Design and Development of FGF-23 Antagonists: Definition of the pharmacophore and initial structure-activity relationships probed by synthetic analogues.

Supporting Information

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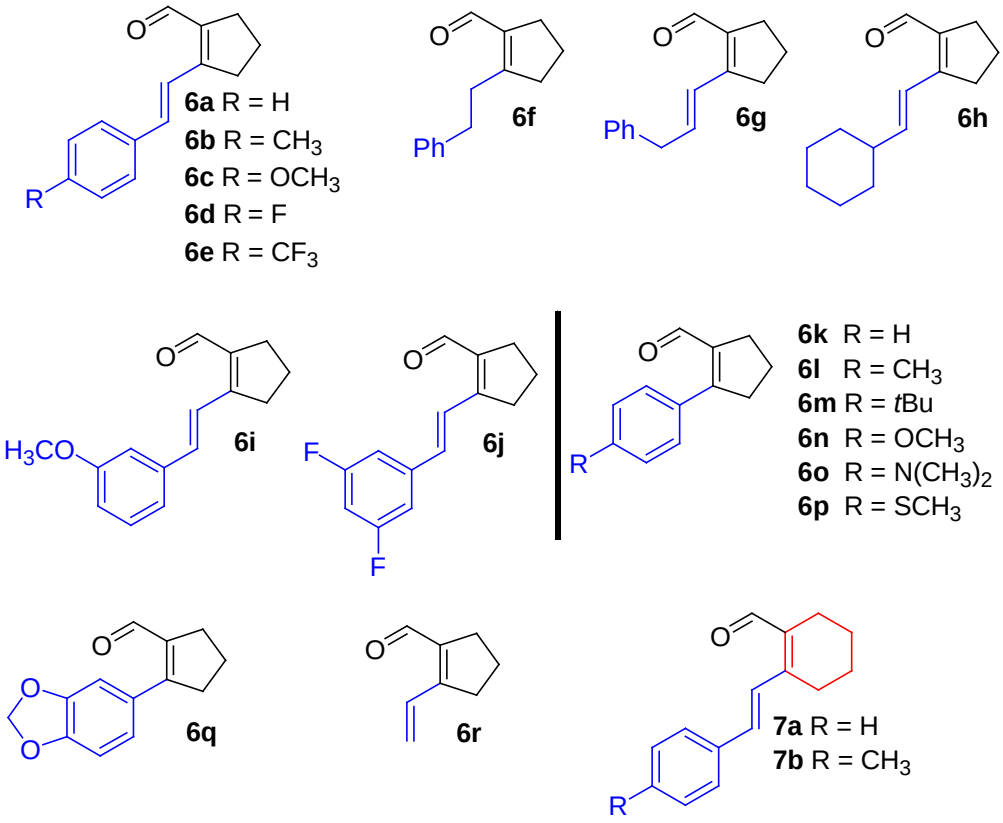
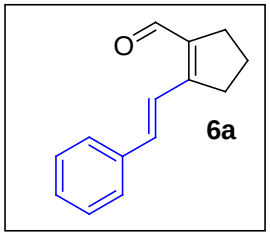
General Procedure for Suzuki-Miyaura Cross-Coupling of Scaffolds: To an 8 mL reaction vial equipped with a magnetic stir bar at ambient temperature was charged $Pd(OAc)_2$, RuPhos, Cs_2CO_3 (3 equiv), the requisite substrate (0.857 mmol), and the desired organoboron reagent (equivalents listed for each reaction). The mixture was slurried in Tol:H_2O (4:1) (0.2 M) and heated to 100 °C for 16 hours upon which time the crude mixture was analyzed by ^1H NMR to ascertain percent conversion of the starting material. Concentration of the crude reaction mixtures under reduced pressure at ambient temperature followed by purification on normal phase silica gel using automated flash-column chromatography with MTBE:hexanes, or EtOAc:hexanes gradient mobile phases afforded the compounds described in the listed yields.

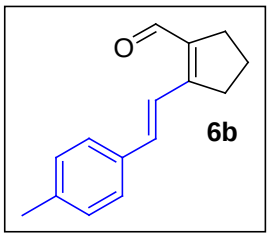
Compounds **6a**, **6k**, and **6r** are known compounds in the primary literature.[1]

Table S1. Aliphatic core aldehyde synthons for analogue preparation.

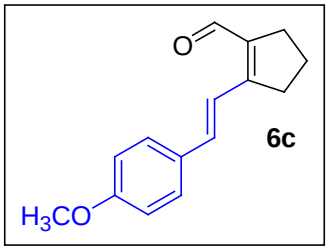




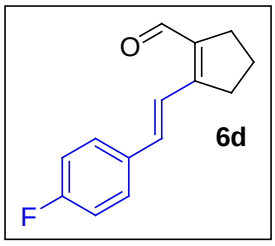
2-Styryl-cyclopent-1-enecarbaldehyde (6a)-precursor to 1: Prepared according to the general procedure discussed above with **4** (1.71 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % Ruphos, and 1.20 equiv potassium styryltrifluoroborate, R_F = 0.62, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with a 5% isocratic hold; isolated yield 0.235 g, 70%; orange solid; mp = 97.5−99.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.33 (s, 1H), 7.68 (d, *J* = 16.0 Hz, 1H), 7.51 (d, *J* = 7.5 Hz, 2H), 7.38 (t, *J* = 7.5 Hz, 2H), 7.35−7.30 (m, 1H), 6.87 (d, *J* = 16.0 Hz, 1H), 2.91 (t, *J* = 7.5 Hz, 2H), 2.72 (t, *J* = 7.5 Hz, 2H), 1.96 (pent, *J* = 7.5 Hz, 2H). Experimental data above was commensurate with results published previously.[2]



2-(2-*p*-Tolyl-vinyl)-cyclopent-1-enecarbaldehyde (6b)-precursor to 8a: Prepared according to the general procedure discussed above with **4** (0.571 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % Ruphos, and 1.05 equiv 4-methylstyrenylboronic acid, R_F = 0.58, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with a 5% isocratic hold; isolated yield 0.050 g, 42%; orange solid; mp = 104.2−107.3 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.33 (s, 1H), 7.64 (d, *J* = 15.9 Hz, 1H), 7.41 (d, *J* = 8.0 Hz, 2H), 7.19 (d, *J* = 8.0 Hz, 2H), 6.84 (d, *J* = 15.9 Hz, 1H), 2.90 (br-t, *J* = 7.7 Hz, 2H), 2.71 (br-t, *J* = 7.7 Hz, 2H), 2.37 (s, 3H), 1.95 (pent, *J* = 7.7 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.6, 158.2, 139.5, 136.8, 133.6, 129.8, 127.3, 119.2, 34.9, 31.2, 21.53, 21.48; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3035, 2952, 2850, 1653, 1616, 1580, 804 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{15}H_{16}O$: 212.1201; found: 212.1208.

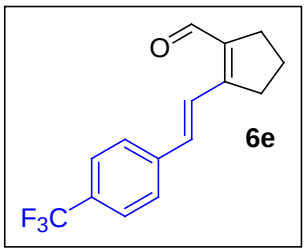


2-[2-(4-Methoxy-phenyl)-vinyl]-cyclopent-1-enecarbaldehyde (6c)-precursor to 8b: Prepared according to the general procedure discussed above with **4** (0.571 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % Ruphos, and 1.05 equiv 4-methoxystyrenylboronic acid, R_F = 0.35, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with a 5% isocratic hold; isolated yield 0.090 g, 69%; orange solid; mp = 107.0−110.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.32 (s, 1H), 7.56 (d, *J* = 15.7 Hz, 1H), 7.48−7.44 (m, 2H), 6.93−6.89 (m, 2H), 6.83 (d, *J* = 15.7 Hz, 1H), 3.84 (s, 3H), 2.89 (br-t, *J* = 7.5 Hz, 2H), 2.71 (br-t, *J* = 7.5 Hz, 2H), 1.95 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.6, 160.6, 158.4, 138.9, 138.5, 129.2, 128.8, 118.0, 114.5, 55.5, 34.9, 31.2, 21.5; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3035, 2918, 2833, 1643, 1604, 1580, 1512, 859 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{15}H_{16}O_2$: 228.1150; found: 228.1144.

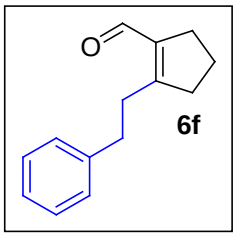


2-[2-(4-Fluoro-phenyl)-vinyl]-cyclopent-1-enecarbaldehyde (6d)-precursor to 8c: Prepared according to the general procedure discussed above with **4** (0.571 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv 4-fluorostyrenylboronic acid, R_F = 0.48, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with a 5% isocratic hold; isolated yield 0.050 g, 41%; brown oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.31 (s, 1H), 7.59 (d, *J* = 16.0 Hz, 1H), 7.50−7.46 (m, 2H), 7.11−7.04 (m, 2H), 6.82 (d, *J* = 16.0 Hz, 1H), 2.89 (br-t, *J* = 7.5 Hz, 2H), 2.71 (br-t, *J* = 7.5 Hz, 2H), 1.96 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.5, 164.2, 162.4, 157.6, 139.9, 135.5, 132.6 (d, *J* = 3.6 Hz), 129.0 (d, *J* = 8.2 Hz), 120.0 (d, *J* = 2.5 Hz), 116.1 (d, *J* = 22.0

Hz), 34.9, 31.4, 21.5; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3044, 2955, 2849, 1652, 1618, 1600, 1592, 1575, 1233, 1217 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{14}H_{13}FO$: 216.0950; found: 216.0950.

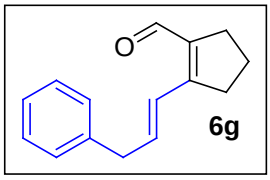


2-[2-(4-Trifluoromethyl-phenyl)-vinyl]-cyclopent-1-enecarbaldehyde (6e)-precursor to 8d: Prepared according to the general procedure discussed above with **4** (0.857 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv 4-trifluoromethylstyrenylboronic acid, R_F = 0.48, 20% MTBE:hexanes; purified using automated flash column chromatography with an MTBE:hexanes gradient mobile phase with a 5% isocratic hold; isolated yield 0.205 g, 90%; pale-orange solid; mp = 98.9−101.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.34 (s, 1H), 7.75 (d, *J* = 16.0 Hz, 1H), 7.63 (d, *J* = 8.5 Hz, 2H), 7.60 (d, *J* = 8.5 Hz, 2H), 6.86 (d, *J* = 16.0 Hz, 1H), 2.91 (t, *J* = 7.5 Hz, 2H), 2.74 (t, *J* = 7.5 Hz, 2H), 1.98 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.4, 156.6, 141.0, 139.7, 134.8, 130.5, 127.29, 127.2X (overlaps with 127.29), 125.8, 123.4, 34.7, 31.3, 21.3; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3051, 3854, 2926, 1639, 1617, 1589, 1322, 1167, 1120, 1066 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{15}H_3F_3O$: 266.0918; found: 266.0908.

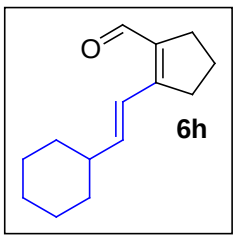


2-Phenethyl-cyclopent-1-enecarbaldehyde (6f)-precursor to 8f: Prepared according to the general procedure discussed above with **4** (0.571 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv potassium phenethyltrifluoroborate, R_F = 0.71, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with a 5% isocratic hold; isolated yield 0.075 g, 66%; orange liquid; ^{1}H NMR (500 MHz, $CDCl_3$):

δ 9.77 (s, 1H), 7.31−7.26 (m, 2H), 7.23−7.18 (m, 1H), 7.17−7.12 (m, 2H), 2.92−2.86 (m, 2H), 2.85−2.80 (m, 2H), 2.61 (br-t, *J* =7.6 Hz, 2H), 2.52 (br-t, *J* = 7.6 Hz, 2H), 1.85 (pent, *J* = 7.6 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 188.0, 164.9, 140.6, 139.1, 128.7, 128.4, 126.6, 38.6, 34.8, 30.6, 30.3, 21.6; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3027, 2951, 2855, 1661, 1627, 750, 700 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{14}H_{16}O$ [M-H]: 199.1117; found: 199.1127.

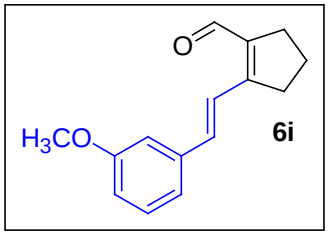


2-(3-Phenyl-propenyl)-cyclopent-1-enecarbaldehyde (6g)-precursor to 8g: Prepared according to the general procedure discussed above with **4** (0.571 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv 3-phenylpropenylboronic acid, R_F = 0.57, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with a 5% isocratic hold; isolated yield 0.0952 g, 79%; yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.17 (s, 1H), 7.33 (t, *J* = 7.5 Hz, 2H), 7.25−7.22 (m, 1H), 7.20 (d, *J* = 7.5 Hz, 2H), 7.02 (d, *J* = 15.5 Hz, 1H), 6.21 (dt, *J* = 15.5, 7.0 Hz, 1H), 3.57 (d, *J* = 7.0 Hz, 2H), 2.75 (t, *J* = 7.5 Hz, 2H), 2.64 (t, *J* = 7.5 Hz, 2H), 1.88 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.8, 158.1, 139.1, 138.7, 138.6, 128.81, 128.80, 126.7, 123.4, 39.8, 35.0, 30.9, 21.4; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3028, 2953, 2857, 1656, 1633, 751, 699 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{15}H_{16}O$: 212.1201; found: 212.1191.

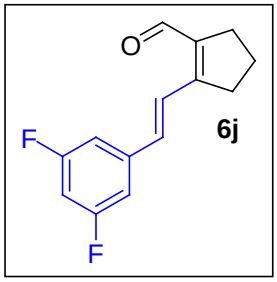


2-(2-Cyclohexyl-vinyl)-cyclopent-1-enecarbaldehyde (6h)-precursor to 8i: Prepared according to the general procedure discussed above with **4** (0.571 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv cyclohexylvinyl boronic acid, R_F = 0.70, 20% MTBE:hexanes; purified using

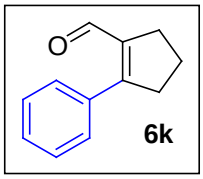
automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with a 2.5% isocratic hold; isolated yield 0.068 g, 58%; orange solid; mp = 46.0−49.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.19 (s, 1H), 6.94 (d, *J* = 16.0 Hz, 1H), 6.02 (dd, *J* = 16.0, 7.5 Hz, 1H), 2.75 (t, *J* = 7.5 Hz, 2H), 2.64 (br-t, *J* = 7.5 Hz, 2H), 2.20−2.12 (m, 1H), 1.88 (pent, *J* = 7.5 Hz, 2H), 1.81−1.73 (br-m, 4H), 1.73−1.66 (br-m, 2H), 1.37−1.11 (br-m, 4H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.9, 159.1, 146.3, 137.9, 120.0, 41.8, 35.1, 32.7, 30.9, 26.1, 26.0, 21.4; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 2924, 2851, 1658, 1630 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{14}H_{20}O$: 204.1514; found: 204.1505.



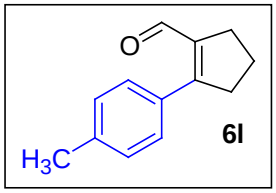
2-[2-(3-Methoxy-phenyl)-vinyl]-cyclopent-1-enecarbaldehyde (6i)-precursor to 8j: Prepared according to the general procedure discussed above with **4** (0.571): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv 3-methoxystyrenyl boronic acid pinacol ester, R_F = 0.39, 20% MTBE:hexanes; purified using automated flash column chromatography with an MTBE:hexanes gradient mobile phase with a 2.5% isocratic hold; isolated yield 0.044 g, 34%; orange solid; mp = 84.2−88.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.34 (s, 1H), 7.66 (d, *J* = 15.8 Hz, 1H), 7.31 (t, *J* = 8.0 Hz, 1H), 7.11 (br-d, *J* = 7.5 Hz, 1H), 7.04−7.02 (br-m, 1H), 6.89 (dd, *J* = 8.0, 2.5 Hz, 1H), 6.84 (d, *J* = 15.9 Hz, 1H), 3.87 (s, 3H), 2.91 (t, *J* = 7.5 Hz, 2H), 2.72 (t, *J* = 7.5 Hz, 2H), 1.97 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.6, 160.1, 157.7, 140.0, 137.8, 136.7, 130.0, 120.4, 120.0, 114.8, 112.6, 55.5, 34.9, 31.3, 21.4; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 2955, 2837, 1651, 1615, 1596, 1584, 779 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{15}H_{16}O_2$: 228.1150; found: 228.1140.



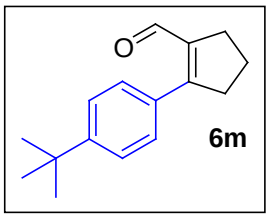
2-[2-(3,5-Difluoro-phenyl)-vinyl]-cyclopent-1-enecarbaldehyde (6j)-precursor to 8k: Prepared according to the general procedure discussed above with **4** (0.571 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv 3,5-difluorostyrenyl boronic acid pinacol ester, R_F = 0.58, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; yellow solid; isolated yield 0.081 g, 61%; yellow solid; mp = 106.3−108.5 °C; ^{1}H NMR (500 MHz, CD_3OD): δ 10.33 (s, 1H), 7.91 (d, *J* = 16.0 Hz, 1H), 7.29−7.23 (m, 2H), 6.94 (d, *J* = 16.0 Hz, 1H), 6.92−6.87 (m, 1H), 2.94 (t, *J* = 7.5 Hz, 2H), 2.67 (t, *J* = 7.5 Hz, 2H), 1.97 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 187.5, 163.5 (dd, *J* = 250, 13 Hz), 156.3, 141.4, 139.8 (t, *J* = 10.9 Hz), 134.2 (t, *J* = 3.0 Hz), 122.6 (t, *J* = 3.1 Hz), 109.9 (dd, *J* = 19.5, 6.5 Hz), 104.2 (t, *J* = 25.5 Hz), 34.8, 31.5, 21.4; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3092, 2959, 2854, 1646, 1623, 1593, 905, 726 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{14}H_{12}F_2O$: 234.0856; found: 234.0861.



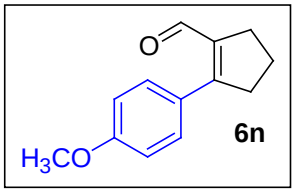
2-Phenyl-cyclopent-1-enecarbaldehyde (6k)-precursor to 8l: Prepared according to the general procedure discussed above with **4** (0.571 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv phenylboronic acid; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.146 g, 95%; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.82 (s, 1H), 7.41−7.38 (m, 3H), 7.36−7.33 (m, 2H), 3.01−2.96 (m, 2H), 2.78−2.73 (m, 2H), 2.01 (pent, *J* = 7.5 Hz, 2H).



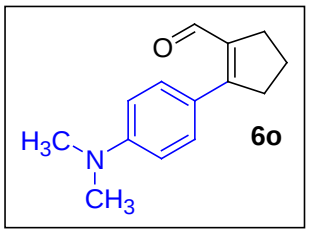
2-*p*-Tolyl-cyclopent-1-enecarbaldehyde (6l)-precursor to 8m: Prepared according to the general procedure discussed above with **4** (0.857 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv potassium 4-methylphenyltrifluoroborate, R_F = 0.68, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.106 g, 66%; orange oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.85 (s, 1H), 7.28−7.26 (br-m, 2H), 7.24−7.20 (br-m, 2H), 3.01−2.96 (m, 2H), 2.78−2.72 (m, 2H), 2.39 (s, 3H), 2.01 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.7, 162.7, 139.6, 139.2, 132.2, 129.3, 128.7, 39.7, 31.2, 21.8, 21.4; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3028, 2954, 2852, 1658, 1607, 817 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{13}H_{14}O$: 186.1045; found: 186.1045.



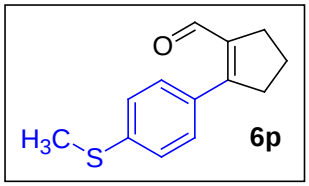
2-(4-*tert*-Butyl-phenyl)-cyclopent-1-enecarbaldehyde (6m)-precursor to 8n: Prepared according to the general procedure discussed above with **4** (0.857 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv potassium 4-*tert*-butylphenyl trifluoroborate, R_F = 0.70, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.142 g, 73%; yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.86 (s, 1H), 7.45−7.41 (m, 2H), 7.34−7.29 (m, 2H), 3.02−2.97 (m, 2H), 2.79−2.73 (m, 2H), 2.01 (pent, *J* = 7.5 Hz, 2H), 1.36 (s, 9H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.7, 162.6, 152.8, 139.3, 132.2, 128.6, 125.6, 39.7, 34.9, 31.4, 31.3, 21.8; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3034, 2960, 2905, 2868, 1659, 1607, 1508, 1233, 833 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{16}H_{20}O$: 228.1514; found: 228.1514.



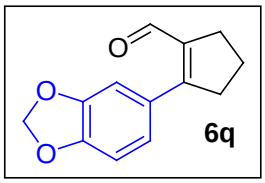
2-(4-Methoxy-phenyl)-cyclopent-1-enecarbaldehyde (6n)-precursor to 8o: Prepared according to the general procedure discussed above with **4** (0.857): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv of potassium 4-methoxyphenyltrifluoroborate, R_F = 0.54, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.153 g, 88%; yellow film; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.85 (s, 1H), 7.40−7.30 (m, 2H), 6.95−6.91 (m, 2H), 3.84 (s, 3H), 2.99−2.93 (m, 2H), 1.99 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.6, 162.4, 160.7, 138.6, 130.3, 122.5, 114.1, 55.5, 39.6, 31.3, 21.7; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 2954, 2837, 1653, 1603, 1509, 1252, 1177, 832 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{13}H_{14}O_2$: 202.0994; found: 202.0996.



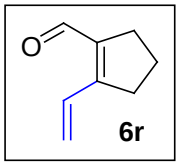
2-(4-Dimethylamino-phenyl)-cyclopent-1-enecarbaldehyde (6o)-precursor to 8p: Prepared according to the general procedure discussed above with **4** (0.857 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv of potassium 4-*N*,*N*-dimethylaminophenyl trifluoroborate, R_F = 0.46, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.096 g, 52%; yellow-orange solid; mp = 111.0−113.6 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.89 (s, 1H), 7.33−7.28 (m, 2H), 6.73−6.69 (m, 2H), 3.02 (s, 6H), 2.99−2.94 (m, 2H), 2.77−2.72 (m, 2H), 1.97 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.7, 163.0, 151.3, 137.0, 130.4, 122.7, 111.7, 40.3, 39.2, 31.3, 21.8; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 2967, 2933, 2830, 1637, 1608, 1585, 1235, 824 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{14}H_{17}NO$: 215.1310; found: 215.1309.



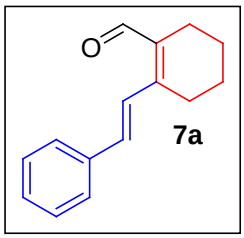
2-(4-Methylsulfanyl-phenyl)-cyclopent-1-enecarbaldehyde (6p)-precursor to 8r: Prepared according to the general procedure discussed above with **4** (0.857 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv of potassium 4-methylsulfanylphenyl trifluoroborate, R_F = 0.54, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.135 g, 72%; yellow solid; mp = 51.2−52.3 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.84 (s, 1H), 7.30−7.25 (m, 4H), 3.00−2.94 (m, 2H), 2.78−2.73 (m, 2H), 2.51 (s, 3H), 2.00 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.3, 161.9, 140.8, 139.4, 131.5, 129.2, 126.0, 39.6, 31.3, 21.8, 15.5; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 2954, 2921, 2833, 1656, 1592, 1096, 819 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{13}H_{14}OS$: 218.0765; found: 218.0759.



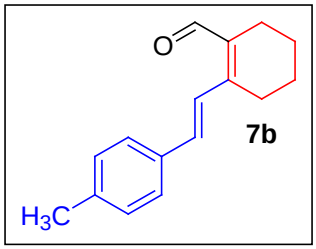
2-Benzo[1,3]dioxol-5-yl-cyclopent-1-enecarbaldehyde (6q)-precursor to 8s: Prepared according to the general procedure discussed above with **4** (0.89 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv of potassium 2-benzo[1,3]dioxol-5-yltrifluoroborate, R_F = 0.57, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.108 g, 56%; pale-yellow solid; mp = 84.9−86.7 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.85 (s, 1H), 6.89−6.83 (m, 3H), 6.02 (s, 2H), 2.96−2.92 (m, 2H), 2.76−2.72 (m, 2H), 1.99 (pent, *J* = 7.5 Hz, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 190.4, 162.0, 148.8, 148.1, 139.2, 129.0, 123.3, 108.9, 108.5, 101.6, 39.8, 31.3, 21.7; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 2960, 2904, 2850, 1652, 1598, 1503, 1487, 1241, 1037 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{13}H_{12}O_3$: 216.0786; found: 216.0783.



2-Vinyl-cyclopent-1-enecarbaldehyde (6r)-precursor to 8u: Prepared according to the general procedure discussed above with **4** (0.86 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv of potassium vinyl trifluoroborates; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.094 g, 90%; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.20 (s, 1H), 7.26 (dd, *J* = 17.0, 10.8 Hz, 1H), 5.55 (d, *J* = 17.0 Hz, 1H), 5.54 (d, *J* = 10.8 Hz, 1H), 2.77 (br-t, *J* = 7.5 Hz, 2H), 2.66 (br-t, *J* = 7.5 Hz, 2H), 1.91 (pent, *J* = 7.5 Hz, 2H). *This compound is extremely unstable at ambient temperature outside of solution. Spectral data was acquired with residual solvent from purification to prevent compound degradation and is consistent with literature values.*[4]

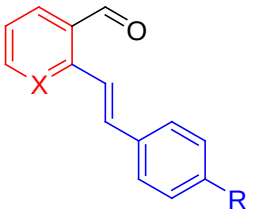


2-Styryl-cyclohex-1-enecarbaldehyde (7a)-precursor to 9a: Prepared according to the general procedure discussed above with **5** (0.747 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv potassium styrenyltrifluoroborate, R_F = 0.72, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 5.0% isocratic hold; isolated yield 0.134 g, 85%; amorphous; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.48 (s, 1H), 7.76 (d, *J* = 16.0 Hz, 1H), 7.49−7.46 (m, 2H), 7.40−7.34 (m, 2H), 7.33−7.28 (m, 1H), 6.87 (d, *J* = 16.0 Hz, 1H), 2.60−2.54 (br-m, 2H), 2.39−2.34 (br-m, 2H), 1.77−1.70 (m, 2H), 1.69−1.63 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ =190.6, 151.8, 136.8, 135.9, 133.6, 129.0, 128.8, 127.1, 123.5, 27.7, 23.4, 22.1, 21.7; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3056, 3024, 2932, 2861, 1658, 1614, 1583, 1148, 749, 691 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{15}H_{16}O$: 212.1201; found: 212.1196.

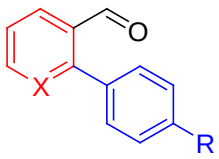


2-(2-*p*-Tolyl-vinyl)-cyclohex-1-enecarbaldehyde (7b)-precursor to 9b: Prepared according to the general procedure discussed above with **5** (0.747): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv of 4-methylstyrenylboronic acid, R_F = 0.72, 20% MTBE:hexanes; purified using automated flash column chromatography using an MTBE:hexanes gradient mobile phase employing a 2.5% isocratic hold; isolated yield 0.104 g, 62%; pale-yellow solid; mp = 82.3−84.6 °C; ^{1}H NMR (500 MHz, CD_3OD): δ 10.41 (s, 1H), 7.84 (d, *J* = 16.0 Hz, 1H), 7.44 (d, *J* = 8.2 Hz, 2H), 7.17 (d, *J* = 8.2 Hz, 2H), 6.92 (d, *J* = 16.0 Hz, 1H), 2.62−2.56 (br-m, 2H), 2.33 (s, 3H), 2.32−2.27 (br-m, 2H), 1.75−1.69 (m, 2H), 1.67−1.61 (m, 2H); ^{13}C NMR (125 MHz, CD_3OD): δ 192.5, 154.4, 139.9, 136.1, 135.3, 135.1, 130.5, 128.2, 123.2, 28.4, 24.2, 23.1, 22.7, 21.3; IR (ATR-CD_3OD): $\bar{\upsilon}_{max}$ = 3014, 2868, 2925, 1644, 1614, 1602, 1578, 1150, 960, 804 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{16}H_{18}O$: 226.1358; found: 226.1353.

Table S2. Pyridine and benzene core aldehyde synthons for analogue preparation.

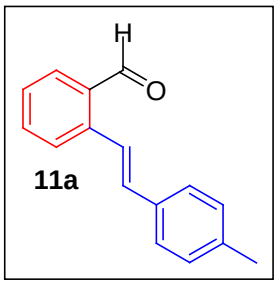


11a X = C; R = CH_3
11b X = C; R = OCH_3
12a X = N; R = H
12b X = N; R = CH_3
12c X = N; R = OCH_3

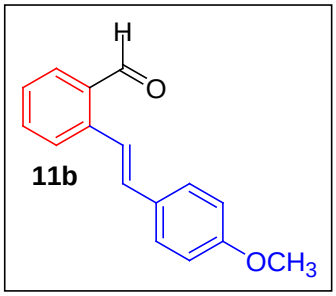


11c X = C; R = OCH_3
12d X = N; R = H
12e X = N; R = CH_3
12f X = N; R = OCH_3

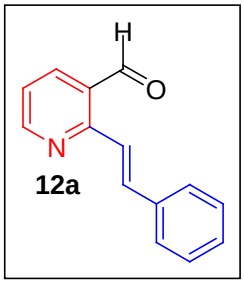
Compounds **11a** and **11b** are known in the primary literature.[3]



2-(2-*p*-Tolyl-vinyl)-benzaldehyde (11a)-precursor to 13a: Prepared according to the general procedure discussed above with 2-bromo-benzaldehyde (0.81 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv potassium 4-methoxyphenyltrifluroborate, R_F = 0.78, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with an isocratic hold at 20%; isolated yield 0.108 g, 60%; yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.34 (s, 1H), 8.00 (d, *J* = 16.0 Hz, 1H), 7.84 (dd, *J* = 7.8, 1.0 Hz, 1H), 7.72 (d, *J* = 7.8 Hz, 1H), 7.58 (t, *J* = 7.8 Hz, 1H), 7.47 (d, *J* = 8.0 Hz, 2H), 7.42 (t, *J* = 7.5 Hz, 1H), 7.20 (d, *J* = 8.0 Hz, 2H), 7.04 (d, *J* = 16.0 Hz, 1H), 2.38 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.8, 140.4, 138.5, 134.3, 134.2, 133.8, 133.0, 132.3, 129.6, 127.5, 127.2, 127.0, 123.7, 21.4; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3022, 2919, 2855, 2733, 1689, 1628, 1564, 1594, 1513, 1478, 1449, 1182, 963, 803, 754, 726, 535 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{16}H_{14}O$: 222.1045; found: 222.1043.

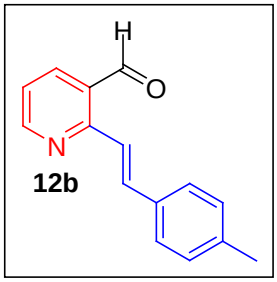


2-[2-(4-Methoxy-phenyl)-vinyl]-benzaldehyde (11b)-precursor to 13b: : Prepared according to the general procedure discussed above with 2-bromo-benzaldehyde (0.81 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv 4-methoxyphenylboronic acid, R_F = 0.57, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with an isocratic hold at 10%; isolated yield 0.104 g, 54%; amorphous; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.33 (s, 1H), 7.91 (d, *J* = 16.3 Hz, 1H), 7.82 (dd, *J* = 7.8, 1.3 Hz, 1H), 7.70 (d, *J* = 8.0 Hz, 1H), 7.57 (dt, *J* = 7.8, 1.3 Hz, 1H), 7.53−7.48 (m, 2H), 7.41 (dt, *J* = 7.8, 1.3 Hz, 1H), 7.01 (d, *J* = 16.3 Hz, 1H), 6.94−6.90 (m, 2H), 3.84 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.8, 159.9, 140.4, 133.8, 133.7, 132.8, 132.3, 129.8, 128.4, 127.3, 127.0, 122.3, 114.3, 55.4; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3035, 2934, 2836, 2740, 1693, 1605, 1594, 1511, 1250, 1175, 1032, 820 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{16}H_{14}O_2$: 238.0994; found: 238.0995.

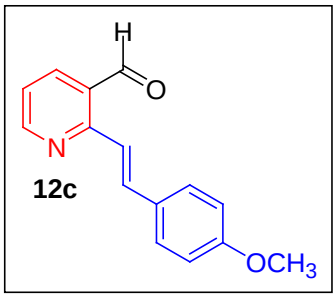


2-Styryl-pyridine-3-carbaldehyde (12a)-precursor to 14a: Prepared according to the general procedure discussed above with 2-bromo-pyridine-3-carbaldehyde (0.81 mmol): 2.5 mol % $Pd(OAc)_2$, 5.0 mol % RuPhos, and 1.20 equiv potassium styryltrifluoroborate, R_F = 0.26, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase; isolated yield 0.138 g, 82%; yellow solid; mp = 59.3−63.1 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.40 (s, 1H), 8.78 (dd, *J* = 4.8, 1.8 Hz, 1H), 8.11 (dd, *J* = 7.8, 1.8 Hz, 1H), 8.10 (d, *J* = 15.6 Hz, 1H), 7.97 (d, *J* = 15.6 Hz, 1H), 7.68−7.64 (m, 2H), 7.43−7.38 (m, 2H), 7.37−7.32 (m, 2H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.4, 156.2, 153.4, 139.8, 138.6, 136.4, 129.4, 129.0,

127.96, 127.93, 122.5, 122.3; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3058, 2862, 2817, 2743, 1700, 1629, 1577, 1551, 1450, 1435, 1398, 772, 690 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{14}H_{11}NO$: 209.0841; found: 209.0833.

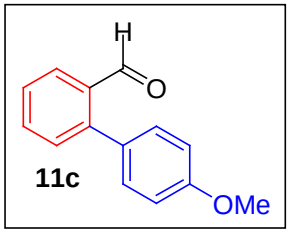


2-(2-*p*-Tolyl-vinyl)-pyridine-3-carbaldehyde (12b)-precursor to 14b: Prepared according to the general procedure discussed above with 2-bromo-pyridine-3-carbaldehyde (0.81 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv trans-2-(4-methylphenyl)vinylboronic acid, R_F = 0.26, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase; isolated yield 0.041 g, 31%; pale-yellow solid; mp = 80.0−82.2 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.41 (s, 1H), 8.77 (dd, *J* = 4.7, 1.9 Hz, 1H), 8.11 (dd, *J* = 7.8, 1.9 Hz, 1H), 8.05 (d, *J* = 15.6 Hz, 1H), 7.95 (d, *J* = 15.6 Hz, 1H), 7.56 (d, *J* = 8.0 Hz, 2H), 7.32 (dd, *J* = 7.8, 4.7 Hz, 1H), 7.21 (d, *J* = 8.0 Hz, 2H), 2.39 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.5, 156.6, 153.6, 139.5, 138.3, 138.3X (overlaps with 138.3), 133.7, 129.7, 127.9, 127.8, 122.1, 121.7, 21.6; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3023, 2857, 2734, 1691, 1627, 1553, 1437, 811 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{15}H_{13}NO$: 223.0997; found: 223.0999.

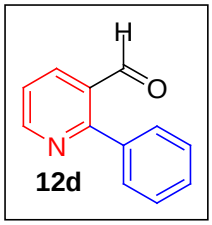


2-[2-(4-Methoxy-phenyl)-vinyl]-pyridine-3-carbaldehyde (12c)-precursor to 14c: Prepared according to the general procedure discussed above with 2-bromo-pyridine-3-carbaldehyde (0.54 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv trans-2-(4-methoxyphenyl)vinylboronic acid, R_F = 0.14, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient

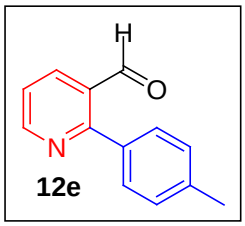
mobile phase; isolated yield 0.101 g, 78%; pale-yellow solid; mp = 64.1−66.0 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.39 (s, 1H), 8.75 (dd, *J* = 4.7, 1.8 Hz, 1H), 8.09 (dd, *J* = 7.8, 1.8 Hz, 1H), 7.99 (d, *J* = 15.6 Hz, 1H), 7.93 (d, *J* = 15.6 Hz, 1H), 7.64−7.58 (m, 2H), 7.30 (dd, *J* = 7.8, 4.7 Hz, 1H), 6.96−6.91 (m, 2H), 3.86 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.6, 160.7, 156.7, 153.6, 139.7, 138.0, 129.4, 129.3, 127.6, 121.8, 120.4, 114.4, 55.5; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 2934, 2837, 2739, 1692, 1626, 1552, 1510, 1252, 1173, 826 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{15}H_{13}NO_2$: 239.0946; found: 239.0954.



4'-Methoxy-biphenyl-2-carbaldehyde (11c)-precursor to (13c): Prepared according to the general procedure discussed above with 2-bromo-benzaldehyde (0.81 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv potassium 4-methoxyphenyl trifluoroborate, R_F = 0.44, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase; isolated yield 0.150 g, 87%; yellow solid; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.00 (s, 1H), 8.00 (dd, *J* = 7.5, 1.4 Hz, 1H), 7.62 (dt, *J* = 7.5, 1.4 Hz, 1H), 7.47−7.72 (m, 2H), 7.36−7.29 (m, 2H), 7.05−6.97 (m, 2H), 3.88 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.8, 159.9, 145.8, 133.9, 133.7, 131.5, 130.9, 130.2, 127.8, 127.5, 114.1, 55.6; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3035, 3961, 3838, 2755, 1691, 1610, 1597, 1515, 1474, 1247, 1179, 836, 766 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{14}H_{12}O_2$: 212.0837; found: 212.0828. *Spectral data acquired was consistent with literature values.*[5]

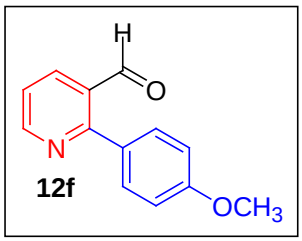


2-Phenyl-pyridine-3-carbaldehyde (12d)-precursor to 14d: Prepared according to the general procedure discussed above with 2-bromo-pyridine-3-carbaldehyde (0.81 mmol): 2.5 mol % $Pd(OAc)_2$, 5.0 mol % RuPhos, and 1.20 equiv potassium phenyltrifluoroborate, R_F = 0.28, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase; isolated yield 0.102 g, 68%; yellow oil; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.07 (d, *J* = 0.8 Hz, 1H), 8.89 (dd, *J* = 4.8, 1.8 Hz, 1H), 8.32 (dd, *J* = 7.9, 1.8 Hz, 1H), 7.63−7.58 (m, 2H), 7.57−7.51 (m, 3H), 7.45 (ddd, *J* = 7.9, 4.8, 1.8 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 191.8, 162.4, 153.6, 137.2, 136.1, 130.5, 129.8, 129.7, 128.8, 122.7; IR (ATR- $CDCl_3$): $\bar{\upsilon}_{max}$ = 3044, 2925, 2858, 2748, 1695, 1615, 1580, 1557, 1433, 1386, 1247, 828, 799, 770 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{12}H_9NO$: 183.0684; found: 183.0836.



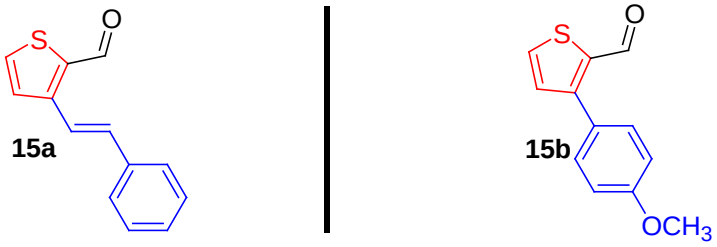
2-*p*-Tolyl-pyridine-3-carbaldehyde (12e)-precursor to 14e: Prepared according to the general procedure discussed above with 2-bromo-pyridine-3-carbaldehyde (0.81 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, 1.20 equiv potassium 4-methylphenyltrifluoroborate, R_F = 0.31, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with an isocratic hold at 25%; isolated yield 0.067 g, 43%; pale-yellow solid; mp = 78.4−81.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.06 (d, *J* = 0.8 Hz, 1H), 8.87 (dd, *J* = 4.8, 1.8 Hz, 1H), 8.29 (dd, *J* = 8.0, 1.8 Hz, 1H), 7.51−7.47 (m, 2H), 7.42 (ddd, *J* = 8.0, 4.8, 0.8 Hz, 1H), 7.35−7.32 (m, 2H), 2.45 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.1, 162.5, 153.6, 140.0, 136.0, 134.4, 130.6, 129.6, 129.5, 122.4, 21.5; IR (ATR-$CDCl_3$): $\bar{\upsilon}_{max}$ = 3040,

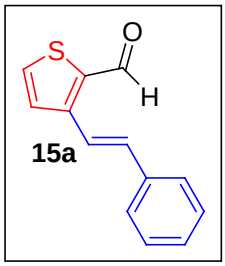
2920, 2858, 2748, 1695, 1580, 1433, 1386, 1247, 828, 799, 770 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{13}H_{11}NO$: 197.0841; found: 197.0836.



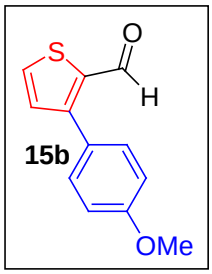
2-(4-Methoxy-phenyl)-pyridine-3-carbaldehyde (12f)-precursor to 14f: Prepared according to the general procedure discussed above with 2-bromo-pyridine-3-carbaldehyde (0.81 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.20 equiv potassium 4-methoxyphenyltrifluoroborate, R_F = 0.18, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase; isolated yield 0.118 g, 69%; white solid; mp = 67.0−68.4 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.06 (d, *J* = 0.8 Hz, 1H), 8.85 (dd, *J* = 4.7, 1.9 Hz, 1H), 8.28 (dd, *J* = 7.9, 1.9 Hz, 1H), 7.57−7.54 (m, 2H), 7.39 (ddd, *J* = 7.9, 4.7, 0.8 Hz, 1H), 7.07−7.03 (m, 2H), 3.91 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 192.1, 162.0, 161.1, 153.6, 136.1, 132.1, 129.7, 129.4, 122.2, 114.3, 56.6; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3056, 2970, 2840, 2747, 1695, 1681, 1607, 1580, 1515, 1432, 1387, 1251, 1176, 840, 776, 730 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{13}H_{11}NO_2$: 213.0790; found: 213.0797.

Table S3. Thiophene core aldehyde synthons for analogue preparation.





3-Styryl-thiophene-2-carbaldehyde (15a)-precursor to 16a: Prepared according to the general procedure discussed above with 3-bromo-thiophene-2-carbaldehyde (0.79 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv potassium styrenyltrifluoroborate, R_F = 0.60, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase; isolated yield 0.068 g, 41%; light-brown solid; mp = 104.1−106.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 10.22 (s, 1H), 7.69 (d, *J* = 16.3 Hz, 1H), 7.68 (d, *J* = 5.0 Hz, 1H), 7.56−7.52 (m, 2H), 7.46 (d, *J* = 5.0 Hz, 1H), 7.42−7.37 (m, 2H), 7.35−7.31 (m, 1H), 7.18 (d, *J* = 16.3 Hz, 1H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 182.8, 146.9, 137.7, 136.4, 134.9, 134.5, 129.1, 129.0, 127.1, 126.9, 119.5; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 3101, 3077, 3023, 2833, 1652, 1626, 1433, 1196, 956, 758, 692 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{13}H_{10}OS$: 214.0452; found: 214.0452.



3-(4-Methoxy-phenyl)-thiophene-2-carbaldehyde (15b)-precursor to 16b: Prepared according to the general procedure discussed above with 3-bromo-thiophene-2-carbaldehyde (0.79 mmol): 5.0 mol % $Pd(OAc)_2$, 10.0 mol % RuPhos, and 1.05 equiv potassium 4-methoxyphenyltrifluroborate, R_F = 0.37, 20% MTBE:hexanes; purified using automated flash column chromatography employing an MTBE:hexanes gradient mobile phase with an isocratic hold at 25%; isolated yield 0.039 g, 23%; light-brown solid; mp = 65.7−68.5 °C; ^{1}H NMR (500 MHz, $CDCl_3$): δ 9.88 (s, 1H), 7.73−7.71 (m, 1H), 7.44−7.39 (m, 2H), 7.20 (d, *J* = 4.9 Hz, 1H), 7.04−6.98 (m, 2H), 3.87 (s, 3H); ^{13}C NMR (125 MHz, $CDCl_3$): δ 184.5, 160.4, 151.5, 138.0, 134.2, 131.0, 130.7, 126.6, 114.5, 55.6; IR (ATR-$CDCl_3$): $\tilde{\upsilon}_{max}$ = 2970, 2838, 1654, 1607, 1500, 1250, 1178, 1029, 754 cm^{-1}; HRMS (EI): *m/z* calculated for $C_{12}H_{10}O_2S$: 218.0402; found: 218.0410.

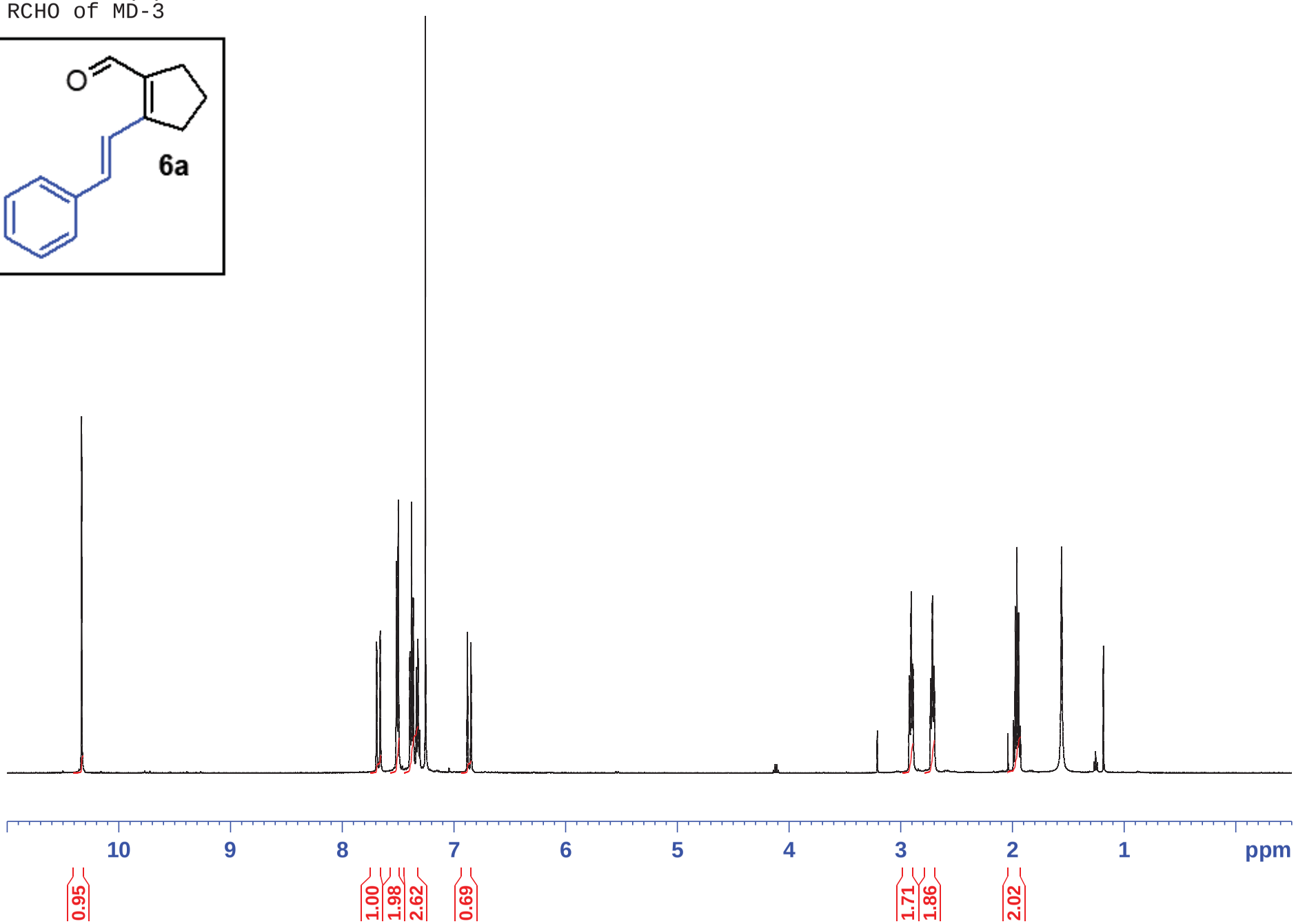
References

[1] For compound **6a** see: Knobloch, K.; Koch, J.; Keller, M.; Eberbach, W. The Dipolar Route to Azepin-3-one Derivatives by Heterocyclization of Linear and Monocyclic Enallenyl Nitrones as the Key Step. *European Journal of Organic Chemistry* **2005**, *13*, 2715−2733. For compound **6k** see: Vogel, C.; Schnippenkoetter, B.; Jones, P. G.; Bubenitschek, P. Cycloalkano-2H-pyrrole as Stable Intermediate in the Rearrangement of β-cycloalkenyl-α-azidoacry Esters to Cycloalkano-1H-pyrrole. *Angewandte Chemie, International Edition* **1993**, *32*, 1051−1052. For compound **6r** see: Yoshida, K.; Takahashi, H.; Imamoto, T. Synthesis of Substituted Benzenes and Phenols by Ring-Closing Olefin Metathesis. *Chemistry-A European Journal* **2008**, *14*, 8246−8261.

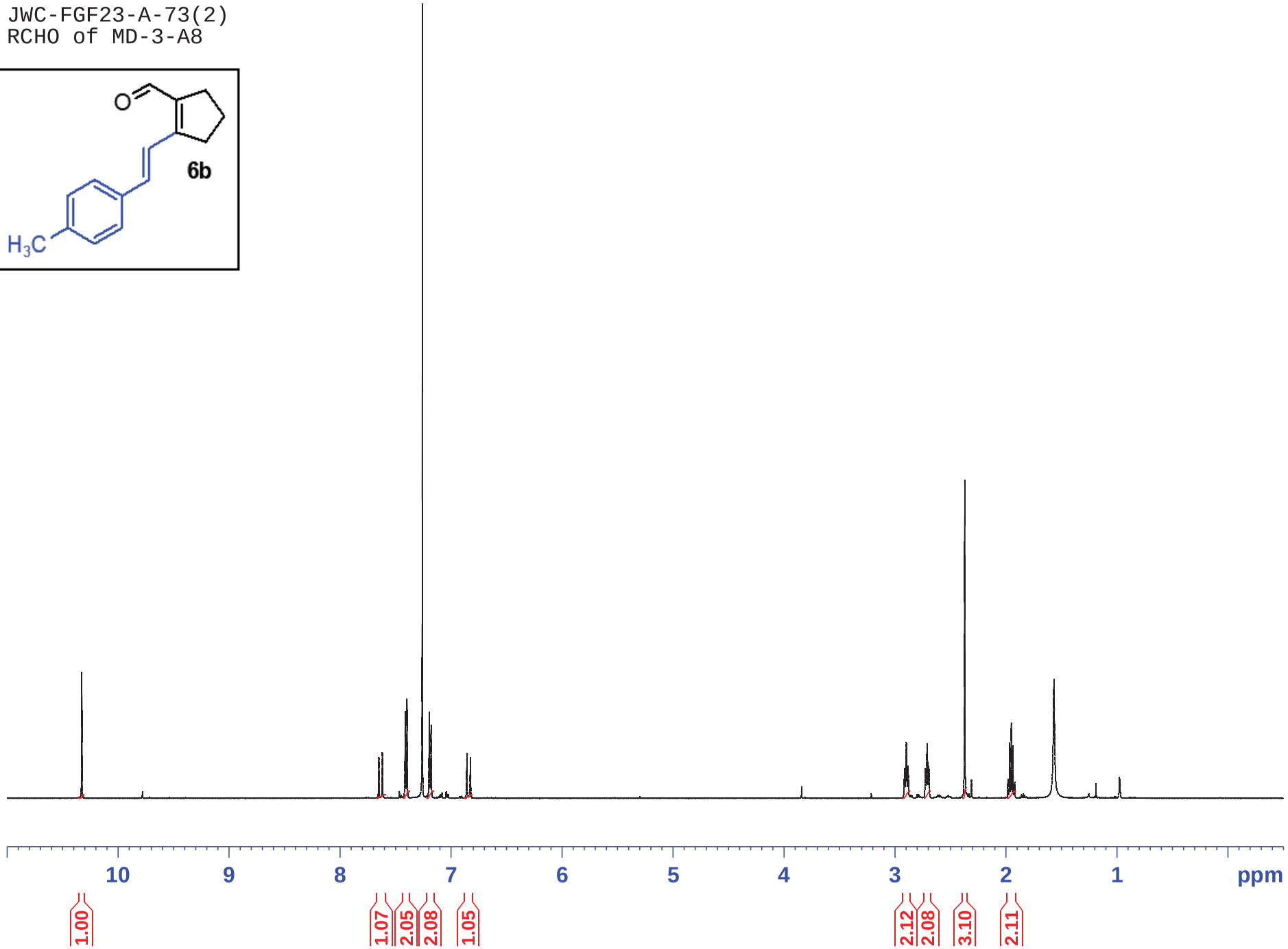
[2] Xiao, Z. S.; Riccardi, D.; Velazquez, H. A.; Chin, A.-L.; Yates, C. R.; Carrick, J. D.; Smith, J. C.; Baudry, J.; Quarles, L. D. Computationally Identified Novel Chemical Compounds that Antagonize FGF-23. *Science Signaling* **2016**, *9(455)*, ra113-*Cover Story*.

[3] For compound **11a** see: Nallasivam, J. L.; Fernandes, R. A. Development of Unimolecular Tetrakis(piperidin-4-ol) as a Ligand for Suzuki-Miyaura Cross-Coupling Reactions: Synthesis of Incrustoporin and Preclamol. *European Journal of Organic Chemistry* **2015**, *16*, 3558−3567. For compound **11b** see: Discovery of Aryl-biphenyl-2-ylmethylpiperazines as Novel Scaffolds for 5-HT7 Ligands and Role of the Aromatic Substitutents Binding to the Target Receptor. *Biorganic and Medicinal Chemistry* **2013**, *21*, 2568−2676.

JWC-B-151(2)
RCHO of MD-3

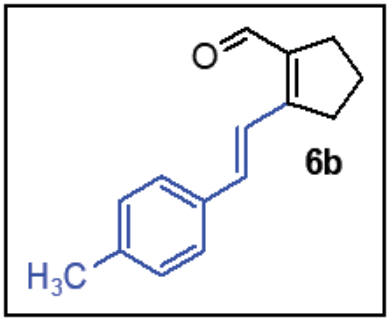


JWC-FGF23-A-73(2)
RCHO of MD-3-A8



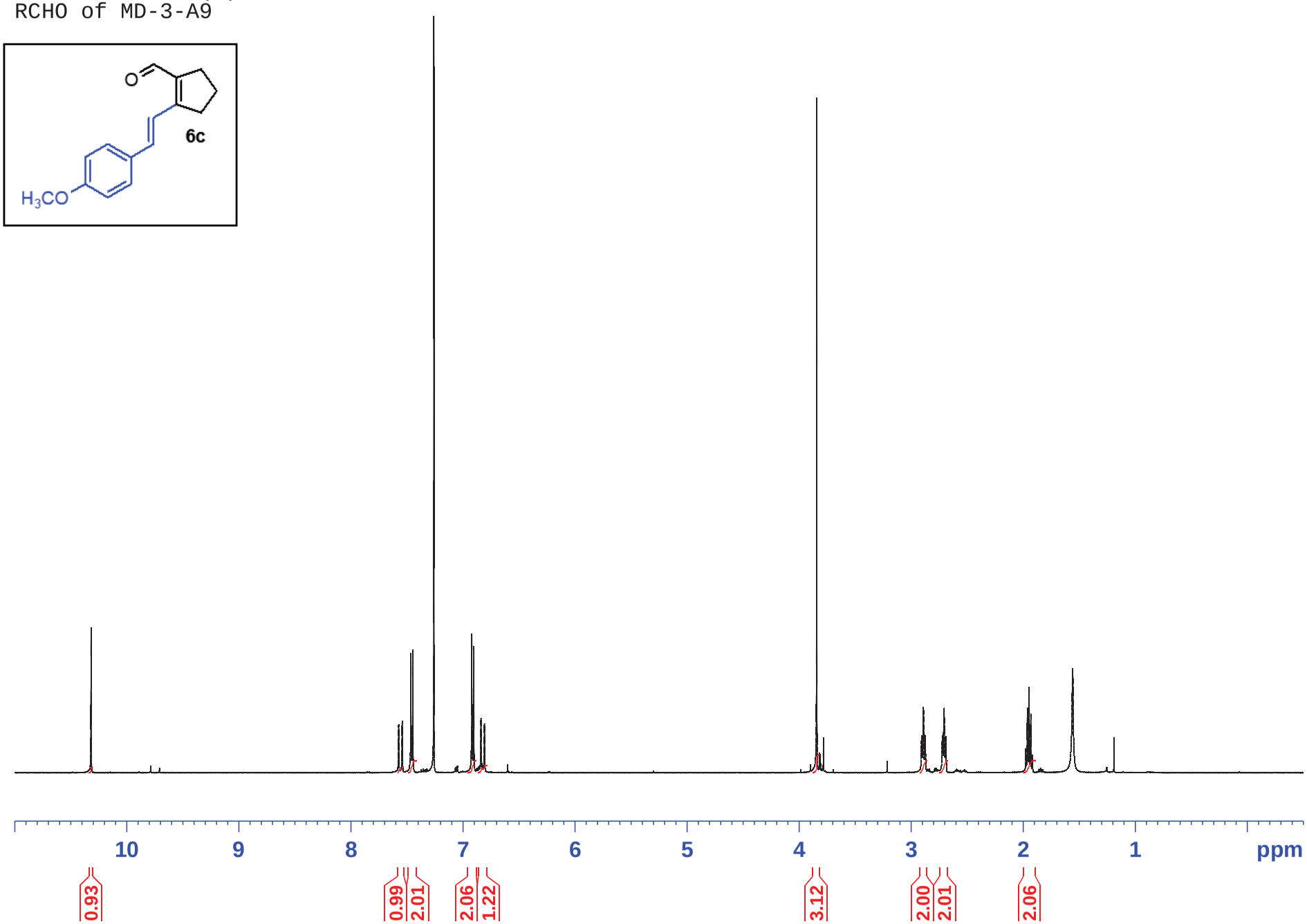
JWC-FGF23-A-73(2)
RCHO of MD-3-A8

187.63
158.20
139.45
139.40
136.83
133.64
129.78
127.30
119.15
34.91
31.24
21.53
21.48



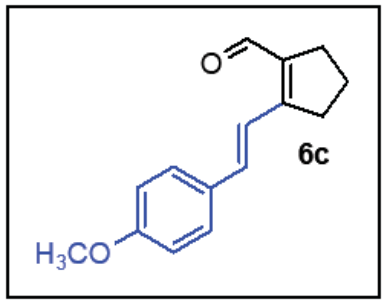
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

JWC-FGF23-A-79(2)
RCHO of MD-3-A9



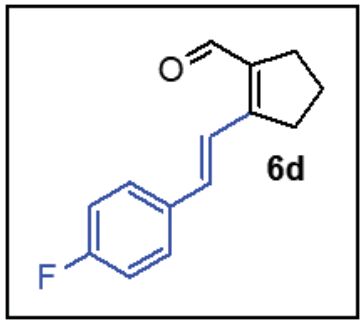
JWC-FGF23-A-75(2)
RCHO of MD-3-A19

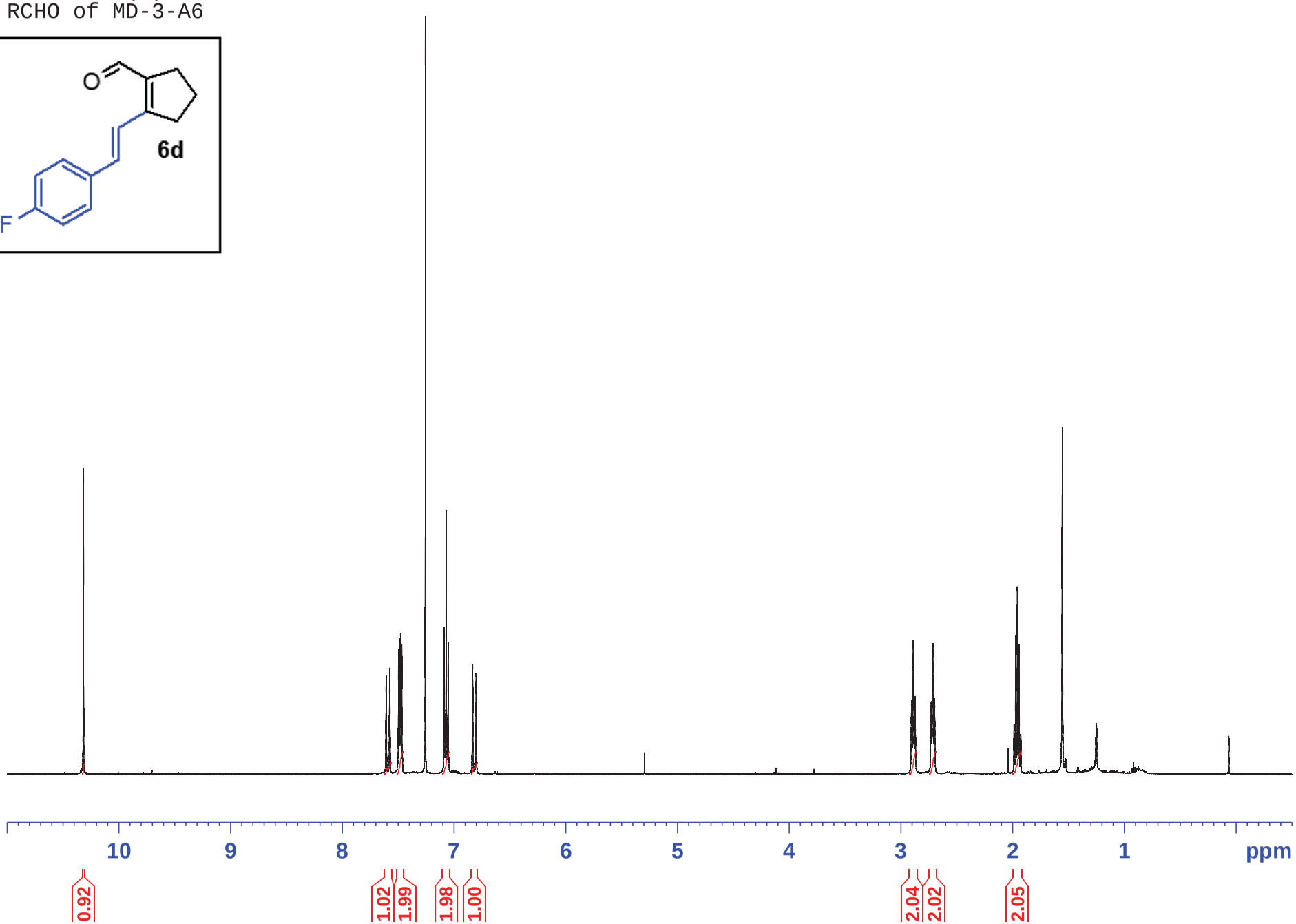
187.57
160.58
158.42
138.87
136.48
129.15
128.79
118.01
114.51
55.52
34.92
31.20
21.50



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

JWC-B-155(4)
RCHO of MD-3-A6





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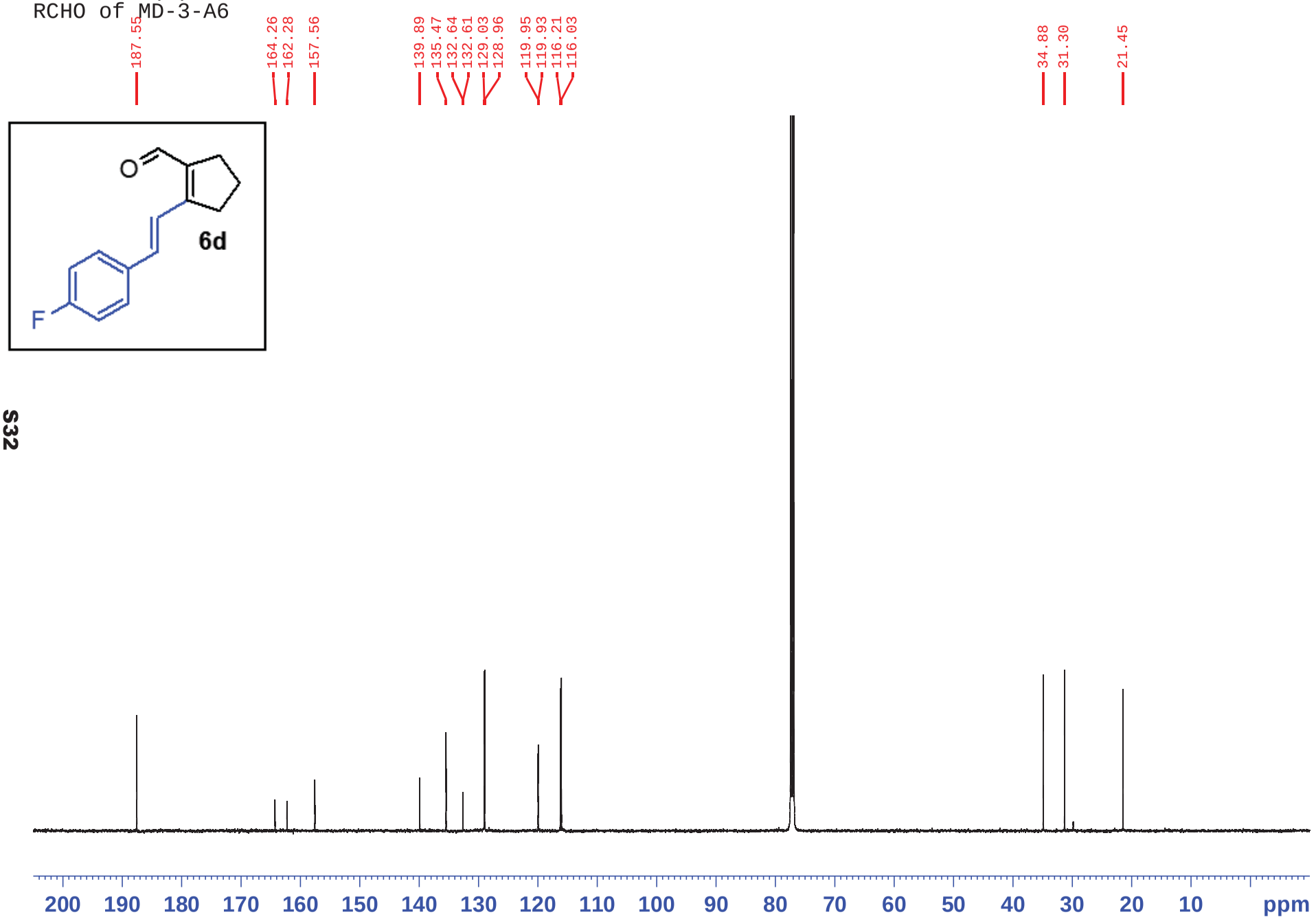
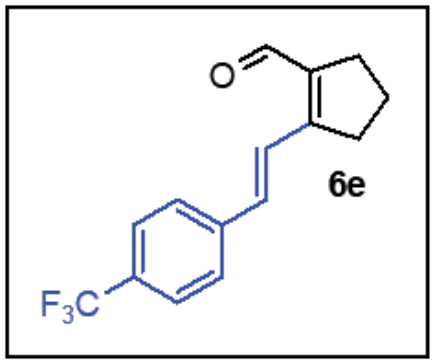
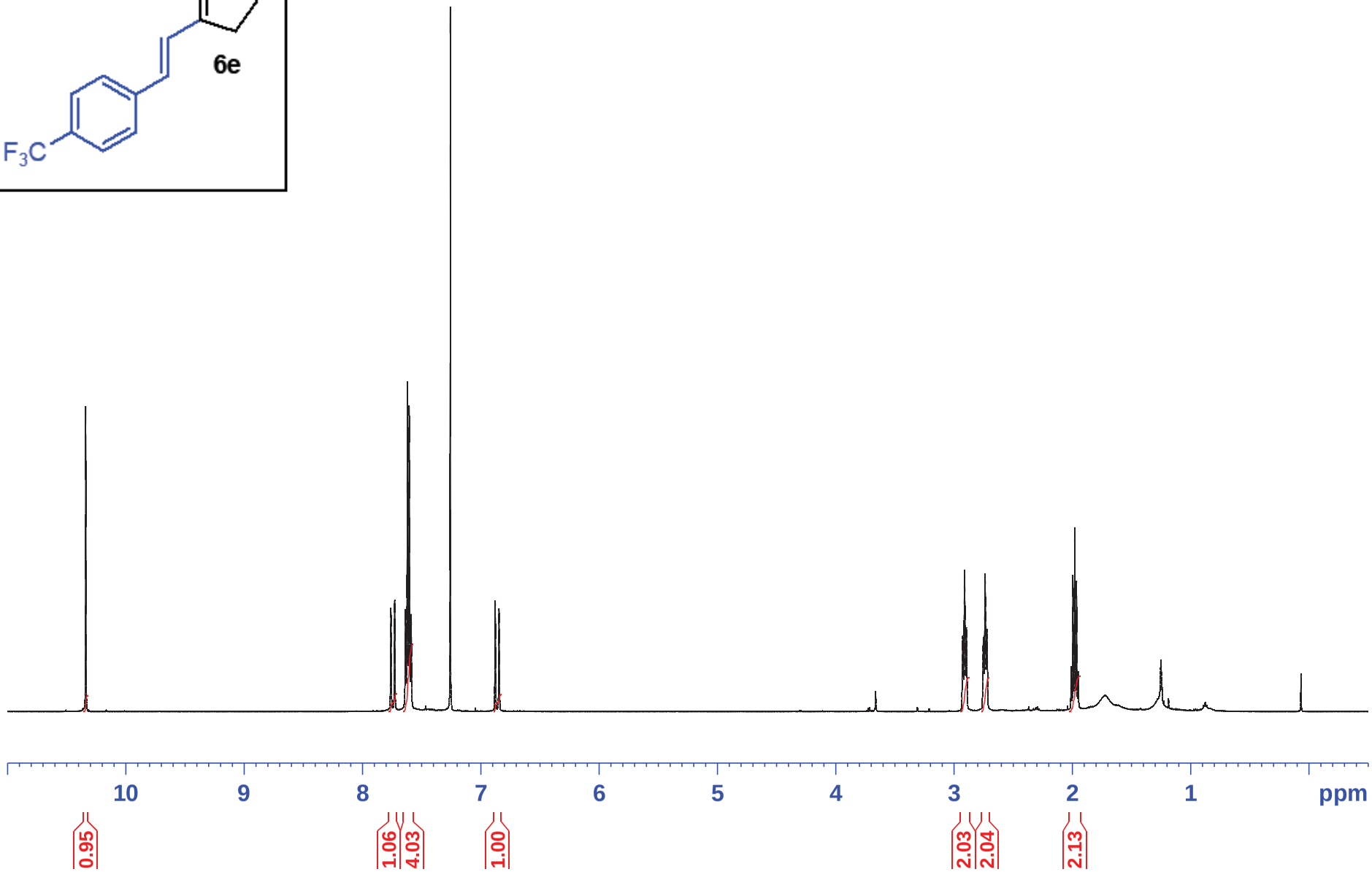
RCHO of MD-3-A6



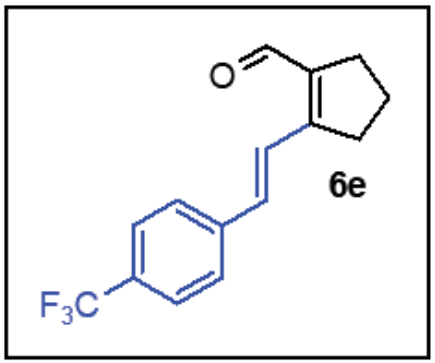
RPD-FGF23-A-17(2)
RCHO of MD-3-A7

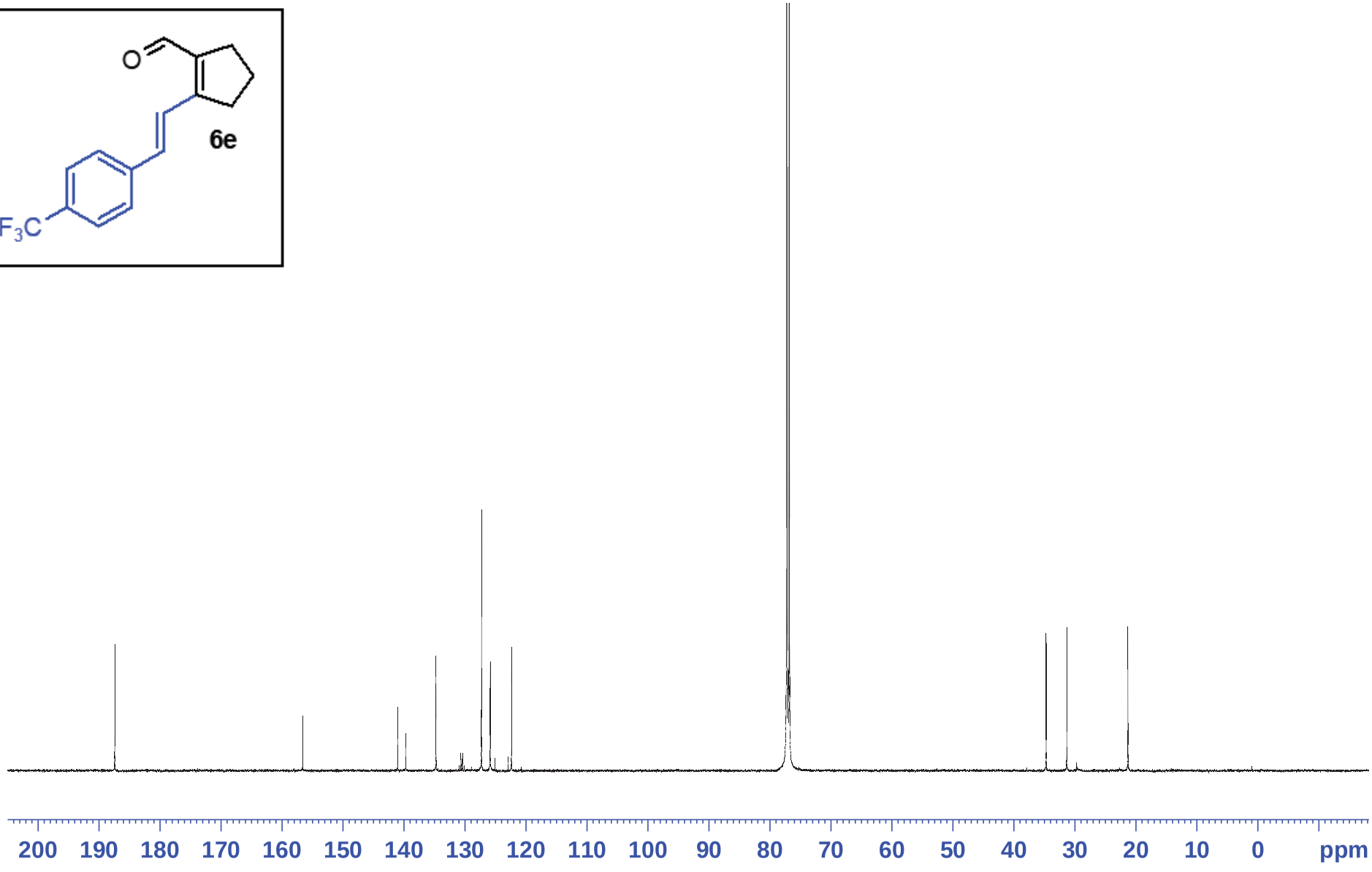




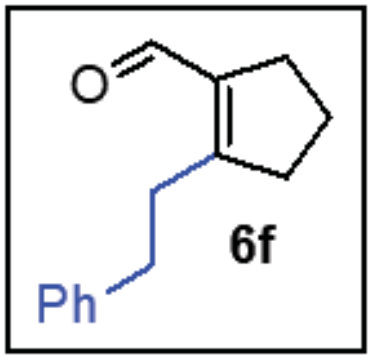
RPD-FGF23-A-17(2)
RCHO of MD-3-A7

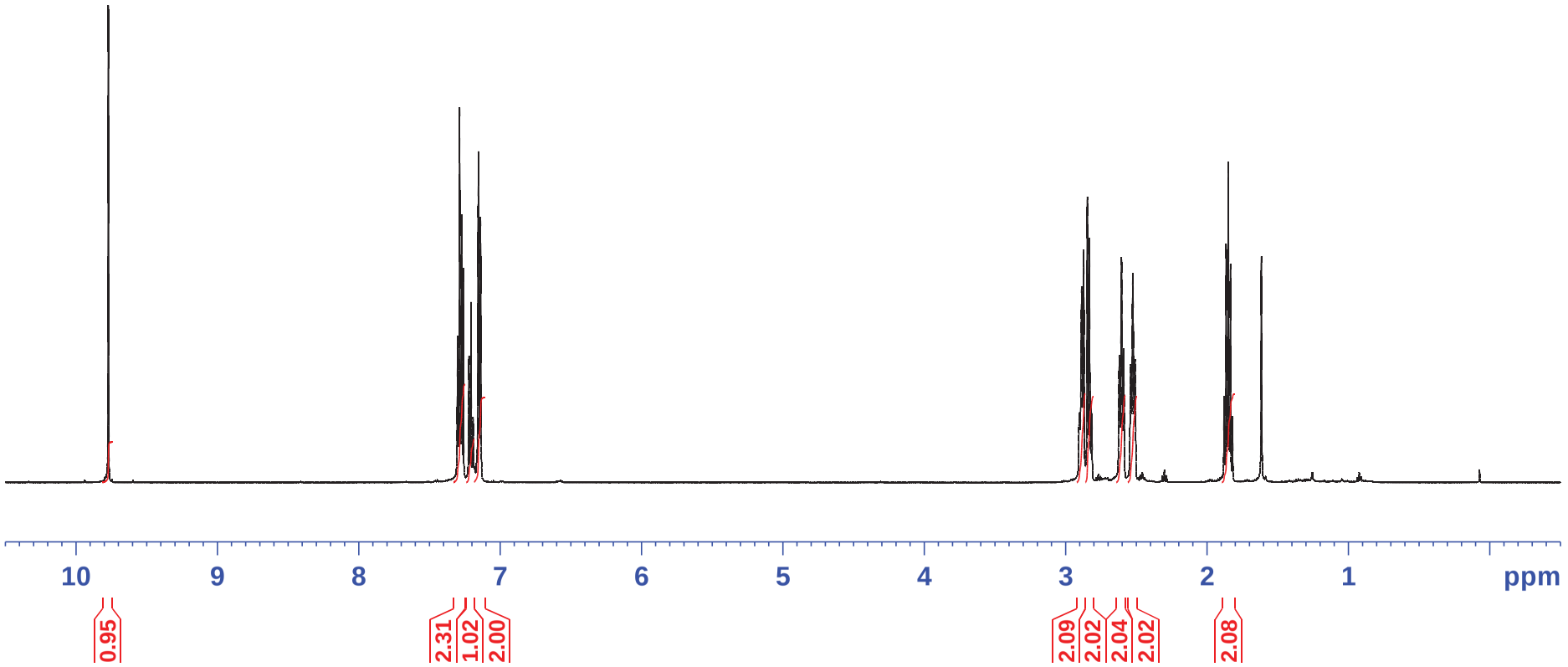
187.41
156.60
141.04
139.70
134.75
130.54
127.29
125.86
122.37
34.71
31.32
21.31





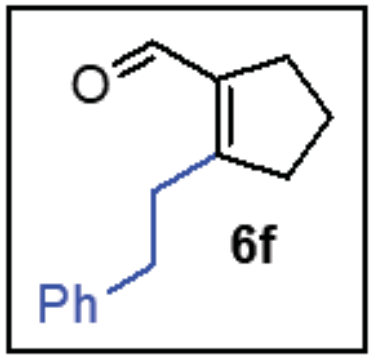
JWC-B-153(2)
RCHO of MD-3-A4

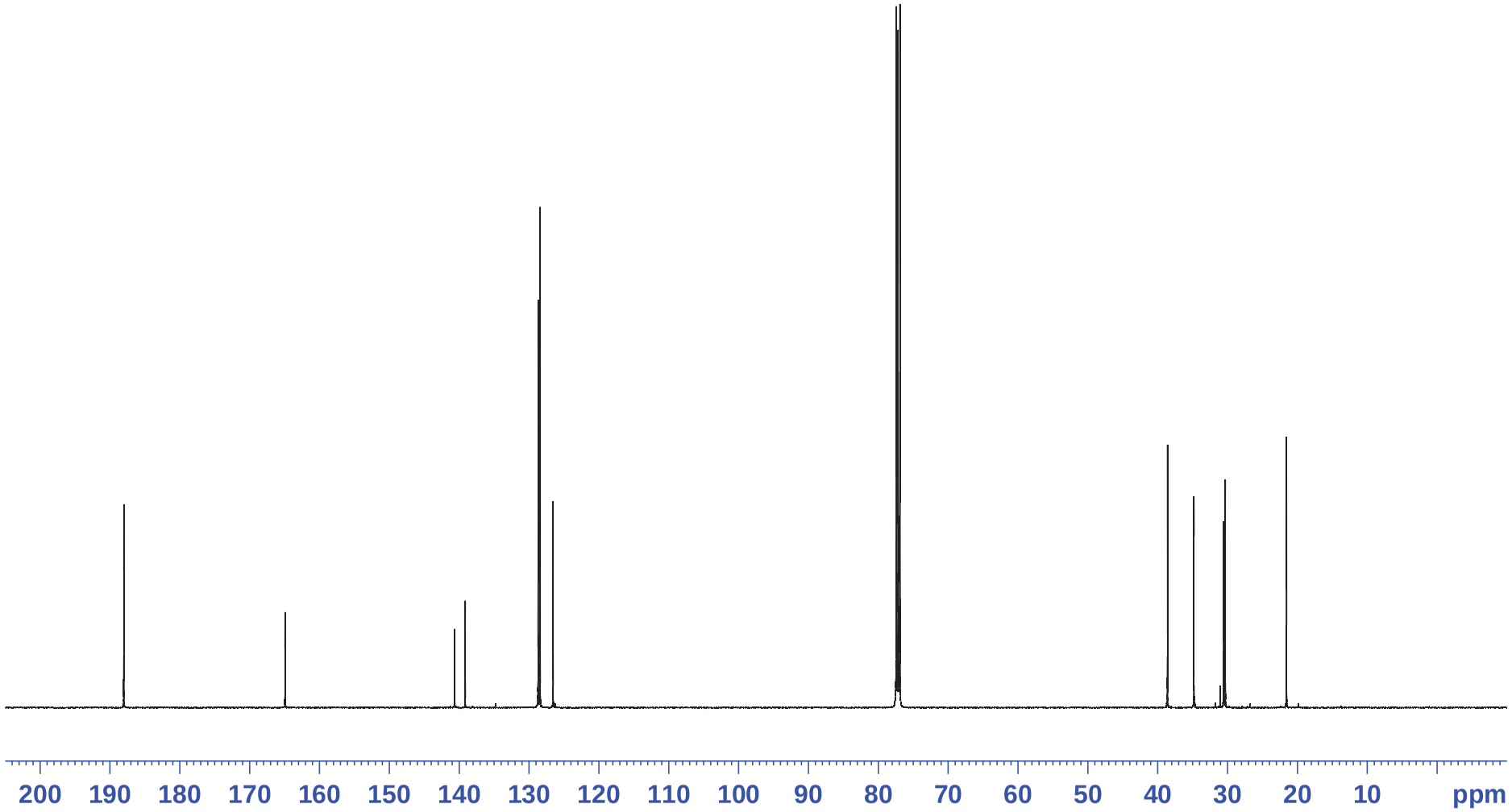




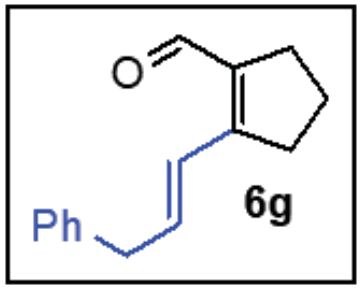
JWC-B-153(2)
RCHO of MD-3-A4

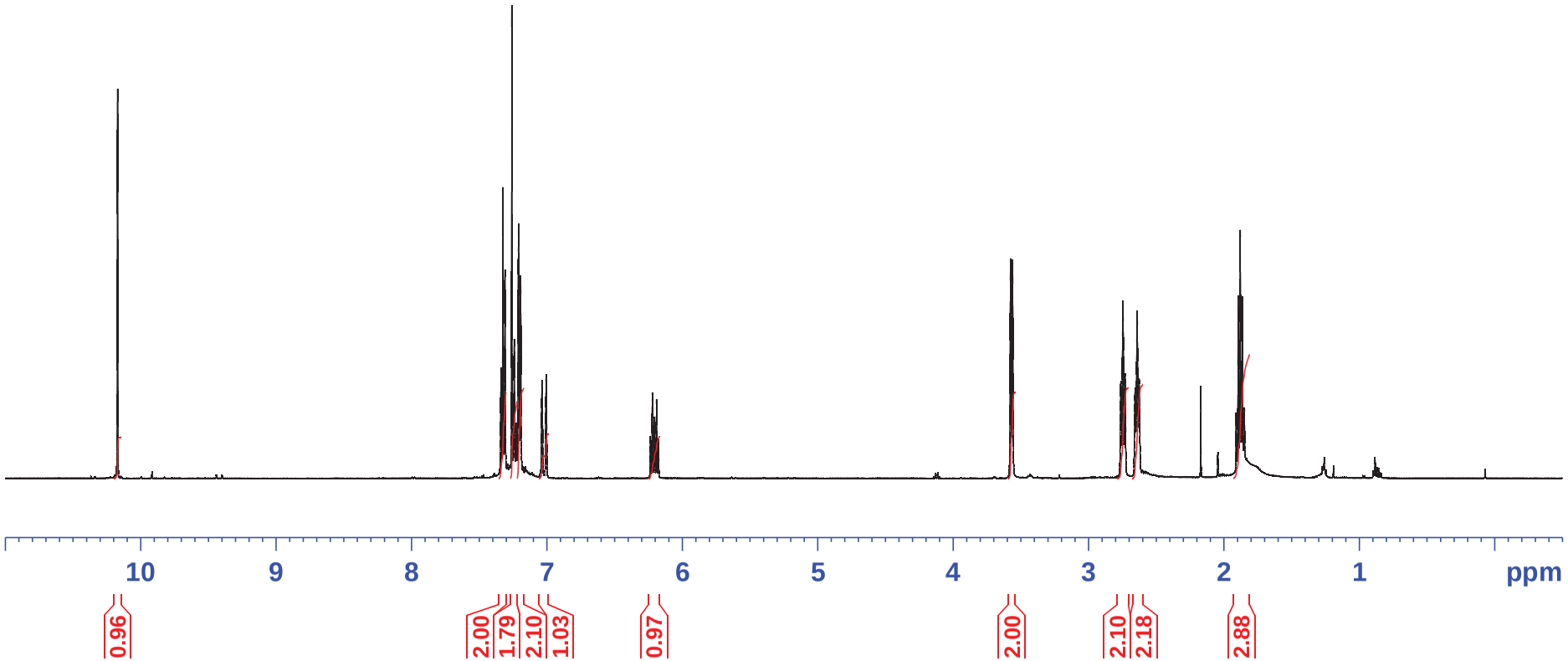
188.00
164.92
140.64
139.13
128.69
128.41
126.55
38.59
34.80
30.56
30.32
21.55





MOI-FGF23-A-99(4)
RCHO of MD-3-A29





MOI-FGF23-A-99(4)
RCHO of MD-3-A29

187.84
158.13
139.06
138.65
138.56
128.81
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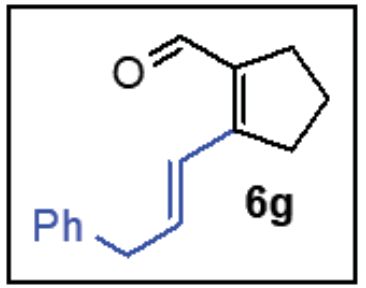
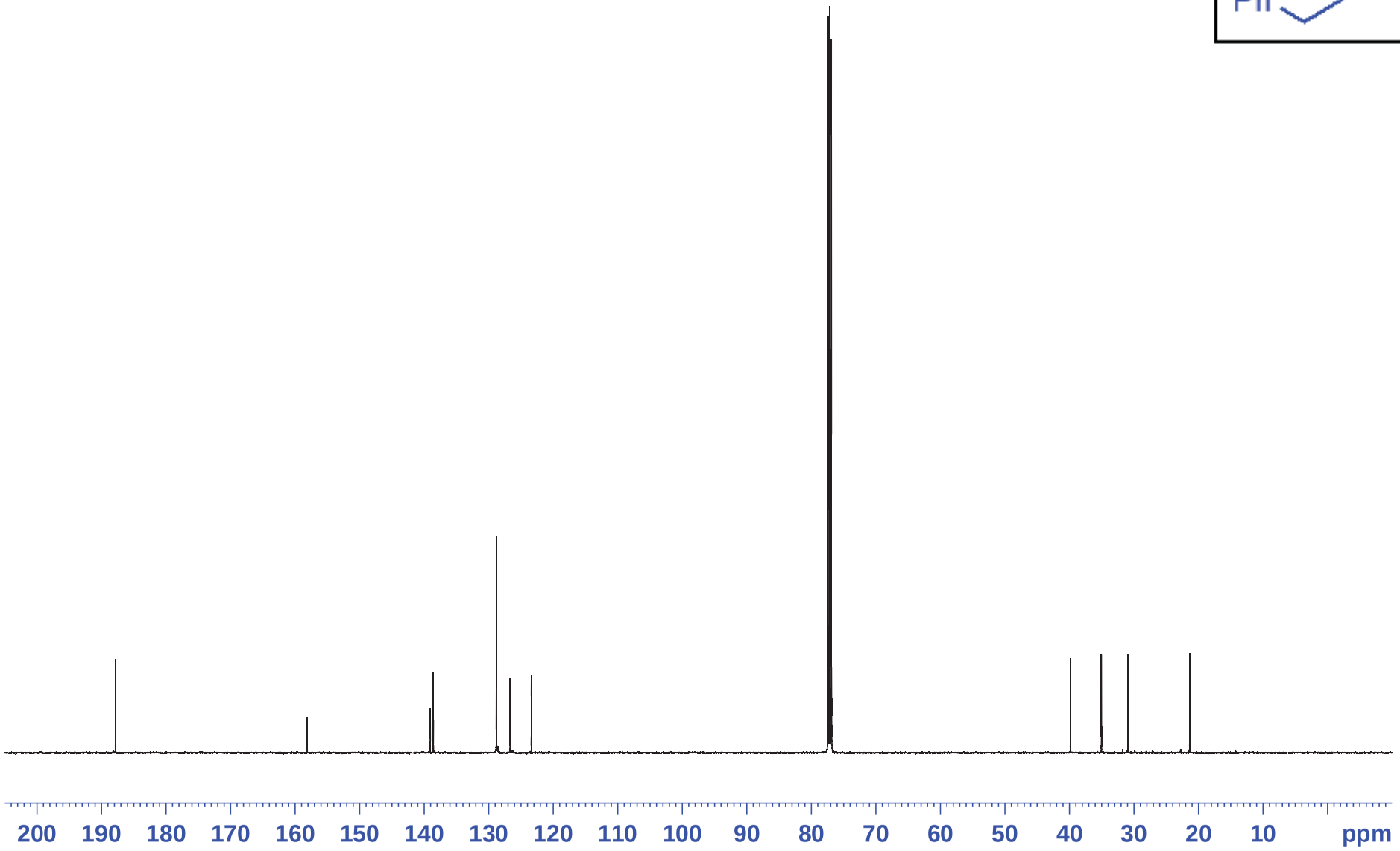
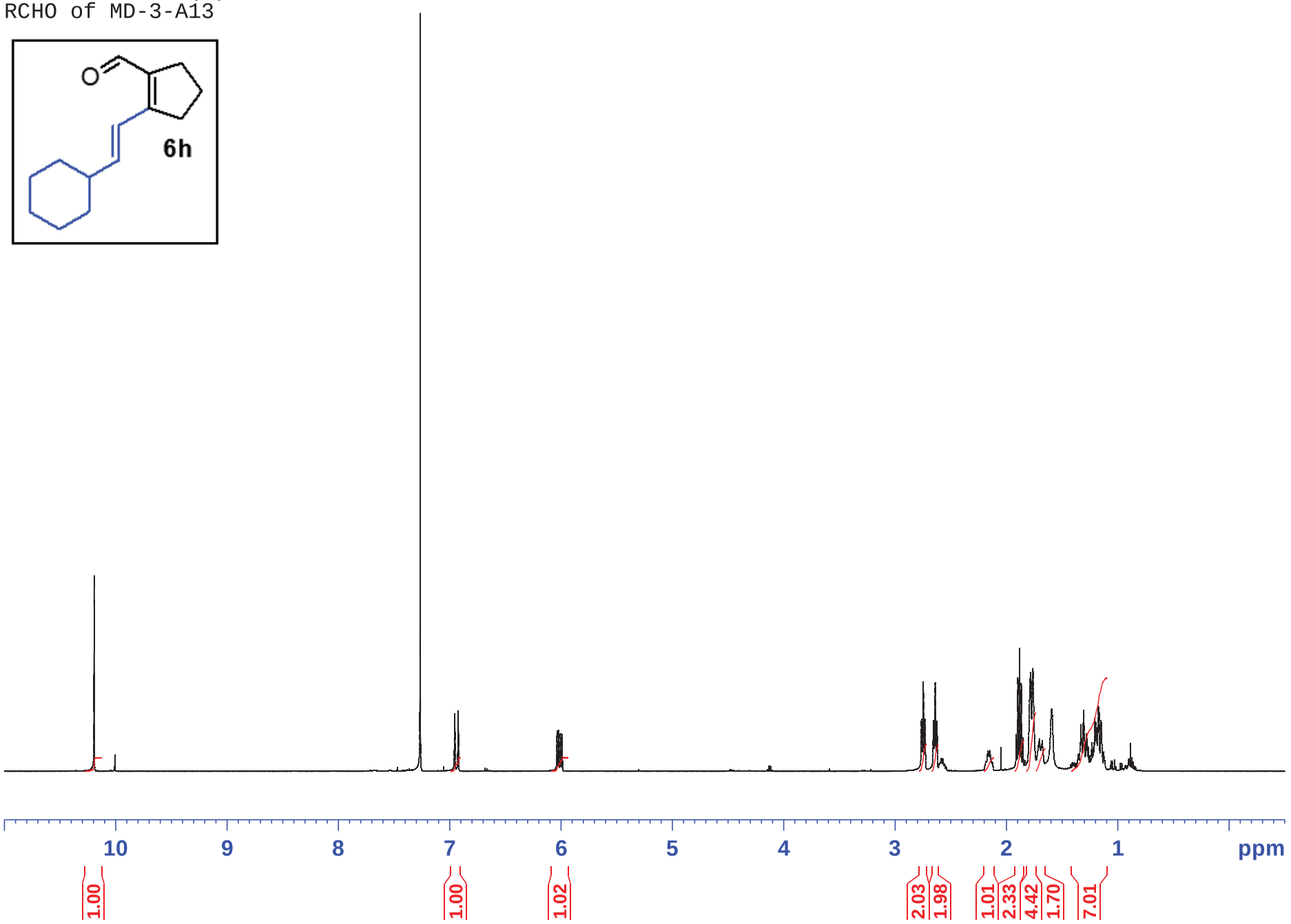
126.68
123.39
39.83
35.03
30.93
21.35



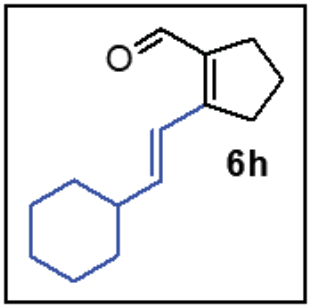


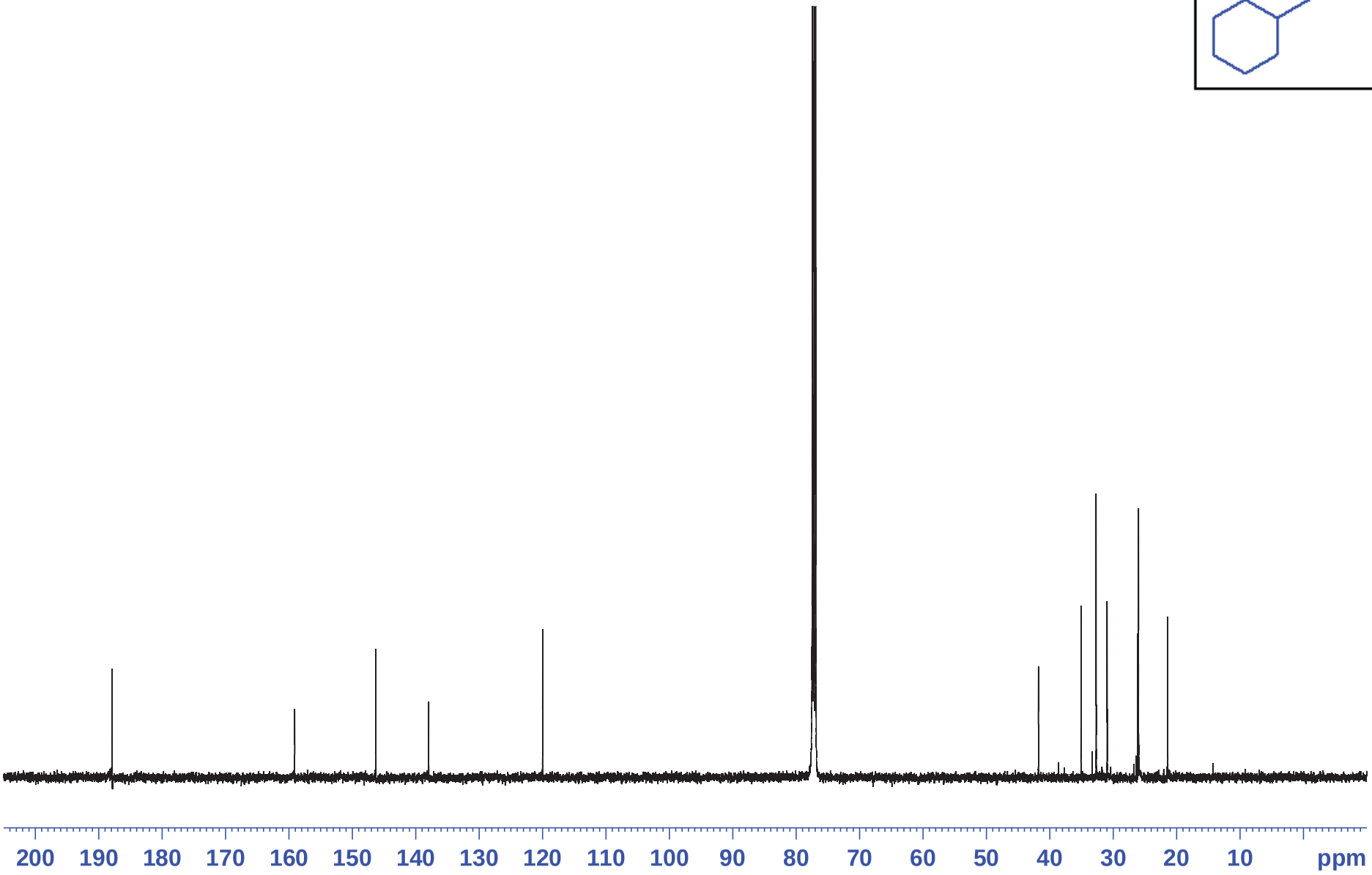
RPD-FGF23-A-47(3)
RCHO of MD-3-A13



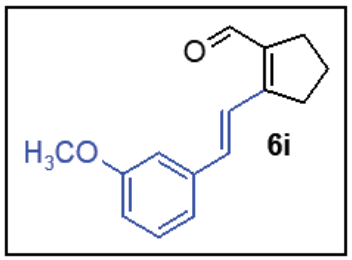
RPD-FGF23-A-133(2)
RCHO of MD-3-A13

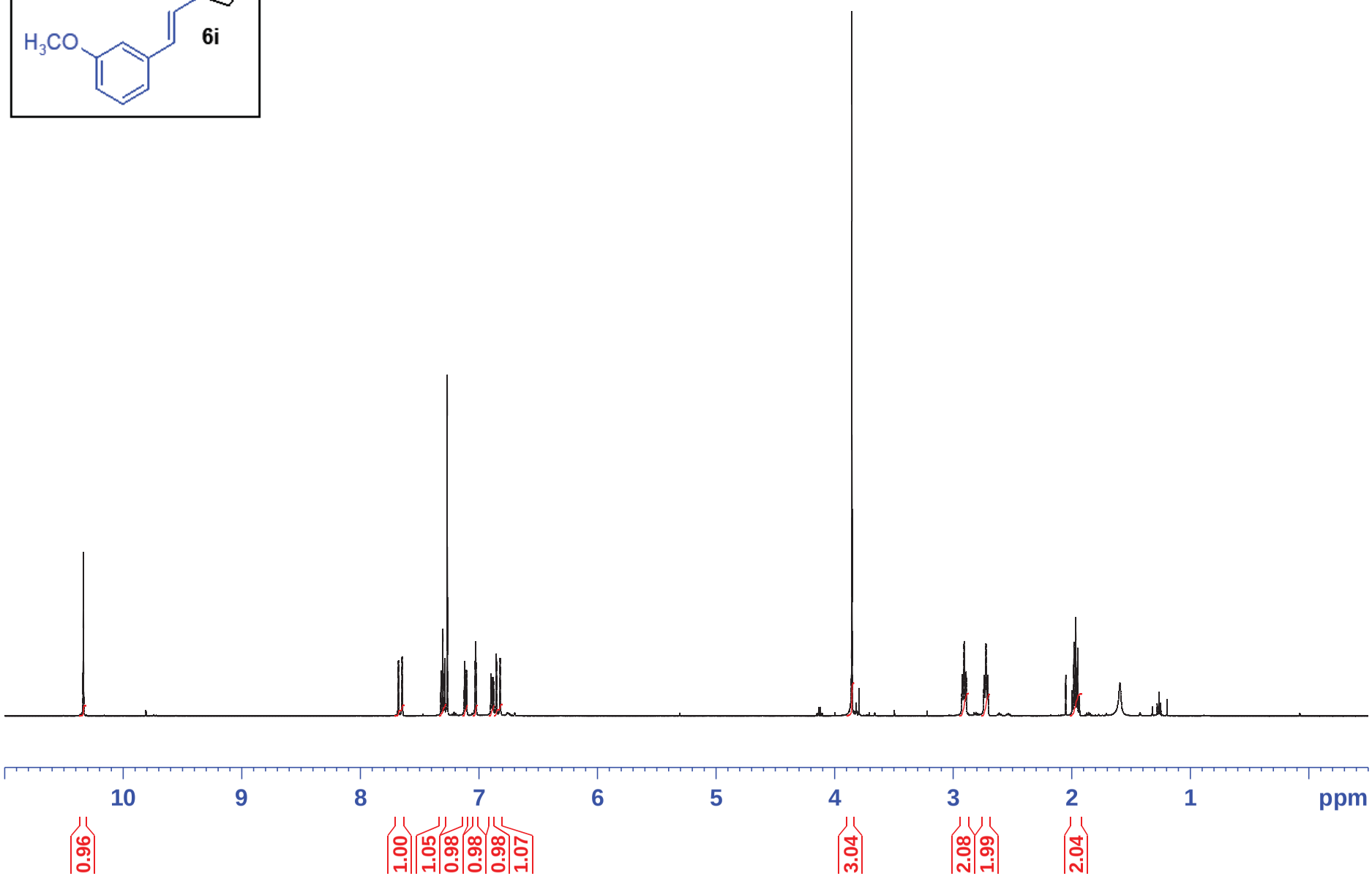
187.89
159.07
146.27
137.93
119.96
41.77
35.05
32.67
30.93
26.13
25.97
21.39





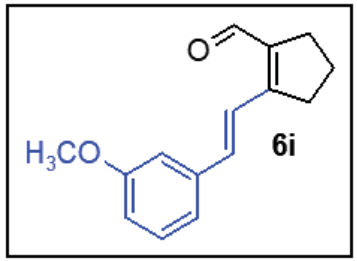
RPD-FGF23-A-129(4)
RCHO of MD-3-A21

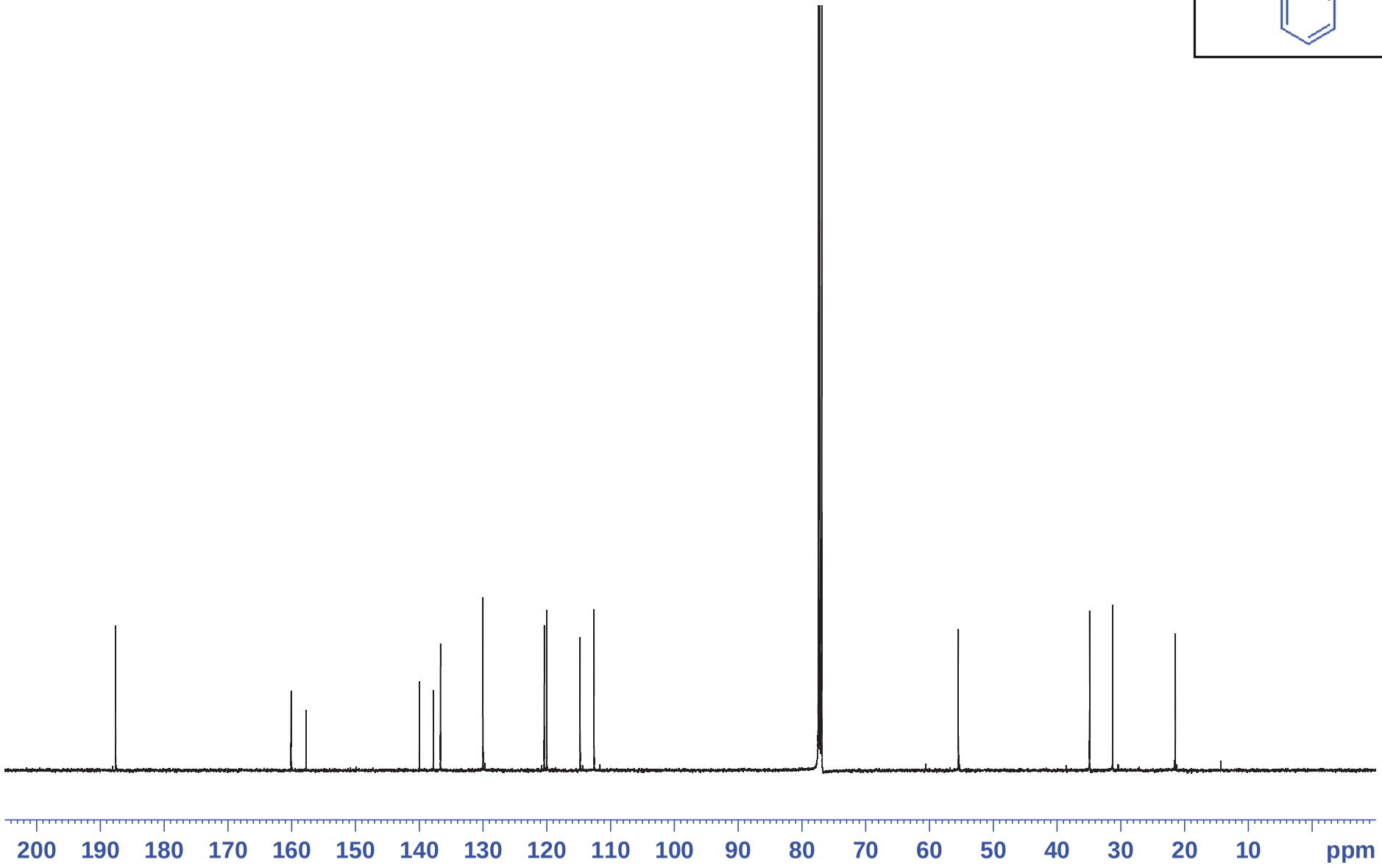




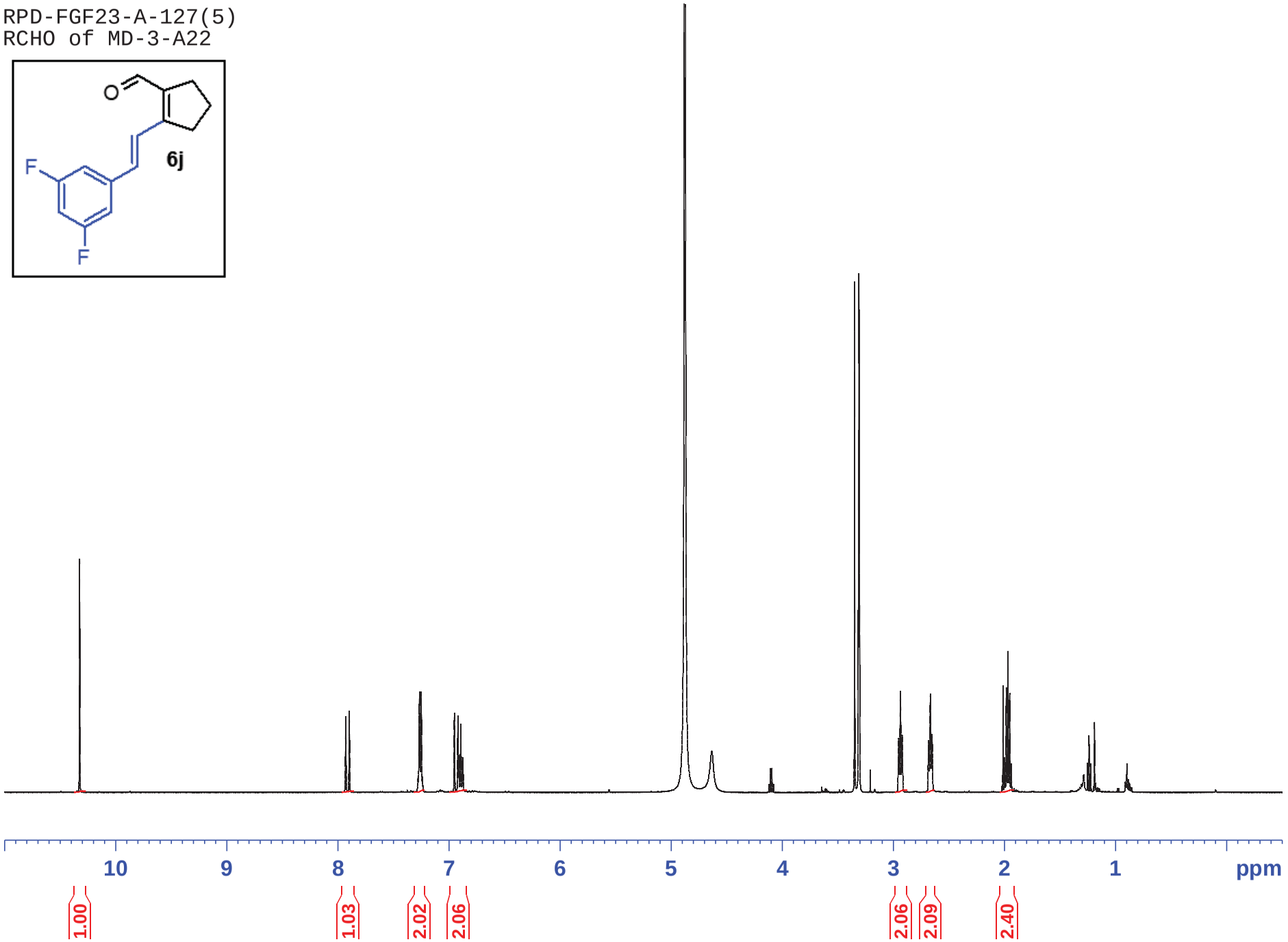
RPD-FGF23-A-127(4)
RCHO of MD-3-A21

187.60
160.10
157.74
139.99
137.80
136.67
130.00
120.40
119.99
114.77
112.58
55.46
34.90
31.28
21.44

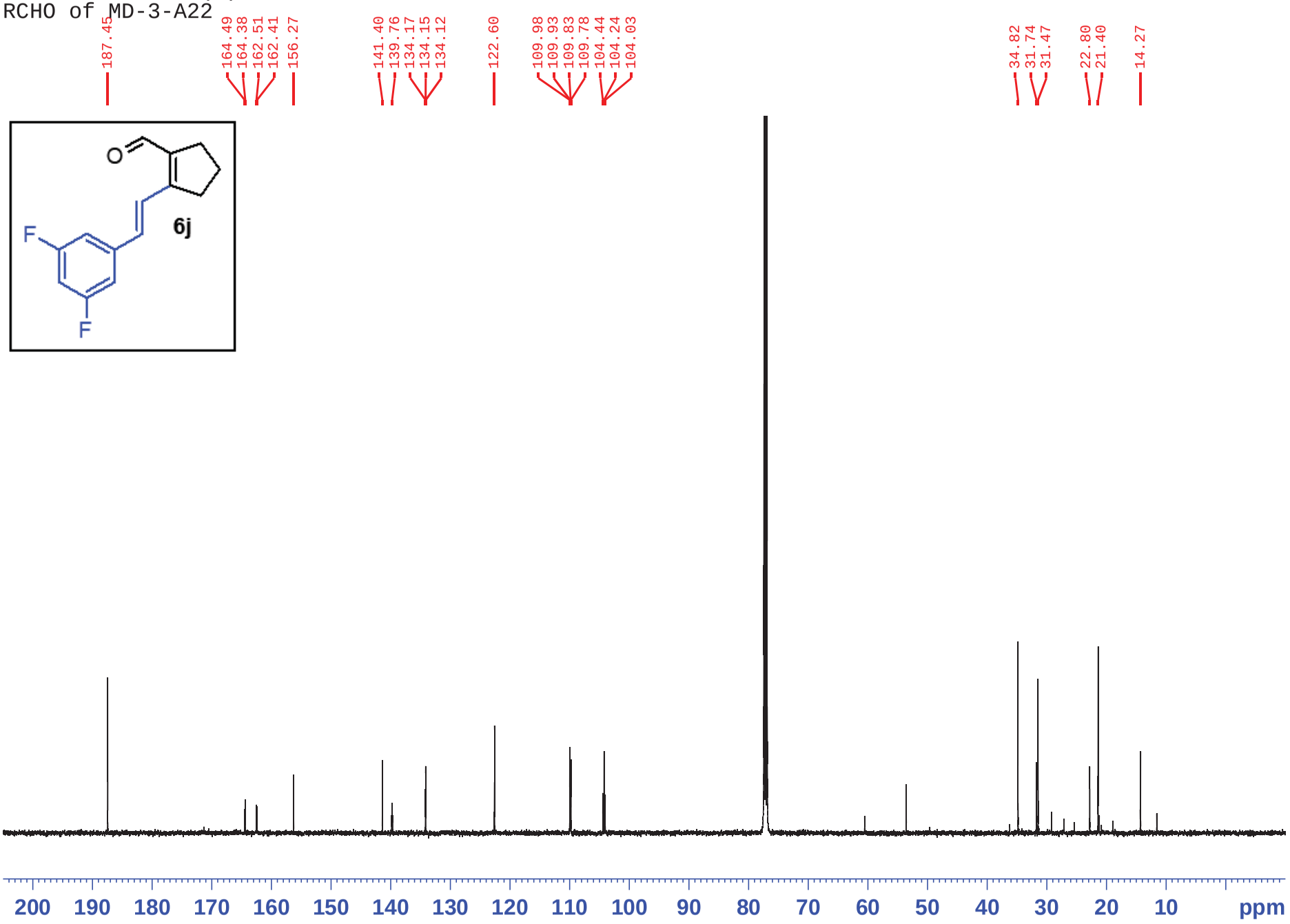




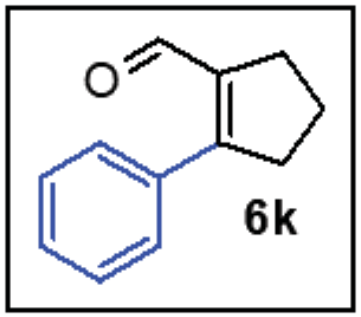
RPD-FGF23-A-127(5)
RCHO of MD-3-A22



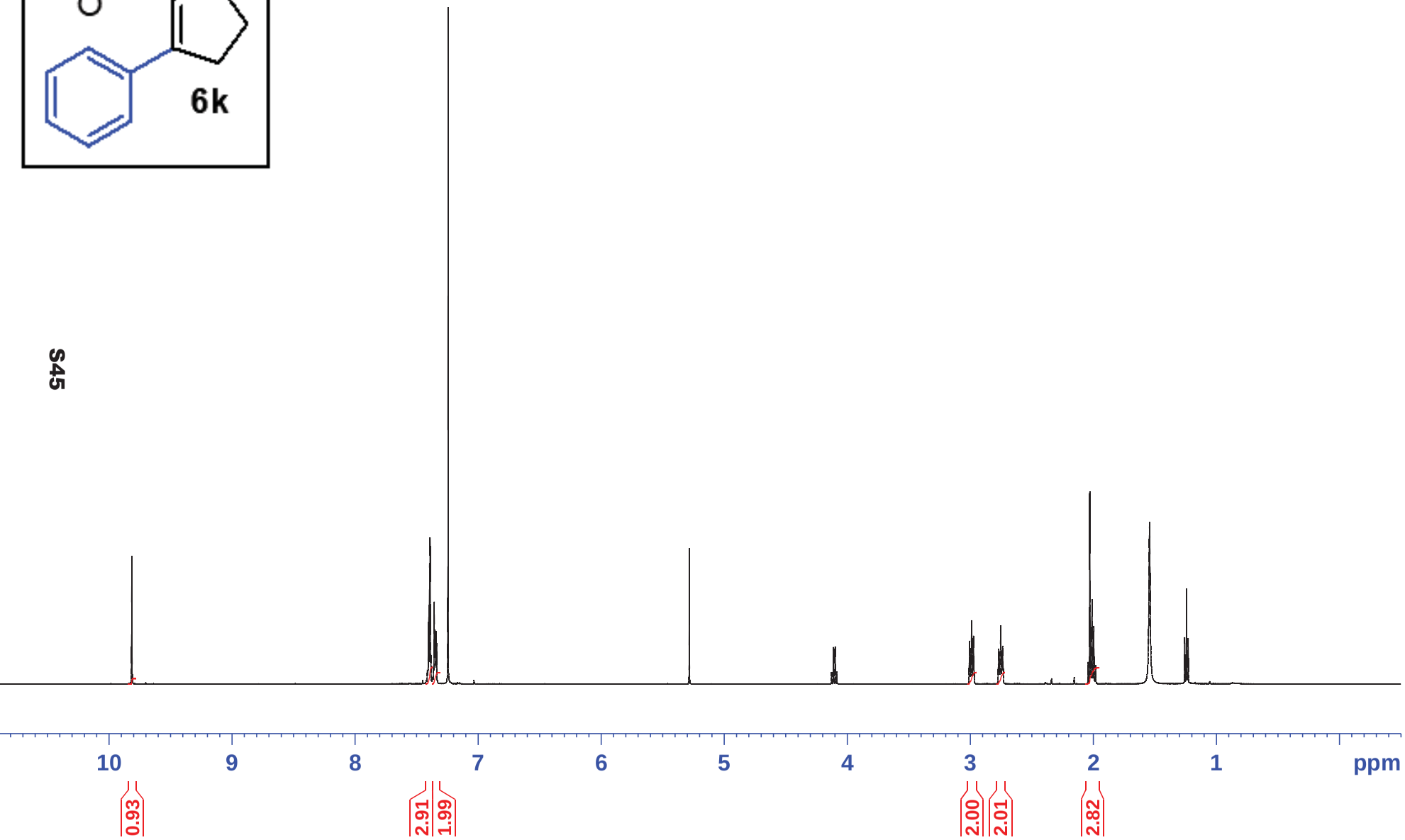
RPD-FGF23-A-127(5)
RCHO of MD-3-A22



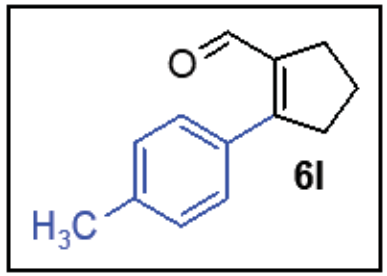
MOI-FGF23-A-95(5)
2-Phenyl-cyclopent-1-enecarbaldehyde

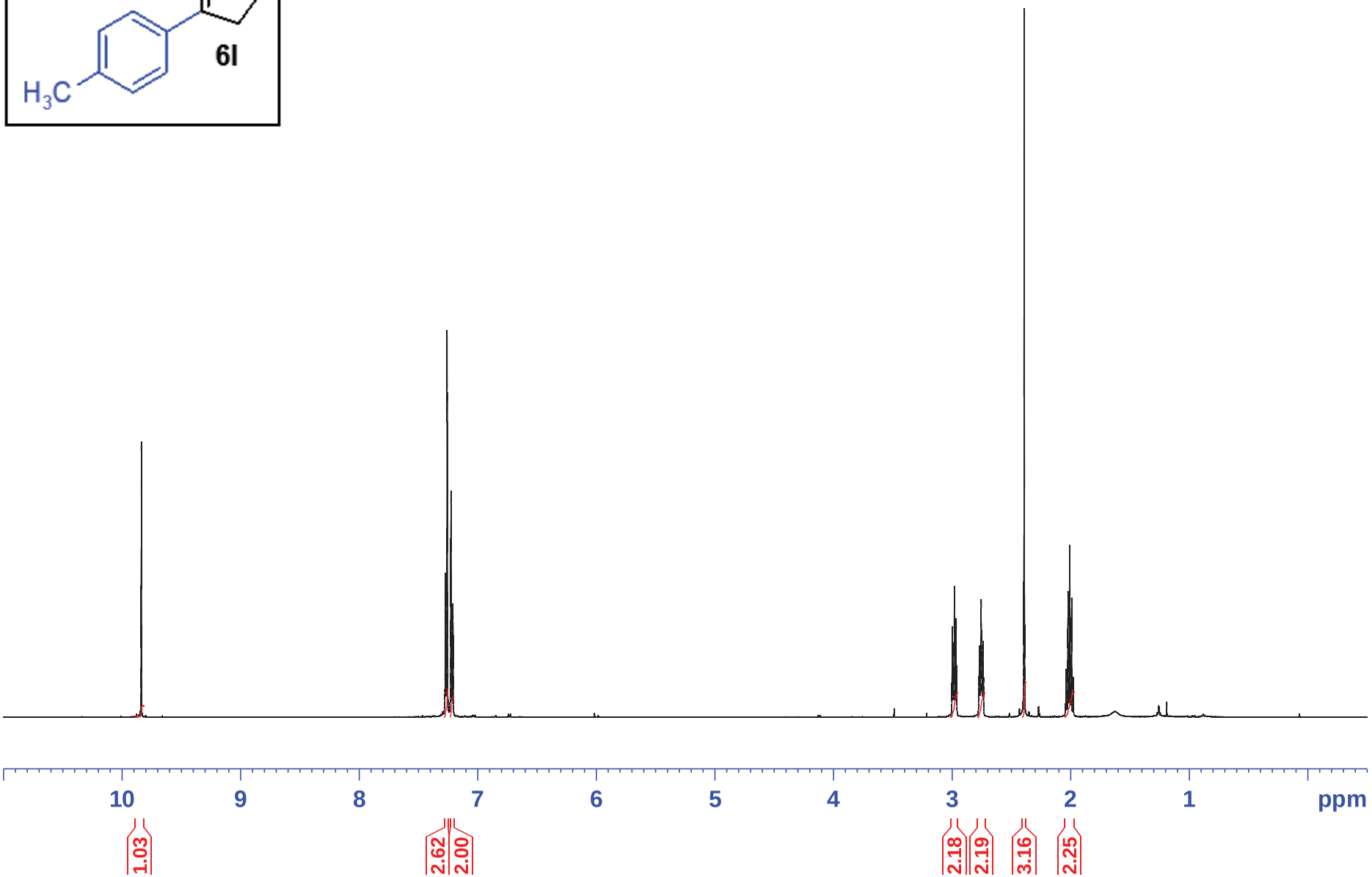


S45

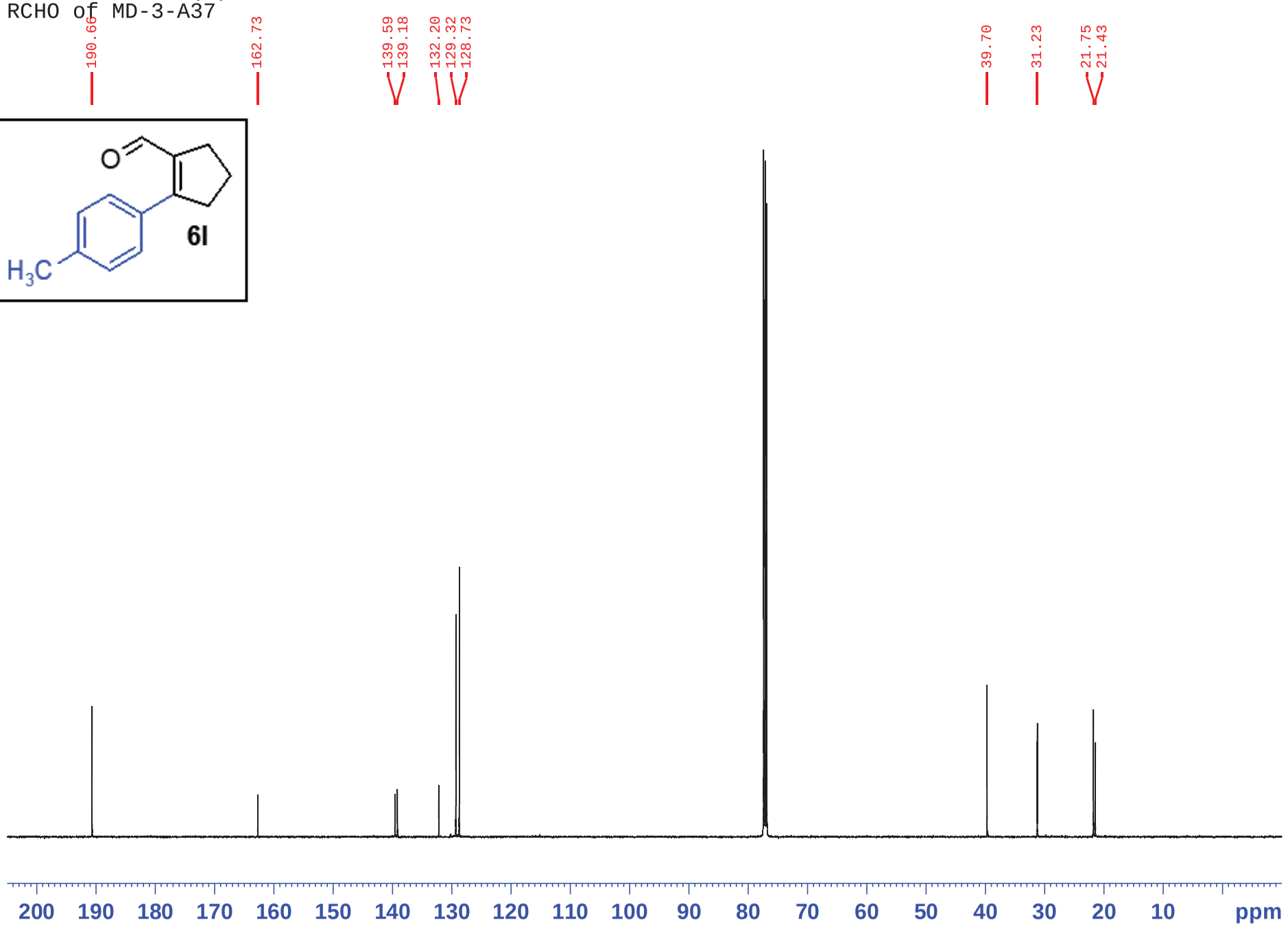


RPD-FGF23-A-81(2)
RCHO of MD-3-A37

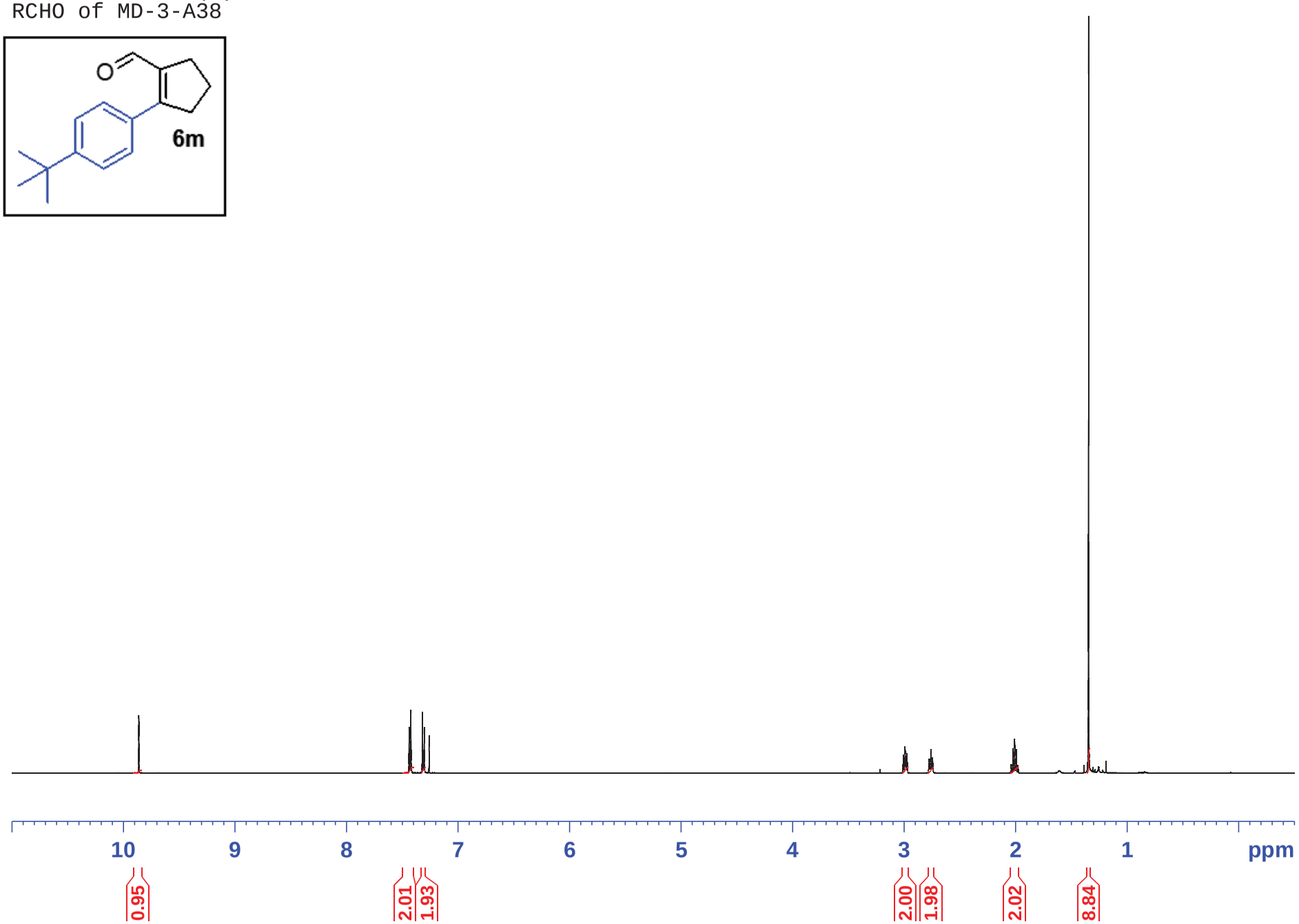




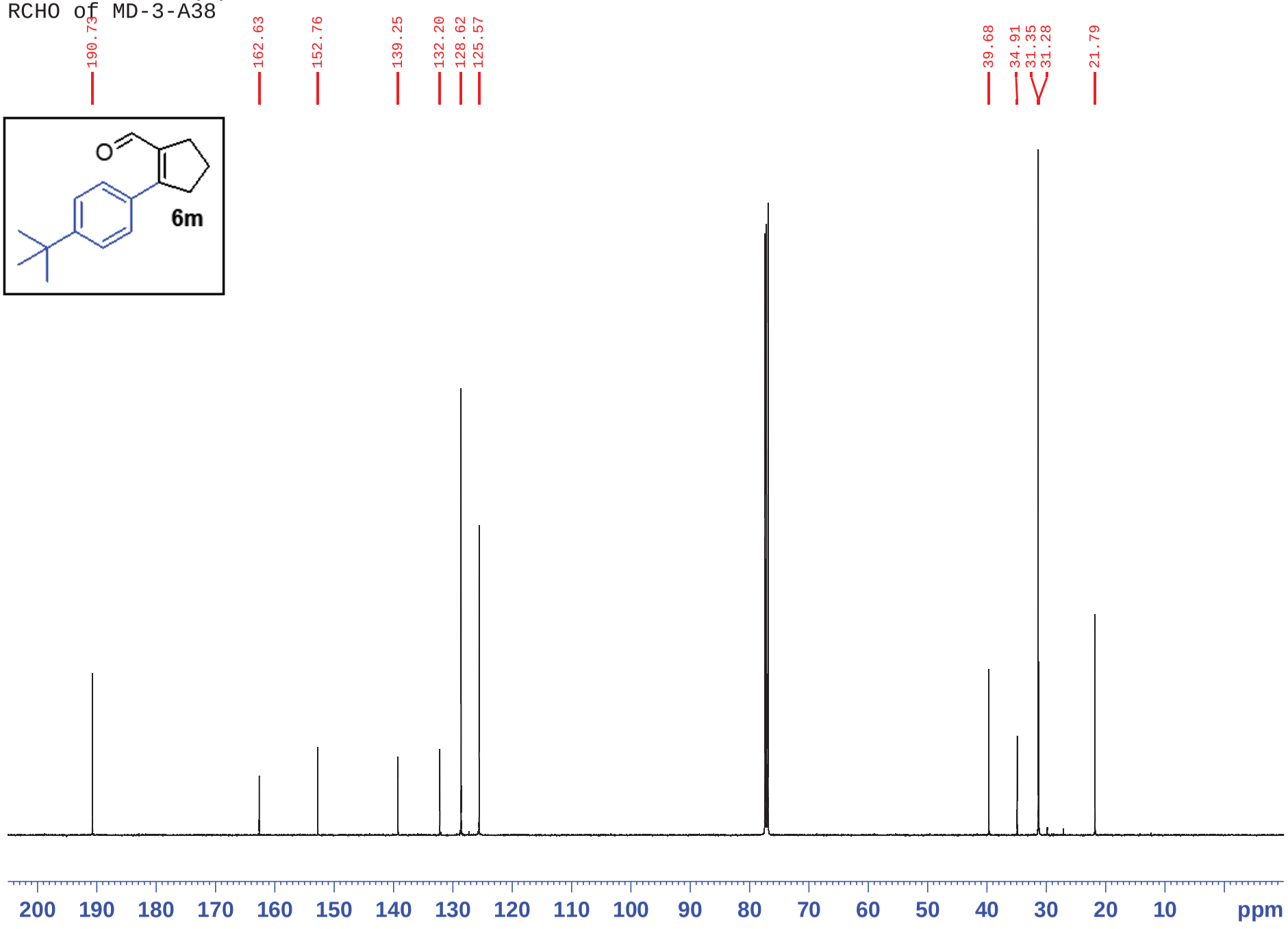
RPD-FGF23-A-81(2)
RCHO of MD-3-A37



RPD-FGF23-A-89(3)
RCHO of MD-3-A38

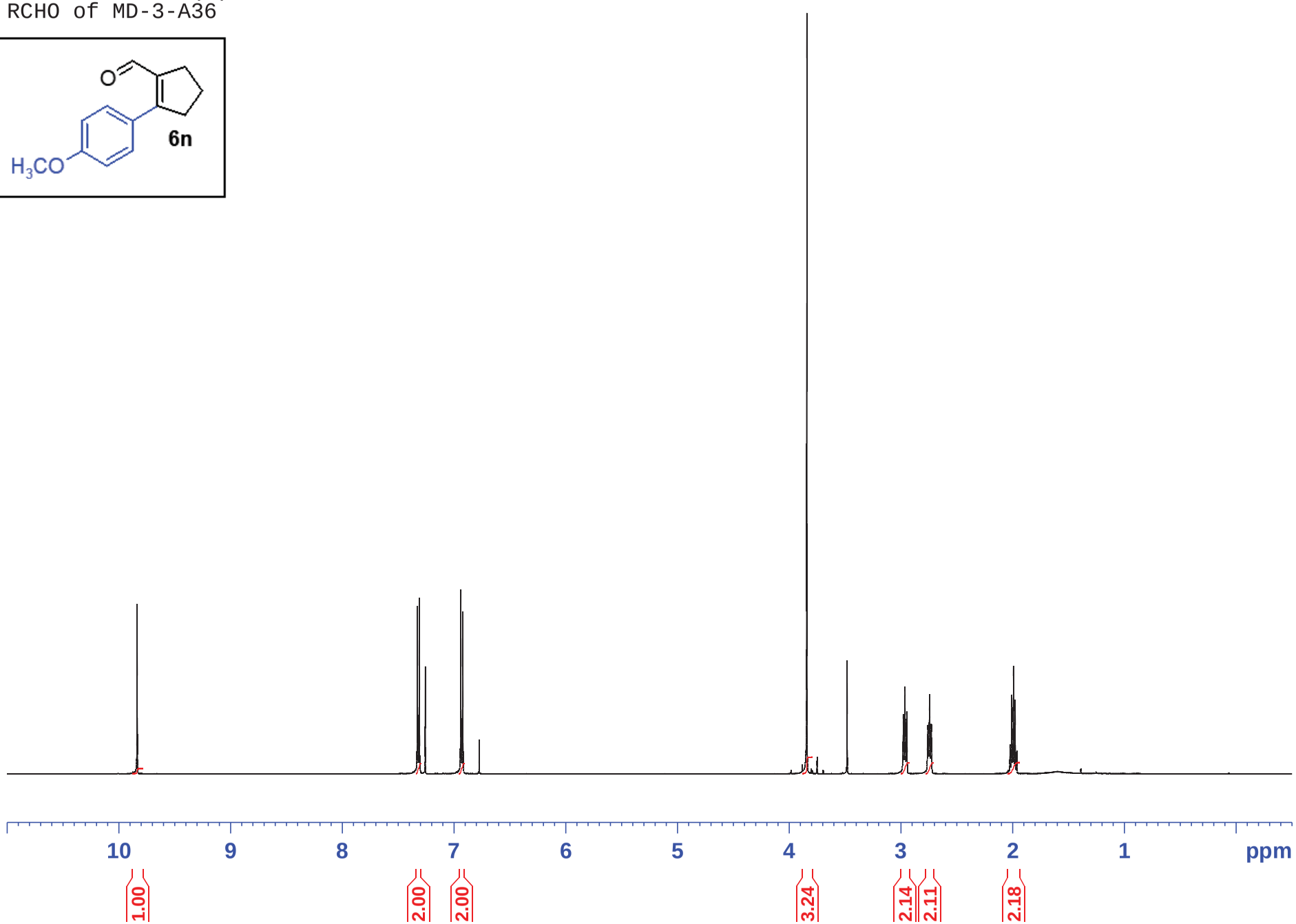


RPD-FGF23-A-97(2)
RCHO of MD-3-A38



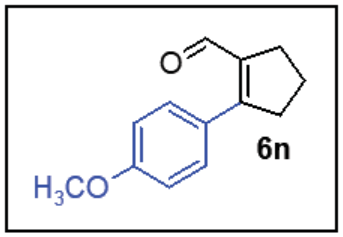
RPD-FGF23-A-77(4)
RCHO of MD-3-A36

S50



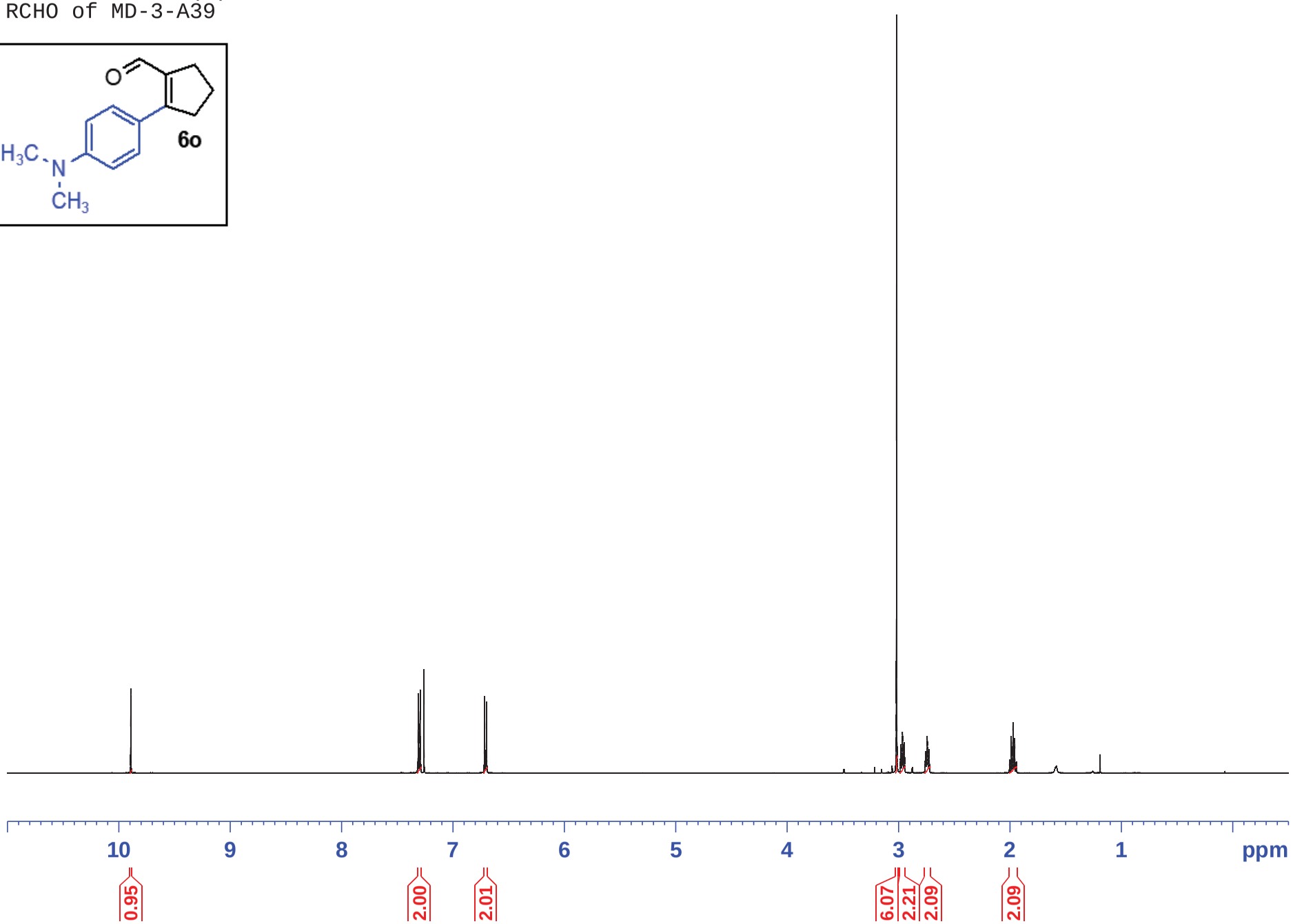
RPD-FGF23-A77(4)
RCHO of MD-3-A36

190.60
162.35
160.72
138.59
130.29
127.53
114.07
55.51
39.60
31.26
21.74

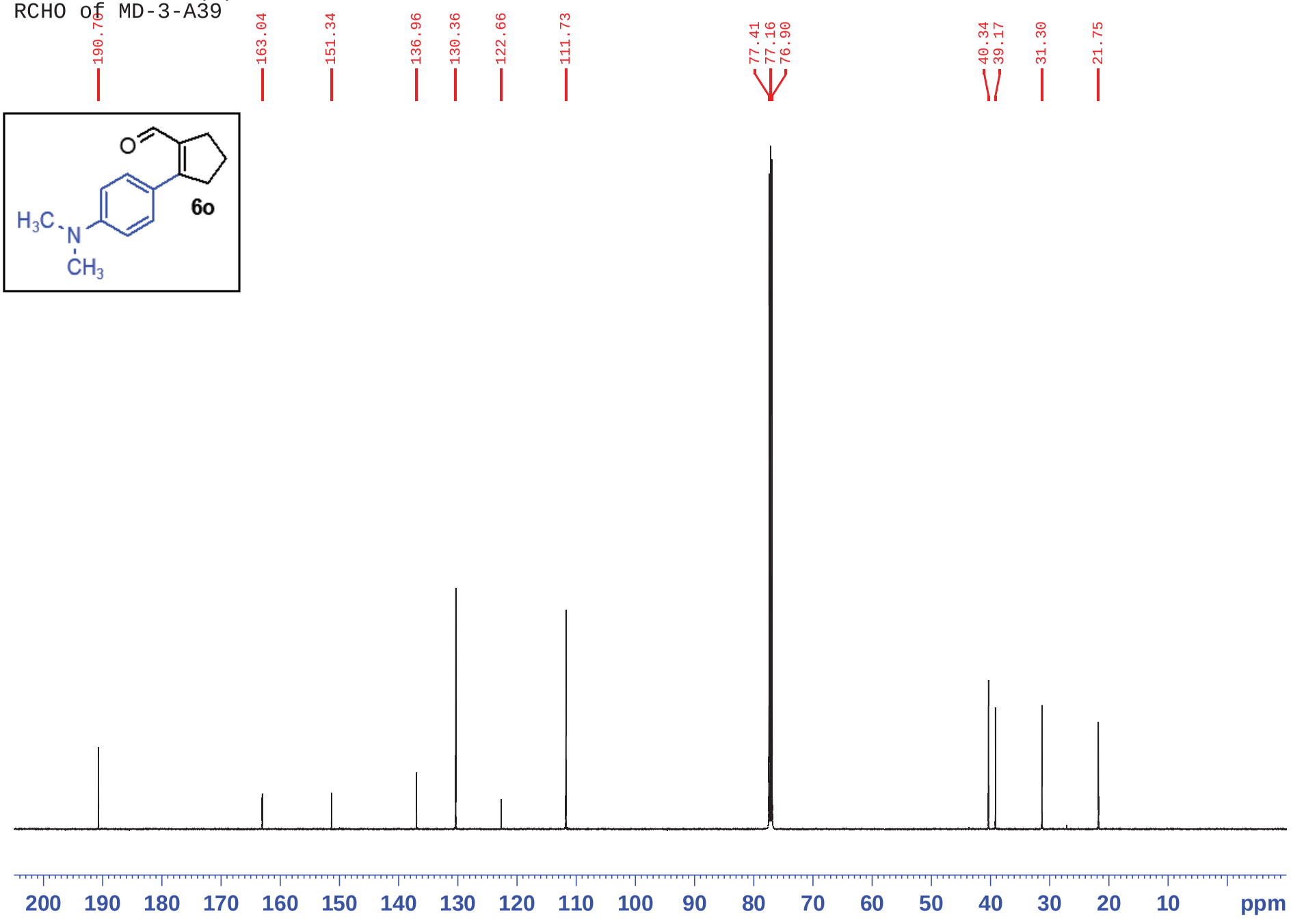


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

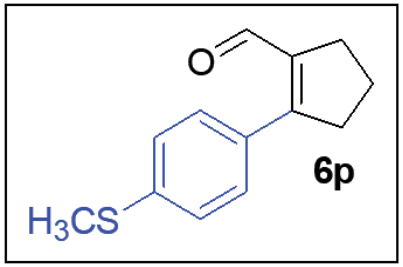
RPD-FGF23-A-79(5)
RCHO of MD-3-A39

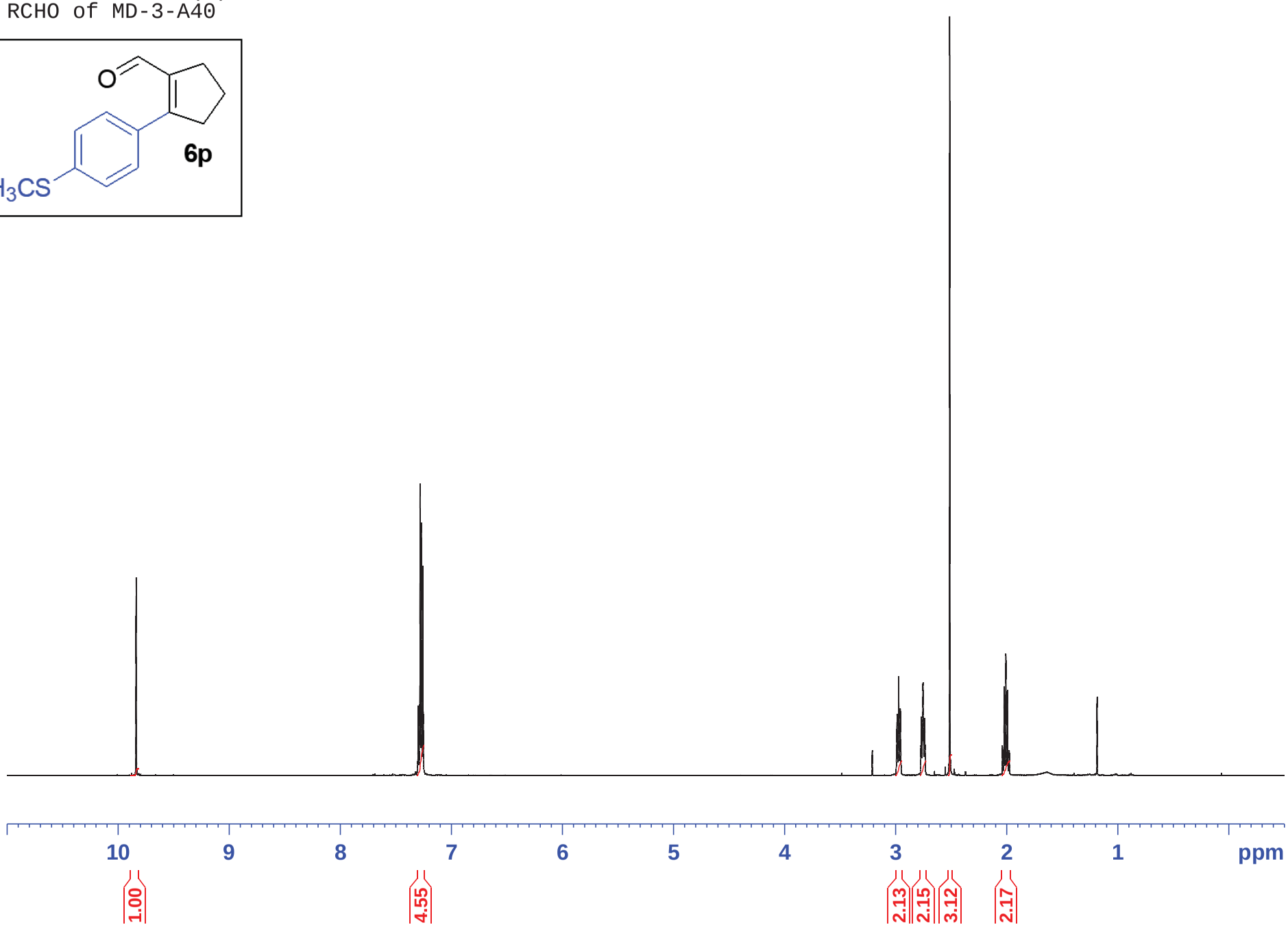


RPD-FGF23-A-79(5)
RCHO of MD-3-A39

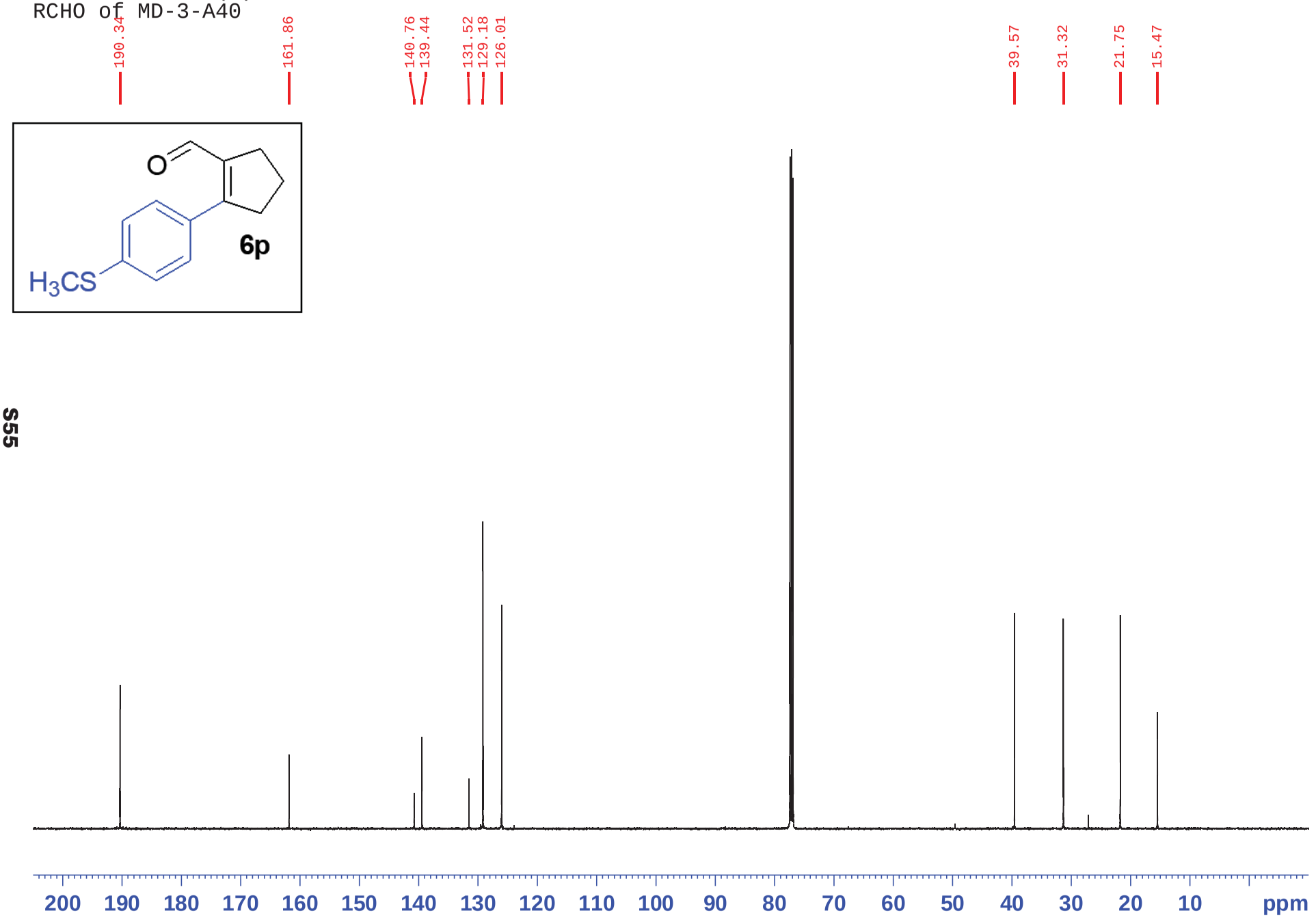


RPD-FGF23-A-83(3)
RCHO of MD-3-A40

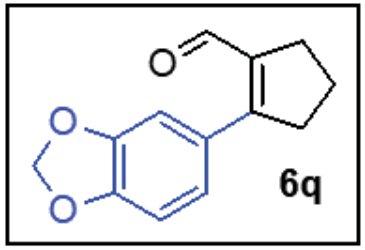


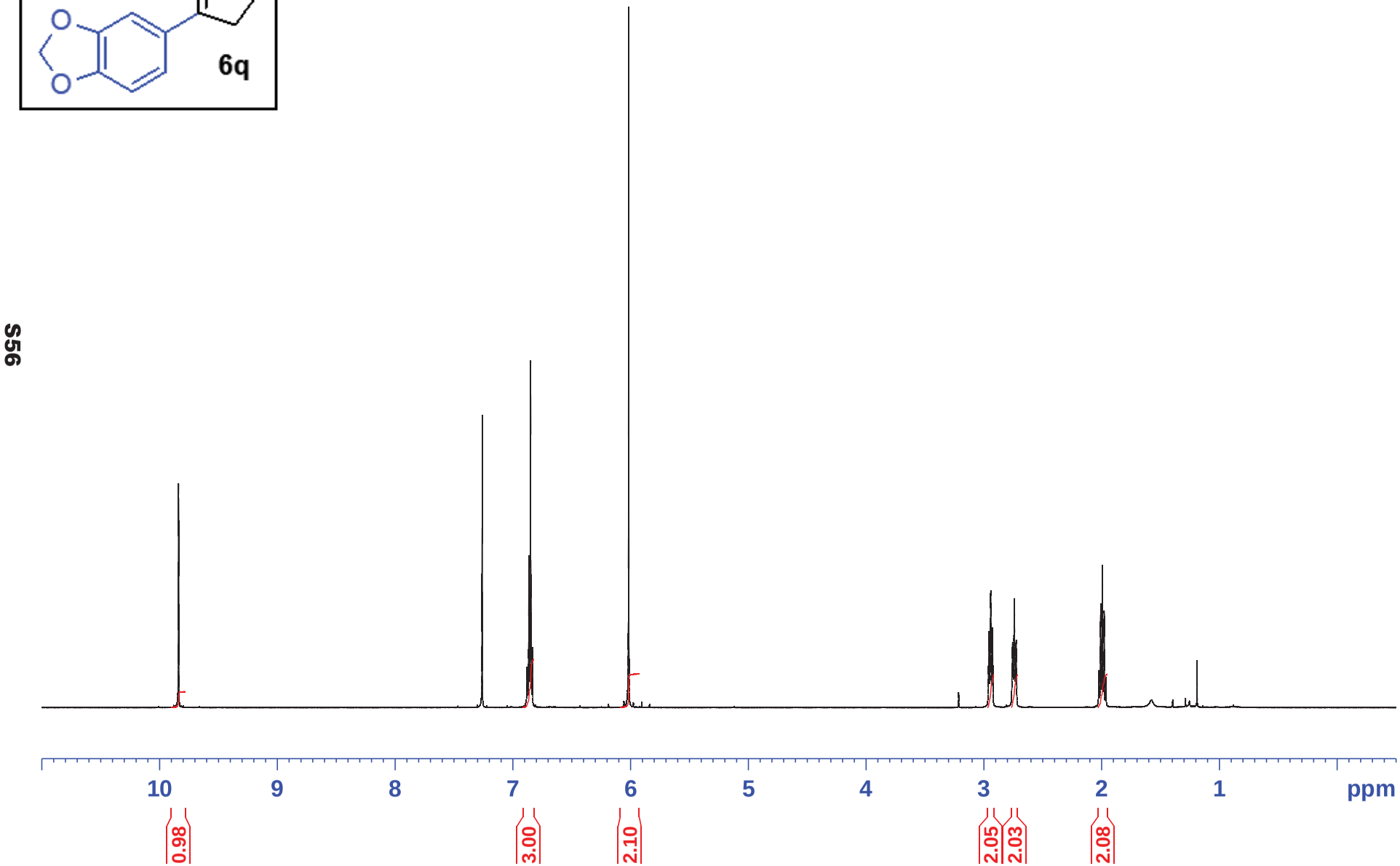


RPD-FGF23-A-83(3)
RCHO of MD-3-A40



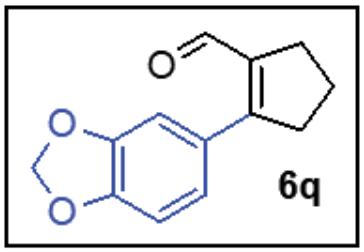
RPD-FGF23-A-97(2)
RCHO of MD-3-A41





RPD-FGF23-A-97(2)
RCHO of MD-3-A41

190.42
161.99
148.80
148.06
139.16
129.03
123.28
108.85
108.47
101.64
77.42
77.16
76.91
39.79
31.30
21.73

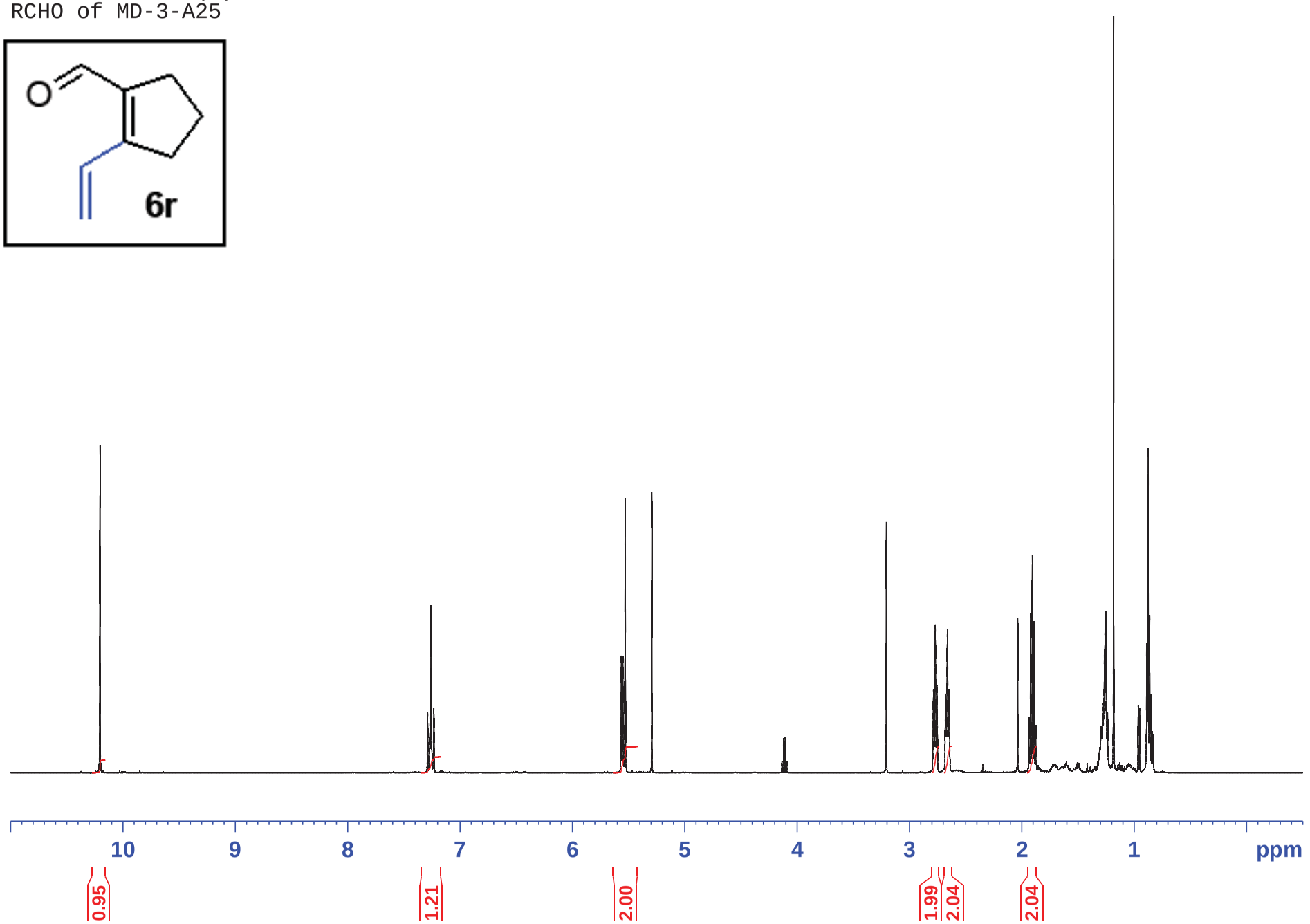


S57

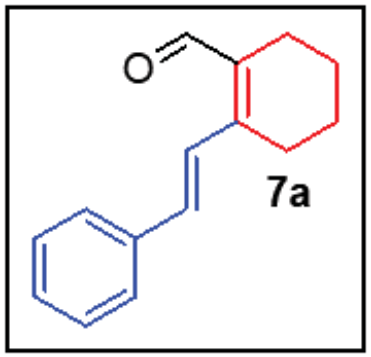
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-25(2)
RCHO of MD-3-A25

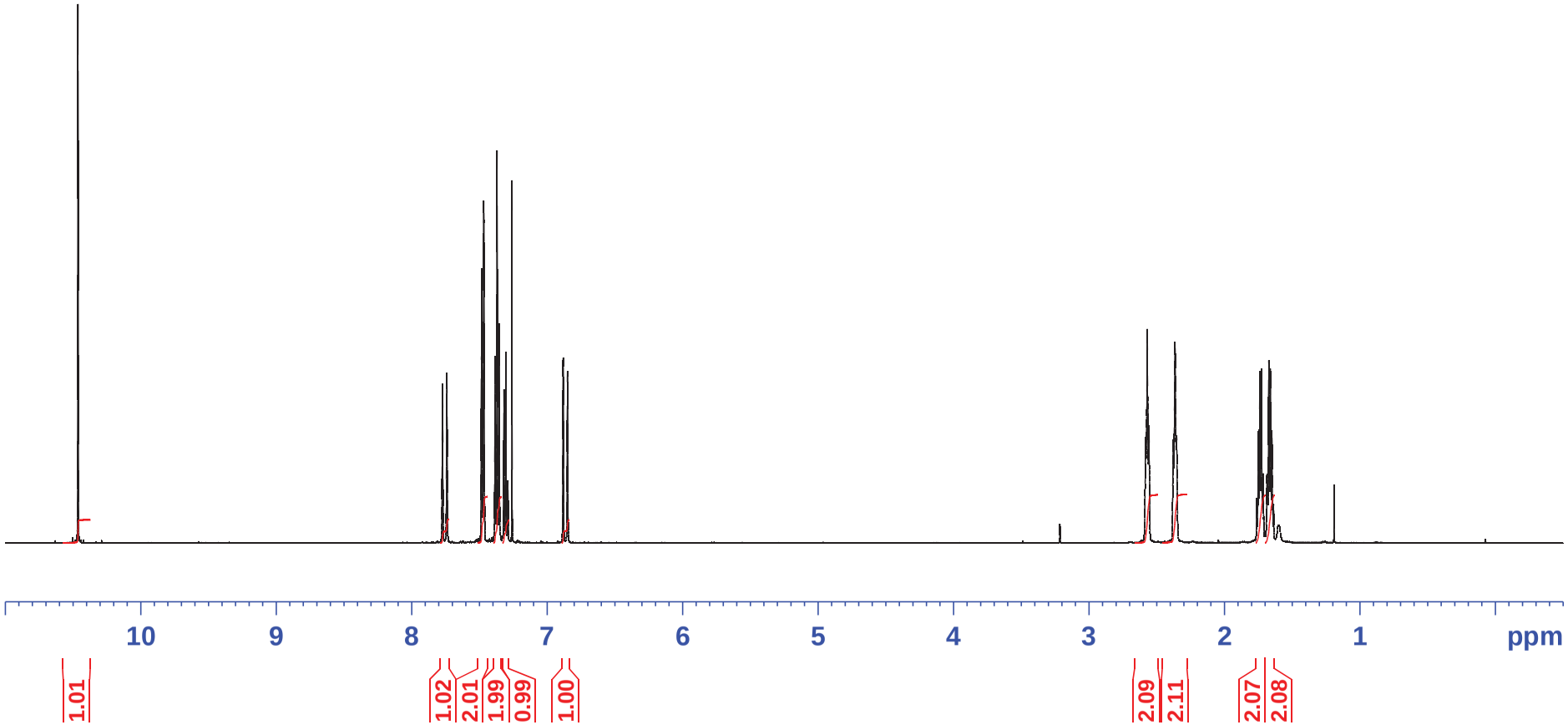
S58



RPD-FGF23-A-69(5)
RCHO of MD-3-A33

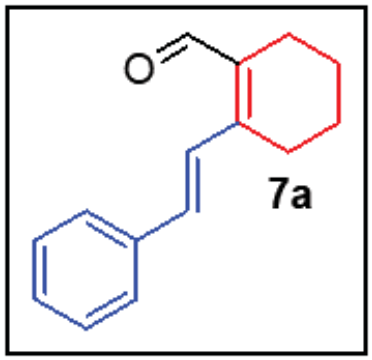


S59



RPD-FGF23-A-69(5)
RCHO of MD-3-A33

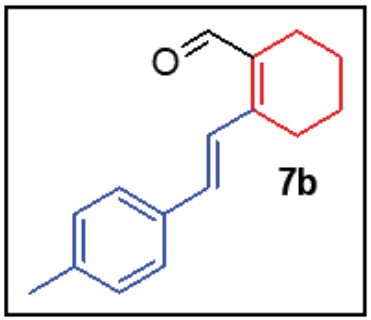
190.60
151.75
136.75
135.90
133.60
128.99
128.78
127.11
123.49
77.41
77.15
76.90
27.70
23.42
22.12
21.66

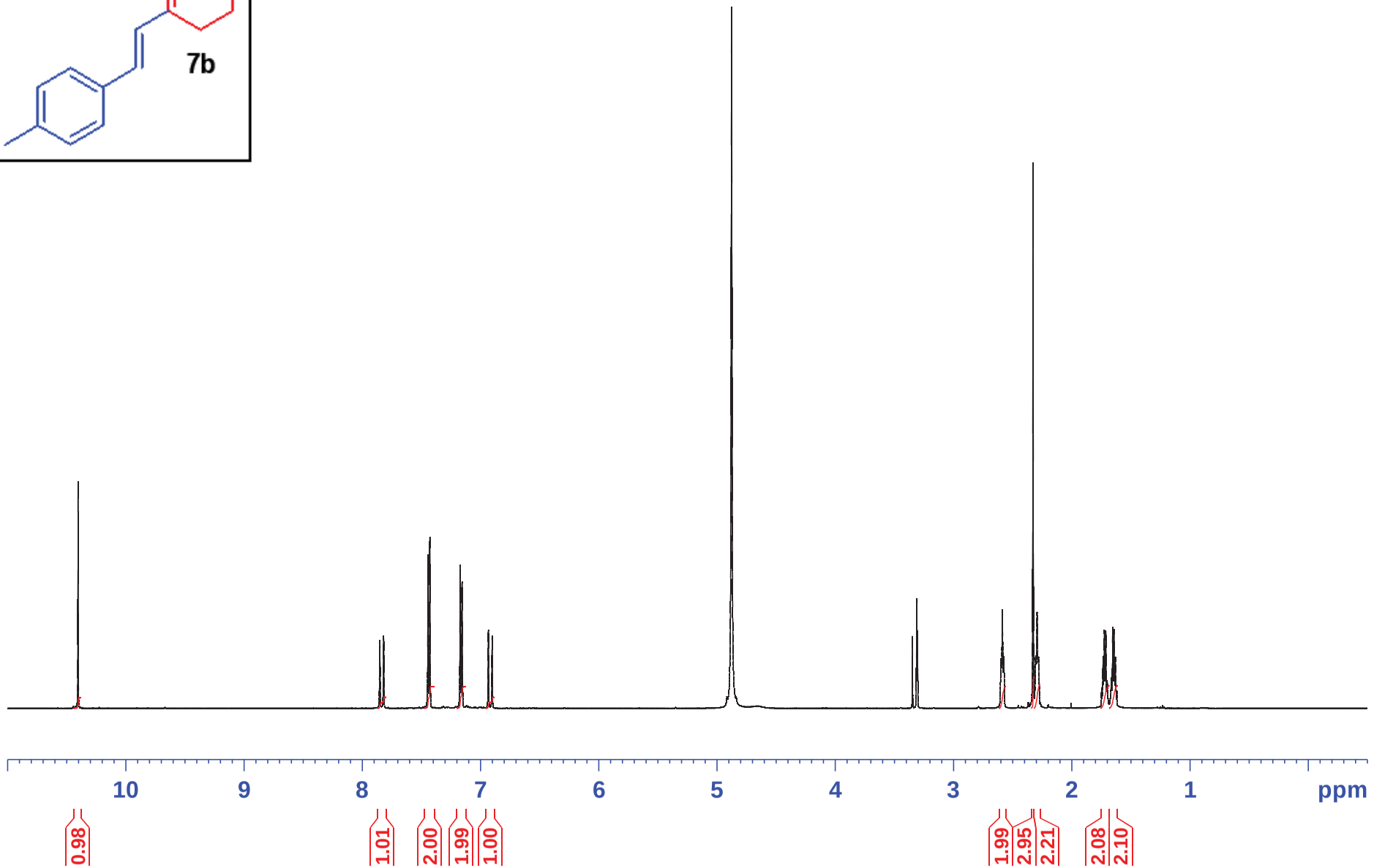


S60

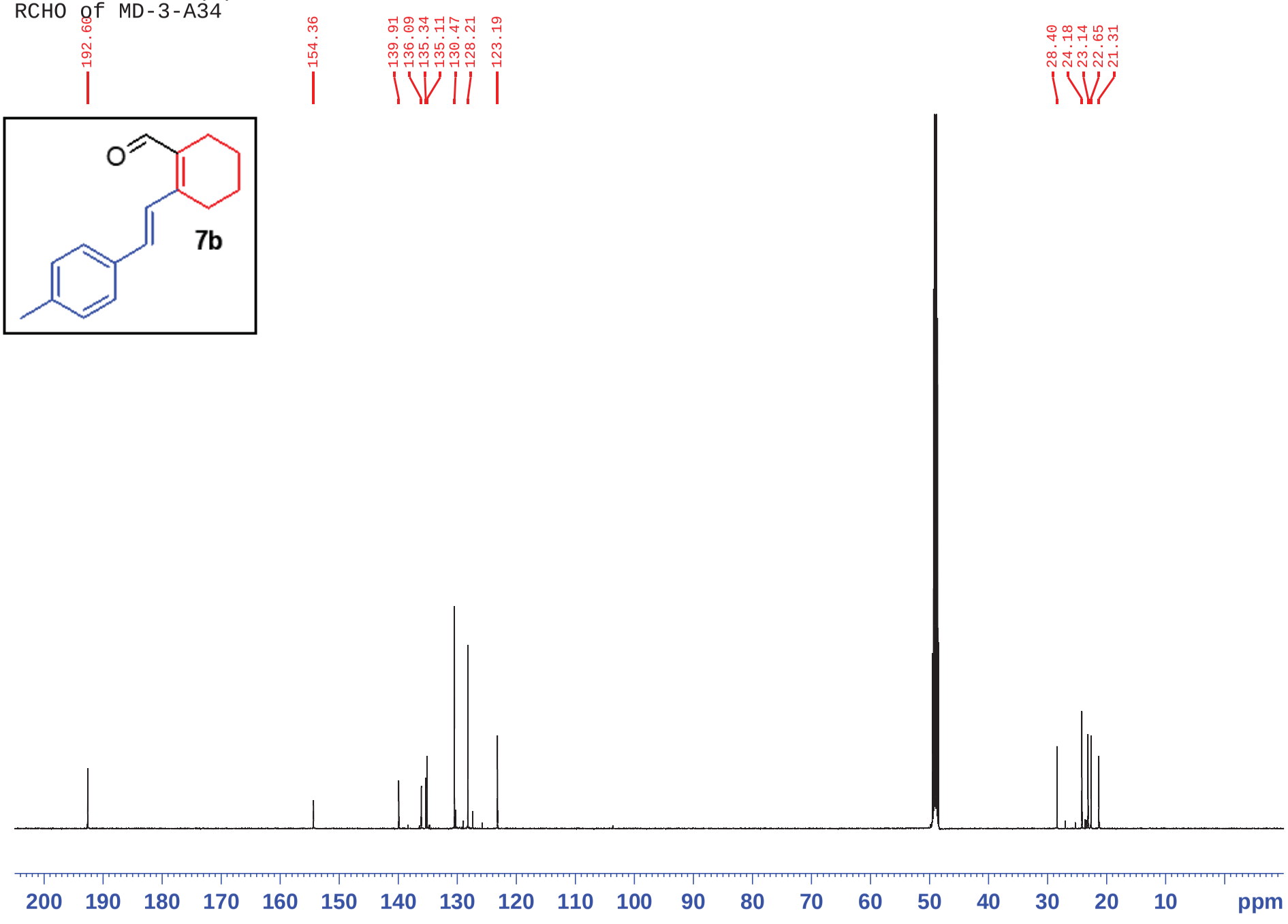
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-71(4)
RCHO of MD-3-A34

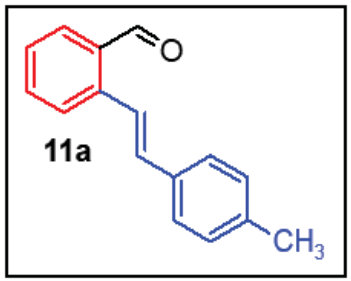


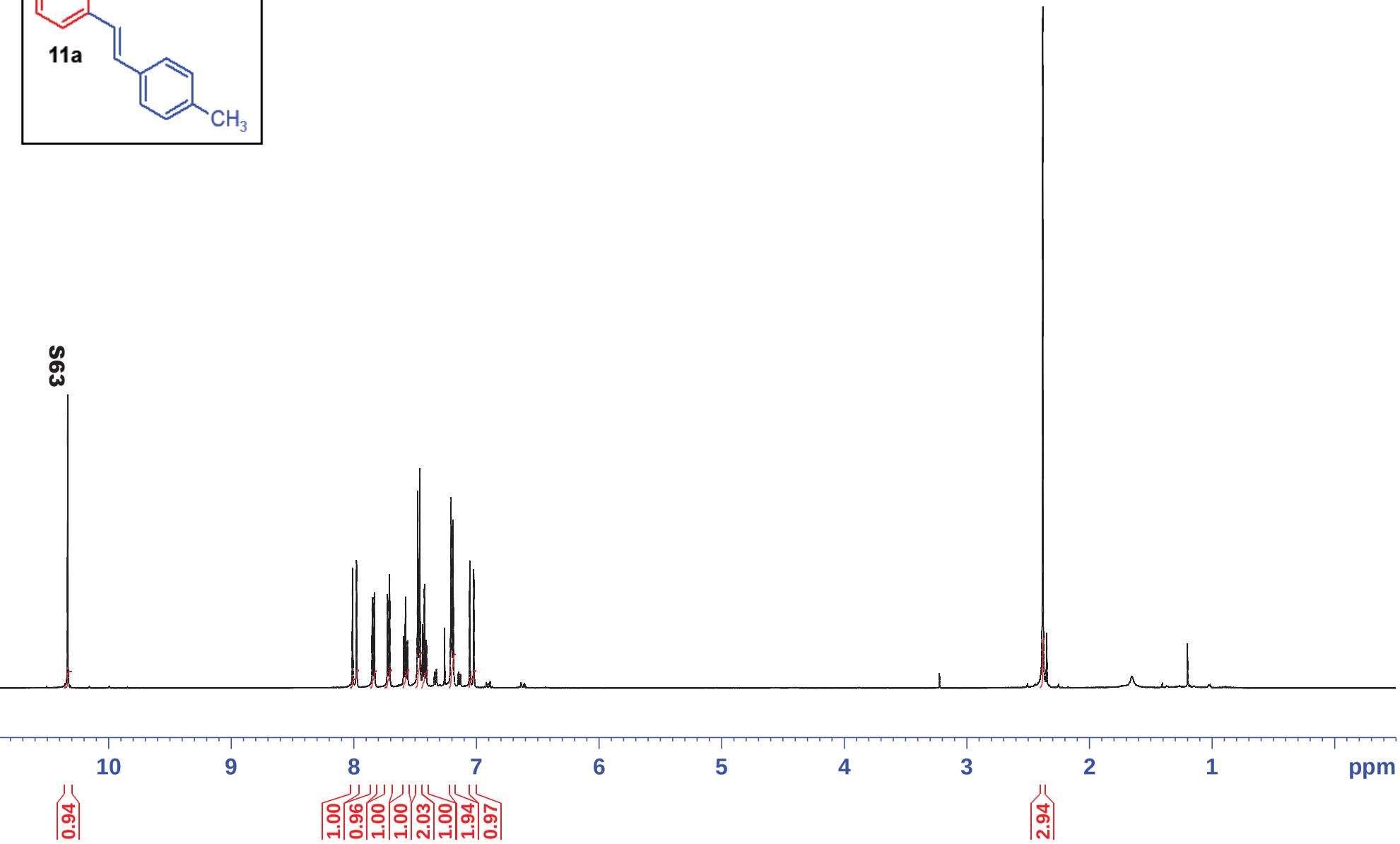


RPD-FGF23-A-71(4)
RCHO of MD-3-A34



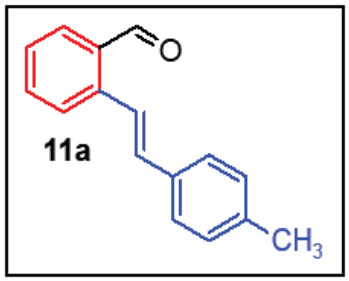
AEL-FGF23-A-141(2)
RCHO of MD-3-A45





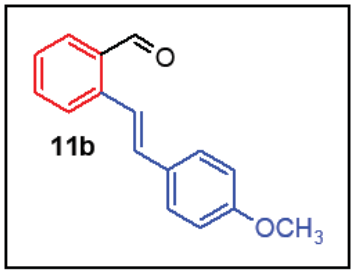
AEL-FGF23-A-141(2)
RCHO of MD-3-A45

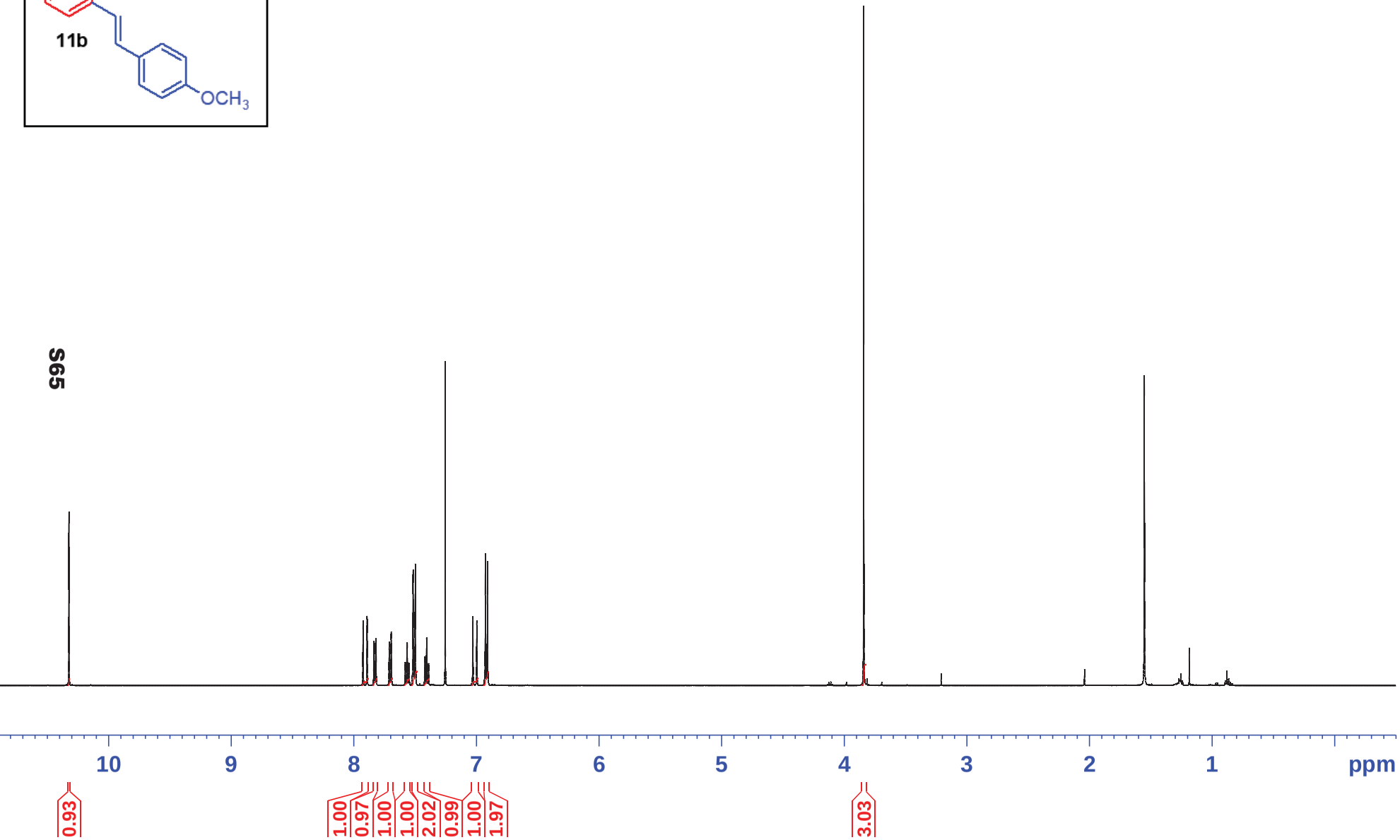
192.09
140.35
138.49
134.26
134.17
133.82
132.98
132.25
129.61
127.53
127.22
127.03
123.74
21.44



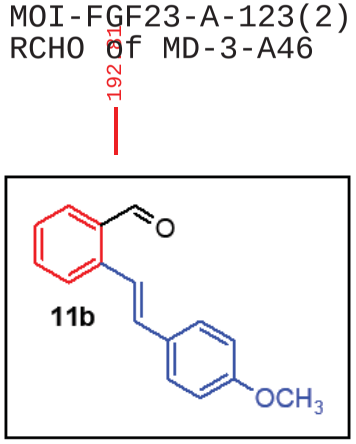
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

MOI-FGF23-A-123(2)
RCHO of MD-3-A46

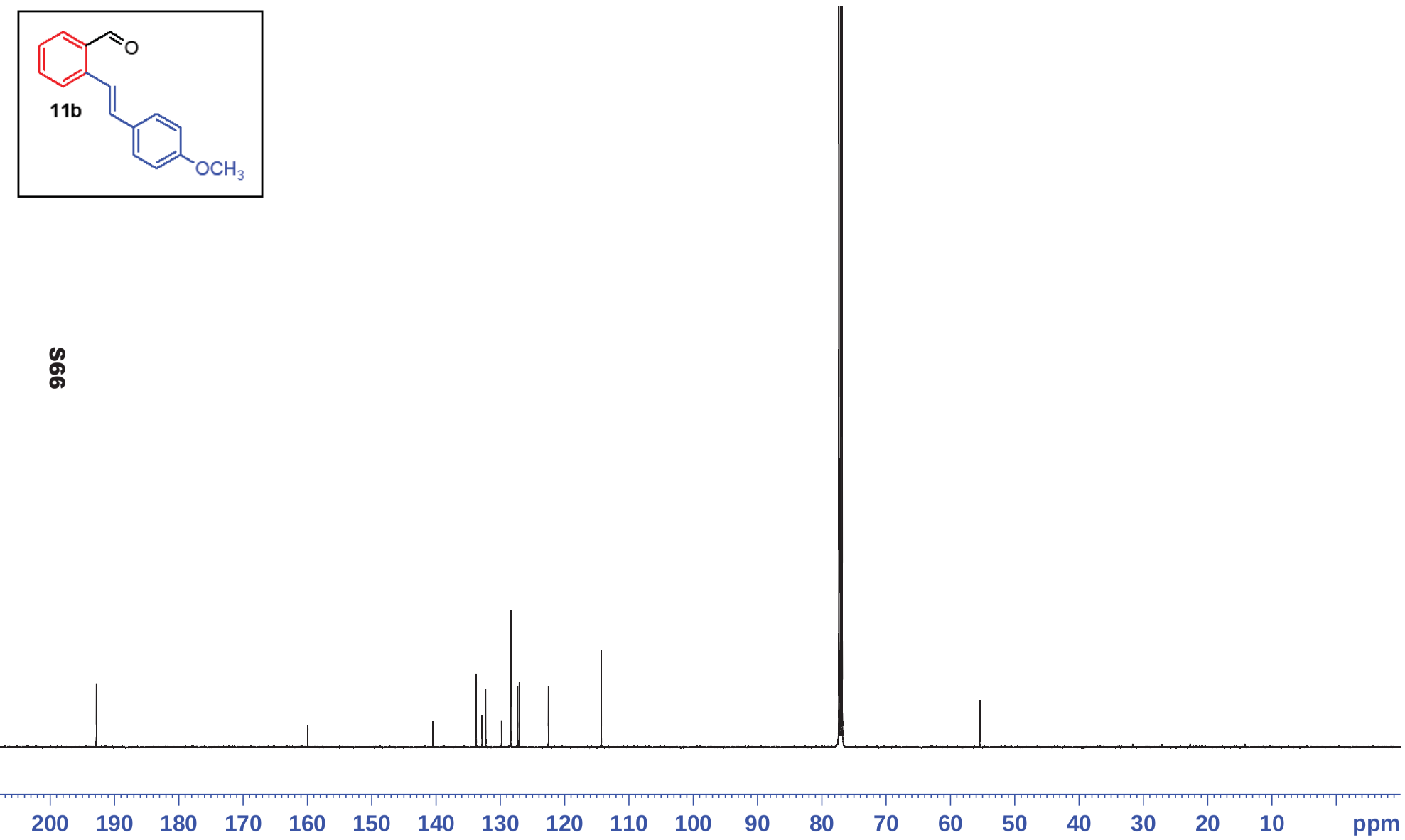




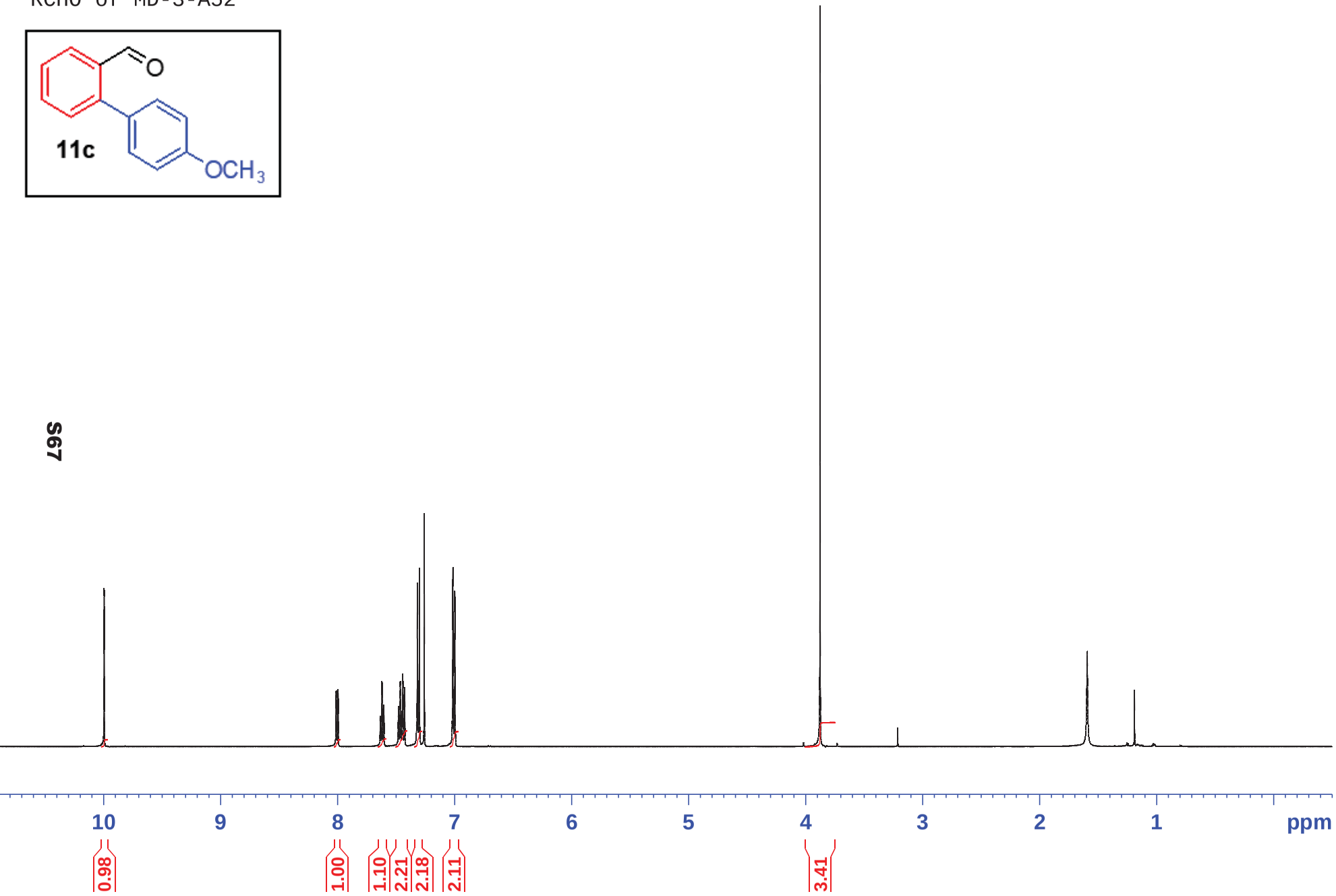
MOI-FGF23-A-123(2)
RCHO of MD-3-A46



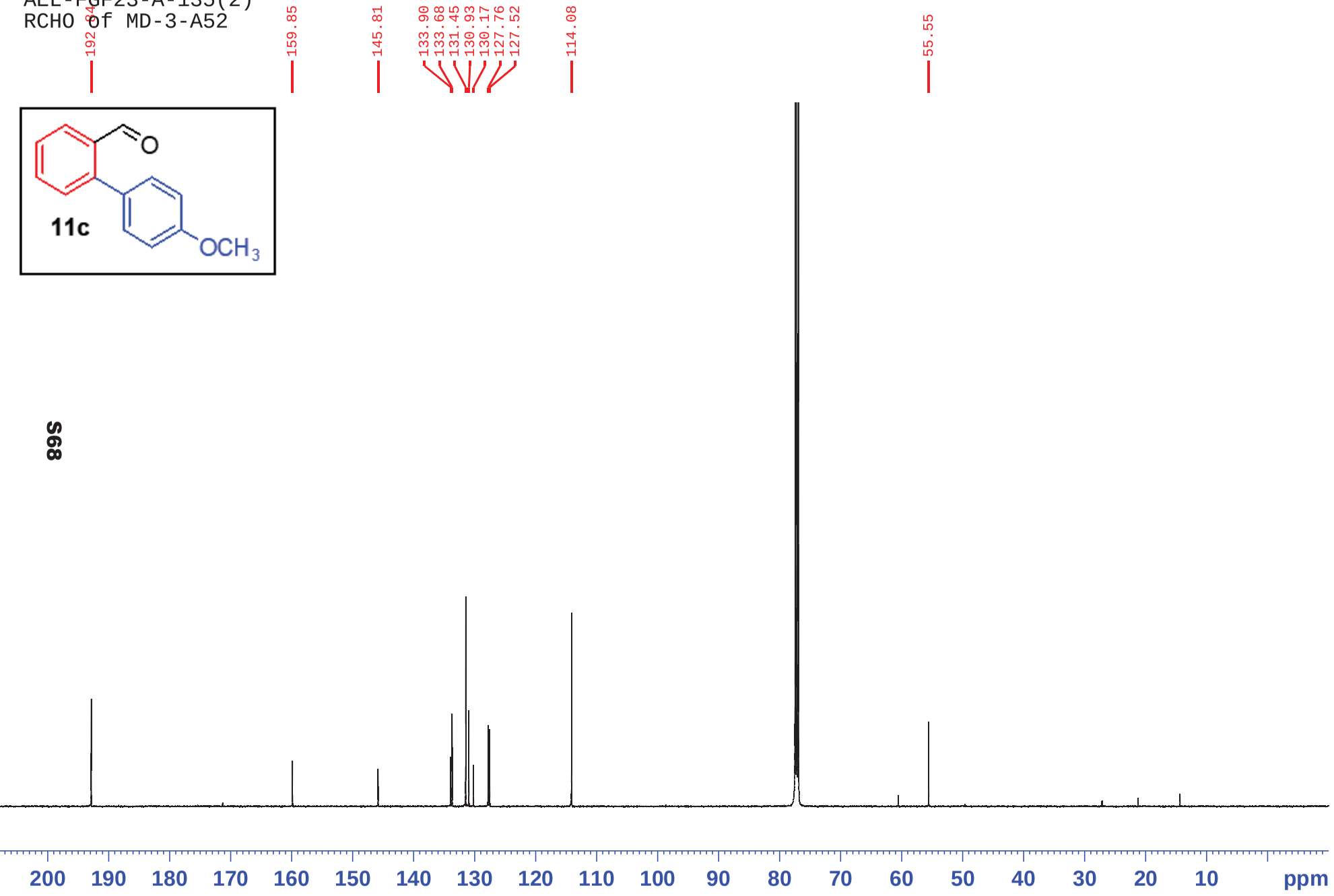
S66



AEL-FGF23-A-135(2)
RCHO of MD-3-A52

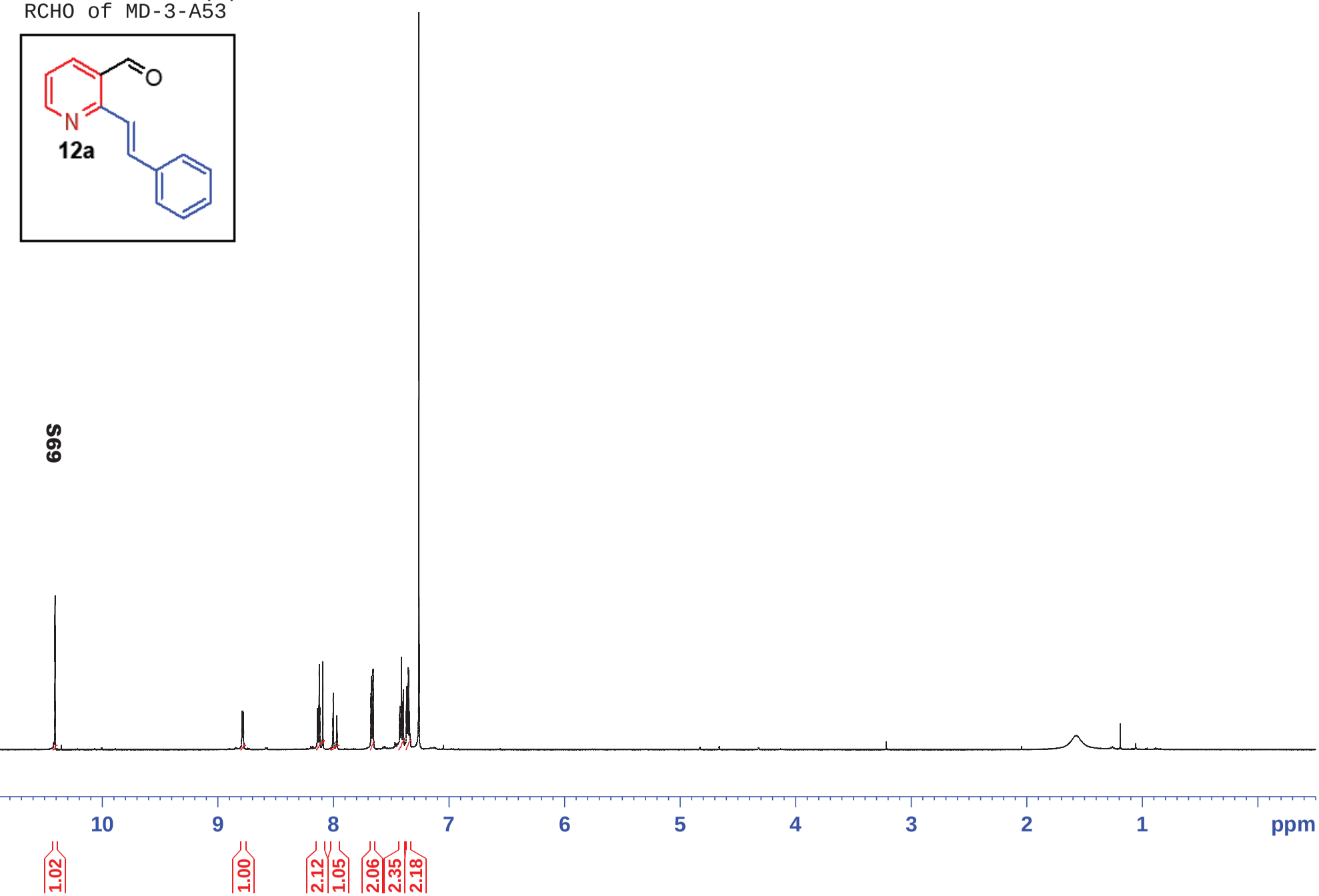


AEL-FGF23-A-135(2)
RCHO of MD-3-A52



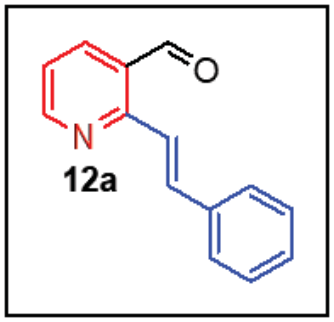
MOI-FGF23-A-55(3)
RCHO of MD-3-A53

69S



MOI-FGF23-A-55(2)
RCHO of MD-3-A53

191.37
156.22
153.43
139.82
138.60
136.43
129.35
128.98
127.96
127.93
122.48
122.31

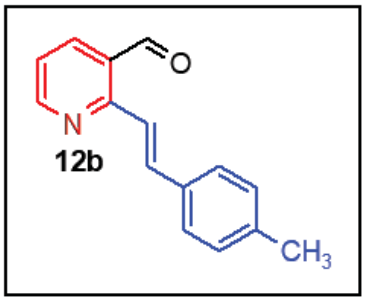


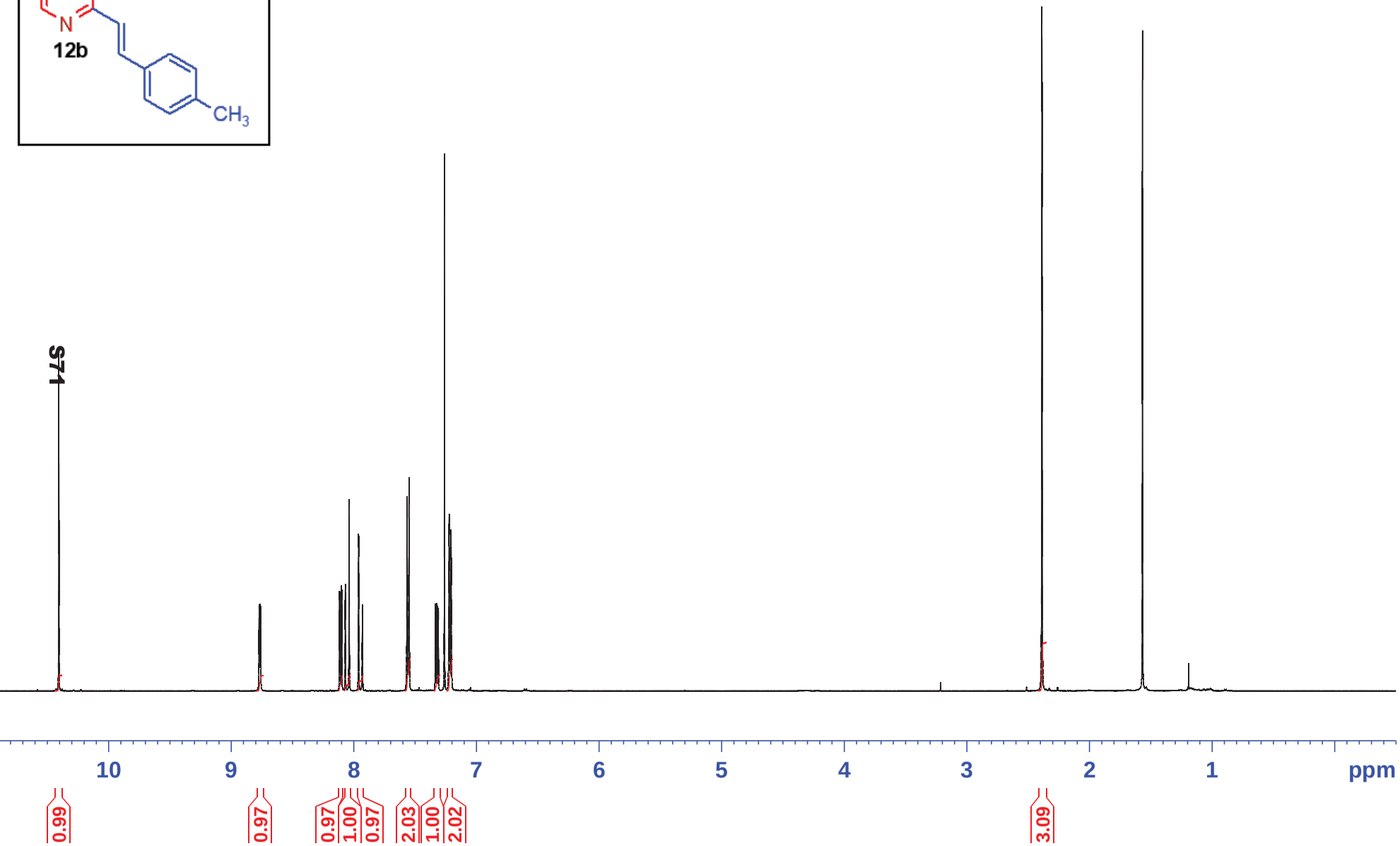
12a

S70

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

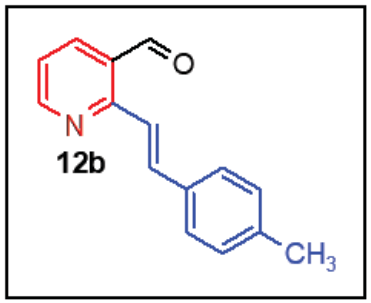
MOI-FGF23-A-115(3)
RCHO of MD-3-A54





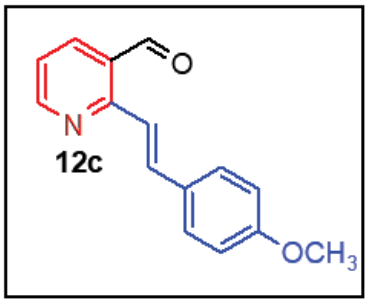
MOI-FGF23-A-115(3)
RCHO of MD-3-A54

191.44
156.56
153.61
139.52
138.33
133.73
129.70
127.86
127.76
122.07
121.65
21.57

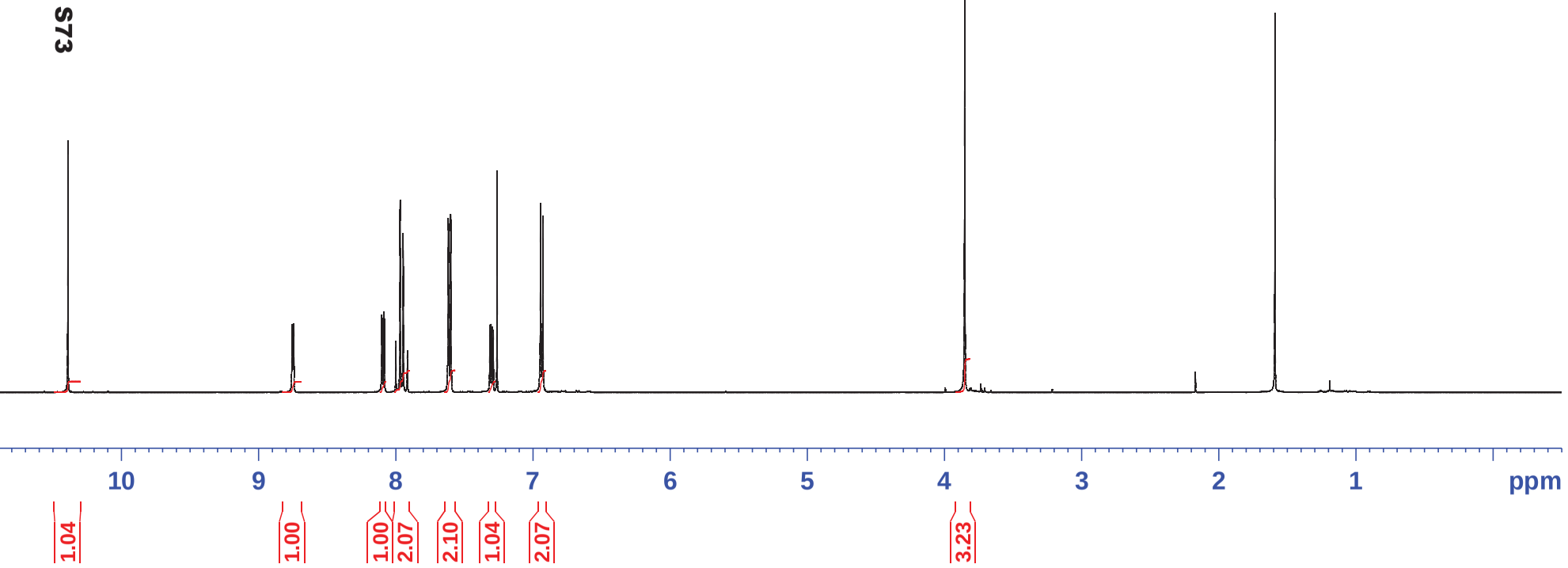


200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

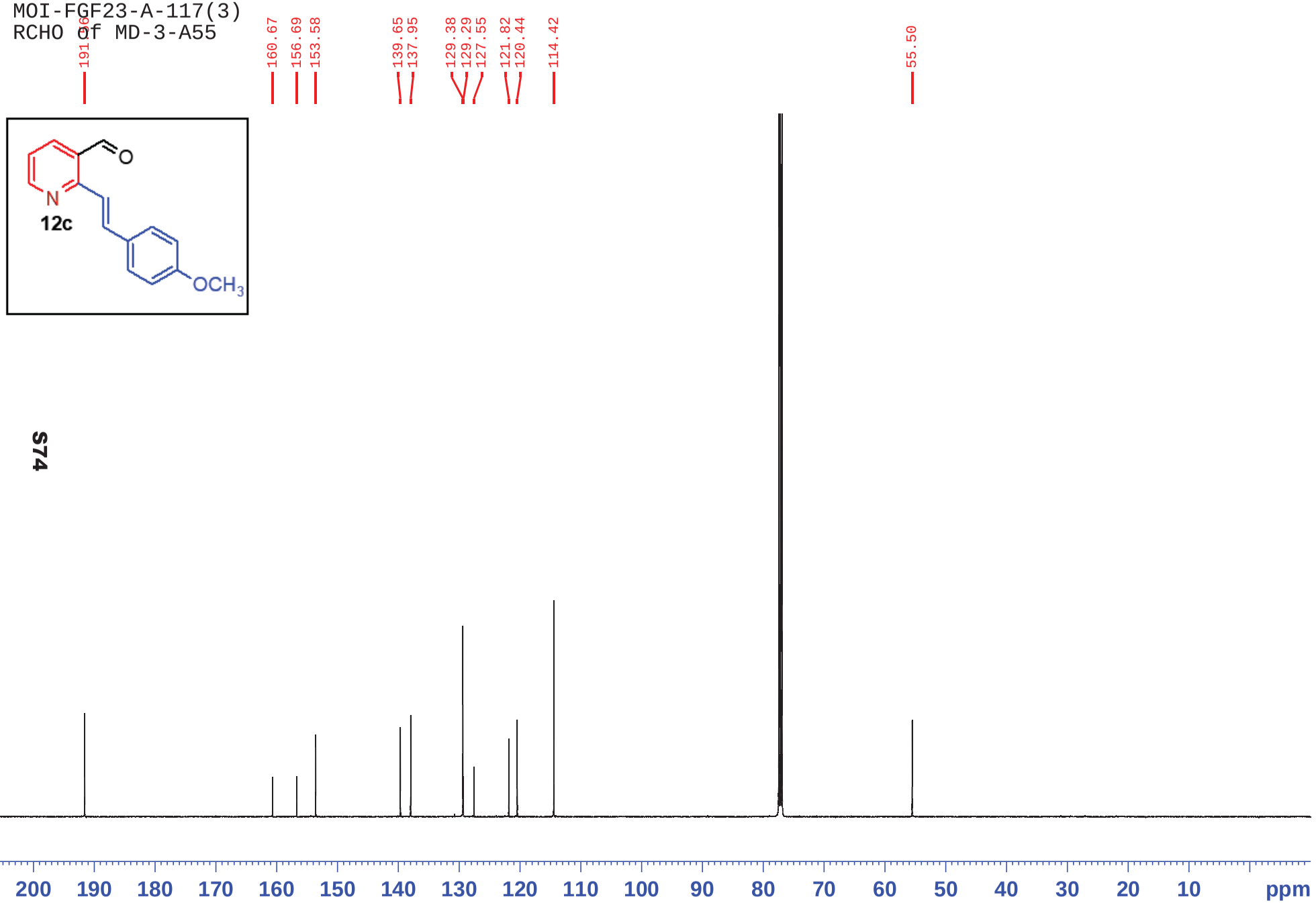
MOI-FGF23-A-117(3)
RCHO of MD-3-A55



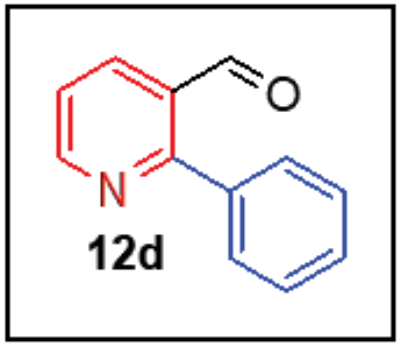
S73

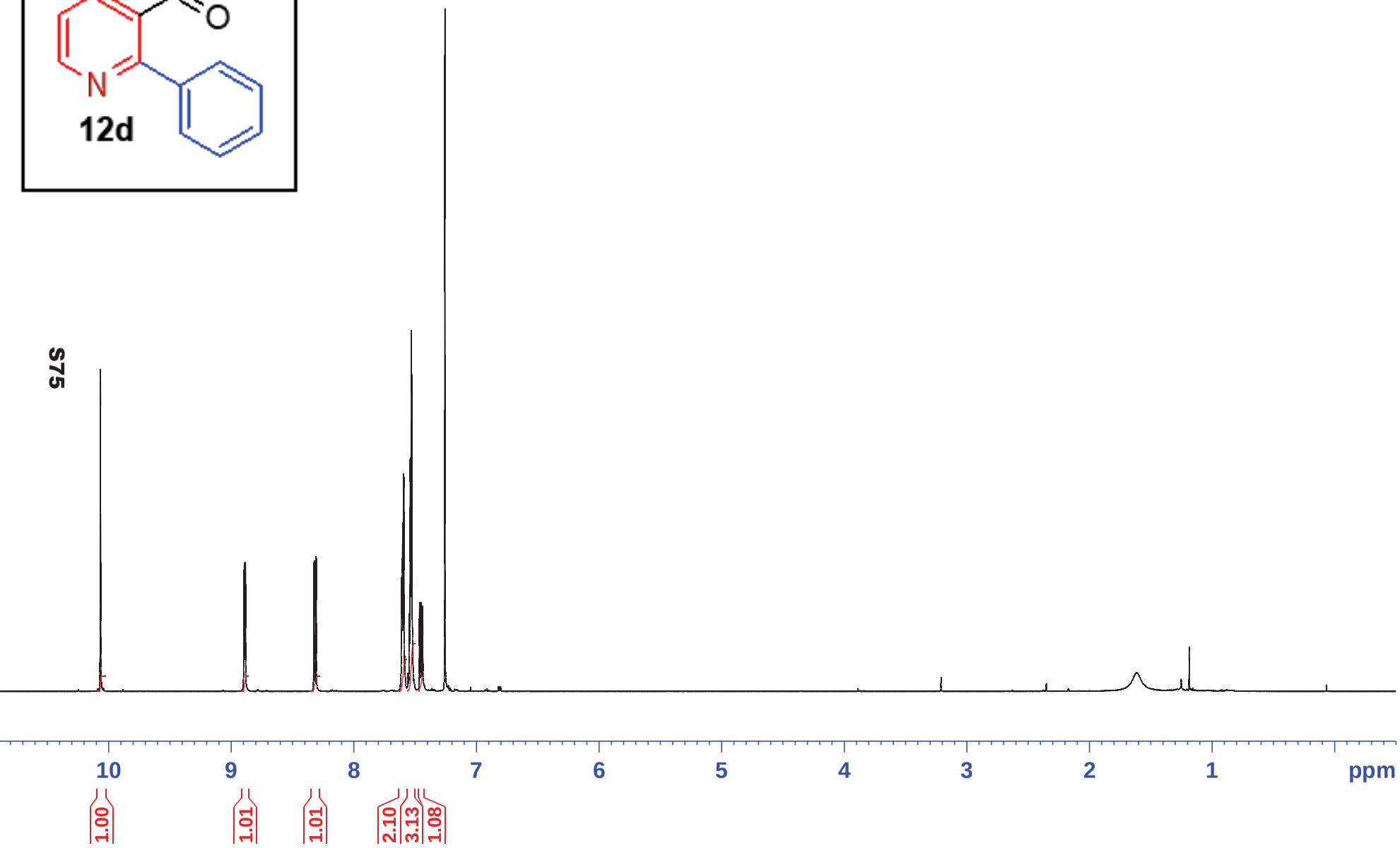


MOI-FGF23-A-117(3)
RCHO of MD-3-A55



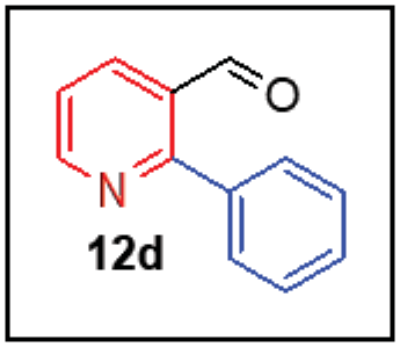
MOI-FGF23-A-57(2)
RCHO of MD-3-A56





MOI-FGF23-A-57(3)
RCHO of MD-3-A56

191.80
162.40
153.59
137.16
136.08
130.54
129.77
129.69
128.80
122.70

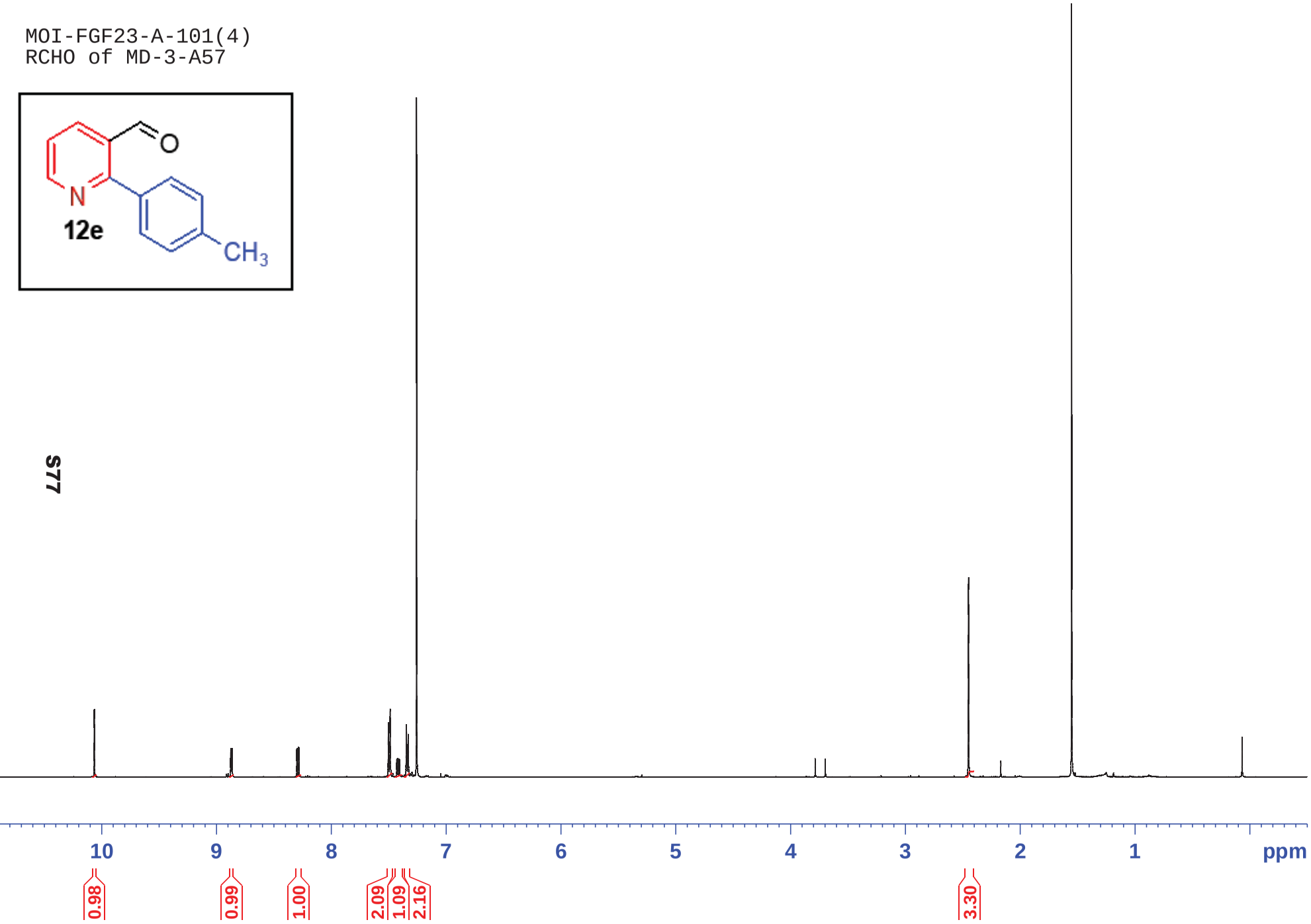


S76

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

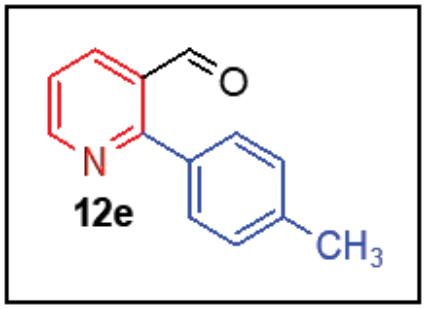
MOI-FGF23-A-101(4)
RCHO of MD-3-A57

S77



MOI-FGF23-A-101(4)
RCHO of MD-3-A57

192.55
162.46
153.60
139.98
136.04
134.40
130.54
129.61
129.54
122.42
21.50

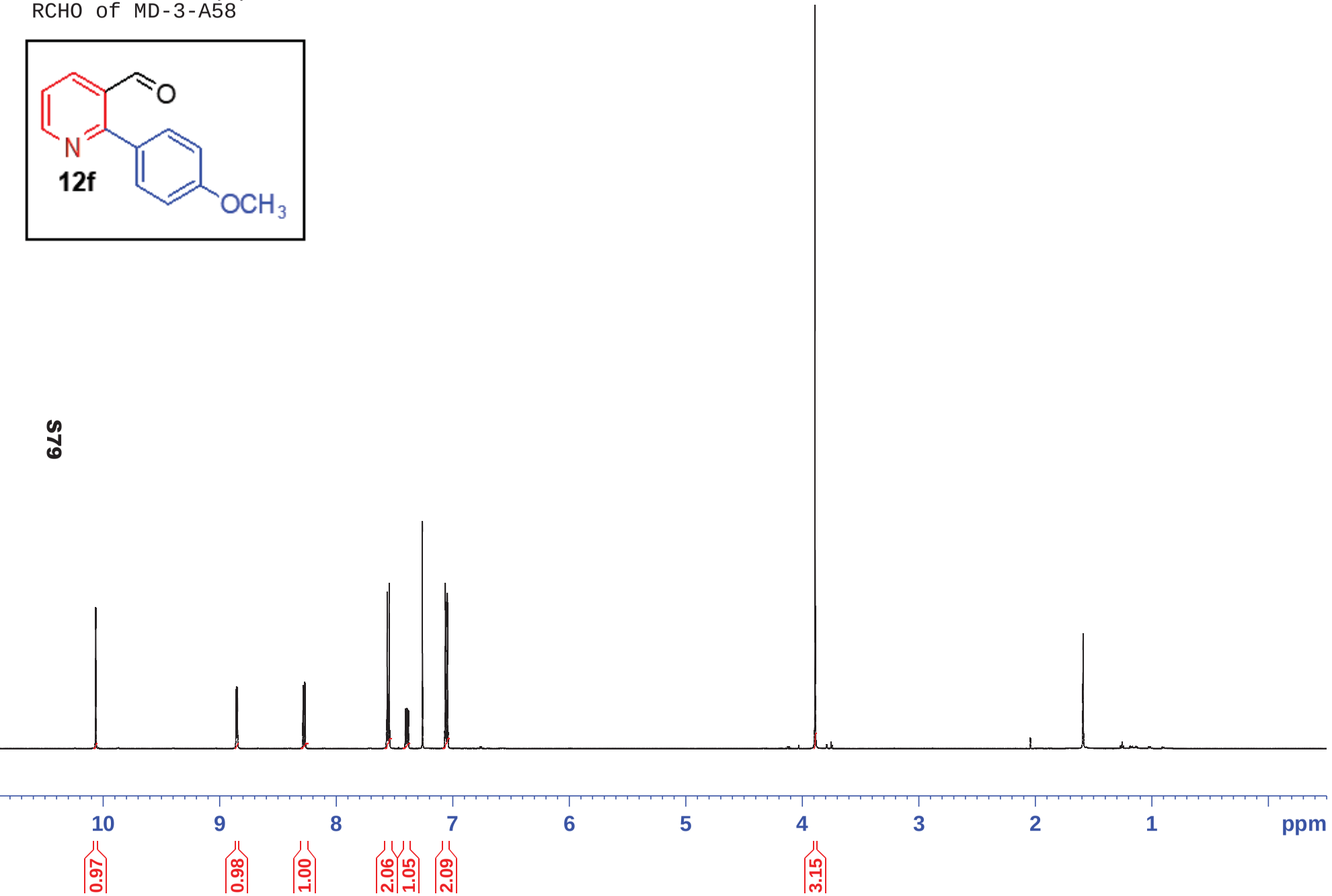


S78

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

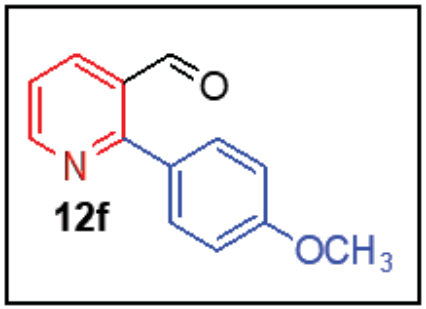
MOI-FGF23-A-61(3)
RCHO of MD-3-A58

S79



MOI-A-61(3)
RCHO of MD-3-A58

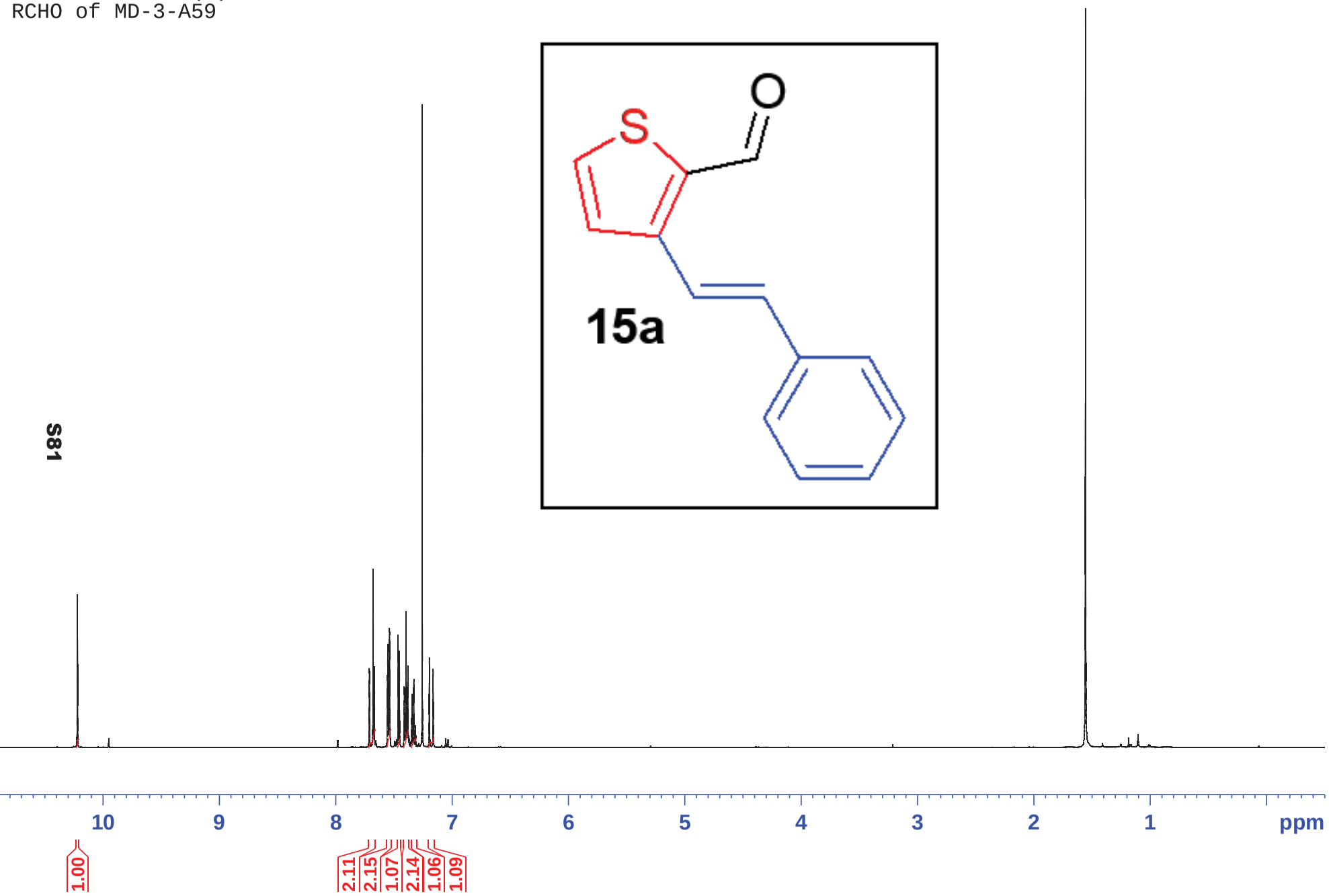
192.08
161.98
161.11
153.56
136.13
132.08
129.71
129.44
122.15
114.33
55.61



S80

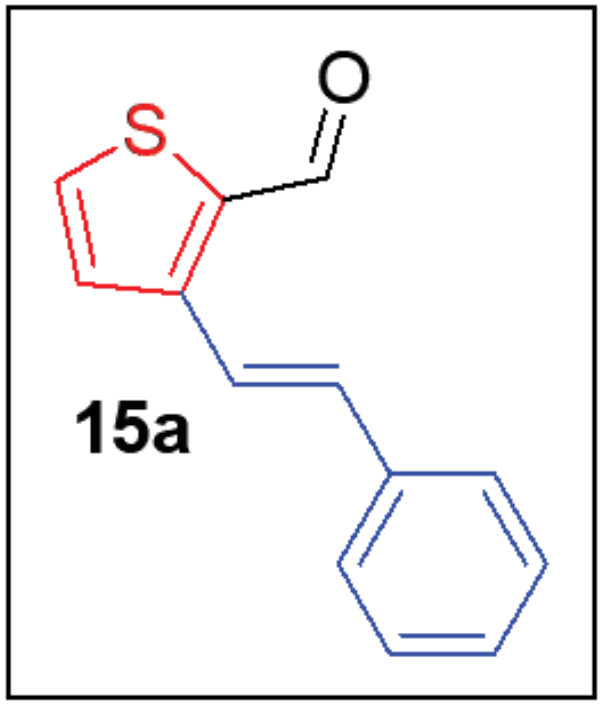
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

MOI-FGF23-A-77(5)
RCHO of MD-3-A59



MOI-FGF23-A-77(5)
RCHO of MD-3-A59

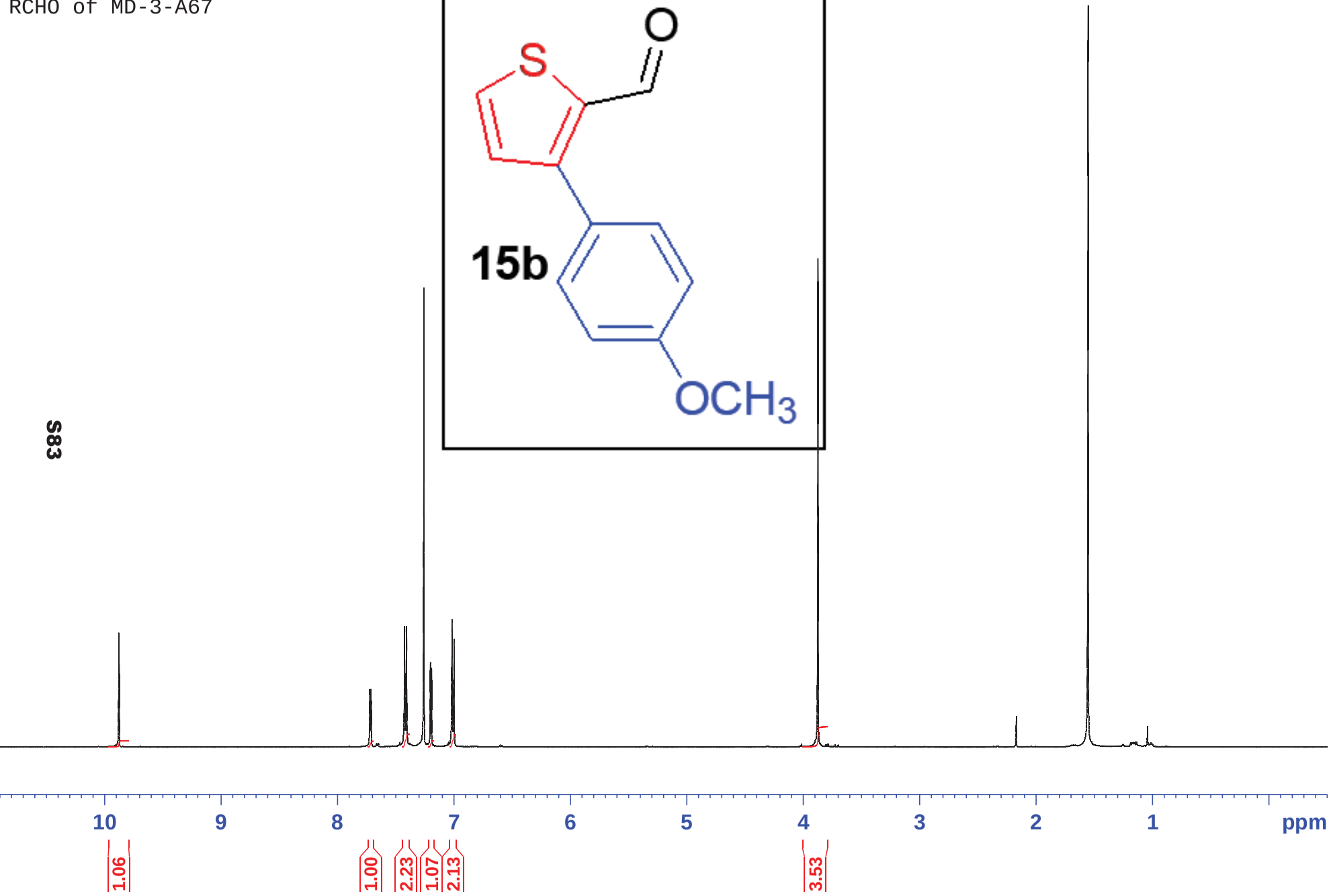
182.18
146.88
137.67
136.40
134.89
134.52
129.05
128.96
127.11
126.94
119.46



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

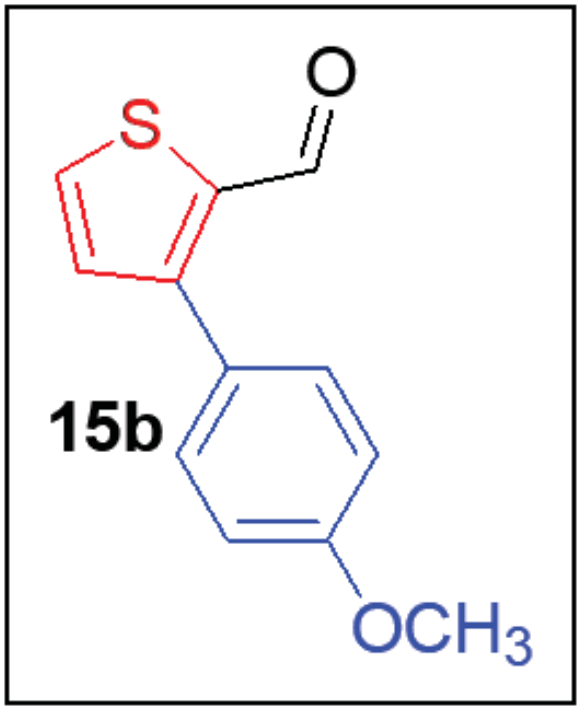
MOI-FGF23-A-107(3)
RCHO of MD-3-A67

S83



MOI-FGF23-A-107(3)
RCHO of MD-3-A67

184.00
160.36
151.49
137.97
134.23
130.99
130.69
126.57
114.45
55.57

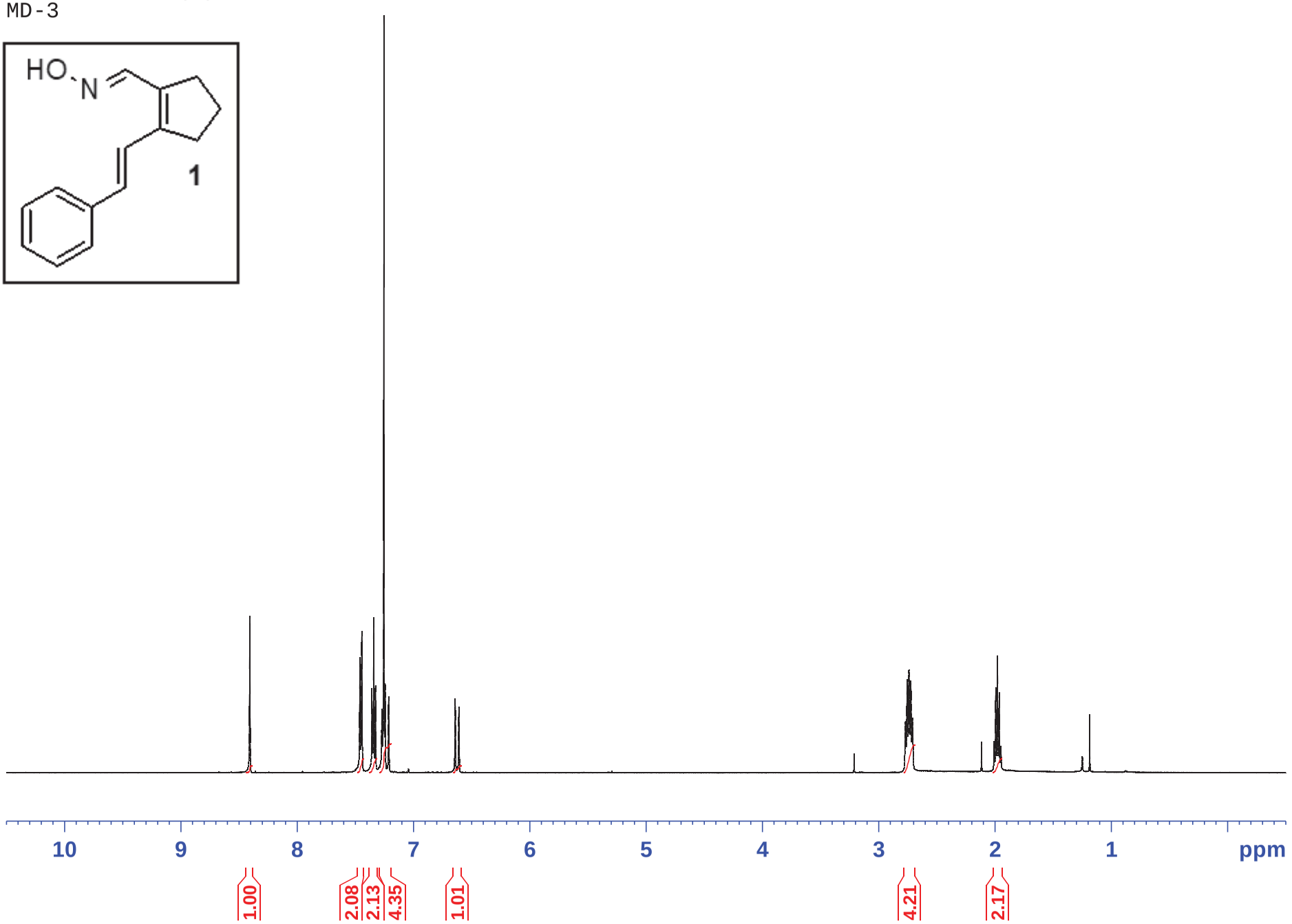


S84

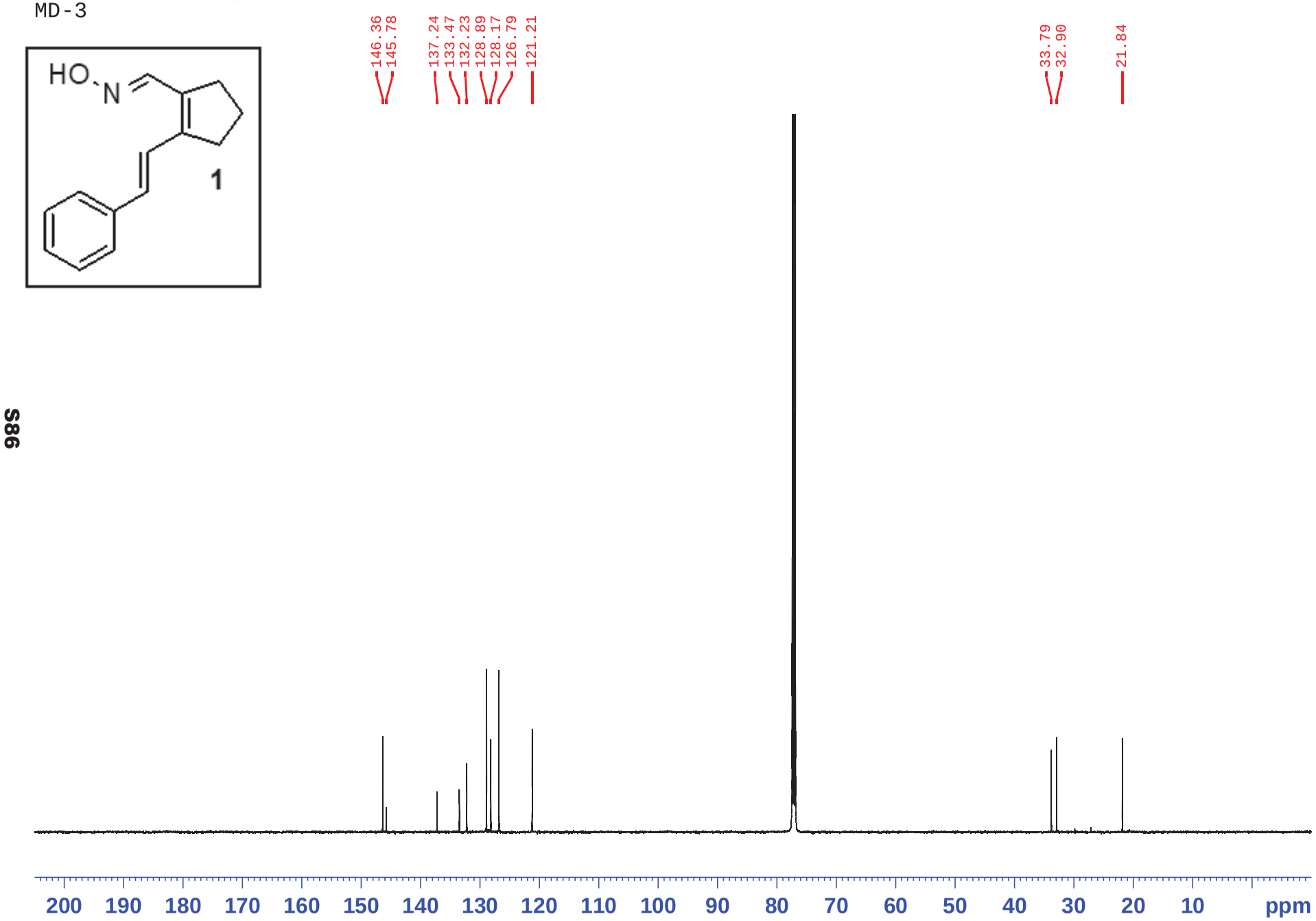
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

JWC-FGF23-A71(2)
MD-3

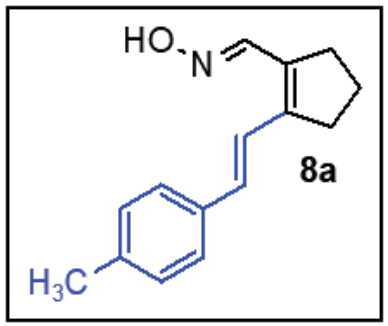
S85

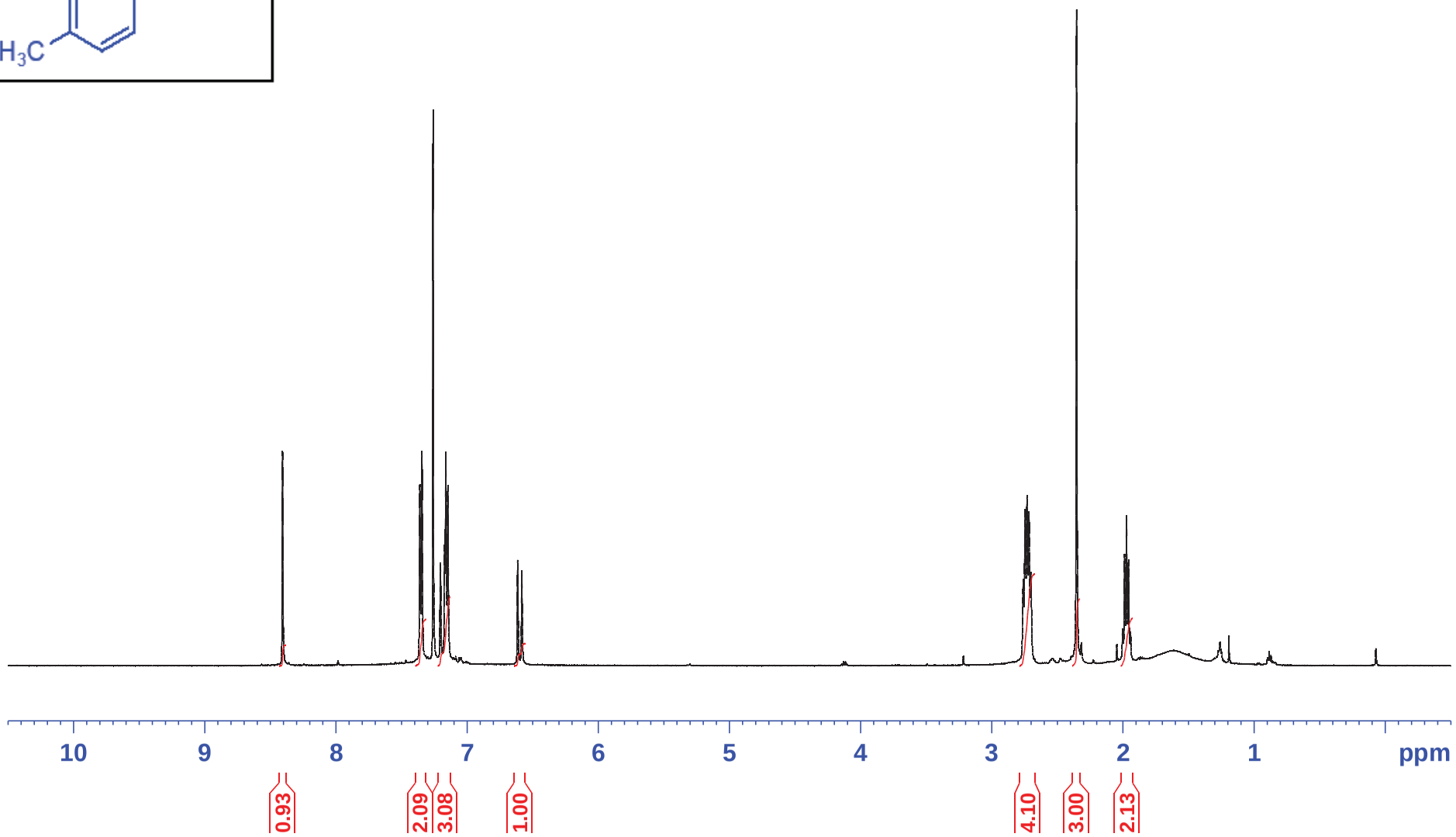


JWC-FGF23-A71(2)
MD-3

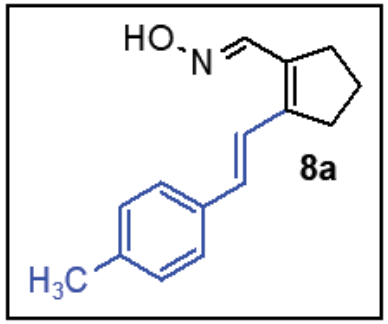


RPD-FGF23-A-35(3)
MD-3-A8





RPD-FGF23-A-35(3)
MD-3-A8

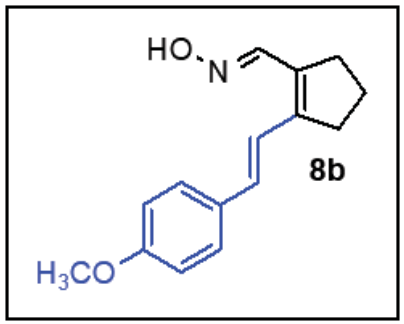


146.41
145.62
138.04
134.36
132.86
131.97
129.49
126.58
120.19
33.65
32.73
21.71
21.29

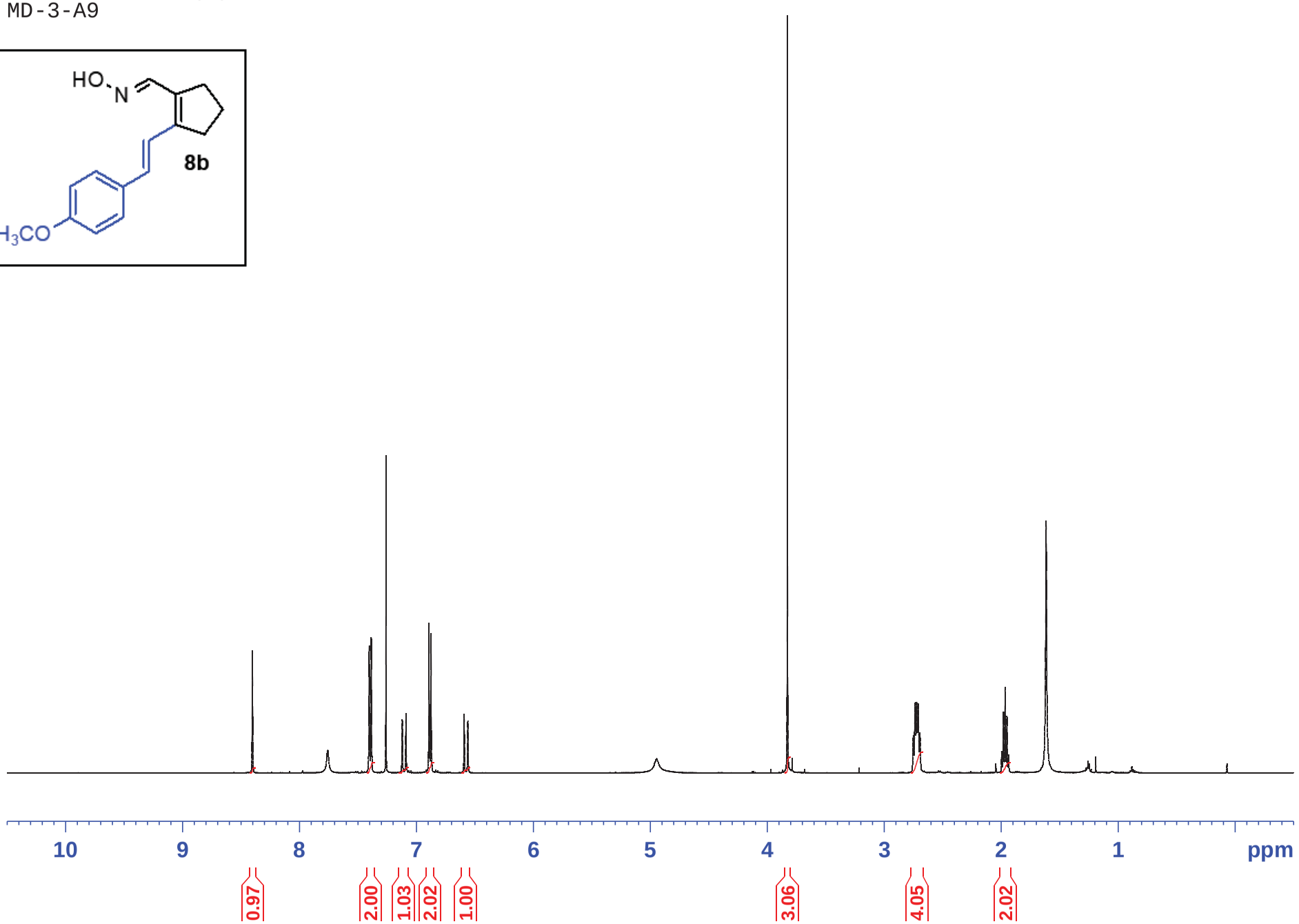
S88

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

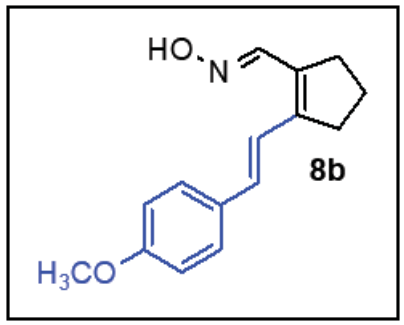
JDC-FGF23-A-21(3)
MD-3-A9



68S



JDC-FGF23-A-21(3)
MD-3-A9

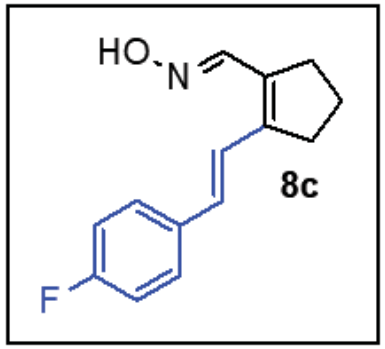


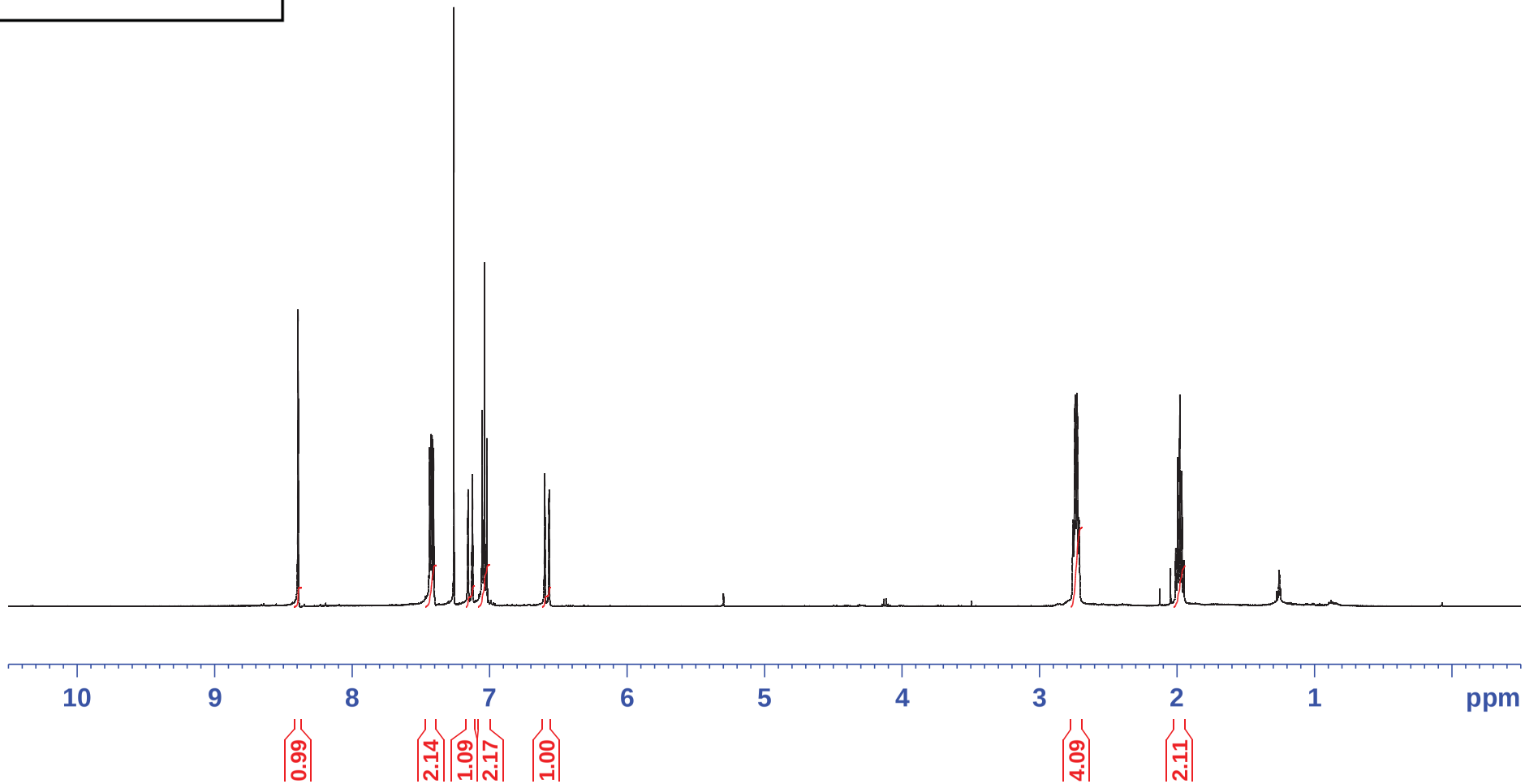
159.75
146.49
145.83
132.42
131.71
130.07
128.06
119.30
114.36
55.48
33.79
32.84
21.84

S90

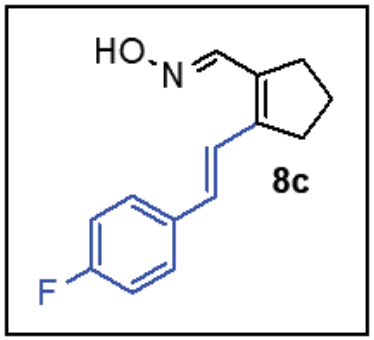
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-33(3)
MD-3-A6





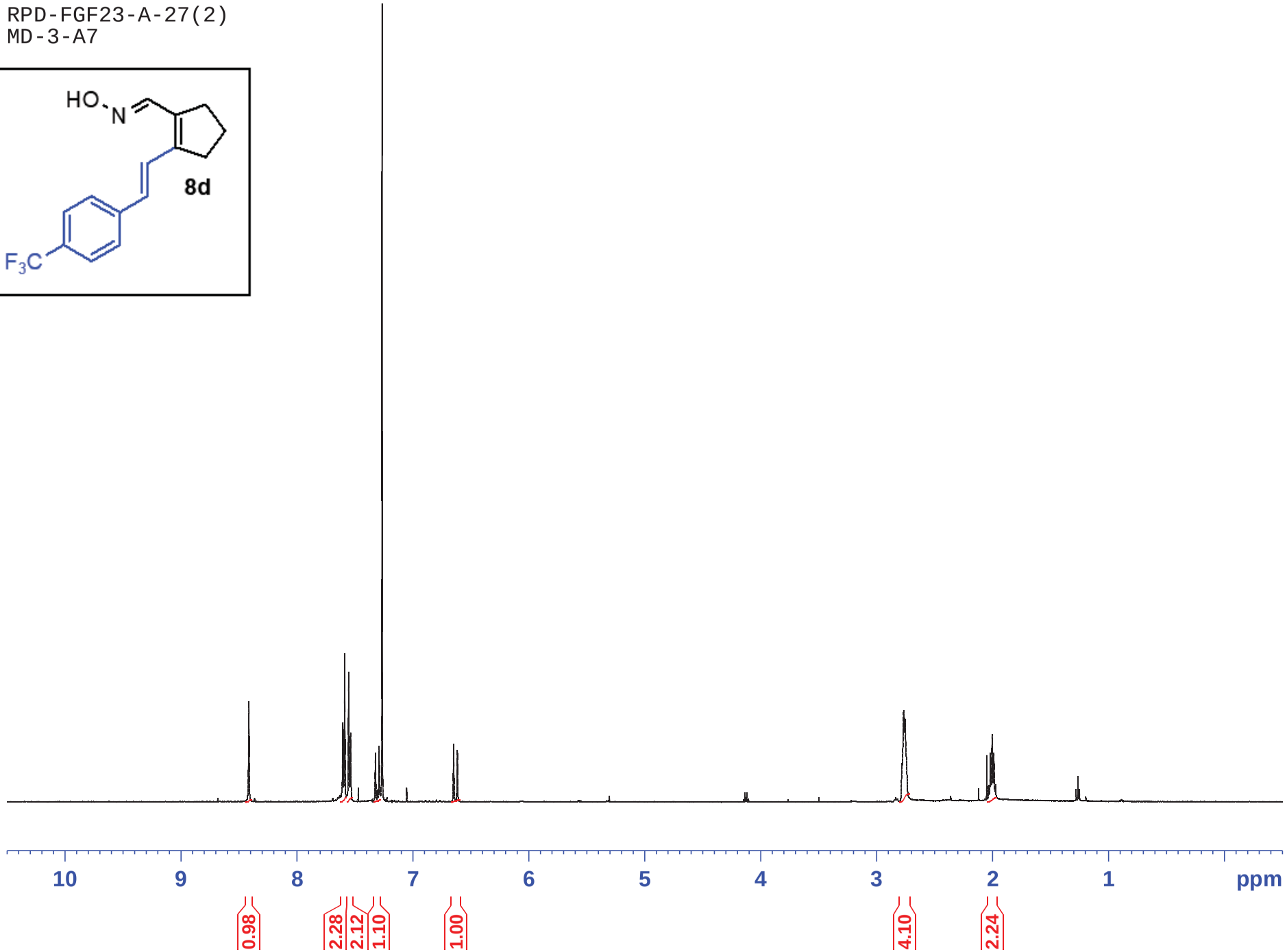
RPD-FGF23-A-33(3)
MD-3-A6



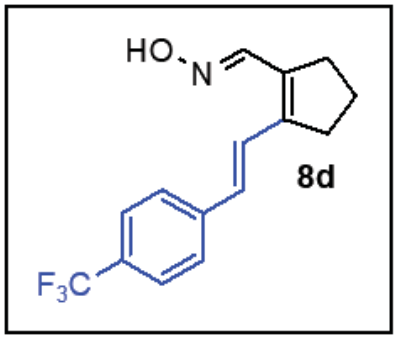
163.68
161.71
146.14
145.59
133.50
133.45
133.43
130.98
128.34
128.28
121.02
121.00
115.98
115.81
77.41
77.15
76.90
33.78
32.90
21.81

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-27(2)
MD-3-A7



RPD-FGF23-A-27(2)
MD-3-A7

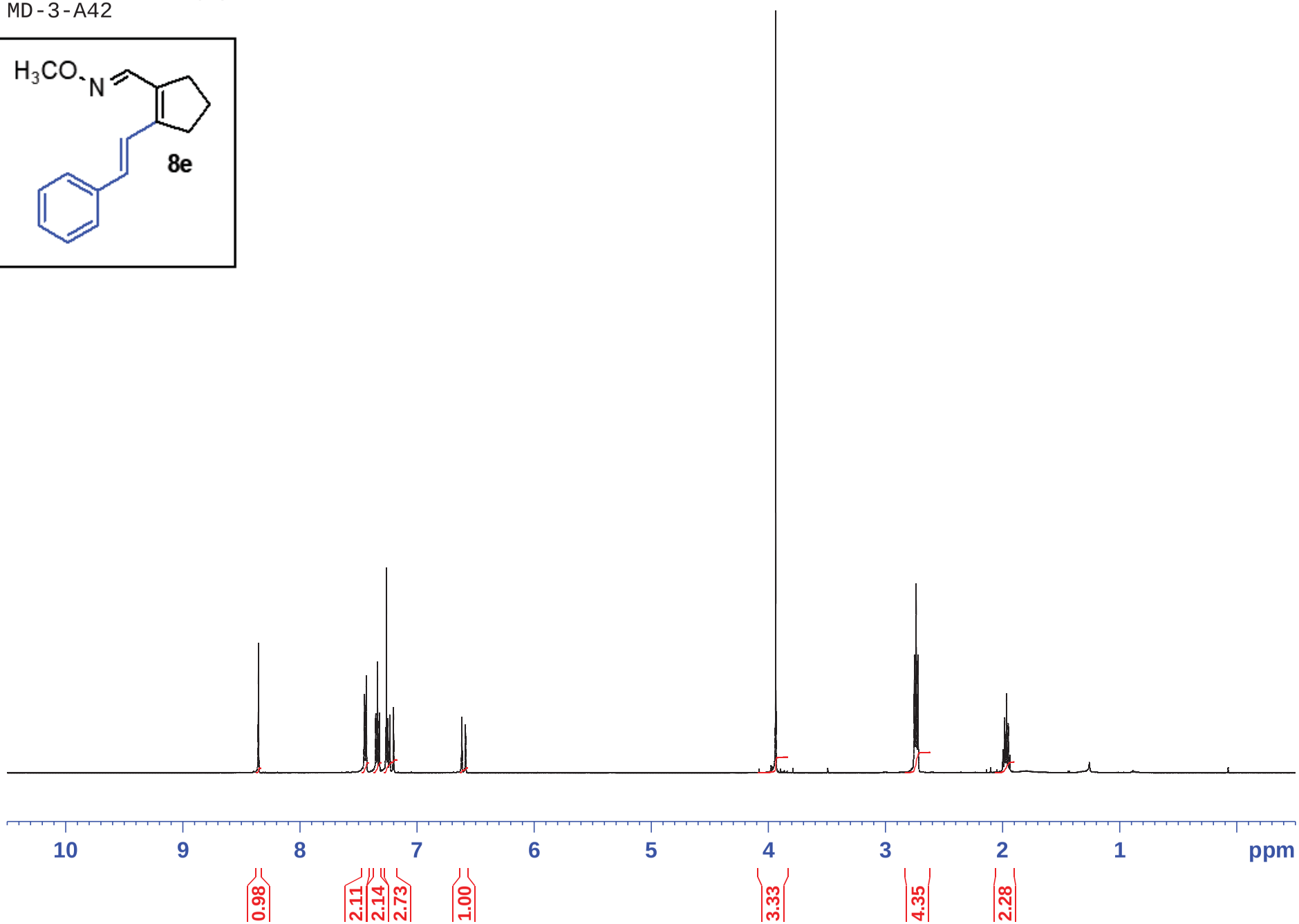


145.92
144.92
140.71
135.26
130.43
129.82
126.83
125.84
123.50
33.71
33.01
21.79

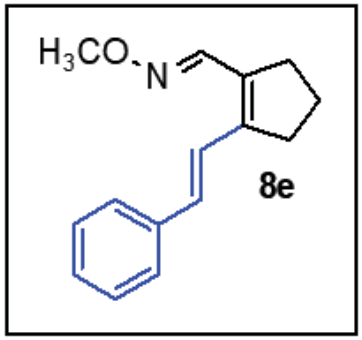
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

JWC-FGF23-A-65(2)
MD-3-A42

S95



JWC-FGF23-A-65(2)
MD-3-A42

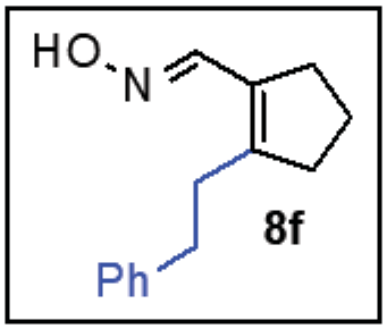


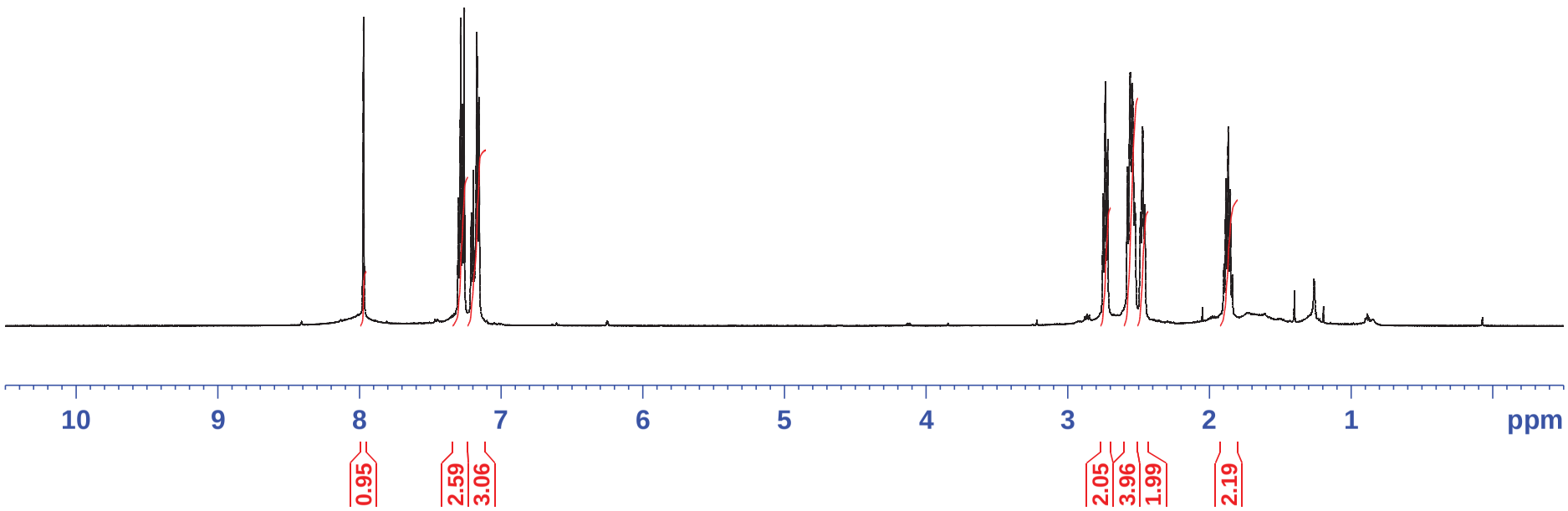
145.09
144.80
137.35
133.89
131.81
128.86
128.05
126.73
121.37
62.03
33.76
33.03
21.90

S96

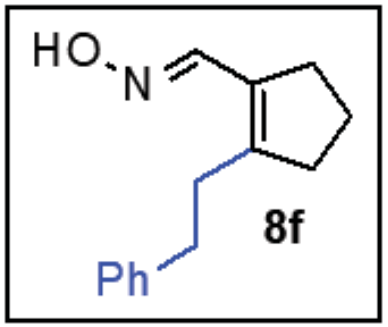
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-31(3)
MD-3-A4





RPD-FGF23-A-31(3)
MD-3-A4

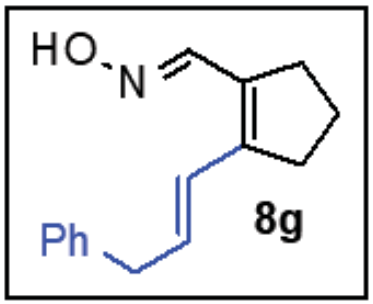


149.89
146.45
141.49
130.00
128.54
128.39
126.22
37.15
34.74
32.08
30.85
21.95

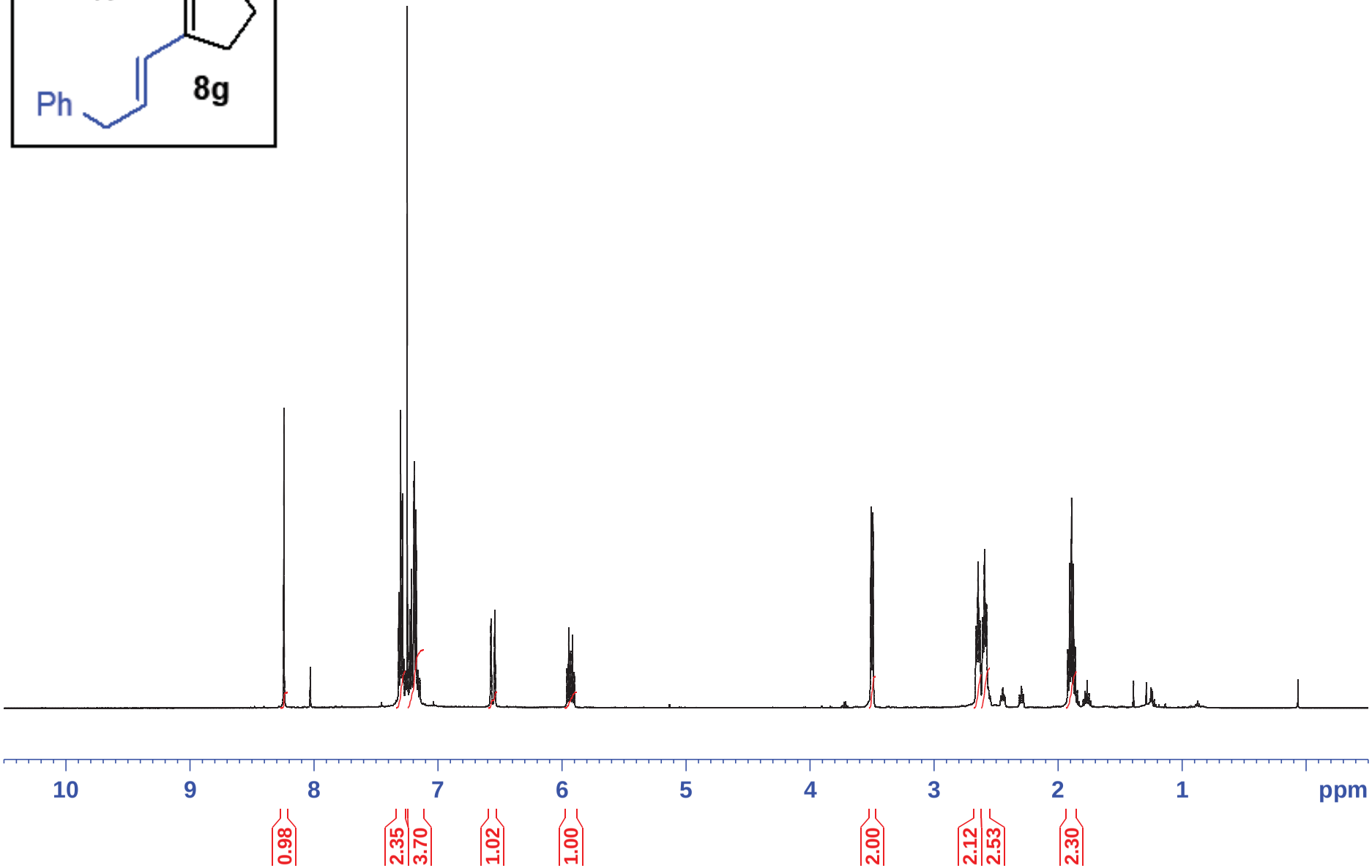
S98

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

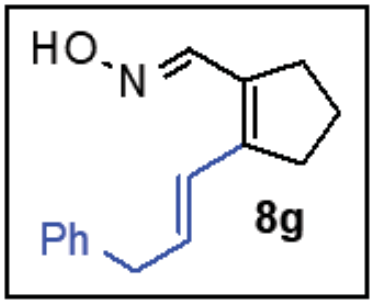
JDC-FGF23-A23(2)
MD-3-A29



66S



JDC-FGF23-A-23(2)
MD-3-A29

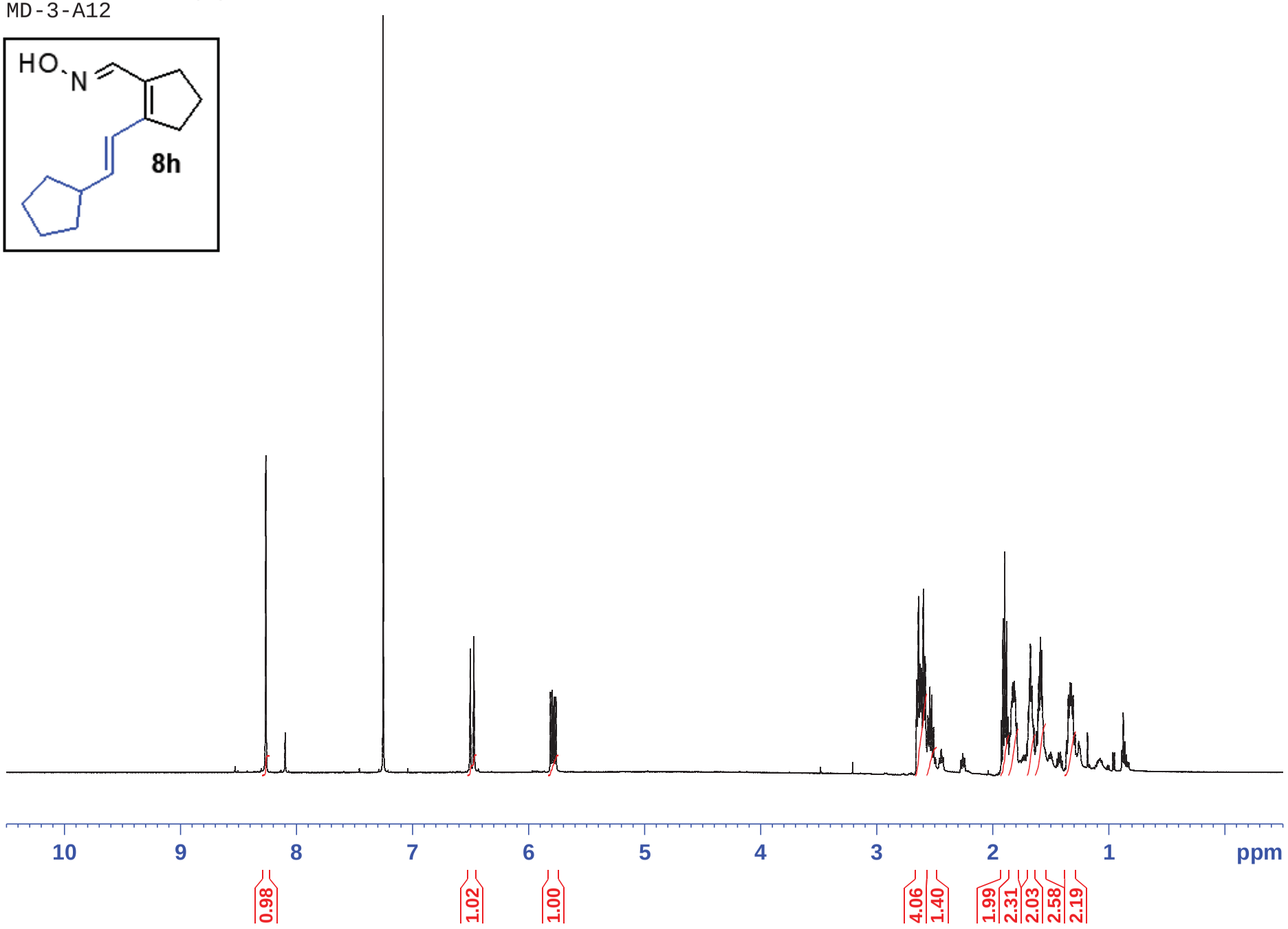


146.17
145.80
139.86
133.69
131.38
128.76
128.71
128.51
126.45
124.23
39.78
33.97
32.63
21.73

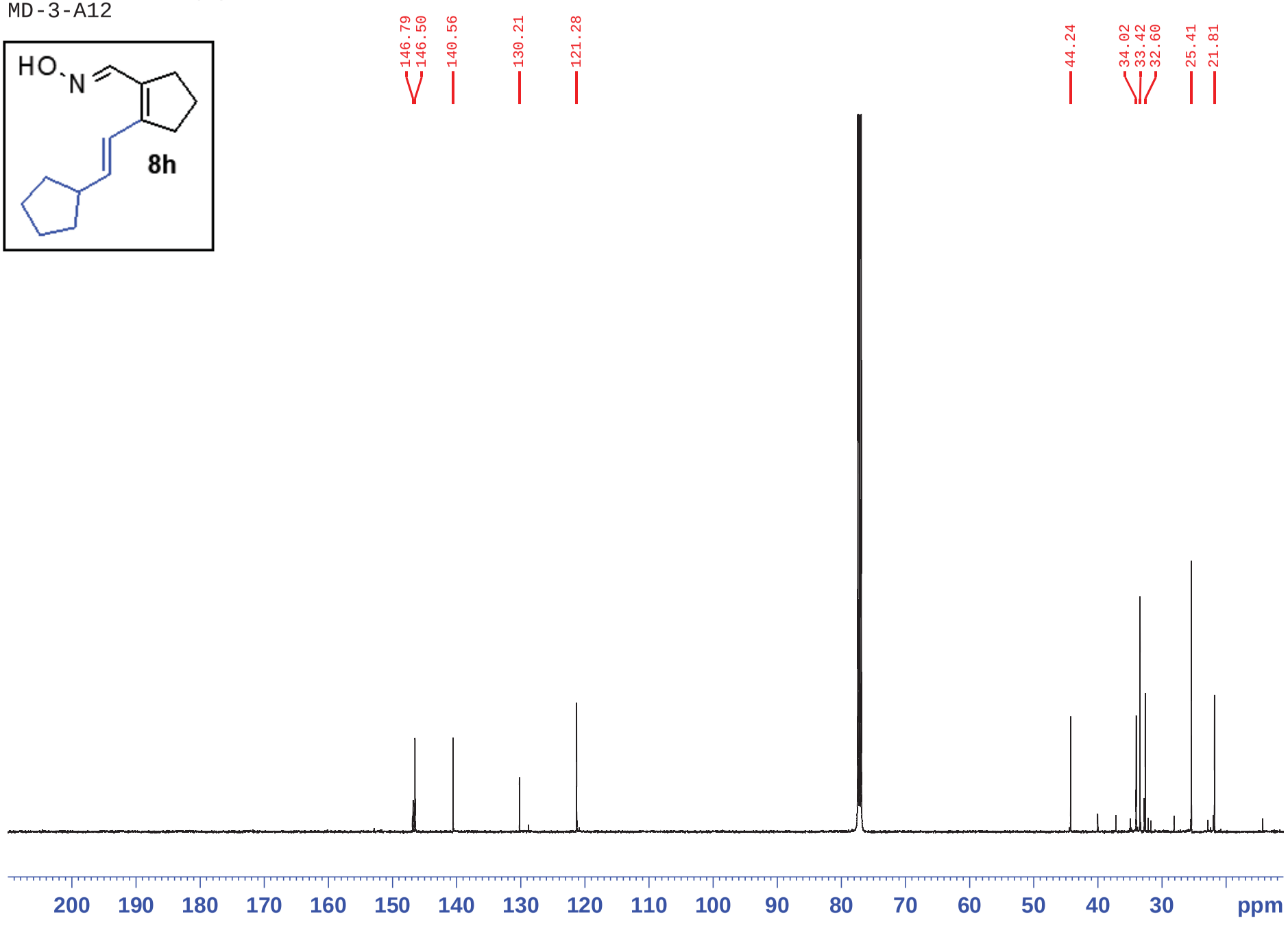
S100

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

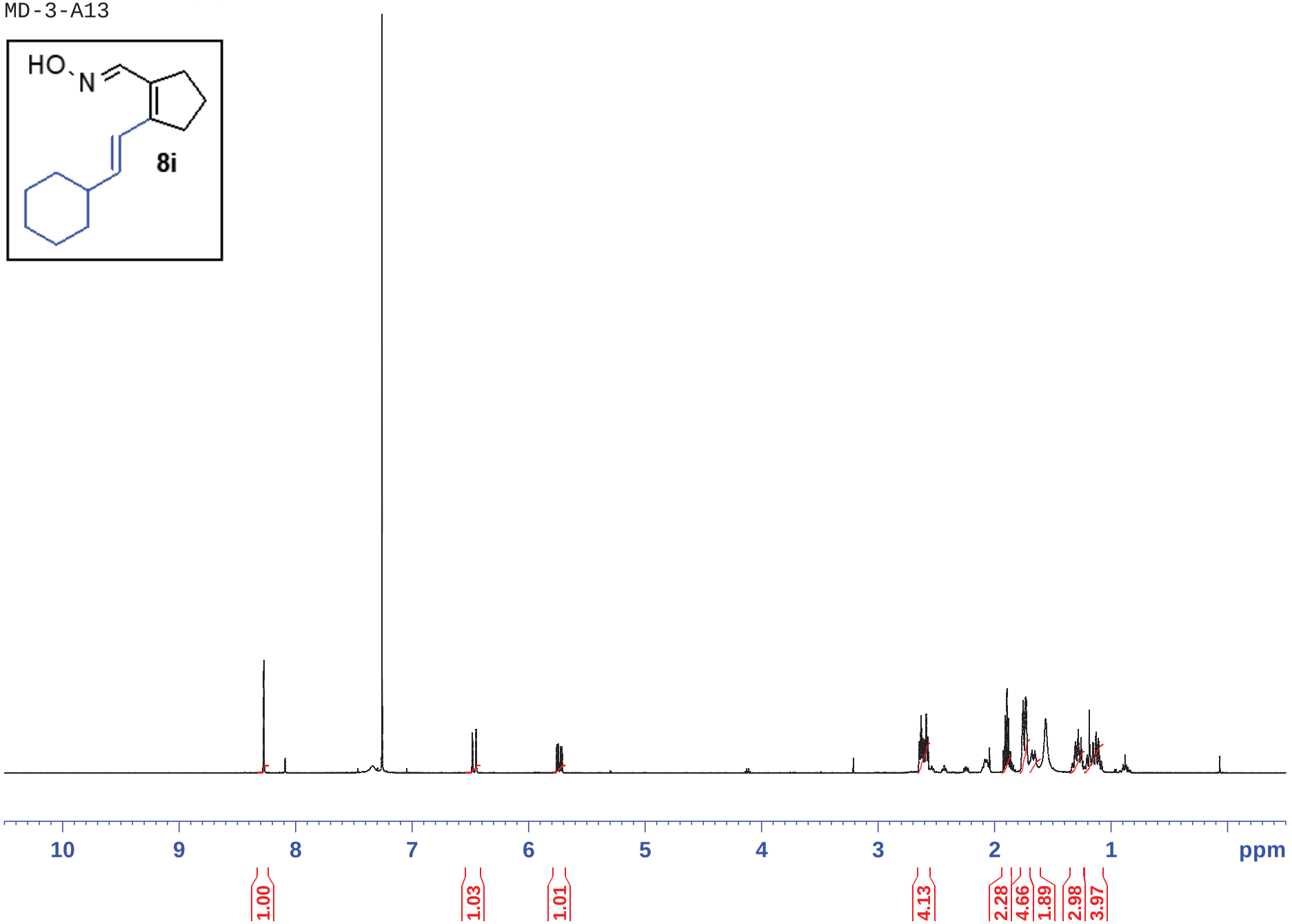
JWC-FGF23-A-43(3)
MD-3-A12



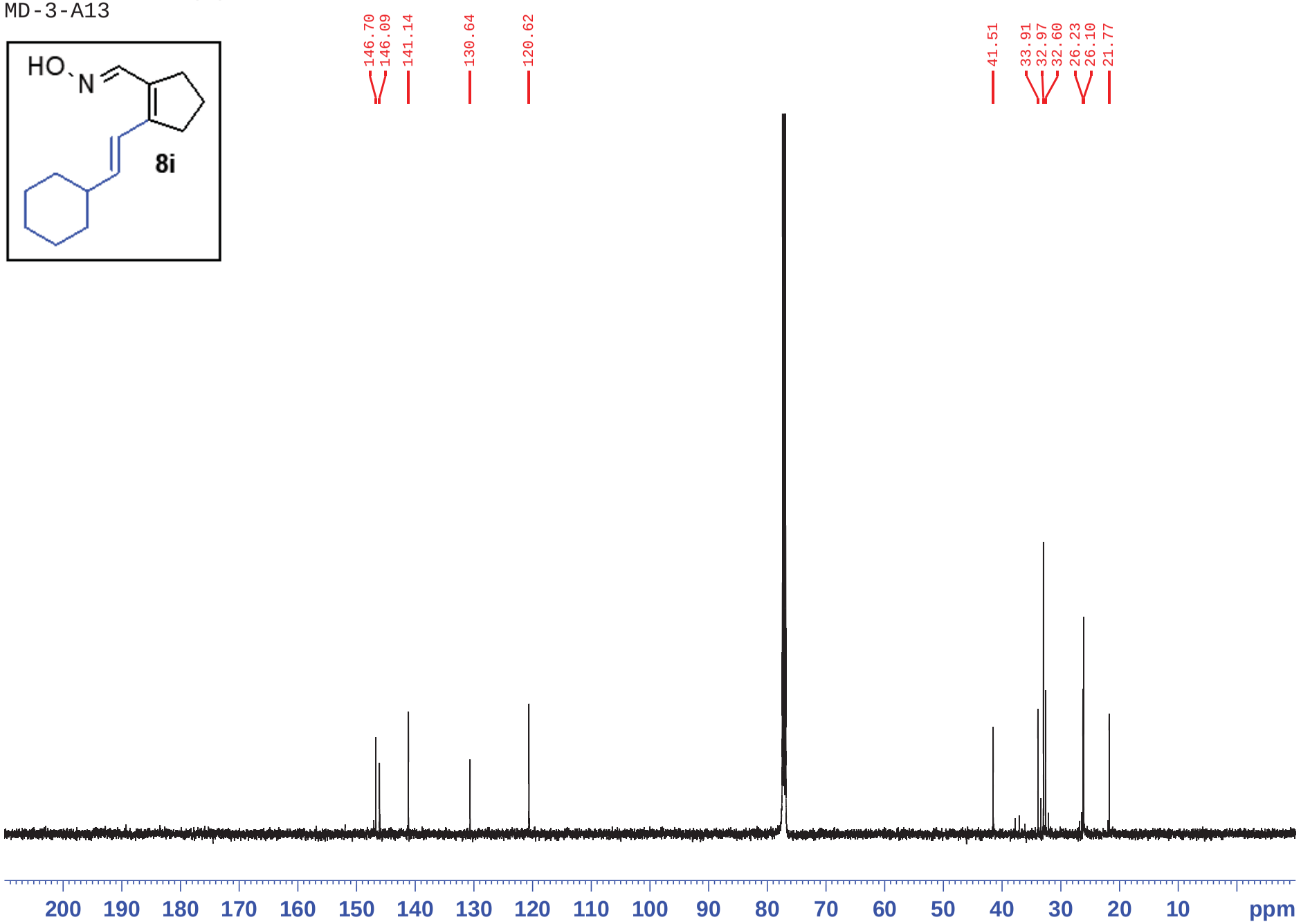
JWC-FGF23-A-43(3)
MD-3-A12



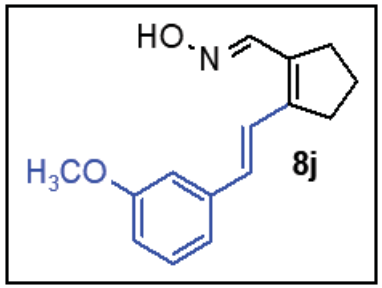
JWC-FGF23-A-47(3)
MD-3-A13

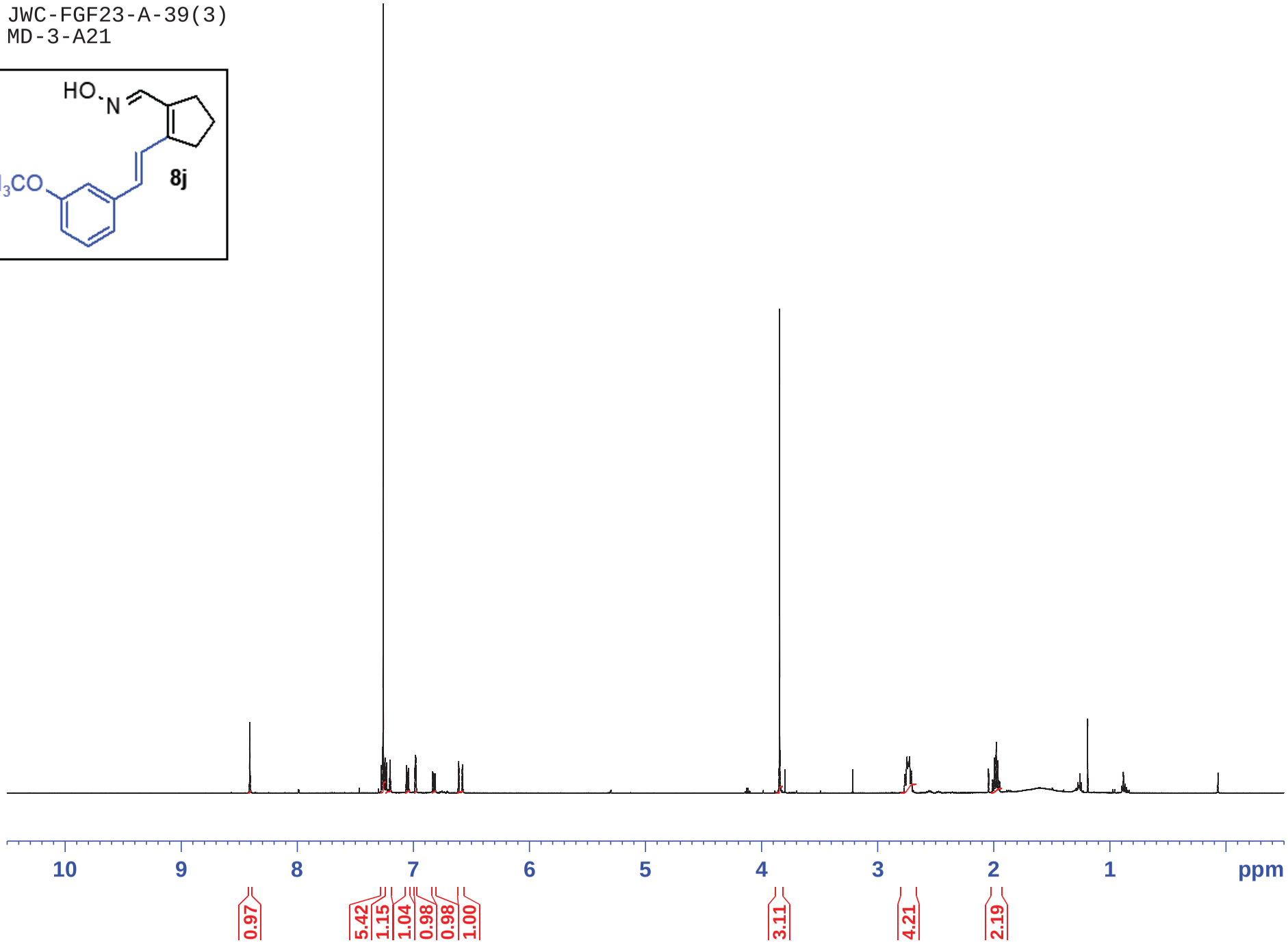


JWC-FGF23-A-47(3)
MD-3-A13

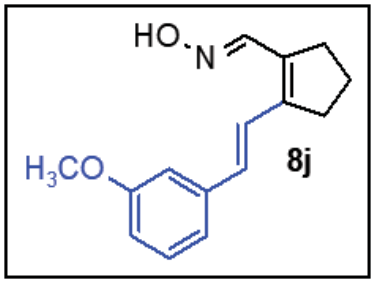


JWC-FGF23-A-39(3)
MD-3-A21





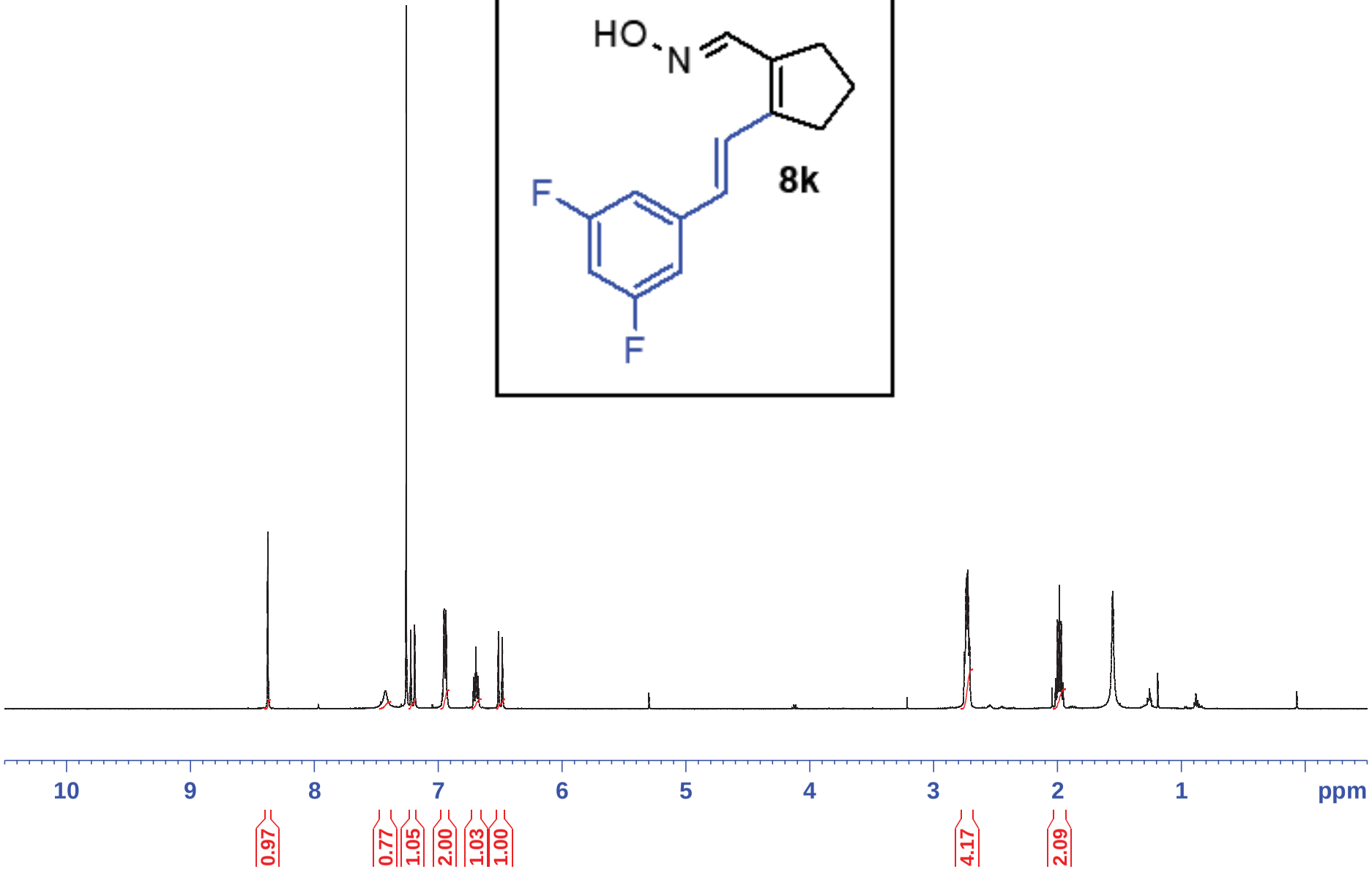
JWC-FGF23-A-39(5)
MD-3-A21



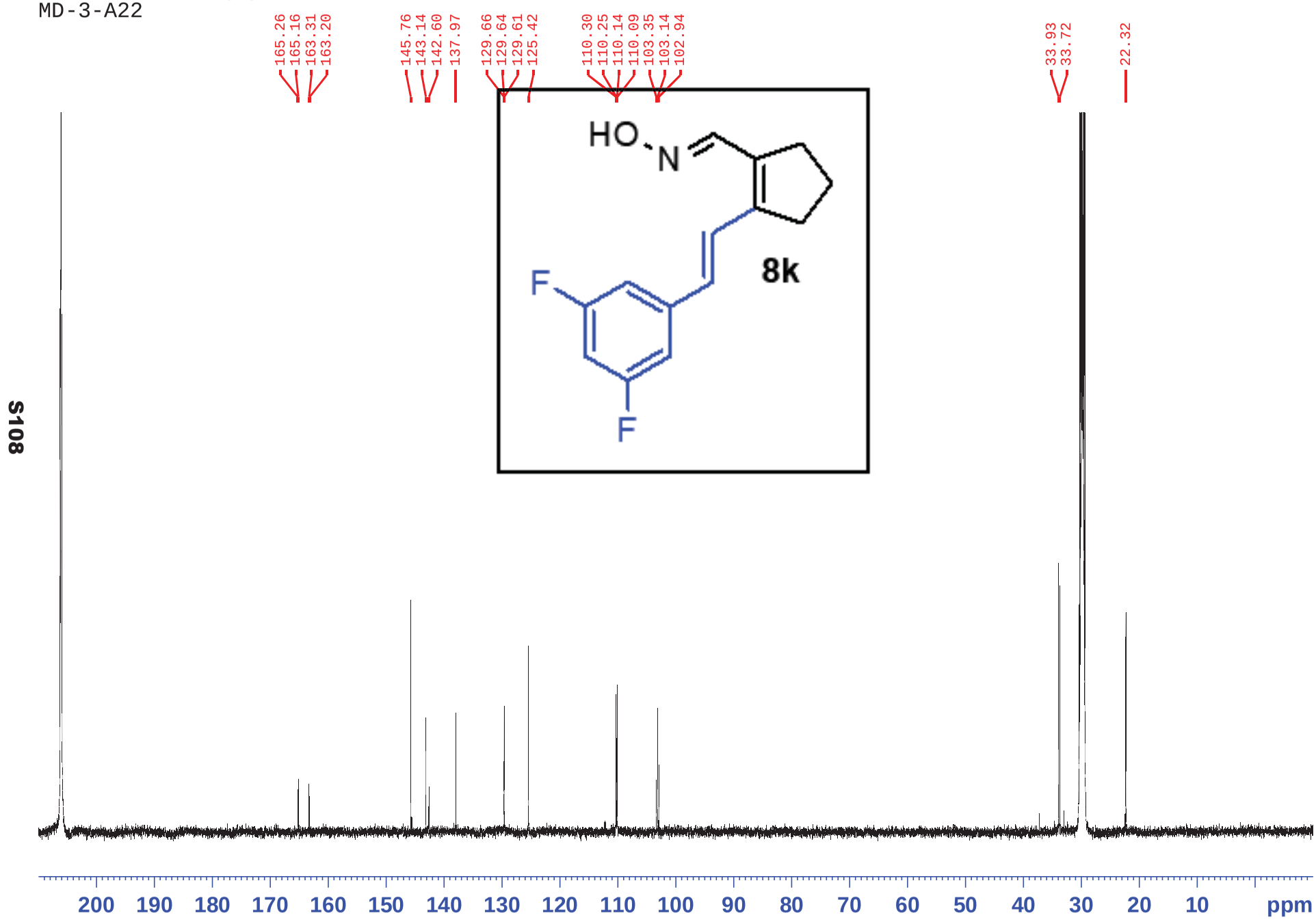
160.06
146.57
145.39
138.72
133.78
132.00
129.85
121.54
119.57
113.98
111.80
55.44
33.78
32.92
21.84

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

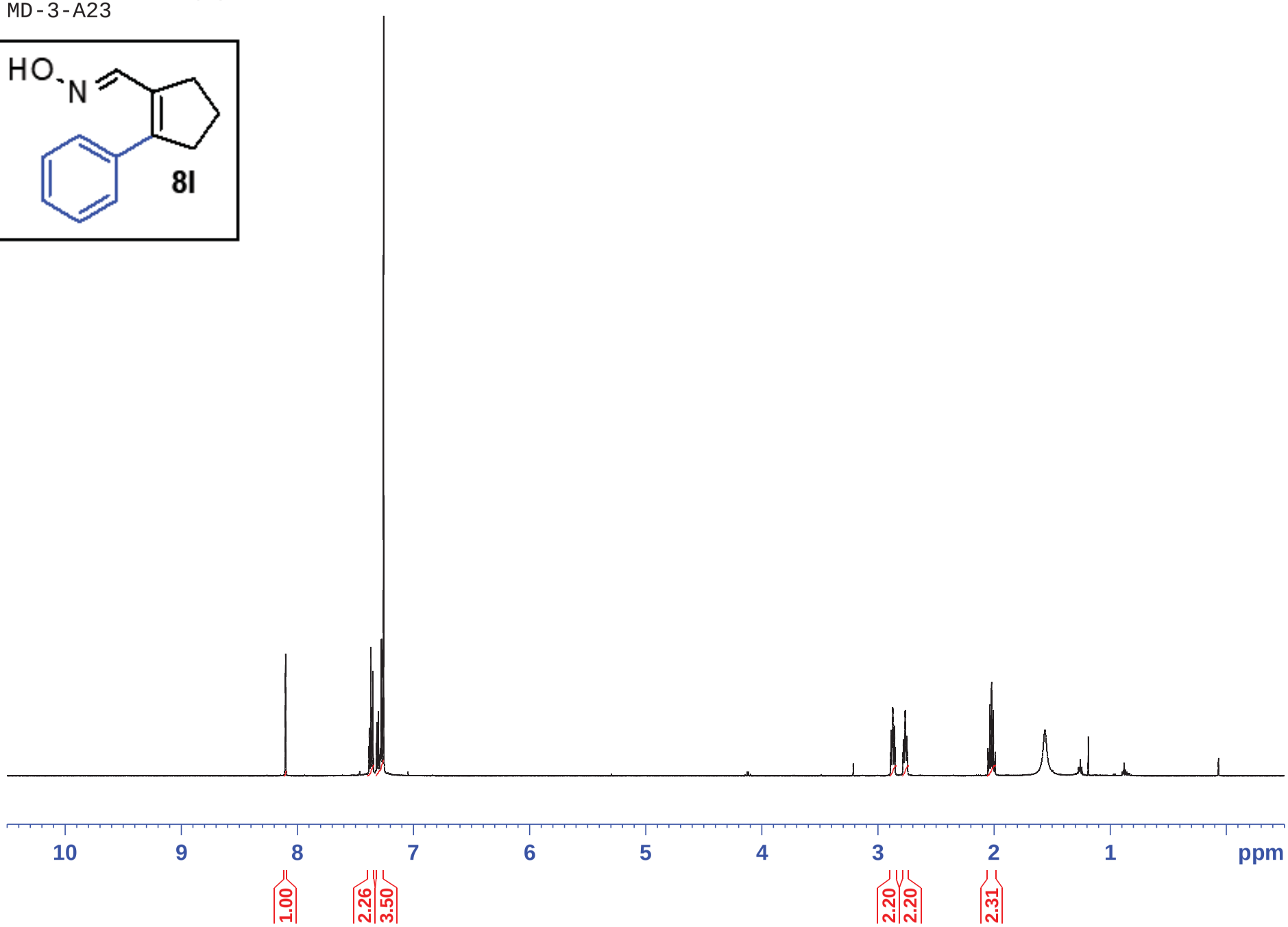
JWC-FGF23-A-41(3)
MD-3-A22



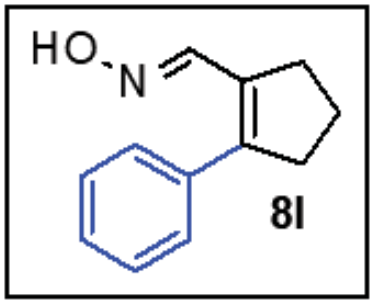
JWC-FGF23-A-41(6)
MD-3-A22



JWC-FGF23-A-33(3)
MD-3-A23



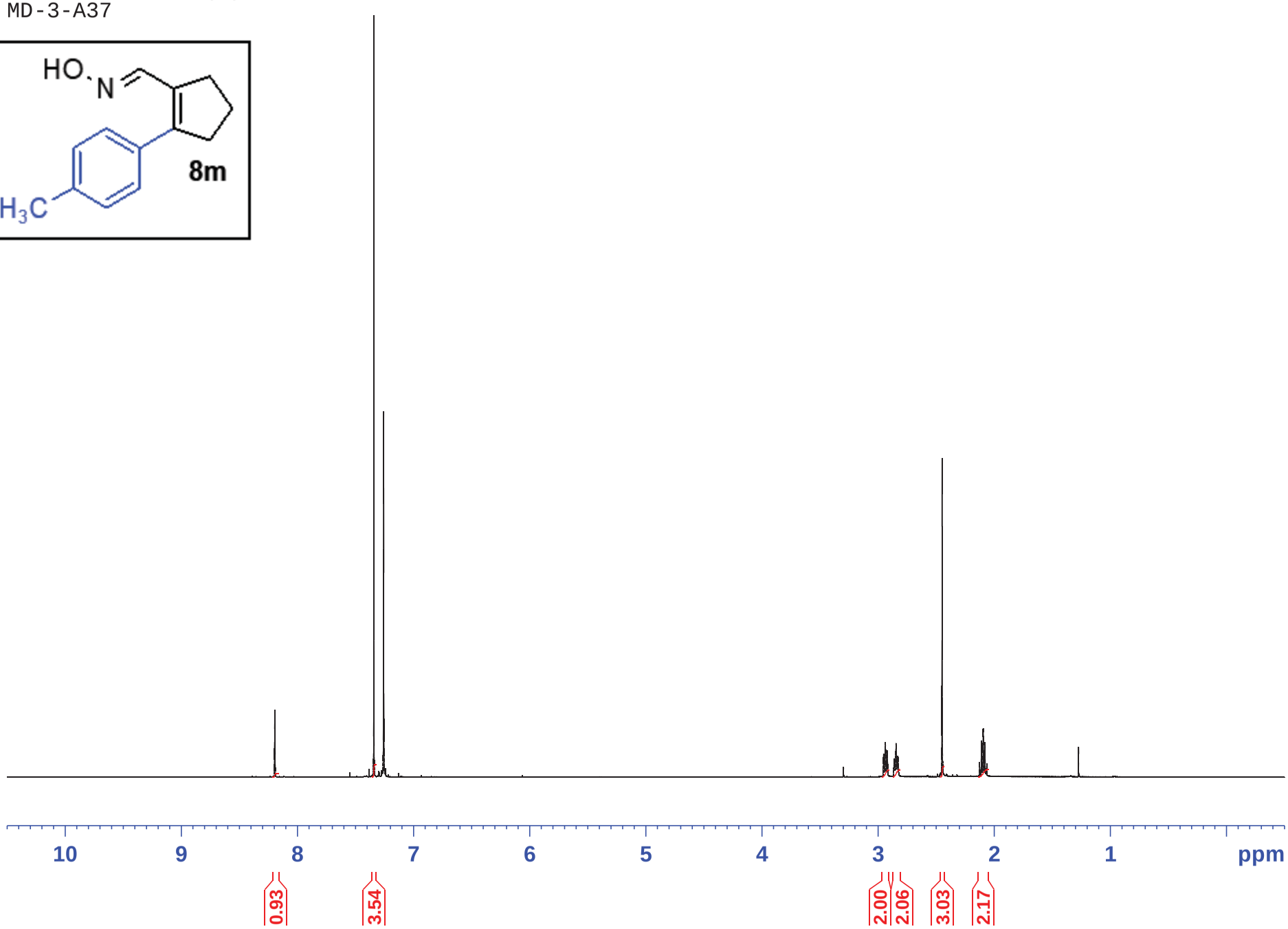
JWC-FGF23-A-33(3)
MD-3-A23



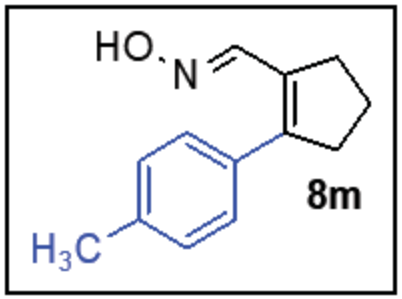
148.58
148.23
136.65
131.76
128.40
128.03
127.82
38.38
32.95
22.03

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-103(2)
MD-3-A37



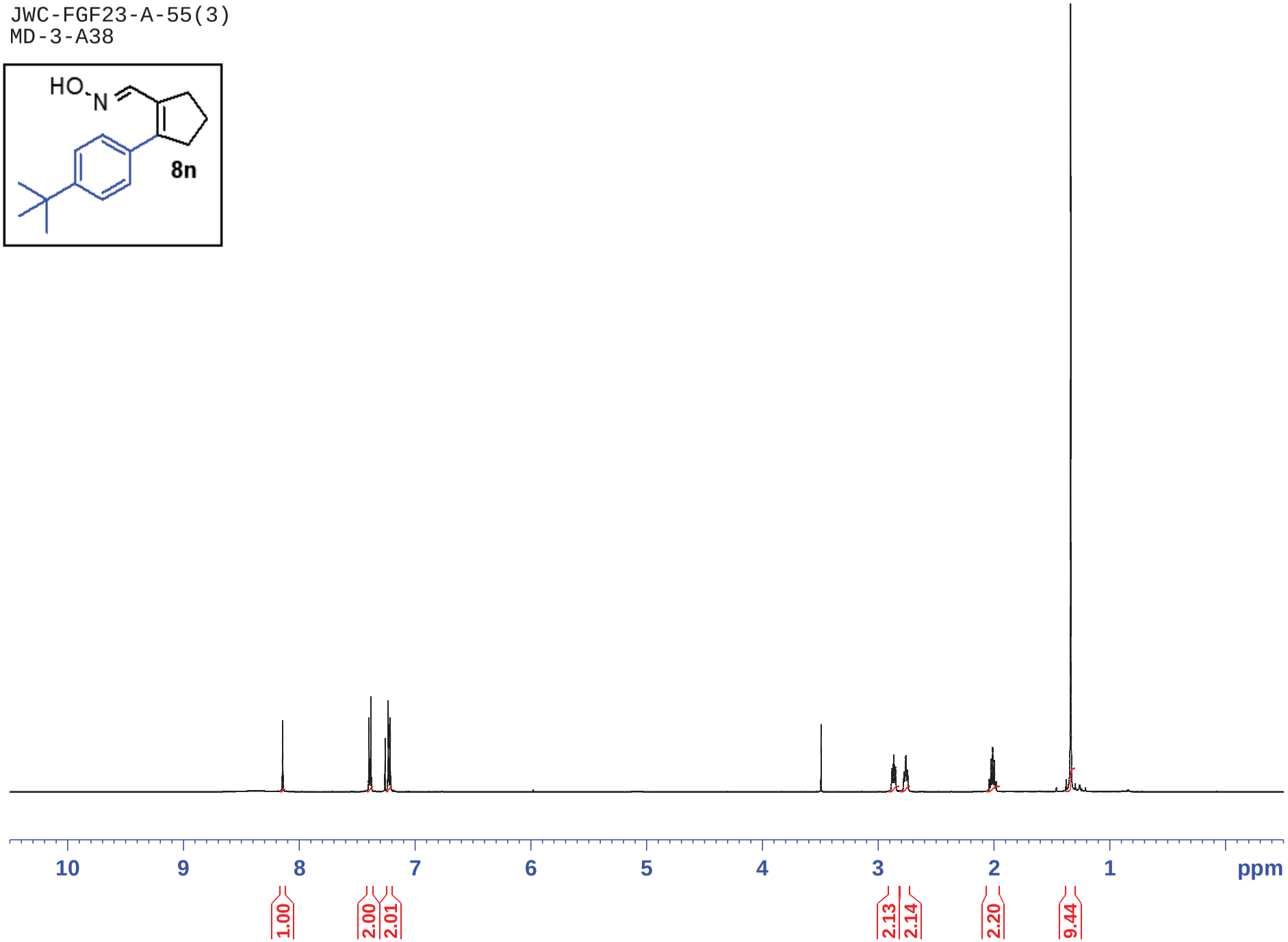
RPD-FGF23-A-103(2)
MD-3-A37



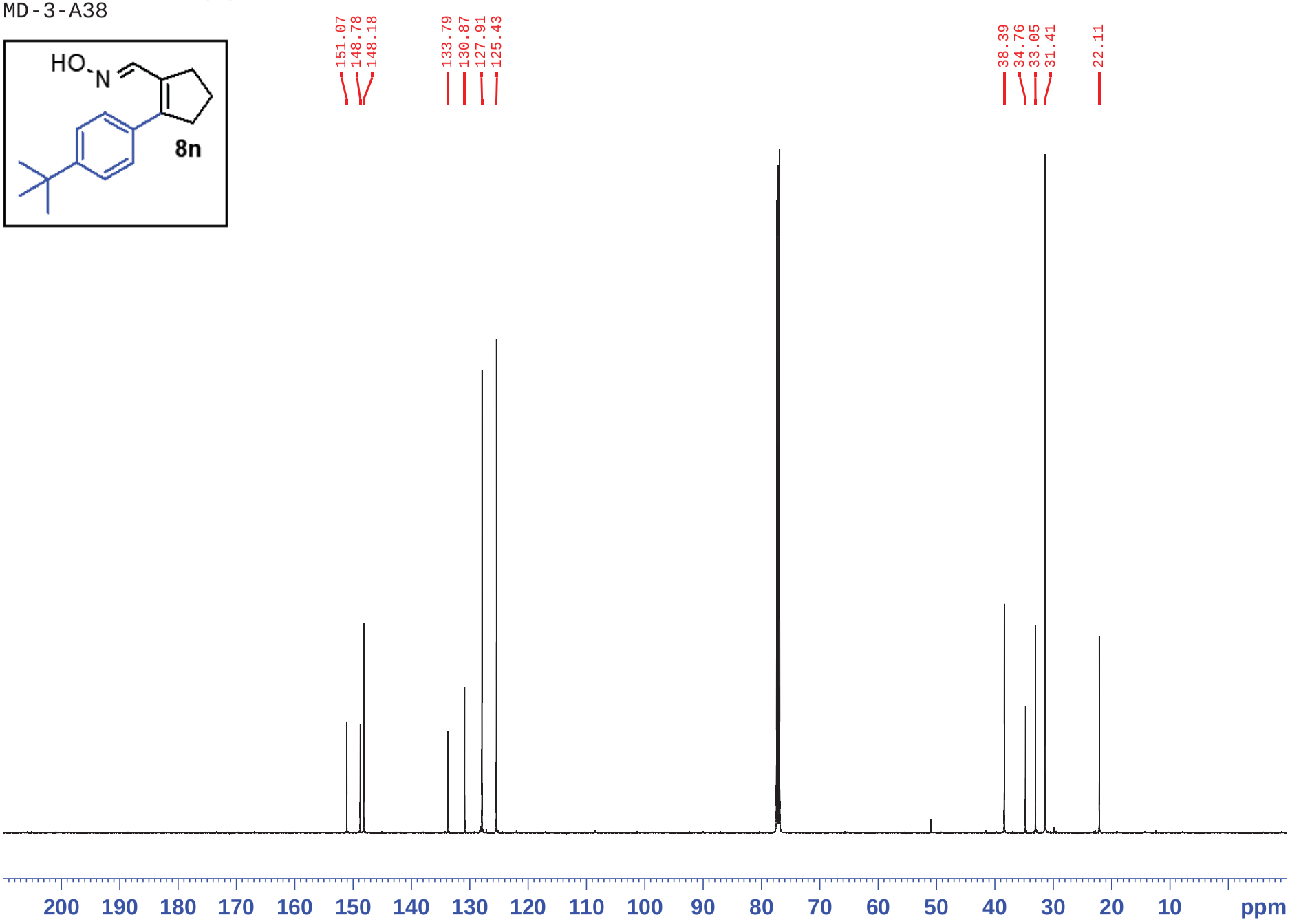
149.24
148.47
137.94
133.77
130.67
129.23
128.08
38.49
33.07
22.13
21.36

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

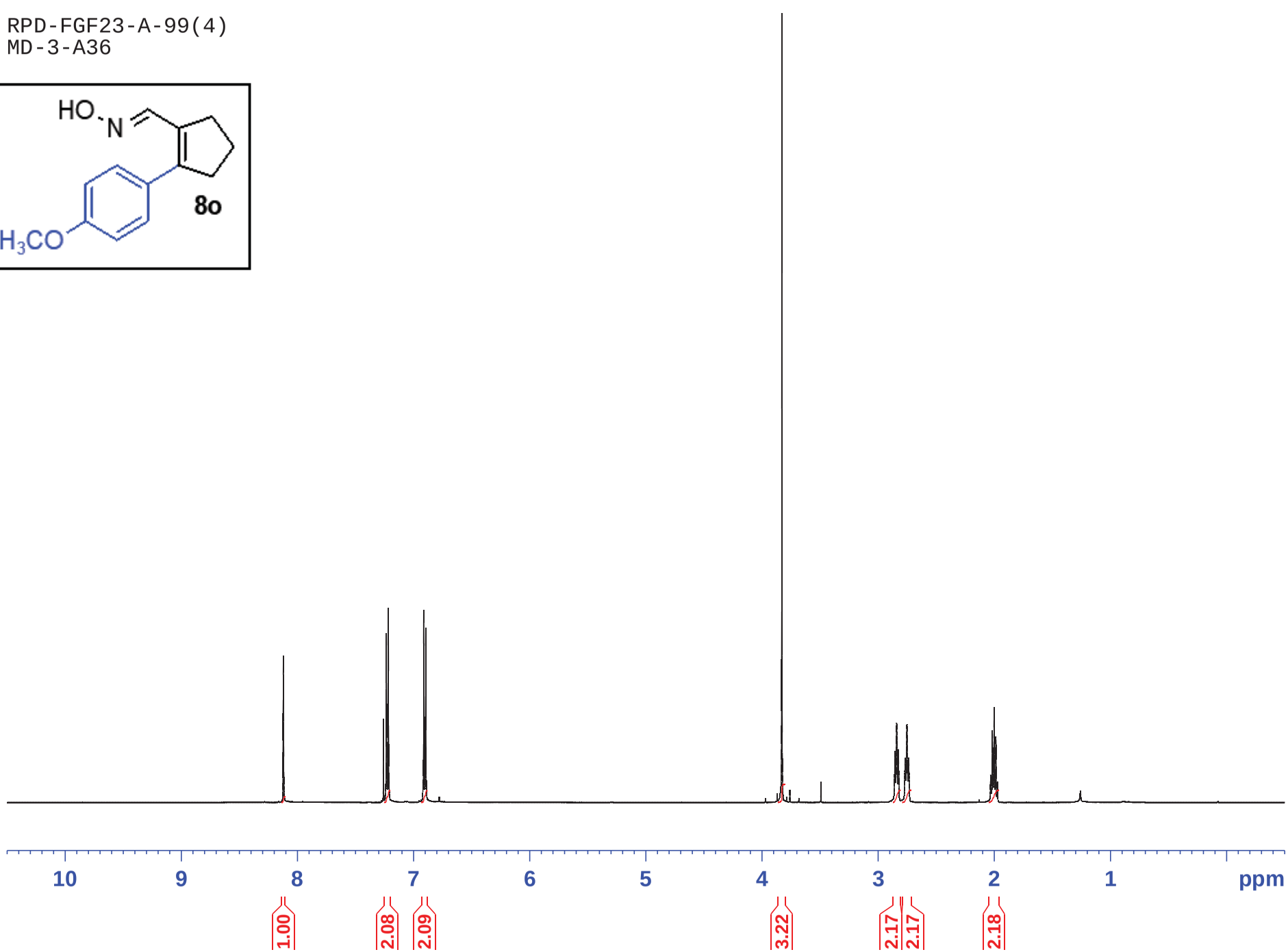
JWC-FGF23-A-55(3)
MD-3-A38



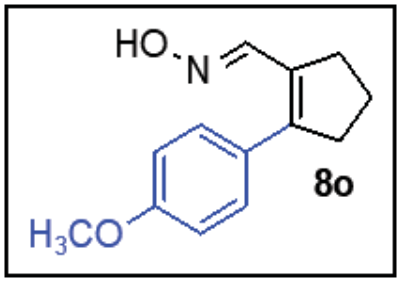
JWC-FGF23-A-101(5)
MD-3-A38



RPD-FGF23-A-99(4)
MD-3-A36



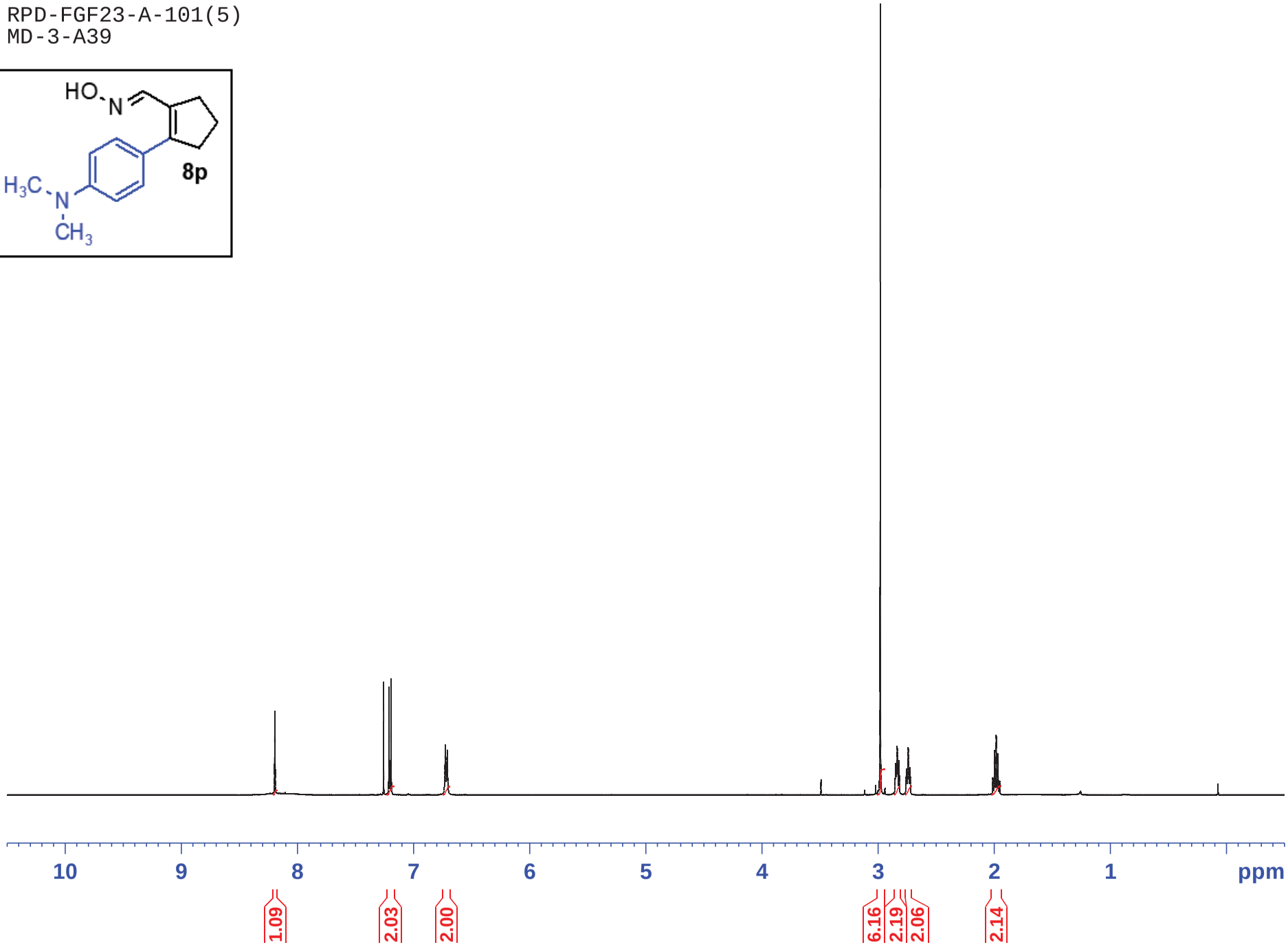
RPD-FGF23-A-99(4)
MD-3-A36



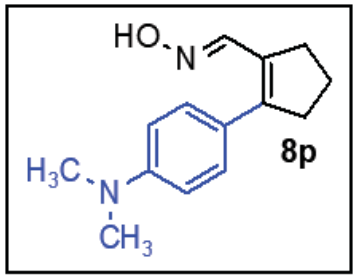
159.46
148.49
148.06
130.13
129.41
129.16
113.95
55.44
38.42
33.05
22.02

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-101(5)
MD-3-A39



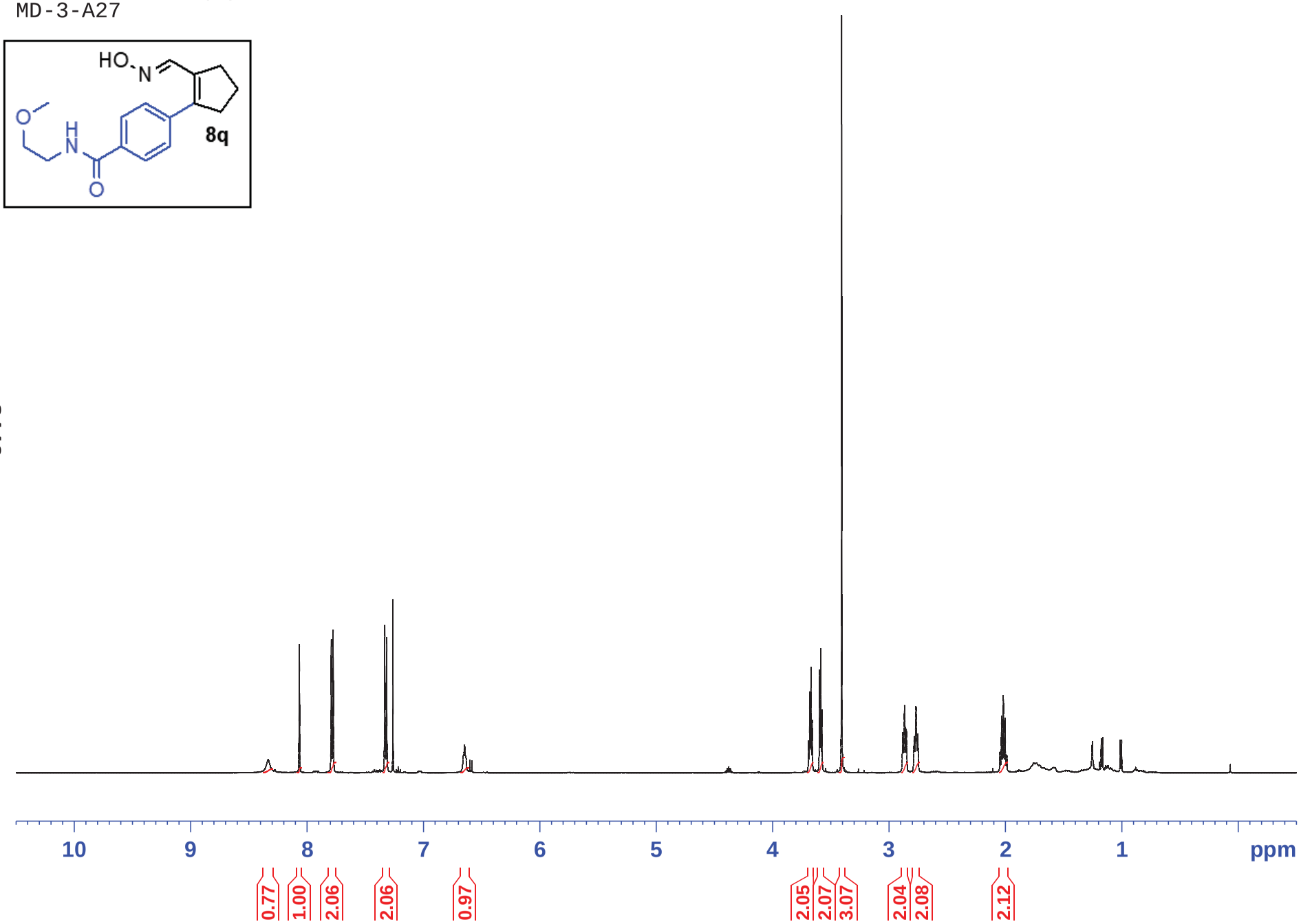
RPD-FGF23-A-105(2)
MD-3-A39



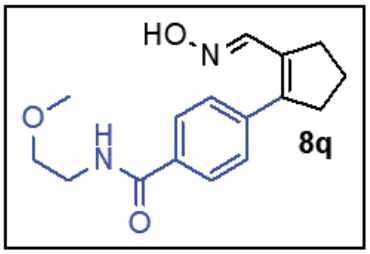
150.20
148.81
148.65
148.59
129.22
128.65
112.22
40.59
38.15
33.11
22.03

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

JWC-FGF23-A-45(5)
MD-3-A27



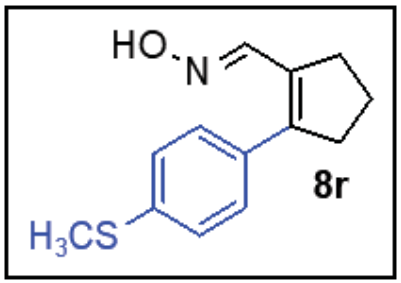
JWC-FGF23-A-45(5)
MD-3-A27



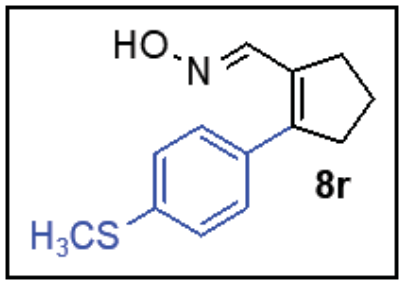
167.18
147.54
147.05
139.99
133.72
133.03
128.27
127.22
71.42
59.00
39.84
38.36
33.27
22.12

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-105(2)
MD-3-A40



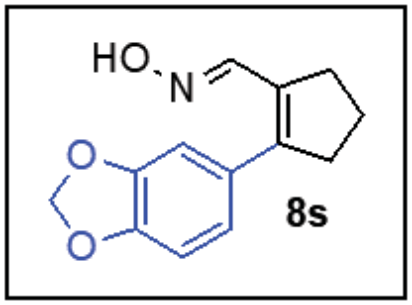
RPD-FGF23-A-105(2)
MD-3-A40

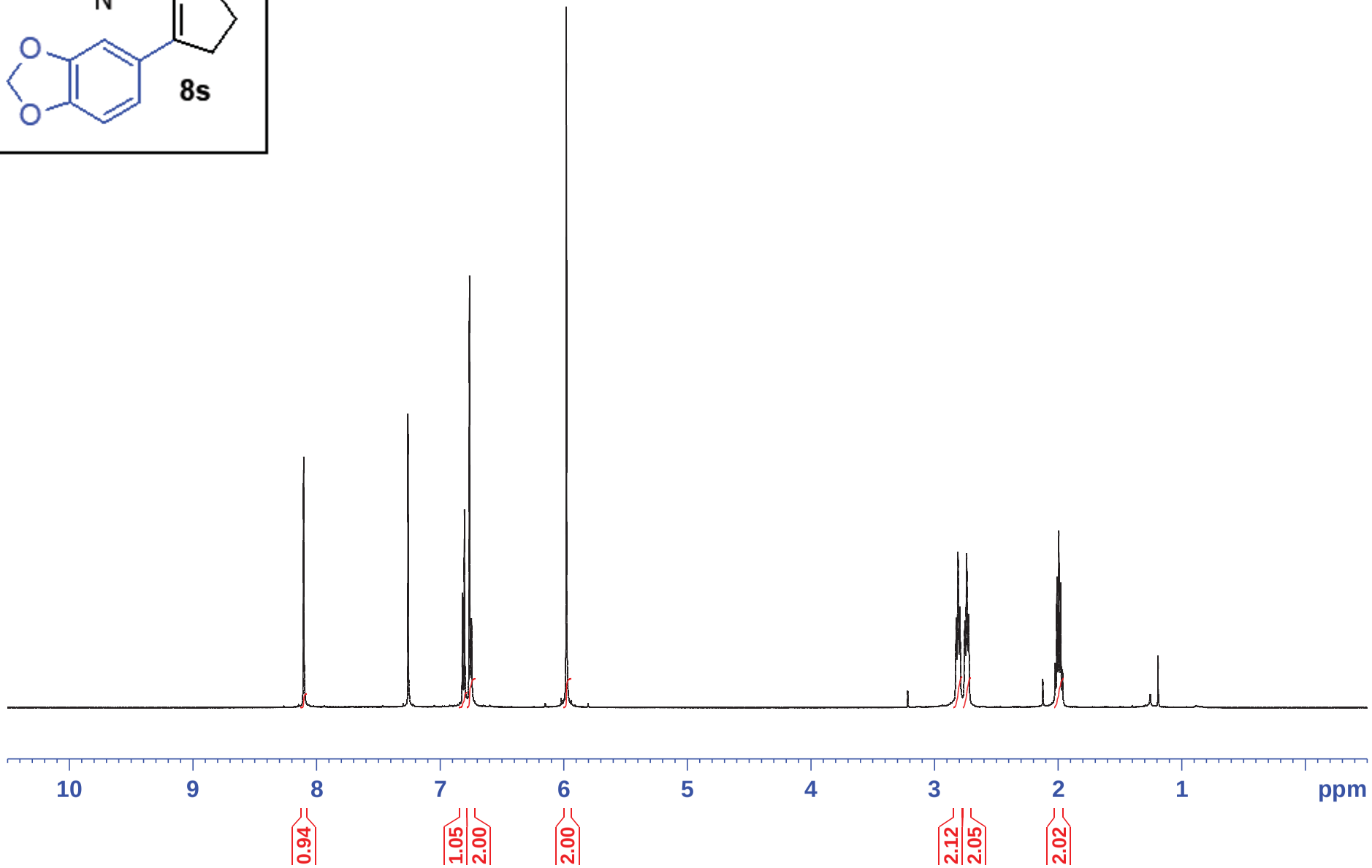


148.33
148.16
138.57
133.36
131.15
128.56
126.40
38.32
33.11
22.09
15.80

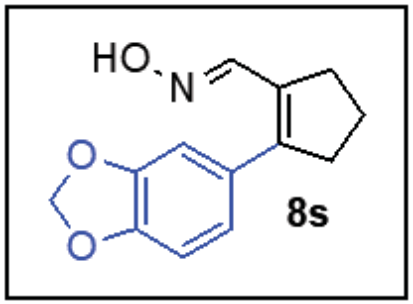
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

JWC-FGF23-A-57(2)
MD-3-A41

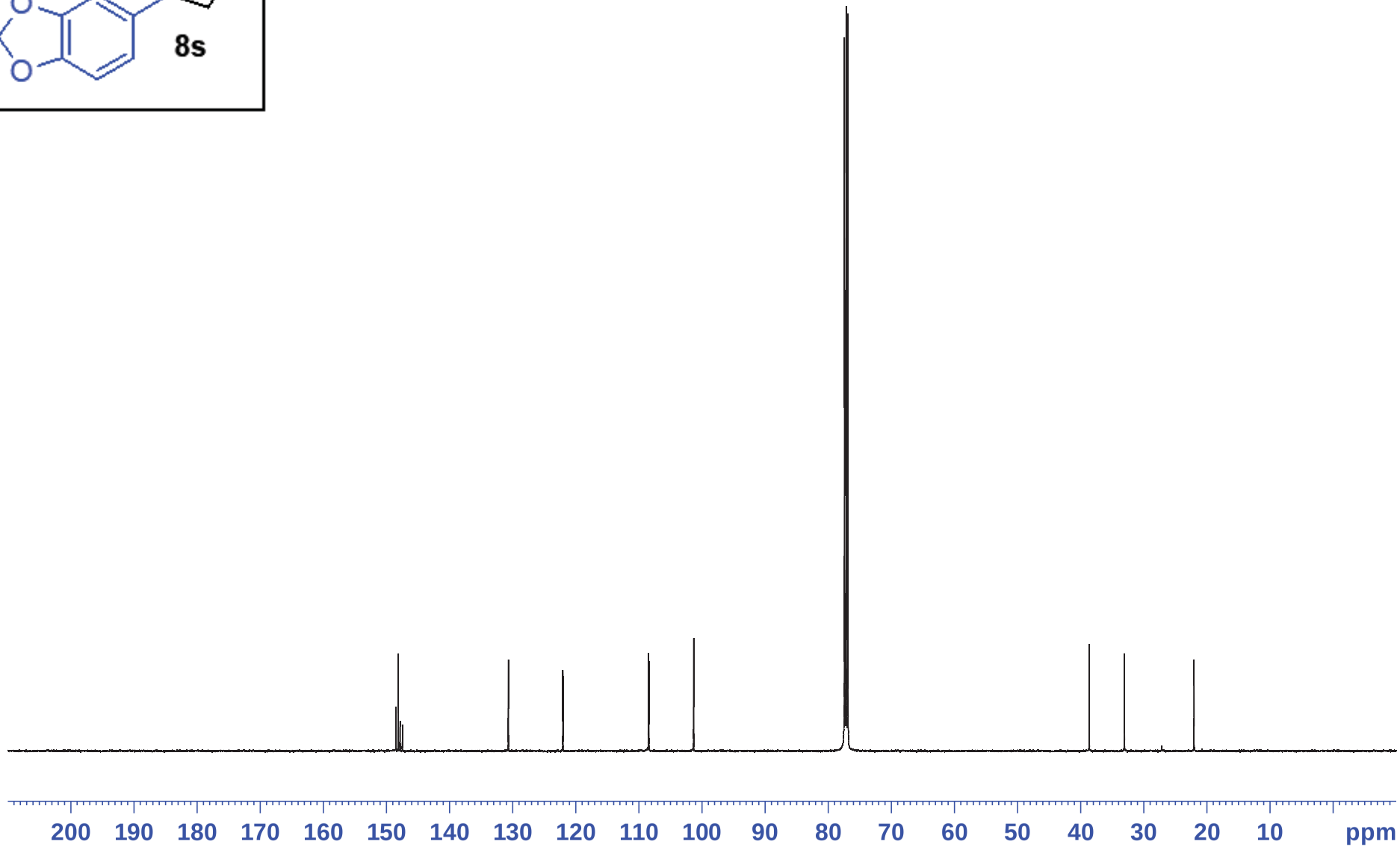




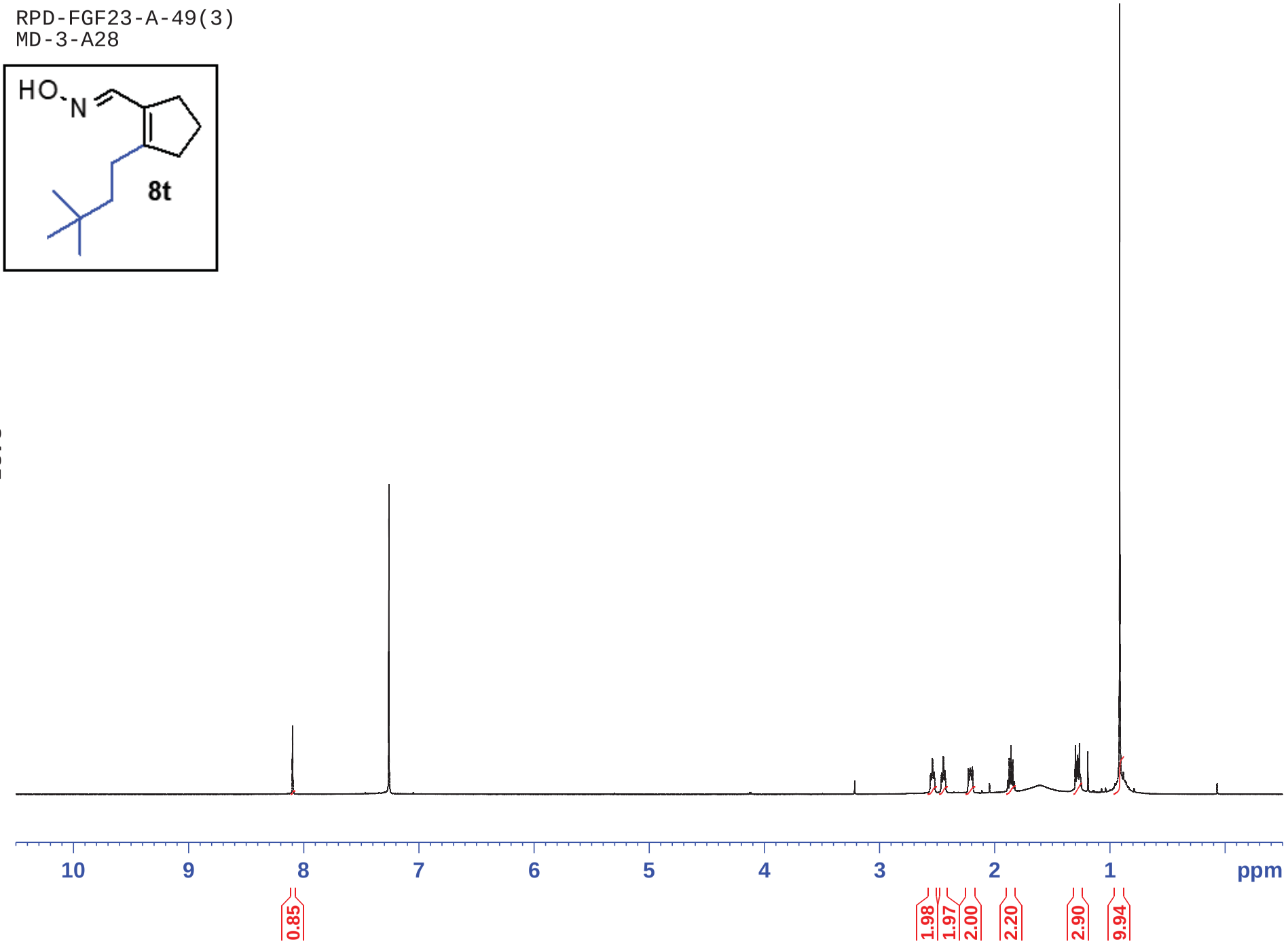
JWC-FGF23-A-57(2)
MD-3-A41



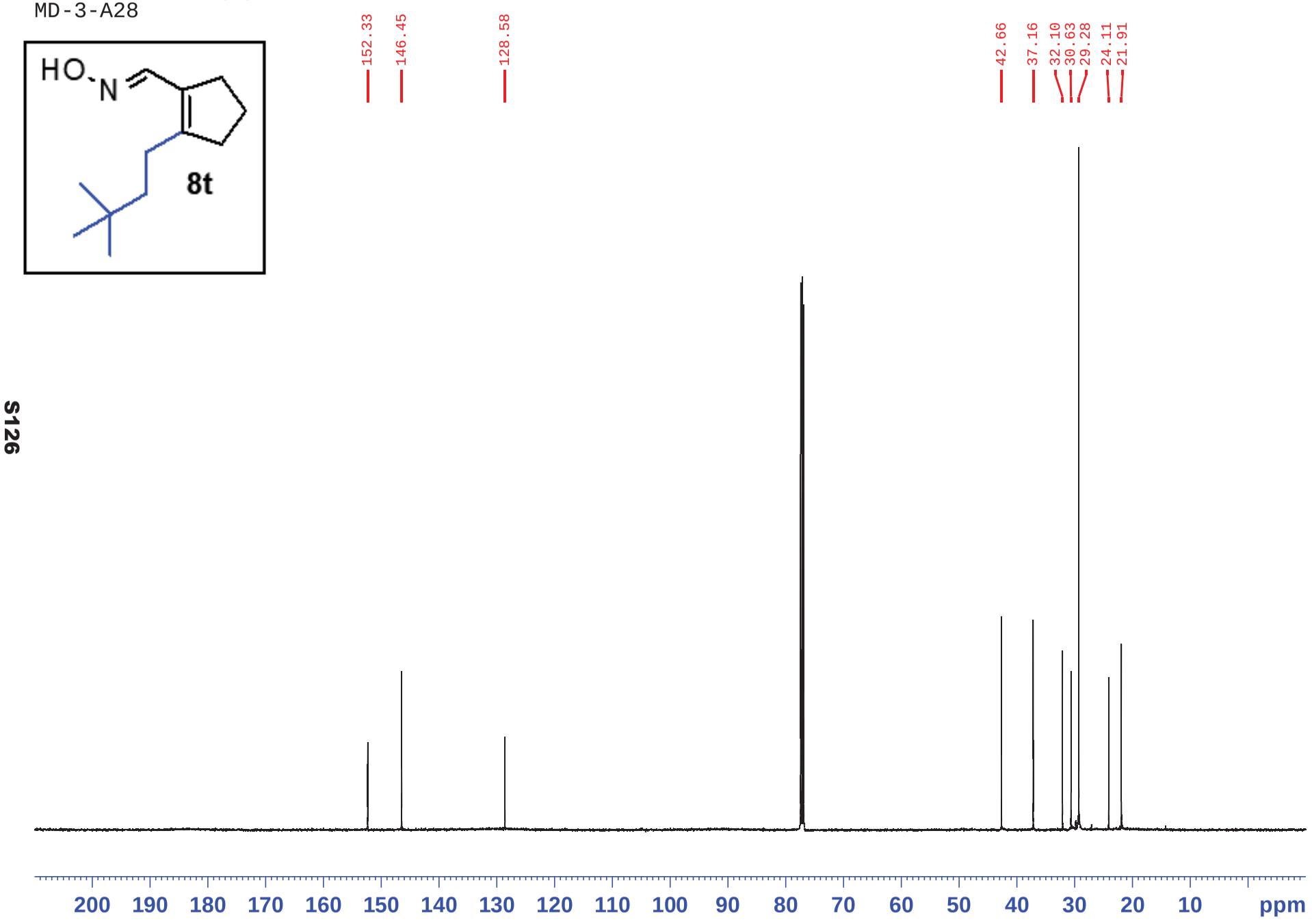
148.49
148.12
147.83
147.47
130.68
122.03
108.48
108.42
101.32
38.63
33.07
22.04



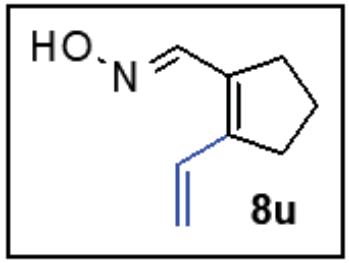
RPD-FGF23-A-49(3)
MD-3-A28

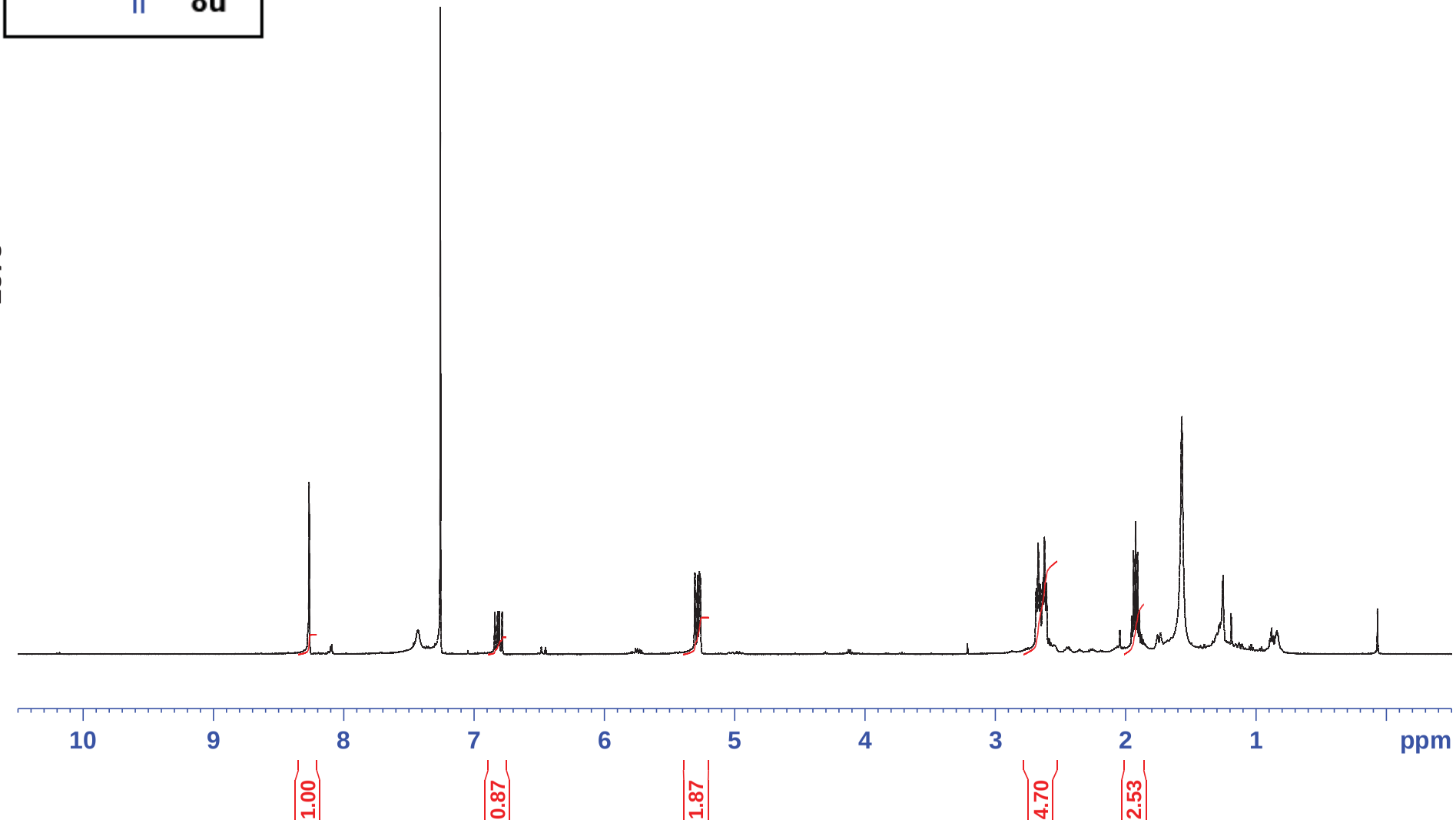


RPD-FGF23-A-49(3)
MD-3-A28

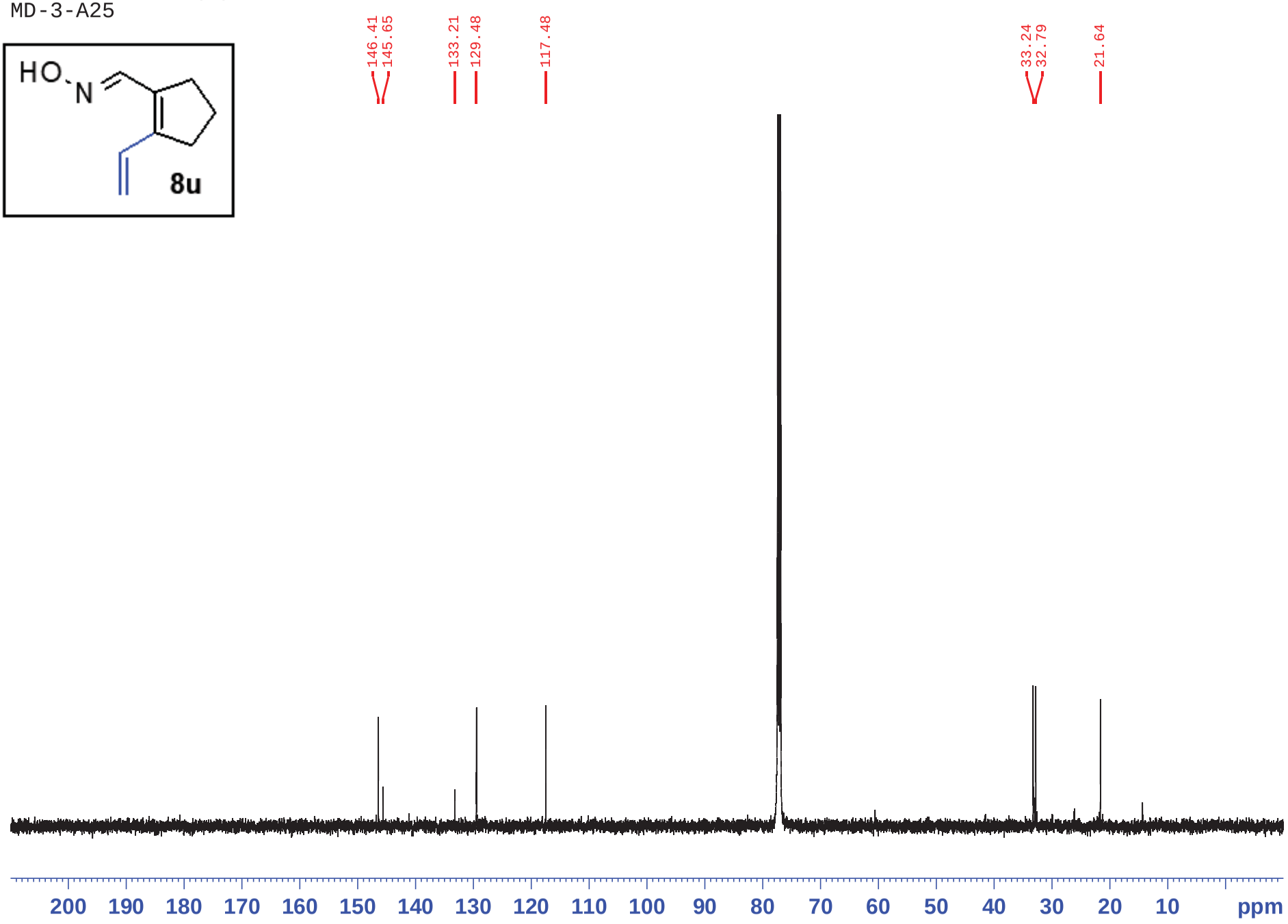


RPD-FGF23-A-37(7)
MD-3-A25



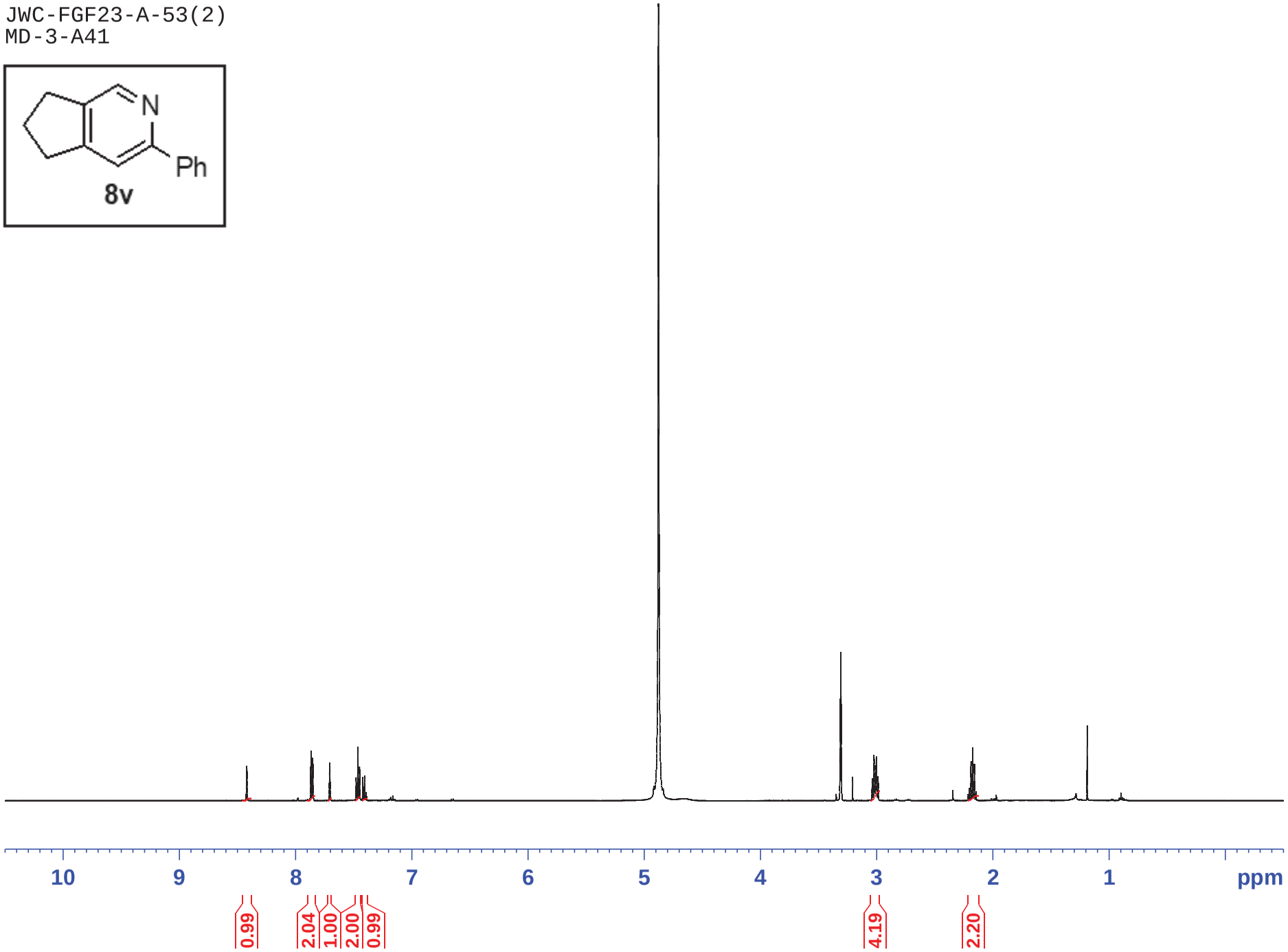


RPD-FGF23-A-37(7)
MD-3-A25

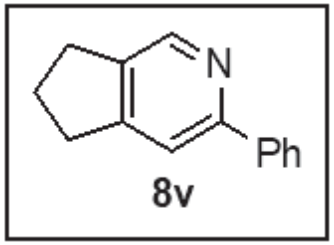


JWC-FGF23-A-53(2)
MD-3-A41

8v



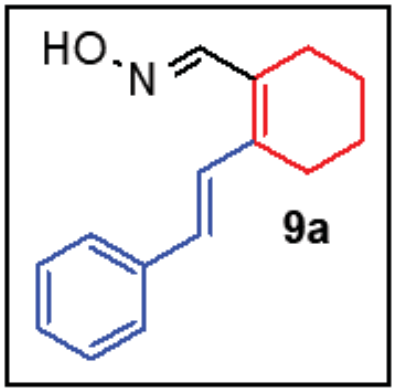
JWC-FGF23-A-53(2)
MD-3-A43

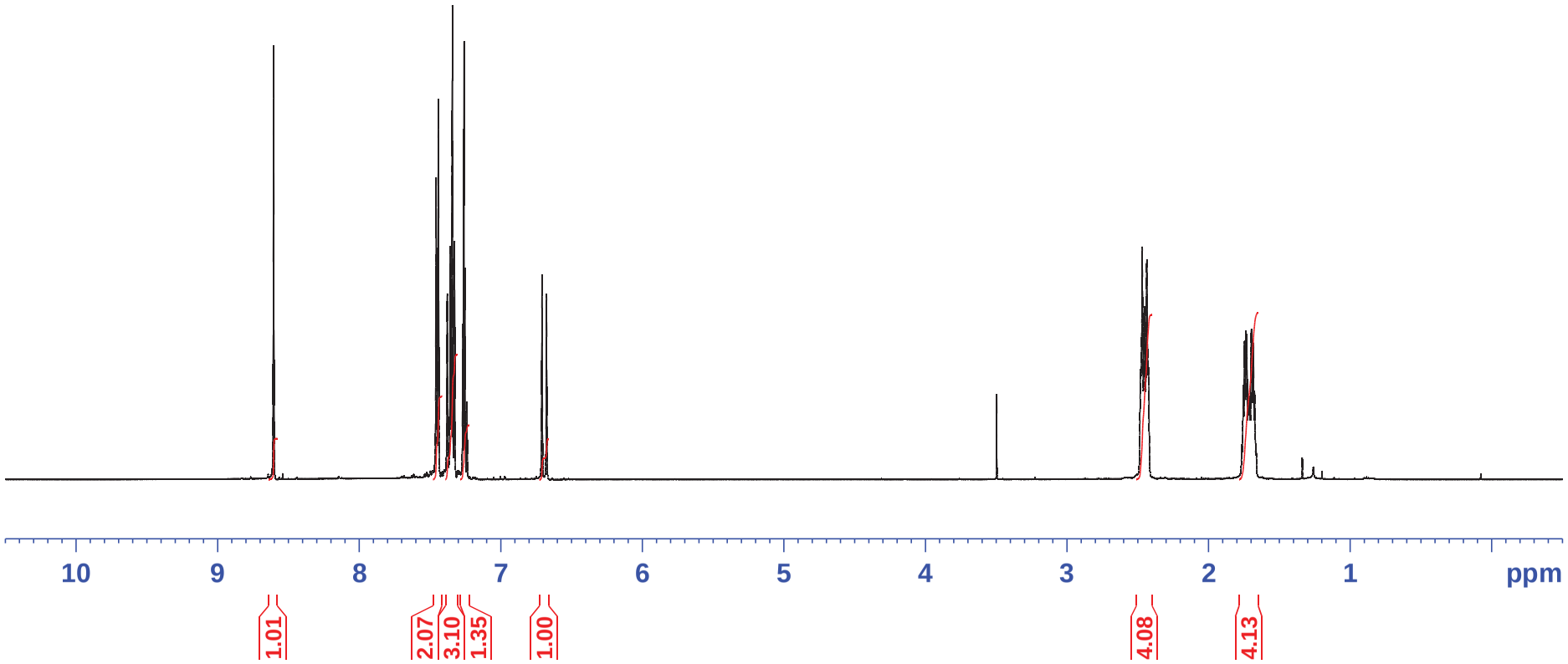


8v

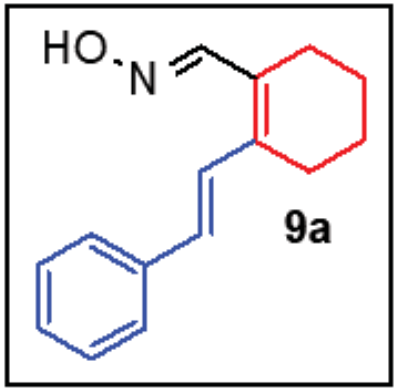
157.47
156.99
145.53
140.82
140.72
129.78
129.75
128.17
118.89
33.67
30.84
26.18

RPD-FGF23-A-91(4)
MD-3-A33





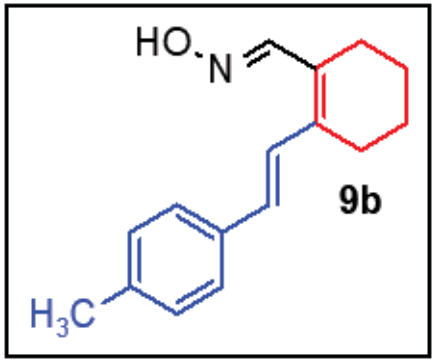
RPD-FGF23-A-91(4)
MD-3-A33

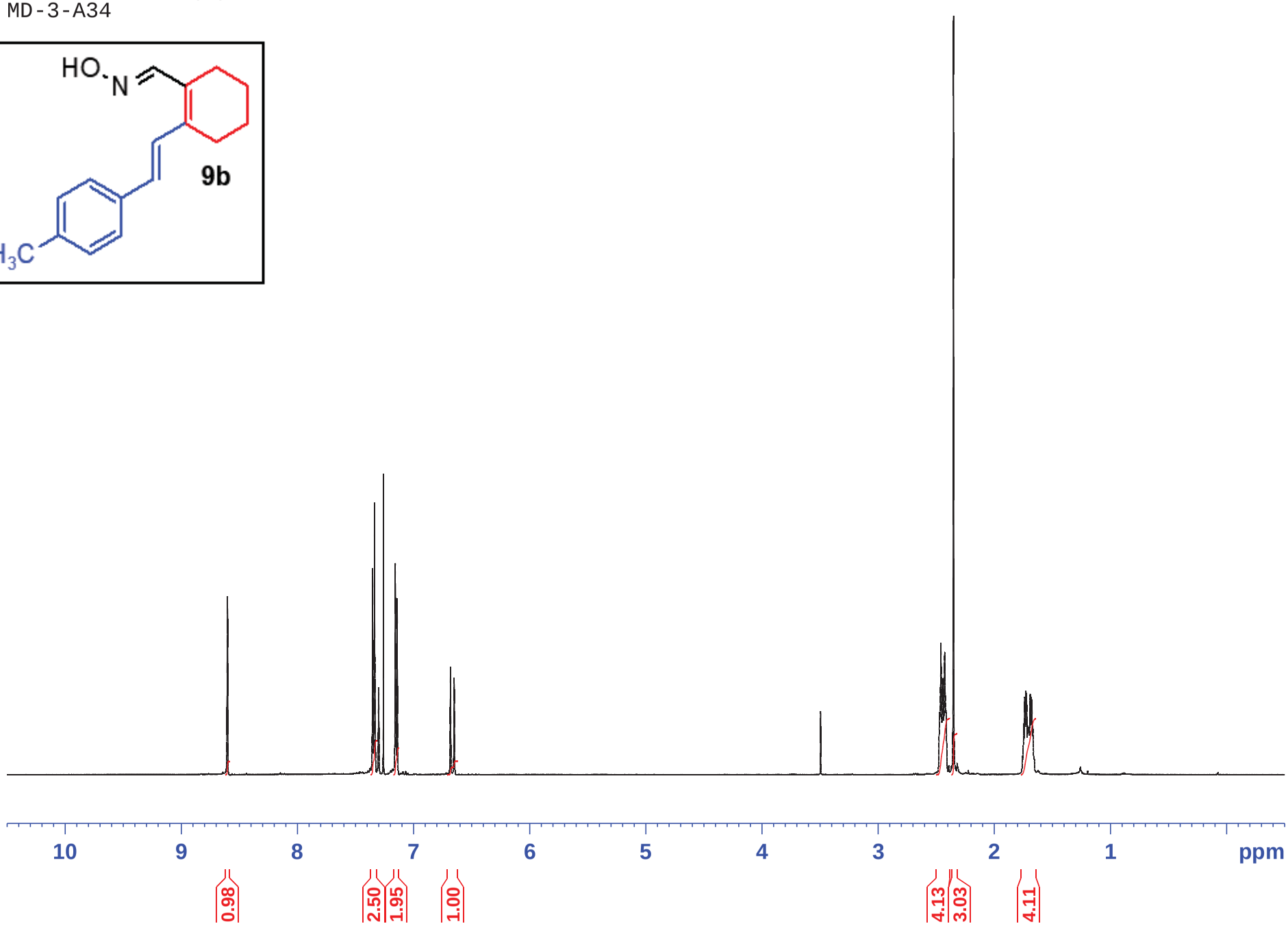


149.12
139.09
137.52
129.64
129.31
128.83
127.94
126.72
124.98
26.87
25.47
22.38
22.05

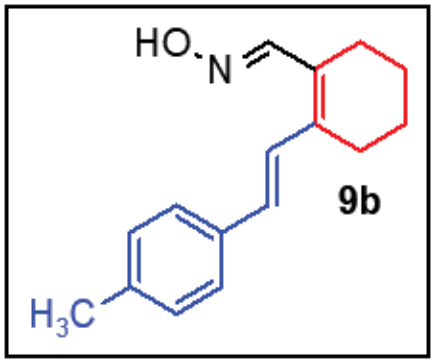
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

RPD-FGF23-A-93(4)
MD-3-A34

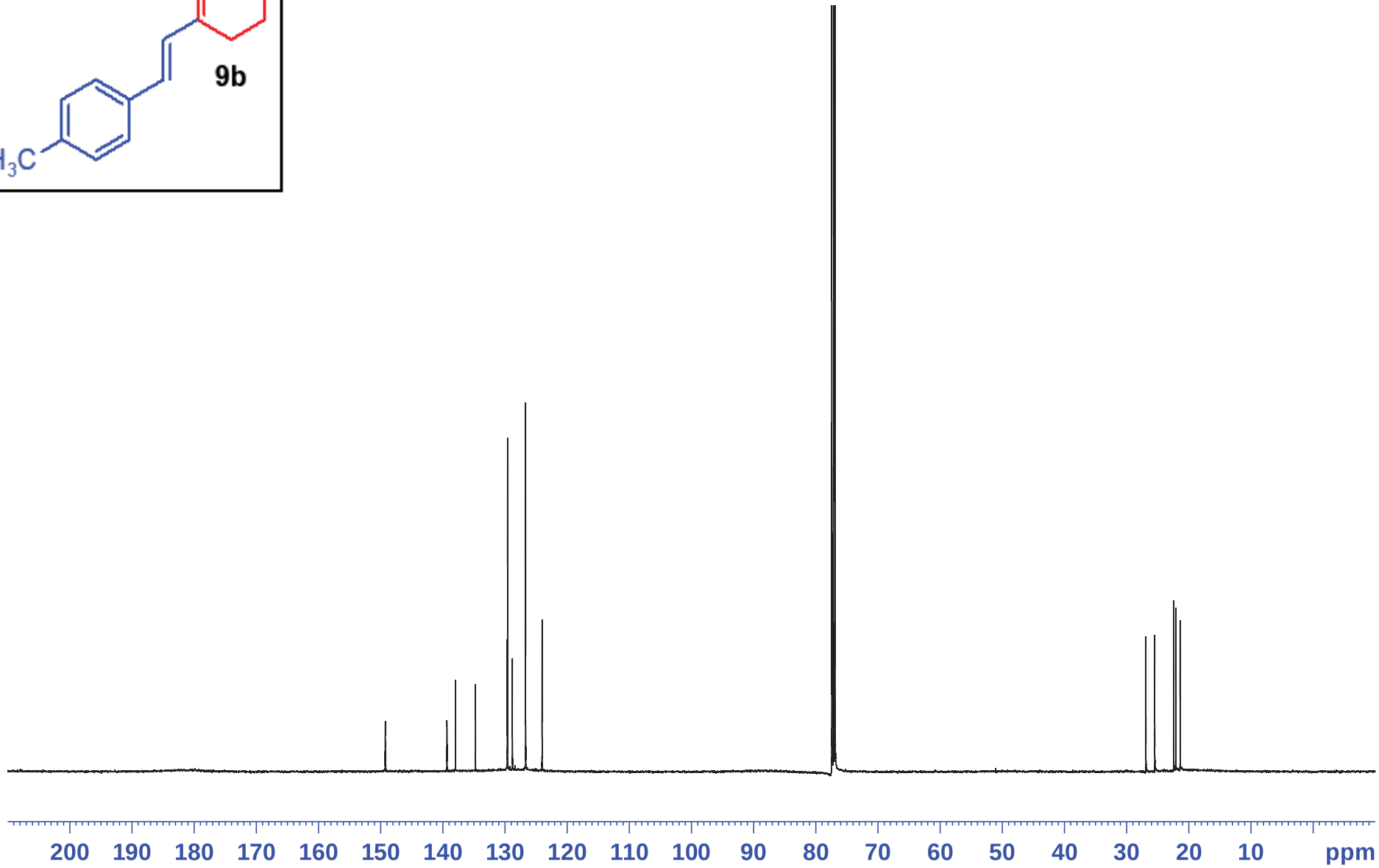




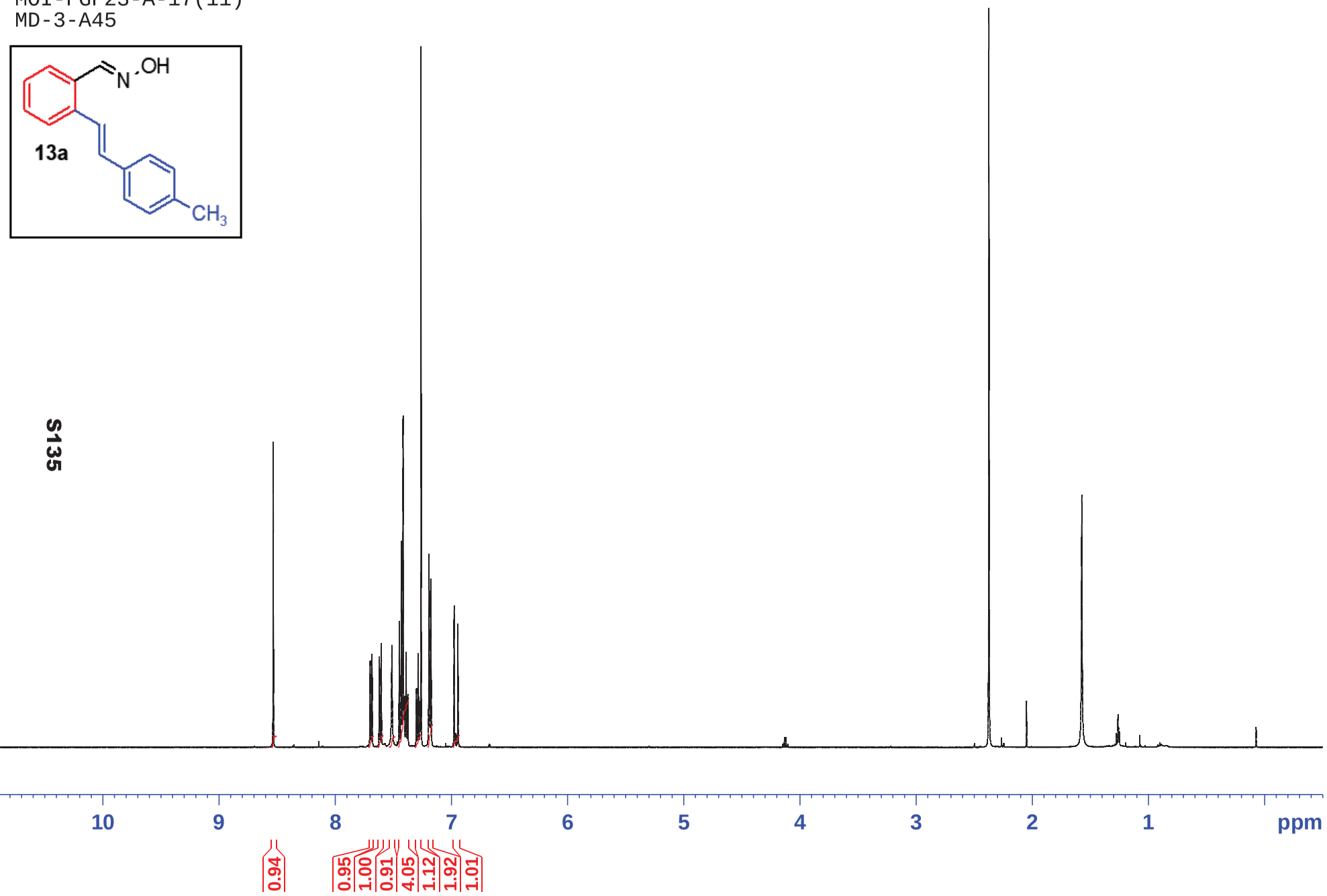
RPD-FGF23-A-93(4)
MD-3-A34



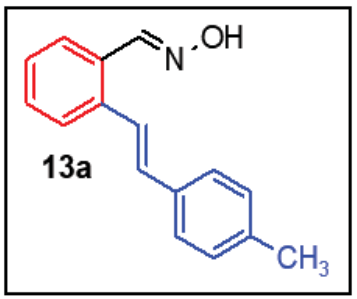
149.25
139.33
137.95
134.74
129.63
129.58
128.80
126.67
124.03
26.89
25.47
22.43
22.10
21.40



MOI-FGF23-A-17(11)
MD-3-A45



MOI-FGF23-A-17(11)
MD-3-A45



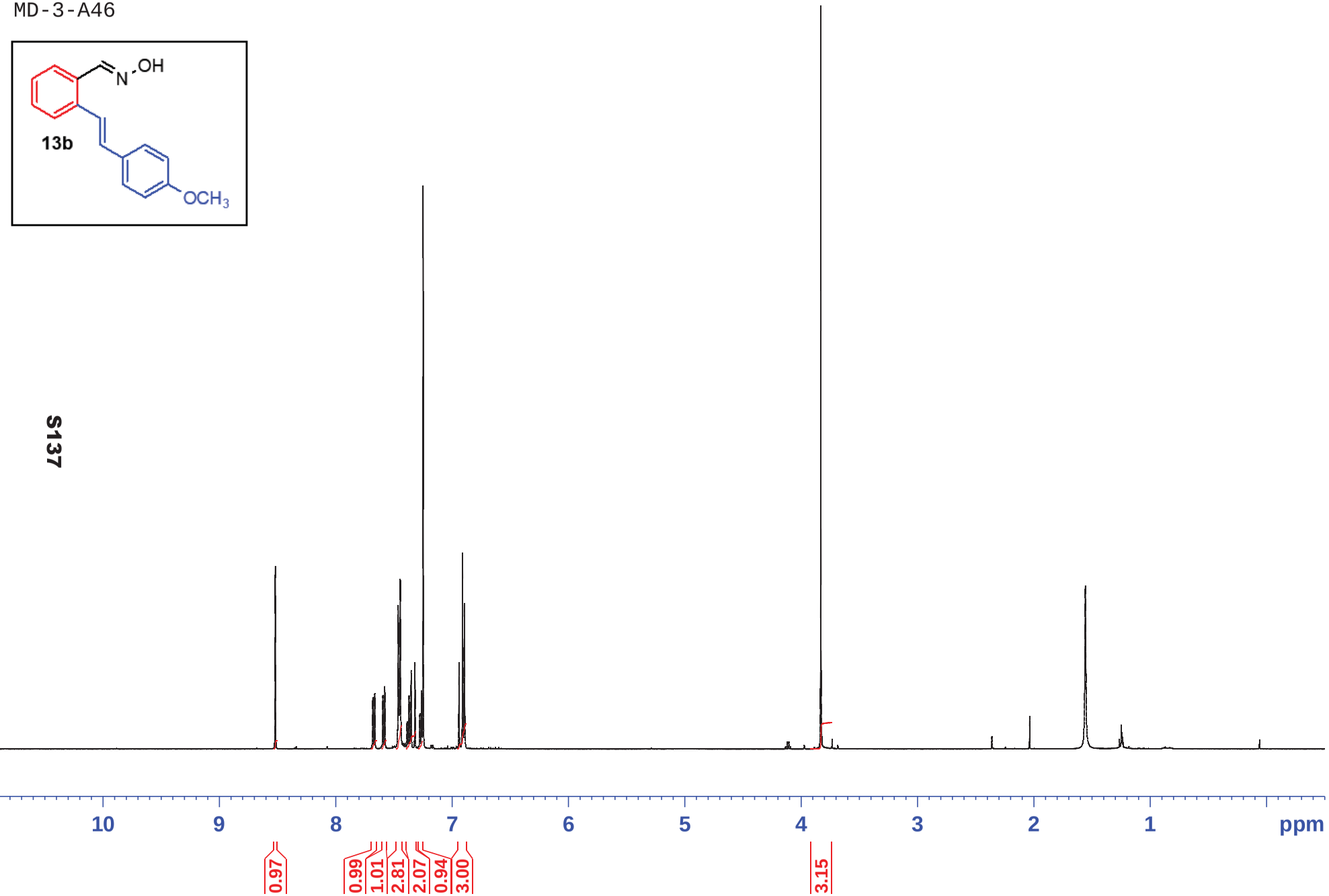
149.60
138.17
137.32
134.50
132.52
130.06
129.59
129.47
127.62
127.60
126.89
126.82
124.62
21.43

S136

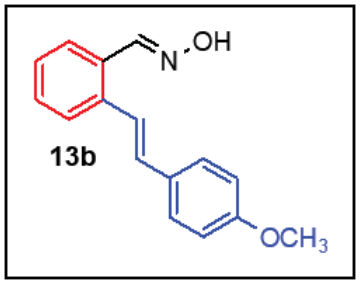
200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

MOI-FGF23-A-21(5)
MD-3-A46

S137



MOI-FGF23-A-21(5)
MD-3-A46

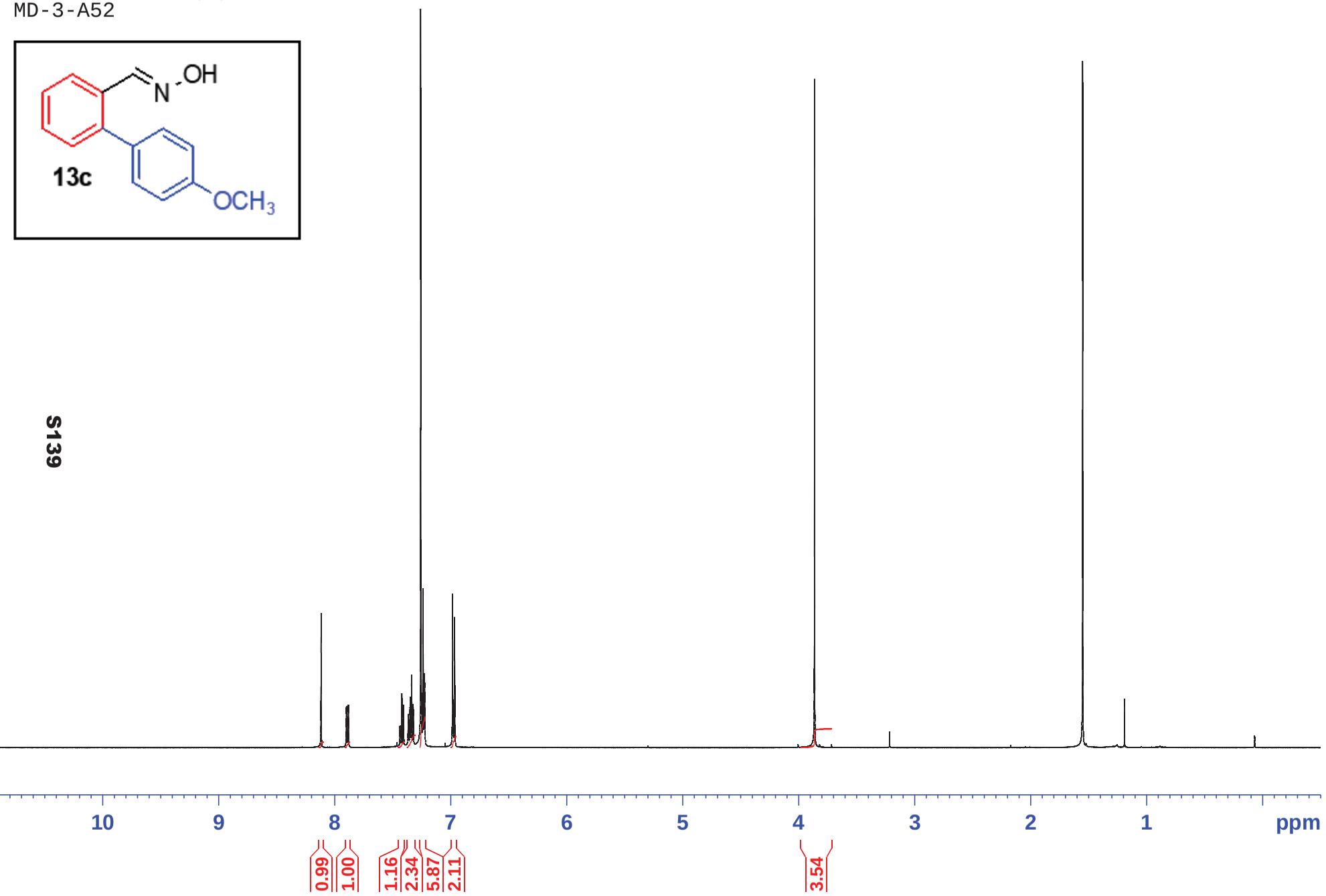


159.68
149.60
137.37
132.02
130.03
129.97
129.27
128.08
127.54
127.37
126.70
123.40
114.24
55.42

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

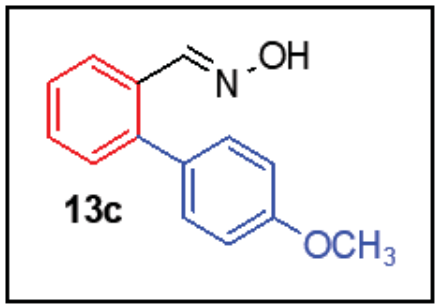
MOI-FGF23-A-71(5)
MD-3-A52

S139



MOI-FGF23-A-71(5)
MD-3-A52

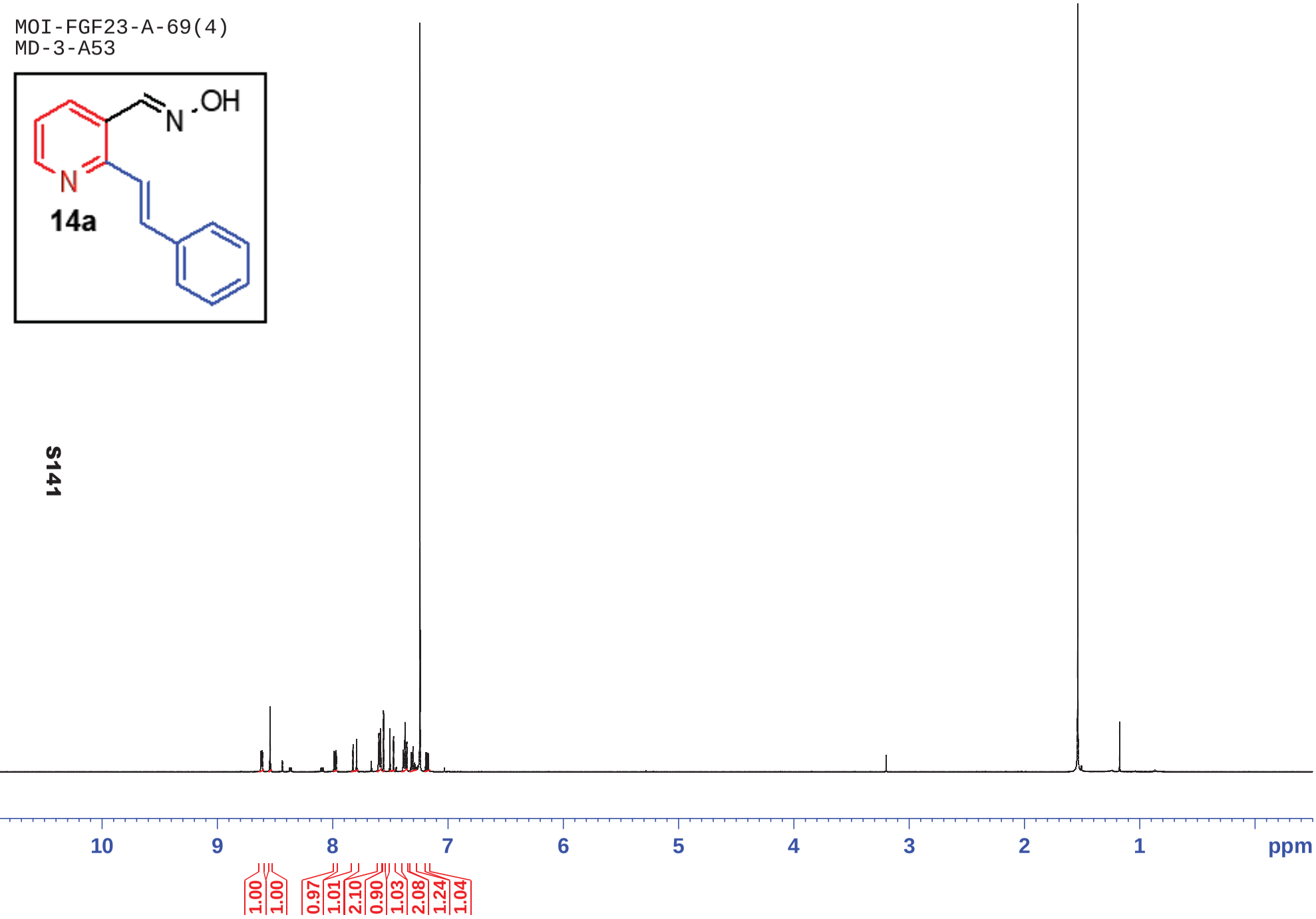
159.33
150.15
142.05
131.96
130.99
130.45
129.84
129.75
127.42
126.25
113.98
55.51



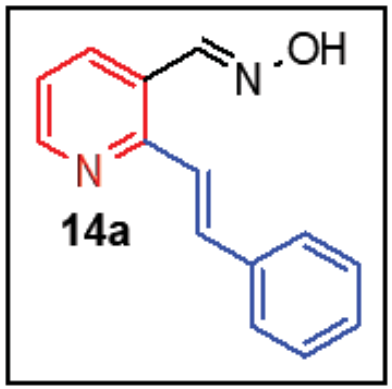
S140

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

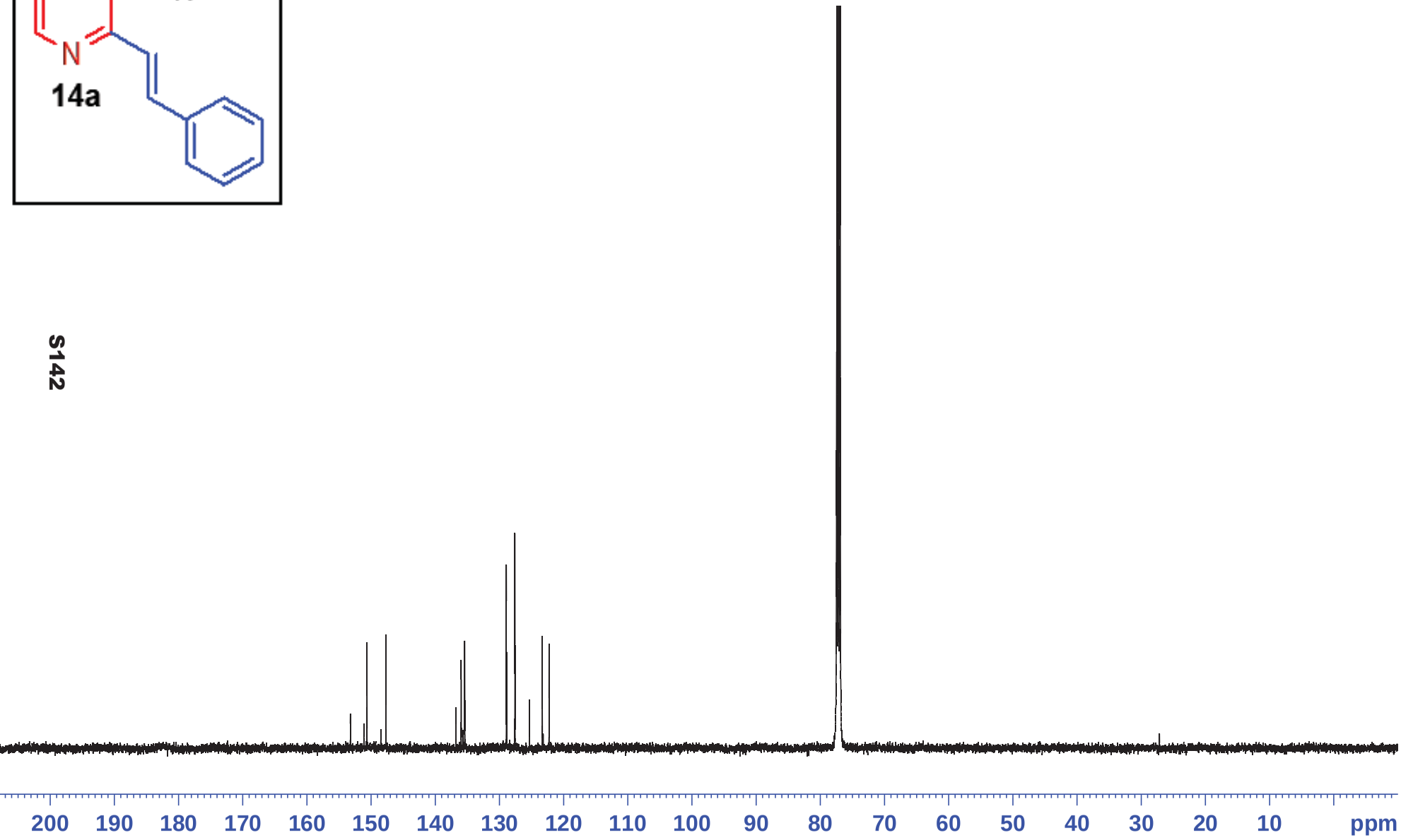
MOI-FGF23-A-69(4)
MD-3-A53



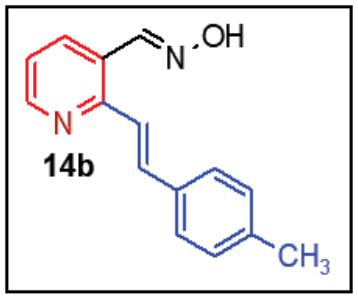
MOI-FGF23-A-69(4)
MD-3-A53

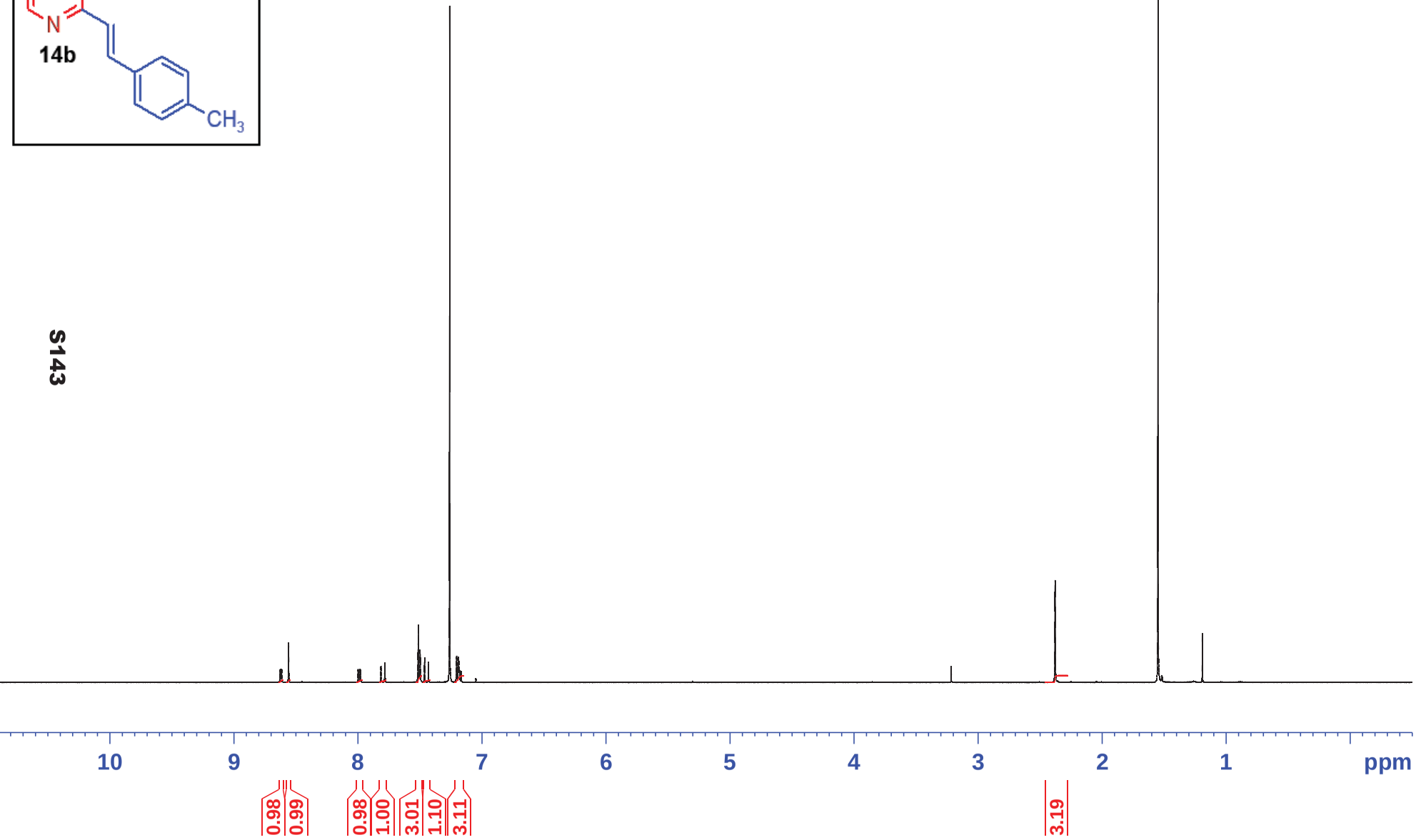


153.22
150.63
147.68
136.77
135.94
135.39
128.90
128.83
127.57
125.30
123.31
122.25

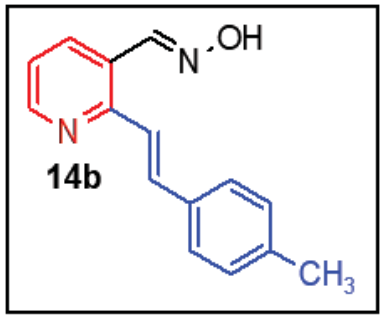


MOI-FGF23-A-85(3)
MD-3-A54





MOI-FGF23-A-85(3)
MD-3-A54

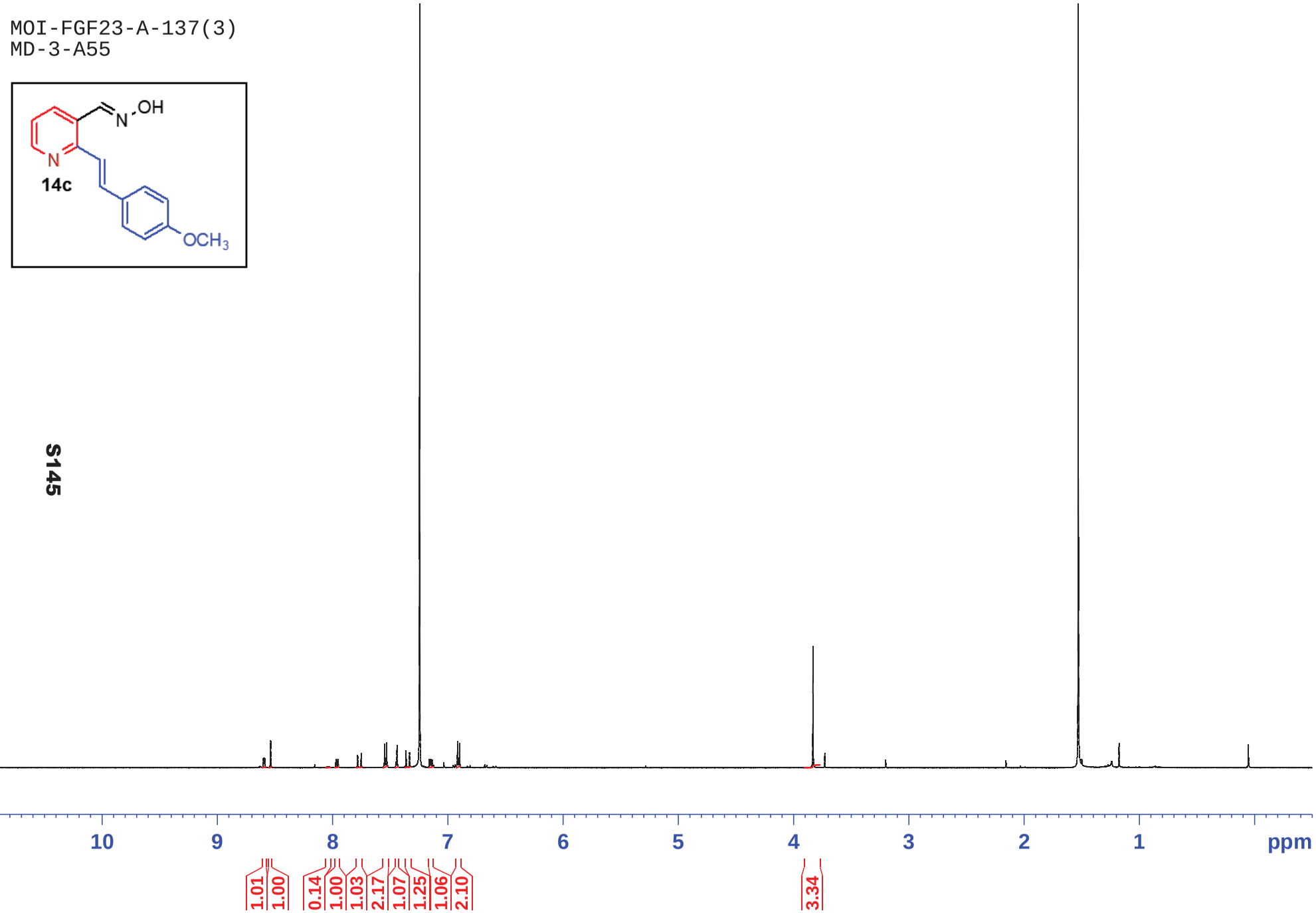


153.05
149.93
147.32
139.22
136.71
135.93
133.83
129.66
127.63
125.52
122.11
121.52
21.54

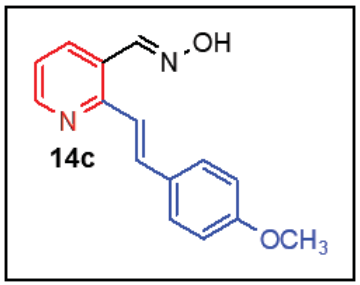
S144

200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

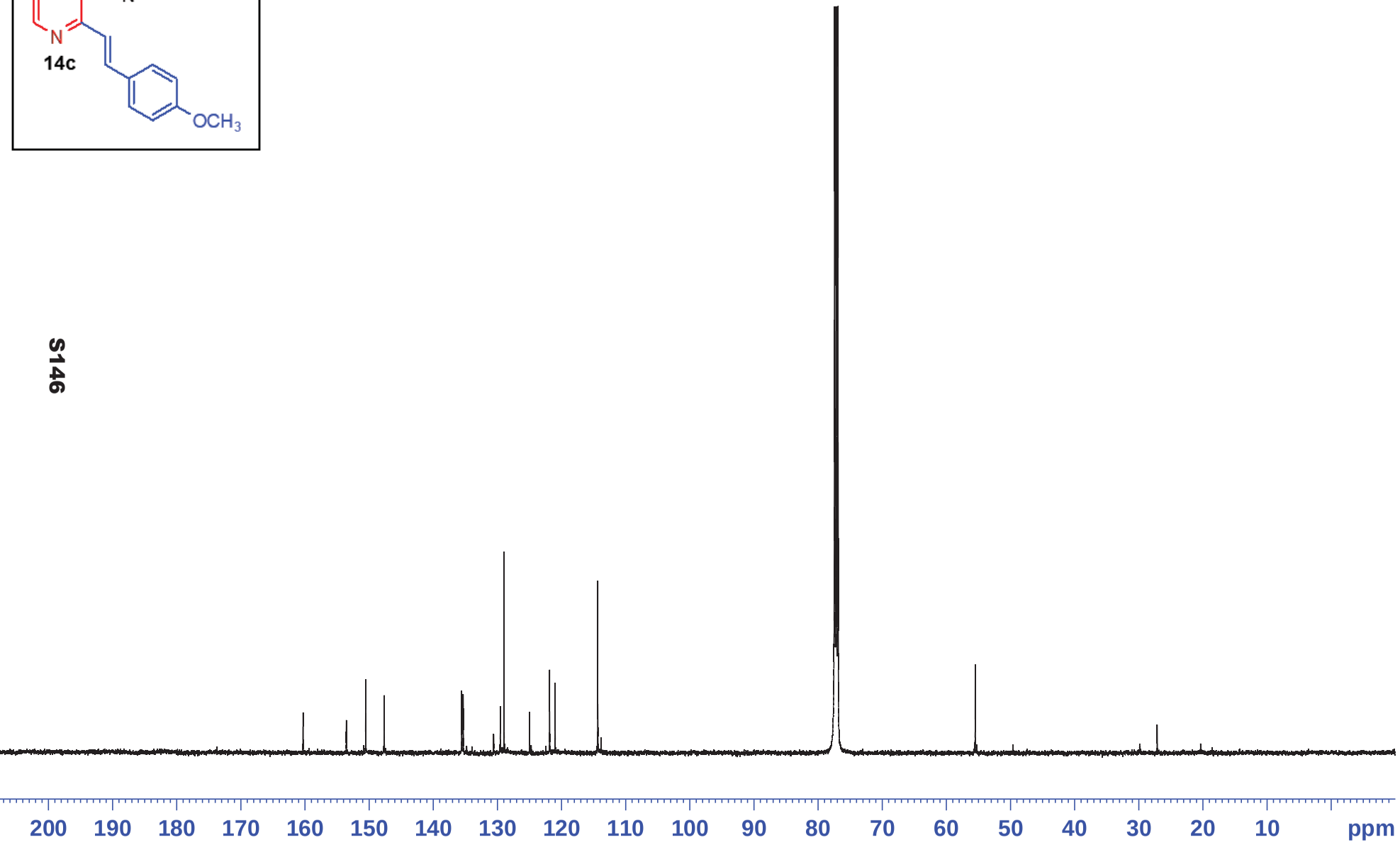
MOI-FGF23-A-137(3)
MD-3-A55



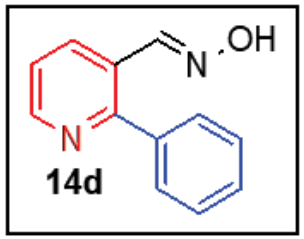
AEL-FGF23-A-139(2)
MD-3-A55



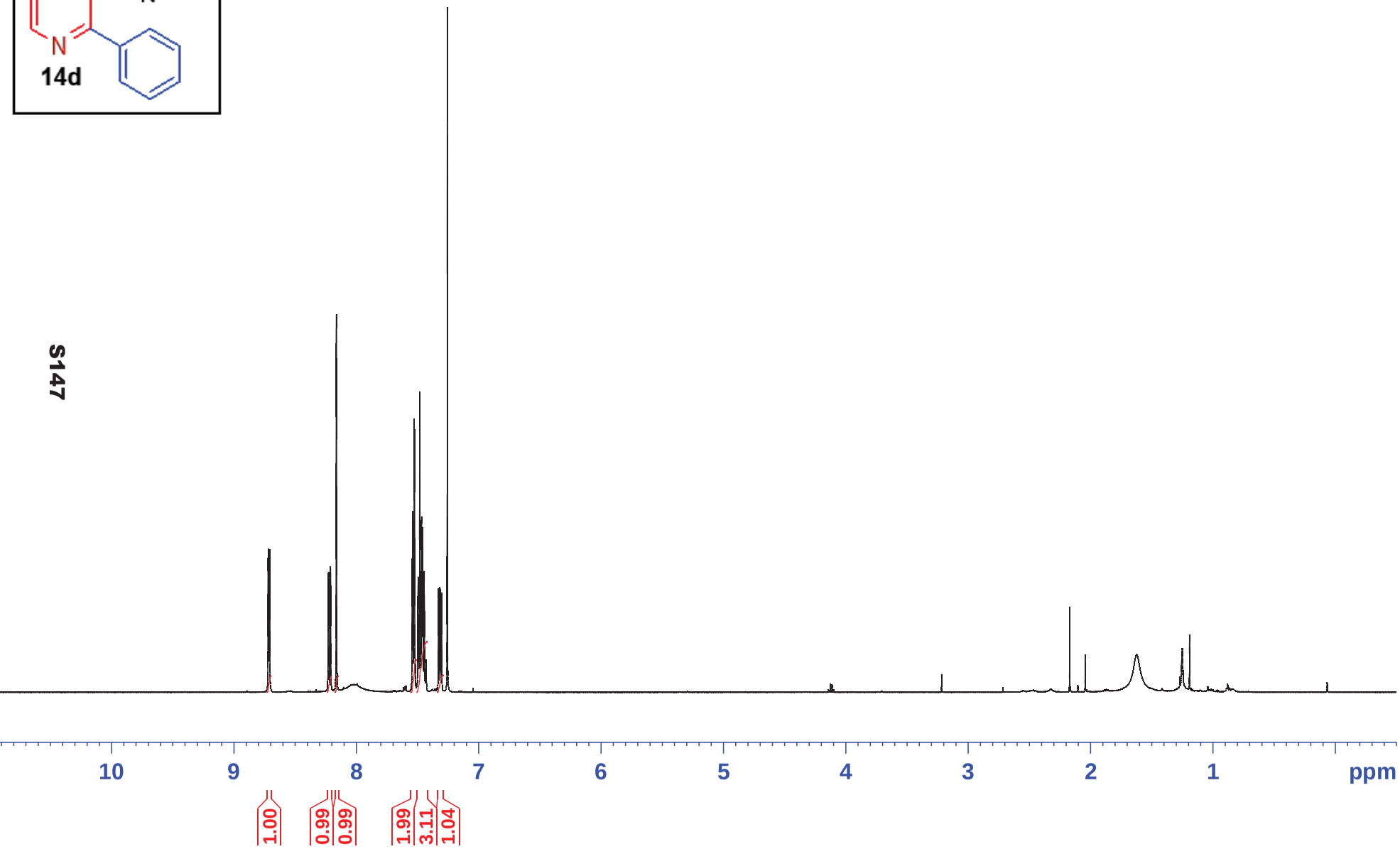
160.30
153.55
150.53
147.64
135.57
135.32
129.54
128.96
124.97
121.85
120.99
114.34
55.48



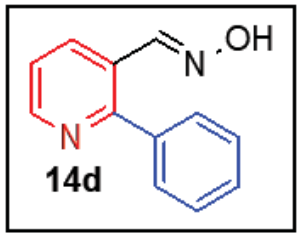
MOI-FGF23-67(6)
MD-3-A56



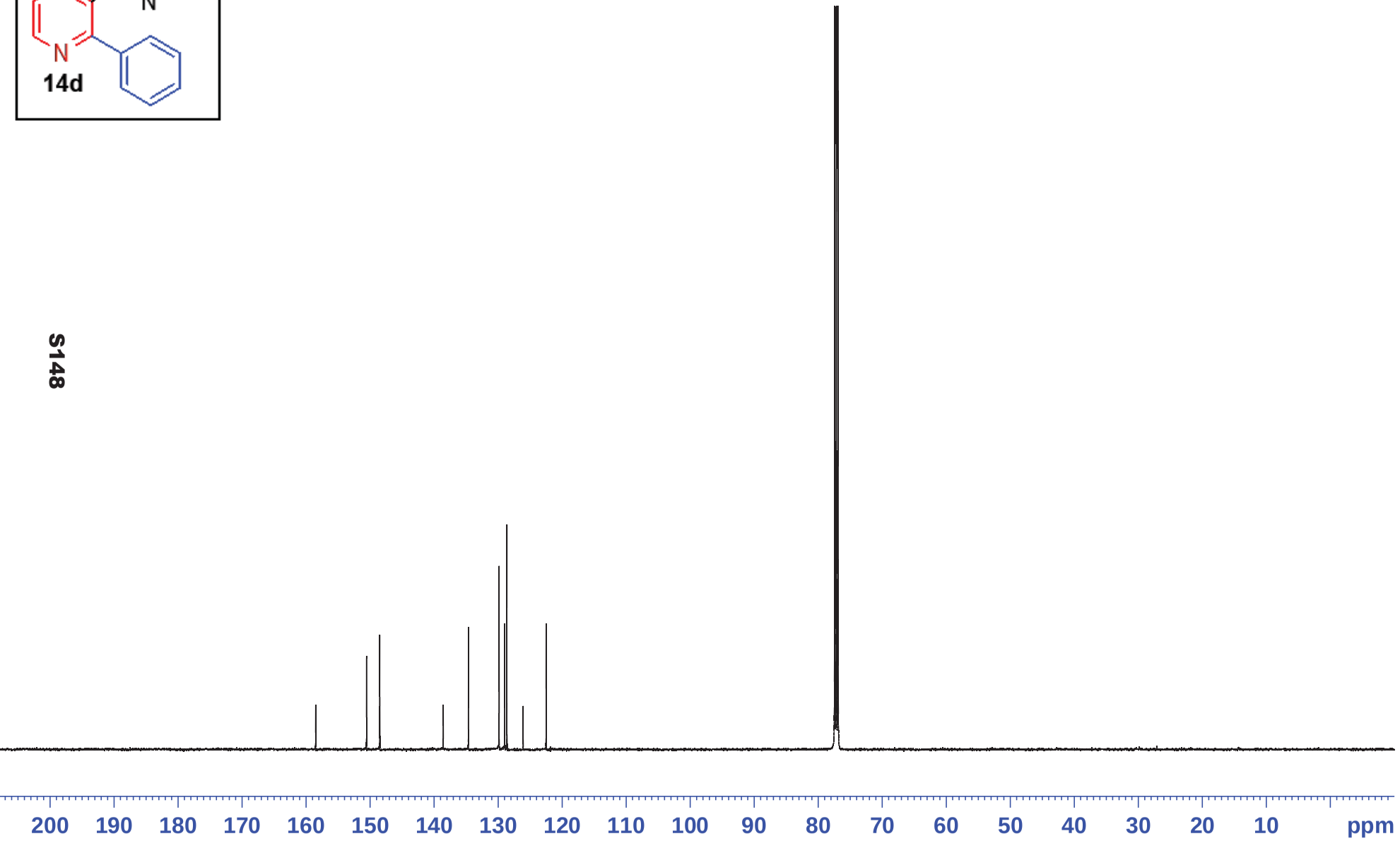
S147



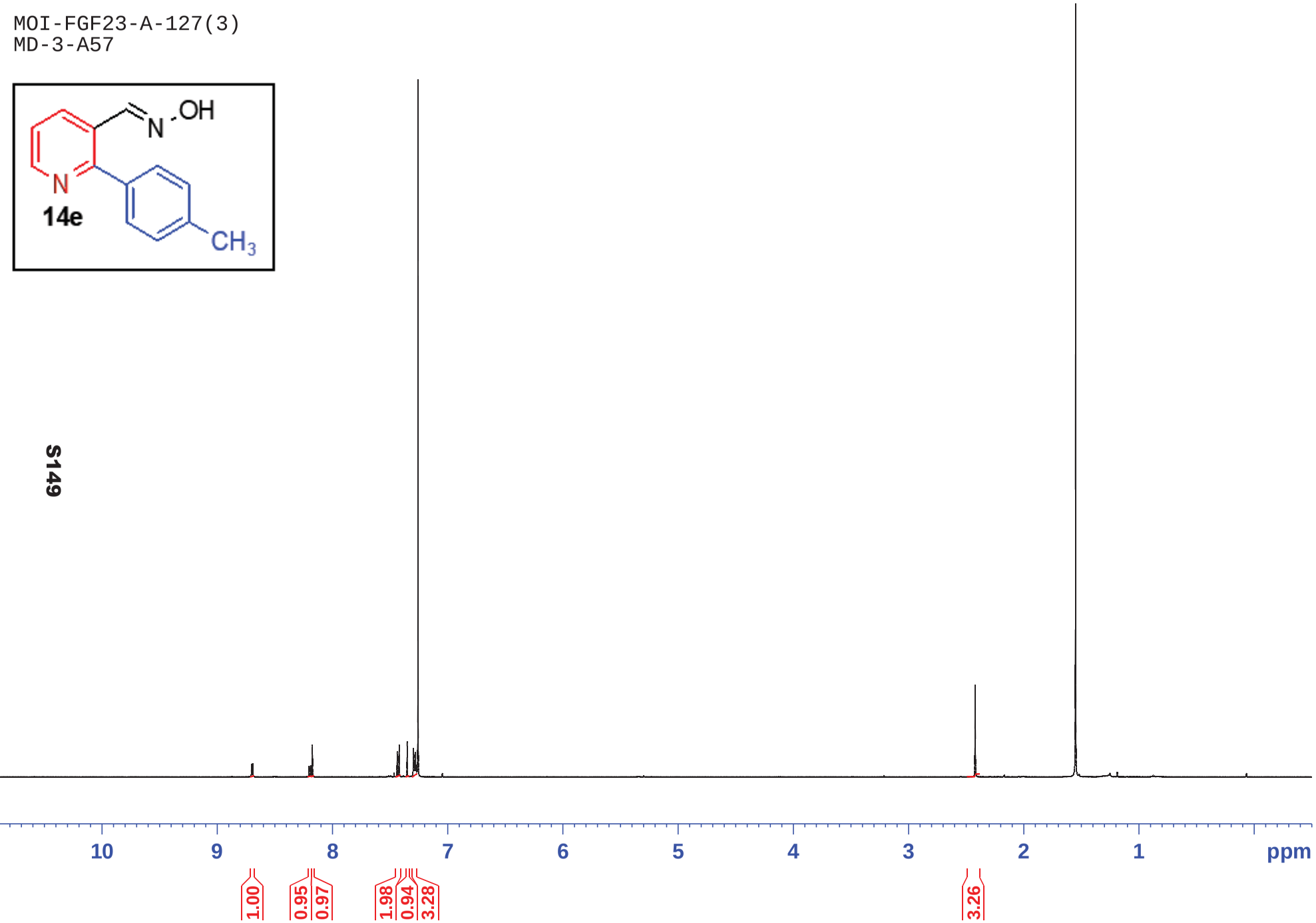
MOI-FGF23-A-67(4)
MD-3-A56



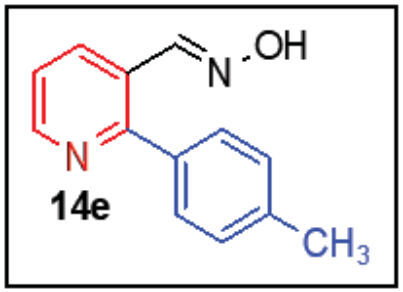
158.49
150.54
148.48
138.56
134.62
129.83
128.97
128.61
126.07
122.49



MOI-FGF23-A-127(3)
MD-3-A57

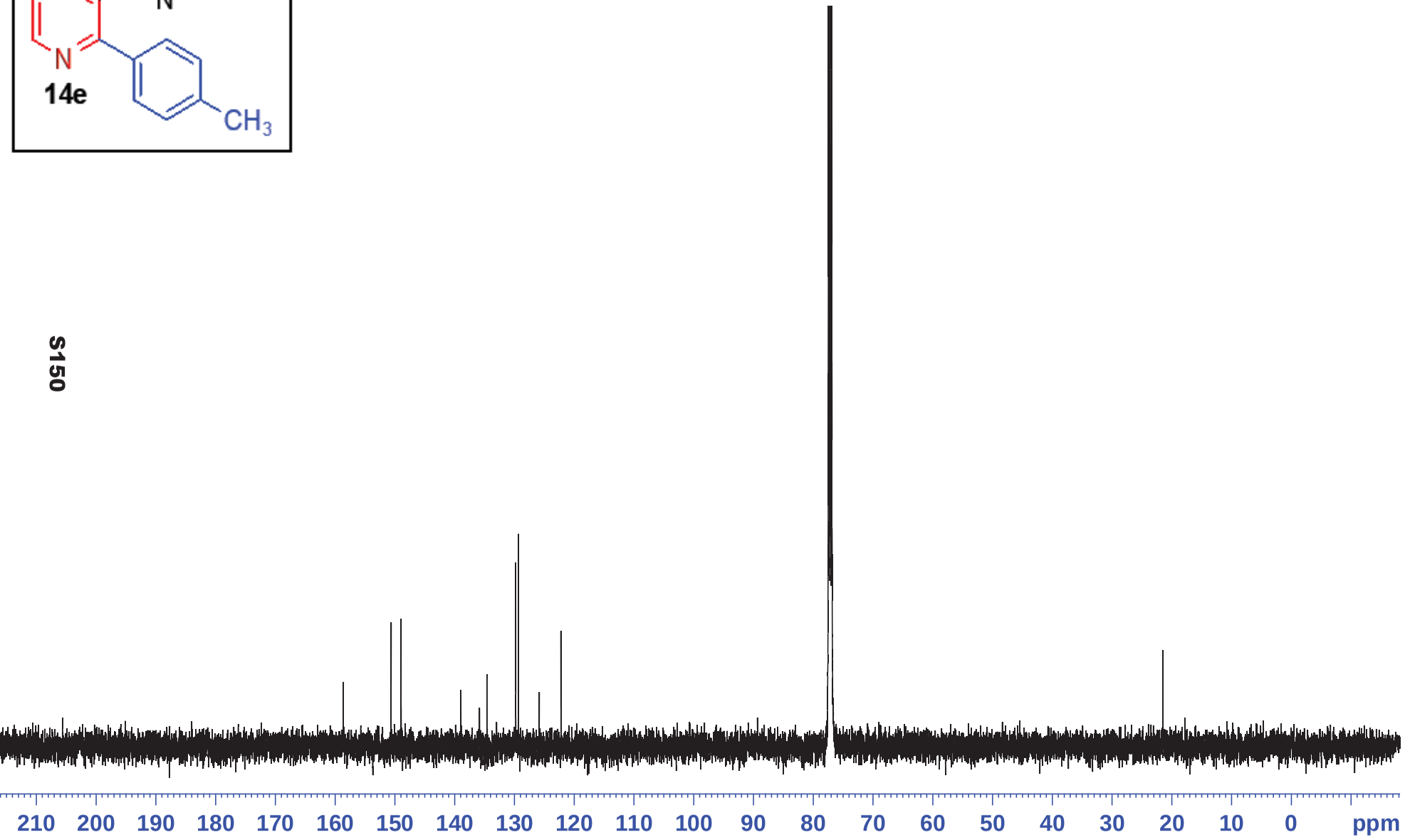


MOI-FGF23-A-127(3)
MD-3-A57



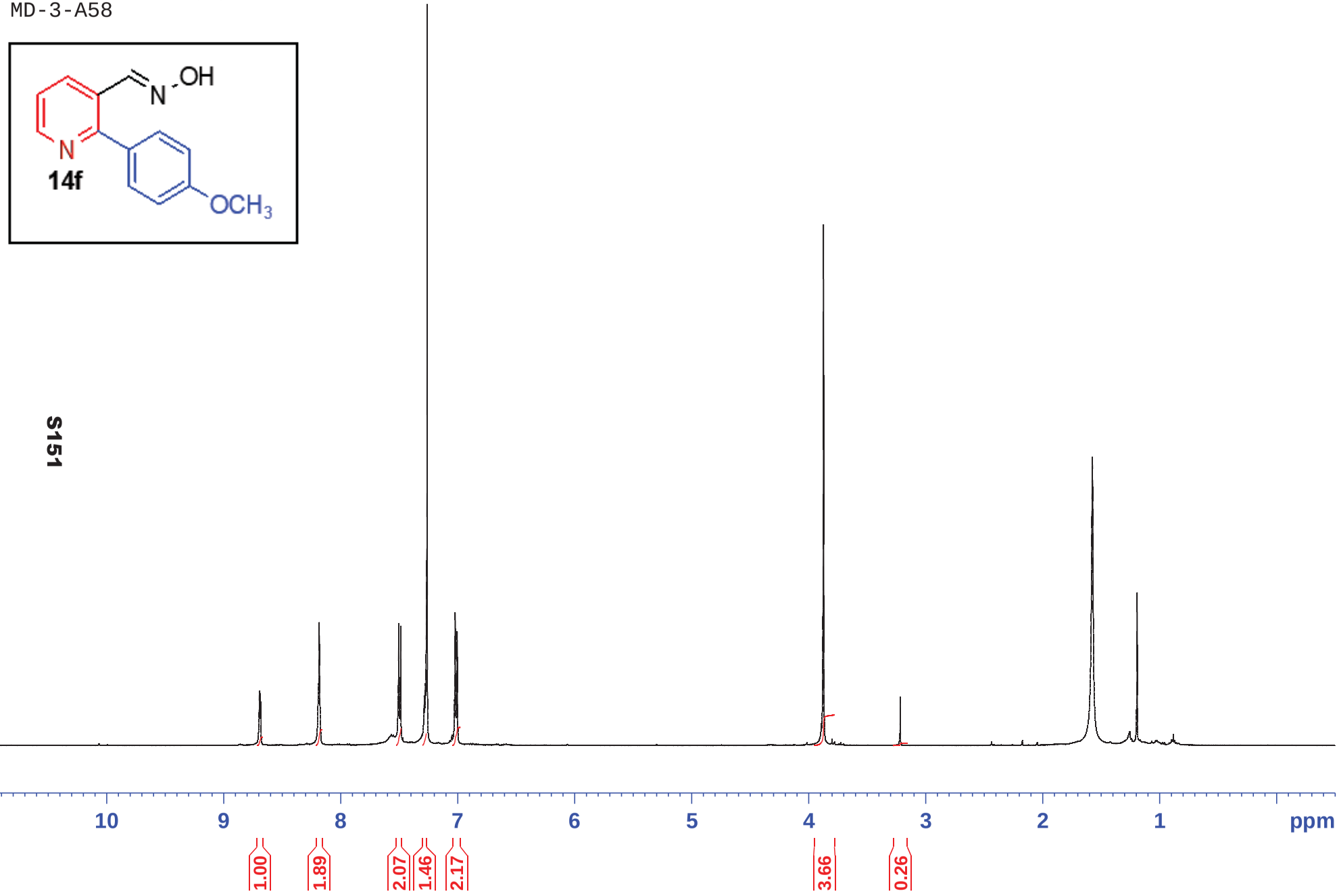
158.61
150.60
148.94
138.94
135.81
134.58
129.77
129.31
125.82
122.22
21.45

S150

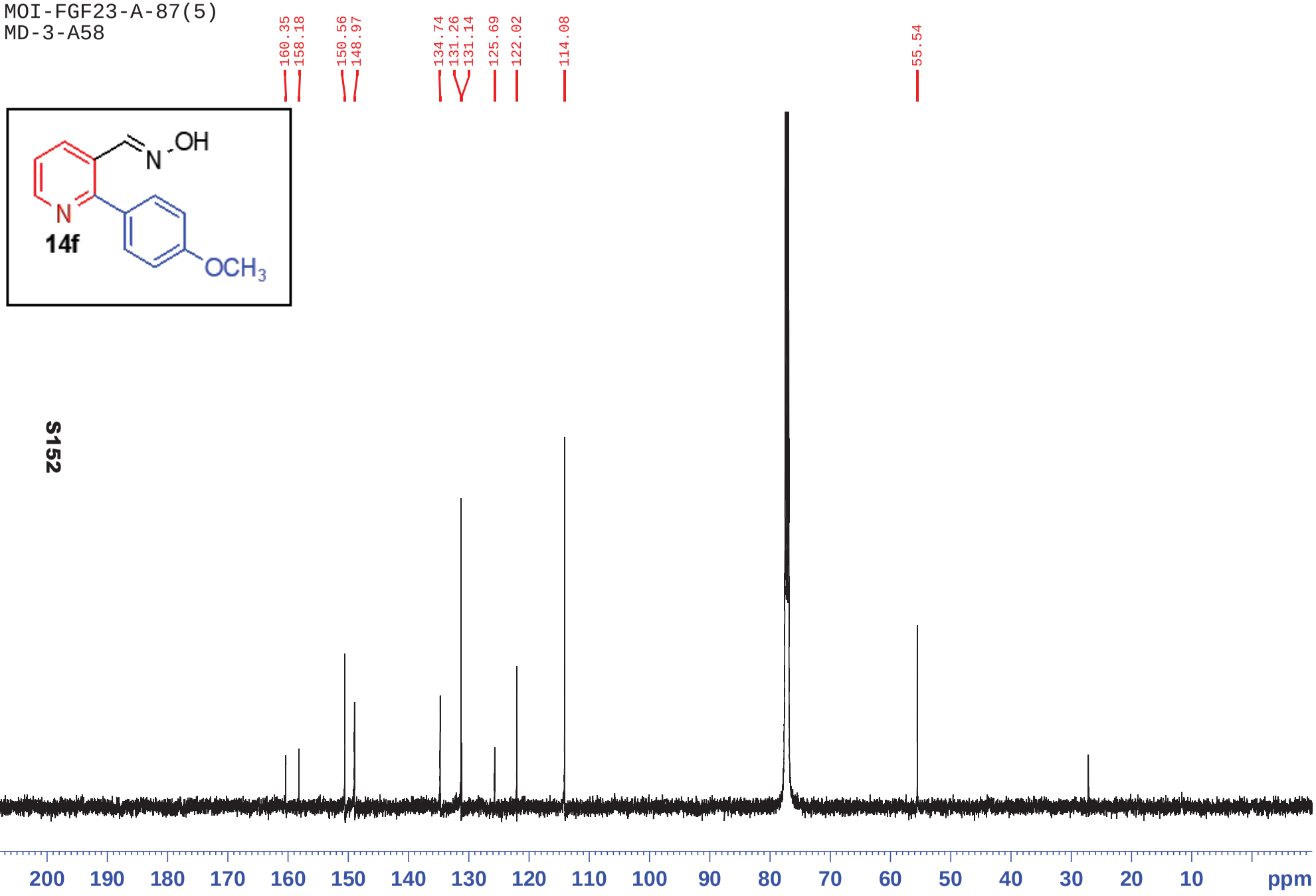


MOI-FGF23-A-87(5)
MD-3-A58

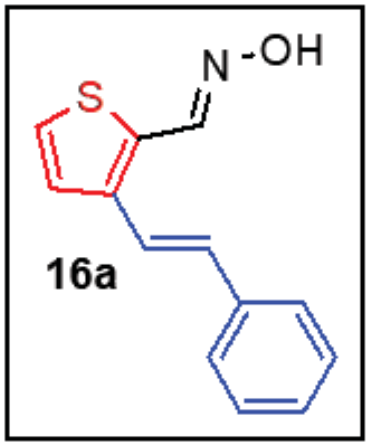
S151



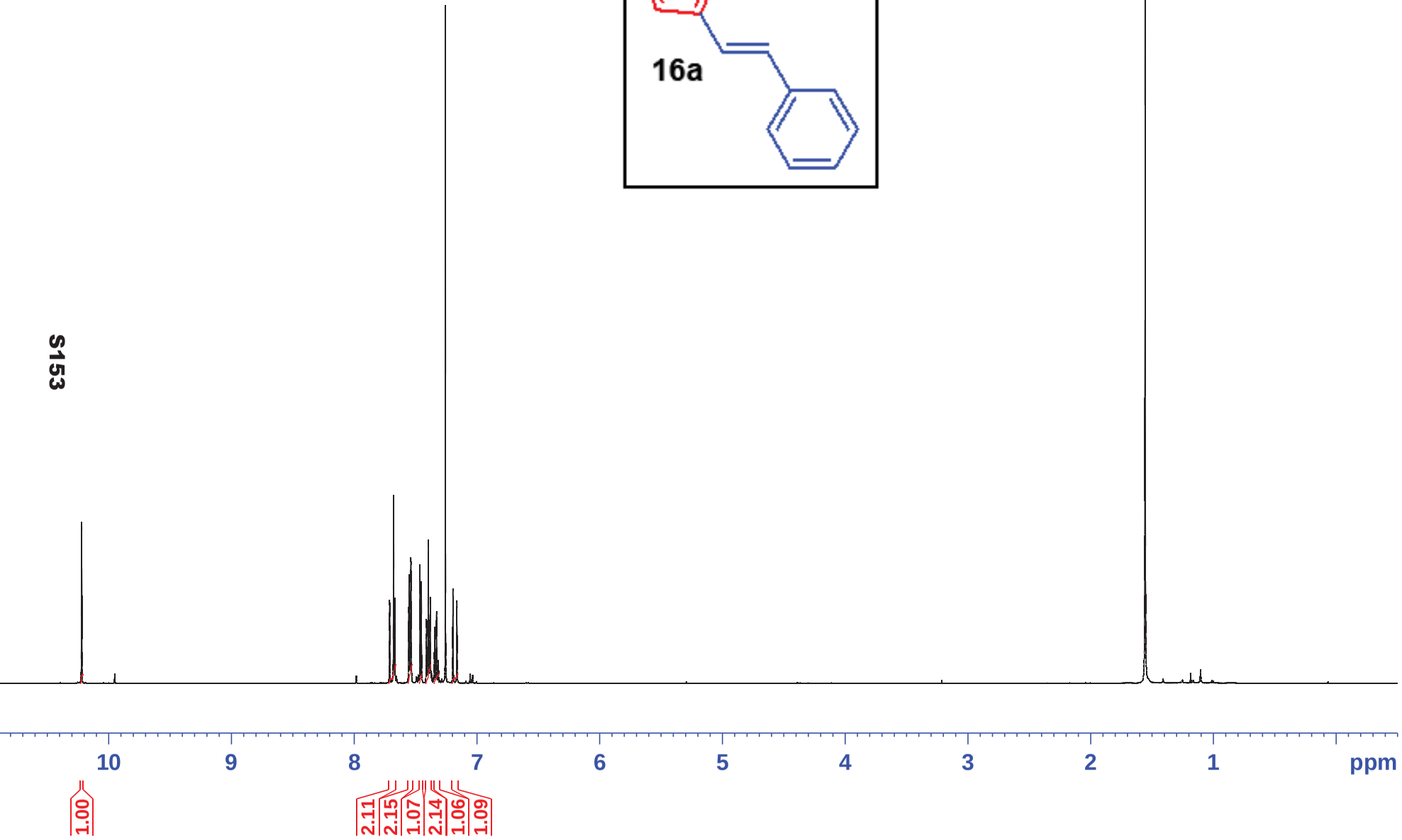
MOI-FGF23-A-87(5)
MD-3-A58



MOI-FGF23-A-77(5)
RCHO of MD-3-A59

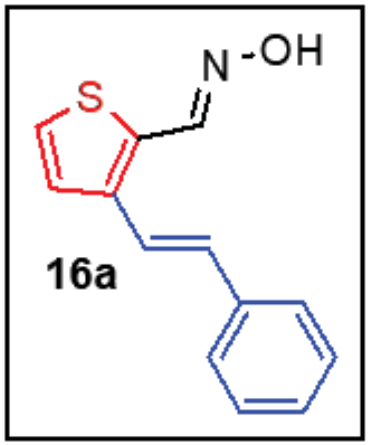


S153



MOI-FGF23-A-77(5)
RCHO of MD-3-A59

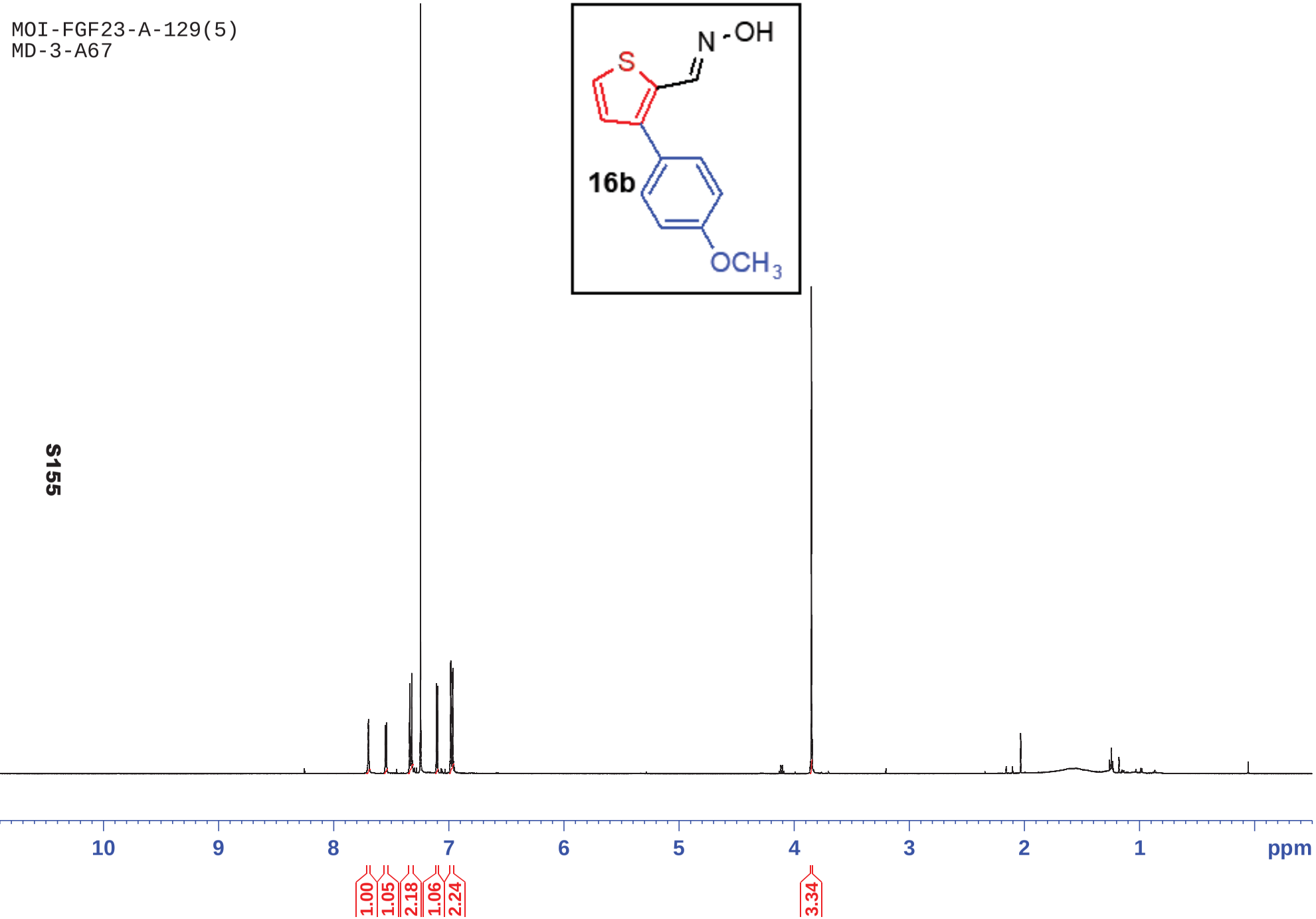
182.18
146.88
137.67
136.40
134.89
134.52
129.05
128.96
127.11
126.94
119.46



200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 ppm

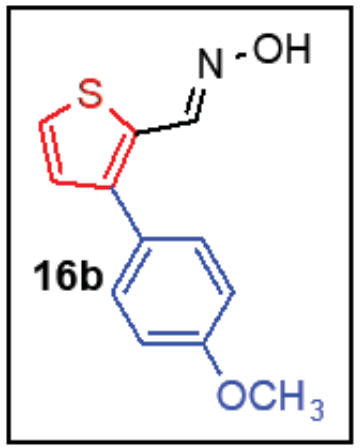
MOI-FGF23-A-129(5)
MD-3-A67

S155



MOI-FGF23-A-129(5)
MD-3-A67

159.60
146.08
141.32
130.85
130.38
128.53
128.15
124.87
114.23
55.52



S156

210 200 190 180 170 160 150 140 130 120 110 100 90 80 70 60 50 40 30 20 10 0 ppm

Compound 1 | Rf 200 | Thursday 20 July 2017 08:56AM

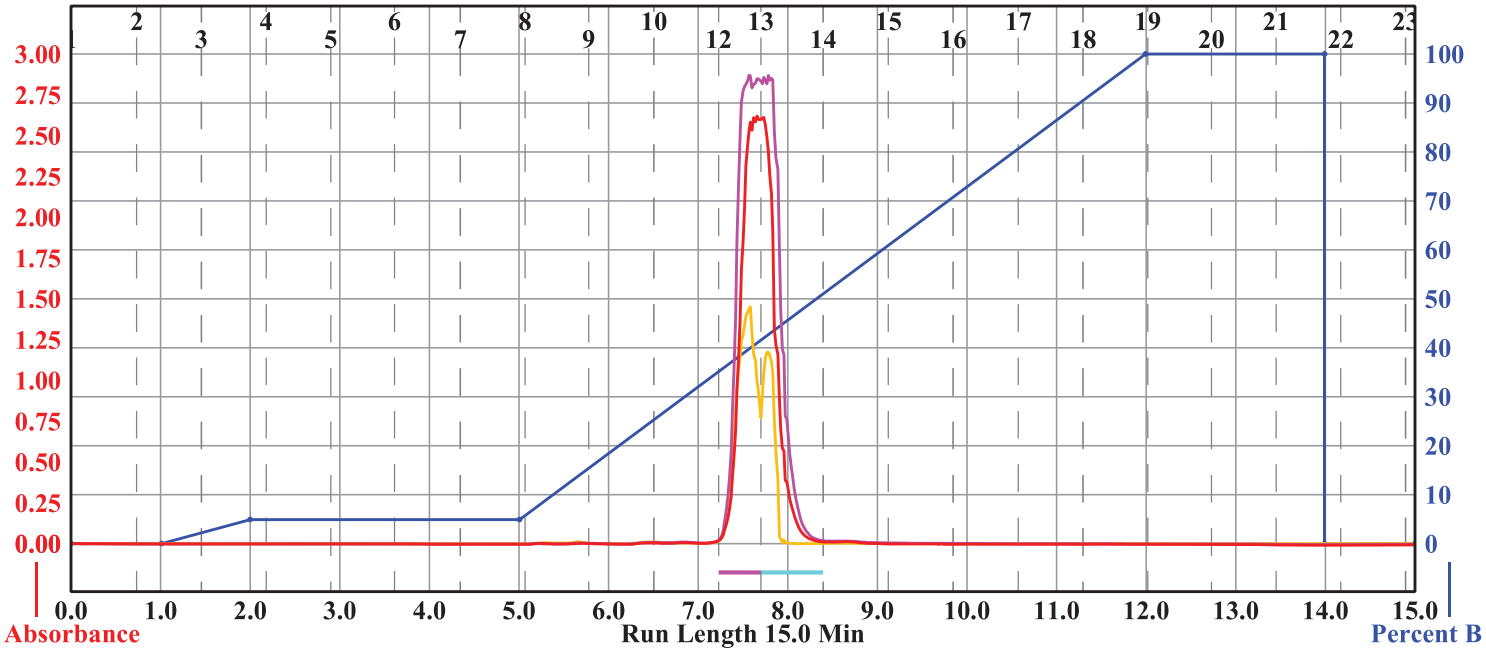
Sample: JWC-FGF23-A-71

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:12	A:12
2	A:13	A:13

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol

S157

Compound 6b | Rf 200 | Tuesday 25 July 2017 11:55AM

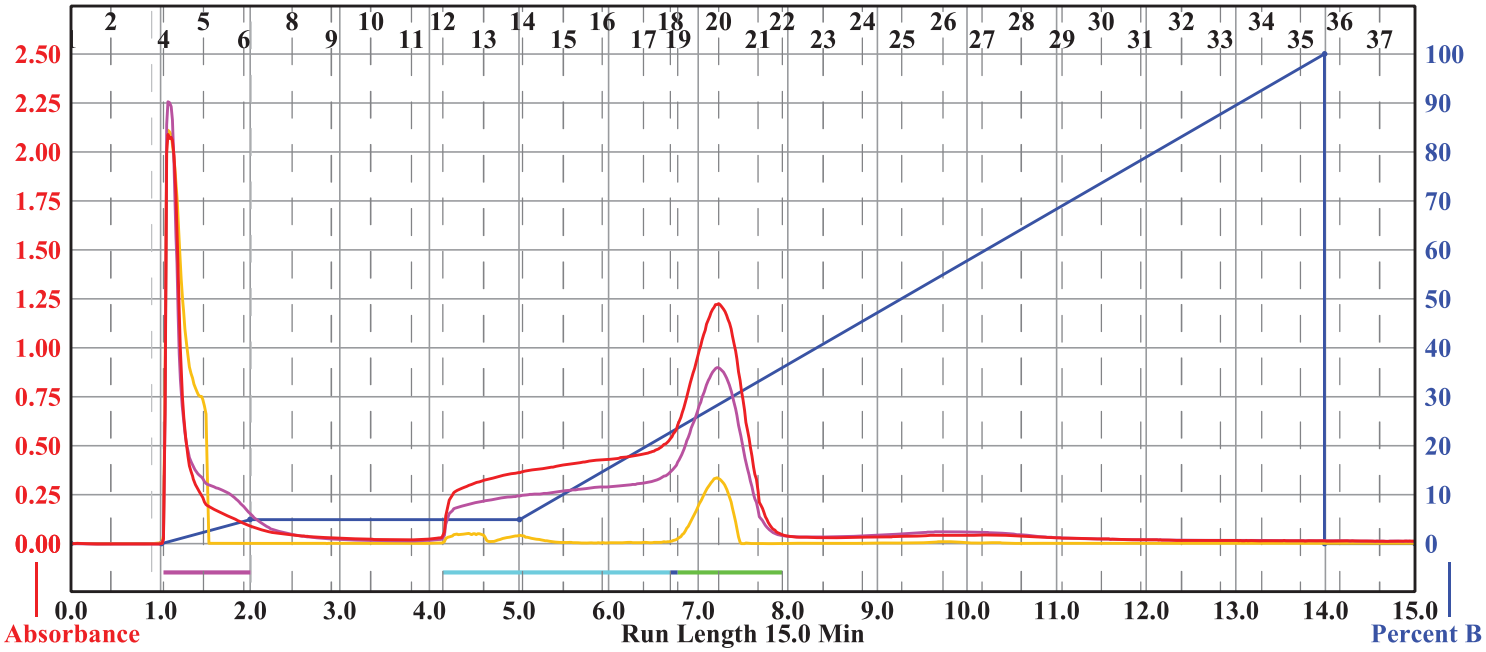
Sample: JWC-FGF23-A-73

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 9.5 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:4	A:6
2	A:12	A:17
3	A:18	A:18
4	A:19	A:21

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
9.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S158

Compound 6c | Rf 200 | Tuesday 25 July 2017 09:25AM

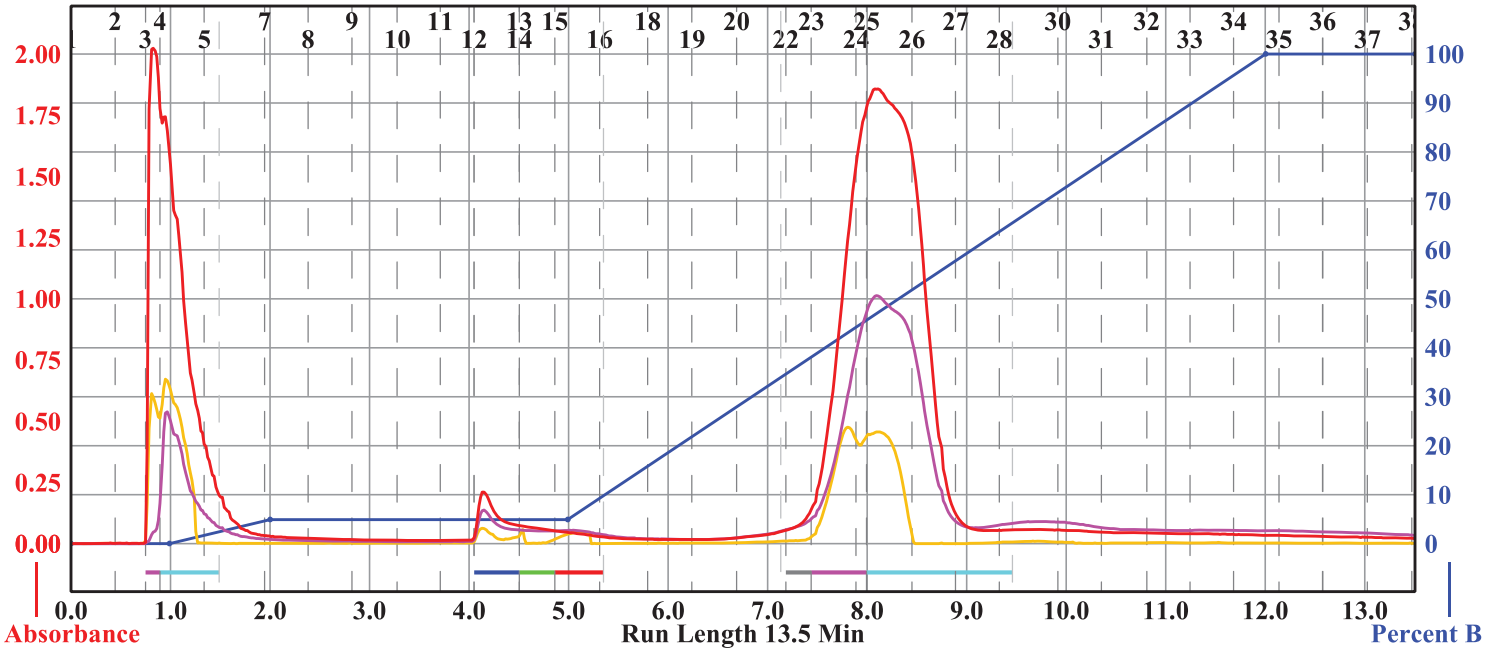
Sample: JWC-FGF23-A-75

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:3
2	A:4	A:5
3	A:12	A:13
4	A:14	A:14
5	A:15	A:16
6	A:22	A:22
7	A:23	A:24
8	A:25	A:28

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
1.5	100.0	A1 hexane	B2 methanol

S159

Compound 6d | Rf 200 | Tuesday 18 July 2017 02:30PM

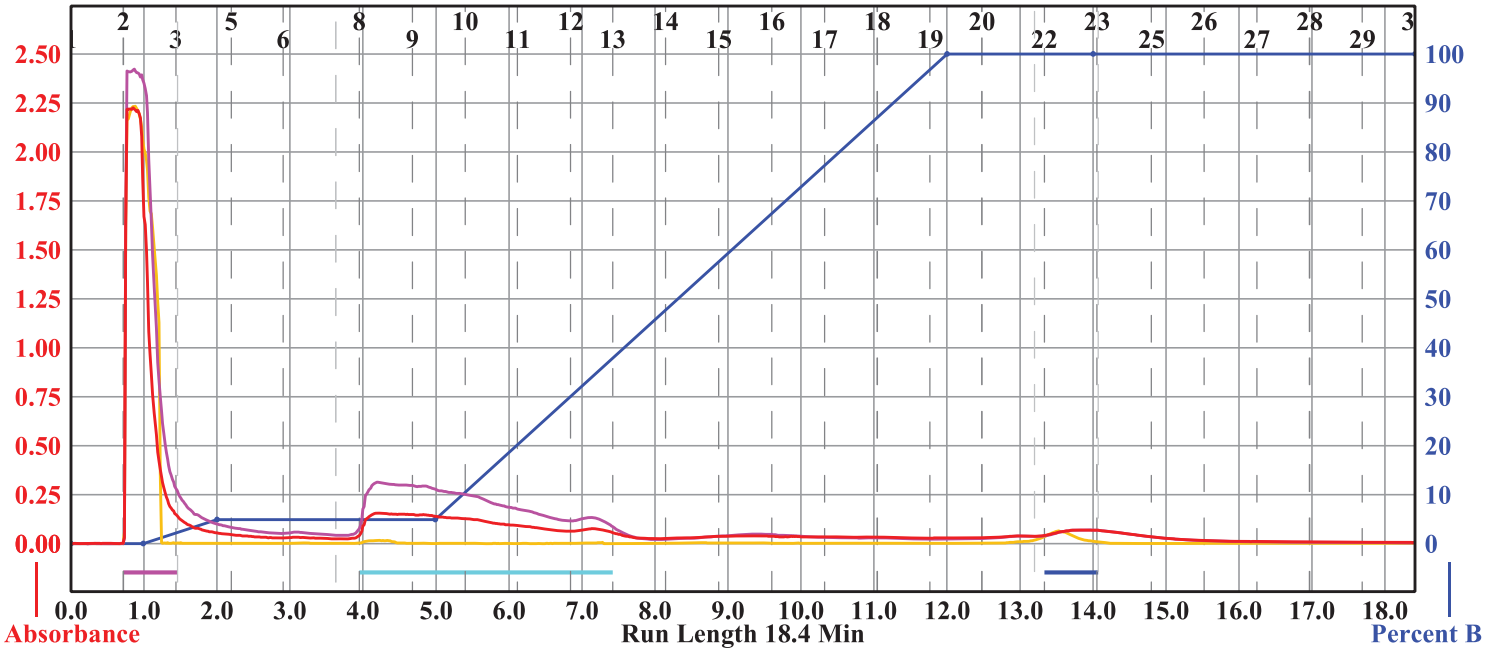
Sample: JWC-B-155

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

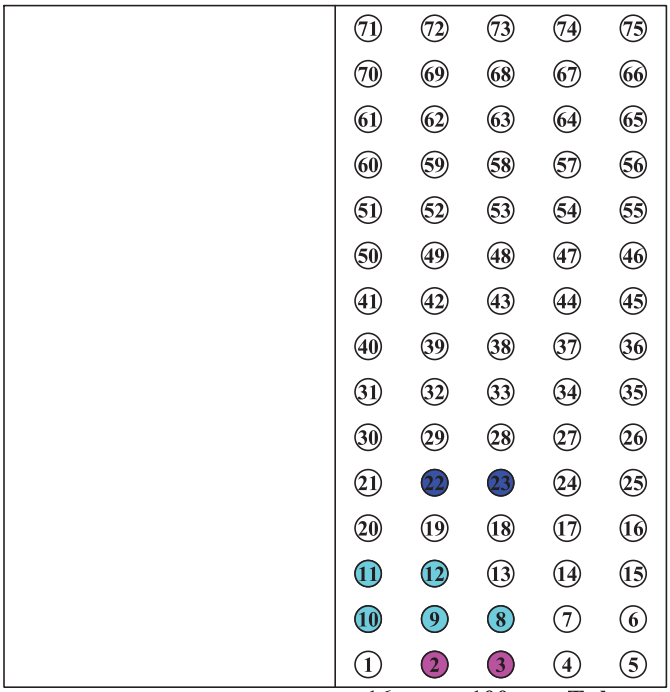
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:3
2	A:8	A:12
3	A:22	A:23

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
4.4	100.0	A1 hexane	B2 methanol

S160

Compound 6e

Rf 200

Thursday 15 December 2016 03:12PM

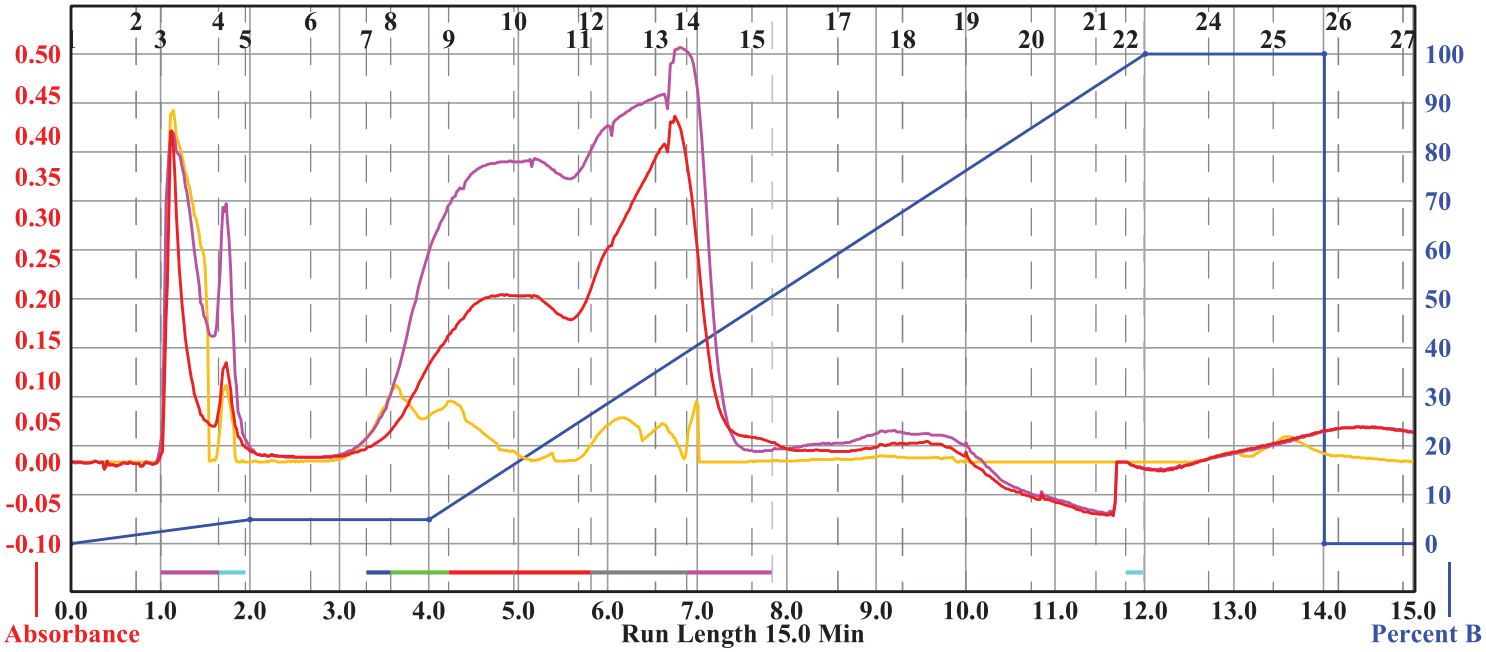
Sample: RPD-FGF23-A-17

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A2 dichloromethane
Solvent: B2 methanol

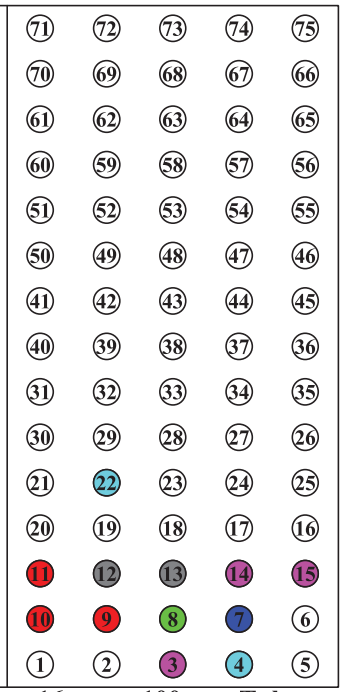
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:3
2	A:4	A:4
3	A:7	A:7
4	A:8	A:8
5	A:9	A:11
6	A:12	A:13
7	A:14	A:15
8	A:22	A:22

Duration	%B	Solvent A	Solvent B
0.0	0.0	A2 dichlorometha	B2 methanol
2.0	4.9	A2 dichlorometha	B2 methanol
2.0	4.9	A2 dichlorometha	B2 methanol
8.0	100.0	A2 dichlorometha	B2 methanol
2.0	100.0	A2 dichlorometha	B2 methanol
0.0	0.0	A2 dichlorometha	B2 methanol
1.0	0.0	A2 dichlorometha	B2 methanol

S161

Compound 6f

Rf 200

Thursday 20 July 2017 01:31PM

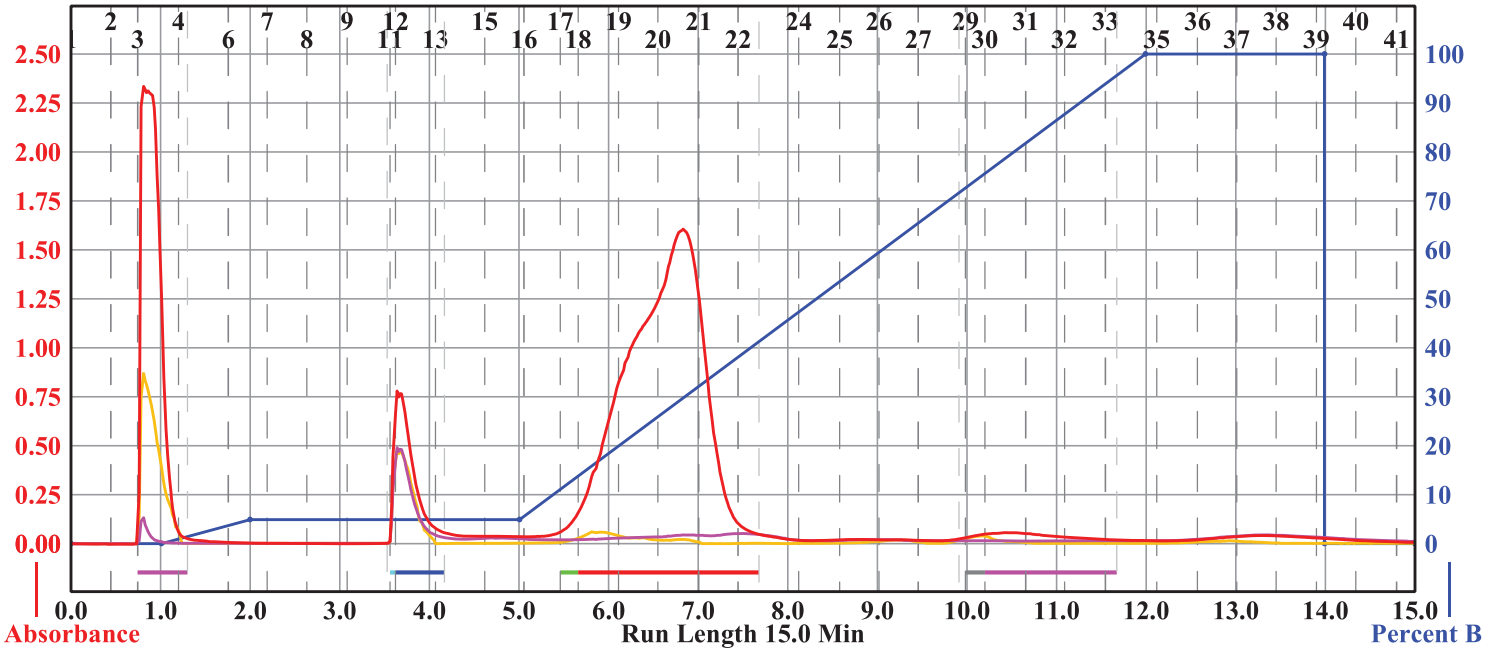
Sample: JWC-B-153

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

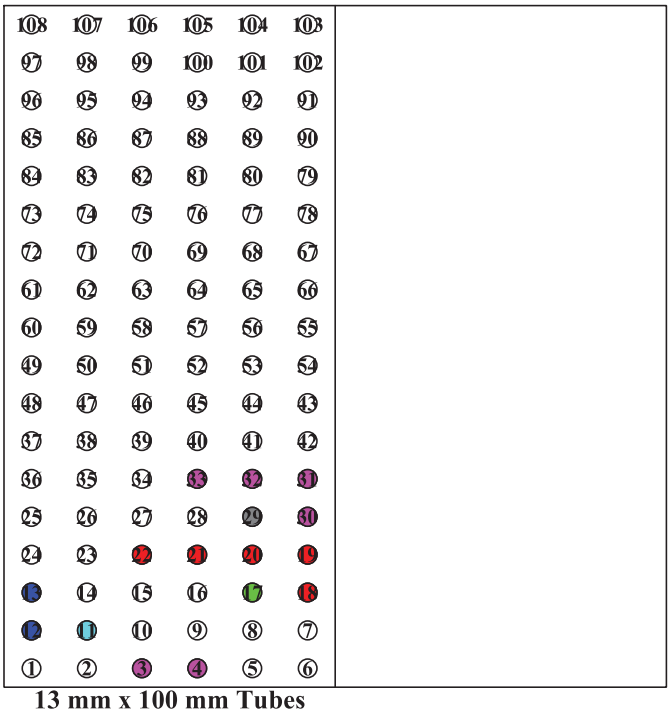
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:4
2	A:11	A:11
3	A:12	A:13
4	A:17	A:17
5	A:18	A:22
6	A:29	A:29
7	A:30	A:33

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol

S162

Compound 6i

Rf 200

Wednesday 19 July 2017 09:52AM

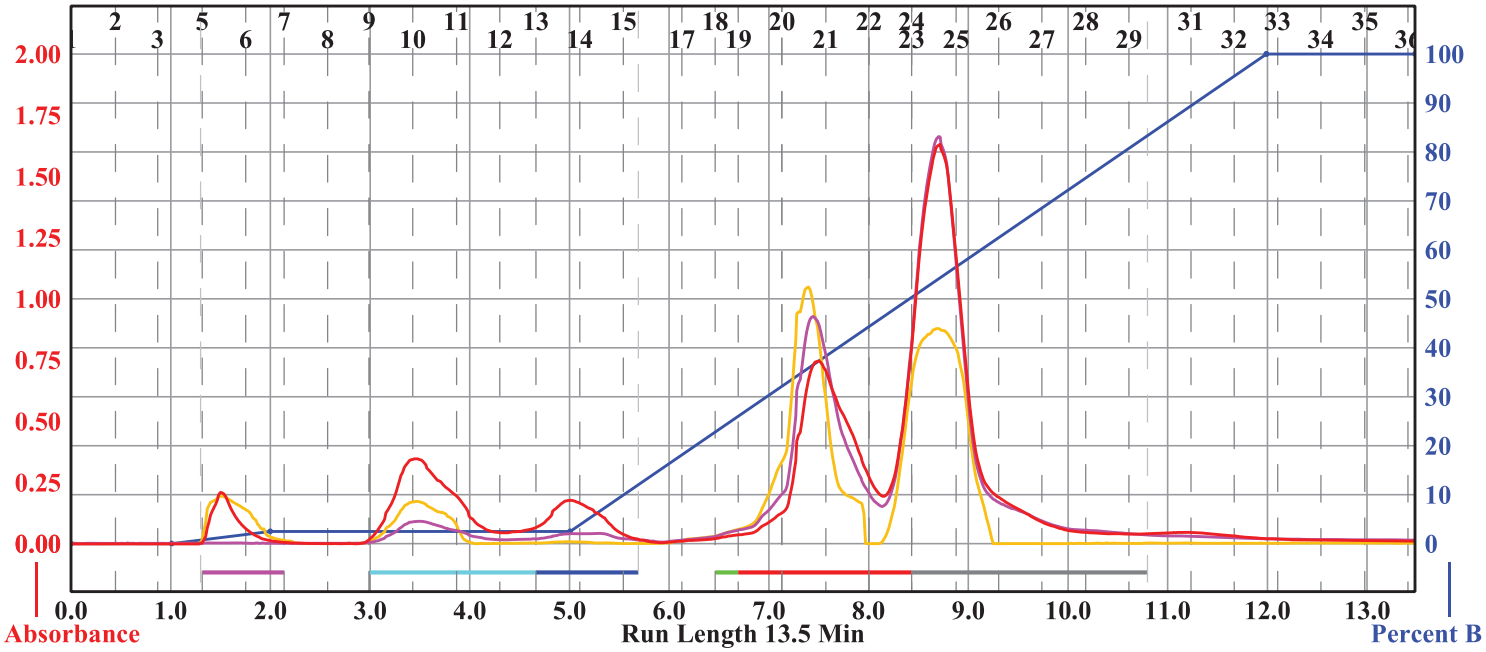
Sample: RPD-FGF23-A-129

4x Column: 3
RediSep Column: Silica 12g
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

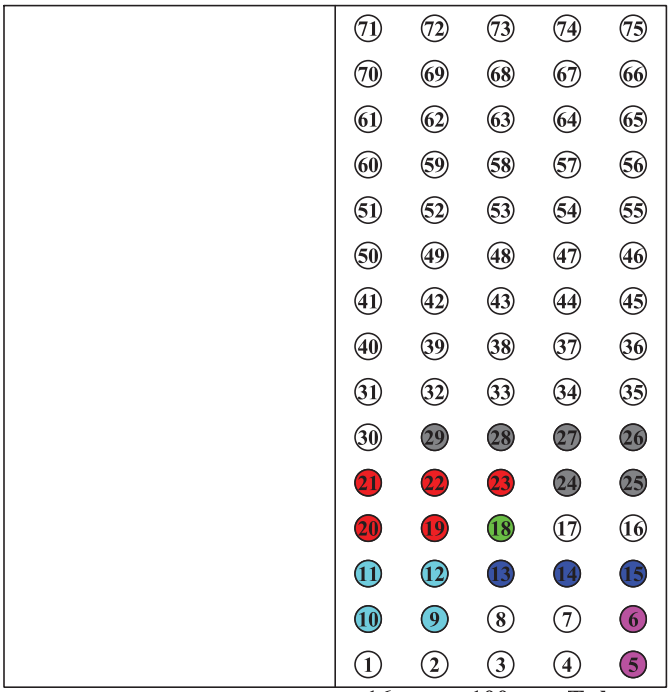
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:5	A:6
2	A:9	A:12
3	A:13	A:15
4	A:18	A:18
5	A:19	A:23
6	A:24	A:29

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
0.0	100.0	A1 hexane	B2 methanol
1.5	100.0	A1 hexane	B2 methanol

S163

Compound 6j | Rf 200 | Monday 17 July 2017 08:58AM

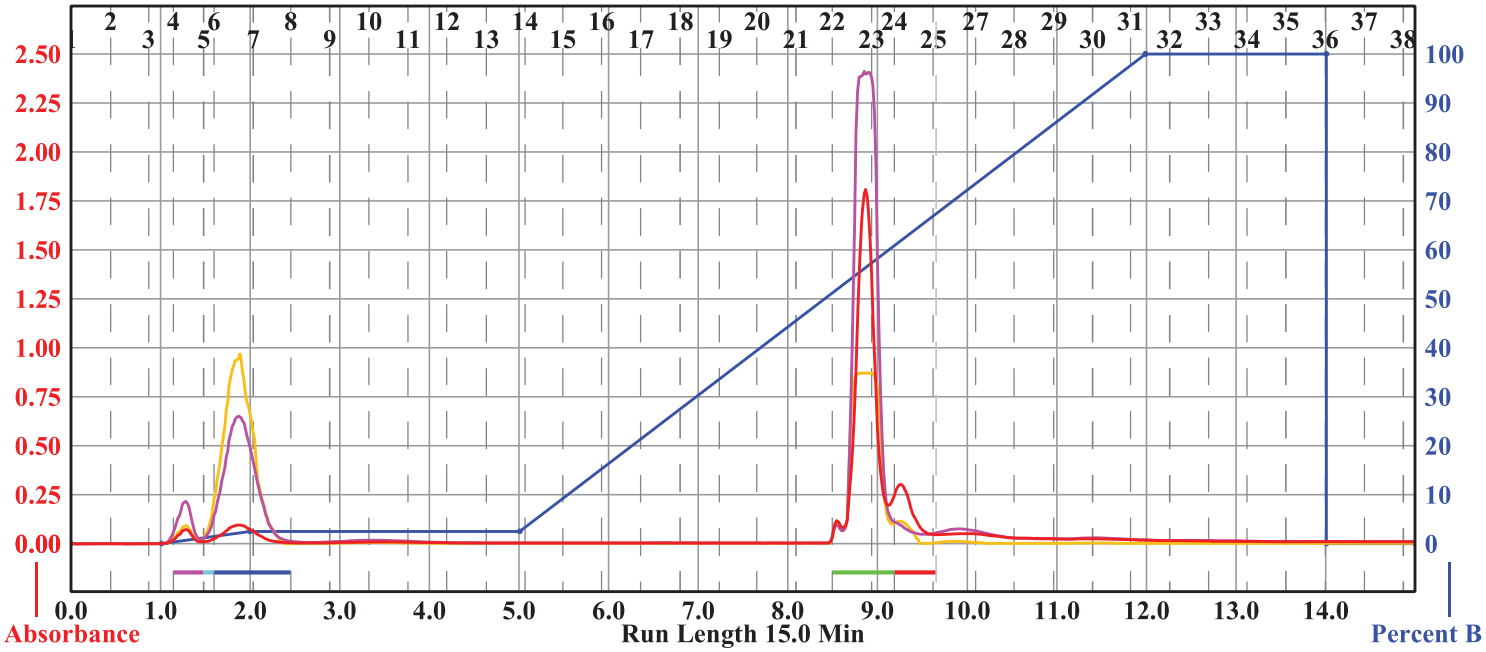
Sample: RPD-FGF23-A-127

4x Column: 2
RediSep Column: Silica 12g
SN: E04150644E16E6 Lot: 2621319040Y
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:4	A:4
2	A:5	A:5
3	A:6	A:7
4	A:22	A:23
5	A:24	A:25

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
0.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S164

Compound 6k

Rf 200

Tuesday 21 March 2017 10:26AM

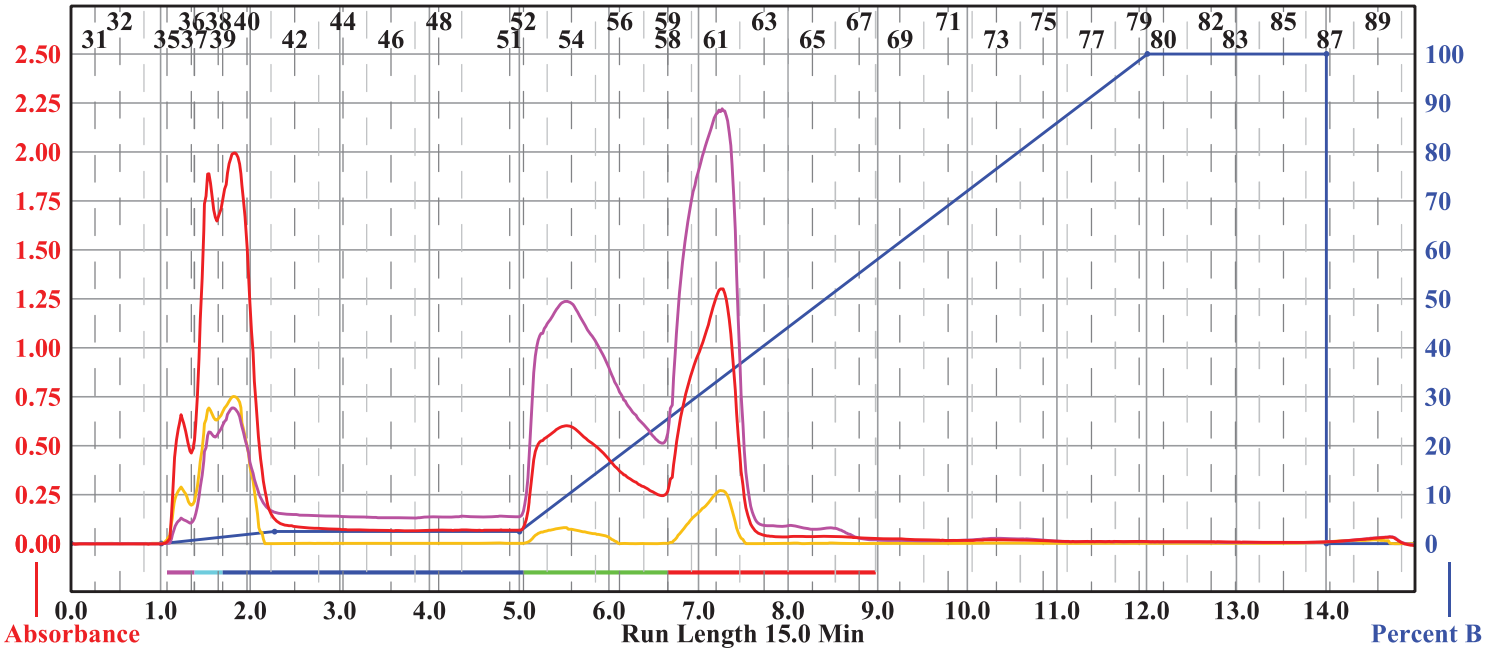
Sample: RPD-FGF23-A-81

4x Column: 2
RediSep Column: Silica 12g
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 3.2 min
Solvent: A1 hexane
Solvent: B2 methanol

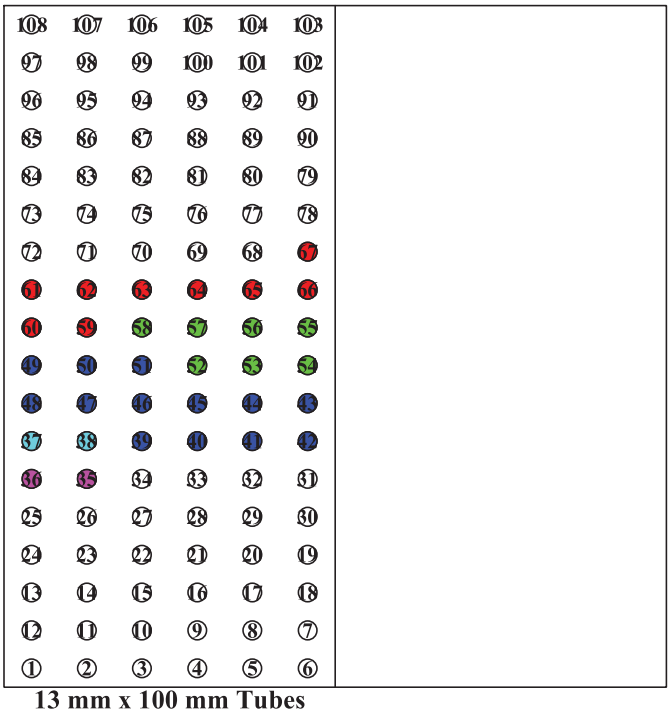
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A



13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:35	A:36
2	A:37	A:38
3	A:39	A:51
4	A:52	A:58
5	A:59	A:67

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.3	2.5	A1 hexane	B2 methanol
2.7	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S165

Compound 6l | Rf 200 | Wednesday 22 March 2017 02:16PM

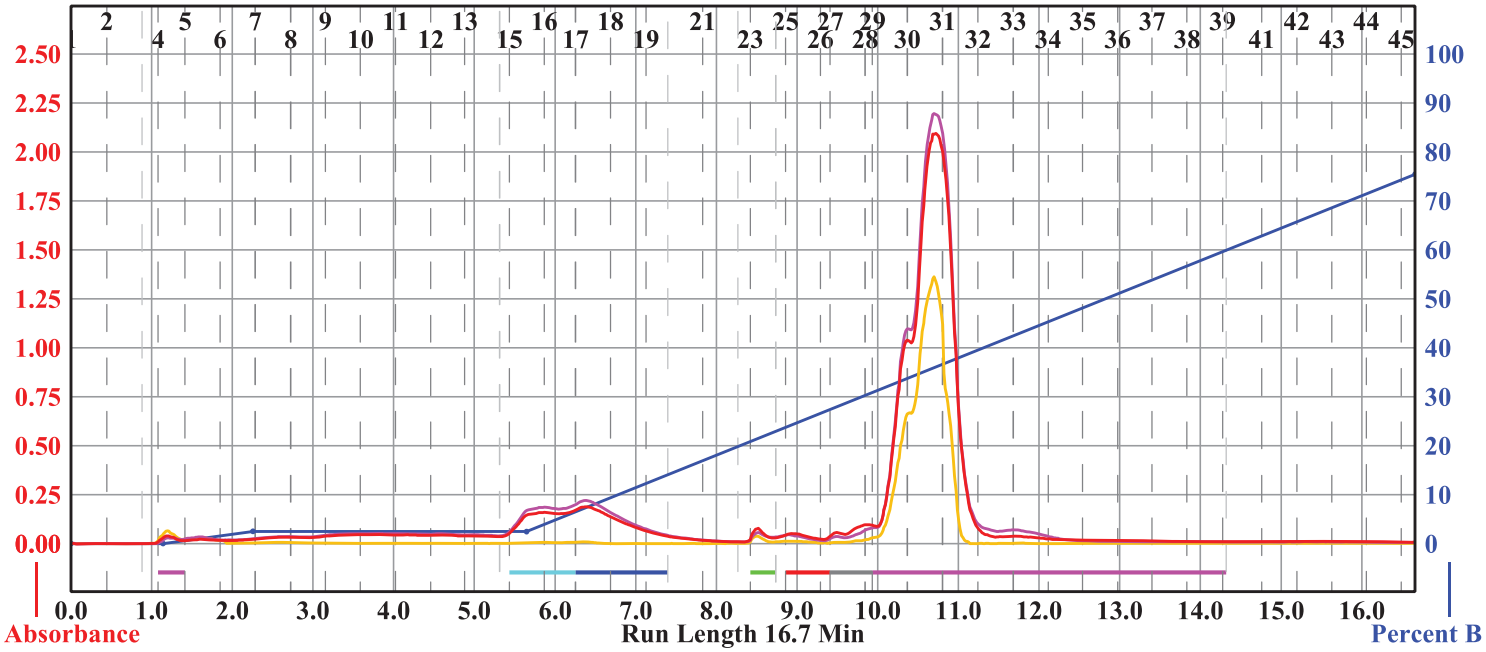
Sample: RPD-FGF23-A-97

4x Column: 1
RediSep Column: Silica 12g
SN: E0415064EFECC4 Lot: 2621319040Y
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

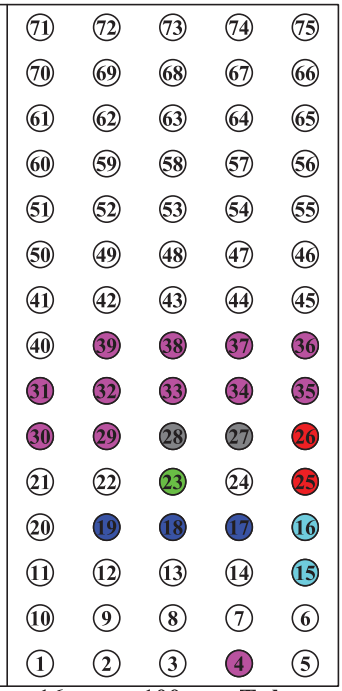
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:4	A:4
2	A:15	A:16
3	A:17	A:19
4	A:23	A:23
5	A:25	A:26
6	A:27	A:28
7	A:29	A:39

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.1	0.0	A1 hexane	B2 methanol
1.1	2.5	A1 hexane	B2 methanol
3.4	2.5	A1 hexane	B2 methanol
11.0	75.4	A1 hexane	B2 methanol

S166

Compound 6m | Rf 200 | Tuesday 21 March 2017 02:44PM

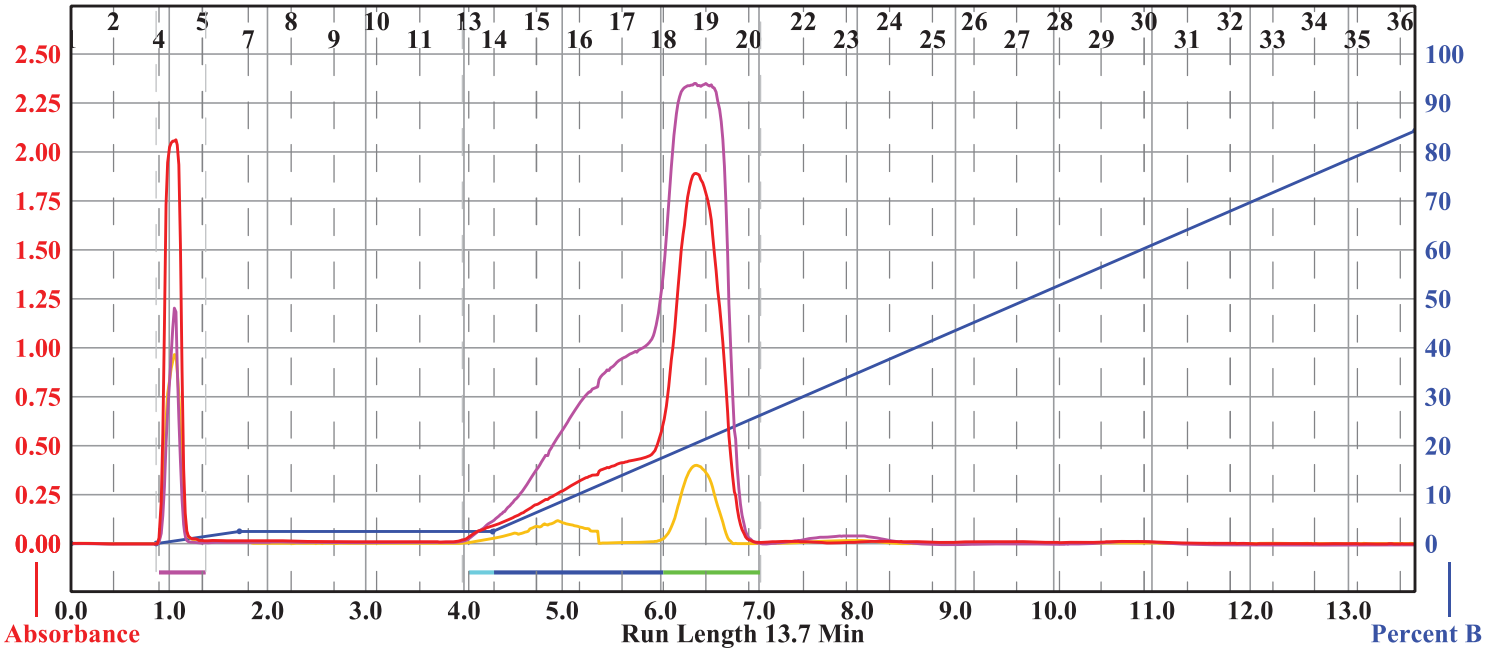
Sample: RPD-FGF23-A-89

4x Column: 1
RediSep Column: Silica 12g
SN: E04150644E1DB3 Lot: 2621319040Y
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

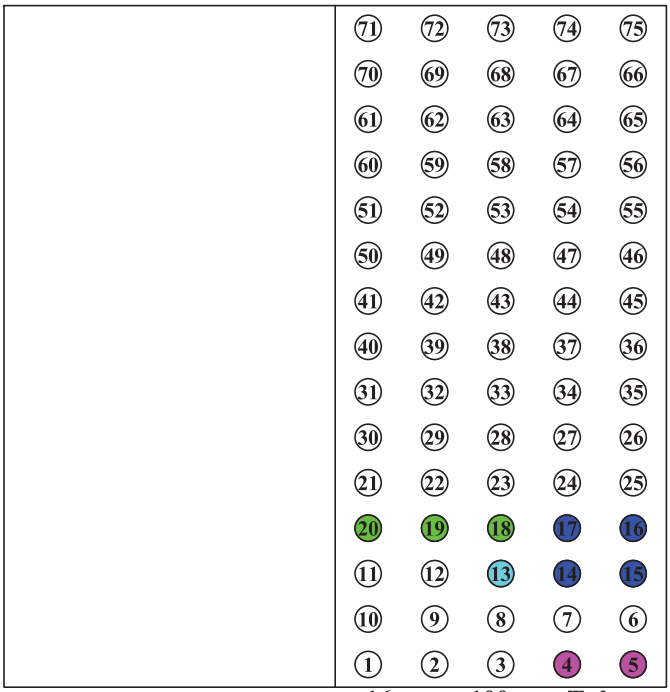
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:4	A:5
2	A:13	A:13
3	A:14	A:17
4	A:18	A:20

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
0.9	0.0	A1 hexane	B2 methanol
0.8	2.5	A1 hexane	B2 methanol
2.6	2.5	A1 hexane	B2 methanol
9.4	84.3	A1 hexane	B2 methanol

S167

Compound 6n | Rf 200 | Monday 20 March 2017 02:57PM

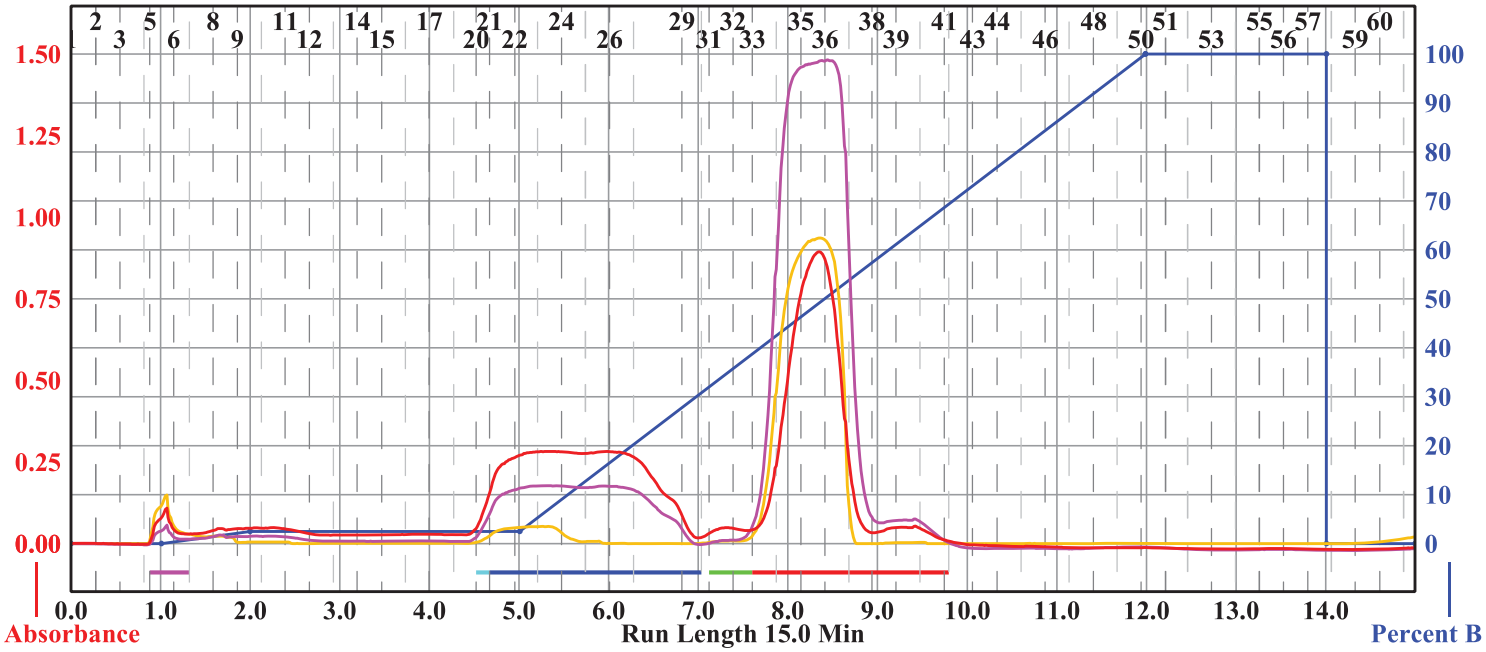
Sample: RPD-FGF23-A-77

4x Column: 2
RediSep Column: Silica 12g
SN: E04150644E2281 Lot: 2621319040Y
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:5	A:6
2	A:20	A:20
3	A:21	A:29
4	A:31	A:32
5	A:33	A:41

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
0.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S168

Compound 6o

Rf 200

Tuesday 21 March 2017 09:19AM

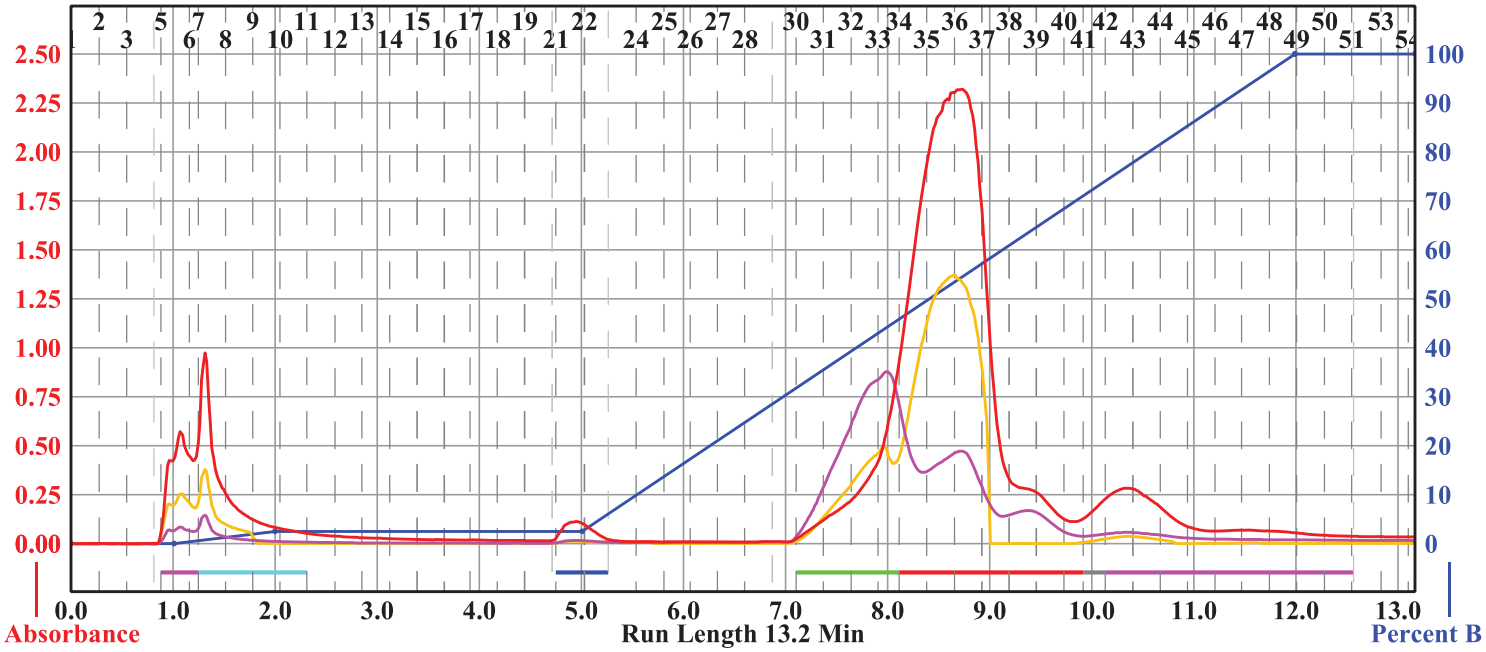
Sample: RPD-FGF23-A-79

4x Column: 2
RediSep Column: Silica 12g
SN: E04150644E23B4 Lot: 2621319040Y
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:5	A:6
2	A:7	A:10
3	A:21	A:22
4	A:30	A:33
5	A:34	A:40
6	A:41	A:41
7	A:42	A:51

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
0.0	100.0	A1 hexane	B2 methanol
1.2	100.0	A1 hexane	B2 methanol

S169

Compound 6p

Rf 200

Wednesday 22 March 2017 02:57PM

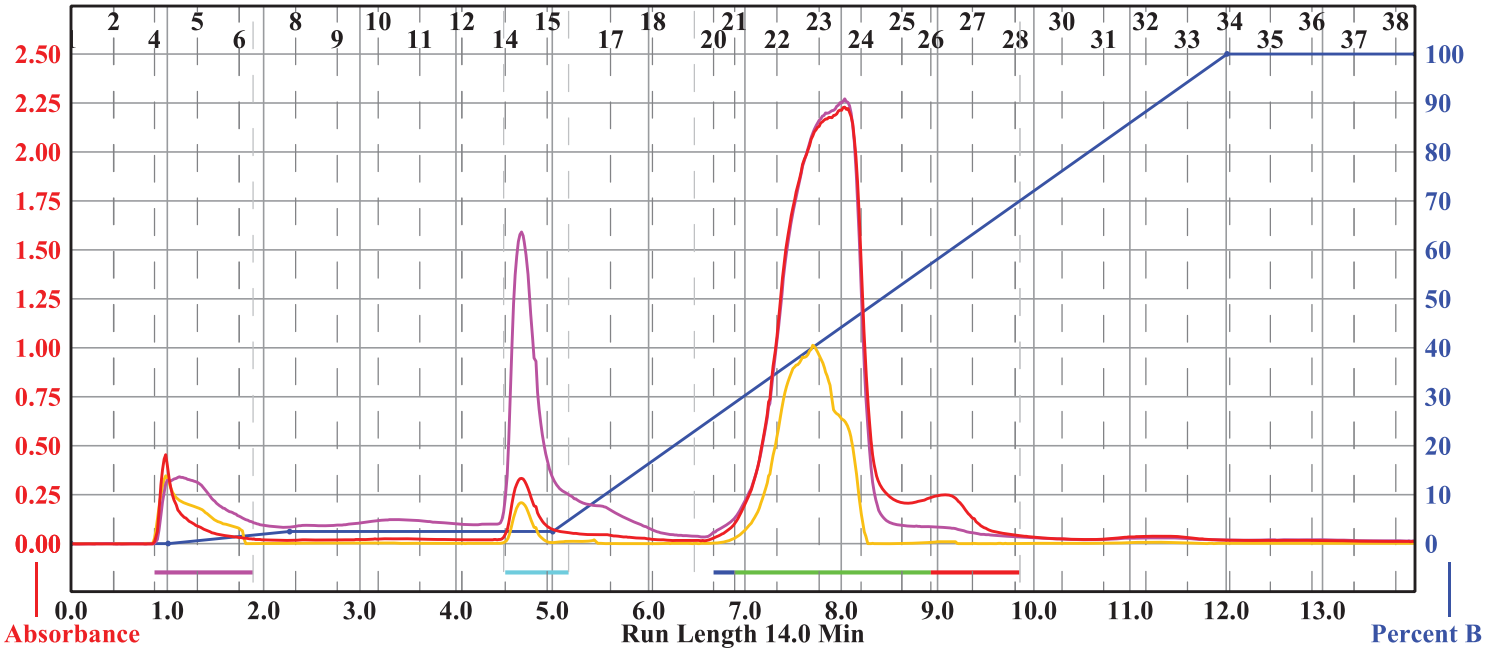
Sample: RPD-FGF23-A-83

4x Column: 2
RediSep Column: Silica 12g
SN: E04150644E1C81 Lot: 2621319040Y
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 3.2 min
Solvent: A1 hexane
Solvent: B2 methanol

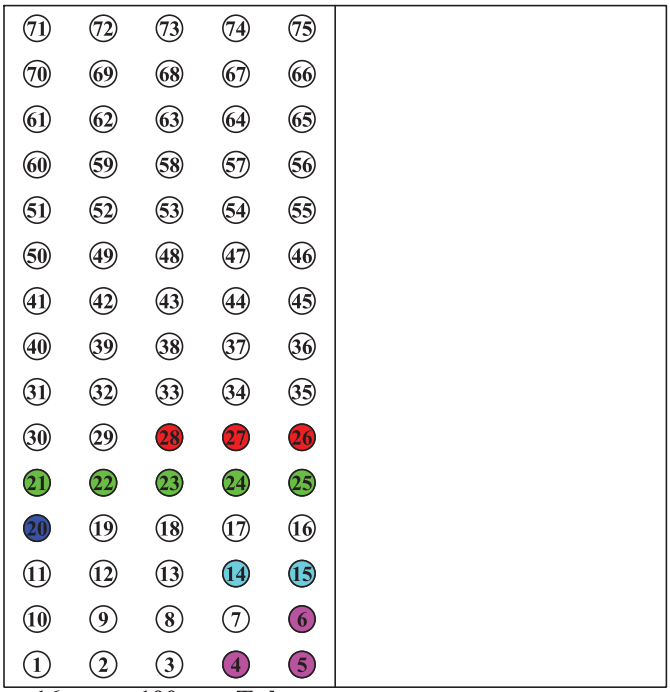
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:4	A:6
2	A:14	A:15
3	A:20	A:20
4	A:21	A:25
5	A:26	A:28

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.3	2.5	A1 hexane	B2 methanol
2.7	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol

S170

Compound 7a | Rf 200 | Wednesday 15 March 2017 01:54PM

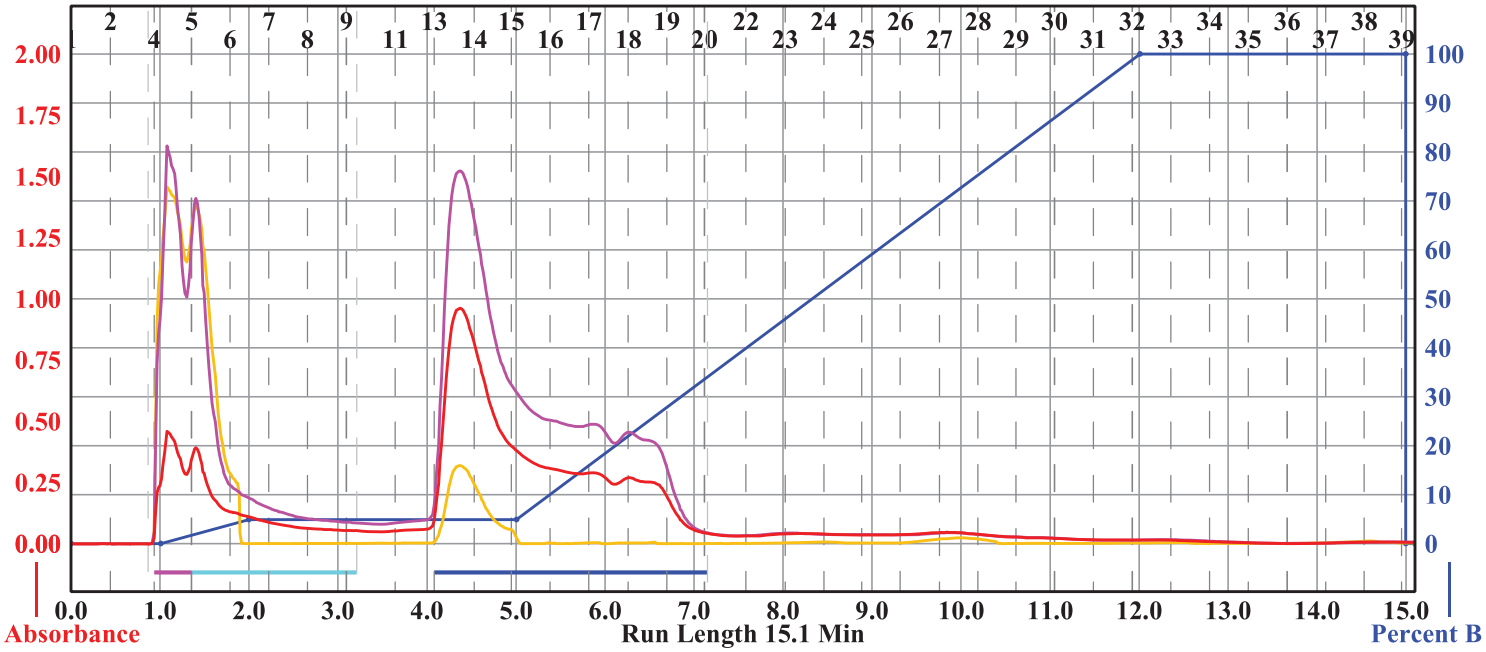
Sample: RPD-FGF23-A-69

4x Column: 2
RediSep Column: Silica 12g
SN: E0415064EFE44C Lot: 2621319040Y
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

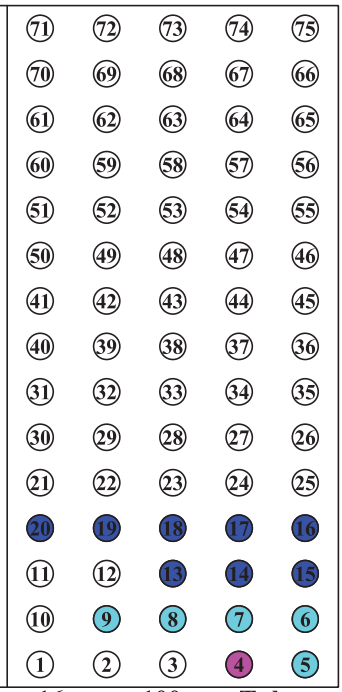
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:4	A:4
2	A:5	A:9
3	A:13	A:20

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
3.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
0.1	0.0	A1 hexane	B2 methanol

S171

Compound 7b | Rf 200 | Thursday 16 March 2017 09:30AM

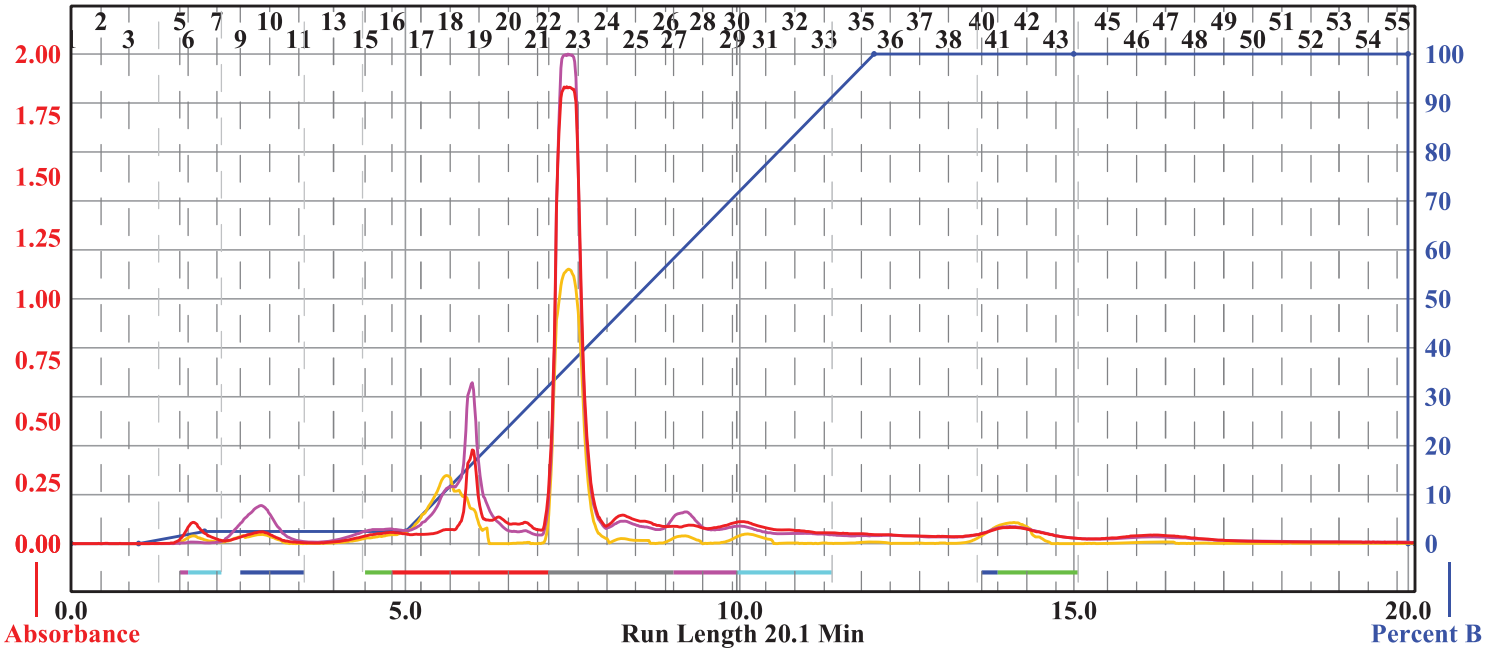
Sample: RPD-FGF23-A-71

4x Column: 2
RediSep Column: Silica 12g
SN: E04150644E16DE Lot: 2621319040Y
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:5	A:5
2	A:6	A:7
3	A:9	A:11
4	A:15	A:15
5	A:16	A:21
6	A:22	A:26
7	A:27	A:29
8	A:30	A:33
9	A:40	A:40
10	A:41	A:43

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
3.0	100.0	A1 hexane	B2 methanol
5.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
0.1	0.0	A1 hexane	B2 methanol

S172

Compound 8b

Rf 200

Friday 13 January 2017 02:00PM

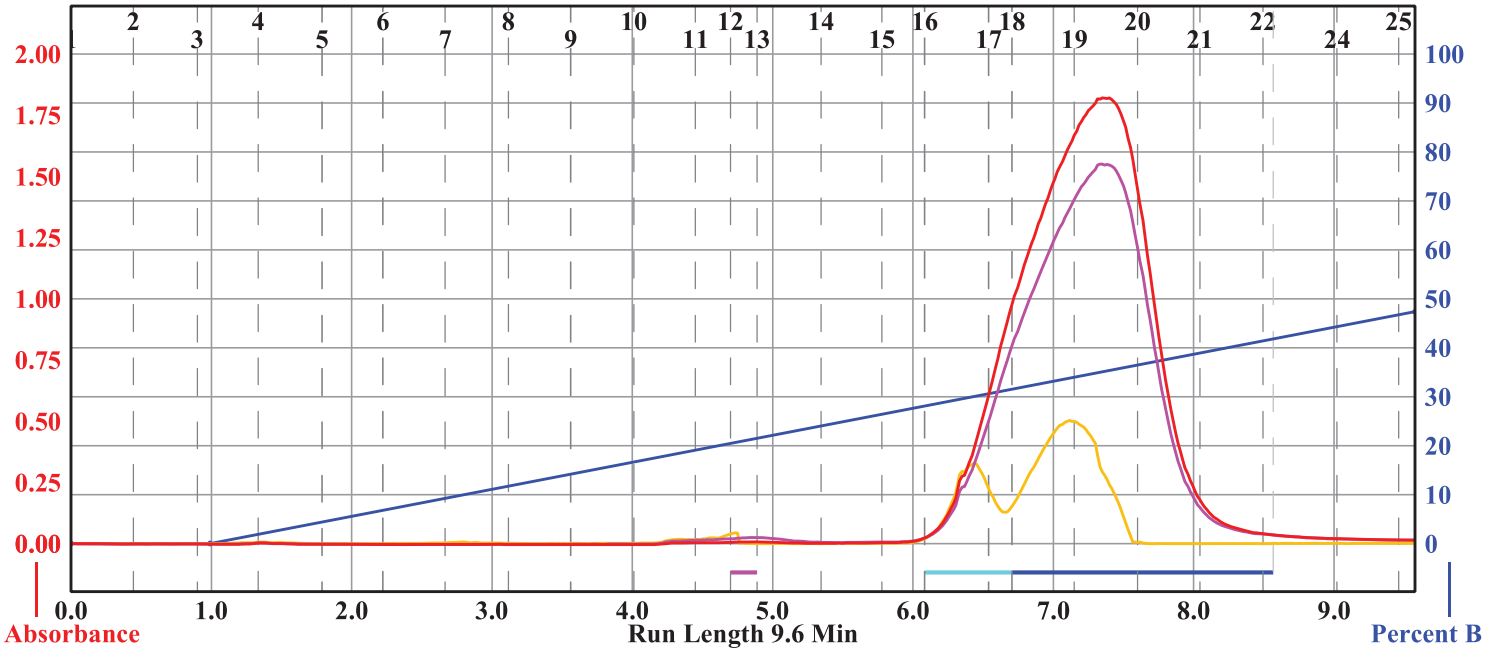
Sample: JDC-FGF23-A-21-2

4x Column: 3
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:12	A:12
2	A:16	A:17
3	A:18	A:22

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
8.6	47.4	A1 hexane	B2 methanol

S173

Compound 8e | Rf 200 | Monday 27 March 2017 03:27PM

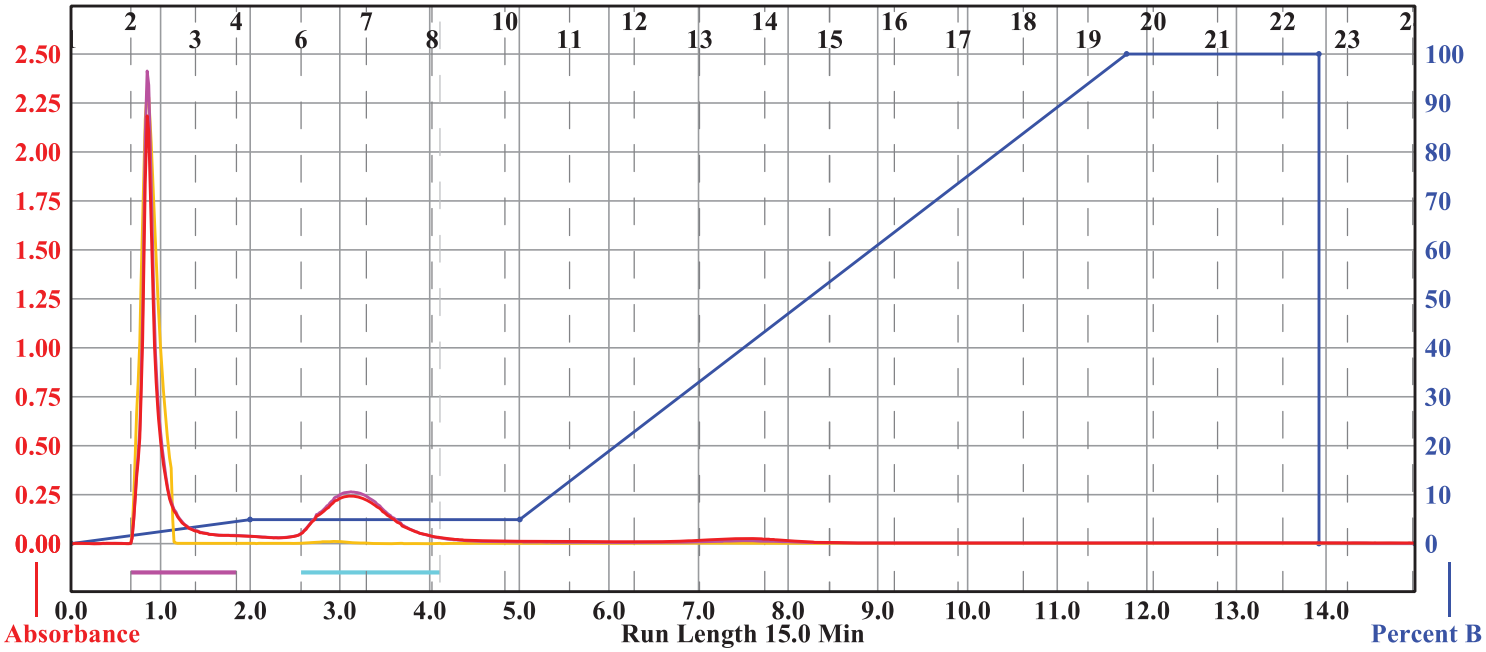
Sample: JWC-FGF23-A-59

4x Column: 2
RediSep Column: Al2O3 pH=7 8g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:2	A:3
2	A:6	A:8

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
2.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
6.8	100.0	A1 hexane	B2 methanol
2.1	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.1	0.0	A1 hexane	B2 methanol

S174

Compound 8f | Rf 200 | Wednesday 11 January 2017 02:19PM

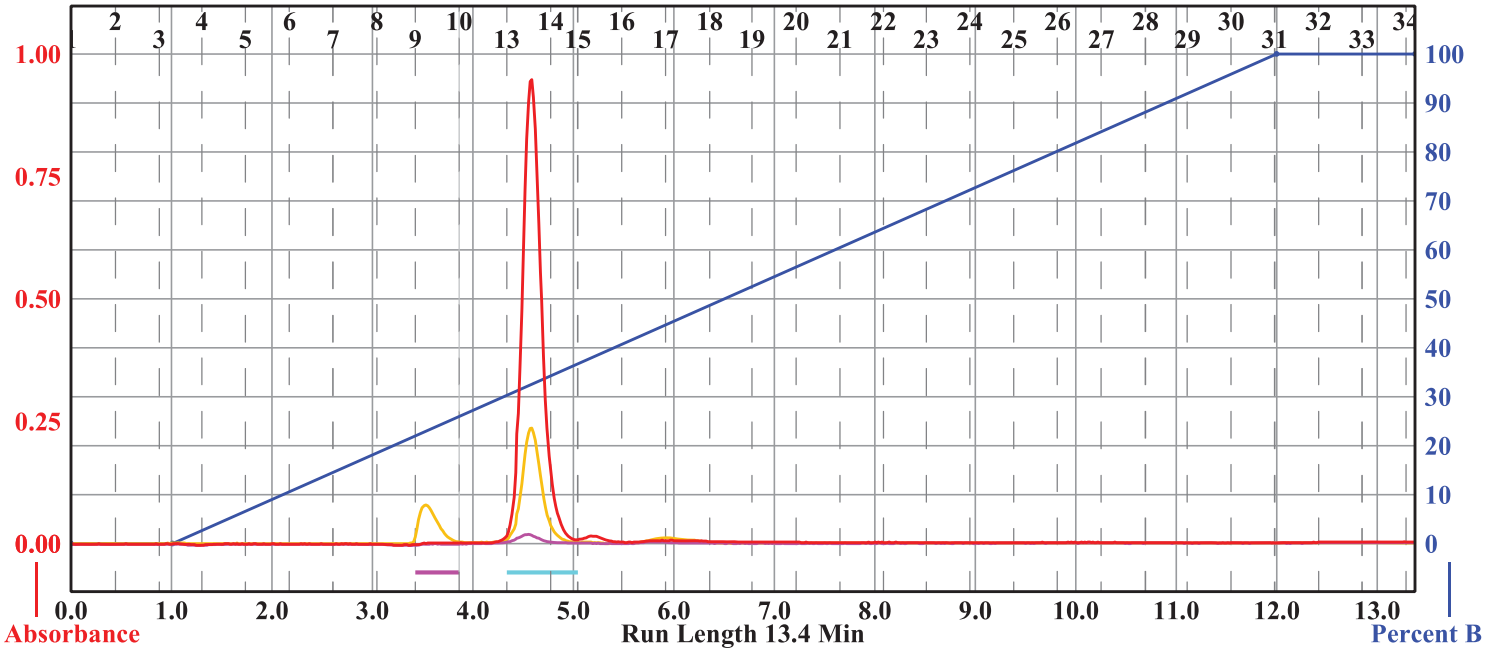
Sample: RPD-FGF23-A-31

4x Column: 1
RediSep Column: Silica 12g Gold
SN: E04150644DB5EF Lot: 2622248050W
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: 13 ml
Non-Peak Tube Volume: 13 ml
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:9	A:10
2	A:13	A:14

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
11.0	100.0	A1 hexane	B2 methanol
1.4	100.0	A1 hexane	B2 methanol

S175

Compound 8h | Rf 200 | Friday 13 January 2017 10:50AM

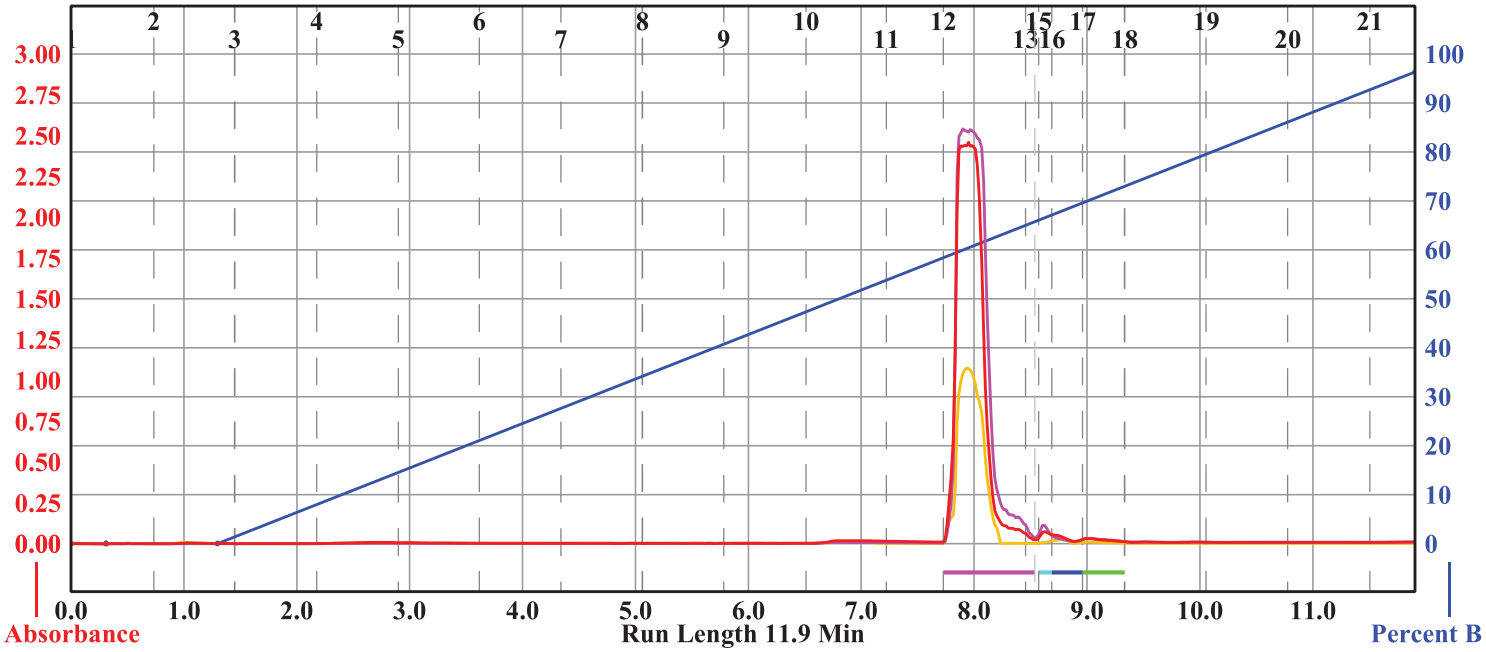
Sample: JWC-FGF23-A-43

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

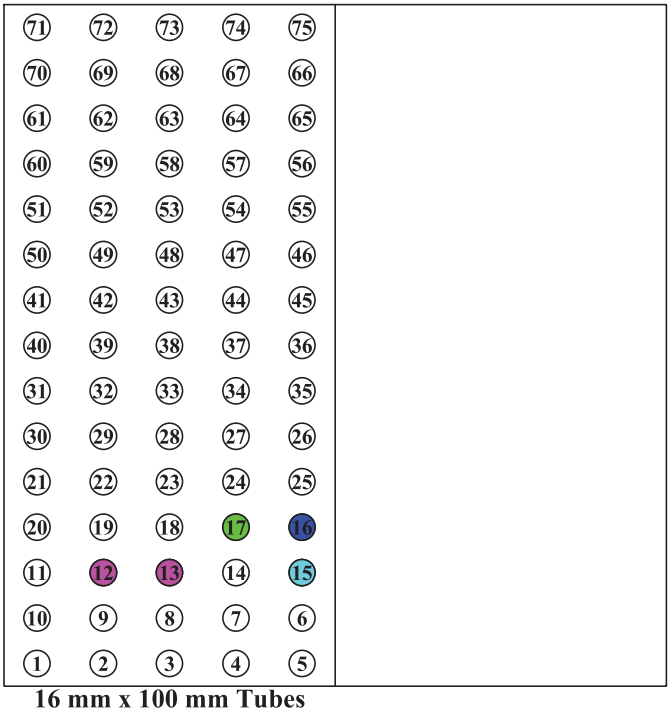
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:12	A:13
2	A:15	A:15
3	A:16	A:16
4	A:17	A:17

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
0.3	0.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
10.6	96.3	A1 hexane	B2 methanol

S176

Compound 8i | Rf 200 | Friday 13 January 2017 11:58AM

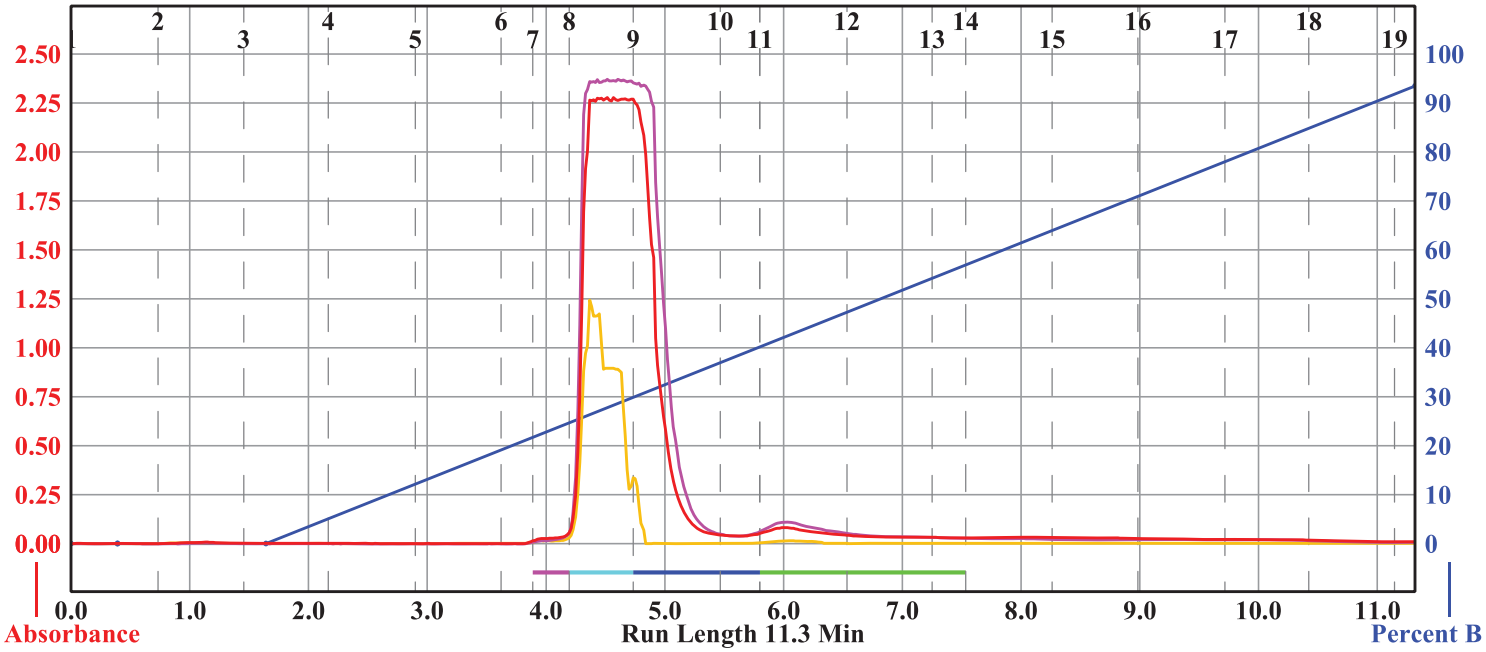
Sample: RPD-FGF23-A-47

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:7	A:7
2	A:8	A:8
3	A:9	A:10
4	A:11	A:13

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
0.4	0.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.2	0.0	A1 hexane	B2 methanol
9.7	93.5	A1 hexane	B2 methanol

S177

Compound 8j | Rf 200 | Tuesday 17 January 2017 09:39AM

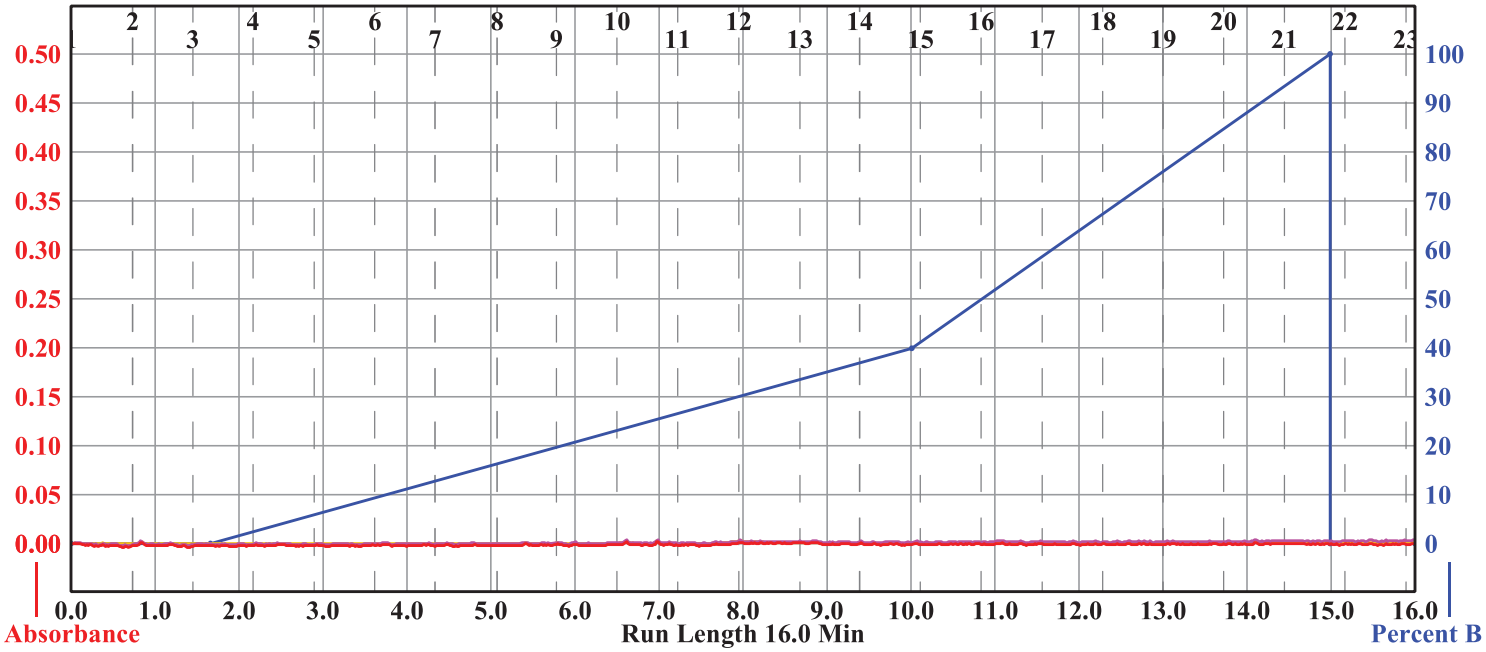
Sample: JWC-FGF23-A-39-2

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.7	0.0	A1 hexane	B2 methanol
8.3	39.9	A1 hexane	B2 methanol
5.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S178

Compound 8k | Rf 200 | Thursday 12 January 2017 09:29AM

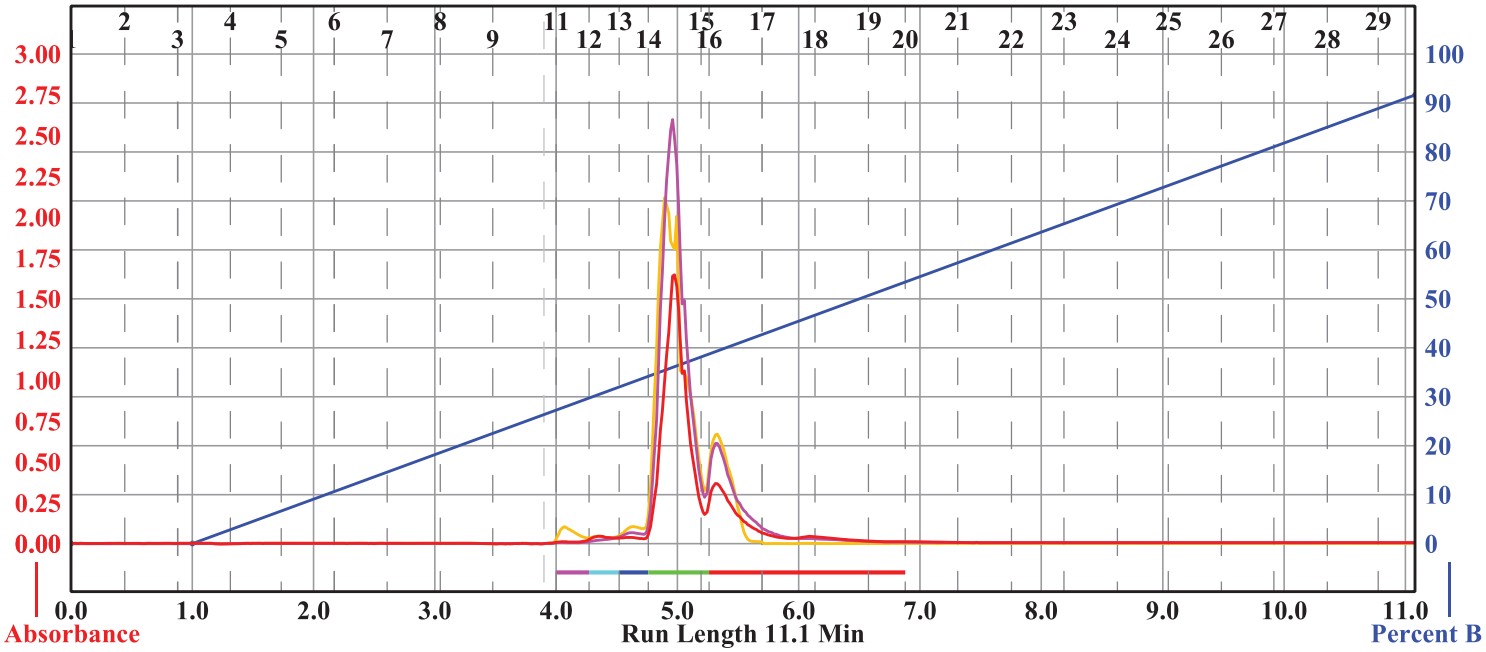
Sample: JWC-FGF23-A-41

4x Column: 2
RediSep Column: Silica 12g Gold
SN: E04150644DD628 Lot: 2622248050W
Flow Rate: 30 ml/min
Equilibration Volume: 100.8 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

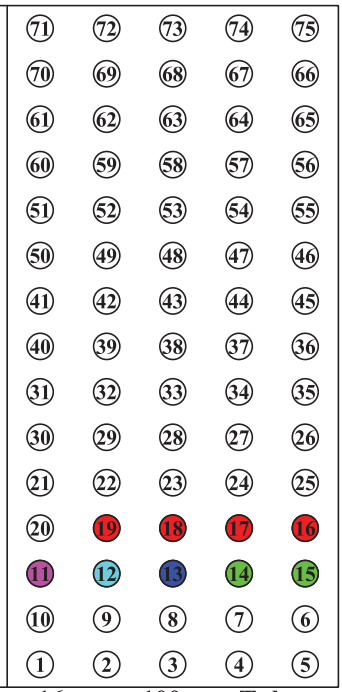
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 1 min
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 1 min
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:11	A:11
2	A:12	A:12
3	A:13	A:13
4	A:14	A:15
5	A:16	A:19

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
10.1	91.6	A1 hexane	B2 methanol

S179

Compound 8l

Rf 200

Wednesday 11 January 2017 09:34AM

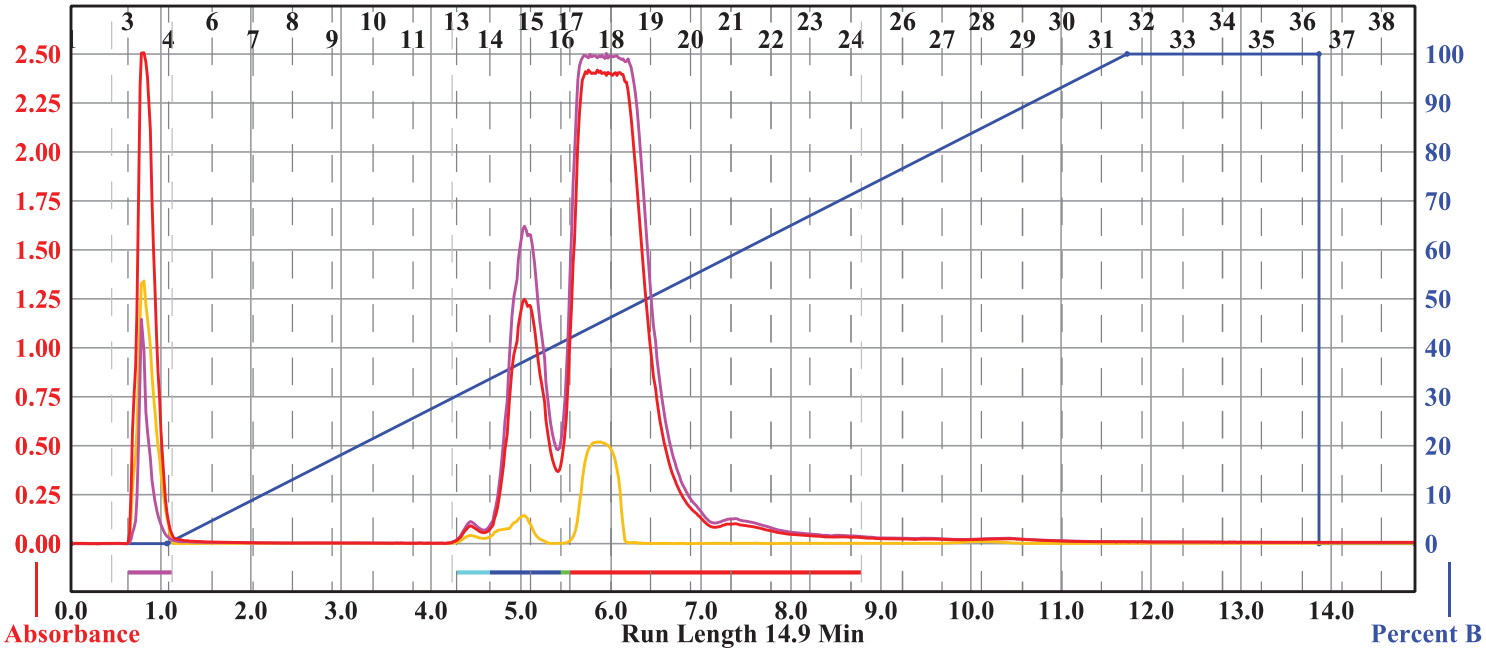
Sample: JWC-FGF23-A-33

4x Column: 2
RediSep Column: Al2O3 pH=7 8g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:4
2	A:13	A:13
3	A:14	A:15
4	A:16	A:16
5	A:17	A:24

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.1	0.0	A1 hexane	B2 methanol
10.7	100.0	A1 hexane	B2 methanol
2.1	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.1	0.0	A1 hexane	B2 methanol

S180

Compound 8 m | Rf 200 | Tuesday 28 March 2017 10:15AM

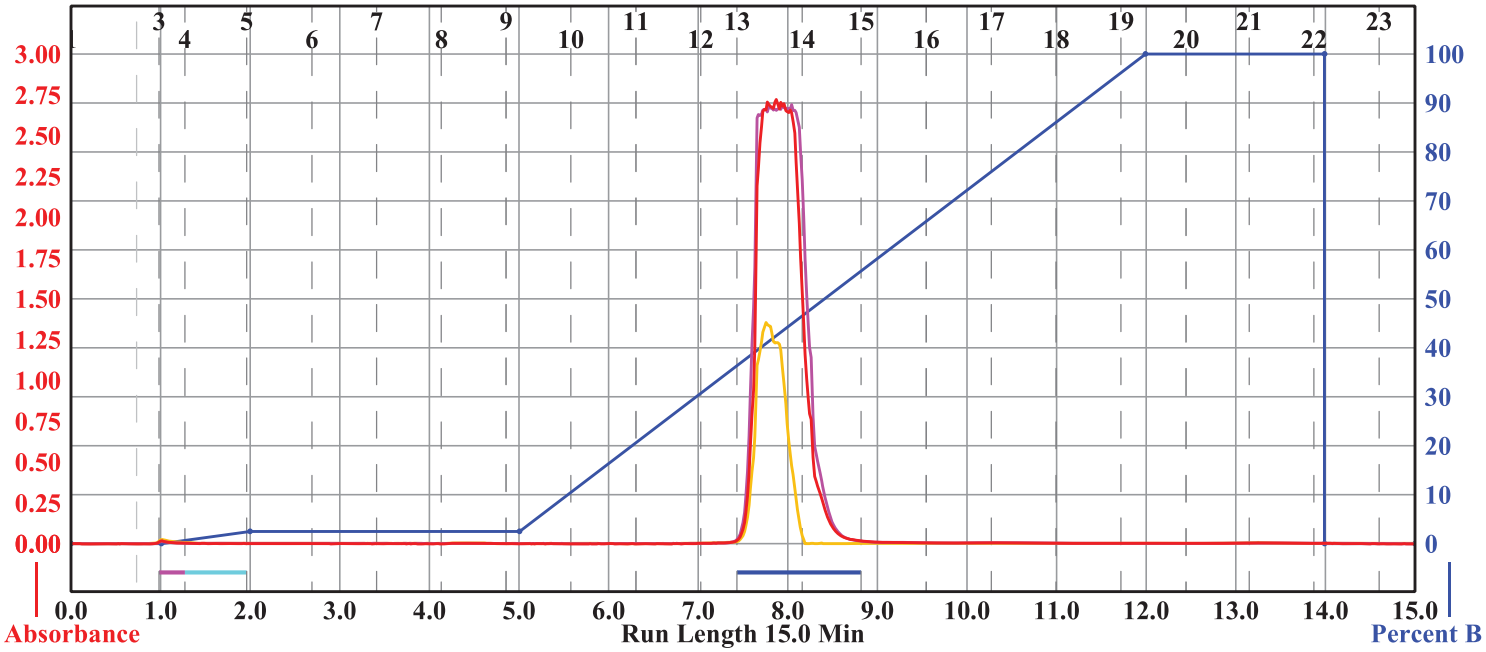
Sample: RPD-FGF23-A-103

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 2.8 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:3
2	A:4	A:4
3	A:13	A:14

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S181

Compound 8n | Rf 200 | Thursday 23 March 2017 10:52AM

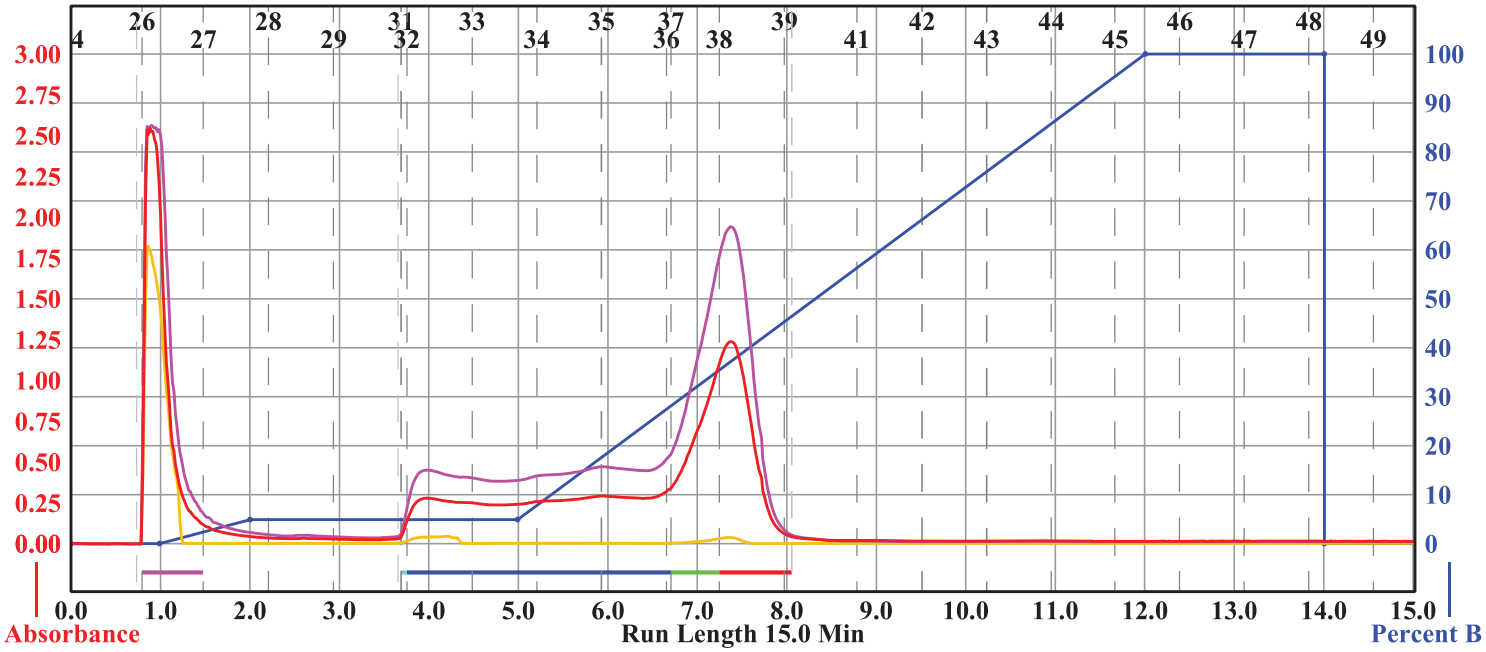
Sample: JWC-FGF23-A-55

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:26	A:26
2	A:31	A:31
3	A:32	A:36
4	A:37	A:37
5	A:38	A:39

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S182

Compound 8o | Rf 200 | Friday 24 March 2017 01:35PM

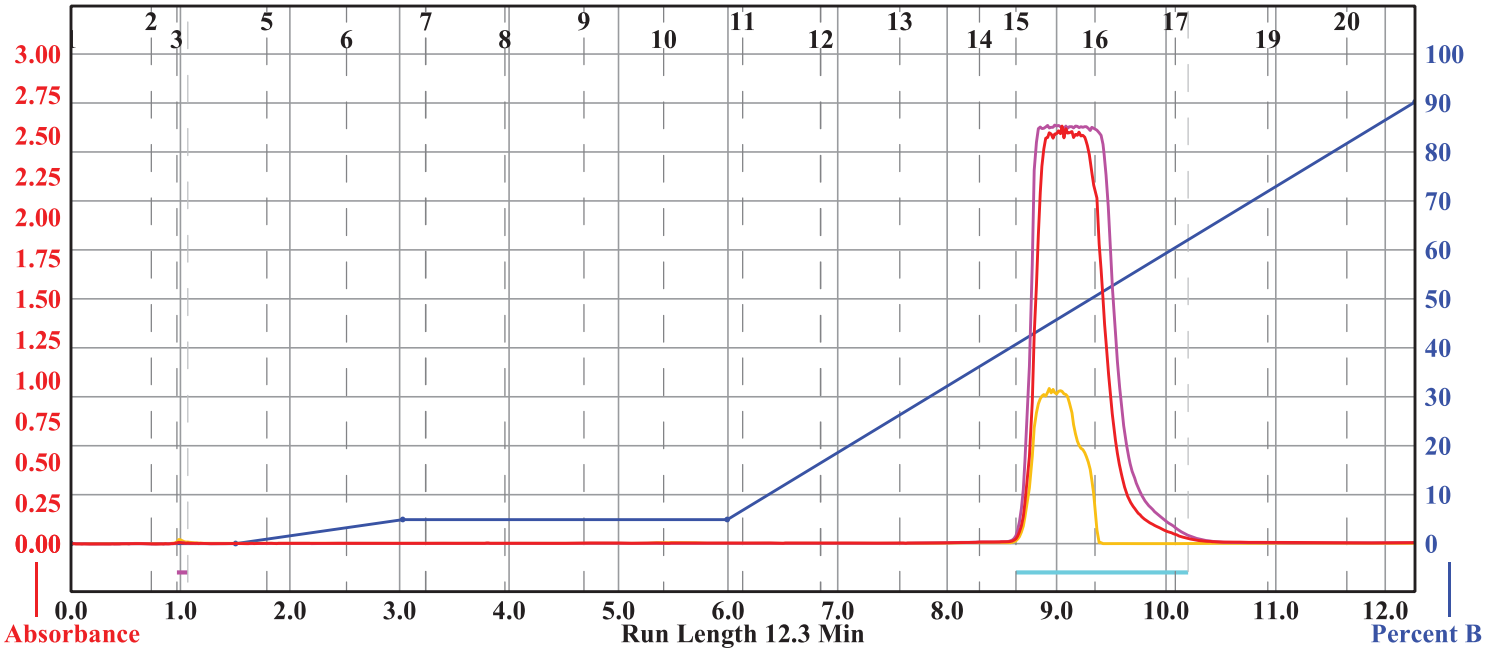
Sample: RPD-FGF23-A-99

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:3
2	A:15	A:17

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.5	0.0	A1 hexane	B2 methanol
1.5	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
6.3	90.2	A1 hexane	B2 methanol

S183

Compound 8p | Rf 200 | Monday 27 March 2017 03:02PM

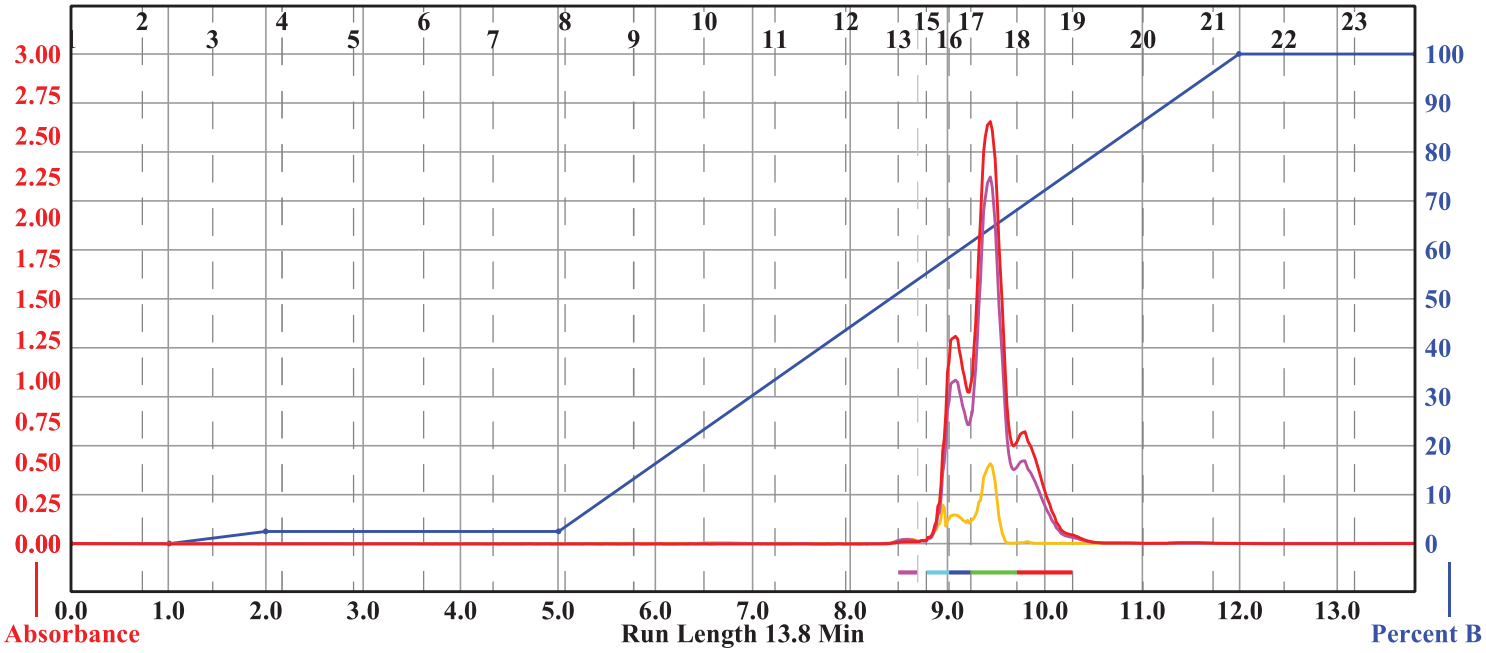
Sample: RPD-FGF23-A-101

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 7.6 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:13	A:13
2	A:15	A:15
3	A:16	A:16
4	A:17	A:17
5	A:18	A:18

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
1.8	100.0	A1 hexane	B2 methanol

S184

Compound 8q

Rf 200

Friday 13 January 2017 12:18PM

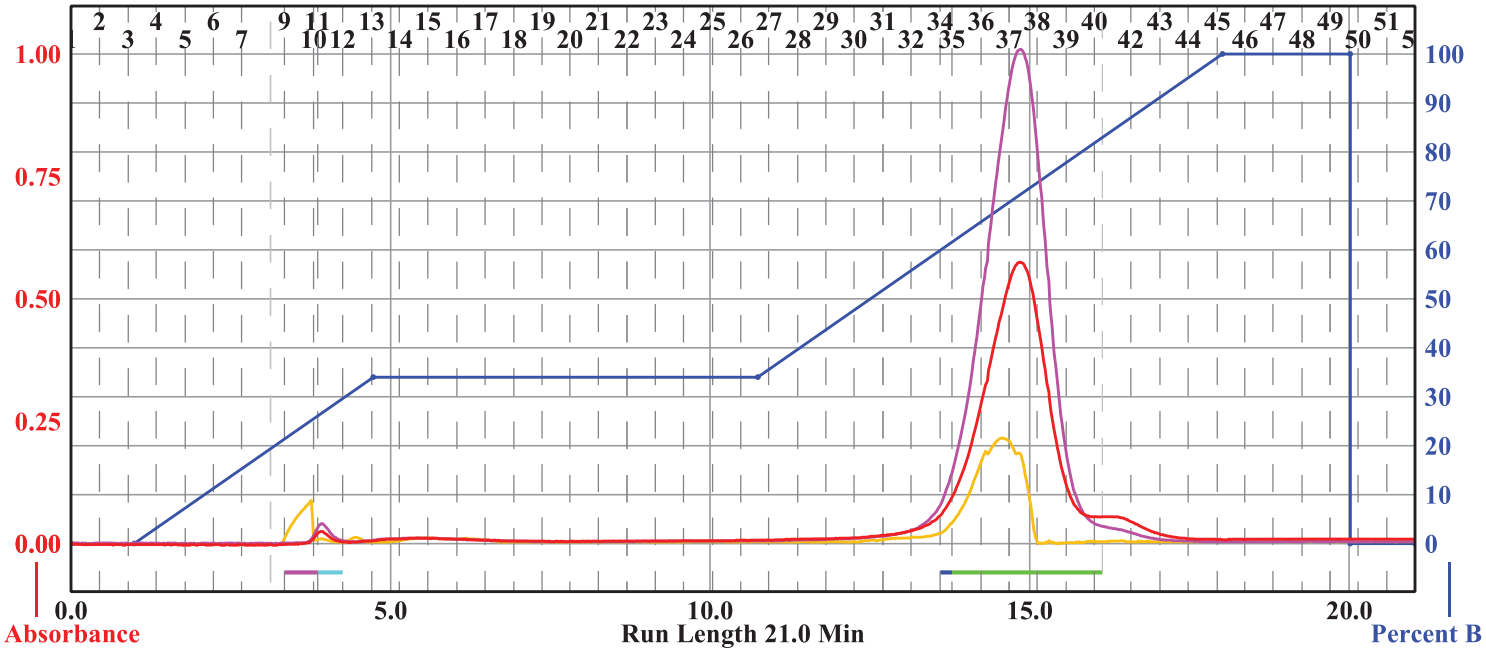
Sample: JWC-FGF23-45

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B1 ethyl acetate

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:9	A:10
2	A:11	A:11
3	A:34	A:34
4	A:35	A:40

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B1 ethyl acetate
1.0	0.0	A1 hexane	B1 ethyl acetate
3.7	34.0	A1 hexane	B1 ethyl acetate
6.0	34.0	A1 hexane	B1 ethyl acetate
7.3	100.0	A1 hexane	B1 ethyl acetate
2.0	100.0	A1 hexane	B1 ethyl acetate
0.0	0.0	A1 hexane	B1 ethyl acetate
1.0	0.0	A1 hexane	B1 ethyl acetate

S185

Compound 8r | Rf 200 | Tuesday 28 March 2017 10:38AM

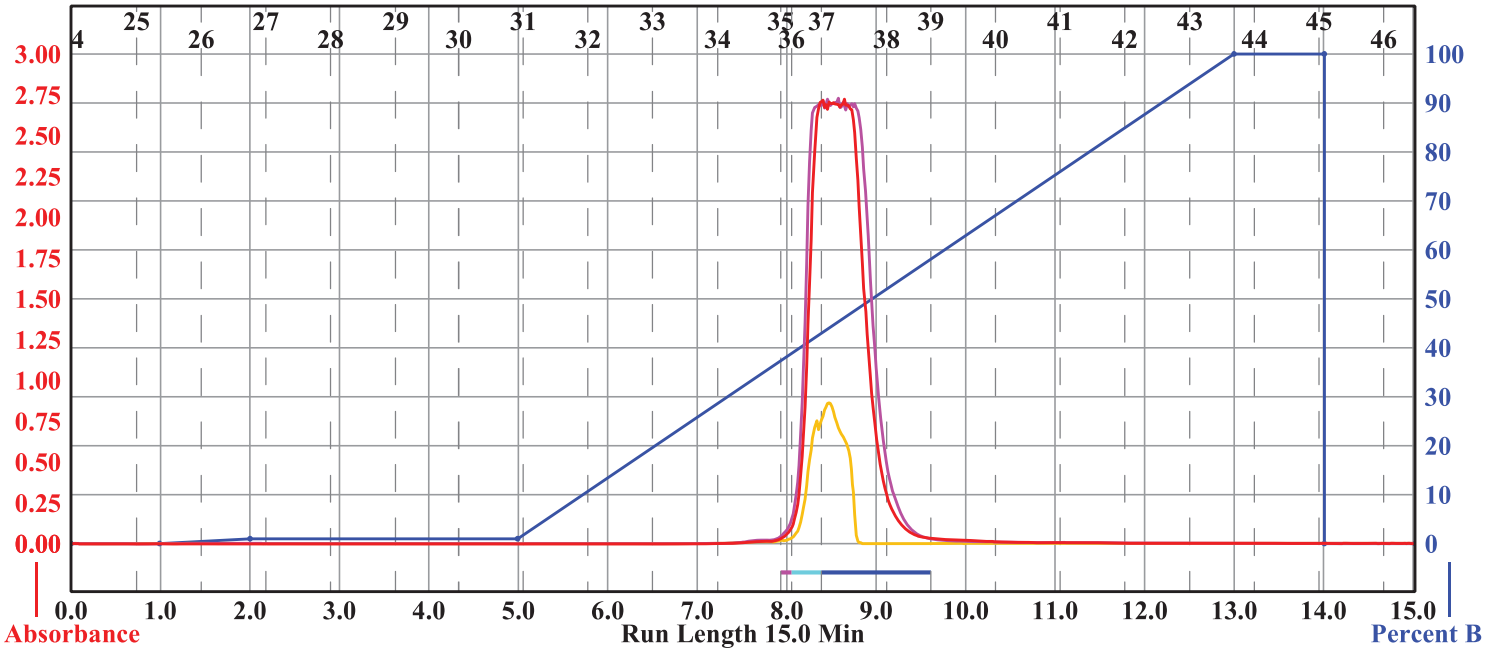
Sample: RPD-FGF23-A-105

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

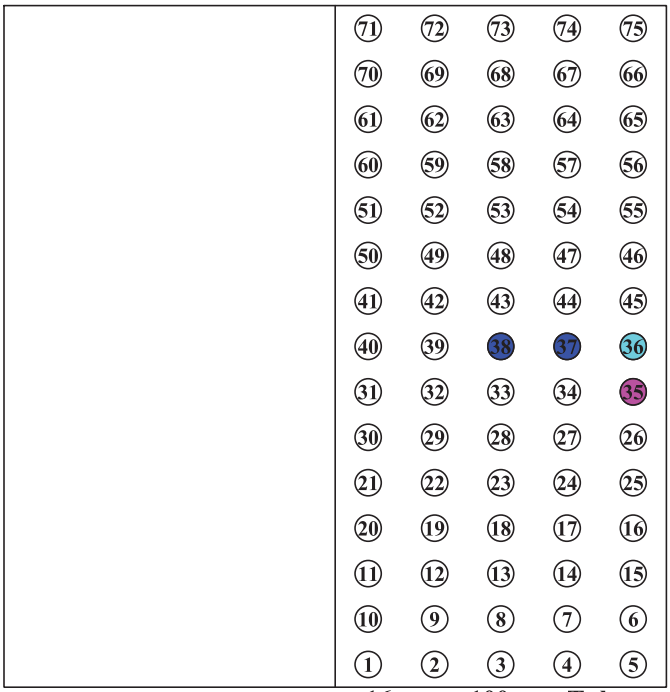
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:35	A:35
2	A:36	A:36
3	A:37	A:38

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	1.0	A1 hexane	B2 methanol
3.0	1.0	A1 hexane	B2 methanol
8.0	100.0	A1 hexane	B2 methanol
1.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S186

Compound 8s | Rf 200 | Thursday 23 March 2017 09:25AM

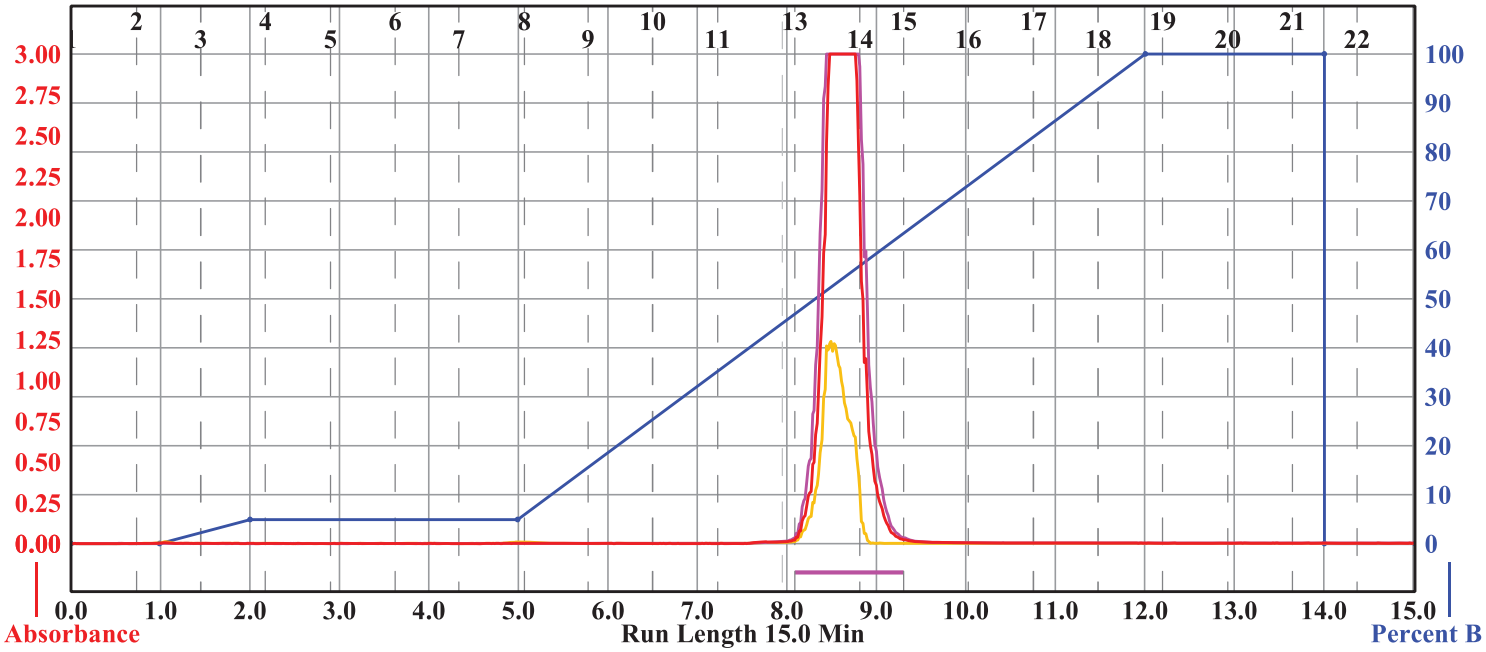
Sample: JWC-FGF23-A-57

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:13	A:14

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S187

Compound 8u | Rf 200 | Friday 13 January 2017 02:18PM

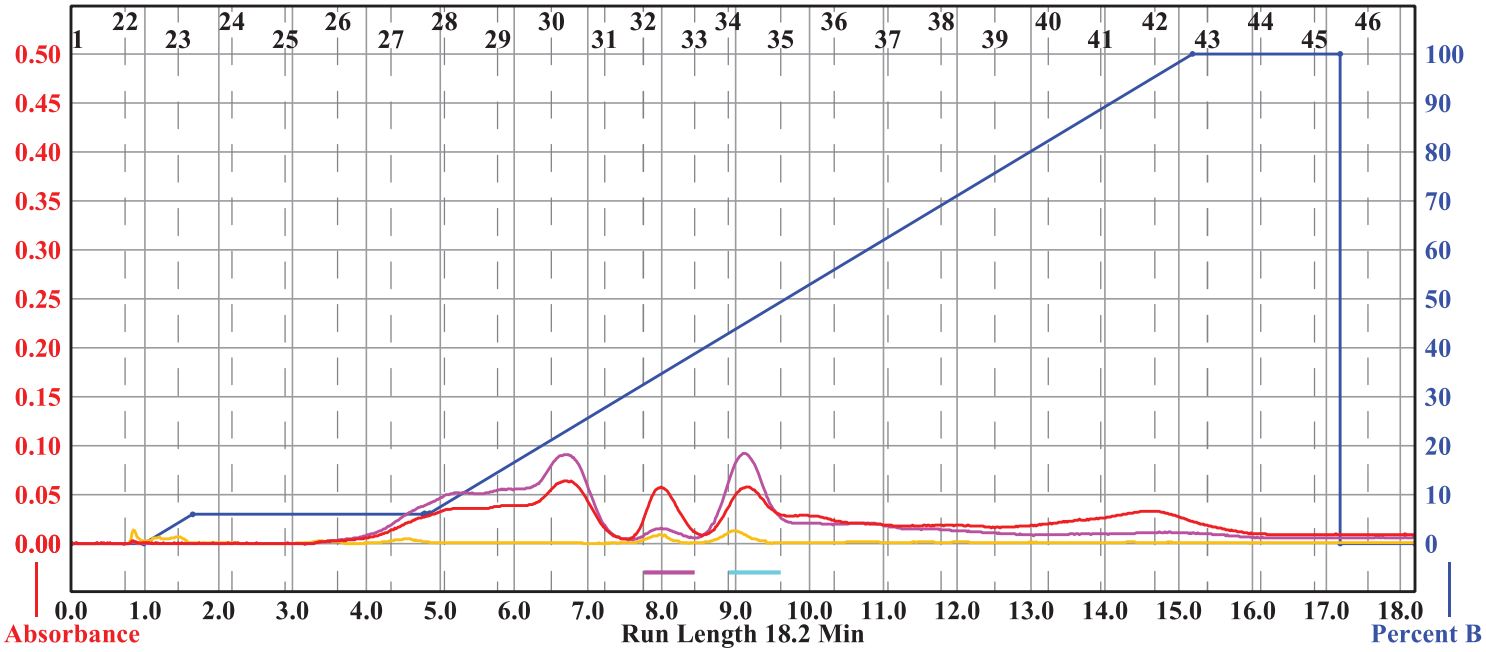
Sample: RPD-FGF23-A-37

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

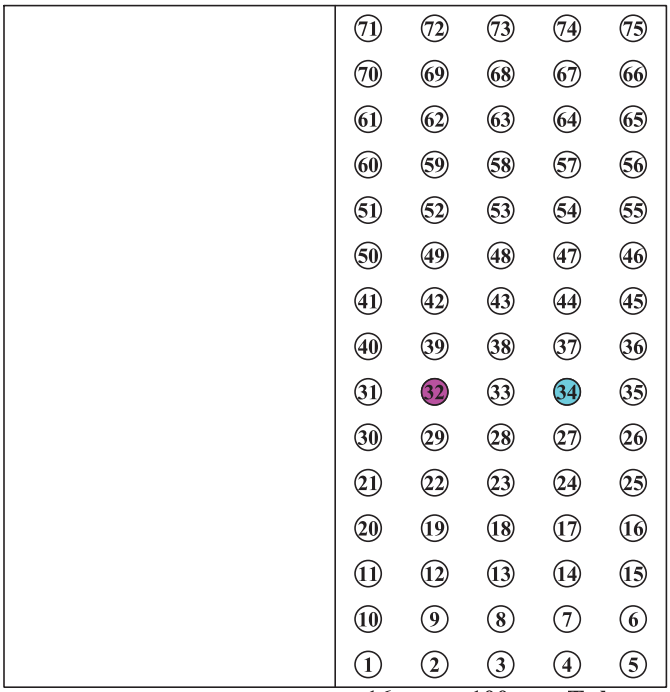
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:32	A:32
2	A:34	A:34

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
0.7	6.0	A1 hexane	B2 methanol
3.1	6.0	A1 hexane	B2 methanol
0.0	6.2	A1 hexane	B2 methanol
0.1	6.2	A1 hexane	B2 methanol
0.0	6.2	A1 hexane	B2 methanol
10.3	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol

S188

Compound 8v | Rf 200 | Thursday 23 March 2017 12:58PM

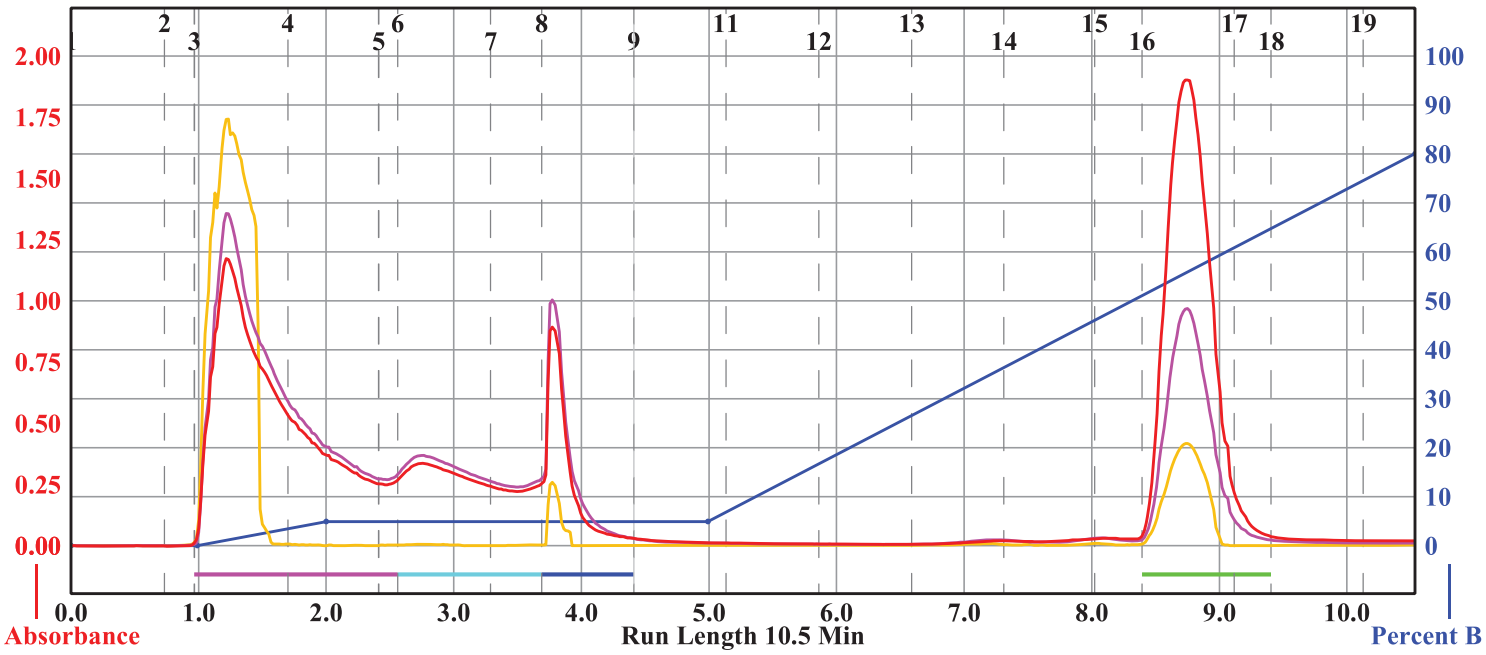
Sample: JWC-FGF23-A-53

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:5
2	A:6	A:7
3	A:8	A:9
4	A:16	A:17

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	4.9	A1 hexane	B2 methanol
3.0	4.9	A1 hexane	B2 methanol
5.5	80.0	A1 hexane	B2 methanol

S189

Compound 9a

Rf 200

Thursday 23 March 2017 12:36PM

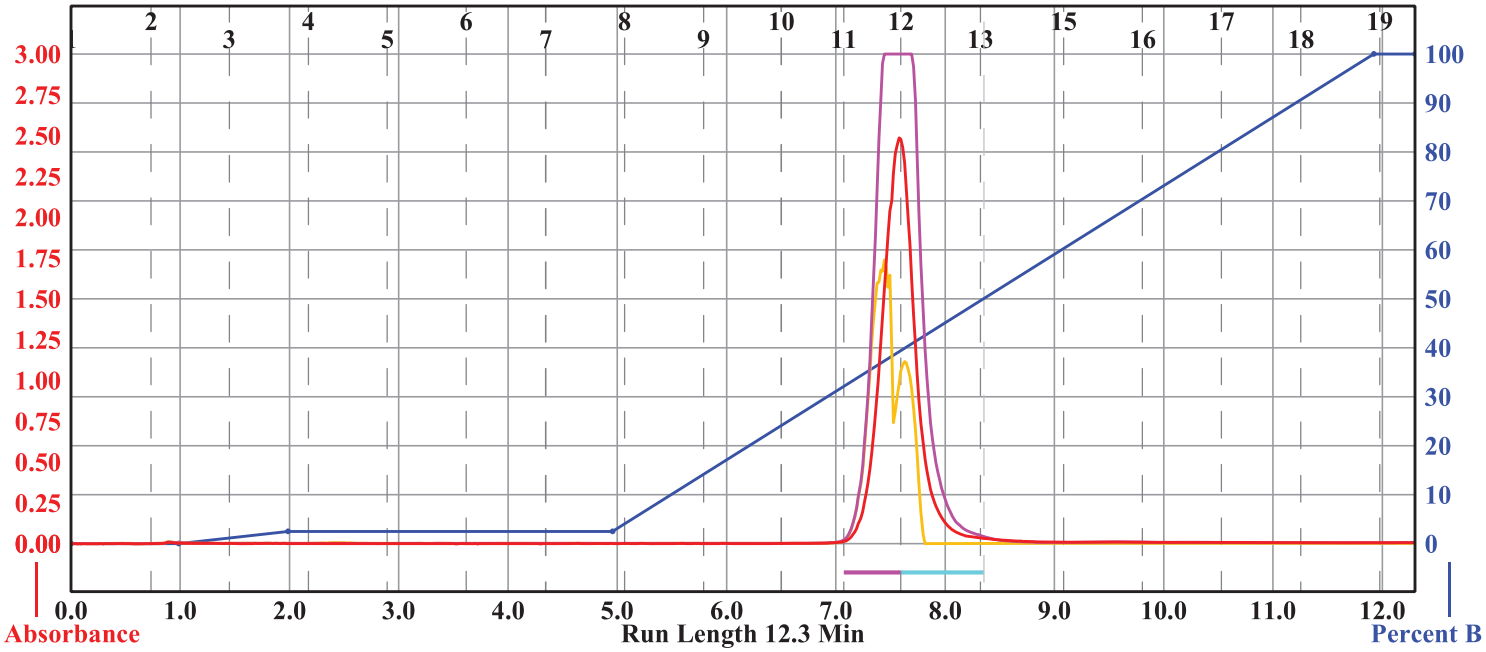
Sample: RPD-FGF23-A-91

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:11	A:11
2	A:12	A:13

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
0.4	100.0	A1 hexane	B2 methanol

S190

Compound 9b | Rf 200 | Thursday 15 March 2018 11:44AM

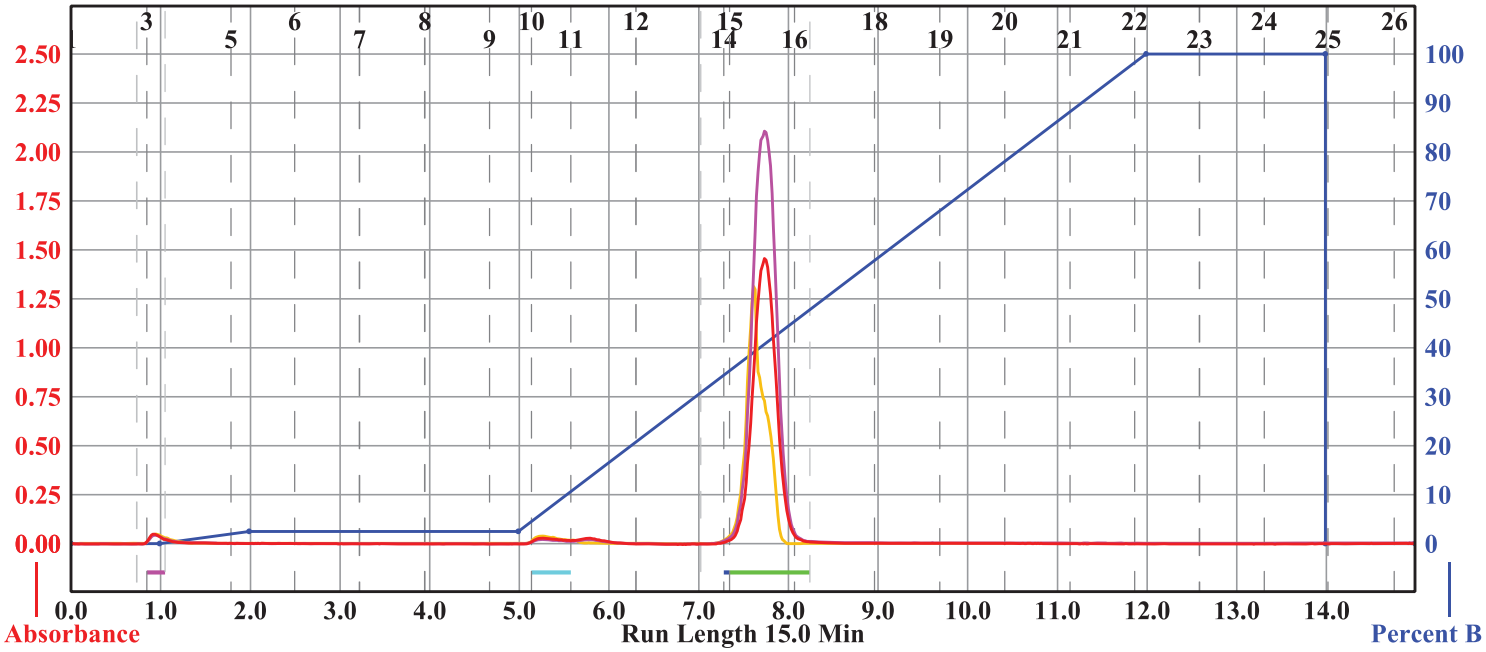
Sample: RPD-FGF23-A-93.2.

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

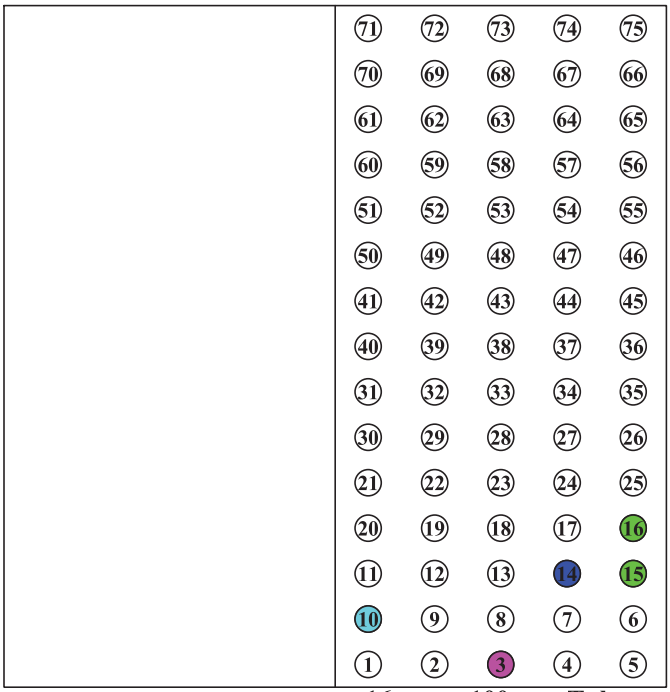
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:3
2	A:10	A:10
3	A:14	A:14
4	A:15	A:16

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	2.5	A1 hexane	B2 methanol
3.0	2.5	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S191

Compound 13a | Rf 200 | Monday 19 June 2017 12:51PM

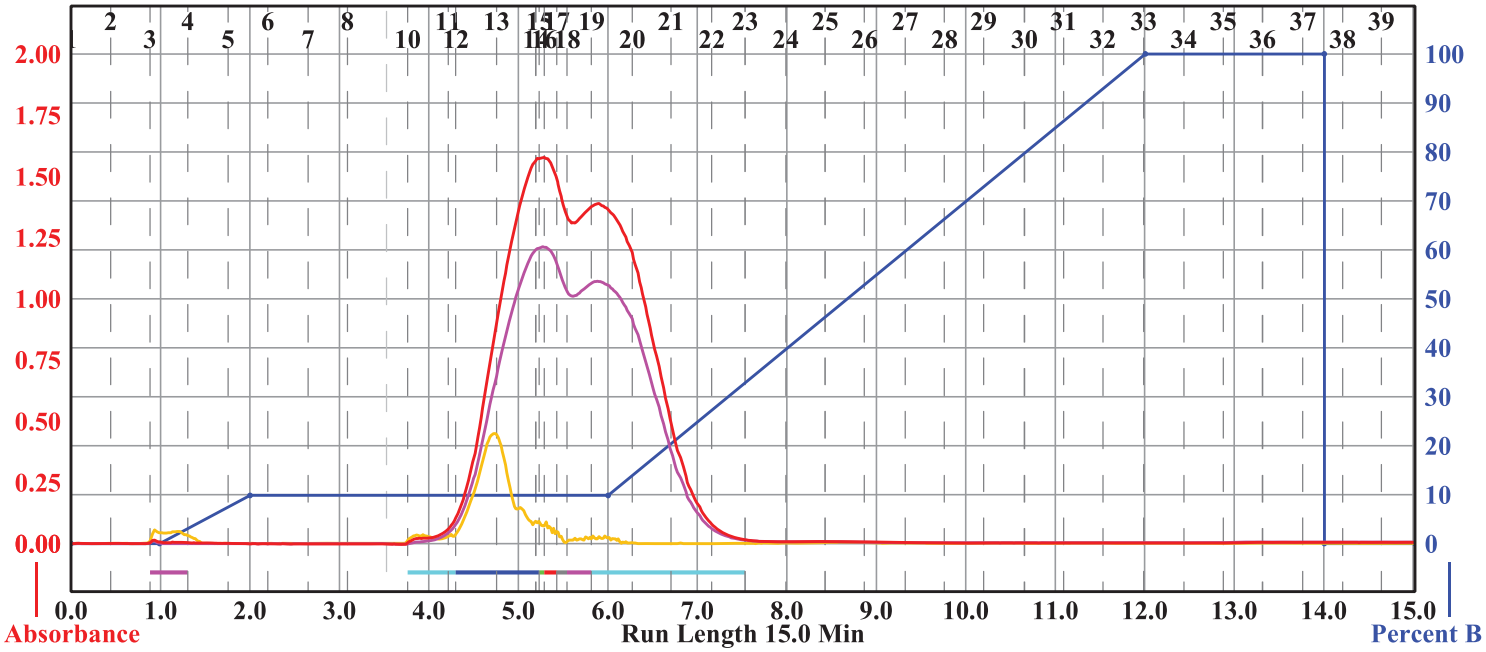
Sample: moi-a-17_3

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

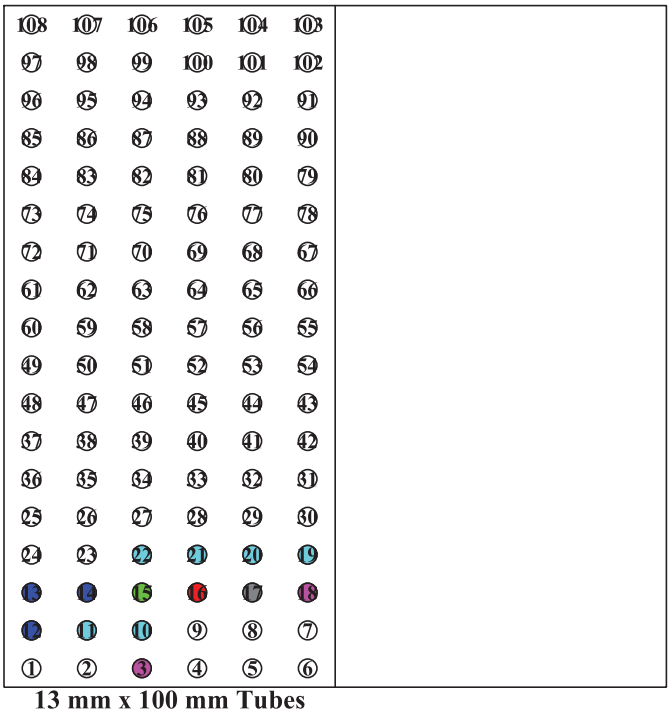
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:3	A:3
2	A:10	A:11
3	A:12	A:14
4	A:15	A:15
5	A:16	A:16
6	A:17	A:17
7	A:18	A:18
8	A:19	A:22

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	9.9	A1 hexane	B2 methanol
4.0	9.9	A1 hexane	B2 methanol
6.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S192

Compound 13b | **Rf 200** | **Tuesday 20 June 2017 01:28PM**

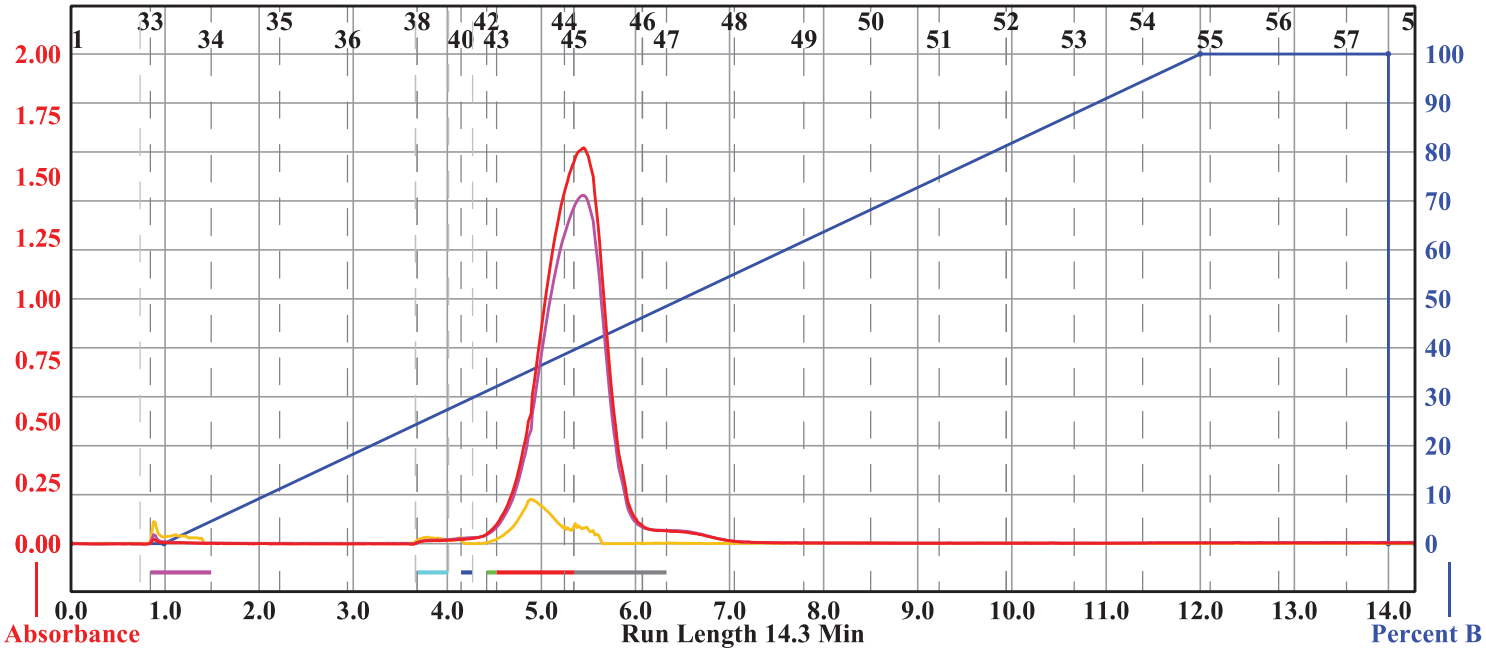
Sample: moi-a-21

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:33	A:33
2	A:38	A:38
3	A:40	A:40
4	A:42	A:42
5	A:43	A:44
6	A:45	A:46

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
11.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
0.3	0.0	A1 hexane	B2 methanol

S193

Compound 13c | Rf 200 | Monday 07 August 2017 12:44PM

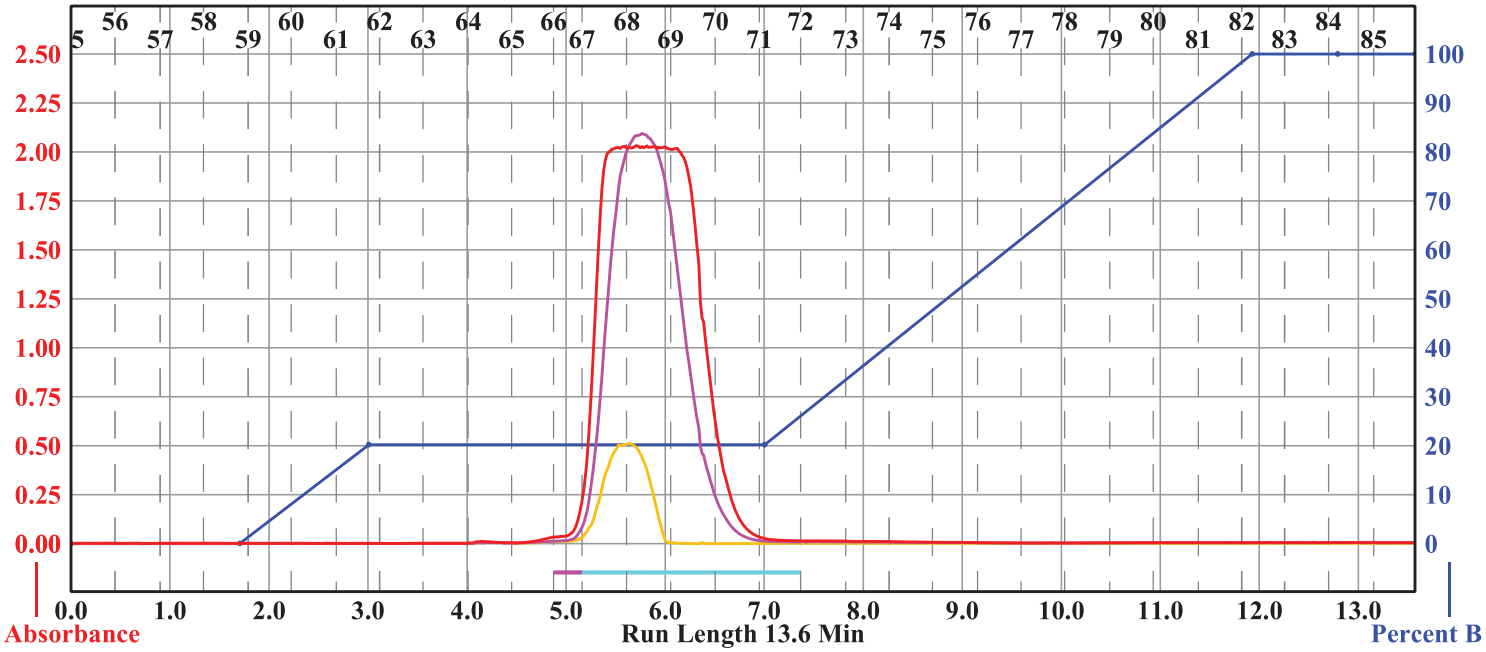
Sample: MoI-A-71-2

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 1.1 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:66	A:66
2	A:67	A:71

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.7	0.0	A1 hexane	B2 methanol
1.3	20.2	A1 hexane	B2 methanol
4.0	20.2	A1 hexane	B2 methanol
4.9	100.0	A1 hexane	B2 methanol
0.9	100.0	A1 hexane	B2 methanol
0.8	100.0	A1 hexane	B2 methanol

S194

Compound 14a | **Rf 200** | **Monday 07 August 2017 12:14PM**

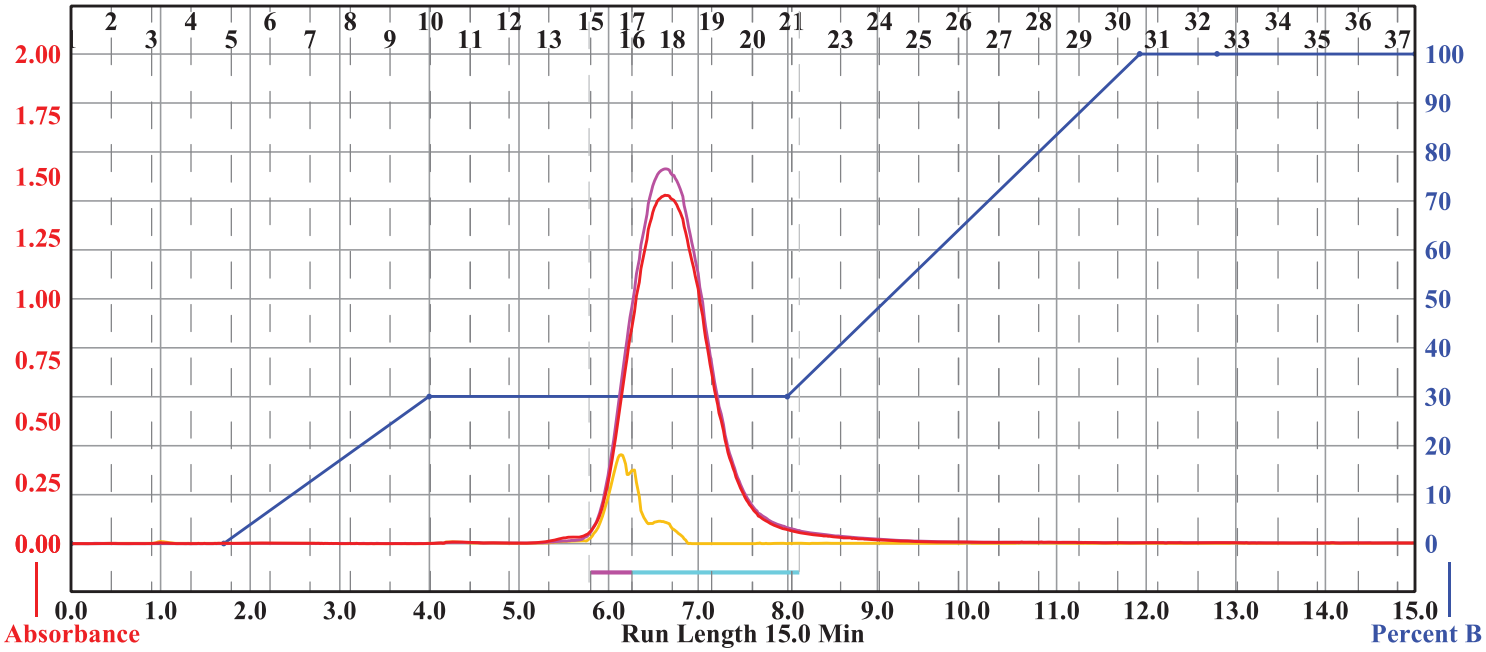
Sample: MOI-A-69-2

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:15	A:16
2	A:17	A:21

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.7	0.0	A1 hexane	B2 methanol
2.3	30.0	A1 hexane	B2 methanol
4.0	30.0	A1 hexane	B2 methanol
3.9	100.0	A1 hexane	B2 methanol
0.9	100.0	A1 hexane	B2 methanol
2.2	100.0	A1 hexane	B2 methanol

S195

Compound 14b | Rf 200 | Monday 07 August 2017 11:49AM

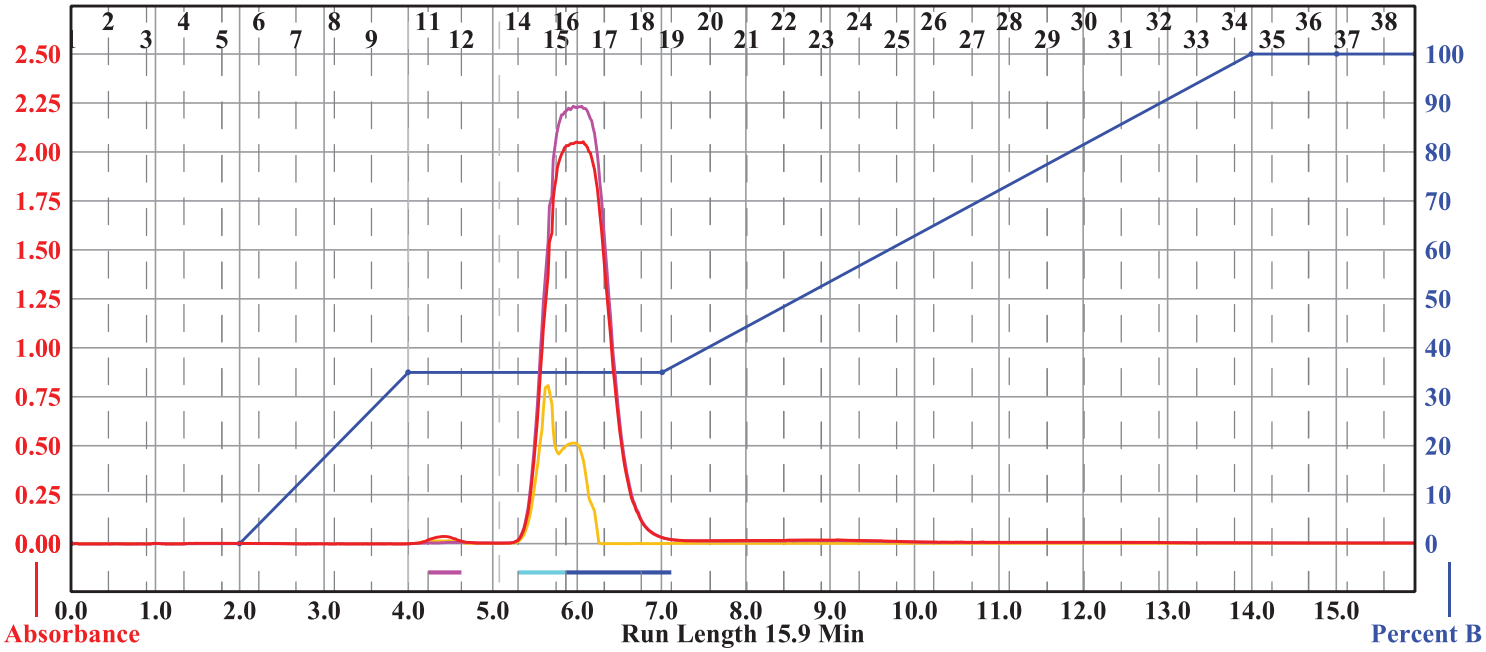
Sample: moi-a-85-2

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:11	A:11
2	A:14	A:15
3	A:16	A:18

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
2.0	0.0	A1 hexane	B2 methanol
2.0	35.0	A1 hexane	B2 methanol
3.0	35.0	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
1.0	100.0	A1 hexane	B2 methanol
0.9	100.0	A1 hexane	B2 methanol

S196

Compound 14c | **Rf 200** | **Tuesday 08 August 2017 11:00AM**

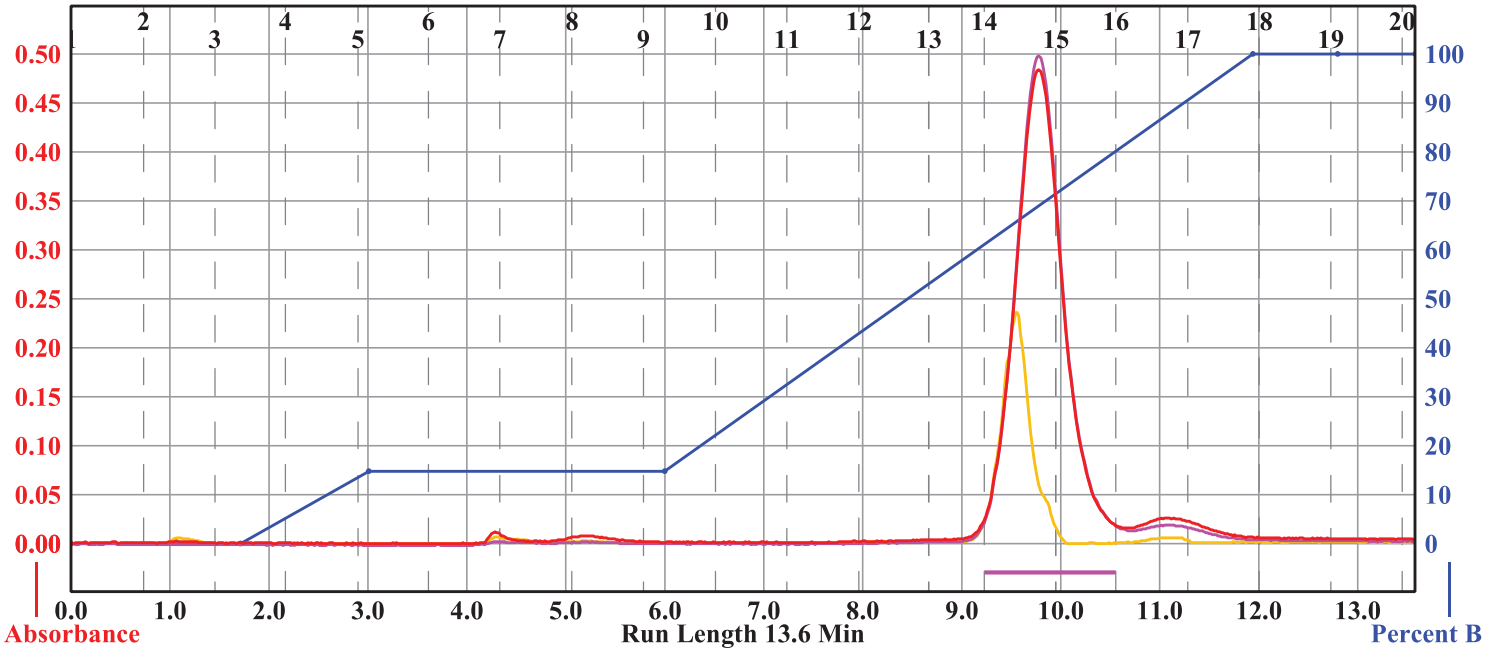
Sample: moi-a-137

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:14	A:15

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.7	0.0	A1 hexane	B2 methanol
1.3	14.8	A1 hexane	B2 methanol
3.0	14.8	A1 hexane	B2 methanol
5.9	100.0	A1 hexane	B2 methanol
0.9	100.0	A1 hexane	B2 methanol
0.8	100.0	A1 hexane	B2 methanol

S197

Compound 14d | **Rf 200** | **Thursday 27 July 2017 11:58AM**

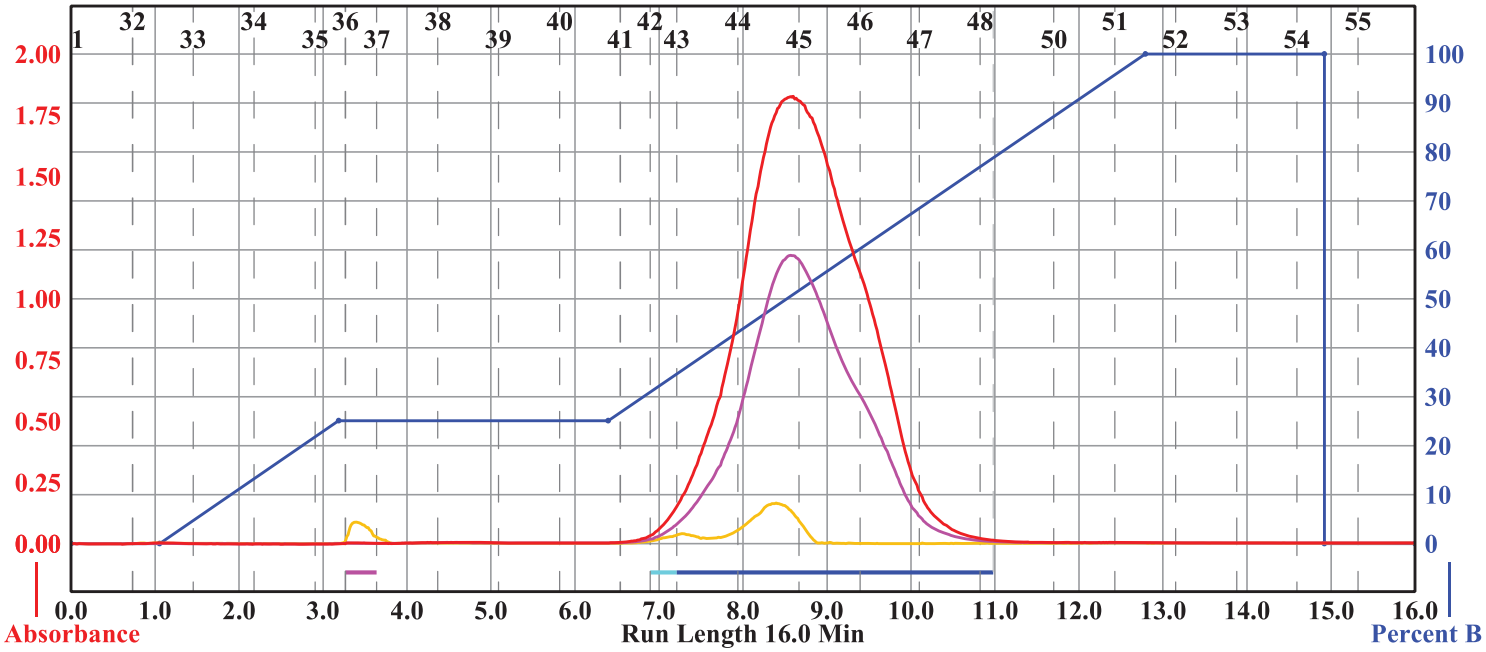
Sample: moi-a-67

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 10.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:36	A:36
2	A:42	A:42
3	A:43	A:48

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.1	0.0	A1 hexane	B2 methanol
2.1	25.1	A1 hexane	B2 methanol
3.2	25.1	A1 hexane	B2 methanol
6.4	100.0	A1 hexane	B2 methanol
2.1	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.1	0.0	A1 hexane	B2 methanol

S198

Compound 14e | Rf 200 | Tuesday 08 August 2017 08:09AM

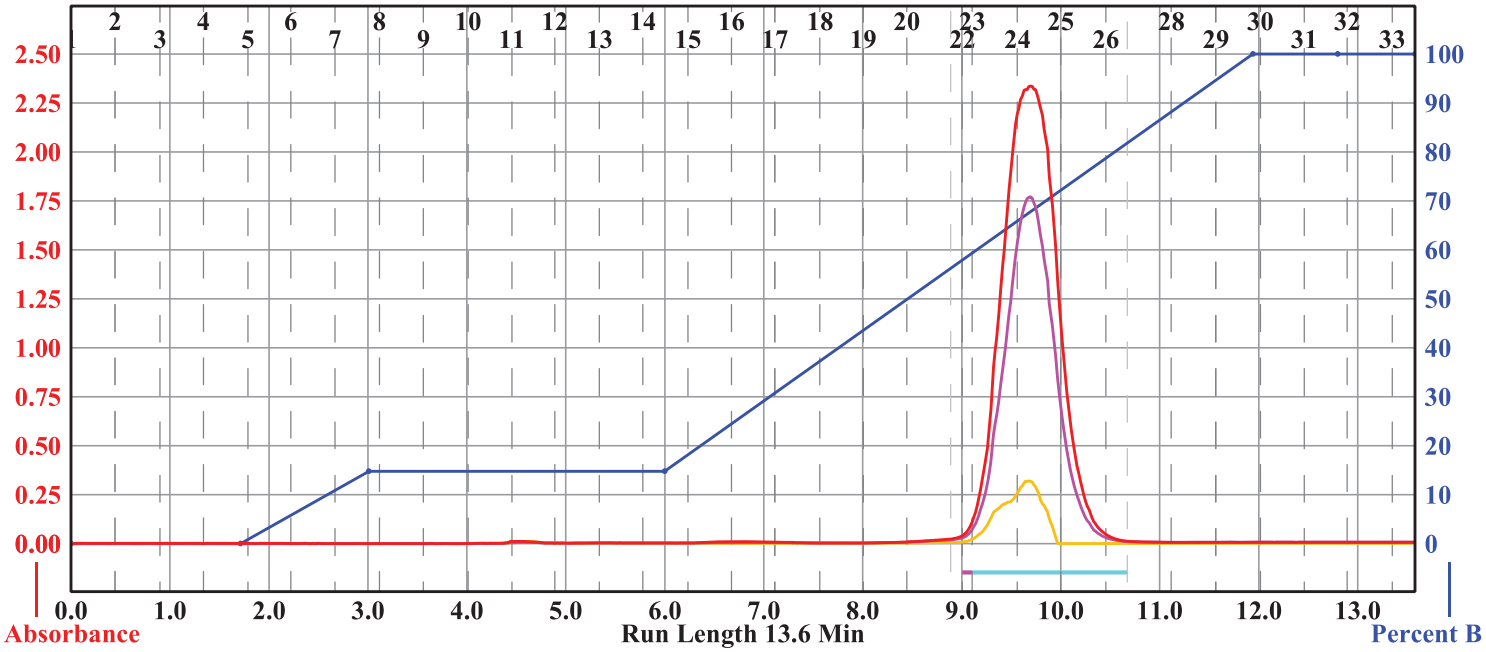
Sample: moi-a-127

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

108	107	106	105	104	103
97	98	99	100	101	102
96	95	94	93	92	91
85	86	87	88	89	90
84	83	82	81	80	79
73	74	75	76	77	78
72	71	70	69	68	67
61	62	63	64	65	66
60	59	58	57	56	55
49	50	51	52	53	54
48	47	46	45	44	43
37	38	39	40	41	42
36	35	34	33	32	31
25	26	27	28	29	30
24	23	22	21	20	19
13	14	15	16	17	18
12	11	10	9	8	7
1	2	3	4	5	6

13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:22	A:22
2	A:23	A:26

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.7	0.0	A1 hexane	B2 methanol
1.3	14.8	A1 hexane	B2 methanol
3.0	14.8	A1 hexane	B2 methanol
5.9	100.0	A1 hexane	B2 methanol
0.9	100.0	A1 hexane	B2 methanol
0.8	100.0	A1 hexane	B2 methanol

S199

Compound 14f

Rf 200

Monday 07 August 2017 03:08PM

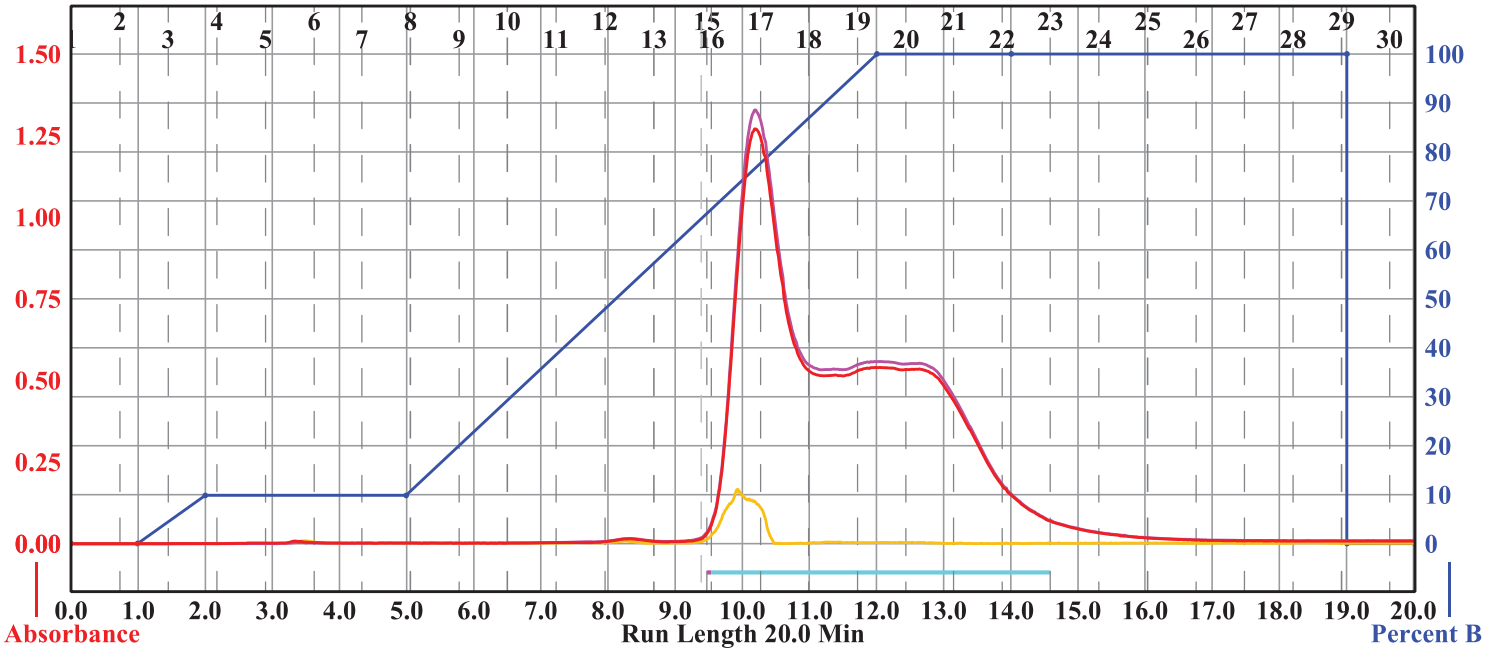
Sample: moi-a-87

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:15	A:15
2	A:16	A:22

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	9.9	A1 hexane	B2 methanol
3.0	9.9	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
5.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S200

Compound 16a

Rf 200

Wednesday 02 August 2017 01:34PM

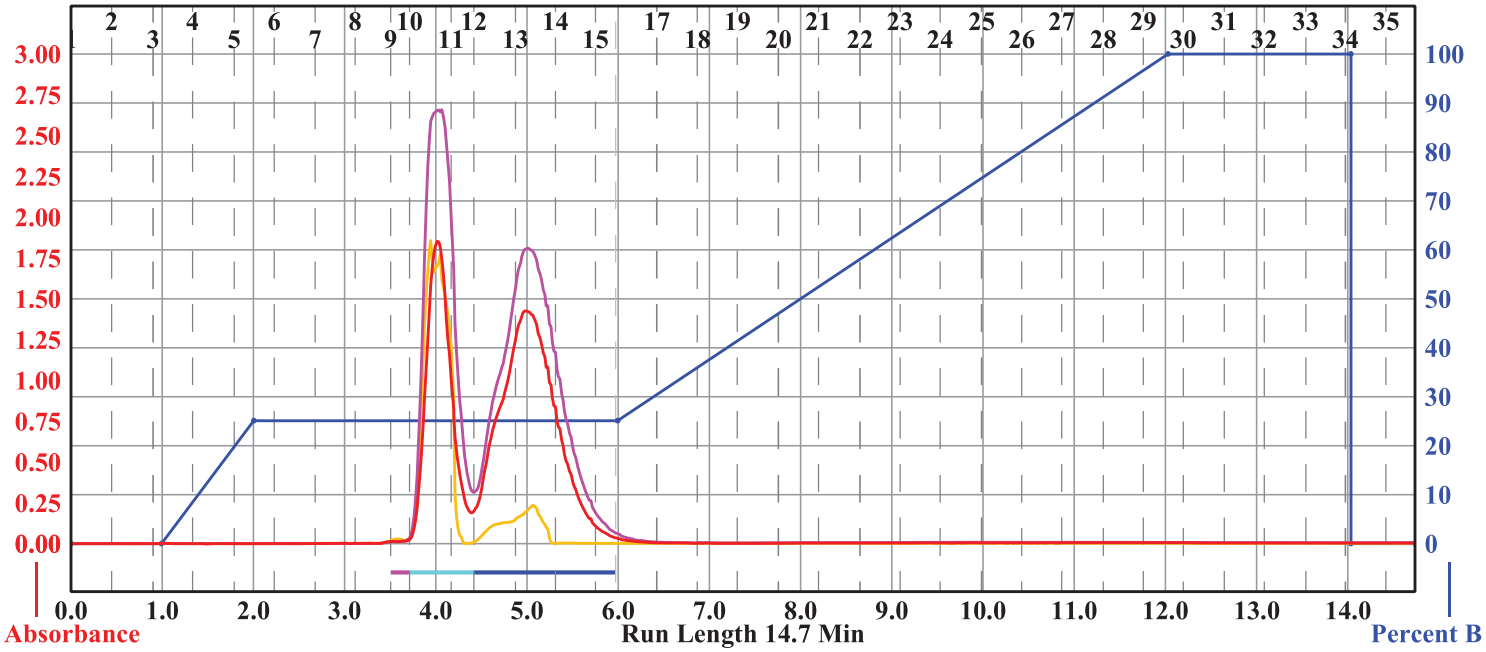
Sample: moi-a-91

4x Column: 1
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.1 min
Solvent: A1 hexane
Solvent: B2 methanol

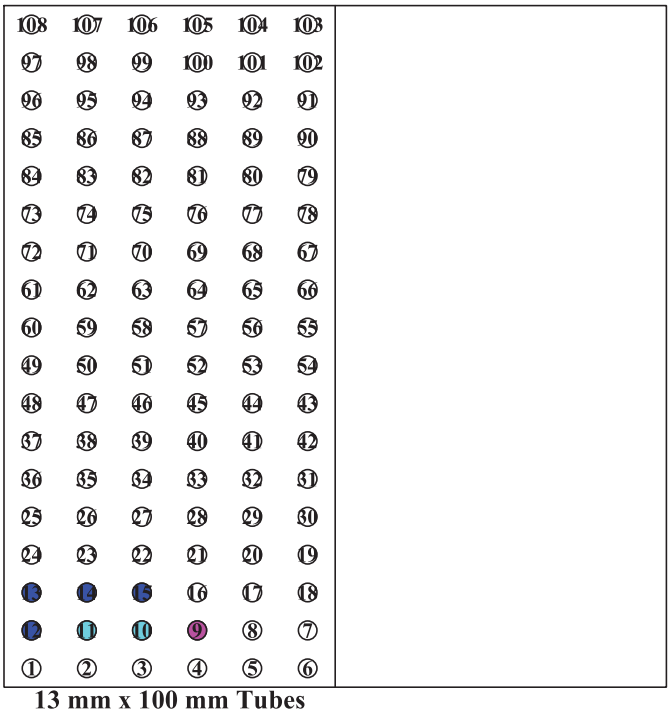
Peak Tube Volume: Max.
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A



13 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:9	A:9
2	A:10	A:11
3	A:12	A:15

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
1.0	25.1	A1 hexane	B2 methanol
4.0	25.1	A1 hexane	B2 methanol
6.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
0.7	0.0	A1 hexane	B2 methanol

S201

Compound 16b | Rf 200 | Tuesday 08 August 2017 12:42PM

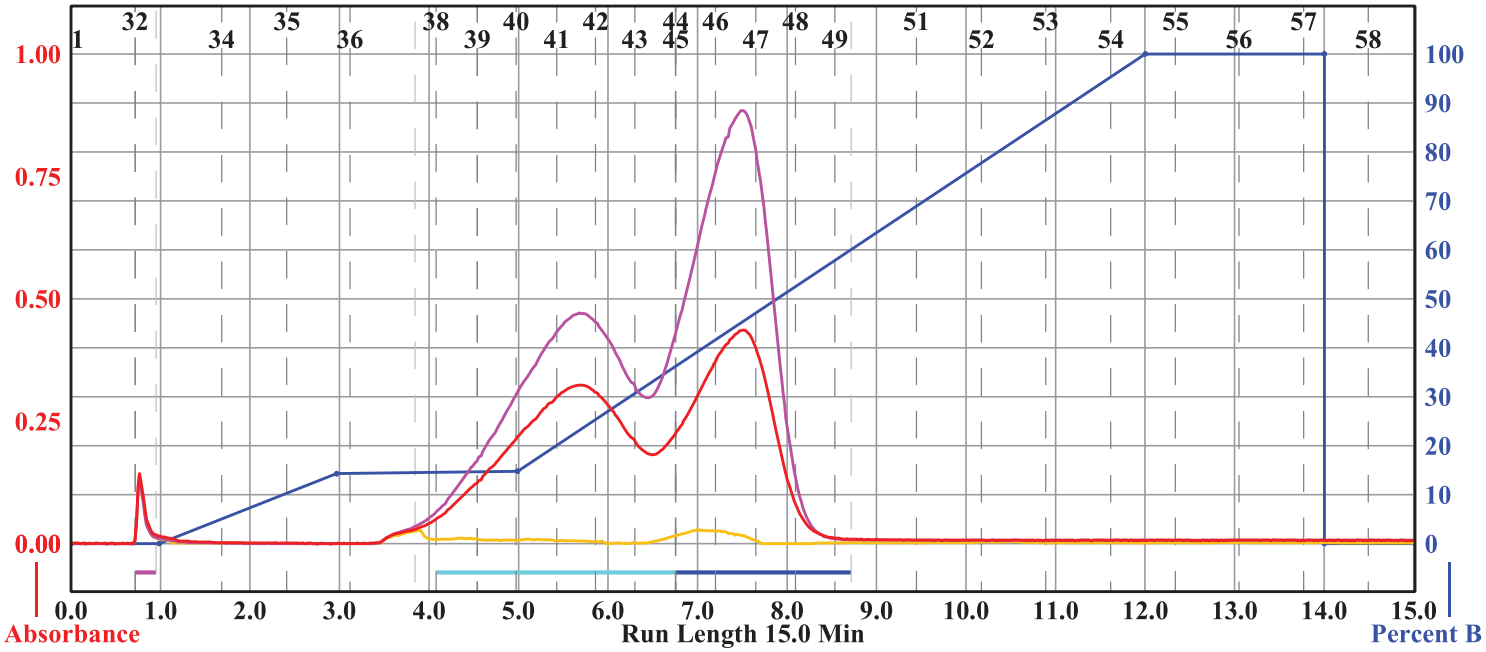
Sample: moi-a-129

4x Column: 2
RediSep Column: Silica 4g
Flow Rate: 18 ml/min
Equilibration Volume: 33.6 ml
Initial Waste: 0.0 ml
Air Purge: 5.0 min
Solvent: A1 hexane
Solvent: B2 methanol

Peak Tube Volume: 8 ml
Non-Peak Tube Volume: Max.
Loading Type: Solid
Wavelength 1 (red): 254nm
Peak Width: 30 sec
Threshold: 0.20 AU
Wavelength 2 (purple): 280nm

All Wavelength (orange): 200nm - 360nm
Peak Width: 30 sec
Threshold: 0.20 AU

Run Notes:



Rack A

71	72	73	74	75
70	69	68	67	66
61	62	63	64	65
60	59	58	57	56
51	52	53	54	55
50	49	48	47	46
41	42	43	44	45
40	39	38	37	36
31	32	33	34	35
30	29	28	27	26
21	22	23	24	25
20	19	18	17	16
11	12	13	14	15
10	9	8	7	6
1	2	3	4	5

16 mm x 100 mm Tubes

Peak #	Start Tube	End Tube
1	A:32	A:32
2	A:38	A:44
3	A:45	A:49

Duration	%B	Solvent A	Solvent B
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol
2.0	14.3	A1 hexane	B2 methanol
2.0	14.8	A1 hexane	B2 methanol
7.0	100.0	A1 hexane	B2 methanol
2.0	100.0	A1 hexane	B2 methanol
0.0	0.0	A1 hexane	B2 methanol
1.0	0.0	A1 hexane	B2 methanol

S202