Supporting Information

'Asymmetric Synthesis of Highly Functionalized Cyclopentanes by a Rhodium- and Scandium-Catalyzed Five-Step Domino Sequence'

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1. Experimental Section

1.1 General Considerations

All reactions were conducted in oven-dried glassware under an inert atmosphere of dry argon. All reagents were used as received from commercial suppliers, unless specified otherwise. Cyclohexane, dichloroethane, ethyl acetate and heptane solvents and scandium(III) triflate were purchased from Aldrich were used as received. Dichloromethane, hexanes and toluene were obtained from a Grubbs-type solvent purification system. ^{1}H NMR spectra were recorded at either 400 MHz on an INOVA-400 spectrometer or at 600 MHz on an INOVA-600 spectrometer. ^{13}C NMR spectra were recorded at 100 or 150 MHz on the same instruments. NMR spectra were recorded in deuterated chloroform ($CDCl_3$) solutions, with residual chloroform (δ 7.27 ppm for ^{1}H NMR and δ 77.23 ppm for ^{13}C NMR) or tetramethylsilane (δ 0.00 ppm for ^{1}H NMR) taken as the internal standard, and were reported in parts per million (ppm). Abbreviations for signal coupling are as follows: s, singlet; d, doublet; t, triplet; q, quartet; m, multiplet. Coupling constants were taken from the spectra directly and are uncorrected. IR spectra were collected on a Nicolet iS10 FT-IR spectrometer as neat films. Mass spectra determinations were carried out on a Thermo Finnigan LTQ-FTMS spectrometer with ESI or APCI ionization. Optical rotations were measured on JASCO P-2000 polarimeter. Analytical TLC was performed on silica gel plates using UV light or stained with 10% vanillin/1% sulfuric acid/ethanol solution. Flash column chromatography was performed with silica gel 60 A (230-400 mesh). **1a-d**[1], Rh_2(*S*-DOSP)$_4$ and Rh_2(*R*-DOSP)$_4$[2], **2a-d**[3], **2e-f**[4], **2g**[5], **2h**[6], **2i**[7], **4**[5], **10**[5] and **13**[8] were all synthesized according to published procedures.

1.2 General Procedure for One-Pot Cyclopentane Synthesis

An oven-dried, 25 mL round-bottomed flask, equipped with a stir bar, was capped with a rubber septum and placed under a dry argon atmosphere. The reaction vessel was charged with Rh_2(*S*-DOSP)$_4$ (19 mg, 0.01 mmol, 0.01 equiv) and the allyl alcohol (1.0 mmol, 1.0 equiv) in heptane (1.0 mL). The solution was cooled to 0 ^{o}C in an ice bath before adding a heptane solution (10 mL) of the diazo compound (1.1 mmol, 1.1 equiv) drop-wise over 30 min. Following addition, the reaction was stirred at 0 ^{o}C for 2 h before warming to rt for 30 min. The rubber septum was removed and the reaction flask was fixed with a reflux condenser and heated to 80 ^{o}C for 24 h or until TLC indicated complete conversion of the [2,3]-rearrangement product to a mixture of oxy-Cope and ene products. Scandium triflate (98 mg, 0.20 mmol, 0.20 equiv) was then added in a single portion and the reaction was heated for an addition 2 h or until TLC indicated complete conversion of the oxy-Cope product to the cyclopentane. The reaction was then cooled to ambient temperature and concentrated *in vacuo*. The product was purified by flash chromatography.

1.3 References

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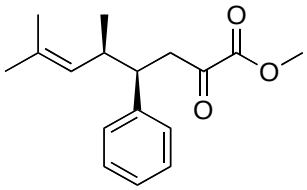
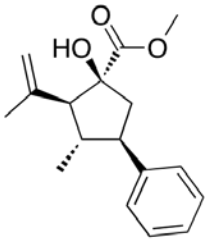
2. Characterization Data



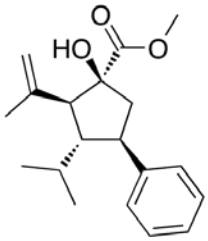
(+)-(4*R*,5*R*)-methyl 5,7-dimethyl-2-oxo-4-phenyloct-6-enoate (6).

A 25 mL round-bottomed flask, equipped with a magnetic stirring bar and reflux condenser, was charged with a solution of (*R*,*E*)-methyl 2-hydroxy-3,3-dimethyl-2-((*E*)-styryl)hex-4-enoate (**4**) (105 mg, 0.38 mmol) in heptane (5 mL). The solution was heated in an oil bath (preheated to 80 ^{o}C) for 15 h, until complete consumption of the starting material was apparent by TLC (SiO_2, pentane/diethyl ether, 10 : 1). The reaction vessel was cooled to ambient temperature and silica gel (500 mg) was added. The mixture was stirred at room temperature for 2 h before concentrating *in vacuo*. The crude was purified on silica gel eluting with pentane : ether (10 : 1), and gave compound **6** as a colorless oil (104 mg, quant.). $[\alpha]^{20}_{D}$ 63.5^{o} (*c* 1.0, $CHCl_3$). ^{1}H NMR (400 MHz, $CDCl_3$): δ 7.26-7.30 (m, 2H), 7.17-7.21 (m, 3H), 4.88 (d, *J* = 10.0 Hz, 1H), 3.77 (s, 3H), 3.20-3.28 (m, 1H), 2.95-3.03 (m, 2H), 2.52-2.62 (m, 1H), 1.68 (d, *J* =1.2 Hz, 3H), 1.65 (d, *J* = 1.2 Hz, 3H), 0.70 (d, *J* = 6.4 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 193.2, 161.4, 143.3, 132.8, 130.2, 128.6, 128.3, 126.7, 53.0, 48.3, 44.6, 39.0, 26.0, 19.5, 18.4,. FTIR (neat): ν_{max}/cm^{-1} 1728, 1452, 1268, 1239, 1096, 1061. HRMS (p-APCI): *m/z* 275.1639 [$(M+H)^+$ requires 275.1642]. HPLC analysis: 82% ee, (*R*,*R*)-Whelk 01, 0.5% isopropanol/hexanes, 0.7 mL/min, UV: 230 nm, t_R: 22.30 min (minor), 34.30 min (major).



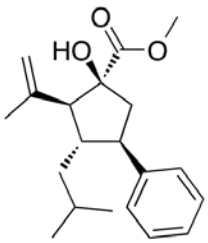
(+)-(1*S*,2*S*,3*S*,4*R*)-methyl 1-hydroxy-3-methyl-4-phenyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (7a).

Prepared by general procedure with methyl styryldiazoacetate (**1a**) (225 mg, 1.1 mmol, 1.0 equiv) and 4-methyl-3-penten-2-ol (**2a**) (101 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **7a** as a white solid (261 mg, 95%). MP = 40-42 ^{o}C. $[\alpha]^{20}_{D}$ 9.3^{o} (*c* 1.03, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.31 (m, 5H), 5.08 (s, 1H), 4.80 (s, 1H), 3.81 (s, 3H), 2.98 (s, 1H, -OH), 2.83 (dd, *J* = 14.4, 10.2 Hz, 1H), 2.76 (ddd, *J* = 10.2, 8.4, 7.8 Hz, 1H), 2.53 (d, *J* = 12 Hz, 1H), 2.29 (m, 1H), 2.03 (dd, *J* = 14.4, 8.4 Hz, 1H), 1.73 (s, 3H), 0.88 (d, *J* = 6.3 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 176.9, 144.1, 141.2, 128.4, 127.8, 126.3, 114.8, 81.3, 63.6, 52.7, 51.7, 46.7, 44.8, 23.4, 16.0. FTIR (neat): ν_{max}/cm^{-1} 3523, 3027, 2950, 1729, 1450, 1431. HRMS (p-APCI): *m/z* 275.1641 [$(M+H)^+$ requires 275.1642]. HPLC analysis: 82% ee, CHIRALCEL ODR, 0.5% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 11.54 min (major), 22.08 min (minor).



(-)-(1*S*,2*S*,3*S*,4*R*)-methyl 1-hydroxy-3-isopropyl-4-phenyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (7b).

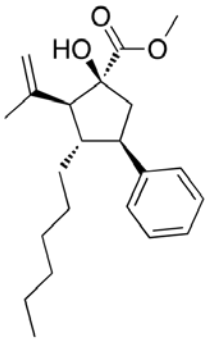
Prepared by general procedure with methyl styryldiazoacetate (**1a**) (226 mg, 1.1 mmol, 1.0 equiv) and 2,5-dimethyl-4-hexen-3-ol (**2b**) (127 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **7b** as a colorless oil (183 mg, 67%). $[\alpha]^{20}_{D}$ -10.2^{o} (*c* 1.07, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.37 (d, *J* = 7.2 Hz, 2H), 7.27 (t, *J* = 7.2 Hz, 2H), 7.16 (t, *J* = 7.2 Hz, 1H), 5.05 (s, 1H), 4.86 (s, 1H), 3.77 (s, 3H), 3.09 (m, 1H), 3.08 (s, 1H), 2.80 (dd, *J* = 14.4, 10.2 Hz, 1H), 2.77 (d, *J* = 12 Hz, 1H), 2.50 (ddd, *J* = 15.0, 12.6, 2.4 Hz, 1H), 1.92 (dd, *J* = 14.4, 7.2 Hz, 1H), 1.77 (s, 3H), 1.75 (m, 1H), 0.87 (d, *J* = 7.2 Hz, 3H), 0.66 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.5, 146.3, 142.2, 128.3, 128.1, 125.9, 114.9, 81.6, 59, 54.2, 52.5, 47.2, 45, 27.7, 23.1, 20.6, 18.5. FTIR (neat): ν_{max}/cm^{-1} 3512, 3023, 2950, 2923, 1725, 1450, 1431. HRMS (p-APCI): *m/z* 285.1847 [(M-OH)$^{+}$ requires 285.1849]. HPLC analysis: 80% ee, CHIRALCEL ODR, 0.5% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 11.27 min (major), 22.50 min (minor).



(+)-(1*S*,2*S*,3*S*,4*R*)-methyl 1-hydroxy-3-isobutyl-4-phenyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (7c).

Prepared by general procedure with methyl styryldiazoacetate (**1a**) (229 mg, 1.1 mmol, 1.0 equiv) and 2,6-dimethyl-2-hepten-4-ol (**2c**) (142 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **7c** as a white solid (232 mg, 73%). MP = 68-69 ^{o}C. $[\alpha]^{20}_{D}$ 8.6^{o} (*c* 0.50, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.37 (d, *J* = 7.2 Hz, 2H), 7.28 (t, *J* = 7.2 Hz, 2H), 7.18 (t, *J* = 7.2 Hz, 1H), 5.06 (s, 1H), 4.85 (s,

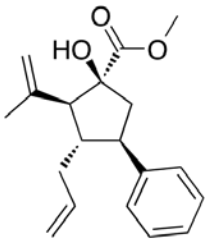
1H), 3.79 (s, 3H), 3.03 (s, 1H), 2.86 (m, 2H), 2.60 (d, *J* = 12 Hz, 1H), 2.50 (m, 1H), 1.94 (dt, *J* = 7.2, 4.8 Hz, 1H), 1.75 (s, 3H), 1.29 (m, 1H), 1.25 (m, 2H), 0.68 (d, *J* = 6.6 Hz, 3H), 0.51 (d, *J* = 6.6 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 176.6, 145.8, 141.7, 128.3, 128, 126.1, 114.8, 81.6, 63.1, 52.6, 51.2, 47.8, 47, 43.8, 25.2, 23.9, 23.5, 21.6. FTIR (neat): ν_{max}/cm^{-1} 3520, 3024, 2950, 1725, 1636, 1450, 1431. HRMS (p-APCI): *m/z* 317.2107 [$(M+H)^+$ requires 317.2111]. HPLC analysis: 80% ee, CHIRALCEL ODR, 0.5% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 10.94 min (major), 23.52 min (minor).



(-)-(1*S*,2*S*,3*S*,4*R*)-methyl 3-hexyl-1-hydroxy-4-phenyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (7d).

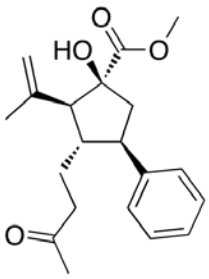
Prepared by general procedure with methyl styryldiazoacetate (**1a**) (228 mg, 1.1 mmol, 1.0 equiv) and 2-methyl-2-decen-4-ol (**2d**) (172 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **7d** as a colorless oil (276 mg, 80%). $[\alpha]^{20}_D$ -3.6^o (*c* 1.28, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.34 (d, *J* = 7.5 Hz, 2H), 7.29 (t, *J* = 7.5 Hz, 2H), 7.19 (t, *J* = 7.5 Hz, 1H), 5.07 (s, 1H), 4.83 (s, 1H), 3.79 (s, 3H), 3.01 (s, 1H), 2.93 (dd, *J* = 18.0, 10.2 Hz, 1H), 2.82 (dd, *J* = 14.1, 11.1 Hz, 1H), 2.65 (d, *J* = 12.6 Hz, 1H), 2.40 (m, 1H), 1.96 (dd, *J* = 14.1, 7.8 Hz, 1H), 1.74 (s, 3H), 1.28-1.39 (m, 2H), 1.13-1.19 (m, 2H), 1.00-1.13 (m, 6H), 0.81 (t, *J* = 7.2 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.7, 145.3, 141.7, 128.3, 128, 126.1, 114.8, 81.4, 61.5, 52.6, 49.4, 49, 47.1, 31.6,

31.4, 29.5, 26, 23.4, 22.5, 14.0. FTIR (neat): ν_{max}/cm^{-1} 3520, 2950, 2923, 2849, 1725, 1632, 1454. HRMS (p-APCI): *m/z* 345.2421 [(M+H)$^+$ requires 345.2424]. HPLC analysis: 78% ee, CHIRALCEL ODR, 0.5% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 9.63 min (major), 19.58 min (minor).



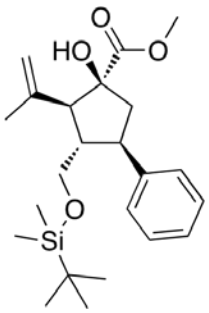
(-)-(1*S*,2*S*,3*S*,4*R*)-methyl 3-allyl-1-hydroxy-4-phenyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (7e).

Prepared by general procedure with methyl styryldiazoacetate (**1a**) (212 mg, 1.0 mmol, 1.0 equiv) and 6-methyl-1,5-heptadien-4-ol (**2e**) (172 mg, 0.9 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **7e** as a colorless oil (232 mg, 86%). $[\alpha]^{20}_D$ -3.3^o (*c* 1.12, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.33 (t, *J* = 7.8 Hz, 2H), 7.31 (d, *J* = 7.8 Hz, 2H), 7.21 (t, *J* = 7.8 Hz, 1H), 5.67 (ddt, *J* = 16.2, 10.2, 7.2 Hz, 1H), 5.10 (s, 1H), 4.97 (s, 1H), 4.94 (dd, *J* = 7.2, 1.2 Hz, 1H), 4.84 (s, 1H), 3.80 (s, 3H), 3.00 (s, 1H), 2.99 (dt, *J* = 10.8, 7.2 Hz, 1H), 2.81 (ddd, *J* = 14.4, 10.8, 1.2 Hz, 1H), 2.67 (d, *J* = 12.0 Hz, 1H), 2.44 (dddd, *J* = 12.0, 11.4, 5.4, 4.8 Hz, 1H), 2.05-2.14 (m, 2H), 2.01 (dd, *J* = 14.4, 7.8 Hz, 1H), 1.73 (s, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): 176.7, 144.5, 141.0, 134.9, 128.4, 128.1, 126.3, 117.1, 115.1, 81.1, 60.1, 52.7, 49.1, 47.5, 46.5, 33.6, 23.5. FTIR (neat): ν_{max}/cm^{-1} 3517, 3074, 2950, 2919, 1725, 1632, 1435. HRMS (p-APCI): *m/z* 283.1690 [(M-OH)$^+$ requires 283.1693]. HPLC analysis: 76% ee, CHIRALCEL ODR, 0.5% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 10.68 min (major), 21.29 min (minor).



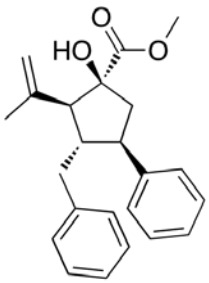
(+)-(1*S*,2*S*,3*S*,4*R*)-methyl 1-hydroxy-3-(3-oxobutyl)-4-phenyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (7f).

Prepared by general procedure with methyl styryldiazoacetate (**1a**) (223 mg, 1.1 mmol, 1.0 equiv) and 5-methyl-1-(2-methyl-1,3-dioxolan-2-yl)4-hexen-3-ol (**2f**) (201 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (5:1), and gave compound **7f** as a colorless oil (149 mg, 45%). $[\alpha]^{20}_{D}$ 32.7^{o} (*c* 0.50, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.34 (dd, *J* = 7.2, 1.2 Hz, 2H), 7.30 (t, *J* = 7.2 Hz, 2H), 7.21 (dt, *J* = 7.2, 1.2 Hz, 1H), 5.10 (s, 1H), 4.89 (s, 1H), 3.80 (s, 3H), 3.00 (s, 1H), 2.82-2.91(m, 2H), 2.62 (d, *J* = 12.6, 1H), 2.43 (dtd, *J* = 12.0, 7.8, 4.2 Hz, 1H), 2.23 (ddd, *J* = 17.4, 9.0, 7.2 Hz, 1H), 2.12 (ddd, *J* = 17.4, 9.0, 4.8 Hz, 1H), 1.94 (dd, *J* = 13.8, 6.9 Hz), 1.81 (s, 3H), 1.76-1.81 (m, 1H), 1.76 (s, 3H), 1.50 (m, 1H). ^{13}C NMR (150 MHz, $CDCl_3$): 208.6, 176.4, 145.1, 141.2, 128.6, 127.9, 126.4, 115.3, 81.3, 62.1, 52.7, 50.0, 48.0, 47.4, 40.7, 29.6, 26.1, 23.3. FTIR (neat): ν_{max}/cm^{-1} 3514, 3063, 3027, 2951, 2928, 1728, 1714, 1638, 1602, 1494, 1436. HRMS (p-APCI): *m/z* 313.1800 [$(M\text{-}OH)^{+}$ requires 313.1798]. HPLC analysis: 90% ee, CHIRALCEL ODR, 1.0% isopropanol/hexanes, 1.0 mL/min, UV: 210 nm, t_R: 11.01 min (major), 35.53 min (minor).



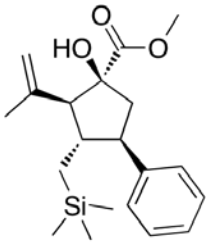
()-(1*S*,2*S*,3*S*,4*R*)-methyl 3-(((*tert*-butyldimethylsilyl)oxy)methyl)-1-hydroxy-4-phenyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (7g).

Prepared by general procedure, in the absence of scandium(III) triflate, with methyl styryldiazoacetate (**1a**) (225 mg, 1.1 mmol, 1.0 equiv) and 1-((*tert*-butyldimethylsilyl)oxy)-4-methyl-3-penten-2-ol (**2g**) (230 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **7g** as a colorless oil (263 mg, 65%). $[\alpha]^{20}_{D}$ -5.4^{o} (*c* 1.10, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.32 (m, 5H), 5.09 (s, 1H), 4.81 (s, 1H), 3.82 (s, 3H), 3.51 (dd, *J* = 10.2, 2.4 Hz, 1H), 3.46 (dd, *J* = 10.2, 2.4 Hz, 1H), 3.35 (dt, *J* = 10.2, 7.8 Hz, 1H), 3.04 (d, *J* = 10.2 Hz, 1H), 3.04 (s, 1H), 2.84 (dd, *J* = 14.4, 10.8 Hz, 1H), 2.35 (dt, *J* = 10.8, 2.4 Hz, 1H), 2.07 (dd, *J* = 14.4, 7.8 Hz, 1H), 1.75 (s, 3H), 0.92 (s, 9H), 0.02 (s, 3H), -0.01 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 176.8, 144.6, 141.2, 128.4, 128.1, 126.2, 114.6, 81.2, 59.3, 56.8, 52.6, 52.3, 45.9, 43.9, 25.8, 23.6, 18.2, -5.6, -5.7. FTIR (neat): ν_{max}/cm^{-1} 3523.4, 3023.5, 2949.8, 2922.7, 2853, 1729.2, 1457.9. HRMS (p-APCI): *m/z* 405.2462 [$(M+H)^+$ requires 405.2456]. HPLC analysis: 76% ee, CHIRALCEL ODR, 0.5% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 8.36 min (major), 10.26 min (minor).



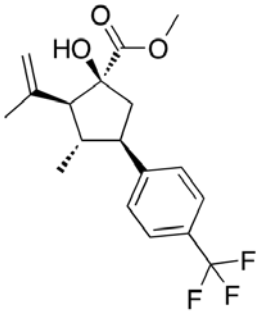
(-)-(1*S*,2*S*,3*S*,4*R*)-methyl 3-benzyl-1-hydroxy-4-phenyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (7h).

Prepared by general procedure with methyl styryldiazoacetate (**1a**) (225 mg, 1.1 mmol, 1.0 equiv) and 4-methyl-1-phenyl-3-penten-2-ol (**2h**) (178 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **7h** as a white solid (147 mg, 42%). MP = 62-64 ^{o}C. $[\alpha]^{20}_{D}$ -34.7^{o} (*c* 1.15, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.32 (d, *J* = 7.2 Hz, 2H), 7.28 (t, *J* = 7.8 Hz, 1H), 7.16-7.19 (m, 2H), 7.13 (t, *J* = 7.2 Hz, 1H), 6.98 (d, *J* = 7.2 Hz, 2H), 5.12 (s, 1H), 4.88 (s, 1H), 3.72 (s, 3H), 2.99 (s, 1H), 2.92 (dt, *J* = 10.8, 7.8 Hz, 1H), 2.67-2.77 (m, 3H), 2.61 (dd, *J* = 14.1, 5.1 Hz, 1H), 2.55 (d, *J* = 12.0 Hz, 1H), 1.90 (dd, *J* = 14.7, 7.2 Hz, 1H), 1.69 (s, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 176.5, 144.4, 141.0, 138.5, 130.0, 128.4, 128.2, 127.8, 126.2, 125.8, 115.5, 81.0, 60.1, 52.6, 49.7, 47.2, 46.7, 35.4, 23.3. FTIR (neat): ν_{max}/cm^{-1} 3524, 3062, 3027, 2951, 2922, 2851, 1731, 1602, 1495,1454, 1438, 1231. HRMS (p-APCI): *m/z* 351.1958 [$(M+H)^+$ requires 351.1955]. HPLC analysis: 92% ee, CHIRALCEL ODR, 0.5% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 15.80 min (major), 23.47 min (minor).



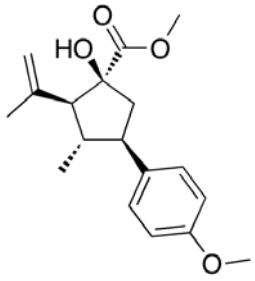
(-)-(1*S*,2*S*,3*S*,4*R*)-methyl 1-hydroxy-4-phenyl-2-(prop-1-en-2-yl)-3-((trimethylsilyl)methyl)cyclopentanecarboxylate (7i).

Prepared by general procedure with methyl styryldiazoacetate (**1a**) (229 mg, 1.1 mmol, 1.0 equiv) and 4-methyl-1-(trimethylsilyl)-3-penten-2-ol (**2i**) (176 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **7i** as a colorless oil (204 mg, 59%). $[\alpha]^{20}_{D}$ -0.6^{o} (*c* 1.73, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.29-7.35 (m, 4H), 7.21 (tt, *J* = 7.2, 1.2 Hz, 1H), 5.13 (s, 1H), 4.85 (s, 1H), 3.81 (s, 3H), 2.98 (s, 1H), 2.82-2.89 (m, 2H), 2.87 (d, *J* = 12.0 Hz, 1H), 2.57 (dddd, *J* = 15.0, 10.2, 7.2, 3.0 Hz, 1H), 1.93 (m, 1H), 1.75 (s, 3H), 0.74 (dd, *J* = 15.0, 3.0 Hz, 1H), 0.61 (dd, *J* = 15.0, 7.8 Hz, 1H), -0.25 (s, 9H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 176.8, 144.4, 141.3, 128.4, 128.2, 126.4, 115.3, 80.9, 64.6, 52.6, 52.1, 47.8, 45.7, 23.6, 19.3, -0.4. FTIR (neat): ν_{max}/cm^{-1} 3523, 3064, 3028, 2951, 2895, 1729, 1638, 1602, 1495, 1455, 1436, 1246, 1201. HRMS (p-APCI): *m/z* 347.2041 [$(M+H)^+$ requires 347.2037]. HPLC analysis: 83% ee, CHIRALCEL ODR, 0.5% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 10.24 min (major), 26.93 min (minor).



(+)-(1*S*,2*S*,3*S*,4*R*)-methyl 1-hydroxy-3-methyl-2-(prop-1-en-2-yl)-4-(4-(trifluoromethyl)phenyl)cyclopentanecarboxylate (9a).

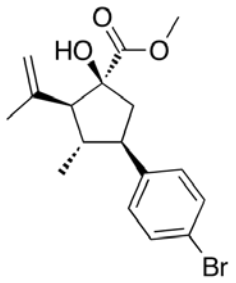
Prepared by general procedure with methyl 4-(trifluoromethyl)styryldiazoacetate (**8a**) (315 mg, 1.1 mmol, 1.0 equiv) and 4-methyl-3-penten-2-ol (**2a**) (102 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (10:1), and gave compound **9a** as a white solid (215 mg, 63%). MP = 71-75 ^{o}C. $[\alpha]^{20}_{D}$ 4.4^{o} (*c* 1.03, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.56 (d, *J* = 7.5 Hz, 2H), 7.44 (d, *J* = 7.5 Hz, 2H), 5.09 (s, 1H), 4.80 (s, 1H), 3.81 (s, 3H), 3.05(s, 1H), 2.81-2.89 (m, 2H), 2.55 (d, *J* = 12.0 Hz, 1H), 2.30 (m, 1H), 2.02 (ddd, *J* = 13.8, 10.2, 7.8 Hz, 1H), 1.73 (s, 3H), 0.89 (d, *J* = 6.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.5, 148.5, 140.8, 128.2, 125.4, 125.3, 125.3, 114.9, 81.4, 63.6, 52.7, 51.4, 46.4, 45.1, 23.3, 15.9. FTIR (neat): ν_{max}/cm^{-1} 3519, 2955, 1731, 1640, 1618, 1438, 1323, 1120, 1067. HRMS (p-APCI): *m/z* 343.1516 [$(M+H)^+$ requires 343.1516]. HPLC analysis: 78% ee, CHIRALCEL ODR, 1.0% isopropanol/hexanes, 1.0 mL/min, UV: 210 nm, t_R: 5.61 min (major), 7.25 min (minor).



(+)-(1*S*,2*S*,3*S*,4*R*)-methyl 1-hydroxy-4-(4-methoxyphenyl)-3-methyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (9b).

Prepared by general procedure with methyl 4-methoxystyryldiazoacetate (**8b**) (255 mg, 1.1 mmol, 1.0 equiv) and 4-methyl-3-penten-2-ol (**2a**) (101 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (4:1), and gave compound **9b** as a

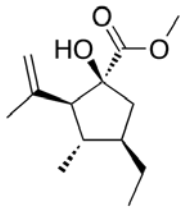
colorless oil (261 mg, 94%). $[\alpha]^{20}_{D}$ 4.3° (*c* 1.25, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.25 (d, *J* = 8.7 Hz, 2H), 6.86 (d, *J* = 8.7 Hz, 2H), 5.07 (s, 1H), 4.79 (s, 1H), 3.80 (s, 3H), 3.79 (s, 3H), 2.98 (s, 1H), 2.80 (dd, *J* = 14.1, 10.2 Hz), 2.70 (dd, *J* = 18.6, 10.2 Hz, 1H), 2.51 (d, *J* = 12.0 Hz, 1H), 2.23 (ddq, *J* = 12.6, 10.4, 6.4 Hz, 1H), 1.98 (dd, *J* = 14.1, 8.2 Hz, 1H), 1.72 (s, 3H), 0.87 (d, *J* = 6.0 Hz, 3H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 176.9, 158.1, 141.2, 136.0, 128.7, 114.7, 113.8, 81.1, 63.5, 55.2, 52.6, 50.9, 46.8, 44.8, 23.3, 16.0. FTIR (neat): ν_{max}/cm^{-1} 3518, 2952, 2835, 1729, 1611, 1512, 1439, 1243,1178, 1036. HRMS (p-APCI): *m/z* 305.1748 $[(M+H)^+$ requires 305.1747]. HPLC analysis: 87% ee, CHIRALCEL ODR, 1.0% isopropanol/hexanes, 1.0 mL/min, UV: 210 nm, t_R: 7.55 min (minor), 11.12 min (major).



(+)-(1*S*,2*S*,3*S*,4*R*)-methyl 4-(4-bromophenyl)-1-hydroxy-3-methyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (9c).

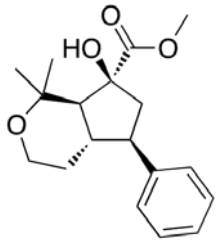
Prepared by general procedure with methyl 4-bromostyryldiazoacetate (**8c**) (319 mg, 1.1 mmol, 1.0 equiv) and 4-methyl-3-penten-2-ol (**2a**) (101 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **9c** as a white solid (171 mg, 48%). MP = 71-72 °C. $[\alpha]^{20}_{D}$ 1.8° (*c* 1.05, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.43 (d, *J* = 8.7 Hz, 2H), 7.20 (d, *J* = 8.7 Hz, 2H), 5.08 (s, 1H), 4.79 (s, 1H), 3.81 (s, 3H), 3.00 (s, 1H), 2.83 (dd, *J* = 14.1, 10.5 Hz, 1H), 2.72 (td, *J* = 10.5, 8.1 Hz, 1H), 2.56 (d, *J* = 6.0 Hz, 1H), 2.24 (tq, *J* = 6.6, 6.0 Hz, 1H), 1.96 (dd, *J* = 14.1, 8.1 Hz, 1H), 1.72 (s, 3H), 0.87 (d, *J* = 6.6 Hz,

3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 176.6, 143.2, 141.0, 131.5, 129.6, 120.0, 114.9, 81.2, 63.5, 52.7, 51.1, 46.5, 44.9, 23.3, 15.9. FTIR (neat): ν_{max}/cm^{-1} 3521, 3072, 2952, 2924, 2868, 1729, 1639, 1487, 1436, 1010. HRMS (p-APCI): *m/z* 335.0640 [(M-OH)$^+$ requires 335.0641]. HPLC analysis: 92% ee, CHIRALCEL ADH, 1.0% isopropanol/hexanes, 1.0 mL/min, UV: 230 nm, t_R: 13.20 min (major), 14.34 min (minor).



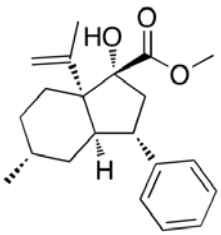
(-)-(1*S*,2*S*,3*S*,4*R*)-methyl 4-ethyl-1-hydroxy-3-methyl-2-(prop-1-en-2-yl)cyclopentanecarboxylate (9d).

Prepared by general procedure with (*E*)-methyl 2-diazo-3-hexenoate (**8d**) (174 mg, 1.1 mmol, 1.0 equiv) and 4-methyl-3-penten-2-ol (**2a**) (102 mg, 1.0 mmol, 1.0 equiv) at rt. The crude was purified on silica gel eluting with hexanes : ethyl acetate (9:1), and gave compound **9d** as a colorless oil (142 mg, 63%). $[\alpha]^{20}_D$ -0.4^o (*c* 2.03, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 5.04 (s, 1H), 4.75 (s, 1H), 3.77 (s, 3H), 2.80 (s, 1H), 2.54 (dd, *J* = 13.2, 9.0 Hz, 1H), 2.38 (d, *J* = 12.0 Hz, 1H), 1.81-1.87 (m, 1H), 1.68 (s, 3H), 1.64-1.70 (m, 1H), 1.49-1.55 (m, 2H), 1.20-1.27 (m, 1H), 0.95 (d, *J* = 6.6 Hz, 3H), 0.91 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (150 MHz, $CDCl_3$): δ 177.3, 141.6, 114.4, 81.1, 63.8, 52.5, 46.6, 44.0, 41.6, 27.1, 23.4, 16.7, 12.5. FTIR (neat): ν_{max}/cm^{-1} 3526, 3072, 2956, 2926, 2874, 1731, 1639, 1457, 1437, 1233, 1077. HRMS (p-APCI): *m/z* 227.1640 [(M+H)$^+$ requires 227.1642]. HPLC analysis: 64% ee, CHIRALCEL ADH, 1.0% isopropanol/hexanes, 0.5 mL/min, UV: 210 nm, t_R: 22.09 min (minor), 22.03 min (major).



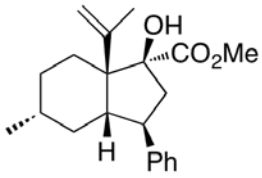
(+)-(4*aS*,5*R*,7*S*,7*aR*)-methyl 7-hydroxy-1,1-dimethyl-5-phenyloctahydrocyclopenta[*c*]pyran-7-carboxylate (12).

Prepared by general procedure with methyl styryldiazoacetate (**1a**) (227 mg, 1.1 mmol, 1.0 equiv) and 1-((*tert*-butyldimethylsilyl)oxy)-5-methyl-4-hexen-3-ol (**11**) (244 mg, 1.0 mmol, 1.0 equiv). The crude was purified on silica gel eluting with hexanes : ethyl acetate (4:1), and gave compound **12** as a white solid (141 mg, 46%). MP = 94-96 ^{o}C. $[\alpha]^{20}_{D}$ 14.0^{o} (*c* 1.07, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.29-7.33 (m, 4H), 7.21-7.23 (m, 1H), 3.85 (s, 3H), 3.69 (ddd, *J* = 12.0, 5.4, 1.2 Hz, 1H), 3.51 (td, *J* = 12.0, 2.4 Hz, 1H), 3.34 (s, 1H), 2.71 (m, 2H), 2.21 (m, 1H), 1.94 (d, *J* = 13.2 Hz, 1H), (1.91 (dd, *J* = 18.9, 12.9 Hz, 1H), 1.74 (dddd, *J* = 12.6, 1.2, 1.2, 1.2 Hz, 1H), 1.39 (qd, *J* = 12.6, 5.4 Hz), 1.33 (s, 3H), 1.13 (s, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 178.2, 143.5, 128.5, 127.6, 126.4, 80.3, 74.9, 60.8, 60.2, 53.0, 50.4, 48.8, 44.5, 32.5, 28.8, 21.1. FTIR (neat): ν_{max}/cm^{-1} 3510, 3026, 2974, 2929, 2861, 1426, 1601, 1436, 1202, 1099. HRMS (p-APCI): *m/z* 305.1748 [$(M+H)^+$ requires 305.1747]. HPLC analysis: 80% ee, CHIRALCEL ODR, 1.0% isopropanol/hexanes, 01.0 mL/min, UV: 210 nm, t_R: 9.63 min (major), 23.30 min (minor).



(-)-(1*R*,3*S*,3*aR*,5*R*,7*aR*)-methyl 1-hydroxy-5-methyl-3-phenyl-7a-(prop-1-en-2-yl)octahydro-1*H*-indene-1-carboxylate (14).

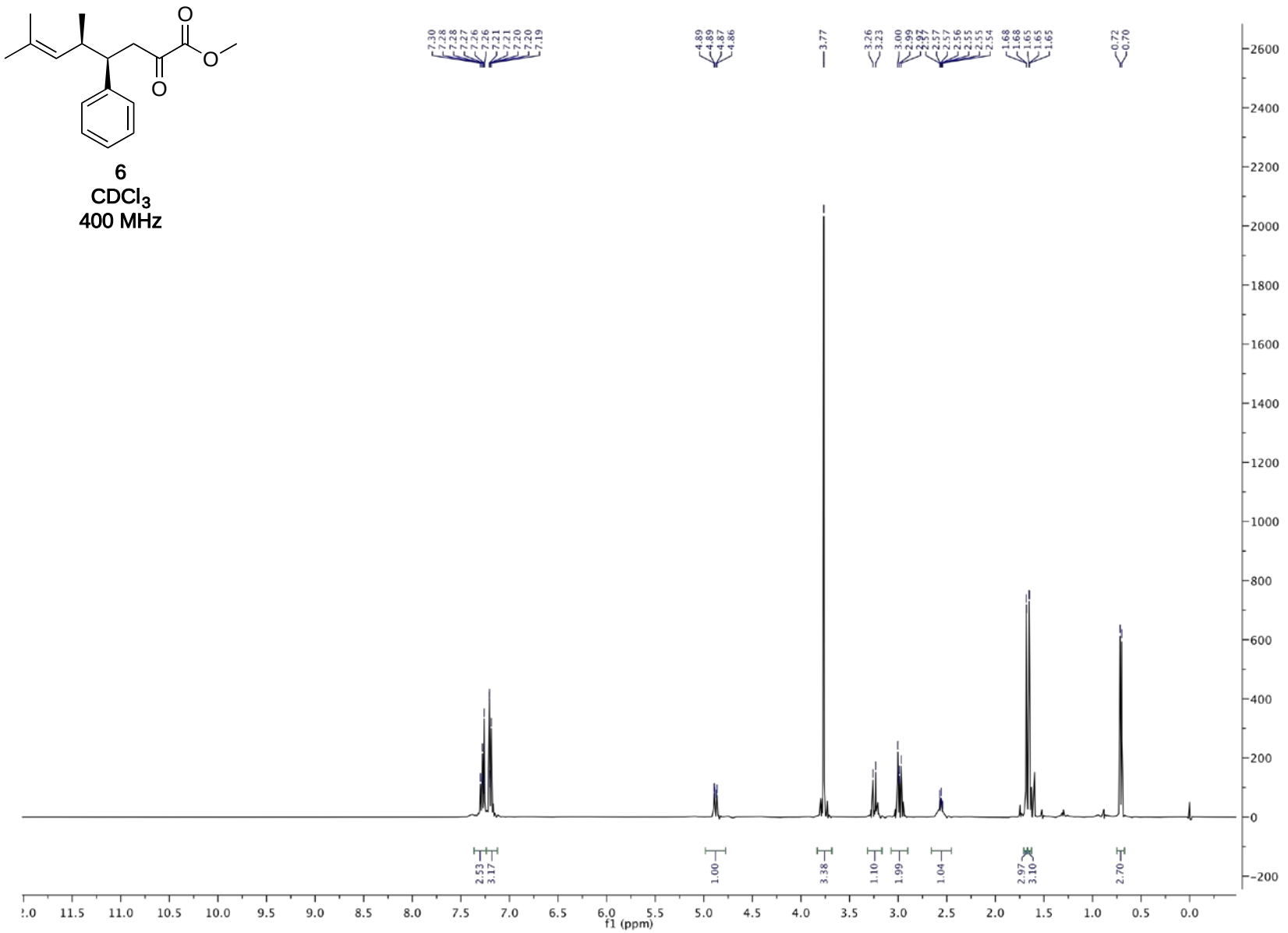
Prepared by general procedure, in the absence of scandium(III) triflate, with methyl styryldiazoacetate (**1a**) (229 mg, 1.1 mmol, 1.0 equiv), (-)-(*R*,*R*)-pulegol (**13**) (155 mg, 1.0 mmol, 1.0 equiv) and Rh_2(*R*-DOSP)$_4$. The crude was purified on silica gel eluting with hexanes : ethyl acetate (7:1), and gave compound **14** as a colorless oil (227 mg, 69%). $[\alpha]^{20}_D$ -5.4^o (*c* 1.10, $CHCl_3$). $[\alpha]^{20}_D$ -4.6^o (*c* 1.09, $CHCl_3$). ^{1}H NMR (600 MHz, $CDCl_3$): δ 7.37 (d, *J* = 6.9 Hz, 2H), 7.30 (t, *J* = 6.9 Hz, 2H), 7.20(t, *J* = 6.9 Hz, 1H), 5.34 (s, 1H), 4.99 (s, 1H), 3.75 (s, 3H), 3.35 (dt, *J* = 11.1, 5.4 Hz, 1H), 3.10 (ddd, *J* = 14.4, 11.7, 2.4 Hz, 1H), 2.96 (d, *J* = 2.4 Hz, 1H), 2.65 (m, 1H), 2.08 (dd, *J* = 14.7, 5.1 Hz, 1H), 1.70-1.81 (m, 2H), 1.68 (s, 3H), 1.59-1.64 (m, 2H), 1.41 (m, 1H), 0.99 (ddd, *J* = 17.7, 12.6, 5.4 Hz, 1H), 0.85 (d, *J* = 6.6 Hz, 3H), 0.77 (dtd, *J* = 16.2, 12.0, 3.0 Hz, 1H). ^{13}C NMR (100 MHz, $CDCl_3$): δ 174.5, 146.2, 143.0, 128.3, 128.2, 126.1, 117.8, 83.9, 59.3, 52.1, 50.0, 44.5, 43.0, 31.6, 30.6, 27.2, 26.4, 22.5, 21.5. FTIR (neat): ν_{max}/cm^{-1} 3540, 3085, 3026, 2950, 2912, 2868, 2847, 1727, 1629, 1604, 1494, 1447. HRMS (p-APCI): *m/z* 329.2113 [(M+H)$^+$ requires 329.2111].

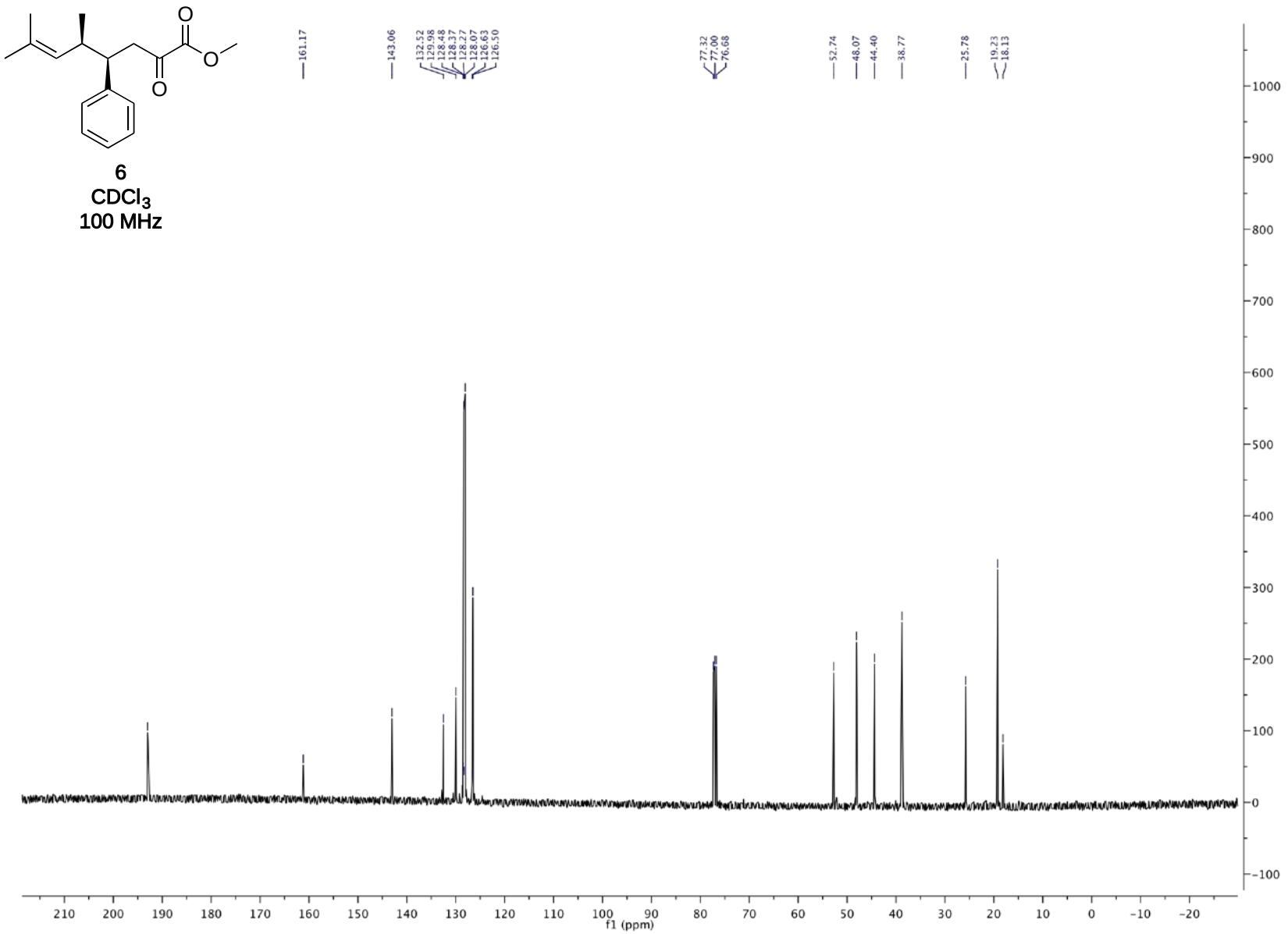


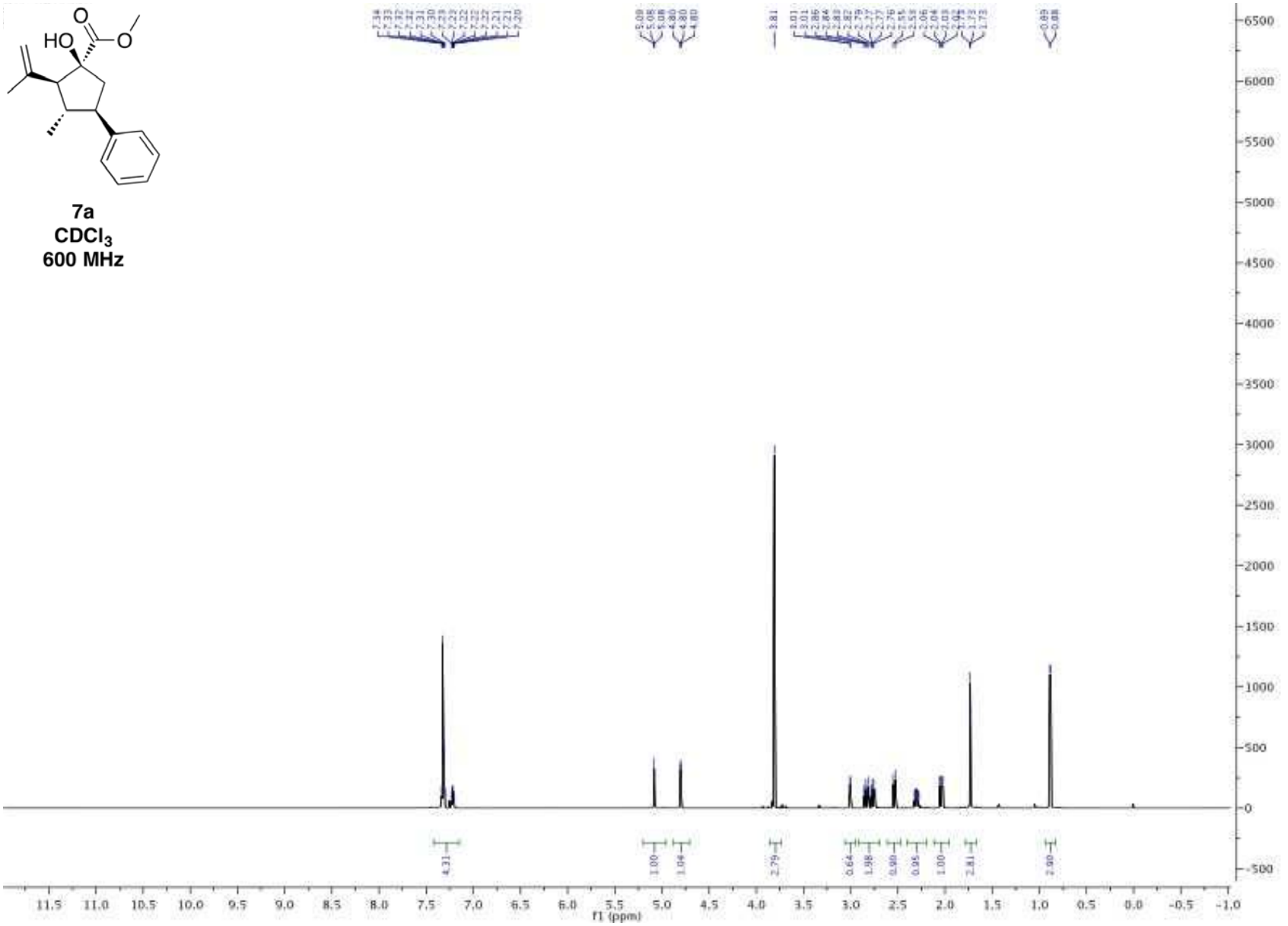
(1*S*,3*R*,3a*S*,5*R*,7*aS*)-methyl 1-hydroxy-5-methyl-3-phenyl-7*a*-(prop-1-en-2-yl)octahydro-1*H*-indene-1-carboxylate (15). Prepared by general procedure, in the absence of scandium(III) triflate, with methyl styryldiazoacetate (**1a**) (229 mg, 1.1 mmol, 1.0 equiv), (-)-(*R*,*R*)-pulegol (**13**) (155 mg, 1.0 mmol, 1.0 equiv) and Rh_2(*S*-DOSP)$_4$. The crude was purified on silica gel

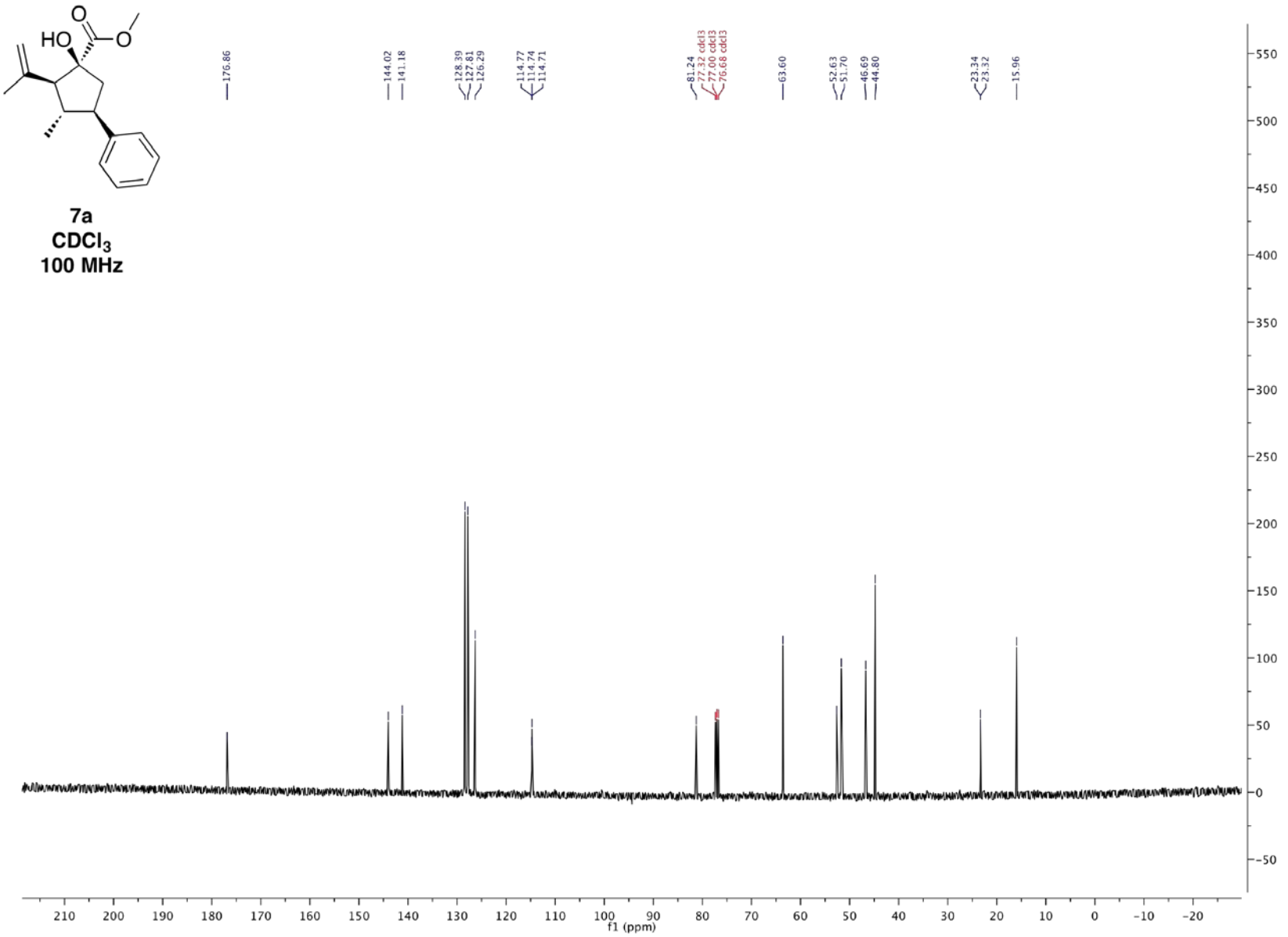
eluting with hexanes : ethyl acetate (7:1), and gave an inseparable mixture of compounds **14** and **15** (1 : 2.1) as a colorless oil (combined yield: 187 mg, 59%).

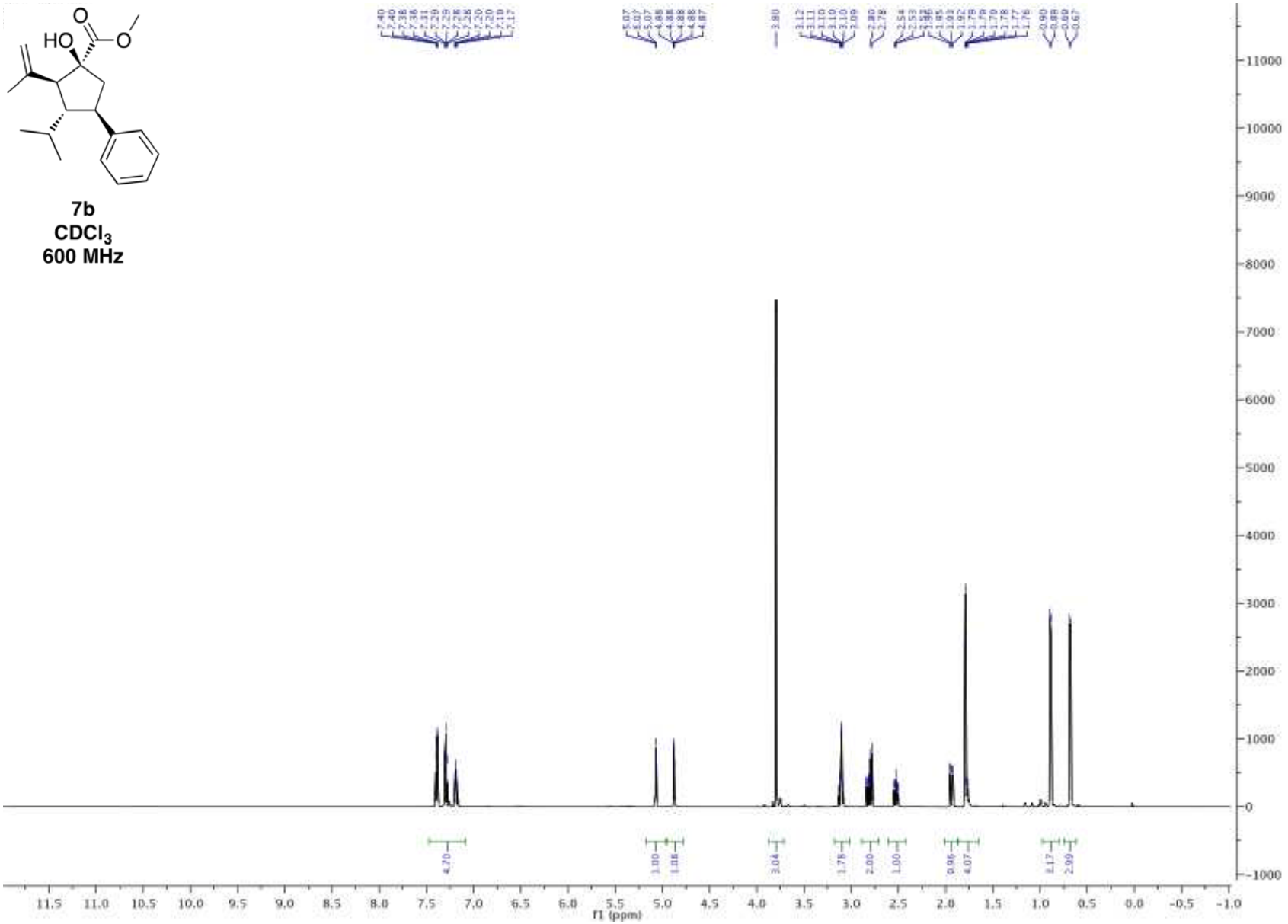
3. NMR Data for New Compounds

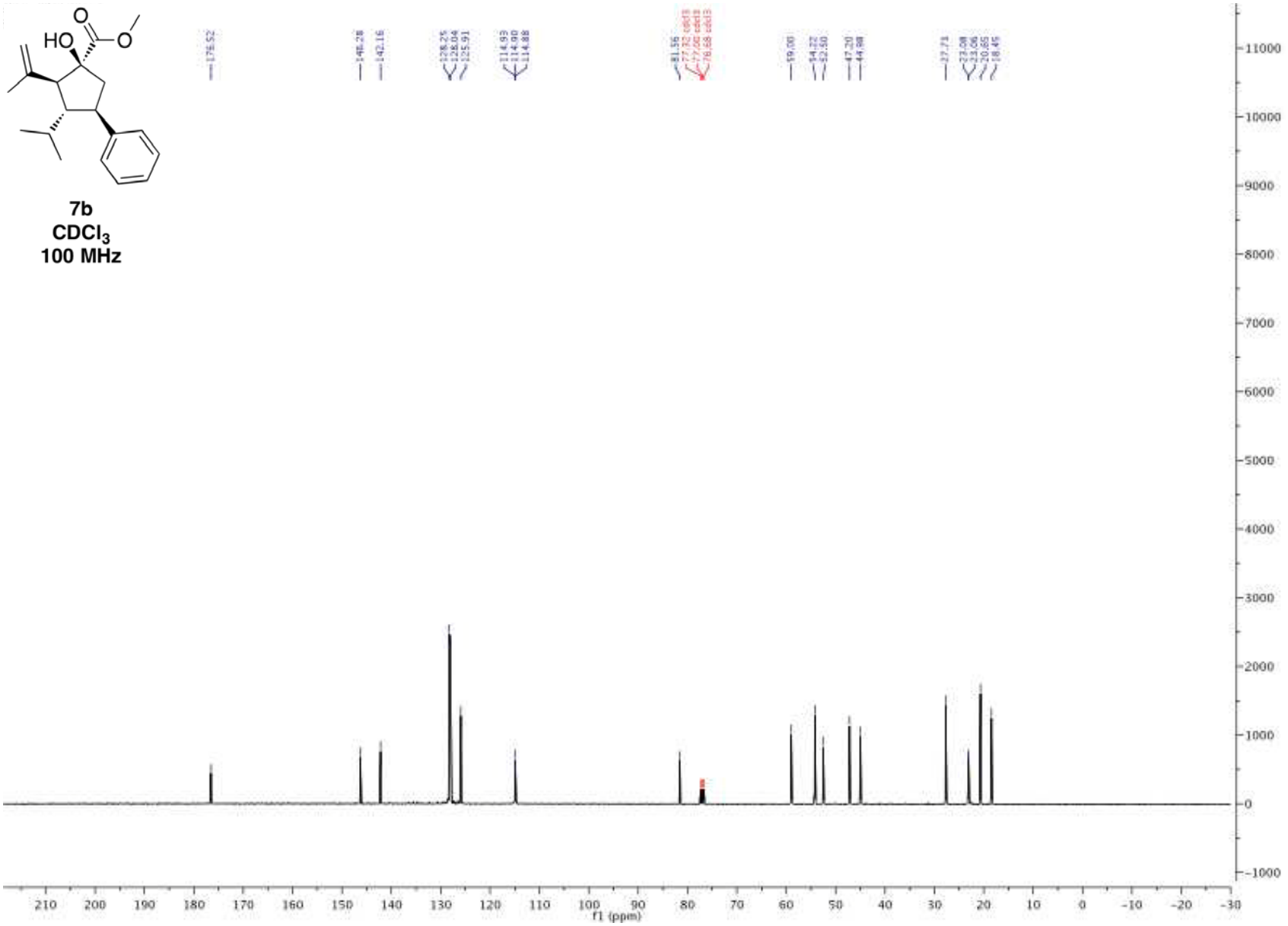

6
CDCl3
400 MHz
f1 (ppm)

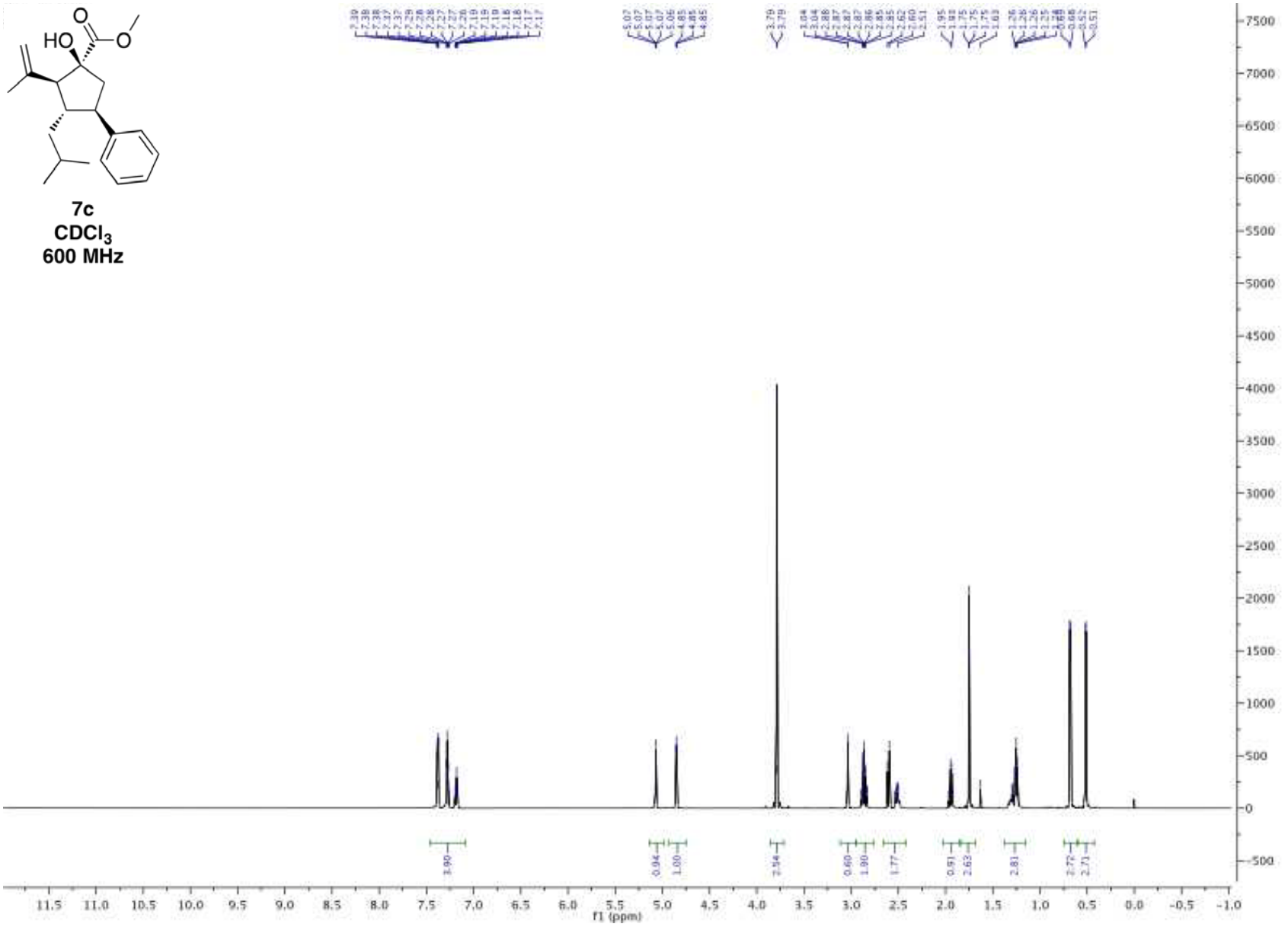


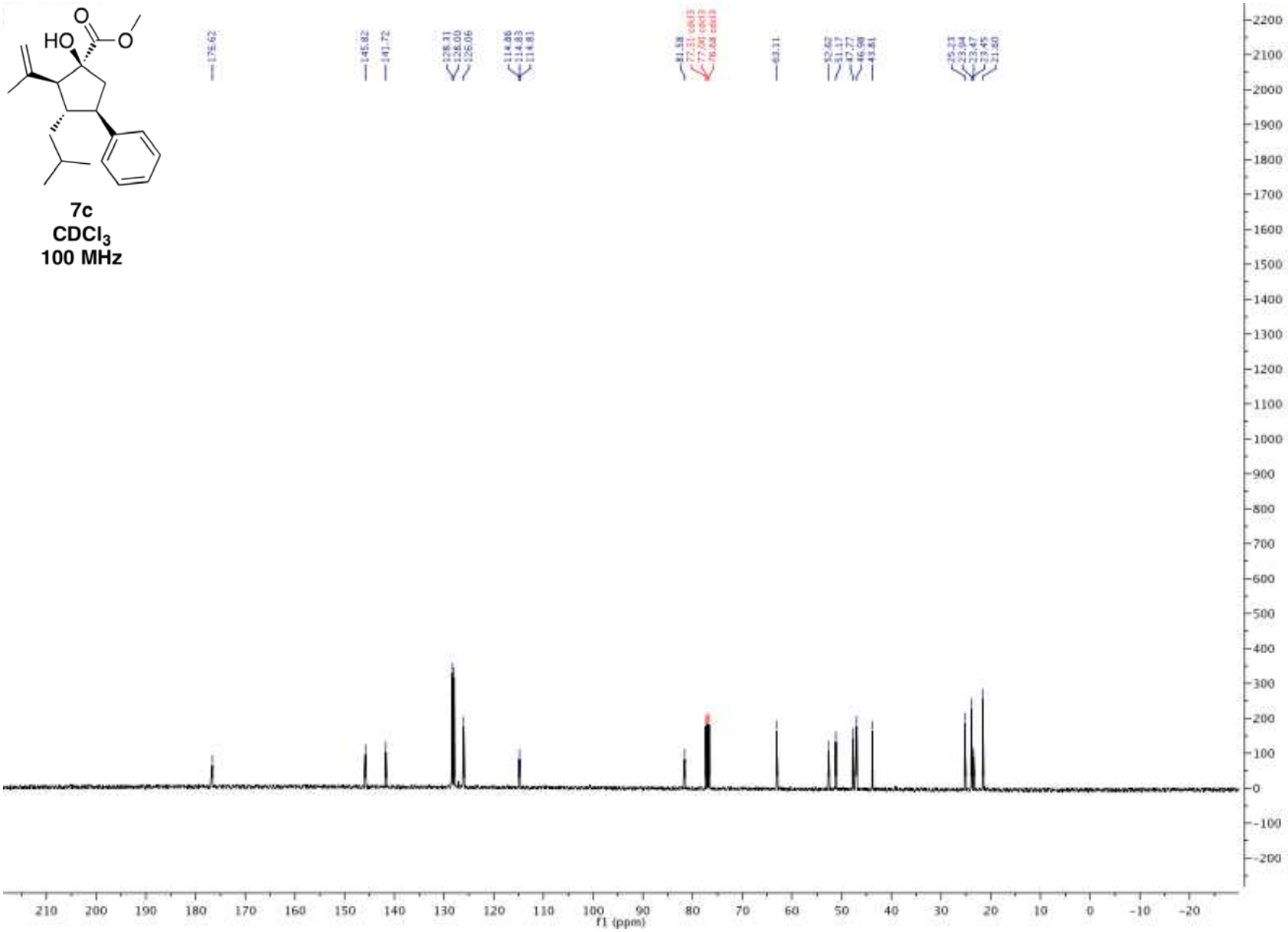


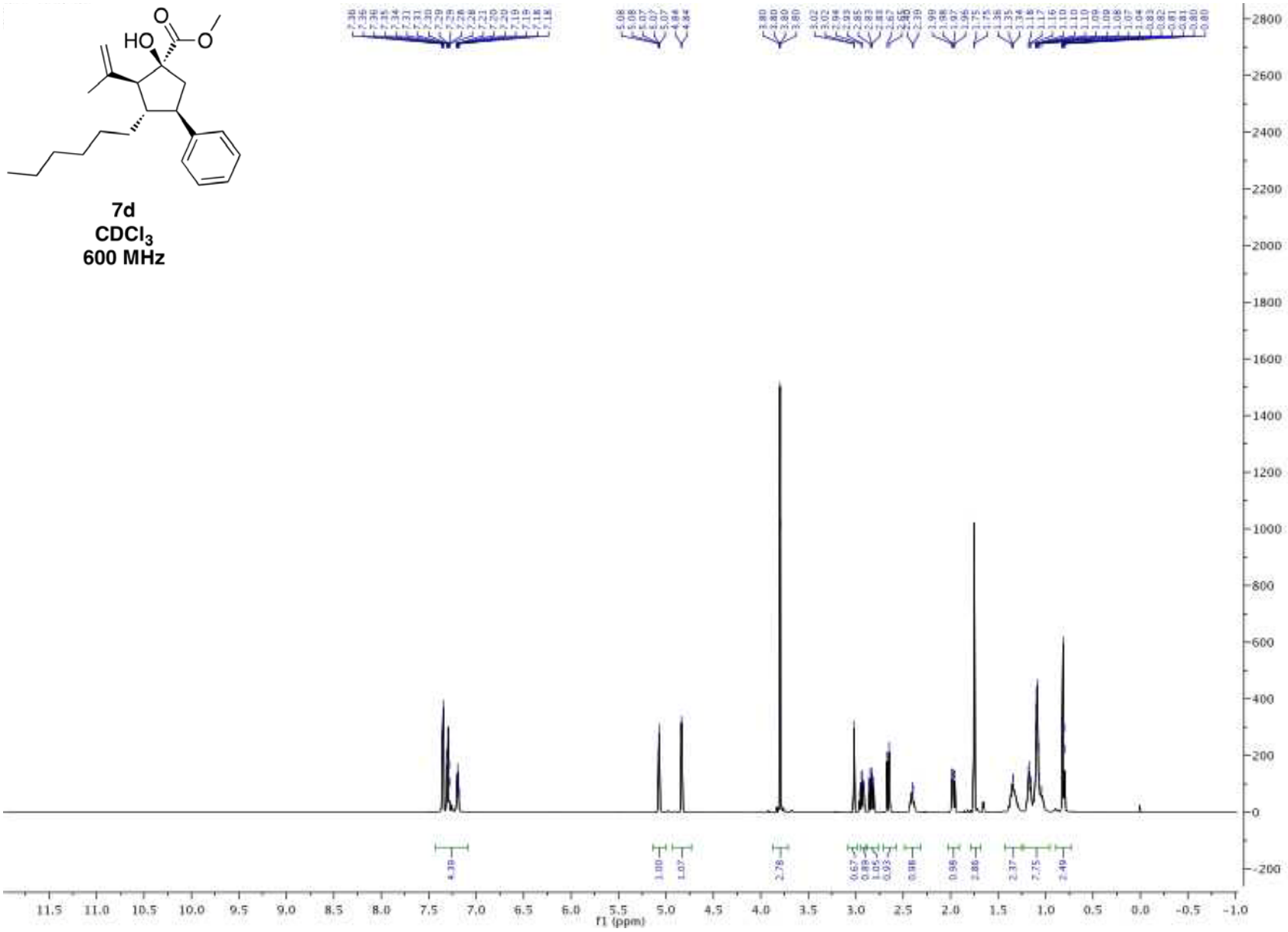


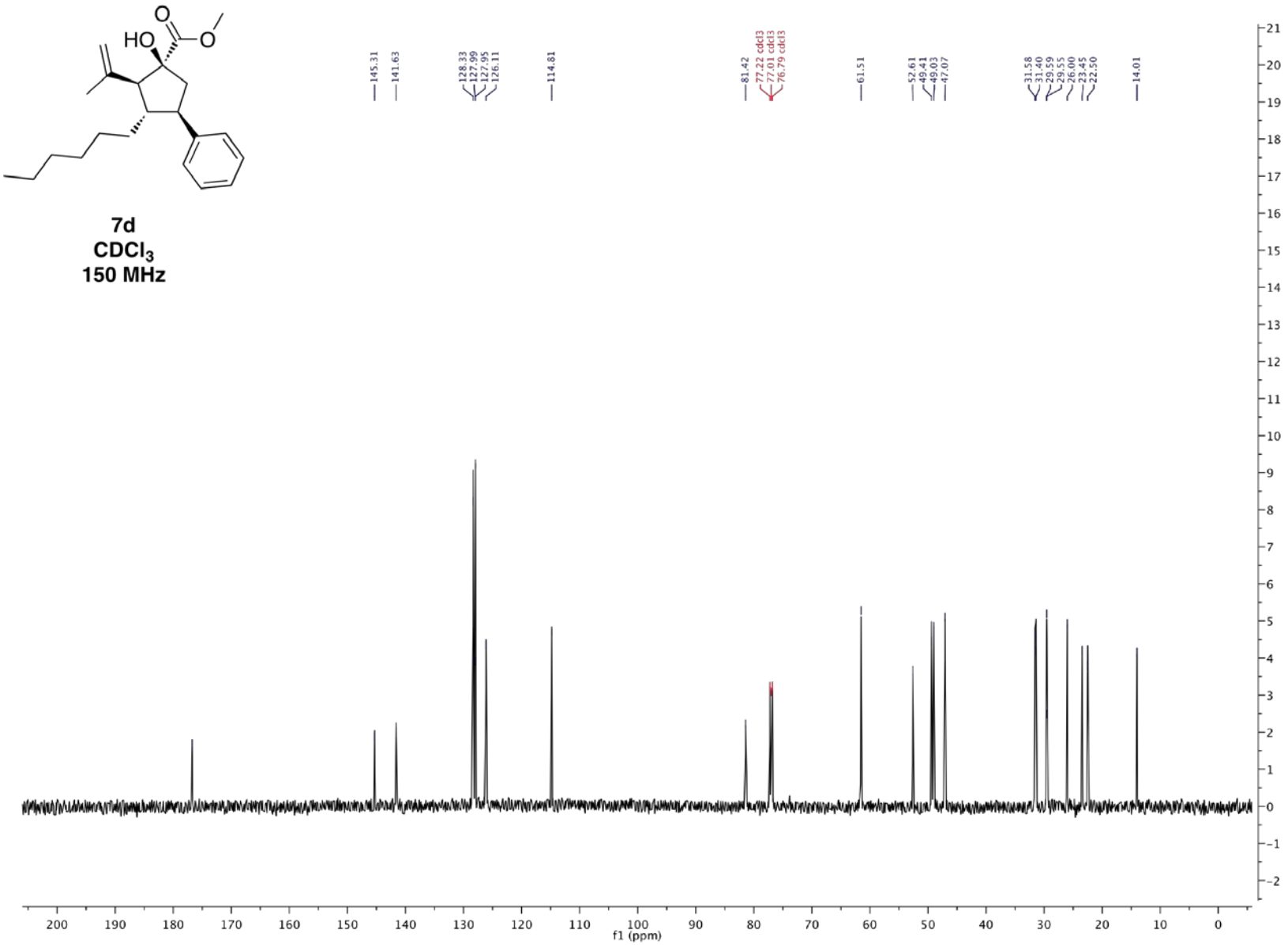


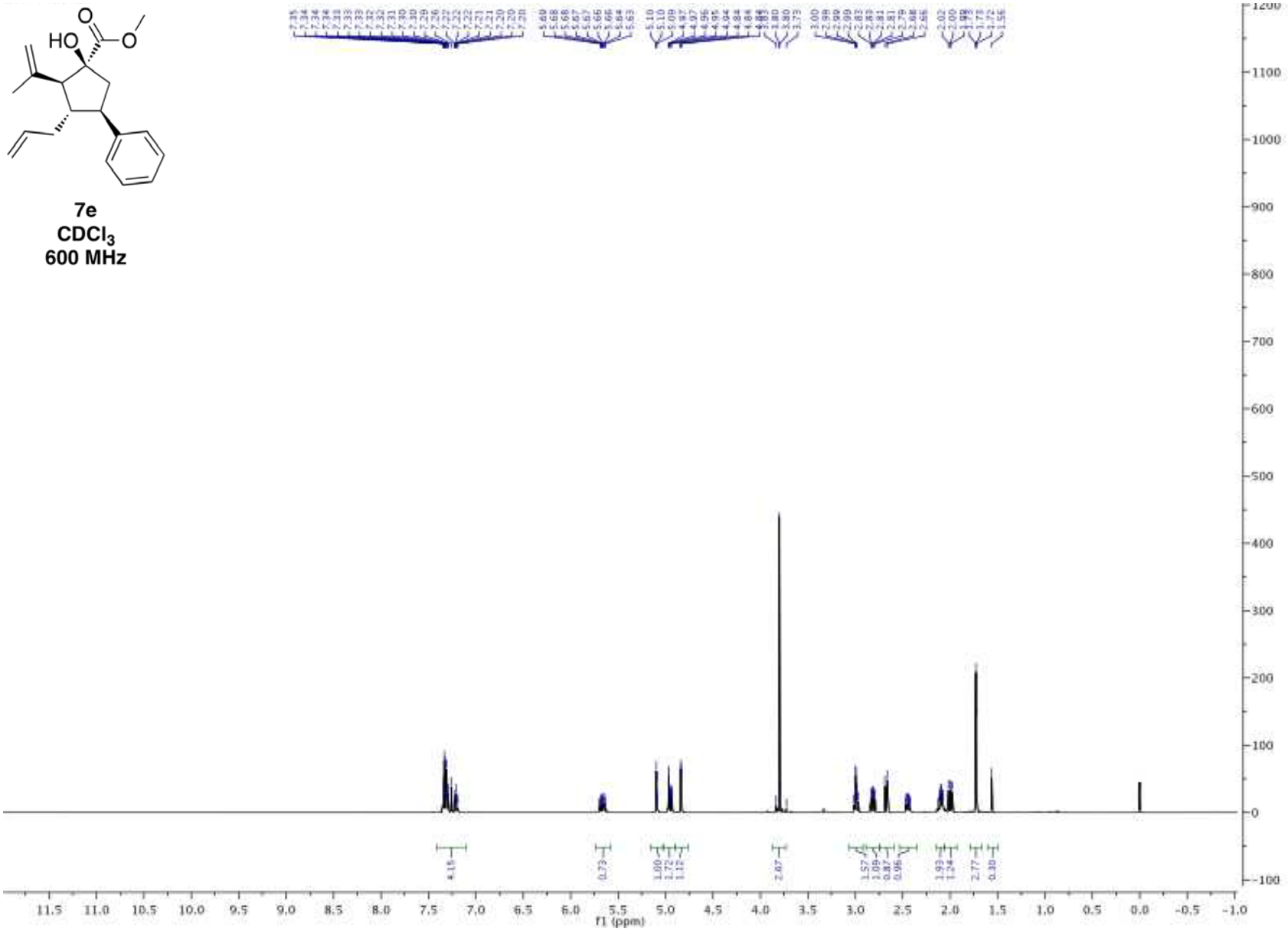


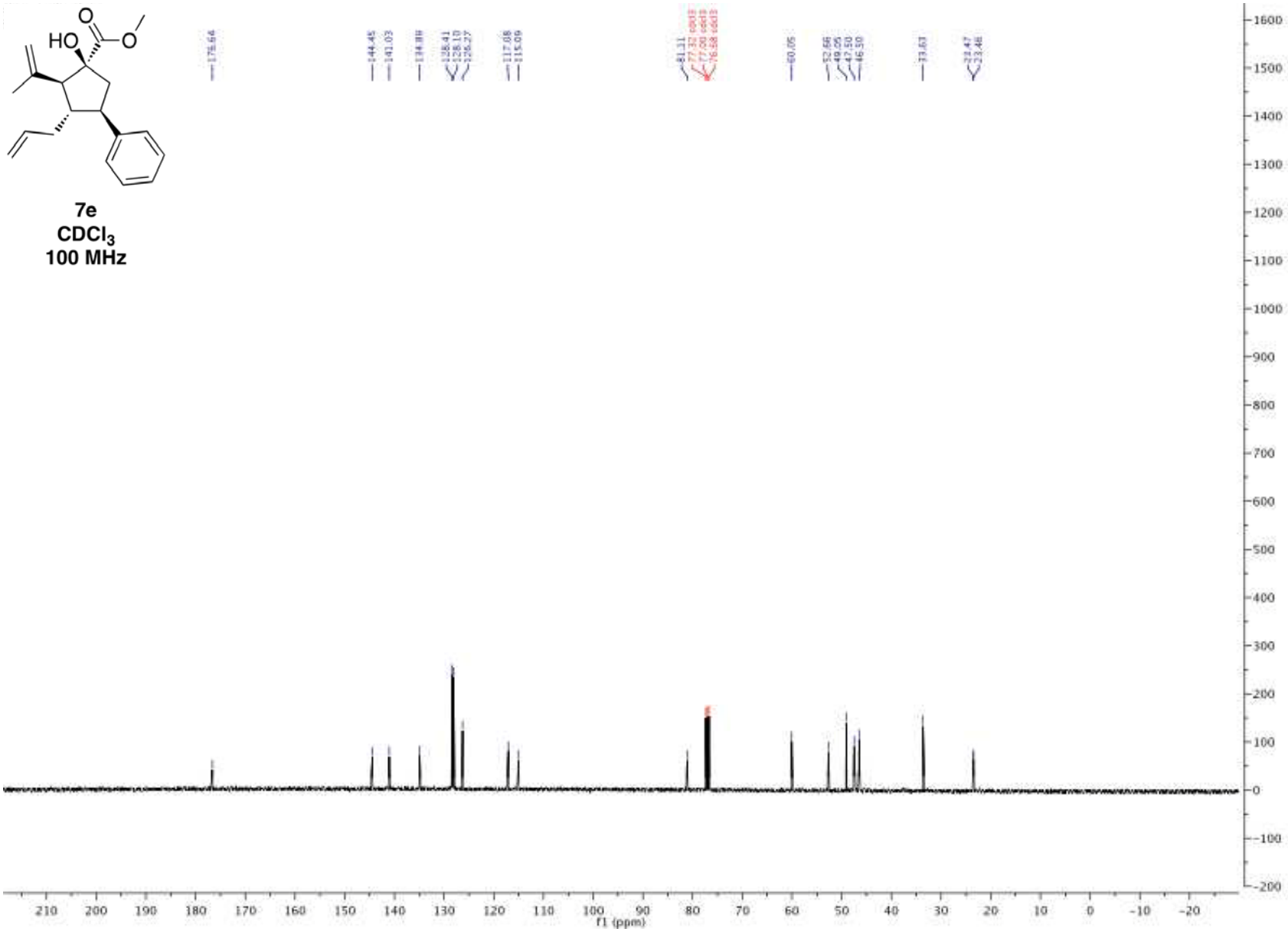


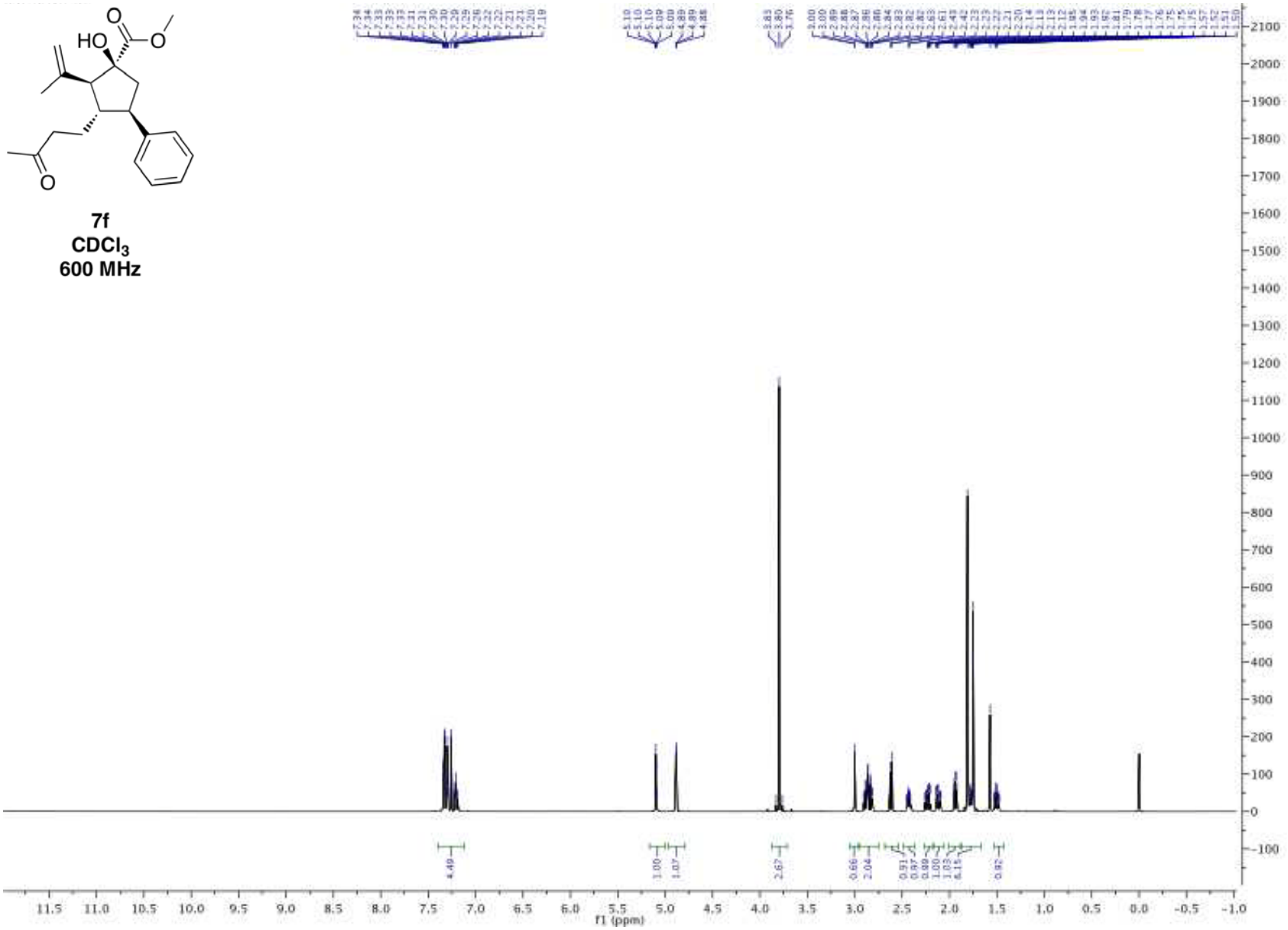


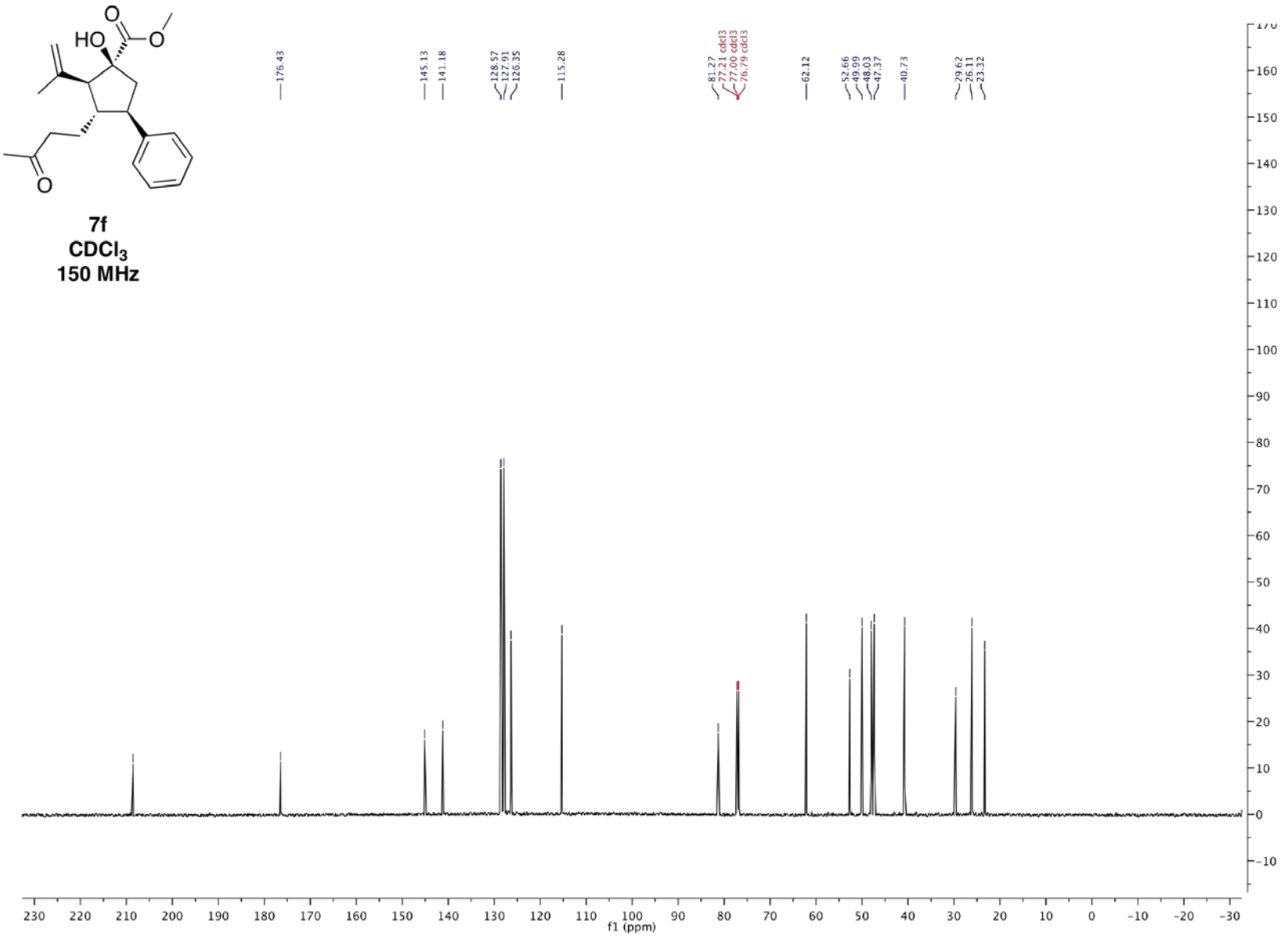


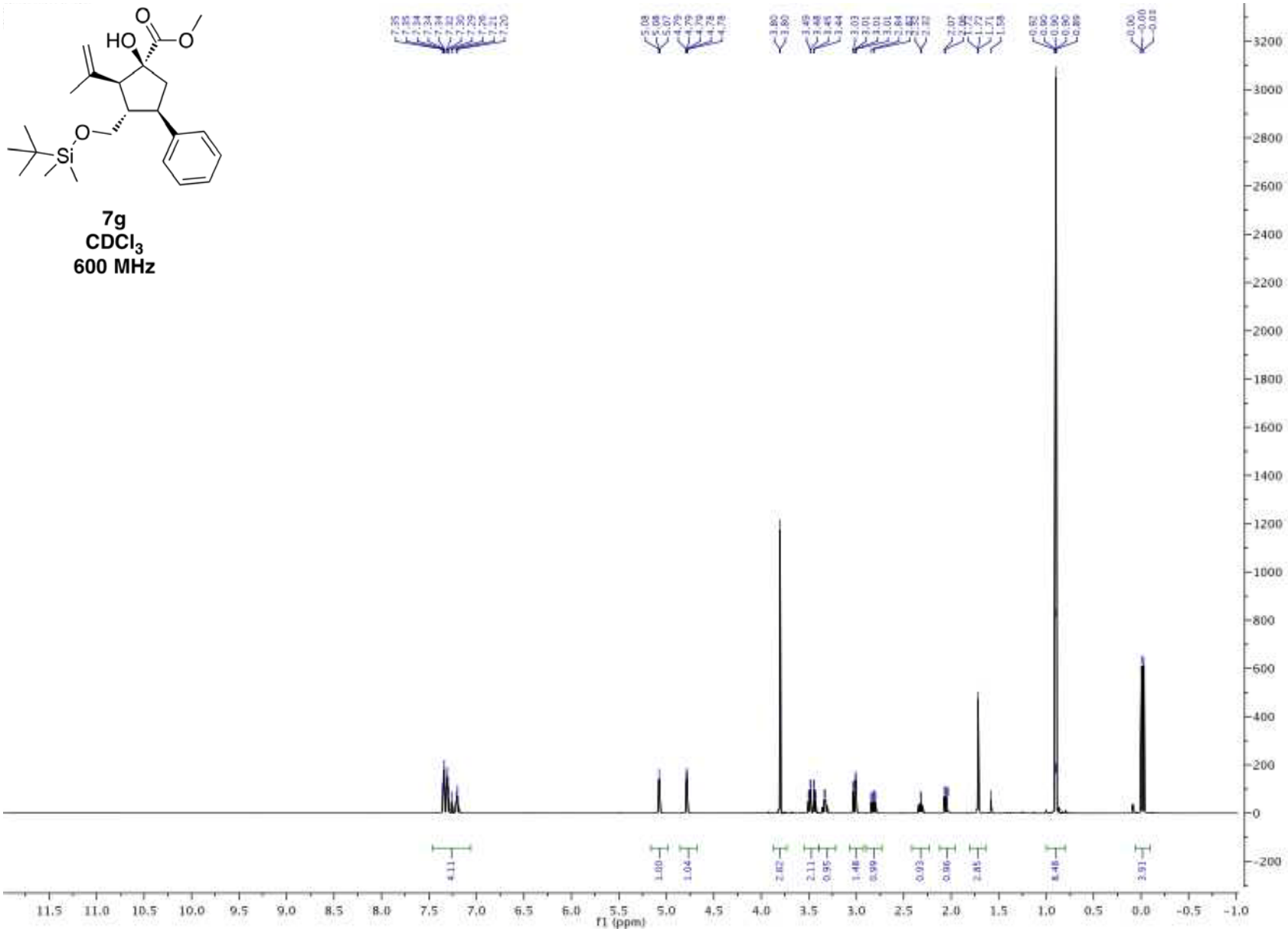


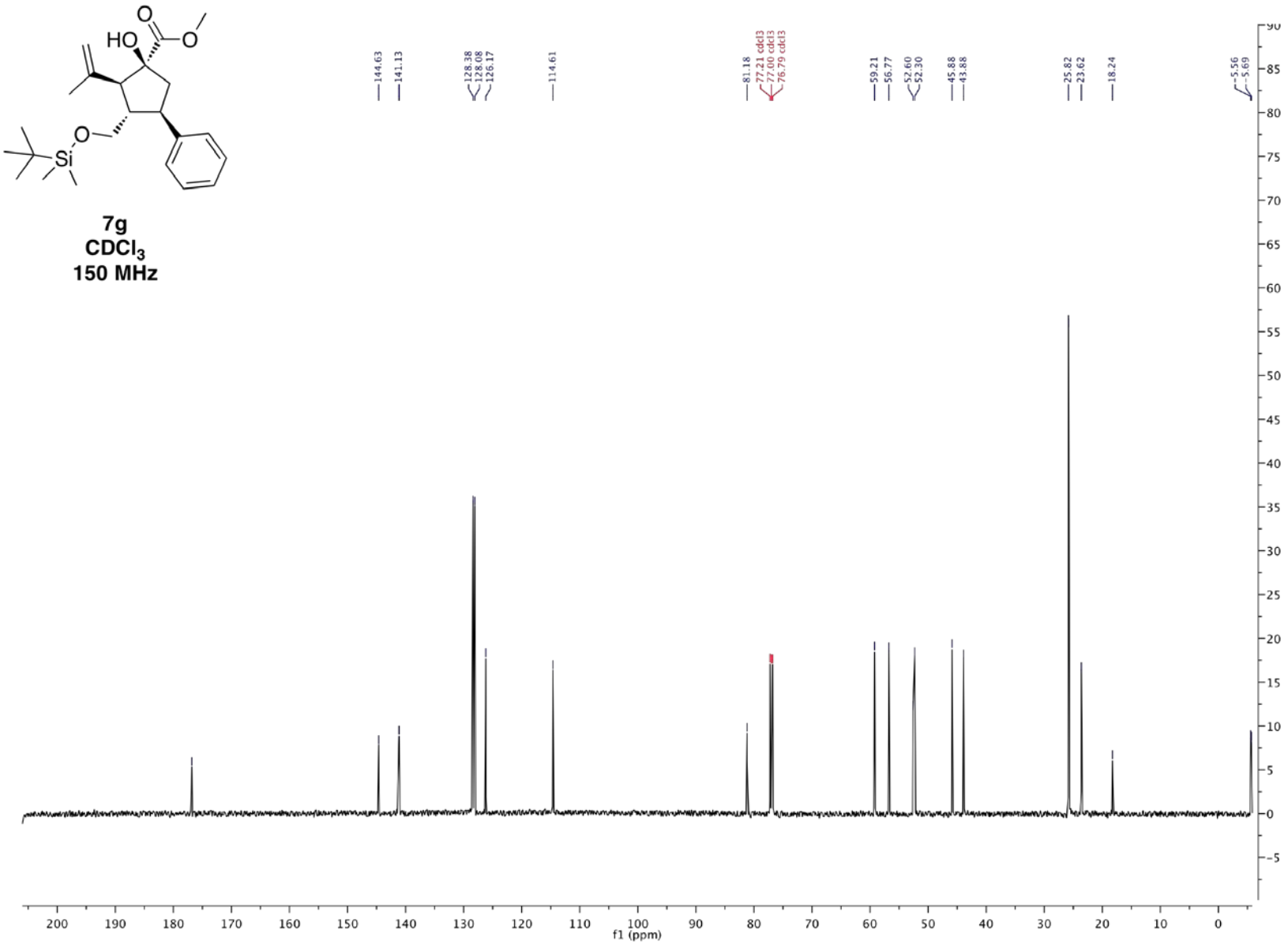








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77.00 cdcl3
76.79 cdcl3
59.21
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52.30
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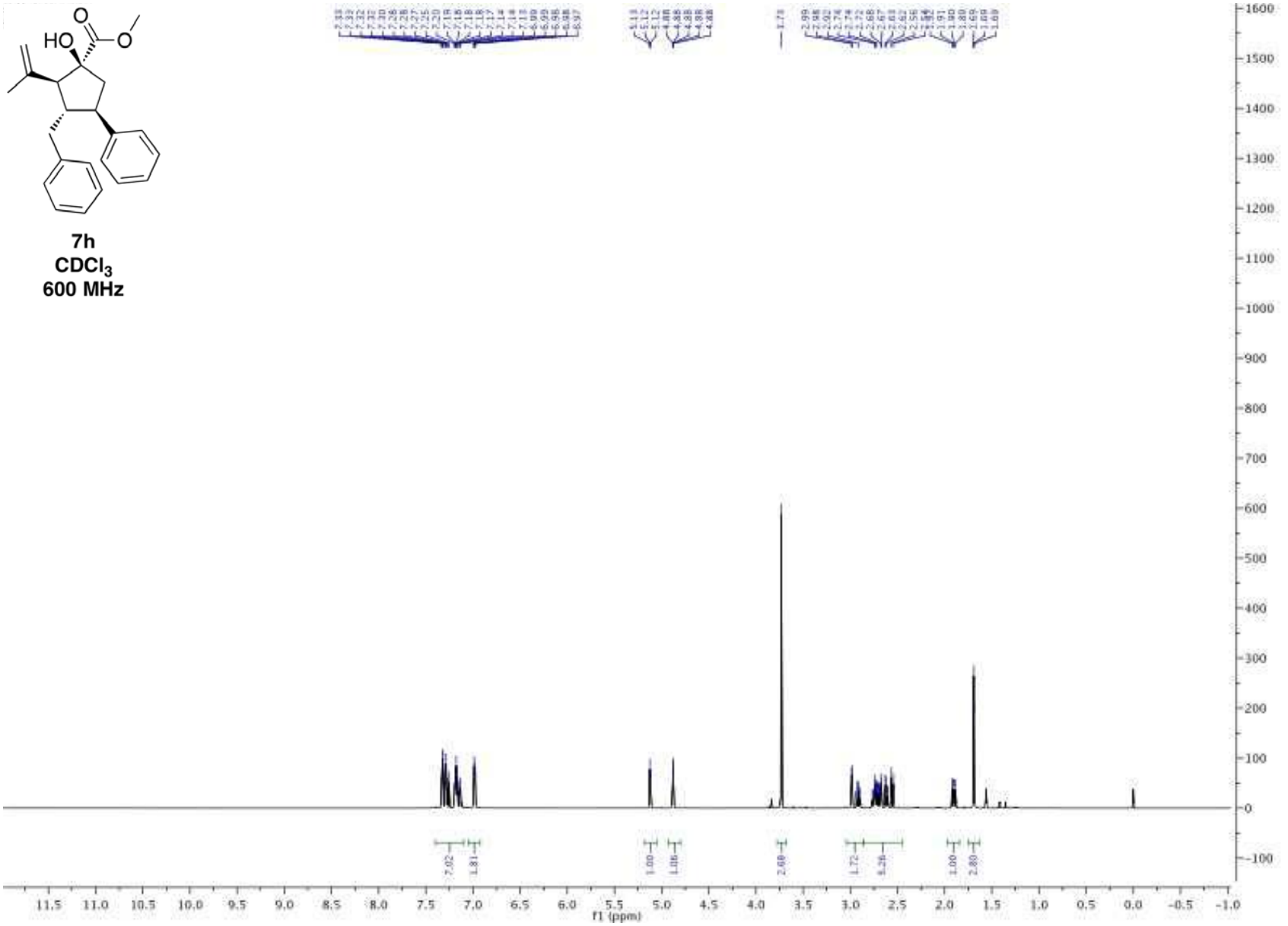
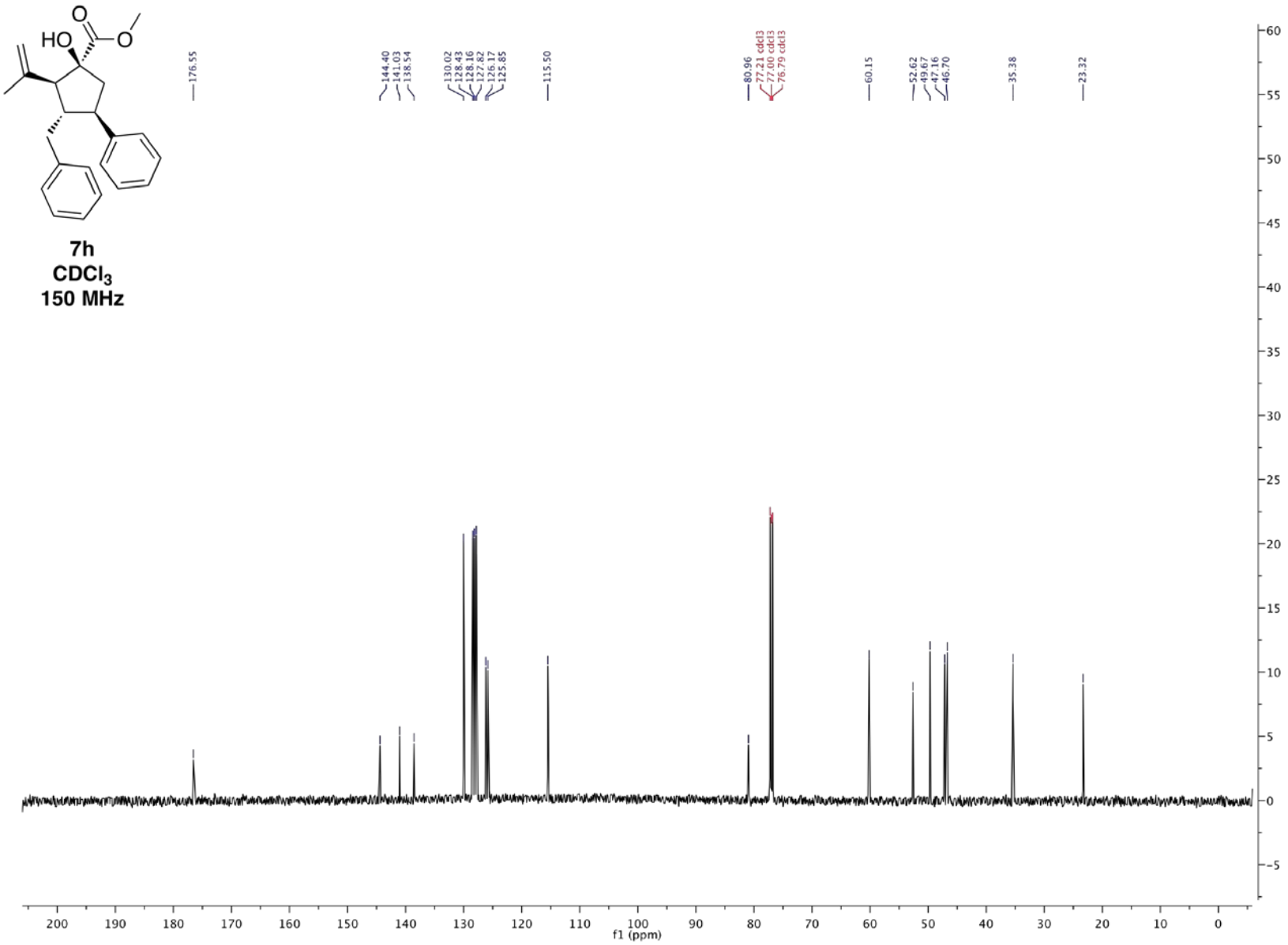
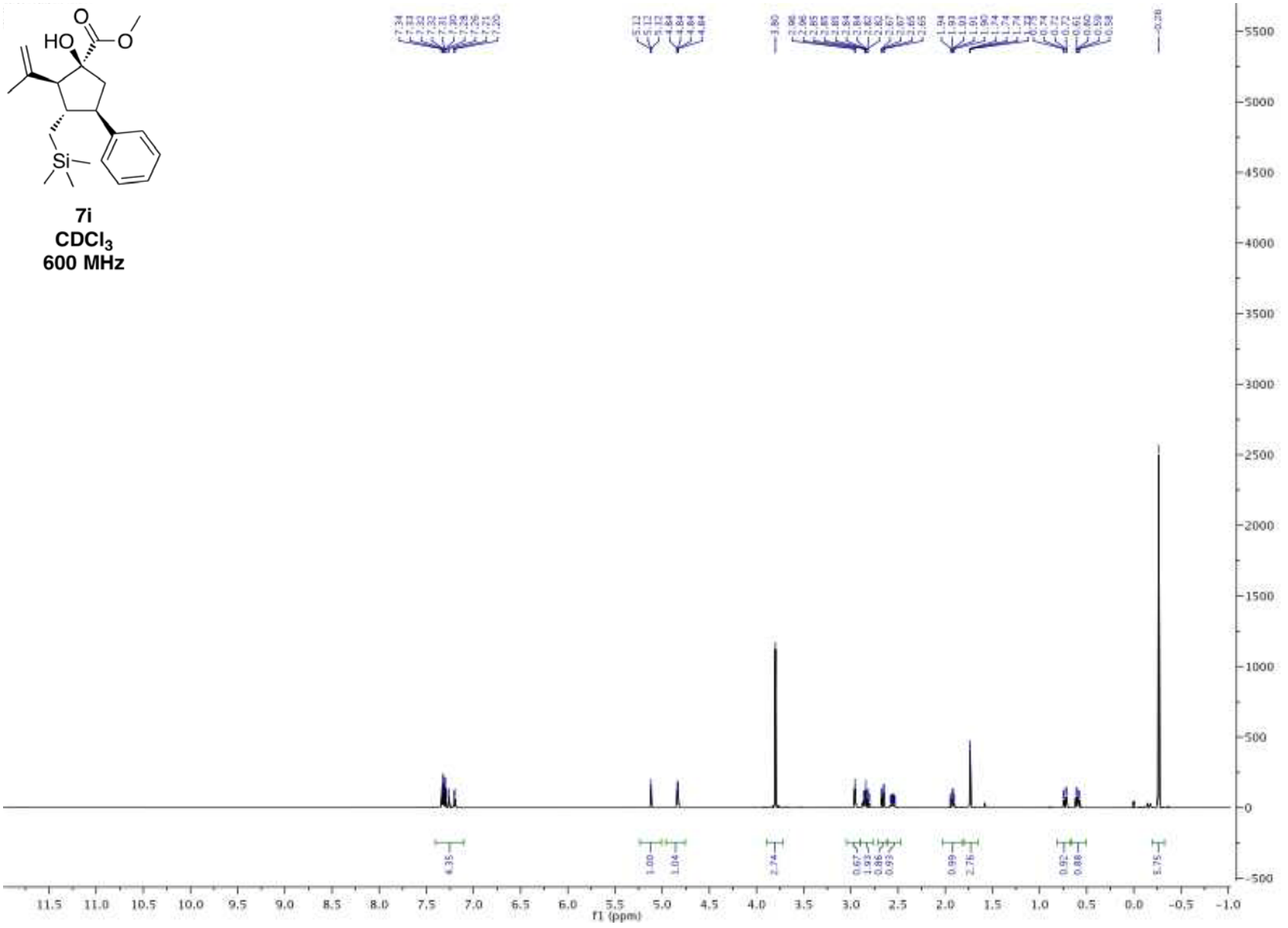
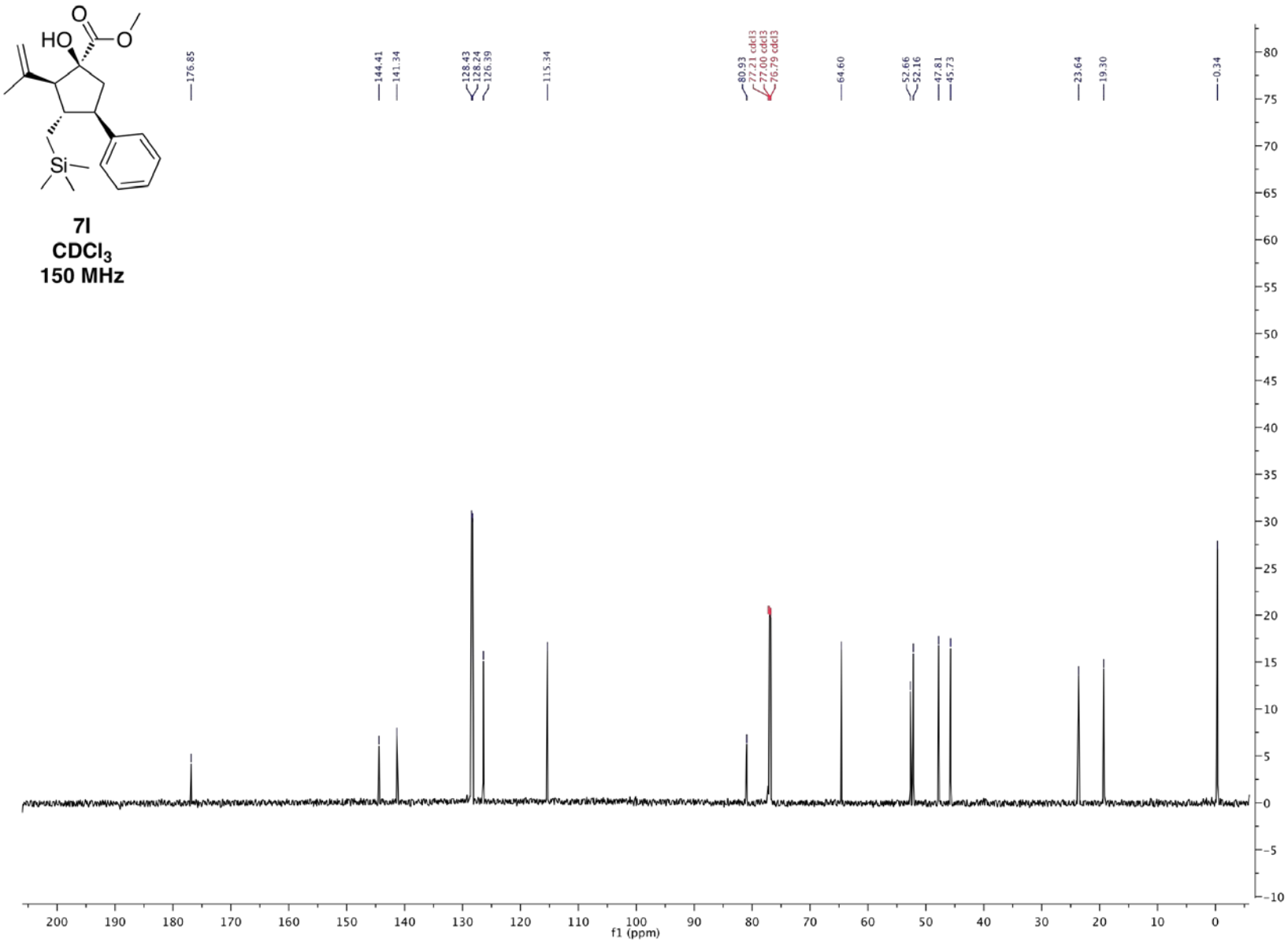
18.24
-5.56
-5.69
f1 (ppm)





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77.21 cdcl3
77.00 cdcl3
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52.66
52.16
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23.64
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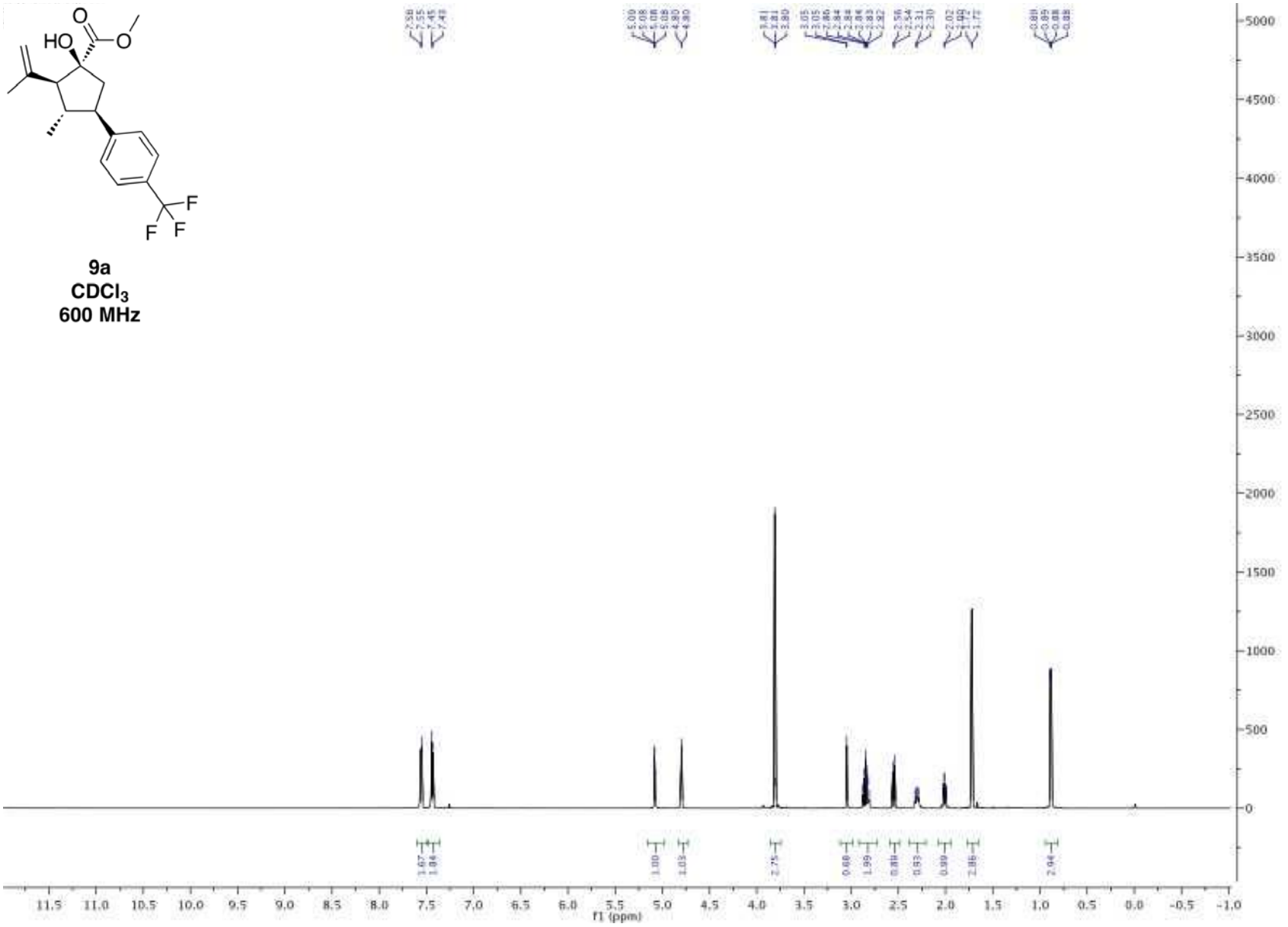
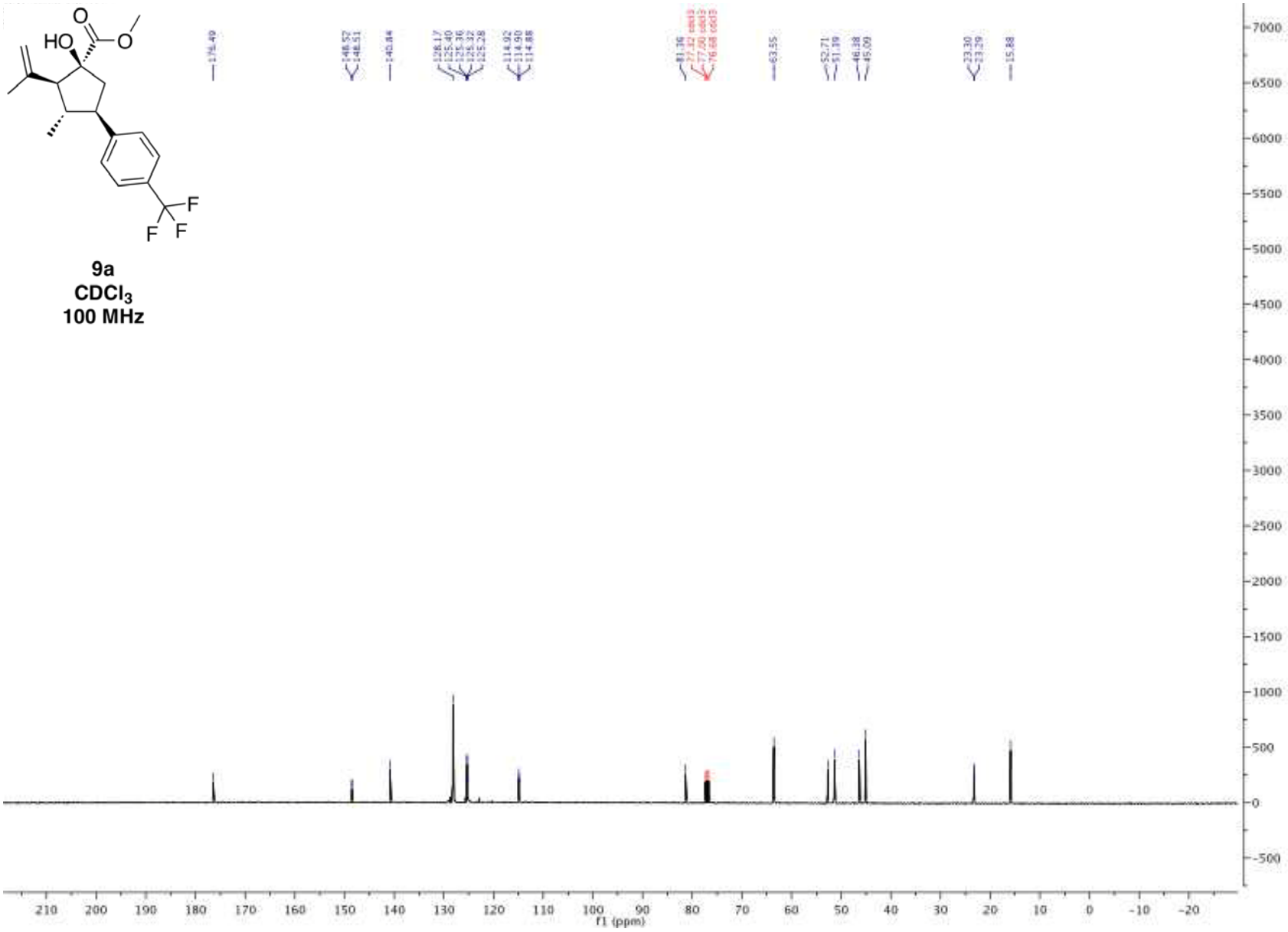
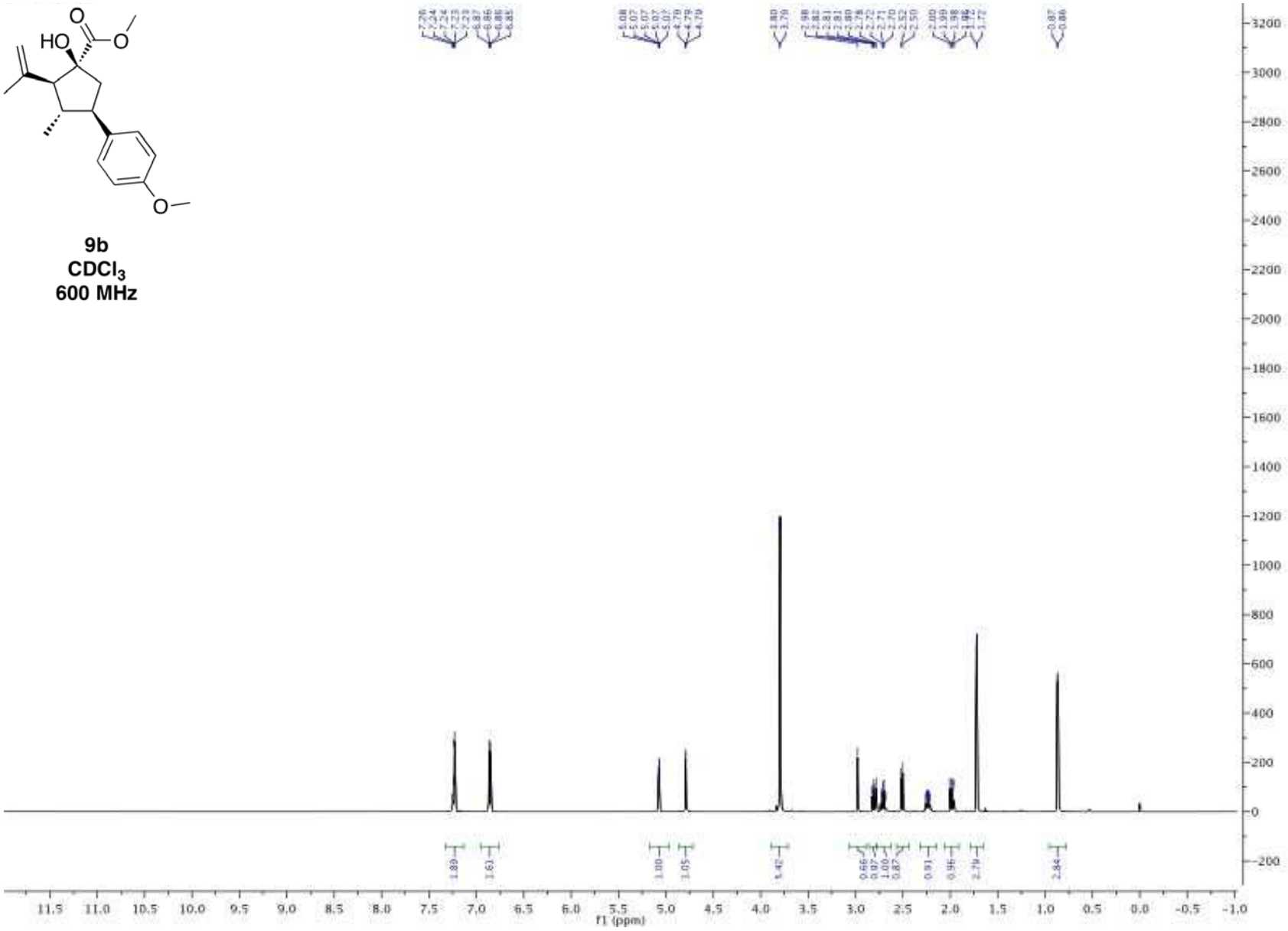
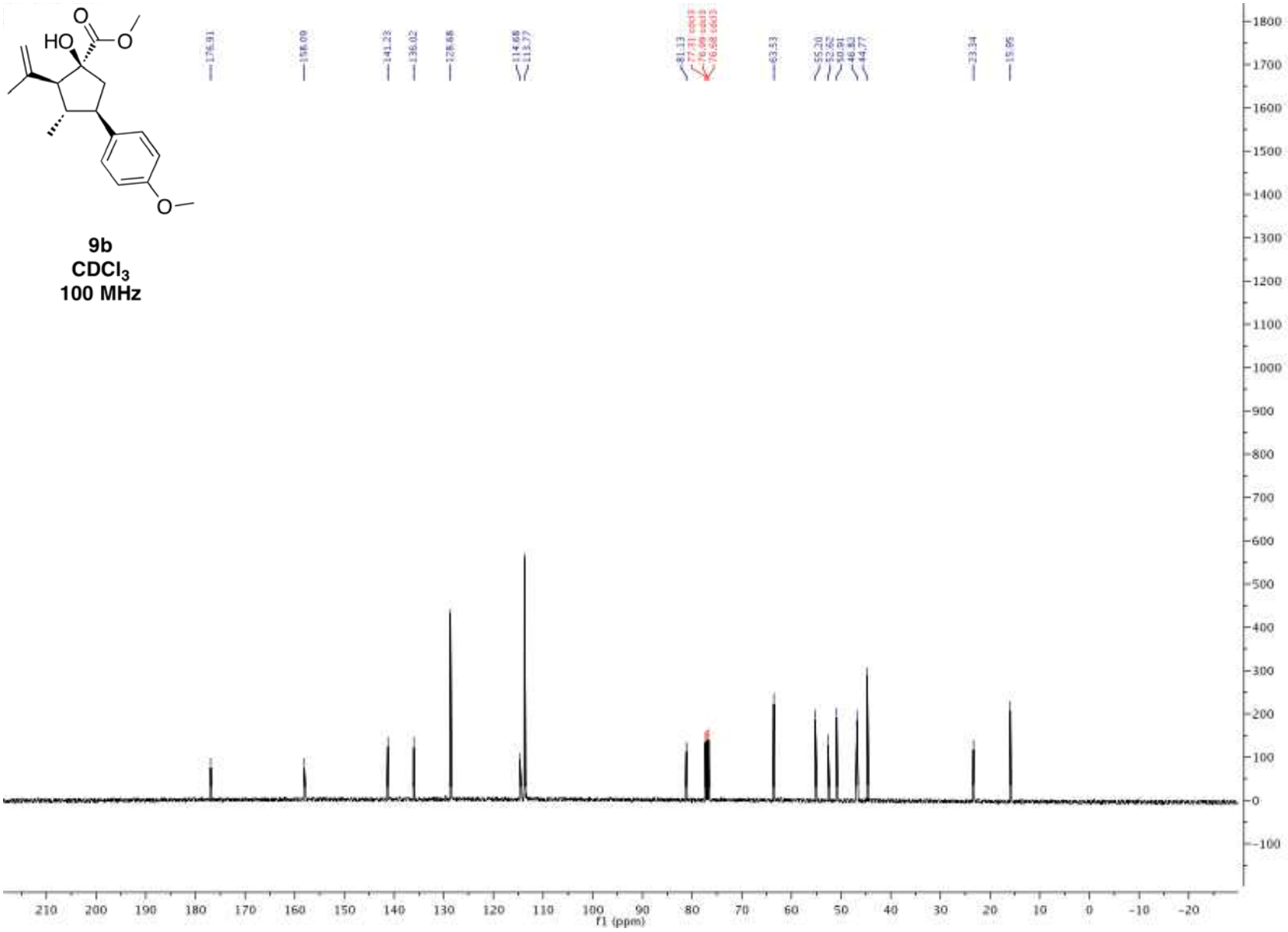
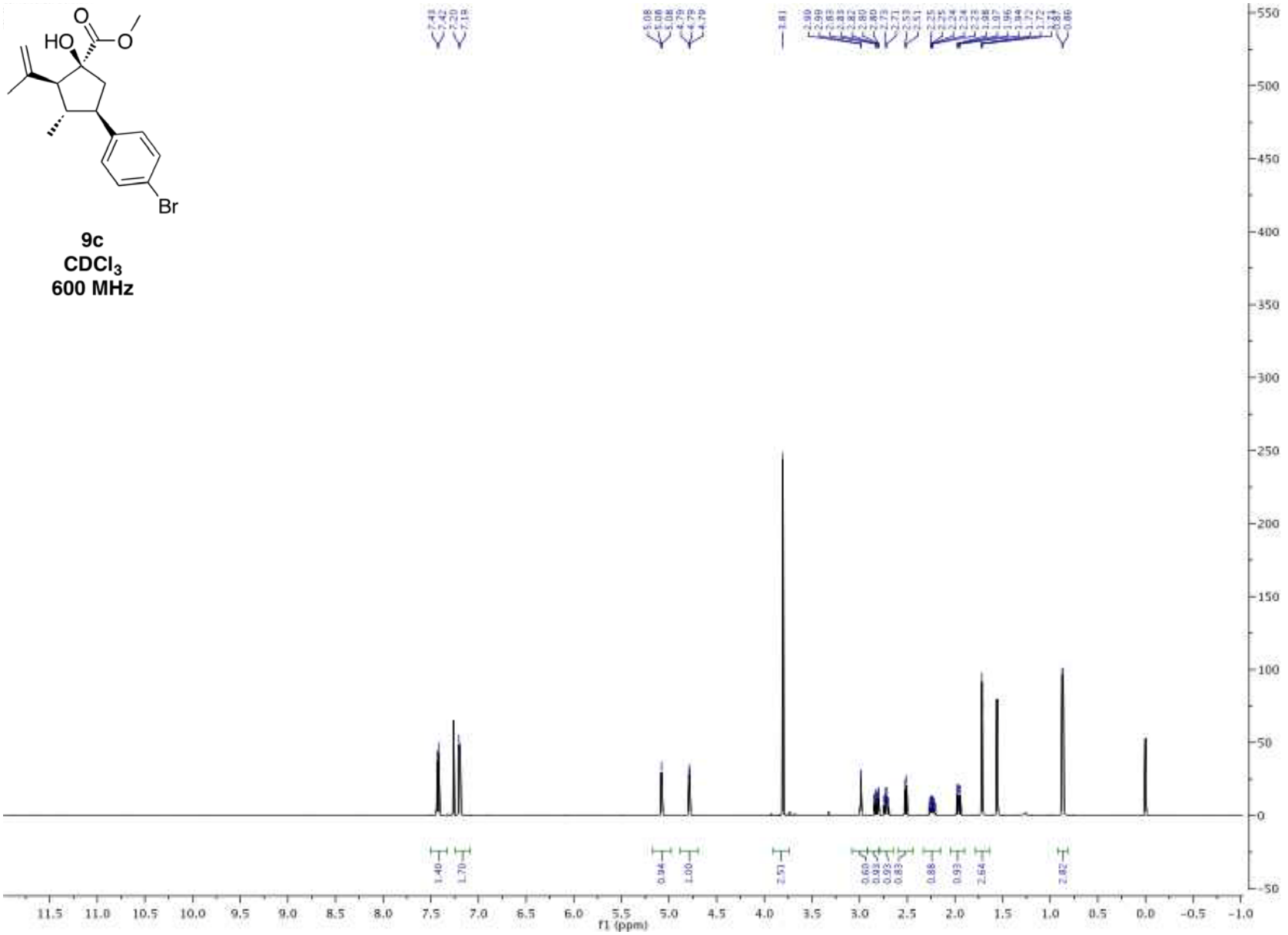
-0.34
f1 (ppm)

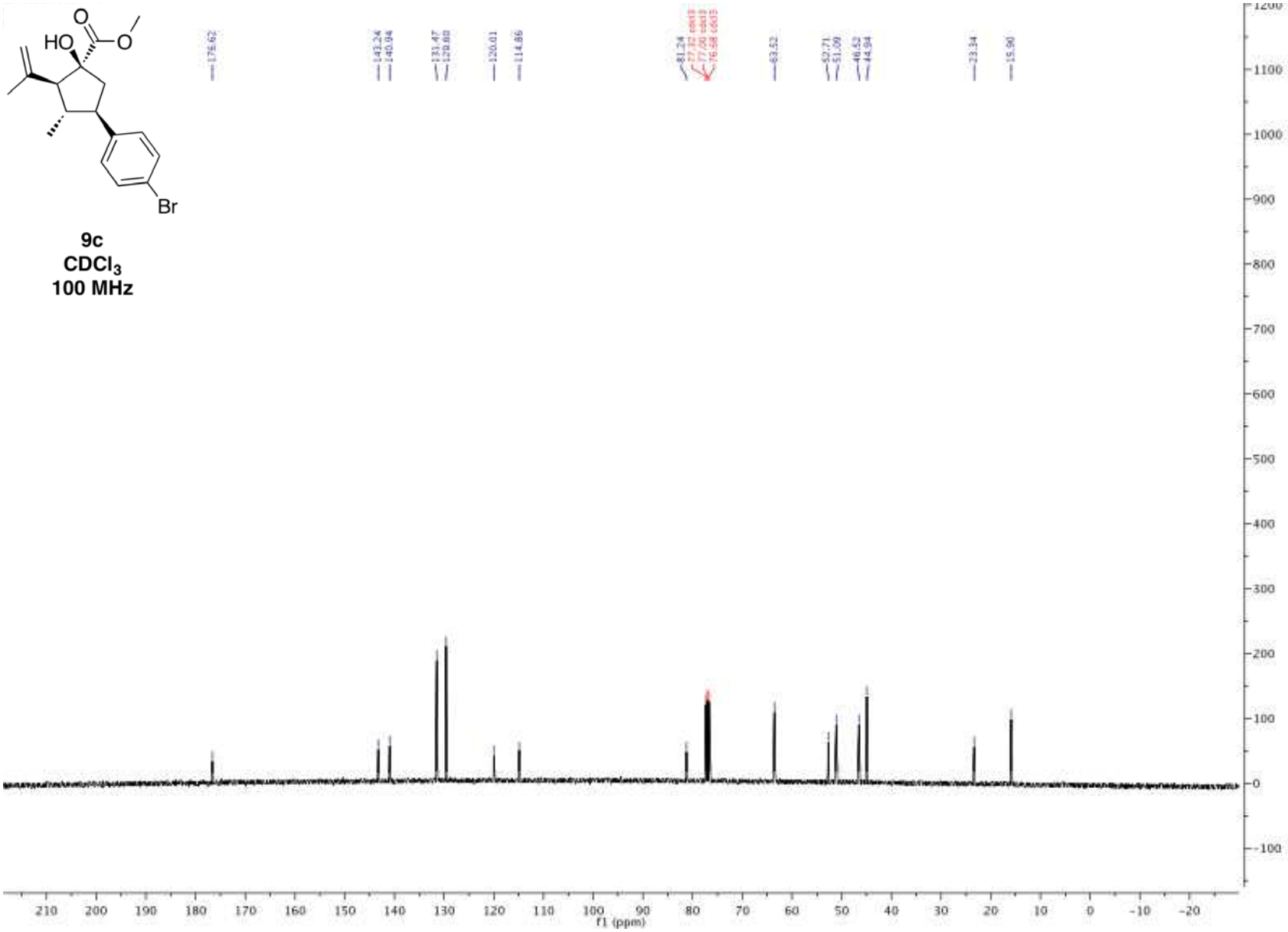


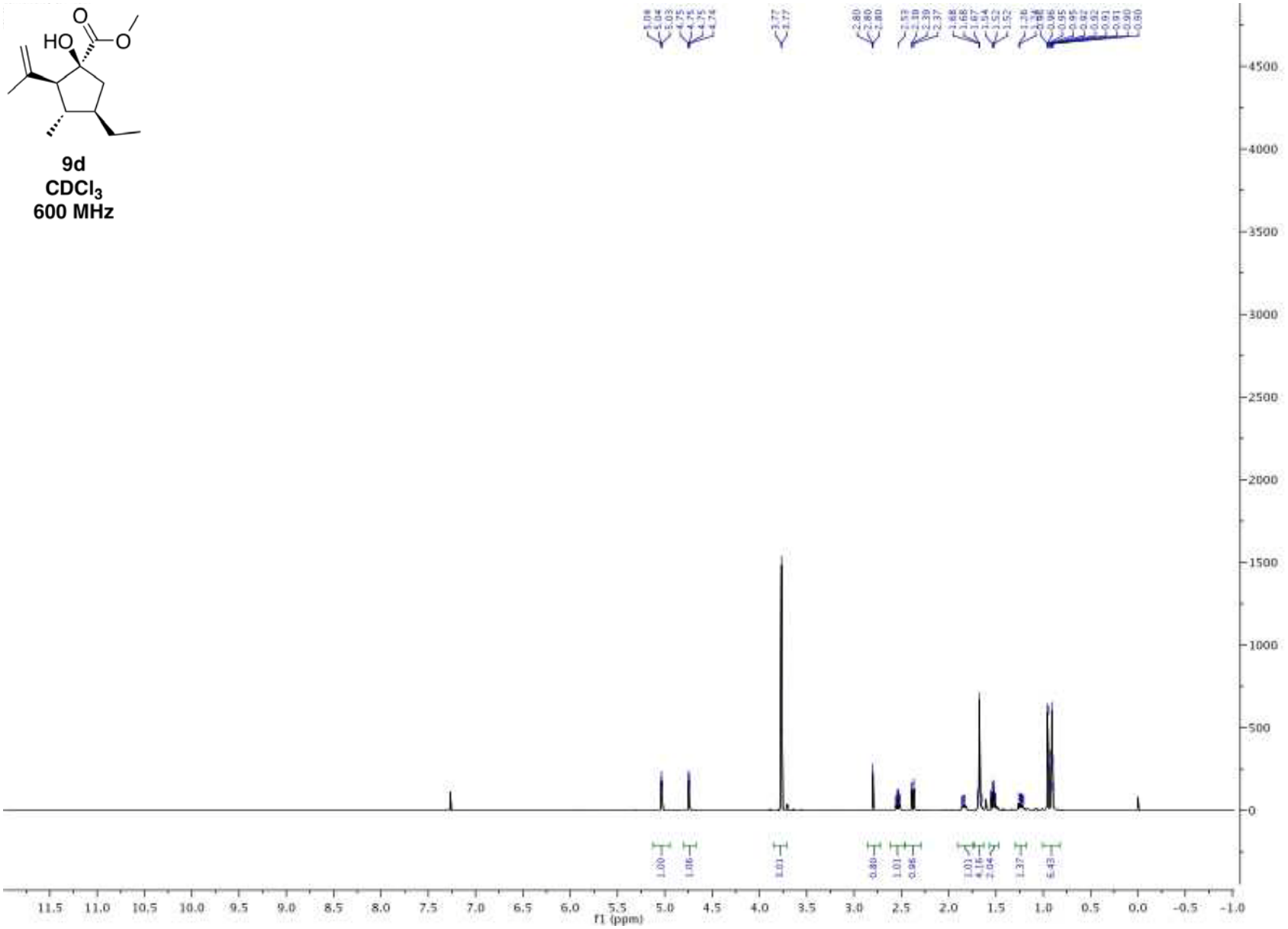


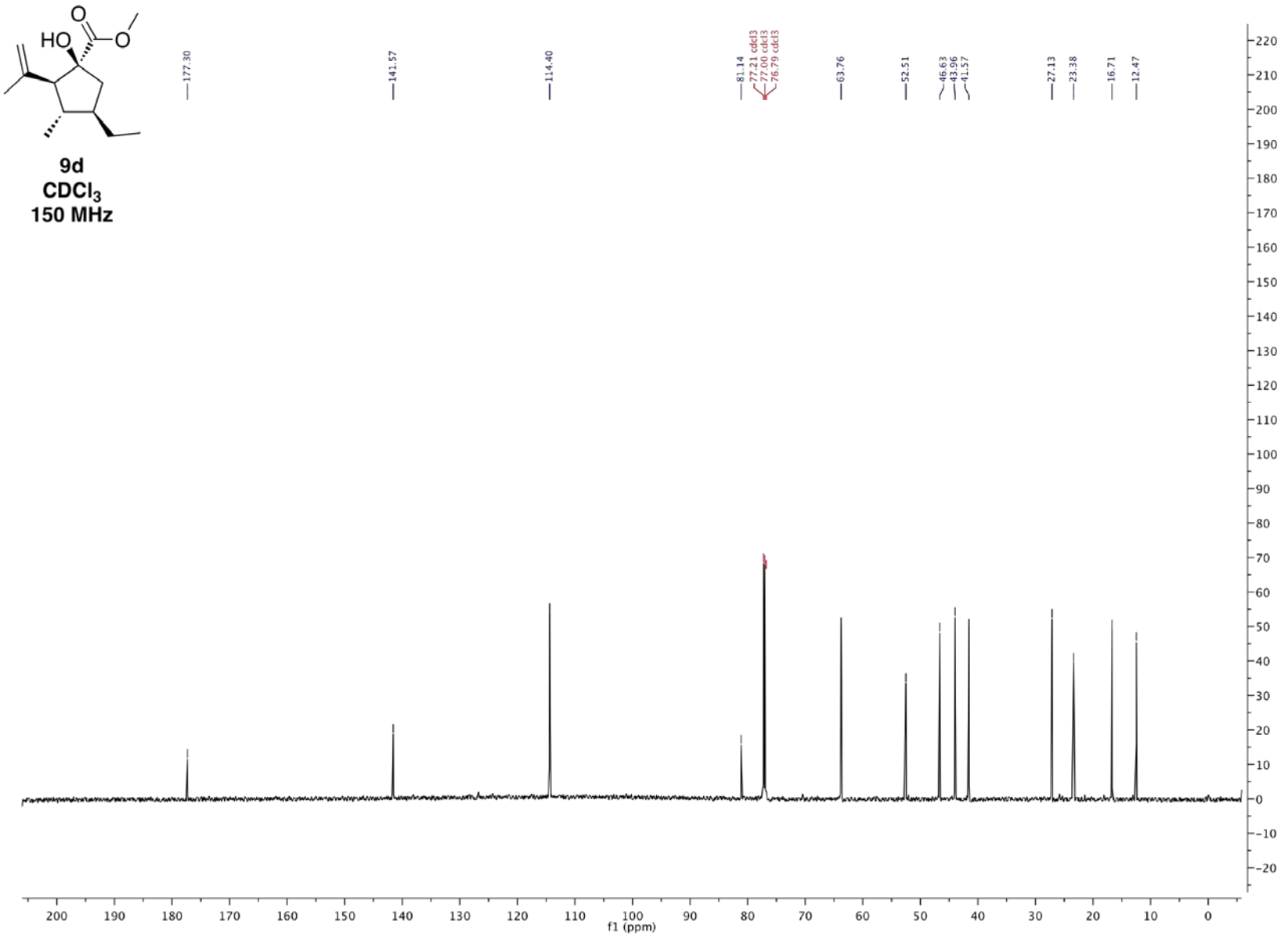


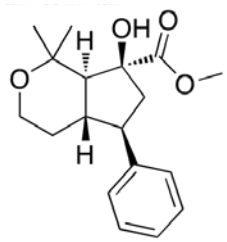












12
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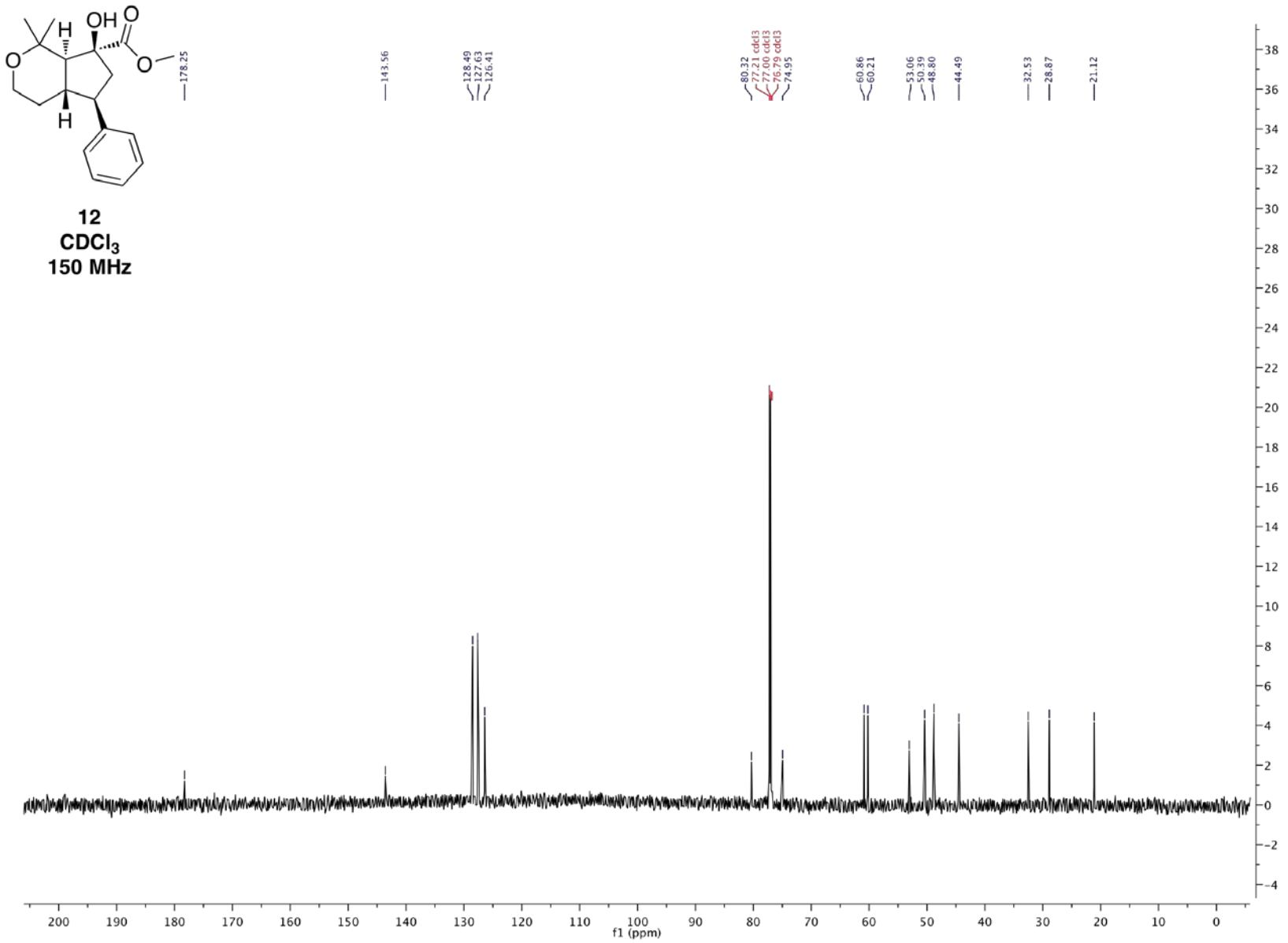
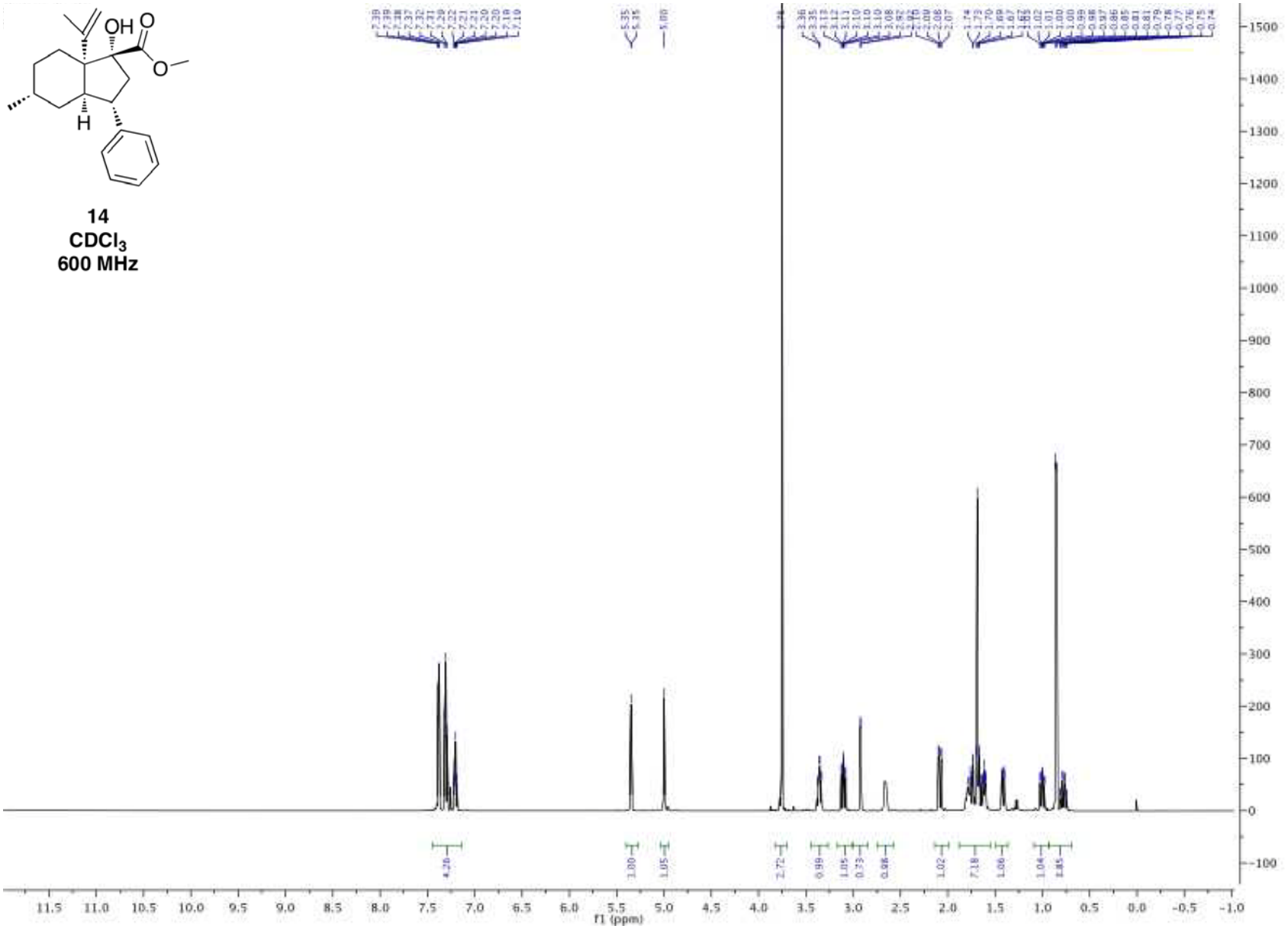
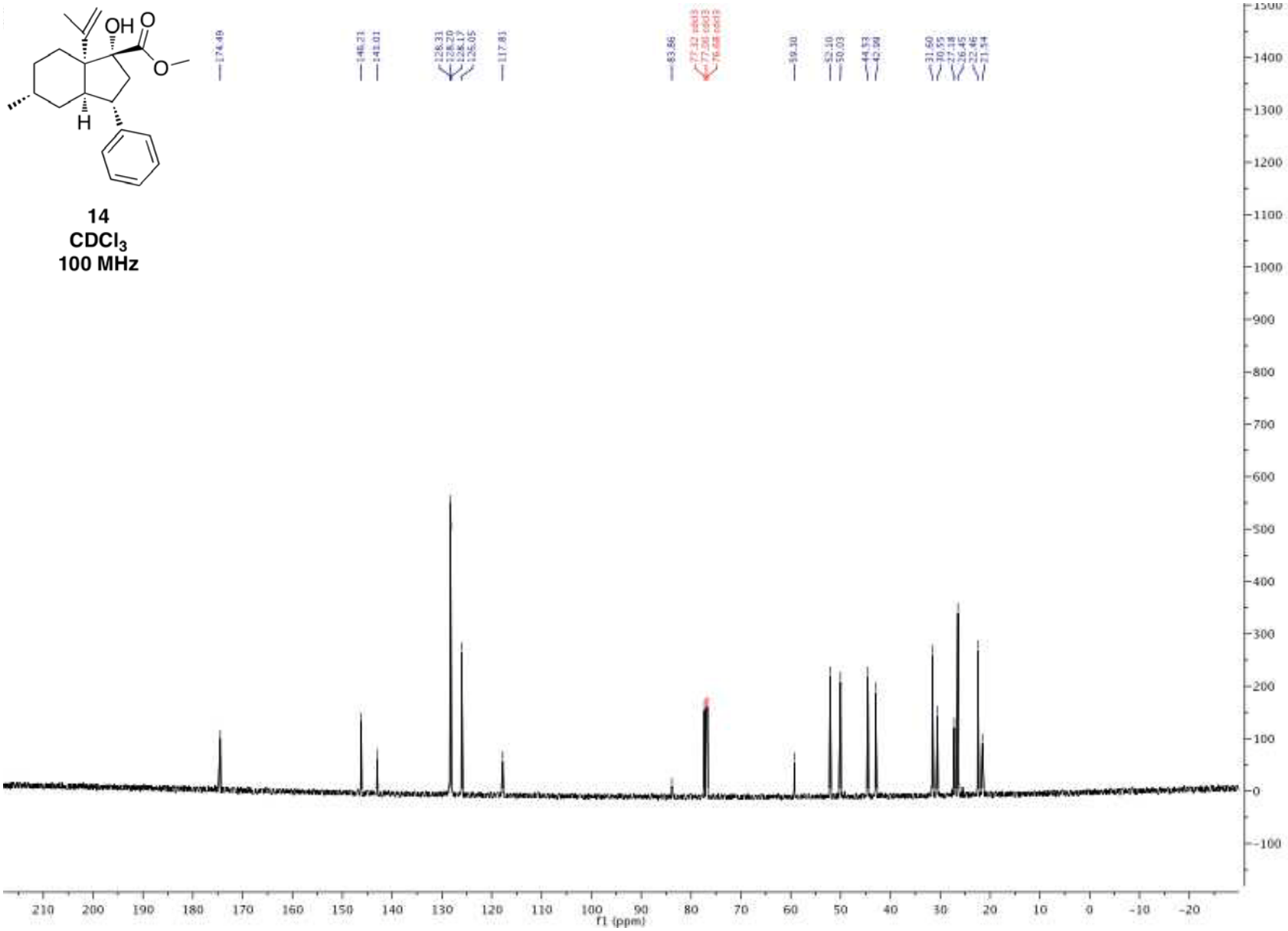
600 MHz



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O
H
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100 MHz
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126.05
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83.86
77.32
77.00
76.68
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44.53
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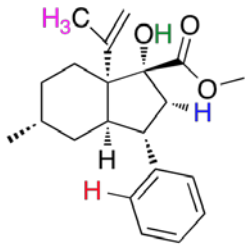
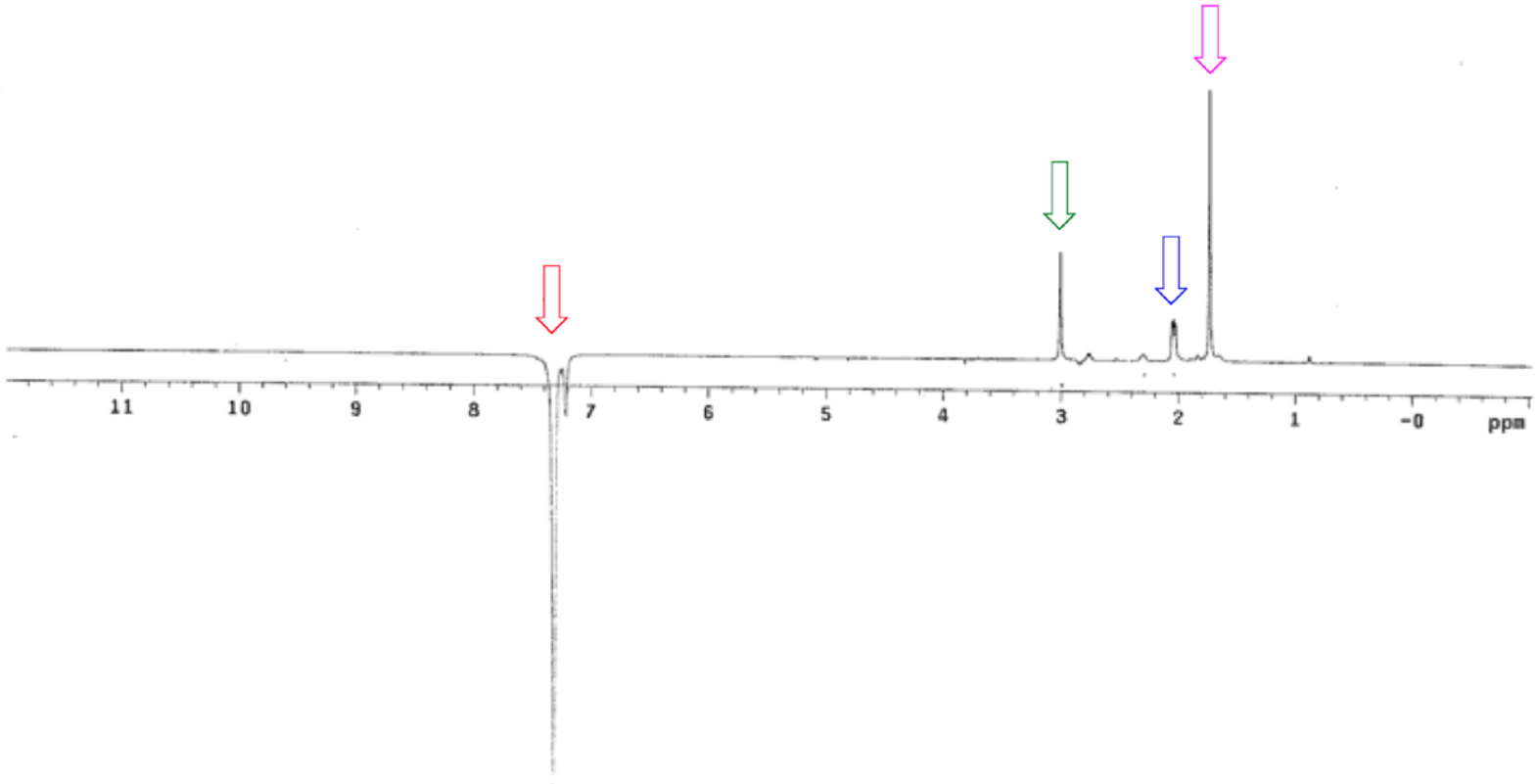
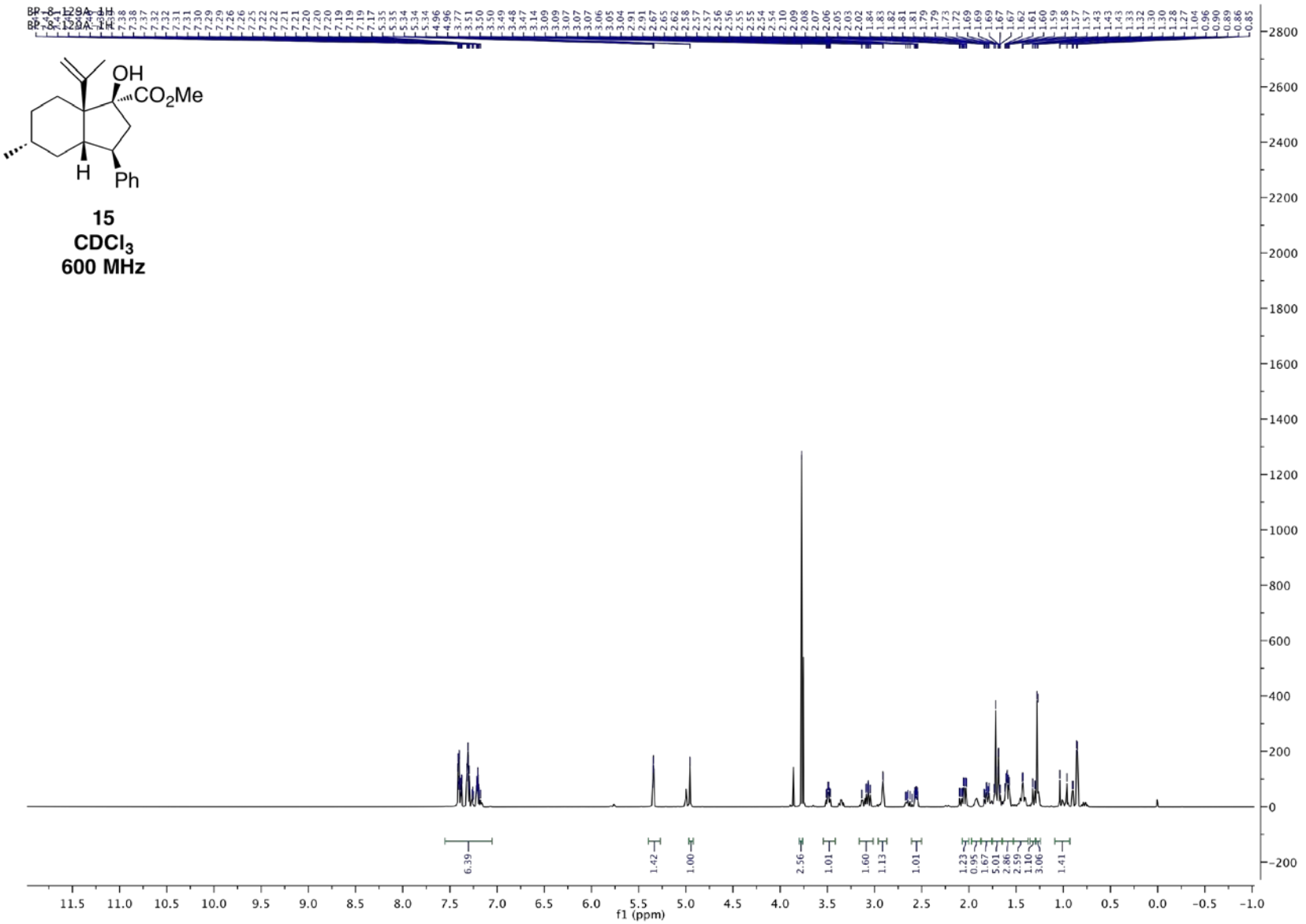
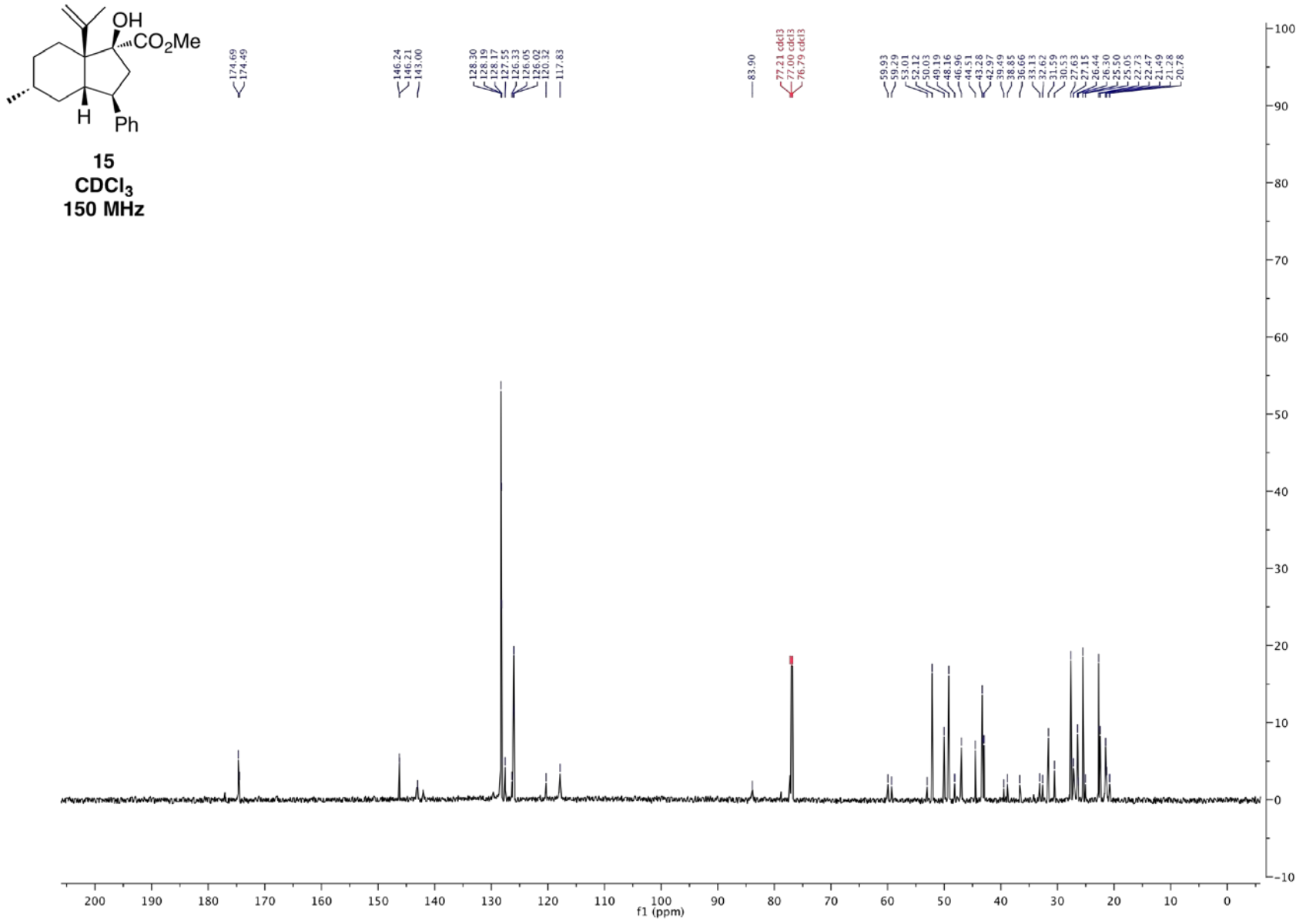
26.45
22.46
21.54
f1 (ppm)



14
$CDCl_3$
600 MHz



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H
Ph
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CDCl3
150 MHz
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128.17
127.55
126.33
126.05
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120.32
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59.93
59.29
53.01
52.12
50.03
49.19
48.16
46.96
44.51
43.28
42.97
39.49
38.85
36.66
33.13
32.62
31.59
30.53
27.63
27.15
26.44
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25.05
22.73
22.47
21.49
21.28
20.78
f1 (ppm)

4. X-Ray Crystallography Data

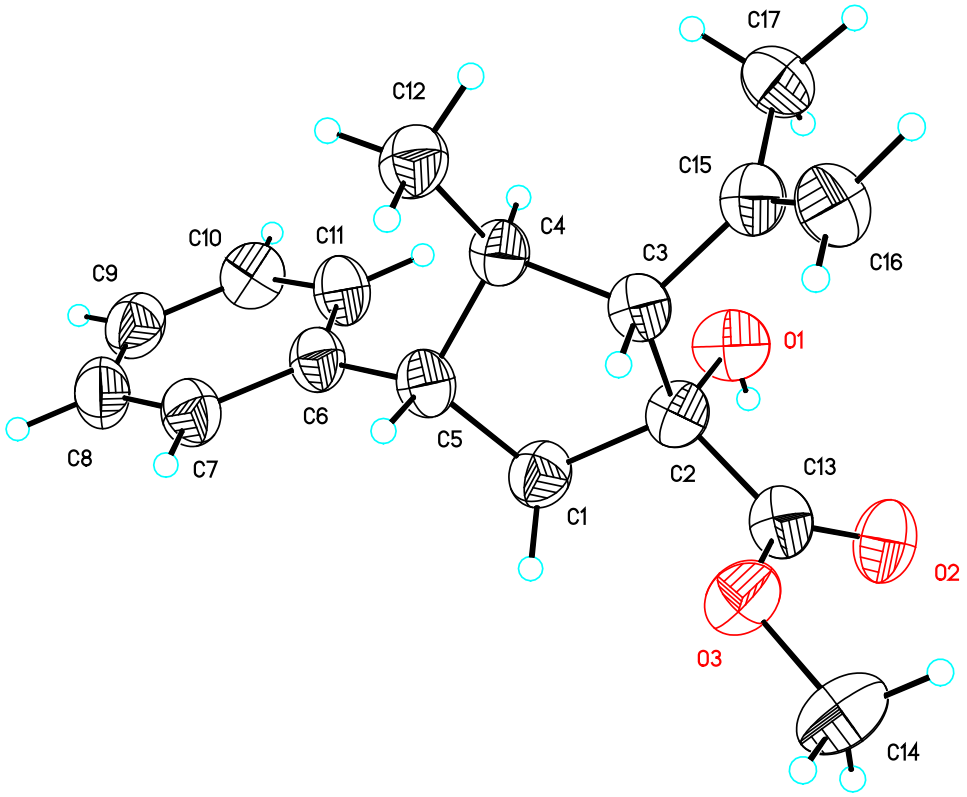


Table 1. Crystal data and structure refinement for **7a**.

Identification code	**7a**	
Empirical formula	C17 H22 O3	
Formula weight	274.35	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	Pbca	
Unit cell dimensions	a = 12.7496(10) Å	⟨= 90°.
	b = 10.6190(12) Å	®= 90°.
	c = 22.4445(18) Å	© = 90°.
Volume	3038.7(5) $Å^3$	
Z	8	
Density (calculated)	1.199 Mg/m^3	
Absorption coefficient	0.646 mm^{-1}	
F(000)	1184	
Crystal size	0.14 x 0.13 x 0.04 mm^3	
Theta range for data collection	3.94 to 69.41°.	
Index ranges	-15<=h<=15, -12<=k<=12, -27<=l<=24	
Reflections collected	15922	
Independent reflections	2779 [R(int) = 0.0829]	
Completeness to theta = 69.41°	97.4 %	
Absorption correction	Semi-empirical from equivalents	

Max. and min. transmission	0.9721 and 0.9150
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2779 / 0 / 190
Goodness-of-fit on F^2	1.066
Final R indices [I>2sigma(I)]	R1 = 0.0761, wR2 = 0.1951
R indices (all data)	R1 = 0.1217, wR2 = 0.2347
Extinction coefficient	0.0009(3)
Largest diff. peak and hole	0.285 and -0.395 e.$Å^{-3}$

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **7a**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	2628(3)	2173(3)	2767(2)	56(1)
C(2)	2095(3)	2524(3)	3365(1)	49(1)
C(3)	1169(3)	1559(3)	3416(1)	47(1)
C(4)	817(3)	1418(3)	2767(1)	47(1)
C(5)	1856(3)	1294(3)	2427(2)	49(1)
C(6)	1793(3)	1553(3)	1765(2)	49(1)
C(7)	2183(3)	697(3)	1352(2)	57(1)
C(8)	2146(3)	947(4)	746(2)	65(1)
C(9)	1725(3)	2047(4)	537(2)	65(1)
C(10)	1326(3)	2910(3)	941(2)	62(1)
C(11)	1361(3)	2662(3)	1546(2)	56(1)
C(12)	71(3)	308(3)	2666(2)	58(1)
C(13)	2834(3)	2453(3)	3891(2)	52(1)
C(14)	3921(4)	1142(4)	4473(2)	78(1)
C(15)	343(3)	1831(3)	3874(1)	51(1)
C(16)	295(4)	1167(4)	4375(2)	62(1)
C(17)	-467(3)	2845(3)	3748(2)	62(1)
O(1)	1691(2)	3769(2)	3330(1)	64(1)

O(2)	3057(2)	3350(2)	4192(1)	67(1)
O(3)	3207(2)	1298(2)	3980(1)	62(1)

Table 3. Bond lengths [Å] and angles [°] for **7a**.

C(1)-C(2)	1.550(5)
C(1)-C(5)	1.556(5)
C(1)-H(1A)	0.9900
C(1)-H(1B)	0.9900
C(2)-O(1)	1.421(4)
C(2)-C(13)	1.513(5)
C(2)-C(3)	1.568(4)
C(3)-C(15)	1.499(5)
C(3)-C(4)	1.533(4)
C(3)-H(3A)	1.0000
C(4)-C(12)	1.531(4)
C(4)-C(5)	1.534(5)
C(4)-H(4A)	1.0000
C(5)-C(6)	1.513(5)
C(5)-H(5A)	1.0000
C(6)-C(11)	1.390(5)
C(6)-C(7)	1.392(5)
C(7)-C(8)	1.386(5)
C(7)-H(7A)	0.9500
C(8)-C(9)	1.368(6)
C(8)-H(8A)	0.9500

C(9)-C(10)	1.386(5)
C(9)-H(9A)	0.9500
C(10)-C(11)	1.384(5)
C(10)-H(10A)	0.9500
C(11)-H(11A)	0.9500
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-O(2)	1.201(4)
C(13)-O(3)	1.330(4)
C(14)-O(3)	1.442(5)
C(14)-H(14A)	0.9800
C(14)-H(14B)	0.9800
C(14)-H(14C)	0.9800
C(15)-C(16)	1.328(5)
C(15)-C(17)	1.519(5)
C(16)-H(16A)	0.99(4)
C(16)-H(16B)	0.93(4)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
O(1)-H(1C)	0.8400

C(2)-C(1)-C(5)	106.9(3)
C(2)-C(1)-H(1A)	110.3
C(5)-C(1)-H(1A)	110.3
C(2)-C(1)-H(1B)	110.3
C(5)-C(1)-H(1B)	110.3
H(1A)-C(1)-H(1B)	108.6
O(1)-C(2)-C(13)	108.4(3)
O(1)-C(2)-C(1)	109.5(3)
C(13)-C(2)-C(1)	113.0(3)
O(1)-C(2)-C(3)	109.9(3)
C(13)-C(2)-C(3)	112.3(3)
C(1)-C(2)-C(3)	103.6(2)
C(15)-C(3)-C(4)	117.7(3)
C(15)-C(3)-C(2)	116.9(3)
C(4)-C(3)-C(2)	102.4(2)
C(15)-C(3)-H(3A)	106.3
C(4)-C(3)-H(3A)	106.3
C(2)-C(3)-H(3A)	106.3
C(12)-C(4)-C(3)	113.4(3)
C(12)-C(4)-C(5)	113.4(3)
C(3)-C(4)-C(5)	103.2(3)
C(12)-C(4)-H(4A)	108.9
C(3)-C(4)-H(4A)	108.9

C(5)-C(4)-H(4A)	108.9
C(6)-C(5)-C(4)	115.2(3)
C(6)-C(5)-C(1)	114.0(3)
C(4)-C(5)-C(1)	104.6(3)
C(6)-C(5)-H(5A)	107.6
C(4)-C(5)-H(5A)	107.6
C(1)-C(5)-H(5A)	107.6
C(11)-C(6)-C(7)	117.3(3)
C(11)-C(6)-C(5)	121.5(3)
C(7)-C(6)-C(5)	121.2(3)
C(8)-C(7)-C(6)	121.1(3)
C(8)-C(7)-H(7A)	119.4
C(6)-C(7)-H(7A)	119.4
C(9)-C(8)-C(7)	120.9(3)
C(9)-C(8)-H(8A)	119.6
C(7)-C(8)-H(8A)	119.6
C(8)-C(9)-C(10)	119.0(4)
C(8)-C(9)-H(9A)	120.5
C(10)-C(9)-H(9A)	120.5
C(11)-C(10)-C(9)	120.2(4)
C(11)-C(10)-H(10A)	119.9
C(9)-C(10)-H(10A)	119.9
C(10)-C(11)-C(6)	121.5(3)

C(10)-C(11)-H(11A)	119.3
C(6)-C(11)-H(11A)	119.3
C(4)-C(12)-H(12A)	109.5
C(4)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(4)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
O(2)-C(13)-O(3)	124.2(3)
O(2)-C(13)-C(2)	123.1(3)
O(3)-C(13)-C(2)	112.7(3)
O(3)-C(14)-H(14A)	109.5
O(3)-C(14)-H(14B)	109.5
H(14A)-C(14)-H(14B)	109.5
O(3)-C(14)-H(14C)	109.5
H(14A)-C(14)-H(14C)	109.5
H(14B)-C(14)-H(14C)	109.5
C(16)-C(15)-C(3)	120.7(3)
C(16)-C(15)-C(17)	120.2(4)
C(3)-C(15)-C(17)	119.1(3)
C(15)-C(16)-H(16A)	123(2)
C(15)-C(16)-H(16B)	123(2)
H(16A)-C(16)-H(16B)	114(3)

C(15)-C(17)-H(17A)	109.5
C(15)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
C(15)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(2)-O(1)-H(1C)	109.5
C(13)-O(3)-C(14)	116.5(3)

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **7a**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	58(2)	60(2)	50(2)	0(2)	0(2)	-7(2)
C(2)	56(2)	37(2)	54(2)	2(1)	-1(2)	-7(1)
C(3)	54(2)	37(1)	49(2)	1(1)	2(2)	-2(1)
C(4)	55(2)	37(2)	48(2)	0(1)	-1(2)	0(1)
C(5)	60(2)	36(2)	52(2)	-2(1)	2(2)	1(1)
C(6)	57(2)	37(2)	52(2)	-5(1)	2(2)	-4(1)
C(7)	64(3)	48(2)	60(2)	-7(2)	0(2)	1(2)
C(8)	72(3)	67(2)	56(2)	-17(2)	4(2)	-5(2)
C(9)	64(3)	80(3)	51(2)	-6(2)	2(2)	-14(2)
C(10)	69(3)	56(2)	61(2)	7(2)	-3(2)	-3(2)
C(11)	73(3)	42(2)	54(2)	-3(1)	4(2)	0(2)
C(12)	62(3)	44(2)	67(2)	-6(2)	-3(2)	-5(2)
C(13)	63(2)	41(2)	53(2)	0(1)	6(2)	-9(2)
C(14)	76(3)	80(3)	78(3)	18(2)	-27(2)	-11(2)
C(15)	60(2)	39(2)	54(2)	-1(1)	1(2)	-4(1)
C(16)	72(3)	56(2)	57(2)	6(2)	10(2)	1(2)
C(17)	62(3)	55(2)	68(2)	2(2)	10(2)	5(2)
O(1)	70(2)	35(1)	88(2)	8(1)	-1(1)	-4(1)

O(2)	87(2)	54(1)	59(2)	-9(1)	-5(1)	-13(1)
O(3)	69(2)	49(1)	68(2)	2(1)	-17(1)	-1(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **7a**.

	x	y	z	U(eq)
H(1A)	3300	1735	2842	67
H(1B)	2771	2941	2531	67
H(3A)	1492	735	3531	56
H(4A)	460	2212	2639	56
H(5A)	2110	410	2479	59
H(7A)	2480	-73	1487	69
H(8A)	2418	348	472	78
H(9A)	1705	2215	122	78
H(10A)	1029	3676	802	74
H(11A)	1083	3262	1817	68
H(12A)	-130	275	2244	87
H(12B)	-558	418	2911	87
H(12C)	423	-478	2776	87
H(14A)	4144	260	4495	117
H(14B)	3569	1379	4844	117
H(14C)	4536	1682	4413	117
H(17A)	-954	2905	4084	92

H(17B)	-858	2628	3386	92
H(17C)	-113	3655	3691	92
H(16A)	-260(30)	1290(30)	4678(16)	60(11)
H(16B)	740(30)	500(40)	4454(16)	67(11)
H(1C)	2190	4282	3302	96

Table 6. Torsion angles [°] for **7a**.

C(5)-C(1)-C(2)-O(1)	-103.1(3)
C(5)-C(1)-C(2)-C(13)	135.9(3)
C(5)-C(1)-C(2)-C(3)	14.1(3)
O(1)-C(2)-C(3)-C(15)	-49.1(4)
C(13)-C(2)-C(3)-C(15)	71.7(4)
C(1)-C(2)-C(3)-C(15)	-166.1(3)
O(1)-C(2)-C(3)-C(4)	81.1(3)
C(13)-C(2)-C(3)-C(4)	-158.1(3)
C(1)-C(2)-C(3)-C(4)	-35.8(3)
C(15)-C(3)-C(4)-C(12)	-62.8(4)
C(2)-C(3)-C(4)-C(12)	167.5(3)
C(15)-C(3)-C(4)-C(5)	174.1(2)
C(2)-C(3)-C(4)-C(5)	44.4(3)
C(12)-C(4)-C(5)-C(6)	75.5(3)
C(3)-C(4)-C(5)-C(6)	-161.4(2)
C(12)-C(4)-C(5)-C(1)	-158.6(3)
C(3)-C(4)-C(5)-C(1)	-35.5(3)
C(2)-C(1)-C(5)-C(6)	139.5(3)
C(2)-C(1)-C(5)-C(4)	12.9(3)
C(4)-C(5)-C(6)-C(11)	53.2(4)
C(1)-C(5)-C(6)-C(11)	-67.6(5)

C(4)-C(5)-C(6)-C(7)	-127.9(3)
C(1)-C(5)-C(6)-C(7)	111.2(4)
C(11)-C(6)-C(7)-C(8)	0.4(6)
C(5)-C(6)-C(7)-C(8)	-178.5(4)
C(6)-C(7)-C(8)-C(9)	0.0(6)
C(7)-C(8)-C(9)-C(10)	-0.3(6)
C(8)-C(9)-C(10)-C(11)	0.2(6)
C(9)-C(10)-C(11)-C(6)	0.2(6)
C(7)-C(6)-C(11)-C(10)	-0.5(6)
C(5)-C(6)-C(11)-C(10)	178.4(3)
O(1)-C(2)-C(13)-O(2)	-3.9(5)
C(1)-C(2)-C(13)-O(2)	117.7(4)
C(3)-C(2)-C(13)-O(2)	-125.5(4)
O(1)-C(2)-C(13)-O(3)	176.5(3)
C(1)-C(2)-C(13)-O(3)	-61.9(4)
C(3)-C(2)-C(13)-O(3)	54.9(4)
C(4)-C(3)-C(15)-C(16)	131.3(4)
C(2)-C(3)-C(15)-C(16)	-106.0(4)
C(4)-C(3)-C(15)-C(17)	-46.4(4)
C(2)-C(3)-C(15)-C(17)	76.2(4)
O(2)-C(13)-O(3)-C(14)	0.2(6)
C(2)-C(13)-O(3)-C(14)	179.9(3)

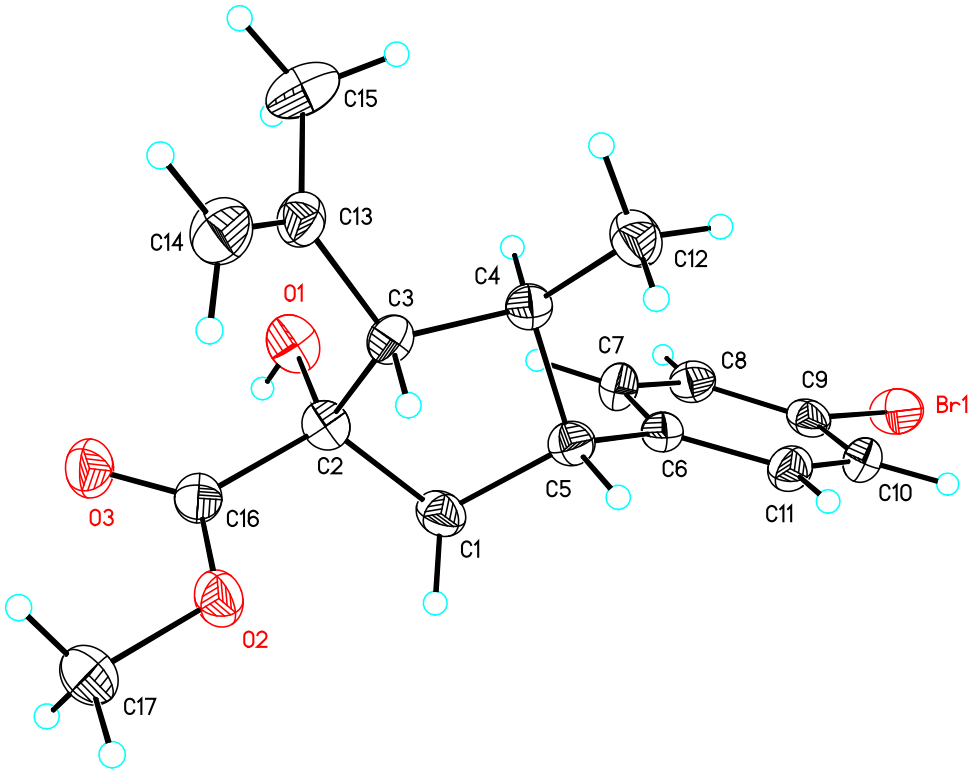


Table 1. Crystal data and structure refinement for **9c**.

Identification code	**9c**	
Empirical formula	C17 H21 Br O3	
Formula weight	353.25	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 5.8343(3) Å	⟨= 90°.
	b = 7.4644(4) Å	®= 90°.
	c = 37.7001(18) Å	© = 90°.
Volume	1641.82(14) $Å^3$	
Z	4	
Density (calculated)	1.429 Mg/m^3	
Absorption coefficient	3.473 mm^{-1}	
F(000)	728	
Crystal size	0.36 x 0.20 x 0.16 mm^3	
Theta range for data collection	2.34 to 69.24°.	
Index ranges	-7<=h<=6, -9<=k<=6, -41<=l<=45	
Reflections collected	13100	
Independent reflections	2837 [R(int) = 0.0216]	
Completeness to theta = 69.24°	97.2 %	

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.6065 and 0.3678
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	2837 / 0 / 190
Goodness-of-fit on F^2	1.028
Final R indices [I>2sigma(I)]	R1 = 0.0243, wR2 = 0.0653
R indices (all data)	R1 = 0.0245, wR2 = 0.0654
Absolute structure parameter	0.024(16)
Largest diff. peak and hole	0.289 and -0.308 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters (Å^{2}x 10^3) for **9c**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
Br(1)	3377(1)	306(1)	8152(1)	45(1)
C(1)	1529(5)	-1691(3)	6375(1)	33(1)
C(2)	2954(4)	-1357(3)	6039(1)	29(1)
C(3)	2374(4)	629(3)	5952(1)	27(1)
C(4)	2487(4)	1498(3)	6320(1)	27(1)
C(5)	1105(4)	171(3)	6548(1)	28(1)
C(6)	1677(4)	245(3)	6941(1)	27(1)
C(7)	3774(4)	-379(3)	7065(1)	31(1)
C(8)	4286(4)	-362(3)	7425(1)	32(1)
C(9)	2681(4)	308(3)	7658(1)	30(1)
C(10)	618(4)	981(3)	7544(1)	33(1)
C(11)	121(4)	942(3)	7184(1)	30(1)
C(12)	1588(5)	3405(3)	6332(1)	37(1)
C(13)	3721(4)	1465(3)	5653(1)	33(1)
C(14)	2814(5)	1564(4)	5333(1)	46(1)
C(15)	6048(5)	2194(5)	5735(1)	54(1)
C(16)	2416(4)	-2559(3)	5726(1)	30(1)
C(17)	-405(5)	-3703(4)	5343(1)	47(1)

O(1)	5321(3)	-1541(2)	6121(1)	39(1)
O(2)	185(3)	-2629(2)	5651(1)	36(1)
O(3)	3871(3)	-3302(2)	5556(1)	42(1)

Table 3. Bond lengths [Å] and angles [°] for **9c**.

Br(1)-C(9)	1.907(2)
C(1)-C(2)	1.538(3)
C(1)-C(5)	1.554(3)
C(1)-H(1A)	0.9900
C(1)-H(1B)	0.9900
C(2)-O(1)	1.422(3)
C(2)-C(16)	1.515(3)
C(2)-C(3)	1.556(3)
C(3)-C(13)	1.507(3)
C(3)-C(4)	1.533(3)
C(3)-H(3A)	1.0000
C(4)-C(12)	1.517(3)
C(4)-C(5)	1.541(3)
C(4)-H(4A)	1.0000
C(5)-C(6)	1.517(3)
C(5)-H(5A)	1.0000
C(6)-C(7)	1.390(3)
C(6)-C(11)	1.392(3)
C(7)-C(8)	1.391(3)
C(7)-H(7A)	0.9500
C(8)-C(9)	1.377(3)

C(8)-H(8A)	0.9500
C(9)-C(10)	1.374(3)
C(10)-C(11)	1.388(3)
C(10)-H(10A)	0.9500
C(11)-H(11A)	0.9500
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-C(14)	1.322(4)
C(13)-C(15)	1.495(4)
C(14)-H(14A)	0.9962
C(14)-H(14B)	0.9202
C(15)-H(15A)	0.9800
C(15)-H(15B)	0.9800
C(15)-H(15C)	0.9800
C(16)-O(3)	1.199(3)
C(16)-O(2)	1.333(3)
C(17)-O(2)	1.451(3)
C(17)-H(17A)	0.9800
C(17)-H(17B)	0.9800
C(17)-H(17C)	0.9800
O(1)-H(1C)	0.8400

C(2)-C(1)-C(5)	106.72(17)
C(2)-C(1)-H(1A)	110.4
C(5)-C(1)-H(1A)	110.4
C(2)-C(1)-H(1B)	110.4
C(5)-C(1)-H(1B)	110.4
H(1A)-C(1)-H(1B)	108.6
O(1)-C(2)-C(16)	108.28(19)
O(1)-C(2)-C(1)	109.24(19)
C(16)-C(2)-C(1)	115.8(2)
O(1)-C(2)-C(3)	110.42(18)
C(16)-C(2)-C(3)	110.84(18)
C(1)-C(2)-C(3)	102.15(18)
C(13)-C(3)-C(4)	118.54(19)
C(13)-C(3)-C(2)	116.00(19)
C(4)-C(3)-C(2)	101.74(17)
C(13)-C(3)-H(3A)	106.6
C(4)-C(3)-H(3A)	106.6
C(2)-C(3)-H(3A)	106.6
C(12)-C(4)-C(3)	114.16(19)
C(12)-C(4)-C(5)	113.9(2)
C(3)-C(4)-C(5)	102.22(17)
C(12)-C(4)-H(4A)	108.8
C(3)-C(4)-H(4A)	108.8

C(5)-C(4)-H(4A)	108.8
C(6)-C(5)-C(4)	113.97(18)
C(6)-C(5)-C(1)	113.99(18)
C(4)-C(5)-C(1)	104.89(17)
C(6)-C(5)-H(5A)	107.9
C(4)-C(5)-H(5A)	107.9
C(1)-C(5)-H(5A)	107.9
C(7)-C(6)-C(11)	118.4(2)
C(7)-C(6)-C(5)	120.70(19)
C(11)-C(6)-C(5)	120.9(2)
C(6)-C(7)-C(8)	121.0(2)
C(6)-C(7)-H(7A)	119.5
C(8)-C(7)-H(7A)	119.5
C(9)-C(8)-C(7)	118.6(2)
C(9)-C(8)-H(8A)	120.7
C(7)-C(8)-H(8A)	120.7
C(10)-C(9)-C(8)	122.0(2)
C(10)-C(9)-Br(1)	119.50(18)
C(8)-C(9)-Br(1)	118.52(18)
C(9)-C(10)-C(11)	118.8(2)
C(9)-C(10)-H(10A)	120.6
C(11)-C(10)-H(10A)	120.6
C(10)-C(11)-C(6)	121.1(2)

C(10)-C(11)-H(11A)	119.5
C(6)-C(11)-H(11A)	119.5
C(4)-C(12)-H(12A)	109.5
C(4)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
C(4)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(14)-C(13)-C(15)	122.1(2)
C(14)-C(13)-C(3)	119.8(2)
C(15)-C(13)-C(3)	118.1(2)
C(13)-C(14)-H(14A)	120.8
C(13)-C(14)-H(14B)	124.6
H(14A)-C(14)-H(14B)	114.3
C(13)-C(15)-H(15A)	109.5
C(13)-C(15)-H(15B)	109.5
H(15A)-C(15)-H(15B)	109.5
C(13)-C(15)-H(15C)	109.5
H(15A)-C(15)-H(15C)	109.5
H(15B)-C(15)-H(15C)	109.5
O(3)-C(16)-O(2)	124.0(2)
O(3)-C(16)-C(2)	122.9(2)
O(2)-C(16)-C(2)	113.0(2)

O(2)-C(17)-H(17A)	109.5
O(2)-C(17)-H(17B)	109.5
H(17A)-C(17)-H(17B)	109.5
O(2)-C(17)-H(17C)	109.5
H(17A)-C(17)-H(17C)	109.5
H(17B)-C(17)-H(17C)	109.5
C(2)-O(1)-H(1C)	109.5
C(16)-O(2)-C(17)	115.0(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **9c**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
Br(1)	50(1)	58(1)	26(1)	0(1)	-3(1)	-16(1)
C(1)	43(1)	28(1)	28(1)	1(1)	-1(1)	-7(1)
C(2)	30(1)	29(1)	28(1)	1(1)	-5(1)	3(1)
C(3)	24(1)	30(1)	26(1)	3(1)	0(1)	4(1)
C(4)	26(1)	28(1)	28(1)	0(1)	-1(1)	0(1)
C(5)	28(1)	30(1)	27(1)	1(1)	-1(1)	-2(1)
C(6)	29(1)	25(1)	27(1)	-1(1)	3(1)	-3(1)
C(7)	30(1)	35(1)	30(1)	-3(1)	4(1)	4(1)
C(8)	29(1)	35(1)	32(1)	2(1)	-2(1)	-2(1)
C(9)	40(1)	27(1)	22(1)	-1(1)	-1(1)	-9(1)
C(10)	33(1)	34(1)	32(1)	-4(1)	10(1)	-1(1)
C(11)	27(1)	30(1)	34(1)	1(1)	3(1)	2(1)
C(12)	46(1)	29(1)	37(1)	-1(1)	1(1)	3(1)
C(13)	39(1)	28(1)	32(1)	3(1)	7(1)	5(1)
C(14)	61(2)	47(2)	30(1)	8(1)	3(1)	-1(1)
C(15)	36(2)	65(2)	60(2)	15(2)	8(1)	-8(1)
C(16)	35(1)	26(1)	29(1)	3(1)	2(1)	2(1)
C(17)	45(2)	56(2)	38(1)	-12(1)	-6(1)	-10(1)

O(1)	37(1)	36(1)	42(1)	-1(1)	-11(1)	10(1)
O(2)	34(1)	42(1)	32(1)	-10(1)	-4(1)	0(1)
O(3)	36(1)	47(1)	44(1)	-13(1)	4(1)	4(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **9c**.

	x	y	z	U(eq)
H(1A)	2371	-2482	6541	39
H(1B)	53	-2268	6314	39
H(3A)	728	655	5878	32
H(4A)	4116	1495	6402	33
H(5A)	-557	466	6520	34
H(7A)	4873	-824	6901	38
H(8A)	5714	-803	7509	38
H(10A)	-450	1464	7708	39
H(11A)	-1304	1398	7102	37
H(12A)	2542	4171	6181	56
H(12B)	1638	3846	6577	56
H(12C)	3	3429	6246	56
H(14A)	1357	930	5276	69
H(14B)	3517	2084	5140	69
H(15A)	6726	2692	5518	81
H(15B)	7026	1230	5825	81
H(15C)	5918	3139	5914	81

H(17A)	-2068	-3672	5307	70
H(17B)	88	-4943	5382	70
H(17C)	367	-3219	5133	70
H(1C)	5610	-2618	6167	58

Table 6. Torsion angles [°] for **9c**.

C(5)-C(1)-C(2)-O(1)	93.5(2)
C(5)-C(1)-C(2)-C(16)	-144.02(19)
C(5)-C(1)-C(2)-C(3)	-23.5(2)
O(1)-C(2)-C(3)-C(13)	57.2(3)
C(16)-C(2)-C(3)-C(13)	-62.8(2)
C(1)-C(2)-C(3)-C(13)	173.3(2)
O(1)-C(2)-C(3)-C(4)	-73.0(2)
C(16)-C(2)-C(3)-C(4)	167.05(19)
C(1)-C(2)-C(3)-C(4)	43.1(2)
C(13)-C(3)-C(4)-C(12)	61.6(3)
C(2)-C(3)-C(4)-C(12)	-169.9(2)
C(13)-C(3)-C(4)-C(5)	-174.91(19)
C(2)-C(3)-C(4)-C(5)	-46.4(2)
C(12)-C(4)-C(5)-C(6)	-79.5(2)
C(3)-C(4)-C(5)-C(6)	156.86(18)
C(12)-C(4)-C(5)-C(1)	155.2(2)
C(3)-C(4)-C(5)-C(1)	31.5(2)
C(2)-C(1)-C(5)-C(6)	-130.0(2)
C(2)-C(1)-C(5)-C(4)	-4.7(2)
C(4)-C(5)-C(6)-C(7)	-70.4(3)
C(1)-C(5)-C(6)-C(7)	50.0(3)

C(4)-C(5)-C(6)-C(11)	109.5(2)
C(1)-C(5)-C(6)-C(11)	-130.2(2)
C(11)-C(6)-C(7)-C(8)	2.0(3)
C(5)-C(6)-C(7)-C(8)	-178.2(2)
C(6)-C(7)-C(8)-C(9)	-0.8(4)
C(7)-C(8)-C(9)-C(10)	-1.0(4)
C(7)-C(8)-C(9)-Br(1)	179.35(18)
C(8)-C(9)-C(10)-C(11)	1.6(4)
Br(1)-C(9)-C(10)-C(11)	-178.81(19)
C(9)-C(10)-C(11)-C(6)	-0.3(3)
C(7)-C(6)-C(11)-C(10)	-1.4(3)
C(5)-C(6)-C(11)-C(10)	178.7(2)
C(4)-C(3)-C(13)-C(14)	-140.0(2)
C(2)-C(3)-C(13)-C(14)	98.4(3)
C(4)-C(3)-C(13)-C(15)	38.8(3)
C(2)-C(3)-C(13)-C(15)	-82.7(3)
O(1)-C(2)-C(16)-O(3)	-9.6(3)
C(1)-C(2)-C(16)-O(3)	-132.7(3)
C(3)-C(2)-C(16)-O(3)	111.6(3)
O(1)-C(2)-C(16)-O(2)	173.4(2)
C(1)-C(2)-C(16)-O(2)	50.4(3)
C(3)-C(2)-C(16)-O(2)	-65.4(3)
O(3)-C(16)-O(2)-C(17)	0.5(4)

C(2)-C(16)-O(2)-C(17)	177.4(2)

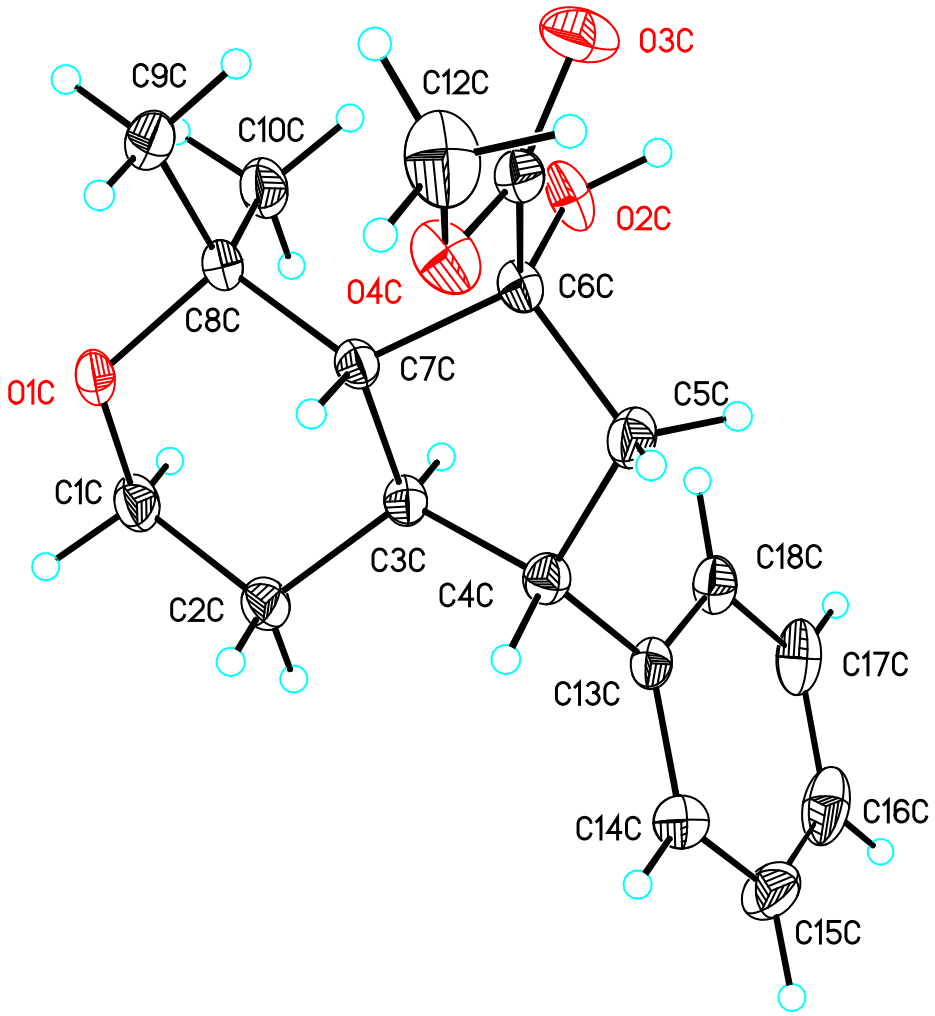
Symmetry transformations used to generate equivalent atoms:

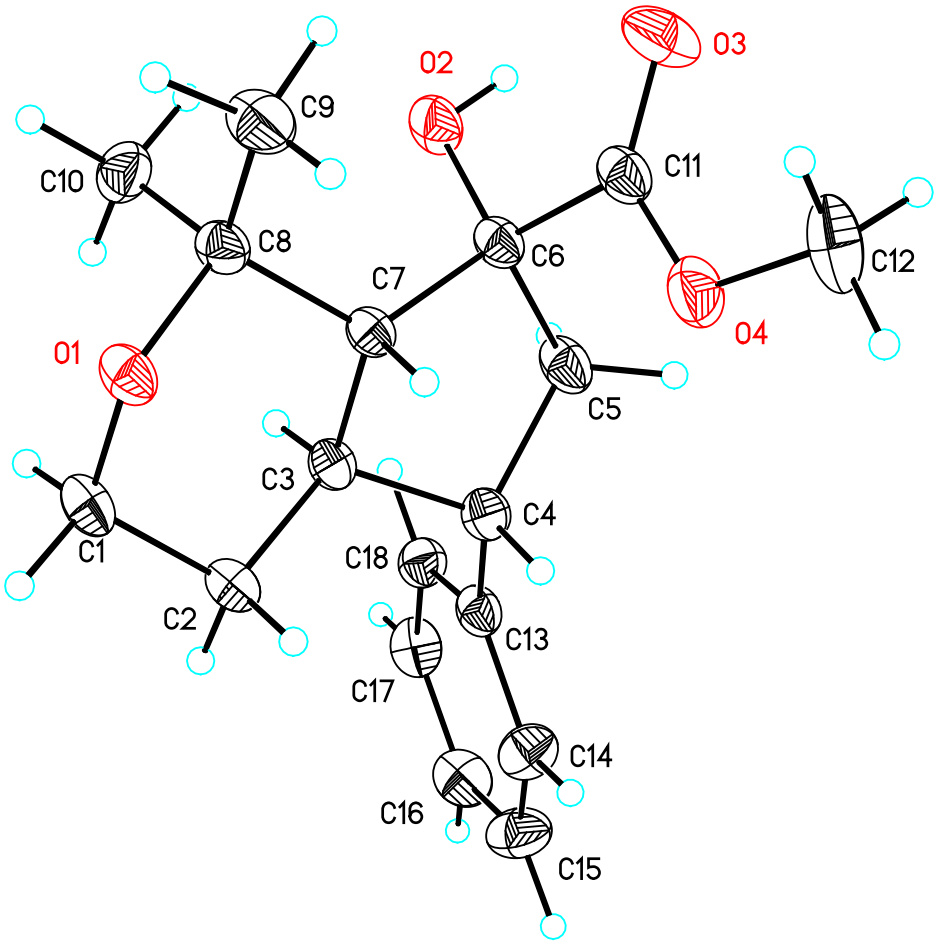
Table 7. Hydrogen bonds for **9c** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(1)-H(1C)...Br(1)#1	0.84	3.05	3.6918(17)	134.5

Symmetry transformations used to generate equivalent atoms:

#1 -x+1,y-1/2,-z+3/2





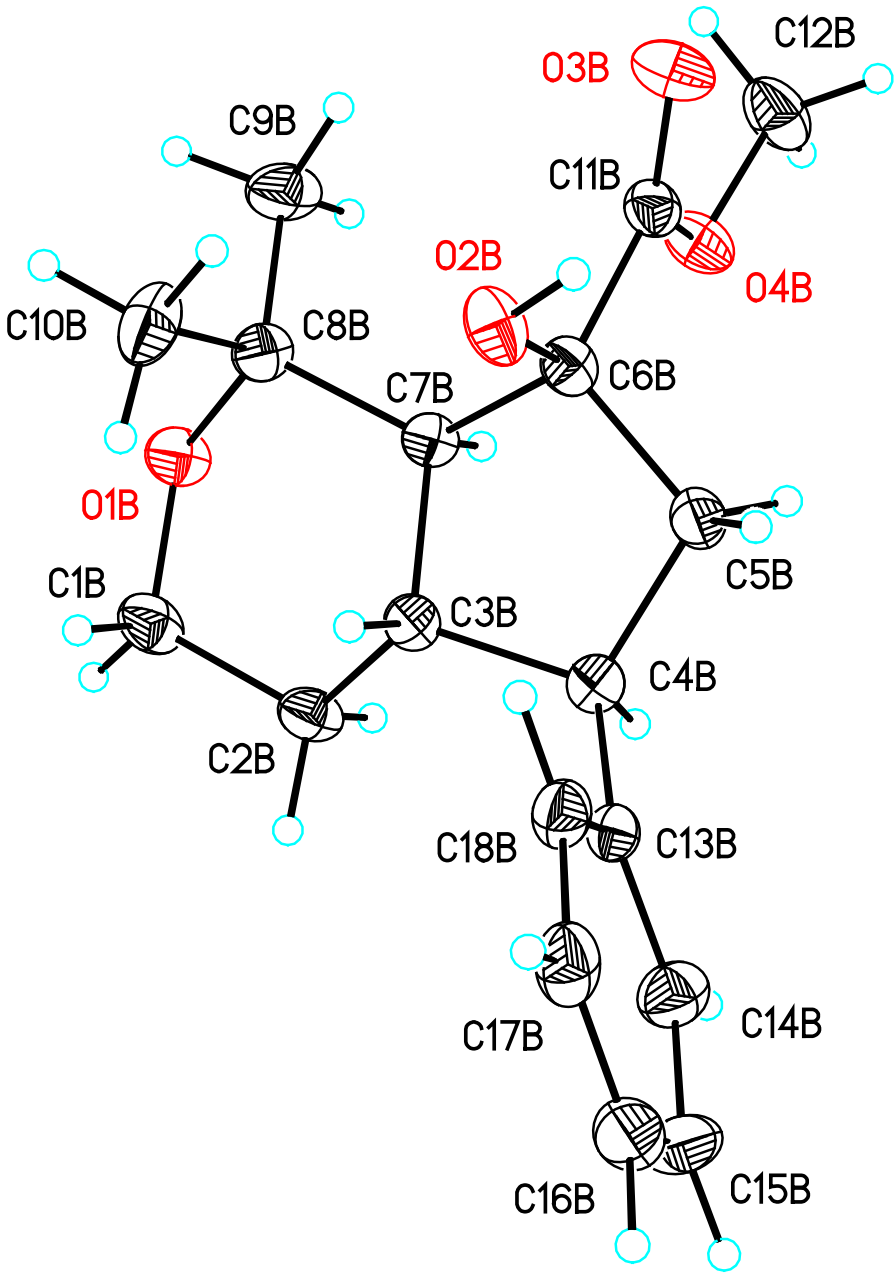


Table 1. Crystal data and structure refinement for **12**.

Identification code	**12**	
Empirical formula	C18 H24 O4	
Formula weight	304.37	
Temperature	173(2) K	
Wavelength	1.54178 Å	
Crystal system	Monoclinic	
Space group	P2(1)/n	
Unit cell dimensions	a = 12.1533(3) Å	⟨= 90°.
	b = 35.1495(7) Å	®= 110.6840(10)°.
	c = 12.2922(3) Å	© = 90°.
Volume	4912.5(2) Å^3	
Z	12	
Density (calculated)	1.235 Mg/m^3	
Absorption coefficient	0.696 mm^{-1}	
F(000)	1968	
Crystal size	0.40 x 0.25 x 0.13 mm^3	
Theta range for data collection	2.51 to 67.32°.	
Index ranges	-14<=h<=14, -37<=k<=41, -14<=l<=13	
Reflections collected	41157	
Independent reflections	8401 [R(int) = 0.1191]	
Completeness to theta = 67.32°	95.1 %	

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9149 and 0.7681
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	8401 / 0 / 596
Goodness-of-fit on F^2	1.132
Final R indices [I>2sigma(I)]	R1 = 0.0801, wR2 = 0.2275
R indices (all data)	R1 = 0.0976, wR2 = 0.2637
Extinction coefficient	0.00078(18)
Largest diff. peak and hole	0.395 and -0.592 e.Å^{-3}

Table 2. Atomic coordinates (x 10^4) and equivalent isotropic displacement parameters ($Å^2$x 10^3) for **12**. U(eq) is defined as one third of the trace of the orthogonalized U^{ij} tensor.

	x	y	z	U(eq)
C(1)	5739(3)	239(1)	1776(3)	38(1)
C(2)	5803(2)	35(1)	2887(2)	35(1)
C(3)	6875(2)	170(1)	3888(2)	31(1)
C(4)	6977(2)	53(1)	5116(2)	34(1)
C(5)	7880(3)	339(1)	5880(3)	41(1)
C(6)	7800(2)	706(1)	5141(3)	33(1)
C(7)	6860(2)	605(1)	3950(2)	30(1)
C(8)	6826(2)	797(1)	2816(3)	36(1)
C(9)	6588(3)	1219(1)	2822(3)	49(1)
C(10)	7898(3)	723(1)	2464(3)	44(1)
C(11)	7414(2)	1047(1)	5692(2)	36(1)
C(12)	5967(3)	1267(1)	6417(3)	63(1)
C(13)	7275(2)	-362(1)	5398(2)	35(1)
C(14)	6475(3)	-601(1)	5627(3)	43(1)
C(15)	6714(3)	-984(1)	5862(3)	48(1)
C(16)	7757(3)	-1137(1)	5859(3)	46(1)
C(17)	8570(3)	-905(1)	5633(3)	43(1)
C(18)	8323(2)	-519(1)	5402(3)	39(1)

O(1)	5771(2)	646(1)	1916(2)	37(1)
O(2)	8893(2)	786(1)	5022(2)	42(1)
O(3)	7947(2)	1339(1)	5977(2)	59(1)
O(4)	6405(2)	973(1)	5850(2)	50(1)
C(1B)	845(3)	1412(1)	-3083(3)	43(1)
C(2B)	1044(3)	1661(1)	-2016(3)	39(1)
C(3B)	2015(2)	1487(1)	-979(2)	32(1)
C(4B)	2237(2)	1667(1)	217(2)	35(1)
C(5B)	2651(3)	1330(1)	1091(3)	38(1)
C(6B)	2612(2)	965(1)	364(2)	33(1)
C(7B)	1706(2)	1073(1)	-833(2)	31(1)
C(8B)	1534(2)	833(1)	-1920(3)	38(1)
C(9B)	1002(3)	446(1)	-1850(3)	50(1)
C(10B)	2626(3)	782(1)	-2250(3)	51(1)
C(11B)	2250(2)	618(1)	910(3)	35(1)
C(12B)	713(3)	360(1)	1449(3)	52(1)
C(13B)	3086(3)	1997(1)	459(3)	35(1)
C(14B)	2748(3)	2363(1)	632(3)	46(1)
C(15B)	3532(3)	2668(1)	798(3)	55(1)
C(16B)	4641(3)	2605(1)	777(3)	50(1)
C(17B)	4995(3)	2242(1)	621(3)	45(1)
C(18B)	4226(2)	1943(1)	467(3)	38(1)
O(1B)	608(2)	1025(1)	-2872(2)	41(1)

O(2B)	3704(2)	899(1)	235(2)	41(1)
O(3B)	2847(2)	346(1)	1282(2)	51(1)
O(4B)	1166(2)	665(1)	939(2)	43(1)
C(1C)	9901(3)	1933(1)	3197(3)	40(1)
C(2C)	9725(3)	1678(1)	2144(3)	41(1)
C(3C)	8787(2)	1853(1)	1079(2)	32(1)
C(4C)	8628(2)	1674(1)	-102(2)	36(1)
C(5C)	8194(3)	2006(1)	-992(3)	40(1)
C(6C)	8257(2)	2375(1)	-268(2)	32(1)
C(7C)	9116(2)	2266(1)	946(2)	32(1)
C(8C)	9255(3)	2509(1)	2026(3)	37(1)
C(9C)	9791(3)	2894(1)	1951(3)	48(1)
C(10C)	8156(3)	2562(1)	2336(3)	48(1)
C(11C)	8656(3)	2710(1)	-832(3)	35(1)
C(12C)	10229(3)	2951(1)	-1345(3)	61(1)
C(13C)	7837(2)	1328(1)	-387(2)	33(1)
C(14C)	8243(3)	977(1)	-606(3)	43(1)
C(15C)	7505(3)	659(1)	-861(3)	54(1)
C(16C)	6373(3)	690(1)	-879(3)	56(1)
C(17C)	5949(3)	1039(1)	-669(3)	48(1)
C(18C)	6676(3)	1353(1)	-430(3)	40(1)
O(1C)	10162(2)	2318(1)	2984(2)	38(1)
O(2C)	7151(2)	2459(1)	-188(2)	44(1)

O(3C)	8063(2)	2976(1)	-1279(2)	55(1)
O(4C)	9763(2)	2661(1)	-790(2)	46(1)

Table 3. Bond lengths [Å] and angles [°] for **12**.

C(1)-O(1)	1.439(3)
C(1)-C(2)	1.521(4)
C(1)-H(1A)	0.9900
C(1)-H(1B)	0.9900
C(2)-C(3)	1.518(4)
C(2)-H(2A)	0.9900
C(2)-H(2B)	0.9900
C(3)-C(4)	1.526(4)
C(3)-C(7)	1.534(3)
C(3)-H(3A)	1.0000
C(4)-C(13)	1.514(3)
C(4)-C(5)	1.538(4)
C(4)-H(4A)	1.0000
C(5)-C(6)	1.561(3)
C(5)-H(5A)	0.9900
C(5)-H(5B)	0.9900
C(6)-O(2)	1.415(3)
C(6)-C(11)	1.529(3)
C(6)-C(7)	1.547(4)
C(7)-C(8)	1.535(4)
C(7)-H(7A)	1.0000

C(8)-O(1)	1.465(3)
C(8)-C(9)	1.513(3)
C(8)-C(10)	1.532(3)
C(9)-H(9A)	0.9800
C(9)-H(9B)	0.9800
C(9)-H(9C)	0.9800
C(10)-H(10A)	0.9800
C(10)-H(10B)	0.9800
C(10)-H(10C)	0.9800
C(11)-O(3)	1.200(3)
C(11)-O(4)	1.333(3)
C(12)-O(4)	1.449(3)
C(12)-H(12A)	0.9800
C(12)-H(12B)	0.9800
C(12)-H(12C)	0.9800
C(13)-C(18)	1.386(4)
C(13)-C(14)	1.387(3)
C(14)-C(15)	1.386(4)
C(14)-H(14A)	0.9500
C(15)-C(16)	1.378(4)
C(15)-H(15A)	0.9500
C(16)-C(17)	1.384(4)
C(16)-H(16A)	0.9500

C(17)-C(18)	1.398(3)
C(17)-H(17A)	0.9500
C(18)-H(18A)	0.9500
O(2)-H(2C)	0.8400
C(1B)-O(1B)	1.435(3)
C(1B)-C(2B)	1.523(4)
C(1B)-H(1BA)	0.9900
C(1B)-H(1BB)	0.9900
C(2B)-C(3B)	1.526(4)
C(2B)-H(2BA)	0.9900
C(2B)-H(2BB)	0.9900
C(3B)-C(7B)	1.530(3)
C(3B)-C(4B)	1.534(4)
C(3B)-H(3BA)	1.0000
C(4B)-C(13B)	1.510(3)
C(4B)-C(5B)	1.557(4)
C(4B)-H(4BA)	1.0000
C(5B)-C(6B)	1.556(3)
C(5B)-H(5BA)	0.9900
C(5B)-H(5BB)	0.9900
C(6B)-O(2B)	1.409(3)
C(6B)-C(11B)	1.529(3)
C(6B)-C(7B)	1.542(4)

C(7B)-C(8B)	1.532(4)
C(7B)-H(7BA)	1.0000
C(8B)-O(1B)	1.471(3)
C(8B)-C(9B)	1.520(3)
C(8B)-C(10B)	1.528(4)
C(9B)-H(9BA)	0.9800
C(9B)-H(9BB)	0.9800
C(9B)-H(9BC)	0.9800
C(10B)-H(10D)	0.9800
C(10B)-H(10E)	0.9800
C(10B)-H(10F)	0.9800
C(11B)-O(3B)	1.192(3)
C(11B)-O(4B)	1.341(3)
C(12B)-O(4B)	1.446(3)
C(12B)-H(12D)	0.9800
C(12B)-H(12E)	0.9800
C(12B)-H(12F)	0.9800
C(13B)-C(14B)	1.390(3)
C(13B)-C(18B)	1.395(4)
C(14B)-C(15B)	1.402(4)
C(14B)-H(14B)	0.9500
C(15B)-C(16B)	1.375(5)
C(15B)-H(15B)	0.9500

C(16B)-C(17B)	1.379(4)
C(16B)-H(16B)	0.9500
C(17B)-C(18B)	1.375(4)
C(17B)-H(17B)	0.9500
C(18B)-H(18B)	0.9500
O(2B)-H(2BC)	0.8400
C(1C)-O(1C)	1.432(3)
C(1C)-C(2C)	1.527(4)
C(1C)-H(1CA)	0.9900
C(1C)-H(1CB)	0.9900
C(2C)-C(3C)	1.529(4)
C(2C)-H(2CA)	0.9900
C(2C)-H(2CB)	0.9900
C(3C)-C(7C)	1.530(3)
C(3C)-C(4C)	1.531(4)
C(3C)-H(3CA)	1.0000
C(4C)-C(13C)	1.513(3)
C(4C)-C(5C)	1.558(4)
C(4C)-H(4CA)	1.0000
C(5C)-C(6C)	1.560(3)
C(5C)-H(5CA)	0.9900
C(5C)-H(5CB)	0.9900
C(6C)-O(2C)	1.413(3)

C(6C)-C(11C)	1.530(3)
C(6C)-C(7C)	1.537(4)
C(7C)-C(8C)	1.538(4)
C(7C)-H(7CA)	1.0000
C(8C)-O(1C)	1.462(4)
C(8C)-C(9C)	1.518(3)
C(8C)-C(10C)	1.525(4)
C(9C)-H(9CA)	0.9800
C(9C)-H(9CB)	0.9800
C(9C)-H(9CC)	0.9800
C(10C)-H(10G)	0.9800
C(10C)-H(10H)	0.9800
C(10C)-H(10I)	0.9800
C(11C)-O(3C)	1.190(3)
C(11C)-O(4C)	1.340(3)
C(12C)-O(4C)	1.450(3)
C(12C)-H(12G)	0.9800
C(12C)-H(12H)	0.9800
C(12C)-H(12I)	0.9800
C(13C)-C(14C)	1.391(3)
C(13C)-C(18C)	1.396(4)
C(14C)-C(15C)	1.397(4)
C(14C)-H(14C)	0.9500

C(15C)-C(16C)	1.373(5)
C(15C)-H(15C)	0.9500
C(16C)-C(17C)	1.388(5)
C(16C)-H(16C)	0.9500
C(17C)-C(18C)	1.379(4)
C(17C)-H(17C)	0.9500
C(18C)-H(18C)	0.9500
O(2C)-H(2CC)	0.8400

O(1)-C(1)-C(2)	111.8(2)
O(1)-C(1)-H(1A)	109.3
C(2)-C(1)-H(1A)	109.3
O(1)-C(1)-H(1B)	109.3
C(2)-C(1)-H(1B)	109.3
H(1A)-C(1)-H(1B)	107.9
C(3)-C(2)-C(1)	109.7(2)
C(3)-C(2)-H(2A)	109.7
C(1)-C(2)-H(2A)	109.7
C(3)-C(2)-H(2B)	109.7
C(1)-C(2)-H(2B)	109.7
H(2A)-C(2)-H(2B)	108.2
C(2)-C(3)-C(4)	117.7(2)
C(2)-C(3)-C(7)	109.3(2)

C(4)-C(3)-C(7)	102.51(19)
C(2)-C(3)-H(3A)	109.0
C(4)-C(3)-H(3A)	109.0
C(7)-C(3)-H(3A)	109.0
C(13)-C(4)-C(3)	114.4(2)
C(13)-C(4)-C(5)	115.3(2)
C(3)-C(4)-C(5)	102.7(2)
C(13)-C(4)-H(4A)	108.0
C(3)-C(4)-H(4A)	108.0
C(5)-C(4)-H(4A)	108.0
C(4)-C(5)-C(6)	107.4(2)
C(4)-C(5)-H(5A)	110.2
C(6)-C(5)-H(5A)	110.2
C(4)-C(5)-H(5B)	110.2
C(6)-C(5)-H(5B)	110.2
H(5A)-C(5)-H(5B)	108.5
O(2)-C(6)-C(11)	109.6(2)
O(2)-C(6)-C(7)	110.8(2)
C(11)-C(6)-C(7)	111.0(2)
O(2)-C(6)-C(5)	110.9(2)
C(11)-C(6)-C(5)	110.8(2)
C(7)-C(6)-C(5)	103.6(2)
C(3)-C(7)-C(8)	112.9(2)

C(3)-C(7)-C(6)	104.9(2)
C(8)-C(7)-C(6)	122.2(2)
C(3)-C(7)-H(7A)	105.1
C(8)-C(7)-H(7A)	105.1
C(6)-C(7)-H(7A)	105.1
O(1)-C(8)-C(9)	103.9(2)
O(1)-C(8)-C(10)	109.3(2)
C(9)-C(8)-C(10)	110.5(2)
O(1)-C(8)-C(7)	105.21(19)
C(9)-C(8)-C(7)	111.7(2)
C(10)-C(8)-C(7)	115.4(2)
C(8)-C(9)-H(9A)	109.5
C(8)-C(9)-H(9B)	109.5
H(9A)-C(9)-H(9B)	109.5
C(8)-C(9)-H(9C)	109.5
H(9A)-C(9)-H(9C)	109.5
H(9B)-C(9)-H(9C)	109.5
C(8)-C(10)-H(10A)	109.5
C(8)-C(10)-H(10B)	109.5
H(10A)-C(10)-H(10B)	109.5
C(8)-C(10)-H(10C)	109.5
H(10A)-C(10)-H(10C)	109.5
H(10B)-C(10)-H(10C)	109.5

O(3)-C(11)-O(4)	123.6(2)
O(3)-C(11)-C(6)	125.6(2)
O(4)-C(11)-C(6)	110.8(2)
O(4)-C(12)-H(12A)	109.5
O(4)-C(12)-H(12B)	109.5
H(12A)-C(12)-H(12B)	109.5
O(4)-C(12)-H(12C)	109.5
H(12A)-C(12)-H(12C)	109.5
H(12B)-C(12)-H(12C)	109.5
C(18)-C(13)-C(14)	117.9(2)
C(18)-C(13)-C(4)	122.0(2)
C(14)-C(13)-C(4)	120.1(2)
C(15)-C(14)-C(13)	121.3(3)
C(15)-C(14)-H(14A)	119.3
C(13)-C(14)-H(14A)	119.3
C(16)-C(15)-C(14)	120.3(3)
C(16)-C(15)-H(15A)	119.8
C(14)-C(15)-H(15A)	119.8
C(15)-C(16)-C(17)	119.5(3)
C(15)-C(16)-H(16A)	120.3
C(17)-C(16)-H(16A)	120.3
C(16)-C(17)-C(18)	119.8(3)
C(16)-C(17)-H(17A)	120.1

C(18)-C(17)-H(17A)	120.1
C(13)-C(18)-C(17)	121.2(2)
C(13)-C(18)-H(18A)	119.4
C(17)-C(18)-H(18A)	119.4
C(1)-O(1)-C(8)	115.4(2)
C(6)-O(2)-H(2C)	109.5
C(11)-O(4)-C(12)	116.4(2)
O(1B)-C(1B)-C(2B)	111.7(2)
O(1B)-C(1B)-H(1BA)	109.3
C(2B)-C(1B)-H(1BA)	109.3
O(1B)-C(1B)-H(1BB)	109.3
C(2B)-C(1B)-H(1BB)	109.3
H(1BA)-C(1B)-H(1BB)	107.9
C(1B)-C(2B)-C(3B)	109.2(2)
C(1B)-C(2B)-H(2BA)	109.8
C(3B)-C(2B)-H(2BA)	109.8
C(1B)-C(2B)-H(2BB)	109.8
C(3B)-C(2B)-H(2BB)	109.8
H(2BA)-C(2B)-H(2BB)	108.3
C(2B)-C(3B)-C(7B)	109.3(2)
C(2B)-C(3B)-C(4B)	117.2(2)
C(7B)-C(3B)-C(4B)	104.3(2)
C(2B)-C(3B)-H(3BA)	108.6

C(7B)-C(3B)-H(3BA)	108.6
C(4B)-C(3B)-H(3BA)	108.6
C(13B)-C(4B)-C(3B)	112.5(2)
C(13B)-C(4B)-C(5B)	114.6(2)
C(3B)-C(4B)-C(5B)	104.64(19)
C(13B)-C(4B)-H(4BA)	108.3
C(3B)-C(4B)-H(4BA)	108.3
C(5B)-C(4B)-H(4BA)	108.3
C(6B)-C(5B)-C(4B)	107.1(2)
C(6B)-C(5B)-H(5BA)	110.3
C(4B)-C(5B)-H(5BA)	110.3
C(6B)-C(5B)-H(5BB)	110.3
C(4B)-C(5B)-H(5BB)	110.3
H(5BA)-C(5B)-H(5BB)	108.5
O(2B)-C(6B)-C(11B)	110.3(2)
O(2B)-C(6B)-C(7B)	108.5(2)
C(11B)-C(6B)-C(7B)	113.3(2)
O(2B)-C(6B)-C(5B)	111.1(2)
C(11B)-C(6B)-C(5B)	111.1(2)
C(7B)-C(6B)-C(5B)	102.2(2)
C(3B)-C(7B)-C(8B)	112.7(2)
C(3B)-C(7B)-C(6B)	102.9(2)
C(8B)-C(7B)-C(6B)	121.9(2)

C(3B)-C(7B)-H(7BA)	106.1
C(8B)-C(7B)-H(7BA)	106.1
C(6B)-C(7B)-H(7BA)	106.1
O(1B)-C(8B)-C(9B)	103.5(2)
O(1B)-C(8B)-C(10B)	109.6(2)
C(9B)-C(8B)-C(10B)	109.8(2)
O(1B)-C(8B)-C(7B)	105.70(19)
C(9B)-C(8B)-C(7B)	111.9(2)
C(10B)-C(8B)-C(7B)	115.6(2)
C(8B)-C(9B)-H(9BA)	109.5
C(8B)-C(9B)-H(9BB)	109.5
H(9BA)-C(9B)-H(9BB)	109.5
C(8B)-C(9B)-H(9BC)	109.5
H(9BA)-C(9B)-H(9BC)	109.5
H(9BB)-C(9B)-H(9BC)	109.5
C(8B)-C(10B)-H(10D)	109.5
C(8B)-C(10B)-H(10E)	109.5
H(10D)-C(10B)-H(10E)	109.5
C(8B)-C(10B)-H(10F)	109.5
H(10D)-C(10B)-H(10F)	109.5
H(10E)-C(10B)-H(10F)	109.5
O(3B)-C(11B)-O(4B)	124.0(2)
O(3B)-C(11B)-C(6B)	125.4(2)

O(4B)-C(11B)-C(6B)	110.6(2)
O(4B)-C(12B)-H(12D)	109.5
O(4B)-C(12B)-H(12E)	109.5
H(12D)-C(12B)-H(12E)	109.5
O(4B)-C(12B)-H(12F)	109.5
H(12D)-C(12B)-H(12F)	109.5
H(12E)-C(12B)-H(12F)	109.5
C(14B)-C(13B)-C(18B)	118.1(2)
C(14B)-C(13B)-C(4B)	121.6(2)
C(18B)-C(13B)-C(4B)	120.3(2)
C(13B)-C(14B)-C(15B)	120.6(3)
C(13B)-C(14B)-H(14B)	119.7
C(15B)-C(14B)-H(14B)	119.7
C(16B)-C(15B)-C(14B)	119.5(3)
C(16B)-C(15B)-H(15B)	120.3
C(14B)-C(15B)-H(15B)	120.3
C(15B)-C(16B)-C(17B)	120.6(3)
C(15B)-C(16B)-H(16B)	119.7
C(17B)-C(16B)-H(16B)	119.7
C(18B)-C(17B)-C(16B)	119.7(3)
C(18B)-C(17B)-H(17B)	120.2
C(16B)-C(17B)-H(17B)	120.2
C(17B)-C(18B)-C(13B)	121.5(2)

C(17B)-C(18B)-H(18B)	119.3
C(13B)-C(18B)-H(18B)	119.3
C(1B)-O(1B)-C(8B)	115.7(2)
C(6B)-O(2B)-H(2BC)	109.5
C(11B)-O(4B)-C(12B)	116.7(2)
O(1C)-C(1C)-C(2C)	111.9(2)
O(1C)-C(1C)-H(1CA)	109.2
C(2C)-C(1C)-H(1CA)	109.2
O(1C)-C(1C)-H(1CB)	109.2
C(2C)-C(1C)-H(1CB)	109.2
H(1CA)-C(1C)-H(1CB)	107.9
C(1C)-C(2C)-C(3C)	109.2(2)
C(1C)-C(2C)-H(2CA)	109.8
C(3C)-C(2C)-H(2CA)	109.8
C(1C)-C(2C)-H(2CB)	109.8
C(3C)-C(2C)-H(2CB)	109.8
H(2CA)-C(2C)-H(2CB)	108.3
C(2C)-C(3C)-C(7C)	109.2(2)
C(2C)-C(3C)-C(4C)	116.9(2)
C(7C)-C(3C)-C(4C)	103.8(2)
C(2C)-C(3C)-H(3CA)	108.9
C(7C)-C(3C)-H(3CA)	108.9
C(4C)-C(3C)-H(3CA)	108.9

C(13C)-C(4C)-C(3C)	114.0(2)
C(13C)-C(4C)-C(5C)	114.1(2)
C(3C)-C(4C)-C(5C)	104.82(19)
C(13C)-C(4C)-H(4CA)	107.8
C(3C)-C(4C)-H(4CA)	107.8
C(5C)-C(4C)-H(4CA)	107.8
C(4C)-C(5C)-C(6C)	106.7(2)
C(4C)-C(5C)-H(5CA)	110.4
C(6C)-C(5C)-H(5CA)	110.4
C(4C)-C(5C)-H(5CB)	110.4
C(6C)-C(5C)-H(5CB)	110.4
H(5CA)-C(5C)-H(5CB)	108.6
O(2C)-C(6C)-C(11C)	109.4(2)
O(2C)-C(6C)-C(7C)	108.7(2)
C(11C)-C(6C)-C(7C)	114.6(2)
O(2C)-C(6C)-C(5C)	110.9(2)
C(11C)-C(6C)-C(5C)	110.1(2)
C(7C)-C(6C)-C(5C)	103.0(2)
C(3C)-C(7C)-C(6C)	103.1(2)
C(3C)-C(7C)-C(8C)	112.8(2)
C(6C)-C(7C)-C(8C)	122.0(2)
C(3C)-C(7C)-H(7CA)	105.9
C(6C)-C(7C)-H(7CA)	105.9

C(8C)-C(7C)-H(7CA)	105.9
O(1C)-C(8C)-C(9C)	104.0(2)
O(1C)-C(8C)-C(10C)	109.4(2)
C(9C)-C(8C)-C(10C)	109.8(2)
O(1C)-C(8C)-C(7C)	105.15(19)
C(9C)-C(8C)-C(7C)	111.2(2)
C(10C)-C(8C)-C(7C)	116.4(2)
C(8C)-C(9C)-H(9CA)	109.5
C(8C)-C(9C)-H(9CB)	109.5
H(9CA)-C(9C)-H(9CB)	109.5
C(8C)-C(9C)-H(9CC)	109.5
H(9CA)-C(9C)-H(9CC)	109.5
H(9CB)-C(9C)-H(9CC)	109.5
C(8C)-C(10C)-H(10G)	109.5
C(8C)-C(10C)-H(10H)	109.5
H(10G)-C(10C)-H(10H)	109.5
C(8C)-C(10C)-H(10I)	109.5
H(10G)-C(10C)-H(10I)	109.5
H(10H)-C(10C)-H(10I)	109.5
O(3C)-C(11C)-O(4C)	123.8(2)
O(3C)-C(11C)-C(6C)	125.3(2)
O(4C)-C(11C)-C(6C)	110.9(2)
O(4C)-C(12C)-H(12G)	109.5

O(4C)-C(12C)-H(12H)	109.5
H(12G)-C(12C)-H(12H)	109.5
O(4C)-C(12C)-H(12I)	109.5
H(12G)-C(12C)-H(12I)	109.5
H(12H)-C(12C)-H(12I)	109.5
C(14C)-C(13C)-C(18C)	118.2(2)
C(14C)-C(13C)-C(4C)	121.2(2)
C(18C)-C(13C)-C(4C)	120.6(2)
C(13C)-C(14C)-C(15C)	120.6(3)
C(13C)-C(14C)-H(14C)	119.7
C(15C)-C(14C)-H(14C)	119.7
C(16C)-C(15C)-C(14C)	120.0(3)
C(16C)-C(15C)-H(15C)	120.0
C(14C)-C(15C)-H(15C)	120.0
C(15C)-C(16C)-C(17C)	120.2(3)
C(15C)-C(16C)-H(16C)	119.9
C(17C)-C(16C)-H(16C)	119.9
C(18C)-C(17C)-C(16C)	119.6(3)
C(18C)-C(17C)-H(17C)	120.2
C(16C)-C(17C)-H(17C)	120.2
C(17C)-C(18C)-C(13C)	121.4(3)
C(17C)-C(18C)-H(18C)	119.3
C(13C)-C(18C)-H(18C)	119.3

C(1C)-O(1C)-C(8C)	115.4(2)
C(6C)-O(2C)-H(2CC)	109.5
C(11C)-O(4C)-C(12C)	116.4(2)

Symmetry transformations used to generate equivalent atoms:

Table 4. Anisotropic displacement parameters ($Å^2 x\ 10^3$) for **12**. The anisotropic displacement factor exponent takes the form: $-2\pi^2[h^2 a^{*2} U^{11} + ... + 2 h k a^* b^* U^{12}]$

	U^{11}	U^{22}	U^{33}	U^{23}	U^{13}	U^{12}
C(1)	46(2)	26(1)	46(2)	-5(1)	22(1)	2(1)
C(2)	38(2)	25(1)	47(2)	-5(1)	21(1)	-2(1)
C(3)	37(1)	22(1)	43(2)	-2(1)	25(1)	1(1)
C(4)	39(2)	27(1)	45(2)	0(1)	26(1)	3(1)
C(5)	59(2)	25(1)	45(2)	0(1)	26(2)	1(1)
C(6)	38(2)	25(1)	41(2)	-2(1)	21(1)	-2(1)
C(7)	37(1)	22(1)	39(2)	-3(1)	22(1)	0(1)
C(8)	41(2)	25(1)	47(2)	1(1)	23(1)	-2(1)
C(9)	70(2)	24(1)	55(2)	3(1)	24(2)	-1(1)
C(10)	45(2)	48(2)	47(2)	5(1)	27(2)	-4(1)
C(11)	43(2)	28(1)	40(2)	-2(1)	19(1)	-1(1)
C(12)	78(3)	63(2)	62(2)	-14(2)	40(2)	20(2)
C(13)	49(2)	23(1)	40(2)	-1(1)	26(1)	0(1)
C(14)	45(2)	37(1)	57(2)	6(1)	33(2)	4(1)
C(15)	53(2)	37(2)	61(2)	10(1)	28(2)	-7(1)
C(16)	61(2)	26(1)	51(2)	6(1)	22(2)	3(1)
C(17)	50(2)	32(1)	54(2)	4(1)	28(2)	11(1)
C(18)	47(2)	31(1)	48(2)	4(1)	30(2)	1(1)

O(1)	44(1)	26(1)	43(1)	-1(1)	18(1)	1(1)
O(2)	40(1)	45(1)	48(1)	-6(1)	24(1)	-5(1)
O(3)	74(2)	29(1)	86(2)	-16(1)	40(2)	-10(1)
O(4)	57(1)	46(1)	61(2)	-17(1)	39(1)	-2(1)
C(1B)	50(2)	30(1)	50(2)	4(1)	21(2)	-8(1)
C(2B)	47(2)	25(1)	49(2)	6(1)	22(2)	-1(1)
C(3B)	38(2)	22(1)	45(2)	0(1)	24(1)	-4(1)
C(4B)	40(2)	25(1)	51(2)	-2(1)	30(1)	-2(1)
C(5B)	53(2)	28(1)	43(2)	-1(1)	27(2)	-8(1)
C(6B)	37(2)	26(1)	44(2)	3(1)	26(1)	-1(1)
C(7B)	35(1)	21(1)	45(2)	1(1)	23(1)	-3(1)
C(8B)	43(2)	25(1)	48(2)	-1(1)	19(1)	2(1)
C(9B)	61(2)	26(1)	57(2)	-2(1)	13(2)	-5(1)
C(10B)	53(2)	53(2)	53(2)	-14(2)	28(2)	7(1)
C(11B)	44(2)	25(1)	43(2)	3(1)	26(1)	-1(1)
C(12B)	61(2)	43(2)	66(2)	13(2)	39(2)	-12(1)
C(13B)	50(2)	21(1)	43(2)	-1(1)	29(2)	-2(1)
C(14B)	56(2)	30(1)	58(2)	-4(1)	30(2)	5(1)
C(15B)	81(3)	24(1)	66(2)	-5(1)	34(2)	-3(1)
C(16B)	65(2)	39(2)	50(2)	-4(1)	26(2)	-21(1)
C(17B)	49(2)	46(2)	48(2)	-5(1)	28(2)	-11(1)
C(18B)	46(2)	32(1)	46(2)	-5(1)	29(2)	-6(1)
O(1B)	50(1)	27(1)	49(1)	2(1)	19(1)	-3(1)

O(2B)	37(1)	47(1)	49(1)	6(1)	26(1)	3(1)
O(3B)	58(1)	32(1)	71(2)	14(1)	33(1)	8(1)
O(4B)	46(1)	34(1)	60(1)	13(1)	34(1)	-1(1)
C(1C)	47(2)	31(1)	43(2)	4(1)	19(2)	-3(1)
C(2C)	54(2)	26(1)	47(2)	5(1)	25(2)	0(1)
C(3C)	40(2)	23(1)	41(2)	0(1)	24(1)	-3(1)
C(4C)	43(2)	24(1)	51(2)	-3(1)	31(2)	-3(1)
C(5C)	56(2)	30(1)	40(2)	0(1)	25(2)	-7(1)
C(6C)	36(2)	25(1)	45(2)	3(1)	25(1)	0(1)
C(7C)	38(2)	22(1)	43(2)	0(1)	24(1)	-2(1)
C(8C)	45(2)	25(1)	46(2)	-2(1)	22(2)	2(1)
C(9C)	60(2)	28(1)	54(2)	-3(1)	17(2)	-5(1)
C(10C)	50(2)	49(2)	53(2)	-8(1)	27(2)	10(1)
C(11C)	46(2)	25(1)	43(2)	0(1)	25(1)	0(1)
C(12C)	69(2)	61(2)	65(2)	14(2)	41(2)	-19(2)
C(13C)	45(2)	23(1)	41(2)	-1(1)	27(1)	-3(1)
C(14C)	49(2)	32(1)	55(2)	-3(1)	28(2)	3(1)
C(15C)	78(2)	27(1)	61(2)	-7(1)	30(2)	-4(1)
C(16C)	72(2)	47(2)	51(2)	-3(2)	26(2)	-29(2)
C(17C)	46(2)	61(2)	44(2)	-4(2)	23(2)	-16(1)
C(18C)	48(2)	36(1)	49(2)	-6(1)	32(2)	-5(1)
O(1C)	42(1)	29(1)	45(1)	2(1)	18(1)	-1(1)
O(2C)	35(1)	59(1)	46(1)	7(1)	23(1)	6(1)

O(3C)	71(2)	31(1)	72(2)	17(1)	39(1)	15(1)
O(4C)	45(1)	46(1)	58(1)	16(1)	32(1)	-3(1)

Table 5. Hydrogen coordinates (x 10^4) and isotropic displacement parameters ($Å^2$x 10^3) for **12**.

	x	y	z	U(eq)
H(1A)	6408	158	1549	45
H(1B)	5003	166	1142	45
H(2A)	5082	87	3061	42
H(2B)	5854	-243	2784	42
H(3A)	7601	87	3746	37
H(4A)	6204	105	5205	41
H(5A)	8682	230	6122	49
H(5B)	7701	399	6587	49
H(7A)	6090	670	4027	37
H(9A)	5897	1261	3040	73
H(9B)	6443	1324	2045	73
H(9C)	7272	1346	3385	73
H(10A)	7798	857	1736	66
H(10B)	7972	450	2353	66
H(10C)	8609	816	3077	66
H(12A)	5227	1183	6489	95
H(12B)	5832	1501	5953	95

H(12C)	6547	1316	7193	95
H(14A)	5748	-500	5622	51
H(15A)	6157	-1141	6026	57
H(16A)	7916	-1400	6011	55
H(17A)	9294	-1008	5634	51
H(18A)	8884	-361	5245	46
H(2C)	9378	856	5667	63
H(1BA)	174	1514	-3741	51
H(1BB)	1552	1421	-3306	51
H(2BA)	308	1680	-1845	47
H(2BB)	1273	1921	-2165	47
H(3BA)	2766	1492	-1140	39
H(4BA)	1471	1762	239	42
H(5BA)	3461	1376	1639	46
H(5BB)	2125	1302	1543	46
H(7BA)	924	1079	-733	37
H(9BA)	298	480	-1645	75
H(9BB)	1577	291	-1255	75
H(9BC)	786	318	-2605	75
H(10D)	2430	623	-2947	76
H(10E)	3249	659	-1607	76
H(10F)	2898	1031	-2406	76
H(12D)	-81	424	1422	78

H(12E)	1229	323	2260	78
H(12F)	685	124	1013	78
H(14B)	1979	2407	638	55
H(15B)	3300	2918	925	66
H(16B)	5167	2812	871	60
H(17B)	5767	2200	621	54
H(18B)	4476	1694	363	45
H(2BC)	4193	823	869	62
H(1CA)	9178	1930	3394	48
H(1CB)	10553	1832	3873	48
H(2CA)	9475	1421	2292	49
H(2CB)	10475	1653	2003	49
H(3CA)	8015	1849	1199	39
H(4CA)	9421	1593	-92	43
H(5CA)	7376	1959	-1520	48
H(5CB)	8702	2029	-1465	48
H(7CA)	9913	2257	880	38
H(9CA)	9861	3044	2644	72
H(9CB)	9286	3029	1256	72
H(9CC)	10573	2858	1904	72
H(10G)	8342	2724	3026	72
H(10H)	7881	2314	2497	72
H(10I)	7539	2683	1683	72

H(12G)	11040	2887	-1262	91
H(12H)	10218	3198	-977	91
H(12I)	9745	2966	-2173	91
H(14C)	9030	952	-583	52
H(15C)	7786	421	-1022	65
H(16C)	5878	472	-1035	67
H(17C)	5163	1061	-690	58
H(18C)	6381	1591	-292	48
H(2CC)	6676	2520	-846	66

Table 6. Torsion angles [°] for **12**.

O(1)-C(1)-C(2)-C(3)	-54.1(3)
C(1)-C(2)-C(3)-C(4)	169.13(19)
C(1)-C(2)-C(3)-C(7)	52.9(2)
C(2)-C(3)-C(4)-C(13)	72.6(3)
C(7)-C(3)-C(4)-C(13)	-167.5(2)
C(2)-C(3)-C(4)-C(5)	-161.6(2)
C(7)-C(3)-C(4)-C(5)	-41.7(2)
C(13)-C(4)-C(5)-C(6)	152.1(2)
C(3)-C(4)-C(5)-C(6)	26.9(2)
C(4)-C(5)-C(6)-O(2)	-120.6(2)
C(4)-C(5)-C(6)-C(11)	117.5(2)
C(4)-C(5)-C(6)-C(7)	-1.7(2)
C(2)-C(3)-C(7)-C(8)	-57.5(3)
C(4)-C(3)-C(7)-C(8)	177.0(2)
C(2)-C(3)-C(7)-C(6)	167.14(18)
C(4)-C(3)-C(7)-C(6)	41.6(2)
O(2)-C(6)-C(7)-C(3)	94.7(2)
C(11)-C(6)-C(7)-C(3)	-143.27(19)
C(5)-C(6)-C(7)-C(3)	-24.3(2)
O(2)-C(6)-C(7)-C(8)	-35.4(3)
C(11)-C(6)-C(7)-C(8)	86.6(3)

C(5)-C(6)-C(7)-C(8)	-154.4(2)
C(3)-C(7)-C(8)-O(1)	57.5(3)
C(6)-C(7)-C(8)-O(1)	-175.89(19)
C(3)-C(7)-C(8)-C(9)	169.6(2)
C(6)-C(7)-C(8)-C(9)	-63.8(3)
C(3)-C(7)-C(8)-C(10)	-63.1(3)
C(6)-C(7)-C(8)-C(10)	63.5(3)
O(2)-C(6)-C(11)-O(3)	0.4(4)
C(7)-C(6)-C(11)-O(3)	-122.3(3)
C(5)-C(6)-C(11)-O(3)	123.1(3)
O(2)-C(6)-C(11)-O(4)	-177.8(2)
C(7)-C(6)-C(11)-O(4)	59.5(3)
C(5)-C(6)-C(11)-O(4)	-55.1(3)
C(3)-C(4)-C(13)-C(18)	62.6(4)
C(5)-C(4)-C(13)-C(18)	-56.3(4)
C(3)-C(4)-C(13)-C(14)	-115.4(3)
C(5)-C(4)-C(13)-C(14)	125.7(3)
C(18)-C(13)-C(14)-C(15)	0.5(5)
C(4)-C(13)-C(14)-C(15)	178.6(3)
C(13)-C(14)-C(15)-C(16)	-0.8(5)
C(14)-C(15)-C(16)-C(17)	0.8(5)
C(15)-C(16)-C(17)-C(18)	-0.5(5)
C(14)-C(13)-C(18)-C(17)	-0.2(5)

C(4)-C(13)-C(18)-C(17)	-178.2(3)
C(16)-C(17)-C(18)-C(13)	0.2(5)
C(2)-C(1)-O(1)-C(8)	59.9(3)
C(9)-C(8)-O(1)-C(1)	-176.5(2)
C(10)-C(8)-O(1)-C(1)	65.5(3)
C(7)-C(8)-O(1)-C(1)	-59.0(2)
O(3)-C(11)-O(4)-C(12)	-1.0(5)
C(6)-C(11)-O(4)-C(12)	177.3(3)
O(1B)-C(1B)-C(2B)-C(3B)	54.6(3)
C(1B)-C(2B)-C(3B)-C(7B)	-53.9(3)
C(1B)-C(2B)-C(3B)-C(4B)	-172.2(2)
C(2B)-C(3B)-C(4B)-C(13B)	-86.8(3)
C(7B)-C(3B)-C(4B)-C(13B)	152.2(2)
C(2B)-C(3B)-C(4B)-C(5B)	148.2(2)
C(7B)-C(3B)-C(4B)-C(5B)	27.3(2)
C(13B)-C(4B)-C(5B)-C(6B)	-125.5(2)
C(3B)-C(4B)-C(5B)-C(6B)	-1.9(3)
C(4B)-C(5B)-C(6B)-O(2B)	91.8(3)
C(4B)-C(5B)-C(6B)-C(11B)	-144.9(2)
C(4B)-C(5B)-C(6B)-C(7B)	-23.7(2)
C(2B)-C(3B)-C(7B)-C(8B)	58.0(3)
C(4B)-C(3B)-C(7B)-C(8B)	-175.9(2)
C(2B)-C(3B)-C(7B)-C(6B)	-168.90(19)

C(4B)-C(3B)-C(7B)-C(6B)	-42.8(2)
O(2B)-C(6B)-C(7B)-C(3B)	-77.0(2)
C(11B)-C(6B)-C(7B)-C(3B)	160.15(19)
C(5B)-C(6B)-C(7B)-C(3B)	40.5(2)
O(2B)-C(6B)-C(7B)-C(8B)	50.5(3)
C(11B)-C(6B)-C(7B)-C(8B)	-72.3(3)
C(5B)-C(6B)-C(7B)-C(8B)	168.0(2)
C(3B)-C(7B)-C(8B)-O(1B)	-57.0(3)
C(6B)-C(7B)-C(8B)-O(1B)	179.97(19)
C(3B)-C(7B)-C(8B)-C(9B)	-169.0(2)
C(6B)-C(7B)-C(8B)-C(9B)	68.0(3)
C(3B)-C(7B)-C(8B)-C(10B)	64.4(3)
C(6B)-C(7B)-C(8B)-C(10B)	-58.7(3)
O(2B)-C(6B)-C(11B)-O(3B)	6.8(4)
C(7B)-C(6B)-C(11B)-O(3B)	128.6(3)
C(5B)-C(6B)-C(11B)-O(3B)	-116.9(3)
O(2B)-C(6B)-C(11B)-O(4B)	-174.4(2)
C(7B)-C(6B)-C(11B)-O(4B)	-52.5(3)
C(5B)-C(6B)-C(11B)-O(4B)	61.9(3)
C(3B)-C(4B)-C(13B)-C(14B)	117.7(3)
C(5B)-C(4B)-C(13B)-C(14B)	-123.0(3)
C(3B)-C(4B)-C(13B)-C(18B)	-59.6(4)
C(5B)-C(4B)-C(13B)-C(18B)	59.8(3)

C(18B)-C(13B)-C(14B)-C(15B)	0.5(5)
C(4B)-C(13B)-C(14B)-C(15B)	-176.8(3)
C(13B)-C(14B)-C(15B)-C(16B)	0.7(5)
C(14B)-C(15B)-C(16B)-C(17B)	-1.5(5)
C(15B)-C(16B)-C(17B)-C(18B)	1.1(5)
C(16B)-C(17B)-C(18B)-C(13B)	0.2(5)
C(14B)-C(13B)-C(18B)-C(17B)	-1.0(5)
C(4B)-C(13B)-C(18B)-C(17B)	176.4(3)
C(2B)-C(1B)-O(1B)-C(8B)	-59.4(3)
C(9B)-C(8B)-O(1B)-C(1B)	175.9(2)
C(10B)-C(8B)-O(1B)-C(1B)	-67.0(3)
C(7B)-C(8B)-O(1B)-C(1B)	58.2(3)
O(3B)-C(11B)-O(4B)-C(12B)	-0.4(4)
C(6B)-C(11B)-O(4B)-C(12B)	-179.2(2)
O(1C)-C(1C)-C(2C)-C(3C)	-54.3(3)
C(1C)-C(2C)-C(3C)-C(7C)	52.9(3)
C(1C)-C(2C)-C(3C)-C(4C)	170.3(2)
C(2C)-C(3C)-C(4C)-C(13C)	83.8(3)
C(7C)-C(3C)-C(4C)-C(13C)	-155.9(2)
C(2C)-C(3C)-C(4C)-C(5C)	-150.7(2)
C(7C)-C(3C)-C(4C)-C(5C)	-30.4(3)
C(13C)-C(4C)-C(5C)-C(6C)	131.9(2)
C(3C)-C(4C)-C(5C)-C(6C)	6.4(3)

C(4C)-C(5C)-C(6C)-O(2C)	-96.4(3)
C(4C)-C(5C)-C(6C)-C(11C)	142.4(2)
C(4C)-C(5C)-C(6C)-C(7C)	19.7(3)
C(2C)-C(3C)-C(7C)-C(6C)	168.81(19)
C(4C)-C(3C)-C(7C)-C(6C)	43.4(2)
C(2C)-C(3C)-C(7C)-C(8C)	-57.7(3)
C(4C)-C(3C)-C(7C)-C(8C)	176.9(2)
O(2C)-C(6C)-C(7C)-C(3C)	79.1(2)
C(11C)-C(6C)-C(7C)-C(3C)	-158.11(19)
C(5C)-C(6C)-C(7C)-C(3C)	-38.6(2)
O(2C)-C(6C)-C(7C)-C(8C)	-48.8(3)
C(11C)-C(6C)-C(7C)-C(8C)	74.0(3)
C(5C)-C(6C)-C(7C)-C(8C)	-166.5(2)
C(3C)-C(7C)-C(8C)-O(1C)	57.8(3)
C(6C)-C(7C)-C(8C)-O(1C)	-178.60(19)
C(3C)-C(7C)-C(8C)-C(9C)	169.8(2)
C(6C)-C(7C)-C(8C)-C(9C)	-66.7(3)
C(3C)-C(7C)-C(8C)-C(10C)	-63.5(3)
C(6C)-C(7C)-C(8C)-C(10C)	60.1(3)
O(2C)-C(6C)-C(11C)-O(3C)	-9.7(4)
C(7C)-C(6C)-C(11C)-O(3C)	-132.0(3)
C(5C)-C(6C)-C(11C)-O(3C)	112.4(3)
O(2C)-C(6C)-C(11C)-O(4C)	172.3(2)

C(7C)-C(6C)-C(11C)-O(4C)	49.9(3)
C(5C)-C(6C)-C(11C)-O(4C)	-65.6(3)
C(3C)-C(4C)-C(13C)-C(14C)	-121.1(3)
C(5C)-C(4C)-C(13C)-C(14C)	118.5(3)
C(3C)-C(4C)-C(13C)-C(18C)	58.5(4)
C(5C)-C(4C)-C(13C)-C(18C)	-61.9(3)
C(18C)-C(13C)-C(14C)-C(15C)	-0.1(5)
C(4C)-C(13C)-C(14C)-C(15C)	179.6(3)
C(13C)-C(14C)-C(15C)-C(16C)	-1.0(5)
C(14C)-C(15C)-C(16C)-C(17C)	1.4(5)
C(15C)-C(16C)-C(17C)-C(18C)	-0.7(5)
C(16C)-C(17C)-C(18C)-C(13C)	-0.4(5)
C(14C)-C(13C)-C(18C)-C(17C)	0.8(5)
C(4C)-C(13C)-C(18C)-C(17C)	-178.9(3)
C(2C)-C(1C)-O(1C)-C(8C)	60.6(3)
C(9C)-C(8C)-O(1C)-C(1C)	-176.5(2)
C(10C)-C(8C)-O(1C)-C(1C)	66.2(3)
C(7C)-C(8C)-O(1C)-C(1C)	-59.6(3)
O(3C)-C(11C)-O(4C)-C(12C)	-0.9(4)
C(6C)-C(11C)-O(4C)-C(12C)	177.2(3)

Symmetry transformations used to generate equivalent atoms:

Table 7. Hydrogen bonds for **12** [Å and °].

D-H...A	d(D-H)	d(H...A)	d(D...A)	<(DHA)
O(2)-H(2C)...O(1B)#1	0.84	1.98	2.817(3)	175.7
O(2B)-H(2BC)...O(1)	0.84	1.99	2.776(3)	154.5
O(2C)-H(2CC)...O(1C)#2	0.84	1.98	2.771(3)	157.2

Symmetry transformations used to generate equivalent atoms:

#1 x+1,y,z+1 #2 x-1/2,-y+1/2,z-1/2