Supporting Information

Use of 4-Cyanocoumarins as Dienophiles in a Facile Synthesis of Highly Substituted Dibenzopyranones

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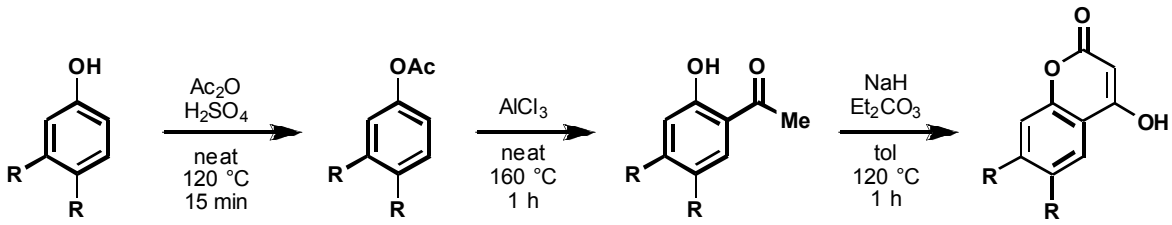
General Experimental: All reactions were carried out in flame-dried flasks under an argon atmosphere unless otherwise noted. The following solvents were dried and distilled from the indicated drying agent under an argon atmosphere: tetrahydrofuran (THF) and diethyl ether (ether) from sodium benzophenone ketyl radical; dichloromethane, benzene, triethylamine, and hexamethylphosphoramide (HMPA) from calcium hydride; diisopropylamine from sodium hydroxide; methanol from magnesium methoxide. Danishefsky's diene **10d** was purchased from Aldrich and used without purification. The TBS analogue of Danishefsky's diene **10e**[1] and 3-cyanocoumarin[2] were prepared as described in the literature. All other reagents were reagent grade and used without purification unless otherwise stated. ^{1}H and ^{13}C NMR spectra were recorded on Bruker spectrometers at 500 MHz for proton and at 125 MHz for carbon. ^{1}H and ^{13}C NMR data are reported in parts per million (δ) downfield from tetramethylsilane. The following abbreviations are used: s (singlet), d (doublet), t (triplet), q (quartet), m (multiplet), b (broad). Infrared spectra were recorded on a Thermo Nicolet Avatar 370 infrared spectrophotometer. Thin-layer chromatography (TLC) was performed using Merck silica gel 60 F254 0.2 mm aluminum-backed plates. Visualization was accomplished using ultraviolet light or one of the following stains: anisaldehyde or potassium permanganate. Flash column chromatography was carried out using ICN Biomedicals silica gel 60 (230-400 mesh). Microwave reactions were performed in a CEM Discover LabMate microwave.

Synthesis of 4-Cyanocoumarins 12a, 12b and 12c.

The 4-cyanocoumarin **5**/**12a** was synthesized from the commercially available 4-hydroxycouma-

rin **3** in two steps, namely bromination followed by cyanation. The 4-cyanocoumarins **12b** and **12c** were also synthesized from the corresponding 4-hydroxycoumarins. These 4-hydroxy-coumarins are commercially available but are expensive. Therefore, they were synthesized in three steps from the corresponding phenols as described in the literature[3] in 63-70% yield.



3b or 3c

6-Chloro-4-hydroxy-7-methylcoumarin (3b). White powder, mp >250 °C. R_f = 0.26 (ethyl acetate) ^{1}H NMR (500 MHz, DMSO-d_6) δ: 7.67 (1H, s), 7.10 (1H, s), 4.68 (1H, s), 4.10-3.83 (1H, bs), 2.31 (3H, s). ^{13}C NMR (125 MHz, DMSO-d_6) δ: 172.7, 164.5, 153.1, 137.9, 127.0, 124.1, 122.3, 118.5, 86.3, 20.0.

4-Hydroxy-6,7-dimethylcoumarin (3c). White powder, mp 236-238 °C. R_f = 0.47 (ethyl acetate).^{1}H NMR (500 MHz, DMSO-d_6) δ: 12.44 (1H, bs), 7.49 (1H, s), 7.09 (1H, s), 5.52 (1H, s), 2.25 (3H, s), 2.13 (3H, s). ^{13}C NMR (125 MHz, DMSO-d_6) δ: 166.2, 162.6, 152.3, 142.7, 132.6, 123.3, 117.0, 113.6, 90.5, 20.0, 19.1.

Preparation of the 4-Cyanocoumarins 12a, 12b, and 12c from the Corresponding 4-Hydroxycoumarins.

To a round bottom flask equipped with a magnetic stir bar and a reflux condenser was added the 4-hydroxycoumarin **3** and toluene (0.5 M). The reaction mixture was heated at 120 °C for 10 min and then Bu_4NBr (1.5 equiv) was added in 2 portions over 5 min. The reaction mixture was stirred vigorously for 15 min until the starting material completely dissolved. P_2O_5 (2.0 equiv) was then added in 3 portions over 15 min and the reaction was heated for 3 h. The reaction mixture was cooled to 23 °C and quenched with a saturated aqueous $NaHCO_3$ solution. The

mixture was poured into a separatory funnel containing dichloromethane. The remaining solid was also transferred with methanol (3x) to the separatory funnel. The aqueous layer was extracted with dichloromethane (3x) and the combined organic layers were then washed with water (2x), brine (1x) and dried over $MgSO_4$. The solvent was removed *in vacuo* to afford a white/orange solid. To a round bottom flask equipped with a magnetic stir bar was added the crude bromide **4**, dimethylformamide (2 M) and CuCN (1.1 equiv). The reaction mixture was heated at 130 °C for 2 h and then cooled to 23 °C. The dimethylformamide was removed *in vacuo* and then the reaction was diluted with dichloromethane and an aqueous NH_4OH solution. The aqueous layer was extracted with dichloromethane (3x) and the combined organic layers were then washed with aqueous NH_4OH (2x), water (2x), brine (1x) and dried over $MgSO_4$. The solvent was removed *in vacuo* to afford the 4-cyanocoumarin, **12**.

4-Bromocoumarin (4). Yellow powder, mp 87-89 °C. R_f = 0.53 (1:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.82 (1H, dd, *J* = 7.9, 1.5 Hz), 7.59 (1H, ddd, *J* = 7.9, 7.9, 1.5 Hz), 7.35 (1H, ddd, *J* = 7.9, 7.9, 1.2 Hz), 7.32 (1H, d, *J* = 7.9 Hz), 6.85 (1H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.7, 152.4, 141.8, 133.2, 128.0, 124.9, 119.5, 118.9, 117.0.

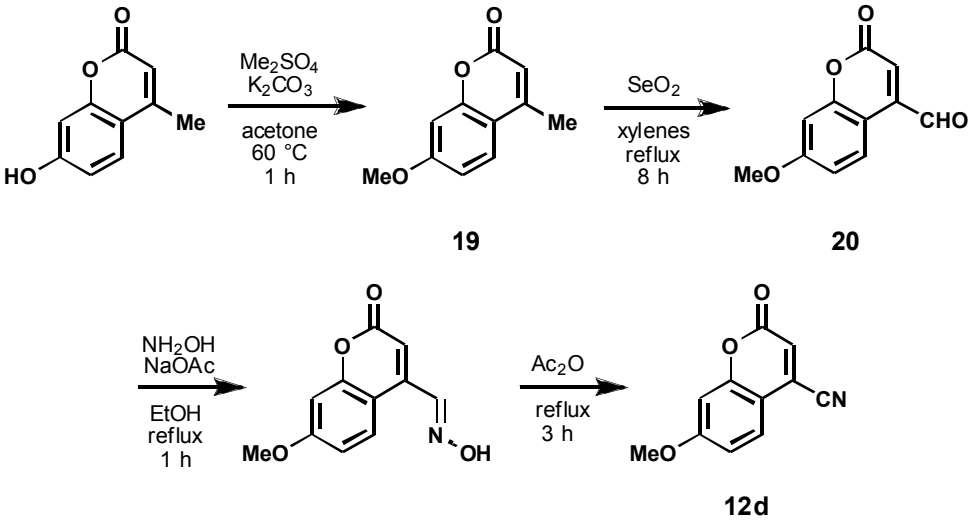
4-Bromo-6-chloro-7-methylcoumarin (4b). Green powder, mp 195-196 °C. R_f = 0.70 (ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.78 (1H, s), 7.21 (1H, s), 6.82 (1H, s), 2.48 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.3, 150.6, 142.4, 139.9, 130.9, 127.5, 119.4, 118.9, 118.0, 20.5. IR (KBr): 1732 cm^{-1}.

4-Bromo-6,7-dimethylcoumarin (4c). Tan powder, mp 140-142 °C. R_f = 0.63 (ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.52 (1H, s), 7.09 (1H, s), 6.74 (1H, s), 2.36 (3H, s), 2.33 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 159.2, 150.7, 143.6, 141.4, 133.9, 127.8, 118.2, 117.4, 116.6, 20.2, 19.3.

4-Cyanocoumarin (5/12a). Small tan crystals, mp 182-184 °C. R_f = 0.44 (2:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.81 (1H, dd, *J* = 7.9, 1.5 Hz), 7.68 (1H, ddd, *J* = 8.2, 7.2, 1.5 Hz), 7.46-7.37 (2H, m), 6.86 (1H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.6, 153.7, 133.9, 127.2, 126.1, 125.5, 123.7, 117.5, 115.3, 112.9. IR (KBr): 1728, 1604 cm^{-1}.

6-Chloro-4-cyano-7-methylcoumarin (12b). Tan powder, mp 154-156 °C. R_f = 0.18 (6:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.76 (1H, s), 7.28 (1H, s), 6.83 (1H, s), 2.51 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 157.4, 151.8, 143.5, 131.6, 126.0, 125.5, 123.5, 119.4, 114.2, 112.6, 20.9. IR (KBr): 1730 cm^{-1}.

4-Cyano-6,7-dimethylcoumarin (12c). Yellow powder, mp 170-172 °C. R_f = 0.34 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.51 (1H, s), 7.17 (1H, s), 6.76 (1H, s), 2.39 (3H, s), 2.36 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 158.3, 152.0, 144.7, 134.7, 126.9, 125.8, 122.2, 117.9, 113.2, 113.0, 20.5, 19.2. IR (KBr): 1726, 1622 cm^{-1}.



Preparation of 4-Cyano-7-methoxycoumarin 12d

4-Cyano-7-methoxycoumarin **12d** was synthesized from commercially available 4-methyl-7-hydroxycoumarin in four steps according to the literature[4] in 43% overall yield.

4-Methyl-7-methoxycoumarin (19). White needles, mp 155-156 °C. R_f = 0.53 (1:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.48 (1H, d, *J* = 8.9 Hz), 6.84 (1H, dd, *J* = 8.9, 1.8 Hz), 6.80 (1H, d, *J* = 2.1 Hz), 6.12 (1H, d, *J* = 0.9 Hz), 3.86 (3H, s), 2.39 (3H, d, *J* = 1.2 Hz). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 162.6, 161.2, 155.3, 152.5, 125.5, 113.5, 112.2, 111.9, 100.8, 55.7, 18.6.

4-Formyl-7-methoxycoumarin (20). Yellow powder, mp 185 °C. R_f = 0.30 (1:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, DMSO-d_6) δ: 10.07 (1H, s), 8.35 (1H, d, *J* = 8.9 Hz), 7.04 (1H, d, *J* = 2.4 Hz), 6.99 (1H, dd, *J* = 9.2, 2.8 Hz), 6.95 (1H, s), 3.84 (3H, s). ^{13}C NMR (125 MHz, DMSO-d_6) δ: 194.1, 162.9, 160.8, 156.3, 143.9, 127.3, 121.7, 113.2, 108.5, 101.4, 56.3. IR (KBr): 1730, 1617 cm^{-1}.

4-Cyano-7-methoxycoumarin (12d). Light green powder, mp 166-167 °C. R_f = 0.25 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.68 (1H, d, *J* = 8.9 Hz), 6.96 (1H, dd, *J* = 8.9, 2.4 Hz), 6.85 (1H, d, *J* = 2.4 Hz), 6.65 (1H, s), 3.91 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 164.4, 158.3, 155.6, 127.0, 126.9, 119.5, 113.7, 113.2, 109.0, 101.4, 56.1.

Preparation of Dienes 10a, 10b, 10c, and 10f.

To a round bottom flask equipped with a magnetic stir bar was added the corresponding commercially available aldehyde[5] or ketone, THF (2 M) and Et_3N (1.5 equiv). The reaction mixture was cooled to 0 °C and then *tert*-butyldimethylsilyl trifluoromethanesulfonate (1.05 equiv) was added over 10 min. Once the reaction was deemed complete by TLC (6-12 h), the reaction mixture was quenched with a saturated $NaHCO_3$ aqueous solution. The aqueous layer was extracted with pentane (3x) and the combined organic layers were then washed with water and brine and dried over $MgSO_4$. The solvent was removed *in vacuo* and the crude product was purified by distillation (100 °C/10 mm Hg) to afford the product as a clear colorless liquid.

1-(1,1-Dimethylethyl)dimethylsilyloxy-1,3-butadiene, 7/10a. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.56 (1H, d, *J* = 11.6 Hz), 6.22 (1H, ddd, *J* = 17.1, 10.1, 10.1 Hz), 5.73 (1H, dd, *J* = 11.6, 11.6 Hz), 4.98 (1H, d, *J* = 17.1, 0.9 Hz), 4.81 (1H, dd, *J* = 10.1, 0.9 Hz), 0.92 (9H, s), 0.16 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 145.3, 133.3, 114.1, 111.8, 25.6, 18.2, -5.3. R_f = 0.75 (3:1, hexanes:ethyl acetate).

(*Z*)-1-(1,1-Dimethylethyl)dimethylsilyloxy-2-(2-propenyl)-1,3-butadiene, 10b. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.45 (1H, s), 6.20 (1H, dd, *J* = 17.1, 10.7 Hz), 5.80 (1H, ddt, *J* = 17.6, 10.7, 6.4 Hz), 5.05 (1H, d, *J* = 17.1 Hz), 5.02 (1H, d, *J* = 17.1 Hz), 4.95 (1H, d, *J* = 10.1 Hz), 4.82 (1H, d, *J* = 11.0 Hz), 3.03 (2H, d, *J* = 6.4 Hz), 0.93 (9H, s), 0.15 (6H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 142.3, 136.2, 135.3, 120.2, 114.5, 108.5, 28.0, 25.5, 18.1, -5.4. R_f = 0.69 (3:1, hexanes:ethyl acetate).

(*E*)-1-(1,1-Dimethylethyl)dimethylsilyloxy-2-methyl-1,3-pentadiene, 10c. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 6.33 (1H, s), 5.97 (1H, dd, *J* = 15.2, 0.9 Hz), 5.46 (1H, dq, *J* = 15.3, 6.4 Hz), 1.75 (3H, dd, *J* = 6.7, 0.9 Hz), 1.70 (3H, s), 0.93 (9H, s), 0.14 (6H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 139.5, 131.0, 120.0, 117.9, 25.6, 18.3, 18.2, 9.4, -5.3. R_f = 0.68 (3:1, hexanes:ethyl acetate).

2-(1,1-Dimethylethyl)dimethylsilyloxy-5-methyl-1,3-pentadiene, 10f. ^{1}H NMR (500 MHz, $CDCl_3$) δ: 5.56 (1H, s), 4.30 (1H, s), 4.16 (1H, s), 1.90 (3H, s), 1.77 (3H, s), 0.93 (9H, s), 0.17 (6H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 155.6, 136.7, 123.0, 94.8, 27.0, 25.7, 19.8, 18.3, -4.4. R_f = 0.63 (3:1, hexanes:ethyl acetate).

General Procedure for the Diels-Alder Reaction.

To a round bottom flask equipped with a reflux condenser and a magnetic stir bar, or to a microwave reaction vial equipped with a magnetic stir bar, was added the 4-cyanocoumarin,

degassed toluene (1 M) and the diene (2 equiv). The reaction mixture was heated at the indicated temperature for the indicated time and then allowed to cool to 23 °C. The solvent was removed *in vacuo* and the crude product was purified by column chromatography (1:1, pentane:dichloro-methane) or recrystallization from hot methanol.

(±) (6a*S*,10*R*,10a*R*)-10-(1,1-Dimethylethyl)dimethylsilyloxy-6-oxo-6a,7,10,10a-tetrahydro-6*H*-benzo[*c*]chromene-10a-carbonitrile, 8n/11a. White needles, mp 155-157 °C. R_f = 0.37 (6:1, hexanes:ethyl acetate).^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.57 (1H, d, *J* = 7.6 Hz), 7.40 (1H, ddd, *J* = 8.2, 8.2, 1.2 Hz), 7.24 (1H, dd, *J* = 7.6, 7.6 Hz), 7.06 (1H, d, *J* = 8.2 Hz), 6.03 (1H, ddd, *J* = 10.0, 4.3, 2.0 Hz), 5.87 (1H, bdd, *J* = 10.0, 4.9 Hz), 4.23 (1H, d, *J* = 4.9 Hz), 3.25 (1H, dd, *J* = 19.0, 4.5 Hz), 3.24 (1H, d, *J* = 7.0 Hz), 2.67 (1H, bd, *J* = 19.0 Hz), 0.67 (9H, s), -0.11 (3H, s), -0.52 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 166.0, 151.8, 130.5, 128.1, 126.7, 125.3, 124.7, 120.2, 118.8, 117.3, 68.9, 44.3, 36.6, 25.2, 22.4, 17.6, -5.1, -6.1. IR (KBr): 1773 cm^{-1}.

(±) (6a*S*,10*R*,10a*R*)-10-(1,1-Dimethylethyl)dimethylsilyloxy-6-oxo-6a,7,10,10a-tetrahydro-9-(2-propenyl)-6*H*-benzo[*c*]chromene-10a-carbonitrile, endo 11b. White needles, mp 143 °C. R_f = 0.46 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.60 (1H, dd, *J* = 7.6, 1.5 Hz), 7.40 (1H, ddd, *J* = 8.2, 7.6, 1.5 Hz), 7.25 (1H, ddd, *J* = 8.5, 8.2, 1.2 Hz), 7.07 (1H, dd, *J* = 8.2, 1.2 Hz), 5.79 (1H, dddd, *J* = 17.1, 10.4, 7.6, 5.5 Hz), 5.73-5.70 (1H, m), 5.19-5.12 (2H, m), 4.16 (1H, s), 3.31 (1H, d, *J* = 19.2 Hz), 3.20 (1H, d, *J* = 7.6 Hz), 2.92 (1H, d, *J* = 15.6 Hz), 2.79 (1H, dd, *J* = 15.6, 7.6 Hz), 2.66 (1H, dd, *J* = 19.2, 7.6 Hz), 0.74 (9H, s), -0.03 (3H, s), -0.82 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 166.2, 151.8, 134.8, 134.5, 130.7, 127.2, 124.9, 123.9, 120.4, 118.9, 117.8, 117.7, 72.0, 45.3, 38.9, 36.3, 25.4, 22.4, 18.0, -4.7, -5.7. IR (KBr): 1779, 1636 cm^{-1}.

(±) (6a*S*,7*S*,10*R*,10a*R*)-7,9-Dimethyl-10-(1,1-dimethylethyl)dimethylsilyloxy-6-oxo-6a,7,10,

10a-tetrahydro-6*H*-benzo[*c*]chromene-10a-carbonitrile, endo 11c. White powder. R_f = 0.47 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.57 (1H, dd, *J* = 7.9, 1.5 Hz), 7.38 (1H, ddd, *J* = 7.9, 7.9, 1.5 Hz), 7.21 (1H, dd, *J* = 7.6, 7.6 Hz), 7.02 (1H, d, *J* = 7.9 Hz), 5.58 (1H, s), 4.11 (1H, s), 3.09-3.03 (1H, m), 2.98 (1H, d, *J* = 4.9 Hz), 1.80 (3H, s), 1.52 (3H, d, *J* = 7.6 Hz), 0.74 (9H, s), -0.02 (3H, s), -0.95 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 164.0, 152.2, 130.8, 130.5, 129.3, 127.1, 124.4, 121.0, 118.8, 117.4, 47.5, 41.2, 30.2, 25.8, 25.4, 22.3, 17.8, 17.6, -4.5, -5.9.

(±) (6a*S*,10*R*,10a*R*)-8-(1,1-Dimethylethyl)dimethylsilyloxy-10-methoxy-6-oxo-6a,7,10,10a-tetrahydro-6*H*-benzo[*c*]chromene-10a-carbonitrile, endo 11e. White powder, mp 143-147 °C. R_f = 0.41 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.62 (1H, dd, *J* = 7.6, 1.5 Hz), 7.41 (1H, dd, *J* = 7.9, 7.9 Hz), 7.25 (1H, dd, *J* = 7.6, 7.6 Hz), 7.09 (1H, d, *J* = 8.2 Hz), 5.17 (1H, d, *J* = 6.1 Hz), 4.00 (1H, d, *J* = 6.1 Hz), 3.28 (1H, d, *J* = 7.3 Hz), 3.18 (1H, d, *J* = 18.6 Hz), 2.94 (3H, s), 2.64 (1H, dd, *J* = 18.6, 7.3 Hz), 0.97 (9H, s), 0.25 (3H, s), 0.23 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 165.9, 153.5, 151.5, 130.6, 126.8, 124.6, 119.7, 118.6, 117.2, 100.5, 78.1, 57.0, 43.5, 38.0, 26.8, 25.5, 17.9, -4.4, -4.7. IR (KBr): 1776, 1665 cm^{-1}.

(±) (6a*S*,10*R*,10a*R*)-2-Chloro-10-(1,1-dimethylethyl)dimethylsilyloxy-3-methyl-6-oxo-6a,7, 10,10a-tetrahydro-6*H*-benzo[*c*]chromene-10a-carbonitrile, endo 13b. Yellow powder. R_f = 0.50 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.51 (1H, s), 6.91 (1H, s), 6.03-5.98 (1H, m), 5.88-5.82 (1H, m), 4.19 (1H, d, *J* = 4.9 Hz), 3.24-3.16 (2H, m), 2.64 (1H, d, *J* = 8.9 Hz), 2.38 (3H, s), 0.67 (9H, s), -0.10 (3H, s), -0.47 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 165.6, 150.0, 138.9, 129.8, 128.1, 126.7, 125.1, 119.3, 119.1, 118.3, 68.8, 43.8, 36.4, 25.1, 22.3, 19.9, 17.6, -5.2, -6.1.

(±) (6a*S*,10*R*,10a*R*)-3-Methoxy-10-(1,1-dimethylethyl)dimethylsilyloxy-6-oxo-6a,7,10,10a-tetrahydro-6*H*-benzo[*c*]chromene-10a-carbonitrile, endo 13d. Yellow powder. R_f = 0.33 (6:1,

hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.42 (1H, dd, *J* = 8.5, 1.2 Hz), 6.76 (1H, d, *J* = 8.5 Hz), 6.57 (1H, s), 6.01-5.95 (1H, m), 5.87-5.81 (1H, m), 4.16 (1H, d, *J* = 5.2 Hz), 3.79 (3H, s), 3.24-3.16 (2H, m), 2.63 (1H, dd, *J* = 19.5, 7.3 Hz), 0.67 (9H, s), -0.12 (3H, s), -0.49 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 166.0, 161.3, 152.6, 128.0, 127.4, 125.3, 118.9, 112.2, 110.4, 102.8, 68.9, 55.6, 43.6, 36.7, 25.1, 22.4, 17.5, -5.2, -6.0.

(±) (6a*S*,7*S*,10a*S*)-7-(1,1-Dimethylethyl)dimethylsilyloxy-6-oxo-6a,7,10,10a-tetrahydro-6*H*-benzo[*c*]chromene-6a-carbonitrile, endo 17. Yellow powder, mp 125-129 °C. R_f = 0.35 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.26 (1H, dd, *J* = 7.9, 7.9 Hz), 7.17 (1H, dd, *J* = 7.9, 7.9 Hz), 7.14 (1H, dd, *J* = 7.9, 0.9 Hz), 6.99 (1H, dd, *J* = 7.9, 0.9 Hz), 5.98 (1H, bd, *J* = 10.4 Hz), 5.67 (1H, bd, *J* = 10.4 Hz), 4.59 (1H, bs), 3.78 (1H, d, *J* = 5.2 Hz), 2.98 (1H, dd, *J* = 18.9, 5.8 Hz), 2.75 (1H, bd, *J* = 18.9 Hz), 0.63 (9H, s), 0.02 (3H, s), -0.13 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 162.8, 152.0, 128.8, 126.1, 126.0, 125.0, 124.8, 120.9, 117.0, 116.8, 67.7, 49.3, 33.0, 25.4, 22.3, 17.7, -5.0, -5.2. IR (KBr): 1764 cm^{-1}.

General Procedure for Elimination/Aromatization.

To a round bottom flask equipped with a magnetic stir bar was added the Diels-Alder adduct and THF (0.2 M). The reaction mixture was cooled to 0 °C and KOtBu (2.5 equiv) was added. After 15 min, the reaction was quenched with an aqueous NH_4Cl solution and diluted with dichloro-methane. The aqueous layer was extracted with dichloromethane (3x) and the combined organic layers were then washed with water (1x), brine (1x) and dried over $MgSO_4$. The solvent was removed *in vacuo* and the crude product was purified by column chromatography (1:2, pentane:dichloromethane; dichloromethane → acetone for **14c**) to afford the pure product as a white powder.

6*H*-Benzo[*c*]chromen-6-one, 14a. mp 89 °C. R_f = 0.32 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.39 (1H, d, *J* = 7.9 Hz), 8.11 (1H, d, *J* = 8.2 Hz), 8.05 (1H, d, *J* = 8.2 Hz),

7.82 (1H, ddd, *J* = 8.2, 8.2, 0.9 Hz), 7.58 (1H, dd, *J* = 7.6, 7.6 Hz), 7.48 (1H, ddd, *J* = 8.2, 8.2, 1.2 Hz), 7.36 (1H, d, *J* = 8.5 Hz), 7.33 (1H, ddd, *J* = 7.9, 7.9, 0.9 Hz). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 161.2, 151.2, 134.8, 134.7, 130.5, 130.4, 128.8, 124.5, 122.7, 121.7, 121.2, 118.0, 117.7. IR (KBr): 1736, 1607 cm^{-1}.

9-(2-Propenyl)-6*H*-benzo[*c*]chromen-6-one, 14b. mp 98 °C. R_f = 0.39 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.31 (1H, d, *J* = 8.2 Hz), 8.05 (1H, d, *J* = 8.2 Hz), 7.91 (1H, s), 7.46 (1H, ddd, *J* = 8.2, 7.9, 1.2 Hz), 7.41 (1H, d, *J* = 8.2 Hz), 7.35 (1H, d, *J* = 8.2 Hz), 7.32 (1H, dd, *J* = 7.6, 7.6 Hz), 6.02 (1H, dddd, *J* = 16.8, 10.3, 6.7, 6.7 Hz), 5.22-5.15 (2H, m), 3.58 (2H, d, *J* = 6.7 Hz). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 161.1, 151.4, 147.8, 135.7, 134.8, 130.7, 130.3, 130.0, 129.6, 124.4, 122.7, 119.3, 118.0, 117.7, 117.4, 40.5. IR (KBr): 1731, 1614 cm^{-1}.

8-Hydroxy-6*H*-benzo[*c*]chromen-6-one, 14c. mp 232 °C. R_f = 0.43 (1:2, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, DMSO-d_6) δ: 10.46 (1H, bs), 8.31 (1H, d, *J* = 8.9 Hz), 8.25 (1H, d, *J* = 7.9 Hz), 7.61 (1H, d, *J* = 2.4 Hz), 7.49 (1H, ddd, *J* = 8.3, 8.3, 1.2 Hz), 7.42 (3H, m). ^{13}C NMR (125 MHz, DMSO-d_6) δ: 160.2, 158.3, 149.6, 129.1, 126.0, 124.7, 124.6, 124.0, 122.7, 121.9, 118.1, 117.0, 113.8. IR (KBr): 3248, 1715 cm^{-1}.

2,3-Dimethyl-6*H*-benzo[*c*]chromen-6-one, 14d. mp 170-172 °C. R_f = 0.44 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.32 (1H, d, *J* = 8.2 Hz), 7.98 (1H, d, *J* = 8.2 Hz), 7.74 (1H, dd, *J* = 8.2, 8.2 Hz), 7.66 (1H, s), 7.48 (1H, dd, *J* = 8.2, 8.2 Hz), 7.03 (1H, s), 2.30 (3H, s), 2.29 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 161.4, 149.3, 139.9, 134.9, 134.5, 133.0, 130.3, 128.0, 122.9, 121.2, 120.8, 118.0, 115.2, 19.9, 19.4. IR (KBr): 1722, 1605 cm^{-1}.

3-Methoxy-6*H*-benzo[*c*]chromen-6-one, 14e. mp 137-138 °C. R_f = 0.26 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.32 (1H, d, *J* = 7.9 Hz), 7.97 (1H, d, *J* = 7.9 Hz), 7.91

(1H, d, *J* = 8.9 Hz), 7.76 (1H, ddd, *J* = 7.9, 7.9, 1.2 Hz), 7.48 (1H, dd, *J* = 7.6, 7.6 Hz), 6.89 (1H, dd, *J* = 8.8, 2.4 Hz), 6.83 (1H, d, *J* = 2.4 Hz), 3.87 (3H, s). ^{13}C NMR (125 MHz, DMSO-d_6) δ: 161.6, 160.8, 152.4, 135.7, 135.1, 130.0, 128.4, 125.1, 122.3, 119.6, 112.7, 111.0, 101.8, 56.1. IR (KBr): 1732, 1620 cm^{-1}.

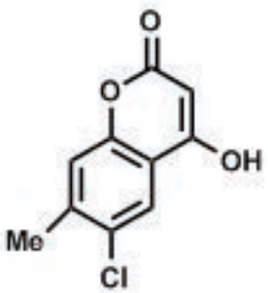
2-Chloro-3-methyl-6*H*-benzo[*c*]chromen-6-one, 14f. mp 182-183 °C. R_f = 0.44 (3:1, hexanes: ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.33 (1H, d, *J* = 7.6 Hz), 7.94 (1H, d, *J* = 7.6 Hz), 7.89 (1H, s), 7.79 (1H, dd, *J* = 7.9, 7.9 Hz), 7.56 (1H, dd, *J* = 7.9, 7.9 Hz), 7.16 (1H, s), 2.42 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.7, 149.4, 138.9, 134.9, 133.7, 130.6, 130.4, 129.0, 122.7, 121.4, 120.8, 119.5, 117.0, 20.2. IR (KBr): 1743, 1604 cm^{-1}.

7,9-Dimethyl-6*H*-benzo[*c*]chromen-6-one, 14g. mp 143-145 °C. R_f = 0.39 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 8.00 (1H, d, *J* = 7.9 Hz), 7.76 (1H, s), 7.43 (1H, dd, *J* = 7.3, 7.3 Hz), 7.31-7.24 (2H, m), 7.18 (1H, s), 2.82 (3H, s), 2.48 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 160.5, 151.4, 144.7, 144.2, 136.0, 133.6, 130.0, 124.0, 122.9, 120.0, 118.3, 117.23, 117.21, 23.7, 22.0. IR (KBr): 1727, 1610 cm^{-1}.

(±)-1,1-Dimethylethyl (1*S*,2*S*,6S)-1-cyano-2-(1,1-dimethylethyl)dimethylsilyloxy-6-(2-hydroxyphenyl)-cyclohex-3-enoate, 18. mp 178 °C. R_f = 0.32 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.18 (1H, dd, *J* = 7.6, 1.5 Hz), 6.98 (1H, ddd, *J* = 7.6, 7.6, 1.5 Hz), 6.81 (1H, ddd, *J* = 7.6, 7.6, 0.9 Hz), 6.67 (1H, dd, *J* = 7.9, 0.9 Hz), 6.65 (1H, bs), 5.95 (1H, ddd, *J* = 10.0, 4.5, 2.5 Hz), 5.58 (1H, bdd, *J* = 10.0, 2.7 Hz), 4.94-4.91 (1H, m), 4.13 (1H, dd, *J* = 11.6, 5.5 Hz) 2.86 (1H, dddd, *J* = 18.0, 11.5, 3.0, 2.8 Hz), 2.22 (1H, bd, *J* = 18.0 Hz), 1.48 (9H, s), 0.94 (9H, s), 0.24 (3H, s), 0.19 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 163.8, 154.2, 128.8, 128.7, 127.0, 126.7, 125.0, 120.3, 119.9, 116.1, 83.8, 74.2, 55.6, 36.6, 28.7, 27.9, 25.7, 18.1, -4.7. IR (KBr): 3380, 2256, 1737 cm^{-1}.

(±) (6a*S*,10*R*,10a*R*)-10-(1,1-Dimethylethyl)dimethylsilyloxy-6-oxo-6a,7,10,10a-tetrahydro-6a-methyl-6*H*-benzo[*c*]chromene-10a-carbonitrile, 16. To a round bottom flask equipped with a magnetic stir bar was added diisopropylamine (2.8 equiv) and THF (0.2 M). The reaction mixture was cooled to 0 °C and *n*-butyllithium (2.5 equiv) was added. After 15 min the reaction mixture was cooled to -78 °C and stirred for 20 min. Freshly distilled HMPA (2 equiv) was added followed by dropwise addition of the Diels-Alder adduct **11a** in THF (1 M). After 30 min, iodomethane (10 equiv) was added dropwise and the reaction was warmed to -40 °C. After 2 h the reaction was quenched with an aqueous NH_4Cl solution and diluted with dichloromethane. The aqueous layer was extracted with dichloromethane (3x) and the combined organic layers were then washed with water (3x), brine (1x) and dried over $MgSO_4$. The solvent was removed *in vacuo* and the crude product was purified by recrystallization from hot methanol to afford the pure product as a white powder. mp 150-152 °C. R_f = 0.56 (3:1, hexanes:ethyl acetate). ^{1}H NMR (500 MHz, $CDCl_3$) δ: 7.52 (1H, dd, *J* = 7.9, 1.5 Hz), 7.39 (1H, ddd, *J* = 7.9, 7.9, 1.5 Hz), 7.24 (1H, ddd, *J* = 7.9, 7.9, 1.5 Hz), 7.02 (1H, dd, *J* = 7.9, 1.2 Hz), 5.98 (1H, ddd, *J* = 10.4, 4.6, 2.8 Hz), 5.82 (1H, ddd, *J* = 10.4, 4.9, 2.8 Hz), 4.22 (1H, d, *J* = 4.9 Hz), 3.30 (1H, ddd, *J* = 19.2, 4.6, 2.8 Hz), 2.26 (1H, ddd, *J* = 19.2, 4.9, 2.8 Hz), 1.38 (3H, s), 0.67 (9H, s), -0.13 (3H, s), -0.58 (3H, s). ^{13}C NMR (125 MHz, $CDCl_3$) δ: 168.9, 151.5, 130.3, 128.7, 127.1, 124.9, 124.8, 119.0, 117.7, 116.7, 70.1, 50.2, 38.7, 31.1, 25.2, 23.2, 17.6, -5.2, -6.1. IR (KBr): 1776 cm^{-1}. An x-ray crystal structure confirmed the stereochemistry of **16**.[6]

default proton parameters



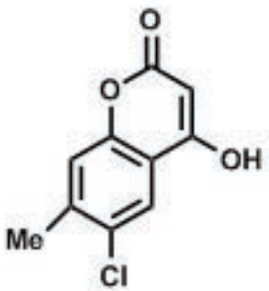
3b

Current Data Parameters
NAME DAA-XII-279-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20081002
Time 15.29
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

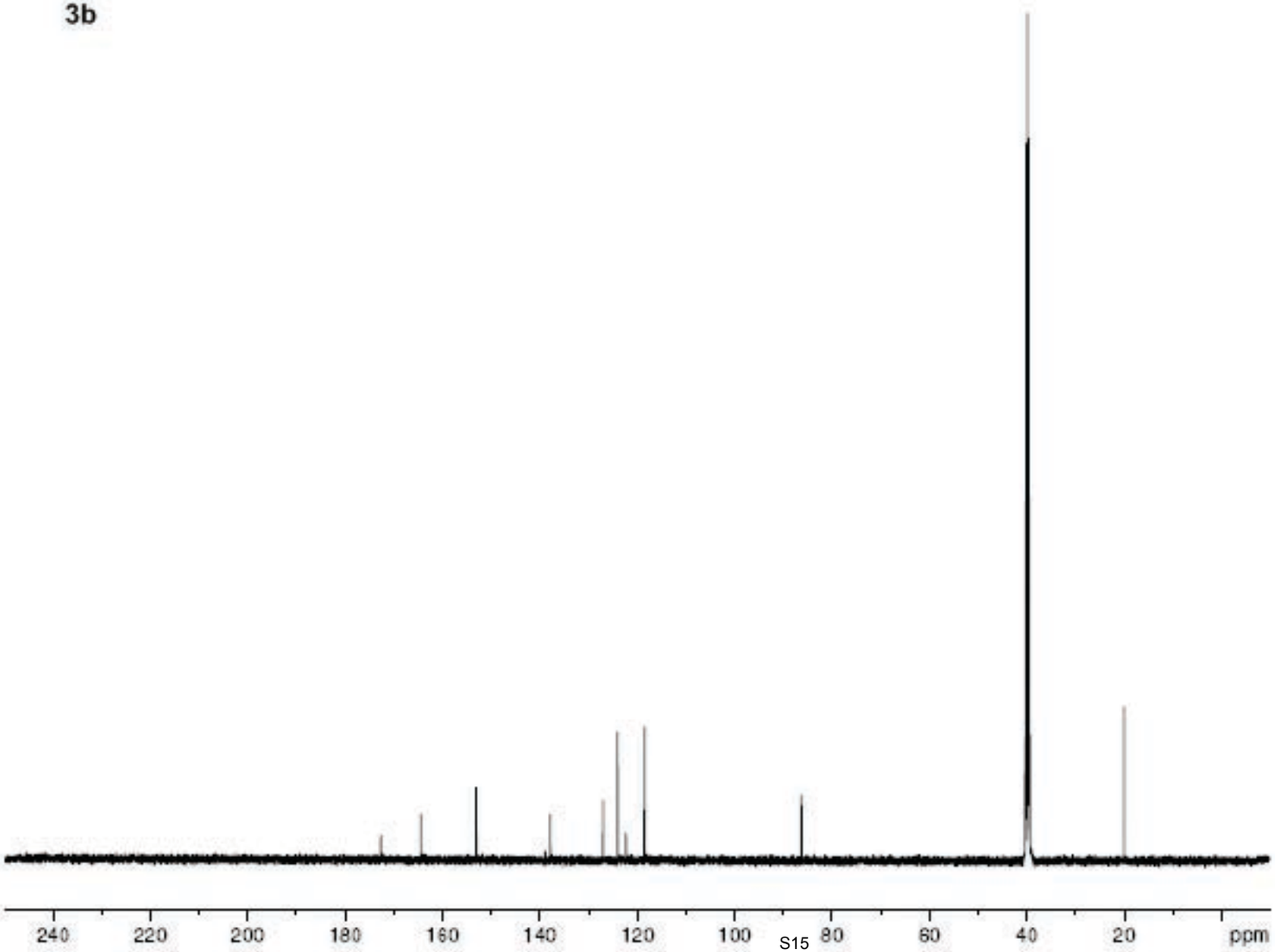
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3b

default carbon parameters (proton decoupled)

172.684
164.514
153.144
137.924
127.039
124.050
122.332
118.501
86.287
40.316
40.149
39.982
39.815
39.648
39.481
39.313
20.011



Current Data Parameters
NAME DAA-XII-279-1
EXPNO 2
PROCNO 1

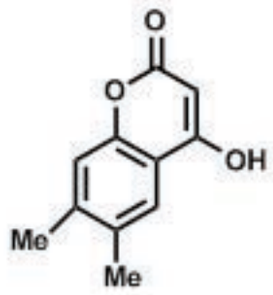
F2 – Acquisition Parameters
Date_ 20081002
Time 15.44
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 – Processing parameters
SI 65536
SF 125.8080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



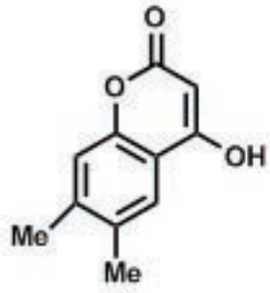
3c

Current Data Parameters
NAME DAA-XII-215-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080904
Time 16.22
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2769001 sec
RG 80.6
DW 50.000 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



3c

default carbon parameters (proton decoupled)

166.179
162.600
152.252
142.650
132.627
123.266
117.017
113.590
90.489
40.264
40.096
39.929
39.763
39.596
39.429
39.261
20.004
19.082

240 220 200 180 160 140 120 100 80 60 40 20 ppm

Current Data Parameters
NAME DAA-XII-215-4
EXPNO 2
PROCNO 1

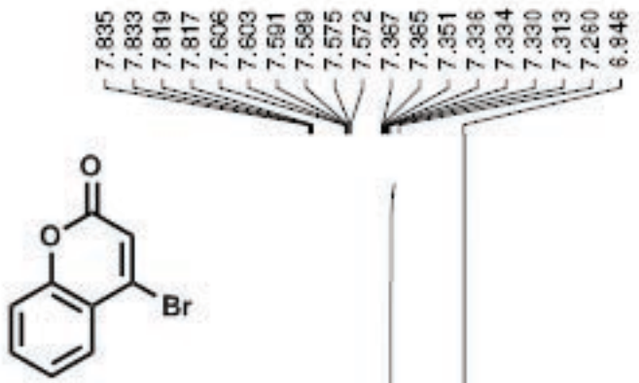
F2 - Acquisition Parameters
Date_ 20080904
Time 18.27
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.4 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

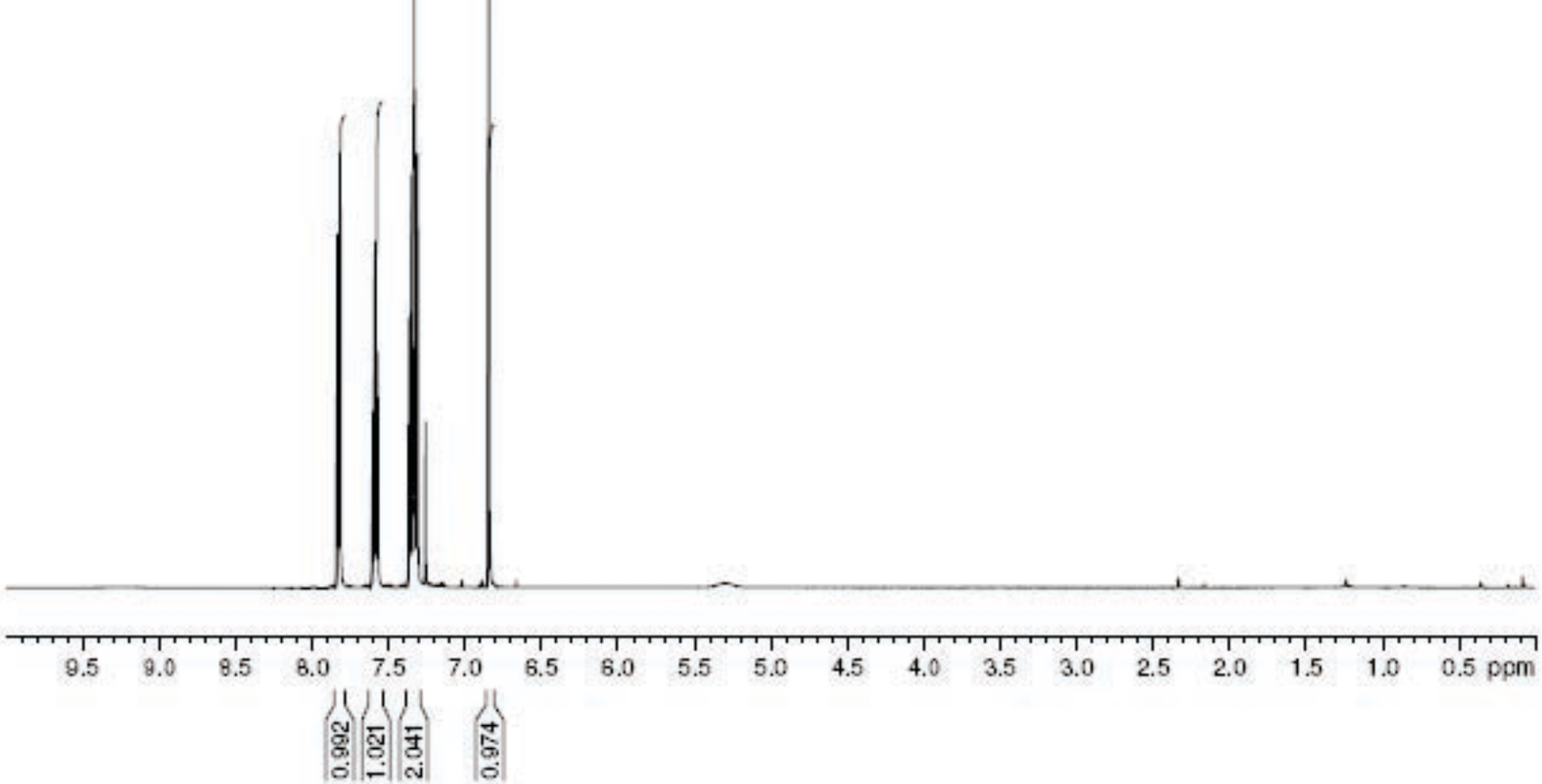
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.9080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Default proton parameters



4

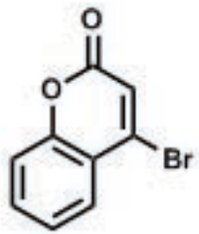


Current Data Parameters
NAME DAA-V-13-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050627
Time 18.23
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2768500 sec
RG 715
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H

F2 – Processing parameters
SI 32768
SF 500.1300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

Default parameters for C–13 with proton decoupling



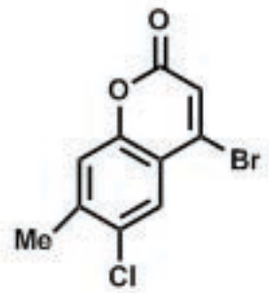
4

Current Data Parameters
NAME DAA–V–13–1
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050627
Time 18.30
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 16384
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 – Processing parameters
SI 32768
SF 125.7577986 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



4b

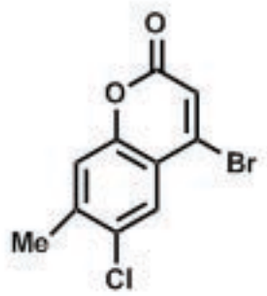
Current Data Parameters
NAME DAA-XII-291-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20081002
Time 15.48
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

default carbon parameters (proton decoupled)



4b

Current Data Parameters
NAME DAA-XII-281-1
EXPNO 2
PROCNO 1

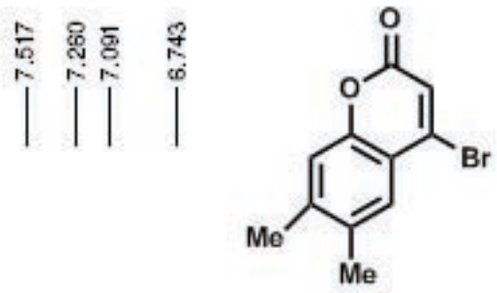
F2 - Acquisition Parameters
Date_ 20081002
Time 15.57
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 812.7
DW 15.300 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.9080873 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



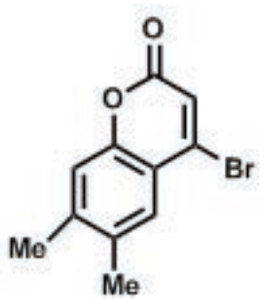
4c

Current Data Parameters
NAME DAA-XII-221-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080911
Time 10.07
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 296.4 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

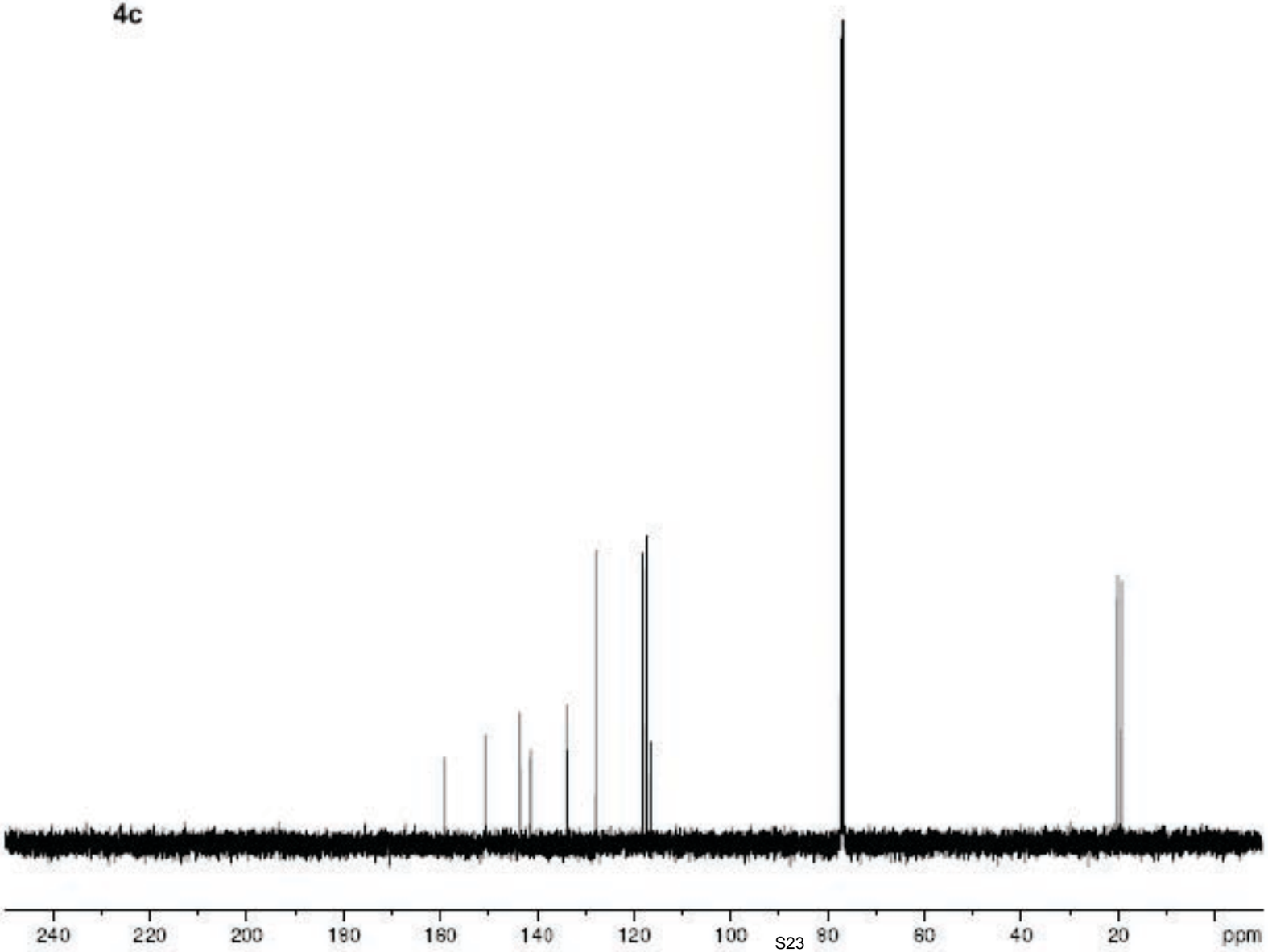
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



4c

default carbon parameters (proton decoupled)

159.173
150.733
143.573
141.352
133.848
127.840
119.209
117.414
116.588
77.253
76.999
76.745
20.164
19.263



Current Data Parameters
NAME DAA-XII-221-1
EXPNO 2
PROCNO 1

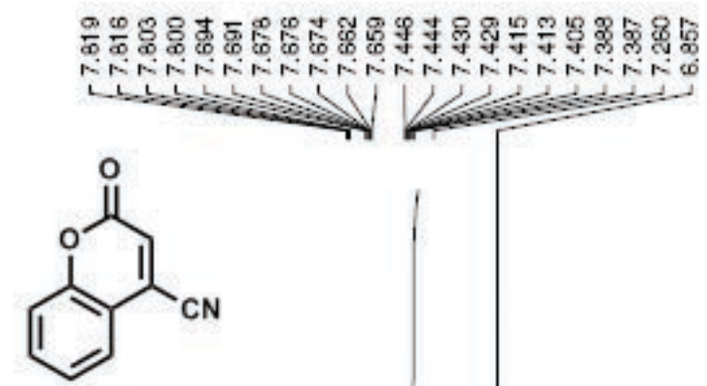
F2 – Acquisition Parameters
Date_ 20080911
Time 10.11
INSTRUM avance500
PROBHD 5 mm DD-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 – Processing parameters
SI 65536
SF 125.8080678 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Default proton parameters

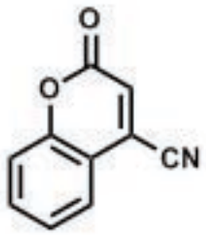


5/12a

Current Data Parameters
NAME DAA-V-149-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050808
Time 13.08
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2768500 sec
RG 1430
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H

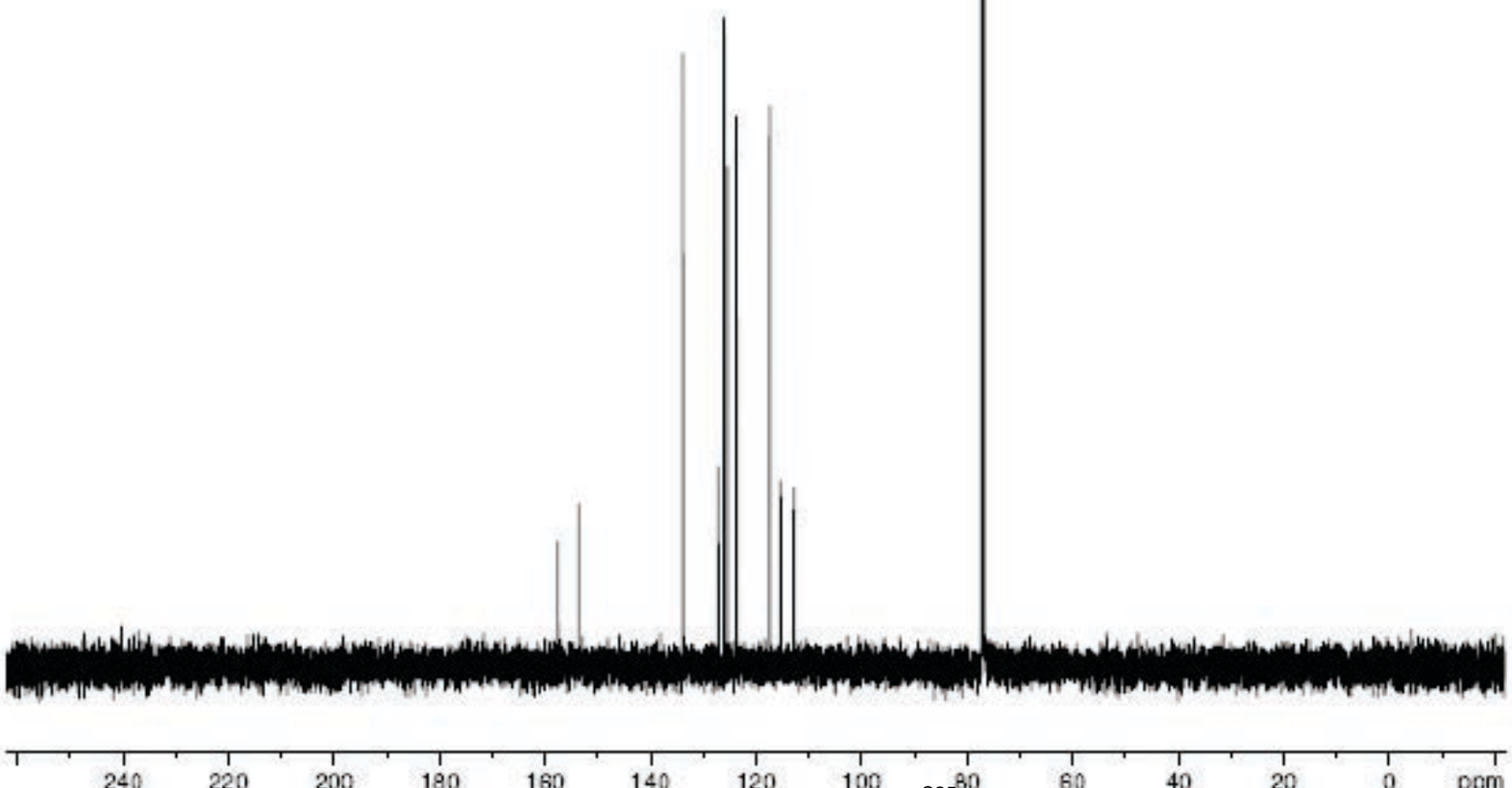
F2 – Processing parameters
SI 32768
SF 500.1300226 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



5/12a

Default parameters for C–13 with proton decoupling

157.641
153.652
133.910
127.176
126.049
125.483
123.736
117.503
115.312
112.994
77.252
76.997
76.744

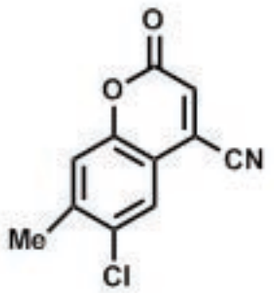


Current Data Parameters
NAME DAA–V–149–1
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050808
Time 13.12
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 – Processing parameters
SI 32768
SF 125.7577932 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



12b

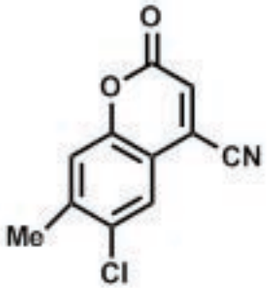
Current Data Parameters
NAME DAA-XII-283-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20081007
Time 6.36
INSTRUM avance500
PROBHD 5 mm DB-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 143.7
DW 50.000 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

default carbon parameters (proton decoupled)



12b

157.412
151.810
143.544
131.617
126.044
125.499
123.492
119.380
114.213
112.630
77.252
76.998
76.744
76.290
20.850

240 220 200 180 160 140 120 100 80 60 40 20 ppm

Current Data Parameters
NAME DAA-XII-293-1
EXPNO 2
PROCNO 1

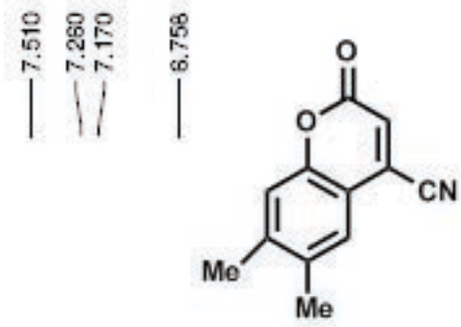
F2 – Acquisition Parameters
Date_ 20081007
Time 6.44
INSTRUM avance500
PROBHD 5 mm DD-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 812.7
DW 15.300 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 – Processing parameters
SI 65536
SF 125.8080863 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



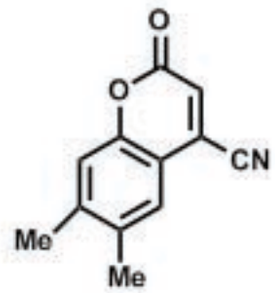
12c

Current Data Parameters
NAME DAA-XII-245-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080917
Time 9.07
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 362
DW 50.000 usec
DE 6.00 usec
TE 298.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

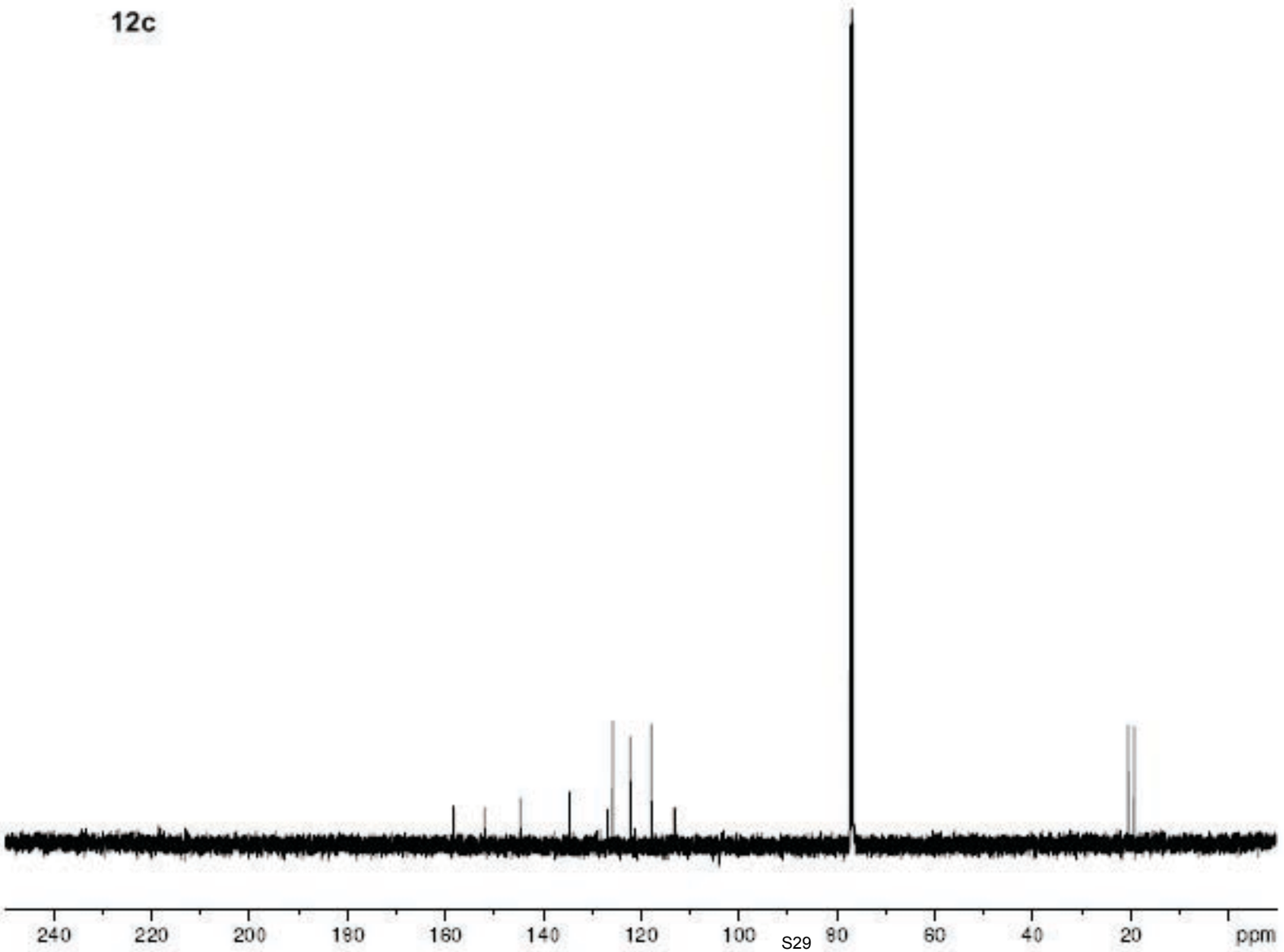
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



12c

default carbon parameters (proton decoupled)

158.316
152.018
144.685
134.717
126.912
125.842
122.216
117.938
113.230
113.046
77.252
76.998
76.744
20.481
19.230



Current Data Parameters
NAME DAA-XII-245-1
EXPNO 3
PROCNO 1

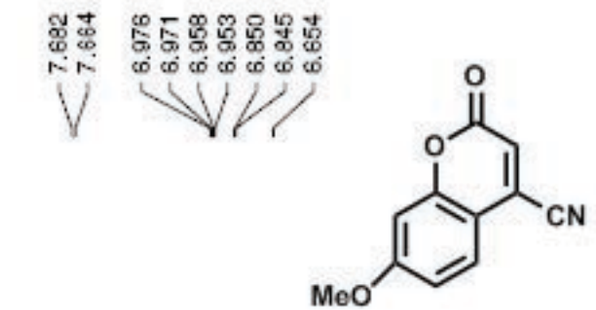
F2 – Acquisition Parameters
Date_ 20080917
Time 9.21
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT DMSO
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.2 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 – Processing parameters
SI 65536
SF 125.9080853 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



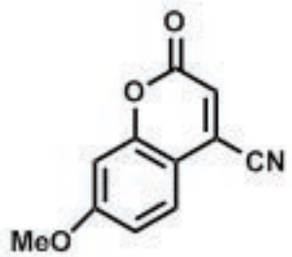
12d

Current Data Parameters
NAME DAA-XII-251-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080920
Time 7.56
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 295.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

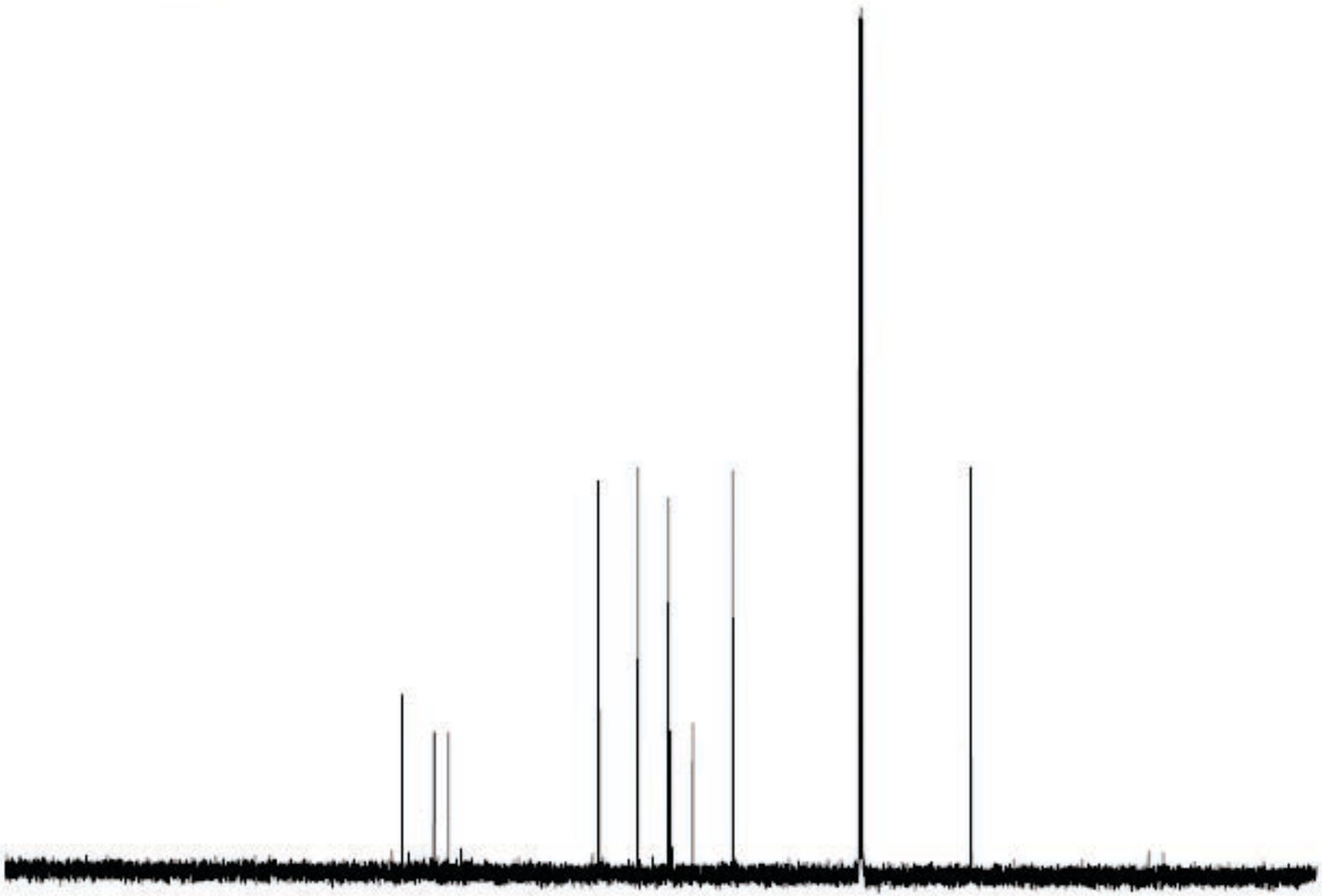
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



12d

default carbon parameters (proton decoupled)

164.393
158.263
155.601
127.027
126.880
119.488
113.679
113.206
109.008
101.359
77.252
76.998
76.744
56.057



Current Data Parameters
NAME DAA-XII-251-1
EXPNO 2
PROCNO 1

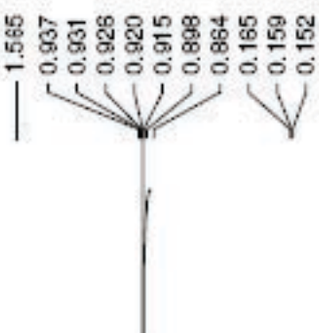
F2 - Acquisition Parameters
Date_ 20080920
Time 8.05
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.9080893 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Default proton parameters



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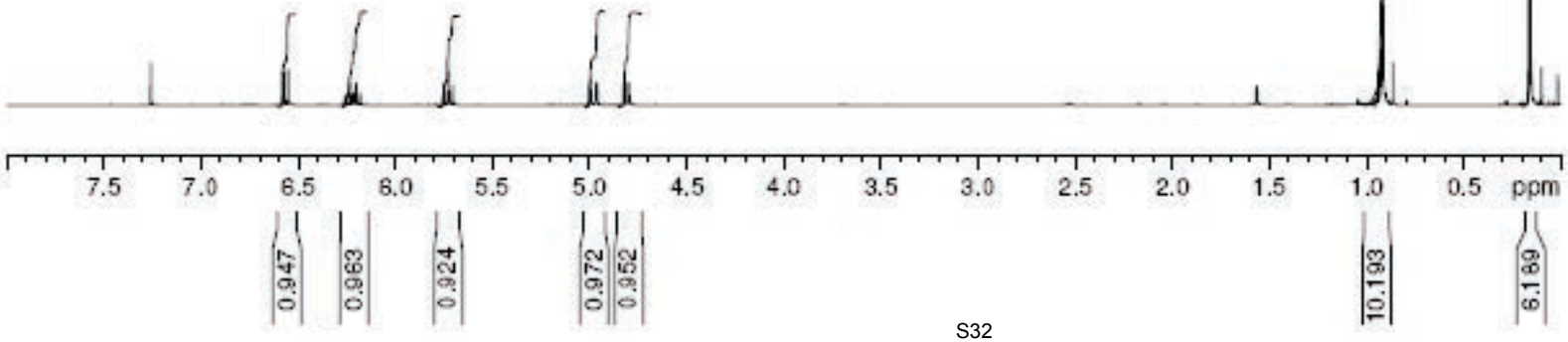
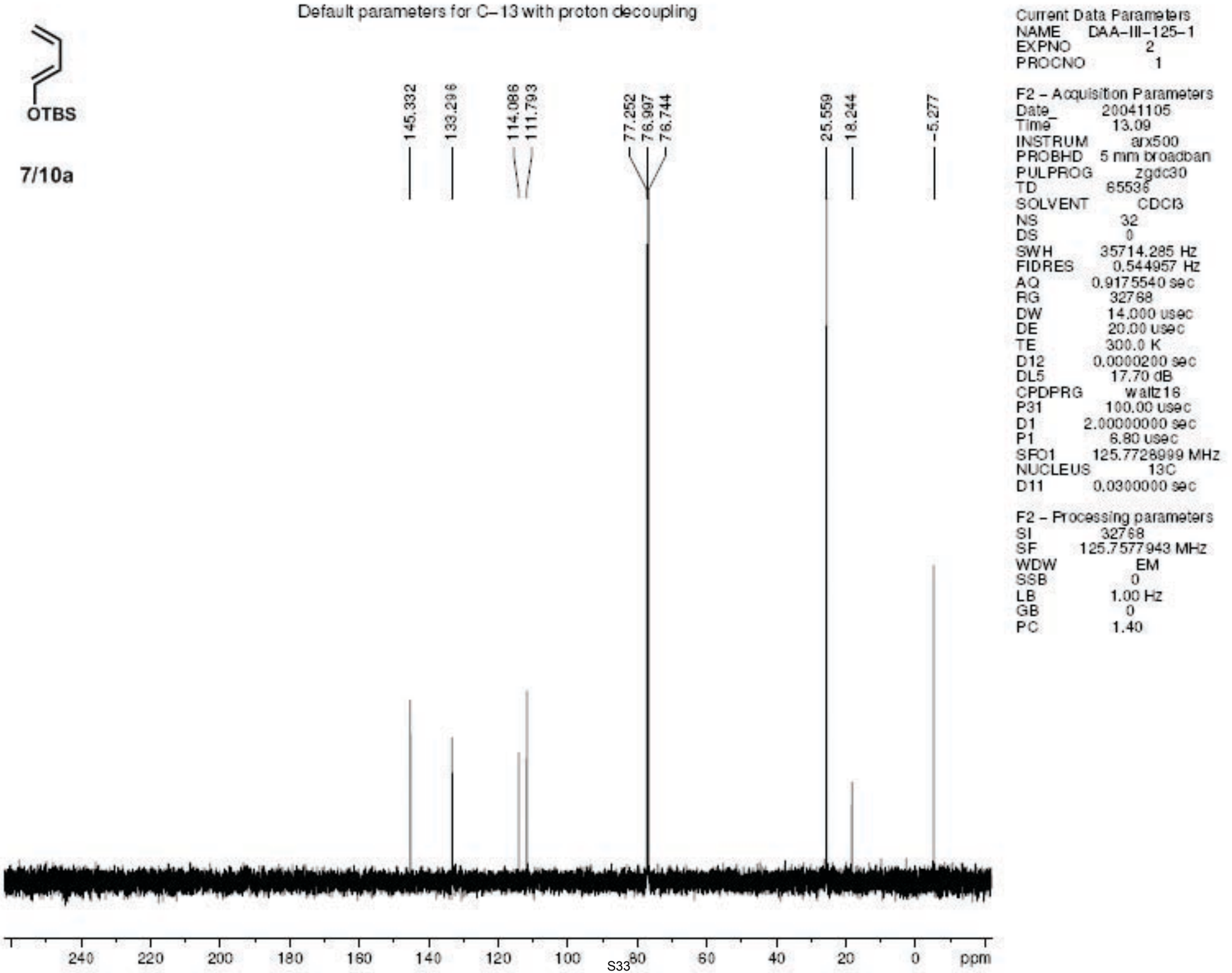
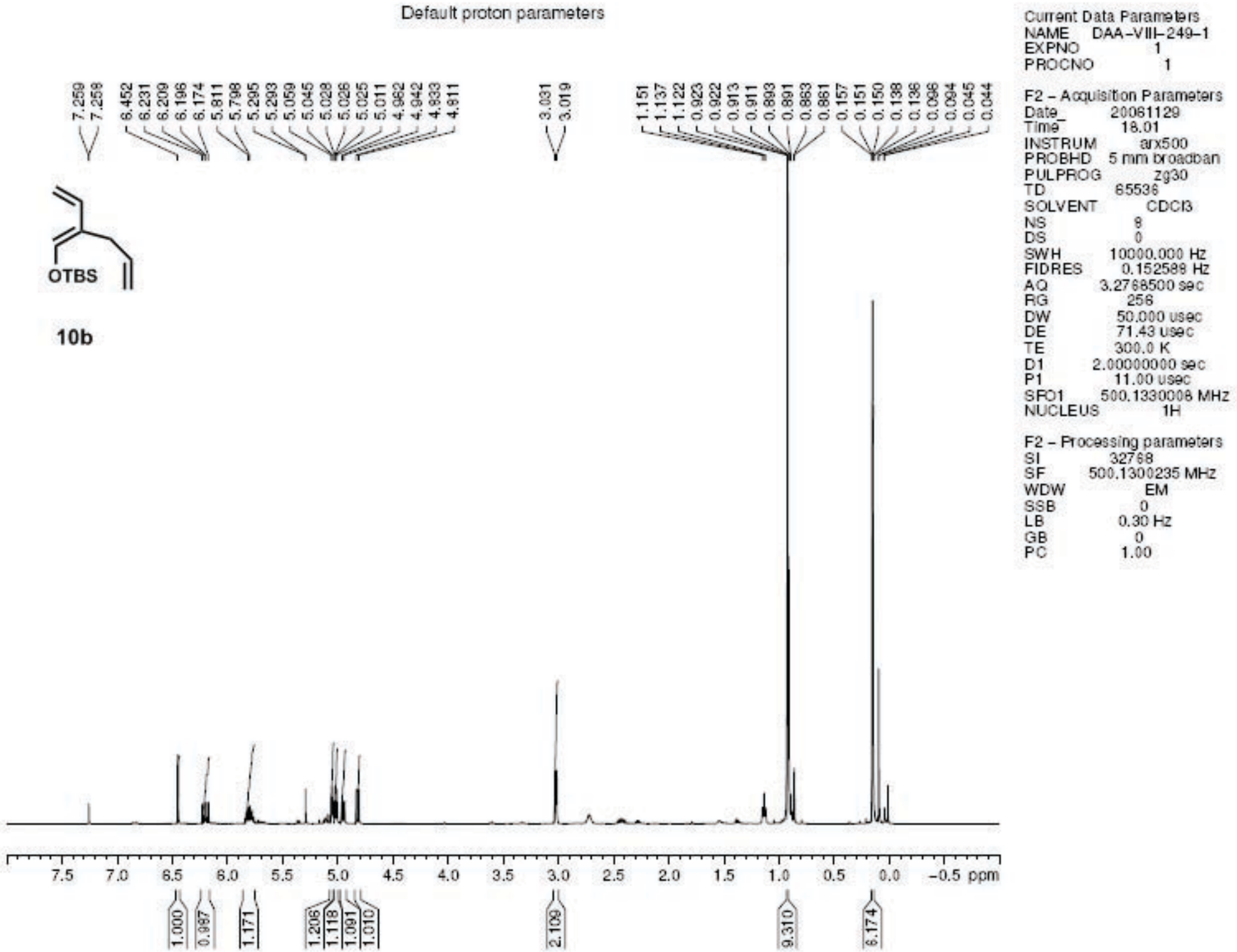
Current Data Parameters
NAME DAA-III-125-1
EXPNO 1
PROCNO 1

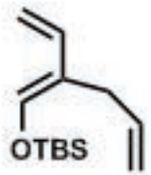
F2 - Acquisition Parameters
Date_ 20041105
Time 13.06
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 32768
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.305176 Hz
AQ 1.6394500 sec
RG 512
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 500.1300232 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00




Default parameters for C–13 with proton decoupling
OTBS
7/10a
145.332
133.296
114.086
111.793
77.252
76.997
76.744
25.559
18.244
-5.277
240
220
200
180
160
140
120
100
80
60
40
20
0
ppm
Current Data Parameters
NAME DAA–III–125–1
EXPNO 2
PROCNO 1
F2 – Acquisition Parameters
Date_ 20041105
Time 13.09
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 32
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec
F2 – Processing parameters
SI 32768
SF 125.7577943 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40


Default proton parameters
7.259
7.258
6.452
6.231
6.209
6.196
6.174
5.811
5.798
5.295
5.293
5.059
5.045
5.028
5.026
5.025
5.011
4.962
4.942
4.933
4.911
3.031
3.019
1.151
1.137
1.122
0.923
0.922
0.913
0.911
0.893
0.891
0.863
0.861
0.157
0.151
0.150
0.138
0.136
0.096
0.094
0.045
0.044
OTBS
10b
Current Data Parameters
NAME DAA-VIII-249-1
EXPNO 1
PROCNO 1
F2 – Acquisition Parameters
Date_ 20061129
Time 18.01
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2768500 sec
RG 256
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H
F2 – Processing parameters
SI 32768
SF 500.1300235 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
-0.5 ppm
1.000
0.997
1.171
1.206
1.118
1.091
1.010
2.109
9.310
6.174



10b

Default parameters for C–13 with proton decoupling

142.310
136.178
135.300
120.203
114.498
108.472
77.247
76.994
76.739
27.971
25.498
18.121
-5.357

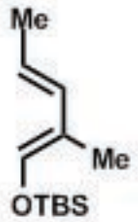
240 220 200 180 160 140 120 100 80 60 40 20 0 ppm

Current Data Parameters
NAME DAA-VIII-249-1
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20061129
Time 18.06
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 – Processing parameters
SI 32768
SF 125.7577954 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



10c

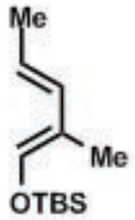
Current Data Parameters
NAME DAA-XII-293-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20081009
Time 17.34
INSTRUM avance500
PROBHD 5 mm DD-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 57
DW 50.000 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

default carbon parameters (proton decoupled)



10c

Current Data Parameters
NAME DAA-XII-293-1
EXPNO 2
PROCNO 1

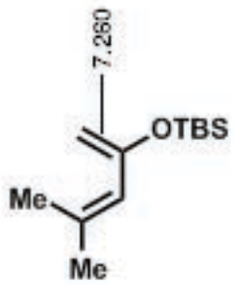
F2 - Acquisition Parameters
Date_ 20081009
Time 17.38
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.8080823 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



10f

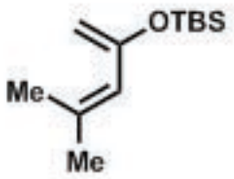
Current Data Parameters
NAME DAA-XII-91-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080530
Time 14.42
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 295.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

default carbon parameters (proton decoupled)



10f

Current Data Parameters
NAME DAA-XII-91-1
EXPNO 2
PROCNO 1

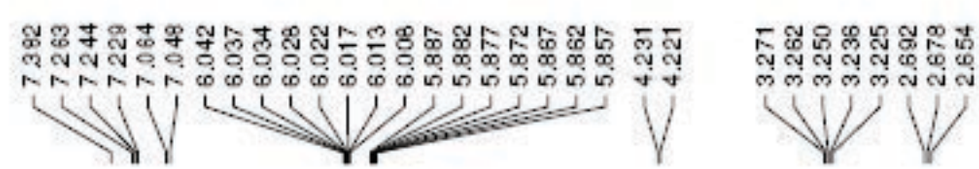
F2 - Acquisition Parameters
Date_ 20080530
Time 14.48
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.739 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 5792.6
DW 15.300 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

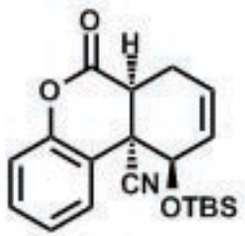
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.9080638 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters





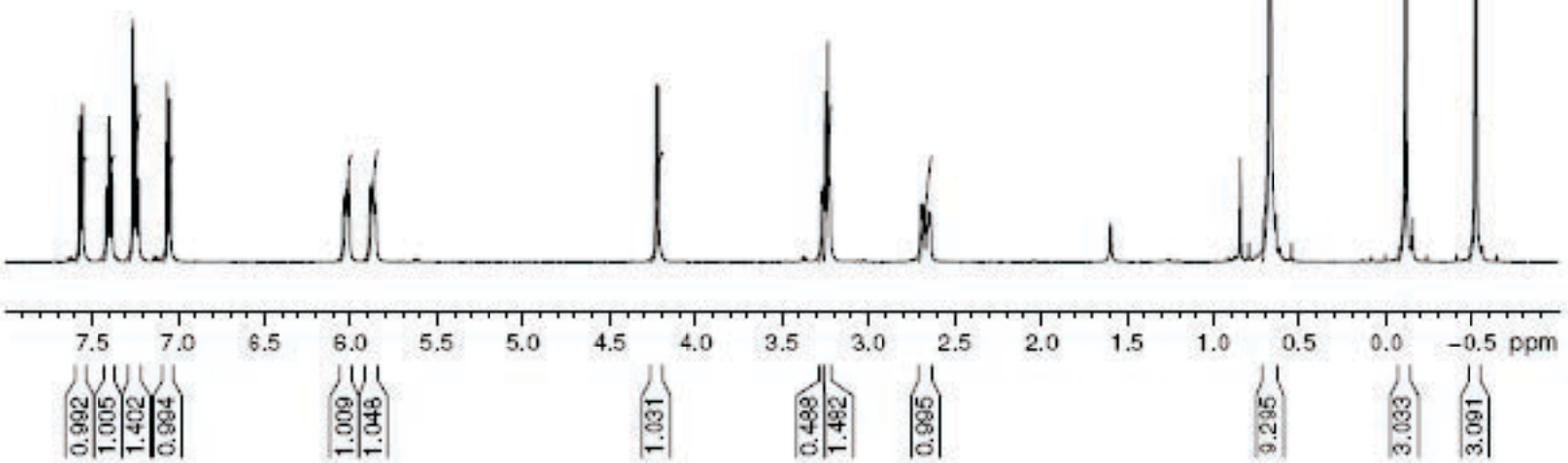
8n/11a

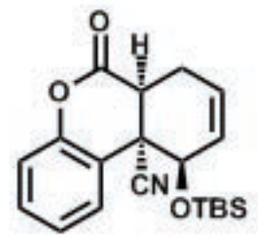
Current Data Parameters
NAME DAA-V-197-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20050831
Time 10.12
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 114
DW 50.000 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





8n/11a

default carbon parameters (proton decoupled)

166.001
151.769
130.528
128.110
126.708
125.270
124.685
120.203
118.810
117.338
77.254
77.000
76.746
68.933
44.273
36.561
25.167
22.372
17.599
-5.131
-6.107

Current Data Parameters
NAME DAA-V-197-
EXPNO 2
PROCNO 1

F2 - Acquisition Paramet
Date_ 20050831
Time 10.17
INSTRUM avance500
PROBHD 5 mm bb-Z Z
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.739 H
FIDRES 0.498653 H
AQ 1.0027661 sec
RG 912.3
DW 15.300 usec
DE 6.00 usec
TE 296.5 K
D1 2.00000000 se
d11 0.03000000 se
MCREST 0.00000000
MCWRK 0.01500000

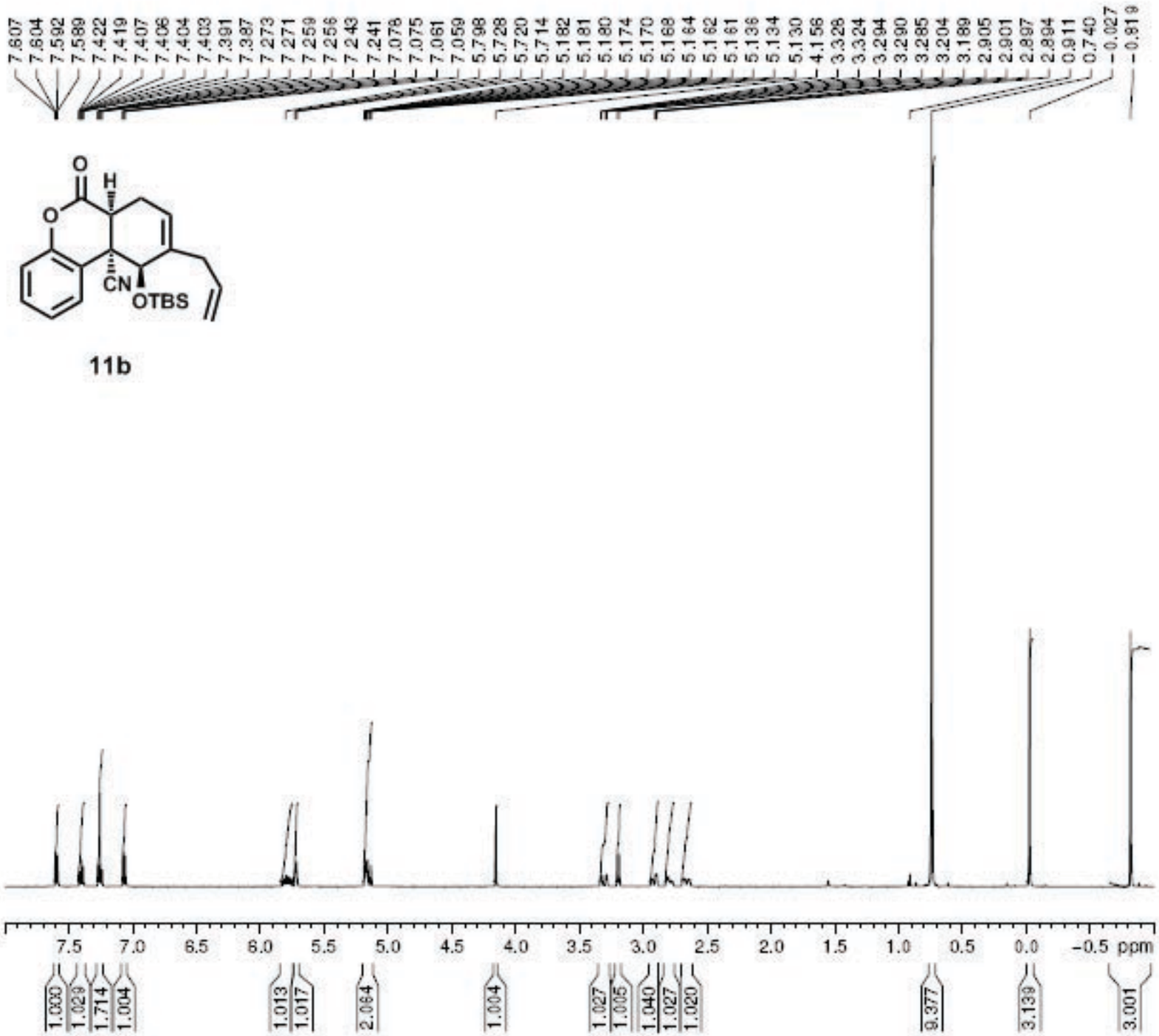
======== CHANNEL f1 =
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 M

======== CHANNEL f2 =
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 M

F2 - Processing paramet
SI 65536
SF 125.9080878 MI
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

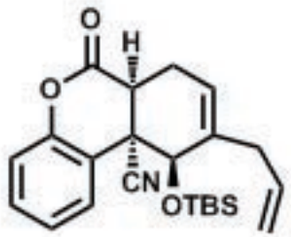
Default proton parameters



Current Data Parameters
NAME DAA-VIII-251-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20061130
Time 15.11
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H

F2 - Processing parameters
SI 32768
SF 500.1300232 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



11b

Default parameters for C–13 with proton decoupling

166.196
151.747
134.811
134.438
130.728
127.202
124.881
123.900
120.393
118.908
117.816
117.653
77.247
76.994
76.739
72.021
45.263
38.924
36.245
25.419
22.399
17.941
-4.681
-5.719

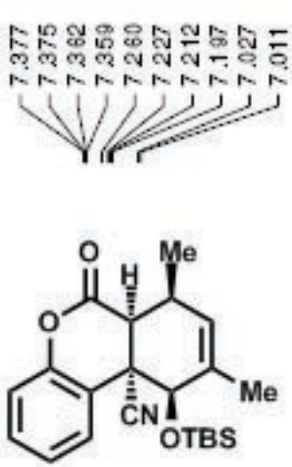
240 220 200 180 160 140 120 100 80 60 40 20 0 ppm

Current Data Parameters
NAME DAA-VIII-251-2
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20061130
Time 23.09
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 16384
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 – Processing parameters
SI 32768
SF 125.7577964 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



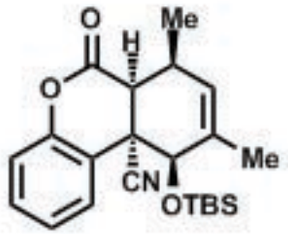
11c

Current Data Parameters
NAME DAA-XII-295-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081009
Time 17.48
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 40.3
DW 50.000 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



11c

default carbon parameters (proton decoupled)

163.979
152.219
130.751
130.542
129.326
127.098
124.395
121.037
119.833
117.385
77.251
76.997
76.742
73.348
47.481
41.159
30.210
25.817
25.414
22.281
17.823
17.626
-4.534
-5.949

Current Data Parameters
NAME DAA-XII-295-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081009
Time 17.52
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.9 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

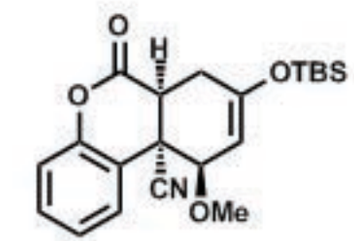
======== CHANNEL f1 ====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.9080943 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

240 230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

default proton parameters



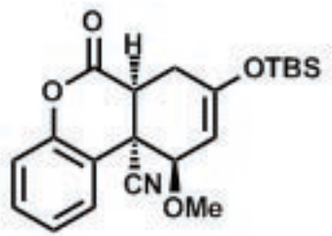
11e

Current Data Parameters
NAME DAA-XII-191-2
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080730
Time 11.54
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT Aceton
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2769001 sec
RG 80.6
DW 50.000 usec
DE 6.00 usec
TE 296.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



11e

default carbon parameters (proton decoupled)

165.914
165.577
153.456
151.520
150.699
149.259
130.623
128.524
126.819
125.102
124.635
121.088
119.666
119.642
117.402
117.328
117.195
102.073
100.451
79.480
79.130
77.252
76.998
76.744
58.307
56.950
44.351
43.469
40.749
37.968
27.215
26.833
25.647
25.495
17.947
-4.355
-4.377

Current Data Parameters
NAME DAA-XII-181-2
EXPNO 2
PROCNO 1

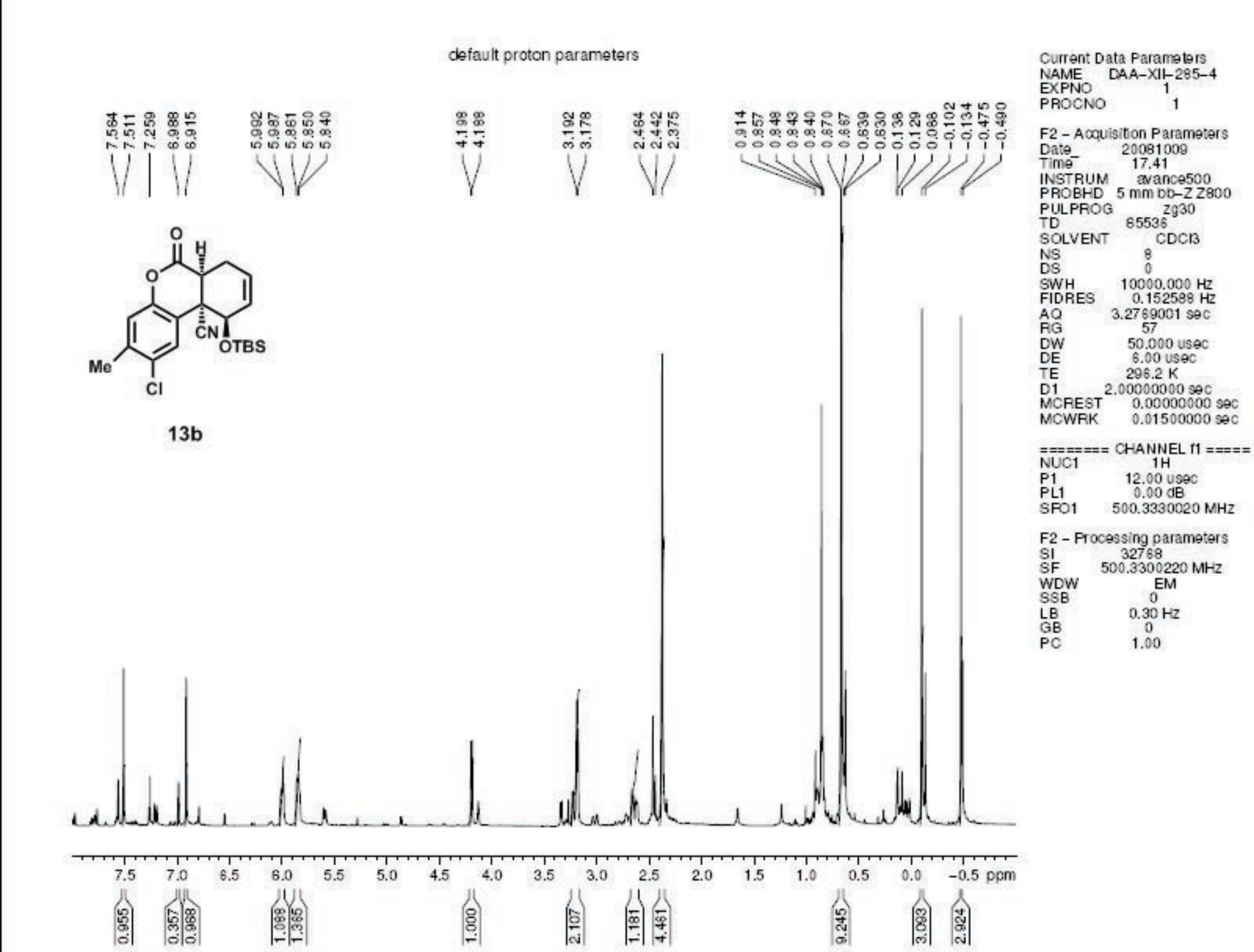
F2 - Acquisition Parameters
Date_ 20080730
Time 11.58
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT DMSO
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 456.1
DW 15.300 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

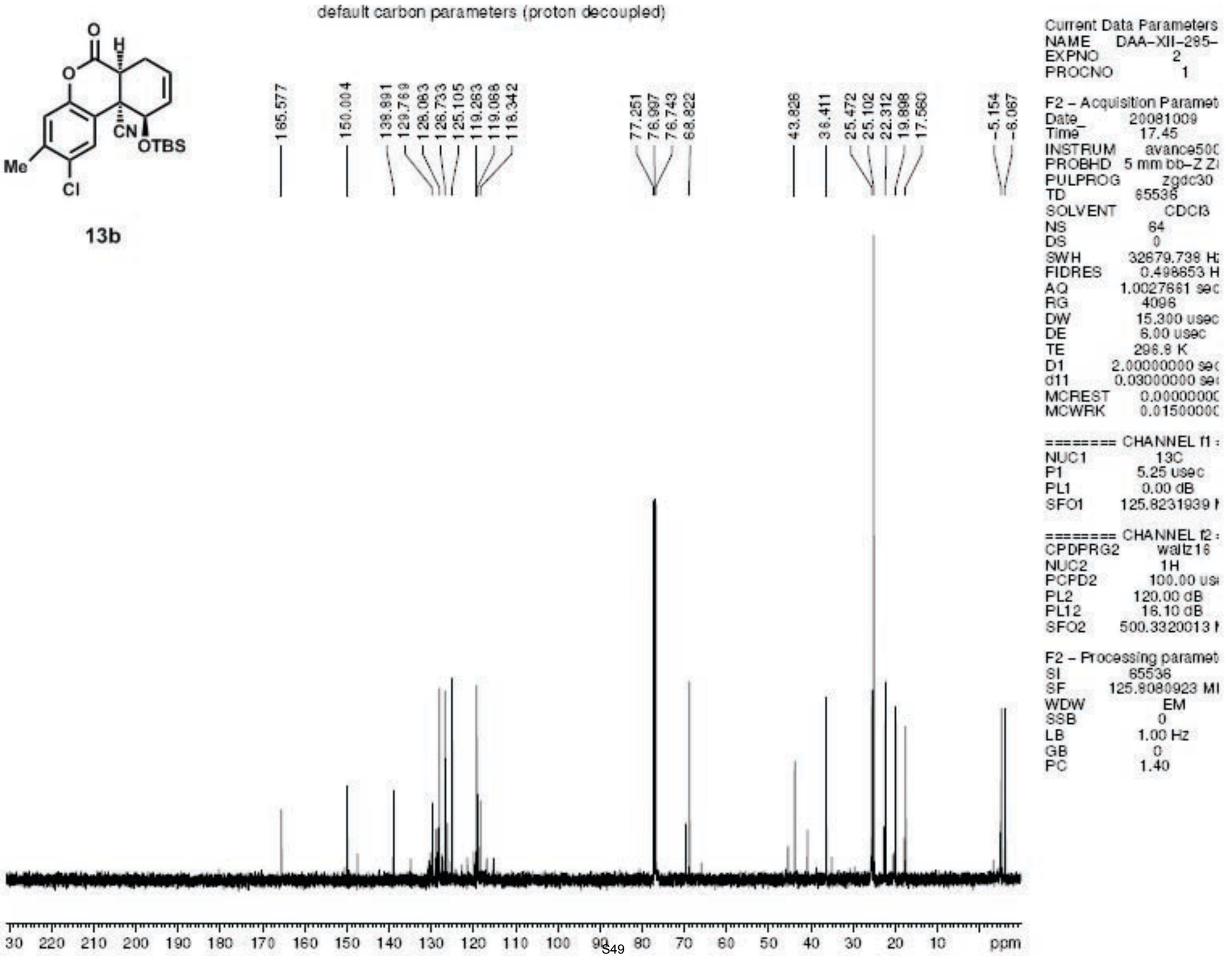
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

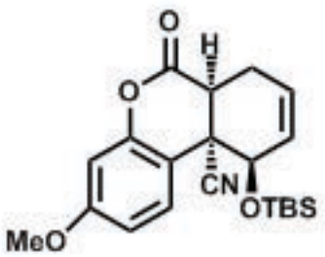
F2 - Processing parameters
SI 65536
SF 125.8080898 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

240 220 200 180 160 140 120 100 80 60 40 20 ppm


default proton parameters
7.564
7.511
7.259
6.988
6.915
5.992
5.987
5.861
5.850
5.840
4.198
4.189
3.192
3.178
2.464
2.442
2.375
0.914
0.857
0.848
0.843
0.840
0.670
0.667
0.639
0.630
0.138
0.129
0.088
-0.102
-0.134
-0.475
-0.490
O
O
H
CN
OTBS
Me
Cl
13b
Current Data Parameters
NAME DAA-XII-285-4
EXPNO 1
PROCNO 1
F2 - Acquisition Parameters
Date_ 20081009
Time 17.41
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 57
DW 50.000 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec
======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00
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
-0.5 ppm
0.955
0.357
0.968
1.069
1.365
1.000
2.107
1.181
4.461
9.245
3.093
2.924


default carbon parameters (proton decoupled)
O
H
O
CN
OTBS
Me
Cl
13b
165.577
150.004
139.891
129.769
128.083
126.733
125.105
119.263
119.068
118.342
77.251
76.997
76.743
68.822
43.826
36.411
25.472
25.102
22.312
19.898
17.560
-5.154
-6.067
Current Data Parameters
NAME DAA-XII-295-
EXPNO 2
PROCNO 1
F2 - Acquisition Paramet
Date_ 20081009
Time 17.45
INSTRUM avance50(
PROBHD 5 mm bb-Z Z
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 H
FIDRES 0.498653 H
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.8 K
D1 2.00000000 se
d11 0.03000000 se
MCREST 0.0000000(
MCWRK 0.0150000(
======== CHANNEL f1 =
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 M
======== CHANNEL f2 =
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 us
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 M
F2 - Processing paramet
SI 65536
SF 125.9080923 MI
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

default proton parameters



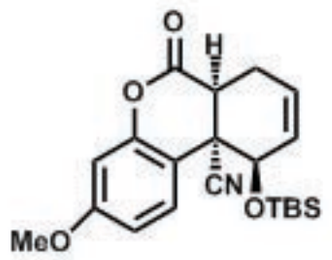
13d

Current Data Parameters
NAME DAA-XII-265-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080923
Time 10.34
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 35.9
DW 50.000 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



13d

default carbon parameters (proton decoupled)

165.999
161.252
152.584
127.969
127.353
125.308
118.929
112.153
110.437
102.750
77.252
76.997
76.743
68.872
55.639
43.625
36.668
25.541
25.138
22.407
17.545
-5.190
-6.030

Current Data Parameters
NAME DAA-XII-265-4
EXPNO 2
PROCNO 1

F2 - Acquisition Parameter
Date_ 20080923
Time 10.40
INSTRUM avance500
PROBHD 5 mm bb-Z Z8
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 8192
DW 15.300 usec
DE 6.00 usec
TE 295.9 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 s
MCWRK 0.01500000 s

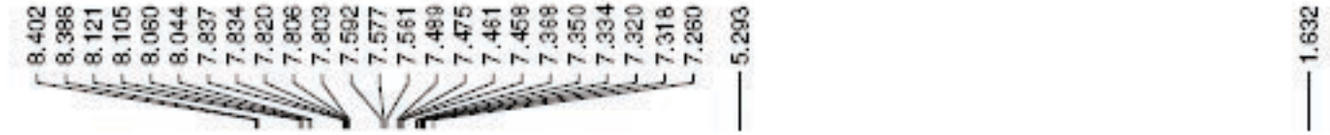
======== CHANNEL f1 =
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 M

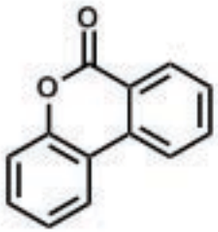
======== CHANNEL f2 =
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 M

F2 - Processing parameter
SI 65536
SF 125.8080938 MH
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

30 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

default proton parameters





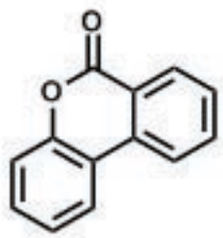
14a

Current Data Parameters
NAME DAA-XII-175-3
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080730
Time 10.25
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 128
DW 50.000 usec
DE 6.00 usec
TE 296.2 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

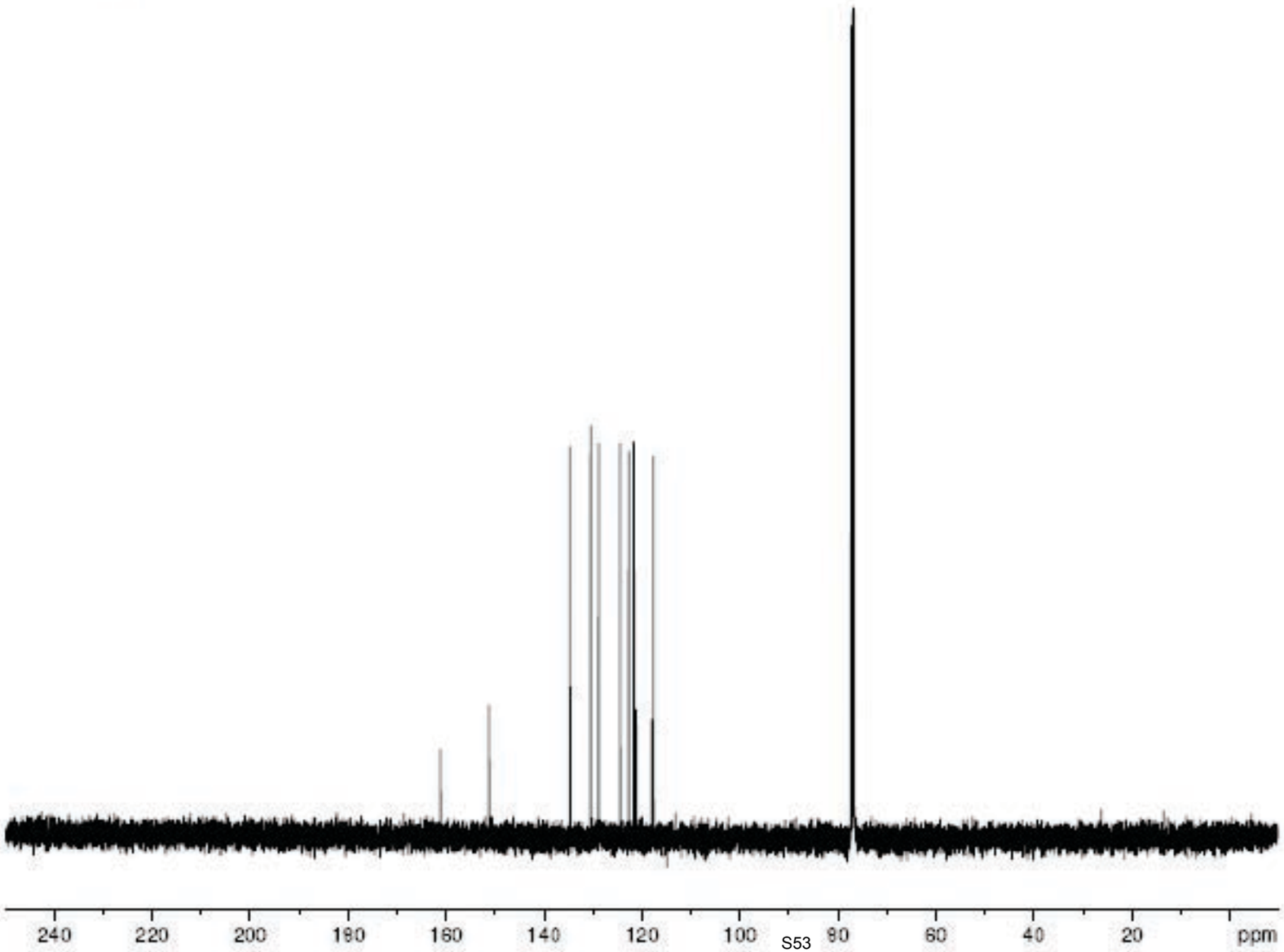
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



14a

default carbon parameters (proton decoupled)

161.159
151.245
134.811
134.724
130.538
130.408
128.950
124.521
122.731
121.651
121.214
117.999
117.745
77.260
77.036
76.751



Current Data Parameters
NAME DAA-XII-175-3
EXPNO 2
PROCNO 1

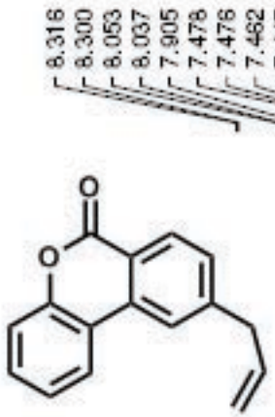
F2 – Acquisition Parameters
Date_ 20080730
Time 10.30
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1625.5
DW 15.300 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 – Processing parameters
SI 65536
SF 125.8080868 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

Default proton parameters



14b

Current Data Parameters

NAME	DAA-XII-193-2
EXPNO	1
PROCNO	1

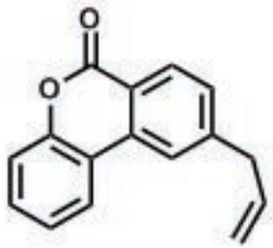
F2 – Acquisition Parameters

Date_	20080730
Time	19.59
INSTRUM	arx500
PROBHD	5 mm broadban
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	8
DS	0
SWH	10000.000 Hz
FIDRES	0.152588 Hz
AQ	3.2768500 sec
RG	715
DW	50.000 usec
DE	71.43 usec
TE	300.0 K
D1	2.00000000 sec
P1	11.00 usec
SFO1	500.1330008 MHz
NUCLEUS	1H

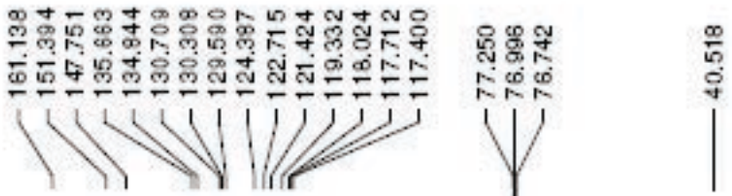
F2 – Processing parameters

SI	32768
SF	500.1300241 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

Default parameters for C–13 with proton decoupling



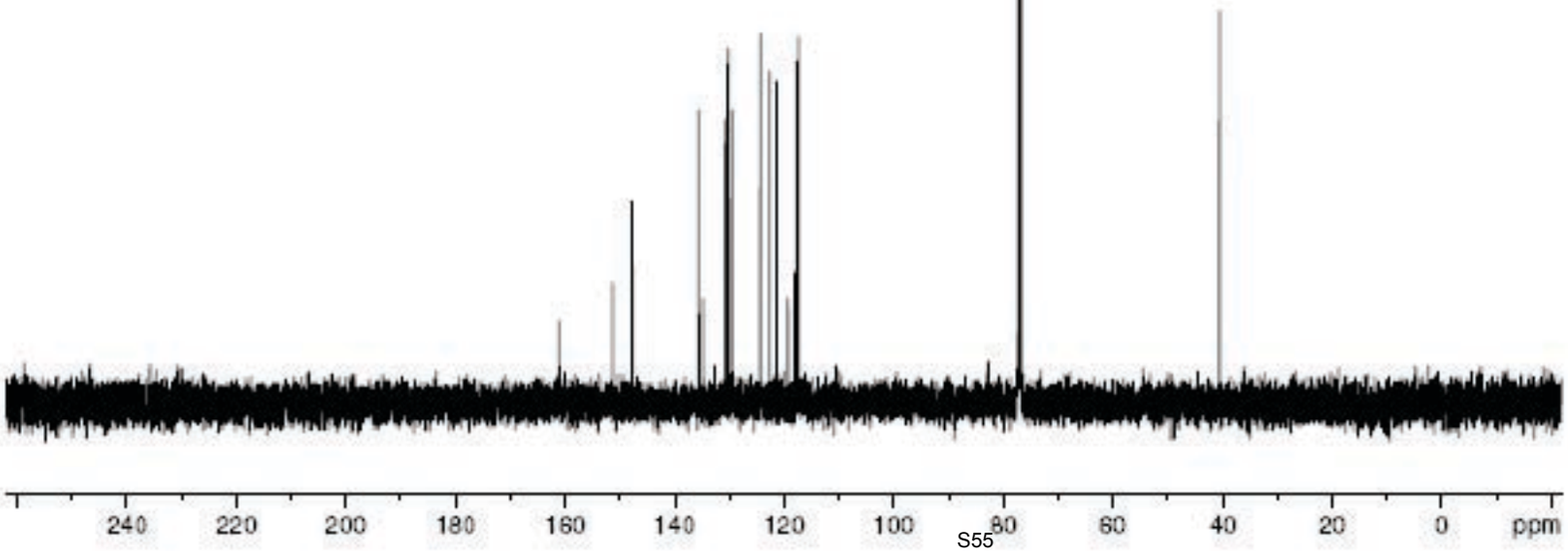
14b



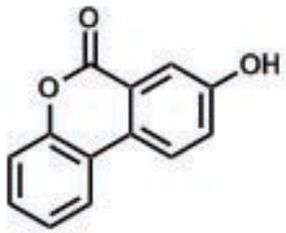
Current Data Parameters
NAME DAA–XII–193–2
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080730
Time 20.12
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 45500
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 – Processing parameters
SI 32768
SF 125.7577975 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

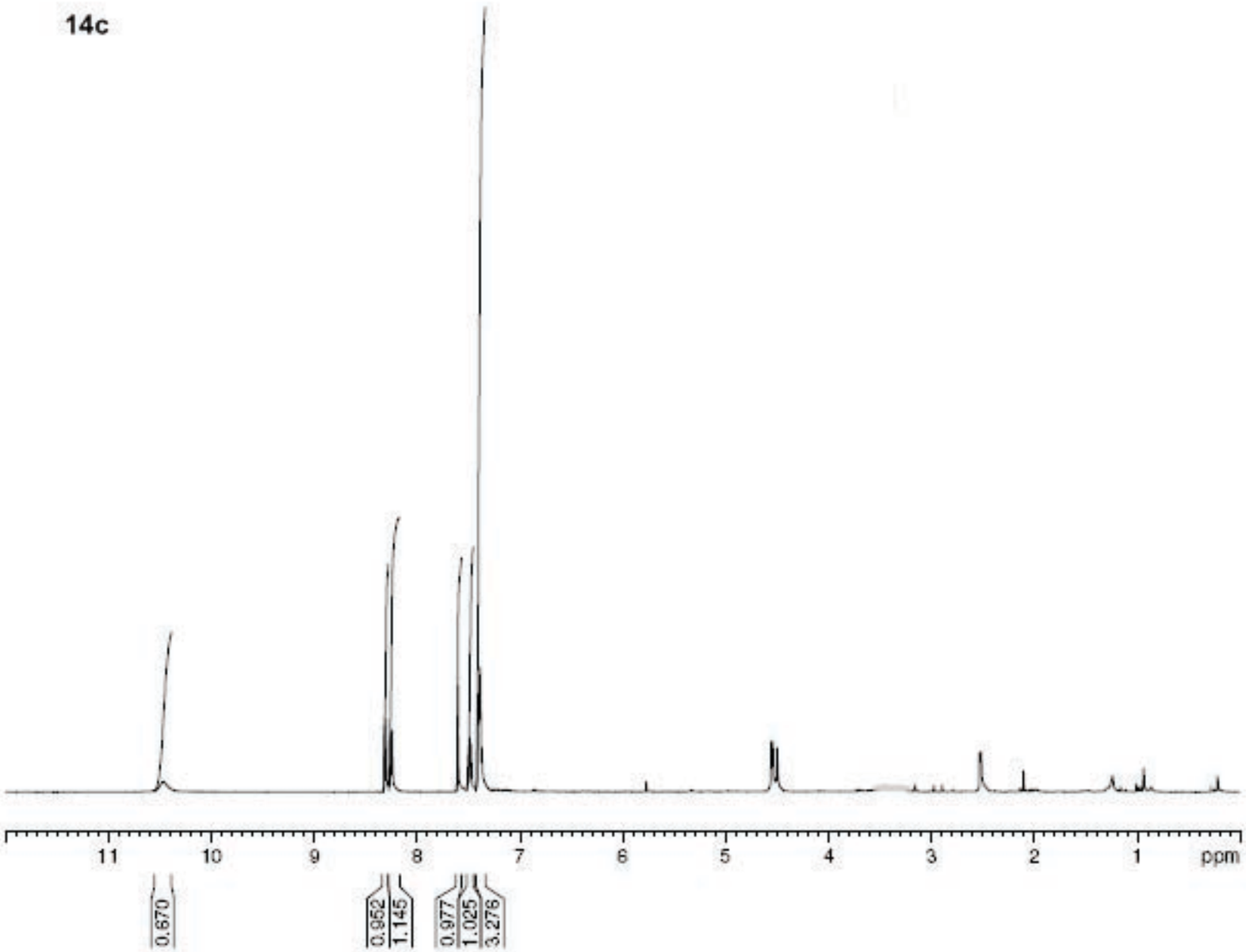


Default proton parameters



8.319
8.302
8.280
8.244
7.609
7.604
7.493
7.491
7.477
7.411
7.405
7.399
7.396
7.393
7.388
7.372

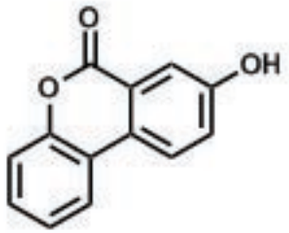
14c



Current Data Parameters
NAME DAA-XII-199-5
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080731
Time 22.05
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H

F2 – Processing parameters
SI 32768
SF 500.1300000 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



14c

Default parameters for C–13 with proton decoupling

160.204
158.288
149.631
129.114
125.946
124.697
124.606
123.968
122.722
121.926
118.103
117.016
113.787

40.008
39.841
39.674
39.507
39.340
39.173
39.006

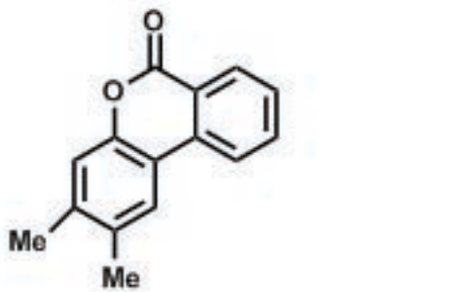
Current Data Parameters
NAME DAA-XII-199-5
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080731
Time 22.13
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 32768
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 – Processing parameters
SI 32768
SF 125.7578497 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

240 220 200 180 160 140 120 100 80 60 40 20 0 ppm

default proton parameters



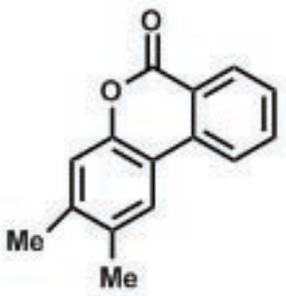
14d

Current Data Parameters
NAME DAA-XII-277-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080929
Time 14.10
INSTRUM avance500
PROBHD 5 mm BB-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 80.6
DW 50.000 usec
DE 6.00 usec
TE 295.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

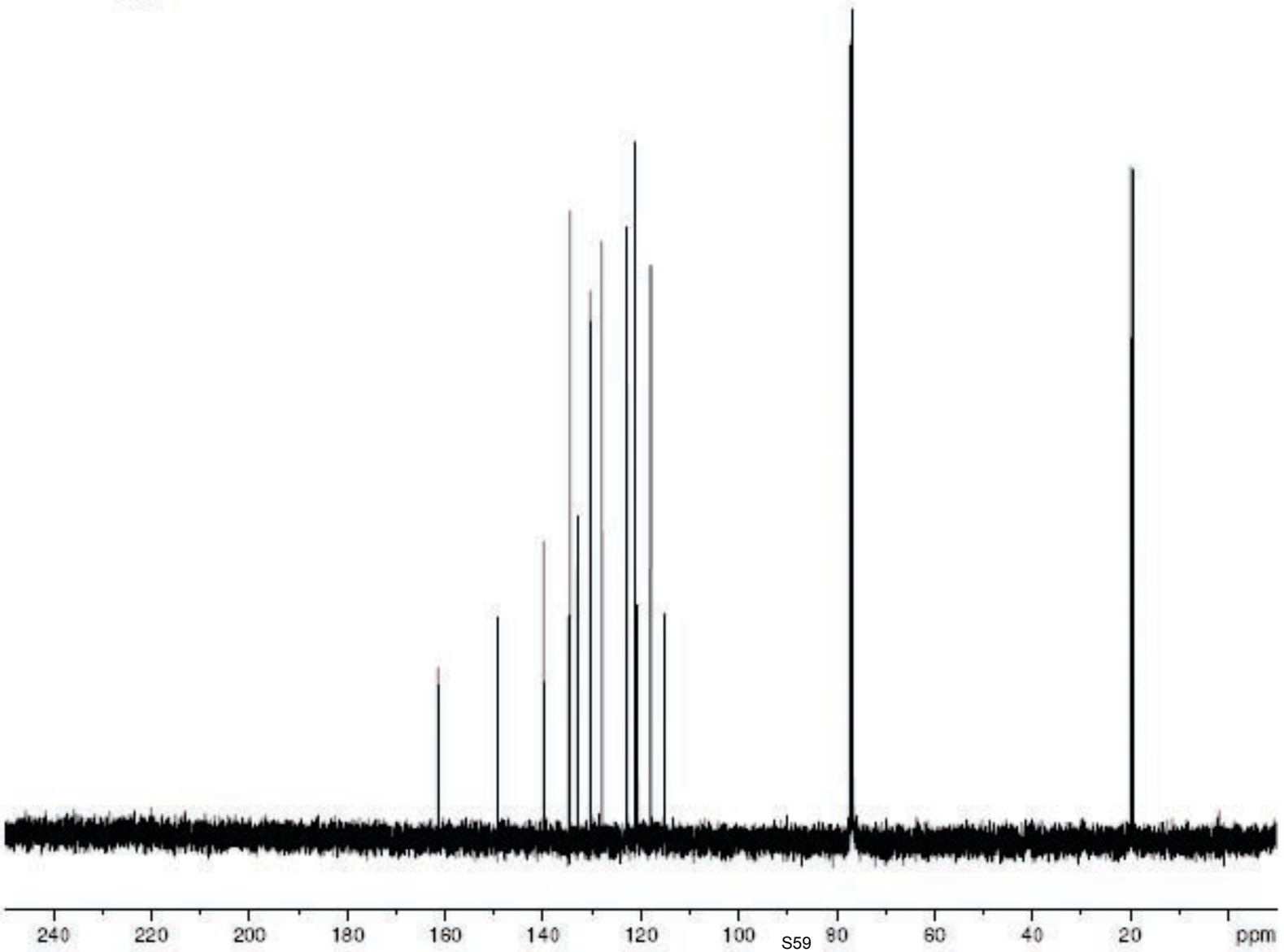
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



14d

default carbon parameters (proton decoupled)

161.443
149.338
139.889
134.870
134.517
132.959
130.336
128.026
122.926
121.232
120.811
118.021
115.227
77.254
77.000
76.746
19.895
19.422



Current Data Parameters
NAME DAA-XII-277-1
EXPNO 2
PROCNO 1

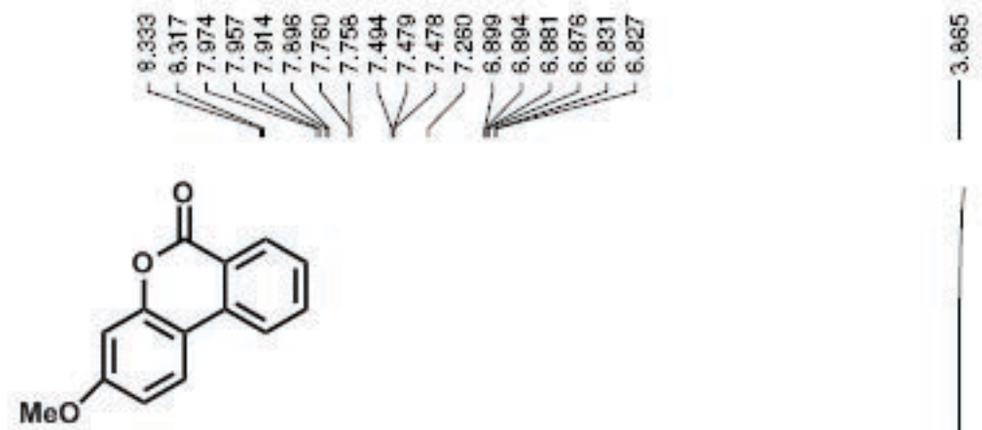
F2 – Acquisition Parameters
Date_ 20080929
Time 14.15
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT DMSO
NS 64
DS 0
SWH 32679.739 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 3649.1
DW 15.300 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

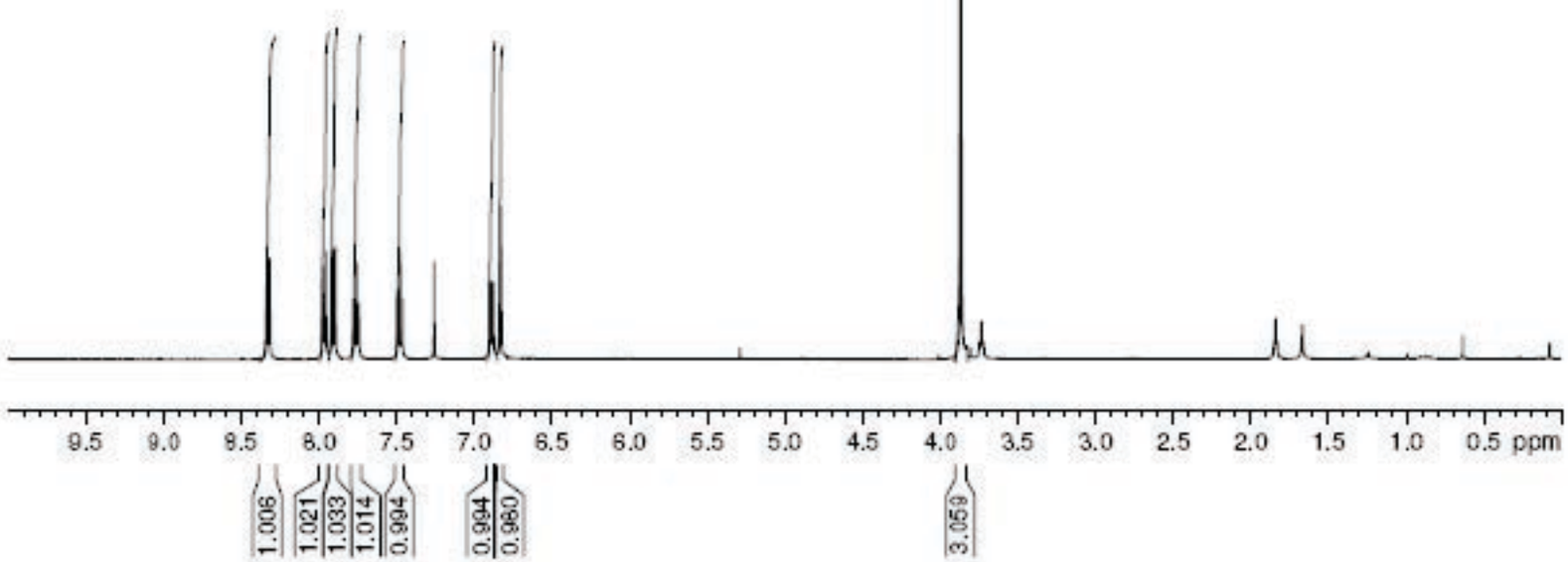
======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 – Processing parameters
SI 65536
SF 125.9080908 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



14e

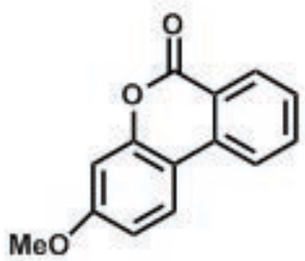


Current Data Parameters
NAME DAA-XII-269-2
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080926
Time 7.49
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 295.5 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



14e

default carbon parameters (proton decoupled)

161.570
160.828
152.437
135.678
135.069
130.025
128.428
125.063
122.307
119.574
112.684
110.988
101.822
56.124
40.336
40.168
40.002
39.835
39.668
39.501
39.334

Current Data Parameters
NAME DAA-XII-269-5
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080928
Time 16.35
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 256
DS 0
SWH 32679.739 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

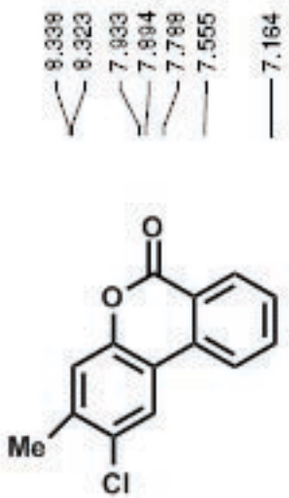
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.9080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

240 220 200 180 160 140 120 100 80 60 40 20 ppm

default proton parameters



14f

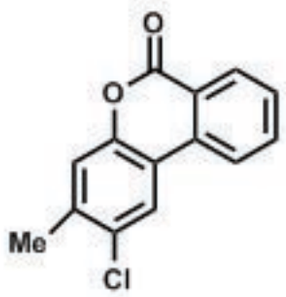
Current Data Parameters
NAME DAA-XIII-15-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081013
Time 13.11
INSTRUM avance500
PROBHD 5 mm BB-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 114
DW 50.000 usec
DE 6.00 usec
TE 295.3 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

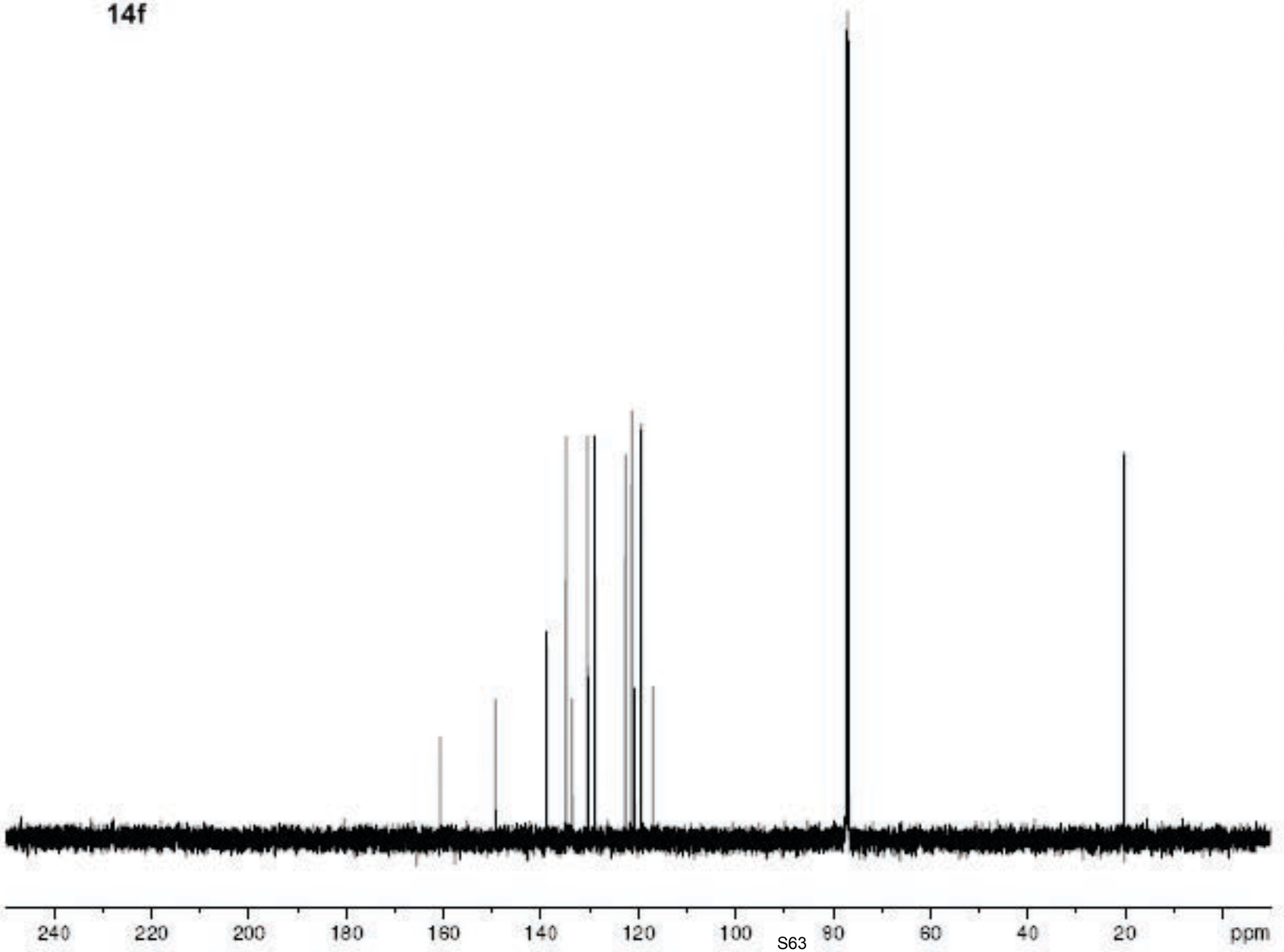
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

default carbon parameters (proton decoupled)



14f

160.693
149.408
138.860
134.880
133.652
130.556
130.351
128.979
122.701
121.419
120.829
119.503
116.988
77.252
76.998
76.743
20.237



Current Data Parameters
NAME DAA-XIII-15-1
EXPNO 2
PROCNO 1

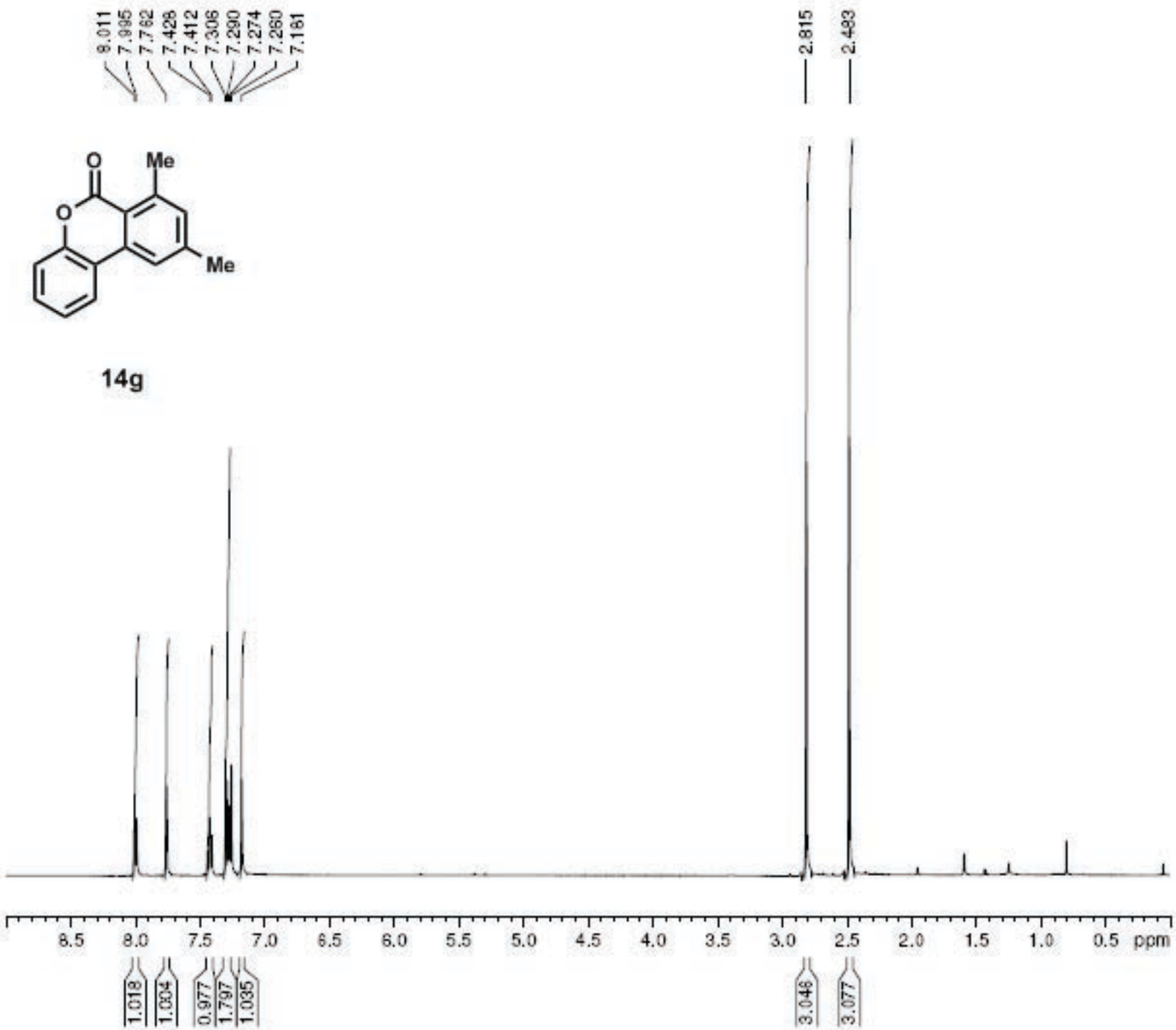
F2 - Acquisition Parameters
Date_ 20081013
Time 13.15
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.739 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.9080898 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters

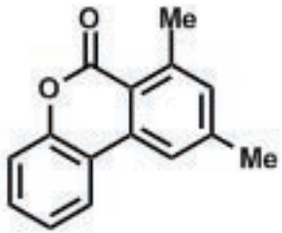


Current Data Parameters
NAME DAA-XIII-17-4
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20081015
Time 10.52
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 295.1 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

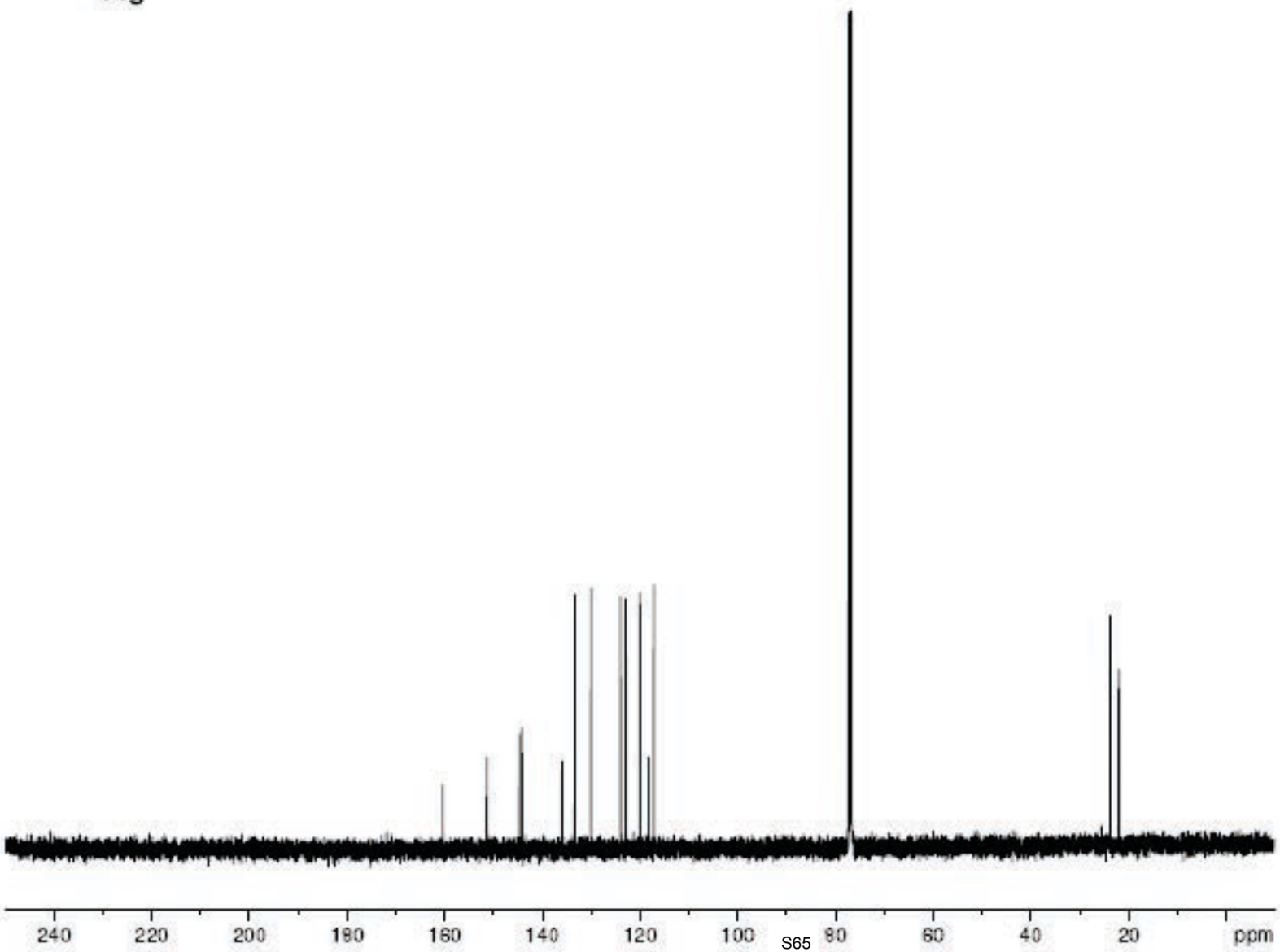
F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



14g

default carbon parameters (proton decoupled)

160.466
151.407
144.719
144.191
136.021
133.357
130.042
124.031
122.935
120.007
118.273
117.235
117.209
77.254
77.030
76.746
23.701
21.949



Current Data Parameters
NAME DAA-XIII-17-4
EXPNO 2
PROCNO 1

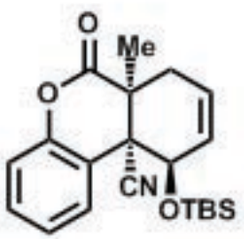
F2 - Acquisition Parameters
Date_ 20081015
Time 11.00
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.739 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 296.0 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.8080863 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



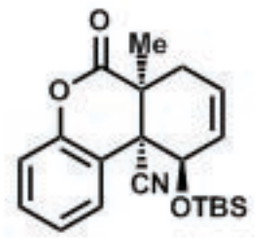
16

Current Data Parameters
NAME DAA-VI-105-1
EXPNO 1
PROCNO 1

F2 - Acquisition Parameters
Date_ 20060127
Time 1.36
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 114
DW 50.000 usec
DE 6.00 usec
TE 294.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 - Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



16

default carbon parameters (proton decoupled)

168.920
151.469
130.328
128.685
127.089
124.846
124.786
119.011
117.860
116.653
77.254
76.999
76.745
70.131
50.148
38.719
31.159
25.596
25.225
23.963
17.617
-5.184
-6.148

Current Data Paramete
NAME DAA-VI-89
EXPNO 2
PROCNO 1

F2 – Acquisition Param
Date_ 20060103
Time 19.56
INSTRUM avance5
PROBHD 5 mm bb-Z
PULPROG zgdc3
TD 65536
SOLVENT CDCl
NS 64
DS 0
SWH 32679.738
FIDRES 0.498653
AQ 1.0027661 s
RG 2896.3
DW 15.300 us
DE 6.00 usec
TE 294.8 K
D1 2.00000000 s
d11 0.03000000 s
MCREST 0.000000
MCWRK 0.015000

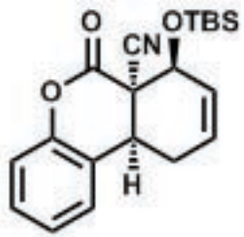
======== CHANNEL f
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.823193

======== CHANNEL f
CPDPRG2 waltz1
NUC2 1H
PCPD2 100.00 u
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.332001

F2 – Processing param
SI 65536
SF 125.9080883
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

0 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

default proton parameters



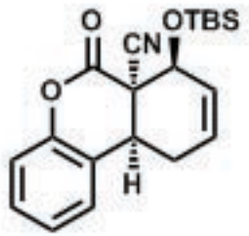
17

Current Data Parameters
NAME DAA-XII-107-2
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080805
Time 18.03
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2769001 sec
RG 114
DW 50.000 usec
DE 6.00 usec
TE 296.7 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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default carbon parameters (proton decoupled)

162.825
151.960
128.779
126.107
126.024
125.001
124.774
120.911
117.006
116.772
77.253
76.999
76.745
67.708
49.257
33.001
25.700
25.348
22.302
17.662
-5.000
-5.188

240 220 200 180 160 140 120 100 80 60 40 20 ppm

Current Data Parameters
NAME DAA-XII-107-2
EXPNO 2
PROCNO 1

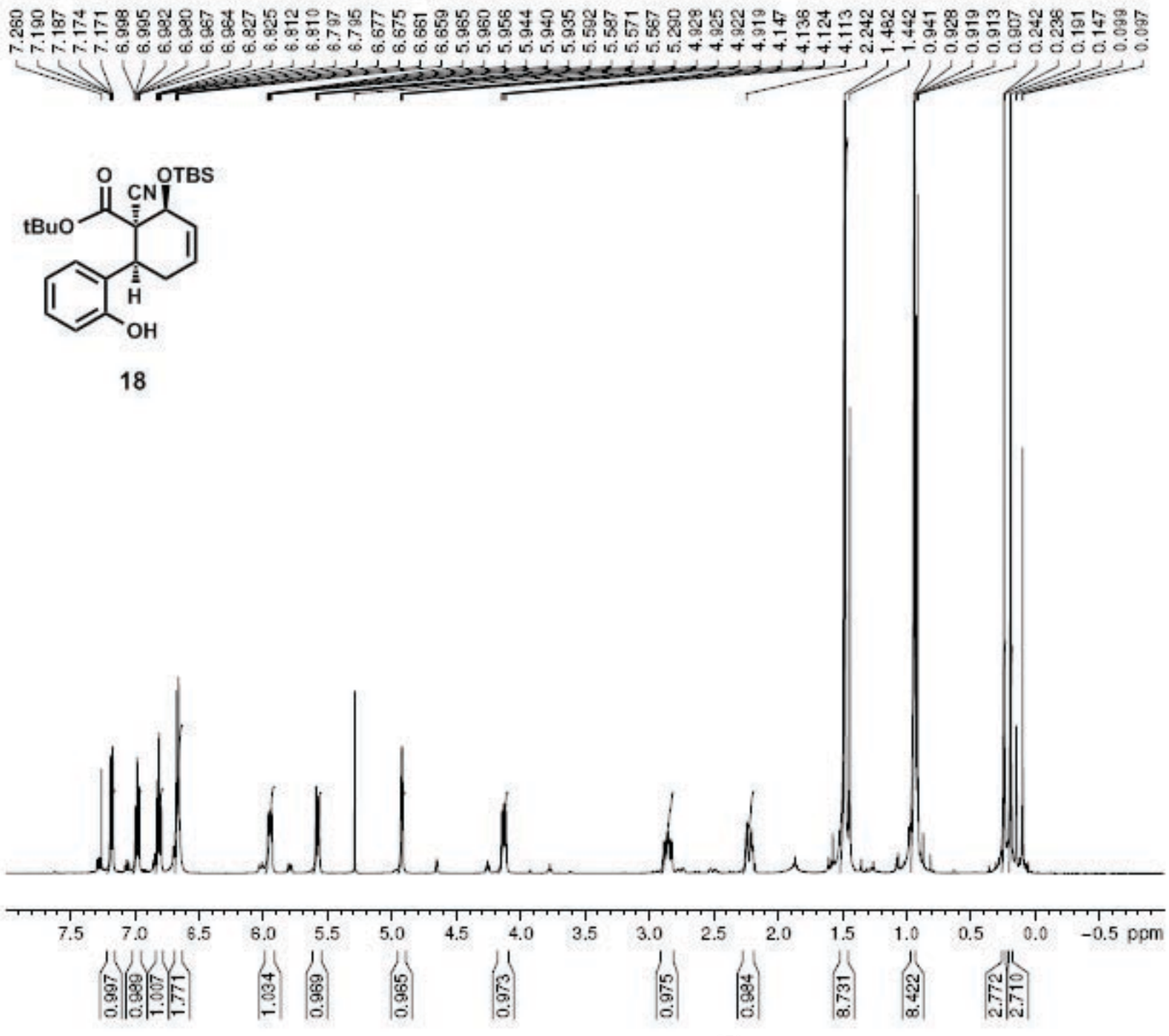
F2 – Acquisition Parameters
Date_ 20080805
Time 18.09
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498853 Hz
AQ 1.0027661 sec
RG 4096
DW 15.300 usec
DE 6.00 usec
TE 297.3 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 ====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 ====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 – Processing parameters
SI 65536
SF 125.9080859 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters

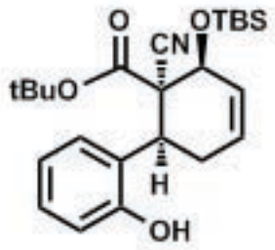


Current Data Parameters
NAME DAA-XII-197-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080801
Time 16.01
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152589 Hz
AQ 3.2769001 sec
RG 40.3
DW 50.000 usec
DE 6.00 usec
TE 296.5 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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default carbon parameters (proton decoupled)

163.836
154.213
128.804
128.734
127.961
126.676
124.977
120.307
119.838
116.069
83.749
77.253
76.999
76.745
74.243
55.630
36.592
29.690
27.883
27.716
25.703
25.614
18.067
-4.690

Current Data Parameters
NAME DAA-XII-197-1
EXPNO 2
PROCNO 1

F2 - Acquisition Parameters
Date_ 20080801
Time 16.06
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 1448.2
DW 15.300 usec
DE 6.00 usec
TE 297.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

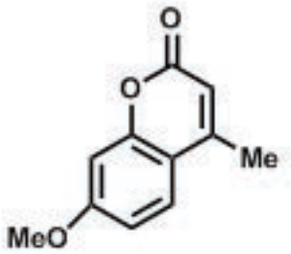
======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 - Processing parameters
SI 65536
SF 125.9080868 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

240 220 200 180 160 140 120 100 80 60 40 20 ppm

Default proton parameters



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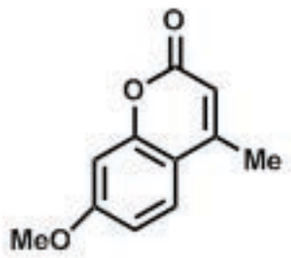
Current Data Parameters
NAME DAA-XII-243-1
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080915
Time 11.28
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2768500 sec
RG 1024
DW 50.000 usec
DE 71.43 usec
TE 300.0 K
D1 2.00000000 sec
P1 11.00 usec
SFO1 500.1330008 MHz
NUCLEUS 1H

F2 – Processing parameters
SI 32768
SF 500.1300244 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



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Default parameters for C–13 with proton decoupling

162.608
161.237
155.263
152.517
125.487
113.534
112.225
111.913
100.806
77.256
77.003
76.749
55.704
18.640

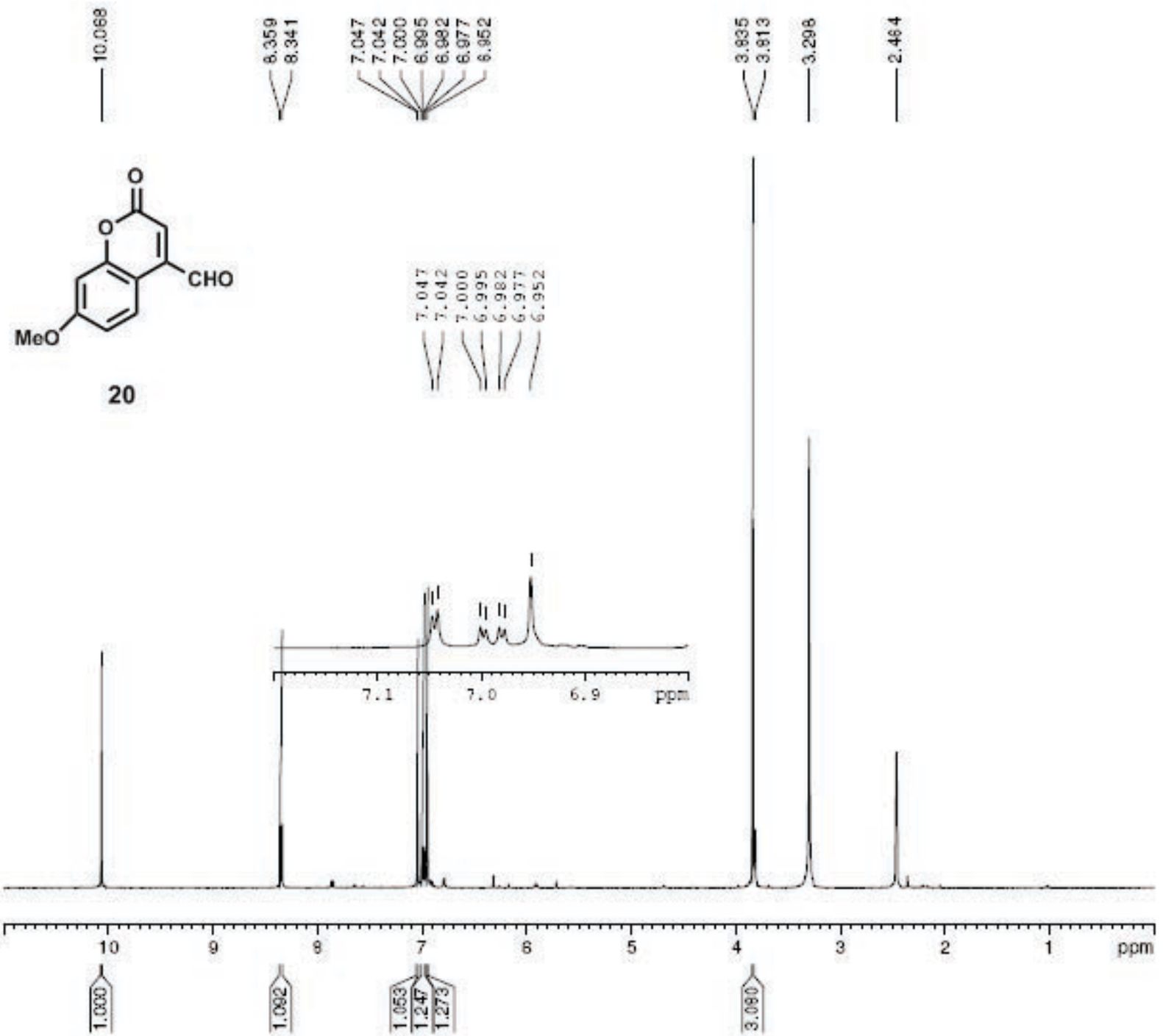
230 220 210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

Current Data Parameters
NAME DAA–XII–243–1
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080915
Time 11.32
INSTRUM arx500
PROBHD 5 mm broadban
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 64
DS 0
SWH 35714.285 Hz
FIDRES 0.544957 Hz
AQ 0.9175540 sec
RG 16384
DW 14.000 usec
DE 20.00 usec
TE 300.0 K
D12 0.0000200 sec
DL5 17.70 dB
CPDPRG waltz16
P31 100.00 usec
D1 2.00000000 sec
P1 6.80 usec
SFO1 125.7728999 MHz
NUCLEUS 13C
D11 0.0300000 sec

F2 – Processing parameters
SI 32768
SF 125.7577975 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

default proton parameters



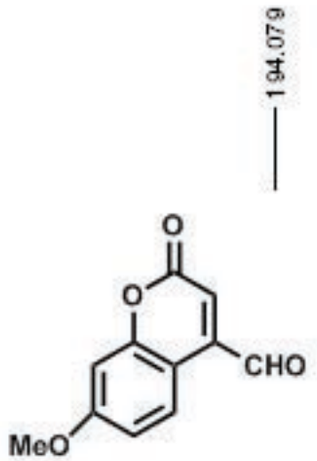
Current Data Parameters
NAME DAA-XII-247-2
EXPNO 1
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080919
Time 13.59
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 0
SWH 10000.000 Hz
FIDRES 0.152588 Hz
AQ 3.2769001 sec
RG 181
DW 50.000 usec
DE 6.00 usec
TE 295.6 K
D1 2.00000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 1H
P1 12.00 usec
PL1 0.00 dB
SFO1 500.3330020 MHz

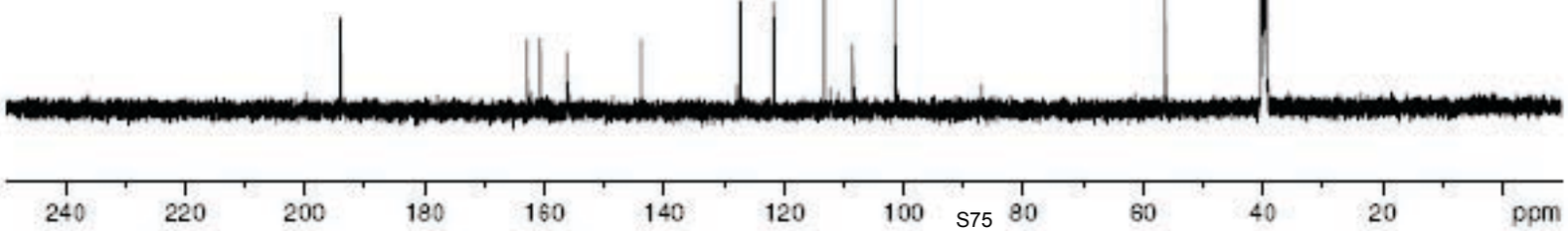
F2 – Processing parameters
SI 32768
SF 500.3300220 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

default carbon parameters (proton decoupled)



20

194.079
162.925
160.756
156.248
143.890
127.258
121.660
113.220
108.544
101.444
56.314
40.326
40.156
39.991
39.824
39.657
39.491
39.324



Current Data Parameters
NAME DAA-XII-247-3
EXPNO 2
PROCNO 1

F2 – Acquisition Parameters
Date_ 20080919
Time 14.47
INSTRUM avance500
PROBHD 5 mm bb-Z Z800
PULPROG zgdc30
TD 65536
SOLVENT CDCl3
NS 128
DS 0
SWH 32679.738 Hz
FIDRES 0.498653 Hz
AQ 1.0027661 sec
RG 2298.8
DW 15.300 usec
DE 6.00 usec
TE 296.1 K
D1 2.00000000 sec
d11 0.03000000 sec
MCREST 0.00000000 sec
MCWRK 0.01500000 sec

======== CHANNEL f1 =====
NUC1 13C
P1 5.25 usec
PL1 0.00 dB
SFO1 125.8231939 MHz

======== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 100.00 usec
PL2 120.00 dB
PL12 16.10 dB
SFO2 500.3320013 MHz

F2 – Processing parameters
SI 65536
SF 125.9080969 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40

References:

1. Kozmin, S.A.; Rawal, V. H. *J. Org. Chem.* **1997**, *62*, 5252.

2. Fringuelli, F.; Piermatti, O.; Pizzo, F. *Synthesis* **2003**, *15*, 2331.

3. (a) Sebille, S.; De Tullio, P.; Becker, B.; Antoine, M.; Boverie, S.; Pirotte, B.; Lebrun, P. *J. Med. Chem*. **2005**, *48*, 614. (b) Jung, J.; Jung, Y.; Park, O. *Synth. Commun.* **2001**, *31*, 1195.

4. (a) Ito, K.; Sawanobori, J. *Synth. Commun.* **1982,** *12*, 665. (b) Schwebel, D.; Soltau, M.; Margaretha, P. *Synthesis* **2001**, *8*, 1111.

5. (*E*)-2-Ethylidenepent-4-enal, the aldehyde used to prepare diene **10b**, is not commercially available and was synthesized in 2 steps according to the procedure of Kieczykowski, G. R.; Schlessinger, R. H.; Sulsky, R. B. *Tetrahedron Lett.* **1976**, 597.

6. We thank Dr. Saeed Khan for his assistance in obtaining the x-ray structure.