

Supporting Information

Development of a Mild and Efficient Protocol for the Protection and *O*-Alkylation of Tertiary Allyl Alcohol of Oxindole with Potassium Carbonate under Microwave Irradiation

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General.....	S1
Typical experimental procedure.....	S2
Characterization of compounds.....	S3
Copies of ¹ H and ¹³ C NMR spectra.....	S11

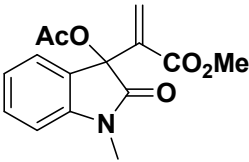
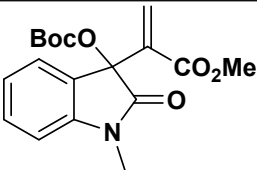
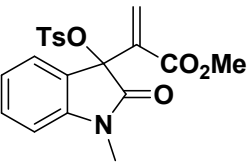
General Remark: All the reactions were carried out in oven-dried glassware. Progress of reactions was monitored by Thin Layer Chromatography (TLC) while purification of crude compounds was done by column chromatography using silica gel (100-200 mesh). Melting points were recorded on a Buchi melting point apparatus and are uncorrected. NMR spectra were recorded at 500 and 300 MHz (based on availability of instruments) 125 and 75 MHz (for ¹³C) respectively on Bruker Avance DPX-500 MHz. and Bruker Avance DPX-300 MHz. Chemical shifts are reported in δ (ppm) relative to TMS (¹H) or CDCl₃ (¹³C) as internal standards. Mass spectra were recorded using JEOL JMS 600H mass spectrometer. IR spectra were recorded on Bomem MB series FT-IR spectrometer, absorbencies are reported in cm⁻¹. Yields refer to quantities obtained after chromatography.

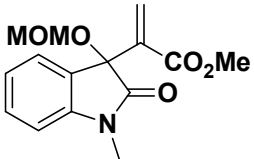
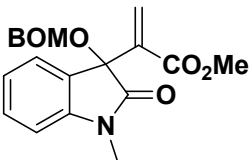
Typical Experimental Procedure: The MBH adduct **1a** (100 mg, 0.404 mmol), potassium carbonate (100 % w/w) and protecting reagents or alkylation reagents (1.5 equiv.) with tetra-butyl ammonium bromide (1.5 equiv.) were made a slurry in 50 ml round bottom flask and subjected to microwave oven of Start S (Milestone-Italy) which is equipped with a magnetic stirrer, a noncontact infrared continuous feedback temperature system under atmospheric pressure for a period of 10-15 minutes. After cooling the reaction mixture to room temperature, the crude mixture was dissolved in EtOAc and

Supporting Information

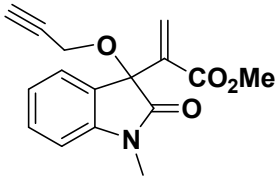
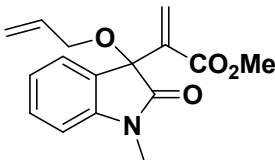
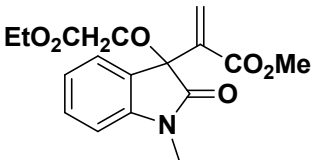
filtered through celite pad then purified by a silica gel column chromatography and afforded the corresponding products in good to moderate yield (45-98%).

Characterization of new compounds:

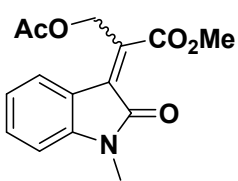
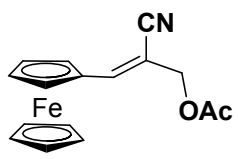
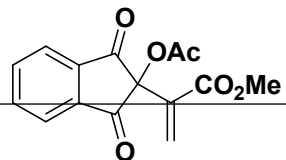
 Compound 3a	IR (CH₂Cl₂) $\gamma_{\max}$: 1130, 1361, 1476, 1492, 1619, 1721, 1732, 2126, 2931, 3218 cm⁻¹; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 2.03(s, 3H), 3.27(s, 3H), 3.77(s, 3H), 6.50(s, 1H), 6.57(s, 1H), 6.91(d, J=7Hz, 1H), 7.20(s, 1H), 7.60(s, 1H), 7.79(d, J=7Hz, 1H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 20.52, 26.99, 54.87, 78.29, 108.29, 115.52, 122.80, 127.61, 129.06, 131.61, 135.47, 135.87, 165.68, 169.87, 173.01; FAB mass: Calcd. For C₁₅H₁₅NO₅ is 289.10; Found : 290.47(M+1).
 Compound 3b	IR (CH₂Cl₂) $\gamma_{\max}$: 1131, 1360, 1472, 1617, 1719, 1731, 2123, 2930, 3215 cm⁻¹; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 1.85-1.89(bs, 9H), 3.28(s, 1H), 3.59(s, 3H), 6.88(s, 1H), 6.89(s, 1H), 7.14-7.15(d, J=6.5Hz, 1H), 7.32-7.34(t, J= 6.5Hz, 1H), 7.40(s, 1H), 7.54(s, 1H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 25.56, 25.60, 26.04, 27.29, 51.85, 76.56, 81.34, 113.26, 133.56, 121.34, 121.97, 131.90, 138.75, 139.29, 144.21, 157.29, 164.03; FAB mass: Calcd. For C₁₅H₁₅NO₅ is 347.14; Found : 348.12(M+1).
 (Data has been presented only)	IR (CH₂Cl₂) $\gamma_{\max}$: 1132, 1223, 1353, 1478, 1490, 1618, 1723, 1735, 2124, 2926, 3212 cm⁻¹; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 2.42(s, 3H), 3.17(s, 3H), 3.94(s, 3H), 6.22(s, 2H), 6.78(s, 1H), 6.99(s, 1H), 7.30-7.37(m, 4H), 7.81-7.83(d, J = 7.5Hz,

protected isomer 3c) Compound 3c	2H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 26.25, 33.14, 55.38, 79.05, 113.05, 113.20, 114.63(2C), 119.20, 122.48, 122.69, 124.21, 129.51, 129.75(2C), 132.18, 132.39, 146.81, 165.74, 166.25; FAB mass: Calcd. For C₂₀H₁₉NO₆S is 401.09; Found : 401.34(M+).
 Compound 3d	IR (CH₂Cl₂) γ_{max}: 1128, 1222, 1475, 1492, 1616, 1725, 1728, 2116, 2925, cm⁻¹; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 2.49 (s, 3H), 3.22(s, 3H), 3.99(s, 3H), 5.32(s, 2H), 6.79-6.80(d, <i>J</i> = 8Hz, 1H), 6.98-6.702(t, <i>J</i> = 7.5Hz, 1H), 7.32-7.35(t, <i>J</i> = 7.5Hz, 1H), 7.43-7.44(d, <i>J</i> = 8Hz, 1H), 7.52-7.54(m, 1H), 7.69-7.71(m, 1H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 25.99, 31.52, 39.37, 52.48, 73.33, 113.76, 125.59, 127.01, 128.18, 129.42, 130.07, 131.52, 148.48, 160.22, 168.74; FAB mass: Calcd. For C₁₅H₁₇NO₅ is 291.11; Found : 292.21(M+1).
 Compound 3e	IR (CH₂Cl₂) γ_{max}: 1135, 1213, 1334, 1465, 1470, 1618, 1724, 1729, 2116, 2915, cm⁻¹; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 3.30(s, 3H), 3.57(s, 3H), 4.12-4.16(dd, <i>J</i> = 11 Hz, 2H), 4.52-4.56(dd, <i>J</i> = 11 Hz, 2H), 6.43(s, 1H), 6.55(s, 1H), 6.86-6.87(d, <i>J</i> = 8 Hz, 1H), 6.98-6.99(t, <i>J</i> = 8Hz, 1H), 7.12-7.14(t, <i>J</i> = 8Hz, 1H), 7.30-7.34(m, 6H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 26.86, 52.19, 66.60, 66.91, 73.41, 79.83, 108.69, 122.83, 123.39, 125.94, 128.16, 128.23, 132.15, 136.25, 136.67(4C), 136.80, 145.60, 163.99, 167.39, 172.63; FAB mass: Calcd. For C₂₁H₂₁NO₅ is 367.14; Found:

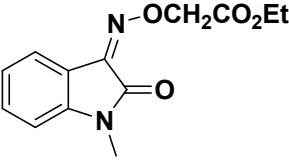
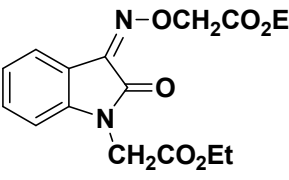
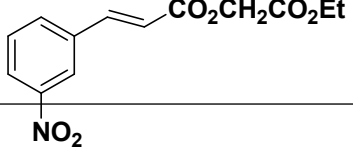
Supporting Information

	368.29(M+1).
 Compound 3f	IR (CH₂Cl₂) $\gamma_{\max}$: 1134, 1233, 1354, 1475, 1470, 1613, 1724, 1735, 2122, 2928, 3210 cm⁻¹; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 2.35(s, 1H), 3.27(s, 3H), 3.57(s, 3H), 3.88-4.03(m, 2H), 6.62(s, 2H), 6.85-6.87(d, J = 7.5Hz, 1H), 7.04-7.06(t, J = 7.0Hz, 1H), 7.12-7.14(d, J = 7.5Hz, 1H), 7.34-7.34(t, J = 7.0Hz, 1H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 26.46, 51.95, 52.91, 74.62, 79.10, 80.41, 108.47, 122.89, 124.55, 125.96, 128.51, 130.75, 137.42, 145.34, 164.57, 173.43; FAB mass: Calcd. For C₁₆H₁₅NO₄ is 285.12; Found: 286.32(M+1).
 Compound 3g	IR (CH₂Cl₂) $\gamma_{\max}$: 1130, 1343, 1481, 1476, 1634, 1725, 1781, 2938 cm⁻¹; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 3.27(s, 3H), 3.56(s, 3H), 3.61-3.64(m, 1H), 3.78-3.80(m, 1H), 5.08–5.10(d, J = 10.5Hz, 1H), 5.80-5.84(m, 1H), 6.59(s, 1H), 6.61(s, 1H), 6.85-6.86(d, J = 7.5Hz, 1H), 7.02-7.05(t, J = 7.5Hz, 1H), 7.10–7.11(d, J = 7 Hz, 1H), 7.33–7.36(t, J = 7 Hz, 1H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 26.43, 51.89, 65.33, 80.28, 108.30, 116.75, 122.82, 124.14, 126.87, 128.25, 130.39, 134.01, 138.03, 145.31, 164.70, 174.19; FAB mass: Calcd. For C₁₆H₁₇NO₄ is 287.12; Found : 288.32(M+1).
 Compound 3h	IR (CH₂Cl₂) $\gamma_{\max}$: 1138, 1214, 1356, 1470, 1616, 1715, 1728, 2116, 2930 cm⁻¹; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 1.20(t, J = 2Hz, 3H), 3.28(s, 3H), 3.57(s, 3H), 3.75-3.78(d, J = 17Hz, 1H), 3.97-4.00(d, J = 17Hz, 1H), 4.10-4.12(q, J = 2Hz,

Supporting Information

	2H), 6.64(s, 1H), 6.72(s, 1H), 6.86-6.87(d, $J = 7.5$Hz, 1H), 7.01-7.04(t, $J = 7.5$Hz, 1H), 7.12-7.14(d, $J = 7.5$Hz, 1H), 7.33-7.37(t, $J = 7.0$Hz, 1H); ^{13}C NMR (CDCl_3/TMS, 125.7 MHz): δ 14.07, 26.47, 51.94, 60.89, 62.25, 80.45, 108.49, 122.90, 124.71, 125.85, 128.78, 130.80, 137.31, 145.28, 164.58, 169.08, 173.40; FAB mass: Calcd. For $\text{C}_{17}\text{H}_{19}\text{NO}_6$ is 333.12; Found: 333.29(M⁺).
 Compound 4a	IR (CH_2Cl_2) γ_{max}: 1131, 1323, 1476, 1617, 1654, 1721, 2226, 2929, cm^{-1}; ^1H NMR (CDCl_3/TMS, 500.1 MHz): δ 2.08(s, 3H), 3.22(s, 3H), 3.95(s, 3H), 5.72(s, 2H), 6.79-6.81(d, $J = 7.5$Hz, 1H), 6.98-7.01(m, 1H), 7.31-7.34(m, 2H); ^{13}C NMR (CDCl_3/TMS, 125.7 MHz): δ 26.03, 29.71, 38.27, 52.83, 108.35, 119.62, 122.58, 124.30, 128.18, 131.57, 136.66, 144.04, 166.81, 166.85, 177.27; FAB mass: Calcd. For $\text{C}_{15}\text{H}_{15}\text{NO}_5$ is 289.09; Found: 290.10 (M+1).
 Compound 6c	IR (CH_2Cl_2) γ_{max}: 1131, 1323, 1470, 1723, 2224, 2928, cm^{-1}; ^1H NMR (CDCl_3/TMS, 500.1 MHz): δ 2.13(s, 3H), 4.20(s, 5H), 4.49-4.50(m, 2H), 4.66(s, 2H), 4.84-4.85(m, 2H), 7.08(s, 1H); ^{13}C NMR (CDCl_3/TMS, 125.7 MHz): δ 20.97, 65.45, 69.96, 70.08(2C), 71.71(7C), 75.75, 100.42, 118.68, 149.86, 170.58 ; FAB mass: Calcd. For $\text{C}_{16}\text{H}_{15}\text{FeNO}_2$ is 309.04; Found: 310.14 (M+1).
	IR (CH_2Cl_2) γ_{max}: 1130, 1325, 1465, 1618, 1721, 2214, 2921, cm^{-1};

Supporting Information

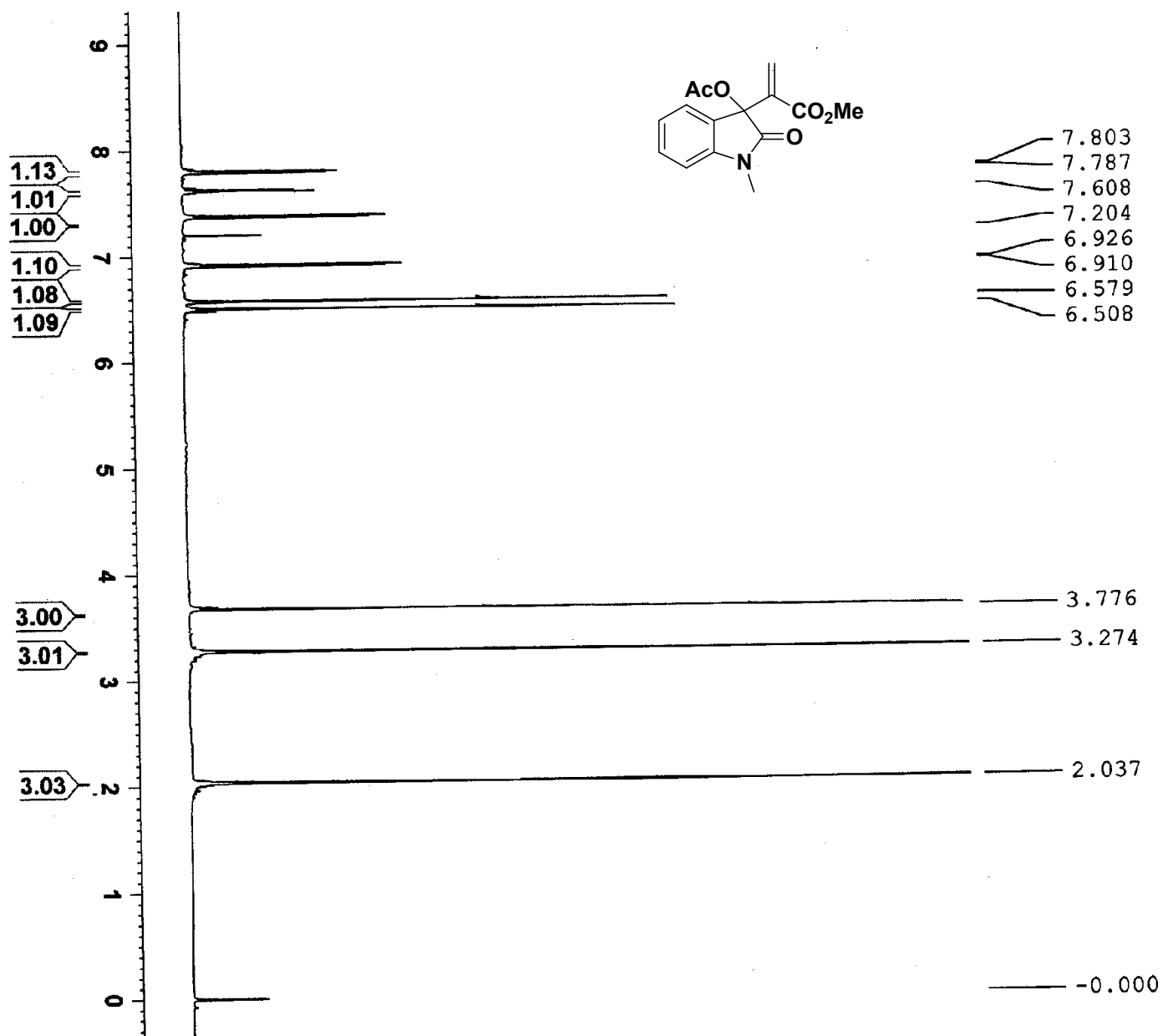
Compound 6d	¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 2.54(s, 3H), 3.82(s, 3H), 7.11(s, 1H), 7.54(s, 1H), 7.87-7.89(m, 2H), 8.01-8.03(m, 2H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 23.25, 50.22, 75.38, 123.83, 124.00, 129.29, 136.00, 136.38, 141.20, 141.30, 152.01, 167.79, 168.29, 199.03, 199.57; FAB mass: Calcd. For C₁₅H₁₂O₆ is 288.06; Found: 289.06 (M+1).
 Compound 6e	IR (CH₂Cl₂) γ_{max}: 1142, 1517, 1615, 1721, 2913, 3072 ; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 1.26-1.28 (t, <i>J</i> = 2Hz, 3H), 3.30(s, 3H), 4.22-4.27(q, <i>J</i> = 2Hz, 2H), 4.49(s, 2H), 6.72-6.73(d, <i>J</i> = 8Hz, 1H), 7.08-7.11(t, <i>J</i> = 7.5Hz, 1H), 7.37-7.40(t, <i>J</i> = 7.5 Hz, 1H), 8.07-8.09(d, <i>J</i> = 7.5Hz 1H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 14.90, 23.46, 41.35, 61.23, 108.55, 115.64, 123.72, 128.75, 132.46, 143.99, 144.62, 163.16, 167.46; FAB mass: Calcd. For C₁₃H₁₄N₂O₄ is 262.09; Found: 263.52. (M+1).
 Compound 6f	IR (CH₂Cl₂) γ_{max}: 1143, 1515, 1617, 1724, 2911, 3085; ¹H NMR (CDCl₃/TMS, 500.1 MHz): δ 1.24-1.31(m, 6H), 4.20-4.28(m, 4H), 4.49(s, 2H), 5.02(s, 2H), 6.72-6.73(d, <i>J</i> = 7.5Hz, 1H), 7.08-7.11(t, <i>J</i> = 7.0Hz, 1H), 7.37-7.40(t, <i>J</i> = 7.0Hz, 1H), 8.07-8.09(d, <i>J</i> = 7.5Hz 1H); ¹³C NMR (CDCl₃/TMS, 125.7 MHz): δ 14.10, 14.15, 41.23, 61.35, 61.90, 72.89, 108.62, 115.64, 123.55, 128.75, 132.99, 143.46, 144.72, 163.12, 167.16, 168.41; FAB mass: Calcd. For C₁₆H₁₈N₂O₆ is 334.12; Found: 335.56 (M+1).
 Compound 6g	IR (CH₂Cl₂) γ_{max}: 1135, 1345, 1654, 1684, 1724, 2860, 2910, 3080;

Supporting Information

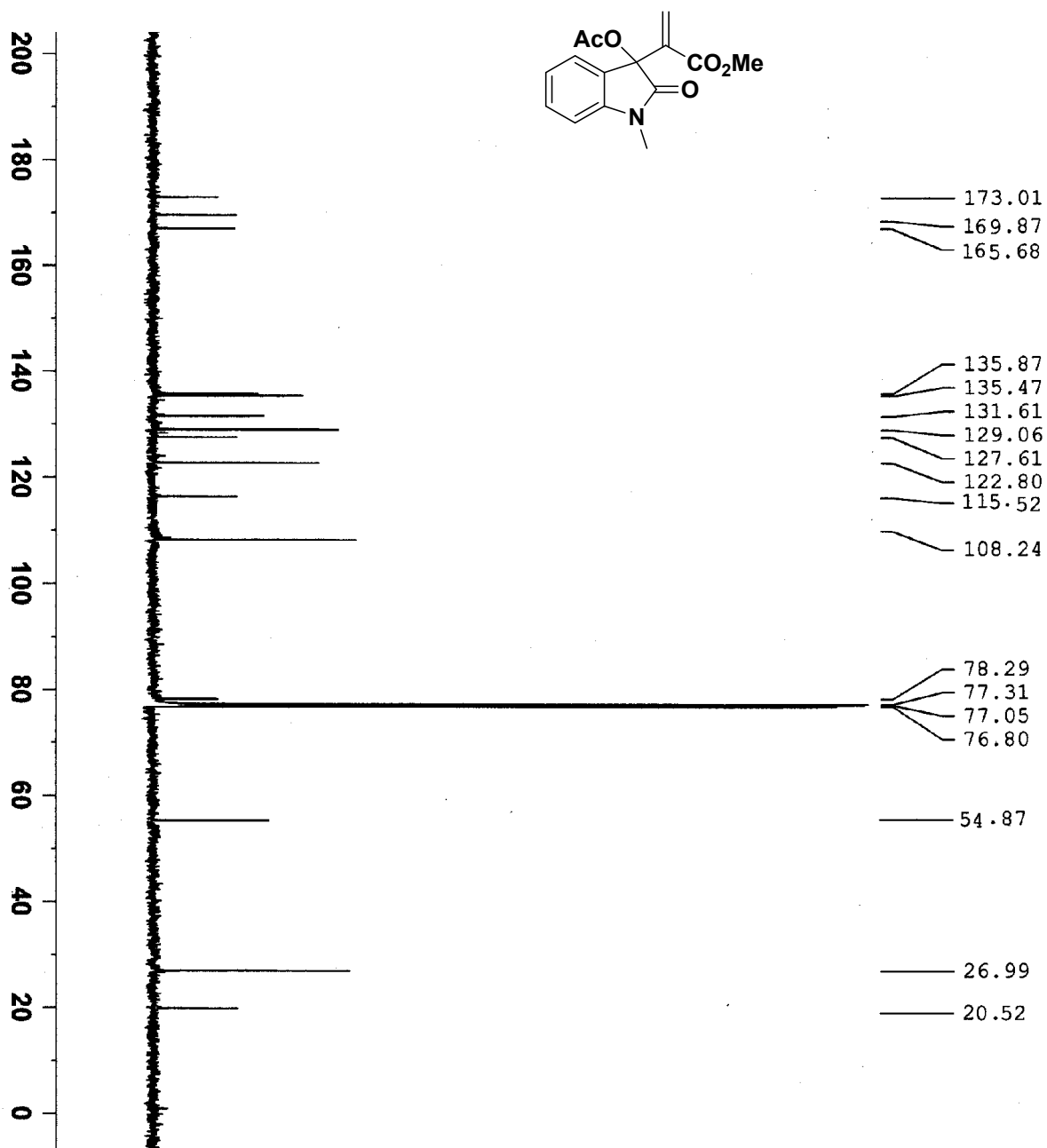
Compound 6g	¹H NMR (CDCl₃/TMS, 300.1 MHz): δ 1.25-1.31(m, 3H), 4.23-4.30(m, 2H), 4.76(s, 2H), 6.62-6.68(d, <i>J</i> = 16Hz, 1H), 7.56-7.61(d, <i>J</i> = 8.7Hz, 2H), 7.68-7.71(d, <i>J</i> = 16Hz, 1H), 8.25-8.28(d, <i>J</i> = 8.7Hz, 2H); ¹³C NMR (CDCl₃/TMS, 75.3 MHz): δ 14.12, 61.12, 61.62, 121.14, 124.22(2C), 128.81(2C), 140.20, 143.16, 148.69, 165.25, 167.62; FAB mass: Calcd. For C₁₃H₁₃NO₆ is 279.07; Found: 280.31(M+1).
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Copies of ^1H and ^{13}C NMR spectra of new compounds:

Compound 3a

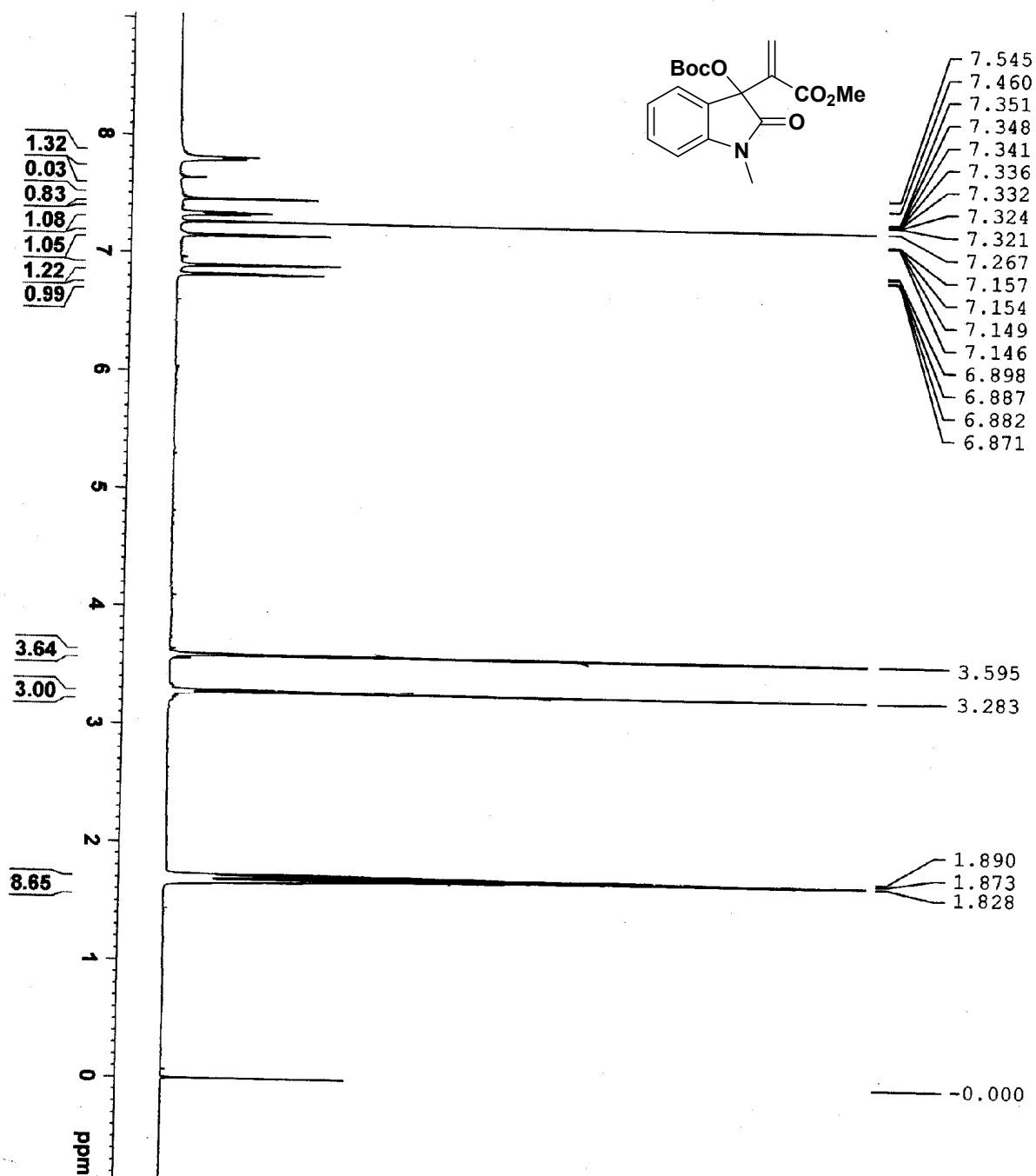


Compound 3a

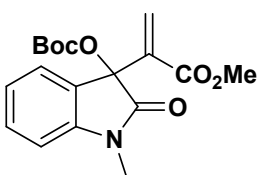
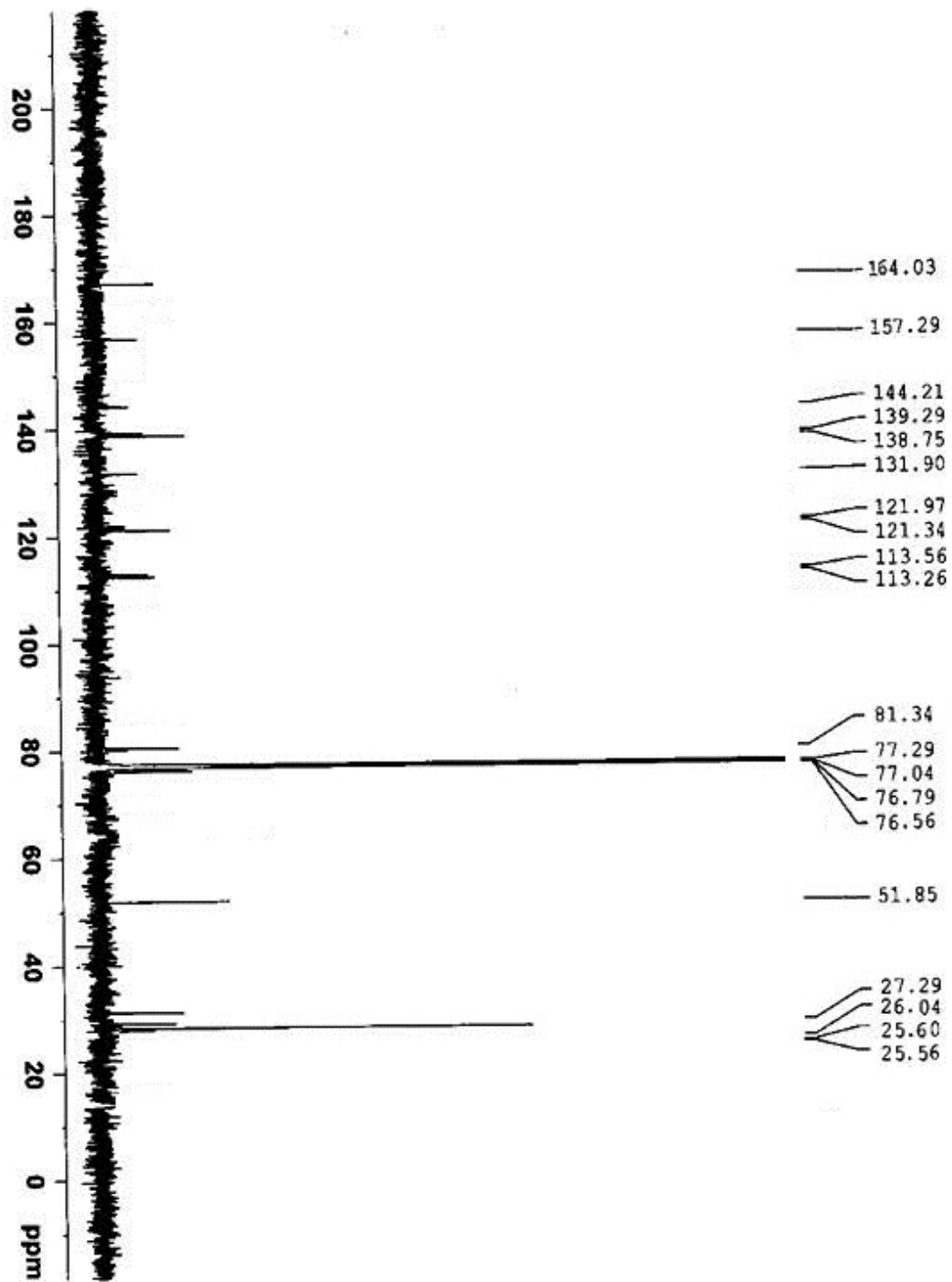


Supporting Information

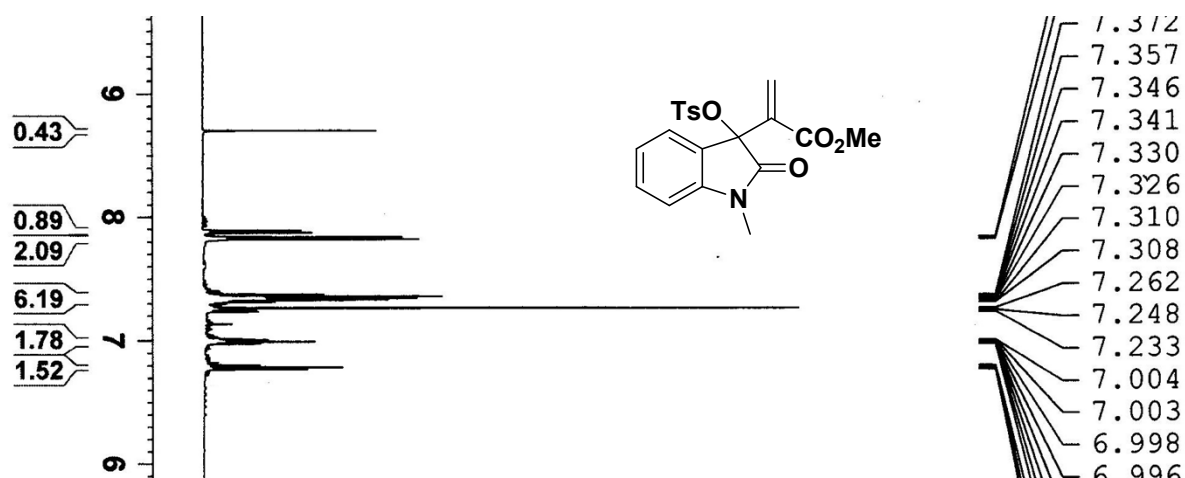
Compound 3b



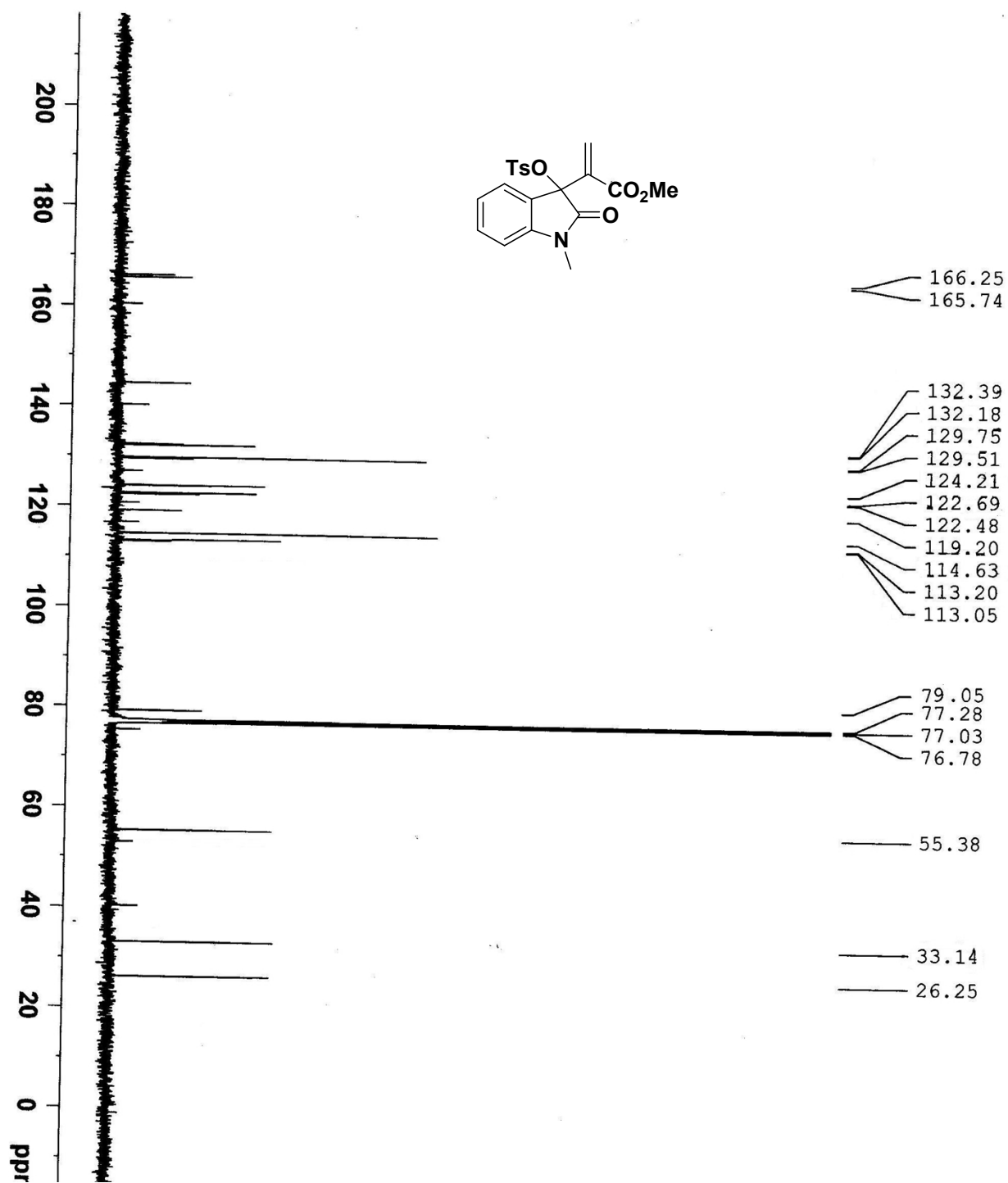
Compound 3b



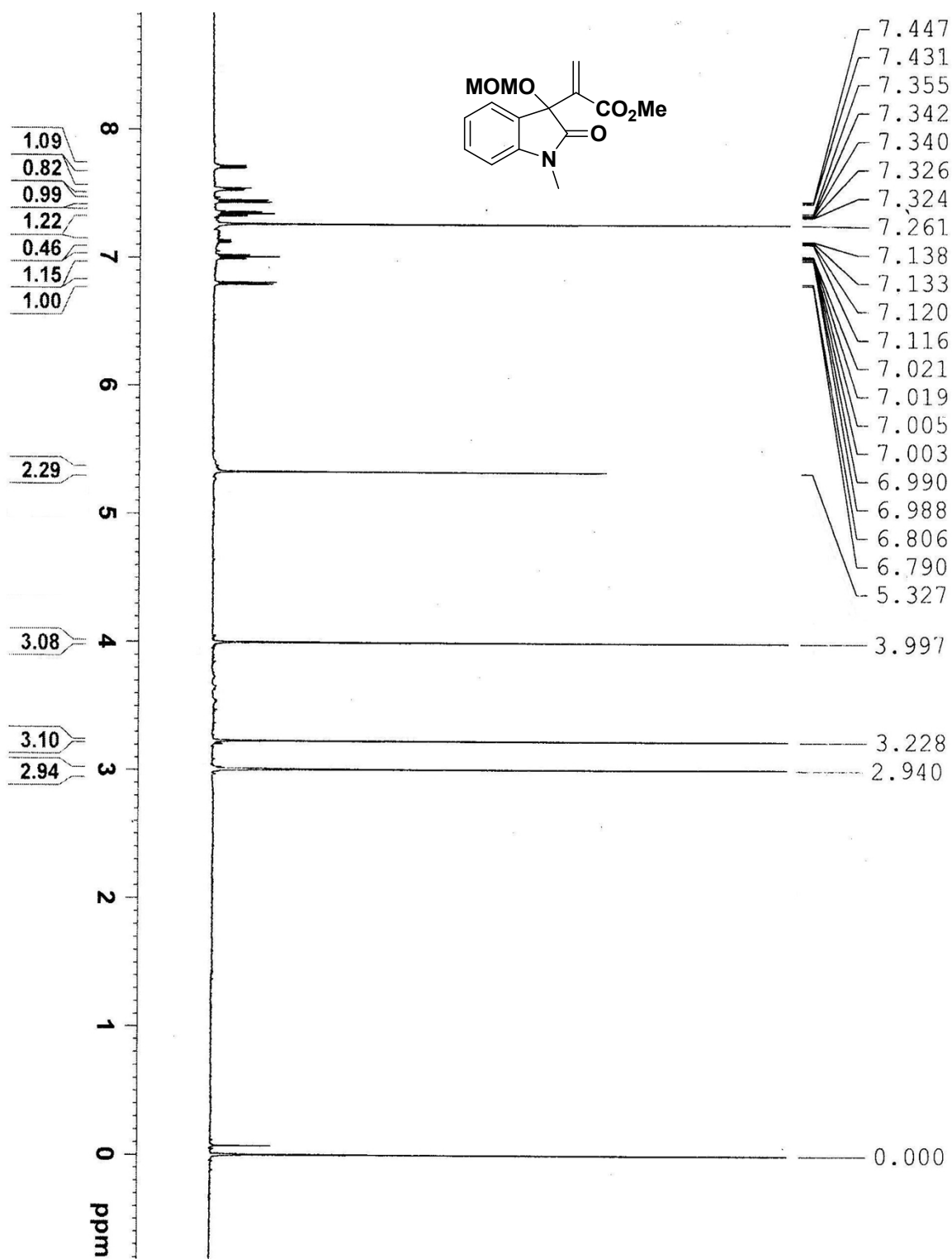
Compound 3c



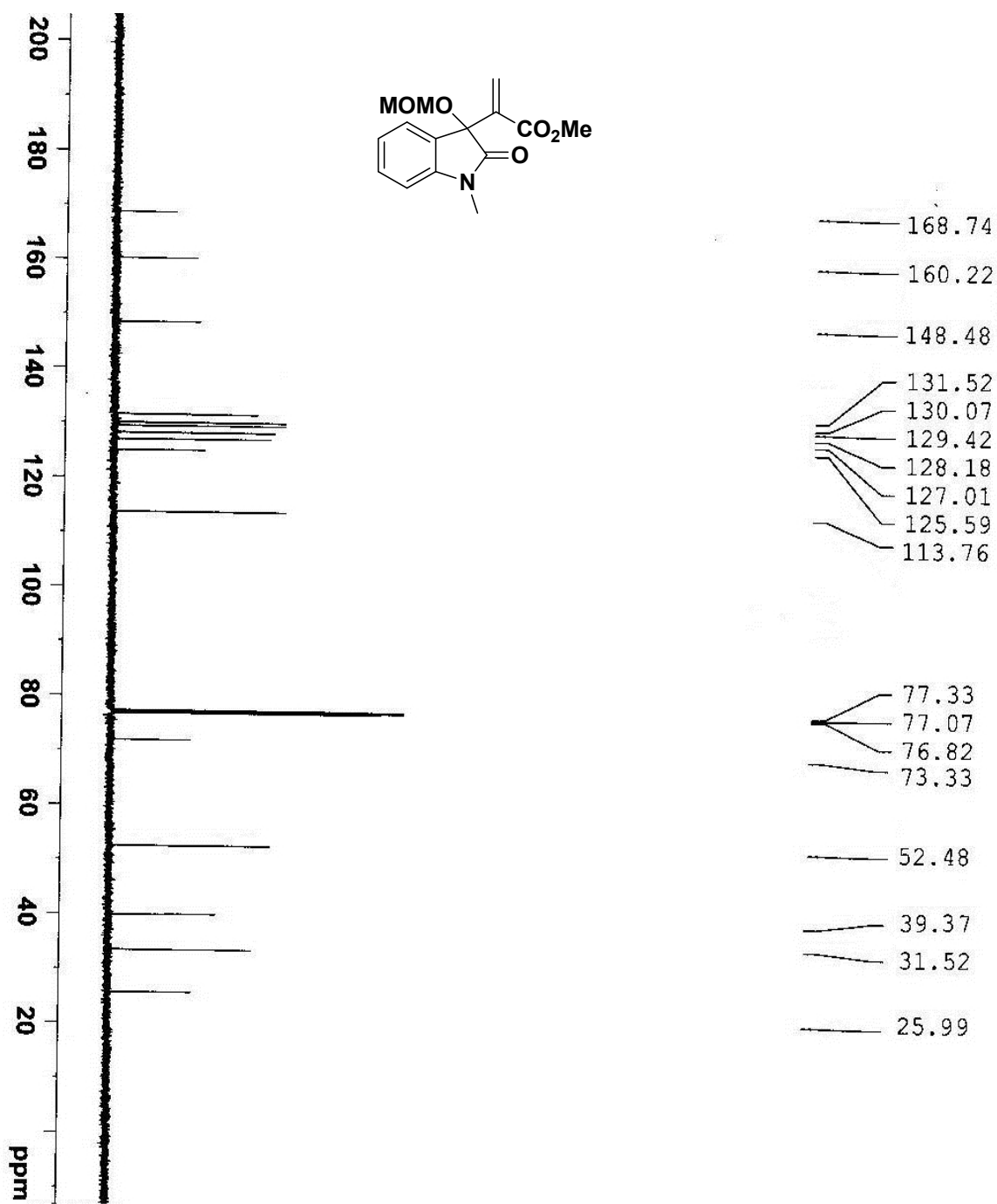
Compound 3c



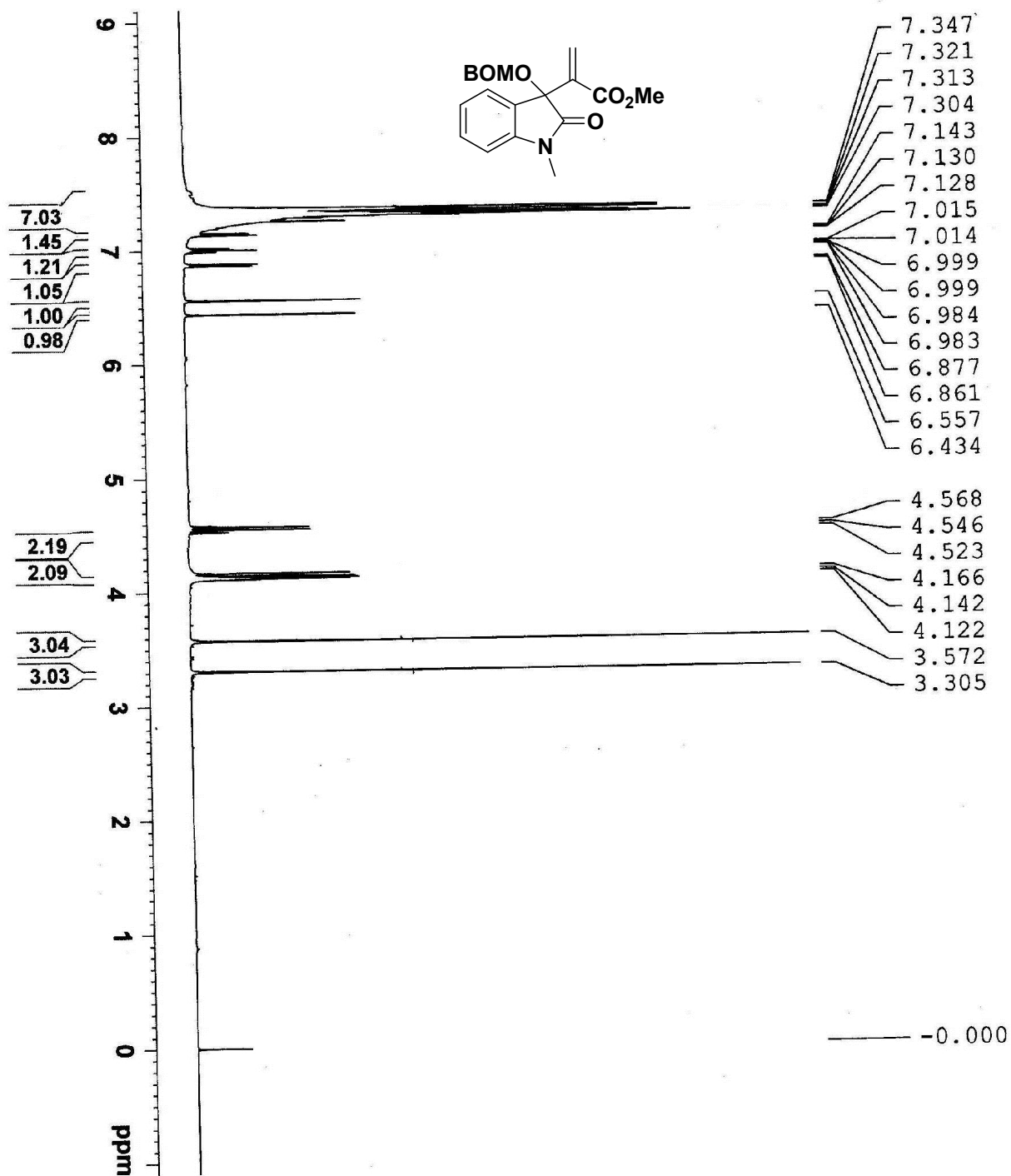
Compound 3d



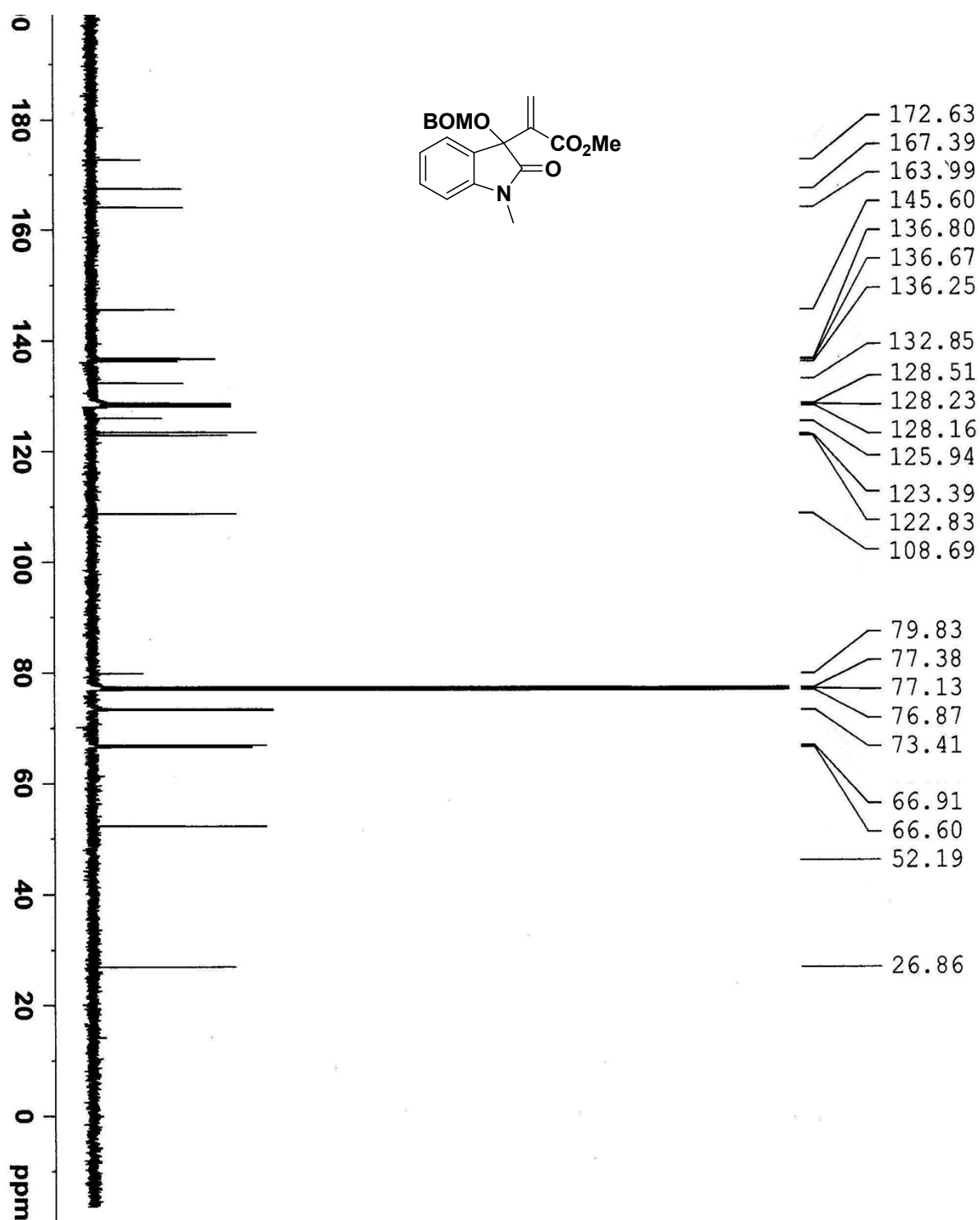
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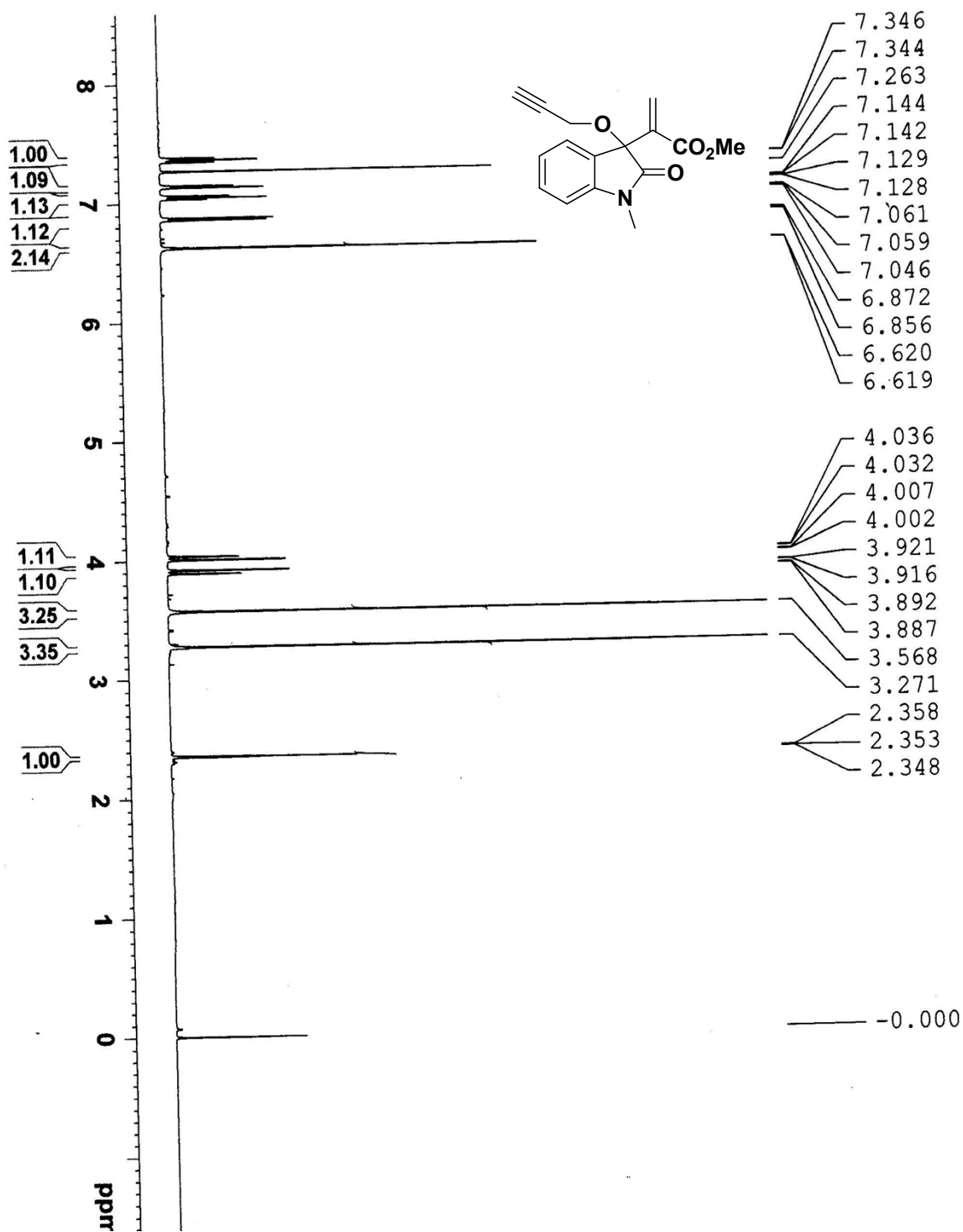
Compound 3e



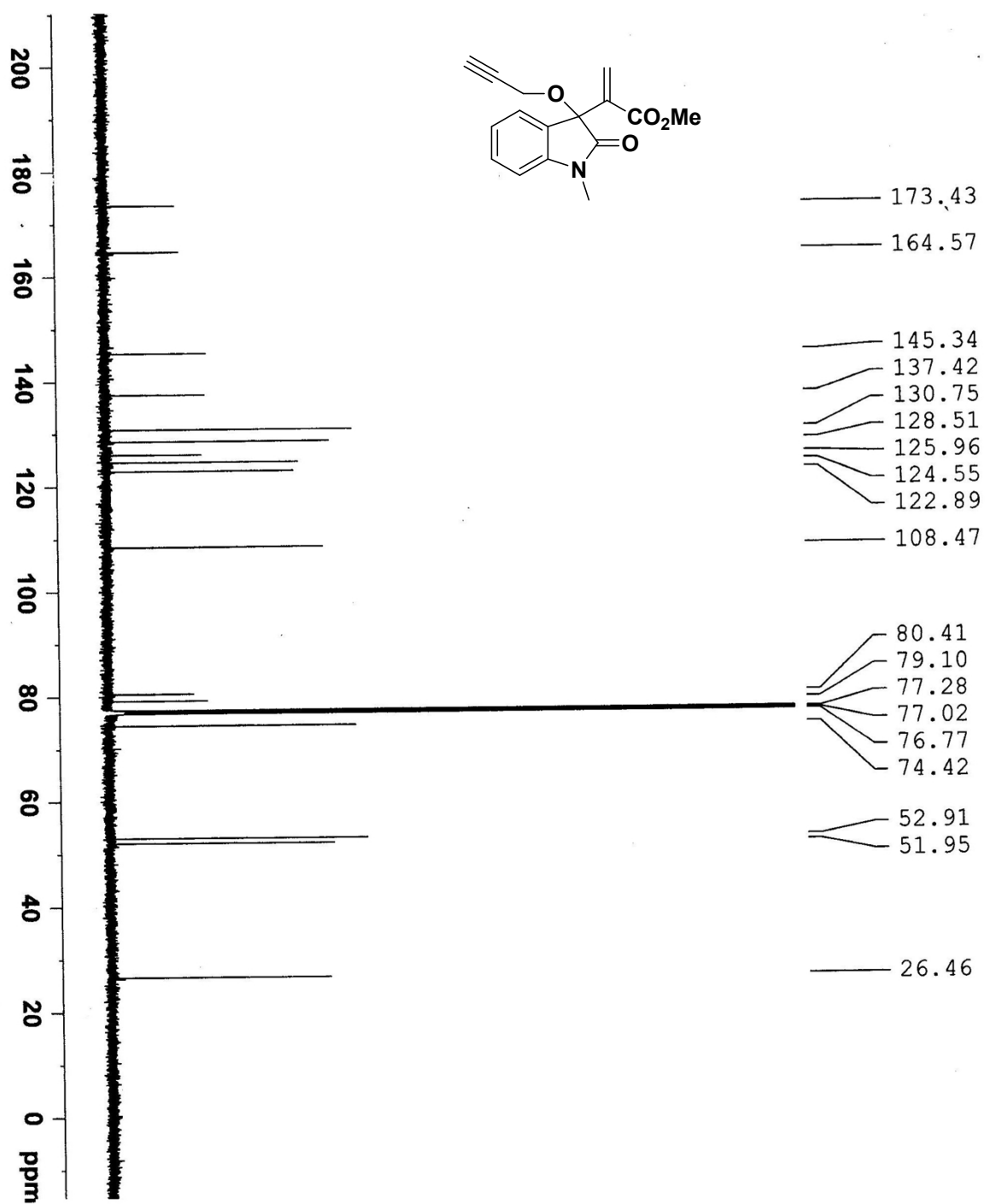
Compound 3e



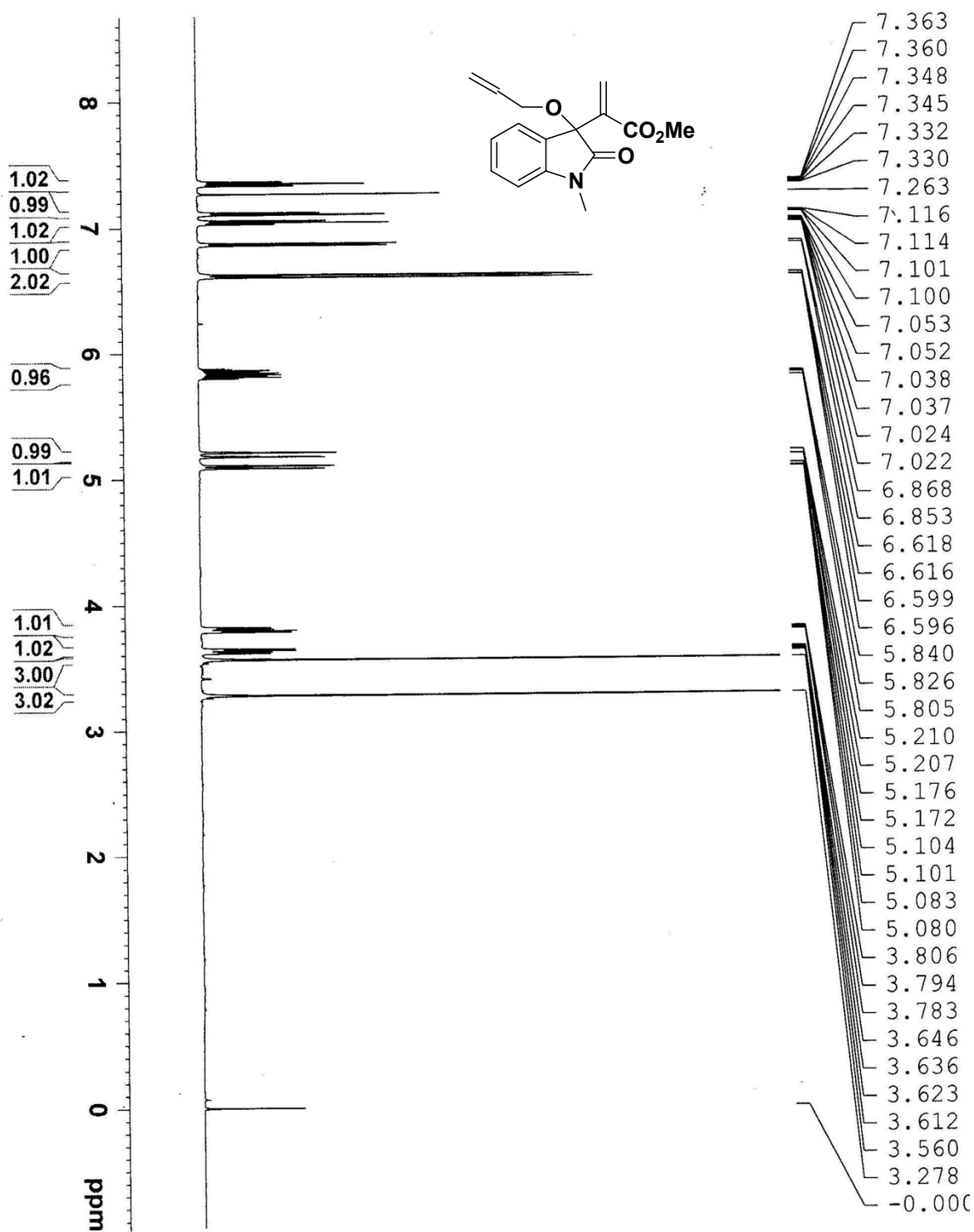
Compound 3f



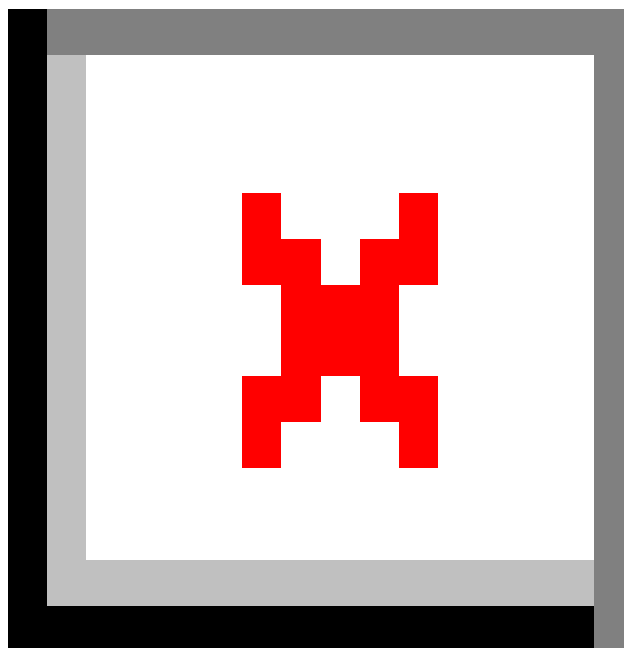
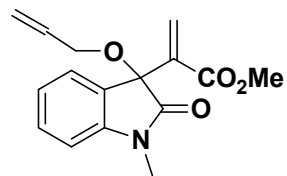
Compound 3f



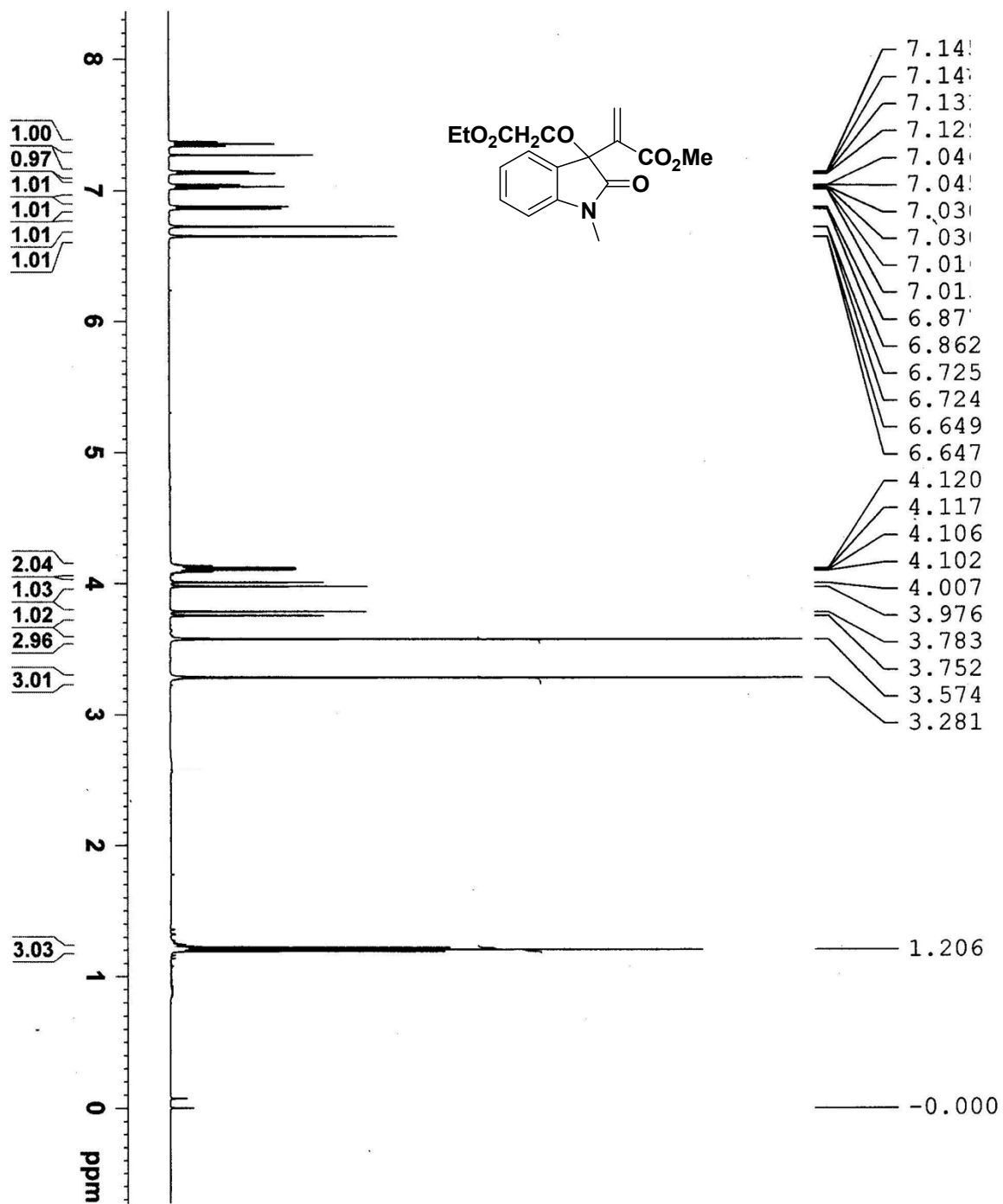
Compound 3g



Compound 3g

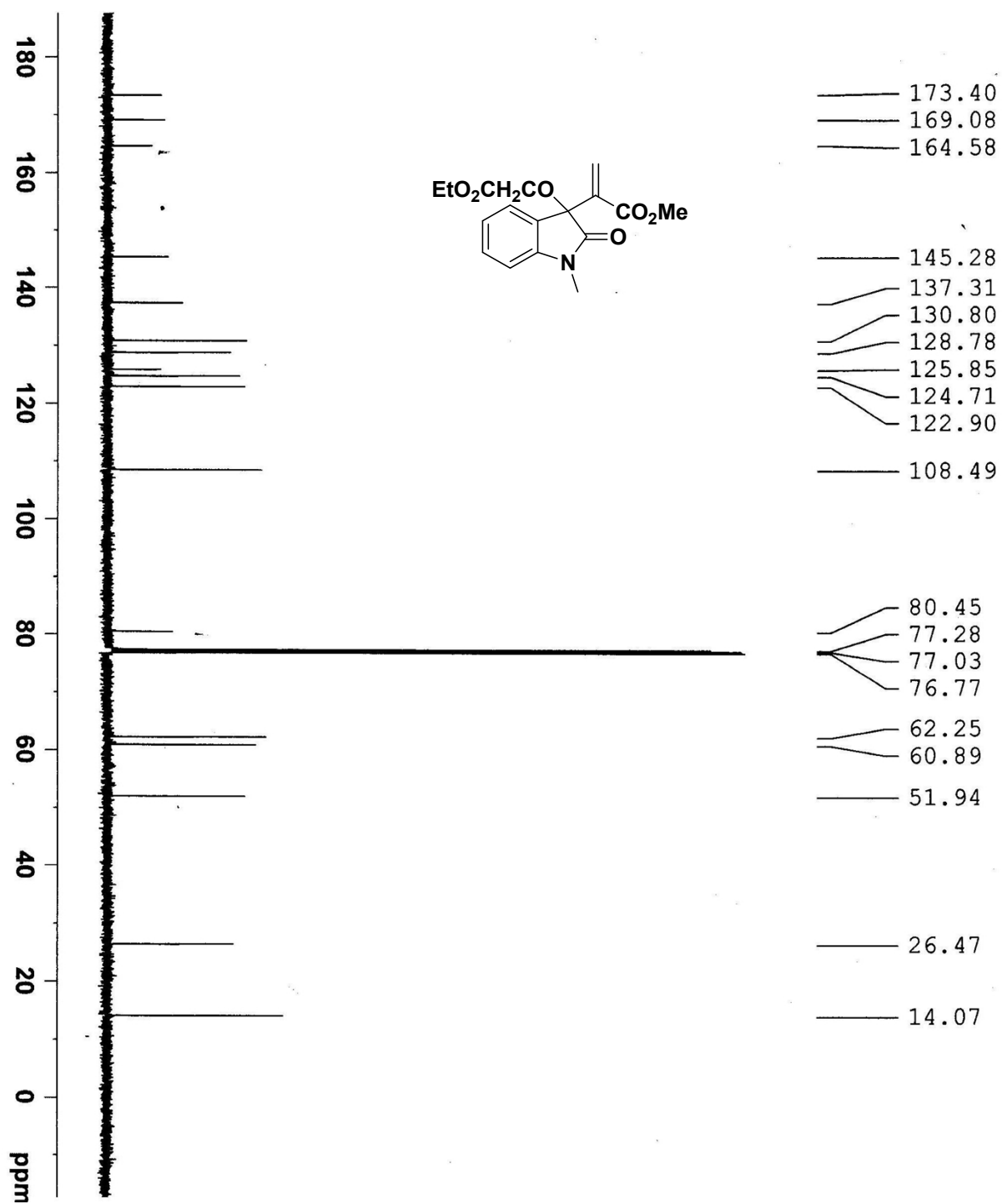


Compound 3h

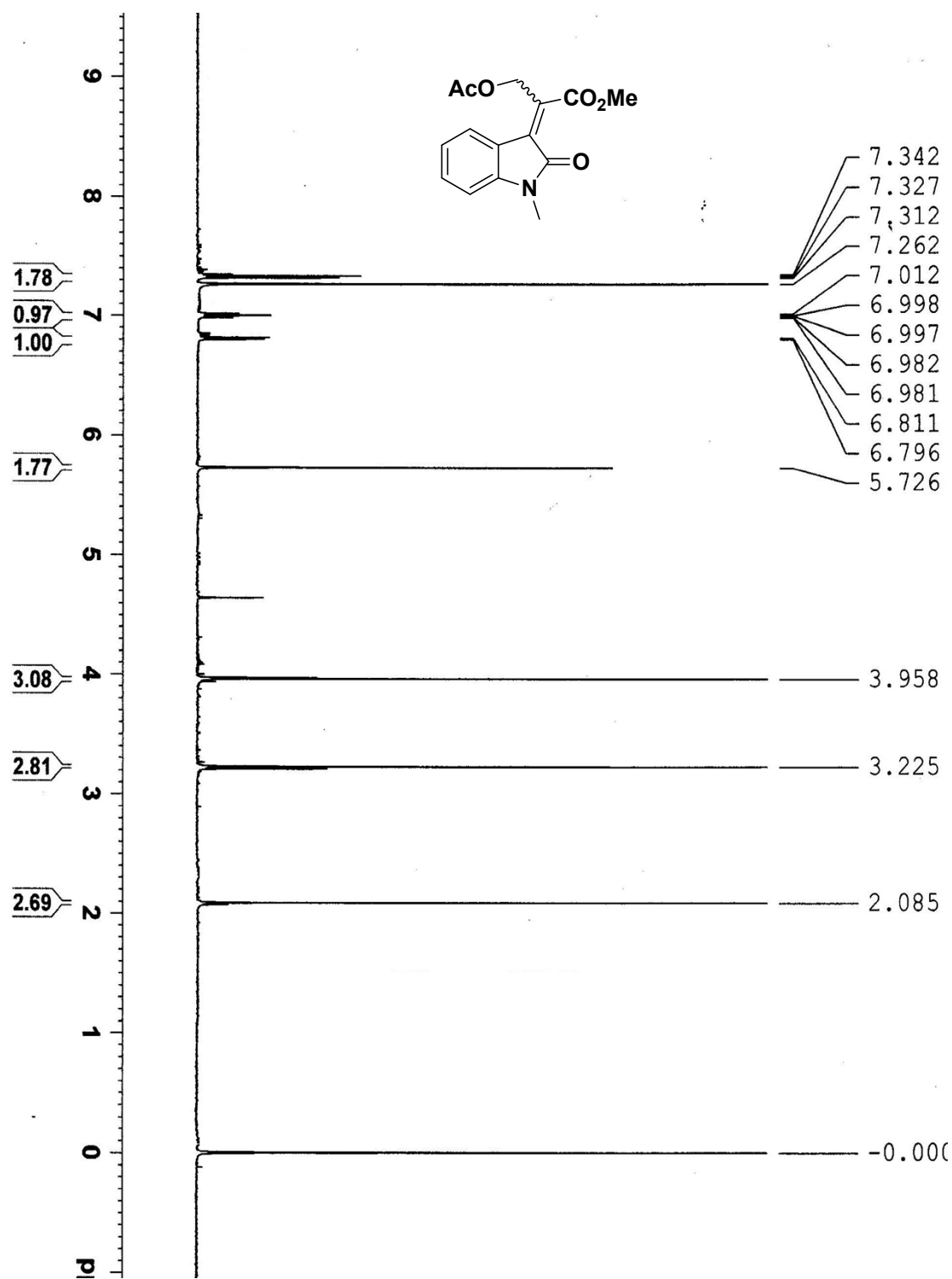


Compound 3h

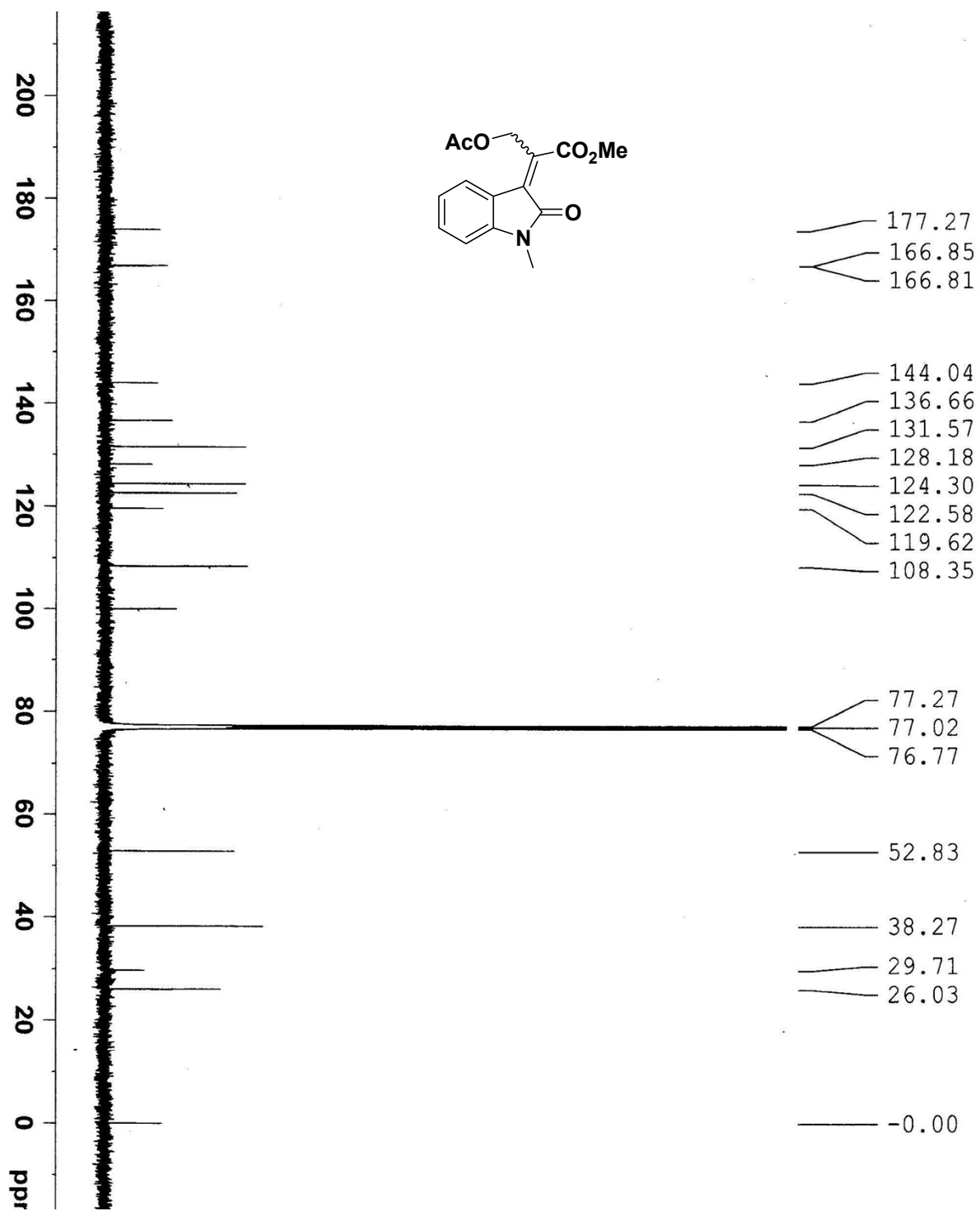
Supporting Information



Compound 4a

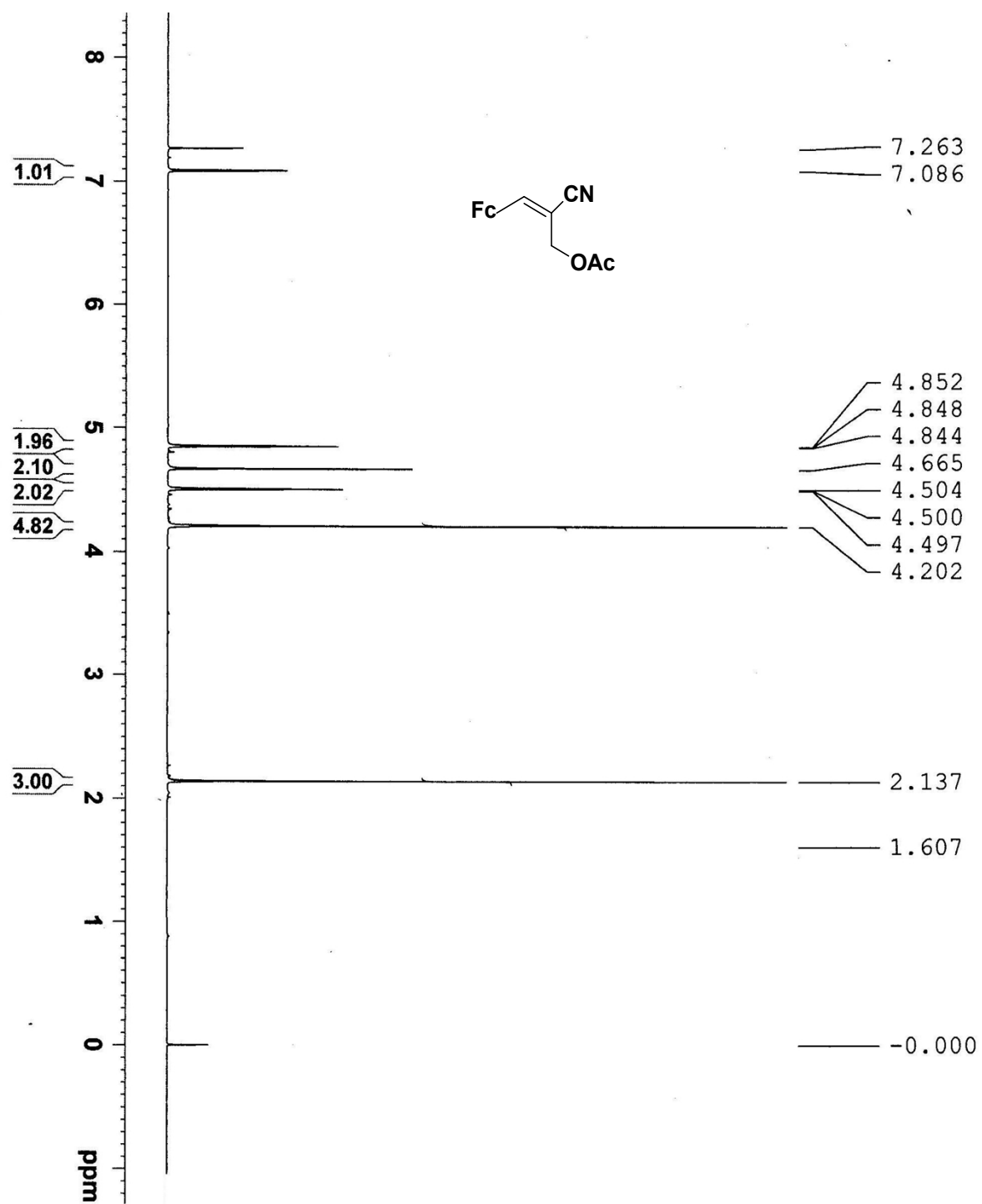


Compound 4a

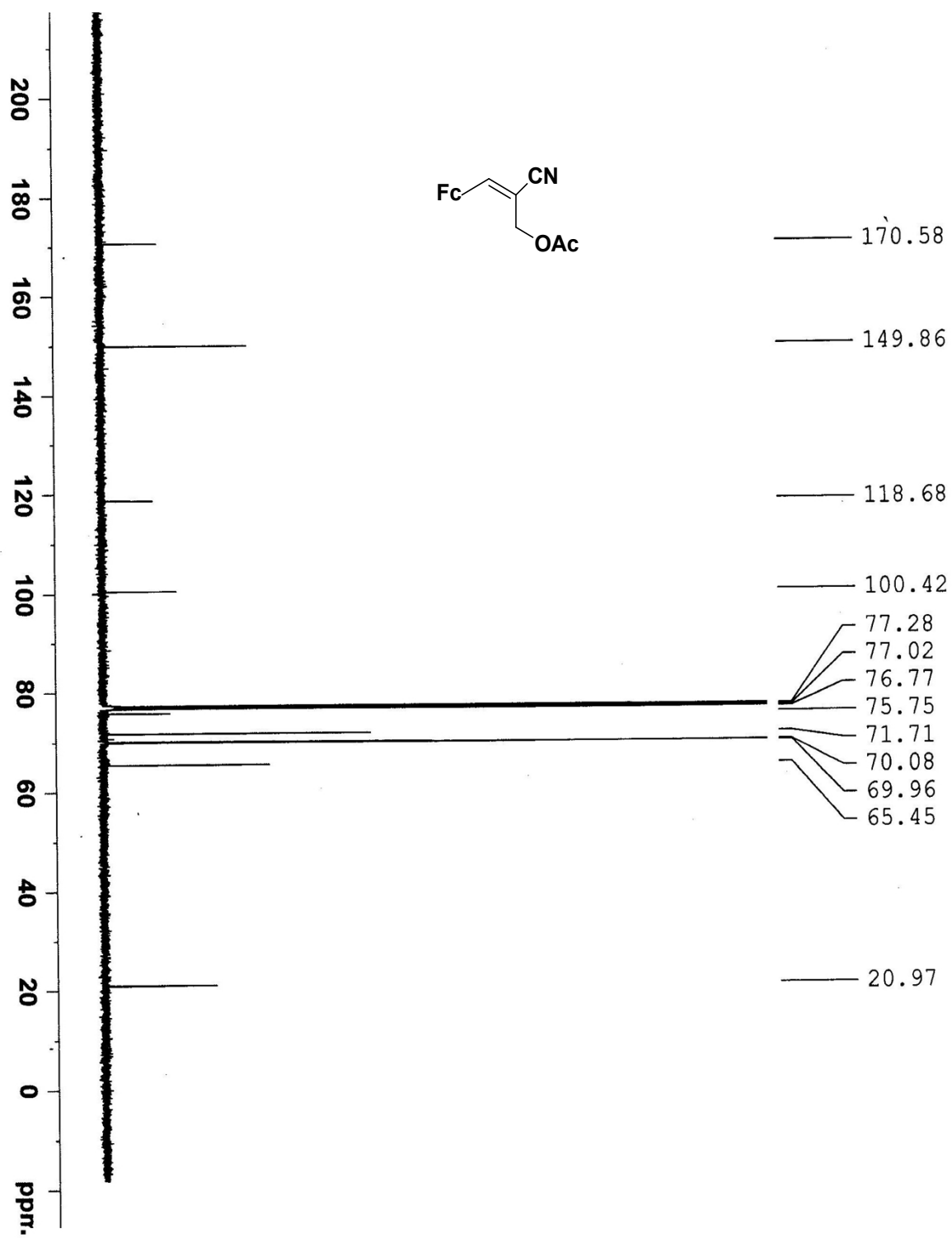


Compound 6c

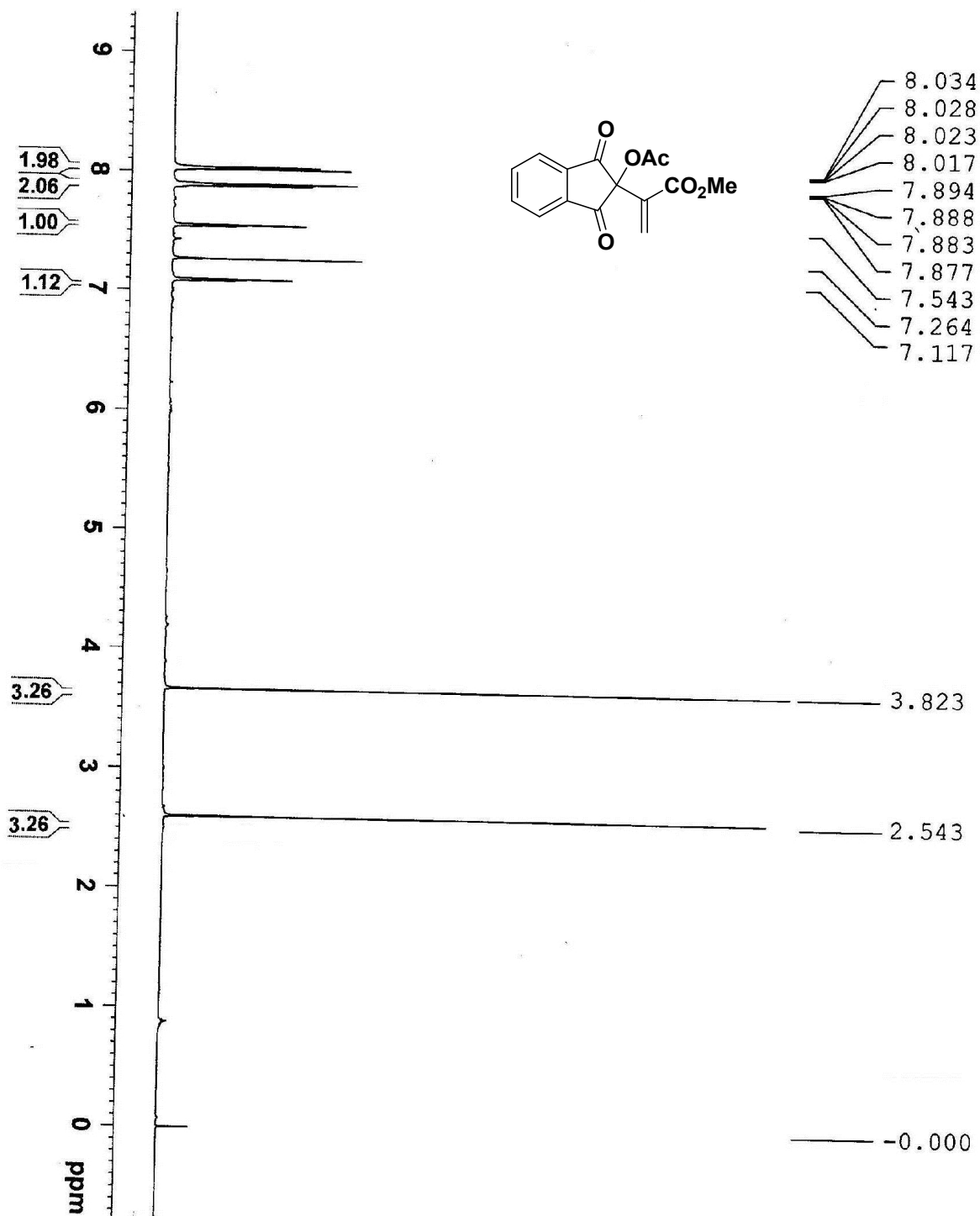
Supporting Information



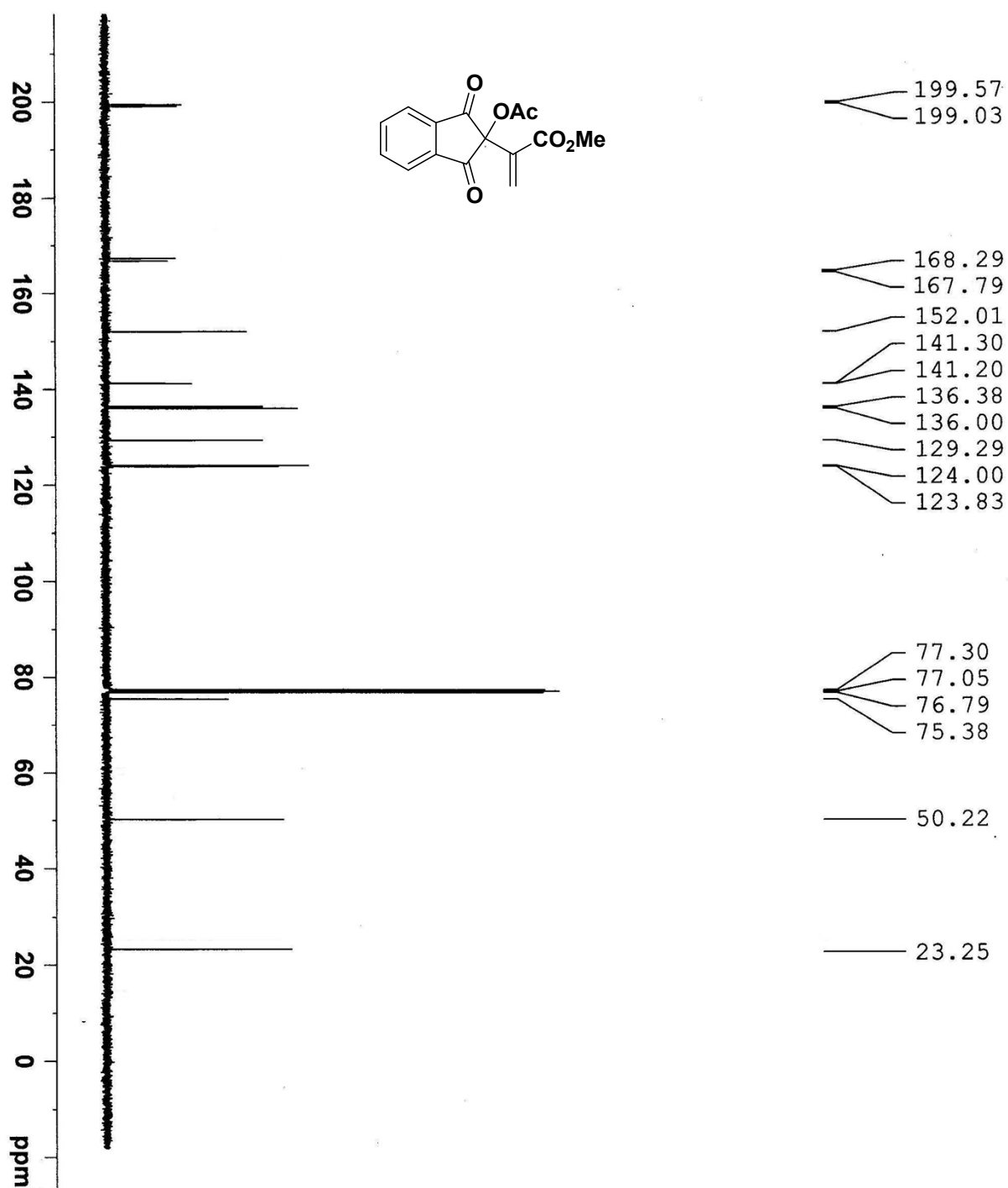
Compound 6c



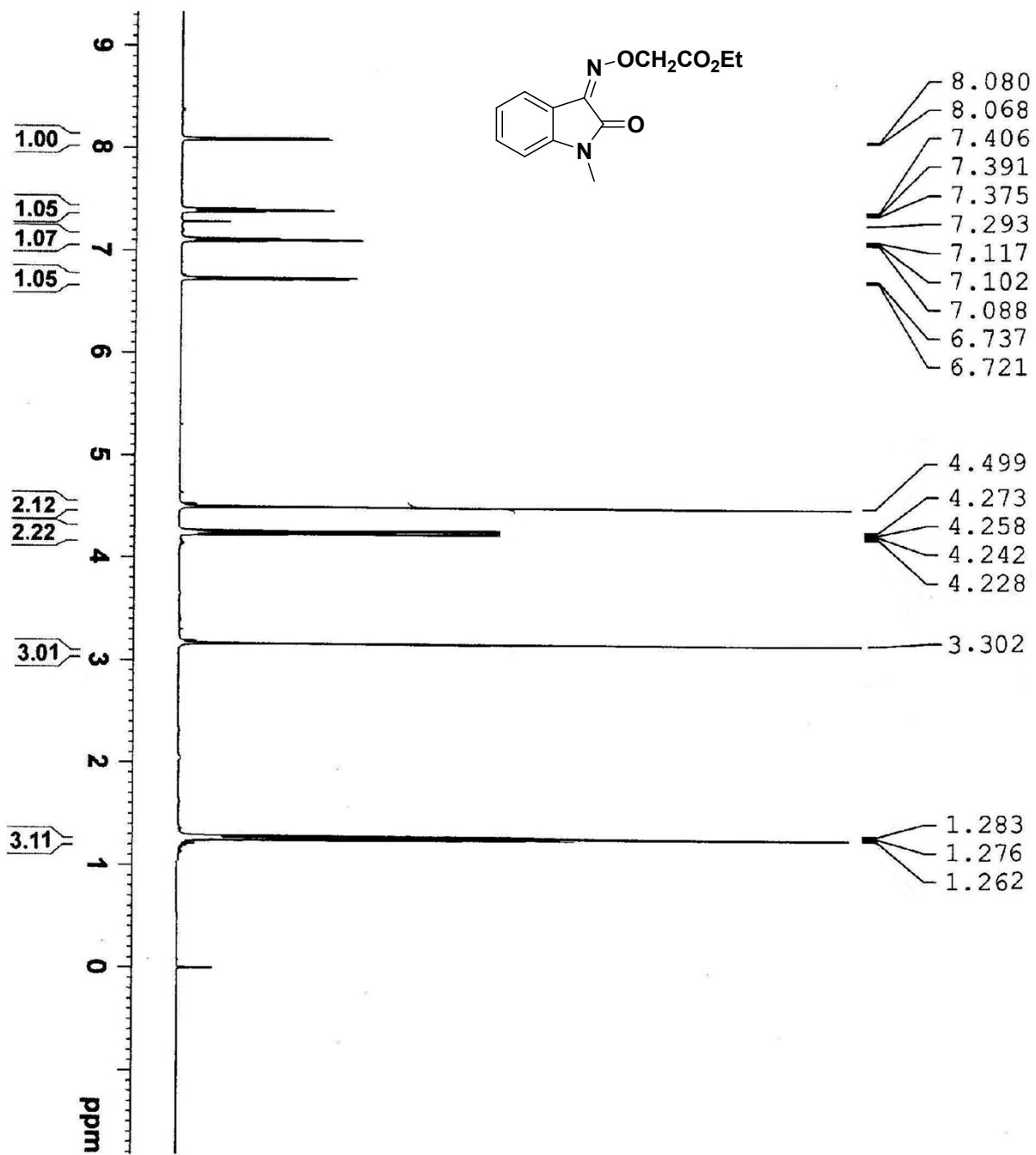
Compound 6d



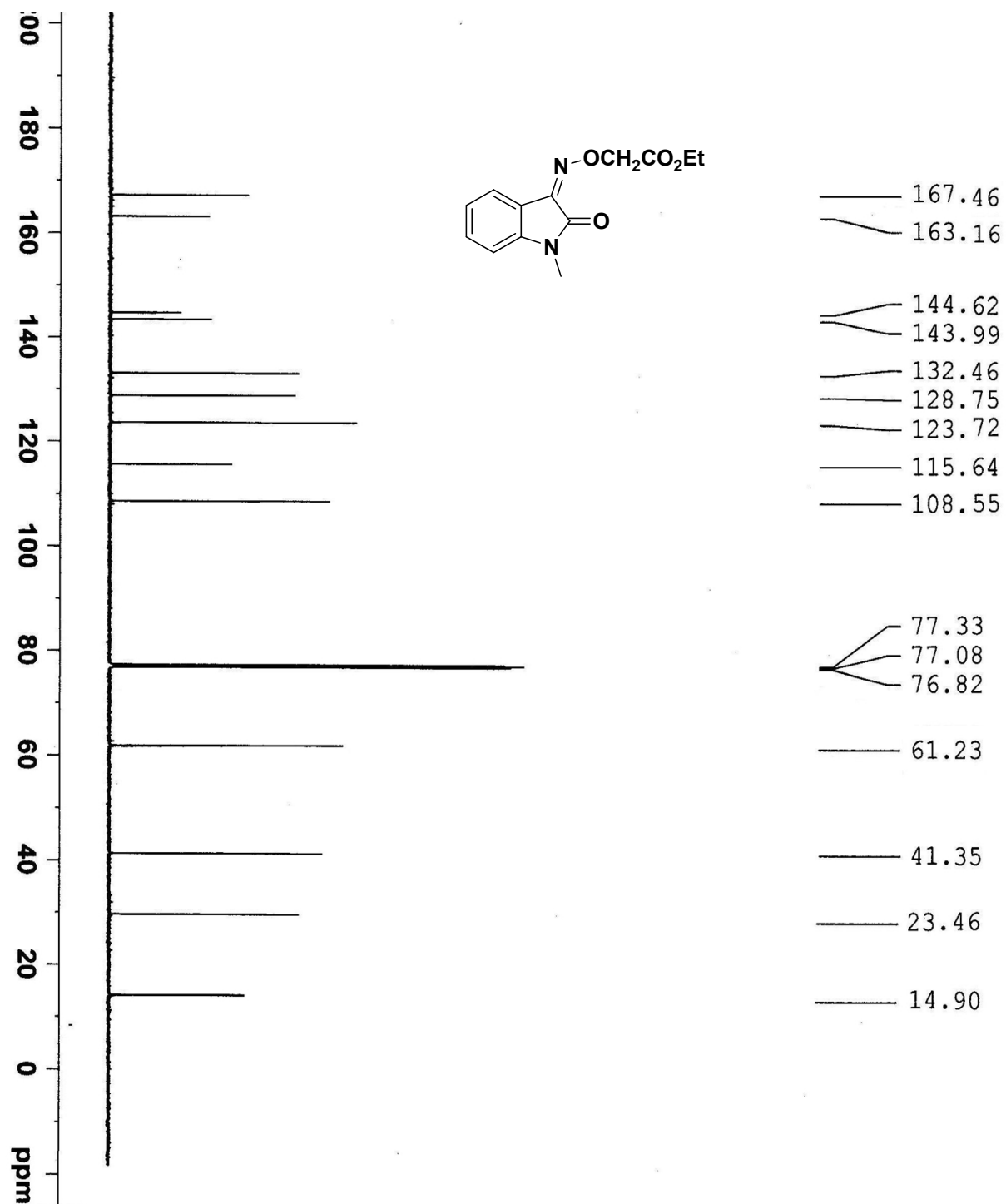
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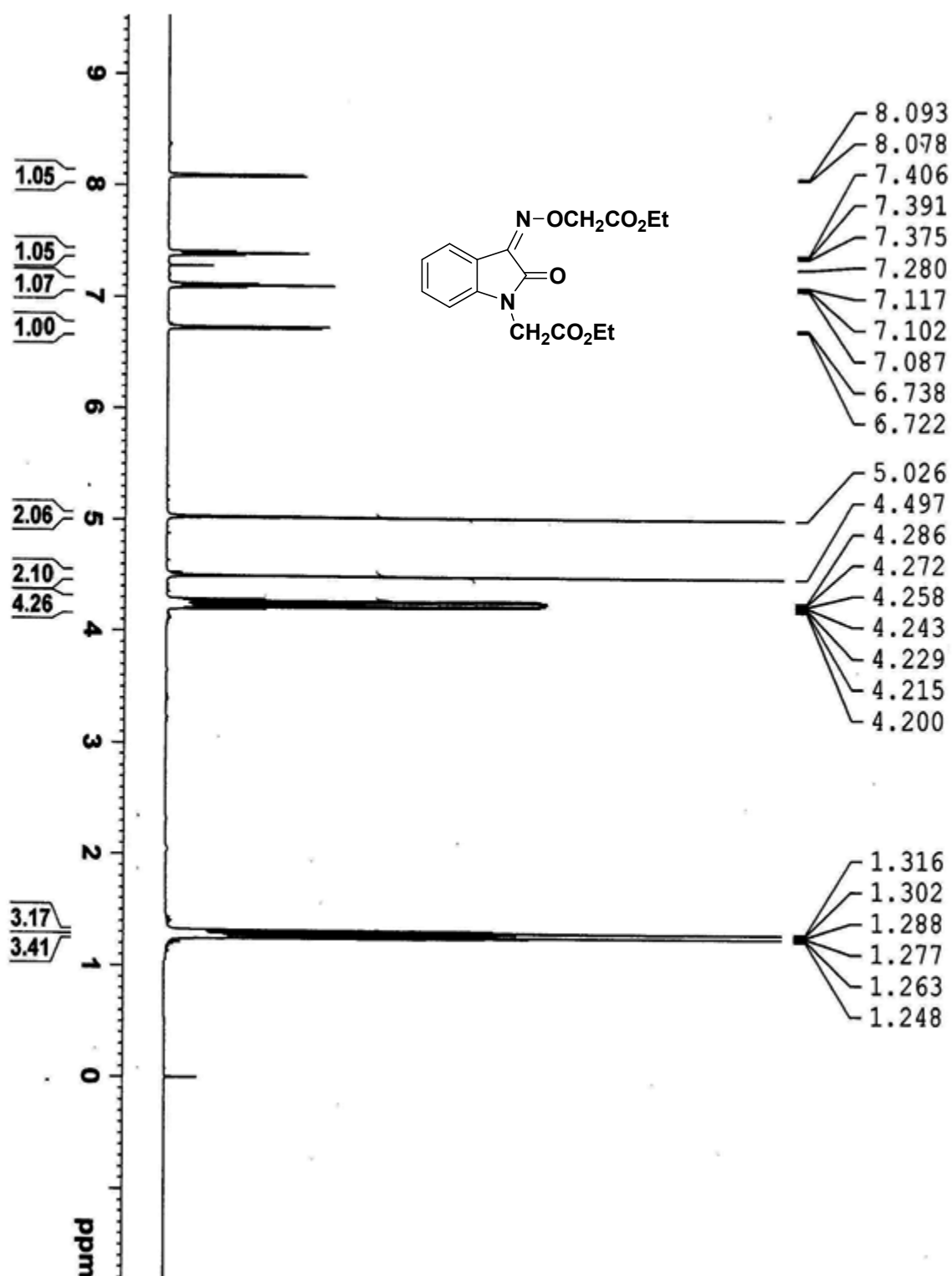
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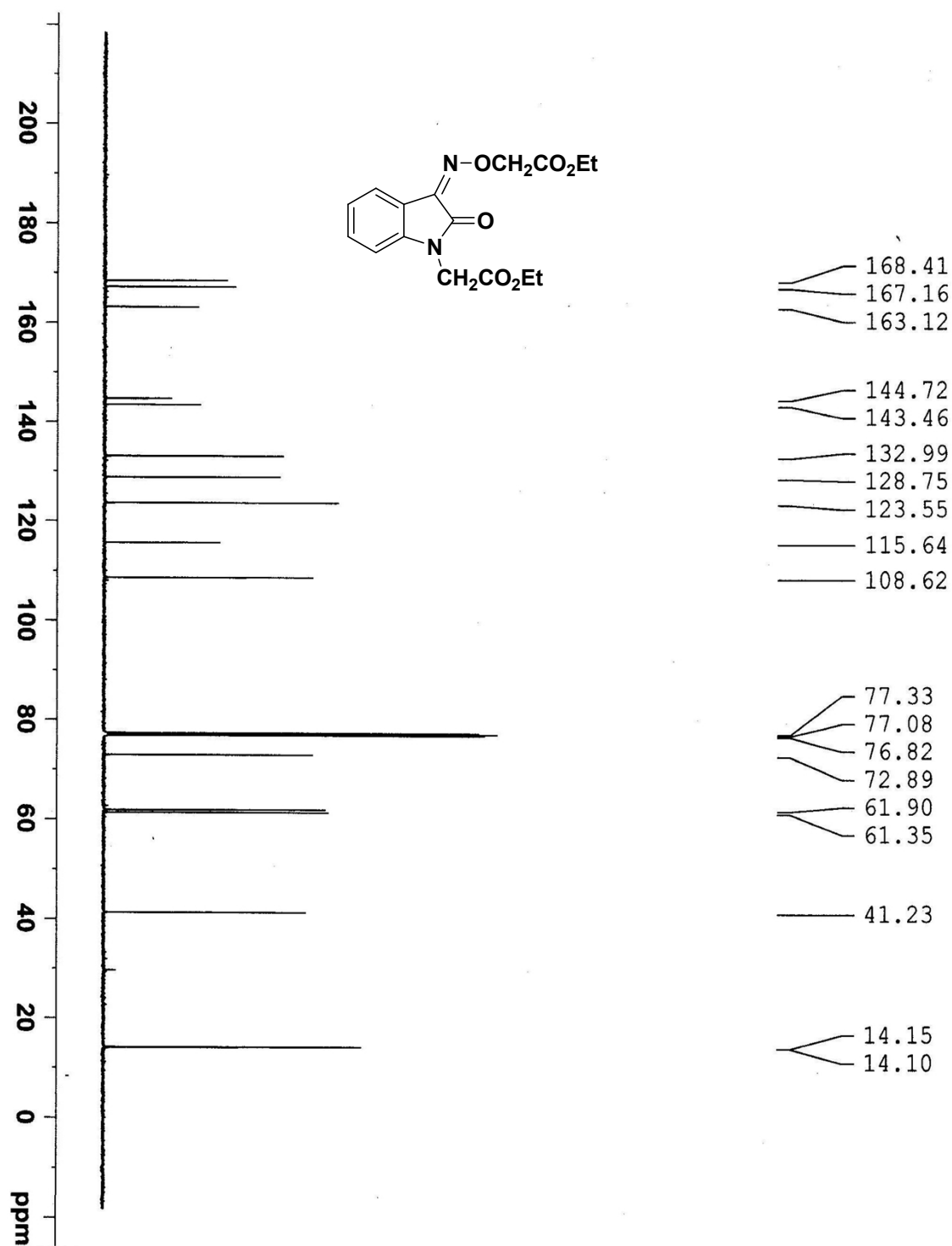
Compound 6e



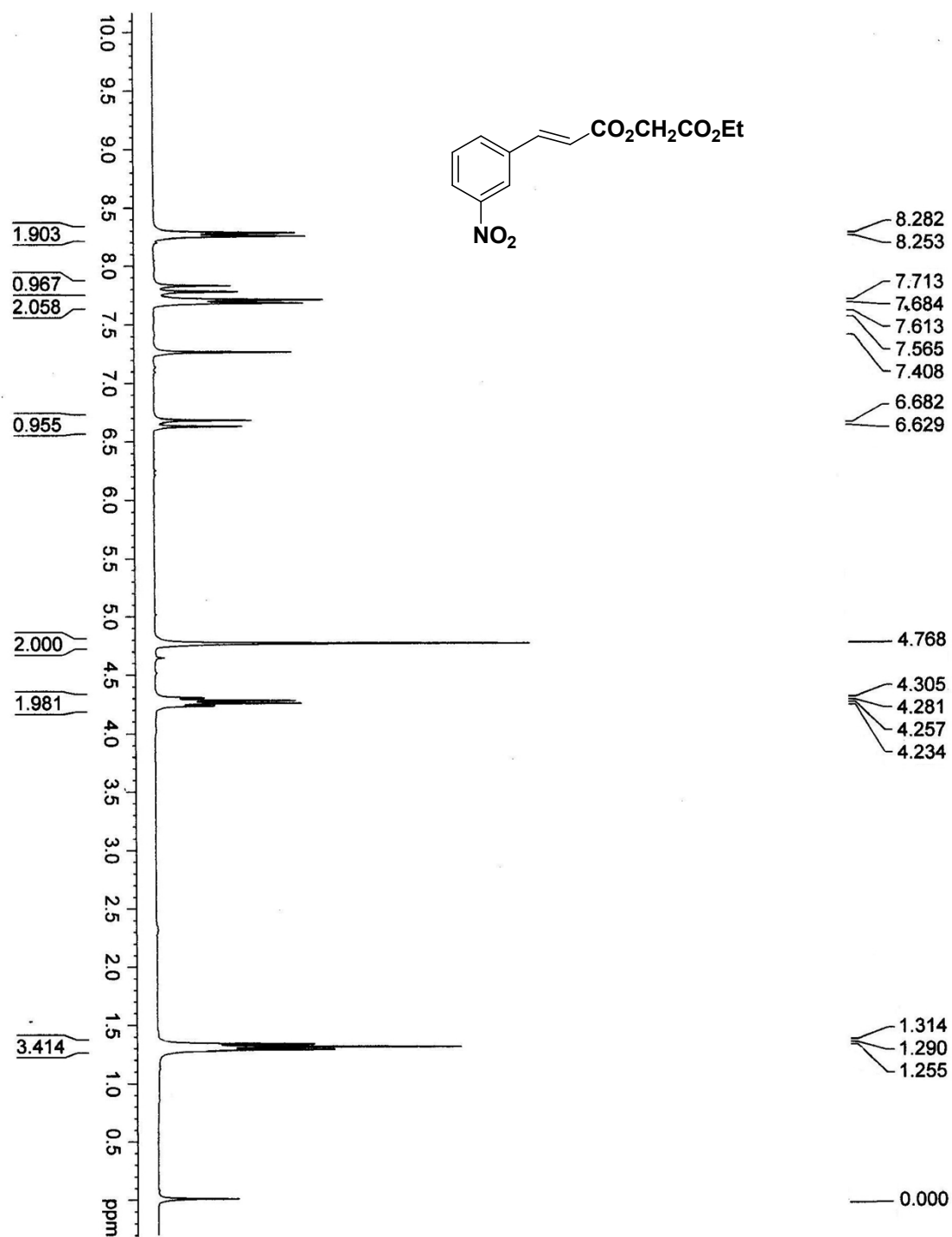
Compound 6f



Compound 6f



Compound 6g



Compound 6g

