Synthesis and NMR Characterization of (*Z*,*Z*,*Z*,*Z*,*E*,*E*,ω)-Heptaprenol

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Supporting Information

Contents

Experimental Procedures.

General Information. All reactions were performed under an atmosphere of nitrogen, unless noted otherwise. Thin-layer chromatography was performed with Whatman reagents 0.25-mm silica gel 60-F plates. Column chromatography was carried out with silica gel 60, 230–400 mesh (0.040–0.063 mm particle size) purchased from EM Science. High-resolution mass spectra were obtained at the Department of Chemistry and Biochemistry, University of Notre Dame by ESI ionization, using Bruker micrOTOF/Q2 mass spectrometer.

NMR Spectroscopy. 1D ^{1}H, ^{13}C{^{1}H}, Dept and 2D homo-COSY and heteronuclear ^{1}H-^{13}C HETCOR spectra were recorded on a Varian INOVA-500 or Varian DirectDrive 600 spectrometer. For compounds **18-OH**, **20-OH**, **7b**, **4**, and **4B2**, 1D ^{1}H and ^{13}C{^{1}H}, and 2D homo- DQF-COSY, TOCSY, ROESY and heteronuclear ^{1}H-^{13}C HSQC, HSQC-TOCSY, HMBC spectra were recorded on a Bruker ADVANCE II 800. The structures of prenol **4** and **4B2** [10 mg of each was dissolved in 600 μL $CDCl_3$ containing 3 mg $Eu(hfc)_3$] were determined by interpretation of the homonuclear DQF-COSY, TOCSY, ROESY and heteronuclear ^{1}H-^{13}C HSQC, HSQC-TOCSY, HMBC NMR spectra. Proton connectivities were derived from the DQF-COSY and TOCSY spectra. ^{13}C resonances corresponding to carbons with directly attached protons were assigned using HSQC and HSQC-TOCSY spectra. HMBC spectra were used to assign resonances of the quaternary carbons and to validate the connectivities established by the other spectra. ROESY spectra were utilized to establish the *E* or *Z* configurations for each C=C double bond. Experiments were performed at 25 ^{o}C using a Bruker AVANCE II spectrometer equipped with a TCI cryoprobe and operating at a ^{1}H resonance frequency of 800.13 MHz. Standard pulse sequences were used.[1-6] Time domain data (t_2 and t_1) for 2D experiments were recorded as 2048 × 1024 complex matrices with 16 and 32 scans per t_1 increment for homonuclear and heteronuclear spectra, respectively. Relaxation delay between individual scans and spin-lock time for TOCSY experiments was 1.4 s and 60 ms, respectively. For ROESY experiments, a relaxation delay of 4 s and a spin-lock time of 400 ms gave 2048 × 512 complex time domain points. Linear prediction to 1024 complex points was applied in

the t_1 domain. The data were zero filled to obtain final 2048 × 2048 complex matrices. In all other homonuclear 2D experiments, zero filling was used only in the t_1 domain to obtain final 2048 × 2048 complex time domain data. In heteronuclear 2D experiments, linear prediction to 2048 complex data points was employed in the t_1 domain, which was zero filled to 4096 to get final 2048 × 4096 complex time domain data. Shifted sine bell weighting functions were applied in both domains prior to double Fourier transformation. In the 3D ^{1}H-^{13}C-HSQC-TOCSY experiments, 1024 × 48 × 128 complex time domain points in t_3, t_2, and t_1 domains were collected with 16 scans per time increment, relaxation delay of 1.3 s, and 60 ms spin-lock. Linear predictions to 128 and 256 complex points were utilized in the t_2 and t_1 domains. Zero filling gave 2048 × 256 × 512 complex matrices. Shifted sine bell weighting functions were applied in all three domains prior to triple Fourier transformation. Spectra were processed using Bruker TopSpin 2.1 software. ^{1}H spectra, the ^{1}H dimension in 2D heteronuclear spectra, and the 1D ^{13}C{^{1}H} spectra were referenced to solvent (DMSO, δ_H 2.5 and δ_C 39.5; $CDCl_3$, δ_H 7.27 and δ_C 77.23). The ^{13}C dimension in the 2D heteronuclear spectra was referenced indirectly.[7]

Six functional group transformations, used for the synthesis of compounds described in this paper, are given as general procedures A – F.

General procedure A. **Deprotection of *O*-THP ether of prenol.**

THP-protected ether (20 mmol) was dissolved in MeOH (100 mL) and $TsOH{\cdot}H_2O$ (0.6 g) was added. The resulting mixture was stirred for 6 h at room temperature. After addition of Et_3N (0.5 mL), the resulting solution was filtered through a layer of silica gel and concentrated under reduced pressure. The crude compound was used for the next step without further purification or it was purified by short-path column chromatography on silica gel.

General procedure B. **Bromination of prenol.**

B1. Triethylamine (3.3 mL, 24 mmol) was added to a solution of alcohol (20 mmol) in anhydrous THF (60 mL) and the solution was cooled down in an ice-water bath. To this solution, MsCl (1.9 mL, 24 mmol) was added dropwise. After stirring for 1 h in an ice-water bath, the reaction mixture was quickly filtered through a layer of Celite to a pre-cooled THF solution of LiBr (2.1 g, 24 mmol in 20 mL of THF) and Celite pad was quickly washed with THF (2 × 10 mL). The combined solution was stirred for 2 h in an ice-water bath. The volatiles were evaporated under reduced pressure and the resulting bromide was taken up in hexanes/ethyl acetate mixture (1:1, 200 mL). The solution was washed with water (50 mL × 2) and brine (100 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The crude compound was used for the next step without further purification or it was purified by short-path column chromatography on silica gel.

B2. Phosphorus tribromide (0.83 mL, 8.7 mmol) in dry ether (20 mL) wad added to a mixture of alcohol (20 mmol) and pyridine (0.15 mL) in dry ether (50 mL) over 1 h in an ice-water bath. After stirring for 2 h in an ice-water bath, it was poured into ice-water (100 mL) and then extracted with hexanes (50 mL× 3). The organic layer was washed with water (50 mL× 2), saturated $NaHCO_3$ (50 mL× 2), and brine (50 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel chromatography.

B3. Same as general procedure C except using LiBr, instead of LiCl

General procedure C. **Chlorination of prenol.**

A cold solution of alcohol (20 mmol) and LiCl (2.1 g, 50 mmol) in DMF (200 mL) was treated with *s*-collidine (6.6 mL, 50 mmol) and MsCl (3.9 mL, 50 mmol) in an ice-water bath. After stirring in an ice-water bath for 2 h, the reaction mixture was diluted with ethyl acetate (800 mL). The mixture was washed with saturated NH_4Cl (300 mL × 2) and brine (300 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure to afford the corresponding chloride. The crude compound was

used for the next step without further purification or it was purified by short-path column chromatography on silica gel.

General procedure D. **Sulfonylation of prenyl halide.**

The halide (chloride or bromide, 20 mmol) in acetonitrile (10 mL) was added dropwise to a suspension of sodium *p*-toluenesulfinate (4.3 g, 24 mmol) in DMF (120 mL) in an ice-water bath. After the reaction mixture was stirred at room temperature for 6 h, it was diluted with ethyl acetate (500 mL). The mixture was washed with water (200 mL × 2) and brine (200 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated to afford the corresponding sulfone. The crude compound was purified by column chromatography on silica gel.

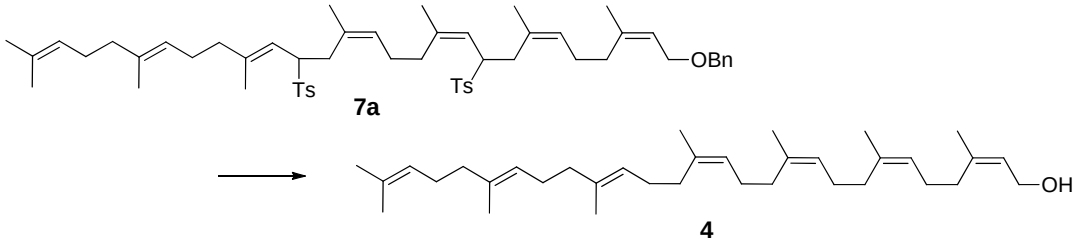
General procedure E. **Coupling of prenyl sulfone and prenyl bromide.**

Sulfone (20 mmol) was dissolved in a mixture of anhydrous THF and HMPA (4:1, 80 mL) and the resulting solution was cooled to –78 ^{o}C. To this cold solution, *n*-BuLi (12.5 mL, 20 mmol, 1.6 M in hexane) was added dropwise and stirring was continued for 1.5 h. A solution of bromide (17.4 mmol) in THF-HMPA (4:1, 20 mL) was added dropwise to a solution of sulfone over 1 h. The mixture was stirred for an additional 5 h. After warming the mixture to 0 ^{o}C, it was poured into ice-water and extracted with hexanes-ether (1:1, 150 mL × 2). The extract was washed with water (150 mL) and brine (150 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The residue was purified by column chromatography on silica gel to afford the product.

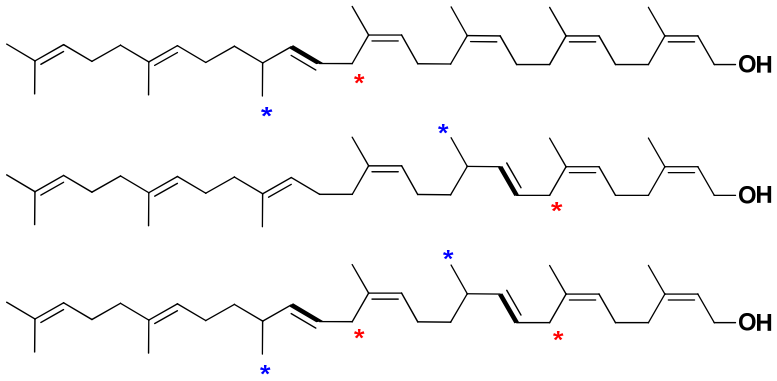
General procedure F. **Deprotection of benzyl ether and desulfonylation.**

Transformation of 7a to 4 and 7b to 4/4B2 are given as representative examples.

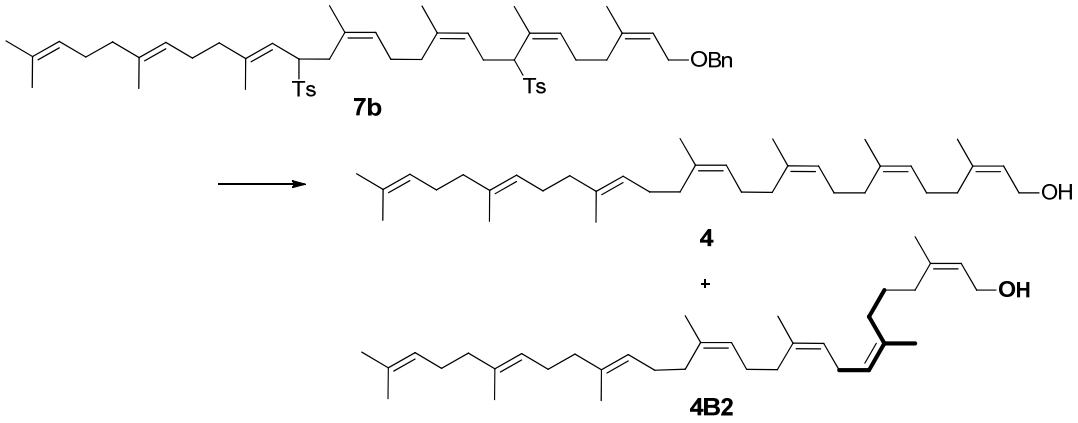
Synthesis of compound 4 from 7a.



Ethylamine (150 mL) was added to a flask containing lithium (1.0 g, 0.14 mol) at –78 °C and ether (50 mL) was added. After stirring for 10 min at –78 °C, an ethereal solution of compound **7a** (6.0 g, 6.7 mmol in 50 mL) was added dropwise to the blue solution over 20 min. After stirring for 40 min at –78 °C, the reaction was quenched by addition of isoprene (10 mL) and MeOH (50 mL). After addition of saturated NH_4Cl (200 mL), the reaction mixture was extracted with ethyl acetate (100 mL × 3). The combined organic layer was washed with brine (150 mL), dried over anhydrous $MgSO_4$, filtered through a layer of silica gel, and concentrated under reduced pressure. The residue (2.8 g) was purified by column chromatography (silica gel, 35 g; i.d. of column, 3/4 inch; ethyl acetate:hexane, 1:7) to afford the desired product (2.0 g, 59%). The purity of **4** is >90% as assessed by ^1H NMR. As isolated by the silica flash chromatography **4** contains < 10% of impurities presumed to arise as a result of double bond isomerization. Neither the number of these impurities nor their structures were determined. The structures of the three impurities anticipated as possibly present are shown below. We anticipate greater purity of **4** can be achieved by column chromatography using silver nitrate-impregnated silica gel.[8-10]

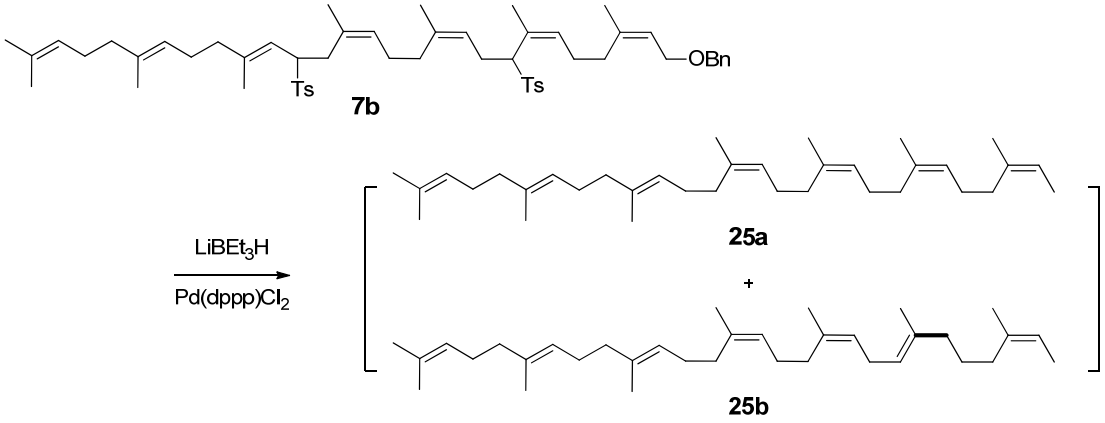


Synthesis of compounds 4/4B2 from 7b.



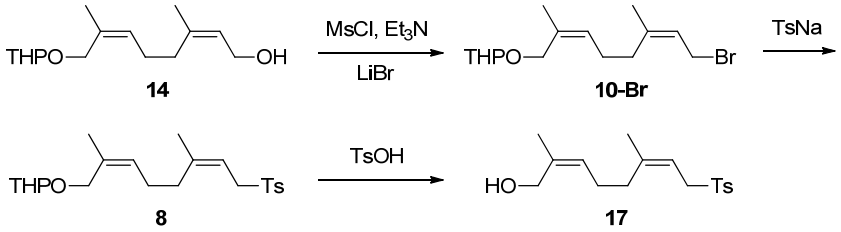
Ethylamine (200 mL) was added to a flask containing lithium (1.9 g, 0.27 mol) at –78 °C and ether (100 mL) was added. After stirring for 10 min at –78 °C, an ethereal solution of compound **7b** (8.0 g, 9.0 mmol in 100 mL) was added dropwise to the blue solution over 20 min. After stirring for 40 min at –78 °C, the reaction was quenched by addition of isoprene (15 mL) and MeOH (70 mL). After addition of saturated NH_4Cl (300 mL), the reaction mixture was extracted with ethyl acetate (150 mL × 3). The combined organic layer was washed with brine (200 mL), dried over anhydrous $MgSO_4$, filtered through a layer of silica gel, and concentrated under reduced pressure. The residue (3.5 g) was purified by column chromatography (silica gel, 50 g; i.d. of column, 1 inch; ethyl acetate:hexanes, 1:7) to afford the mixture of **4**/**4B2** (2.8 g, 1:3 mixture, 63%). The mixture was further subjected to column chromatography (silica gel, 50 g; i.d. of column, 3/4 inch; ethyl acetate:hexanes, 1:15) to afford **4** (0.23 g), **4B2** (0.81 g), and **4**/**4B2** mixture (1.6 g).

Deprotection of benzyl ether and desulfonylation of 7b using $LiBEt_3H/Pd(dppp)Cl_2$.



$LiBEt_3H$ (10 mL, 10 mmol, 1.0 M solution in THF) was added dropwise over 15 min to a cold solution of compound **7b** (1.8 g, 2.0 mmol) and bis(diphenylphosphino)propanepalladium(II) dichloride ($Pd(dppp)Cl_2$ (0.24 g, 0.40 mmol) in anhydrous THF (40 mL) in an ice-water bath. Stirring was continued for 4 h, and the reaction mixture was diluted with hexanes:ether (1:1, 200 mL). The mixture was washed with saturated NH_4Cl (100 mL × 2) and brine (100 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. ^1H NMR analysis indicated that the crude material has at least two products. TLC analysis showed that the major products were considerably less polar than expected for **4** (heptaprenol). The major material obtained from column chromatography (silica gel, 15 g; i.d. of column, 1/2 inch; hexanes) of this crude product was 0.78 g of a 1:3 mixture (by ^1H NMR analysis) of hydrocarbons (arising from reductive desulfonylation and deoxygenation). The possible structures of the two isolated reaction products are formulated as **25a** and **25b** (major). These structures are suggested by the absence any aromatic resonances, and the further absence of the resonance anticipated for the methylene of (either a free or an *O*-Bn-protected) allyl alcohol in ^1H NMR spectrum. As a result, further evaluation of this method for desulfonylation was not made.

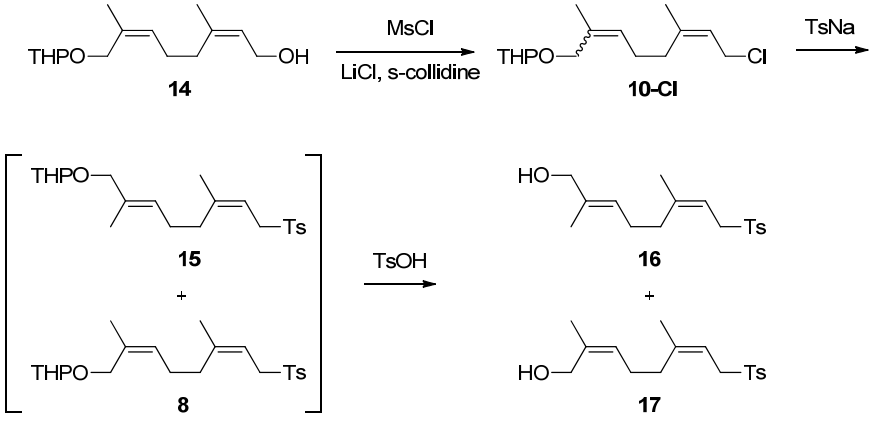
Synthesis of compounds 17 from 14 using general procedures B1/D/A



Triethylamine (17 mL, 122 mmol) was added to a solution of **14** (26 g, 102 mmol) in anhydrous THF (300 mL) and the solution was cooled down in an ice-water bath. To this solution, MsCl (9.4 mL, 122 mmol) was added dropwise. After stirring for 1 h in an ice-water bath, the reaction mixture was quickly filtered through a layer of Celite to a pre-cooled THF solution of LiBr (10.4 g, 120 mmol in 20 mL of THF) and Celite pad was quickly washed with THF (2 × 100 mL). The combined solution was stirred for 2 h in an ice-water bath. The volatiles were evaporated under reduced pressure. The crude bromide was taken up in hexanes/ethyl acetate mixture (1:1, 500 mL). The solution was washed with water (300

mL) and brine (300 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure. The crude compound (**10-Br**, ~27 g) in acetonitrile (60 mL) was added dropwise to a suspension of sodium *p*-toluenesulfinate (21.4 g, 120 mmol) in DMF (250 mL) in an ice-water bath. After the reaction mixture was stirred at room temperature for 6 h, it was diluted with ethyl acetate (800 mL). The mixture was washed with water (400 mL × 2) and brine (400 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated to afford crude compound. It was purified by column chromatography (silica gel, 350 g; i.d. of column, 3 inch; ethyl acetate:hexanes, 1:5 to 1:3) to give **8** (31 g, 78%) and **8/15** mixture (3 g). A small amount of **8** was taken for the next step. Compound **8** (1.0 g, 2.5 mmol) was dissolved in MeOH (10 mL) and $TsOH{\cdot}H_2O$ (0.1 g) was added. The resulting mixture was stirred for 6 h at room temperature. After addition of Et_3N (50 μL), the resulting solution was filtered through a layer of silica gel and concentrated under reduced pressure. The crude compound was purified by column chromatography (silica gel, 10 g; i.d. of column, 1/2 inch; ethyl acetate:hexanes, 1:1) to give **17** (0.63 g, 80%).

Synthesis of compounds 16/17 from 14 using general procedures C/D/A



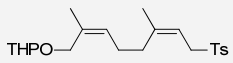
A cold solution of **14** (10 g, 39 mmol) and LiCl (4.1 g, 97 mmol) in DMF (300 mL) was treated with *s*-collidine (13 mL, 98 mmol) and MsCl (7.6 mL, 98 mmol) in an ice-water bath. After stirring at ice-water temperature for 2 h, the reaction mixture was diluted with ethyl acetate (800 mL). The mixture was washed with saturated NH_4Cl (300 mL × 2) and brine (300 mL × 2), dried over anhydrous $MgSO_4$, filtered, and concentrated under reduced pressure to afford **10-Cl**. The crude compound (**10-Cl**, ~10 g)

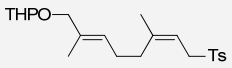
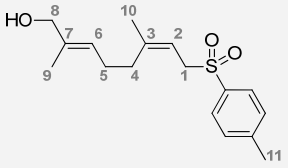
in acetonitrile (20 mL) was added dropwise to a suspension of sodium *p*-toluenesulfinate (8.2 g, 46 mmol) in DMF (200 mL) in an ice-water bath. After the reaction mixture was stirred at room temperature for 6 h, it was diluted with ethyl acetate (600 mL). The mixture was washed with water (200 mL × 2) and brine (200 mL), dried over anhydrous $MgSO_4$, filtered, and concentrated to afford a mixture of compounds **8** and **15** (8.2 g, 53% combined yield). NMR analysis showed a 5:4 ratio of **8**:**15**. A sample of the mixture (1.0 g) was purified by column chromatography (silica gel, 15 g; i.d. of column, 1/2 inch; ethyl acetate:hexanes, 1:5) to give **8** (0.35 g) and **15** (0.30 g).

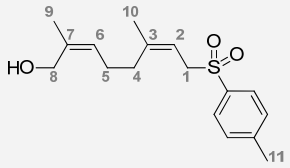
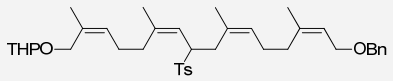
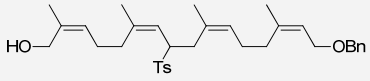
The **8**/**15** mixture (2.0 g, 5.1 mmol) was dissolved in MeOH (20 mL) and $TsOH{\cdot}H_2O$ (0.2 g) was added. The resulting mixture was stirred for 6 h at room temperature. After addition of Et_3N (100 μL), the resulting solution was filtered through a layer of silica gel and concentrated under reduced pressure. The crude compound was purified by column chromatography (silica gel, 30 g; i.d. of column, 3/4 inch; ethyl acetate:hexanes, 1:1) to give **16/17** as a mixture (1.3 g, 81% combined yield). The mixture was subjected to column chromatography (silica gel, 20 g; i.d. of column, 1/2 inch; ethyl acetate:hexanes, 2:3) to give **16** (0.30 g), **17** (0.40 g), and a **16/17** mixture (0.50 g).

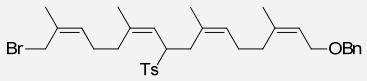
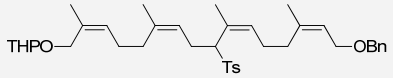
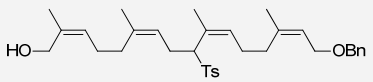
Table S1. Characterization data of synthesized compounds.

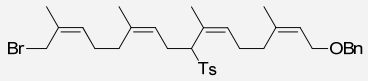
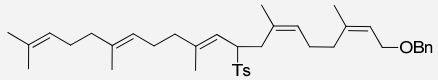
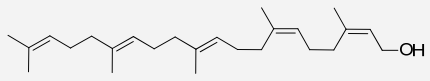
Spectral data of synthesized compounds are given in Table S1. The used preparation procedure used for each compound is indicated. When the compound was known in the literature, the literature is given.

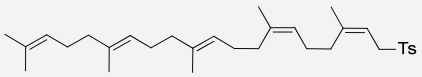
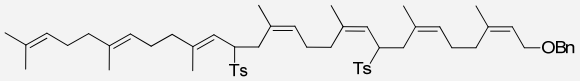
Compound No	
8	
Procedure used	B1 or B3 followed by D
Yield	31 g (78%, B1) and 2 g (57%, B3) from **14**
Lit	Ref[11]
	No spectroscopic characterization is given.
^{1}H NMR	(500 MHz, $CDCl_3$) δ 1.46 - 1.61 (m, 4H), 1.64 - 1.68 (m, 1H), 1.70 (2 × s, 6H), 1.73 - 1.83 (m, 3H), 1.93 (q, *J* = 7.6 Hz, 2H), 2.41 (s, 3H), 3.45 - 3.51 (m, 1H), 3.77 (dd, *J* =

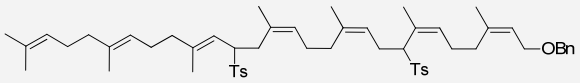
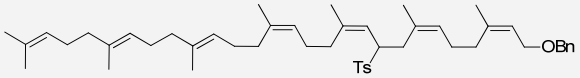
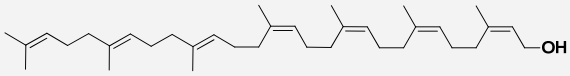
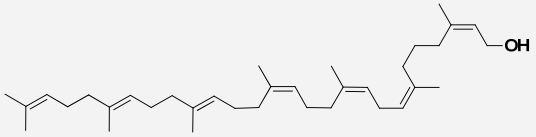
	7.8, 3.2 Hz, 2H), 3.80 - 3.86 (m, 1H), 4.00 (dd, *J* = 20.5, 11.4 Hz, 2H), 4.53 (t, *J* = 3.4 Hz, 1H), 5.18 (q, *J* = 7.2 Hz, 2H), 7.30 (d, *J* = 8.0 Hz, 2H), 7.72 (d, *J* = 8.2 Hz, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 19.3 (CH_2), 21.5 (CH_3), 21.6 (CH_3), 23.4 (CH_3), 25.4 (CH_2), 25.5 (CH_2), 30.5 (CH_2), 31.9 (CH_2), 55.7 (CH_2), 62.0 (CH_2), 65.0 (CH_2), 97.1 (CH), 111.2 (vinyl CH), 128.1 (vinyl CH), 128.3 (CH), 129.5 (CH), 132.6 (C), 135.8 (C), 144.4 (C), 145.3 (C)
HRMS (ESI)	calcd for $C_{22}H_{32}NaO_4S$ ($M+Na^+$) 415.1914, found 415.1912
15	
Procedure used	C followed by D
Yield	8.2 g (53%, as 5:4 mixture of **8**:**15**) from **14** 0.3 g (**15**, isolated from 1.0 g of **8**/**15** mixture)
^{1}H NMR	(500 MHz, $CDCl_3$) δ 1.43 - 1.55 (m, 4H), 1.56 (s, 3H), 1.62 - 1.68 (m, 1H), 1.69 (s, 3H), 1.78 (t, *J* = 7.7 Hz, 3H), 1.84 - 1.94 (m, 2H), 2.39 (s, 3H), 3.41 - 3.51 (m, 1H), 3.71 - 3.77 (m, 2H), 3.78 - 3.86 (m, 1H), 4.02 (d, *J* = 11.6 Hz, 1H), 4.53 (t, *J* = 3.6 Hz, 1H), 5.16 (t, *J* = 7.7 Hz, 1H), 5.24 (dd, *J* = 7.0, 6.2 Hz, 1H), 7.28 (d, *J* = 8.2 Hz, 2H), 7.69 (d, *J* = 8.2 Hz, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 13.9 (CH_3), 19.3 (CH_2), 21.5 (CH_3), 23.3 (CH_3), 25.3 (CH_2), 25.5 (CH_2), 30.5 (CH_2), 31.3 (CH_2), 55.8 (CH_2), 62.0 (CH_2), 72.5 (CH_2), 97.4 (CH), 111.0 (vinyl CH), 126.3 (vinyl CH), 128.2 (CH), 129.4 (CH), 132.6 (C), 135.7 (C), 144.3 (C), 145.4 (C)
HRMS (ESI)	calcd for $C_{22}H_{32}NaO_4S$ ($M+Na^+$) 415.1914, found 415.1902
16	
Procedure used	A
Yield	1.3 g (81%) from **8**/**15** mixture 0.3 g (**16**, isolated from **16**/**17** mixture)
^{1}H NMR	(600 MHz, DMSO-d_6) δ 1.47 (s, 3H, H-9), 1.67 (s, 3H, H-10), 1.81 (s, 4H, H-4, H-5), 2.38 (s, 3H, H-11), 3.72 (s, 2H, H-8), 3.94 (d, *J* = 7.7 Hz, 2H, H-1), 5.05 (t, *J* = 7.8 Hz, 1H, H-2), 5.20 (br. s., 1H, H-6), 7.41 (d, *J* = 7.9 Hz, 2H), 7.71 (d, *J* = 8.3 Hz, 2H)
^{13}C NMR	(151 MHz, DMSO-d_6) δ 13.4 (C-9), 21.0 (C-11), 23.2 (C-10), 25.2, 31.2 (C-4, C-5), 54.8 (C-1), 66.3 (C-8), 111.3 (C-2), 122.7 (C-6), 127.9, 129.6, 135.7, 136.1, 144.1, 144.7
HRMS (ESI)	calcd for $C_{17}H_{24}NaO_3S$ ($M+Na^+$) 331.1338, found 331.1334

17	
Procedure used	A
Yield	0.6 g (80%) from **8**
Lit	Ref[11]
	No spectroscopic characterization is given.
^{1}H NMR	(600 MHz, $CDCl_3$) δ 1.69 (s, 3H, H-10), 1.72 (s, 3H, H-9), 1.76 - 1.86 (m, 2H, H-4), 1.89 - 2.03 (m, 2H, H-5), 2.40 (s, 3H, H-11), 3.74 (d, *J* = 7.9 Hz, 2H, H-1), 4.00 (s, 2H, H-8), 5.12 (q, *J* = 8.2 Hz, 2H, H-6, H-2), 7.29 (d, *J* = 8.1 Hz, 2H), 7.69 (d, *J* = 8.1 Hz, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 21.3 (C-9), 21.5 (C-11), 23.4 (C-10), 25.5 (C-5), 32.0 (C-4), 55.7 (C-1), 61.0 (C-8), 110.9 (C-2), 126.7 (C-6), 128.2, 129.5, 135.3, 135.7, 144.5, 145.7
HRMS (ESI)	calcd for $C_{17}H_{24}NaO_3S$ ($M+Na^+$) 331.1338, found 331.1330
18	
Procedure used	E
Yield	35 g (63%) from **8**
Lit	Ref[11]
	No spectroscopic characterization is given.
^{1}H NMR	(500 MHz, $CDCl_3$) δ 1.60, 1.64, 1.72 (4 × s, 12H), 1.44 -1.91 (m, 10H), 1.92 - 2.10 (m, 4H), 2.41 (s, 3H), 2.46 (dt, *J* = 11.4, 6.6 Hz, 1H), 2.68 (d, *J* = 13.2 Hz, 1H), 3.44 - 3.57 (m, 1H), 3.80 - 3.90 (m, 2H), 3.91 - 4.07 (m, 4H), 4.41 - 4.51 (m, 2H), 4.54 (br. s., 1H), 4.95 (d, *J* = 10.6 Hz, 1H), 5.07 - 5.20 (m, 2H), 5.41 (t, *J* = 6.4 Hz, 1H), 7.21 - 7.40 (m, 6H), 7.66 - 7.74 (m, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 19.3, 19.4, 21.5, 21.6, 23.2, 23.4, 23.5, 25.4, 25.4, 26.4, 30.1, 30.1, 30.5, 32.0, 32.1, 32.1, 62.0, 63.1, 63.2, 65.0, 65.0, 66.2, 72.0, 97.2, 97.3, 117.8, 122.1, 127.4, 127.7, 128.0, 128.1, 128.1, 128.2, 129.1, 129.3, 130.4, 132.4, 132.4, 134.8, 138.4, 139.9, 144.3, 144.6, 144.6
HRMS (ESI)	calcd for $C_{39}H_{54}NaO_5S$ ($M+Na^+$) 657.3584, found 657.3587
18-OH	
Procedure used	A
Yield	14 g (93%) from **18**
Lit	Ref[11]

	No spectroscopic characterization is given.
^{1}H NMR/^{13}C NMR	Full assignment is given in Table S3.
HRMS (ESI)	calcd for $C_{34}H_{46}NaO_4S$ ($M+Na^+$) 573.3009, found 573.3010
19	
Procedure used	B2
Yield	10 g (74%) from **18-OH**
Lit	Ref[11]
	No spectroscopic characterization
^{1}H NMR	^{1}H NMR (600 MHz, $CDCl_3$) δ 1.59, 1.68, 1.71, 1.78 (4 × s, 12H), 1.65 - 1.91 (m, 4H), 1.94 - 2.11 (m, 4H), 2.42 (s, 3H), 2.66 (d, *J* = 13.2 Hz, 1H), 3.79 - 3.94 (m, 3H), 3.97 (d, *J* = 6.8 Hz, 2H), 4.48 (s, 2H), 4.99 (d, *J* = 10.6 Hz, 1H), 5.12 - 5.27 (m, 2H), 5.40 (t, *J* = 6.1 Hz, 1H), 7.23 - 7.37 (m, 6H), 7.72 (d, *J* = 8.1 Hz, 2H)
^{13}C NMR	(151 MHz, $CDCl_3$) δ 21.6, 21.8, 23.3, 23.4, 23.7, 26.0, 26.4, 30.4, 31.4, 31.9, 32.0, 63.3, 66.2, 72.0, 118.0, 122.1, 127.4, 127.7, 128.1, 128.2, 129.1, 129.3, 130.2, 130.4, 132.2, 134.8, 138.4, 139.8, 144.2, 144.4
HRMS (ESI)	calcd for $C_{34}H_{45}BrNaO_3S$ ($M+Na^+$) 635.2165, found 635.2170
20	
Procedure used	E
Yield	38 g (83%) from **11**
^{1}H NMR	(500 MHz, $CDCl_3$) δ 1.61, 1.63, 1.77, 1.79 (4 × s, 12H), 1.47 - 1.90 (m, 10H), 1.96 - 2.06 (m, 2H), 2.11 (q, *J* = 7.3 Hz, 2H), 2.41 (s, 3H), 2.59 (m, *J* = 11.7, 6.3 Hz, 1H), 2.74 - 2.82 (m, 1H), 3.51 (dd, *J* = 11.0, 5.0 Hz, 1H), 3.85 - 3.99 (m, 3H), 3.99 - 4.13 (m, 2H), 4.43 - 4.50 (m, 2H), 4.59 (t, *J* = 3.1 Hz, 1H), 4.82 (t, *J* = 6.4 Hz, 1H), 5.29 (t, *J* = 6.9 Hz, 1H), 5.33 - 5.45 (m, 2H), 7.24 - 7.39 (m, 6H), 7.71 (d, *J* = 8.0 Hz, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 19.4 (CH_2), 21.5 (CH_3), 21.7 (CH_3), 23.2 (CH_3), 23.3 (CH_2), 25.4 (CH_2), 25.8 (CH_2), 25.9 (CH_2), 30.6 (CH_2), 31.3 (CH_2), 32.1 (CH_2), 62.0 (CH_2), 62.1 (CH_2), 65.1 (CH_2), 66.1 (CH, CH_2), 72.1 (CH_2), 97.3 (CH), 119.4, 122.4, 126.3, 127.5, 127.6, 128.3, 128.6, 128.7, 129.4, 132.3, 135.1, 135.3, 138.1, 138.3, 139.3, 144.3
HRMS (ESI)	calcd for $C_{39}H_{54}NaO_5S$ ($M+Na^+$) 657.3584, found 657.3591
20-OH	
Procedure used	A
Yield	16 g (80%) from **20**
^{1}H NMR/^{13}C NMR	Full assignment is given in Table S3.

HRMS (ESI)	calcd for $C_{34}H_{46}NaO_4S$ ($M+Na^+$) 573.3009, found 573.2995
21	
Procedure used	B1 or B2
Yield	3.4 g (55%) or 10.8 (88%) from **20-OH**
^{1}H NMR	^{1}H NMR (500 MHz, $CDCl_3$) δ 1.40 - 1.57 (m, 1H), 1.64, 1.68, 1.82, 1.86 (4 × s, 12H), 1.73 - 1.94 (m, 3H), 2.01 - 2.19 (m, 4H), 2.44 (s, 3H), 2.56 - 2.67 (m, 1H), 2.78 - 2.88 (m, 1H), 3.90 - 4.03 (m, 5H), 4.49 (s, 2H), 4.88 (t, *J* = 6.5 Hz, 1H), 5.27 - 5.45 (m, 3H), 7.28 - 7.40 (m, 7H), 7.74 (d, *J* = 8.2 Hz, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 21.6, 21.9, 23.3, 23.4, 26.0, 26.3, 31.4, 31.4, 32.0, 66.2, 66.3, 72.1, 119.8, 122.4, 126.4, 127.5, 127.7, 128.3, 128.8, 129.4, 130.6, 132.1, 135.3, 137.7, 138.3, 139.4, 144.4
HRMS (ESI)	calcd for $C_{34}H_{45}BrNaO_3S$ ($M+Na^+$) 635.2165, found 635.2170
22	
Procedure used	E
Yield	9.0 g (71%) from **12**
^{1}H NMR	(500 MHz, acetone-d_6) δ 1.58, 1.59, 1.61, 1.65, 1.74 (5 × s, 15H), 1.09 - 1.30 (m, 3H), 1.59 (d, *J* = 4.6 Hz, 6H), 1.61 (s, 3H), 1.65 (s, 4H), 1.74 (s, 3H), 1.92 - 2.12 (m, 14H), 2.43 (s, 3H), 2.50 (dd, *J* = 13.0, 11.4 Hz, 1H), 2.70 (dd, *J* = 13.2, 2.4 Hz, 1H), 3.98 (td, *J* = 11.0, 3.2 Hz, 1H), 4.02 (d, *J* = 6.8 Hz, 2H), 4.48 (s, 2H), 4.95 (d, *J* = 10.0 Hz, 1H), 5.06 - 5.16 (m, 2H), 5.21 (br. s., 1H), 5.33 - 5.44 (m, 1H), 7.23 - 7.29 (m, 1H), 7.31 - 7.37 (m, 3H), 7.42 (d, *J* = 8.2 Hz, 2H), 7.75 (d, *J* = 8.2 Hz, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 15.9 (CH_3), 16.3 (CH_3), 17.6 (CH_3), 21.6 (CH_3), 23.4 (CH_3), 23.4 (CH_3), 25.6 (CH_3), 26.1 (CH_2), 26.4 (CH_2), 26.6 (CH_2), 29.6 (CH_2), 32.1 (CH_2), 39.6 (CH_2), 39.7 (CH_2), 63.5 (CH), 66.3 (CH_2), 72.1 (CH_2), 117.0 (CH), 122.1 (CH), 123.3 (CH), 124.1 (CH), 127.4 (CH), 127.7 (CH), 127.7 (CH), 127.8 (CH), 128.3 (CH), 129.1 (CH), 129.3 (CH), 130.6 (CH), 131.3 (CH), 134.9, 135.5, 138.4, 139.9, 144.2, 145.1
HRMS (ESI)	calcd for $C_{39}H_{54}NaO_3S$ (M+Na) 625.3686, found 625.3680
23	
Procedure used	F
Yield	3.6 g (71%) from **22**
Lit	Ref[8]
	^{1}H, ^{13}C, HRMS
^{1}H NMR	(500 MHz, $CDCl_3$) δ 1.59, 1.60, 1.67, 1.68, 1.73 (6 × s, 18H), 1.93 - 2.11 (m, 16H),

	4.04 - 4.19 (m, 2H), 5.06 - 5.16 (m, 4H), 5.43 (t, J = 6.8 Hz, 1H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 15.9 (CH_3), 17.6 (CH_3), 23.3 (CH_3), 23.4 (CH_3), 25.6 (CH_3), 26.2 (CH_2), 26.5 (CH_2), 26.7 (CH_2), 31.8 (CH_2), 32.1 (CH_2), 39.6 (CH_2), 58.8 (CH_2), 123.9 (CH), 124.1 (CH), 124.3 (CH), 124.4 (CH), 124.5 (CH), 131.1, 134.8, 135.1, 136.0, 139.5
HRMS (ESI)	calcd for $C_{25}H_{42}NaO$ (M+Na) 381.3128, found 381.2975
24	
Procedure used	B2 followed by D
Yield	2.6 g (55%) from **23**
^{1}H NMR	(500 MHz, $CDCl_3$) δ 1.59, 1.65, 1.67, 1.72 (6 × s, 18H), 1.74 - 1.81 (m, 2H), 1.87 (q, J = 7.3 Hz, 3H), 1.93 - 2.09 (m, 14H), 2.43 (s, 3H), 3.77 (d, J = 7.8 Hz, 2H), 4.94 (t, J = 6.9 Hz, 1H), 5.09 (m, 3H), 5.19 (t, J = 7.6 Hz, 1H), 7.31 (m, J = 8.0 Hz, 2H), 7.73 (m, J = 8.2 Hz, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 15.9, 17.6, 21.6, 23.3, 23.5, 25.6, 25.7, 26.5, 26.5, 26.7, 31.8, 32.0, 39.7, 55.9, 111.0, 123.8, 123.9, 124.1, 124.3, 128.4, 129.5, 131.2, 134.9, 135.2, 135.9, 136.0, 144.4, 145.7
HRMS (ESI)	calcd for $C_{32}H_{48}NaO_2S$ (M+Na) 519.3267, found 519.3261
7a	
Procedure used	E
Yield	14 g (77%) from **19**
Lit	Ref[11]
	No spectroscopic characterization is given.
^{1}H NMR	(500 MHz, $CDCl_3$) δ 1.41 - 1.53 (m, 2H), 1.55, 1.56, 1.58. 1.59, 1.64, 1.67, 1.72 (8 × s, 24H), 1.72 - 1.83 (m, 3H), 1.77 - 1.81 (m, 1H), 1.87 - 2.07 (m, 18H), 2.38 - 2.48 (m, 10H), 2.61 - 2.71 (m, 1H), 2.74 (d, J = 13.6 Hz, 1H), 3.75 - 3.89 (m, 3H), 3.93 - 4.03 (m, 2H), 4.46 - 4.51 (m, 2H), 4.87 - 5.10 (m, 7H), 5.17 (d, J = 8.8 Hz, 1H), 5.41 (t, J = 6.8 Hz, 1H), 7.24 - 7.38 (m, 11H), 7.65 - 7.76 (m, 5H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 15.9, 15.9, 16.2, 16.2, 17.6, 21.6, 23.2, 23.2, 23.3, 23.4, 23.4, 23.5, 25.6, 25.6, 26.1, 26.1, 26.5, 26.6, 29.4, 29.5, 30.2, 31.7, 31.8, 32.1, 39.6, 39.7, 39.7, 56.0, 63.1, 63.3, 63.3, 66.3, 72.0, 117.0, 117.8, 117.8, 122.1, 123.3, 124.1, 124.1, 127.3, 127.4, 127.7, 128.0, 128.1, 128.3, 128.5, 129.1, 129.2, 129.3, 129.3, 129.3, 129.5, 130.4, 130.7, 130.8, 131.3, 134.7, 134.8, 135.5, 135.6, 138.4, 139.9, 144.3, 144.3, 144.4, 144.5, 145.0
HRMS (ESI)	calcd for $C_{56}H_{76}NaO_5S_2$ (M+Na) 915.5026, found 915.5019

7b	
Procedure used	E
Yield	17 g (83%) from **21**
^{1}H NMR/^{13}C NMR	Full assignment is given in Table S4.
HRMS (ESI)	calcd for $C_{56}H_{76}O_5S_2$ (M-H$^-$) 891.5050, found 891.5070
7c	
Procedure used	E
Yield	3.6 g (93%) from **24**
^{1}H NMR	(500 MHz, $CDCl_3$) δ 1.43 - 1.54 (m, 2H), 1.60, 1.64, 1.65, 1.68, 1.73 (8 × s, 24H), 1.74 - 1.82 (m, 2H), 1.87 - 2.12 (m, 16H), 2.41 (s, 3H), 2.46 (dd, *J* = 13.0, 11.6 Hz, 1H), 2.72 (dd, *J* = 13.2, 1.8 Hz, 1H), 3.85 (td, *J* = 10.9, 2.6 Hz, 1H), 3.99 (d, *J* = 6.8 Hz, 2H), 4.49 (s, 2H), 4.90 (t, *J* = 6.0 Hz, 1H), 4.97 (d, *J* = 10.6 Hz, 1H), 5.07 - 5.27 (m, 4H), 5.42 (t, *J* = 6.4 Hz, 1H), 7.24 - 7.41 (m, 7H), 7.71 (d, *J* = 8.2 Hz, 2H)
^{13}C NMR	(126 MHz, $CDCl_3$) δ 15.9, 17.5, 21.4, 23.2, 23.2, 23.3, 23.5, 25.6, 26.4, 26.4, 26.5, 26.6, 30.0, 31.7, 32.0, 32.1, 39.6, 39.6, 63.1, 66.2, 72.0, 117.5, 122.0, 123.8, 123.9, 124.0, 124.2, 127.3, 127.6, 127.9, 128.2, 129.0, 129.2, 130.4, 134.8, 135.1, 135.6, 138.4, 139.8, 144.1, 144.8
HRMS (ESI)	calcd for $C_{49}H_{70}NaO_3S$ (M+Na) 761.4938, found 761.4942
4	
Procedure used	F
Yield	2.0 g, 59% from **7a** (>90% purity) 2.8 g, 63% (as 1:3 mixture of **4**:**4B2**) from **7b** 0.2 g (**4**, isolated from **4**/**4B2** mixture) 1.6 g, 68% from **7c** (>91% purity) Compound **4** was isolated as colorless oil and was stored at 4 ^{o}C.
Lit	Ref[8] ^{1}H, ^{13}C, HRMS are given.
^{1}H NMR/^{13}C NMR	Full assignment is given in Table S2.
HRMS (ESI)	calcd for $C_{35}H_{58}NaO$ (M+Na$^+$) 517.4380, found 517.4348
4B2	
Procedure used	F
Yield	2.8 g, 63% (as 1:3 mixture of **4**:**4B2**) from **7b**

	0.8 g (**4B2**, isolated from **4**/**4B2** mixture)
^{1}H NMR/^{13}C NMR	Full assignment is given in Table S2.
HRMS (ESI)	calcd for $C_{35}H_{58}NaO$ ($M+Na^{+}$) 517.4380, found 517.4340

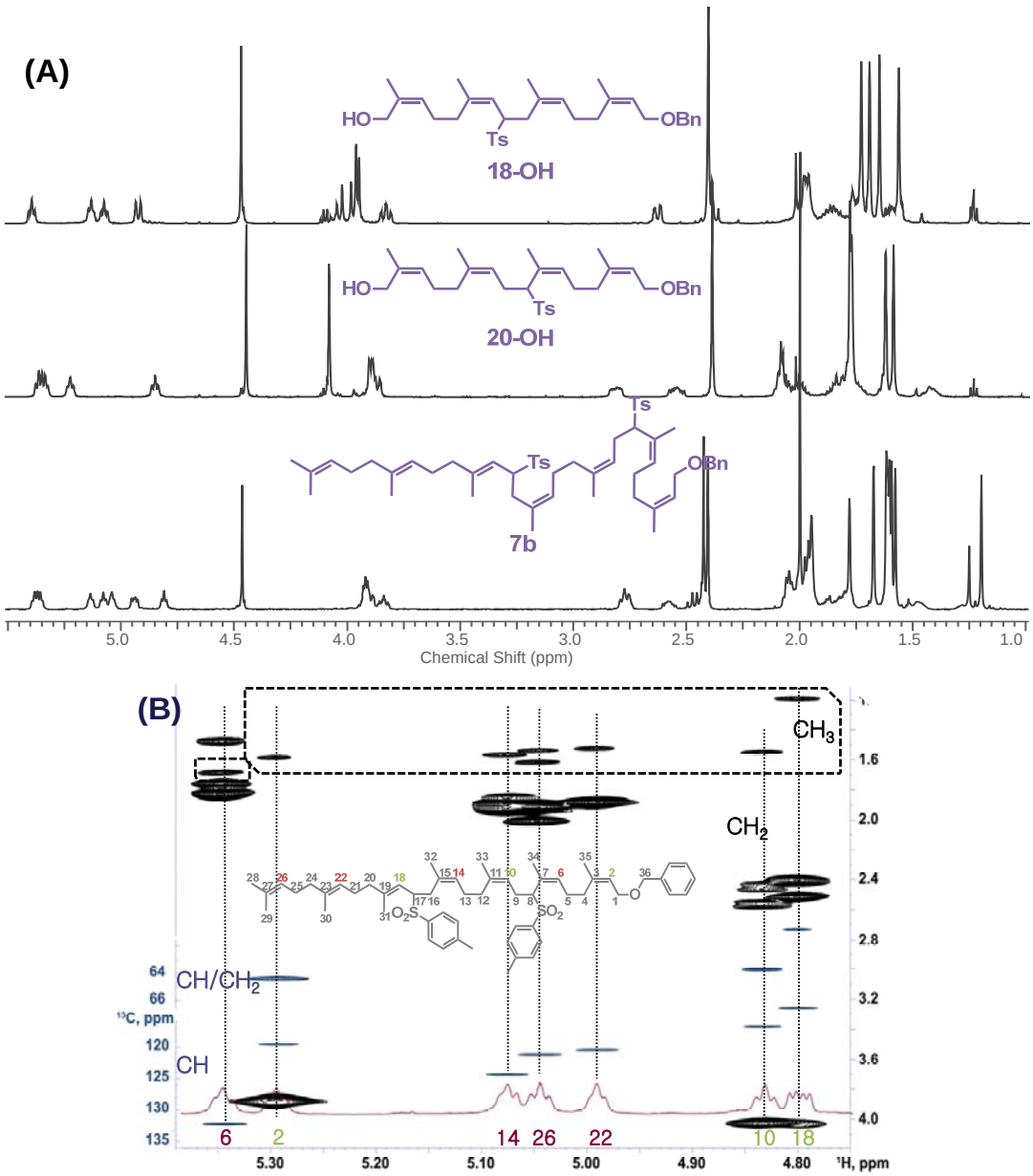
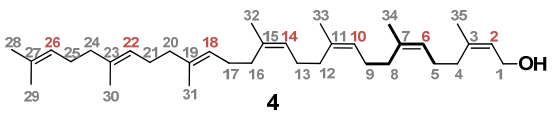


Figure S1. ^{1}H NMR spectra of intermediates (**18-OH, 20-OH** and **7b**) (A). ^{1}H-^{13}C-HSQC-TOCSY (crosspeaks in blue) and TOCSY (crosspeak in black) of **7b** (800.13 MHz).

Stereochemical verification within each route was by NMR. Notably, intermediates **18-OH, 20-OH** and **7b** had well-resolved ^{1}H resonances (Figure S1A, Tables 1 and 2). Figure S1B further shows the ^{1}H-^{13}C-HSQC-TOCSY spectrum of **7b** (crosspeaks in blue with respect to the y-axis on the left) and TOCSY spectrum (crosspeaks in black with respect to the y-axis on the right). The seven vinyl resonances (of H-2, -6, -10, -14, -18, -22, and -28) in **7b** were each resolved, and each resonance showed crosspeaks to the proximal CH/CH$_2$/CH$_3$ carbons. The ^{1}H and ^{13}C resonance assignments for three compounds are given in Tables S1 and S2.

Table S2. ^{1}H and ^{13}C NMR data[a] for **4** and **4B2** in $CDCl_3$ at 298 K (800.13 MHz, obtained using a solution of 10 mg of **4** (or **4B2**) and 3 mg of $Eu(hcf)_3$ in 600 µL of $CDCl_3$).



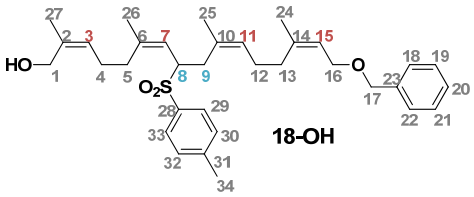
4

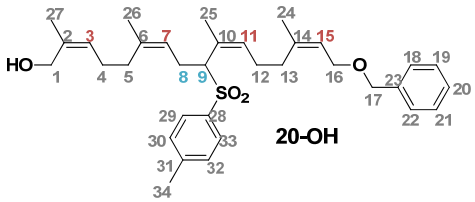
4B2

	4				**4B2**			
Carbon	**Type**	δ_C	δ_H	HMBC[b]	**Type**	δ_C	δ_H	HMBC[b]
1	**CH_2**	63.38	6.08		**CH_2**	65.60	6.95	
2	**CH**	125.24	6.62		**CH**	125.62	7.08	
3	**C**	140.68			**C**	141.76		
4	**CH_2**	32.81	2.70	2, 3, 5, 35	**CH_2**	32.69	2.79	2, 3, 5, 6, 35
5	**CH_2**	26.65	2.45	3, 4, 6, 7	**CH_2**	27.20	1.96	3, 4, 6, 7
6	**CH**	124.74	5.41	5, 8, 34	**CH_2**	40.00	2.29	4, 5, 7, 8, 34
7	**C**	136.20			**C**	135.03		
8	**CH_2**	32.30	2.11 2.18	9, 10	**CH**	124.04	5.27	6, 7, 34
9	**CH_2**	26.43	2.12 2.18	10, 11	**CH_2**	27.19	2.77	7, 8, 10
10	**CH**	124.96	5.23	8, 9, 33	**CH**	124.27	5.18	9, 12, 33
11	**C**	135.33			**C**	135.32		
12	**CH_2**	32.29	2.11 2.18	11, 14, 33	**CH_2**	32.53	2.10	10, 11, 33
13	**CH_2**	26.61	2.12 2.18	12, 14, 15	**CH_2**	26.62	2.10	12, 14, 15
14	**CH**	124.93	5.19	13, 32	**CH**	125.21	5.17	13, 16, 32
15	**C**	135.40			**C**	135.69		
16	**CH_2**	32.00	2.08 2.10	15, 17, 18, 32	**CH_2**	32.26	2.07	14, 17, 18
17	**CH_2**	26.61	2.09 2.11	16, 18, 19	**CH_2**	26.89	2.08	15, 16, 18, 19
18	**CH**	124.15	5.18	16, 17, 20, 31	**CH**	124.43	5.16	17, 20, 31
19	**C**	135.13			**C**	135.42		
20	**CH_2**	39.73	2.00	18, 19, 21	**CH_2**	40.01	2.00	19, 21, 31
21	**CH_2**	26.65	2.02 2.08	20, 22, 23	**CH_2**	26.90	2.07	20, 22, 23
22	**CH**	124.23	5.14	20, 21, 24, 30	**CH**	124.49	5.13	18, 21, 22, 24, 30
23	**C**	134.87			**C**	135.18		
24	**CH_2**	39.83	2.012	22, 23, 25, 26, 30	**CH_2**	39.97	1.99	22, 23, 25, 26, 30
25	**CH_2**	26.73	2.008 2.07	24, 26, 27	**CH_2**	27.03	2.07	23, 26, 27
26	**CH**	124.38	5.12	28, 29	**CH**	124.66	5.11	24, 25, 28, 29
27	**C**	131.20			**C**	131.49		
28	**CH_3**	25.69	1.70	26, 27, 29	**CH_3**	25.96	1.69	26, 27, 29
29	**CH_3**	17.66	1.62	26, 27, 28	**CH_3**	17.94	1.613	26, 27, 28
30	**CH_3**	15.98	1.62	22, 23, 24	**CH_3**	16.25	1.611	22, 23, 24
31	**CH_3**	15.98	1.64	18, 19, 20	**CH_3**	16.25	1.63	18, 19, 20
32	**CH_3**	23.47	1.73	14, 15, 16	**CH_3**	23.722	1.717	14, 15, 16
33	**CH_3**	23.47	1.75	10, 11, 12	**CH_3**	23.716	1.720	10, 11, 12
34	**CH_3**	23.44	1.79	6, 7, 8	**CH_3**	16.33	1.75	6, 7, 8
35	**CH_3**	23.85	2.04	2, 3, 4	**CH_3**	24.28	2.14	2, 3, 4

[a] Due to overlaps among signals, the values for the coupling constants or for ΣJ could not be always extracted. [b] HMBC correlations, optimized for 8 Hz, are from the stated ^{1}H resonance to the indicated carbon resonances.

Table S3. ^{1}H and ^{13}C NMR data for **18-OH** and **20-OH** in DMSO at 298 K (800.13 MHz).

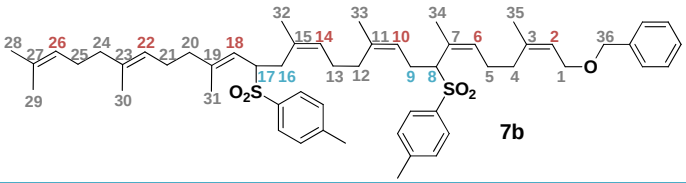




		18-OH				20-OH			
Carbon	**Type**	δ_C	δ_H	^{1}H multiplicity[a] $J(H_i,H_j)$ [Hz]	HMBC[b]	δ_C	δ_H	^{1}H multiplicity[a] $J(H_i,H_j)$ [Hz]	HMBC[b]
1	**CH_2**	59.78	3.83	s	2, 3, 27	59.86	3.91	bs	2, 3, 27
2	**C**	136.26				136.50			
3	**CH**	125.39	4.96	t, 6.7	1, 4, 5, 27	125.41	5.06	t, ΣJ = 14.6	1, 4, 5, 27
4	**CH_2**	25.51	1.69 1.78	mt mt	2, 3, 5, 6, 12	25.70	2.02	mt	2, 3, 5, 6
5	**CH_2**	32.29	1.52 1.77	mt mt	4, 6, 7, 26	32.29	1.90 1.95	mt mt	3, 4, 6, 7, 26
6	**C**	144.88				138.02			
7	**CH**	117.71	4.86	d, 10.4	5, 8, 9, 26	119.91	4.84	t, ΣJ = 13.5	5, 9, 26
8	**CH**	62.58	3.97	ddd, 10.9, 10.9, 3.0	6, 7, 9, 10	23.42 (CH_2)	2.47 2.61	mt, ΣJ = 38.0 mt, ΣJ = 26.0	6, 7, 9, 10
9	**CH_2**	30.31	2.34 2.49	dd, 13.0, 11.3 dd, 13.0, 2.0	7, 8, 10, 11, 25	65.61 (CH)	4.02	dd, 11.4, 3.3	7, 8, 10, 11, 25
10	**C**	130.79				126.15			
11	**CH**	128.05	5.13	t, 6.4	9, 12, 13, 25	135.60	5.35	t, ΣJ = 13.4	9, 10, 12, 13, 25
12	**CH_2**	26.43	1.93	mt	10, 11, 13, 14	25.88	1.48 1.83	mt mt	10, 11, 13, 14
13	**CH_2**	32.07	1.93 1.97	mt mt	12, 14, 15, 24	31.38	1.76 1.82	mt mt	11, 12, 14, 15, 24
14	**C**	139.23				138.85			
15	**CH**	122.81	5.33	t, 6.7	13, 14, 16, 24	122.99	5.30	dd, 6.2, 7.1	13, 14, 16, 24
16	**CH_2**	66.24	3.93	d, 6.7	14, 15, 17	66.24	3.87 3.89	dd, 6.2, 12.0 dd, 7.1, 12.0	14, 15, 17
17	**CH_2**	71.52	4.41	s	16, 18, 22, 23	71.52	4.39	s	16, 18, 22, 23
18, 22	**CH**	127.82	7.29	d, 7.1	17, 19, 20, 21	127.69	7.27	d, 7.3	17, 20
19, 21	**CH**	128.59	7.32	t, 7.1	18, 20, 22, 23	128.56	7.31	t, 7.3	23
20	**CH**	127.75	7.26	t, 7.1	18, 19, 21, 22	127.72	7.25	t, 7.3	18, 22
23	**C**	139.04				139.04			
24	**CH_3**	23.50	1.67	s	13, 14, 15	23.42	1.58	bs	13, 14, 15
25	**CH_3**	23.82	1.56	s	9, 10, 11	19.24	1.70	bs	9, 10, 11
26	**CH_3**	23.45	1.59	d	5, 6, 7	23.47	1.58	bs	5, 6, 7
27	**CH_3**	21.59	1.64	s	1, 2, 3	21.66	1.69	bs	1, 2, 3
28	**C**	135.09				135.61			
29, 33	**CH**	129.22	7.70	d, 8.2	30, 31, 32	128.85	7.70	d, 8.2	31
30, 32	**CH**	129.95	7.41	d, 8.2	28, 29, 33, 34	130.06	7.39	d, 8.2	28, 34
31	**C**	144.69				144.69			
34	**CH_3**	21.51	2.38	s	30, 31, 32	21.44	2.37	s	30, 31, 32

[a] Due to overlaps among signals, the values for the coupling constants or for ΣJ could not be always extracted. [b] HMBC correlations, optimized for 8 Hz, are from the stated ^{1}H resonance to the indicated carbon.

Table S4. ^{1}H and ^{13}C NMR data for **7b** in DMSO at 298 K (800.13 MHz).



7b

Carbon	Type	δ_C	δ_H	^{1}H multiplicity[a] $J(H_i,H_i)$, [Hz]	HMBC[b]
1	**CH_2**	65.75	3.90 3.87	dd, 11.8, 6.8 dd, 11.8, 6.8	2, 3, 36
2	**CH**	122.53	5.30	t, 6.8	1, 3, 4, 35
3	**C**	138.48			
4	**CH_2**	30.94	1.76 1.82	mt mt	2, 3, 5, 6, 35
5	**CH_2**	25.43	1.48 1.83	mt mt	3, 4, 6, 7
6	**CH**	135.14	5.35	t, 6.3	4, 5, 8, 34
7	**C**	125.69			
8	**CH**	65.04	4.02	mt	6, 7, 9, 10, 34
9	**CH_2**	22.97	2.45 2.56	mt mt	7, 8, 10, 11
10	**CH**	119.66	4.83	t, 6.9	8, 11, 13, 33
11	**C**	137.20			
12	**CH_2**	31.49	1.85 1.93	mt mt	10, 11, 13, 14, 33
13	**CH_2**	25.77	1.90 1.93	mt mt	11, 12, 14, 15
14	**CH**	127.32	5.08	t, 6.8	12, 13, 16, 32
15	**C**	130.49			
16	**CH_2**	29.20	2.41 2.52	mt mt	15, 17, 18, 32
17	**CH**	62.10	4.03	mt	16, 18, 19
18	**CH**	116.73	4.80	ddd, 10.3, 5.1, 0.9	16, 17, 20, 31
19	**C**	144.57			
20	**CH_2**	39.09	1.88	mt	18, 19, 21, 22, 31
21	**CH_2**	25.60	1.89	mt	19, 20, 22, 23
22	**CH**	123.36	4.99	t, 5.5	20, 21, 24, 30
23	**C**	134.68			
24	**CH_2**	39.17	1.91	mt	22, 23, 25, 26, 30
25	**CH_2**	26.19	2.01	mt	23, 24, 26, 27
26	**CH**	124.07	5.05	t, 7.0	24, 25, 28, 29
27	**C**	130.68			
28	**CH_3**	25.46	1.62	t, 1.0	26, 27, 29
29	**CH_3**	17.47	1.54	bs	26, 27, 28
30	**CH_3**	15.69	1.52	s	22, 23, 24
31	**CH_3**	16.08	1.19	d, 0.9	18, 19, 20
32	**CH_3**	23.12	1.57	bs	15, 16, 17
33	**CH_3**	23.00	1.55	d, 1.0	10, 11, 12
34	**CH_3**	18.79	1.68	d, 5.1	6, 7, 8
35	**CH_3**	22.90	1.59	dd, 3.9, 1.1	2, 3, 4
36	**CH_2**	71.07	4.39	s	1, 37, 38, 42
37	**C**	138.59			
38, 42	**CH**	127.33	7.28	bd, 7.3	36, 40
39, 41	**CH**	128.18	7.32	t, 7.3	37
40	**CH**	127.30	7.26	tt, 7.3, 1.3	38, 42
43	**C**	135.10			
44	**CH**	128.40	7.696	d, 8.2	
45, 47	**CH**	129.63	7.398	dq, 8.2, 0.6	
46	**C**	144.32			
48	**CH**	128.40	7.698	d, 8.2	
49	**CH_3**	21.04	2.37	bs	
50	**C**	134.68			
51, 55	**CH**	128.77	7.71	d, 8.2	
52, 54	**CH**	129.43	7.41	dq, 8.2, 0.6	
53	**C**	144.21			
56	**CH_3**	21.06	2.39	bs	

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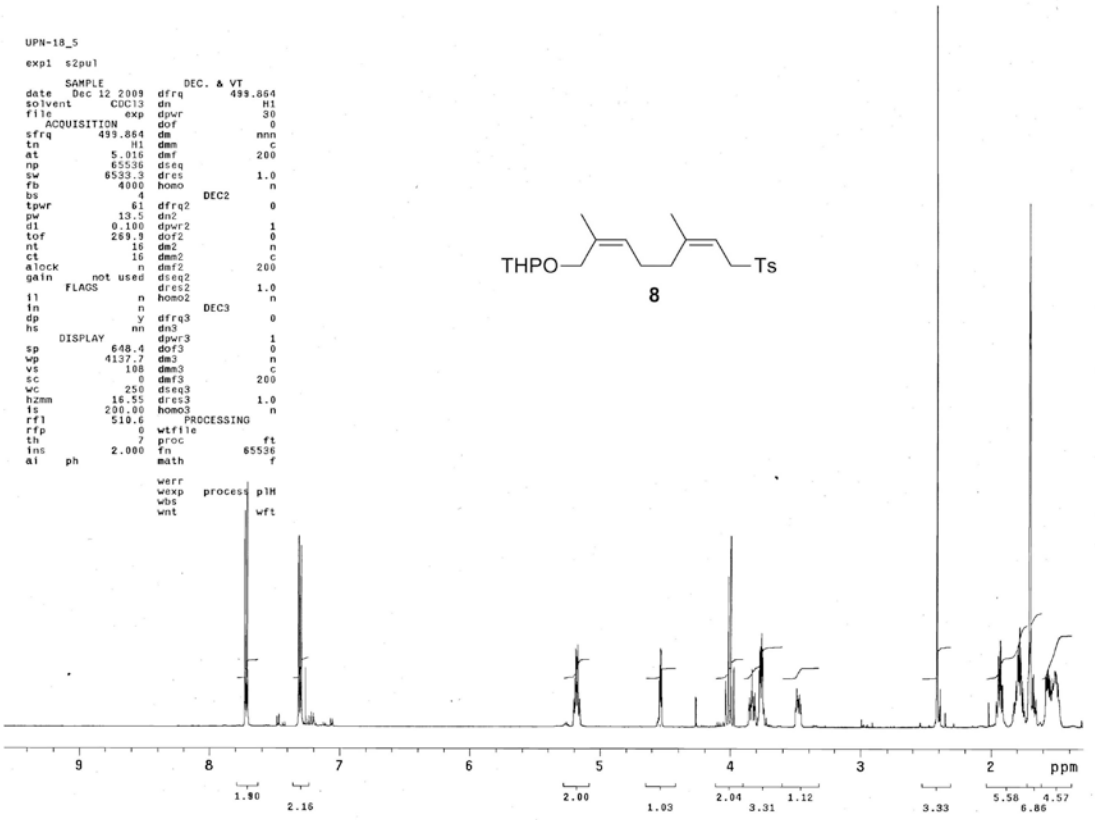
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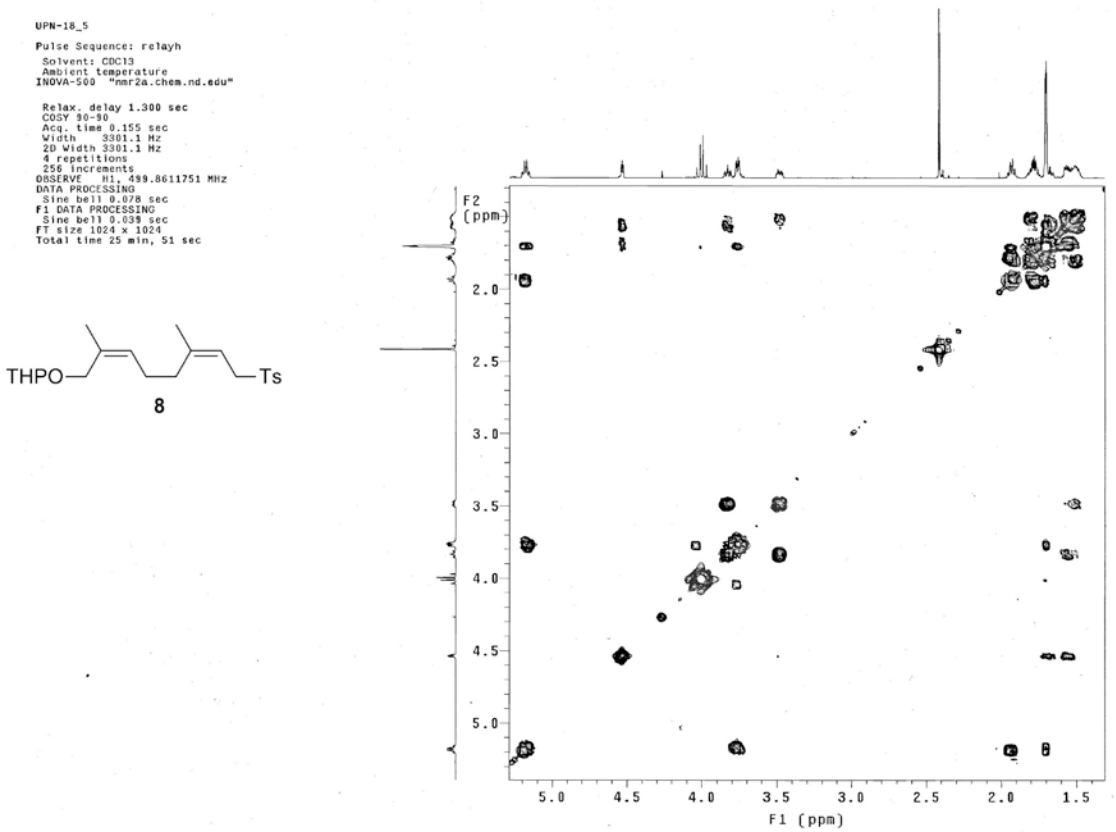
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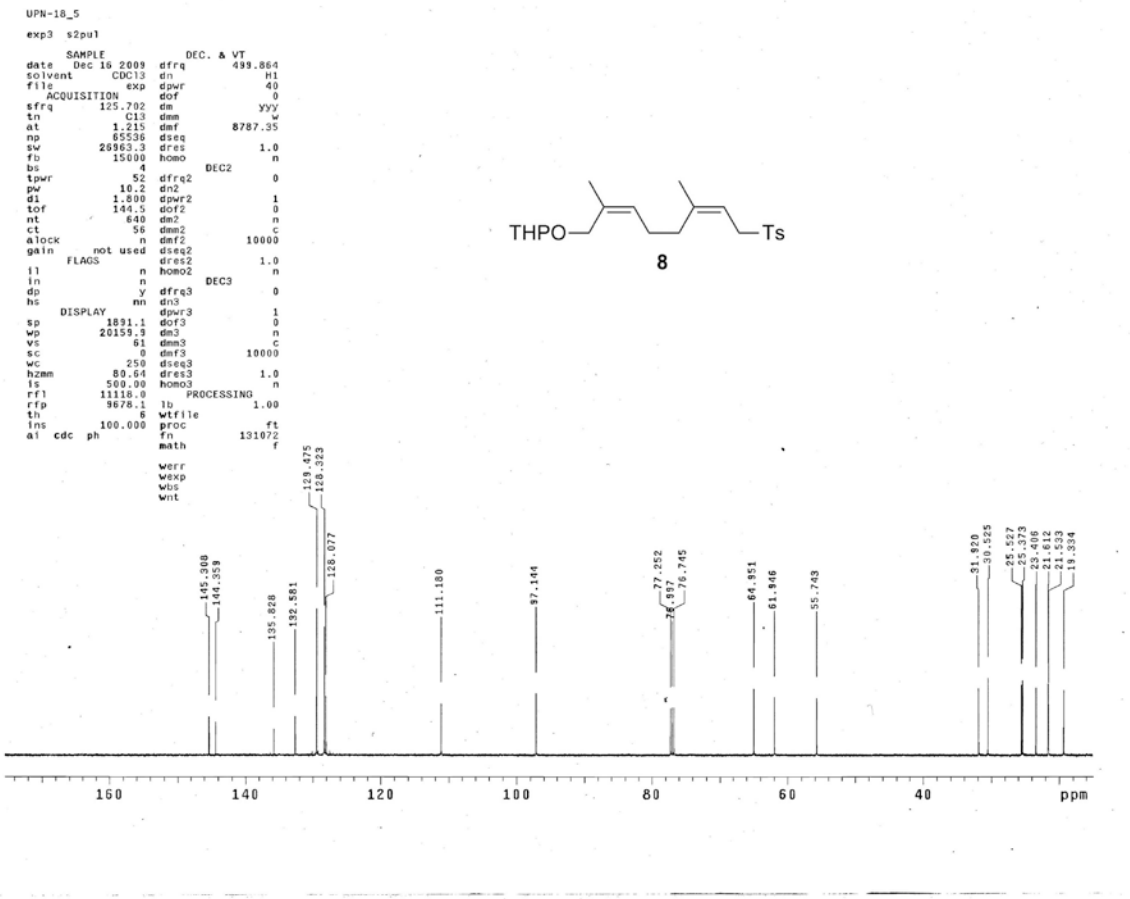
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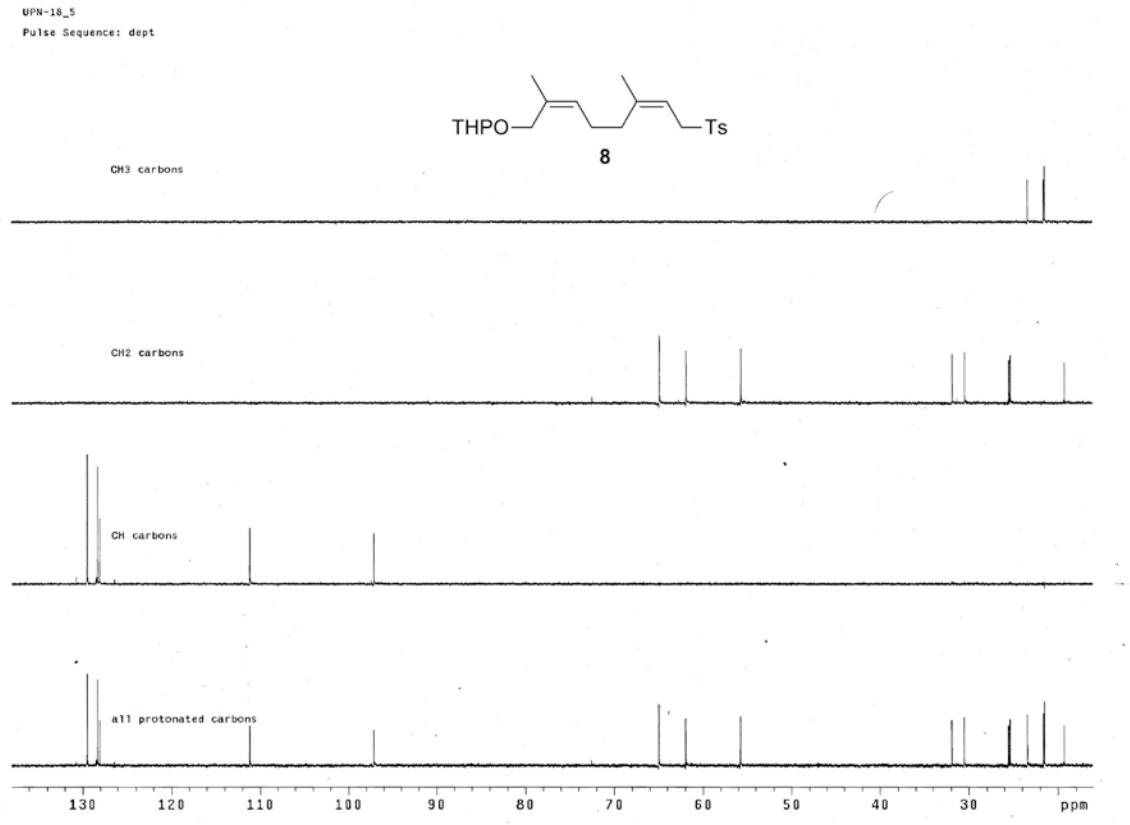
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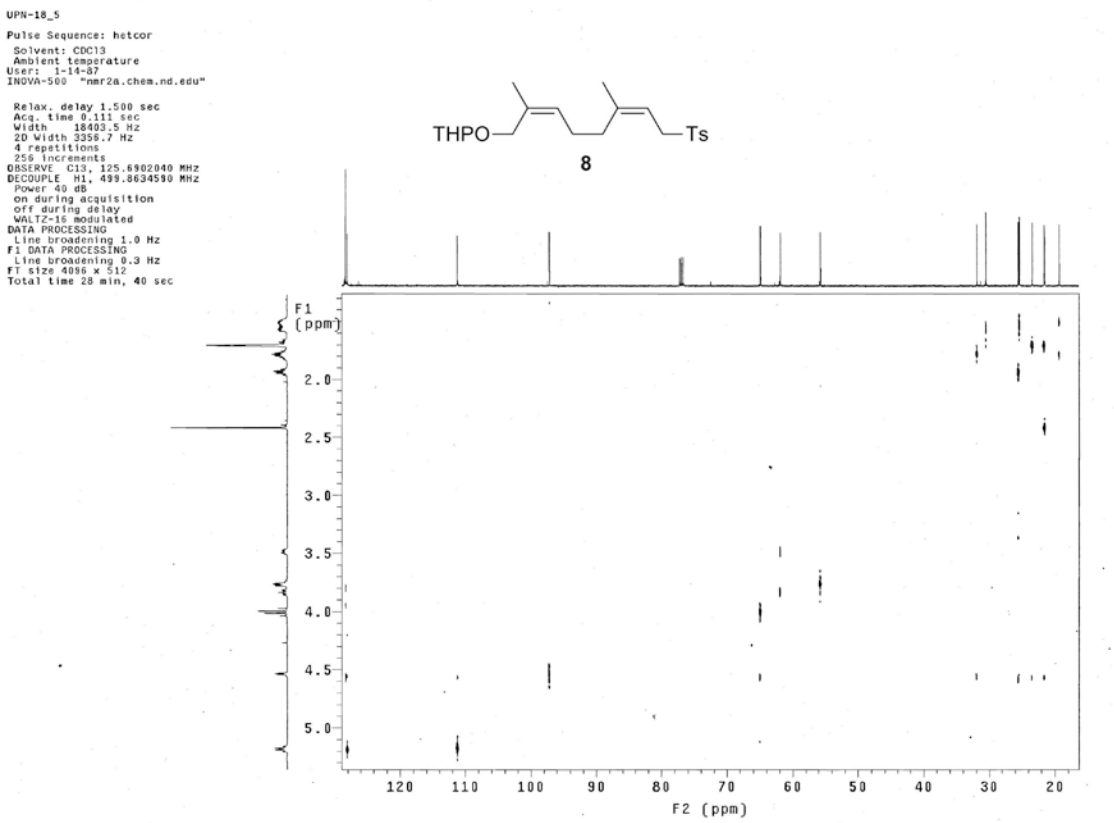
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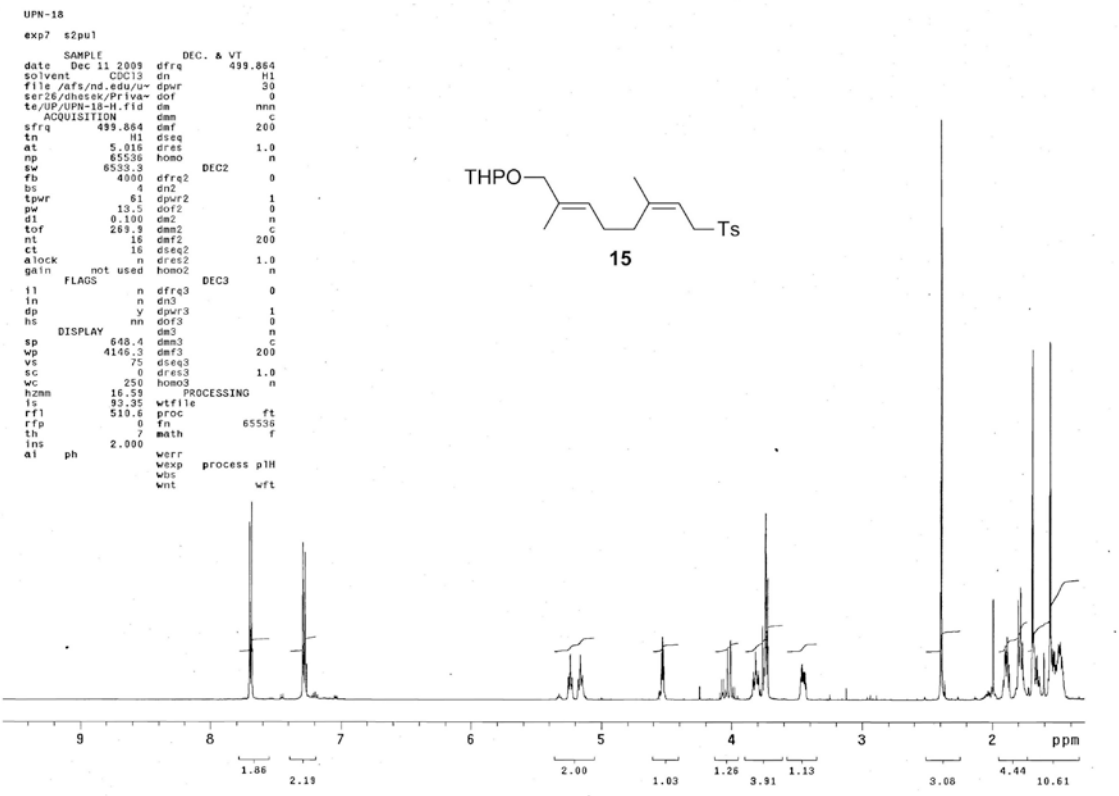
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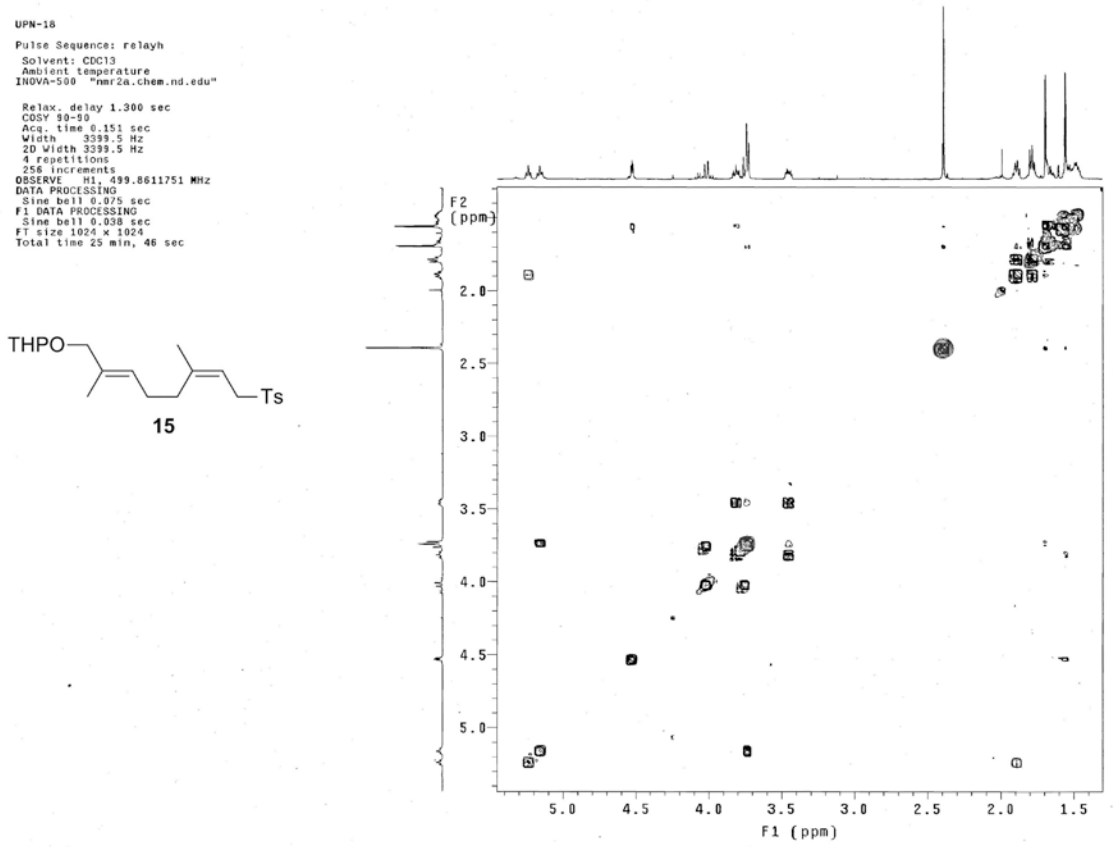
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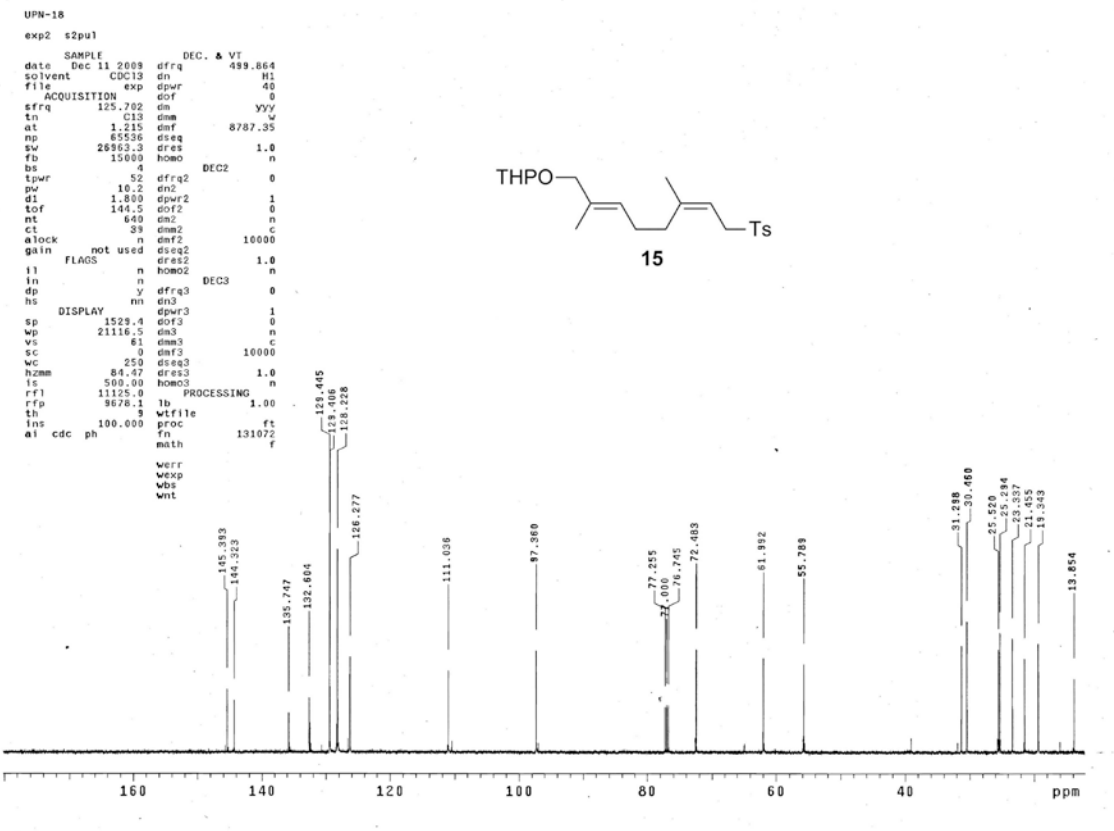
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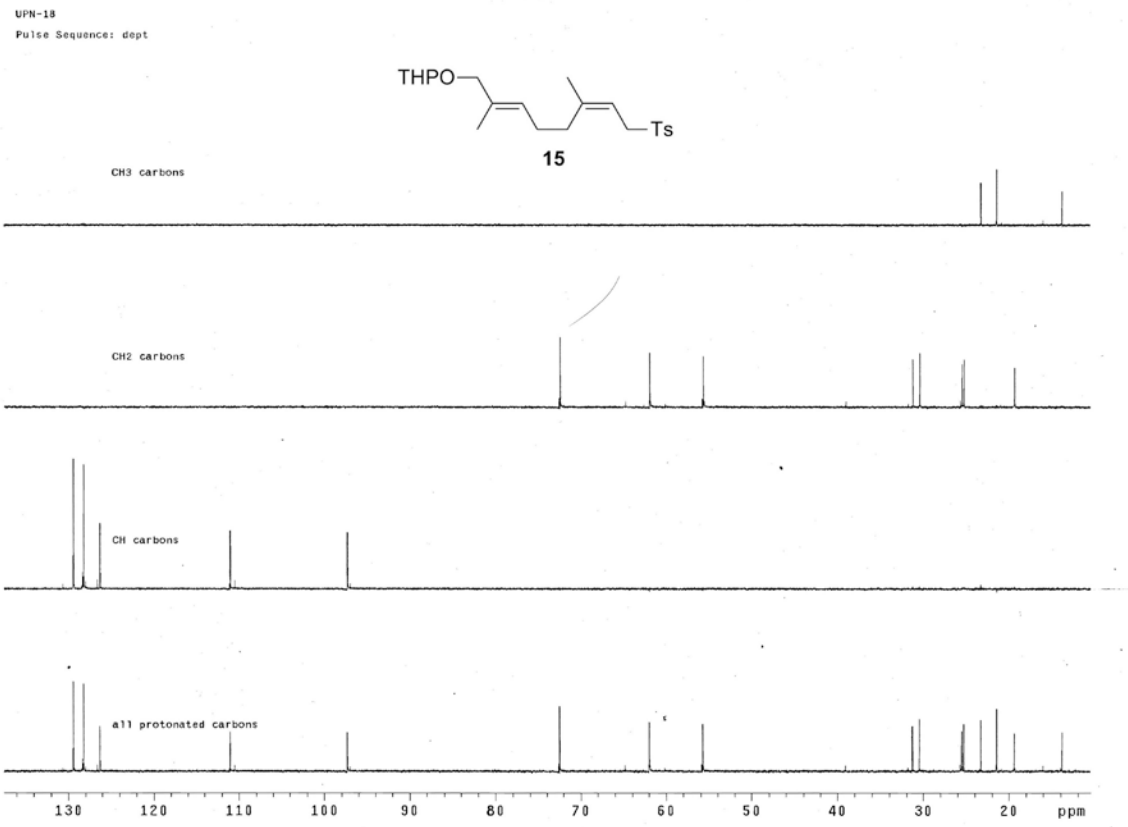
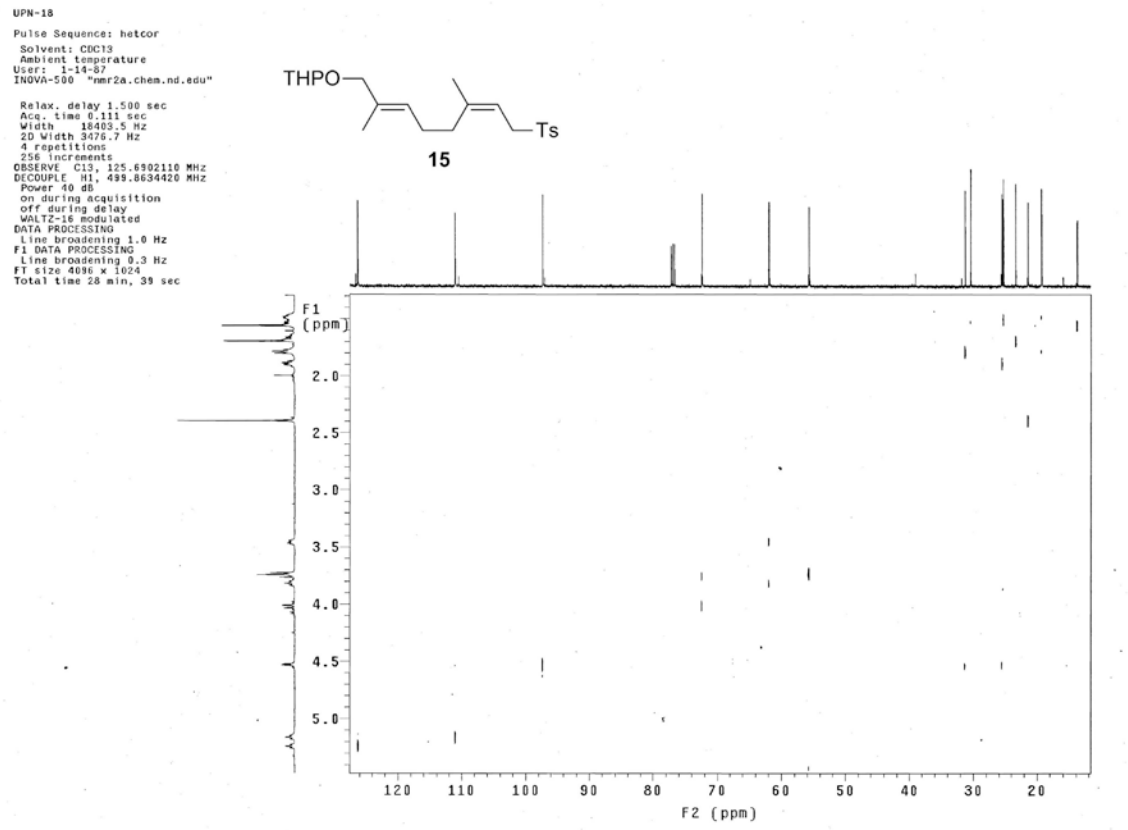
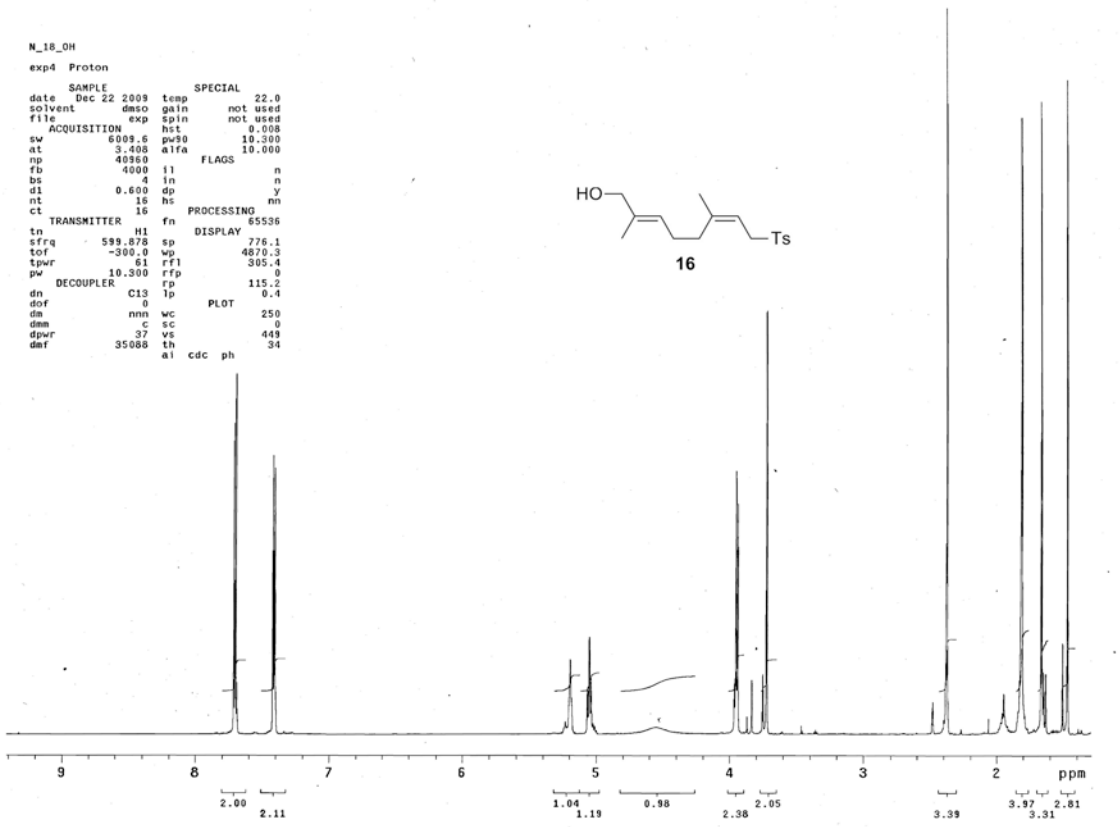
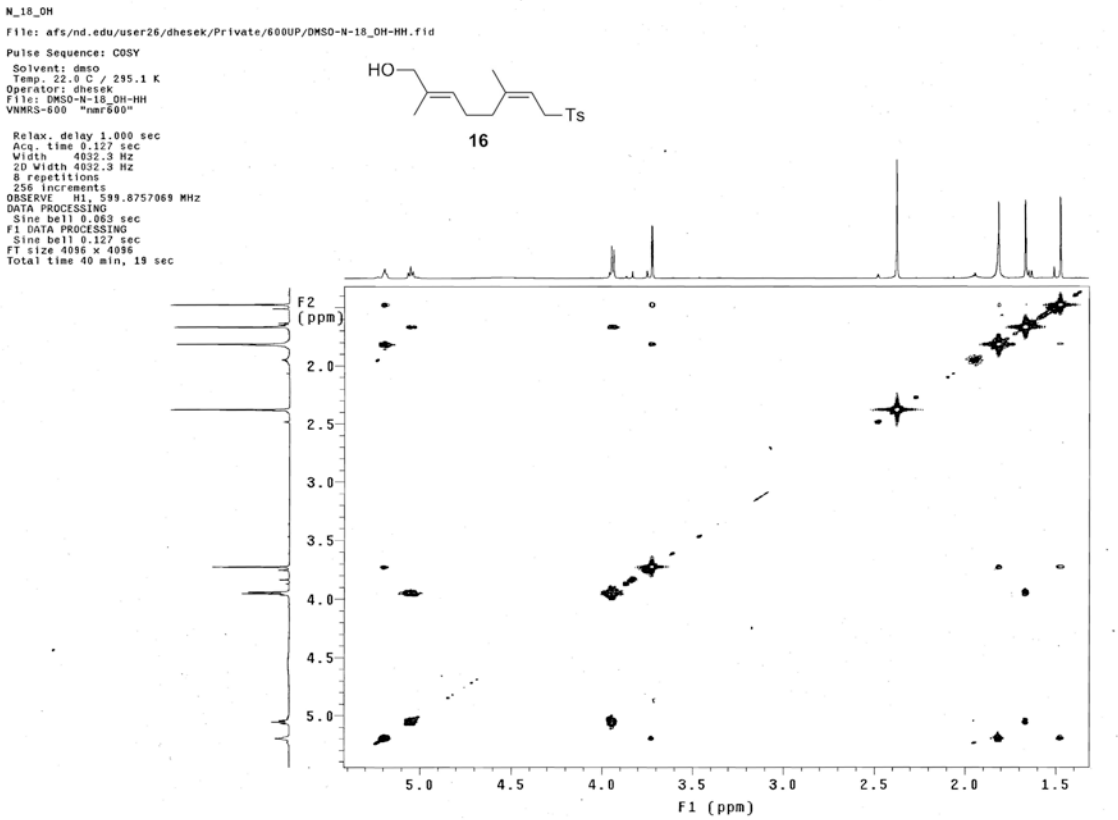
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
THPO
Ts
15
145.383
144.323
135.747
132.604
129.445
129.406
128.228
126.277
111.036
97.360
77.255
77.000
76.745
72.483
61.992
55.789
31.288
30.460
25.520
25.264
23.337
21.455
19.343
13.854
ppm


UPN-18
Pulse Sequence: dept
THPO
Ts
15
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
130
120
110
100
90
80
70
60
50
40
30
20
ppm


UPN-18
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 16403.5 Hz
2D Width 3476.7 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6902110 MHz
DECOUPLE H1, 499.8634420 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 28 min, 39 sec
THPO
Ts
15
F1 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
120
110
100
90
80
70
60
50
40
30
20
F2 (ppm)


N_18_OH
exp4 Proton
SAMPLE
date Dec 22 2009
solvent dmso
file exp
ACQUISITION
sw 6009.6
at 3.408
np 40960
fb 4000
bs 4
d1 0.600
nt 16
ct 16
TRANSMITTER
tn H1
sfrq 599.878
tof -300.0
tpwr 61
pw 10.300
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
SPECIAL
temp 22.0
gain not used
spin not used
hst 0.008
pw90 10.300
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp 776.1
wp 4870.3
rfl 305.4
rfp 0
rp 115.2
lp 0.4
PLOT
wc 250
sc 0
vs 449
th 34
ai cdc ph
HO
Ts
16
9
8
7
6
5
4
3
2
ppm
2.00
2.11
1.04
1.19
0.98
2.38
2.05
3.39
3.97
3.31
2.81

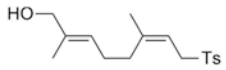

N_18_OH
File: afs/nd.edu/user26/dhesek/Private/600UP/DMSO-N-18_OH-HH.fid
Pulse Sequence: COSY
Solvent: dmso
Temp. 22.0 C / 295.1 K
Operator: dhesek
File: DMSO-N-18_OH-HH
VNMRS-600 "nmr600"
Relax. delay 1.000 sec
Acq. time 0.127 sec
Width 4032.3 Hz
2D Width 4032.3 Hz
8 repetitions
256 increments
OBSERVE H1, 599.8757069 MHz
DATA PROCESSING
Sine bell 0.063 sec
F1 DATA PROCESSING
Sine bell 0.127 sec
FT size 4096 x 4096
Total time 40 min, 19 sec
HO
Ts
16
F2 (ppm)
F1 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
1.5

N_18_OH

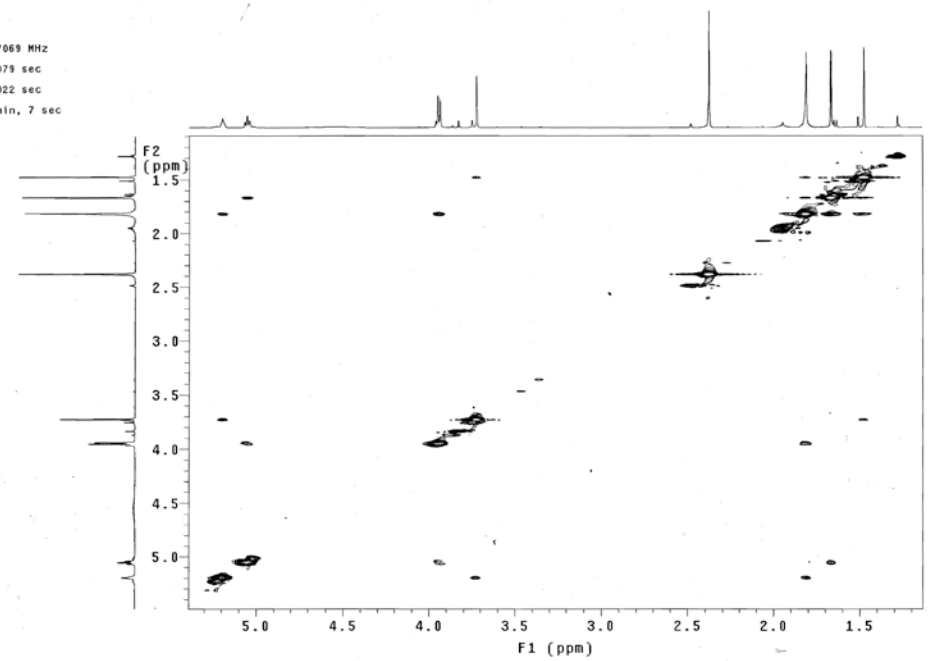
File: xp

Pulse Sequence: ROESY
Solvent: dmso
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"

Relax. delay 3.830 sec
Mixing 0.500 sec
Acq. time 0.170 sec
Width 6009.6 Hz
2D Width 6009.6 Hz
40 repetitions
2 x 256 increments
OBSERVE H1, 599.8757069 MHz
DATA PROCESSING
Gauss apodization 0.079 sec
F1 DATA PROCESSING
Gauss apodization 0.022 sec
FT size 2048 x 2048
Total time 25 hr, 50 min, 7 sec



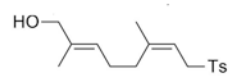
16



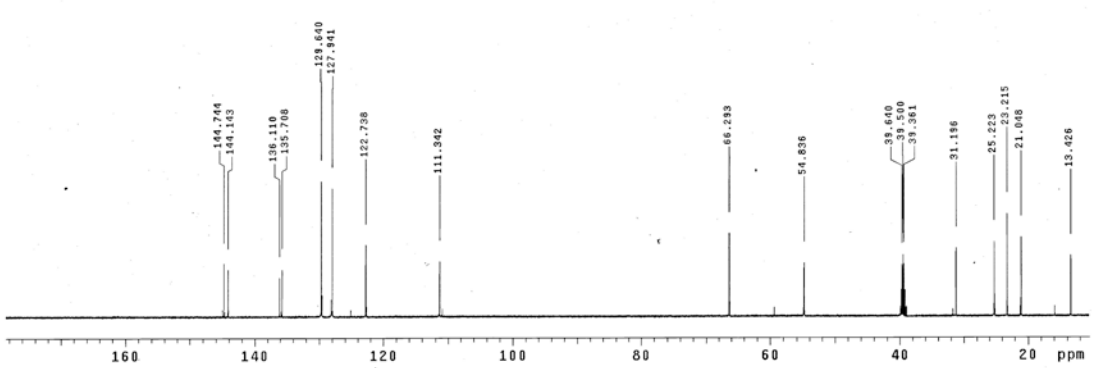
N_18_OH

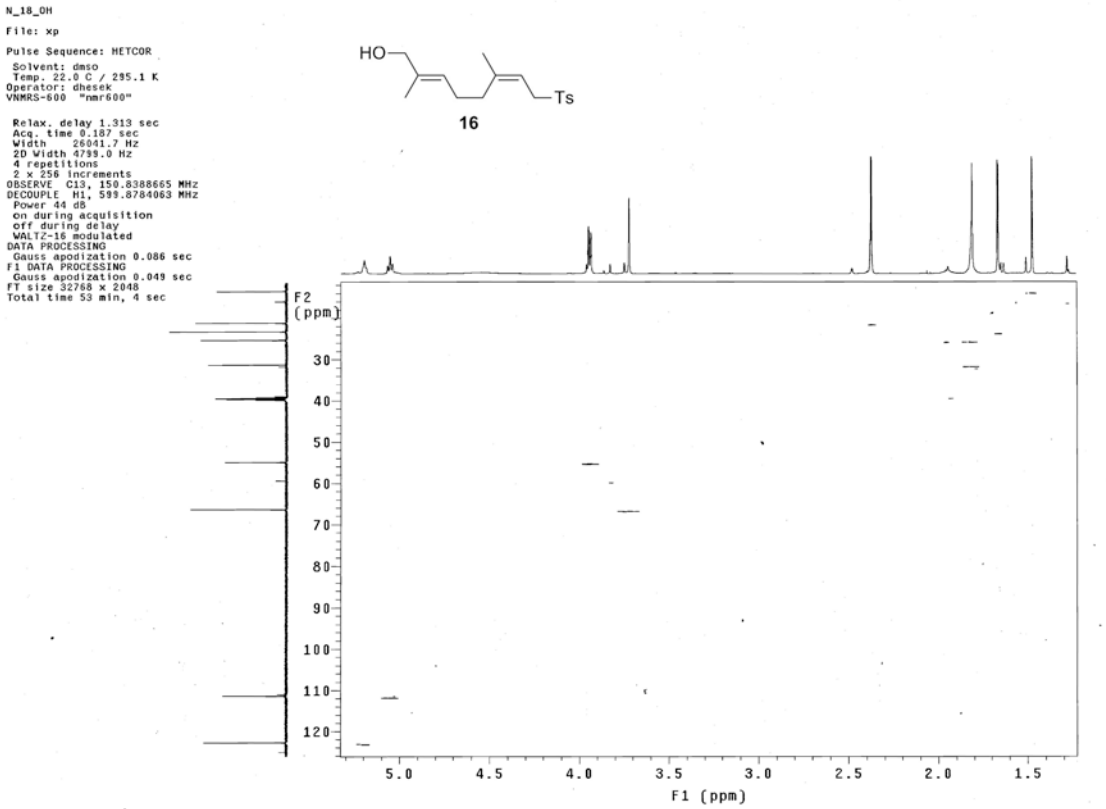
exp8 Carbon

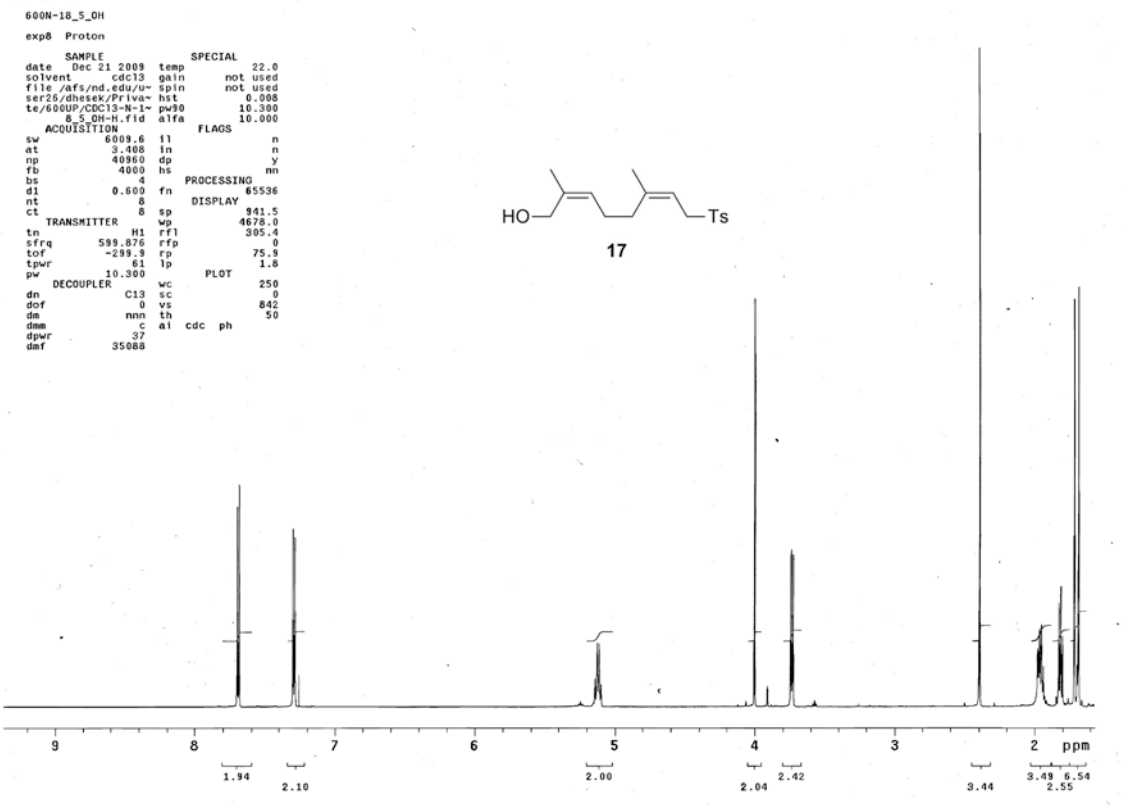
SAMPLE		SPECIAL	
date	Dec 23 2009	temp	22.0
solvent	dmso	gain	not used
file	exp	spin	not used
ACQUISITION		hst	0.008
sw	36764.7	pw90	7.400
at	1.783	alfa	10.000
np	131072	FLAGS	
fb	17000	il	n
bs	4	in	n
d1	1.220	dp	y
nt	256	hs	nn
ct	60	PROCESSING	
TRANSMITTER		lb	0.50
tn	C13	fn	262144
sfrq	150.855	DISPLAY	
tof	1542.6	sp	1587.0
tpwr	58	wp	25338.6
pw	7.400	rf1	8571.2
DECOUPLER		rfp	5958.1
dn	H1	rp	-114.2
dof	0	lp	7.9
dm	yyy	PLOT	
dmm	w	wc	250
dpwr	44	sc	0
dmf	15198	vs	76
		th	8
		ai cdc ph	



16




N_18_OH
File: xp
Pulse Sequence: HETCOR
Solvent: dmso
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"
Relax. delay 1.313 sec
Acq. time 0.187 sec
Width 26041.7 Hz
2D Width 4799.0 Hz
4 repetitions
2 x 256 increments
OBSERVE C13, 150.8388665 MHz
DECOUPLE H1, 599.8784063 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Gauss apodization 0.086 sec
F1 DATA PROCESSING
Gauss apodization 0.049 sec
FT size 32768 x 2048
Total time 53 min, 4 sec
HO
Ts
16
F2 (ppm)
F1 (ppm)


600N-18_5_OH
exp8 Proton
SAMPLE
date Dec 21 2009
solvent cdcl3
file /afs/nd.edu/u~
ser26/dhesek/Priva~
te/600UP/CDCl3-N-1~
8_5_OH-H.fid
ACQUISITION
sw 6009.6
at 3.408
np 40960
fb 4000
bs 4
d1 0.600
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 599.876
tof -299.9
tpwr 61
pw 10.300
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
SPECIAL
temp 22.0
gain not used
spin not used
hst 0.008
pw90 10.300
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
fn 65536
DISPLAY
sp 941.5
wp 4678.0
rfl 305.4
rfp 0
rp 75.9
lp 1.8
PLOT
wc 250
sc 0
vs 842
th 50
ai cdc ph
HO
Ts
17
ppm
1.94
2.10
2.00
2.04
2.42
3.44
3.49
6.54
2.55

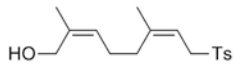
600N-18_5_OH

File: afs/nd.edu/user26/dhesek/Private/600UP/N-18_5_OH-HH.fid

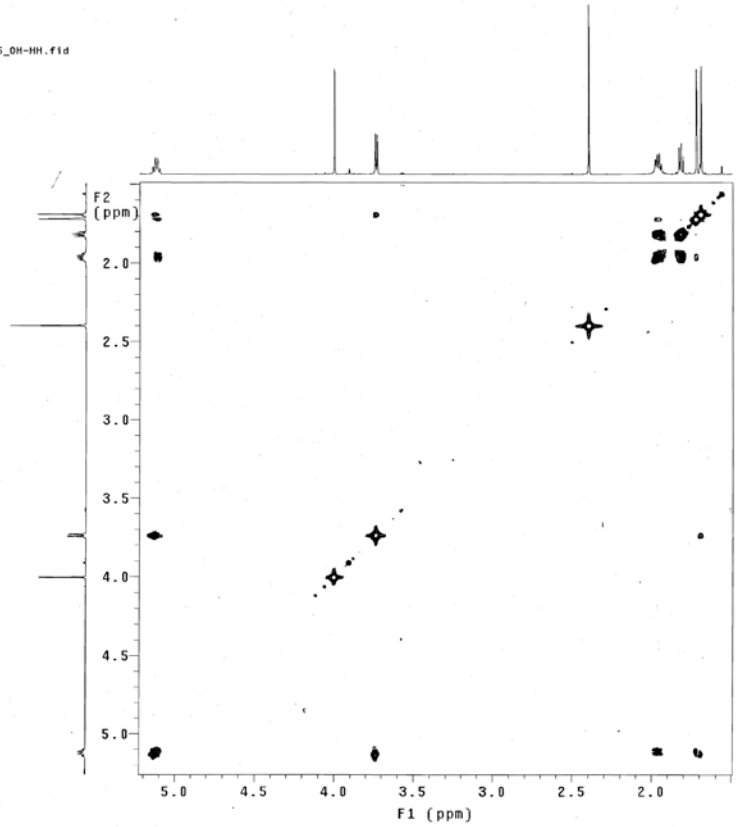
Pulse Sequence: COSY

Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: dhesek
File: N-18_5_OH-HH
VNMRS-600 "nmr600"

Relax. delay 1.000 sec
Acq. time 0.242 sec
Width 4223.0 Hz
2D Width 4223.0 Hz
8 repetitions
512 increments
OBSERVE H1, 599.8728575 MHz
DATA PROCESSING
Sine bell 0.121 sec
F1 DATA PROCESSING
Sine bell 0.078 sec
FT size 4096 x 4096
Total time 1 hr, 30 min, 14 sec



17



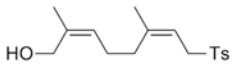
600N-18_5_OH

File: xp

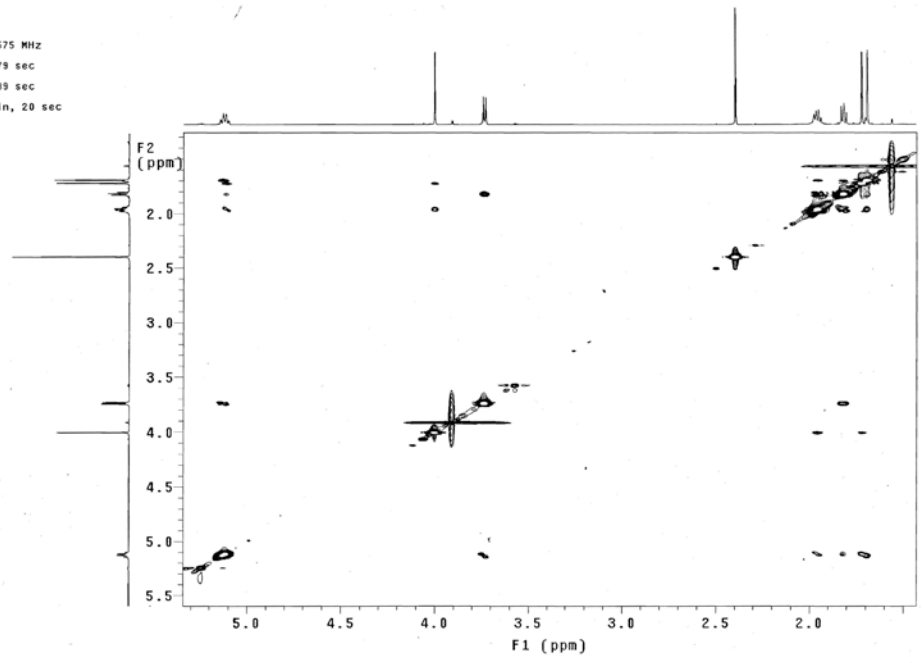
Pulse Sequence: ROESY

Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"

Relax. delay 3.830 sec
Mixing 0.500 sec
Acq. time 0.170 sec
Width 6009.6 Hz
2D Width 6009.6 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 599.8728575 MHz
DATA PROCESSING
Gauss apodization 0.079 sec
F1 DATA PROCESSING
Gauss apodization 0.039 sec
FT size 2048 x 2048
Total time 20 hr, 40 min, 20 sec



17



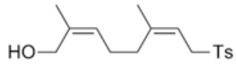
600N-18_5_OH

File: xp

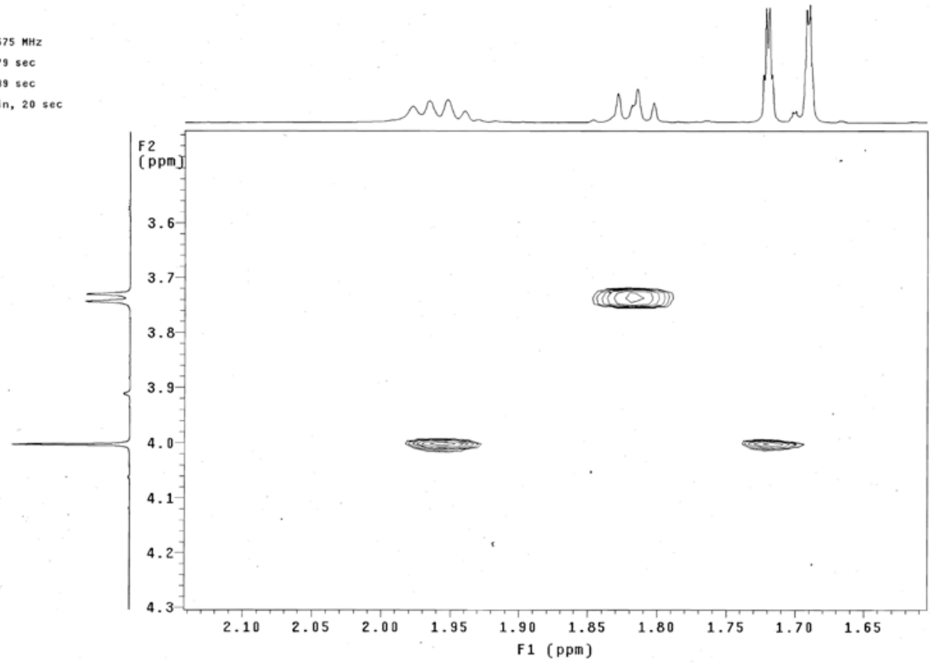
Pulse Sequence: ROESY

Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"

Relax. delay 3.830 sec
Mixing 0.500 sec
Acq. time 0.170 sec
Width 6009.6 Hz
2D Width 6009.6 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 599.8728575 MHz
DATA PROCESSING
Gauss apodization 0.079 sec
F1 DATA PROCESSING
Gauss apodization 0.039 sec
FT size 2048 x 2048
Total time 20 hr, 40 min, 20 sec



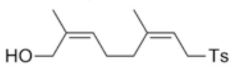
17



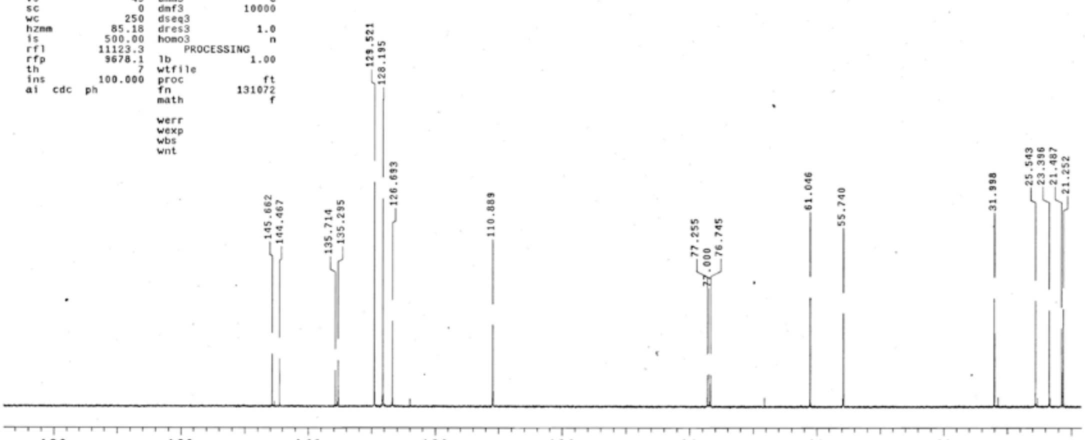
UPN-18_5_OH

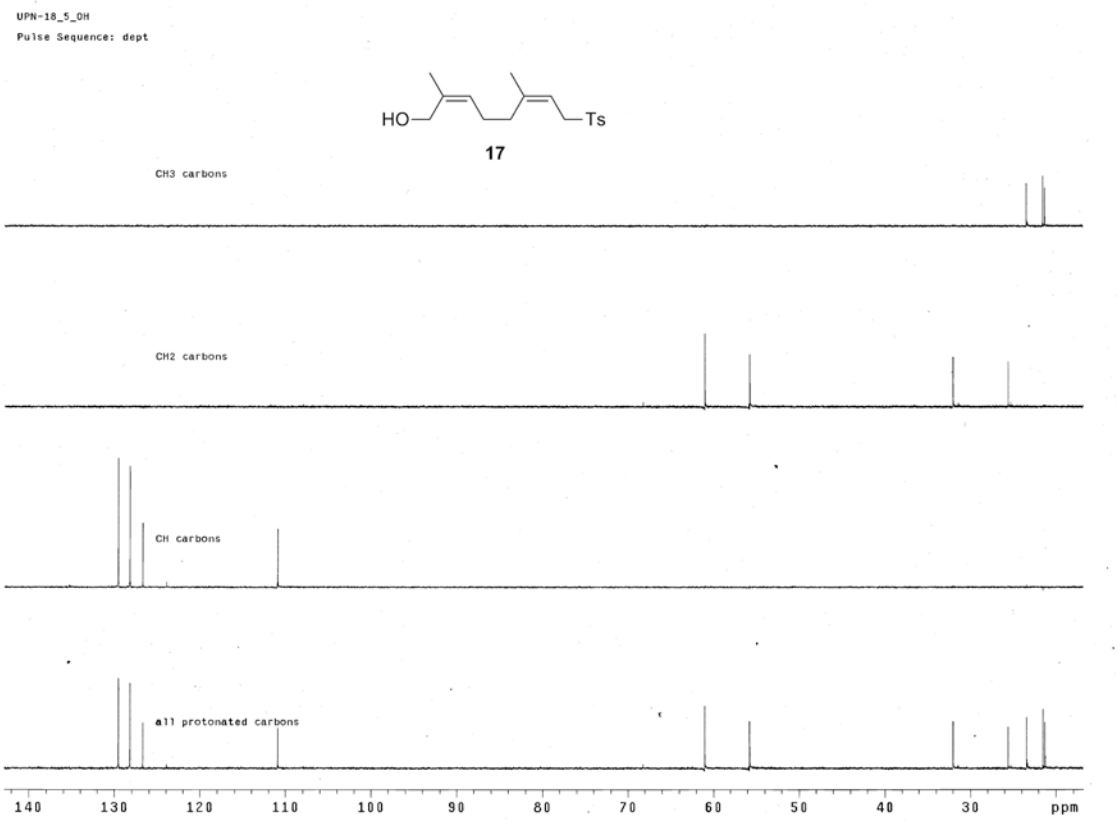
exp2 s2pul

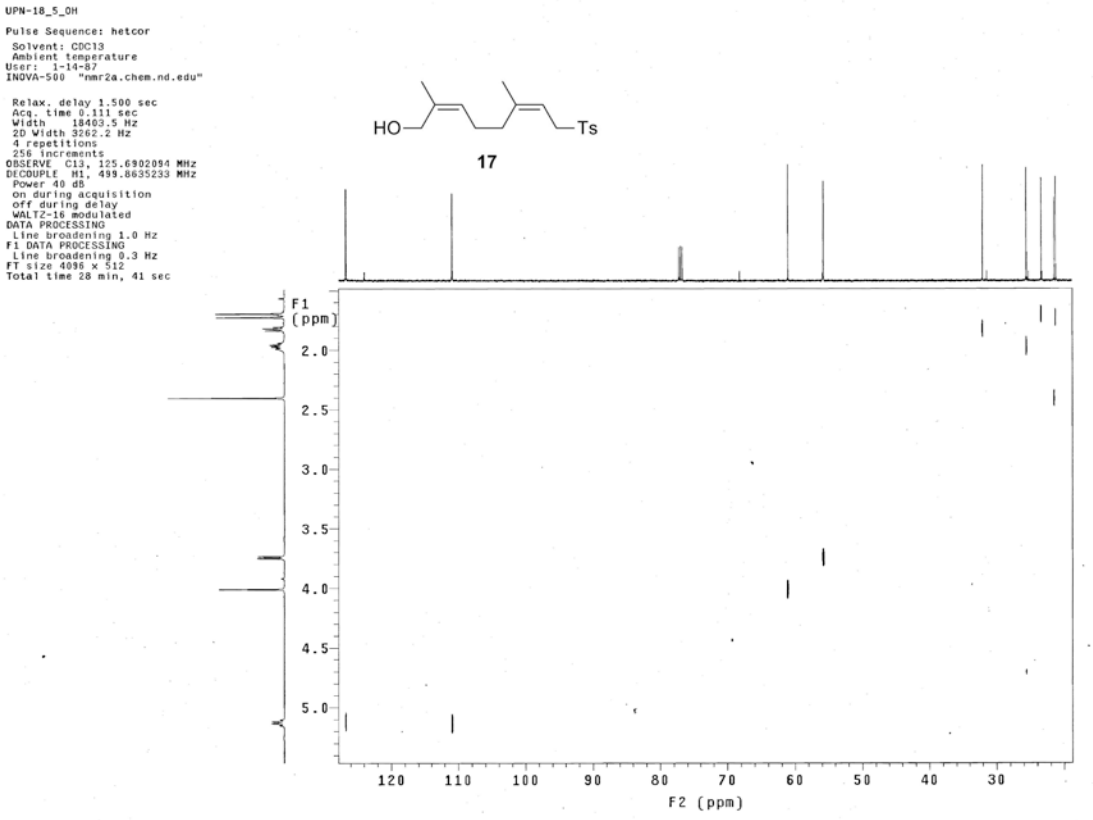
SAMPLE		DEC. & VT	
date	Dec 13 2009	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	640	dm2	n
ct	75	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	2310.7	dof3	0
wp	21293.8	dm3	n
vs	45	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	85.18	dres3	1.0
is	500.00	homo3	n
rfl	11123.3	PROCESSING	
rfp	9678.1	lb	1.00
th	7	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

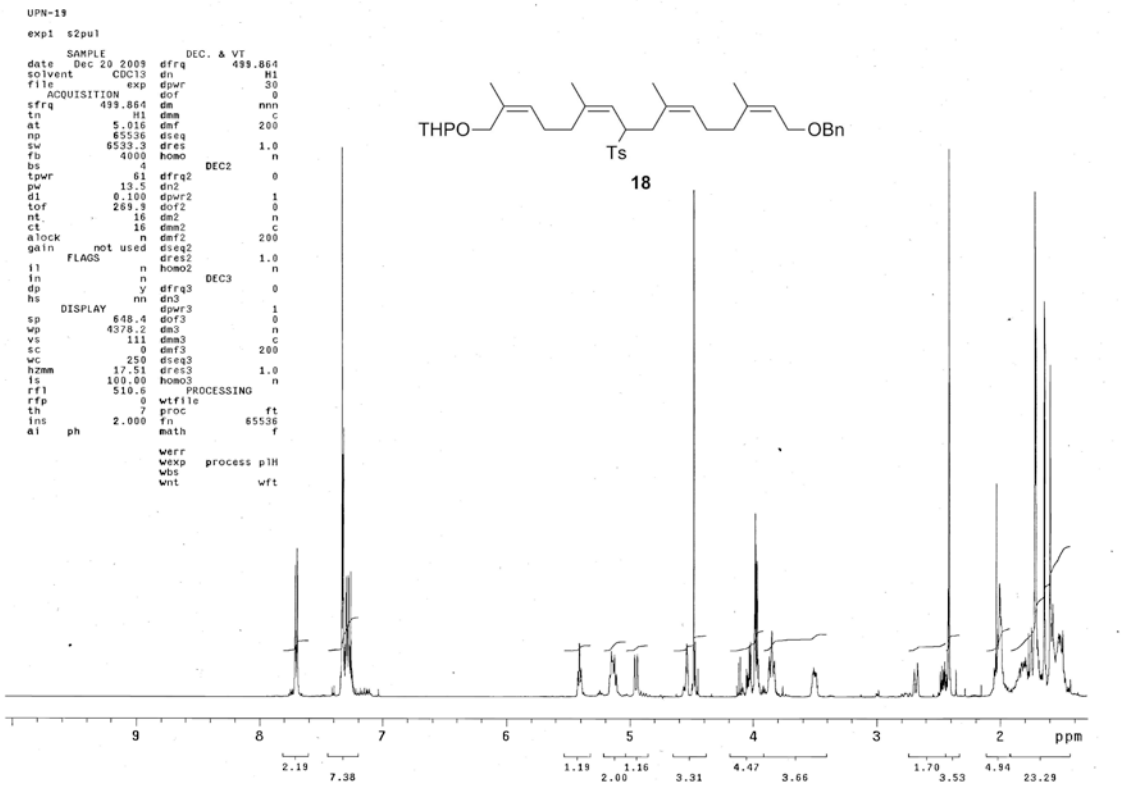


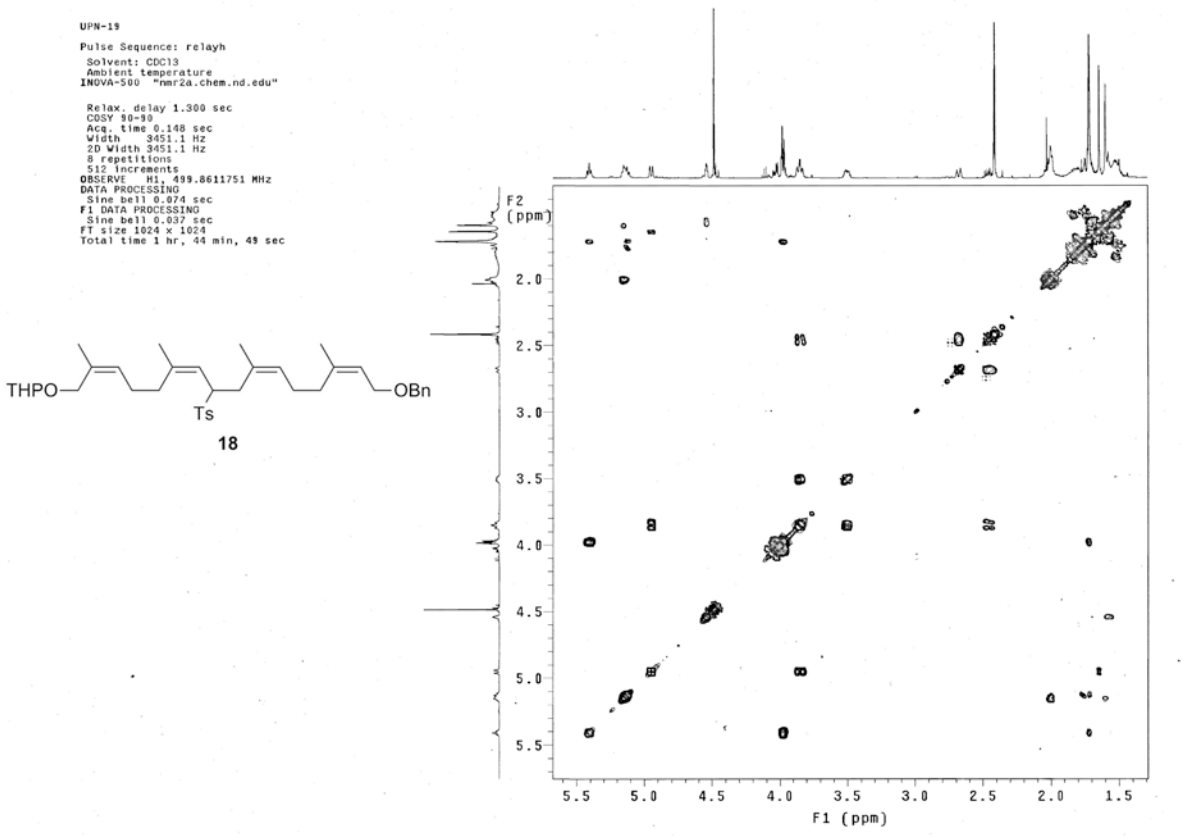
17

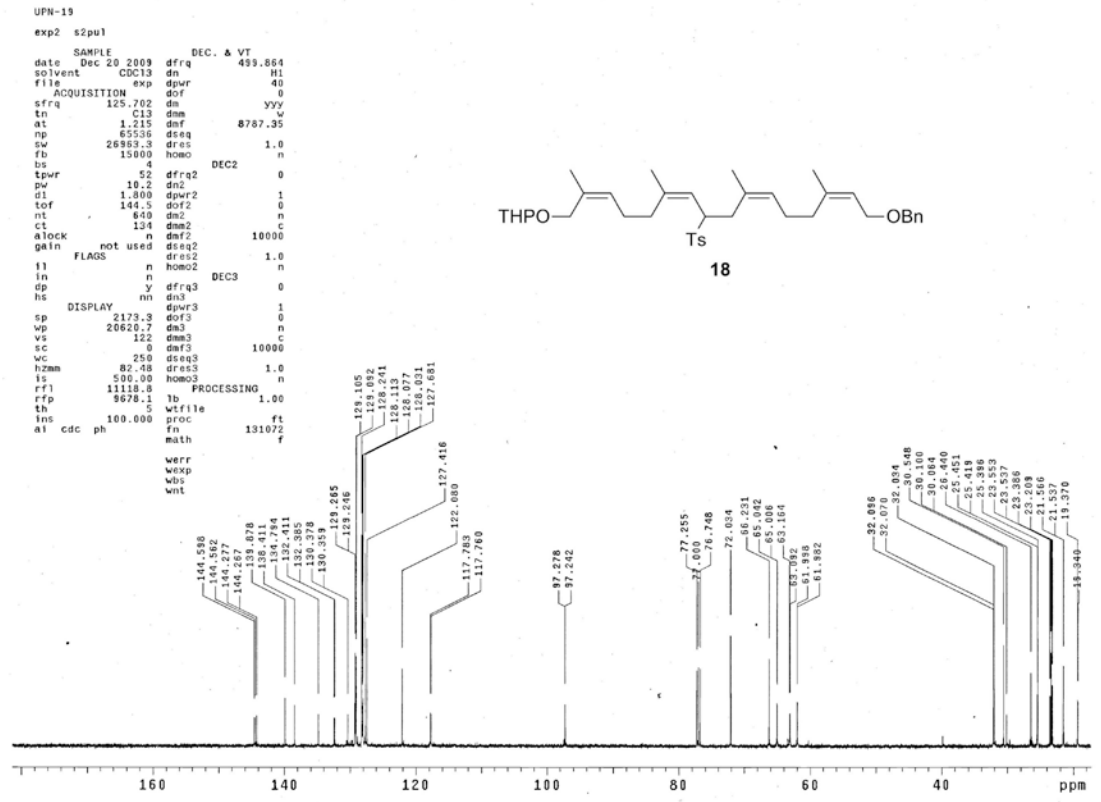


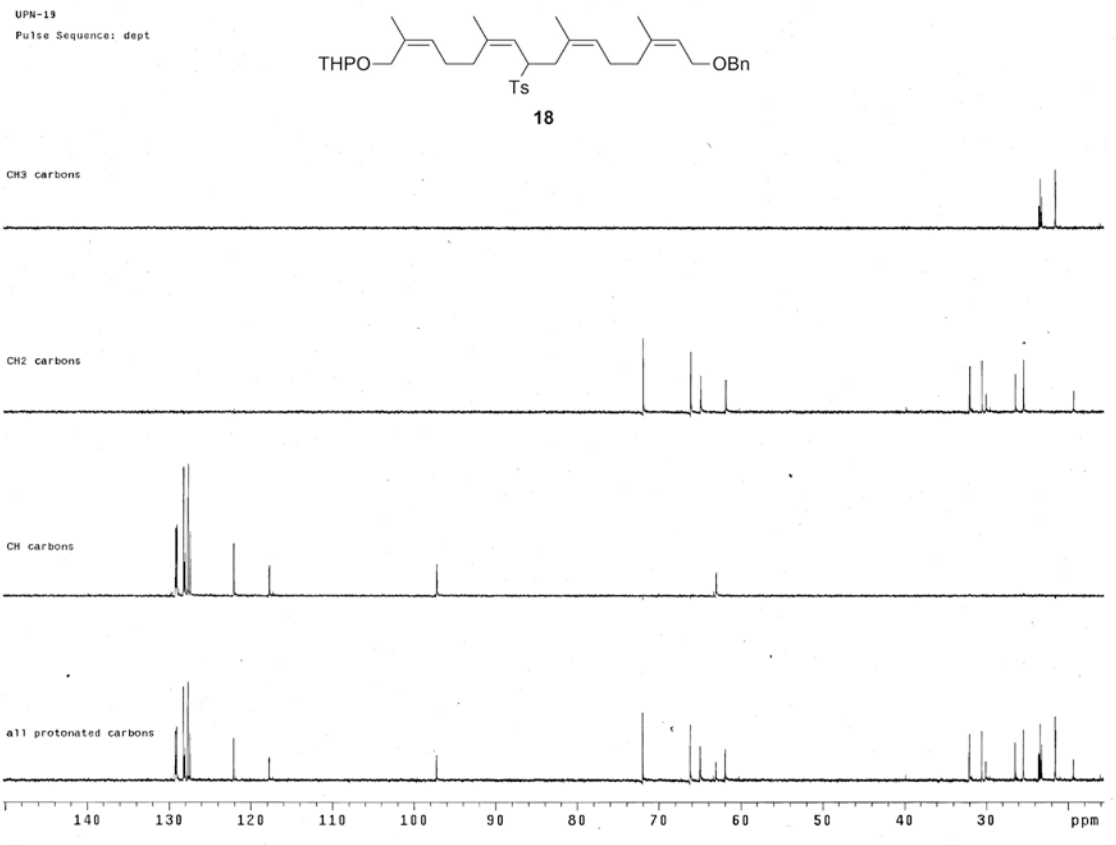

UPN-18_5_OH
Pulse Sequence: dept
HO
Ts
17
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
140
130
120
110
100
90
80
70
60
50
40
30
ppm

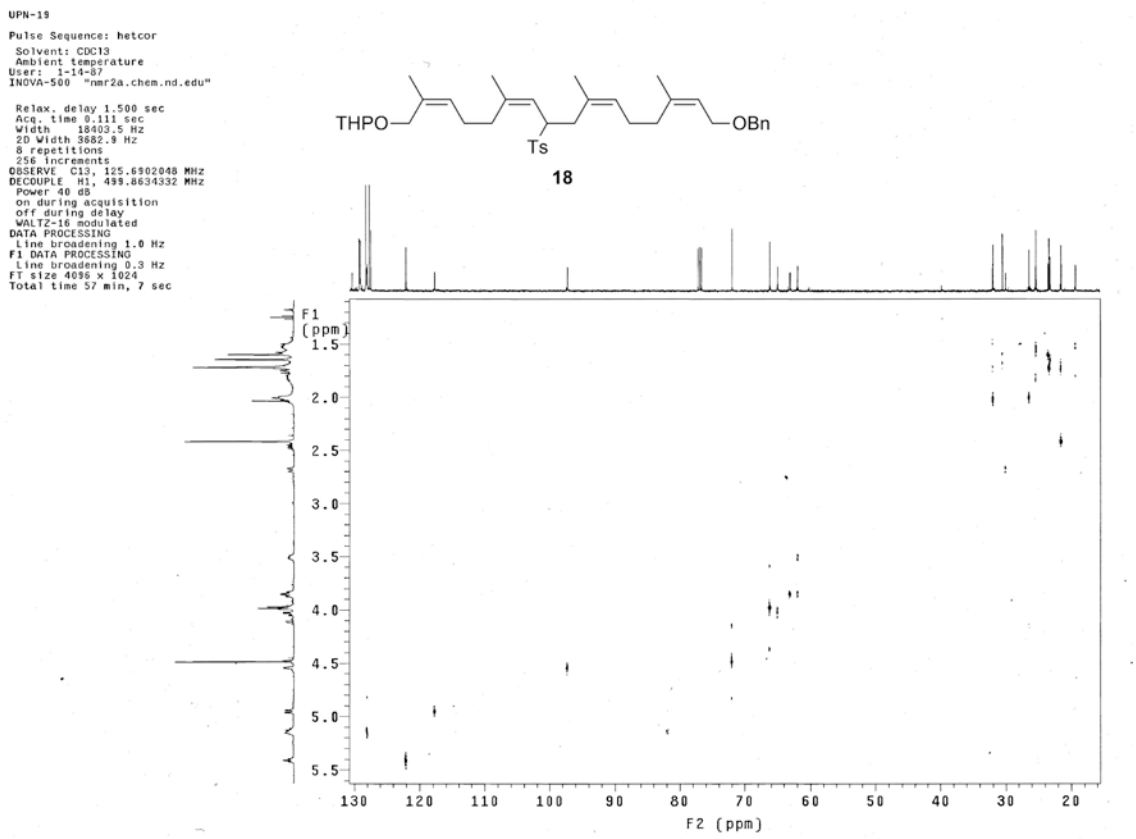

UPN-18_5_OH
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3262.2 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6902094 MHz
DECOUPLE H1, 499.8635233 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 41 sec
HO
Ts
17
F1 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
120
110
100
90
80
70
60
50
40
30
F2 (ppm)

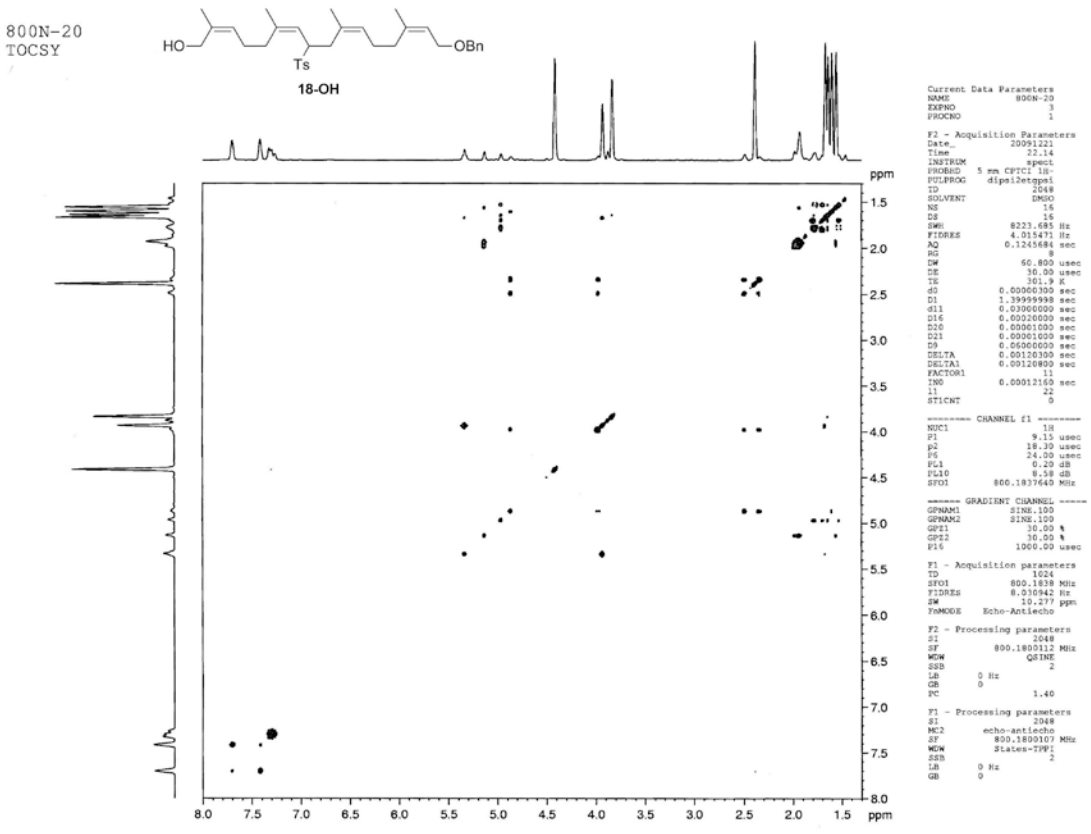

UPN-19
exp1 s2pul
SAMPLE
date Dec 20 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
dl 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 648.4
wp 4378.2
vs 111
sc 0
wc 250
hzmm 17.51
is 100.00
rfl 510.6
rfp 0
th 7
ins 2.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
THPO
OBn
Ts
18
9
8
7
6
5
4
3
2
ppm
2.19
7.38
1.19
1.16
2.00
3.31
4.47
3.66
1.70
3.53
4.94
23.29

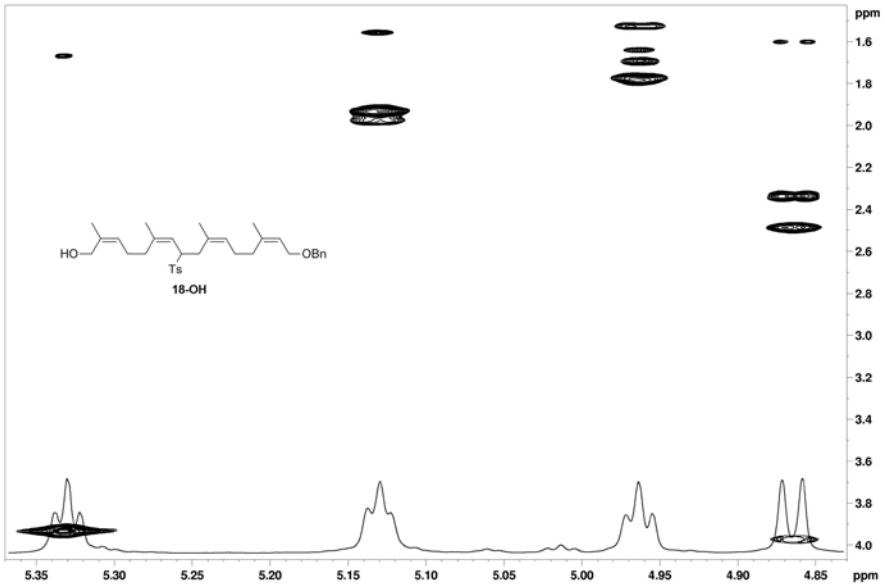

UPN-19
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.148 sec
Width 3451.1 Hz
2D Width 3451.1 Hz
8 repetitions
512 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.074 sec
F1 DATA PROCESSING
Sine bell 0.037 sec
FT size 1024 x 1024
Total time 1 hr, 44 min, 48 sec
THPO
OBn
Ts
18
F2 (ppm)
F1 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
1.5

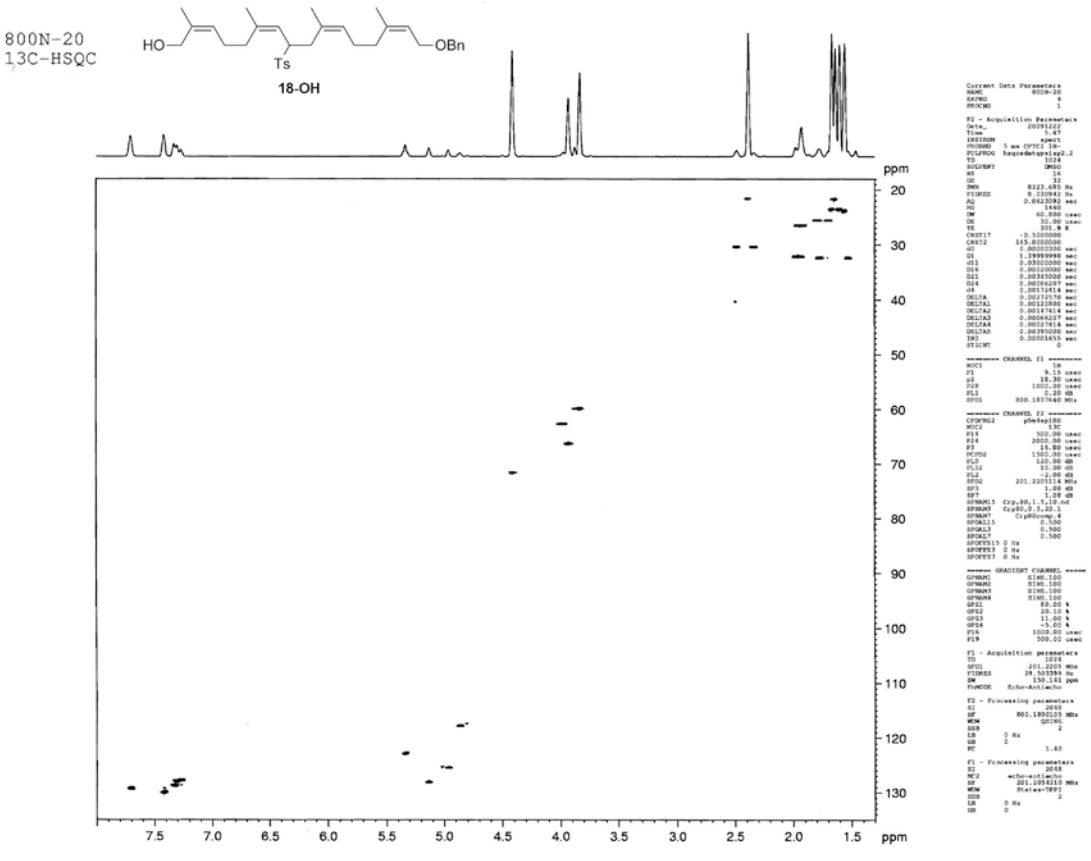

UPN-19
exp2 s2pul
SAMPLE
date Dec 20 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15800
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 134
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 2173.3
wp 20620.7
vs 122
sc 0
wc 250
hzmm 82.48
is 500.00
rfl 11118.8
rfp 9678.1
th 5
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
THPO
OBn
Ts
18
144.598
144.562
144.277
144.267
138.878
138.411
134.794
132.411
132.385
130.378
130.359
129.265
129.248
129.105
129.092
128.241
128.113
128.077
126.031
127.681
127.416
122.080
117.783
117.760
97.278
97.242
77.255
77.000
76.748
72.034
66.231
65.042
65.006
63.164
63.092
61.998
61.982
32.086
32.070
32.034
30.548
30.100
30.064
28.440
25.451
25.419
25.386
23.553
23.527
23.386
23.209
21.566
21.537
19.370
19.340
160
140
120
100
80
60
40
ppm

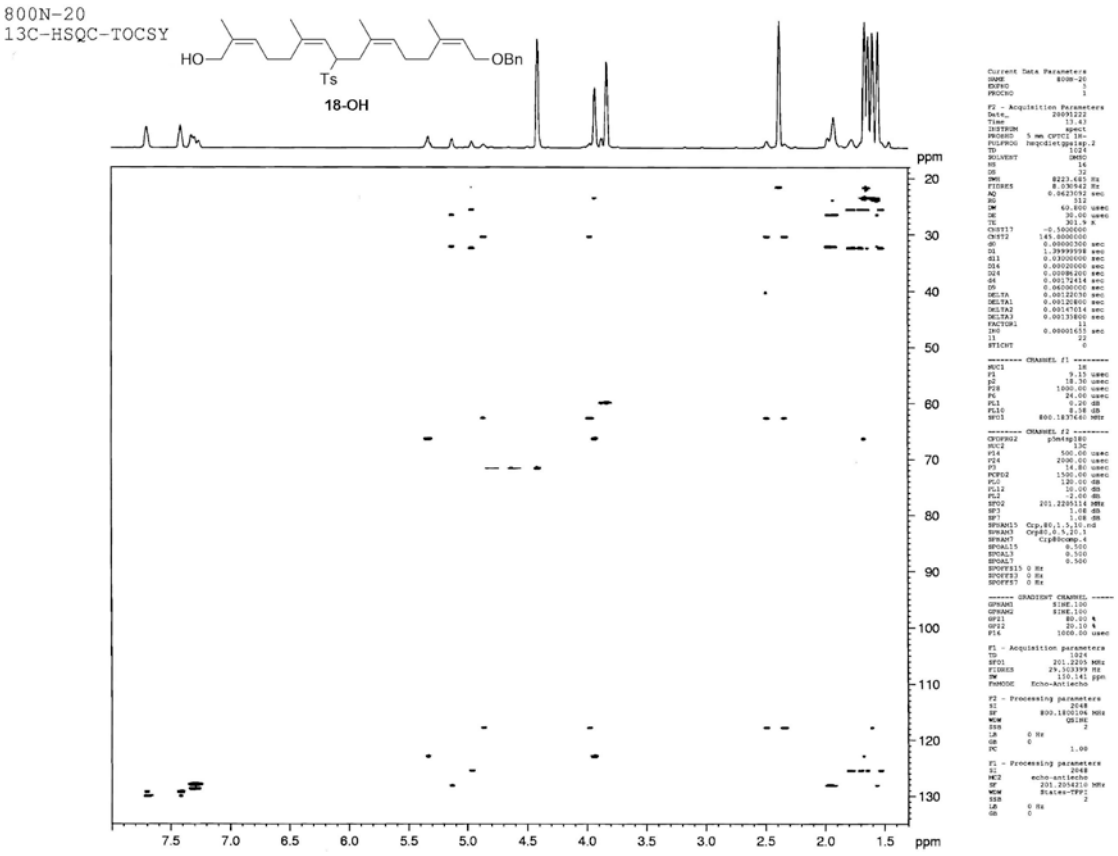

UPN-19
Pulse Sequence: dept
THPO
OBn
Ts
18
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
140
130
120
110
100
90
80
70
60
50
40
30
ppm

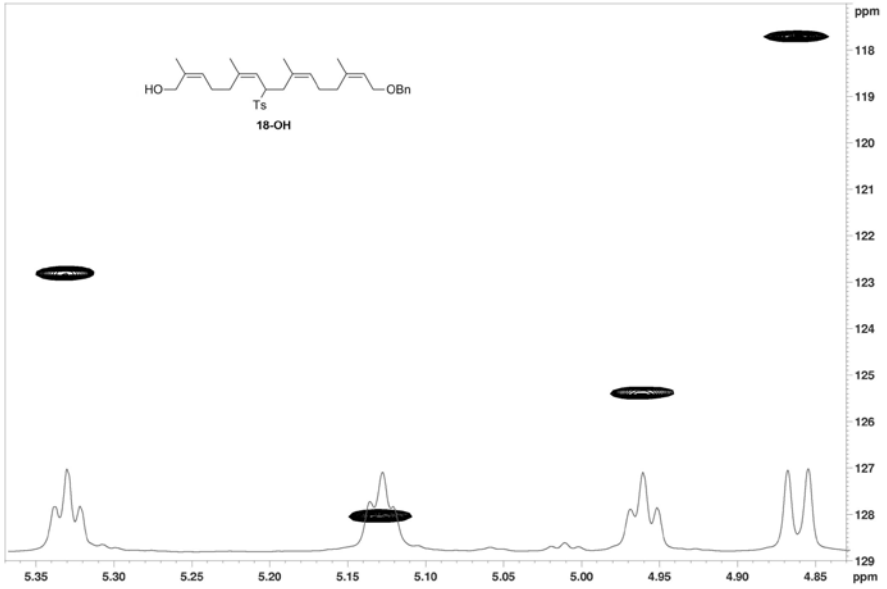

UPN-19
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3682.9 Hz
8 repetitions
256 increments
OBSERVE C13, 125.6902048 MHz
DECOUPLE H1, 499.8634332 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 57 min, 7 sec
THPO
OBn
Ts
18
F1 (ppm)
F2 (ppm)

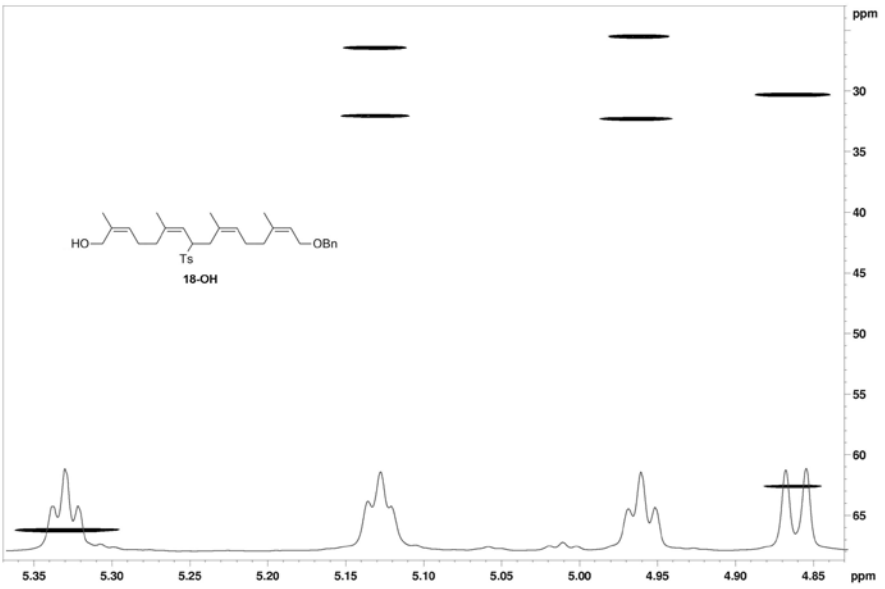

800N-20
TOCSY
HO
OBn
Ts
18-OH
ppm
Current Data Parameters
NAME 800N-20
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20091221
Time 22.14
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG dipsi2etgpsi
TD 2048
SOLVENT DMSO
NS 16
DS 16
SWH 8223.685 Hz
FIDRES 4.015471 Hz
AQ 0.1245684 sec
RG 8
DW 60.800 usec
DE 30.00 usec
TE 301.9 K
D0 0.00000300 sec
D1 1.39999998 sec
D11 0.03000000 sec
D16 0.00020000 sec
D20 0.00001000 sec
D21 0.00001000 sec
D9 0.06900000 sec
DELTA 0.00120300 sec
DELTA1 0.00120900 sec
FACTOR1 11
IN0 0.00012160 sec
l1 22
STSCNT 0
CHANNEL f1
NUC1 1H
P1 9.15 usec
p2 18.30 usec
P6 24.00 usec
PL1 0.20 dB
PL10 9.58 dB
SFO1 800.1837640 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 1024
SFO1 800.1838 MHz
F1DRES 8.030942 Hz
SW 10.277 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 2048
SF 800.1800112 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 2048
MC2 echo-antiecho
SF 800.1800107 MHz
WDW States-TPPI
SSB 2
LB 0 Hz
GB 0


HO
OBn
Ts
18-OH
ppm
1.6
1.8
2.0
2.2
2.4
2.6
2.8
3.0
3.2
3.4
3.6
3.8
4.0
5.35
5.30
5.25
5.20
5.15
5.10
5.05
5.00
4.95
4.90
4.85
ppm

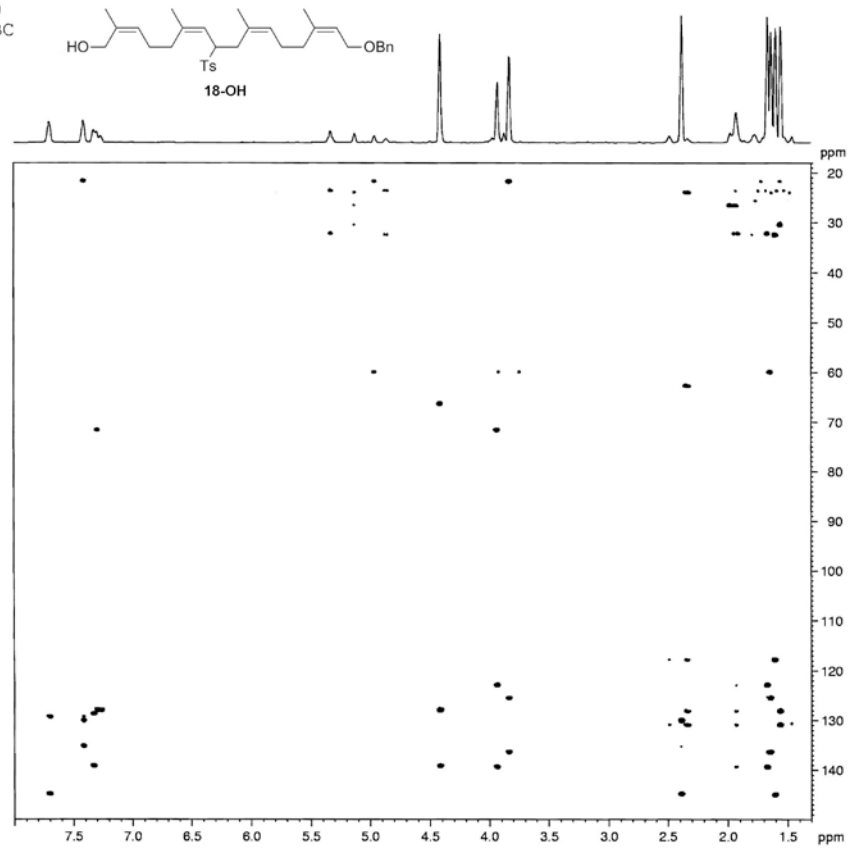

800N-20
13C-HSQC
HO
OBn
Ts
18-OH
ppm
20
30
40
50
60
70
80
90
100
110
120
130
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm


800N-20
13C-HSQC-TOCSY
HO
OBn
Ts
18-OH
ppm
20
30
40
50
60
70
80
90
100
110
120
130
7.5
7.0
6.5
6.0
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
ppm


HO
OBn
Ts
18-OH
ppm
118
119
120
121
122
123
124
125
126
127
128
129
5.35
5.30
5.25
5.20
5.15
5.10
5.05
5.00
4.95
4.90
4.85
ppm



800N-20
13C-HMBC




```
Current Data Parameters
NAME                  800N-20
EXPNO                       7
PROCNO                      1

F2 - Acquisition Parameters
Date_              20091223
Time                  13.53
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG    hmbcgpl2ndqf
TD                      2048
SOLVENT                 DMSO
NS                        60
DS                        64
SWH                 8223.685 Hz
FIDRES              4.015471 Hz
AQ                  0.1245684 sec
RG                       2300
DW                    60.800 usec
DE                    30.00 usec
TE                    301.9 K
CNST13              8.0000000
CNST6             125.0000000
CNST7             175.0000000
d0                  0.00000300 sec
D1                  1.39999998 sec
D16                 0.00200000 sec
d6                  0.06250000 sec
DELTA1              0.00280000 sec
DELTA2              0.00165714 sec
DELTA3              0.06129600 sec
IN0                 0.00001310 sec

======== CHANNEL f1 ========
NUC1                      1H
P1                     9.15 usec
p2                    18.30 usec
PL1                    0.20 dB
SFO1           800.1837640 MHz

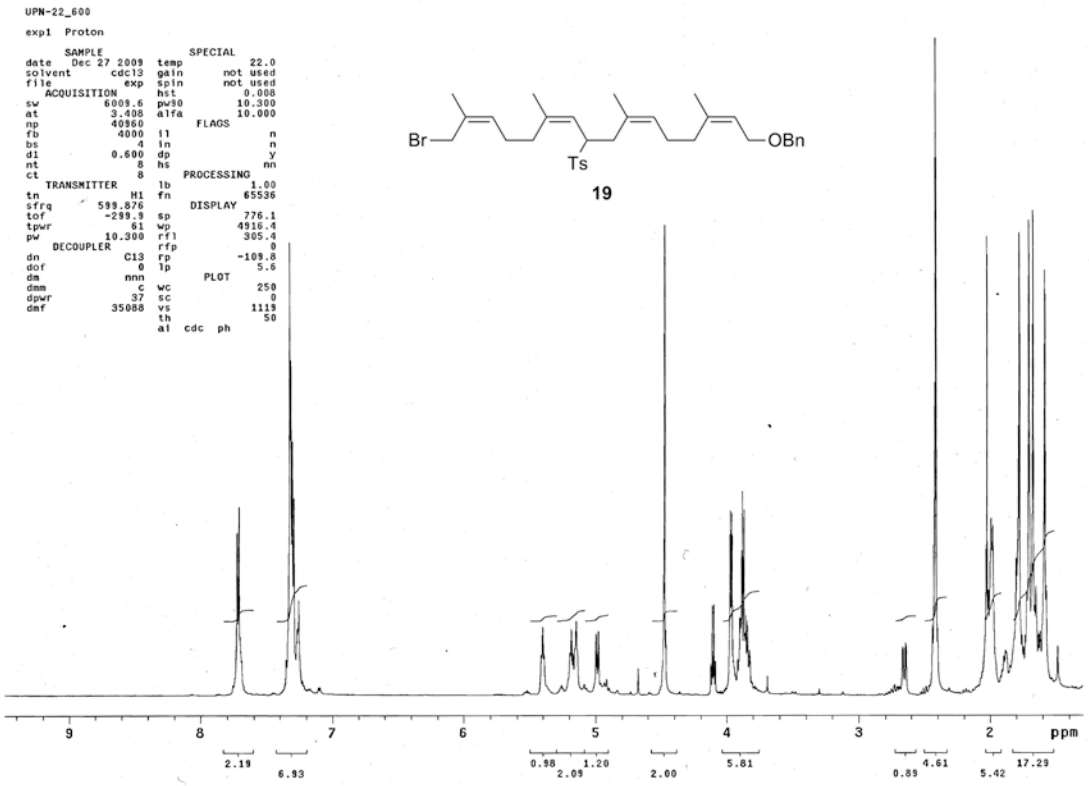
======== CHANNEL f2 ========
NUC2                     13C
P3                    14.80 usec
PL2                   -2.00 dB
SFO2           201.2245355 MHz

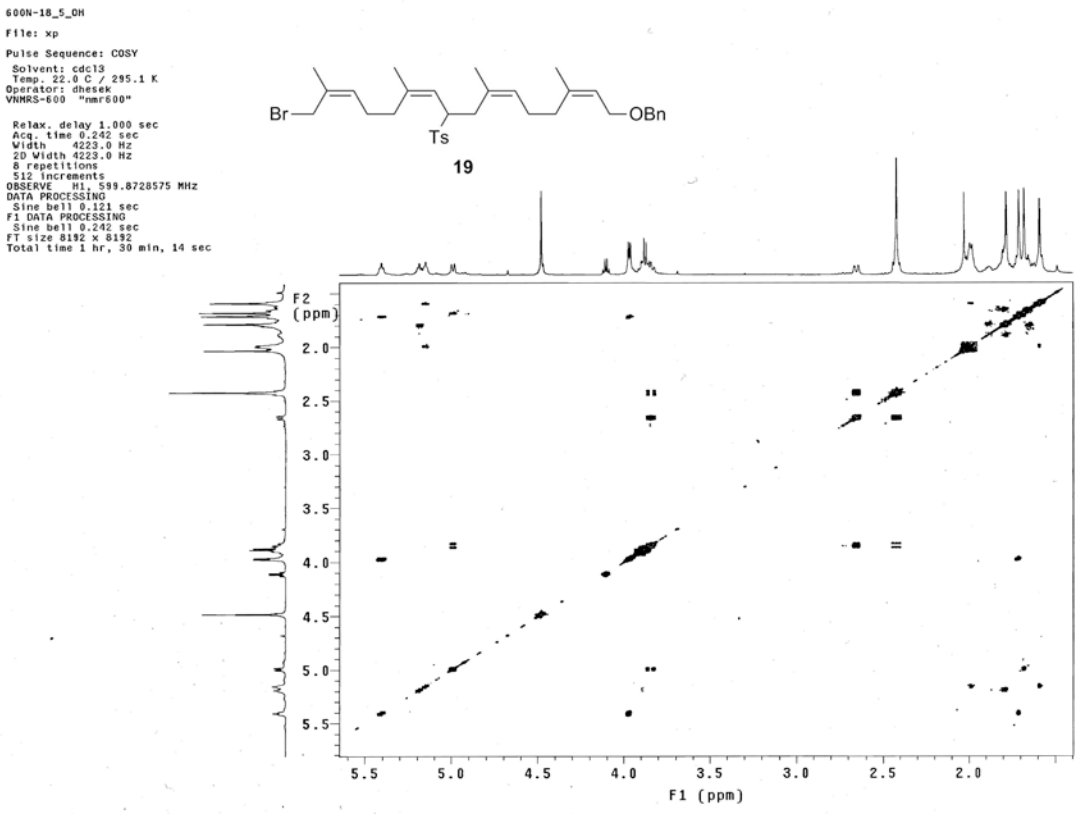
====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPNAM3              SINE.100
GPNAM4              SINE.100
GPNAM5              SINE.100
GPNAM6              SINE.100
GPZ1                  50.00 %
GPZ2                  30.00 %
GPZ3                  40.10 %
GPZ4                  15.00 %
GPZ5                 -10.00 %
GPZ6                  -5.00 %
P16                1000.00 usec

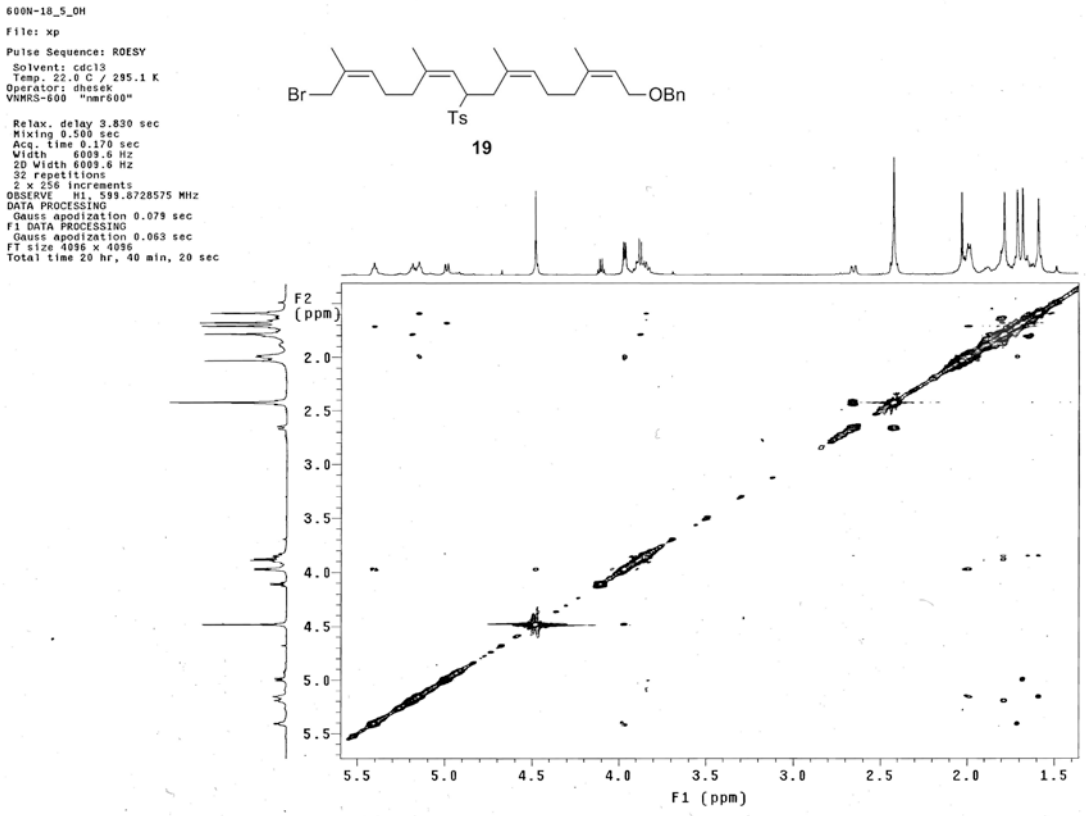
F1 - Acquisition parameters
TD                      1024
SFO1           201.2245 MHz
FIDRES            37.273376 Hz
SW                 169.678 ppm
FnMODE                    QF

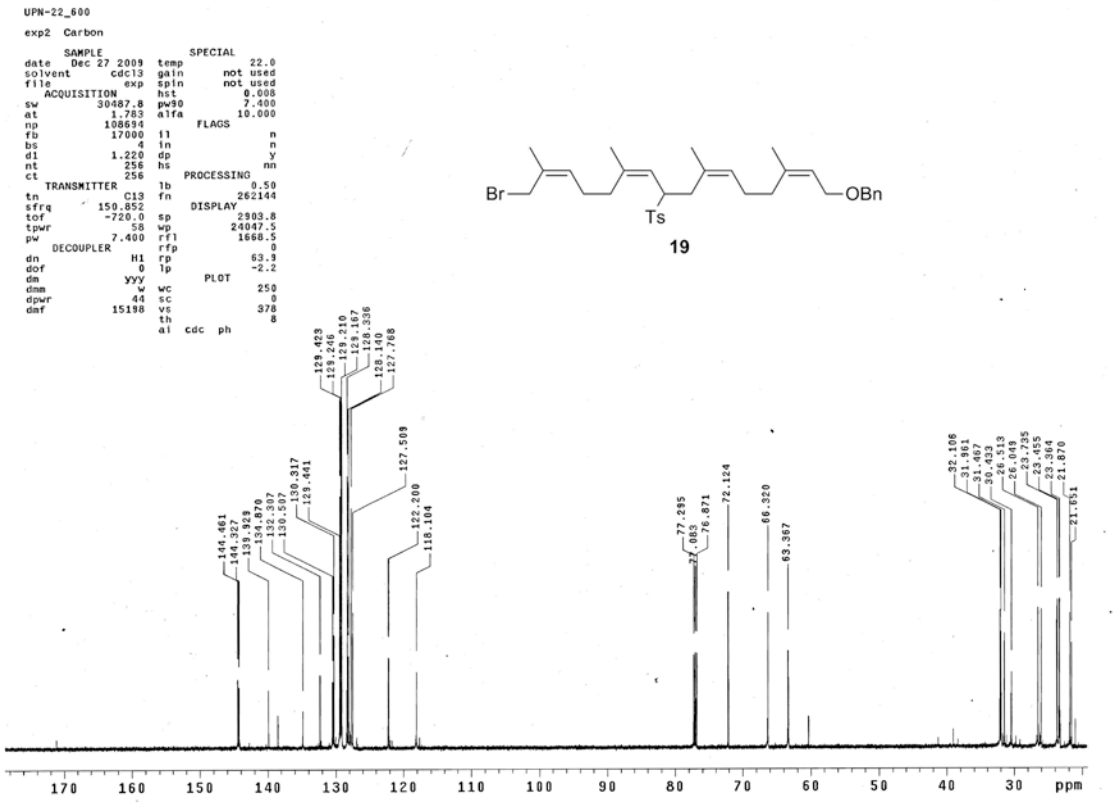
F2 - Processing parameters
SI                      2048
SF             800.1800115 MHz
WDW                    QSINE
SSB          0
LB           0 Hz
GB           0
PC                     1.40

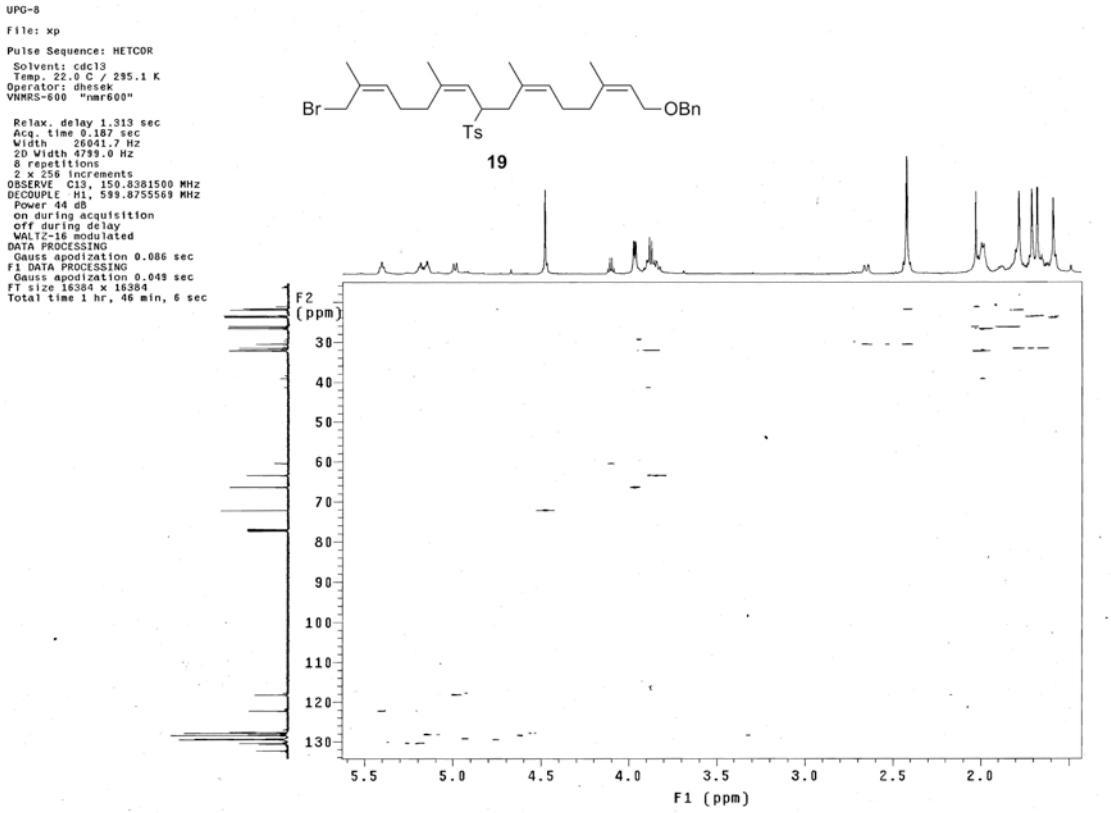
F1 - Processing parameters
SI                      4096
MC2                       QF
SF             201.2054218 MHz
WDW             States-TPPI
SSB          0
LB           0 Hz
GB           0
```

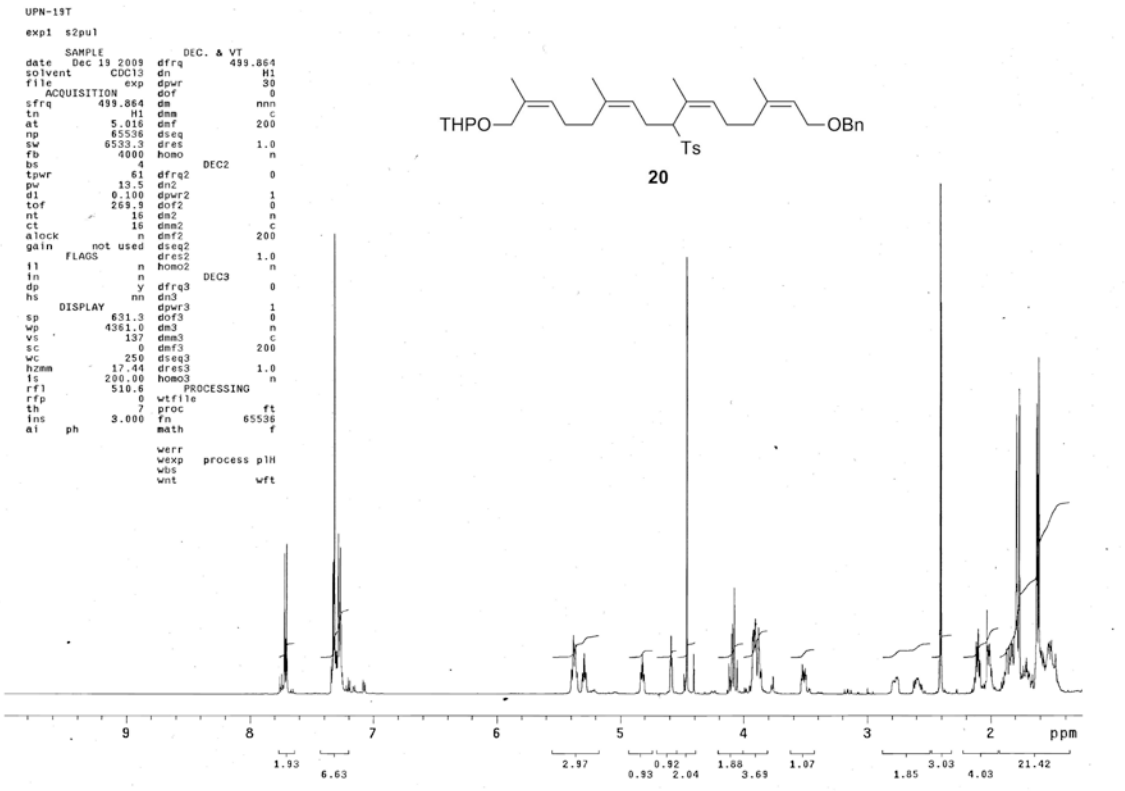

UPN-22_600
exp1 Proton
SAMPLE
date Dec 27 2009
solvent cdcl3
file exp
ACQUISITION
sw 6009.6
at 3.408
np 40960
fb 4000
bs 4
d1 0.600
nt 8
ct 8
TRANSMITTER
tn H1
sfrq 599.876
tof -299.9
tpwr 61
pw 10.300
DECOUPLER
dn C13
dof 0
dm nnn
dmm c
dpwr 37
dmf 35088
SPECIAL
temp 22.0
gain not used
spin not used
hst 0.008
pw90 10.300
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 1.00
fn 65536
DISPLAY
sp 776.1
wp 4916.4
rfl 305.4
rfp 0
rp -109.8
lp 5.6
PLOT
wc 250
sc 0
vs 1119
th 50
al cdc ph
Br
OBn
Ts
19
9
8
7
6
5
4
3
2
ppm
2.19
6.93
0.98
1.20
2.09
2.00
5.81
0.89
4.61
5.42
17.29

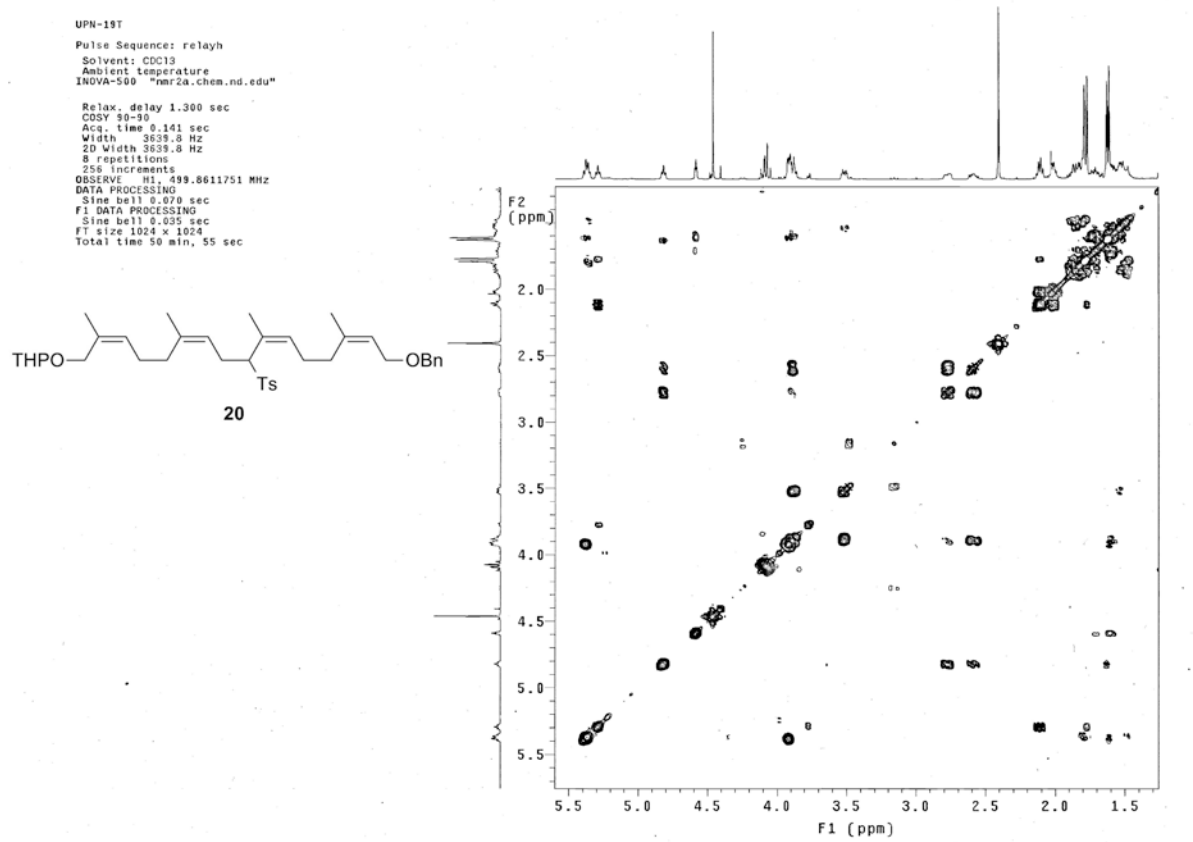

600N-18_5_OH
File: xp
Pulse Sequence: COSY
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"
Relax. delay 1.000 sec
Acq. time 0.242 sec
Width 4223.0 Hz
2D Width 4223.0 Hz
8 repetitions
512 increments
OBSERVE H1, 599.8728575 MHz
DATA PROCESSING
Sine bell 0.121 sec
F1 DATA PROCESSING
Sine bell 0.242 sec
FT size 8192 x 8192
Total time 1 hr, 30 min, 14 sec
Br
OBn
Ts
19
F2 (ppm)
F1 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5

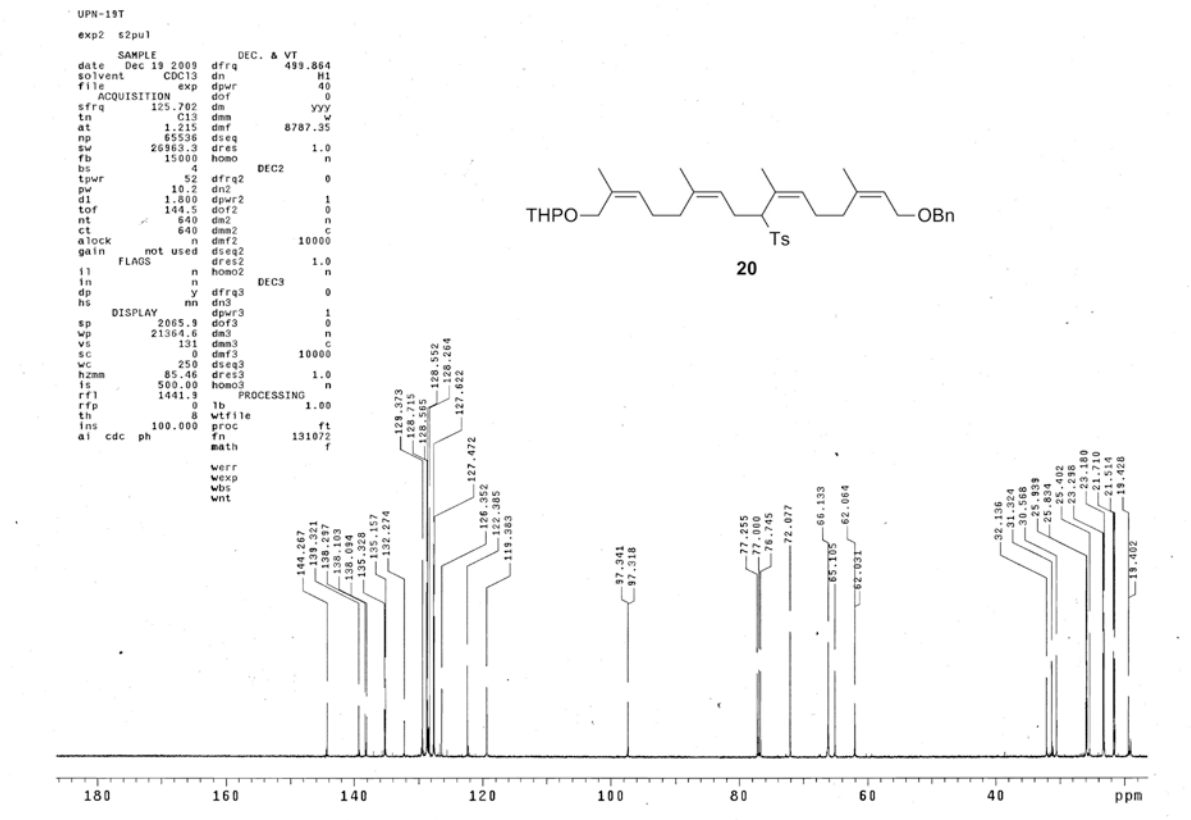

600N-18_5_OH
File: xp
Pulse Sequence: ROESY
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"
Relax. delay 3.830 sec
Mixing 0.500 sec
Acq. time 0.170 sec
Width 6009.6 Hz
20 Width 6009.6 Hz
32 repetitions
2 x 256 increments
OBSERVE H1, 599.8728575 MHz
DATA PROCESSING
Gauss apodization 0.079 sec
F1 DATA PROCESSING
Gauss apodization 0.063 sec
FT size 4096 x 4096
Total time 20 hr, 40 min, 20 sec
Br
OBn
Ts
19
F2 (ppm)
F1 (ppm)

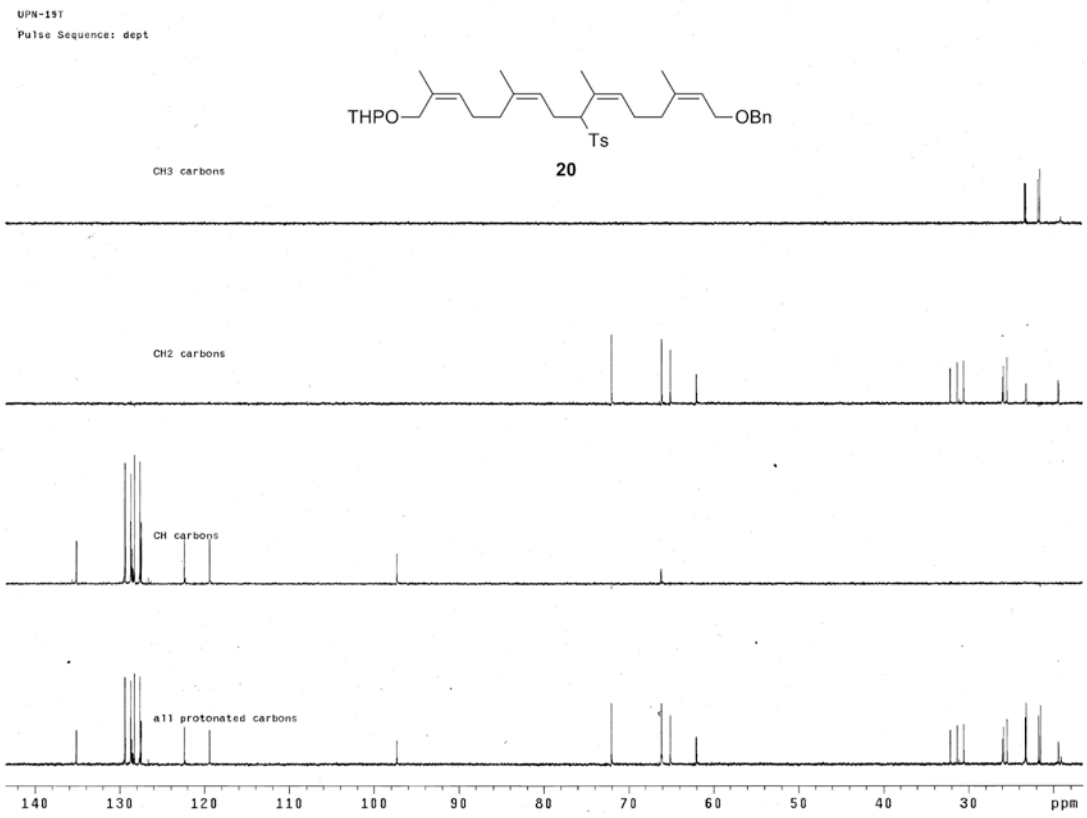

UPN-22_600
exp2 Carbon
SAMPLE
date Dec 27 2009
solvent cdcl3
file exp
ACQUISITION
sw 30487.8
at 1.783
np 108694
fb 17000
bs 4
d1 1.220
nt 256
ct 256
TRANSMITTER
tn C13
sfrq 150.852
tof -720.0
tpwr 58
pw 7.400
DECOUPLER
dn H1
dof 0
dm yyy
dmm w
dpwr 44
dmf 15198
SPECIAL
temp 22.0
gain not used
spin not used
hst 0.008
pw90 7.400
alfa 10.000
FLAGS
il n
in n
dp y
hs nn
PROCESSING
lb 0.50
fn 262144
DISPLAY
sp 2903.8
wp 24047.5
rfl 1668.5
rfp 0
rp 63.8
lp -2.2
PLOT
wc 250
sc 0
vs 378
th 8
al cdc ph
Br
OBn
Ts
19
144.461
144.327
134.870
132.307
130.507
130.317
129.441
129.403
129.245
129.210
129.167
128.336
128.140
127.768
127.509
122.200
118.164
77.295
77.083
76.871
72.124
66.320
63.367
32.106
31.961
31.467
30.483
26.513
26.043
23.735
23.455
23.364
21.870
21.651
ppm

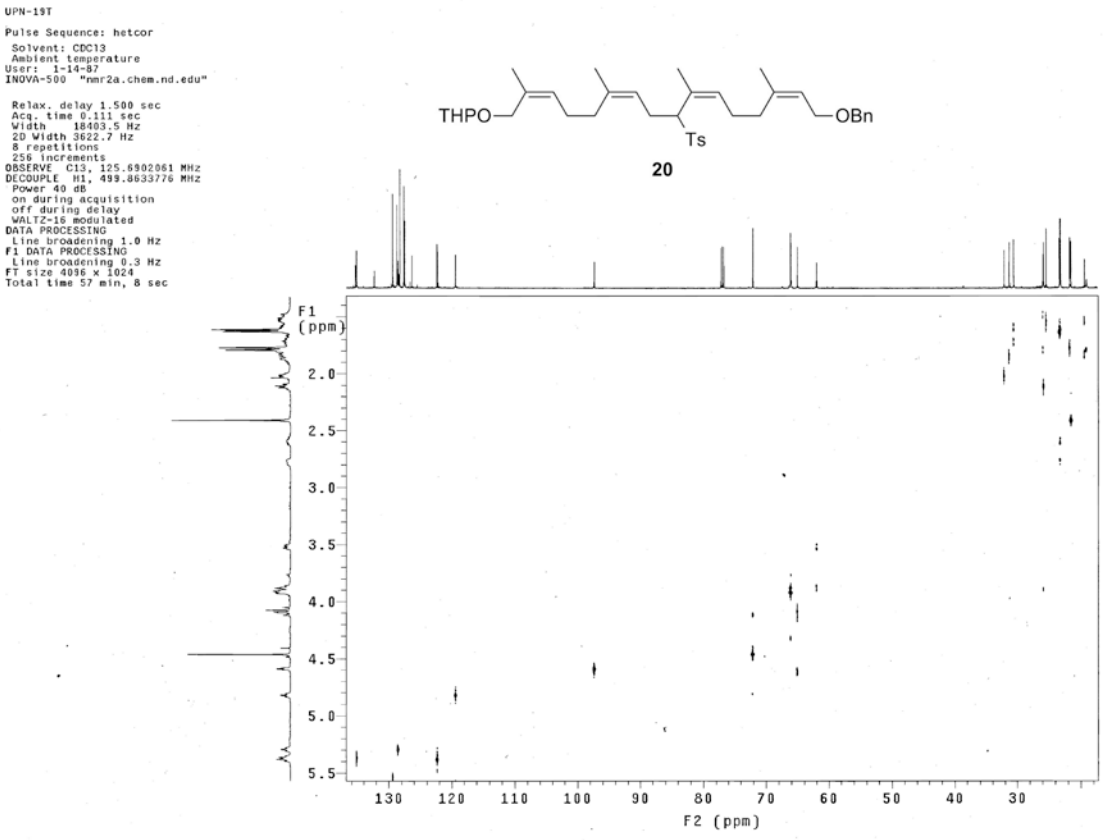

UPG-8
File: xp
Pulse Sequence: HETCOR
Solvent: cdcl3
Temp. 22.0 C / 295.1 K
Operator: dhesek
VNMRS-600 "nmr600"
Relax. delay 1.313 sec
Acq. time 0.187 sec
Width 26041.7 Hz
2D Width 4799.0 Hz
8 repetitions
2 x 256 increments
OBSERVE C13, 150.8381500 MHz
DECOUPLE H1, 599.8755569 MHz
Power 44 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Gauss apodization 0.086 sec
F1 DATA PROCESSING
Gauss apodization 0.049 sec
FT size 16384 x 16384
Total time 1 hr, 46 min, 6 sec
Br
Ts
OBn
19
F2 (ppm)
F1 (ppm)


UPN-19T
exp1 s2pul
SAMPLE
date Dec 19 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 631.3
wp 4361.0
vs 137
sc 0
wc 250
hzmm 17.44
is 200.00
rfl 510.6
rfp 0
th 7
ins 3.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
THPO
Ts
OBn
20
ppm
1.93
6.63
2.97
0.93
0.92
2.04
1.88
3.69
1.07
1.85
3.03
4.03
21.42

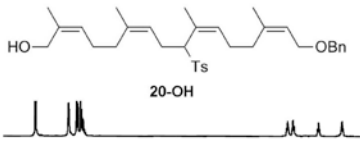

UPN-19T
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.141 sec
Width 3639.8 Hz
2D Width 3639.8 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 50 min, 55 sec
THPO
OBn
Ts
20
F2 (ppm)
F1 (ppm)

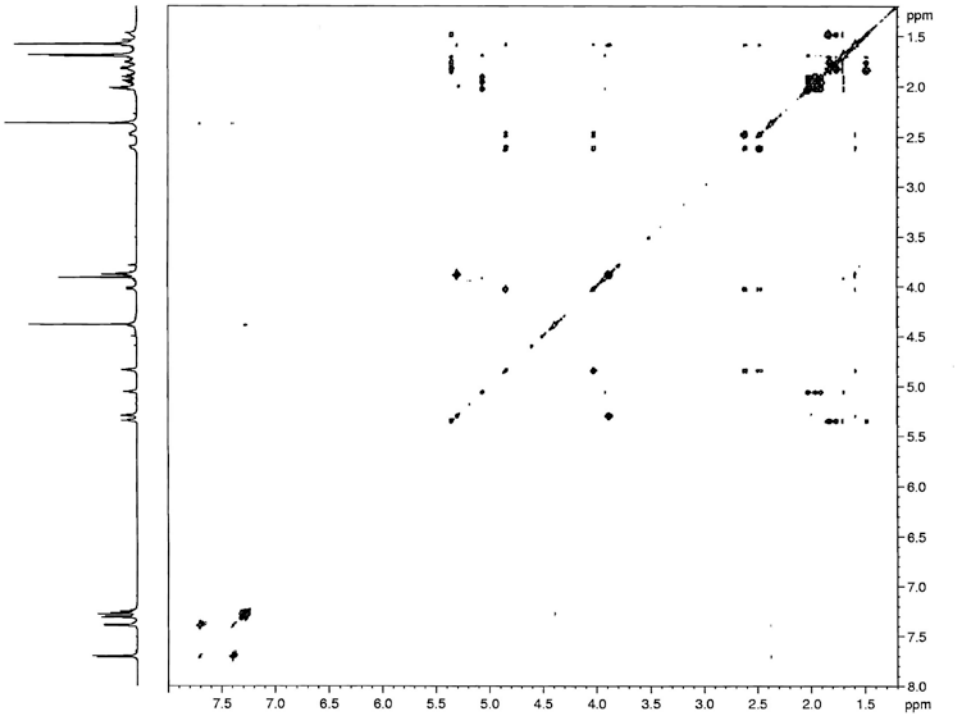

UPN-19T
exp2 s2pul
SAMPLE
date Dec 19 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 640
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 2065.9
wp 21364.6
vs 131
sc 0
wc 250
hzmm 85.46
is 500.00
rfl 1441.9
rfp 0
th 8
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
THPO
OBn
Ts
20
144.267
139.321
138.287
138.103
138.094
135.328
135.157
132.274
129.373
128.715
128.565
128.552
128.264
127.622
127.472
126.352
126.065
119.383
97.341
97.318
77.255
77.000
76.745
72.077
66.133
65.105
62.064
62.031
32.136
31.324
30.568
25.939
25.834
25.402
23.298
23.180
21.710
21.514
19.428
19.402
ppm


UPN-19T
Pulse Sequence: dept
THPO
OBn
Ts
20
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
140
130
120
110
100
90
80
70
60
50
40
30
ppm


UPN-19T
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3622.7 Hz
8 repetitions
256 increments
OBSERVE C13, 125.6902061 MHz
DECOUPLE H1, 499.8633776 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 57 min, 8 sec
THPO
OBn
Ts
20
F1 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
130
120
110
100
90
80
70
60
50
40
30
F2 (ppm)

800N-20T
TOCSY






```
Current Data Parameters
NAME            DMSO-N-20T
EXPNO                    3
PROCNO                   1

F2 - Acquisition Parameters
Date_          20091225
Time              22.11
INSTRUM            spect
PROBHD   5 mm CPTCI 1H-
PULPROG    dipsi2etgpsi
TD                 2048
SOLVENT            DMSO
NS                   16
DS                   16
SWH           8223.685 Hz
FIDRES        4.015471 Hz
AQ            0.1245684 sec
RG                  4.5
DW                60.800 usec
DE                30.00 usec
TE                301.9 K
d0            0.00000300 sec
D1            1.39999998 sec
d11           0.03000000 sec
D16           0.00020000 sec
D20           0.00001000 sec
D21           0.00001000 sec
D9            0.06000000 sec
DELTA         0.00120300 sec
DELTA1        0.00120800 sec
FACTOR1              11
IN0           0.00012160 sec
l1                   22
ST1CNT                0

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.30 usec
p2                18.60 usec
P6                24.00 usec
PL1                0.20 dB
PL10               8.43 dB
SFO1        800.1837640 MHz

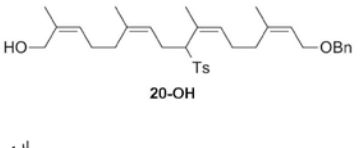
====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPZ1               30.00 %
GPZ2               30.00 %
P16              1000.00 usec

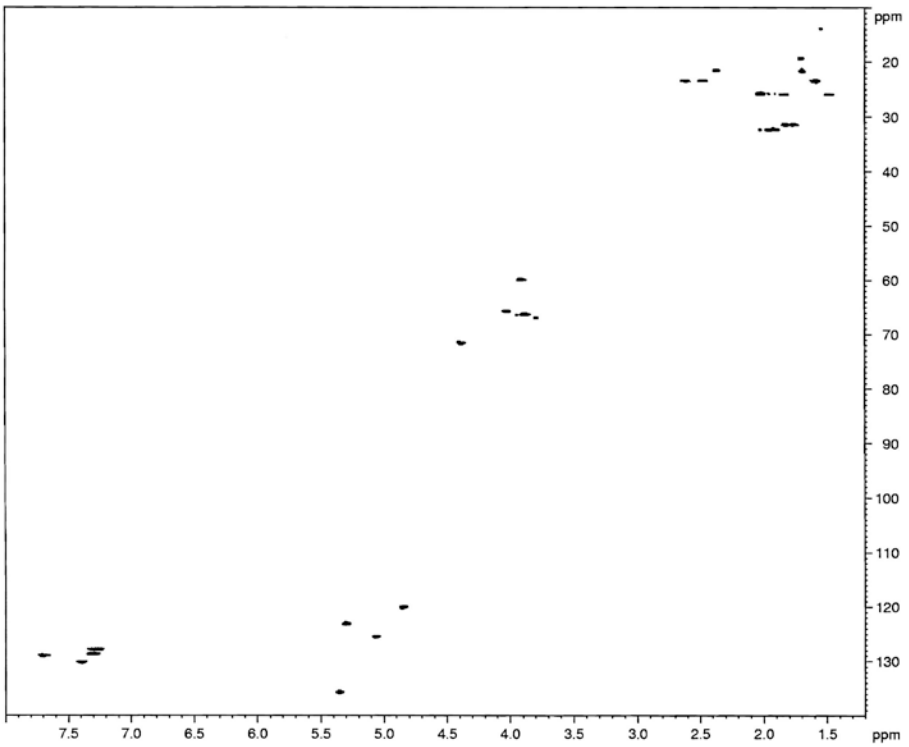
F1 - Acquisition parameters
TD                 1024
SFO1          800.1838 MHz
FIDRES        8.030942 Hz
SW               10.277 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                 2048
SF          800.1800157 MHz
WDW                QSINE
SSB                   2
LB         0 Hz
GB         0
PC                 1.40

F1 - Processing parameters
SI                 2048
MC2        echo-antiecho
SF          800.1800158 MHz
WDW          States-TPPI
SSB                   2
LB         0 Hz
GB         0
```

800N-20T
13C-HSQC






```
Current Data Parameters
NAME            DMSO-N-20T
EXPNO                    4
PROCNO                   1

F2 - Acquisition Parameters
Date_          20091226
Time              5.44
INSTRUM            spect
PROBHD   5 mm CPTCI 1H-
PULPROG  hsqcedetgpsisp2.2
TD                 1024
SOLVENT            DMSO
NS                   16
DS                   64
SWH           8223.685 Hz
FIDRES        8.030942 Hz
AQ            0.0623092 sec
RG                  512
DW                60.800 usec
DE                30.00 usec
TE                301.9 K
CNST17         -0.5000000
CNST2        145.0000000
d0            0.00002300 sec
D1            1.39999998 sec
d11           0.03000000 sec
D16           0.00020000 sec
D21           0.00345000 sec
D24           0.00086207 sec
d4            0.00172414 sec
DELTA         0.00272540 sec
DELTA1        0.00120800 sec
DELTA2        0.00147614 sec
DELTA3        0.00086207 sec
DELTA4        0.00027414 sec
DELTA5        0.00395000 sec
IN0           0.00001655 sec
ST1CNT                0

======== CHANNEL f1 ========
NUC1                 1H
P1                 9.30 usec
p2                18.60 usec
P28             1000.00 usec
PL1                0.20 dB
SFO1        800.1837640 MHz

======== CHANNEL f2 ========
CPDPRG2        pMetap180
NUC2                13C
P14              500.00 usec
P24             2000.00 usec
P3                14.80 usec
PCPD2           1500.00 usec
PL0              120.00 dB
PL12              10.00 dB
PL2                2.00 dB
SFO2        201.2203514 MHz
SP3                1.08 dB
SP7                1.58 dB
SPNAM15  Crp,80,1.5,10.ad
SPNAM3   Crp80,0.5,20.1
SPNAM7   Crp80comp.4
SPOAL15           0.500
SPOAL3            0.500
SPOAL7            0.500
SPOFFS15 0 Hz
SPOFFS3  0 Hz
SPOFFS7  0 Hz

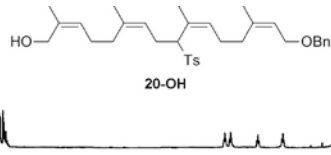
====== GRADIENT CHANNEL =====
GPNAM1          SINE.100
GPNAM2          SINE.100
GPNAM3          SINE.100
GPNAM4          SINE.100
GPZ1               80.00 %
GPZ2               20.10 %
GPZ3               11.00 %
GPZ4               -5.00 %
P16              1000.00 usec
P19               300.00 usec

F1 - Acquisition parameters
TD                 1024
SFO1          201.2205 MHz
FIDRES        29.503399 Hz
SW              190.141 ppm
FnMODE     Echo-Antiecho

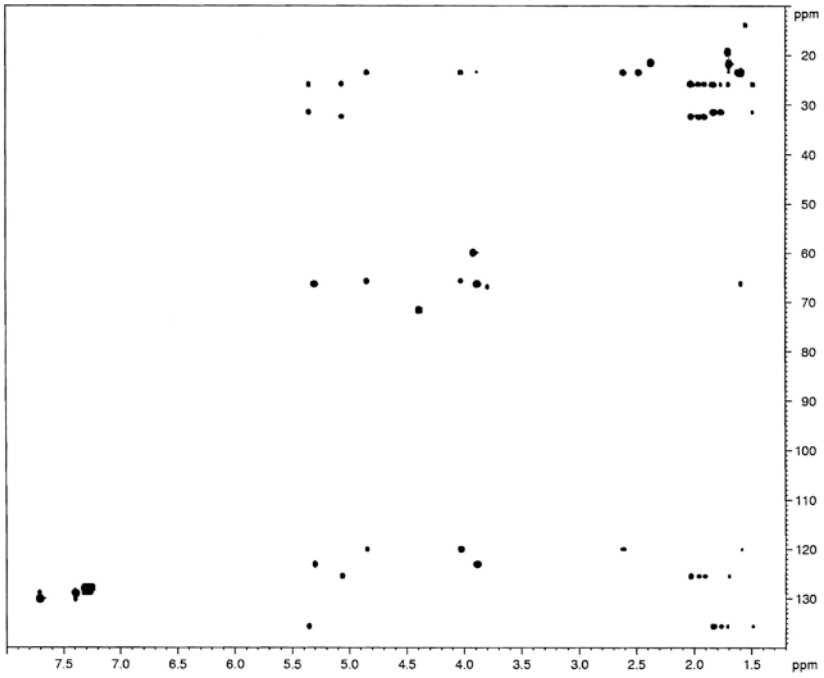
F2 - Processing parameters
SI                 2048
SF          800.1800136 MHz
WDW                QSINE
SSB                   2
LB         0 Hz
GB         0
PC                 1.40

F1 - Processing parameters
SI                 2048
MC2        echo-antiecho
SF          201.2054210 MHz
WDW          States-TPPI
SSB                   2
LB         0 Hz
GB         0
```

800N-20T
13C-HSQC-TOCSY



20-OH



Current Data Parameters
NAME DMSO-N-20T
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091226
Time 12.36
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hsqcdietgpsisp.2
TD 1024
SOLVENT DMSO
NS 16
DS 16
SWH 8223.685 Hz
FIDRES 8.030942 Hz
AQ 0.0623092 sec
RG 456
DW 60.800 usec
DE 30.00 usec
TE 301.9 K
CNST17 -0.5000000
CNST2 145.0000000
d0 0.00000300 sec
D1 1.29999998 sec
d11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
d4 0.00172414 sec
D9 0.04000000 sec
DELTA 0.00122060 sec
DELTA1 0.00120400 sec
DELTA2 0.00147014 sec
DELTA3 0.00135400 sec
FACTOR1 11
IN0 0.00001655 sec
11 22
SFICNT 0

======== CHANNEL f1 ========
NUC1 1H
P1 9.30 usec
p2 18.60 usec
PL1 1000.00 usec
P6 24.00 usec
PL1 0.20 dB
PL10 8.49 dB
SFO1 800.1837640 MHz

======== CHANNEL f2 ========
CPDPRG2 gbsetgp180
NUC2 13C
P14 500.00 usec
P24 2000.00 usec
P3 14.80 usec
PCPD2 1300.00 usec
PL0 120.00 dB
PL12 10.00 dB
PL2 -2.00 dB
SFO2 201.2205114 MHz
SP3 1.08 dB
SP7 1.08 dB
SPNAM15 Crp,80,1.5,10.nd
SPNAM3 Crp80,0.5,20.1
SPNAM7 Crp80comp.4
SPOAL15 0.500
SPOAL3 0.500
SPOAL7 0.500
SPOFFS15 0 Hz
SPOFFS3 0 Hz
SPOFFS7 0 Hz

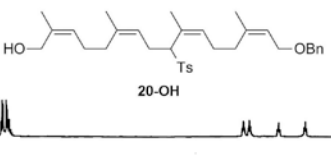
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 1024
SFO1 201.2205 MHz
FIDRES 29.503399 Hz
SW 150.141 ppm
FnMODE Echo-Antiecho

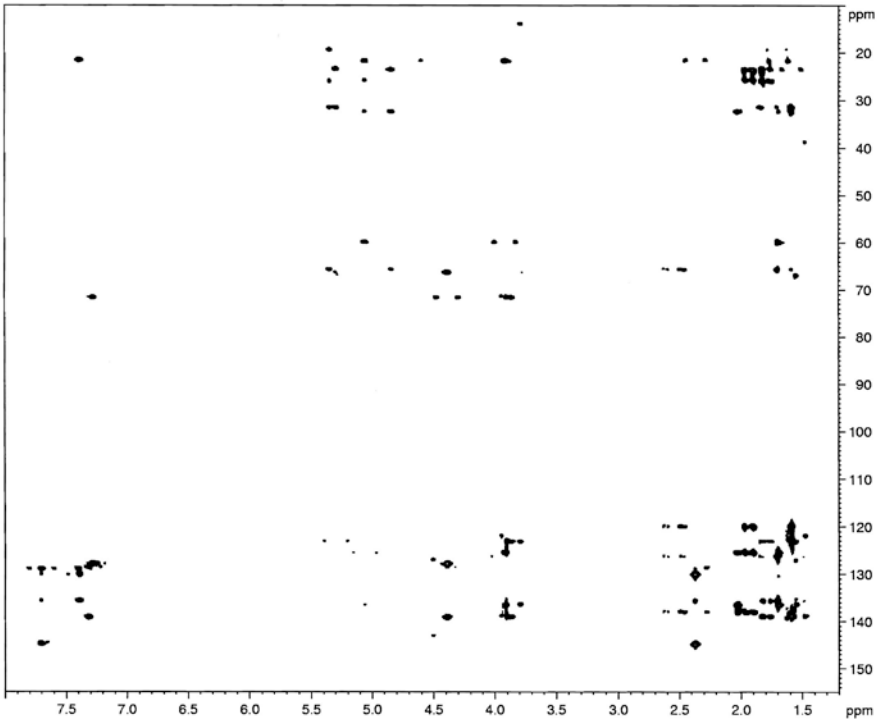
F2 - Processing parameters
SI 2048
SF 800.1800150 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 2048
MC2 echo-antiecho
SF 201.2054219 MHz
WDW States-TPPI
SSB 2
LB 0 Hz
GB 0

800N-20T
13C-HMBC



20-OH



Current Data Parameters
NAME DMSO-N-20T
EXPNO 7
PROCNO 1

F2 - Acquisition Parameters
Date_ 20091227
Time 11.49
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 80
DS 64
SWH 8223.685 Hz
FIDRES 4.015471 Hz
AQ 0.1245684 sec
RG 2050
DW 60.800 usec
DE 30.00 usec
TE 301.9 K
CNST13 8.0000000
CNST6 125.0000000
CNST7 175.0000000
d0 0.00000300 sec
D1 1.29999998 sec
D16 0.00020000 sec
d6 0.06250000 sec
DELTA1 0.00280000 sec
DELTA2 0.00165714 sec
DELTAJ 0.06129600 sec
IN0 0.00001310 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.30 usec
p2 18.60 usec
PL1 0.20 dB
SFO1 800.1837640 MHz

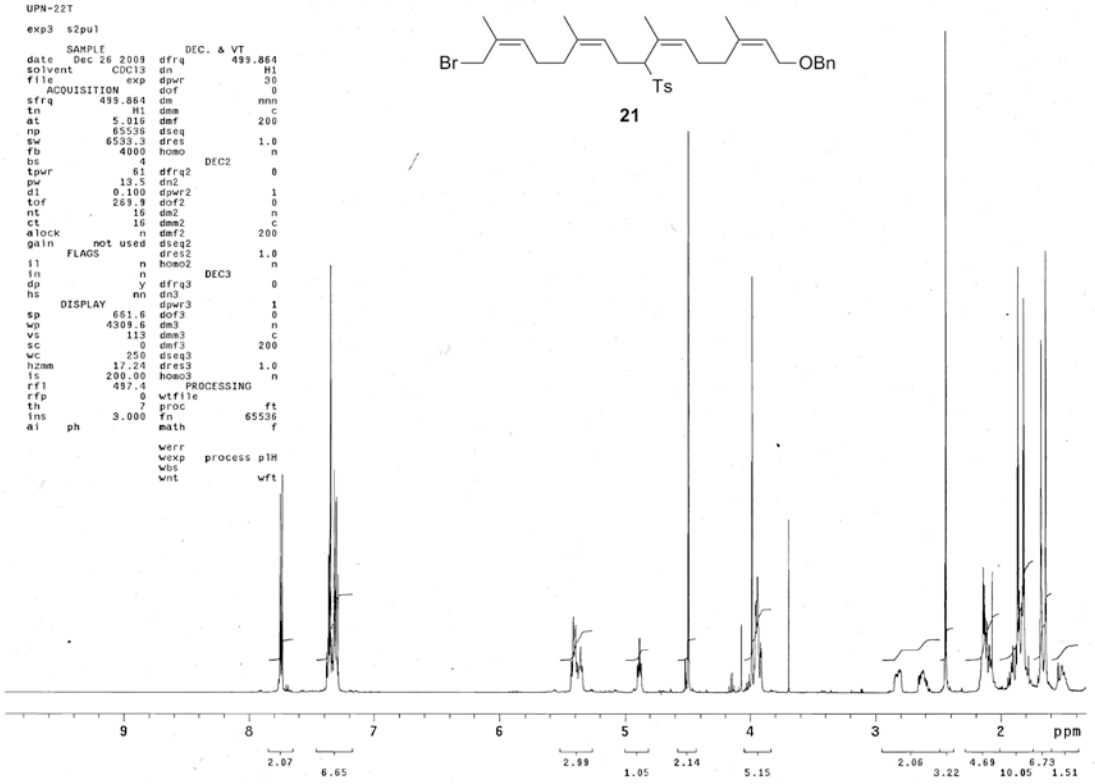
======== CHANNEL f2 ========
NUC2 13C
P3 14.80 usec
PL2 -2.00 dB
SFO2 201.2245355 MHz

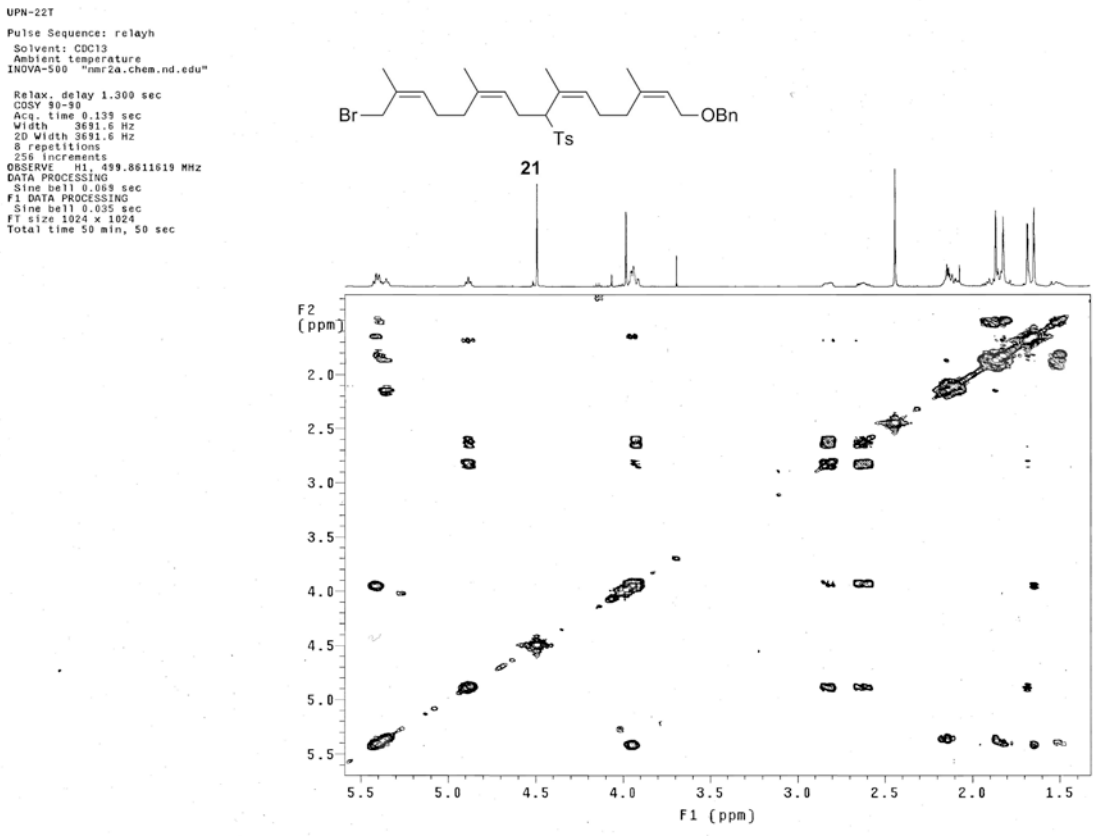
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPNAM5 SINE.100
GPNAM6 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %
GPZ5 -10.00 %
GPZ6 -5.00 %
P16 1000.00 usec

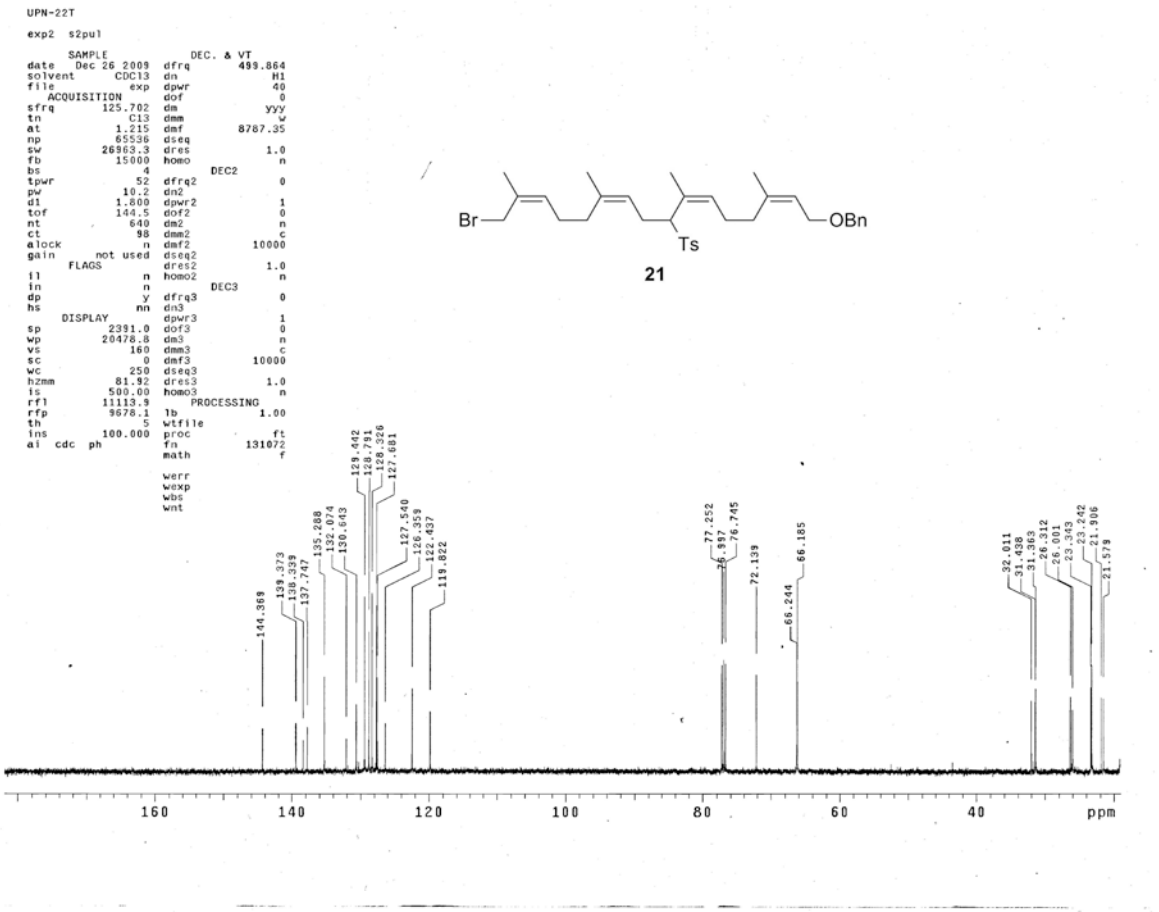
F1 - Acquisition parameters
TD 512
SFO1 201.2245 MHz
FIDRES 74.546753 Hz
SW 189.678 ppm
FnMODE QF

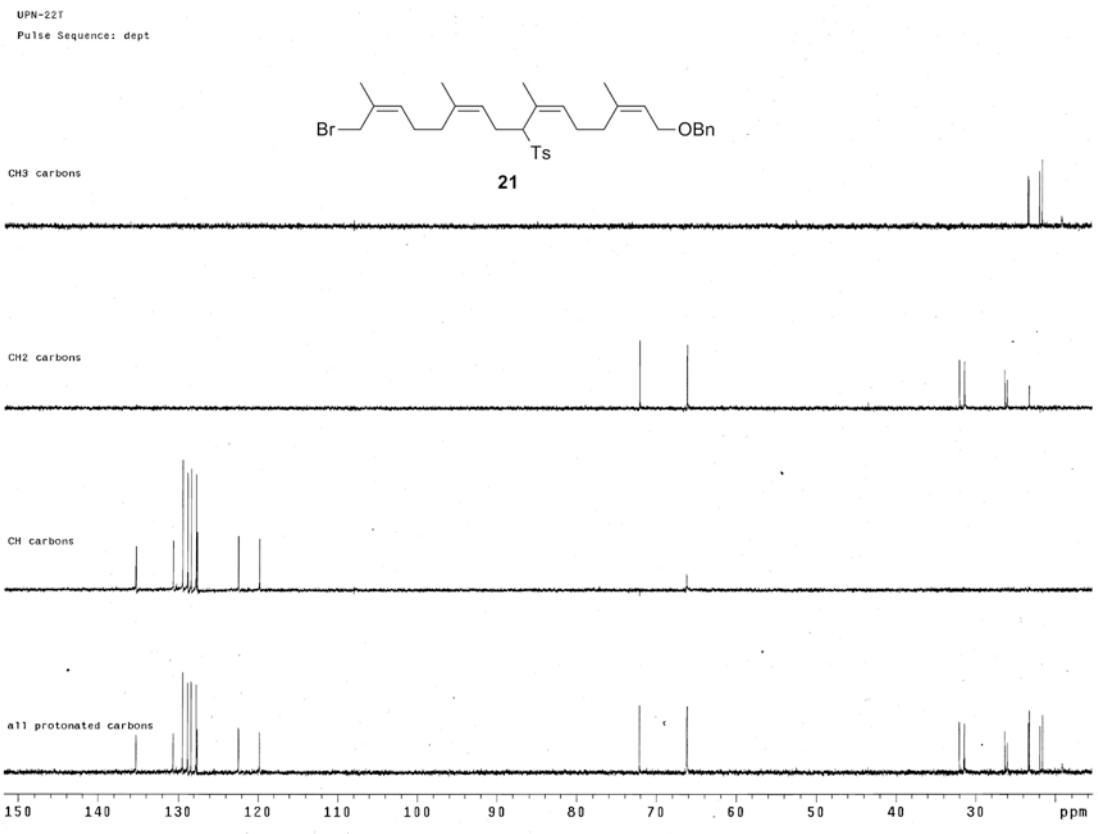
F2 - Processing parameters
SI 2048
SF 800.1800156 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

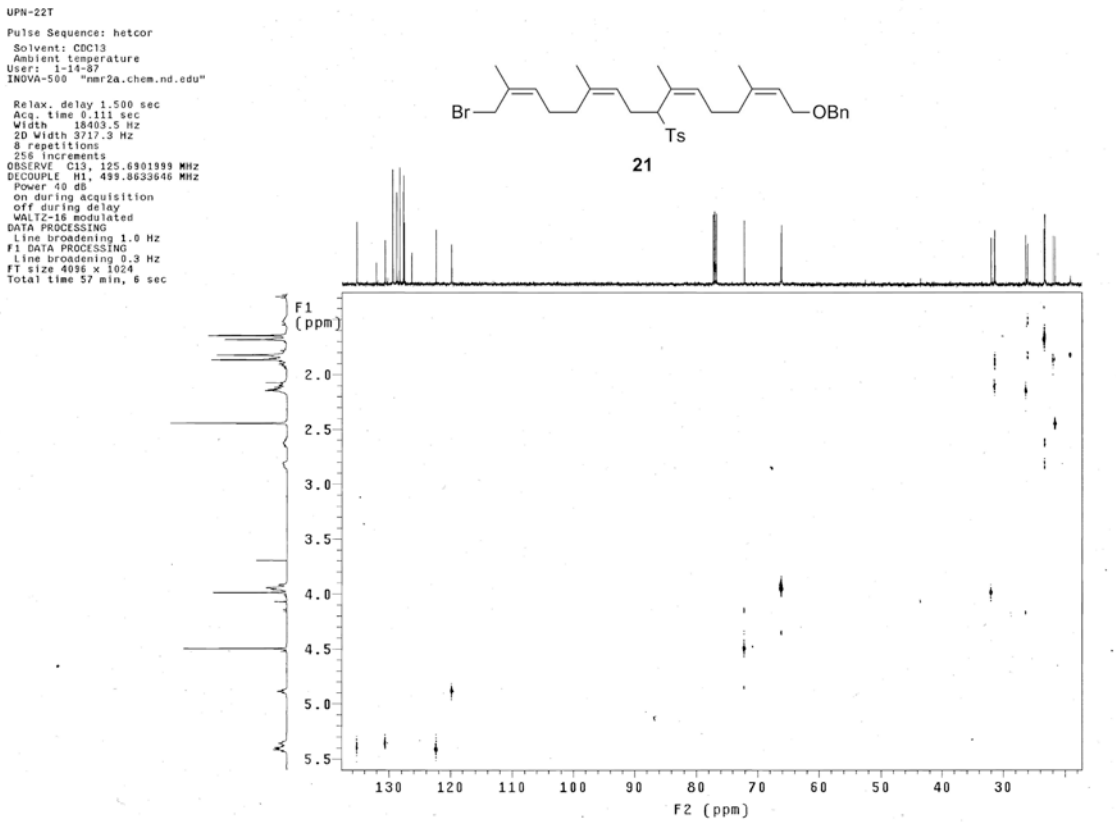
F1 - Processing parameters
SI 2048
MC2 QF
SF 201.2054221 MHz
WDW States-TPPI
SSB 0
LB 0 Hz
GB 0

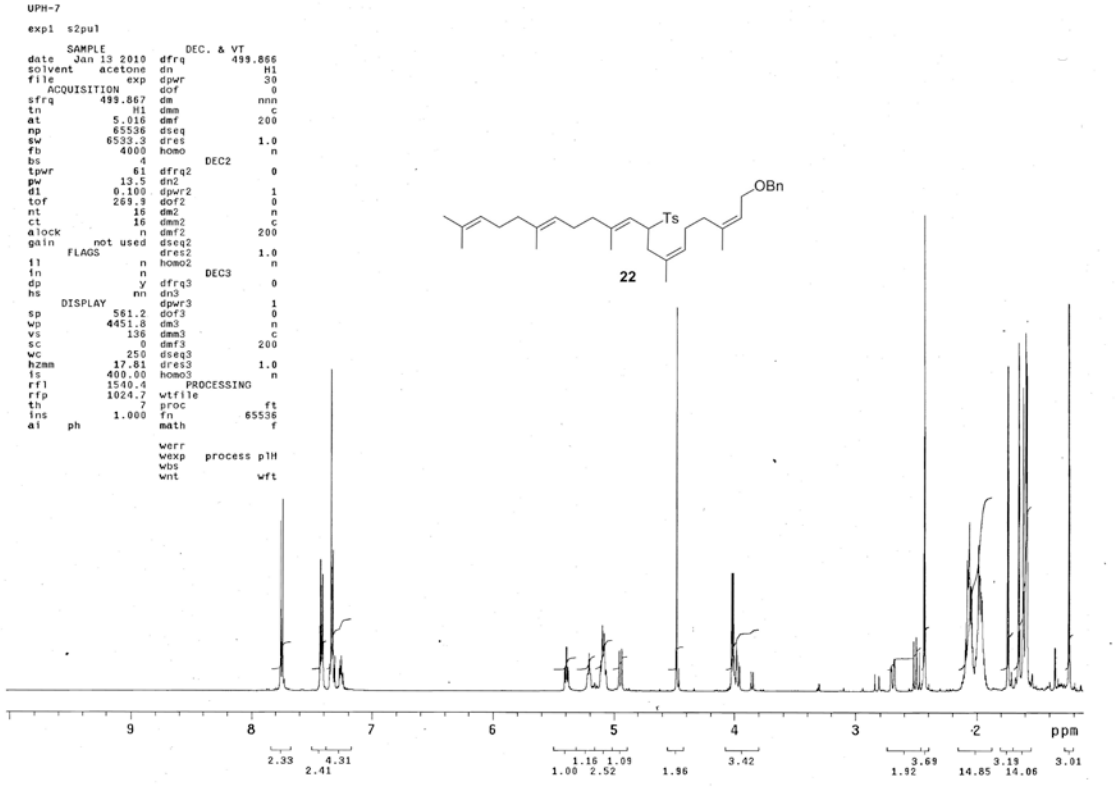

UPN-22T
exp3 s2pul
SAMPLE
date Dec 26 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 661.6
wp 4309.6
vs 113
sc 0
wc 250
hzmm 17.24
is 200.00
rfl 497.4
rfp 0
th 7
ins 3.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process p1H
wbs
wnt wft
Br
OBn
Ts
21
9
8
7
6
5
4
3
2
ppm
2.07
6.65
2.99
1.05
2.14
5.15
2.06
3.22
4.69
10.05
6.73
1.51

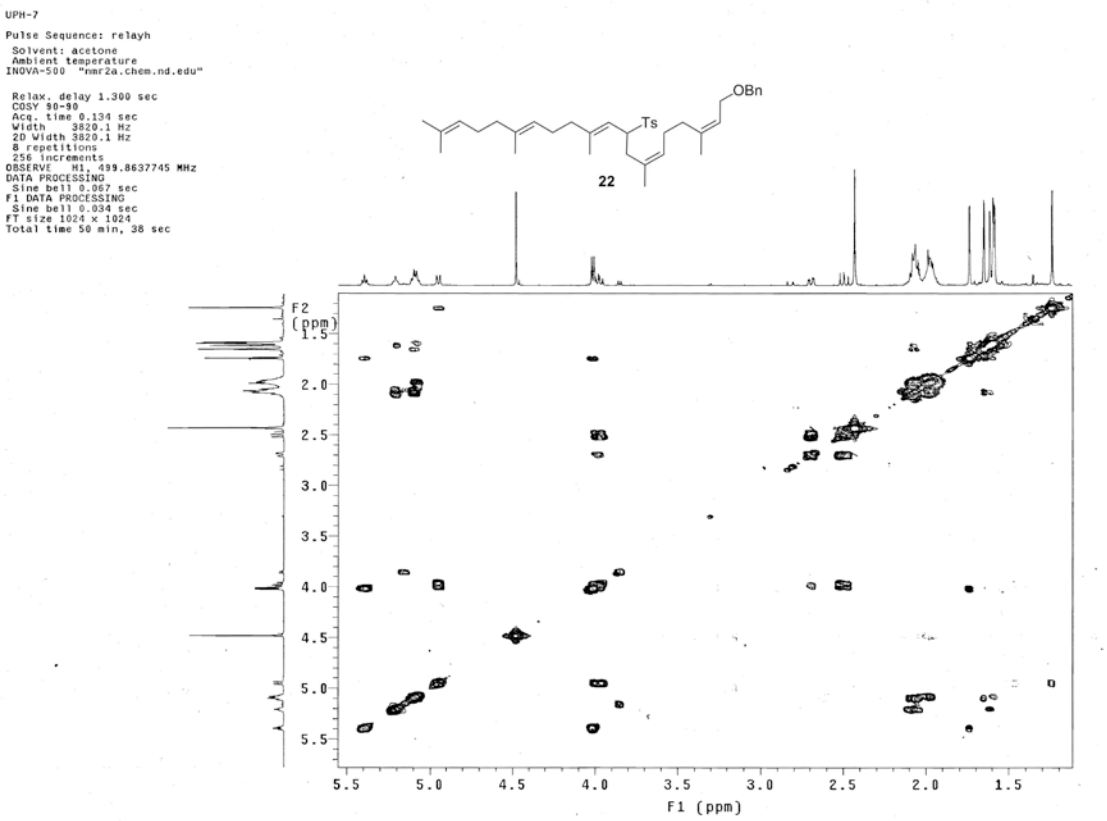

UPN-22T
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.139 sec
Width 3691.6 Hz
2D Width 3691.6 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611619 MHz
DATA PROCESSING
Sine bell 0.069 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 50 min, 50 sec
Br
OBn
Ts
21
F2 (ppm)
F1 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
1.5

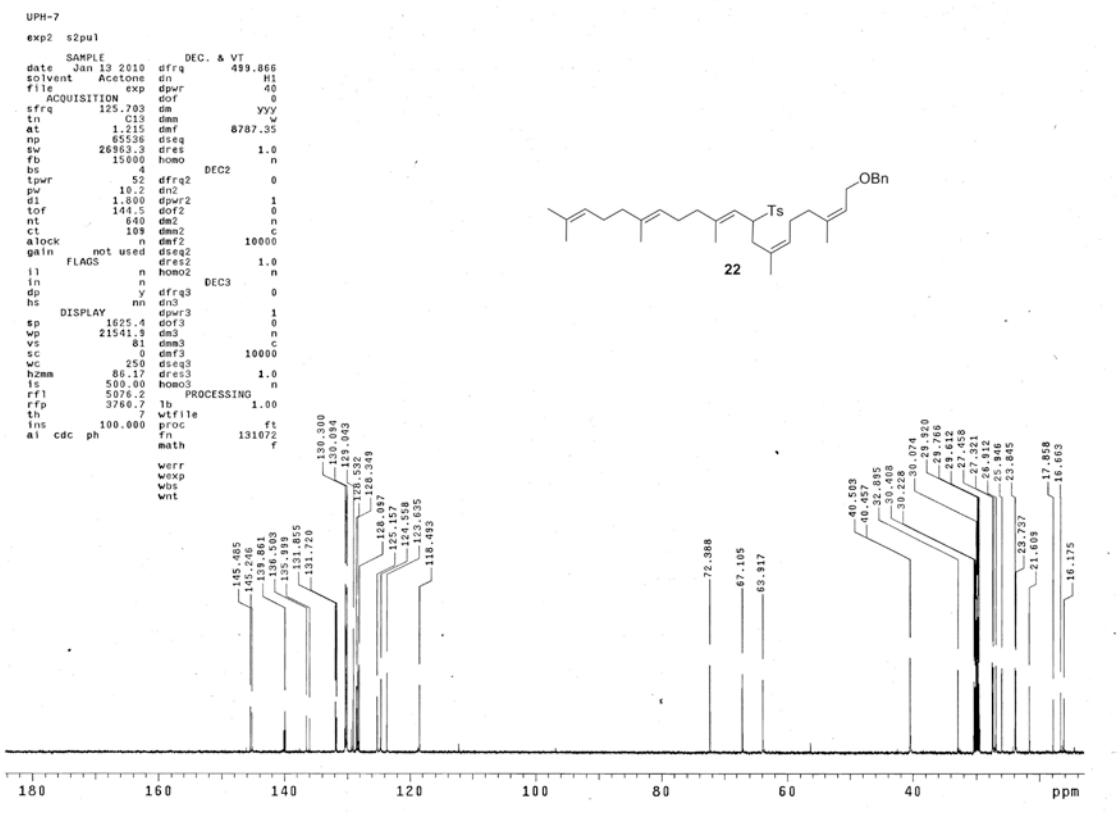

UPN-22T
exp2 s2pul
SAMPLE
date Dec 26 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26363.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 98
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 2391.0
wp 20478.8
vs 160
sc 0
wc 250
hzmm 81.92
is 500.00
rfl 11113.9
rfp 9678.1
th 5
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Br
OBn
Ts
21
144.369
139.373
138.339
137.747
135.268
132.074
130.643
129.442
128.791
128.376
127.681
127.540
126.359
122.437
119.822
77.252
76.997
76.745
72.139
66.244
66.185
32.011
31.438
31.363
26.312
26.001
23.343
23.242
21.906
21.579
160
140
120
100
80
60
40
ppm

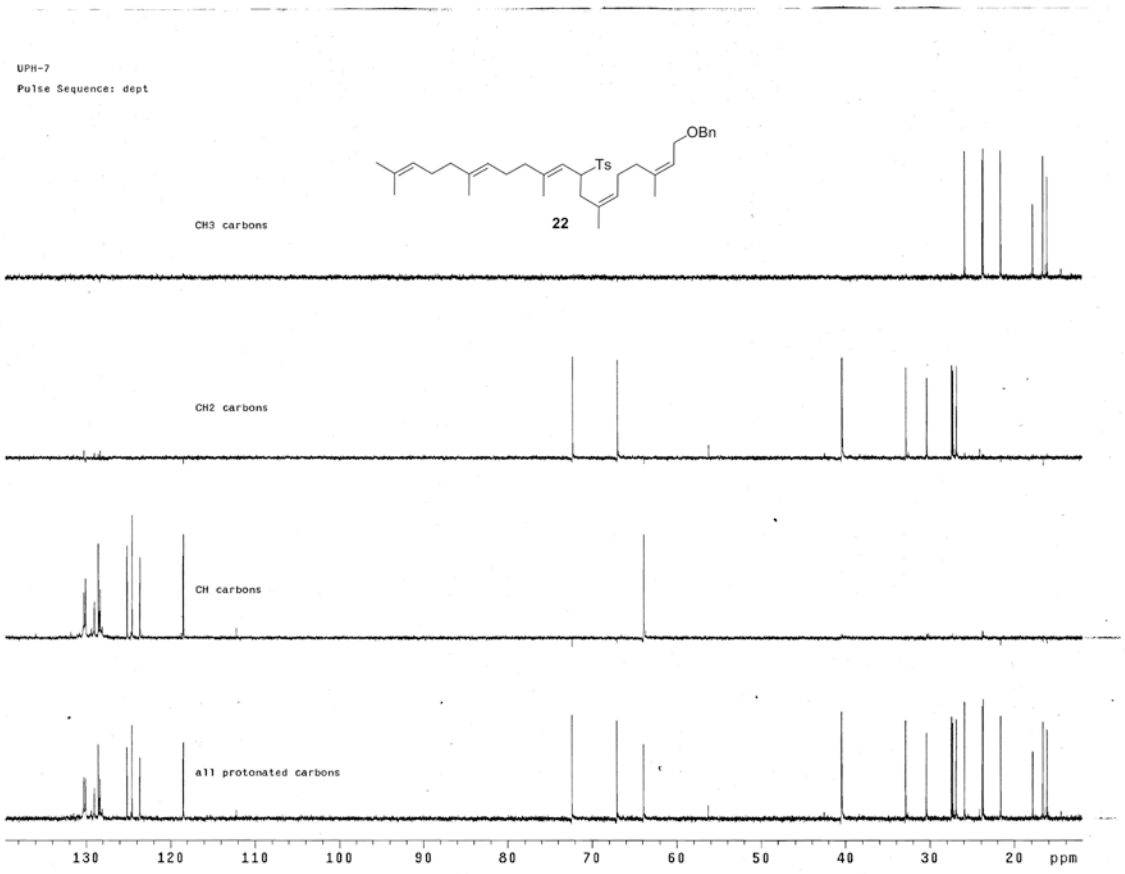

UPN-22T
Pulse Sequence: dept
Br
OBn
Ts
21
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
150
140
130
120
110
100
90
80
70
60
50
40
30
ppm

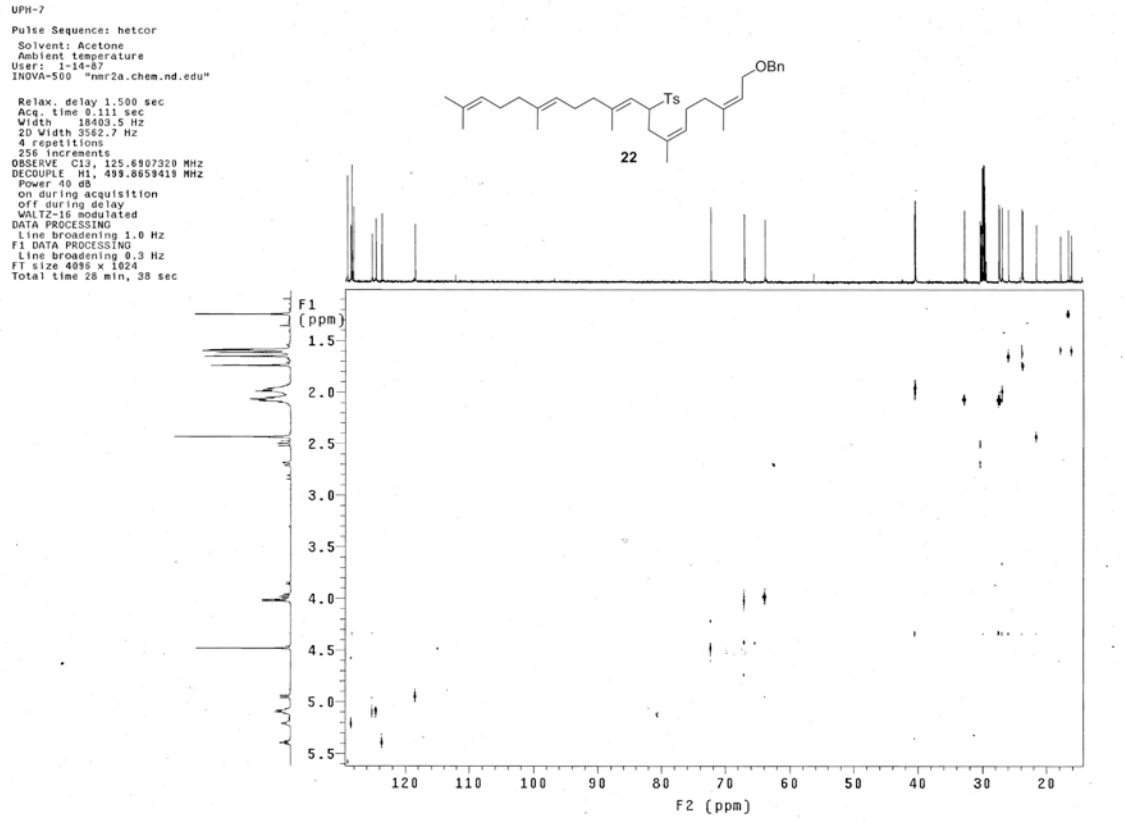

UPN-22T
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3717.3 Hz
8 repetitions
256 increments
OBSERVE C13, 125.6901999 MHz
DECOUPLE H1, 499.8633646 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 57 min, 6 sec
Br
OBn
Ts
21
F1 (ppm)
F2 (ppm)

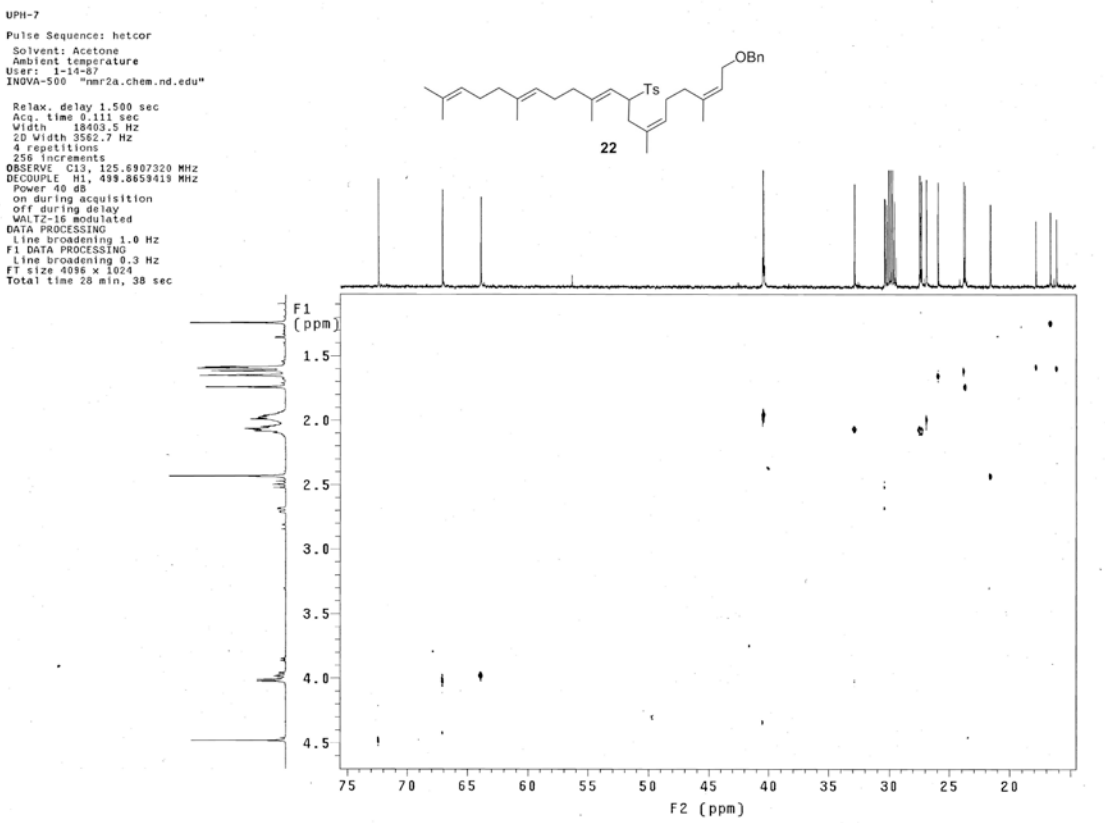

UPH-7
exp1 s2pul
SAMPLE
date Jan 13 2010
solvent acetone
file exp
ACQUISITION
sfrq 499.867
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 561.2
wp 4451.8
vs 136
sc 0
wc 250
hzmm 17.81
is 400.00
rfl 1540.4
rfp 1024.7
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
OBn
Ts
22
ppm
2.33
4.31
2.41
1.16
1.09
1.00
2.52
1.96
3.42
3.69
1.92
14.85
3.19
14.06
3.01

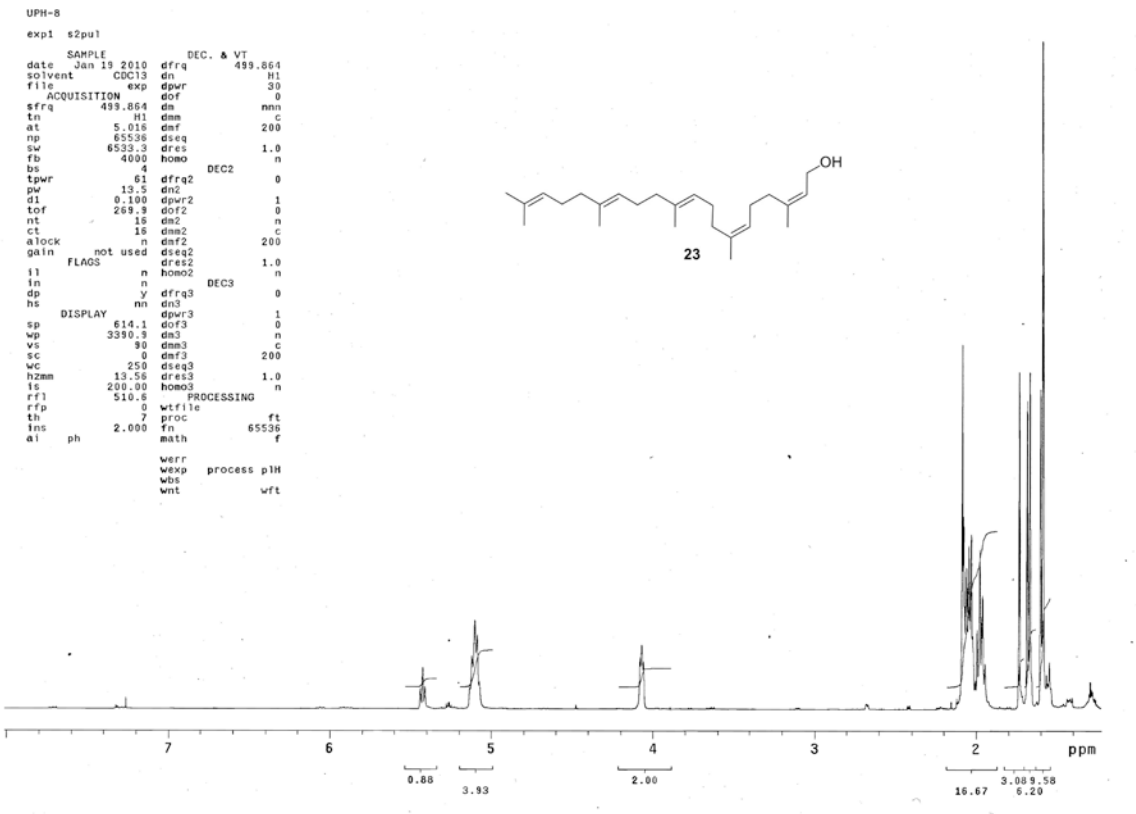

UPH-7
Pulse Sequence: relayh
Solvent: acetone
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.134 sec
Width 3820.1 Hz
2D Width 3820.1 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8637745 MHz
DATA PROCESSING
Sine bell 0.067 sec
F1 DATA PROCESSING
Sine bell 0.034 sec
FT size 1024 x 1024
Total time 50 min, 38 sec
OBn
Ts
22
F2 (ppm)
F1 (ppm)

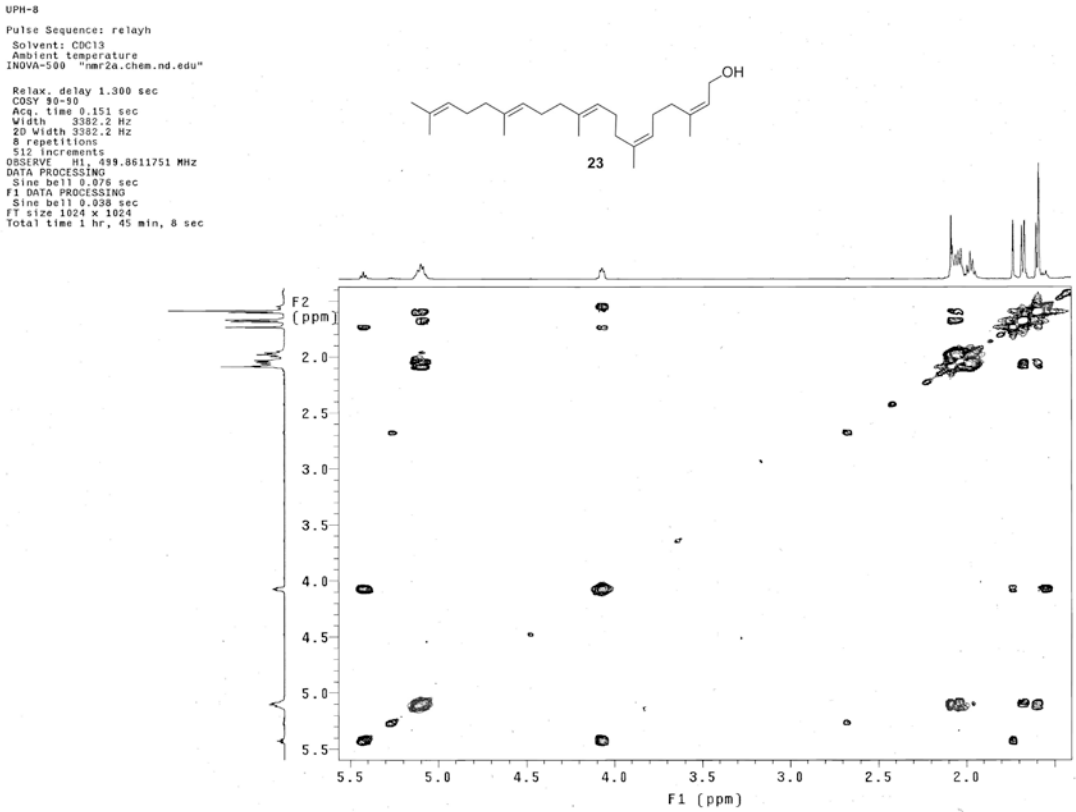

UPH-7
exp2 s2pul
SAMPLE
date Jan 13 2010
solvent Acetone
file exp
ACQUISITION
sfrq 125.703
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 108
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1625.4
wp 21541.9
vs 81
sc 0
wc 250
hzmm 86.17
is 500.00
rfl 5076.2
rfp 3760.7
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.866
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
OBn
Ts
22
145.485
145.246
139.861
136.503
135.999
131.855
131.720
130.300
130.044
129.043
128.532
128.349
128.097
125.157
124.558
123.635
118.493
72.388
67.105
63.917
40.503
40.457
40.411
32.895
30.408
30.228
30.074
29.920
29.766
29.613
27.459
27.321
26.912
25.946
23.845
23.737
21.603
17.858
16.663
16.175
180
160
140
120
100
80
60
40
ppm

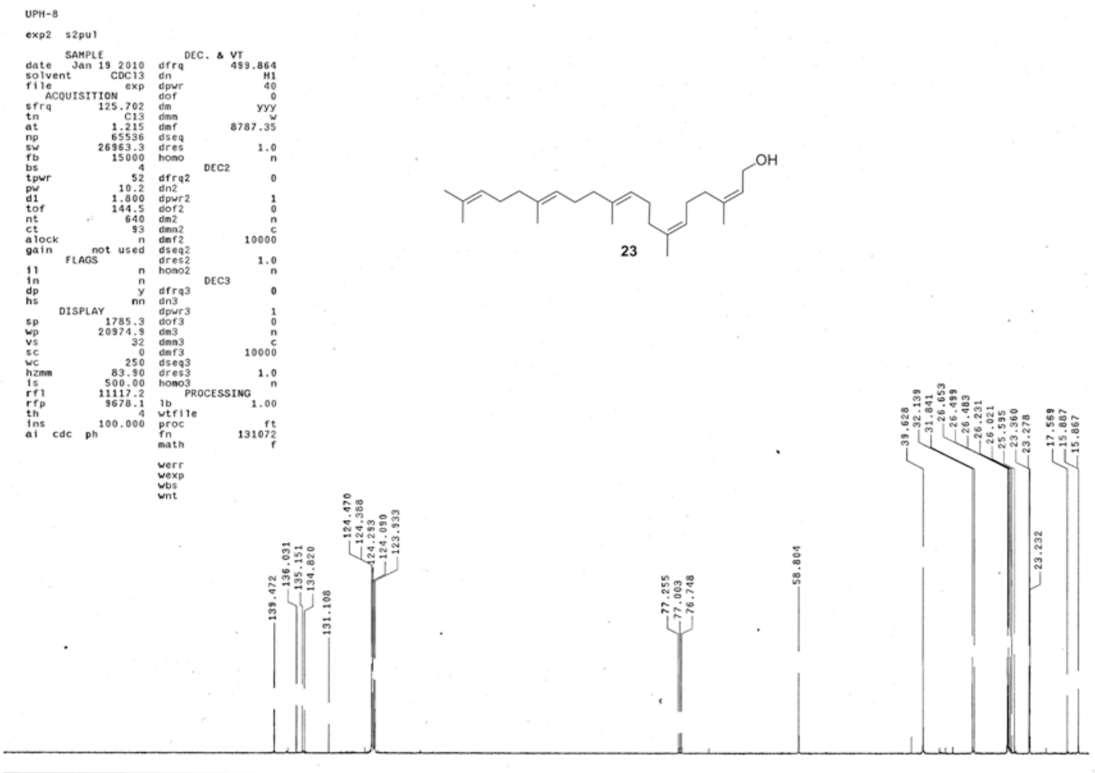

UPH-7
Pulse Sequence: dept
OBn
Ts
22
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
130
120
110
100
90
80
70
60
50
40
30
20
ppm

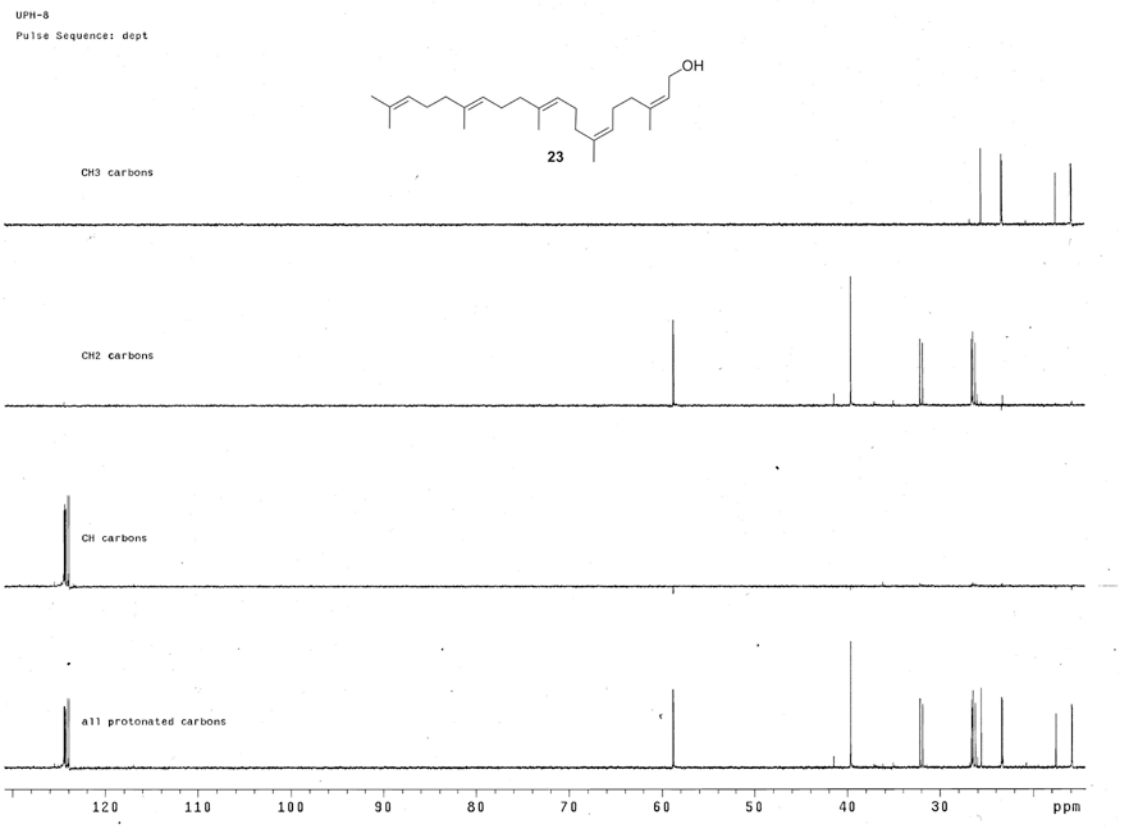

UPH-7
Pulse Sequence: hetcor
Solvent: Acetone
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3562.7 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6907320 MHz
DECOUPLE H1, 499.8659419 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 28 min, 38 sec
OBn
Ts
22
F1 (ppm)
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
120
110
100
90
80
70
60
50
40
30
20
F2 (ppm)

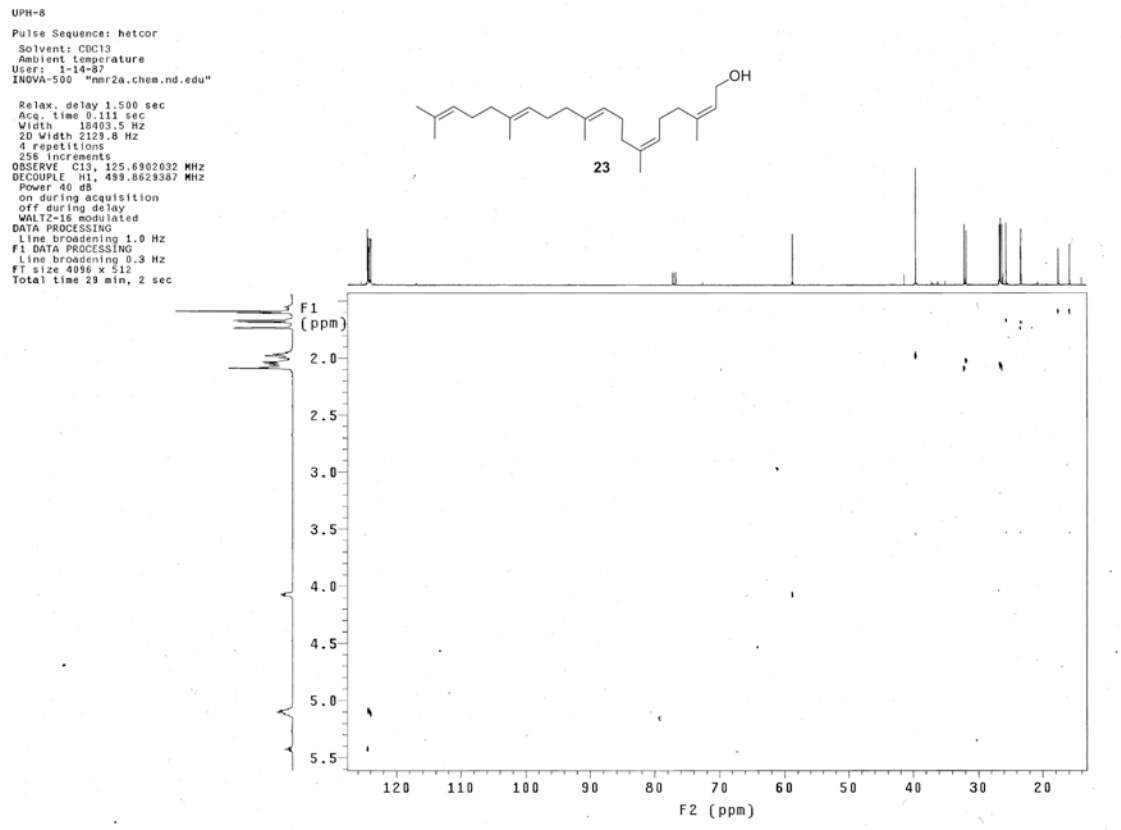

UPH-7
Pulse Sequence: hetcor
Solvent: Acetone
Ambient temperature
User: 1-14-67
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3562.7 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6907320 MHz
DECOUPLE H1, 499.8659419 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 28 min, 38 sec
OBn
Ts
22
F1 (ppm)
F2 (ppm)

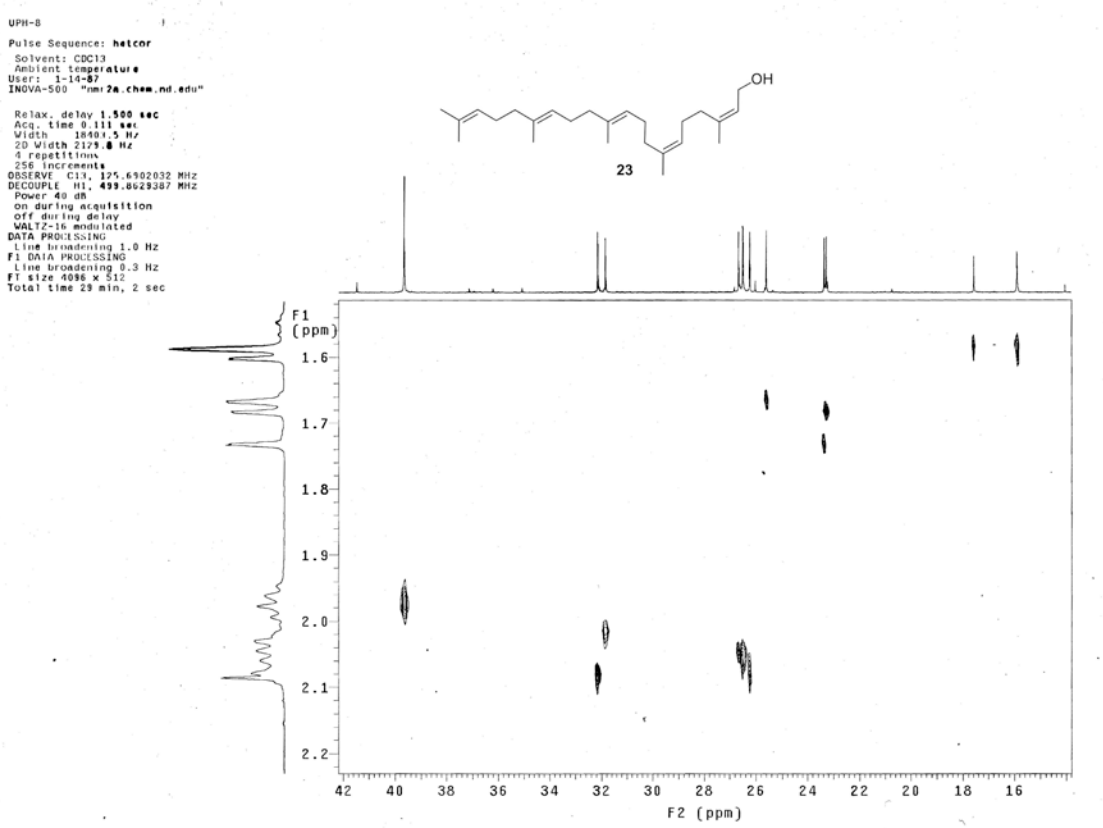

UPH-8
exp1 s2pul
SAMPLE
date Jan 19 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 614.1
wp 3390.9
vs 90
sc 0
wc 250
hzmm 13.56
is 200.00
rfl 510.6
rfp 0
th 7
ins 2.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
OH
23
ppm
0.88
3.93
2.00
16.67
3.08
9.58
6.20

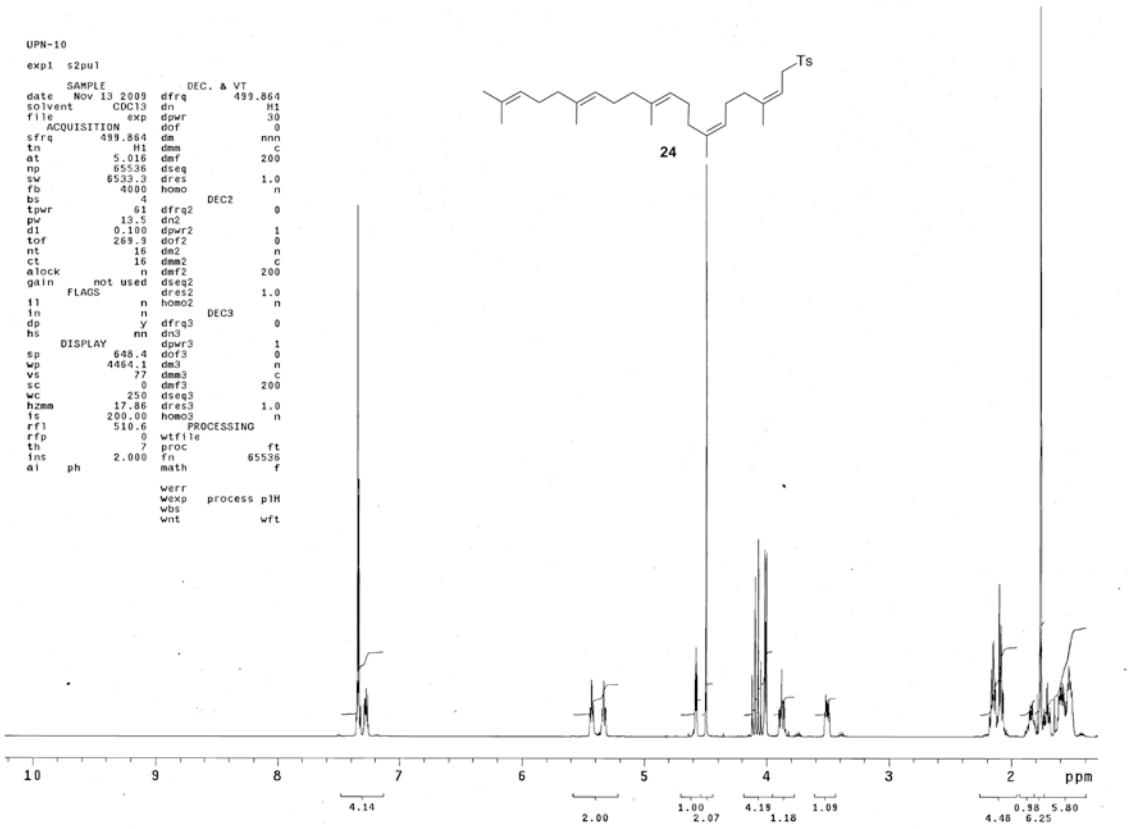

UPH-8
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.151 sec
Width 3382.2 Hz
2D Width 3382.2 Hz
8 repetitions
512 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.076 sec
F1 DATA PROCESSING
Sine bell 0.038 sec
FT size 1024 x 1024
Total time 1 hr, 45 min, 8 sec
OH
23
F2 (ppm)
F1 (ppm)

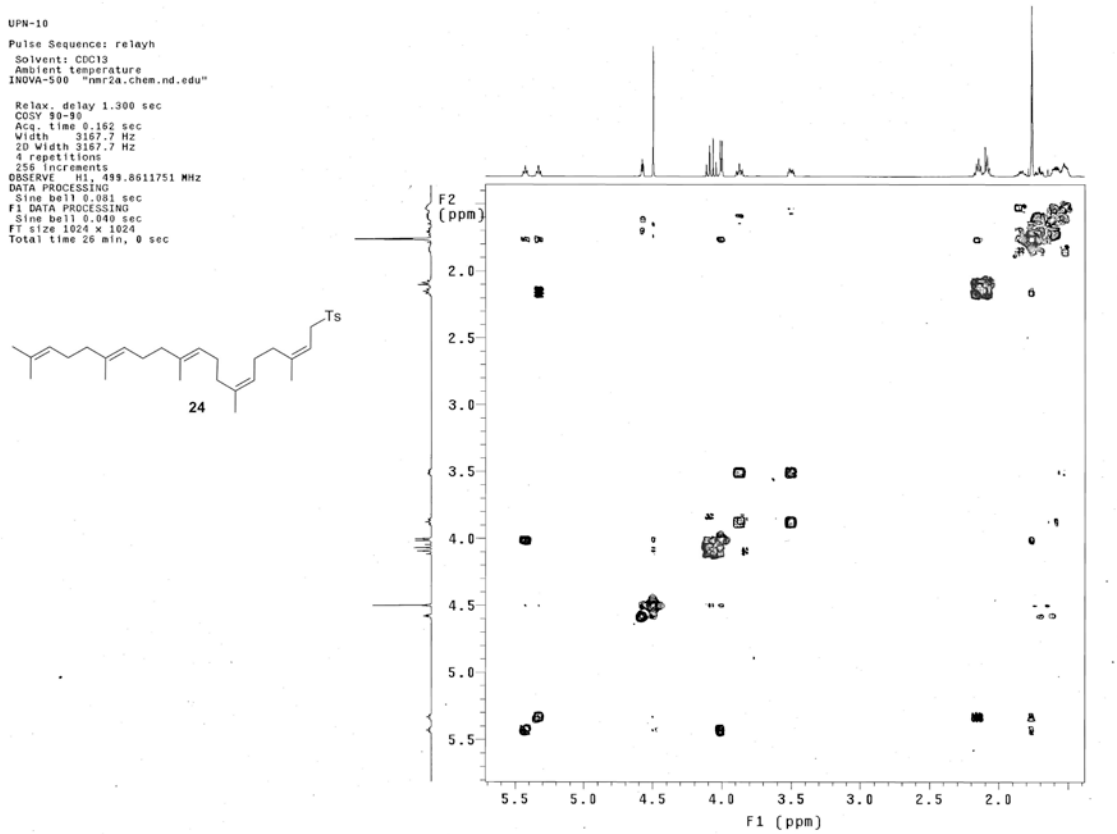

UPH-8
exp2 s2pul
SAMPLE
date Jan 19 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 93
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1785.3
wp 20974.9
vs 32
sc 0
wc 250
hzmm 83.90
is 500.00
rfl 11117.2
rfp 9678.1
th 4
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
OH
23
139.472
136.031
135.151
134.820
131.108
124.470
124.368
124.283
124.090
123.933
77.255
77.003
76.748
58.804
39.628
32.139
31.841
26.653
26.499
26.483
26.231
26.021
25.595
23.360
23.278
23.232
17.569
15.887
15.867
ppm

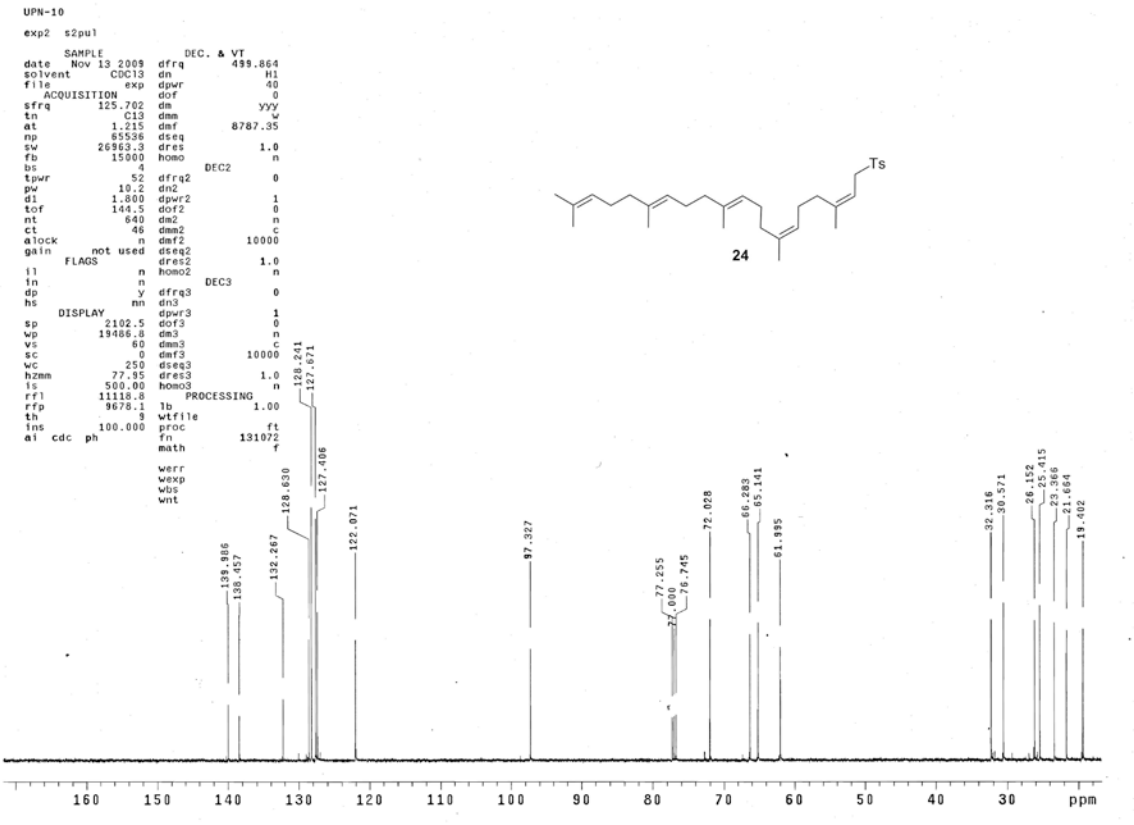

UPH-8
Pulse Sequence: dept
OH
23
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
120
110
100
90
80
70
60
50
40
30
ppm

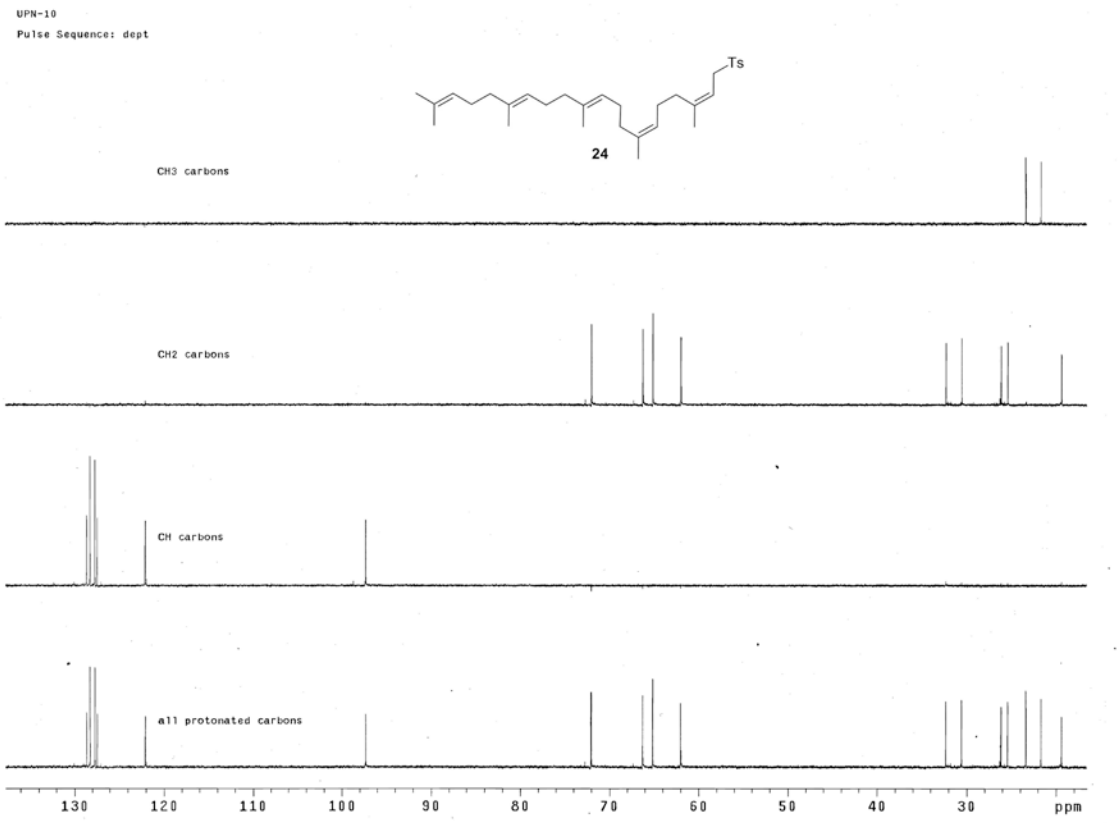

UPH-8
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2129.8 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6902032 MHz
DECOUPLE H1, 499.8629387 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 29 min, 2 sec
OH
23
F1
(ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
120
110
100
90
80
70
60
50
40
30
20
F2 (ppm)

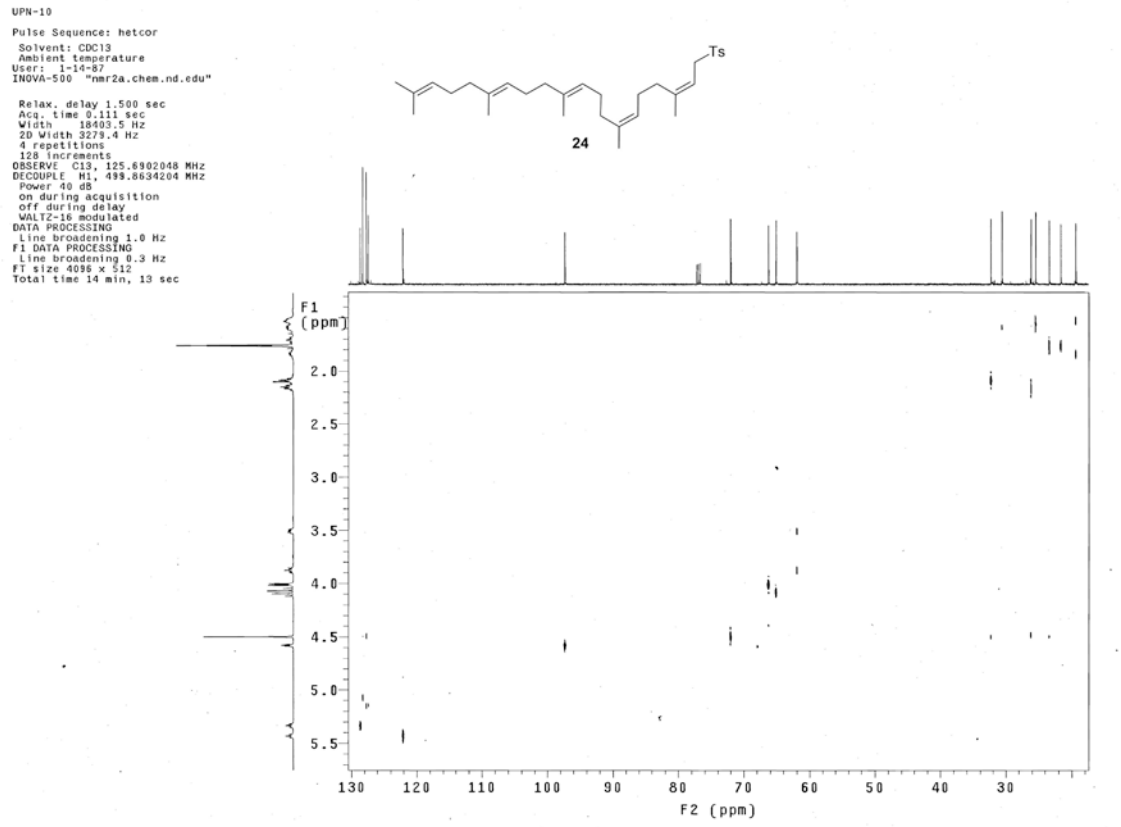

UPH-8
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18401.5 Hz
2D Width 2179.8 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6902032 MHz
DECOUPLE H1, 499.8629387 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 29 min, 2 sec
OH
23
F1 (ppm)
F2 (ppm)

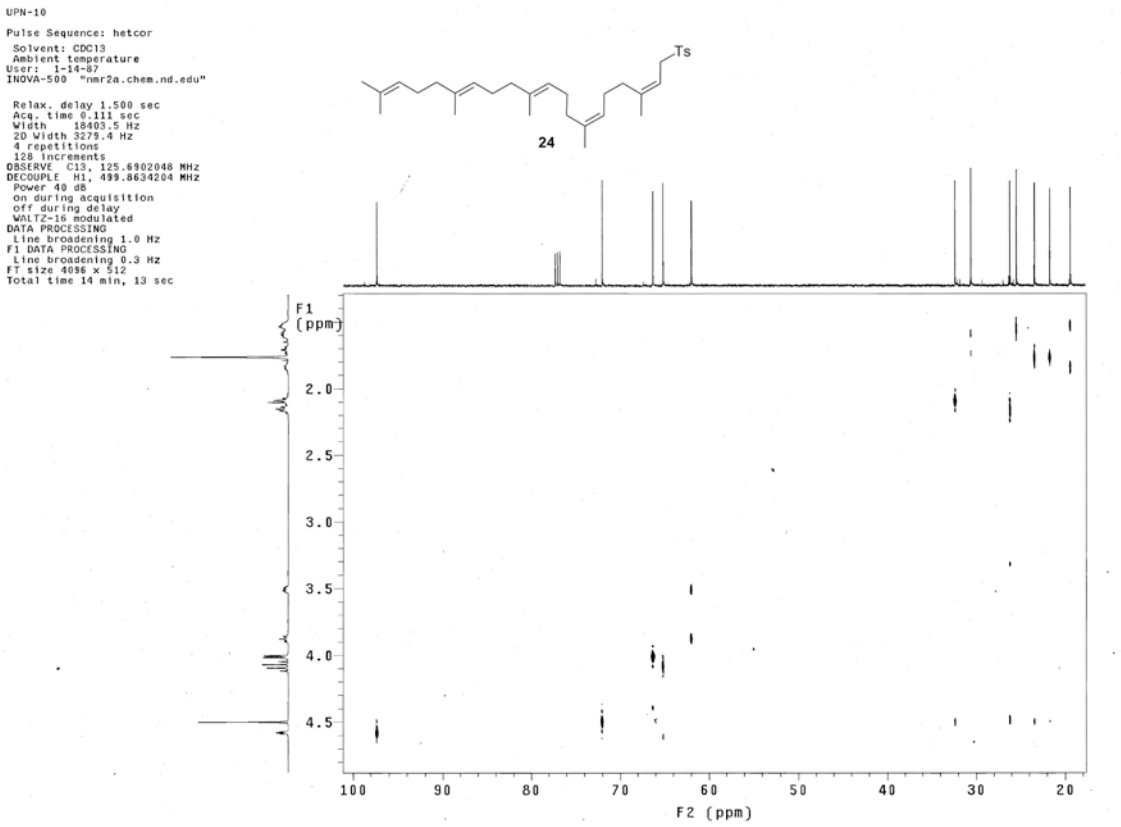

UPN-10
expl s2pul
SAMPLE
date Nov 13 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 648.4
wp 4464.1
vs 77
sc 0
wc 250
hzmm 17.86
is 200.00
rf1 510.6
rfp 0
th 7
ins 2.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process p1H
wbs
wnt wft
Ts
24
ppm
4.14
2.00
1.00
2.07
4.19
1.18
1.09
0.98
4.48
5.80
6.25

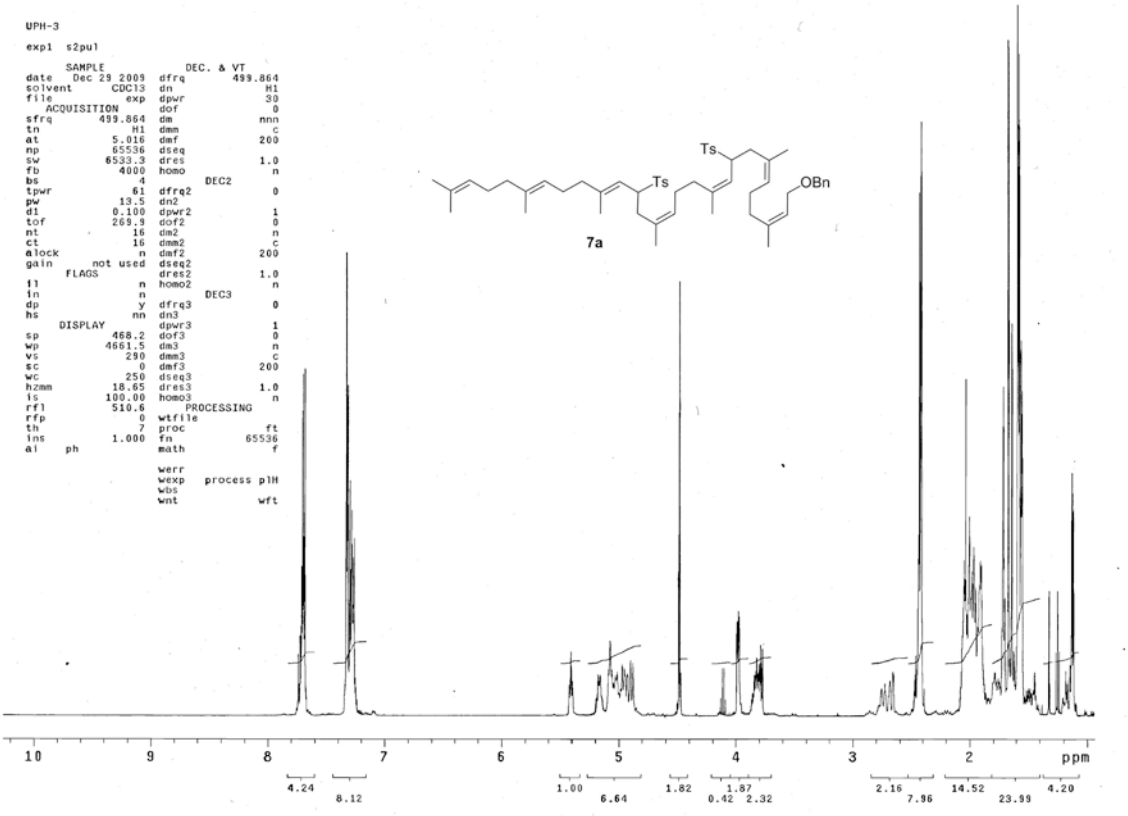

UPN-10
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.162 sec
Width 3167.7 Hz
2D Width 3167.7 Hz
4 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.081 sec
F1 DATA PROCESSING
Sine bell 0.040 sec
FT size 1024 x 1024
Total time 26 min, 0 sec
Ts
24
F2 (ppm)
F1 (ppm)

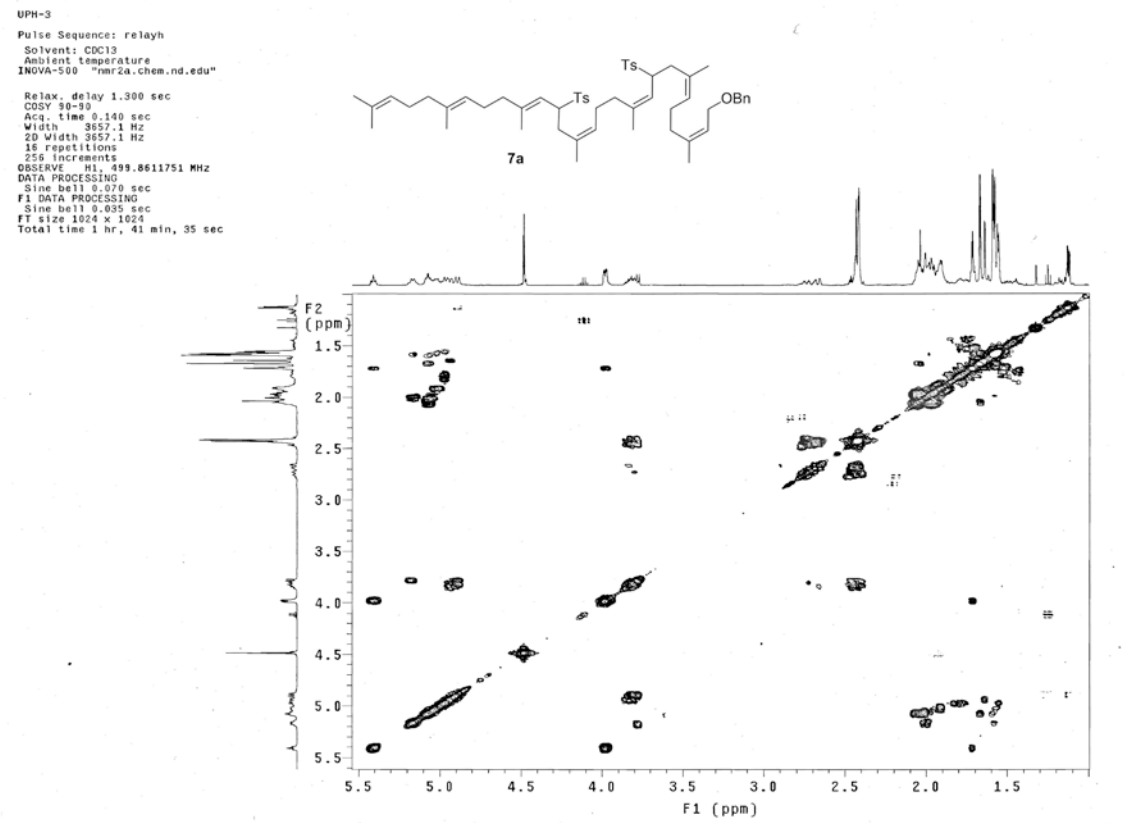

UPN-10
exp2 s2pul
SAMPLE
date Nov 13 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 46
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 2102.5
wp 19486.8
vs 60
sc 0
wc 250
hzmm 77.95
is 500.00
rfl 11118.8
rfp 9678.1
th 9
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Ts
24
139.986
138.457
132.267
128.630
128.241
127.671
127.406
122.071
97.327
77.255
77.000
76.745
72.028
66.283
65.141
61.995
32.316
30.571
26.152
25.415
23.366
21.664
19.402
ppm

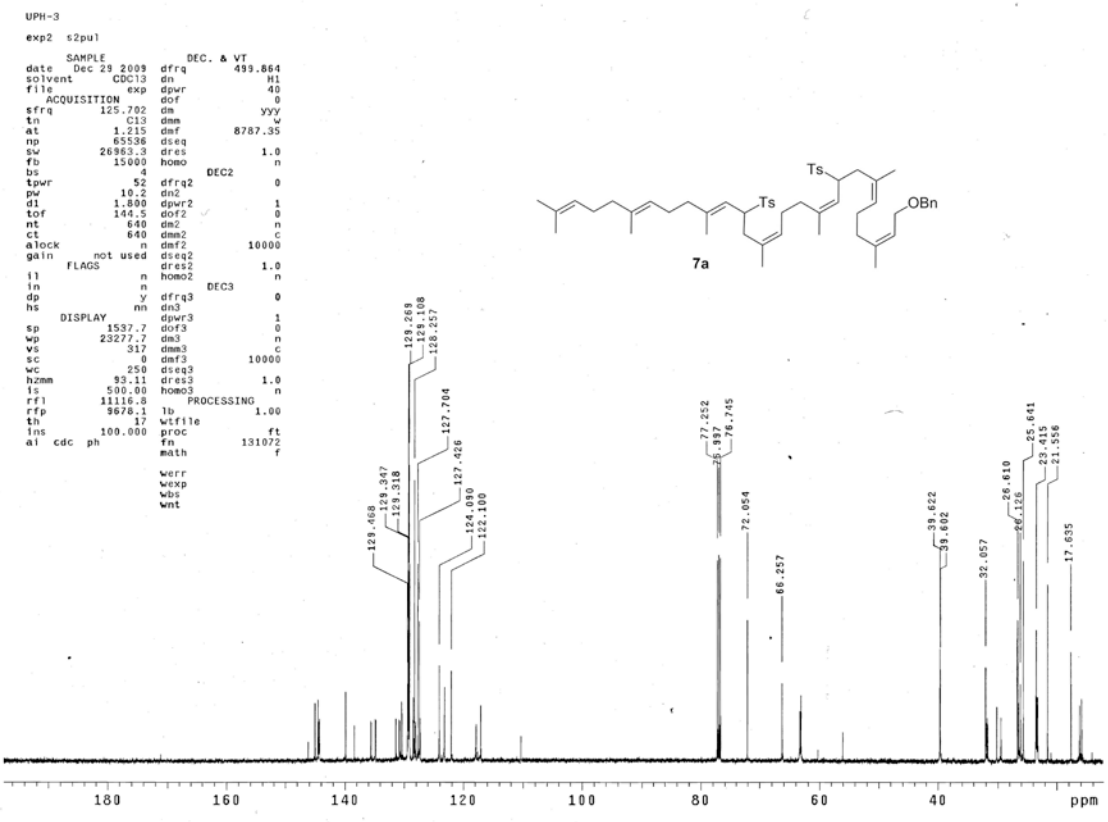

UPN-10
Pulse Sequence: dept
Ts
24
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
130
120
110
100
90
80
70
60
50
40
30
ppm

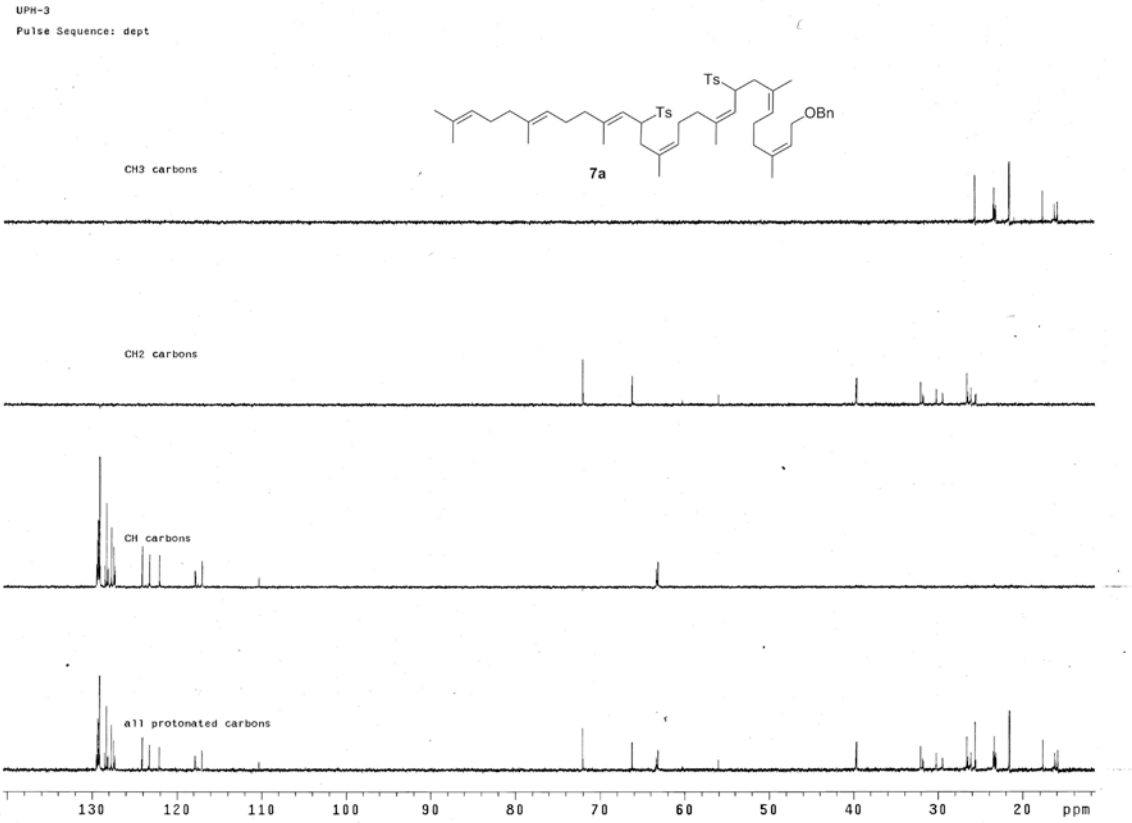

UPN-10
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3279.4 Hz
4 repetitions
128 increments
OBSERVE C13, 125.6902048 MHz
DECOUPLE H1, 499.8634204 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 14 min, 13 sec
Ts
24
F1 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
130
120
110
100
90
80
70
60
50
40
30
F2 (ppm)

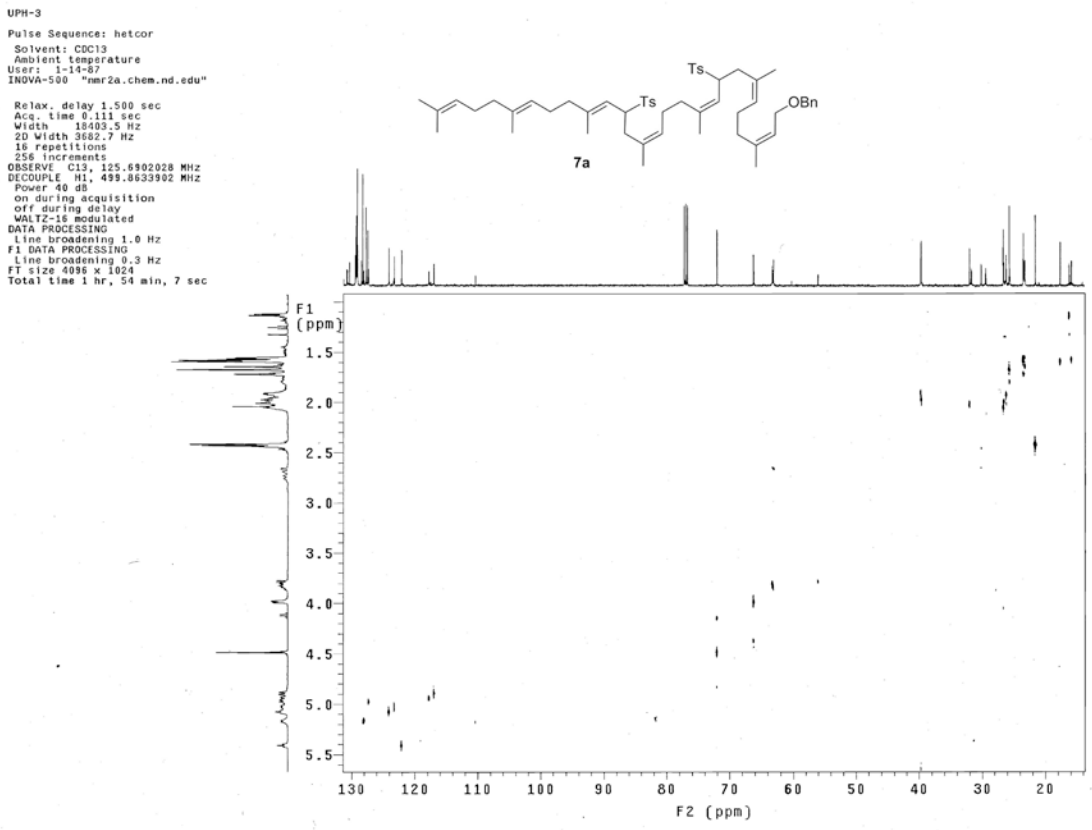

UPN-10
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3279.4 Hz
4 repetitions
128 increments
OBSERVE C13, 125.6902048 MHz
DECOUPLE H1, 499.8634204 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 14 min, 13 sec
Ts
24
F1 (ppm)
F2 (ppm)


UPH-3
exp1 s2pul
SAMPLE
date Dec 29 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 468.2
wp 4661.5
vs 290
sc 0
wc 250
hzmm 18.65
is 100.00
rfl 510.6
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
Ts
Ts
OBn
7a
ppm
4.24
8.12
1.00
6.64
1.82
1.87
0.42
2.32
2.16
7.96
14.52
23.99
4.20

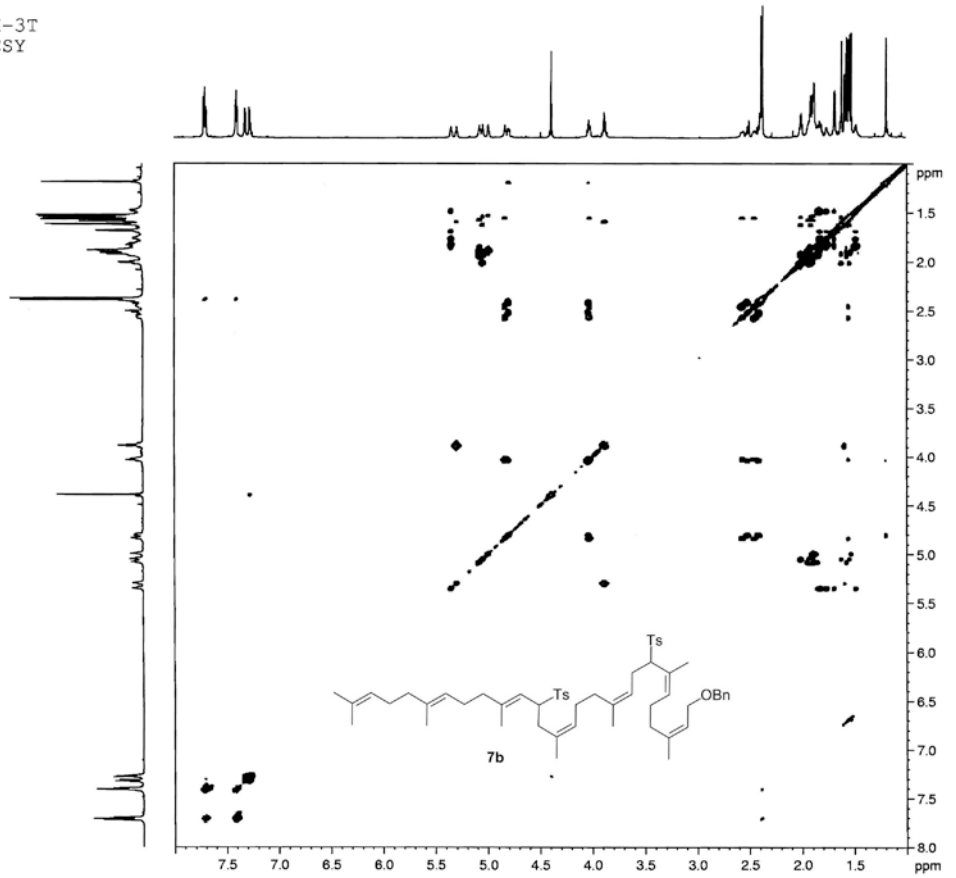

UPH-3
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.140 sec
Width 3657.1 Hz
2D Width 3657.1 Hz
16 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.070 sec
F1 DATA PROCESSING
Sine bell 0.035 sec
FT size 1024 x 1024
Total time 1 hr, 41 min, 35 sec
Ts
Ts
OBn
7a
F2 (ppm)
F1 (ppm)


UPH-3
exp2 s2pul
SAMPLE
date Dec 29 2009
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 640
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1537.7
wp 23277.2
vs 317
sc 0
wc 250
hzmm 93.11
is 500.00
rfl 11116.8
rfp 9678.1
th 17
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq n
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2 n
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3 n
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Ts
Ts
OBn
7a
129.468
129.347
129.318
129.269
129.108
128.257
127.704
127.426
124.090
122.100
77.252
76.997
76.745
72.054
66.257
39.622
39.602
32.057
26.610
26.126
25.641
23.415
21.556
17.635
ppm


UPH-3
Pulse Sequence: dept
Ts
Ts
OBn
7a
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
130
120
110
100
90
80
70
60
50
40
30
20
ppm


UPH-3
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-07
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3682.7 Hz
16 repetitions
256 increments
OBSERVE C13, 125.6902028 MHz
DECOUPLE H1, 499.8633902 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 1024
Total time 1 hr, 54 min, 7 sec
Ts
Ts
OBn
7a
F1 (ppm)
1.5
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
130
120
110
100
90
80
70
60
50
40
30
20
F2 (ppm)

UPH-3T
TOCSY




```
Current Data Parameters
NAME                  UPH-3T
EXPNO                      3
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100210
Time                   1.49
INSTRUM                spect
PROBHD   5 mm CPTCI 1H-
PULPROG    dipsi2etgpsi
TD                      1024
SOLVENT                 DMSO
NS                        16
DS                        16
SWH                8223.685 Hz
FIDRES             8.030942 Hz
AQ                 0.0623092 sec
RG                      14.2
DW                    60.800 usec
DE                    30.00 usec
TE                    301.9 K
d0                 0.00000300 sec
D1                 1.39999998 sec
d11                0.03000000 sec
D16                0.00020000 sec
D20                0.00001000 sec
D21                0.00001000 sec
D9                 0.06000000 sec
DELTA              0.00120300 sec
DELTA1             0.00120800 sec
FACTOR1                   11
IN0                0.00012160 sec
l1                        22
STICNT                     0

======== CHANNEL f1 ========
NUC1                      1H
P1                      9.30 usec
p2                     18.60 usec
P6                     24.00 usec
PL1                     0.20 dB
PL10                    8.43 dB
SFO1           800.1837640 MHz

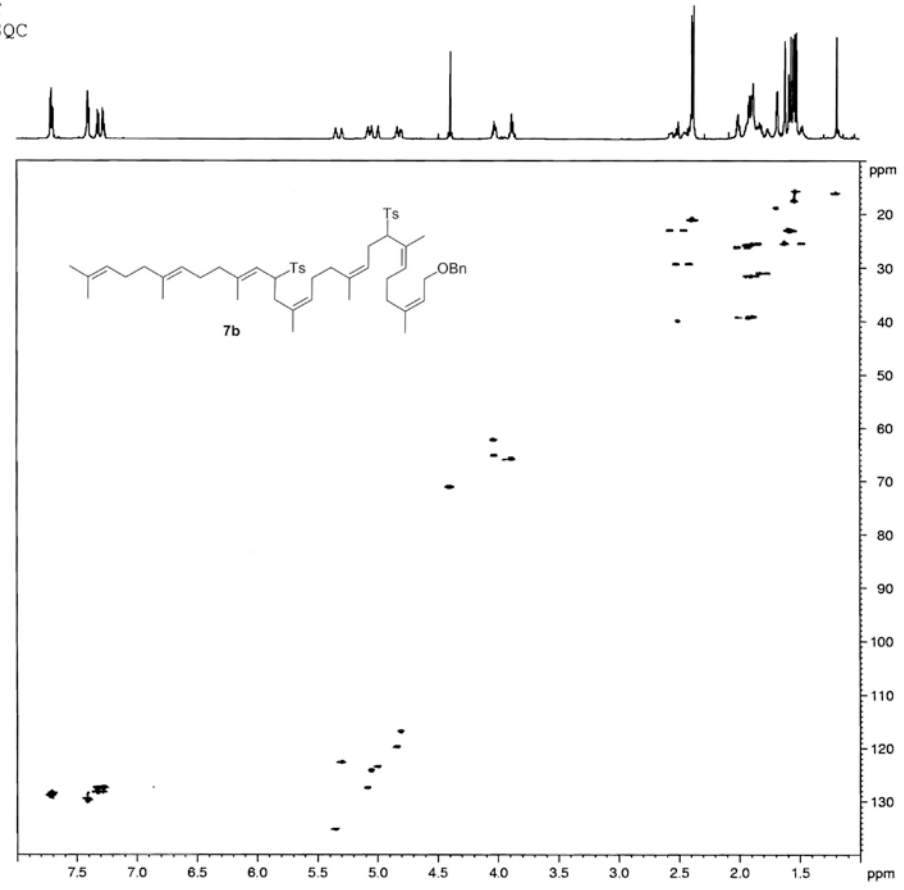
====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPZ1                  30.00 %
GPZ2                  30.00 %
P16                 1000.00 usec

F1 - Acquisition parameters
TD                      1024
SFO1            800.1838 MHz
FIDRES             8.030942 Hz
SW                   16.277 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                      2048
SF             800.1800073 MHz
WDW                    QSINE
SSB                        2
LB          0 Hz
GB          0
PC                      1.40

F1 - Processing parameters
SI                      2048
MC2          echo-antiecho
SF              800.1800072 MHz
WDW             States-TPPI
SSB                        2
LB          0 Hz
GB          0
```


UPH-3T
13C-HSQC




```
Current Data Parameters
NAME                  UPH-3T
EXPNO                      6
PROCNO                     1

F2 - Acquisition Parameters
Date_              20100210
Time                   9.06
INSTRUM                spect
PROBHD   5 mm CPTCI 1H-
PULPROG  hsqcedetgpsisp2.2
TD                      1024
SOLVENT                 DMSO
NS                        32
DS                        96
SWH                8223.685 Hz
FIDRES             8.030942 Hz
AQ                 0.0623092 sec
RG                      2050
DW                    60.800 usec
DE                    30.00 usec
TE                    301.9 K
CNST17             -0.5000000
CNST2             145.0000000
d0                 0.00000300 sec
D1                 1.39999998 sec
d11                0.03000000 sec
D16                0.00020000 sec
D21                0.00345000 sec
D24                0.00086207 sec
d6                 0.00172414 sec
DELTA              0.00272540 sec
DELTA1             0.00120800 sec
DELTA2             0.00143414 sec
DELTA3             0.00064207 sec
DELTA4             0.00027414 sec
DELTA5             0.00391000 sec
IN0                0.00001655 sec
STICNT                     0

======== CHANNEL f1 ========
NUC1                      1H
P1                      9.30 usec
p2                     18.60 usec
P28                  1000.00 usec
PL1                     0.20 dB
SFO1           800.1837640 MHz

======== CHANNEL f2 ========
CPDPRG2            p5m4sp180
NUC2                      13C
P14                   500.00 usec
P24                  2000.00 usec
P3                     14.80 usec
PCPD2                1500.00 usec
PL0                   120.00 dB
PL12                   10.00 dB
PL2                    -1.00 dB
SFO2           201.2205114 MHz
SF3                    1.08 dB
SF7                    1.08 dB
SPNAM15  Cxp,80,1.5,10.nd
SPNAM3   Cxp80,0.5,20.1
SPNAM7      Cxp80comp.4
SPOAL15               0.500
SPOAL3                0.500
SPOAL7                0.500
SPOFFS15 0 Hz
SPOFFS3  0 Hz
SPOFFS7  0 Hz

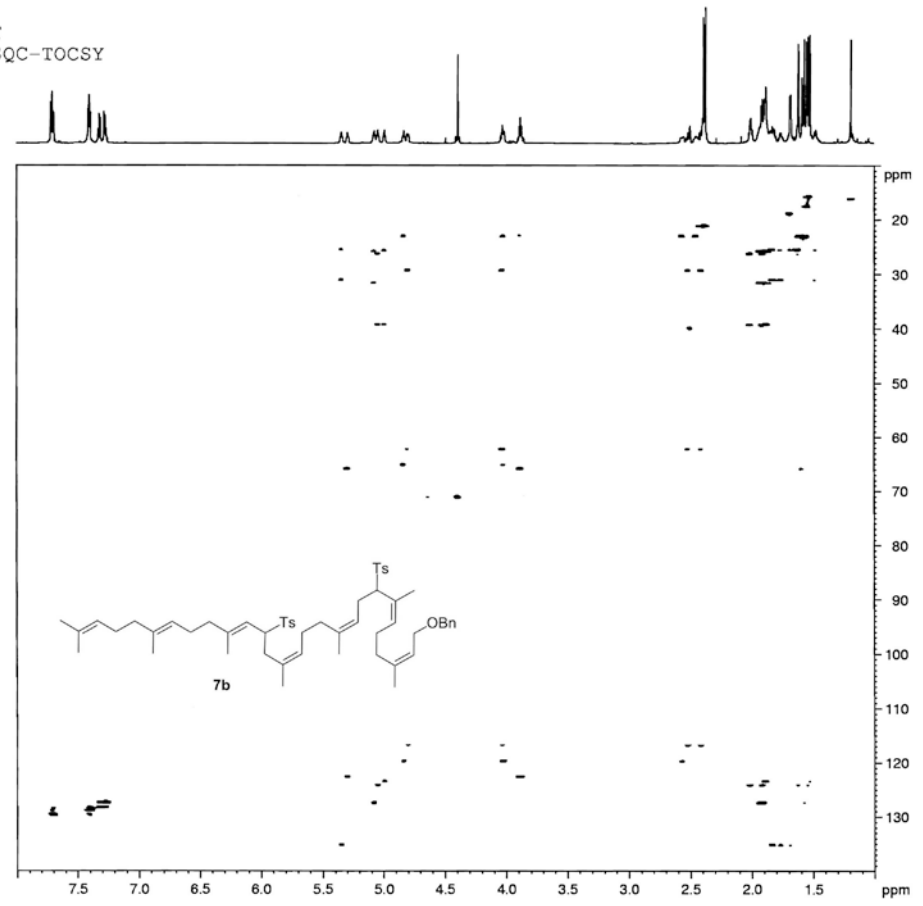
====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPNAM3              SINE.100
GPNAM4              SINE.100
GPZ1                  80.00 %
GPZ2                  25.13 %
GPZ3                  11.00 %
GPZ4                  -5.00 %
P16                 1000.00 usec
P19                  500.00 usec

F1 - Acquisition parameters
TD                      1024
SFO1           201.2205 MHz
FIDRES            29.503399 Hz
SW                   150.141 ppm
FnMODE     Echo-Antiecho

F2 - Processing parameters
SI                      2048
SF             800.1800054 MHz
WDW                    QSINE
SSB                        3
LB          0 Hz
GB          0
PC                      1.40

F1 - Processing parameters
SI                      2048
MC2          echo-antiecho
SF             201.2055121 MHz
WDW             States-TPPI
SSB                        3
LB          0 Hz
GB          0
```

UPH-3T
13C-HSQC-TOCSY



Current Data Parameters
NAME UPH-3T
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100210
Time 22.46
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hsqcdietgpsisp.2
TD 1024
SOLVENT DMSO
NS 32
DS 16
SWH 8223.685 Hz
FIDRES 8.030942 Hz
AQ 0.6623092 sec
RG 1620
DW 60.800 usec
DE 30.00 usec
TE 301.9 K
CNST17 -0.5000000
CNST2 145.0000000
d0 0.00000300 sec
D1 1.50000000 sec
d11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
d4 0.00172414 sec
D9 0.04000000 sec
DELTA 0.00122040 sec
DELTA1 0.00120800 sec
DELTA2 0.00147014 sec
DELTA3 0.00135800 sec
FACTOR1 11
IN0 0.00001655 sec
l1 22
ST1CNT 0

======== CHANNEL f1 ========
NUC1 1H
P1 9.30 usec
p2 18.40 usec
P28 1000.00 usec
P6 24.00 usec
PL1 0.20 dB
PL10 8.43 dB
SFO1 800.1837640 MHz

======== CHANNEL f2 ========
CPDPRG2 p5etap180
NUC2 13C
P14 500.00 usec
P24 2000.00 usec
P3 14.80 usec
PCPD2 1300.00 usec
PL0 120.00 dB
PL12 10.00 dB
PL2 -2.00 dB
SFO2 201.2205114 MHz
SP3 1.00 dB
SP7 1.00 dB
SPNAM15 Crp,40,1.5,10.nd
SPNAM3 Crp80,0.5,80.1
SPNAM7 Crp80comp.4
SPOAL15 0.500
SPOAL3 0.500
SPOAL7 0.500
SPOFFS15 0 Hz
SPOFFS3 0 Hz
SPOFFS7 0 Hz

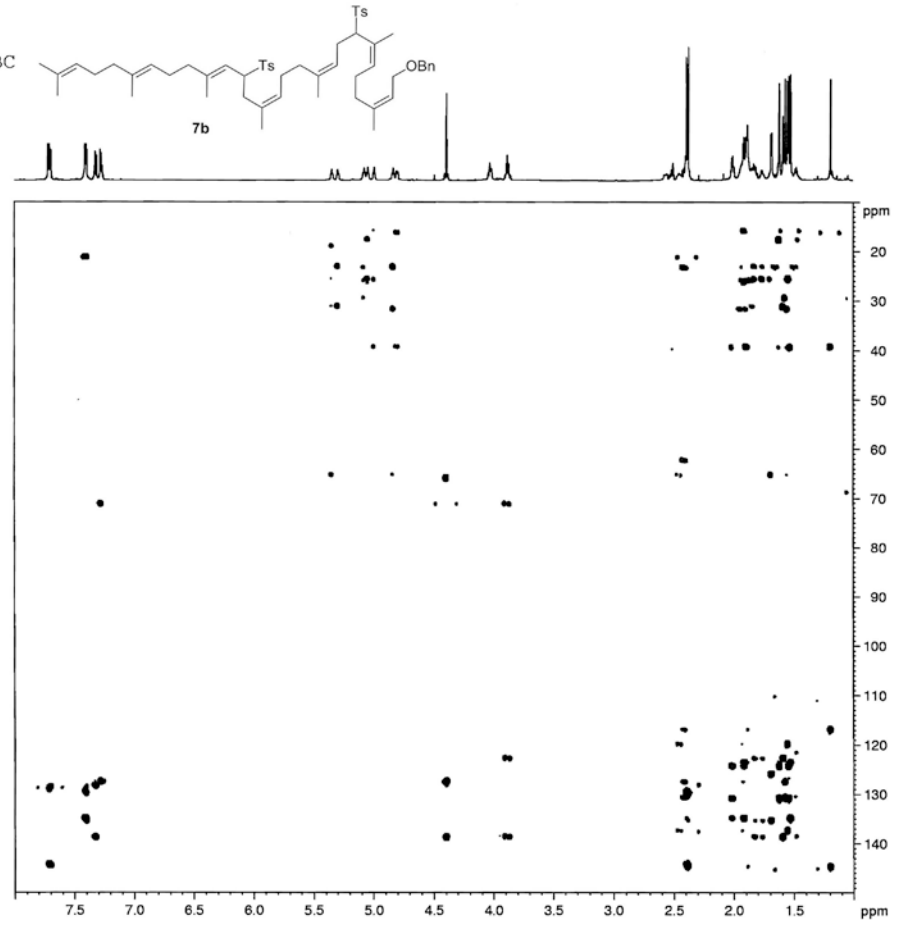
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 1024
SFO1 201.2205 MHz
FIDRES 29.503399 Hz
SW 150.141 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 800.1800058 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 2048
MC2 echo-antiecho
SF 201.2055122 MHz
WDW States-TPPI
SSB 2
LB 0 Hz
GB 0

UPH-3T
13C-HMBC



Current Data Parameters
NAME UPH-3T
EXPNO 6
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100211
Time 13.54
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT DMSO
NS 80
DS 128
SWH 8223.685 Hz
FIDRES 4.015471 Hz
AQ 0.1245684 sec
RG 3250
DW 60.800 usec
DE 30.00 usec
TE 301.9 K
CNST13 8.0000000
CNST6 125.0000000
CNST7 175.0000000
d0 0.00000300 sec
D1 1.39999998 sec
D16 0.00020000 sec
d6 0.06250000 sec
DELTA1 0.00280000 sec
DELTA2 0.00165714 sec
DELTA3 0.06129600 sec
IN0 0.00001310 sec

======== CHANNEL f1 ========
NUC1 1H
P1 9.30 usec
p2 18.60 usec
PL1 0.20 dB
SFO1 800.1837640 MHz

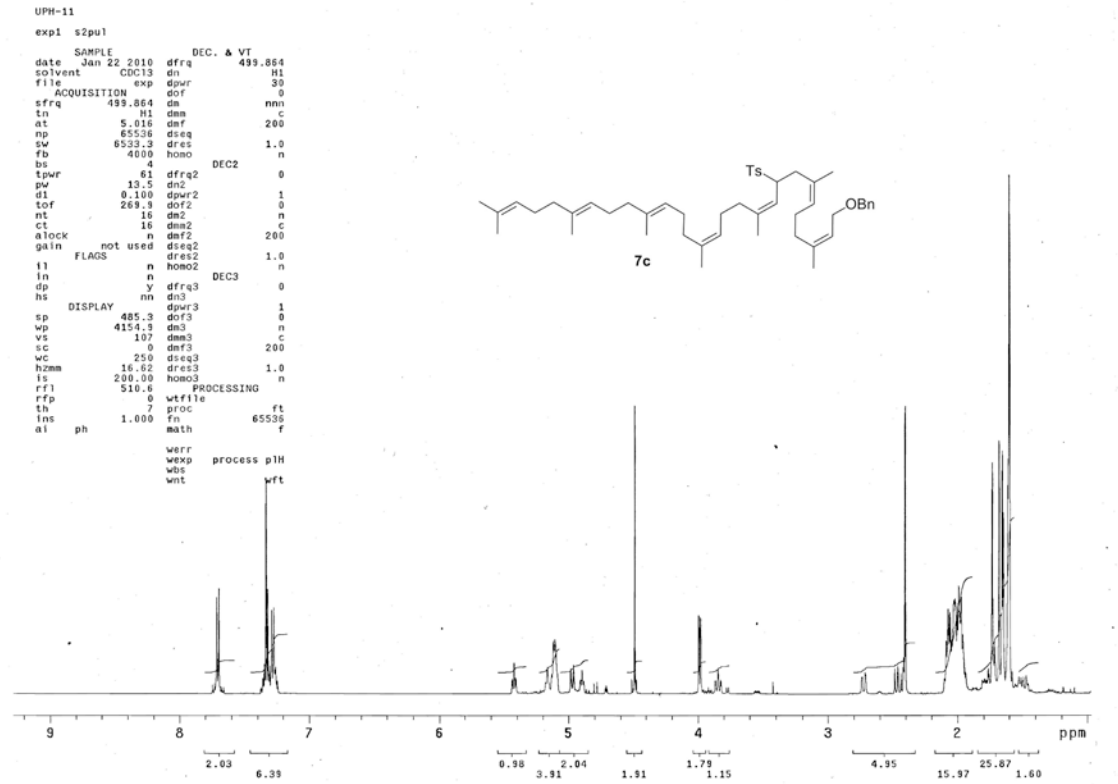
======== CHANNEL f2 ========
NUC2 13C
P3 14.80 usec
PL2 -2.00 dB
SFO2 201.2245355 MHz

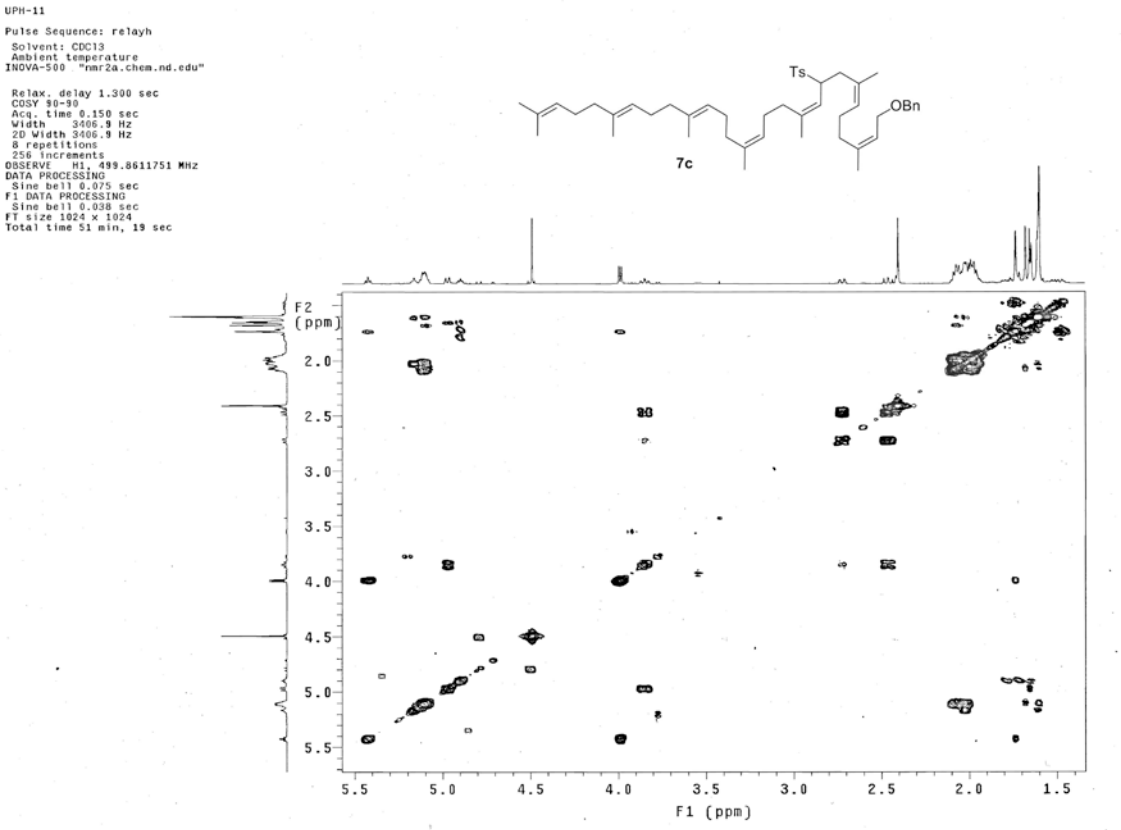
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPNAM5 SINE.100
GPNAM6 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %
GPZ5 -10.00 %
GPZ6 -5.00 %
P16 1000.00 usec

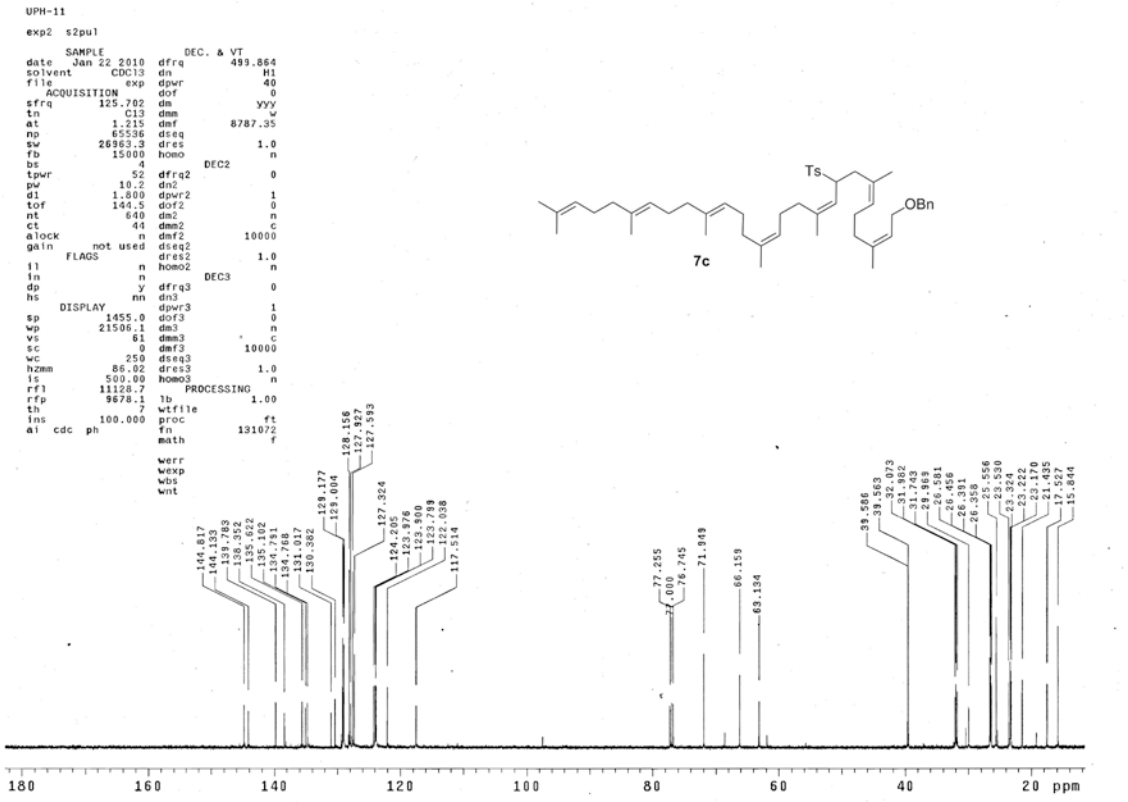
F1 - Acquisition parameters
TD 512
SFO1 201.2245 MHz
FIDRES 74.546753 Hz
SW 189.678 ppm
FnMODE QF

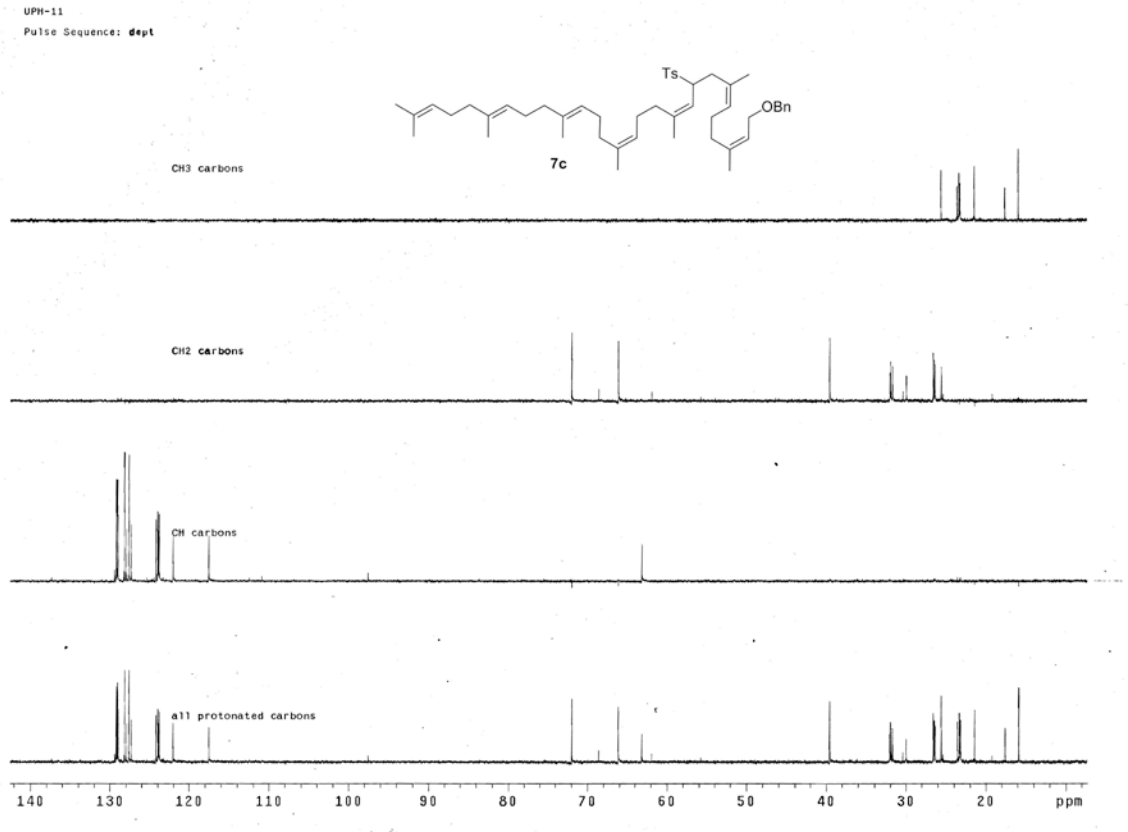
F2 - Processing parameters
SI 2048
SF 800.1800066 MHz
WDW QSINE
SSB 0
LB 0 Hz
GB 0
PC 1.40

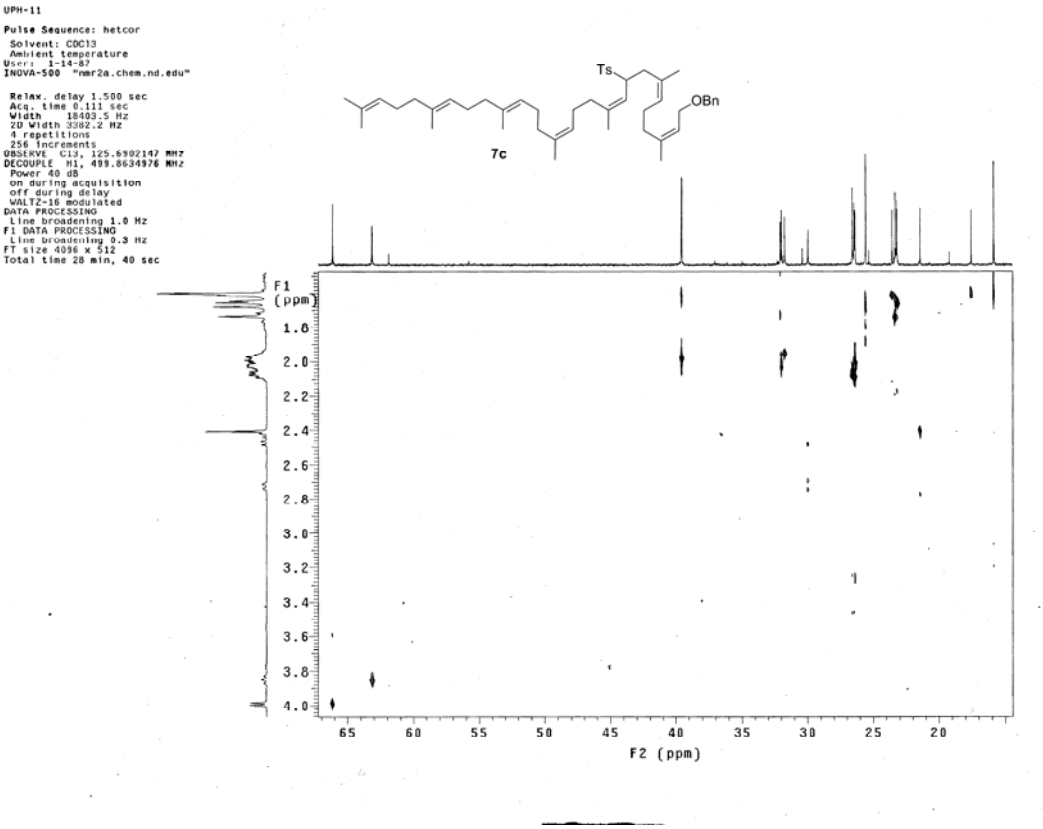
F1 - Processing parameters
SI 2048
MC2 QF
SF 201.2055102 MHz
WDW States-TPPI
SSB 0
LB 0 Hz
GB 0

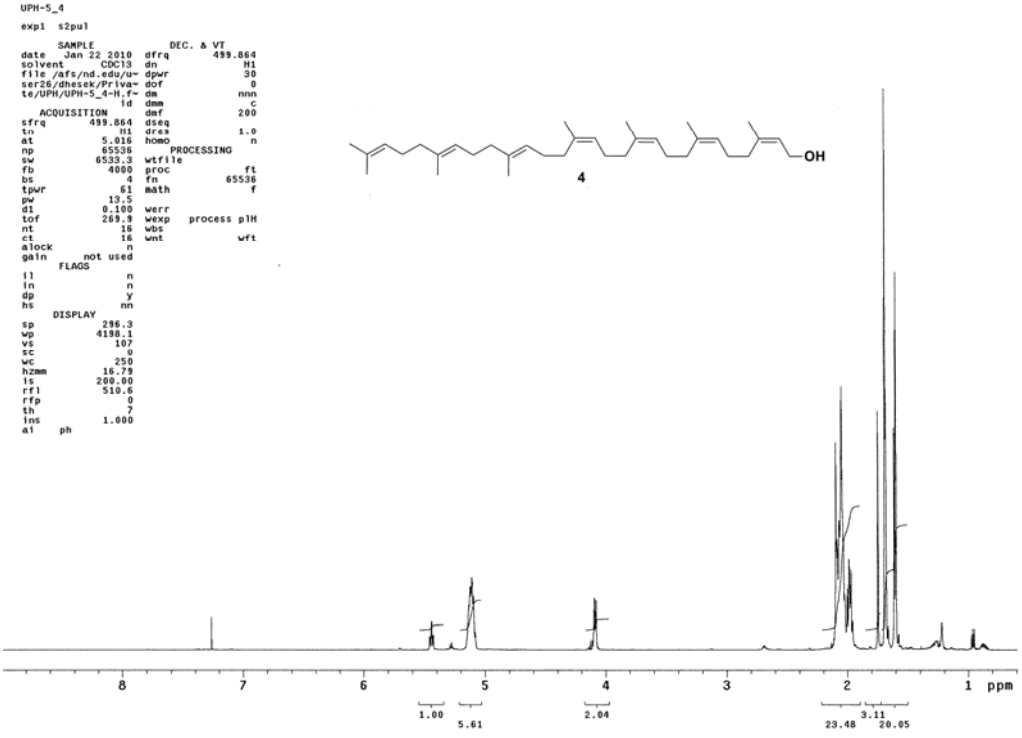

UPH-11
exp1 s2pul
SAMPLE
date Jan 22 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.8
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 485.3
wp 4154.3
vs 107
sc 0
wc 250
hzmm 16.62
is 200.00
rfl 510.6
rfp 0
th 7
ins 1.000
ai ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 200
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 200
dseq3
dres3 1.0
homo3 n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
Ts
OBn
7c
9
8
7
6
5
4
3
2
ppm
2.03
6.39
0.98
3.91
2.04
1.91
1.79
1.15
4.95
15.97
25.87
1.60


UPH-11
Pulse Sequence: relayh
Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.150 sec
Width 3406.9 Hz
2D Width 3406.9 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.075 sec
F1 DATA PROCESSING
Sine bell 0.038 sec
FT size 1024 x 1024
Total time 51 min, 19 sec
Ts
OBn
7c
F2 (ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
5.5
5.0
4.5
4.0
3.5
3.0
2.5
2.0
1.5
F1 (ppm)


UPH-11
exp2 s2pul
SAMPLE
date Jan 22 2010
solvent CDCl3
file exp
ACQUISITION
sfrq 125.702
tn C13
at 1.215
np 65536
sw 26963.3
fb 15000
bs 4
tpwr 52
pw 10.2
d1 1.800
tof 144.5
nt 640
ct 44
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 1455.0
wp 21506.1
vs 61
sc 0
wc 250
hzmm 86.02
is 500.00
rfl 11128.7
rfp 9678.1
th 7
ins 100.000
ai cdc ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 40
dof 0
dm yyy
dmm w
dmf 8787.35
dseq
dres 1.0
homo n
DEC2
dfrq2 0
dn2
dpwr2 1
dof2 0
dm2 n
dmm2 c
dmf2 10000
dseq2
dres2 1.0
homo2 n
DEC3
dfrq3 0
dn3
dpwr3 1
dof3 0
dm3 n
dmm3 c
dmf3 10000
dseq3
dres3 1.0
homo3 n
PROCESSING
lb 1.00
wtfile
proc ft
fn 131072
math f
werr
wexp
wbs
wnt
Ts
OBn
7c
144.817
144.133
139.783
138.252
135.622
135.102
134.791
134.768
131.017
130.382
128.177
128.156
127.927
127.593
127.324
124.205
123.586
123.300
123.799
122.038
117.514
77.255
77.000
76.745
71.949
66.159
63.134
39.586
39.563
32.073
31.982
31.743
29.969
29.581
26.456
26.391
26.358
25.556
23.530
23.324
23.222
23.170
21.435
17.527
15.844
180
160
140
120
100
80
60
40
20 ppm


UPH-11
Pulse Sequence: dept
Ts
OBn
7c
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
140
130
120
110
100
90
80
70
60
50
40
30
20
ppm


UPH-11
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3382.2 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6902147 MHz
DECOUPLE H1, 499.8634976 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 40 sec
Ts
OBn
7c
F1 (ppm)
F2 (ppm)

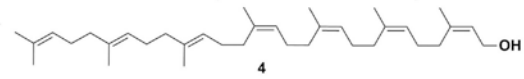

UPH-5_4
exp1 s2pul
SAMPLE
date Jan 22 2010
solvent CDCl3
file /afs/nd.edu/u~
ser26/dhesek/Priva~
te/UPH/UPH-5_4-H,f~
1d
ACQUISITION
sfrq 499.864
tn H1
at 5.016
np 65536
sw 6533.3
fb 4000
bs 4
tpwr 61
pw 13.5
d1 0.100
tof 269.9
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
hs nn
DISPLAY
sp 296.3
wp 4198.1
vs 107
sc 0
wc 250
hzmm 16.78
is 200.00
rfl 510.6
rfp 0
th 7
lns 1.000
al ph
DEC. & VT
dfrq 499.864
dn H1
dpwr 30
dof 0
dm nnn
dmm c
dmf 200
dseq
dres 1.0
homo n
PROCESSING
wtfile
proc ft
fn 65536
math f
werr
wexp process plH
wbs
wnt wft
OH
4
ppm
1.00
5.61
2.04
3.11
23.48
20.05

UPH-5_3

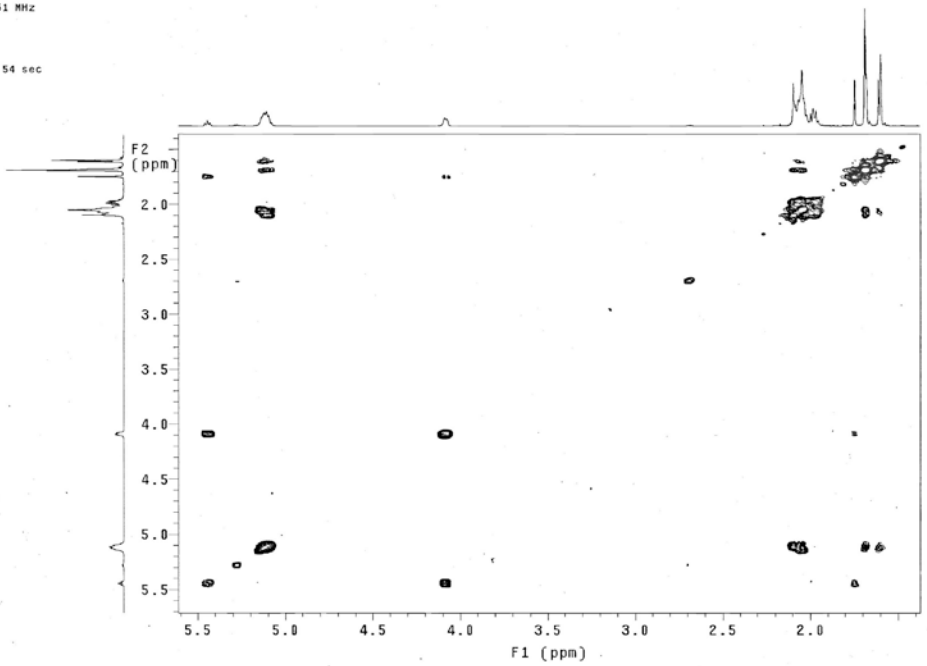
Pulse Sequence: relayh

Solvent: CDCl3
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.163 sec
Width 3133.4 Hz
20 Width 3133.4 Hz
32 repetitions
512 increments
OBSERVE H1, 499.8611751 MHz
DATA PROCESSING
Sine bell 0.082 sec
F1 DATA PROCESSING
Sine bell 0.041 sec
FT size 1024 x 1024
Total time 7 hr, 4 min, 54 sec



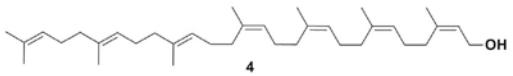
4



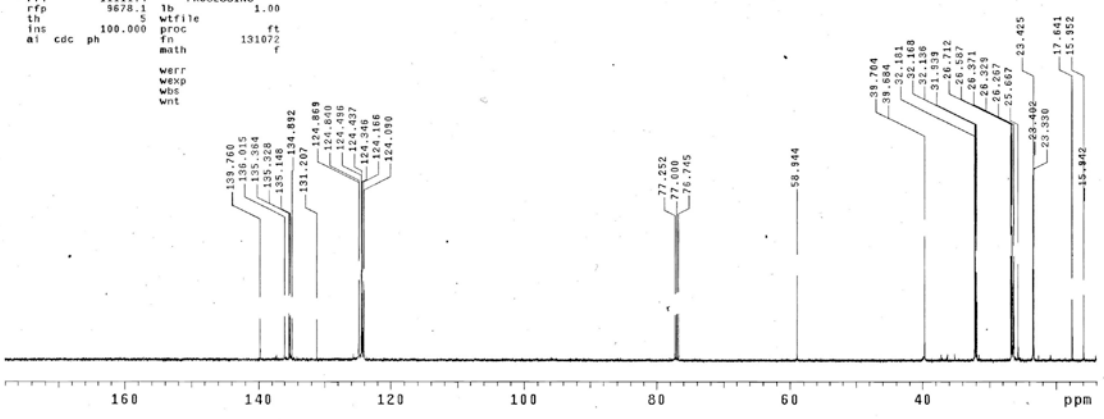
UPH-5_4

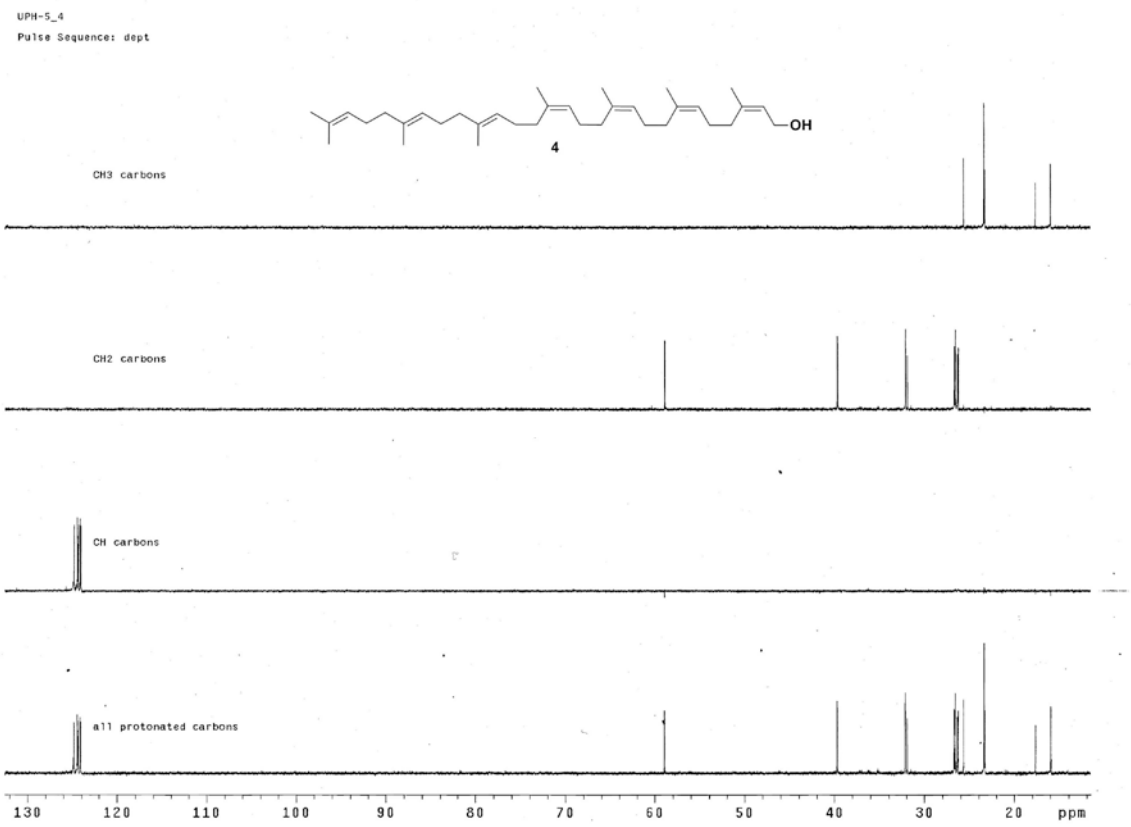
exp2 s2pul

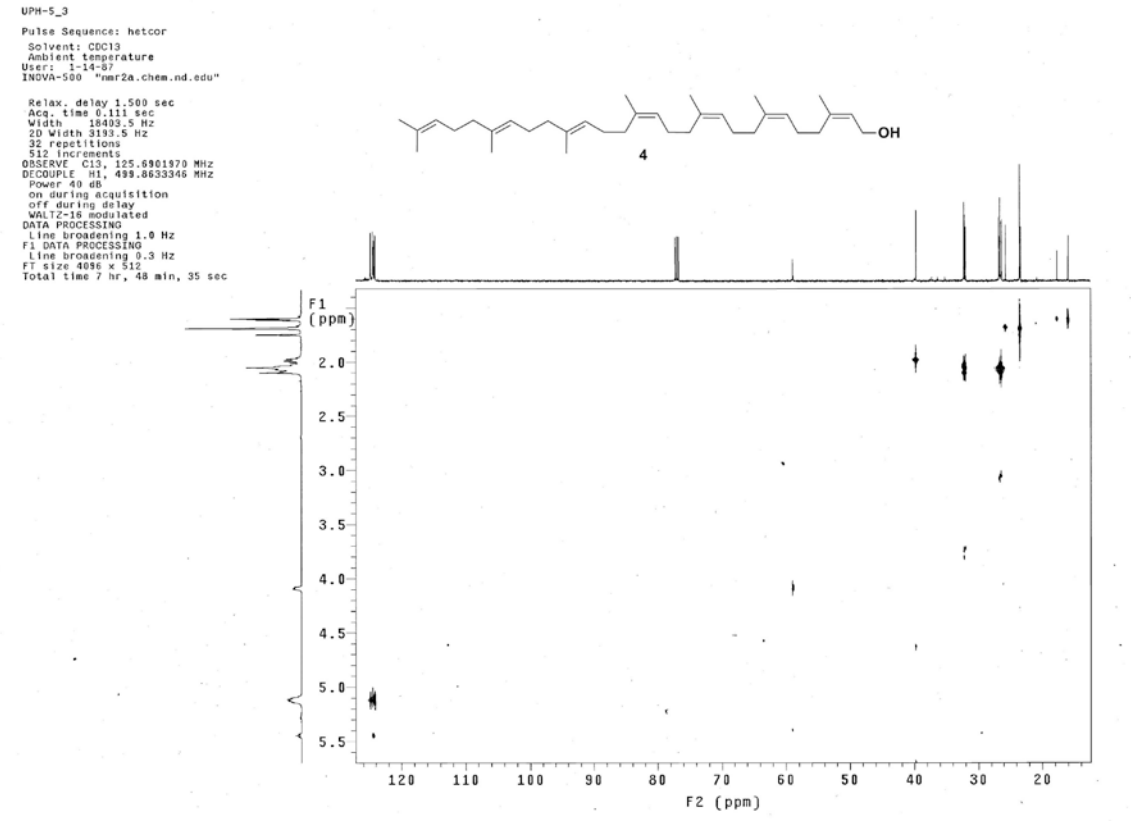
SAMPLE		DEC. & VT	
date	Jan 22 2010	dfrq	499.864
solvent	CDCl3	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.702	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26983.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	640	dm2	n
ct	151	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
il	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	1755.7	dof3	0
wp	20620.7	dm3	n
vs	81	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	82.48	dres3	1.0
is	500.00	homo3	n
rfl	11111.4	PROCESSING	
rfp	9678.1	lb	1.00
th	5	wtfile	
ins	100.000	proc	ft
ai cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

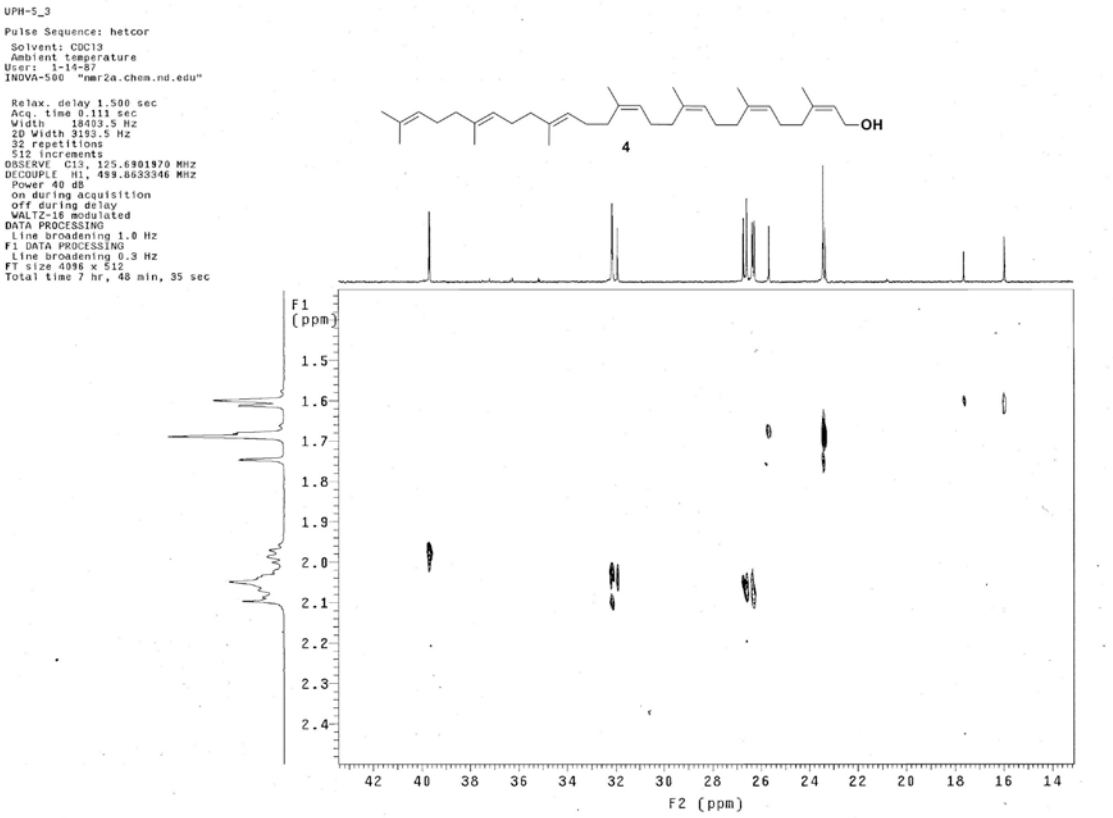


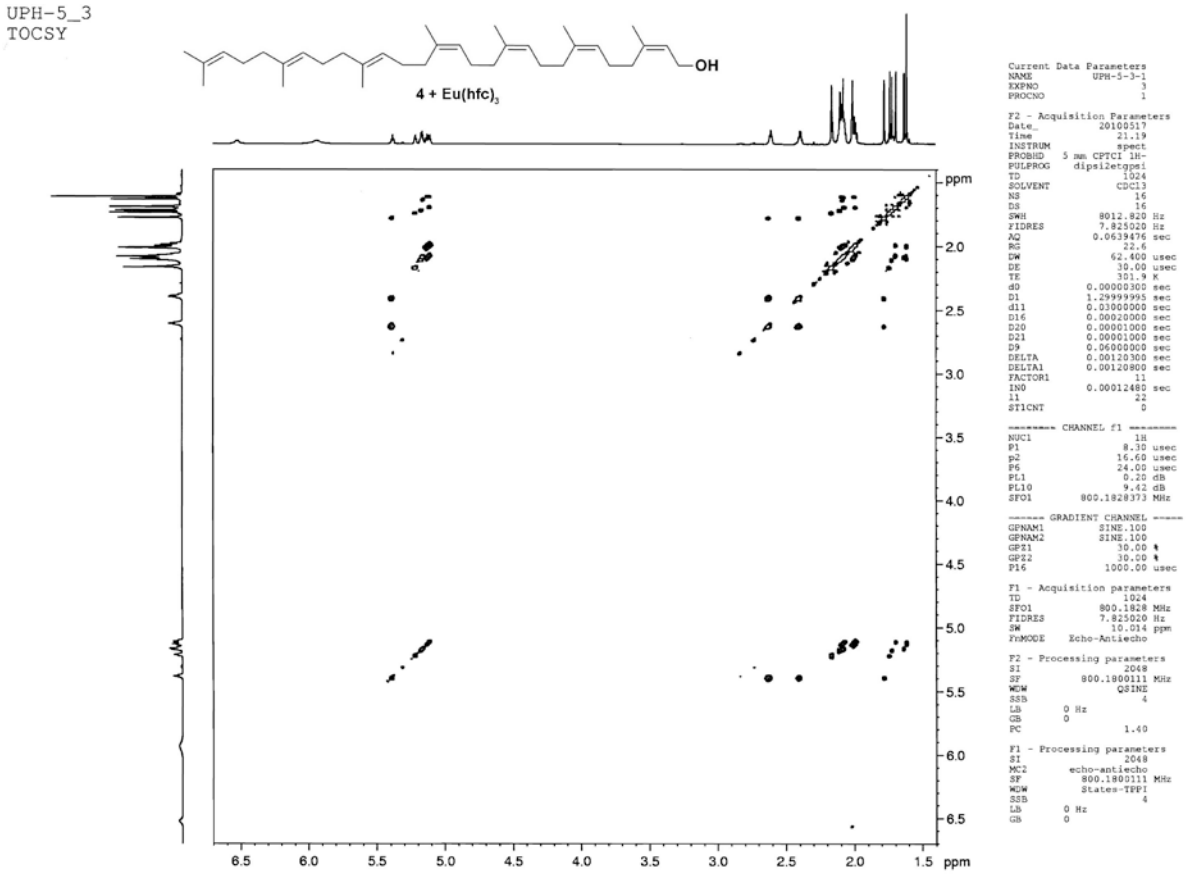
4



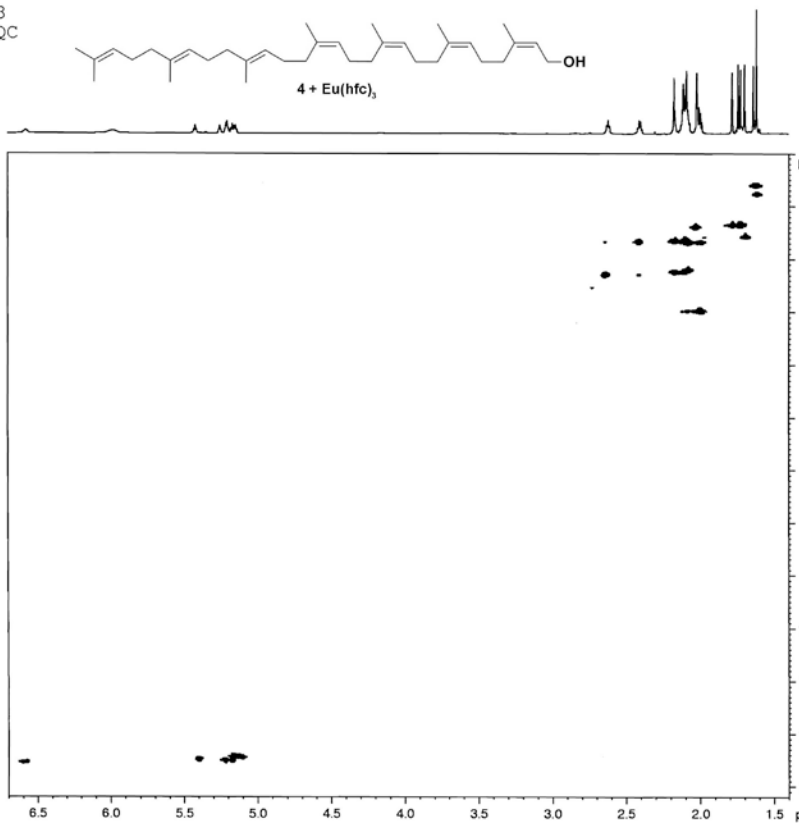

UPH-5_4
Pulse Sequence: dept
OH
4
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
130
120
110
100
90
80
70
60
50
40
30
20
ppm


UPH-5_3
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
20 Width 3193.5 Hz
32 repetitions
512 increments
OBSERVE C13, 125.6901970 MHz
DECOUPLE H1, 499.8633346 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 7 hr, 48 min, 35 sec
OH
4
F1
(ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
120
110
100
90
80
70
60
50
40
30
20
F2 (ppm)


UPH-5_3
Pulse Sequence: hetcor
Solvent: CDCl3
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 3193.5 Hz
32 repetitions
512 increments
OBSERVE C13, 125.6901970 MHz
DECOUPLE H1, 499.8633346 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 7 hr, 48 min, 35 sec
OH
4
F1 (ppm)
F2 (ppm)


UPH-5_3
TOCSY
OH
4 + Eu(hfc)3
ppm
Current Data Parameters
NAME UPH-5-3-1
EXPNO 3
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100517
Time 21.19
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG dipsi2etgpsi
TD 1024
SOLVENT CDCl3
NS 16
DS 16
SWH 8012.820 Hz
FIDRES 7.825020 Hz
AQ 0.0639476 sec
RG 22.6
DW 62.400 usec
DE 30.00 usec
TE 301.9 K
d0 0.00000300 sec
D1 1.29999995 sec
d11 0.03000000 sec
D16 0.00020000 sec
D20 0.00001000 sec
D21 0.00001000 sec
D9 0.06000000 sec
DELTA 0.00120300 sec
DELTA1 0.00120800 sec
FACTOR1 11
IN0 0.00012480 sec
l1 22
ST1CNT 0
CHANNEL f1
NUC1 1H
P1 8.30 usec
p2 16.60 usec
P6 24.00 usec
PL1 0.20 dB
PL10 9.42 dB
SFO1 800.1828373 MHz
GRADIENT CHANNEL
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 1024
SFO1 800.1828 MHz
FIDRES 7.825020 Hz
SW 10.014 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 2048
SF 800.1800111 MHz
WDW QSINE
SSB 4
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 2048
MC2 echo-antiecho
SF 800.1800111 MHz
WDW States-TPPI
SSB 4
LB 0 Hz
GB 0

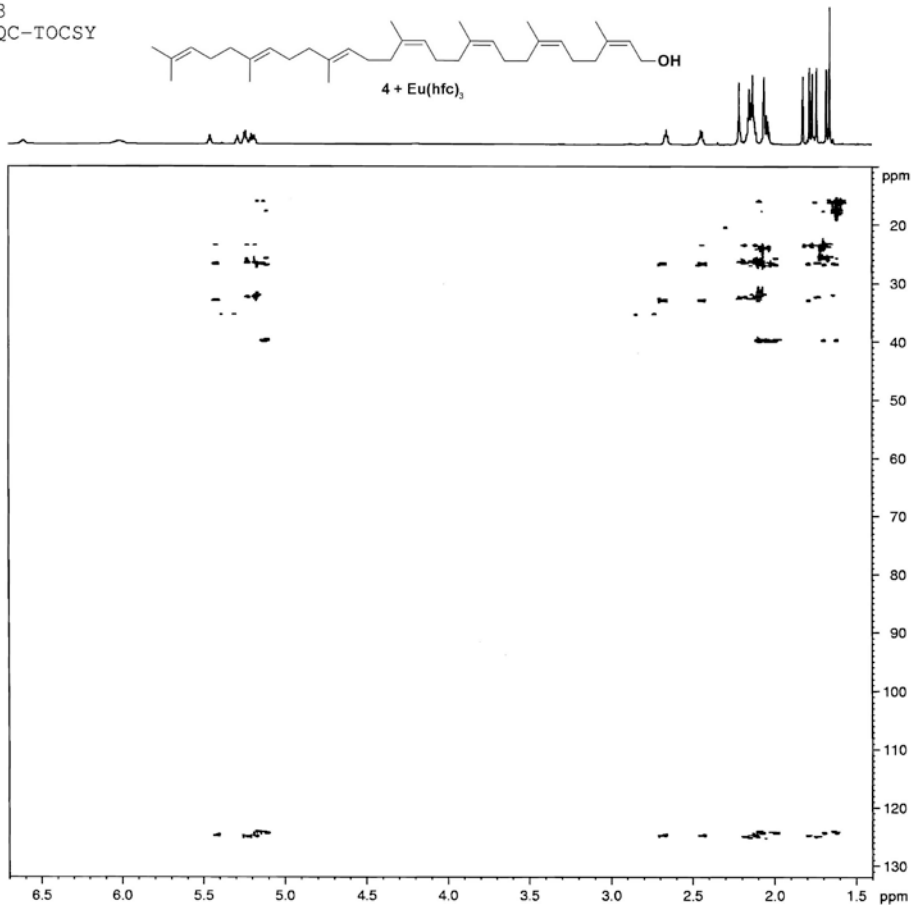
UPH-5_3
13C-HSQC



Current Data Parameters
NAME UPH-5-3-1
EXPNO 4
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100518
Time 4.10
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hsqcdetgpsisp2.2
TD 1024
SOLVENT CDCl3
NS 24
DS 96
SWH 8012.820 Hz
FIDRES 7.825020 Hz
AQ 0.0639676 sec
RG 4100
DW 62.400 usec
DE 30.00 usec
TE 301.9 K
CNST17 -0.5000000
CNST2 145.0000000
d0 0.00000300 sec
D1 1.39999991 sec
d11 0.03000000 sec
D16 0.00020000 sec
D24 0.00085200 sec
d4 0.00172414 sec
D9 0.00600000 sec
DELTA 0.00171826 sec
DELTA1 0.00120800 sec
DELTA2 0.00147414 sec
DELTA3 0.00064207 sec
DELTA4 0.00027414 sec
DELTA5 0.00297006 sec
ING 0.00001655 sec
IN0 0.00000655 sec
NUC1 1H
P1 8.30 usec
p2 16.60 usec
PL0 120.00 dB
PL1 0.20 dB
SFO1 800.1823373 MHz
CPDPRG2 p5m4sp180
NUC2 13C
P14 500.00 usec
P24 2000.00 usec
P3 14.80 usec
PCPD2 1500.00 usec
PL0 120.00 dB
PL12 10.00 dB
PL2 -2.00 dB
SFO2 201.2205114 MHz
SF3 1.08 dB
SF7 1.08 dB
SPNAM15 Crp,80,1.5,10.nd
SPNAM3 Crp80,6.5,20.1
SPNAM7 Crp80comp.4
SPOAL15 0.500
SPOAL3 0.500
SPOAL7 0.500
SPOFFS15 0 Hz
SPOFFS3 0 Hz
SPOFFS7 0 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM7 SINE.100
GPNAM4 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
GPZ3 11.00 %
GPZ4 5.00 %
P16 1000.00 usec
P19 500.00 usec
F1 - Acquisition parameters
TD 1024
SFO1 201.2215 MHz
FIDRES 31.503107 Hz
SW 150.141 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 2048
SF 800.1800130 MHz
WDW QSINE
SSB 3
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 4096
MC2 echo-antiecho
SF 201.2054210 MHz
WDW States-TPPI
SSB 3
LB 0 Hz
GB 0

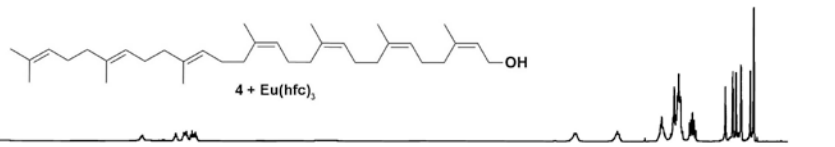
UPH-5_3
13C-HSQC-TOCSY

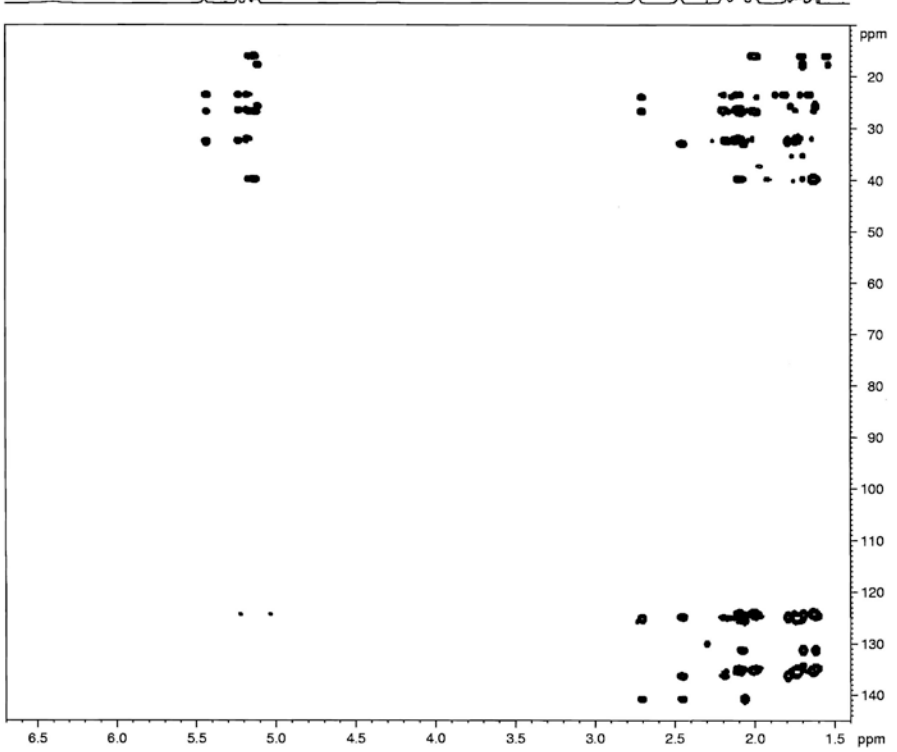


Current Data Parameters
NAME UPH-5-3-1
EXPNO 5
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100518
Time 23.11
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hsqcdietgpsisp2.2
TD 1024
SOLVENT CDCl3
NS 32
DS 128
SWH 8012.820 Hz
FIDRES 7.825020 Hz
AQ 0.0639676 sec
RG 2300
DW 62.400 usec
DE 30.00 usec
TE 301.9 K
CNST17 -0.5000000
CNST2 145.0000000
d0 0.00000300 sec
D1 1.39999998 sec
d11 0.03000000 sec
D16 0.00020000 sec
D24 0.00085200 sec
d4 0.00172414 sec
D9 0.06000000 sec
DELTA 0.00171826 sec
DELTA1 0.00120800 sec
DELTA2 0.00147014 sec
DELTA3 0.00135800 sec
FACTOR1 11
IN0 0.00001655 sec
l1 22
ST1CNT 0
======== CHANNEL f1 ========
NUC1 1H
P1 8.10 usec
p2 16.20 usec
P28 1000.00 usec
P6 24.00 usec
PL1 0.20 dB
PL10 9.63 dB
SFO1 800.1828373 MHz
======== CHANNEL f2 ========
CPDPRG2 p5m4sp180
NUC2 13C
P14 500.00 usec
P24 2000.00 usec
P3 14.80 usec
PCPD2 1500.00 usec
PL0 120.00 dB
PL12 10.00 dB
PL2 -2.00 dB
SFO2 201.2205114 MHz
SF3 1.08 dB
SF7 1.08 dB
SPNAM15 Crp,80,1.5,10.nd
SPNAM3 Crp80,6.5,20.1
SPNAM7 Crp80comp.4
SPOAL15 0.500
SPOAL3 0.500
SPOAL7 0.500
SPOFFS15 0 Hz
SPOFFS3 0 Hz
SPOFFS7 0 Hz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 1024
SFO1 201.2205 MHz
FIDRES 29.503399 Hz
SW 150.141 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 2048
SF 800.1800130 MHz
WDW QSINE
SSB 4
LB 0 Hz
GB 0
PC 1.00
F1 - Processing parameters
SI 2048
MC2 echo-antiecho
SF 201.2054289 MHz
WDW States-TPPI
SSB 4
LB 0 Hz
GB 0

UPH-5_3
13C-HMBC






```
Current Data Parameters
NAME                 UPH-5-3-1
EXPNO                        6
PROCNO                       1

F2 - Acquisition Parameters
Date_              20100519
Time                   13.25
INSTRUM                spect
PROBHD    5 mm CPTCI 1H-
PULPROG    hmbcgp12ndqf
TD                     2048
SOLVENT                CDCl3
NS                       64
DS                      128
SWH              8223.685 Hz
FIDRES           4.015471 Hz
AQ               0.1245684 sec
RG                     4600
DW                 60.800 usec
DE                 30.00 usec
TE                 301.9 K
CNST13             8.0000000
CNST6            125.0000000
CNST7            175.0000000
d0                 0.00000300 sec
D1                 1.39999998 sec
D16                0.00020000 sec
d6                 0.06250000 sec
DELTA1             0.00280000 sec
DELTA2             0.00165714 sec
DELTA3             0.06129600 sec
ING                0.00001310 sec

======== CHANNEL f1 ========
NUC1                     1H
P1                    8.10 usec
p2                   16.20 usec
PL1                   0.20 dB
SFO1           800.1828373 MHz

======== CHANNEL f2 ========
NUC2                    13C
P3                   14.80 usec
PL2                  -2.00 dB
SFO2           201.2245355 MHz

====== GRADIENT CHANNEL =====
GPNAM1              SINE.100
GPNAM2              SINE.100
GPNAM3              SINE.100
GPNAM4              SINE.100
GPNAM5              SINE.100
GPNAM6              SINE.100
GPZ1                 50.00 %
GPZ2                 30.00 %
GPZ3                 40.10 %
GPZ4                 15.00 %
GPZ5                -10.00 %
GPZ6                 -5.00 %
P16                1000.00 usec

F1 - Acquisition parameters
TD                       512
SFO1           201.2245 MHz
FIDRES          74.546753 Hz
SW                189.678 ppm
FnMODE                   QF

F2 - Processing parameters
SI                       2048
SF             800.1800130 MHz
WDW                    QSINE
SSB                      0
LB                       0 Hz
GB                       0
PC                     1.40

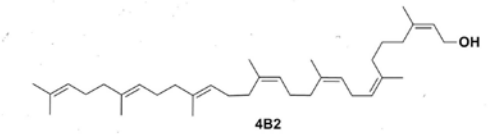
F1 - Processing parameters
SI                       4096
MC2                      QF
SF             201.2054289 MHz
WDW                    States-TPPI
SSB                      0
LB                       0 Hz
GB                       0
```

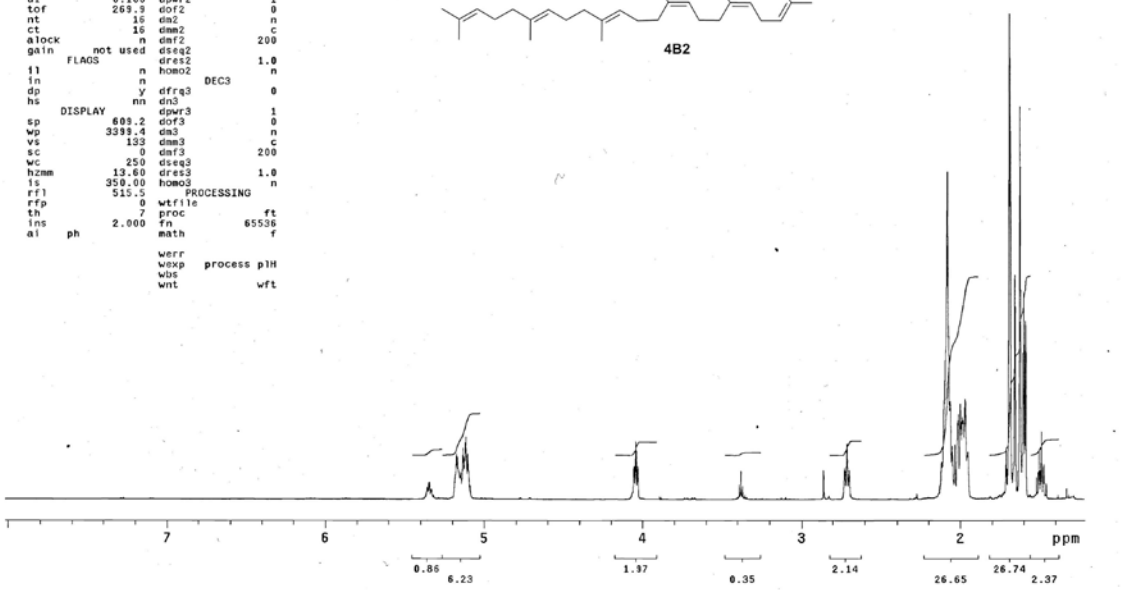
UPH_5_2

```
exp4  s2pul

      SAMPLE                 DEC. & VT
date   Jan  4 2010    dfrq          499.866
solvent     acetone   dn                 H1
file             exp  dpwr               30
    ACQUISITION       dof                 0
sfrq          499.867  dm                nnn
tn                 H1  dmm                 c
at               5.016 dmf               200
np               65536 dseq
sw              6533.3 dres               1.0
fb               4000  homo                n
bs                  4              DEC2
tpwr               61  dfrq2               0
pw               13.5  dn2
d1              0.100  dpwr2               1
tof             269.9  dof2                0
nt                 16  dm2                 n
ct                 16  dmm2                c
alock               n  dmf2              200
gain       not used    dseq2
     FLAGS             dres2             1.0
fl                  n  homo2               n
in                  n              DEC3
dp                  y  dfrq3               0
hs                 nn  dn3
    DISPLAY           dpwr3               1
sp               608.2  dof3                0
wp              3399.4  dm3                 n
vs                 133  dmm3                c
sc                   0  dmf3              200
wc                 250  dseq3
hzmm             13.60  dres3             1.0
is              350.00  homo3               n
rfl              515.5        PROCESSING
rfp                  0  wtfile
th                   7  proc                ft
ins              2.000  fn               65536
ai    ph               math                f

                        werr
                        wexp   process plH
                        wbs
                        wnt                 wft
```



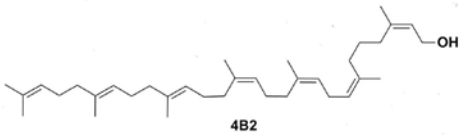


UPH_5_2

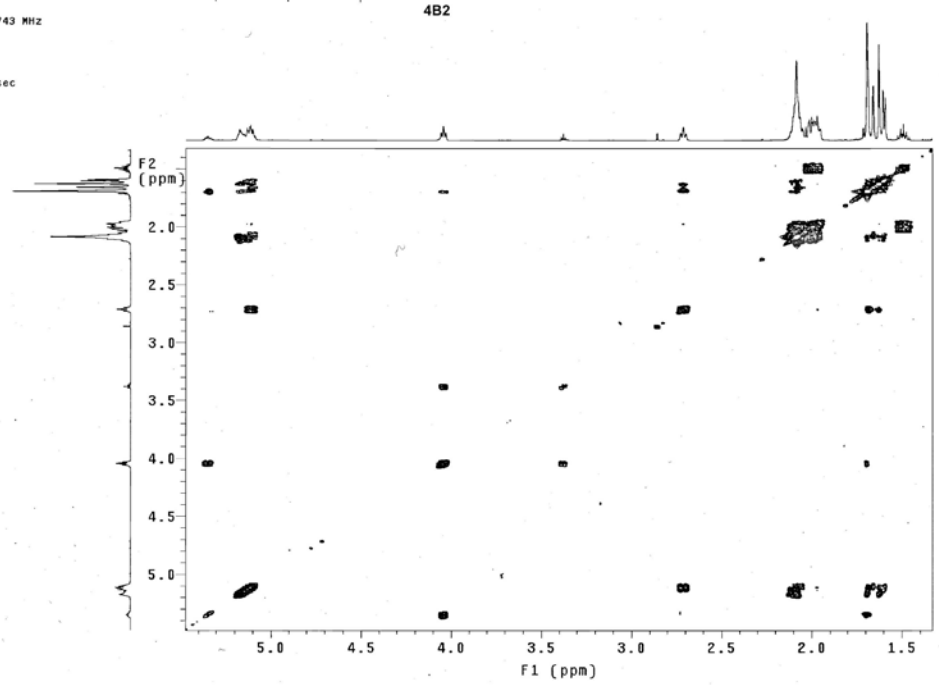
Pulse Sequence: relayh

Solvent: acetone
Ambient temperature
INOVA-500 "nmr2a.chem.nd.edu"

Relax. delay 1.300 sec
COSY 90-90
Acq. time 0.227 sec
Width 2257.8 Hz
2D Width 2257.8 Hz
8 repetitions
256 increments
OBSERVE H1, 499.8637743 MHz
DATA PROCESSING
Sine bell 0.113 sec
F1 DATA PROCESSING
Sine bell 0.057 sec
FT size 1024 x 1024
Total time 54 min, 36 sec



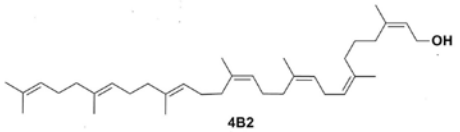
4B2



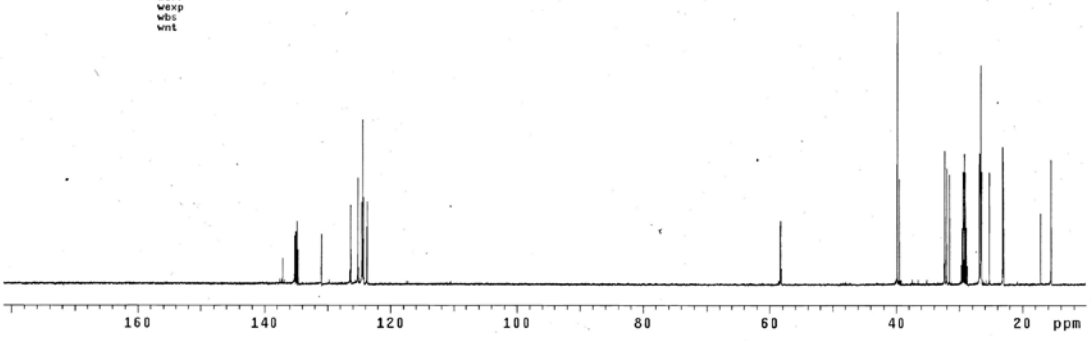
UPH_5_2

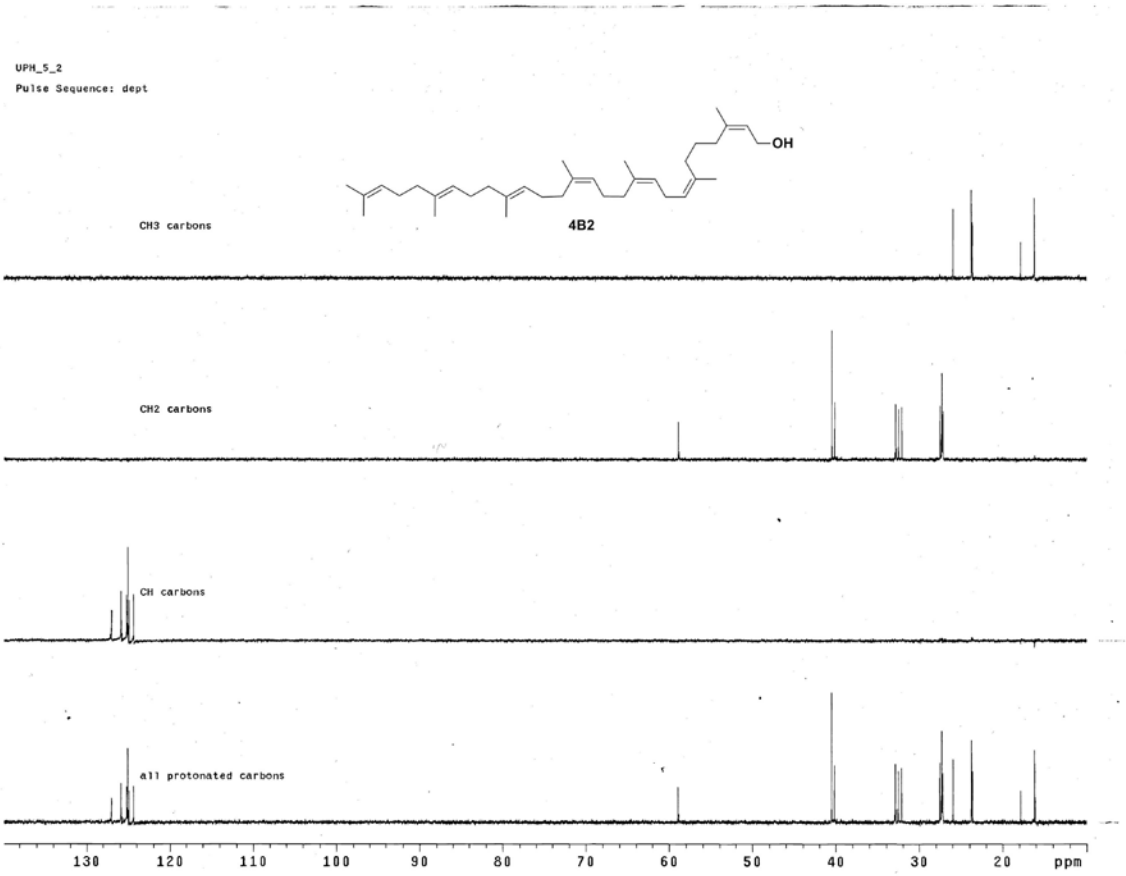
exp5 s2pul

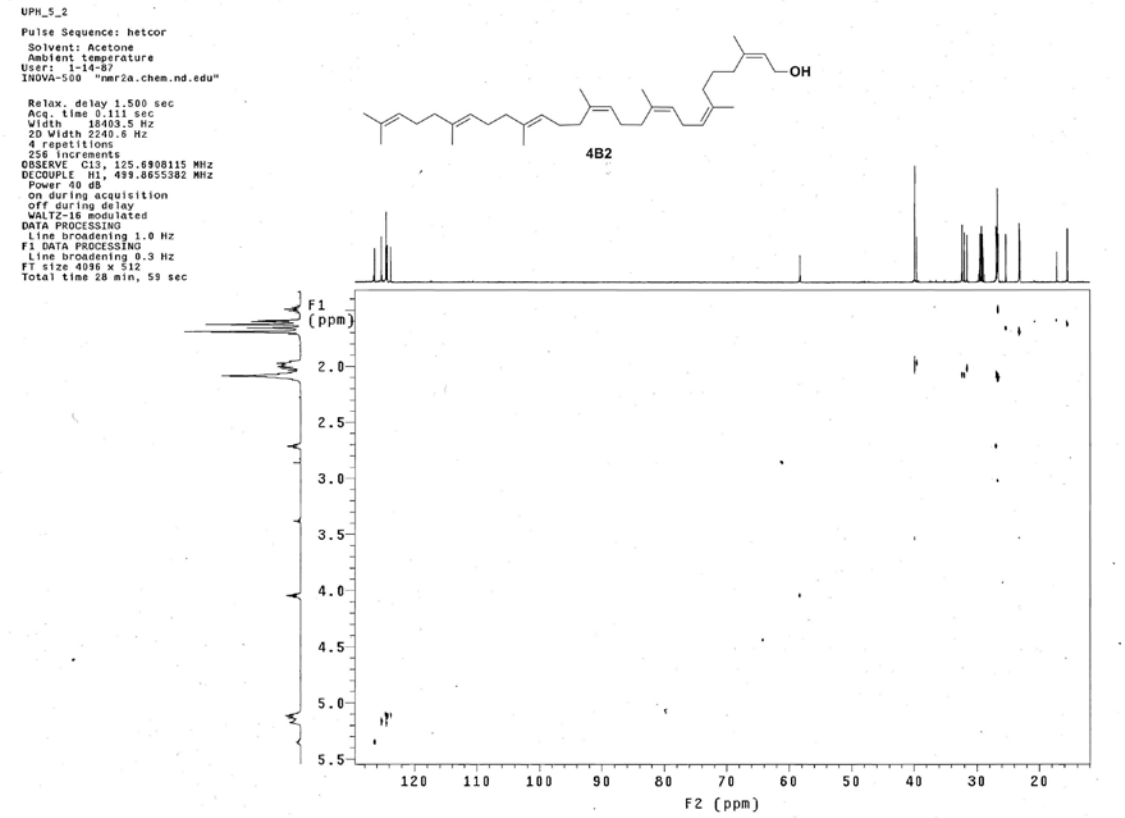
SAMPLE		DEC. & VT	
date	Jan 4 2010	dfrq	499.866
solvent	Acetone	dn	H1
file	exp	dpwr	40
ACQUISITION		dof	0
sfrq	125.703	dm	yyy
tn	C13	dmm	w
at	1.215	dmf	8787.35
np	65536	dseq	
sw	26963.3	dres	1.0
fb	15000	homo	n
bs	4	DEC2	
tpwr	52	dfrq2	0
pw	10.2	dn2	
d1	1.800	dpwr2	1
tof	144.5	dof2	0
nt	640	dm2	n
ct	311	dmm2	c
alock	n	dmf2	10000
gain	not used	dseq2	
FLAGS		dres2	1.0
fl	n	homo2	n
in	n	DEC3	
dp	y	dfrq3	0
hs	nn	dn3	
DISPLAY		dpwr3	1
sp	1272.7	dof3	0
wp	21506.1	dm3	n
vs	96	dmm3	c
sc	0	dmf3	10000
wc	250	dseq3	
hzmm	86.02	dres3	1.0
is	500.00	homo3	n
rfl	1395.0	PROCESSING	
rfp	0	lb	1.00
th	4	wtfile	
ins	100.000	proc	ft
al cdc ph		fn	131072
		math	f
		werr	
		wexp	
		wbs	
		wnt	

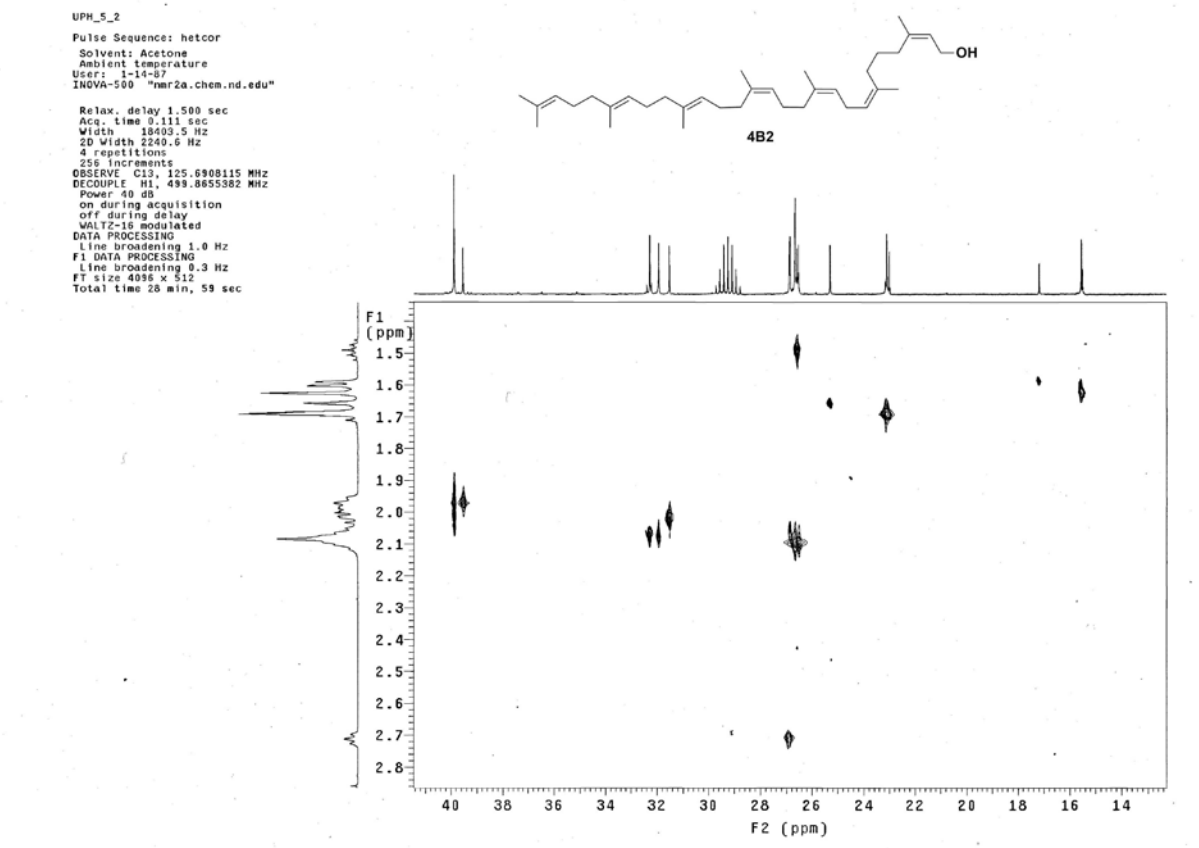


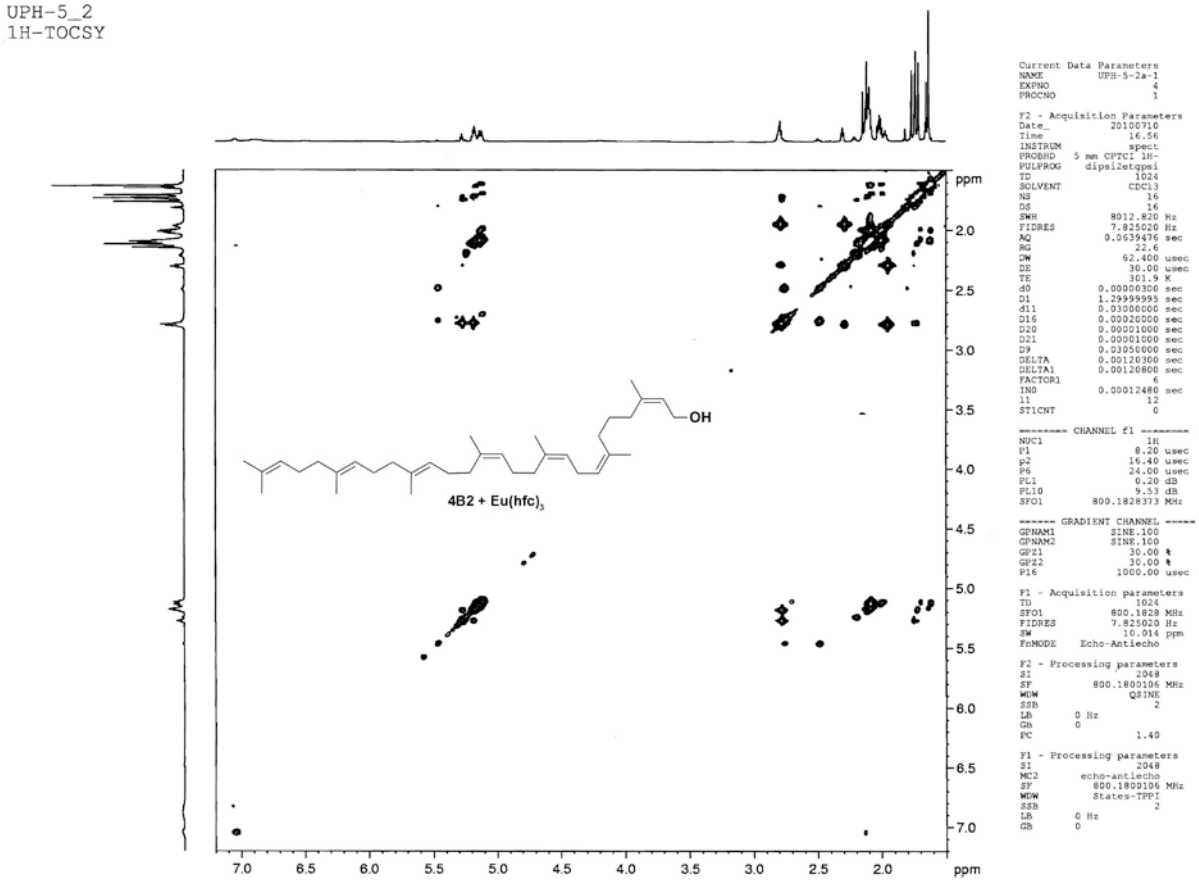
4B2



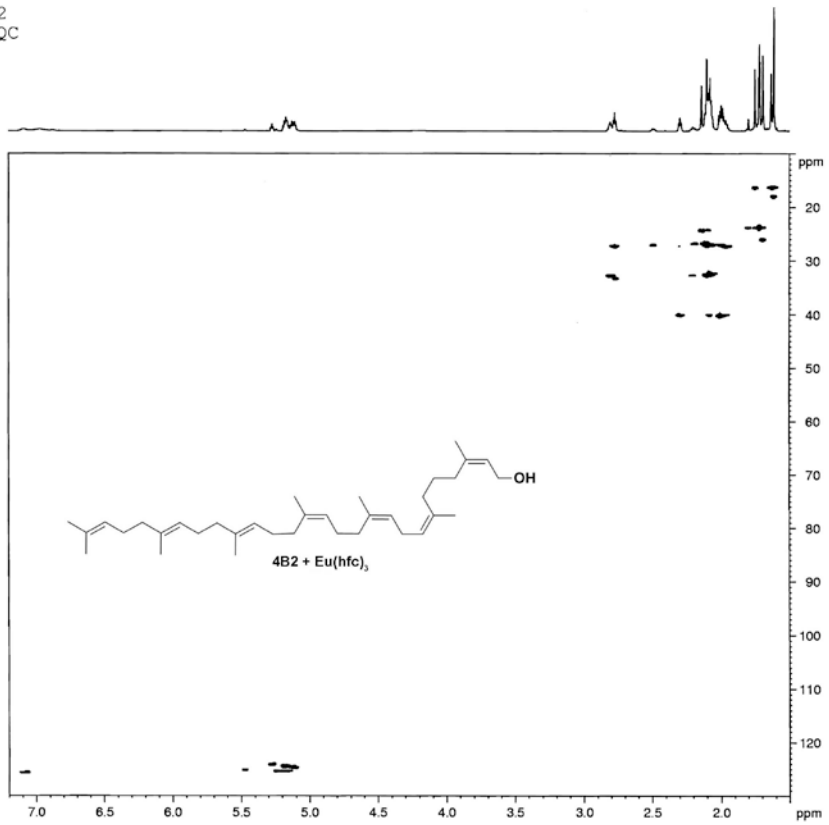

UPH_5_2
Pulse Sequence: dept
OH
4B2
CH3 carbons
CH2 carbons
CH carbons
all protonated carbons
130
120
110
100
90
80
70
60
50
40
30
20
ppm


UPH_5_2
Pulse Sequence: hetcor
Solvent: Acetone
Ambient temperature
User: 1-14-07
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2240.6 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6808115 MHz
DECOUPLE H1, 499.8655382 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 59 sec
OH
4B2
F1
(ppm)
2.0
2.5
3.0
3.5
4.0
4.5
5.0
5.5
120
110
100
90
80
70
60
50
40
30
20
F2 (ppm)


UPH_5_2
Pulse Sequence: hetcor
Solvent: Acetone
Ambient temperature
User: 1-14-87
INOVA-500 "nmr2a.chem.nd.edu"
Relax. delay 1.500 sec
Acq. time 0.111 sec
Width 18403.5 Hz
2D Width 2240.6 Hz
4 repetitions
256 increments
OBSERVE C13, 125.6908115 MHz
DECOUPLE H1, 499.8655382 MHz
Power 40 dB
on during acquisition
off during delay
WALTZ-16 modulated
DATA PROCESSING
Line broadening 1.0 Hz
F1 DATA PROCESSING
Line broadening 0.3 Hz
FT size 4096 x 512
Total time 28 min, 59 sec
OH
4B2
F1 (ppm)
F2 (ppm)


UPH-5_2
1H-TOCSY
OH
4B2 + Eu(hfc)3
ppm
Current Data Parameters
NAME UPH-5-2a-1
EXPNO 4
PROCNO 1
F2 - Acquisition Parameters
Date_ 20100710
Time 16.56
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG dipsi2etqpsi
TD 1024
SOLVENT CDCl3
NS 16
DS 16
SWH 8012.820 Hz
FIDRES 7.825020 Hz
AQ 0.0639476 sec
RG 22.6
DW 62.400 usec
DE 30.00 usec
TE 301.9 K
d0 0.00000350 sec
D1 1.29999995 sec
d11 0.03000000 sec
D16 0.00020000 sec
D20 0.00001000 sec
D21 0.00001000 sec
D9 0.03050000 sec
DELTA 0.00120300 sec
DELTA1 0.00120800 sec
FACTOR1 6
IN0 0.00012480 sec
l1 12
ST1CNT 0
======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
p2 16.40 usec
P6 24.00 usec
PL1 0.20 dB
PL10 9.53 dB
SFO1 800.1828373 MHz
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 30.00 %
GPZ2 30.00 %
P16 1000.00 usec
F1 - Acquisition parameters
TD 1024
SFO1 800.1828 MHz
FIDRES 7.825020 Hz
SW 10.014 ppm
FnMODE Echo-Antiecho
F2 - Processing parameters
SI 2048
SF 800.1800106 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40
F1 - Processing parameters
SI 2048
MC2 echo-antiecho
SF 800.1800106 MHz
WDW States-TPPI
SSB 2
LB 0 Hz
GB 0

UPH-5_2
13C-HSQC



Current Data Parameters
NAME UPH-5-2a-1
EXPNO 8
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100711
Time 6.60
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hsqcedetgpsisp2.2
TD 1024
SOLVENT CDCl3
NS 24
DS 96
SWH 8012.820 Hz
FIDRES 7.825020 Hz
AQ 0.0639476 sec
RG 8250
DW 62.400 usec
DE 30.00 usec
TE 301.9 K
CNST17 -0.5000000
CNST2 145.0000000
d0 0.00000303 sec
D1 1.29999995 sec
d11 0.03000000 sec
D16 0.00020000 sec
D21 0.00345000 sec
D24 0.00086207 sec
d4 0.00172414 sec
DELTA 0.00272760 sec
DELTA1 0.00120803 sec
DELTA2 0.00167414 sec
DELTA3 0.00064207 sec
DELTA4 0.00027414 sec
DELTA5 0.00079000 sec
INO 0.00001655 sec
SYSCNT 0

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
p2 16.40 usec
P28 1000.00 usec
PL1 0.20 dB
SFO1 800.1828373 MHz

======== CHANNEL f2 ========
CPDPRG2 p5m4sp180
NUC2 13C
P14 500.00 usec
P24 2000.00 usec
P3 14.80 usec
PCPD2 1500.00 usec
PL2 120.00 dB
PL12 10.00 dB
PL2 -2.00 dB
SFO2 201.2205114 MHz
SP3 1.08 dB
SP7 1.08 dB
SPNAM15 Crp,80,1.5,10.nd
SPNAM3 Crp80,0.5,20.1
SPNAM7 Crp80comp.4
SPOAL15 0.500
SPOAL3 0.500
SPOAL7 0.500
SPOFFS15 0 Hz
SPOFFS3 0 Hz
SPOFFS7 0 Hz

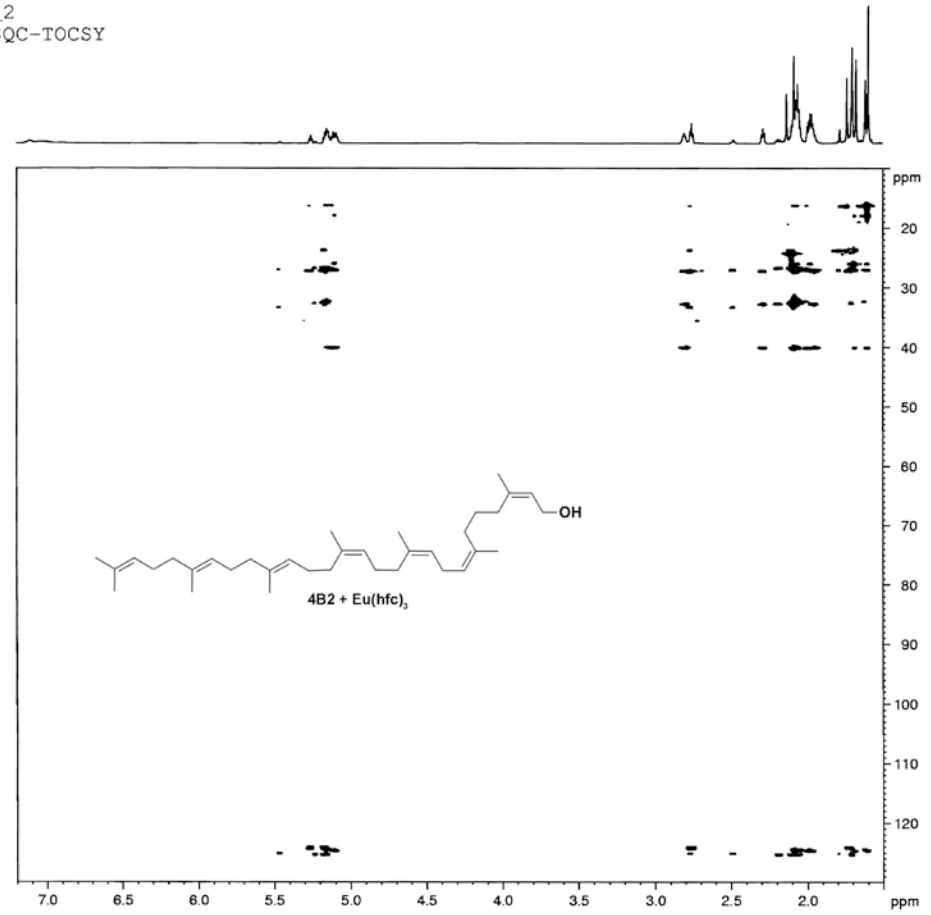
======= GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
GPZ3 11.08 %
GPZ4 -5.00 %
P16 1000.00 usec
P19 500.00 usec

F1 - Acquisition parameters
TD 1024
SFO1 201.2203 MHz
FIDRES 29.503399 Hz
SW 150.141 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 800.1800106 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 4096
MC2 echo-antiecho
SF 201.2053682 MHz
WDW States-TPPI
SSB 2
LB 0 Hz
GB 0

UPH-5_2
13C-HSQC-TOCSY



Current Data Parameters
NAME UPH-5-2a-1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100711
Time 16.20
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hsqcdietgpsisp.2
TD 1024
SOLVENT CDCl3
NS 32
DS 128
SWH 8012.820 Hz
FIDRES 7.825020 Hz
AQ 0.0639476 sec
RG 2050
DW 62.400 usec
DE 30.00 usec
TE 301.9 K
CNST17 -0.5000000
CNST2 145.0000000
d0 0.00000300 sec
D1 1.39999998 sec
d11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
d4 0.00172414 sec
D9 0.04100000 sec
DELTA 0.00121840 sec
DELTA1 0.00120800 sec
DELTA2 0.00167914 sec
DELTA3 0.00135800 sec
FACTOR1 12
INO 0.00001655 sec
l1 24
SYSCNT 0

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
p2 16.40 usec
P28 1000.00 usec
P5 24.00 usec
PL1 0.20 dB
PL10 9.93 dB
SFO1 800.1828373 MHz

======== CHANNEL f2 ========
CPDPRG2 p5m4sp180
NUC2 13C
P14 500.00 usec
P24 2000.00 usec
P3 14.80 usec
PCPD2 1500.00 usec
PL2 120.00 dB
PL12 10.00 dB
PL2 -2.00 dB
SFO2 201.2205114 MHz
SP3 1.08 dB
SP7 1.08 dB
SPNAM15 Crp,80,1.5,10.nd
SPNAM3 Crp80,0.5,20.1
SPNAM7 Crp80comp.4
SPOAL15 0.500
SPOAL3 0.500
SPOAL7 0.500
SPOFFS15 0 Hz
SPOFFS3 0 Hz
SPOFFS7 0 Hz

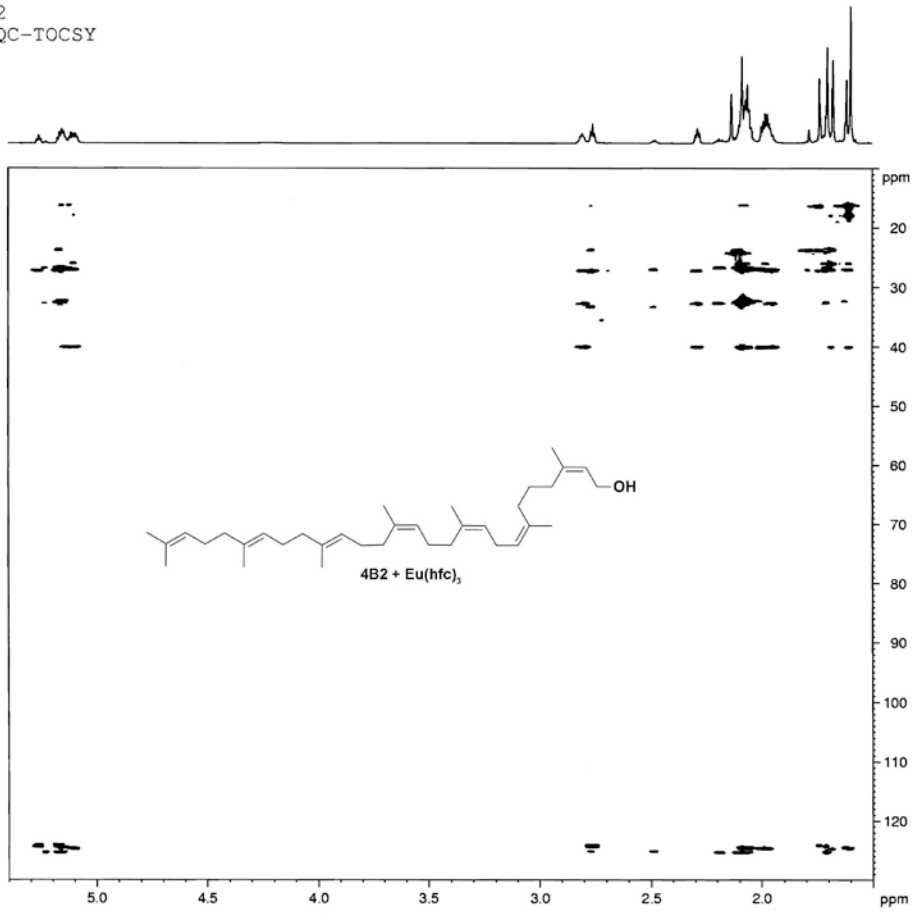
======= GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ1 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 1024
SFO1 201.2203 MHz
FIDRES 29.503399 Hz
SW 150.141 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 800.1800190 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 4096
MC2 echo-antiecho
SF 201.2053681 MHz
WDW States-TPPI
SSB 2
LB 0 Hz
GB 0

UPH-5_2
13C-HSQC-TOCSY



Current Data Parameters
NAME UPH-5-2a-1
EXPNO 10
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100711
Time 16.20
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hsqcdietgpsisp.2
TD 1024
SOLVENT CDCl3
NS 32
DS 128
SWH 8012.820 Hz
FIDRES 7.825020 Hz
AQ 0.0639476 sec
RG 2050
DW 62.400 usec
DE 20.00 usec
TE 301.9 K
CNST17 -0.3000000
CNST2 145.0000000
d0 0.00000300 sec
D1 1.39999998 sec
d11 0.03000000 sec
D16 0.00020000 sec
D24 0.00086200 sec
d4 0.00172414 sec
D9 0.06100000 sec
DELTA 0.00121840 sec
DELTA1 0.00120800 sec
DELTA2 0.00147014 sec
DELTA3 0.00135810 sec
FACTOR1 32
IN0 0.00001655 sec
l1 24
NTINT 0

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
P2 16.40 usec
P28 1000.00 usec
P6 24.00 usec
PL1 0.20 dB
PL16 9.53 dB
SFO1 800.1828373 MHz

======== CHANNEL f2 ========
CPDPRG2 ptm4sp180
NUC2 13C
P14 500.00 usec
P24 2000.00 usec
P3 14.80 usec
PCPD2 1500.00 usec
PL0 120.00 dB
PL12 10.00 dB
PL2 -2.00 dB
SFO2 201.2205114 MHz
SP3 1.08 dB
SP7 1.08 dB
SPNAM15 Crp,80,1.3,10.ns
SPNAM3 Crp80,0.5,20.1
SPNAM7 Crp80comp.4
SPOAL15 0.500
SPOAL3 0.500
SPOAL7 0.500
SPOFFS15 0 Hz
SPOFFS3 0 Hz
SPOFFS7 0 Hz

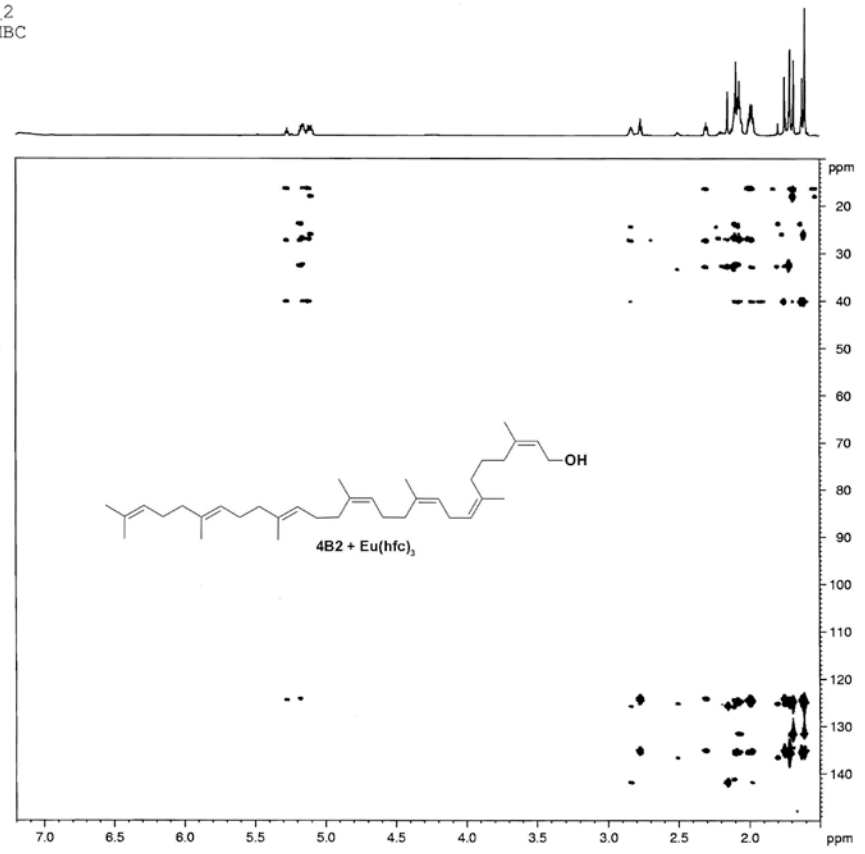
------- GRADIENT CHANNEL -----
GPNAM1 SINE.100
GPNAM2 SINE.100
GPZ10 80.00 %
GPZ2 20.10 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 1024
SFO1 201.2205 MHz
FIDRES 29.503399 Hz
SW 150.141 ppm
FnMODE Echo-Antiecho

F2 - Processing parameters
SI 2048
SF 800.1800190 MHz
WDW QSINE
SSB 2
LB 0 Hz
GB 0
PC 1.00

F1 - Processing parameters
SI 4096
MC2 echo-antiecho
SF 201.2053681 MHz
WDW States-TPPI
SSB 2
LB 0 Hz
GB 0

UPH-5_2
13C-HMBC



Current Data Parameters
NAME UPH-5-2a-1
EXPNO 12
PROCNO 1

F2 - Acquisition Parameters
Date_ 20100712
Time 14.52
INSTRUM spect
PROBHD 5 mm CPTCI 1H-
PULPROG hmbcgpl2ndqf
TD 2048
SOLVENT CDCl3
NS 64
DS 128
SWH 8223.685 Hz
FIDRES 4.015471 Hz
AQ 0.1245684 sec
RG 5790
DW 60.800 usec
DE 30.00 usec
TE 301.9 K
CNST13 8.0000000
CNST6 125.0000000
CNST7 175.0000000
d0 0.00000300 sec
D1 1.39999998 sec
D16 0.00020000 sec
d6 0.06250000 sec
DELTA1 0.00280000 sec
DELTA2 0.00165714 sec
DELTA3 0.06129600 sec
IN0 0.00001310 sec

======== CHANNEL f1 ========
NUC1 1H
P1 8.20 usec
p2 16.40 usec
PL1 0.20 dB
SFO1 800.1828373 MHz

======== CHANNEL f2 ========
NUC2 13C
P3 14.80 usec
PL2 -2.00 dB
SFO2 201.2245355 MHz

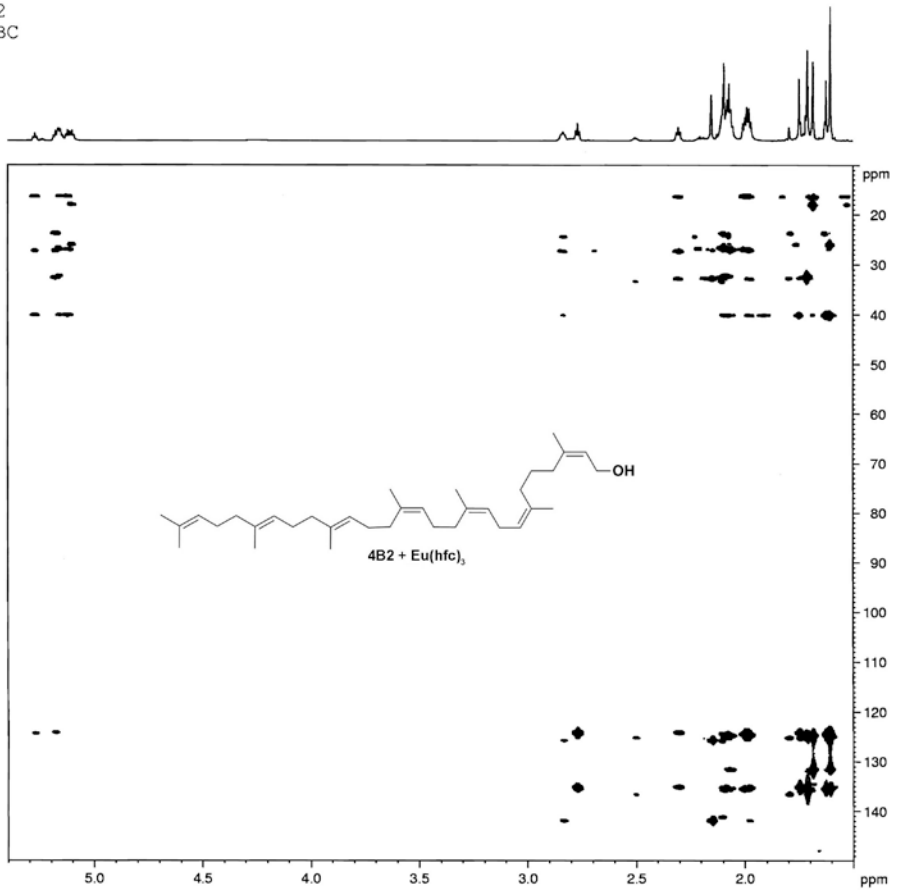
====== GRADIENT CHANNEL =====
GPNAM1 SINE.100
GPNAM2 SINE.100
GPNAM3 SINE.100
GPNAM4 SINE.100
GPNAM5 SINE.100
GPNAM6 SINE.100
GPZ1 50.00 %
GPZ2 30.00 %
GPZ3 40.10 %
GPZ4 15.00 %
GPZ5 -10.00 %
GPZ6 -5.00 %
P16 1000.00 usec

F1 - Acquisition parameters
TD 512
SFO1 201.2245 MHz
FIDRES 74.546753 Hz
SW 189.678 ppm
FnMODE QF

F2 - Processing parameters
SI 2048
SF 800.1800169 MHz
WDW QSINE
SSB 4
LB 0 Hz
GB 0
PC 1.40

F1 - Processing parameters
SI 4096
MC2 QF
SF 201.2053682 MHz
WDW States-TPPI
SSB 4
LB 0 Hz
GB 0

UPH-5_2
13C-HMBC

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Current Data Parameters
NAME                 UPH-5-2a-1
EXPNO                        12
PROCNO                        1

F2 - Acquisition Parameters
Date_              20100712
Time                    14.52
INSTRUM                 spect
PROBHD    5 mm CPTCI 1H-
PULPROG      hmbcgpl2ndqf
TD                        2048
SOLVENT                 CDCl3
NS                           64
DS                          128
SWH                 8223.685 Hz
FIDRES              4.015471 Hz
AQ                  0.1245684 sec
RG                       5790
DW                     60.800 usec
DE                     30.00 usec
TE                     301.9 K
CNST13               8.0000000
CNST6              125.0000000
CNST7              175.0000000
d0                   0.00000300 sec
D1                   1.39999998 sec
D16                  0.00020000 sec
d6                   0.06250000 sec
DELTA1               0.00280000 sec
DELTA2               0.00165714 sec
DELTA3               0.06129600 sec
IN0                  0.00001310 sec

======== CHANNEL f1 ========
NUC1                       1H
P1                       8.20 usec
p2                      16.40 usec
PL1                      0.20 dB
SFO1               800.1828373 MHz

======== CHANNEL f2 ========
NUC2                      13C
P3                      14.80 usec
PL2                     -2.00 dB
SFO2               201.2245355 MHz

====== GRADIENT CHANNEL =====
GPNAM1                 SINE.100
GPNAM2                 SINE.100
GPNAM3                 SINE.100
GPNAM4                 SINE.100
GPNAM5                 SINE.100
GPNAM6                 SINE.100
GPZ1                      50.00 %
GPZ2                      30.00 %
GPZ3                      40.10 %
GPZ4                      15.00 %
GPZ5                     -10.00 %
GPZ6                      -5.00 %
P16                    1000.00 usec

F1 - Acquisition parameters
TD                           512
SFO1               201.2245 MHz
FIDRES             74.546753 Hz
SW                   189.678 ppm
FnMODE                       QF

F2 - Processing parameters
SI                         2048
SF                 800.1800169 MHz
WDW                       QSINE
SSB                           4
LB          0 Hz
GB          0
PC                         1.40

F1 - Processing parameters
SI                         4096
MC2                          QF
SF                 201.2053682 MHz
WDW                  States-TPPI
SSB                           4
LB          0 Hz
GB          0
```