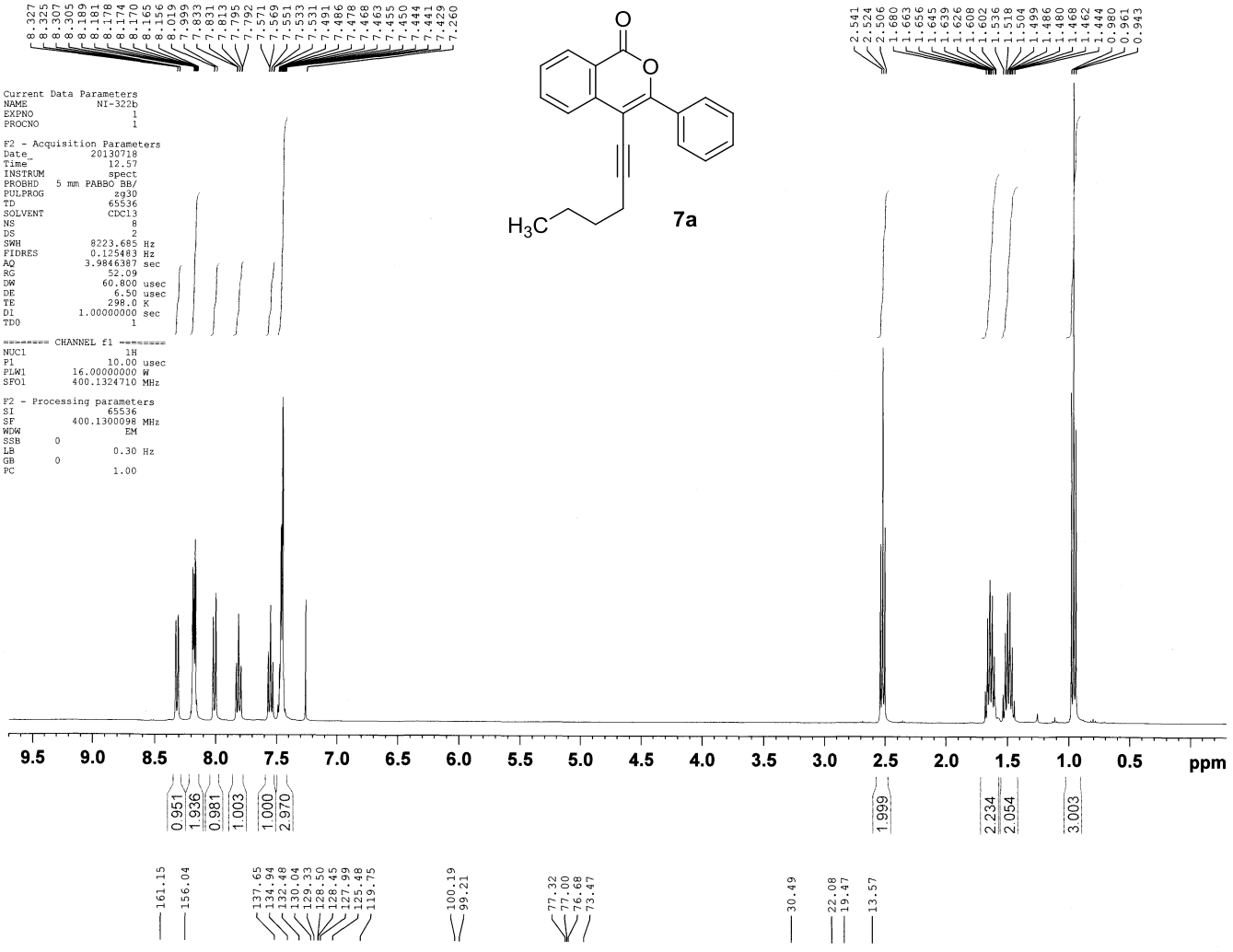
Table S4. Crystallographic data of **12**

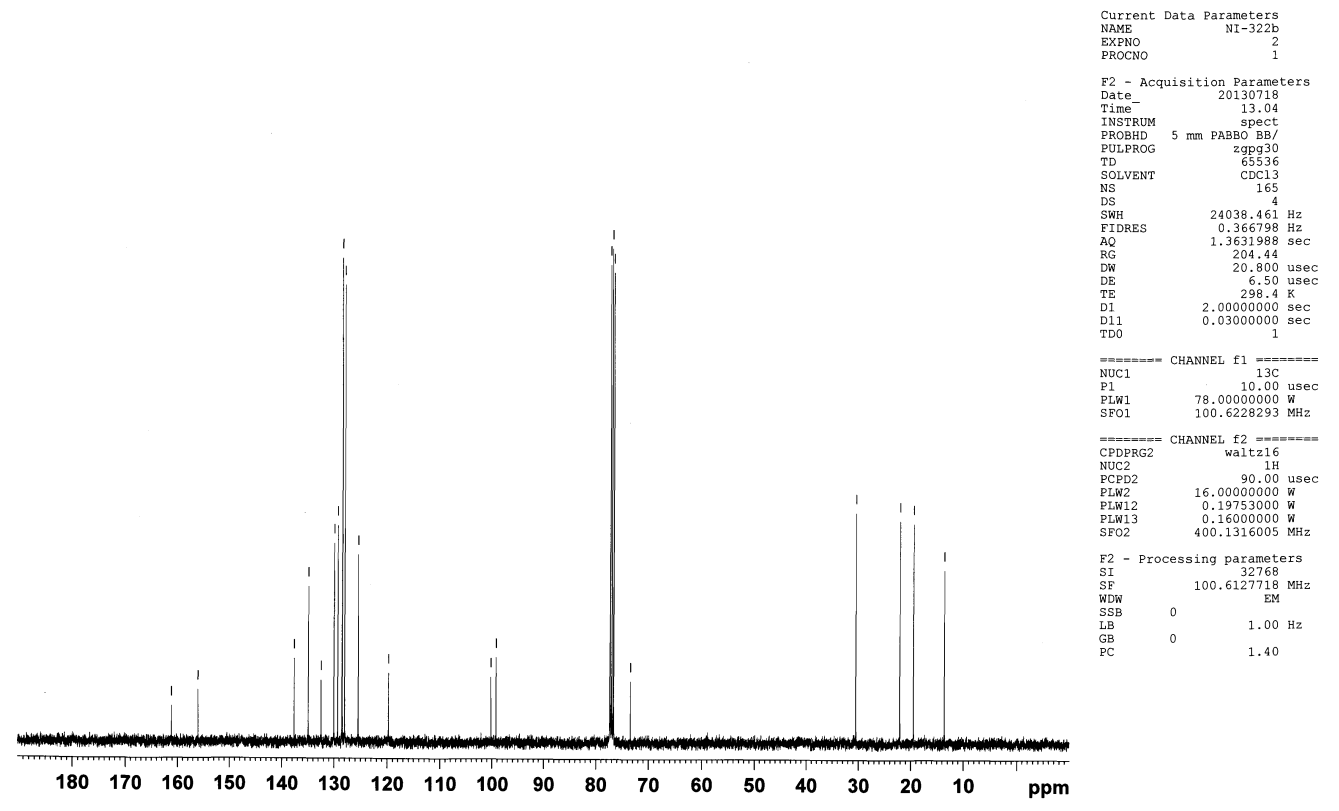


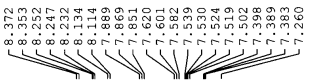
$C_{27}H_{24}O_3$	γ = 92.531 (1)°
M_r = 396.46	V = 981.26 (12) Å^3
Triclinic, $P\bar{1}$	Z = 2
Hall symbol: -P 1	F(000) = 420
a = 5.7885 (4) Å	D_x = 1.342 Mg m^{-3}
b = 10.4613 (8) Å	Mo $K\alpha$ radiation, λ = 0.71073 Å
c = 16.3808 (12) Å	μ = 0.09 mm^{-1}
α = 92.216 (1)°	T = 90 K
β = 97.607 (1)°	0.29 × 0.11 × 0.10 mm
Radiation source: fine-focus sealed tube	R_{int} = 0.021
Graphite monochromator	θ_{max} = 27.6°, θ_{min} = 1.3°
11621 measured reflections	h = -7→7
4505 independent reflections	k = -13→13

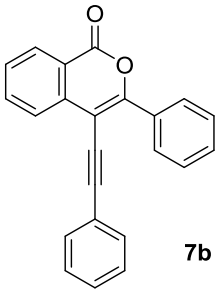
3883 reflections with $I > 2\sigma(I)$	l = -21→21
Refinement on F^2	Primary atom site location: structure-invariant direct methods
Least-squares matrix: full	Secondary atom site location: difference Fourier map
$R[F^2 > 2\sigma(F^2)]$ = 0.039	Hydrogen site location: inferred from neighbouring sites
$wR(F^2)$ = 0.112	H atoms treated by a mixture of independent and constrained refinement
S = 0.98	$w = 1/[\sigma^2(F_o^2) + (0.0641P)^2 + 0.3713P]$ where $P = (F_o^2 + 2F_c^2)/3$
4505 reflections	$(\Delta/\sigma)_{max} < 0.001$
275 parameters	$\Delta\rangle_{max}$ = 0.36 e Å^{-3}
0 restraints	$\Delta\rangle_{min}$ = -0.25 e Å^{-3}

7. ^{1}H and ^{13}C NMR spectra of all new compounds









7b

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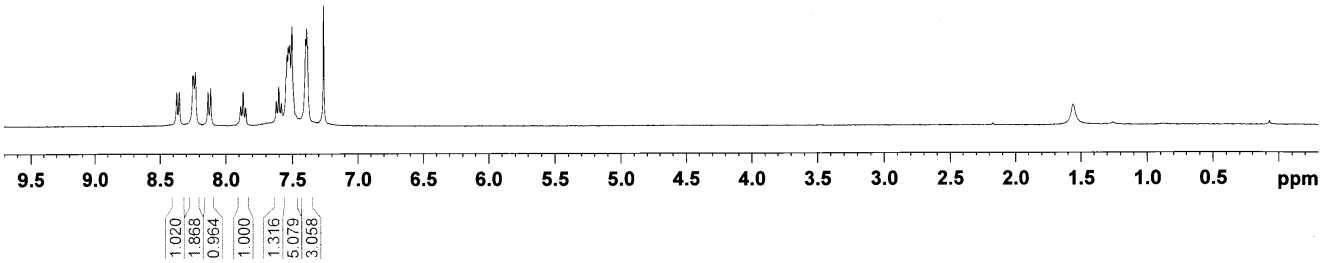

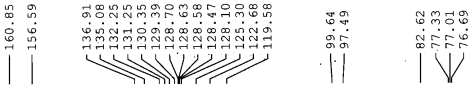


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PLW13                0.16000000 W
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PC                         1.40
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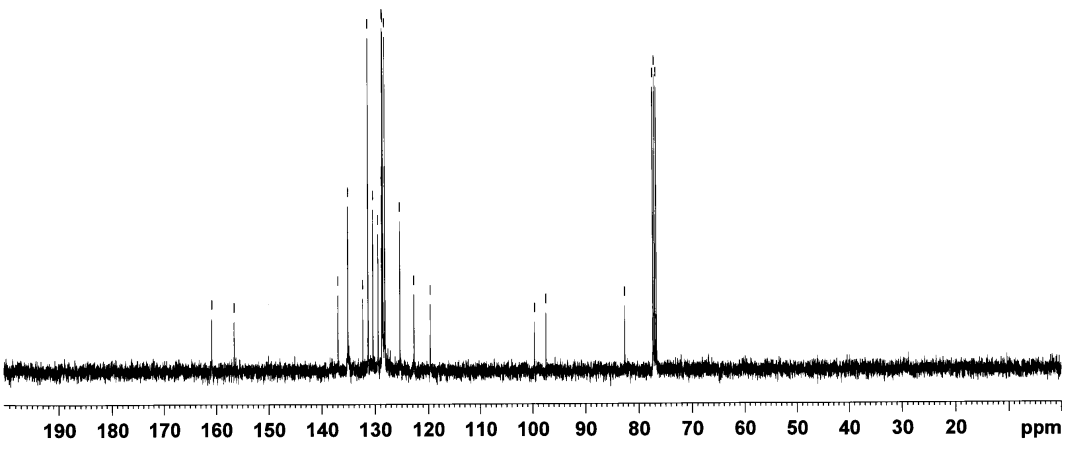

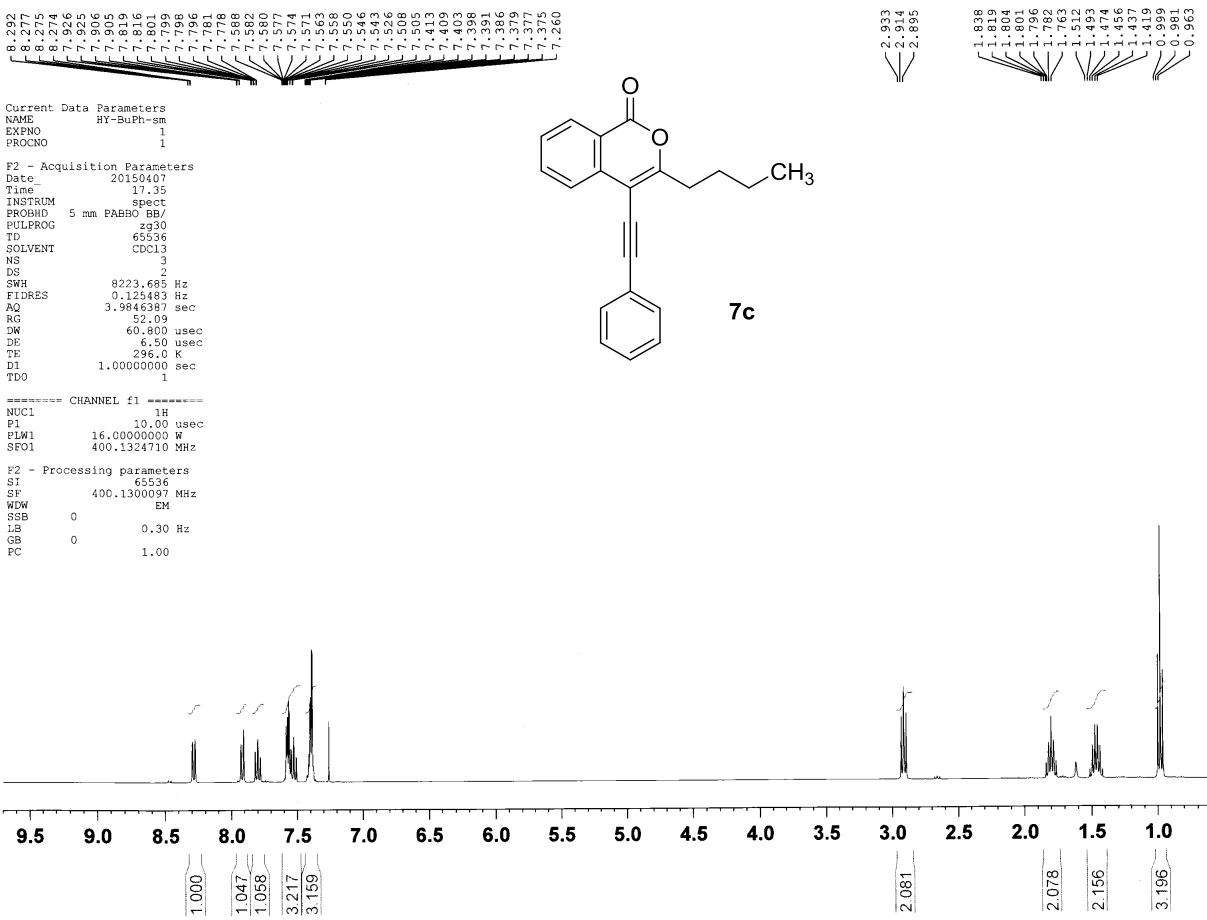
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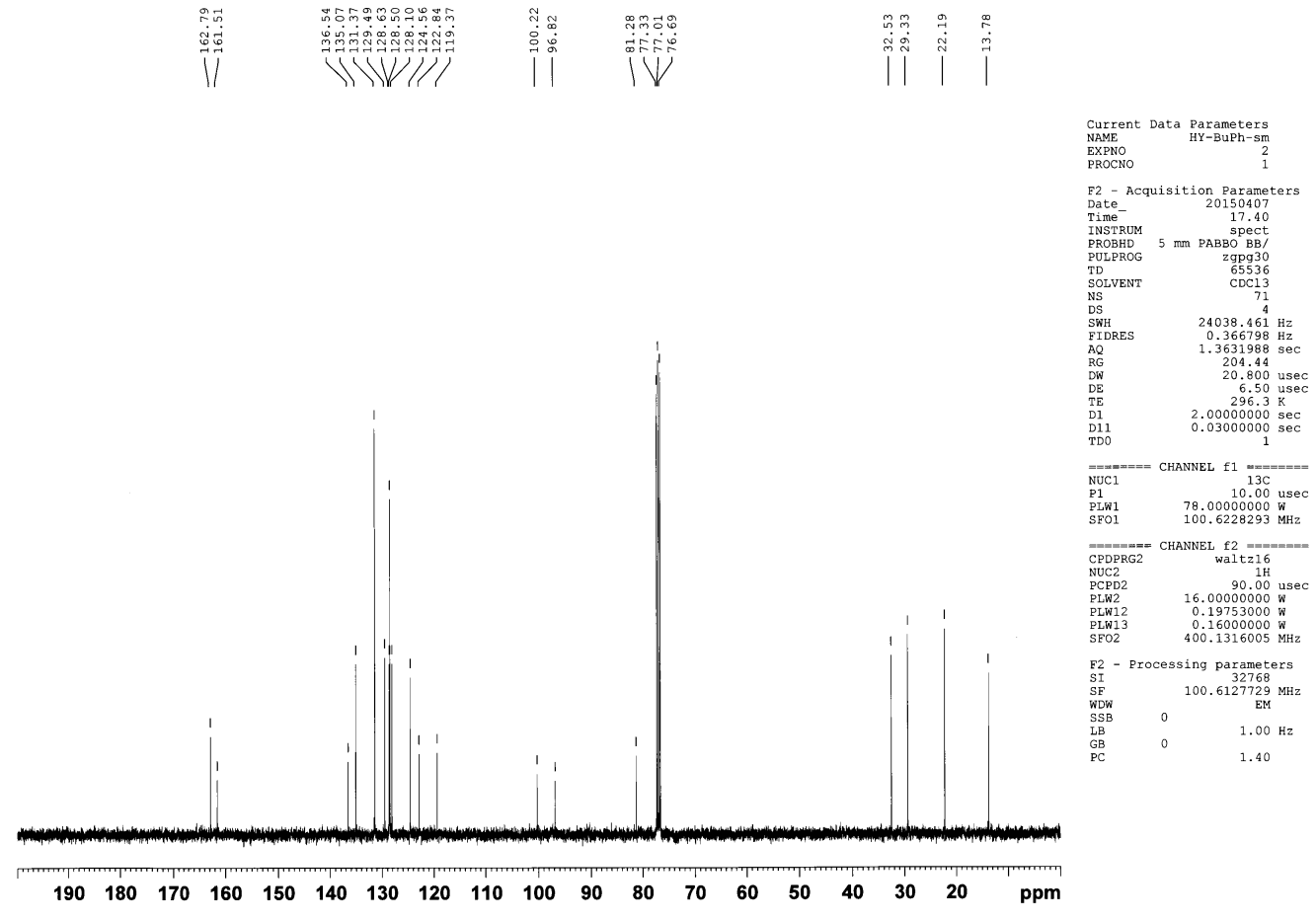
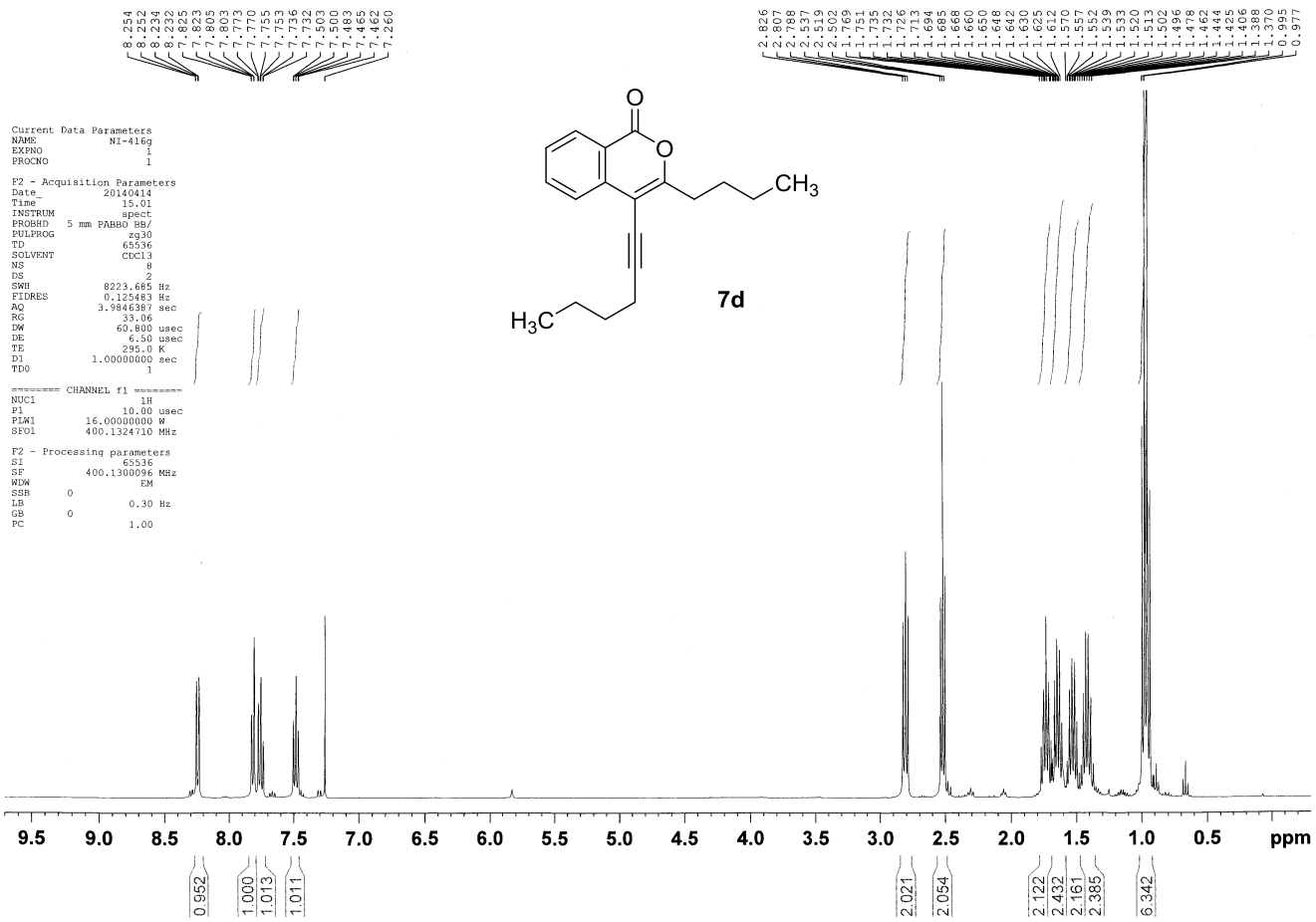
8.277
8.275
8.274
8.274
7.926
7.925
7.906
7.905
7.819
7.816
7.801
7.799
7.798
7.796
7.781
7.778
7.588
7.582
7.580
7.577
7.577
7.574
7.571
7.563
7.558
7.550
7.546
7.543
7.526
7.508
7.505
7.413
7.409
7.403
7.398
7.391
7.386
7.379
7.377
7.375
7.260
2.933
2.914
2.895
1.838
1.819
1.804
1.801
1.796
1.782
1.763
1.512
1.493
1.474
1.456
1.437
1.419
0.999
0.981
0.963
Current Data Parameters
NAME HY-BuPh-sm
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150407
Time 17.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 52.09
DW 60.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
CH3
7c
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.000
1.047
1.058
3.217
3.159
2.081
2.078
2.156
3.196


162.79
161.51
136.54
135.07
131.37
129.49
128.63
128.50
128.10
124.56
122.84
119.37
100.22
96.82
81.28
77.33
77.01
76.69
32.53
29.33
22.19
13.78
Current Data Parameters
NAME HY-BuPh-sm
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150407
Time 17.40
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 71
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127729 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm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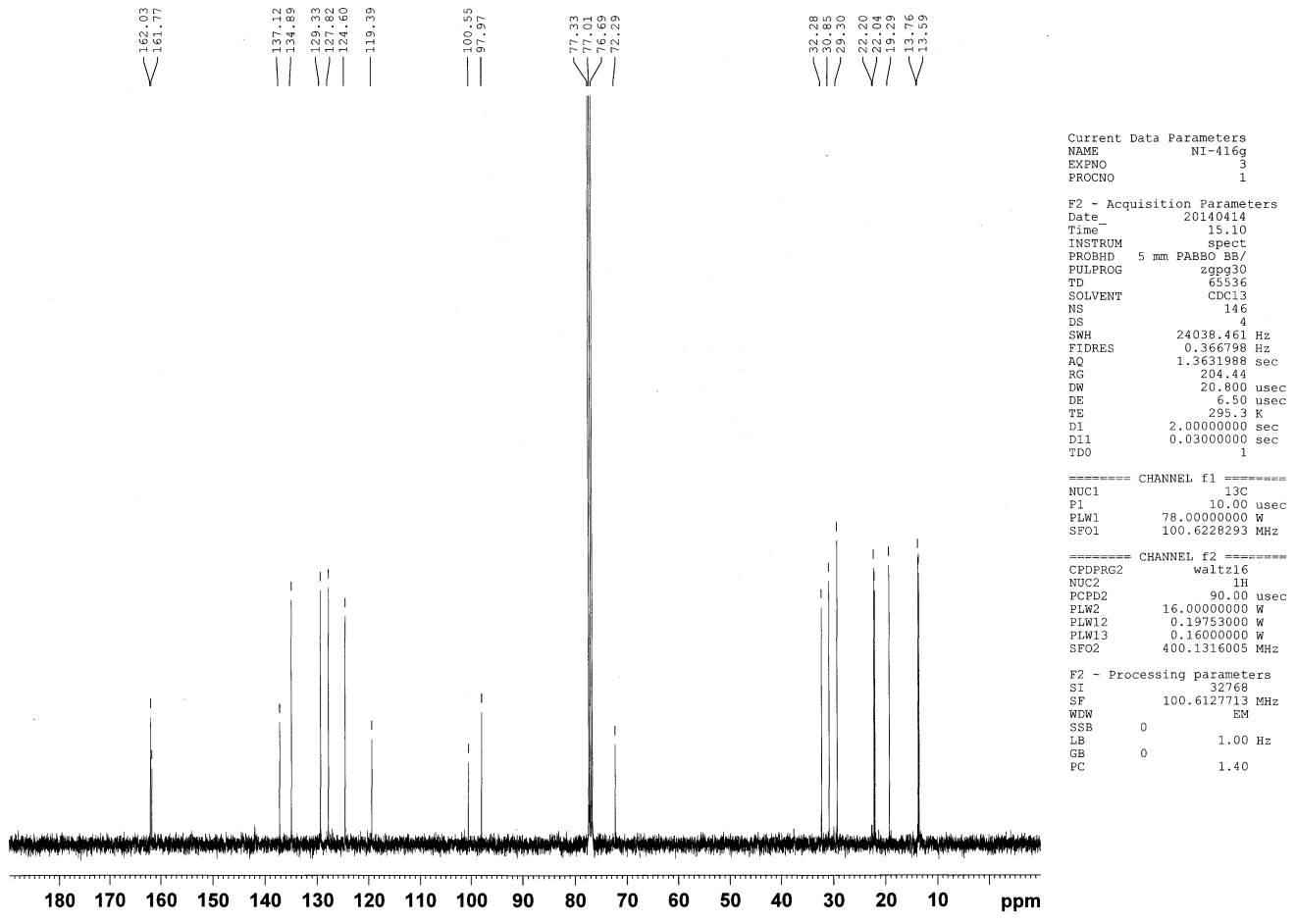
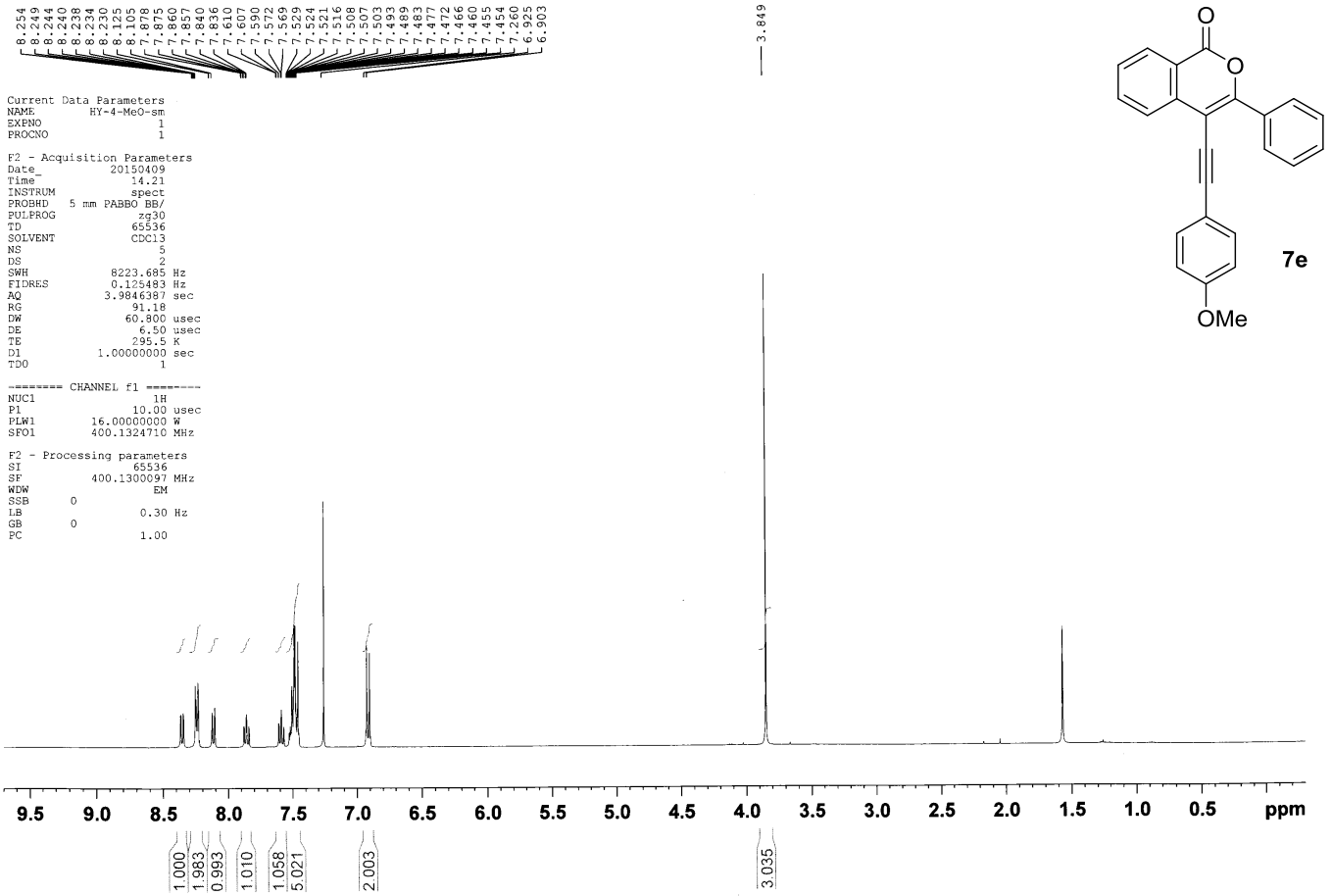
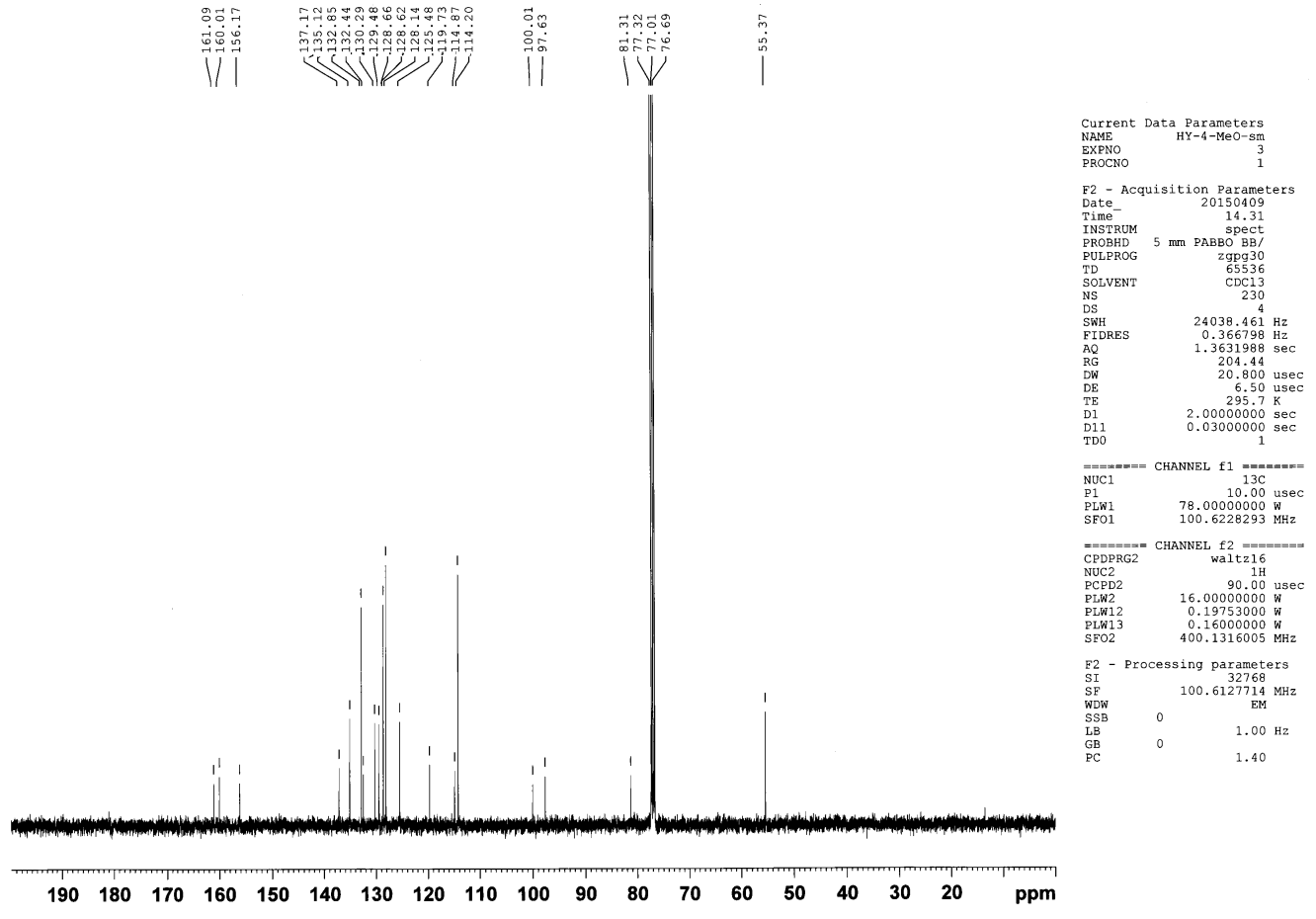
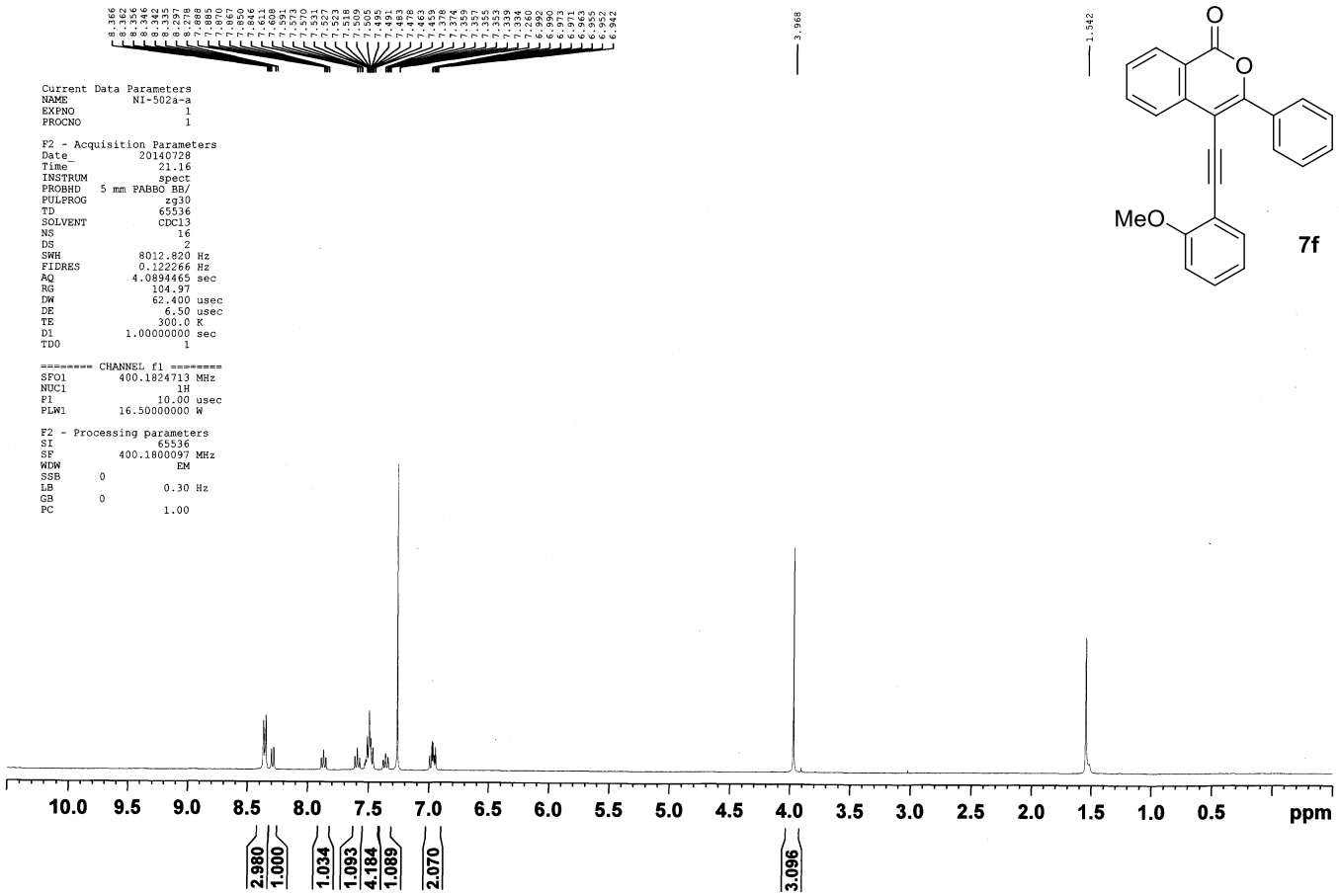
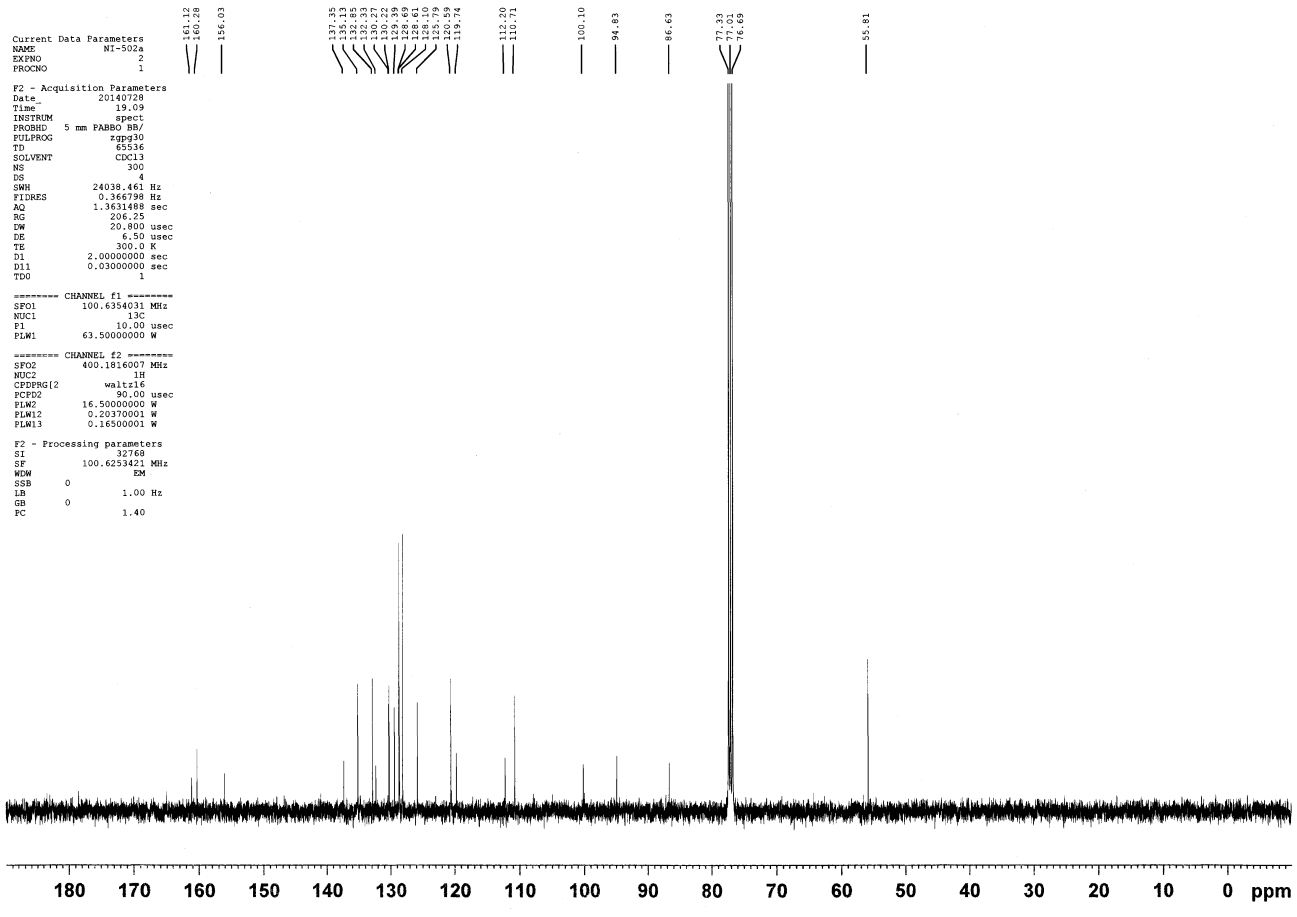
8.252
8.234
8.232
7.825
7.823
7.805
7.803
7.773
7.770
7.755
7.753
7.736
7.732
7.503
7.500
7.483
7.465
7.462
7.260
2.826
2.807
2.788
2.537
2.519
2.502
1.769
1.751
1.735
1.732
1.726
1.713
1.694
1.685
1.668
1.660
1.650
1.648
1.642
1.630
1.625
1.612
1.570
1.557
1.552
1.539
1.533
1.520
1.513
1.502
1.496
1.478
1.462
1.444
1.425
1.406
1.388
1.370
0.995
0.977
Current Data Parameters
NAME NI-416g
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140414
Time_ 15.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 295.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
CH3
H3C
7d
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.952
1.000
1.013
1.011
2.021
2.054
2.122
2.432
2.161
2.385
6.342

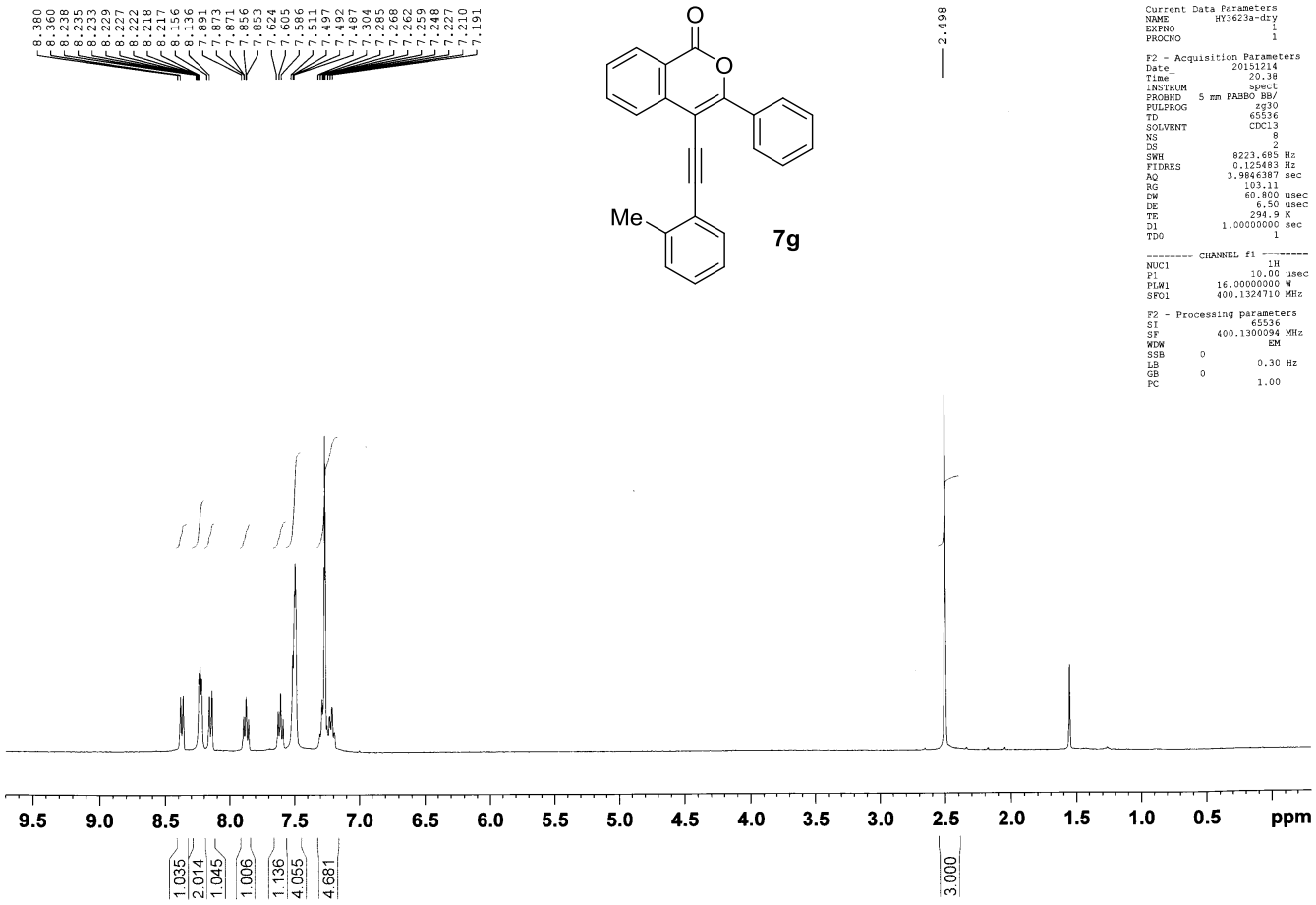

162.03
161.77
137.12
134.89
129.33
127.82
124.60
119.39
100.55
97.97
77.33
77.01
76.69
72.29
32.28
30.85
29.30
22.20
22.04
19.29
13.76
13.59
Current Data Parameters
NAME NI-416g
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140414
Time_ 15.10
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 146
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127713 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

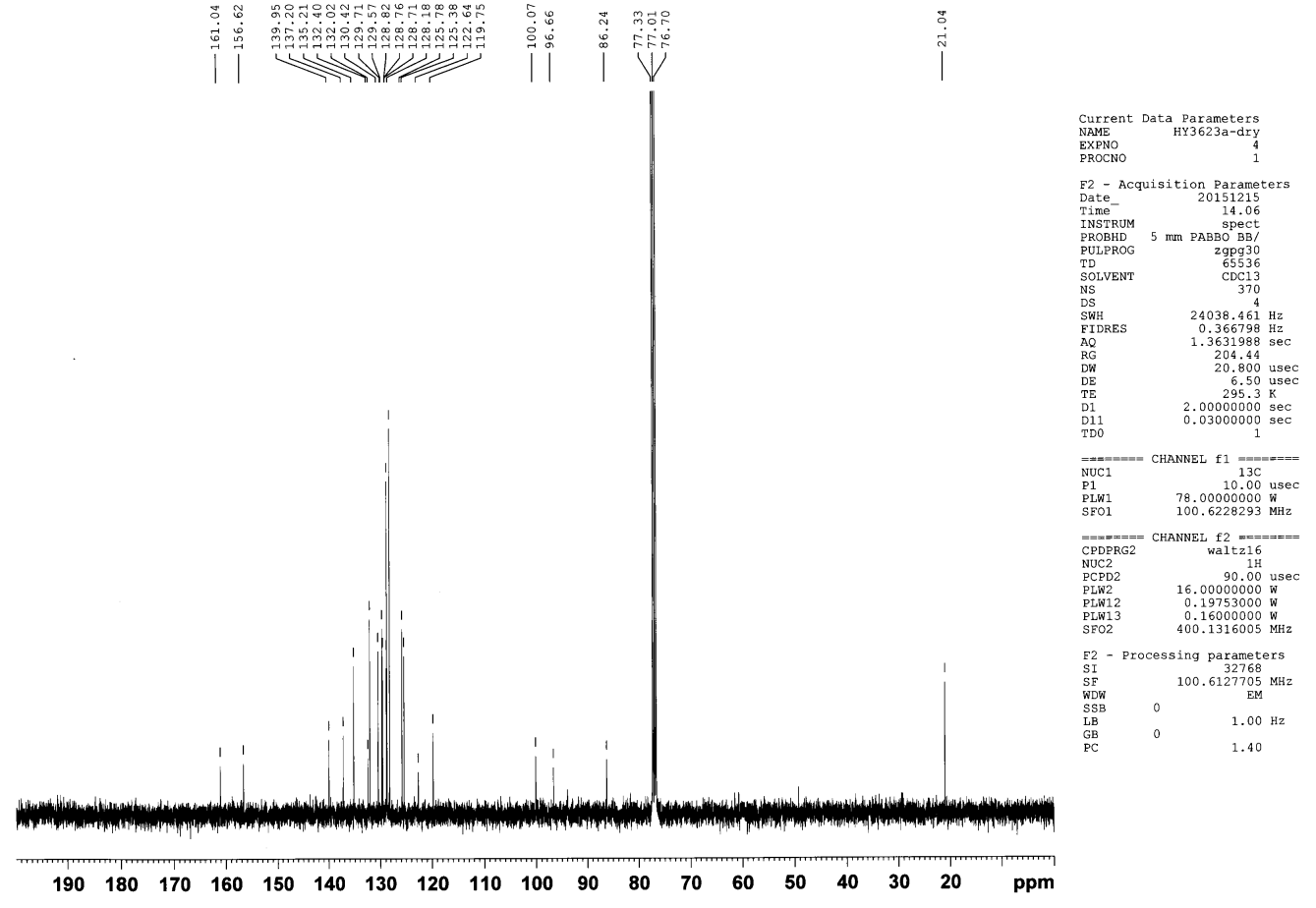

8.254
8.249
8.244
8.240
8.238
8.234
8.230
8.125
8.105
7.878
7.875
7.860
7.857
7.840
7.836
7.610
7.607
7.590
7.572
7.569
7.529
7.524
7.521
7.516
7.508
7.507
7.493
7.489
7.483
7.477
7.472
7.466
7.460
7.455
7.454
7.260
6.925
6.903
3.849
Current Data Parameters
NAME HY-4-MeO-sm
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150409
Time 14.21
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 91.18
DW 60.800 usec
DE 6.50 usec
TE 295.5 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
OMe
7e
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.000
1.983
0.993
1.010
1.058
5.021
2.003
3.035


161.09
160.01
156.17
137.17
135.12
132.85
132.44
130.29
129.48
128.66
128.62
128.14
125.48
119.73
114.87
114.20
100.01
97.63
81.31
77.32
77.01
76.69
55.37
Current Data Parameters
NAME HY-4-MeO-sm
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150409
Time 14.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 230
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


8.366
8.362
8.356
8.346
8.342
8.335
8.297
8.288
8.280
7.885
7.870
7.867
7.850
7.846
7.611
7.608
7.591
7.573
7.570
7.531
7.527
7.523
7.518
7.509
7.505
7.495
7.491
7.483
7.478
7.463
7.459
7.378
7.374
7.359
7.357
7.355
7.353
7.339
7.334
7.260
6.992
6.990
6.973
6.971
6.963
6.955
6.952
6.942
3.968
1.542
Current Data Parameters
NAME NI-502a-a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140728
Time 21.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 104.97
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1824713 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1800097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
O
O
MeO
7f
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.980
1.000
1.034
1.093
4.184
1.089
2.070
3.096


161.12
160.28
156.03
137.35
135.13
132.85
132.53
130.27
130.22
129.89
129.69
128.61
128.10
125.79
125.59
119.74
112.20
110.71
100.10
94.83
86.63
77.33
77.01
76.69
55.81
Current Data Parameters
NAME NI-502a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140728
Time 19.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 206.25
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6354031 MHz
NUC1 13C
P1 10.00 usec
PLW1 63.50000000 W
======== CHANNEL f2 ========
SFO2 400.1816007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.50000000 W
PLW12 0.20370001 W
PLW13 0.16500001 W
F2 - Processing parameters
SI 32768
SF 100.6253421 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


8.380
8.360
8.238
8.235
8.233
8.229
8.227
8.222
8.218
8.217
8.156
8.136
7.891
7.873
7.871
7.856
7.853
7.624
7.605
7.586
7.511
7.497
7.492
7.487
7.304
7.285
7.268
7.262
7.259
7.248
7.227
7.210
7.191
2.498
O
O
Me
7g
Current Data Parameters
NAME HY3623a-dry
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151214
Time 20.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 103.11
DW 60.800 usec
DE 6.50 usec
TE 294.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300094 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.035
2.014
1.045
1.006
1.136
4.055
4.681
3.000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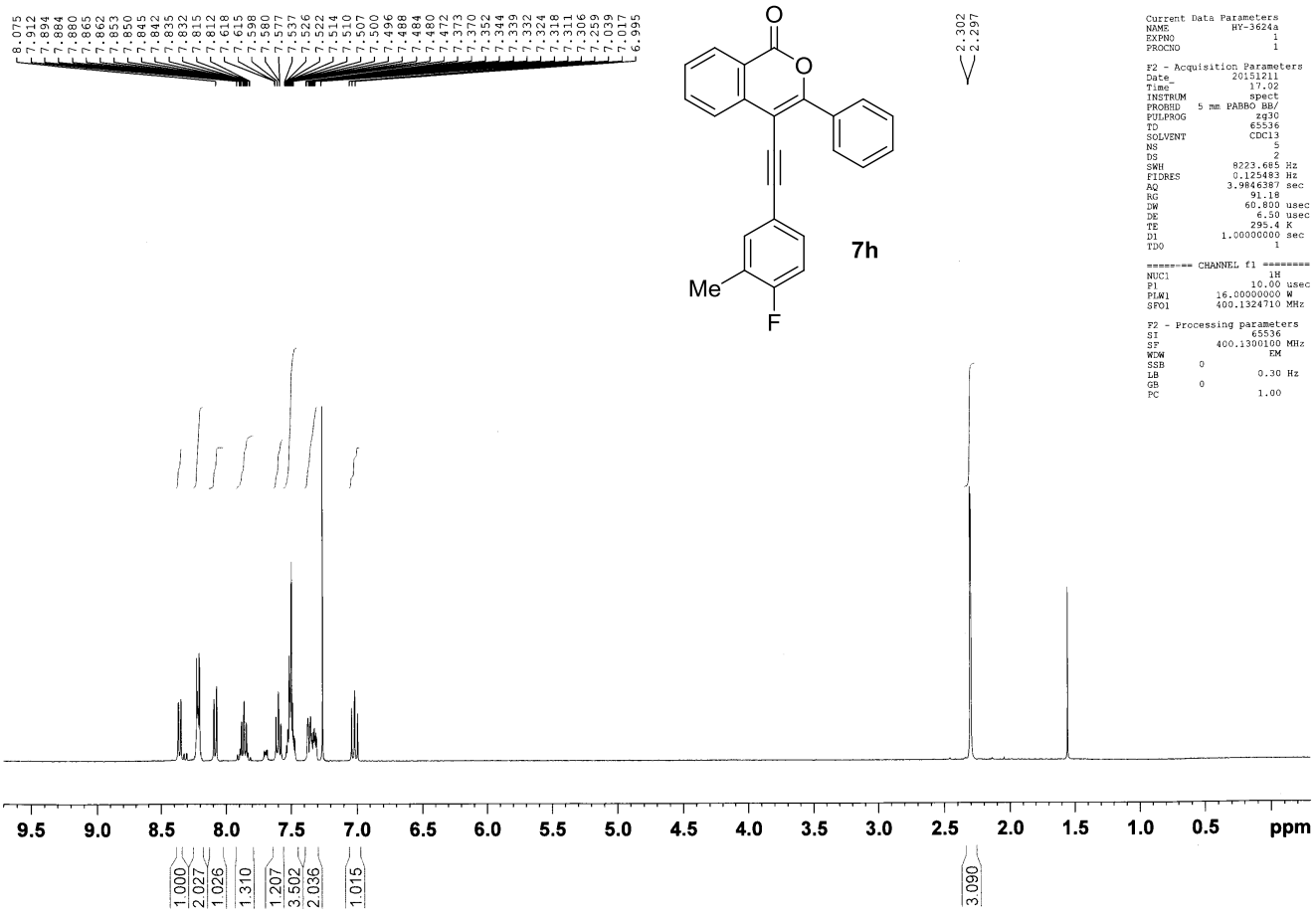
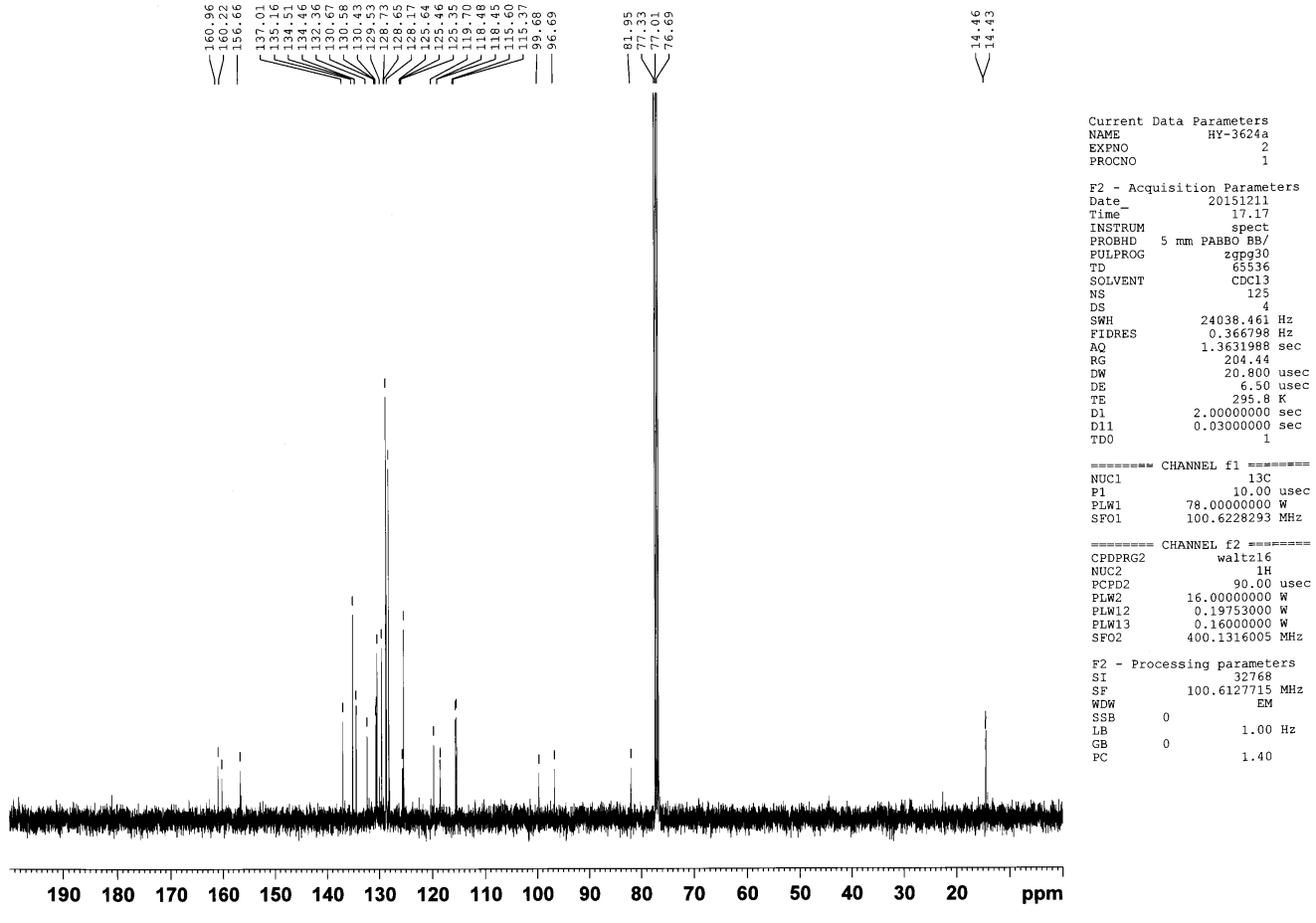
156.62
139.95
137.20
135.21
132.40
132.02
130.42
129.71
129.57
128.82
128.76
128.71
128.18
125.78
125.38
122.64
119.75
100.07
96.66
86.24
77.33
77.01
76.70
21.04
Current Data Parameters
NAME HY3623a-dry
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151215
Time 14.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 370
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127705 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm


8.075
7.912
7.894
7.884
7.880
7.865
7.862
7.853
7.850
7.845
7.842
7.835
7.832
7.815
7.812
7.618
7.615
7.598
7.580
7.577
7.537
7.526
7.522
7.514
7.510
7.507
7.500
7.496
7.488
7.484
7.480
7.472
7.373
7.370
7.352
7.344
7.339
7.332
7.324
7.318
7.311
7.306
7.259
7.039
7.017
6.995
2.302
2.297
O
O
Me
F
7h
Current Data Parameters
NAME HY-3624a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151211
Time 17.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 5
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 91.18
DW 60.800 usec
DE 6.50 usec
TE 295.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.000
2.027
1.026
1.310
1.207
3.502
2.036
1.015
3.090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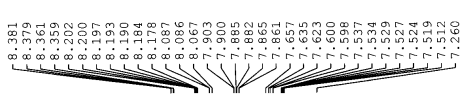
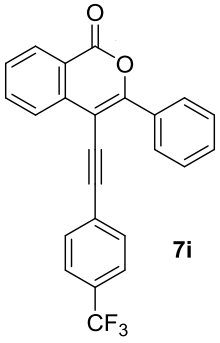
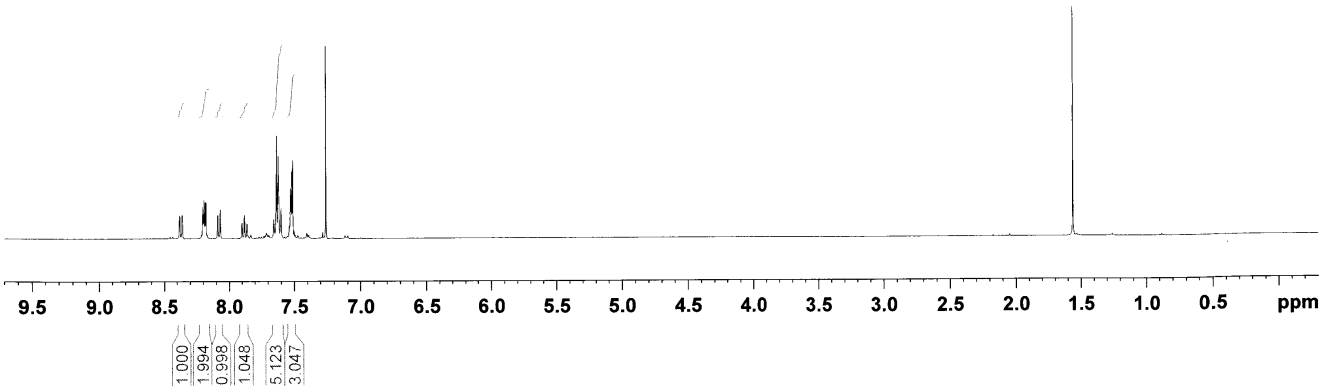
160.22
156.66
137.01
135.16
134.51
134.46
132.36
130.67
130.58
130.43
129.53
128.73
128.65
128.17
125.64
125.46
125.35
119.70
118.48
118.45
115.60
115.37
99.68
96.69
81.95
77.33
77.01
76.69
14.46
14.43
Current Data Parameters
NAME HY-3624a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151211
Time 17.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 125
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127715 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm

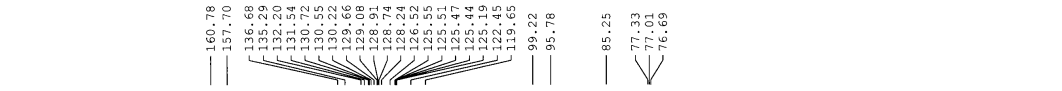


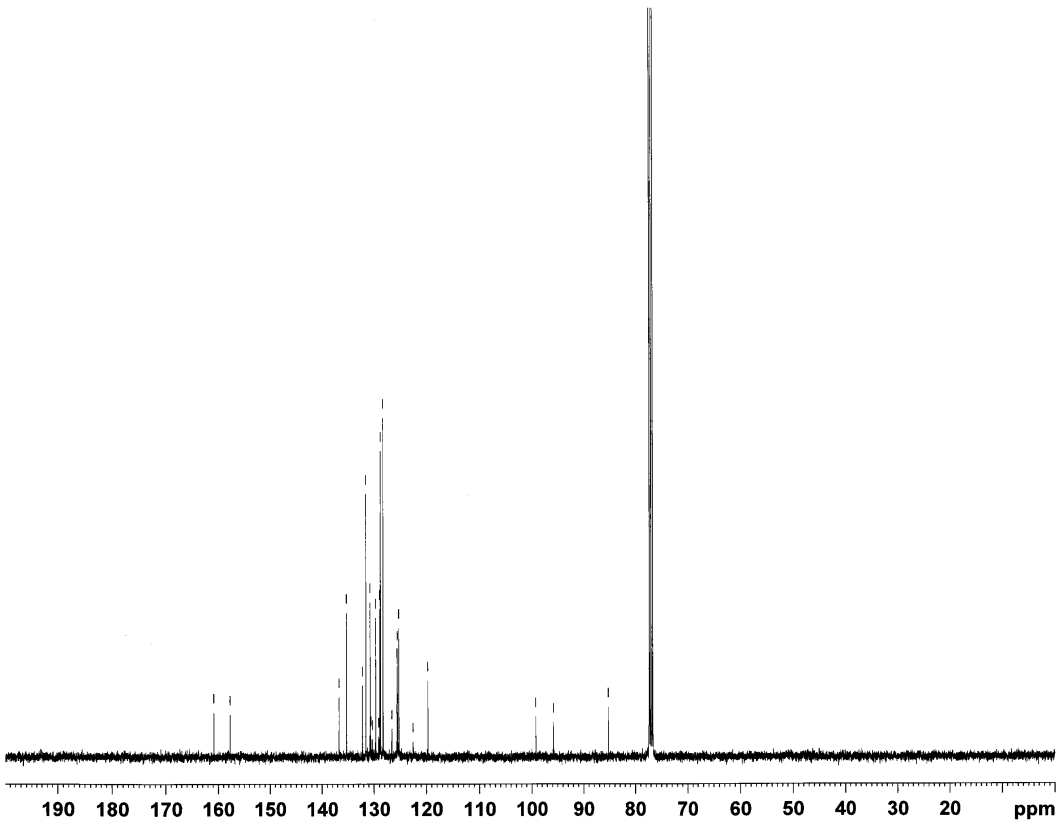


7i

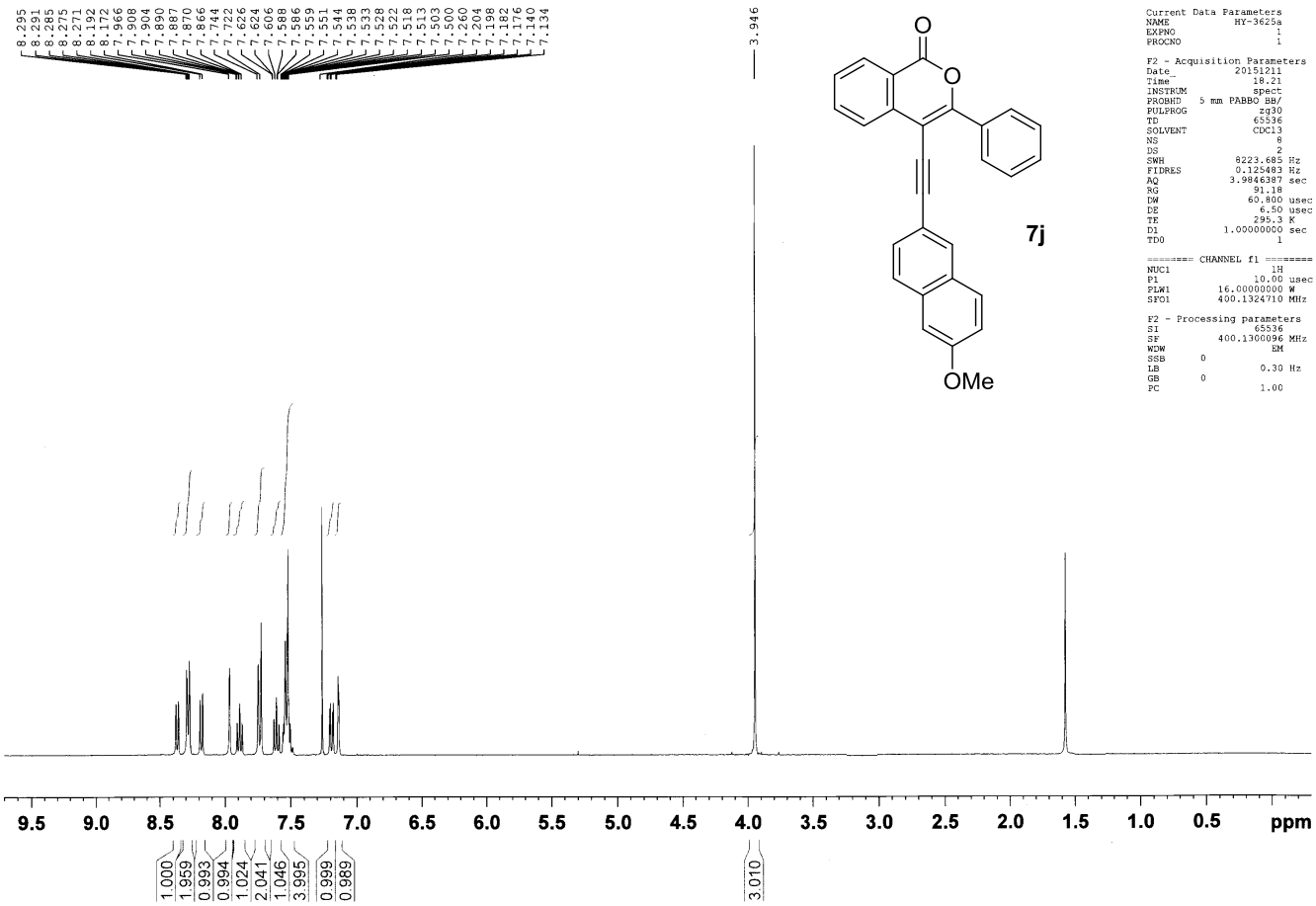
```
Current Data Parameters
NAME                  HY-3646a
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20160518
Time                     13.46
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                  zg30
TD                      65536
SOLVENT                  CDCl3
NS                           4
DS                           2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9846387 sec
RG                       103.11
DW                       60.800 usec
DE                        6.50 usec
TE                       296.1 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       10.00 usec
PLW1                16.00000000 W
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                      65536
SF                  400.1300097 MHz
WDW                        EM
SSB                  0
LB                       0.30 Hz
GB                   0
PC                       1.00
```



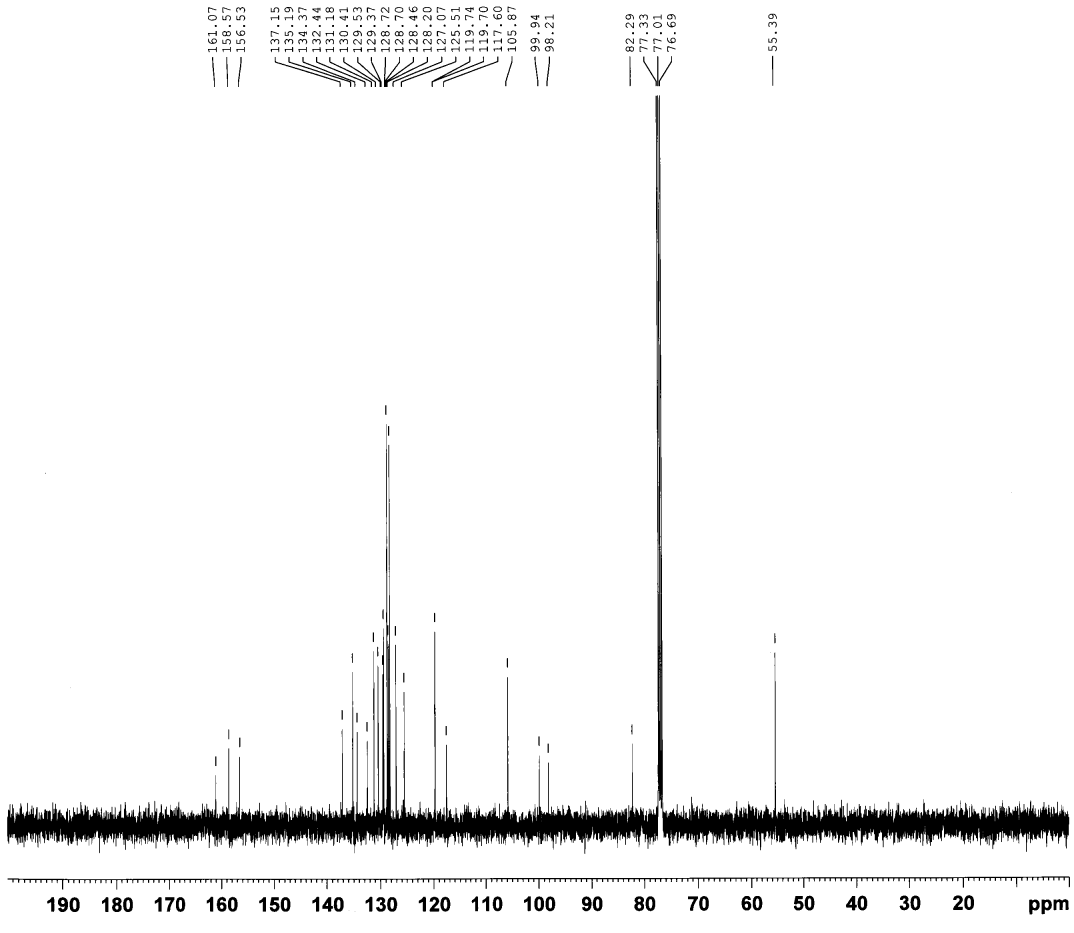




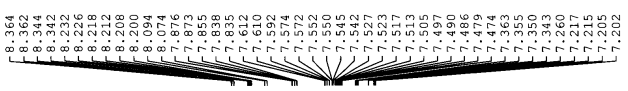

```
Current Data Parameters
NAME                  HY-3646a
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                 20160518
Time                     13.56
INSTRUM                  spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                      65536
SOLVENT                  CDCl3
NS                         788
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                       204.44
DW                       20.800 usec
DE                        6.50 usec
TE                       296.4 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       10.00 usec
PLW1                78.00000000 W
SFO1               100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    90.00 usec
PLW2                16.00000000 W
PLW12                0.19753000 W
PLW13                0.16000000 W
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                      32768
SF                 100.6127708 MHz
WDW                        EM
SSB                  0
LB                       1.00 Hz
GB                   0
PC                       1.40
```

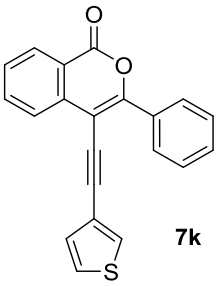



```
Current Data Parameters
NAME                  HY-3625a
EXPNO                        1
PROCNO                       1

F2 - Acquisition Parameters
Date_                20151211
Time                    18.21
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CDCl3
NS                           8
DS                           2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                  3.9846387 sec
RG                       91.18
DW                      60.800 usec
DE                       6.50 usec
TE                      295.3 K
D1                  1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        1H
P1                       10.00 usec
PLW1                16.00000000 W
SFO1               400.1324710 MHz

F2 - Processing parameters
SI                          65536
SF                 400.1300096 MHz
WDW                            EM
SSB                          0
LB                        0.30 Hz
GB                           0
PC                        1.00
```



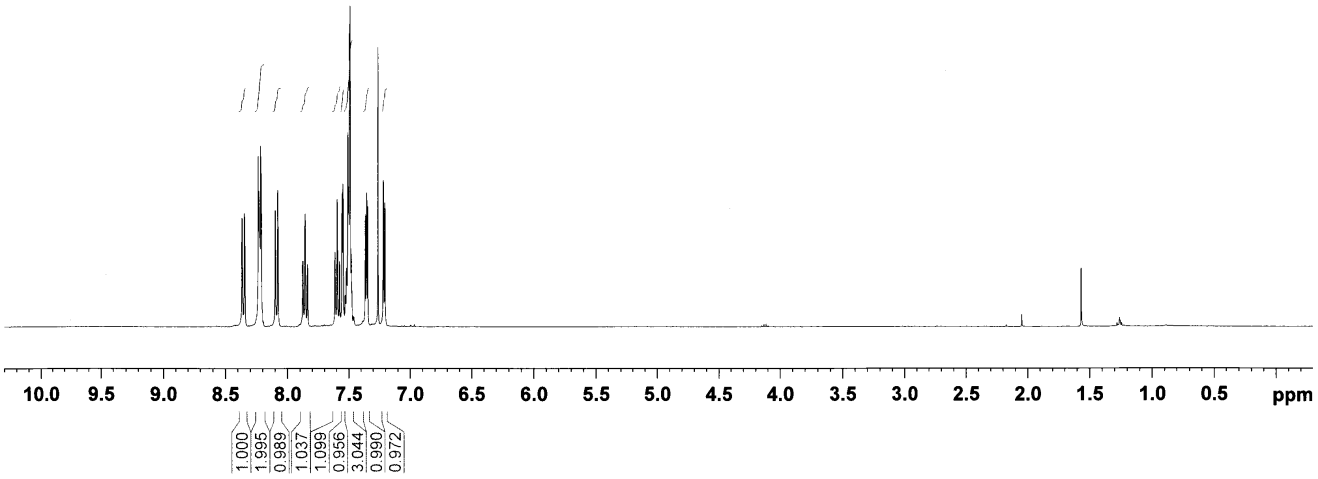

```
Current Data Parameters
NAME                  HY-3625a
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                20151211
Time                    18.24
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                     65536
SOLVENT                CDCl3
NS                         248
DS                           4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                  1.3631988 sec
RG                      204.44
DW                      20.800 usec
DE                       6.50 usec
TE                      295.7 K
D1                  2.00000000 sec
D11                 0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                       13C
P1                       10.00 usec
PLW1                78.00000000 W
SFO1               100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                        1H
PCPD2                    90.00 usec
PLW2                16.00000000 W
PLW12                0.19753000 W
PLW13                0.16000000 W
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                          32768
SF                 100.6127709 MHz
WDW                            EM
SSB                          0
LB                        1.00 Hz
GB                           0
PC                        1.40
```

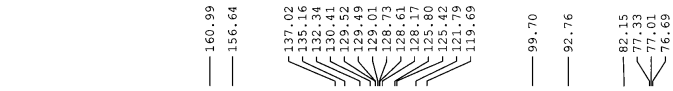


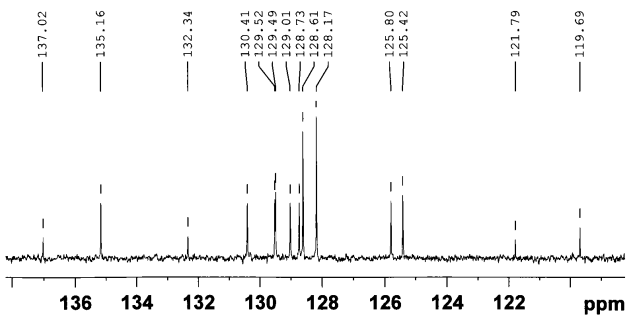


7k

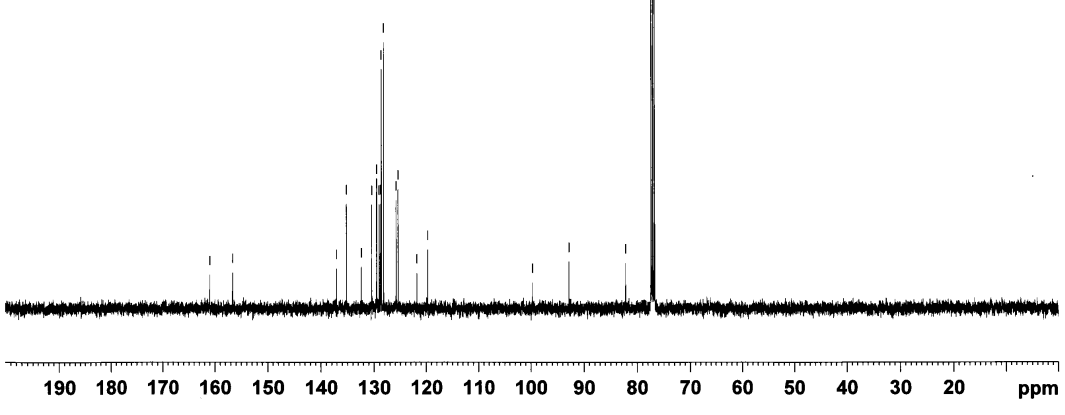
```
Current Data Parameters
NAME                  HY-3633b1
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20151218
Time_                     16.59
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                   zg30
TD                       65536
SOLVENT                  CDCl3
NS                           8
DS                           2
SWH                   8223.685 Hz
FIDRES                0.125483 Hz
AQ                    3.9846387 sec
RG                        91.18
DW                       60.800 usec
DE                        6.50 usec
TE                       295.0 K
D1                    1.00000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                         1H
P1                       10.00 usec
PLW1                 16.00000000 W
SFO1                400.1324710 MHz

F2 - Processing parameters
SI                       65536
SF                  400.1300095 MHz
WDW                         EM
SSB                  0
LB                        0.30 Hz
GB                   0
PC                        1.00
```

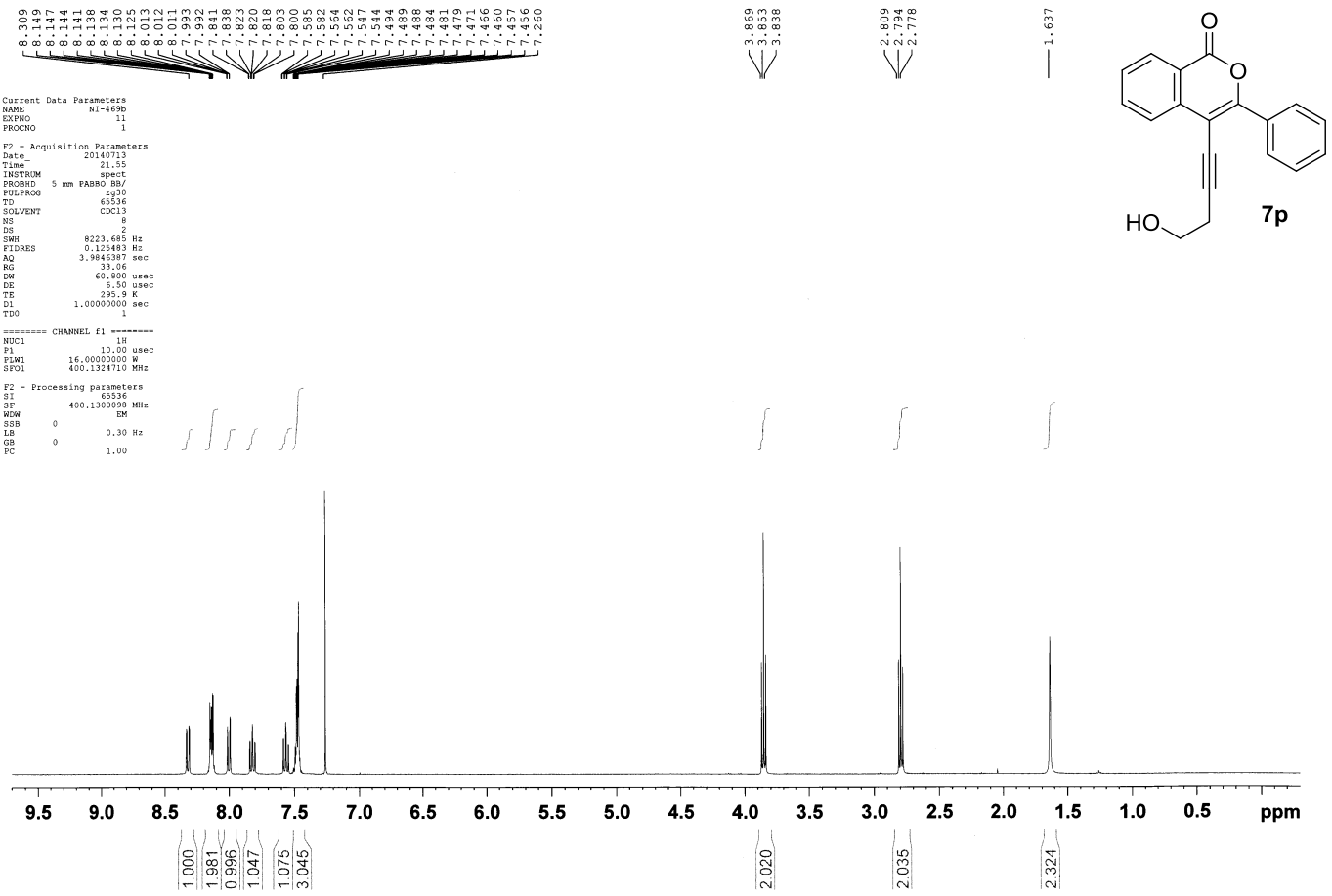


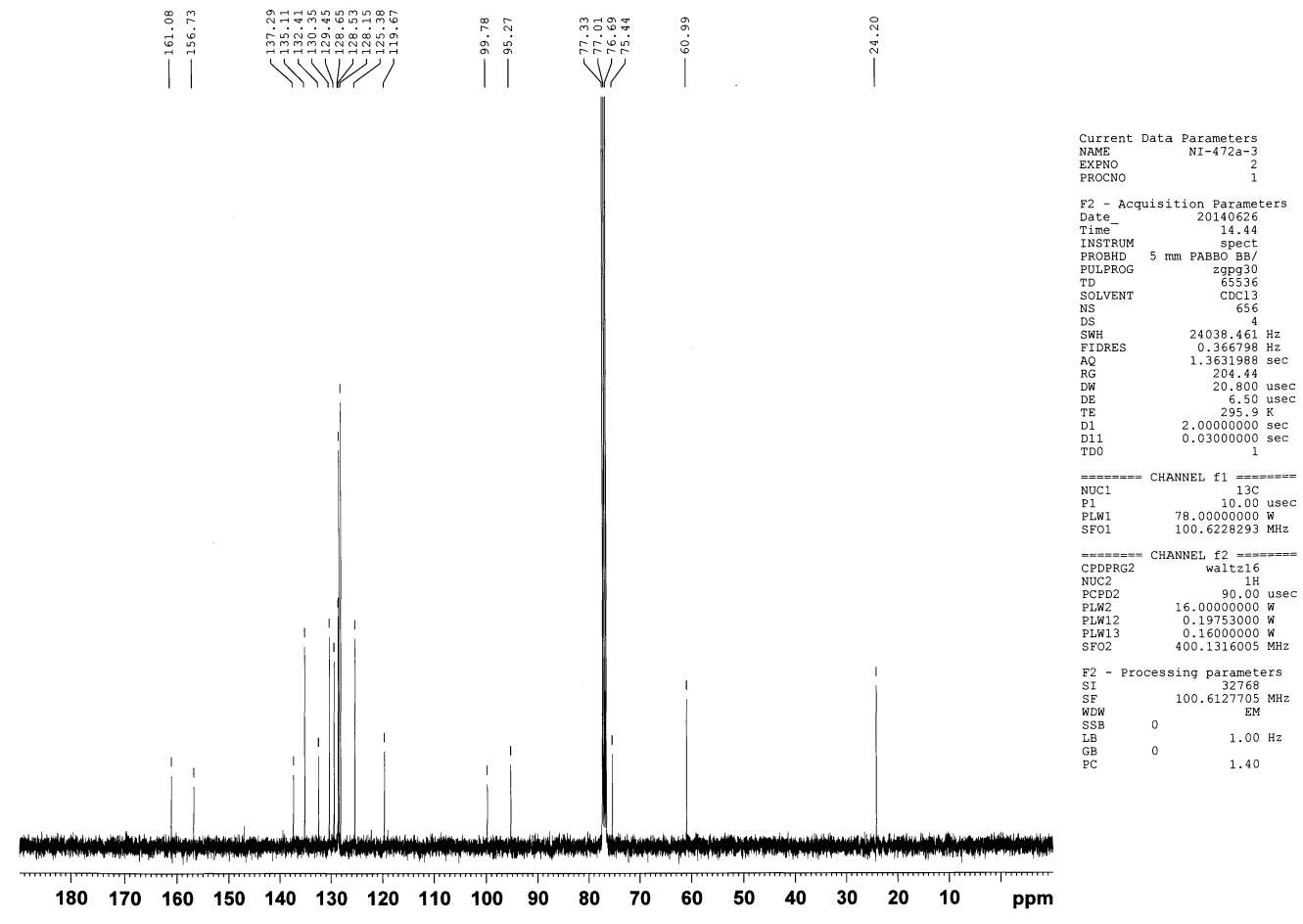


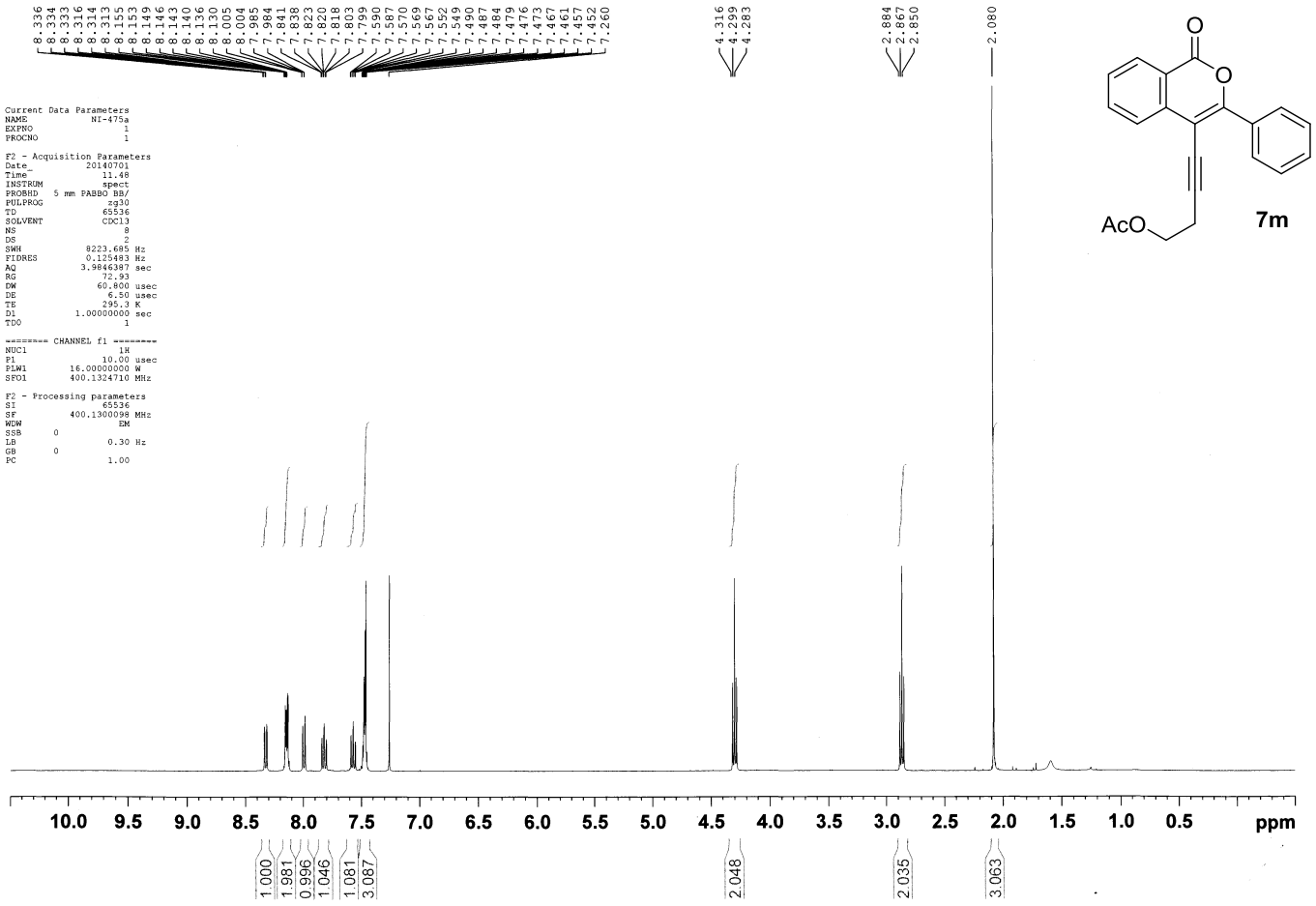


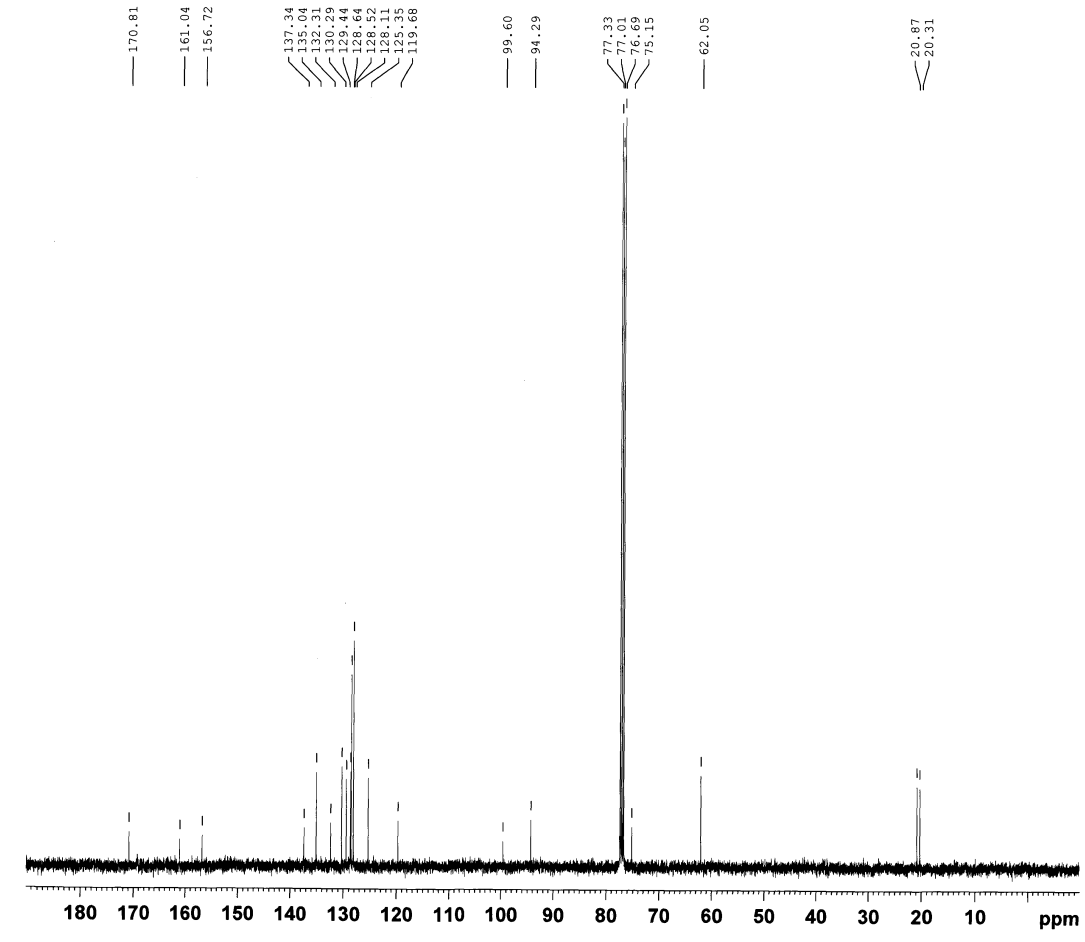

```
Current Data Parameters
NAME                  HY-3633b1
EXPNO                         2
PROCNO                        1

F2 - Acquisition Parameters
Date_                  20151218
Time_                     17.04
INSTRUM                   spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                       65536
SOLVENT                  CDCl3
NS                         221
DS                           4
SWH                  24038.461 Hz
FIDRES                0.366798 Hz
AQ                    1.3631988 sec
RG                       204.44
DW                       20.800 usec
DE                        6.50 usec
TE                       295.3 K
D1                    2.00000000 sec
D11                   0.03000000 sec
TD0                          1

======== CHANNEL f1 ========
NUC1                        13C
P1                       10.00 usec
PLW1                78.00000000 W
SFO1               100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2                 waltz16
NUC2                         1H
PCPD2                    90.00 usec
PLW2                16.00000000 W
PLW12                0.19753000 W
PLW13                0.16000000 W
SFO2               400.1316005 MHz

F2 - Processing parameters
SI                       32768
SF                 100.6127714 MHz
WDW                         EM
SSB                  0
LB                        1.00 Hz
GB                   0
PC                        1.40
```




8.309
8.149
8.147
8.144
8.141
8.138
8.134
8.130
8.125
8.013
8.012
8.011
7.993
7.992
7.841
7.838
7.823
7.820
7.818
7.803
7.800
7.585
7.582
7.564
7.562
7.547
7.544
7.494
7.489
7.488
7.484
7.481
7.479
7.471
7.466
7.460
7.457
7.456
7.260
3.869
3.853
3.838
2.809
2.794
2.778
1.637
O
O
HO
7p
Current Data Parameters
NAME NI-469b
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140713
Time 21.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 295.9 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300090 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.000
1.981
0.996
1.047
1.075
3.045
2.020
2.035
2.324


161.08
156.73
137.29
135.11
132.41
130.35
129.45
128.65
128.53
128.15
125.38
119.67
99.78
95.27
77.33
77.01
76.69
75.44
60.99
24.20
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Current Data Parameters
NAME NI-472a-3
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140626
Time 14.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 656
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127705 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





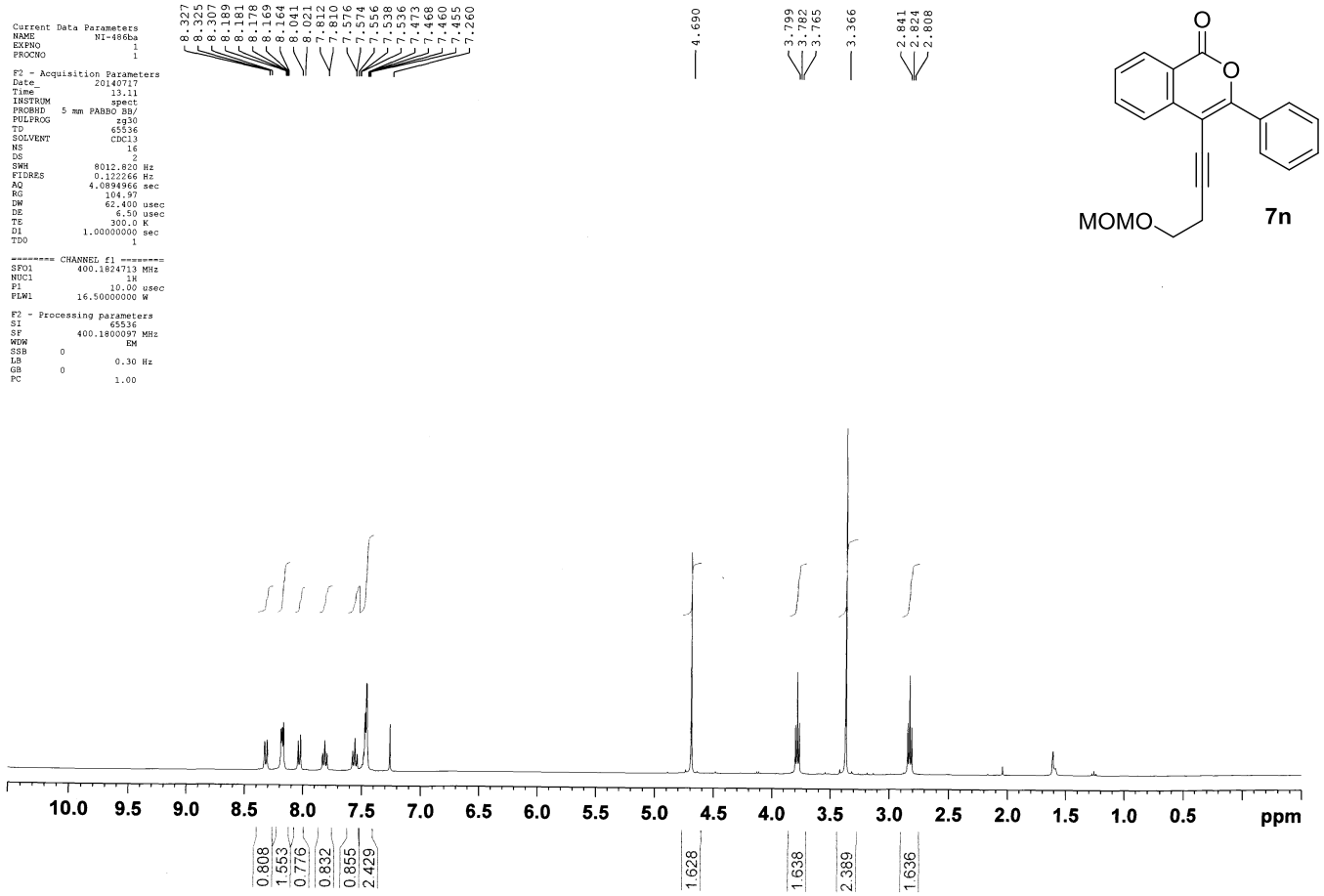
Current Data Parameters
NAME NI-475a
EXPNO 2
PROCNO 1

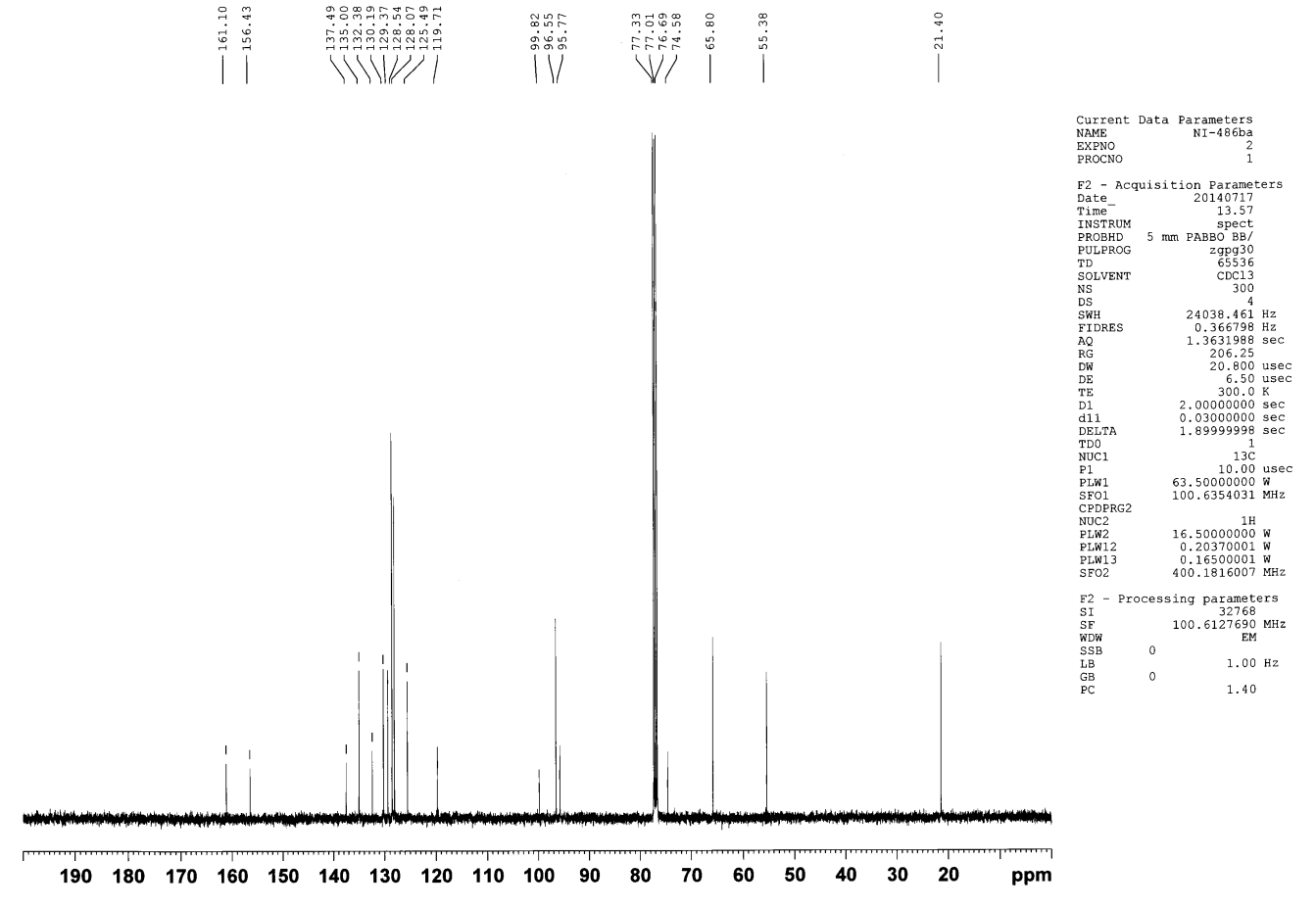
F2 - Acquisition Parameters
Date_ 20140701
Time 11.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 196
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

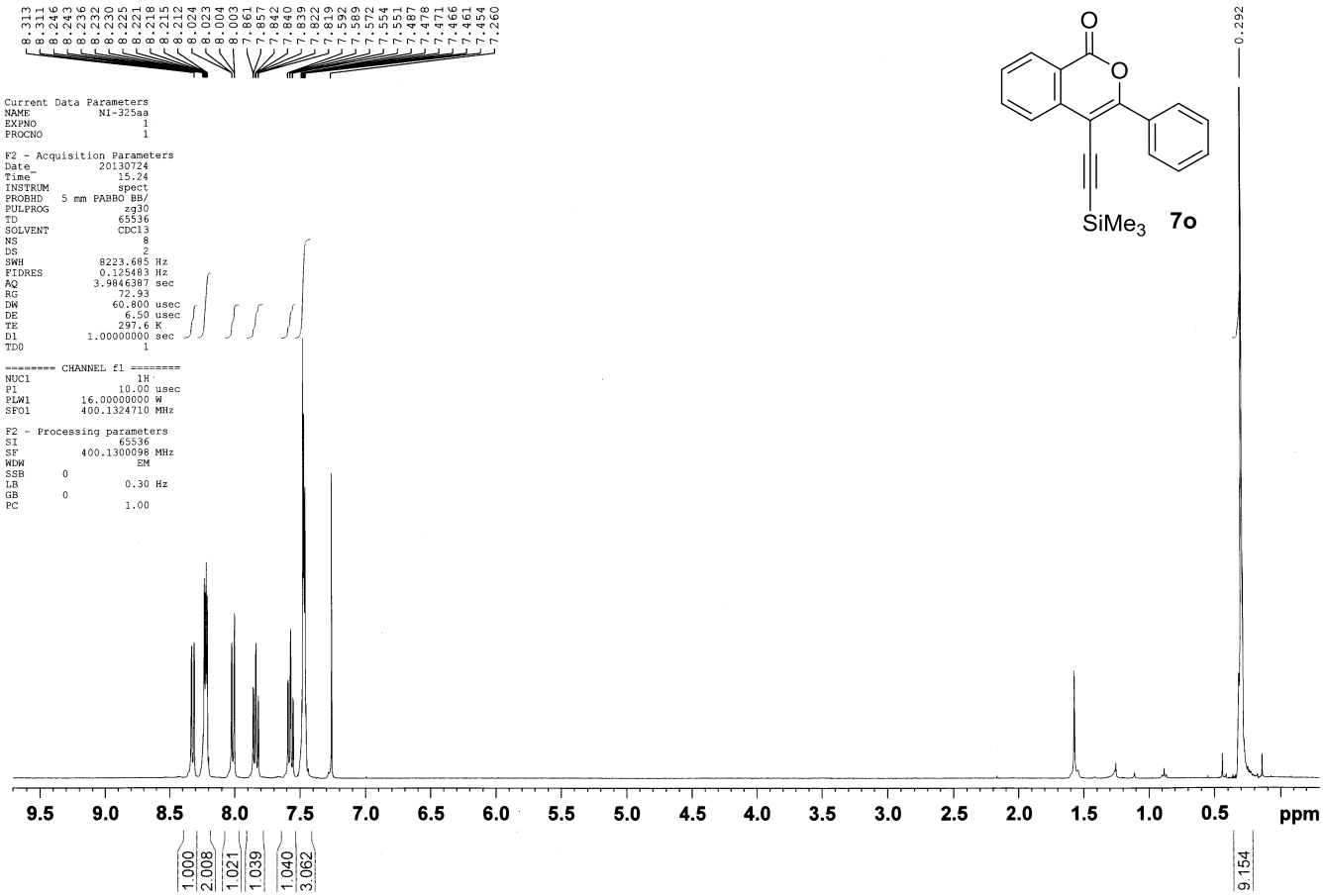
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz

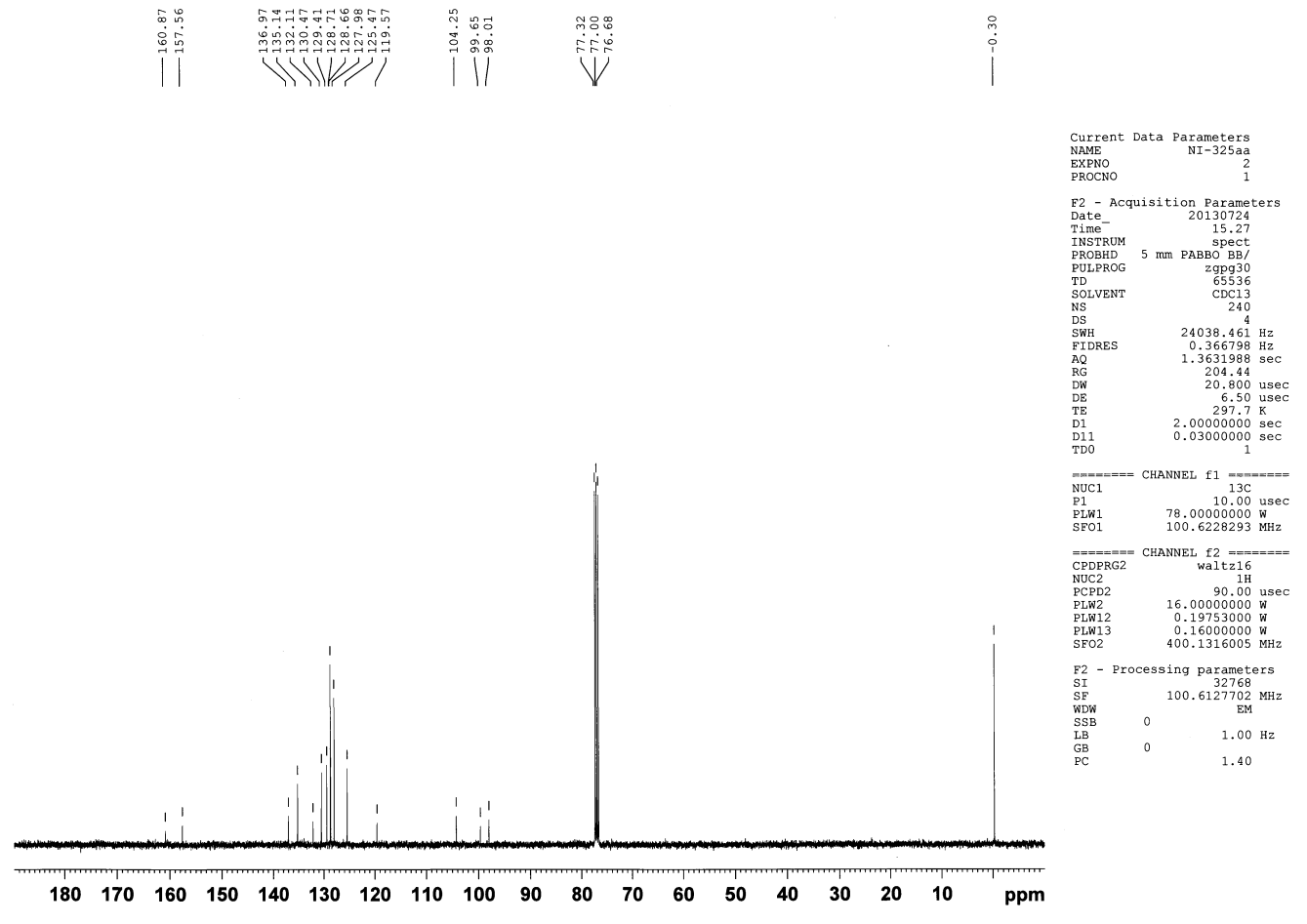
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz

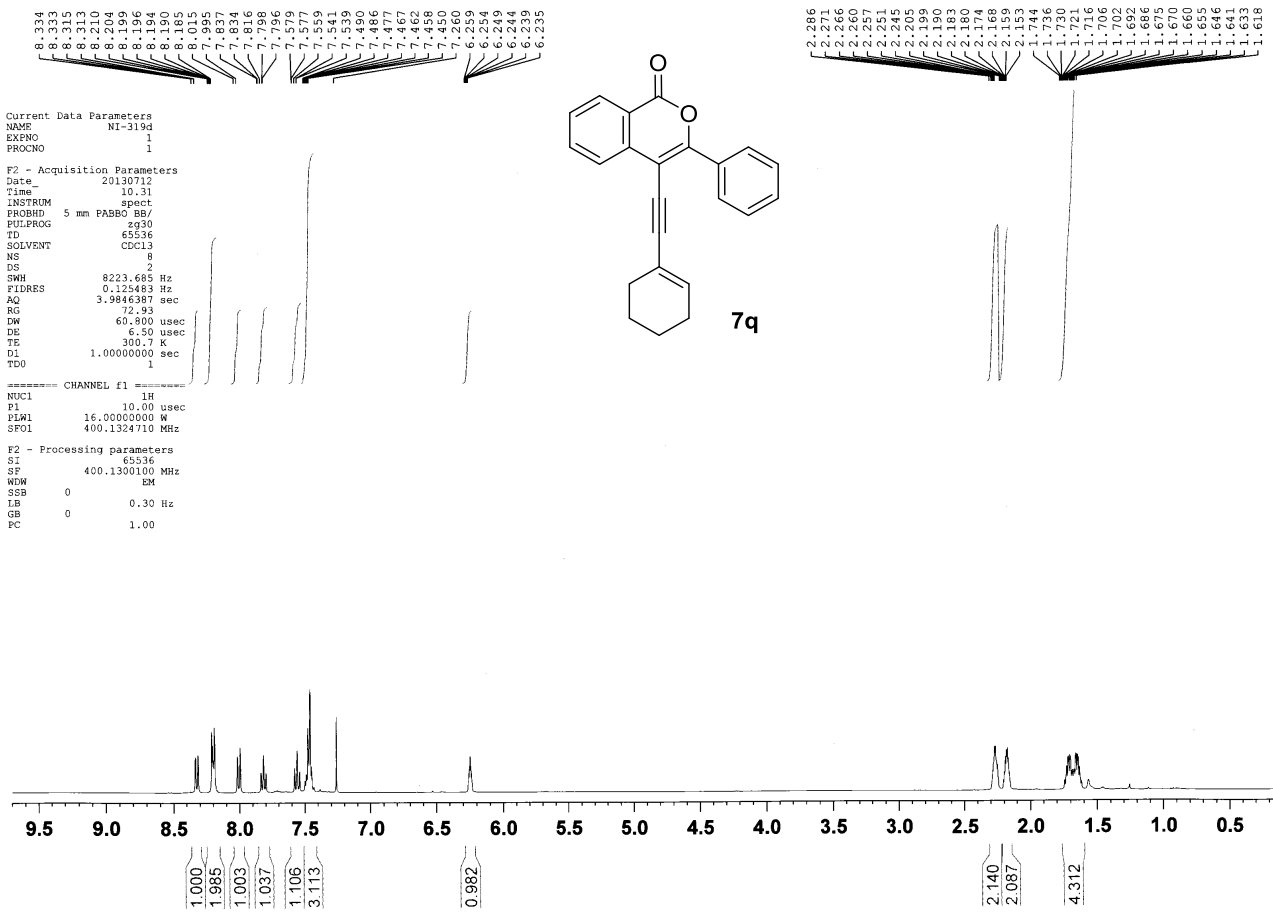
F2 - Processing parameters
SI 32768
SF 100.6127711 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

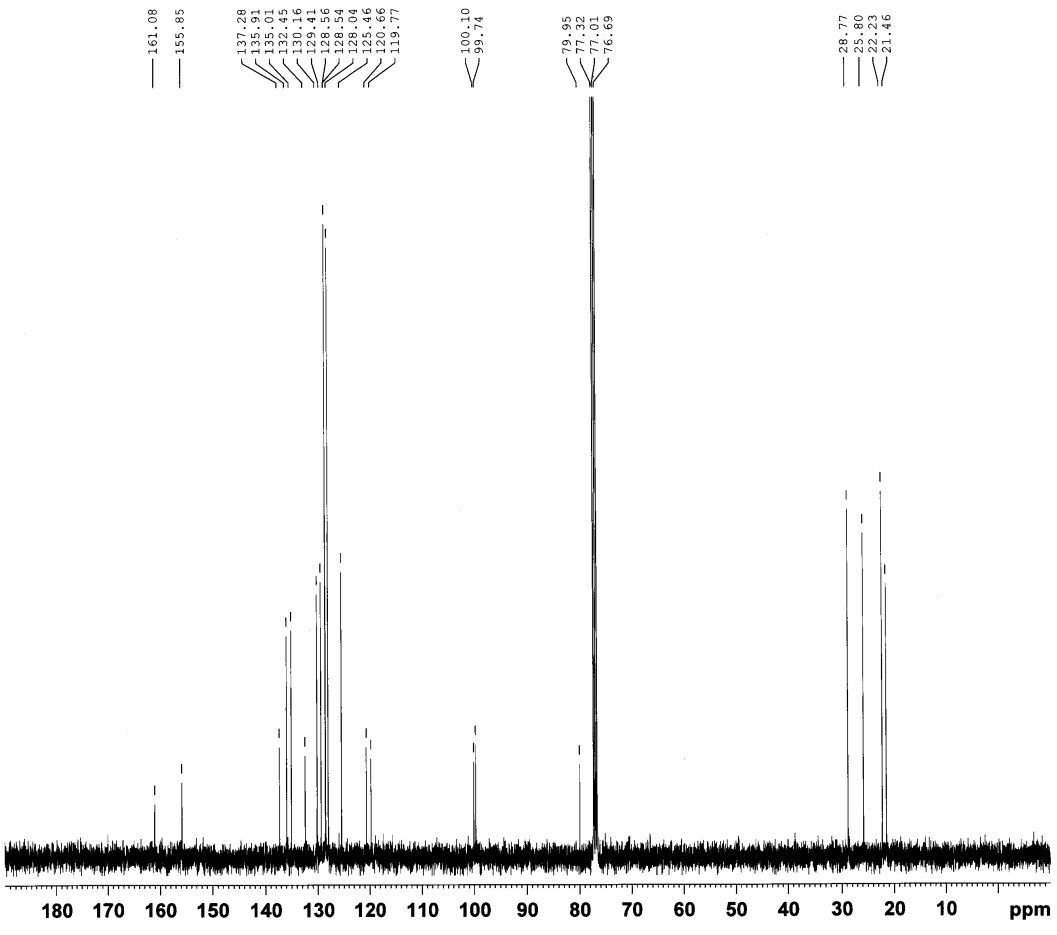

Current Data Parameters
NAME NI-486ba
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140717
Time 13.11
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 104.97
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1824713 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1800097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
8.327
8.325
8.307
8.189
8.181
8.178
8.169
8.164
8.041
8.021
7.812
7.810
7.576
7.574
7.556
7.538
7.536
7.473
7.468
7.460
7.455
7.260
4.690
3.799
3.782
3.765
3.366
2.841
2.824
2.808
O
O
MOMO
7n
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
0.808
1.553
0.776
0.832
0.855
2.429
1.628
1.638
2.389
1.636


161.10
156.43
137.49
135.00
132.38
130.19
129.37
128.54
128.07
125.49
119.71
99.82
96.55
95.77
77.33
77.01
76.69
74.58
65.80
55.38
21.40
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Current Data Parameters
NAME NI-486ba
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140717
Time 13.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 206.25
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
NUC1 13C
P1 10.00 usec
PLW1 63.50000000 W
SFO1 100.6354031 MHz
CPDPRG2
NUC2 1H
PLW2 16.50000000 W
PLW12 0.20370001 W
PLW13 0.16500001 W
SFO2 400.1816007 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


8.313
8.311
8.246
8.243
8.236
8.232
8.230
8.225
8.221
8.218
8.215
8.212
8.024
8.023
8.004
8.003
7.861
7.857
7.842
7.840
7.839
7.822
7.819
7.592
7.589
7.572
7.554
7.551
7.487
7.478
7.471
7.466
7.461
7.454
7.260
0.292
O
O
SiMe3
7o
Current Data Parameters
NAME NI-325aa
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130724
Time 15.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 72.93
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.000
2.008
1.021
1.039
1.040
3.062
9.154


160.87
157.56
136.97
135.14
132.11
130.47
129.41
128.71
128.66
127.98
125.47
119.57
104.25
99.65
98.01
77.32
77.00
76.68
-0.30
Current Data Parameters
NAME NI-325aa
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130724
Time 15.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 240
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127702 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm





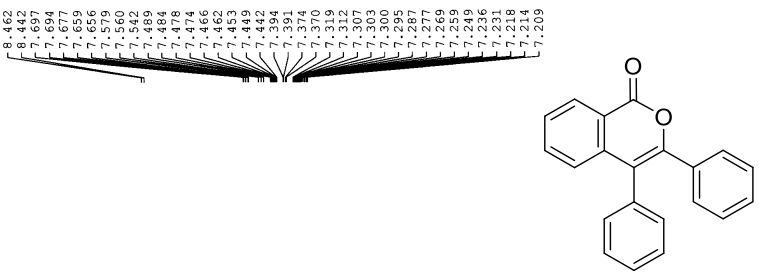
Current Data Parameters
NAME NI-319d
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20130712
Time 10.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 216
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 300.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

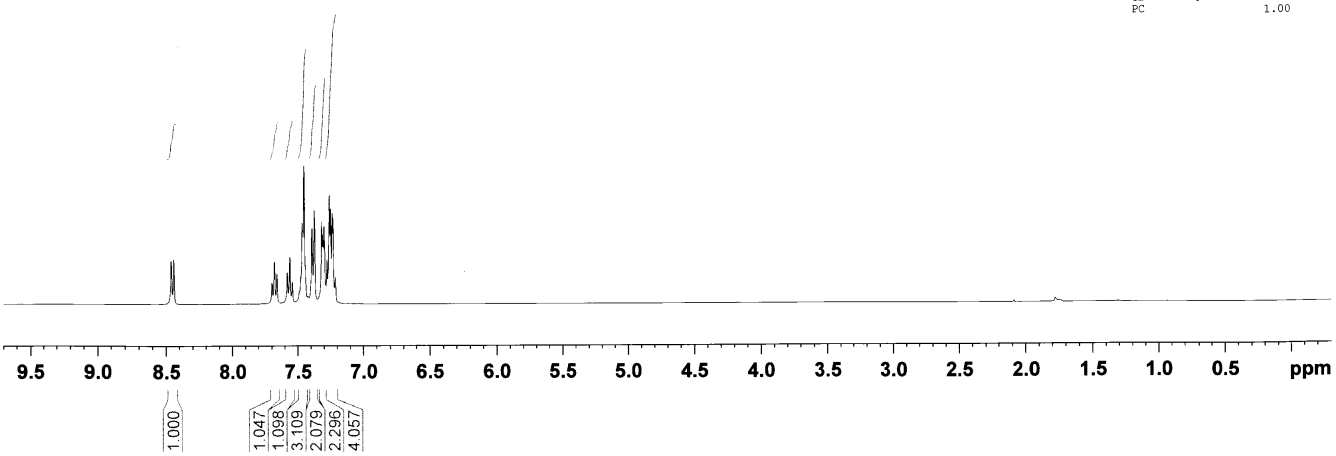
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz

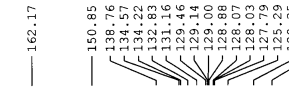
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz

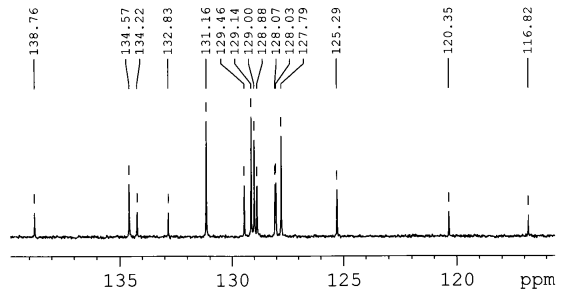
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40



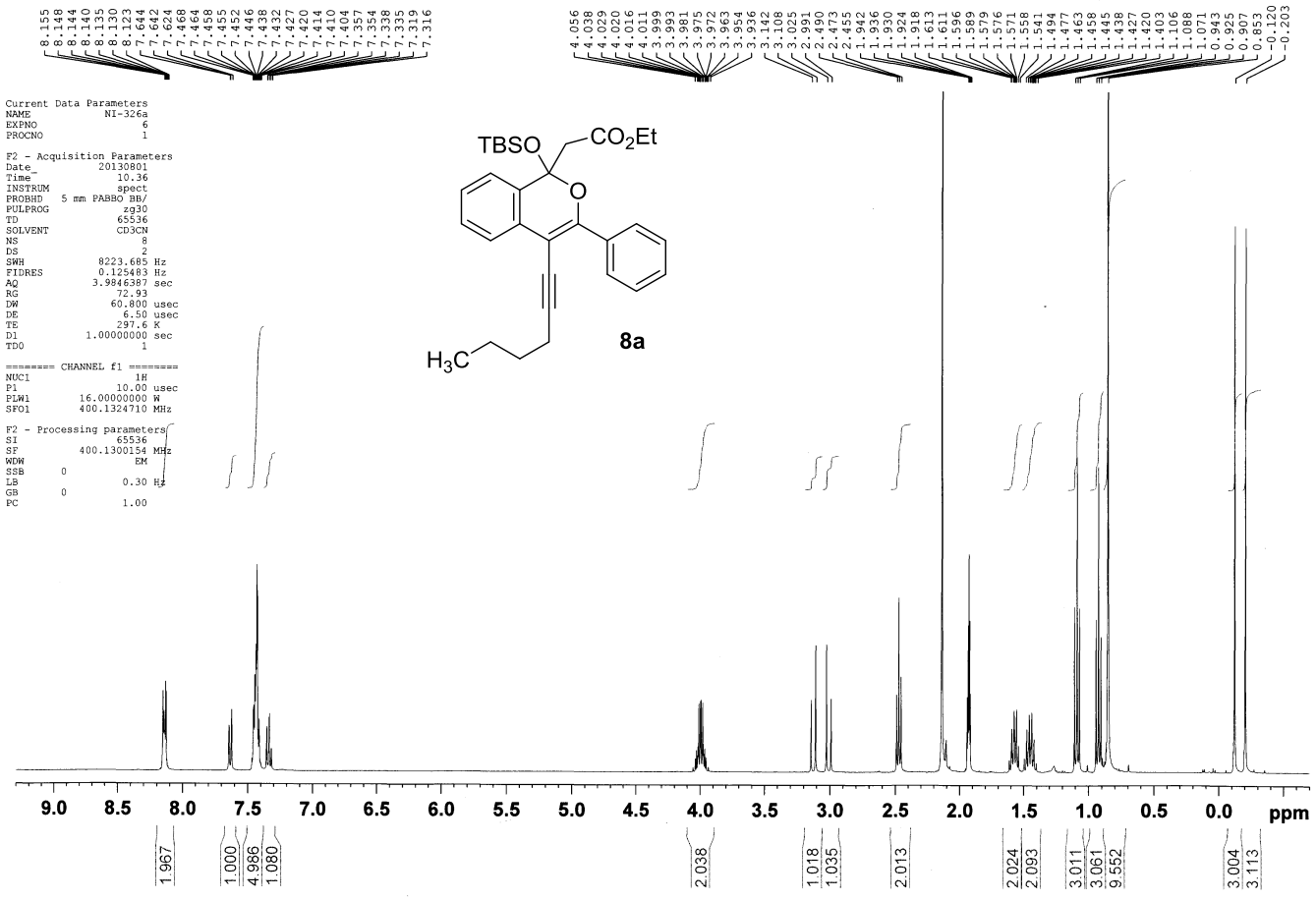

```
Current Data Parameters
NAME      HY-NI-suPhPh-sm
EXPNO                  1
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150420
Time              20.47
INSTRUM           spect
PROBHD  5 mm PABBO BB/
PULPROG            zg30
TD                65536
SOLVENT           CDCl3
NS                    8
DS                    2
SWH            8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG                33.06
DW               60.800 usec
DE                6.50 usec
TE                295.9 K
D1           1.00000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                  1H
P1                10.00 usec
PLW1        16.00000000 W
SFO1       400.1324710 MHz

F2 - Processing parameters
SI                65536
SF           400.1299922 MHz
WDW                  EM
SSB       0
LB                0.30 Hz
GB        0
PC                1.00
```

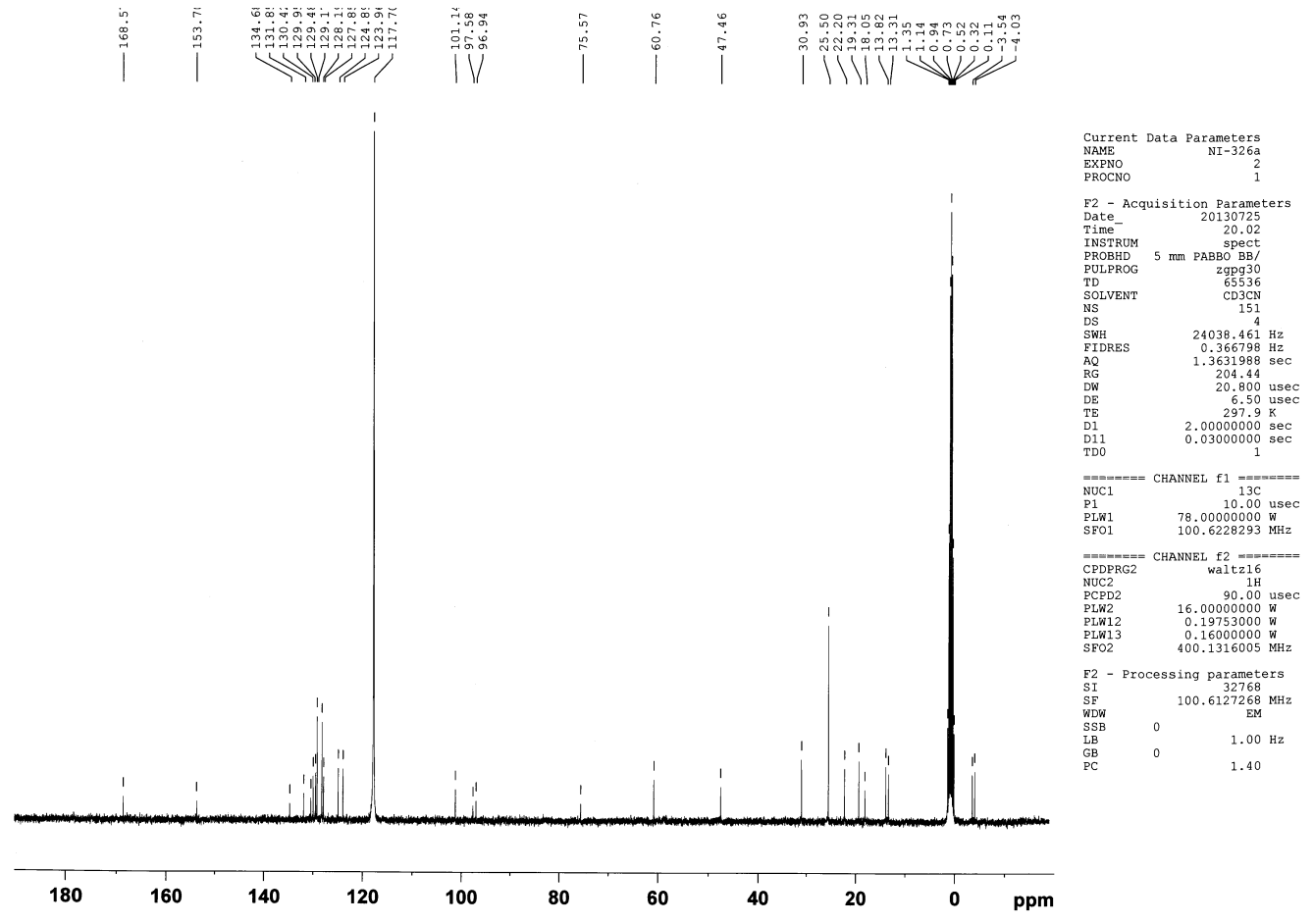


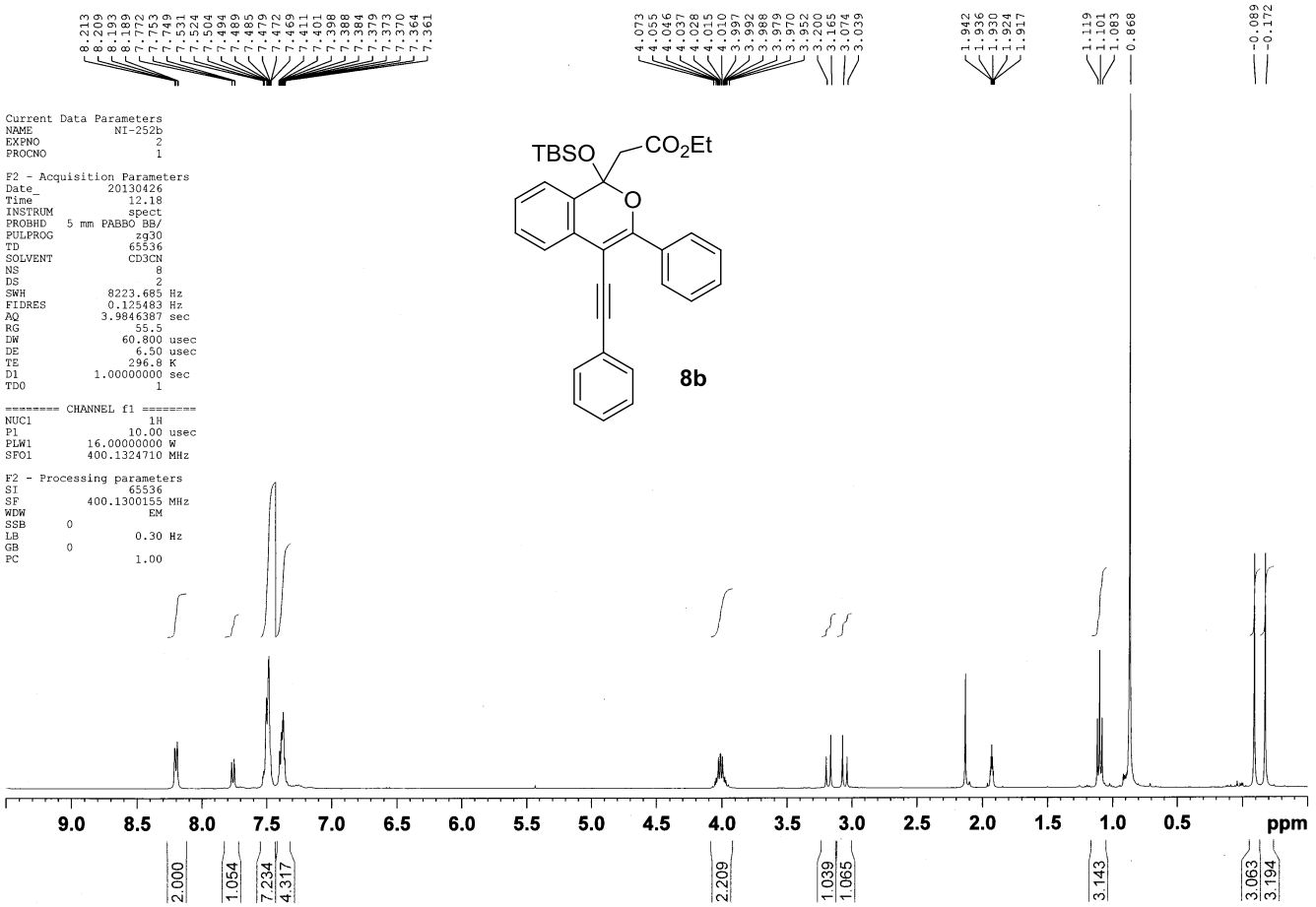




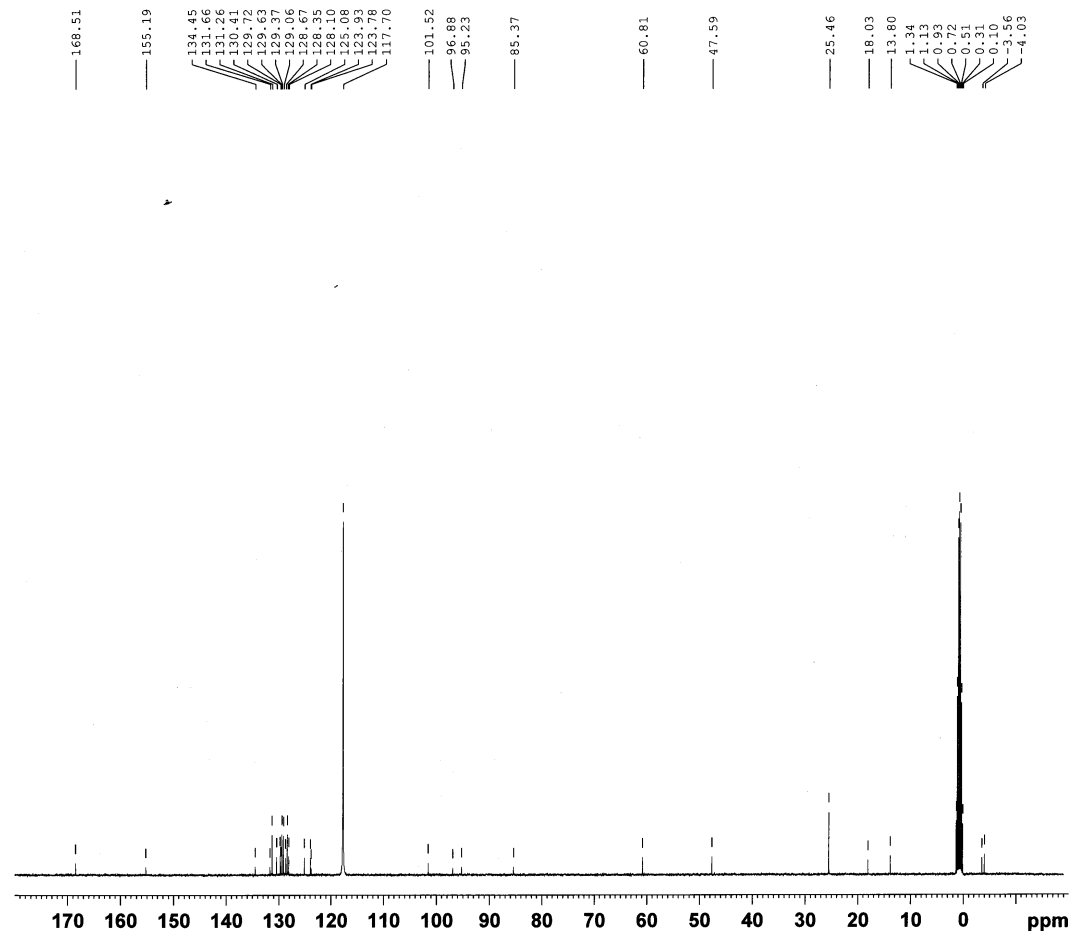

```
Current Data Parameters
NAME      HY-NI-suPhPh-sm
EXPNO                  2
PROCNO                 1

F2 - Acquisition Parameters
Date_           20150420
Time              20.40
INSTRUM           spect
PROBHD  5 mm PABBO BB/
PULPROG           zgpg30
TD                65536
SOLVENT           CDCl3
NS                   34
DS                    4
SWH           24038.461 Hz
FIDRES        0.366798 Hz
AQ            1.3631988 sec
RG                204.44
DW               20.800 usec
DE                6.50 usec
TE                295.9 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                   1

======== CHANNEL f1 ========
NUC1                 13C
P1                10.00 usec
PLW1        78.00000000 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2             90.00 usec
PLW2        16.00000000 W
PLW12        0.19753000 W
PLW13        0.16000000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                32768
SF           100.6127780 MHz
WDW                  EM
SSB       0
LB                1.00 Hz
GB        0
PC                1.40
```


8.155
8.148
8.144
8.140
8.135
8.130
8.123
7.644
7.642
7.624
7.468
7.464
7.458
7.455
7.452
7.446
7.438
7.432
7.427
7.420
7.414
7.410
7.404
7.357
7.354
7.338
7.335
7.319
7.316
4.056
4.038
4.029
4.020
4.016
4.011
3.999
3.993
3.981
3.975
3.972
3.963
3.954
3.936
3.142
3.108
3.025
2.991
2.490
2.473
2.455
1.942
1.936
1.930
1.924
1.918
1.613
1.611
1.596
1.589
1.579
1.576
1.571
1.558
1.541
1.494
1.477
1.463
1.458
1.445
1.438
1.427
1.420
1.403
1.106
1.088
1.071
0.943
0.925
0.907
0.853
-0.120
-0.203
Current Data Parameters
NAME NI-326a
EXPNO 6
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130801
Time_ 10.36
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 72.93
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
H3C
8a
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
0.0
ppm
1.967
1.000
4.986
1.080
2.038
1.018
1.035
2.013
2.024
2.093
3.011
3.061
9.552
3.004
3.113

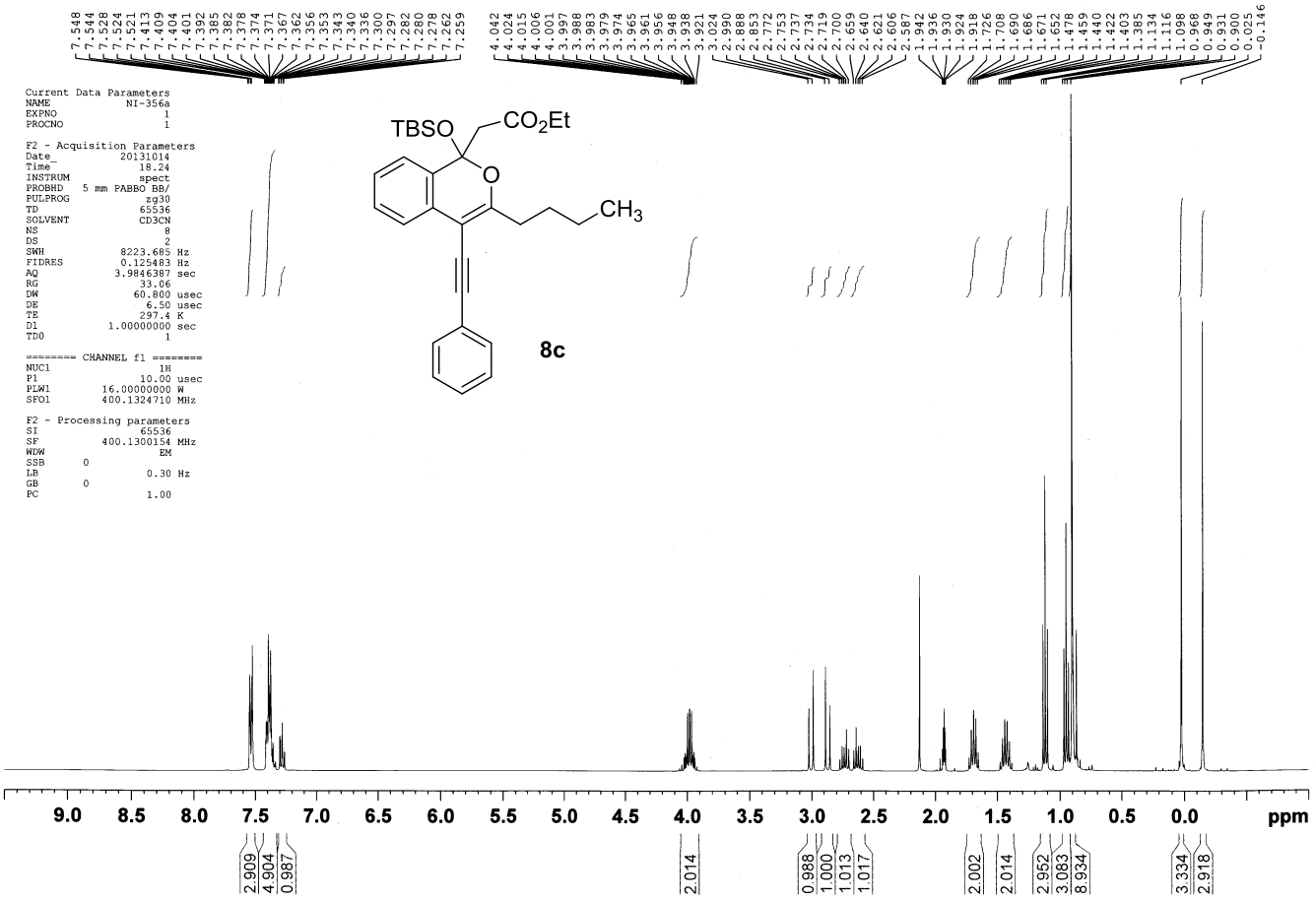

168.5
153.7
134.6
131.8
130.4
129.9
129.4
129.1
128.1
127.8
124.8
123.9
117.7
101.1
97.58
96.94
75.57
60.76
47.46
30.93
25.50
22.20
19.31
18.05
13.82
13.31
1.35
1.14
0.94
0.73
0.52
0.32
0.11
-3.54
-4.03
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME NI-326a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130725
Time_ 20.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 151
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127268 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

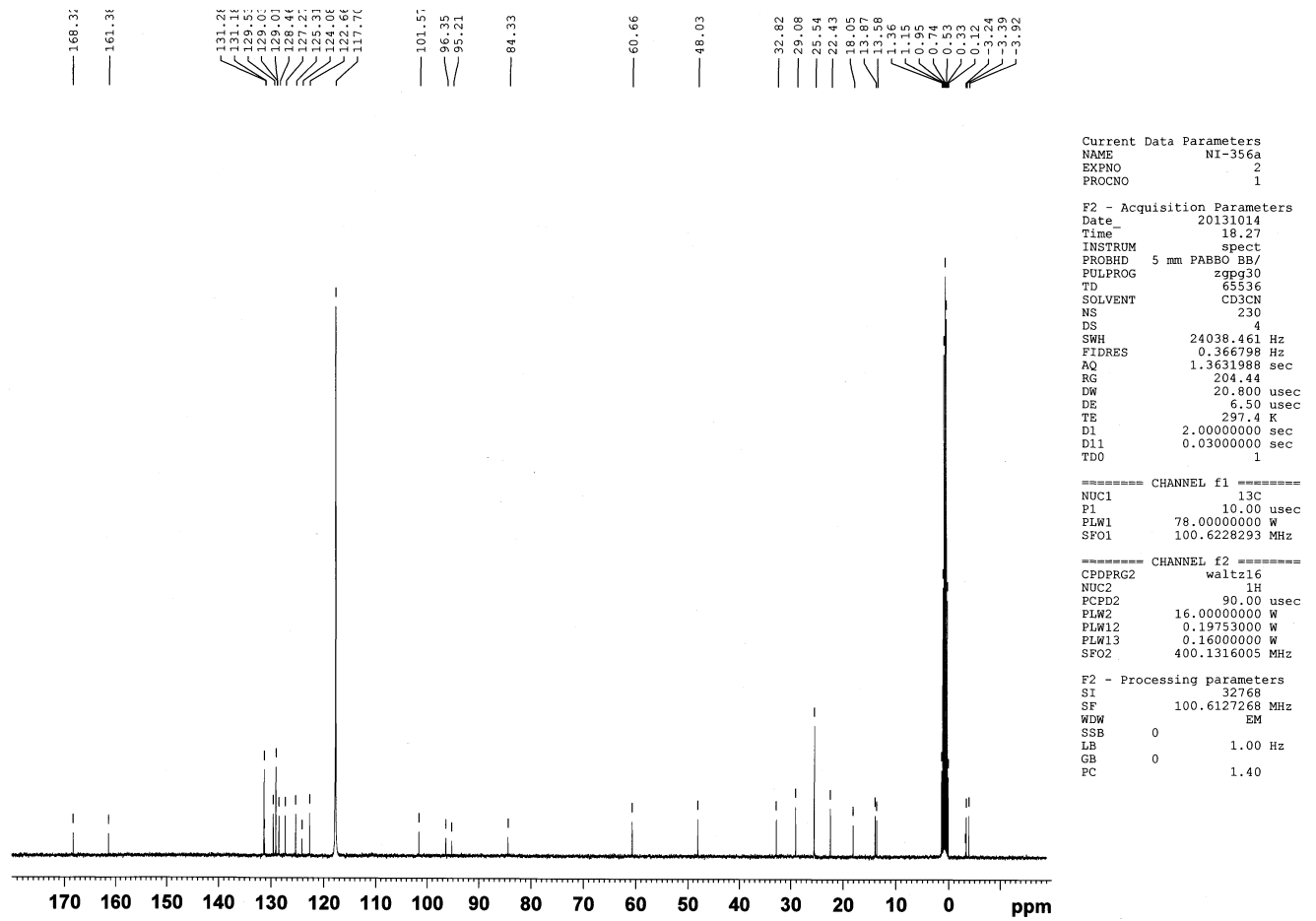


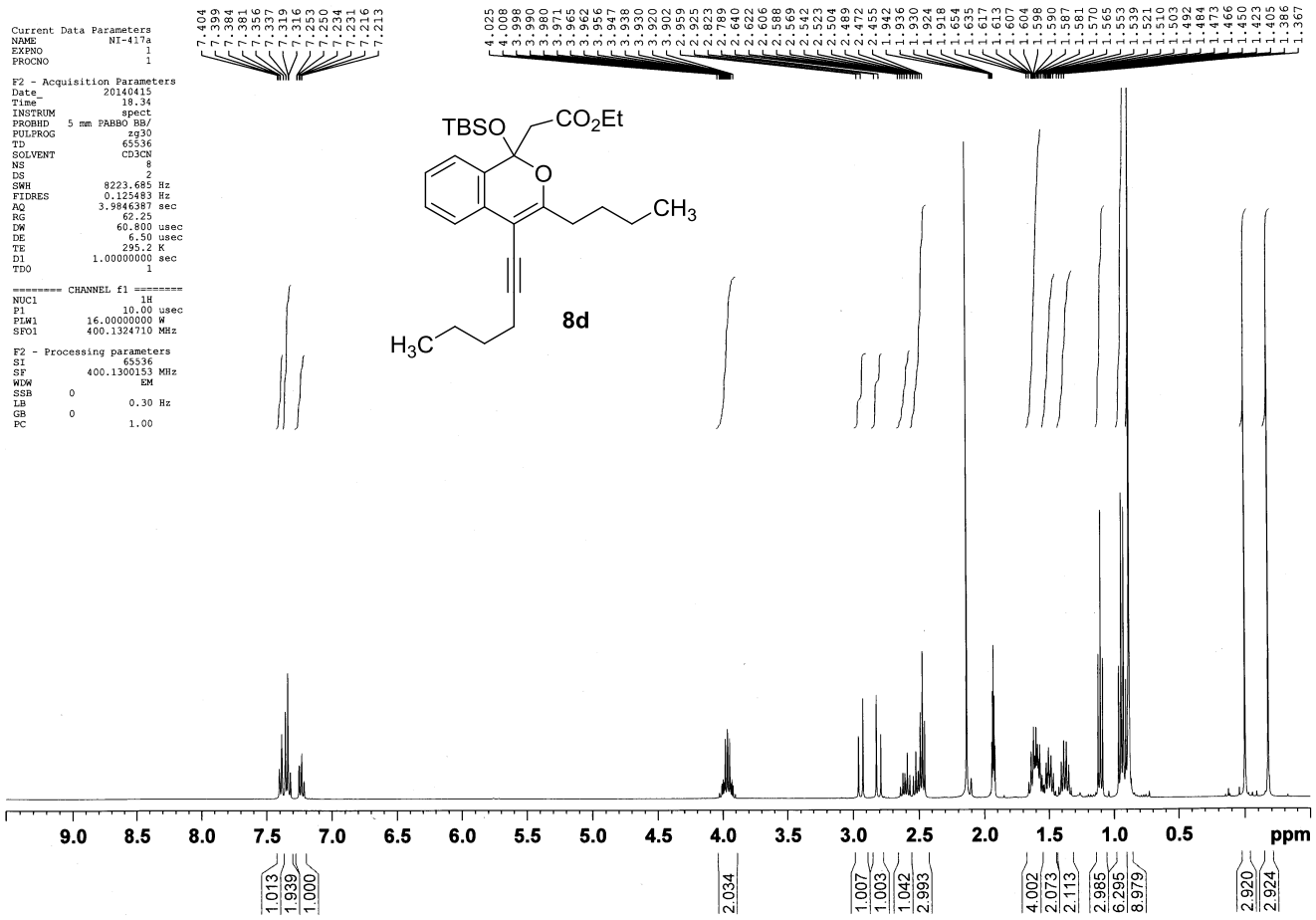

```
Current Data Parameters
NAME                  NI-252b
EXPNO                       2
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130426
Time                  12.18
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG                zg30
TD                    65536
SOLVENT               CD3CN
NS                        8
DS                        2
SWH                8223.685 Hz
FIDRES             0.125483 Hz
AQ                 3.9846387 sec
RG                      55.5
DW                    60.800 usec
DE                     6.50 usec
TE                    296.8 K
D1                 1.00000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                       1H
P1                      10.00 usec
PLW1               16.00000000 W
SFO1              400.1324710 MHz

F2 - Processing parameters
SI                        65536
SF                400.1300155 MHz
WDW                          EM
SSB             0
LB                      0.30 Hz
GB              0
PC                        1.00
```

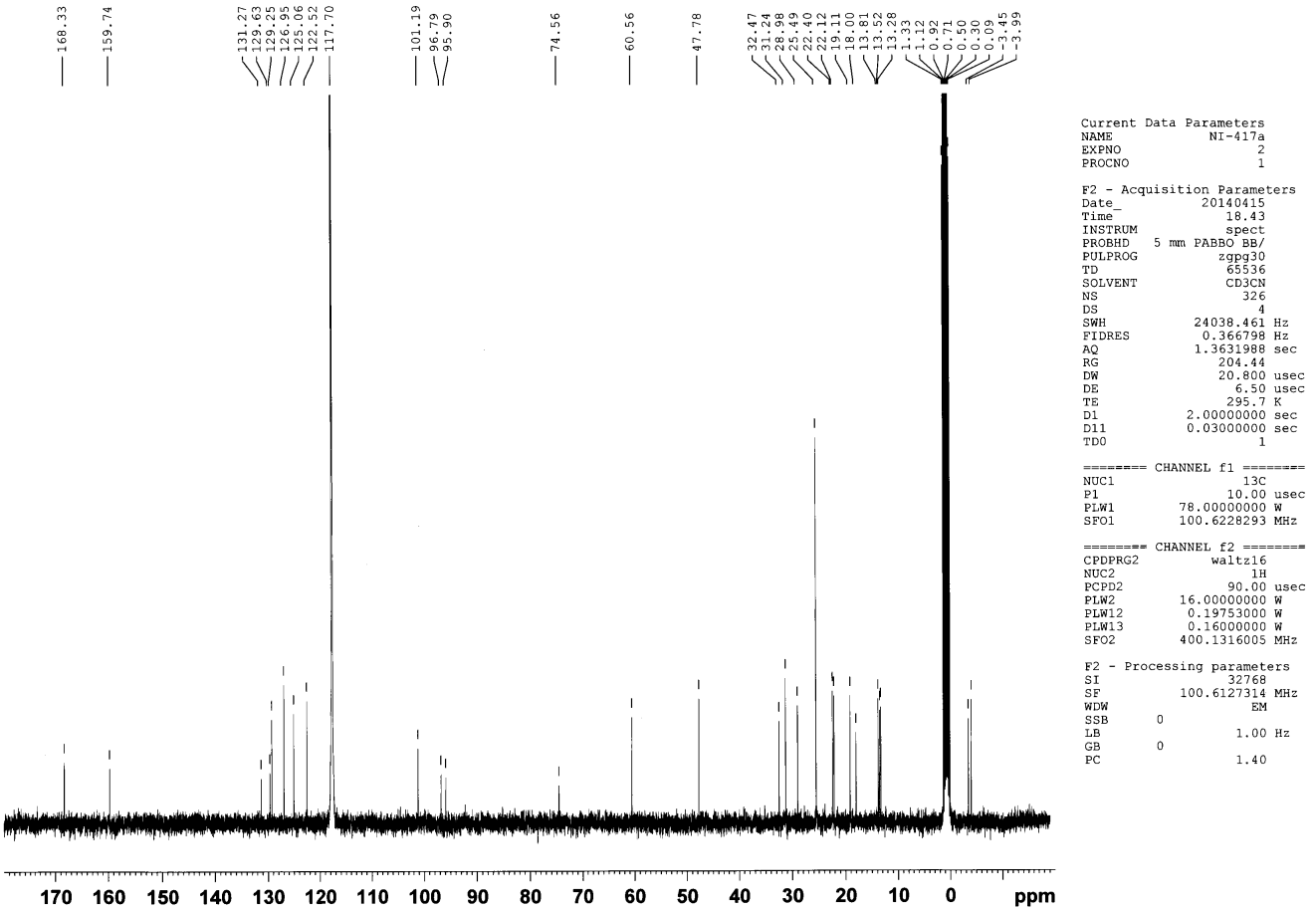


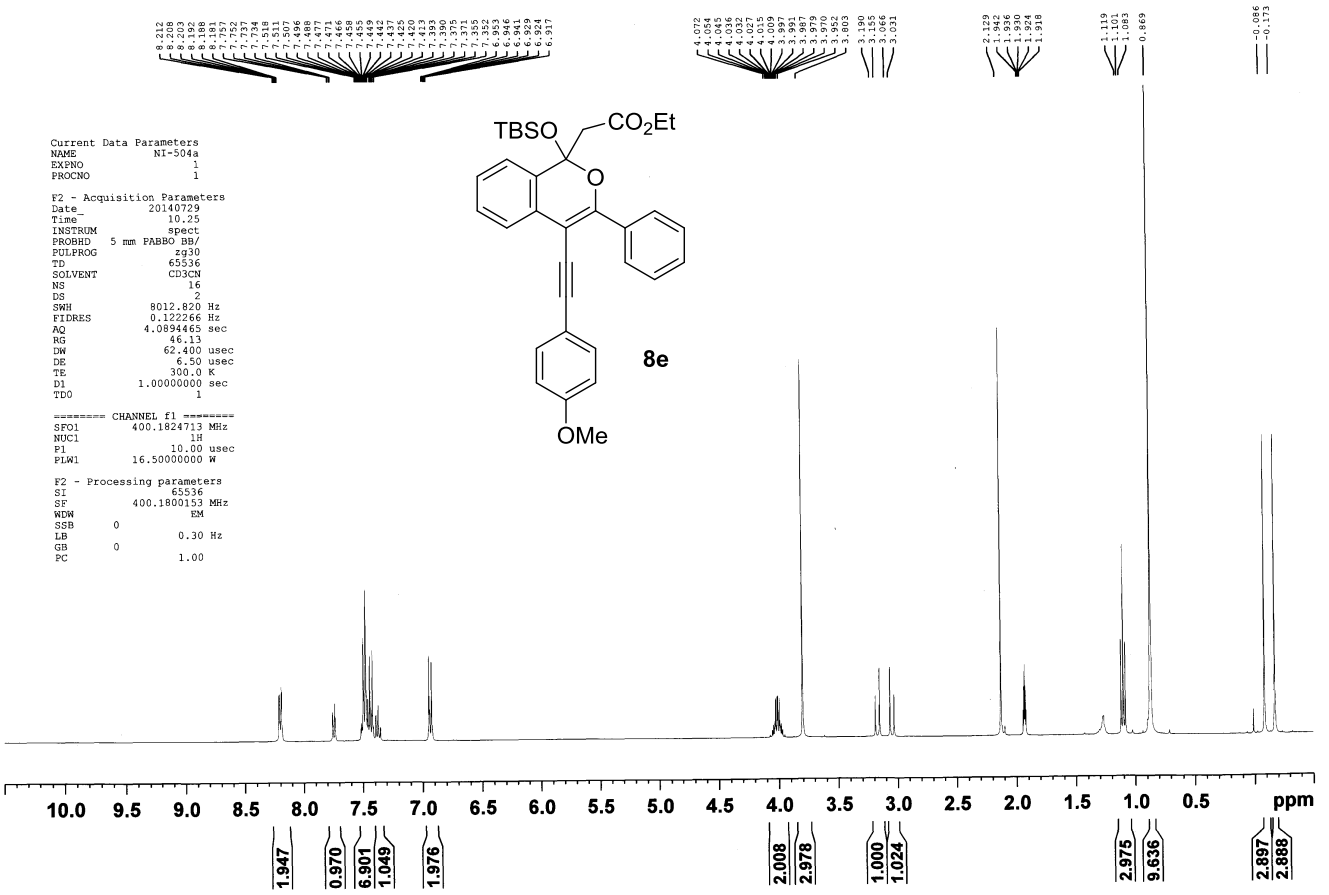

```
Current Data Parameters
NAME                  NI-252b
EXPNO                       3
PROCNO                      1

F2 - Acquisition Parameters
Date_              20130426
Time                  12.26
INSTRUM               spect
PROBHD   5 mm PABBO BB/
PULPROG               zgpg30
TD                    65536
SOLVENT               CD3CN
NS                      303
DS                        4
SWH               24038.461 Hz
FIDRES             0.366798 Hz
AQ                 1.3631988 sec
RG                    204.44
DW                    20.800 usec
DE                     6.50 usec
TE                    297.3 K
D1                 2.00000000 sec
D11                0.03000000 sec
TD0                         1

======== CHANNEL f1 ========
NUC1                      13C
P1                      10.00 usec
PLW1               78.00000000 W
SFO1              100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2             waltz16
NUC2                       1H
PCPD2                  80.00 usec
PLW2               7.09999990 W
PLW12              0.24961001 W
PLW13              0.15975000 W
SFO2              400.1316005 MHz

F2 - Processing parameters
SI                        32768
SF                100.6127300 MHz
WDW                          EM
SSB             0
LB                      1.00 Hz
GB              0
PC                        1.40
```

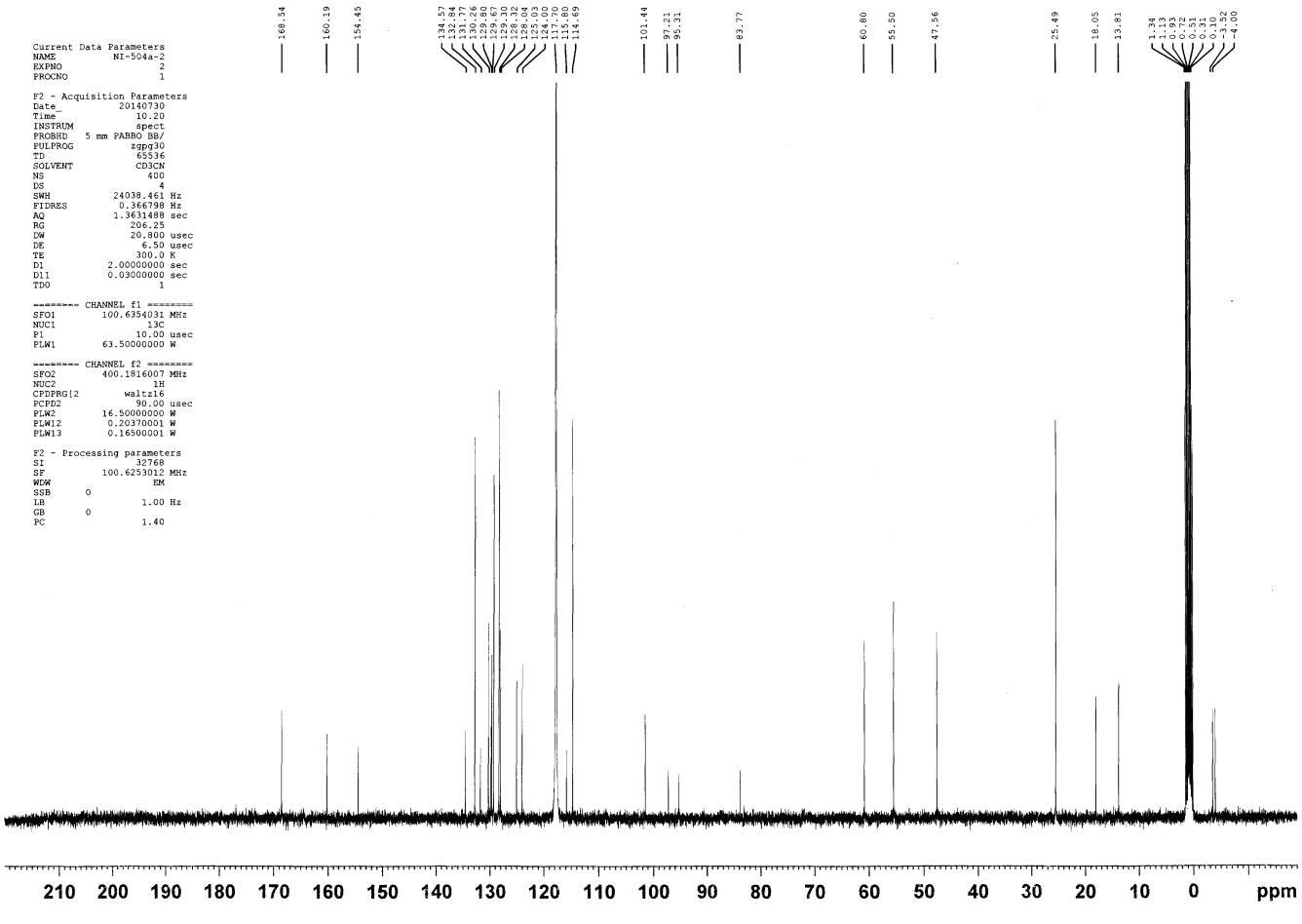

Current Data Parameters
NAME NI-356a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131014
Time 18.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 297.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
CH3
8c
ppm

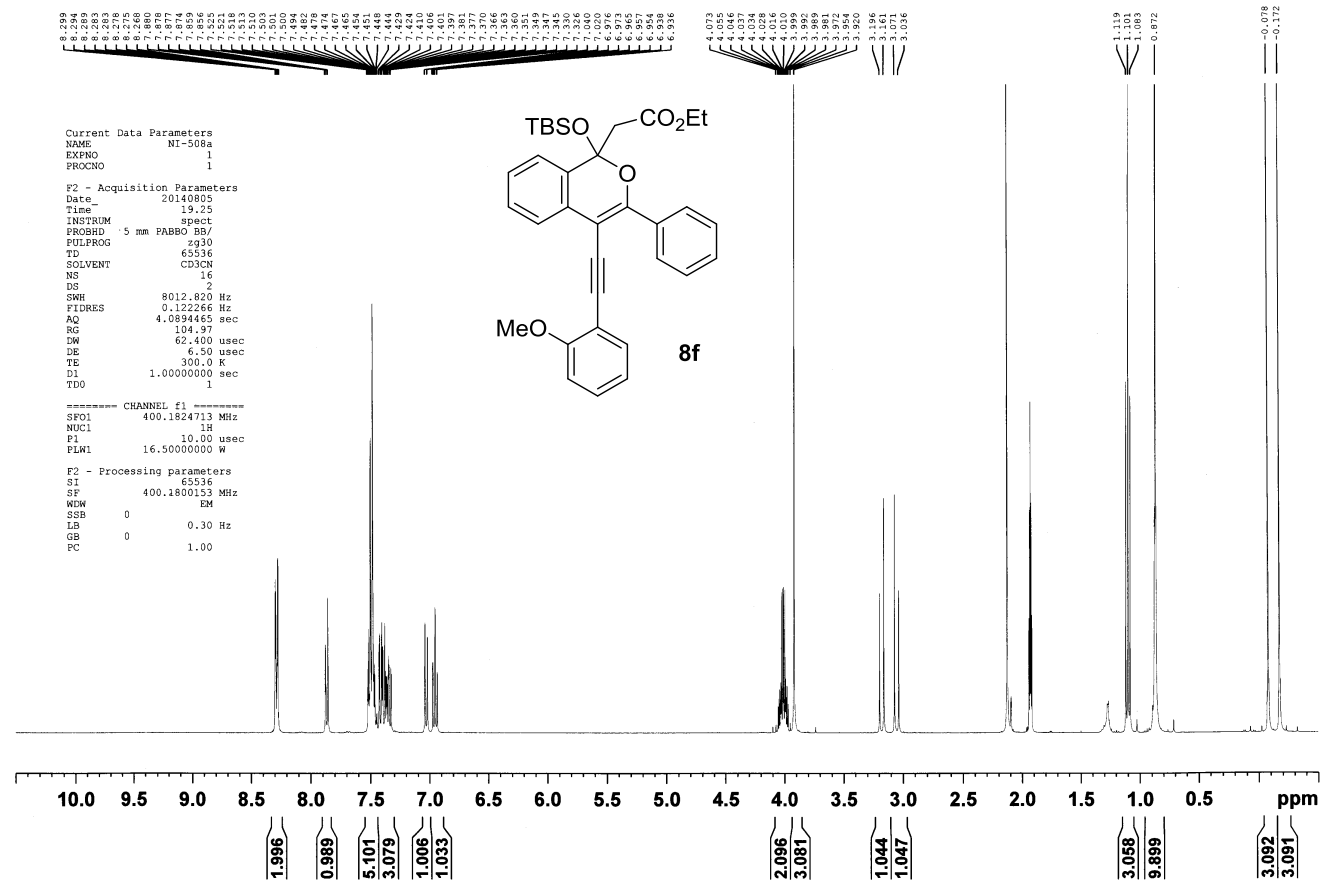

Current Data Parameters
NAME NI-356a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131014
Time 18.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 230
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127268 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
ppm

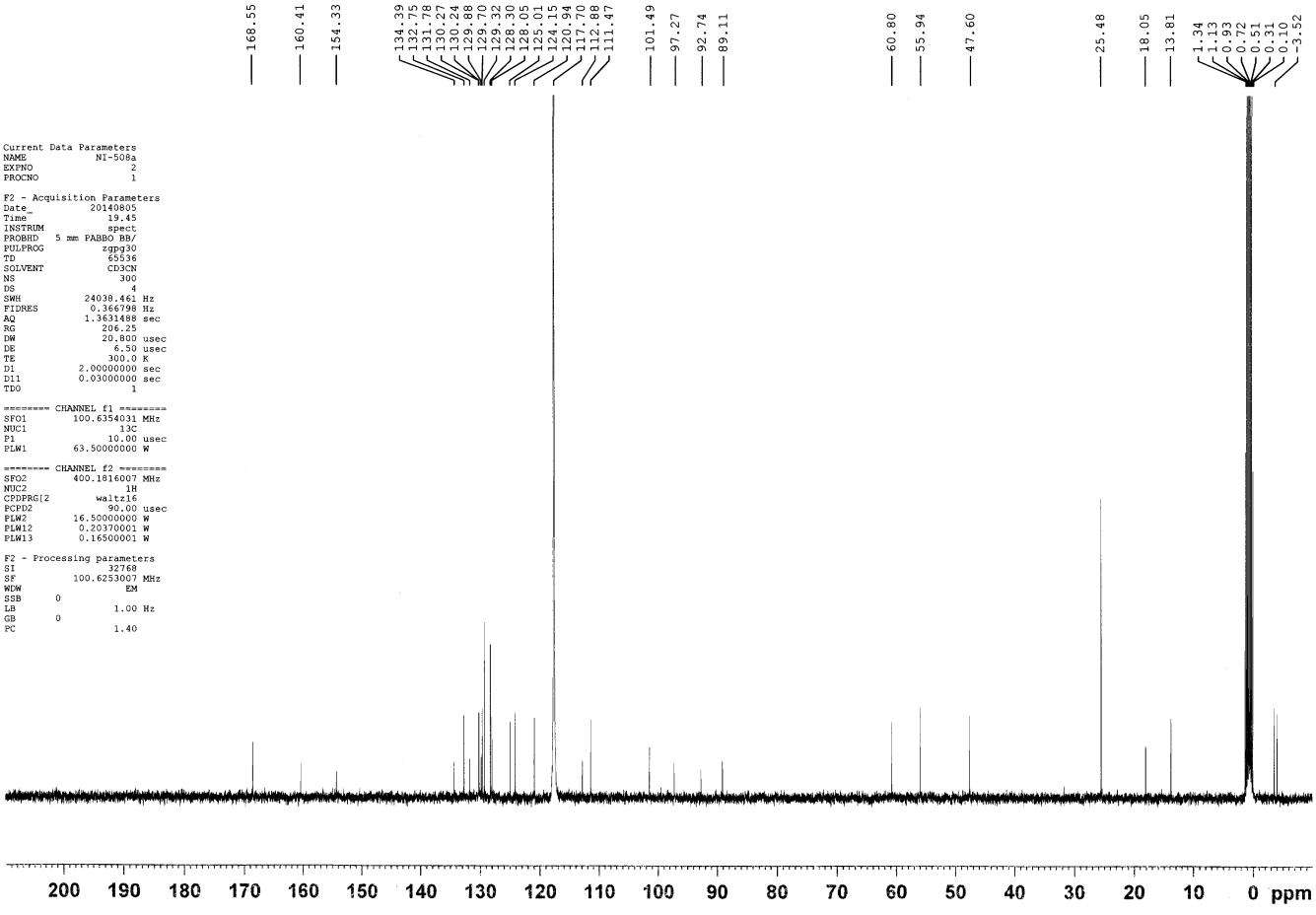

Current Data Parameters
NAME NI-417a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140415
Time 18.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 62.25
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
CH3
H3C
8d
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

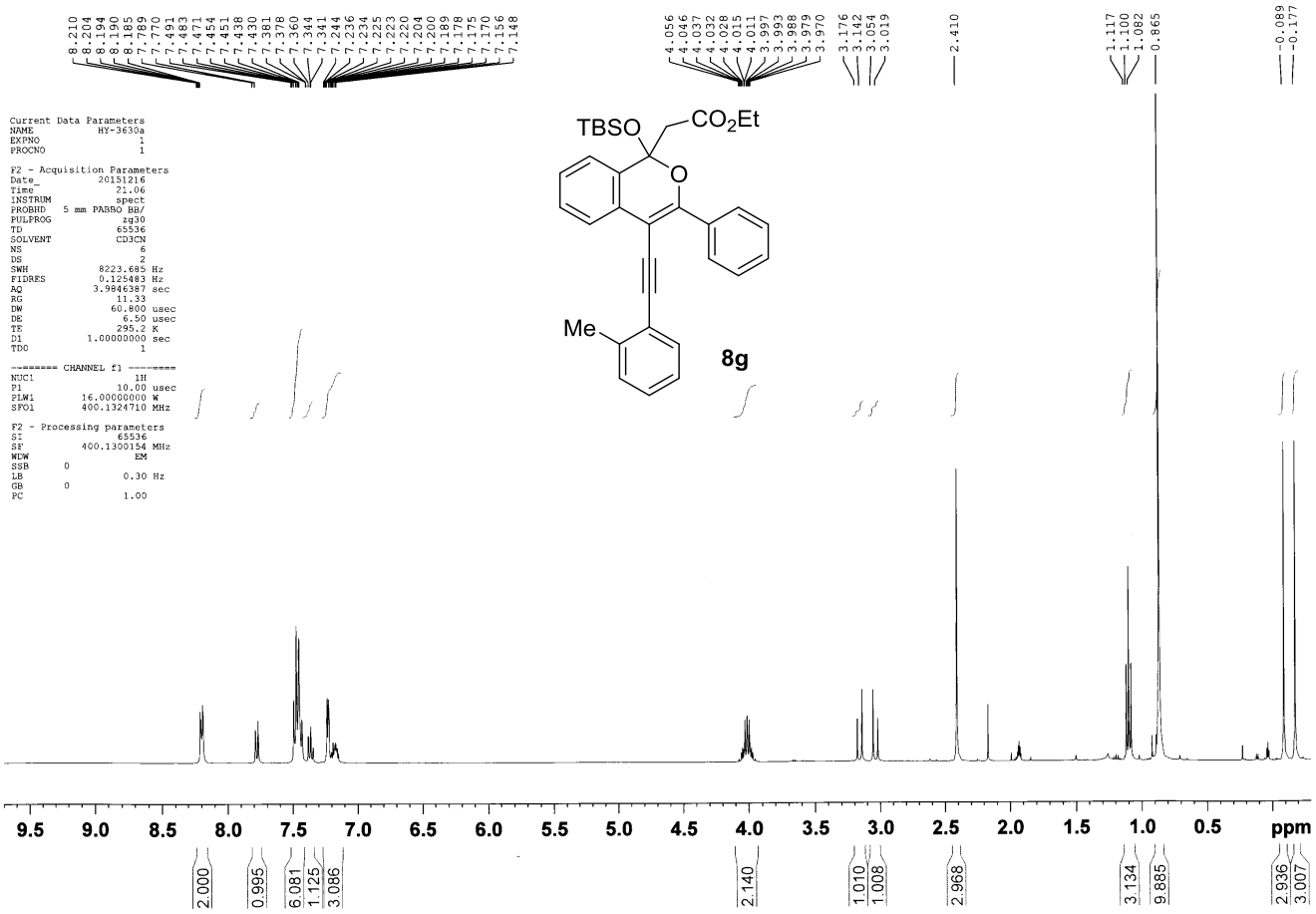

Current Data Parameters
NAME NI-417a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140415
Time 18.43
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 326
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127314 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

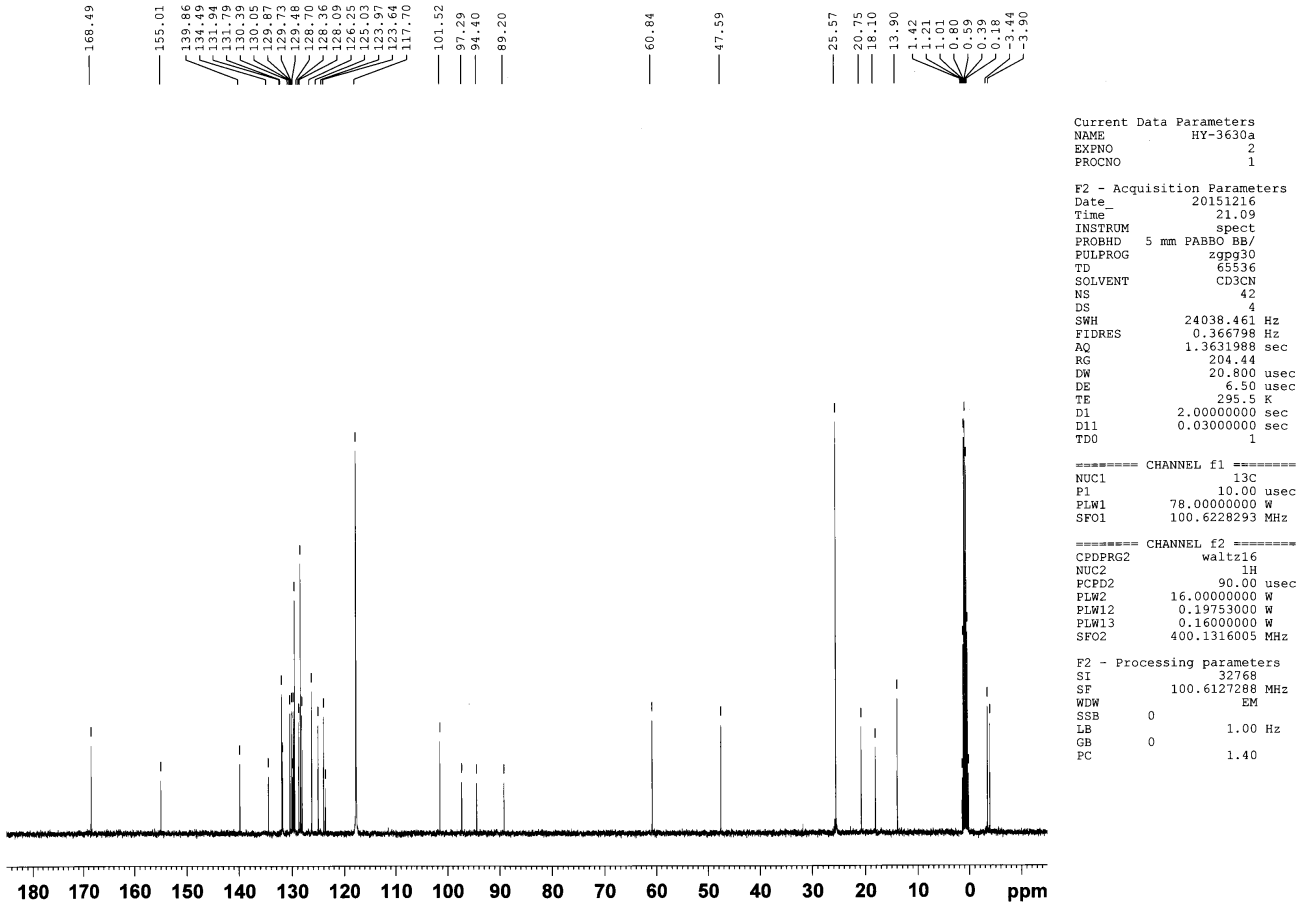

Current Data Parameters
NAME NI-504a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140729
Time_ 10.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 46.13
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1824713 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1800153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
OMe
8e
8.212 8.208 8.203 8.192 8.188 8.181 7.757 7.752 7.737 7.734 7.518 7.511 7.507 7.496 7.488 7.477 7.471 7.466 7.458 7.455 7.449 7.442 7.437 7.425 7.420 7.413 7.393 7.380 7.375 7.371 7.355 7.352 6.953 6.946 6.941 6.929 6.924 6.917
4.072 4.054 4.045 4.036 4.032 4.022 4.015 4.009 3.997 3.987 3.981 3.979 3.970 3.962 3.803 3.190 3.155 3.066 3.031
2.129 1.942 1.936 1.930 1.924 1.918
1.119 1.101 1.083 0.869
-0.086 -0.173
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.947 0.970 6.901 1.049 1.976 2.008 2.978 1.000 1.024 2.975 9.636 2.897 2.888

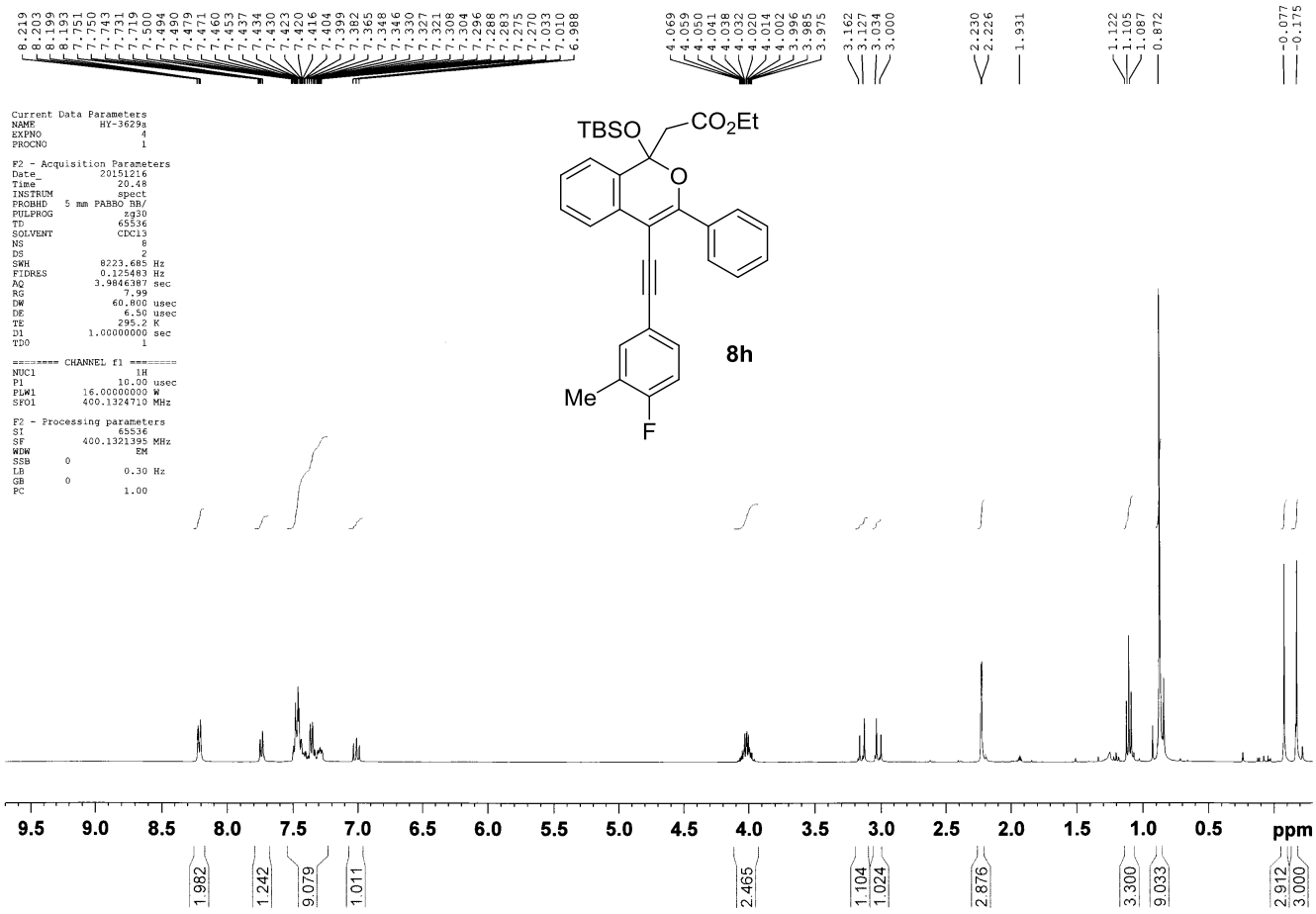

Current Data Parameters
NAME NI-504a-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140730
Time_ 10.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 400
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 206.25
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6354031 MHz
NUC1 13C
P1 10.00 usec
PLW1 63.50000000 W
======== CHANNEL f2 ========
SFO2 400.1816007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.50000000 W
PLW12 0.20370001 W
PLW13 0.16500001 W
F2 - Processing parameters
SI 32768
SF 100.6253012 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
168.54 160.19 154.45 134.57 132.84 131.77 130.26 129.80 129.67 129.30 128.32 128.04 125.03 124.00 117.70 115.80 114.69 101.44 97.21 95.31 83.77 60.80 55.50 47.56 25.49 18.05 13.81 1.34 1.13 0.93 0.72 0.31 0.21 0.10 -3.52 -4.00
210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm


TBSO
CO2Et
O
MeO
8f
Current Data Parameters
NAME NI-508a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140805
Time 19.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 104.97
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1824713 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1800153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
1.996
0.989
5.101
3.079
1.006
1.033
2.096
3.081
1.044
1.047
3.058
9.899
3.092
3.091
ppm

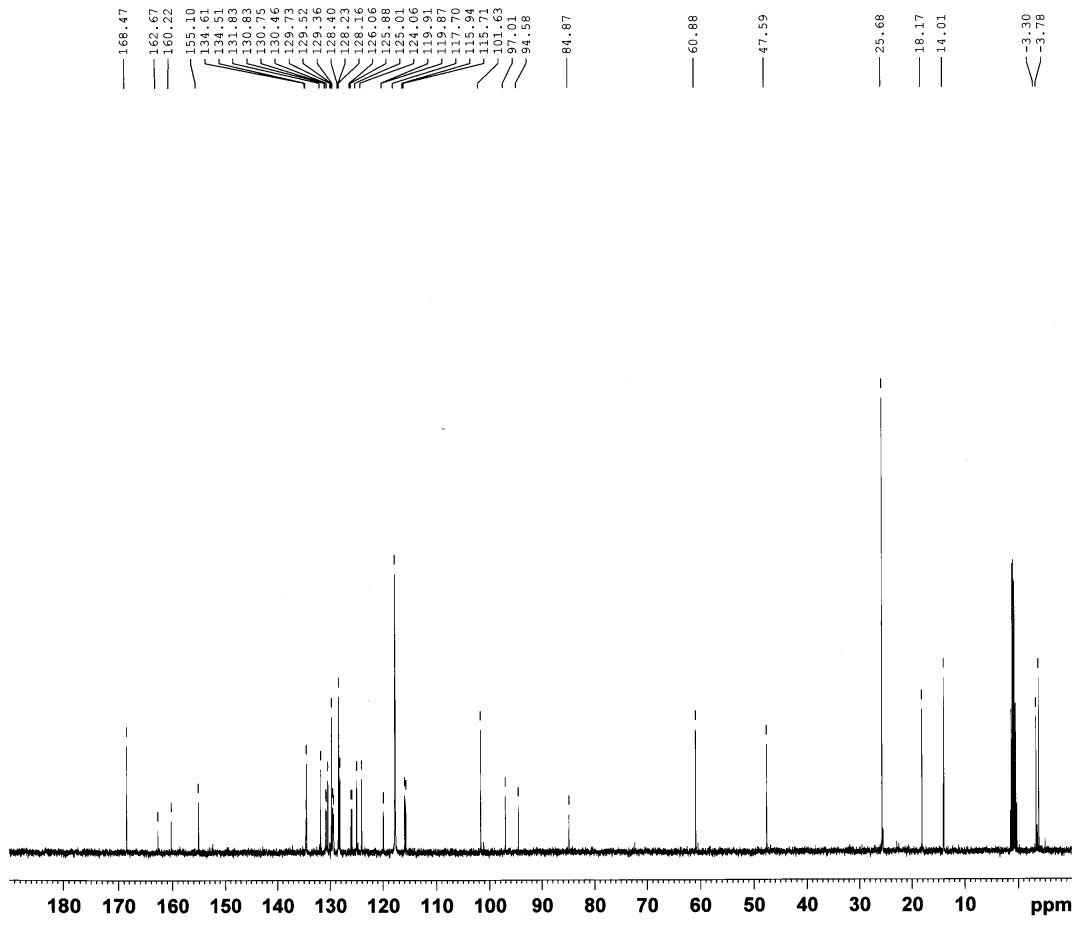

Current Data Parameters
NAME NI-508a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140805
Time 19.45
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 206.25
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6354031 MHz
NUC1 13C
P1 10.00 usec
PLW1 63.50000000 W
======== CHANNEL f2 ========
SFO2 400.1816007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.50000000 W
PLW12 0.20370001 W
PLW13 0.16500001 W
F2 - Processing parameters
SI 32768
SF 100.6253007 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
168.55
160.41
154.33
134.39
132.75
131.78
130.27
130.24
129.88
129.70
129.32
128.30
128.05
125.01
124.15
120.94
117.70
112.88
111.47
101.49
97.27
92.74
89.11
60.80
55.94
47.60
25.48
18.05
13.81
1.34
1.13
0.93
0.72
0.51
0.31
0.10
-3.52
ppm


8.210
8.204
8.194
8.190
8.185
7.789
7.770
7.491
7.483
7.471
7.454
7.451
7.438
7.430
7.381
7.378
7.360
7.344
7.341
7.244
7.236
7.234
7.225
7.223
7.220
7.204
7.200
7.189
7.178
7.175
7.170
7.156
7.148
4.056
4.046
4.037
4.032
4.028
4.015
4.011
3.997
3.993
3.988
3.979
3.970
3.176
3.142
3.054
3.019
2.410
1.117
1.100
1.082
0.865
0.089
0.177
TBSO
CO2Et
O
Me
8g
Current Data Parameters
NAME HY-3630a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151216
Time 21.06
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 6
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 11.33
DW 60.800 usec
DE 6.50 usec
TE 295.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.000
0.995
6.081
1.125
3.086
2.140
1.010
1.008
2.968
3.134
9.885
2.936
3.007

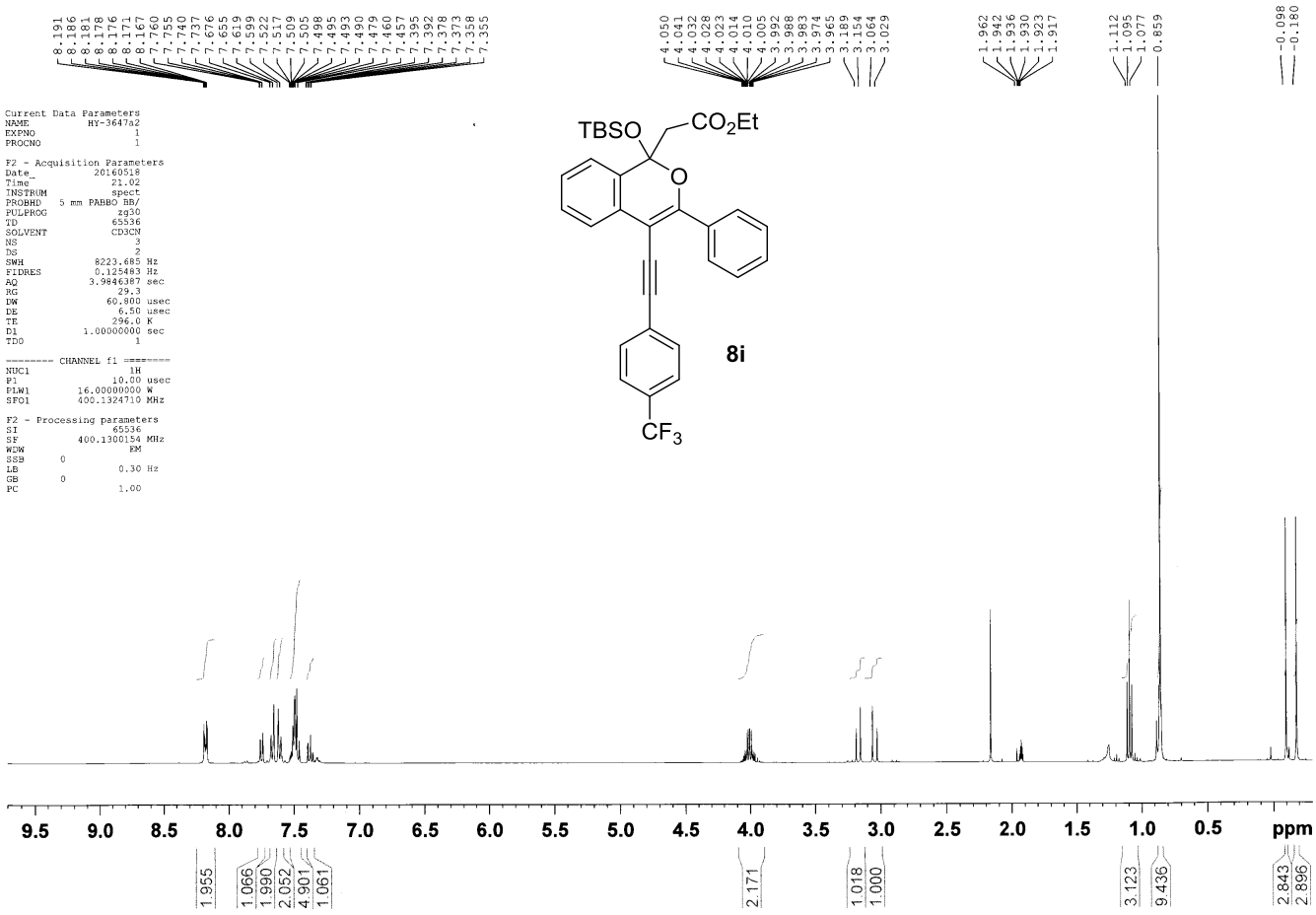

168.49
155.01
139.86
134.49
131.94
131.79
130.39
130.05
129.87
129.73
129.48
128.70
128.36
128.09
126.25
125.03
123.97
123.64
117.70
101.52
97.29
94.40
89.20
60.84
47.59
25.57
20.75
18.10
13.90
1.42
1.21
1.01
0.80
0.59
0.39
0.18
-3.44
-3.90
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Current Data Parameters
NAME HY-3630a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151216
Time 21.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 42
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127288 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

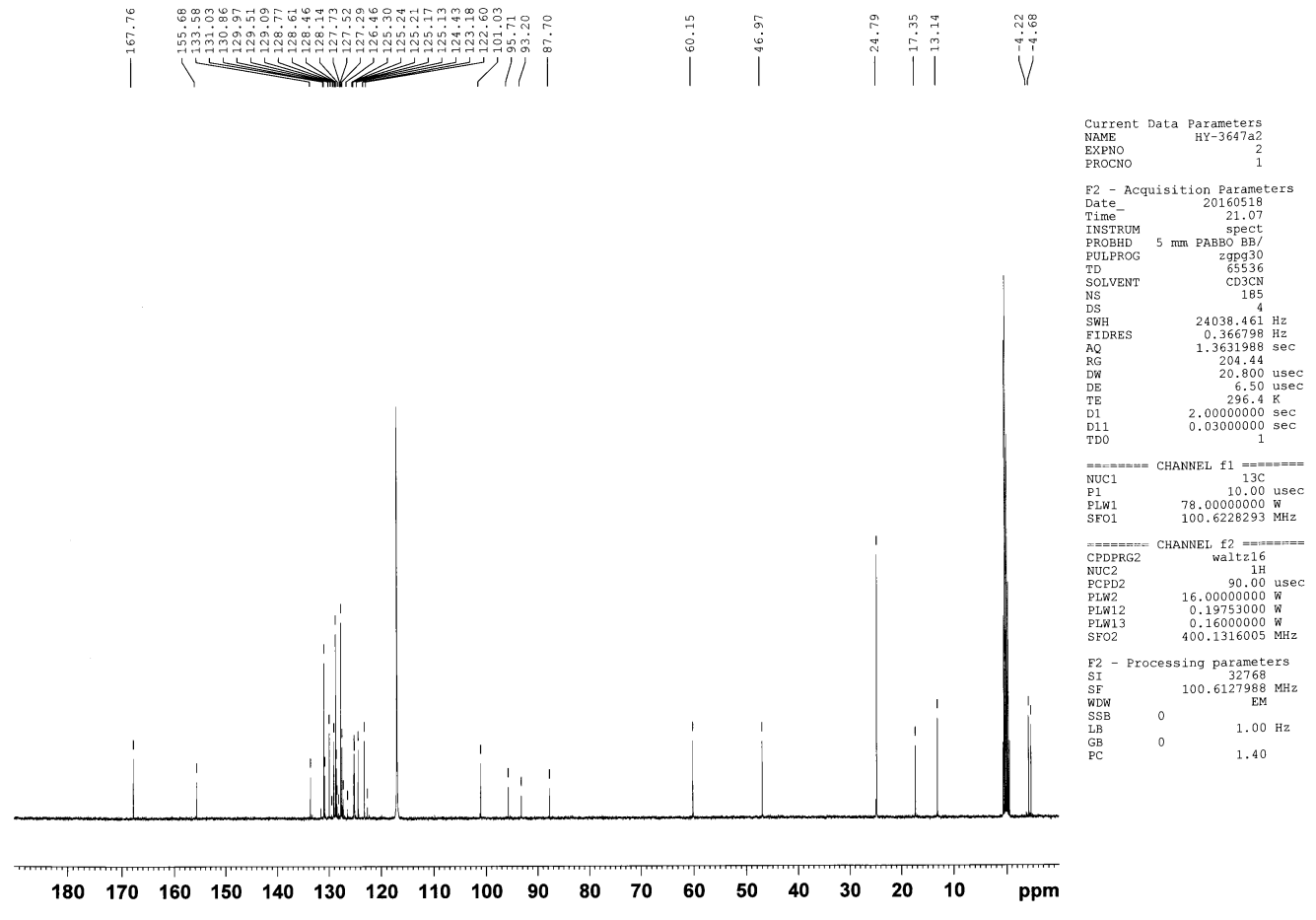


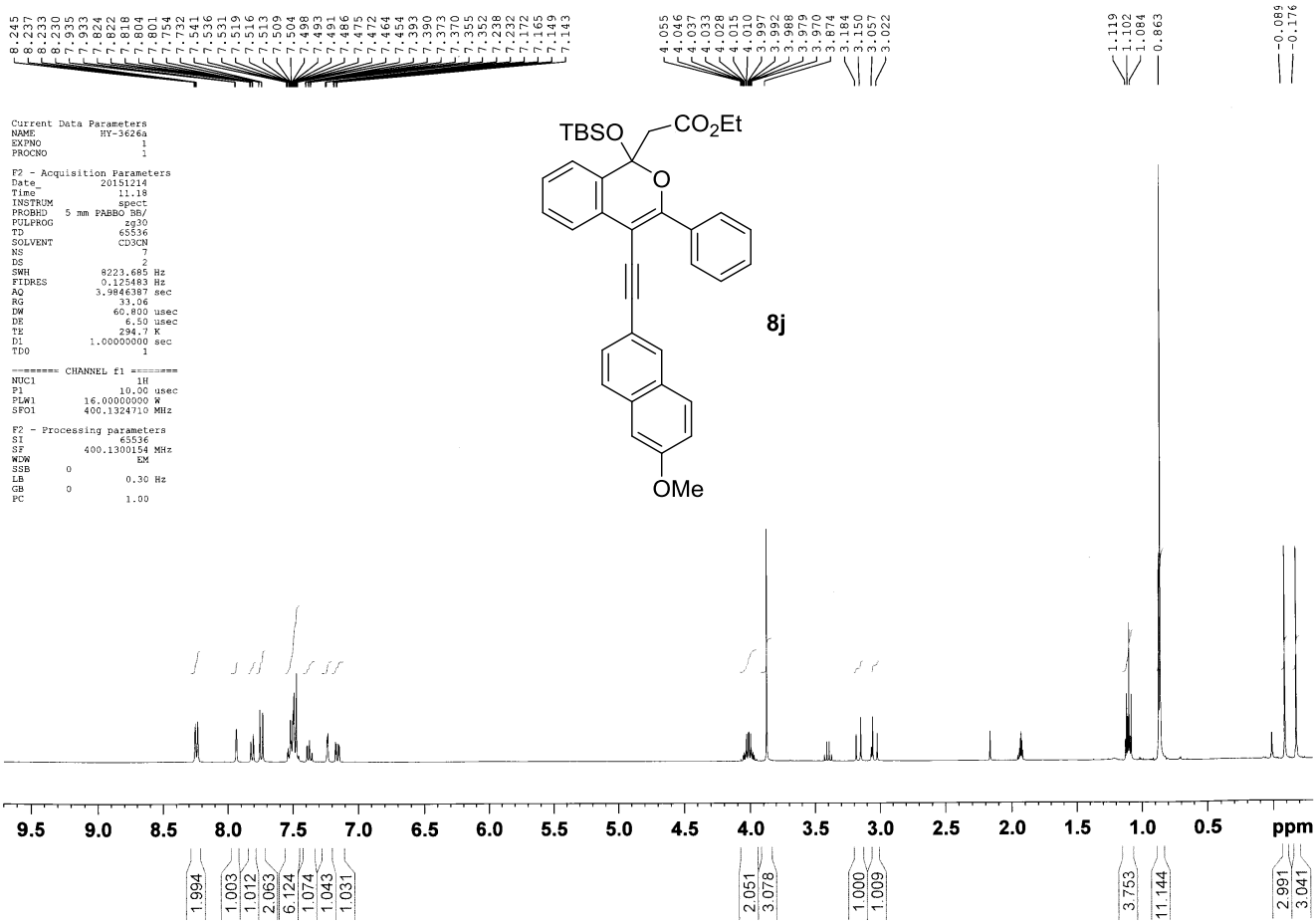

```
Current Data Parameters
NAME              HY-3629a
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_            20151216
Time                20.48
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG             zg30
TD                 65536
SOLVENT            CDCl3
NS                     8
DS                     2
SWH              8223.685 Hz
FIDRES           0.125483 Hz
AQ               3.9846387 sec
RG                  7.99
DW                 60.800 usec
DE                  6.50 usec
TE                 295.2 K
D1           1.00000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                  1H
P1                 10.00 usec
PLW1         16.00000000 W
SFO1        400.1324710 MHz

F2 - Processing parameters
SI                 65536
SF          400.1321395 MHz
WDW                   EM
SSB       0
LB                 0.30 Hz
GB        0
PC                 1.00
```

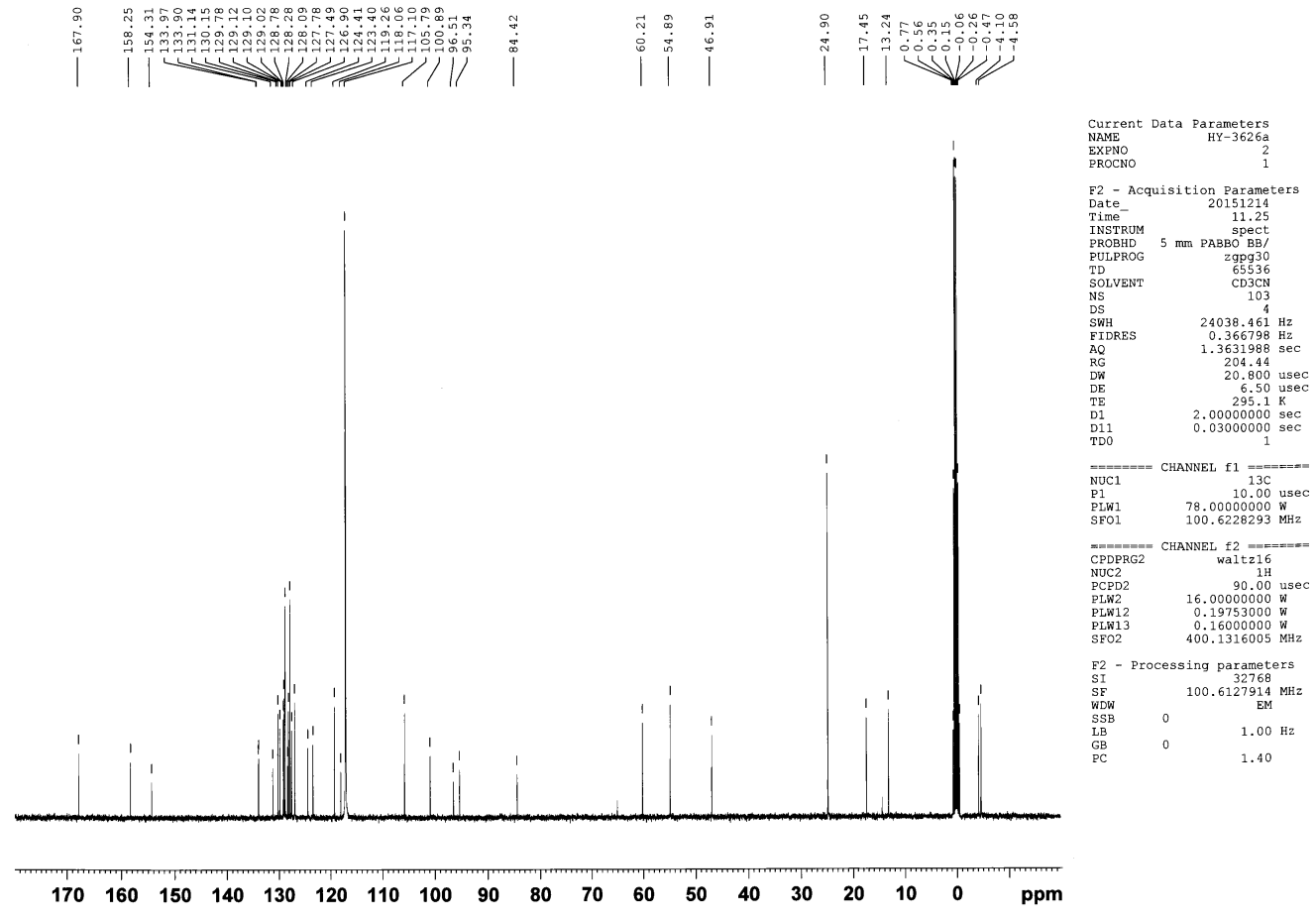


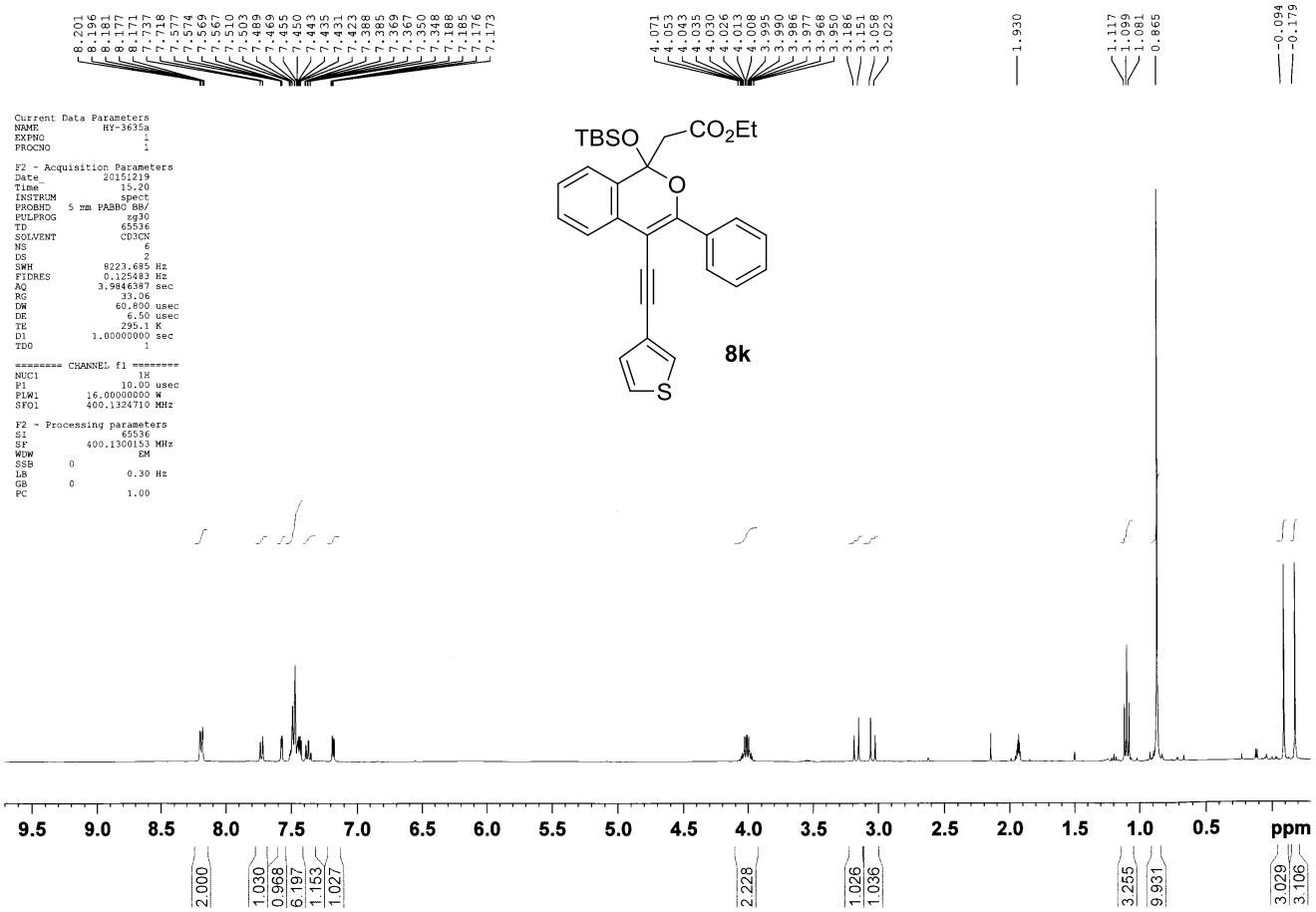

```
Current Data Parameters
NAME              HY-3629a
EXPNO                    2
PROCNO                   1

F2 - Acquisition Parameters
Date_            20151216
Time                20.42
INSTRUM             spect
PROBHD   5 mm PABBO BB/
PULPROG          zgpg30
TD                 65536
SOLVENT            CDCl3
NS                    15
DS                     4
SWH             24038.461 Hz
FIDRES           0.366798 Hz
AQ               1.3631988 sec
RG                 204.44
DW                 20.800 usec
DE                  6.50 usec
TE                 295.3 K
D1           2.00000000 sec
D11          0.03000000 sec
TD0                    1

======== CHANNEL f1 ========
NUC1                 13C
P1                 10.00 usec
PLW1        78.00000000 W
SFO1       100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2          waltz16
NUC2                  1H
PCPD2              90.00 usec
PLW2        16.00000000 W
PLW12        0.19753000 W
PLW13        0.16000000 W
SFO2       400.1316005 MHz

F2 - Processing parameters
SI                 32768
SF          100.6132584 MHz
WDW                   EM
SSB       0
LB                 1.00 Hz
GB        0
PC                 1.40
```


8.191
8.186
8.181
8.178
8.176
8.171
8.167
7.760
7.755
7.740
7.737
7.676
7.655
7.619
7.599
7.522
7.517
7.509
7.505
7.498
7.495
7.493
7.490
7.479
7.460
7.457
7.395
7.392
7.378
7.373
7.358
7.355
4.050
4.041
4.032
4.028
4.023
4.014
4.010
4.005
3.992
3.988
3.983
3.974
3.965
3.189
3.154
3.064
3.029
1.962
1.942
1.936
1.930
1.923
1.917
1.112
1.095
1.077
0.859
-0.098
-0.180
Current Data Parameters
NAME HY-3647a2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160518
Time 21.02
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 3
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 29.3
DW 60.800 usec
DE 6.50 usec
TE 296.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
8i
CF3
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.955
1.066
1.990
2.052
4.901
1.061
2.171
1.018
1.000
3.123
9.436
2.843
2.896


167.76
155.68
133.58
131.03
130.86
129.97
129.51
129.09
128.77
128.61
128.46
128.14
127.73
127.52
127.29
126.46
125.30
125.24
125.21
125.17
125.13
124.43
123.18
122.60
101.03
95.71
93.20
87.70
60.15
46.97
24.79
17.35
13.14
-4.22
-4.68
Current Data Parameters
NAME HY-3647a2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160518
Time 21.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 185
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127988 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.245
8.237
8.233
8.230
7.935
7.933
7.824
7.822
7.818
7.804
7.801
7.754
7.732
7.541
7.536
7.531
7.519
7.516
7.513
7.509
7.504
7.498
7.493
7.491
7.486
7.475
7.472
7.464
7.454
7.393
7.390
7.373
7.370
7.355
7.352
7.238
7.232
7.172
7.165
7.149
7.143
4.055
4.046
4.037
4.033
4.028
4.015
4.010
3.997
3.992
3.988
3.979
3.970
3.874
3.184
3.150
3.057
3.022
1.119
1.102
1.084
0.863
-0.089
-0.176
Current Data Parameters
NAME HY-3626a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151214
Time 11.18
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 7
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 294.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300154 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
8j
OMe
9.5
9.0
8.5
8.0
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
0.5
ppm
1.994
1.003
1.012
2.063
6.124
1.074
1.043
1.031
2.051
3.078
1.000
1.009
3.753
11.144
2.991
3.041


167.90
158.25
154.31
133.97
133.90
131.14
130.15
129.78
129.12
129.10
129.02
128.78
128.28
128.09
127.78
127.49
126.90
124.41
123.40
119.26
118.06
117.10
105.79
100.89
96.51
95.34
84.42
60.21
54.89
46.91
24.90
17.45
13.24
0.77
0.56
0.35
0.15
-0.06
-0.26
-0.47
-4.10
-4.58
Current Data Parameters
NAME HY-3626a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151214
Time 11.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 103
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127914 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
10
0
ppm


8.201
8.196
8.181
8.177
8.171
7.737
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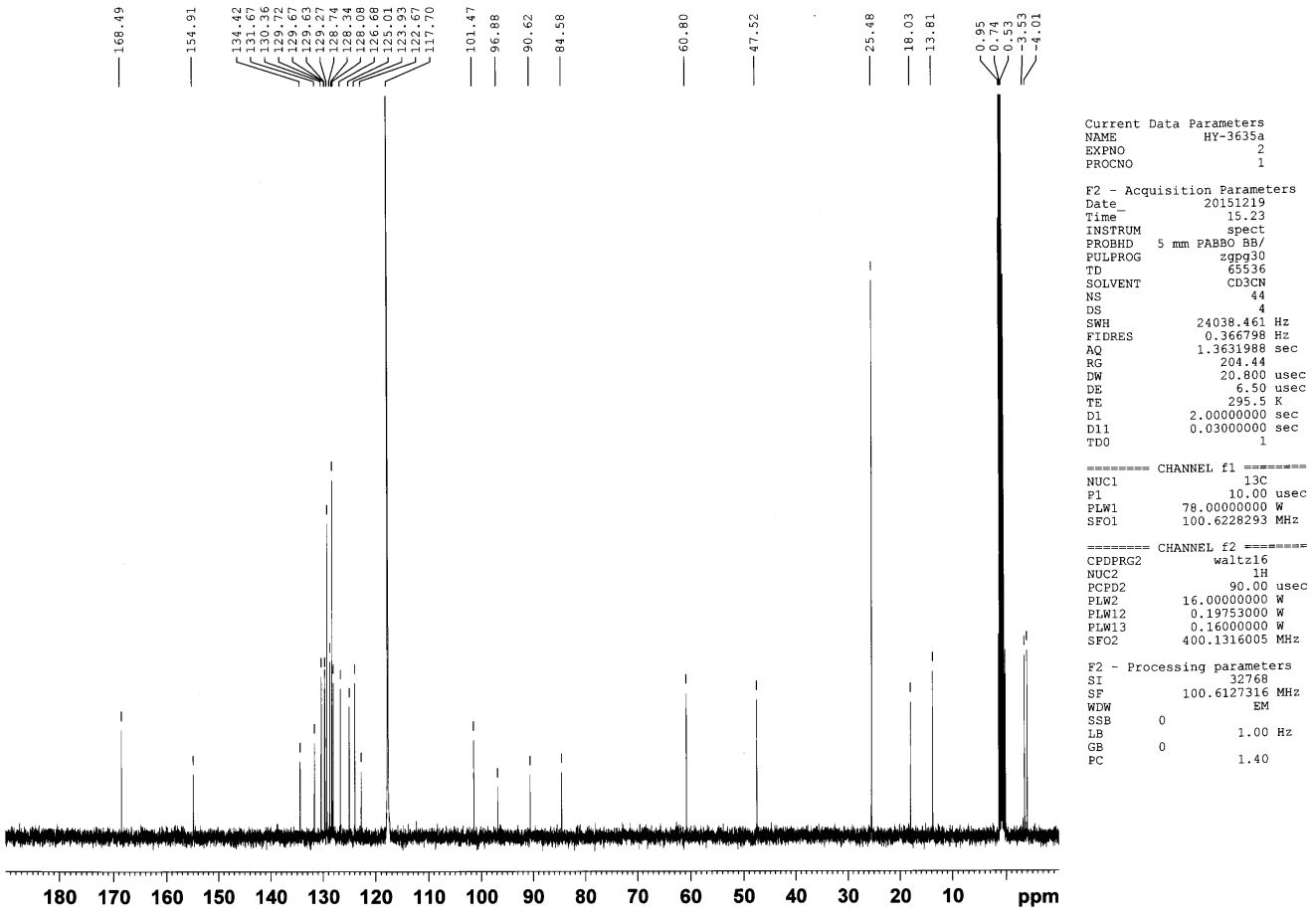
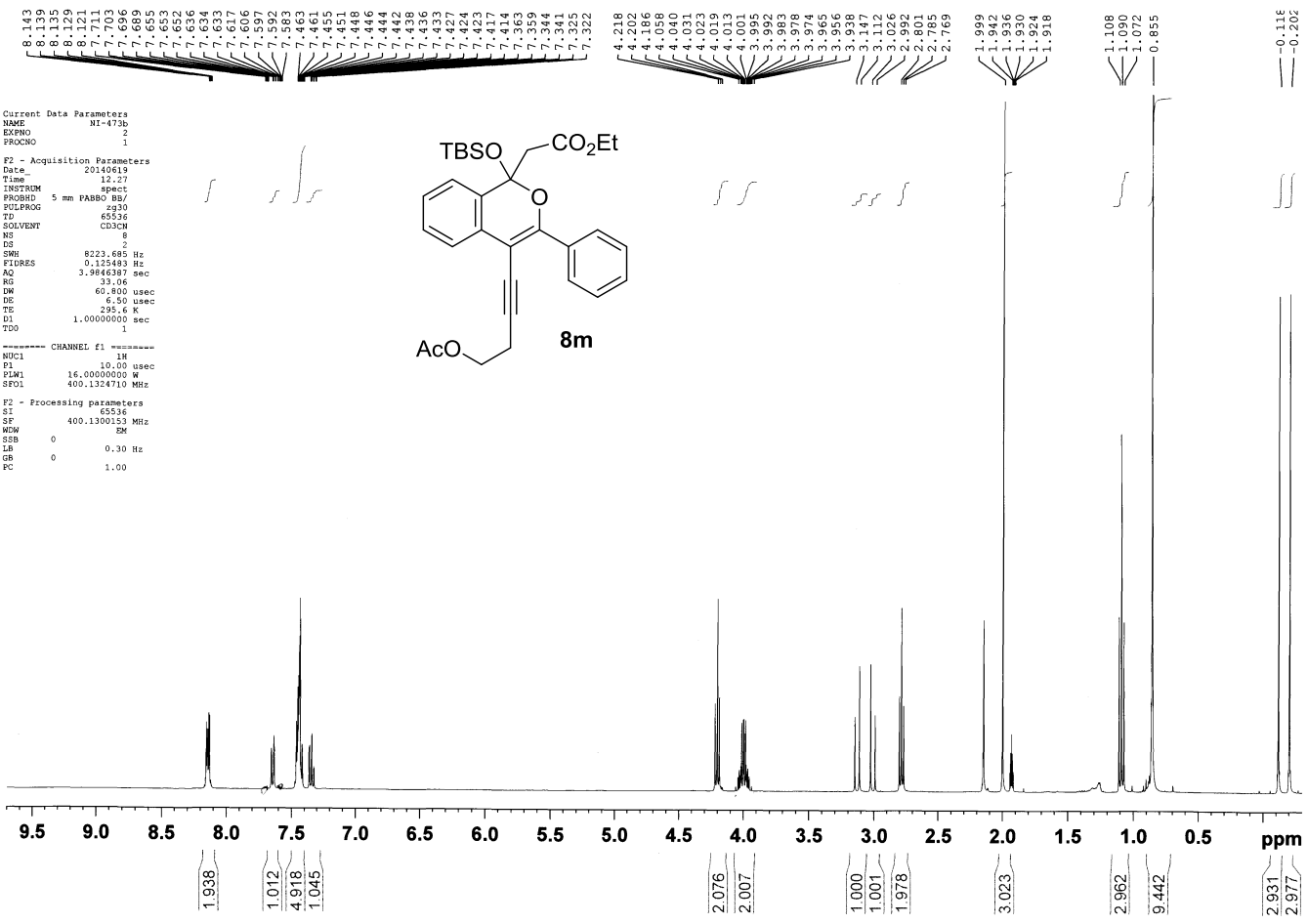
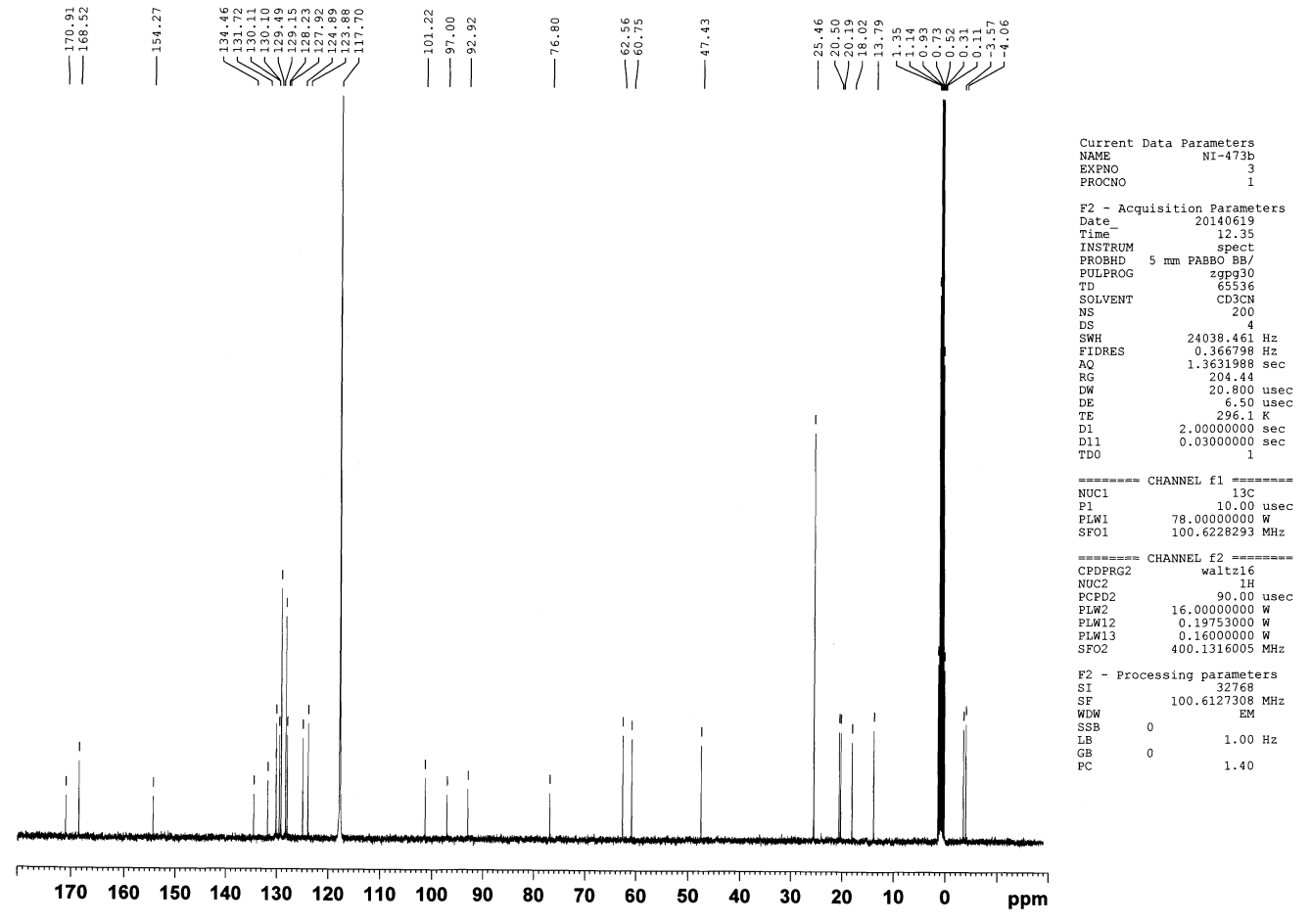
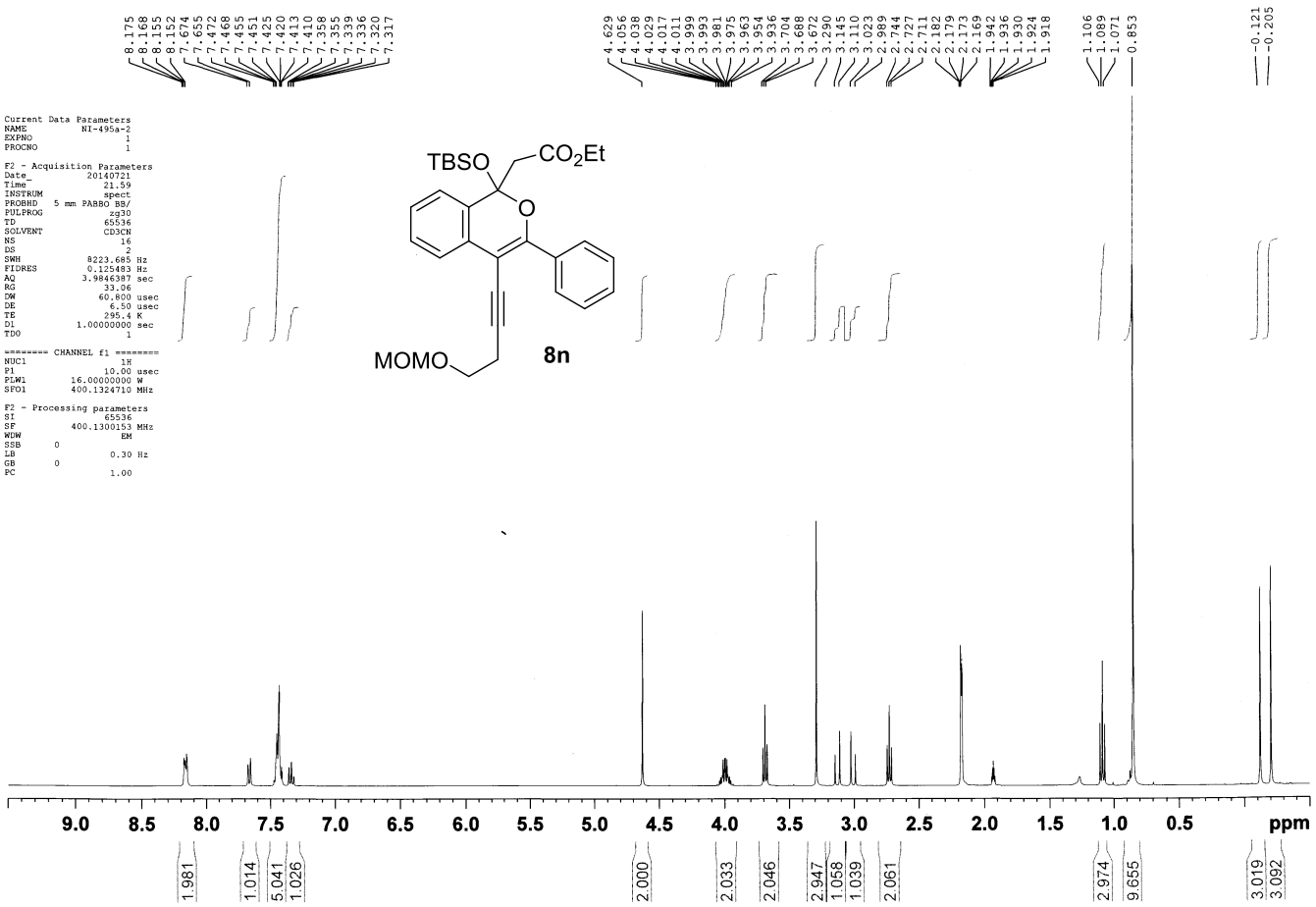
7.577
7.574
7.569
7.567
7.510
7.503
7.489
7.469
7.455
7.450
7.443
7.435
7.431
7.423
7.388
7.385
7.369
7.367
7.350
7.348
7.188
7.185
7.176
7.173
4.071
4.053
4.043
4.035
4.030
4.026
4.013
4.008
3.995
3.990
3.986
3.977
3.968
3.950
3.856
3.151
3.058
3.023
1.930
1.117
1.099
1.081
0.865
-0.094
-0.179
Current Data Parameters
NAME HY-3635a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151219
Time 15.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 6
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 295.1 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
S
8k
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.000
1.030
0.968
6.197
1.153
1.027
2.228
1.026
1.036
3.255
9.931
3.029
3.106

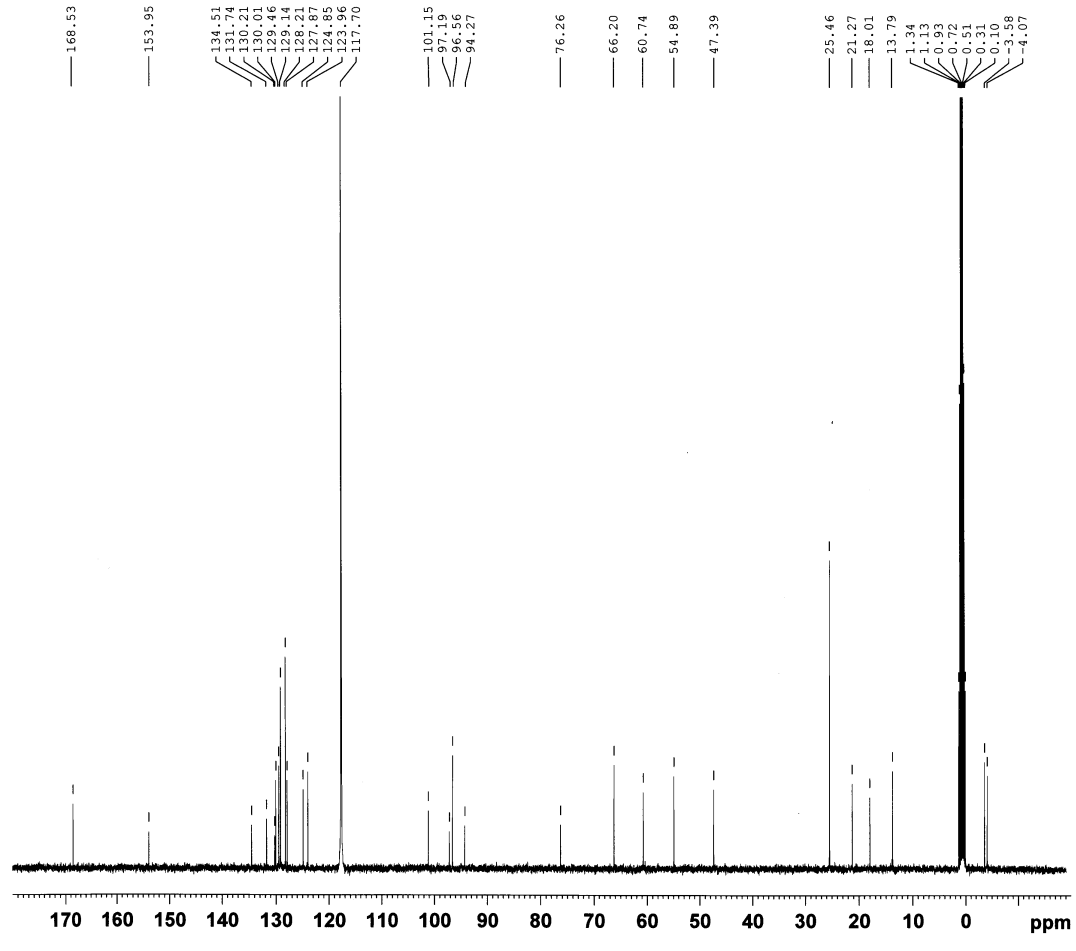

168.49
154.91
134.42
131.67
130.36
129.72
129.67
129.63
129.27
128.74
128.34
128.08
126.68
125.01
123.93
122.67
117.70
101.47
96.88
90.62
84.58
60.80
47.52
25.48
18.03
13.81
0.95
0.74
0.53
-3.53
-4.01
Current Data Parameters
NAME HY-3635a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151219
Time 15.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 44
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.5 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127316 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm


8.143 8.139 8.135 8.129 8.121 7.711 7.703 7.696 7.689 7.655 7.653 7.652 7.636 7.634 7.633 7.617 7.606 7.597 7.592 7.583 7.463 7.461 7.455 7.451 7.448 7.446 7.444 7.442 7.438 7.436 7.433 7.427 7.424 7.423 7.417 7.414 7.363 7.359 7.344 7.341 7.325 7.322
4.218 4.202 4.186 4.058 4.040 4.031 4.023 4.019 4.013 4.001 3.995 3.992 3.983 3.978 3.974 3.965 3.956 3.938 3.147 3.112 3.026 2.992 2.801 2.785 2.769
1.999 1.942 1.936 1.930 1.924 1.918
1.108 1.090 1.072 0.855
-0.118 -0.202
Current Data Parameters
NAME NI-473b
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140619
Time 12.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
AcO
8m
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.938 1.012 4.918 1.045 2.076 2.007 1.000 1.001 1.978 3.023 2.962 9.442 2.931 2.977

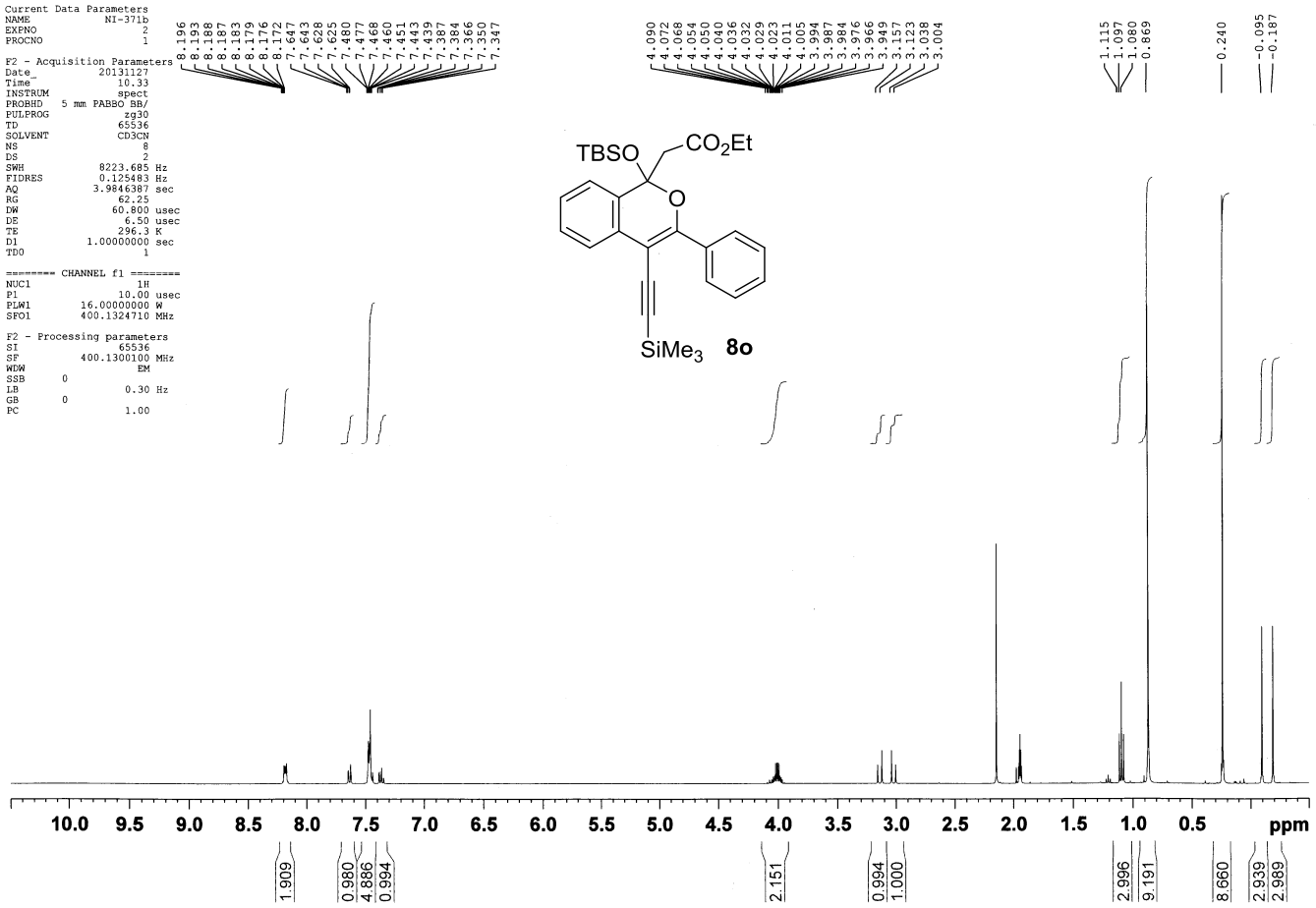

170.91 168.52 154.27 134.46 131.72 130.11 130.10 129.49 129.15 128.23 127.92 124.89 123.88 117.70 101.22 97.00 92.92 76.80 62.56 60.75 47.43 25.46 20.50 20.19 18.02 13.79 1.35 1.14 0.93 0.73 0.52 0.31 0.11 -3.57 -4.06
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Current Data Parameters
NAME NI-473b
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140619
Time 12.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 200
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127308 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

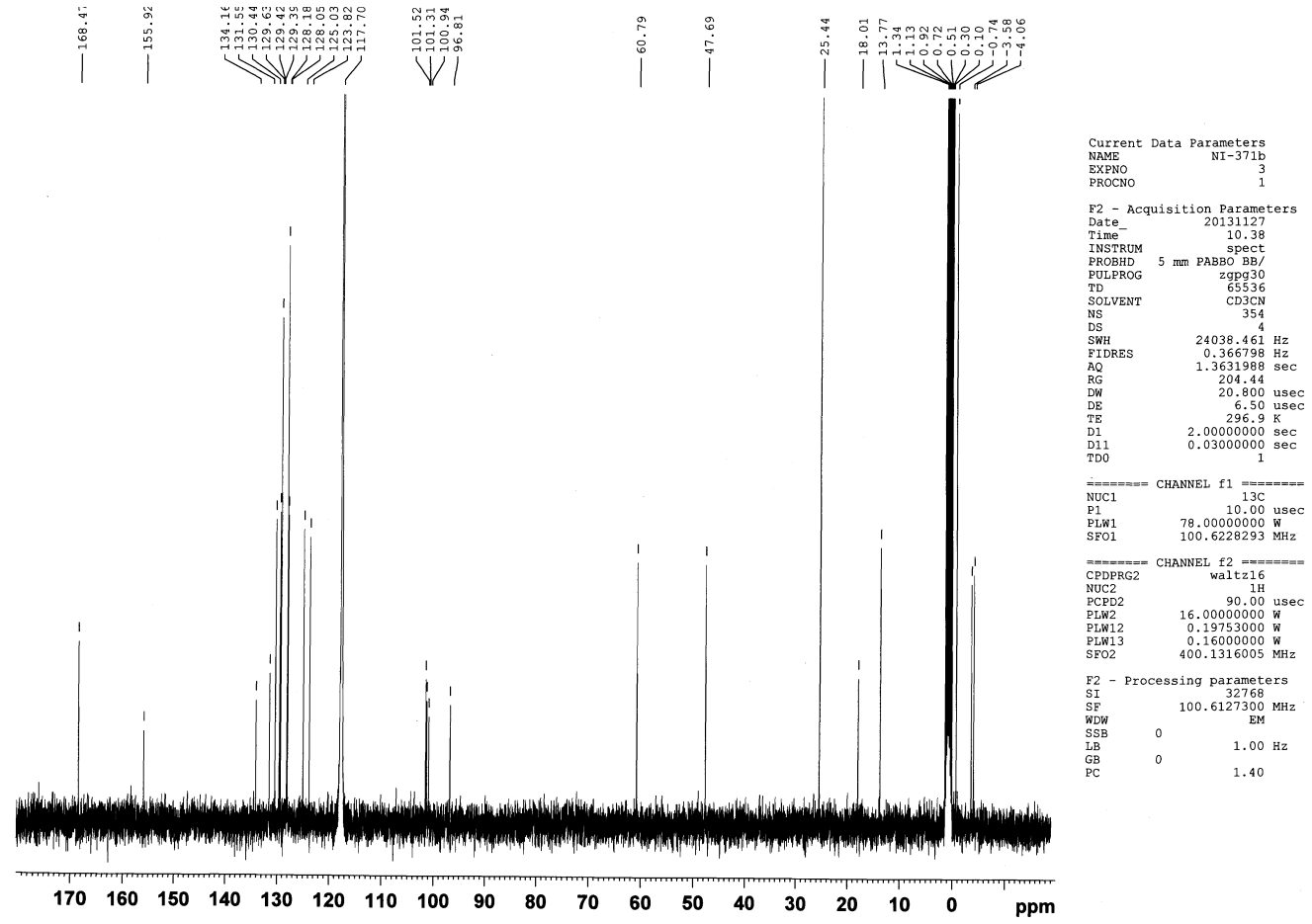



```
Current Data Parameters
NAME                  NI-495a-2
EXPNO                         1
PROCNO                        1

F2 - Acquisition Parameters
Date_                20140721
Time                    21.59
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zg30
TD                     65536
SOLVENT                CD3CN
NS                        16
DS                         2
SWH                 8223.685 Hz
FIDRES              0.125483 Hz
AQ                3.9846387 sec
RG                     33.06
DW                    60.800 usec
DE                     6.50 usec
TE                    295.4 K
D1                1.00000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                       1H
P1                     10.00 usec
PLW1             16.00000000 W
SFO1            400.1324710 MHz

F2 - Processing parameters
SI                         65536
SF                400.1300153 MHz
WDW                           EM
SSB                0
LB                     0.30 Hz
GB                 0
PC                     1.00
```




```
Current Data Parameters
NAME                   NI-495a
EXPNO                        2
PROCNO                       1

F2 - Acquisition Parameters
Date_                20140721
Time                    18.27
INSTRUM                 spect
PROBHD    5 mm PABBO BB/
PULPROG                 zgpg30
TD                     65536
SOLVENT                CD3CN
NS                       184
DS                         4
SWH                24038.461 Hz
FIDRES              0.366798 Hz
AQ                1.3631988 sec
RG                    204.44
DW                    20.800 usec
DE                     6.50 usec
TE                    295.8 K
D1                2.00000000 sec
D11               0.03000000 sec
TD0                        1

======== CHANNEL f1 ========
NUC1                      13C
P1                     10.00 usec
PLW1             78.00000000 W
SFO1            100.6228293 MHz

======== CHANNEL f2 ========
CPDPRG2                waltz16
NUC2                       1H
PCPD2                  90.00 usec
PLW2             16.00000000 W
PLW12             0.19753000 W
PLW13             0.16000000 W
SFO2            400.1316005 MHz

F2 - Processing parameters
SI                         32768
SF                100.6127311 MHz
WDW                           EM
SSB                0
LB                     1.00 Hz
GB                 0
PC                     1.40
```





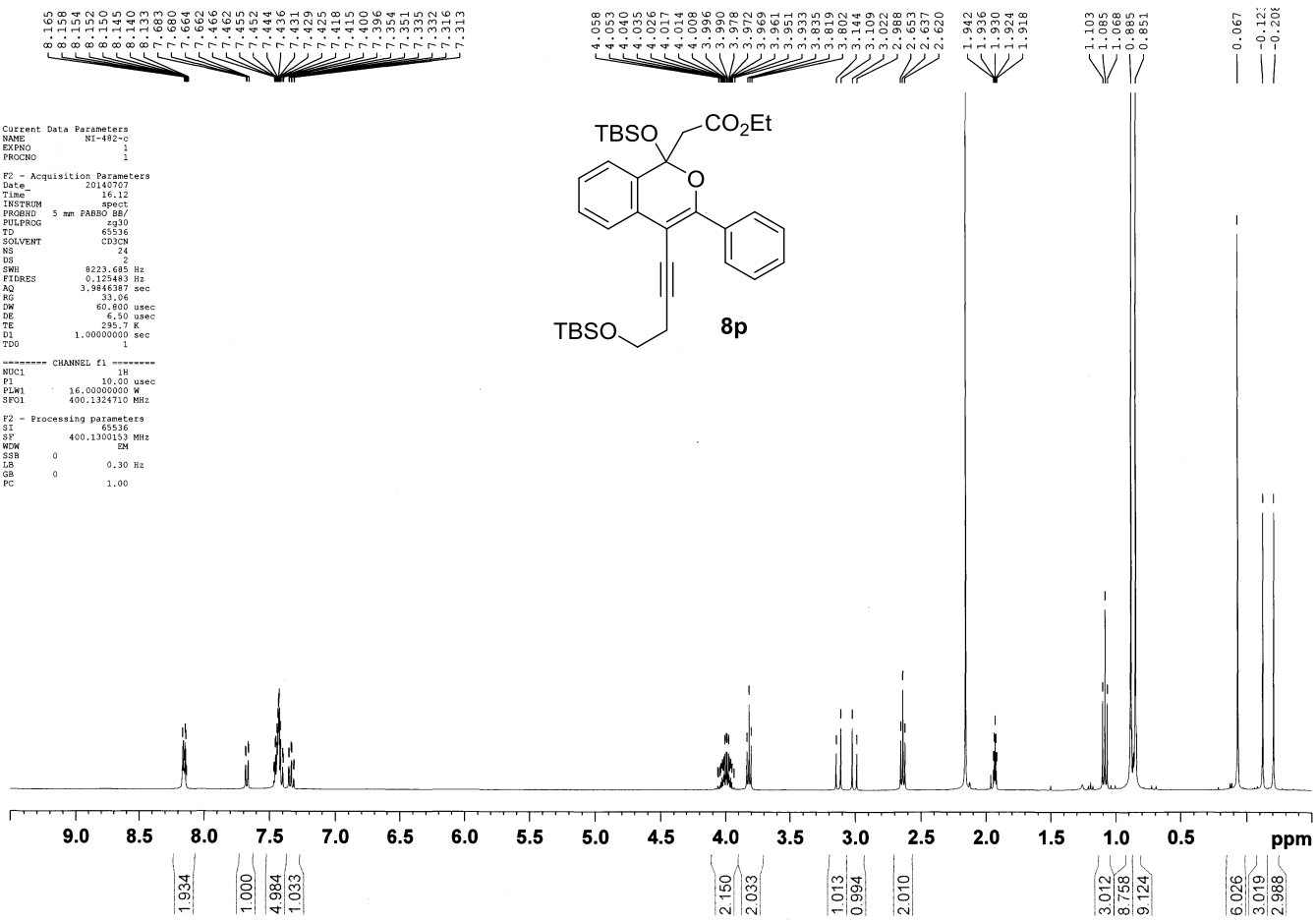
Current Data Parameters
NAME NI-371b
EXPNO 3
PROCNO 1

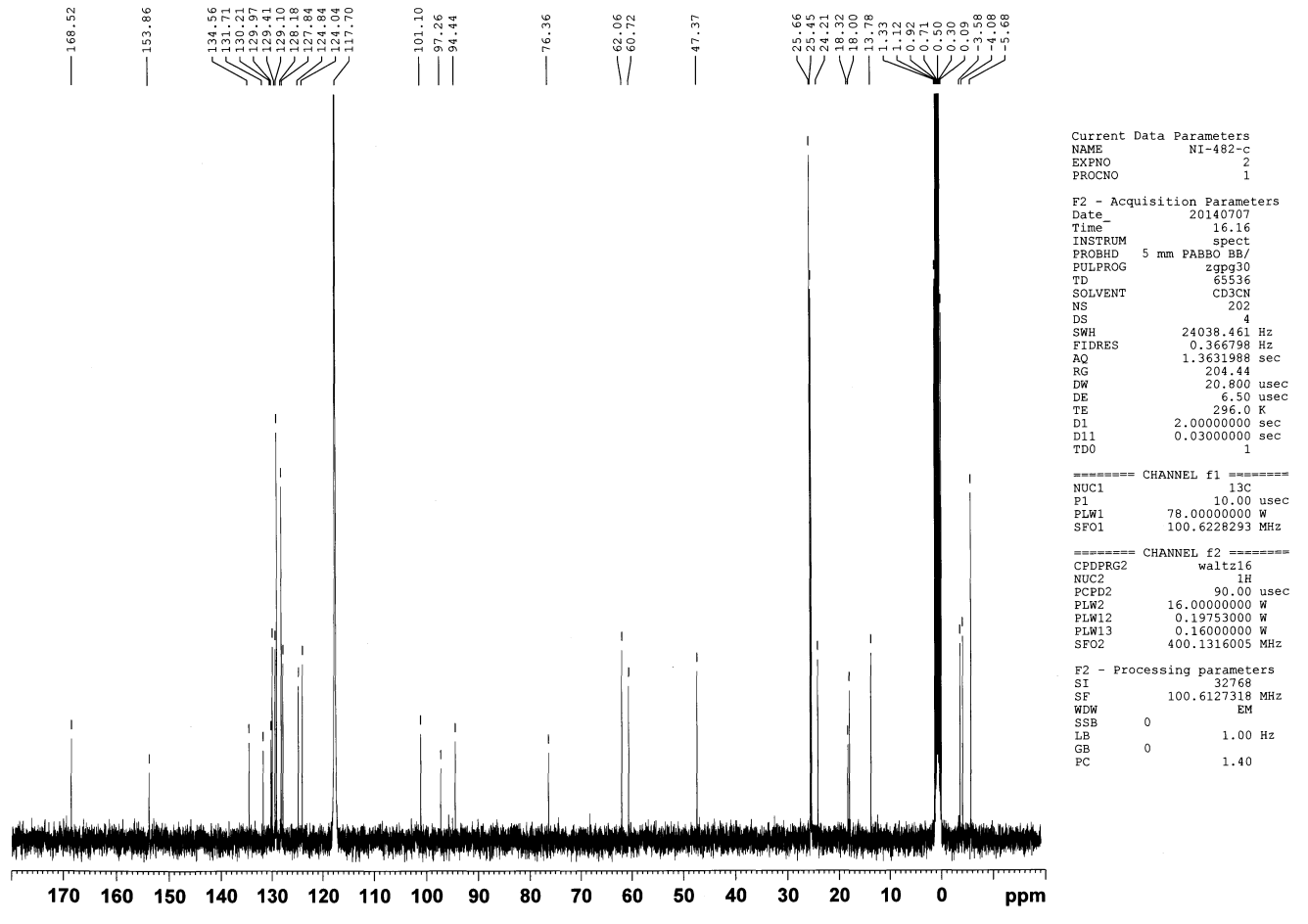
F2 - Acquisition Parameters
Date_ 20131127
Time_ 10.38
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 354
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

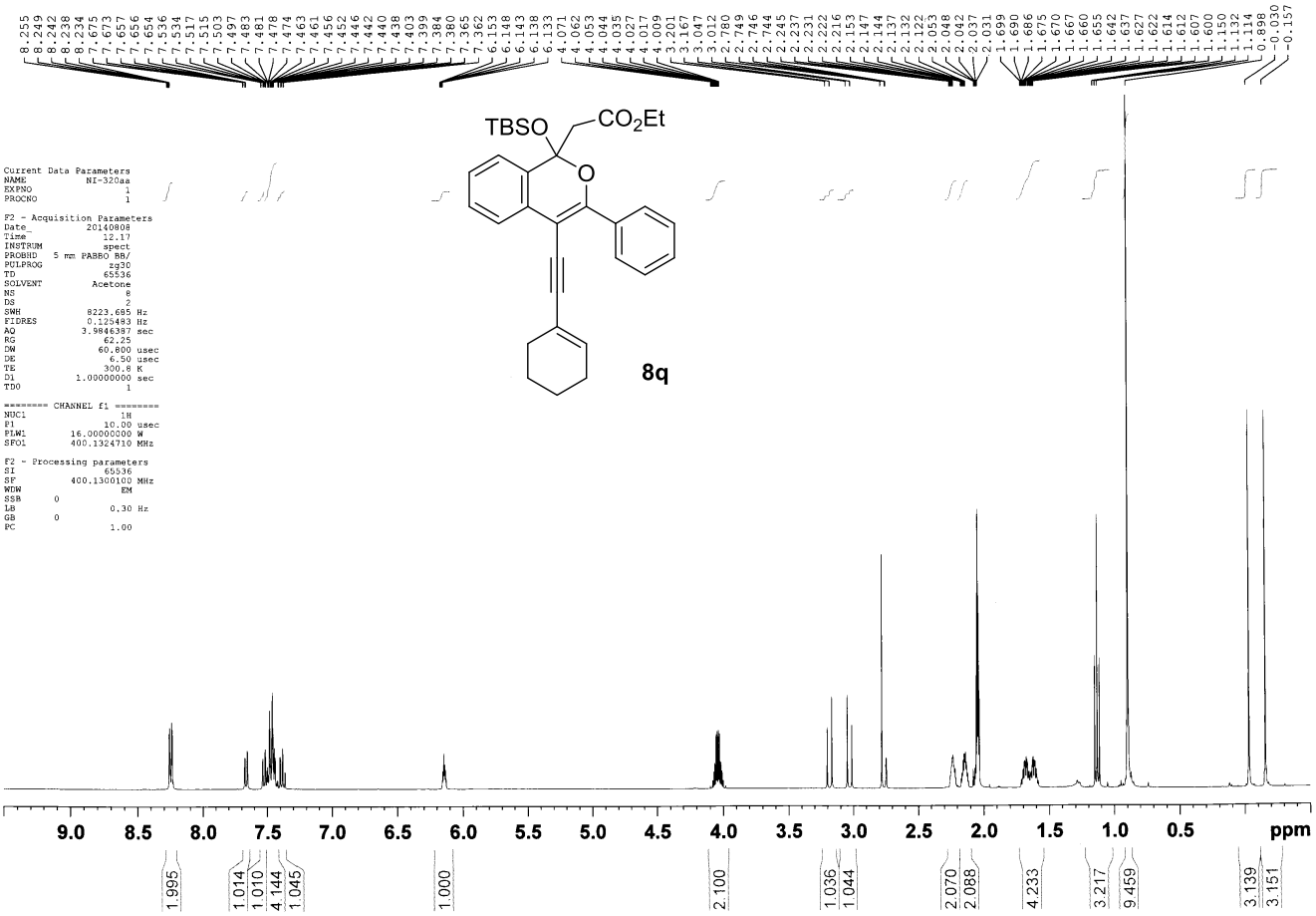
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz

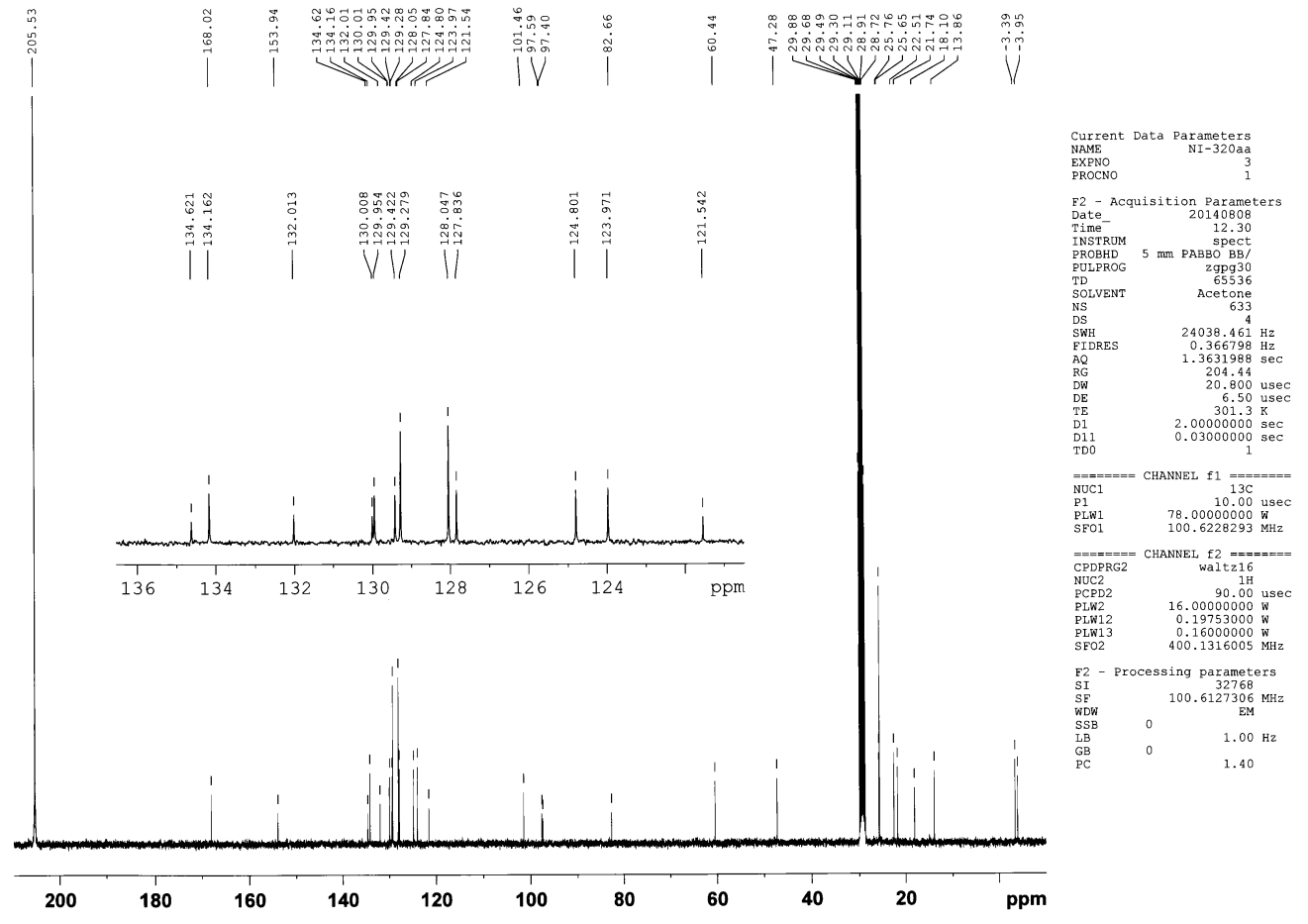
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz

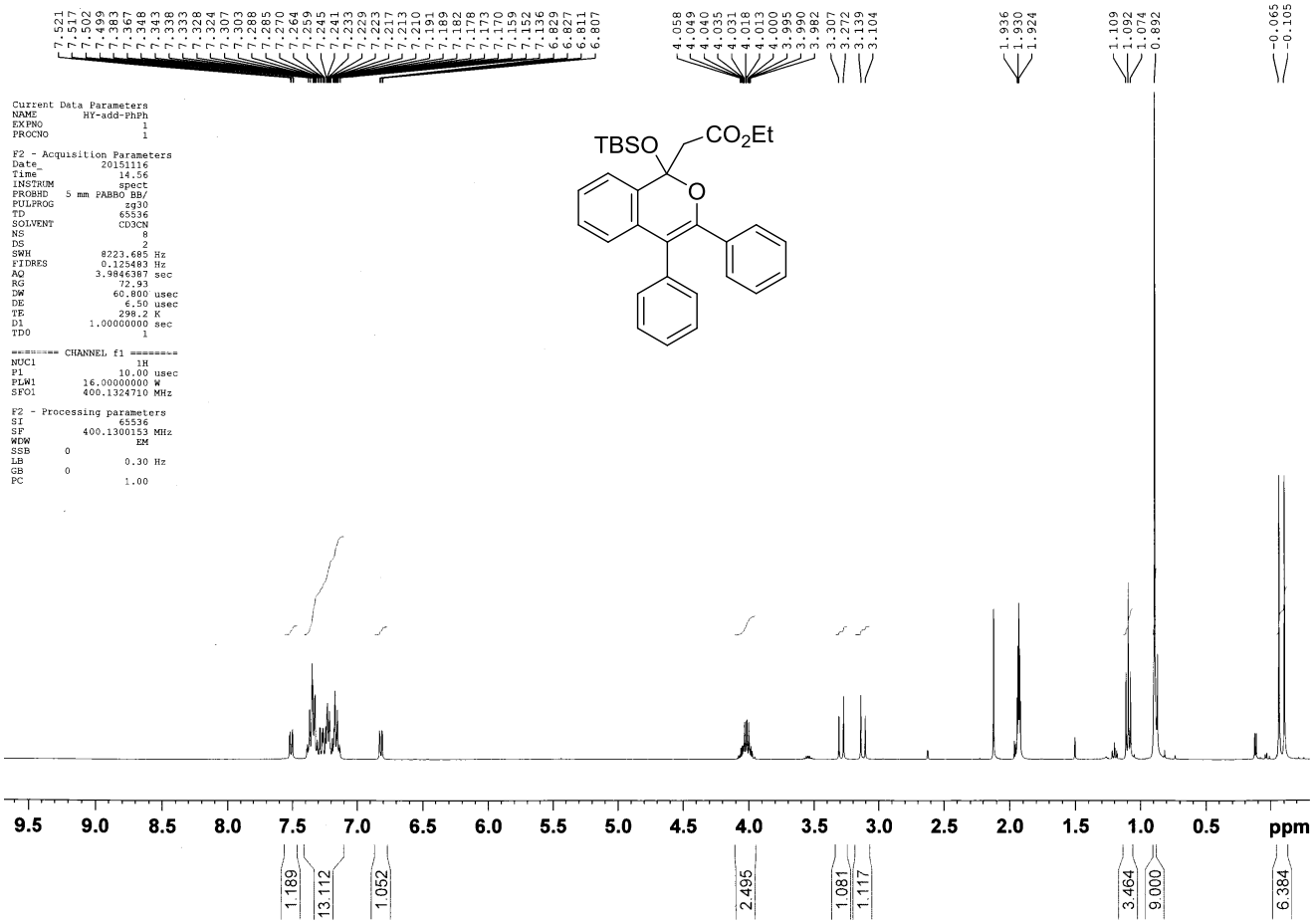
F2 - Processing parameters
SI 32768
SF 100.6127300 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

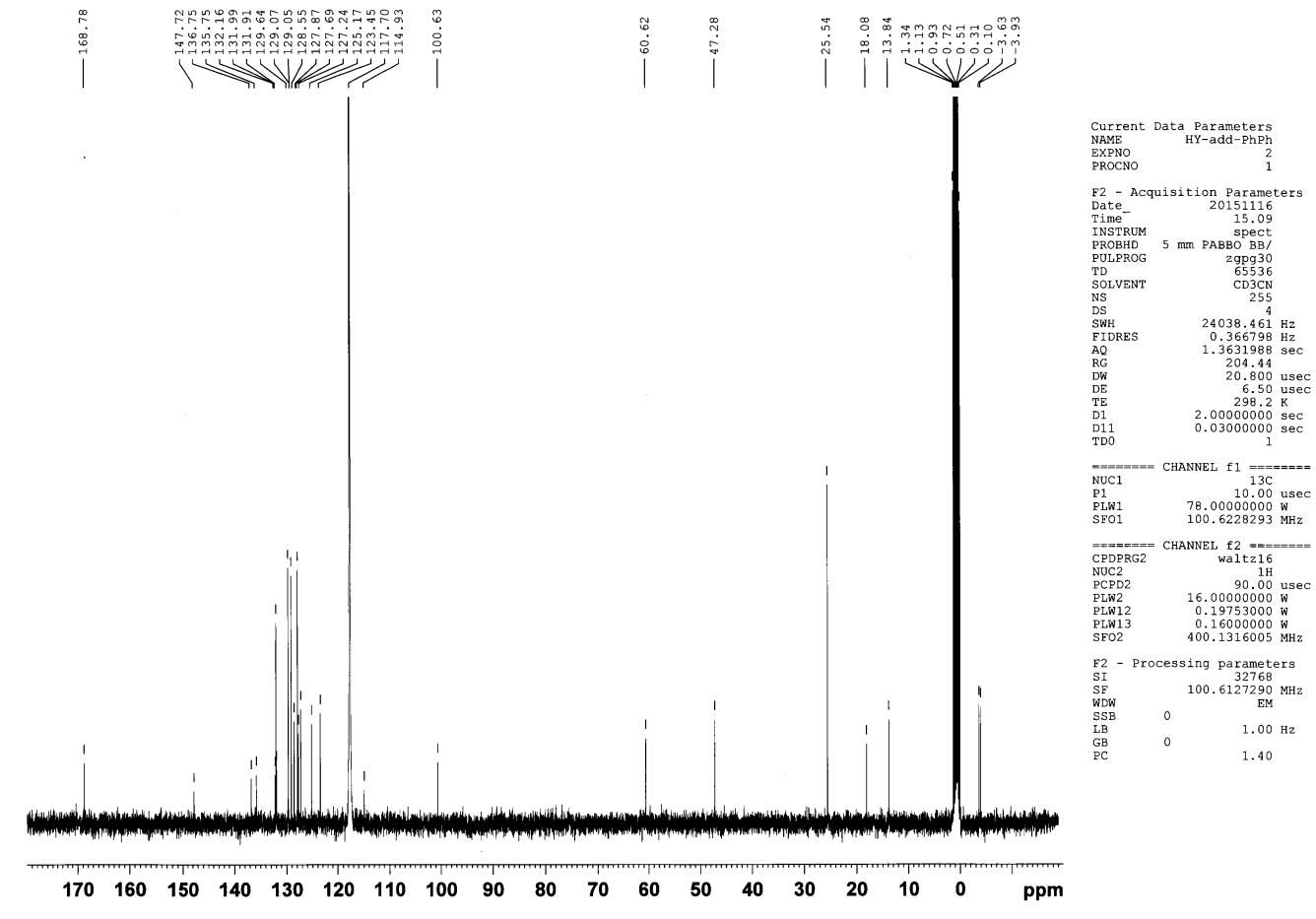

8.165 8.158 8.154 8.152 8.150 8.145 8.140 8.133 7.683 7.680 7.664 7.662 7.466 7.462 7.455 7.452 7.444 7.436 7.431 7.429 7.425 7.418 7.415 7.400 7.396 7.354 7.351 7.335 7.332 7.316 7.313
4.058 4.053 4.040 4.035 4.026 4.017 4.014 4.008 3.996 3.990 3.978 3.972 3.969 3.961 3.951 3.933 3.835 3.819 3.802 3.144 3.109 3.022 2.988 2.653 2.637 2.620
1.942 1.936 1.930 1.924 1.918
1.103 1.085 1.068 0.885 0.851
0.067
Current Data Parameters
NAME NI-482-c
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140707
Time 16.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 24
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 295.7 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
TBSO
8p
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.934 1.000 4.984 1.033 2.150 2.033 1.013 0.994 2.010 3.012 8.758 9.124 6.026 3.019 2.988

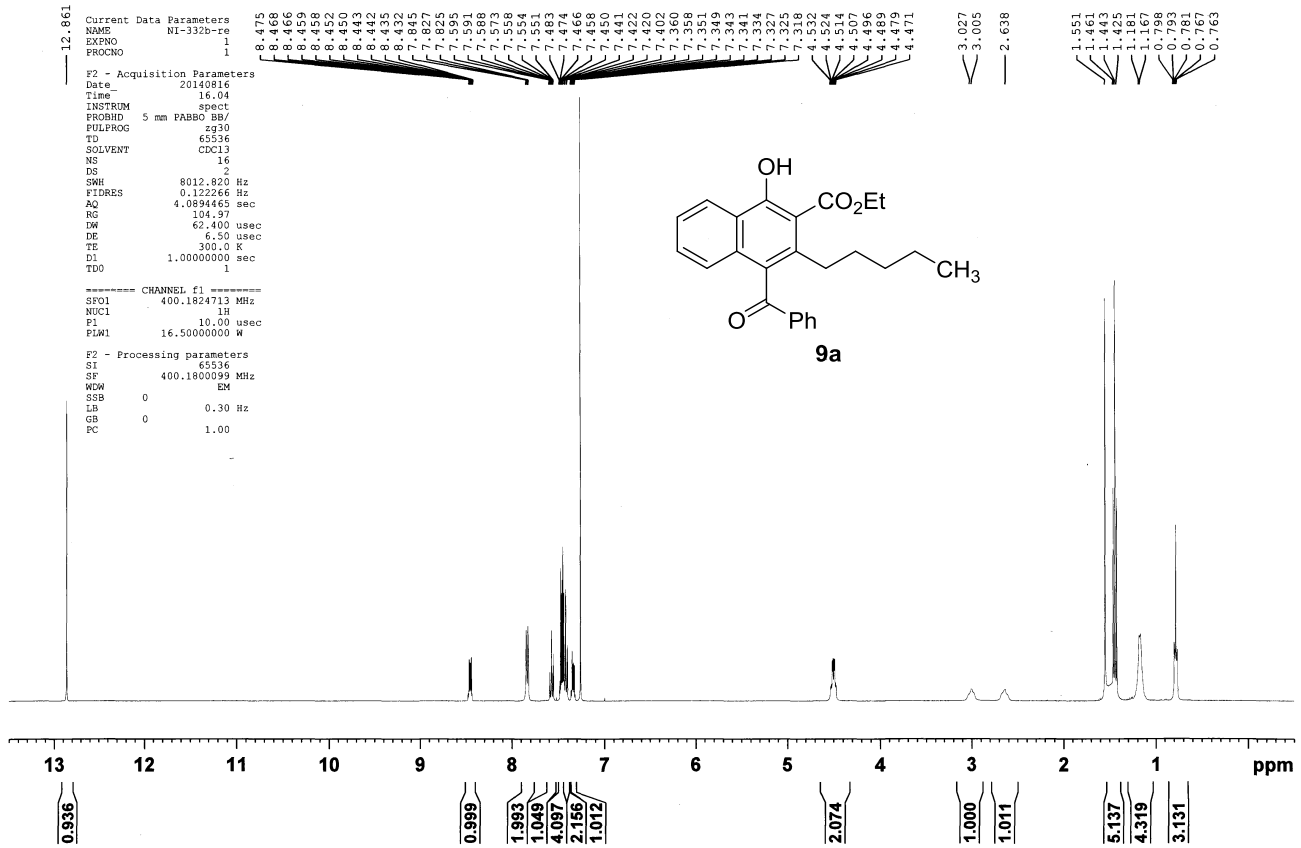

168.52 153.86 134.56 131.71 130.21 129.97 129.41 129.10 128.18 127.84 124.84 124.04 117.70 101.10 97.26 94.44 76.36 62.06 60.72 47.37 25.66 25.45 24.21 18.32 18.00 13.78 1.33 1.12 0.92 0.71 0.50 0.30 0.09 -3.58 -4.08 -5.68
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Current Data Parameters
NAME NI-482-c
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140707
Time 16.16
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 202
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127318 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

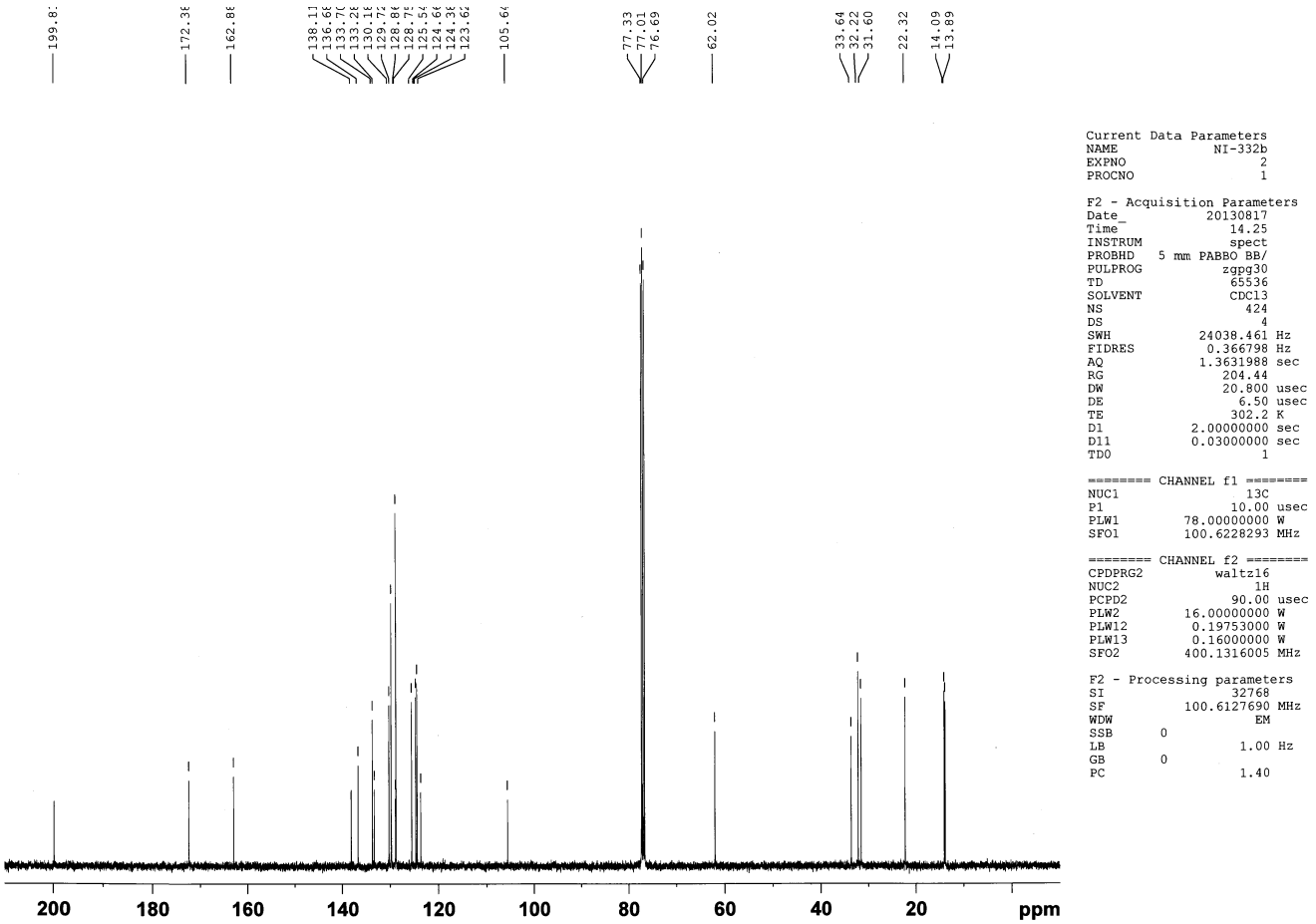

TBSO
CO2Et
O
8q
Current Data Parameters
NAME NI-320aa
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140808
Time 12.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT Acetone
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846397 sec
RG 62.25
DW 60.800 usec
DE 6.50 usec
TE 300.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.995 1.014 1.010 4.144 1.045 1.000 2.100 1.036 1.044 2.070 2.088 4.233 3.217 9.459 3.139 3.151


205.53 168.02 153.94 134.62 134.16 132.01 130.01 129.95 129.42 129.28 128.05 127.84 123.80 121.54 101.46 97.59 97.40 82.66 60.44 47.28 29.88 29.68 29.49 29.30 29.11 28.91 28.72 25.76 25.65 22.51 21.74 18.10 13.86 -3.39 -3.95
134.621 134.162 132.013 130.008 129.954 129.422 129.279 128.047 127.836 124.801 123.971 121.542
136 134 132 130 128 126 124 ppm
200 180 160 140 120 100 80 60 40 20 ppm
Current Data Parameters
NAME NI-320aa
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140808
Time 12.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT Acetone
NS 633
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 301.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127306 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.521 7.517 7.502 7.499 7.483 7.367 7.348 7.343 7.338 7.333 7.328 7.324 7.307 7.303 7.288 7.285 7.270 7.264 7.259 7.245 7.241 7.233 7.229 7.223 7.217 7.213 7.210 7.191 7.189 7.182 7.178 7.173 7.170 7.159 7.152 7.136 6.829 6.827 6.811 6.807
4.058 4.049 4.040 4.035 4.031 4.018 4.013 4.000 3.995 3.990 3.982
3.307 3.272 3.139 3.104
1.936 1.930 1.924
1.109 1.092 1.074 0.892
-0.065 -0.105
Current Data Parameters
NAME HY-add-PhPh
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 14.56
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CD3CN
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 72.93
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300153 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
TBSO
CO2Et
O
9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
1.189 13.112 1.052 2.495 1.081 1.117 3.464 9.000 6.384


168.78
147.72 136.75 135.75 132.16 131.99 131.91 129.64 129.07 129.05 128.55 127.87 127.69 127.24 125.17 123.45 117.70 114.93
100.63
60.62
47.28
25.54
18.08
13.84
1.34 1.13 0.93 0.72 0.51 0.31 0.10 -3.63 -3.93
170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm
Current Data Parameters
NAME HY-add-PhPh
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151116
Time 15.09
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CD3CN
NS 255
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127290 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





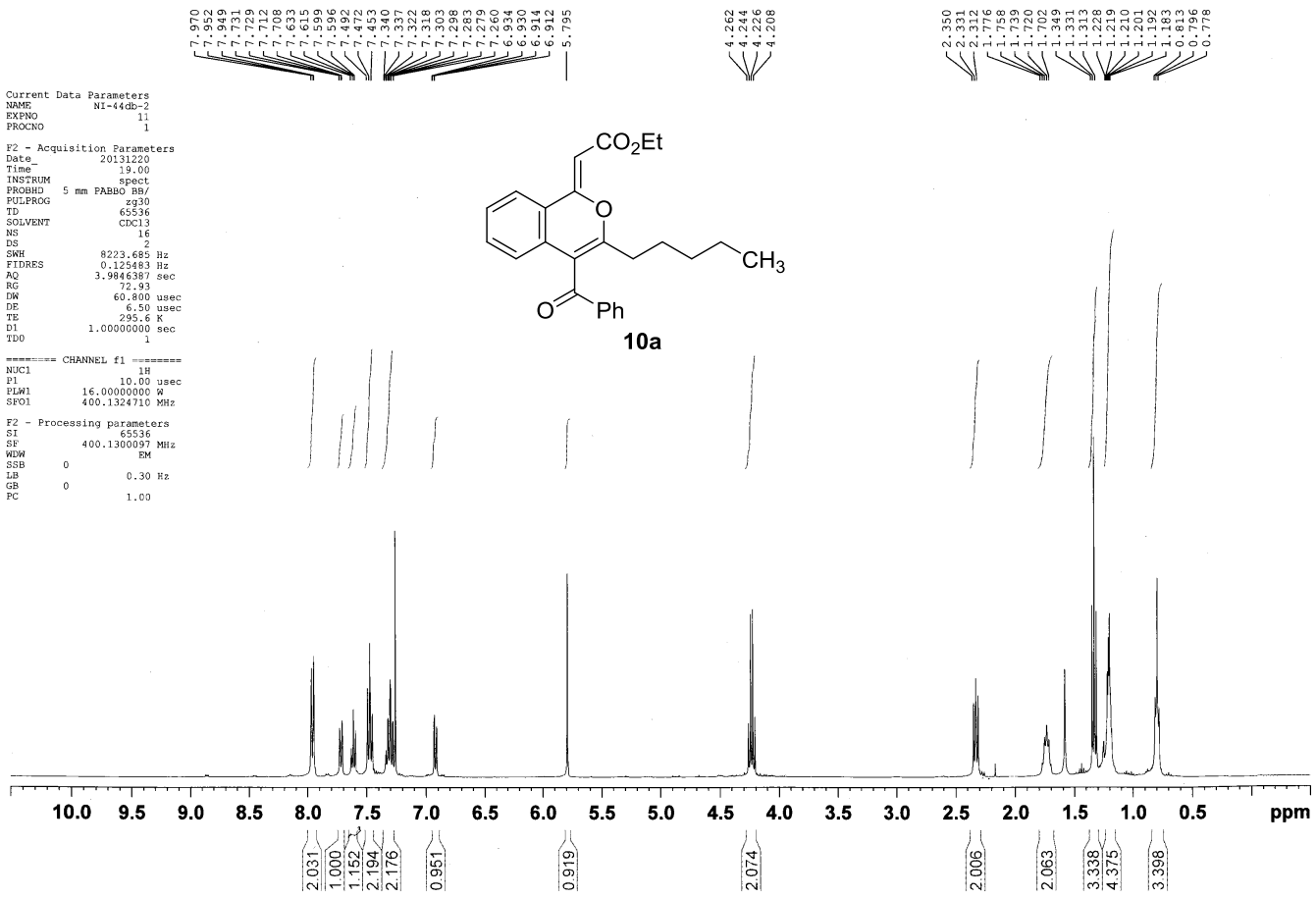
Current Data Parameters
NAME NI-332b
EXPNO 2
PROCNO 1

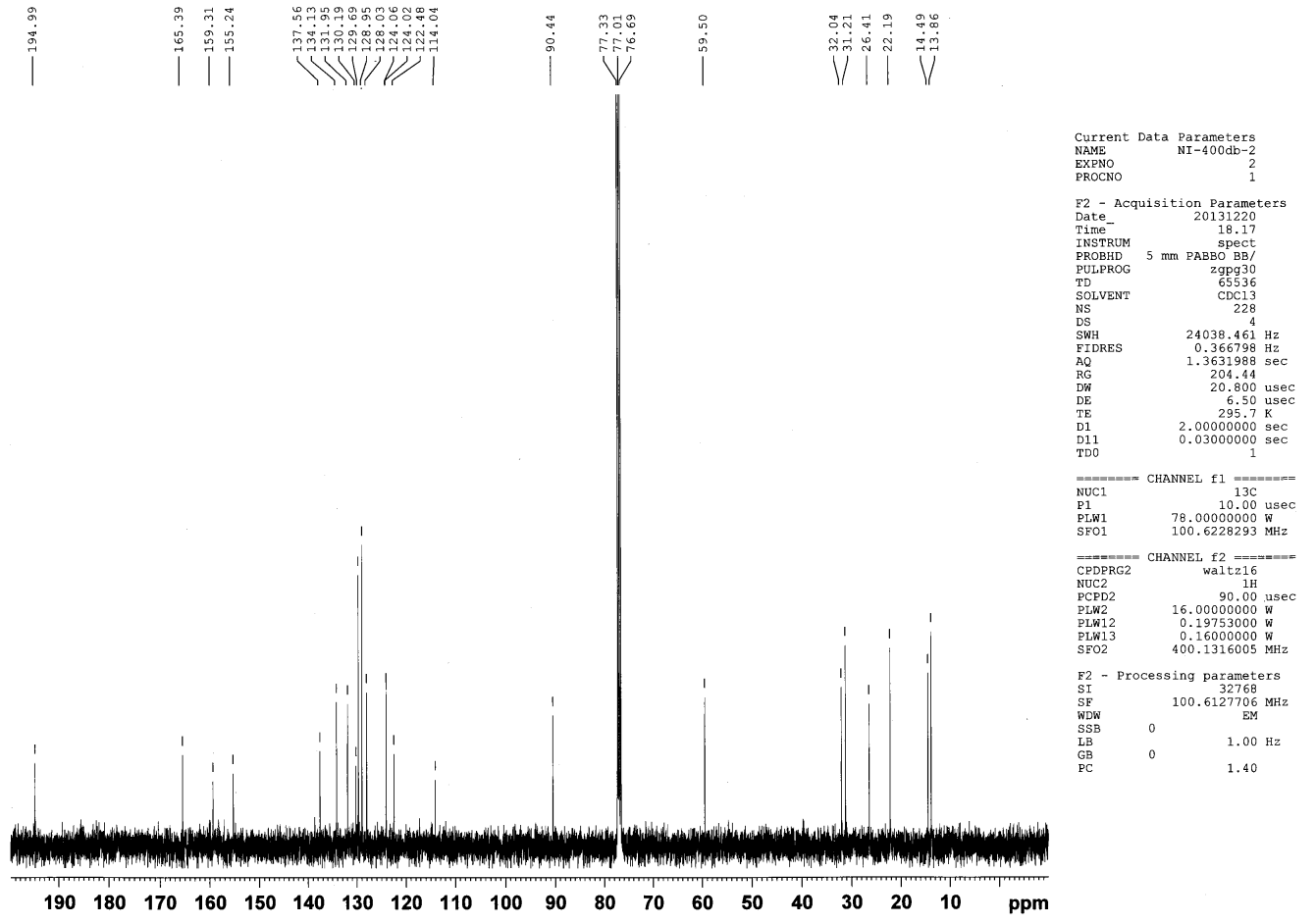
F2 - Acquisition Parameters
Date_ 20130817
Time 14.25
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 424
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 302.2 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

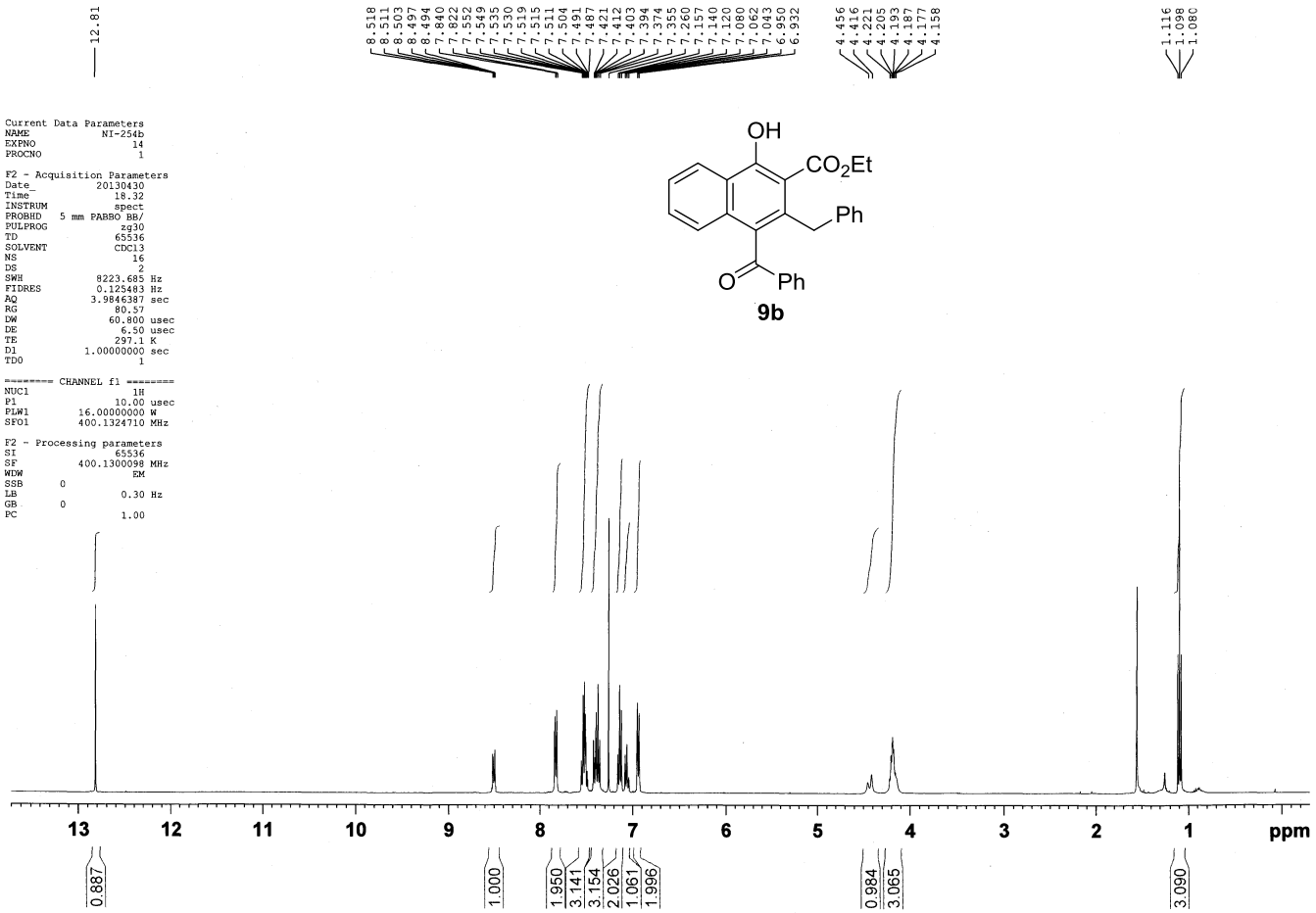
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz

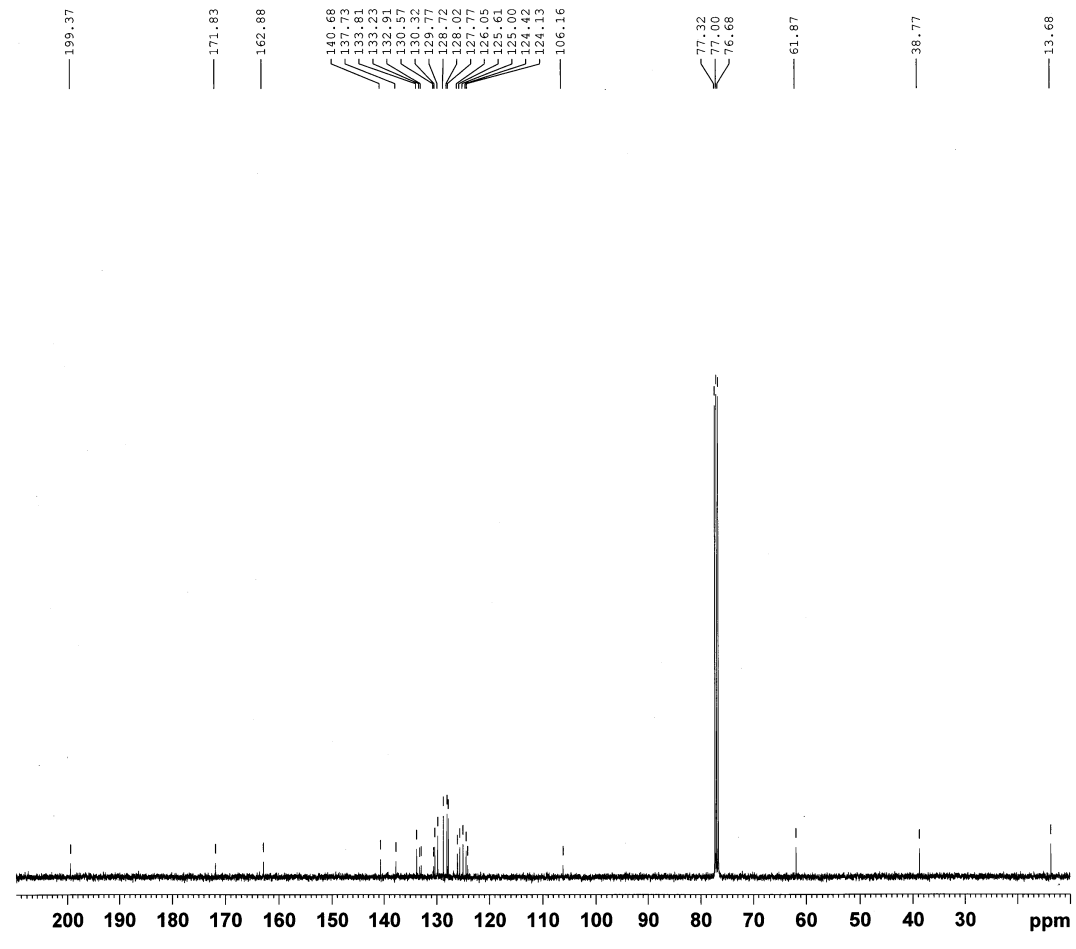
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz

F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


7.970
7.952
7.949
7.731
7.729
7.712
7.708
7.633
7.615
7.599
7.596
7.492
7.472
7.453
7.340
7.337
7.322
7.318
7.303
7.298
7.283
7.279
7.260
6.934
6.930
6.914
6.912
5.795
4.262
4.244
4.226
4.208
2.350
2.331
2.312
1.776
1.758
1.739
1.720
1.702
1.349
1.331
1.313
1.228
1.219
1.210
1.201
1.192
1.183
0.813
0.796
0.778
Current Data Parameters
NAME NI-44db-2
EXPNO 11
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131220
Time_ 19.00
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 72.93
DW 60.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
CO2Et
O
CH3
O
Ph
10a
10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm
2.031
1.000
1.152
2.194
2.176
0.951
0.919
2.074
2.006
2.063
3.338
4.375
3.398


194.99
165.39
159.31
155.24
137.56
134.13
131.95
130.19
129.69
128.95
128.03
124.06
124.02
122.48
114.04
90.44
77.33
77.01
76.69
59.50
32.04
31.21
26.41
22.19
14.49
13.86
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Current Data Parameters
NAME NI-400db-2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131220
Time_ 18.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 228
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127706 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40





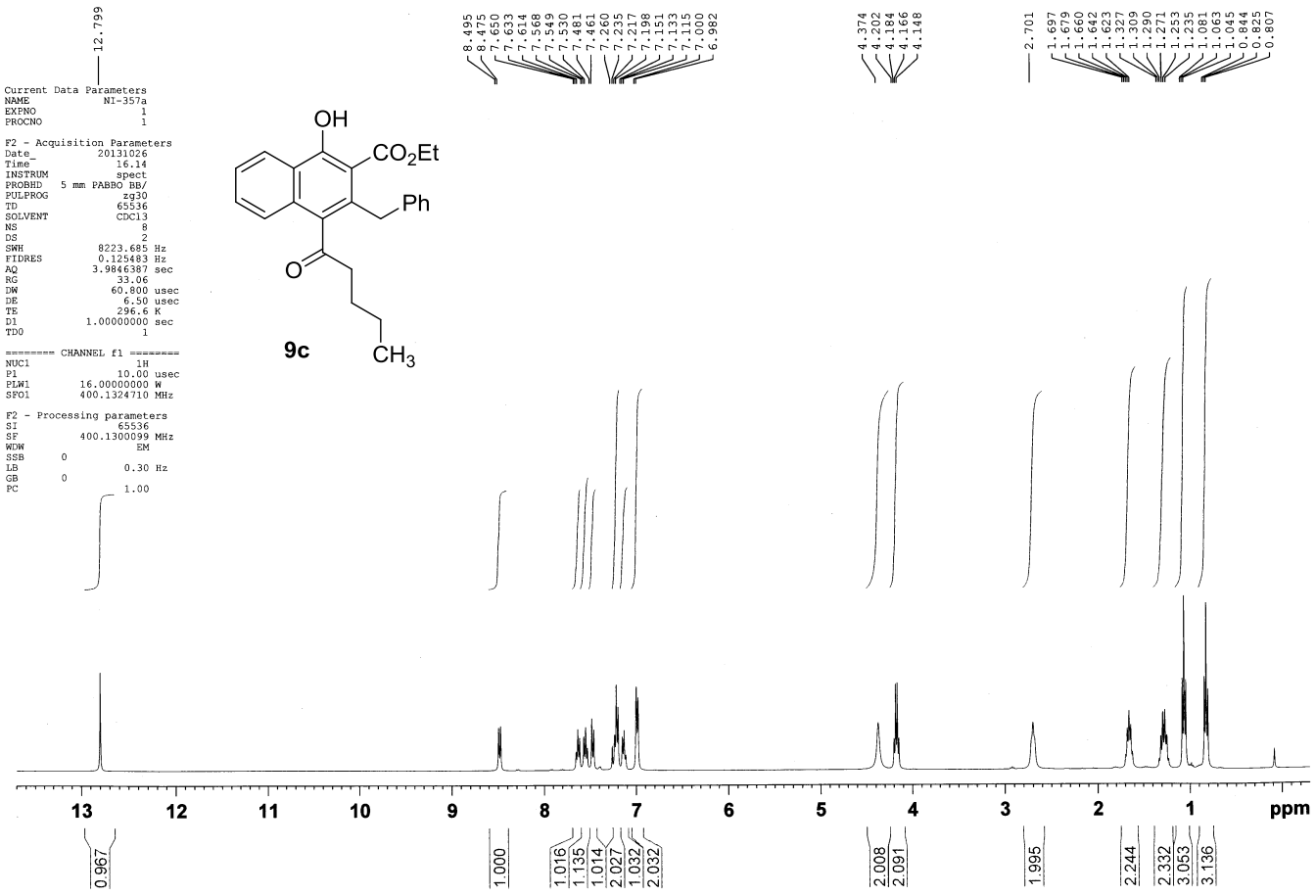
Current Data Parameters
NAME NI-254b
EXPNO 5
PROCNO 1

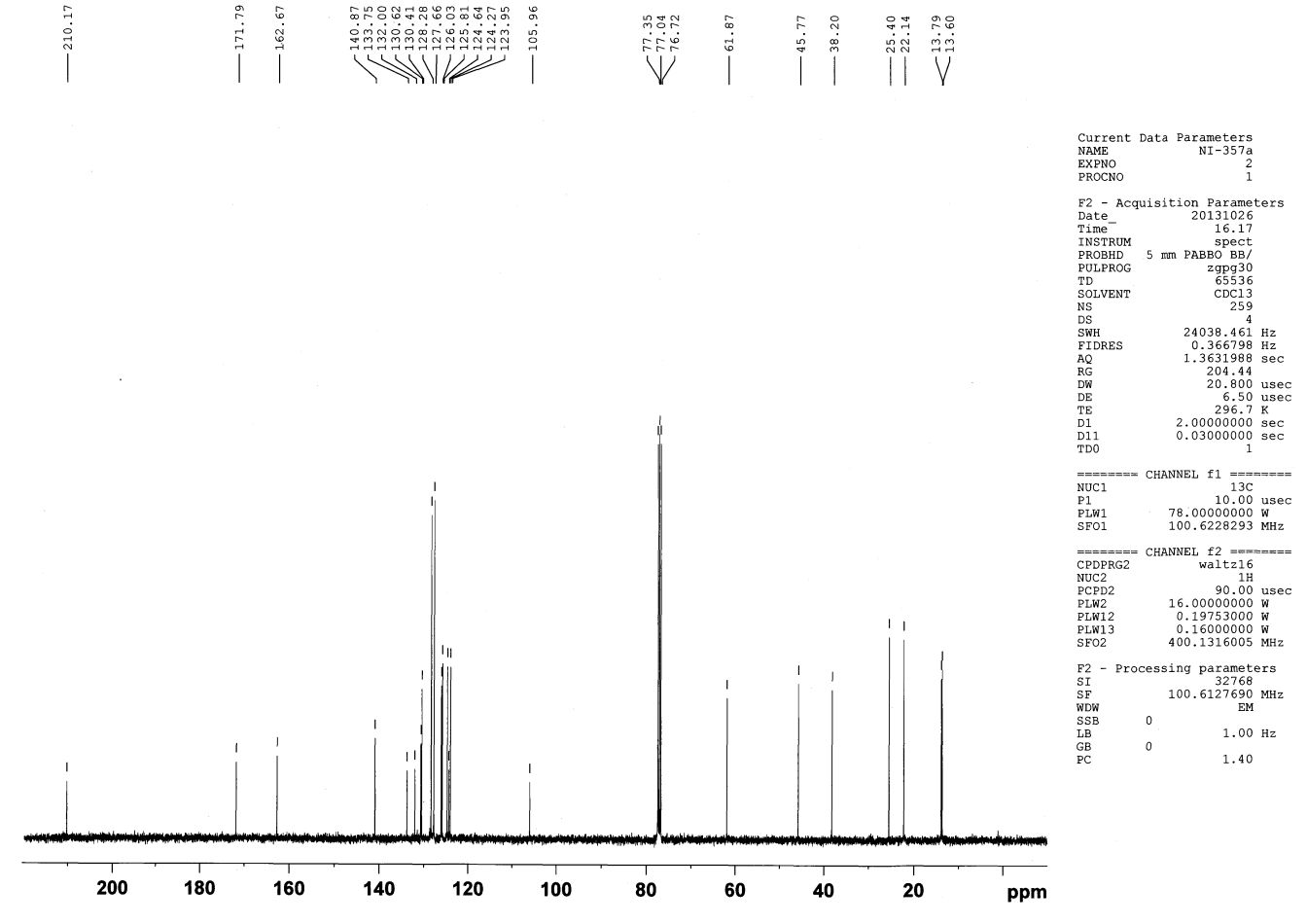
F2 - Acquisition Parameters
Date_ 20130430
Time 12.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 459
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

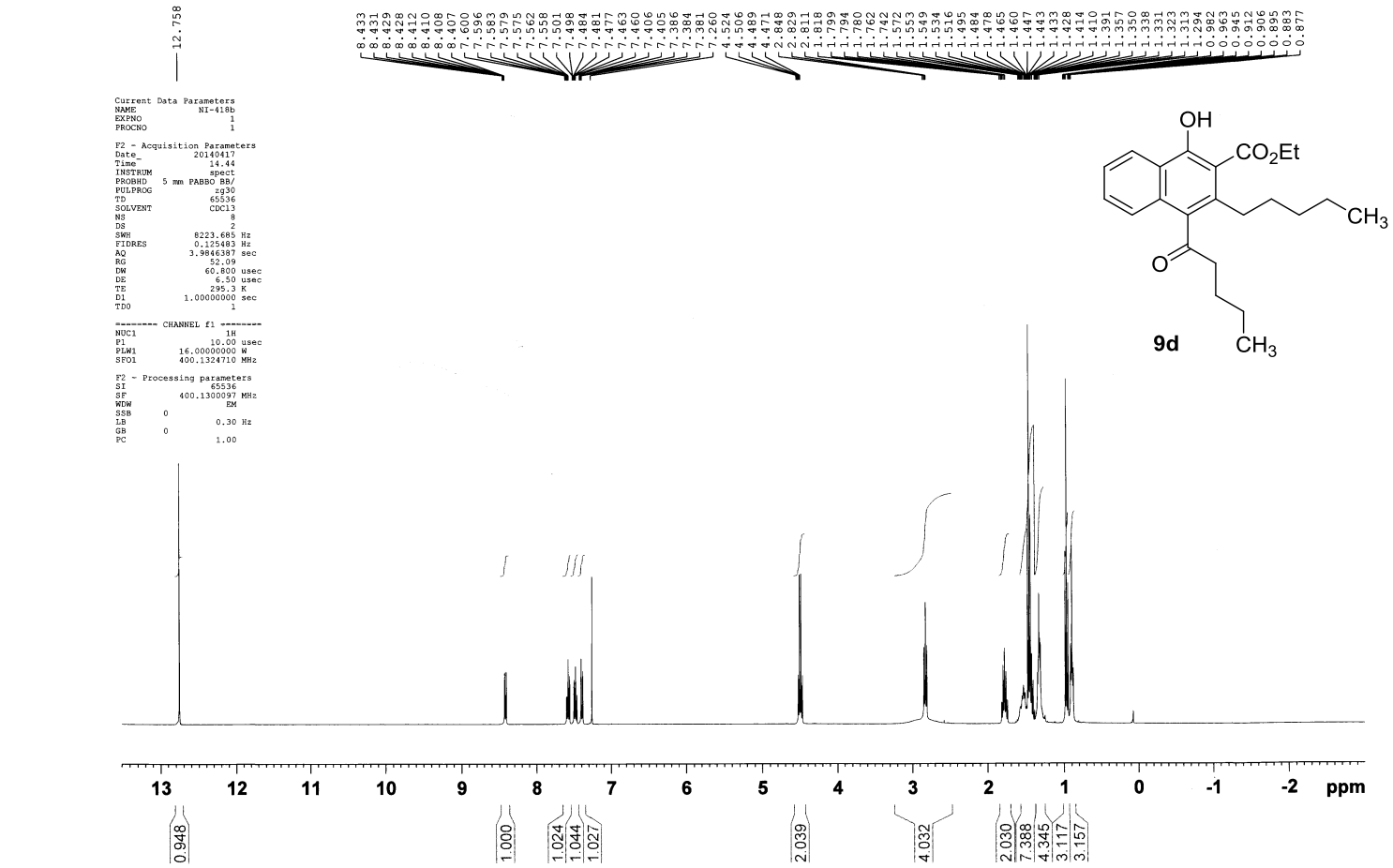
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz

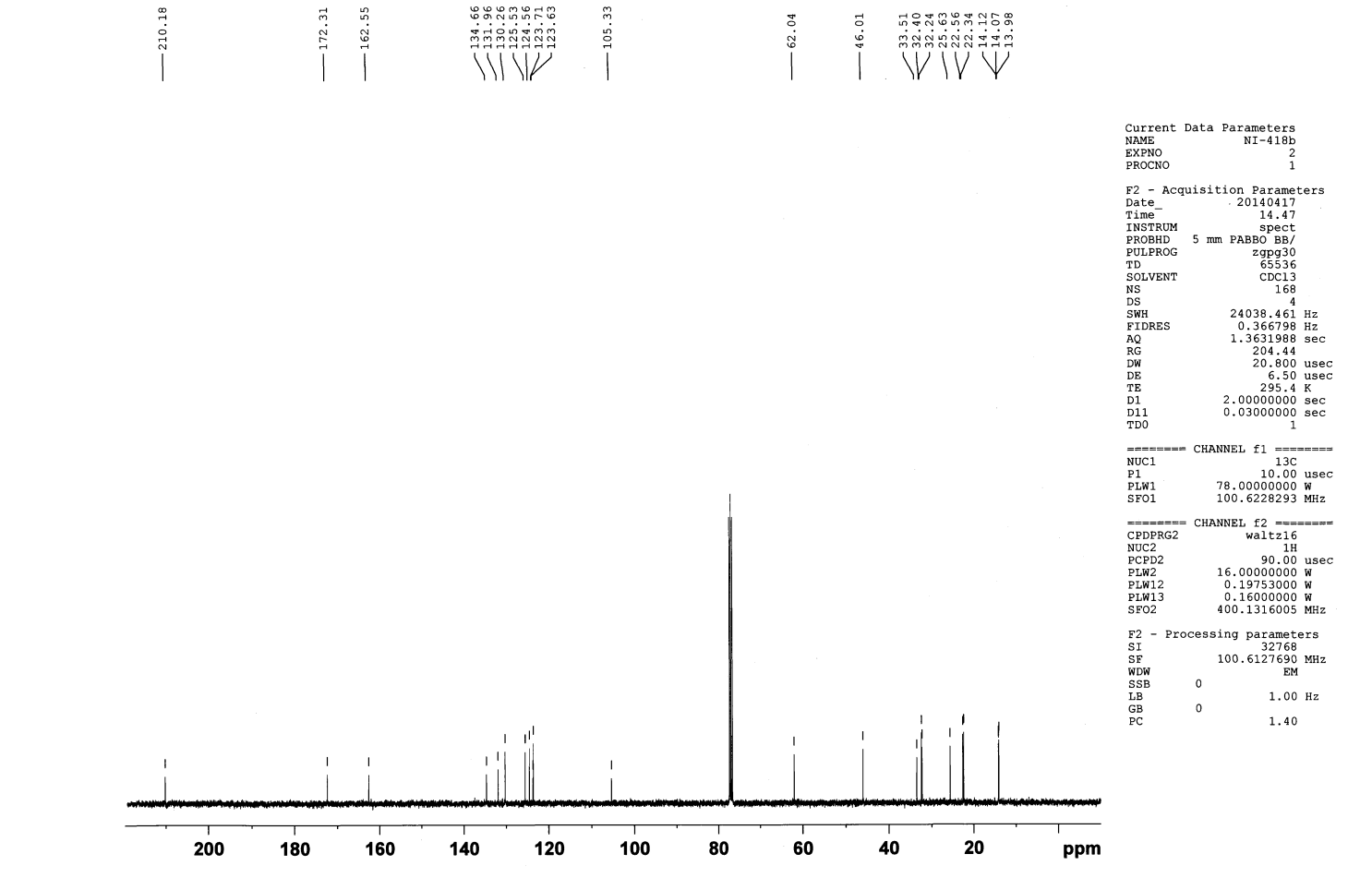
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PLW2 7.09999990 W
PLW12 0.24961001 W
PLW13 0.15975000 W
SFO2 400.1316005 MHz

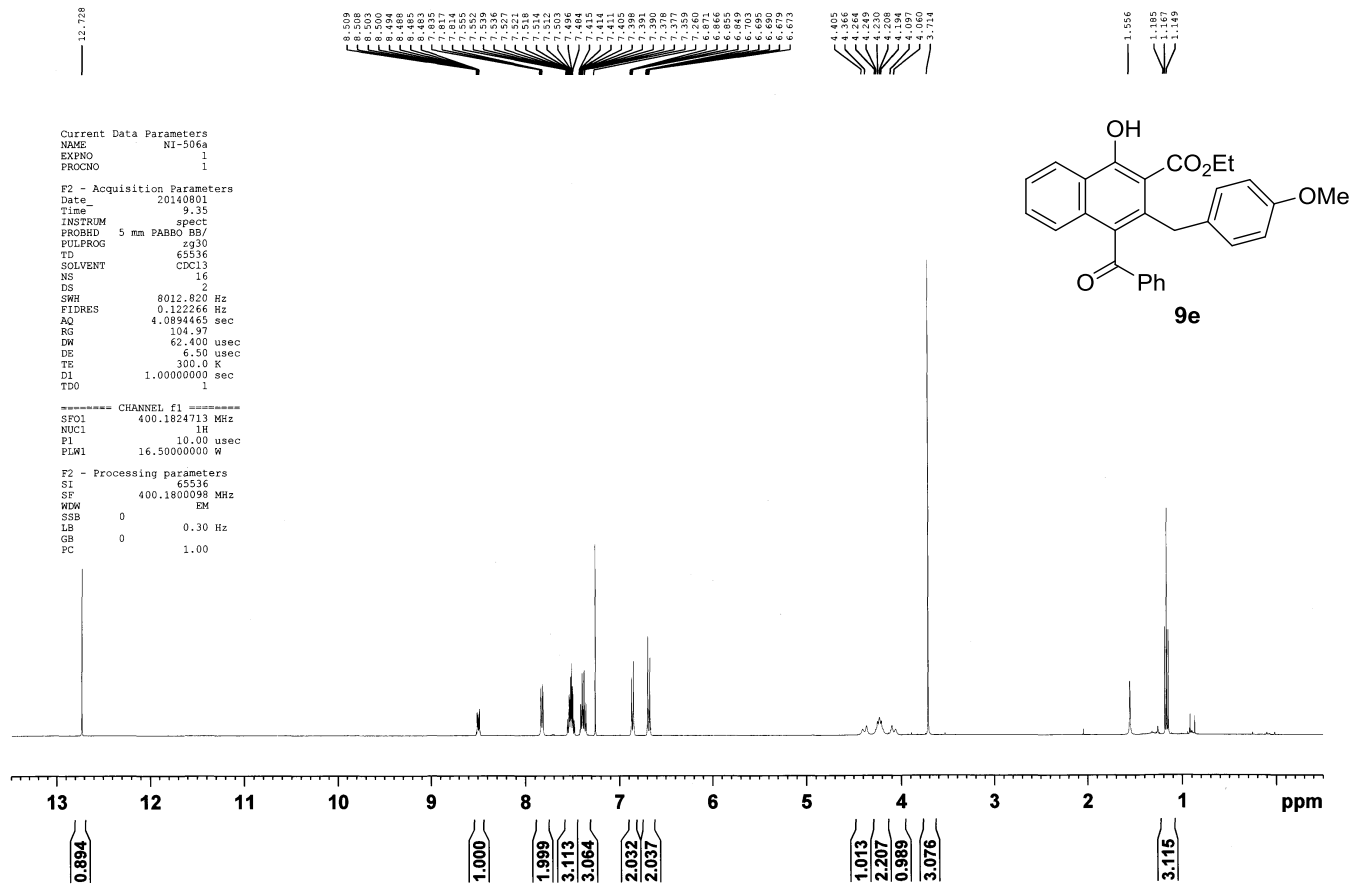
F2 - Processing parameters
SI 32768
SF 100.6127708 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

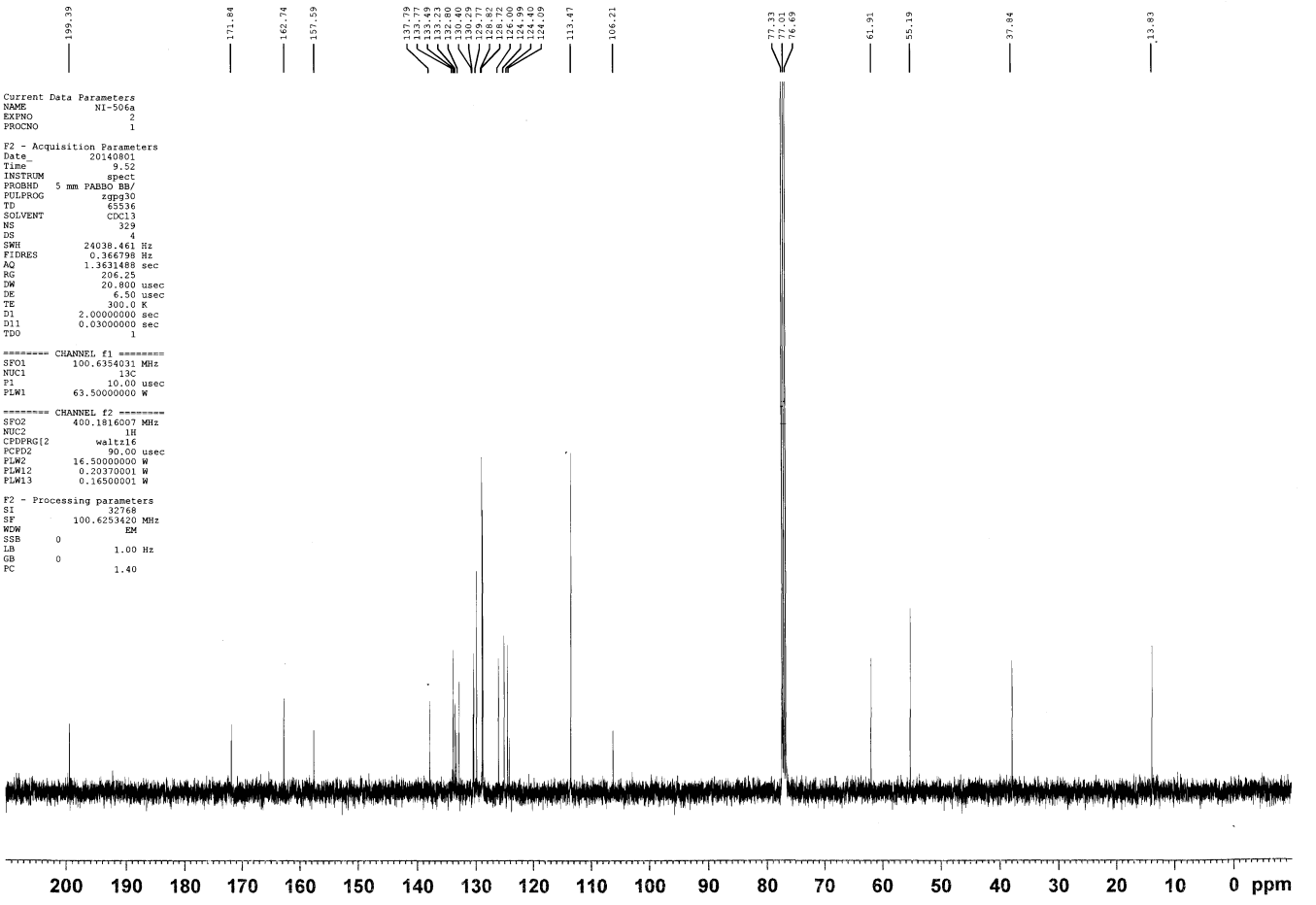

12.799
8.495
8.475
7.650
7.633
7.614
7.568
7.549
7.530
7.481
7.461
7.260
7.235
7.217
7.198
7.151
7.133
7.115
7.000
6.982
4.374
4.202
4.184
4.166
4.148
2.701
1.697
1.679
1.660
1.642
1.623
1.327
1.309
1.290
1.271
1.253
1.235
1.081
1.063
1.045
0.844
0.825
0.807
Current Data Parameters
NAME NI-357a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131026
Time 16.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
Ph
O
9c
CH3
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.967
1.000
1.016
1.135
1.014
2.027
1.032
2.032
2.008
2.091
1.995
2.244
2.332
3.053
3.136

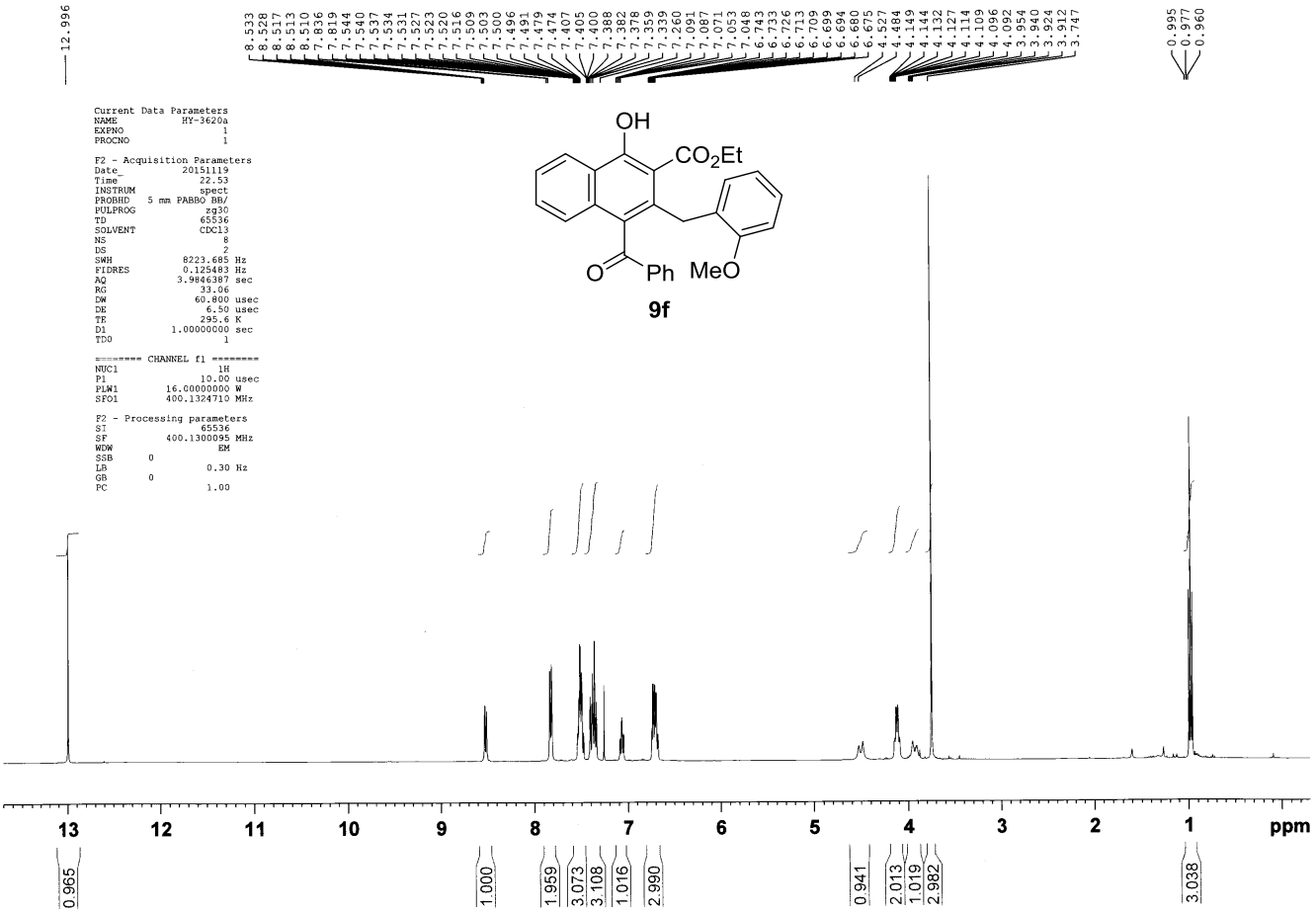

210.17
171.79
162.67
140.87
133.75
132.00
130.62
130.41
128.28
127.66
126.03
125.81
124.64
124.27
123.95
105.96
77.35
77.04
76.72
61.87
45.77
38.20
25.40
22.14
13.79
13.60
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME NI-357a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131026
Time 16.17
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 259
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

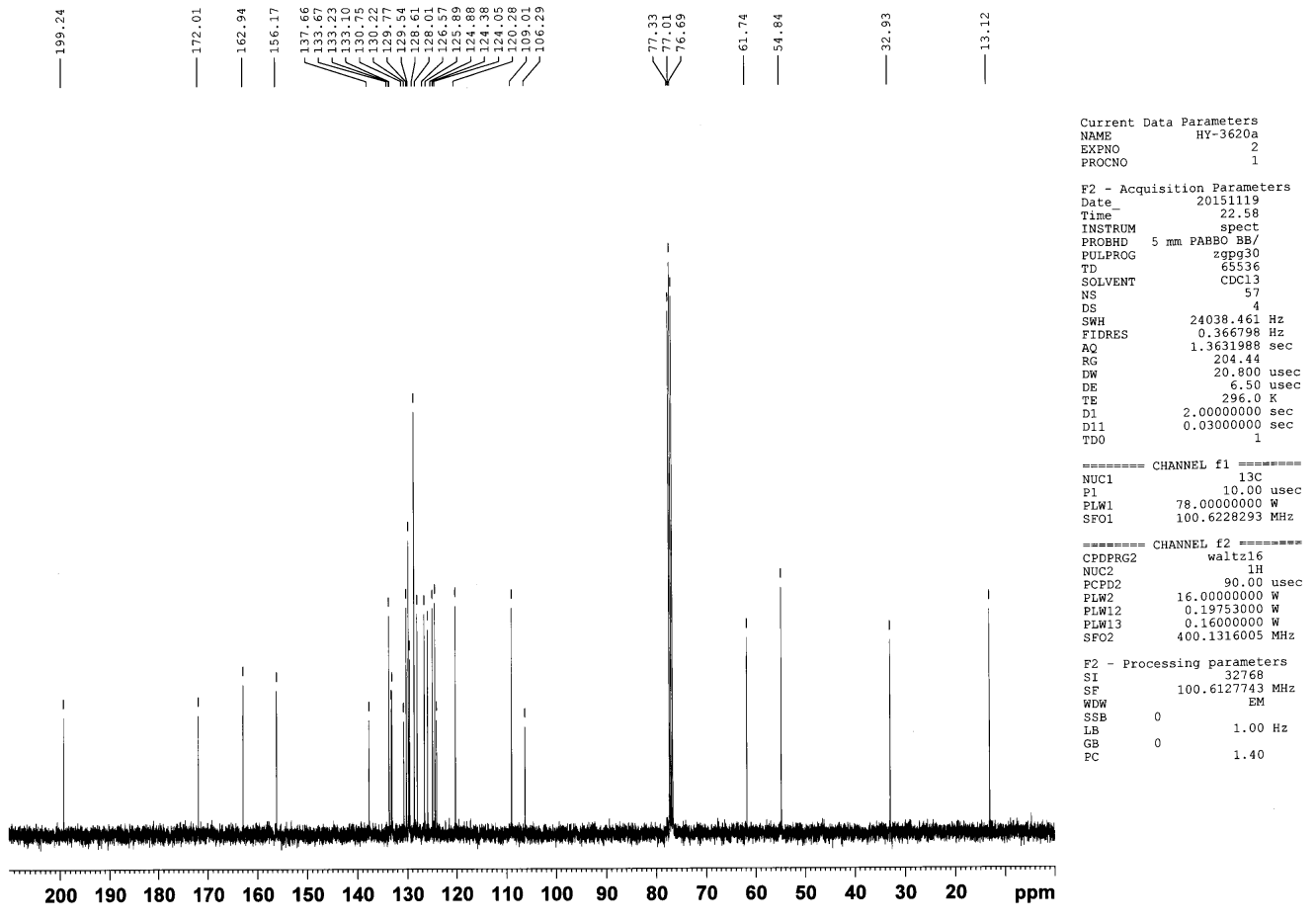

12.758
8.433
8.431
8.429
8.428
8.412
8.410
8.408
8.407
7.600
7.596
7.583
7.579
7.575
7.562
7.558
7.501
7.498
7.484
7.481
7.477
7.463
7.460
7.406
7.405
7.386
7.384
7.381
7.260
4.524
4.506
4.489
4.471
2.848
2.829
2.811
1.818
1.799
1.794
1.780
1.762
1.742
1.572
1.553
1.549
1.534
1.516
1.495
1.484
1.478
1.465
1.460
1.447
1.443
1.433
1.428
1.414
1.410
1.391
1.357
1.350
1.338
1.331
1.323
1.313
1.294
0.982
0.963
0.945
0.912
0.906
0.895
0.883
0.877
Current Data Parameters
NAME NI-418b
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140417
Time 14.44
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 52.09
DW 60.800 usec
DE 6.50 usec
TE 295.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300097 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
CH3
O
9d
CH3
13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1 -2 ppm
0.948
1.000
1.024
1.044
1.027
2.039
4.032
2.030
7.388
4.345
3.117
3.157

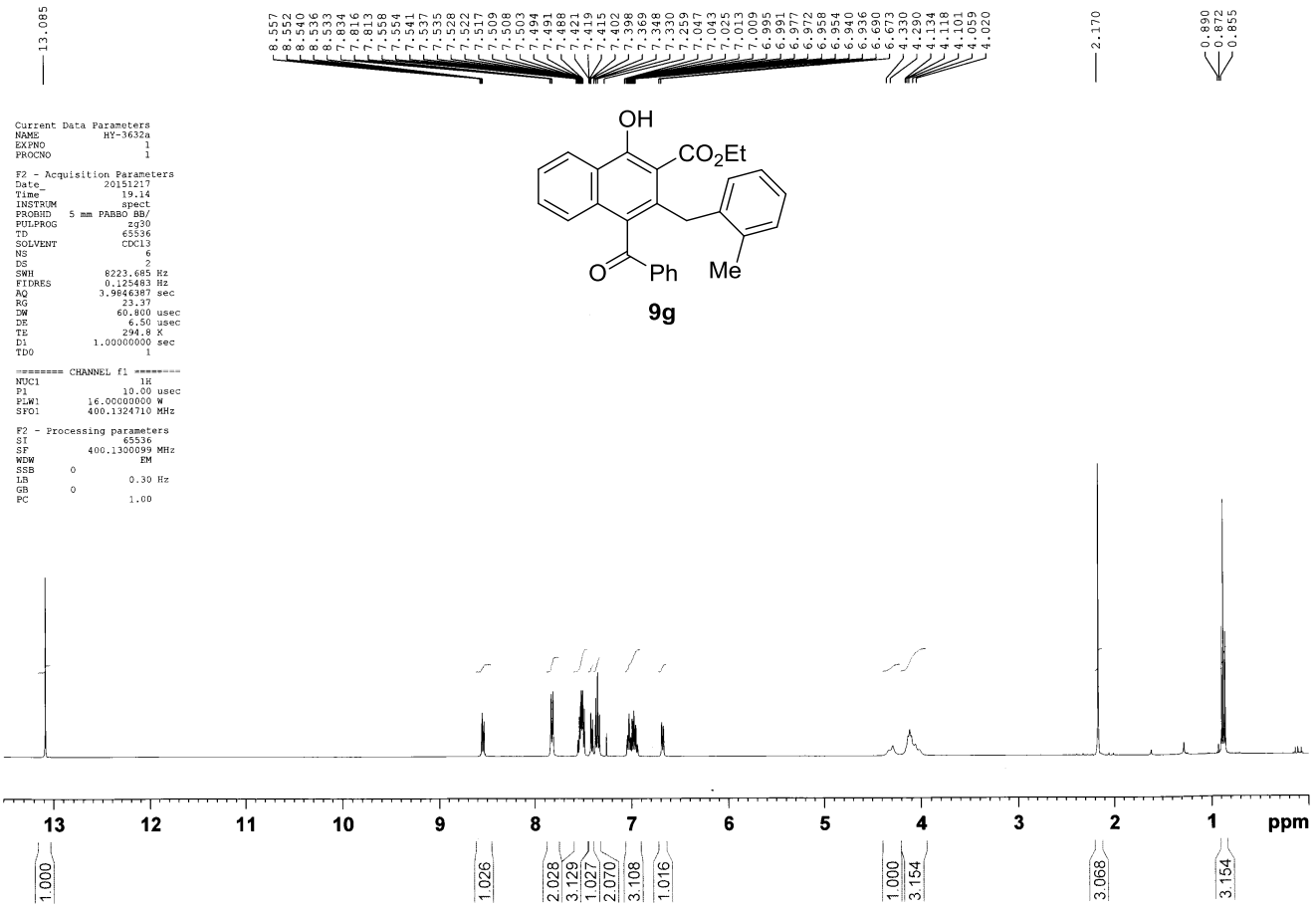

210.18
172.31
162.55
134.66
131.96
130.26
125.53
124.56
123.71
123.63
105.33
62.04
46.01
33.51
32.40
32.24
25.63
22.56
22.34
14.12
14.07
13.98
Current Data Parameters
NAME NI-418b
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140417
Time 14.47
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 168
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127690 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 180 160 140 120 100 80 60 40 20 ppm

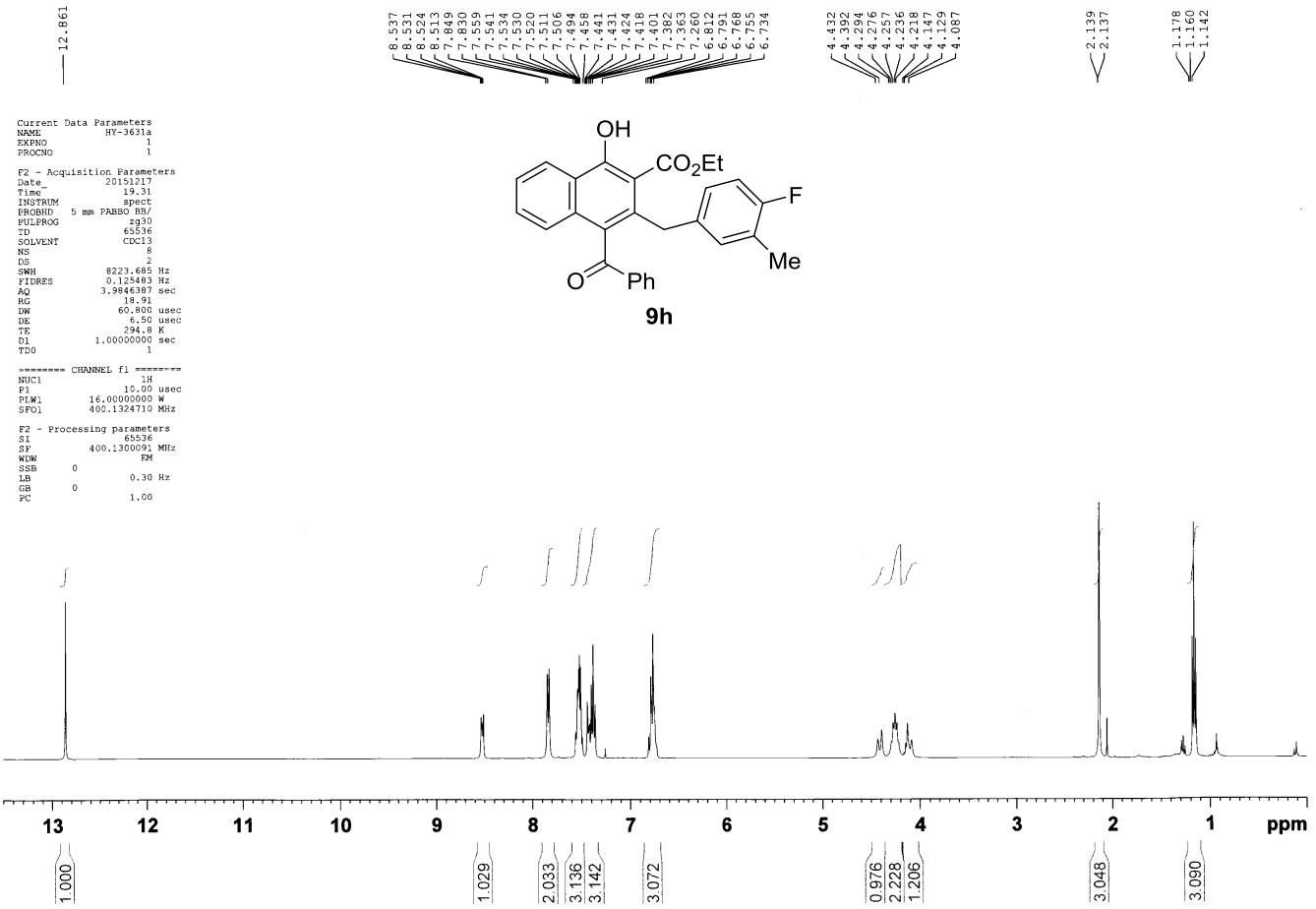

12.728
8.509
8.508
8.505
8.500
8.494
8.488
8.483
7.835
7.817
7.814
7.555
7.552
7.539
7.536
7.527
7.521
7.518
7.514
7.512
7.503
7.496
7.486
7.415
7.414
7.411
7.400
7.398
7.391
7.390
7.378
7.377
7.359
7.260
6.872
6.866
6.855
6.849
6.695
6.690
6.679
6.673
4.405
4.396
4.264
4.249
4.230
4.208
4.194
4.097
4.060
3.714
1.556
1.185
1.167
1.149
Current Data Parameters
NAME NI-506a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140801
Time 9.35
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894465 sec
RG 104.97
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1824713 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1800098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
OMe
O
Ph
9e
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.894
1.000
1.999
3.113
3.064
2.032
2.037
1.013
2.207
0.989
3.076
3.115

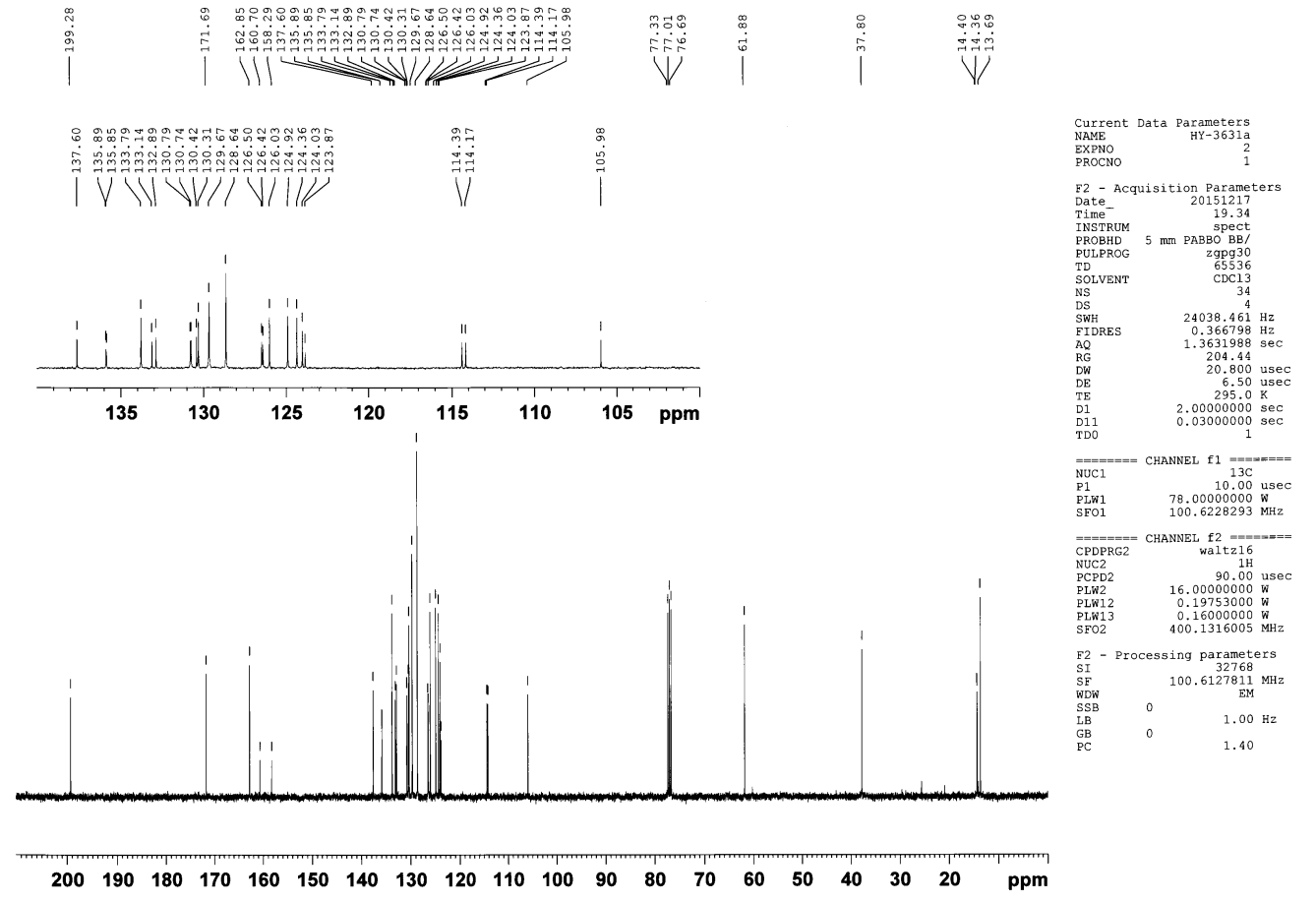

199.39
171.84
162.74
157.59
137.79
133.77
133.49
133.23
132.80
130.40
130.09
129.77
128.82
128.72
126.90
126.43
124.40
124.09
113.47
106.21
77.33
77.01
76.69
61.91
55.19
37.84
13.83
Current Data Parameters
NAME NI-506a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140801
Time 9.52
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 329
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631488 sec
RG 206.25
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 100.6354031 MHz
NUC1 13C
P1 10.00 usec
PLW1 63.50000000 W
======== CHANNEL f2 ========
SFO2 400.1816007 MHz
NUC2 1H
CPDPRG[2 waltz16
PCPD2 90.00 usec
PLW2 16.50000000 W
PLW12 0.20370001 W
PLW13 0.16500001 W
F2 - Processing parameters
SI 32768
SF 100.6253420 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

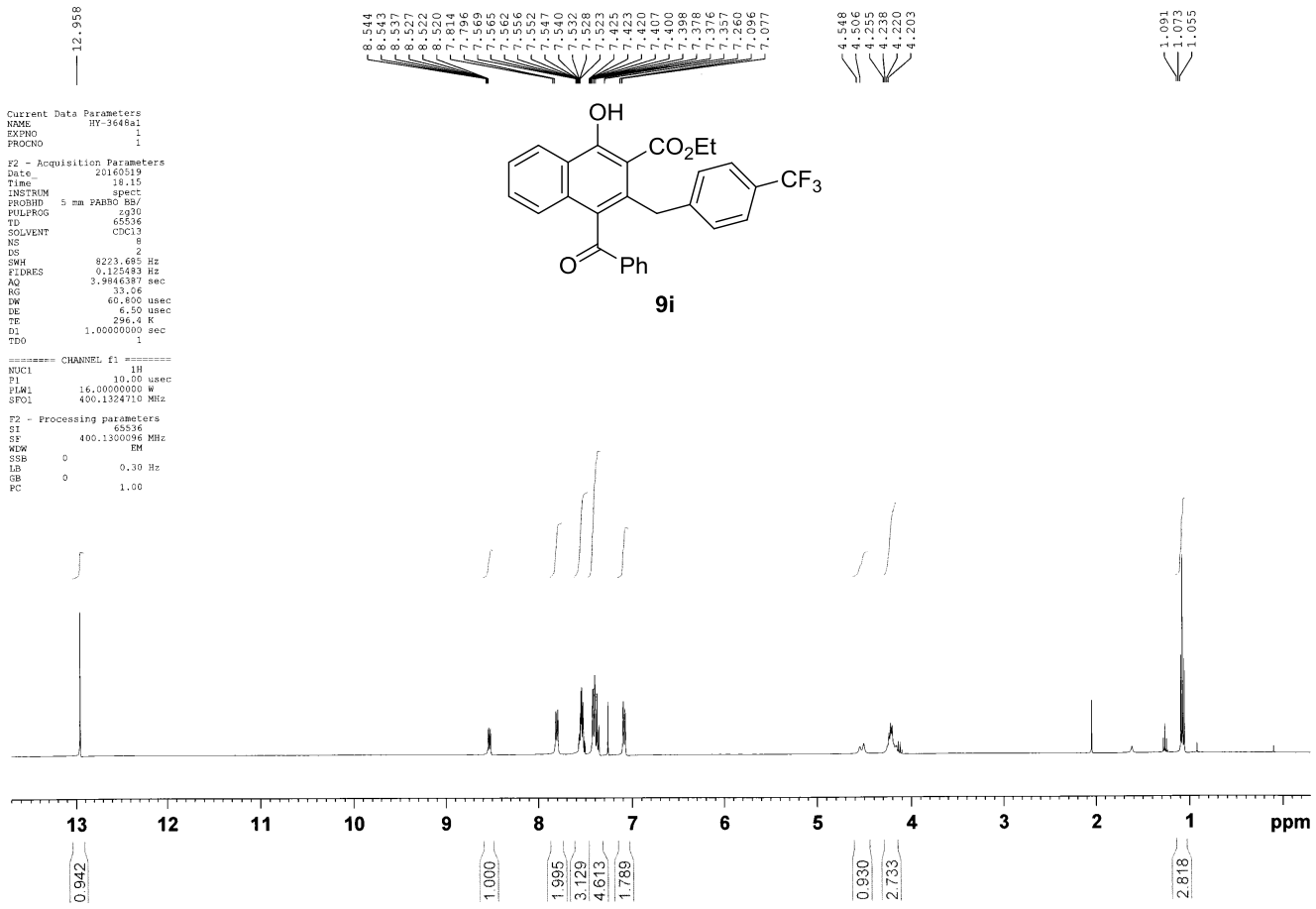

12.996
8.533
8.528
8.517
8.513
8.510
7.836
7.819
7.544
7.540
7.537
7.534
7.531
7.527
7.523
7.520
7.516
7.509
7.503
7.500
7.496
7.491
7.479
7.474
7.407
7.405
7.400
7.388
7.382
7.378
7.359
7.339
7.320
7.261
7.091
7.087
7.071
7.053
7.048
6.743
6.733
6.726
6.713
6.709
6.699
6.694
6.680
6.675
4.527
4.484
4.149
4.144
4.132
4.127
4.114
4.109
4.096
4.092
3.954
3.940
3.924
3.912
3.747
0.995
0.977
0.960
Current Data Parameters
NAME HY-3620a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151119
Time 22.53
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 295.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
O
Ph
MeO
9f
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.965
1.000
1.959
3.073
3.108
1.016
2.990
0.941
2.013
1.019
2.982
3.038

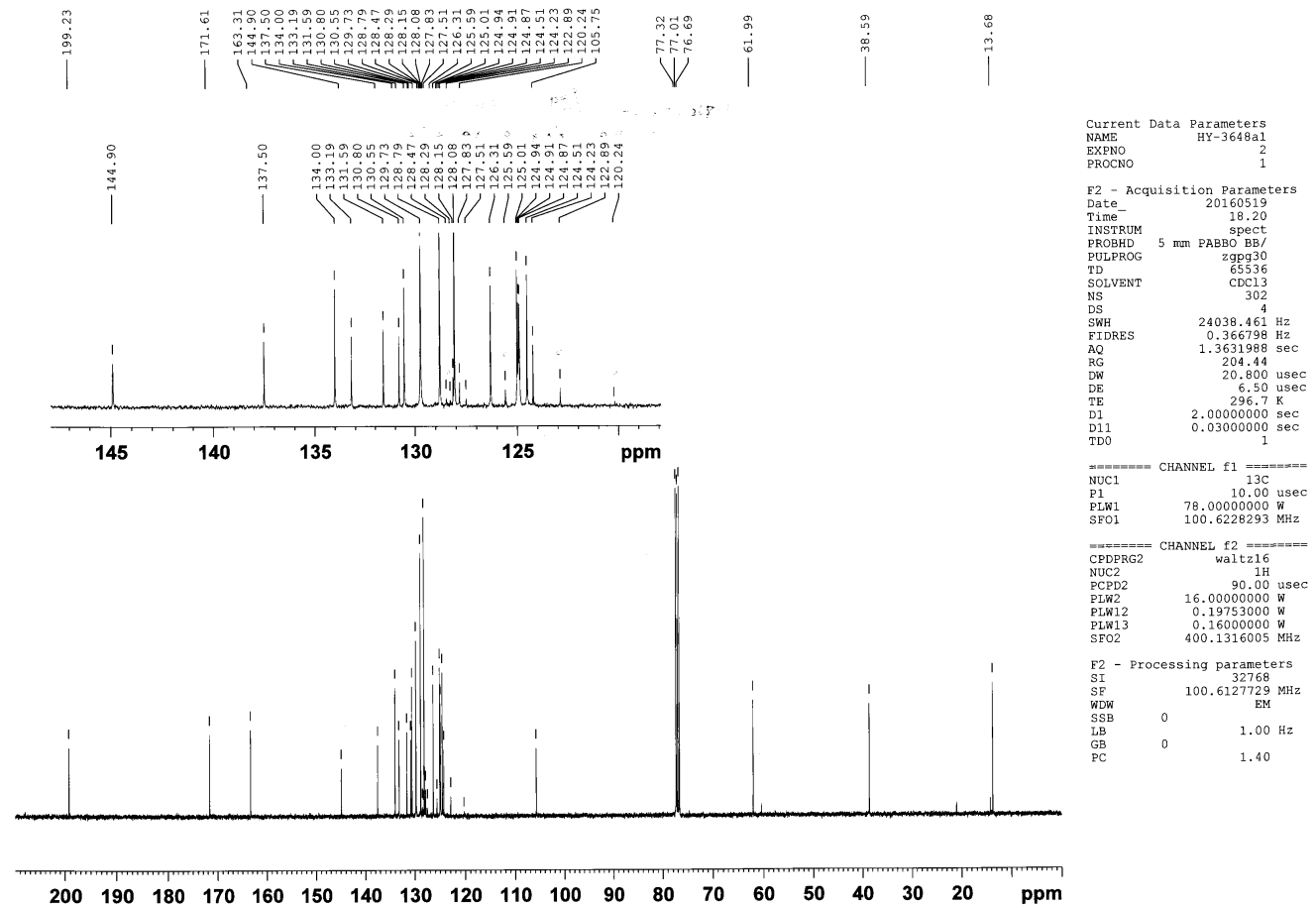

199.24
172.01
162.94
156.17
137.66
133.67
133.23
133.10
130.75
130.22
129.77
129.54
128.61
128.01
126.57
125.89
124.88
124.38
124.05
120.28
109.01
106.29
77.33
77.01
76.69
61.74
54.84
32.93
13.12
Current Data Parameters
NAME HY-3620a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151119
Time 22.58
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 57
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127743 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm

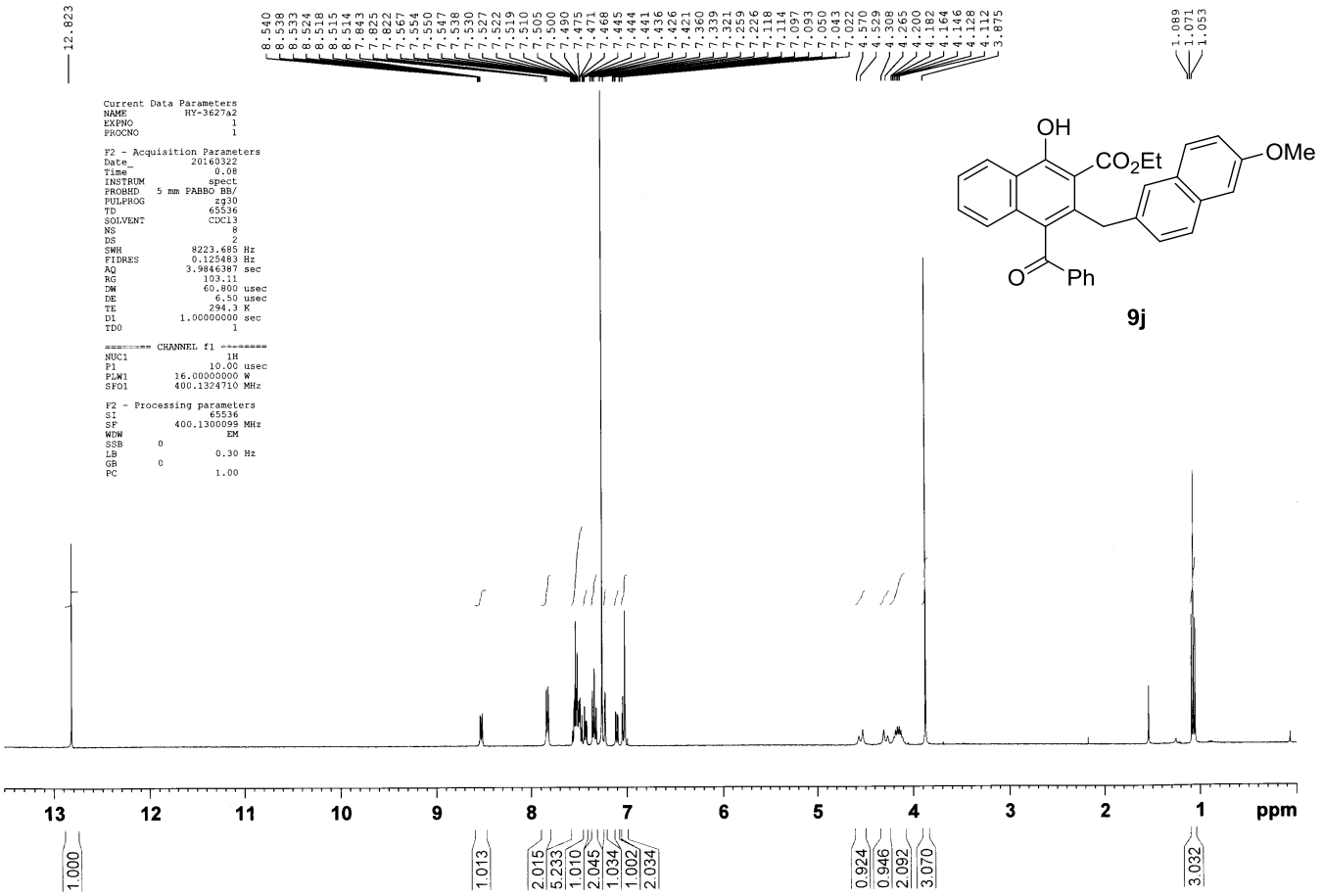

13.085
8.557
8.552
8.540
8.536
8.533
7.834
7.816
7.813
7.558
7.554
7.541
7.537
7.535
7.528
7.522
7.517
7.509
7.508
7.503
7.494
7.491
7.488
7.421
7.419
7.415
7.402
7.398
7.369
7.348
7.330
7.259
7.047
7.043
7.025
7.013
7.009
6.995
6.991
6.977
6.972
6.958
6.954
6.940
6.936
6.690
6.673
4.330
4.290
4.134
4.118
4.101
4.059
4.020
2.170
0.890
0.872
0.855
OH
CO2Et
O
Ph
Me
9g
Current Data Parameters
NAME HY-3632a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151217
Time 19.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 6
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 23.37
DW 60.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.000
1.026
2.028
3.129
1.027
2.070
3.108
1.016
1.000
3.154
3.068
3.154
199.26
171.91
163.11
139.23
137.56
134.80
133.74
133.31
132.83
130.65
130.30
129.67
129.24
128.62
126.66
125.98
125.79
125.48
124.92
124.42
124.08
106.30
77.33
77.01
76.69
61.80
36.42
19.63
13.16
140 135 130 125 ppm
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Current Data Parameters
NAME HY-3632a
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151217
Time 19.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 11
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 294.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127773 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

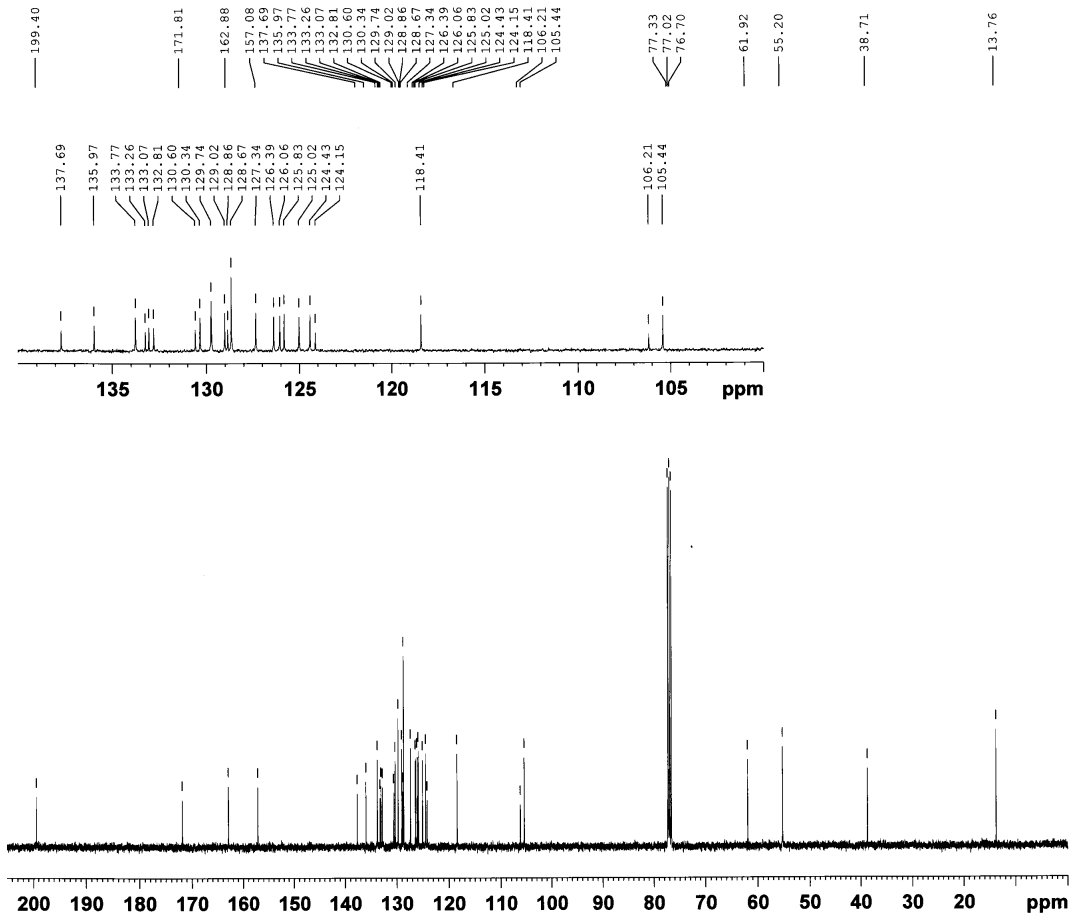

12.861
8.537
8.531
8.524
8.513
7.849
7.830
7.559
7.541
7.534
7.530
7.520
7.511
7.506
7.494
7.458
7.441
7.431
7.424
7.418
7.401
7.382
7.363
7.260
6.812
6.791
6.768
6.755
6.734
4.432
4.392
4.294
4.276
4.257
4.236
4.218
4.147
4.129
4.087
2.139
2.137
1.178
1.160
1.142
OH
CO2Et
F
Me
O
Ph
9h
Current Data Parameters
NAME HY-3631a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151217
Time 19.31
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 18.91
DW 60.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300091 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.000
1.029
2.033
3.136
3.142
3.072
0.976
2.228
1.206
3.048
3.090

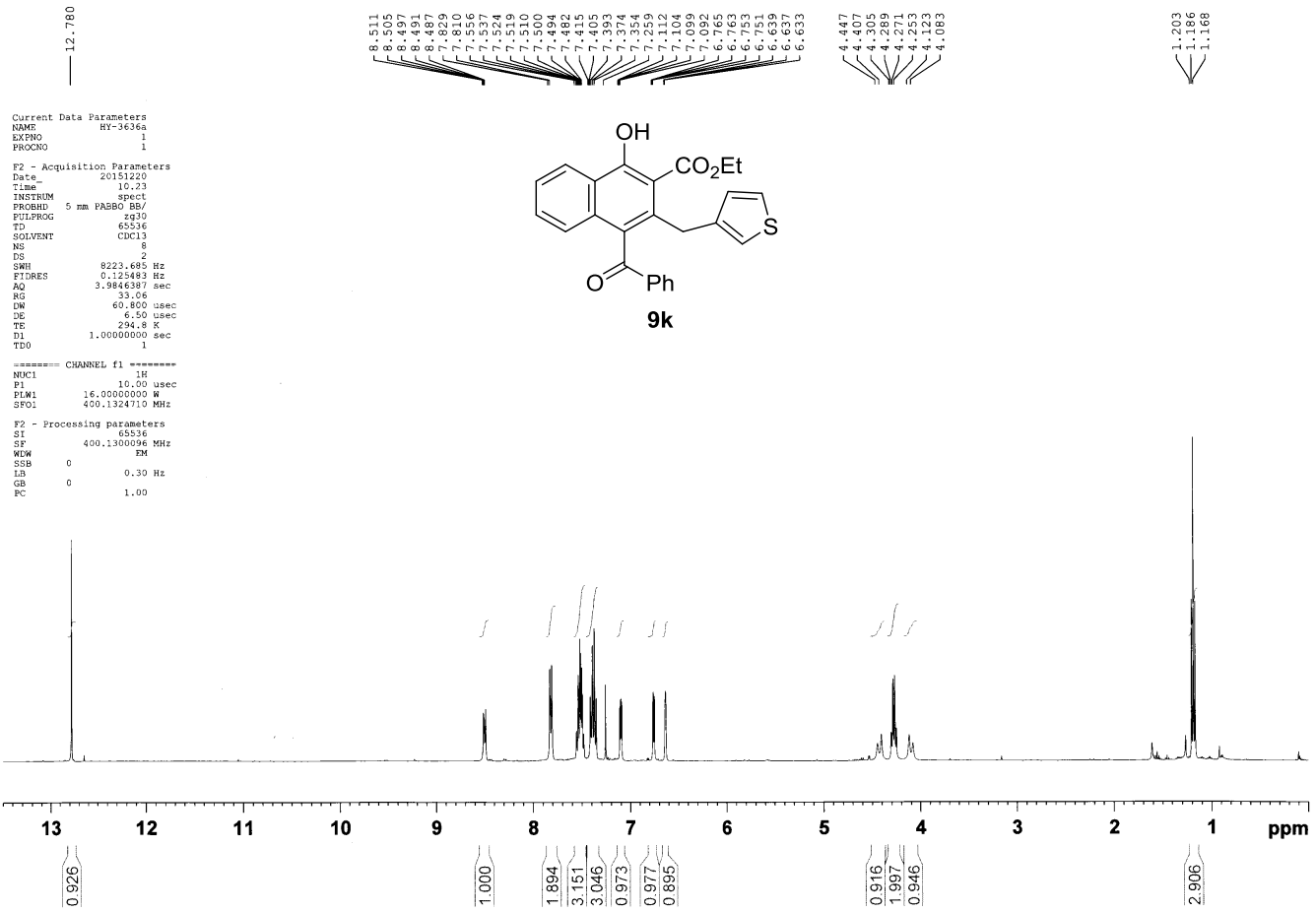

199.28
171.69
162.85
160.70
158.29
137.60
135.89
135.85
133.79
133.14
132.89
130.79
130.74
130.42
130.31
129.67
128.64
126.50
126.42
126.03
124.92
124.36
124.03
123.87
114.39
114.17
105.98
77.33
77.01
76.69
61.88
37.80
14.40
14.36
13.69
135 130 125 120 115 110 105 ppm
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Current Data Parameters
NAME HY-3631a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151217
Time 19.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 34
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127811 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


12.958
8.544
8.543
8.537
8.527
8.522
8.520
7.814
7.796
7.569
7.565
7.562
7.556
7.552
7.547
7.540
7.532
7.528
7.523
7.425
7.423
7.420
7.407
7.400
7.398
7.376
7.367
7.360
7.096
7.077
4.548
4.506
4.255
4.238
4.220
4.203
1.091
1.073
1.055
OH
CO2Et
CF3
O
Ph
9i
Current Data Parameters
NAME HY-3648a1
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160519
Time 18.15
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.695 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 296.4 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.942 1.000 1.995 3.129 4.613 1.789 0.930 2.733 2.818


199.23
171.61
163.31
144.90
137.50
134.00
133.19
131.59
130.80
130.55
129.73
128.79
128.47
128.29
128.15
128.08
127.83
127.51
126.31
125.59
125.01
124.94
124.91
124.87
124.51
124.23
122.89
120.24
105.75
77.32
77.01
76.69
61.99
38.59
13.68
145 140 135 130 125 ppm
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Current Data Parameters
NAME HY-3648a1
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160519
Time 18.20
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 302
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.7 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127729 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


12.823
8.540
8.538
8.533
8.524
8.518
8.515
8.514
7.843
7.825
7.822
7.567
7.554
7.550
7.547
7.538
7.530
7.527
7.522
7.519
7.510
7.505
7.500
7.490
7.475
7.471
7.468
7.445
7.444
7.441
7.436
7.421
7.360
7.339
7.321
7.251
7.226
7.118
7.114
7.097
7.093
7.050
7.043
7.022
4.570
4.529
4.308
4.265
4.200
4.182
4.164
4.146
4.128
4.112
3.875
1.089
1.071
1.053
Current Data Parameters
NAME HY-3627a2
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160322
Time 0.08
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 103.11
DW 60.800 usec
DE 6.50 usec
TE 294.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
OMe
O
Ph
9j
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
1.000
1.013
2.015
5.233
1.010
2.045
1.034
1.002
2.034
0.924
0.946
2.092
3.070
3.032


199.40
171.81
162.88
157.08
137.69
135.97
133.77
133.26
133.07
132.81
130.60
130.34
129.74
129.02
128.86
128.67
127.34
126.39
126.06
125.83
125.02
124.43
124.15
118.41
106.21
105.44
77.33
77.02
76.70
61.92
55.20
38.71
13.76
135
130
125
120
115
110
105
ppm
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm
Current Data Parameters
NAME HY-3627a2
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20160322
Time 0.27
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 162
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 294.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127739 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

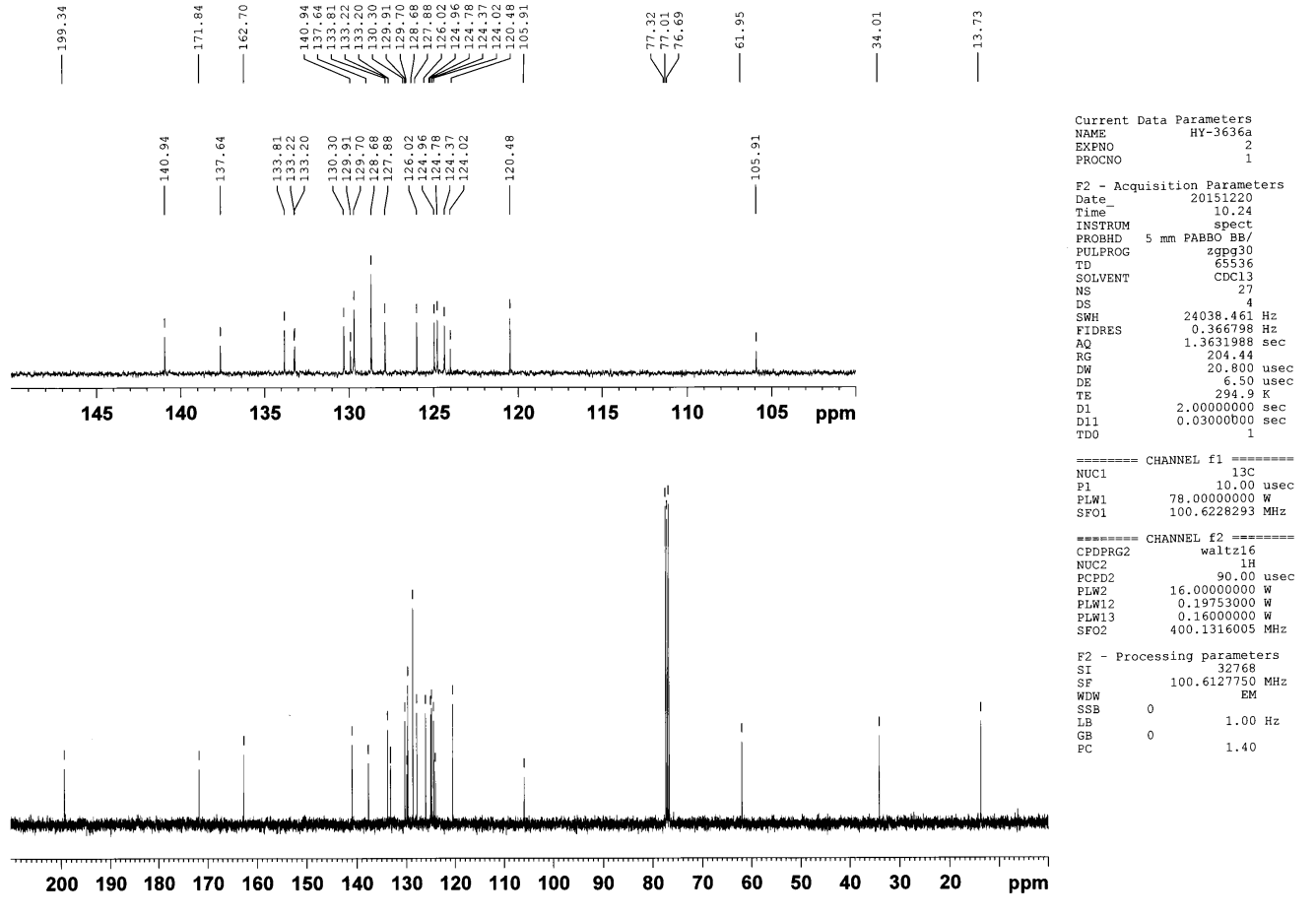


Current Data Parameters
NAME HY-3636a
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20151220
Time 10.23
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1

======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz

F2 - Processing parameters
SI 65536
SF 400.1300096 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



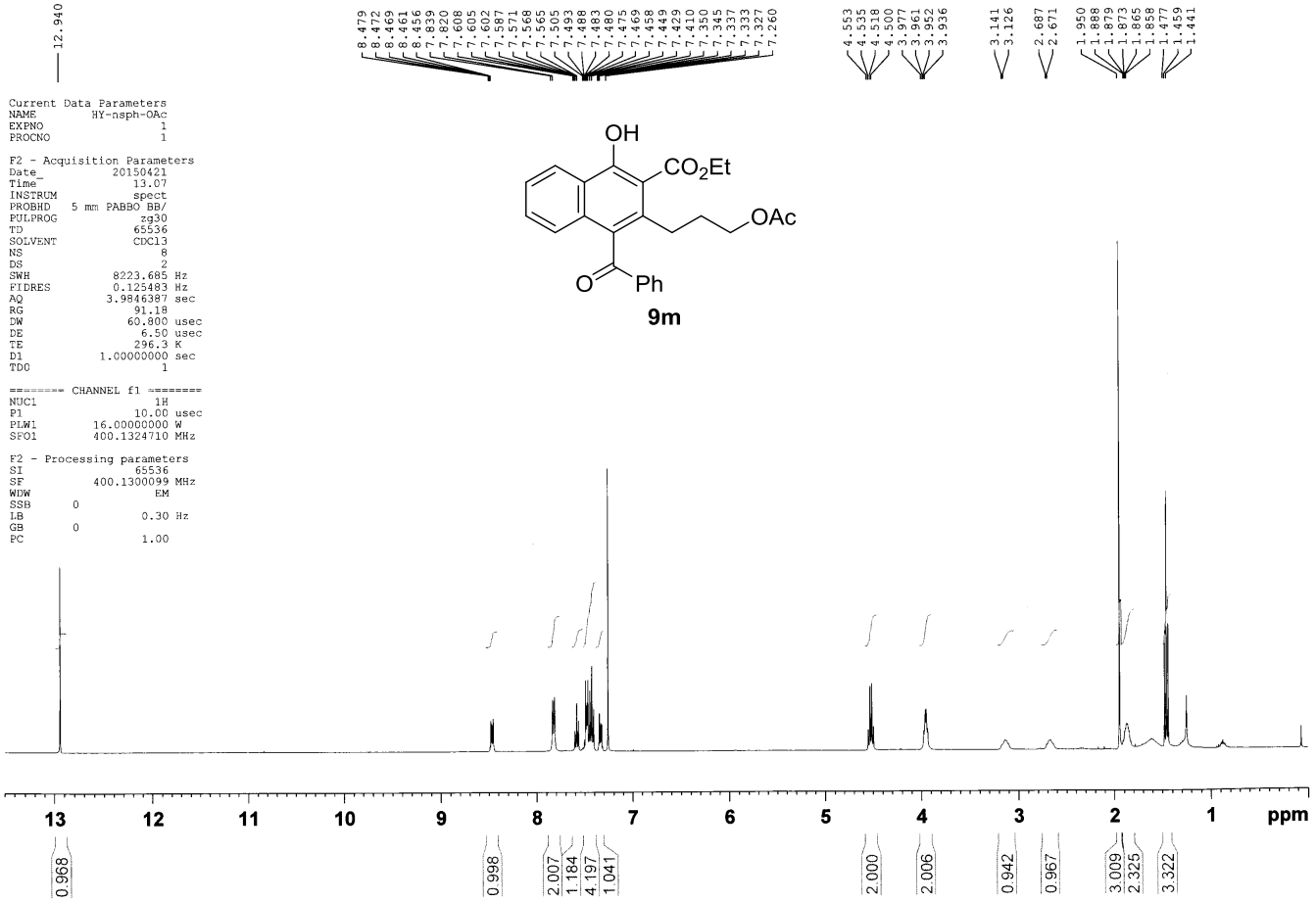
Current Data Parameters
NAME HY-3636a
EXPNO 2
PROCNO 1

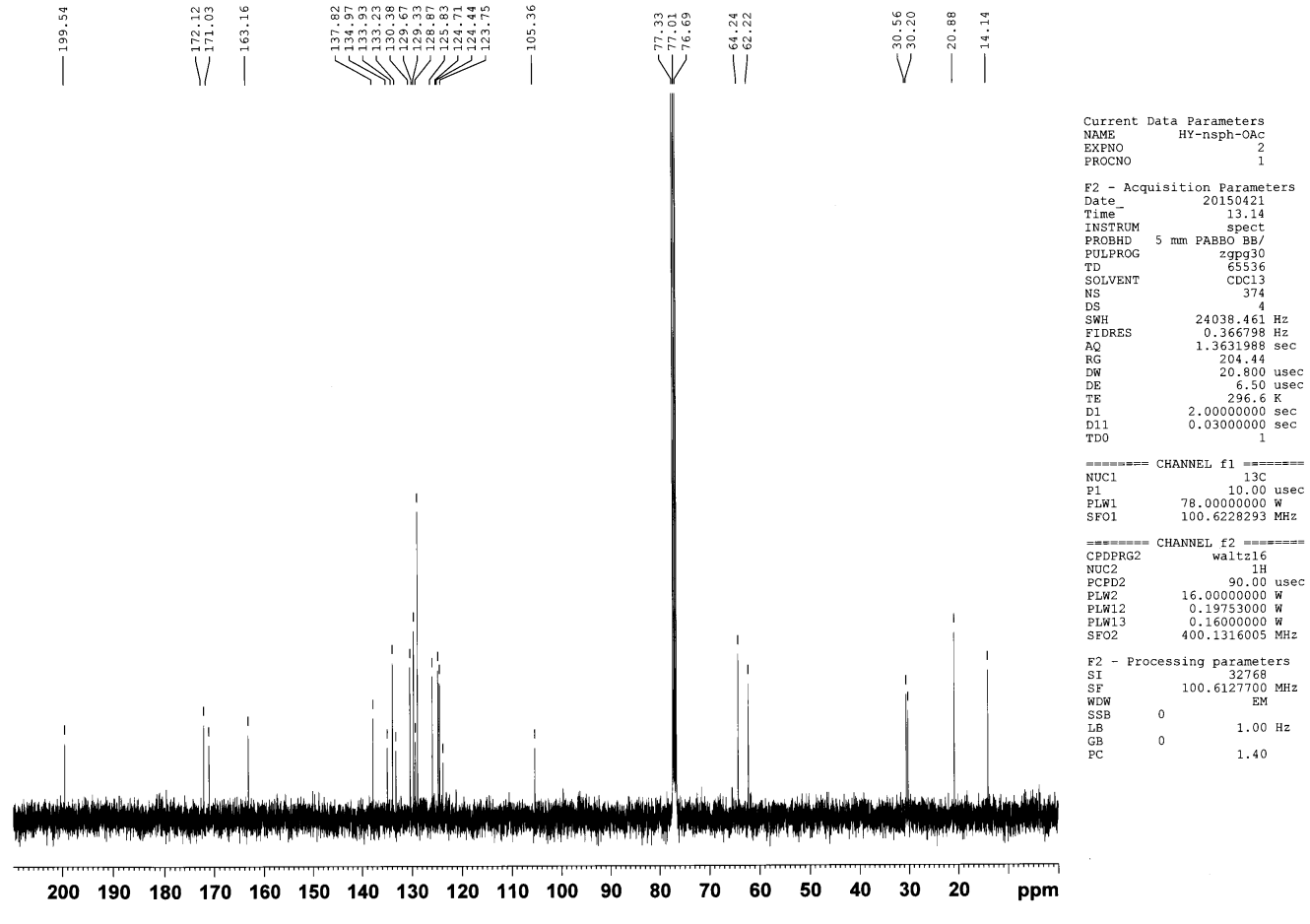
F2 - Acquisition Parameters
Date_ 20151220
Time 10.24
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 27
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 294.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1

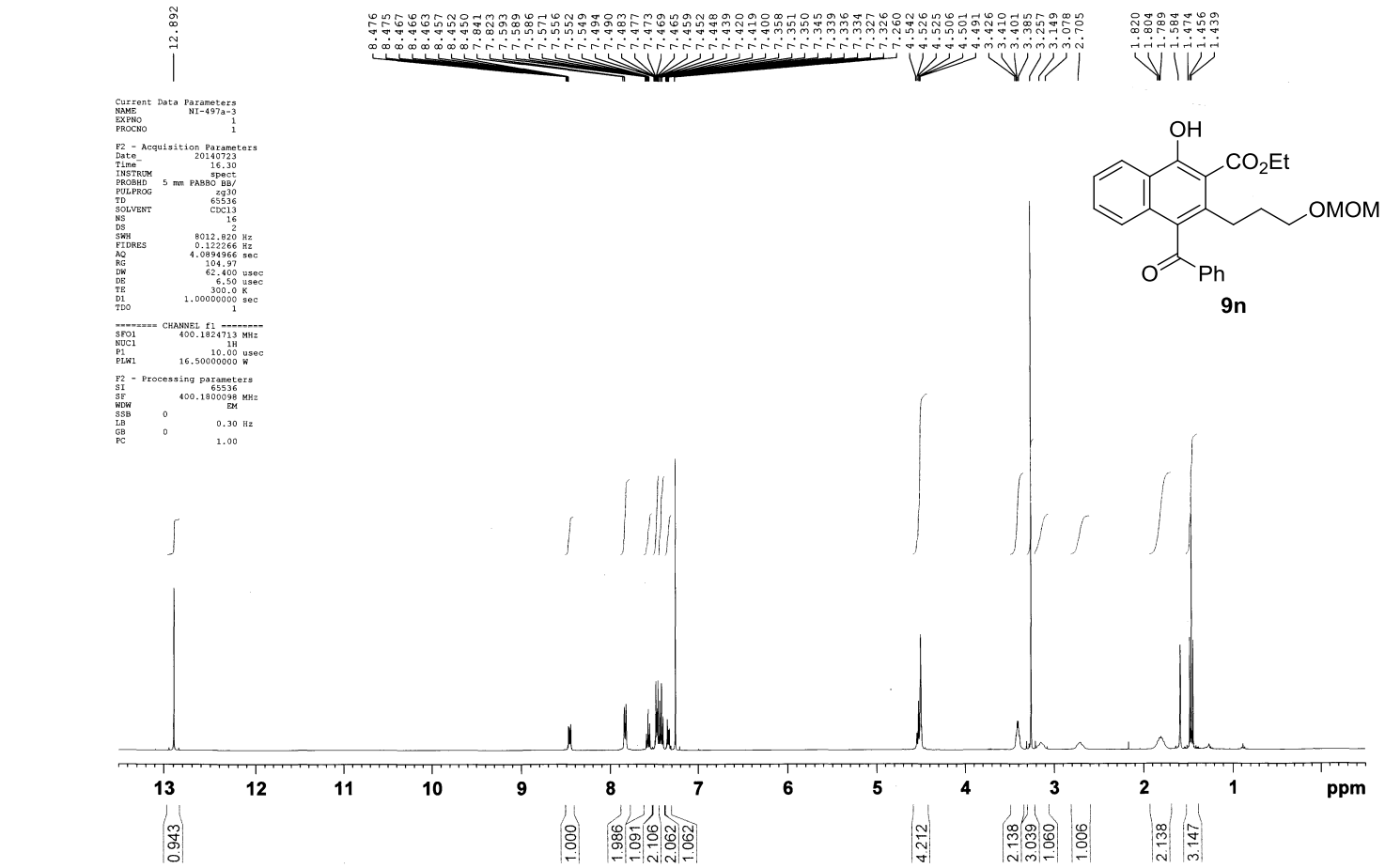
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz

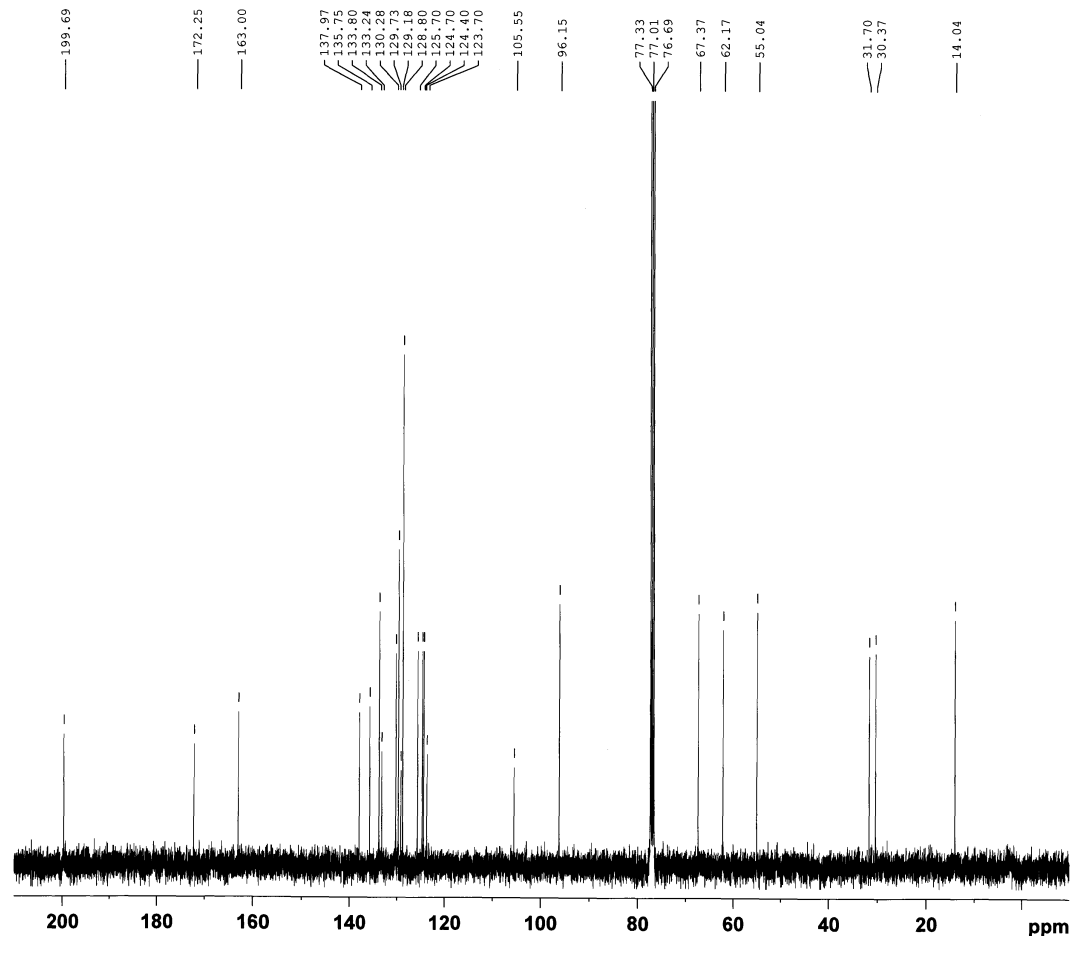
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz

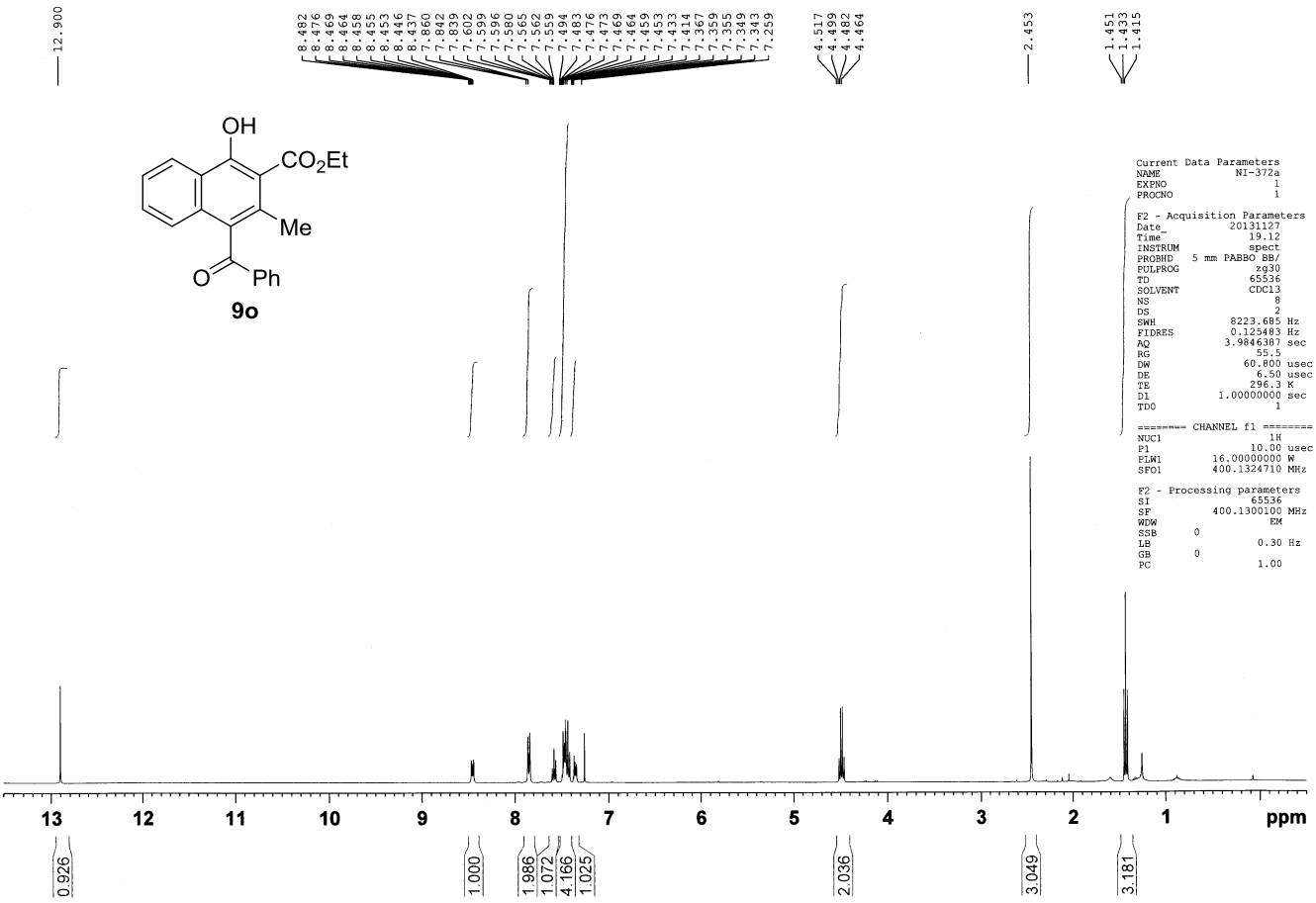
F2 - Processing parameters
SI 32768
SF 100.6127750 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


12.940
8.479
8.472
8.469
8.461
8.456
7.839
7.820
7.608
7.605
7.602
7.587
7.571
7.568
7.565
7.505
7.493
7.488
7.483
7.480
7.475
7.469
7.458
7.449
7.429
7.410
7.350
7.345
7.337
7.333
7.327
7.260
4.553
4.535
4.518
4.500
3.977
3.961
3.952
3.936
3.141
3.126
2.687
2.671
1.950
1.888
1.879
1.873
1.865
1.858
1.477
1.459
1.441
Current Data Parameters
NAME HY-nsph-OAc
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150421
Time 13.07
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 91.18
DW 60.800 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300099 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
OAc
O
Ph
9m
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.968
0.998
2.007
1.184
4.197
1.041
2.000
2.006
0.942
0.967
3.009
2.325
3.322


199.54
172.12
171.03
163.16
137.82
134.97
133.93
133.23
130.38
129.67
129.33
128.87
125.83
124.71
124.44
123.75
105.36
77.33
77.01
76.69
64.24
62.22
30.56
30.20
20.88
14.14
Current Data Parameters
NAME HY-nsph-OAc
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150421
Time 13.14
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 374
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.6 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127700 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
190
180
170
160
150
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


12.892
8.476
8.475
8.467
8.466
8.463
8.457
8.452
8.450
7.841
7.823
7.593
7.589
7.586
7.571
7.556
7.552
7.549
7.494
7.490
7.483
7.477
7.473
7.469
7.465
7.459
7.452
7.448
7.439
7.420
7.419
7.400
7.358
7.351
7.350
7.345
7.339
7.336
7.334
7.327
7.326
7.260
4.542
4.526
4.525
4.506
4.501
4.491
3.426
3.410
3.401
3.385
3.257
3.149
3.078
2.705
1.820
1.804
1.789
1.584
1.474
1.456
1.439
Current Data Parameters
NAME NI-497a-3
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140723
Time 16.30
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8012.820 Hz
FIDRES 0.122266 Hz
AQ 4.0894966 sec
RG 104.97
DW 62.400 usec
DE 6.50 usec
TE 300.0 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
SFO1 400.1824713 MHz
NUC1 1H
P1 10.00 usec
PLW1 16.50000000 W
F2 - Processing parameters
SI 65536
SF 400.1800098 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
OMOM
O
Ph
9n
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
0.943
1.000
1.986
1.091
2.106
2.062
1.062
4.212
2.138
3.039
1.060
1.006
2.138
3.147


199.69
172.25
163.00
137.97
135.75
133.80
133.24
130.28
129.73
129.18
128.80
125.70
124.70
124.40
123.70
105.55
96.15
77.33
77.01
76.69
67.37
62.17
55.04
31.70
30.37
14.04
200
180
160
140
120
100
80
60
40
20
ppm
Current Data Parameters
NAME NI-497a-3
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20140723
Time 16.48
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 300
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 206.25
DW 20.800 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
d11 0.03000000 sec
DELTA 1.89999998 sec
TD0 1
NUC1 13C
P1 10.00 usec
PLW1 63.50000000 W
SFO1 100.6354031 MHz
CPDPRG2
NUC2 1H
PLW2 16.50000000 W
PLW12 0.20370001 W
PLW13 0.16500001 W
SFO2 400.1816007 MHz
F2 - Processing parameters
SI 32768
SF 100.6253420 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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8.464
8.458
8.455
8.453
8.446
8.437
7.860
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7.839
7.602
7.599
7.596
7.580
7.565
7.562
7.559
7.494
7.483
7.476
7.473
7.469
7.464
7.459
7.453
7.433
7.414
7.367
7.359
7.355
7.349
7.343
7.259
4.517
4.499
4.482
4.464
2.453
1.451
1.433
1.415
OH
CO2Et
Me
O
Ph
9o
Current Data Parameters
NAME NI-372a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131127
Time 19.12
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 55.5
DW 60.800 usec
DE 6.50 usec
TE 296.3 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13
12
11
10
9
8
7
6
5
4
3
2
1
ppm
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1.000
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1.072
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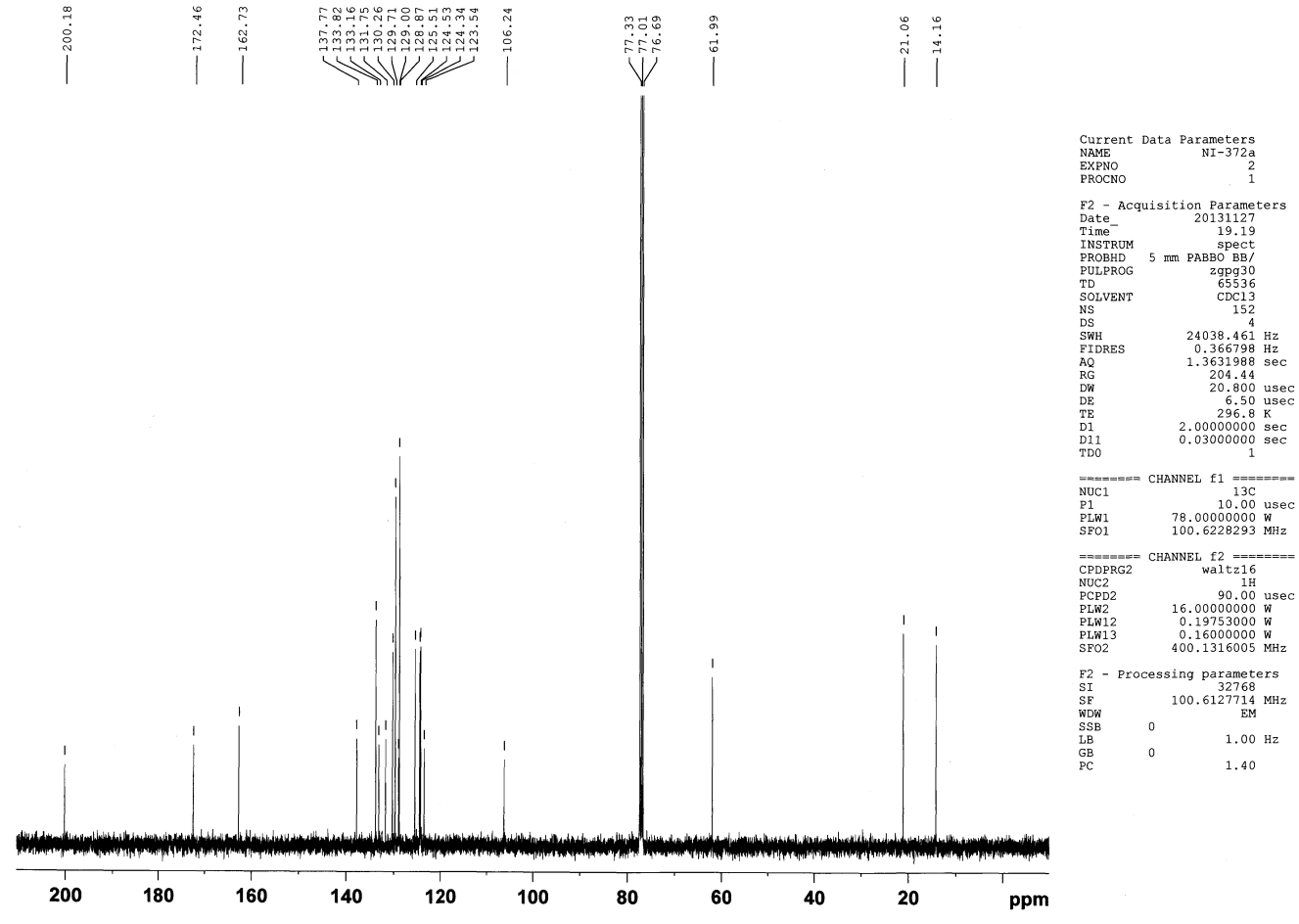
2.036
3.049
3.181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133.82
133.16
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130.26
129.71
129.00
128.87
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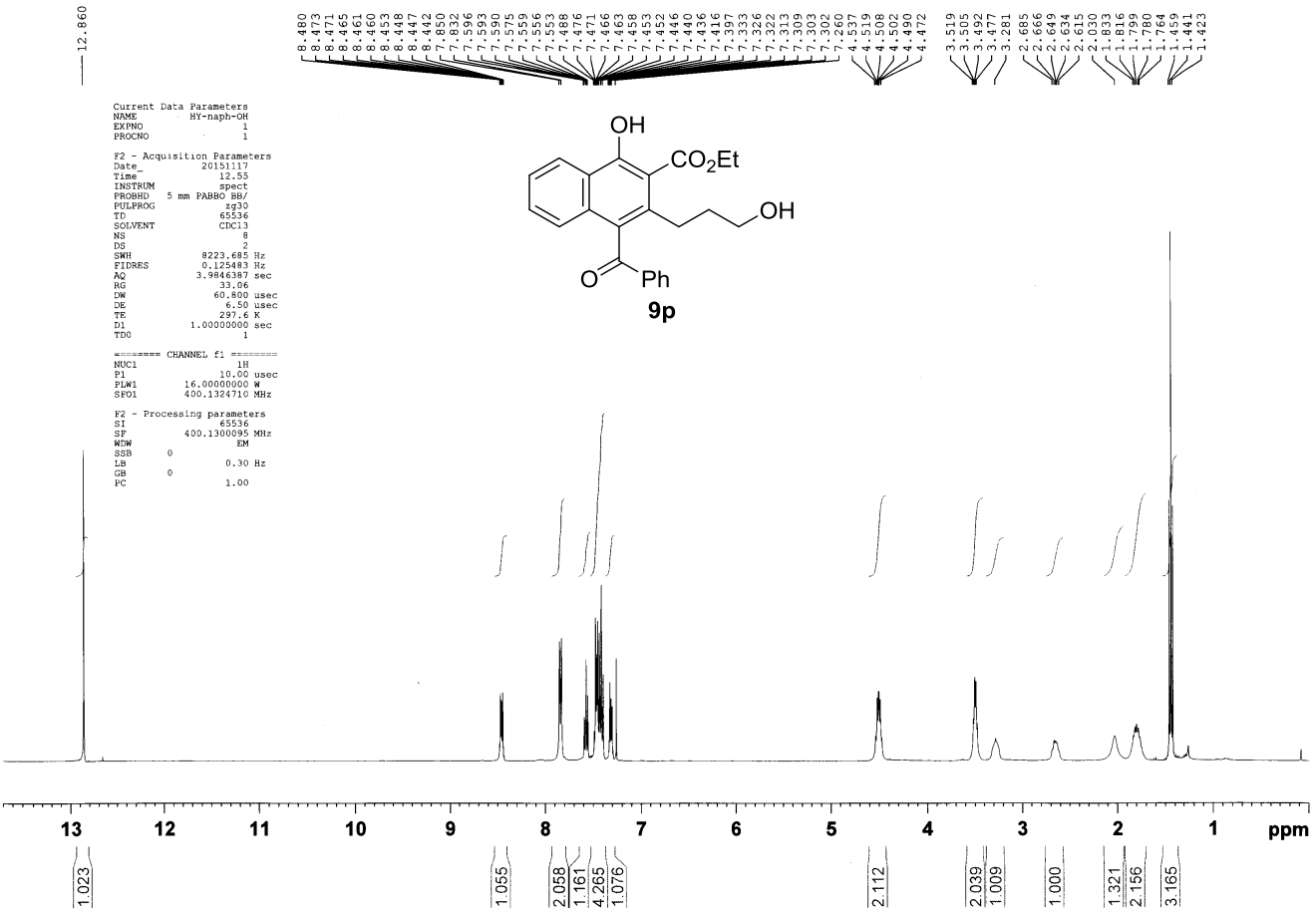
124.53
124.34
123.54
106.24
77.33
77.01
76.69
61.99
21.06
14.16
Current Data Parameters
NAME NI-372a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20131127
Time 19.19
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDC13
NS 152
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.8 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127714 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
200
180
160
140
120
100
80
60
40
20
ppm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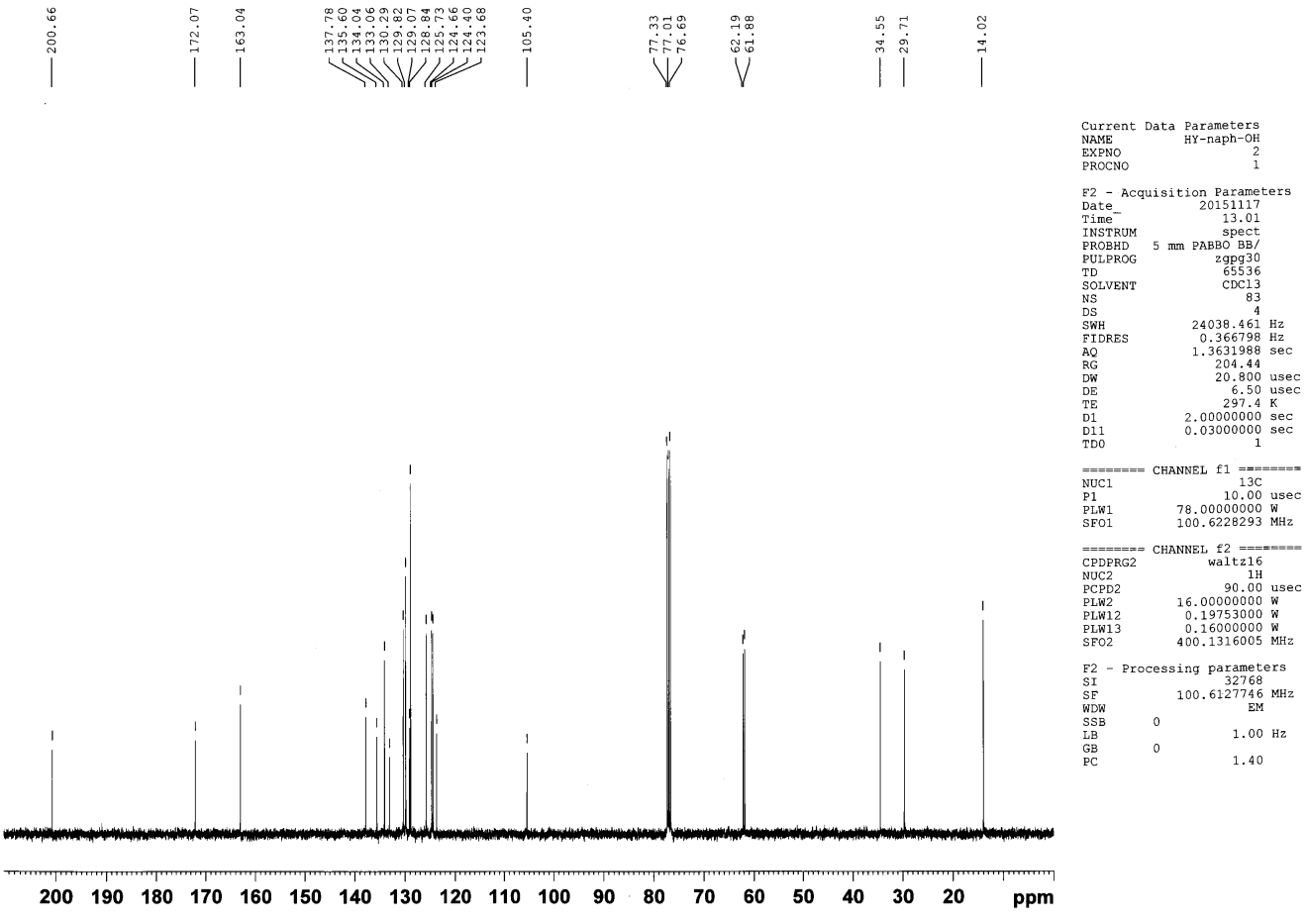
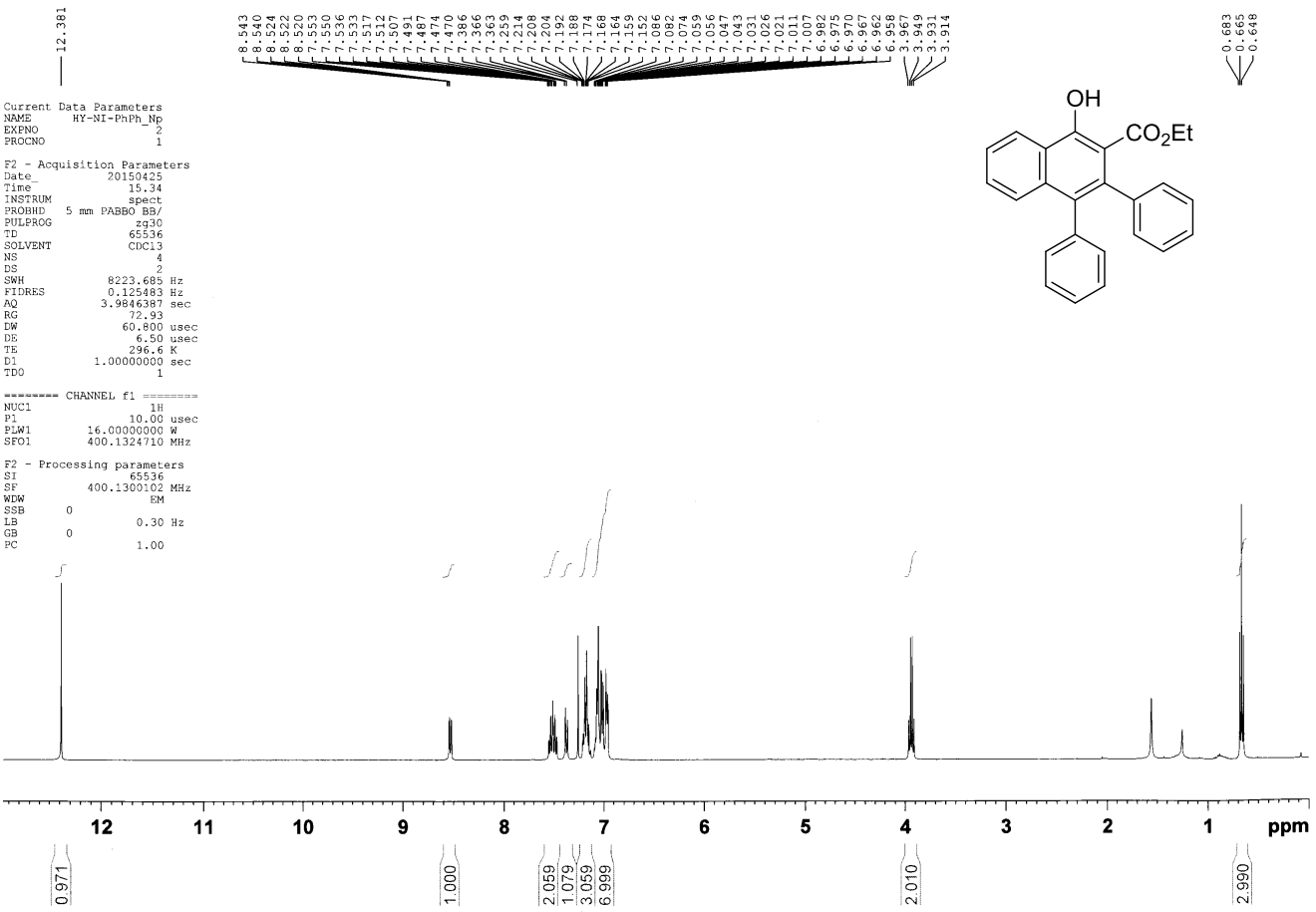
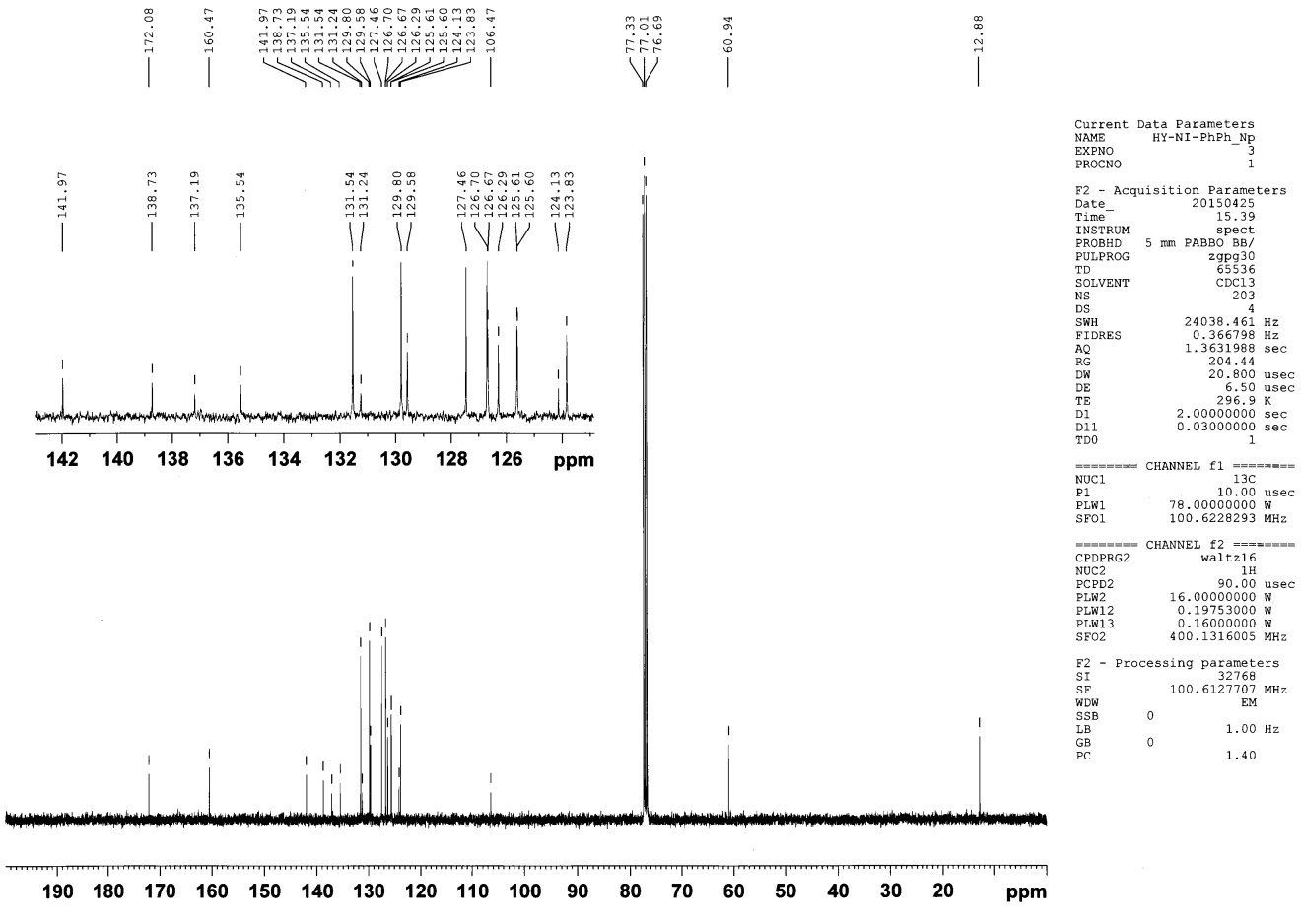
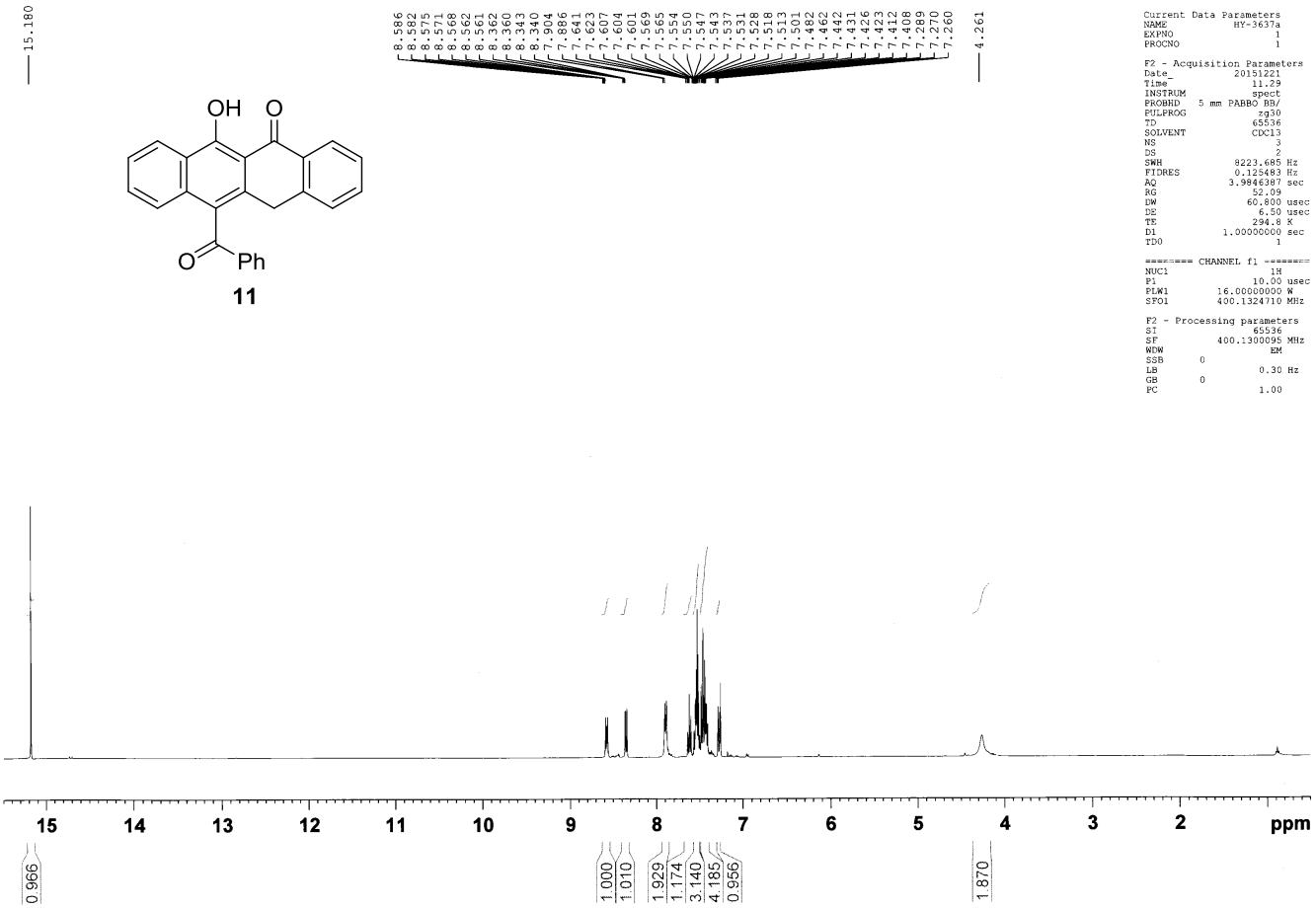
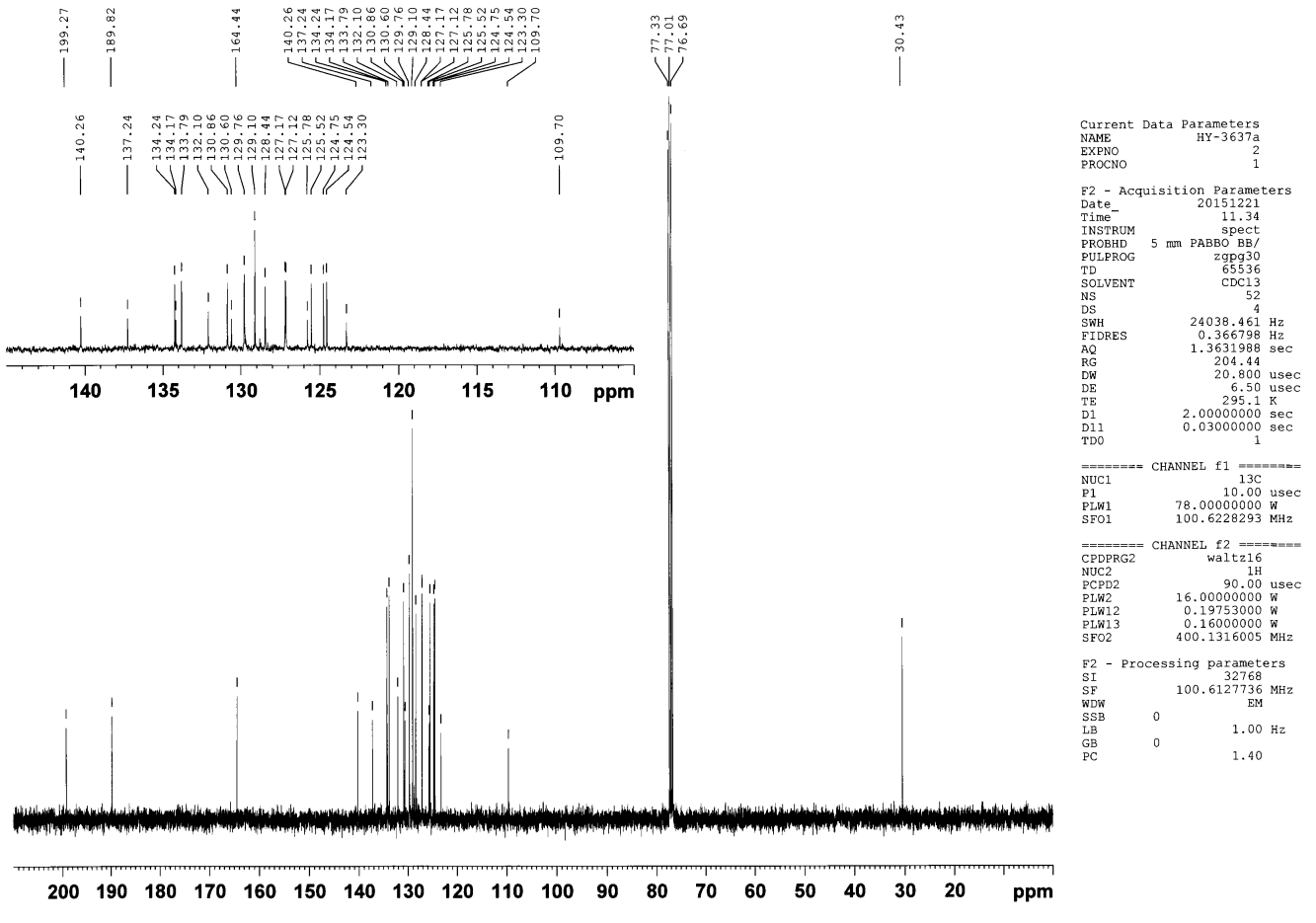
8.461
8.460
8.453
8.448
8.447
8.442
7.850
7.832
7.596
7.593
7.590
7.575
7.559
7.556
7.553
7.488
7.476
7.471
7.466
7.463
7.458
7.453
7.452
7.446
7.440
7.436
7.416
7.397
7.333
7.326
7.322
7.313
7.309
7.303
7.302
7.260
4.537
4.519
4.508
4.502
4.490
4.472
3.519
3.505
3.492
3.477
3.281
2.685
2.666
2.649
2.634
2.615
2.030
1.833
1.816
1.799
1.780
1.764
1.459
1.441
1.423
OH
CO2Et
OH
O
Ph
9p
Current Data Parameters
NAME HY-naph-OH
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151117
Time 12.55
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 33.06
DW 60.800 usec
DE 6.50 usec
TE 297.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.023
1.055
2.058
1.161
4.265
1.076
2.112
2.039
1.009
1.000
1.321
2.156
3.165

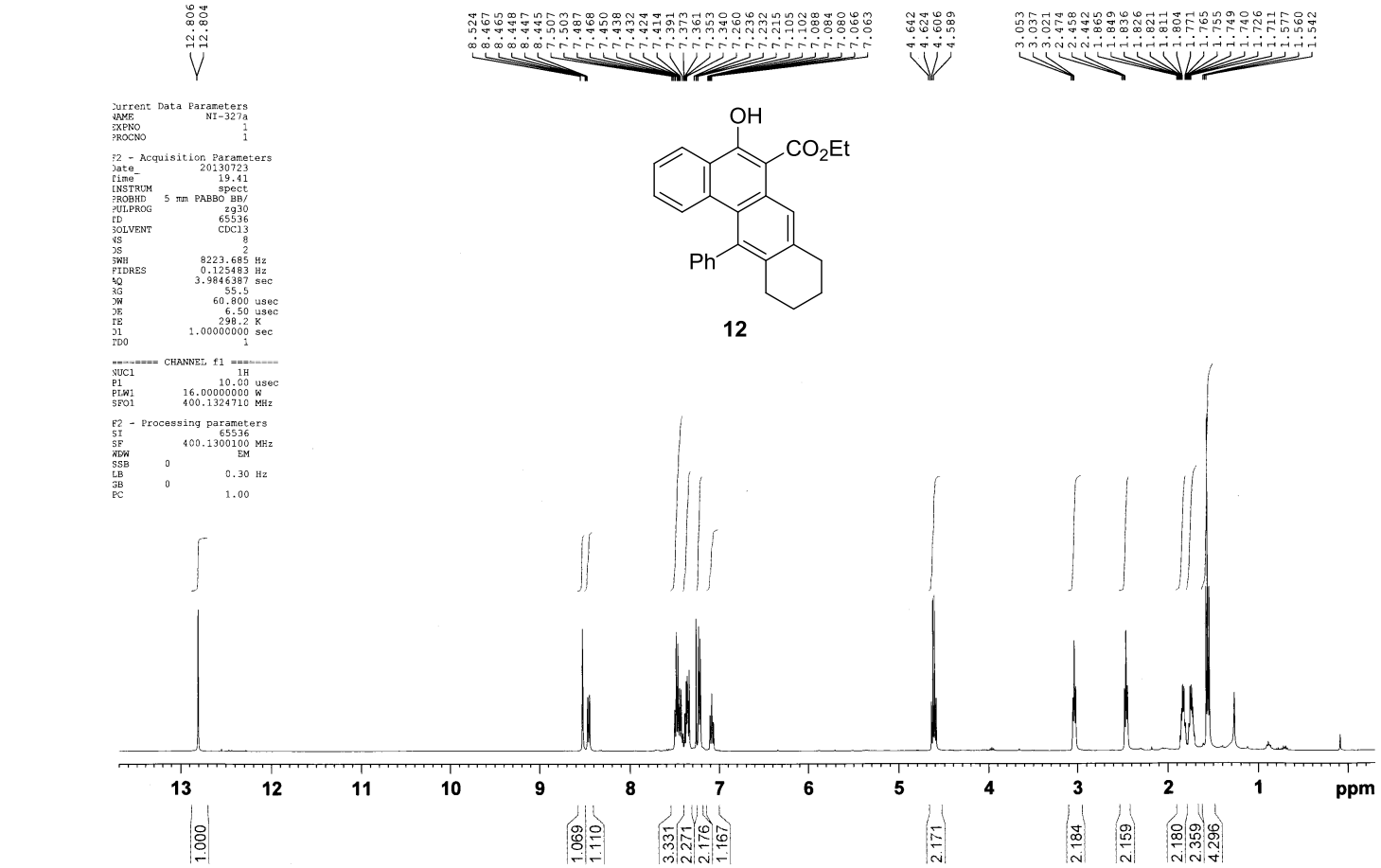

200.66
172.07
163.04
137.78
135.60
134.04
133.06
130.29
129.82
129.07
128.84
125.73
124.66
124.40
123.68
105.40
77.33
77.01
76.69
62.19
61.88
34.55
29.71
14.02
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Current Data Parameters
NAME HY-naph-OH
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151117
Time 13.01
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 83
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 297.4 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127746 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


12.381
8.543
8.540
8.524
8.522
8.520
7.553
7.550
7.536
7.533
7.517
7.512
7.507
7.491
7.487
7.474
7.470
7.386
7.366
7.363
7.259
7.214
7.208
7.204
7.192
7.188
7.174
7.168
7.164
7.159
7.152
7.086
7.082
7.074
7.059
7.056
7.047
7.043
7.031
7.026
7.021
7.011
7.007
6.982
6.975
6.970
6.967
6.962
6.958
3.967
3.949
3.931
3.914
0.683
0.665
0.648
OH
CO2Et
Current Data Parameters
NAME HY-NI-PhPh_Np
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150425
Time 15.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 4
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 72.93
DW 60.800 usec
DE 6.50 usec
TE 296.6 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300102 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
12 11 10 9 8 7 6 5 4 3 2 1 ppm
0.971
1.000
2.059
1.079
3.059
6.999
2.010
2.990


172.08
160.47
141.97
138.73
137.19
135.54
131.54
131.24
129.80
129.58
127.46
126.70
126.67
126.29
125.61
125.60
124.13
123.83
106.47
77.33
77.01
76.69
60.94
12.88
142 140 138 136 134 132 130 128 126 ppm
190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Current Data Parameters
NAME HY-NI-PhPh_Np
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20150425
Time 15.39
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 203
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 296.9 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127707 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


15.180
8.586
8.582
8.575
8.571
8.568
8.562
8.561
8.362
8.360
8.343
8.340
7.904
7.886
7.641
7.623
7.607
7.604
7.601
7.569
7.565
7.554
7.550
7.547
7.543
7.537
7.531
7.528
7.518
7.513
7.501
7.482
7.462
7.442
7.431
7.426
7.423
7.412
7.408
7.289
7.270
7.260
4.261
OH
O
O
Ph
11
Current Data Parameters
NAME HY-3637a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151221
Time 11.29
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 3
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 52.09
DW 60.800 usec
DE 6.50 usec
TE 294.8 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300095 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
15 14 13 12 11 10 9 8 7 6 5 4 3 2 ppm
0.966
1.000
1.010
1.929
1.174
3.140
4.185
0.956
1.870


199.27
189.82
164.44
140.26
137.24
134.24
134.17
133.79
132.10
130.86
130.60
129.76
129.10
128.44
127.17
127.12
125.78
125.52
124.75
124.54
123.30
109.70
77.33
77.01
76.69
30.43
140 135 130 125 120 115 110 ppm
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 ppm
Current Data Parameters
NAME HY-3637a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20151221
Time 11.34
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 52
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 295.1 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127736 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40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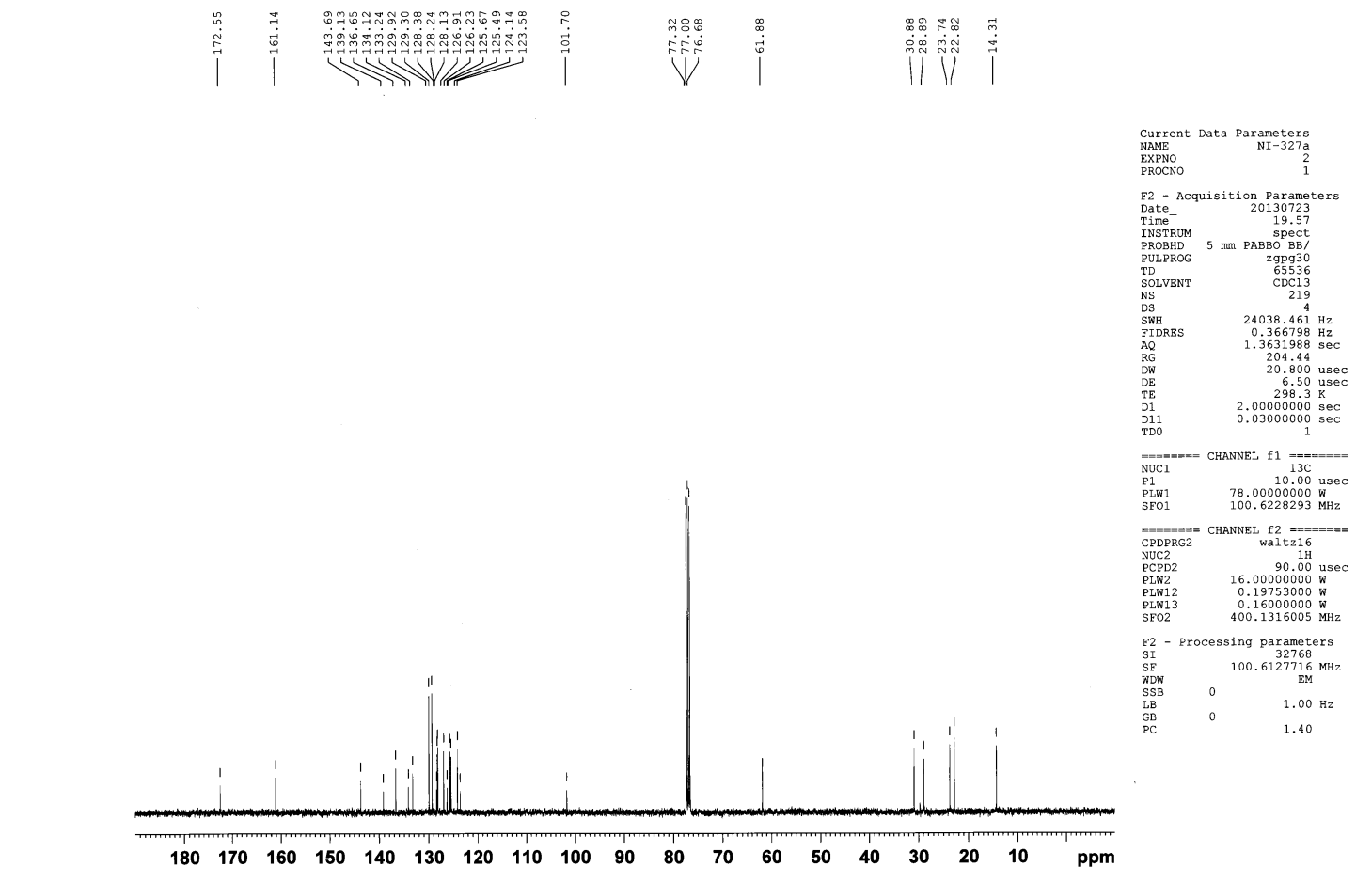
8.467
8.465
8.448
8.447
8.445
7.507
7.503
7.487
7.468
7.450
7.438
7.432
7.424
7.414
7.391
7.373
7.361
7.353
7.340
7.260
7.236
7.232
7.215
7.105
7.102
7.088
7.084
7.080
7.066
7.063
4.642
4.624
4.606
4.589
3.053
3.037
3.021
2.474
2.458
2.442
1.865
1.849
1.836
1.826
1.821
1.811
1.804
1.771
1.765
1.755
1.749
1.740
1.726
1.711
1.577
1.560
1.542
Current Data Parameters
NAME NI-327a
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130723
Time 19.41
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 55.5
DW 60.800 usec
DE 6.50 usec
TE 298.2 K
D1 1.00000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 1H
P1 10.00 usec
PLW1 16.00000000 W
SFO1 400.1324710 MHz
F2 - Processing parameters
SI 65536
SF 400.1300100 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
OH
CO2Et
Ph
12
13 12 11 10 9 8 7 6 5 4 3 2 1 ppm
1.000
1.069
1.110
3.331
2.271
2.176
1.167
2.171
2.184
2.159
2.180
2.359
4.296


172.55
161.14
143.69
139.13
136.65
134.12
133.24
129.92
129.30
128.38
128.24
128.13
126.91
126.23
125.67
125.49
124.14
123.58
101.70
77.32
77.00
76.68
61.88
30.88
28.89
23.74
22.82
14.31
180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm
Current Data Parameters
NAME NI-327a
EXPNO 2
PROCNO 1
F2 - Acquisition Parameters
Date_ 20130723
Time 19.57
INSTRUM spect
PROBHD 5 mm PABBO BB/
PULPROG zgpg30
TD 65536
SOLVENT CDCl3
NS 219
DS 4
SWH 24038.461 Hz
FIDRES 0.366798 Hz
AQ 1.3631988 sec
RG 204.44
DW 20.800 usec
DE 6.50 usec
TE 298.3 K
D1 2.00000000 sec
D11 0.03000000 sec
TD0 1
======== CHANNEL f1 ========
NUC1 13C
P1 10.00 usec
PLW1 78.00000000 W
SFO1 100.6228293 MHz
======== CHANNEL f2 ========
CPDPRG2 waltz16
NUC2 1H
PCPD2 90.00 usec
PLW2 16.00000000 W
PLW12 0.19753000 W
PLW13 0.16000000 W
SFO2 400.1316005 MHz
F2 - Processing parameters
SI 32768
SF 100.6127716 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

8. References

1. H. Yanai, T. Yoshino, M. Fujita, H. Fukaya, A. Kotani, F. Kusu, T. Taguchi, *Angew. Chem. Int. Ed.* **2013**, *52*, 1560–1563.
2. (a) F. J. Waller, A. G. M. Barrett, D. C. Braddock, D. Ramprasad, R. M. McKinnell, A. J. P. White, D. J. Williams, R. J. Ducray, *J. Org. Chem.* **1999**, *64*, 2910–2913; (b) H. Yanai, A. Takahashi, T. Taguchi, *Tetrahedron* **2007**, *63*, 12149–12159.
3. Yao, T.; Larock, R. C. *J. Org. Chem.* **2003**, *68*, 5936–5942.
4. Vasilevsky, S. F.; Mikhailovskaya, T. F.; Mamatyuk, V. I.; Salnikov, G. E.; Bogdanchikov, G. A.; Manoharan, M.; Alabugin, I. V. *J. Org. Chem.*, **2009**, *74*, 8106–8117.