

Catalytic Enantioselective Allylation of Dienals Through the Intermediacy of Unsaturated π -Allyl Complexes

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Supporting Information

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General Information

^1H NMR spectra were recorded on either a Varian Gemini-400 (400 MHz), or a Varian Inova-500 (500 MHz) spectrometer. Chemical shifts are reported in ppm with the solvent resonance as the internal standard (CDCl_3 : 7.26 ppm). Data are reported as follows: chemical shift, integration, multiplicity (s = singlet, d = doublet, t = triplet, q = quartet, p = pentet, br = broad, m = multiplet), coupling constants (Hz), and assignment. ^{13}C NMR spectra were recorded on either a Varian Gemini-400 (100 MHz), or a Varian Inova-500 (125 MHz) spectrometer with complete proton decoupling. Chemical shifts are reported in ppm with the solvent resonance as the internal standard (CDCl_3 : 77.23 ppm). Infrared (IR) spectra were recorded on a Bruker alpha spectrophotometer, ν_{max} cm^{-1} . Bands are characterized as broad (br), strong (s), medium (m), and weak (w). High resolution mass spectrometry (ESI) was performed at the Mass Spectrometry Facility, Boston College.

Liquid Chromatography was performed using forced flow (flash chromatography) on silica gel (SiO_2 , 230 \times 450 Mesh) purchased from Silicycle. Thin Layer Chromatography was performed on 25 μm silica gel plates purchased from Silicycle. Visualization was performed using ultraviolet light (254 nm), phosphomolybdic acid (PMA) in ethanol, or potassium permanganate (KMnO_4) in water. Analytical chiral gas-liquid chromatography (GLC) was performed on a Hewlett-Packard 6890 Series chromatograph equipped with a split mode capillary injection system, a flame ionization detector, and a Supleco β -Dex 120 column with helium as the carrier gas. Analytical chiral supercritical fluid chromatography (SFC) was performed on a Berger Instruments Supercritical Chromatograph equipped with an Alcott auto sampler and a Knauer UV detector with methanol as the modifier. Analytical high performance liquid chromatography (HPLC) was performed on a Shimadzu chromatography equipped with two LC-10APvp pumps, SPD-10AVvp UV detector and SIL-10ADvp injector. Optical rotations were measured on a Rudolph Analytical Research Autopol IV Polarimeter.

All reactions were conducted in oven- or flame-dried glassware under an inert atmosphere of nitrogen or argon. Tetrahydrofuran (THF) and dichloromethane (DCM) were purified using a Pure Solv MD-4 solvent purification system from Innovative Technology Inc. Bis(1,5-cyclooctadiene)nickel(0) [$\text{Ni}(\text{cod})_2$] and tricyclohexylphosphine (PCy_3) were purchased from Strem Chemicals, Inc. Acetic acid and dimethyl malonate were distilled under reduced pressure. Hoveyda-Grubbs catalyst second generation (HG-II) refers to [1,3-Bis(2,4,6-trimethylphenyl)-2-imidazolidinylidene] dichloro(*o*-isopropoxyphenylmethylene)ruthenium.¹ All other reagents were purchased from either Fisher or Aldrich and used without further purification. (*R,R*)- $^t\text{BuTADDOLPPh}$ (**L1**)², (*R,R*)-xylylTADDOLPPh (**L2**)¹, (*R,R*)-TADDOLPPh (**L3**)¹, (*R,R*)-

¹ Garber, S.B.; Kingsbury, J.S.; Gray, B.L.; Hoveyda, A.H. *J. Am. Chem. Soc.* **2000**, *122*, 8168.

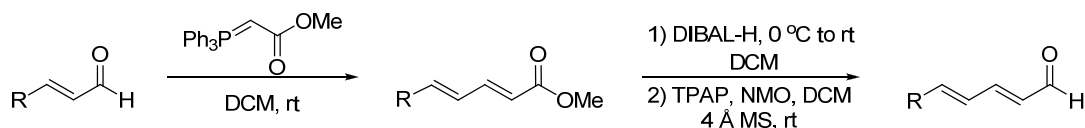
² Sieber, J. D.; Morken, J. P. *J. Am. Chem. Soc.* **2008**, *130*, 4978.

TADDOLPN(C₄H₈) (**L4**)³ and (*R,R*)-TADDOLPN(C₄H₈) (**L5**)² were prepared according to literature procedures.

Experimental Procedures

Preparation of Dienals

Representative Procedure for the Synthesis of Dienals

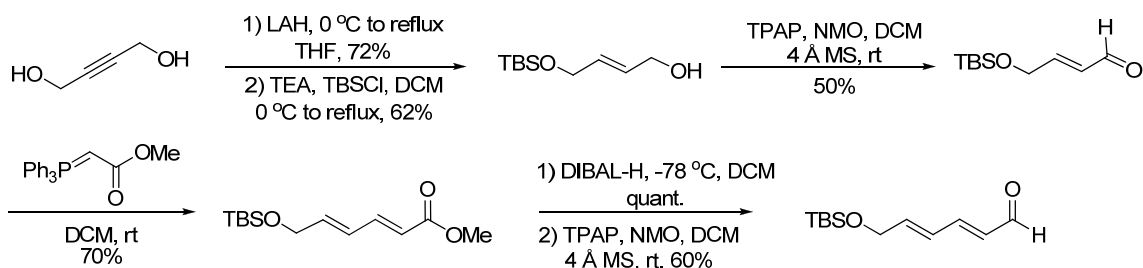


The following dienals were prepared from commercially available α,β -unsaturated aldehydes: (*2E,4E*)-deca-2,4-dienal (Table 2, entries 3 and 4)⁴ and (*2E,4E*)-phenylpenta-2,4-dienal (Table 2, entry 5)³. Spectral data are in accordance with the literature references.

(*2E,4E*)-5-cyclohexylpenta-2,4-dienal was prepared from (*E*)-3-cyclohexylacrylaldehyde (Table 2, entry 8), which was originally synthesized from cyclohexanecarboxaldehyde according to general procedure. Spectral data are in accordance with the literature reference.³

(*2E,4E*)-5-(furan-2-yl)penta-2,4-dienal (Table 2, entry 9) was prepared according to the literature procedure.⁵ Spectral data are in accordance with the literature reference.

Preparation of (*2E,4E*)-6-(*tert*-butyldimethylsilyloxy)hexa-2,4-dienal



(*2E,4E*)-6-(*tert*-butyldimethylsilyloxy)hexa-2,4-dienal (Table 2, entry 7). A light yellow oil. $R_f = 0.48$ (3:1 hexane:EtOAc); ¹H NMR (400 Hz,

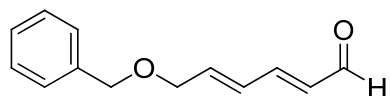
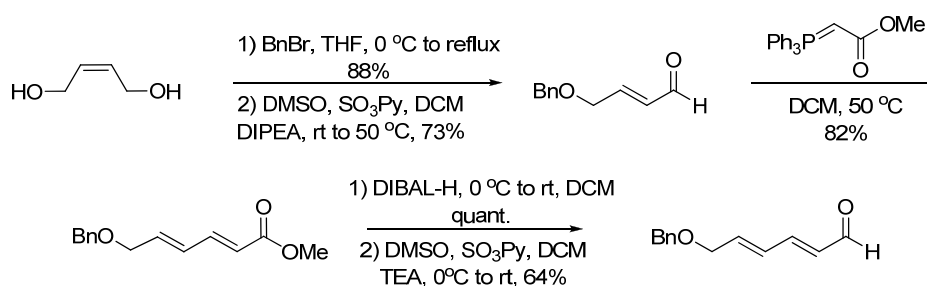
³ Burks, H. E.; Morken, J. P. *J. Am. Chem. Soc.* **2007**, *129*, 8766.

⁴ Rein, T.; Kann, N.; Åkermark, B.; Helquist, P. *J. Org. Chem.* **1990**, *55*, 5312.

⁵ Bellassoued, M.; Salemkour, M. *Tetrahedron.* **1996**, *52*, 4507.

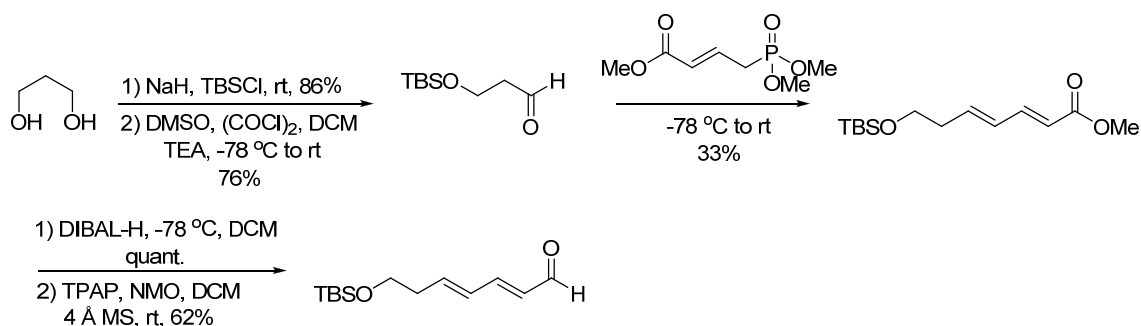
CDCl₃): δ 0.09 (6H, s, Si(CH₃)₂), 0.93 (9H, s, SiC(CH₃)₃), 4.34 (2H, dd, $J = 4.2$ Hz, 1.8 Hz, SiOCH₂CH=CH), 6.15 (1H, dd, $J = 15.2$ Hz, 8.0 Hz, C(O)HCH=CH), 6.32 (1H, dt, $J = 15.2$ Hz, 4.0 Hz, SiOCH₂CH=CH), 6.56 (1H, ddt, $J = 15.2$ Hz, 10.8 Hz, 2.0 Hz, SiOCH₂CH=CH), 7.13 (1H, dd, $J = 15.4$ Hz, 11.0 Hz, C(O)HCH=CH), 9.57 (1H, d, $J = 8.4$ Hz, C(O)H); ¹³C NMR (100 Hz, CDCl₃): δ 193.9, 151.7, 144.4, 131.5, 127.0, 63.1, 26.1, 18.7, -5.05 ppm; IR (neat): 2954.7 (m), 2930.0 (m), 2886.1 (w), 2856.8 (w), 2728.7 (m), 1684.6 (s), 1645.7 (s), 1602.7 (w), 1643.4 (w), 1362.1 (w), 1264.2 (m), 1161.5 (m), 1131.98 (s) cm⁻¹; HRMS (ESI+) for C₁₂H₂₃O₂Si [M+H]: calculated 227.1467, found: 227.1475.

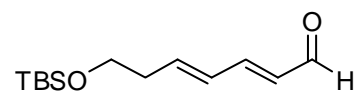
Preparation of (2E,4E)-6-(benzyloxy)hexa-2,4-dienal



(2E,4E)-6-(benzyloxy)hexa-2,4-dienal (Table 2, entry 6). A yellow oil. $R_f = 0.37$ (3:1 hexane:EtOAc); ¹H NMR (400 Hz, CDCl₃): δ 4.18 (2H, d, $J = 5.2$ Hz, OCH₂CH=CH), 4.57 (2H, s, PhCH₂O), 6.16 (1H, dd, $J = 15.2$ Hz, 8.0 Hz, C(O)HCH=CH), 6.32 (1H, dt, $J = 15.2$ Hz, 5.2 Hz, OCH₂CH=CH), 6.58 (1H, ddm, $J = 15.2$ Hz, 10.8 Hz, OCH₂CH=CH), 7.13 (1H, dd, $J = 15.4$ Hz, 11.0 Hz, C(O)HCH=CH), 7.31-7.36 (5H, m, Ph-H), 9.58 (1H, d, $J = 8.0$ Hz, C(O)H); ¹³C NMR (125 Hz, CDCl₃): δ 194.0, 151.4, 141.2, 137.9, 132.0, 126.2, 128.7, 128.1, 128.0, 73.1, 69.7 ppm; IR (neat): 3030.9 (w), 2845.9 (br), 2735.3 (w), 1678.8 (s), 1643.4 (s), 1602.1(w), 1469.6 (w), 1453.3 (m), 1391.1 (w), 1360.4 (m), 1161.9 (m), 1101.9 (s) cm⁻¹; HRMS (ESI+) for C₁₃H₁₅O₂ [M+H]: calculated 203.1072, found: 203.1082.

Preparation of (2E,4E)-7-(tert-butyldimethylsilyloxy)hepta-2,4-dienal




(2E,4E)-7-(tert-butyldimethylsilyloxy)hepta-2,4-dienal (Table 2, entry 10). A yellow oil. $R_f = 0.65$ (3:1 hexane:EtOAc); $^1\text{H NMR}$ (400 Hz, CDCl_3): δ 0.05 (6H, s, $\text{Si}(\text{CH}_3)_2$), 0.89 (9H, s, $\text{SiC}(\text{CH}_3)_3$), 2.43 (2H, q, $J = 6.4$ Hz, $\text{CH}_2\text{CH}_2\text{CH}=\text{CH}$), 3.73 (2H, t, $J = 6.4$ Hz, $\text{CH}_2\text{CH}_2\text{CH}=\text{CH}$), 6.09 (1H, dd, $J = 15.2$ Hz, 8.0 Hz, $\text{C}(\text{O})\text{HCH}=\text{CH}$), 6.26-6.41 (2H, m, $\text{CH}_2\text{CH}=\text{CH}$ and $\text{CH}_2\text{CH}=\text{CH}$), 7.09 (1H, dd, $J = 15.2$ Hz, 10.0 Hz, $\text{C}(\text{O})\text{HCH}=\text{CH}$), 9.55 (1H, d, $J = 7.6$ Hz, $\text{C}(\text{O})\text{H}$); $^{13}\text{C NMR}$ (100 Hz, CDCl_3): δ 194.0, 152.6, 143.7, 130.6, 130.4, 62.1, 36.9, 26.1, 18.6, -5.0 ppm; IR (neat): 2953.4 (m), 2928.7 (m), 2885.7 (w), 2856.8 (m), 2738.5 (w), 1685.1 (s), 1640.9 (s), 1600.3 (w), 1471.1 (w), 1289.1 (w), 1254.8 (m), 1098.7 (s), 936.7 (m) cm^{-1} ; HRMS (ESI+) for $\text{C}_{13}\text{H}_{25}\text{O}_2\text{Si}$ [$\text{M}+\text{H}$]: calculated 241.1624, found: 241.1633.

Procedure for Non-Catalyzed Allylation (Scheme 1)

An oven-dried 2-dram vial equipped with a magnetic stir-bar was charged with 33.7 mg (0.351 mmol) of sorbic aldehyde in a dry-box under an argon atmosphere, followed by 0.70 mL of THF and 70.7 mg (0.421 mmol) of allylboronic acid pinacol ester, sequentially. The vial was capped, taped with electrical tape, removed from the dry-box, and allowed to stir at ambient temperature for 15 hours. After this time period, deionized water was added and the mixture was allowed to stir for another 10 minutes. The aqueous layer was washed with CH_2Cl_2 ($\times 3$), and the combined organic layers were dried with Na_2SO_4 and concentrated in vacuo. Analysis of the unpurified reaction mixture using $^1\text{H NMR}$ was used to determine $[E,Z]:[E,E]$ ratio.

Representative Procedure for Ni-Catalyzed Allylation at Ambient Temperature (Scheme 1 and Table 1)

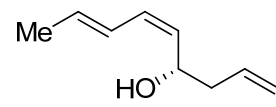
An oven-dried 2-dram vial equipped with a magnetic stir-bar was charged with 9.6 mg (0.0351 mmol) of bis(1,5-cyclooctadiene)nickel, 20.1 mg (0.0351 mmol) of chiral ligand **L3**, and 0.70 mL of THF in a dry-box under an argon atmosphere. The vial was capped and stirred for 5 minutes. Next, 33.7 mg (0.351 mmol) of sorbic aldehyde was added, followed by 70.7 mg (0.421 mmol) of allylboronic acid pinacol ester. The vial was capped, taped with electrical tape, removed from the dry-box, and allowed to stir at ambient temperature for 18 hours. After this time period, deionized water was added and the mixture was allowed to stir for another 10 minutes. The aqueous layer was washed with CH_2Cl_2 ($\times 3$), and the combined organic layers were dried with Na_2SO_4 and concentrated in vacuo. Analysis of the unpurified reaction mixture using $^1\text{H NMR}$ was used to determine $[E,Z]:[E,E]$ ratio. Silica gel chromatography (10:1 hexane:EtOAc) afforded 37.0 mg (77%) of a light yellow oil of the allylation product as a mixture of isomers.

Representative Procedure for Ni-Catalyzed Allylation at -35 °C (Table 2)

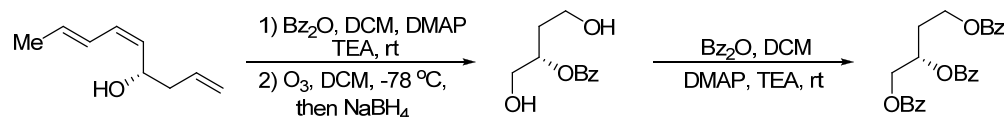
In a dry-box freezer: An oven-dried 2-dram vial equipped with a magnetic stir-bar was charged with 9.6 mg (0.0351 mmol) of bis(1,5-cyclooctadiene)nickel, 20.1 mg (0.0351 mmol) of chiral ligand **L3**, and 0.70 mL of THF in a dry-box under an argon atmosphere. The vial was capped and stirred for 5 minutes. Next, 33.7 mg (0.351 mmol) of sorbic aldehyde was added, and the vial was capped and put into the freezer (temperature: -35 °C) inside the dry-box without stirring. Meanwhile, a syringe containing 70.7 mg (0.421 mmol) of allylboronic acid pinacol ester was put into the same freezer. After 30 minutes, the allylboronic acid pinacol ester was quickly transferred to the reaction vial and the vial resealed. The reaction was kept in the dry-box freezer for 18 hours, and deionized water was added. After stirring for another 10 minutes, the aqueous layer was washed with CH₂Cl₂ (×3), and the combined organic layers were dried with Na₂SO₄ and concentrated in vacuo. Analysis of the unpurified reaction mixture using ¹H NMR was used to determine [*E,Z*]:[*E,E*] ratio. Silica gel chromatography (10:1 hexane:EtOAc) afforded 27.1 mg (56%) of a light yellow oil of the allylation product as a mixture of isomers.

In a cryo-cool: An oven-dried 6-dram vial equipped with a magnetic stir-bar was charged with 9.6 mg (0.035 mmol) of bis(1,5-cyclooctadiene)nickel, 20.1 mg (0.035 mmol) of chiral ligand **L3**, and 0.70 mL of THF in a dry-box under an argon atmosphere. The vial was capped and stirred for 5 minutes. To a separate oven-dried 1-mL vial was added 33.7 mg (0.351 mmol) of sorbic aldehyde. To a third oven-dried 1-mL vial was added 117 mg (0.701 mmol) of allylboronic acid pinacol ester. After removal of the magnetic stir-bar from the 6-dram vial, the two uncapped 1-mL vials were transferred carefully into the 6-dram vial without mixing the reaction with the two reagents. The vial was capped, taped with electrical tape, removed from the dry-box, and cooled in a cryo-cool at -35 °C. After 30 minutes, the vial was gently shaken to mix the contents of the three vials and then put back to the cryo-cool for another 18 hours. After this time period, 0.6 mL (1.2 mmol) of acetaldehyde was added, followed by warming to ambient temperature over 30 minutes. Deionized water was added, and the mixture was lightly shaken for another 10 minutes. The aqueous layer was washed with CH₂Cl₂ (×3), and the combined organic layers were dried with Na₂SO₄ and concentrated in vacuo. Analysis of the unpurified reaction mixture using ¹H NMR was used to determine [*E,Z*]:[*E,E*] ratio. Silica gel chromatography (10:1 hexane:EtOAc) afforded 33.9 mg (70%) of a light yellow oil of the allylation product as a mixture of isomers.

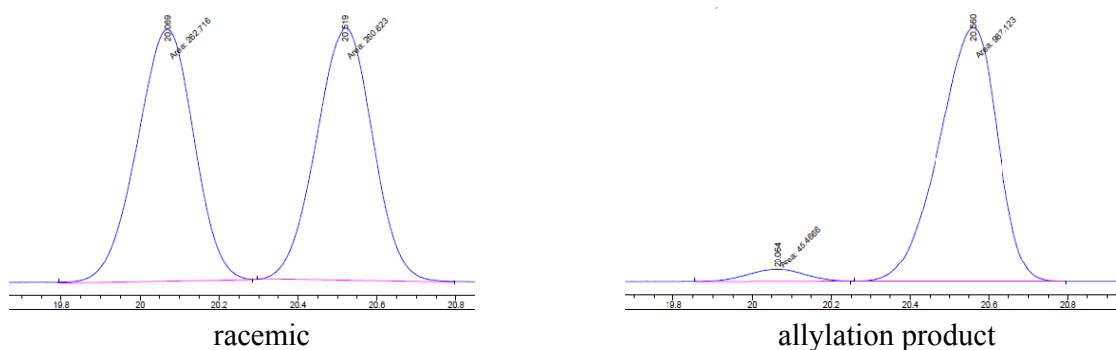
Characterization and Proof of Stereochemistry


(*S,5Z,7E*)-nona-1,5,7-trien-4-ol. A light yellow oil. Single isomer. $R_f = 0.42$ (3:1 hexane:EtOAc); $^1\text{H NMR}$ (400 Hz, CDCl_3): δ 1.60 (1H, d, $J = 3.6$ Hz, OH), 1.79 (3H, dd, $J = 6.4$ Hz, 1.6 Hz, CH_3), 2.32 (2H, ddt, $J = 6.8, 6.0, 1.2$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 4.63 (1H, dddd, $J = 8.8, 6.0, 6.0, 3.2$ Hz, CHOH), 5.12-5.18 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.30 (1H, dd, $J = 10.8, 8.8$ Hz, $\text{CH}=\text{CHCHOH}$), 5.73-5.88 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$ and $\text{CH}_3\text{CH}=\text{CH}$), 6.04 (1H, dd, $J = 11.2, 10.8$ Hz, $\text{CH}=\text{CHCHOH}$), 6.35 (1H, ddq, $J = 14.8, 11.4, 1.2$ Hz, $\text{CH}_3\text{CH}=\text{CH}$); $^{13}\text{C NMR}$ (100 Hz, CDCl_3): δ 134.3, 132.1, 130.7, 130.4, 126.6, 118.3, 67.3, 42.3, 18.5 ppm; IR (neat): 3349.4 (br), 3076.5 (w), 3021.8 (m), 2978.7 (w), 2914.2 (m), 2852.4 (m), 1654.9 (m), 1641.0 (m), 1433.7 (m), 1376.2 (m), 1306.3 (m), 1019.6 (s), 982.2 (s), 946.3 (s), 912.7 (s) cm^{-1} ; HRMS (ESI+) for C_9H_{13} [$\text{M}+\text{H}-\text{H}_2\text{O}$]: calculated 121.1017, found: 121.1017; $[\alpha]_D^{20} = -2.3$ ($c = 0.42, \text{CHCl}_3$).

Proof of Stereochemistry: Enantioselectivities were determined by comparison of the acylated product with authentic racemic material prepared using tricyclohexylphosphine as the achiral ligand in the allylation reaction. Absolute stereochemistry was determined by converting the allylation product to benzoate, followed by ozonolysis/reduction, and converting the corresponding diol to tribenzoate, as shown below. The resulting tribenzoate was compared on chiral SFC to butane-1,2,4-triyl tribenzoate and (*S*)-butane-1,2,4-triyl tribenzoate which were derived from commercially available butane-1,2,4-triol and (*S*)-1,2,4-triol respectively.

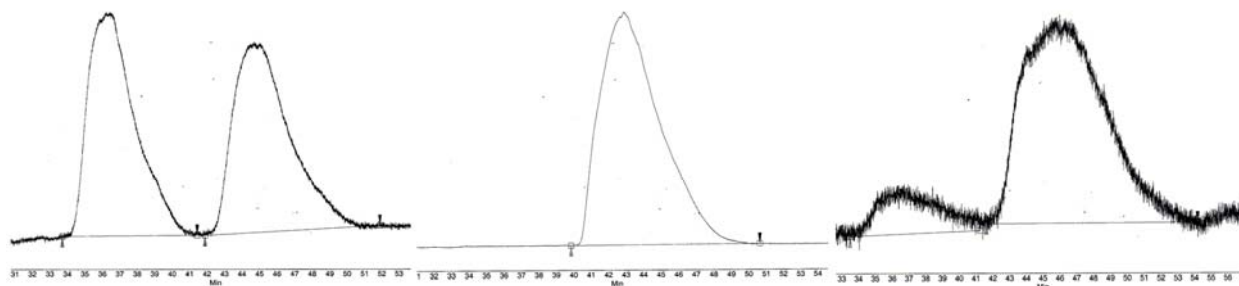


Chiral GLC (β -dex, supelco, 100 °C, 20 psi) - analysis of the acetate of (*SZ,7E*)-nona-1,5,7-trien-4-ol



Peak #	RetTime [min]	Type	Width [min]	Area [pA*s]	Height [pA]	Area %
1	20.064	MM	0.1692	45.46656	4.47819	4.40316
2	20.560	MM	0.1709	987.12329	96.24561	95.59684

Chiral SFC (OD-H, Chiralpak, 220 nm, 3.0 mL/min, 3.0% MeOH, 150 psi, 50 °C) – analysis of butane-1,2,4-triyl tribenzoate



racemic tribenzoate

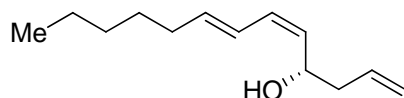
Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
33.71	36.39	41.43	0.00	49.69	572.5	1800.0	49.694
41.88	44.69	51.89	0.00	50.31	489.7	1822.1	50.306
				100.00	1062.2	3622.1	100.000

S-tribenzoate

Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
39.85	42.98	50.63	0.00	100.00	7144.1	28677.1	100.000
				100.00	7144.1	28677.1	100.000

tribenzoate derived from allylation product

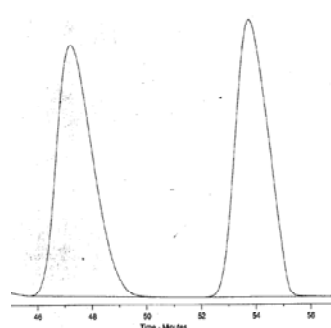
Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
33.50	36.39	41.24	0.00	14.00	64.1	273.1	14.001
41.62	45.73	54.18	0.00	86.00	292.2	1677.2	85.999
				100.00	356.3	1950.3	100.000



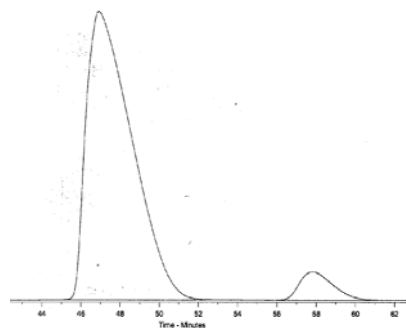
(*S,5Z,7E*)-trideca-1,5,7-trien-4-ol. A colorless oil. Mixture of isomers (24:1 [*E,Z*]:[*E,E*]). $R_f = 0.59$ (3:1 hexane:EtOAc); ^1H NMR (400 Hz, CDCl_3): δ 0.88 (3H, t, $J = 6.8$ Hz, CH_3), 1.26-1.41 (6H, m, $\text{CH}_3(\text{CH}_2)_3$), 1.71 (1H, s, OH), 2.10 (2H, q, $J = 7.2$ Hz, $\text{CH}_2\text{CH}=\text{CH}$), 2.32 (2H, t, $J = 6.8$ Hz, CH_2CHOH), 4.62 (1H, dt, $J = 8.0, 6.4$ Hz, CHOH), 5.11-5.17 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.30 (1H, dd, $J = 10.4, 9.2$ Hz, $\text{CH}=\text{CHCHOH}$), 5.71-5.87 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$ and $\text{CH}_2\text{CH}=\text{CH}$), 6.03 (1H, t, $J = 11.0$ Hz, $\text{CH}=\text{CHCHOH}$), 6.31 (1H, ddt, $J = 14.8, 11.2, 0.8$ Hz, $\text{CH}_2\text{CH}=\text{CH}$); ^{13}C NMR (100 Hz, CDCl_3): δ 137.8, 134.3, 130.9, 130.5, 125.2, 118.3, 67.3, 42.3, 33.0, 31.7, 29.1, 22.7, 14.3 ppm; IR (neat): 3367.7 (br), 3076.4 (m), 2956.4 (s), 2925.4 (m), 2856.7 (m), 1692.3 (m), 1641.0 (m), 1458.8 (m), 1433.1 (m), 1378.4 (m), 1307.7 (m), 1021.2 (s), 984.4 (s), 946.9 (s), 912.2 (s) cm^{-1} ; HRMS (ESI+) for $\text{C}_{13}\text{H}_{21}$ [$\text{M}+\text{H}-\text{H}_2\text{O}$]: calculated 177.1643, found: 177.1639; $[\alpha]_D^{20} = -1.2$ ($c = 1.41$, CHCl_3).

Proof of Stereochemistry: Enantioselectivities were determined by comparison with authentic racemic material prepared using tricyclohexylphosphine as the achiral ligand in the allylation reaction. Absolute stereochemistry was determined by converting the allylation product to butane-1,2,4-triyl tribenzoate as described for (*S,5Z,7E*)-nona-1,5,7-trien-4-ol. The resulting tribenzoate was compared on chiral SFC to butane-1,2,4-triyl tribenzoate and (*S*)-butane-1,2,4-triyl tribenzoate derived from commercially available butane-1,2,4-triol and (*S*)-1,2,4-triol, respectively.

Chiral HPLC (AS, Chiralcel, 0.5 mL/min, 0% Isopropanol, 220 nm) – analysis of (*5Z,7E*)-trideca-1,5,7-trien-4-ol



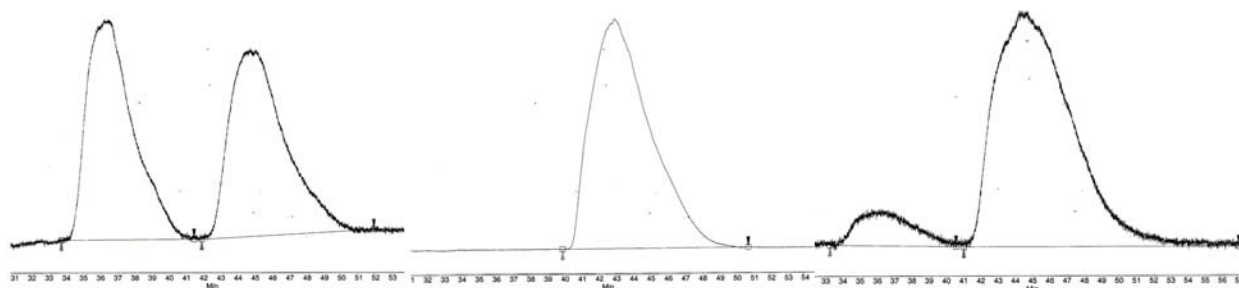
Racemic



allylation product

Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	46.94		0.00	N/A	100919500	93.288	BB	2.64
2	57.83		0.00	N/A	7261621	6.712	BB	1.88

Chiral SFC (OD-H, Chiralpak, 220 nm, 3.0 mL/min, 3.0% MeOH, 150 psi, 50 °C) – analysis of butane-1,2,4-triyl tribenzoate



racemic tribenzoate

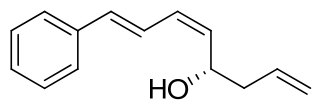
S-tribenzoate

tribenzoate derived from allylation product

Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
33.71	36.39	41.43	0.00	49.69	572.5	1800.0	49.694
41.88	44.69	51.89	0.00	50.31	489.7	1822.1	50.306
				100.00	1062.2	3622.1	100.000

Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
39.85	42.90	50.63	0.00	100.00	7144.1	26077.1	100.000
				100.00	7144.1	26077.1	100.000

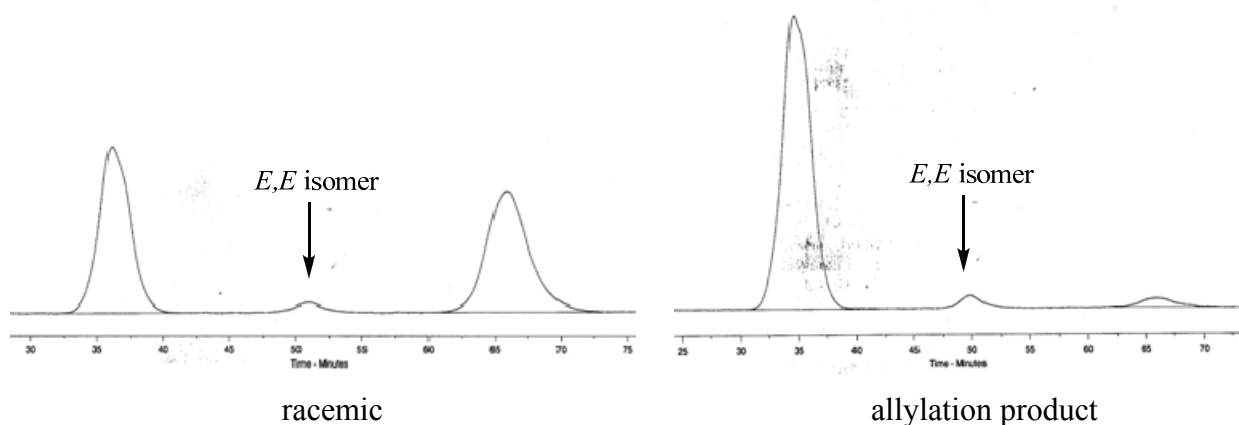
Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [μV.Min]	Area [%]
33.25	35.94	40.55	0.00	9.83	130.9	516.3	9.829
41.01	44.37	56.98	0.00	90.17	903.5	4736.3	90.171
				100.00	1034.4	5252.6	100.000



(*S,5Z,7E*)-8-phenylocta-1,5,7-trien-4-ol. A yellow oil. Mixture of isomers (24:1 [*E,Z*]:[*E,E*]). $R_f = 0.33$ (3:1 hexane:EtOAc); $^1\text{H NMR}$ (400 Hz, CDCl_3): δ 1.87 (1H, s, OH), 2.38 (2H, t, $J = 6.8$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 4.76 (1H, dt, $J = 8.0, 6.8$ Hz, CHOH), 5.14-5.21 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.51 (1H, dd, $J = 10.8, 8.8$ Hz, $\text{CH}=\text{CHCHOH}$), 5.85 (1H, ddt, $J = 17.2, 10.0, 7.2$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 6.24 (1H, t, $J = 11.2$ Hz, $\text{CH}=\text{CHCHOH}$), 6.59 (1H, d, $J = 15.2$, PhCH=CH), 7.07 (1H, dd, $J = 15.6, 11.2$ Hz, PhCH=CH), 7.25 (1H, t, $J = 7.2$ Hz, para-Ph-H), 7.33 (2H, dd, $J = 8.0, 7.2$ Hz, meta-Ph-H), 7.42 (2H, dd, $J = 8.0, 0.8$ Hz, ortho-Ph-H); $^{13}\text{C NMR}$ (100 Hz, CDCl_3): δ 137.1, 134.6, 134.1, 133.3, 130.6, 128.8, 128.0, 128.6, 123.8, 118.5, 67.5, 42.3 ppm; IR (neat): 3361.3 (br), 3077.1 (m), 3027.4 (m), 2978.4 (m), 2906.9 (m), 1638.9 (m), 1597.4 (w), 1493.4 (m), 1448.8 (m), 1306.3 (m), 989.2 (s), 946.1 (s), 916.2 (s), 858.4 (s) cm^{-1} ; HRMS (ESI+) for $\text{C}_{14}\text{H}_{15}$ [$\text{M}+\text{H}-\text{H}_2\text{O}$]: calculated 183.1174, found: 183.1177; $[\alpha]_{\text{D}}^{20} = +3.22$ ($c = 1.41$, CHCl_3).

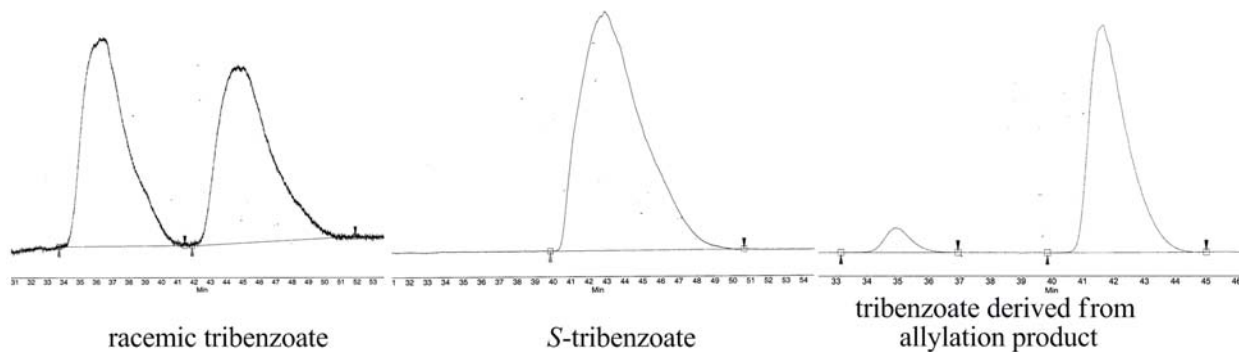
Proof of Stereochemistry: Enantioselectivities were determined by comparison with authentic racemic material prepared using tricyclohexylphosphine as the achiral ligand in the allylation reaction. Absolute stereochemistry was determined by converting the allylation product to butane-1,2,4-triyl tribenzoate as described for (*S,5Z,7E*)-nona-1,5,7-trien-4-ol. The resulting tribenzoate was compared on chiral SFC to butane-1,2,4-triyl tribenzoate and (*S*)-butane-1,2,4-triyl tribenzoate derived from commercially available butane-1,2,4-triol and (*S*)-1,2,4-triol, respectively.

Chiral HPLC (OD, Chiralcel, 1.0 mL/min, 1.0% Isopropanol, 220 nm) – analysis of (*5Z,7E*)-8-phenylocta-1,5,7-trien-4-ol



Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	34.73		0.00	N/A	9043018	96.063	BB	2.68
2	65.83		0.00	N/A	370651	3.937	BB	3.31

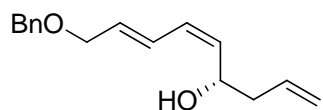
Chiral SFC (OD-H, Chiralpak, 220 nm, 3.0 mL/min, 3.0% MeOH, 150 psi, 50 °C) – analysis of butane-1,2,4-triyl tribenzoate



Start	Time	End	RT Offset	Quantity	Height	Area	Area
[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
33.71	36.39	41.43	0.00	49.69	572.5	1800.0	49.694
41.88	44.69	51.89	0.00	50.31	489.7	1822.1	50.306
				100.00	1062.2	3622.1	100.000

Start	Time	End	RT Offset	Quantity	Height	Area	Area
[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
39.85	42.90	50.53	0.00	100.00	7144.1	26077.1	100.000
				100.00	7144.1	26077.1	100.000

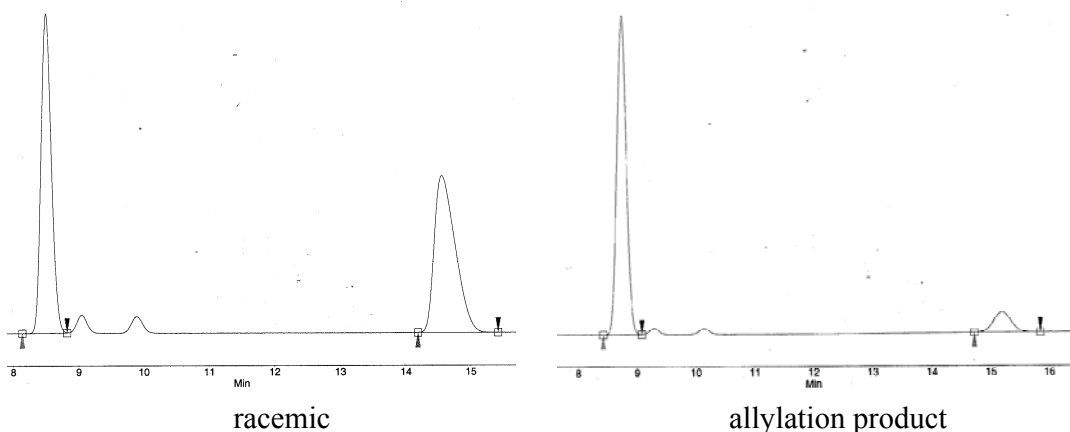
Start	Time	End	RT Offset	Quantity	Height	Area	Area
[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
33.16	34.93	36.96	0.00	7.02	157.6	156.7	7.025
39.86	41.67	45.01	0.00	92.98	1470.5	2074.2	92.975
				100.00	1628.2	2231.0	100.000



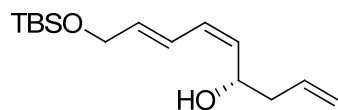
(S,5Z,7E)-9-(benzyloxy)nona-1,5,7-trien-4-ol. A yellow oil. Mixture of isomers (18:1 [*E,Z*]:[*E,E*]). $R_f = 0.24$ (3:1 hexane:EtOAc); $^1\text{H NMR}$ (500 Hz, CDCl_3): δ 1.67 (1H, d, $J = 2.5$ Hz, OH), 2.32 (2H, tt, $J = 7.0, 0.5$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 4.09 (2H, d, $J = 4.5$ Hz, $\text{OCH}_2\text{CH}=\text{CH}$), 4.53 (1H, s, PhCH_2O), 4.63 (1H, m, CHOH), 5.13-5.18 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.44 (1H, dd, $J = 10.5, 9.5$ Hz, $\text{CH}=\text{CHCHOH}$), 5.77-5.88 (2H, m, $\text{OCH}_2\text{CH}=\text{CH}$ and $\text{CH}_2\text{CH}=\text{CH}_2$), 6.09 (1H, t, $J = 11.5$ Hz, $\text{CH}=\text{CHCHOH}$), 6.58 (1H, ddd, $J = 15.5, 12.0, 1.5$ Hz, $\text{OCH}_2\text{CH}=\text{CH}$), 7.28-7.36 (5H, m, Ph-H); $^{13}\text{C NMR}$ (125 Hz, CDCl_3): δ 138.3, 134.1, 133.3, 132.2, 129.8, 128.6, 180.0, 127.9, 127.4, 118.6, 72.5, 70.4, 67.2, 42.2 ppm; IR (neat): 3390.1 (br), 3066.2 (m), 3027.8 (m), 292.8 (m), 2853.4 (m), 1640.5 (m), 143.8 (m), 1358.5 (m), 1305.9 (w), 1206.6 (w), 1102.2 (s), 1046.5 (s), 1027.1 (s), 989.5 (s), 915.6 (s), 737.5 (s) cm^{-1} ; HRMS (ESI+) for $\text{C}_{16}\text{H}_{19}\text{O}$ [$\text{M}+\text{H}-\text{H}_2\text{O}$]: calculated 227.1436, found: 227.1438; $[\alpha]_D^{20} = -4.32$ ($c = 0.66$, CHCl_3).

Proof of Stereochemistry: Enantioselectivities were determined by comparison with authentic racemic material prepared using tricyclohexylphosphine as the achiral ligand in the allylation reaction. The absolute stereochemistry was assigned by analogy.

Chiral SFC (AD-H, Chiralpak, 220 nm, 3.0 mL/min, 3.0% MeOH, 150 psi, 50 °C) – analysis of (5Z,7E)-9-(benzyloxy)nona-1,5,7-trien-4-ol



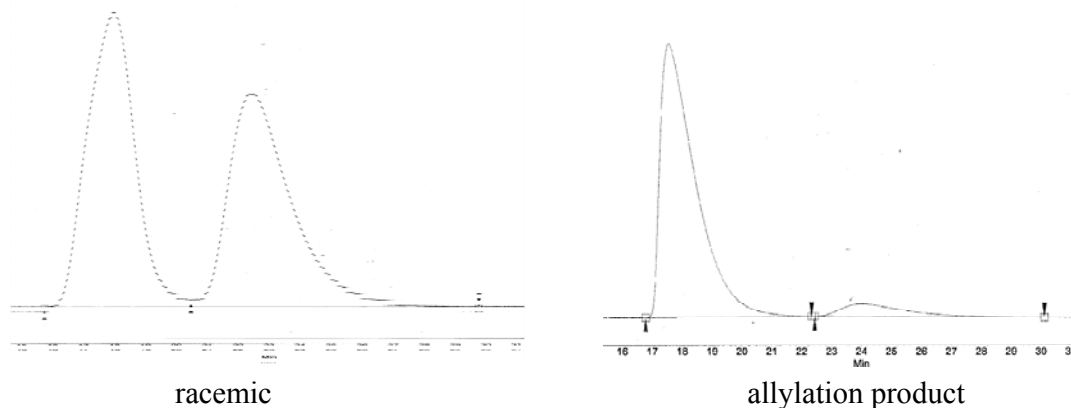
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [$\mu\text{V}\cdot\text{Min}$]	Area [%]
1	UNKNOWN	8.42	8.73	9.08	0.00	89.43	11834.1	2205.7	89.426
2	UNKNOWN	14.71	15.18	15.84	0.00	10.57	733.2	260.8	10.574
Total						100.00	12567.3	2466.5	100.000

**(*S,5Z,7E*)-9-(*tert*-butyldimethylsilyloxy)nona-1,5,7-trien-4-ol.** A

light yellow oil. Single isomer. $R_f = 0.43$ (3:1 hexane:EtOAc); $^1\text{H NMR}$ (400 Hz, CDCl_3): δ 0.08 (6H, s, $\text{Si}(\text{CH}_3)_2$), 0.92 (1H, s, $\text{Si}(\text{CH}_3)_3$), 1.63 (1H, d, $J = 3.2$ Hz, OH), 2.32 (2H, t, $J = 6.8$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 4.24 (2H, dd, $J = 4.4, 1.6$ Hz, $\text{SiOCH}_2\text{CH}=\text{CH}$), 4.64 (1H, dddd, $J = 8.8, 6.4, 6.0, 2.4$ Hz, CHOH), 5.12-5.18 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.40 (1H, dd, $J = 10.8, 8.8$ Hz, $\text{CH}=\text{CHCHOH}$), 5.76-5.86 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$ and $\text{SiOCH}_2\text{CH}=\text{CH}$), 6.08 (1H, dd, $J = 11.2, 10.8$ Hz, $\text{CH}=\text{CHCHOH}$), 6.54 (1H, ddq, $J = 14.0, 11.2, 1.2$ Hz, $\text{SiOCH}_2\text{CH}=\text{CH}$); $^{13}\text{C NMR}$ (100Hz, CDCl_3): δ 135.2, 134.2, 132.5, 129.8, 124.4, 118.4, 67.5, 63.5, 42.3, 26.2, 18.7, -4.93, -4.94 ppm; IR (neat): 3380.1 (br), 3077.2 (w), 3009.9 (m), 2954.4 (m), 2895.7 (m), 2856.4 (m), 1641.2 (w), 1471.5 (m), 1362.4 (m), 1253.7 (m), 1100.9 (br), 1007.2 (br), 831.9 (s), 744.2 (s) cm^{-1} ; HRMS (ESI+) for $\text{C}_{15}\text{H}_{27}\text{OSi}$ [$\text{M}+\text{H}-\text{H}_2\text{O}$]: calculated 251.1831, found: 251.1821; $[\alpha]_D^{20} = -5.94$ ($c = 0.83$, CHCl_3).

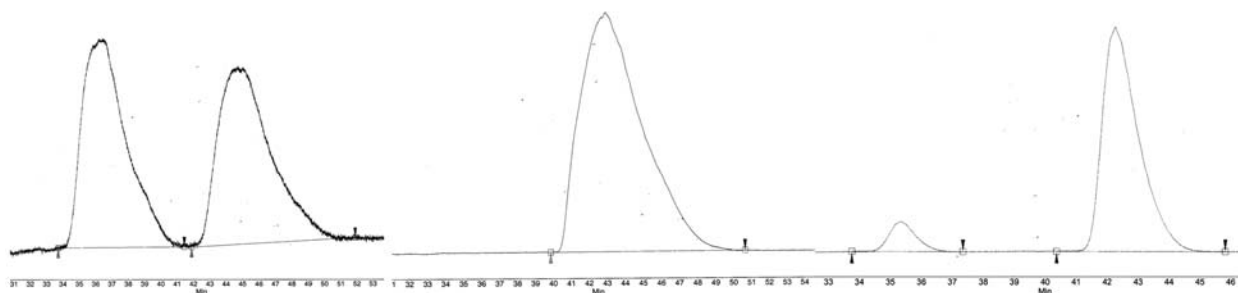
Proof of Stereochemistry: Enantioselectivities were determined by comparison with authentic racemic material prepared using tricyclohexylphosphine as the achiral ligand in the allylation reaction. Absolute stereochemistry was determined by converting the allylation product to butane-1,2,4-triyl tribenzoate as described for (*S,5Z,7E*)-nona-1,5,7-trien-4-ol. The resulting tribenzoate was compared on chiral SFC to butane-1,2,4-triyl tribenzoate and (*S*)-butane-1,2,4-triyl tribenzoate derived from commercial available butane-1,2,4-triol and (*S*)-1,2,4-triol, respectively.

Chiral SFC (AD-H, Chiralpak, 220 nm, 1.0 mL/min, 1.5% MeOH, 150 psi, 50 °C) – analysis of (*S,5Z,7E*)-9-(*tert*-butyldimethylsilyloxy)nona-1,5,7-trien-4-ol



Index	Name	Min				Quantity	Height	Area	Area
		Start	Time	End	RT Offset				
		[Min]	[Min]	[Min]	[Min]	[% Area]	[μV]	[$\mu\text{V}\cdot\text{Min}$]	[%]
1	UNKNOWN	16.79	17.55	22.33	0.00	92.41	5295.4	7256.0	92.408
2	UNKNOWN	22.43	24.04	30.11	0.00	7.59	240.4	596.1	7.592
Total						100.00	5535.8	7852.1	100.000

Chiral SFC (OD-H, Chiralpak, 220 nm, 3.0 mL/min, 3.0% MeOH, 150 psi, 50 °C) – analysis of butane-1,2,4-triyl tribenzoate



racemic tribenzoate

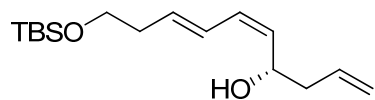
Start	Time	End	RT Offset	Quantity	Height	Area	Area
[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
33.71	36.39	41.43	0.00	49.69	572.5	1800.0	49.694
41.88	44.69	51.89	0.00	50.31	489.7	1822.1	50.306
				100.00	1062.2	3622.1	100.000

S-tribenzoate

Start	Time	End	RT Offset	Quantity	Height	Area	Area
[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
39.85	42.90	50.63	0.00	100.00	7144.1	28977.1	100.000
				100.00	7144.1	28977.1	100.000

tribenzoate derived from allylation product

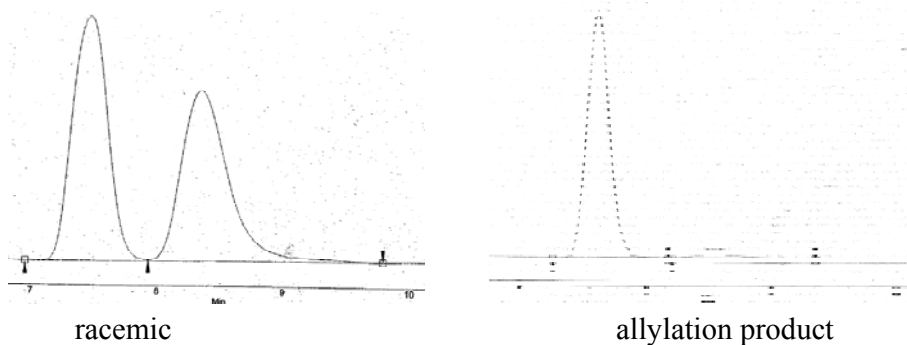
Start	Time	End	RT Offset	Quantity	Height	Area	Area
[Min]	[Min]	[Min]	[Min]	[% Area]	[μ V]	[μ V.Min]	[%]
33.76	35.36	37.35	0.00	8.75	121.7	119.7	8.749
40.37	42.29	45.82	0.00	91.25	921.6	1248.7	91.251
				100.00	1043.3	1368.4	100.000



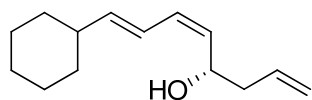
(S,5Z,7E)-10-(tert-butyldimethylsilyloxy)deca-1,5,7-trien-4-ol. A yellow oil. Single isomer. $R_f = 0.51$ (3:1 hexane:EtOAc); $^1\text{H NMR}$ (400 Hz, CDCl_3): δ 0.05 (6H, s, $\text{Si}(\text{CH}_3)_2$), 0.89 (9H, s, $\text{Si}(\text{CH}_3)_3$), 1.61 (1H, d, $J = 4.2$ Hz, OH), 2.33 (4H, m, $\text{CH}_2\text{CH}=\text{CH}_2$ and $\text{CH}_2\text{CH}=\text{CH}$), 3.66 (2H, t, $J = 6.6$ Hz, SiOCH_2), 4.62 (1H, m, CHOH), 5.12-5.18 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.33 (1H, dd, $J = 10.8, 8.8$ Hz, $\text{CH}=\text{CHCHOH}$), 5.71-5.87 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$ and $\text{CH}_2\text{CH}=\text{CH}$), 6.04 (1H, t, $J = 11.2$ Hz, $\text{CH}=\text{CHCHOH}$), 6.38 (1H, ddt, $J = 14.8, 11.2, 1.2$ Hz, $\text{CH}_2\text{CH}=\text{CH}$); $^{13}\text{C NMR}$ (125 Hz, CDCl_3): δ 134.3, 133.8, 131.1, 130.7, 127.0, 118.5, 67.3, 62.9, 42.2, 36.6, 26.1, 18.6, -5.1 ppm; IR (neat): 3361.4 (br), 3077.2 (w), 2954.0 (s), 2929.6 (s), 2898.1 (m), 2857.5 (s), 1471.6 (m), 1255.6 (m), 1100.5 (s), 948.7 (m), 836.2 (s) cm^{-1} ; HRMS (ESI+) for $\text{C}_{16}\text{H}_{29}\text{OSi}$ [$\text{M}+\text{H}-\text{H}_2\text{O}$]: calculated 265.1988, found: 265.1986; $[\alpha]_{\text{D}}^{20} = 2.34$ ($c = 1.37$, CHCl_3).

Proof of Stereochemistry: Enantioselectivities were determined by comparison with authentic racemic material prepared using tricyclohexylphosphine as the achiral ligand in the allylation reaction. The absolute stereochemistry was assigned by analogy.

Chiral SFC (AD-H, Chiralpak, 220 nm, 1.0 mL/min, 2.0% MeOH, 150 psi, 50 °C) – analysis of (S,Z,E)-10-(tert-butyldimethylsilyloxy)deca-1,5,7-trien-4-ol



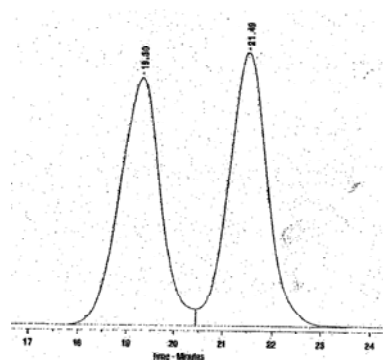
Index	Name	Start [Min]	Time [Min]	End [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [$\mu\text{V}\cdot\text{Min}$]	Area [%]
1	UNKNOWN	7.54	7.84	8.41	0.00	94.17	5158.7	1045.1	94.171
2	UNKNOWN	8.46	8.83	9.63	0.00	5.83	206.1	64.7	5.829
Total						100.00	5364.8	1109.8	100.000



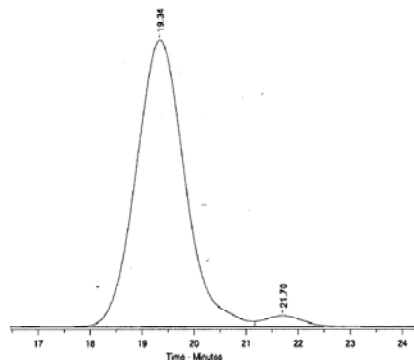
(S,5Z,7E)-8-cyclohexylocta-1,5,7-trien-4-ol. A light yellow oil. Mixture of isomers (38:1 [*E,Z*]:[*E,E*]). $R_f = 0.43$ (3:1 hexane:EtOAc); $^1\text{H NMR}$ (400 Hz, CDCl_3): δ 1.05–1.74 (11H, m, (CH_2)₅ and OH), 2.03 (1H, dtt, $J = 7.6, 7.2, 3.6$ Hz, $\text{CHCH}=\text{CH}$), 2.32 (2H, t, $J = 6.2$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 4.63 (1H, dddd, $J = 8.8, 6.4, 6.0, 2.4$ Hz, CHOH), 5.11–5.18 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.31 (1H, dd, $J = 10.0, 8.8$ Hz, $\text{CH}=\text{CHCHOH}$), 5.70 (1H, dd, $J = 15.2, 7.2$ Hz, $\text{CHCH}=\text{CH}$), 5.83 (1H, dtt, $J = 17.2, 10.0, 7.2$ Hz, $\text{CHCH}=\text{CH}_2$), 6.03 (1H, dd, $J = 11.2, 10.8$ Hz, $\text{CH}=\text{CHCHOH}$), 6.27 (1H, dtt, $J = 15.2, 11.2, 0.8$ Hz, $\text{CHCH}=\text{CH}$) ppm; $^{13}\text{C NMR}$ (100 Hz, CDCl_3): δ 143.4, 134.4, 131.2, 130.7, 122.7, 118.3, 67.4, 42.3, 41.2, 33.02, 32.99, 26.4, 26.2 ppm; IR (neat): 3338.5 (br), 3075.3 (w), 3008.0 (w), 2978.6 (w), 2921.9 (s), 2850.1 (s), 1641.2 (m), 1447.5 (m), 1349.3 (br), 1020.2 (s), 984.0 (s), 947.5 (s), 921.1 (s), 839.9 (m) cm^{-1} ; HRMS (ESI+) for $\text{C}_{14}\text{H}_{21}$ [$\text{M}+1-\text{H}_2\text{O}$]: calculated 189.1643, found: 189.1639; $[\alpha]_D^{20} = -0.91$ ($c = 1.24$, CHCl_3).

Proof of Stereochemistry: Enantioselectivities were determined by comparison with authentic racemic material prepared using tricyclohexylphosphine as the achiral ligand in the allylation reaction. The absolute stereochemistry was assigned by analogy.

Chiral HPLC (AS, Chiralcel, 220 nm, 0.5 mL/min, 0.5% Isopropanol) – analysis of (5Z,7E)-8-cyclohexylocta-1,5,7-trien-4-ol

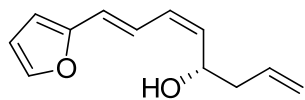


racemic



allylation product

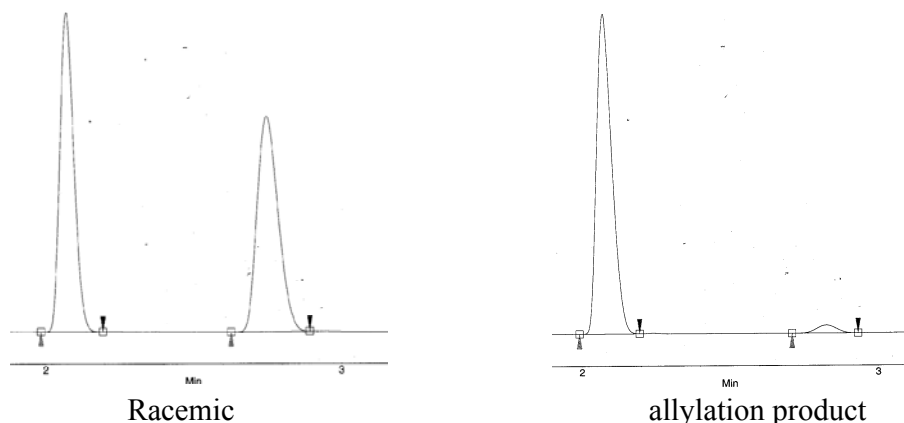
Peak #	Ret. Time	Name	Amount	Amt %	Area	Area %	Type	Width
1	19.34		0.00	N/A	41412050	97.035	BV	0.99
2	21.70		0.00	N/A	1265217	2.965	VB	0.92



(S,5Z,7E)-8-(furan-2-yl)octa-1,5,7-trien-4-ol. A yellow oil. Mixture of isomers (18:1 [E,Z]:[E,E]). $R_f = 0.28$ (3:1 hexane:EtOAc); $^1\text{H NMR}$ (400Hz, CDCl_3): δ 1.76 (1H, d, $J = 3.2$ Hz, OH), 2.36 (2H, ddt, $J = 7.2, 6.4, 1.2$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 4.75 (1H, dddd, $J = 9.6, 6.4, 6.0, 3.2$ Hz, CHOH), 5.13-5.21 (2H, m, $\text{CH}_2\text{CH}=\text{CH}_2$), 5.49 (1H, dd, $J = 10.0, 9.6$ Hz, $\text{CH}=\text{CHCHOH}$), 5.85 (1H, ddt, $J = 17.2, 10.4, 7.2$ Hz, $\text{CH}_2\text{CH}=\text{CH}_2$), 6.16 (1H, ddt, $J = 12.0, 10.4, 0.8$ Hz, $\text{CH}=\text{CHCHOH}$), 6.29-6.40 (3H, m, ArCH=CH, ortho-Ar and meta-Ar), 6.96 (1H, dd, $J = 15.6, 11.6$ Hz, ArCH=CH), 7.38 (1H, d, $J = 1.6$ Hz, para-Ar); $^{13}\text{C NMR}$ (100Hz, CDCl_3): δ 153.1, 142.6, 134.2, 133.6, 130.2, 122.4, 122.0, 118.6, 111.9, 109.4, 67.4, 42.3 ppm; IR (neat): 3381.7 (br), 3118.2 (w), 3076.0 (w), 3010.9 (m), 2978.2 (w), 2929.4 (m), 1676.1 (w), 1638.5 (m), 1609.6 (w), 1483.7 (m), 1152.1 (m), 1014.4 (s), 984.1 (s), 942.4 (s), 925.2 (s), 735.8 (s) cm^{-1} ; HRMS (ESI+) for $\text{C}_{12}\text{H}_{13}\text{O}$ [M+1- H_2O]: calculated 173.0966, found: 173.0971; $[\alpha]_D^{20} = 0.65$ ($c = 1.12$, CHCl_3).

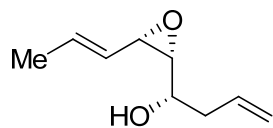
Proof of Stereochemistry: Enantioselectivities were determined by comparison with authentic racemic material prepared using tricyclohexylphosphine as the achiral ligand in the allylation reaction. The absolute stereochemistry was assigned by analogy.

Chiral SFC (AD-H, Chiralpak, 220 nm, 5.0 mL/min, 5.0% MeOH, 150 psi, 50 °C) – analysis of (5Z,7E)-8-(furan-2-yl)octa-1,5,7-trien-4-ol

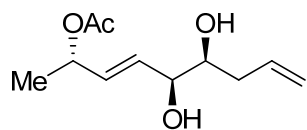


Index	Name	Start Time [Min]	End Time [Min]	RT Offset [Min]	Quantity [% Area]	Height [μV]	Area [$\mu\text{V}\cdot\text{Min}$]	Area [%]	
1	UNKNOWN	1.99	2.07	2.19	0.00	96.87	32872.4	1966.2	96.873
2	UNKNOWN	2.71	2.82	2.93	0.00	3.13	820.9	63.5	3.127
Total						100.00	33693.3	2029.7	100.000

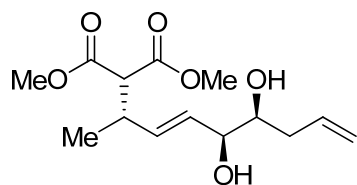
Procedure for Functionalization of (*S*,*5Z*,*7E*)-nona-1,5,7-trien-4-ol (Scheme 2)



(*S*)-1-((*2R*,*3S*)-3-((*E*)-prop-1-enyl)oxiran-2-yl)but-3-en-1-ol. A flame-dried 25-mL round-bottom-flask equipped with a magnetic stir-bar was charged with 221 mg (1.60 mmol) of (*S*,*5Z*,*7E*)-nona-1,5,7-trien-4-ol, 8.01 mL of THF, and 336 mg (4.01 mmol) of NaHCO₃. The flask was sealed with septum and cooled in a cryo-cool to -20 °C under N₂. After 30 minutes, mCPBA (77%) was added as a white powder, and the mixture was allowed to stir at this temperature for another 4 hours. Then, the reaction was filtered through a plug of silica gel (prewashed with 5% triethylamine in ether), washed with ether, and concentrated under reduced pressure. The crude material was purified by silica gel (prewashed with 5% triethylamine in ether) chromatography (3:1 pentane:ether) to afford 195.0 mg (79%) of the title compound as light yellow oil and as a mixture of diastereomers (40:1 *d.r.*). $R_f = 0.59$ (1:1 hexane/EtOAc, stain in KMnO₄). ¹H NMR (400 Hz, CDCl₃): δ 1.76 (3H, dd, $J = 6.8, 1.6$ Hz, CH₃), 2.12 (1H, s, OH), 2.33 (2H, m, CH₂CH=CH₂), 3.06 (1H, dd, $J = 8.0, 4.4$ Hz, CHOCHCHOH), 3.49 (1H, dd, $J = 8.0, 4.4$ Hz, CHOCHCHOH), 3.60 (1H, dtd, $J = 7.6, 6.0, 4.0$ Hz, CHOH), 5.12-5.18 (2H, m, CH₂CH=CH₂), 5.33 (1H, ddd, $J = 15.2, 8.0, 1.2$ Hz, CH₃CH=CH), 5.82 (1H, ddt, $J = 17.2, 10.4, 6.8$ Hz, CH₂CH=CH₂), 5.97 (1H, dq, $J = 15.2, 6.4$ Hz, CH₃CH=CH); ¹³C NMR (100Hz, CDCl₃): δ 133.9, 133.5, 124.9, 118.4, 69.4, 61.8, 58.0, 38.7, 18.3 ppm; IR (neat): 3415.0 (br), 3077.0 (w), 3003.5 (m), 2970.6 (m), 2919.7 (m), 2857.1 (w), 1668.3 (w), 1641.9 (m), 1436.2 (m), 1378.2 (w), 1295.1 (br), 1047.3 (m), 963.5 (s), 914.4 (s), 873.4 (m) cm⁻¹; HRMS (ESI+) for C₉H₁₃O [M+H-H₂O]: calculated: 137.0966, found: 137.0973; $[\alpha]_D^{20} = +1.53$ ($c = 0.70$, CHCl₃).



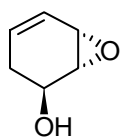
(*2S*,*5S*,*6S*,*E*)-5,6-dihydroxynona-3,8-dien-2-yl acetate. An oven-dried 2-dram vial was charged with 7.5 mg (0.0065 mmol) of tetrakis(triphenylphosphine)palladium, 0.20 mL of THF, and 5.8 mg (0.098 mmol) of acetic acid in a dry-box under an argon atmosphere. The vial was capped with a septum, taped with electrical tape, and removed from the dry-box. The reaction mixture was cooled in a ice-water bath for 30 minutes, and then 10.0 mg (0.0650 mmol) of (*S*)-1-((*2R*,*3S*)-3-((*E*)-prop-1-enyl)oxiran-2-yl)but-3-en-1-ol was added dropwise to the reaction vial as a solution in 0.14 mL of THF. The reaction was allowed to stir at 0 °C for 1 hour, followed by another 3 hours at ambient temperature. After this time period, the reaction was cooled to 0 °C again, followed by addition of about 10 μL of H₂O₂ (30% in water). Saturated NaHCO₃ and ether were added and stirring continuously for 5 minutes, and the layers were separated. The aqueous layer was extracted with ether (×3), and the organic layers were filtered through a plug of MgSO₄ (top) and silica gel (bottom). Solvent was evaporated *in vacuo*. The crude material was purified by silica gel chromatography (1:1 pentane:ether) to afford 8.1 mg (58%) of the title compound (10:1 *I,4:I,2*) as a colorless oil. $R_f = 0.2$ (1:1 hexane/EtOAc, stain in KMnO₄). ¹H NMR (500 Hz, CDCl₃): δ 1.32 (3H, d, $J = 6.5$ Hz, CH₃CHOAc), 2.05 (3H, s, CH₃C(O)), 2.15-2.38 (4H, m, CH₂CH=CH₂ and 2 OHs), 3.55 (1H, ddt, $J = 8.0, 6.0, 4.0$ Hz, CHOHCH₂), 3.98 (1H, m, CH=CHCHOH), 5.13-5.17 (2H, m, CH₂CH=CH₂), 5.36 (1H, p, $J = 6.5$ Hz, CH₃CHOAc), 5.77-5.89 (3H, m, CH=CH and CH₂CH=CH₂); ¹³C NMR (125 Hz, CDCl₃): δ 170.5, 134.3, 133.2, 130.9, 118.7, 74.9, 73.6, 70.4, 37.8, 21.6, 20.4 ppm; IR (neat): 3387.1 (br), 3114.3 (w), 2979.2 (w), 2930.9 (w), 2033.3 (w), 2005.8 (w), 1735.7 (s), 1641.5 (w), 1432.0 (w), 1372.4 (m), 1242.5 (s), 1043.2 (m) cm⁻¹; HRMS (ESI+) for C₁₁H₁₇O₃ [M+H-H₂O]: calculated: 197.1178, found: 197.1181; $[\alpha]_D^{20} = -31.85$ ($c = 0.43$, CHCl₃).



Dimethyl 2-((2S,5S,6S,E)-5,6-dihydroxynona-3,8-dien-2-yl)malonate. An oven-dried 2-dram vial was charged with 7.5 mg (0.0065 mmol) of tetrakis(triphenylphosphine)palladium, 0.20 mL of THF, and 12.9 mg (0.0975 mmol) of dimethylmalonate in a dry-box under an argon atmosphere. The vial was capped with septum, taped with electrical tape, and removed from the dry-box. Next, 10.0 mg

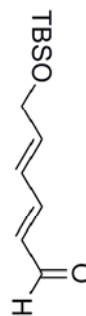
(0.0650 mmol) of (*S*)-1-((2*R*,3*S*)-3-((*E*)-prop-1-enyl)oxiran-2-yl)but-3-en-1-ol was added to the reaction vial as a solution in THF (0.14 mL). The reaction was allowed to stir at ambient temperature for 14 hours. After this time period, the reaction was cooled to 0 °C, followed by addition of about 10 μ L of H₂O₂ (30% in water). Saturated NaHCO₃ and ether were added and stirring continuously for 5 minutes, and the layers were separated. The aqueous layer was extracted with ether (\times 3), and the organic layers were filtered through a plug of MgSO₄ (top) and silica gel (bottom). Solvent was evaporated *in vacuo*. The crude material was purified by silica gel chromatography (3:1 hexane:EtOAc) to afford 16.0 mg (86%) of the title compound (17:1 *d.r.*) as a colorless oil. R_f = 0.2 (1:1 hexane:EtOAc, stain in KMnO₄). ¹H NMR (500 Hz, CDCl₃): δ 1.11 (3H, d, J = 7.0 Hz, CHCH₃), δ 2.15 (1H, dddt, J = 14.5, 7.5, 8.0, 1.0 Hz, CH_aH_bCH=CH₂), δ 2.27-2.35 (3H, m, CH_aH_bCH=CH₂ and 2 OHs), δ 2.99 (1H, ddq, J = 8.5, 7.5, 7.0 Hz, CHCH₃), δ 3.33 (1H, d, J = 8.5 Hz, C(O)CHC(O)), δ 3.51 (1H, ddd, J = 8.0, 6.0, 4.0 Hz, CHOHCHOHCH₂), δ 3.70 (3H, s, (OCH₃)_a), δ 3.73 (3H, s, (OCH₃)_b), δ 3.92 (1H, dd, J = 6.5, 4.5 Hz, CHOHCHOHCH₂), δ 5.12-5.17 (2H, m, CH₂CH=CH₂), δ 5.55 (1H, ddd, J = 15.5, 7.0, 1.0 Hz, CH=CHCHOH), δ 5.72 (1H, ddd, J = 16.0, 8.5, 1.0 Hz, CH=CHCHOH), δ 5.84 (1H, dddd, J = 17.5, 10.5, 7.5, 6.5 Hz, CH₂CH=CH₂); ¹³C NMR (125 Hz, CDCl₃):

δ 168.78, 168.76, 135.2, 134.5, 130.8, 118.4, 75.2, 73.6, 57.6, 52.7, 52.6, 37.6, 37.0, 18.3 ppm; IR (neat): 3433.4 (br), 2955.5 (m), 1734.9 (s), 1641.3 (w), 1534.7 (m), 1243.5 (br), 1159.9 (m), 1062.7 (m), 1018.7 (m), 976.3 (m) cm⁻¹; HRMS (ESI+) for C₁₄H₂₁O₅ [M+H-H₂O]: calculated: 269.1389, found: 269.1382; $[\alpha]_D^{20}$ = -19.86 (c = 0.88, CHCl₃).



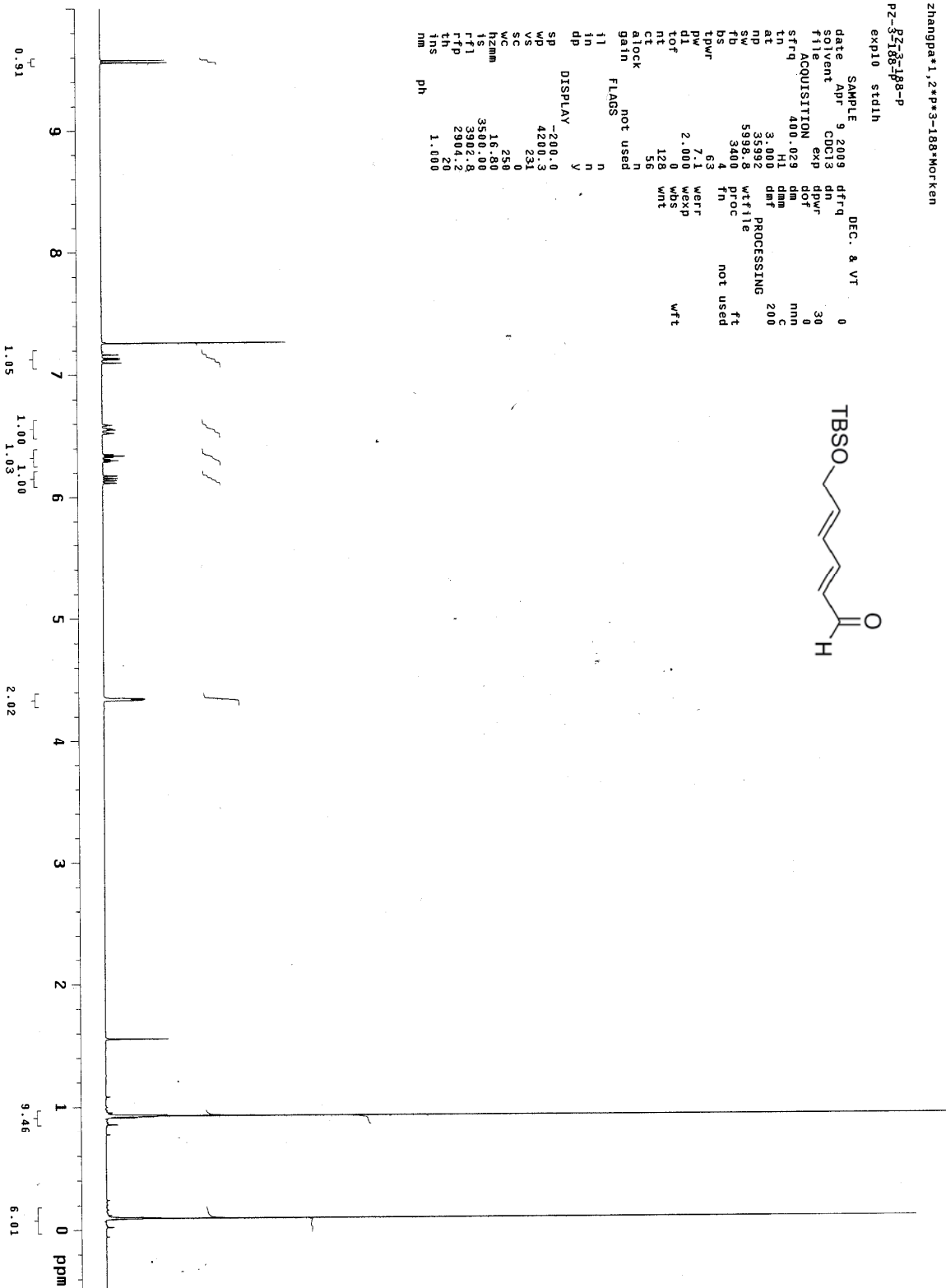
(1*R*,2*S*,6*S*)-7-oxabicyclo[4.1.0]hept-4-en-2-ol. A flame-dried round-bottom-flask was charged with 2.0 mg (0.0032 mmol) of Hoveyda-Grubbs II catalyst, 6.0 mL of CH₂Cl₂, and 10.0 mg (0.0650 mmol) of (*S*)-1-((2*R*,3*S*)-3-((*E*)-prop-1-enyl)oxiran-2-yl)but-3-en-1-ol in CH₂Cl₂ (0.5 mL). The reaction was allowed to stir at ambient temperature under N₂ for 2 hours. After this time period, solvent was evaporated *in vacuo*, and the crude material was purified by silica gel chromatography (2:1 pentane:ether) to afford 5.9 mg (81%) of the title compound (17:1 *d.r.*) as a colorless oil. R_f = 0.23 (1:1 hexane:EtOAc, stain in KMnO₄). ¹H NMR (500 Hz, CDCl₃): δ 1.53 (1H, d, J = 8.5 Hz, OH), δ 2.27 (1H, ddt, J = 17.5, 6.5, 2.0 Hz, CH_aH_b), δ 2.37 (1H, dddd, J = 17.0, 6.0, 3.5, 3.0 Hz, CH_aH_b), δ 3.34 (1H, td, J = 4.0, 1.5 Hz, CHOHCHOH), δ 3.51 (1H, ddd, J = 4.0, 2.5, 2.0 Hz, CHOH), δ 4.43 (1H, m, CHOHCHOH), δ 5.82 (1H, ddm, J = 9.5, 6.0 Hz, CH₂CH=CH), δ 6.06 (1H, dt, CH₂CH=CH); ¹³C NMR (125 Hz, CDCl₃): δ 129.6, 124.1, 64.1, 55.9, 47.0, 30.6 ppm; IR (neat): 3412.5 (br), 3041.1 (w), 2993.0 (w), 2923.0 (s), 2852.8 (m), 1641.8 (w), 1418.5 (m), 1398.1 (m), 1050.9 (s), 1036.0 (s), 981.1 (s), 952.2 (m), 885.9 (s), 805.2 (s), 771.3 (s) cm⁻¹; HRMS (ESI+) for C₆H₉O₂ [M+H]: calculated: 113.0603, found: 113.0597; $[\alpha]_D^{20}$ = -79.15 (c = 0.42, CHCl₃).

Spectral Data



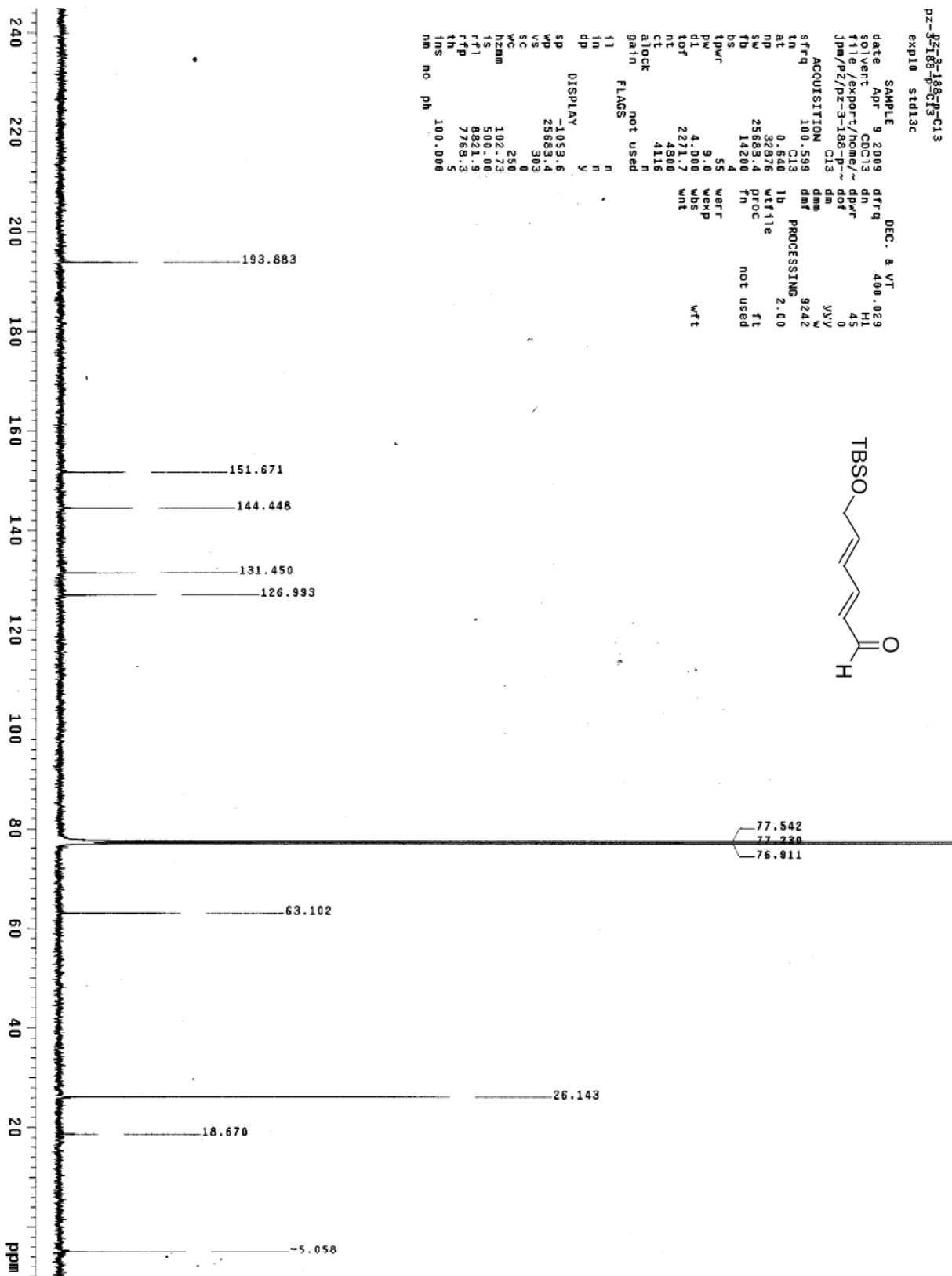
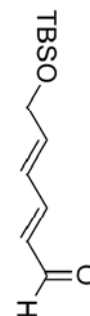
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ct 56
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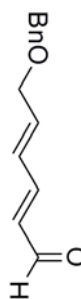


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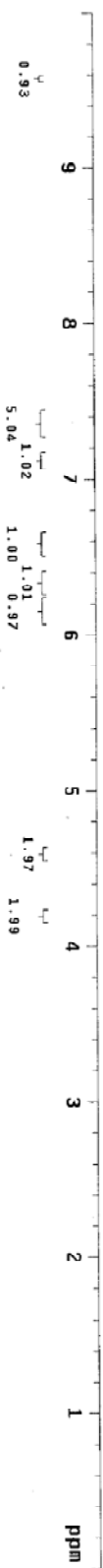
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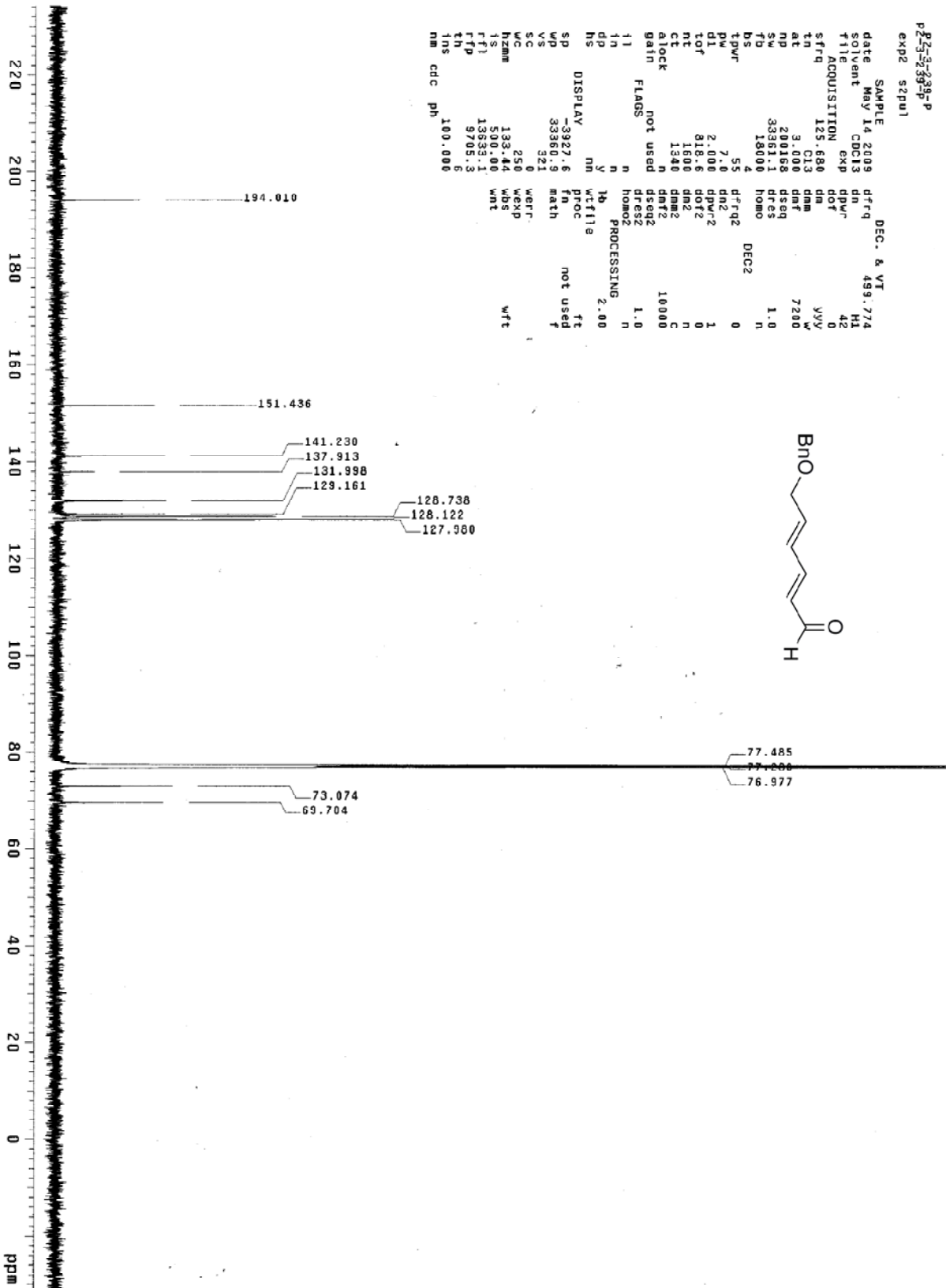


p2-33-239-p
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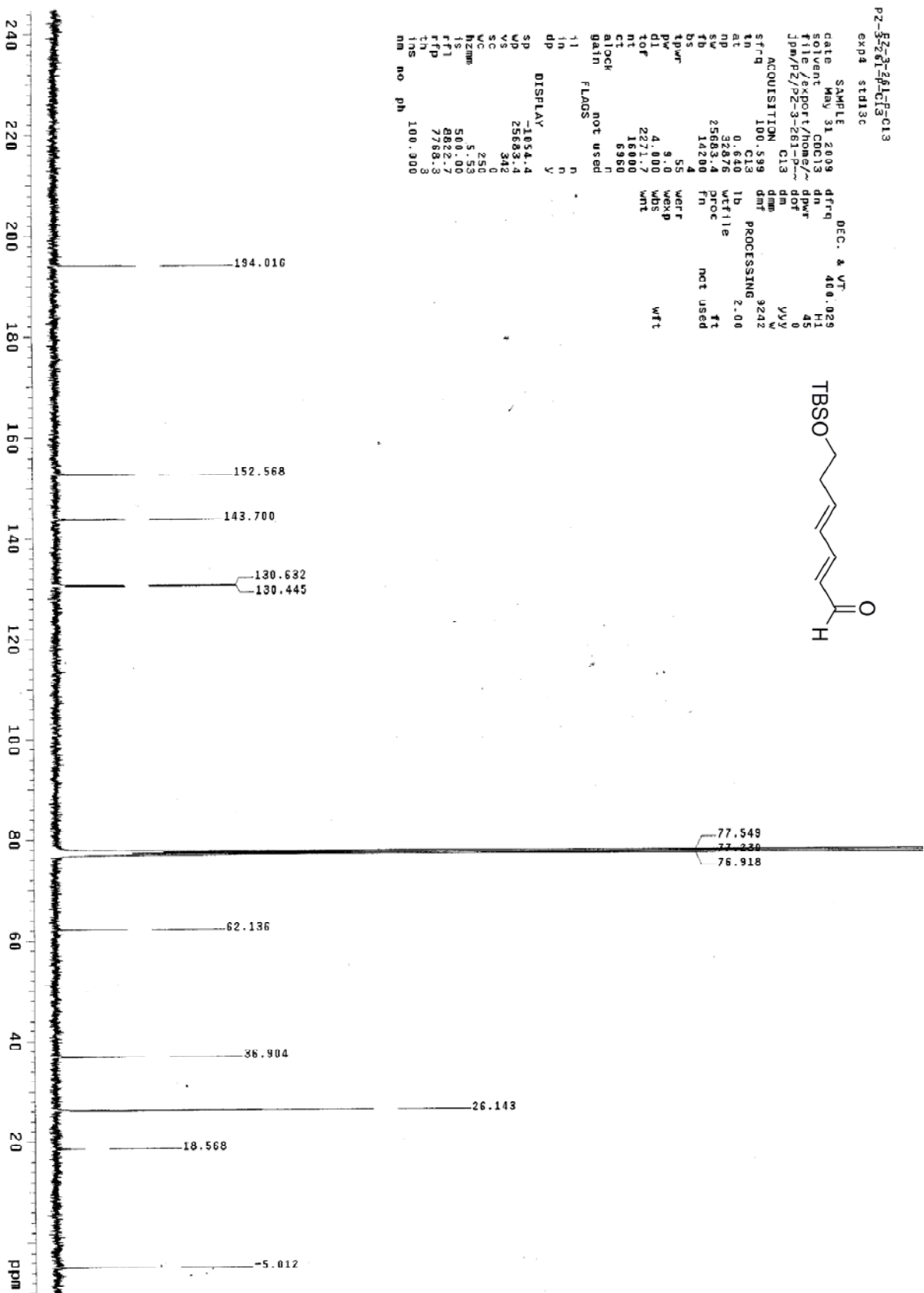
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tof		wrt			
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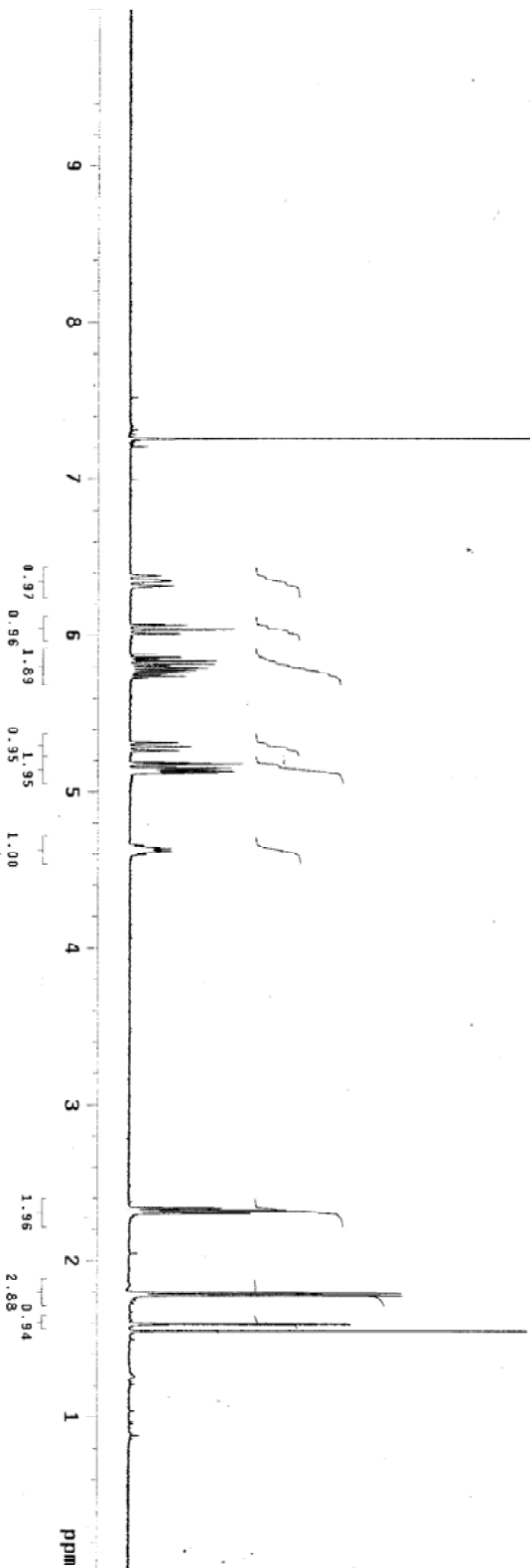
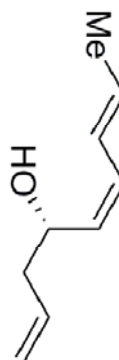
zhangpa*1, 2*p*3-178*Morken

P2-3176-3P

exps std1h

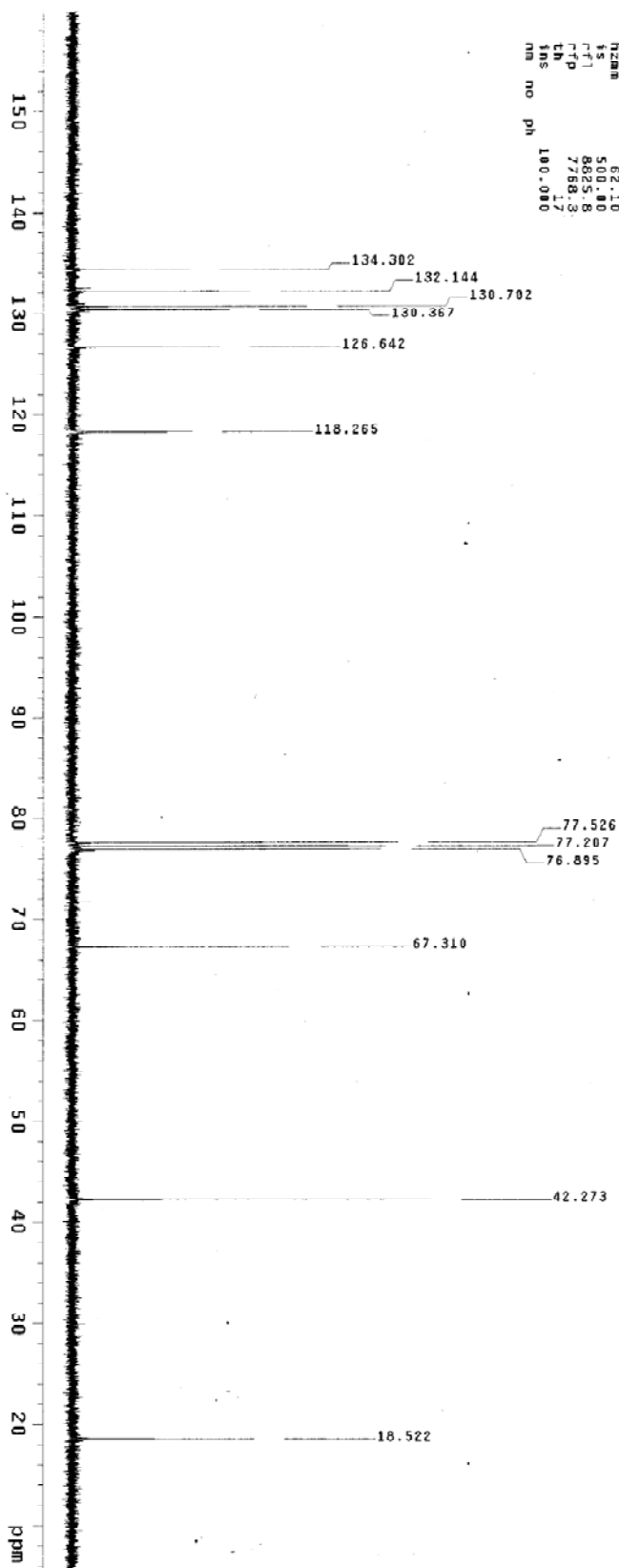
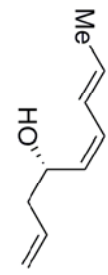
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zhangjda*1.2*pp*3-147*NO-Morken
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 l49 n
 l50 n
 l51 n
 l52 n
 l53 n
 l54 n
 l55 n
 l56 n
 l57 n
 l58 n
 l59 n
 l60 n
 l61 n
 l62 n
 l63 n
 l64 n
 l65 n
 l66 n
 l67 n
 l68 n
 l69 n
 l70 n
 l71 n
 l72 n
 l73 n
 l74 n
 l75 n
 l76 n
 l77 n
 l78 n
 l79 n
 l80 n
 l81 n
 l82 n
 l83 n
 l84 n
 l85 n
 l86 n
 l87 n
 l88 n
 l89 n
 l90 n
 l91 n
 l92 n
 l93 n
 l94 n
 l95 n
 l96 n
 l97 n
 l98 n
 l99 n
 l100 n
 no ph 100.080



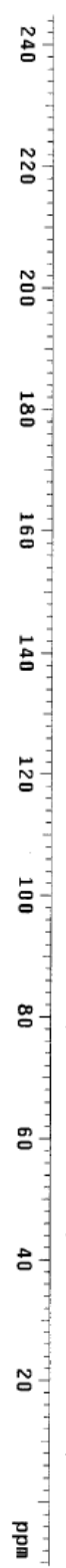
zhangjupr-1,6-f-4a-c-3-140-nmr.km

02-23-18-21

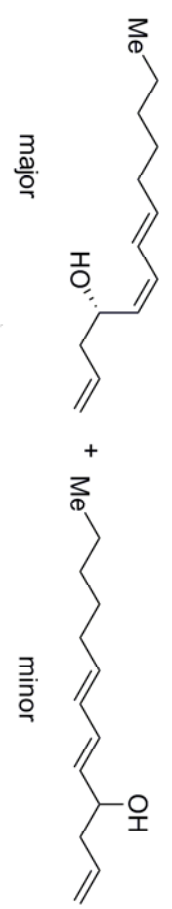
expb std13c

```

SAMPLE DEC. 8 VT
date Feb 21 2009 dfrq 400.029
cont CDCl3 d1 45
f1s 45 d1 45
f1s ACQUISITION exp dmf yyy
sfrq 100.599 dm
in C13 dmf 9242
at 0.540 dm
np 32876 PROCESSED 2.00
sw 25683.4 lbfile
fb 14200 wfile
ds 4 proc
tpwr 55 fn
pw 9.0 weff
dl 4.000 weff
tof 2271.7 wexp
nt 1600 wds
ct 72 wft
alock not used
gain n
flags not used
11 n
in n
dp y
DISPLAY -1056.7
sp 25683.4
wp 44
vs 0
sc 250
wc hzmm 24.84
ls 500.00
ftl 8925.9
ftp 7789.3
tms 16
nm no 100.000
    
```

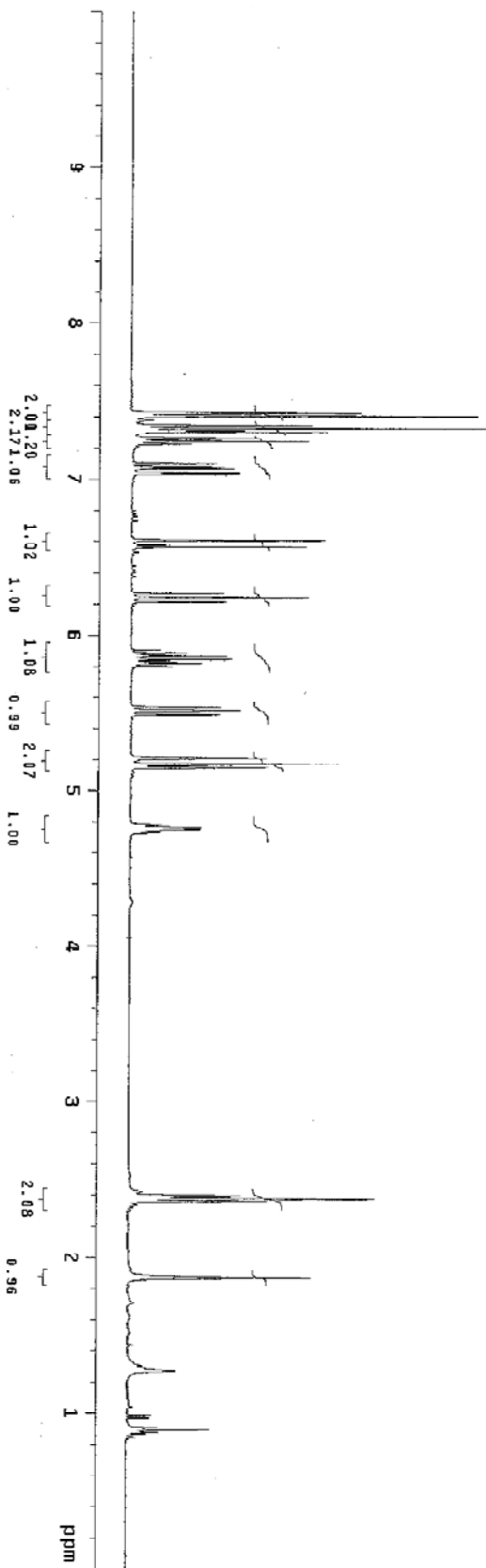
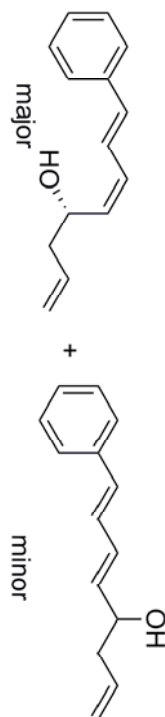


- 137.824
- 134.341
- 130.858
- 130.523
- 125.177
- 118.265
- 77.542
- 77.230
- 76.911
- 67.318
- 42.273
- 38.047
- 31.660
- 29.096
- 22.753
- 14.275



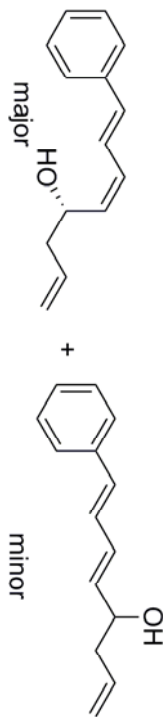
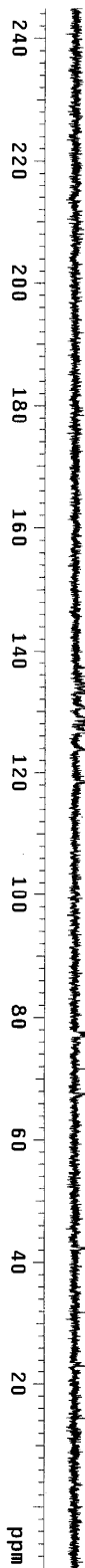
p2-82-3-157-9
expi stahh

SAMPLE		DEC. & VT	
date	Mar 18 2009	dfrq	0
solvent	CDCl3	dn	30
file	/export/home/~	dpwr	0
jpw	p2-8-157-9	dot	0
ACQUISITION		dm	nnn
strq	460.025	dmp	c
tn	HI	dnt	200
at	3.001	PROCESSING	
np	38982	wfitle	ft
sw	5898.8	proc	not used
fb	3401	fn	
bs	4	weir	
tpwr	7.1	wexp	
pl	2.000	wbs	wft
lrf	1	wnt	
nr	32		
ct	24		
clock	n		
gath	n		
flags			
rl	n		
in	n		
dp	y		
sp	DISPLAY		
wp	-0.1		
wv	4000.7		
wc	57		
sc	0		
uc	250		
hzmm	16.00		
ts	1456.21		
rfl	1801.1		
rtp	0		
th	9		
ins	1.000		
nm			



p2-RZ-3-157-P-C13
 exp2 std13c

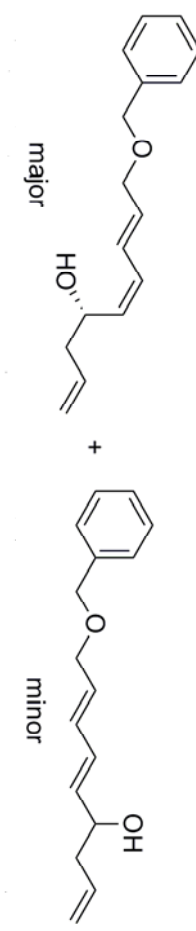
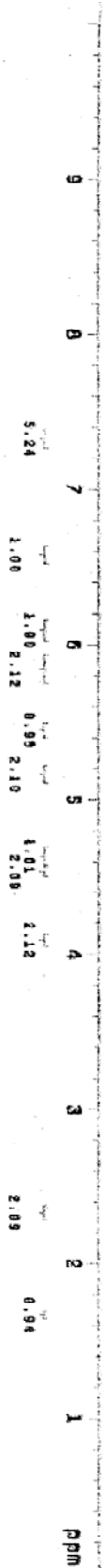
date	Mar 16 2009	dfreq	DEC. & VT	400.029
solvent	CDCl3	dn		H1
file	exp	dpwr		45
title	exp	dot		0
sfreq	100.593	dm		VVY
z1	0.543	dmf		9242
nd	32876	lb	PROCESSING	2.00
sw	25683.4	wtfile		
fb	14200	proc		
bs	4	fn		
tpwr	55	ft		
pw	9.0	not used		
dl	4.000	werr		
tof	2271.7	wexp		
nt	1500	wbs		
ct	60	wnt		
atlock	not used			
gain	not used			
flags				
i1	n			
in	n			
dp	y			
display				
sp	1062.2			
wp	23983.4			
v	94			
sc	0			
wc	250			
hzmm	102.73			
is	500.00			
rfl	8830.5			
rffp	7768.3			
th	13			
ims	100.000			
nm				

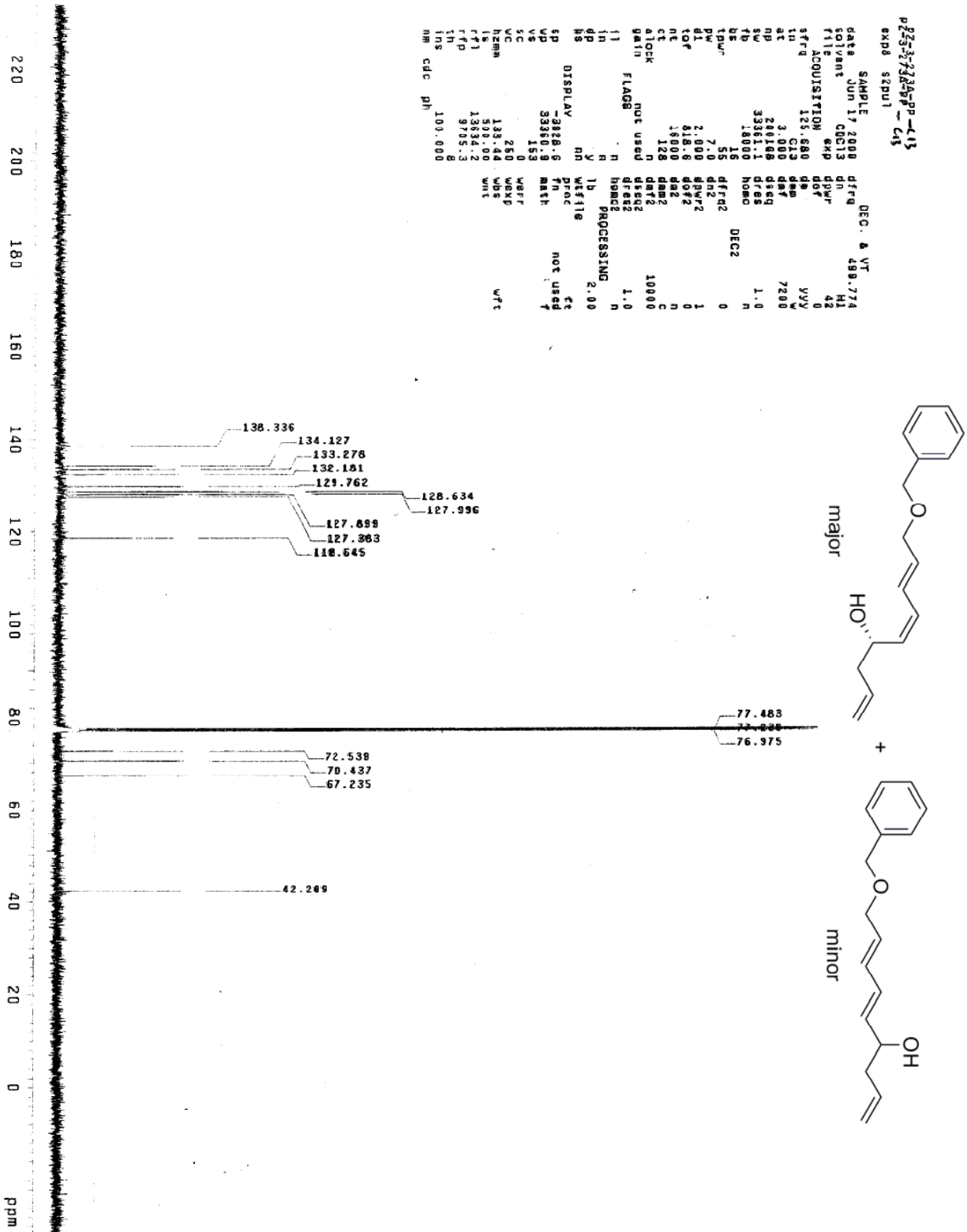


137.146
 134.606
 134.099
 133.305
 130.593
 128.769
 128.014
 126.712
 123.767
 118.522
 77.542
 77.222
 76.911
 67.489
 42.304


```

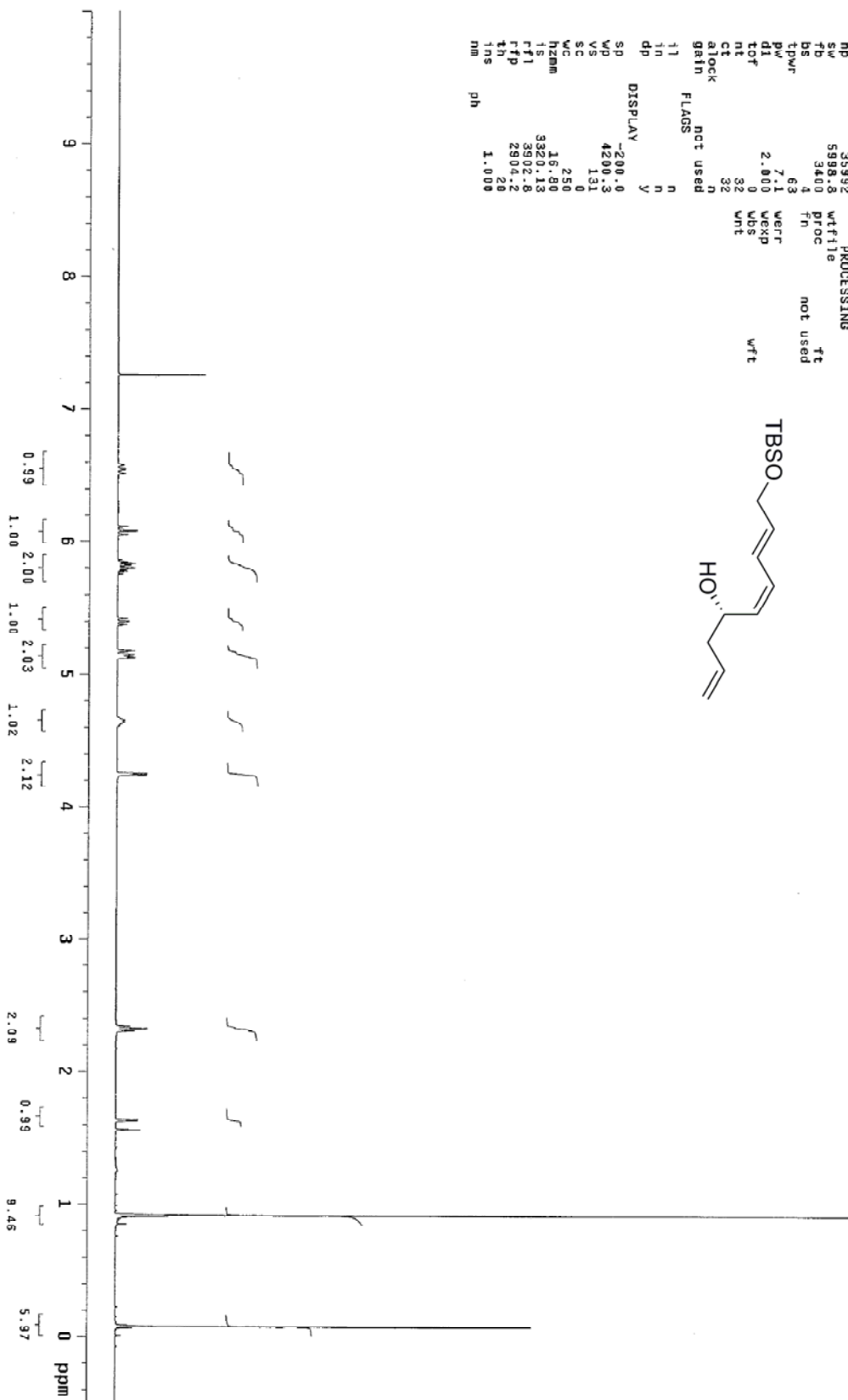
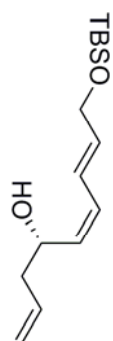
#2527345pp
exp# szpol
SAMPLE 2009 JUN 11 499.774 DEC. & VT
SOLVENT C6Cl6 30 M1
FILE ACQUISITION EXP 30 M1
EXP# 499.773 0
SFRQ 499.773 0
LN HI 200
AT 5.089 200
NP 7803.3 1.0
SW 4088 0
TD 4088 0
DS 4088 0
TPWR 4.5 SFRQ2 DEQ2 0
PW 4.5 SFRQ2 0
DI 9 DWR2 2
TOT 497.0 DOT2 2
NI 32 SFR2 2
CI 32 SFR2 2
ALOCK N SFR2 200
gain not used N SFR2 200
FLAGS not used N SFR2 200
11 N NOMOS PROCESSING 1.0
IN N WIT1 1.0
DP Y Proc not used Y
HS N N N N
SP DISPLAY N N N N
WD 499.78 -0.1 math
VS 104 wcrp
SC 0 wcrp
HZMM 19.99 WDS
IS 483.67 WTE
FT1 4148.17
FTP 3828.17
TH 1.000
INS 1.000
    
```





P2-32189-189-P
 exp10 st11h

date	SAMPLE	8 2003	DEC. 8 VI	0
solvent	CCC13			
file	exp			30
ACQUISITION	exp			0
stfrq	400.029			nnn
tn	H1	dmf		c
at	3.000	dmf		200
mp	3599.2	wtfile	PROC	ft
sw	5998.8	proc	not used	
fb	3400	fn		
bs				
tpwr	63	werr	7.1	wft
dl	2.000	wexp	0	
tof	0	wbs	32	
nt	32	unt		
ct	32			
atlock	n			
gain	not used			
FLAGS	not used			
l1	n			
l2	n			
l3	n			
dp	y			
DISPLAY	-200.0			
SP	4200.3			
VP	131			
VS	0			
WC	250			
SC	0			
hzmm	16.90			
ts	320.13			
ts1	3072.8			
tfp	2904.2			
th	42			
tns	1.000			
nm				
ph				

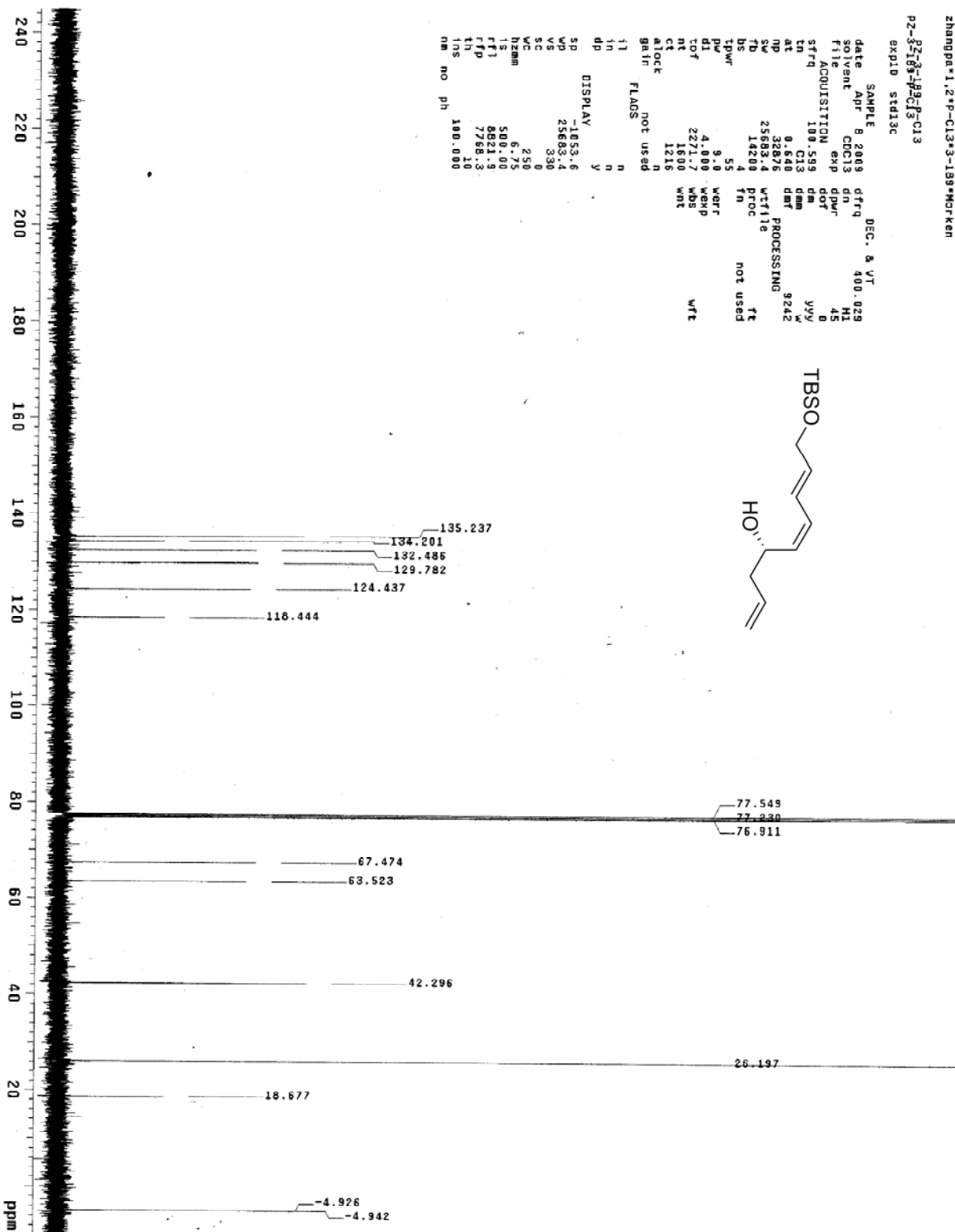


zhangpa*1,2*P-Cl3*3-139*Morken

PZ-3278-199-Cl3
 expid st013c

```

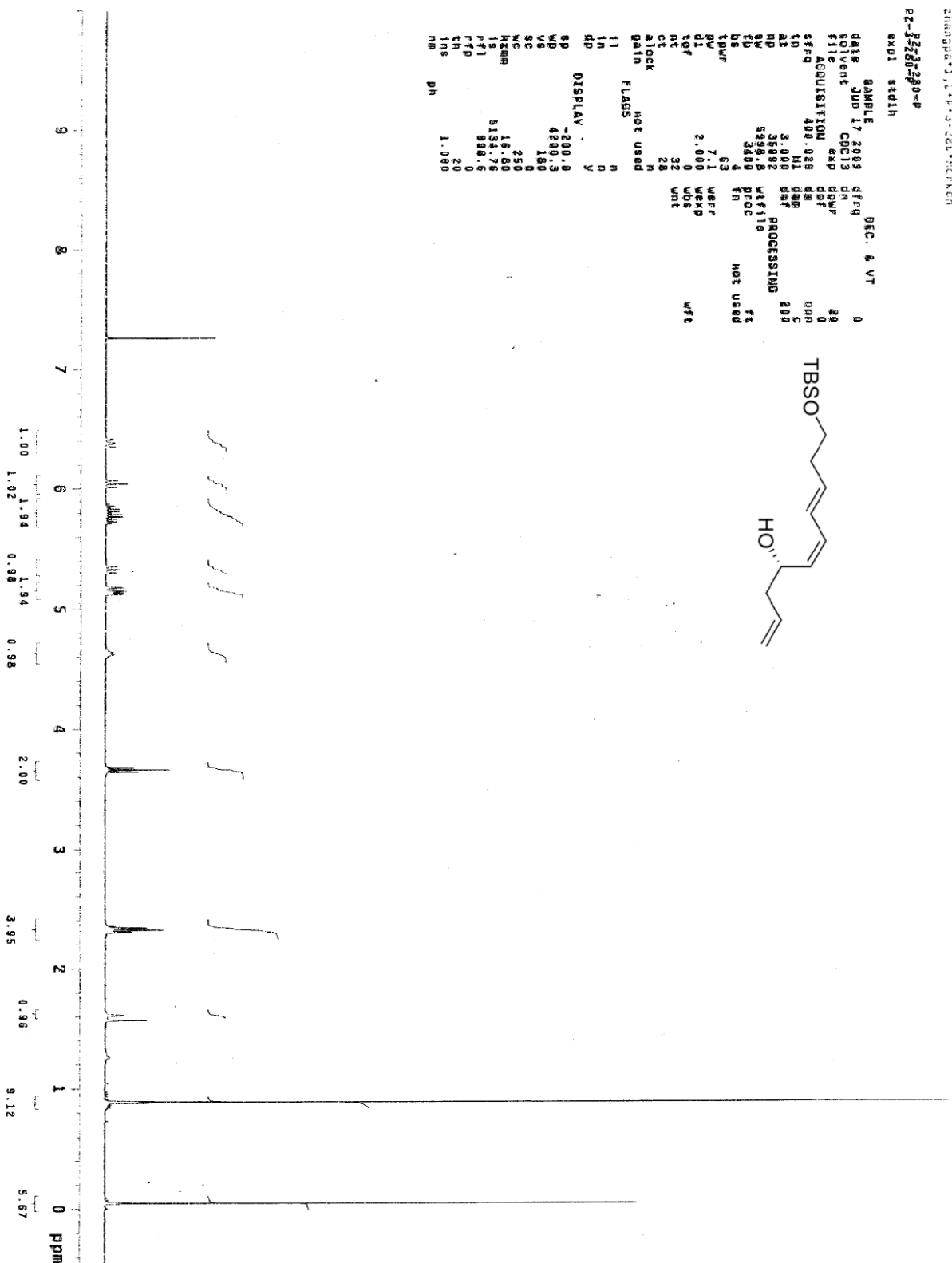
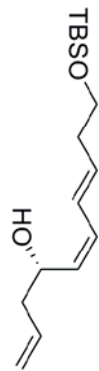
SAMPLE      date Apr  8 2009   dfrq 400.029   DEC. & VT
solvent     dm             d0
file        exp dot    ddr      H1
ACQUISITION 100.599   dm       45
sfreq       C13      dm       0
tn          C13      dm       0
at          0.640   dm       0
np          32876   wt11le  9242
sw          25683.4 wt11le  9242
td          14200   Proc     not used
bs          4       fn
tpwr       55
pw         3.0     warrt
pr         4.007   wexp
tof        227.60  wms
nr         4450   wnt
ct         1216
atlock     not used
gain       not used
flags      not used
il         n
in         n
dp         y
DISPLAY   y
sp         -1053.6
wp         25683.4
vs         330
sc         0
wc         250
ncmm       6.75
f1         802.00
f2         802.00
ffp        7764.33
th         1.0
ins        100.000
nm no ph
    
```

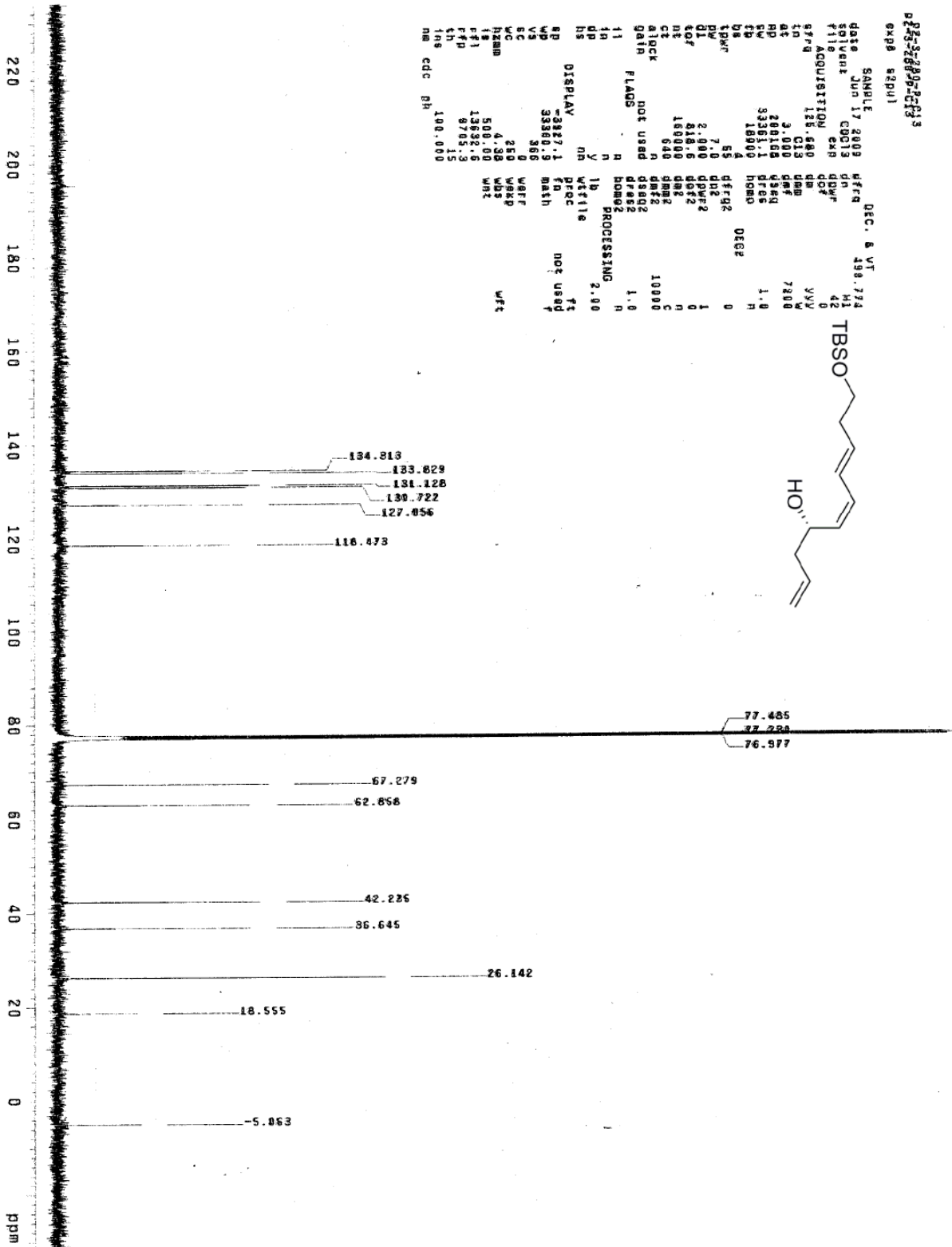


ZHANG001.L.F3-2007.MORKEN

PZ-3286-20-B
ex01 stdh

date	JUN 17 2009	time	17:00
solvent	CDCl3	temp	30
site	ACQUISITION	exp	0
sfreq	400.029	qpc	0
in	H1	qmr	0
az	3.000	psf	200
rd	35002	proc	PROCESSED
sw	5298.0	wtfile	ft
fd	3400	proc	not used
ds	4	ft	
tdwp	65	not used	
dv	7.1	wexp	
va	2.000	wdp	
vcf	32	wt	
ncf	28		
ct	28		
stock	not used		
gain	not used		
fl	n		
in	n		
dd	y		
display	y		
sp	-200.0		
wp	4200.3		
vs	180		
sc	250		
wc	250		
hsmr	16.00		
rs	3154.78		
ft1	999.6		
ft2	20		
ft3	20		
ft4	20		
ft5	20		
mm	1.080		

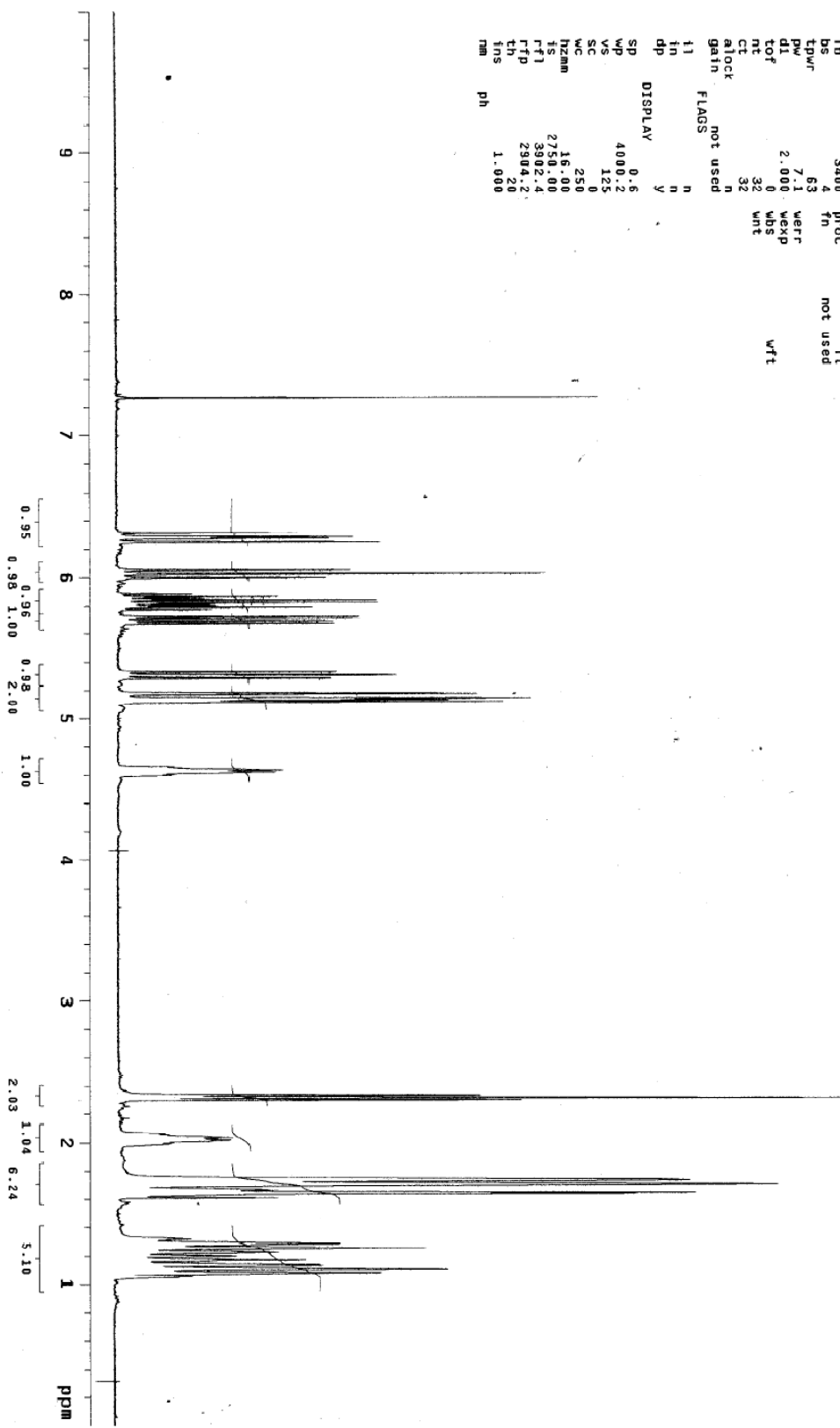
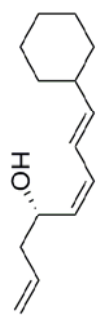




zhangpa1,2*p*3-191*Morken

PZ-3-191-P
 exp10 stdin

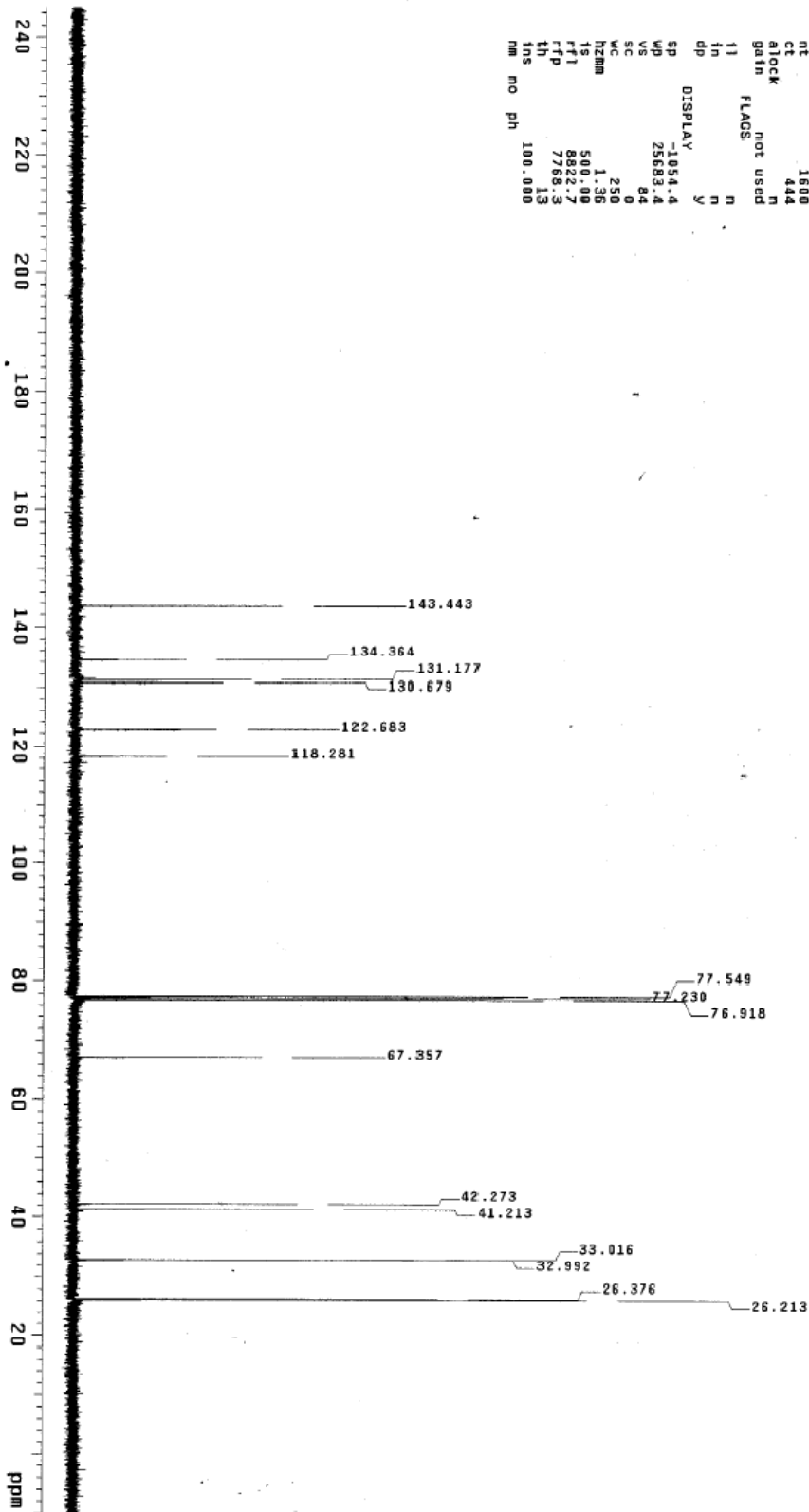
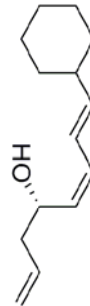
date	Apr 9 2009	DEC. & VT	0
solvent	CDCl3	dn	30
file	exp	dpwr	0
ACQUISITION	exp	dot	nmn
sfreq	400.029	dm	c
tn	H1	dmn	200
at	3.000	dmf	PROCESSING
np	33992	wtfile	200
sw	5938.8	proc	ft
fb	3400	fn	not used
bs	4		
epwr	63	verf	
pw	7.1	wexp	
dl	2.000	wds	wft
cof	0	wit	
ct	32		
cl	32		
alock	not used		
gain	not used		
flags	not used		
ij	n		
in	n		
dp	y		



Pz-3-191-191-C13
 expt10 std13c

SAMPLE DEC. & VT
 date Apr 9 2009 dfrq 400.029
 solvent CDCl3 dn H1
 title /EXPORT/home/w dpvr 45
 jpw/pz-3-191-P-~ dot 0
 yyy w
 ACQUISITION C13 dm
 sfrq 100.539 dnt PROCESSING 3242
 tn C13
 at 0.640 wftfile ft
 np 32876 proc not used
 sw 258854.4 fn
 fd 14200 wprt
 TDW 55 wbsd
 PW 9.0 wds
 dt 4.000 wnt
 tof 2271.7
 nt 1600
 ct 444
 atlock n
 gain not used
 flags n
 ii n
 in n
 dp DISPLAY Y

SP -1094.4
 WP 25683.4
 VS 84
 SC 0
 WC 250
 nzm 1.36
 IS 500.90
 FTI 8822.7
 TPI 7785.3
 S0 100.83
 S1 100.000
 nm no ph



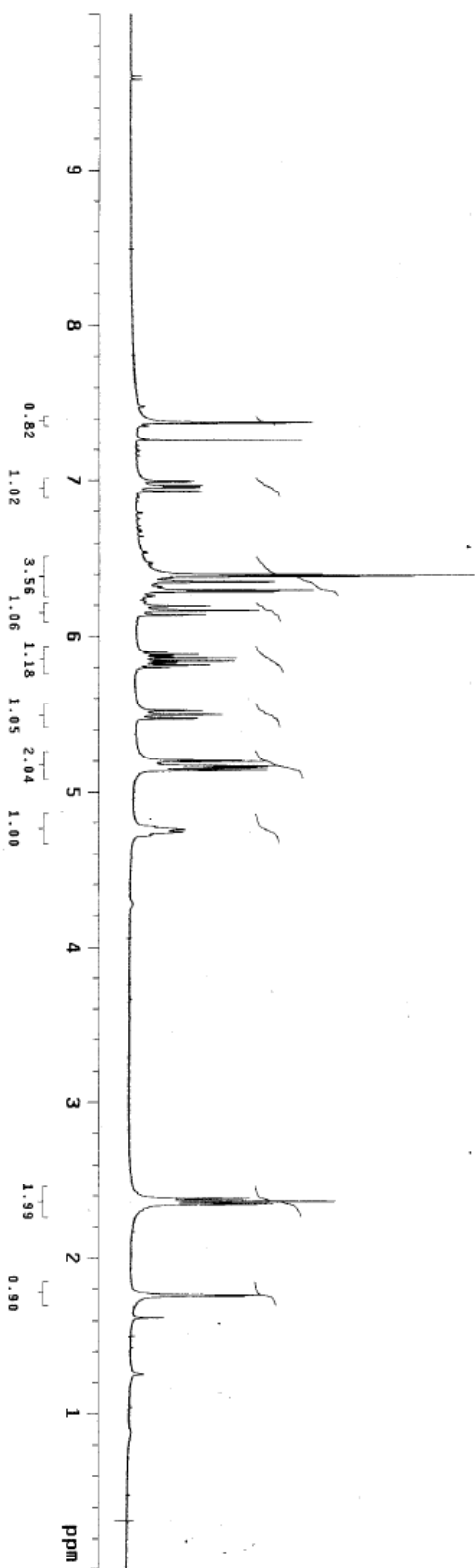
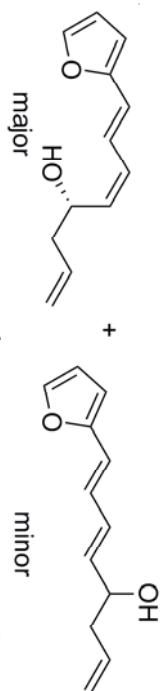
zhangpa*1,2*pp*3-159*Morken

p2-3-159-159-pp
exp2 std1h

```

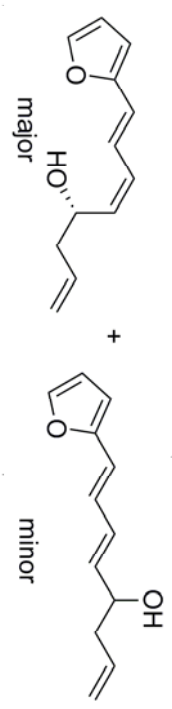
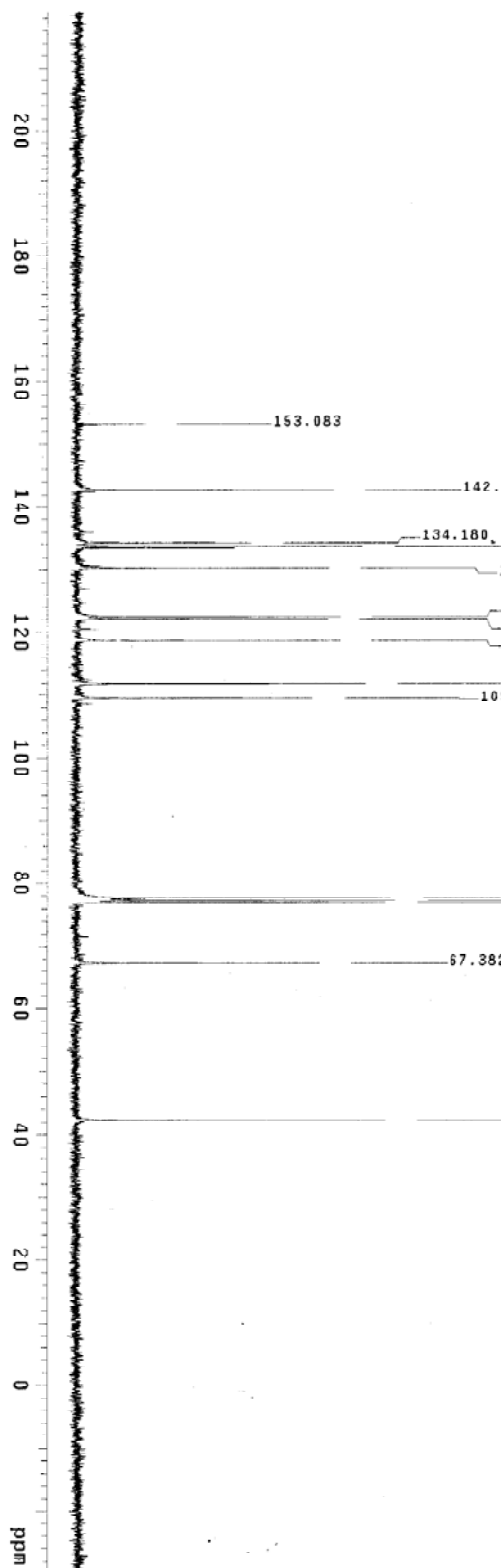
SAMPLE Mar 18 2009 DEC. 8 VT 0
solvent CDC13 dn 30
title exp dpvr 0
ACQUISITION dm 0 nm
sfrq 400.029 dm
tn H1 dm
at 3.000 dm
np 38592 wftfile 200
qw 5980.0 wftfile
hs 349 proc tn
tpwr 63 tn not used
pw 7.1 werr
dl 2.000 wexp
tof 0 wbs
nt 32 wnt
ct 32
atlock n
gain not used
flags n
i1 n
i2 n
i3 n
i4 n
i5 n
i6 n
i7 n
i8 n
i9 n
i10 n
i11 n
i12 n
i13 n
i14 n
i15 n
i16 n
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i32 n
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i36 n
i37 n
i38 n
i39 n
i40 n
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i43 n
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i48 n
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i88 n
i89 n
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i93 n
i94 n
i95 n
i96 n
i97 n
i98 n
i99 n
i100 n

```



P2-3139-133-013130
 exp1 std13c

SAMPLE DEC. & VT
 date Mar 17 2013 dfrq 399.758
 solvent CDCl3 dn n1
 file /223-159/223-159.d01 dn 1
 gcafb/223-130-11d dn 0
 yyy v
 ACQUISITION
 sfrq 100.531 dmf 9259
 tn C13 dseq
 at 2.000 dres 1.0
 np 100000 homo n
 sw 25000.0 temp 25.0
 fb 14000 PROCESSING
 2.00
 bs 4 lb
 tpwr 59 wittle
 pw 9.7 ft
 dl 1.000 fn not used
 tof 0 math f
 nt 10000
 ct 952 warr
 atlock 5 wexp
 gain not used
 wnt
 fl n
 fh n
 dp n
 hs n
 DISPLAY
 sp -2988.5
 wp 24999.6
 vs 50
 sc 0
 wc 250
 hzmm 5.87
 ls 500.00
 rfi 10752.1
 rfp 7763.3
 th 9
 hrs 100.000
 nm

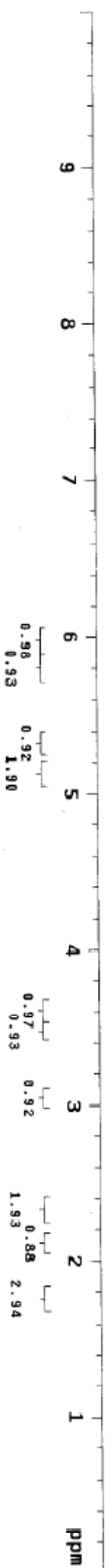
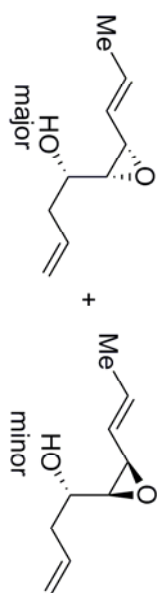


zhangpa*1,2*p*3-192*Mor*ken

STANDARD DISPERSE P2-5-072-P

expt stdIn

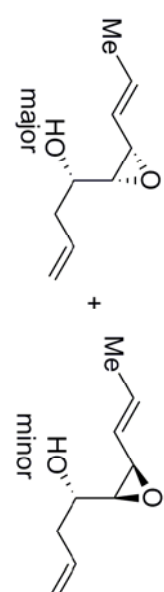
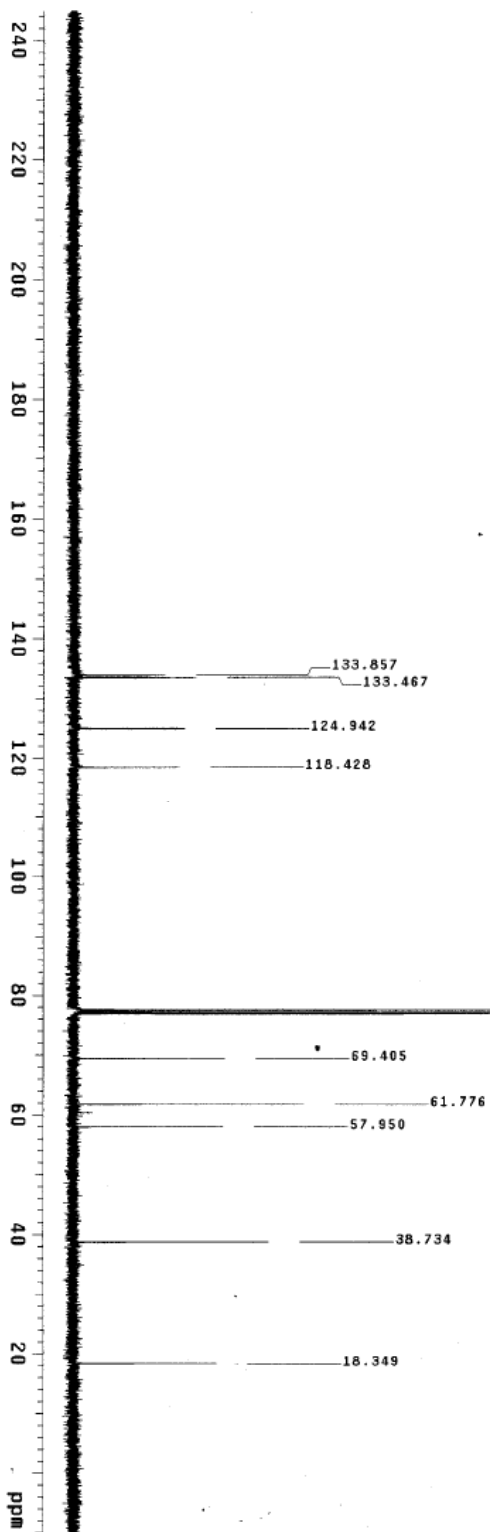
date	Apr 16 2009	DEC. & VT	0
solvent	CDCl3	dn	30
TI	exp	dpwr	0
ACQUISITION	exp	doF	0
sfreq	400.029	da	mm
tn	3.000	HI dnm	5
at	35992	daf	200
np	5998.8	wifile	PROCESSING
sw	3400	proc	ft
fb	3400	fn	not used
bs	4		
tpwr	63	werr	7.1
pw	2.000	wexp	0
dl	0	whs	32
tof	0	wht	32
nt	32		
ct	32		
atlock	n		
gain	not used		
FLAGS	not used		
l1	n		
l2	n		
l3	n		
dp	y		
DISPLAY	-0.1		
sp	4000.2		
wp	76		
vs	0		
sc	250		
vc	0		
hzm	16.00		
hzm	3014.70		
ts	3903.1		
FTF	2904.2		
tn	20		
ins	1.000		
nm			
ph			



p2-3-192-192-P-C13
 expt1 std13c

```

SAMPLE DEC. & VT
date Apr 16 2009 dfrq 400.029
solvent CDCl3 dn H1
F1 ACQUISITION exp dpr 45
sfreq 100.599 dm yyj 45
tn C13 dnm
at 0.640 daf 9242
np 32876 PROCESSED
sw 25683.4 wf1le ft
fb 14200 proc not used
bs 4 tn
tpwr 55
pw 9.10 werr
di 4.1000 wexp
tot 2271.7 wst
ct 44.17 wnt
cl 1036
alock not used
gain not used
fl flags n
in n
dp DISPLAY y
sp -1052.1
wp 25683.4
vs 189
sc 25.0
v 0
hzmm 102.73
is 500.00
rf1 8823.5
rfp 7771.3
th 12
ins 100.000
nm no ph
    
```

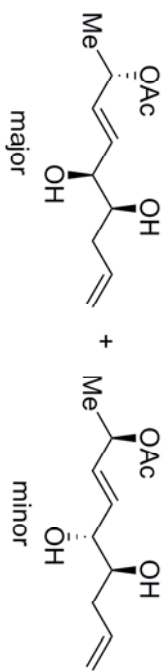
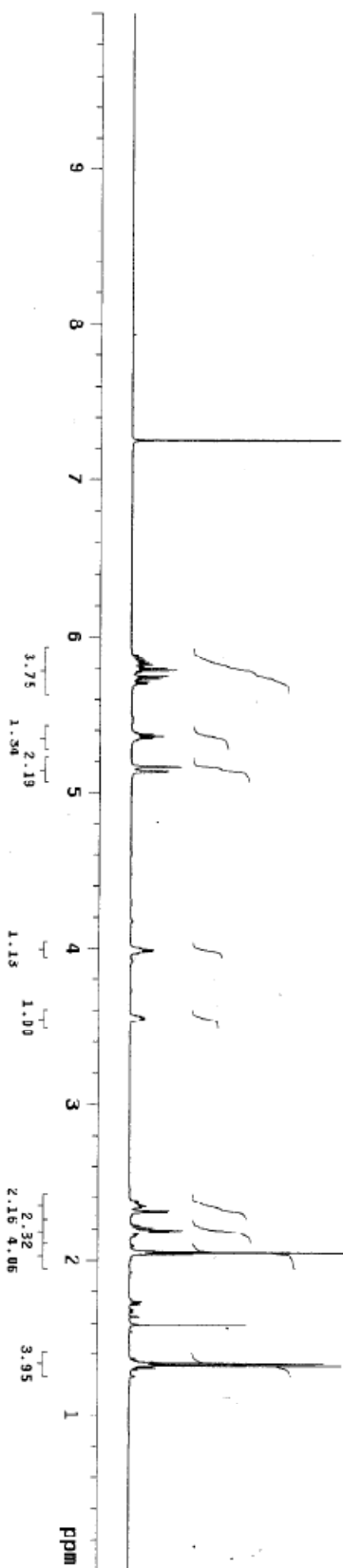


```

P2-3-2008-BPP
expt 12pu1

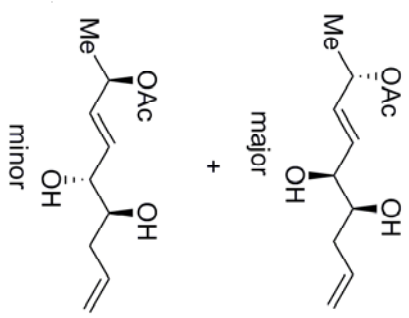
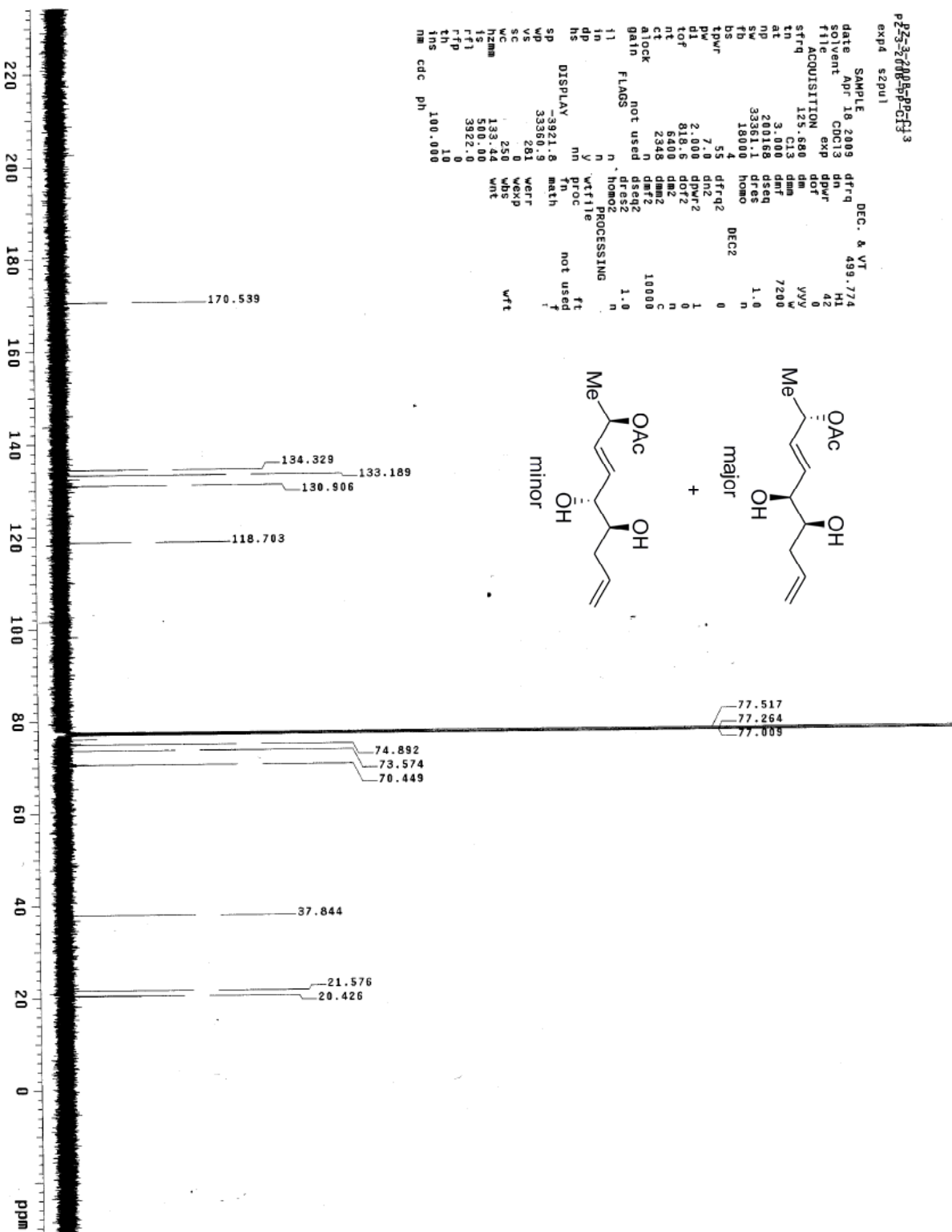
SAMPLE
date Apr 18 2009 dfreq DEC. 4 VT 499.774
solvent CDCl3 dn H1
file ACQUISITION exp dn H1
sfrq 499.774 dm dof 30
tn H1 dm nm 0
at 5.008 dmf c 200
nd 70058 dsen
sw 7005.9 dres 1.0
fb 4000 homo
bs 4
tdwr 57 dfrq2 DEC2
pv 4.6 dn2 0
sl 0 dpvr2 1
tof 497.0 dot2 0
nt 54 dm2 n
ct 54 dm2 c
atock n dmf2 200
gain not used dseq2
flans not used dres2
f1 n homo2 1.0
f2 n
f3 n PROCESSING
f4 n
f5 n
f6 n
f7 n
f8 n
f9 n
f10 n
f11 n
f12 n
f13 n
f14 n
f15 n
f16 n
f17 n
f18 n
f19 n
f20 n
f21 n
f22 n
f23 n
f24 n
f25 n
f26 n
f27 n
f28 n
f29 n
f30 n
f31 n
f32 n
f33 n
f34 n
f35 n
f36 n
f37 n
f38 n
f39 n
f40 n
f41 n
f42 n
f43 n
f44 n
f45 n
f46 n
f47 n
f48 n
f49 n
f50 n
f51 n
f52 n
f53 n
f54 n
f55 n
f56 n
f57 n
f58 n
f59 n
f60 n
f61 n
f62 n
f63 n
f64 n
f65 n
f66 n
f67 n
f68 n
f69 n
f70 n
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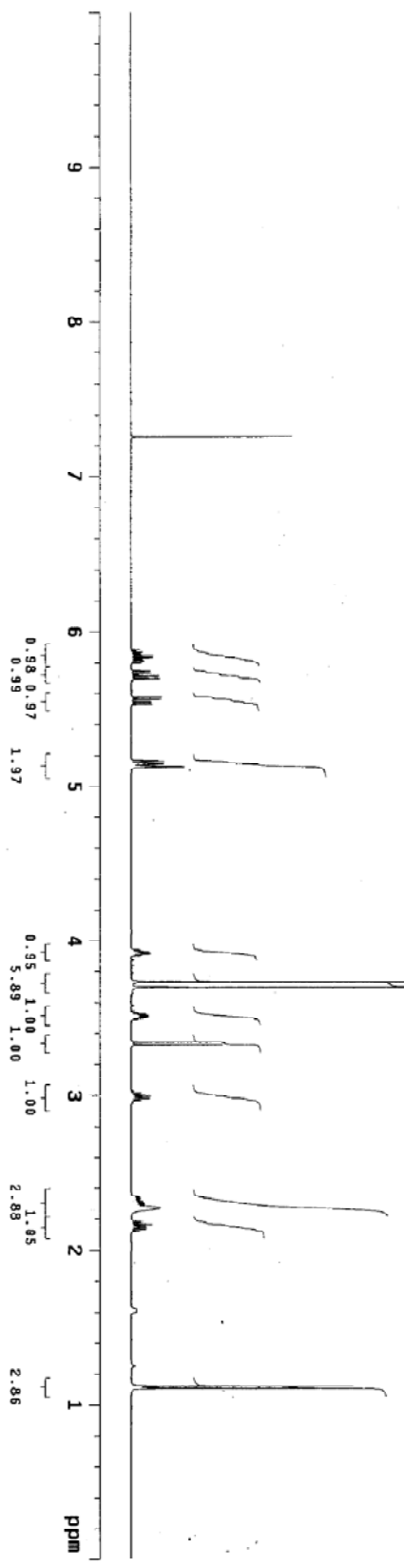
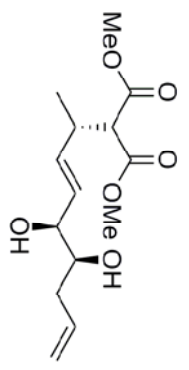
PZ-3-2008-PP-C13
 expd szpul

SAMPLE DEC. & VT
 date Apr 18 2009 dfrq 499.774
 solvent CDCl3 d1 42
 F1 ACQUISITION exp dpwr 0
 strq 125.680 dm yyy w
 tn C13 dmm 7200
 at 3.000 dmf
 np 200168 dseq 1.0
 sw 33361.1 dres
 fb 18000 homo
 bs 55 dfrq2 DEC2 0
 tpwr 7.0 dnc
 pv 2.00 dpr2 1
 dlf 8.00 dpr2
 tof 5400 dms
 nt 2348 dms2
 alock not used
 gain not used dres2 1.0
 n homo2 n
 in n wfile PROCESSING
 dp y n proc not used
 hs n math math
 DISPLAY -3821.8 wft
 sp wp 33360.9
 vs sc 281 wgrd
 v 250 wkd
 hzmm 133.44
 fs 500.00
 rfi 3922.0
 rfp 0
 th 10
 ns 100.000
 nm cdc ph



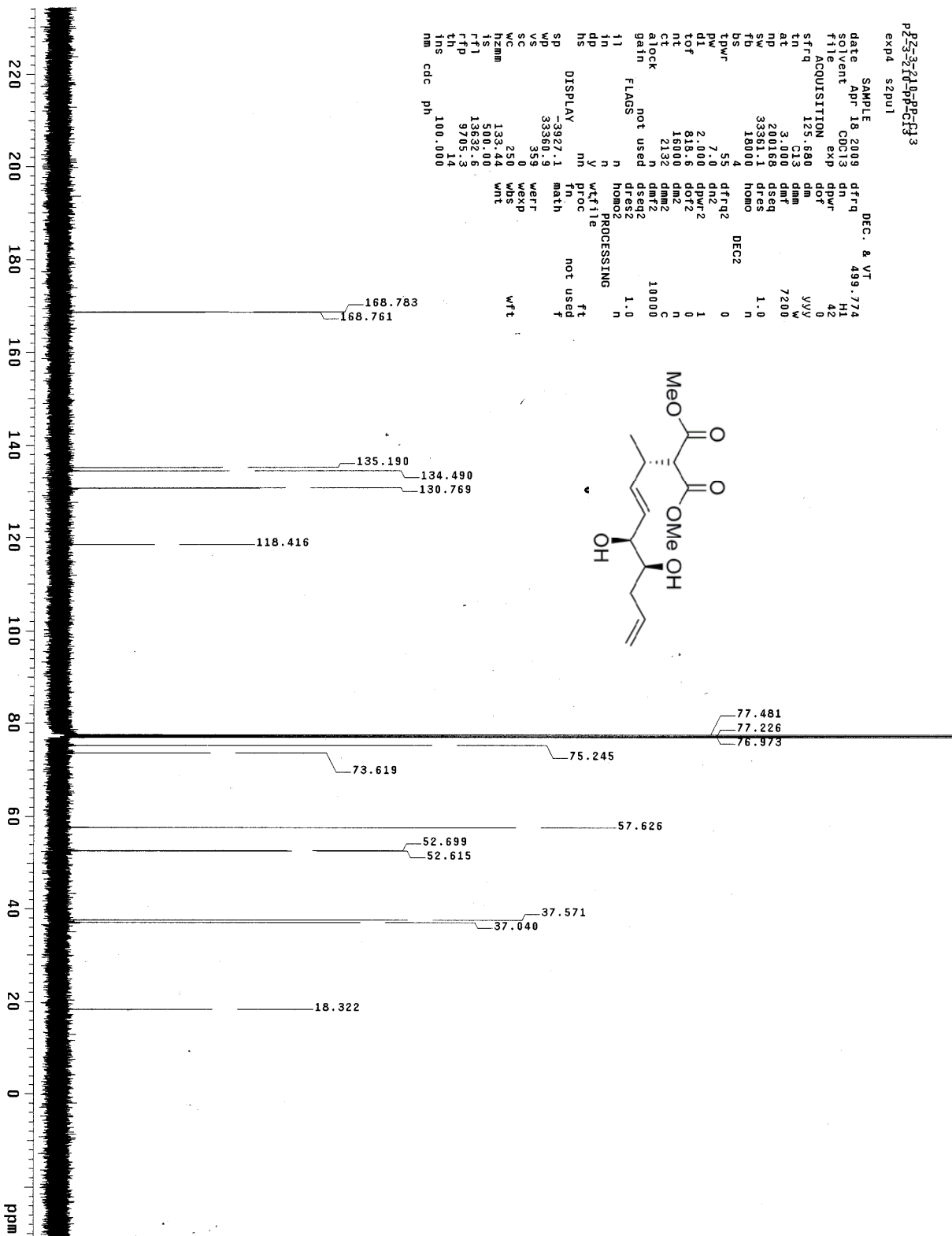
p273-3-21-14-PP
 exp4 s2pu1

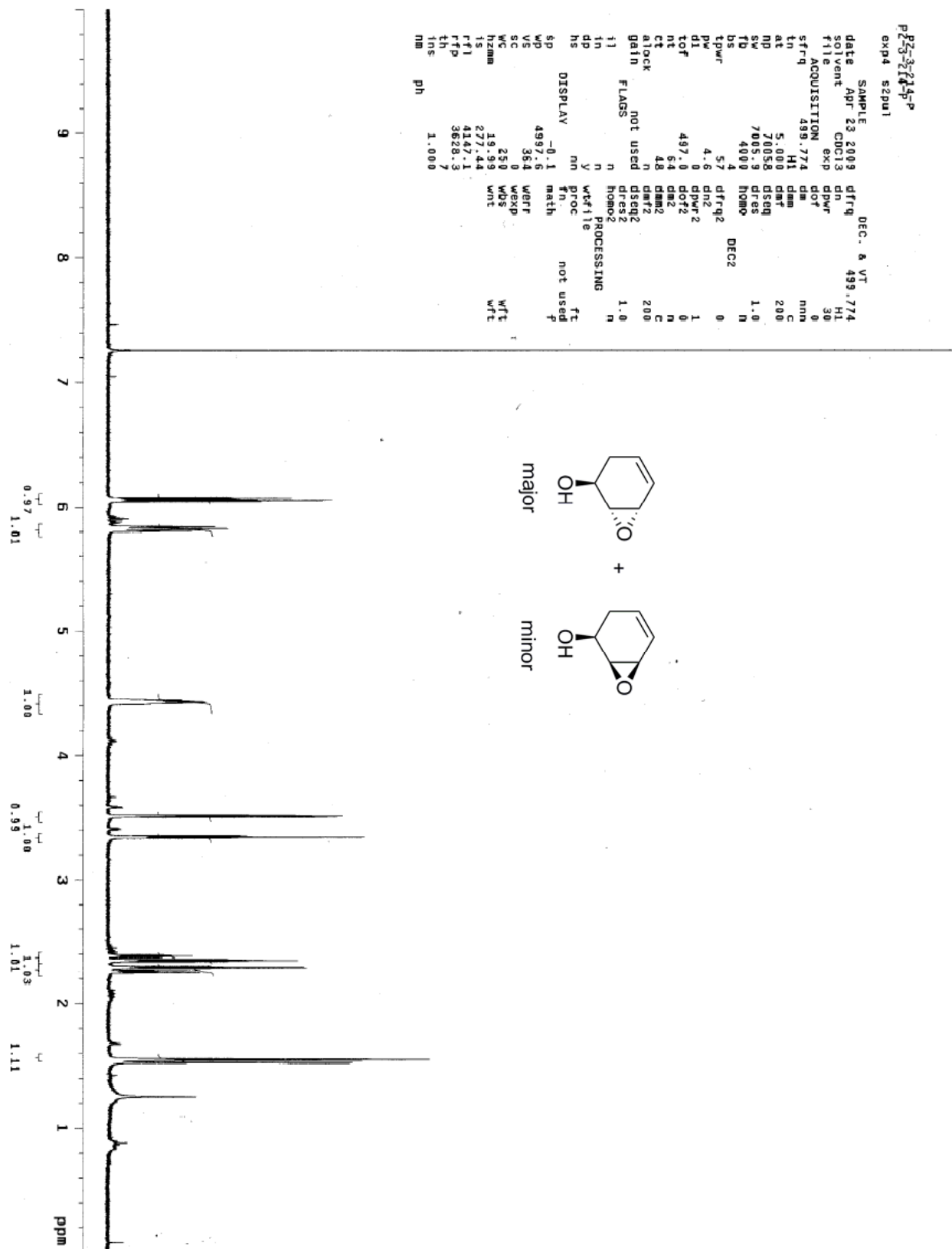
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solvent	CDCl3	dn	H1	30
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sfreq	499.774	dm	dof	0
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at	5.000	dmf	c	200
np	70058	dseq	res	1.0
sw	7065.9	dres	homo	n
fb	4000	homo	DECZ	0
bs	4	dfreq2	dn2	57
tpwr	57	dpwr2	dot2	0
pw	4.6	dm2	dmf2	64
dl	0	dm2	dmf2	48
tot	497.0	dm2	dmf2	48
nt	64	dm2	dmf2	48
ct	48	dm2	dmf2	48
atlock	n	dseq2	dres2	1.0
gain	not used	homo2	homo2	n
flags	not used	homo2	homo2	n
fl	n	PROCESsing	ft	not used
tn	n	wf file	fn	math
dp	an	proc	ft	not used
hs	an	math	wf	wt
sp	DISPLAY	math	wf	wt
wp	-0.1	werr	wf	wt
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sc	106	wbs	wf	wt
wc	0	wnt	wf	wt
h2nm	19.99			
rs	737.06			
rtf1	4147.8			
rtf2	3628.3			
tn	7			
ins	1.000			
nm				



PZ-3-210-PP-C13
 exp4 szpu1

SAMPLE DEC. & VT
 date Apr 18 2009 dfrq 499.774
 solvent CDCl3 dn H1
 file CDC13 exp dpwr 42
 ACQUISITION exp dof 0
 sfrq 125.580 om vvy
 tn 0.13 dmf W
 ac 3.0 dmf 7200
 su 200160 dresq
 fb 33961.1 dres 1.0
 bs 18000 homo 1.0
 4
 tpwr 55 dfrq2 DEC2
 pw 7.0 dn2 0
 di 2.000 dpwr2 1
 tof 818.6 dof2 0
 nt 16000 dm2 n
 ct 2132 dmm2 c
 alock n dmf2 10000
 gain not used dres2
 flags not used dres2 1.0
 11 n homo2
 in n wctfile PROCESSING
 dn y n
 ds n n
 DISPLAY 3927.1 ft
 sp mn proc not used f
 wf 33960 math
 wf 959 werr
 wf 0 wexp
 wf 250 wbs
 wf 133.44 wnt
 hzmm 500.00
 ts 13632.6
 rft1 9705.3
 rft 14
 th 14
 ins 100.000
 nm cdc ph





P23-3714-PC13
 exp4 s2pui

SAMPLE DEC. & VI
 date Apr 23 2009 dtrq 459.774
 solvent CDCl3 dn H1
 file exp dpwr 42
 ACQUISITION dot 0
 srrq 125.680 dm yyy
 tn C13 dm W
 at 3.000 dnt 7200
 np 200105 dscq 1.0
 sw 33581.1 ftes
 hd 18004 homo
 powr 55 dtrq2 DEC2 0
 dv 7.0 dn2
 dl 2.000 dpwr2 1
 tof 816.6 dot2 0
 nt 16000 cam2 n
 ct 1544 cam2 C
 atlock n cam2 10000
 gain not used dscq2 1.0
 11 n homo2 1.0
 1n n
 1p y
 1b PROCESsing 2.00
 hs n
 DISPLAY nn wfile ft
 sp 3925.5 T proc not used f
 vp 33581.9 math
 sc 445 0
 wc 0 wrr
 hzmm 133.44 wxp
 ts 500.00 wds
 rfp 13631.1 wtt
 th 9705.3
 17
 100.060
 nm cdc ph

