Supplementary information: Enantioselective Synthesis of *epi*-Gallocatechin-3-gallate (EGCG), the Active Polyphenol Component from Green Tea. Lianhai Li, Tak Hang Chan*, Department of Chemistry, McGill University, Montreal, Quebec, H3A 2K6 Canada

Experimental

General Procedures. Chemicals were used as obtained from commercial sources unless specified otherwise. CH_2Cl_2 was freshly distilled over CaH_2 and used immediately. THF was freshly distilled over Na/benzophenone and used immediately. Anhydrous DMF was obtained by distillation over CaH_2 under vacuum. Literature procedures were used for preparation of the following chemicals: Dess-Martin periodinane[1], methyl 3,4,5-tris(benzyloxy)benzoate[2], 3,4,5-tris(benzyloxy)benzoic acid[3]. The synthesis of naturally occurring (-)-EGCG is described in detail as a representative example. The procedure for the synthesis of racemic ***(1R*, 2R*)*-3-[2,4-bis-(benzyloxy)-6-hydroxyphenyl]-1-[3,4,5-tris(benzyloxy)phenyl]propane-1,2-diol (7c),** which was used to prepare racemic EGCG, is also presented.

Preparation of Silica Gel Supported H_2SO_4 for the Friedel-Crafts Cinnamylation: Silica gel (30 g) was added to a mixture of H_2SO_4 (98%, 10 g) and hexane (100 mL); the mixture was shaken for 5 min, and the solvent was removed with an evaporator. The residue was left in the rotavac for 1 h at 30 °C, then hexane (100 mL) was added, and the mixture was shaken vigorously by hand for 5 min. The solvent was removed by rotary evaporation at 60 °C for 4 h. The resulting catalyst was kept sealed for future use.

3,4,5-Tris(benzyloxy)benzyl alcohol: LAH (1.50 g, 39 mmol) was added in 10 batches to a stirred solution of methyl 3,4,5-tris(benzyloxy)benzoate (18.4 g, 40 mmol) in THF (125 mL) at 0 °C under an Ar atmosphere. The mixture was stirred at rt for 2 h after the addition. Then hexane (125 mL) was added. This was followed by dropwise addition of a saturated solution of NH_4HF_2 (5 mL). The mixture was stirred at rt for 1 h and then filtered and washed with ethyl acetate. The filtrate was dried (Na_2SO_4) and evaporated to afford the desired alcohol (15.4 g, 89% yield) of sufficient purity for the next step. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.52-7.25 (m, 15 H), 6.67 (s, 2 H), 5.11 (s, 4H), 5.06 (s, 2 H), 4.56 (s, 2H), 1.91 (br s, 1H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 153.2, 138.0, 137.8, 137.3, 137.0, 128.9, 128.8, 128.4, 128.1, 128.0, 127.7, 106.4, 75.5, 71.3, 65.6.

Ethyl (*E*)-3,4,5-tris(benzyloxy)cinnamate: At rt under an Ar atmosphere, PDC (6.8 g, 18.1 mmol) was added to a stirred mixture of 3,4,5-tris(benzyloxy)benzyl alcohol (15.4 g, 36.1 mmol) with 4 A molecular sieves (10 g) in CH_2Cl_2 (150 mL). The mixture was stirred at rt overnight, and Et_2O (150 mL) was added to quench the reaction. The mixture was filtered through a layer of silica gel, and the solid was thoroughly washed with Et_2O. The solvent was evaporated, and the residue was dried in high vacuum for 2 h to yield the aldehyde as white solid. The aldehyde thus obtained was dissolved in THF (120 mL), and to this solution triethyl phosphonoacetate (9.43 g, 42 mmol) was added. The mixture was cooled in an ice bath, and NaH (1.68 g, 60% dispersion in mineral oil, 42 mmol) was added to this solution in 10 batches. The reaction was allowed to proceed at rt for 2 h and then sat. $NaHCO_3$ solution was added. The organic layer was separated, and the aqueous layer was extracted with ethyl acetate. The organic phases were combined, dried (Na_2SO_4), filtered and evaporated to afford a solid. This solid was washed with hexane

to remove the mineral oil and the excess of triethyl phosphonoacetate to yield the desired compound (16.3 g, 91% yield) in sufficient purity for the next step. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.56 (d, J = 16.4 Hz, 1 H), 7.45-7.25 (m, 15 H), 6.83 (s, 2 H), 6.29 (d, J = 16.4 Hz, 1 H), 5.13 (s, 4H), 5.11 (s, 2 H), 5.26 (q, J = 7.6 Hz, 2H), 1.35 (t, J = 7.6 Hz, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 167.2, 153.2, 144.7, 140.6, 137.8, 137.0, 130.2, 128.8, 128.6, 128.5, 128.3, 128.2, 127.7, 117.7, 107.9, 75.5, 71.4, 60.7, 14.6.

(*E*)-3,4,5-Tris(benzyloxy)cinnamyl alcohol (4c): To a solution of ethyl (*E*)-3,4,5-tris(benzyloxy)cinnamate (16.3 g, 33 mmol) in THF (125 mL) at -78 °C under an Ar atmosphere, DIBAL (48 mL, 1.5 M in toluene, 72 mmol) was added dropwise. The mixture was stirred at -78 °C for 1 h and then at rt for another 1 h. Then the mixture was cooled to 0 °C and poured into a stirred mixture of hexane (250 mL) and saturated aqueous Na_2SO_4 solution (5 mL). The resulting mixture was stirred until a large quantity of solid was formed. The mixture was filtered, and the solid was thoroughly washed with ethyl acetate. The organic solutions were combined and dried (Na_2SO_4). The residue after evaporation of the solvent was washed again with hexane, and the solid was collected to afford the desired product (14.2 g, 95% yield). ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.45-7.25 (m, 15 H), 6.68 (s, 2 H), 6.48 (d, J = 16.4 Hz, 1 H), 6.18 (dt, J = 16.4 , 5.2 Hz, 1 H), 5.11 (s, 4H), 5.06 (s, 2 H), 4.29 (d, J = 5.2 Hz, 2H), 1.6 (br s, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 153.2, 138.5, 137.3, 132.6, 131.2, 128.9, 128.8, 128.4, 128.2, 128.1, 128.0, 127.6, 106.4, 75.5, 71.4, 63.7.

[3,5-Bis(benzyloxy)-2-[3-[3,4,5-tris(benzyloxy)phenyl]allyl]phenoxy]-*tert*-butyl-dimethylsilane (18): At rt under an Ar atmosphere, 25% H_2SO_4/SiO_2 (160 mg, 0.4 mmol) was added in one batch to the stirred mixture of 3,5-bis(benzyloxy)phenol (306

mg, 1 mmol) and (*E*)-3,4,5-tris(benzyloxy)cinnamyl alcohol (452 mg, 1 mmol) in a solvent mixture of CH_2Cl_2 (2 mL) and CS_2 (2 mL). The resulting mixture was stirred at rt for 3 h and then filtered through a layer of silica gel. The solvent was evaporated, and the residue was purified by column chromatography on silica gel (5% EtOAc/C_6H_6) to afford (*E*)-3-[2,4-bis(benzyloxy)6-hydroxyphenyl]-1-[3,4,5-tris(benzyloxy)phenyl]-propene (468 mg) which was not pure but was used as obtained in the next step. The alkene thus obtained was dissolved in dry DMF (5 mL), and to this solution imidazole (145 mg) and TBSCl (165 mg) were added successively. The resulting mixture was stirred at rt overnight, and then saturated Na_2CO_3 solution was added to quench the reaction. The mixture was extracted with EtOAc. The organic layers were combined, dried (Na_2SO_4), and evaporated. The residue was purified by flash chromatography on silica gel (benzene) to afford the desired compound (428 mg). The ^{1}H NMR and ^{13}C NMR spectra showed that the compound was contaminated by some impurities (about 10% according to ^{1}H NMR), which were hard to be removed by flash chromatography. This material was used as obtained above without further purification for the next step. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.45-7.25 (m, 25 H), 6.58 (s, 2 H), 6.32-6.10 (m, 4 H), 5.06 (s, 4H), , 5.03 (s, 4 H), 5.01 (s, 2H), 3.49 (d, J = 4.4 Hz, 1 H), 1.00 (s, 9 H), 0.20 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 158.6, 156.4, 155.0, 153.0, 138.2, 137.7, 137.5, 137.3, 134.3, 129.6, 129.2, 128.9, 128.8, 128.7, 128.6, 128.5, 128.4, 128.2, 128.1, 128.0, 127.9, 127.7, 127.6, 127.5, 112.2, 106.0, 98.7, 94.1, 75.5, 71.4, 71.2, 70.4, 70.3, 27.1, 26.1, -3.8.

(+)-(*1R,2R*)-3-[2,4-Bis(benzyloxy)-6-hydroxyphenyl]-1-[3,4,5-tris(benzyloxy)phenyl]propane-1,2-diol ((+)-7c): AD-mix-α (1.42 g) and methanesulfonamide (95 mg) were dissolved in a solvent mixture of *t*-BuOH (6 mL) and

H_2O (6 mL). The resulting mixture was stirred at rt for 5 min. Then the mixture was cooled to 0 °C and a solution of compound **18** (428 mg) in dichloromethane (6 mL) was added. After the mixture had been stirred overnight, a total of four batches of methanesulfonamide (48 mg each) and AD-mix-α (0.71 g each) were added in 24 h intervals. After another 24 h of stirring at 0 °C, TLC showed that the reaction was completed. Then a 10% $Na_2S_2O_3$ solution was added to quench the reaction. The mixture was filtered through a layer of Celite, and the filtrate was extracted with EtOAc. The organic layer was combined, dried (Na_2SO_4) and evaporated. The residue was re-dissolved in THF (3 mL), and TBAF (1 mL, 1 M in THF) was added. The resulting mixture was stirred at rt for 4 h, and then saturated sodium bicarbonate solution was added. The mixture was extracted with EtOAc, and the organic layers were combined, dried (Na_2SO_4) and evaporated. The residue was purified by flash chromatography on silica gel (10-25% EtOAc/benzene) to afford the desired compound (318 mg, 41% yield based on **4c**) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.45-7.25 (m, 25 H), 6.59 (s, 2 H), 6.26 (d, J = 2.0 Hz, 1 H), 6.21 (d, J = 2.0 Hz, 1 H), 5.05-4.95 (m, 8H), 4.89 (s, 2 H), 4.43 (d, J = 5.6 Hz, 1 H), 3.92 (ddd, J = 8.4, 5.6, 3.6 Hz, 1 H), 2.90 (dd, J = 14.8, 3.6 Hz, 1 H), 2.75 (dd, J = 14.8, 8.4 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.3, 158.1, 157.5, 153.0, 138.3, 138.0, 137.1, 136.3, 128.9, 128.8, 128.7, 128.6, 128.4, 128.3, 128.1, 128.0, 127.9, 127.7, 126.8, 106.4, 106.2, 96.0, 93.7, 77.5, 77.1, 76.9, 75.3, 71.3, 70.0, 26.9; $[\alpha]_D$ = +11.54 (c = 1.0, $CHCl_3$). By using the same procedure, **(-)-(*1S,2S*)-3-[2,4-bis(benzyloxy)-6-hydroxyphenyl]-1-[3,4,5-tris(benzyloxy)phenyl]propane-1,2-diol ((-)-7c)** ($[\alpha]_D$ = -11.49 (c = 1.0, $CHCl_3$)) was prepared with identical NMR spectra as the (+)-isomer.

***(1R*,2R*)*-3-[2,4-Bis(benzyloxy)-6-hydroxyphenyl]1-[3,4,5-tris(benzyloxy)phenyl]propane-1,2-diol (7c):** At rt and under an Ar atmosphere, 25% H_2SO_4/SiO_2 (160 mg, 0.4 mmol) was added in one batch to a stirred mixture of 3,5-bis(benzyloxy)phenol[4] (306 mg, 1 mmol) and (*E*)-3,4,5-tris(benzyloxy)cinnamyl alcohol (452 mg, 1 mmol) in a solvent mixture of CH_2Cl_2 (2mL) and CS_2 (2 mL). The resulting mixture was stirred at rt for 3 h and then filtered through a layer of silica gel. The solvent was evaporated, and the residue was purified by column chromatography on silica gel (5% EtOAc/C_6H_6) to afford (*E*)-3-[2,4-bis(benzyloxy)-6-hydroxyphenyl]1-[3,4,5-tris(benzyloxy)phenyl]propene (463 mg which was not pure but was used as obtained in the next step. The alkene thus obtained was dissolved in acetone (4.5 mL), and to this solution were added water (1.5 mL), NMO (130 mg, 50% in water) and OsO_4 (0.1 mL, as a suspension in 2-propanol which contained 10 mg of OsO_4 per mL, well-shaken before use). The resulting mixture was stirred at rt overnight, and then 10% $Na_2S_2O_3$ solution was added. The mixture was filtered through a layer of celite, and the filtrate was extracted with EtOAc. The organic layers were combined, dried (Na_2SO_4) and evaporated. The residue was purified by flash chromatography on silica gel (10-25% EtOAc/benzene) to afford the desired compound (340 mg, 50% yield) with identical NMR spectra as (+)-**7c**.

(-)-(*2S,3R*)-*trans*-5,7-Bis(benzyloxy)-2-[3,4,5-tris(benzyloxy)phenyl]chroman-3-ol ((-)-13c): To a solution of (-)-**7c** (318 mg, 0.41 mmol) in 1,2-dichloroethane (5 mL) was added triethyl orthoformate (0.12 mL), followed by PPTS (60 mg, 0.24 mmol). The mixture was stirred at rt for 20 min and then heated to 60 ^{0}C for about 8 h until TLC showed that the reaction had been completed. The reaction mixture was filtered through

a layer of silica gel and evaporated. The residue was redissolved in DME (4 mL) and MeOH (4 mL), K_2CO_3 (60 mg) was added, and the mixture was stirred at rt overnight. The solvent was evaporated, and the residue was purified by flash chromatography on silica gel (5% EtOAc/C_6H_6) to afford the desired product (233 mg, 75% yield) as a white solid. ^{1}H NMR ($CDCl_3$, 300 MHz) δ 7.55-7.30 (m, 25 H), 6.78 (s, 2 H), 6.34 (d, J = 2.4 Hz, 1 H), 6.29 (d, J = 2.4 Hz, 1 H), 5.15 (s, 2 H), 5.14 (s, 2 H), 5.11 (s, 2 H), 5.10 (s, 2 H), 5.07 (s, 2 H), 5.04 (s, 2 H), 4.65 (d, J = 8.1 Hz, 1 H), 4.01 (ddd, J = 9.0, 8.1, 6.0 Hz, 1 H), 3.15 (dd, J = 16.5, 6.0 Hz, 1 H), 2.75 (dd, J = 16.5, 9.0 Hz, 1 H), 1.76 (br s, 2 H); ^{13}C NMR ($CDCl_3$, 75 MHz) δ 159.1, 158.0, 155.4, 153.3, 138.9, 138.0, 137.2, 137.1, 133.6, 129.0, 128.9, 128.8, 128.7, 128.5, 128.3, 128.2, 128.1, 128.0, 127.9. 127.8, 127.4, 106.9, 102.6, 94.6, 94.2, 82.1, 75.5, 71.4, 70.4, 70.2, 68.5, 27.9; $[\alpha]_D$ = -7.12 (c = 1.0, $CHCl_3$). By using the same procedure as described above, (+)-**13c** ($[\alpha]_D$ = + 7.21 (c = 1.0, $CHCl_3$)) and the racemate **13c** were also prepared with identical NMR spectra as the (-)- isomer.

(-)-(*2S,3S*)-*cis*-5,7-Bis(bnzyloxy)-2-[3,4,5-tris(benzyloxy)phenyl]chroman-3-ol ((-)-15c): Dess-Martin reagent (150 mg) was added in one batch to a stirred solution of (-)-**13c** (180 mg, 0.24 mmol) in CH_2Cl_2 (5 mL) under an Ar atmosphere, and the mixture was stirred at rt for 2 h. Then saturated aqueous sodium bicarbonate solution (3 mL) and 10% aqueous $Na_2S_2O_3$ aqueous solution (3 mL) were added to quench the reaction. The resulting mixture was stirred until a clear two phase solution formed, the organic layer was separated, and the aqueous layer was extracted with EtOAc. The combined organic layers were dried (Na_2SO_4) and evaporated. The residue was dissolved in C_6H_6 and was

filtered through a layer of silica gel to remove the pink color. The filtrate was evaporated, the residue (about 180 mg) was dissolved in THF (3 mL), and the solution was cooled to -78 °C. Then L-selectride (0.33 mL, 1 M solution in THF, 0.33 mmol) was added dropwise under an Ar atmosphere. The resulting solution was stirred at -78 °C for 6 h and then at rt for 2 h. The reaction was quenched by the addition of saturated aqueous sodium bicarbonate solution (3 mL), and the resulting mixture was stirred at rt overnight. The organic layer was separated and the aqueous layer was extracted with ethyl acetate. The combined organic layers were dried (Na_2SO_4) and evaporated. The residue was purified by flash chromatography on silica gel (5% EtOAc/C_6H_6) to afford the desired product (145 mg, 80% yield) as a white solid. ^{1}H NMR ($CDCl_3$, 400 MHz) δ 7.55-7.30 (m, 25 H), 6.86 (s, 2 H), 6.34 (d, J = 2.4 Hz, 1 H), 6.33 (d, J = 2.4 Hz, 1 H), 5.15 (s, 4 H), 5.11 (s, 2 H), 5.05 (s, 4 H), 4.92 (s, 1 H), 4.24 (s, , 1 H), 3.05 (d, J = 17.2 Hz, 1 H), 2.95 (dd, J = 17.2, 4.0 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 159.0, 158.6, 155.4, 153.3, 138.5, 138.1, 137.2, 137.1, 134.1, 128.9, 128.8, 128.7, 128.5, 128.3, 128.2, 128.1, 127.9, 127.5, 106.2, 101.2, 94.9, 94.4, 78.8, 75.5, 71.5, 70.4, 70.2, 66.7, 28.4; $[\alpha]_D$ = -9.62 (c = 1.0, $CHCl_3$). By using the same procedure as described above, (+)-**15c** ($[\alpha]_D$ = + 9.41 (c = 1.0, $CHCl_3$)) and racemate **15c** were also prepared with identical NMR spectra as the (-)-isomer.

(-)-(*2S,3S*)-*cis*-5,7-Bis(benzyloxy)-2-[3,4,5-tris(benzyloxy)phenyl]chroman-3-yl 3,4,5-Tris(benzyloxy)benzoate ((-)-17c): A solution of 3,4,5-tris(benzyloxy)benzoic acid (170 mg, 0.39 mmol) was refluxed with $(COCl)_2$ (0.5 mL) in CH_2Cl_2 (6 mL) for 2 h. The excess of $(COCl)_2$ and the solvent were removed by distillation and the residue was dried under vacuum for 3 h and dissolved in CH_2Cl_2 (2 mL). This solution was added

dropwise to a solution of (-)-**15c** (145 mg, 0.19 mmol) and DMAP (58 mg, 0.47 mmol) in dichloromethane (15 mL) at 0 °C. The mixture was stirred at rt overnight, then $NaHCO_3/H_2O$ was added, the organic layer was separated, and the aqueous layer was extracted with ethyl acetate. The organic layers were combined, dried (Na_2SO_4) and evaporated. The residue was purified by flash chromatography on silica gel ($EtOAc/C_6H_6$) to afford the desired compound (188 mg, 83% yield). ^1H NMR ($CDCl_3$, 400 MHz) δ 7.5-7.20 (m, 40 H), 6.76 (s, 2 H), 6.44 (d, J = 2.4 Hz, 1 H), 6.38 (d, J = 2.4 Hz, 1 H), 5.72-5.70 (m, 1 H), 5.22-4.90 (m, 13 H), 4.82 and 4.69 (AB q, J = 11.2 Hz, 4 H), 3.16 (dd, J = 17.6, 4.4 Hz, 1 H), 3.10 (dd, J = 17.6, 2.4 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 165.1, 159.1, 158.3, 155.9, 153.1, 152.6, 142.9, 138.6, 138.0, 137.7, 137.1, 137.0, 136.9, 136.7, 133.5, 128.9 (2), 128.8, 128.7, 128.6 (2), 128.5 128.4, 128.3 (2), 128.2 (2), 128.1 (2), 128.0, 127.8 (2), 127.5, 125.2, 109.3, 106.9, 101.2, 94.8, 94.2, 78.2, 75.4, 75.3, 71.4, 71.3, 70.4, 70.2, 68.5, 26.5; $[\alpha]_D$ = -44.62 (c = 1.0, $CHCl_3$). By using the same procedure as described above, (+)-**17c** ($[\alpha]_D$ = + 45.12 (c = 1.1, $CHCl_3$)) and the racemate **17c** were also prepared with identical NMR spectra as the (-)-isomer.

Preparation of (-)-EGCG ((-)-3b): Under an H_2 atmosphere, $Pd(OH)_2$ (50 mg, 20% on carbon) was added to a solution of (-)-**17c** (60 mg, 0.051 mmol) in a solvent mixture of THF/MeOH (1:1 v/v, 14 mL). The resulting mixture was stirred at rt until TLC showed that the reaction was completed (about 6 h). Then the reaction mixture was filtered through cotton to remove the catalyst. The filtrate was evaporated, and the residue was purified by flash chromatography on silica gel with $EtOAc/CH_2Cl_2$ (7:3) to afford the

desired compound (21 mg, 91% yield) as a white solid. ^{1}H NMR (acetone-d_6/D_2O (2:1), 400 MHz) δ 6.96 (s, 2 H), 6.60 (s, 2 H), 5.97 (d, J = 2.4 Hz, 1 H), 5.36 (d, J = 1.2 Hz, 1 H), 4.96 (s, 1 H), 2.94 (dd, J = 17.2, 4.4 Hz, 1 H), 3.10 (dd, J = 17.2, 1.6 Hz, 1 H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ 166.4, 156.6, 156.0, 145.5, 145.2, 138.5, 132.4, 129.9, 120.5, 109.4, 106.1, 98.1, 95.8, 95.0, 77.4, 69.5, 25.9; $[\alpha]_D$ = -148.1 (c = 1.0, THF). The compound obtained as described above had completely identical NMR spectra as the commercially available sample (Sigma, $[\alpha]_D$ = -147.6 (c = 1.0, THF)). By using the same procedure as described above, **(+)-EGCG ((+)-3b)** ($[\alpha]_D$ = -148.2 (c = 1.0, THF)) and the racemic form of EGCG (**3b**) were also obtained.

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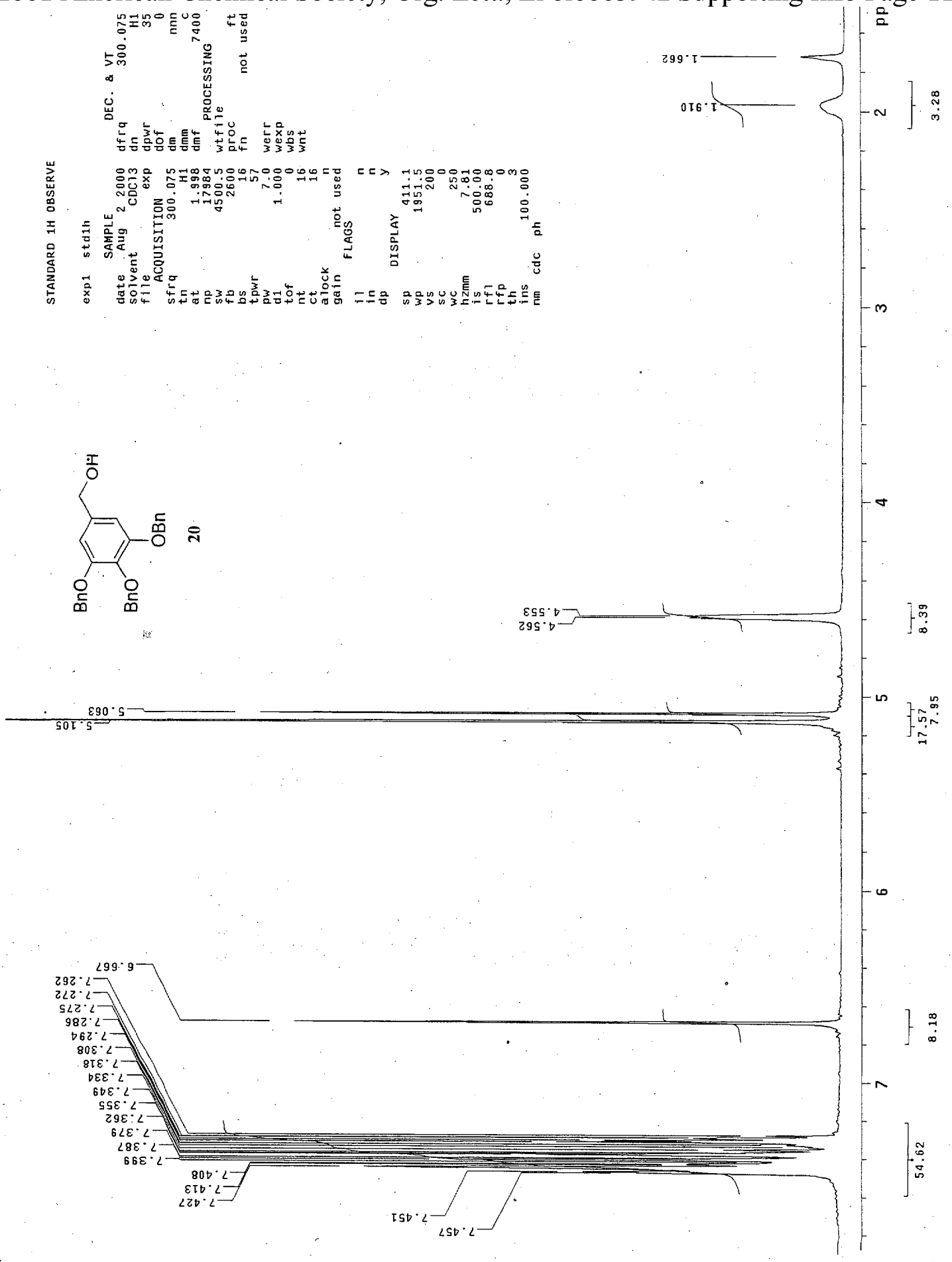

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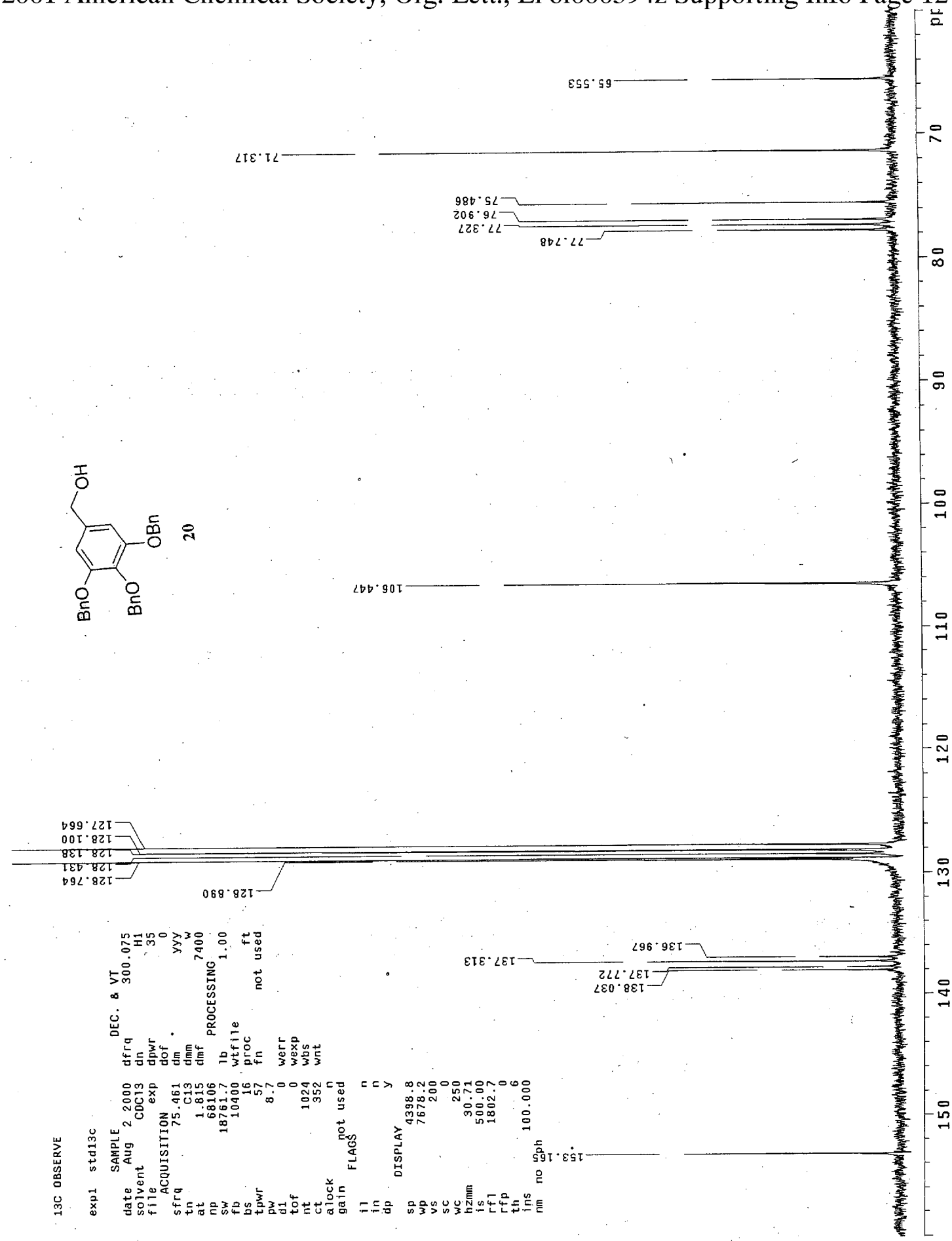
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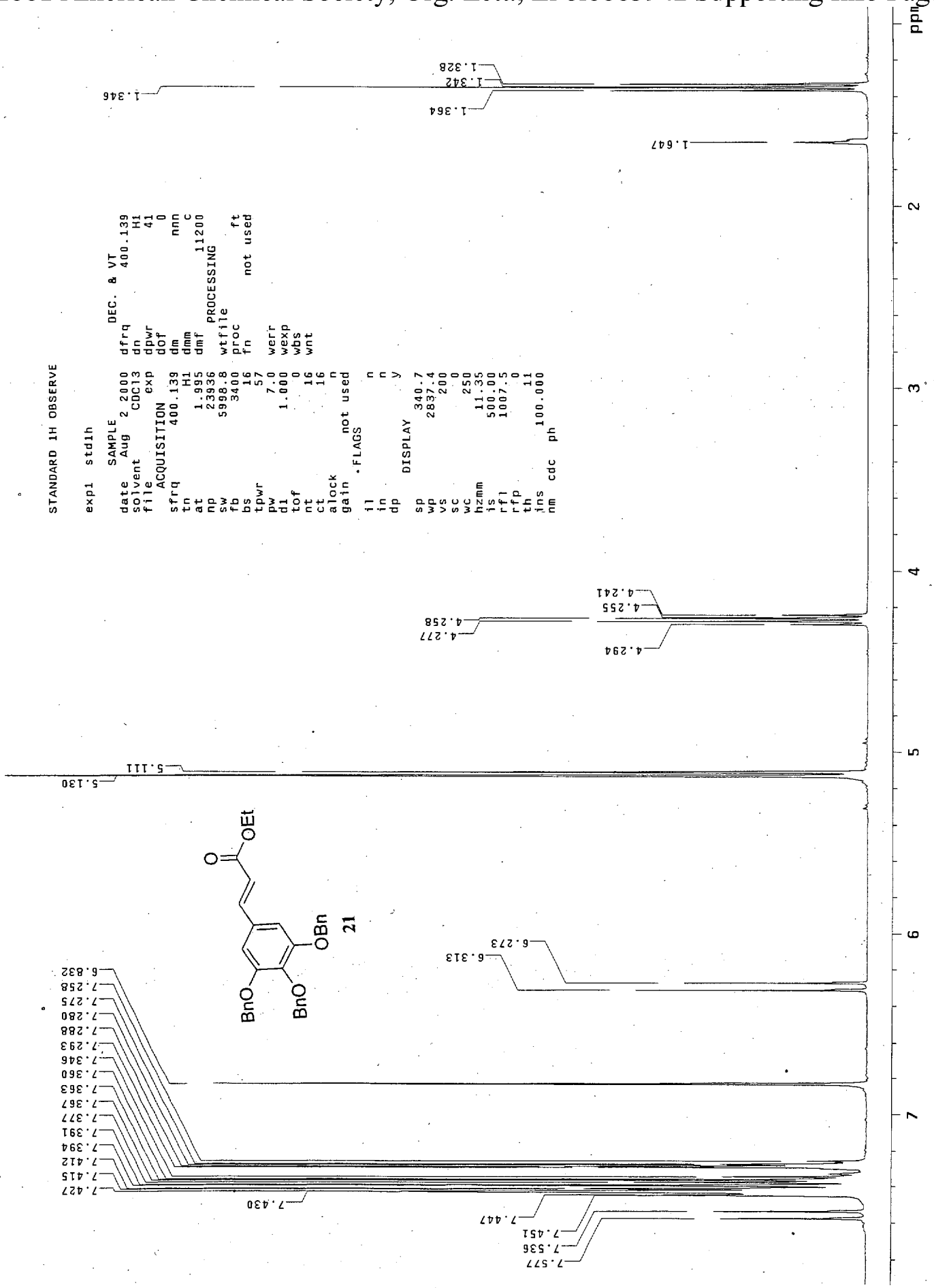
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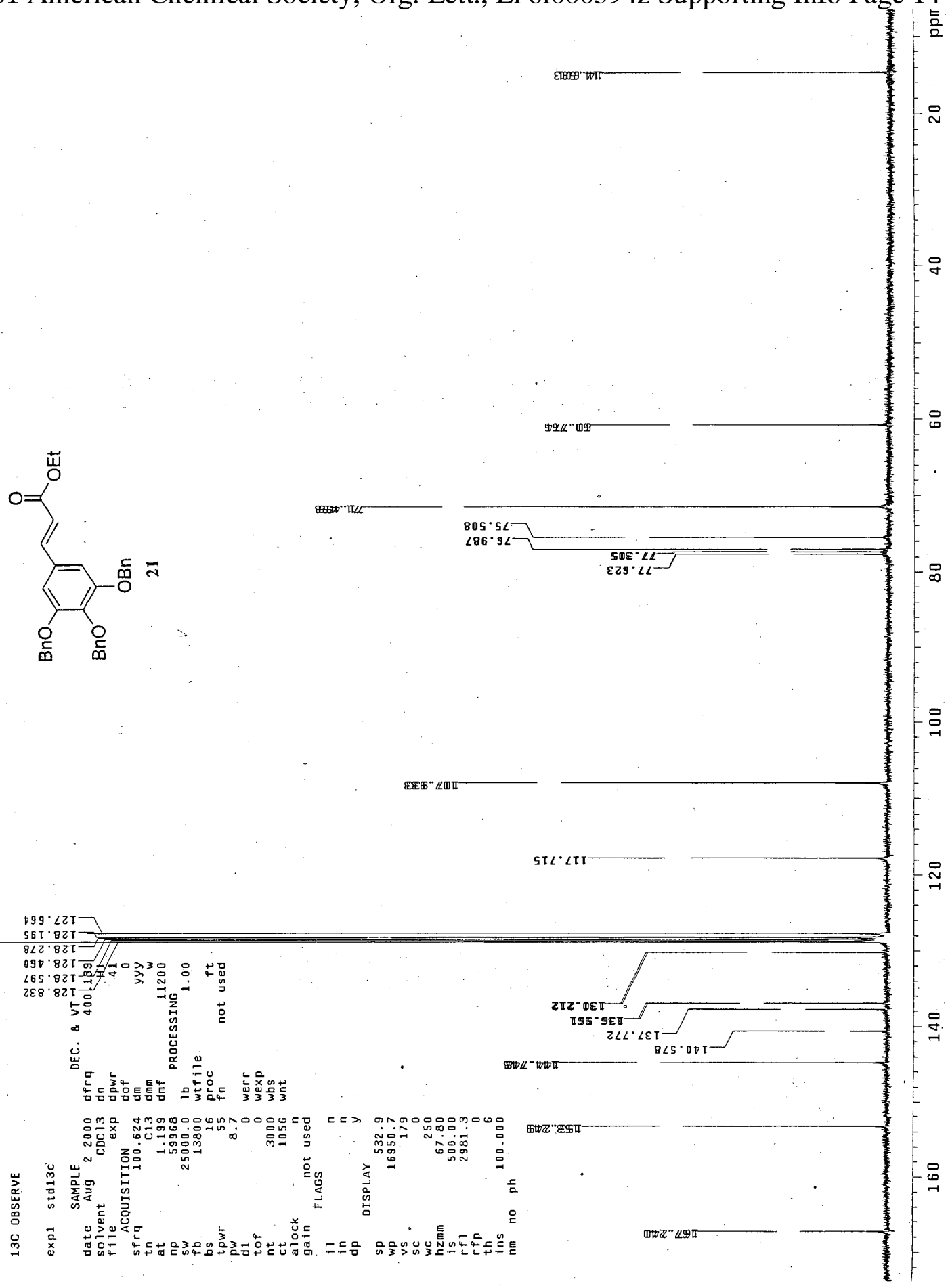
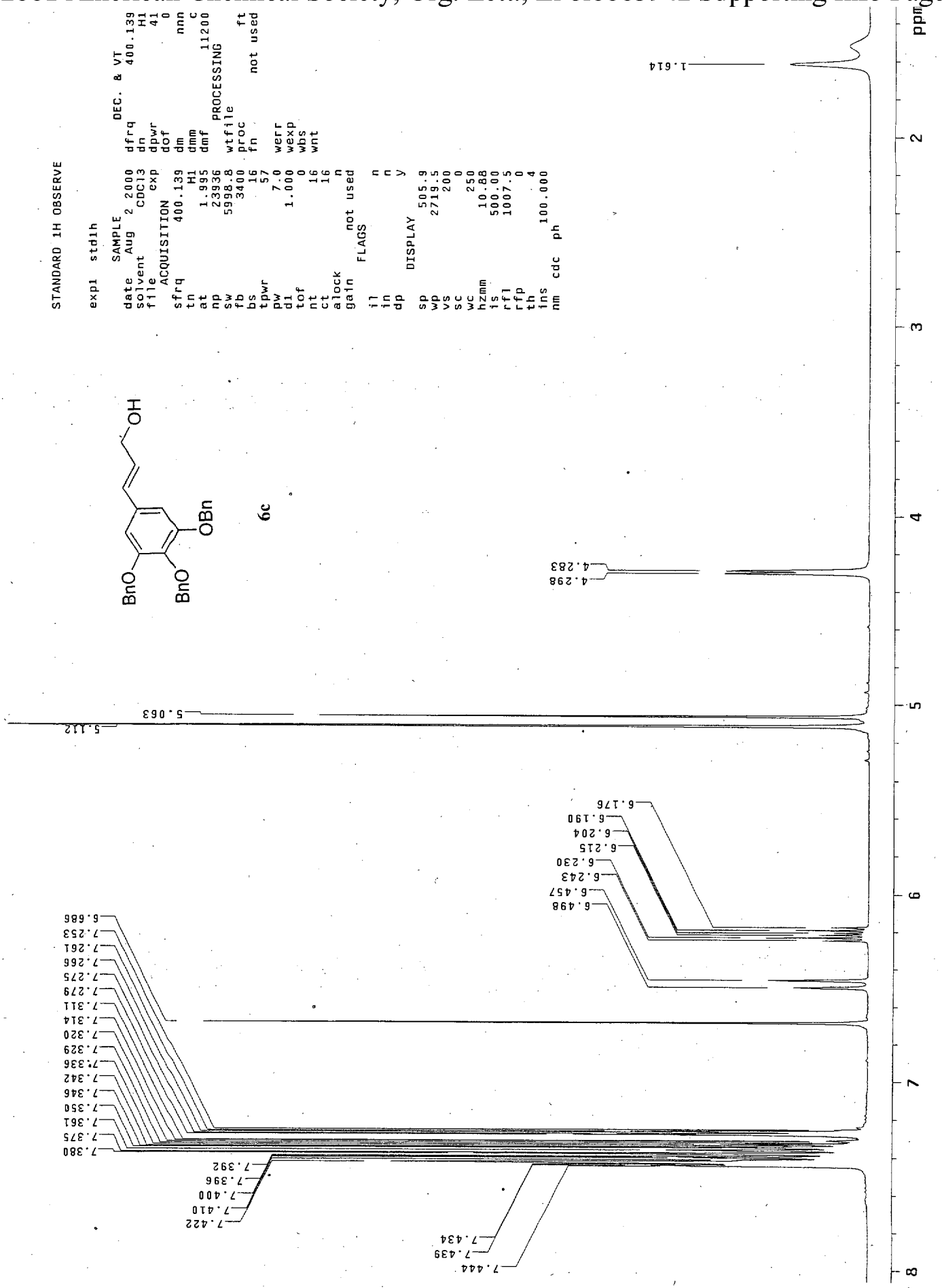
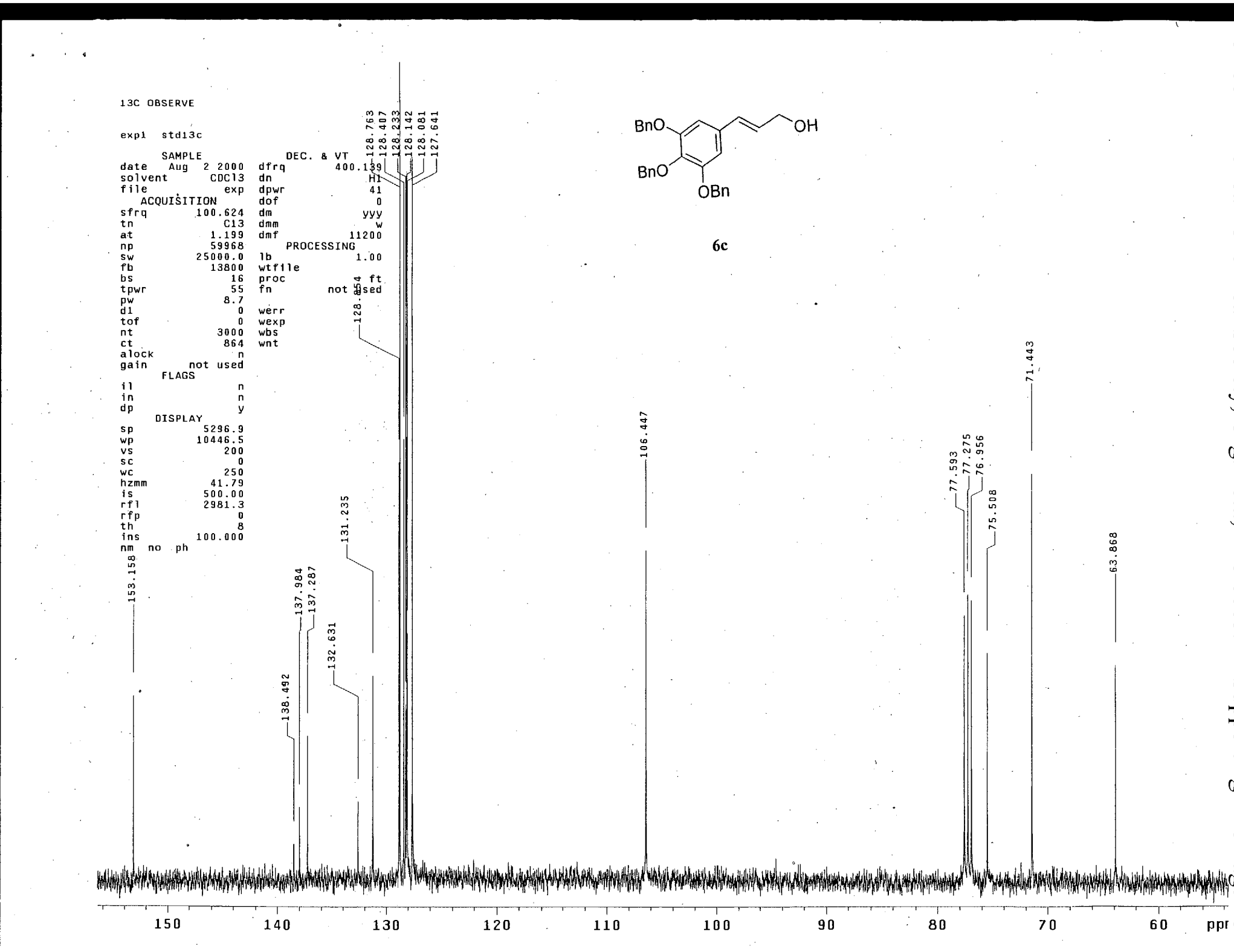
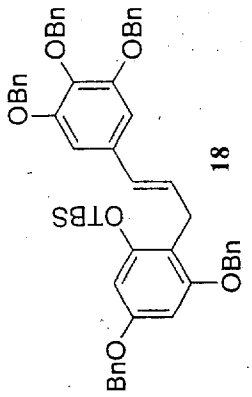

O
OEt
OBn
BnO
BnO
21
ppm
20
40
60
80
100
120
140
160
14.6813
60.7764
71.4888
75.508
76.987
77.305
77.623
107.9383
117.715
127.664
128.195
128.278
128.460
128.597
128.832
130.212
136.961
137.772
140.578
144.7488
153.2249
167.2240
13C OBSERVE
exp1 std13c
SAMPLE
date Aug 2 2000
solvent CDCl3
file exp
ACQUISITION
sfrq 100.624
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 16
tpwr 55
pw 8.7
d1 0
tof 0
nt 3000
ct 1056
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp 532.9
wp 16950.7
vs 179
sc 0
wc 250
hzmm 67.80
is 500.00
rfl 2981.3
rfp 0
th 6
ins 100.000
nm no ph
DEC. & VT
dfrq 400.139
dn H1
dpwr 41
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile ft
proc not used
fn
werr
wexp
wbs
wnt

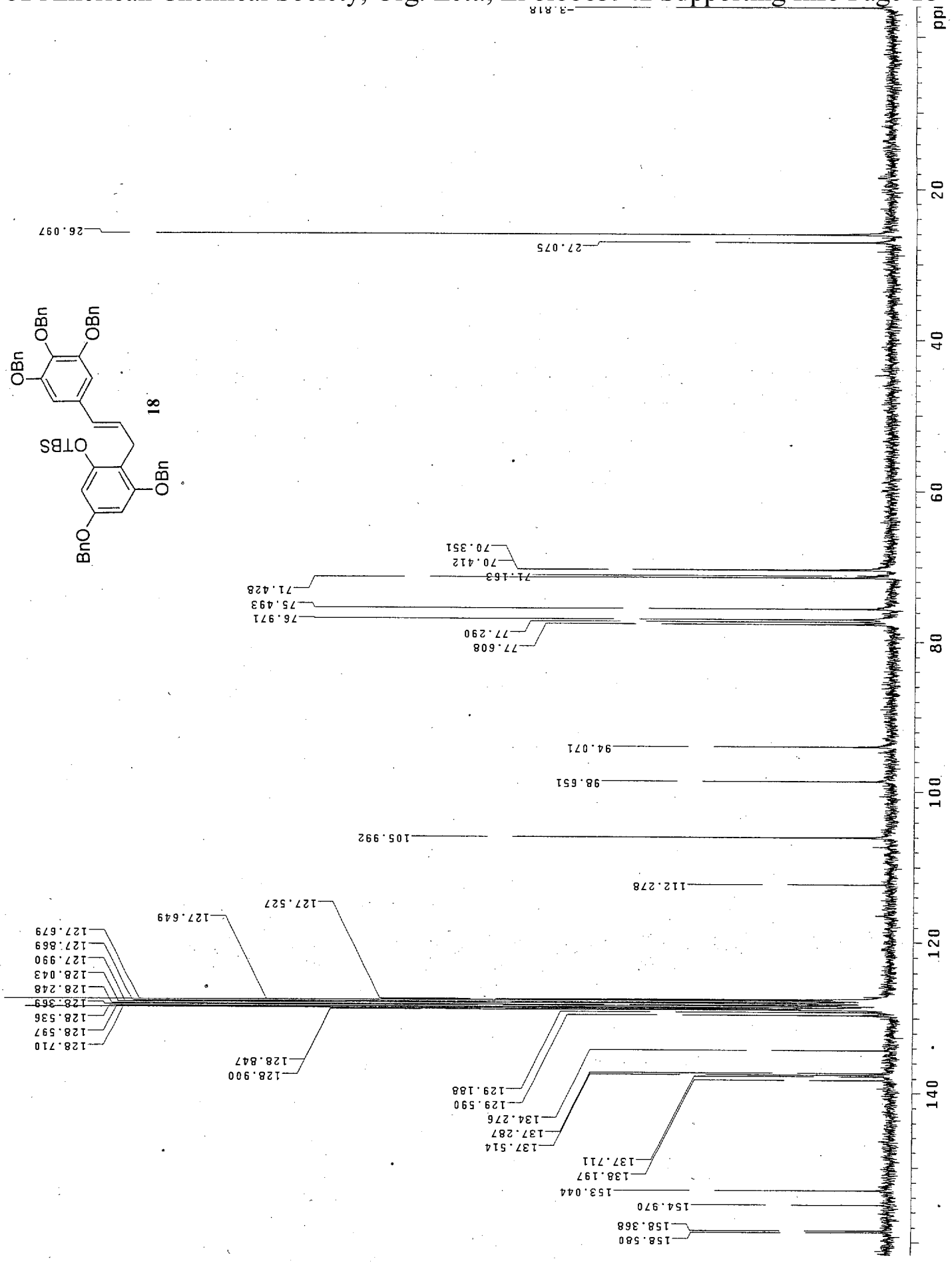

STANDARD 1H OBSERVE
expl stdlh
SAMPLE
date Aug 2 2000
solvent CDCl3
file exp
ACQUISITION
sfrq 400.139
tn H1
at 1.995
np 23936
sw 5998.8
fb 3400
bs 16
tpwr 57
pw 7.0
d1 1.000
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp 505.9
wp 2719.5
vs 200
sc 0
wc 250
hzmm 10.88
is 500.00
rfl 1007.5
rfp 0
th 4
ins 100.000
nm cdc ph
DEC. & VT
dfrq 400.139 H1
dn H1
dpwr 41
dof 0
dm nnn
dmm c
dmf 11200
PROCESSING
wtfile ft
proc not used
fn not used
werr
wexp
wbs
wnt
OH
OBn
BnO
BnO
6c
1.614
4.283
4.298
5.063
5.112
6.176
6.190
6.204
6.215
6.230
6.243
6.457
6.498
6.686
7.253
7.261
7.266
7.275
7.279
7.311
7.314
7.320
7.329
7.336
7.342
7.346
7.350
7.361
7.375
7.380
7.392
7.396
7.400
7.410
7.422
7.434
7.439
7.444
ppm
1
2
3
4
5
6
7
8




OBn
OBn
OBn
OTBS
OBn
BnO

18

STANDARD 1H OBSERVE
expl std1h
SAMPLE
date Mar 17 2000
solvent CDCl3
file exp
ACQUISITION
sfrq 400.139
tn H1
at 1.995
np 23936
sw 5998.8
fb 3400
bs 16
tpwr 57
pw 7.0
d1 1.000
tof 0
nt 16
ct 16
alock n
gain not used
FLAGS
il n
in n
dp y
DISPLAY
sp 32.8
wp 3066.2
vs 200
sc 0
wc 250
hzmm 12.26
is 500.00
rfl 1007.5
rfp 0
th 10
ins 100.000
nm cdc ph
DEC. & VT
dfrq 400.139
dn H1
dpwr 41
dof 0
dm nnn
dmm c
dmf 11200
PROCESSING
wtfile
proc ft
fn not used
werr
wexp
wbs
wnt
pp
1
2
3
4
5
6
7


OBn
OBn
OBn
OTBS
OBn
BnO
18
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26.097
27.075
70.351
70.412
71.163
71.428
75.493
76.971
77.290
77.608
94.071
98.651
105.992
112.278
127.527
127.649
127.679
127.869
127.990
128.043
128.248
128.369
128.536
128.597
128.710
128.847
128.900
129.188
129.590
134.276
137.287
137.514
137.711
138.197
153.044
154.970
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158.580
ppm
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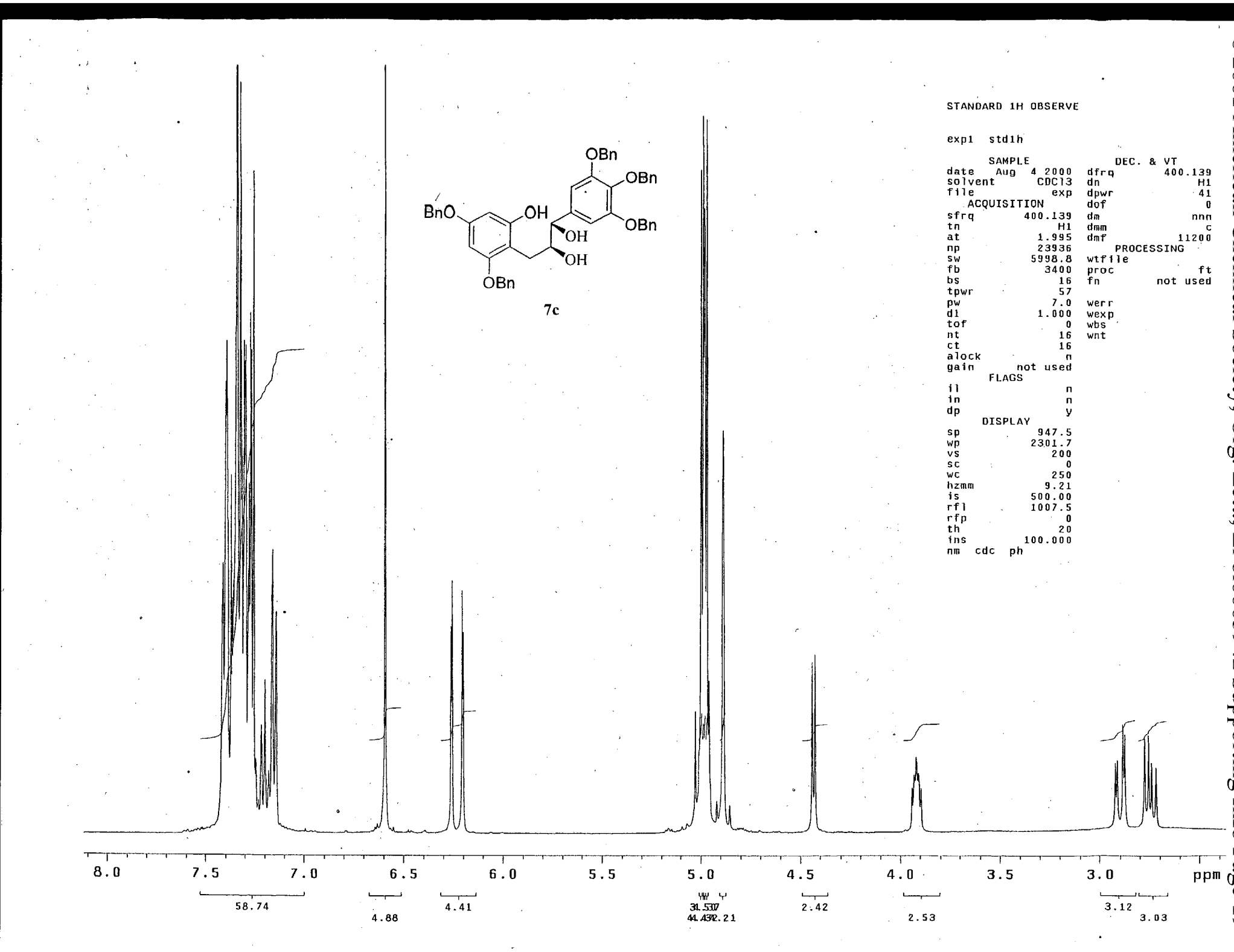
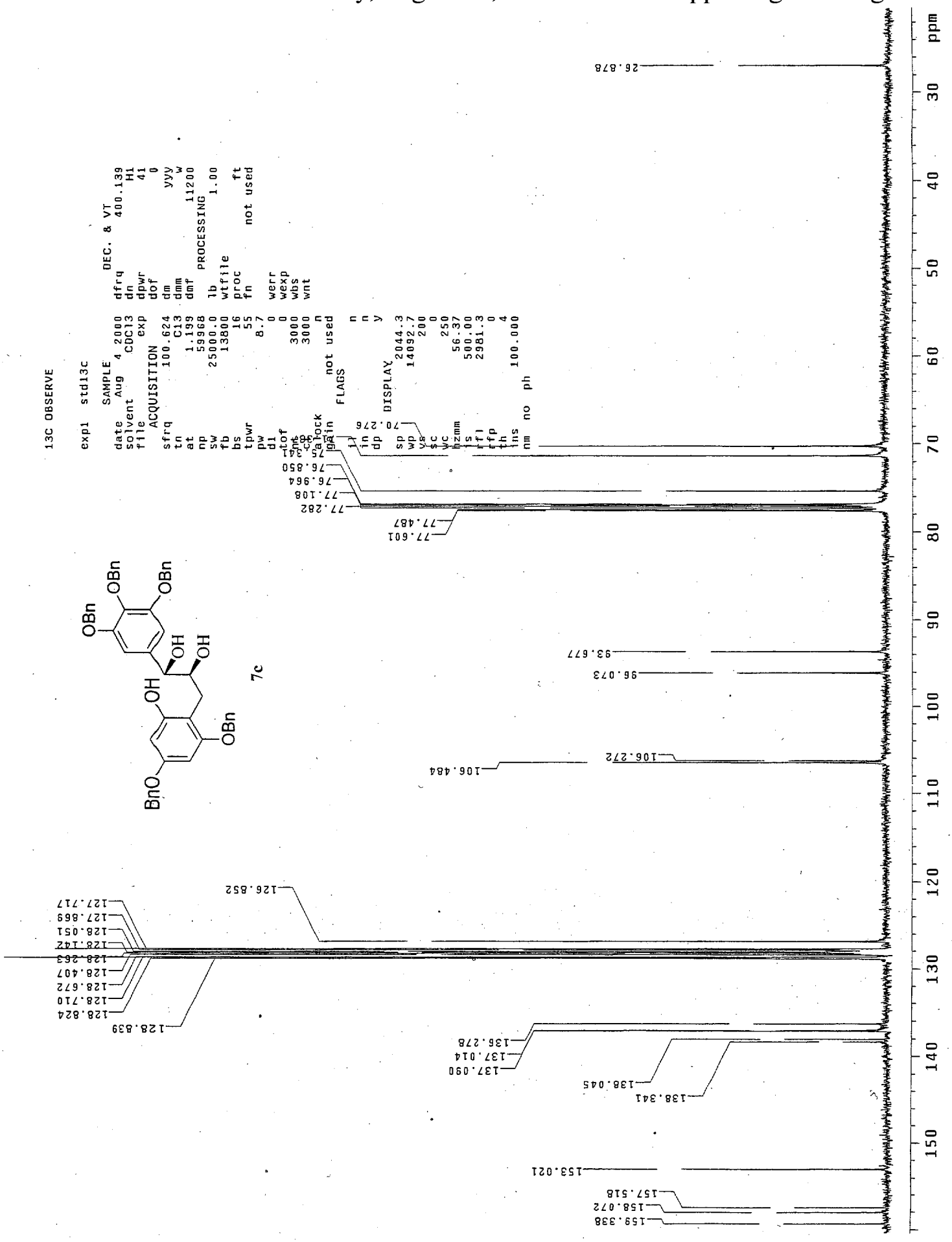
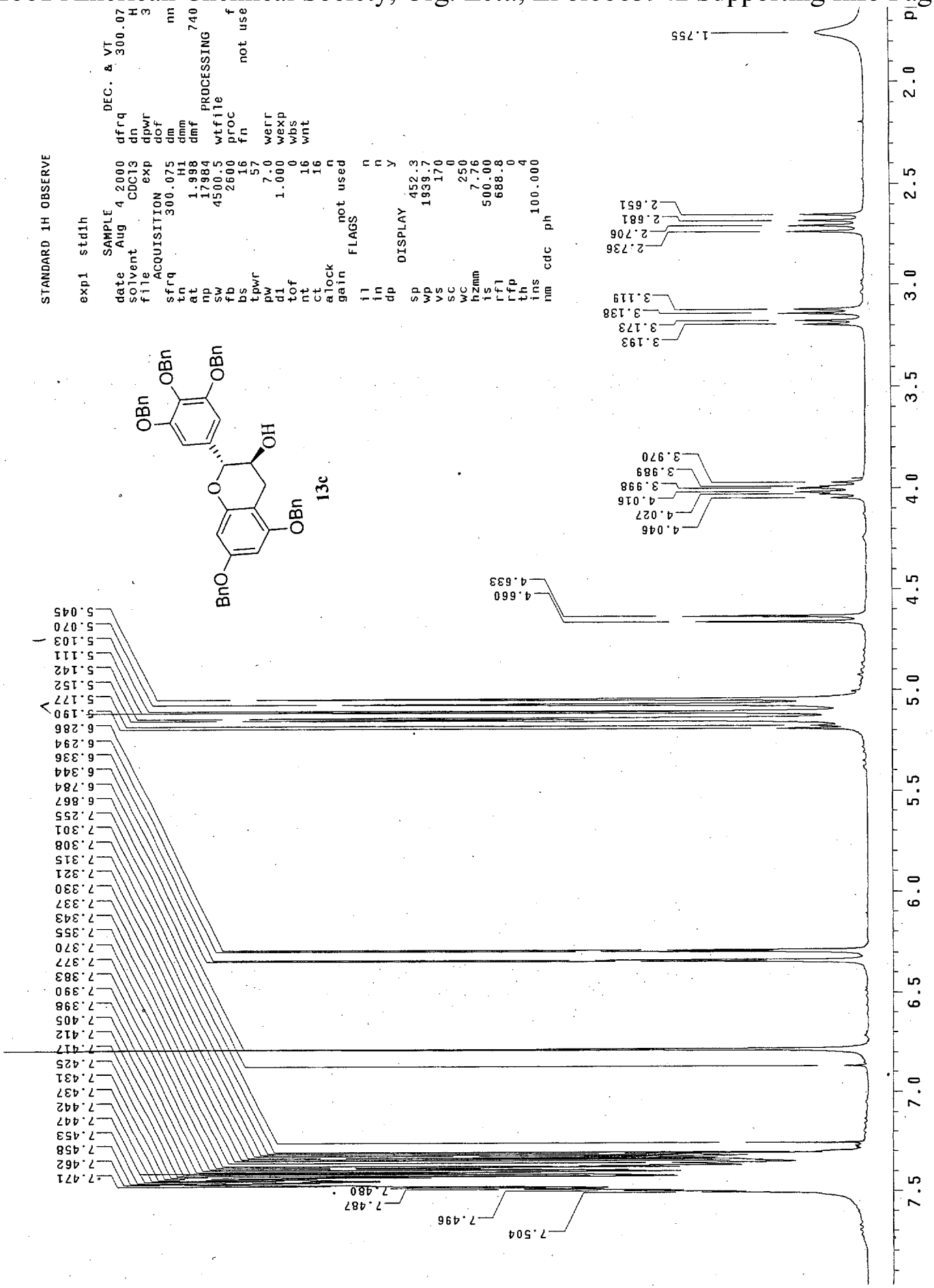
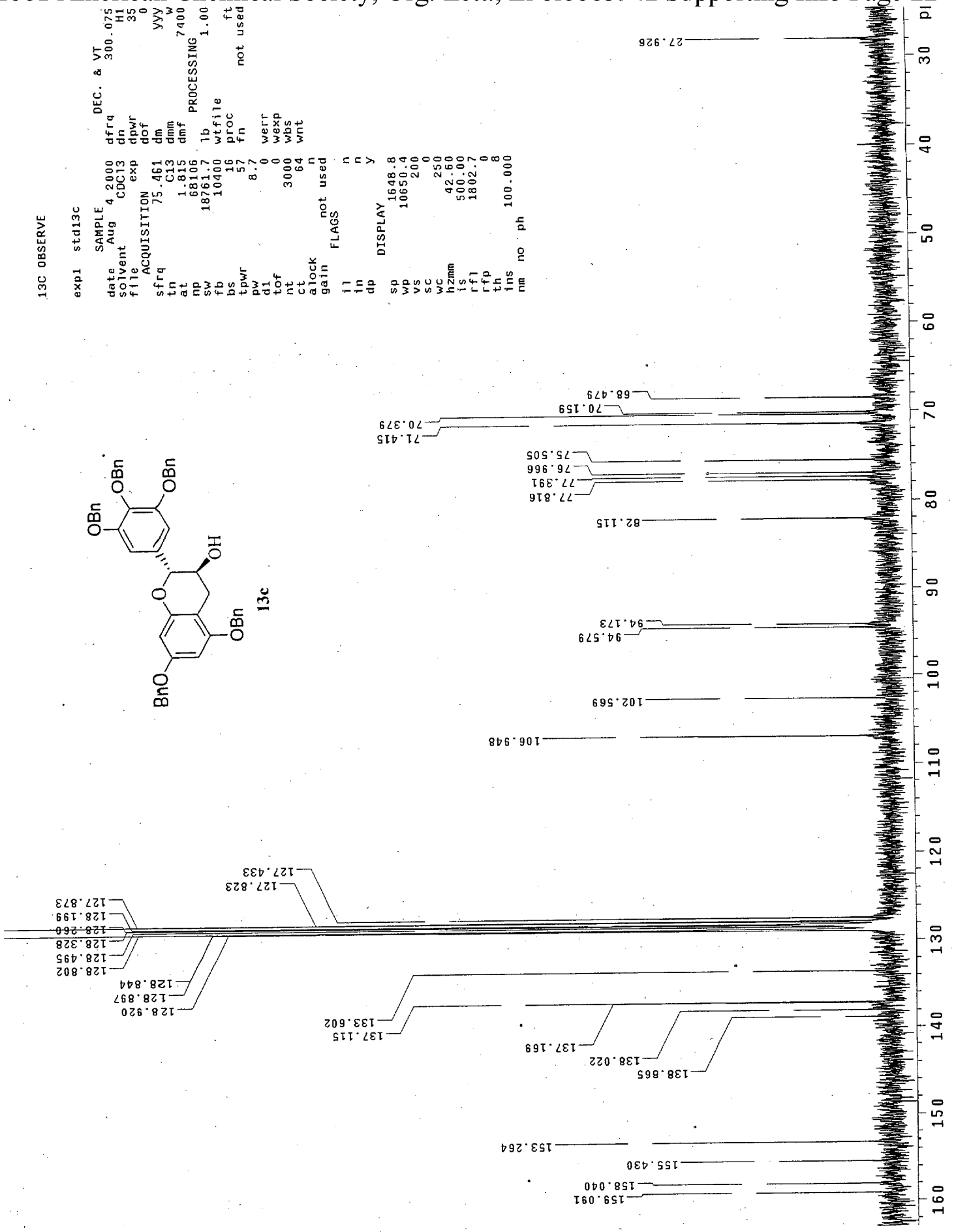
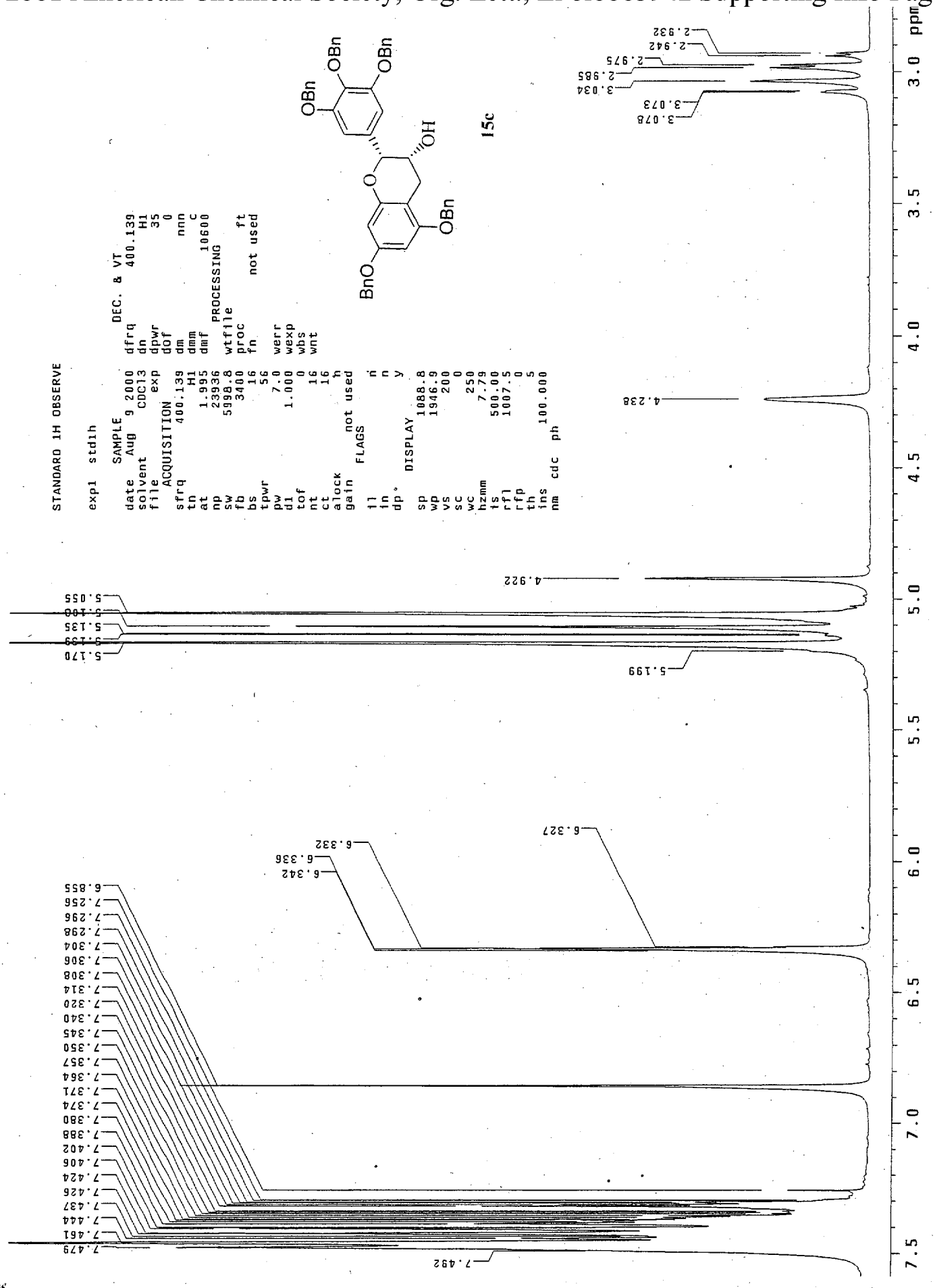
80
100
120
140

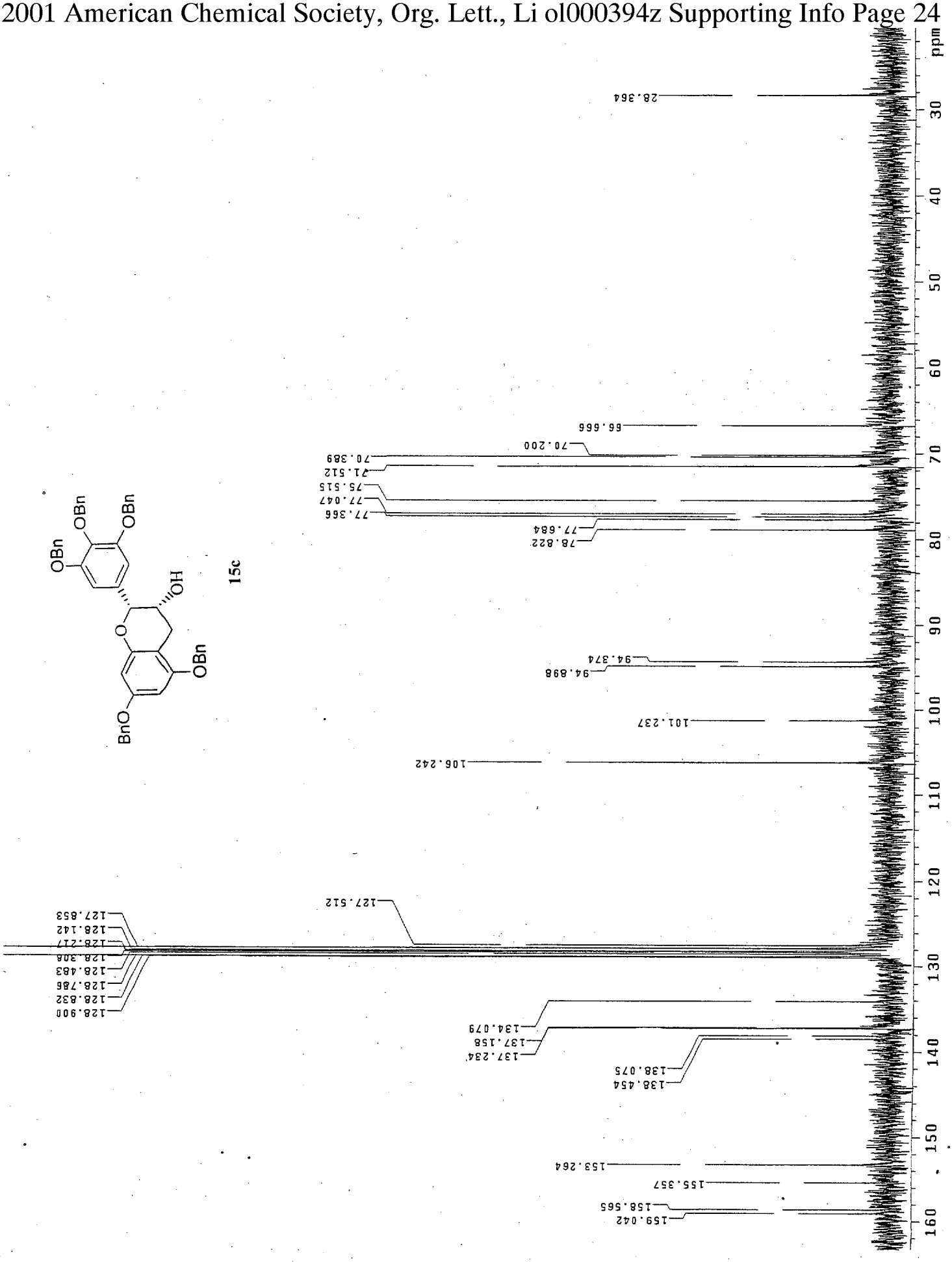



13C OBSERVE
expl std13c
SAMPLE
date Aug 4 2000
solvent CDCl3
file exp
ACQUISITION
sfrq 100.624
tn C13
at 1.199
np 59968
sw 25000.0
fb 13800
bs 16
tpwr 55
pw 8.7
d1 0
tof 0
nt 3000
ct 3000
alock n
gain not used
FLAGS
DISPLAY
DEC. & VT
dfrq 400.139
dn H1
dpwr 41
dof 0
dm yyy
dmm w
dmf 11200
PROCESSING
lb 1.00
wtfile ft
proc not used
fn
werr
wexp
wbs
wnt
ppm
7c
OBn
OH
BnO








ppm
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50
60
70
80
90
100
110
120
130
140
150
160
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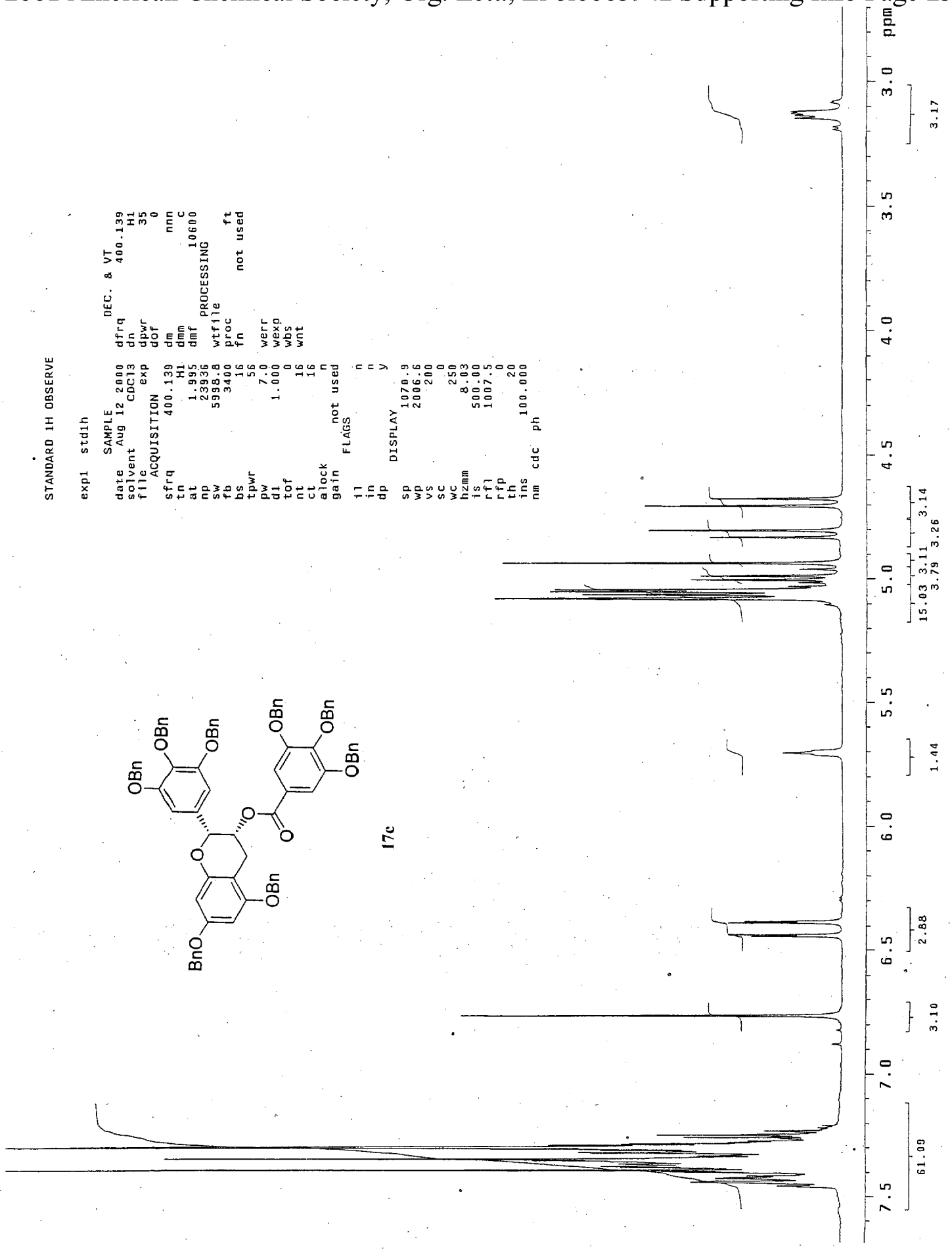
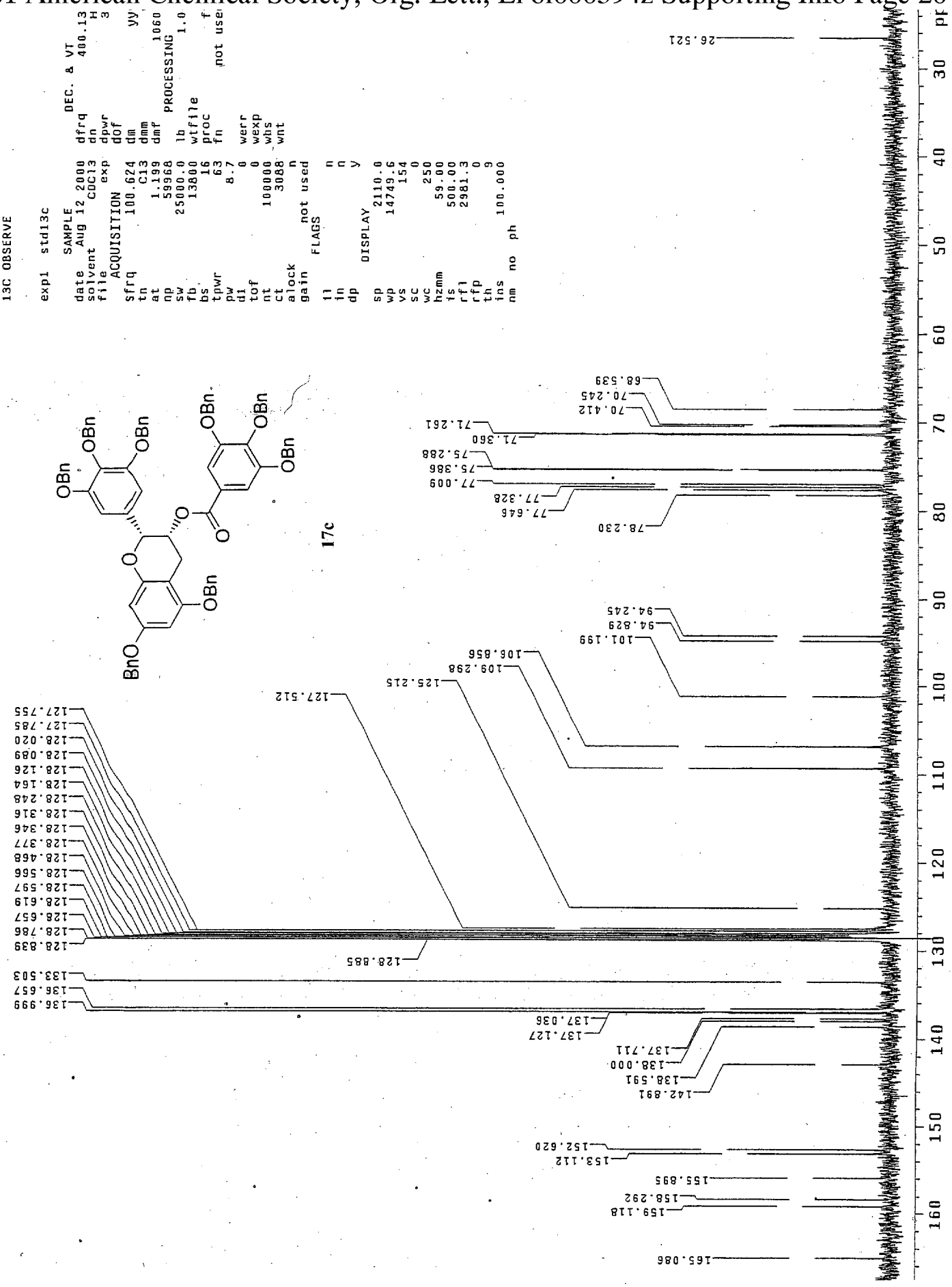
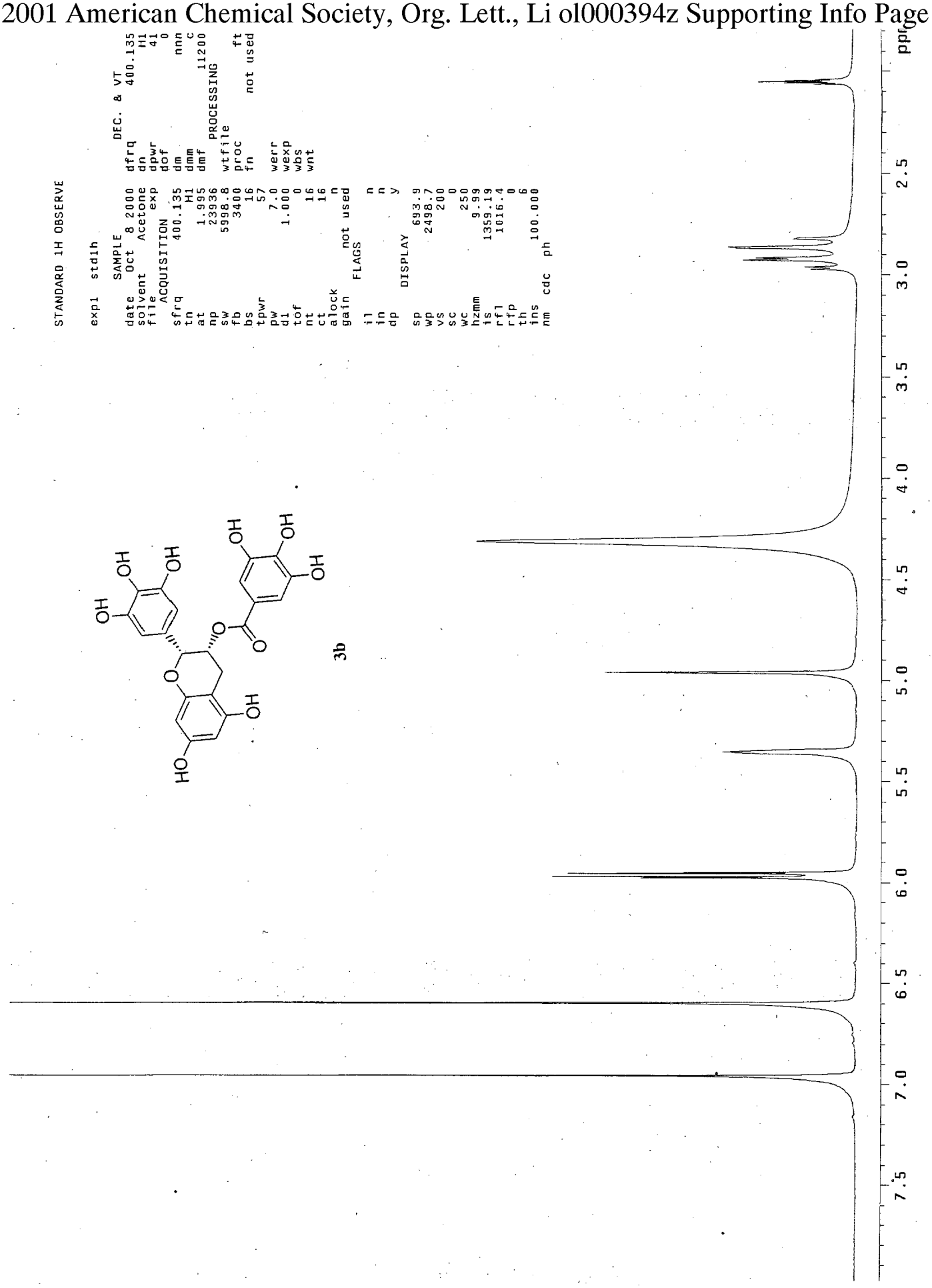
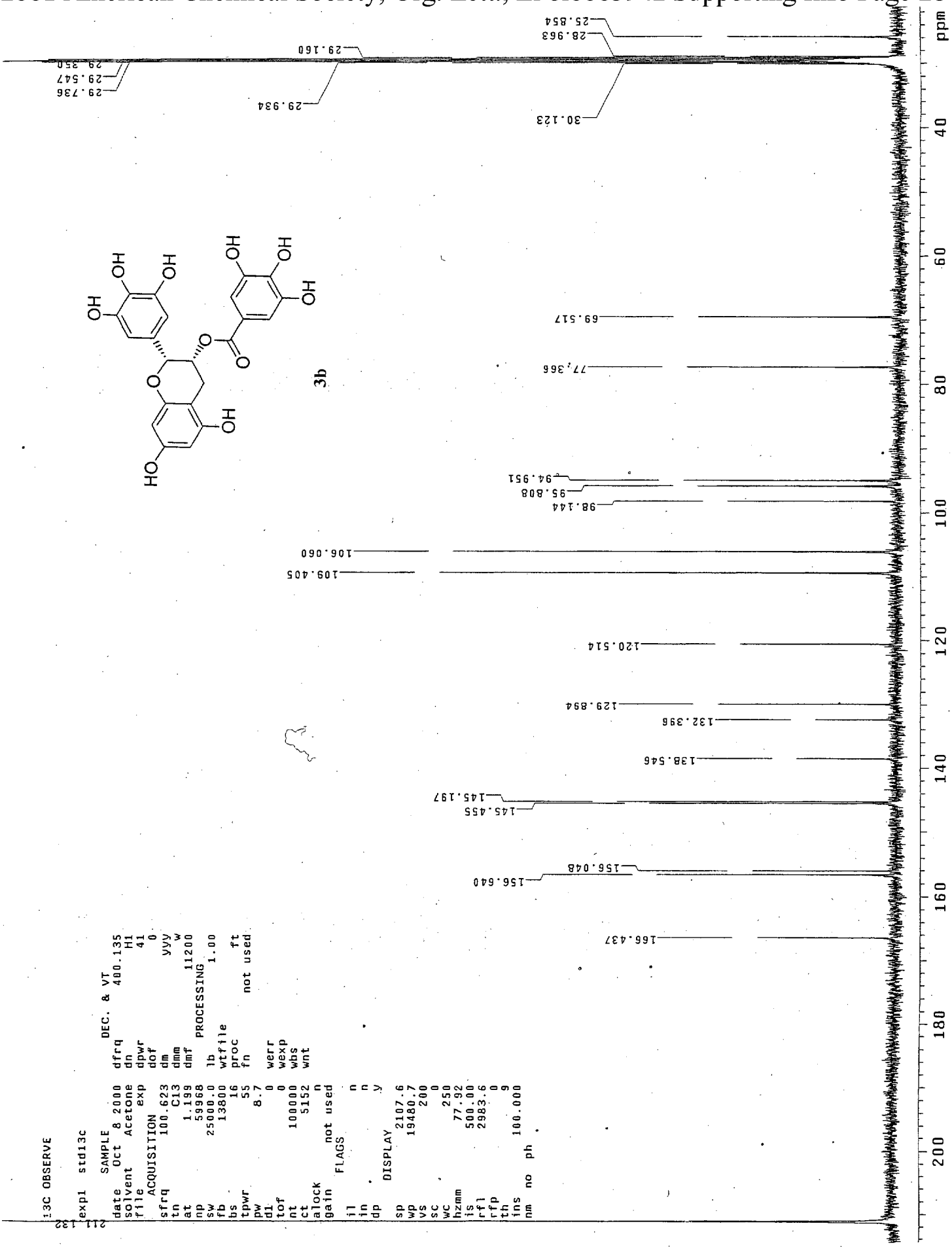
66.666
70.200
70.389
71.512
75.515
77.047
77.366
77.684
78.822
94.374
94.898
101.237
106.242
127.512
127.853
128.142
128.217
128.308
128.483
128.786
128.832
128.900
134.079
137.158
137.234
138.075
138.454
153.264
155.357
158.565
159.042
OBn
OBn
OBn
OH
O
OBn
BnO

15c










```
211.132
!3C OBSERVE
expl  std13c
                SAMPLE                DEC. & VT
date   Oct  8 2000   dfrq    400.135
solvent   Acetone    dn          H1
file        exp      dpwr        41
ACQUISITION          dof          0
sfrq    100.623      dm         yyy
tn          C13      dmm          w
at        1.199      dmf      11200
np        59968    PROCESSING
sw     25000.0      lb        1.00
fb       13800      wtfile       ft
bs          16      proc
tpwr        55      fn       not used
pw         8.7
d1           0      werr
tof          0      wexp
nt      100000      wbs
ct        5152      wnt
alock        n
gain     not used
               FLAGS
il                n
in                n
dp                y
               DISPLAY
sp         2107.6
wp        19480.7
vs            200
sc              0
wc            250
hzmm        77.92
is         500.00
rfl        2983.6
rfp             0
th              9
ins        100.000
nm   no  ph
```