Electronic Supplementary Information (ESI)

Enantioselective Total Synthesis of Decytospolide A and Decytospolide B Using an Achmatowicz Reaction

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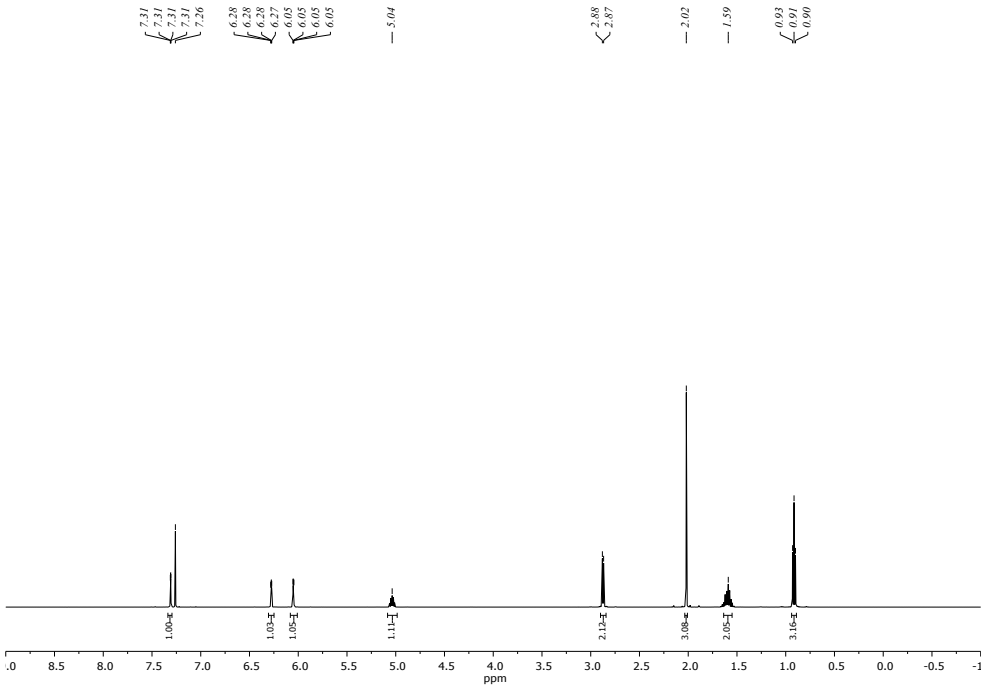
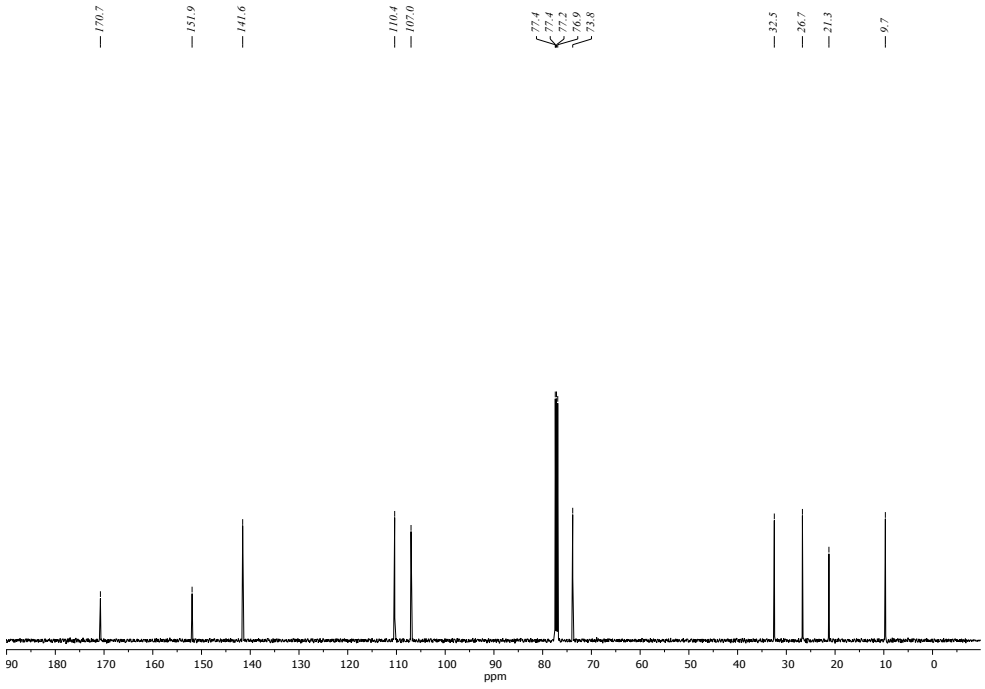
General experimental conditions

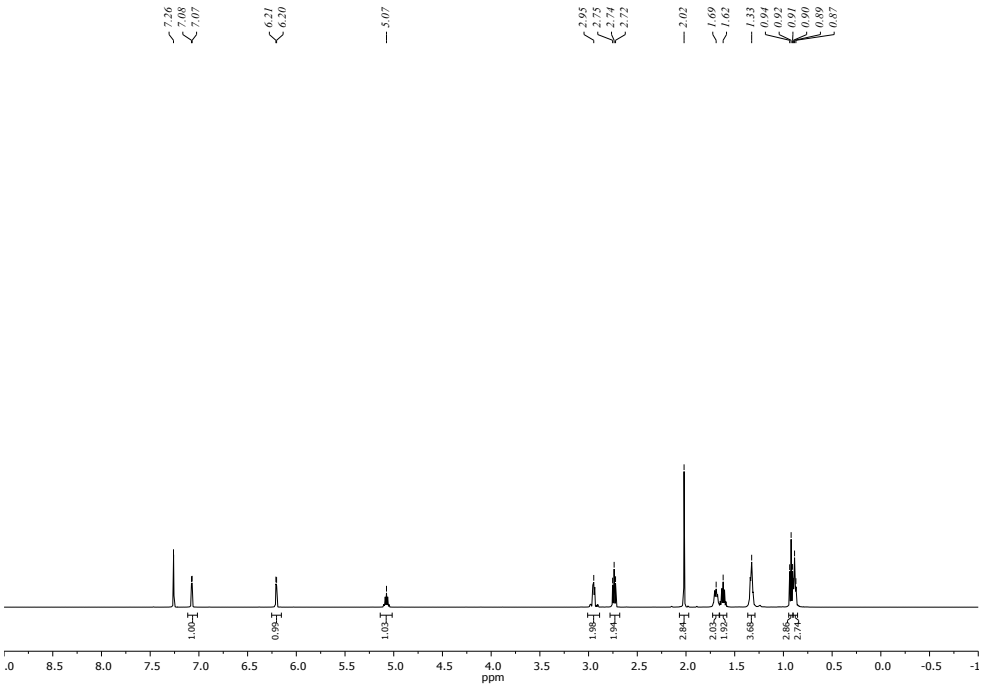
Chemicals and reagents were purchased from commercial suppliers and used without further purification. Anhydrous solvents were obtained as follows: dichloromethane and toluene from calcium hydride, diethyl ether and tetrahydrofuran from sodium/benzophenone. All other solvents were reagent grade. All moisture-sensitive reactions were either carried out in flame- or oven-dried (120 °C) glassware under an argon atmosphere. TLC analysis was conducted using glass-backed thin-layer silica gel chromatography plates (60 Å, 250 μm thickness, F254 indicator). Column chromatography was performed using silica gel, 230-400 mesh, 60 Å pore diameter. ^{1}H and ^{13}C NMR spectra were recorded on either Bruker ARX400, Bruker DRX-500, Bruker AV500HD. Chemical shift (δ values) are reported in parts per million and are referenced to the residual solvent signal ($CDCl_3$ ^{1}H singlet = 7.26, ^{13}C triplet = 77.16). Characteristic splitting patterns due to spin-spin coupling are identified as follows: s = singlet, d = doublet, t = triplet, q = quartet, quint = quintet, sep = septet, m = multiplet, dd = doublet of doublets, ddd = doublet of doublet of doublets, td = triplet of doublets, dq = doublet of quartets, brs = broad singlet, app = apparent. All coupling constants are measured in hertz (Hz). Optical rotations were recorded by a Perkin Elmer 341 polarimeter. LRMS and HRMS spectra were recorded at the Purdue University Department of Chemistry Mass Spectrometry Center. HPLC data was obtained on an Agilent 1290 Infinity II.



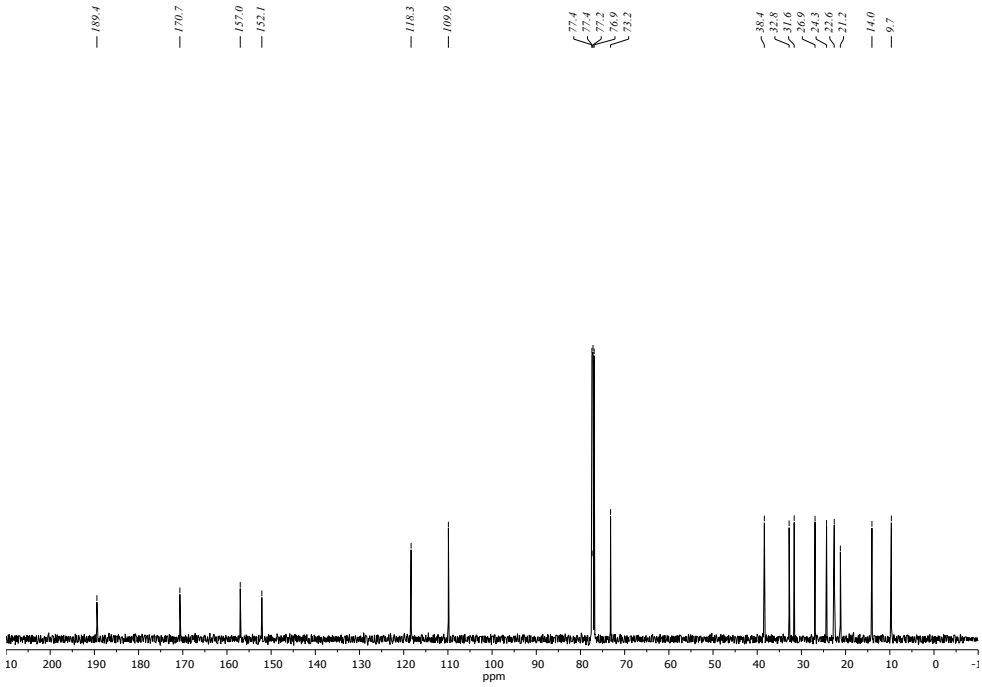
^{1}H NMR (500 MHz, $CDCl_3$) of Acetate **12**



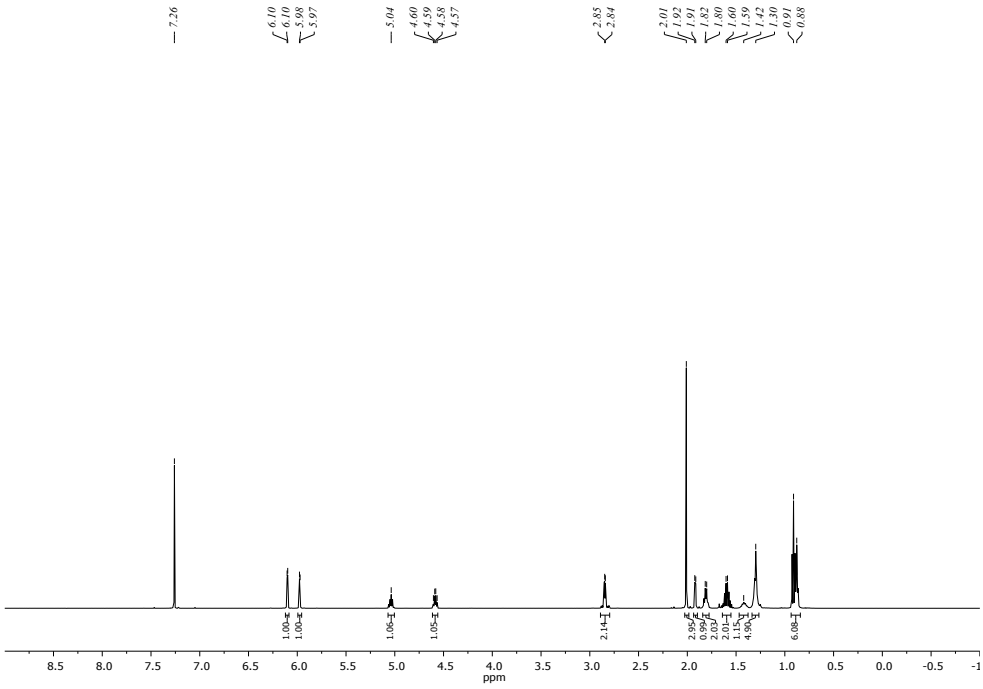
^{13}C NMR (125 MHz, $CDCl_3$) of Acetate **12**



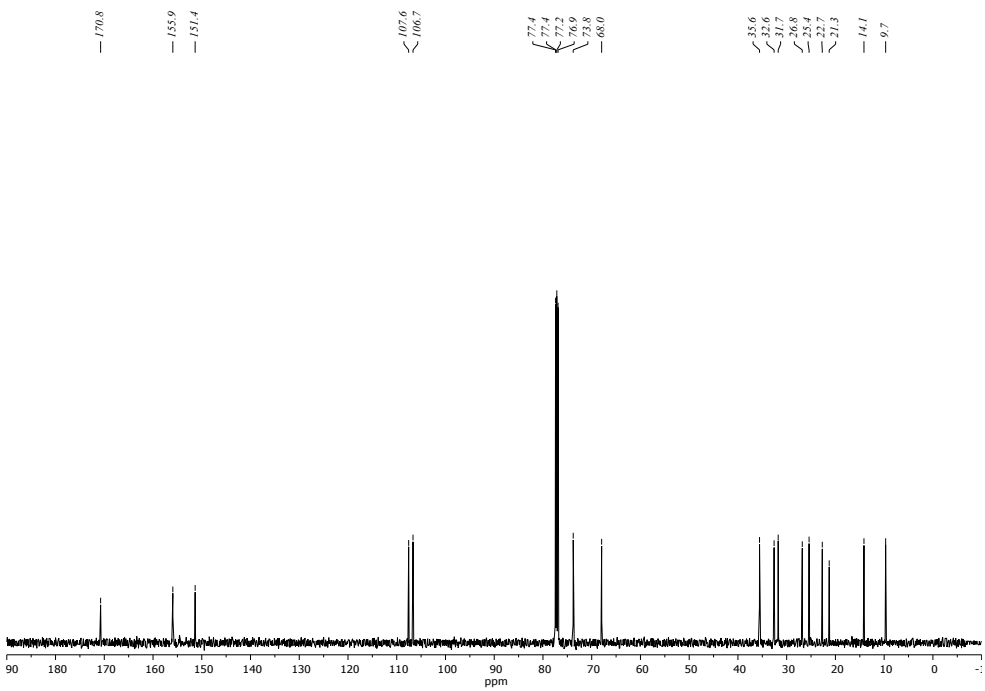
^{1}H NMR (500 MHz, $CDCl_3$) of Furanyl ketone **13**



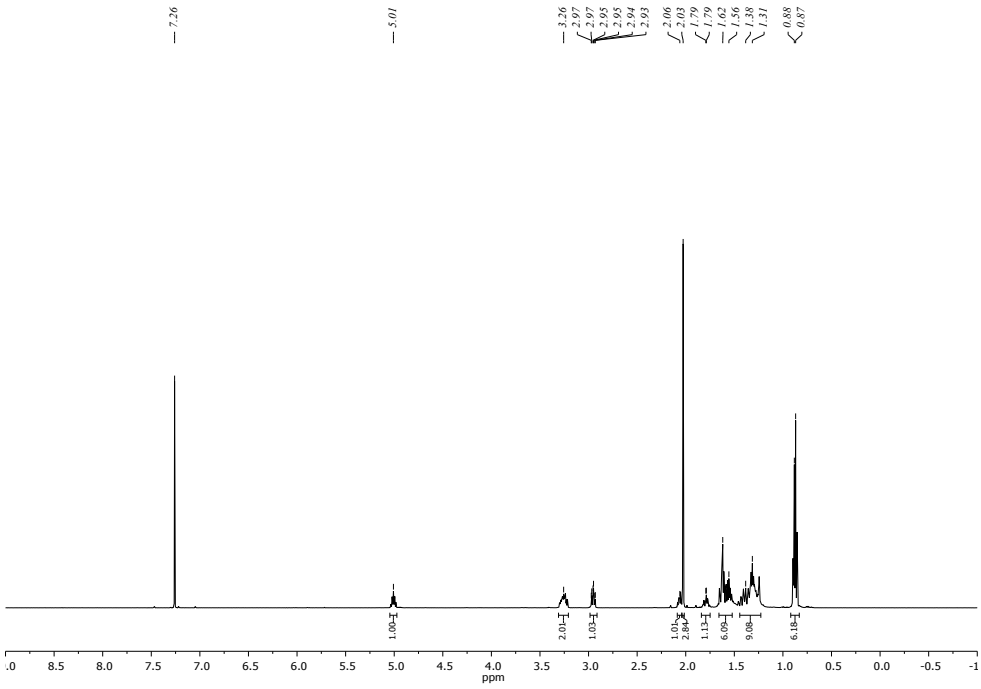
^{13}C NMR (125 MHz, $CDCl_3$) of Furanyl ketone **13**



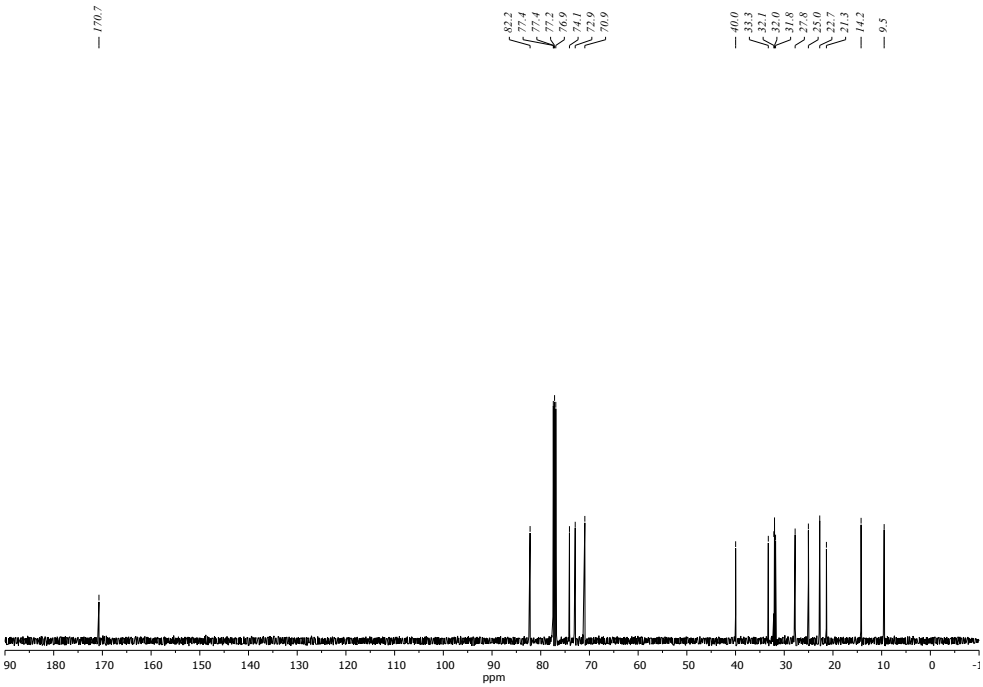
^{1}H NMR (500 MHz, $CDCl_3$) of Alcohol **10**



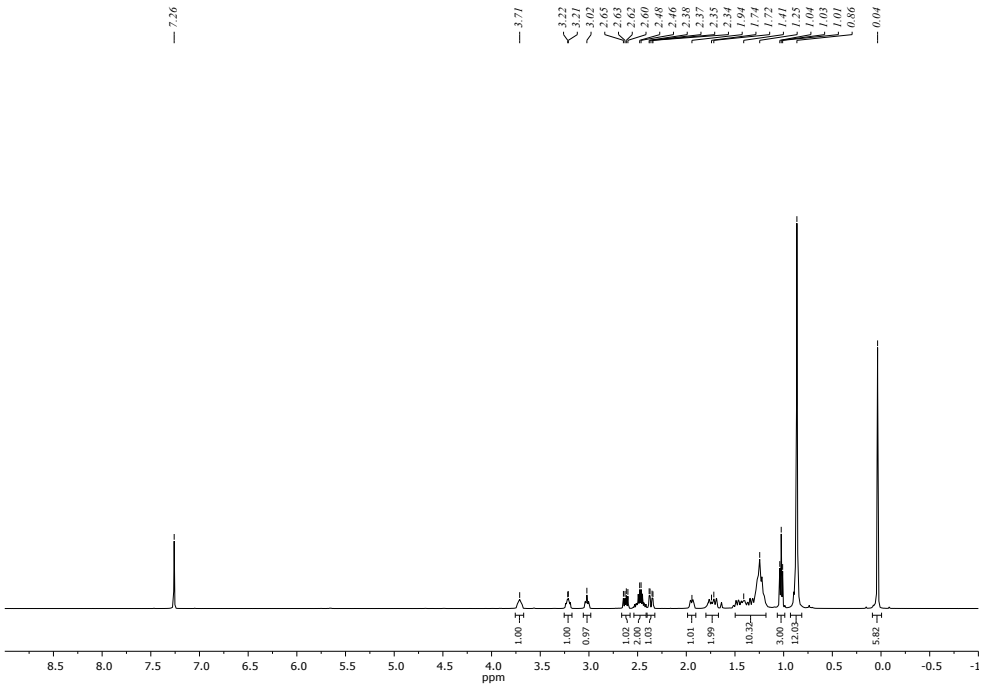
^{13}C NMR (125 MHz, $CDCl_3$) of Alcohol **10**



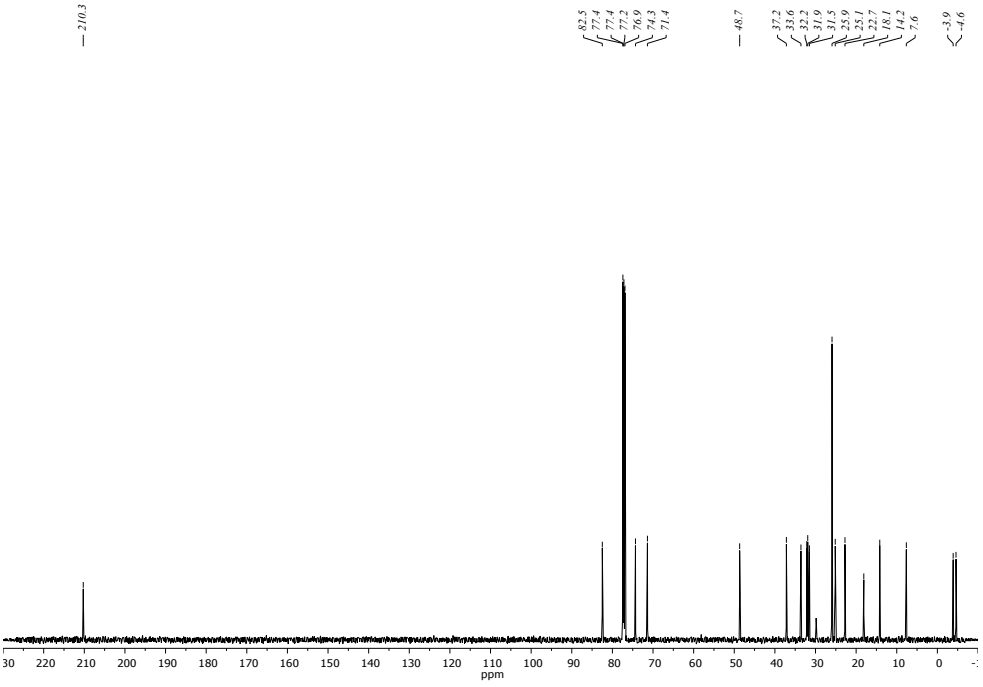
^{1}H NMR (500 MHz, $CDCl_3$) of tetrahydrpyran **8**



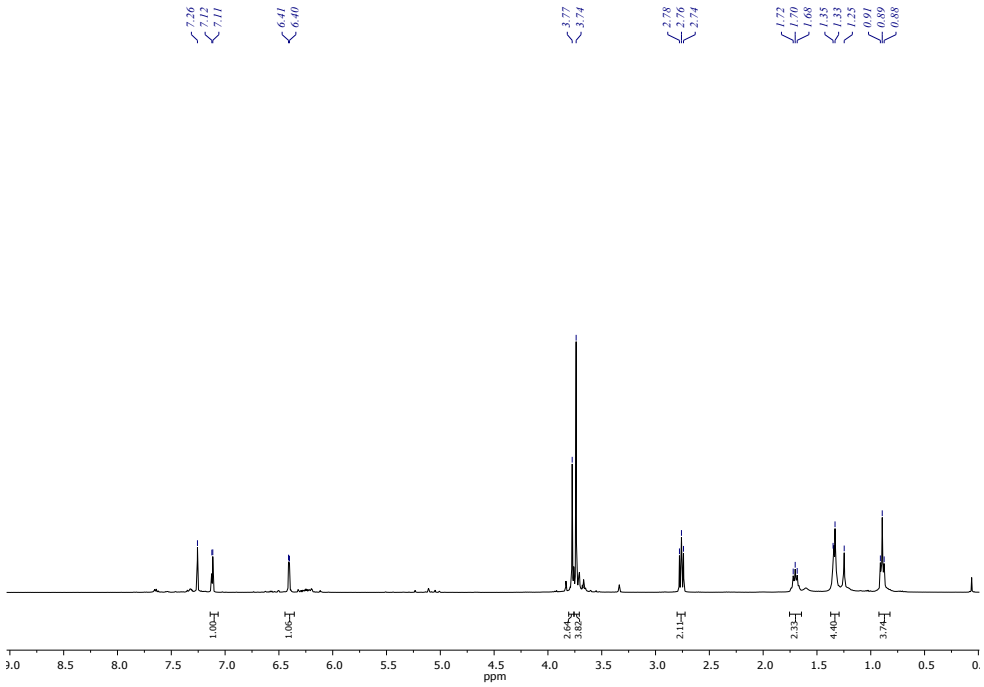
^{13}C NMR (125 MHz, $CDCl_3$) of tetrahydropyran **8**



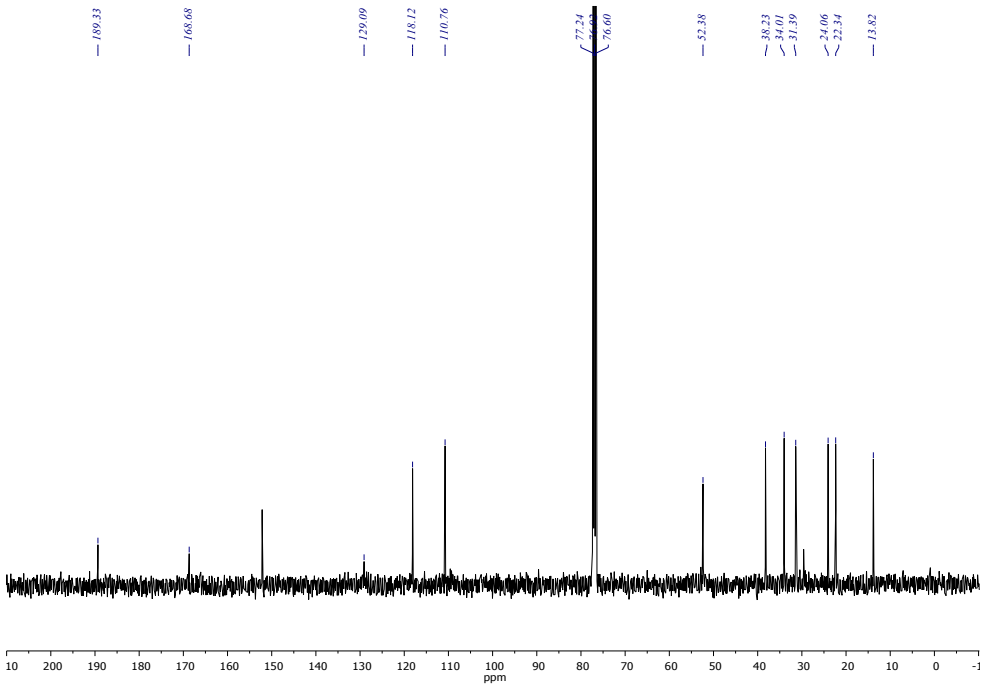
^{1}H NMR (500 MHz, $CDCl_3$) of Ketone **15**



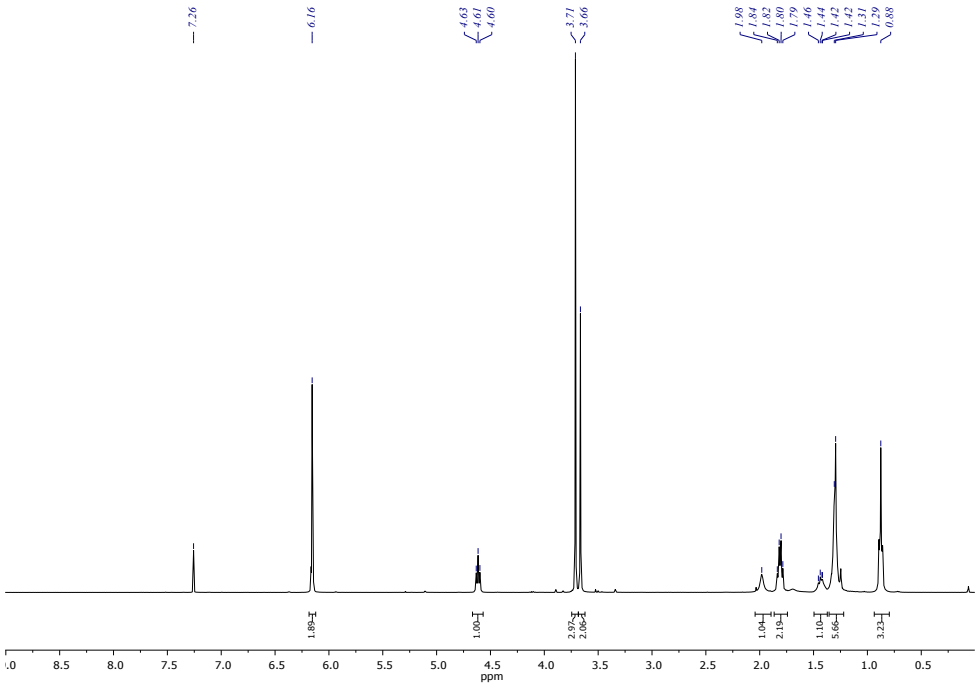
^{13}C NMR (125 MHz, $CDCl_3$) of Ketone **15**



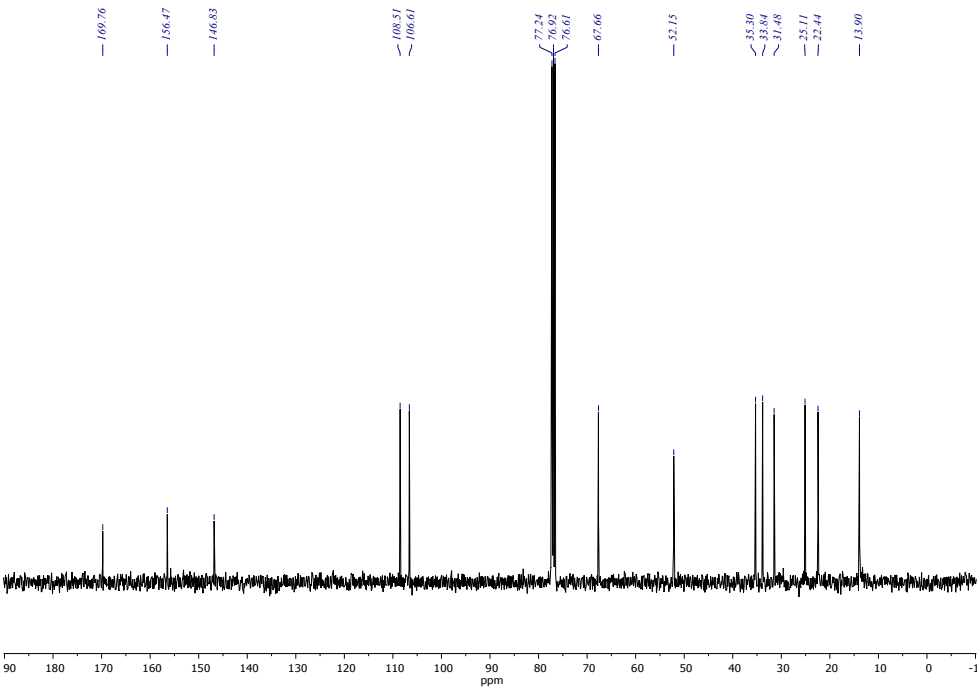
^{1}H NMR (400 MHz, $CDCl_3$) of Ketone **18**



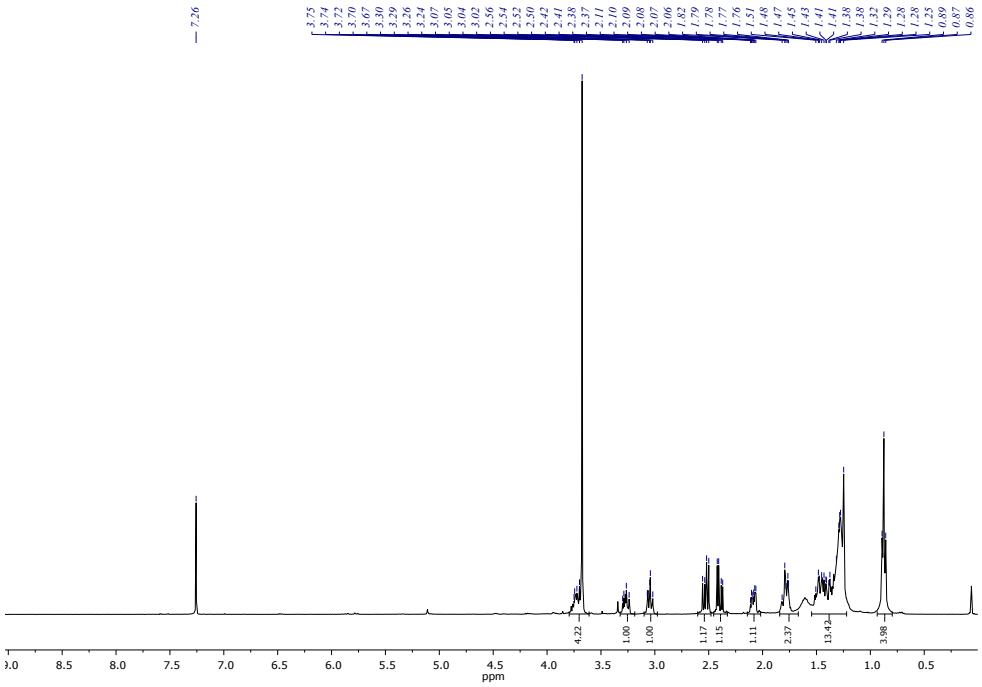
^{13}C NMR (100 MHz, $CDCl_3$) of Ketone **18**



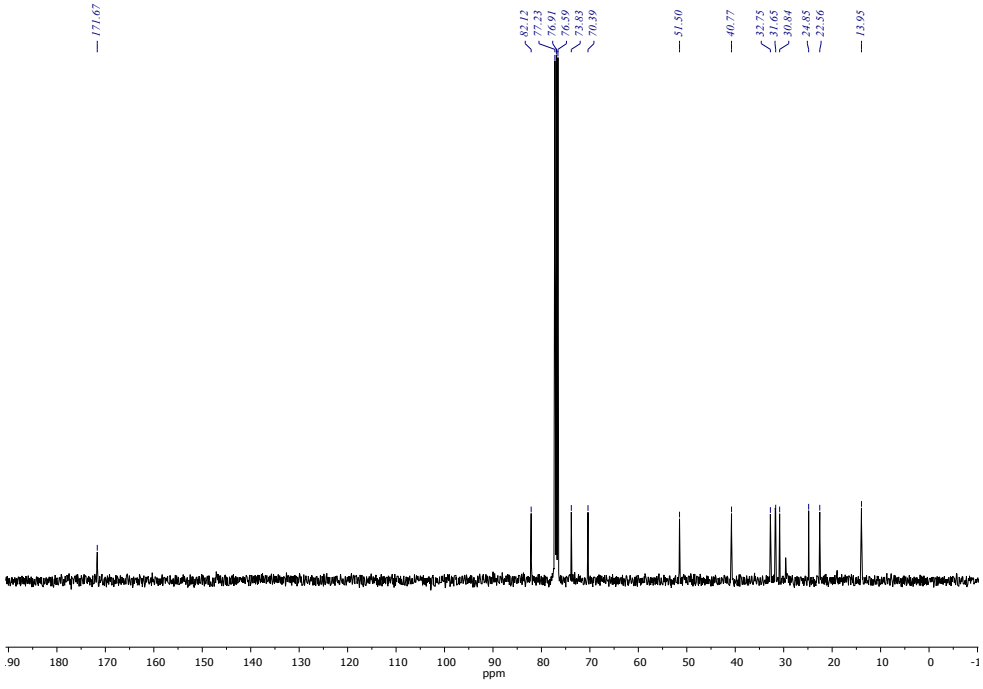
^{1}H NMR (400 MHz, $CDCl_3$) of Alcohol **19**



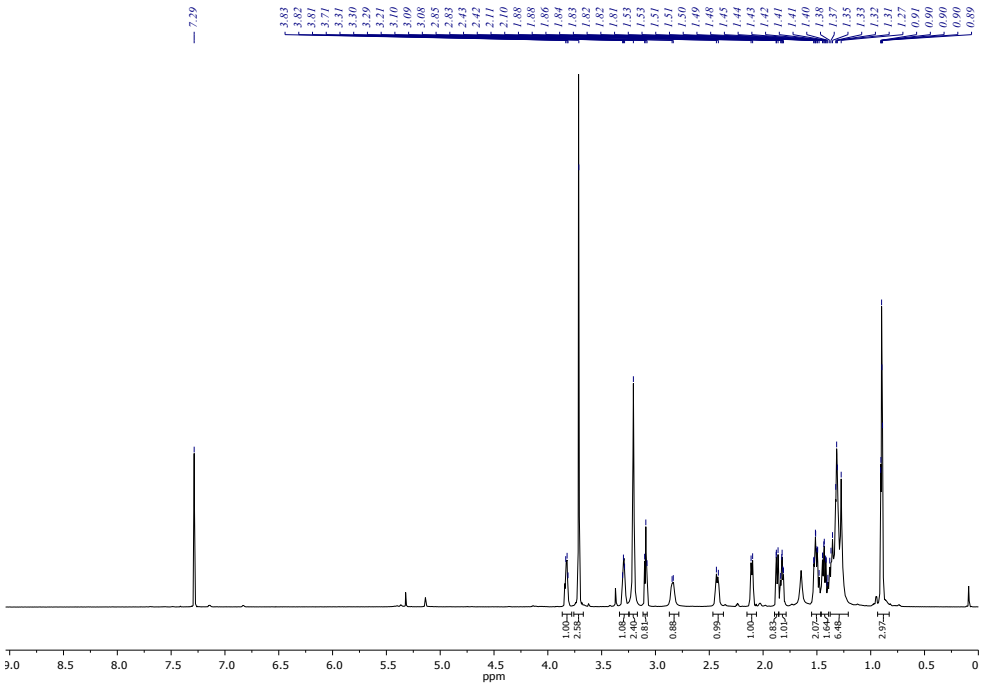
^{13}C NMR (100 MHz, $CDCl_3$) of Alcohol **19**



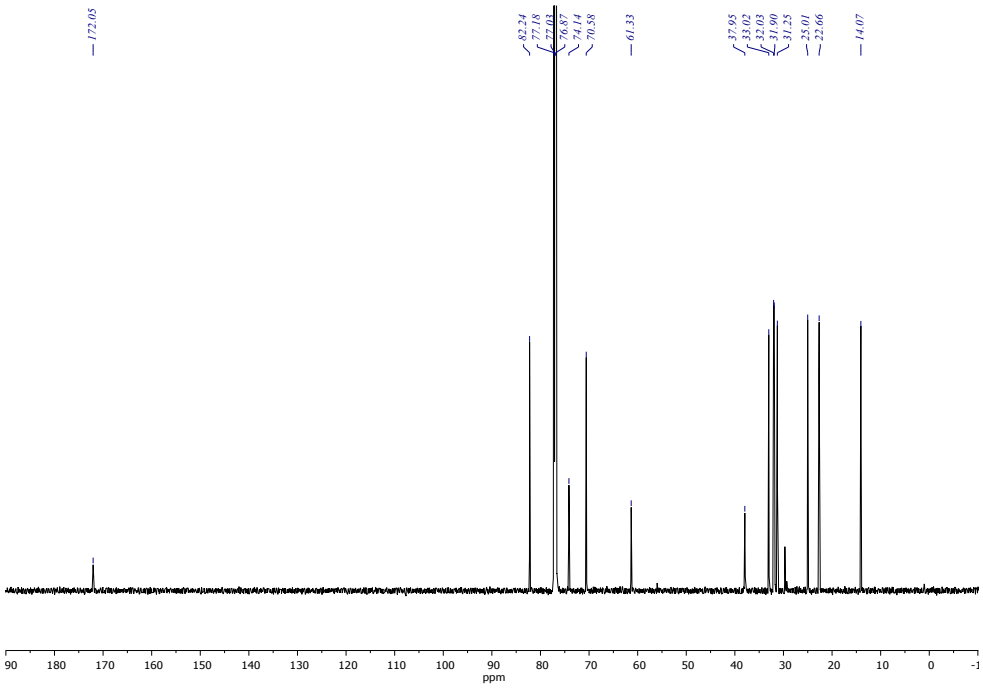
^{1}H NMR (400 MHz, $CDCl_3$) of Alcohol **20**



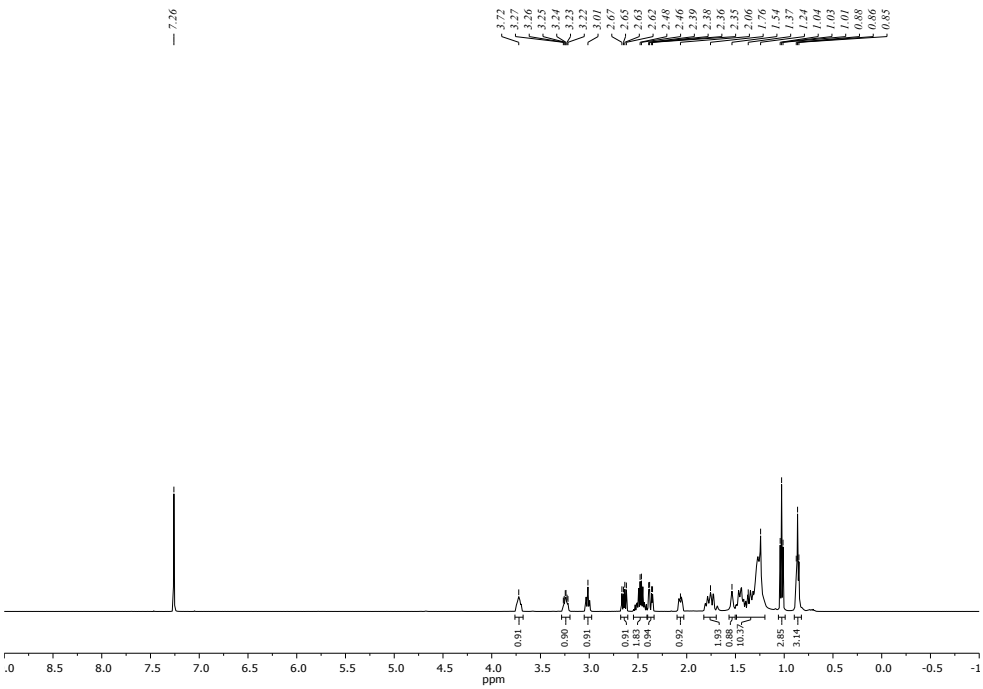
^{13}C NMR (100 MHz, $CDCl_3$) of Alcohol **20**



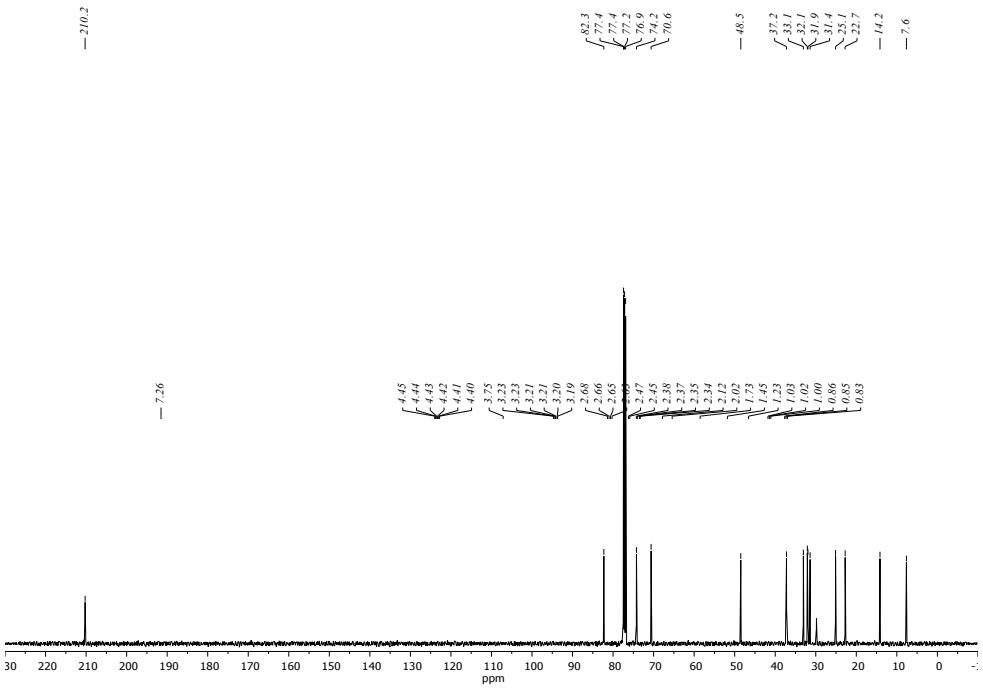
^{1}H NMR (800 MHz, $CDCl_3$) of Amide **21**



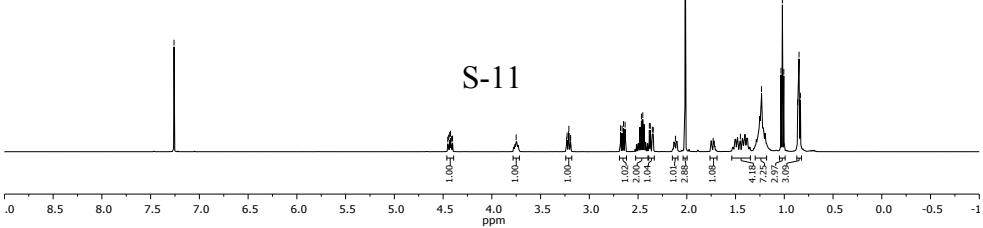
^{13}C NMR (200 MHz, $CDCl_3$) of Amide **21**



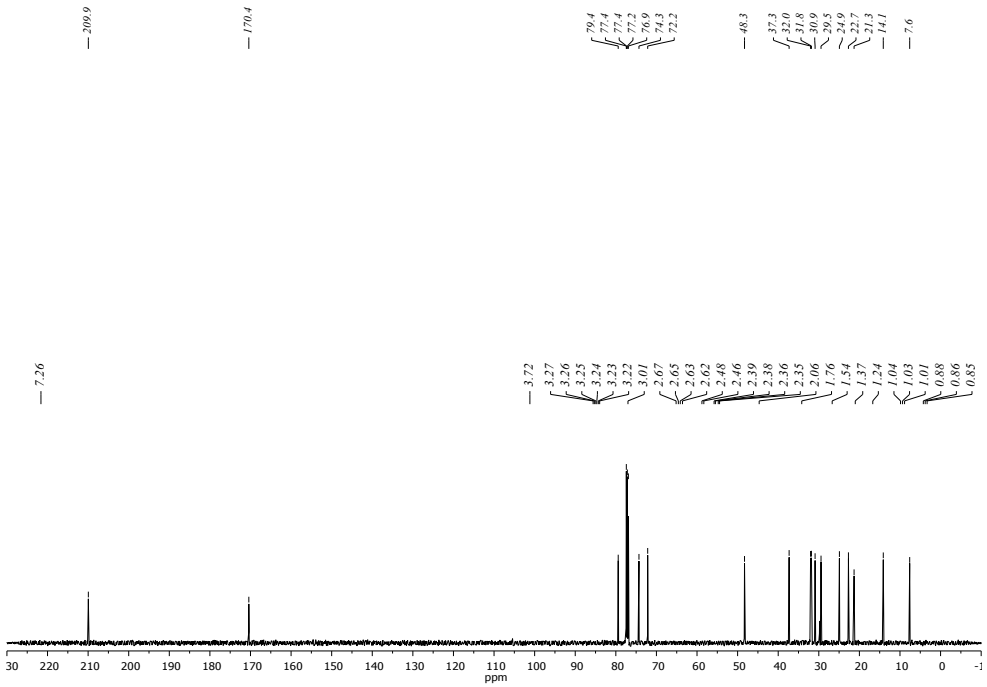
^{1}H NMR (500 MHz, $CDCl_3$) of Decytospolide A, **6**



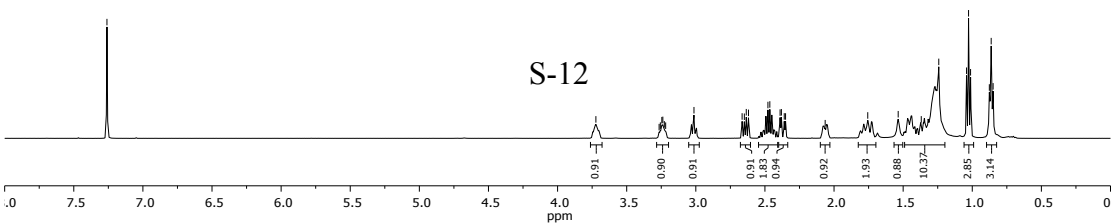
^{13}C NMR (125 MHz, $CDCl_3$) of Decytospolide A, **6**



^{1}H NMR (500 MHz, $CDCl_3$) of Decytospolide B, **7**



^{13}C NMR (125 MHz, $CDCl_3$) of Decytospolide B, **7**



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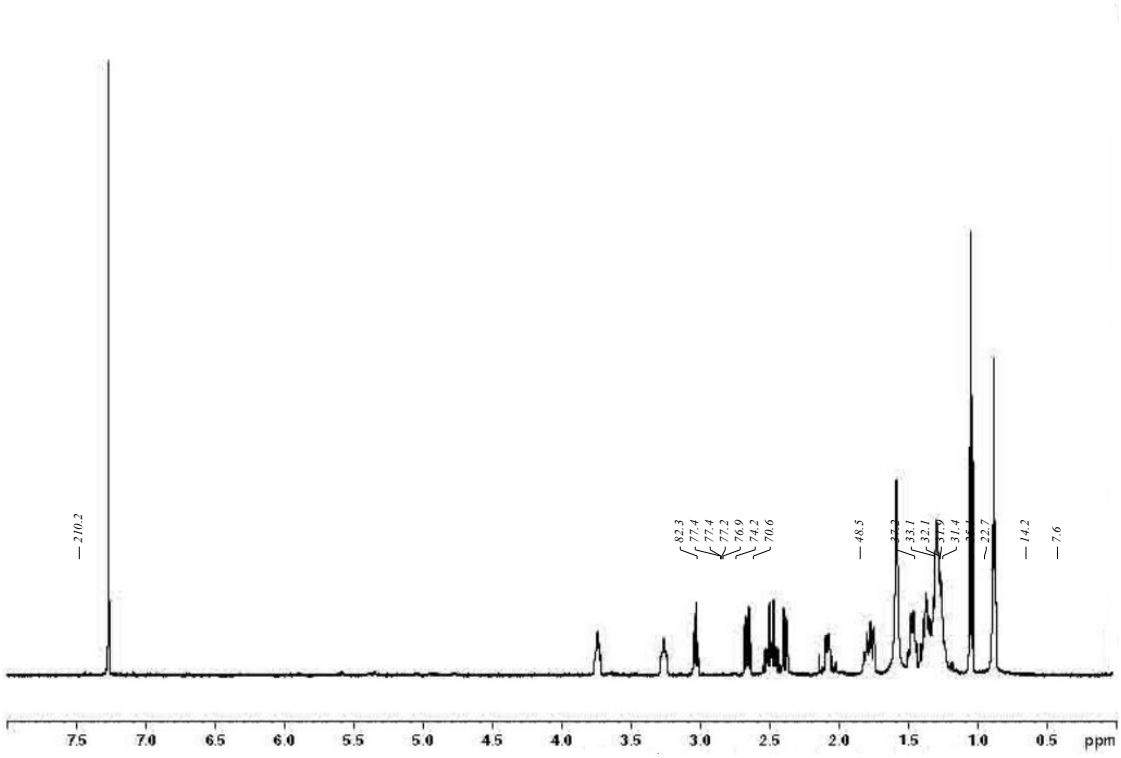
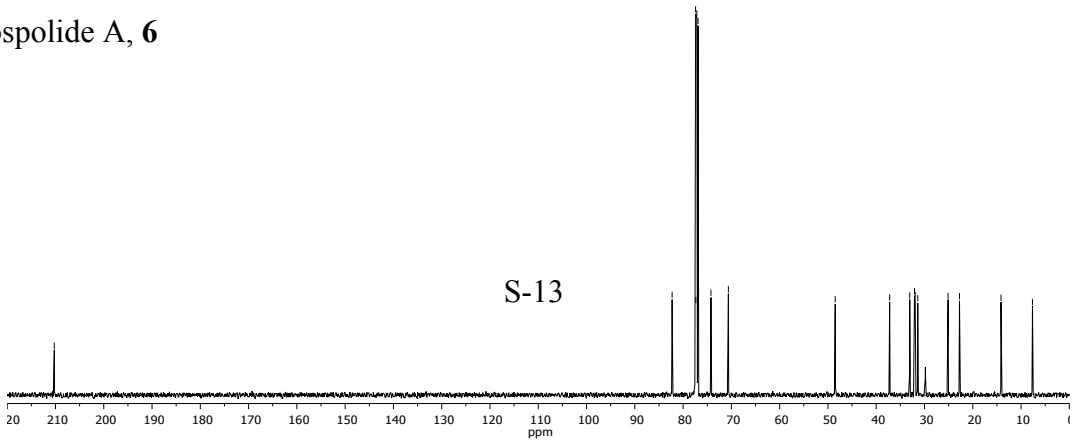
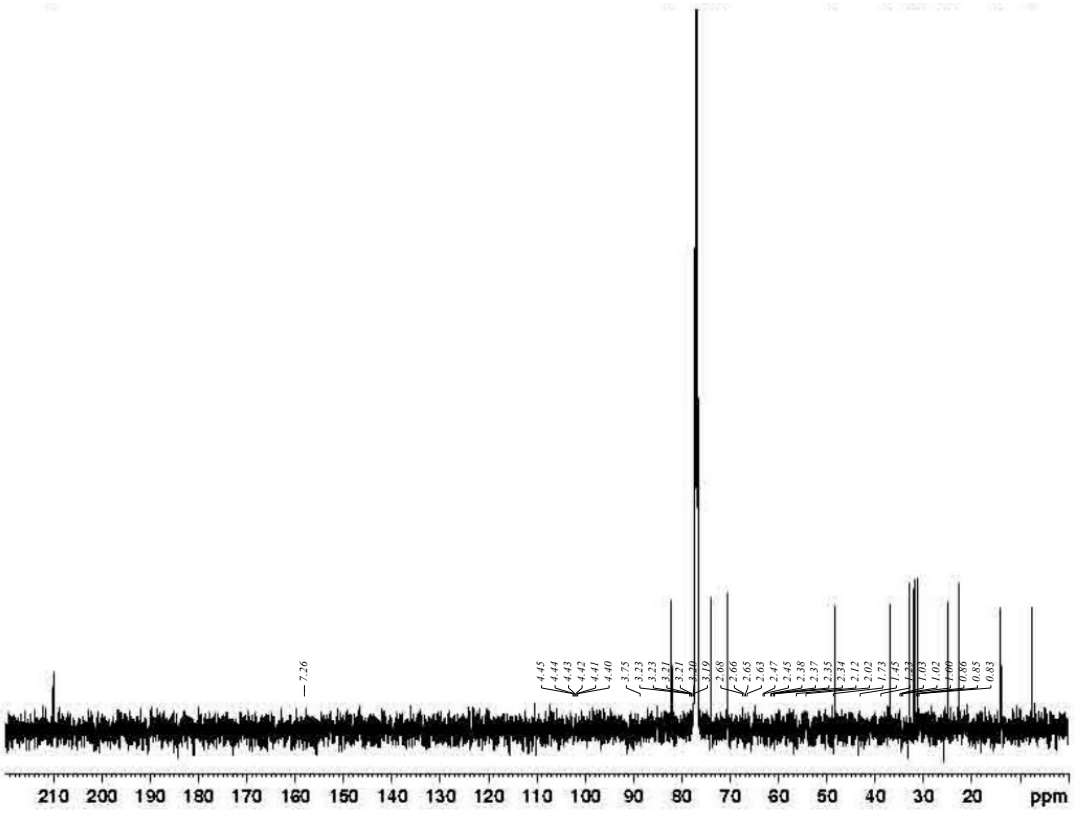
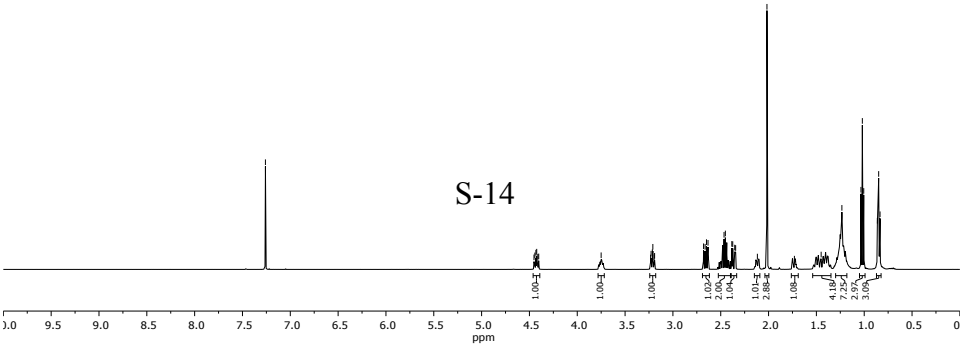


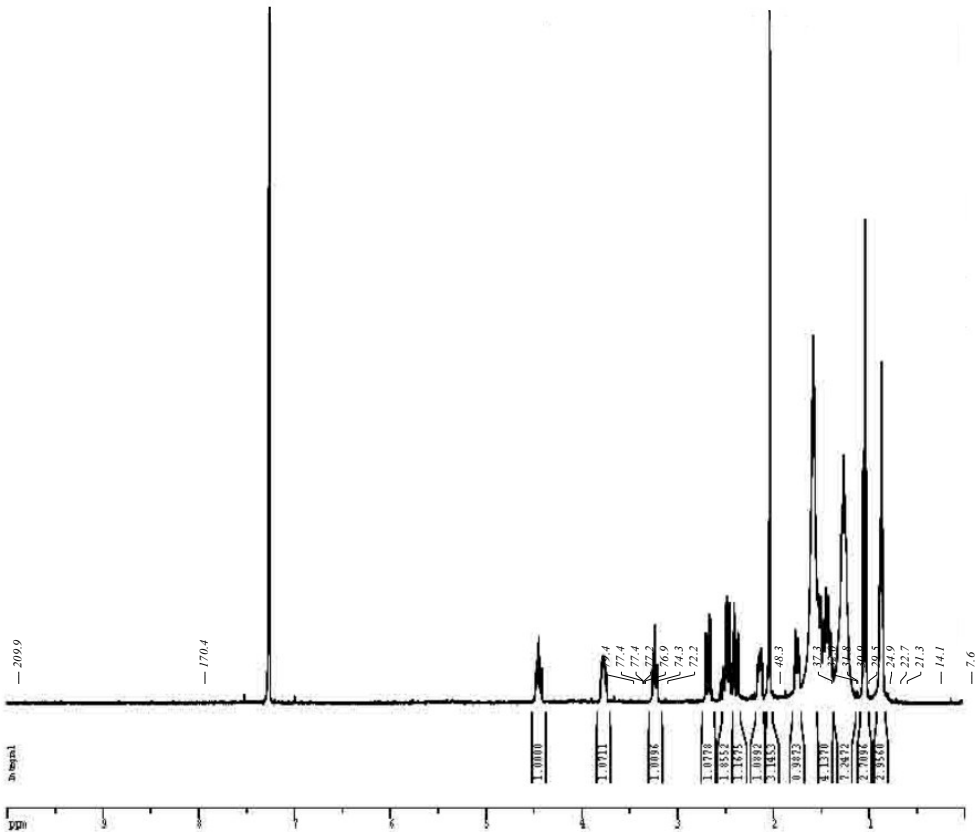
NMR (500 MHz, $CDCl_3$) of Synthetic (top) and Natural (bottom) Decytospolide A, **6**



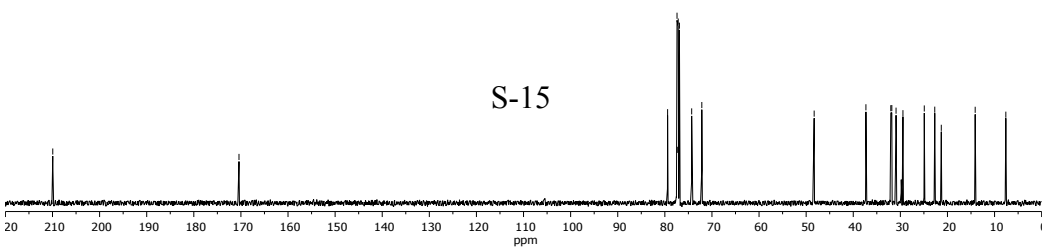


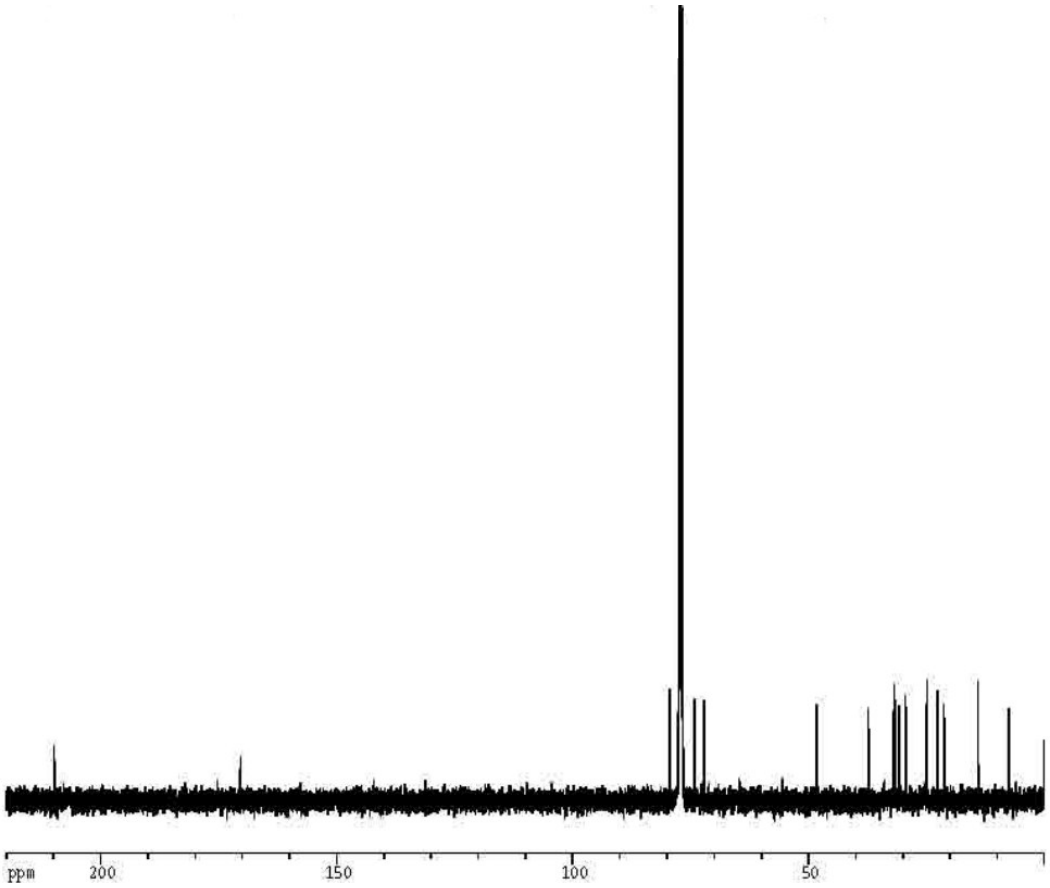
^{13}C NMR (125 MHz, $CDCl_3$) of Synthetic (top) and Natural (bottom) Decytospolide A, **6**





^{1}H NMR (500 MHz, $CDCl_3$) of Synthetic (top) and Natural (bottom) Decytospolide B, **7**





^{13}C NMR (125 MHz, $CDCl_3$) of Synthetic (top) and Natural (bottom) Decytospolide B, **7**