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Supporting Information

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Supporting Information

Rh(I)-Catalyzed Two-Component [(5+2)+1] Cycloaddition Approach toward [5-8-5] Ring Systems

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Contents

1. General Methods of Synthesis

Air and moisture sensitive reactions were carried out in oven-dried glassware sealed with rubber septa under a positive pressure of dry CO mixed gas or nitrogen from a balloon, unless otherwise indicated. Similarly sensitive liquids and solutions were transferred via syringe. Reactions were stirred using Teflon-coated magnetic stir bars. Elevated temperatures were maintained using thermostat-controlled silicone oil baths. Organic solutions were concentrated using a Büchi rotary evaporator with a desktop vacuum pump. Tetrahydrofuran and diethyl ether were distilled from sodium and benzophenone prior to use. Dioxane (extra dry, water < 50 ppm), $[Rh(CO)_2Cl]_2$ was commercially available and used as received. Analytical TLC was performed with 0.25 mm silica gel 60F plates with a 254 nm fluorescent indicator. The TLC plates were visualized by ultraviolet light and treatment with acidic *p*-anisaldehyde stain followed by gentle heating. Purification of products was accomplished by flash chromatography on silica gel and the purified compounds show a single spot by analytical TLC.

NMR spectra were measured on a Varian INOVA 600 (^{1}H at 600 MHz, ^{13}C at 150 MHz) magnetic resonance spectrometer. Data for ^{1}H-NMR spectra are reported as follows: chemical shift (ppm: s = singlet, d = doublet, t = triplet, q = quartet, dd = doublet of doublets, td = triplet of doublets, ddd = doublet of doublet of doublets, tdd = triplet of doublet of doublets, m = multiplet), coupling constant (Hz), and integration. Data for ^{13}C-NMR are reported in terms of chemical shift (ppm) relative to residual solvent peak. Infrared spectra were recorded on an AVATAR 330 Fourier transform spectrometer (FT-IR) and are reported in wavenumbers (cm^{-1}). Mass spectra (MS) and high-resolution mass spectra (HRMS) were recorded on a VG-ZAB-HS mass spectrometer (EI, 70 eV).

Abbreviations:

THF = tetrehydrofuran

PE = petroleum ether

EA = ethyl acetate

DCE = 1,2-dichloroethane

PCC: Pyridinium Chlorochromate

PDC: Pyridinium Dichromate

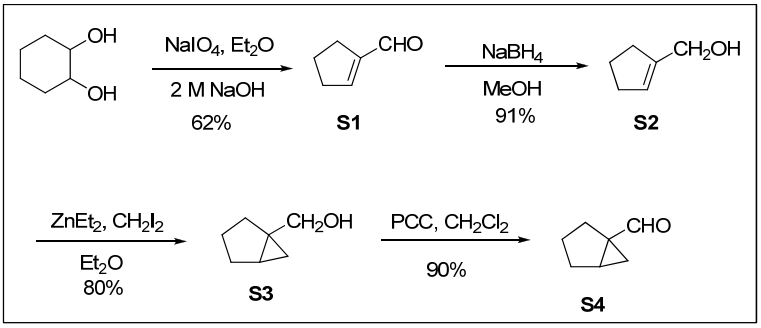
DIBAL-H: Diisobutylaluminum Hydride

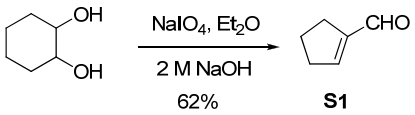
DEAD = diethyl azodicarboxylate

m.p. = melting point

2. General Procedure for the Preparation of Ene-VCP Substrates

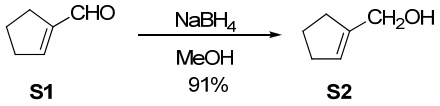
(1) Procedure for the Preparation of Bicyclo[3.1.0]hexane-1-carboaldehyde (S4)





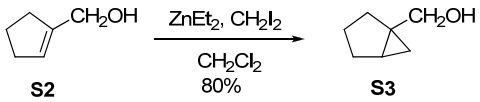
Cyclopent-1-enecarboaldehyde (S1)[1]

To a solution of sodium periodate (20.3 g, 94.8 mmol) in H_2O (150 mL) at 0 °C was added 1,2-diol-cyclohexane (10.0 g, 86.2 mmol) dropwise over 10 min. After stirring for 10 min, a solution of Et_2O (80 mL) and 2 N NaOH (40 mL) was added consecutively. Then the reaction was stirred for 1 h at room temperature. The mixture was extracted with Et_2O (40 mL x 3). After washing with saturated water and brine, the combined organic layer was dried over $MgSO_4$ and concentrated. The residue was purified by flash column chromatography to afford 4.7 g (62%) **S1** as a colorless oil.



Cyclopentenylmethanol (S2)[2]

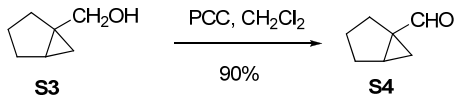
A solution of 5.17 g (53.8 mmol) **S1** dissolved in 50 mL of methanol was cooled to 0°C. Then $NaBH_4$ (0.6 g, 16 mmol) was added in small portion to the above prepared solution over 10 minutes. After 2 h, the reaction solution was evaporated to about one-half volume under reduced pressure. The reaction was then quenched with water and the resulting aqueous phase was extracted with ether and washed with brine. The combined ether phase was dried over anhydrous Na_2SO_4. After removal of the solvent, the residue was purified by flash column chromatography to give 4.8 g (91%) **S2** as a colorless liquid.



Bicyclo[3.1.0]hexane-1-methanol (S3)[3]

Diethyl zinc solution (19.2 mL, 1.5 M in hexane, 28.8 mmol) and CH_2I_2 (4.6 mL, 57.4 mmol) were sequentially added to a solution of **S2** (2.8 g, 28.7 mmol) in anhydrous CH_2Cl_2 (40 mL) at 0 °C. The reaction mixture gradually became a white suspension while stirred at 0 °C. The reaction was monitored by TLC. After 12 h, the reaction was quenched with saturated NH_4Cl. The resulting mixture

was extracted with ether and the combined organic phase was dried over $MgSO_4$ and concentrated. The residue was purified by flash column chromatography to afford 2.5 g (80%) **S3** as a colorless oil.

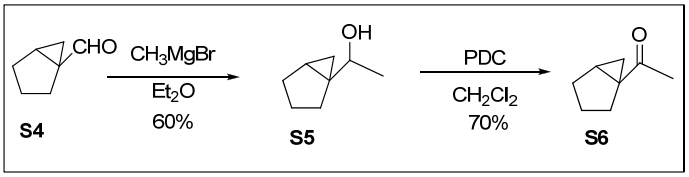


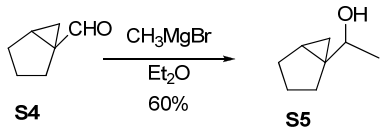
Bicyclo[3.1.0]hexane-1-carboaldehyde (S4)

SiO_2 (6.9 g) was added to a solution of 1.8 g (16.0 mmol) **S3** in 100 mL of CH_2Cl_2. Two equivalents of PCC (6.9 g, 32.0mmol) were added dropwise to the above solution over 10 minutes. The resulting mixture was stirred at room temperature for 2 h. Then the reaction mixture was filtered and the precipitate was washed with excess Et_2O. The organic layers were collected and dried over $MgSO_4$. After concentrated, the residue was purified by flash column chromatography to afford the desired product **S4** 1.58 g as a colorless oil in 90% yield.

^{1}H-NMR (600 MHz, $CDCl_3$): δ 8.99 (s, 1H), 2.18-2.13 (m, 1H), 1.97-1.94 (m, 1H), 1.84-1.73 (m, 4H), 1.37-1.28 (m, 2H), 1.1 (t, J = 6.0 Hz, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 200.7, 42.12, 28.57, 26.26, 24.54, 20.84, 15.39. IR (FT-IR): ν = 2941, 2868, 1684, 1450, 1288, 1227, 1192, 1153, 1040, 958, 930 cm^{-1}. MS (EI): m/z (%) = 110 (M^+, 100), 95 (53), 81 (100), 67 (65), 41 (100). HRMS calcd for $C_7H_{10}O$: 110.0732. Found: 110.0728.

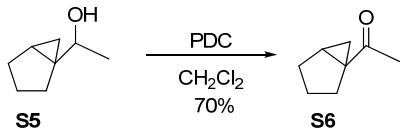
(2) Procedure for the Preparation of 1-(Bicyclo[3.1.0]hexan-1-yl)ethanone (S6)





1-(Bicyclo[3.1.0]hexan-1-yl)ethanol (S5)[4]

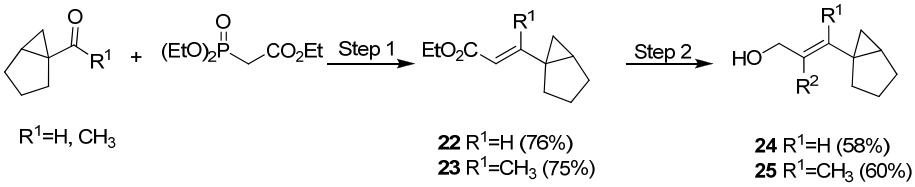
Methylmagnesium bromide (15.0 mL, 3 M, 45.0 mmol) was dissolved in 270 mL anhydrous dimethyl ether under N_2, and then cooled to 0 ^{o}C. **S4** (45.0 mmol, in 10 mL Et_2O) was added slowly to the above solution. The solution was then stirred under 0 ^{o}C for 30 minutes before it was poured into the mixture of 100 g ice and 100 mL 1 M H_2SO_4 (aq). After extracted with Et_2O, washed with water, and brine, dried over $MgSO_4$, and concentrated in vacuo, the crude mixture was purified by flash column chromotography to afford 3.4 g (60 %) **S5** as a light yellow liquid.



1-(Bicyclo[3.1.0]hexan-1-yl)ethanone (S6)[5]

1-(Bicyclo[3.1.0]hexan-1-yl)ethanol **S5** (3.4 g, 27.0mmol) was dissolved in 100 mL anhydrous CH_2Cl_2 and cooled to 0 ^oC. Then PDC (11.1 g, 30 mmol) was added in batches and the resulting solution was stirred for 10 h at room temperature. The product mixture was filtered through a short silica gel column. Then the filtrate was concentrated to get the product with 70% yield (2.7 g) as a light yellow liquid.

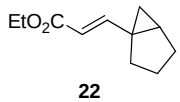
(3) General Procedure for the Preparation of Vinyl Cyclopropyl Alcohols



Step 1: To a flask containing NaH (1.2 equiv.) and THF at 0 °C was added triethyl phosphonoacetate (1.2 equiv.). After stirring at room temperature for 30 min, the corresponding cyclopropyl carbonyl compound (1.0 equiv.) was added dropwise and the reaction was allowed to stir overnight. After quenching with brine, extracting with Et_2O, and drying over $MgSO_4$, concentration of the organic phase in vacuo gave a crude oil that was further purified by flash chromatography (petroleum ether/EtOAc = 9:1). **22** or **23** was obtained as a clear, colorless oil.

Step 2: To a Schlenk flask charged with ester **22** or **23 (**1.0 equiv.) in THF at -78 °C was added DIBAL-H (1 M in toluene, 2.2 equiv.) dropwise. The reaction was warmed to room temperature overnight and was quenched with ethylacetate and aqueous potassium tartrate tetrahydrate. Stirring was continued until the solution was clear. Extracted with Et_2O, washed with brine, dried over $MgSO_4$, evaporation and purification by flash column chromatography (petroleum ether/EtOAc = 4:1) provided alcohol **24** or **25** as a clear, colorless oil.

Physical Data

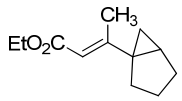


22

(*E*)-Ethyl 3-(bicyclo[3.1.0]hexan-1-yl)acrylate (22)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 6.80 (d, J = 15.6 Hz, 1H), 5.79 (d, J = 15.6 Hz, 1H), 4.18 (q, J = 7.2 Hz, 2H), 1.87-1.84 (m, 1H), 1.78-1.75 (m, 3H), 1.69-1.67(m, 1H), 1.56-1.54 (m, 1H), 1.28 (t, J = 7.2 Hz, 3H), 0.95 (t, J = 5.4 Hz, 1H), 0.87-0.84 (m, 2H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 167.1, 155.2, 116.2, 59.9, 31.8, 29.1, 28.1, 27.0, 20.7, 16.9, 14.3. IR (FT-IR): ν = 2934, 2862, 1638, 1446, 1308, 1275, 1209, 1161, 1095, 1044, 983 cm^{-1}. MS (EI): m/z (%) = 180 (M^+, 35), 151 (33), 79 (47), 43 (100). HRMS

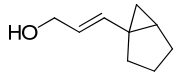
calcd for $C_{11}H_{16}O_2$: 180.1150. Found: 180.1148.



23

(*E*)-Ethyl 3-(bicyclo[3.1.0]hexan-1-yl)but-2-enoate (23)

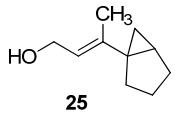
^{1}H-NMR (600 MHz, $CDCl_3$): δ 6.74 (s, 1H), 4.17 (q, J = 7.2 Hz, 2H), 1.98-1.92 (m, 1H), 1.95 (s, 3H), 1.88 (d, J = 1.8 Hz, 1H), 1.84-1.78 (m, 1H), 1.73 (dd, J = 12.0 and 7.8 Hz, 1H), 1.68-1.63 (m, 1H), 1.40-1.38 (m,1H), 1.28 (t, J = 5.4 Hz, 3H), 0.88-0.84 (m, 1H), 0.78 (t, J = 4.8 Hz, 1H), 0.67-0.64 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 168.8, 145.4, 127.9, 60.3, 31.2, 28.8, 27.5, 27.0, 21.3, 14.7, 14.3, 13.2. IR (FT-IR): ν = 2956, 2924, 1708, 1461, 1261, 1111, 1090, 745 cm^{-1}. MS (EI): m/z (%) = 194 (M^+, 95), 165 (77), 147 (63), 121(100), 79 (75). HRMS calcd for $C_{12}H_{18}O_2$: 194.1307. Found: 194.1306.



24

(*E*)-3-(Bicyclo[3.1.0]hexan-1-yl)prop-2-en-1-ol (24)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.65-5.57 (m, 2H), 4.10 (d, J = 5.4 Hz, 2H), 1.86-1.81 (m, 1H), 1.77-1.69 (m, 3H), 1.66-1.61 (m, 1H), 1.53 (bs, 1H), 1.27-1.25 (m, 1H), 1.23-1.19 (m, 1H), 0.69 (t, J = 4.8 Hz, 1H), 0.59-0.57 (m,1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 138.8, 124.6, 63.8, 30.2, 29.2, 27.2, 26.6, 20.8, 14.5. IR (FT-IR): ν = 3327, 2932, 2860, 1664, 1451, 1040, 1006, 966 cm^{-1}. MS (EI): m/z (%) = 138 (M^+, 8.0), 120 (15), 91 (52), 79 (100), 41 (66). HRMS calcd for $C_9H_{14}O$: 138.1045. Found: 138.1044.

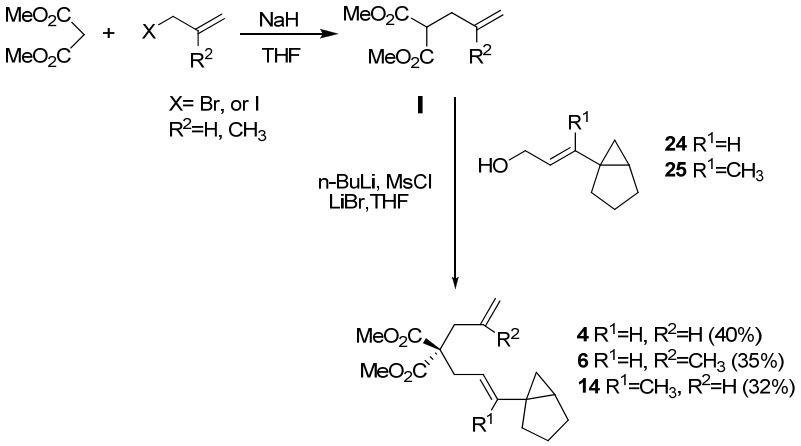


25

(*E*)-3-(Bicyclo[3.1.0]hexan-1-yl)but-2-en-1-ol (25)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.52-5.49 (m, 1H), 4.18 (d, J = 6.6 Hz, 2H), 1.80-1.73 (m, 4H), 1.67 (bs, 1H), 1.60 (s, 3H), 1.39-1.37 (m, 1H), 1.27-1.22 (m, 2H), 0.62 (dd, J = 8.4 and 4.8 Hz , 1H), 0.54 (t, J = 4.2 Hz, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 142.2, 122.0, 59.6, 34.9, 30.8, 27.4, 23.8, 20.9, 14.3, 12.5. IR (FT-IR): ν = 3330, 2930, 2859, 1649, 1448, 1128, 997, 933 cm^{-1}. MS (EI): m/z (%) = 152 (M^+, 11), 123 (47), 93 (75), 79 (73), 43 (66). HRMS calcd for $C_{10}H_{16}O$: 152.1201. Found: 152.1200.

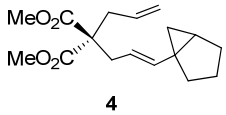
(4) General Procedure for the Preparation of Geminal Diester-Tether Substrates 4, 6, and 14



Step 1: To a flask charged with NaH (1.8 equiv.) and THF at 0 °C was added dimethyl malonate (1.8 equiv.) dropwise. After stirred for 30 min, allyl iodide or allyl bromide (1.0 equiv.) was added and the reaction mixture was stirred at room temperature overnight. Quenching with brine, extracting with Et_2O, drying over $MgSO_4$, evaporation, and purification by flash column chromatography (petroleum ether/EtOAc = 9:1) provided **I** as a clear colorless oil.

Step 2: (a) To a 250 mL flask containing alcohol **24** or **25** (1.0 equiv.) in THF at -78 °C was added *n*-BuLi (1.6 M in hexane, 1.2 equiv.), followed 10 min later by methane sulfonyl chloride (1.2 equiv.), and then immediately by lithium bromide (4.5 equiv.) in one portion. The reaction mixture was stirred for 30 min. (b) To a second flask charged with NaH (1.6 equiv.) in THF was added **I** (1.6 equiv.) dropwise over 15 min. After stirred for 30 min at room temperature, the solution was cooled to -78 °C and the solution of the first flask was transferred in via cannula. The reaction mixture was stirred for 2 h at -78 °C, then allowed to warm to room temperature. Quenching with water, extracting with Et_2O, drying over $MgSO_4$, evaporation and purification by flash column chromatography (petroleum ether/EtOAc = 9:1) provided geminal diester derivatives **4**, **6**, and **14** as clear colorless oils.

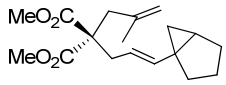
Physical Data



4

(*E*)-Dimethyl 2-allyl-2-(3-(bicyclo[3.1.0]hexan-1-yl)allyl)malonate (4)

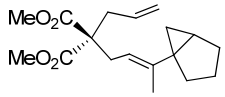
^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.69-5.61 (m 1H), 5.42 (d, J = 15.6 Hz, 1H), 5.21-5.17 (m, 1H), 5.12-5.07 (m, 2H), 3.71(s, 6H), 2.65-2.58 (m, 4H), 1.76-1.75 (m ,2H), 1.68-1.65 (m, 2H), 1.62-1.59 (m, 2H), 1.21-1.16 (m, 1H), 0.62 (t, J = 4.8 Hz, 1H), 0.51-0.49 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 171.5, 140.6, 132.7, 119.21, 119.17, 58.3, 52.5, 52.4, 37.2, 36.1, 30.7, 29.7, 27.4, 26.5, 21.1, 14.4. IR (FT-IR): ν = 2952, 2860, 1734, 1436, 1208, 994, 920, 858 cm^{-1}. MS (EI): m/z (%) = 292 (M^+, 4.0), 219 (47), 120 (86), 91 (82), 79 (100). HRMS calcd for $C_{17}H_{24}O_4$: 292.1675. Found: 292.1676.



6

(*E*)-Dimethyl 2-(3-(bicyclo[3.1.0]hexan-1-yl)allyl)-2-(2-methylallyl)malonate (6)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.40 (d, J = 15.3 Hz, 1H), 5.25-5.20 (m, 1H), 4.86-4.85 (m, 1H), 4.73(t, J = 0.6 Hz, 1H), 3.69 (s, 6H), 2.69 (s, 2H), 2.61 (d, J = 7.2 Hz, 2H), 1.76-1.70 (m, 2H), 1.68-1.66 (m, 2H), 1.67 (s, 3H), 1.63-1.58 (m, 2H), 1.19-1.15 (m, 1H), 0.61 (t, J = 4.8 Hz, 1H), 0.51-0.48 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 171.5, 140.4, 139.9, 119.3, 115.4, 57.7, 51.9, 40.2, 35.8, 30.3, 29.4, 27.1, 26.1, 23.0, 20.7, 14.0. IR (FT-IR): ν = 2950, 2859, 1735, 1436, 1272, 1199, 1176, 968 cm^{-1}. MS (EI): m/z (%) = 306 (M^+, 5.0), 187 (36), 165 (34), 151(100), 120 (68), 79 (78). HRMS calcd for $C_{18}H_{26}O_4$: 306.1831. Found: 306.1845.

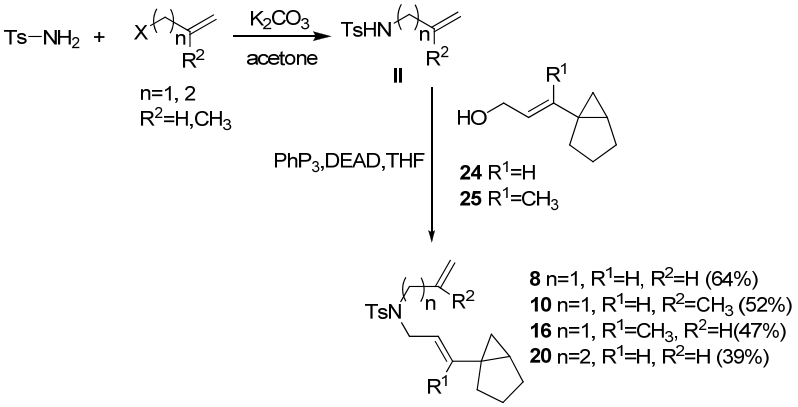


14

(*E*)-Dimethyl 2-allyl-2-(3-(bicyclo[3.1.0]hexan-1-yl)but-2-enyl)malonate (14)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.69-5.62 (m, 1H), 5.08-5.05 (m, 3H), 3.70 (s, 6H), 2.63-2.61 (m, 4H), 1.81-1.64 (m, 5H), 1.56(s, 3H), 1.29- 1.23 (m, 2H), 0.51-0.47 (m, 2H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 171.5, 141.6, 132.6, 118.9, 116.3, 66.2, 58.0, 52.2, 37.0, 35.4, 31.2, 31.1, 27.6, 22.3, 12.5. IR (FT-IR): ν = 2951, 2859, 1735, 1436, 1269, 1139, 920, 862 cm^{-1}. MS (EI): m/z (%) = 306 (M^+, 6.0), 233 (20), 147 (30), 134 (100), 119 (52), 79 (46). HRMS calcd for $C_{18}H_{26}O_4$: 306.1831. Found: 306.1830.

(5) General Procedure for the Preparation of Tosylamide-Tether Substrates 8, 10, 16, and 20

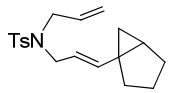


Step 1: To a flask was added $TsNH_2$ (1.1 equiv.), K_2CO_3 (1.5 equiv.) and acetone at 0 °C. After stirring for 30 min, allyl bromide or 4-bromobut-1-ene (1.0 equiv.) was added and the reaction mixture was refluxed for 7 h. Quenching with brine, extraction with Et_2O, drying over $MgSO_4$, evaporation, and purification by flash column chromatography (petroleum ether/EtOAc = 2:1) provided **II** as a white solid.

Step 2: To a solution of alcohol **24** or **25** (1.0 equiv.), **II** (1.2 equiv.), and PPh_3 (2.2 equiv.) in THF was

added DEAD (2.2 equiv.) at room temperature and the reaction mixture was stirred for 20 h at room temperature. The resulting mixture was diluted with Et_2O, washed with water and brine, and dried over Na_2SO_4. After removal of the solvent, the residue was purified by flash column chromatography with Et_3N-impregnated silica gel (petroleum ether/EtOAc = 9:1) to give **8**, **10**, **16,** and **20** as colorless oils.

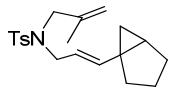
Physical Data



8

(*E*)-N-Allyl-N-(3-(bicyclo[3.1.0]hexan-1-yl)allyl)-4-methylbenzenesulfonamide (8)

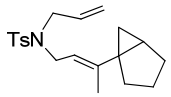
^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.69 (d, J = 8.4 Hz, 2H), 7.28 (d, J = 7.8 Hz, 2H), 5.66-5.59 (m, 1H), 5.39 (d, J = 10.2 Hz, 1H), 5.16-5.11 (m, 3H), 3.80-3.75 (m, 4H), 2.42 (s, 3H), 1.75-1.58 (m, 6H), 0.88-0.84 (m, 1H), 0.64 (t, J = 4.8 Hz ,1H), 0.48-0.47 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 143.0, 141.0, 133.0, 129.5, 127.3, 119.4, 118.6, 49.0, 48.8, 30.2, 29.1, 27.2, 26.5, 21.5, 20.8, 14.4. IR (FT-IR): ν = 2917, 2860, 1340, 1157, 1090, 1032, 952, 814, 753 cm^{-1}. MS (EI): m/z (%) = 331 (M^+, 2.0), 250 (18), 224 (38), 155 (48), 91 (100), 41 (84). HRMS calcd for $C_{19}H_{25}NO_2S$: 331.1606. Found: 331.1614.



10

(*E*)-N-(3-(Bicyclo[3.1.0]hexan-1-yl)allyl)-N-(2-methylallyl)-4-methylbenzenesulfonamide (10)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.69 (d, J = 7.8 Hz, 2H), 7.28 (d, J = 7.8 Hz, 2H), 5.57-5.50 (m, 1H), 5.34 (d, J = 15.6 Hz, 1H), 5.06-4.84 (m, 2H), 3.74-3.67 (m, 4H), 2.42 (s, 3H), 1.70 (s, 3H), 1.69-1.53 (m, 6H), 0.88-0.83 (m, 1H), 0.60 (t, J = 4.8Hz, 1H), 0.45-0.40 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 142.9, 141.2, 140.4, 133.4, 129.2, 127.3, 118.9, 114.2, 52.5, 48.8, 30.2, 29.0, 27.2, 26.4, 21.5, 21.4, 20.8, 14.3. IR (FT-IR): ν = 2927, 2859, 1337, 1157, 1093, 1002, 912, 814, 763 cm^{-1}. MS (EI): m/z (%) = 345 (M^+, 4.0), 264 (16), 238 (38), 190 (46), 91 (100), 55 (100). HRMS calcd for $C_{20}H_{27}NO_2S$: 345.1763. Found: 345.1767.

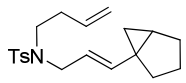


16

(*E*)-N-Allyl-N-(3-(bicyclo[3.1.0]hexan-1-yl)but-2-enyl)-4-methylbenzenesulfonamide (16)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.69 (d, J = 8.4 Hz, 2H), 7.29 (d, J = 7.8 Hz, 2H), 5.69-5.63 (m, 1H), 5.15-5.11 (m, 2H), 5.01-4.98 (m, 1H), 3.83 (d, J = 6.6 Hz, 2H), 3.77 (d, J = 6.0 Hz, 2H), 2.42(s, 3H), 1.70-1.68 (m, 2H), 1.64-1.60 (m, 2H), 1.59-1.58 (m,2H), 1.57 (s, 3H), 0.89-0.83 (m, 2H), 0.47-0.45 (m,1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 142.9, 142.5, 133.3, 129.53, 129.51, 127.2, 118.3, 117.1, 49.4, 44.6, 35.0, 30.8, 27.4, 23.6, 21.5, 20.9, 14.3, 12.3. IR (FT-IR): ν = 2926, 2855, 1339, 1156, 1089, 1037, 922, 814, 756 cm^{-1}. MS (EI): m/z (%) = 345 (M^+, 2.0), 238 (32), 224 (36), 155 (64), 91 (100), 41 (78).

HRMS calcd for $C_{20}H_{27}O_2S$: 345.1763. Found: 345.1771.

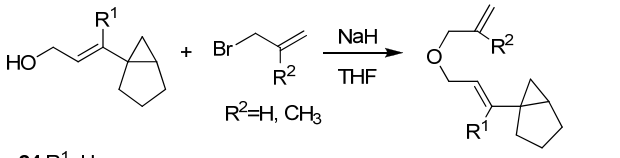


20

(*E*)-N-(3-(Bicyclo[3.1.0]hexan-1-yl)allyl)-N-(but-3-enyl)-4-methylbenzenesulfonamide (20)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.69 (d, J = 8.4 Hz, 2H), 7.28 (d, J = 7.8 Hz, 2H), 5.74-5.68 (m, 1H), 5.44 (d, J = 18 Hz, 1H), 5.20-5.15 (m, 1H), 5.05-5.01 (m, 2H), 3.78-3.74 (m, 2H), 3.18-3.15 (m, 2H), 2.42 (s, 3H), 2.27 (q, J = 6.6 Hz, 2H), 1.73-1.58 (m, 6H), 1.19-1.18 (m, 1H), 0.64 (t, J = 5.4Hz, 1H), 0.49 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ. 143.2, 140.9, 137.7, 135.2, 129.7, 127.4, 120.2, 117.1, 50.3, 46.6, 33.3, 30.5, 29.4, 27.4, 26.7, 21.7, 21.0, 14.6. IR (FT-IR): ν = 2930, 2860, 1338, 1156, 1090, 1031, 926, 813, 746 cm^{-1}. MS (EI): m/z (%) = 345 (M^+, 6.0), 304 (12), 190 (14), 155 (20), 121 (100), 91 (68), 79 (58). HRMS calcd for $C_{20}H_{27}O_2S$: 345.1762. Found: 345.1757.

(6) General Procedure for the Preparation of Ether-Tethered Substrates 12 and 18

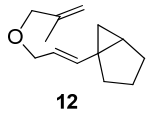


24 R^1=H
25 R^1=CH_3

12 R^1=H, R^2=CH_3 (34%)
18 R^1=CH_3, R^2=H (65%)

To a solution of NaH (1.2 equiv.) in THF was added a solution of alcohol **24** or **25** (1.0 equiv.) in THF at 0 °C and the mixture was refluxed for 2 h. The solution was cooled to room temperature, then allyl bromide (1.2 equiv.) was added and the mixture was refluxed overnight. After being cooled to room temperature, the reaction was quenched with water and extracted with Et_2O. Washed with brine, dried over $MgSO_4$, evaporation to remove the solvent, and purification by flash column chromatography (petroleum ether/EtOAc = 9:1) gave **12** and **18** as colorless oils.

Physical Data

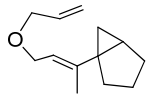


12

(*E*)-1-(3-(2-Methylallyloxy)prop-1-enyl)bicyclo[3.1.0]hexane (12)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.61-5.53 (m, 2H), 4.96-4.95 (m, 1H), 4.89-4.88 (m, 1H), 3.91 (d, J = 5.4 Hz, 2H), 3.88(s, 2H), 1.87-1.82 (m, 1H), 1.74 (s, 3H), 1.72-1.68 (m, 2H), 1.64-1.60 (m, 2H), 1.27-1.20 (m, 2H), 0.68 (t, 1H), 0.59-0.57 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 142.6, 140.1, 122.2, 112.2, 74.1, 71.1, 30.5, 29.4, 27.5, 26.7, 21.1, 19.8, 14.8. IR (FT-IR): ν = 2929, 2859, 1450, 1113, 1077, 971, 891 cm^{-1}. MS (EI): m/z (%) = 192 (M^+, 12), 121 (32), 119 (42), 93 (74), 86 (100), 67

(70), 47 (100). HRMS calcd for $C_{13}H_{20}O$: 192.1514. Found: 192.1520.



18

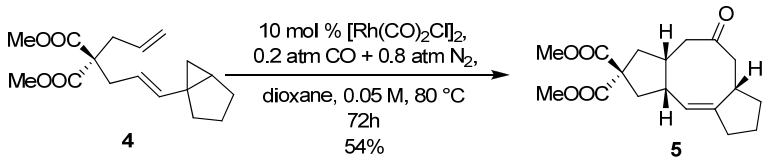
(*E*)-1-(4-(Allyloxy)but-2-en-2-yl)bicyclo[3.1.0]hexane (18)

^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.94 (m, 1H), 5.45 (m, 1H), 5.27 (m, 1H), 5.17 (dd, J = 10.2 and 1.2 Hz, 1H), 4.01 (d, J = 7.2Hz, 2H), 3.96 (d, J = 2.4Hz, 2H), 1.81-1.78 (m, 2H), 1.77-1.69 (m, 2H), 1.62-1.59 (m, 2H), 1.59 (s, 3H), 1.25-1.20 (m, 1H), 0.62-0.60 (m, 1H), 0.26 (t, J = 4.8Hz, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 142.5, 135.1, 119.5, 116.9, 71.1, 66.9, 35.0, 30.8, 27.5, 23.7, 21.0, 14.5, 12.5. IR (FT-IR): ν = 2926, 2860, 1450, 1066, 920, 800 cm^{-1}. MS (EI): m/z (%) = 192 (M^+, 6.0), 134 (22), 121 (30), 91 (38), 81 (48), 91 (68), 41 (100). HRMS calcd for $C_{13}H_{20}O$: 192.1514. Found: 192.1515.

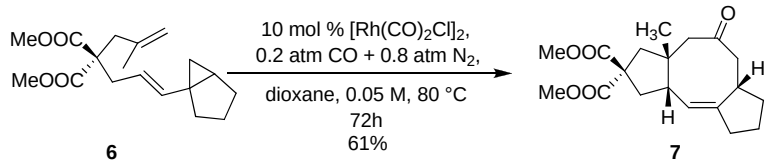
3. General Procedure for the [(5 + 2) + 1] Cycloaddition Reactions

$[Rh(CO)_2Cl]_2$ (10 mol % to the substrate) was charged in a base-washed, oven-dried Schlenk flask under an atmosphere of nitrogen, and then a solution of the ene-VCP substrate in degassed dioxane (0.05 M) was added. The solution was bubbled with the mixed gas of CO and N_2 (0.2 atm CO + 0.8 atm N_2) for 5 min. The reaction mixture was stirred at 80 °C under balloon pressured mixed gas of CO and N_2 (0.2 atm CO + 0.8 atm N_2) until TLC indicated the completion of the reaction (72 h). After cooled to room temperature, the mixture was concentrated and the residue was purified by flash column chromatography with silica gel to afford the cycloaddition product.

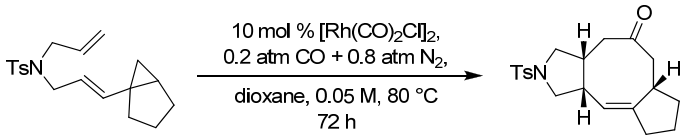
4. Summary of all [(5 + 2) + 1] Cycloaddition Reactions



4 **5**



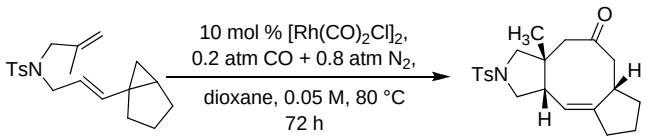
6 **7**



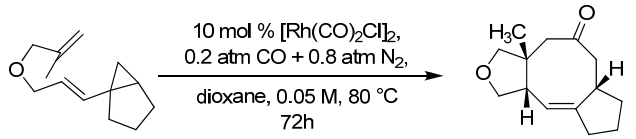
8

72% conversion, 58% yield
(79% based on recovered starting material)

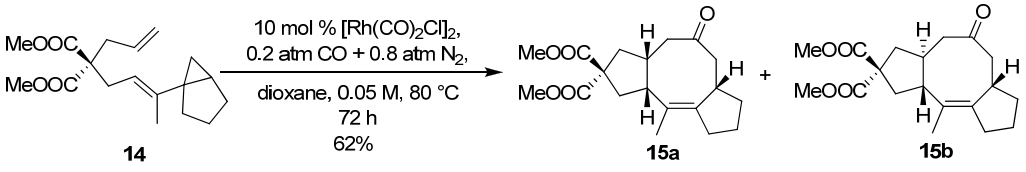
9



10 37% **11**

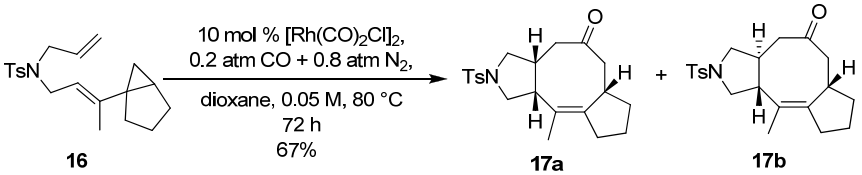


12 44% **13**



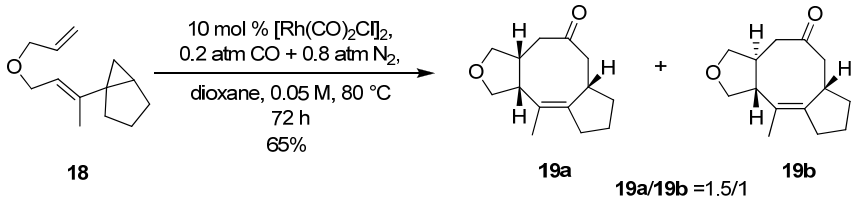
14 **15a** **15b**

15a/15b = 1/6



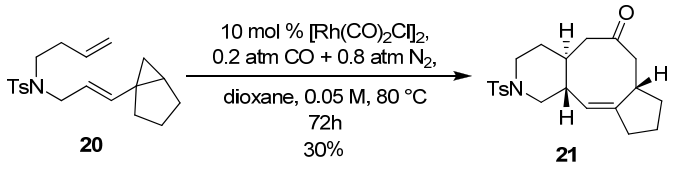
16 **17a** **17b**

17a/17b = 1/1.3



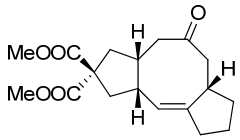
18 **19a** **19b**

19a/**19b** =1.5/1



20 **21**

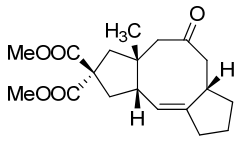
5. Physical Data for Cycloadducts



5

(3R*, 7R*, 11S*)-5,5-Bis(methoxylcarbonyl)tricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (5)

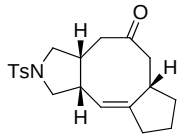
Light yellow oil, 54% yield. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.28-5.24 (m, 1H), 3.74 (s, 3H), 3.73(s, 3H), 2.69-2.58 (m, 2H), 2.52-2.43 (m, 4H), 2.42-2.32 (m, 4H), 2.28-2.19 (m, 2H), 2.05-1.95 (m, 1H), 1.78-1.69 (m, 2H), 1.64-1.56 (m, 1H), 1.49-1.45 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 212.2, 173.1, 172.9, 147.5, 121.8, 59.0, 53.3, 52.82, 52.81, 43.2, 41.4, 41.0, 40.0, 39.5, 37.8, 34.5, 33.2, 24.1. IR (FT-IR): ν = 2916, 2848, 1731, 1434, 1245, 1166, 848 cm^{-1}. MS (EI): m/z (%) = 320 (M^{+}, 38), 302 (55), 203 (90), 91 (100), 79 (94), 41 (58). HRMS calcd for $C_{18}H_{24}O_5$: 320.1624. Found: 320.1621.



7

(3R*, 7R*, 11S*)-5,5-Bis(methoxylcarbonyl)-7-methyltricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (7)

Yellow oil, 61% yield. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.39 (d, J = 1.8 Hz, 1H), 3.73 (s, 3H), 3.71 (s, 3H), 2.61-2.50 (m, 4H), 2.44-2.38 (m, 2H), 2.29-2.18(m, 4H), 2.03 (dd, J = 13.8 and 9.0 Hz, 1H), 1.89-1.84 (m, 1H), 1.69-1.65 (m, 1H), 1.61-1.56 (m, 2H), 1.53-1.50 (m, 1H), 1.20 (s, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 211.6, 173.9, 173.5, 147.4, 128.1, 57.9, 53.56, 53.53, 52.0, 51.2, 47.5, 47.1, 44.7, 41.2, 36.4, 35.3, 34.8, 26.4, 23.4. IR (FT-IR): ν = 2954, 2924, 1732, 1434, 1264, 1198, 1065, 847 cm^{-1}. MS (EI): m/z (%) = 334 (M^{+}, 84), 316 (100), 217 (40), 122 (46), 91 (52), 41(44). HRMS calcd for $C_{19}H_{26}O_5$: 334.1780. Found: 334.1772.

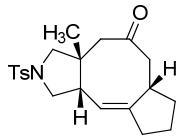


9

(3S*, 7S*, 11S*)-N-Tosyl-5-azatricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (9)

Light yellow oil, 72% conversion, 58% yield (79% based on recovered starting material). ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.72 (d, J = 7.8 Hz 2H), 7.34 (d, J = 8.4 Hz, 2H), 4.96 (dd, J = 8.4 and 1.8 Hz, 1H), 3.41 (d, J = 10.2 Hz, 1H), 3.39 (d, J = 9.6, 1H), 3.22 (dd, J = 10.2 and 4.8 Hz, 1H), 2.82 (t, J = 10.2 Hz, 1H), 2.61-2.47 (m, 3H), 2.45 (s, 3H), 2.42-2.33 (m, 3H), 2.32-2.16 (m, 3H), 1.99-1.96 (m, 1H), 1.73-1.70 (m, 1H), 1.62-1.58 (m, 1H), 1.48-1.47 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 211.9, 149.5 144.3, 130.4, 128.1, 119.7, 54.5, 53.9, 51.8, 41.5, 39.6, 38.7, 35.2, 34.0, 30.4, 30.0, 24.8, 22.2. IR (FT-IR): ν = 2955, 2921, 1700, 1466, 1343, 1162, 1090, 1045, 803 cm^{-1}. MS (EI): m/z (%) = 359 (M^{+}, 24), 301 (90), 204 (78), 146 (72), 91 (100), 42 (86). HRMS calcd for $C_{20}H_{25}NO_3S$: 359.1555. Found:

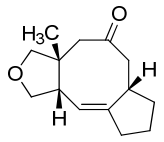
359.1550.



11

(3S*, 7S*, 11S*)-N-Tosyl-7-methyl-5-azatricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (11)

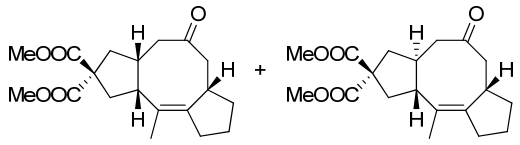
Colorless crystals, 37% yield, m.p.150 °C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.73 (d, J = 7.8 Hz, 2H), 7.34 (d, J = 7.8 Hz, 2H), 4.83 (dd, J = 8.4 and 1.2 Hz, 1H), 3.34 (dd, J = 9.8 and 4.8 Hz, 1H), 3.31 (d, J = 10.2 Hz, 1H), 3.10 (d, J = 9.6 Hz, 1H), 2.91(d, 9.0Hz, 1H), 2.63 (d, J = 11.4 Hz, 1H), 2.57 (m, 1H), 2.45 (s, 3H), 2.40-2.30 (m, 3H), 2.28-2.19 (m,3H), 1.97-1.90 (m, 1H), 1.72-1.67 (m, 1H), 1.60-1.57 (m, 1H), 1.44-1.42 (m, 1H), 0.87 (s, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 210.6, 149.0, 144.2, 134.9, 130.3, 127.9, 121.7, 59.3, 53.69, 53.66, 48.1, 47.5, 43.8, 38.0, 35.0, 33.4, 24.3, 23.9, 22.2. IR (FT-IR): ν = 2957, 2869, 1697, 1384, 1344, 1156, 1091, 1051, 738 cm^{-1}. MS (EI): m/z (%) = 373 (M^+, 30), 315 (60), 218 (78), 160 (94), 91 (76), 42 (100). HRMS calcd for $C_{21}H_{27}NO_3S$: 373.1711. Found: 373.1720. This structure was confirmed by X-ray crystallographic analysis.



13

(3S*, 7S*, 11S*)-7-Methyl-5-oxatricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (13)

Light yellow oil, 44% yield. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 5.38 (dd, J = 7.8 and 1.8 Hz, 1H), 4.13 (q, J = 13.8 Hz, 1H), 3.81 (d, J = 8.4, 1H), 3.52 (d, J = 7.2 Hz, 1H), 3.38 (d, J = 7.8Hz, 1H), 2.71-2.68 (m, 2H), 2.53-2.37 (m, 5H), 2.03 (d, J = 11.4 Hz, 1H), 1.98-1.93 (m, 1H), 1.78-1.72 (m, 1H), 1.65-1.61 (m, 1H), 1.49-1.46 (m, 1H), 1.21 (s, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 210.9, 148.7, 123.5, 79.3, 75.0, 53.4, 48.7, 47.1, 44.7, 38.2, 35.0, 33.2, 24.4, 24.3. IR (FT-IR): ν = 2954, 2873, 1695, 14548, 1055, 882, 754 cm^{-1}. MS (EI): m/z (%) = 220 (M^+, 6.0), 162 (100), 135 (18), 91 (30), 79 (32), 41 (28). HRMS calcd for $C_{14}H_{20}O_2$: 220.1463. Found: 220.1473.



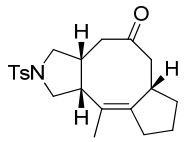
15a (1 : 6) 15b

(3R*, 7S*, 11S*)-2-Methyl-5,5-bis(methoxylcarbonyl)tricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (15b)

Yellow oil, 62% yield. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 3.75 (s, 3H), 3.74 (s, 3H), 3.33-3.29 (m, 1H), 2.71-2.66 (m, 1H), 2.59-2.51 (m, 3H), 2.50-2.46 (m, 1H), 2.45-2.38 (m, 2H), 2.37-2.22 (m, 4H), 1.97-1.88 (m, 1H), 1.83-1.74 (m, 1H), 1.82-1.74 (m, 3H), 1.59 (s, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 211.2, 173.0, 172.9, 142.3, 125.7, 57.7, 52.9, 52.7, 52.6, 48.9, 45.2, 40.0, 39.2, 37.6, 37.4, 32.7, 29.6, 22.9, 15.4. IR (FT-IR): ν = 2954, 2923, 1731, 1435, 1257, 1199, 1066, 864 cm^{-1}. MS (EI): m/z (%) =

334 (M^+, 94), 316 (82), 256 (54), 145 (82), 91 (80), 43 (100). HRMS calcd for $C_{19}H_{26}O_5$: 334.1780. Found: 334.1775.

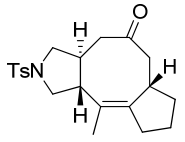
Compound **15a** is the minor product generated in the [(5+2)+1] cycloaddition, which is inseparable from the major product **15b**. Therefore, no characterization data for **15a** was provided.



17a

(3S*, 7S*, 11S*)-2-Methyl-N-tosyl-5-azatricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (17a)

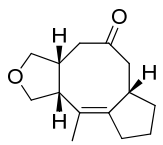
Colorless crystals, 29% yield, m.p.154-155 °C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.71(d, J = 7.8 Hz, 2H), 7.33 (d, J = 7.8 Hz, 2H), 3.63 (d, J = 9.6 Hz, 1H), 3.48 (dd, J = 9.0 and 6.0 Hz, 1H), 3.18 (dd, J = 6.0 and 5.4 Hz, 1H), 2.86 (t, J = 7.2 Hz 1H), 2.82 (d, J = 10.8 Hz, 1H), 2.56 (d, J = 6.6 Hz, 1H), 2.52-2.46 (m, 1H), 2.30-2.34 (m, 1H), 2.27-2.22 (m, 2H), 2.09 (dd, J = 4.2 and 3.6Hz, 1H), 2.01-1.99 (m, 1H), 1.81-1.78 (m, 2H), 1.73-1.71 (m, 1H), 1.66 (s, 1H), 1.60-1.56 (m, 2H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 211.8, 143.5, 142.7, 134.0, 129.8, 127.2, 123.5, 52.6, 51.4, 50.8, 43.9, 40.6, 40.0, 39.2, 34.8, 30.4, 23.0, 21.5, 16.2. IR (FT-IR): ν = 2955, 2922, 1697, 1378, 1342, 1160, 1094, 1055, 816 cm^{-1}. MS (EI): m/z (%) = 373 (M^+, 42), 315 (10), 218 (100), 160 (48), 91 (78), 42 (50). HRMS calcd for $C_{21}H_{27}NO_3S$: 373.1711. Found: 373.1714. This structure was confirmed by X-ray crystallographic analysis.



17b

(3S*, 7R*, 11S*)-2-Methyl-N-tosyl-5-azatricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (17b)

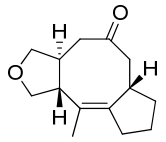
White solid, 38% yield, m.p.142-144 °C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.71(d, J = 8.4 Hz, 2H), 7.33 (d, J = 8.4 Hz, 2H), 3.53 (d, J = 8.4 Hz, 1H), 3.45 (dd, J = 8.4 and 7.8 Hz, 1H), 3.27 (t, J = 10.5 Hz, 1H), 3.17 (t, J = 6.0 Hz, 1H), 2.99 (t, J = 9.9 Hz, 1H), 2.72 (dd, J = 8.4 Hz,1H), 2.54-2.51 (m, 1H), 2.48 (d, J = 10.8 Hz, 1H), 2.45 (s, 3H), 2.36-2.25 (m, 3H), 2.20-2.04 (m, 1H), 1.87 (m, 1H), 1.77-1.59 (m, 3H), 1.54-1.52 (m, 1H), 1.45 (s, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 209.8, 143.9, 143.5, 133.7, 129.7, 127.5, 123.1, 53.0, 52.2, 50.4, 48.1, 43.0, 39.0, 37.5, 32.6, 29.3, 22.3, 21.5, 15.7. IR (FT-IR): ν = 2955, 2922, 1697, 1378, 1342, 1160, 1094, 1055, 816 cm^{-1}. MS (EI): m/z (%) = 373 (M^+, 42), 315 (10), 218 (100), 160 (48), 91 (78), 42 (50). HRMS calcd for $C_{21}H_{27}NO_3S$: 373.1711. Found: 373.1714.



19a

(3S*, 7S*, 11S*)-2-Methyl-5-oxatricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (19a)

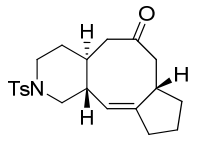
Light yellow oil, 39% yield. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 4.11 (d, J = 8.4 Hz, 1H), 4.01 (t, J = 7.8Hz, 1H), 3.84 (dd, J = 5.4 and 3.6 Hz, 1H), 3.21 (dd, J = 8.4 Hz and 7.8, 1H), 2.94 (t, J = 12Hz, 1H), 2.70-2.68 (m, 1H), 2.57-2.46 (m, 2H), 2.38-2.36 (m, 2H), 2.38-2.36 (m, 2H), 2.26-2.20 (m, 2H), 2.19-1.74 (m, 3H), 1.71 (s, 3H), 1.67-1.57 (m, 2H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 212.1, 142.0, 124.0, 71.3, 71.0, 52.5, 45.2, 41.2, 40.0, 39.3, 34.9, 30.3, 23.0, 16.5. IR (FT-IR): v = 2946, 2860, 1689, 1438, 1047, 897, 754 cm^{-1}. MS (EI): m/z (%) = 220 (M^+, 50), 134 (48), 105 (60), 91 (87), 79 (68), 41 (100). HRMS calcd for $C_{14}H_{20}O_2$: 220.1463. Found: 220.1468.



19b

(3S*, 7R*, 11S*)-2-Methyl-5-oxatricyclo[9.3.0.0^{3,7}]tetradec-1-ene-9-one (19b)

Light yellow oil, 26% yield. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 4.05 (t, J = 12 Hz, 1H), 3.95 (t, J = 12 Hz, 1H), 3.84 (dd, J = 12 and 12.6 Hz, 1H), 3.43 (dd, J = 13.2 Hz and 13.8 Hz, 1H), 2.98 (d, J = 13.2 Hz, 1H), 2.69-2.60 (m, 2H), 2.42-2.30 (m, 2H), 2.28-2.16 (m, 1H), 2.15-2.00 (m, 1H), 1.62-1.56 (m, 2H), 1.53 (s, 3H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 210.4, 143.4, 124.1, 72.8, 70.7, 53.1, 49.9, 43.3, 41.0, 37.3, 32.5, 29.1, 22.3, 15.9. IR (FT-IR): v = 2946, 2860, 1689, 1438, 1047, 897, 754 cm^{-1}. MS (EI): m/z (%) = 220 (M^+, 50), 134 (48), 105 (60), 91 (87), 79 (68), 41 (100). HRMS calcd for $C_{14}H_{20}O_2$: 220.1463. Found: 220.1468.



21

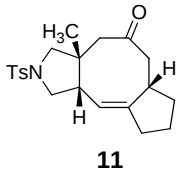
(3S*, 8R*, 12S*)-N-Tosyl-5-azatricyclo[10.3.0.0^{3,8}]pentadec-1-ene-10-one (21)

Colorless crystals, 30% yield, m.p.186 °C. ^{1}H-NMR (600 MHz, $CDCl_3$): δ 7.63 (d, J = 8.4 Hz, 2H), 7.32 (d, J = 7.8 Hz, 2H), 4.95 (dd, J = 8.4 and 1.8 Hz, 1H), 3.80 (m, 1H), 3.73 (m, 1H), 3.01(m, 1H), 2.55 (dd, J = 12 and 4.8 Hz, 1H), 2.49 (dd, J = 12 and 5.4Hz, 1H), 2.44 (s, 3H), 2.40-2.36 (m, 1H), 2.32-2.23 (m, 3H), 2.20-2.15 (m, 2H), 2.01-1.60 (m, 5H), 1.63-1.49 (m, 1H), 1.31-1.26 (m, 1H), .089-0.83 (m, 1H). ^{13}C-NMR (150 MHz, $CDCl_3$): δ 212.1, 149.5, 144.1, 133.9, 130.4, 128.2, 120.9, 53.5, 51.8, 47.0, 46.8, 42.3, 38.1, 37.9, 33.8, 32.9, 31.6, 24.2, 22.2. IR (FT-IR): v = 2954, 2923, 1697, 1340, 1215, 1163, 1090 1022, 750 cm^{-1}. MS (EI): m/z (%) = 373 (M^+, 30), 316 (8.0), 218 (100), 198(28), 91 (56), 42 (34). HRMS calcd for $C_{21}H_{27}NO_3S$: 373.1712. Found: 373.1716. This structure was confirmed by X-ray crystallographic analysis.

6. X-Ray Structure for Cycloadducts 11, 17a, 21, and NOESY Correlations.

CCDC 673996, 756195, 673997 contain the supplementary crystallographic data for **11**, **17a**, and **21**. These data can be obtained free of charge from The Cambridge Crystallographic Data Centre via www.ccdc.cam.ac.uk/data_request/cif.

Figure S1. Chemical Structure and ORTEP Figure of Cycloadduct **11**



11

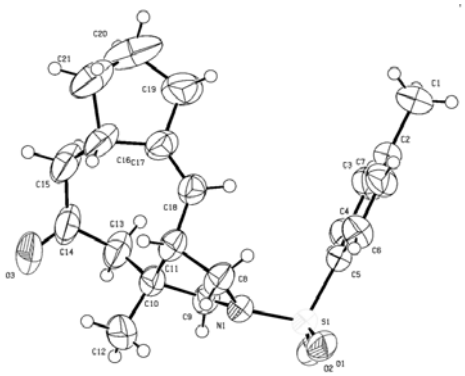
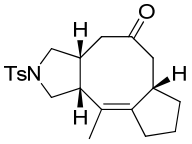


Figure S2. Chemical Structure and ORTEP Figure of Cycloadduct **17a**



17a

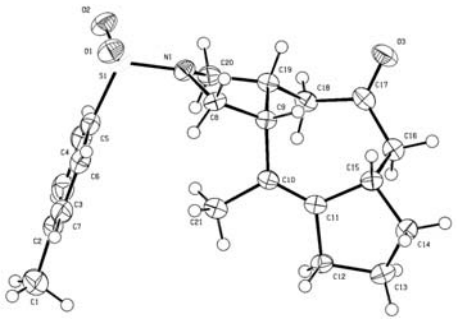
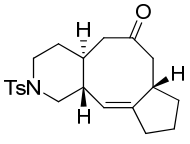


Figure S3. Chemical Structure and ORTEP Figure of Cycloadduct **21**



21

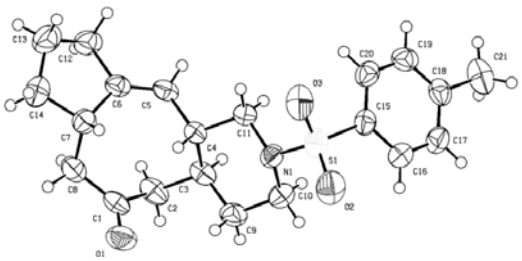


Figure S4. Selected NOESY Correlations for **5**.

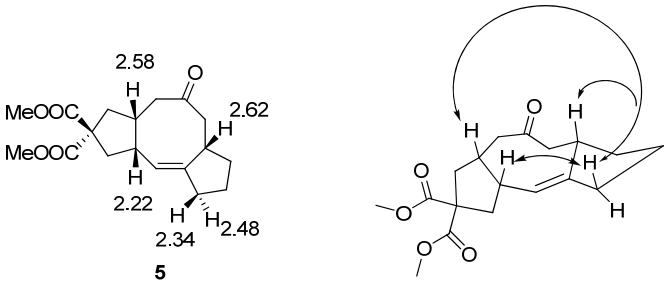


Figure S5. Selected NOESY Correlations for **7**.

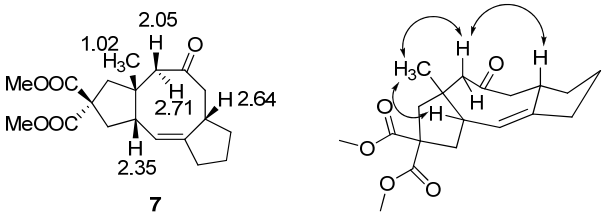


Figure S6. Selected NOESY Correlations for **9**.

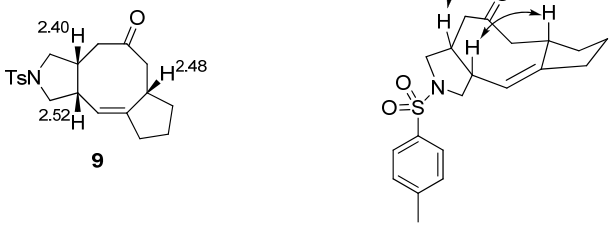
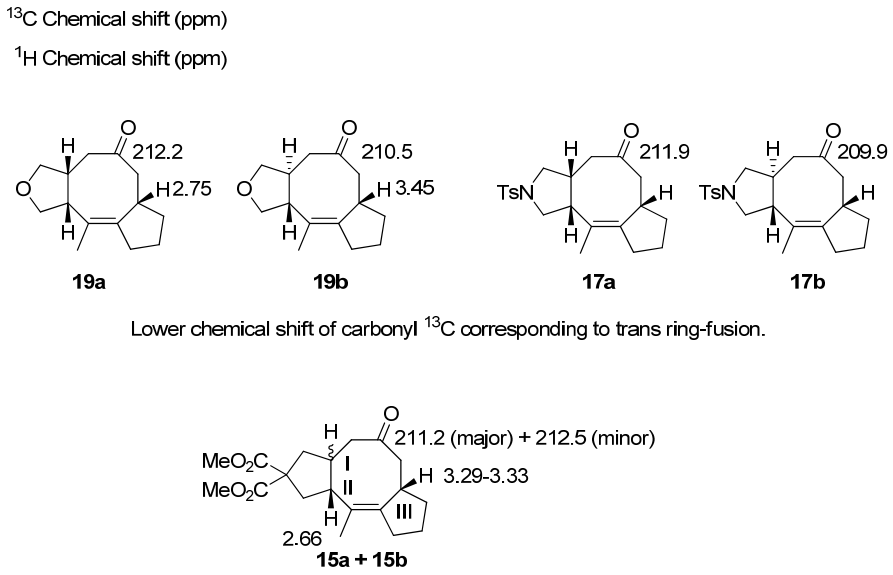


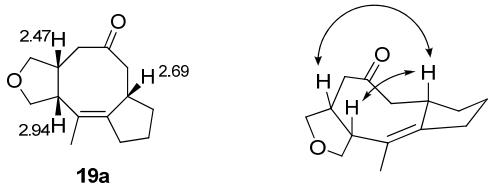
Figure S7. Selected NOESY Correlations for **13**.

Figure S8. Stereochemistry Assignment for **15a/b**.



Since the carbonyl carbons in trans compounds have lower ^{13}C NMR shift compared to those in the *cis* compounds (**19b** vs. **19a**, **17b** vs. **17a**), we assigned the major product in **15a/b** is **15b**, which has a *trans* configuration. The ratio of **15a/15b** is determined by ^{1}H NMR.

Figure S9. Selected NOESY Correlations for **19a**.



7. Stereochemistry Determination

Compounds **5, 7, 9, 13,** and **19a** have a *cis*-fused A/B rings system and this assignment is supported by NOESY experiments. The stereochemistry of compound **7** was deduced by analogy to **11**. Compounds **15b** and **19b** have a *trans*-fused A/B rings system. This is deduced by analogy to the previous experimental findings (Y. Wang, J. Wang, J. C. Su, F. Huang, L. Jiao, Y. Liang, D. Z. Yang, S. W. Zhang, P. A. Wender, Z.-X. Yu, *J. Am. Chem. Soc.* **2007**, *129*, 10060). The assignment of the structure of **15b** was mainly based on ^{13}C NMR (see section 6 above). The configuration between the substituents at positions II and III is *cis*, as shown by XRD analysis of cycloadducts **11**, **17a**, and **21**. This can be well understood based on the computational model, thus, all the [(5+2)+1] cycloadducts were assigned to the same *cis* configuration for the substituents at positions II and III.

8. DFT Calculations and Cartesian Coordinates of Computed Species

Computational Details. All of the calculations were performed with the Gaussian 03 program.[6] B3LYP[7] functional was used to locate all the transition structures and intermediates. 6-31G* basis set is applied for all elements except for Rh, which uses the basis set of LANL2DZ+ECP.[8, 9] This method has been successfully applied to predict structures and reaction mechanisms for reactions of dirhodium tetracarboxylate complexes and other Rh-catalyzed cycloadditions.[10] Frequency calculations at the same level have been performed to confirm each stationary point to be either a minimum or a transition structure. The reported energies are Zero-point energy-corrected electronic energies ($\Delta E_{0\,K}$), enthalpies ($\Delta H_{298\,K}$), and Gibbs free energies ($\Delta G_{298\,K}$).

Table S1. The Computed Energies and Other Thermal Parameters for the Three-Component [(5+2)+1] Reaction (in Hartree)

Structure	E_{ele}	$E_{0\,K}$	$H_{298\,K}$	$G_{298\,K}$
S1	−1034.237214	−1034.4569113	−1034.222700	−1034.279901
TS	−1034.223202	−1034.4409475	−1034.208968	−1034.265139
S1a	−1034.232408	−1034.450763	−1034.217636	−1034.275472

S1:

C	-3.949464	0.107980	-0.338061
C	-3.623324	-0.716143	0.929695
C	-2.298877	-0.126013	1.463275
C	-1.581837	0.450140	0.237762
C	-2.590310	0.529405	-0.885573
C	-1.533615	-0.536514	-1.010051
C	-0.432100	1.364175	0.457246
C	0.345945	1.894117	-0.584199
Rh	0.896213	-0.197757	-0.114266
O	3.568510	0.428140	1.033618
C	2.537656	0.182668	0.578320
Cl	1.397667	-2.491898	-0.349142
H	-0.009496	1.794917	-1.610730
C	1.330364	3.018330	-0.376903
H	-0.298529	1.738617	1.470398
H	-1.692857	-0.860984	2.002663
H	-2.499208	0.705518	2.152354
H	-4.425390	-0.671937	1.672204

H	-3.490297	-1.772268	0.672367
H	-4.520305	1.008981	-0.080060
H	-4.541535	-0.458522	-1.065921
H	-2.541984	1.353903	-1.592664
H	-1.775503	-1.579205	-0.833947
H	-0.814242	-0.388467	-1.817963
H	2.204223	2.907585	-1.027105
H	0.861533	3.981499	-0.621974
H	1.678963	3.067739	0.659771

TS:

C	-1.152627	-0.702760	-1.207171
C	-1.463327	0.584989	0.324166
Rh	0.735349	-0.171359	-0.139513
Cl	1.271546	-2.506598	-0.254187
C	-2.267339	0.271750	-0.903203
C	-3.620746	-0.302724	-0.420411
C	-3.643308	-0.050131	1.106972
C	-2.155436	-0.046453	1.521803
C	-0.395917	1.537415	0.434792
C	0.367025	1.924348	-0.692407
C	1.459072	2.958146	-0.606116
O	3.428990	0.249494	1.274248
C	2.436561	0.114712	0.712135
H	2.274149	2.727570	-1.300231
H	1.069912	3.949383	-0.878970
H	1.878449	3.024813	0.403076
H	-0.077853	1.822468	-1.681599
H	-0.149342	1.915100	1.424296
H	-1.792321	-1.067433	1.681507
H	-1.948123	0.512609	2.440749
H	-4.084023	0.932301	1.316547
H	-4.229853	-0.794658	1.653602
H	-4.466923	0.171800	-0.926409
H	-3.672980	-1.374987	-0.640044

H	-2.353034	1.086943	-1.624149
H	-1.325631	-1.735420	-0.919194
H	-0.645069	-0.578858	-2.167279

S1a:

C	-2.049183	-0.311600	-0.879838
C	-0.820993	-1.160525	-1.217556
C	-0.538734	1.681786	-0.179643
C	0.331143	1.496991	-1.301968
Rh	0.586421	-0.201884	-0.006904
C	1.512204	2.397445	-1.540314
Cl	1.932887	-2.141509	-0.125635
O	2.701675	0.893194	2.105782
C	1.919391	0.591602	1.327164
H	1.904859	2.816391	-0.607946
H	1.225356	3.232016	-2.196975
H	2.318933	1.851951	-2.040629
H	-0.089979	1.066885	-2.208207
H	-0.887421	-2.191281	-0.864999
H	-0.519231	-1.150294	-2.268039
H	-0.300900	2.446424	0.558895
C	-1.513922	0.726153	0.124982
C	-2.366305	0.724702	1.377582
C	-3.673094	0.042224	0.917593
C	-3.188647	-1.009688	-0.097534
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H	-1.883538	0.123601	2.159678
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H	-3.983576	-1.372965	-0.755944
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9. References

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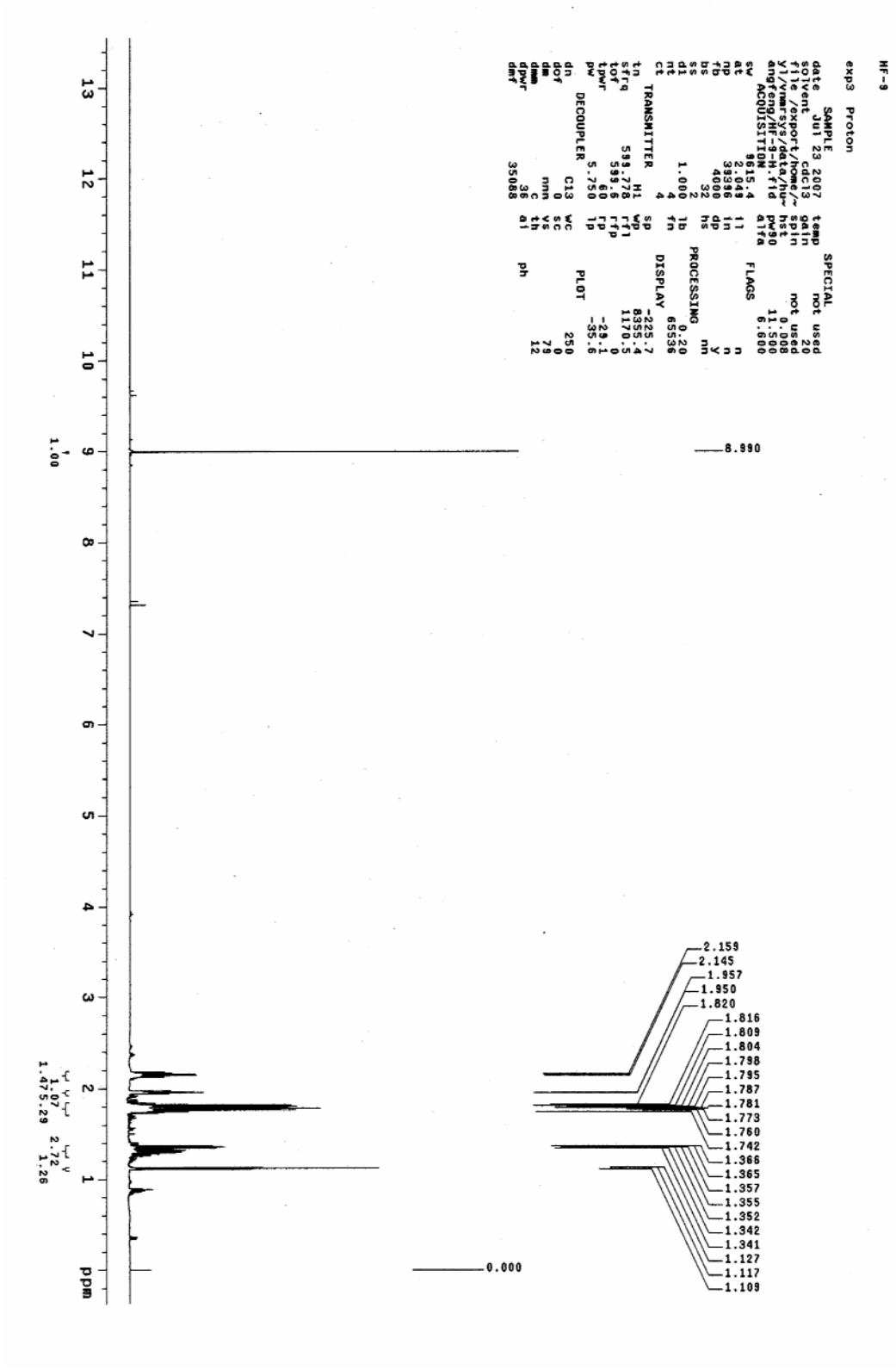
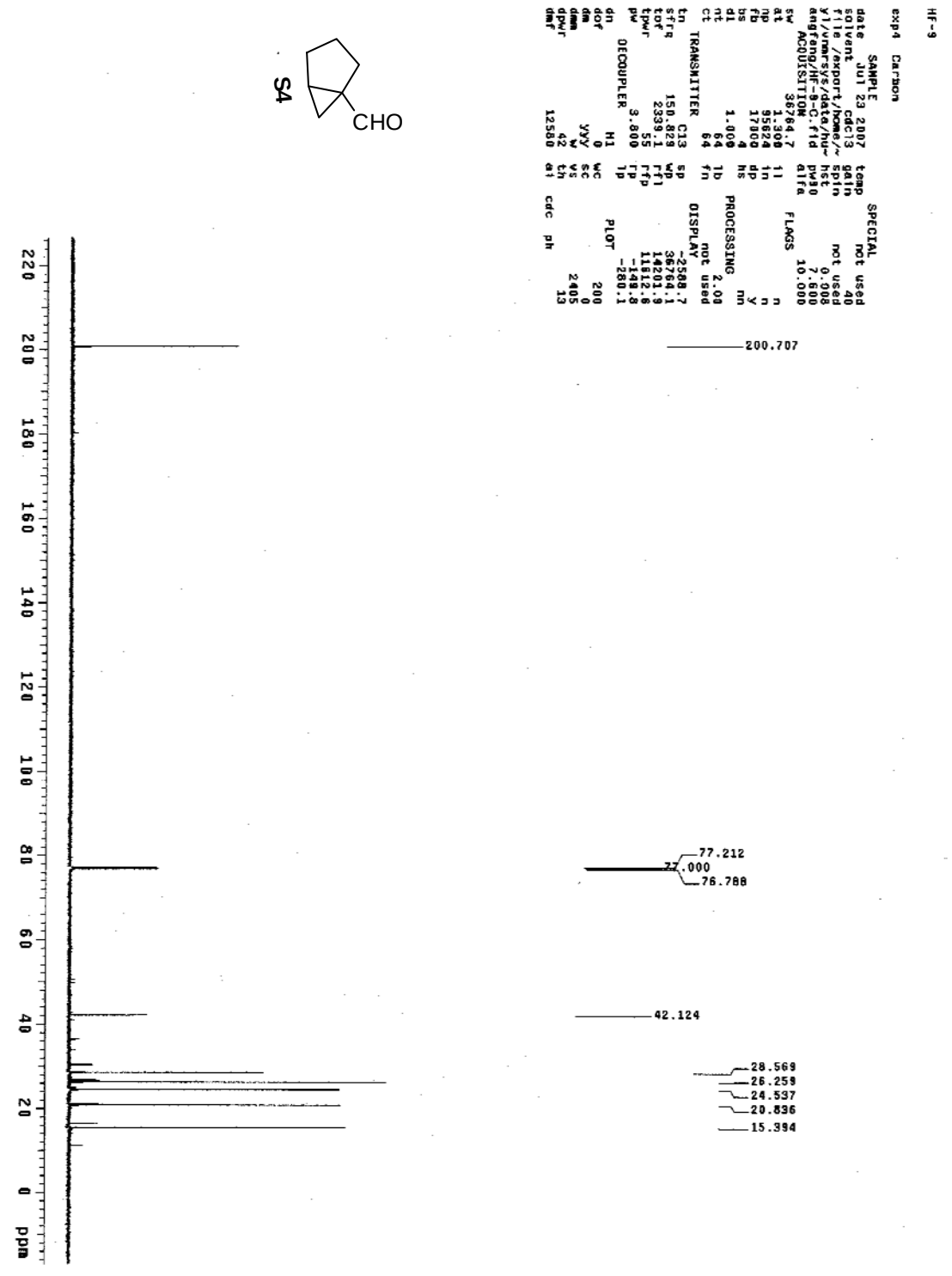
10. ^{1}H and ^{13}C Spectra for all New Compounds

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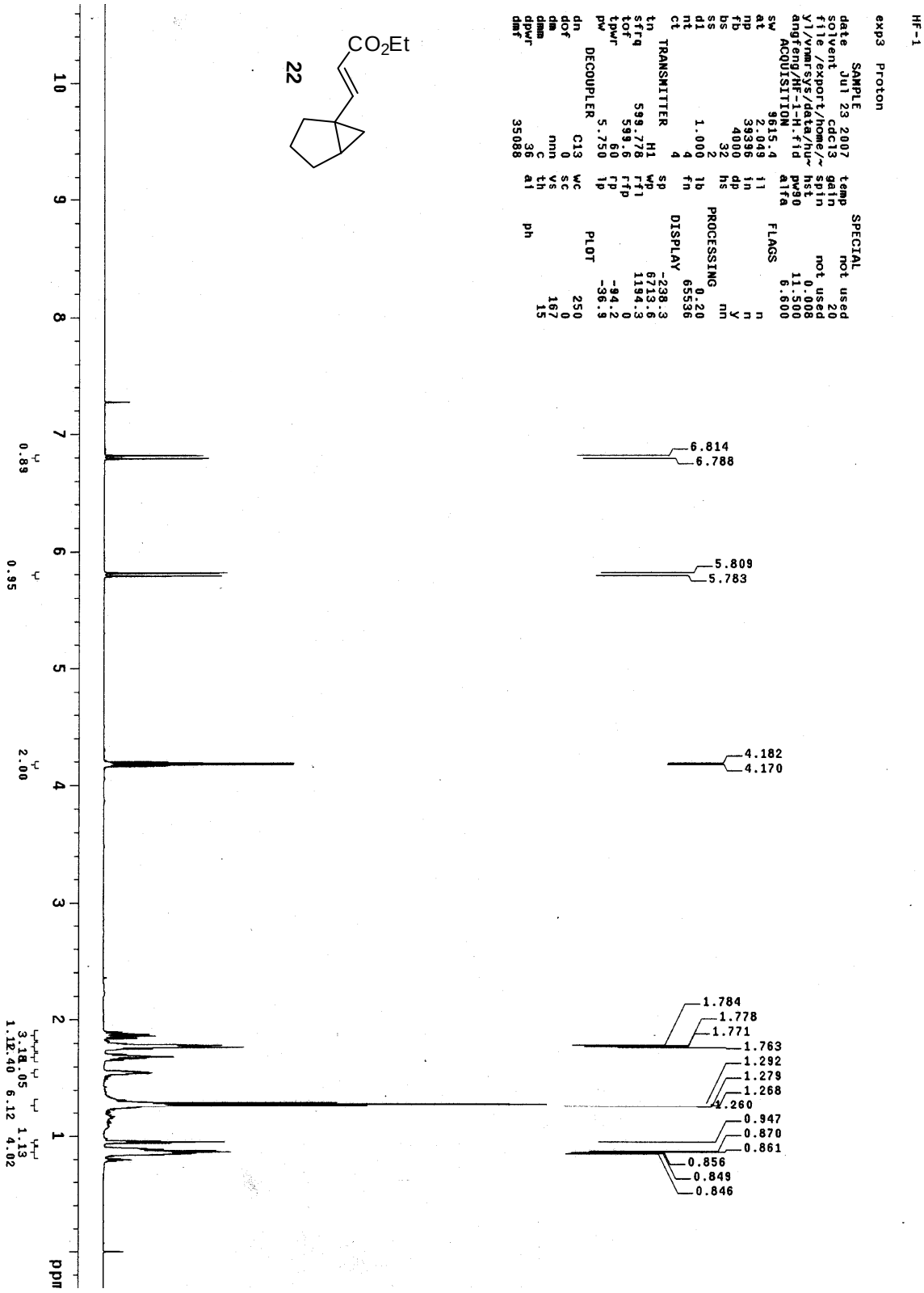
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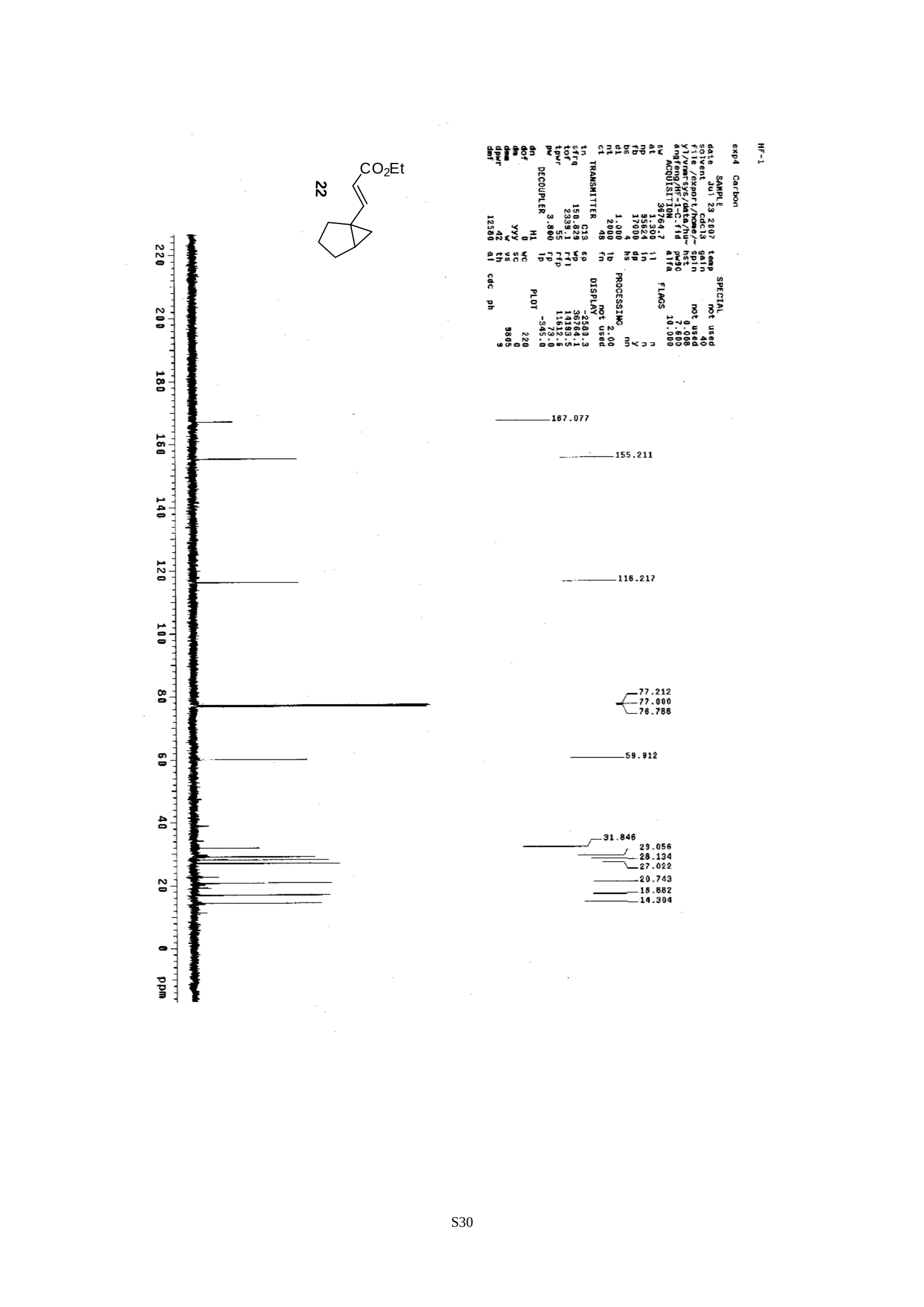
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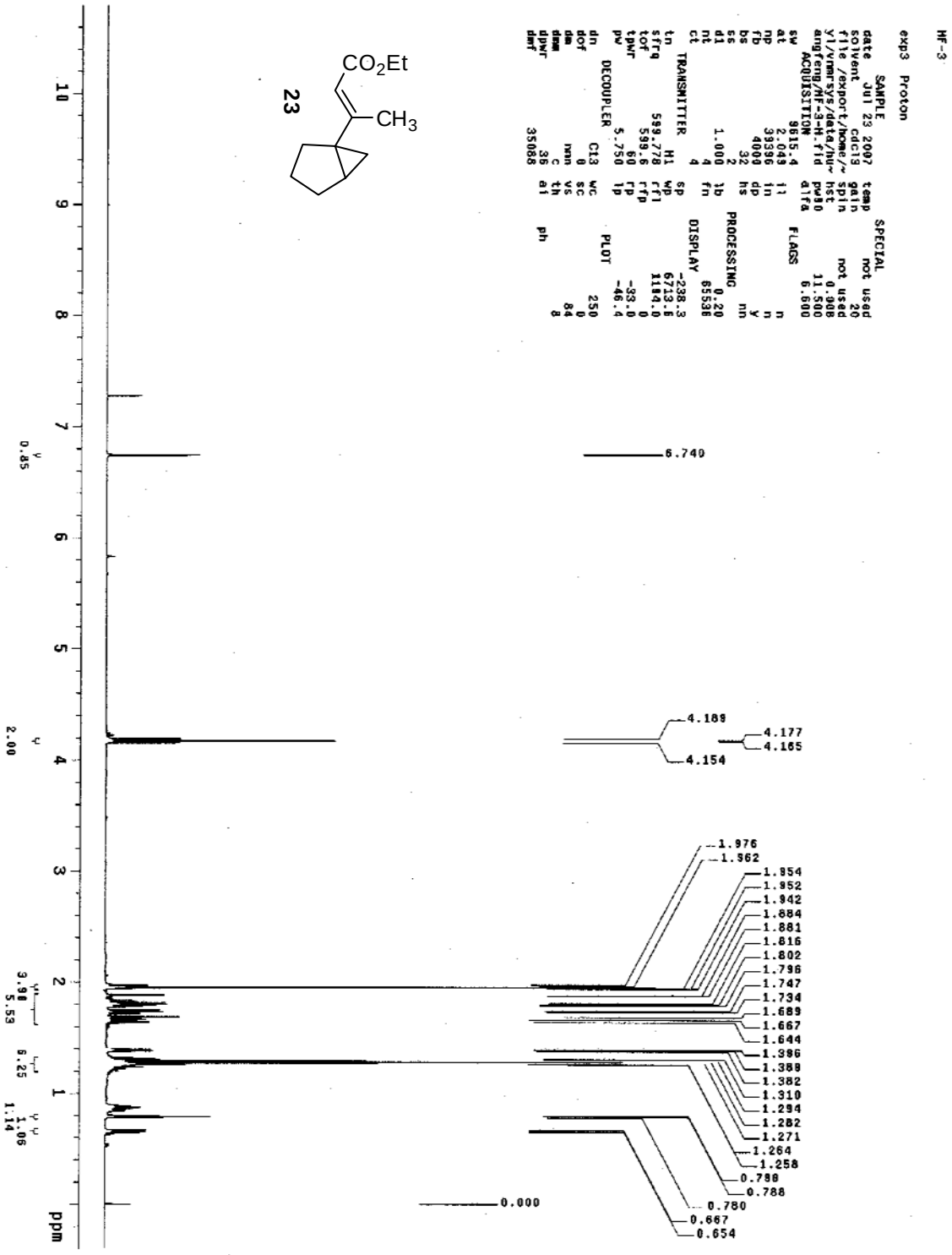
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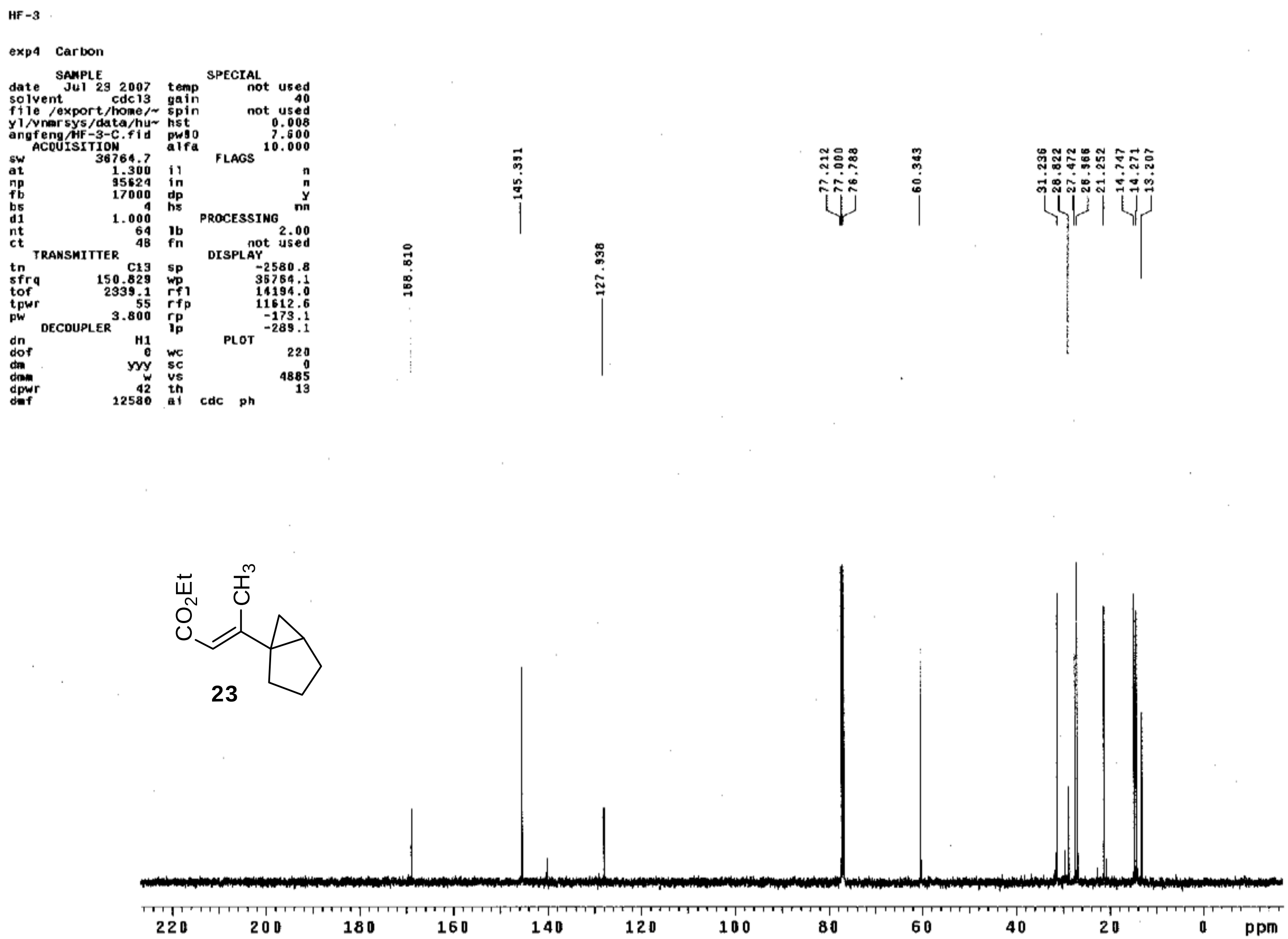
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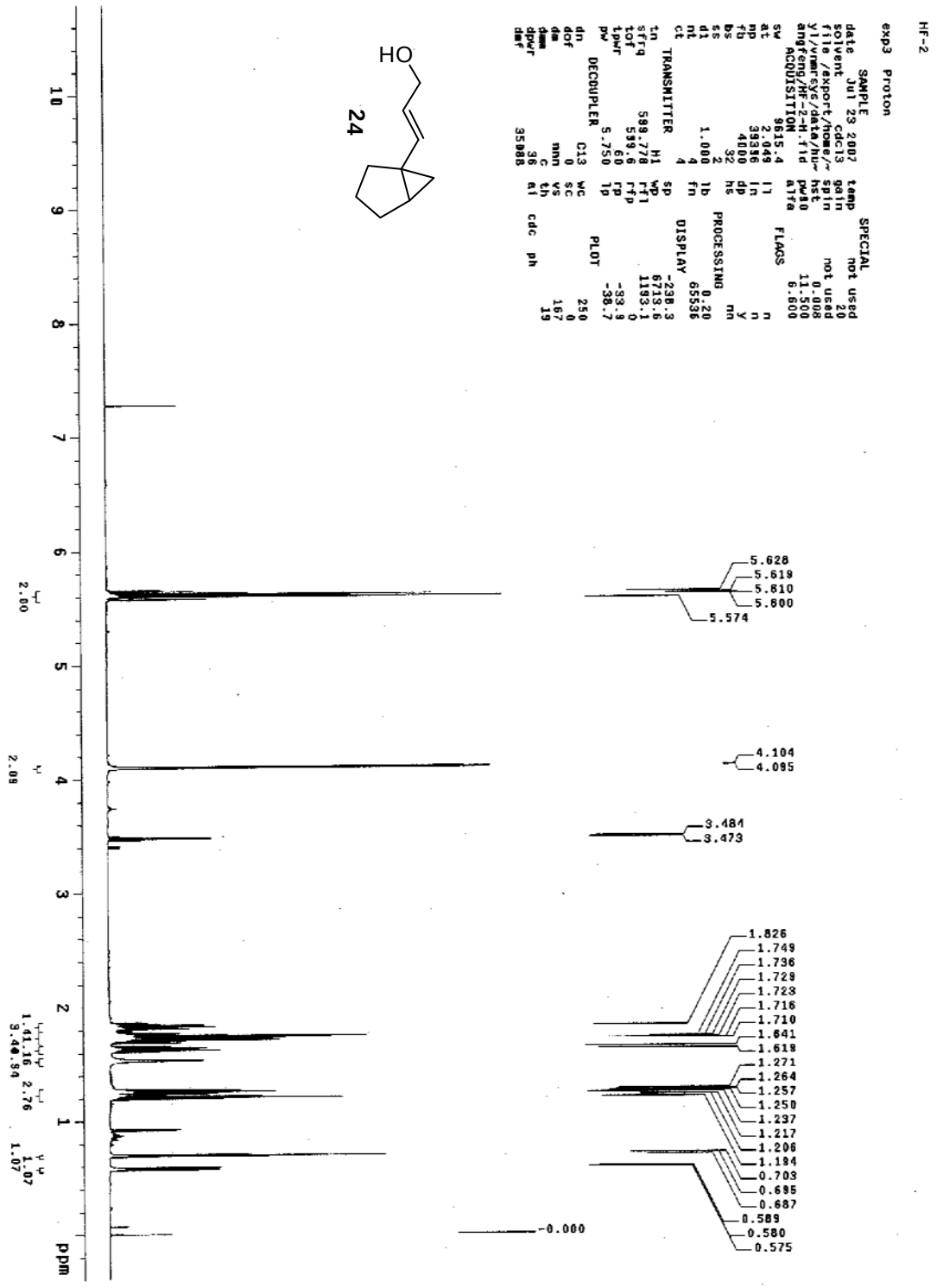
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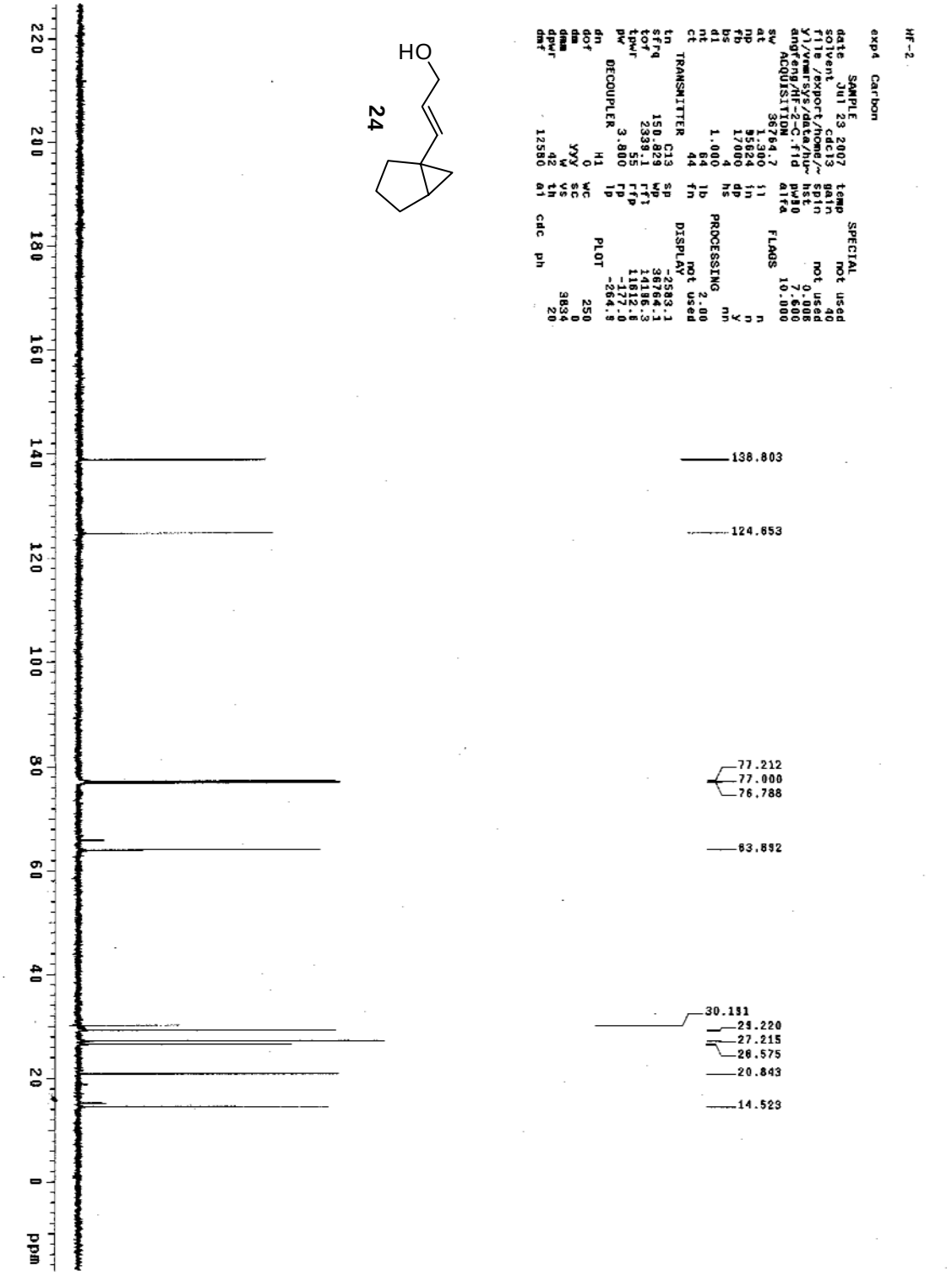
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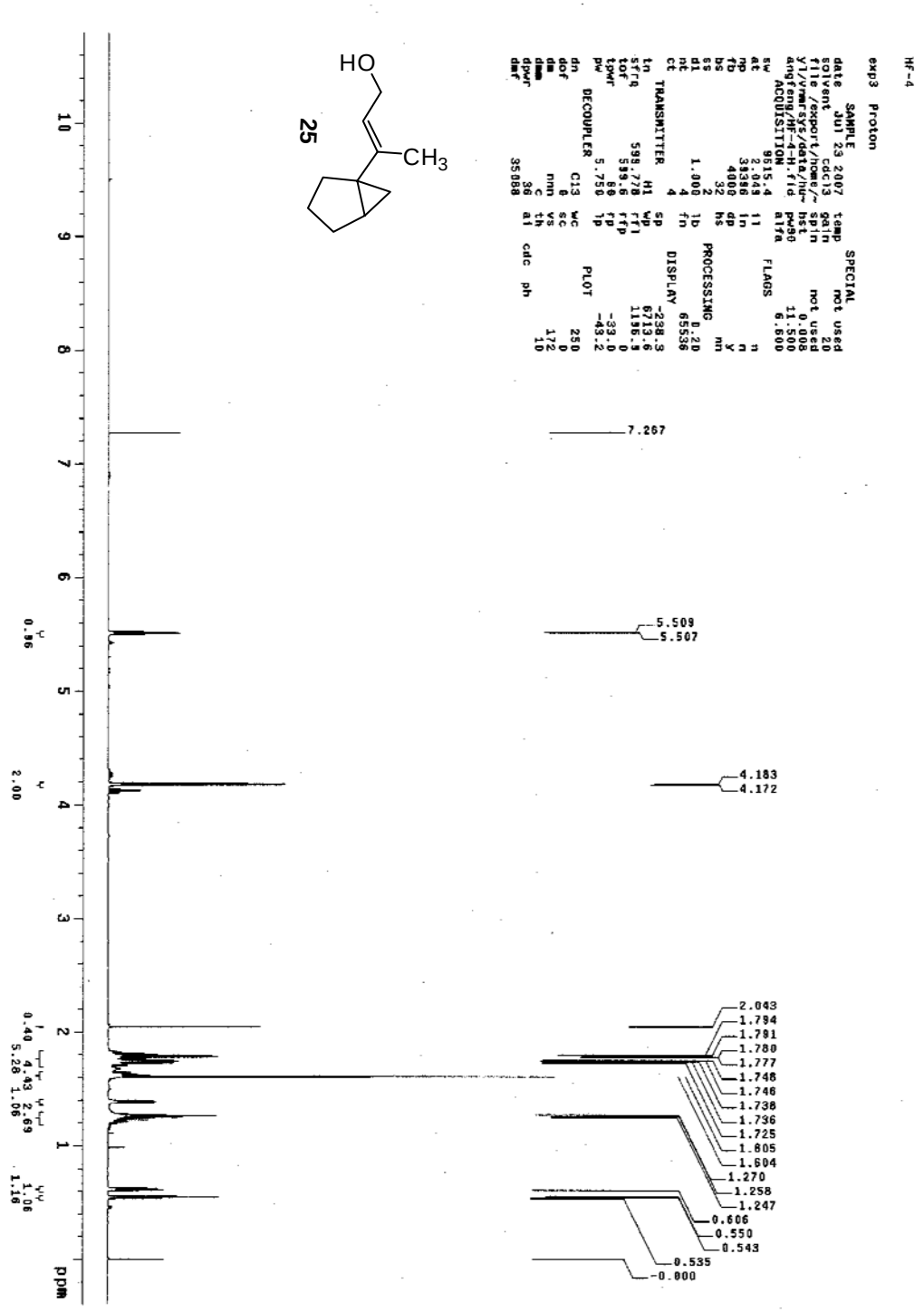
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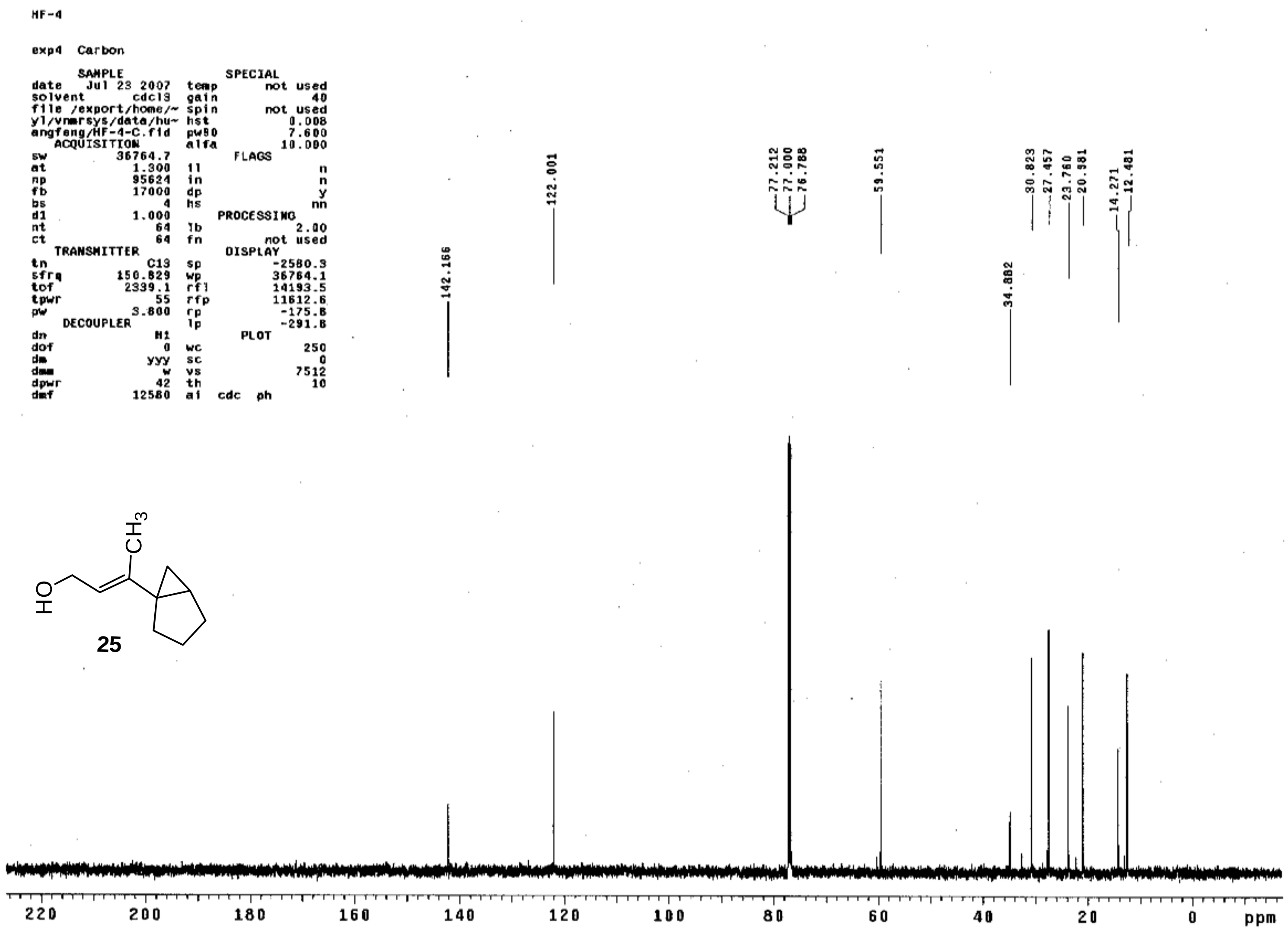

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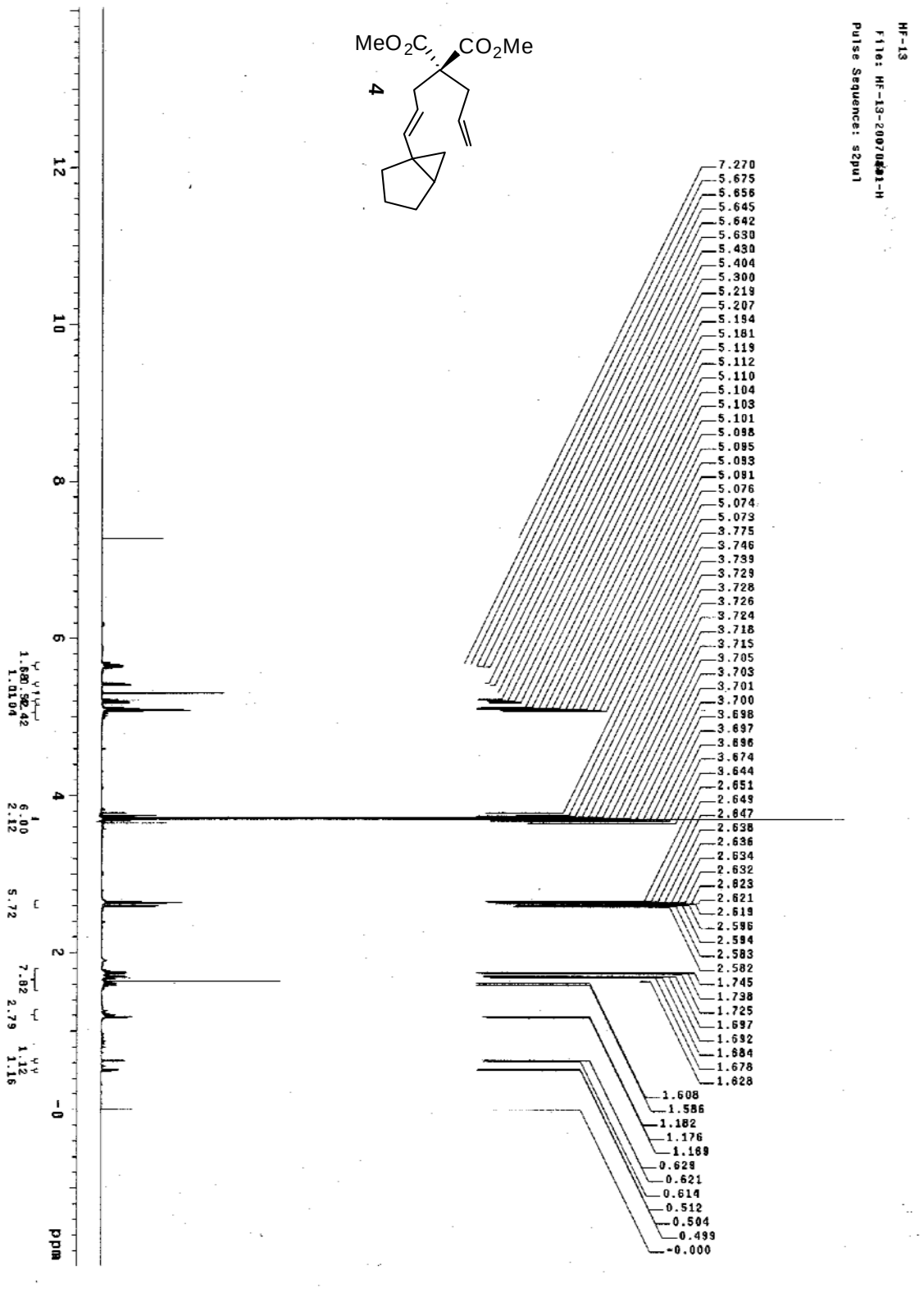
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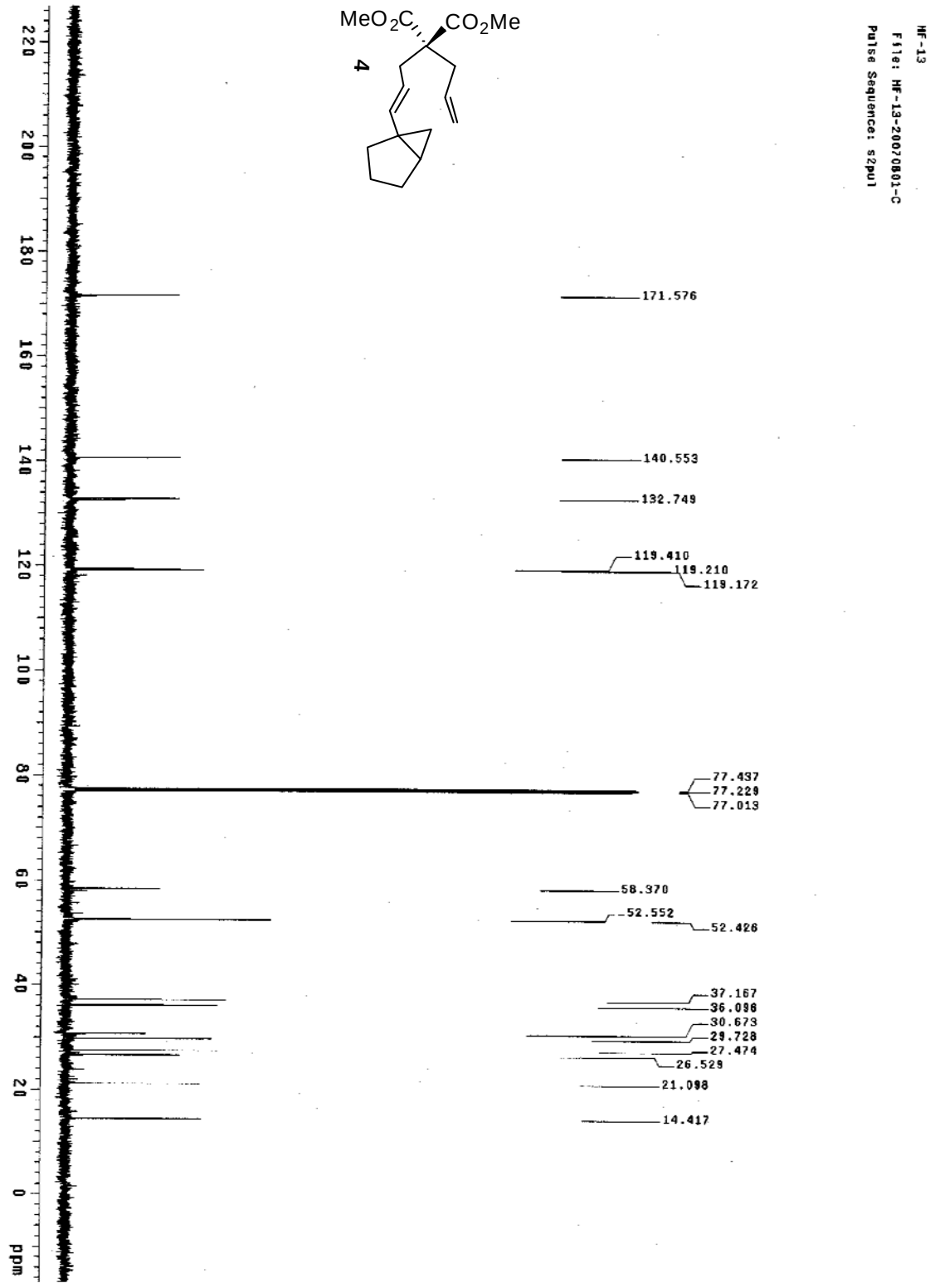
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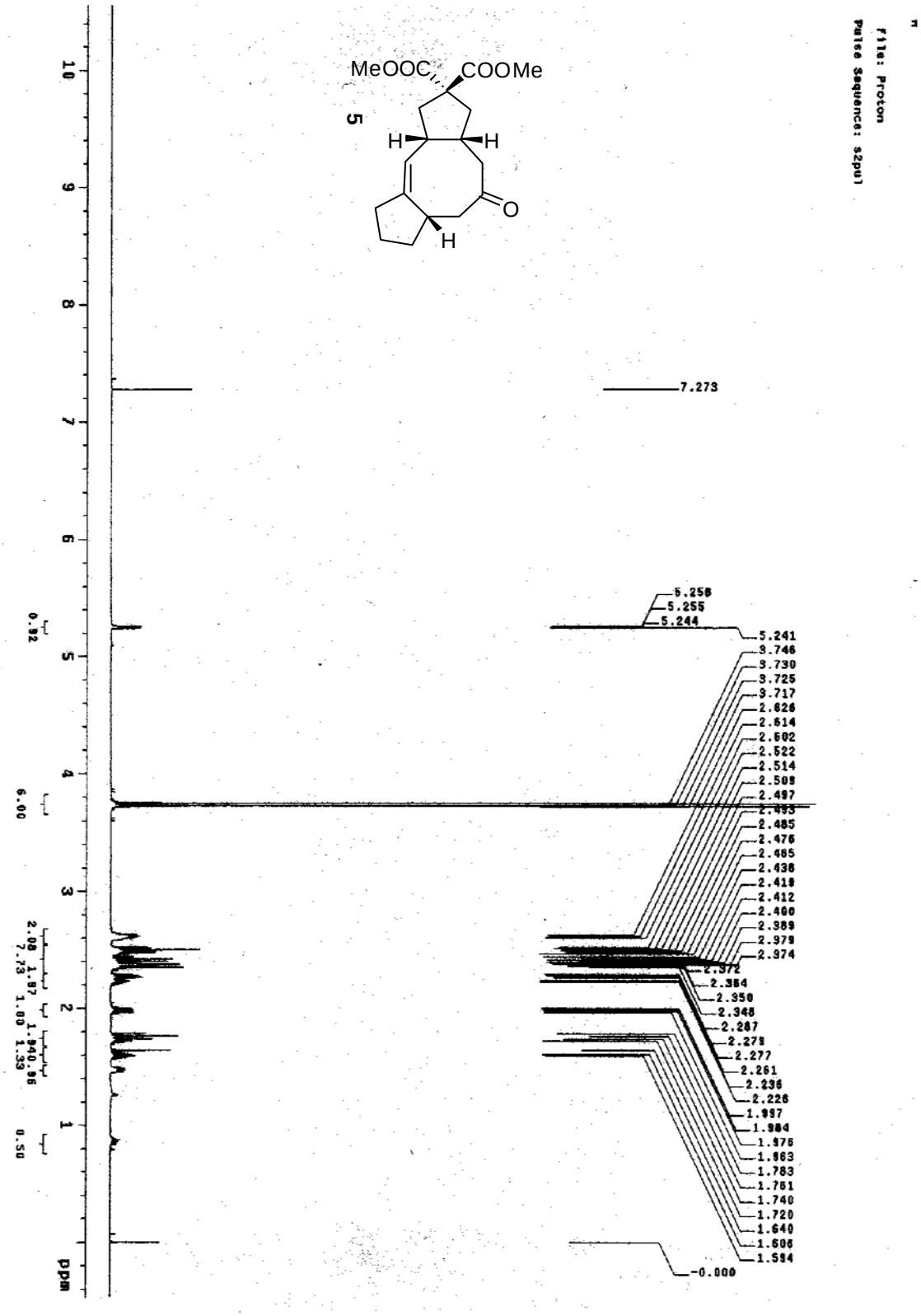
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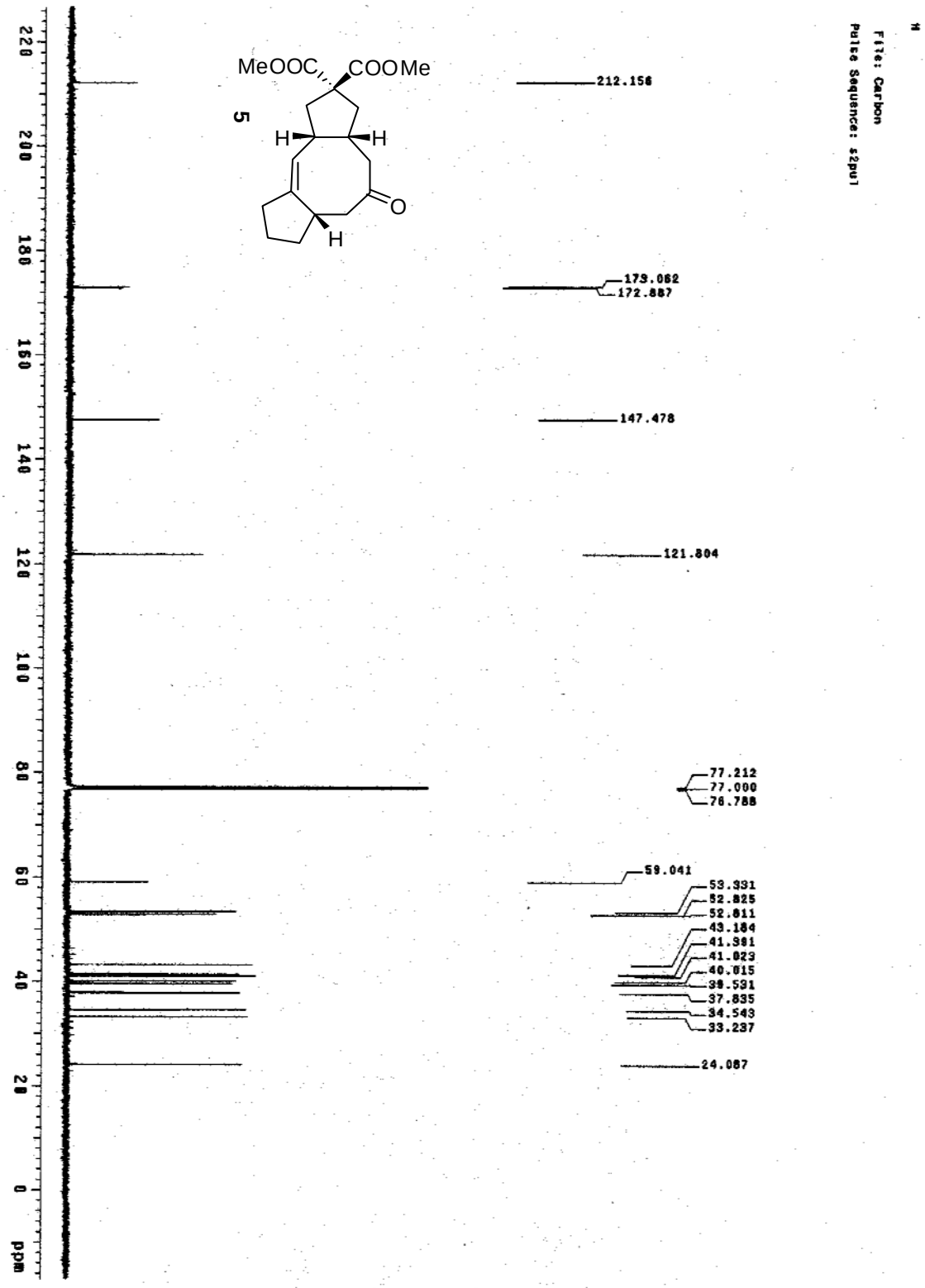
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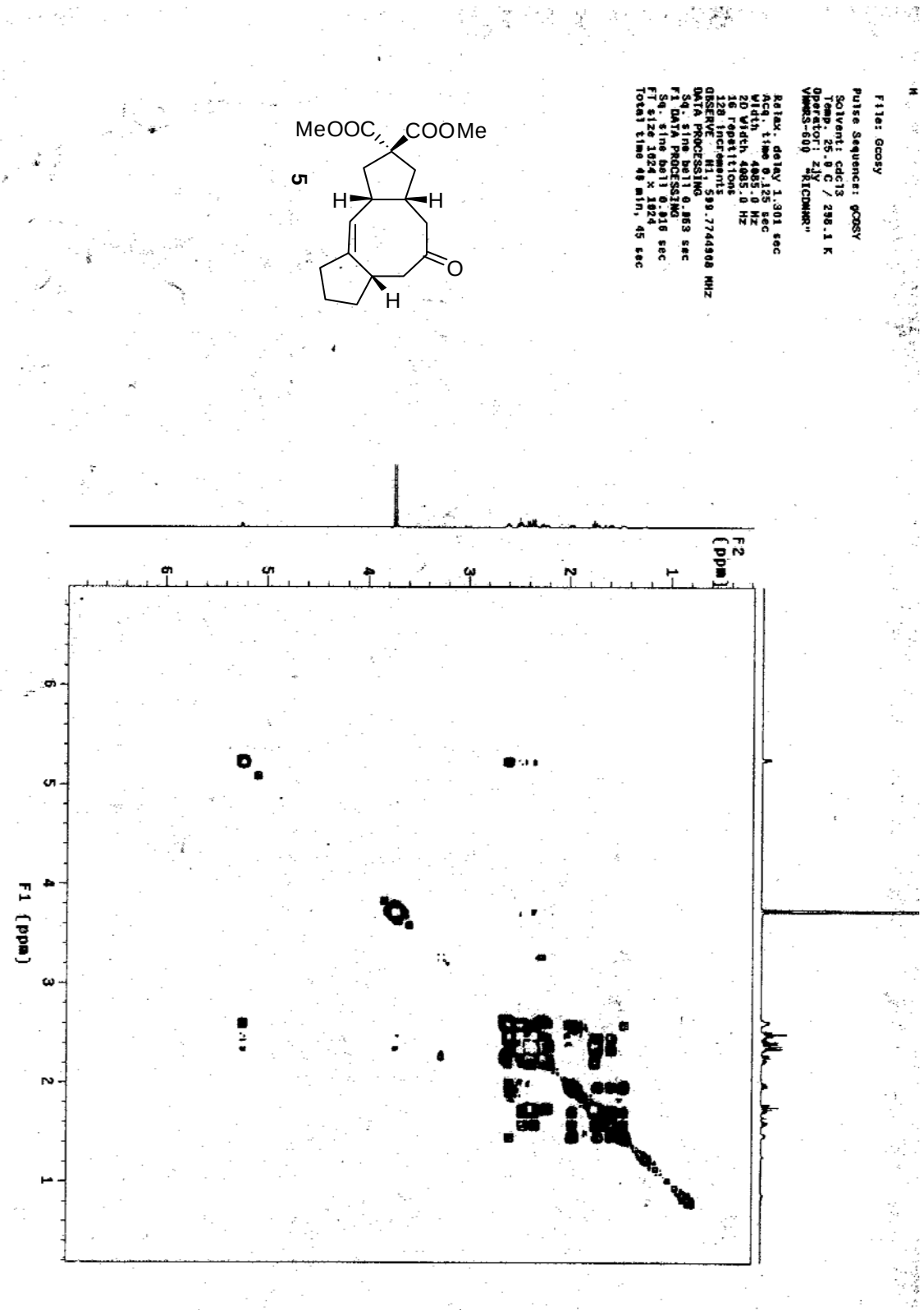
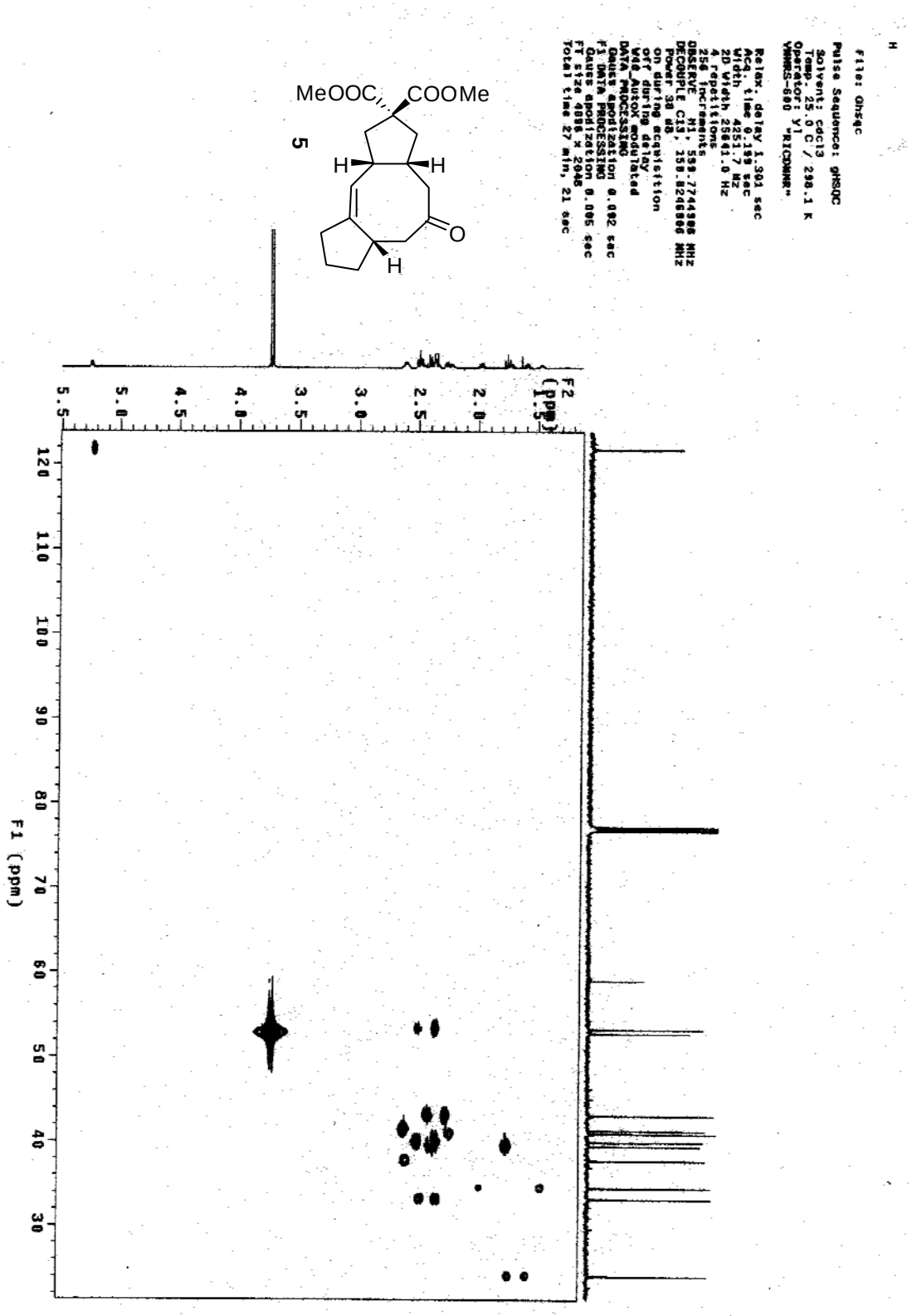
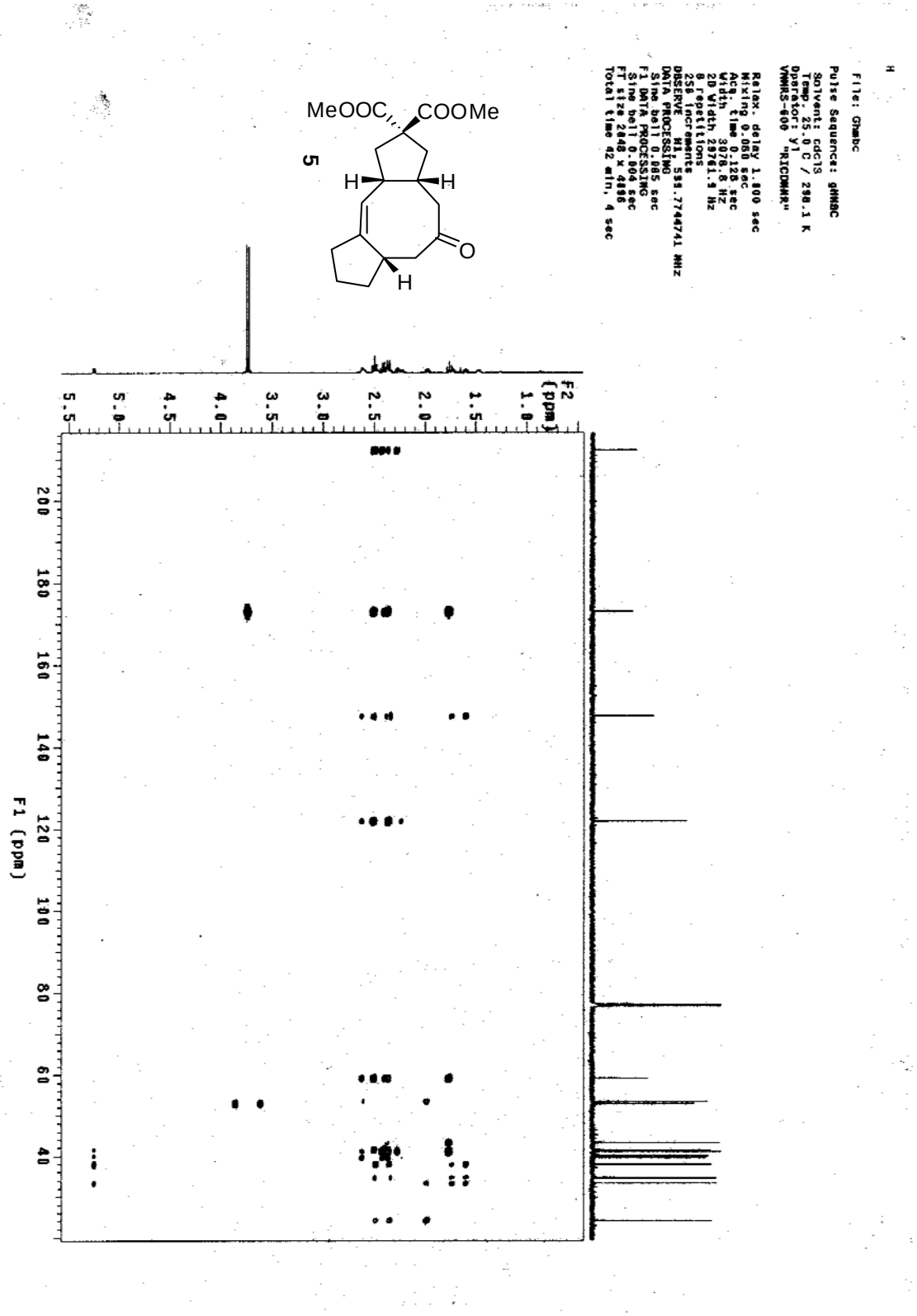
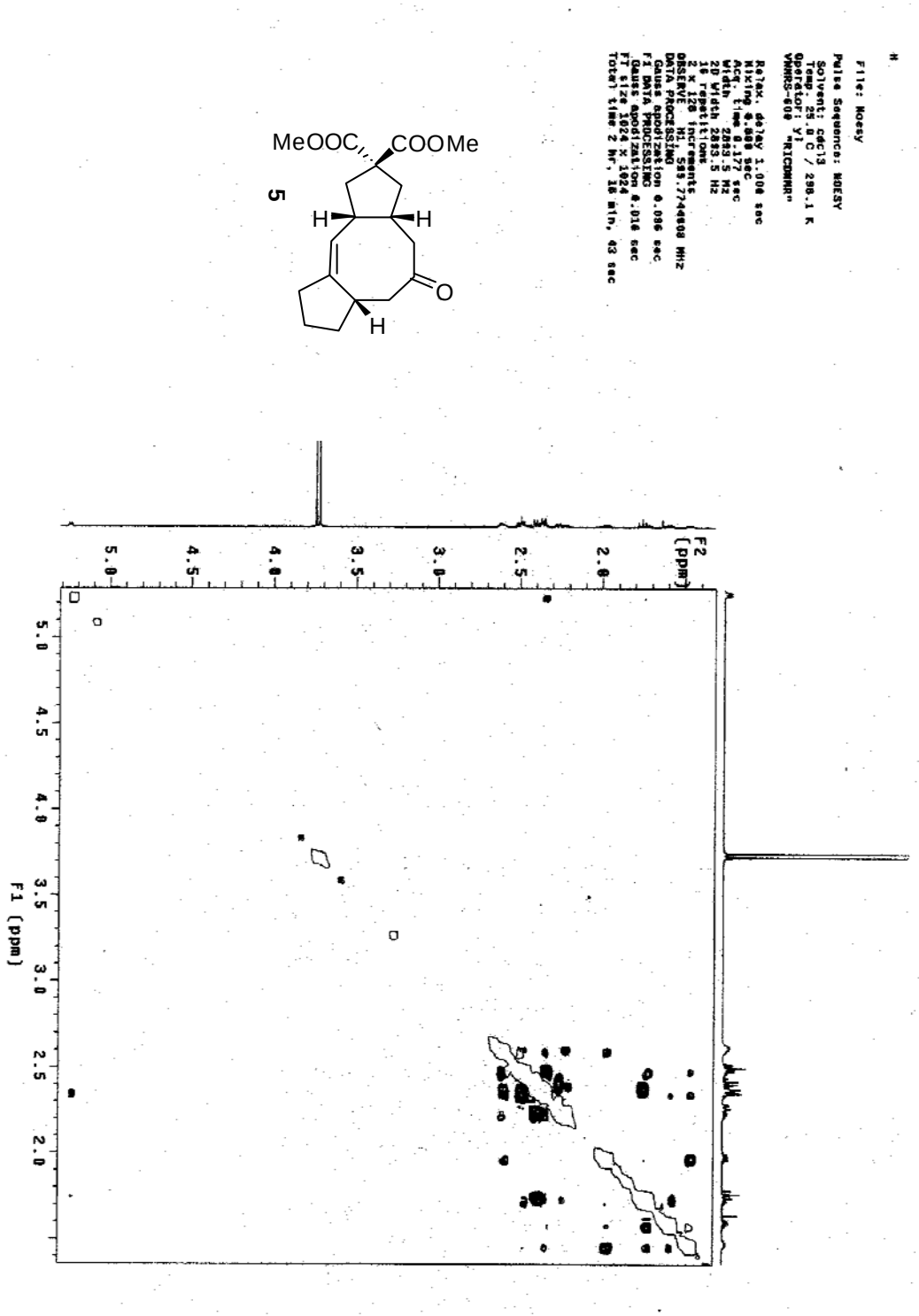
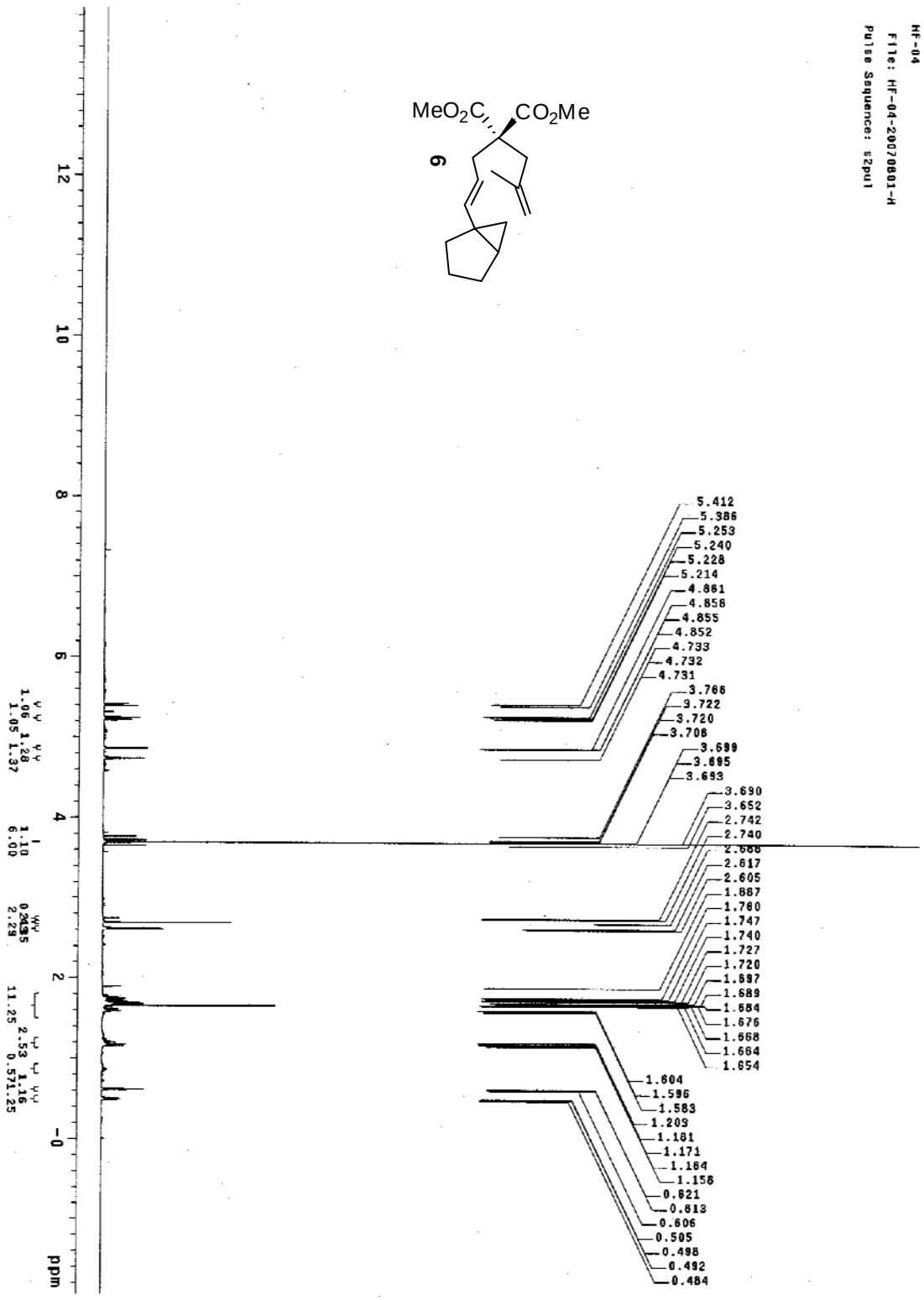
24.087
ppm

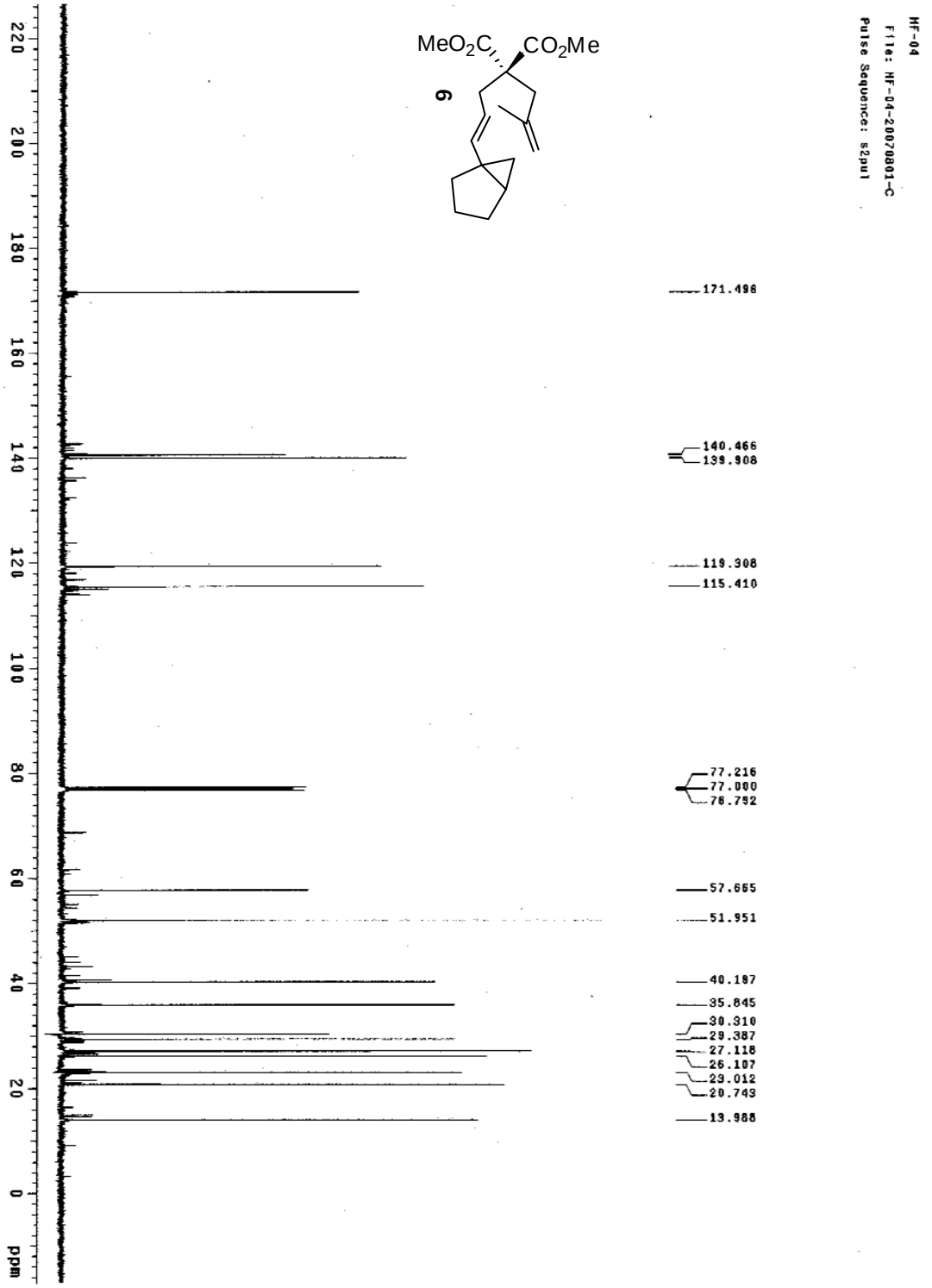

MeOOC
COOMe
5
H
H
H
O
F2 (ppm)
F1 (ppm)

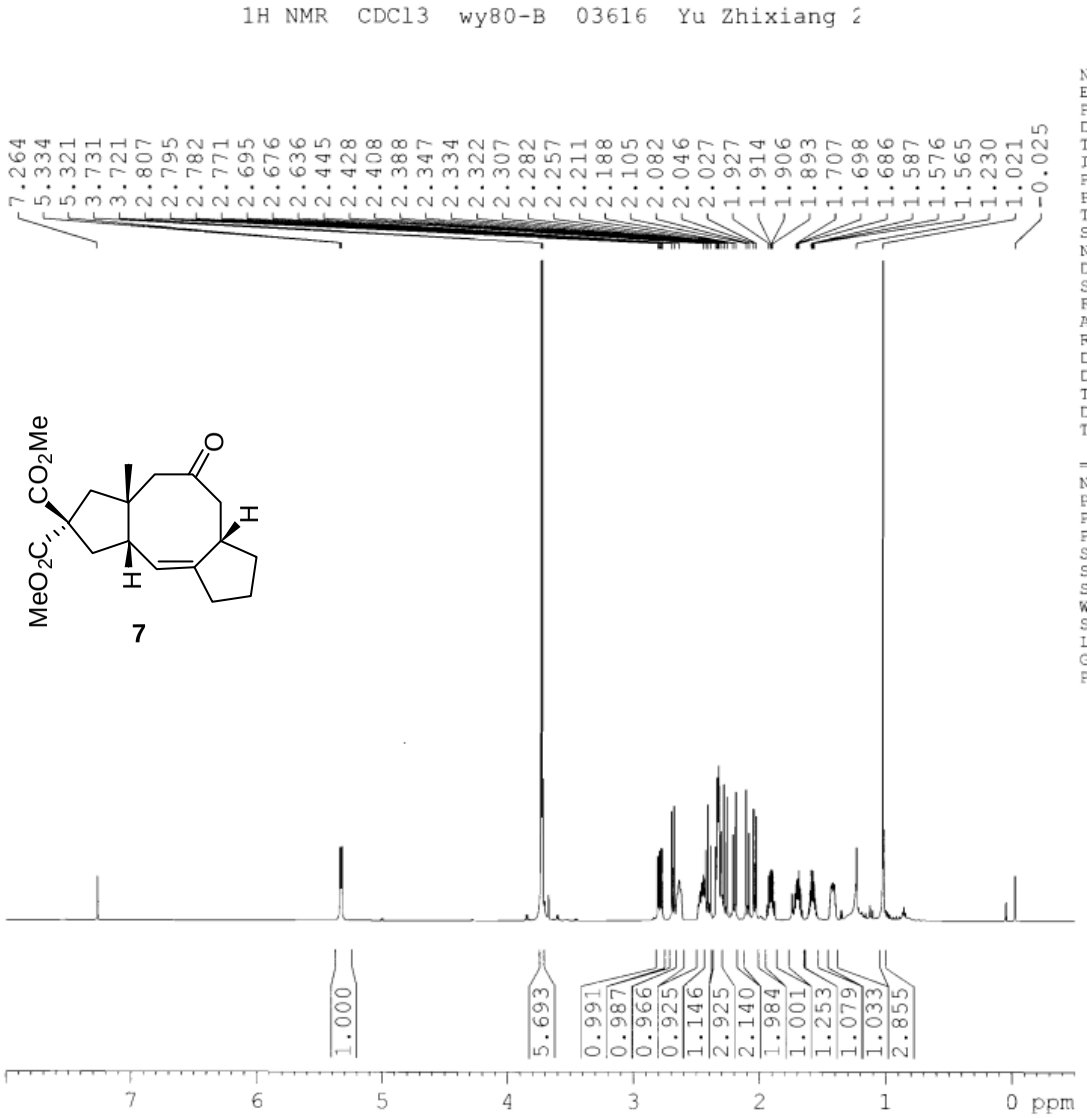

MeOOC
COOMe
5
H
H
O
H
F2 (ppm)
F1 (ppm)


MeOOC
COOMe
5
H
H
H
O
F2 (ppm)
F1 (ppm)


MeOOC
COOMe
5
H
H
O
H
F2 (ppm)
F1 (ppm)

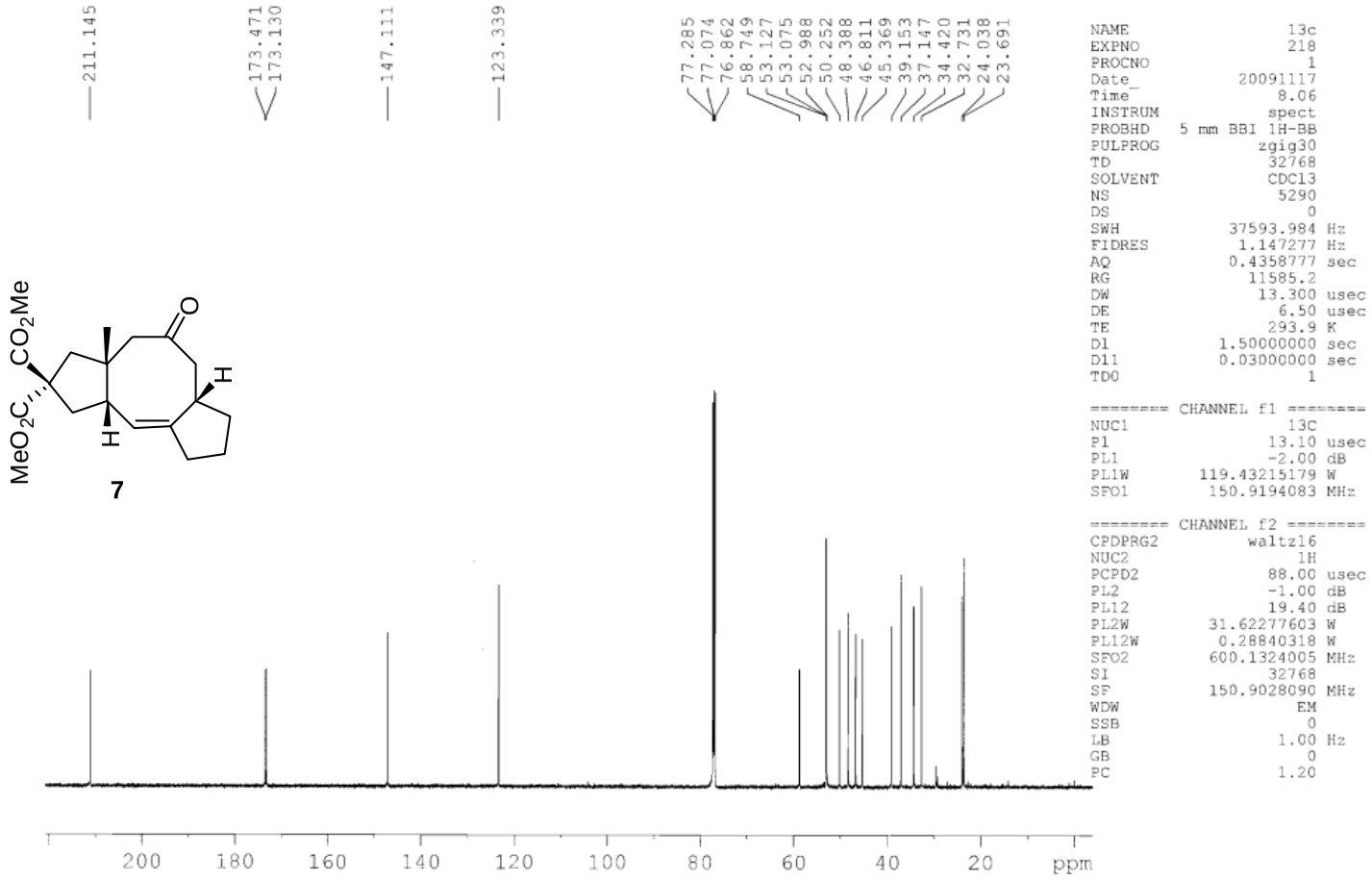

HF-04
File: HF-04-20070801-H
Pulse Sequence: s2pul
MeO2C
CO2Me
6
5.412
5.386
5.253
5.240
5.228
5.214
4.861
4.858
4.855
4.852
4.733
4.732
4.731
3.766
3.722
3.720
3.706
3.699
3.695
3.693
3.690
3.652
2.742
2.740
2.666
2.617
2.605
1.887
1.760
1.747
1.740
1.727
1.720
1.697
1.689
1.684
1.676
1.668
1.664
1.654
1.604
1.596
1.583
1.209
1.181
1.171
1.164
1.156
0.621
0.613
0.606
0.505
0.498
0.492
0.484
ppm


HF-04
File: HF-04-20070801-C
Pulse Sequence: s2pul
MeO2C
CO2Me
6
171.496
140.466
139.908
119.908
115.410
77.216
77.000
76.792
57.665
51.951
40.187
35.845
30.310
29.387
27.118
26.107
23.012
20.743
13.988
220
200
180
160
140
120
100
80
60
40
20
0
ppm

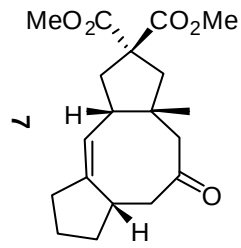


NAME	1h
EXPNO	787
PROCNO	1
Date_	20091116
Time_	20.28
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219786 sec
RG	32
DW	27.800 usec
DE	6.00 usec
TE	293.5 K
D1	5.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.60 usec
PL1	-1.00 dB
PL1W	31.62277603 W
SFO1	600.1348010 MHz
SI	65536
SF	600.1300187 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

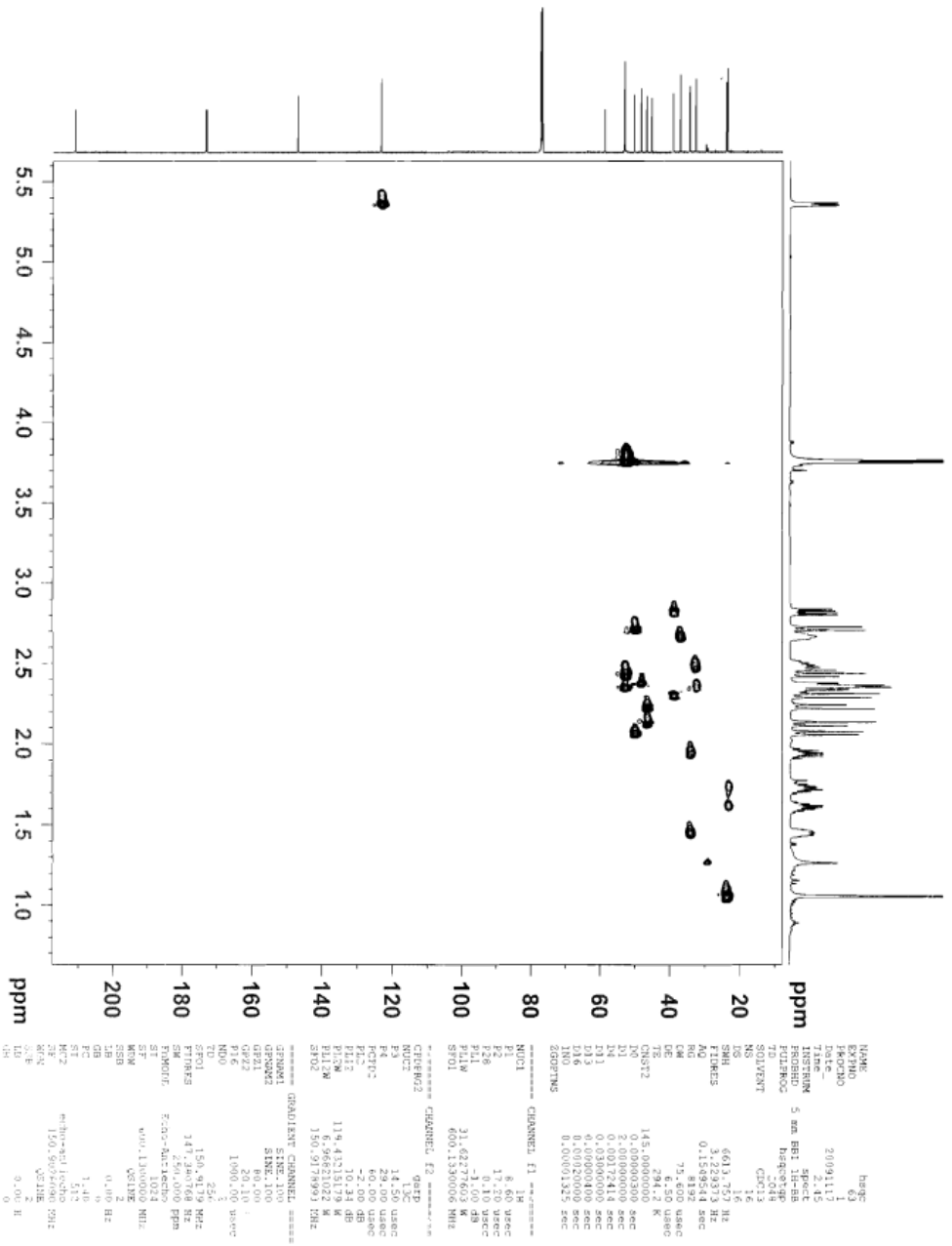
13C NMR CDCl3 wy80-B 03616 Yu Zhixiang 20091116

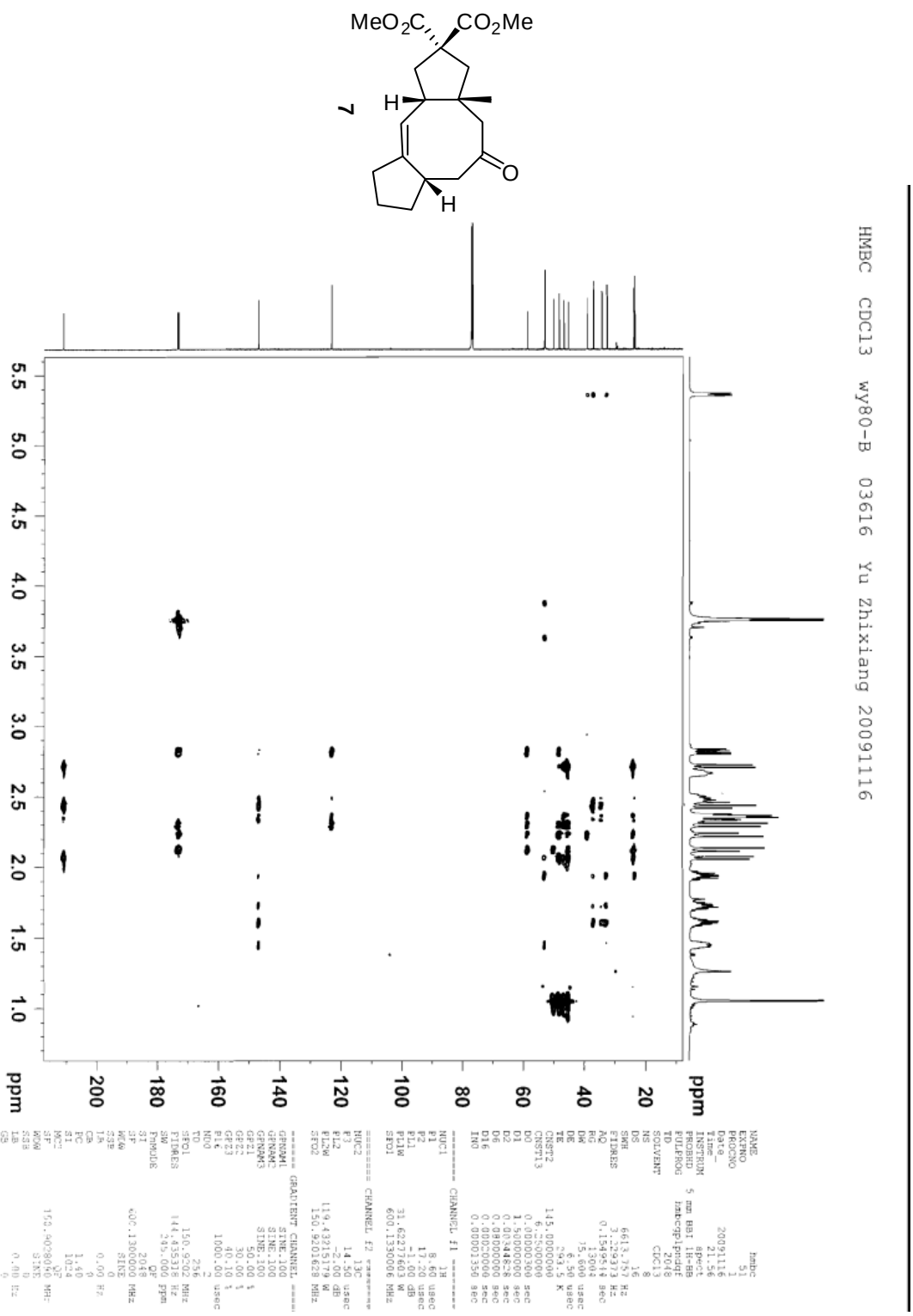


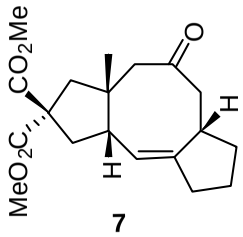
NAME	13c
EXPNO	218
PROCNO	1
Date_	20091117
Time	8.06
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	zgig30
TD	32768
SOLVENT	CDCl3
NS	5290
DS	0
SWH	37593.984 Hz
FIDRES	1.147277 Hz
AQ	0.4358777 sec
RG	11585.2
DW	13.300 usec
DE	6.50 usec
TE	293.9 K
D1	1.50000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.10 usec
PL1	-2.00 dB
PL1W	119.43215179 W
SFO1	150.9194083 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	88.00 usec
PL2	-1.00 dB
PL12	19.40 dB
PL2W	31.62277603 W
PL12W	0.28840318 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028090 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.20


MeO2C
CO2Me
H
O
H

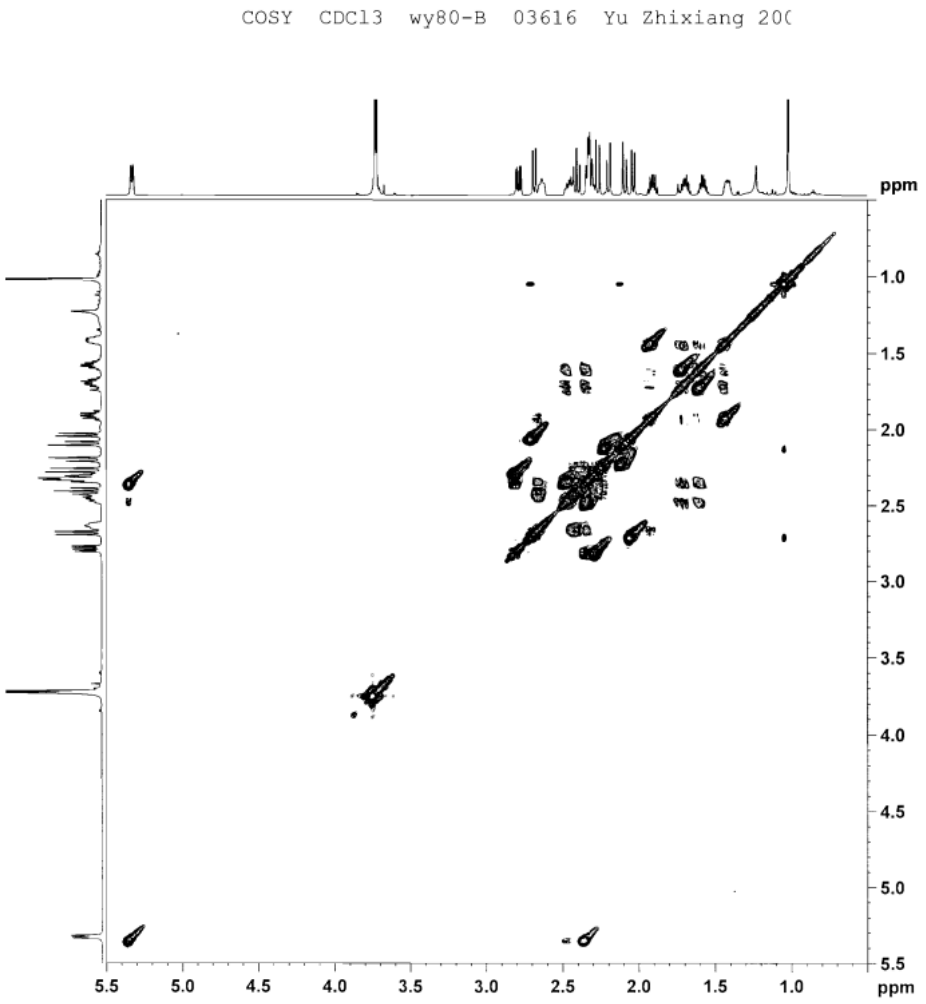
7


HSQC CDCl3 wy80-B 03616 Yu Zhixiang 20091116
ppm
ppm


MeO2C
CO2Me
H
H
O
7
HMBC CDC13 wy80-B 03616 Yu Zhixiang 20091116
ppm
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
200
180
160
140
120
100
80
60
40
20

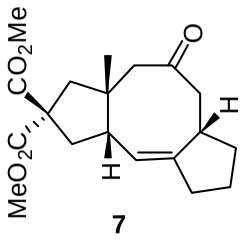


7

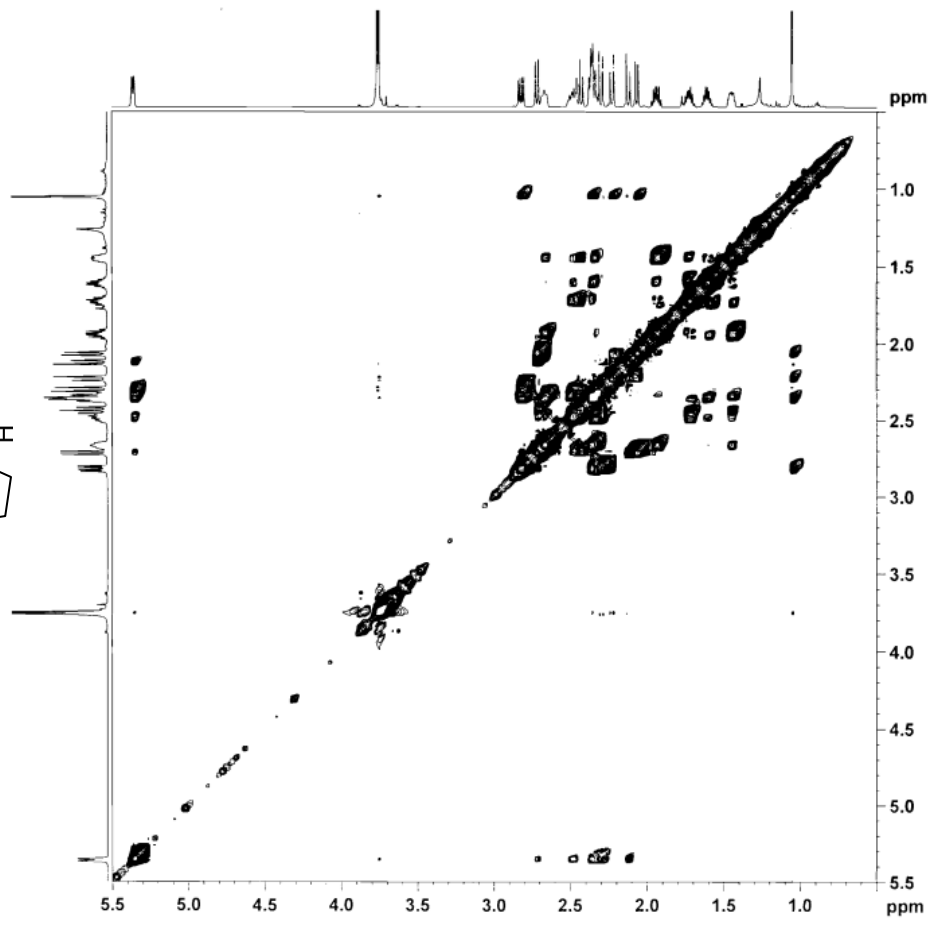


NAME	cosy	
EXPNO	89	
PROCNO	1	
Date_	20091117	
Time	8.09	
INSTRUM	spect	
PROBHD	5 mm BBI 1H-BB	
PULPROG	cosygpqf	
TD	2048	
SOLVENT	CDC13	
NS	4	
DS	16	
SWH	6613.757	Hz
FIDRES	3.229373	Hz
AQ	0.1549544	sec
RG	32	
DW	75.600	usec
DE	6.50	usec
TE	293.8	K
D0	0.00000300	sec
D1	1.50000000	sec
D13	0.00000400	sec
D16	0.00002000	sec
IN0	0.00015120	sec
======== CHANNEL f1 ========		
NUC1	1H	
P0	4.30	usec
P1	8.60	usec
PL1	-1.00	dB
PL1W	31.62277603	W
SFO1	600.1330006	MHz
====== GRADIENT CHANNEL =====		
GPNAM1	SINE.100	
GPZ1	10.00	%
P16	1000.00	usec
ND0	1	
TD	256	
SFO1	600.133	MHz
FIDRES	25.835022	Hz
SW	11.021	ppm
FnMODE	QF	
SI	1024	
SF	600.1300000	MHz
WDW	SINE	
SSB	0	
LB	0.00	Hz
GB	0	
PC	1.40	
SI	1024	
MC2	QF	
SF	600.1300000	MHz
WDW	SINE	
SSB	0	
LB	0.00	Hz
GB	0	

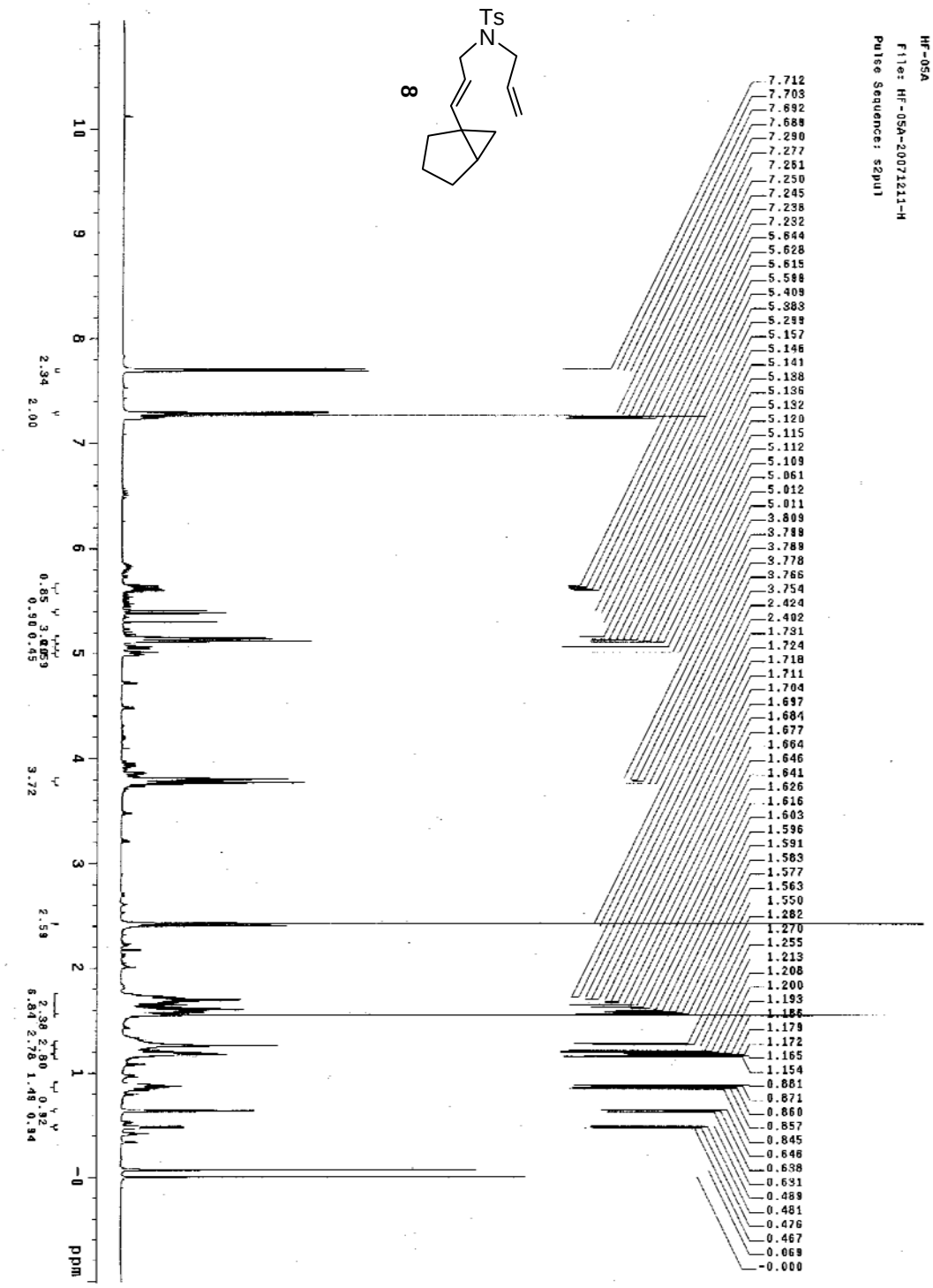
NOESY CDCl3 wy80-B 03616 Yu Zhixiang 2(

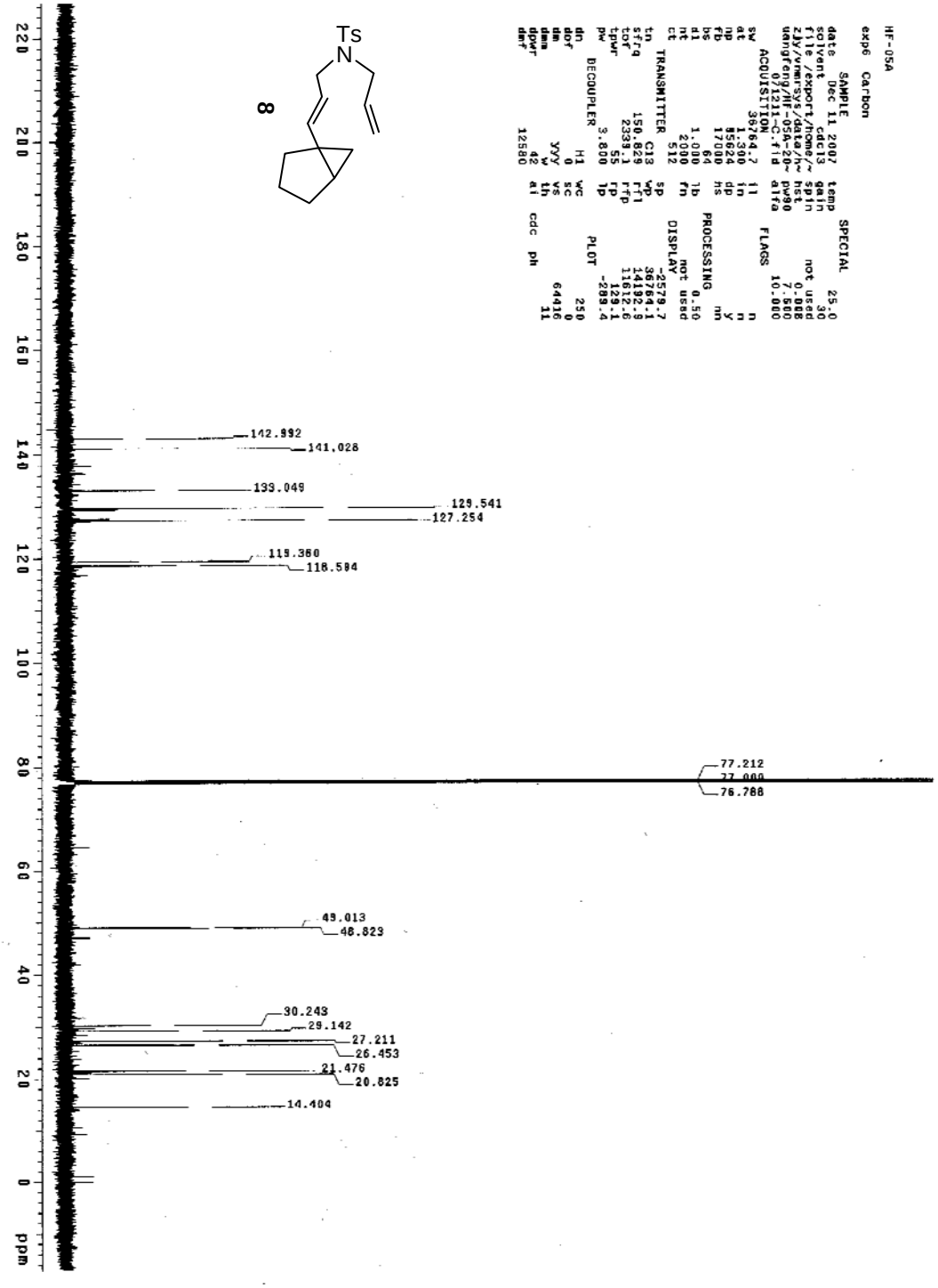


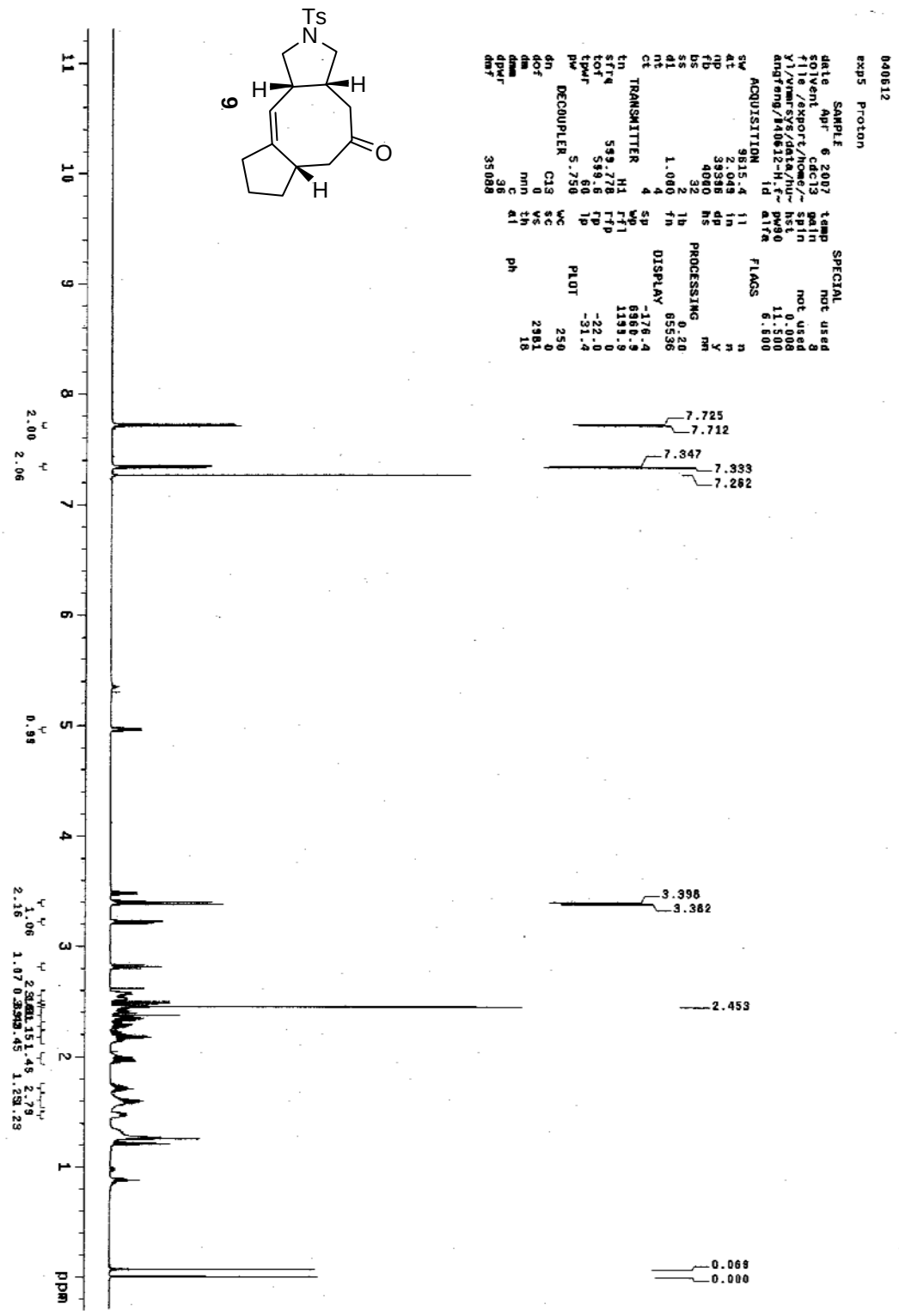
7

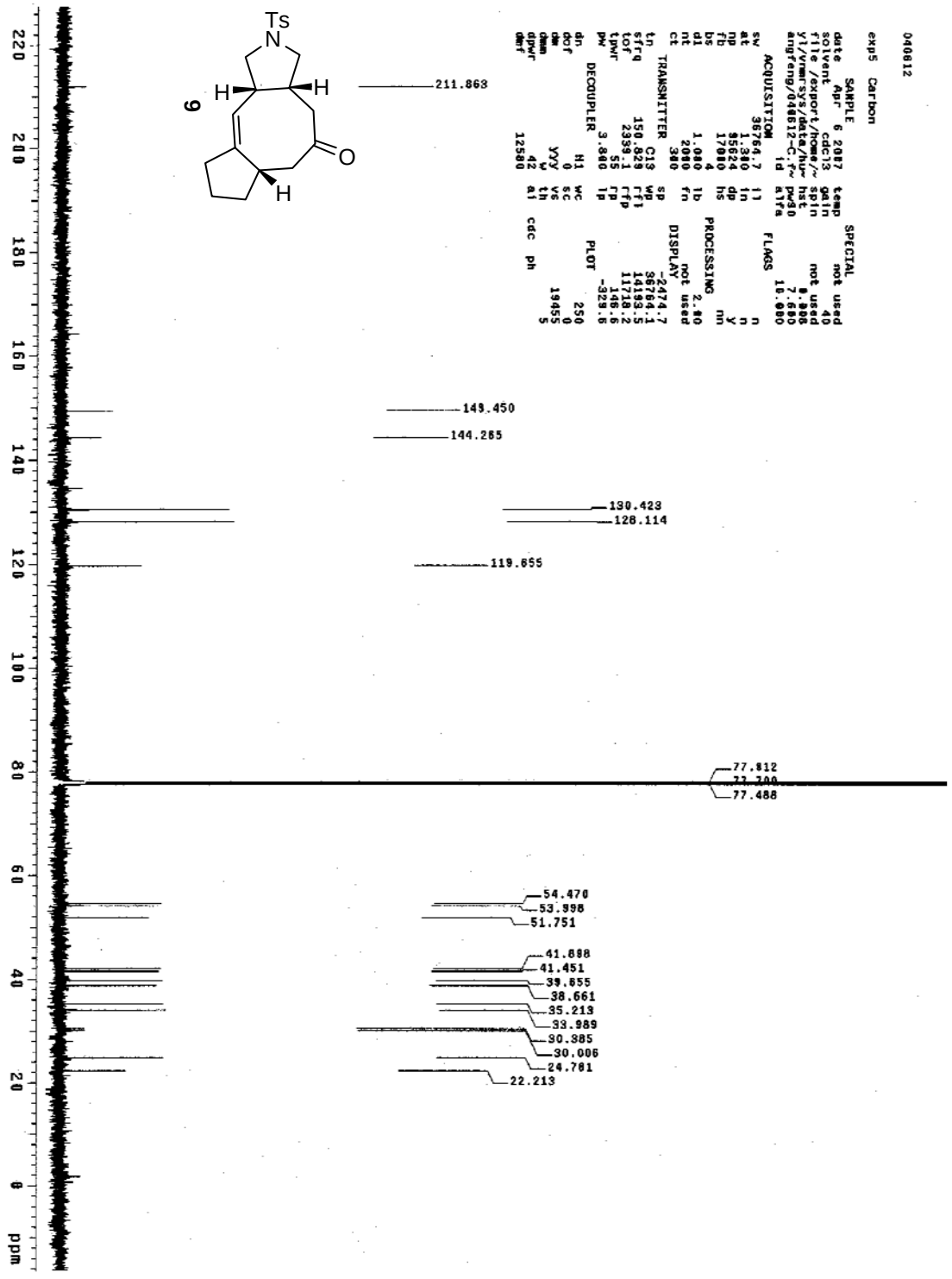


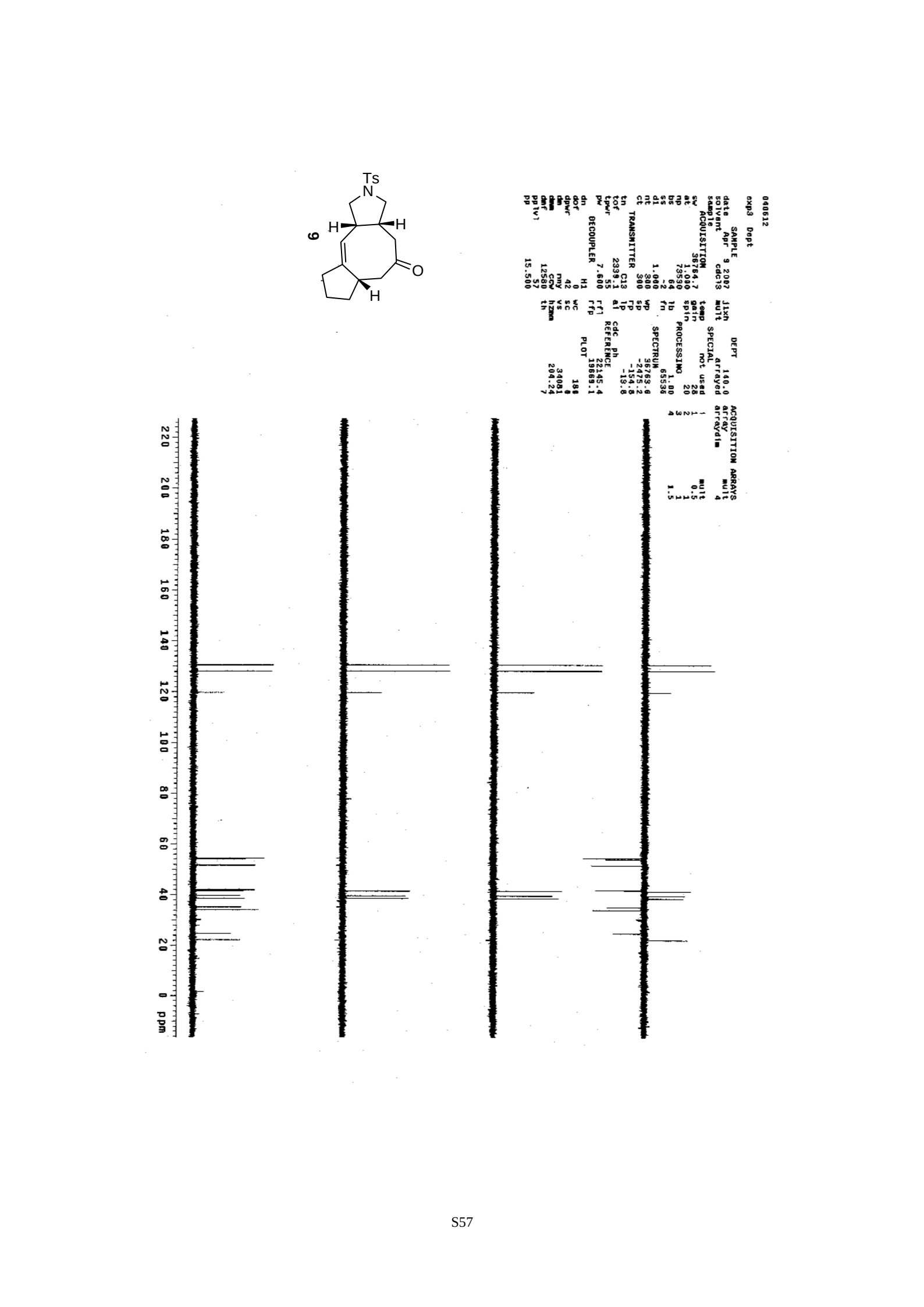
NAME	noesy
EXPNO	119
PROCNO	1
Date_	20091117
Time_	8.39
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	noesygpph
TD	2048
SOLVENT	CDCl3
NS	4
DS	16
SWH	6613.757 Hz
FIDRES	3.229373 Hz
AQ	0.1549544 sec
RG	32
DW	75.600 usec
DE	6.50 usec
TE	293.7 K
D0	0.00006465 sec
D1	1.50000000 sec
D8	0.80000001 sec
D16	0.00020000 sec
IN0	0.00015120 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	8.60 usec
P2	17.20 usec
PL1	-1.00 dB
PL1W	31.62277603 W
SFO1	600.1330006 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPZ1	40.00 %
GPZ2	-40.00 %
P16	1000.00 usec
ND0	1
TD	256
SFO1	600.133 MHz
FIDRES	25.835022 Hz
SW	11.021 ppm
FnMODE	States-TPPI
SI	1024
SF	600.1300000 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	States-TPPI
SF	600.1300000 MHz
WDW	QSINE
SSB	2
LB	0.00 Hz
GB	0

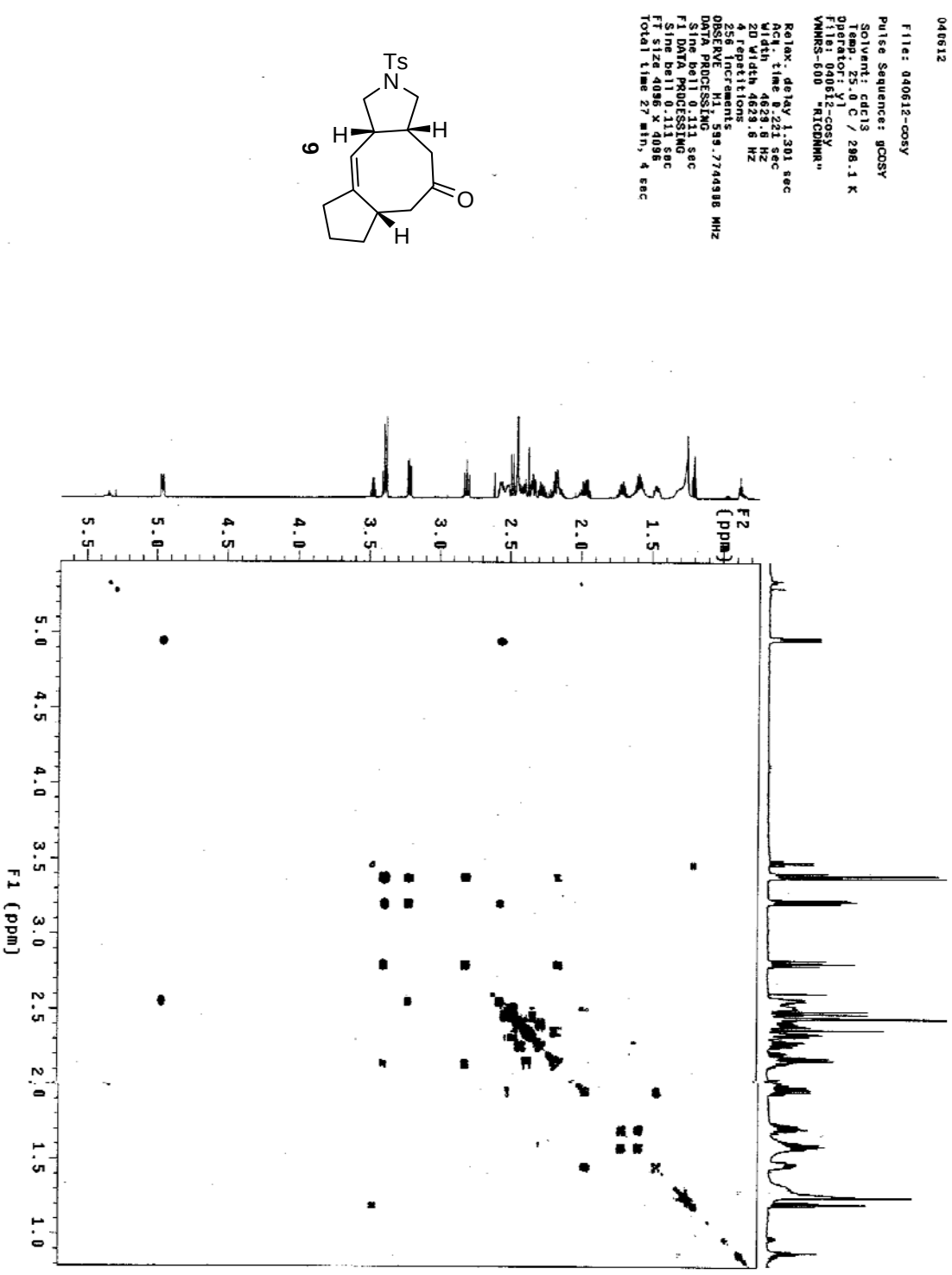

Ts
N
8
HF-05A
File: HF-05A-20071211-H
Pulse Sequence: s2pul
7.712
7.703
7.692
7.689
7.290
7.277
7.251
7.250
7.245
7.238
7.232
5.644
5.628
5.615
5.598
5.409
5.383
5.299
5.157
5.146
5.141
5.138
5.136
5.132
5.120
5.115
5.112
5.109
5.061
5.012
5.011
3.809
3.788
3.769
3.778
3.766
3.754
2.424
2.402
1.731
1.724
1.718
1.711
1.704
1.697
1.684
1.677
1.664
1.646
1.641
1.626
1.616
1.603
1.596
1.591
1.583
1.577
1.563
1.550
1.282
1.270
1.255
1.213
1.208
1.200
1.193
1.186
1.179
1.172
1.165
1.154
0.881
0.871
0.860
0.857
0.845
0.646
0.638
0.631
0.489
0.481
0.476
0.467
0.069
-0.000
2.34
2.00
0.85
0.90
0.45
3.72
2.59
6.84
2.38
2.78
2.60
1.48
0.92
0.94
ppm

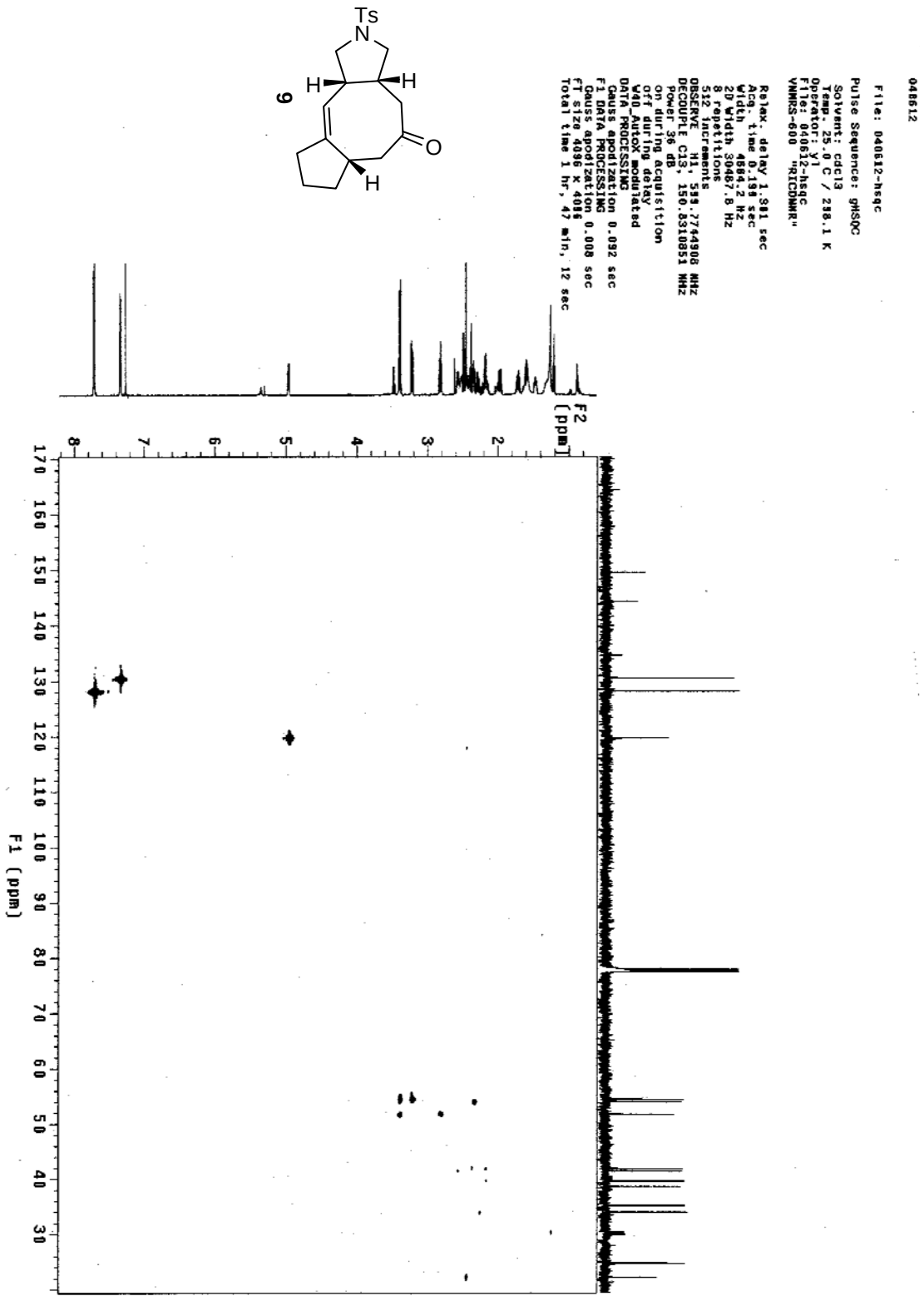

HF-05A
exp6 Carbon
SAMPLE
date Dec 11 2007
solvent cdcl3
file /export/home/~
zjy/vnmrsys/data/h~
wangfeng/HF-05A-20~
0/1211-C.fid
ACQUISITION
sw 36764.7
at 1.500
np 55004
fb 17004
bs 60
bs 4
nt 2000
ct 512
TRANSMITTER
tn C13
sfrq 150.829
tof 2339.1
tpwr 55
pw 3.800
DECOUPLER
dn H1
dof 0
dm yyy
dmm wc
dmf 12580
temp
gain
spin
hst
pw90
alfa
FLAGS
PROCESSING
lb
fn
DISPLAY
sp
wp
rfl
rfp
rp
lp
PLOT
wc
sc
vs
th
cdc
ph
al
Ts
N
8
142.892
141.028
133.049
129.541
127.254
115.360
118.584
77.212
77.000
76.788
49.013
48.823
30.243
29.142
27.211
26.453
21.476
20.825
14.404
220
200
180
160
140
120
100
80
60
40
20
0
ppm

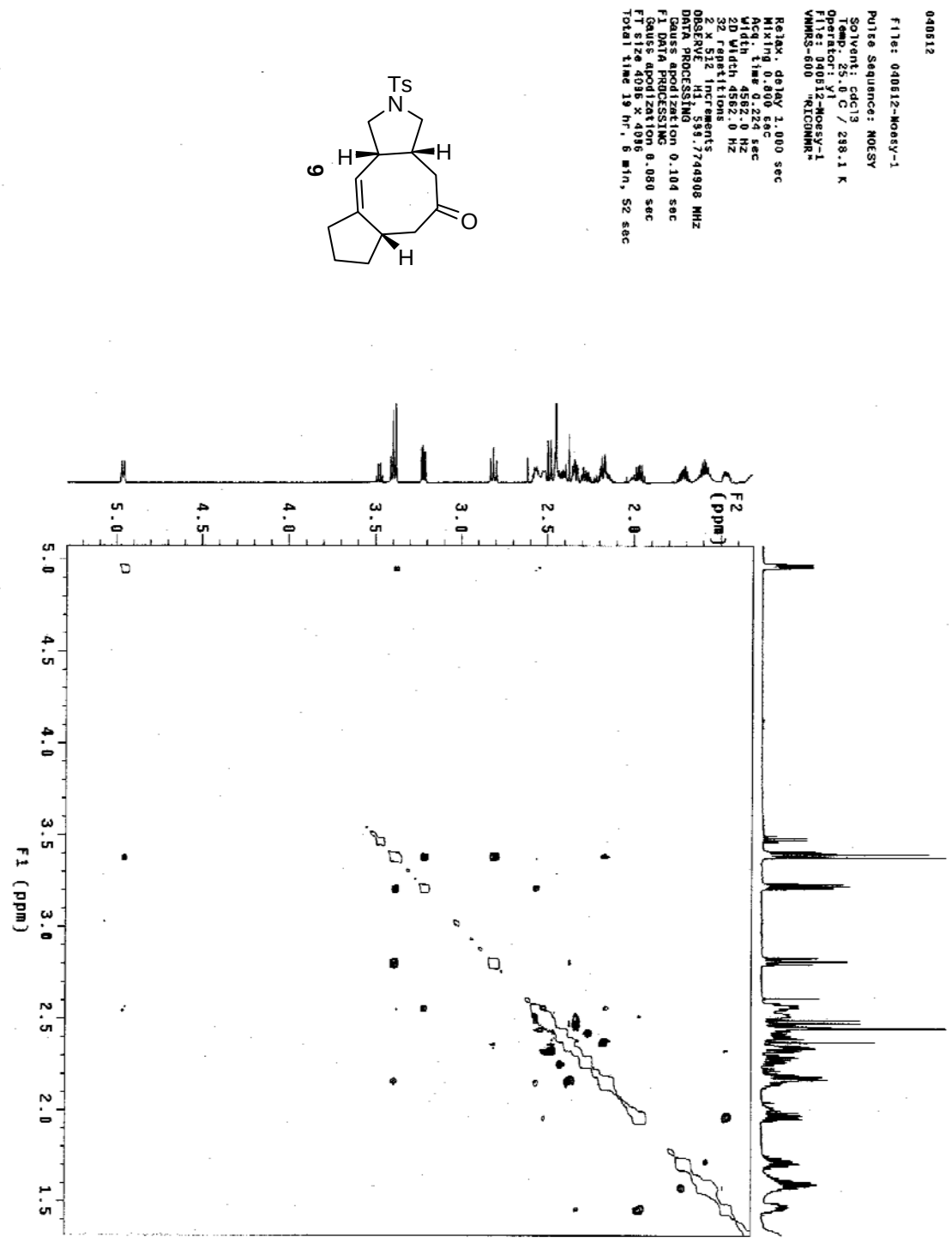

Ts
N
H
H
H
O
9
7.725
7.712
7.347
7.333
7.282
3.398
3.382
2.453
0.068
0.000
ppm

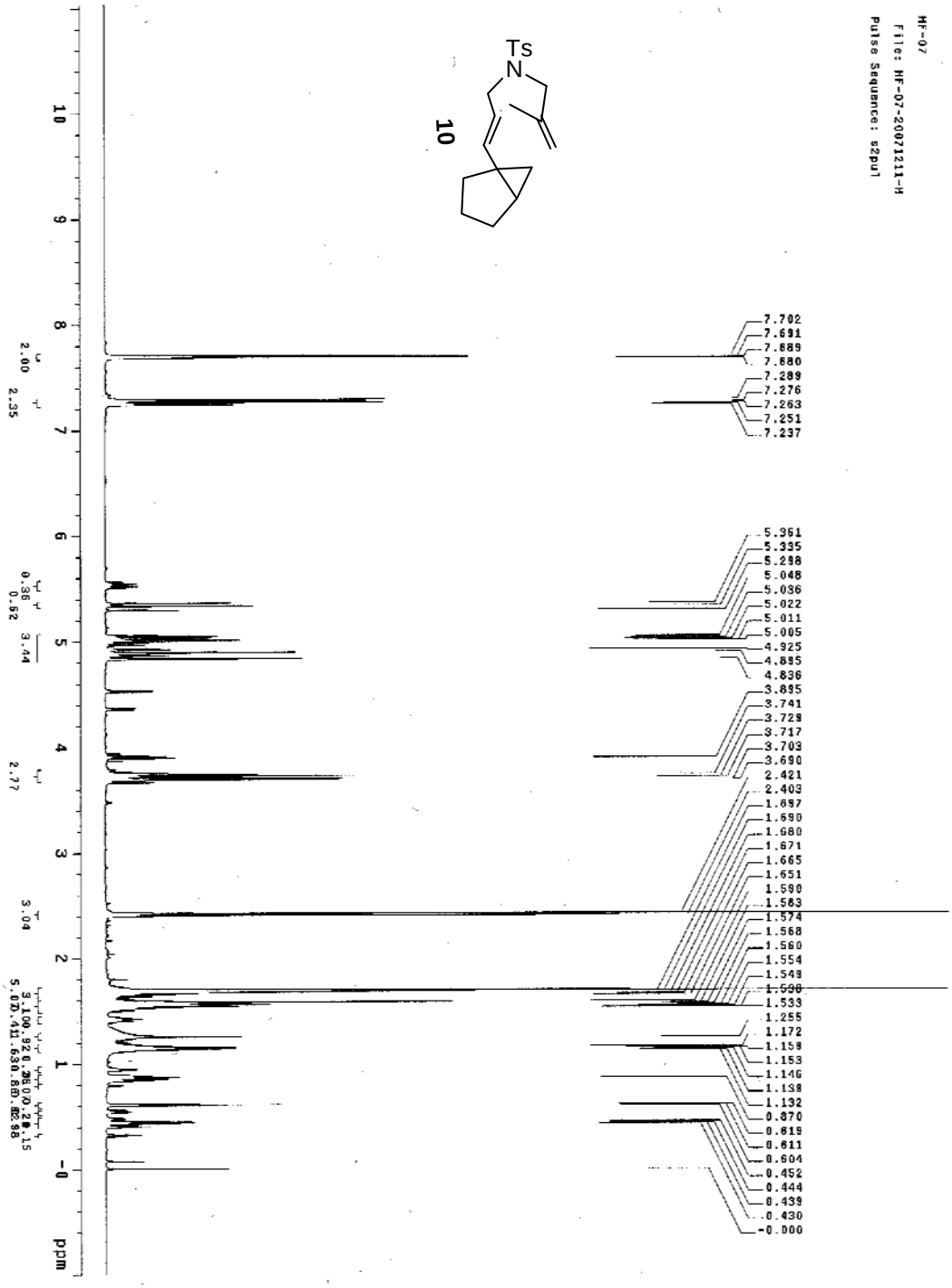

040812
exp5 Carbon
SAMPLE
date Apr 6 2007
solvent cdcl3
file /export1/home/...
yl/vnmrsys/data/hu...
angfeng/040812-C-f...
SPECIAL
temp not used
gain 40
spin not used
hst 8.896
pw90 7.680
id 10.000
ACQUISITION
FLAGS
PROCESSING
DISPLAY
DECOUPLER
PLOT
Ts
N
H
H
H
O
9
211.863
149.450
144.285
130.423
128.114
119.655
77.812
77.488
54.470
53.998
51.751
41.888
41.451
39.655
38.661
35.213
33.989
30.385
30.006
24.781
22.213
ppm

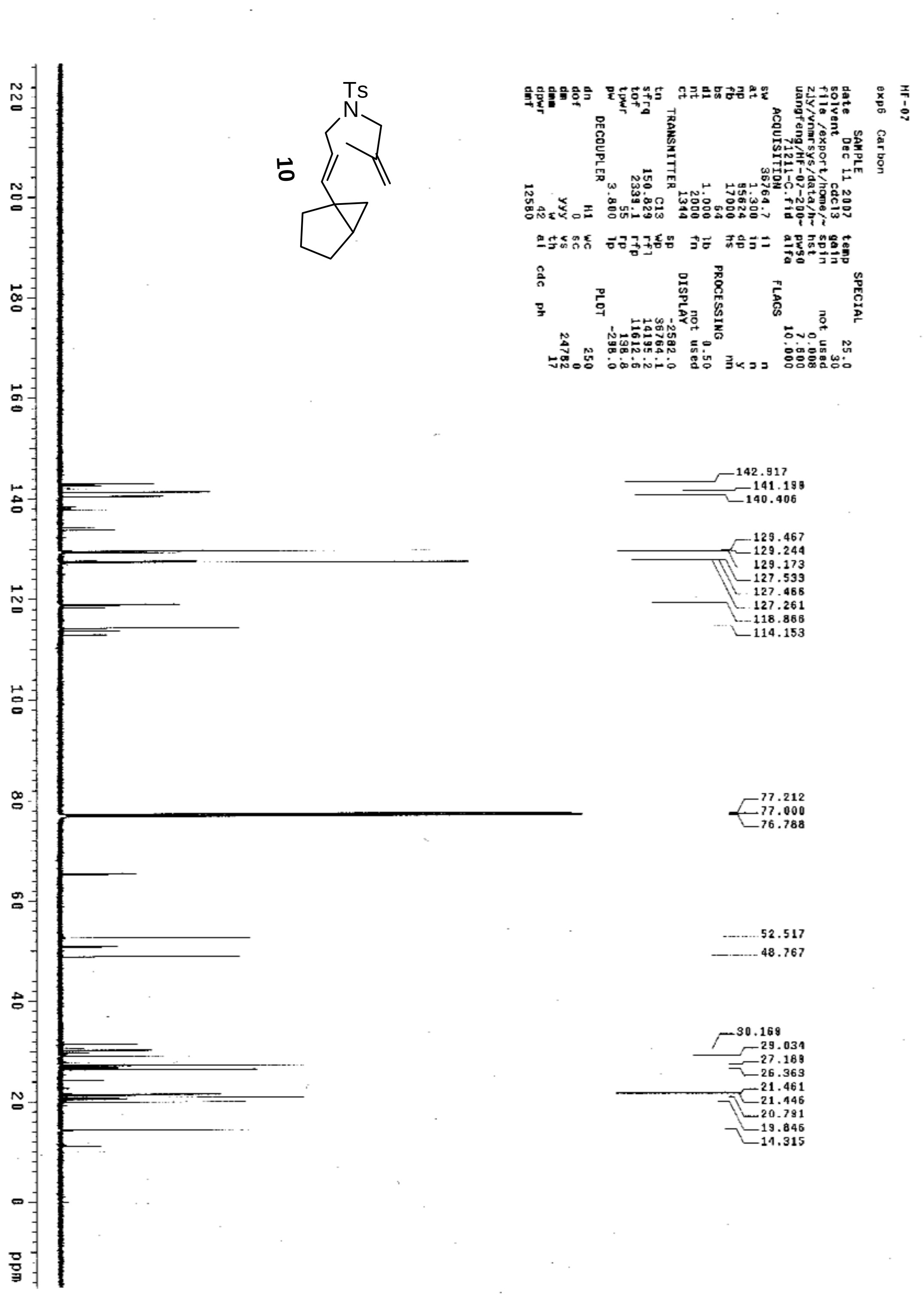

040612
exp3 Dept
SAMPLE
date Apr 9 2007
solvent cdcl3
sample
ACQUISITION
sw 36764.7
at 1.000
np 73530
bs 64
ss -2
d1 1.000
nt 300
ct 300
TRANSMITTER
tn C13
tof 2339.1
tpwr 55
pw 7.600
DECOUPLER
dn H1
dof 0
dpwr 42
dm nny
dmm ccw
dmf 12580
pplvl 57
pp 15.500
jlxh DEPT 140.0
mult SPECIAL arrayed
temp not used 28
gain 20
spin
PROCESSING 1.00
lb 65536
fn SPECTRUM 36763.6
wp -2475.2
sp -154.8
rp -19.8
lp
al
cdc ph
REFERENCE 22145.4
rf1 19669.1
rfp PLOT 188
wc 0
sc 34081
vs 204.24
hzmm 7
th
ACQUISITION ARRAYS
array mult
arraydim 4
mult
1 0.5
1 1
1
1.5
Ts
N
H
H
H
O
9
220 200 180 160 140 120 100 80 60 40 20 0 ppm

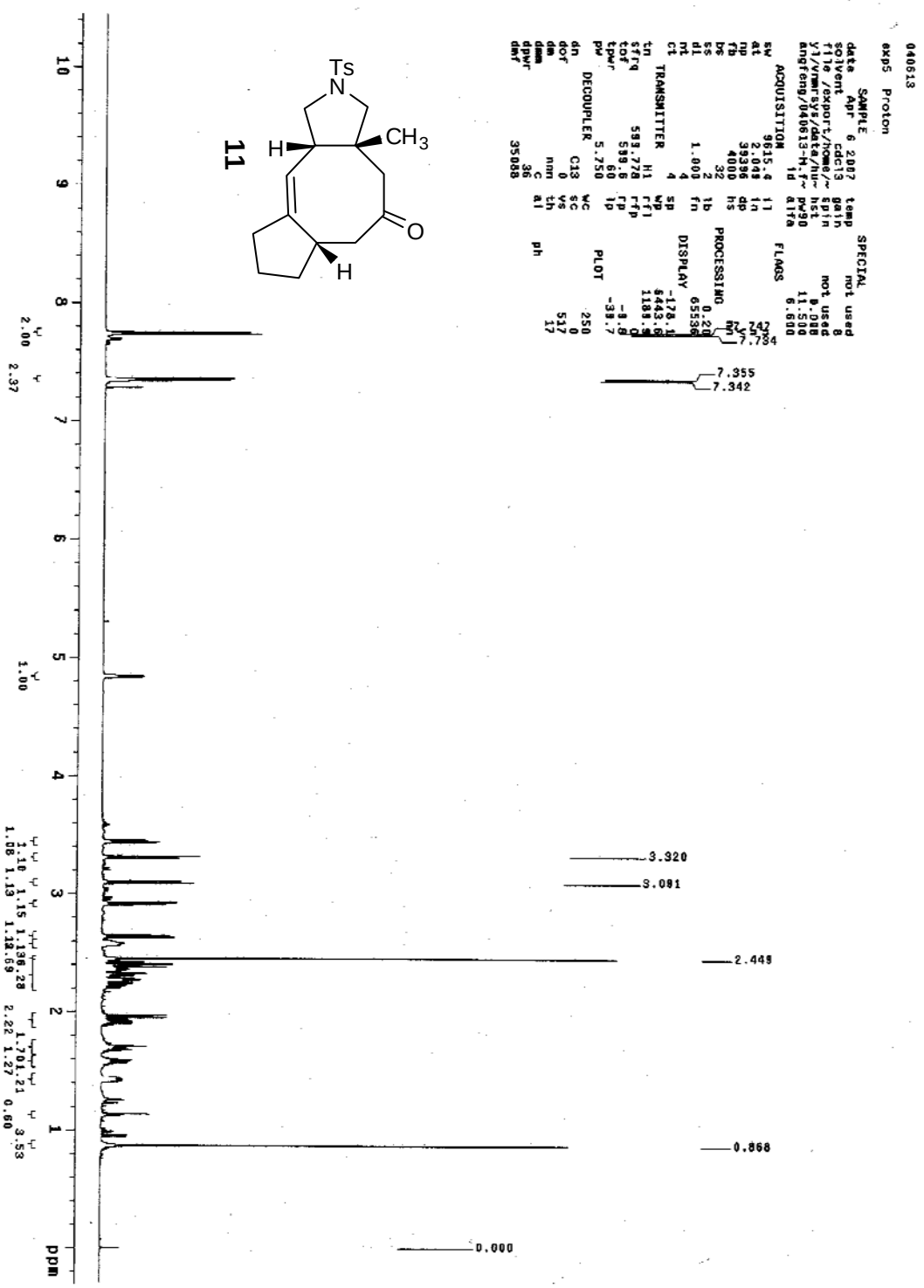

040612
File: 040612-cosy
Pulse Sequence: gCOSY
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Yl
File: 040612-cosy
VNMRS-600 "RICDNMR"
Relax. delay 1.301 sec
Acq. time 0.221 sec
Width 4628.8 Hz
2D Width 4628.8 Hz
4 repetitions
256 increments
OBSERVE H1 599.7744198 MHz
DATA PROCESSING
Sine bell 0.111 sec
F1 DATA PROCESSING
Sine bell 0.111 sec
FT size 4096 x 4096
Total time 27 min, 4 sec
Ts
N
H
H
H
O
9
F2 (ppm)
F1 (ppm)

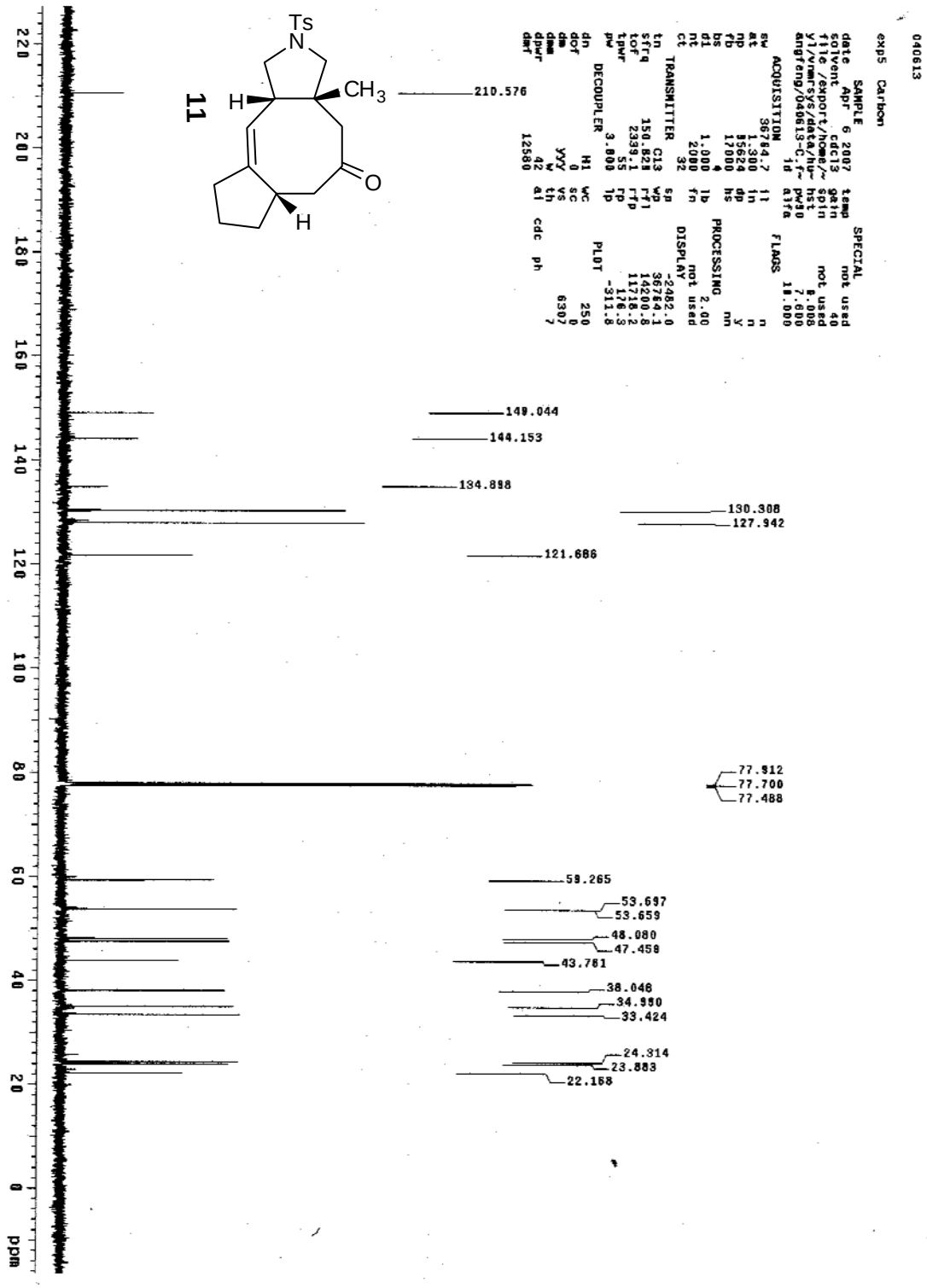

Ts
N
H
H
O
H
9
040612
File: 040612-hsqc
Pulse Sequence: gHSQC
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: Yl
File: 040612-hsqc
VNMRS-600 "RICDNMR"
Relax. delay 1.981 sec
Acq. time 0.198 sec
Width 4884.2 Hz
2D Width 30487.8 Hz
8 repetitions
512 increments
OBSERVE H1, 599.7744908 MHz
DECOUPLE C13, 150.8310851 MHz
Power 36 dB
on during acquisition
off during delay
W40_AutoX modulated
DATA PROCESSING
Gauss apodization 0.092 sec
F1 DATA PROCESSING
Gauss apodization 0.008 sec
FT size 4096 x 4016
Total time 1 hr, 47 min, 12 sec
F2 (ppm)
F1 (ppm)

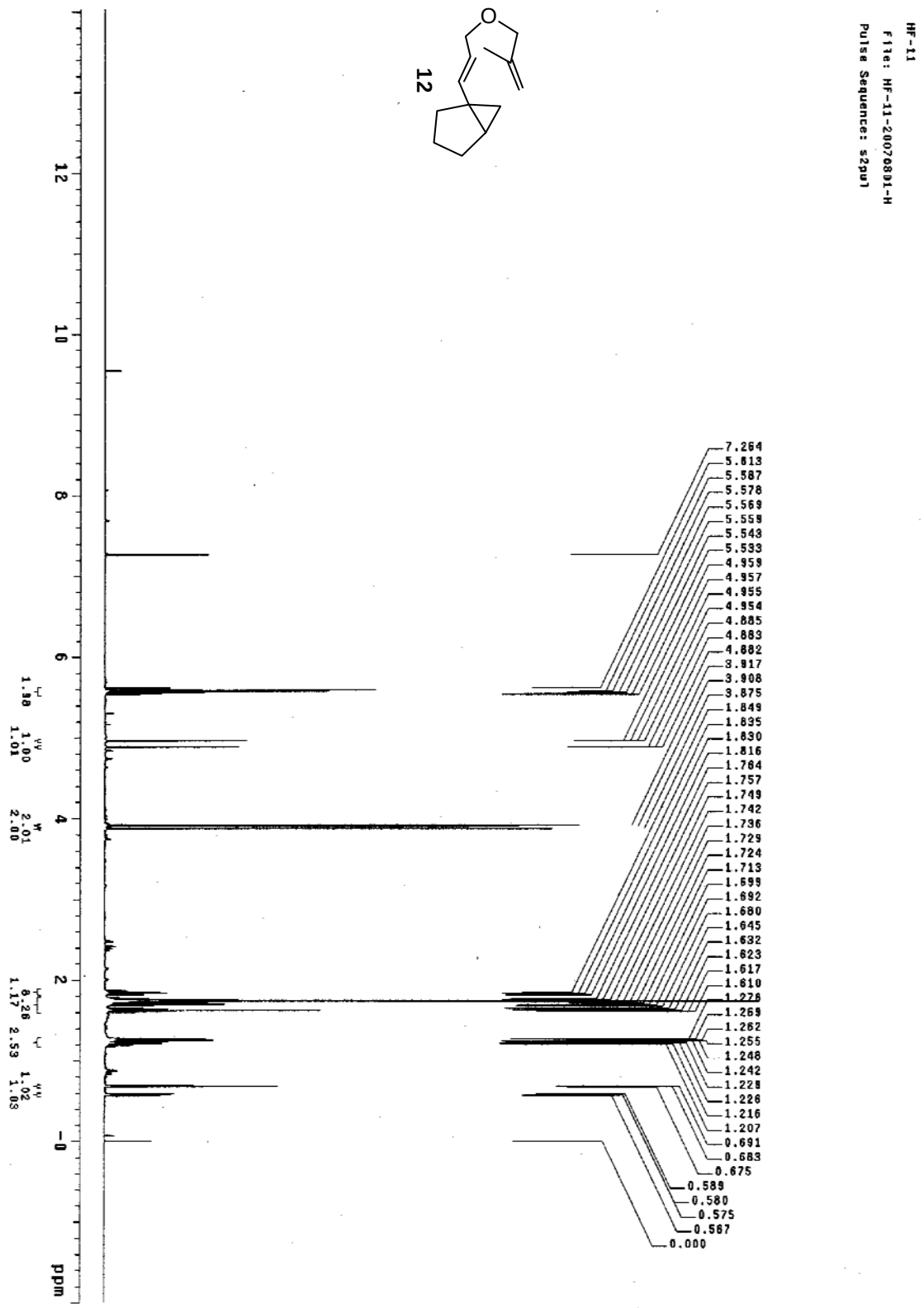

040612
File: 040612-Noesy-1
Pulse Sequence: NOESY
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: yl
File: 040612-Noesy-1
VNMRS-600 "RICONMR"
Relax. delay 1.000 sec
Mixing time 0.800 sec
Acq. time 0.224 sec
Width 4562.0 Hz
2D Width 4562.0 Hz
32 repetitions
2 x 512 increments
OBSERVE H1, 599.7744908 MHz
DATA PROCESSING
Gauss apodization 0.104 sec
F1 DATA PROCESSING
Gauss apodization 0.080 sec
FT size 4096 x 4096
Total time 19 hr, 6 min, 52 sec
Ts
N
H
H
O
H
9
F2 (ppm)
F1 (ppm)

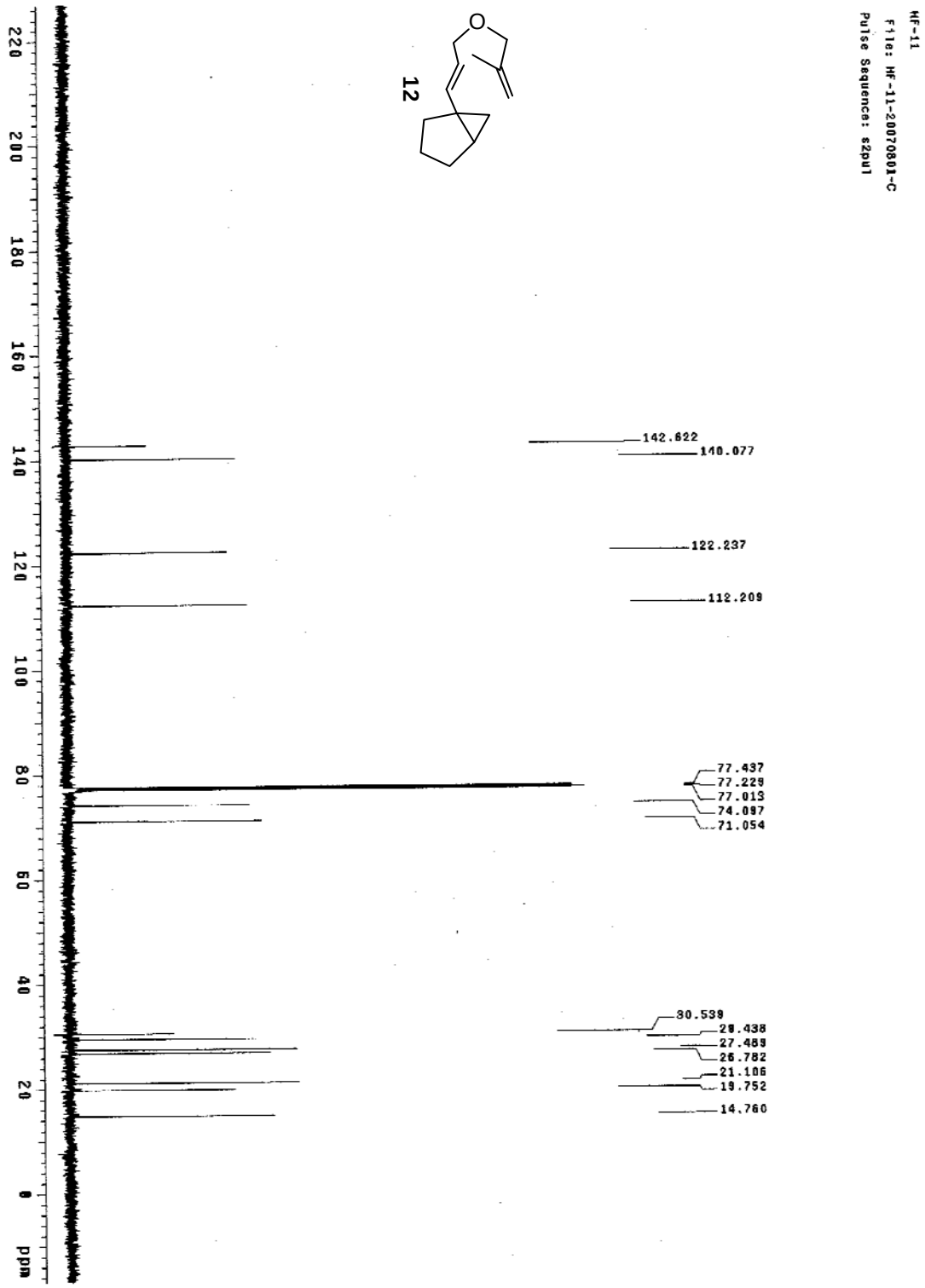

HF-07
File: HF-07-20071211-H
Pulse Sequence: s2pul
Ts
N
10
7.702
7.691
7.689
7.680
7.289
7.276
7.263
7.251
7.237
5.361
5.335
5.298
5.048
5.036
5.022
5.011
5.005
4.925
4.895
4.836
3.895
3.741
3.729
3.717
3.703
3.690
2.421
2.403
1.697
1.690
1.680
1.671
1.665
1.651
1.590
1.583
1.574
1.568
1.560
1.554
1.548
1.538
1.533
1.255
1.172
1.159
1.153
1.146
1.138
1.132
0.870
0.619
0.611
0.604
0.452
0.444
0.439
0.430
-0.000
2.00
2.35
0.36
0.52
3.44
2.77
3.04
ppm

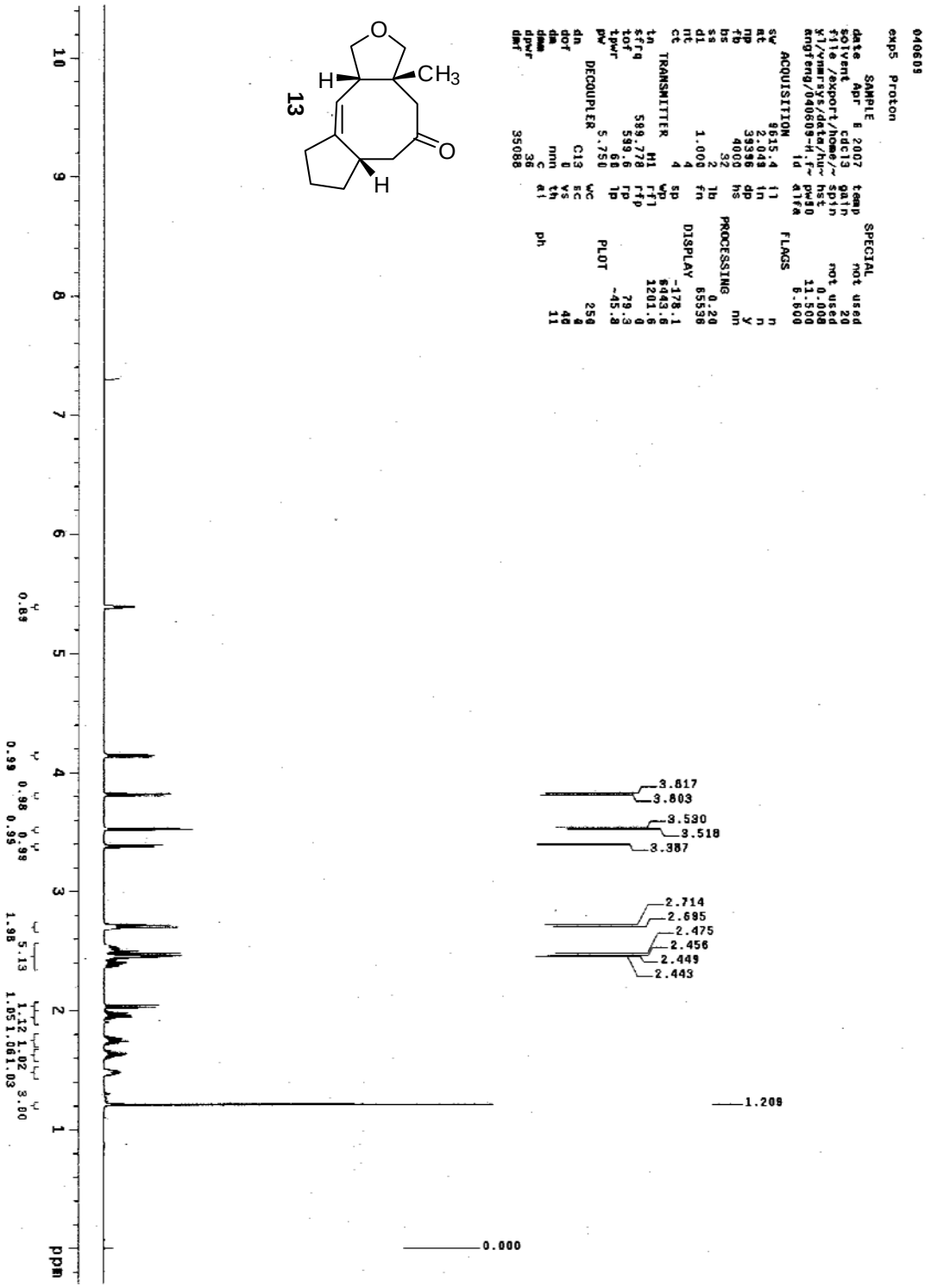

Ts
N
10
142.917
141.198
140.406
129.467
129.244
129.173
127.533
127.466
127.261
118.866
114.153
77.212
77.000
76.788
52.517
48.767
30.169
29.034
27.189
26.363
21.461
21.446
20.791
19.846
14.315
ppm

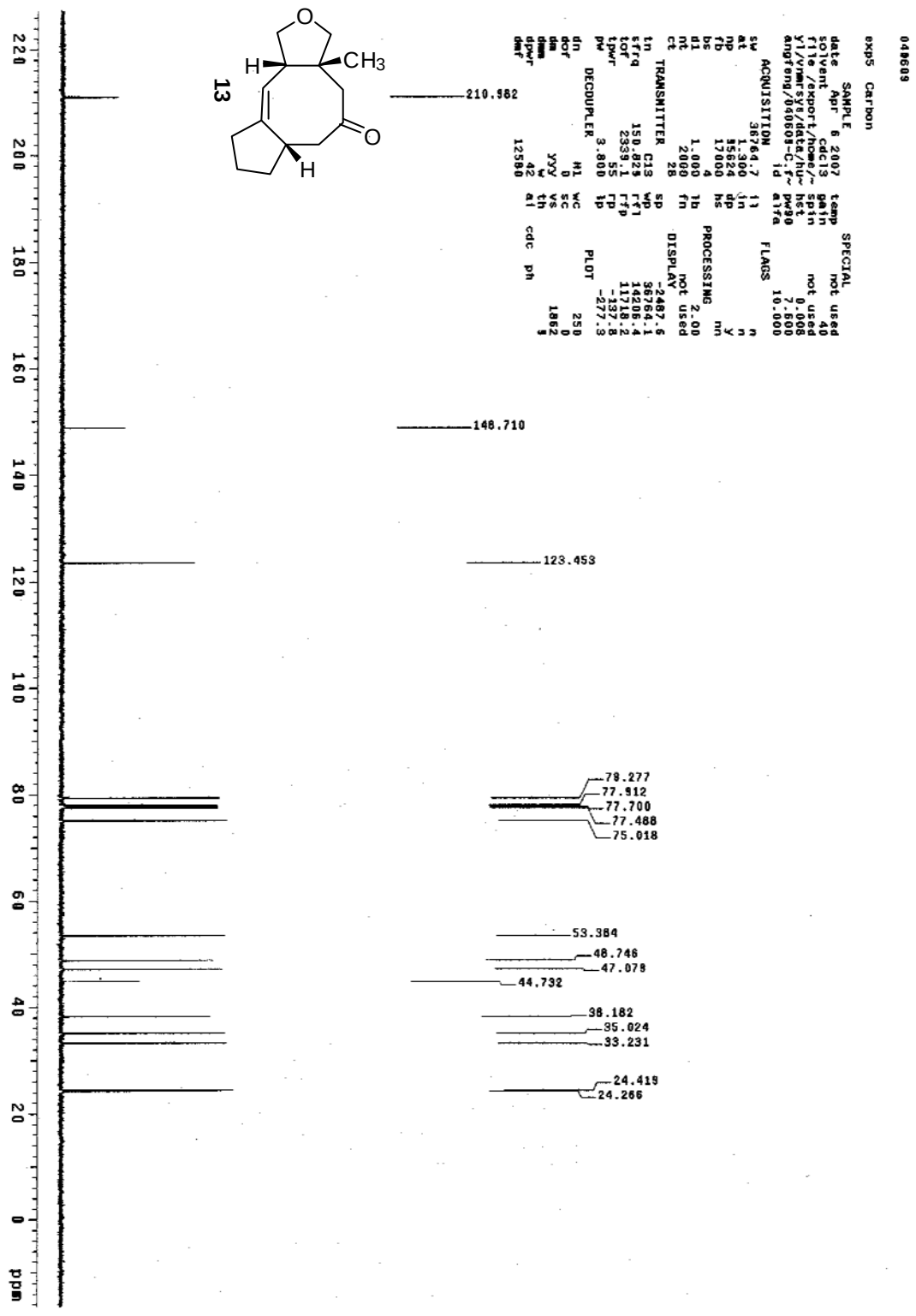

040813
exp5 Proton
11
Ts
N
H
CH3
H
O
7.784
7.355
7.342
3.320
3.091
2.448
0.868
0.000
ppm


11
Ts
N
CH3
H
H
O
210.576
149.044
144.153
134.888
130.308
127.942
121.686
77.912
77.700
77.488
59.265
53.697
53.659
48.080
47.458
43.761
38.046
34.950
33.424
24.314
23.883
22.168
ppm


HF-11
File: HF-11-20070801-H
Pulse Sequence: s2pul
12
7.264
5.613
5.587
5.578
5.569
5.559
5.543
5.533
4.959
4.957
4.955
4.954
4.885
4.883
4.882
3.917
3.908
3.875
1.849
1.835
1.830
1.816
1.764
1.757
1.749
1.742
1.736
1.729
1.724
1.713
1.699
1.692
1.680
1.645
1.632
1.623
1.617
1.610
1.276
1.269
1.262
1.255
1.248
1.242
1.228
1.226
1.216
1.207
0.691
0.683
0.675
0.589
0.580
0.575
0.567
0.000
ppm


HF-11
File: HF-11-20070801-C
Pulse Sequence: s2pul
12
O
142.622
140.077
122.237
112.209
77.437
77.229
77.013
74.097
71.054
30.539
28.438
27.469
26.782
21.106
19.752
14.760
220
200
180
160
140
120
100
80
60
40
20
0
ppm

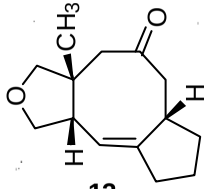

13
O
H
CH3
O
H
3.617
3.603
3.530
3.518
3.387
2.714
2.695
2.475
2.456
2.449
2.443
1.209
0.000
ppm


13
O
H
CH3
O
H
210.982
148.710
123.453
79.277
77.912
77.700
77.488
75.018
53.384
48.746
47.078
44.732
38.182
35.024
33.231
24.419
24.266
220
200
180
160
140
120
100
80
60
40
20
0
ppm

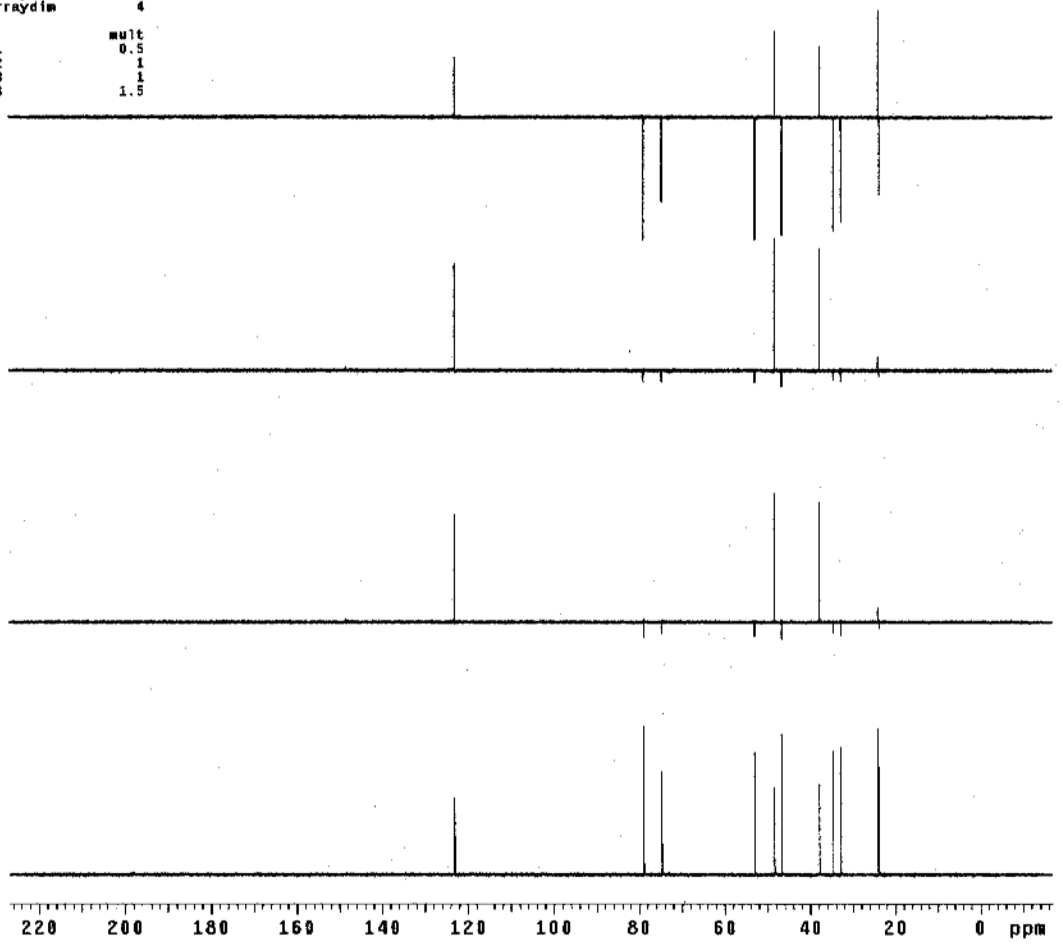
040609

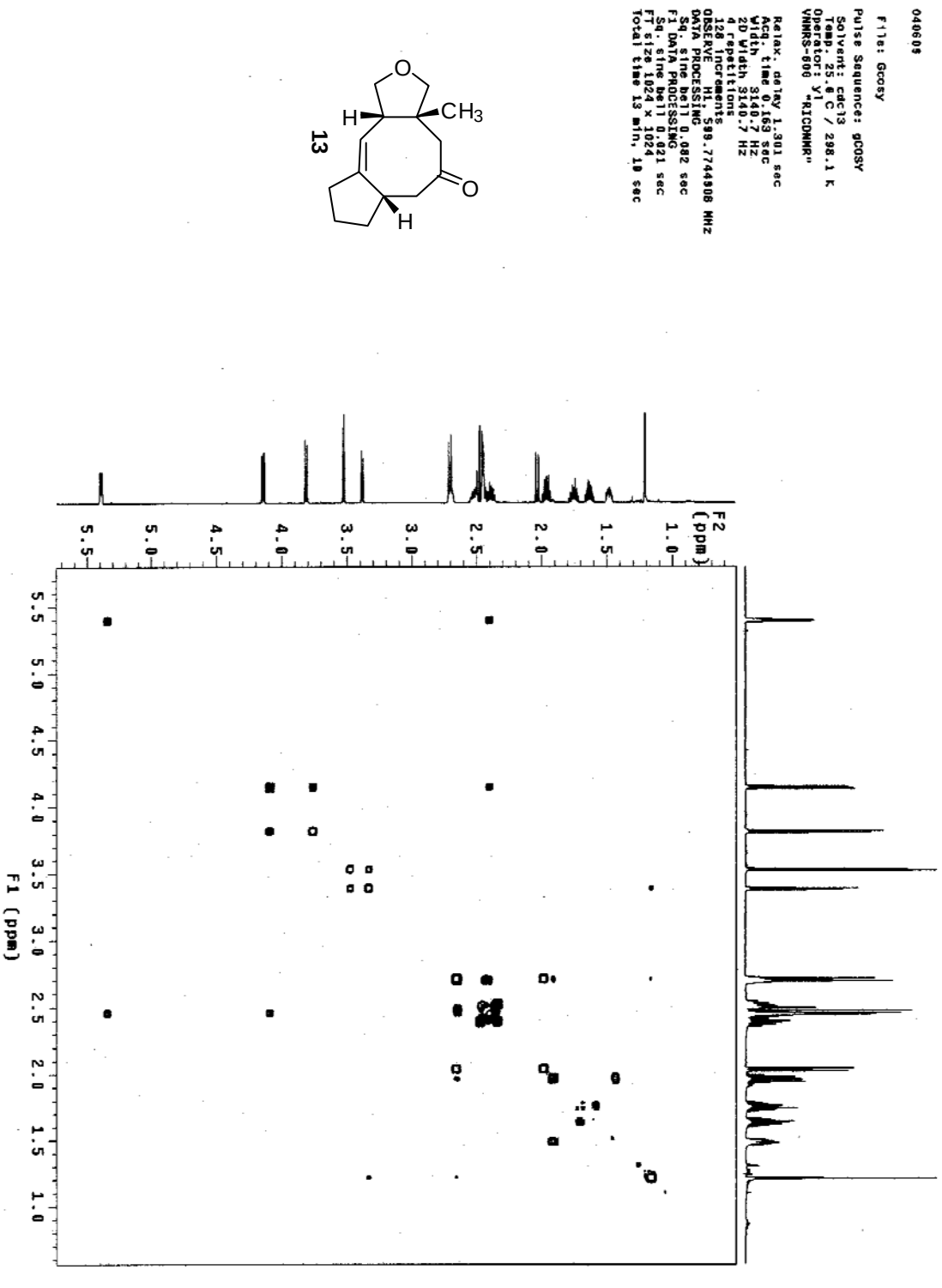
exp5 Dept

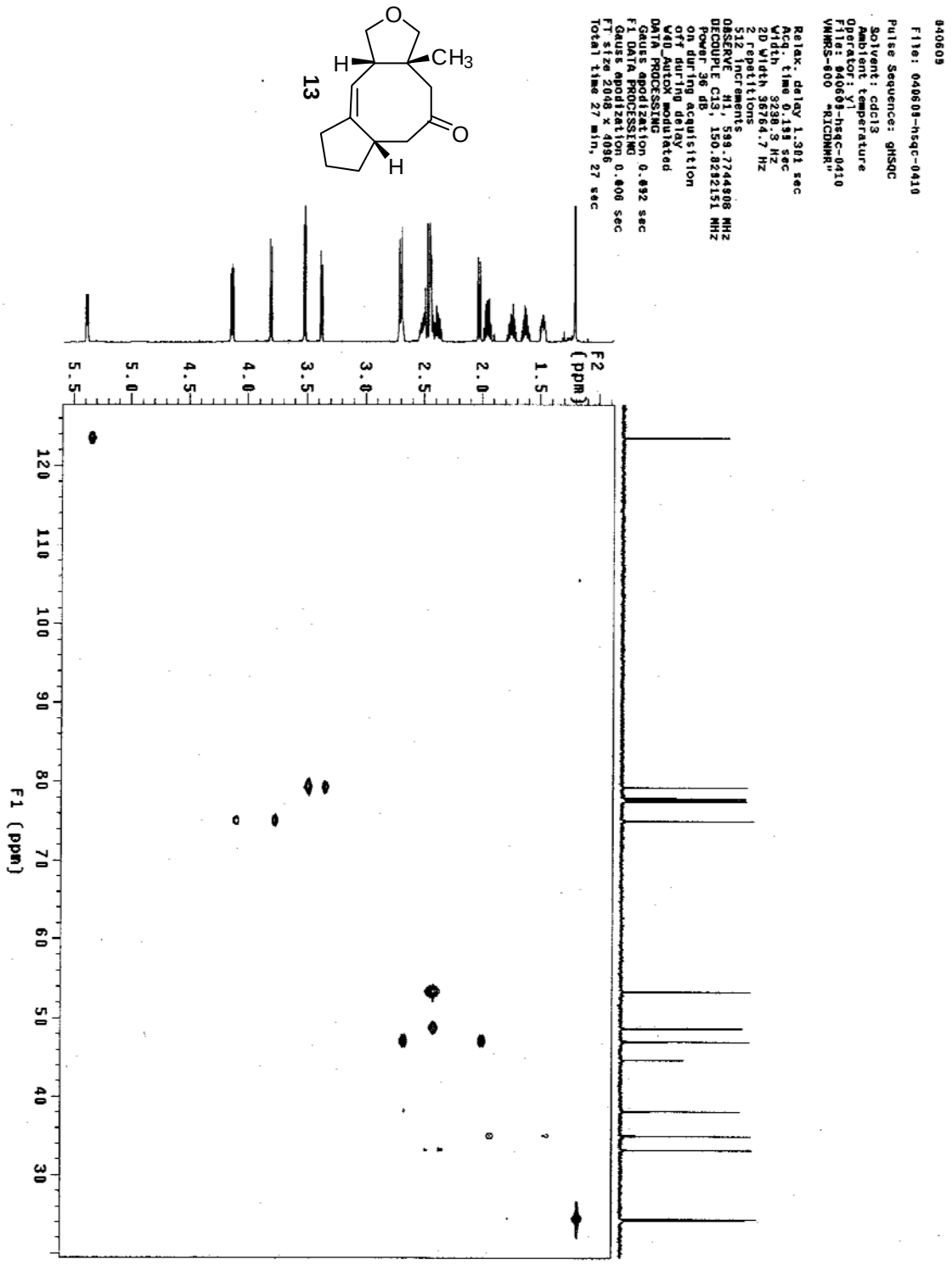
SAMPLE		DEPT		ACQUISITION ARRAYS	
date	Apr 9 2007	jlxh	140.0	array	mult
solvent	cdcl3	mult	arrayed	arraydim	4
sample		SPECIAL			
ACQUISITION		temp	not used	i	mult
sw	36764.7	gain	28	1	0.5
at	1.000	spin	20	2	1
np	73530	PROCESSING		3	1
bs	64	lb	1.00	4	1.5
ss	-2	fn	65536		
d1	1.000	SPECTRUM			
nt	32	wp	36763.6		
ct	32	sp	-2545.8		
TRANSMITTER		rp	-114.6		
tn	C13	lp	37.6		
tof	2339.1	ai	cdc ph		
tpwr	55	REFERENCE			
pw	7.600	rfl	2546.9		
DECOUPLER		rfp	0		
dn	H1	PLOT			
dof	0	wc	180		
dpwr	42	sc	0		
dm	nny	vs	2800		
dmm	ccw	hzmm	204.24		
dmf	12580	th	8		
pplvl	57				
pp	15.500				

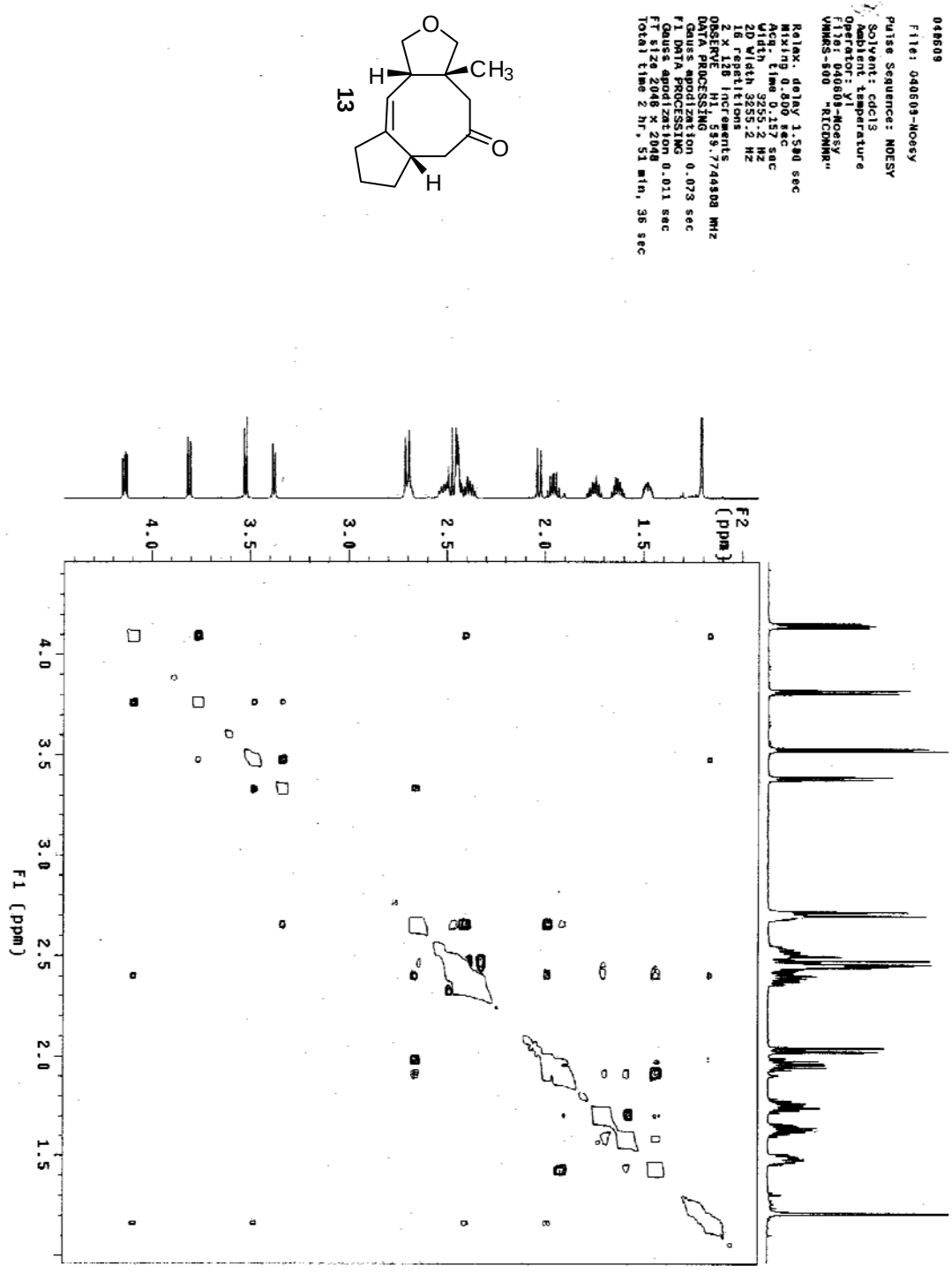


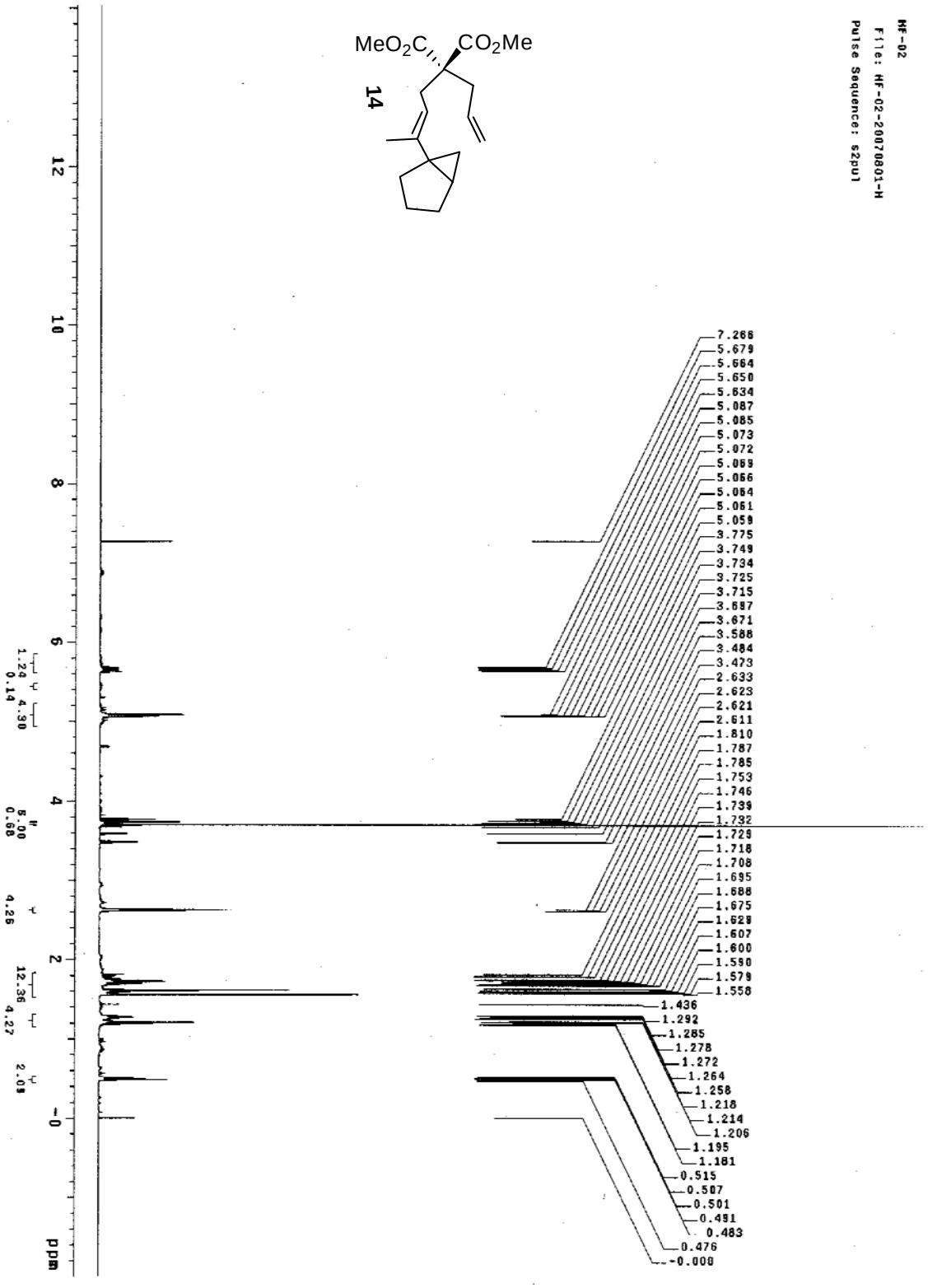
13

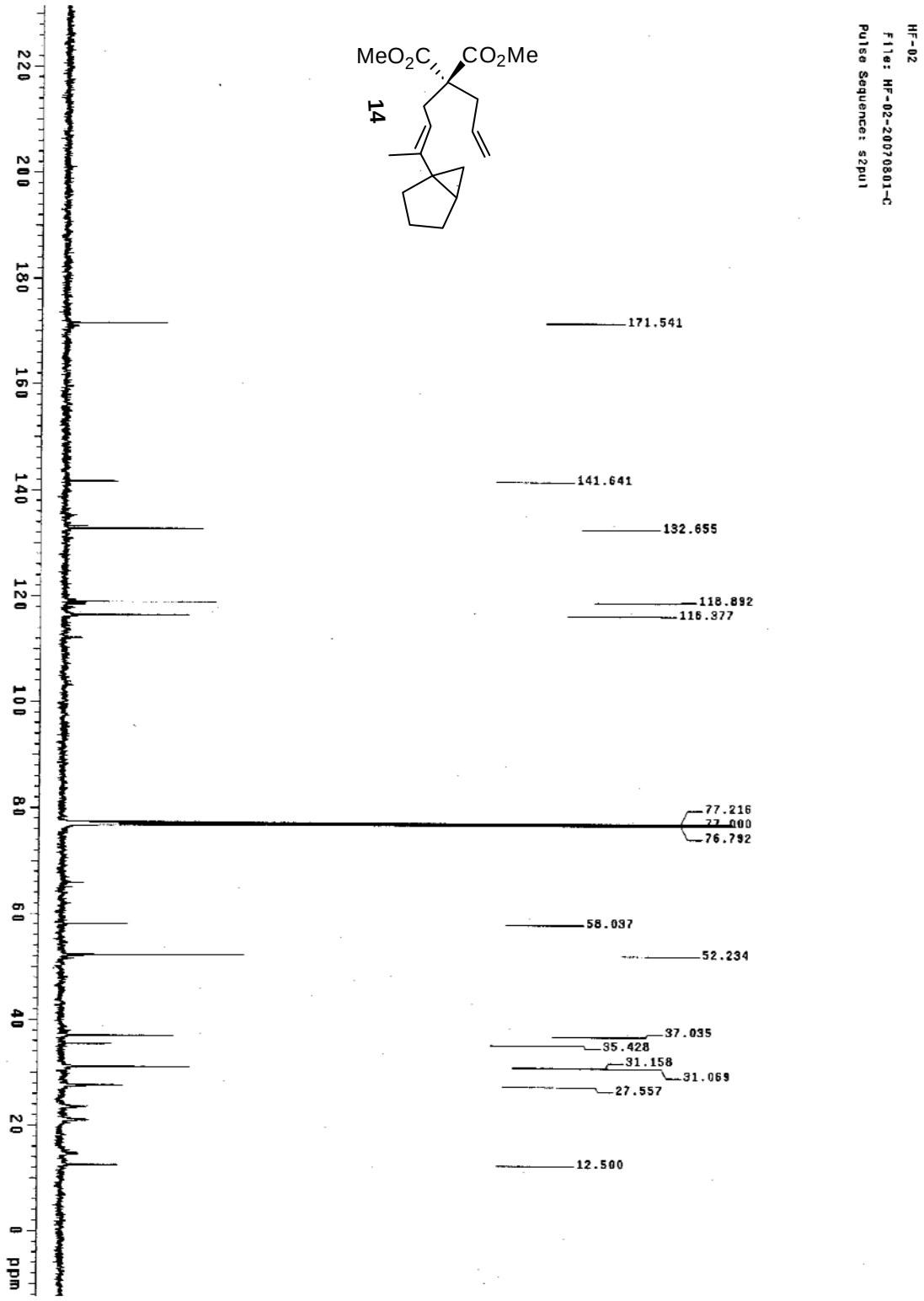


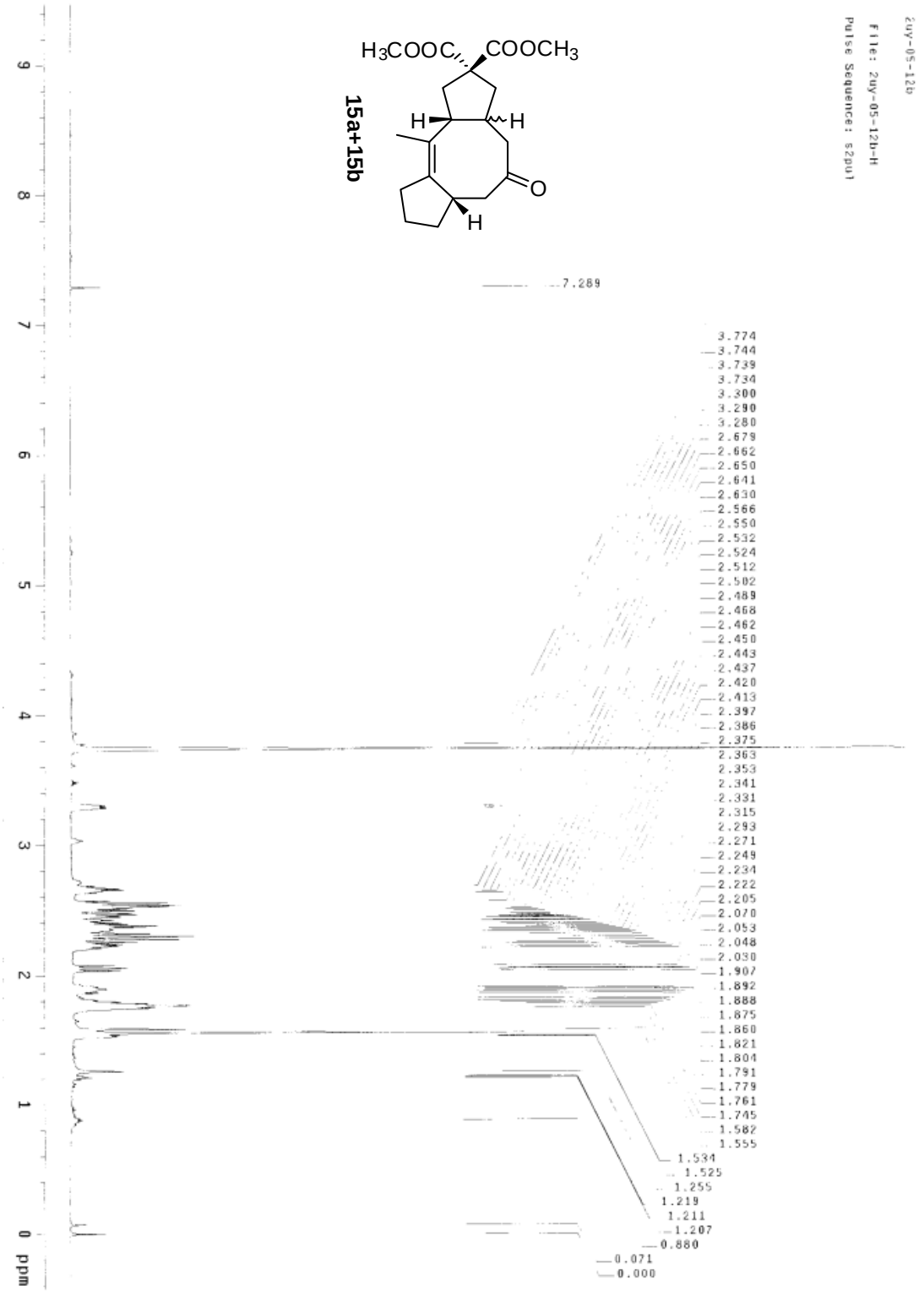

040609
File: Gcosy
Pulse Sequence: gCOSY
Solvent: cdcl3
Temp. 25.6 C / 298.1 K
Operator: yl
VNMRS-600 "RICDNMR"
Relax. delay 1.301 sec
Acq. time 0.169 sec
Width 5140.7 Hz
2D width 5140.7 Hz
4 transients
128 increments
OBSERVE H1, 599.7744908 MHz
DATA PROCESSING
Sq. sine bell 0.082 sec
F1 DATA PROCESSING
Sq. sine bell 0.021 sec
FT size 1024 x 1024
Total time 13 min, 10 sec
O
H
CH3
O
H
13
F2 (ppm)
F1 (ppm)

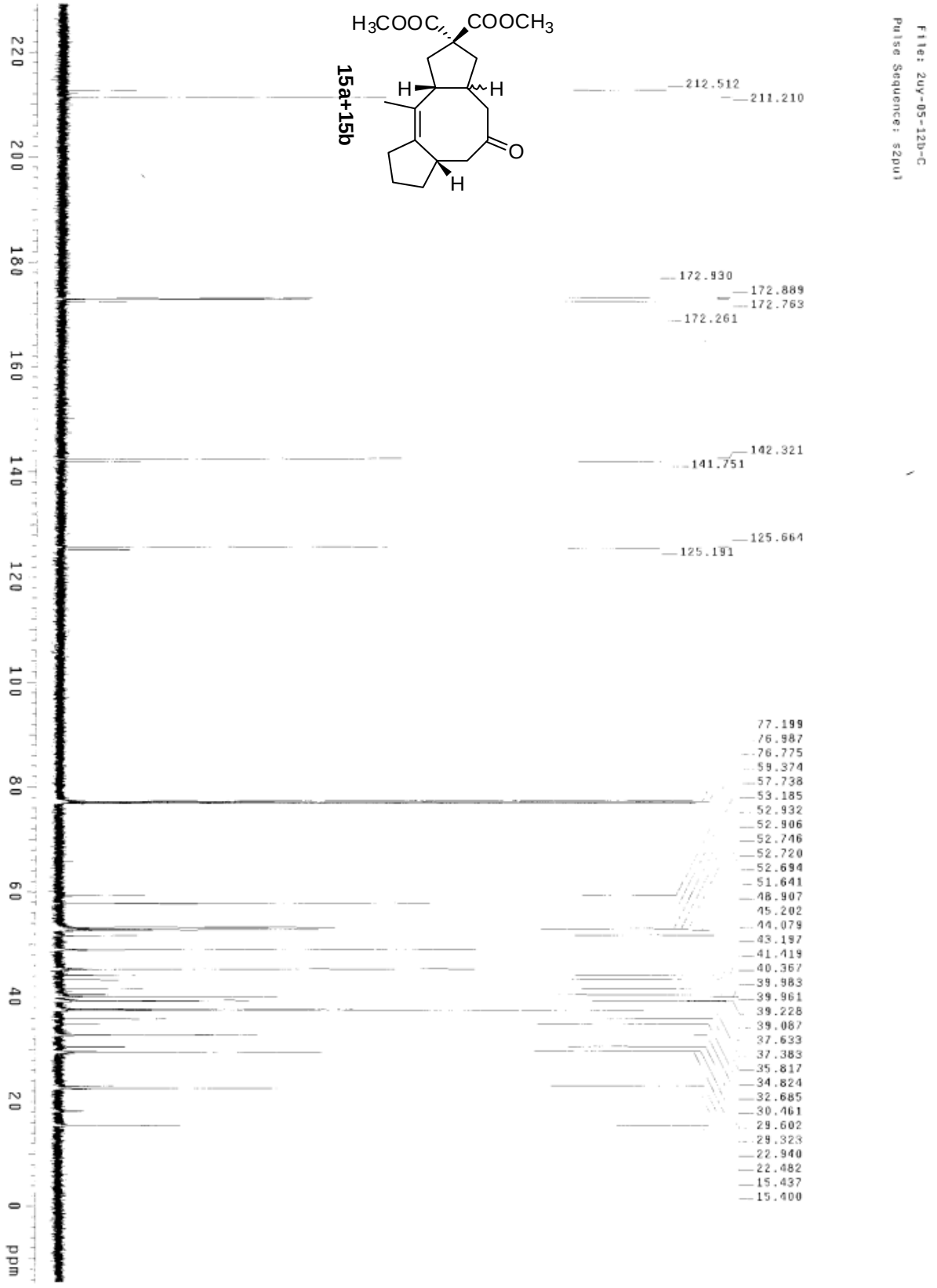

040609
File: 040609-hsqc-0410
Pulse Sequence: gHSQC
Solvent: cdcl3
Ambient temperature
operator:yl
File: 040609-hsqc-0410
VNMRS-600 "RICDNMR"
Relax. delay 1.301 sec
Acq. time 0.198 sec
Width 9238.3 Hz
2D Width 36764.7 Hz
2 repetitions
512 increments
OBSERVE H1, 599.7744808 MHz
DECOUPLE C13, 150.8282151 MHz
Power 36 dB
on during acquisition
off during delay
WALTZ modulated
DATA PROCESSING
Gauss apodization 0.892 sec
F1 DATA PROCESSING
Gauss apodization 0.800 sec
FT size 2048 x 4096
Total time 27 min, 27 sec
O
H
CH3
13
O
H
F2 (ppm)
F1 (ppm)

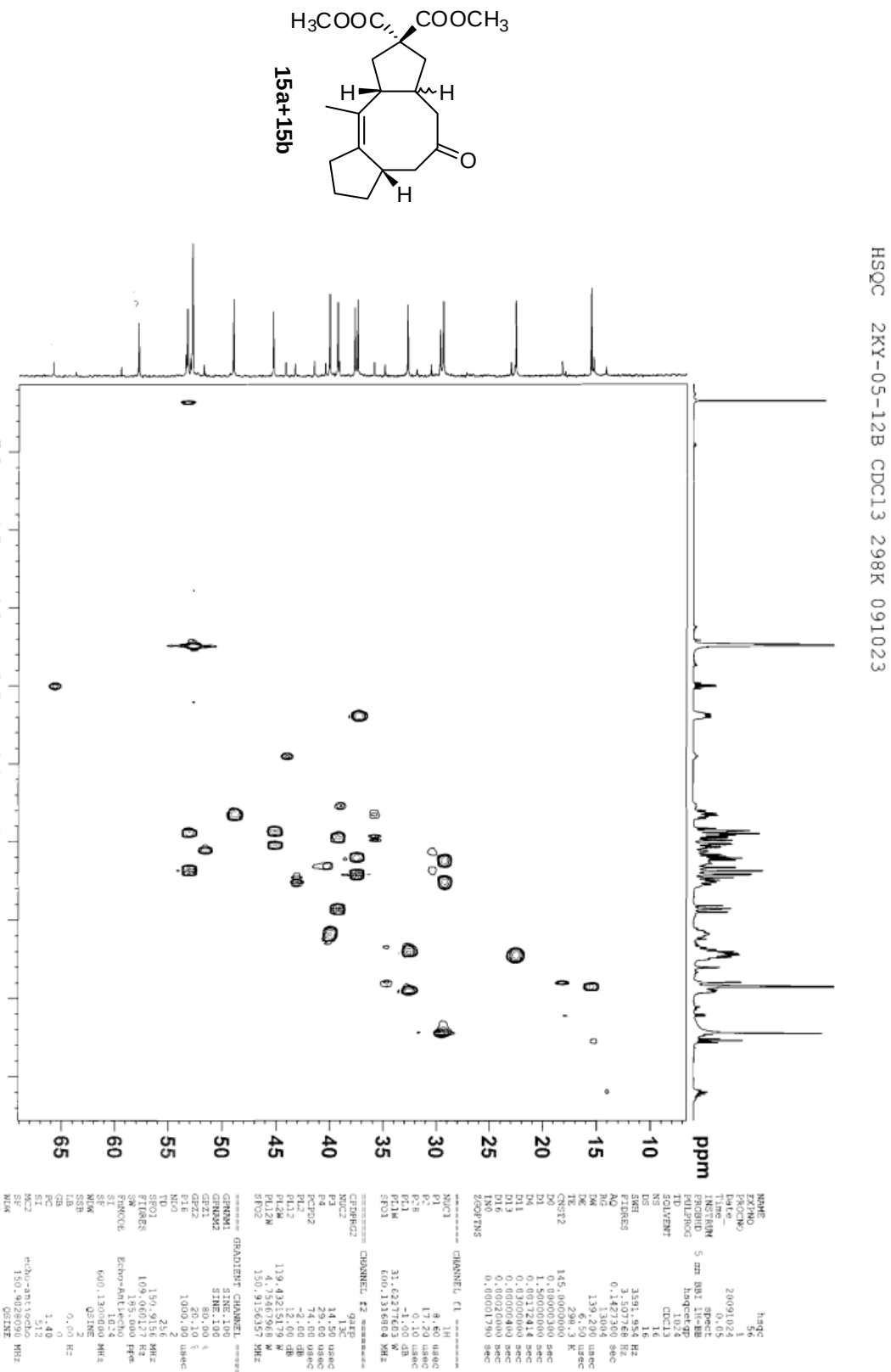

13
O
H
CH3
O
H
048609
File: 040609-Noesy
Pulse Sequence: NOESY
Solvent: cdcl3
Ambient temperature
Operator: yjl
File: 040609-Noesy
VNMRS-500 "RICNMR"
Relax. delay 1.500 sec
Mixing 0.800 sec
Acq. time 0.157 sec
Width 3255.2 Hz
2D Width 3255.2 Hz
16 repetitions
2 x 128 increments
OBSERVE H1 599.7744508 MHz
DATA PROCESSING
Gauss apodization 0.073 sec
F1 DATA PROCESSING
Gauss apodization 0.011 sec
FT size 2048 x 2048
Total time 2 hr, 51 min, 36 sec
F2 (ppm)
F1 (ppm)
4.0
3.5
3.0
2.5
2.0
1.5


MeO2C
CO2Me
14
MF-02
File: MF-02-20070801-H
Pulse Sequence: s2pul
7.268
5.679
5.664
5.650
5.634
5.087
5.085
5.073
5.072
5.069
5.066
5.064
5.061
5.059
3.775
3.749
3.734
3.725
3.715
3.687
3.671
3.588
3.484
3.473
2.633
2.623
2.621
2.611
1.810
1.787
1.785
1.753
1.746
1.739
1.732
1.728
1.718
1.708
1.695
1.688
1.675
1.629
1.607
1.600
1.590
1.579
1.558
1.436
1.292
1.285
1.278
1.272
1.264
1.258
1.218
1.214
1.206
1.195
1.181
0.515
0.507
0.501
0.491
0.483
0.476
-0.000
ppm

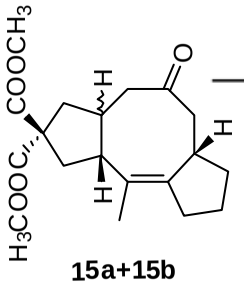

HF-02
File: HF-02-20070801-C
Pulse Sequence: s2pul
MeO2C
CO2Me
14
171.541
141.641
132.655
118.892
118.377
77.216
77.000
76.792
58.037
52.234
37.035
35.428
31.158
31.069
27.557
12.500
220
200
180
160
140
120
100
80
60
40
20
0
ppm


2uy-05-12b
File: 2uy-05-12b-H
Pulse Sequence: s2pul
15a+15b
H3COOC
COOCH3
H
H
O
H
7.289
3.774
3.744
3.739
3.734
3.300
3.290
3.280
2.679
2.662
2.650
2.641
2.630
2.566
2.550
2.532
2.524
2.512
2.502
2.489
2.468
2.462
2.450
2.443
2.437
2.420
2.413
2.397
2.386
2.375
2.363
2.353
2.341
2.331
2.315
2.293
2.271
2.249
2.234
2.222
2.205
2.070
2.053
2.048
2.030
1.907
1.892
1.888
1.875
1.860
1.821
1.804
1.791
1.779
1.761
1.745
1.582
1.555
1.534
1.525
1.255
1.219
1.211
1.207
0.880
0.071
0.000
ppm

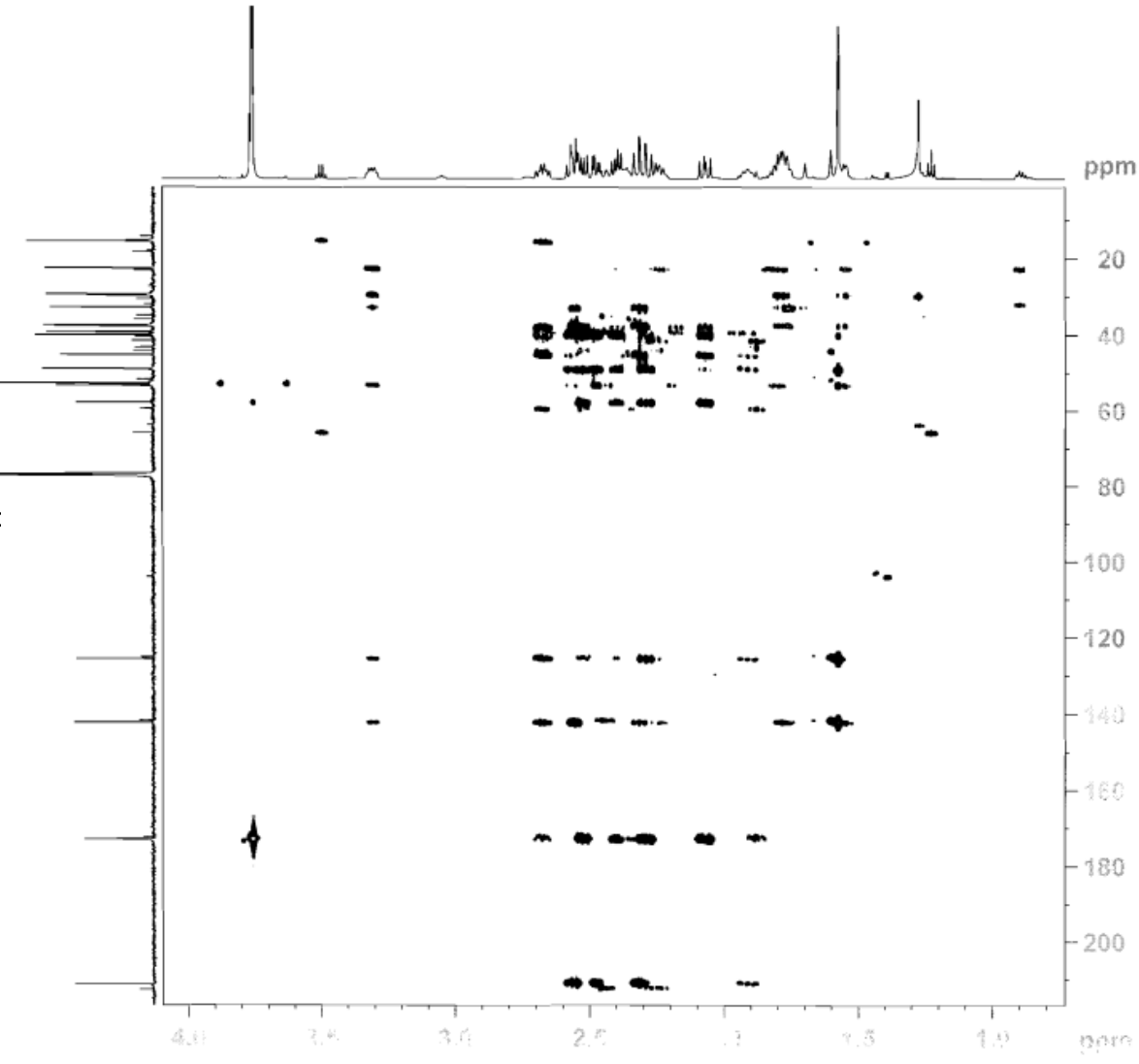

H3COOC
COOCH3
15a+15b
H
H
O
H
Pulse Sequence: s2pul
File: 2uy-05-12b-C
212.512
211.210
172.930
172.889
172.763
172.261
142.321
141.751
125.664
125.191
77.199
76.987
76.775
59.374
57.738
53.185
52.932
52.906
52.746
52.720
52.694
51.641
48.907
45.202
44.079
43.197
41.419
40.367
39.983
39.961
39.228
39.087
37.633
37.383
35.817
34.824
32.685
30.461
29.602
29.323
22.940
22.482
15.437
15.400
220
200
180
160
140
120
100
80
60
40
20
0
ppm


H3COOC
COOCH3
15a+15b
H
H
O
H
HSQC 2KY-05-12B CDC13 298K 091023
ppm
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
1.0
65
60
55
50
45
40
35
30
25
20
15
10

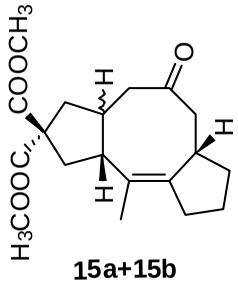
HMBC 2KY-05-12B CDCl3 298K 091023



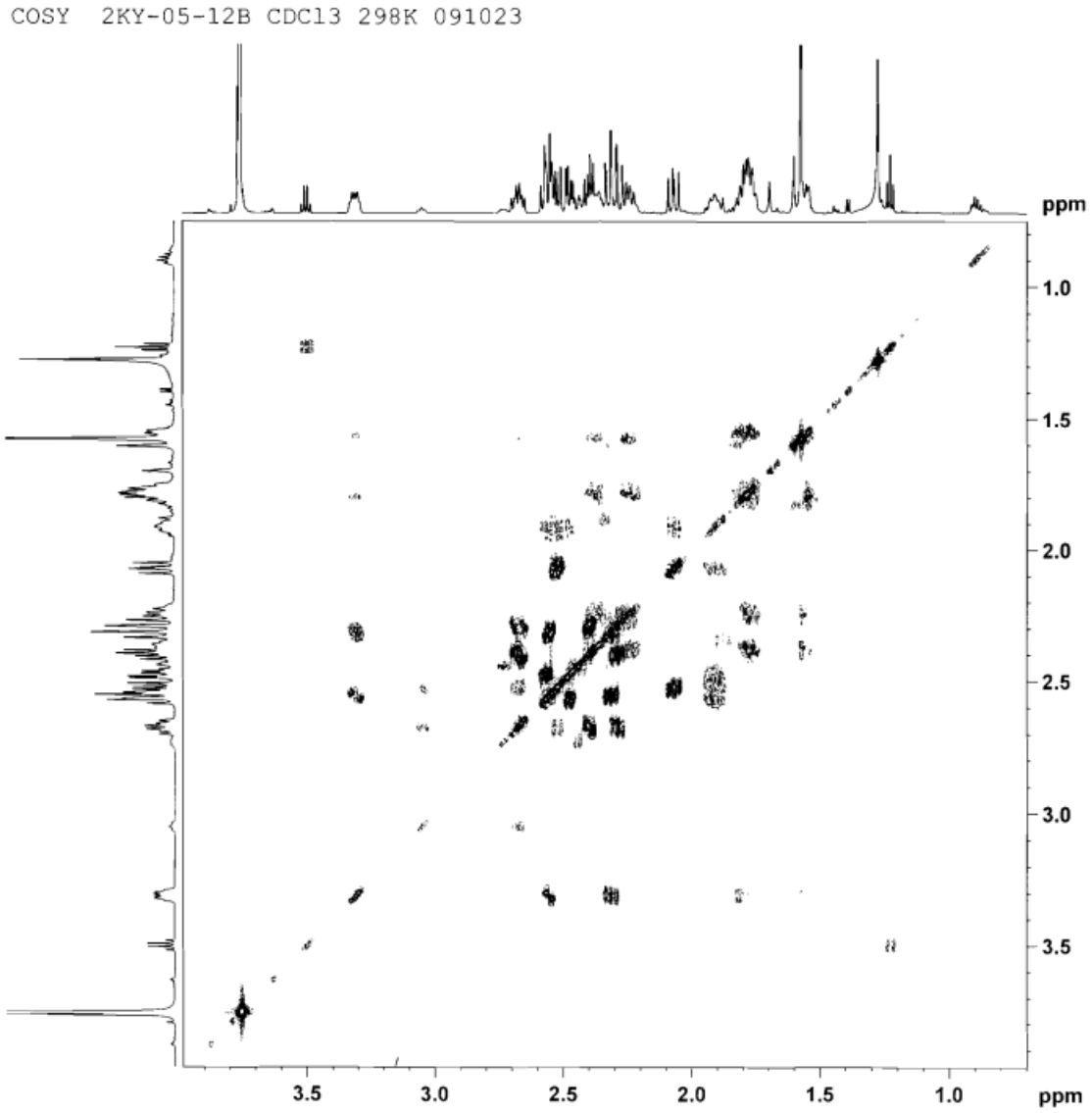
15a+15b



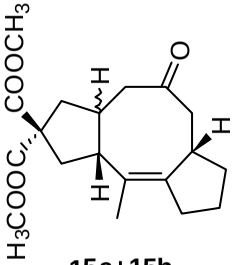

```
NAME                hmbc
EXPNO                 47
PROCNO                 1
Date_           20091023
Time                21.56
INSTRUM             spect
PROBHD    5 mm BBI 1H-BB
PULPROG    hmbcgplpndqf
TD                  2048
SOLVENT             CDCl3
NS                    16
DS                    16
SWH             3591.954 Hz
FIDRES          1.753884 Hz
AQ              0.2852708 sec
RG                  26008
DW                139.200 usec
DE                  6.50 usec
TE                298.3 K
CNST2         145.0000000
CNST13          6.2500000
D0              0.00000300 sec
D1              1.50000000 sec
D2              0.00344828 sec
D6              0.08000000 sec
D16             0.00020000 sec
IN0             0.00001350 sec

======== CHANNEL f1 ========
NUC1                  1H
P1                  9.60 usec
P2                 17.20 usec
PL1                -1.00 dB
PL1W         31.62277603 W
SFO1         600.1316804 MHz

======== CHANNEL f2 ========
NUC2                 13C
P3                 14.50 usec
PL2                -2.00 dB
PL2W        119.43215179 W
SFO2         150.9201628 MHz

====== GRADIENT CHANNEL =====
GPNAM1           SINE.100
GPNAM2           SINE.100
GPNAM3           SINE.100
GPZ1               50.00 %
GPZ2               30.00 %
GPZ3               40.10 %
P16              1000.00 usec
ND0                    2
TD                   256
SFO1           150.9202 MHz
FIDRES         144.435318 Hz
SW                245.000 ppm
FnMODE                QF
SI                  2048
SF           600.1300000 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
PC                  1.40
SI                  1024
MC2                   QF
SF           150.9028090 MHz
WDW                 SINE
SSB                    0
LB                  0.00 Hz
GB                     0
```



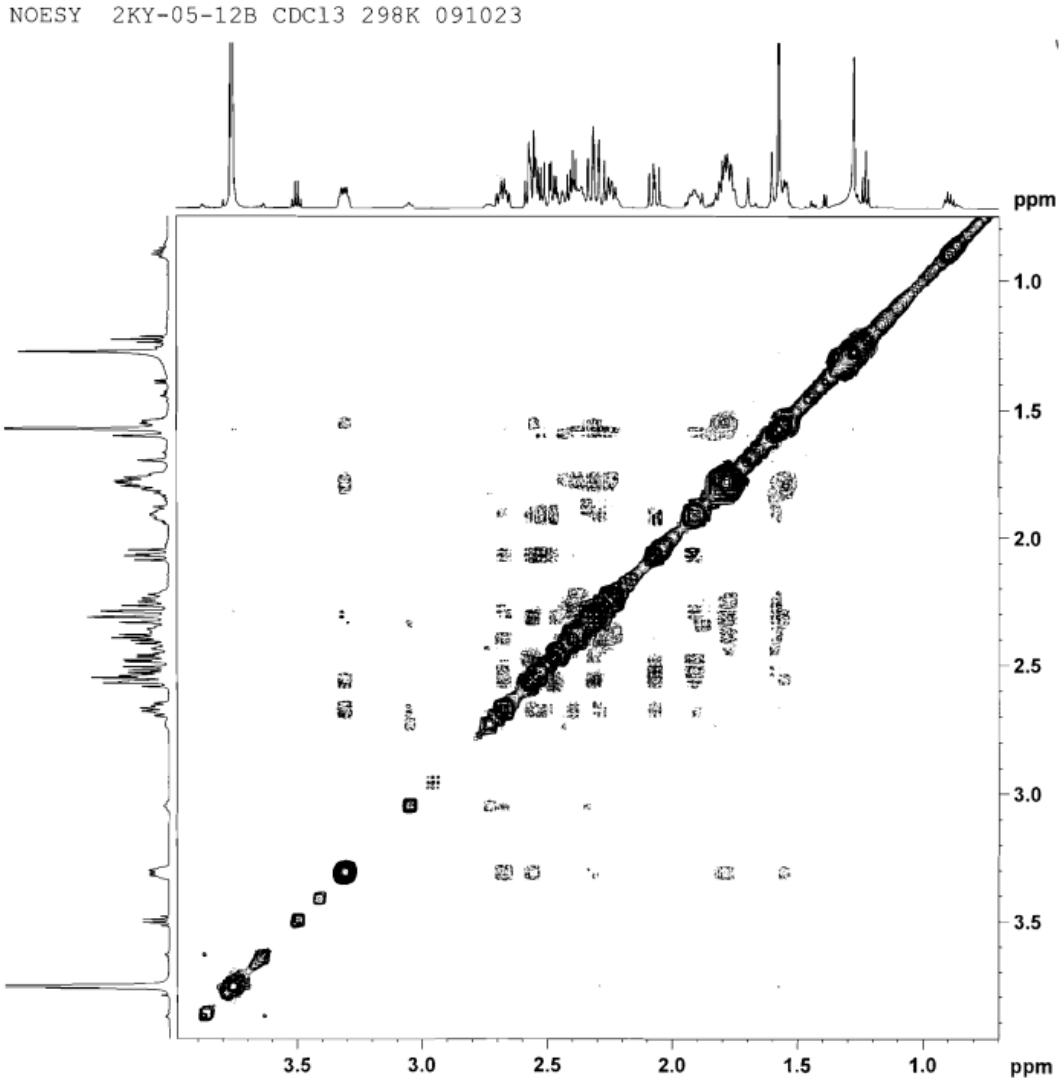
15a+15b



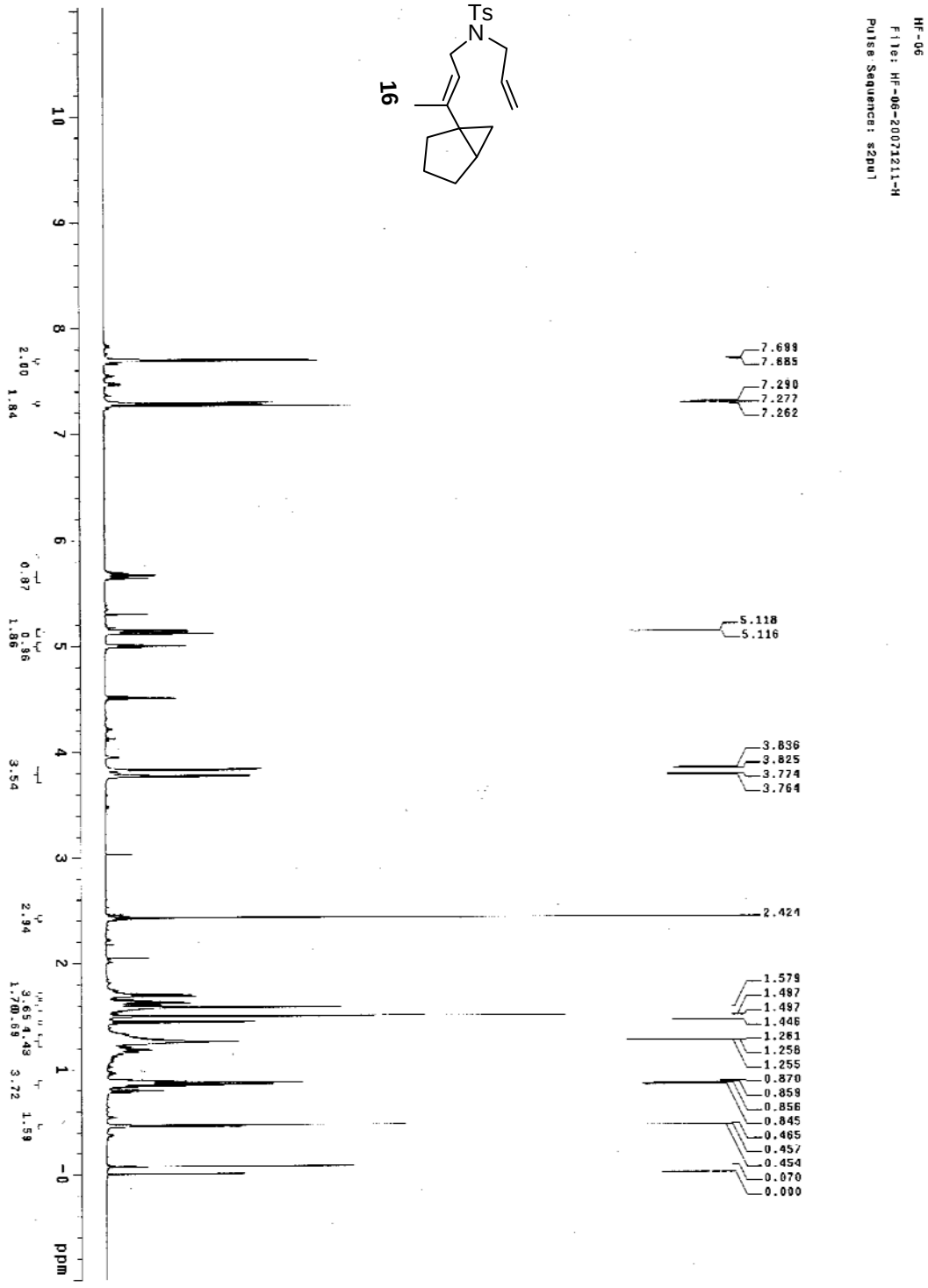
NAME	cosy
EXPNO	79
PROCNO	1
Date_	20091023
Time	19.50
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	cosygpqf
TD	2048
SOLVENT	CDC13
NS	16
DS	16
SWH	3591.954 Hz
FIDRES	1.753884 Hz
AQ	0.2852708 sec
RG	181
DW	139.200 usec
DE	6.50 usec
TE	298.2 K
D0	0.00000300 sec
D1	1.50000000 sec
D13	0.00000400 sec
D16	0.00002000 sec
IN0	0.00027840 sec
======== CHANNEL f1 ========	
NUC1	1H
P0	4.30 usec
P1	8.60 usec
PL1	-1.00 dB
PL1W	31.62277603 W
SFO1	600.1316804 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPZ1	10.00 %
P16	1000.00 usec
ND0	1
TD	256
SFO1	600.1317 MHz
FIDRES	14.031126 Hz
SW	5.985 ppm
FnMODE	QF
SI	1024
SF	600.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	600.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

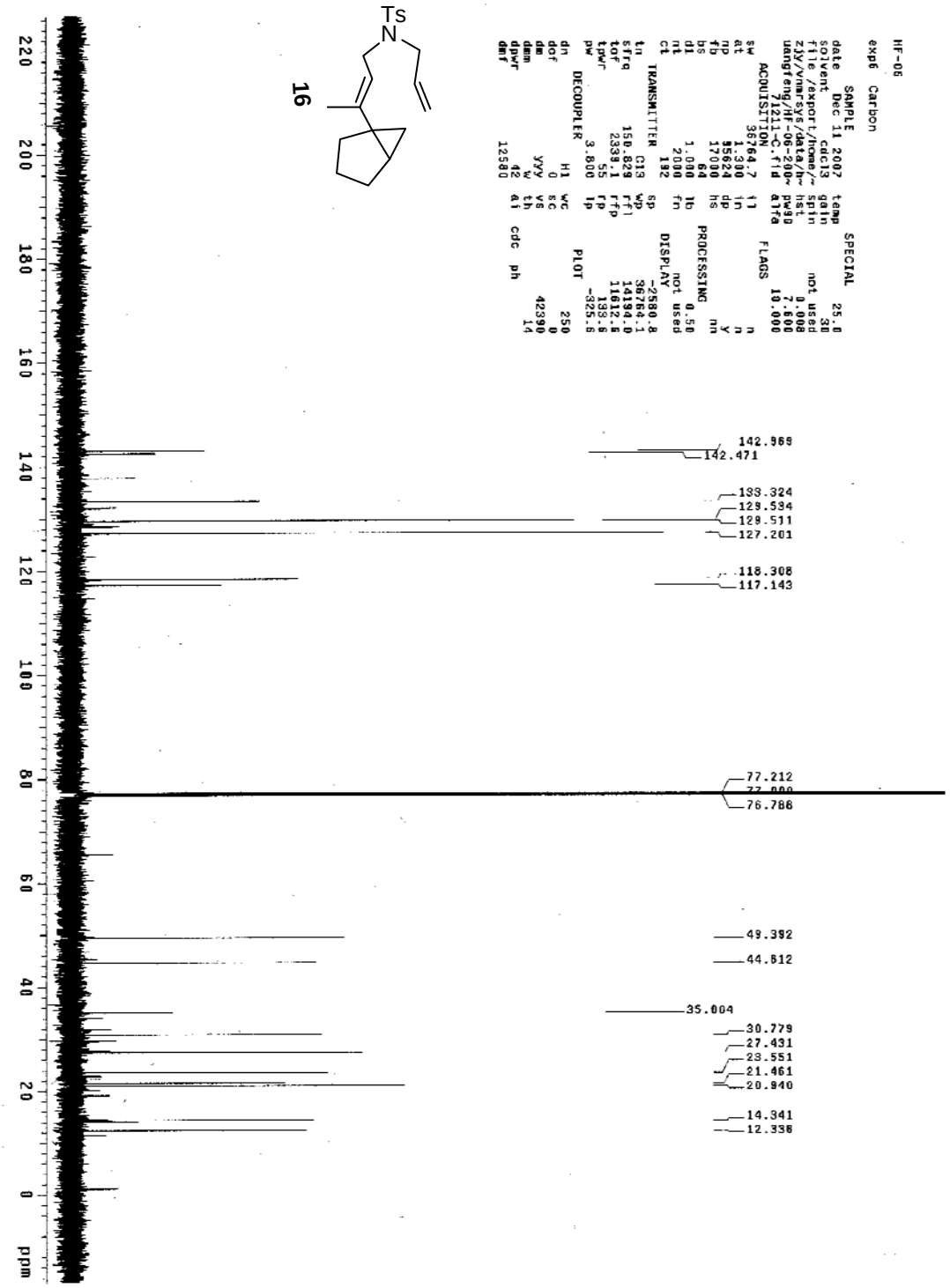


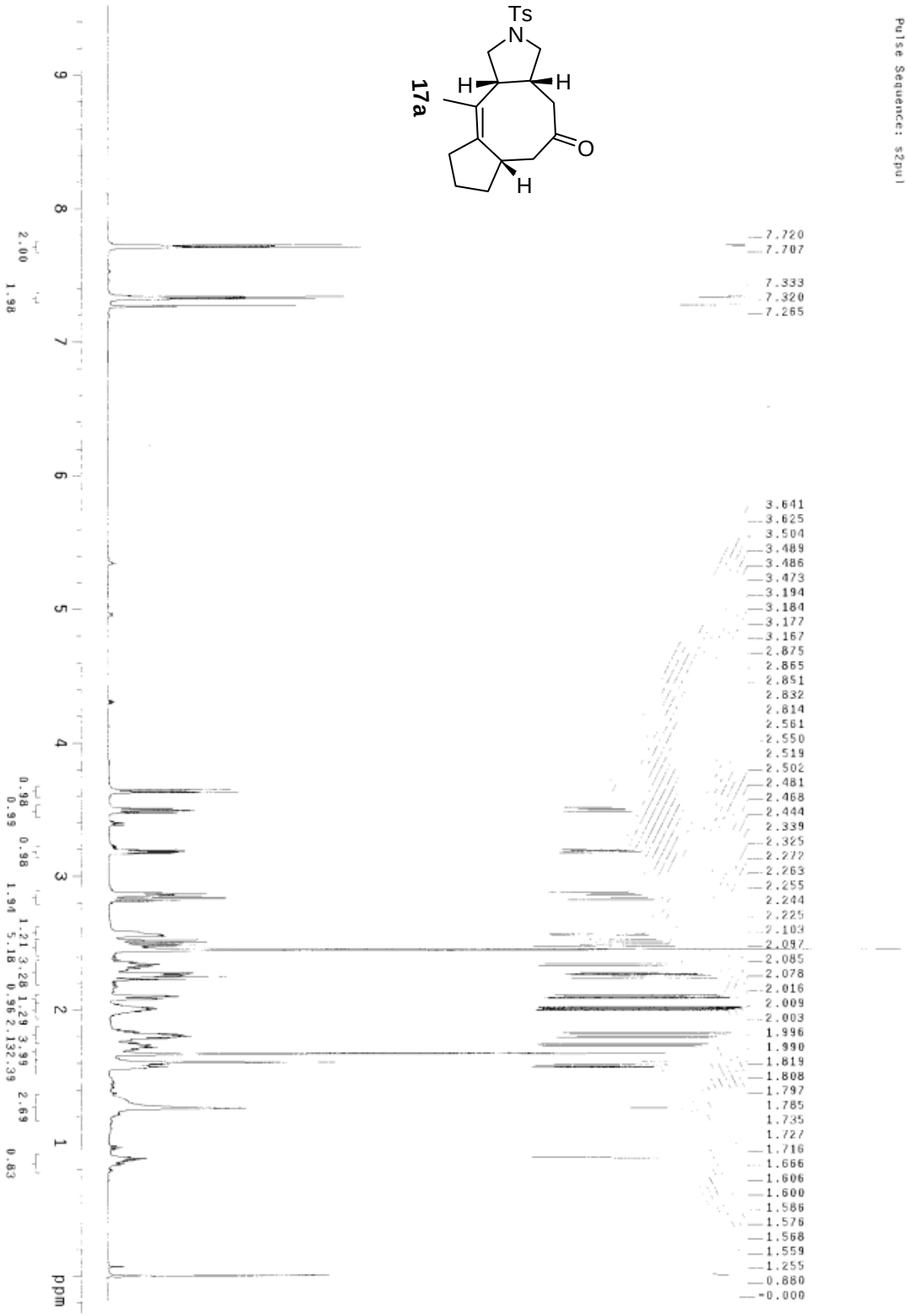
15a+15b

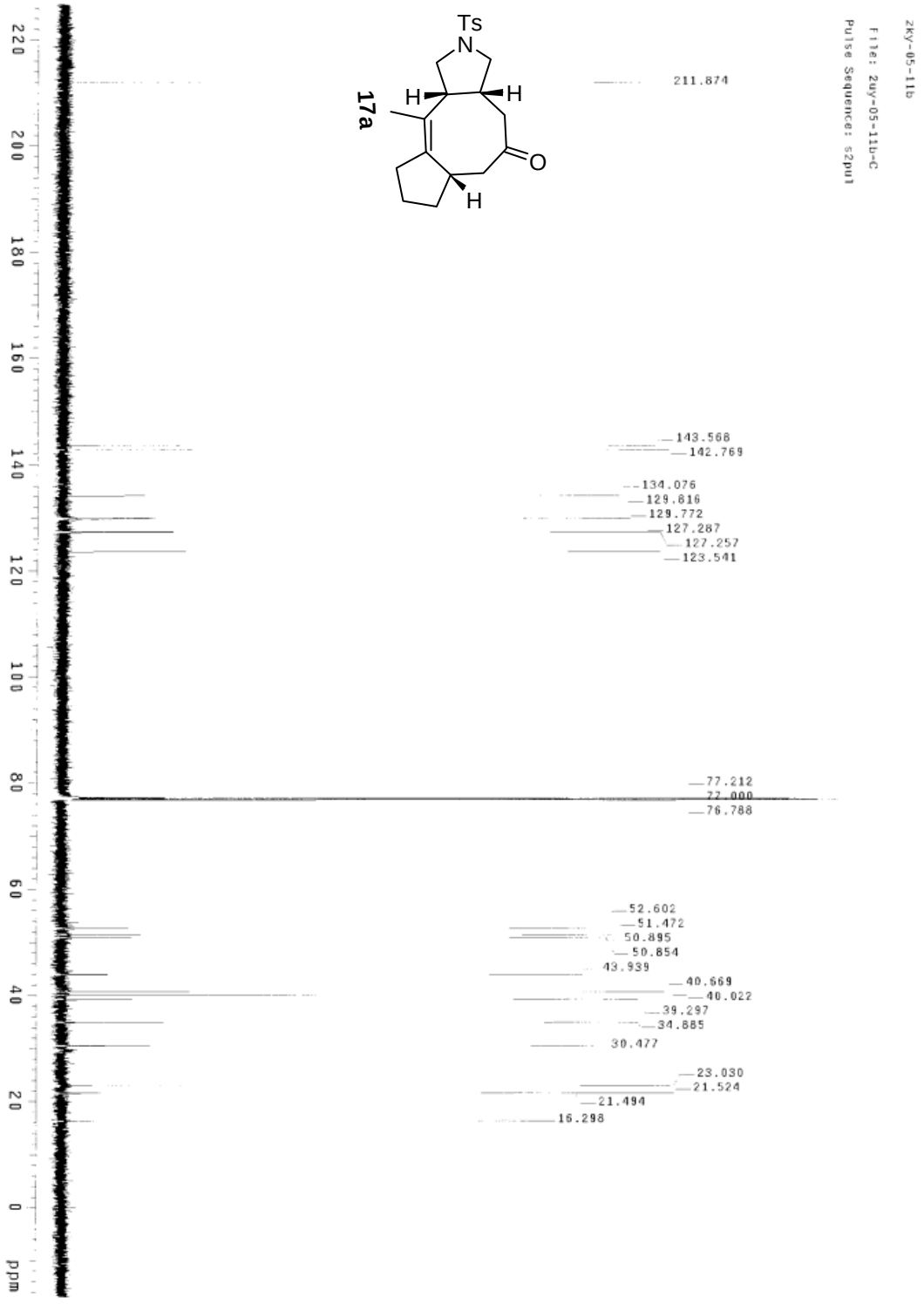


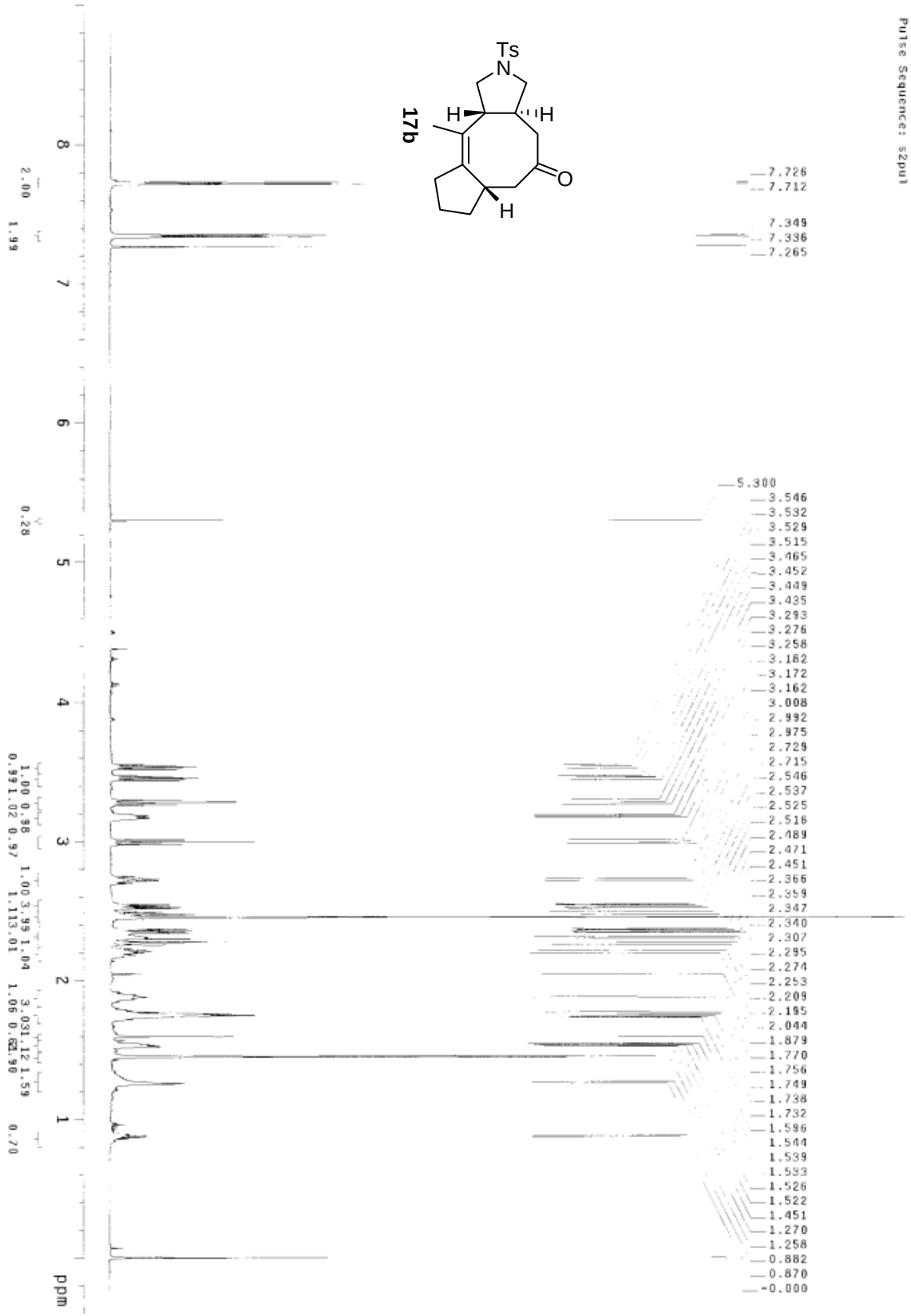
NAME	noesy	
EXPNO	100	
PROCNO	1	
Date_	20091024	
Time	1.59	
INSTRUM	spect	
PROBHD	5 mm BBI 1H-BB	
PULPROG	noesygpph	
TD	2048	
SOLVENT	CDC13	
NS	16	
DS	16	
SWH	3591.954	Hz
FIDRES	1.753884	Hz
AQ	0.2852708	sec
RG	45.3	
DW	139.200	usec
DE	6.50	usec
TE	298.3	K
D0	0.00012825	sec
D1	1.50000000	sec
D8	0.80000001	sec
D16	0.00020000	sec
IN0	0.00027840	sec
======== CHANNEL f1 ========		
NUC1	1H	
P1	8.60	usec
P2	17.20	usec
PL1	-1.00	dB
PL1W	31.62277603	W
SFO1	600.1316804	MHz
====== GRADIENT CHANNEL =====		
GPNAM1	SINE.100	
GPNAM2	SINE.100	
GPZ1	40.00	%
GPZ2	-40.00	%
P16	1000.00	usec
ND0	1	
TD	256	
SFO1	600.1317	MHz
FIDRES	14.031126	Hz
SW	5.985	ppm
FnMODE	States-TPPI	
SI	1024	
SF	600.1300000	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	
PC	1.40	
SI	1024	
MC2	States-TPPI	
SF	600.1300000	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	

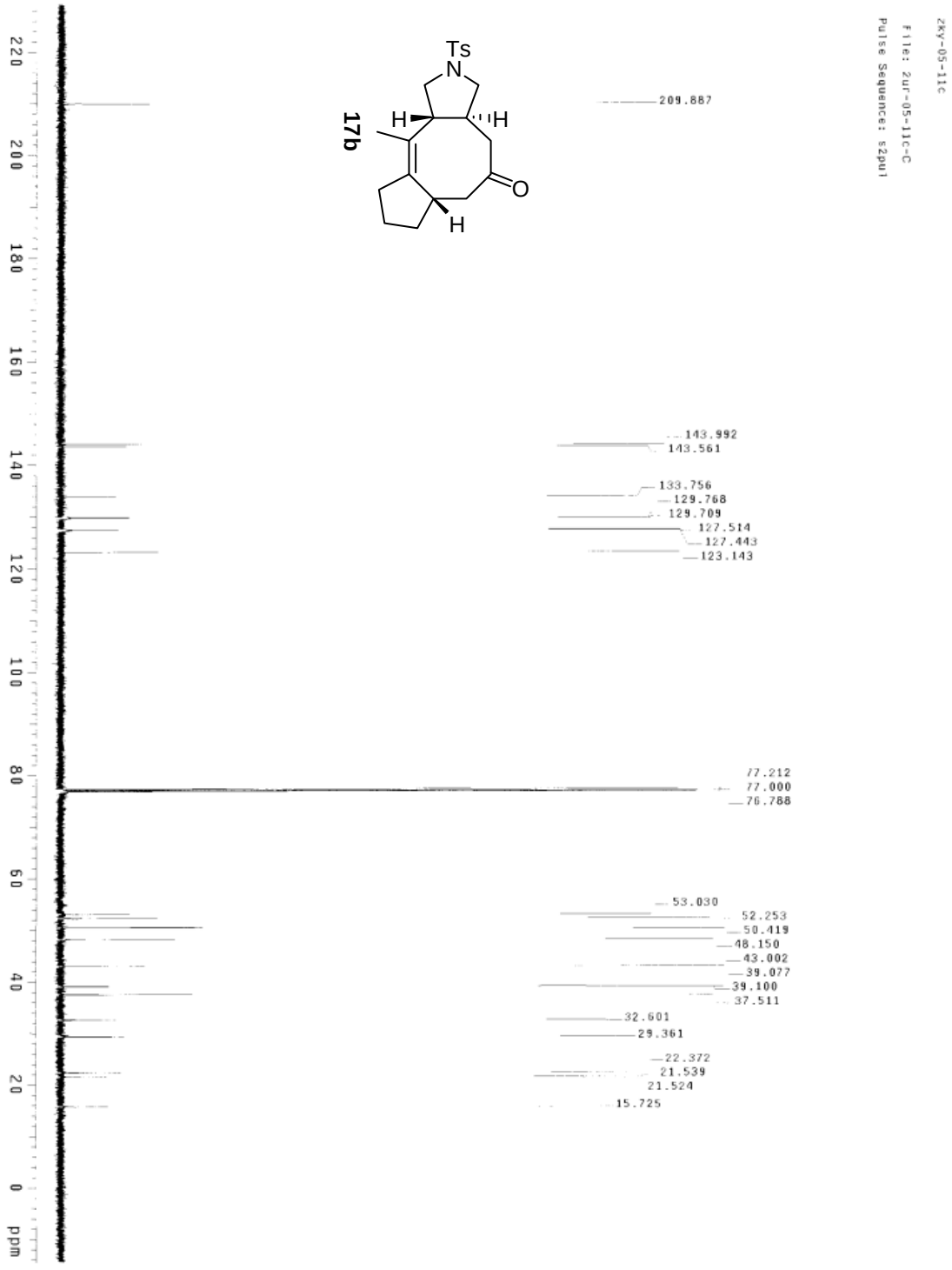

HF-06
File: HF-06-20071211-H
Pulse Sequence: s2pul
Ts
N
16
7.699
7.685
7.290
7.277
7.262
5.118
5.116
3.836
3.825
3.774
3.764
2.424
1.579
1.497
1.497
1.446
1.261
1.258
1.255
0.870
0.858
0.856
0.845
0.465
0.457
0.454
0.070
0.000
ppm

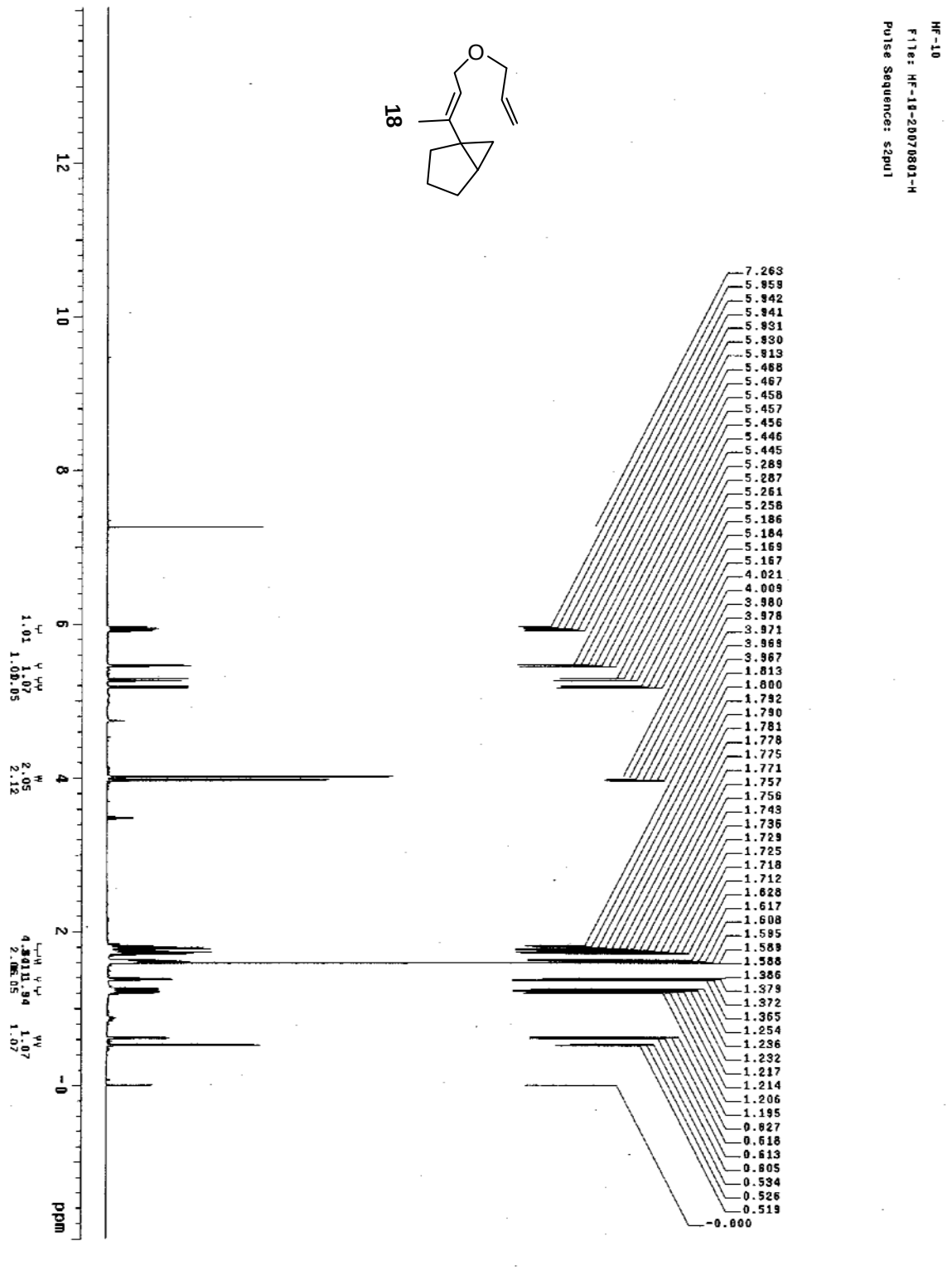

Ts
N
16
142.969
142.471
133.324
129.534
128.511
127.201
118.308
117.143
77.212
77.000
76.788
49.392
44.612
35.004
30.779
27.431
23.551
21.461
20.940
14.341
12.336
ppm

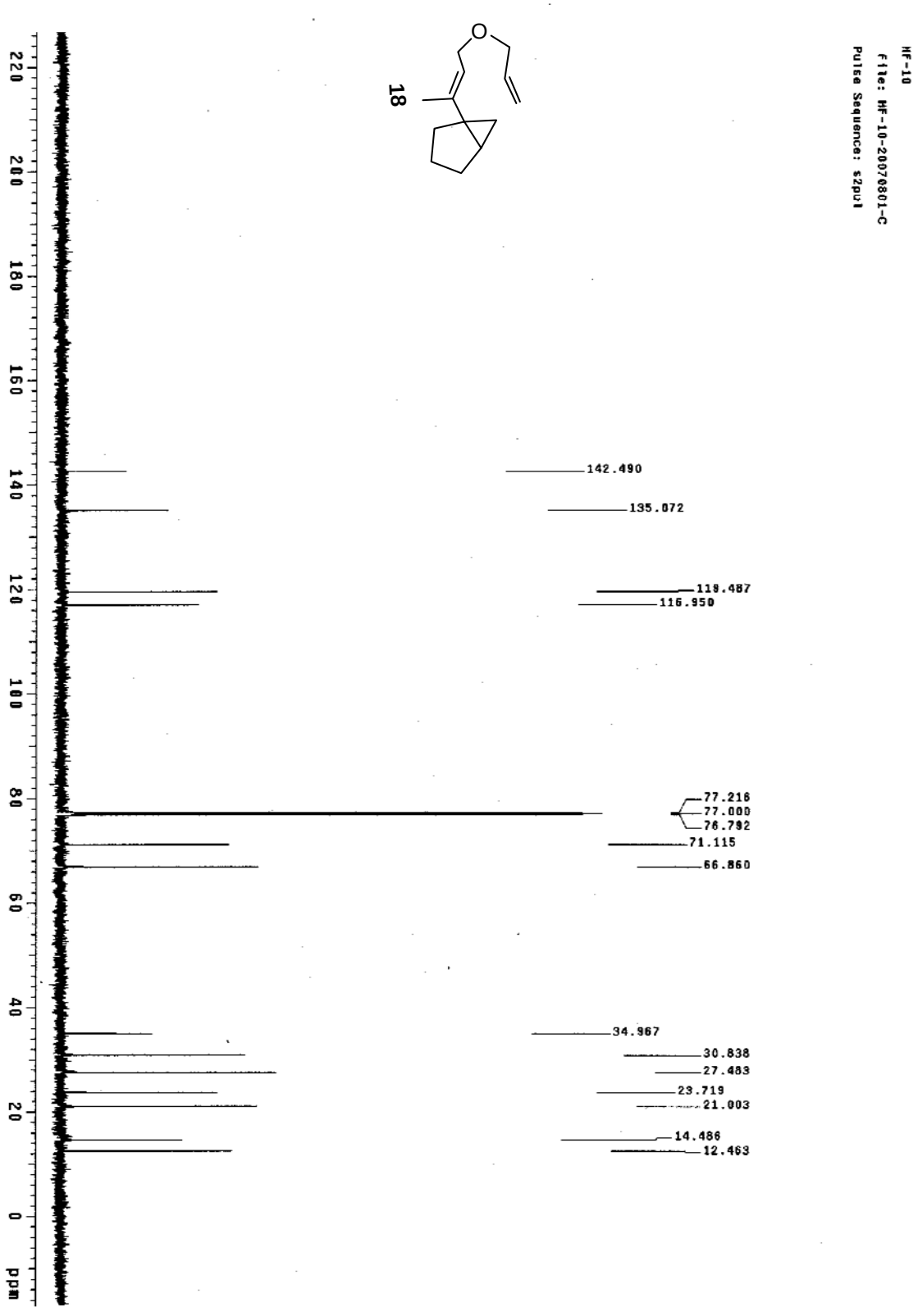

ZKY-05-11B
file: zuy-05-11b-H
Pulse Sequence: s2pul
Ts
N
H
H
O
H
17a
7.720
7.707
7.333
7.320
7.265
3.641
3.625
3.504
3.489
3.486
3.473
3.194
3.184
3.177
3.167
2.875
2.865
2.851
2.832
2.814
2.561
2.550
2.519
2.502
2.481
2.468
2.444
2.339
2.325
2.272
2.263
2.255
2.244
2.225
2.103
2.097
2.085
2.078
2.016
2.009
2.003
1.996
1.990
1.819
1.808
1.797
1.785
1.735
1.727
1.716
1.666
1.606
1.600
1.586
1.576
1.568
1.559
1.255
0.880
-0.000
ppm

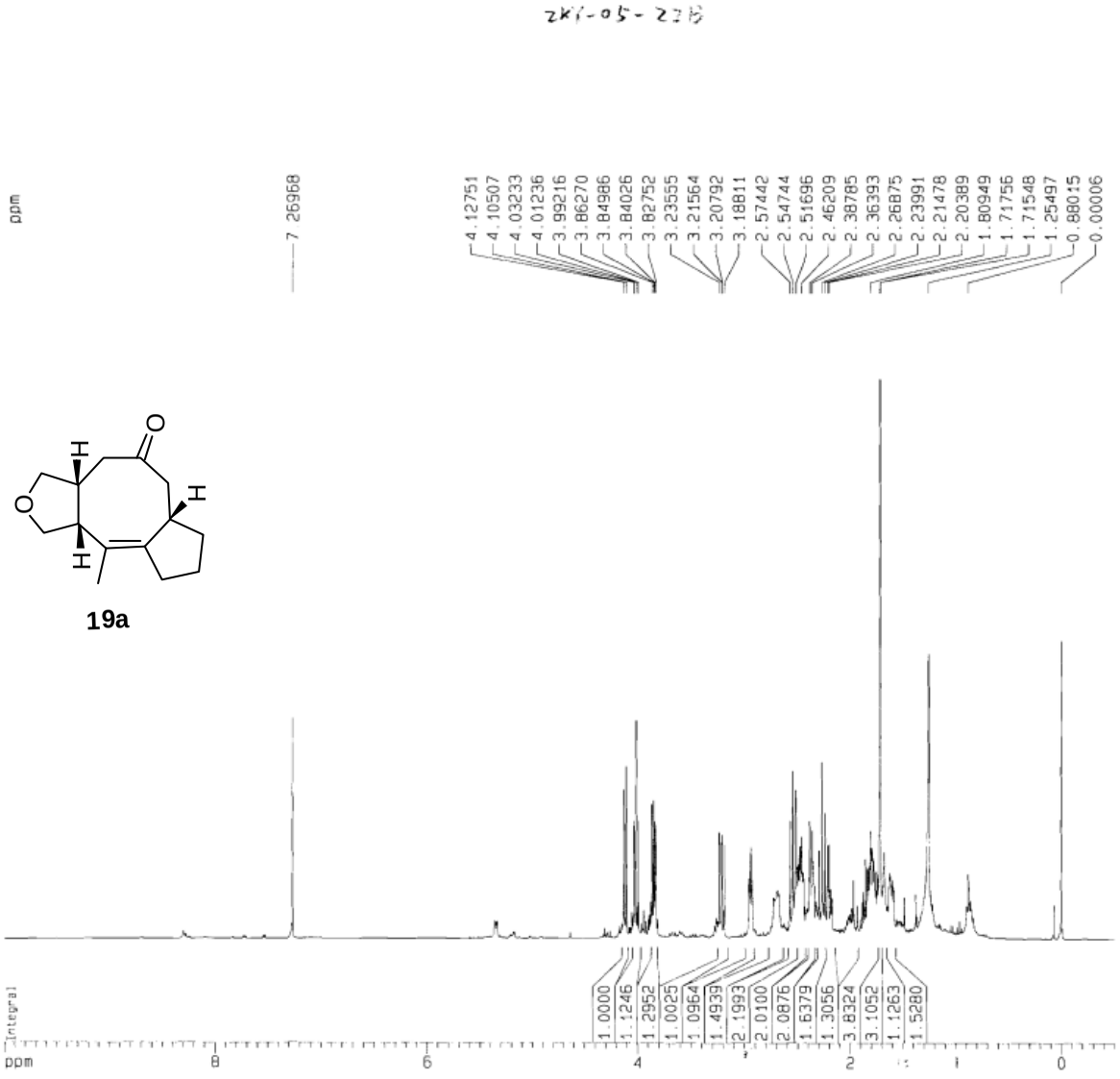

zky-05-11b
File: 2uy-05-11b-C
Pulse Sequence: s2pul
Ts
N
H
H
O
H
17a
211.874
143.568
142.769
134.076
129.816
129.772
127.287
127.257
123.541
77.212
77.000
76.788
52.602
51.472
50.895
50.854
43.939
40.669
40.022
39.297
34.885
30.477
23.030
21.524
21.494
16.298
220
200
180
160
140
120
100
80
60
40
20
0
ppm


File: 2ur-05-11c-H
Pulse Sequence: s2pul
Ts
N
H
H
H
O
17b
7.726
7.712
7.349
7.336
7.265
5.300
3.546
3.532
3.529
3.515
3.465
3.452
3.449
3.435
3.293
3.276
3.258
3.182
3.172
3.162
3.008
2.992
2.975
2.729
2.715
2.546
2.537
2.525
2.516
2.489
2.471
2.451
2.366
2.359
2.347
2.340
2.307
2.295
2.274
2.253
2.209
2.195
2.044
1.879
1.770
1.756
1.749
1.738
1.732
1.596
1.544
1.539
1.533
1.526
1.522
1.451
1.270
1.258
0.882
0.870
-0.000
ppm


Ts
N
H
H
O
H
17b
209.887
143.992
143.561
133.756
129.768
129.709
127.514
127.443
123.143
77.212
77.000
76.788
53.030
52.253
50.419
48.150
43.002
39.077
39.100
37.511
32.601
29.361
22.372
21.539
21.524
15.725
220
200
180
160
140
120
100
80
60
40
20
0
ppm

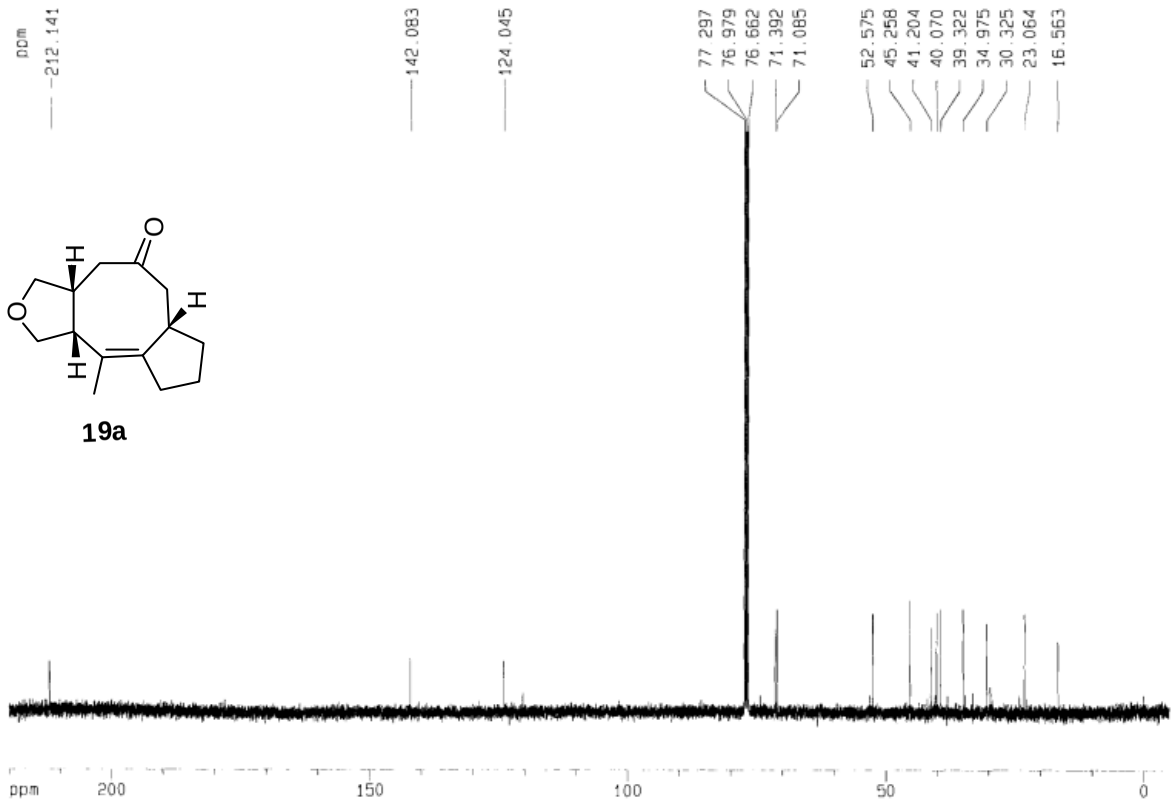

HF-10
File: HF-18-20070801-H
Pulse Sequence: s2pul
18
7.263
5.959
5.942
5.941
5.931
5.930
5.913
5.468
5.467
5.458
5.457
5.456
5.446
5.445
5.289
5.287
5.261
5.258
5.186
5.184
5.169
5.167
4.021
4.009
3.980
3.978
3.971
3.969
3.967
1.813
1.800
1.792
1.790
1.781
1.778
1.775
1.771
1.757
1.756
1.743
1.736
1.729
1.725
1.718
1.712
1.628
1.617
1.608
1.595
1.589
1.588
1.386
1.379
1.372
1.365
1.254
1.236
1.232
1.217
1.214
1.206
1.195
0.827
0.618
0.613
0.605
0.534
0.526
0.519
-0.000
ppm


HF-10
file: HF-10-20070801-C
Pulse Sequence: s2pul
18
O
220
200
180
160
140
120
100
80
60
40
20
0
ppm
142.490
135.072
119.487
116.950
77.216
77.000
76.782
71.115
66.860
34.967
30.838
27.483
23.719
21.003
14.486
12.463



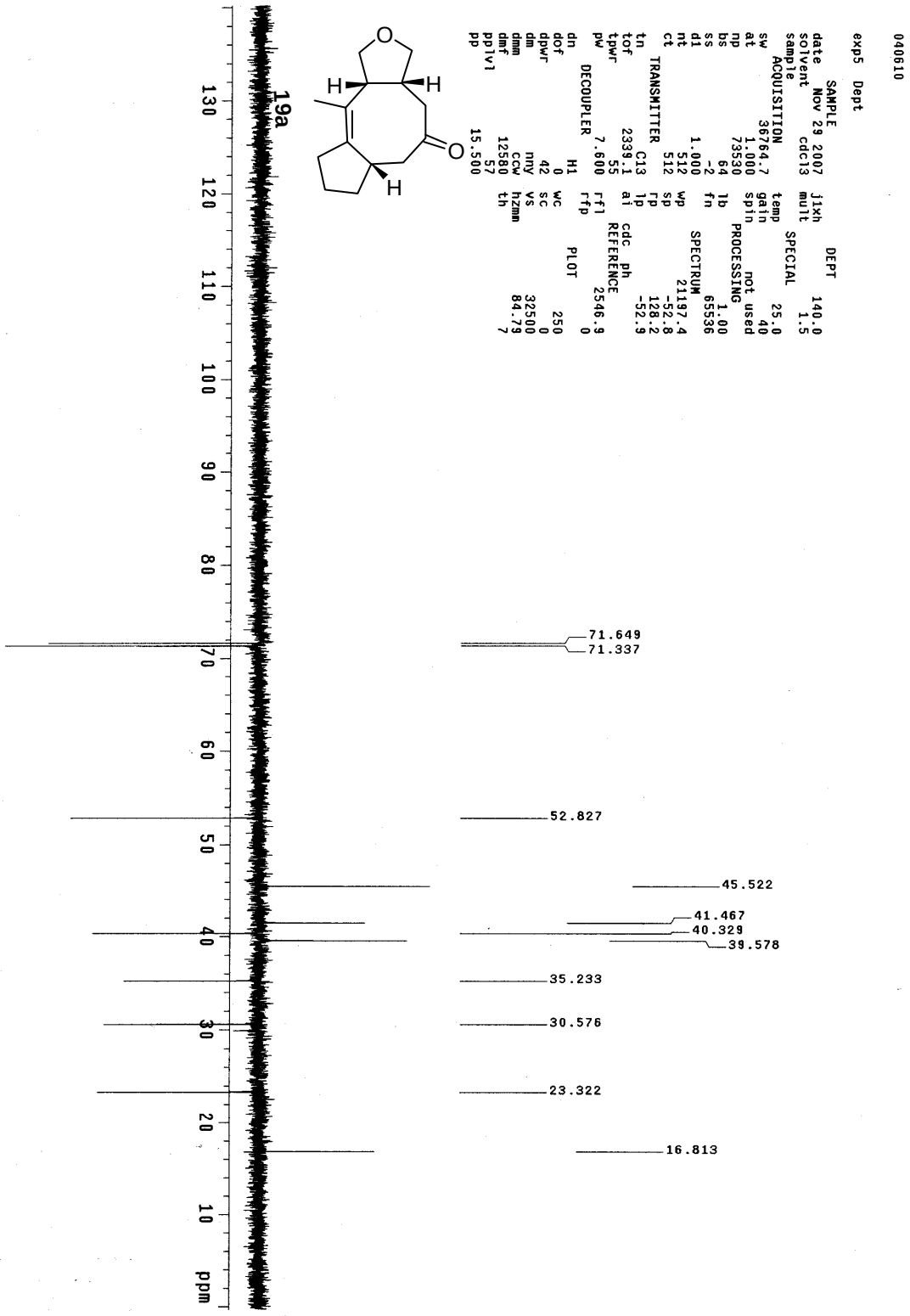
19a

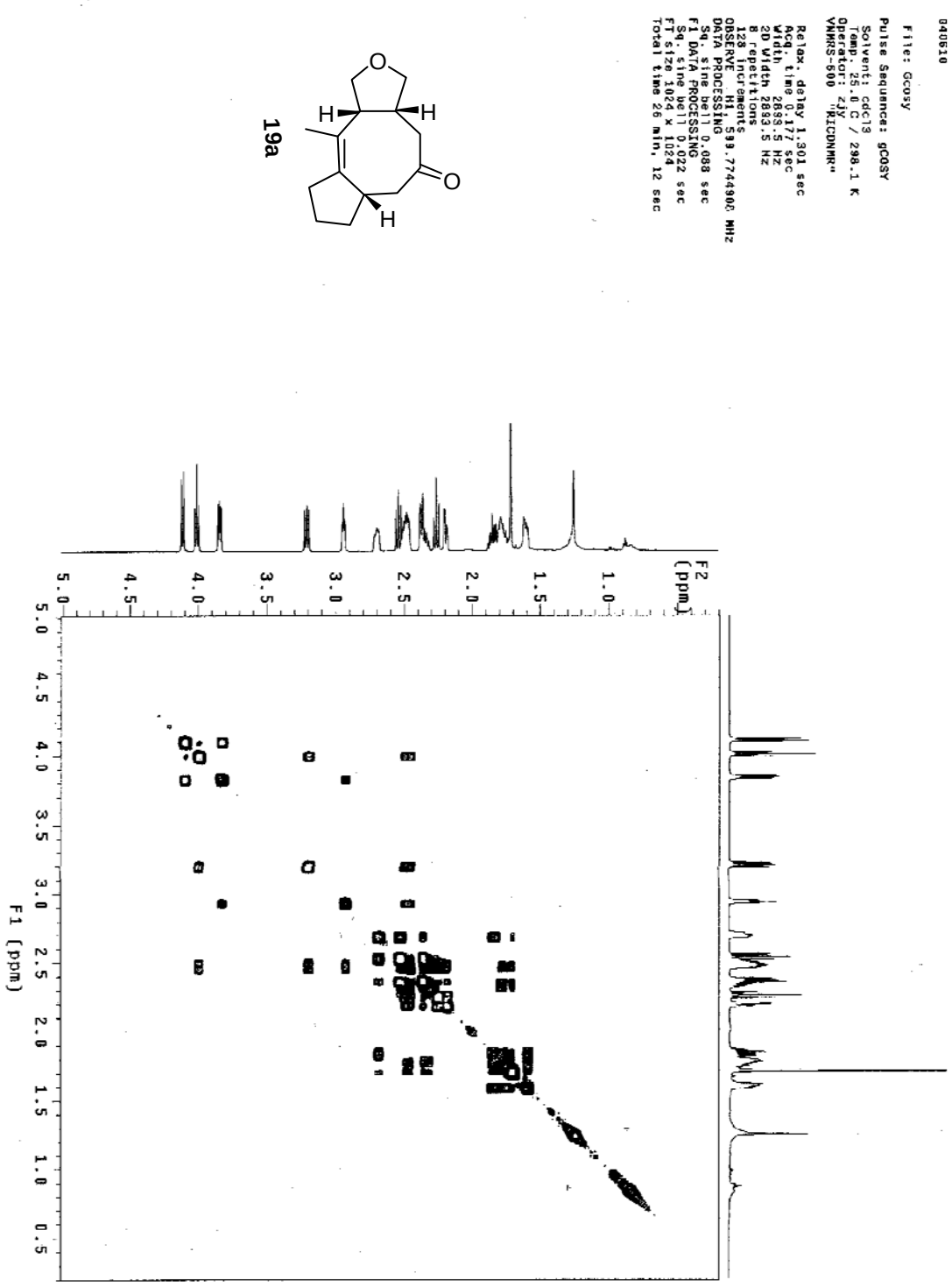
```
Current Data Parameters
NAME                 h03485
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_              20091028
Time                 13.56
INSTRUM              ARX400
PROBHD     5 mm Multinu
PULPROG                 zg
TD                   32768
SOLVENT              CDCl3
NS                      32
DS                       0
SWH               6097.561 Hz
FIDRES            0.186083 Hz
AQ                2.6870260 sec
RG                     360
DW                82.000 usec
DE                102.50 usec
TE                300.0 K
D1                2.00000000 sec
P1                3.00 usec
DE                102.50 usec
SFO1           400.1321971 MHz
NUCLEUS                1H

F2 - Processing parameters
SI                   16384
SF             400.1300055 MHz
WDW                     no
SSB                      0
LB                0.00 Hz
GB                       0
PC                4.00

1D NMR plot parameters
CX                20.00 cm
F1P               10.000 ppm
F1                4001.30 Hz
F2P               -0.500 ppm
F2                -200.07 Hz
PPMCM             0.52500 ppm/cm
HZCM              210.06825 Hz/cm
```

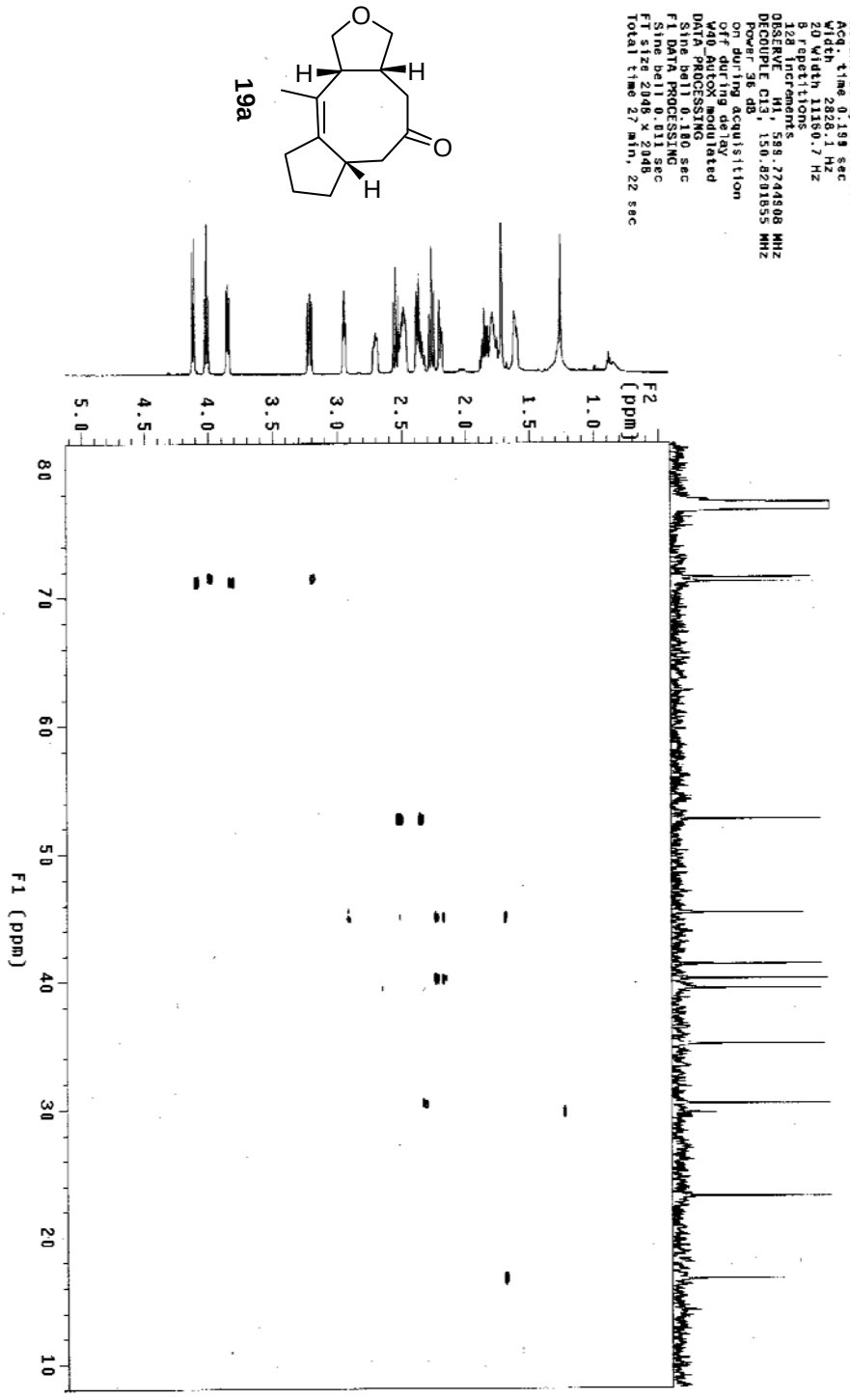


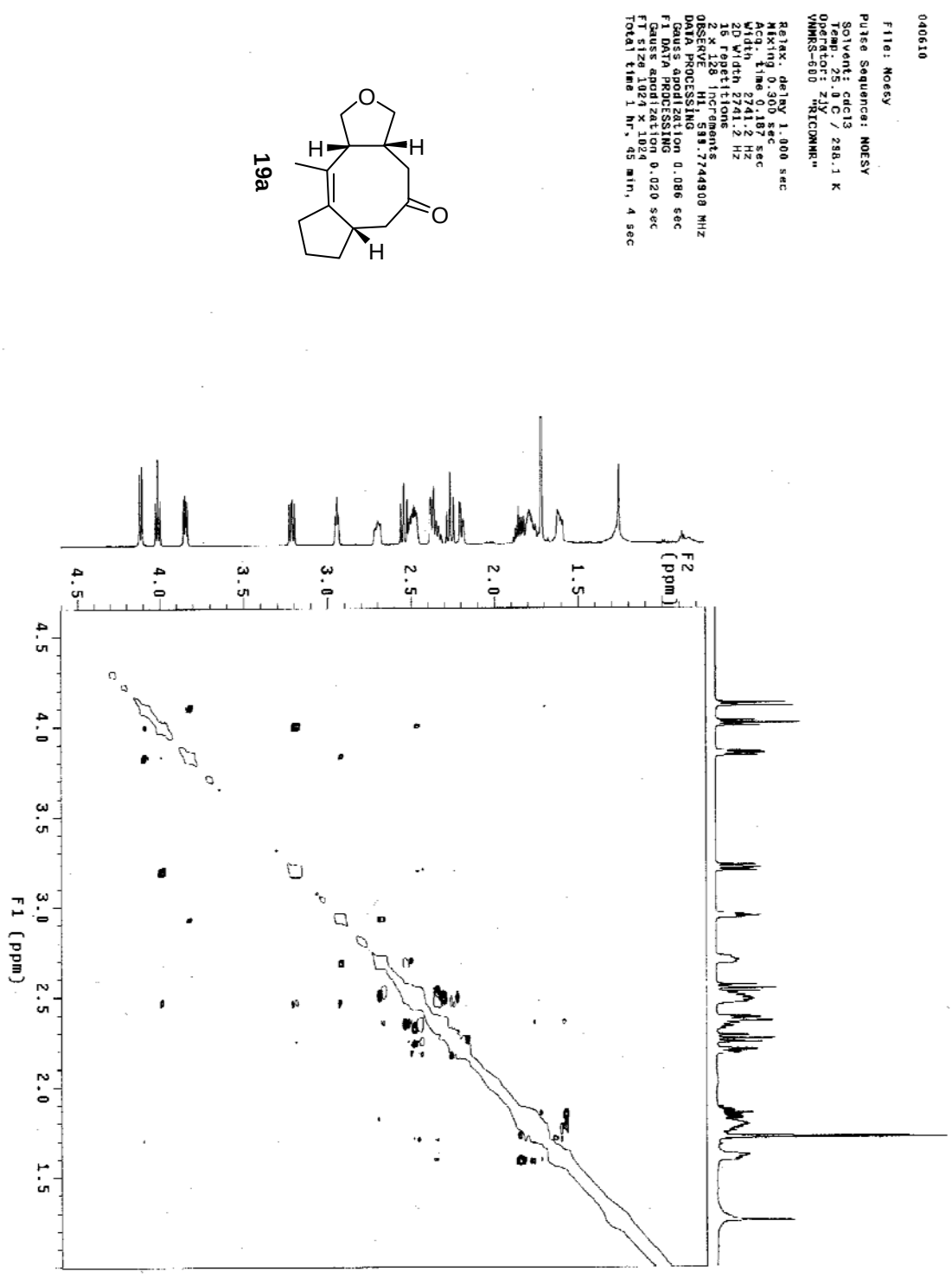
19a

```
Current Data Parameters
NAME                 c03485
EXPNO                     1
PROCNO                    1

F2 - Acquisition Parameters
Date_             20091028
Time                 13.59
INSTRUM              ARX400
PROBHD     5 mm Multinu
PULPROG              zgdc
TD                   32768
SOLVENT              CDCl3
NS                    1409
DS                       0
SWH              25000.000 Hz
FIDRES           0.762939 Hz
AQ               0.6554100 sec
RG                     512
DW                20.000 usec
DE                25.00 usec
TE                300.0 K
D12              0.00002000 sec
DL5                 24.00 dB
CPDPRG              waltz16
P31                100.00 usec
D1               2.00000000 sec
P1                  3.50 usec
DE                25.00 usec
SFO1            100.6233680 MHz
NUCLEUS                 13C
D11              0.03000000 sec

F2 - Processing parameters
SI                   32768
SF              100.6127735 MHz
WDW                     EM
SSB                      0
LB                    0.50 Hz
GB                       0
PC                    1.00

1D NMR plot parameters
CX                   20.00 cm
F1P                 220.000 ppm
F1                22134.81 Hz
F2P                  -5.000 ppm
F2                 -503.07 Hz
PPMCM              11.25000 ppm/cm
HZCM            1131.89368 Hz/cm
```

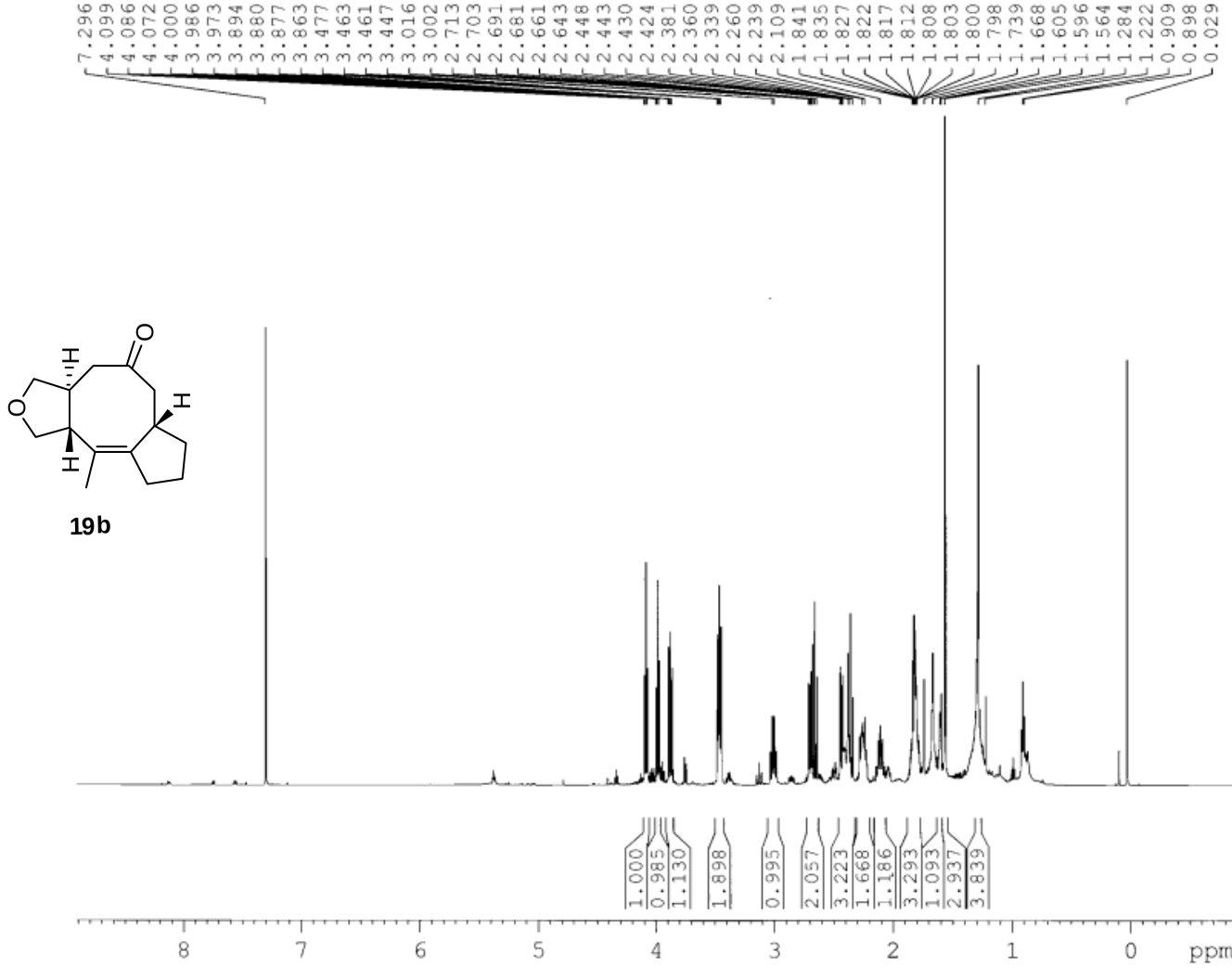

040610
exp5 Dept
SAMPLE
date Nov 29 2007
solvent cdcl3
sample
ACQUISITION
sw 36764.7
at 1.000
np 73530
bs 64
ss -2
d1 1.000
nt 512
ct 512
TRANSMITTER
tn C13
tof 2339.1
tpwr 55
pw 7.600
DECOUPLER
dn H1
dof 0
dpwr 42
dm nny
dmm ccw
dmf 12580
pplvl 57
pp 15.500
jlxh
mult
temp
gain
spin
PROCESSING
lb
fn
SPECTRUM
wp
sp
rp
lp
al
cdc ph
REFERENCE
rfl 2546.9
rfp 0
PLOT
wc 250
sc 0
vs 32500
hzmm 84.79
th 7
DEPT
140.0
1.5
SPECIAL
25.0
40
not used
1.00
65536
21197.4
-52.8
128.2
-52.9
19a
71.649
71.337
52.827
45.522
41.467
40.329
39.578
35.233
30.576
23.322
16.813
ppm


040610
File: Gcosy
Pulse Sequence: gCOSY
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: zjy
VNMRS-600 "RICDNMR"
Relax. delay 1.301 sec
Acq. time 0.177 sec
Width 2693.5 Hz
2D Width 2693.5 Hz
8 repetitions
128 increments
OBSERVE H1, 599.7744900 MHz
DATA PROCESSING
Sq. sine bell 0.088 sec
F1 DATA PROCESSING
Sq. sine bell 0.022 sec
FT size 1024 x 1024
Total time 26 min, 12 sec
19a
O
H
H
O
H
F2 (ppm)
F1 (ppm)


040610
File: 040610-GHSQC-2
Pulse Sequence: ghsqc
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: zjy
File: 040610-GHSQC-2
VNMRS-600 "RICDNMR"
Relax. delay 1.301 sec
Acq. time 0.159 sec
Width 2828.1 Hz
20 Width 11160.7 Hz
8 repetitions
128 increments
OBSERVE H1, 599.7744908 MHz
DECOUPLE C13, 150.8201855 MHz
Power 36 dB
on during acquisition
off during delay
W40_AutoX modulated
DATA PROCESSING
Sine bell 0.180 sec
F1 DATA PROCESSING
Sine bell 0.011 sec
FT size 2048 x 2048
Total time 27 min, 22 sec
19a
O
H
H
O
H
F2 (ppm)
F1 (ppm)


040610
File: Noesy
Pulse Sequence: NOESY
Solvent: cdcl3
Temp. 25.0 C / 298.1 K
Operator: zjy
VNMRS-600 "RICOWNR"
Relax. delay 1.000 sec
Mixing 0.300 sec
Acq. time 0.187 sec
Width 2741.2 Hz
2D Width 2741.2 Hz
16 repetitions
2 x 128 increments
OBSERVE H1, 599.7744900 MHz
DATA PROCESSING
Gauss apodization 0.086 sec
F1 DATA PROCESSING
Gauss apodization 0.020 sec
F1 size 1024 x 1024
Total time 1 hr, 45 min, 4 sec
19a
O
H
H
O
H
F2 (ppm)
F1 (ppm)
4.5
4.0
3.5
3.0
2.5
2.0
1.5

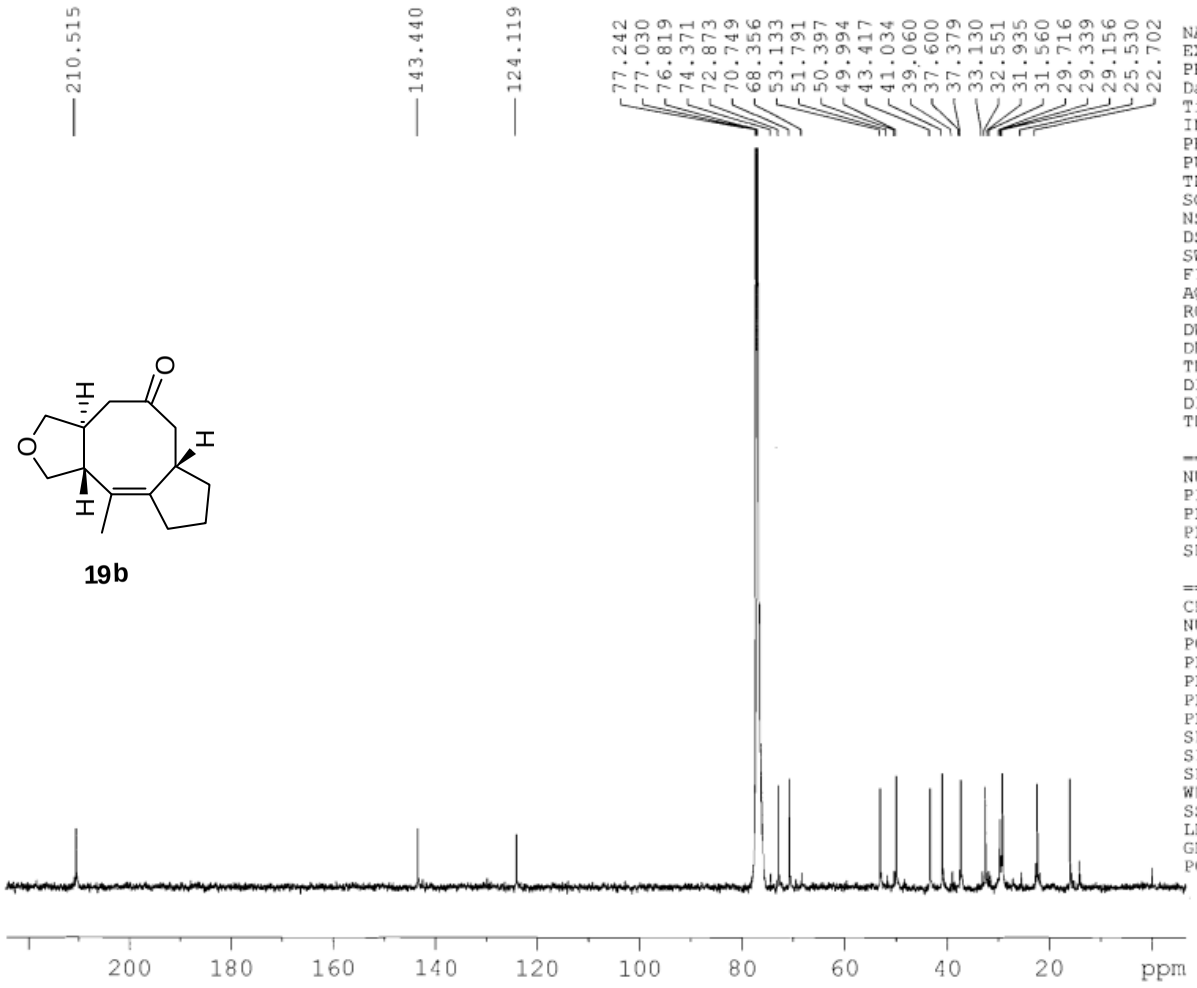
1H NMR CDCl3 ZKY-05-22C Yu Zhixiang 2009103



19b

NAME	1h
EXPNO	745
PROCNO	1
Date_	20091030
Time	23.12
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	zg30
TD	65536
SOLVENT	CDCl3
NS	16
DS	0
SWH	17985.611 Hz
FIDRES	0.274439 Hz
AQ	1.8219786 sec
RG	161.3
DW	27.800 usec
DE	6.00 usec
TE	294.7 K
D1	2.00000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	1H
P1	8.60 usec
PL1	-1.00 dB
PL1W	31.62277603 W
SFO1	600.1348010 MHz
SI	65536
SF	600.1300000 MHz
WDW	EM
SSB	0
LB	0.30 Hz
GB	0
PC	1.00

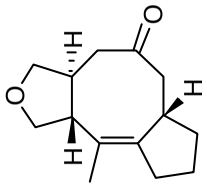
13C-NMR CDCl3 ZKY-05-22C Yu Zhixiang 20091031



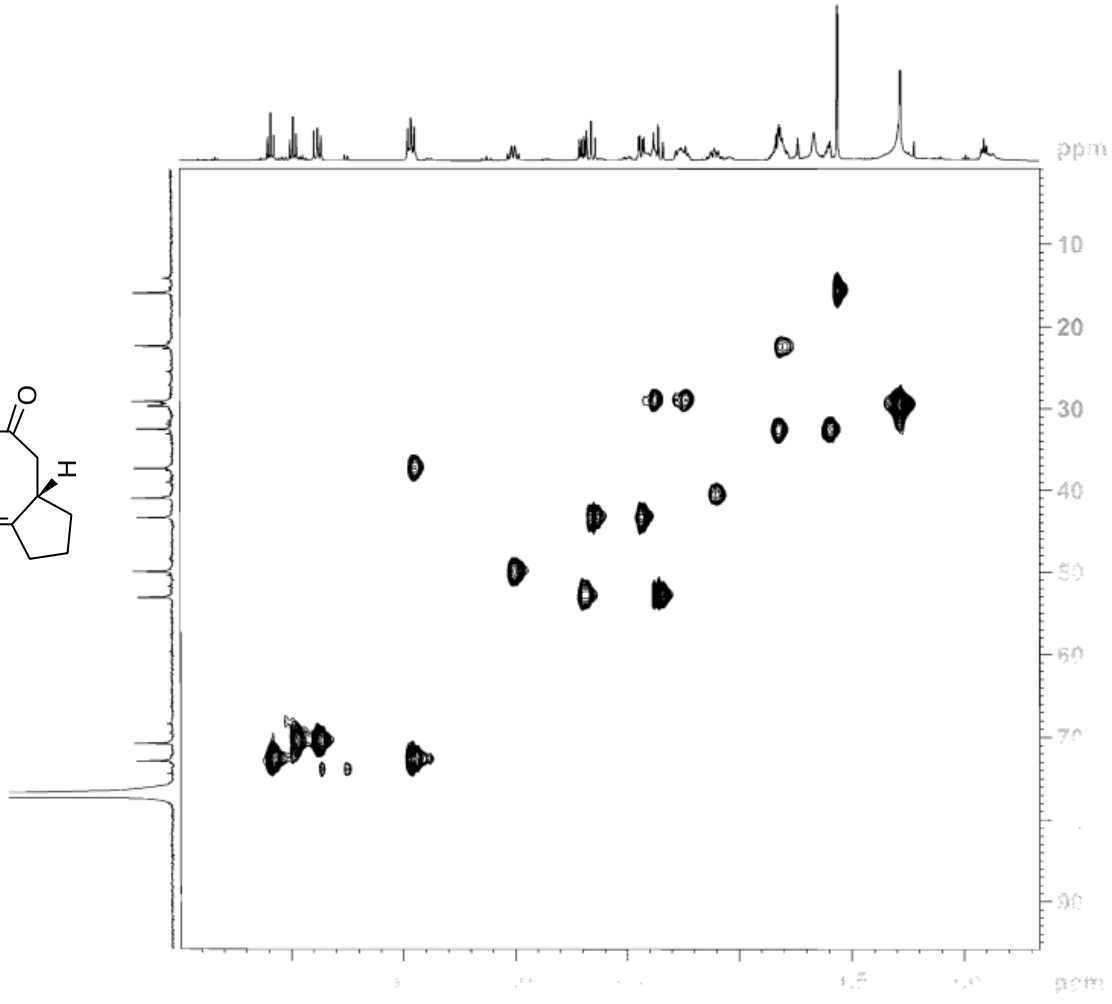
19b

NAME	13c
EXPNO	199
PROCNO	1
Date_	20091101
Time	9.23
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	zgig30
TD	65536
SOLVENT	CDCl3
NS	24576
DS	0
SWH	37593.984 Hz
FIDRES	0.573639 Hz
AQ	0.8716921 sec
RG	9195.2
DW	13.300 usec
DE	6.50 usec
TE	295.6 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1
======== CHANNEL f1 ========	
NUC1	13C
P1	13.10 usec
PL1	-2.00 dB
PL1W	119.43215179 W
SFO1	150.9194083 MHz
======== CHANNEL f2 ========	
CPDPRG2	waltz16
NUC2	1H
PCPD2	88.00 usec
PL2	-1.00 dB
PL12	19.40 dB
PL2W	31.62277603 W
PL12W	0.28840318 W
SFO2	600.1324005 MHz
SI	32768
SF	150.9028090 MHz
WDW	EM
SSB	0
LB	5.00 Hz
GB	0
PC	1.20

HSQC CDCl3 ZKY-05-22C Yu Zhixiang 20091030

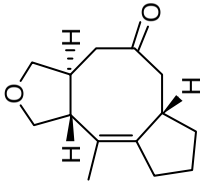


19b

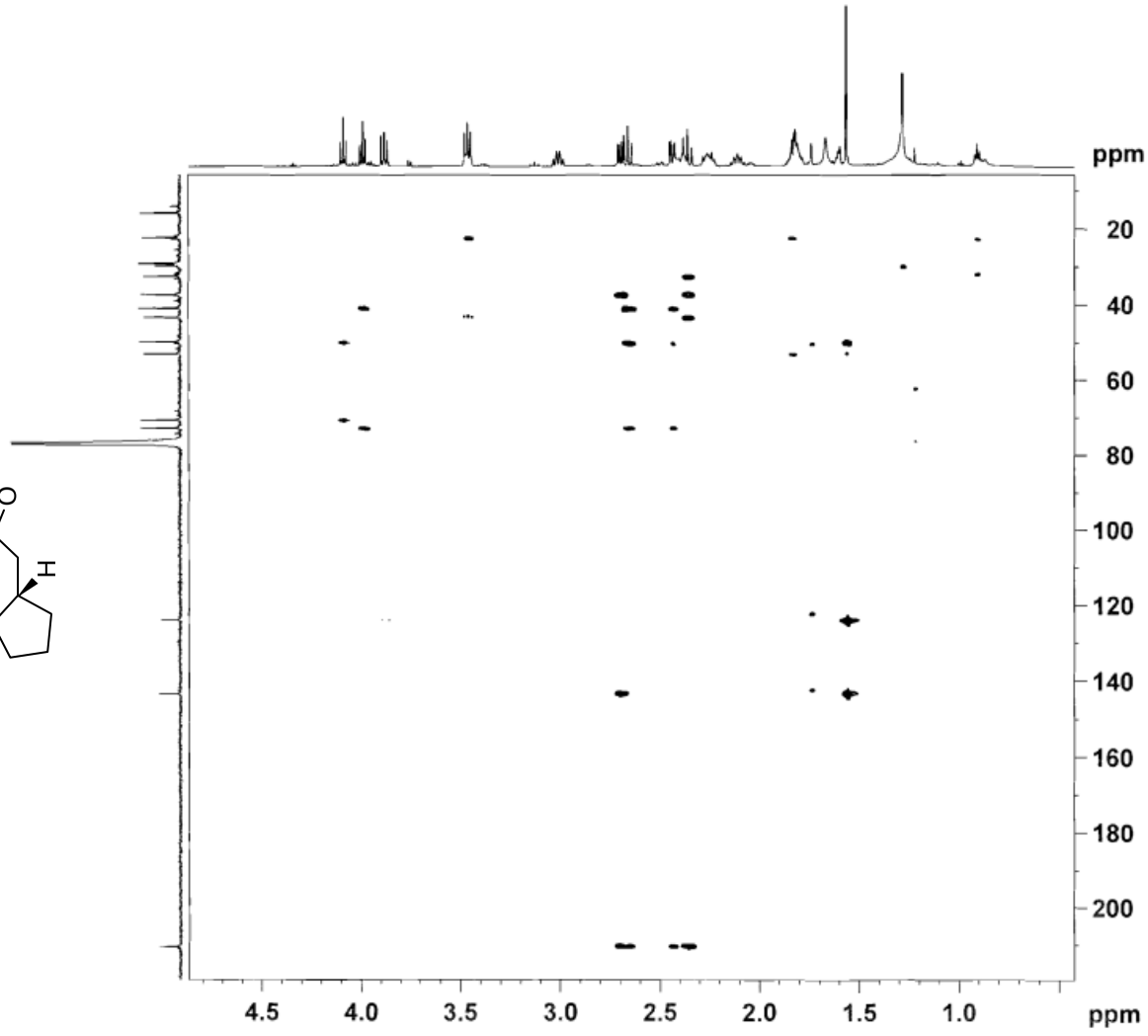



```
NAME                 hsqc
EXPNO                  59
PROCNO                  1
Date_             20091031
Time                 4.54
INSTRUM             spect
PROBHD   5 mm BBI 1H-BB
PULPROG          hsqcetgp
TD                   2048
SOLVENT              CDCl3
NS                      8
DS                     16
SWH               6613.757 Hz
FIDRES            3.229373 Hz
AQ                0.154954 sec
RG                 23170.5
DW                75.600 usec
DE                 6.50 usec
TE                 295.3 K
CNST2           145.0000000
D0                0.00000300 sec
D1                2.00000000 sec
D4                0.00172414 sec
D11               0.03000000 sec
D13               0.00000400 sec
D16               0.00020000 sec
IN0               0.00001325 sec
ZGOPTNS

======== CHANNEL f1 ========
NUC1                    1H
P1                    8.60 usec
P2                   17.20 usec
P28                   0.10 usec
PL1                  -1.00 dB
PL1W           31.62277603 W
SFO1           600.1330006 MHz

======== CHANNEL f2 ========
CPDPRG2              garp
NUC2                   13C
P3                   14.50 usec
P4                   29.00 usec
PCPD2                60.00 usec
PL2                  -2.00 dB
PL12                 10.34 dB
PL2W          119.43215179 W
PL12W          6.96821022 W
SFO2           150.9178993 MHz

====== GRADIENT CHANNEL =====
GPNAM1            SINE.100
GPNAM2            SINE.100
GPZ1                 80.00 %
GPZ2                 20.10 %
P16                1000.00 usec
ND0                     2
TD                    256
SFO1           150.9179 MHz
FIDRES         147.380768 Hz
SW                250.000 ppm
FnMODE      Echo-Antiecho
SI                   1024
SF             600.1300000 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
PC                   1.40
SI                    512
MC2         echo-antiecho
SF             150.9028090 MHz
WDW                 QSINE
SSB                     2
LB                   0.00 Hz
GB                      0
```

HMBC CDCl3 ZKY-05-22C Yu Zhixiang 20091030

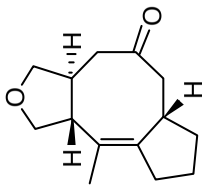


19b

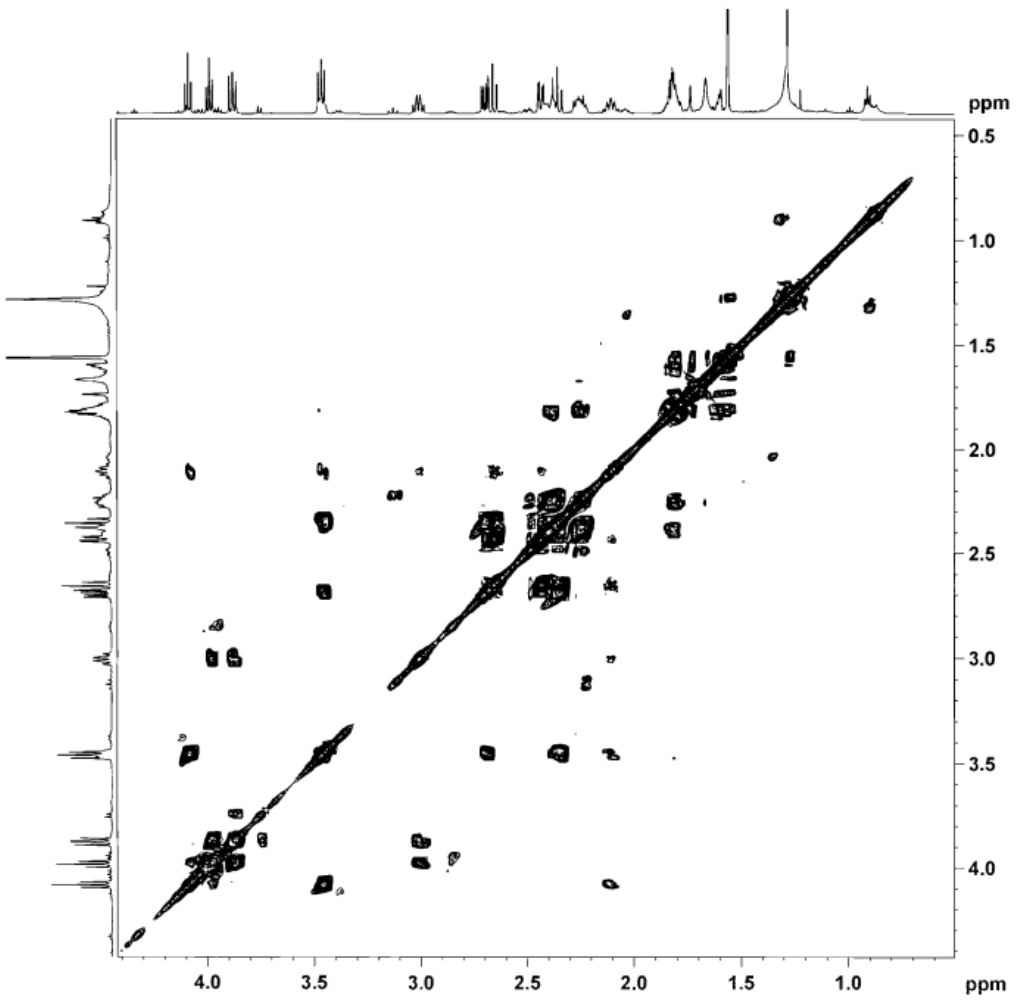


NAME	hmbc
EXPNO	49
PROCNO	1
Date_	20091031
Time	2.55
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	hmbcgplpndqf
TD	2048
SOLVENT	CDCl3
NS	8
DS	16
SWH	6613.757 Hz
FIDRES	3.229373 Hz
AQ	0.1549544 sec
RG	26008
DW	75.600 usec
DE	6.50 usec
TE	295.2 K
CNST2	145.0000000
CNST13	6.2500000
D0	0.00000100 sec
D1	1.50000000 sec
D2	0.00344828 sec
D6	0.08000000 sec
D16	0.00020000 sec
IN0	0.00001350 sec
======== CHANNEL f1 ========	
NUC1	1H
P1	8.60 usec
P2	17.20 usec
PL1	-1.00 dB
PL1W	31.62277603 W
SFO1	600.1330006 MHz
======== CHANNEL f2 ========	
NUC2	13C
P3	14.50 usec
PL2	-2.00 dB
PL2W	119.43215179 W
SFO2	150.9201628 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPNAM2	SINE.100
GPNAM3	SINE.100
GPZ1	50.00 %
GPZ2	30.00 %
GPZ3	40.10 %
P16	1000.00 usec
ND0	2
TD	256
SFO1	150.9202 MHz
FIDRES	144.435318 Hz
SW	245.000 ppm
FnMODE	QF
SI	2048
SF	600.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	150.9028090 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

COSY CDCl3 ZKY-05-22C Yu Zhixiang 20091030

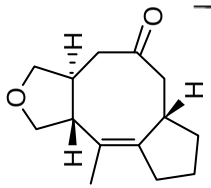


19b

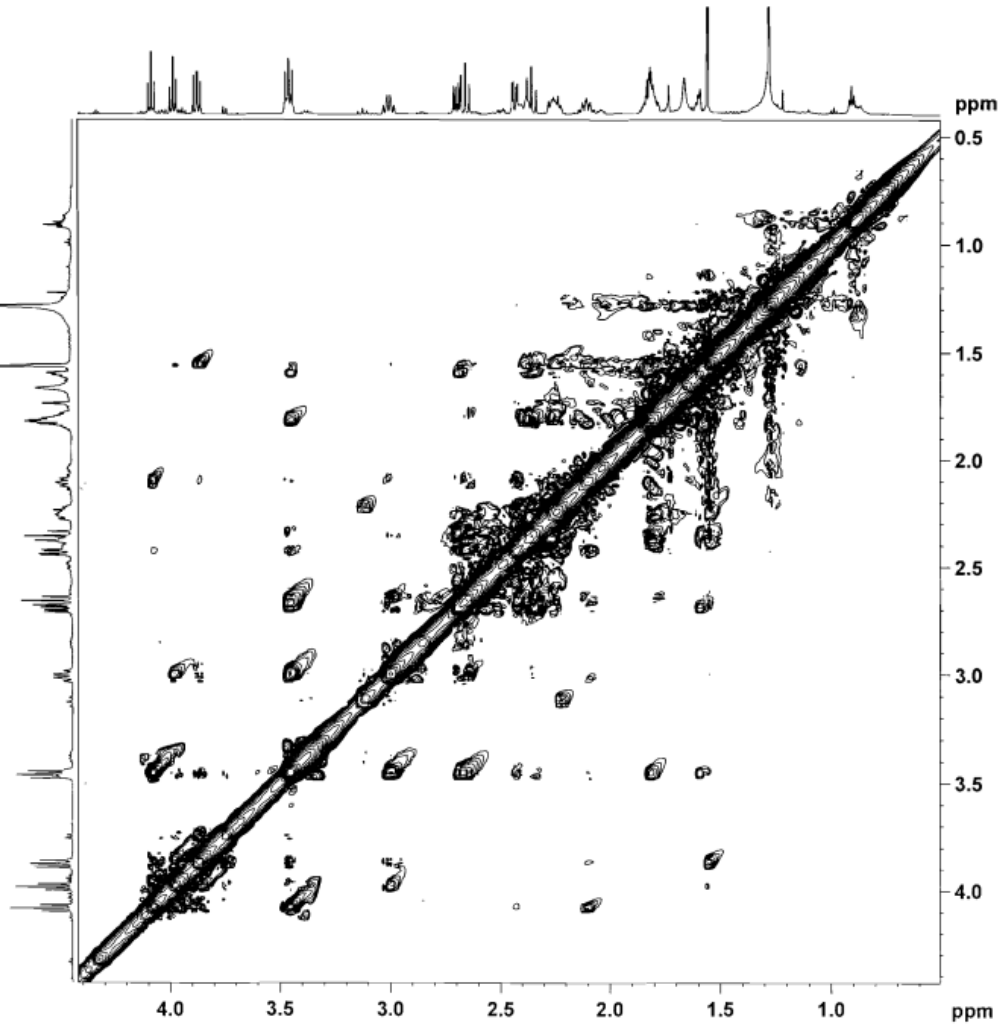


NAME	cosy
EXPNO	84
PROCNO	1
Date_	20091031
Time	3.56
INSTRUM	spect
PROBHD	5 mm BBI 1H-BB
PULPROG	cosygpqf
TD	2048
SOLVENT	CDCl3
NS	8
DS	16
SWH	6613.757 Hz
FIDRES	3.229373 Hz
AQ	0.1549544 sec
RG	574.7
DW	75.600 usec
DE	6.50 usec
TE	295.0 K
D0	0.00000300 sec
D1	1.50000000 sec
D13	0.00000400 sec
D16	0.00002000 sec
IN0	0.00015120 sec
======== CHANNEL f1 ========	
NUC1	1H
P0	4.30 usec
P1	8.60 usec
PL1	-1.00 dB
PL1W	31.62277603 W
SFO1	600.1330006 MHz
====== GRADIENT CHANNEL =====	
GPNAM1	SINE.100
GPZ1	10.00 %
P16	1000.00 usec
ND0	1
TD	256
SFO1	600.133 MHz
FIDRES	25.835022 Hz
SW	11.021 ppm
FnMODE	QF
SI	1024
SF	600.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0
PC	1.40
SI	1024
MC2	QF
SF	600.1300000 MHz
WDW	SINE
SSB	0
LB	0.00 Hz
GB	0

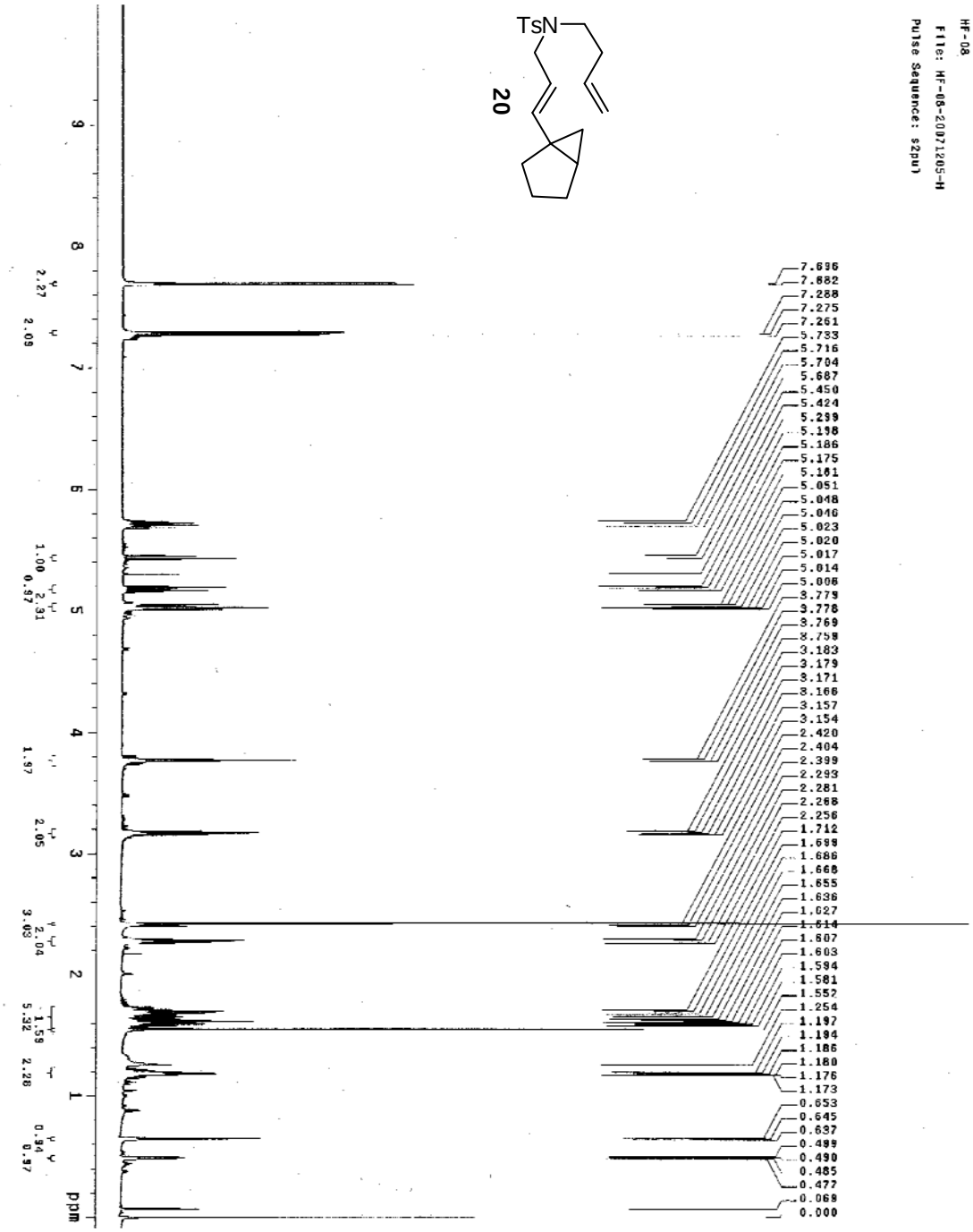
NOESY CDCl3 ZKY-05-22C Yu Zhixiang 20091031



19b



NAME	noesy	
EXPNO	108	
PROCNO	1	
Date_	20091031	
Time	10.17	
INSTRUM	spect	
PROBHD	5 mm BBI 1H-BB	
PULPROG	noesygpph	
TD	2048	
SOLVENT	CDCl3	
NS	8	
DS	16	
SWH	5387.931	Hz
FIDRES	2.630826	Hz
AQ	0.1901972	sec
RG	161.3	
DW	92.800	usec
DE	6.50	usec
TE	295.5	K
D0	0.00008185	sec
D1	2.00000000	sec
D8	0.50000000	sec
D16	0.00020000	sec
IN0	0.00018560	sec
======== CHANNEL f1 ========		
NUC1	1H	
P1	8.60	usec
P2	17.20	usec
PL1	-1.00	dB
PL1W	31.62277603	W
SFO1	600.1324005	MHz
====== GRADIENT CHANNEL =====		
GPNAM1	SINE.100	
GPNAM2	SINE.100	
GPZ1	40.00	%
GPZ2	-40.00	%
P16	1000.00	usec
ND0	1	
TD	256	
SFO1	600.1324	MHz
FIDRES	21.046597	Hz
SW	8.978	ppm
FnMODE	States-TPPI	
SI	1024	
SF	600.1300000	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	
PC	1.40	
SI	1024	
MC2	States-TPPI	
SF	600.1300000	MHz
WDW	QSINE	
SSB	2	
LB	0.00	Hz
GB	0	


HF-08
File: HF-08-20071205-H
Pulse Sequence: s2pul
TsN
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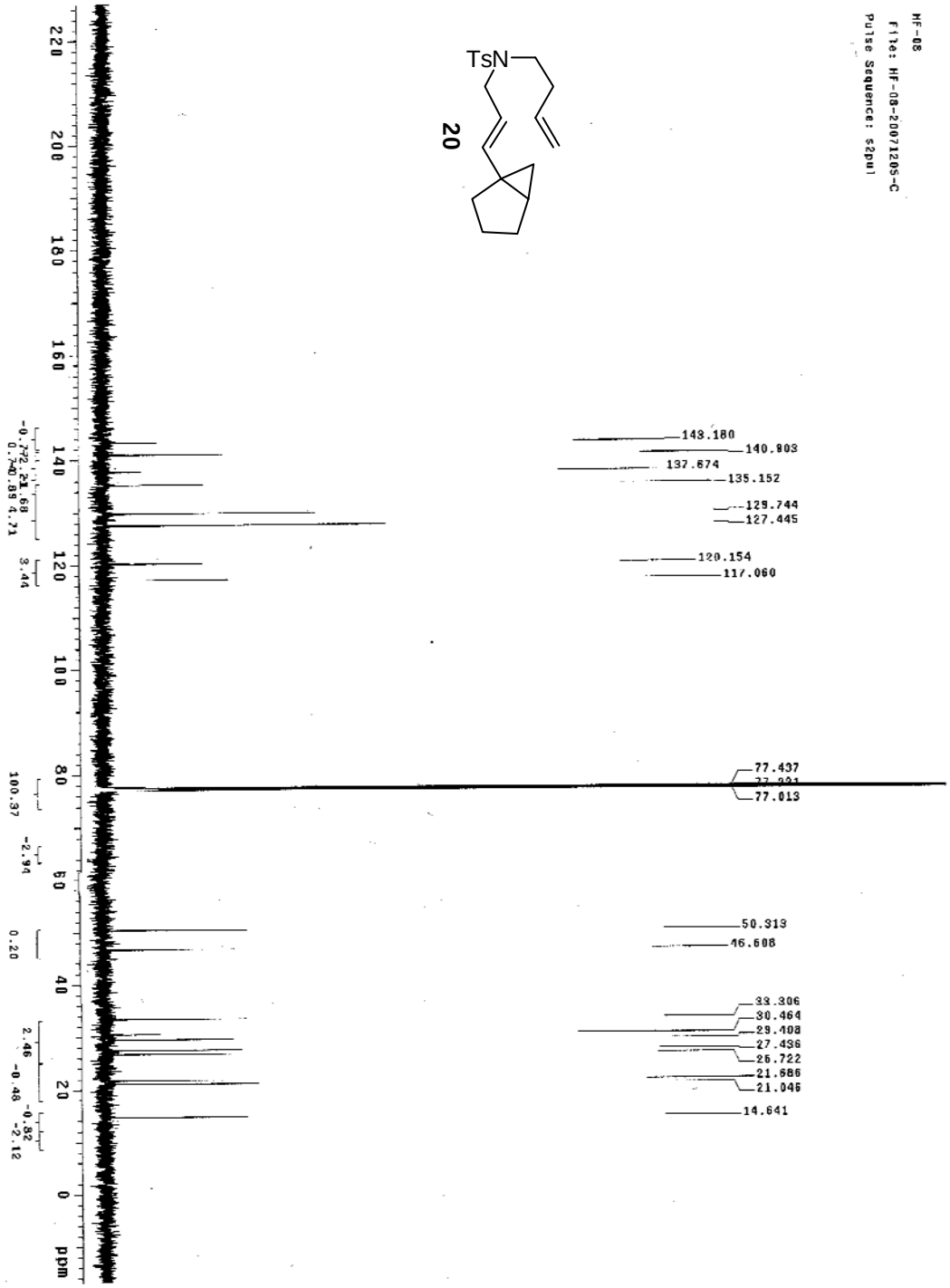
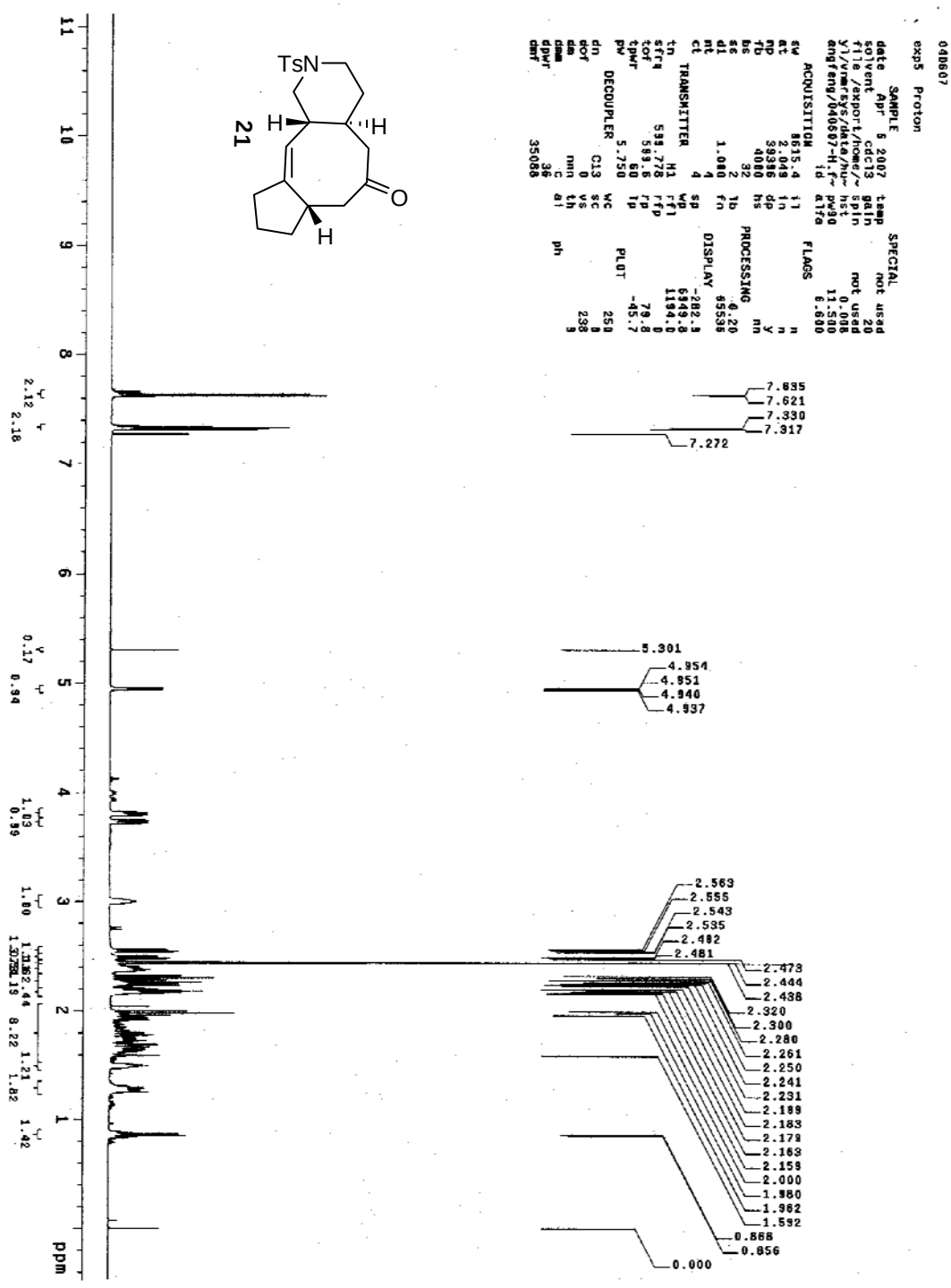
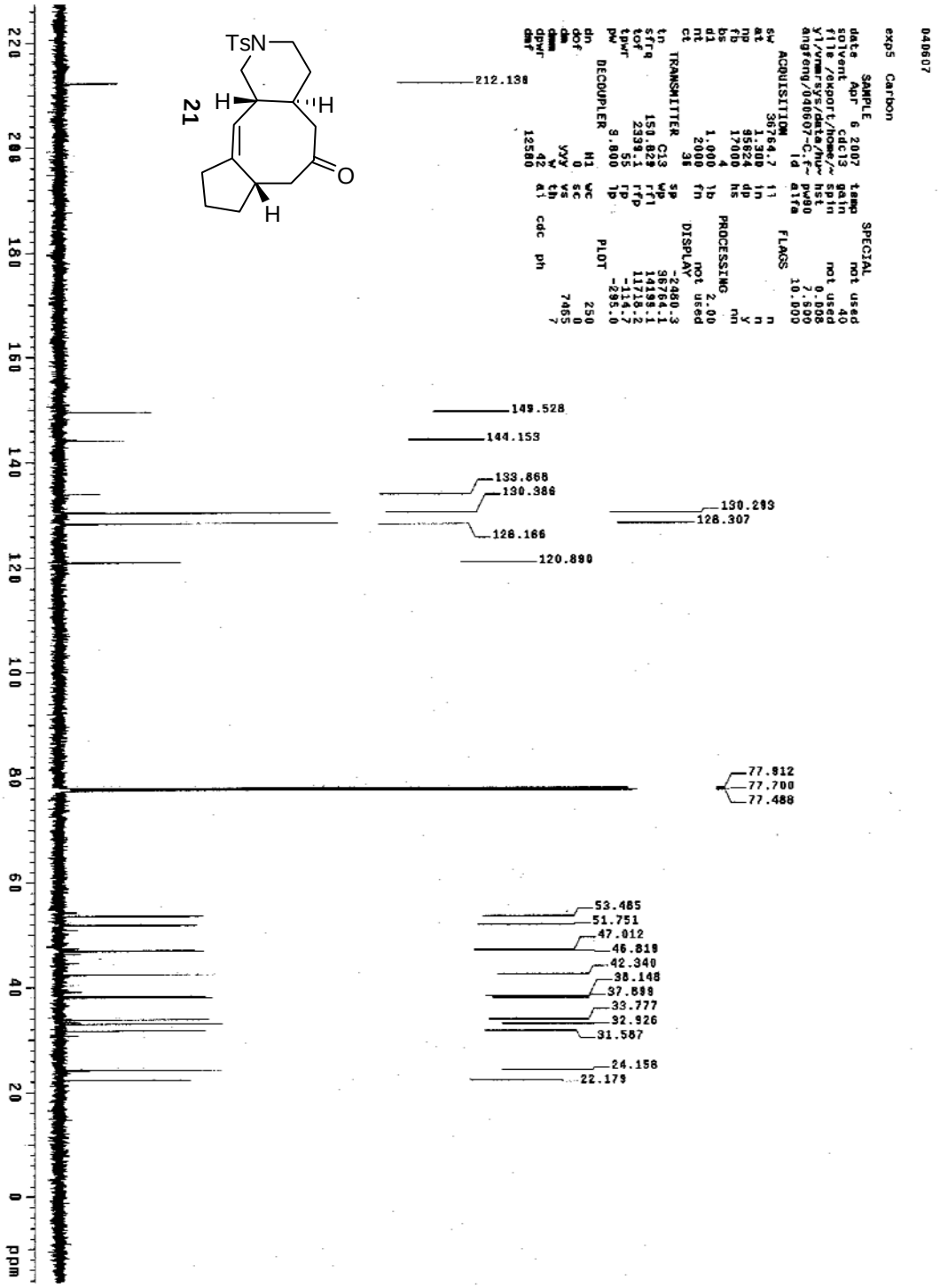
7.882
7.288
7.275
7.261
5.733
5.716
5.704
5.687
5.450
5.424
5.299
5.198
5.186
5.175
5.161
5.051
5.048
5.040
5.023
5.020
5.017
5.014
5.008
3.779
3.778
3.769
3.758
3.183
3.179
3.171
3.166
3.157
3.154
2.420
2.404
2.399
2.293
2.281
2.268
2.256
1.712
1.698
1.686
1.668
1.655
1.636
1.627
1.614
1.607
1.603
1.594
1.581
1.557
1.254
1.197
1.194
1.186
1.180
1.176
1.173
0.653
0.645
0.637
0.499
0.490
0.485
0.477
0.069
0.000
2.27
2.09
1.00
2.31
0.97
1.97
2.05
3.03
2.04
1.59
5.22
2.28
0.94
0.97
9
8
7
6
5
4
3
2
1
ppm


HF-08
File: HF-08-20071205-C
Pulse Sequence: s2pul
TsN
20
148.180
140.903
137.674
135.152
129.744
127.445
120.154
117.060
77.437
77.013
50.319
46.608
33.306
30.464
29.408
27.436
25.722
21.686
21.046
14.641
ppm


TsN
H
H
H
O
21
7.835
7.621
7.330
7.317
7.272
5.301
4.954
4.951
4.940
4.937
2.563
2.555
2.543
2.535
2.482
2.481
2.478
2.444
2.438
2.320
2.300
2.280
2.261
2.250
2.241
2.231
2.189
2.183
2.179
2.168
2.158
2.000
1.980
1.962
1.592
0.868
0.856
0.000
ppm


040607
exp5 Carbon
SAMPLE
date Apr 6 2007
solvent cdcl3
file /export/home/~
yl/vnmrsys/data/hu~
angfeng/040607-C.f~
ACQUISITION
sw 36764.7
at 1.000
np 73524
fb 17000
bs 4
d1 1.000
nt 2000
ct 38
TRANSMITTER
tn C13
sfrq 150.829
tof 2339.1
tpwr 55
pw 8.800
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 42
dmf 12580
temp
gain
spin
hst
pw90
alfa
FLAGS
SPECIAL
not used
40
not used
0.008
7.500
10.000
PROCESSING
DISPLAY
PLOT
cdc ph
TsN
H
H
O
H
21
212.138
149.528
144.153
133.868
130.386
130.293
128.307
128.166
120.890
77.812
77.700
77.488
53.485
51.751
47.012
46.819
42.340
38.148
37.899
33.777
32.926
31.587
24.158
22.179
220
200
180
160
140
120
100
80
60
40
20
0
ppm