α-Benzoyloxylation of β-Keto Sulfides at Ambient Temperature

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General Methods

^{1}H NMR spectra were recorded on a Varian 500 spectrometer at ambient temperature with $CDCl_3$ as solvent. Data are reported as follows: chemical shifts (δ), multiplicity, coupling constants and integration. ^{13}C NMR spectra were recorded operating at 126 MHz at 27 °C with $CDCl_3$ as solvent. Chemical shifts (δ) are given in ppm. Coupling constants (*J*) are reported in Hz. Infrared spectra were recorded on a FT-IR spectrophotometer and are reported in wavenumbers. High resolution mass spectra (HRMS) were obtained using a Bruker High Resolution Mass Spectrometer in fast atom bombardment (FAB+) ionization mode or acquired using an Bruker micrOTOF-Q II 10027. Analytical thin layer chromatography was performed using 0.25 mm silica gel 60-F plates. Flash chromatography was performed using columns of 230-400 mesh silica gel 60 (0.040-0.063 mm). Yields refer to chromatographically pure materials. Toluene was distilled from sodium benzophenone-ketyl under argon prior to use. Benzoyl peroxide (reagent grade, ≥ 98%) was purchased from Sigma-Aldrich and used as received. Benzoyl peroxides **2q-t** were prepared according to the reported procedure.[1]

[1]W.-Y. Yu, W. N. Sit, Z. Zhou and A. S.-C. Chan*, Org. Lett.*, 2009, 11 (**15**), 3174–3177.

General procedure for the α-Benzoyloxylation of β-Keto Sulfides.

A solution of a β-keto sulfides **1** (333 μmol) and benzoyl peroxide **2** (366 μmol) in Toluene (0.1 mL) was stirred in a sealed tube reactor at room temperature (4-5 days). The resulting reaction mixture was concentrated in a vacuum, diluted with CH_2Cl_2 and washed with saturated $NaHCO_3$ aqueous solution. The organic layer was dried over anhydrous Na_2SO_4, filtered and concentrated to give the crude product which was purified by column chromatography using petroleum ether/ethyl ether to afford the corresponding O-benzoyloxylation products **3**. *Caution*: Aryl acylperoxides are potentially explosive and should be handled with care.

3a: Yield 78% (0.074 g); colorless oil. IR (neat): 3059, 2970, 1719, 1601, 1581, 1475, 1356, 1317, 1266, 1177, 1088, 1065, 1025, 950, 735, 708, 689 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.11 – 8.10 (m, 2H), 7.61 – 7.56 (m, 1H), 7.55 – 7.49 (m, 2H), 7.48 – 7.46 (m, 2H), 7.35 – 7.34 (m, 3H), 6.44 (s, 1H), 2.29 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.3, 165.4, 134.0, 133.8, 130.3, 130.1, 129.4, 129.3, 128.72, 128.6, 82.4, 26.5. HRMS (ESI) Calcd. for $C_{16}H_{14}O_3S$ ($M+Na^+$) m/z 309,0556, found 309,0570.

3b: Yield 76% (0.076 g); yellow oil. IR (neat): 3067, 2923, 1788, 1765, 1718, 1600, 1492, 1451, 1356, 1317, 1267, 1220, 1176, 1087, 1065, 1022, 994, 809, 705 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.12 – 8.06 (m, 2H), 7.61 (t, J = 7.5 Hz, 1H), 7.49 – 7.43 (m, 4H), 7.15 (d, J = 7.9 Hz, 2H), 6.39 (s, 1H), 2.34 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.4, 165.4, 139.8, 134.5, 133.8, 130.2, 130.1, 129.1, 128.7, 126.0, 82.5, 26.5, 21.3. HRMS (ESI) Calcd. for $C_{17}H_{16}O_3S$ ($M+Na^+$) m/z 323,0712, found 323,0715.

3c: Yield 78% (0.082 g); yellow oil. IR (neat): 3067, 2838, 1717, 1590, 1568, 1493, 1451, 1356, 1319, 1290, 1269, 1245, 1176, 1088, 1065, 1026, 829, 800, 707 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.10 (dd, J = 8.2, 1.1 Hz, 2H), 7.61 (dd, J = 10.6, 4.3 Hz, 1H), 7.49 – 7.46 (m, 4H), 6.88 – 6.85 (m, 2H), 6.33 (s, 1H), 3.80 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.5, 165.4, 161.0, 136.8, 133.7, 130.0, 129.1, 128.7, 119.7, 114.9, 82.6, 55.4, 26.6. HRMS (ESI) Calcd. for $C_{17}H_{16}O_4S$ ($M+Na^+$) m/z 339,0662, found 339,0666.

3d: Yield 77% (0.093 g); yellow solid; m. p. = 49-51°C. IR (neat): 3066, 2947, 1739, 1715, 1685, 1600, 1470, 1450, 1357, 1321, 1267, 1250, 1173, 1085, 1065, 1005, 814, 703 cm-1. ^1H NMR (500

MHz, $CDCl_3$) δ 8.09 – 8.07 (m, 2H), 7.61 (dd, *J* = 10.6, 4.3 Hz, 1H), 7.49 – 7.45 (m, 4H), 7.41 – 7.40 (m, 2H), 6.43 (s, 1H), 2.29 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 196.9, 165.2, 135.4, 133.9, 132.5, 130.2, 130.0, 128.7, 128.5, 124.0, 81.8, 26.4. HRMS (ESI) Calcd. for $C_{16}H_{13}O_3SBr$ ($M+Na^+$) m/z 386,9661, found 386,9655.

3e: Yield 69% (0.070 g); colorless oil. IR (neat): 3097, 3068, 2928, 1738, 1717, 1602, 1587, 1489, 1451, 1356, 1317, 1267, 1222, 1177, 1156, 1102, 1087, 1065, 1027, 833, 708 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.12 – 8.10 (m, 2H), 7.63 (dd, J = 10.7, 4.3 Hz, 1H), 7.58 – 7.52 (m, 2H), 7.50 (t, J = 7.8 Hz, 2H), 7.08 – 7.04 (m, 2H), 6.41 (s, 1H), 2.31 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.1, 165.3, 163.7 (d, *J* = 250.5 Hz), 136.8 (d, *J* = 8.6 Hz), 133.9, 130.0, 128.9, 128.7, 124.7 (d, *J* = 3.4 Hz), 116.6 (d, *J* = 22.0 Hz), 82.1, 26.5. HRMS (ESI) Calcd. for $C_{16}H_{13}O_3SF$ ($M+Na^+$) m/z 327,0462, found 327,0470.

3f: Yield 62% (0.068 g); yellow oil. IR (neat): 3104, 3068, 2920, 1719, 1599, 1578, 1514, 1477, 1451, 1388, 1265, 1177, 1086, 1064, 1027, 954, 851, 740, 708 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.19 – 8.16 (m, 2H), 8.11 – 8.09 (m, 2H), 7.68 – 7.65 (m, 3H), 7.50 (t, *J* = 7.8 Hz, 2H), 6.58 (s, 1H), 2.35 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 196.4, 165.2, 147.6, 139.9, 134.2, 131.9, 130.1, 128.8, 128.5, 124.2, 81.0, 26.4. HRMS (ESI) Calcd. for $C_{16}H_{13}NO_5S$ ($M+Na^+$) m/z 354,0407, found 354,0420 .

3g: Yield 76% (0.087 g); orange oil. IR (neat): 3070, 2955, 2914, 1740, 1712, 1602, 1584, 1566, 1470, 1452, 1431, 1355, 1290, 1245, 1174, 1088, 1064, 1024, 949, 897, 826, 800, 742, 709 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.11 – 8.08 (m, 2H), 7.94 (dd, *J* = 7.8, 1.4 Hz, 1H), 7.66 (dd, *J* = 8.1, 0.6 Hz, 1H), 7.62 – 7.59 (m, 1H), 7.49 – 7.44 (m, 3H), 7.31 (td, *J* = 7.8, 1.0 Hz, 1H), 6.62 (s, 1H), 3.93 (s, 3H), 2.36 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.8, 166.9, 165.6, 135.3, 133.9, 132.7, 131.2, 130.4, 130.19, 130.12, 128.9, 128.7, 126.9, 81.7, 52.5, 26.5. HRMS (ESI) Calcd. for $C_{18}H_{16}O_5S$ ($M+Na^+$) m/z 367,0611, found 367,0617.

3h: Yield 85% (0.085 g); yellow oil. IR (neat): 3068, 2920, 2855, 1789, 1718, 1602, 1592, 1475, 1451, 1416, 1356, 1317, 1266, 1209, 1176, 1106, 1087, 1065, 1024, 951, 780, 708 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.11 – 8.09 (m, 2H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.48 (t, *J* = 7.8 Hz, 2H), 7.36 (d, *J* = 8.1 Hz, 2H), 7.22 (t, *J* = 7.6 Hz, 1H), 7.16 (d, *J* = 7.5 Hz, 1H), 6.42 (s, 1H), 2.32 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.4, 165.4, 139.2, 134.7, 133.8, 131.0, 130.2, 130.1, 129.6, 129.2, 129.1, 128.7, 82.5, 26.5, 21.3. HRMS (ESI) Calcd. for $C_{17}H_{16}O_3S$ ($M+Na^+$) m/z 323,0712, found 323,0721.

3i: Yield 81% (0.081 g); colorless oil. IR (neat): 3062, 2918, 2850, 1788, 1718, 1691, 1601, 1585, 1471, 1451, 1420, 1356, 1317, 1266, 1211, 1175, 1088, 1087, 1064, 1022, 993, 733 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (dd, *J* = 8.2, 0.9 Hz, 2H), 7.62 – 7.56 (m, 2H), 7.47 (t, *J* = 7.8 Hz, 2H), 7.25 – 7.22 (m, 2H), 7.18 – 7.14 (m, 1H), 6.40 (s, 1H), 2.50 (s, 3H), 2.29 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.8, 165.5, 141.4, 135.0, 133.8, 130.9, 130.0, 129.7, 129.5, 129.0, 128.7, 126.9, 82.8, 26.4, 21.1. HRMS (ESI) Calcd. for $C_{17}H_{16}O_3S$ ($M+Na^+$) m/z 323,0712, found 323,0718.

3J: Yield 84% (0.093 g); yellow oil. IR (neat): 3073, 3055, 2924, 1736, 1719, 1621, 1600, 1583, 1497, 1449, 1423, 1354, 1311, 1265, 1174, 1160, 1085, 1063, 1022, 895, 864, 818, 742 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.13 – 8.12 (m, 2H), 8.06 – 8.04 (m, 1H), 7.82 – 7.78 (m, 2H), 7.77 – 7.75 (m, 1H), 7.62 – 7.58 (m, 2H), 7.50 – 7.45 (m, 4H), 6.51 (s, 1H), 2.30 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.3, 165.4, 133.8, 133.7, 133.5, 133.3, 130.4, 130.1, 129.1, 129.0, 128.7, 127.9, 127.8, 127.26, 127.21, 126.9, 82.4, 26.6. HRMS (ESI) Calcd. for $C_{20}H_{16}O_3S$ ($M+Na^+$) m/z 359,0712, found 359,0714.

3k: Yield 88% (0.098 g); yellow oil. IR (neat): 3070, 3056, 1714, 1601, 1579, 1476, 1453, 1437, 1356, 1316, 1265, 1176, 1087, 1064, 1024, 996, 741, 709 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.08 (d, *J* = 8.1 Hz, 2H), 7.64 (d, *J* = 7.8 Hz, 2H), 7.60 (td, *J* = 7.6, 0.8 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H), 7.37 – 7.33 (m, 1H), 7.31 (t, *J* = 7.6 Hz, 2H), 6.66 (s, 1H), 2.27 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.7, 165.2, 136.0, 133.8, 130.3, 130.0, 129.4, 129.0, 128.6, 125.6, 77.0, 26.1. HRMS (ESI) Calcd. for $C_{16}H_{14}O_3Se$ ($M+Na^+$) m/z 355,0000, found 355,0001.

3l: Yield 82% (0.103 g); yellow oil. IR (neat): 3062, 3028, 2923, 1787, 1764, 1718, 1601, 1581, 1495, 1452, 1440, 1317, 1265, 1211, 1174, 1087, 1065, 1019, 993, 748 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 – 8.08 (m, 2H), 7.62 – 7.59 (m, 1H), 7.48 – 7.46 (m, 4H), 7.35 – 7.25 (m, 5H), 7.21 – 7.18 (m, 1H), 7.15 – 7.14 (m, 2H), 6.42 (s, 1H), 3.09 – 3.04 (m, 1H), 2.87 – 2.81 (m, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.0, 165.4, 140.6, 134.1, 133.8, 130.1, 129.9, 129.39, 129.37, 129.0, 128.7, 128.6, 128.5, 126.3, 82.1, 40.7, 29.5. HRMS (ESI) Calcd. for $C_{23}H_{20}O_3S$ ($M+Na^+$) m/z 399,1025, found 399,1024.

3m: Yield 79% (0.086 g); yellow oil. IR (neat): 3063, 2974, 2935, 2870, 1785, 1758, 1710, 1602, 1585, 1477, 1448, 1316, 1272, 1219, 1176, 896, 794, 745, 706 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 – 8.09 (m, 3H), 7.68 – 7.65 (m, 1H), 7.54 – 7.48 (m, 3H), 7.46-7.44 (m, 1H), 7.35 – 7.33 (m, 2H), 6.74 (s, 1H), 1.33 (s, 9H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 204.4, 165.7, 134.4, 133.6, 130.1, 129.9, 129.3, 129.1, 129.0, 128.6, 79.1, 43.5, 27.5. HRMS (ESI) Calcd. for $C_{19}H_{20}O_3S$ ($M+Na^+$) m/z 351,1025, found 351,1025.

3n: Yield 76% (0.087 g); viscous yellow oil. IR (neat): 3063, 2979, 1722, 1693, 1599, 1580, 1475, 1446, 1314, 1269, 1233, 1177, 1087, 1065, 1023, 948, 746, 709 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.15 – 8.13 (m, 2H), 8.02 – 8.00 (m, 2H), 7.61 – 7.58 (m, 2H), 7.49 – 7.45 (m, 6H), 7.37 – 7.33 (m, 1H), 7.31 – 7.30 (m, 2H), 7.28 (s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 188.8, 165.7, 134.5, 134.2, 133.8, 133.7, 130.1, 129.9, 129.5, 129.2, 129.1, 128.9, 128.8, 128.6, 79.8. HRMS (ESI) Calcd. For $C_{21}H_{16}O_3S$ ($M+Na^+$) m/z 371.0712, found 371.0711.

3o: Yield 68% (0.068 g); yellow oil. IR (neat): 3058, 2955, 1790, 1758, 1725, 1602, 1454, 1357, 1319, 1265, 1226, 1174, 1088, 1066, 1026, 996, 734 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 – 8.08 (m, 2H), 7.60 – 7.58 (m, 3H), 7.49 – 7.47 (m, 2H), 7.36 – 7.34 (m, 3H), 6.46 (s, 1H), 3.70 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 166.6, 165.4, 134.6, 133.8, 130.16, 130.12, 129.5, 129.2, 129.0, 128.6, 77.4, 52.9. HRMS (ESI) Calcd. for $C_{16}H_{14}O_4S$ ($M+Na^+$) m/z 325,0505, found 325,0506.

3p: Yield 78% (0.070 g); yellow oil. IR (neat): 3062, 2932, 2850, 1785, 1732, 1694, 1602, 1585, 1475, 1448, 1316, 1300, 1240, 1220, 1177, 1081, 1058, 1023, 998, 936, 733, 706 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.03 (dd, J = 8.3, 1.1 Hz, 2H), 7.68 – 7.65 (m, 3H), 7.51 – 7.48 (m, 3H), 7.46 – 7.44 (m, 2H), 6.77 (s, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 163.8, 136.0, 134.5, 130.8, 130.7, 130.2, 129.9, 129.7, 128.9, 113.8, 65.5. HRMS (ESI) Calcd. for $C_{15}H_{11}NO_2S$ ($M+Na^+$) m/z 292,0403, found 292,0401.

3q: Yield 77% (0.081 g); yellow oil. IR (neat): 3068, 3003, 2964, 2838, 2540, 1717, 1687, 1589, 1570, 1493, 1452, 1290, 1245, 1176, 1088, 1066, 1025, 829, 801, 707 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.07 – 8.05 (m, 2H), 7.56 – 7.54 (m, 2H), 7.35 – 7.33 (m, 3H), 6.97 – 6.95 (m, 2H), 6.42 (s, 1H), 3.88 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.6, 165.1, 164.1, 134.0, 132.2, 130.1, 129.4, 129.3, 121.4, 114.0, 82.2, 55.6, 26.5. HRMS (ESI) Calcd. for $C_{17}H_{16}O_4S$ ($M+Na^+$) m/z 339,0662, found 339,0676.

3r: Yield 77% (0.077 g); yellow oil. IR (neat): 3069, 2921, 1781, 1715, 1609, 1573, 1440, 1409, 1356, 1267, 1223, 1174, 1083, 1019, 1000, 950, 895, 839, 748 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.99 (d, J = 8.1 Hz, 2H), 7.56 – 7.54 (m, 2H), 7.35 – 7.33 (m, 3H), 7.29 – 7.26 (m, 2H), 6.43 (s, 1H), 2.43 (s, 3H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.5, 165.5, 144.7, 134.0, 130.19, 130.11, 129.46, 129.41, 129.3, 126.3, 82.3, 26.5, 21.9. HRMS (ESI) Calcd. for $C_{17}H_{16}O_3S$ ($M+Na^+$) m/z 323,0712, found 323,0714.

3s: Yield 81% (0.097 g); white solid; m. p. = 77-79°C. IR (neat): 3070, 3044, 2941, 2858, 1738, 1714, 1589, 1481, 1322, 1260, 1219, 1094, 1064, 1007, 952, 840, 741 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.95 (d, J = 8.4 Hz, 2H), 7.62 (d, J = 8.4 Hz, 2H), 7.53 (dd, J = 7.4, 1.6 Hz, 2H), 7.35 (dd, J = 8.4,

4.6 Hz, 3H), 6.42 (s, 1H), 2.30 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 196.9, 164.8, 134.0, 132.1, 131.5, 129.8, 129.5, 129.4, 129.1, 128.0, 82.6, 26.5. HRMS (ESI) Calcd. for $C_{16}H_{13}O_3SBr$ ($M+Na^+$) m/z 386,9661, found 386,9667.

3t: Yield 68% (0.075 g); yellow solid; m. p. = 91-94°C. IR (neat): 3112, 3078, 3068, 2936, 1734, 1714, 1604, 1515, 1439, 1342, 1316, 1269, 1196, 1174, 1098, 1014, 955, 874, 842, 737 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.33 – 8.31 (m, 2H), 8.27 – 8.25 (m, 2H), 7.54 – 7.53 (m, 2H), 7.37 – 7.36 (m, 3H), 6.46 (s, 1H), 2.34 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 196.3, 163.7, 151.0, 134.4, 134.0, 131.2, 129.6, 129.56, 129.50, 123.8, 83.1, 26.5. HRMS (ESI) Calcd. for $C_{16}H_{13}NO_5S$ ($M+Na^+$) m/z 354,0407, found 354,0399.

3u: Yield 80% (0.080 g); colorless oil. IR (neat): 3062, 3030, 2966, 1715, 1689, 1600, 1580, 1493, 1451, 1356, 1317, 1269, 1177, 1088, 1066, 1025, 951, 899, 744 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 7.94 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.59 – 7.56 (m, 1H), 7.43 – 7.40 (m, 2H), 7.36 – 7.29 (m, 4H), 7.26 – 7.25 (m, 1H), 6.17 (s, 1H), 3.96 (d, *J* = 13.4 Hz, 1H), 3.84 (d, *J* = 13.4 Hz, 1H), 2.28 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.7, 165.6, 137.0, 133.6, 130.0, 129.2, 128.88, 128.81, 128.5, 127.5, 79.8, 34.5, 26.0. HRMS (ESI) Calcd. for $C_{17}H_{16}O_3S$ ($M+Na^+$) m/z 323,0712, found 323,0714.

3v: Yield 66% (0.052 g); colorless oil. IR (neat): 3066, 2974, 2931, 2873, 1788, 1715, 1601, 1582, 1493, 1451, 1356, 1317, 1269, 1210, 1176, 1089, 1066, 1024, 950, 899, 708 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.09 (dd, *J* = 8.3, 1.2 Hz, 2H), 7.61 – 7.58 (m, 1H), 7.48 – 7.45 (m, 2H), 6.24 (s, 1H), 2.78 (dq, *J* = 12.9, 7.4 Hz, 1H), 2.64 (dq, *J* = 12.8, 7.5 Hz, 1H), 2.39 (s, 3H), 1.34 (t, *J* = 7.5 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 197.8, 165.7, 133.7, 130.0, 129.1, 128.6, 80.0, 26.1, 24.2, 15.1. HRMS (ESI) Calcd. for $C_{12}H_{14}O_3S$ ($M+Na^+$) m/z 261,0556, found 261,0553.

3w: Yield 75% (0.098 g); yellow oil. IR (neat): 3063, 2957, 2914, 1740, 1717, 1683, 1598, 1580, 1471, 1449, 1352, 1268, 1201, 1176, 1089, 1065, 1024, 949, 899, 743, 710 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.10 – 8.08 (m, 2H), 8.05 – 8.03 (m, 2H), 7.96 – 7.94 (m, 2H), 7.62 – 7.55 (m, 3H), 7.50 – 7.40 (m, 6H), 7.19 (s, 1H), 4.41 (d, *J* = 16.0 Hz, 1H), 4.12 (d, *J* = 16.0 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 194.0, 189.4, 165.7, 135.4, 134.1, 133.8, 133.77, 133.73, 130.1, 129.0, 128.9, 128.86, 128.83, 128.6, 128.5, 75.5, 36.8. HRMS (ESI) Calcd. for $C_{23}H_{18}O_4S$ ($M+Na^+$) m/z 413,0818, found 413,0822.

3aa: Yield 75% (0.075 g); yellow oil. IR (neat): 3067, 2995, 2937, 1714, 1687, 1601, 1580, 1474, 1453, 1353, 1317, 1272, 1223, 1177, 1083, 1062, 1023, 747, 709 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.02 (d, *J* = 7.3 Hz, 2H), 7.59 (t, *J* = 7.5 Hz, 1H), 7.57 – 7.54 (m, 2H), 7.46 (t, *J* = 7.8 Hz, 2H), 7.40 (t, *J* = 7.4 Hz, 1H), 7.33 (t, *J* = 7.4 Hz, 2H), 2.20 (s, 3H), 1.89 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$)

δ 199.6, 165.3, 137.0, 133.7, 130.2, 130.1, 130.0, 129.5, 129.1, 128.6, 92.6, 24.7, 22.8. HRMS (ESI) Calcd. for $C_{17}H_{16}O_3S$ ($M+Na^+$) m/z 323,0712, found 323,0718.

3ab: Yield 77% (0.080 g); colorless oil. IR (neat): 3059, 2977, 2938, 2880, 1714, 1600, 1585, 1474, 1454, 1436, 1354, 1265, 1208, 1177, 1087, 1063, 1024, 939, 891, 853, 750, 711 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.03 – 8.01 (m, 2H), 7.61 (t, *J* = 7.5 Hz, 1H), 7.50 – 7.45 (m, 4H), 7.39 – 7.36 (m, 1H), 7.32 – 7.29 (m, 2H), 2.43 – 2.36 (m, 1H), 2.19 (s, 3H), 2.18 – 2.14 (m, 1H), 1.03 (t, *J* = 7.4 Hz, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 200.1, 164.8, 137.0, 133.7, 130.0, 129.9, 129.5, 129.2, 129.0, 128.7, 96.2, 27.3, 25.4, 8.1. HRMS (ESI) Calcd. for $C_{18}H_{18}O_3S$ ($M+Na^+$) m/z 337,0869, found 337,0872.

3ac: Yield 67% (0.080 g); yellow solid; m. p. = 98-102°C. IR (neat): 3066, 2925, 2853, 1788, 1720, 1680, 1598, 1446, 1368, 1313, 1274, 1212, 1175, 1114, 1078, 1060, 1020, 996, 975, 755, 738 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.18 – 8.17 (m, 2H), 7.98 – 7.96 (m, 2H), 7.62 – 7.60 (m, 2H), 7.58 – 7.55 (m, 1H), 7.45 – 7.40 (m, 4H), 7.37 – 7.33 (m, 4H), 1.98 (s, 3H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 193.0, 164.9, 137.4, 134.4, 133.6, 132.7, 130.1, 129.9, 129.5, 129.3, 129.2, 129.1, 128.6, 128.4, 93.0, 24.4. HRMS (ESI) Calcd. for $C_{22}H_{18}O_3S$ ($M+Na^+$) m/z 385,0869, found 385,0878.

3ad: Yield 63% (0.091 g); yellow oil. IR (neat): 3060, 3030, 2891, 1788, 1766, 1723, 1600, 1580, 1493, 1452, 1313, 1269, 1174, 1087, 1064, 1024, 993, 864, 714 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.12 – 8.11 (m, 2H), 7.69 – 7.66 (m, 1H), 7.55 – 7.52 (m, 2H), 7.48 – 7.46 (m, 2H), 7.28 – 7.20 (m, 3H), 7.18 – 7.16 (m, 1H), 7.15 – 7.13 (m, 3H), 7.02 – 6.96 (m, 4H), 6.88 – 6.86 (m, 2H), 3.64 (dd, *J* = 46.8, 16.3 Hz, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.0, 164.4, 137.0, 135.6, 134.2, 133.8, 130.2, 129.7, 129.6, 129.2, 129.1, 128.9, 128.8, 128.5, 128.3, 128.2, 126.7, 126.6, 97.8, 42.8. HRMS (ESI) Calcd. for $C_{28}H_{22}O_3S$ ($M+Na^+$) m/z 461,1182, found 461,1197.

3ae: Yield 80% (0.079 g); yellow oil. IR (neat): 3061, 2979, 1795, 1716, 1600, 1581, 1476, 1453, 1391, 1273, 1212, 1174, 1113, 1019, 992, 748 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.02 – 8.01 (m, 2H), 7.60 – 7.57 (m, 3H), 7.45 (t, *J* = 7.8 Hz, 2H), 7.35-7.30 (m, 3H), 3.34 (ddd, *J* = 17.8, 9.8, 8.2 Hz, 1H), 3.24 (ddd, *J* = 17.5, 10.5, 4.9 Hz, 1H), 2.85 (ddd, *J* = 12.4, 10.5, 8.1 Hz, 1H), 2.35 (ddd, *J* = 12.4, 9.8, 4.9 Hz, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 195.7, 165.3, 134.8, 133.9, 130.6, 130.0, 129.5, 129.3, 129.0, 128.7, 95.8, 40.0, 27.3. HRMS (ESI) Calcd. for $C_{17}H_{14}O_3S$ ($M+Na^+$) m/z 321,0556, found 321,0571.

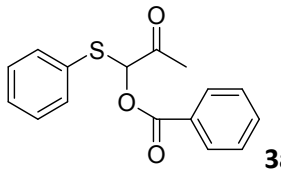
3af: Yield 68% (0.070 g); white solid; m. p. = 97-101°C. IR (neat): 3070, 3048, 2977, 2944, 1743, 1709, 1599, 1585, 1452, 1435, 1403, 1313, 1270, 1152, 1116, 1085, 1065, 1025, 979, 919, 753, 743 cm^{-1}. ^1H NMR (500 MHz, $CDCl_3$) δ 8.06 – 8.03 (m, 2H), 7.58 – 7.56 (m, 3H), 7.46 – 7.43 (m, 2H),

7.38 – 7.37 (m, 1H), 7.34 – 7.31 (m, 2H), 2.77 – 2.71 (m, 1H), 2.60 – 2.57 (m, 2H), 2.34 – 2.30 (m, 1H), 2.14 – 2.08 (m, 2H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 203.9, 165.2, 136.3, 133.6, 130.0, 129.9, 129.5, 129.09, 129.07, 128.6, 91.6, 33.9, 33.8, 17.6. HRMS (ESI) Calcd. for $C_{18}H_{16}O_3S$ ($M+Na^+$) m/z 335,0712, found 335,0703.

3ag: Yield 63% (0.068 g); white solid; m. p. = 99-102°C. IR (neat): 3071, 2947, 2929, 2865, 1699, 1681, 1601, 1580, 1453, 1419, 1323, 1292, 1272, 1229, 1113, 1067, 1024, 935, 901, 751, 707 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.04 – 8.02 (m, 2H), 7.60 – 7.57 (m, 1H), 7.52 – 7.50 (m, 2H), 7.47 – 7.44 (m, 2H), 7.37 – 7.34 (m, 1H), 7.31 – 7.28 (m, 2H), 2.78 (ddt, *J* = 13.5, 9.0, 5.9 Hz, 2H), 2.54 (ddd, *J* = 8.9, 5.1, 3.4 Hz, 1H), 2.31 – 2.26 (m, 1H), 2.00 – 1.95 (m, 3H), 1.91 – 1.87 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 199.3, 165.0, 136.5, 133.5, 129.98, 129.91, 129.8, 129.0, 128.9, 128.5, 93.2, 38.8, 37.9, 26.1, 22.9. HRMS (ESI) Calcd. for $C_{19}H_{18}O_3S$ ($M+Na^+$) m/z 349,0869, found 349,1796.

3ah: Yield 78% (0.088 g); white solid; m. p. = 106-110°C. IR (neat): 3063, 2957, 2936, 2867, 1713, 1599, 1579, 1476, 1453, 1273, 1233, 1174, 1089, 1067, 1022, 989, 944, 759, 736, 711 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.01 – 7.99 (m, 2H), 7.60 – 7.57 (m, 1H), 7.49 – 7.43 (m, 4H), 7.37 – 7.34 (m, 1H), 7.28 (t, *J* = 7.4 Hz, 2H), 2.85 – 2.79 (m, 1H), 2.76 – 2.72 (m, 1H), 2.68 – 2.63 (m, 1H), 2.02 (ddd, *J* = 15.1, 9.8, 2.3 Hz, 1H), 1.90 – 1.82 (m, 2H), 1.74 – 1.71 (m, 3H), 1.62 – 1.59 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 202.5, 164.8, 137.0, 133.5, 130.0, 129.86, 129.82, 129.6, 128.9, 128.6, 95.9, 40.1, 36.1, 28.9, 24.4, 23.7. HRMS (ESI) Calcd. for $C_{20}H_{20}O_3S$ ($M+Na^+$) m/z 363,1025, found 363,1022.

3ai: Yield 73% (0.085 g); colorless oil. IR (neat): 3068, 2941, 2918, 2855, 1727, 1703, 1600, 1580, 1468, 1450, 1254, 1219, 1100, 1095, 1066, 1025, 909, 834, 751, 709 cm^{-1}. ^{1}H NMR (500 MHz, $CDCl_3$) δ 8.06 (d, *J* = 8.1 Hz, 2H), 7.59 (t, *J* = 7.4 Hz, 1H), 7.45 (t, *J* = 7.7 Hz, 2H), 7.35 – 7.30 (m, 3H), 7.23 – 7.20 (m, 2H), 2.90 – 2.86 (m, 2H), 2.48 – 2.40 (m, 1H), 2.38 – 2.35 (m, 1H), 1.99-1.94 (m, 1H), 1.81 – 1.74 (m, 1H), 1.67 – 1.53 (m, 4H), 1.48 – 1.41 (m, 1H), 1.29 – 1.24 (m, 1H). ^{13}C NMR (126 MHz, $CDCl_3$) δ 204.2, 164.3, 136.6, 133.5, 130.17, 130.12, 129.61, 129.60, 128.9, 128.6, 94.8, 36.8, 32.0, 29.2, 25.84, 24.7, 23.3. HRMS (ESI) Calcd. for $C_{21}H_{22}O_3$ ($M+Na^+$) m/z 377.1181, found 377,1201.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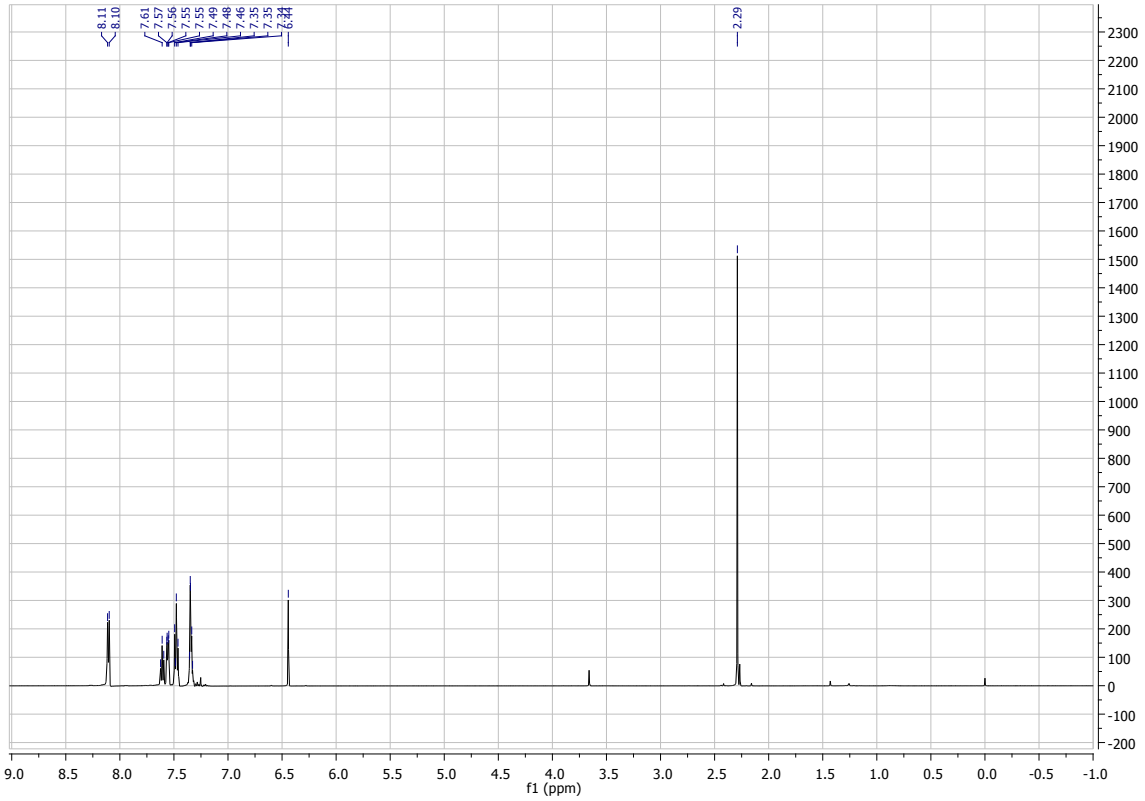
3a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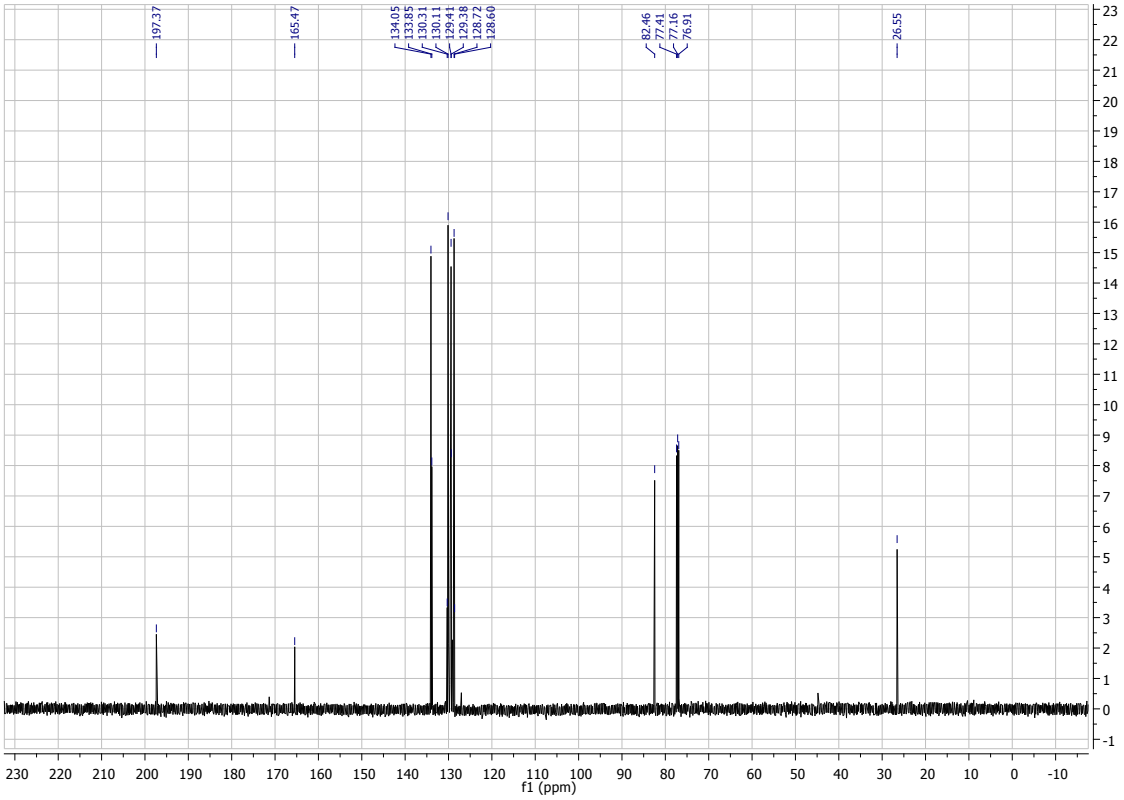
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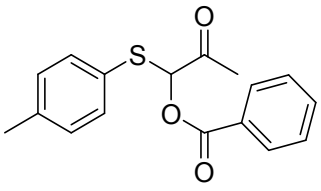
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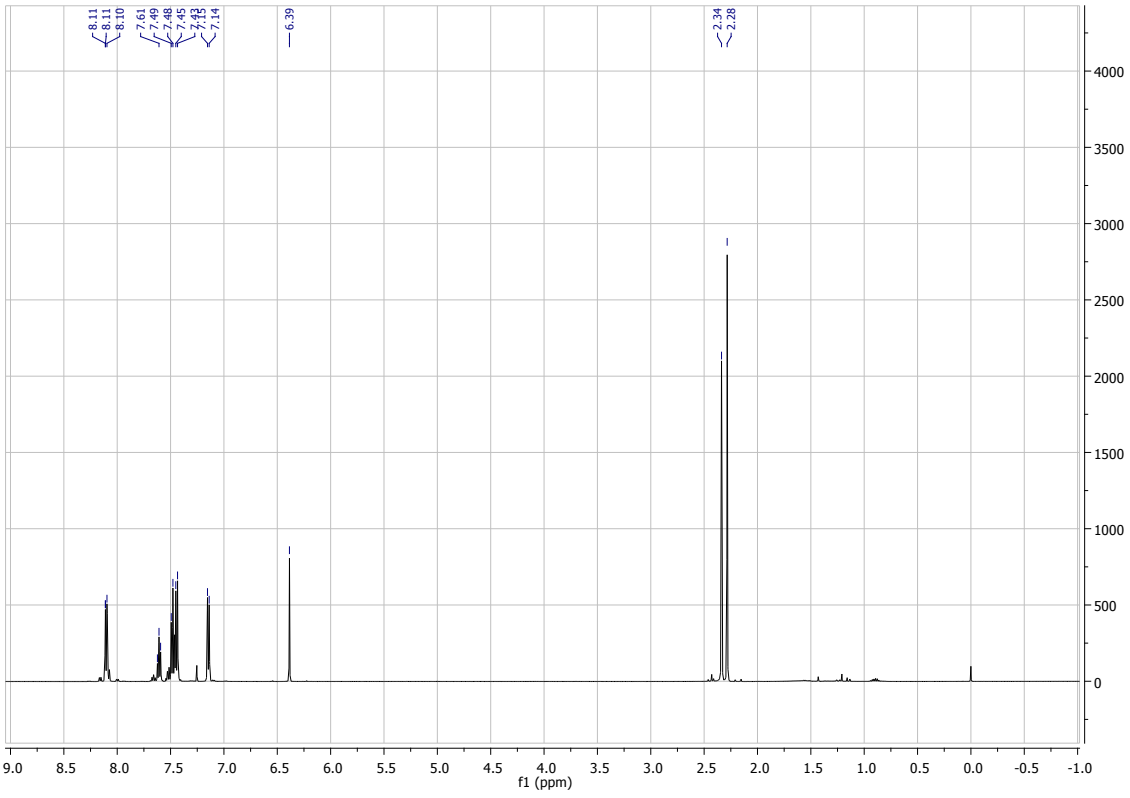
O
O

3b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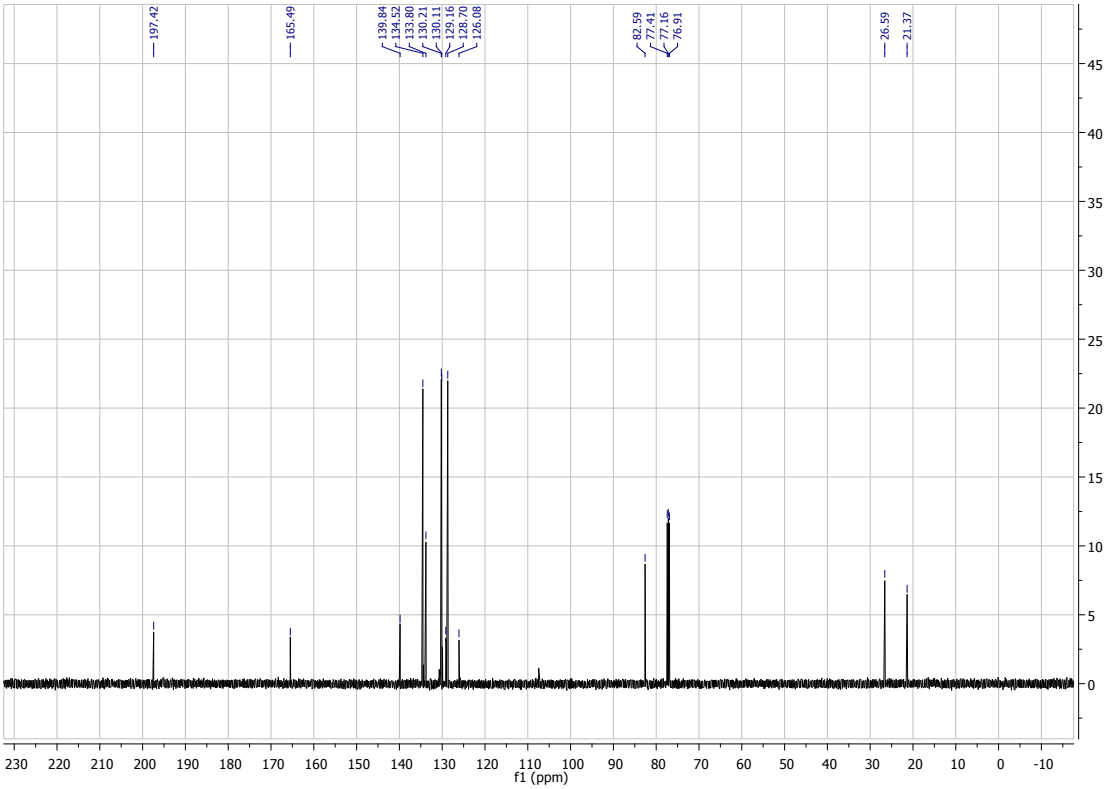
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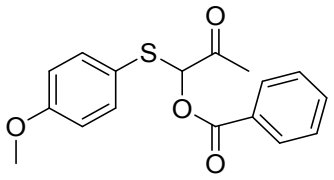
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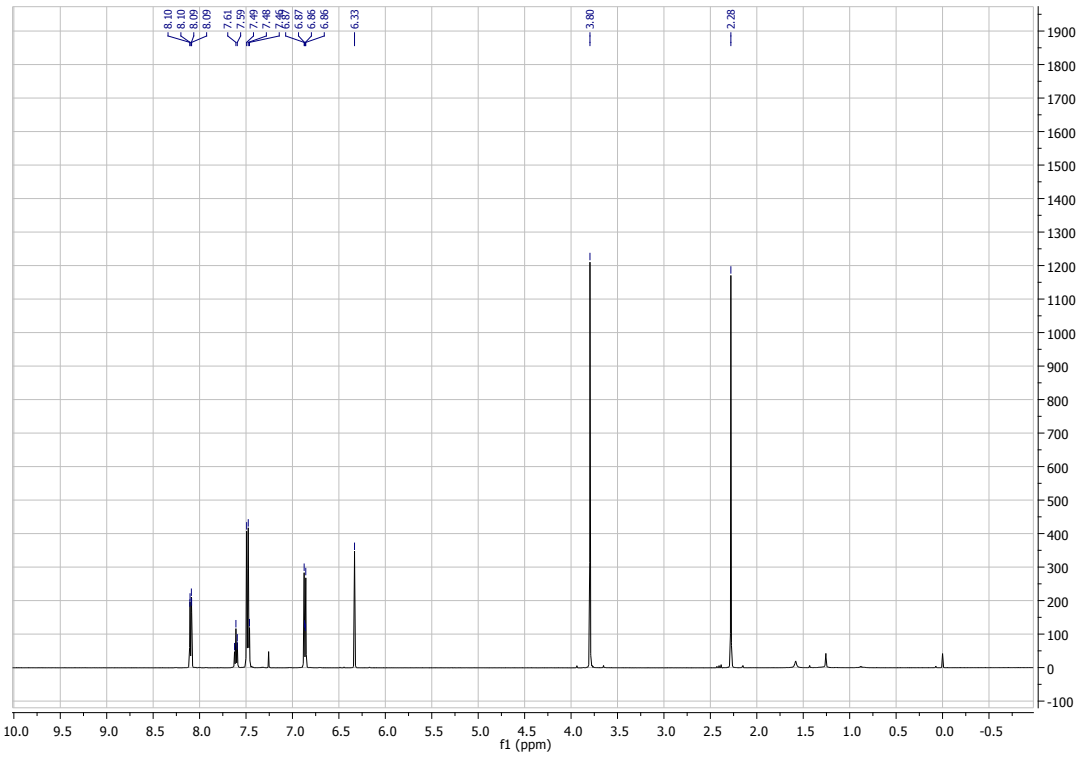
S
O
O
O

3c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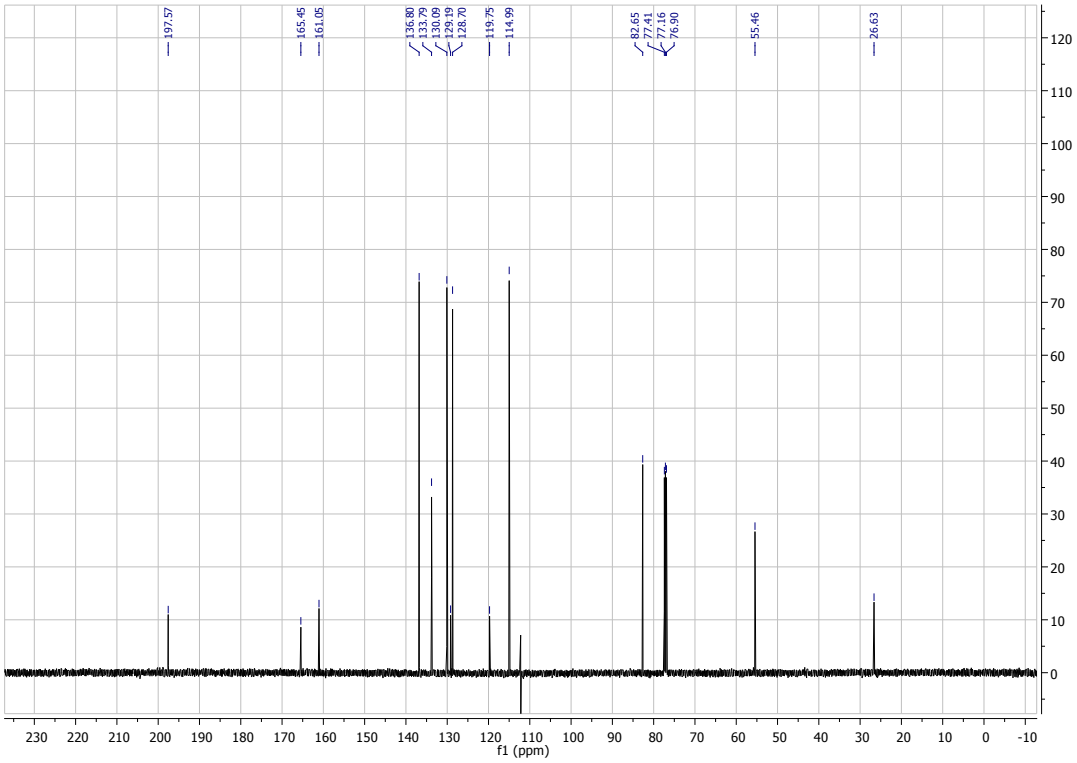
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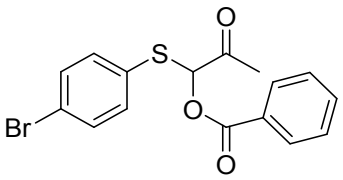
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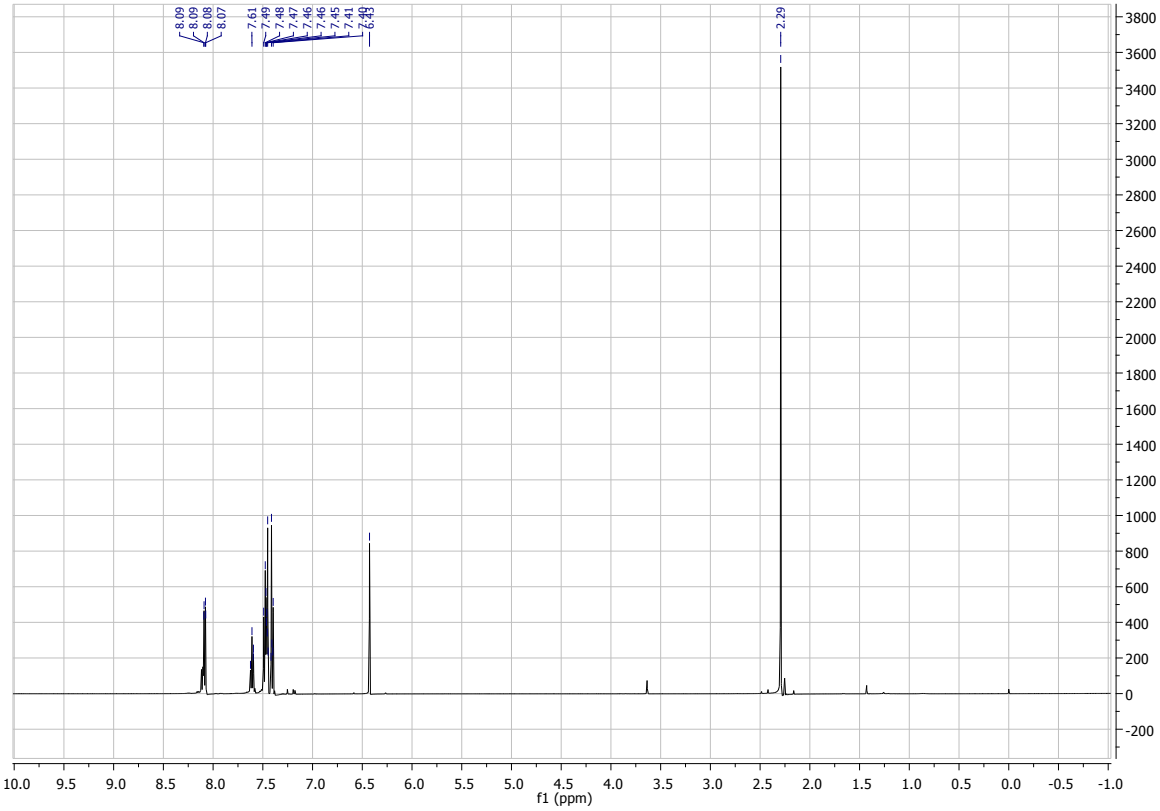
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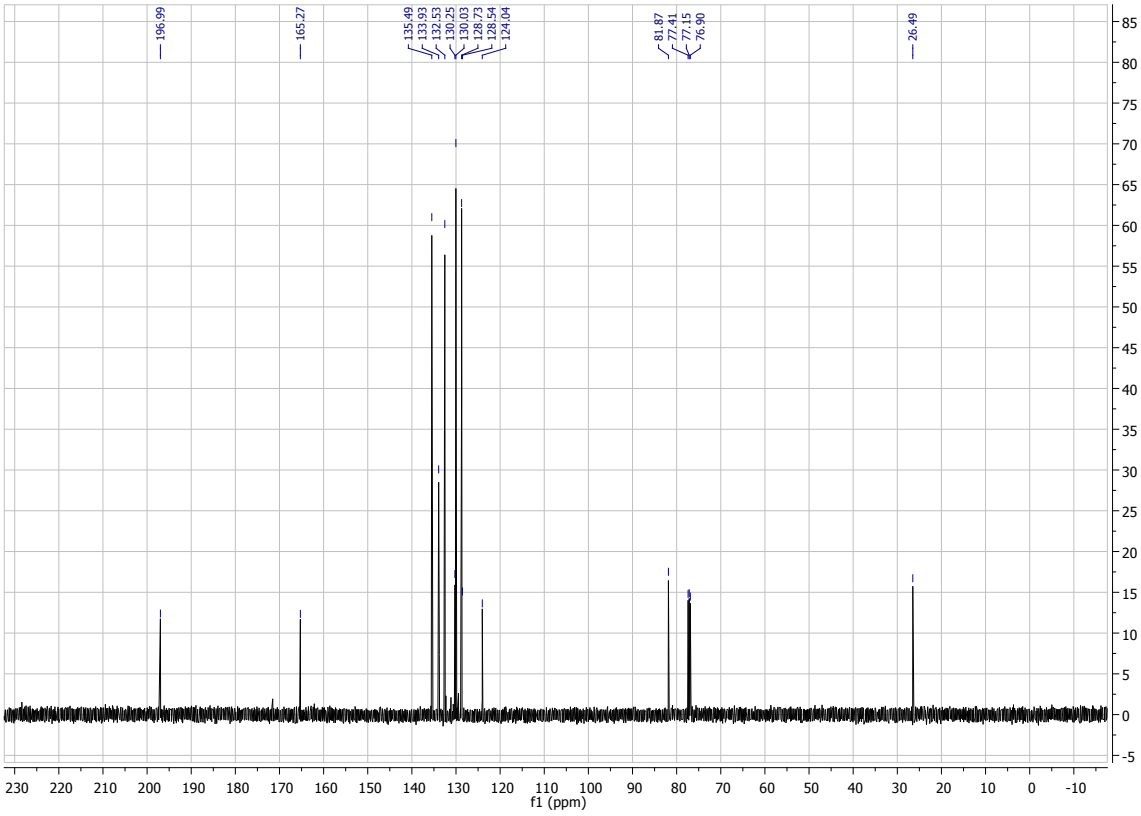
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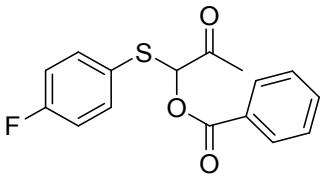
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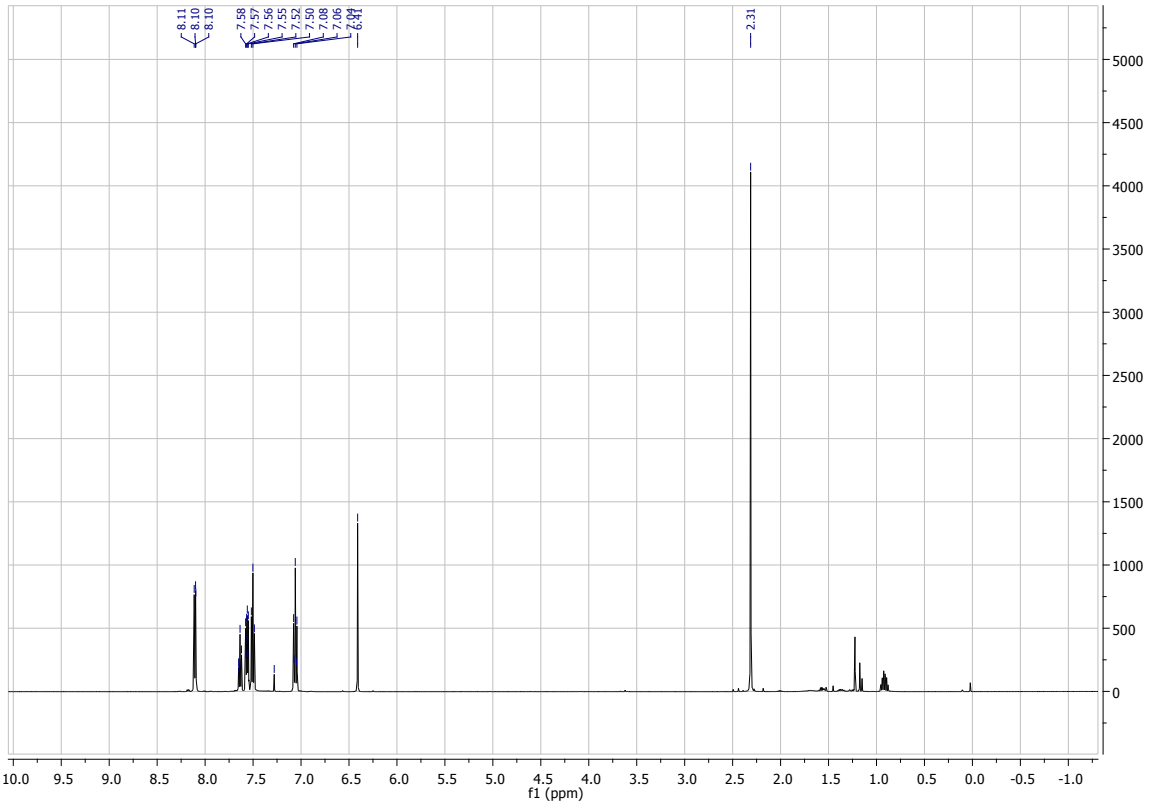
F
O
O

3e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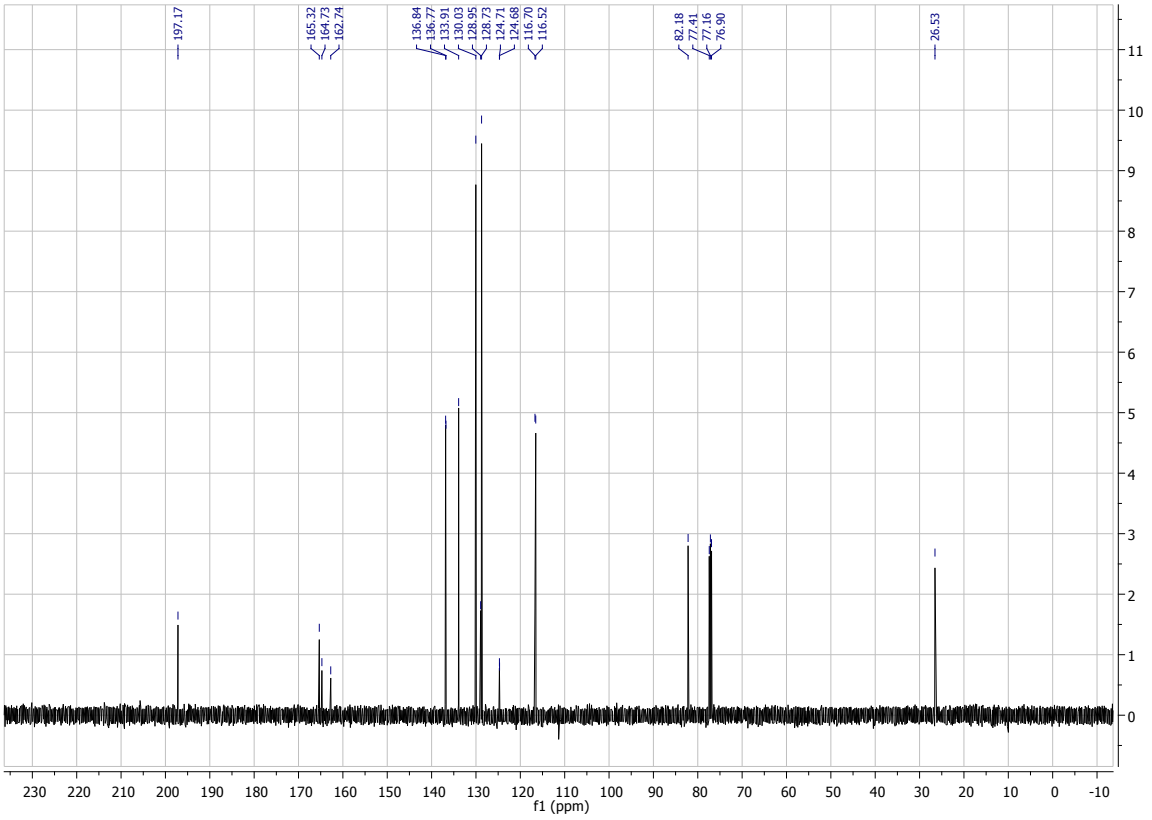
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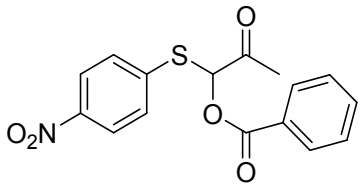
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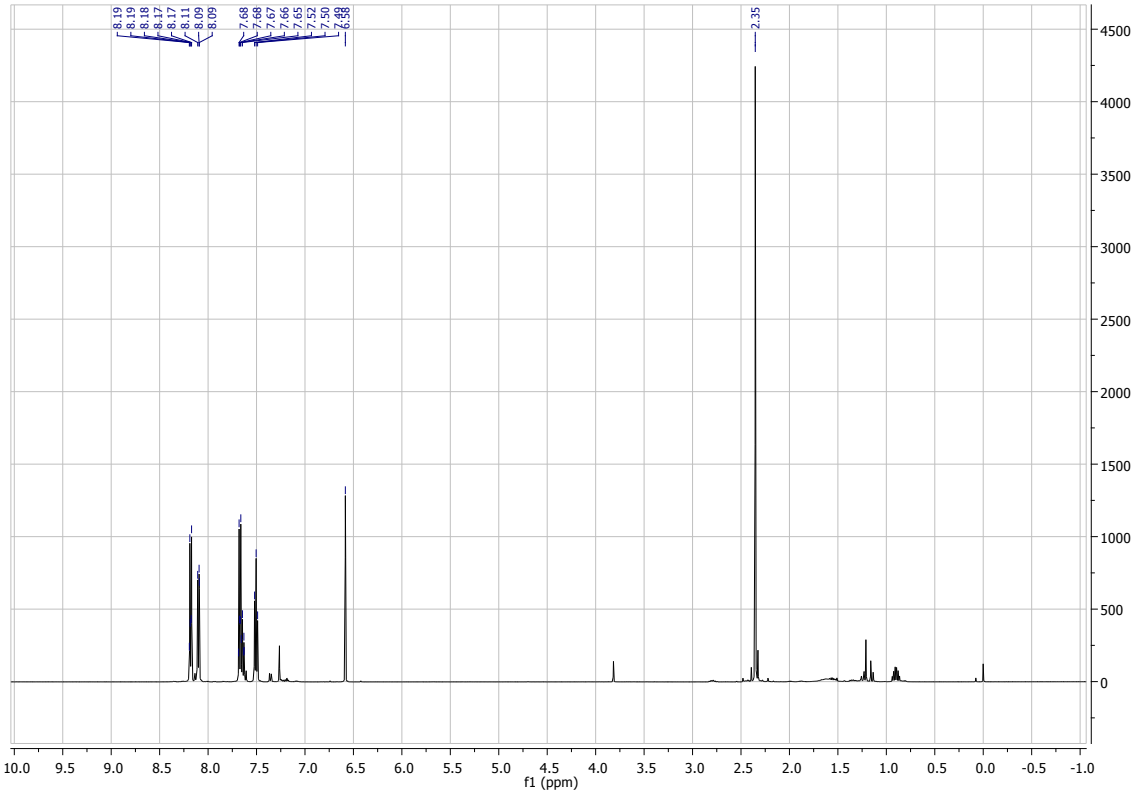
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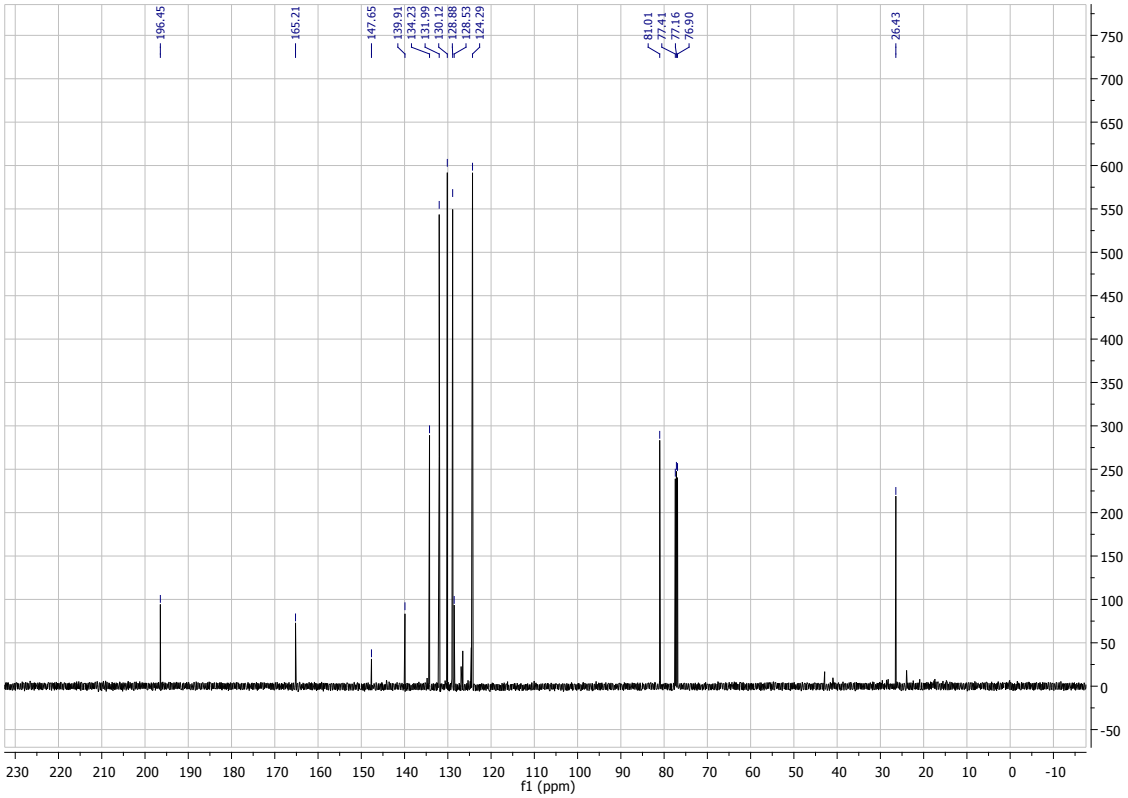
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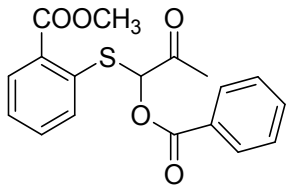
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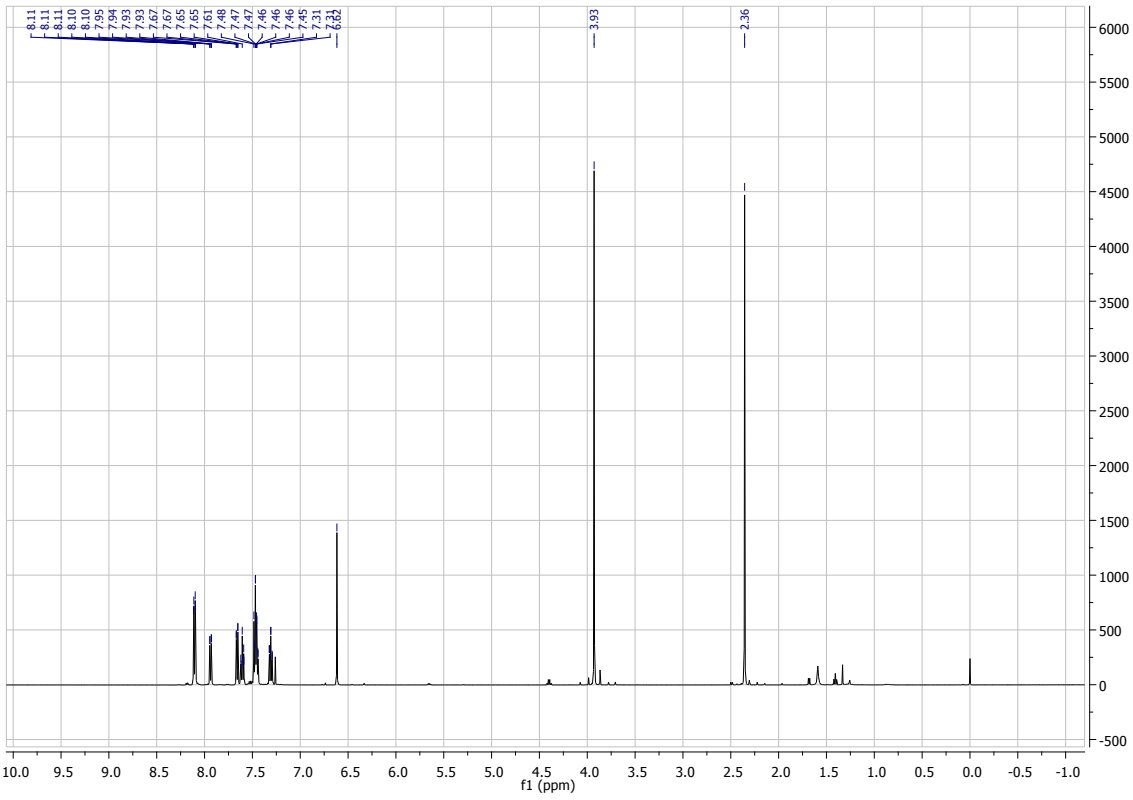
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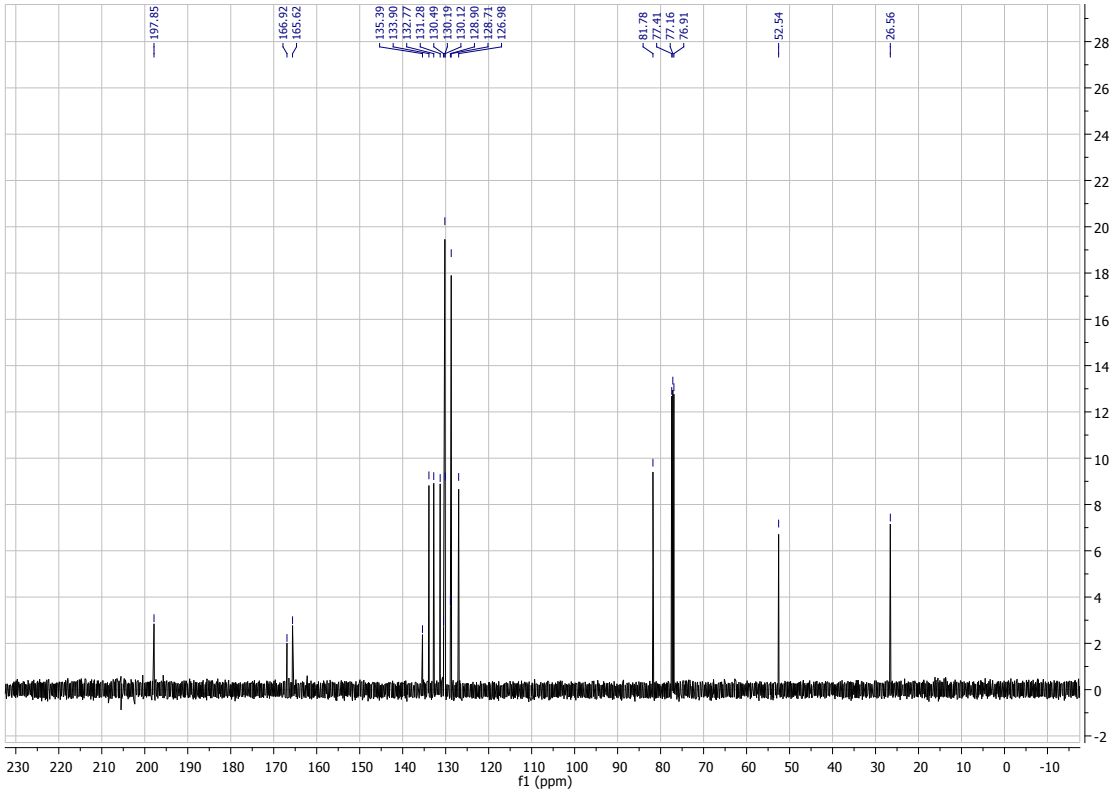
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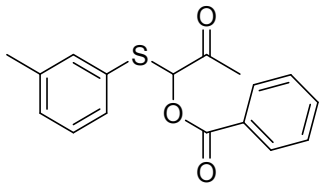
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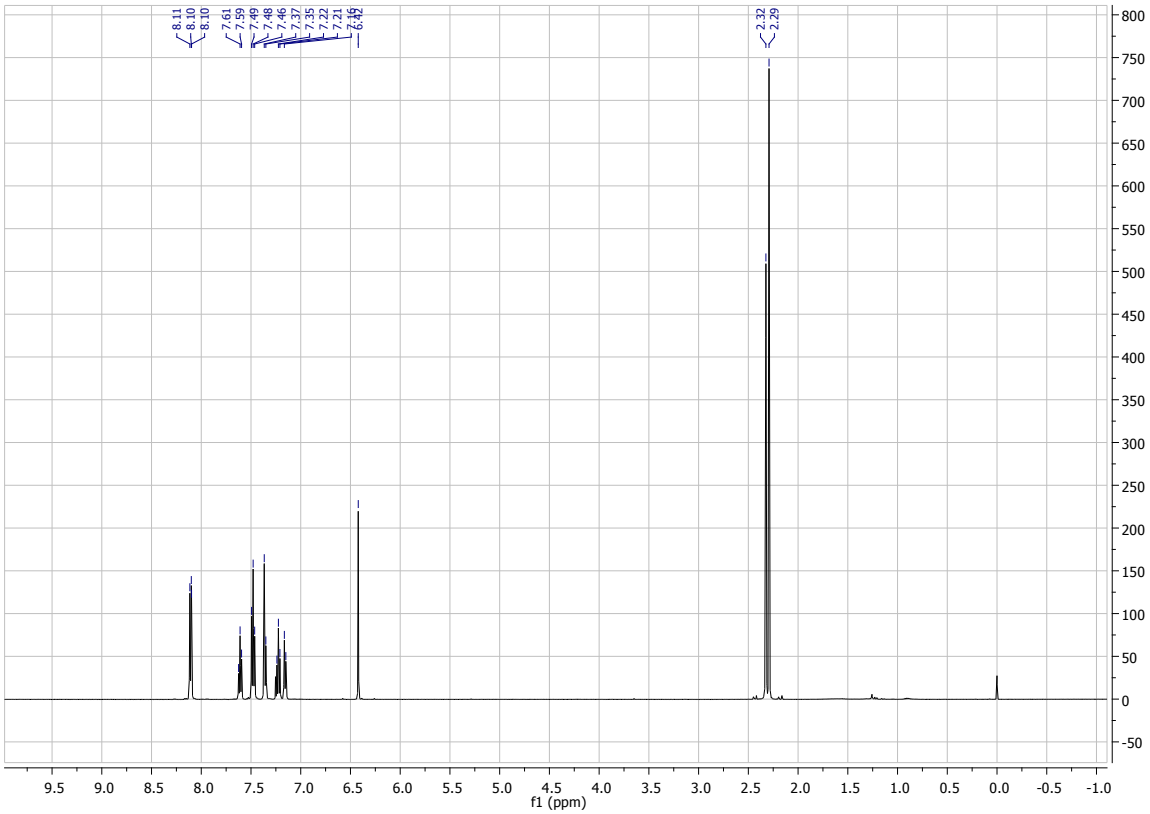
3h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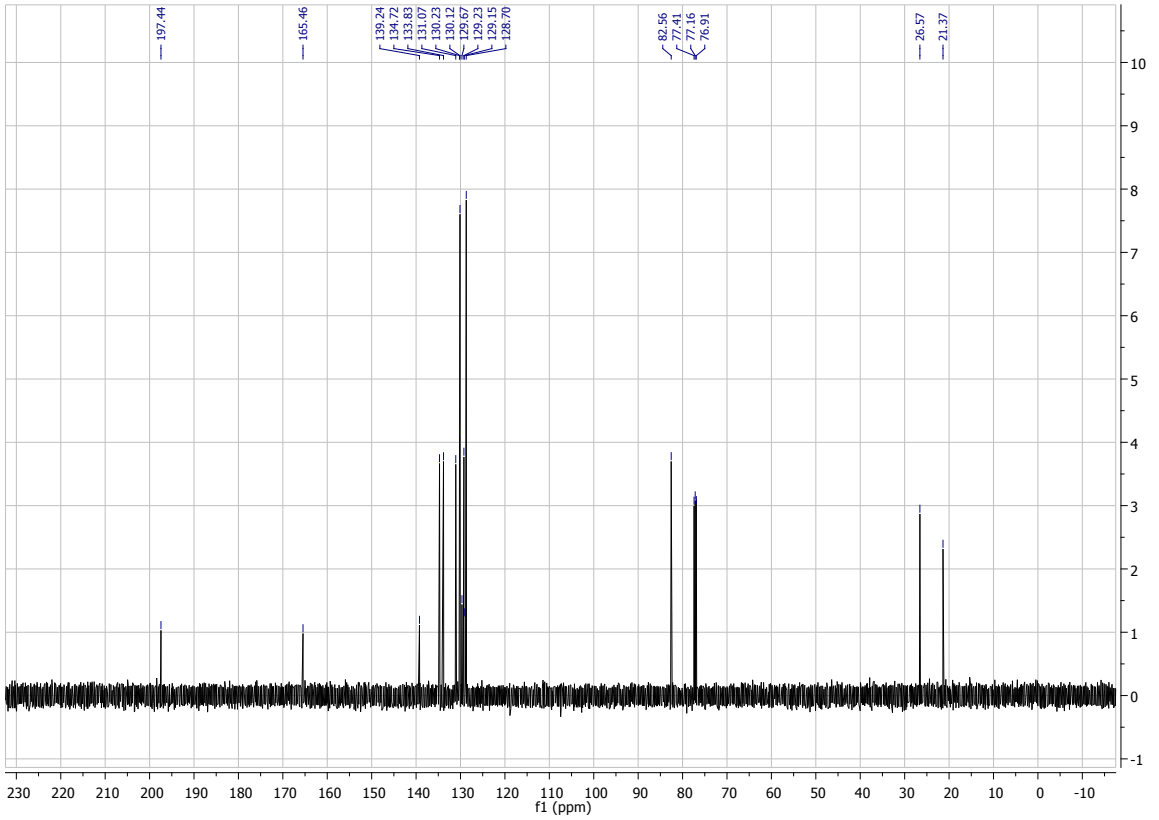
2.29
f1 (ppm)


197.44
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134.72
133.83
131.07
130.23
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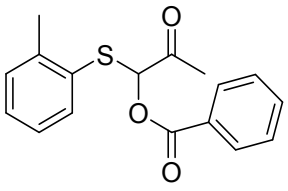
129.15
128.70
82.56
77.41
77.16
76.91
26.57
21.37
f1 (ppm)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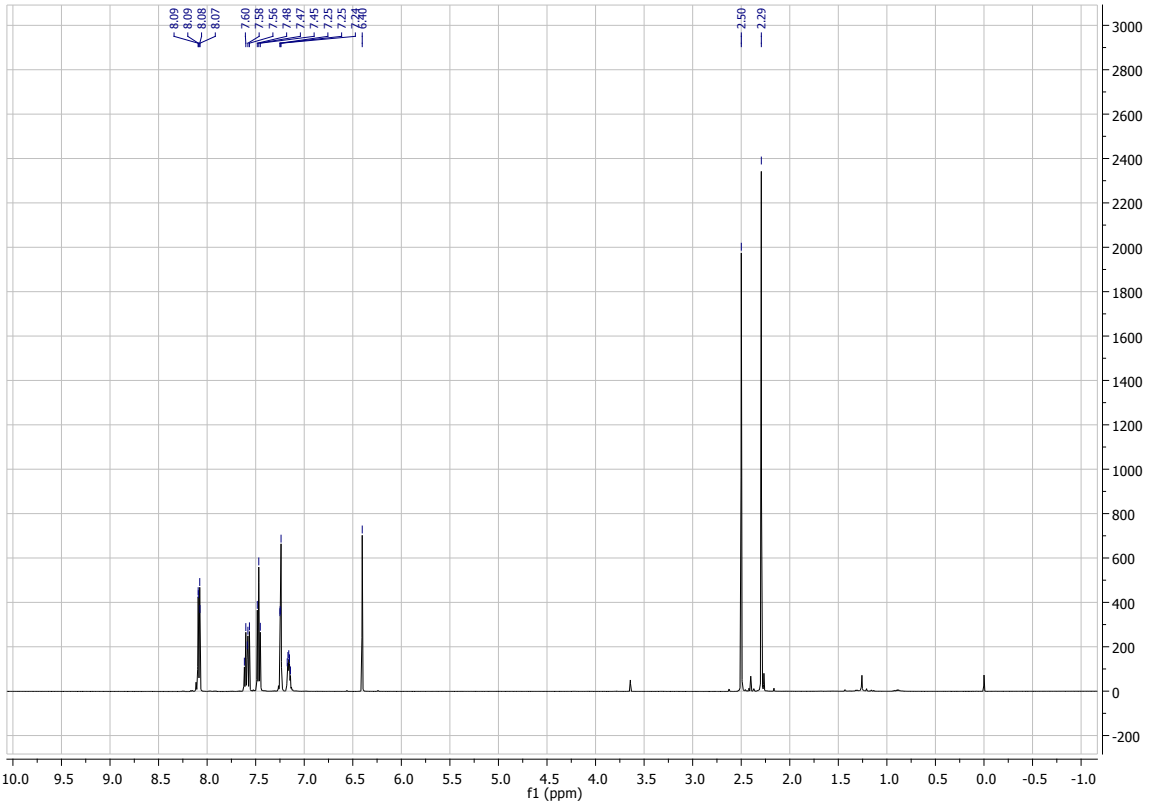
3i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8.07
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7.45
7.25
7.25
7.24
6.40
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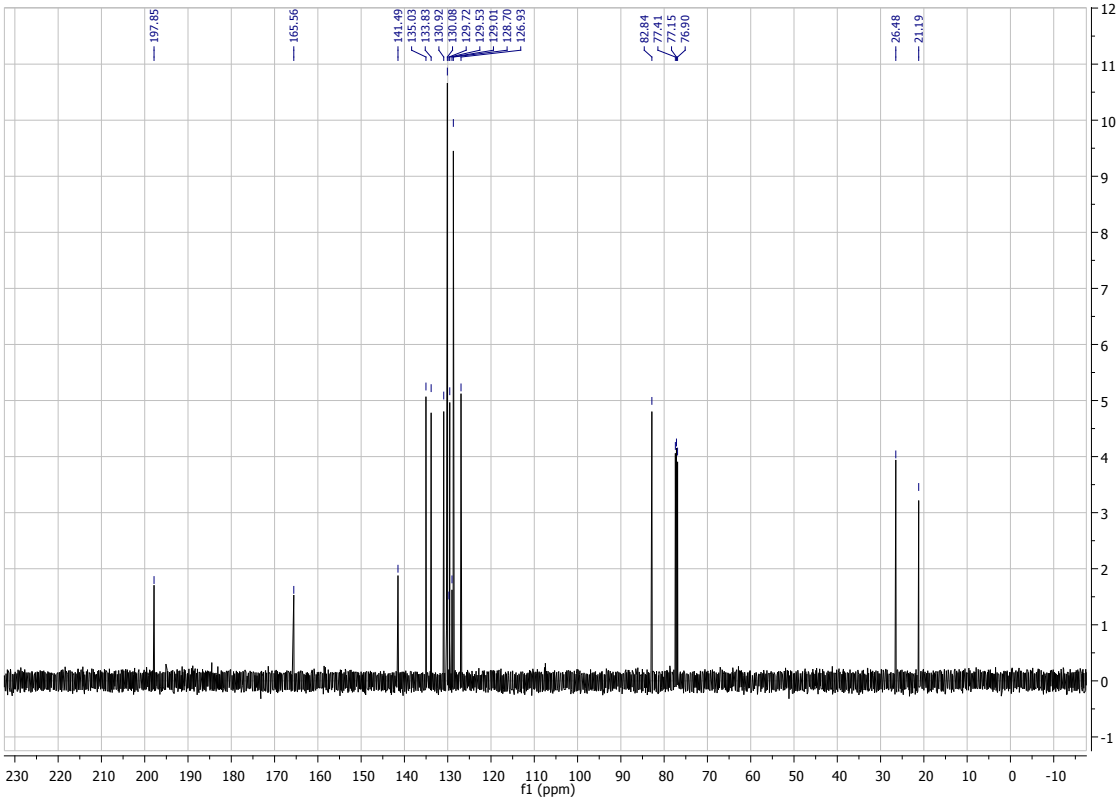
2.29
f1 (ppm)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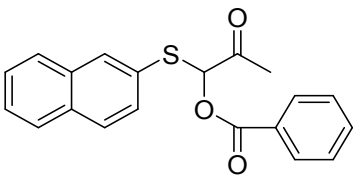
76.90
26.48
21.19
f1 (ppm)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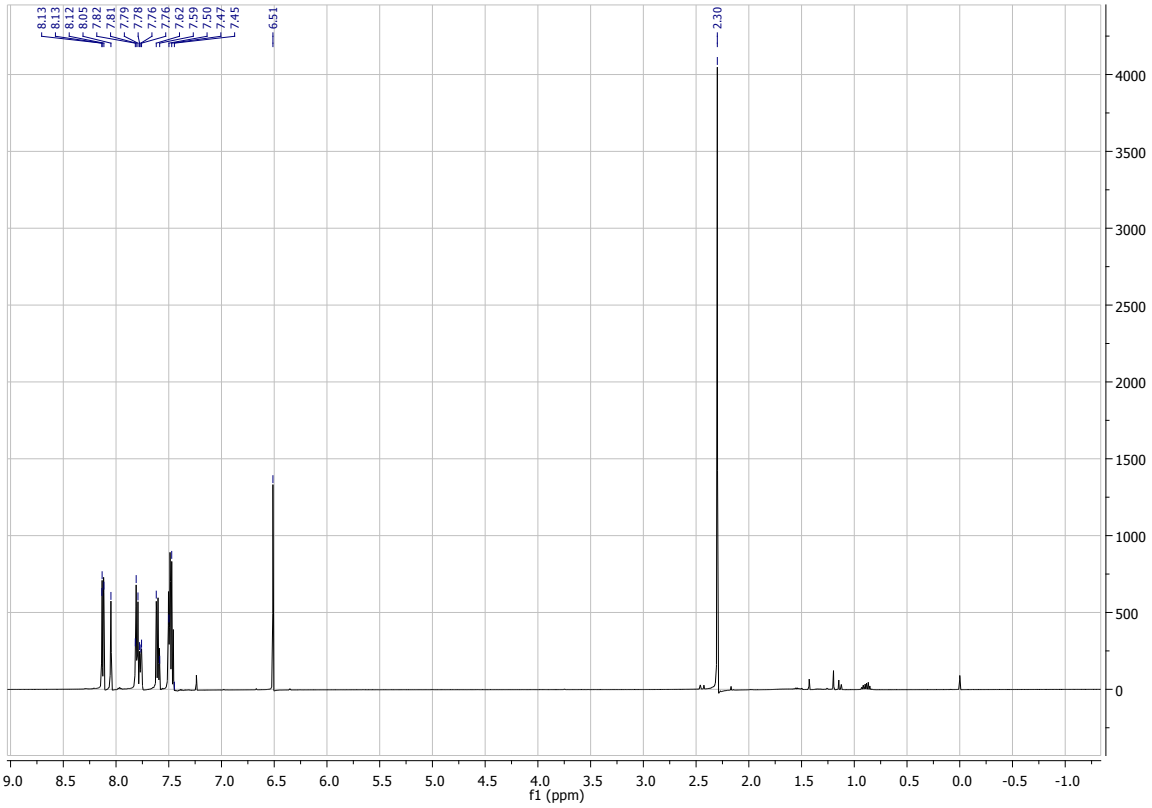
O

3j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8.13
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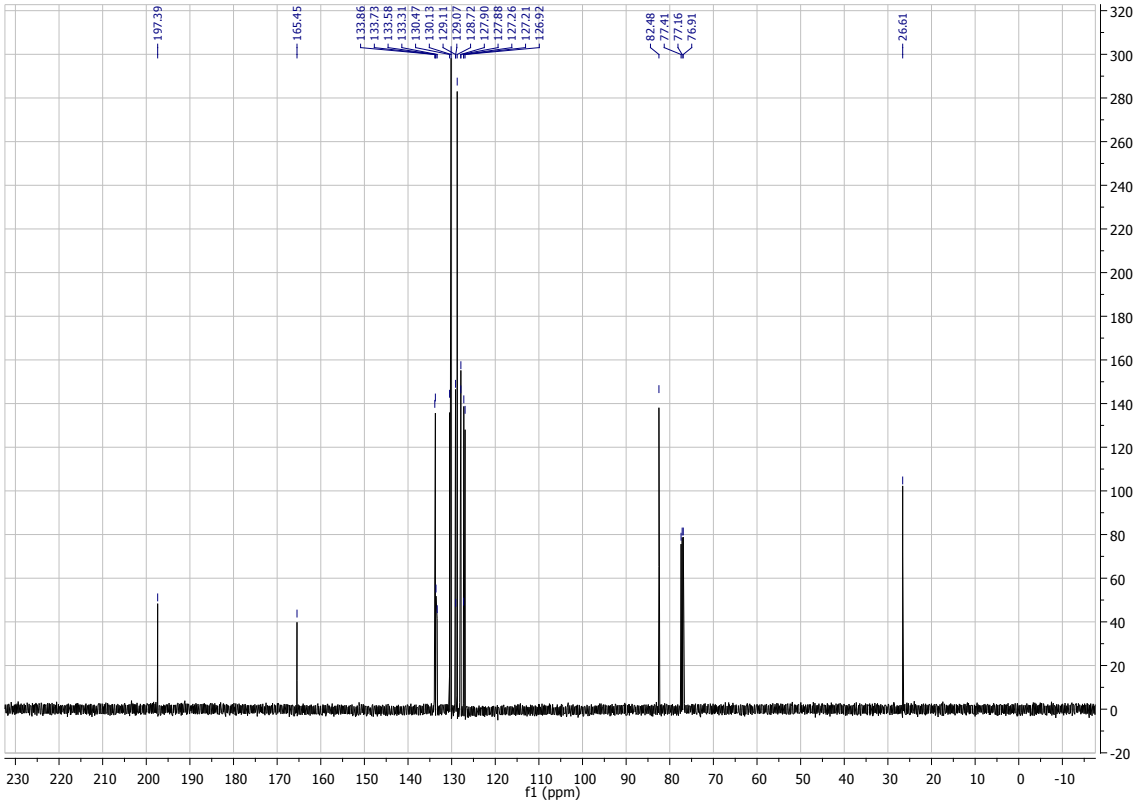
2.30
f1 (ppm)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82.48
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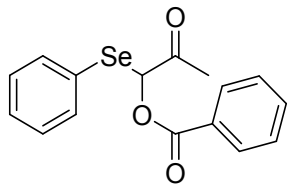
77.16
76.91
26.61
f1 (ppm)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Se
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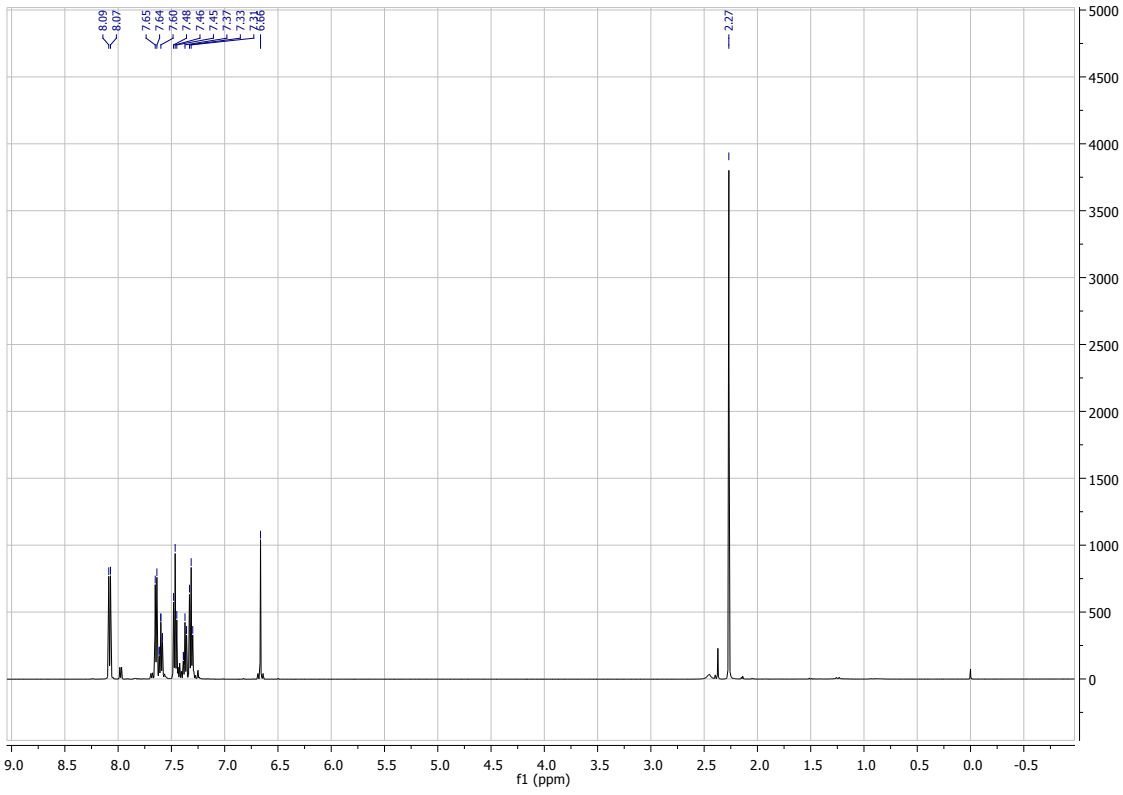
3k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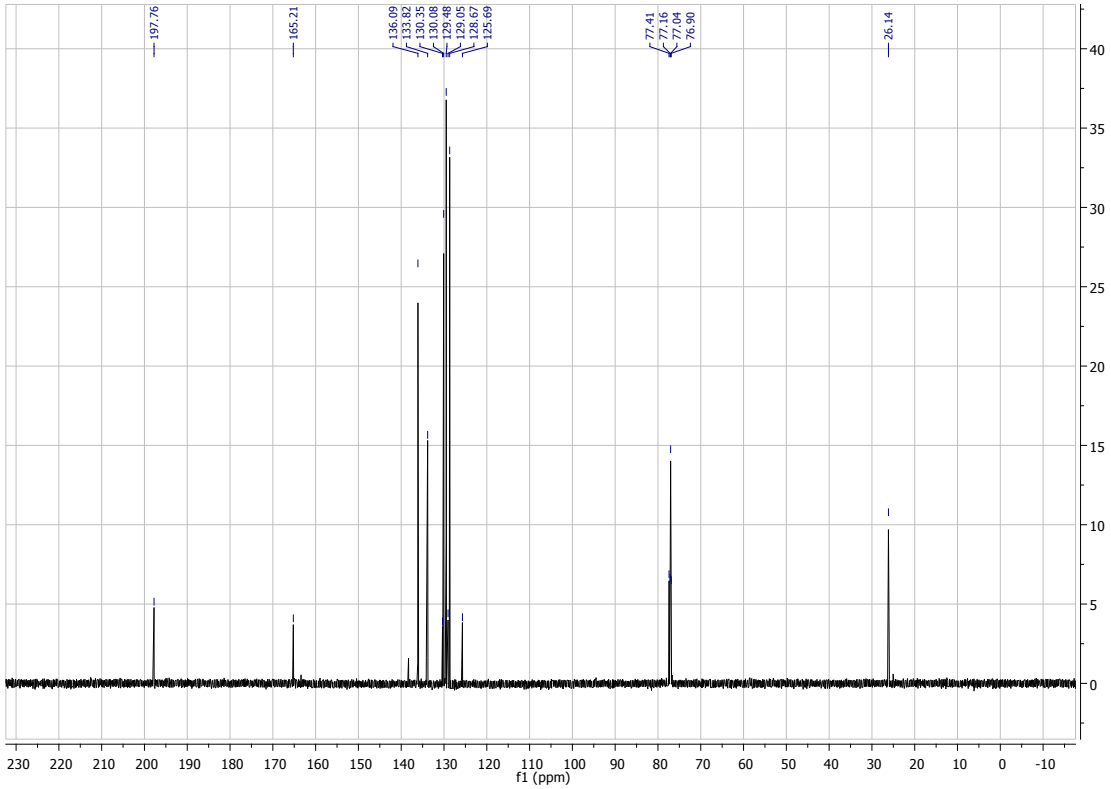
7.45
7.37
7.33
6.86
2.27
f1 (ppm)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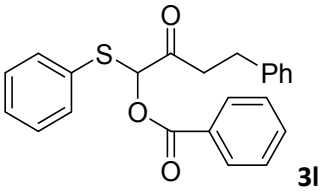
26.14
f1 (ppm)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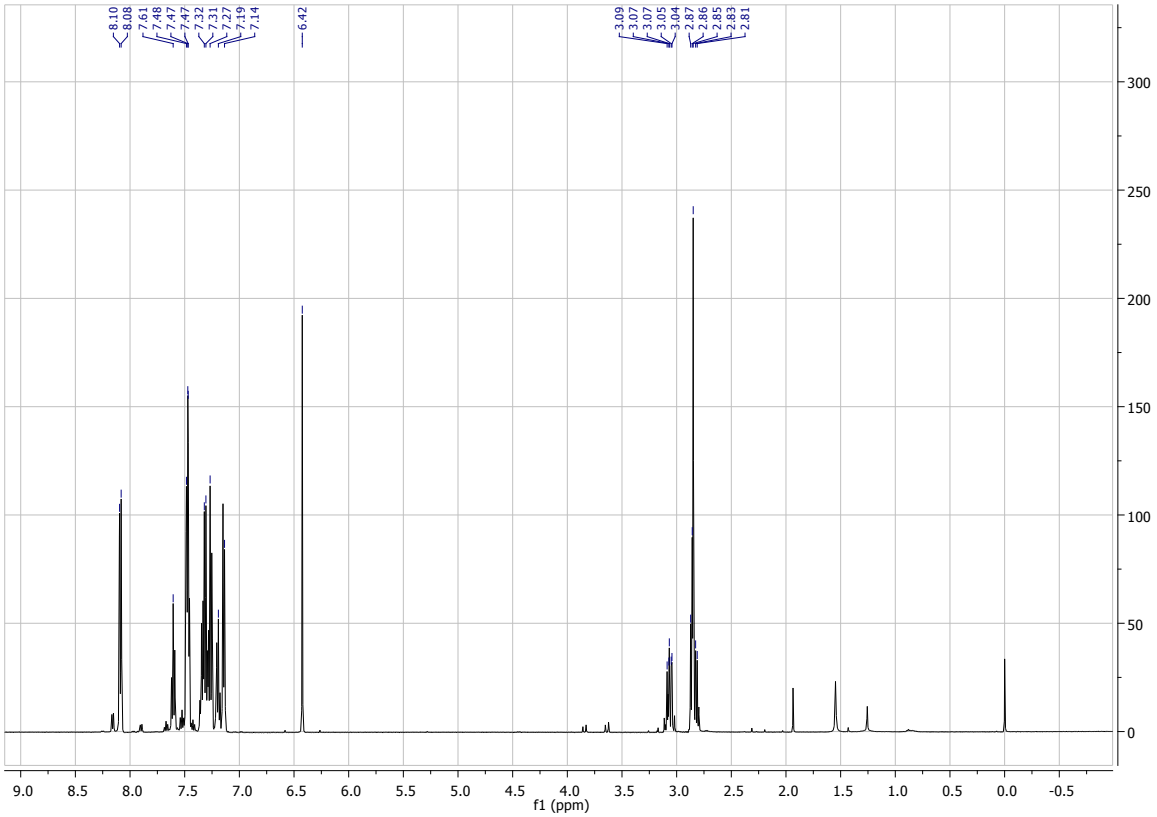
3l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7.47
7.32
7.31
7.27
7.19
7.14
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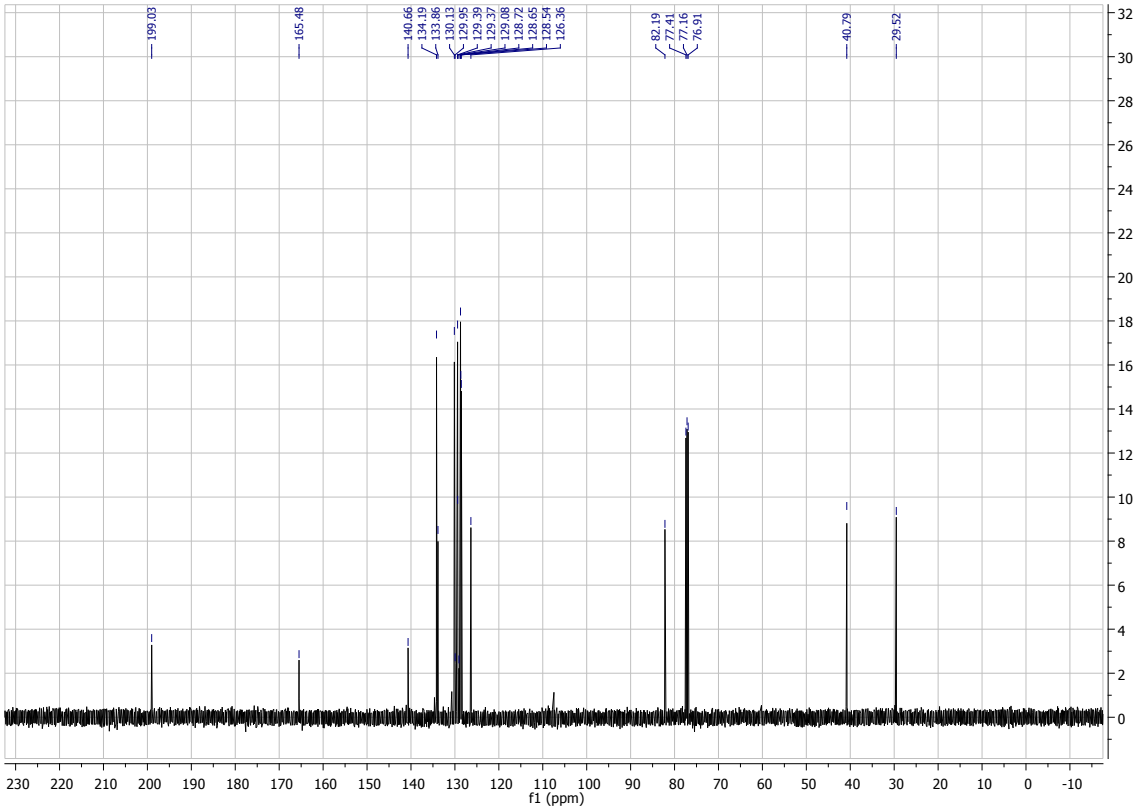
3.07
3.07
3.05
3.04
2.87
2.86
2.85
2.83
2.81
f1 (ppm)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129.39
129.37
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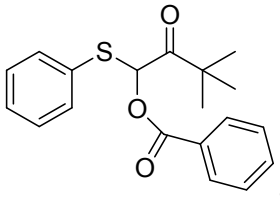
40.79
29.52
f1 (ppm)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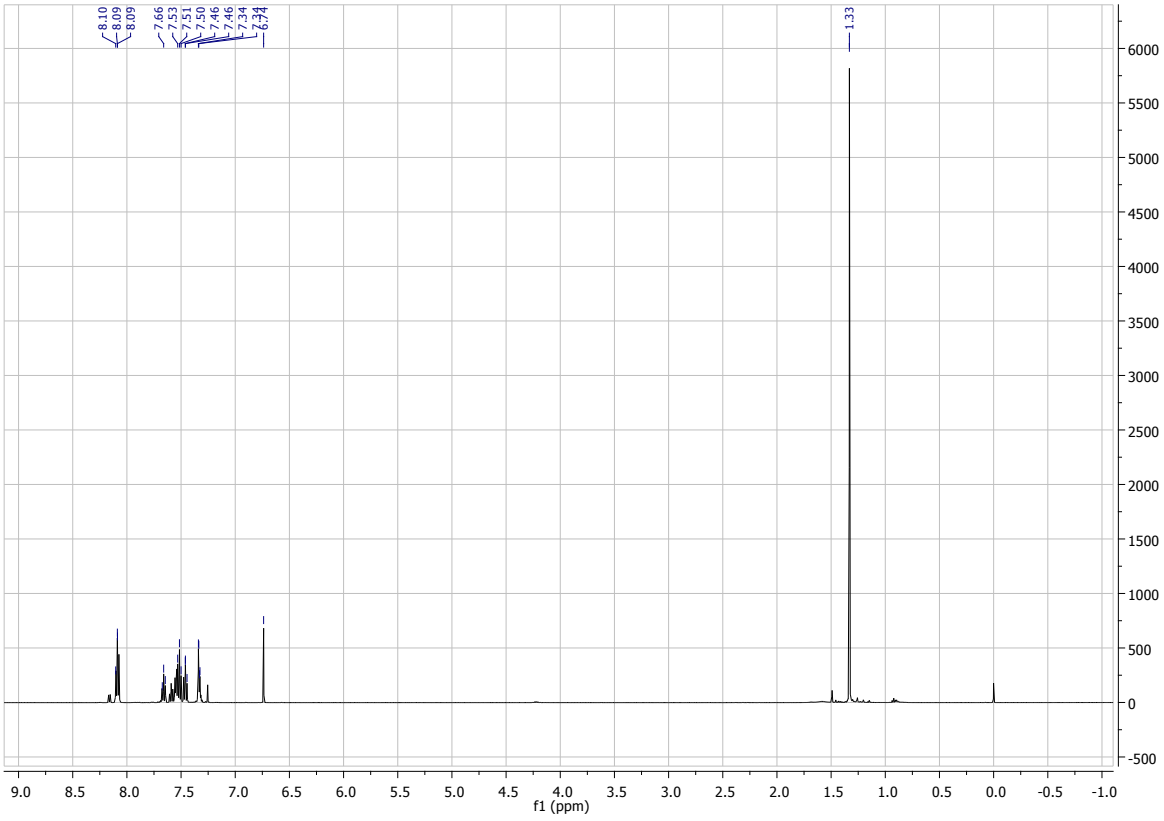
O
O

3m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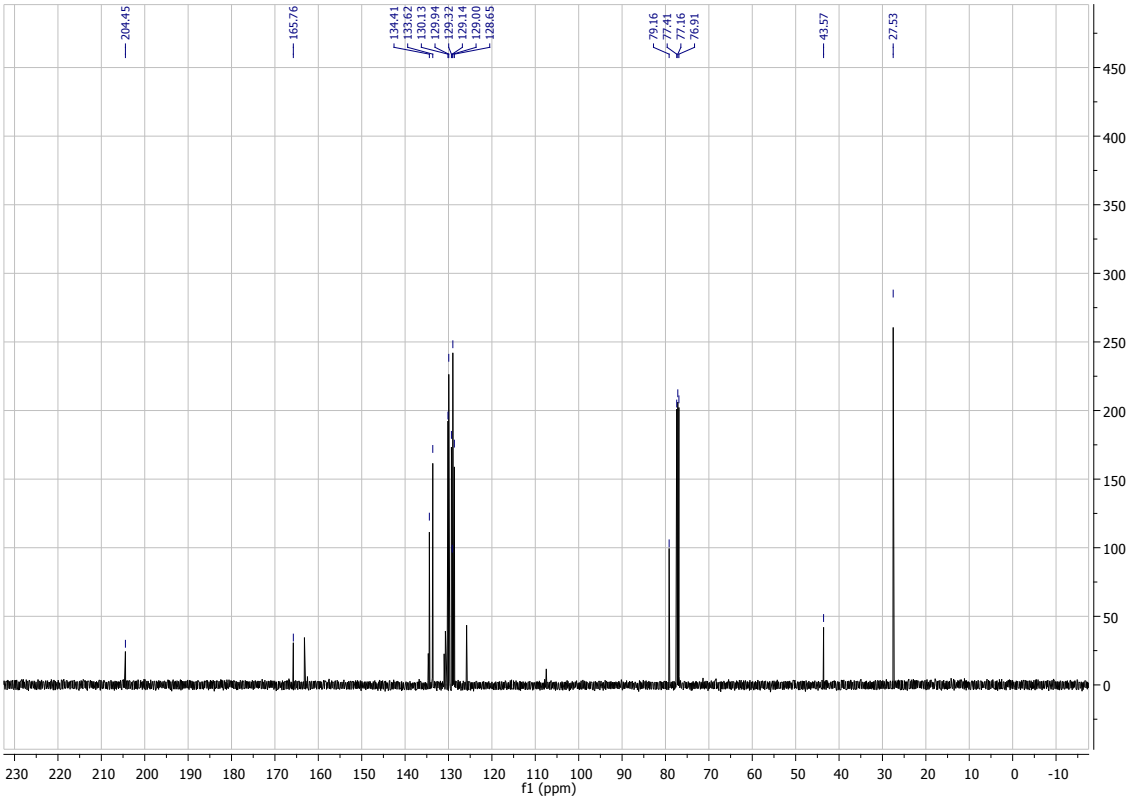
7.50
7.46
7.46
7.34
7.34
1.33
f1 (ppm)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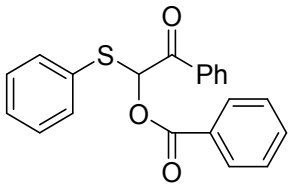
79.16
77.41
77.16
76.91
43.57
27.53
f1 (ppm)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Ph
O
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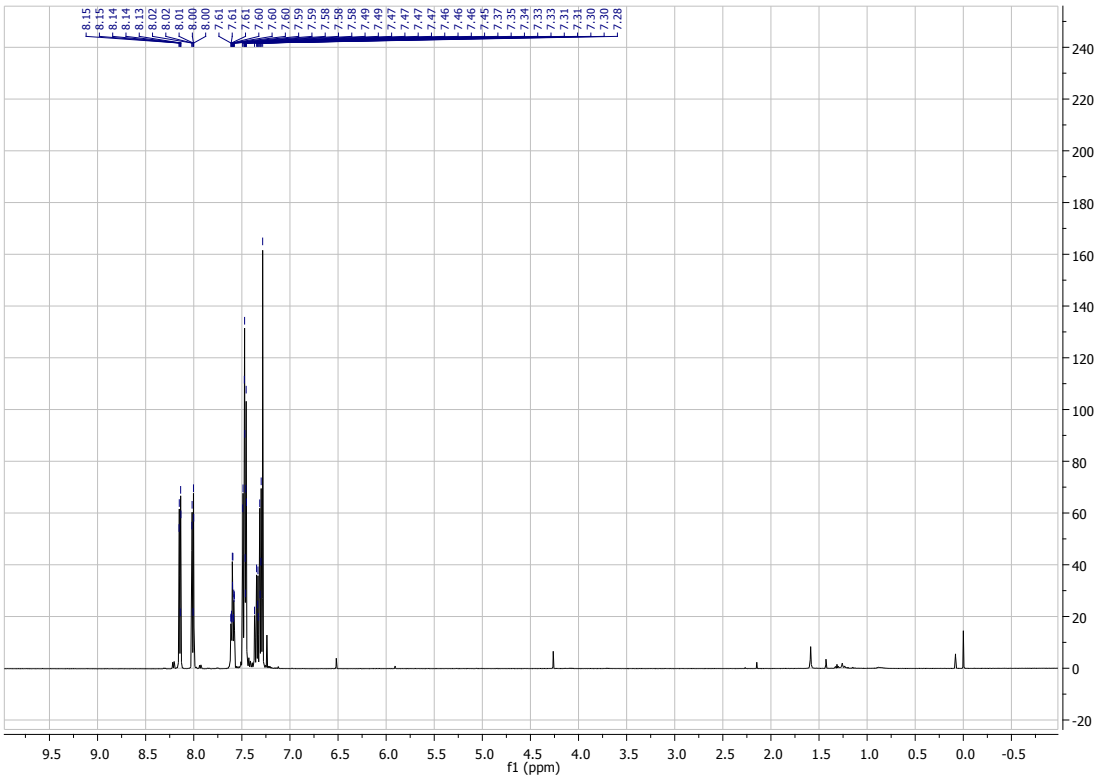
3n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8.01
8.00
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7.60
7.59
7.58
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7.46
7.45
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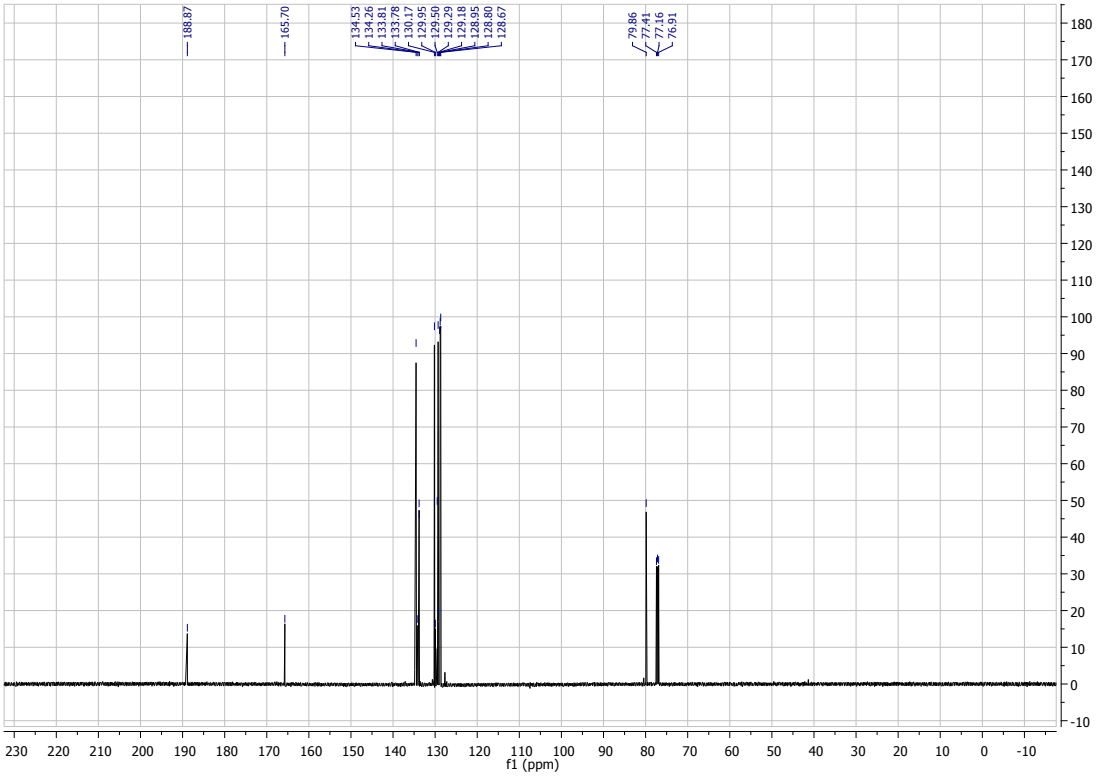
7.35
7.34
7.33
7.31
7.30
7.28
f1 (ppm)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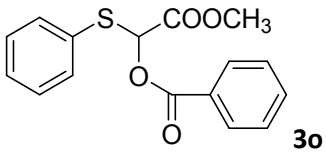
128.80
128.67
79.86
77.41
77.16
76.91
f1 (ppm)


S
COOCH3
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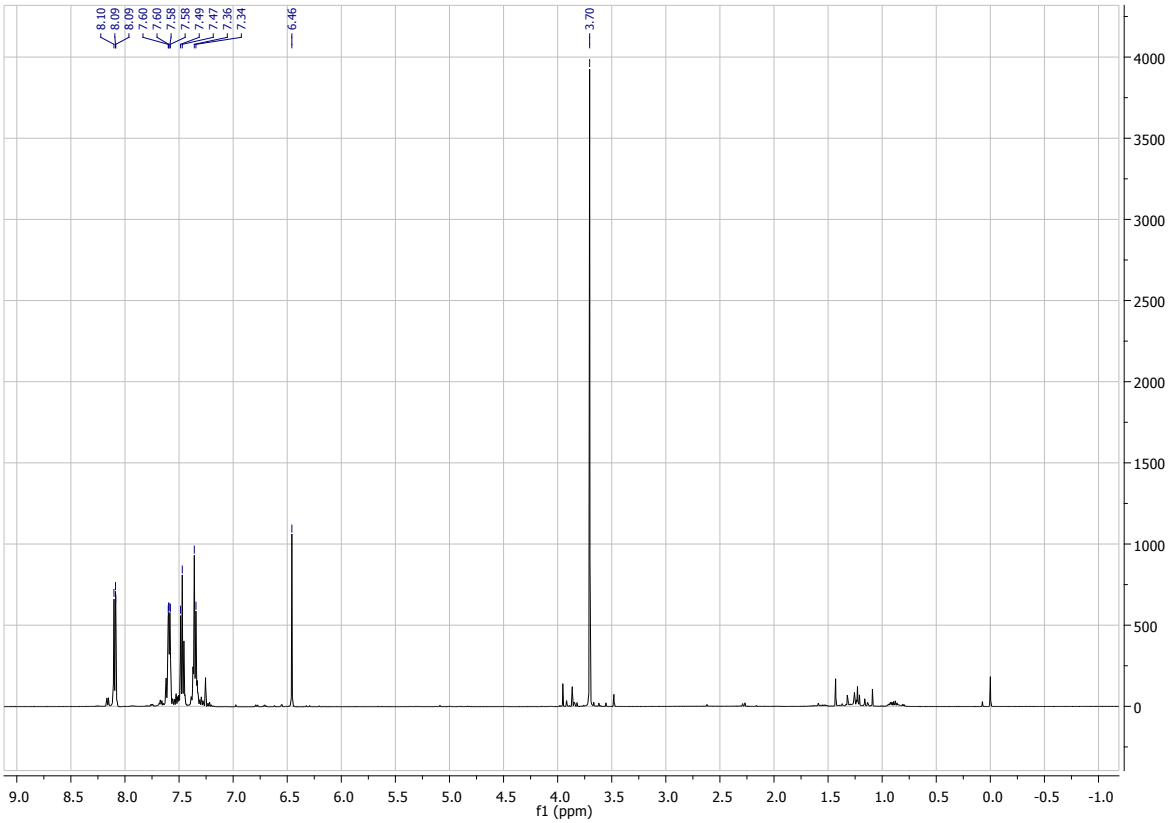
O

3o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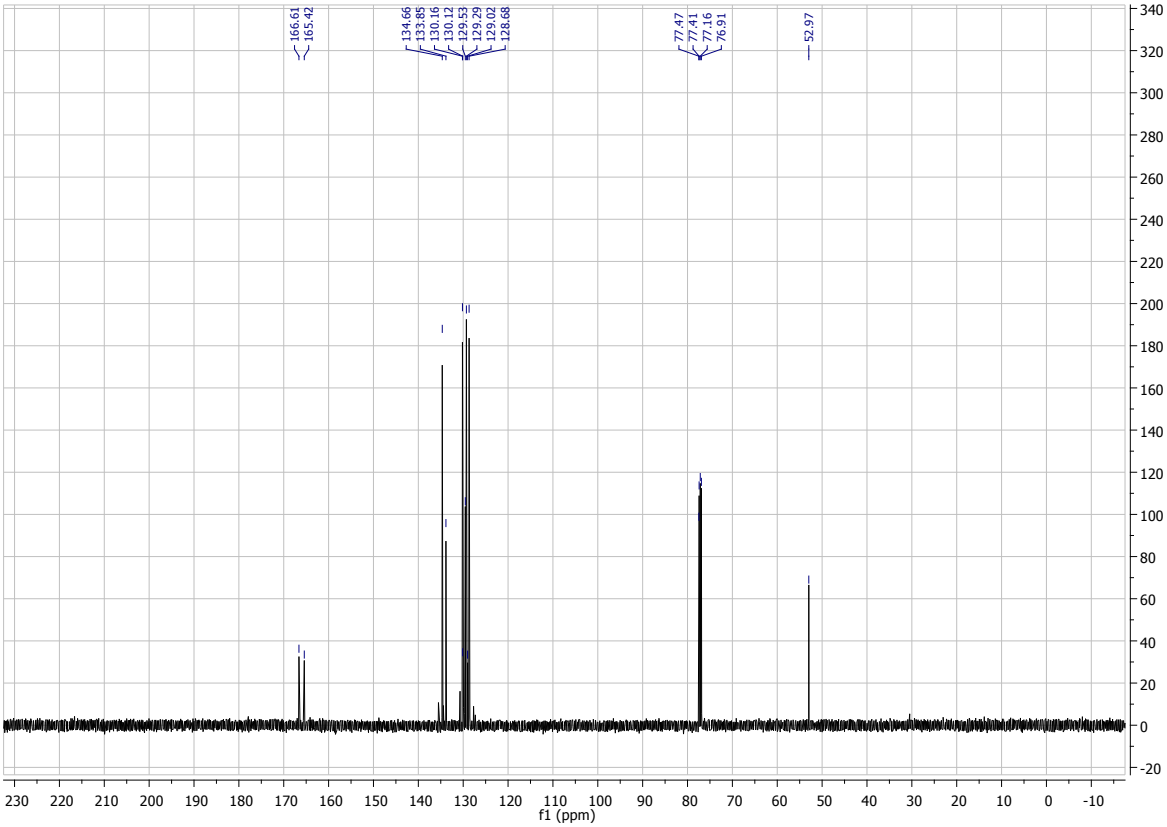
7.34
6.46
3.70
f1 (ppm)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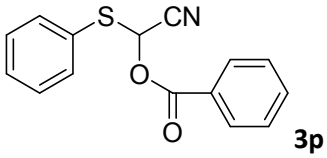
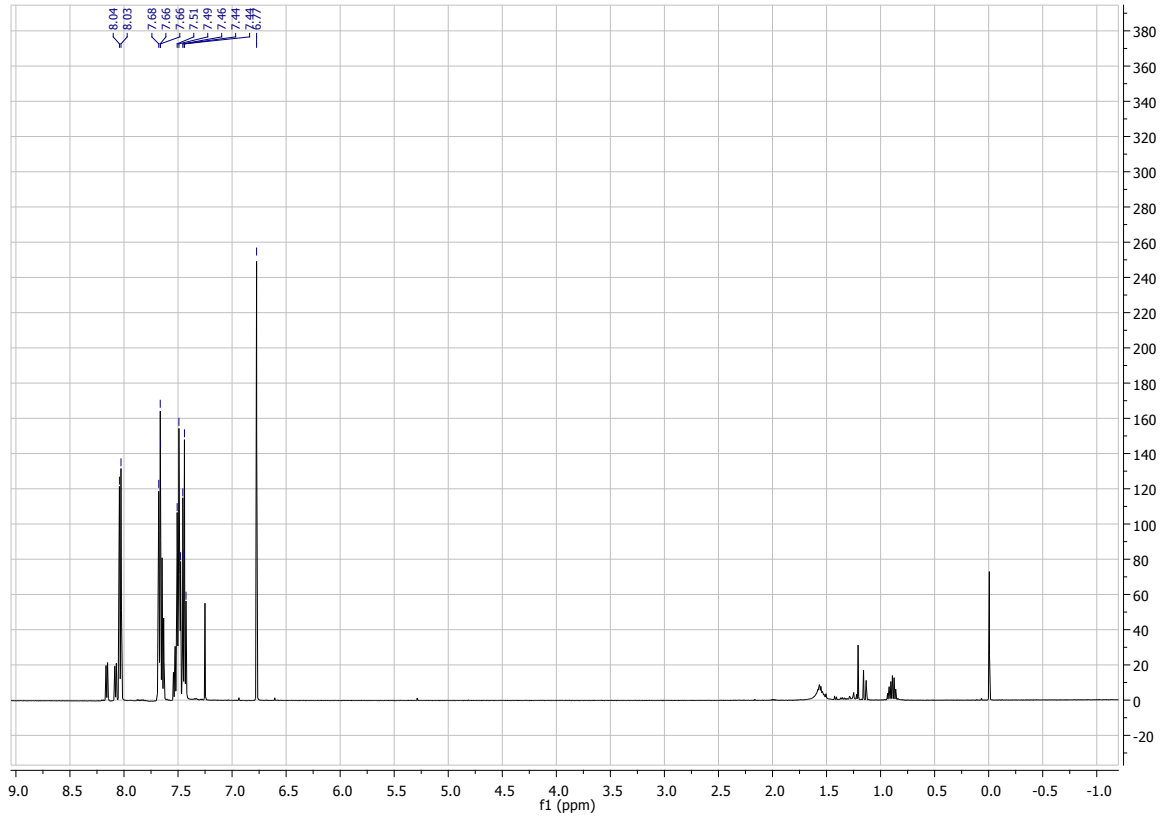
76.91
52.97
f1 (ppm)


S
CN
O
O

3p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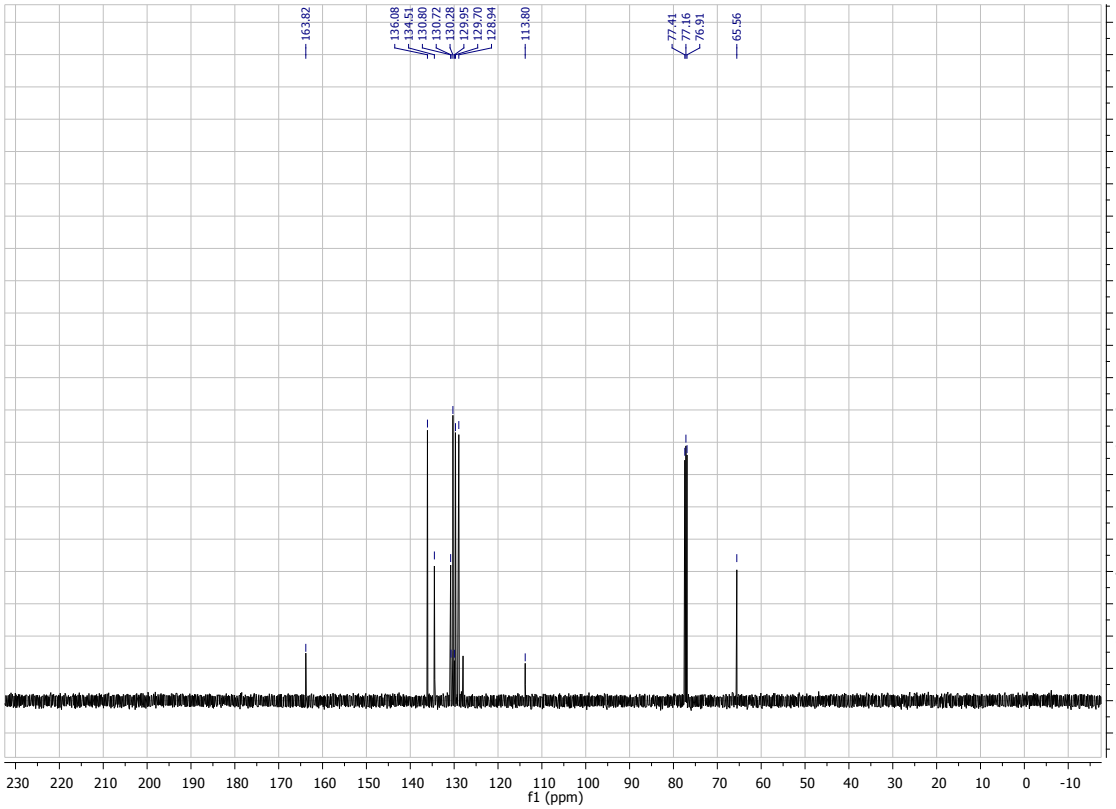
7.44
7.27
f1 (ppm)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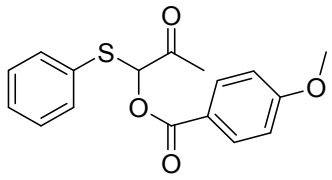
77.16
76.91
65.56
f1 (ppm)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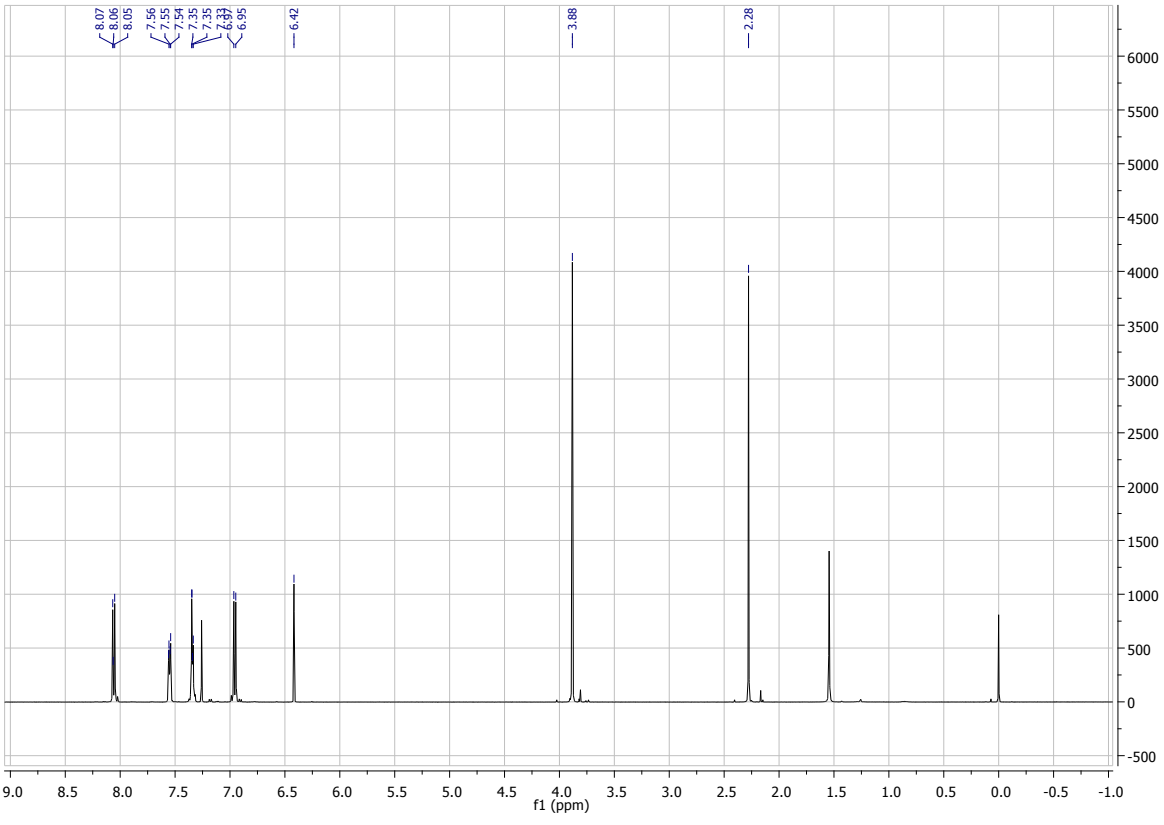
S
O
O
O

3q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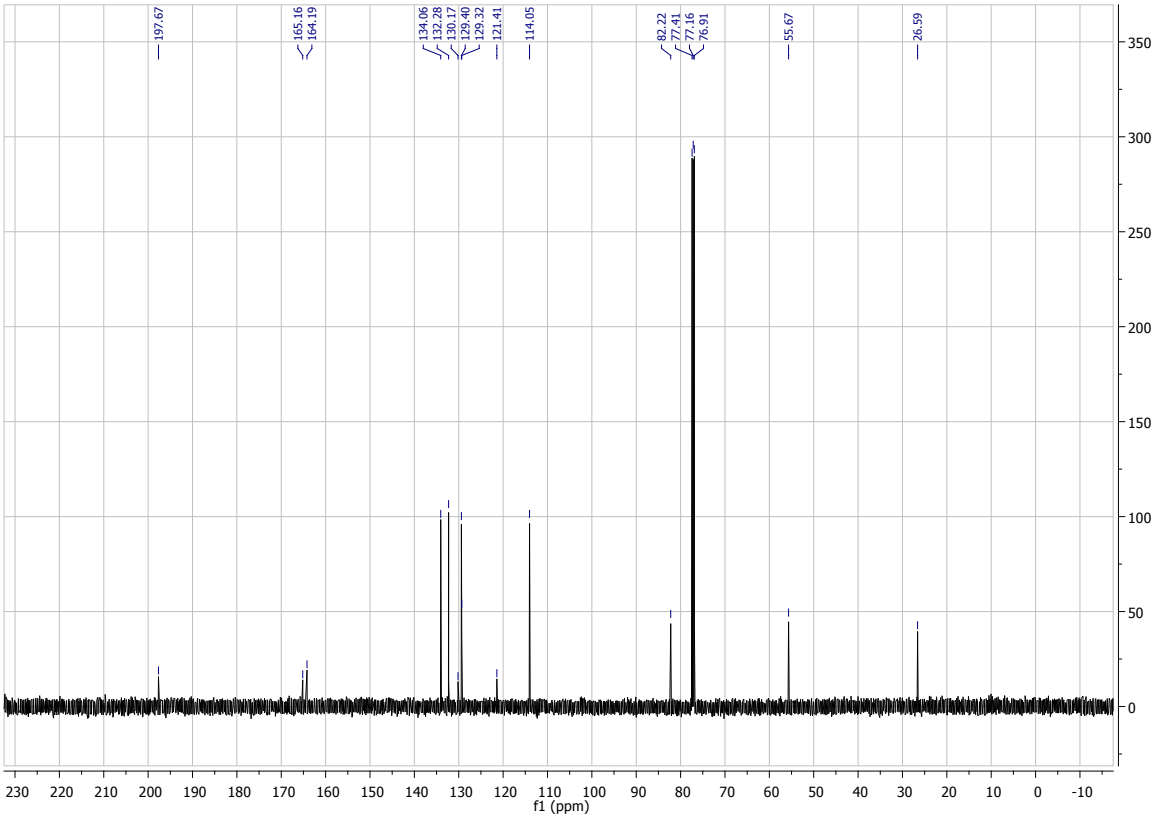
f1 (ppm)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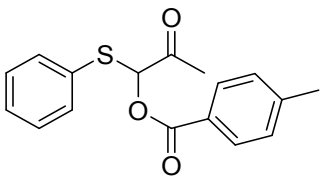
55.67
26.59
f1 (ppm)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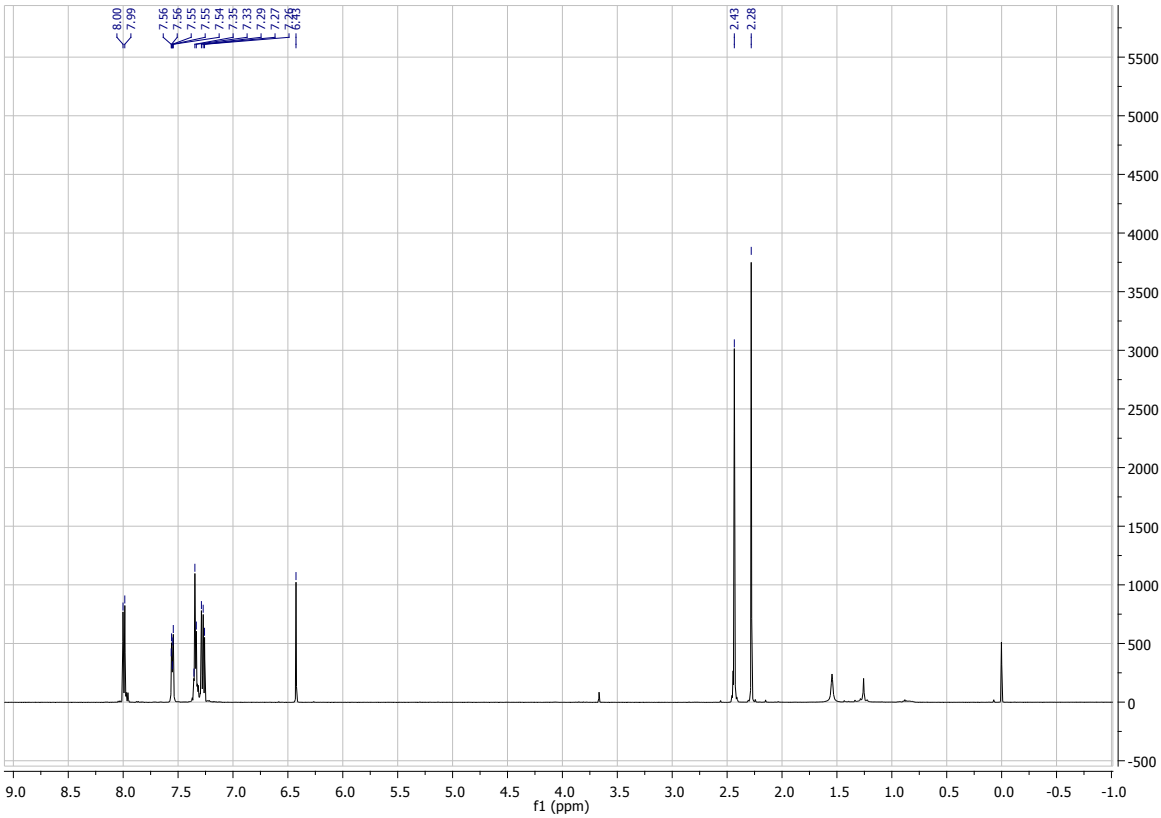
S
O
O

3r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7.29
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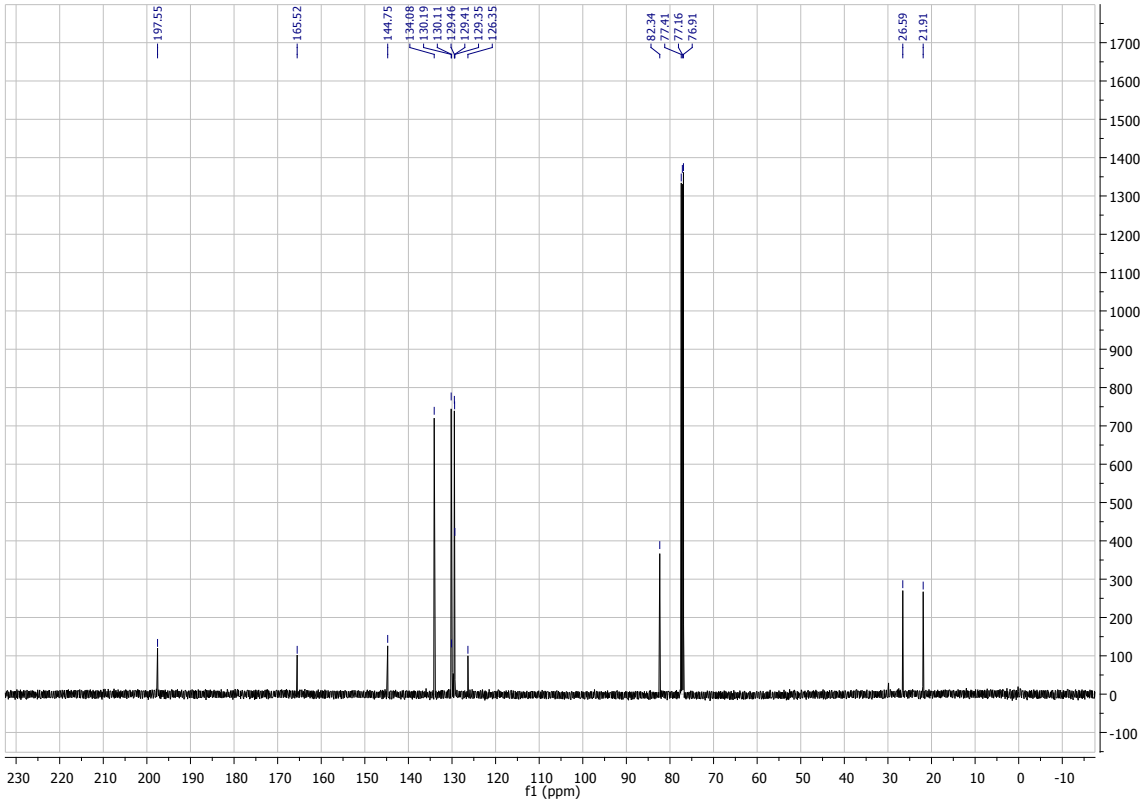
2.28
f1 (ppm)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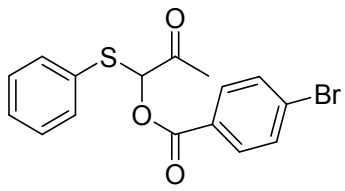
26.59
21.91
f1 (ppm)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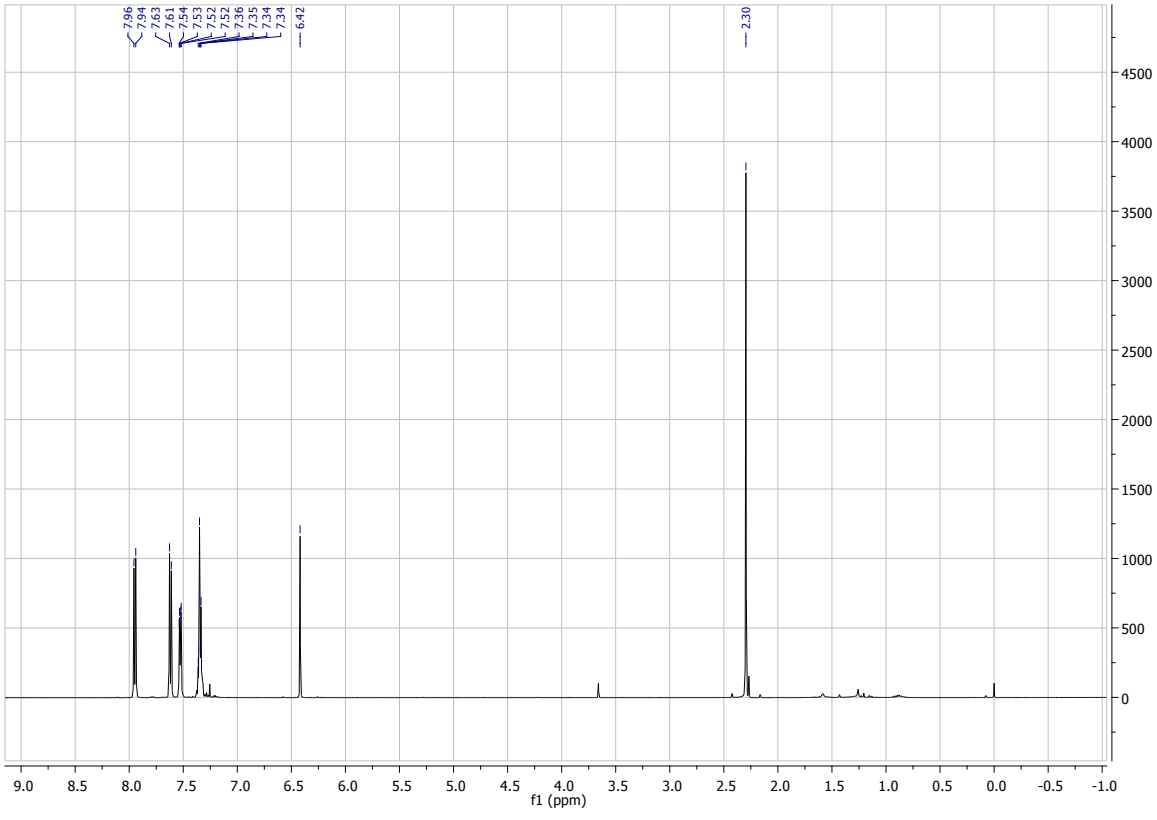
S
O
O
Br

3s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7.35
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7.34
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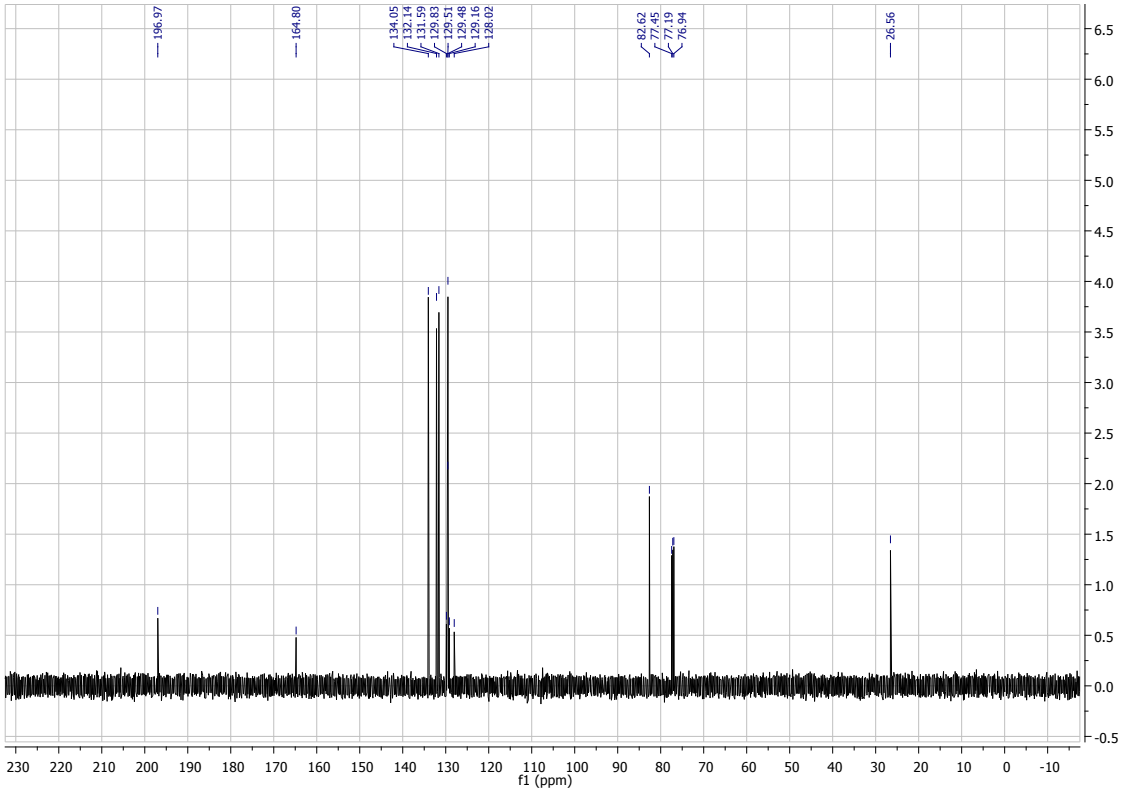
2.30
f1 (ppm)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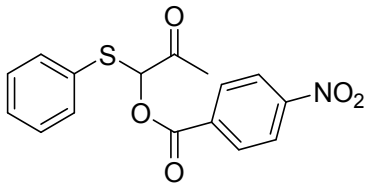
26.56
f1 (ppm)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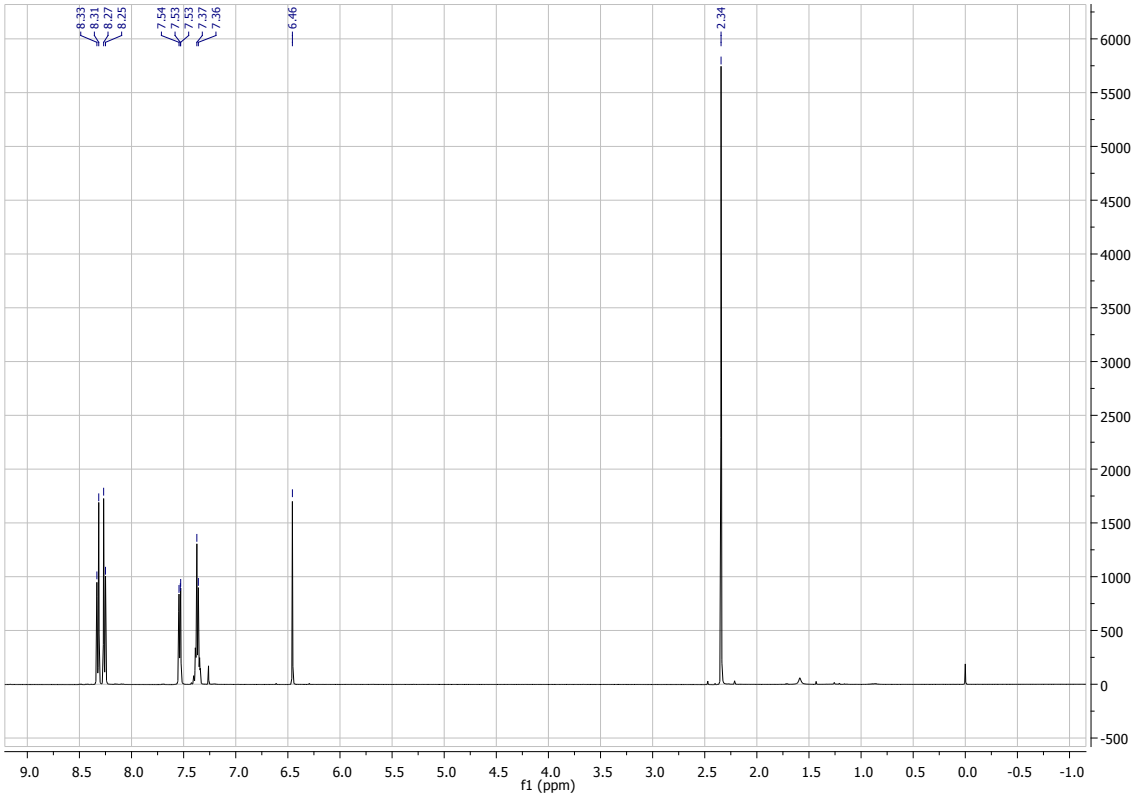
NO2
O

3t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7.53
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7.36
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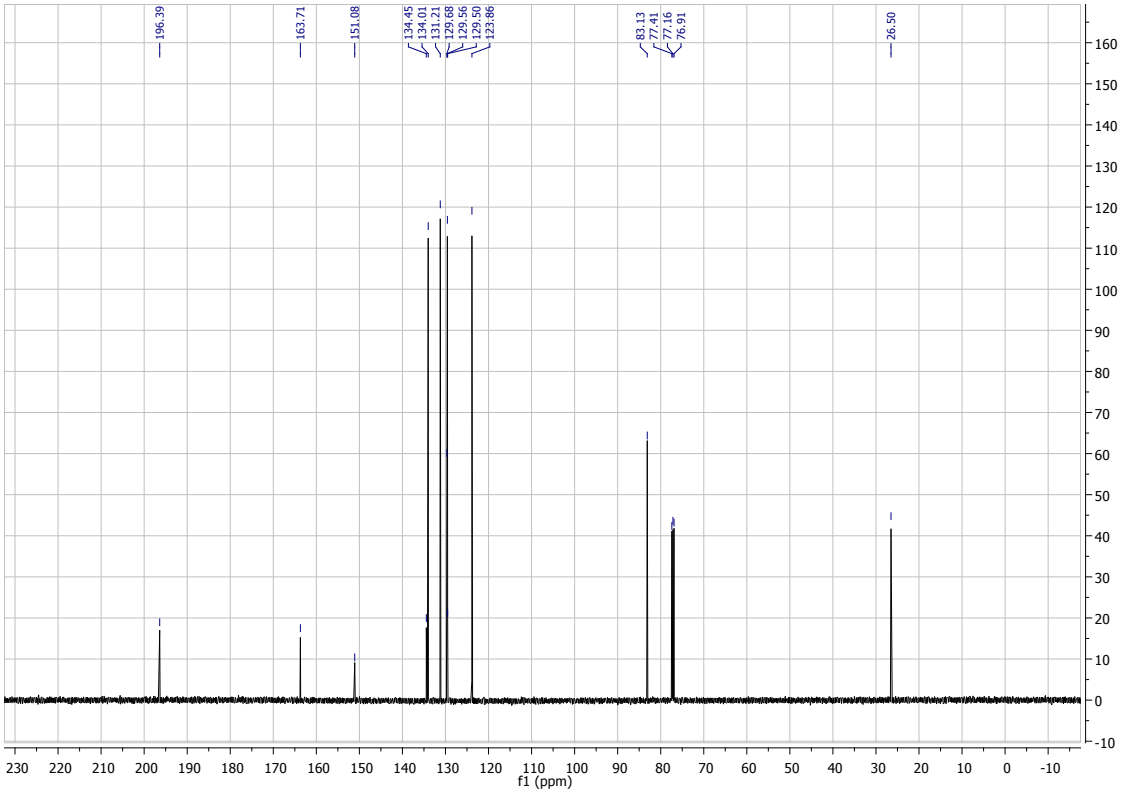
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123.86
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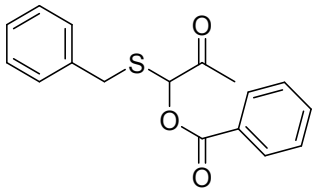
77.16
76.91
26.50
f1 (ppm)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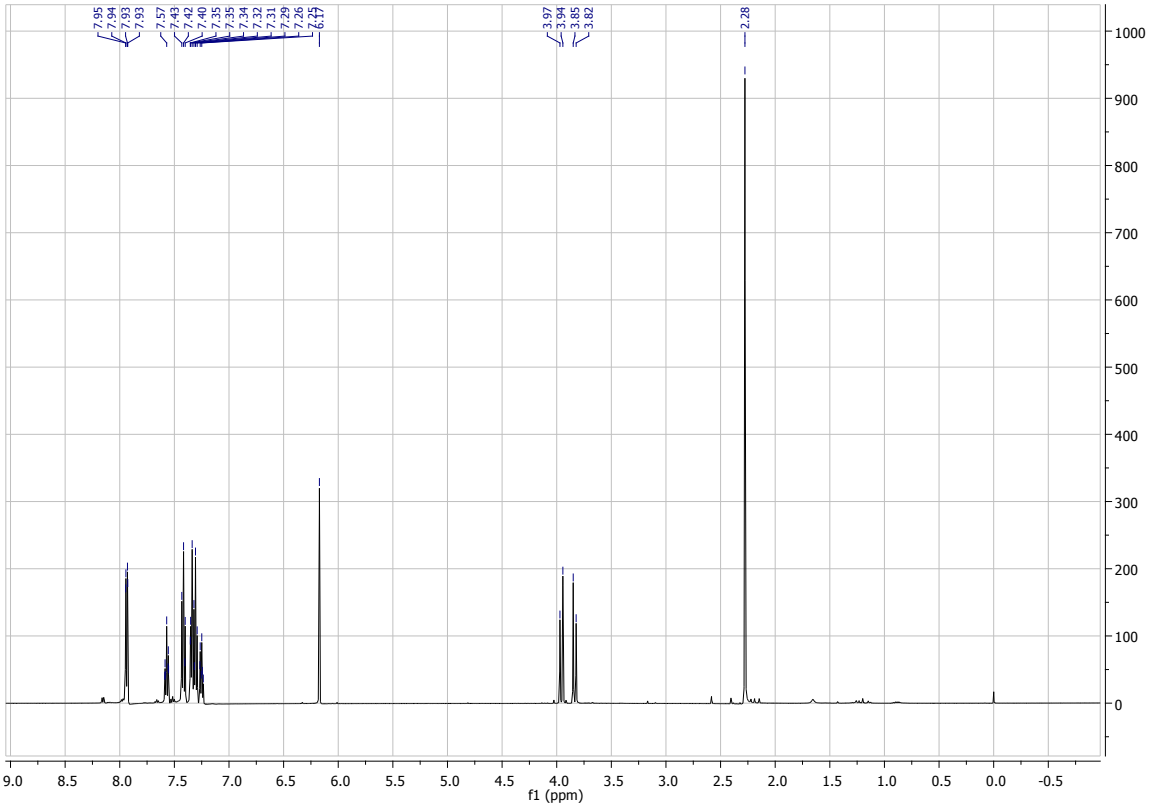
3u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7.26
6.17
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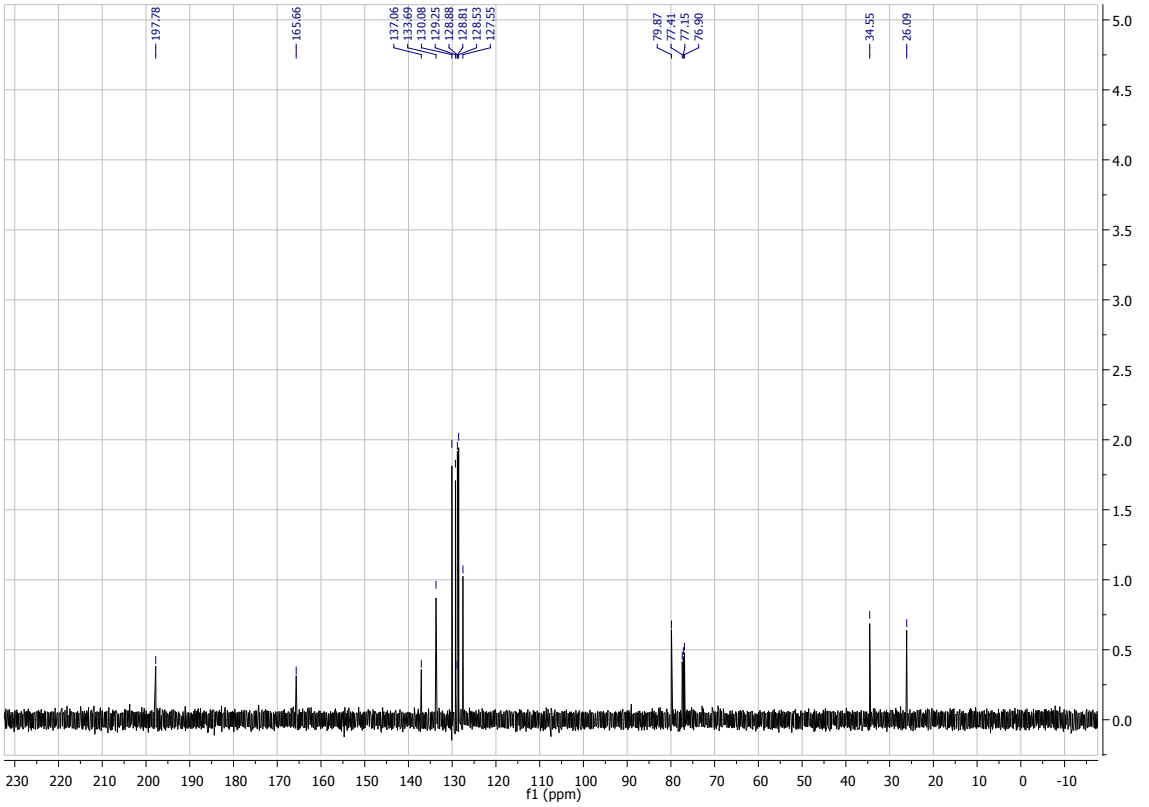
3.85
3.82
2.28
f1 (ppm)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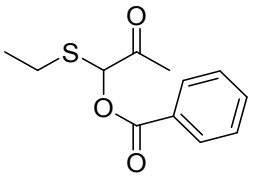
26.09
f1 (ppm)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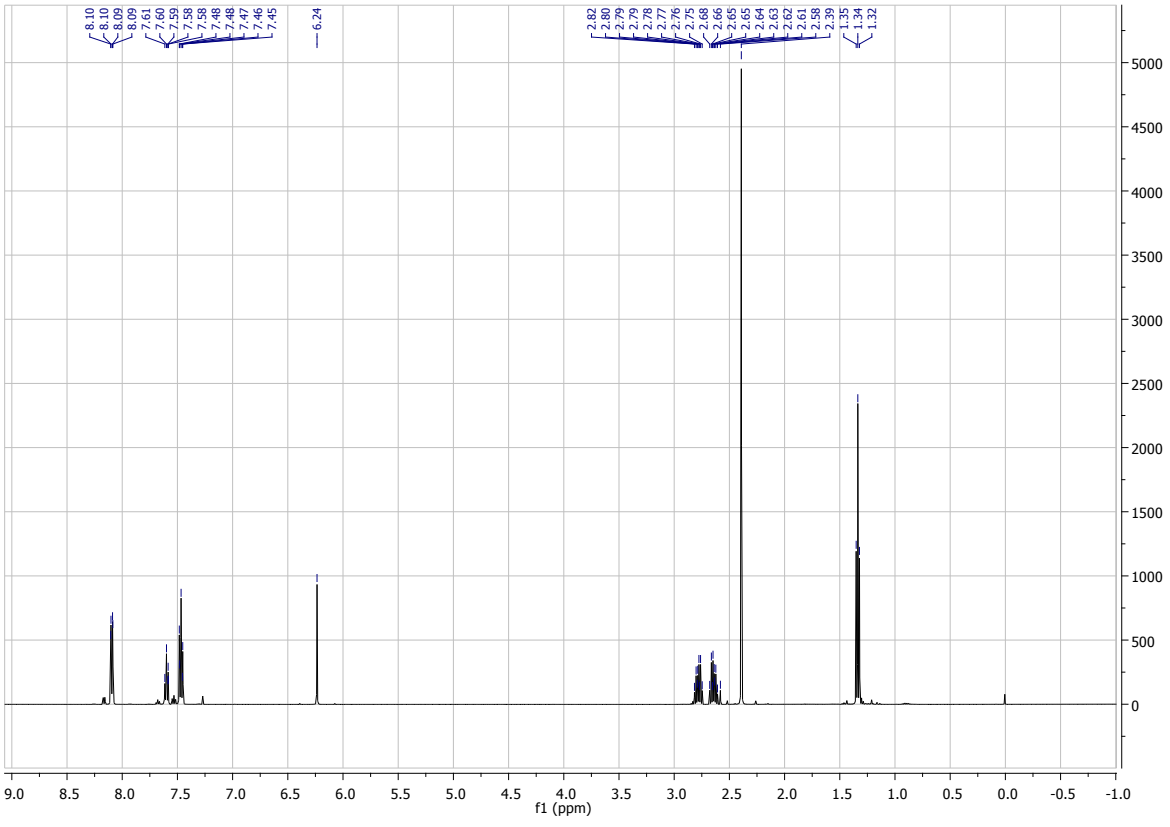
3v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7.47
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7.45
6.24
2.82
2.80
2.79
2.79
2.78
2.77
2.76
2.75
2.68
2.66
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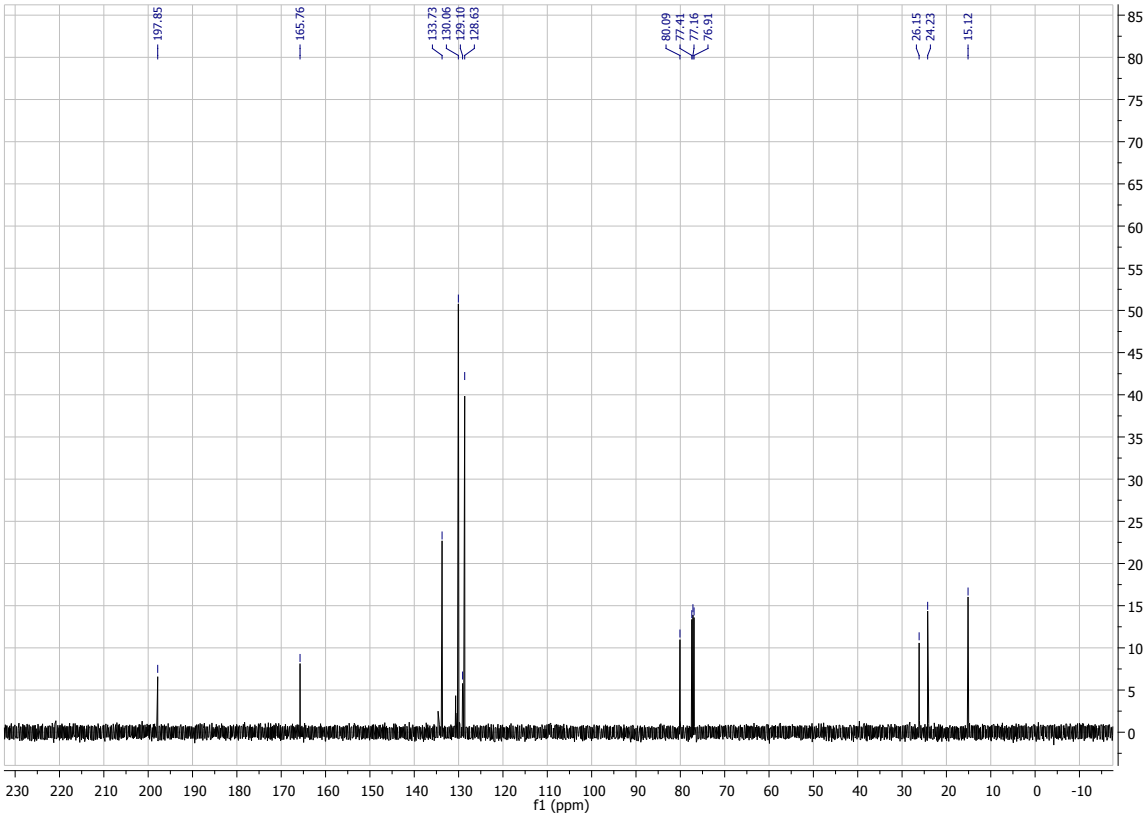
1.34
1.32
f1 (ppm)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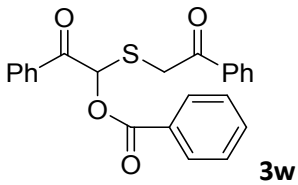
77.41
77.16
76.91
26.15
24.23
15.12
f1 (ppm)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Ph
S
Ph
O
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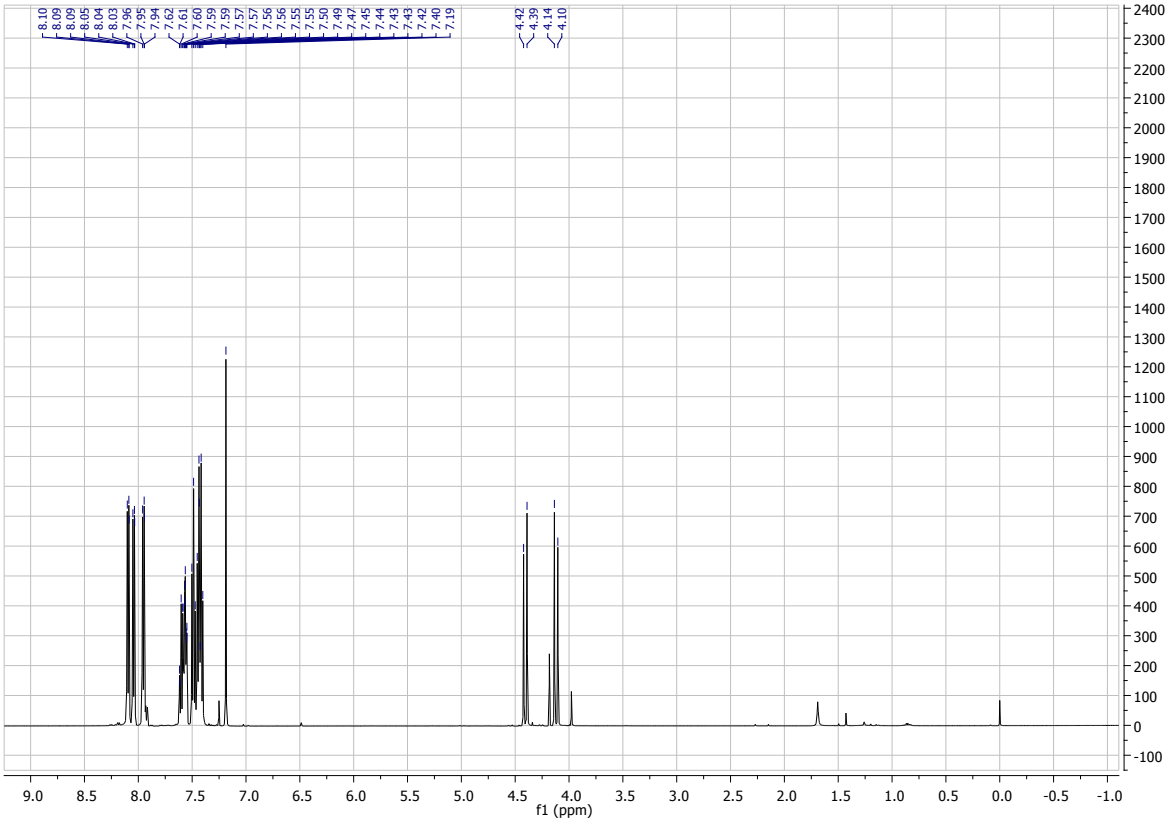
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7.56
7.56
7.55
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7.50
7.49
7.47
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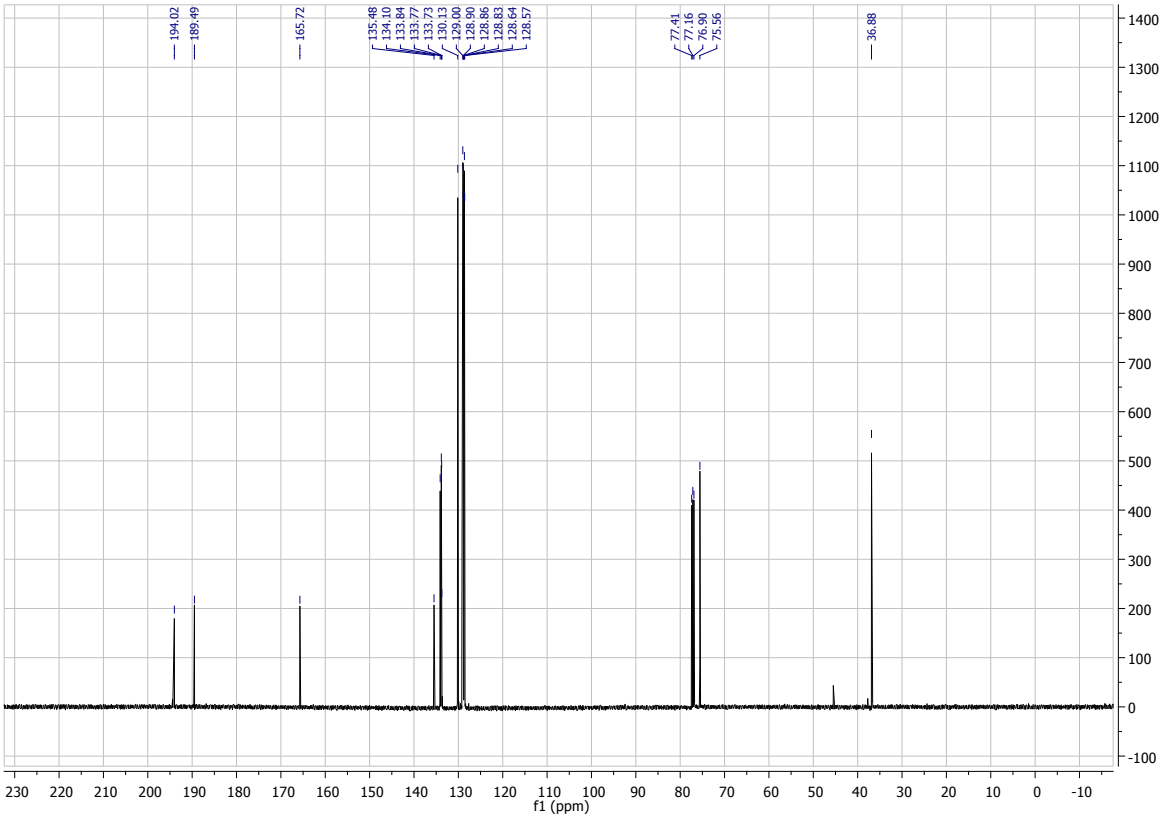
4.39
4.14
4.10
f1 (ppm)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76.90
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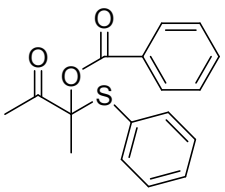
36.88
f1 (ppm)



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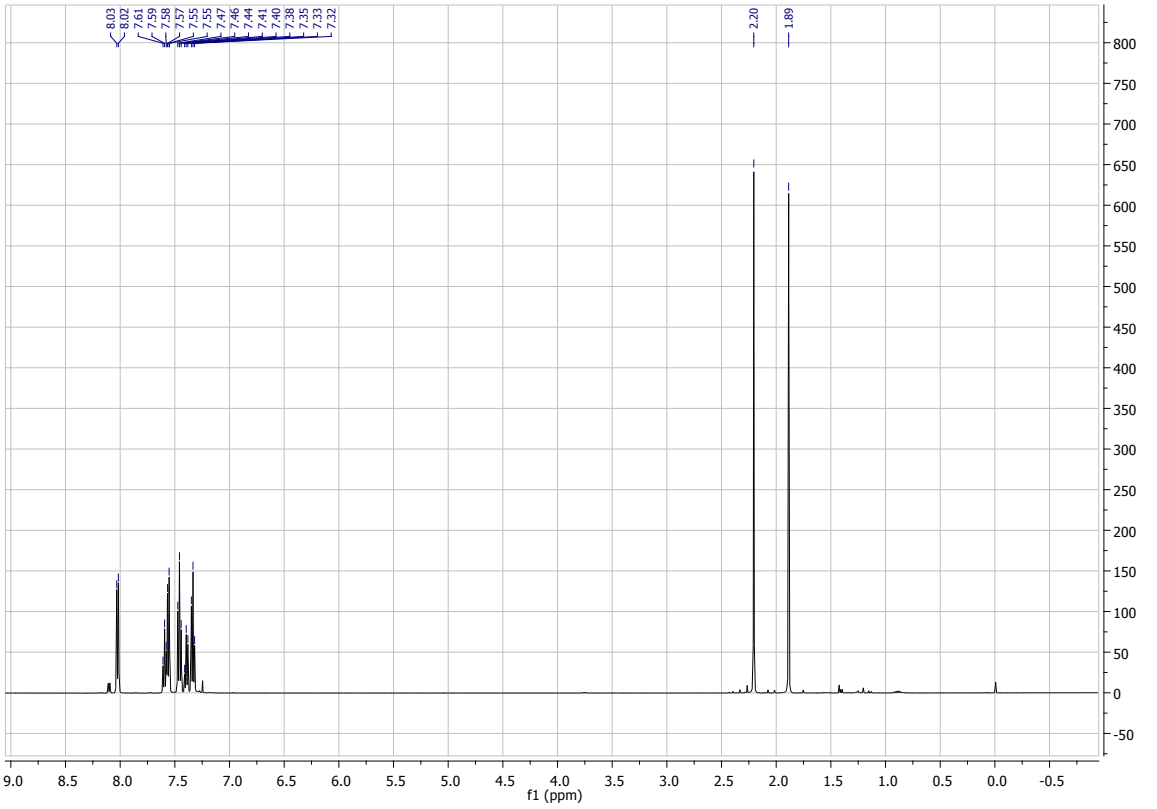
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7.33
7.32
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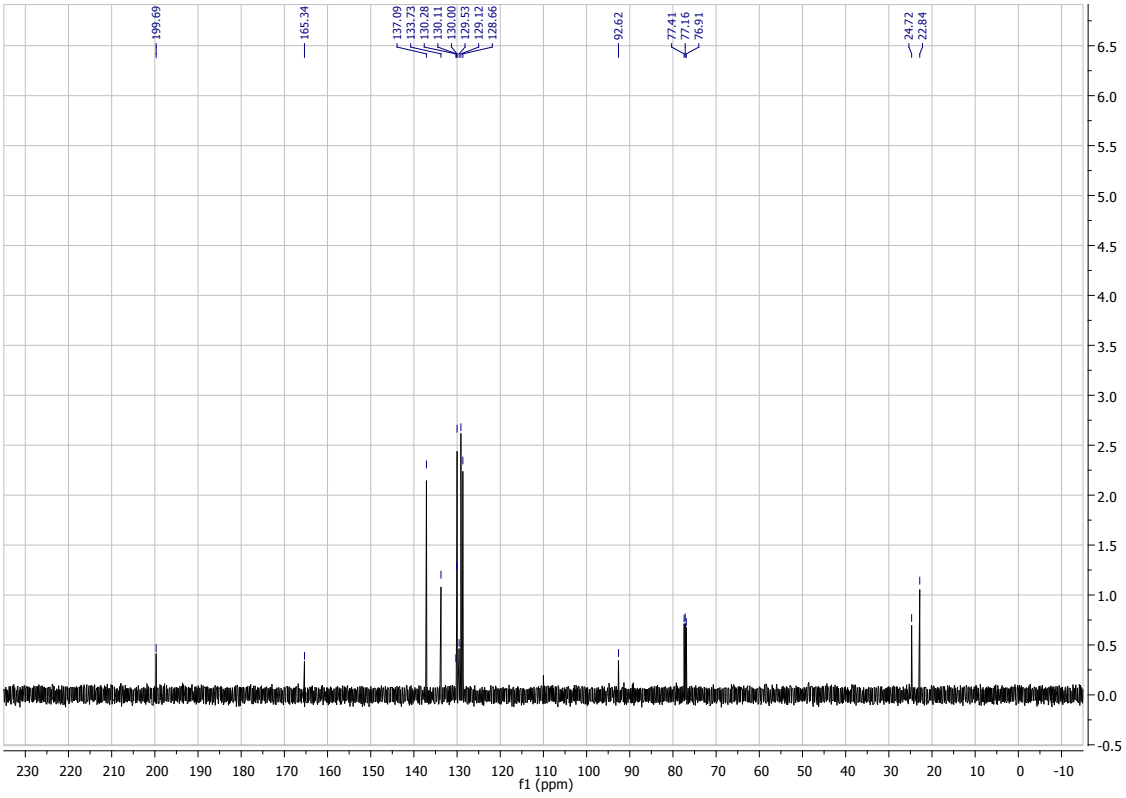
f1 (ppm)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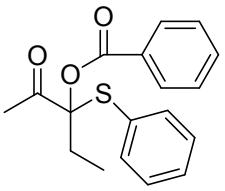
24.72
22.84
f1 (ppm)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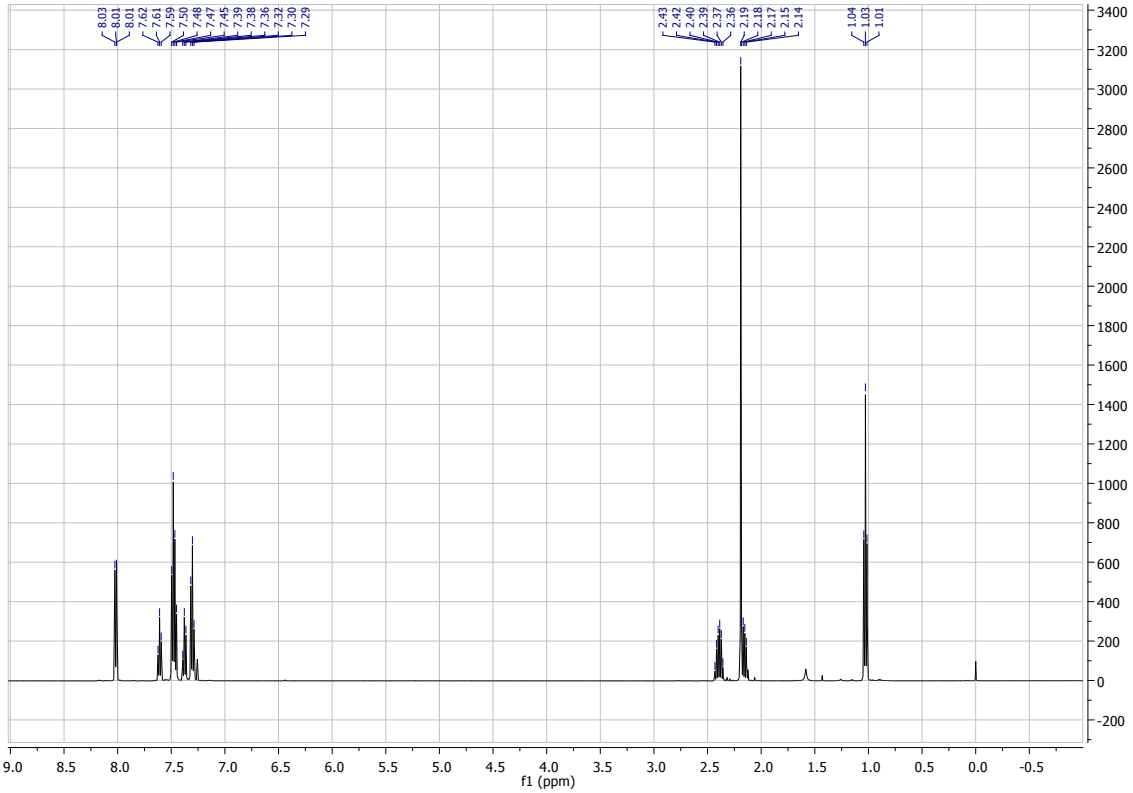
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7.45
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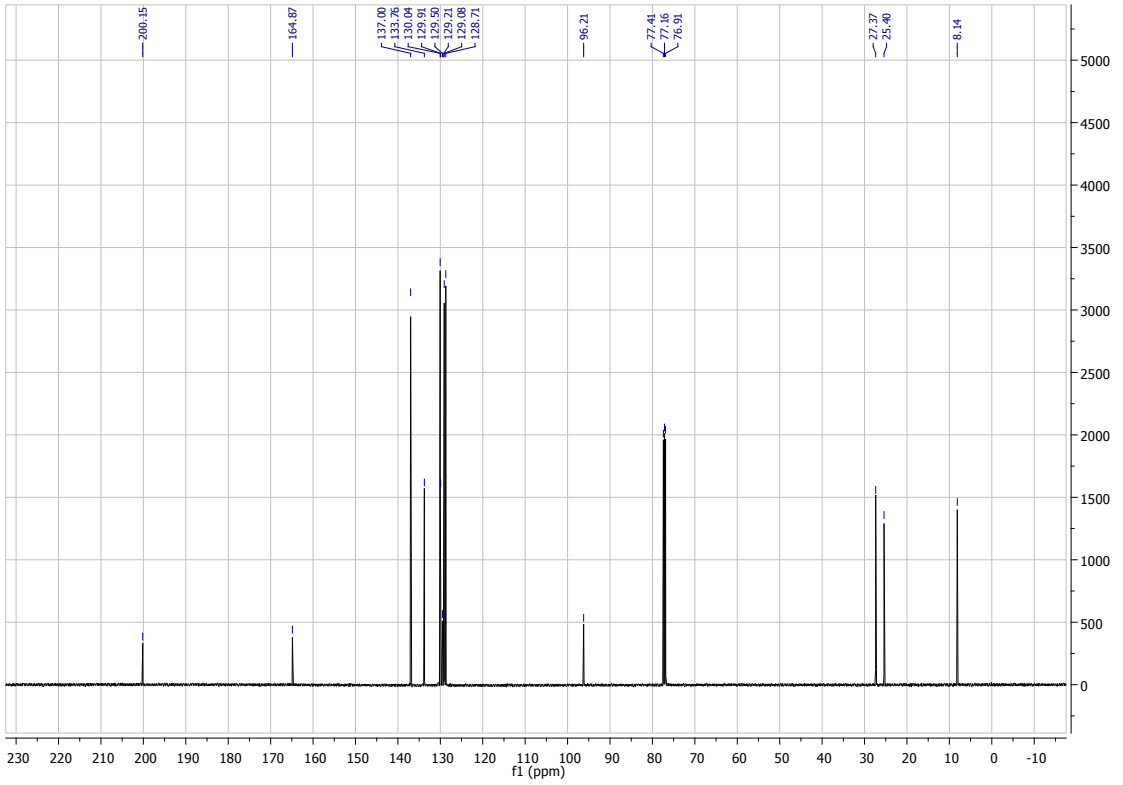
7.38
7.36
7.32
7.30
7.29
2.43
2.42
2.40
2.39
2.37
2.36
2.19
2.18
2.17
2.15
2.14
1.04
1.03
1.01
f1 (ppm)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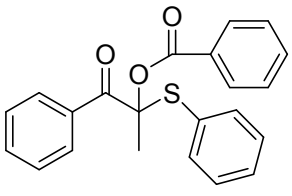
77.41
77.16
76.91
27.37
25.40
8.14
f1 (ppm)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O
S

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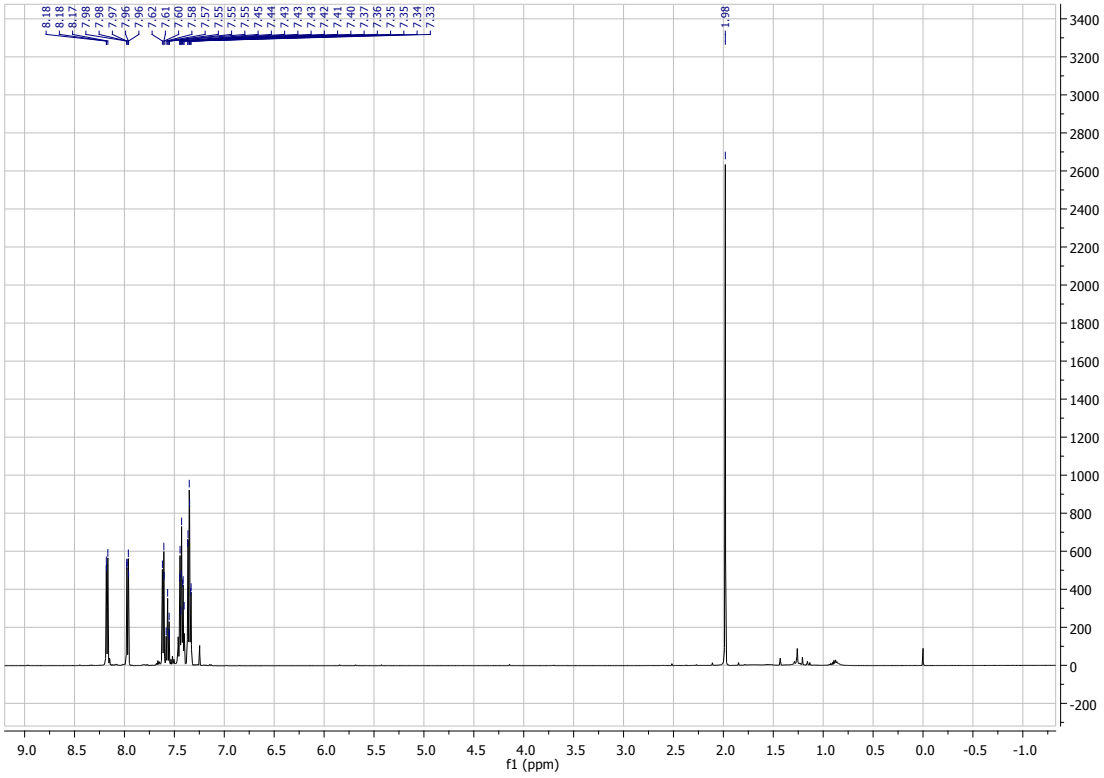
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7.55
7.55
7.55
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7.44
7.43
7.43
7.43
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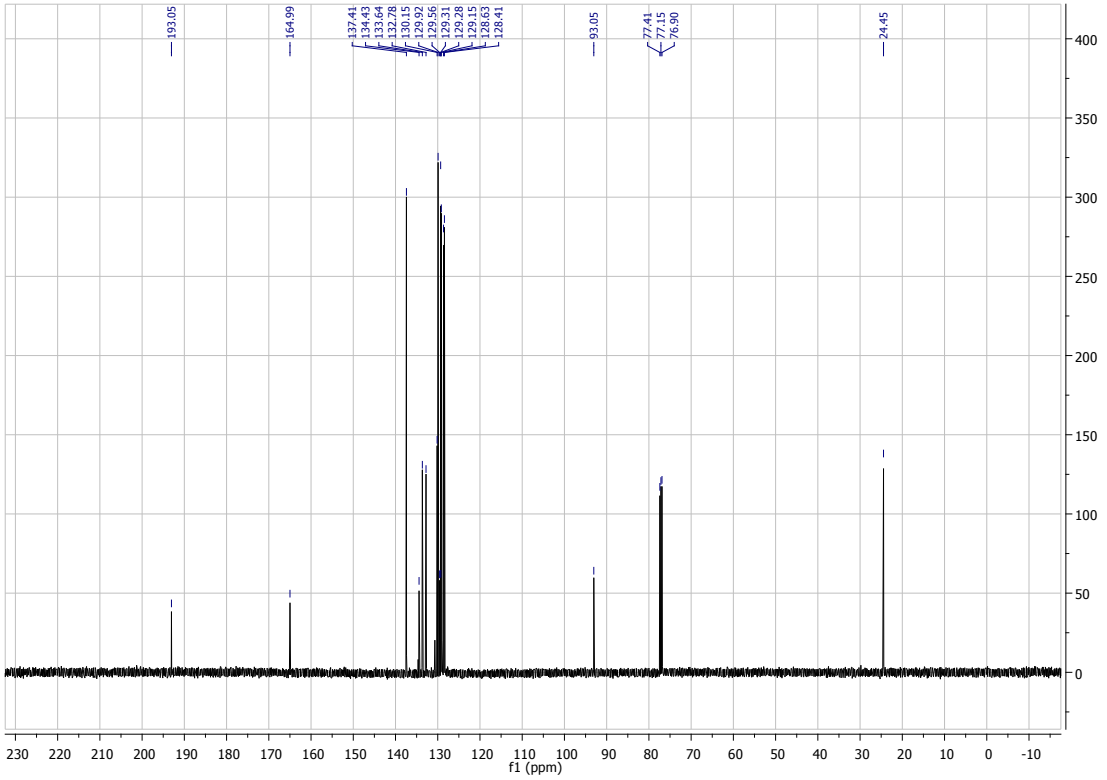
7.36
7.35
7.35
7.34
7.33
1.98
f1 (ppm)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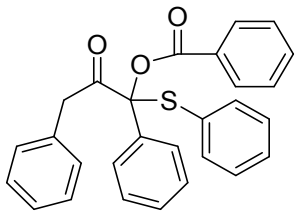
77.15
76.90
24.45
f1 (ppm)



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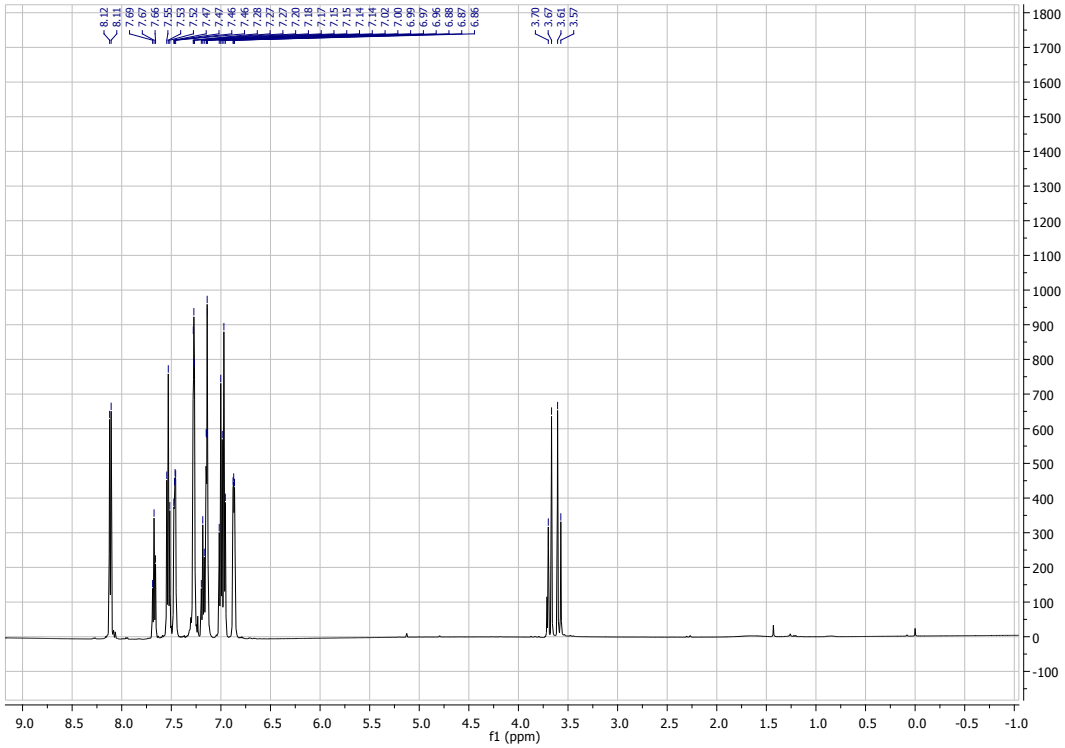
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7.47
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7.46
7.28
7.27
7.27
7.20
7.18
7.17
7.15
7.15
7.14
7.14
7.02
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6.96
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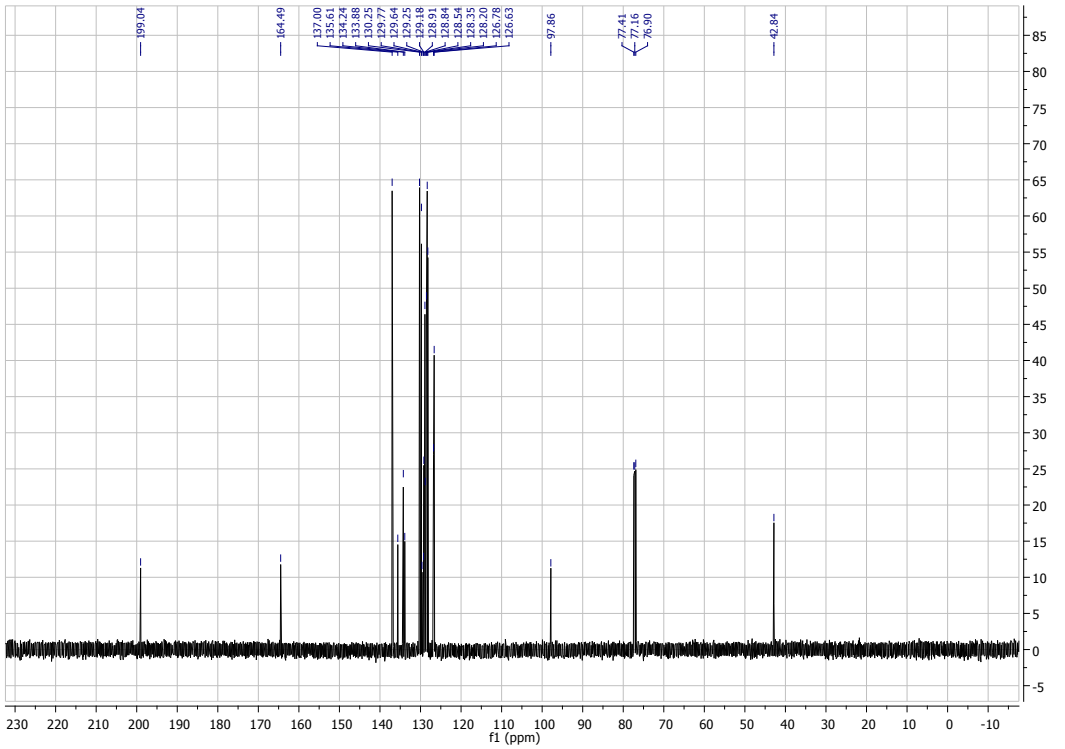
6.87
6.86
3.70
3.67
3.61
3.57
f1 (ppm)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128.91
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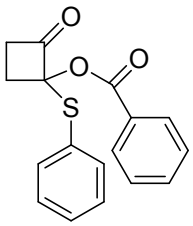
128.20
126.78
126.63
97.86
77.41
77.16
76.90
42.84
f1 (ppm)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S

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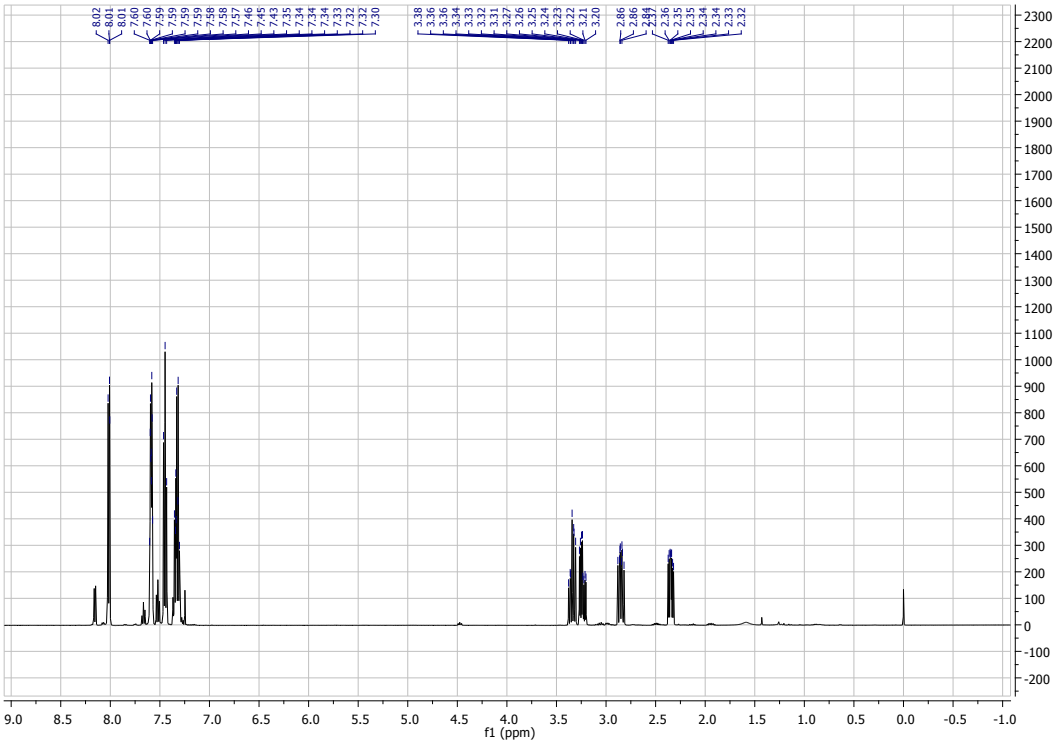
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7.58
7.57
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7.45
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3.24
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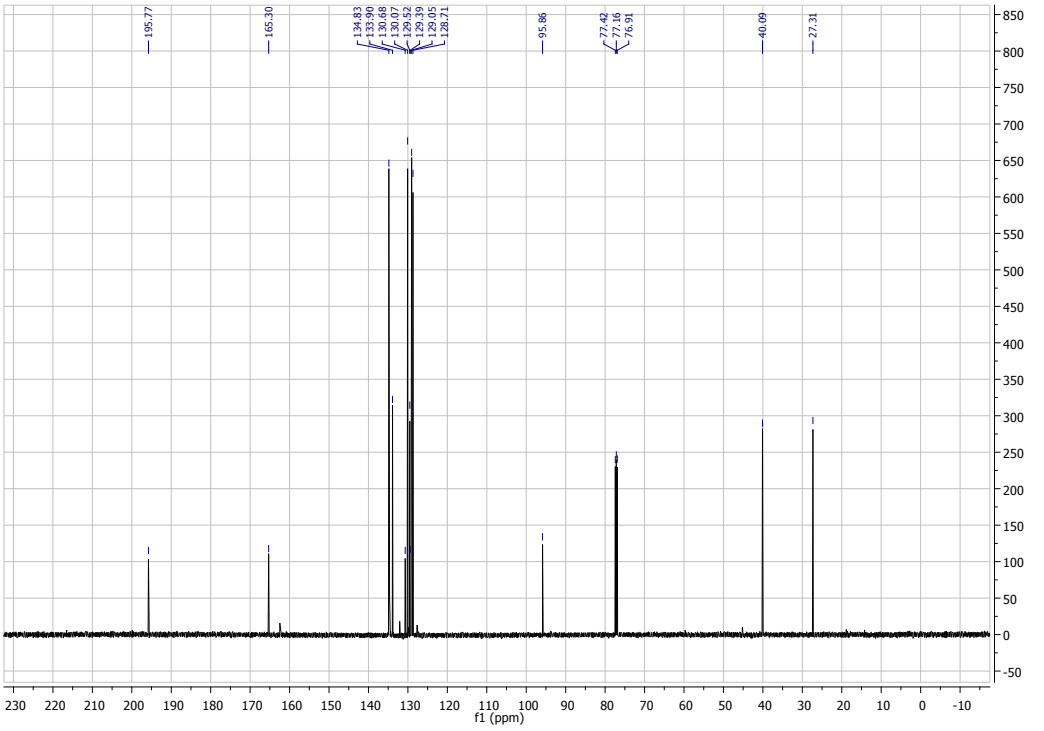
2.32
f1 (ppm)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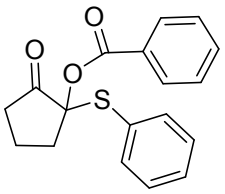
27.31
f1 (ppm)



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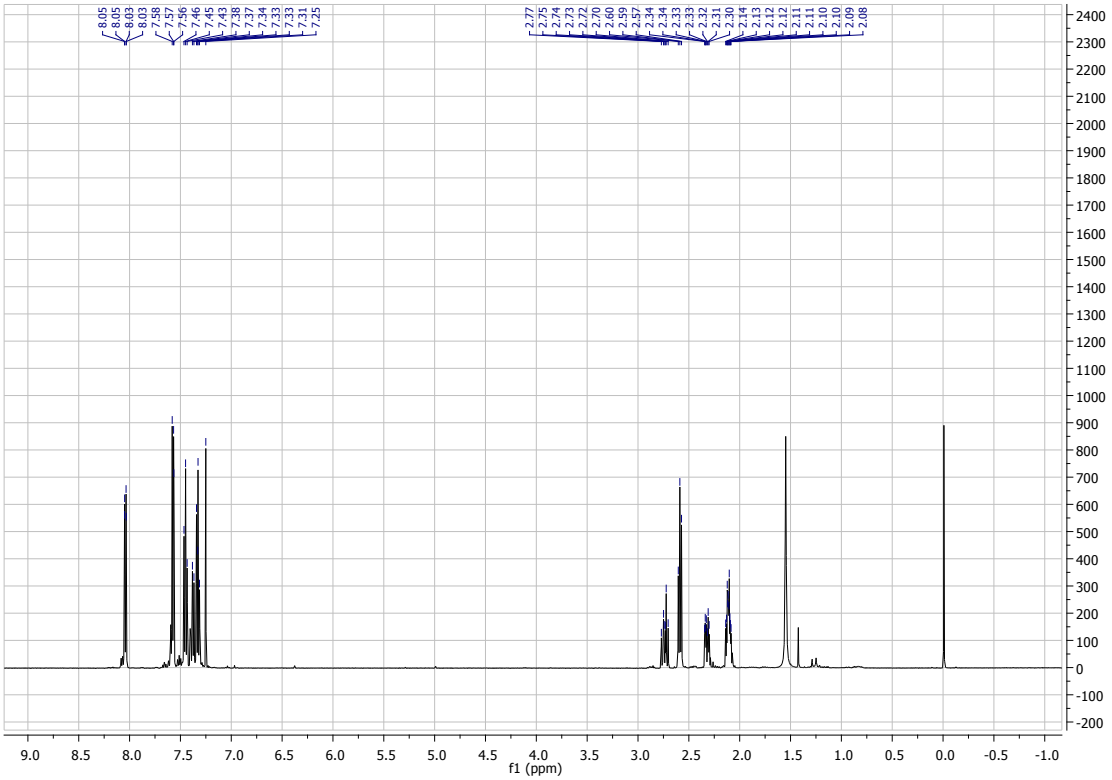
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7.33
7.33
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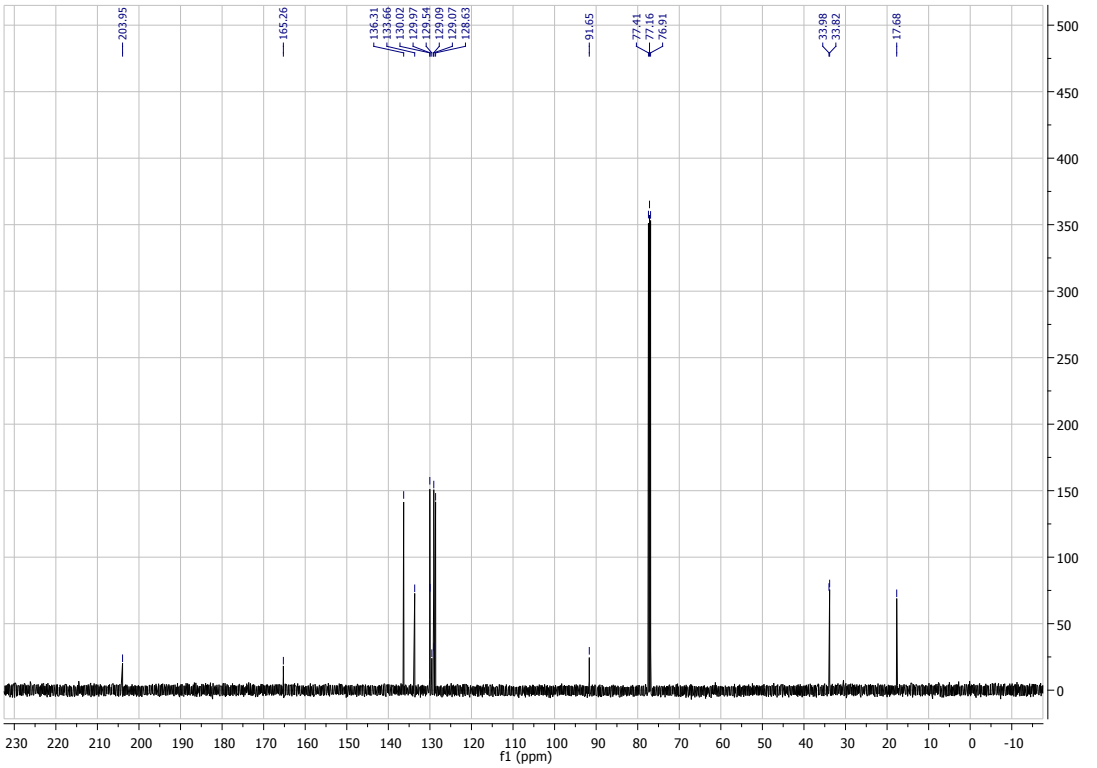
2.10
2.09
2.08
f1 (ppm)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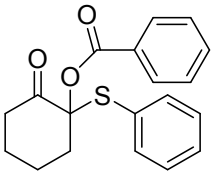
33.98
33.82
17.68
f1 (ppm)



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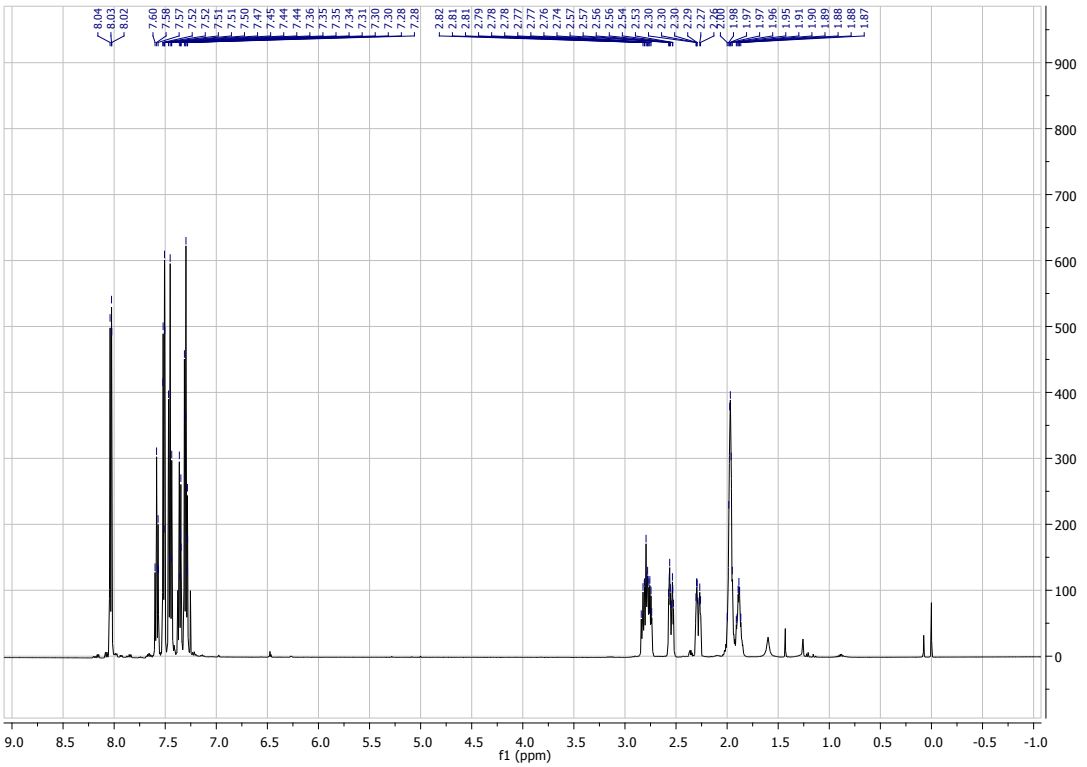
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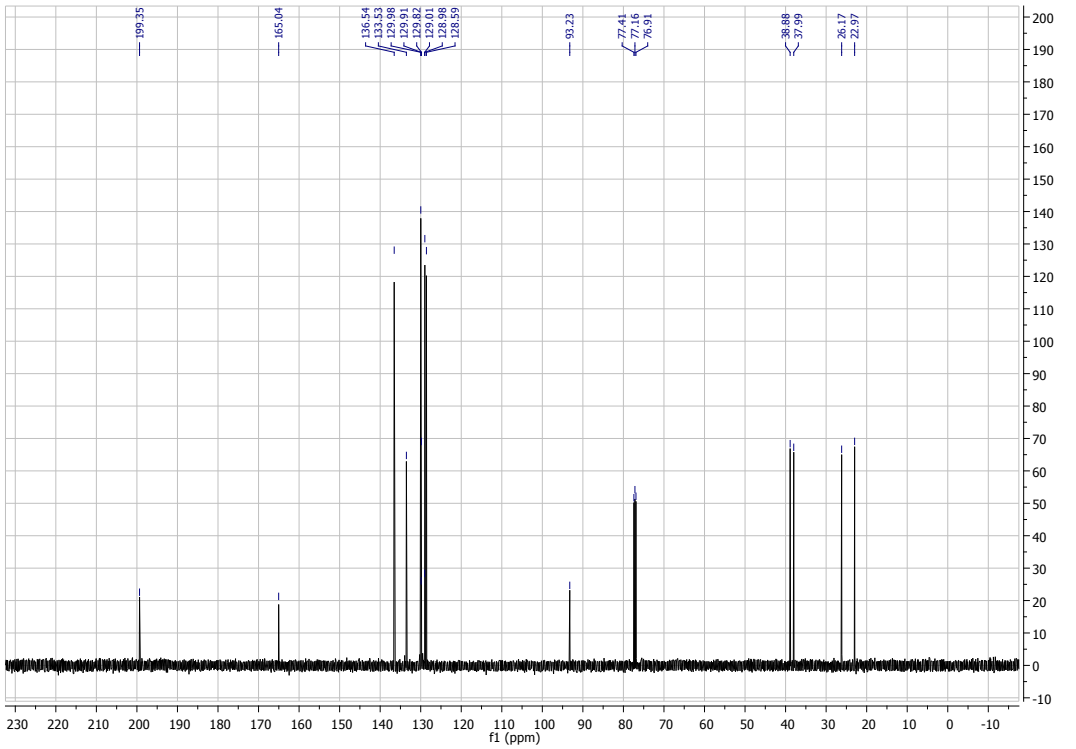
f1 (ppm)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76.91
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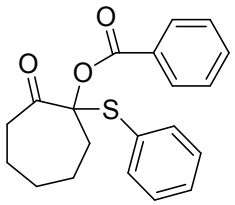
37.99
26.17
22.97
f1 (ppm)



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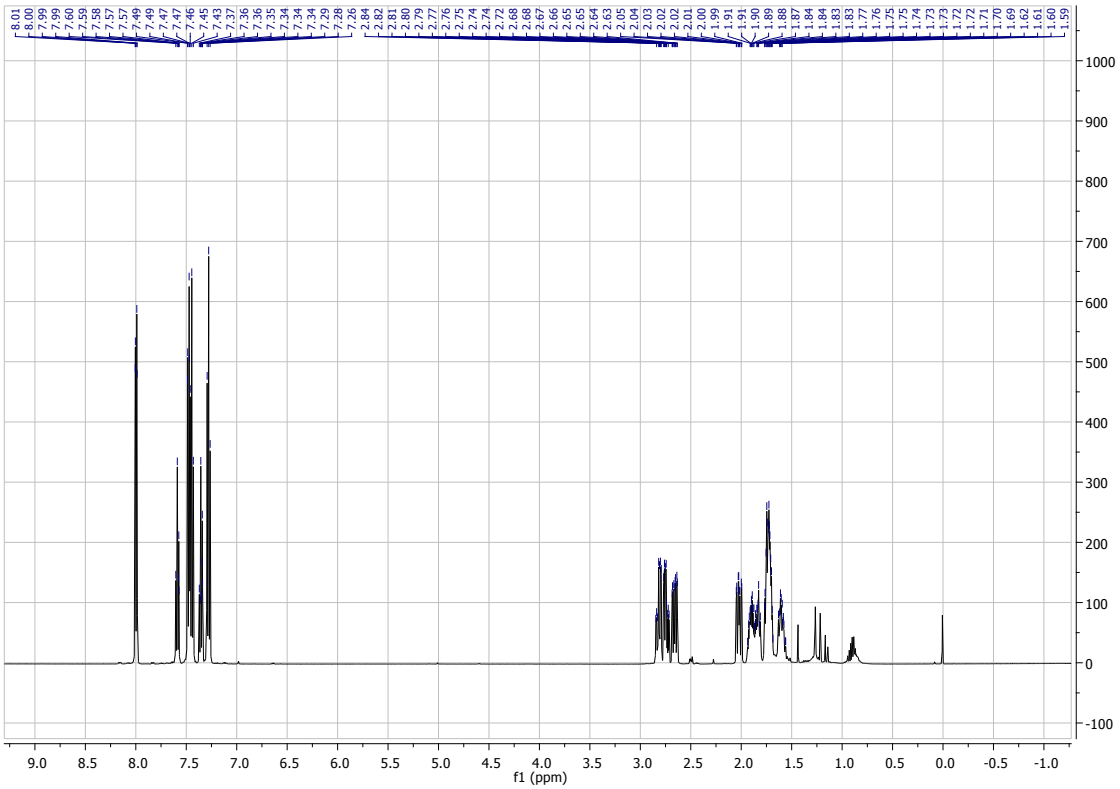
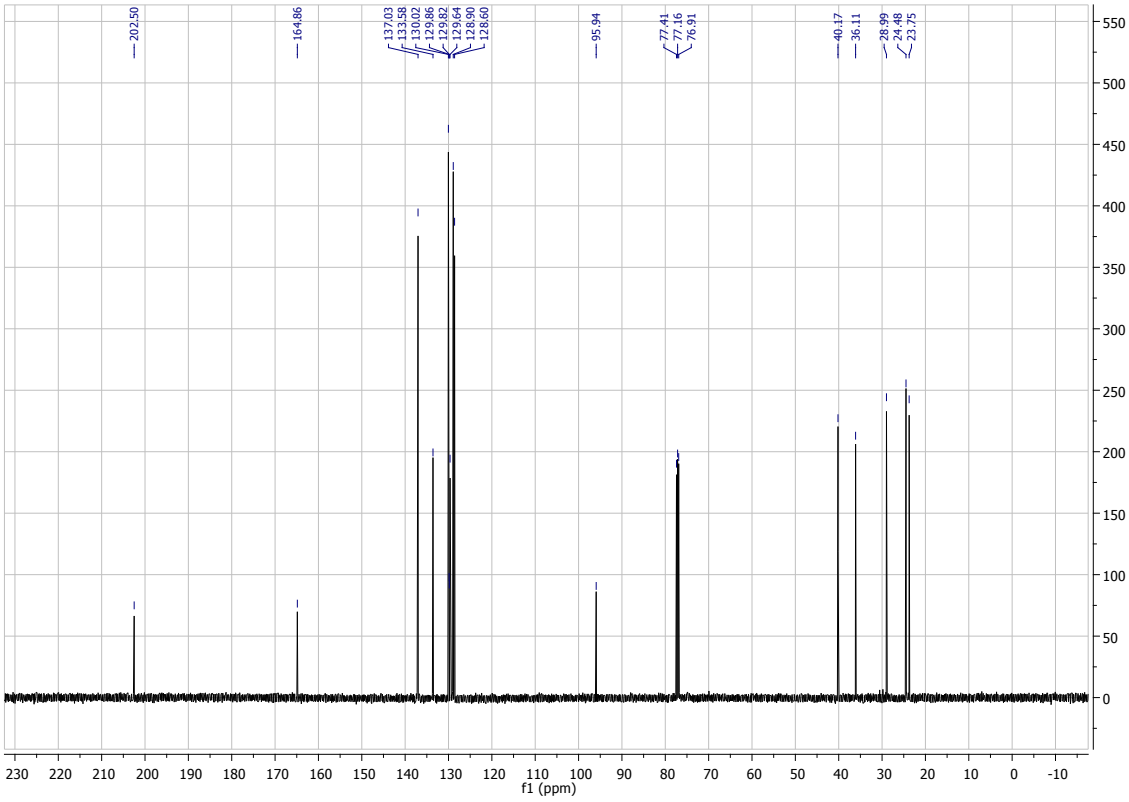
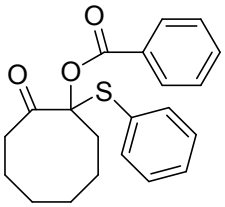
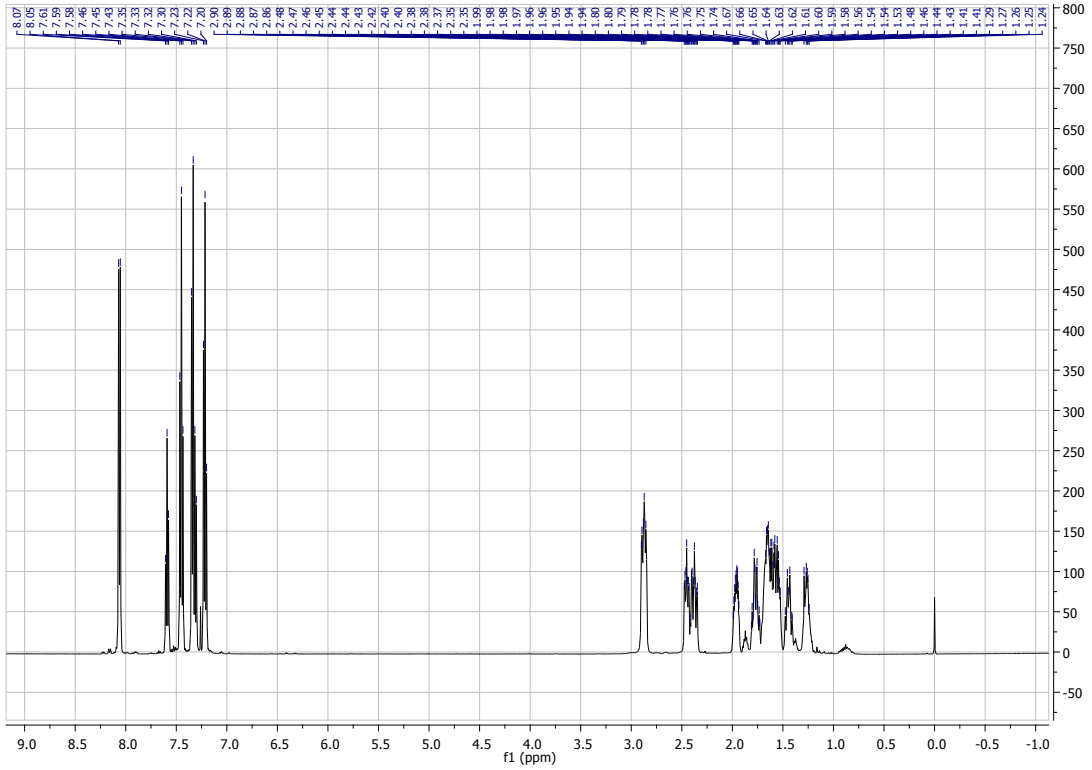





O
O
O
S

3ai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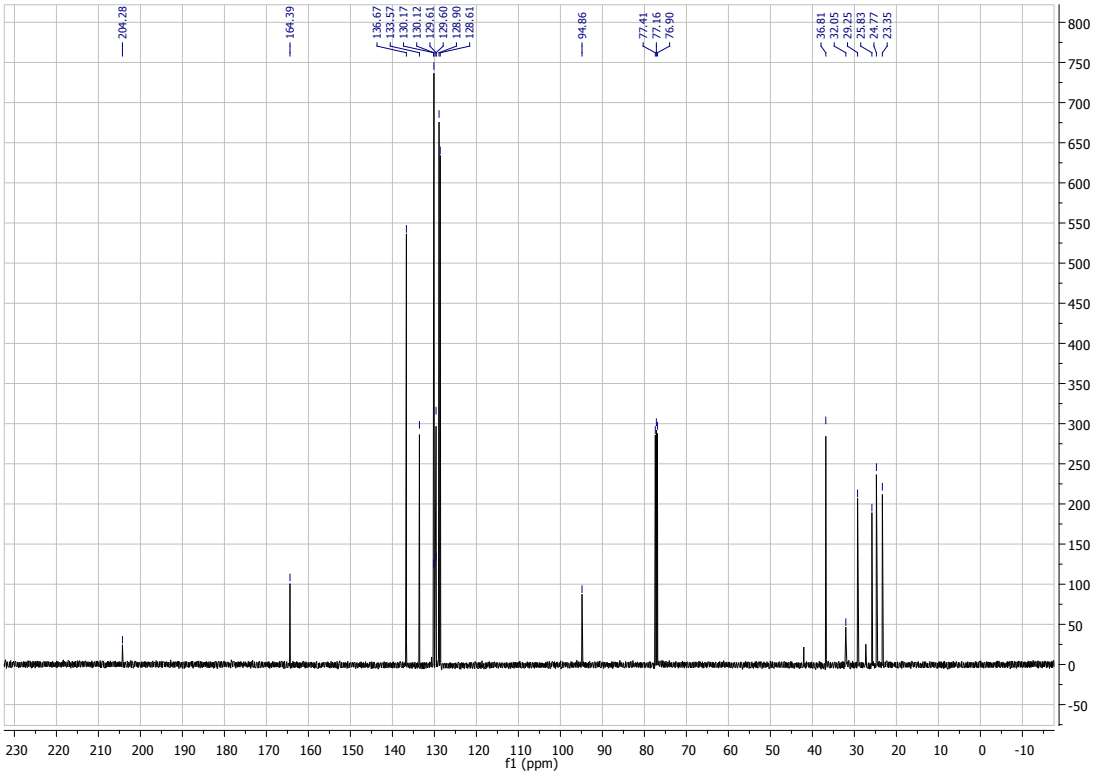
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36.81
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25.83
24.77
23.35
f1 (ppm)