

Supporting Information

Asymmetric Total Synthesis and Revision of the Absolute Configuration of 4-Keto-Clonostachydiol

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Table of Contents

1. Experimental procedures and data for the compounds

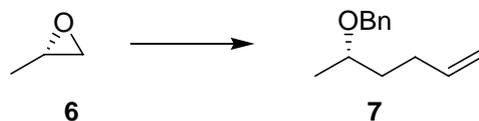
	<u>Page</u>
Benzyl ether 7	S3
Diol 8a	S3
MOM ether 9 and 9a	S4
Alcohol 10 and 10a	S5
Enoate 11 and 11a	S6
Enoate alcohol 5 and 5a	S7
Diol 13 and 13a	S7
Acid 4 and 4a	S8
Ester 14a	S8
Dihydroxy ester 15 and 15a	S9
Dihydroxy acid 3 and 3a	S10
Macrolide 16a	S11
4-Keto-Clonostachydiol 1a	S11
4- <i>epi</i> -Clonostachydiol 17	S12
MTPA Esters of 17	S12
MTPA Esters of 1a	S13
Compare the NMR of 4-Keto-Clonostachydiol.....	S14-S15
Compare the H NMR 4- <i>epi</i> -Clonostachydiol.....	S16

2. Spectra (¹H and ¹³C NMR) of compounds

	<u>Page</u>
Benzyl ether 7	S17-S18
Diol 8 and 8a	S19-S22
TBS ether.....	S23-S26
MOM ether 9 and 9a	S27-S30
Alcohol 10 and 10a	S31-S34
Enoate 11 and 11a	S35-S38
Enoate alcohol 5 and 5a	S39-S42
Diol 13 and 13a	S43-S46
Acid 4 and 4a	S47-S50
Enoate 14 and 14a	S51-S54
Dihydroxy enoate 15 and 15a	S55-S58
Dihydroxy acid 3 and 3a	S59-S62
Macrolide 16 and 16a	S63-S66
Macrolide ketone.....	S67-S70
4-Keto-Clonostachydiol 1 and 1a	S71-S79
4- <i>epi</i> -Clonostachydiol 17	S80-S81
MTPA Esters of 17	S82-S83
MTPA Esters of 1a	S84-S85

1. Experimental procedures and data for the compounds

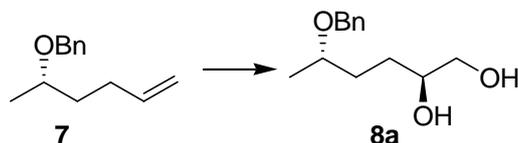
Synthesis of Benzyl ether **7**



A 100 mL three-necked flask containing magnesium powder (1.58 g, 66 mmol) and a stirring bar was dried in an oven at 100 °C for 2 h and cooled to room temperature under dry Ar stream. A portion of allyl bromide (5.5 mL, 60 mmol) in anhydrous THF (80 mL) was introduced over 1 h at 0 °C. After 2 h stir at room temperature, the mixture was cooled to -40 °C. CuI (1.14 g, 6 mmol) was added followed by additional 30 min stir. (*S*)-Propylene oxide (2.1 mL, 30 mmol) in dry THF (5 mL) was added dropwise over 10 min. The mixture was stirred at -40 °C for 1 h and warmed to room temperature over 2 h. The reaction was quenched by saturated aqueous NH₄Cl and extracted by ether, washed with H₂O and brine, dried over Na₂SO₄, and then ether evaporated below 10 °C to give the (*S*)-5-heptene-2-ol as colorless oil, which was used in the next step without purification.

Sodium hydride (60% dispersion in mineral oil, 2.43 g, 60.9 mmol) was washed with hexanes, dried under Ar, suspended in THF (20 mL) and cooled to 0 °C. (*S*)-5-Heptene-2-ol in THF (10 mL) was added dropwise via syringe into the flask. The reaction mixture was allowed to stir at 0 °C for 1 h. Benzyl bromide (3.6 mL, 30.5 mmol) was added dropwise and the mixture was heated at reflux for 6 h. The reaction was quenched with saturated NH₄Cl, extracted with EtOAc, and dried over Na₂SO₄. Concentration in vacuo and purification by flash column chromatography (hexanes/EtOAc 50:1) gave 4.85 g (85% yield) of benzyl ether **7**: $[\alpha]_D^{20} = +17$ (*c* 1.1, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.24-7.34 (m, 5 H), 5.78-5.85 (m, 1 H), 4.93-5.03 (m, 2 H), 4.57 (d, *J* = 11.6 Hz, 1 H), 4.45 (d, *J* = 11.6 Hz, 1 H), 3.53 (m, *J* = 6 Hz, 1 H), 2.11-2.20 (m, 2 H), 1.66-1.73 (m, 1 H), 1.48-1.57 (m, 1 H), 1.20 (d, *J* = 6 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 139.0, 138.7, 128.3, 127.4, 114.5, 74.2, 70.3, 35.8, 29.8, 19.6; IR (film): ν_{\max} 3378, 2829, 1453, 1065, 738, 698 cm⁻¹.

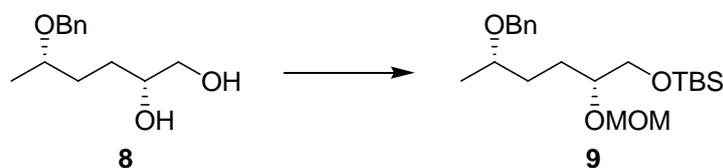
Synthesis of diol **8a**



8a (1.019g, 91%) was obtained from **7** (950 mg, 5 mmol) with AD-mix- α (7.0 g, 5 mmol) by the same operation as the synthesis of **8**: $[\alpha]_D^{20} = +24$ (*c* 0.9, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 7.26-7.34 (m, 5 H), 4.58 (d, *J* = 11.1 Hz, 1 H), 4.44 (d, *J* = 11.1 Hz, 1 H), 3.54-3.67 (m, 3 H), 3.38-3.43 (m, 1 H), 3.22 (br, 1 H), 2.70 (br, 1 H), 1.45-1.69 (m, 4 H), 1.21 (d, *J* = 6.6 Hz, 3 H); ¹³C NMR (75 MHz, CDCl₃): δ 138.5, 128.4, 127.7, 127.6, 74.9, 72.1, 70.4, 66.7, 32.6, 29.0, 19.3; IR (film): ν_{\max} 3386, 2926, 1092, 996, 910 cm⁻¹; HRMS (ESIMS) calcd for C₁₃H₂₁O₃ [M+H]⁺

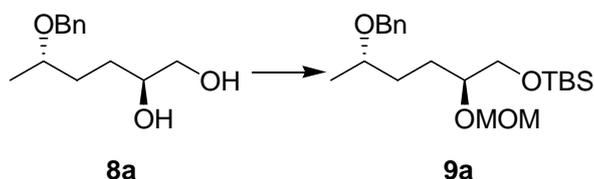
225.1485, found 225.1477.

Synthesis of MOM ether **9** and **9a**



To a magnetically stirred solution of diol **8** (1.25 g, 5.5 mmol) in dry THF (10 mL) were added imidazole (524 mg, 7.7 mmol) and *tert*-butyldimethylsilyl chloride (1.06 g, 7.2 mmol). After 24 h stir at room temperature, the mixture was diluted with Et₂O (20 mL) and H₂O (20 mL) and stirred until a clear separation of the phases. The mixture was then extracted with Et₂O. The combined organic layers were washed with brine, dried over Na₂SO₄, and condensed in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc 10:1) to give 1.77 g of the TBS ether (beta) (95%) as colorless oil: $[\alpha]_D^{20} = +13$ (*c* 1.1, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 7.26-7.35 (m, *J* = 12 Hz, 5 H), 4.58 (d, 1H), 4.45 (d, *J* = 12 Hz, 1 H), 3.54-3.61 (m, 3 H), 3.41 (dd, *J* = 8.1, 10.5 Hz, 1 H), 2.62 (s, 1 H), 1.51-1.60 (m, 4 H), 1.22 (d, *J* = 6.6 Hz, 3 H), 0.87 (s, 9 H), 0.04 (s, 6 H); ¹³C NMR (75 MHz, CDCl₃): δ 138.8, 128.3, 127.6, 127.4, 74.5, 71.6, 70.2, 67.1, 32.3, 28.5, 25.8, 19.4, 18.2, -5.4; IR (film): ν_{\max} 3439, 2930, 2858, 1254, 1093, 838, 778 cm⁻¹; HRMS (ESIMS) calcd for C₁₉H₃₅O₃Si [M+H]⁺ 339.2350, found 339.2358.

Methoxymethyl chloride (1.7 mL, 22 mmol) was added dropwise to a stirred solution of the TBS ether (1.84 g, 5.5 mmol) and *i*-Pr₂EtN (4.8 mL, 27 mmol) in CH₂Cl₂ (50 mL) at 0 °C followed by addition of Bu₄Ni (96 mg). After overnight stir at room temperature, the mixture was quenched with H₂O and extracted CH₂Cl₂. The combined organic layers were washed with brine, dried over MgSO₄, and condensed in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc 20:1) to give 1.85 g of MOM ether **9** (89%) as colorless oil: $[\alpha]_D^{20} = +12$ (*c* 0.95, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 7.26-7.34 (m, 5 H), 4.77 (d, *J* = 6.3 Hz, 1 H), 4.65 (d, *J* = 6.3 Hz, 1 H), 4.57 (d, *J* = 12 Hz, 1 H), 4.46 (d, *J* = 12 Hz, 1 H), 3.51-3.64 (m, 4 H), 3.37 (s, 3 H), 1.50-1.75 (m, 4 H), 1.21 (d, *J* = 6.6 Hz, 3 H), 0.89 (s, 9H), 0.03(s, 6H); ¹³C NMR (75 MHz, CDCl₃): δ 139.0, 128.3, 127.6, 127.3, 96.2, 78.1, 74.6, 70.2, 65.8, 55.4, 32.1, 27.4, 25.9, 19.5, 18.3, -5.4; IR (film): ν_{\max} 3423, 2928, 2857, 1462, 1102, 1039, 839, 776 cm⁻¹; HRMS (ESIMS) calcd for C₂₁H₃₉O₄Si [M+H]⁺ 383.2612, found 383.2622.



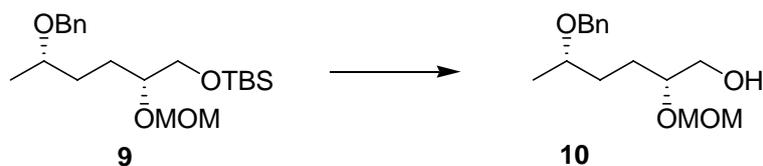
9a (926 mg, 86%) was obtained from **8a** (641 mg, 2.86 mmol) by the same operation as the synthesis of **9**.

TBS ester (alfa): $[\alpha]_D^{20} = +15$ (*c* 2.05, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 7.26-7.35 (m, 5H), 4.58 (d, *J* = 11.7, 1 H), 4.45 (d, *J* = 11.7 Hz, 1 H), 3.54-3.65 (m, 3 H), 3.39-3.45 (m, 1 H), 2.61 (s, 1 H), 1.145-1.71 (m, 4 H), 1.24(d, *J* = 6.6 Hz, 3 H), 0.87 (s, 9 H), 0.04 (s, 6 H); ¹³C NMR (75

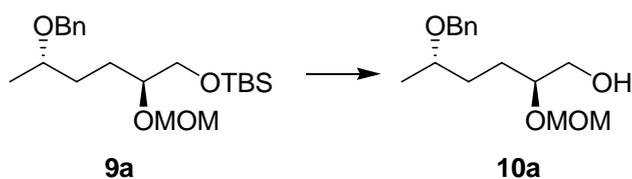
MHz, CDCl₃): δ 138.8, 128.3, 127.6, 127.4, 74.9, 71.9, 70.3, 67.2, 32.7, 28.9, 25.8, 19.5, 18.2, -5.4; IR (film): ν_{\max} 3437, 2930, 2858, 1254, 1093, 838, 779 cm⁻¹; HRMS (ESIMS) calcd for C₁₉H₃₅O₃Si [M+H]⁺ 339.2350, found 339.2345.

9a: $[\alpha]_{\text{D}}^{20} = -4$ (c 0.9, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 7.25-7.36 (m, 5H), 4.77 (d, *J* = 5.1 Hz, 1H), 4.65 (d, *J* = 5.1 Hz, 1H), 4.57 (d, *J* = 11.7 Hz, 1H), 4.46 (d, *J* = 11.7 Hz, 1H), 3.49-3.64 (m, 4H), 3.39 (s, 3H), 1.46-1.76 (m, 4H), 1.24 (d, *J* = 7.2 Hz, 3H), 0.89 (s, 9H), 0.05 (s, 6H); ¹³C NMR (75 MHz, CDCl₃): δ 139.0, 128.3, 127.6, 127.4, 96.3, 78.4, 75.0, 70.3, 65.8, 55.5, 32.4, 27.8, 25.9, 19.6, 18.3, -5.5; IR (film): ν_{\max} 3396, 2930, 2858, 1464, 1101, 1039, 839, 776 cm⁻¹; HRMS (ESIMS) calcd for C₂₁H₃₉O₄Si [M+H]⁺ 383.2612, found 383.2610.

Synthesis of Alcohol **10** and **10a**

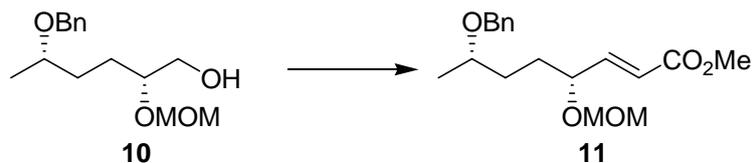


TBAF (1 M in THF, 14 mL, 14 mmol) was added to a stirred solution of the MOM ether **9** (1.695 g, 4.5 mmol) in THF (6 mL) at room temperature, and the mixture was stirred for 6 h at room temperature. The reaction mixture was then diluted with ether. The organic layer was washed with H₂O and brine, dried over MgSO₄ and condensed in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc 2:1) to give 1.068 g of **10** (89%) as colorless oil: $[\alpha]_{\text{D}}^{20} = -7$ (c 1.0, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 7.25-7.34 (m, 5 H), 4.72 (d, *J* = 6.9 Hz, 1 H), 4.66 (d, *J* = 6.9 Hz, 1 H), 4.57 (d, *J* = 11.7 Hz, 1 H), 4.43 (d, *J* = 11.7 Hz, 1 H), 3.46-3.61 (m, 4 H), 3.41 (s, 3 H), 3.15 (m, 1 H), 1.47-1.70 (m, 4 H), 1.20 (d, *J* = 6.3 Hz, 3 H); ¹³C NMR (75 MHz, CDCl₃): δ 138.8, 128.3, 127.6, 127.4, 96.9, 82.0, 74.3, 70.2, 65.5, 55.6, 32.2, 27.3, 19.4; IR (film): ν_{\max} 3441, 2932, 2887, 1452, 1036, 917, 739 cm⁻¹; HRMS (ESIMS) calcd for C₁₅H₂₅O₄ [M+H]⁺ 269.1747, found 269.1755.



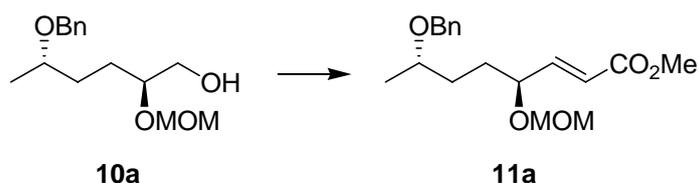
10a (460 mg, 87%) was obtained from **9a** (746 mg, 1.97 mmol) by the same operation as the synthesis of **10**. $[\alpha]_{\text{D}}^{20} = +45$ (c 0.9, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 7.26-7.34 (m, 5 H), 4.72 (d, *J* = 7.2 Hz, 1 H), 4.66 (d, *J* = 6.9 Hz, 1 H), 4.57 (d, *J* = 11.7 Hz, 1 H), 4.43 (d, *J* = 11.7 Hz, 1 H), 3.48-3.60 (m, 4 H), 3.42 (s, 3 H), 2.84 (s, 1 H), 1.53-1.64 (m, 4 H), 1.20 (d, *J* = 6 Hz, 3 H); ¹³C NMR (75 MHz, CDCl₃): δ 138.8, 128.3, 127.6, 127.4, 97.0, 82.4, 74.7, 70.3, 65.7, 55.6, 32.6, 27.7, 19.5; IR (film): ν_{\max} 3421, 2928, 2866, 1096, 1034, 917, 738 cm⁻¹; HRMS (ESIMS) calcd for C₁₅H₂₅O₄ [M+H]⁺ 269.1747, found 269.1739.

Synthesis of Enoate **11** and **11a**



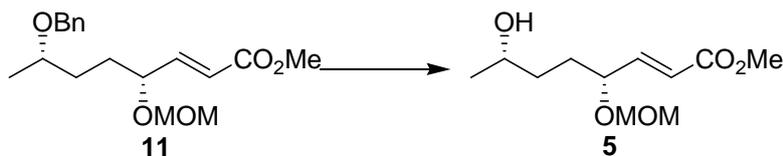
DMSO (1.2 mL, 12 mmol) in CH_2Cl_2 (2 mL) was added dropwise to a solution of oxalyl chloride (1.0 mL, 6 mmol) in dry CH_2Cl_2 (10 mL) at -78°C within 15 min. The mixture was stirred for 30 min and alcohol **10** (1.05 g, 4 mmol) in CH_2Cl_2 (20 mL) was added dropwise within 15 min. The reaction mixture was stirred for 30 min at -78°C and 30 min at -60°C , then Et_3N (3 mL, 16 mmol) was added dropwise and stirred for another 1 h. The reaction mixture was poured into saturated solution of NaHCO_3 (20 mL) and the organic layer was separated. The aqueous layer was extracted with CH_2Cl_2 and the combined organic layers were washed with brine, dried over Na_2SO_4 , concentrated to give the crude aldehyde.

Methyl diethylphosphonoacetate (1.26 g, 6 mmol) in dry THF (10 mL) was added dropwise to a suspension of NaH (191 mg, 60%) in THF (20 mL) at 0°C . After 30 min, the mixture became homogeneous followed by addition of the crude aldehyde in THF (10 mL). After 2 h stir, the mixture was quenched by saturated NH_4Cl and extracted with Et_2O . The combined organic layers were washed with brine, dried over MgSO_4 , and condensed in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/ EtOAc 10:1) to give 1.02 g of enoate **11** (79%) as colorless oil: $[\alpha]_D^{20} = +47$ (c 1.2, CHCl_3); ^1H NMR (300 MHz, CDCl_3): δ 7.25-7.34 (m, 5 H), 6.81 (dd, $J = 15.9, 6.3$ Hz, 1 H), 5.96 (d, $J = 15.9$ Hz, 1 H), 4.55-4.63 (m, 3 H), 4.42 (d, $J = 11.7$ Hz, 1 H), 4.14-4.20 (m, 1 H), 3.74 (s, 3 H), 3.53 (m, 1 H), 3.37 (s, 3 H), 1.50-1.79 (m, 4 H), 1.20 (d, $J = 5.7$ Hz, 3 H); ^{13}C NMR (75 MHz, CDCl_3): δ 166.6, 148.0, 138.8, 128.3, 127.6, 127.4, 121.5, 94.6, 75.0, 74.2, 70.3, 55.6, 51.6, 31.9, 30.5, 19.5; IR (film): ν_{max} 3401, 2924, 1723, 1272, 1152, 1035, 738 cm^{-1} ; HRMS (ESIMS) calcd for $\text{C}_{18}\text{H}_{26}\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 345.1672, found 345.1674.

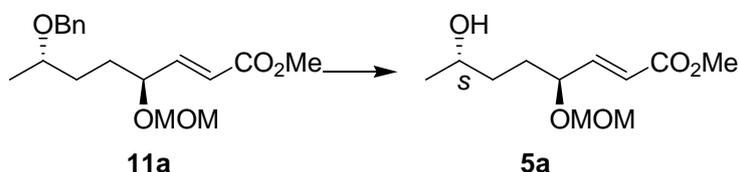


11a (918 mg, 81%) was obtained from **10a** (943 mg, 3.5 mmol) by the same operation as the synthesis of **11**: $[\alpha]_D^{20} = -20$ (c 0.93, CHCl_3); ^1H NMR (400 MHz, CDCl_3): δ 7.26-7.34 (m, 5 H), 6.82 (dd, $J = 15.6, 6.4$ Hz, 1 H), 5.98 (dd, $J = 15.6, 0.8$ Hz, 1 H), 4.56-4.64 (m, 3 H), 4.43 (d, $J = 11.6$ Hz, 1 H), 4.17-4.21 (m, 1 H), 3.75 (s, 3 H), 3.53 (m, 1 H), 3.37 (s, 3 H), 1.58-1.79 (m, 4 H), 1.20 (d, $J = 6$ Hz, 3 H); ^{13}C NMR (100 MHz, CDCl_3): δ 166.6, 148.1, 138.8, 128.3, 127.6, 127.5, 121.5, 94.6, 75.3, 74.6, 70.4, 55.7, 51.6, 32.2, 30.8, 19.6; IR (film): ν_{max} 3431, 2950, 1723, 1275, 1035, 985, 739 cm^{-1} ; HRMS (ESIMS) calcd for $\text{C}_{18}\text{H}_{26}\text{O}_5\text{Na}$ $[\text{M}+\text{Na}]^+$ 345.1672, found 345.1669.

Synthesis of Enoate Alcohol **5** and **5a**

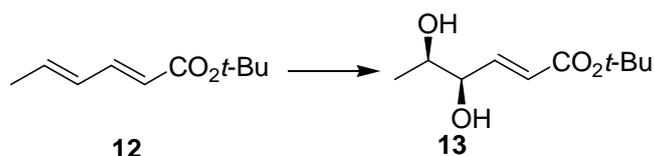


To a solution of enoate **11** (1.01 g, 3 mmol) in CH₂Cl₂/H₂O (40 mL; 19:1), DDQ (2.73 g, 12 mmol) as added and the mixture was refluxed for 3 h. The reaction was then quenched with saturated NaHCO₃ solution, filtered and washed with CH₂Cl₂ (20 mL). The filtrate was washed with H₂O, brine, dried over MgSO₄, and condensed in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc, 2:1) to give 646 mg of **5** (84%) as syrup: $[\alpha]_D^{20} = +61$ (*c* 1.03, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 6.82 (dd, *J* = 15.6 Hz, 6.4 Hz, 1 H), 6.00 (dd, *J* = 16, 1.2 Hz, 1 H) 4.61 (dd, *J* = 16.8, 6.9 Hz, 2 H), 4.24 (m, 1 H), 3.84 (m, 1 H), 3.74 (s, 3 H), 3.38 (s, 3 H), 1.50-1.79 (m, 4 H), 1.19 (d, *J* = 6 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 166.6, 147.8, 121.6, 94.6, 75.0, 67.6, 55.7, 51.6, 34.4, 30.9, 23.6; IR (film): ν_{\max} 3427, 2954, 1722, 1275, 1034 cm⁻¹; HRMS (ESIMS) calcd for C₁₁H₂₀O₅Na [M+Na]⁺ 255.1203, found 255.1210.

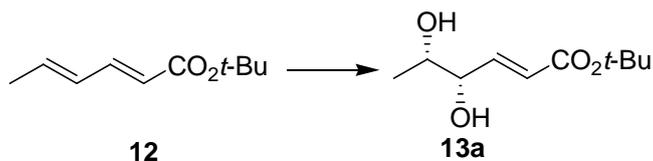


5a (557 mg, 86%) was obtained from **11a** (900 mg, 2.8 mmol) by the same operation as the synthesis of **5**: $[\alpha]_D^{20} = -44$ (*c* 0.95, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 6.78 (dd, *J* = 15.6 Hz, 6.3 Hz, 1 H), 5.95 (d, *J* = 15.6 Hz, 1 H) 4.57 (dd, *J* = 7.2, 12.9 Hz, 1 H), 4.20 (m, 1 H), 3.75 (m, 1 H), 3.70 (s, 3 H), 3.34 (s, 3 H), 2.07 (s, 1 H), 1.42-1.76 (m, 4 H), 1.15 (d, *J* = 6.3 Hz, 3 H); ¹³C NMR (75 MHz, CDCl₃): δ 166.5, 147.8, 121.5, 94.6, 75.1, 67.7, 55.6, 51.6, 34.4, 31.0, 23.5; IR (film): ν_{\max} 3427, 2953, 1723, 1275, 1035 cm⁻¹; HRMS (ESIMS) calcd for C₁₁H₂₀O₅Na [M+Na]⁺ 255.1203, found 255.1201.

Synthesis of Dihydroxy Enoate **13** and **13a**

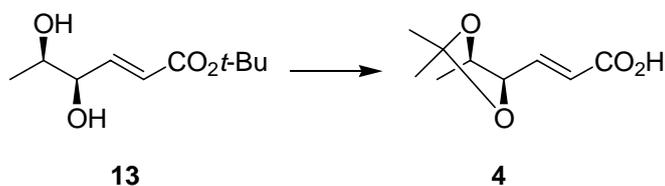


To a well-stirred solution of AD-mix-β (7.0 g, 5 mmol) in *t*-BuOH/H₂O (50 mL 1/1 v/v) was added methansulfonamide (475 mg, 5 mmol) at room temperature. The clear yellow solution was cooled to 0 °C and added unsaturated ester **12** (840 mg, 5 mmol). The solution was stirred vigorously at 0 °C. After stirring for 48 h, the reaction was quenched with 7.5 g of solid Na₂SO₃, warmed to room temperature and stirred for additional 50 min. The mixture was extracted with EtOAc, dried over MgSO₄, and condensed in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc 2:1) to give 616 mg of dihydroxy enoate **13** (61%) as colorless oil: 89% ee; $[\alpha]_D^{20} = +11$ (*c* 0.9, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 6.79 (dd, *J* = 15.9 Hz, 5.4 Hz, 1 H), 6.03 (d, *J* = 15.9 Hz, 1 H), 4.00 (m, 1 H), 3.70 (m, 1 H), 2.88 (s, 2 H), 1.47 (s, 9 H), 1.21 (d, *J* = 6.6 Hz, 3 H); ¹³C NMR (75 MHz, CDCl₃): δ 165.8, 145.5, 124.1, 80.8, 75.6, 70.2, 28.0, 18.9; IR (film): ν_{\max} 3406, 2977, 1712, 1156, 983 cm⁻¹.

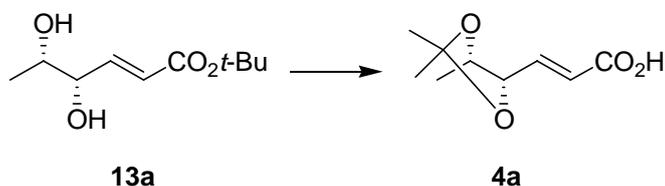


13a (596 mg, 59%) was obtained from **12** (840 mg, 5 mmol) by the same operation as the synthesis of **13**: 88% ee; $[\alpha]_D^{20} = +11$ (*c* 0.9, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 6.79 (dd, *J* = 15.6 Hz, 5.4 Hz, 1 H), 6.03 (dd, *J* = 15.9 Hz, 1.2 Hz, 1 H), 4.00 (m, 1 H), 3.69 (m, 1 H), 3.01 (s, 2 H), 1.46 (s, 9 H), 1.21 (d, *J* = 6.6 Hz, 3 H); ¹³C NMR (75 MHz, CDCl₃): δ 165.9, 145.5, 124.1, 80.8, 75.6, 70.2, 28.0, 19.0.

Synthesis of Acid **4** and **4a**:

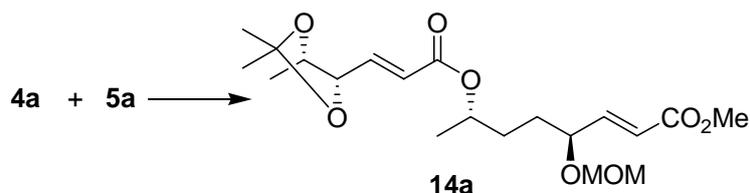


Dihydroxy enoate **13** (566 mg, 2.80 mmol) was stirred in a 1:1 mixture of TFA and CH₂Cl₂ (56 mL). After 1 h stir, the mixture was concentrated. The residue was treated with CHCl₃ (30 mL) and 2,2-dimethoxypropane (30 mL) at 0 °C. To the stirring solution was added *p*-TsOH (53 mg, 0.28 mmol) and H₂O (51 mg, 0.28 mmol). After 45 min, the reaction mixture was diluted with CH₂Cl₂ (100 mL) and washed with H₂O. The organic phase was collected, and the aqueous phase was re-extracted with CH₂Cl₂. The organic layers were combined, dried over MgSO₄, and condensed in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc, 2:1) to give 469 mg of acid **4** (90%) as yellow oil: $[\alpha]_D^{20} = -17$ (*c* 0.9, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 11.5 (br, 1 H), 6.98 (dd, *J* = 15.6, 6.4 Hz, 1 H), 6.15 (dd, *J* = 15.6, 1.2 Hz, 1 H), 4.4-4.13 (m, 1 H), 3.82-3.89 (m, 1 H), 1.43 (d, *J* = 14 Hz, 6 H), 1.33 (d, *J* = 6 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 171.2, 146.2, 121.8, 109.5, 81.4, 76.4, 27.2, 26.6, 16.7; IR (film): ν_{\max} 3471, 2958, 1707, 1378, 1244, 1096 cm⁻¹; HRMS (ESIMS) calcd for C₉H₁₄O₄Na [M+Na]⁺ 209.0784, found 209.0788.



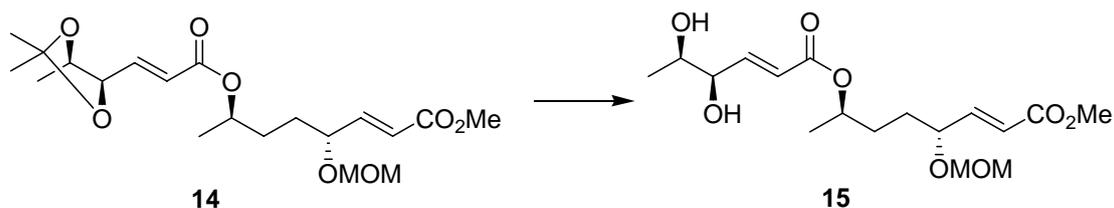
4a (62 mg, 92%) was obtained from **13a** (522 mg, 2.6 mmol) by the same operation as the synthesis of **4**: $[\alpha]_D^{20} = +17$ (*c* 1, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 6.99 (dd, *J* = 15.6, 5.2 Hz, 1 H), 6.16 (dd, *J* = 15.6, 1.2 Hz, 1 H), 4.11-4.15 (m, 1 H), 3.83-3.89 (m, 1 H), 1.44 (d, *J* = 14.8 Hz, 6 H), 1.35 (d, *J* = 6 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 171.0, 146.3, 121.7, 109.5, 81.5, 76.4, 27.3, 26.6, 16.7; HRMS (ESIMS) calcd for C₉H₁₄O₄Na [M+Na]⁺ 209.0784, found 209.0793.

Synthesis of Enoate **14a**

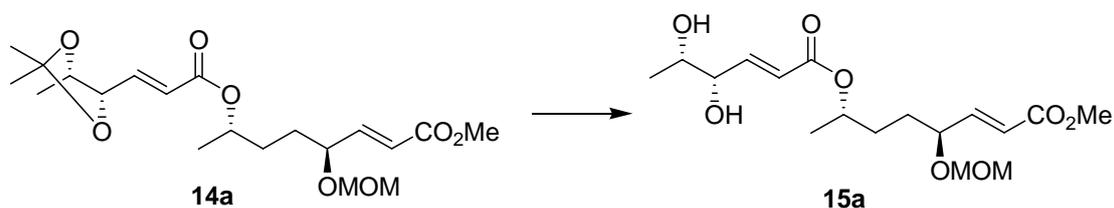


To a solution of acid **4a** (441 mg, 2.37 mmol) in dry THF (2 mL) was added anhydrous Et₃N (0.41 mL) and after stirring for 10 min, TCBC (648 mg, 2.66 mmol). This mixture was stirred for 3 h. The reaction was filtered and the filtrate was concentrated to dryness. To the concentrated filtrate was added dry toluene (8 mL), DMAP (309 mg, 2.5 mmol) and enoate alcohol **5a** (500 mg, 2.15 mmol) in toluene (4 mL). The mixture was heated at 90 °C for 2 h. The crude product was purified by silica gel chromatography (hexanes/EtOAc 10:1) to give 654 mg of enoate **14a** (76%) as colorless oil: $[\alpha]_D^{20} = -30$ (*c* 1.2, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 6.81 (m, 2 H), 6.09 (d, *J* = 15.3 Hz, 1 H), 5.98 (d, *J* = 15.9 Hz, 1 H), 4.97 (m, 1 H), 4.58 (dd, *J* = 6.3, 14.7 Hz, 2 H), 4.18 (m, 1 H), 4.05 (m, 1 H), 3.83 (m, 1 H), 3.73 (s, 3 H), 3.35 (s, 3 H), 1.64 (br, 4 H), 1.41 (d, *J* = 7.2 Hz, 6 H), 1.30 (d, *J* = 5.7 Hz, 3 H), 1.23 (d, *J* = 6 Hz, 3 H); ¹³C NMR (75 MHz, CDCl₃): δ 166.5, 165.4, 147.6, 143.3, 123.1, 121.8, 109.2, 94.6, 81.6, 76.4, 74.8, 70.9, 55.7, 51.6, 31.4, 30.7, 27.2, 26.6, 19.9, 16.6; HRMS (ESIMS) calcd for C₂₀H₃₆NO₈ [M+NH₄]⁺ 418.2435, found 418.2443

Synthesis of Dihydroxy Enoate **15** and **15a**:

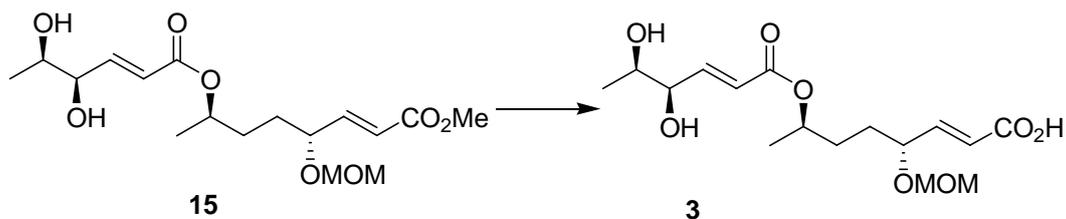


Enoate **14** (105 mg, 0.26 mmol) was dissolved in AcOH (4 mL) and H₂O (1 mL) and the mixture stirred at room temperature 2 days. Brine (20 mL) was added and the mixture was extracted with EtOAc. The combined organic layers were washed with NaHCO₃, brine, dried over Na₂SO₄, and condensed in vacuo. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc, 1:1) to give 68 mg of **15** (72%) as colorless oil: $[\alpha]_D^{20} = +31$ (*c* 0.9, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 6.91 (dd, *J* = 15.6, 4.8 Hz, 1 H), 6.80 (dd, *J* = 15.6, 6 Hz, 1 H), 6.12 (d, *J* = 15.2 Hz, 1 H), 5.99 (d, *J* = 15.6 Hz, 1 H), 4.98 (m, 1 H), 4.61 (dd, *J* = 17.2, 6.8 Hz, 2 H), 4.21 (m, 1 H), 4.06 (m, 1 H), 3.75 (s, 3 H), 3.72 (m, 1 H), 3.37 (s, 3 H), 2.48 (s, 2 H), 1.66 (m, 4 H), 1.25 (d, *J* = 6 Hz, 6 H). ¹³C NMR (100 MHz, CDCl₃): δ 166.6, 165.7, 147.7, 146.4, 122.8, 121.8, 94.7, 75.6, 74.9, 70.9, 70.2, 55.7, 51.7, 31.3, 30.6, 20.0, 19.1; IR (film): ν_{\max} 3420, 2926, 1775, 1721, 1275, 1096, 1041 cm⁻¹; HRMS (ESIMS) calcd for C₁₇H₂₉O₈ [M+H]⁺ 361.1857, found 361.1850.

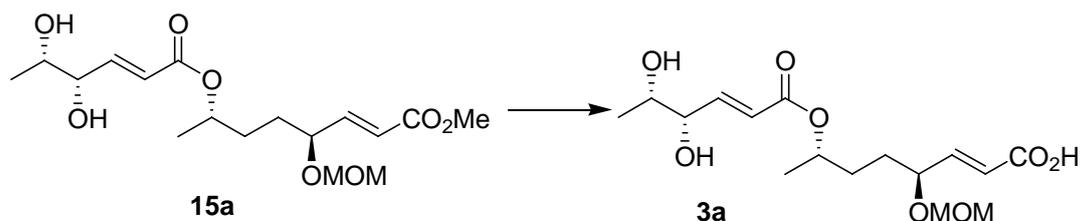


15a (60 mg, 74%) was obtained from **14a** (90 mg, 0.23 mmol) by the same operation as the synthesis of **15**: $[\alpha]_D^{20} = -50$ (*c* 1.1, CHCl₃); ¹H NMR (300 MHz, CDCl₃): δ 6.86 (dd, *J* = 15.3, 4.8 Hz, 1 H), 6.75 (dd, *J* = 15.6, 6.3 Hz, 1 H), 6.07 (d, *J* = 15.3 Hz, 1 H), 5.94 (d, *J* = 15.3 Hz, 1 H), 4.93 (s, 1 H), 4.56 (dd, *J* = 17.2, 6.8 Hz, 2 H), 4.16 (m, 1 H), 4.01 (m, 1 H), 3.70 (s, 3 H), 3.66 (m, 1 H), 3.32 (s, 3 H), 3.01 (s, 2 H), 1.61 (m, 4 H), 1.18-1.27 (m, 6 H). ¹³C NMR (75 MHz, CDCl₃): δ 166.6, 165.8, 147.6, 146.6, 122.5, 121.7, 94.6, 75.5, 74.8, 70.9, 70.1, 55.6, 51.6, 31.2, 30.5, 19.9, 19.0; HRMS (ESIMS) calcd for C₁₇H₂₉O₈ [M+H]⁺ 361.1857, found 361.1865.

Synthesis of Dihydroxy Acid **3** and **3a**:

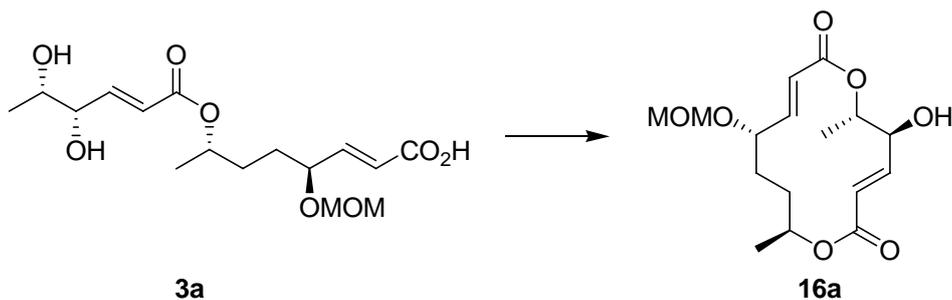


Dihydroxy enoate **15** (84 mg, 0.23 mmol) in THF (2 mL) and H₂O (2 mL) was added LiOH·H₂O (10 mg, 0.25 mmol). This solution was allowed to stir for 6 h. The mixture was diluted by EtOAc (10 mL) and was washed with H₂O (5 mL). The aqueous layer was neutralized (pH 4.0–6.0) with 1 M HCl and the aqueous layer was extracted with EtOAc. The combined organic layers were washed with brine, dried over Na₂SO₄, and concentrated to give crude dihydroxy acid **3** (56 mg, 69%): $[\alpha]_D^{20} = +54$ (*c* 0.3, MeOH); ¹H NMR (400 MHz, CDCl₃): δ 6.85-6.93 (m, 2 H), 6.12 (dd, *J* = 15.6, 1.2 Hz, 1 H), 5.98 (d, *J* = 16 Hz, 1 H), 4.98 (m, 1 H), 4.62 (dd, *J* = 14.6, 7.2 Hz, 2 H), 4.23 (m, 1 H), 4.08 (m, 1 H), 3.74 (m, 1 H), 3.368 (s, 3 H), 1.67 (m, 4 H), 1.23-1.26 (m, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 170.1, 166.0, 149.7, 146.6, 122.6, 121.5, 94.7, 75.6, 74.9, 71.1, 70.3, 55.7, 31.2, 30.5, 19.9, 19.0; IR (film): ν_{\max} 3420, 2935, 1712, 1276, 1037, 986 cm⁻¹; HRMS (ESIMS) calcd for C₁₆H₂₆O₈Na [M+Na]⁺ 369.1520, found 369.1515.



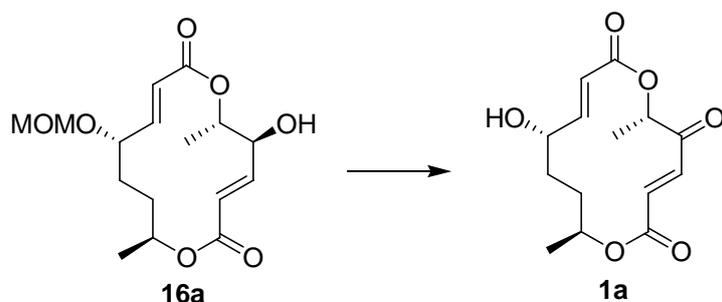
3a (290 mg, 71%) was obtained from **15a** (425 mg, 1.18 mmol) by the same operation as the synthesis of **3**: $[\alpha]_D^{20} = -50$ (*c* 1.4, MeOH); ¹H NMR (400 MHz, CDCl₃): δ 6.85-6.94 (m, 2 H), 6.12 (d, *J* = 15.6 Hz, 1 H), 5.99 (d, *J* = 16 Hz, 1 H), 4.98 (s, 1 H), 4.62 (m, 2 H), 4.24 (m, 1 H), 4.07 (m, 1 H), 3.74 (m, 1 H), 3.38 (s, 3 H), 1.67 (s, 4 H), 1.23-1.26 (m, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 170.2, 166.0, 149.8, 146.6, 122.7, 121.4, 94.7, 75.6, 74.9, 71.1, 70.3, 55.7, 31.2, 30.5, 19.9, 19.0; HRMS (ESIMS) calcd for C₁₆H₂₆O₈Na [M+Na]⁺ 369.1520, found 369.1529.

Synthesis of Macrolide **16a**:



16a (45mg, 77%) was obtained from **3a** (62 mg, 0.18 mmol) by the same operation as the synthesis of **16**: $[\alpha]_D^{20} = -15$ (*c* 0.6, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 6.79 (m, 2 H), 6.14 (dd, *J* = 15.6, 2 Hz, 1 H), 5.90 (dd, *J* = 16, 4.2 Hz, 1 H), 5.31 (m, 1 H), 5.18 (m, 1 H), 4.61 (m, 2 H), 4.48 (m, 1 H), 4.44 (m, 1 H), 3.36 (s, 1 H), 2.04-2.09 (m, 1 H), 1.60-1.77 (m, 3 H), 1.45 (d, *J* = 6.3 Hz, 3 H), 1.20 (d, *J* = 6.4 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 165.8, 165.7, 150.1, 146.7, 122.2, 121.5, 94.6, 74.0, 73.4, 71.1, 69.4, 55.5, 27.2, 26.1, 17.4, 16.0; HRMS (ESIMS) calcd for C₁₆H₂₈NO₇ [M+NH₄]⁺ 346.1860, found 346.1865.

Synthesis of 4-keto-Clonostachydiol **1a**:

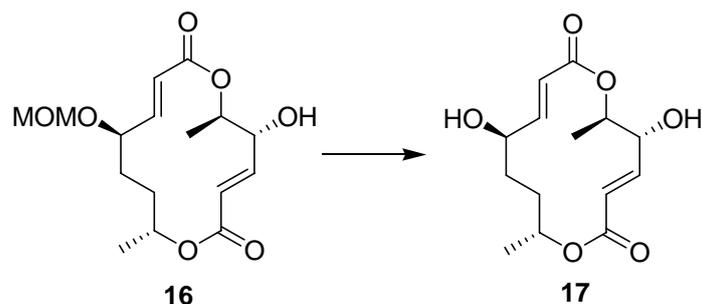


1a (4 mg, 68%) was obtained from **16a** (6 mg, 0.018 mmol) by the same operation as the synthesis of **1**.

Ketone: $[\alpha]_D^{20} = +31$ (*c* 0.3, CHCl₃); ¹H NMR (400 MHz, CDCl₃): δ 7.31 (d, *J* = 16 Hz, 1 H), 7.02 (dd, *J* = 16, 4 Hz, 1 H), 6.48 (d, *J* = 16 Hz, 1 H), 6.13 (dd, *J* = 16, 1.2 Hz, 1 H), 5.31 (q, *J* = 7.2 Hz, 1 H), 5.04 (m, 1 H), 4.64 (m, 2 H), 4.44 (m, 1 H), 3.38 (s, 3 H), 1.81-1.92 (m, 3 H), 1.60 (m, 1 H), 1.56 (d, *J* = 6.4 Hz, 3 H), 1.28 (d, *J* = 6.4 Hz, 3 H); ¹³C NMR (100 MHz, CDCl₃): δ 199.5, 165.7, 164.1, 150.1, 135.3, 131.2, 120.6, 94.7, 75.6, 74.0, 72.0, 55.7, 28.3, 28.1, 18.5, 16.4; HRMS (ESIMS) calcd for C₁₆H₂₈NO₇ [M+NH₄]⁺ 344.1704, found 344.1714.

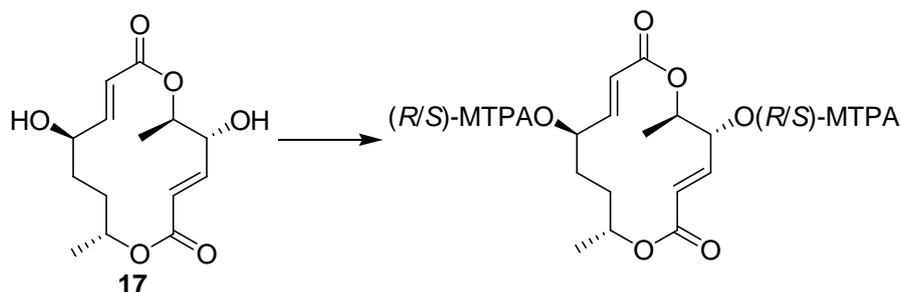
1a: $[\alpha]_D^{20} = +74$ (*c* 0.15, MeOH); ¹H NMR (400 MHz, DMSO-*d*₆): δ 7.37 (d, *J* = 16 Hz, 1 H), 7.20 (dd, *J* = 15.6, 3.2 Hz, 1 H), 6.39 (d, *J* = 16 Hz, 1 H), 5.94 (dd, *J* = 16, 2 Hz, 1 H), 5.20 (d, *J* = 4 Hz, 1 H), 5.16 (q, *J* = 7.2 Hz, 1 H), 4.92 (m, 1 H), 4.36 (m, 1 H), 1.89 (m, 1 H), 1.68 (m, 1 H), 1.56 (m, 1 H), 1.49 (d, *J* = 7.2 Hz, 4 H), 1.20 (d, *J* = 6.4 Hz, 3 H); ¹³C NMR (100 MHz, DMSO-*d*₆): δ 200.2, 165.8, 164.0, 154.5, 136.8, 129.9, 118.1, 75.4, 72.0, 68.7, 30.5, 27.7, 18.6, 16.0; HRMS (ESIMS) calcd for C₁₄H₂₂NO₆ [M+NH₄]⁺ 300.1442, found 300.1433.

Synthesis of 4-*epi*-Clonostachydiol **17**:



Trifluoroacetic acid (1.5 mL) was added to a solution of **16** (13 mg, 0.04 mmol) in CH₂Cl₂ (0.5 mL) at 0 °C. After stirring for 2 h at room temperature, the solvent was evaporated. The crude product was purified by column chromatography on silica gel (hexanes/EtOAc 1:1) to give 4-*epi*-clonostachydiol **17** (8 mg, 85%) as colorless oil: $[\alpha]_D^{20} = -31$ (*c* 1.0, CH₃OH); ¹H NMR (400 MHz, CD₃OD): δ 6.83 (dd, *J* = 15.6, 4 Hz, 1 H), 6.76 (dd, *J* = 15.6, 3.2 Hz, 1 H), 6.14 (dd, *J* = 15.6, 1.6 Hz, 1 H), 5.85 (dd, *J* = 15.6, 1.6 Hz, 1 H), 5.25 (dq, *J* = 6.4, 2 Hz, 1 H), 5.14 (m, 1 H), 4.57 (m, 1 H), 4.41 (m, 1 H), 1.97 (m, 1 H), 1.71 (m, 1 H), 1.64 (m, 1 H), 1.51 (m, 1 H), 1.38 (d, *J* = 6.8 Hz, 3 H), 1.21 (d, *J* = 6.8 Hz, 3 H); ¹³C NMR (100 MHz, CD₃OD): δ 168.0, 167.9, 153.6, 149.7, 123.1, 121.7, 74.0, 73.0, 71.1, 70.7, 29.4, 27.0, 17.8, 16.5; HRMS (ESIMS) calcd for C₁₄H₂₂NO₆ [M+NH₄]⁺ 302.1598, found 302.1607.

Synthesis of (*R*)- and (*S*)-MTPA Esters of **17**:

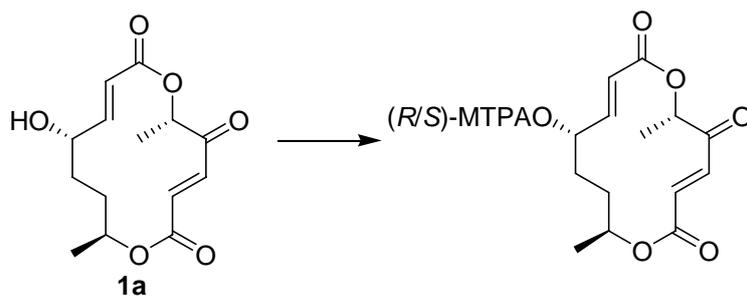


To a solution of **17** (1 mg, 0.0035 mmol) in CH₂Cl₂ (0.5 mL) was added (*R*)- or (*S*)-MTPACl (3.6 mg, 0.014 mmol) and DMAP (4 mg, 0.036 mmol). After stirring for 8 h, the crude product was purified by column chromatography on silica gel (hexanes/EtOAc 10:1) to give (*S*)- or (*R*)-MTPA ester of **17**, respectively.

(*S*)-MTPA Ester of **17**: 1 mg, 40% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.53-7.55 (m, 2 H), 7.36-7.49 (m, 8 H), 6.68 (dd, *J* = 15.6, 3.6 Hz, 1 H), 6.59 (dd, *J* = 16, 4.8 Hz, 1 H), 5.77 (m, 2 H), 5.75 (d, *J* = 15.6 Hz, 1 H), 5.70 (dd, *J* = 16.0, 1.6 Hz, 1 H), 5.38 (m, 1 H), 5.14 (m, 1 H), 3.55 (s, 3 H), 3.51 (s, 3 H), 2.18 (m, 1 H), 1.78 (m, 1 H), 1.66 (m, 1 H), 1.43 (m, 1 H), 1.25 (d, *J* = 6.4 Hz), 1.19 (d, *J* = 6.8 Hz).

(*R*)-MTPA Ester of **17**: 1.2 mg, 48% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.56 (m, 2 H), 7.48 (m, 2 H), 7.41-7.44 (m, 6 H), 6.67 (m, 1 H), 6.63 (m, 1 H), 5.79 (s, 1 H), 5.78 (d, *J* = 16 Hz, 1 H), 5.67 (m, 1 H), 5.58 (dd, *J* = 16.0, 1.6 Hz, 1 H), 5.42 (dq, *J* = 6.4, 2.4 Hz, 1 H), 5.07 (m, 1 H), 3.62 (s, 3 H), 3.52 (s, 3 H), 2.12 (m, 1 H), 1.67 (m, 1 H), 1.54 (m, 1 H), 1.34 (d, *J* = 6.8 Hz, 3 H), 1.25 (m, 1 H), 1.14 (d, *J* = 6.4 Hz, 3 H).

Synthesis of (*R*)- and (*S*)-MTPA Esters of **1a**:



To a solution of **1a** (1 mg, 0.0035 mmol) in CH₂Cl₂ (0.5 mL) was added (*R*)- or (*S*)-MTPACl (1.8 mg, 0.007 mmol) and DMAP (2 mg, 0.016 mmol). After stirring for 8 h, the crude product was purified by column chromatography on silica gel (hexanes/EtOAc 10:1) to give (*S*)- or (*R*)-MTPA ester of **1a**, respectively.

(*S*)-MTPA Ester of **17**: 1 mg, 57% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.43-7.55 (m, 5 H), 7.26 (d, *J* = 16 Hz, 1 H), 7.01 (dd, *J* = 16.0, 4.0 Hz, 1 H), 6.47 (d, *J* = 16 Hz, 1 H), 5.97 (dd, *J* = 16.0, 1.2 Hz, 1 H), 5.76 (m, 1 H), 5.32 (q, *J* = 7.2 Hz, 1 H), 5.00 (m, 1 H), 3.53 (s, 3 H), 1.99 (m, 2 H), 1.74 (m, 1 H), 1.56 (d, *J* = 7.2 Hz, 3 H), 1.45 (m, 1 H), 1.27 (d, *J* = 4.4 Hz, 3 H).

(*R*)-MTPA Ester of **17**: 1.1 mg, 63% yield. ¹H NMR (400 MHz, CDCl₃): δ 7.50-7.52 (m, 2 H), 7.41-7.44 (m, 3 H), 7.28 (d, *J* = 16.4 Hz, 1 H), 6.98 (dd, *J* = 16.0, 4.0 Hz, 1 H), 6.45 (d, *J* = 16.4 Hz, 1 H), 5.82 (dd, *J* = 16.0, 1.6 Hz, 1 H), 5.72 (m, 1 H), 5.30 (q, *J* = 7.2 Hz, 1 H), 5.02 (m, 1 H), 3.55 (s, 3 H), 2.08 (m, 1 H), 1.97 (m, 1 H), 1.85 (m, 1 H), 1.59 (m, 1 H), 1.55 (d, *J* = 7.2 Hz, 3 H), 1.28 (d, *J* = 6.4 Hz, 3 H).

Compare the ^{13}C NMR of 4-keto-colonstachydiol

Natural product		Synthetic product	
Literature 2	Literature 1	1	1a
15.90	16.0	15.9	16.0
18.54	18.7	18.6	18.6
27.58	27.7	27.6	27.7
30.41	30.5	30.4	30.5
68.55	68.7	68.6	68.7
71.87	72.1	72.0	72.0
75.21	75.4	75.4	75.4
117.93	118.2	118.1	118.1
129.64	130.0	129.9	129.9
136.59	136.9	136.8	136.8
154.17	154.5	154.5	154.5
163.71	164.1	164.0	164.0
165.51	165.9	165.8	165.8
199.71	200.3	200.3	200.2

Compare the ^1H NMR of 4-keto-colonstachydiol

Natural product						Synthetic product					
Literature 2			Literature 1			1			1a		
δ_{H}		$J(\text{Hz})$									
1.21	3	d,6	1.30	3	d,6.3	1.20	3	d,6.4	1.20	3	d,6.4
1.50	3	d,7.5	1.59	3	d,7.4	1.49	3	d,7.2	1.49	3	d,7.2
1.51	1	m	1.62	2	m	1.50	1	m	1.50	1	m
1.55	1	m				1.56	1	m	1.56	1	m
1.70	1	m	1.78	1	m	1.68	1	m	1.68	1	m
1.89	1	m	1.99	1	m	1.89	1	m	1.89	1	m
4.36	1	m	4.46	1	bd,8.6	4.36	1	m	4.36	1	m
4.92	1	m	5.02	1	q,6.3	4.92	1	m	4.92	1	m
5.16	1	q,7.5	5.26	1	q,7.4	5.16	1	q,7.2	5.16	1	q,7.2
5.94	1	dd,16 2	6.04	1	dd,15.7 1.4	5.93	1	dd,15.6 2.0	5.94	1	dd,15.6 2
6.38	1	d,16	6.49	1	d,16.2	6.39	1	d,16	6.39	1	d,16
7.19	1	dd,16 2	7.30	1	dd,15.7 3.0	7.20	1	dd,15.6 3.2	7.20	1	dd,15.6 3.2
7.37	1	d,16	7.47	1	d,16.2	7.37	1	d,16	7.37	1	d,16

Compare the ^1H NMR of 4-*epi*-clonostachydiol

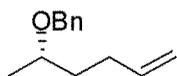
4- <i>epi</i> -Clonostachydiol Literature 1			Synthetic product 17		
J					
1.21	3	d,6.5	1.21	3	d,6.8
1.38	3	d,6.5	1.38	3	d,6.8
1.50	1	m	1.51	1	m
1.66	1	m	1.64	1	m
1.70	1	m	1.71	1	m
1.97	1	m	1.97	1	m
4.40	1	m	4.41	1	m
4.57	1	m	4.57	1	m
5.12	1	m	5.14	1	m
5.25	1	dq,6.5 2.1	5.25	1	dq,6.4 2
5.85	1	dd,15.8 1.9	5.85	1	dd,15.6 1.6
6.14	1	dd,15.7 2.0	6.14	1	dd,15.6 1.6
6.77	1	dd,15.7 3.0	6.76	1	dd,15.6 3.2
6.84	1	dd,15.8 4.3	6.83	1	dd,15.6 4

2. Spectra (¹H and ¹³C NMR) of compounds

7.369
7.364
7.347
7.340
7.332
7.312
7.279
7.272
7.268
7.264
7.240
5.847
5.821
5.804
5.795
5.787
5.779
5.029
5.025
4.986
4.982
4.957
4.955
4.932
4.929
4.928
4.583
4.561
4.554
4.459
4.429
3.556
3.541
3.525
3.510
2.197
2.181
2.178
2.174
2.169
2.165
2.162
2.158
2.145
2.143
2.126
2.123
2.106
1.732
1.730
1.714
1.710
1.698
1.696
1.691
1.681
1.675
1.673
1.658
1.571
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1.534
1.523
1.518
1.499
1.483
1.211
1.196

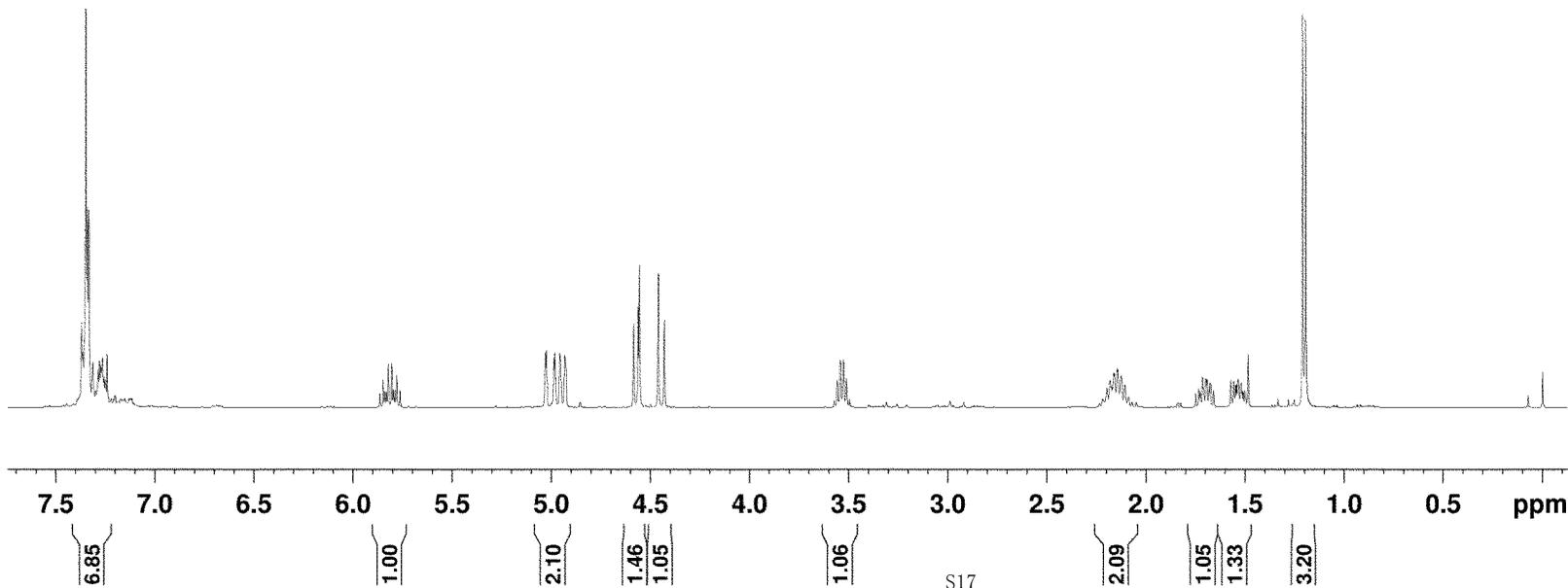
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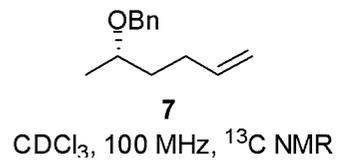
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EXPNO     10
PROCNO    1
Date_     20090113
Time      11.46
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         114
DW         60.800 usec
DE         6.50 usec
TE         289.6 K
D1         1.00000000 sec
TDO        1
  
```



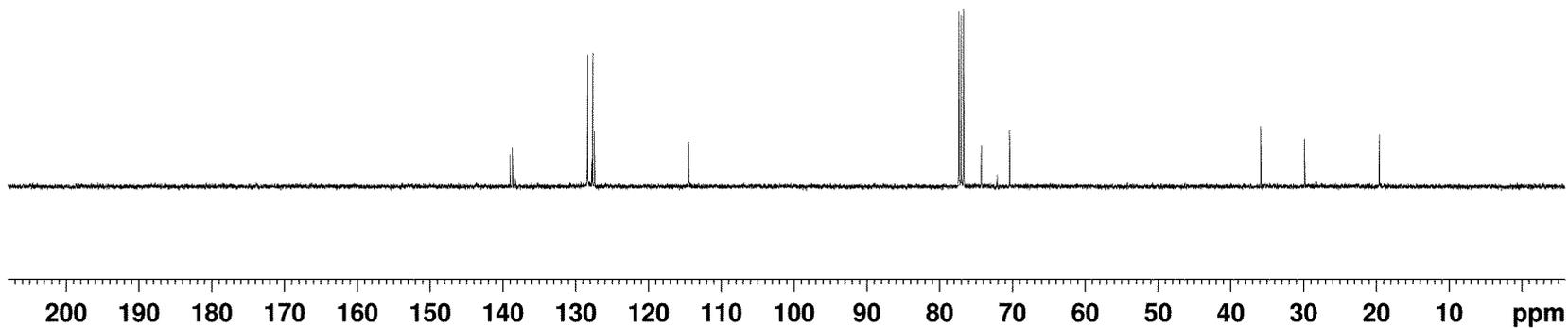
7

CDCl₃, 400 MHz, ¹H NMR





138.99
 138.65
 128.30
 127.63
 127.38
 114.46
 77.32
 77.00
 76.68
 74.23
 70.32
 35.84
 29.80
 19.55



```

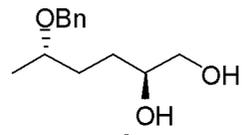
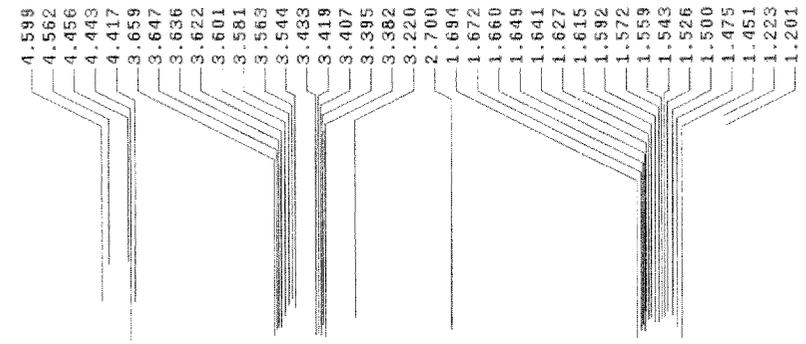
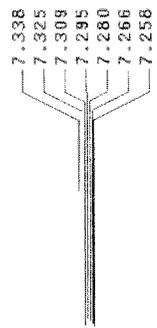
NAME      hanjj0113
EXPNO     11
PROCNO    1
Date_     20090113
Time      11.50
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        64
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        290.3 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

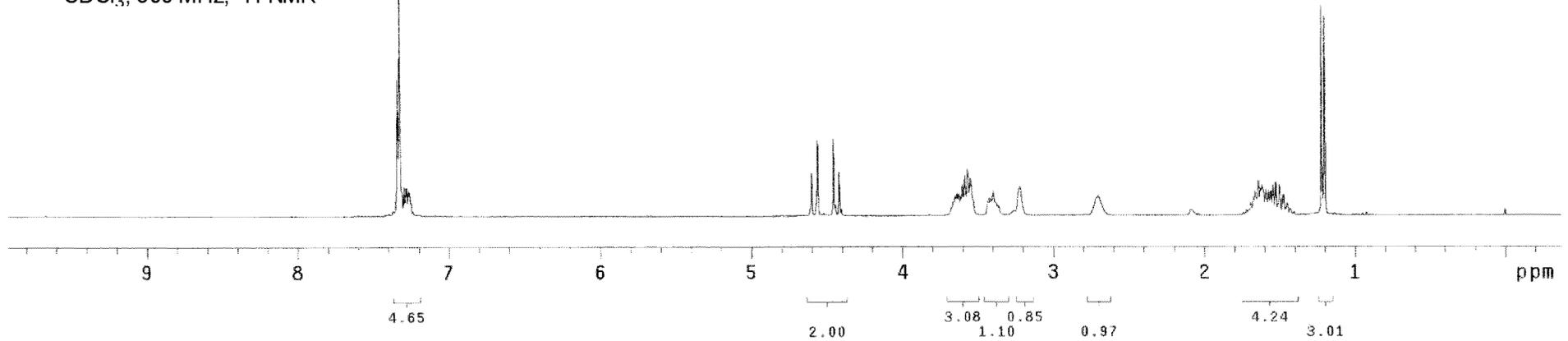
===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz
  
```

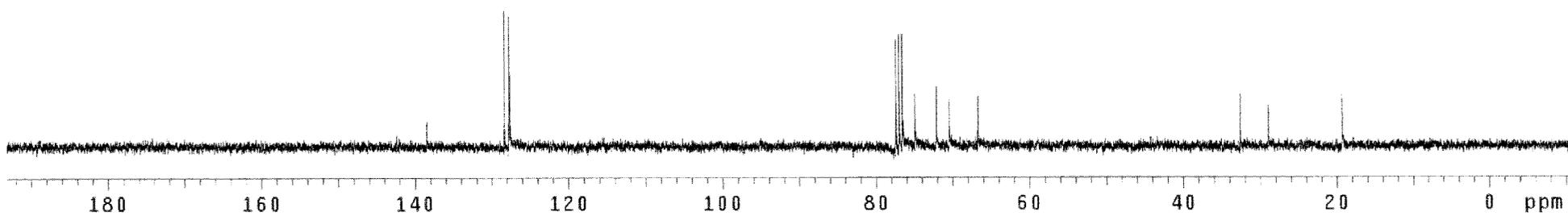
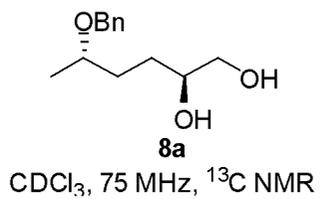
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===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       -2.00 dB
PL12      15.50 dB
PL13      15.50 dB
PL2W      18.19349861 W
PL12W     0.32353121 W
PL13W     0.32353121 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6127741 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```



CDCl₃, 300 MHz, ¹H NMR





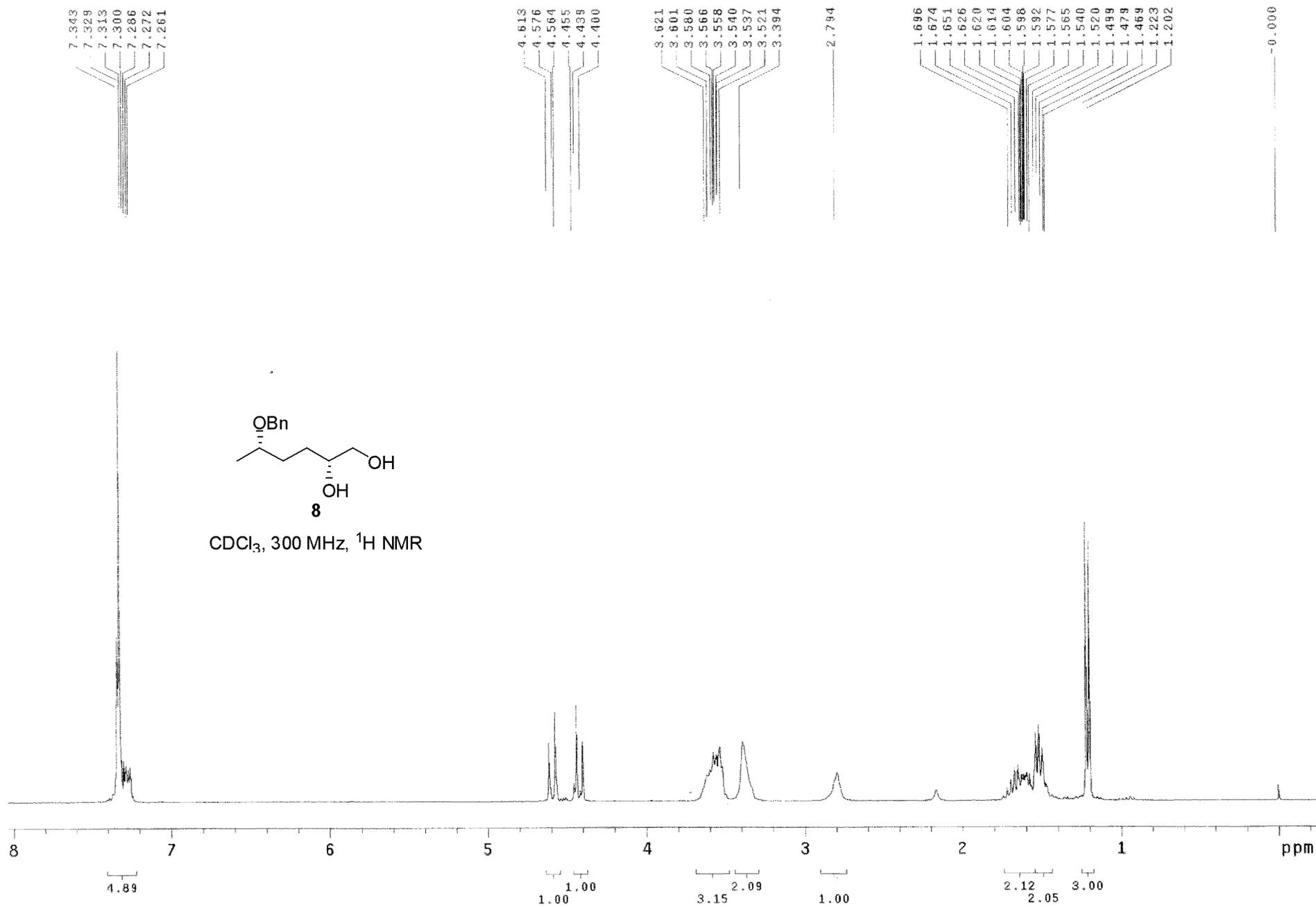
138.458

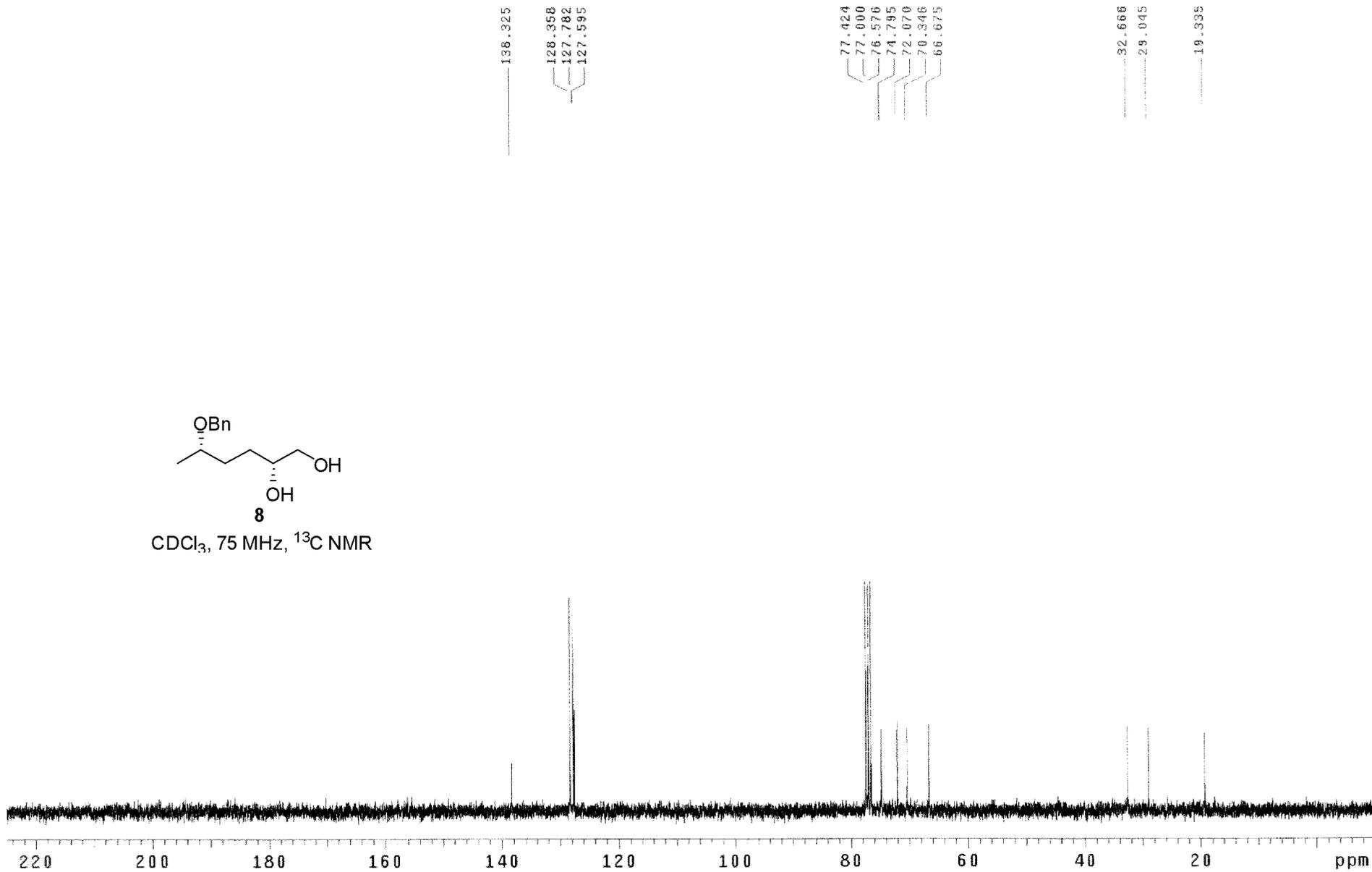
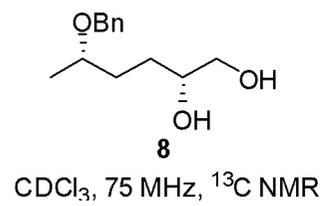
128.358
127.728
127.572

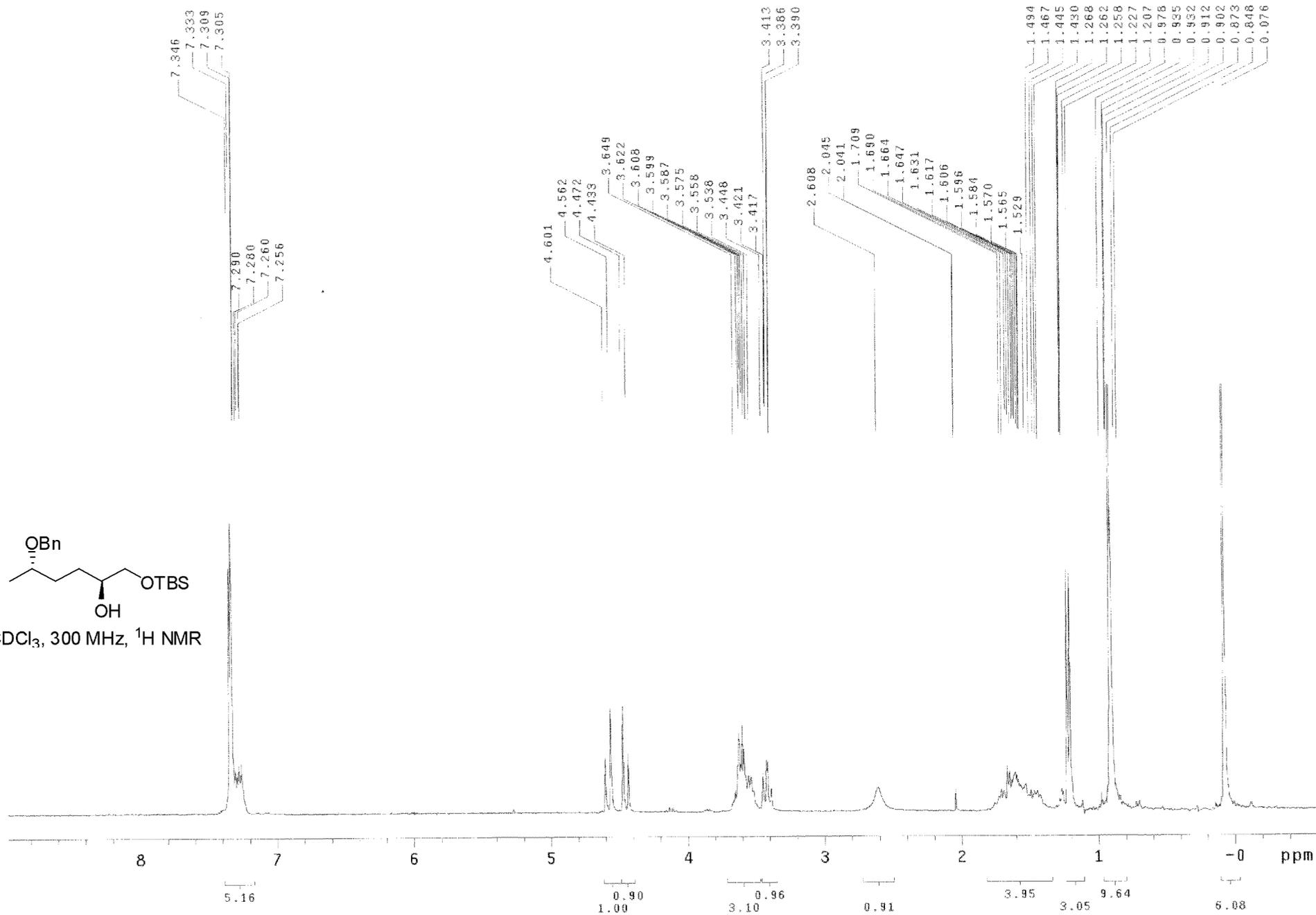
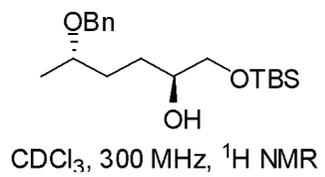
77.424
77.000
76.576
74.917
72.120
70.445
66.663

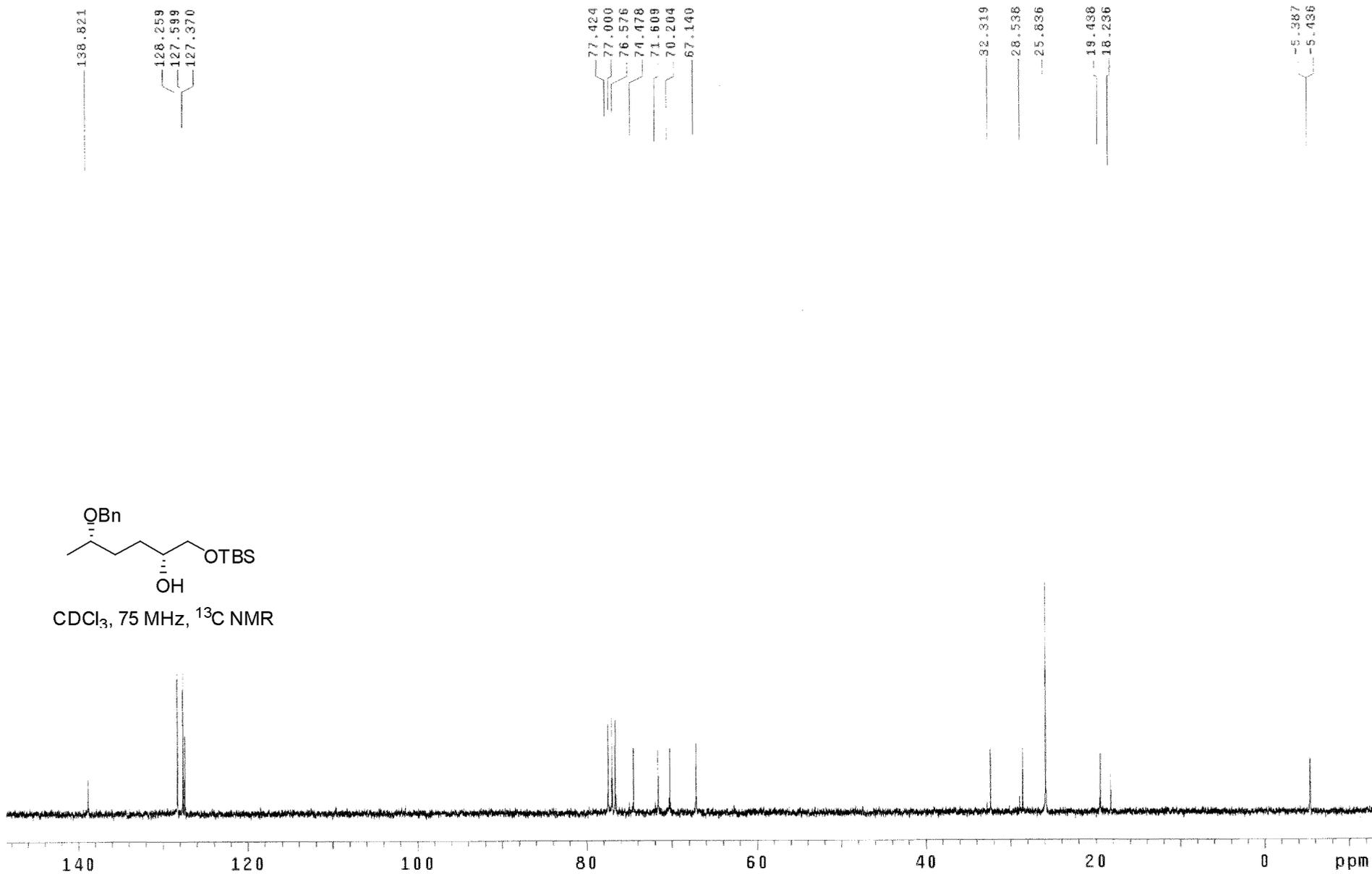
32.586
28.973

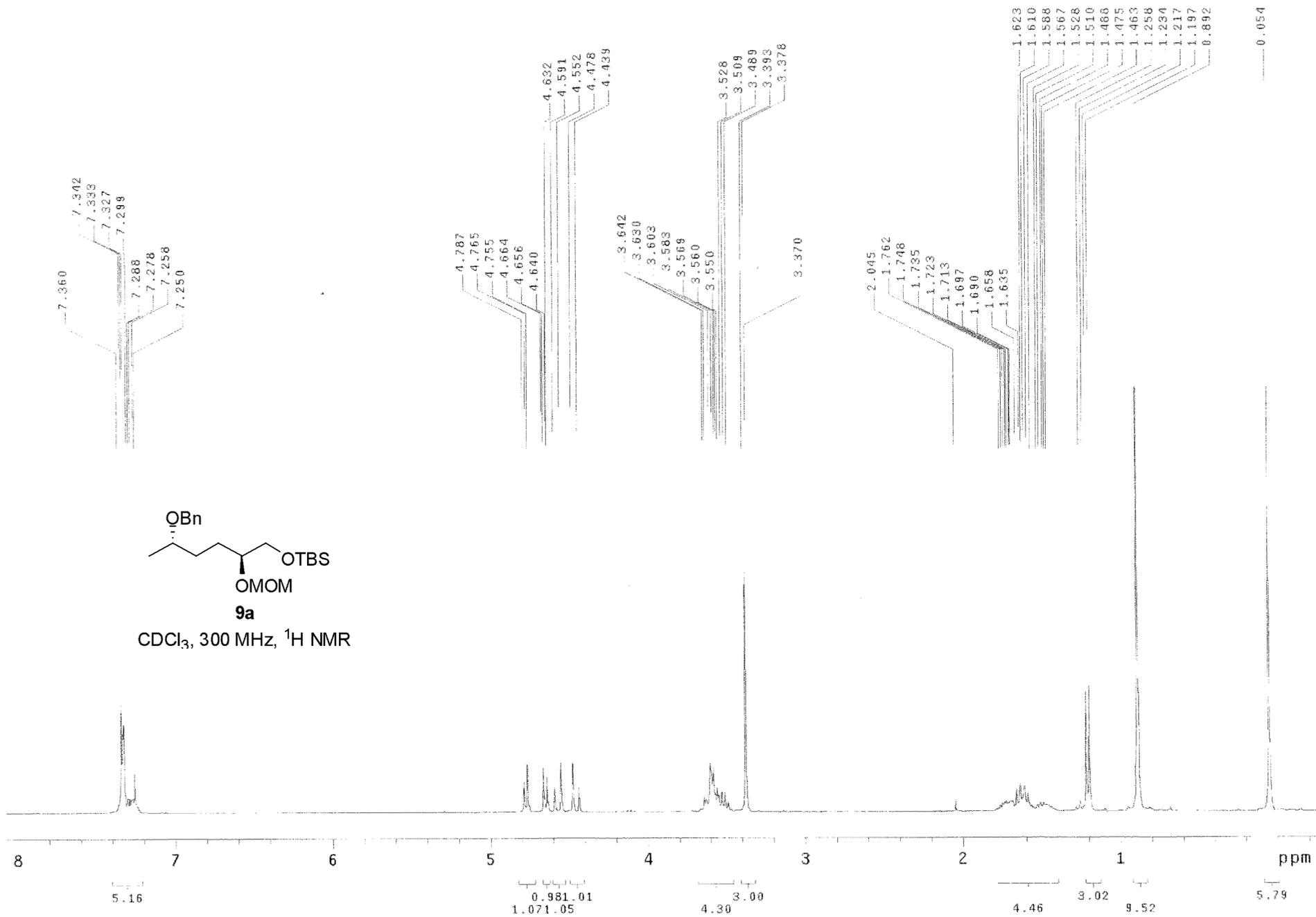
19.300

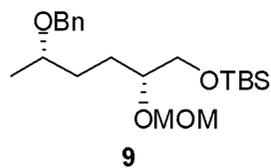
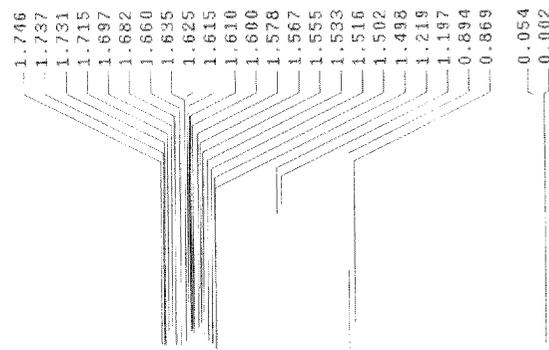
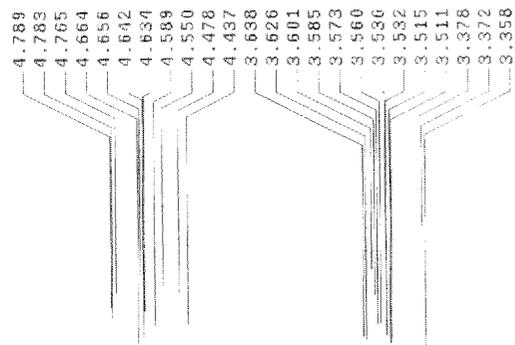
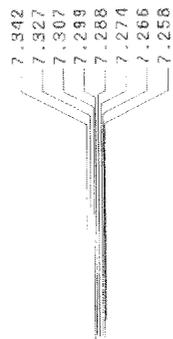




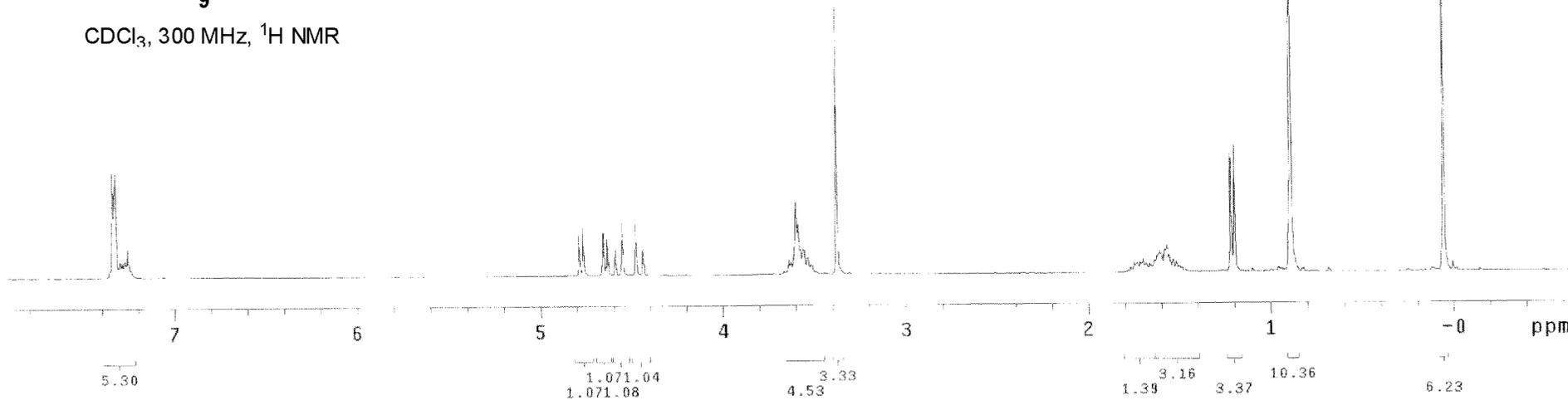


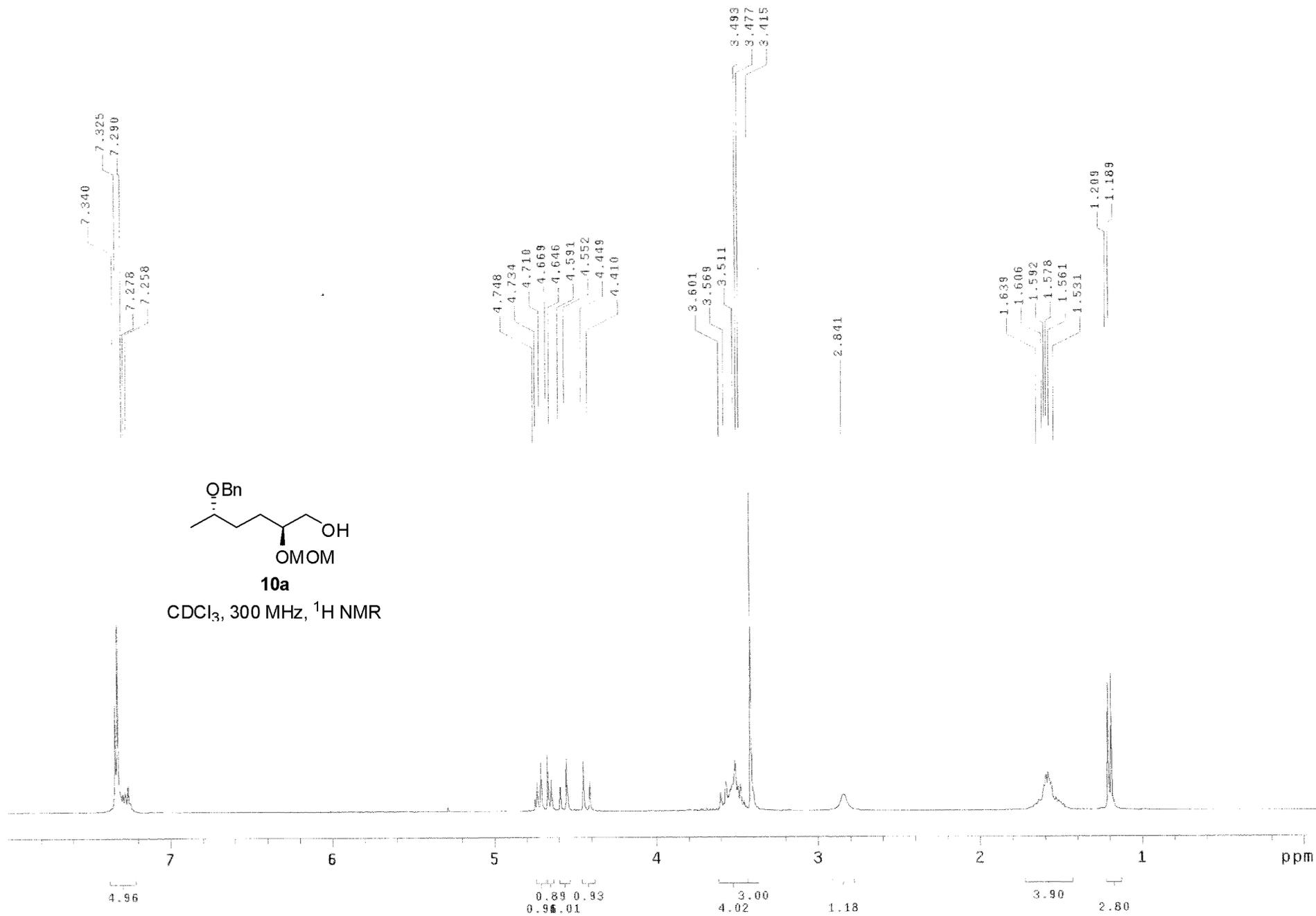


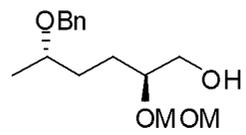




CDCl₃, 300 MHz, ¹H NMR

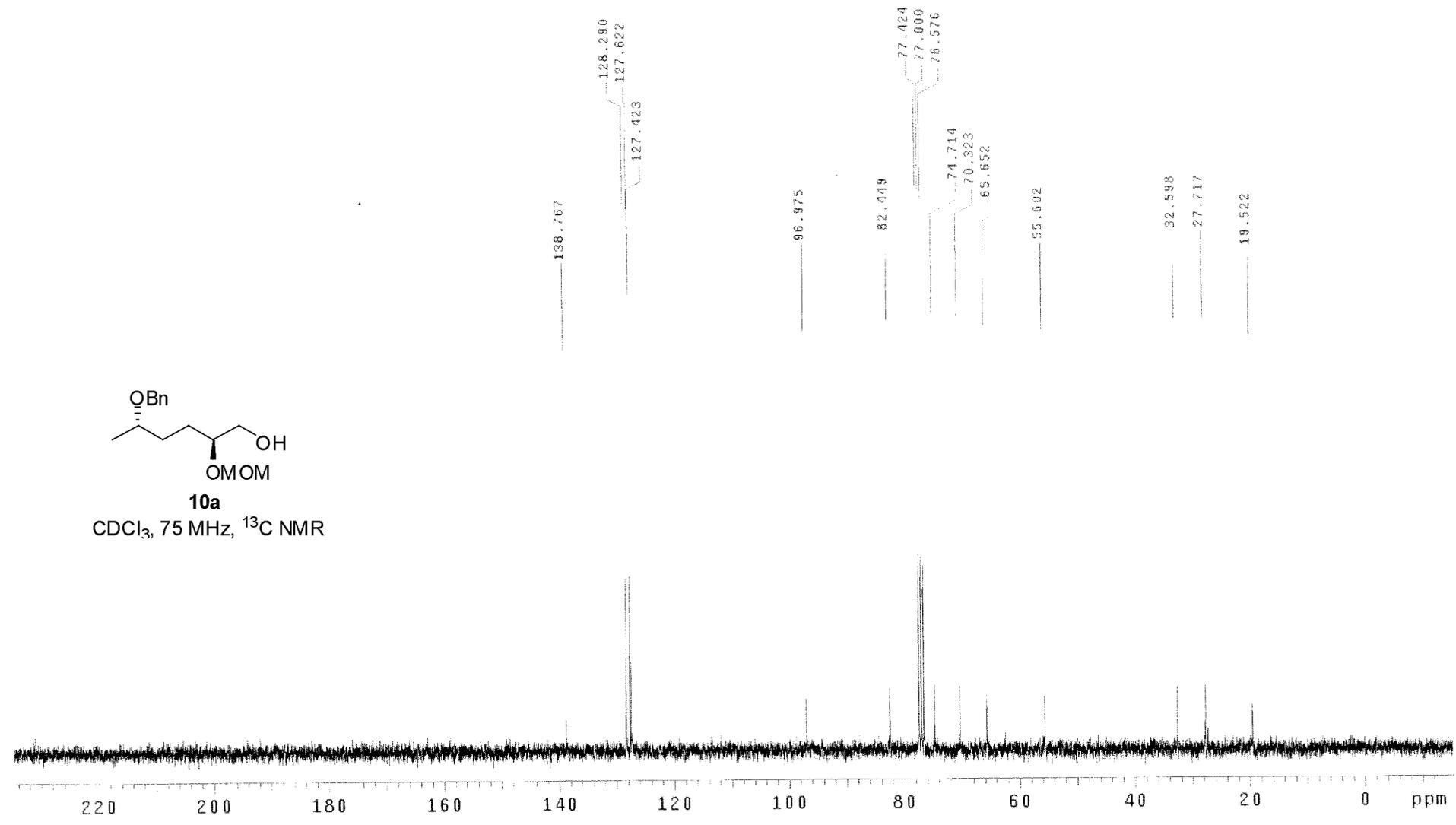


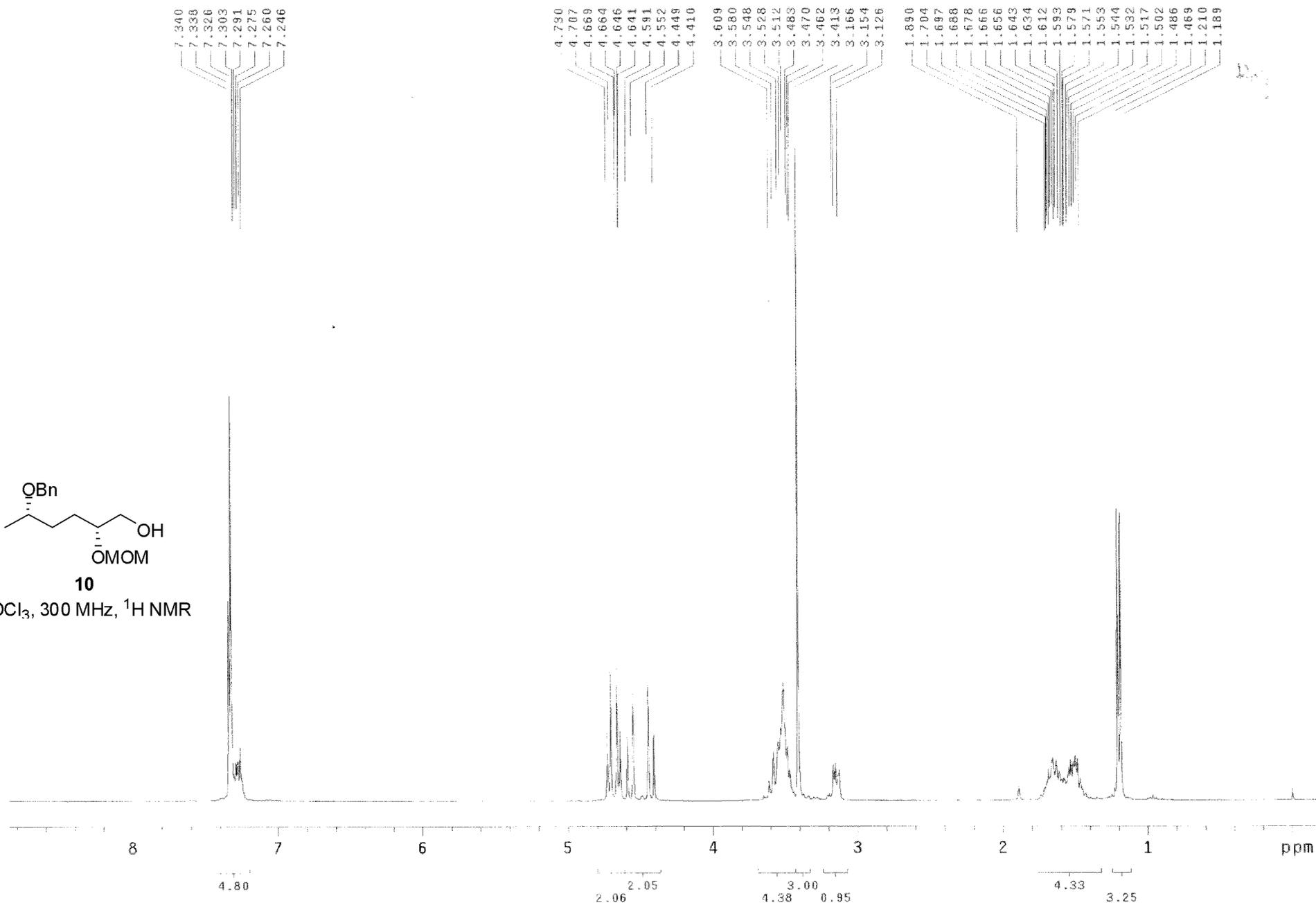
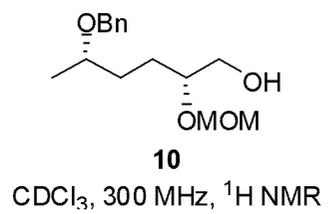


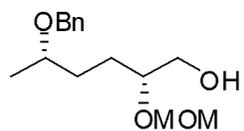


10a

CDCl₃, 75 MHz, ¹³C NMR

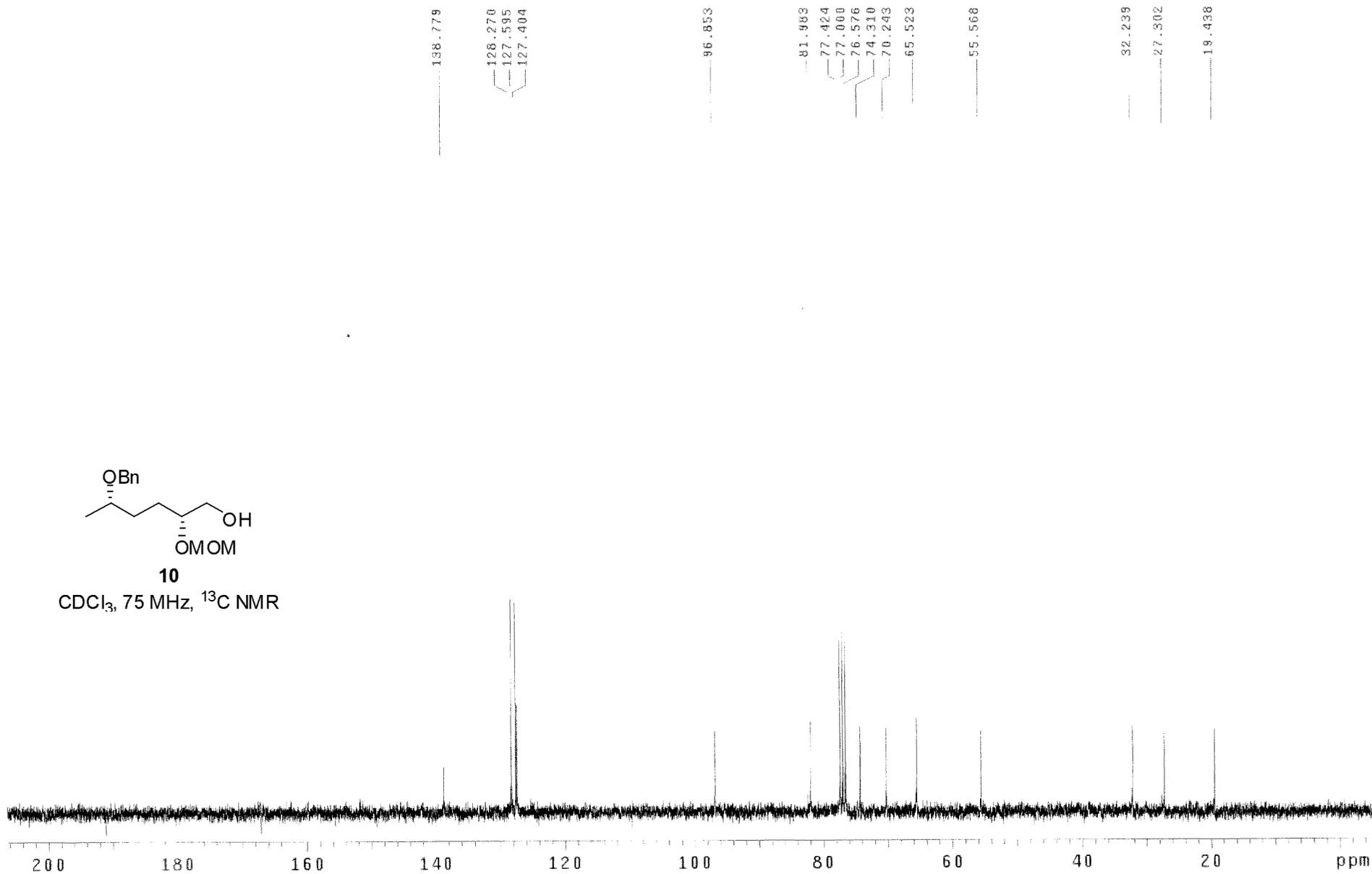






10

CDCl₃, 75 MHz, ¹³C NMR



7.340
7.329
7.290
7.279
7.268
7.262
6.850
6.834
6.810
6.795

6.003
6.001
5.964
5.962

4.635
4.618
4.605
4.588
4.577
4.571
4.556
4.445
4.416
4.210
4.203
4.188
4.173
3.748
3.561
3.542
3.526
3.511
3.496
3.368

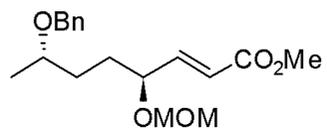
1.787
1.780
1.773
1.766
1.752
1.745
1.662
1.641
1.620
1.614
1.608
1.593
1.576
1.209
1.194

```

NAME      hanjj0113whea
EXPNO     10
PROCNO    1
Date_     20090113
Time      18.52
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         203
DW         60.800 usec
DE         6.50 usec
TE         291.8 K
D1         1.00000000 sec
TD0        1
  
```

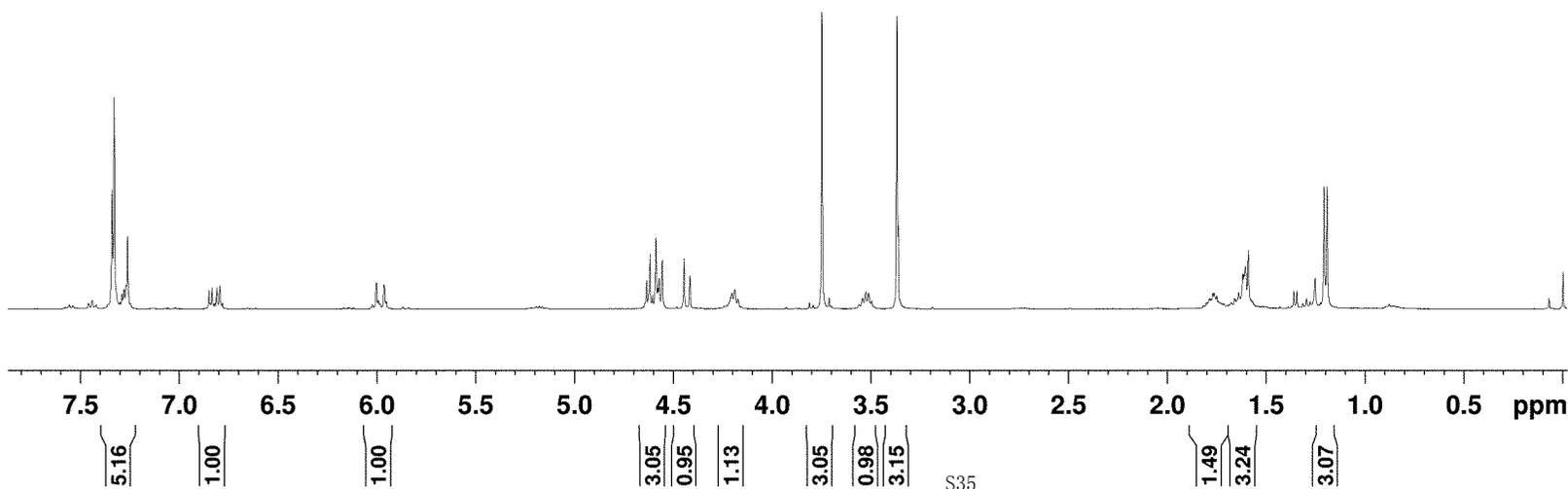
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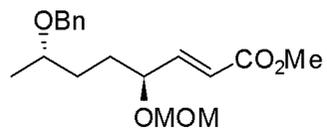
===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300043 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



11a

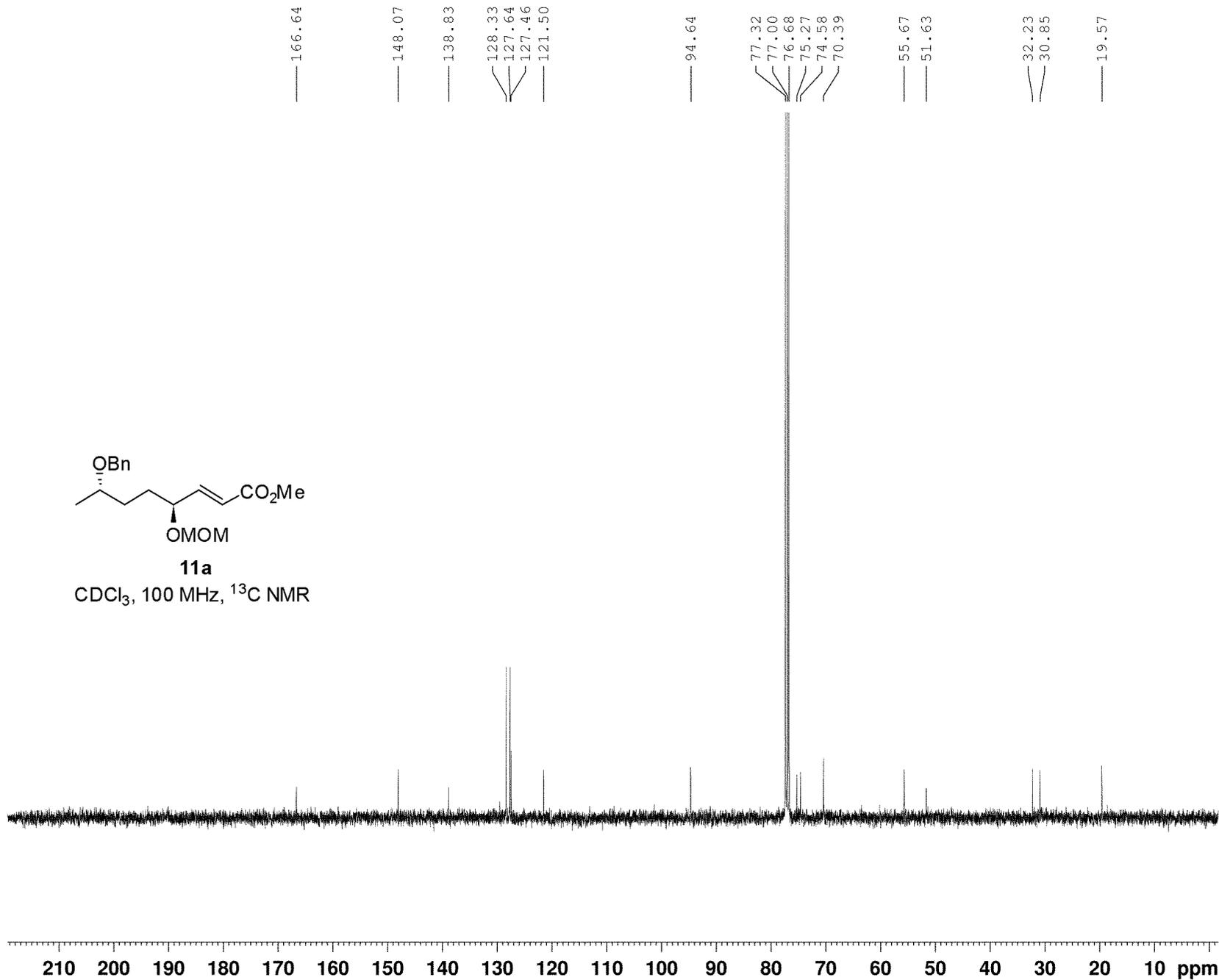
CDCl₃, 400 MHz, ¹H NMR





11a

CDCl₃, 100 MHz, ¹³C NMR



```

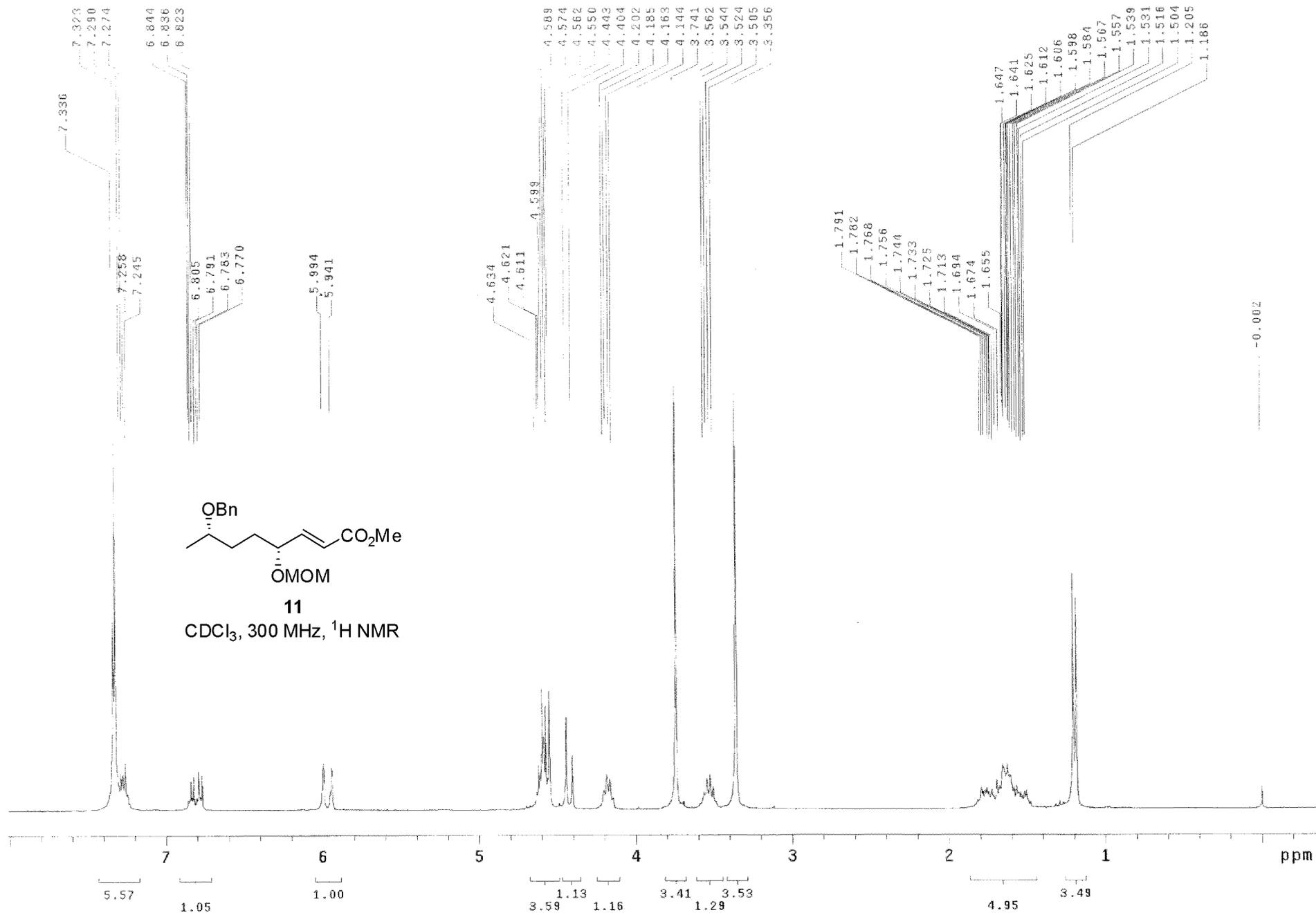
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EXPNO     12
PROCNO    1
Date_     20090113
Time      19.02
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         150
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         292.5 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

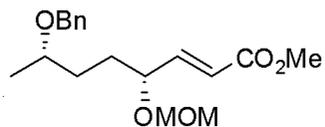
```

===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1       100.6228298 MHz
  
```

```

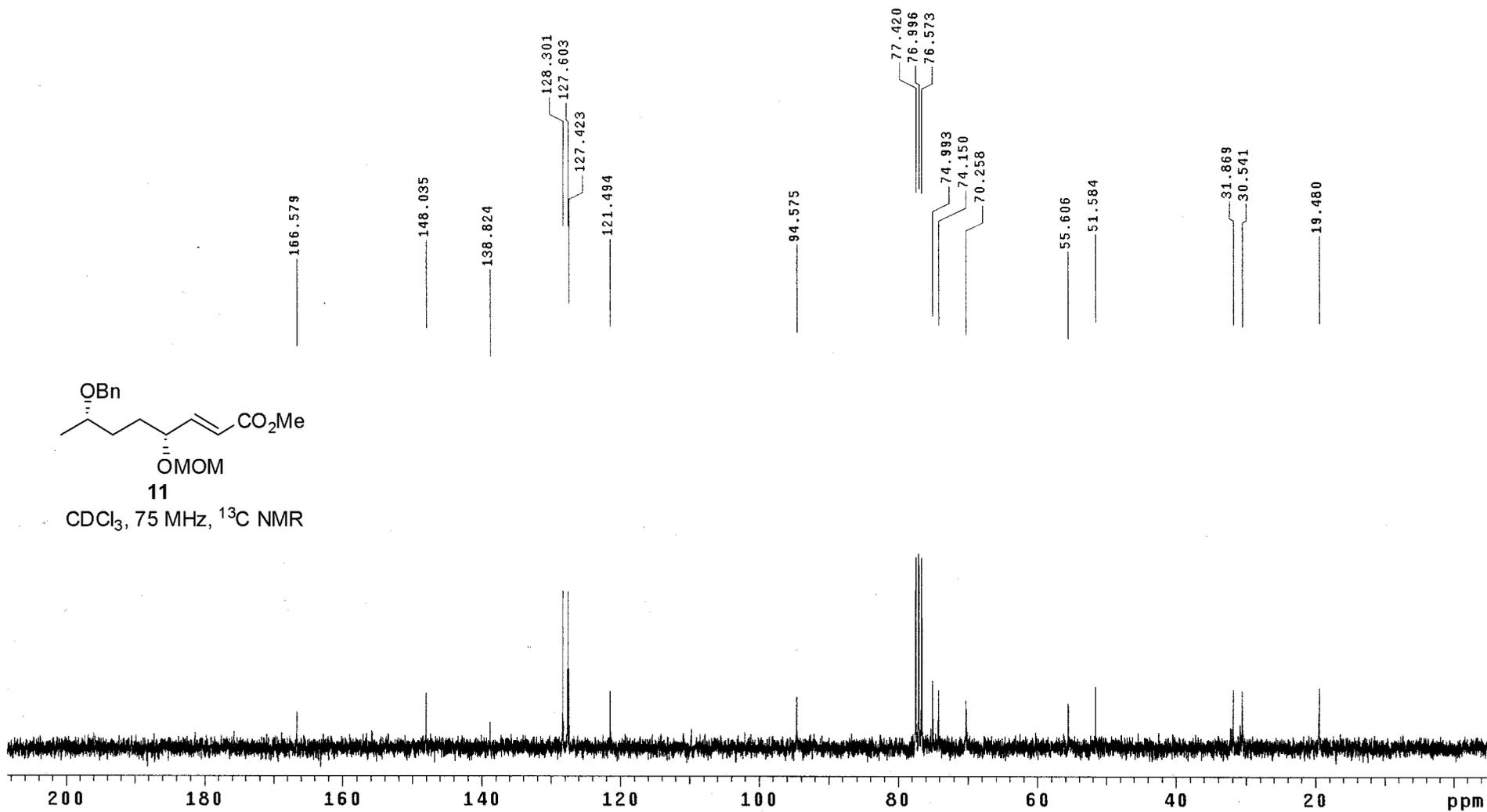
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -2.00 dB
PL12       15.50 dB
PL13       15.50 dB
PL2W       18.19349861 W
PL12W      0.32353121 W
PL13W      0.32353121 W
SFO2       400.1316005 MHz
SI         32768
SF         100.6127707 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

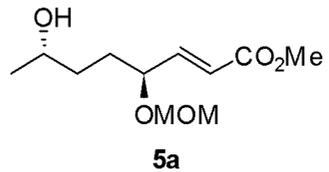




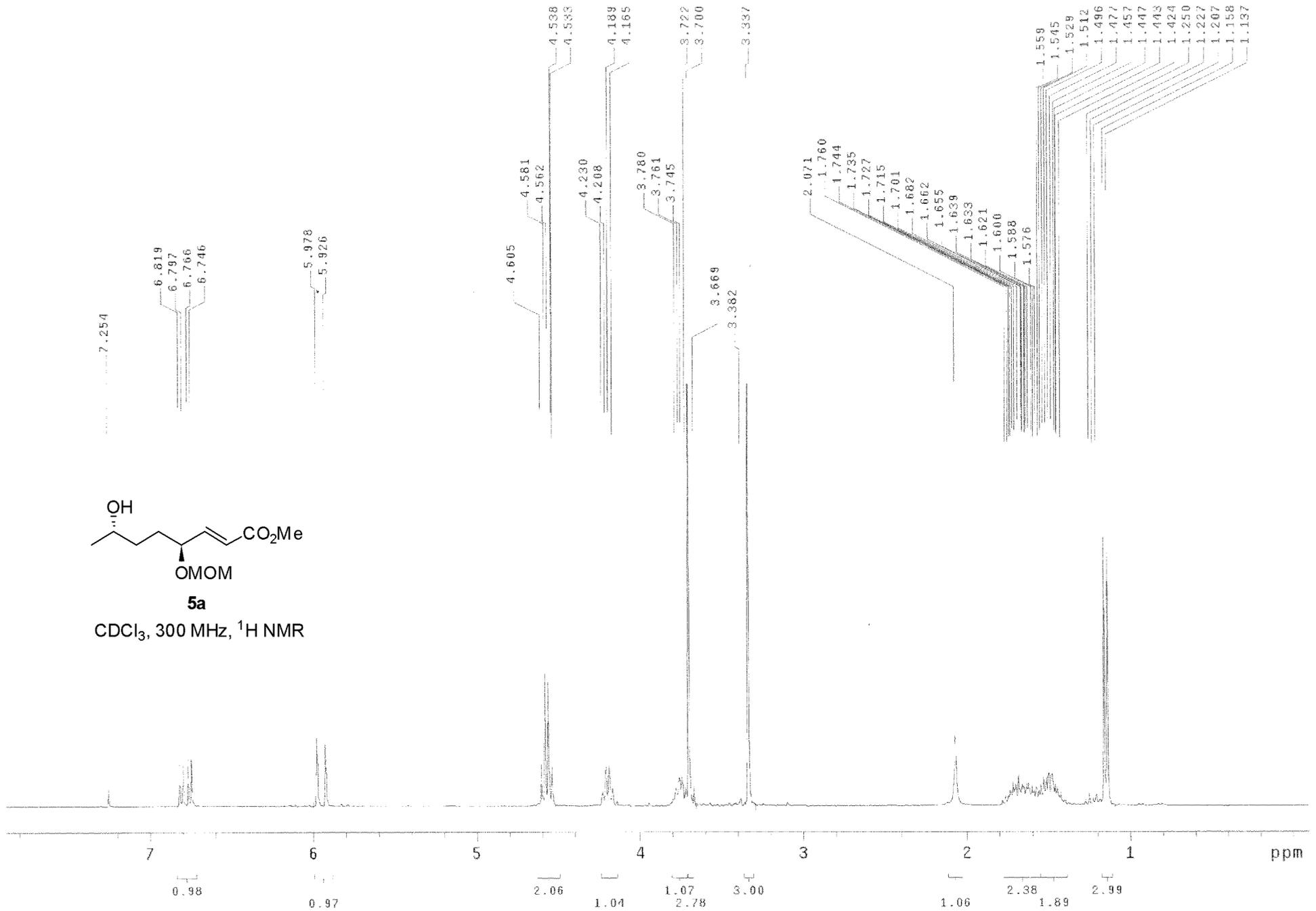
11

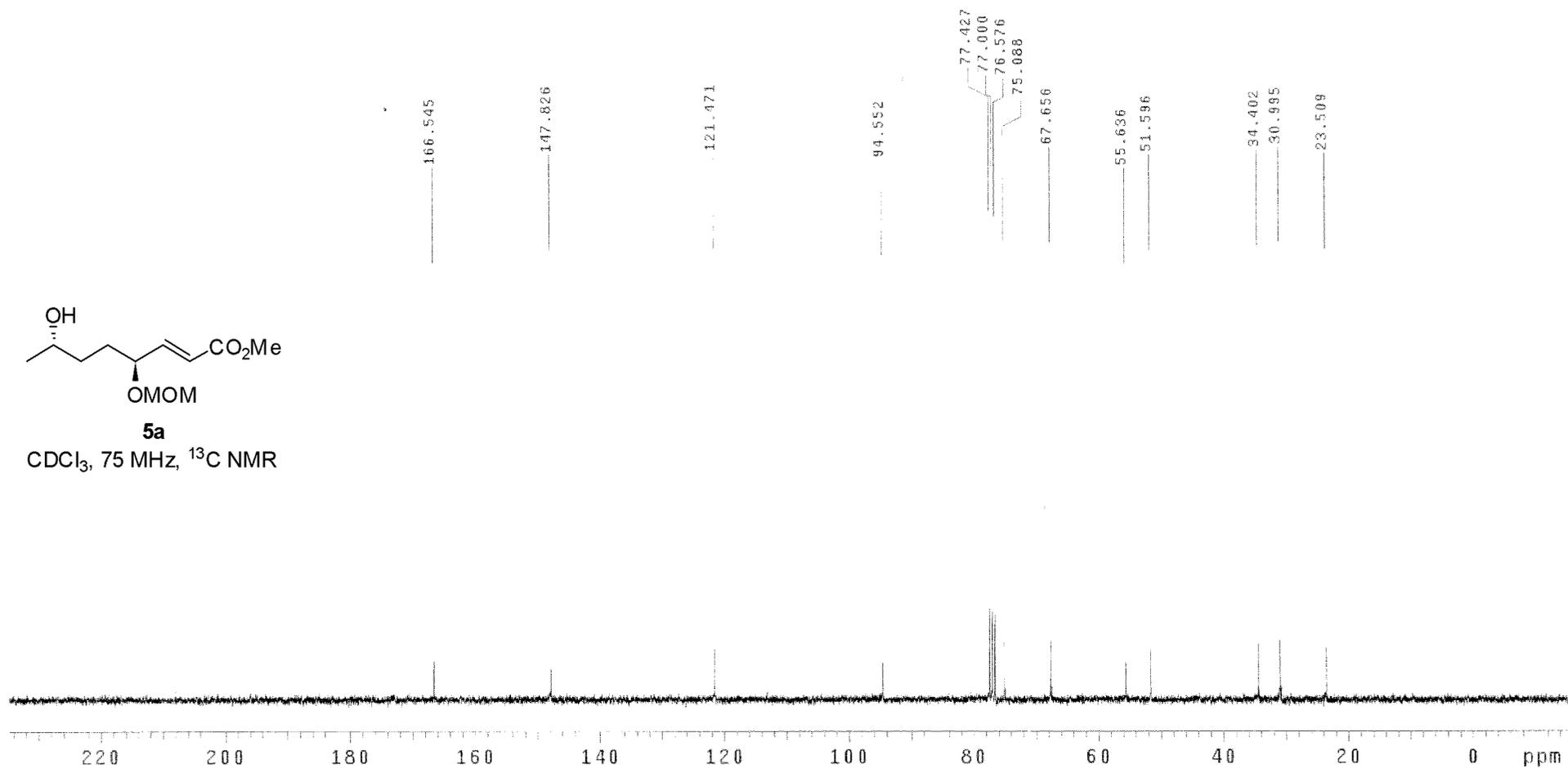
CDCl₃, 75 MHz, ¹³C NMR

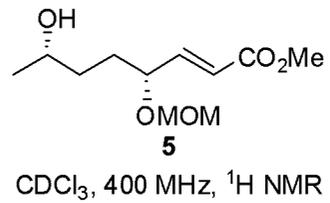
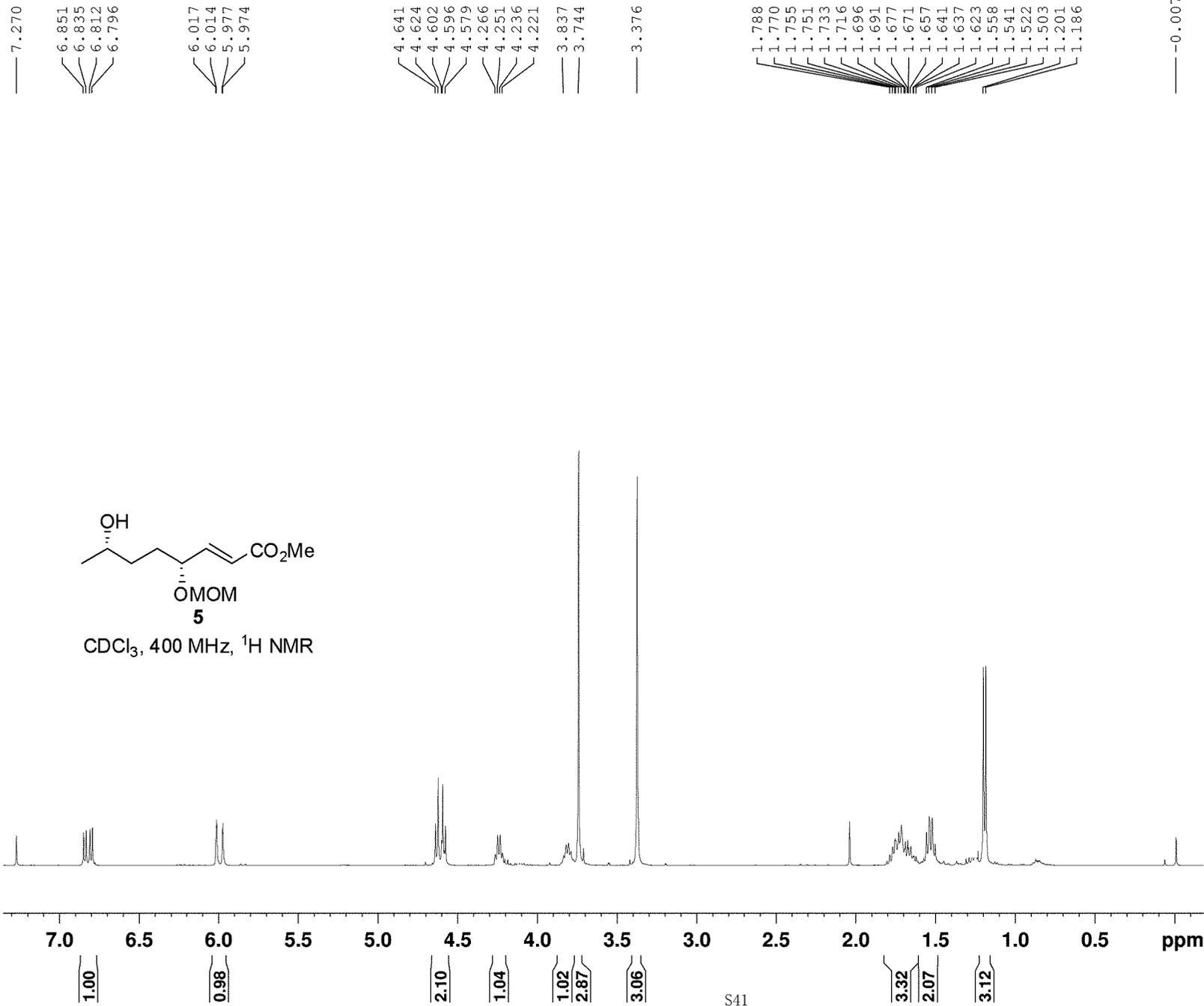




CDCl₃, 300 MHz, ¹H NMR



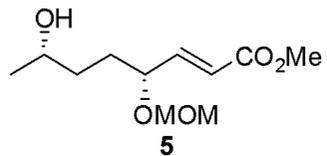




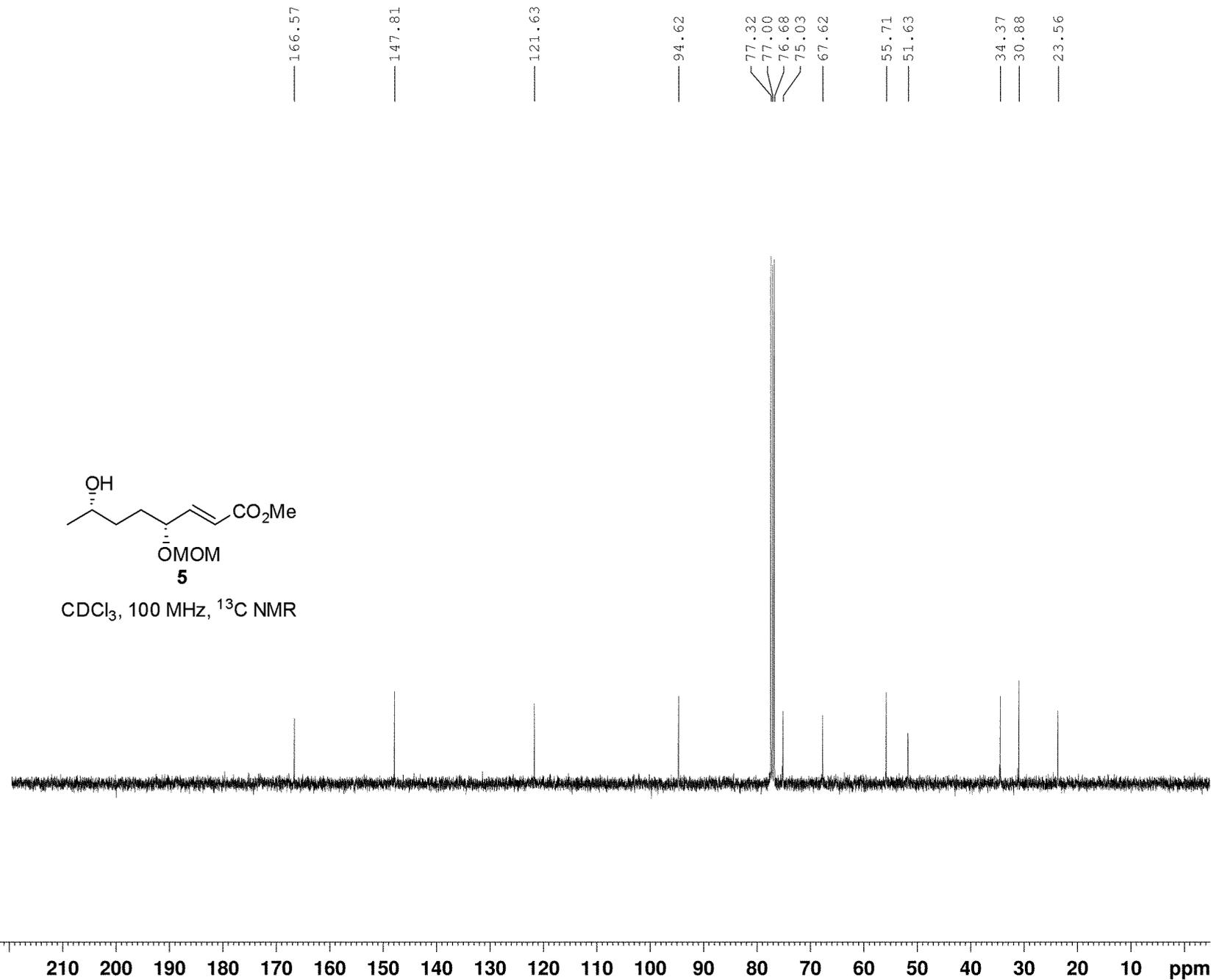
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EXPNO     10
PROCNO    1
Date_     20090110
Time      18.17
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         8
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         144
DW         60.800 usec
DE         6.50 usec
TE         291.7 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.60 usec
PL1        0.00 dB
PL1W       11.47932053 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300012 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



CDCl₃, 100 MHz, ¹³C NMR



```

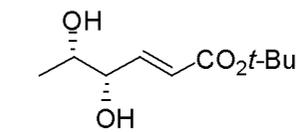
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EXPNO     11
PROCNO    1
Date_     20090110
Time      18.22
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         64
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG         2050
DW        20.800 usec
DE         6.50 usec
TE        292.3 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz
  
```

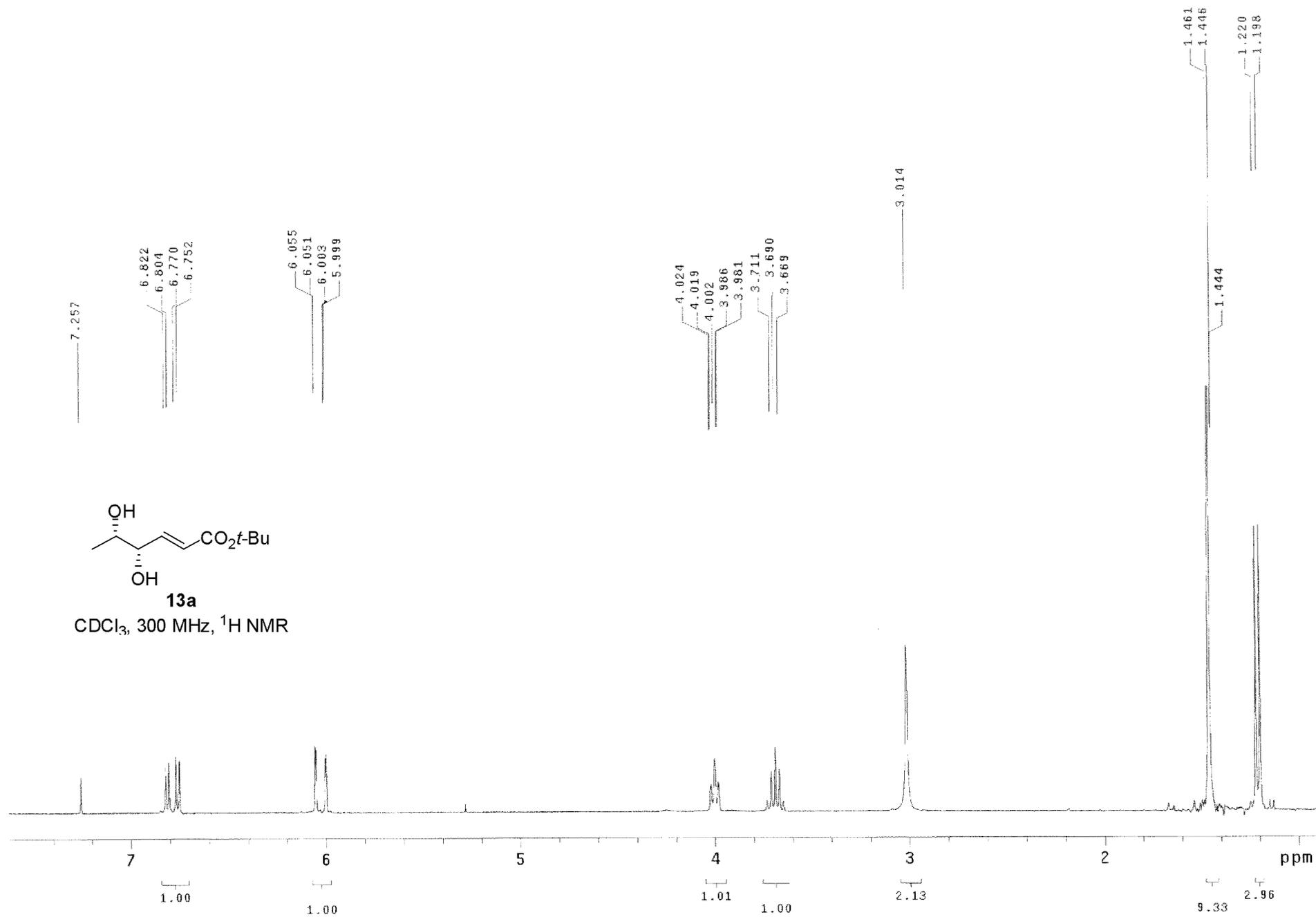
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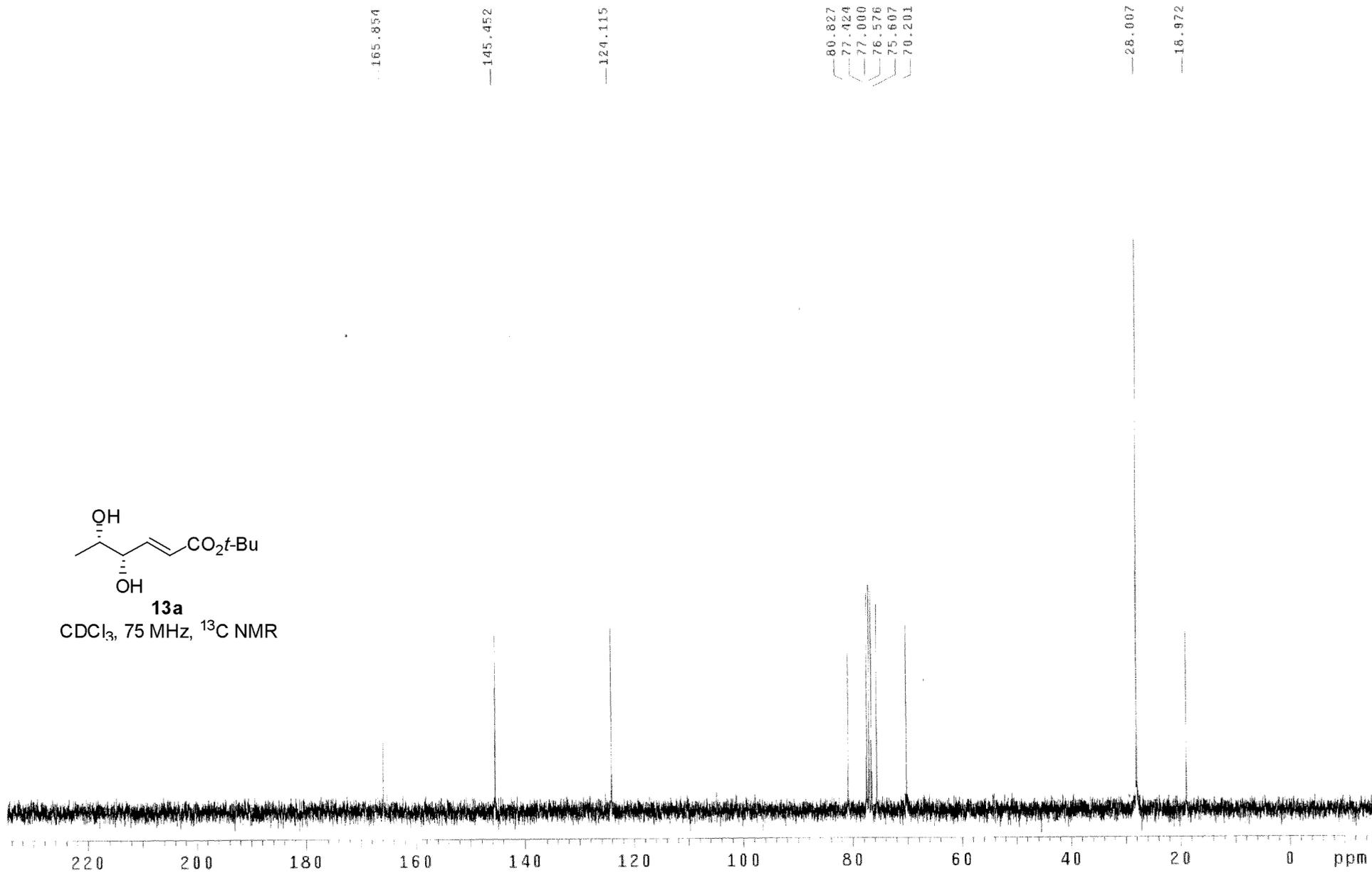
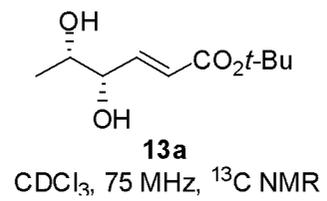
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       -2.00 dB
PL12      15.50 dB
PL13      15.50 dB
PL2W      18.19349861 W
PL12W     0.32353121 W
PL13W     0.32353121 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6127718 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

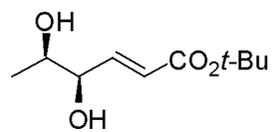
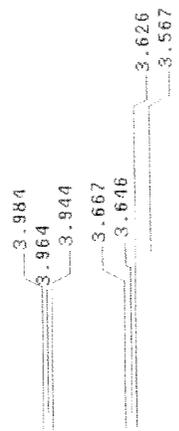
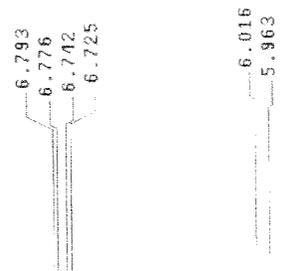


13a

CDCl₃, 300 MHz, ¹H NMR

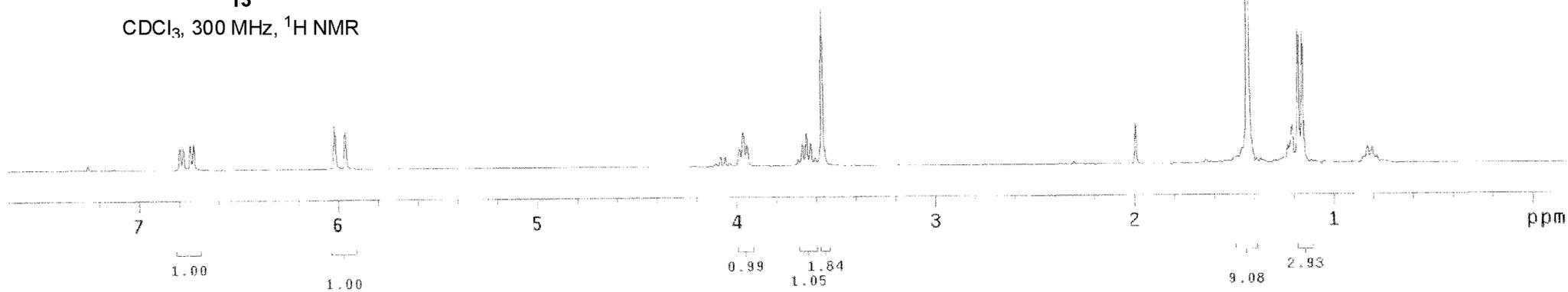


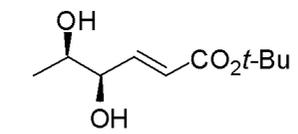




13

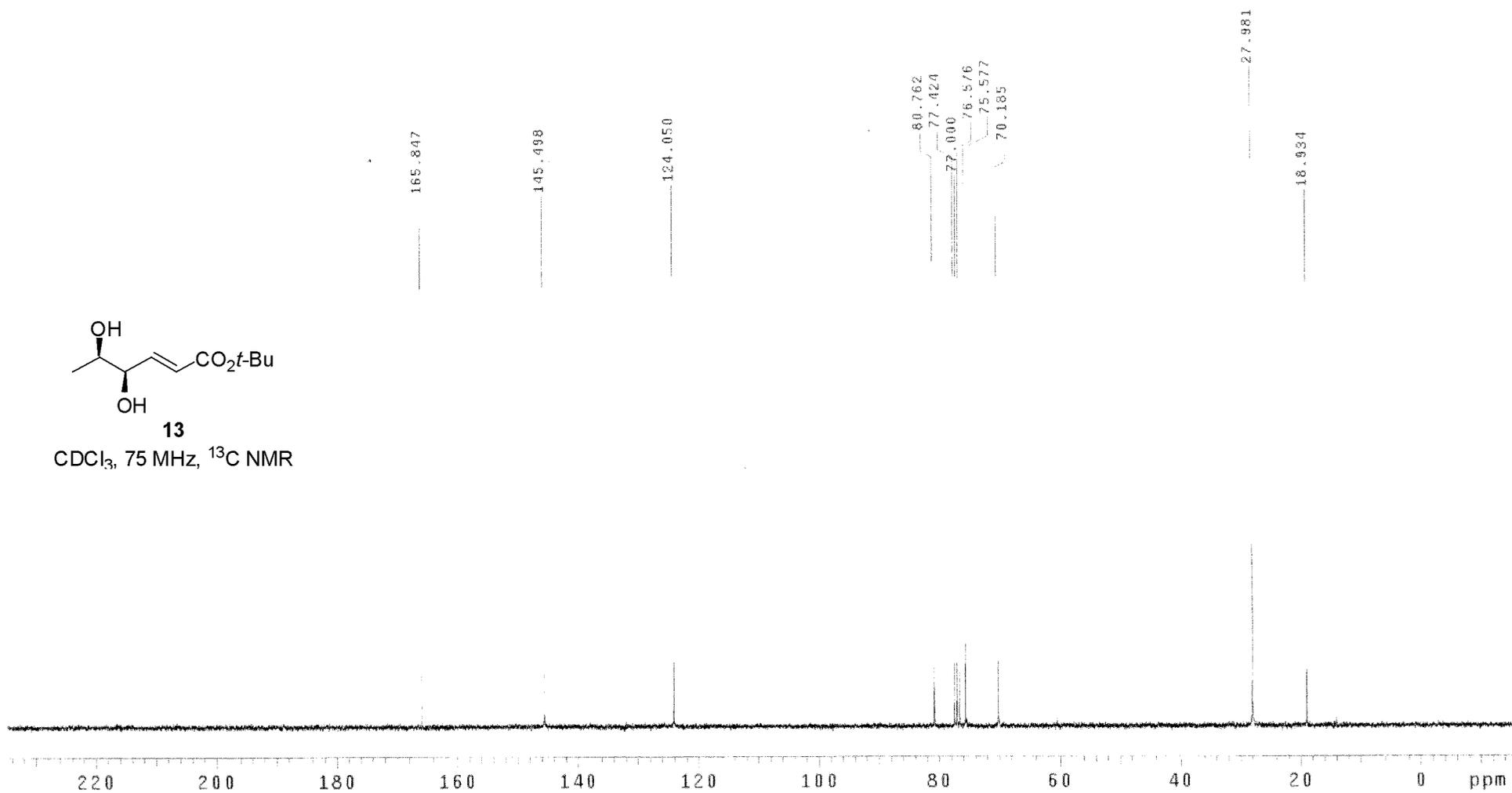
CDCl₃, 300 MHz, ¹H NMR

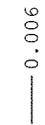
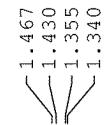
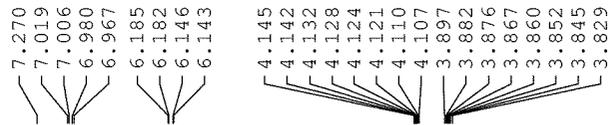
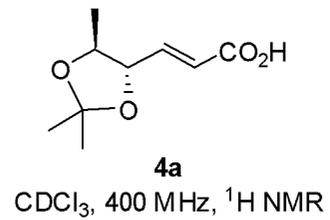




13

CDCl₃, 75 MHz, ¹³C NMR



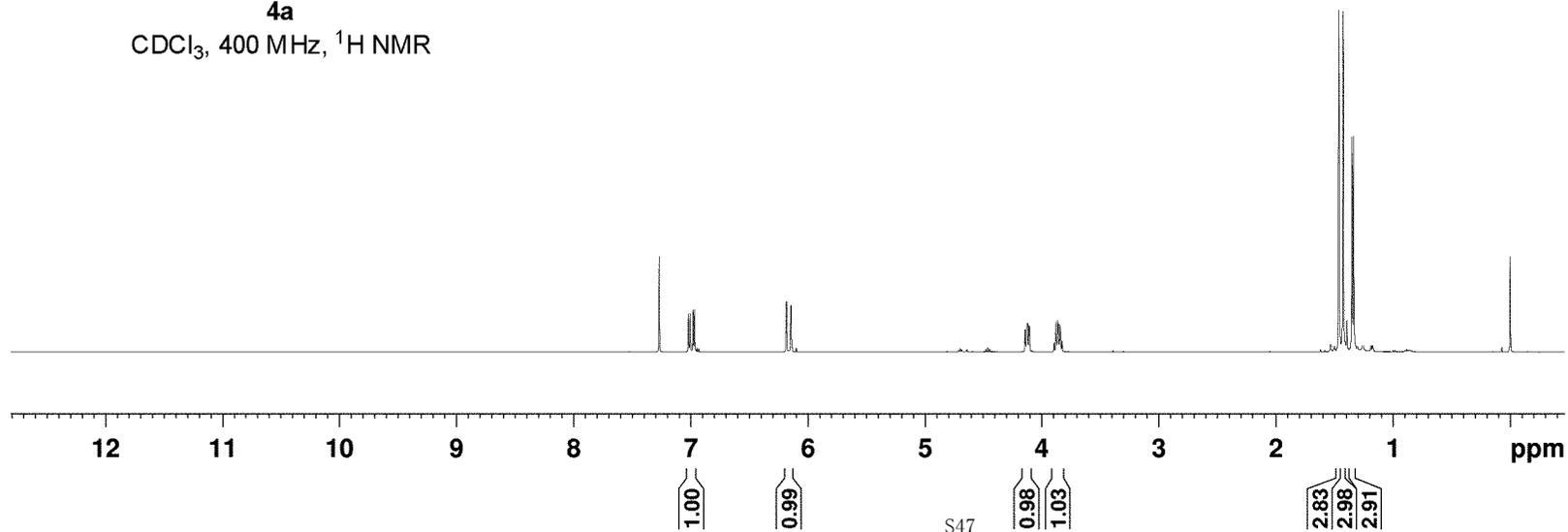


```

NAME      hanjj0117
EXPNO     10
PROCNO    1
Date_     20090117
Time      22.08
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         32
DS         2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ         3.9846387 sec
RG         456
DW         60.800 usec
DE         6.50 usec
TE         290.9 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SFO1      400.1324710 MHz
SI         32768
SF         400.1300013 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



170.79

146.25

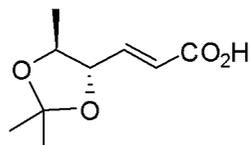
121.70

109.46

81.46
77.32
77.00
76.68
76.42

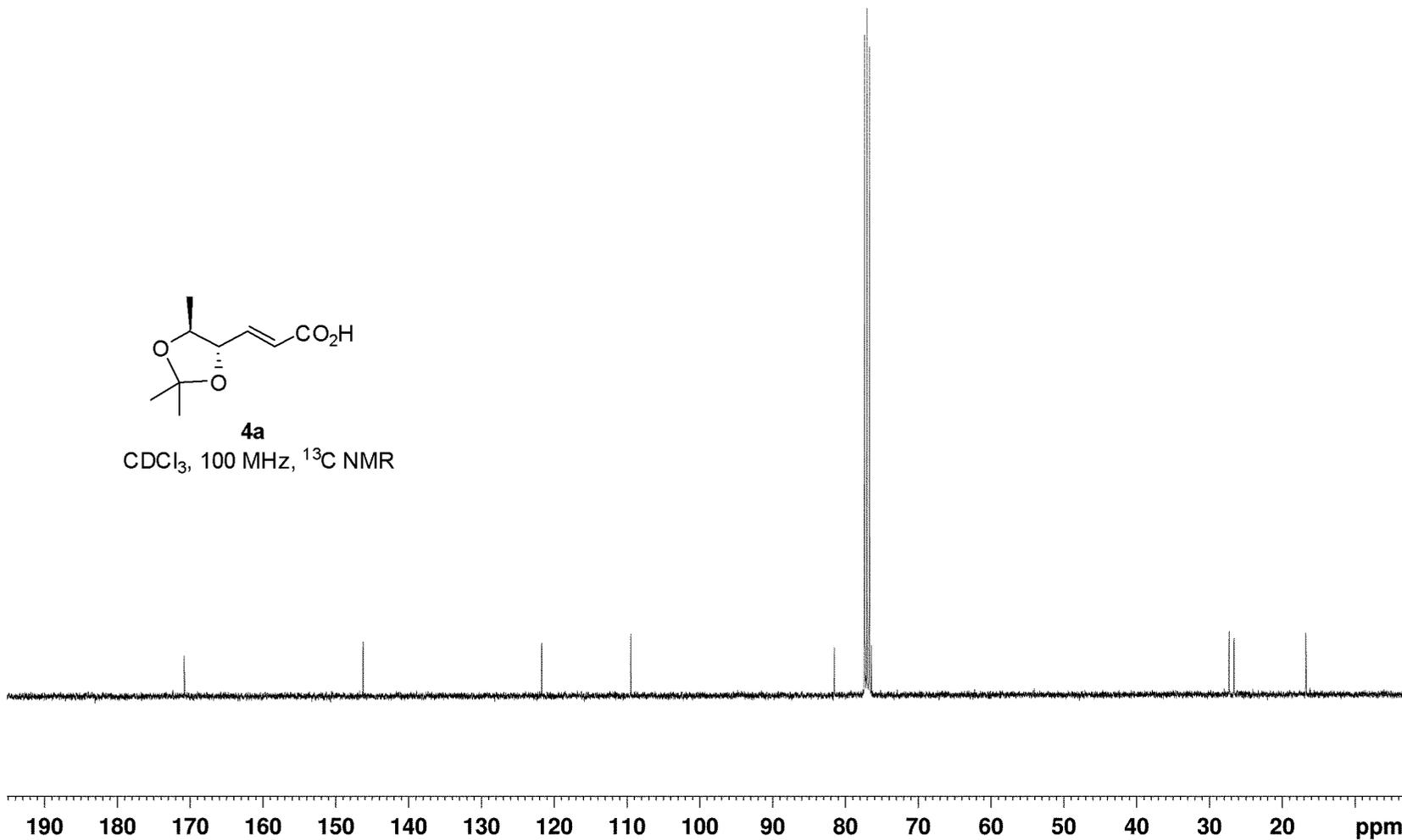
27.27
26.59

16.71



4a

CDCl₃, 100 MHz, ¹³C NMR



```
NAME      hanjj0117
EXPNO     11
PROCNO    1
Date_     20090117
Time      22.39
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        500
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        50.8
DW        20.800 usec
DE        6.50 usec
TE        291.7 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1
```

```
===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz
```

```
===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       -2.00 dB
PL12      15.50 dB
PL13      15.50 dB
PL2W      18.19349861 W
PL12W     0.32353121 W
PL13W     0.32353121 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6127700 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
```

11.545

7.270
7.004
6.990
6.965
6.951
6.167
6.164
6.128
6.125

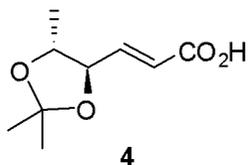
4.135
4.132
4.121
4.118
4.114
4.111
4.100
4.098
3.886
3.871
3.865
3.856
3.849
3.840
3.834
3.819

1.452
1.417
1.340
1.325

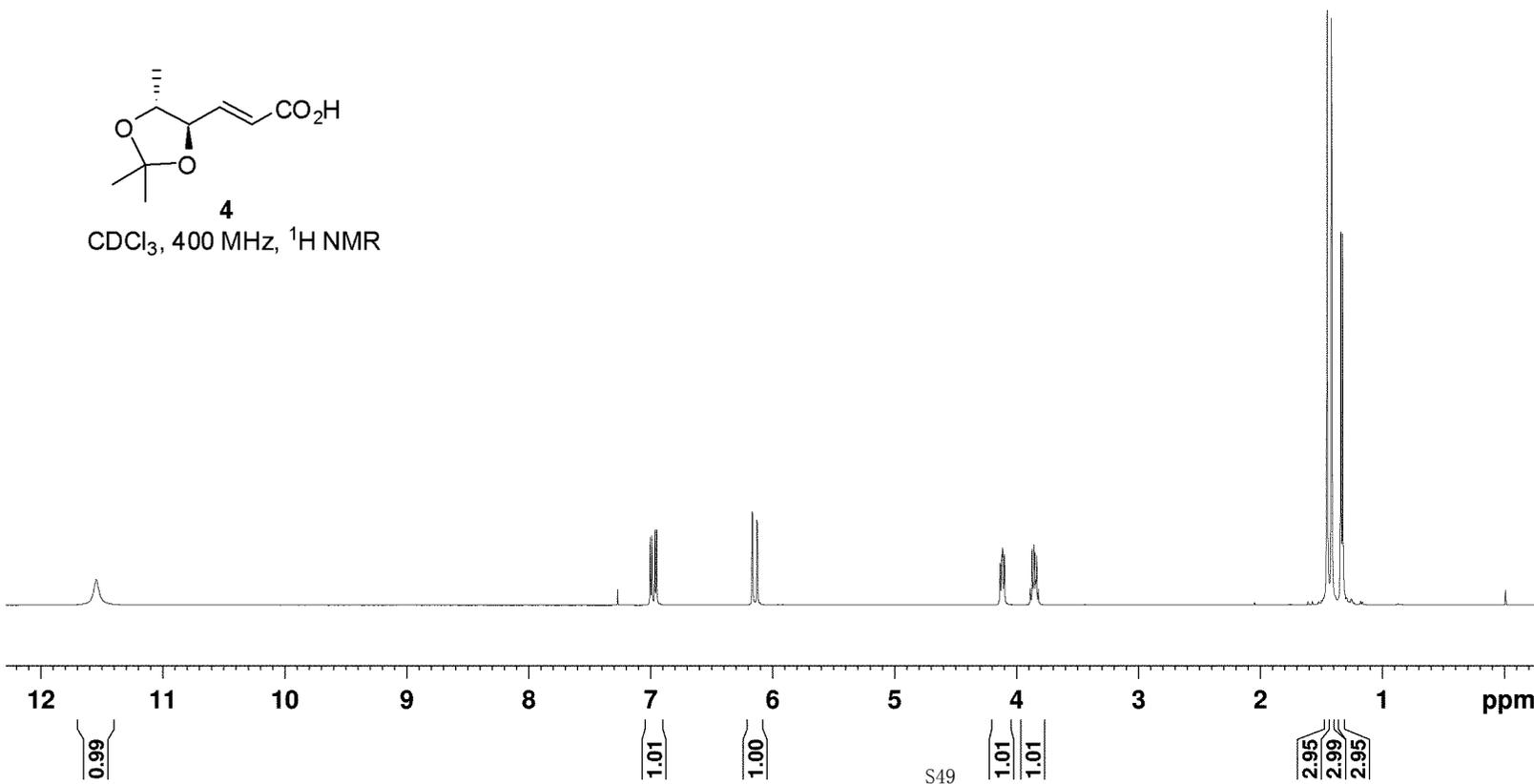
-0.011

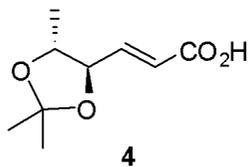
NAME hanjj090107
EXPNO 10
PROCNO 1
Date_ 20090107
Time 21.05
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 16
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 71.8
DW 60.800 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.60 usec
PL1 0.00 dB
PL1W 11.47932053 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300008 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00

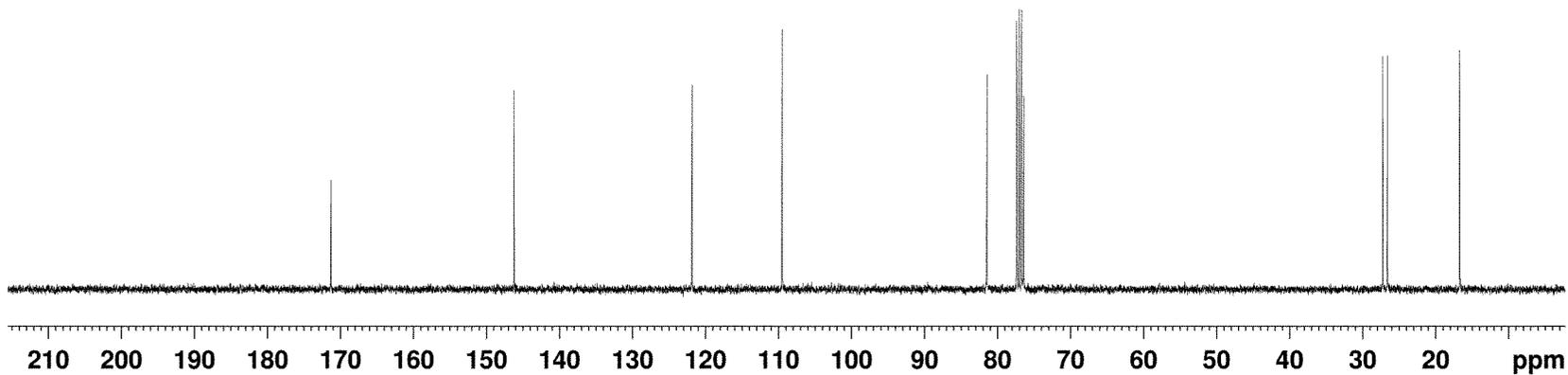


CDCl₃, 400 MHz, ¹H NMR





4
CDCl₃, 100 MHz, ¹³C NMR



S50

81.41
77.32
77.00
76.68
76.40

27.21
26.55
16.65

```

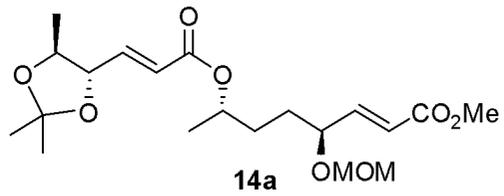
NAME      hanjj090107
EXPNO     11
PROCNO    1
Date_     20090107
Time      21.10
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         64
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         293.3 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

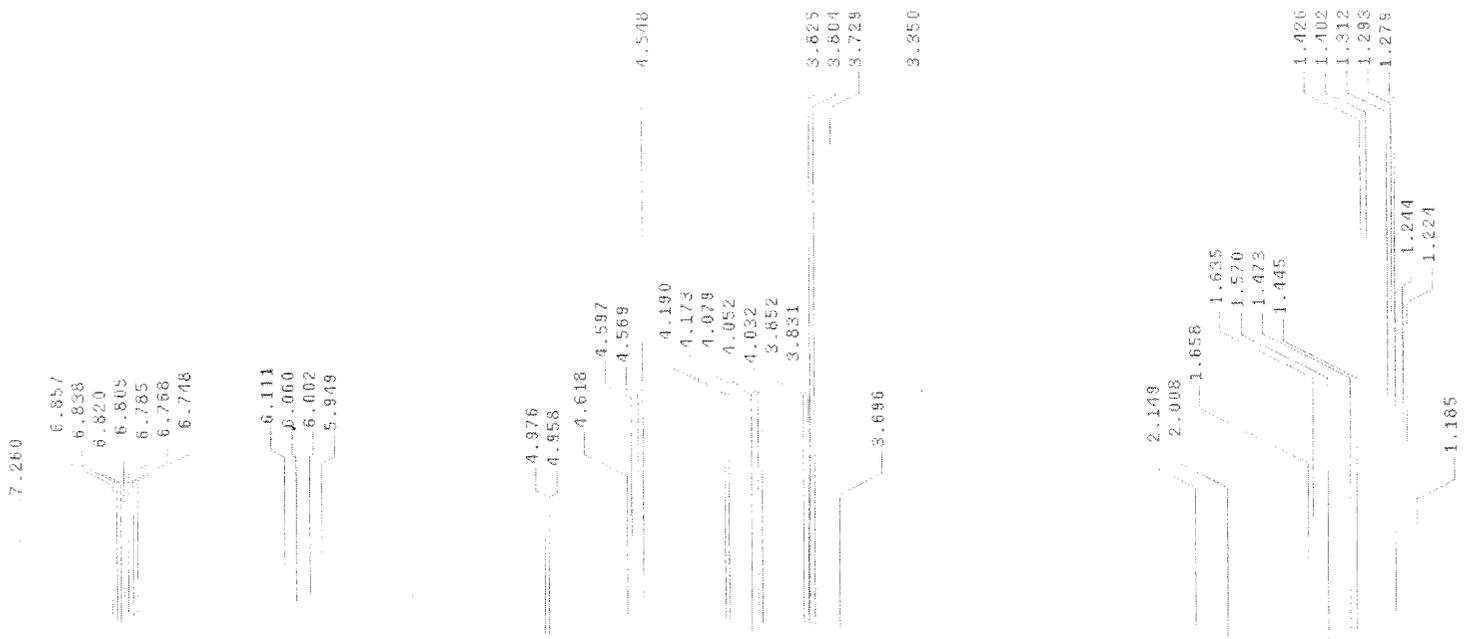
===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz
  
```

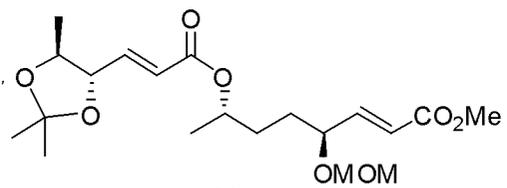
```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2       -2.00 dB
PL12      15.50 dB
PL13      15.50 dB
PL2W      18.19349861 W
PL12W     0.32353121 W
PL13W     0.32353121 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6127712 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

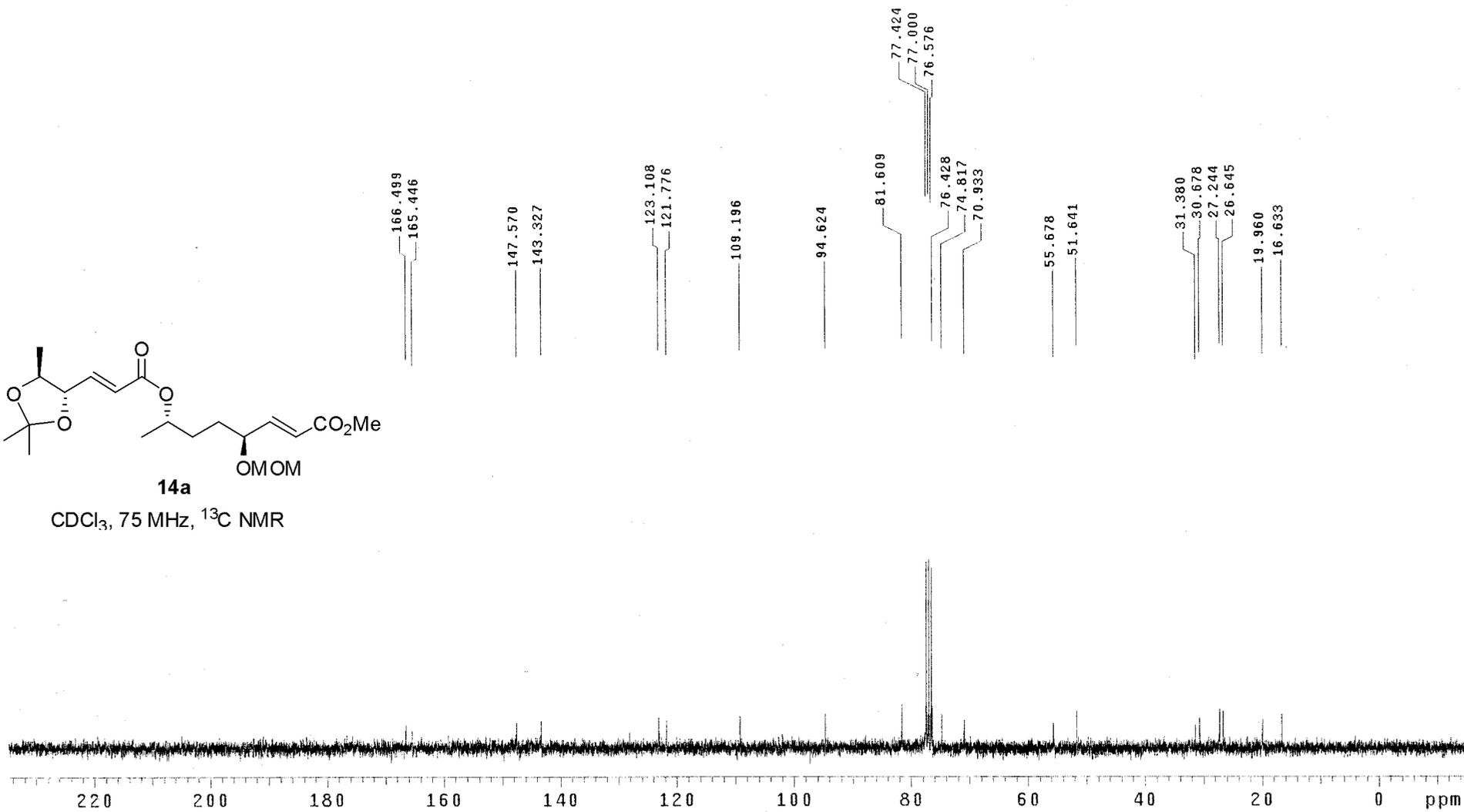


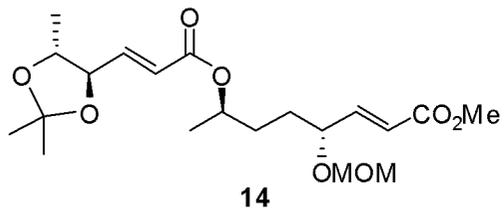
CDCl₃, 300 MHz, ¹H NMR



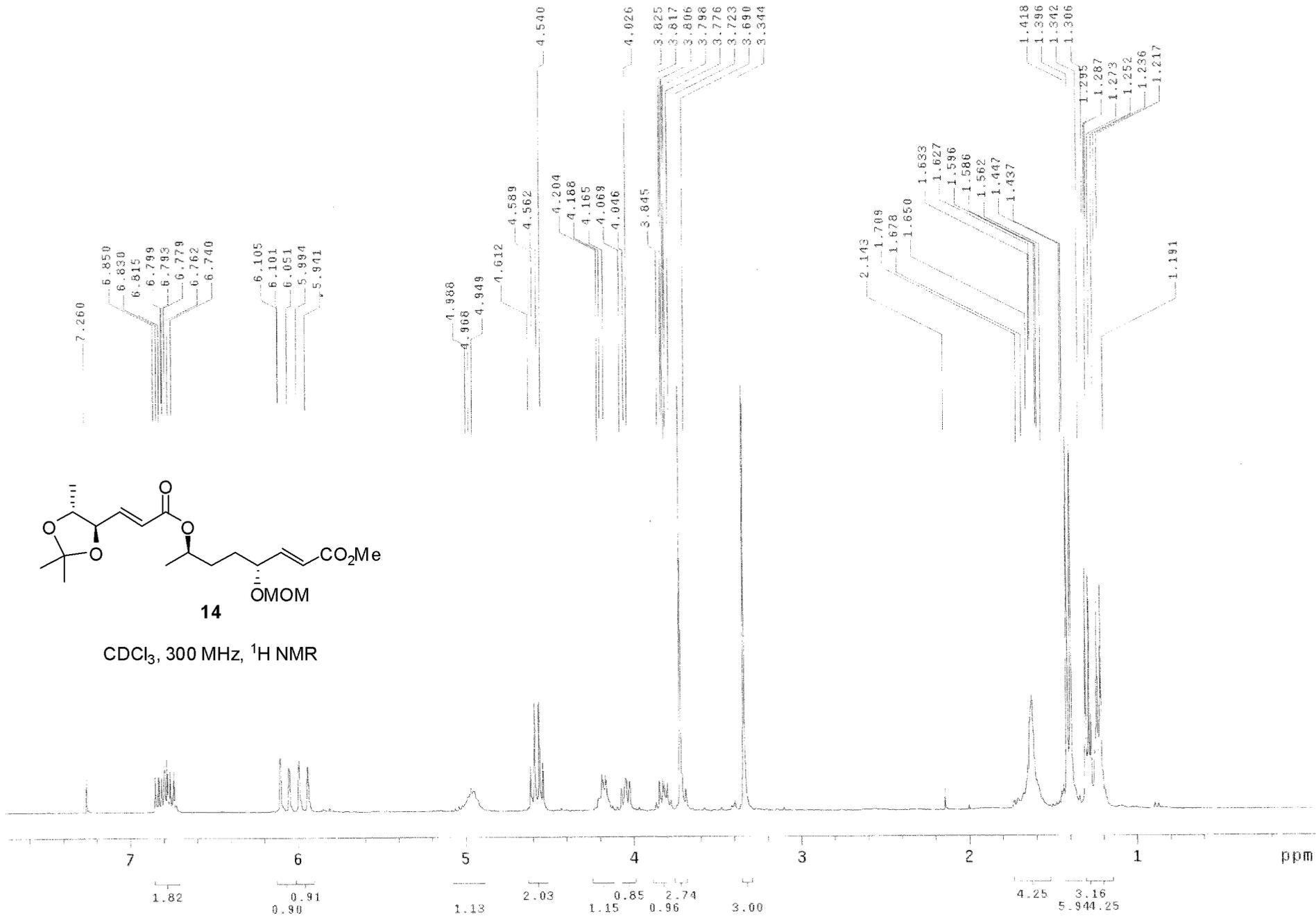


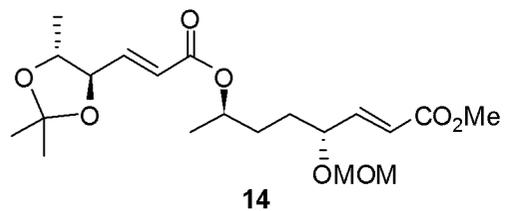
CDCl₃, 75 MHz, ¹³C NMR



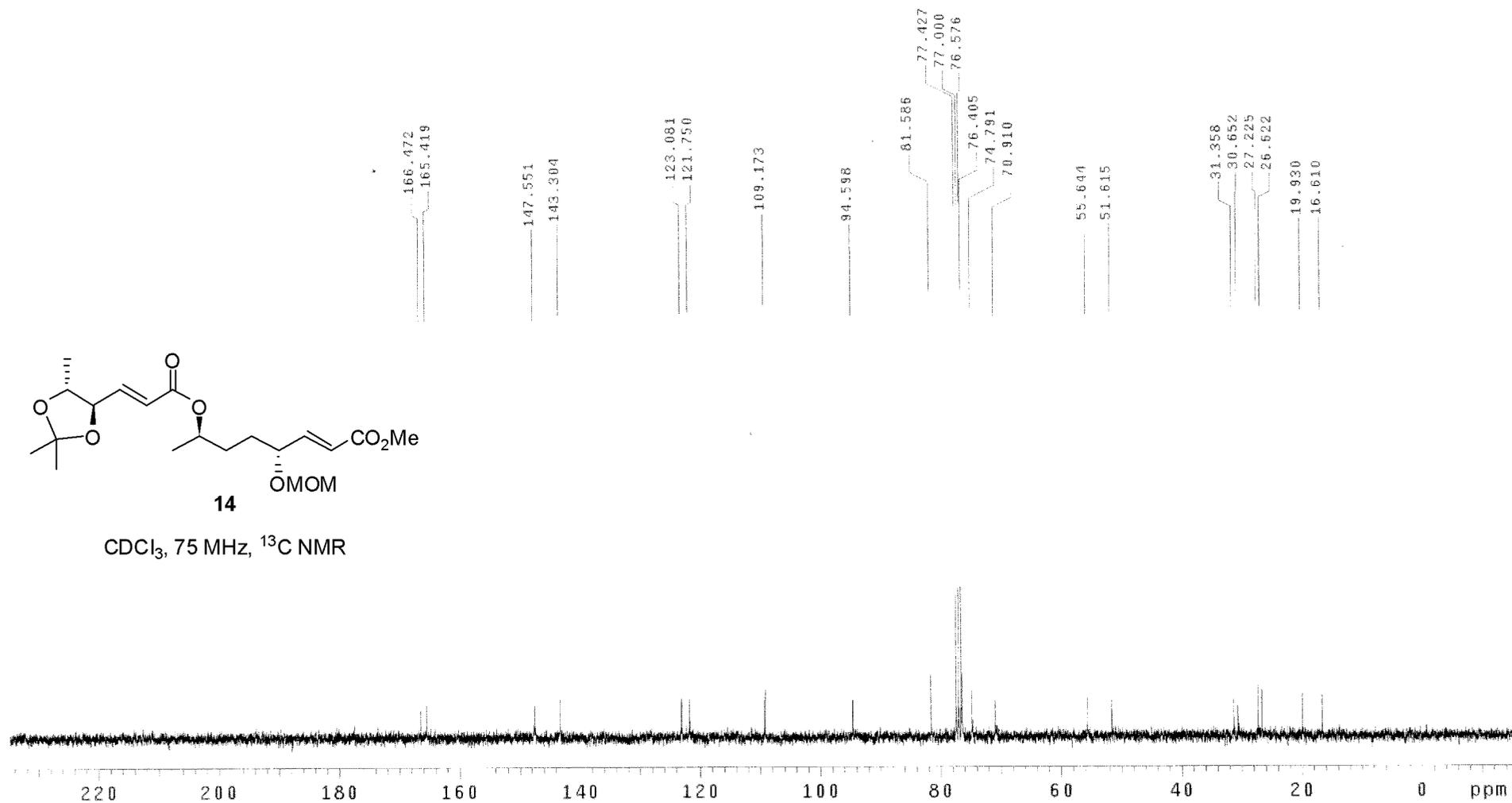


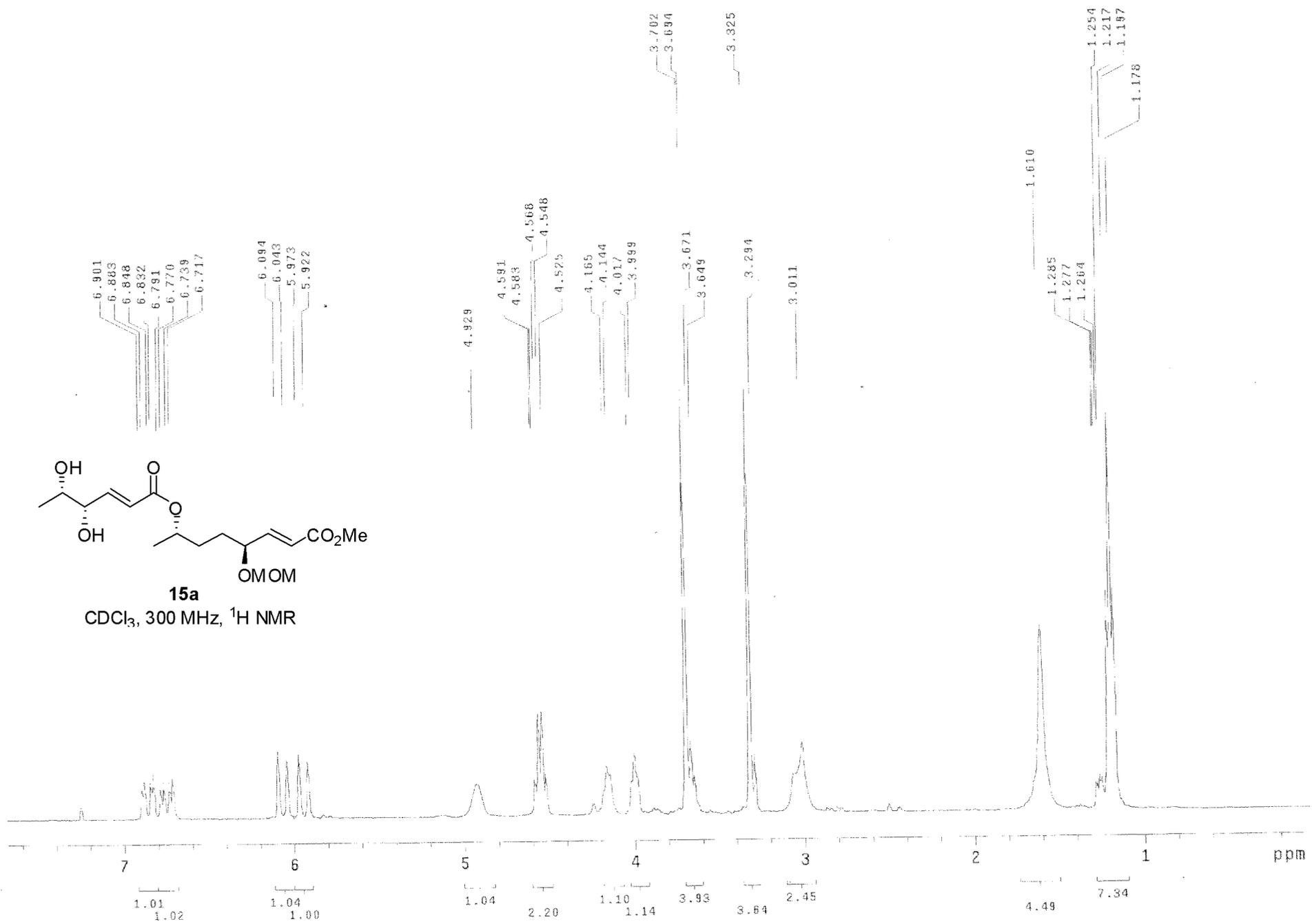
CDCl₃, 300 MHz, ¹H NMR

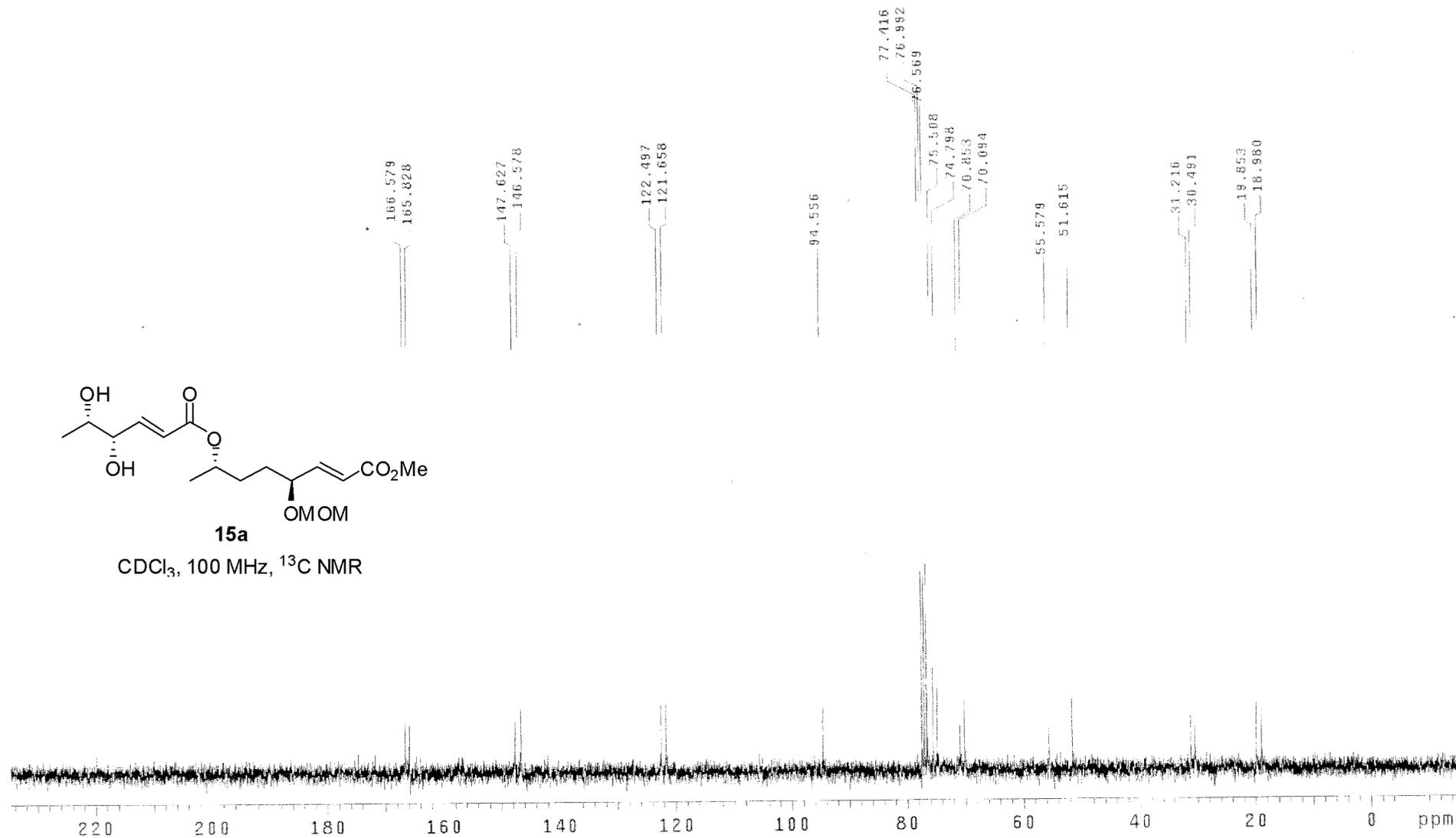




CDCl₃, 75 MHz, ¹³C NMR







7.270
6.934
6.922
6.895
6.883
6.824
6.808
6.785
6.769

6.144
6.106
6.012
5.973

5.001
4.986
4.974
4.632
4.615
4.589
4.572
4.227
4.210
4.192
4.075
4.062
4.050
3.748
3.723
3.716

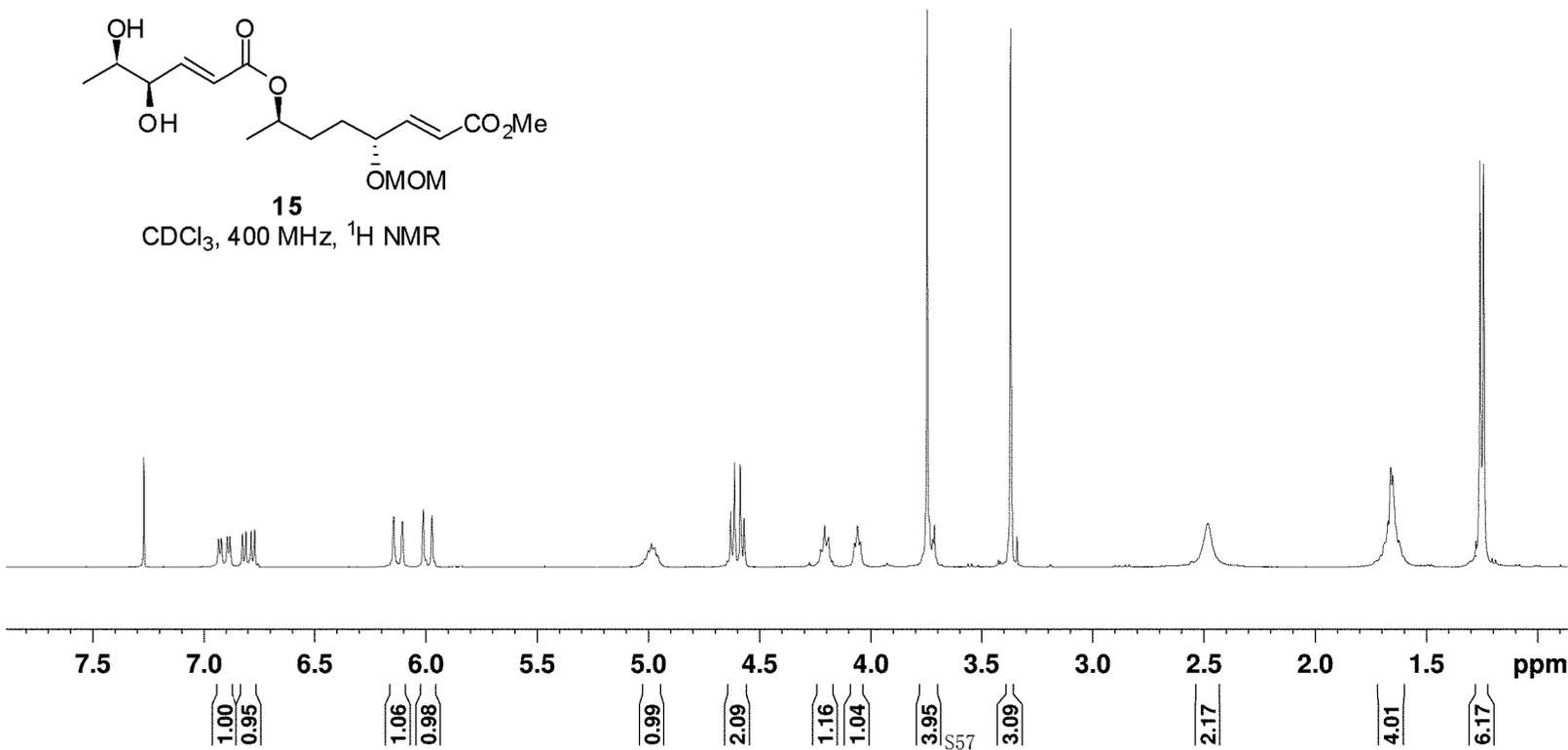
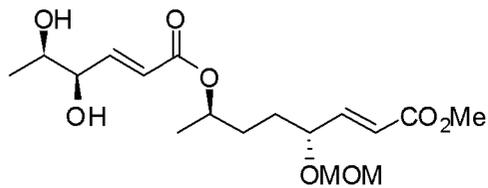
2.484

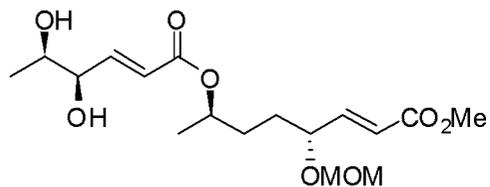
1.675
1.662
1.653

1.260
1.245

NAME laohan1127
EXFNO 10
PROCNO 1
Date_ 20081127
Time 2.26
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDCl3
NS 8
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 101
DW 60.800 usec
DE 6.50 usec
TE 292.5 K
D1 1.00000000 sec
TD0 1

===== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1300011 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00





15
CDCl₃, 100 MHz, ¹³C NMR

166.60
165.74

147.66
146.36

122.79
121.76

94.65

77.32
77.00
76.68
75.63
74.86
70.92
70.17

55.69
51.69

31.29
30.59

19.95
19.15

```

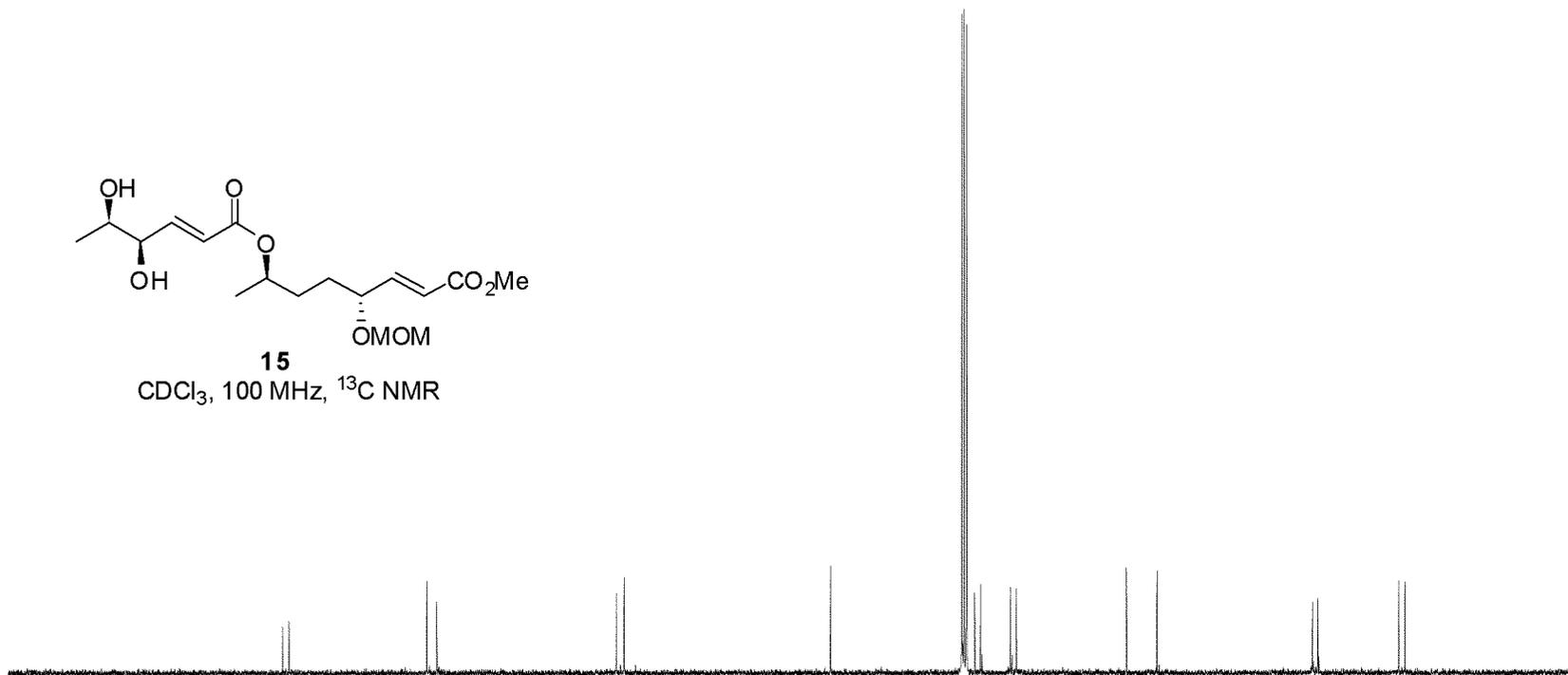
NAME      laohan1127
EXPNO     11
PROCNO    1
Date_     20081127
Time      2.57
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         512
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         912
DW         20.800 usec
DE         6.50 usec
TE         293.6 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         9.70 usec
PL1       -2.00 dB
PL1W      56.13311005 W
SFO1      100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      80.00 usec
PL2        -2.10 dB
PL12       13.90 dB
PL13       13.90 dB
PL2W      17.72104263 W
PL12W     0.44513249 W
PL13W     0.44513249 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6127707 MHz
WDW        EM
SSB         0
LB          1.00 Hz
GB          0
PC          1.40
  
```



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

7.270
6.937
6.925
6.909
6.897
6.886
6.870
6.855

6.143
6.104
6.009
5.970

4.986
4.975
4.668
4.646
4.640
4.628
4.610
4.593
4.239
4.227
4.085
4.073
4.060
3.778
3.763
3.748
3.732
3.382

1.666

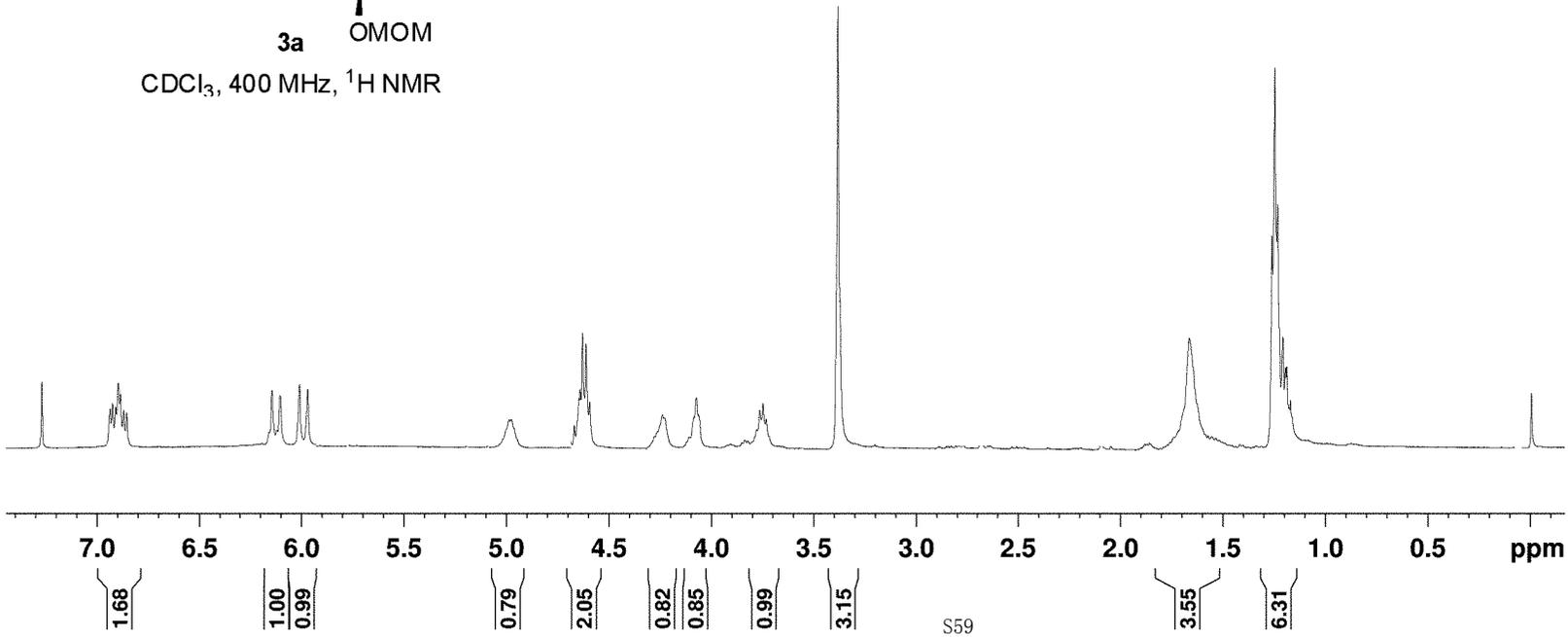
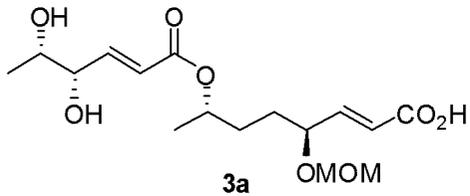
1.263
1.249
1.235

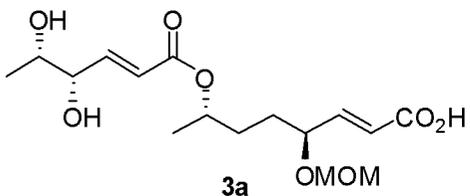
```

NAME      hanjjacid
EXPNO     10
PROCNO    1
Date_     20081223
Time      13.17
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         2
SWH       8223.685 Hz
FIDRES    0.125483 Hz
AQ         3.9846387 sec
RG         144
DW         60.800 usec
DE         6.50 usec
TE         291.2 K
D1         1.00000000 sec
TD0       1
  
```

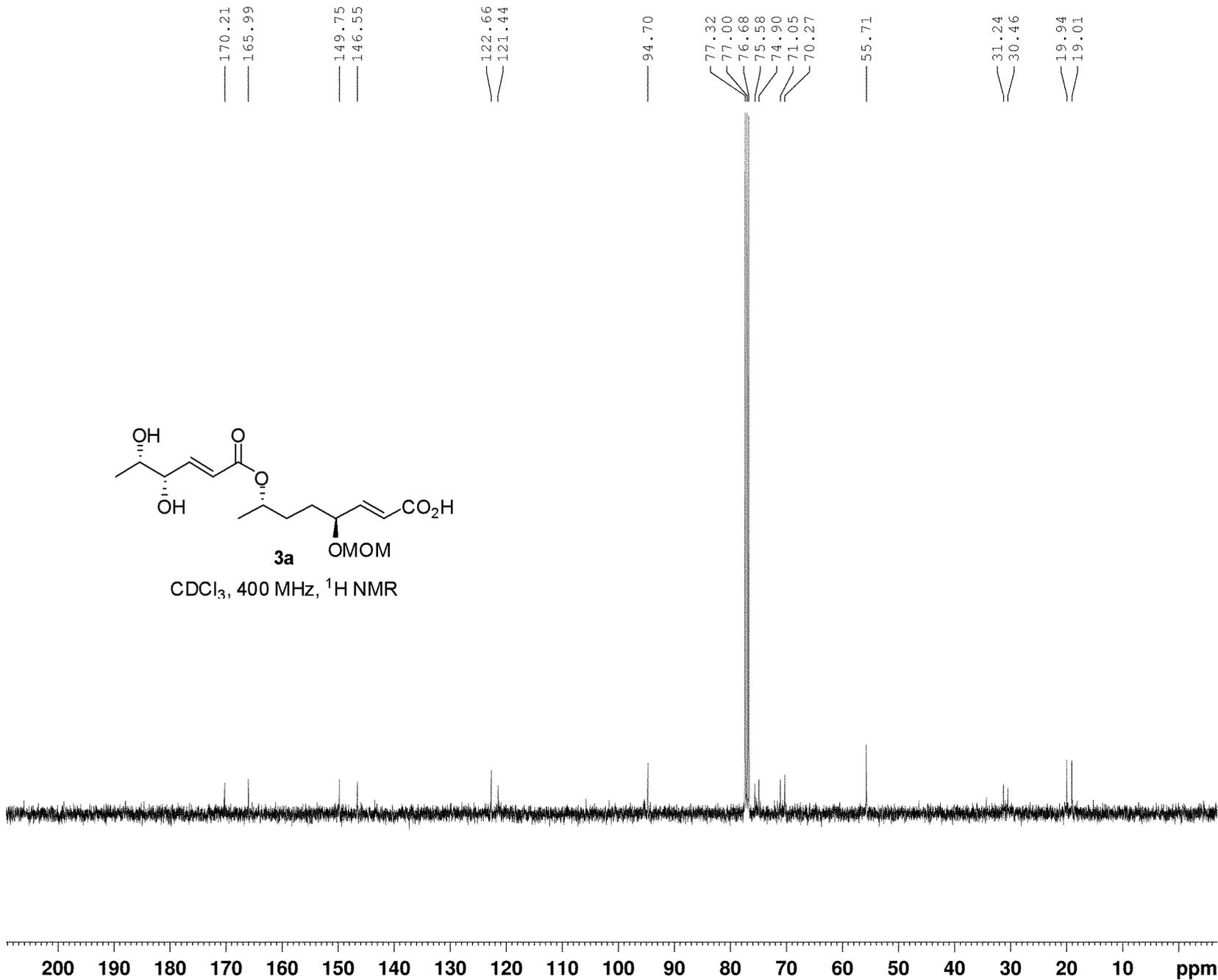
```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300011 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```





CDCl₃, 400 MHz, ¹H NMR



```

NAME      hanjjacid
EXPNO     11
PROCNO    1
Date_     20081223
Time      22.34
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpgg30
TD        65536
SOLVENT   CDCl3
NS        100
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        292.4 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1

```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz

```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       -2.00 dB
PL12      15.50 dB
PL13      15.50 dB
PL2W      18.19349861 W
PL12W     0.32353121 W
PL13W     0.32353121 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6127714 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40

```

6.933
6.920
6.901
6.894
6.886
6.881
6.862
6.847
6.139
6.136
6.100
6.097
6.005
5.965

4.979
4.968
4.643
4.636
4.625
4.607
4.589
4.234
4.222
4.083
4.080
4.068
4.055
3.757
3.741
3.725
3.378

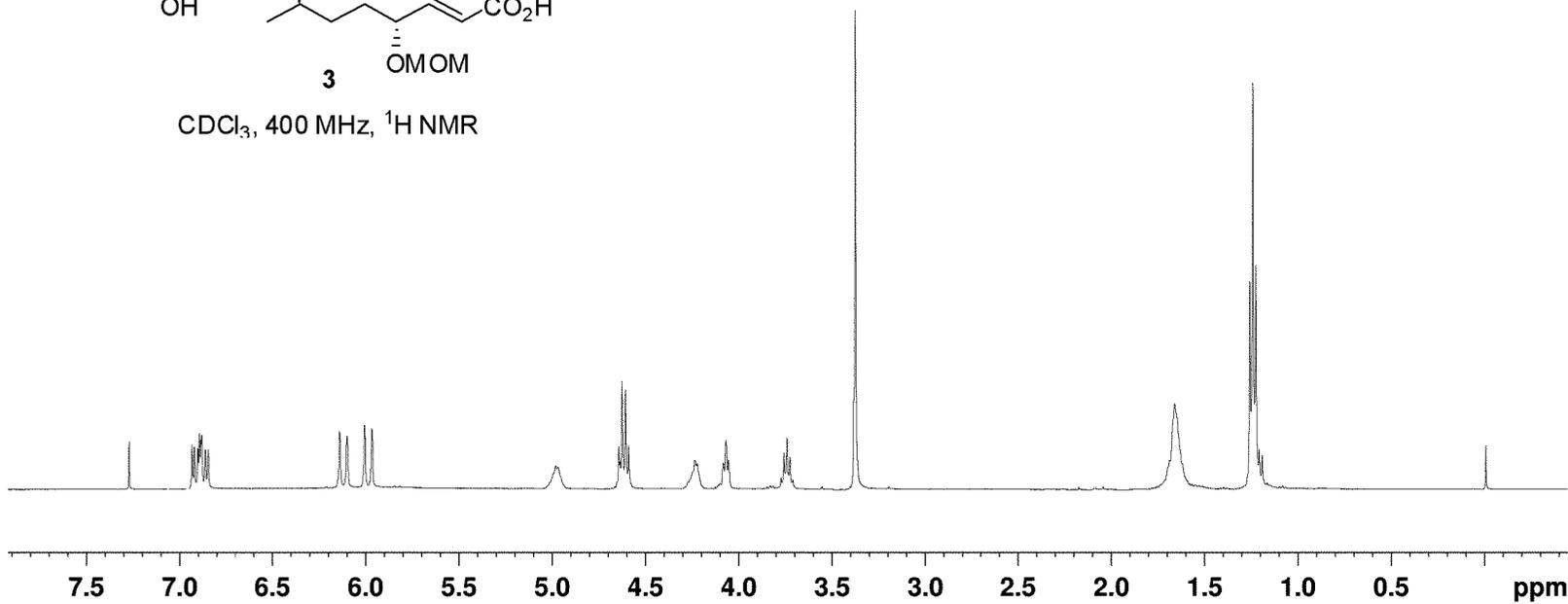
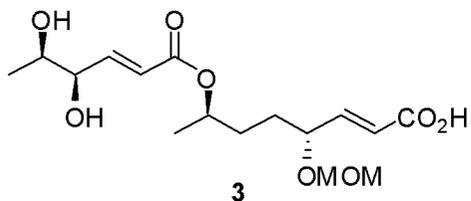
1.689
1.662
1.259
1.243
1.227

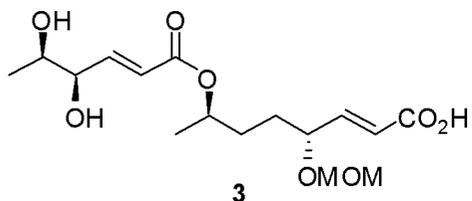
```

NAME      hanjjbacid
EXPNO     10
PROCNO    1
Date_     20081223
Time      13.27
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         114
DW         60.800 usec
DE         6.50 usec
TE         291.2 K
D1         1.00000000 sec
D10        1
  
```

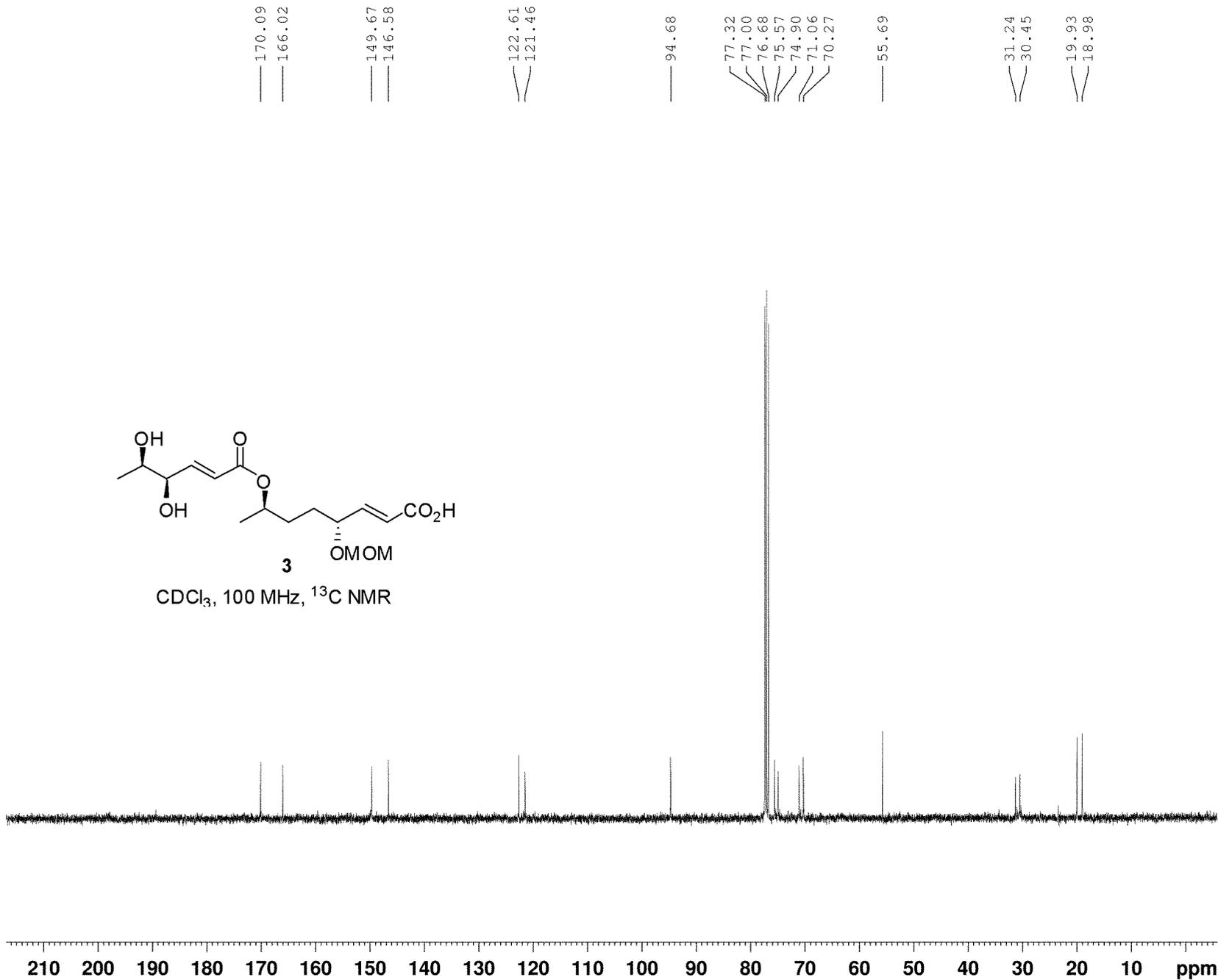
```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300008 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```





CDCl₃, 100 MHz, ¹³C NMR



NAME	hanjjbacid
EXPNO	11
PROCNO	1
Date_	20081223
Time	23.04
INSTRUM	spect
PROBHD	5 mm PABBO BB-
PULPROG	zgpg30
TD	65536
SOLVENT	CDCl3
NS	150
DS	4
SWH	24038.461 Hz
FIDRES	0.366798 Hz
AQ	1.3631988 sec
RG	2050
DW	20.800 usec
DE	6.50 usec
TE	292.1 K
D1	2.00000000 sec
D11	0.03000000 sec
TD0	1

===== CHANNEL f1 =====

NUC1	13C
P1	9.40 usec
PL1	-2.00 dB
PL1W	57.32743073 W
SFO1	100.6228298 MHz

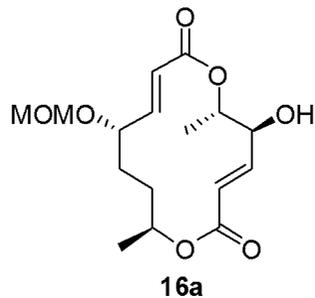
===== CHANNEL f2 =====

CPDPRG2	waltz16
NUC2	1H
PCPD2	90.00 usec
PL2	-2.00 dB
PL12	15.50 dB
PL13	15.50 dB
PL2W	18.19349861 W
PL12W	0.32353121 W
PL13W	0.32353121 W
SFO2	400.1316005 MHz
SI	32768
SF	100.6127722 MHz
WDW	EM
SSB	0
LB	1.00 Hz
GB	0
PC	1.40

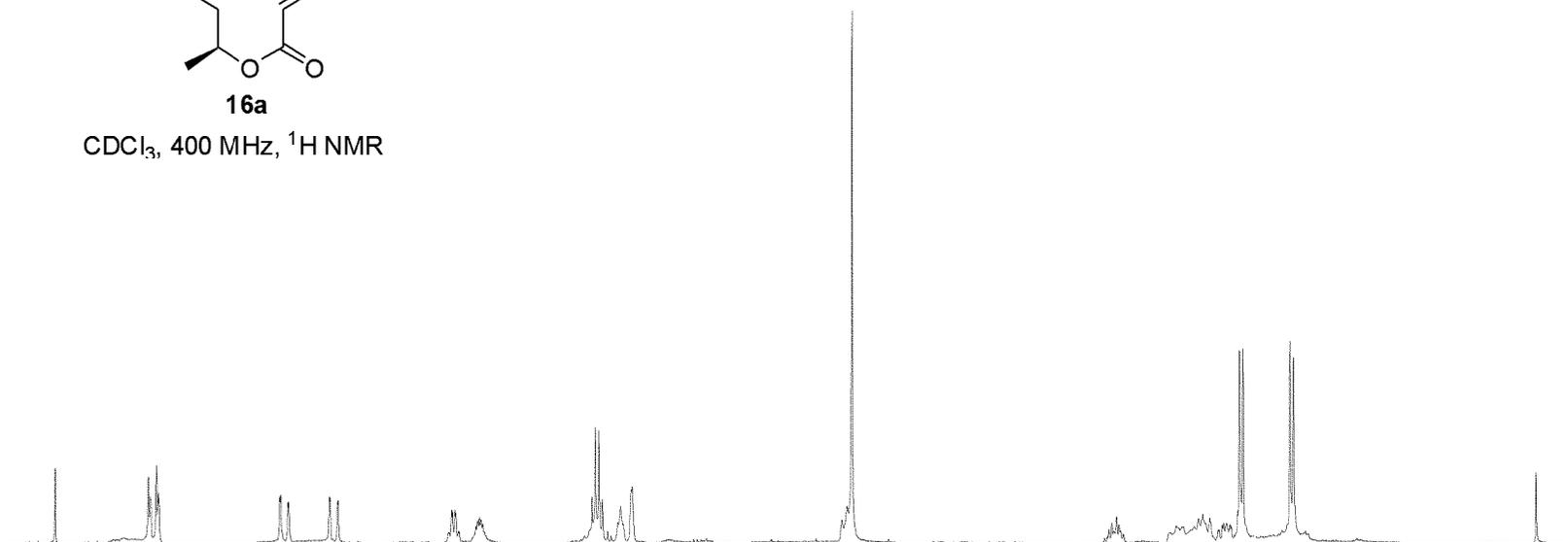
6.814
6.806
6.803
6.775
6.766
6.762
6.166
6.162
6.127
6.122
5.923
5.920
5.883
5.880
5.339
5.335
5.323
5.318
5.306
5.301
5.289
5.285
5.203
5.195
5.187
5.178
5.170
4.634
4.617
4.600
4.583
4.506
4.493
4.480
4.442
4.436

3.358

2.092
2.079
2.055
2.043
1.766
1.735
1.730
1.677
1.656
1.635
1.621
1.600
1.561
1.554
1.539
1.532
1.517
1.511
1.502
1.495
1.464
1.455
1.438
1.416
1.406
1.207
1.191



CDCl₃, 400 MHz, ¹H NMR



7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

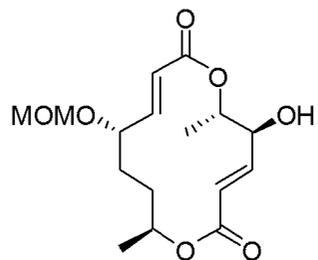
1.73 1.00 0.94 0.88 0.93 2.05 0.78 0.84 3.10 1.05 3.65 2.76 3.21

```

NAME      hanjjester
EXPNO     10
PROCNO    1
Date_     20081223
Time      13.21
INSTRUM   spect
PROBHD    5 mm FABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         8
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         161
DW         60.800 usec
DE         6.50 usec
TE         291.2 K
D1         1.00000000 sec
TDO        1
  
```

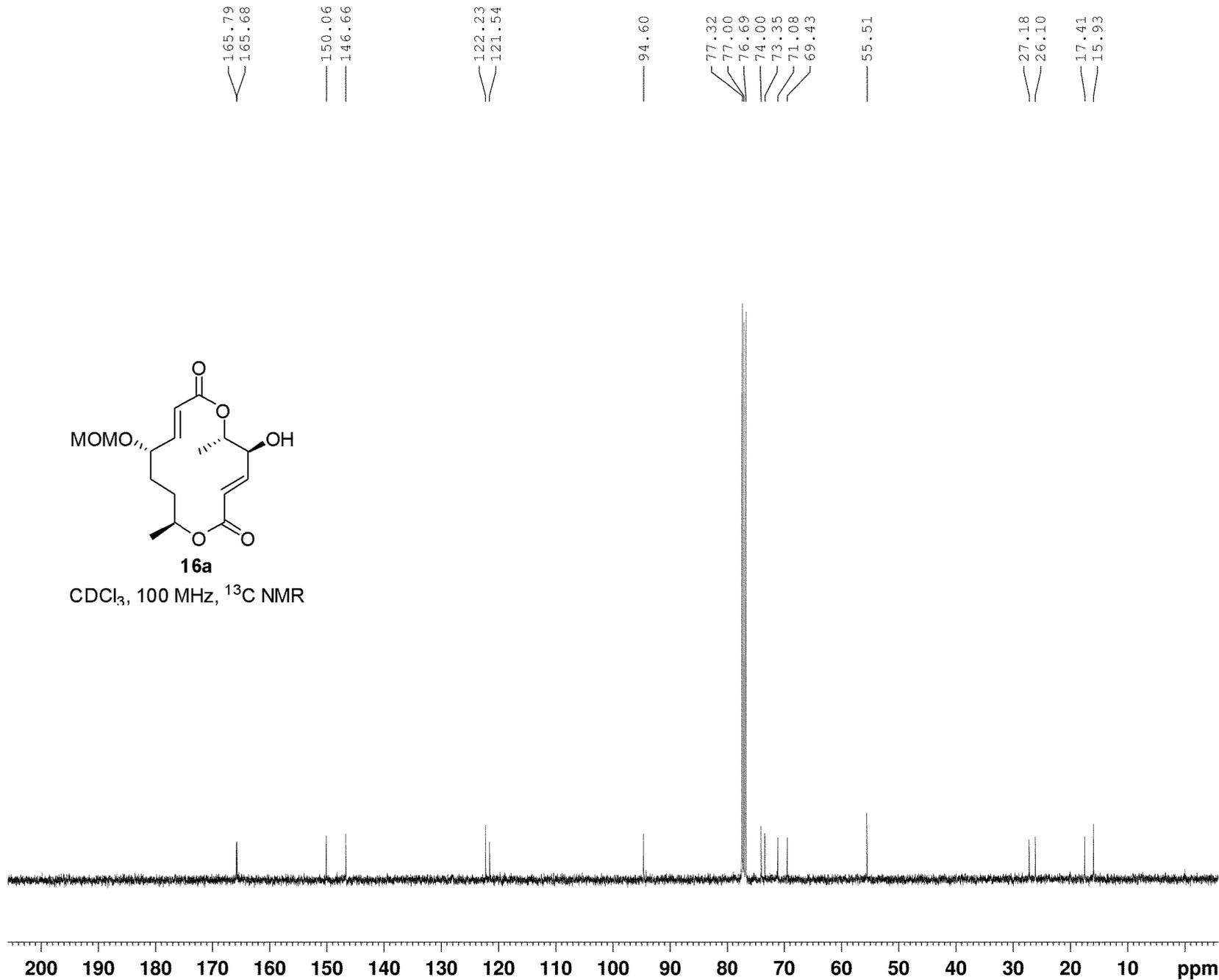
```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SFO1      400.1324710 MHz
SI         32768
SF         400.1300008 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



16a

CDCl₃, 100 MHz, ¹³C NMR



165.79
165.68

150.06
146.66

122.23
121.54

94.60

77.32
77.00
76.69
74.00
73.35
71.08
69.43

55.51

27.18
26.10

17.41
15.93

```

NAME      hanjtester
EXPNO     11
PROCNO    1
Date_     20081223
Time      22.51
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         200
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         292.0 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1      100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2        -2.00 dB
PL12       15.50 dB
PL13       15.50 dB
PL2W       18.19349861 W
PL12W      0.32353121 W
PL13W      0.32353121 W
SFO2      400.1316005 MHz
S1         32768
SF         100.6127714 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

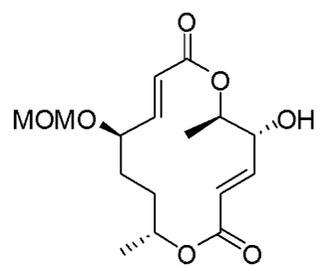
6.814
6.811
6.803
6.801
6.774
6.772
6.764
6.761
6.176
6.172
6.137
6.132
5.923
5.919
5.883
5.879
5.336
5.331
5.319
5.314
5.303
5.297
5.286
5.281
5.199
5.191
5.183
5.175
5.166
4.632
4.615
4.609
4.597
4.592
4.580
4.492
4.447
4.442
4.438
4.435
4.431
3.356
2.091
2.077
2.068
2.064
2.052
2.042
2.034
1.761
1.751
1.746
1.738
1.730
1.724
1.659
1.654
1.649
1.639
1.633
1.628
1.619
1.598
1.558
1.552
1.537
1.530
1.453
1.437
1.206
1.190

```

NAME      hanjjbester
EXPNO     10
PROCNO    1
Date_     20081223
Time      13.31
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         4
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         114
DW         60.800 usec
DE         6.50 usec
TE         291.1 K
D1         1.00000000 sec
TD0        1
  
```

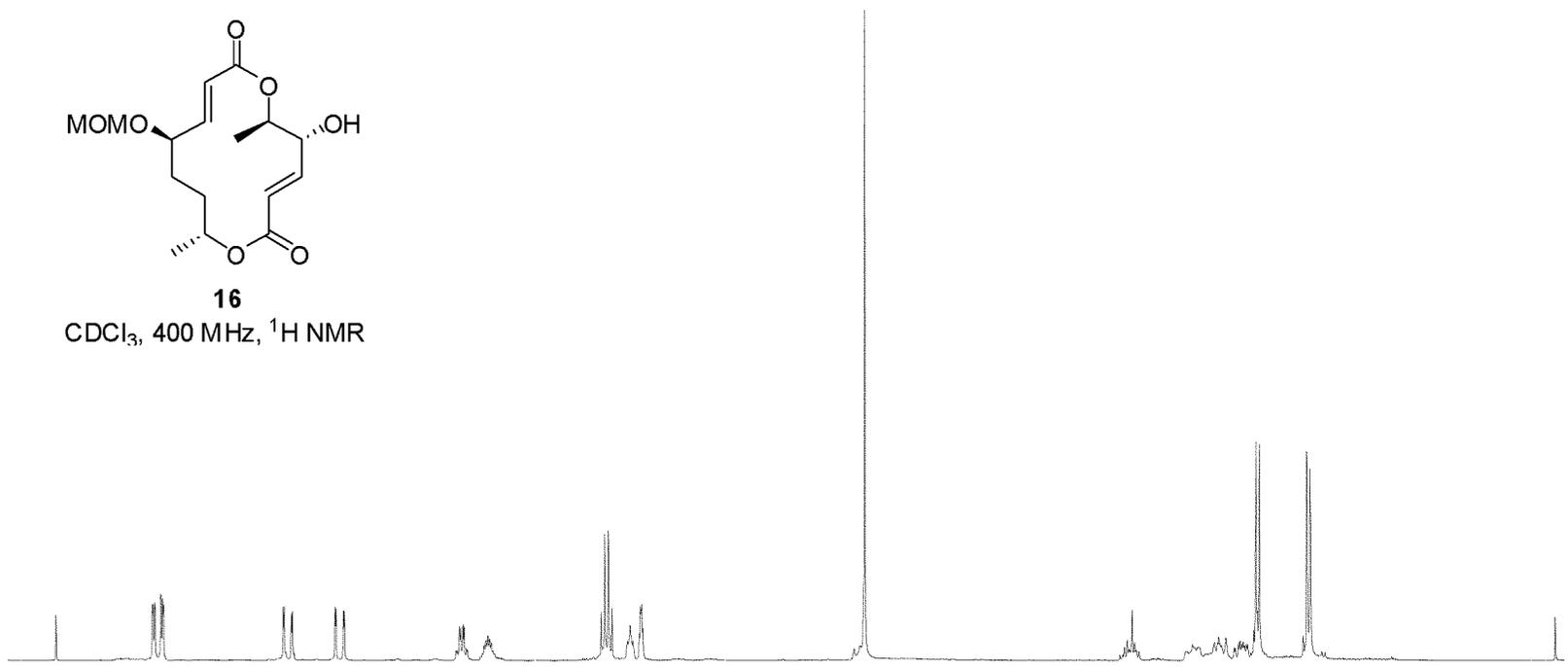
```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1299963 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



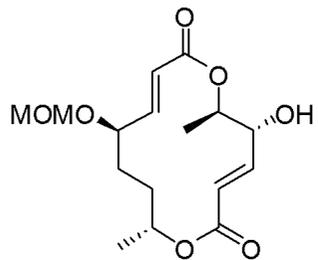
16

CDCl₃, 400 MHz, ¹H NMR



7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

1.74 1.00 0.90 0.91 0.96 2.07 0.83 0.90 3.09 0.97 0.86 1.23 0.88 2.77 2.82



16
CDCl₃, 100 MHz, ¹³C NMR

165.82
165.68

149.98
146.76

122.19
121.53

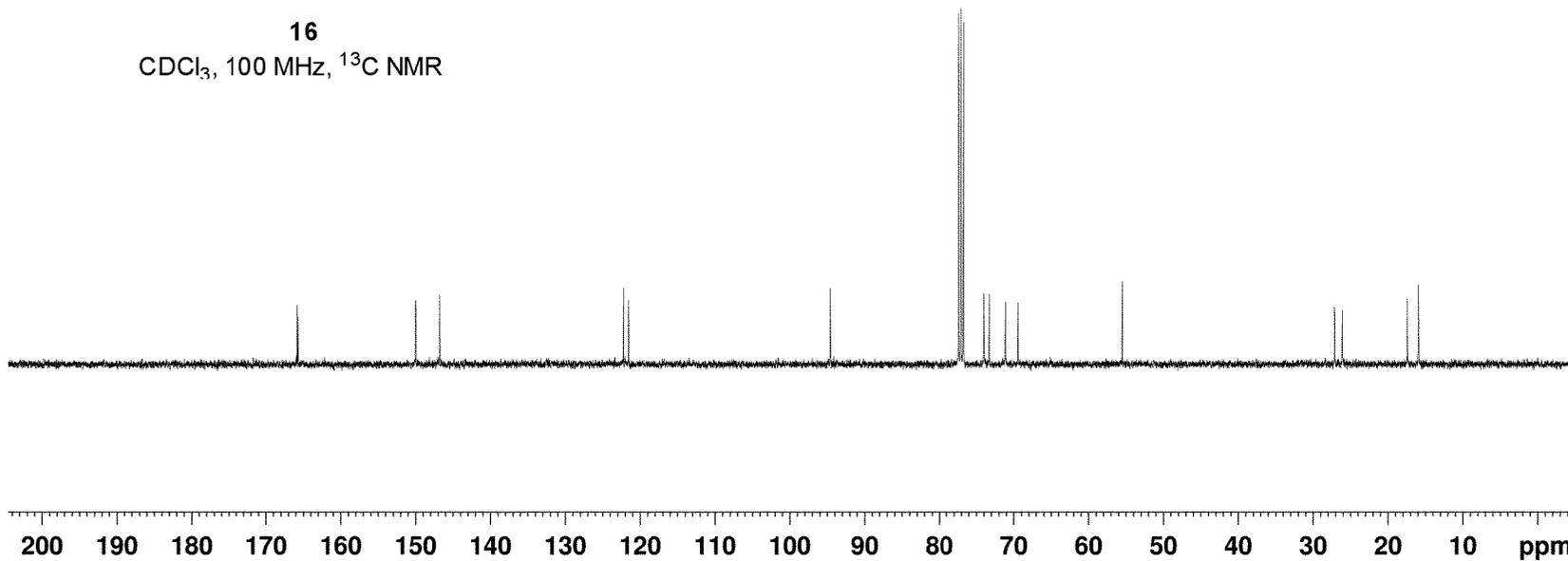
94.57

77.32
77.00
76.68
73.96
73.24
71.07
69.39

55.47

27.09
26.06

17.38
15.91



```

NAME      hanjbbester
EXPNO     11
PROCNO    1
Date_     20081223
Time      23.23
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        100
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        2050
DW        20.800 usec
DE        6.50 usec
TE        292.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.40 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     90.00 usec
PL2       -2.00 dB
PL12      15.50 dB
PL13      15.50 dB
PL2W      18.19349861 W
PL12W     0.32353121 W
PL13W     0.32353121 W
SFO2      400.1316005 MHz
S1        32768
SF        100.6127736 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

7.334
7.294
7.270
7.046
7.035
7.006
6.996
6.504
6.464
6.156
6.152
6.117
6.113
5.347
5.329
5.311
5.293
5.068
5.062
5.052
5.046
5.037
5.031
4.678
4.661
4.647
4.636
4.629
4.619
4.454
4.445
4.435

3.384

1.956
1.936
1.920
1.900
1.891
1.873
1.857
1.837
1.831
1.821
1.815
1.800
1.610
1.595
1.562
1.544
1.288
1.272

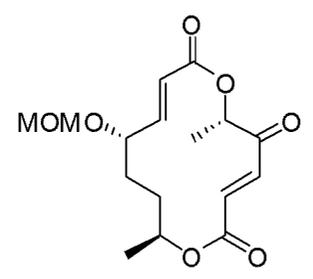
0.004

```

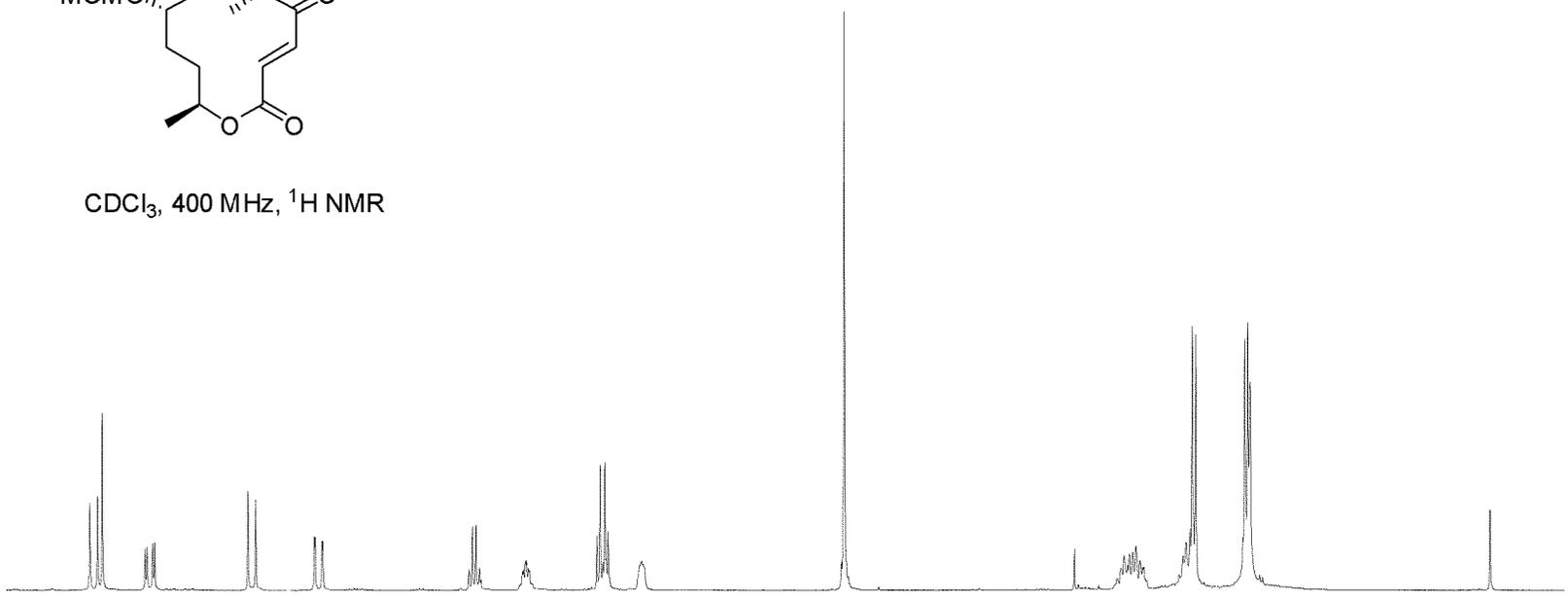
NAME      hanjj081227keto
EXPNO     10
PROCNO    1
Date_     20081227
Time      21.13
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         32
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         322
DW         60.800 usec
DE         6.50 usec
TE         291.9 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1       1H
P1         14.60 usec
PL1        0.00 dB
PL1W       11.47932053 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300012 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



CDCl₃, 400 MHz, ¹H NMR



7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

1.00 0.98 0.97 0.94 0.99 0.98 2.34 0.97 3.35 3.26 1.08 3.35 4.00


```

NAME      hanjj0114bketo
EXPNO     10
PROCNO    1
Date_     20090115
Time      18.42
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         16
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         181
DW         60.800 usec
DE         6.50 usec
TE         291.7 K
D1         1.00000000 sec
TD0        1

```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SFO1      400.1324710 MHz
SI        32768
SF        400.1300012 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

```

7.331
7.290
7.270
7.043
7.032
7.003
6.993

6.499
6.459

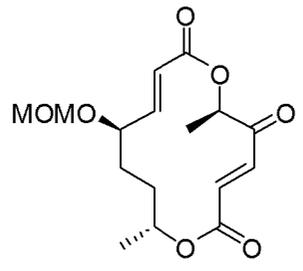
6.152
6.148
6.113
6.109

5.343
5.324
5.306
5.288
5.064
5.058
5.048
5.043
5.033
5.027

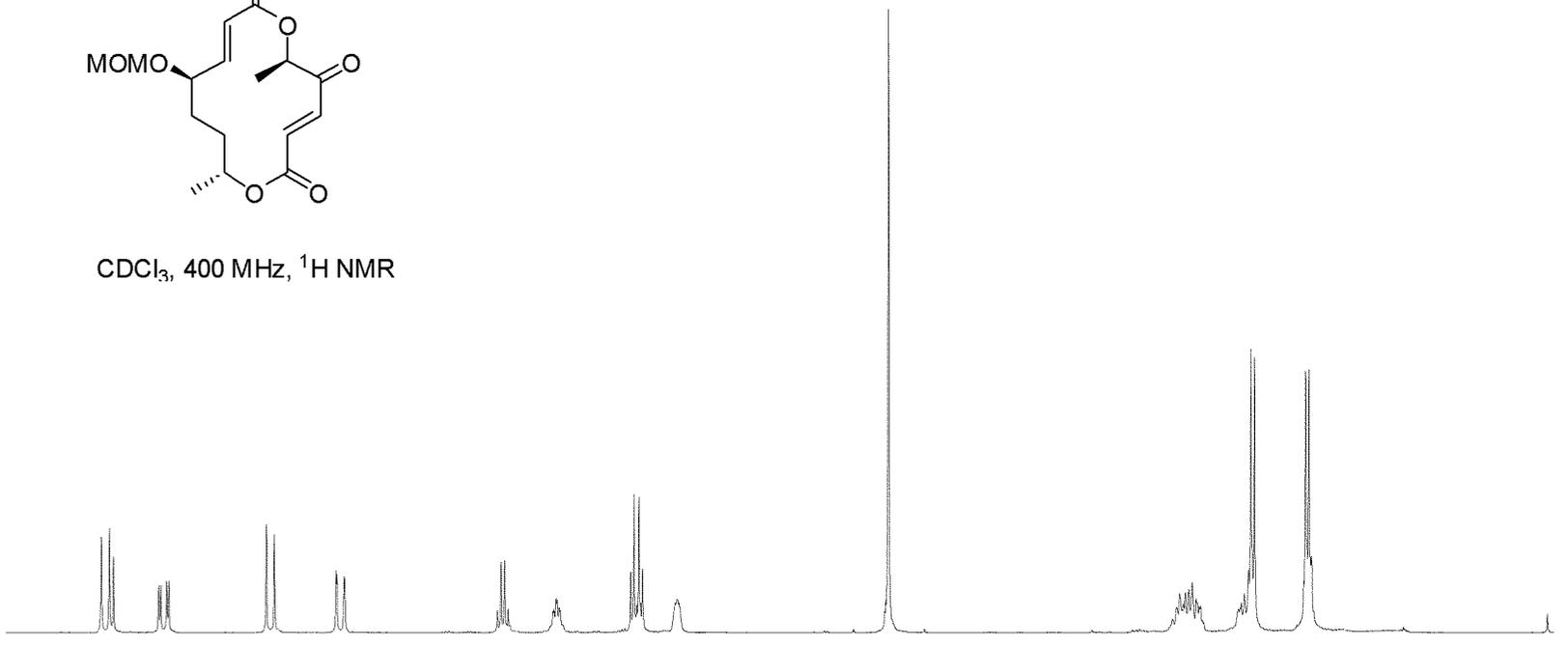
4.674
4.657
4.632
4.615
4.451
4.442
4.432

3.380

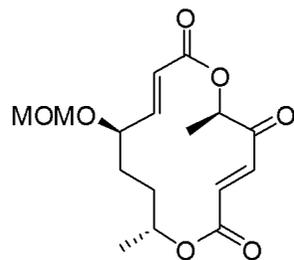
1.952
1.947
1.932
1.916
1.904
1.897
1.887
1.870
1.853
1.833
1.828
1.818
1.812
1.621
1.613
1.607
1.592
1.570
1.559
1.540
1.284
1.268



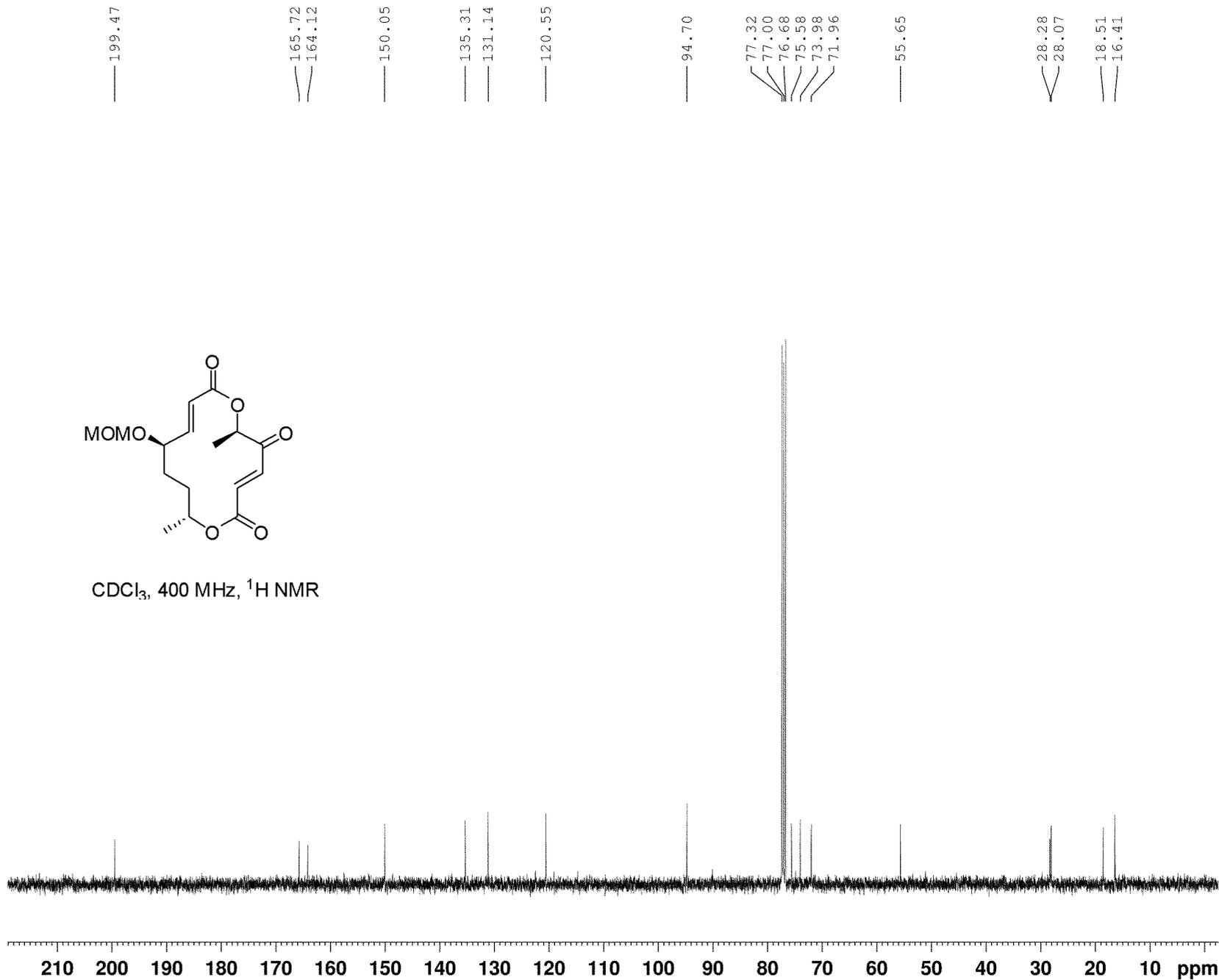
CDCl₃, 400 MHz, ¹H NMR



1.04
1.03
1.03
1.00
1.01
1.06
2.35
1.04
3.26
3.38
1.02
3.29
3.40



CDCl₃, 400 MHz, ¹H NMR



199.47
165.72
164.12
150.05
135.31
131.14
120.55
94.70
77.32
77.00
76.68
75.58
73.98
71.96
55.65
28.28
28.07
18.51
16.41

```

NAME      hanjj0114bketo
EXPNO     11
PROCNO    1
Date_     20090115
Time      18.49
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         80
DS         4
SWH        24038.461 Hz
FIDRES     0.366798 Hz
AQ         1.3631988 sec
RG         50.8
DW         20.800 usec
DE         6.50 usec
TE         292.5 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
PL1        -2.00 dB
PL1W       57.32743073 W
SFO1      100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2      90.00 usec
PL2        -2.00 dB
PL12       15.50 dB
PL13       15.50 dB
PL2W       18.19349861 W
PL12W      0.32353121 W
PL13W      0.32353121 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6127710 MHz
WDW        EM
SSB         0
LB         1.00 Hz
GB         0
PC         1.40
  
```

7.388
7.347
7.264
7.153
7.145
7.114
7.105

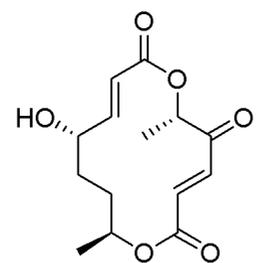
6.489
6.449
6.165
6.161
6.125
6.121

5.360
5.342
5.324
5.305

5.038
5.032
5.021
5.016
5.005

4.999
4.548
4.536

2.034
2.028
2.017
2.011
2.000
1.994
1.983
1.977
1.896
1.890
1.880
1.875
1.858
1.852
1.843
1.837
1.826
1.820
1.779
1.763
1.757
1.741
1.727
1.706
1.691
1.641
1.624
1.607
1.586
1.567
1.549
1.297
1.281



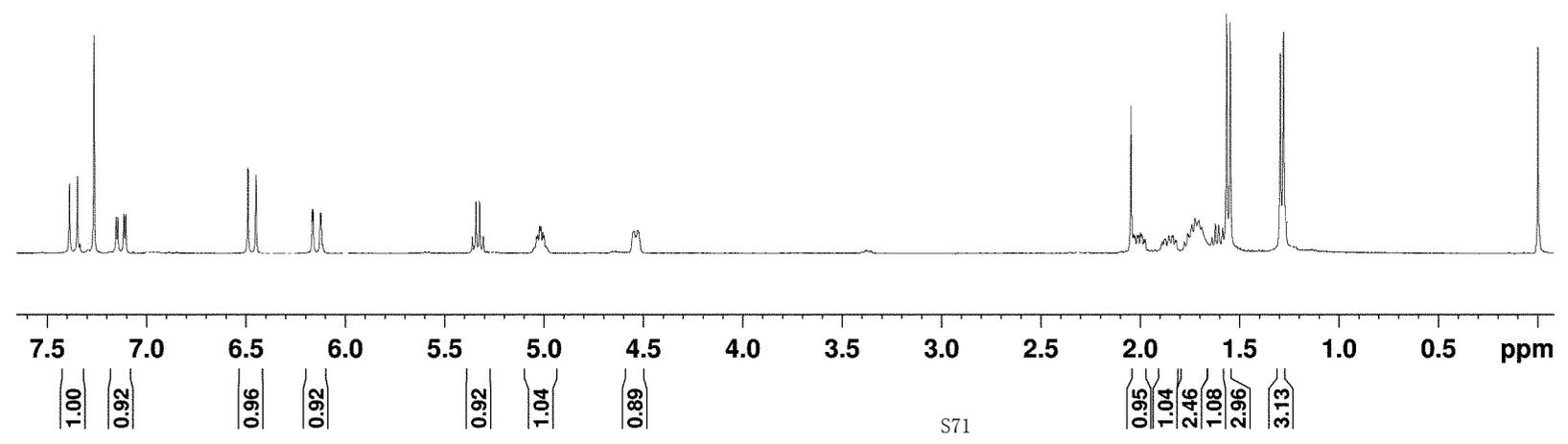
1a

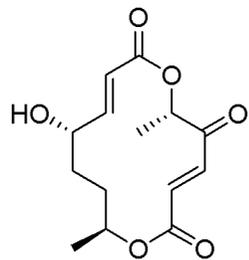
CDCl₃, 400MHz ¹H NMR

```

NAME      hanjj1229atm
EXPNO     10
PROCNO    1
Date_     20081229
Time      23.00
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDC13
NS         32
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         575
DW         60.800 usec
DE         6.50 usec
TE         292.0 K
D1         1.00000000 sec
TD0        1

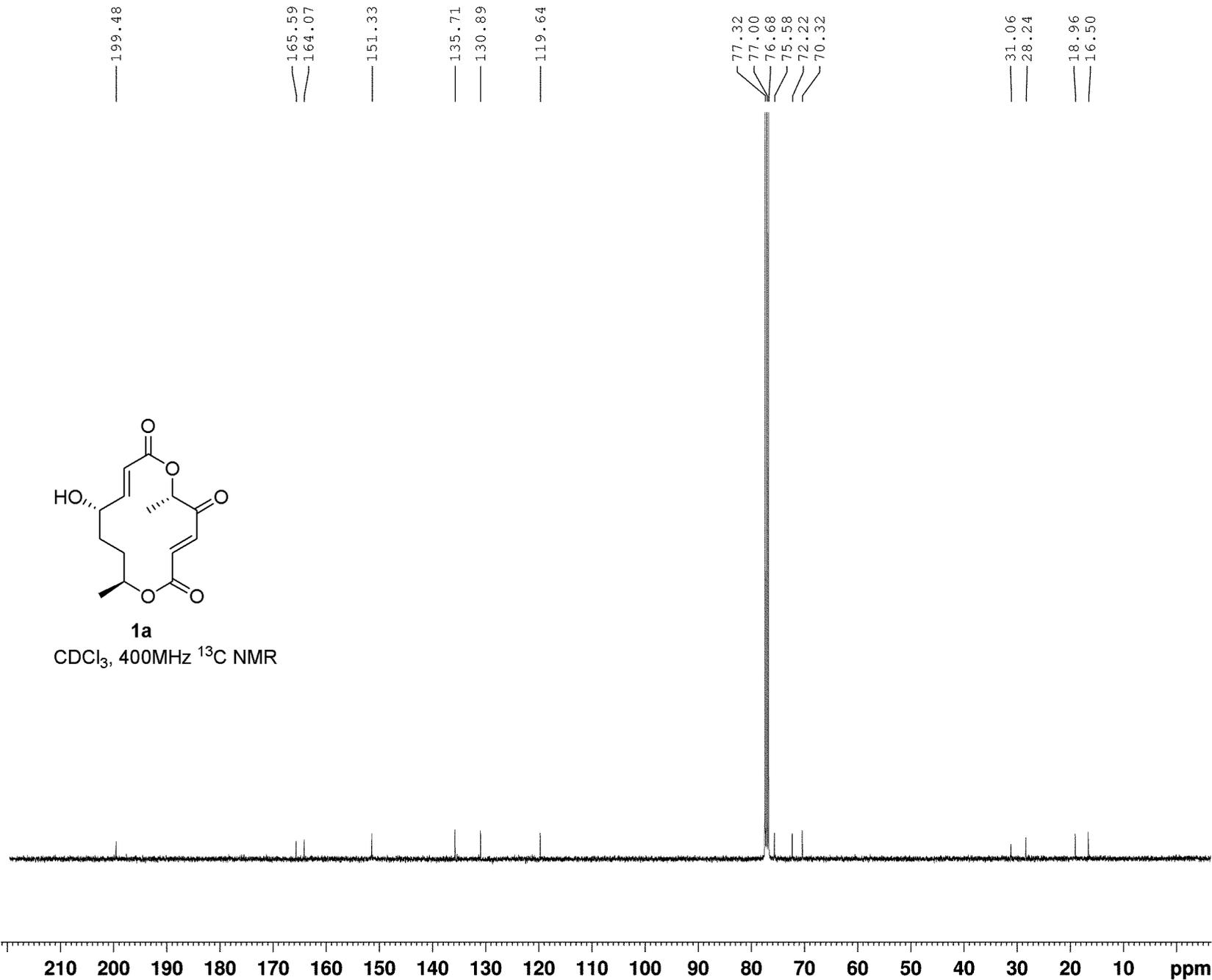
===== CHANNEL f1 =====
NUC1       1H
P1         14.60 usec
PL1        0.00 dB
PL1W       11.47932053 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300035 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```





1a

CDCl₃, 400MHz ¹³C NMR



199.48

165.59
164.07

151.33

135.71

130.89

119.64

77.32
77.00
76.68
75.58
72.22
70.32

31.06
28.24

18.96
16.50

```

NAME      hanjj1229atm
EXPNO     11
PROCNO    1
Date_     20081230
Time      0.58
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         2048
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         292.4 K
D1         2.00000000 sec
D11        0.03000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waitz16
NUC2       1H
PCPD2     90.00 usec
PL2       -2.00 dB
PL12      15.50 dB
PL13      15.50 dB
PL2W      18.19349861 W
PL12W     0.32353121 W
PL13W     0.32353121 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6127700 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

7.392
7.351
7.224
7.216
7.185
7.177

6.406
6.366

5.959
5.955
5.920
5.915

5.203
5.193
5.186
5.167
5.149
5.131

4.937
4.931
4.921
4.915
4.905

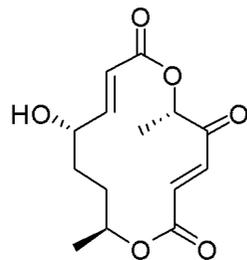
4.899
4.364
4.357

3.328

2.504
2.500
2.496
1.908
1.896
1.891
1.876
1.865
1.713
1.699
1.681
1.665
1.660
1.591
1.575
1.558
1.543
1.536
1.502
1.484
1.212
1.196

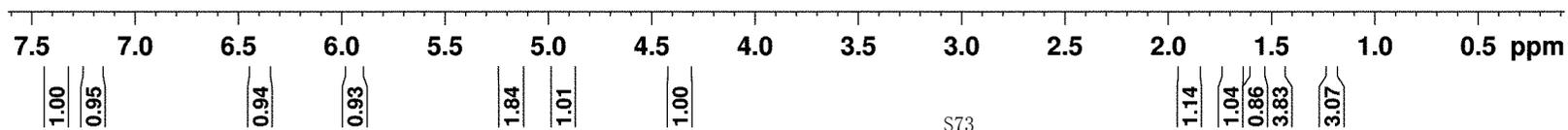
NAME hanjjatm
EXPNO 10
PROCNO 1
Date_ 20090326
Time 2.48
INSTRUM spect
PROBHD 5 mm PABBO BB-
PULPROG zg30
TD 65536
SOLVENT CDC13
NS 32
DS 2
SWH 8223.685 Hz
FIDRES 0.125483 Hz
AQ 3.9846387 sec
RG 362
DW 60.800 usec
DE 6.50 usec
TE 292.7 K
D1 1.00000000 sec
TD0 1

==== CHANNEL f1 =====
NUC1 1H
P1 14.70 usec
PL1 -1.00 dB
PL1W 13.75590801 W
SFO1 400.1324710 MHz
SI 32768
SF 400.1318992 MHz
WDW EM
SSB 0
LB 0.30 Hz
GB 0
PC 1.00



1a

DMSO-d₆, 400 MHz, ¹H NMR



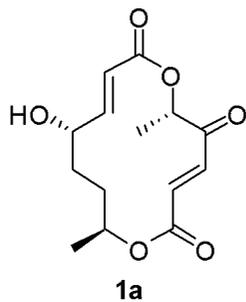
7.376
7.336
7.210
7.202
7.171
7.163

6.388
6.347

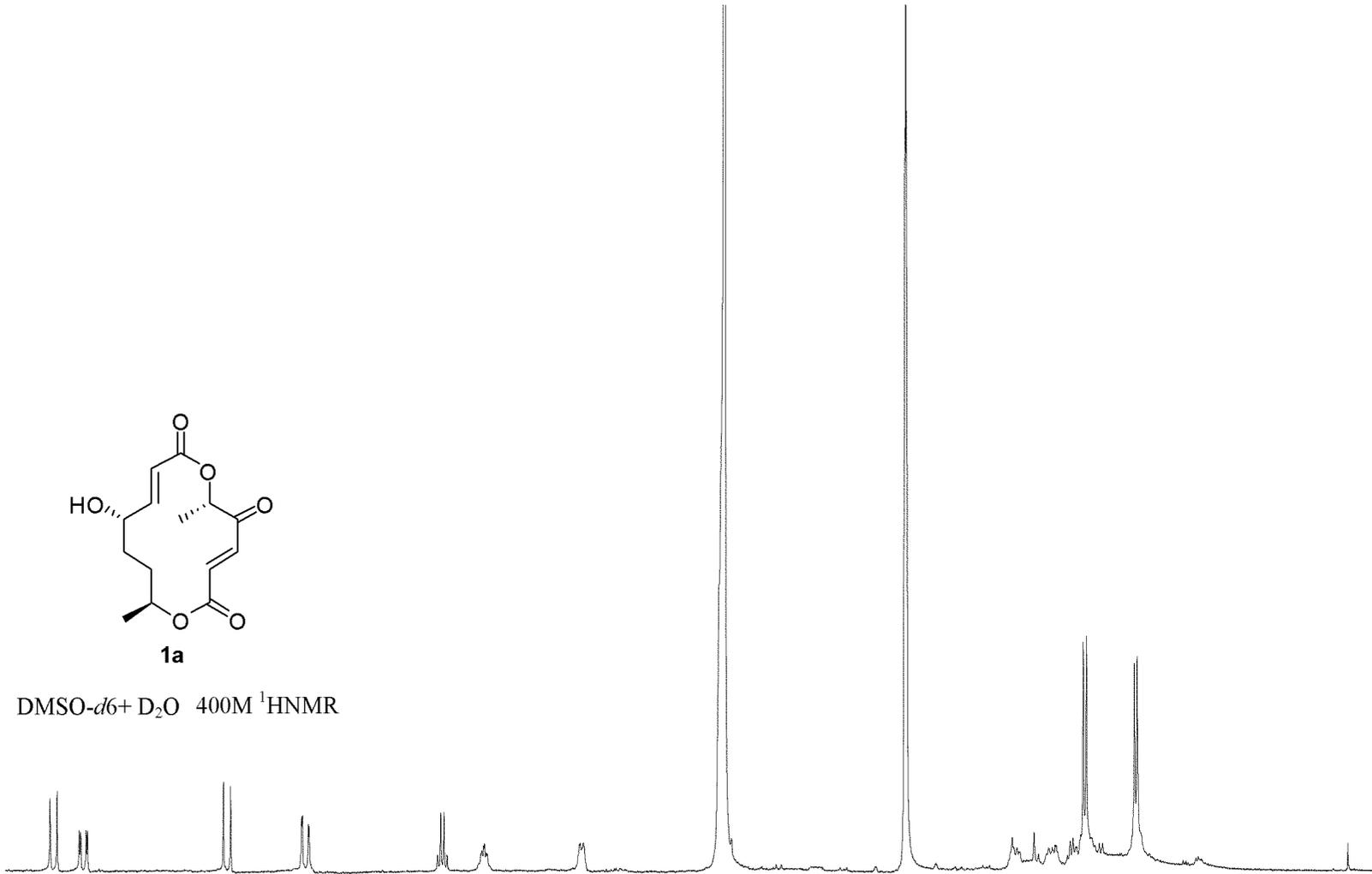
5.943
5.939
5.904
5.899

5.167
5.149
5.131
5.113
4.916
4.906
4.900
4.890
4.884
4.354
4.337

1.895
1.867
1.856
1.688
1.665
1.650
1.644
1.581
1.565
1.548
1.527
1.490
1.472
1.198
1.182



DMSO-*d*₆+ D₂O 400M ¹HNMR



1.00 0.98 0.89 1.09 1.00 1.07 1.08 1.45 0.88 1.73 1.36 3.91 2.92

```

NAME      hanjj0944atm
EXPNO     10
PROCNO    1
Date_     20090404
Time      13.50
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   DMSO
NS         32
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         362
DW         60.800 usec
DE         6.50 usec
TE         293.2 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.70 usec
PL1        -1.00 dB
PL1W       13.75590801 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1299973 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

```

— 200.23
 — 165.81
 — 164.01
 — 154.46
 — 136.84
 — 129.89
 — 118.14

— 75.37
 — 72.01
 — 68.68

— 30.47
 — 27.65
 — 18.61
 — 15.96

```

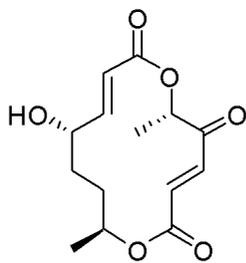
NAME      hanjjbtm025
EXPNO     11
PROCNO    1
Date_     20090325
Time      22.09
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   CDCl3
NS        1024
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        912
DW        20.800 usec
DE        6.50 usec
TE        295.9 K
D1        2.0000000 sec
D11       0.0300000 sec
TD0       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.70 usec
PL1       -2.00 dB
PL1W      56.13311005 W
SFO1      100.6228298 MHz
  
```

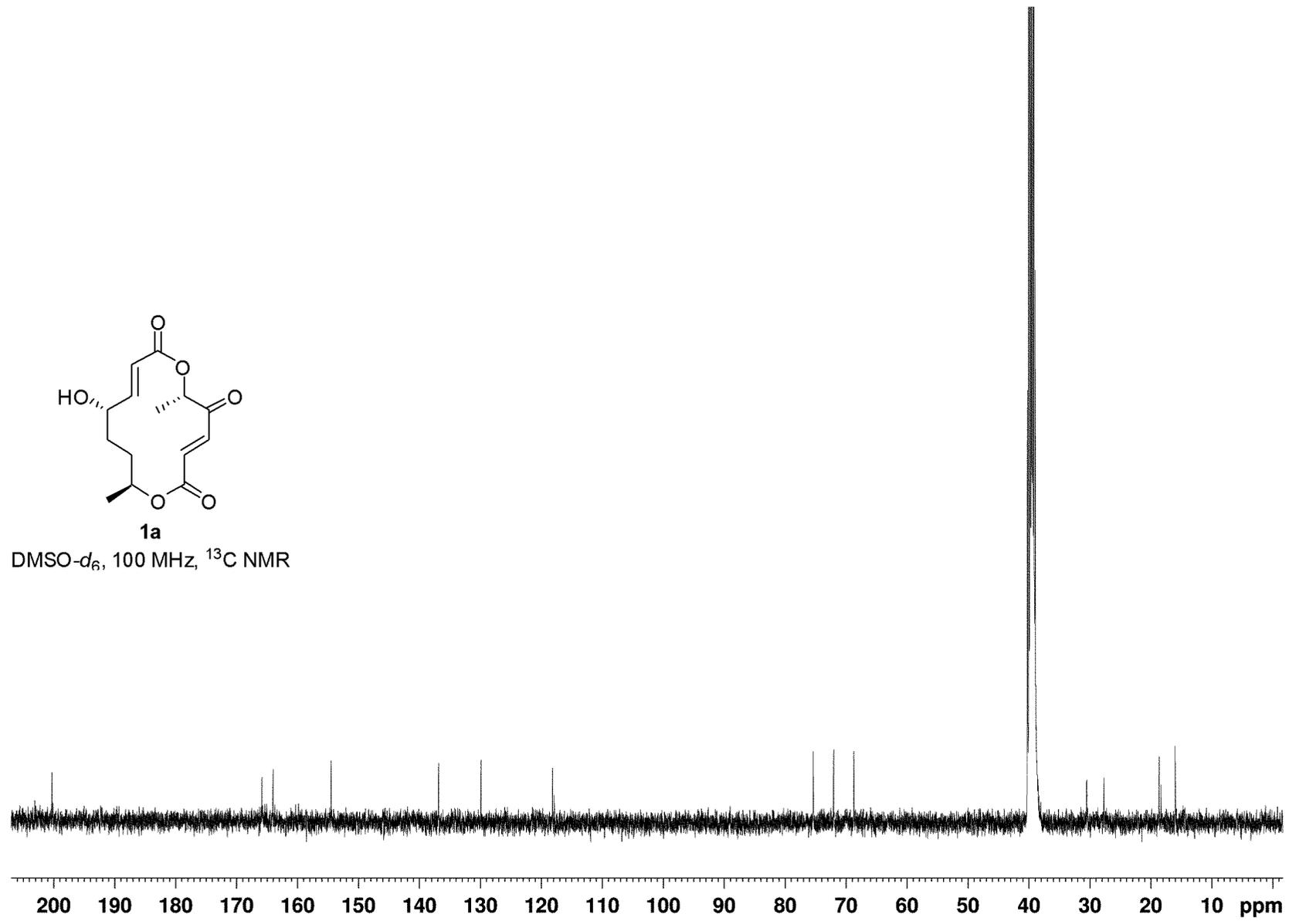
```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -2.10 dB
PL12      13.90 dB
PL13      13.90 dB
PL2W      17.72104263 W
PL12W     0.44513249 W
PL13W     0.44513249 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6132891 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```



1a

DMSO-d₆, 100 MHz, ¹³C NMR

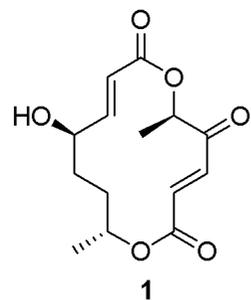


7.388
7.348
7.265
7.154
7.146
7.115
7.106

6.489
6.449
6.164
6.125
6.120

5.359
5.340
5.322
5.304
5.037
5.031
5.021
5.015
5.005
4.998
4.547
4.528

2.049
2.045
2.035
2.029
2.017
2.011
2.000
1.994
1.983
1.977
1.896
1.890
1.880
1.875
1.858
1.852
1.843
1.837
1.826
1.820
1.777
1.761
1.755
1.739
1.726
1.711
1.704
1.689
1.568
1.550
1.297
1.281
0.000



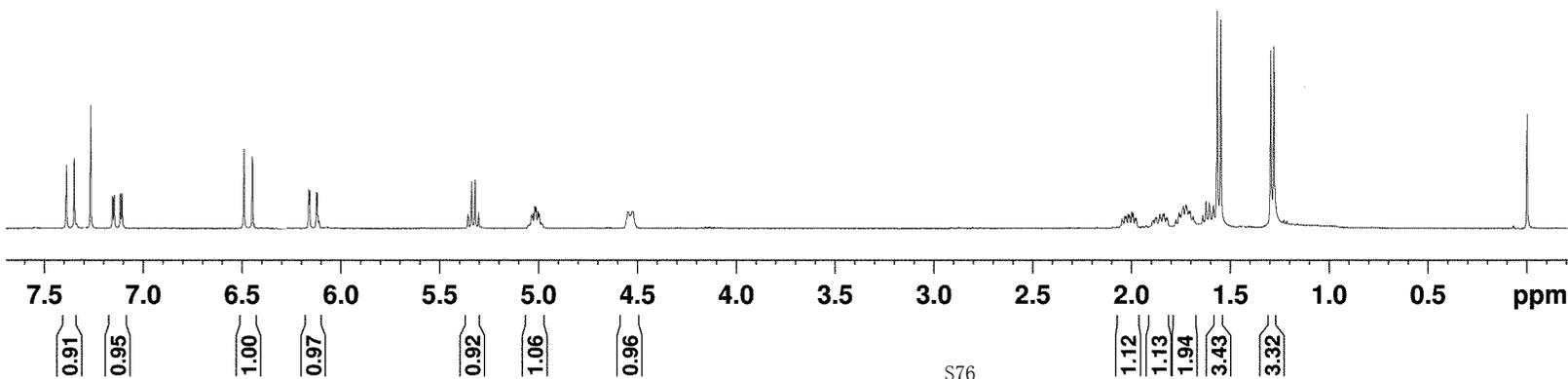
CDCl₃ 400MHz ¹H NMR

```

NAME          hanjjbtm
EXPNO         20
PROCNO        1
Date_         20090116
Time          22.28
INSTRUM       spect
PROBHD        5 mm PABBO BB-
PULPROG       zg30
TD            65536
SOLVENT       CDC13
NS            16
DS            2
SWH           8223.685 Hz
FIDRES        0.125483 Hz
AQ            3.9846387 sec
RG            456
DW            60.800 usec
DE            6.50 usec
TE            290.8 K
D1            1.00000000 sec
TD0           1
  
```

```

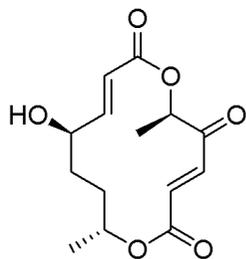
===== CHANNEL f1 =====
NUC1          1H
P1            14.60 usec
PL1           0.00 dB
PL1W         11.47932053 W
SFO1          400.1324710 MHz
SI            32768
SF            400.1300031 MHz
WDW           EM
SSB           0
LB            0.30 Hz
GB            0
PC            1.00
  
```



199.49
 165.60
 164.07
 151.35
 135.70
 130.89
 119.62

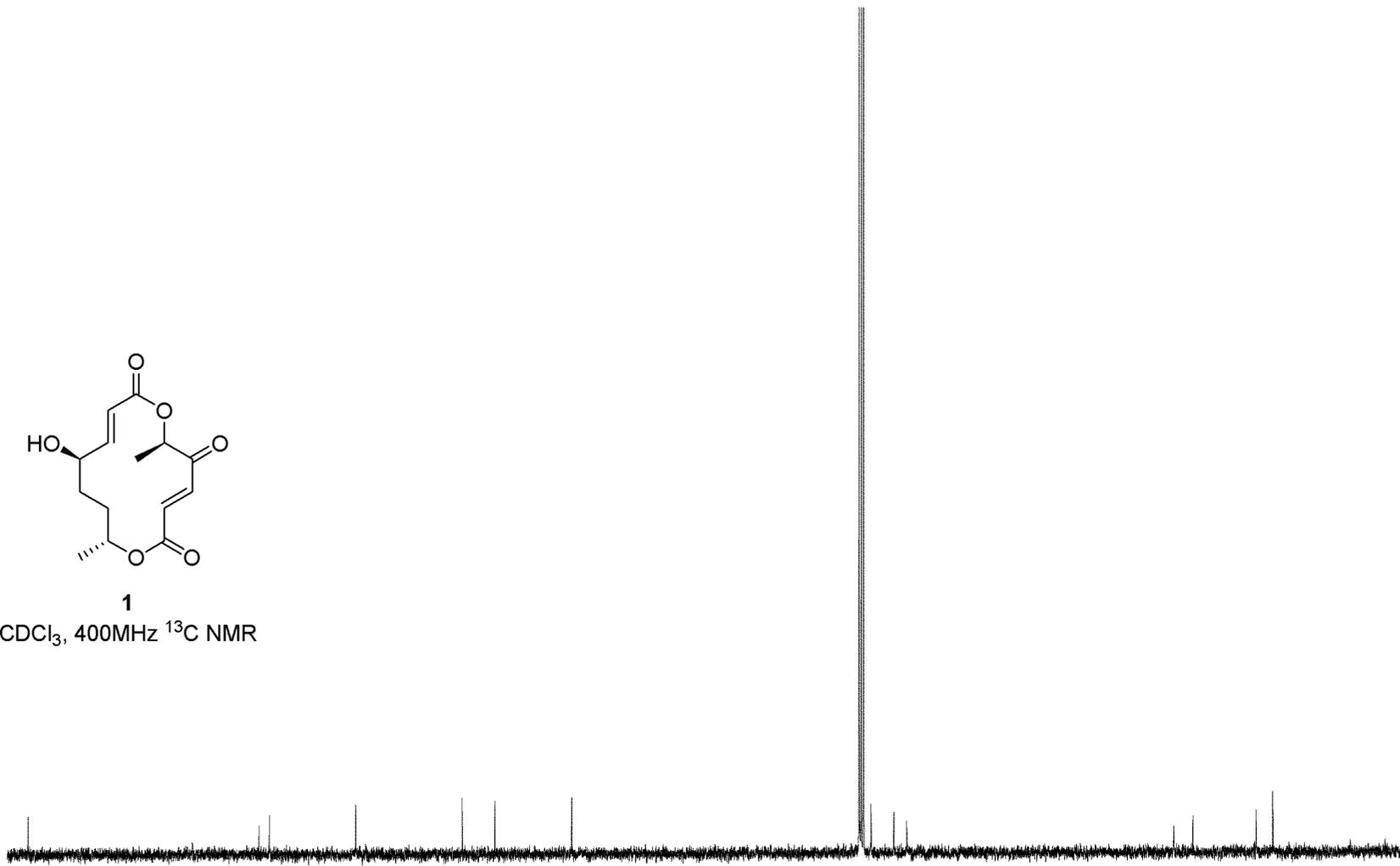
77.32
 77.00
 76.68
 75.58
 72.23
 70.32

31.04
 28.23
 18.96
 16.50



1

CDCl₃, 400MHz ¹³C NMR



```

NAME      hanjjbtm
EXPNO     21
PROCNO    1
Date_     20090116
Time      22.52
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CDCl3
NS         400
DS         4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ         1.3631988 sec
RG         2050
DW         20.800 usec
DE         6.50 usec
TE         291.4 K
D1         2.0000000 sec
D11        0.0300000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1         9.40 usec
PL1       -2.00 dB
PL1W      57.32743073 W
SFO1      100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2       1H
PCPD2     90.00 usec
PL2       -2.00 dB
PL12      15.50 dB
PL13      15.50 dB
PL2W      18.19349861 W
PL12W     0.32353121 W
PL13W     0.32353121 W
SFO2      400.1316005 MHz
SI         32768
SF         100.6127707 MHz
WDW        EM
SSB        0
LB         1.00 Hz
GB         0
PC         1.40
  
```

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

7.394
7.353
7.227
7.219
7.188
7.180

6.408
6.368

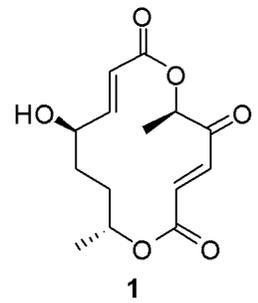
5.956
5.952
5.917
5.912

5.215
5.204
5.183
5.165
5.147
5.129
4.935
4.929
4.919
4.914
4.904
4.898

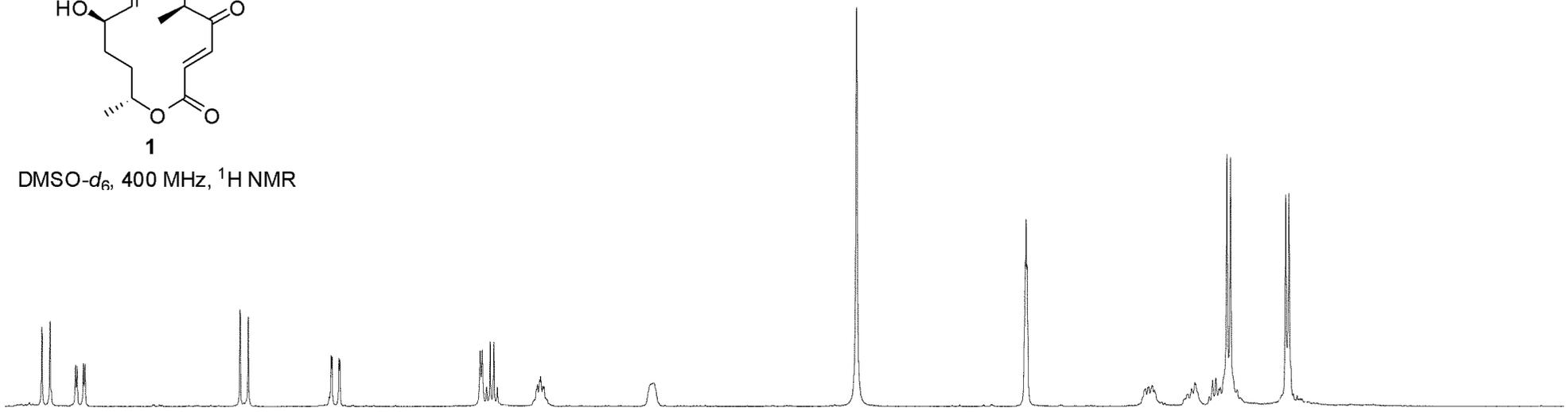
4.362
4.356

3.343

2.504
2.500
2.496
1.907
1.895
1.890
1.873
1.863
1.710
1.696
1.678
1.662
1.657
1.590
1.573
1.557
1.541
1.535
1.502
1.484
1.210
1.194

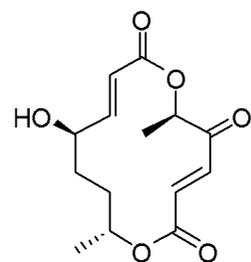


DMSO-d₆, 400 MHz, ¹H NMR



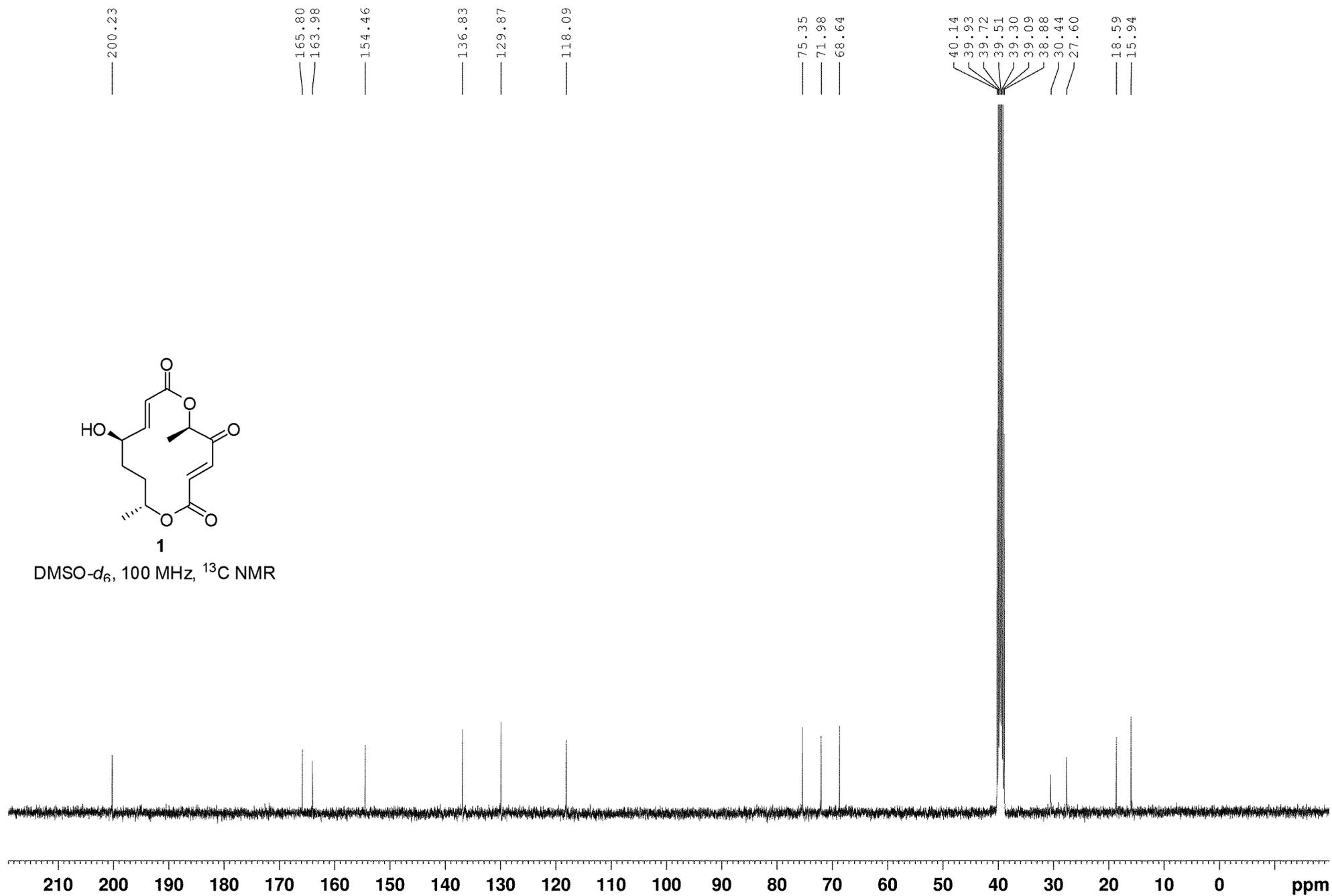
7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

1.00 0.96 0.96 0.98 0.86 1.00 1.07 0.97 1.20 1.07 0.92 4.10 3.21



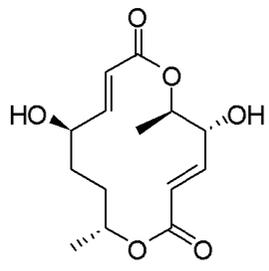
1

DMSO-*d*₆, 100 MHz, ¹³C NMR



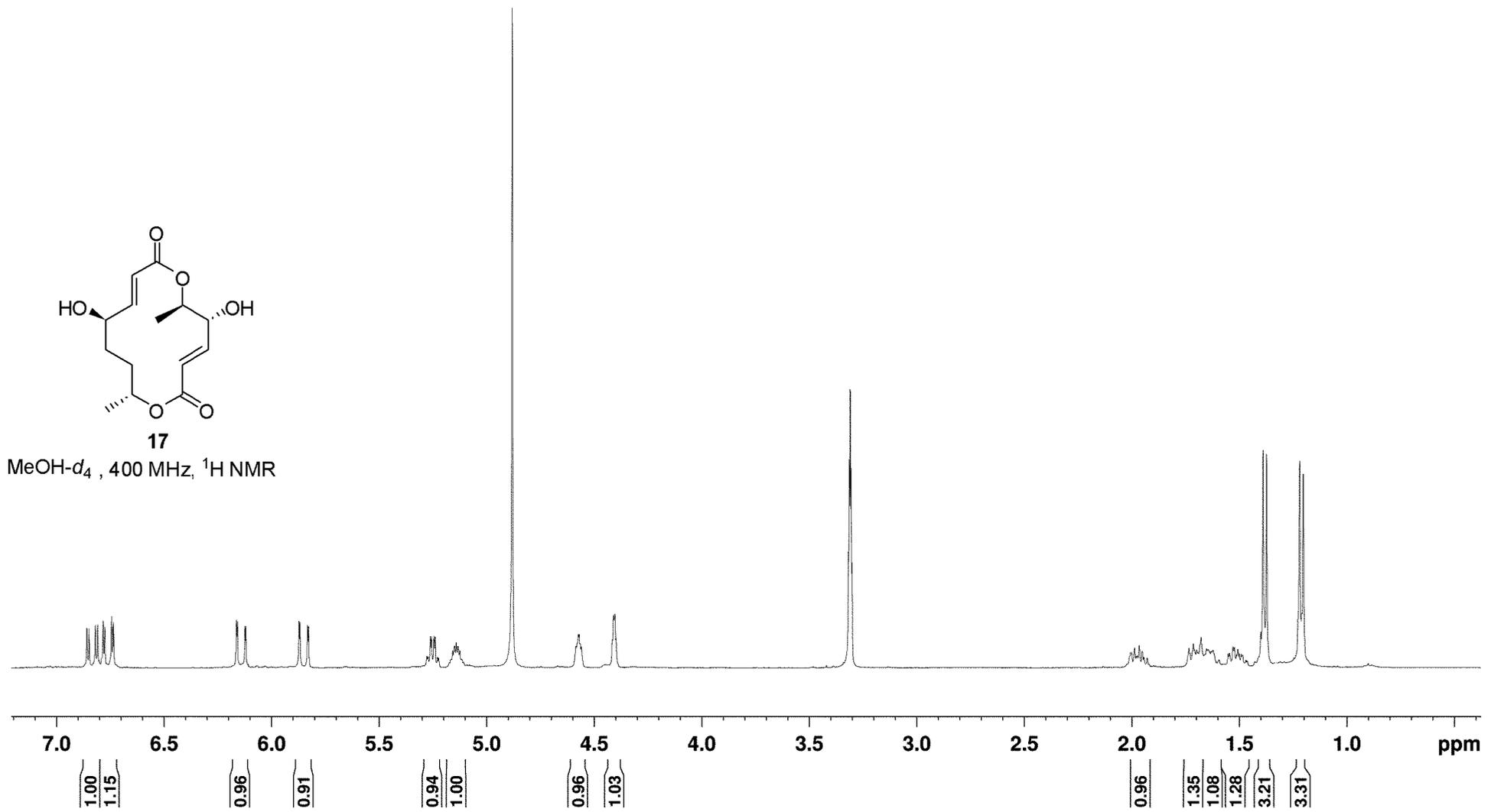
6.858
6.848
6.819
6.808
6.783
6.775
6.743
6.736
6.164
6.160
6.125
6.121
5.874
5.869
5.834
5.830
5.279
5.274
5.262
5.257
5.246
5.241
5.229
5.224
5.167
5.159
5.150
5.142
5.134
5.126
5.118
4.882
4.585
4.580
4.575
4.571
4.566
4.561
4.416
4.411
4.405
4.400

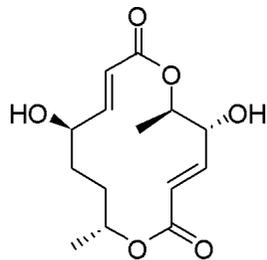
2.002
1.988
1.979
1.974
1.966
1.952
1.944
1.929
1.735
1.730
1.714
1.709
1.700
1.694
1.679
1.653
1.647
1.632
1.623
1.552
1.546
1.530
1.523
1.514
1.508
1.502
1.492
1.486
1.470
1.463
1.391
1.374



17

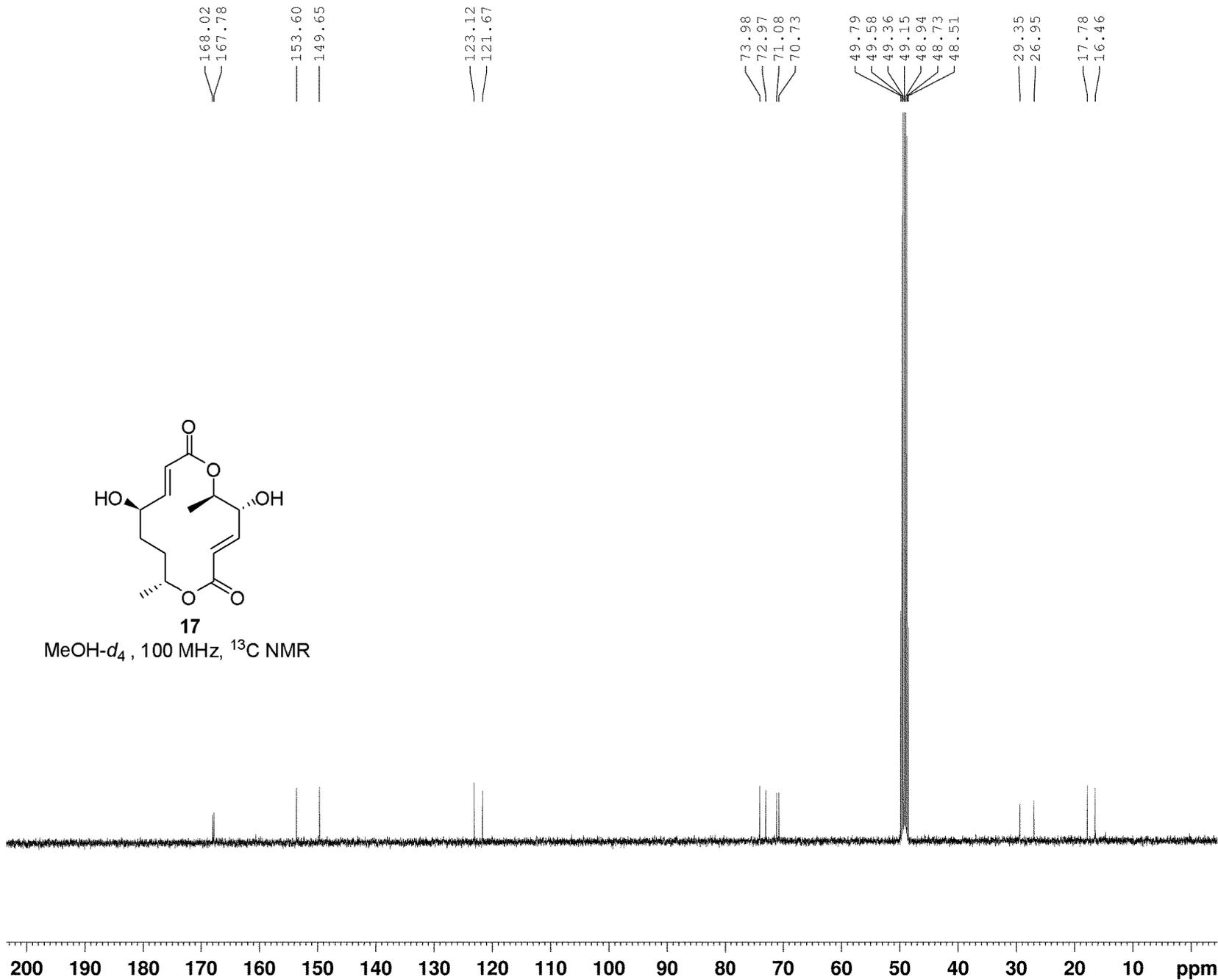
MeOH-*d*₄ , 400 MHz, ¹H NMR





17

MeOH-*d*₄, 100 MHz, ¹³C NMR



168.02
167.78

153.60
149.65

123.12
121.67

73.98
72.97
71.08
70.73

49.79
49.58
49.36
49.15
48.94
48.73
48.51

29.35
26.95

17.78
16.46

```

NAME      hanjbb2diol
EXPNO     11
PROCNO    1
Date_     20090115
Time      20.36
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD        65536
SOLVENT   MeOD
NS        256
DS        4
SWH       24038.461 Hz
FIDRES    0.366798 Hz
AQ        1.3631988 sec
RG        71.8
DW        20.800 usec
DE        6.50 usec
TE        300.0 K
D1        2.00000000 sec
D11       0.03000000 sec
TD0       1
  
```

```

===== CHANNEL f1 =====
NUC1      13C
P1        9.70 usec
PL1       -2.00 dB
PL1W      56.13311005 W
SFO1      100.6228298 MHz
  
```

```

===== CHANNEL f2 =====
CPDPRG2   waltz16
NUC2      1H
PCPD2     80.00 usec
PL2       -2.10 dB
PL12      13.90 dB
PL13      13.90 dB
PL2W      17.72104263 W
PL12W     0.44513249 W
PL13W     0.44513249 W
SFO2      400.1316005 MHz
SI        32768
SF        100.6126112 MHz
WDW       EM
SSB       0
LB        1.00 Hz
GB        0
PC        1.40
  
```

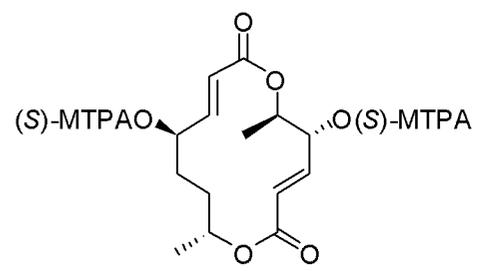
7.552
7.542
7.533
7.492
7.471
7.458
7.454
7.445
7.441
7.427
7.419
7.401
7.380
7.362
7.262
6.700
6.691
6.661
6.652
6.612
6.600
6.572
6.560
5.798
5.771
5.765
5.726
5.722
5.686
5.682
5.410
5.404
5.393
5.388
5.377
5.371
5.360
5.158
5.150
5.142
5.134
5.125
5.125
3.554
3.512
2.204
2.189
2.167
2.152
2.131
1.806
1.786
1.769
1.749
1.691
1.681
1.661
1.653
1.642
1.633
1.554
1.469
1.463
1.442
1.432
1.426
1.409
1.388
1.256
1.240
1.196
1.179

```

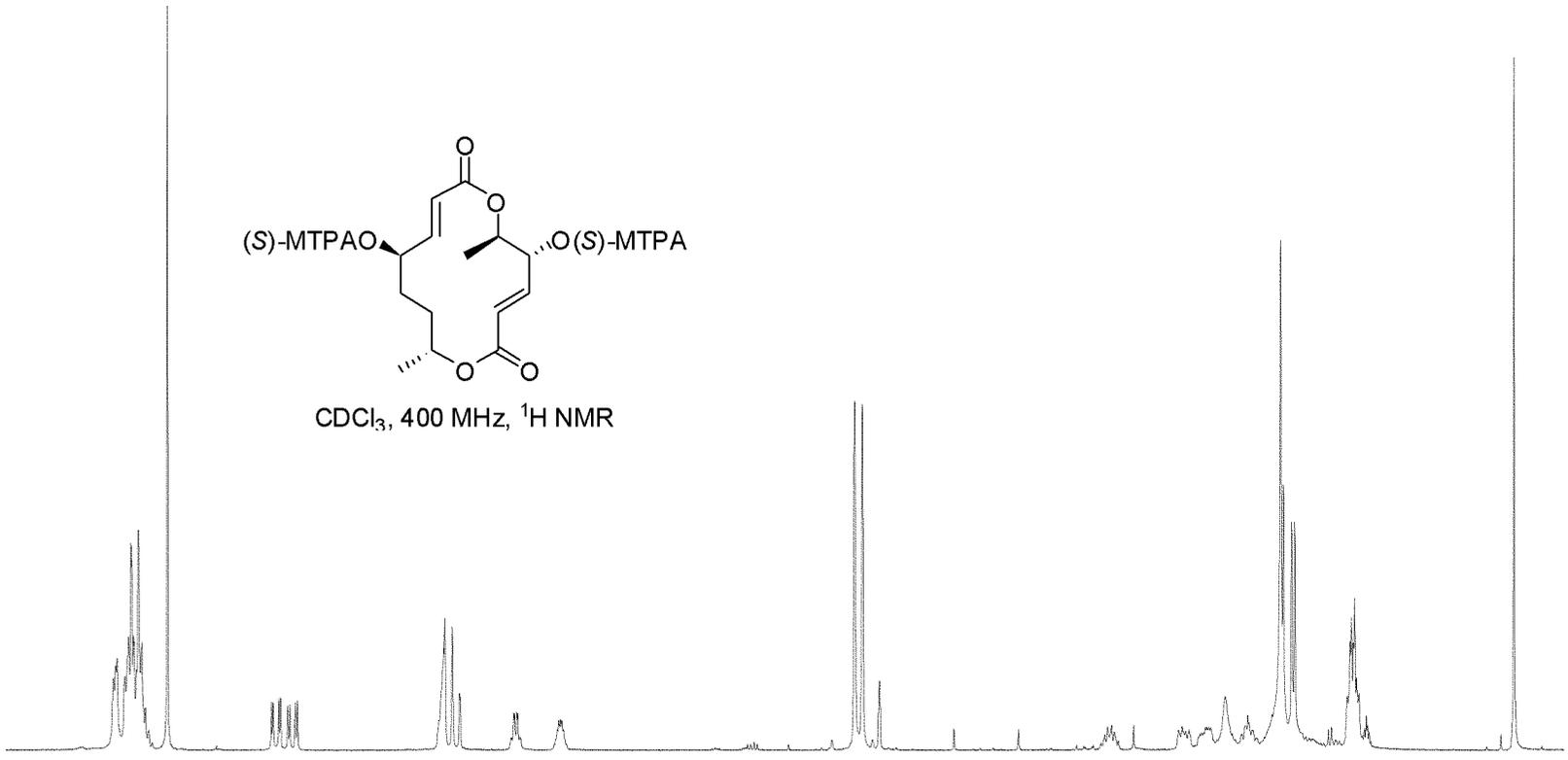
NAME      hanjjsmtpadiol
EXPNO     10
PROCNO    1
Date_     20090117
Time      22.49
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         64
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         575
DW         60.800 usec
DE         6.50 usec
TE         290.9 K
D1         1.00000000 sec
TD0        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SFO1      400.1324710 MHz
SI         32768
SF         400.1300043 MHz
WDW       EM
SSB       0
LB         0.30 Hz
GB         0
PC         1.00
  
```



CDCl₃, 400 MHz, ¹H NMR



8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

2.26 9.35 1.00 0.99 2.46 1.48 0.99 0.97 3.06 2.99 1.00 1.06 1.30 2.01 1.49 7.33 3.64

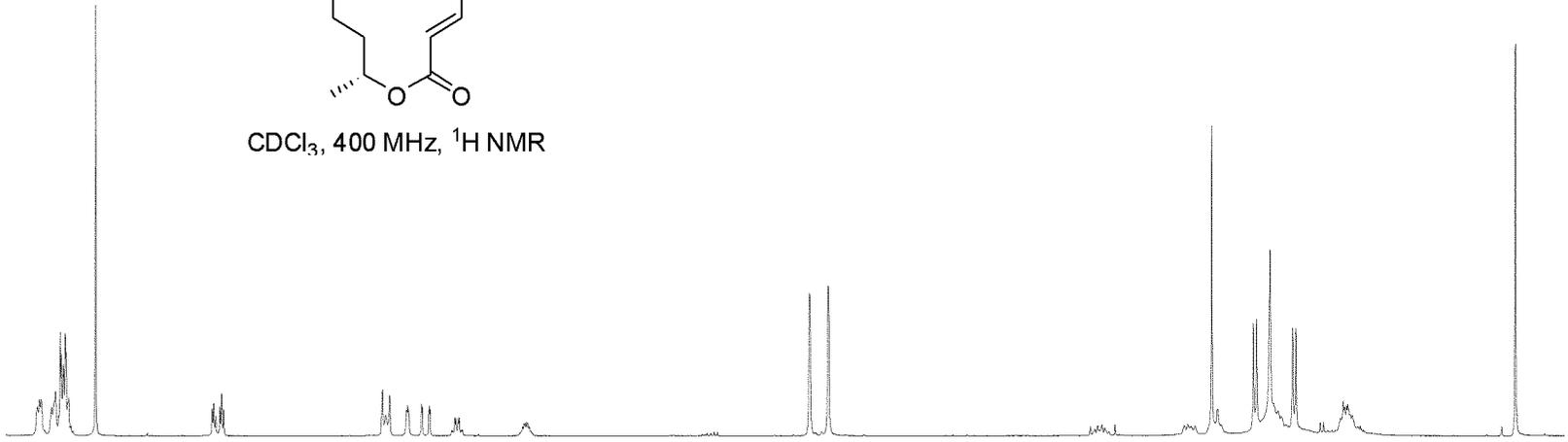
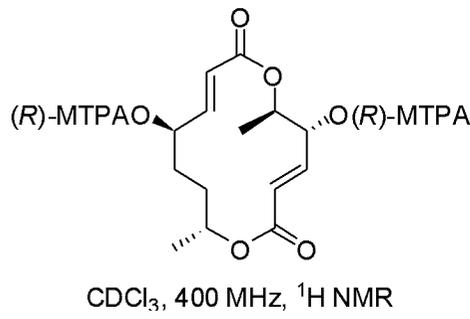
7.567
7.557
7.548
7.495
7.481
7.475
7.460
7.450
7.444
7.436
7.433
7.425
7.422
7.414
7.411
7.407
7.270
6.673
6.667
6.665
6.656
6.634
6.625
6.615
5.802
5.785
5.766
5.761
5.683
5.677
5.673
5.670
5.664
5.602
5.599
5.563
5.559
5.543
5.425
5.414
5.409
5.398
5.392
5.084
5.076
5.068
5.060
5.051
3.617
3.615
3.521
2.182
2.160
2.145
2.138
2.123
2.109
1.702
1.683
1.666
1.646
1.561
1.543
1.531
1.548
1.331
1.263
1.245
1.222
1.147
1.131

```

NAME      hjjxmtpadiol
EXPNO     10
PROCNO    1
Date_     20090217
Time      16.40
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         96
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         575
DW         60.800 usec
DE         6.50 usec
TE         292.0 K
D1         1.00000000 sec
TDO        1
  
```

```

===== CHANNEL f1 =====
NUC1      1H
P1        14.60 usec
PL1       0.00 dB
PL1W      11.47932053 W
SF01      400.1324710 MHz
SI        32768
SF        400.1300015 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00
  
```



7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 ppm

2.15 2.16 6.03 0.94 1.07 2.01 1.02 1.00 1.00 1.02 2.98 3.07 0.96 1.33 4.74 3.52 8.49 3.67

7.451
7.443
7.431
7.426
7.283
7.270
7.243
7.034
7.024
6.994
6.984
6.485
6.445
5.989
5.985
5.949
5.945
5.762
5.751
5.347
5.329
5.311
5.293
5.019
5.013
5.004
4.998
4.989
4.982

3.533

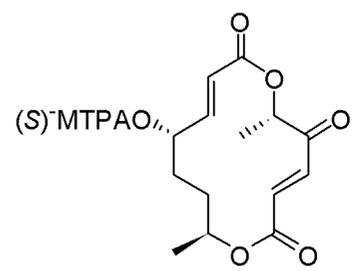
2.010
1.994
1.979
1.964
1.774
1.768
1.746
1.729
1.712
1.570
1.552
1.479
1.462
1.456
1.438
1.424
1.273
1.262

0.008

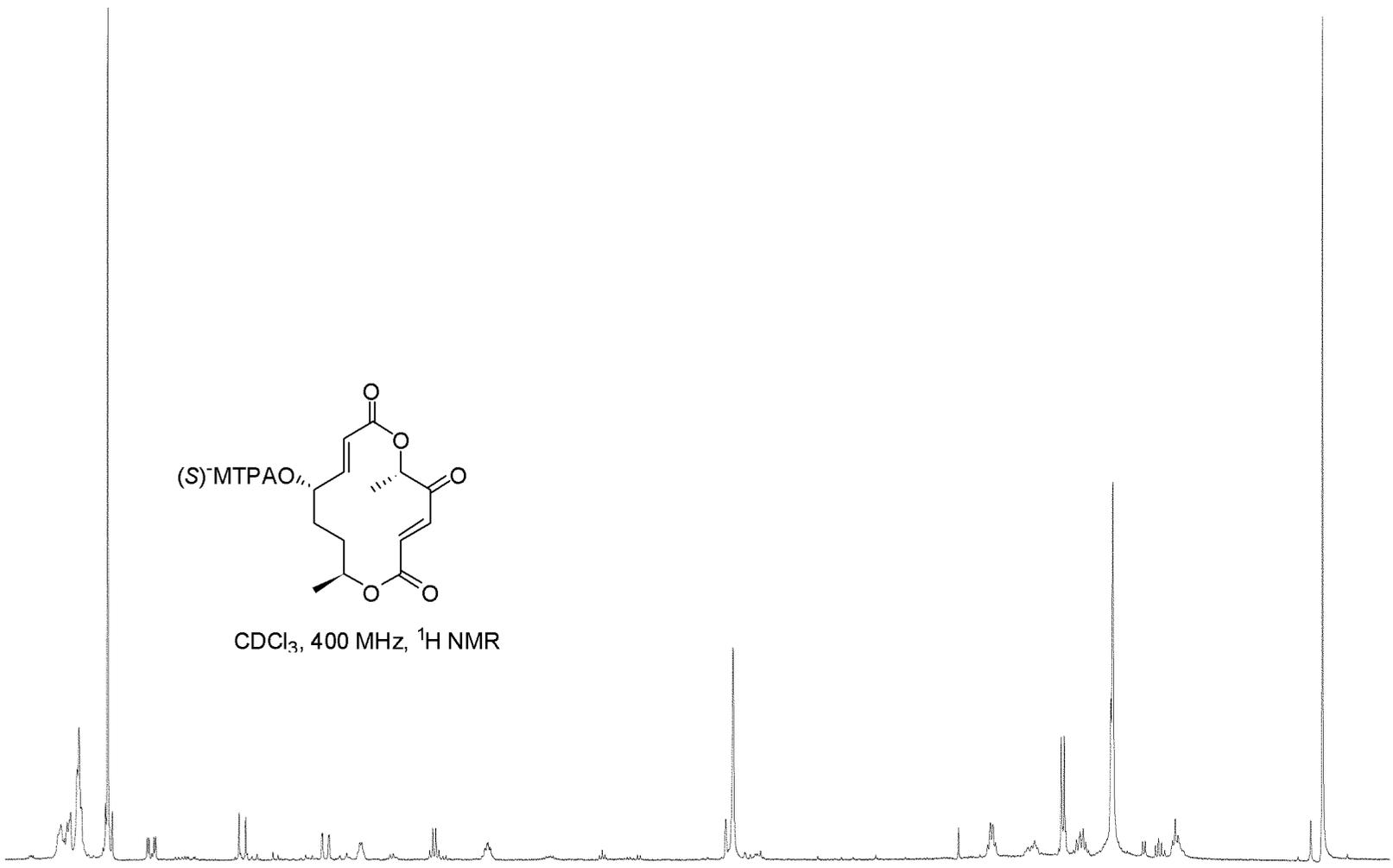
```

NAME      hanjjmosher2
EXPNO     10
PROCNO    1
Date_     20090107
Time      13.39
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         80
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         812
DW         60.800 usec
DE         6.50 usec
TE         291.9 K
D1         1.00000000 sec
TD0        1

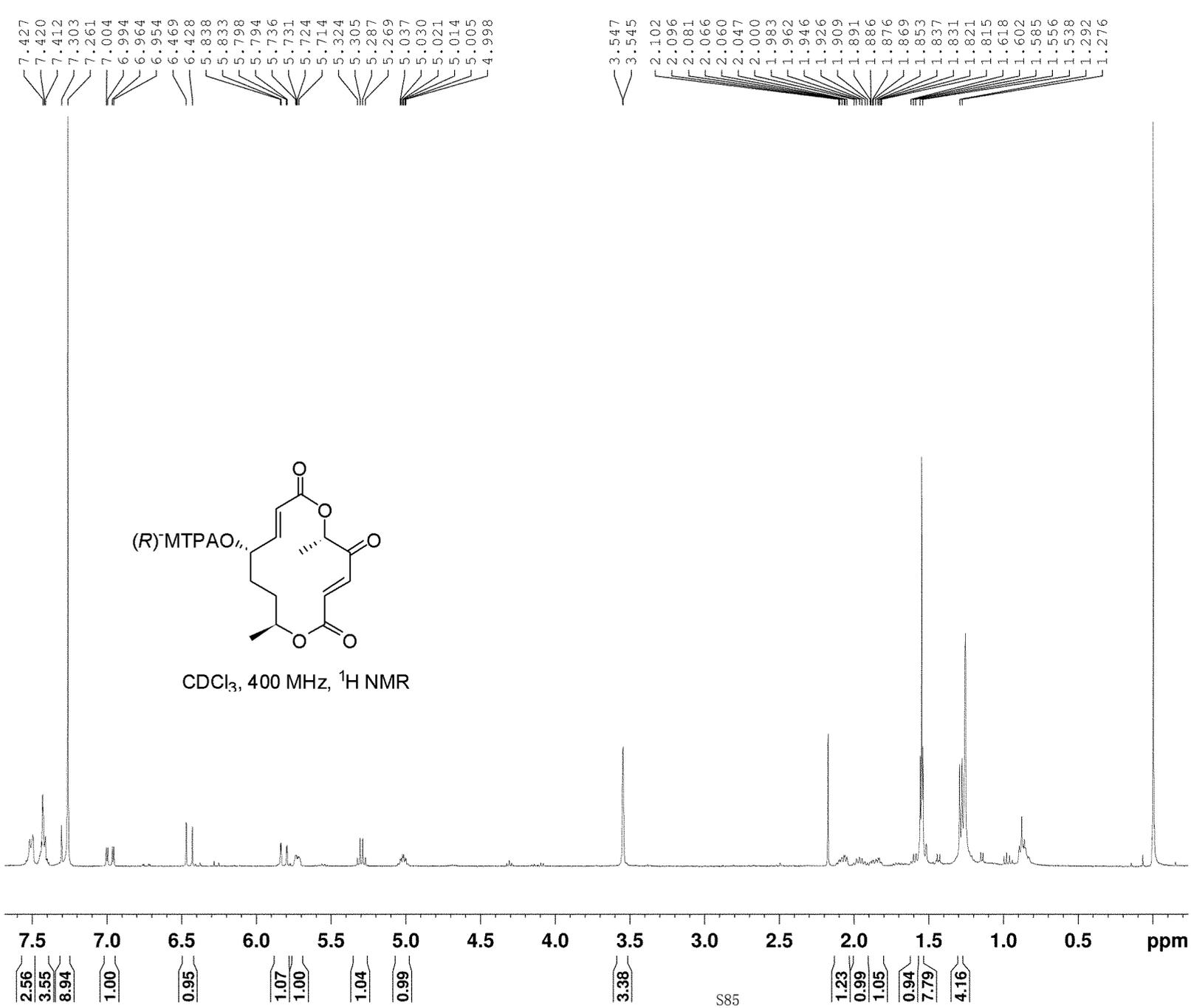
===== CHANNEL f1 =====
NUC1       1H
P1         14.60 usec
PL1        0.00 dB
PL1W       11.47932053 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300012 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00
  
```



CDCl₃, 400 MHz, ¹H NMR



2.15 2.44 6.23 9.46 1.03 1.00 0.96 0.93 1.00 1.09 5.16 2.34 1.97 4.00 2.57 9.97



```

NAME      hanjjmosher1
EXPNO     10
PROCNO    1
Date_     20090107
Time      14.20
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zg30
TD         65536
SOLVENT   CDCl3
NS         80
DS         2
SWH        8223.685 Hz
FIDRES     0.125483 Hz
AQ         3.9846387 sec
RG         812
DW         60.800 usec
DE         6.50 usec
TE         292.4 K
D1         1.00000000 sec
TD0        1

===== CHANNEL f1 =====
NUC1       1H
P1         14.60 usec
PL1        0.00 dB
PL1W       11.47932053 W
SFO1       400.1324710 MHz
SI         32768
SF         400.1300050 MHz
WDW        EM
SSB        0
LB         0.30 Hz
GB         0
PC         1.00

```