Electronic Supplementary Information

Iron-surfactant nanocomposite catalyzed benzylic oxidation in water

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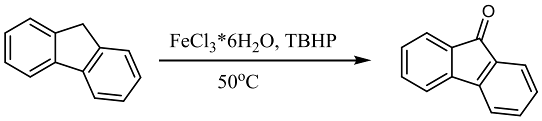
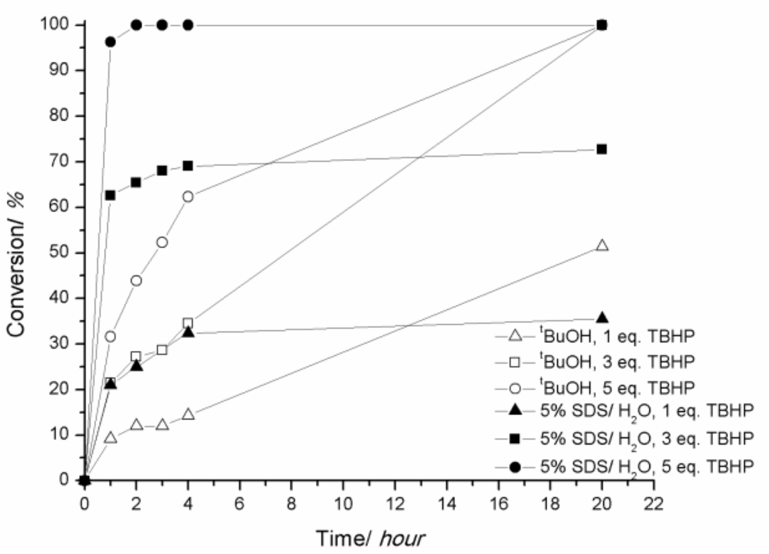
General

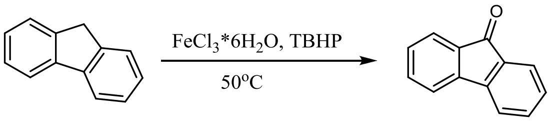
Unless otherwise indicated, all starting materials were obtained from commercial suppliers, and were used without further purification. Analytical thin-layer chromatography (TLC) was performed on Merck DC pre coated TLC plates with 0.25 mm Kieselgel 60 F_{254}. Visualization was performed with a 254 nm UV lamp. The ^{1}H and ^{13}C NMR spectra were recorded on a Bruker Avance-250 spectrometer in $CDCl_3$. Chemical shifts are expressed in parts per million (δ) using residual solvent protons as internal standards (δ 7.26 for ^{1}H, δ 77.0 for ^{13}C). Coupling constants (*J*) are reported in Hertz (Hz). Splitting patterns are designated as s (singlet), d (doublet), t (triplet), m (multiplet). Combination gas chromatography and low resolution mass spectrometry was obtained on an Agilent 6890N Gas Chromatograph (30 m x 0.25 mm column with 0.25 μm HP-5MS coating, He carrier gas) and Agilent 5973 Mass Spectrometer (Ion source: EI+, 70eV, 230^{o}C; interface: 300^{o}C). IR spectra were obtained on a Bruker Alpha spectrometer on a single-reflection diamond ATR unit. All melting points were measured on Büchi 501 apparatus and are uncorrected. High-resolution mass spectra were recorded on an Agilent Technologies 6210 Time of Flight mass spectrometer.

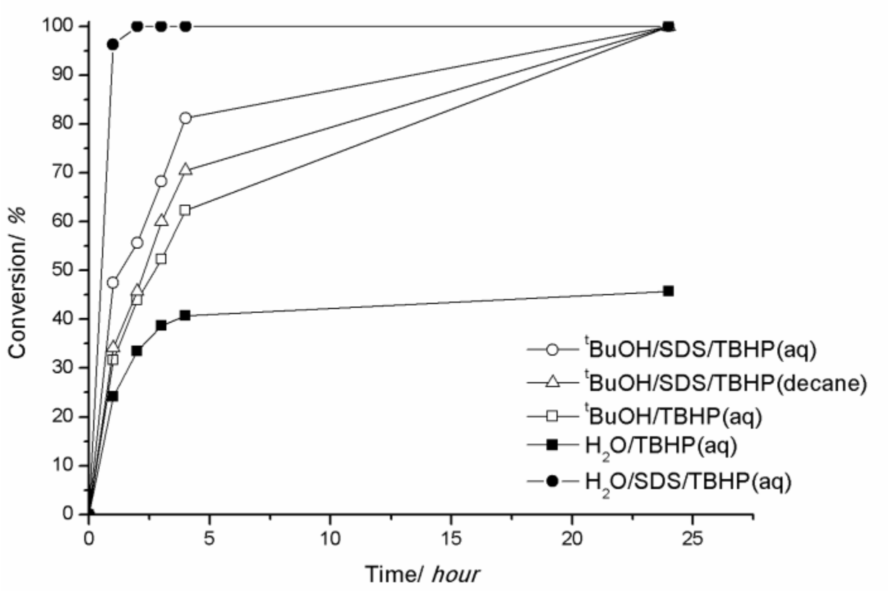
Effect of TBHP loading and the role of SDS in different solvents.





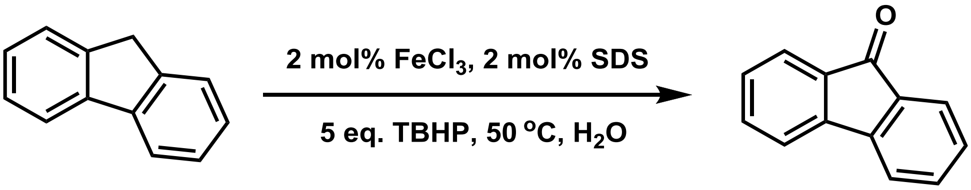
Effect of TBHP loading and the role of SDS in different solvents. Fluorene (1 mmol) TBHP (70 % aq, 5 mmol); $FeCl_3$*6 H_2O (0.02 mmol, 2%) 5 w/w% SDS solution in water or ^{t}BuOH (1 mL), at 50°C, % composition of the product determined by GC.

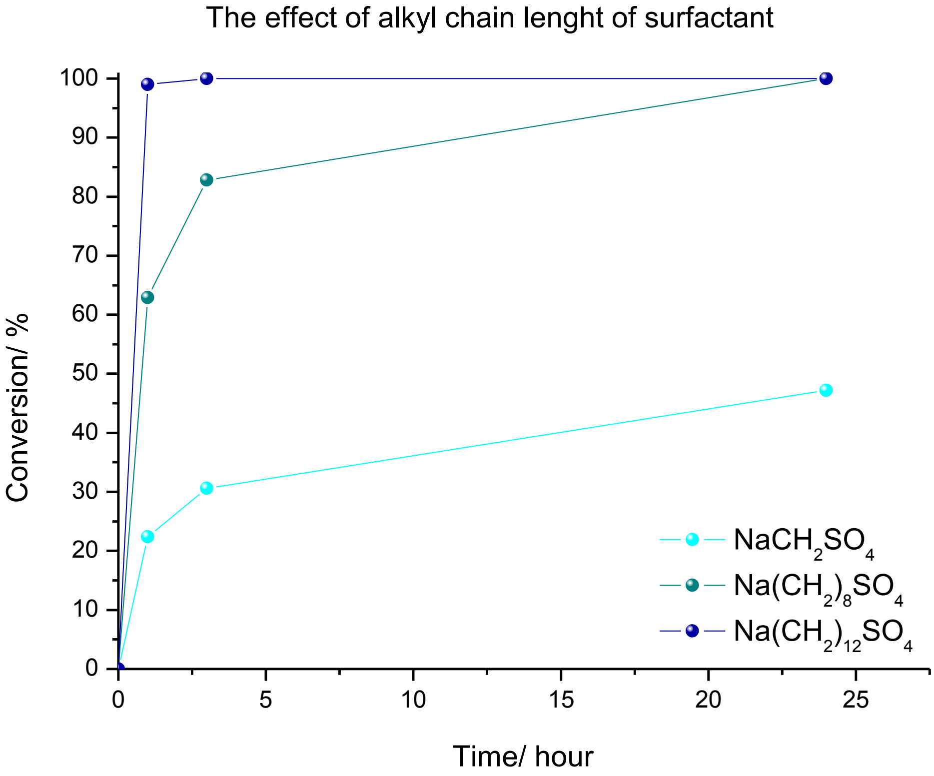




Fluorene (1 mmol) TBHP (70% aq, 5 mmol); $FeCl_3$*6 H_2O (0.02 mmol, 2%) 5 w/w% SDS solution in water or ^{t}BuOH (1 mL), at 50°C. % composition of the product determined by GC

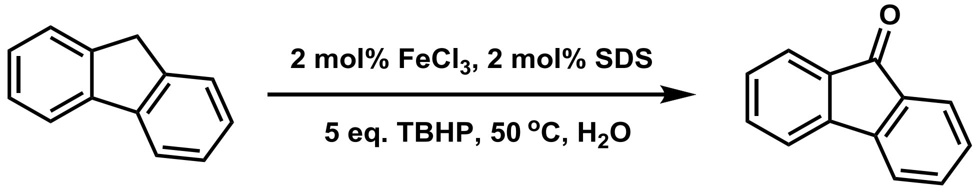
The influence of the carbon chain length on the conversion

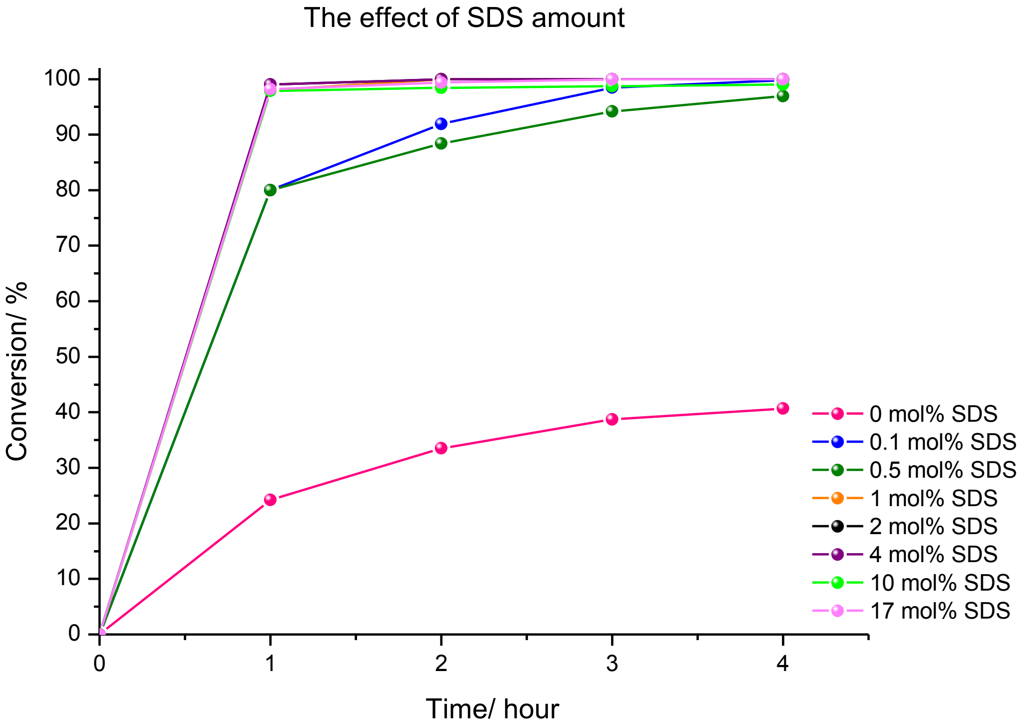




A screw capped vial with stir bar was charged with the 2 mol% $FeCl_3$, 2 mol% SMS, SOS or SDS and water. Fluorene (1 eq.) was added followed by 5 eq. TBHP (70% aq.). The reaction vessel was closed, and stirred for 24 hours at 50°C. Samples were taken and analyzed by GC.

The effect of SDS amount on the conversion

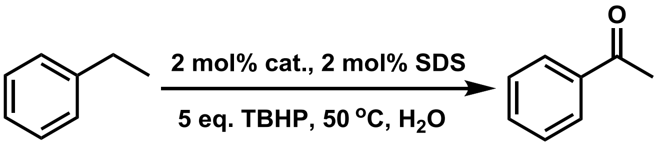


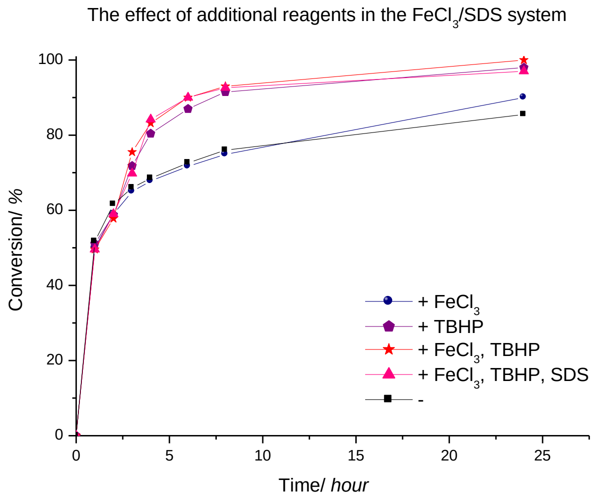


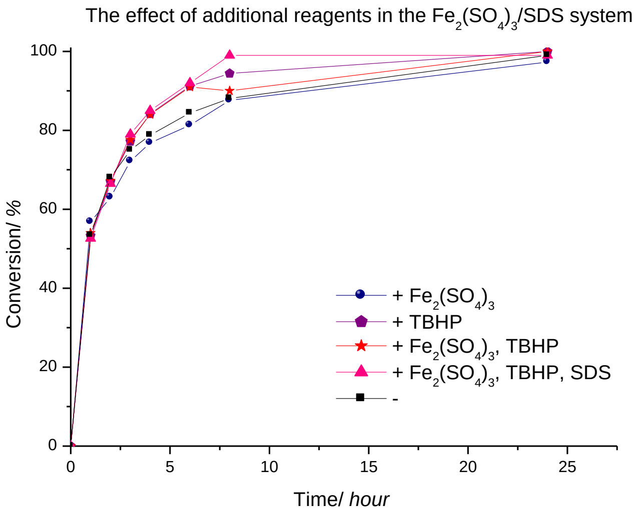
A screw capped vial with stir bar was charged with the 2 mol% $FeCl_3$, x mol% SDS and water. Fluorene (1 eq.) was added followed by 5 eq. TBHP (70% aq.). The reaction vessel was closed, and stirred for 4 hours at 50°C. Samples were taken and analyzed by GC.

Addition of excess TBHP, $FeCl_3$/$Fe_2(SO_4)_3$, SDS after 2 hours

However, addition of 2 mol% iron salt was sufficient to reach full conversion of fluorene to fluorenone in short reaction time, the oxidation of ethylbenzene to acetophenone was not completed in the presence of either 2 mol% $FeCl_3$ or 2 mol% $Fe_2(SO_4)_3$ (60% and 80% conversions were obtained after 24 hours respectively). In order to reach full conversion of this less reactive substrate we added a second portion of additives after 2 hours into the reaction mixture. Addition of TBHP ensures the completion of the reaction in 24 hours, and joint addition of extra TBHP/$FeCl_3$ or TBHP/$FeCl_3$/SDS did not cause better acceleration.[12] Interestingly, reactivation of the catalyst system in case of $Fe_2(SO_4)_3$ did not occurred as significantly as in the case of $FeCl_3$.



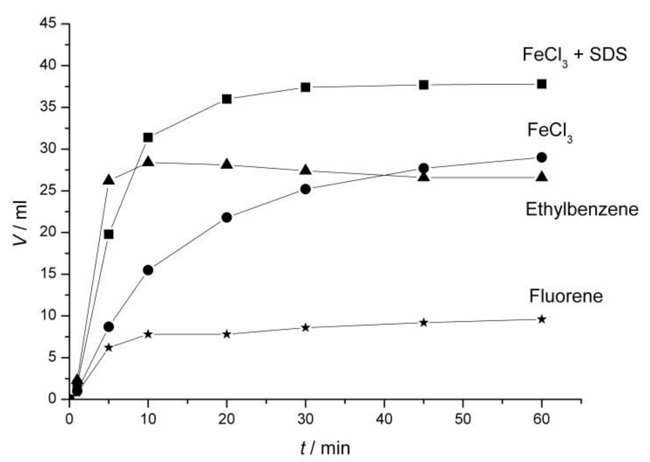




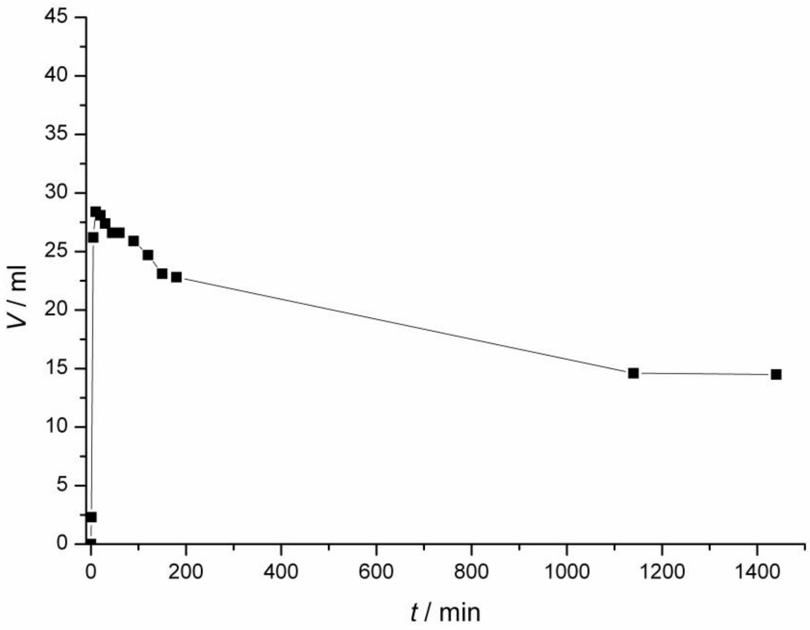
A screw capped vial with stir bar was charged with the 2 mol% catalyst, 2 mol% SDS and water. Ethyl benzene or fluorene (1 eq.) was added followed by 5 eq. TBHP (70% aq.). The reaction vessel was closed, and stirred for 2 hours at 50°C. Then second portion of iron salt, SDS, TBHP was added, and stirring was continued for further 22 hours. Samples were taken and analyzed by GC.

Oxygen evolution measurement

When ethylbenzene substrate was in the reaction mixture of $FeCl_3$, SDS and TBHP in water the oxygen slowly absorbed back into the solution. Oxygen volume was measured with gas burette:



Oxygen evolution experiments. TBHP (70% aq, 5 mmol, 5 eq.); $FeCl_3$*6 H_2O (0.05 mmol, 5%), SDS (0.05 mmol, 5%), H_2O (1 ml) at 50°C. % composition of the product determined by GC. ■: $FeCl_3$+ SDS. ●: $FeCl_3$▲ $FeCl_3$+ SDS + ethylbenzene (1 mmol, 1 eq.), ✿ $FeCl_3$+ SDS + Fluorene (1 mmol, 1eq.).


V / ml
t / min
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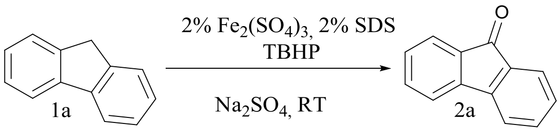
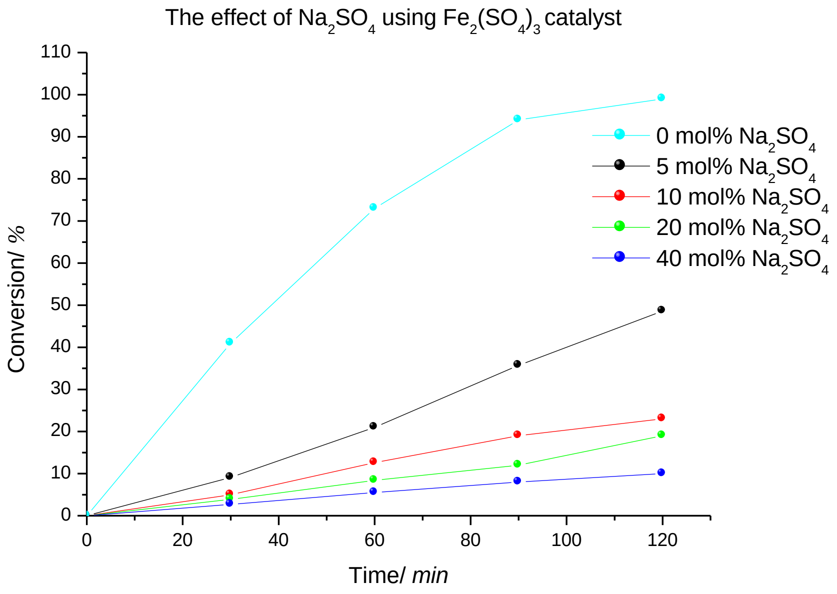
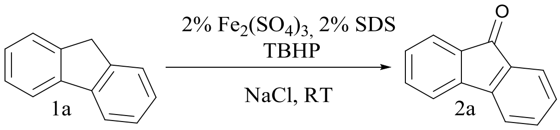
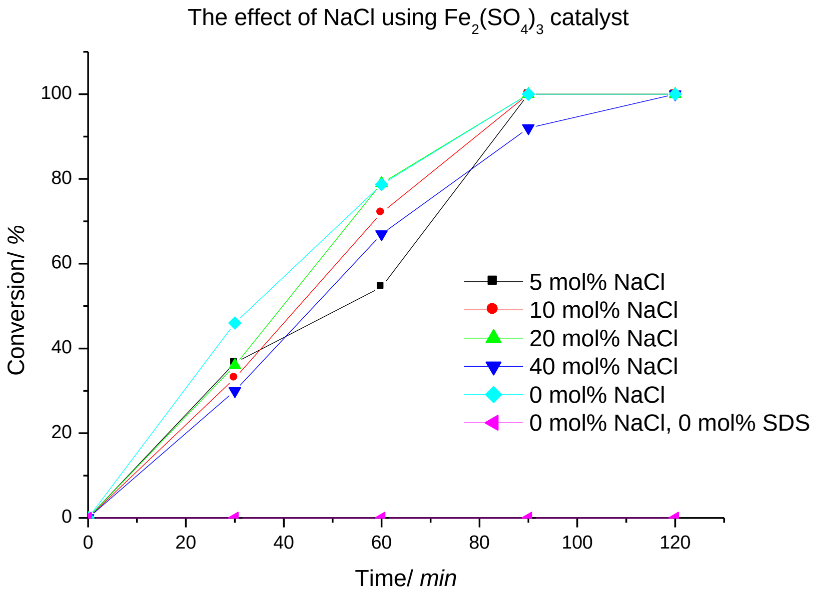
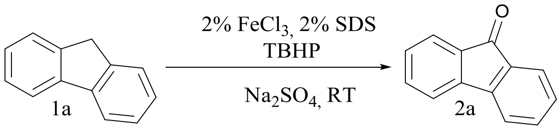
The influence of sulfate, chloride ions

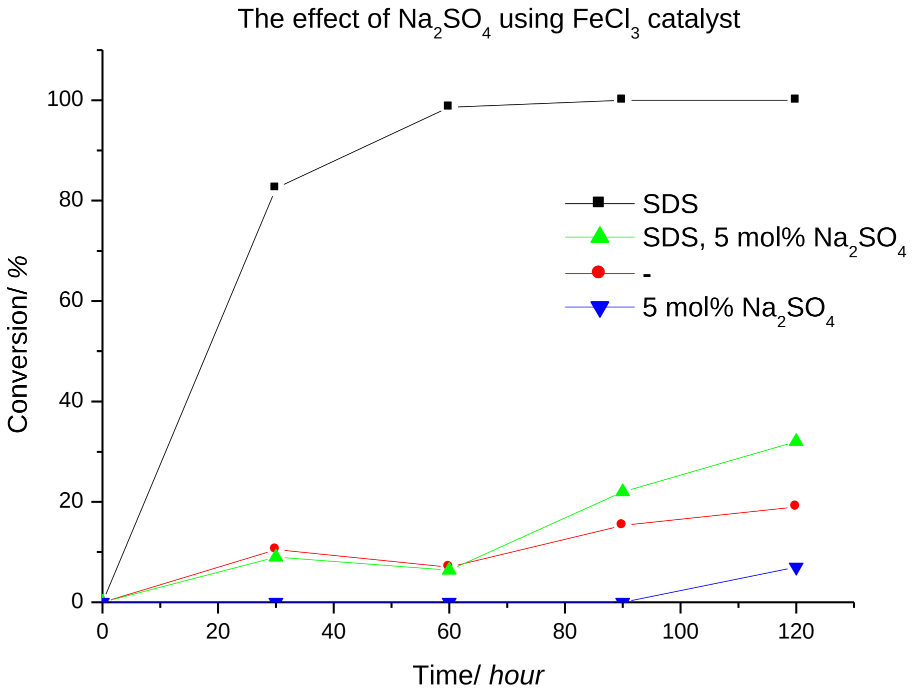






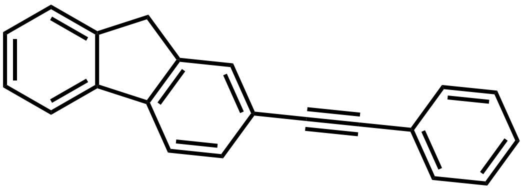






A screw capped vial with stir bar was charged with 83 mg (0.5 mmol, 1 eq.) fluorene, the 2 mol% catalyst, 2.9 mg (0.01 mmol, 2 mol%) SDS and the appropriate salt (Na_2SO_4 or NaCl). 0.5 mL water was added followed by 323 μL TBHP (2.5 mmol, 5 eq., 70% aq.). The reaction vessel was closed, and stirred for 2 hours at 50°C. Samples were taken and analyzed with GC.

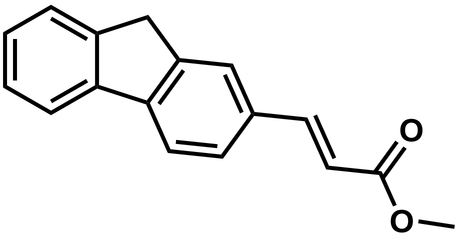
Preparation of starting materials



2-(phenylethynyl)-9*H*-fluorene[1]

A round-bottom flask was charged with 292 mg (1 mmol, 1 eq.) 2-iodo-9H-fluorene, 21 mg (0.03 mmol, 3 mol%) bis(triphenylphosphine)palladium(II) dichloride, and 5.7 mg (0.03 mmol, 3 mol%) copper(I) iodide. The flask was fit with a rubber septum, and purged with argon. DIPA (4 mL) and 165 µL phenylacetylene (1.5 mmol, 1.5 eq.) was added via syringe and the reaction was stirred at 50 °C for 24 hours. The reaction mixture was filtered, than diethyl ether was added. The solution washed with 10 % HCl (aq.) and water, dried with $MgSO_4$, and concentrated under vacuo. The crude product was purified with column chromatography (in hexane-EtOAc).

White solid. 113 mg (0.42 mmol, yield: 42%) Rf: 0.65 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.82-7.76 (m, 3H), 7.64-7.57 (m, 4H), 7.46-7.33 (m, 5H), 3.92 (s, 2H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 143.4, 143.1, 141.7, 141, 131.5, 130.4, 128.30, 128.08, 127.07, 126.83, 125.00, 123.39, 121.16, 120.12, 119.7, 90.2, 89.3, 36.7 ppm. ν_{max} 2919, 1451, 832, 753, 730, 670 cm^{-1} MS (EI, 70 eV): *m/z* (%): 266 (100, [M$^+$]), 189 (10), 133 (15). m.p.: 164-168°C



(E)-methyl 3-(9*H*-fluoren-2-yl)acrylate[2]

A round-bottom flask was charged with 584 mg (2 mmol, 1 eq.) 2-iodo-9*H*-fluorene, and 22.4 mg (0.1 mmol, 5 mol%) palladium(II) acetate. The flask was fitted with a rubber septum, and purged with argon. DMF (2mL), 418 µL TEA (3 mmol, 1.5 eq.) and 182 µL (3 mmol, 1.5 eq.) methyl acrylate was added via syringe and the reaction was stirred at 80 °C for 24 hours. After completion, the reaction mixture was filtered, and poured onto ice. The precipitated crude product was filtered, and purified by column chromatography (in hexane-EtOAc).

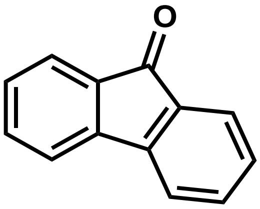
White solid. 309 mg (1.2 mmol, yield: 62%) Rf: 0.48 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.68-7.61 (m, 3H), 7.55 (s, 1H), 7.42 (t, 2H *J* = 6.16 Hz), 7.30-7.19 (m, 2H), 6.36 (d, 1H *J* = 15.95 Hz), 3.76 (s, 2H), 3.71 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 168.1, 145.7, 144.4, 144.3, 144.2, 141.3, 133.3, 127.9, 127.9, 127.4, 125.6, 124.9, 120.8, 120.6, 117.1, 52.1, 37.2 ppm. ν_{max} 2946, 2928, 1709, 1636, 1322, 1169, 977, 836, 741 cm^{-1} MS (EI, 70 eV): *m/z* (%): 250 (100, [M$^+$]), 218 (75), 189 (86), 165 (42), 94 (45). m.p.: 134-137°C

General procedure for Fe catalyzed benzylic oxidation:

A test tube with a septum cap and a stir bar was charged with the catalyst, SDS and water. The starting material and TBHP was added with stirring. The reaction vessel was closed and the reaction was stirred for x hours at 50°C. After cooling to ambient temperature, the solution was washed with EtOAc, dried over magnesium sulfate and concentrated under vacuo. The crude product was purified with chromatography (in hexane/EtOAc).

Substrate	Catalyst	SDS/ mol%	TBHP/ eq.	Time/ h	Conversion in 5 h	
					SDS	without SDS
fluorene	2 mol% $FeCl_3$	2	5	2h	100	20
2-ethyl tiophene				3h	63	50
9H-xanthene				24h	100	36
9,10-dihydroanthracene			10	24h	100	40
2-(phenylethynyl)-9H-fluorene	2 mol% $Fe_2(SO_4)_3$	2	5 + 5 after 24h	48h	25	4
(E)-methyl 3-(9H-fluoren-2-yl)acrylate				48h	74	16
2-iodo-9H-fluorene			5	24h	52	0
methyl 7-iodo-9H-fluorene-4-carboxylate				24h	100	7
diphenylmethane				24h	93	34
2-bromo-9H-fluorene	2 mol% $Fe_2(SO_4)_3$	4	5 + 4 after 5 h	24h	90	16
2,7-dibromo-9H-fluorene					9	5
(methoxymethyl)benzene	2.5 mol% $Fe_2(SO_4)_3$	5	5	24h	49	2
2-benzylphenyl acetate					58	47
N-(2-benzylphenyl)acetamide					87	39
propylbenzene					49	27
butylbenzene					47	26
ethylbenzene					72	72
methyl 2-phenylacetate			5 + 5 after 8 h		40	11
1-ethyl-4-methoxybenzene	5 mol% $FeCl_3$	5	5 + 5 after 1.5 h	2.5h	73	24
isochroman			5	10 min	92	27
1,3-dihydroisobenzofuran			5	10 min	100	35
2-phenylacetonitrile	5 mol% $Fe_2(SO_4)_3$	5	6	24	100	18
1-tosyl-1,2,3,4-tetrahydroquinoline			5 + 1 after 8h	24	22	3
1-benzylindole			5	24	8	5
2,3-cyclopentenopyridine					3	0
1,2,3,4-tetrahydronaphthalene					67	29
benzyl alcohol					93	0

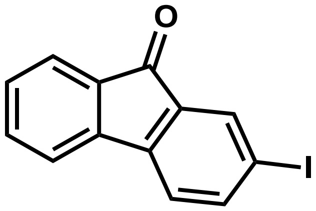
Products



Fluorene-9-one(2a)[3]

A conical test tube was charged with $FeCl_3$ (5.4 mg, 0.02 mmol, 2 mol%), SDS (5.8 mg, 0.02 mmol, 2 mol%), and 1 mL of water. Then fluorene (166 mg, 1 mmol) was added followed by TBHP (0.65 mL, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 2 hours at 50 °C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

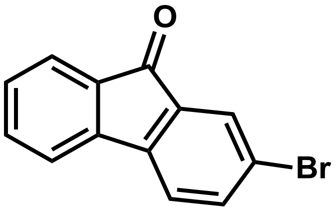
Yellow solid 180 mg (1 mmol, quantitative yield). Rf: 0.50 (in hexane:EtOAc = 5:1) ^1H NMR (250 MHz, $CDCl_3$): δ = 7.45 (t, 8H, *J* = 46.0 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 193.8, 144.3, 134.6, 128.9, 124.1, 120.2 ppm. ν_{max} 1710, 1597, 1449, 1295, 1149, 916, 731 440 cm^{-1} MS (EI, 70 eV): *m/z* (%): 180 (100, [M^+]), 152 (50), 126 (15), 76 (25), 63 (10). m.p. 79,5-80 °C (lit.): 80-83 °C



2-iodo-9*H*-fluoren-9-one (2b)[4]

A conical test tube was charged with $Fe_2(SO_4)_3$ (1.6 mg, 0.006 mmol, 2 mol%), SDS (1.7 mg, 0.006 mmol, 2 mol%), and 0.34 mL of water. Then 2-iodo-9*H*-fluorene (100 mg, 0.3 mmol) was added followed by TBHP (0.19 mL, 1.5 mmol). The reaction vessel was closed with rubber septa, and stirred for 5 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

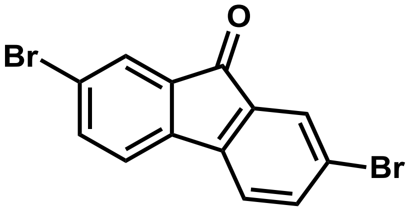
Yellow solid 83 mg (0.27 mmol, yield: 79%). Rf: 0.51 (in hexane:EtOAc = 5:1) ^1H NMR (250 MHz, $CDCl_3$): δ = 7.90 (s, 1H), 7.76 (dd, 1H J_1 = 1.58 Hz, J_2 = 7.74 Hz), 7.61 (d, 1H *J* = 7.42 Hz) 7.48-7.43 (m, 2H), 7.34-7.28 (m, 1H), 7.21 (d, 1H *J* = 7.74 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 192.3, 143.6, 143.5, 143.0, 135.6, 134.9, 133.1, 129.5, 124.4, 121.9, 120.4, 93.9 ppm. ν_{max} 2921, 2851, 1713, 1587, 1437, 1403, 1254, 1184, 1106, 816, 758, 731, 654, 453 cm^{-1} MS (EI, 70 eV): *m/z* (%): 306 (75, [M^{+}]), 151 (100), 139 (25), 75 (30). m.p. 141-143°C (lit.): 142-144 °C



2-bromo-9*H*-fluoren-9-one (2d)[3]

A conical test tube was charged with $Fe_2(SO_4)_3$ (16 mg, 0.04 mmol, 2 mol%), SDS (23 mg, 0.08 mmol, 4 mol%), and 2 mL of water. Then 2-bromo-9*H*-fluorene (488 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 4 hours at 50°C, then TBHP (1.0 ml, 8 mmol, 4 eq.) was added and the reaction mixture was stirred for further 20 hours (24 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

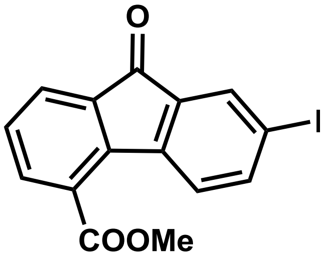
Yellow solid 445 mg (1.72 mmol, yield: 86%). Rf: 0.77 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.59 (d, 1H *J* = 1.74), 7.51 (d, 1H *J* = 7.42), 7.46 (dd, 1H J_1 = 1.90 J_2 = 7.90), 7.38-7.35 (m, 2H), 7.24-7.16 (m, 2H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 192.7, 144.0, 143.3, 137.4, 136.1, 135.4, 134.0, 129.8, 127.8, 124.9, 123.3, 122.1, 120.8 ppm. ν_{max} 1714, 1592, 1441, 1185, 818, 733, 658, 456 cm^{-1} MS (EI, 70 eV): *m/z* (%): 258(80, [M^{+}]), 151(100), 75(20). m.p. 139-141 °C (lit.): 146-148 °C



2,7-dibromo-9*H*-fluoren-9-one (2e) [5,6]

A conical test tube was charged with $Fe_2(SO_4)_3$ (16 mg, 0.04 mmol, 2 mol%), SDS (23 mg, 0.08 mmol, 4 mol%), and 2 mL of water. Then 2,7-dibromo-9*H*-fluorene (648 mg, 2 mmol) was added followed by TBHP (1.3 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 4 hours at 50°C, then TBHP (1 ml, mmol, 4 eq.) was added and the reaction mixture was stirred for further 20 hours (24 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

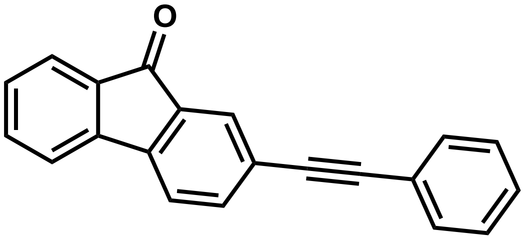
Yellow solid 645 mg (1.9 mmol, yield: 95% Rf: 0.63 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.70 (s, 2H), 7.58 (dd, 2H, J_1 = 1.74 Hz J_2 = 8 Hz). 7.34 (d, 2H J = 8.10 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 190.8, 142.2, 137.4, 135.2, 127.7, 123.2, 121.8 ppm. ν_{max} 3080, 1719, 1590, 1443, 1242, 1178, 1052, 902, 821, 680, 589, 470 cm^{-1} MS (EI, 70 eV): *m/z* (%): 338 (75, [M$^+$]), 231 (20), 150 (100), 98 (23), 75 (42). m.p.: 200-201°C (lit.): 203-205°C



Methyl 7-iodo-9-oxo-9*H*-fluorene-4-carboxylate (2f)

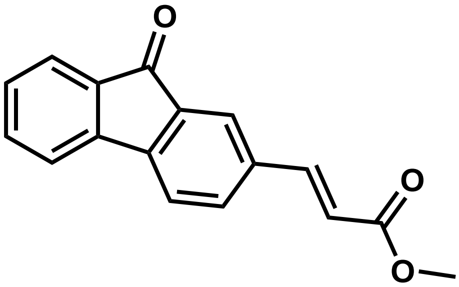
A conical test tube was charged with $Fe_2(SO_4)_3$ (1.4 mg, 0.0034 mmol, 2 mol%), SDS (1 mg, 0.0034 mmol, 2 mol%), and 0.17 mL of water. Then methyl 7-iodo-9*H*-fluorene-4-carboxylate (60.6 mg, 0.17 mmol) was added followed by TBHP (0.11 mL, 0.85 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

Yellow solid 50.6 mg (0.14 mmol, yield: 80%). Rf: 0.50 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.07-7.78 (m, 5H), 7.37 (t, 1H J = 7.50 Hz), 3.99 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 191.5, 166.7, 143.5, 142.3, 136.5, 136.5, 135.8, 134.7, 132.9, 129.0, 128.0, 127.5, 126.8, 95.4, 52.6 ppm. ν_{max} 2923, 2851, 1717, 1579, 1433, 1292, 1272, 1242, 1193, 1175, 1137, 989, 833, 754, 740 cm^{-1}. MS (EI, 70 eV): *m/z* (%): 364 (98, [M$^+$]), 333(42), 305 (40), 207 (60), 150 (100), 138 (25), 75 (27). m.p. 180°C HRMS calcd. for $C_{15}H_9IO_3$ [M+H]$^+$ 364.9669 found 364.9676



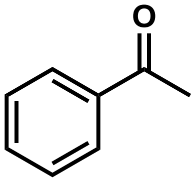
2-(phenylethynyl)-9*H*-fluoren-9-one (2g)[7]

Yellow solid. 36 mg (0.13 mmol, yield: 64%) Rf: 0.51 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.69 (d, 1H J = 0.95 Hz), 7.58-7.51 (m, 2H), 7.47-7.37 (m, 5H), 7.30-7.17 (m, 4H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 193.0, 143.9, 143.6, 137.6, 134.8, 134.2, 134.1, 131.6, 129.3, 128.5, 128.4, 127.2, 124.4, 124.1, 122.7, 120.6, 120.2, 91.0, 88.6 ppm. ν_{max} 2917, 2850, 1714, 1600, 755, 733, 688 cm^{-1} MS (EI, 70 eV): *m/z* (%): 280 (100, [M^+]), 250 (30), 207 (40), 140 (15). m.p.: 140-143 °C



(E)-methyl 3-(9-oxo-9*H*-fluoren-2-yl)acrylate (2h)

Yellow solid. 140 mg (0.53mmol, yield: 53%) Rf: 0.29 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.74 (s, 1H), 7.64 – 7.47 8m, 6H), 7.31-7.28 (m, 1H), 6.42 (d, 1H), 3.79 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 192.9, 167.0, 145.6, 143.6, 143.2, 135.3, 134.9, 134.8, 134.6, 134.4, 129.6, 124.4, 122.7, 120.7, 118.5, 51.8 ppm. ν_{max} 3059, 2917, 1706, 1580, 1437, 1317, 1169, 1004, 840, 719 cm^{-1} MS (EI, 70 eV): *m/z* (%): 264 (100, [M^+]), 233 (98), 205 (40), 176 (75), 151 (42), 88 (45). m.p.: 169-171 °C

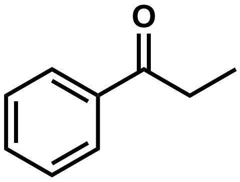


Acetophenone (2i)[3]

A conical test tube was charged with $Fe_2(SO_4)_3$ (20 mg, 0.05 mmol, 2.5 mol%), SDS (28.8 mg, 0.1 mmol, 5 mol%), and 2 mL of water. Then ethylbenzene (245 µl, 212 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50 °C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

A conical test tube was charged with $Fe_2(SO_4)_3$ (40 mg, 0.1 mmol, 5 mol%), SDS (28.8 mg, 0.1 mmol, 5 mol%), and 2 mL of water. Then benzyl-acohol (207.2 µl, 216.3 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

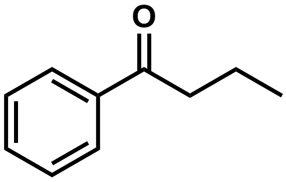
Colorless oil 180 mg (1.5 mmol, yield: 75%, from ethylbenzene), 235 mg (1.96 mmol, yield: 98% from 1-phenyl-1-ethanol) Rf: 0.49 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.89-7.86 (m, 2H), 7.51-7.348 (m, 3H), 2.52 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.2, 137.0, 133.0, 128.5, 26.5 ppm. ν_{max} 2926, 2855, 1684, 1599, 1467, 1358, 1262, 954, 758, 688, 587 cm^{-1} MS (EI, 70 eV): *m/z* (%): 120 (32, [M$^+$]), 105 (100), 77 (85), 51 (45).



Propiophenone (2j)[8]

A conical test tube was charged with $Fe_2(SO_4)_3$ (5 mg, 0.0125 mmol, 2.5 mol%), SDS (14.4 mg, mmol, 5 mol%), and 355 μl of water. Then propylbenzene (139.4 μl, 120.18 mg, 1 mmol) was added followed by TBHP (645 μl, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50 °C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

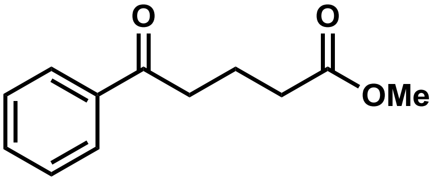
Colorless oil 56 mg (0.42 mmol, yield: 42%), Rf: 0.65 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.87 (d, 2H, J = 6.95 Hz), 7.48-7.34 (m, 3H, J = 7.42 Hz), 2.92 (q, 2H, J = 14.44 Hz), 1.14 (t, 3H, J = 7.20 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 200.2, 137.3, 133.3, 128.9, 128.3, 32.2, 8.6 ppm. ν_{max} 2978, 2938, 1685, 1449, 1319, 1218, 1180, 1014, 950, 743, 689 cm^{-1} MS (EI, 70 eV): *m/z* (%): 134 (15, [M$^+$]), 105 (100), 77 (50), 51 (15).



Butirophenone (2k)[9]

A conical test tube was charged with $Fe_2(SO_4)_3$ (5 mg, 0.0125 mmol, 2.5 mol%), SDS (14.4mg, mmol, 5 mol%), and 355 μl of water. Then butylbenzene (156.1 μl, 134.2 mg, 1 mmol) was added followed by TBHP (645 μl, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

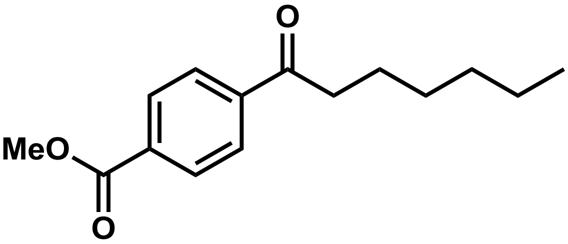
Colorless oil 63.8 mg (0.43 mmol, yield: 43%), Rf: 0.67 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.86 (d, 2H, *J* = 8.53 Hz), 8.16 (m, 3H), 2.85 (t, 2H, *J* = 7.27 Hz), 1.68 (d, 2H, *J* = 22.00 Hz), 0.92 (t, 3H, *J* = 7.42) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 200.8, 137.5, 133.2, 128.9, 18.4, 40.9, 18.1, 14.3 ppm. ν_{max} 2962, 2933, 2874, 1683, 1448, 1315, 1272, 1212, 1001, 753, 745, 689 cm^{-1} MS (EI, 70 eV): *m/z* (%): 148 (15, [M$^+$]), 120 (10), 105 (100), 77 (45), 51 (15).



Methyl 5-oxo-5-phenylpentanoate (2l)[10]

A conical test tube was charged with $Fe_2(SO_4)_3$ (5mg, 0.0125mmol, 2.5 mol%), SDS (14.4 mg, mmol, 5 mol%), and 355 μl of water. Then 5-phenylvaleric acid (192.2 mg, 1 mmol) was added followed by TBHP (645 μl, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 48 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

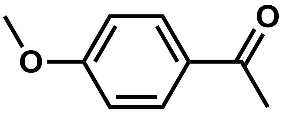
Colorless oil 56 mg (0.42 mmol, yield: 42%), Rf: 0.36 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.88 (d, 2H, *J* = 7.11 Hz), 7.52-7.35 (m, 3H), 3.61 (s, 3H), 2.98 (t, 2H, *J* = 7.19 Hz), 2.38 (t, 2H, *J* = 7.19 Hz), 2.00 (quint., 2H, *J* = 7.30) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 200.0, 174.1, 137.2, 133.5, 129.0, 128.4, 52.0, 37.8, 33.5, 19.7 ppm. ν_{max} 2951, 1732, 1682, 1448, 1437, 1366, 1209, 1175, 1149, 1015, 742, 690 cm^{-1} MS (EI, 70 eV): *m/z* (%): 206 (2, [M$^+$]), 175 (5), 147 (6), 133 (5), 120 (12), 105 (100), 77 (40).



Methyl 4-heptanoylbenzoate (2m)[11]

A conical test tube was charged with $Fe_2(SO_4)_3$ (5 mg, 0.0125 mmol, 2.5 mol%), SDS (14.4 mg, mmol, 5 mol%), and 355 μl of water. Then 4-heptylbenzoic acid (234.3 mg, 1 mmol) was added followed by TBHP (645 μl, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 48 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

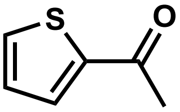
White solid 100.8 mg (0.40 mmol, yield: 40%), Rf: 0.53 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.08 (2, 2H, *J* = 8.37 Hz), 7.97 (d, 2H, *J* = 8.53 Hz), 3.91 (s, 3H), 2.95 (t, 2H, *J* = 7.35 Hz), 1.70 (quint., 2H, *J* = 14.45 Hz), 1.36-1.25 (m, 6H), 0.85 (t, 3H, *J* = 6.64 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 200.3, 166.6, 140.7, 134.0, 1301, 128.3, 52.8, 39.3, 32.0, 29.3, 24.5, 22.8, 14.4 ppm. ν_{max} 2957, 2929, 2871, 1720, 1675, 1435, 1278, 1239, 1194, 1108, 958, 764, 687 cm^{-1} MS (EI, 70 eV): *m/z* (%): 248 (3, [M$^+$]), 217 (5), 189 (15), 178 (80), 163 (100), 147 (20), 135 (25), 120 (10), 104 (15), 76 (10). m.p.: 73-75°C



1-(4-methoxyphenyl)ethanone (2n)[12]

A conical test tube was charged with $FeCl_3$ (27 mg, 0.1 mmol, 5 mol%), SDS (28.8 mg, 0.1 mmol, 5 mol%), and mL of water. Then 1-ethyl-4-methoxybenzene (283 µl, 272 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 90 min at 50°C, then TBHP (1.29 ml, 10 mmol, 5 eq) was added and the reaction mixture was stirred for further 1 hour (2.5 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

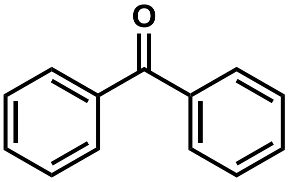
White solid 115 mg (0.77 mmol, yield: 40%) Rf: 0.21 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.82 (d, 2H *J* = 8.85 Hz), 6.81 (d, 2H, *J* = 8.85 Hz), 3.74 (s, 3H), 2.43 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 196.5, 163.3, 130.3, 113.4, 55.2, 26.1 ppm. ν_{max} 2965, 2842, 1666, 1597, 1356, 1247, 1019, 830, 575 cm^{-1} MS (EI, 70 eV): *m/z* (%): 150 (40, [M$^+$]), 135 (100), 107 (22), 92 (30), 77 (40). m.p. 32-35 °C (lit.): 37-39 °C



1-(thiophen-2-yl)ethanone (2p)[13]

A conical test tube was charged with $FeCl_3$ (16.2 mg, 0.06 mmol, 2 mol%), SDS (17.4 mg, 0.06 mmol, 2 mol%), and 3 mL of water. Then 2-ethylthiophene (340 µl, 224.3 mg, 3 mmol) was added followed by TBHP (1.94 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 3 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

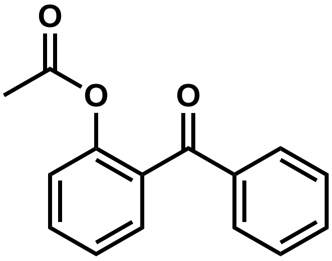
Brown oil 123 mg (0.98 mmol, yield: 33%) Rf: 0.58 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.66 (dd, 1H J_1 = 1.11 Hz, J_2 = 3.79 Hz), 7.60 (dd, 1H J_1 = 1.11 Hz, J_2 = 5.06 Hz), 7.09 (dd, 1H J_1 = 4.90 Hz, J_2 = 3.79 Hz), 2.52 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 191.1, 144.9, 134.2, 132.9, 128.5 ppm. ν_{max} 2978, 2930, 1660, 1413, 1357, 1271, 857, 721, 590 cm^{-1} MS (EI, 70 eV): *m/z* (%): 126 (40, [M$^+$]), 111 (100), 83 (15), 57 (12).



Benzophenone (2q)[3]

A conical test tube was charged with $Fe_2(SO_4)_3$ (16 mg, 0.04 mmol, 2 mol%), SDS (11.6 mg, 0.04 mmol, 2 mol%), and 3 mL of water. Then diphenylmethane (333 µl, 336.5 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

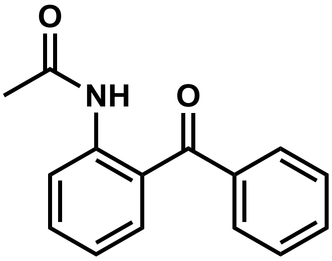
Colorless oil 326 mg (1.79 mmol yield: 90 %) Rf: 0.53 (in hexane:EtOAc = 3:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.71 (d, 4H *J* = 6.79 Hz), 7.50 (t, 2H J = 7.35 Hz), 7.39 (t, 4H, *J* = 7.27 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 196.6, 137.4, 132.3, 129.9, 128.2 cm^{-1} ν_{max} 3060, 1655, 1446, 1274, 940, 918, 694, 637, 437 cm^{-1} MS (EI, 70 eV): *m/z* (%): 182 (75, [M$^+$]), 105 (100), 77 (72), 51 (30).



2-benzoylphenyl acetate (2r)[14]

A conical test tube was charged with $Fe_2(SO_4)_3$ (10 mg, 0.025 mmol, 2.5 mol%), SDS (14.4 mg, 0.05 mmol, 5 mol%), and 1 mL of water. Then 2-benzylphenyl acetate (226 mg, 1 mmol) was added followed by TBHP (645 µL, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

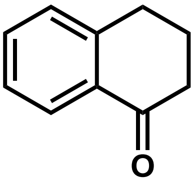
Yellow oil 68 mg (0.28 mmol, yield: 28% Rf: 0.72 (in hexane:EtOAc =2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.77 (d, 2H *J* = 7.27 Hz), 7.55 (t, 2H *J* = 6.48 Hz), 7.45 (t, 2H *J* = 7.35 Hz), 7.33 (t, 1H *J* = 7.11 Hz), 7.20 (d, 1H *J* = 7.90 Hz), 1.94 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 194.8, 169.1, 148.7, 137.5, 133.0, 132.2, 131.4, 130.4, 129.7, 128.3, 125.6, 123.2, 20.5 ppm. ν_{max} 1765, 1662, 1448, 1184, 1102, 1009, 906, 761, 698, 633 cm^{-1} MS (EI, 70 eV): *m/z* (%): 240 (5, [M^+]), 197 (100), 121 (45), 105 (30), 77 (43).



N-(2-benzoylphenyl)acetamide (2s)[15]

A conical test tube was charged with $Fe_2(SO_4)_3$ (10 mg, 0.025 mmol, 2.5 mol%), SDS (14.4 mg, 0.05 mmol, 5 mol%), and 1 mL of water. Then N-(2-benzylphenyl)acetamide (225 mg, 1 mmol) was added followed by TBHP (645 μL, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

Off-white solid 148 mg (0.62 mmol, yield: 62% Rf: 0.51 (in hexane:EtOAc =2:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 10.84 (s, 1H), 8.64 (d, 1H *J* = 8.21 Hz), 7.71 (d, 2H *J* = 7.11 Hz), 7.63-7.46 (m, 5H), 7.09 (t, 1H *J* = 7.58 Hz), 2.23 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 199.5, 169.0, 140.3, 138.4, 134.1, 133.3, 132.4, 129.7, 128.2, 123.1, 121.9, 121.3, 25.0 ppm. ν_{max} 1698, 1580, 1518, 1444, 1289, 1256, 1156, 919, 751, 698, 607 cm^{-1} MS (EI, 70 eV): *m/z* (%): 239 (12, [M^+]), 196 (100), 167 (15), 134 (17), 120 (40), 105 (12), 92 (15), 77 (35). m.p.: 75-77°C (lit.: 81°C)

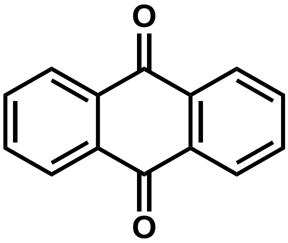


3,4-dihydronaphthalen-1(2*H*)-one (2t)[3]

A conical test tube was charged with $Fe_2(SO_4)_3$ (60 mg, 0.15 mmol, 5 mol%), SDS (43.3 mg, 0.15 mmol, 5 mol%), and 3 mL of water. Then 1,2,3,4-tetrahydronaphthalene (408.6 μl, 396.3

mg, 3 mmol) was added followed by TBHP (1.94 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

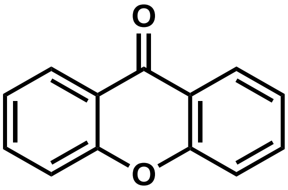
Yellow oil 114 mg (0.78 mmol, yield: 26%) Rf: 0.52 (in hexane:EtOAc = 4:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.22 (d, 1H, J = 8.06 Hz), 7.38-7.32 (m, 1H), 7.21-7.12 (m, 2H), 2.85 (t, 2H, J = 6.08 Hz), 2.54 (t, 2H, J = 6.55 Hz), 2.02 (q, 2H, J = 12.64 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 198.7, 144.9, 133.8, 133.0, 129.1, 127.5, 127.0, 39.6, 30.1, 23.7 ppm. ν_{max} 2976, 2933, 1681, 1599, 1299, 1284, 762, 734, 553 cm^{-1} MS (EI, 70 eV): *m/z* (%): 146 (70, [M$^+$]), 131 (15), 118 (100), 103 (2), 90 (70), 77 (5)



Anthracene-9,10-dione (2v)[16]

A conical test tube was charged with $FeCl_3$ (10.8 mg, 0.04 mmol, 2 mol%), SDS (11.5 mg, 0.04 mmol, 2 mol%), and 2 mL of water. Then 9,10-dihydroanthracene (360 mg, 2 mmol) was added followed by TBHP (2.58 mL, 20 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

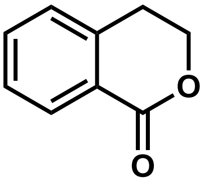
Off-white solid 445 mg (2.1 mmol, yield: 88%) Rf: 0.39 (in hexane:EtOAc = 3:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.32 (s, 4H), 7.81 (s, 4H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 183.1, 134.1, 133.5, 127.2 ppm. ν_{max} 1673, 1573, 1281, 1168, 935, 808, 691, 620 cm^{-1} MS (EI, 70 eV): *m/z* (%): 208 (100, [M$^+$]), 180 (100), 152 (100), 126 (15), 76 (42). m.p. 279-281°C



9*H*-xanthen-9-one (2w)[3]

A conical test tube was charged with $FeCl_3$ (10.8 mg, 0.04 mmol, 2 mol%), SDS (11.5 mg, 0.04 mmol, 2 mol%), and 2 mL of water. Then 9*H*-xanthene (364 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

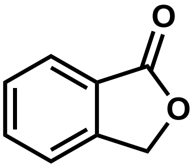
Off-white solid 380 mg (1.94 mmol, yield: 97%) Rf: 0.39 (in hexane:EtOAc = 3:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.32 (d, 2H *J* = 7.74 Hz), 7.70 (t, 2H, *J* = 7.5 Hz), 7.46 (d, 2H *J* = 8.06 Hz), 7.35 (t, 2H *J* = 7.19 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 177.1, 156.0, 134.7, 126.6, 123.8, 121.7, 117.9 ppm. ν_{max} 1653, 1615, 1478, 1454, 1329, 1144, 755, 670, 625 cm^{-1} MS (EI, 70 eV): *m/z* (%): m.p. 174 °C (lit.): 172-174°C



Isochroman-1-one (2x)[3]

A conical test tube was charged with $FeCl_3$ (40.5 mg, 0.15 mmol, 5 mol%), SDS (43.3 mg, 0.15 mmol, 5 mol%), and 3 mL of water. Then isochroman (377.3 µl, 402.5 mg, 3 mmol) was added followed by TBHP (1.94 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 10 min at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

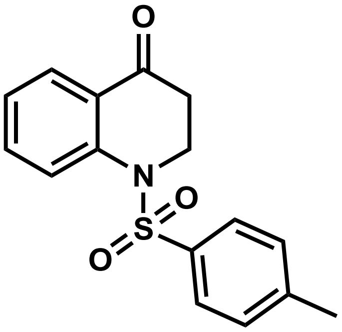
Colorless oil 123 mg (0.83 mmol, yield: 28%) Rf: 0.16 (in hexane:EtOAc = 3:1) ^{1}H NMR (250 MHz, DMSO-d_6): δ = 8.00 (d, 1H *J* = 7.58 Hz), 7.46 (t, 1H *J* = 7.50 Hz), 7.30 (t, 1H *J* = 7.424 Hz), 7.19 (d, 1H *J* = 7.58 Hz), 4.44 (t, 2H *J* = 6.00 Hz), 2.98 (t, 2H *J* = 6.00 Hz) ppm. ^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 163.4, 140.2, 133.6, 129.4, 127.6, 124.9, 67.1, 27.0 ppm. ν_{max} 2900, 1715, 1392, 1292, 1240, 1118, 1089, 1027, 743, 693, 491. MS (EI, 70 eV): *m/z* (%): 148 (50, [M$^+$]), 118 (100), 90 (85), 63 (20).



Isobenzofuran-1(3H)-one (2y)[3]

A conical test tube was charged with $FeCl_3$ (40.5 mg, 0.15 mmol, 5 mol%), SDS (43.3 mg, mmol, 5 mol%), and 3 mL of water. Then 1,3-dihydroisobenzofuran (330.7 µl, 360.5 mg, mmol) was added followed by TBHP (1.94 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 10 min at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

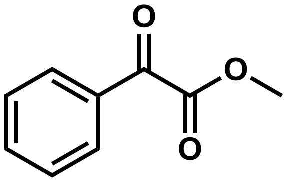
White solid 133 mg (0.99 mmol, yield: 33%) Rf: 0.53 (in hexane:EtOAc = 3:1) ^{1}H NMR (250 MHz, DMSO-d_6): δ = 7.83-7.72 (m, 2H), 7.66-7.52 (m, 2H), 5.39 (s, 2H) ppm. ^{13}C NMR (62.5 MHz, DMSO-d_6): δ = 170.64, 147.2, 134.1, 128.8, 124.8, 124.8, 122.9, 69.8 ppm. ν_{max} 2916, 1745, 1708, 1466, 1437, 1366, 1216, 1048, 999, 737, 679, 472 cm^{-1}. MS (EI, 70 eV): *m/z* (%): 134 (50, [M$^+$]), 105 (100), 77 (45), 51 (15). m.p. 72°C (lit.): 71-74°C



1-tosyl-2,3-dihydroquinolin-4(1H)-one (2z)[17]

A conical test tube was charged with $Fe_2(SO_4)_3$ (20 mg, 0.05 mmol, 5 mol%), SDS (14.4 mg, 0.05 mmol, 5 mol%), and 1 mL of water. Then 1-tosyl-1,2,3,4-tetrahydroquinoline (287.1 mg, 1 mmol) was added followed by TBHP (0.65 mL, 5 mmol). The reaction vessel was closed with rubber septa, and stirred for 8 hours at 50°C, then TBHP (0.13 ml, 1 mmol, 1 eq.) was added and the reaction mixture was stirred for further 16 hours (24 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

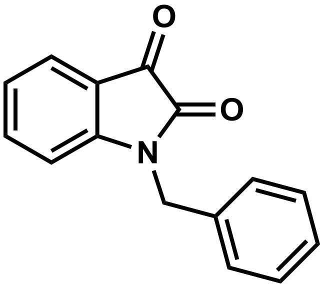
White solid 90.1 mg (0.3 mmol, yield: 30%) Rf.: 0.72 (in hexane:EtOAc = 3:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.93 (d, 1H, *J* = 7.74 Hz), 7.85 (d, 1H, *J* = 8.37 Hz), 7.59-7.53 (m, 3H), 7.29-7.20 (m, 3H), 4.22 (t, 2H, *J* = 6.40 Hz), 2.37 (t, 2H, *J* = 6.24 Hz), 2.37 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 193.1, 145.0, 142.8, 137.2, 135.1, 130.5, 128.2, 127.3, 126.1, 126.0, 125.0, 46.6, 36.9, 22.0 ppm. ν_{max} 3064, 2923, 1680, 1595, 1349, 1294, 1162, 1076, 922, 772, 728, 682, 575, 550, 539 cm^{-1}. MS (EI, 70 eV): *m/z* (%): 301 (10, [M$^+$]), 155 (20), 146 (75), 128 (4), 117 (15), 104 (4), 91 (100), 77 (10), 65 (20). m.p.: 138-140°C



Methyl 2-oxo-2-phenylacetate (2aa)[18]

A conical test tube was charged with $Fe_2(SO_4)_3$ (20 mg, 0.1 mmol, 2.5 mol%), SDS (28.8 mg, 0.1 mmol, 5 mol%), and 2 mL of water. Then methyl 2-phenylacetate (285 µl, 300.3 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 8 hours at 50°C, then TBHP (1.29 ml, 10 mmol, 5 eq.) was added and the reaction mixture was stirred for further 16 hours (24 hours total reaction time). After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

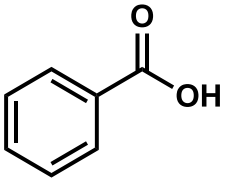
Colorless oil 264 mg (1.6 mmol, yield: 80%) Rf: 0.41 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 8.05 (dd, 2H J_1 = 7.11 Hz, J_2 = 1.42 Hz), 7.65 (t, 1H J = 7.5 Hz), 7.53-7.45 (m, 2H), 3.97 (s, 3H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 186.5, 164.0, 135.0, 132.2, 130.1, 128.8, 52.7 ppm. ν_{max} 2956, 1737, 1689, 1204, 1172, 1003, 677 cm^{-1} MS (EI, 70 eV): *m/z* (%): 164(2, [M$^+$]), 105(100), 77(52), 51(20).



1-benzyl isatin (2bb)[19]

A conical test tube was charged with $Fe_2(SO_4)_3$ (5mg, 0,025 mmol, 5 mol%), SDS (1.8 mg, mmol, 5 mol%), and 0.266 mL of water. Then 1-benzylindole (52 mg, 0.25 mmol) was added followed by TBHP (0.484mL, 0.75mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified with chromatography (hexane/EtOAc).

Orange solid. 12.3 mg (0.05 mmol, yield: 21%, Purity 92%) Rf: 0.40 (in hexane:EtOAc = 3:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 7.55 (d, 1H, J = 6.63 Hz), 7.41 (t, 1H, J = 7.82 Hz), 7.55 (d, 1H, J = 6.63 Hz), 7.28-7.19 (m, 5H), 7.02 (t, 1H, J = 7.66 Hz), 6.01 (d, 1H, J = 8.06 Hz), 4.86 (s, 2H) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 183.6, 158.7, 151.1, 138.7, 134.9, 129.4, 127.8, 124.3, 118.1, 111.4, 44.4 ppm. ν_{max} 2925, 1727, 1608, 1467, 1345, 1174, 1091, 1001, 852, 751, 691, 469 cm^{-1} MS (EI, 70 eV): *m/z* (%): 237 (35, [M$^+$]), 207 (5), 180 (40), 146 (100), 104 (10), 91 (50), 77 (10). m.p.: 110-120°C (lit.): 133-135°C



Benzoic acid (2cc)[20]

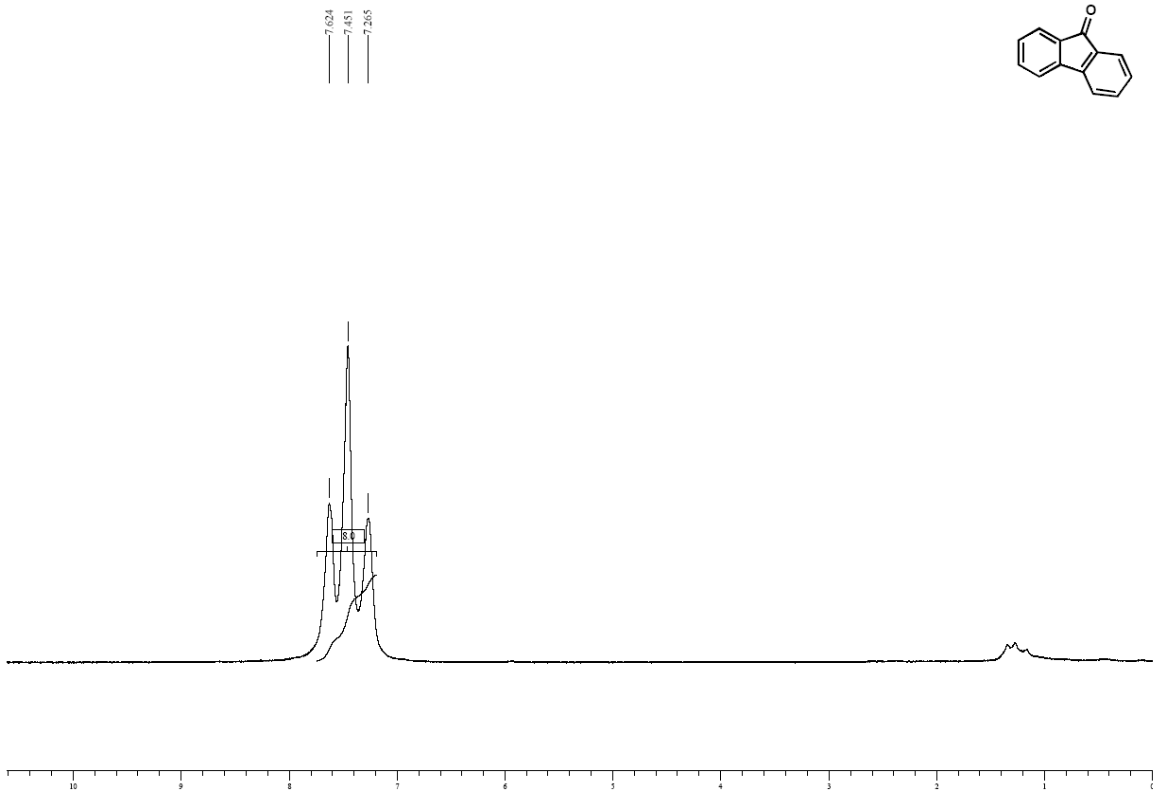
A conical test tube was charged with $Fe_2(SO_4)_3$ (30mg, 0.075mmol, 5 mol%), SDS (43.3mg, mmol, 5 mol%), and 1.1 mL of water. Then 2-phenylacetonitrile (345 µl, 351 mg, 3 mmol) was added followed by TBHP (1.9 mL, 15 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

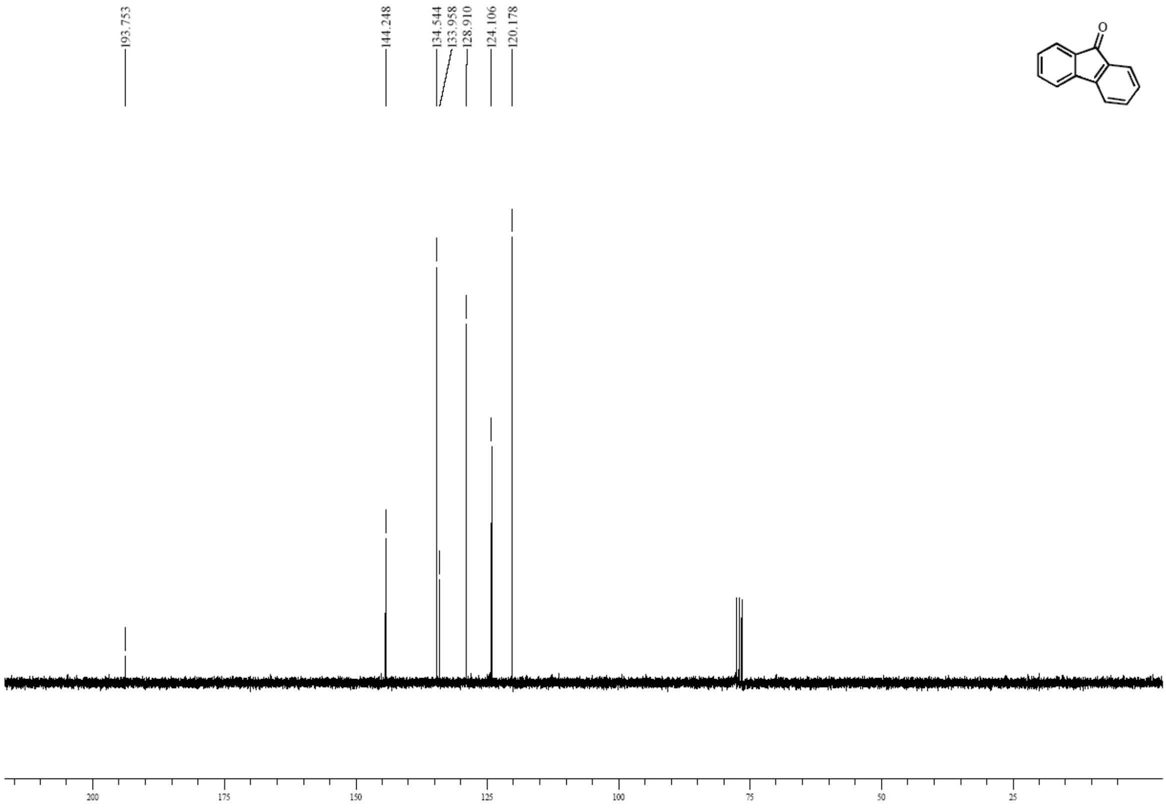
A conical test tube was charged with $Fe_2(SO_4)_3$ (20 mg, mmol, 5 mol%), SDS (28.8 mg, mmol, 5 mol%), and 0.710 mL of water. Then benzylalcohol (207 µl, 216 mg, 2 mmol) was added followed by TBHP (1.29 mL, 10 mmol). The reaction vessel was closed with rubber septa, and stirred for 24 hours at 50°C. After cooling to room temperature, the solution was washed with EtOAc, dried over magnesium sulfate, and concentrated. The crude product was purified by chromatography (hexane/EtOAc).

White solid 168 mg (1.38 mmol, yield: 46% from 2-phenylacetonitrile), 99.7 mg (0.82 mmol, yield: 41% from benzylalcohol) Rf: 0.32 (in hexane:EtOAc = 5:1) ^{1}H NMR (250 MHz, $CDCl_3$): δ = 12.71 (s, 1H), 8.16 (d, 1H, *J* = 7.42 Hz), 7.64 (t, 1H, *J* = 7.35 Hz), 7.50 (t, 1H, *J* = 7.50 Hz) ppm. ^{13}C NMR (62.5 MHz, $CDCl_3$): δ = 173.1, 134.3, 130.7, 129.8, 128.9 ppm. ν_{max} 3070, 2603, 2551, 1678, 1581, 1419, 1288, 1180, 930, 804, 704, 545 cm^{-1} MS (EI, 70 eV): *m/z* (%): 122 (90, [M$^+$]), 105 (100), 94 (5), 77 (80), 51 (40). m.p.: 121-123°C (lit.): 121-125°C

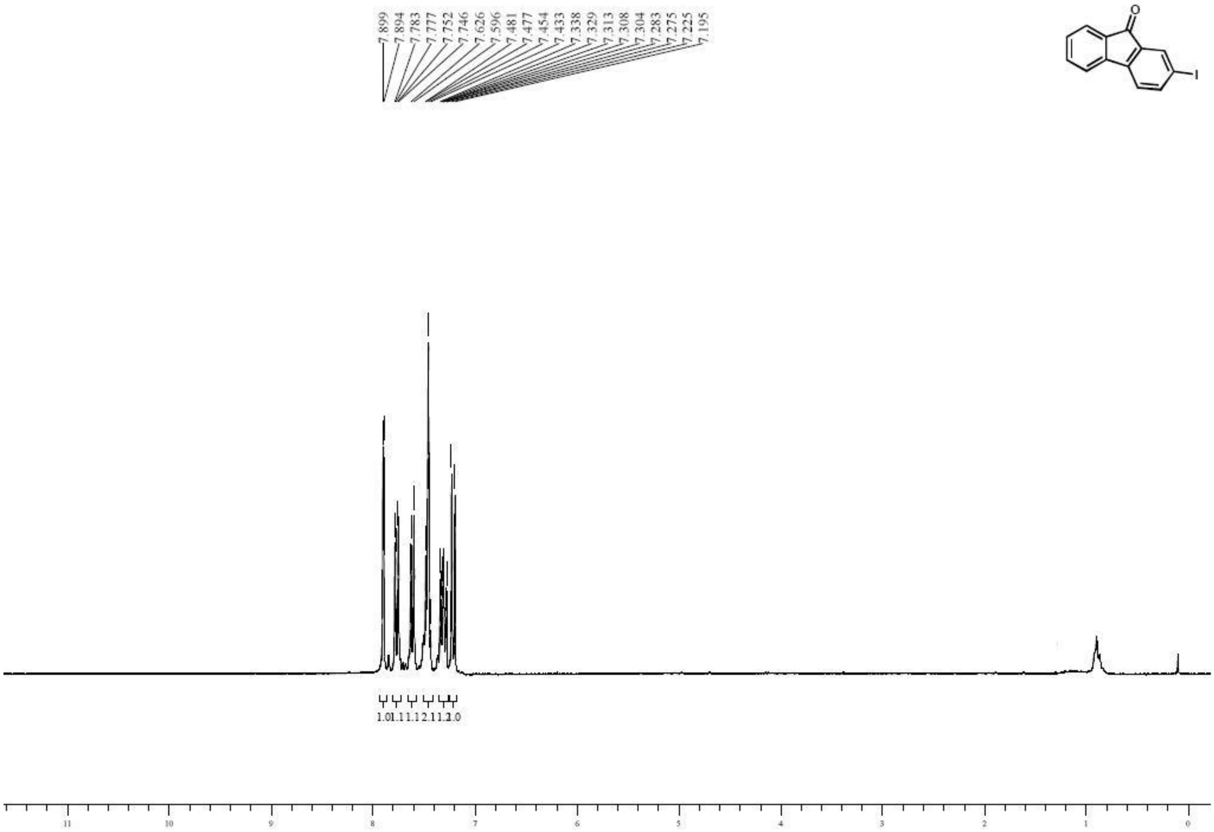
NMR spectrum

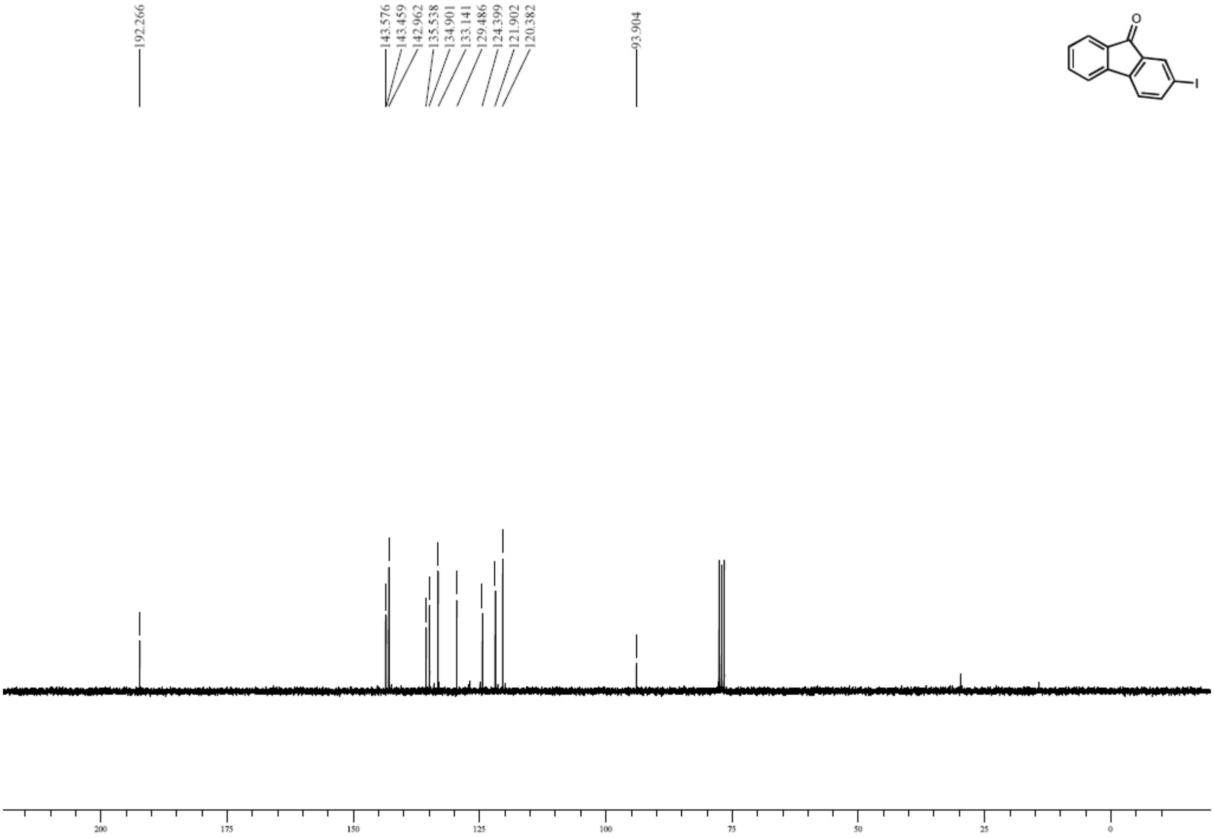
Fluorene-9-one (2a)[3]



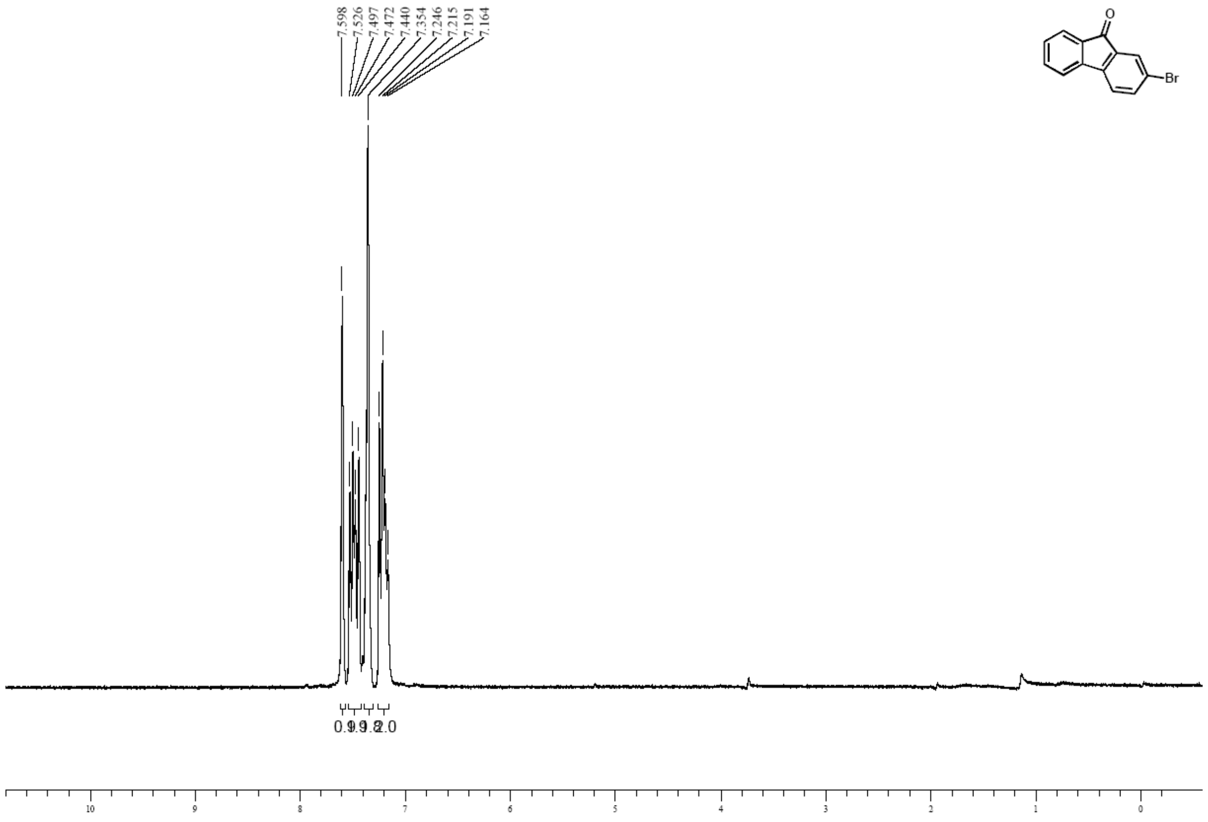


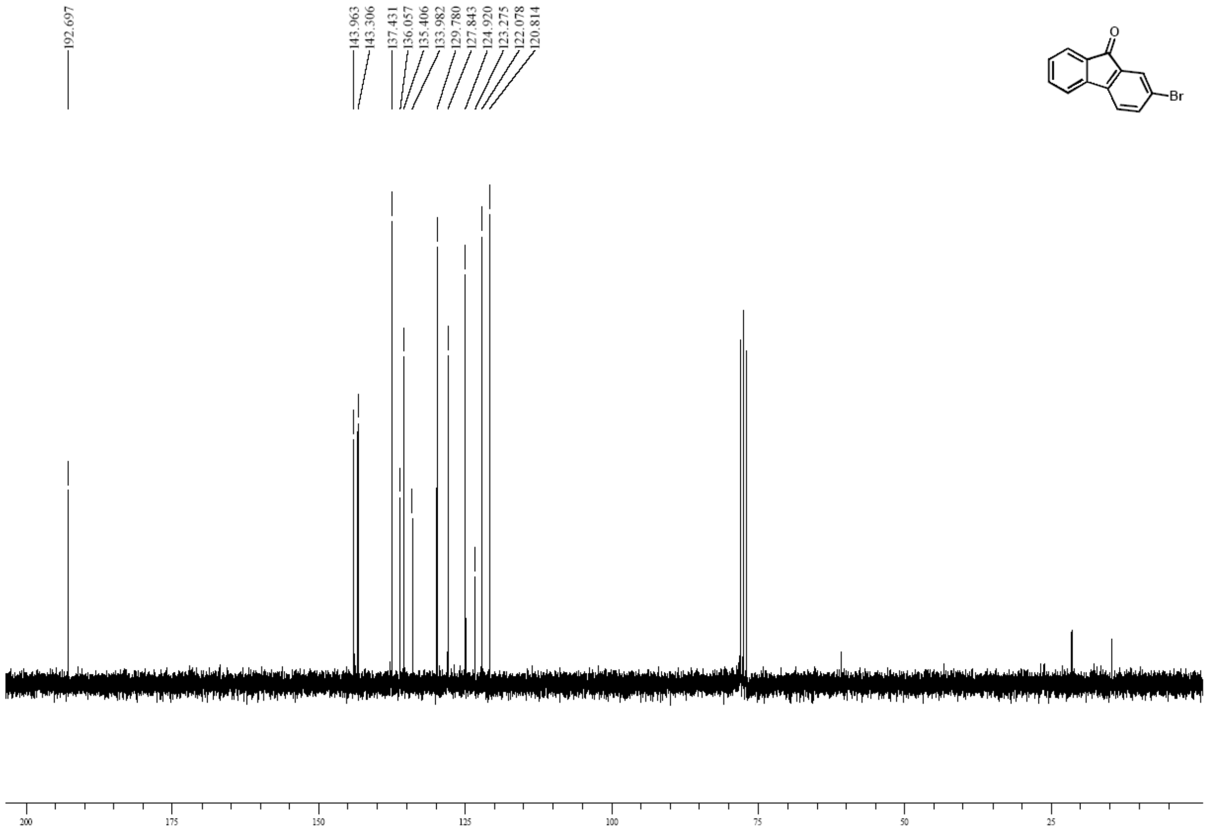
2-iodo-9*H*-fluoren-9-one (2b)[4]



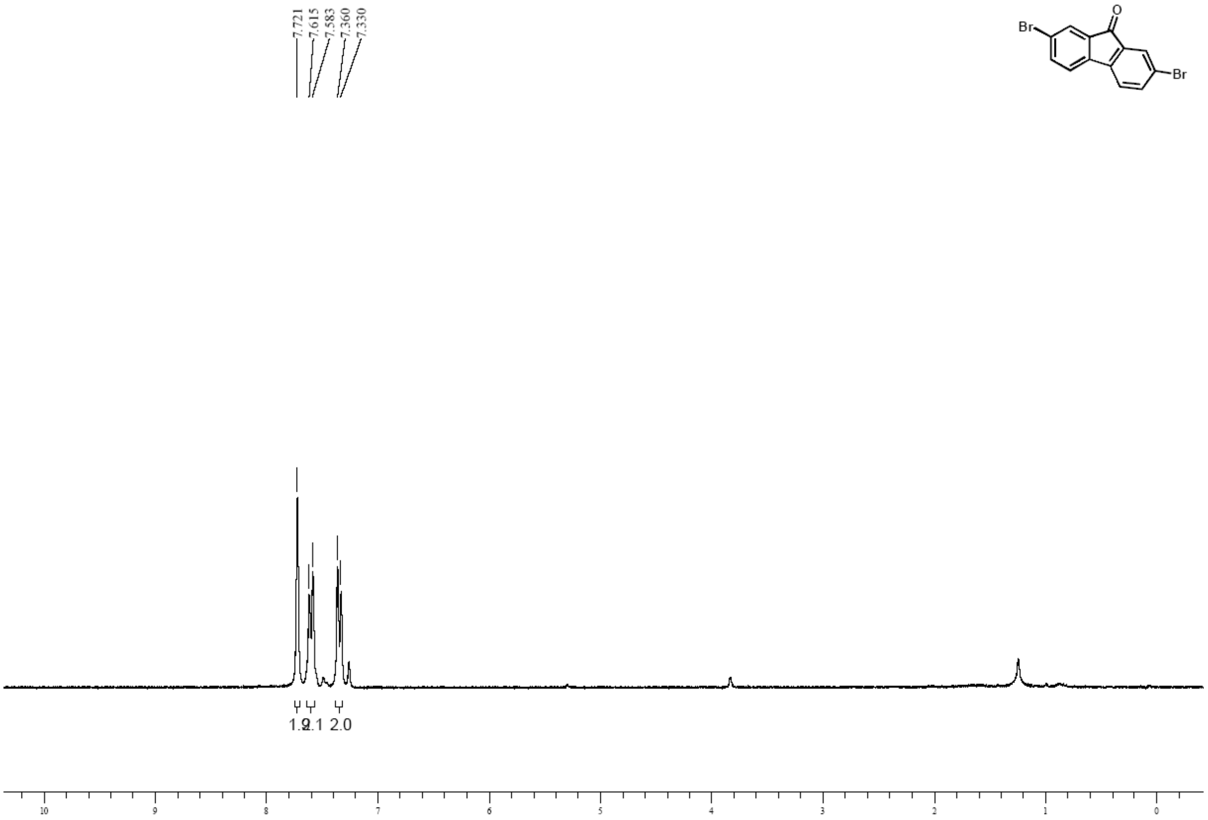


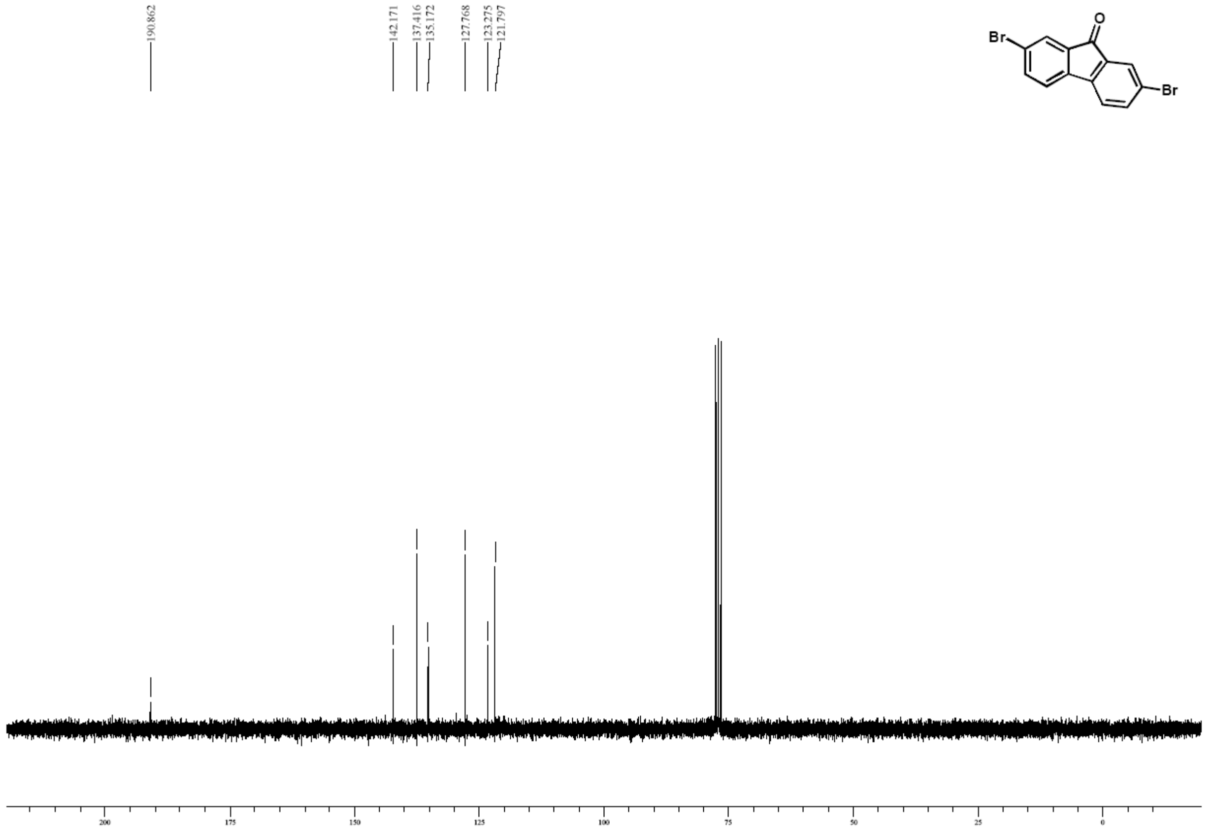
2-bromo-9*H*-fluoren-9-one (2d)[3]



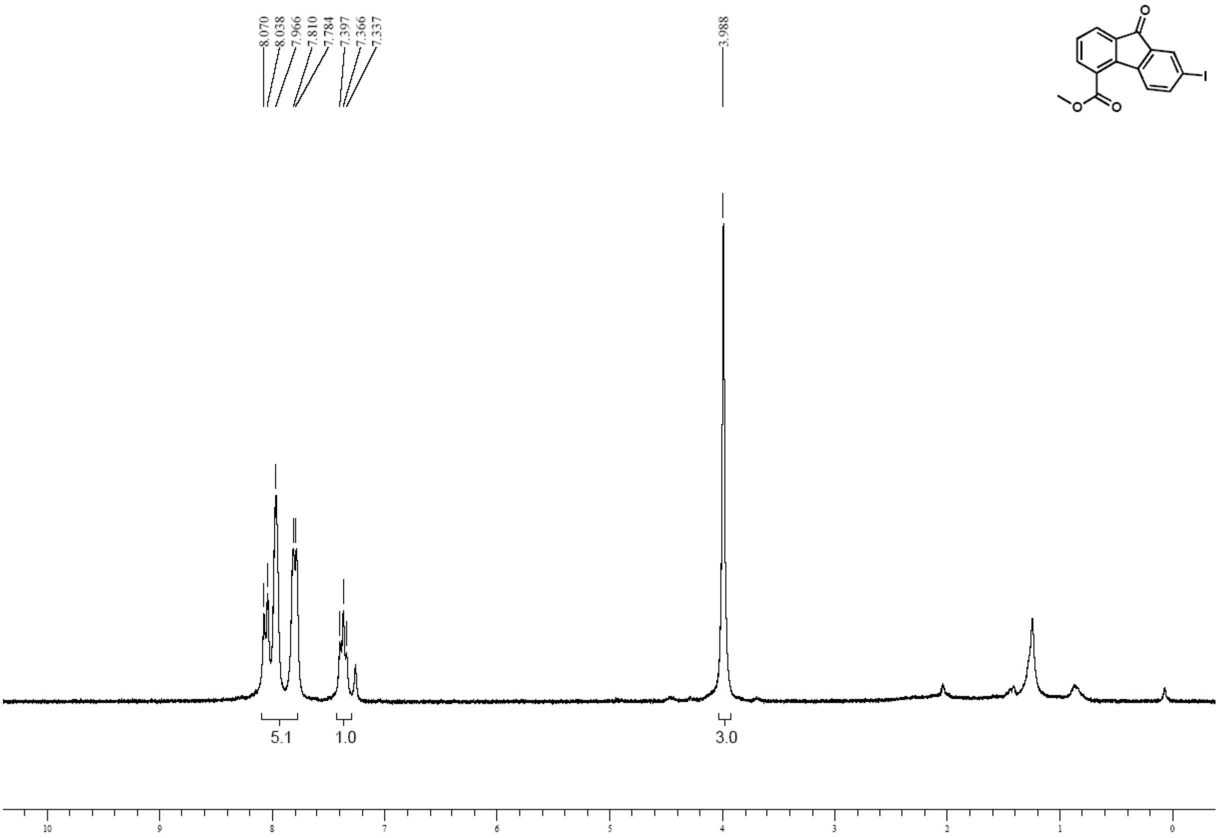


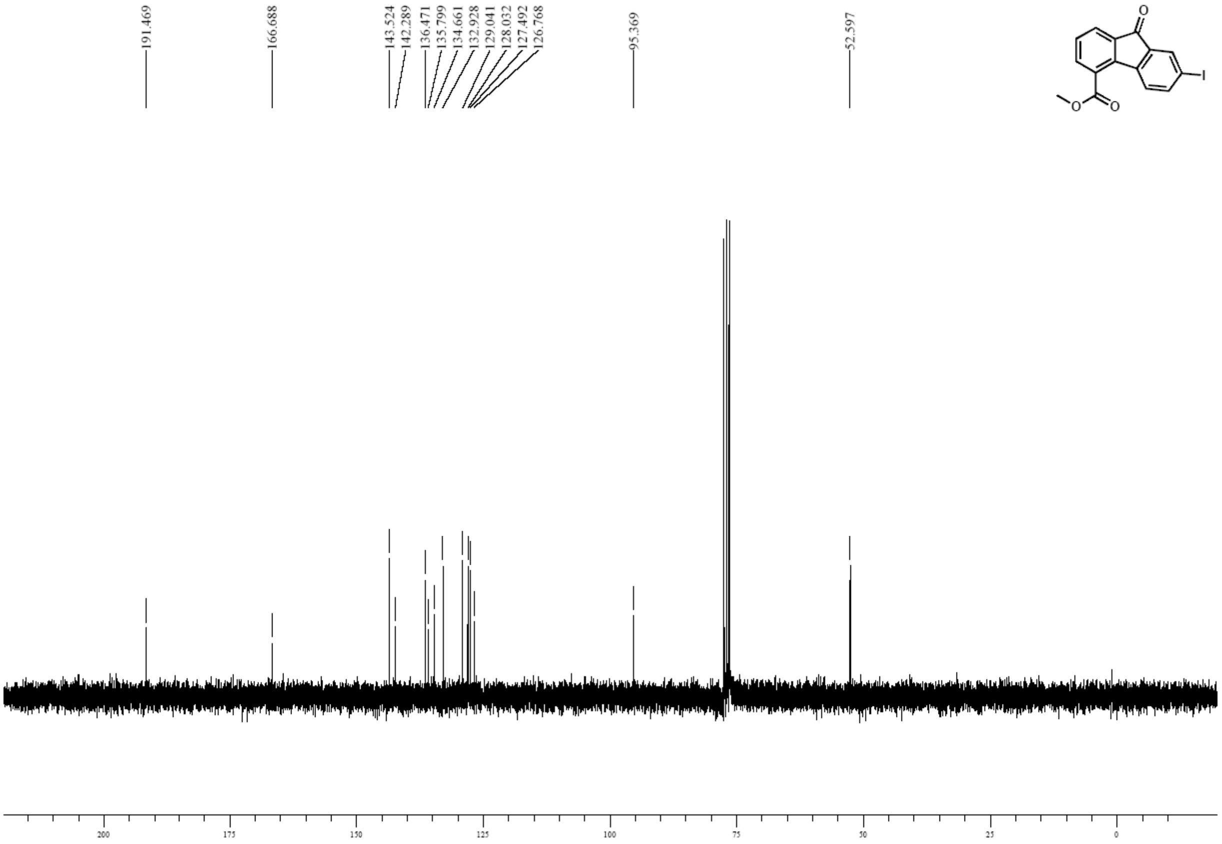
2,7-dibromo-9*H*-fluoren-9-one (2e)[5,6]



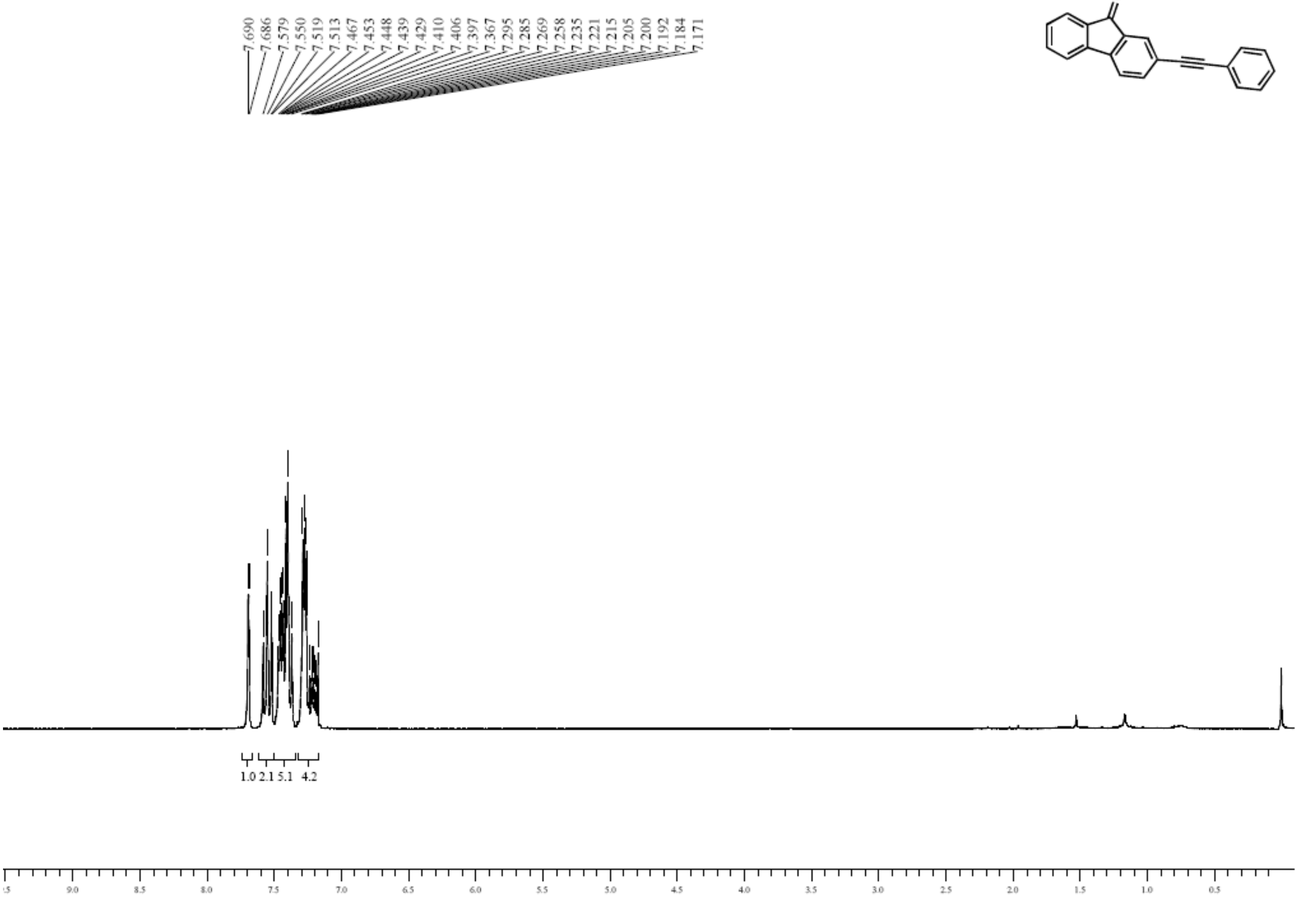


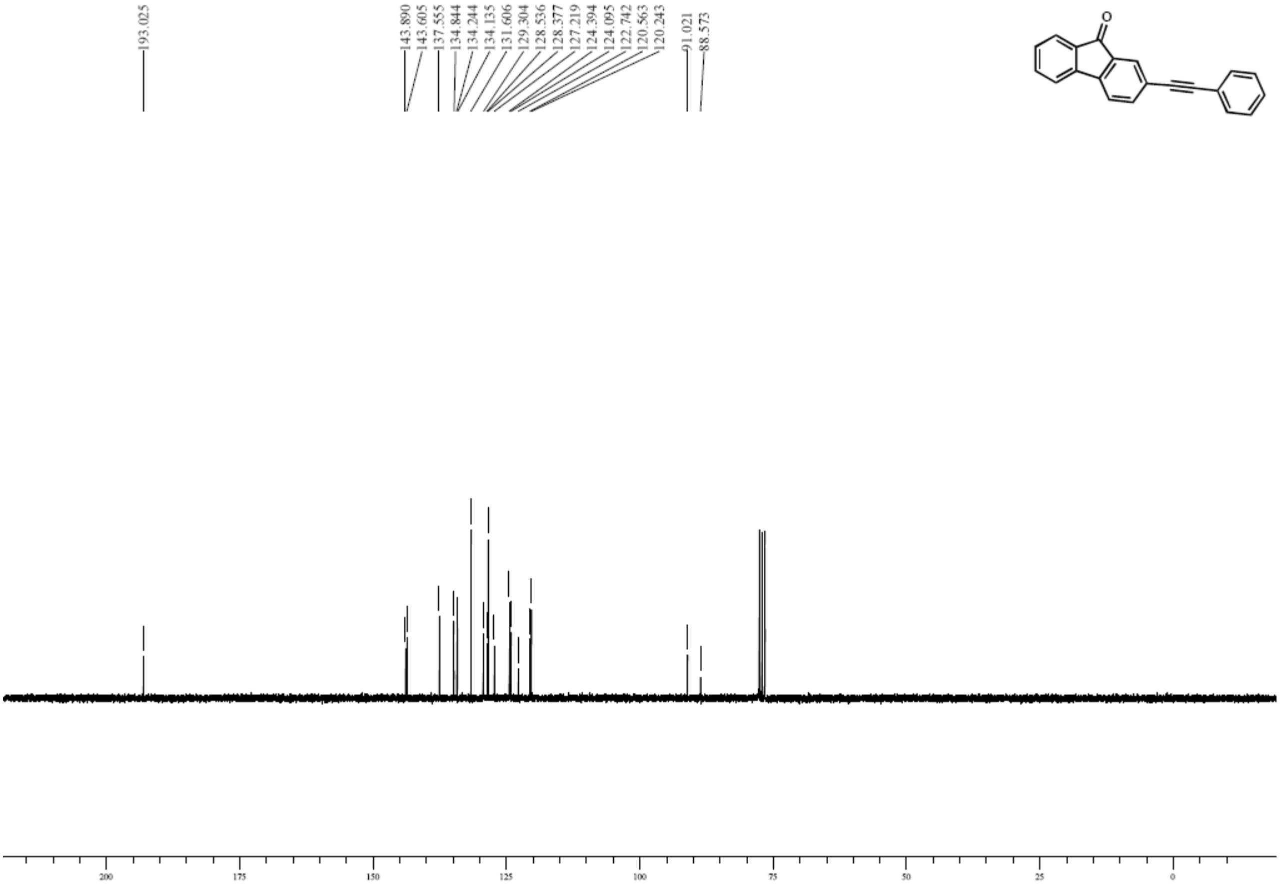
Methyl 7-iodo-9-oxo-9*H*-fluorene-4-carboxylate (2f)



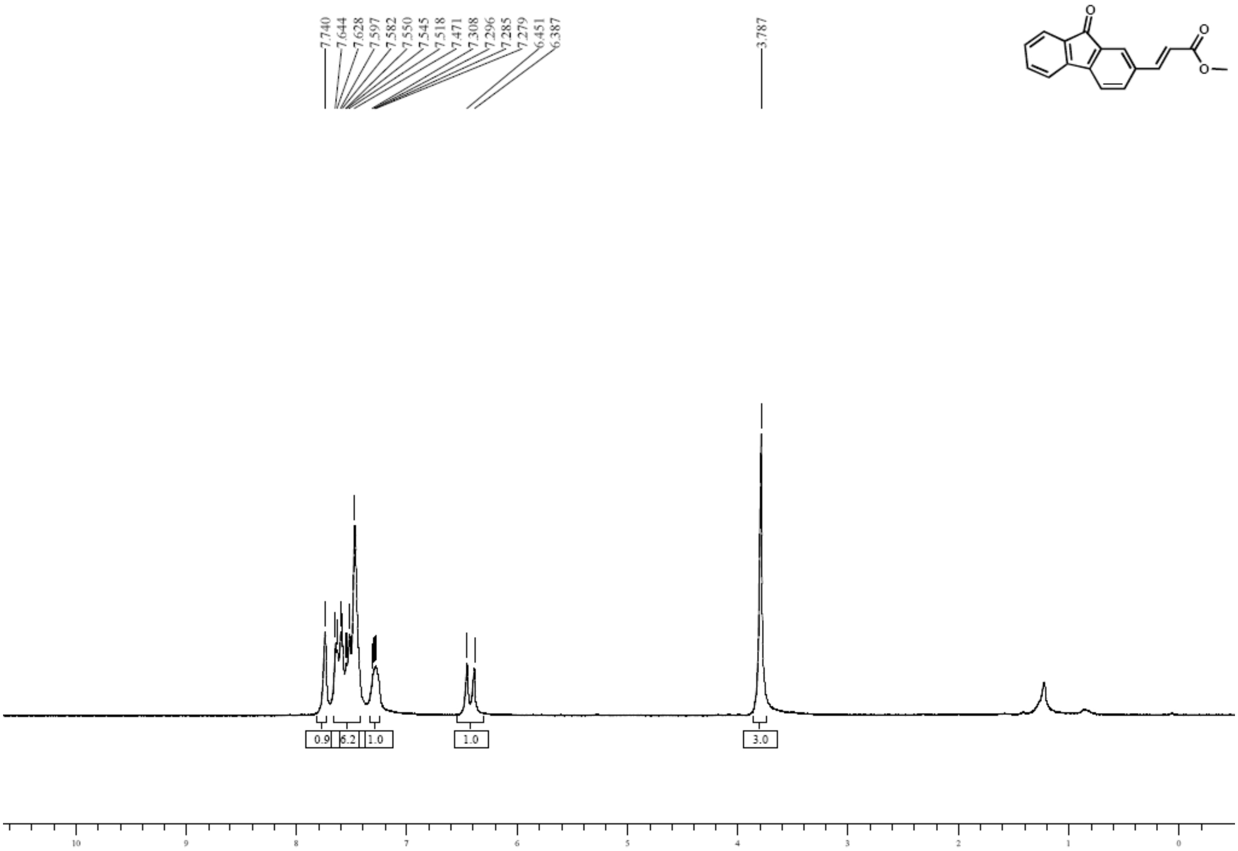


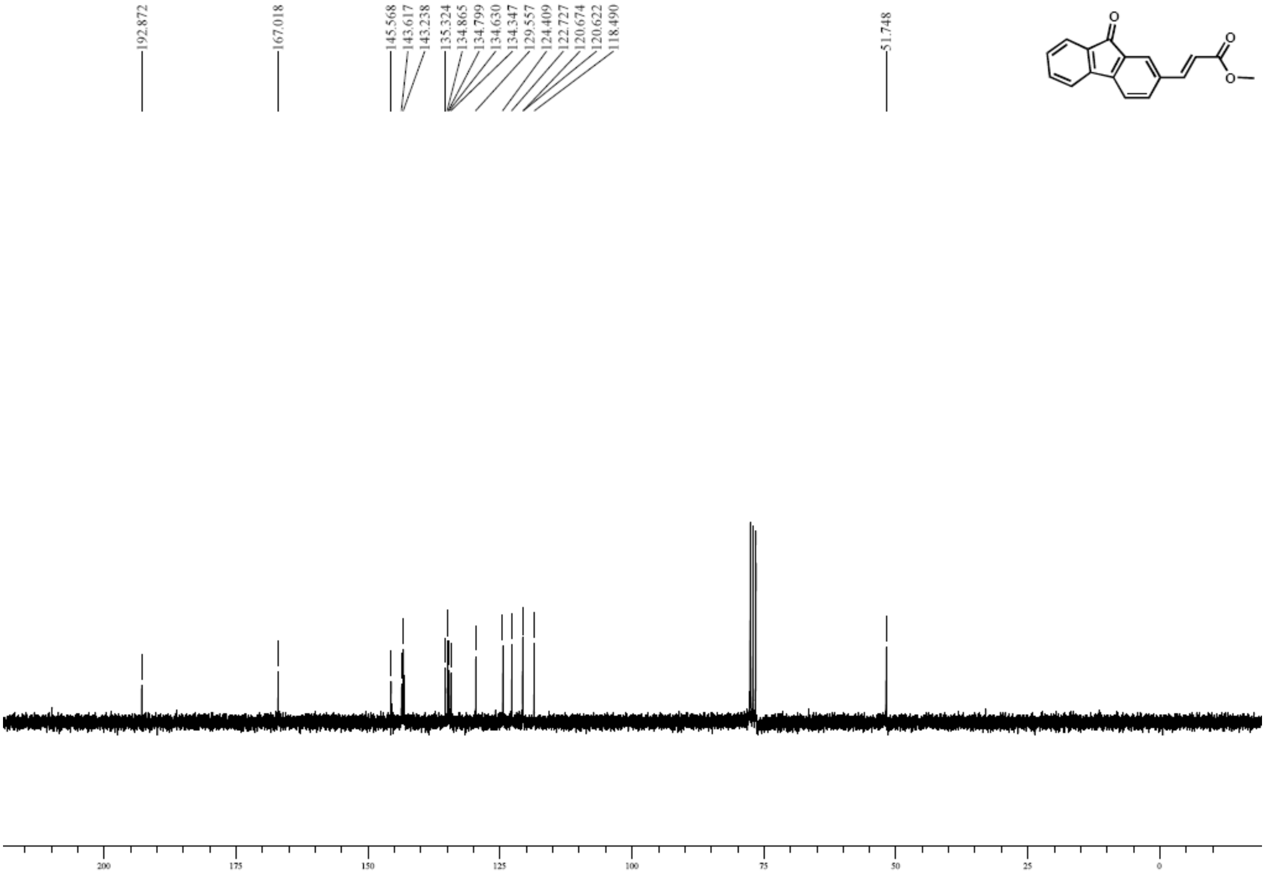
2-(phenylethynyl)-9*H*-fluoren-9-one (2g)[7]



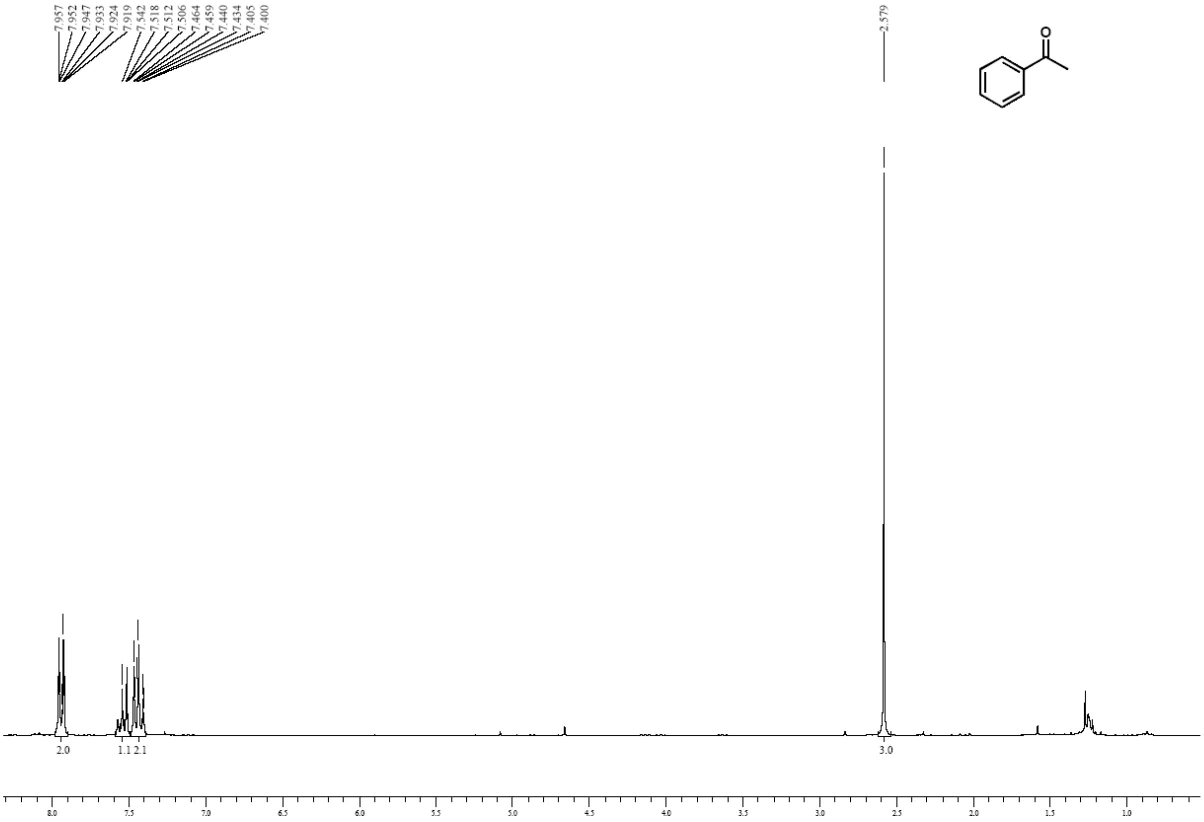


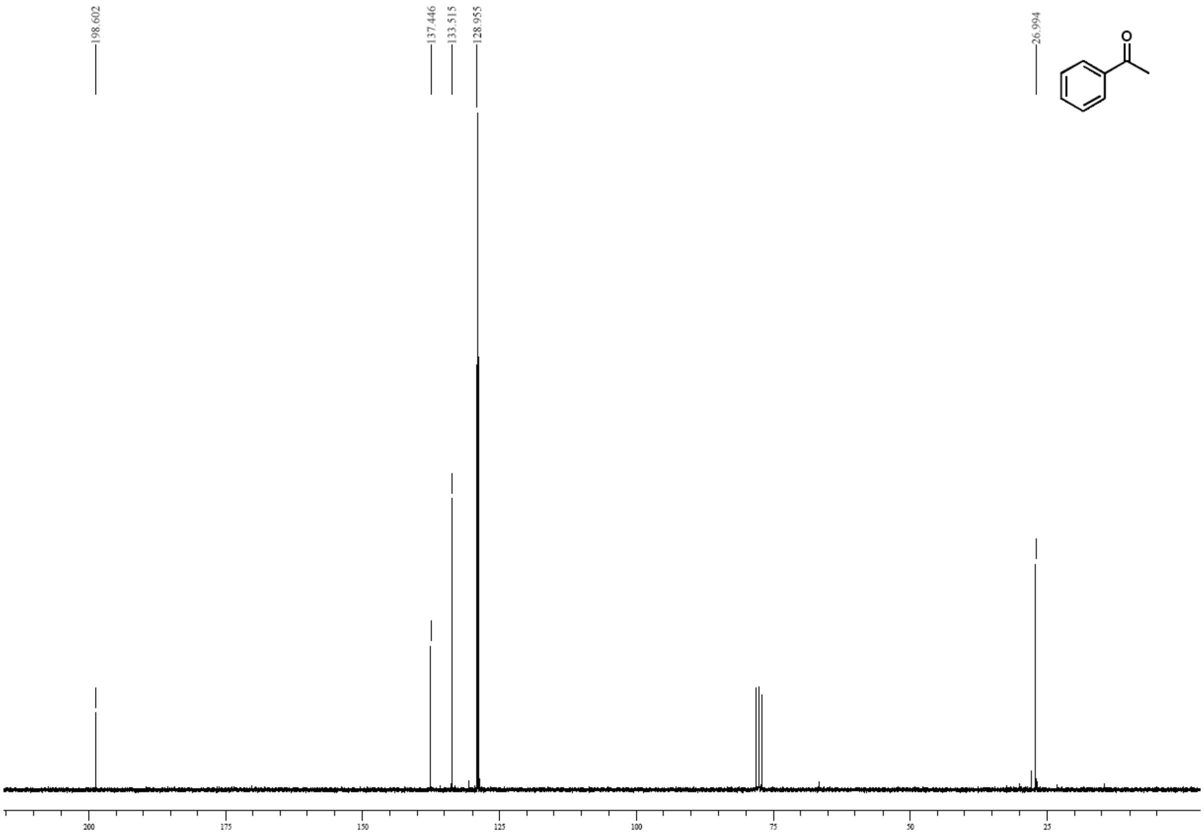
(E)-methyl 3-(9-oxo-9*H*-fluoren-2-yl)acrylate (2h)



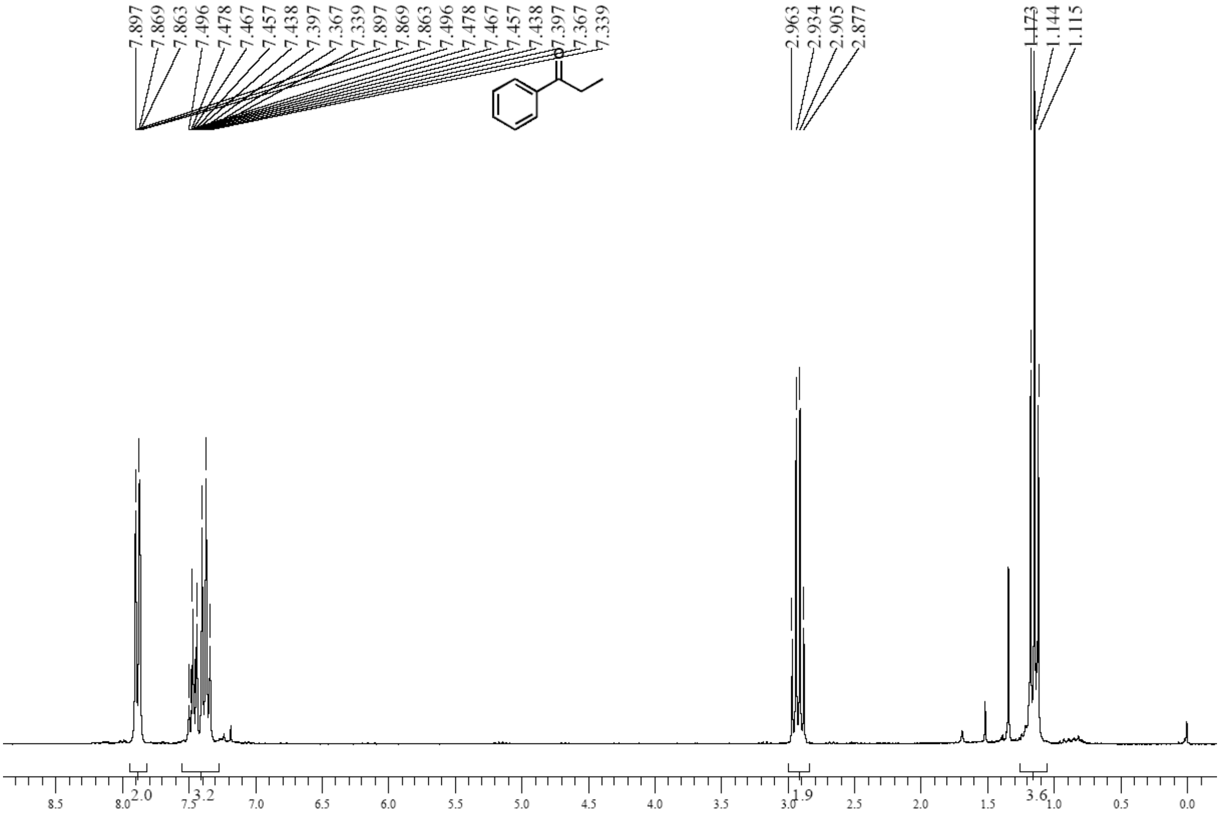


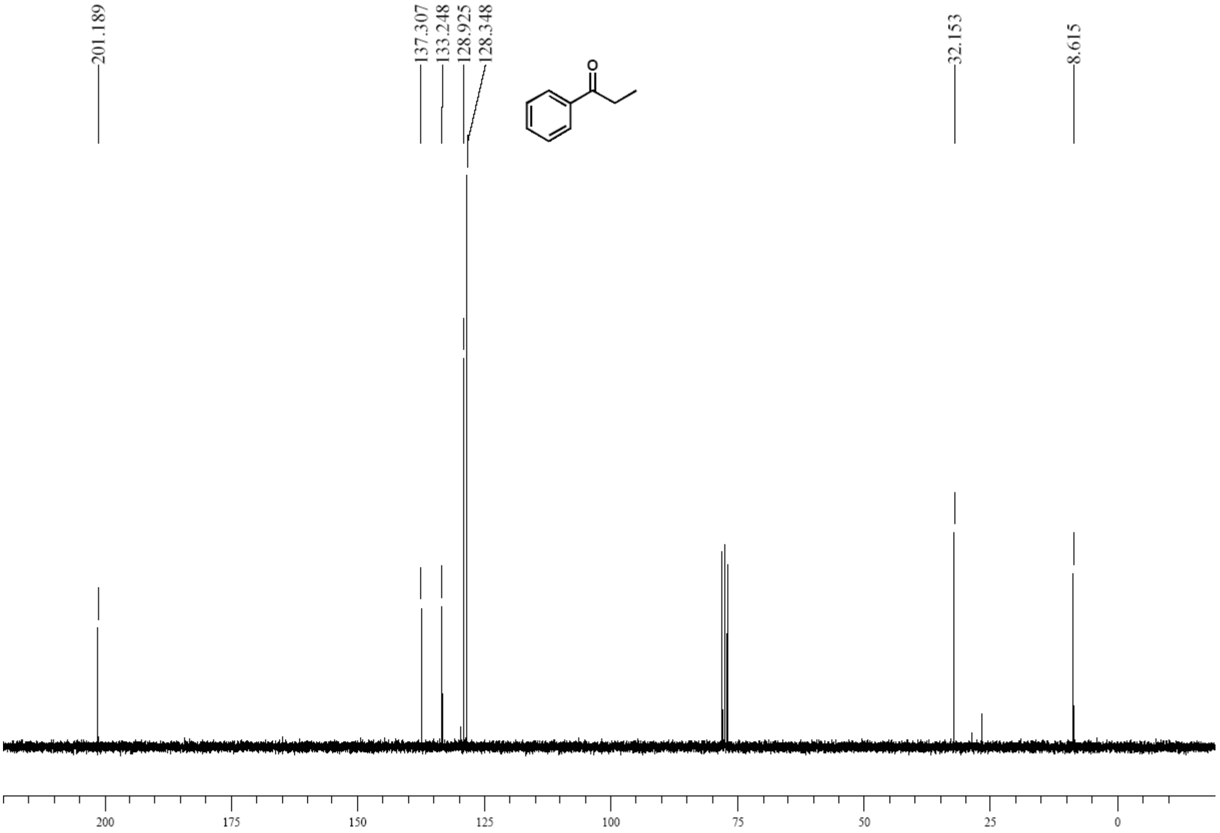
Acetophenone (2i)[3]



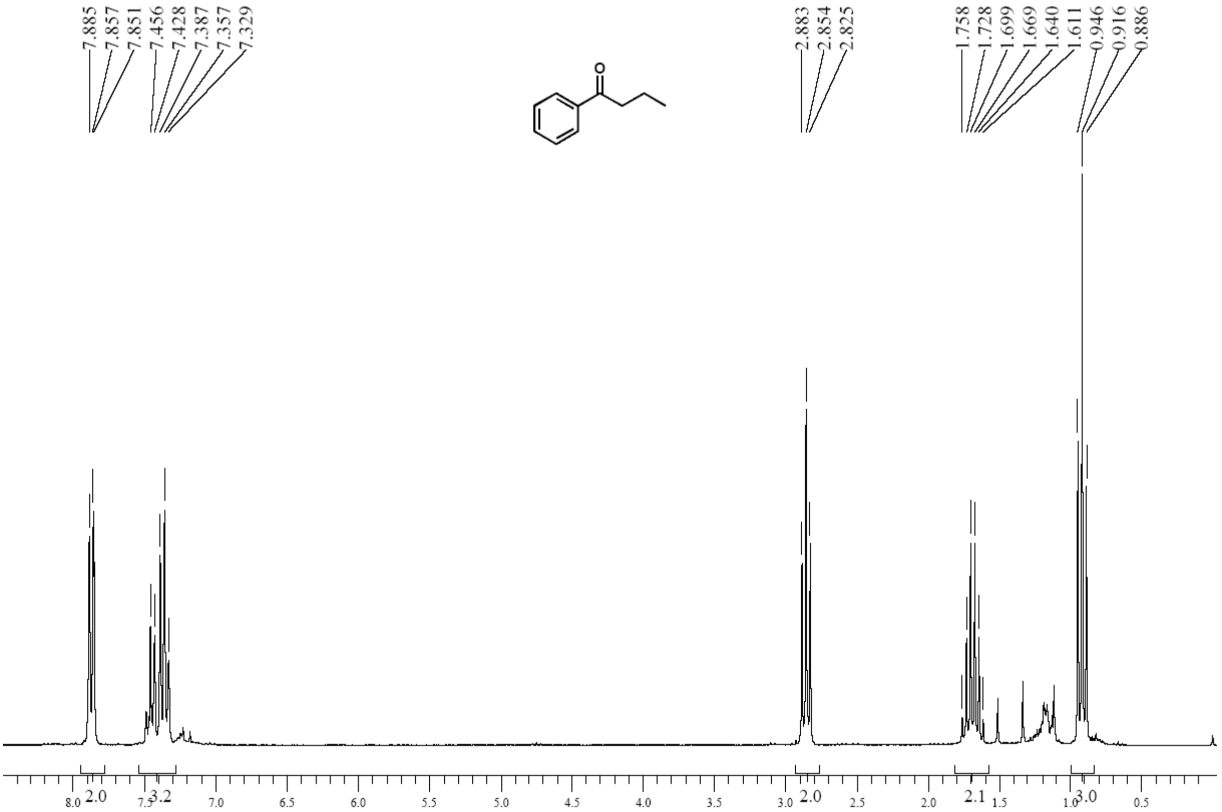


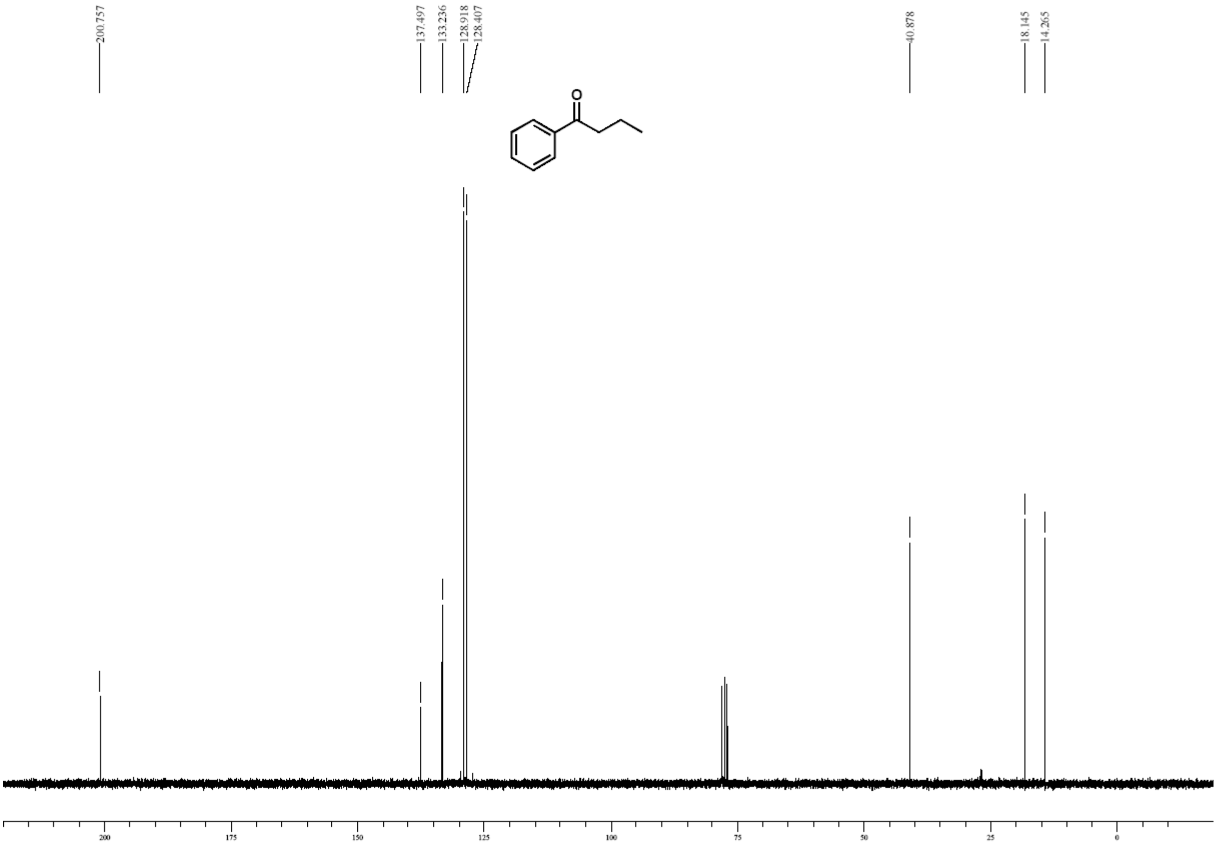
Propiophenone (2j)[8]



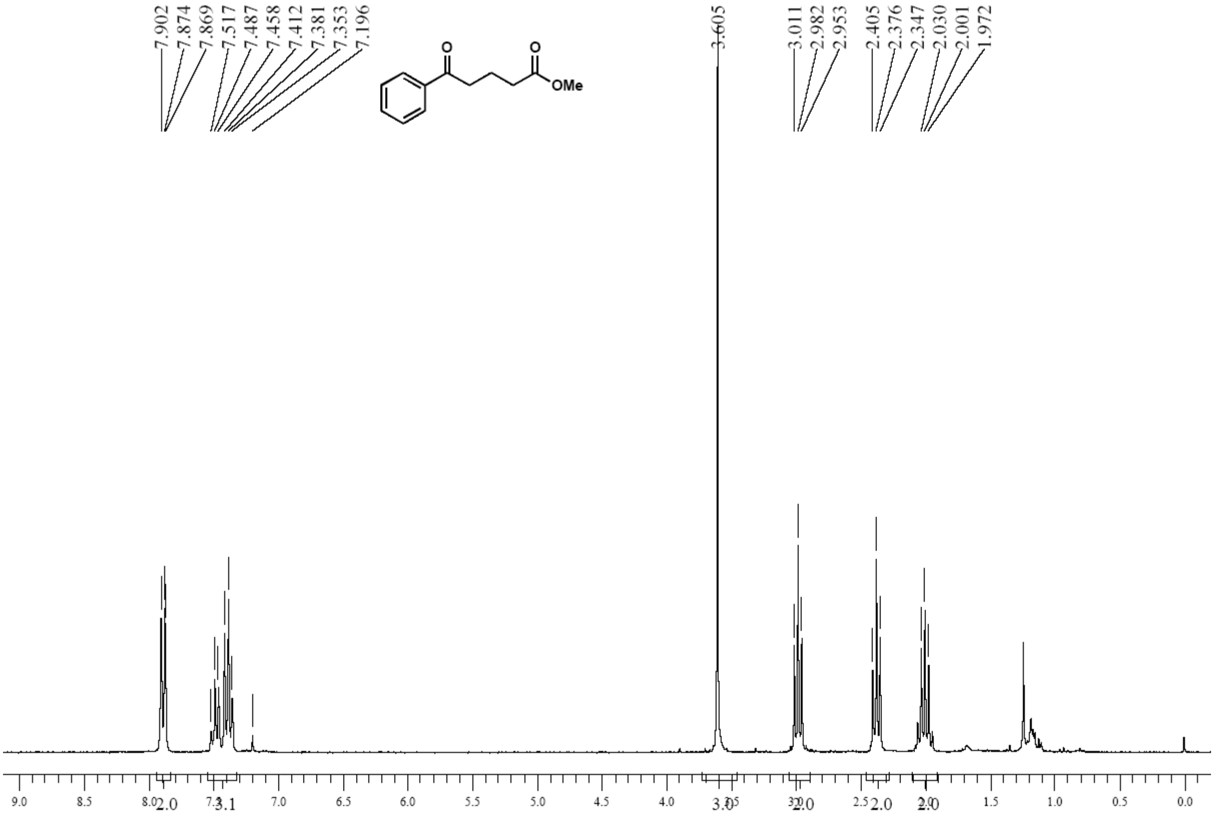


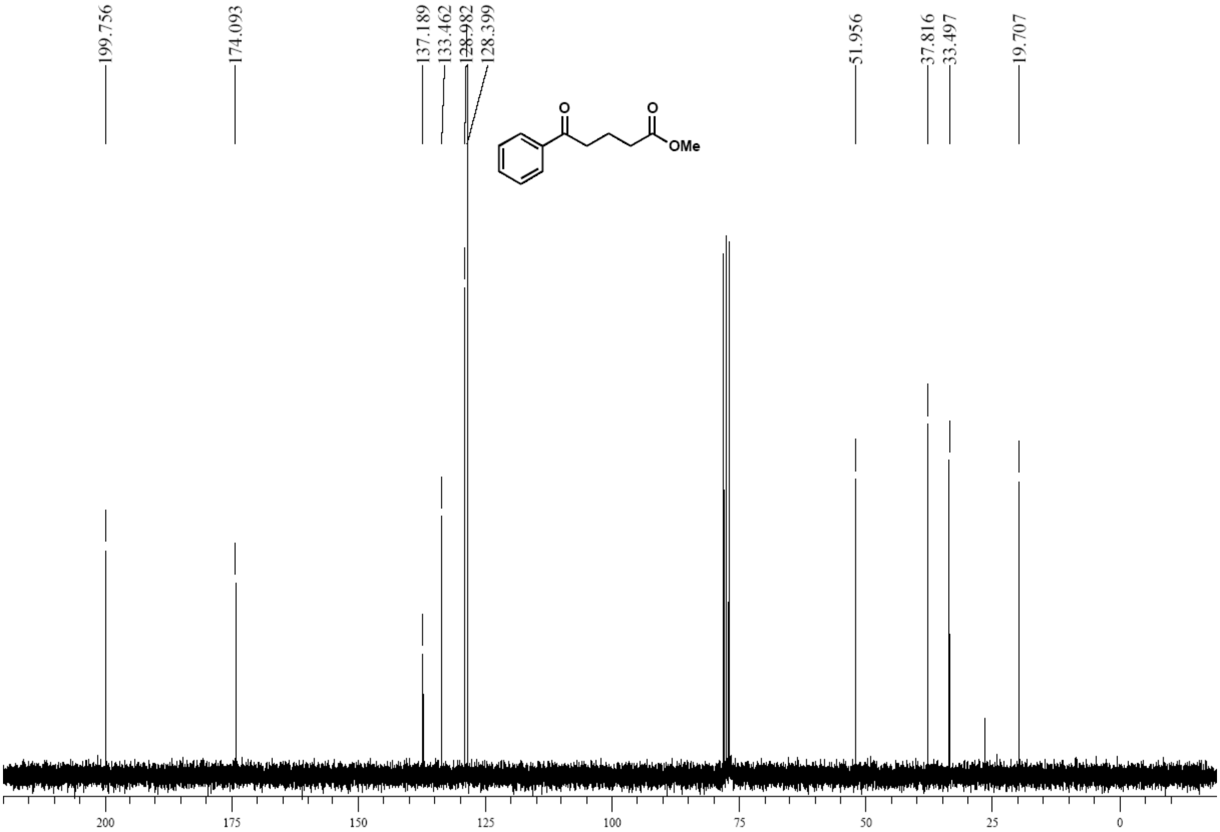
Butirophenone (2k)[9]



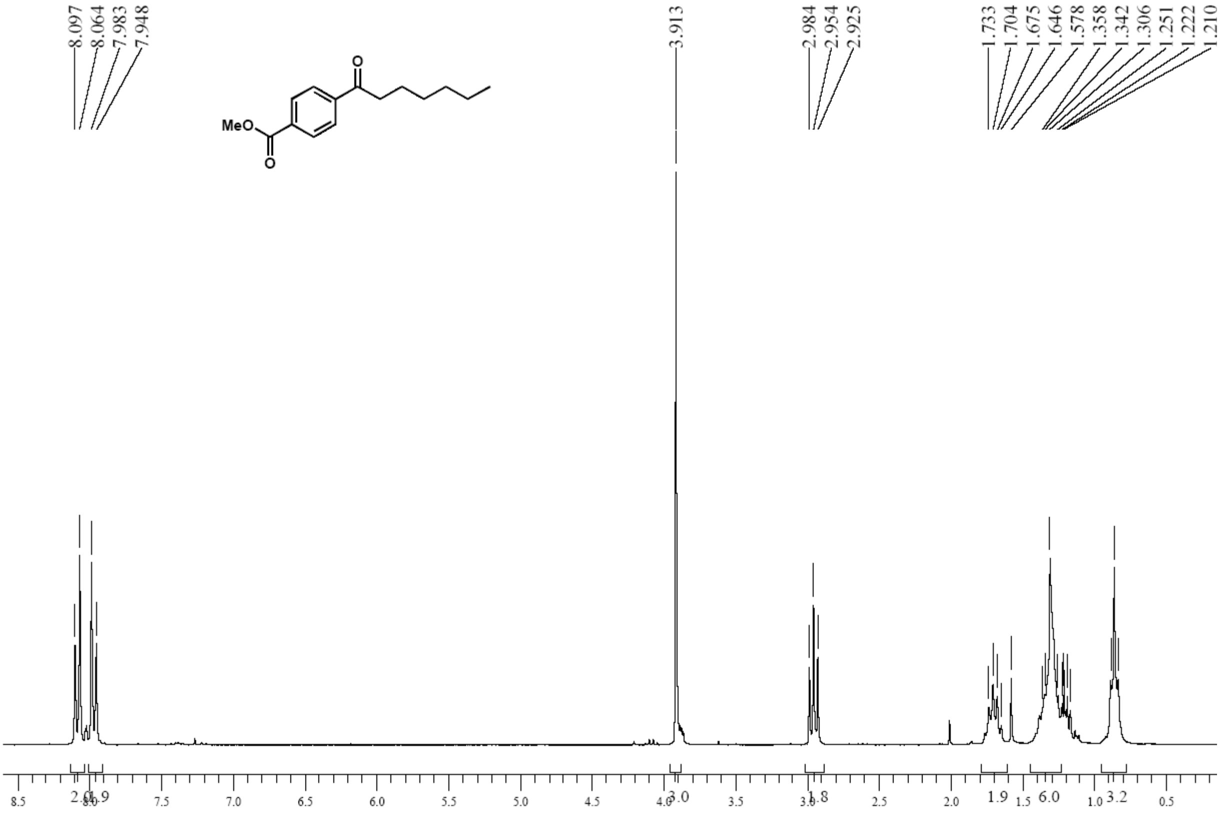


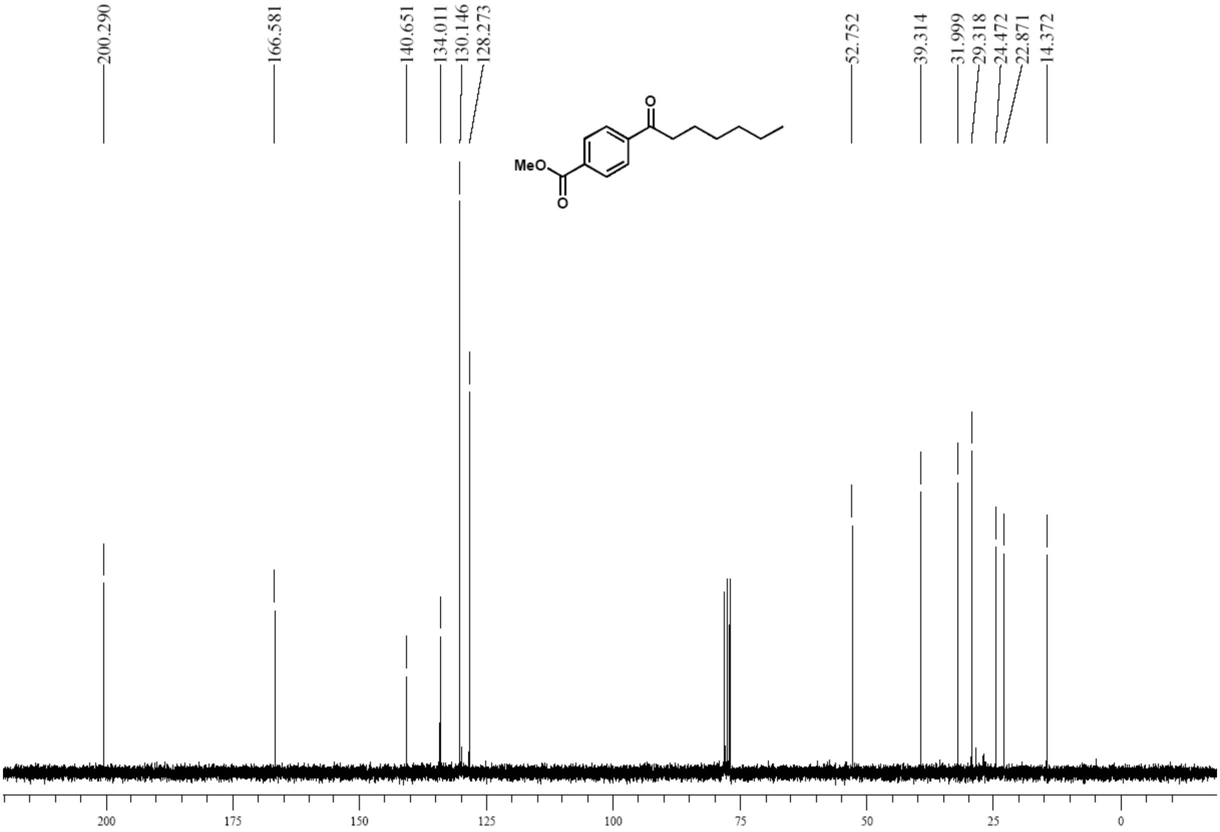
Methyl 5-oxo-5-phenylpentanoate (2l)[10]



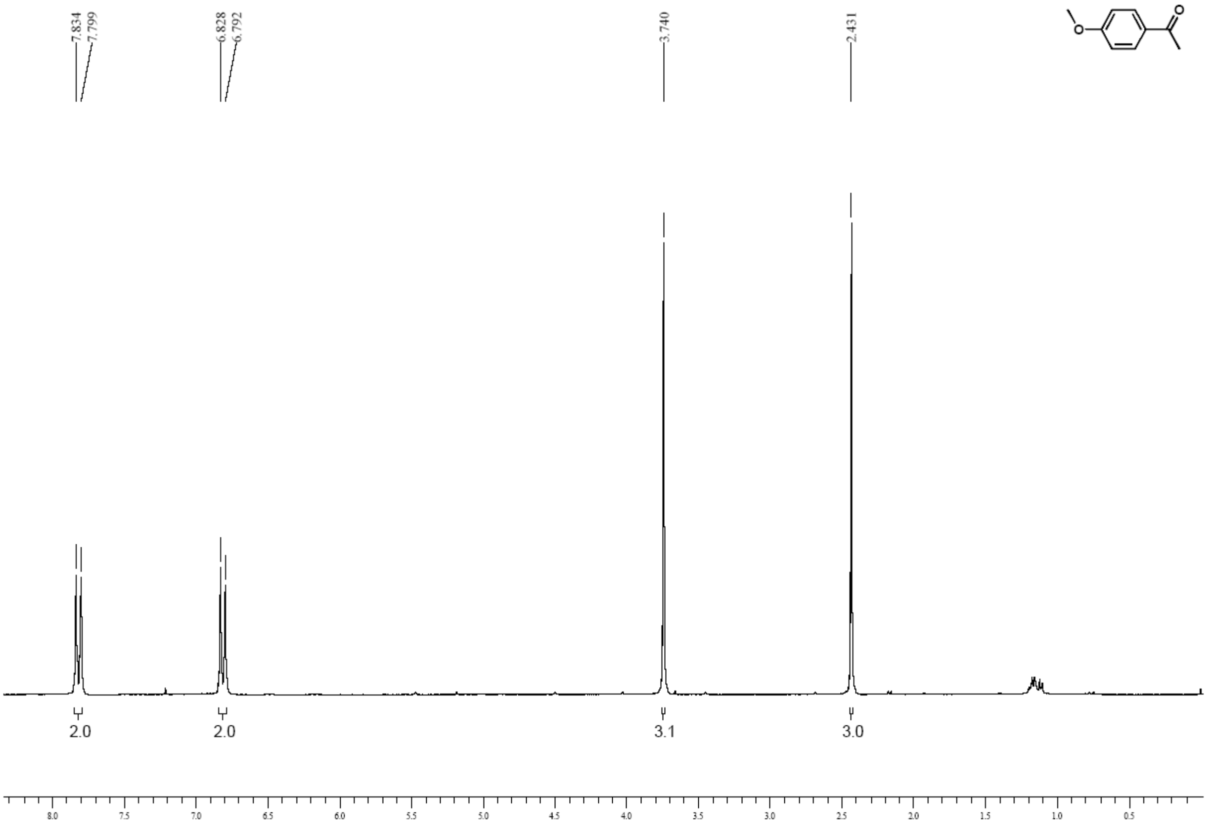


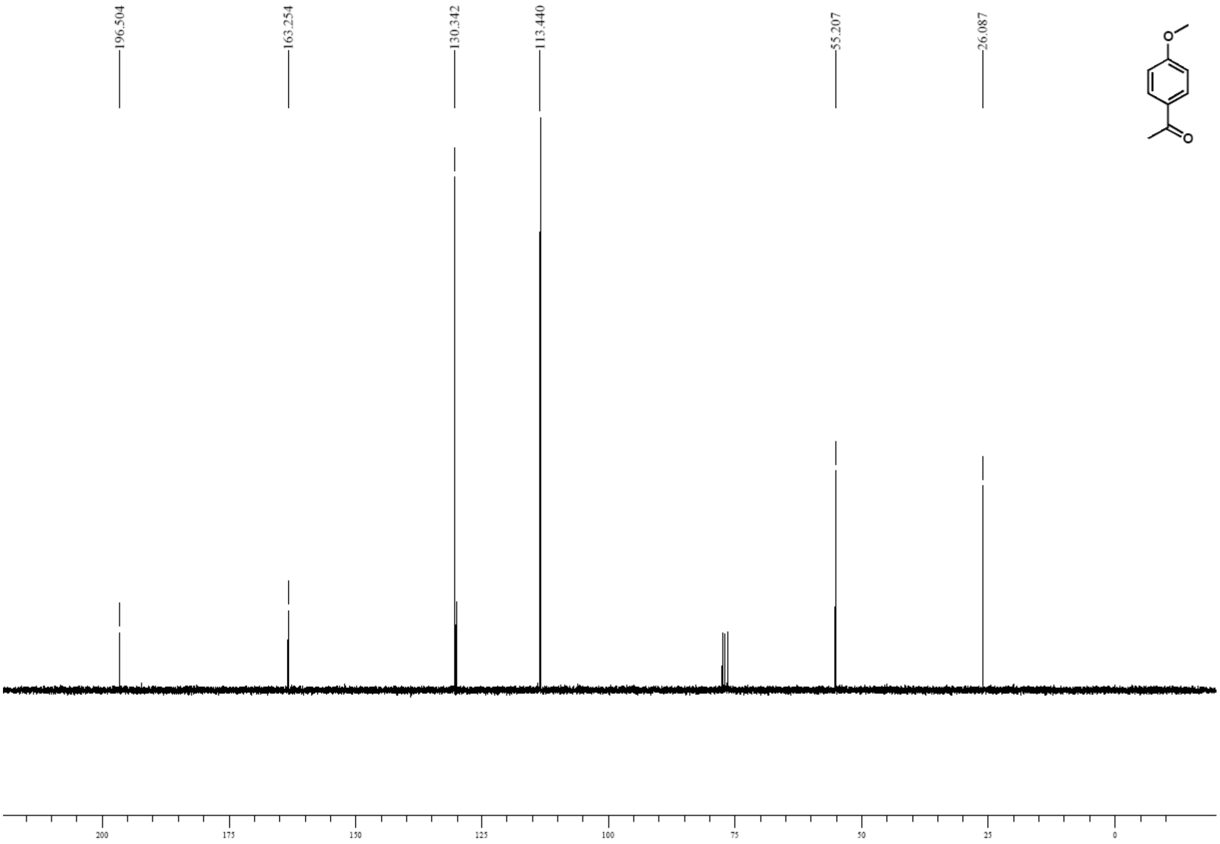
Methyl 4-heptanoylbenzoate (2m)[11]



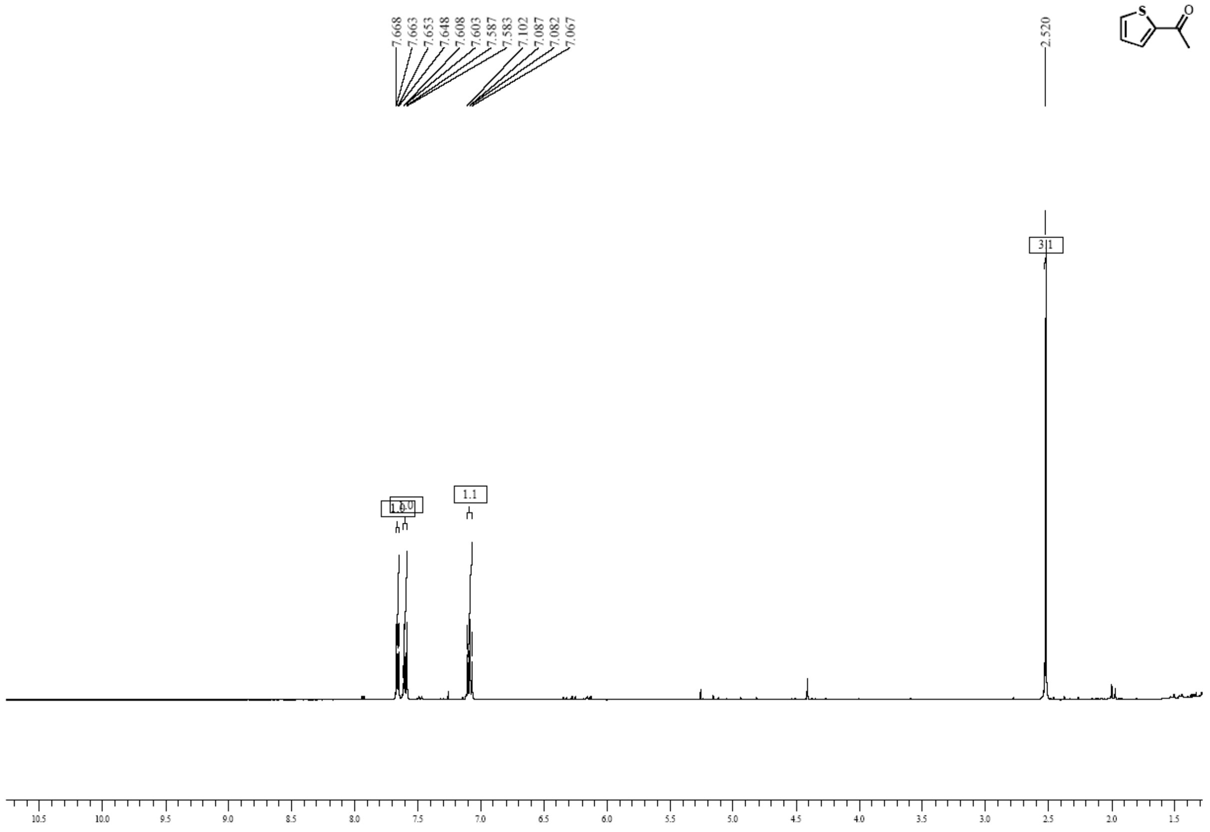


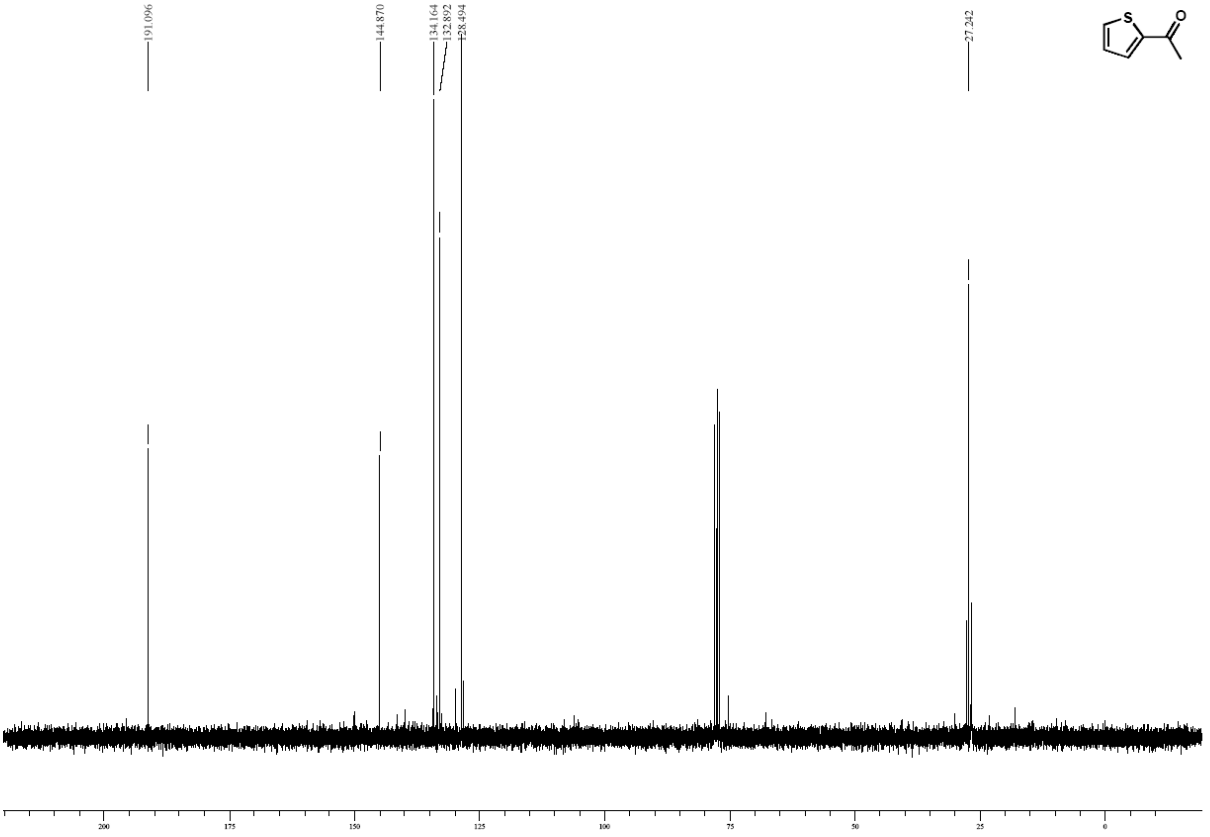
1-(4-methoxyphenyl)ethanone (2n)[12]



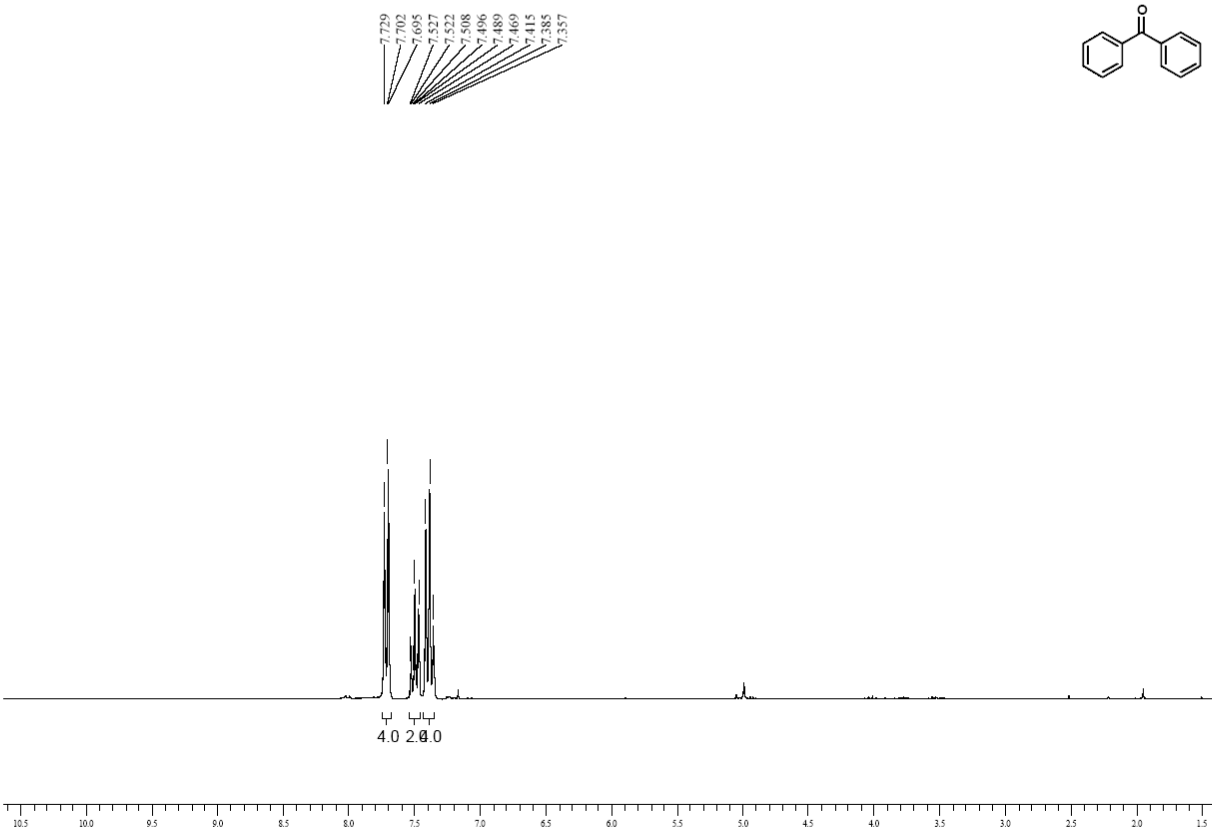


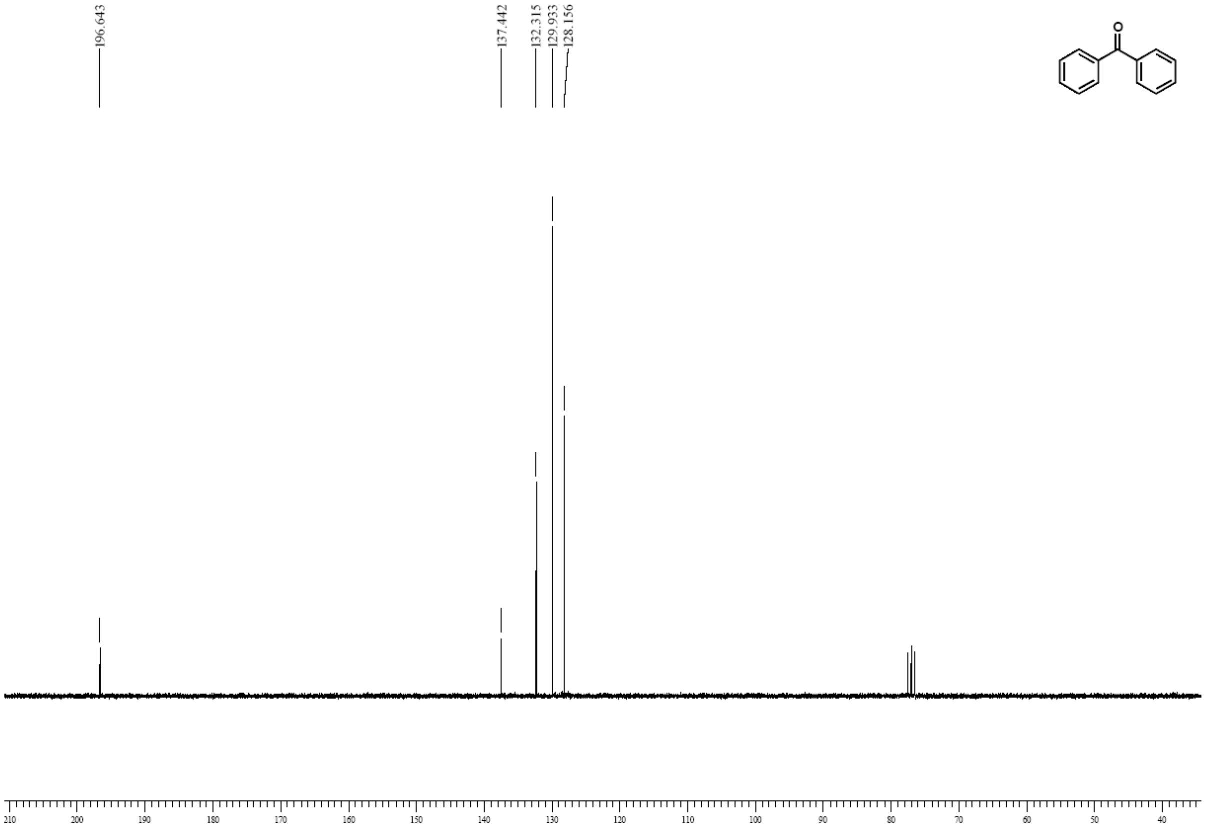
1-(thiophen-2-yl)ethanone (2p)[13]



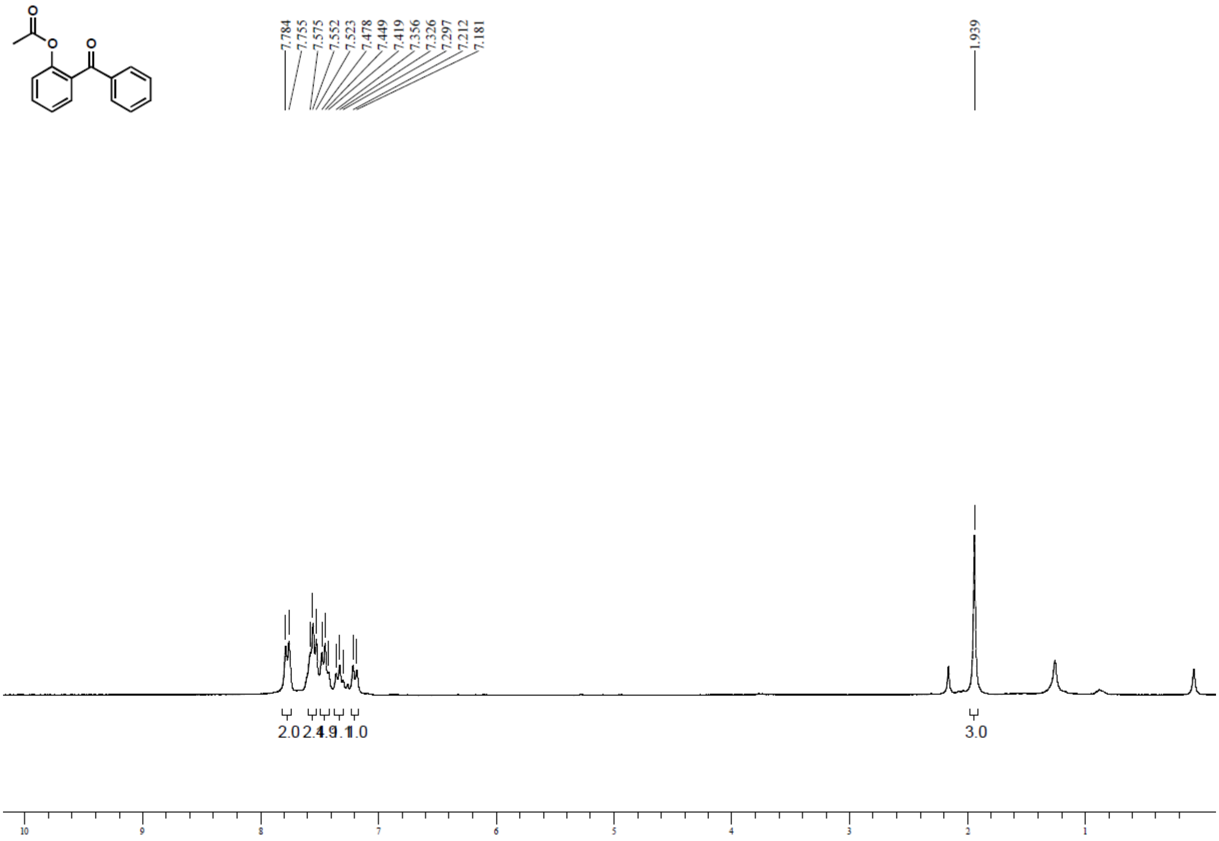


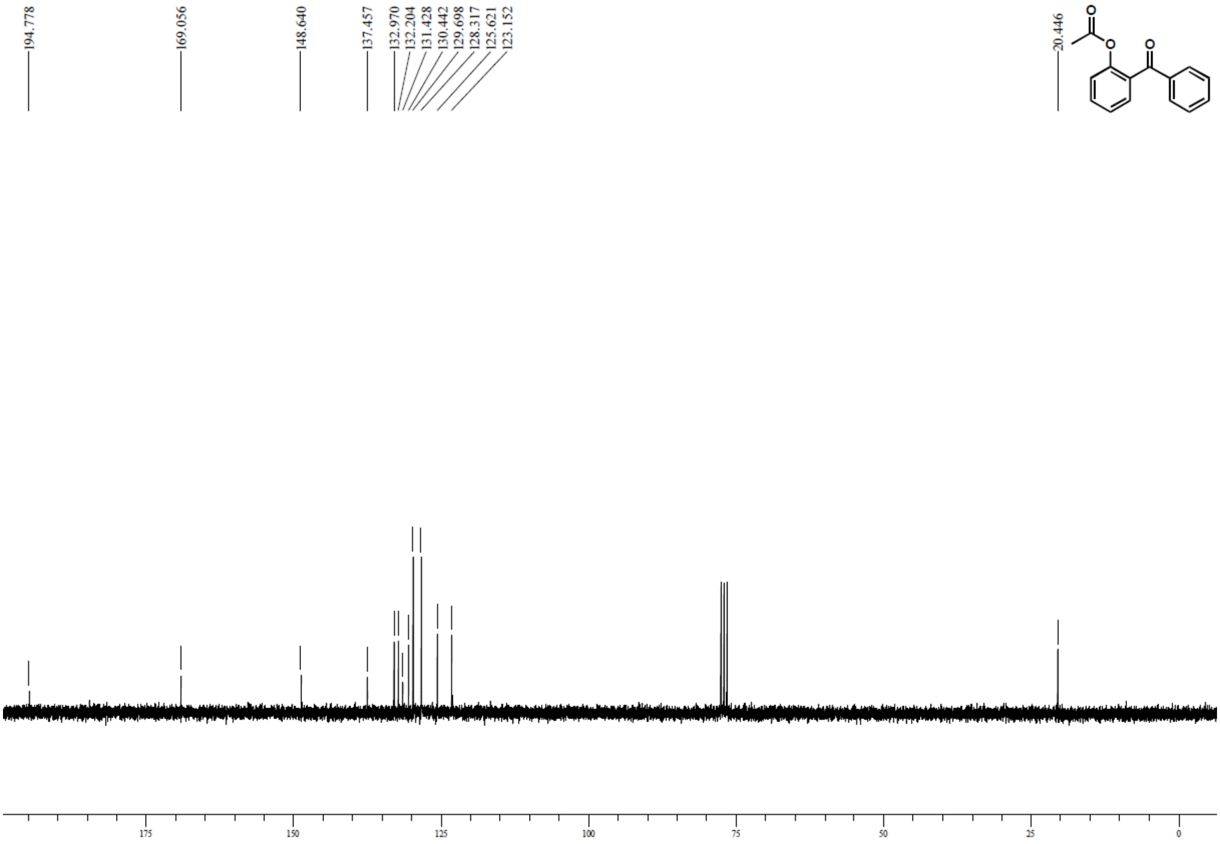
Benzophenone (2q)[3]



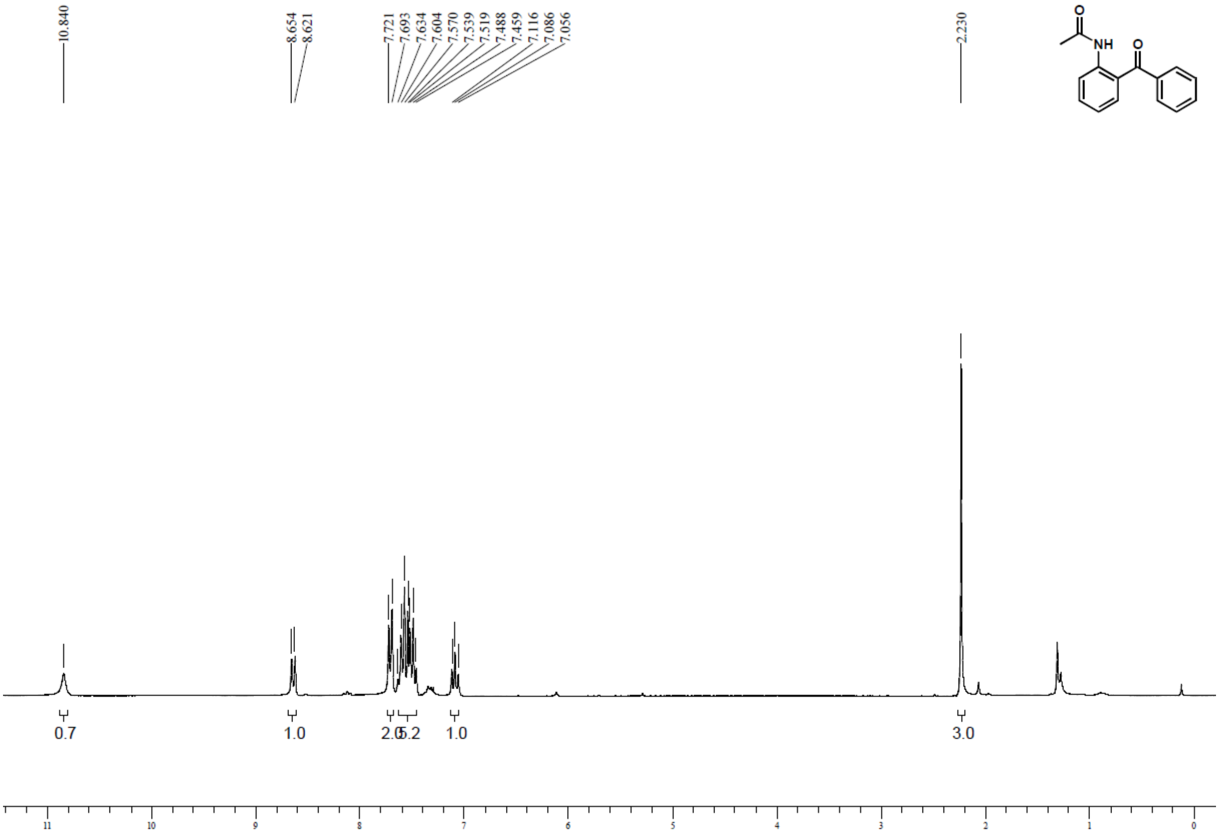


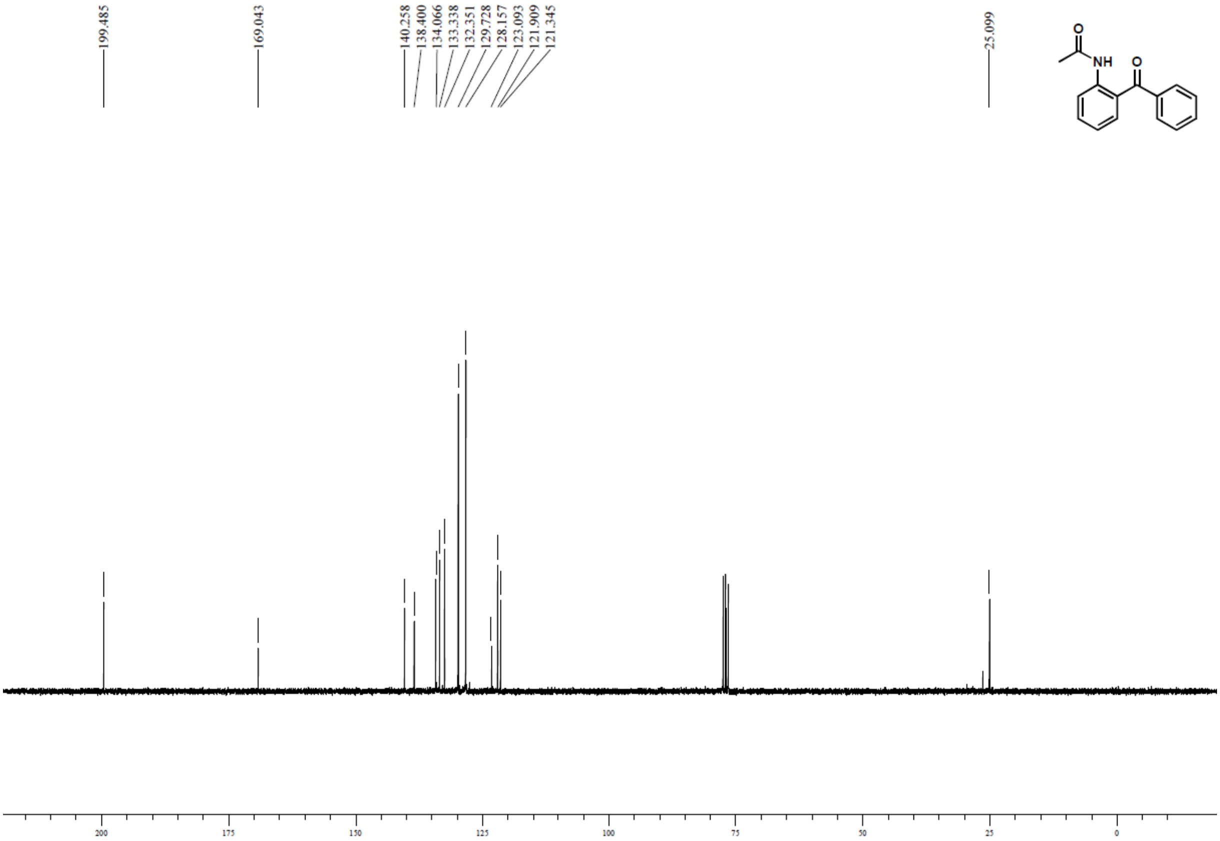
2-benzoylphenyl acetate (2r)[14]



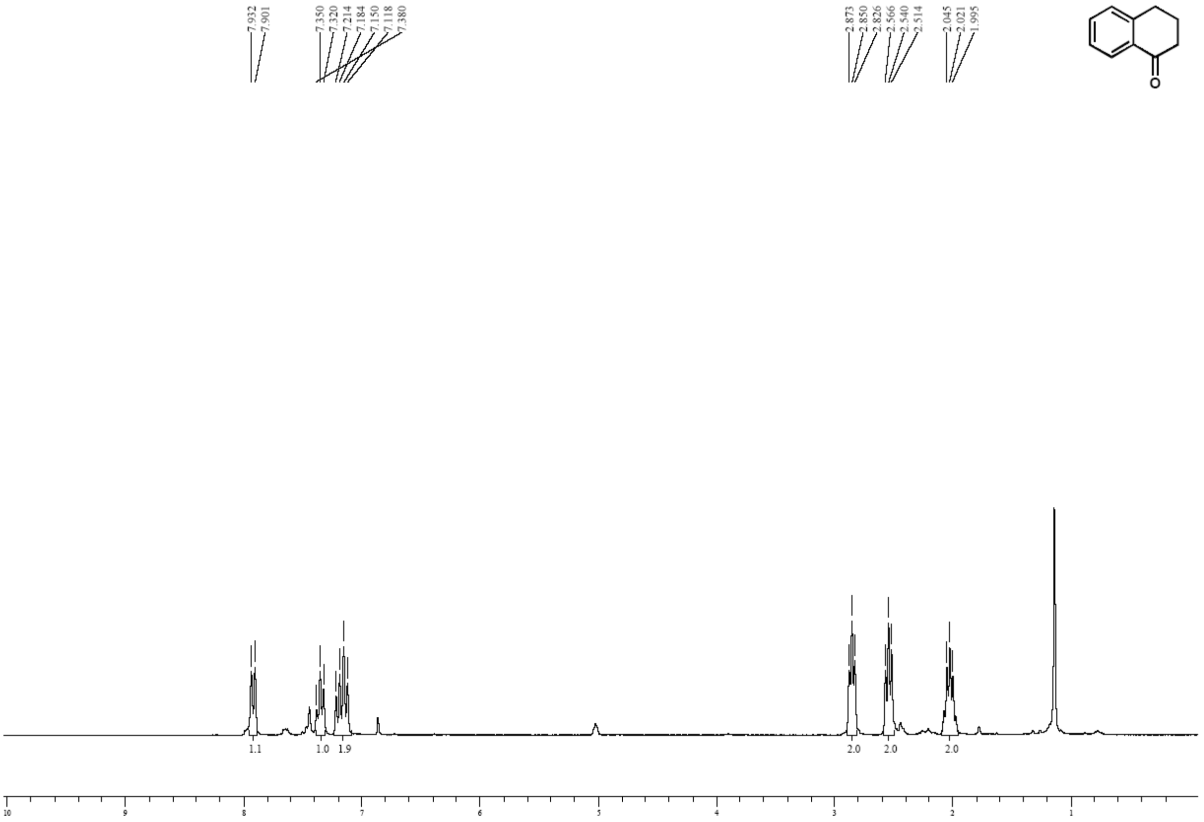


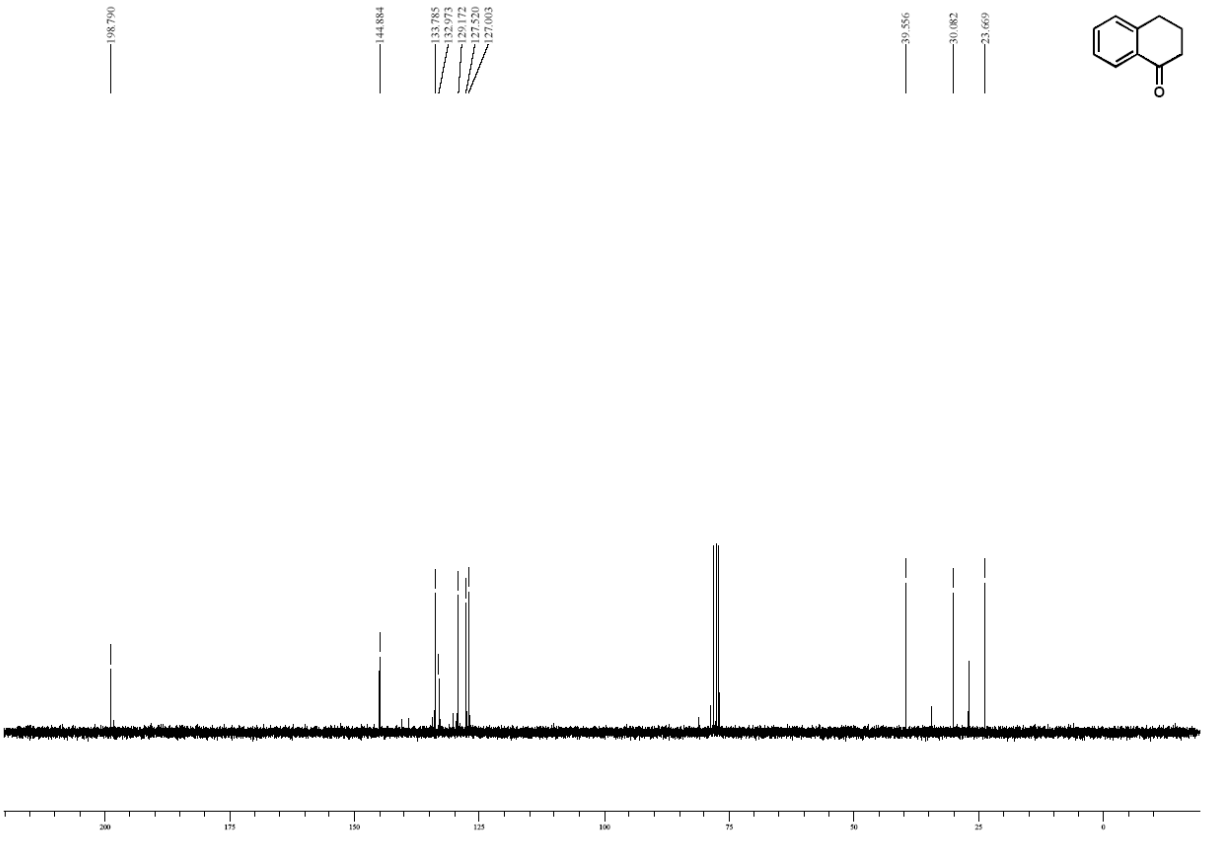
N-(2-benzoylphenyl)acetamide (2s)[15]



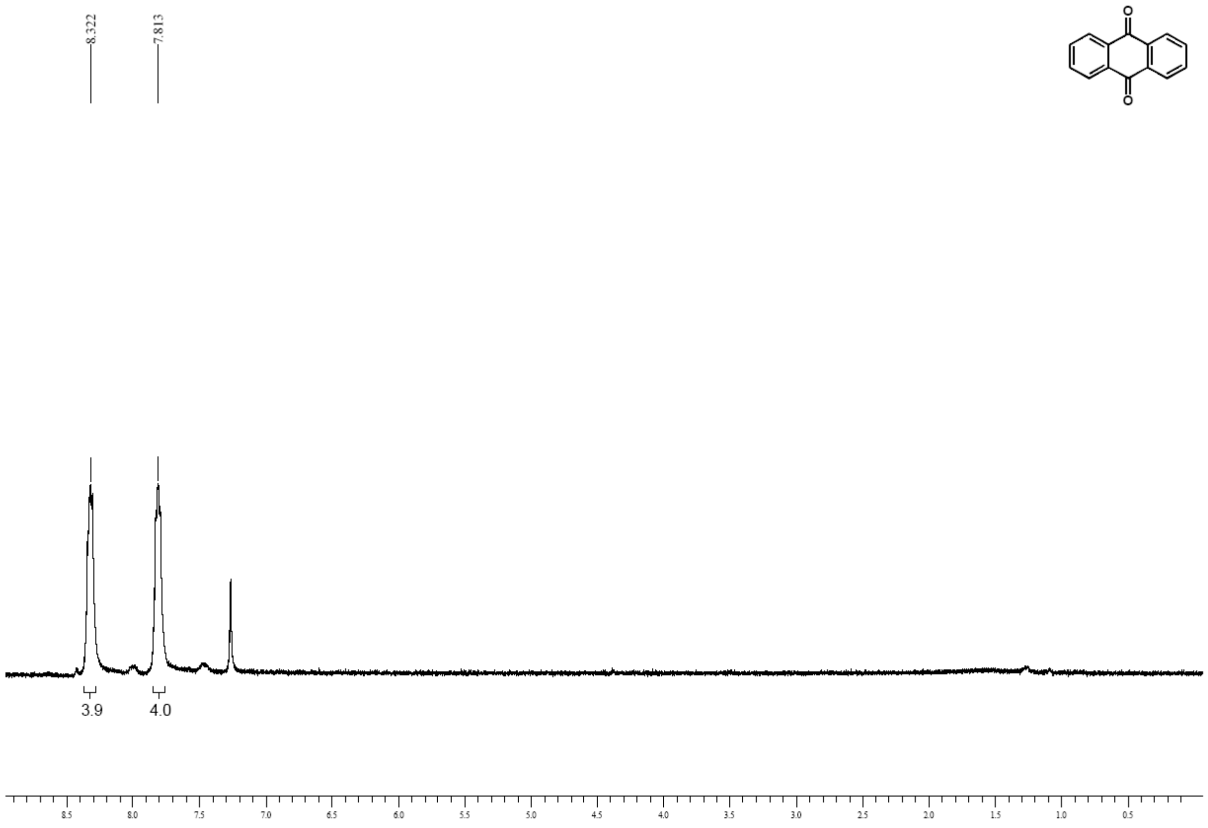


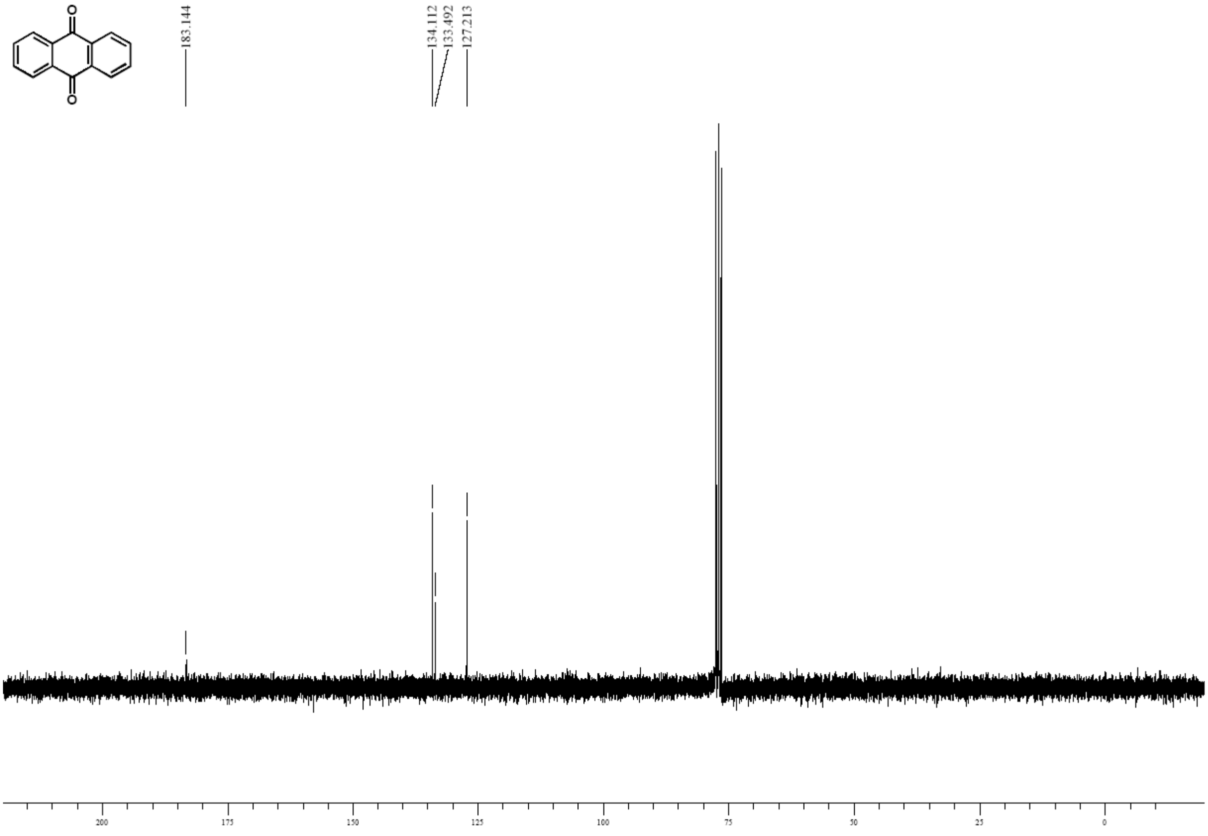
3,4-dihydronaphthalen-1(2*H*)-one (2t)[3]



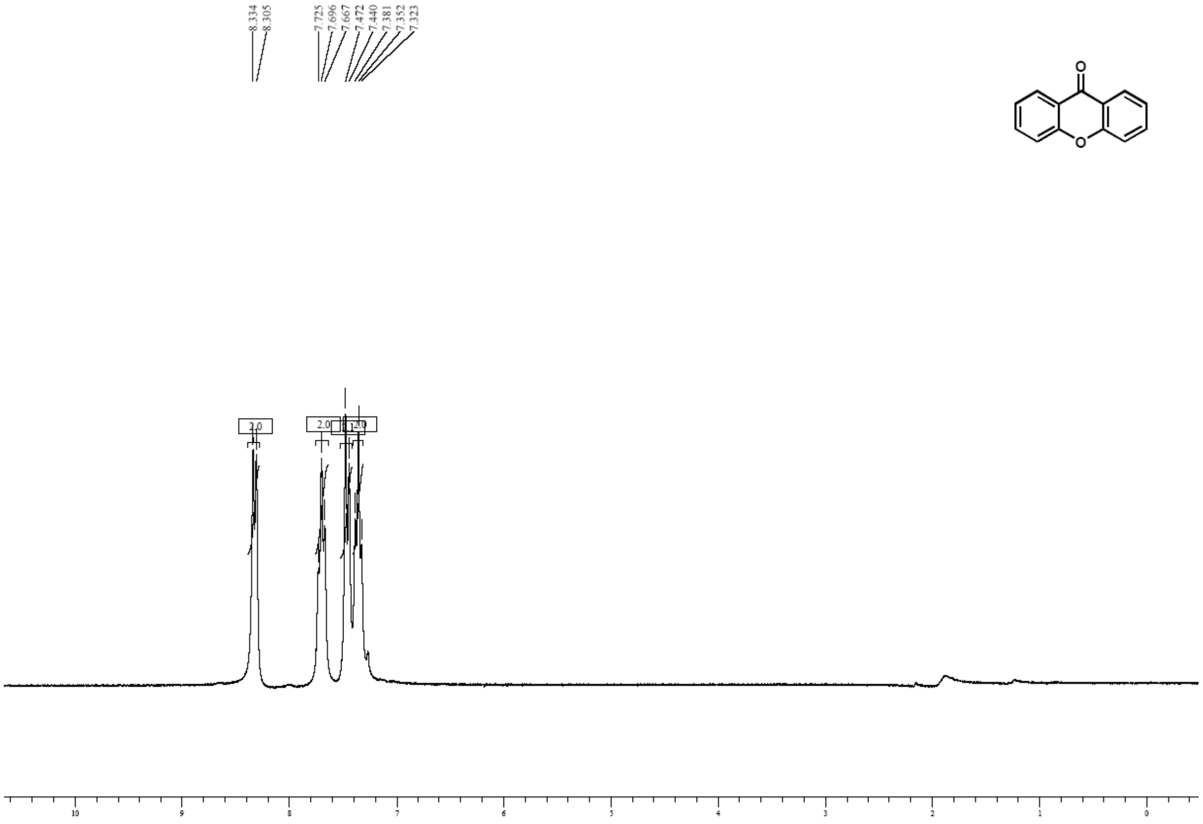


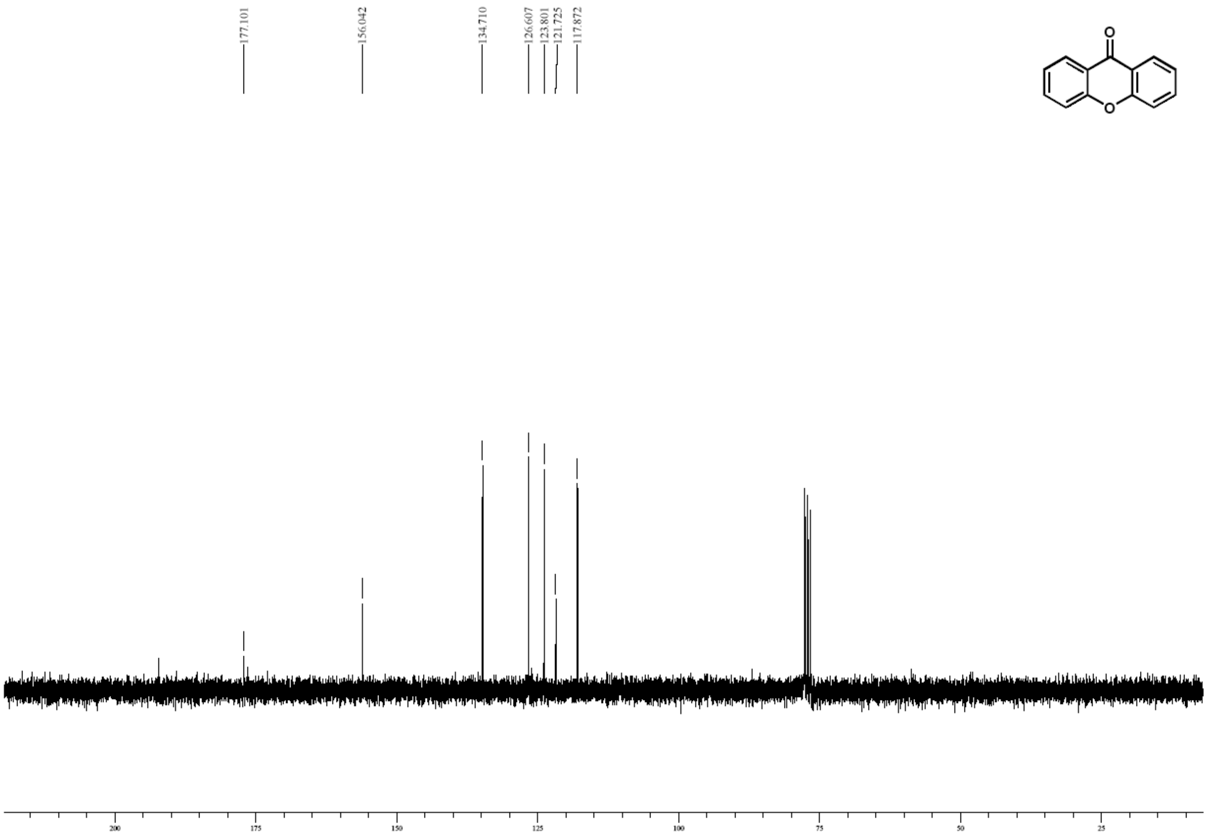
Anthracene-9,10-dione (2v)[16]



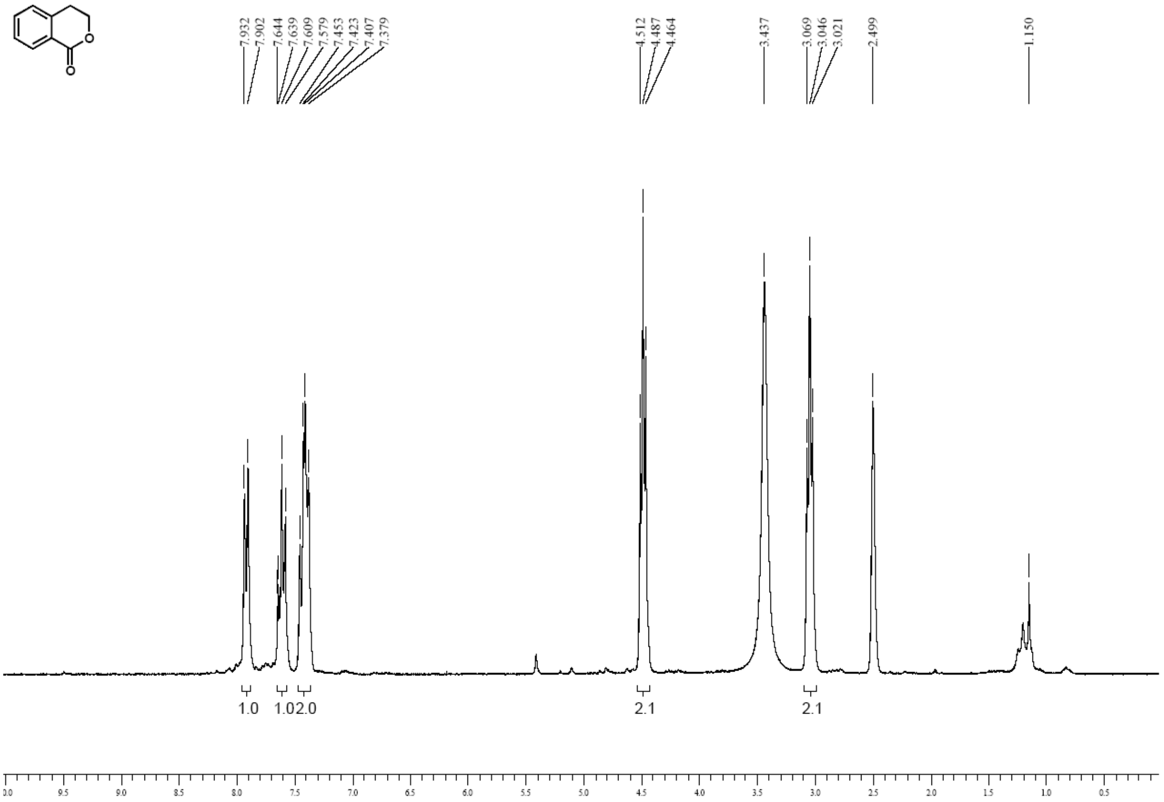


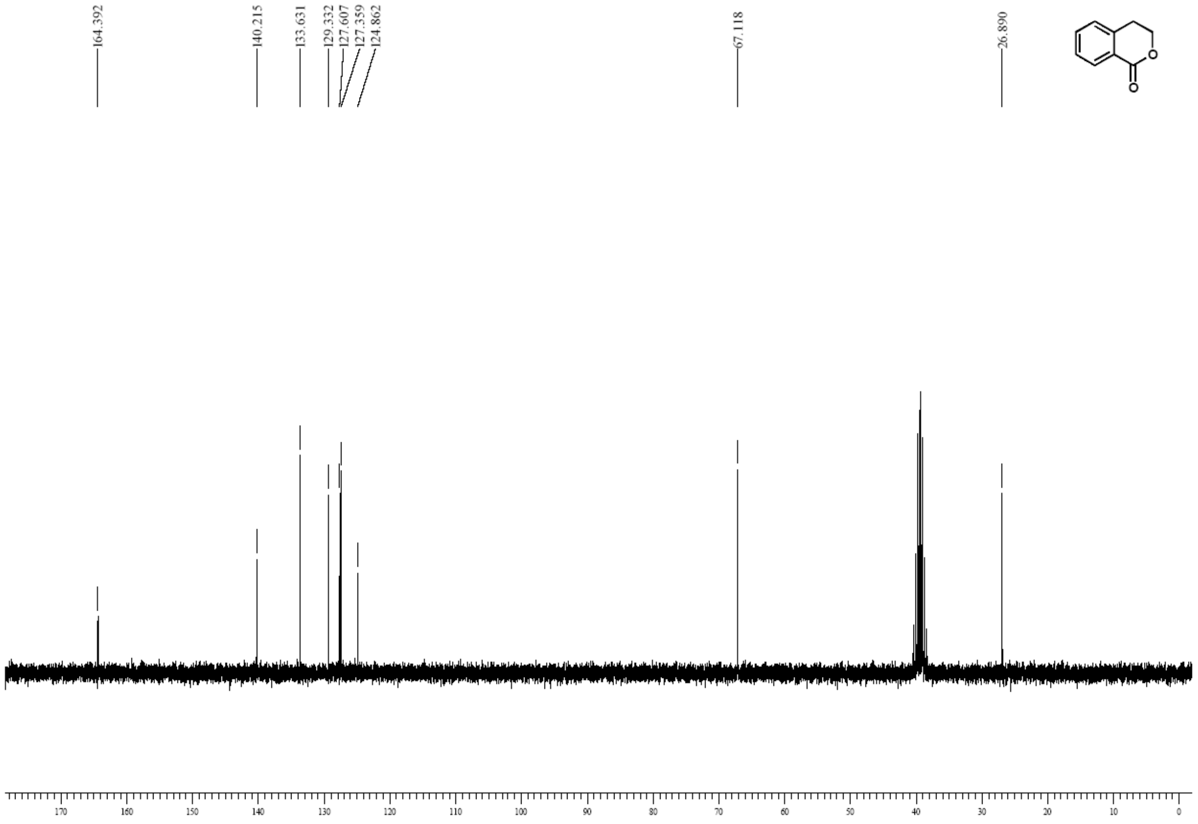
9*H*-xanthen-9-one (2w)[3]



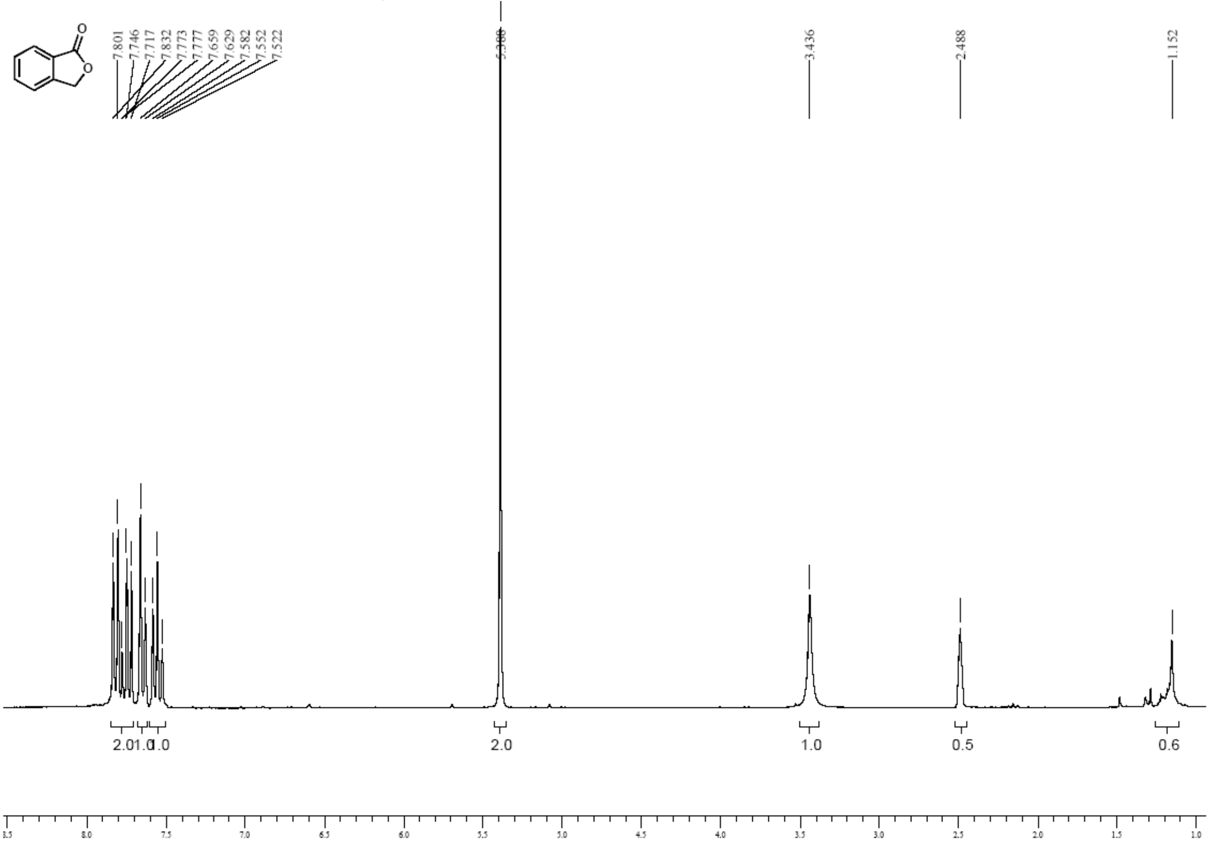


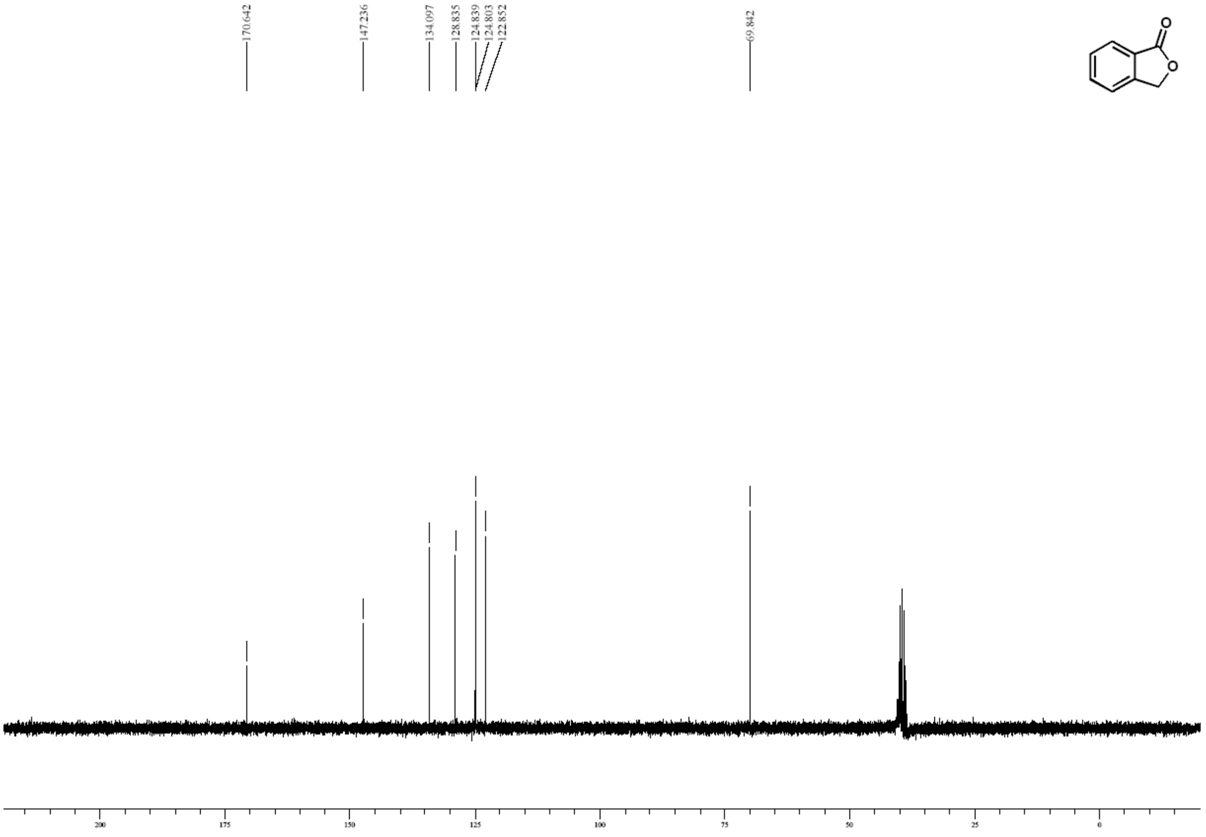
Isochroman-1-one (2x)[3]



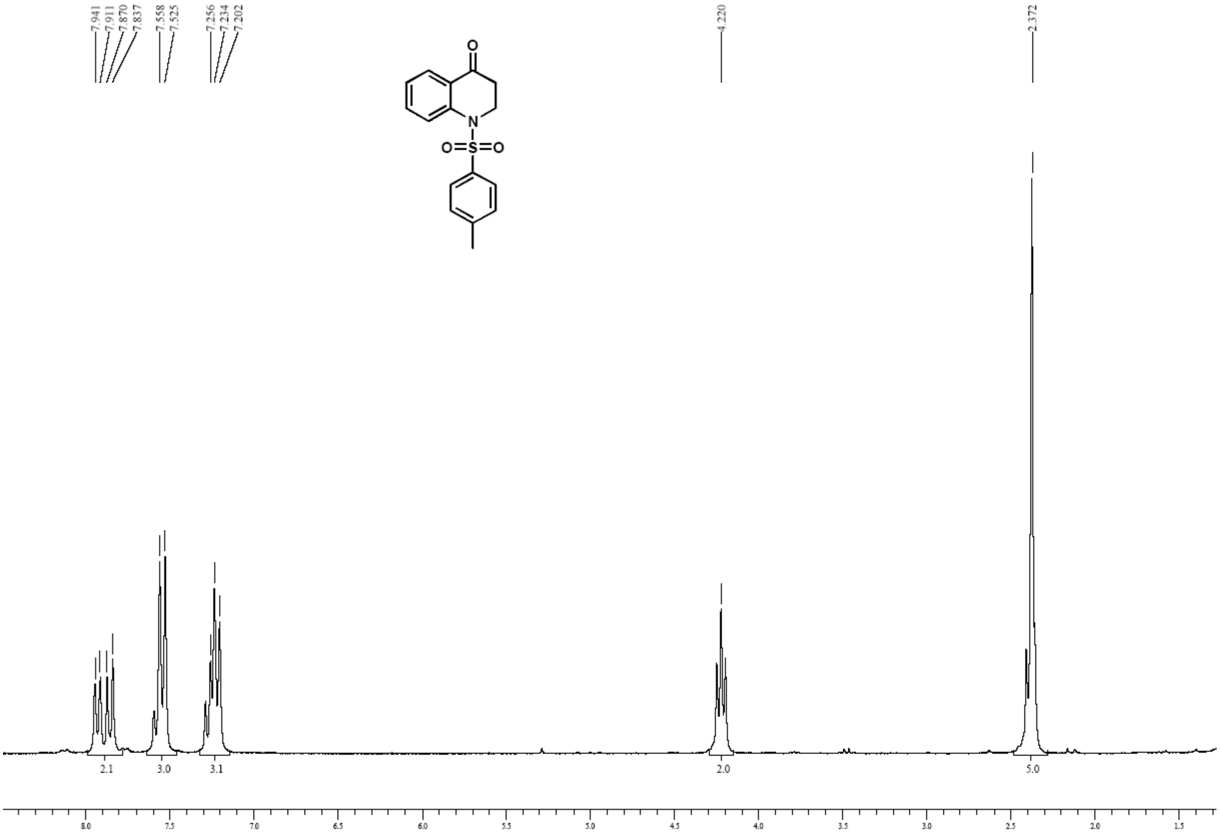


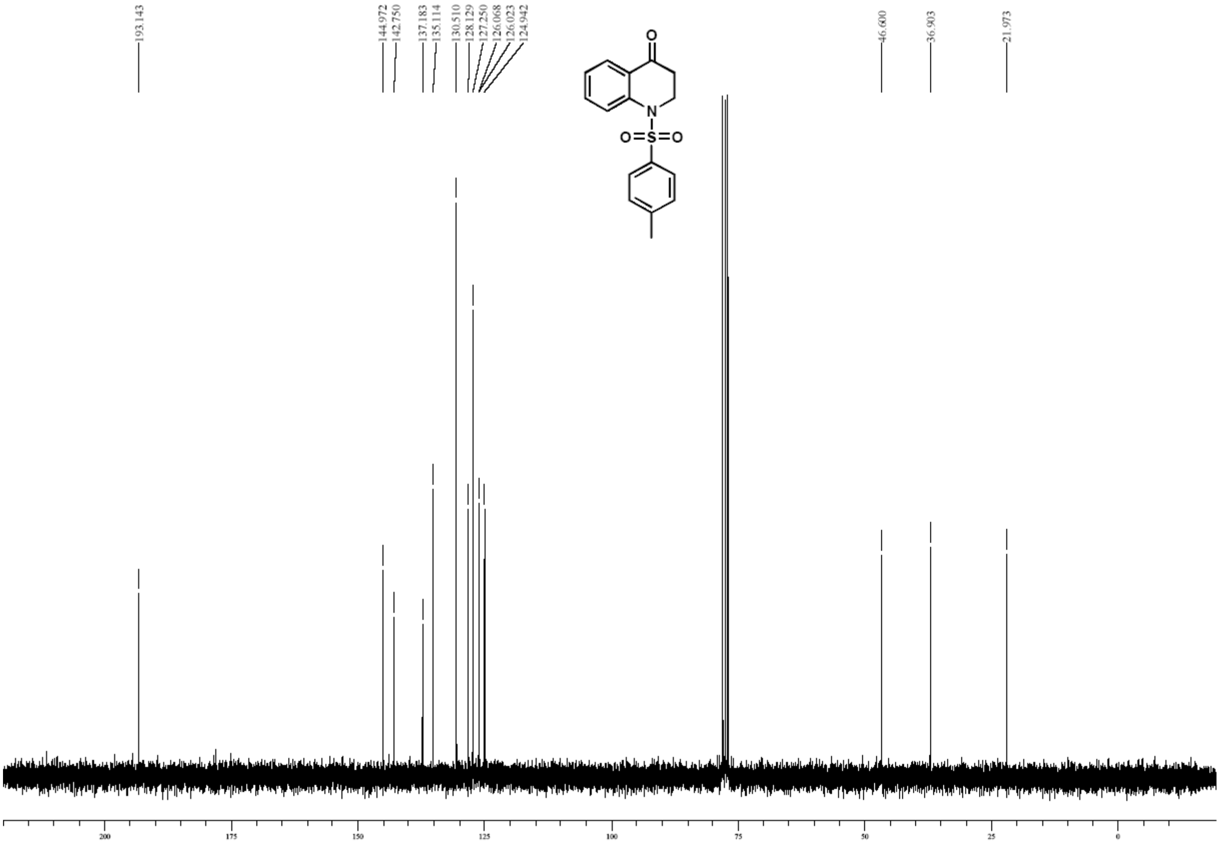
Isobenzofuran-1(3*H*)-one (2y)[3]



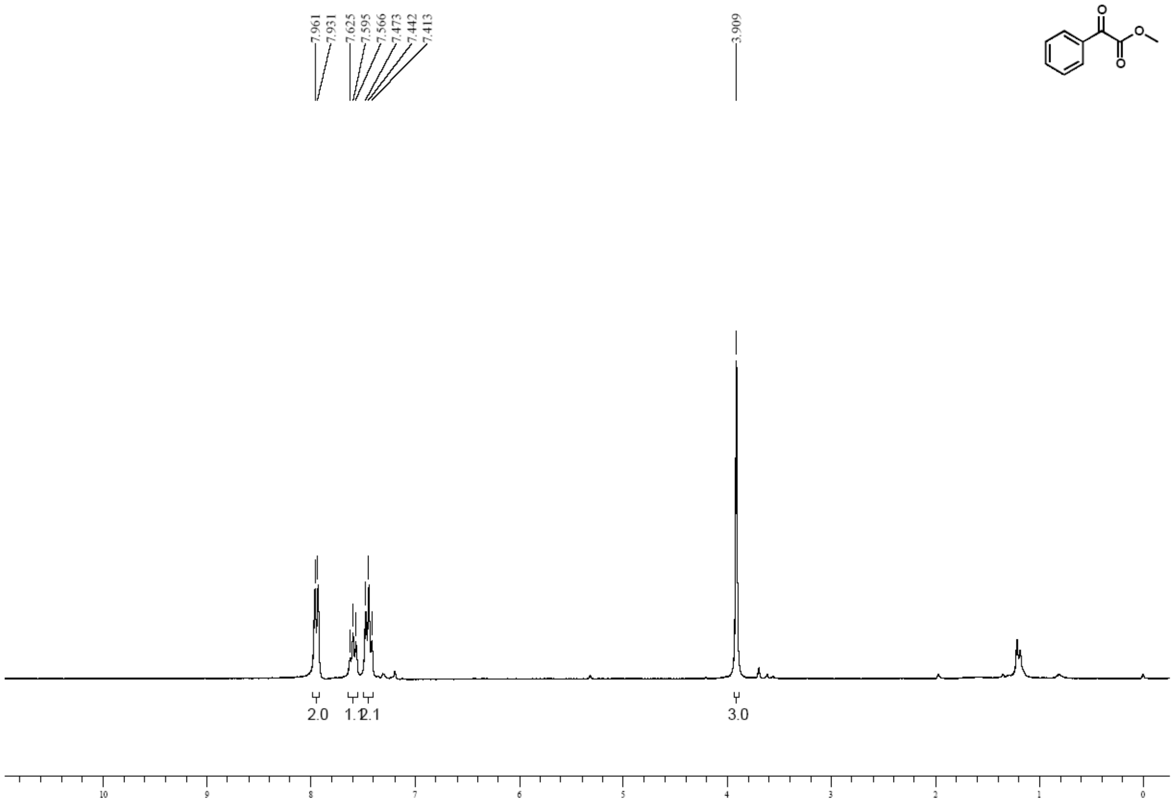


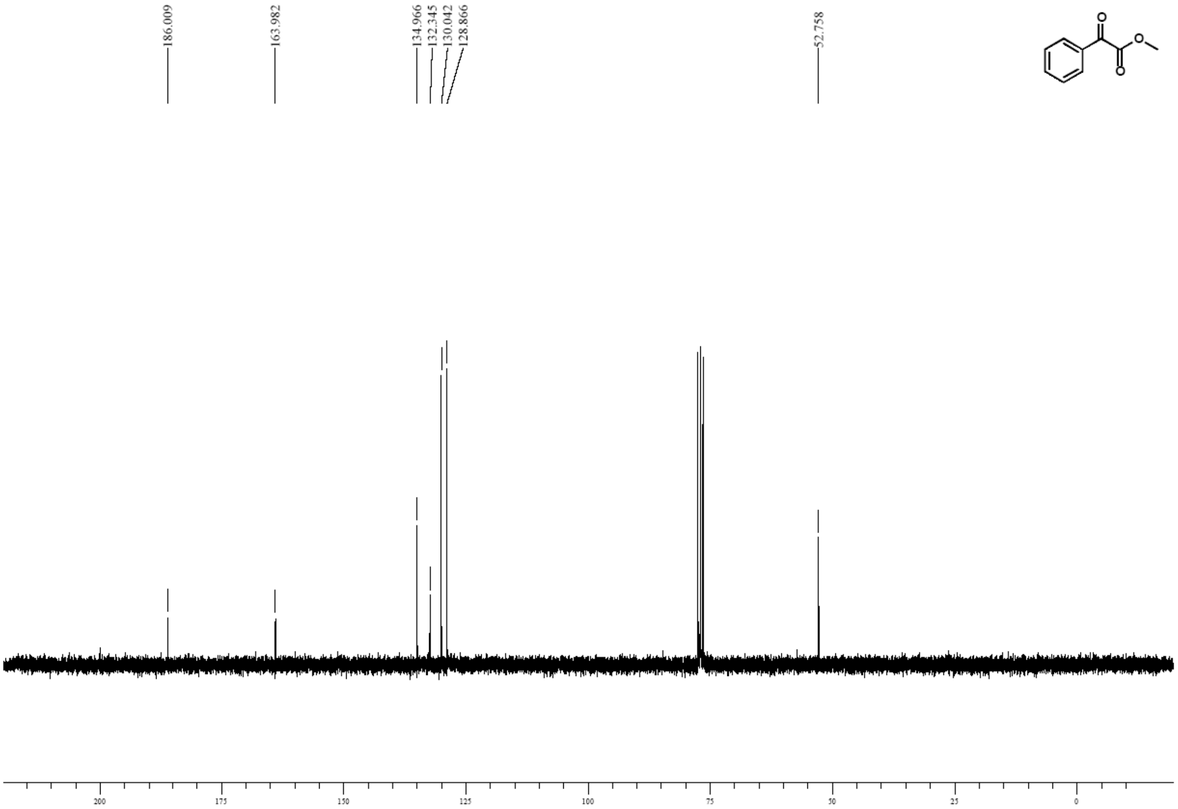
1-tosyl-2,3-dihydroquinolin-4(1*H*)-one (2z)[17]



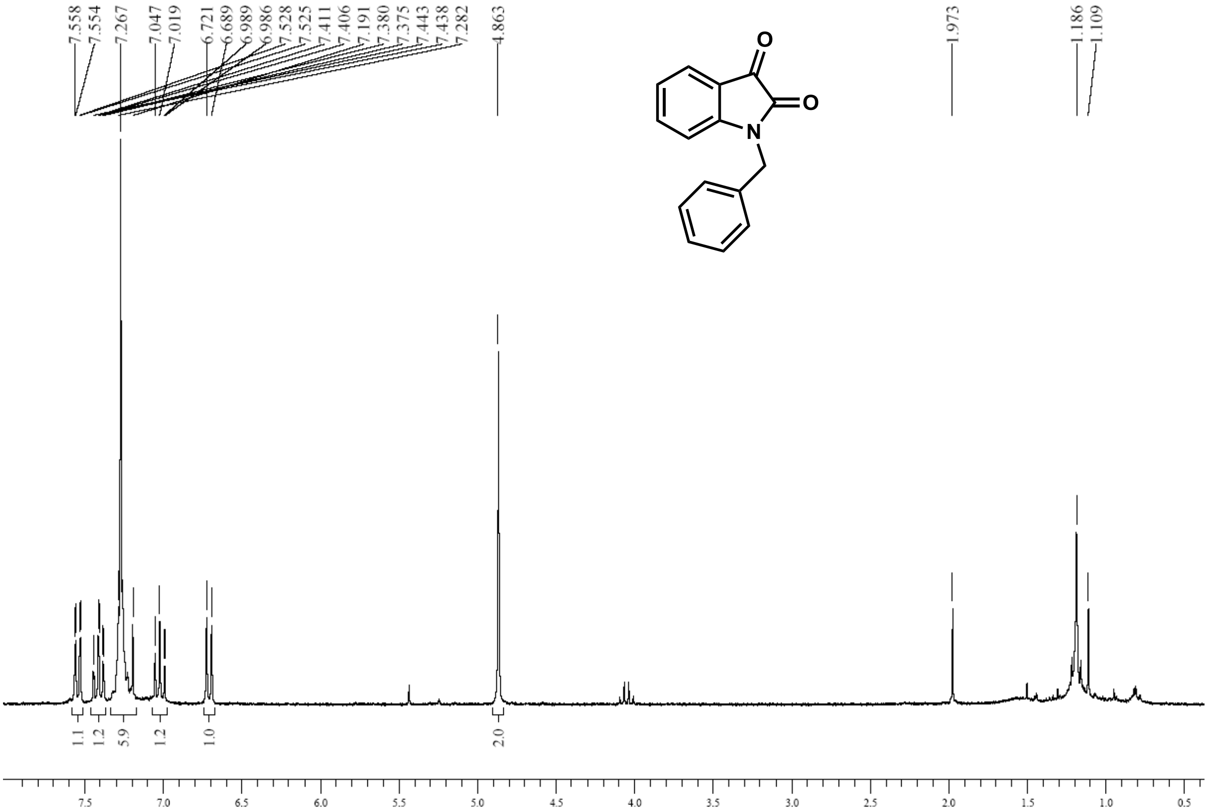


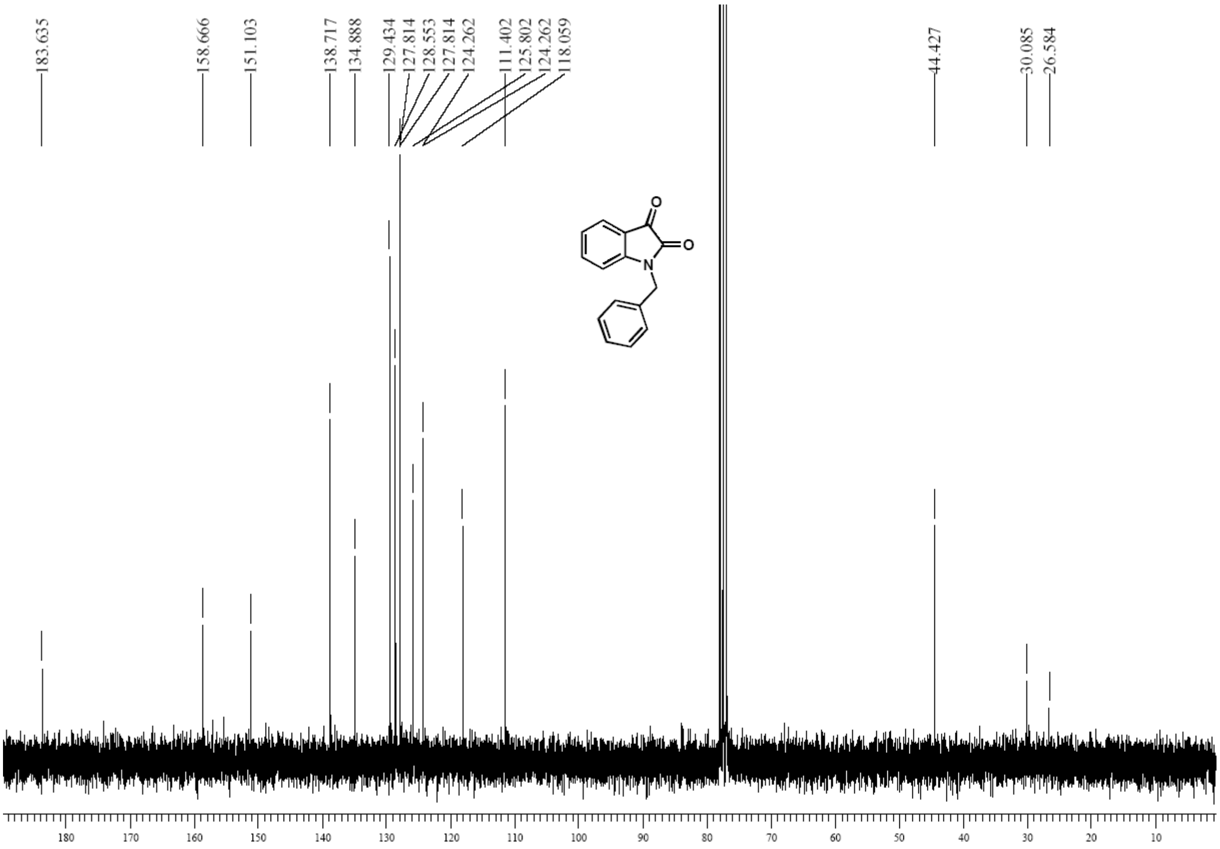
Methyl 2-oxo-2-phenylacetate (2aa)[18]



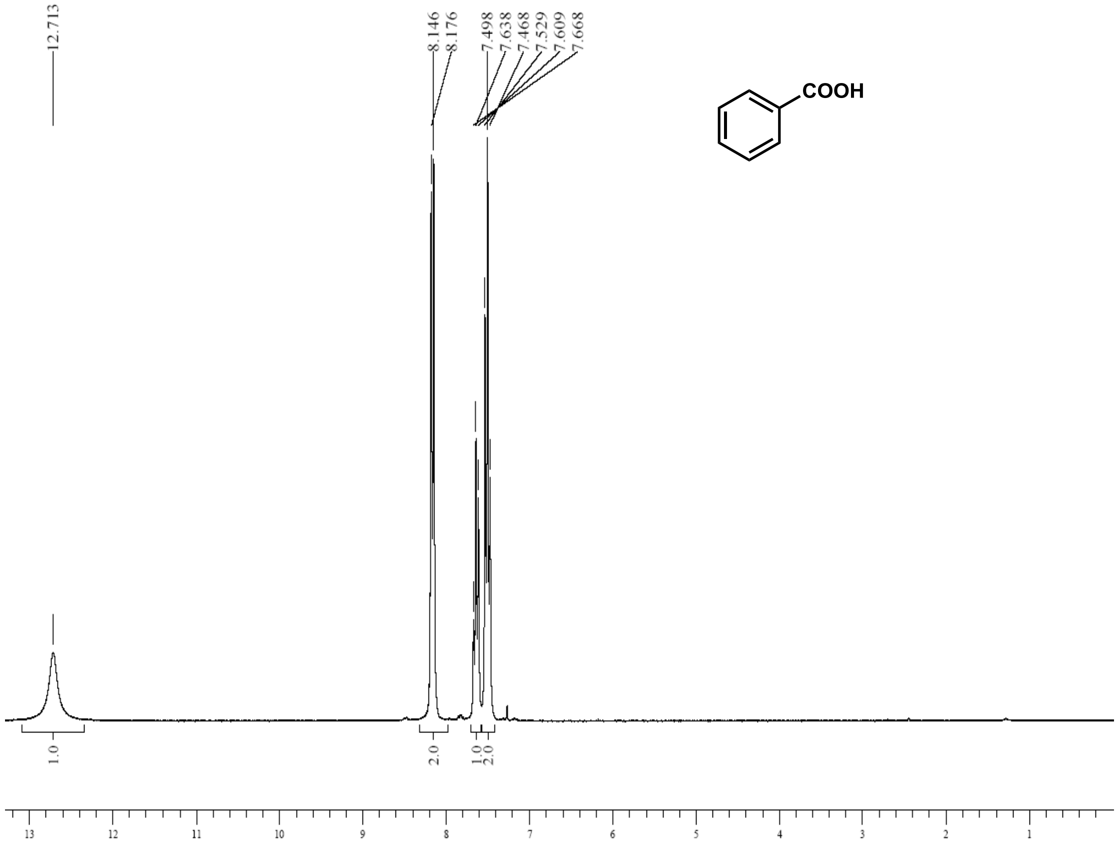


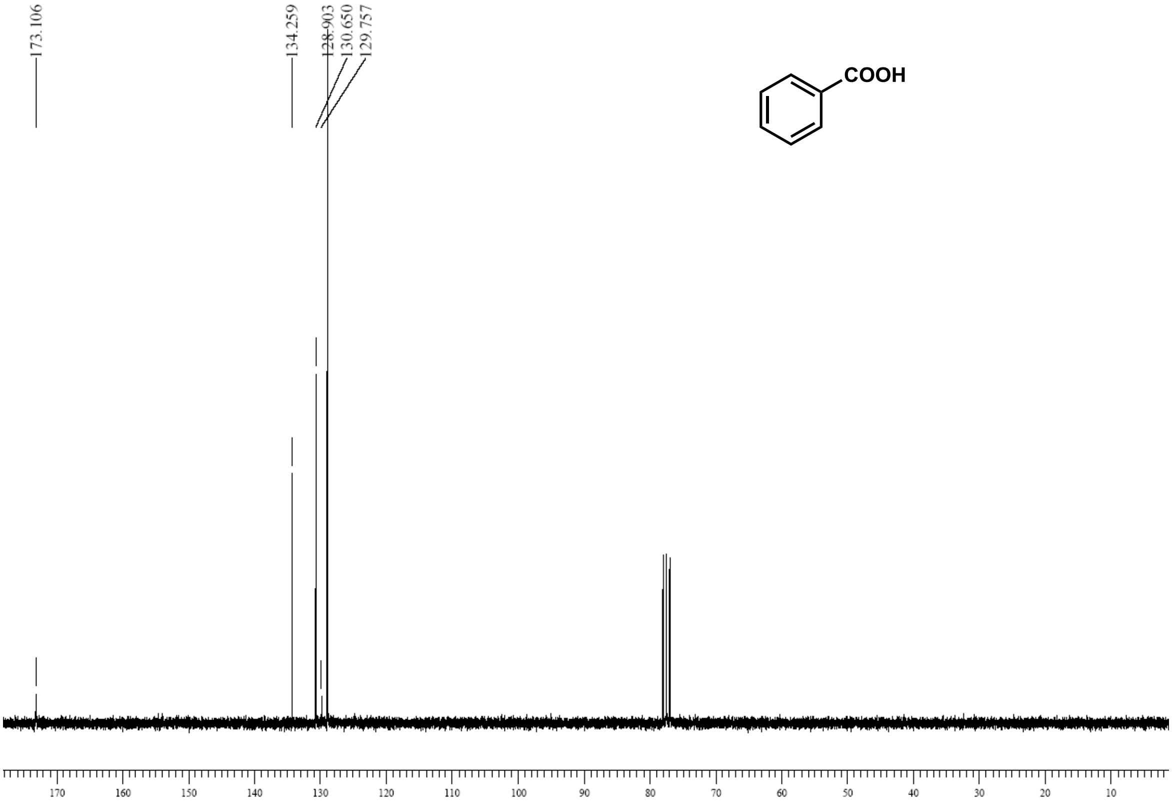
1-benzyl isatin (2bb)[19]





Benzoic acid (2cc)[20]





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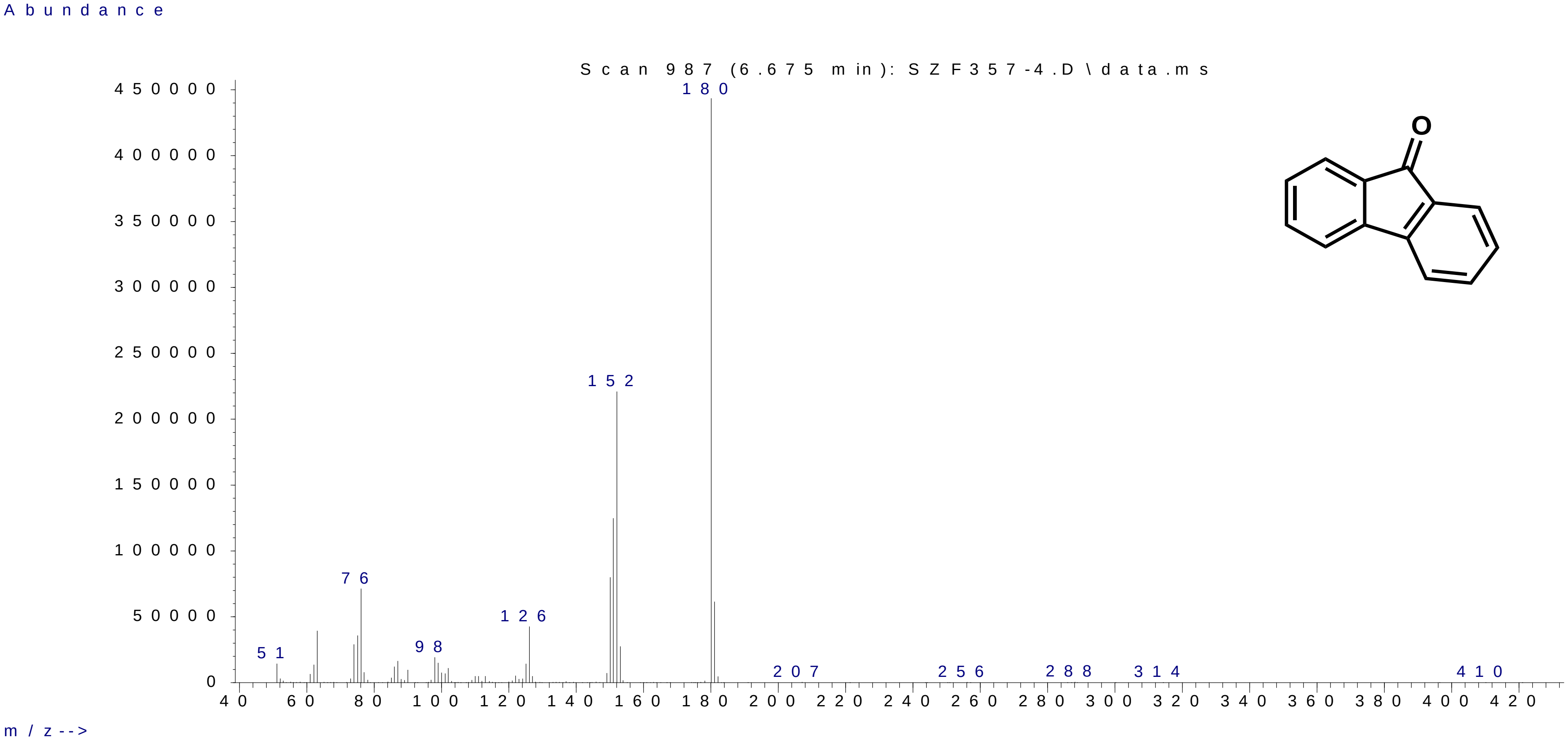
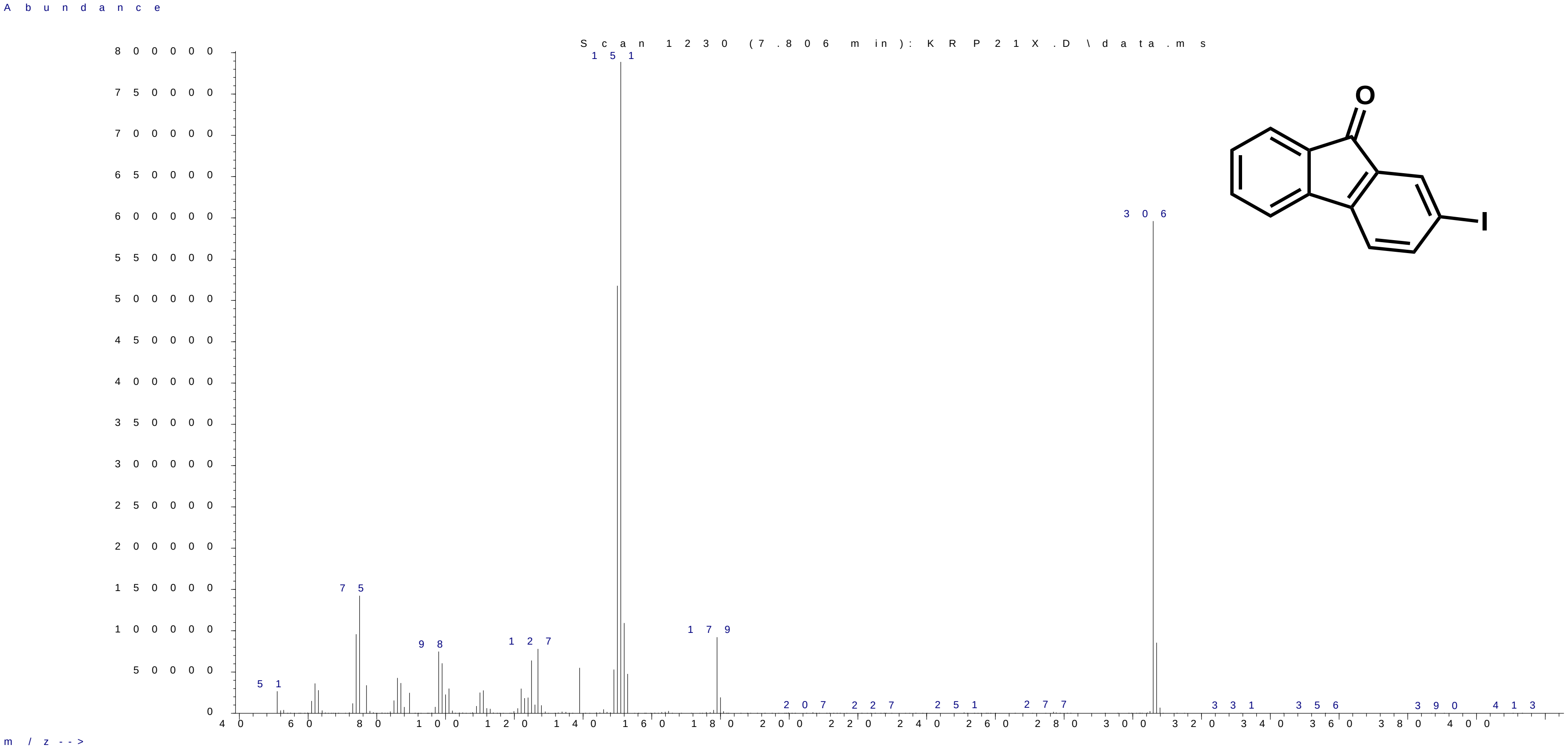
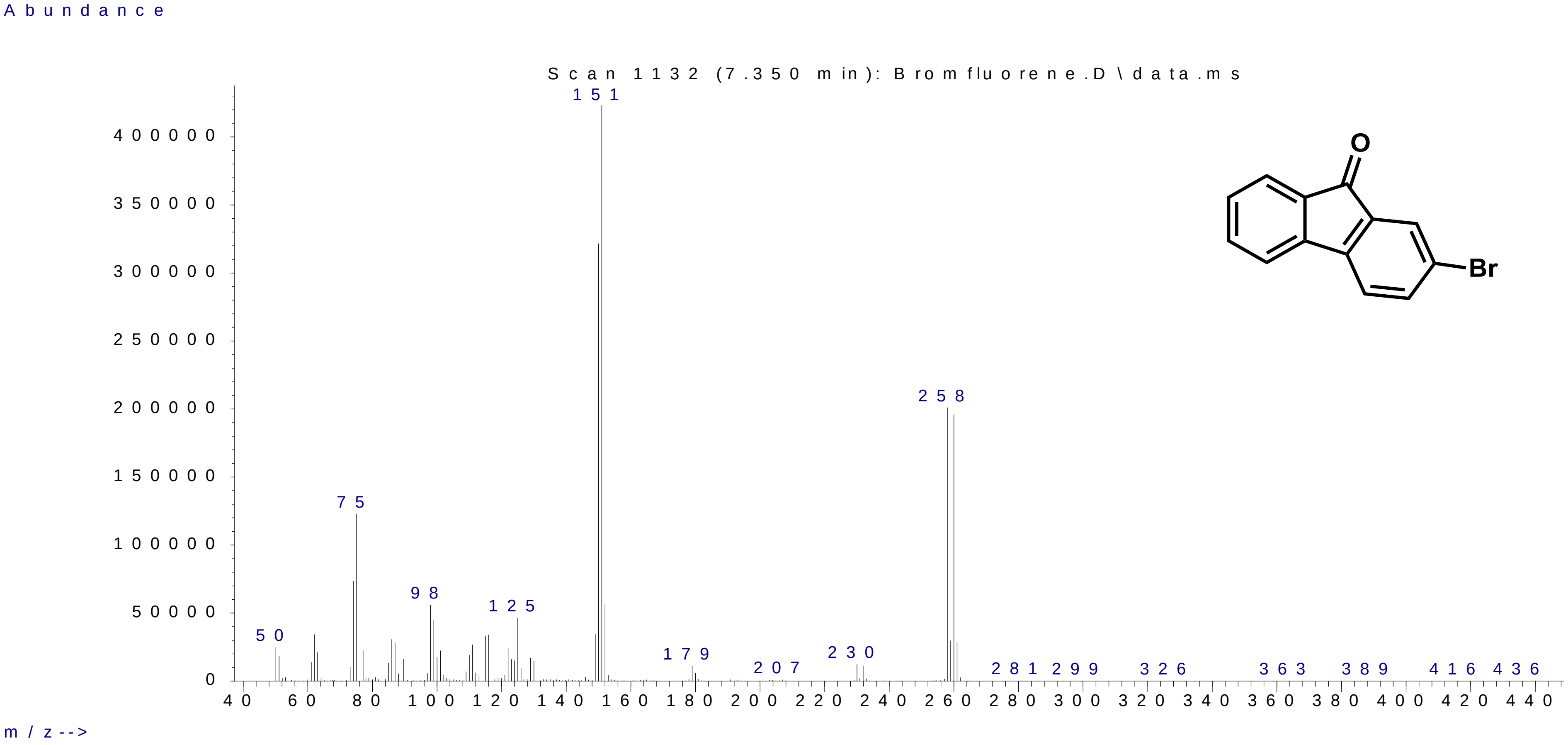
Fluorene-9-one (2a)[3]



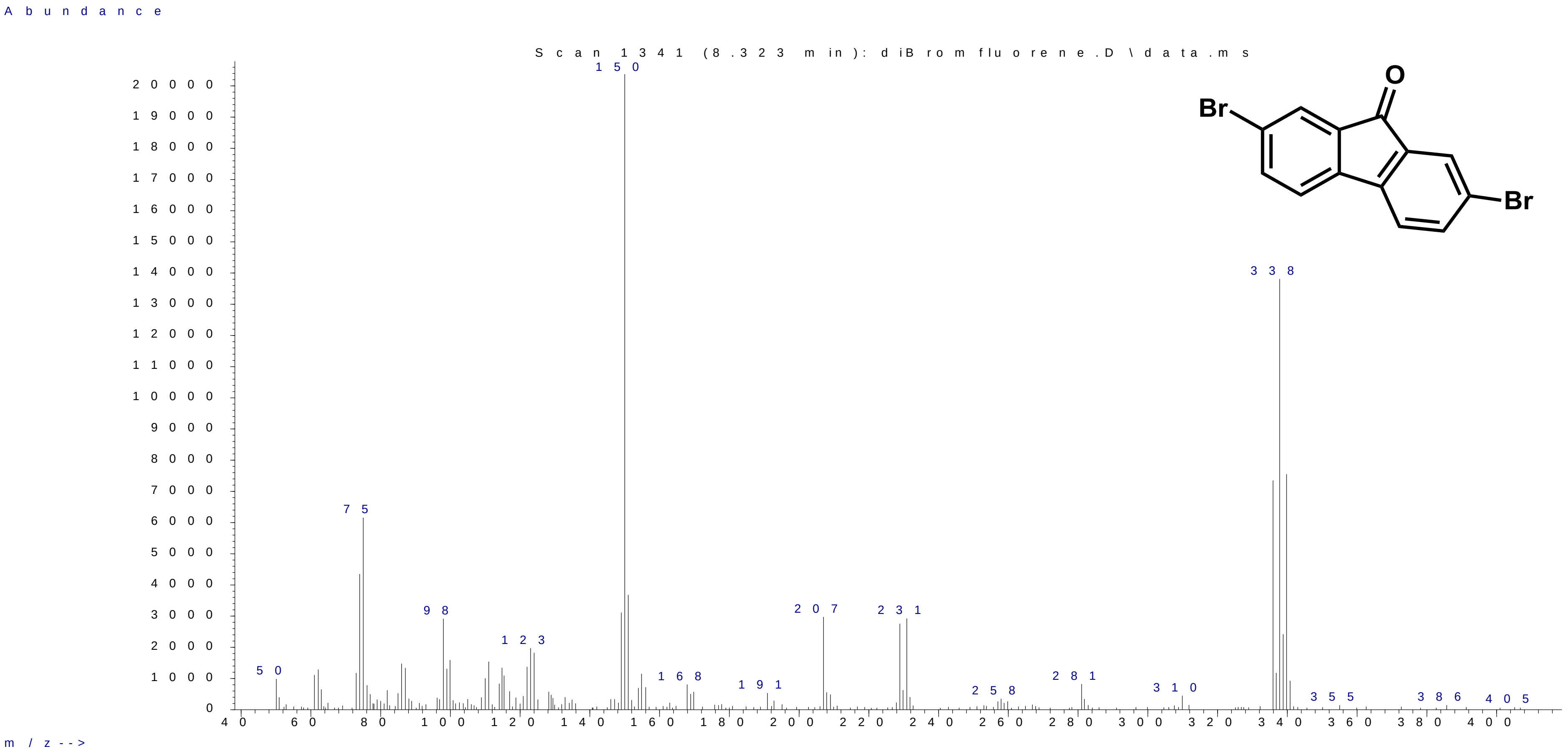
2-iodo-9*H*-fluoren-9-one (2b)[4]



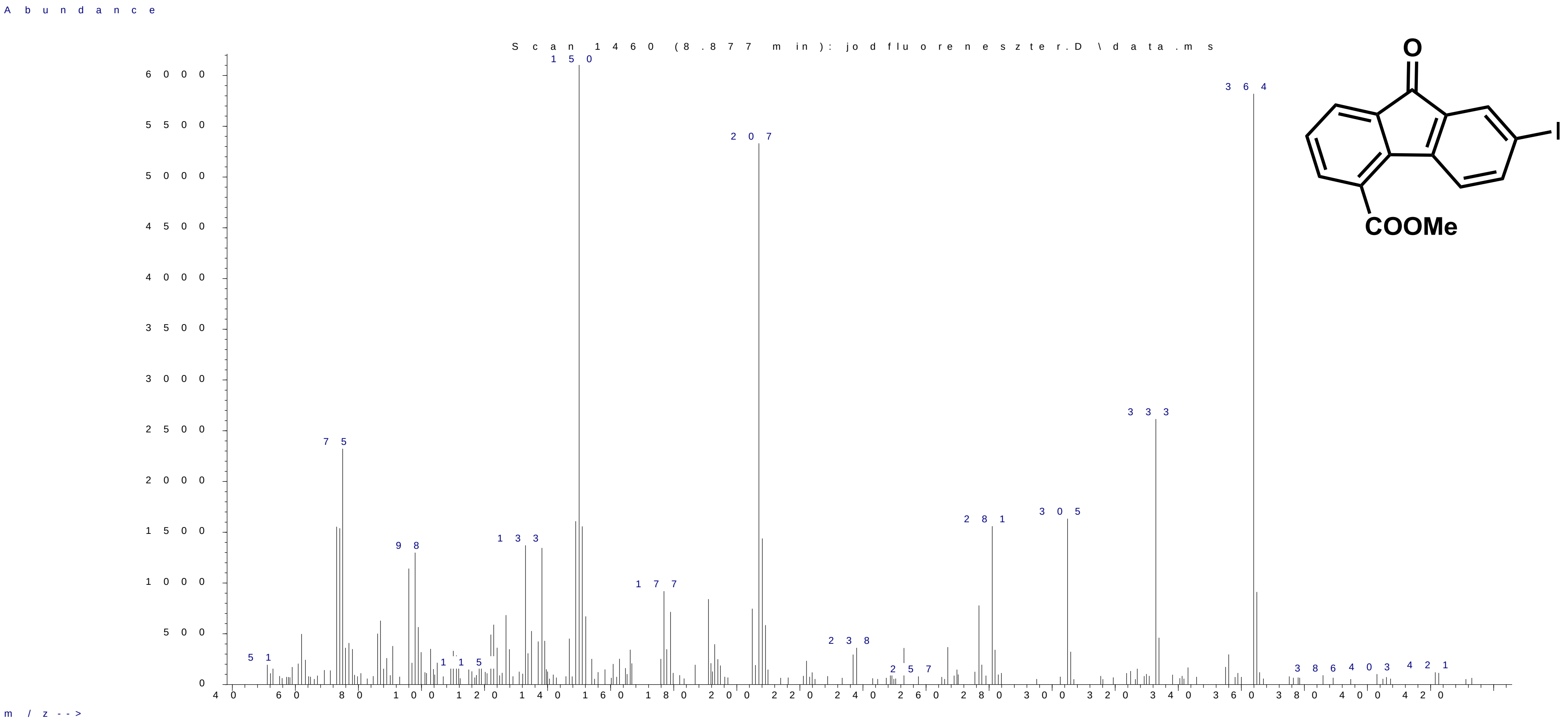
2-bromo-9*H*-fluoren-9-one (2d)[3]



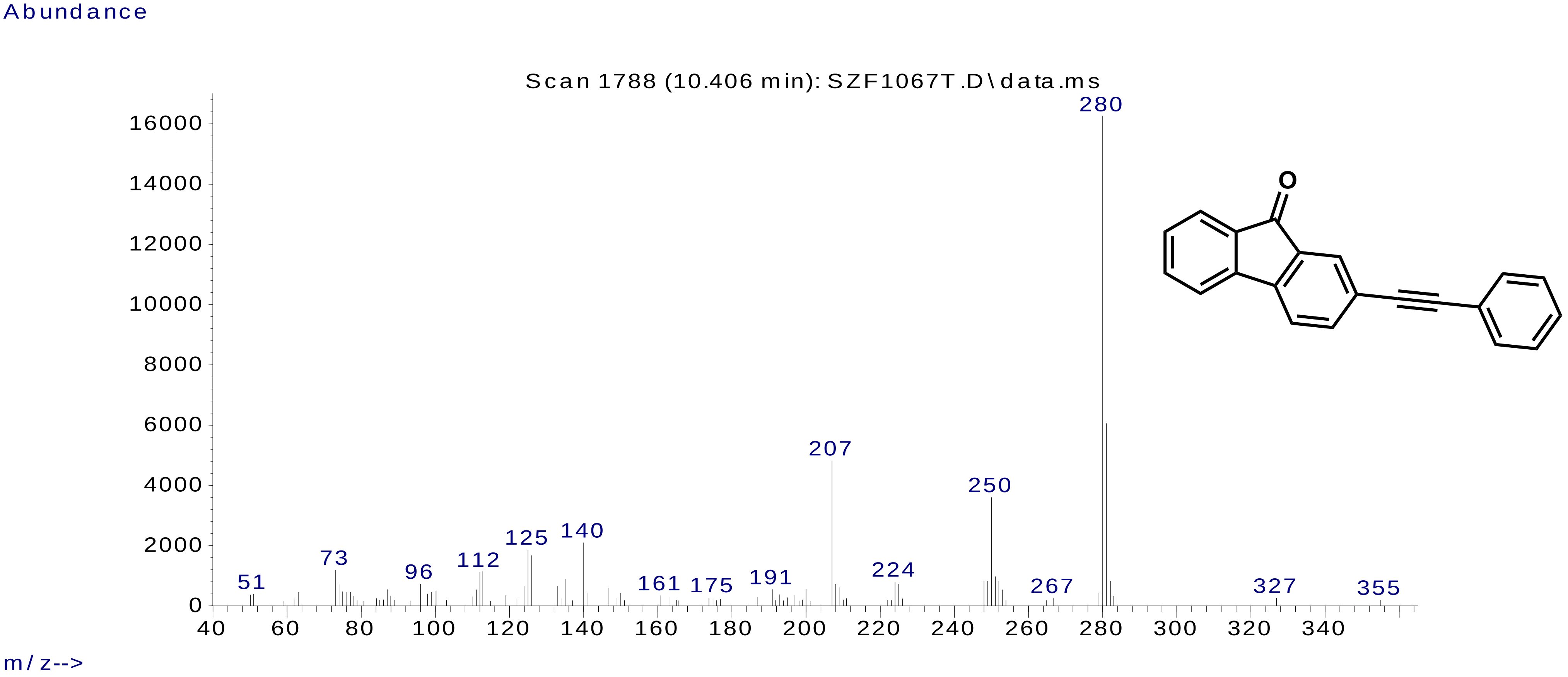
2,7-dibromo-9*H*-fluoren-9-one (2e) [5,6]



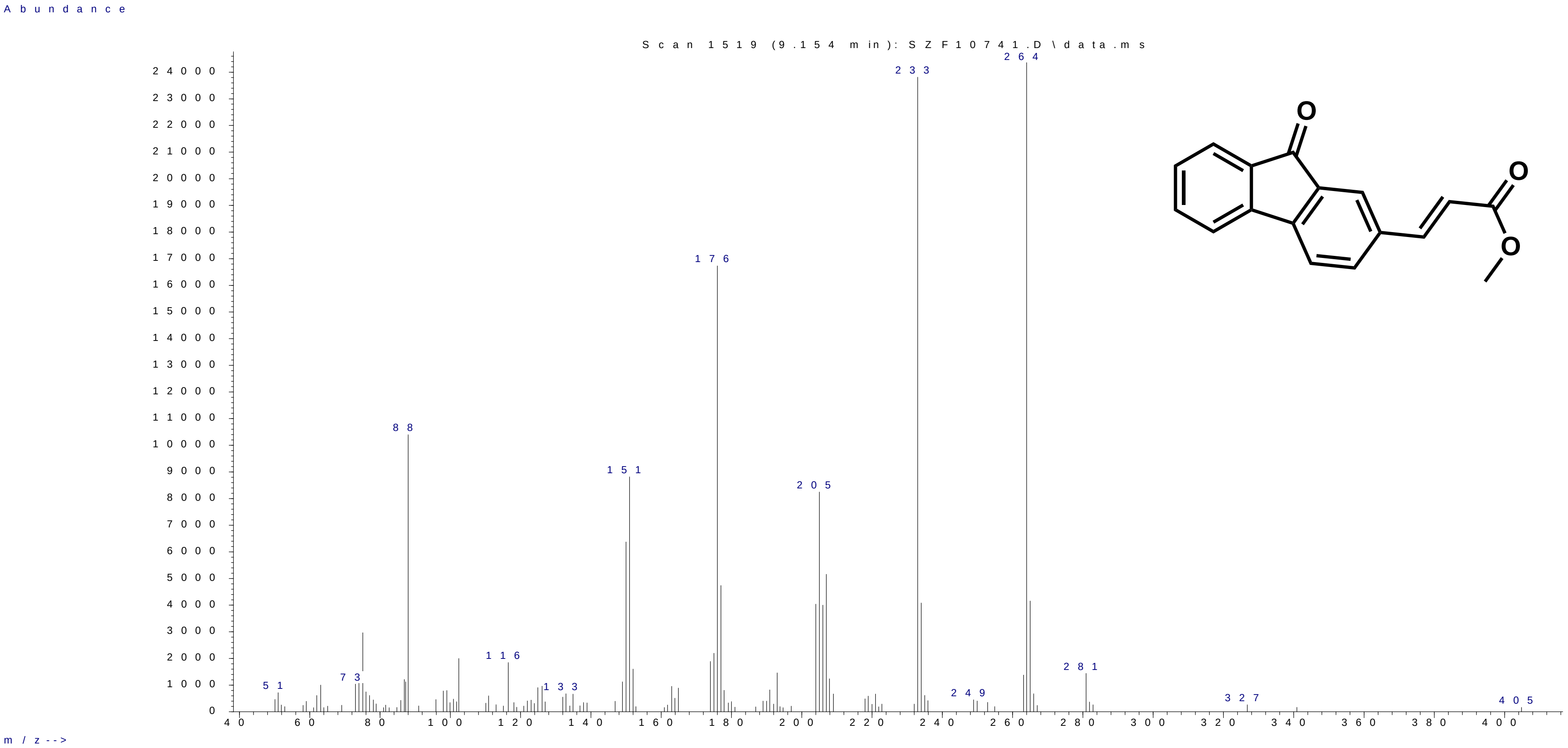
Methyl 7-iodo-9-oxo-9*H*-fluorene-4-carboxylate (2f)



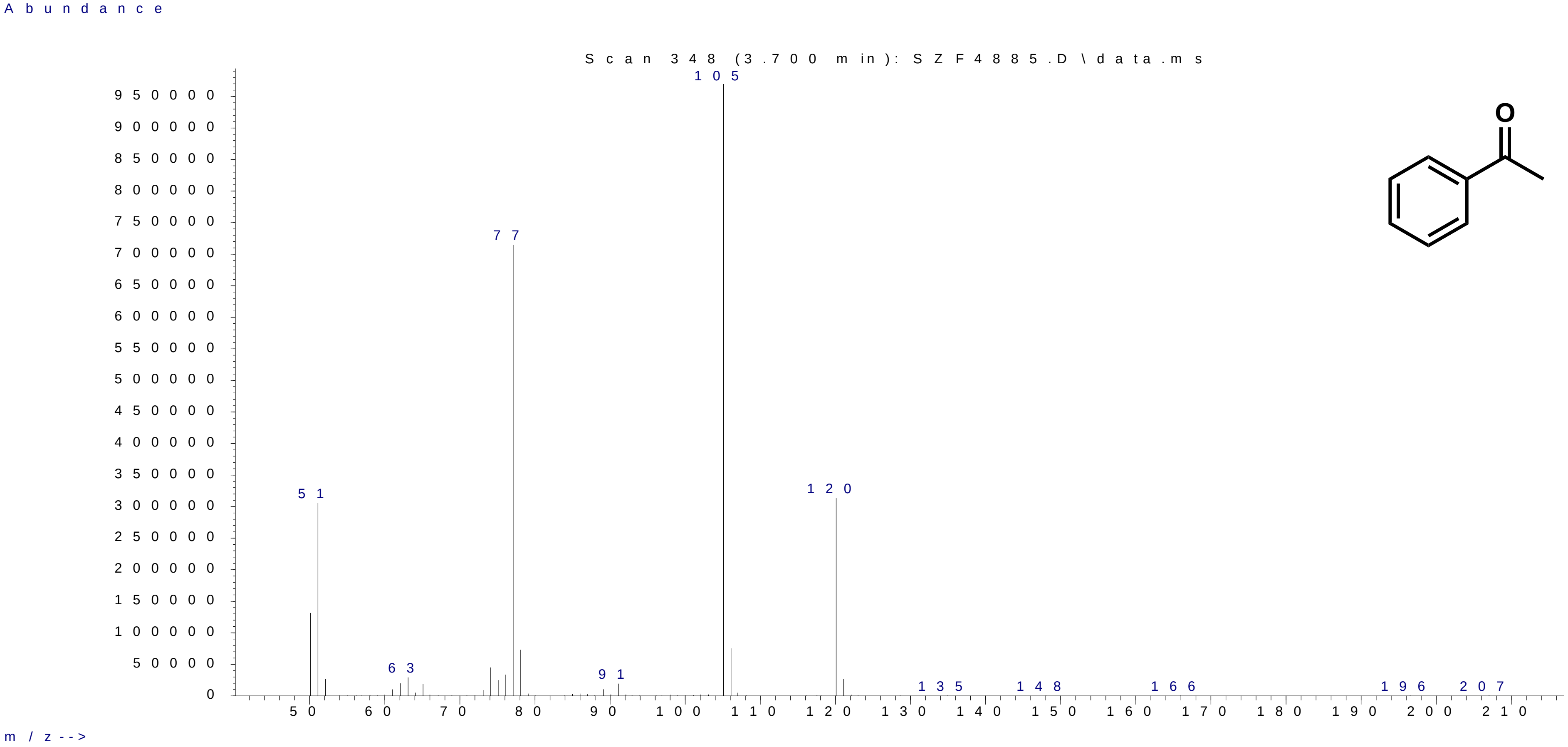
2-(phenylethynyl)-9*H*-fluoren-9-one (2g)[7]



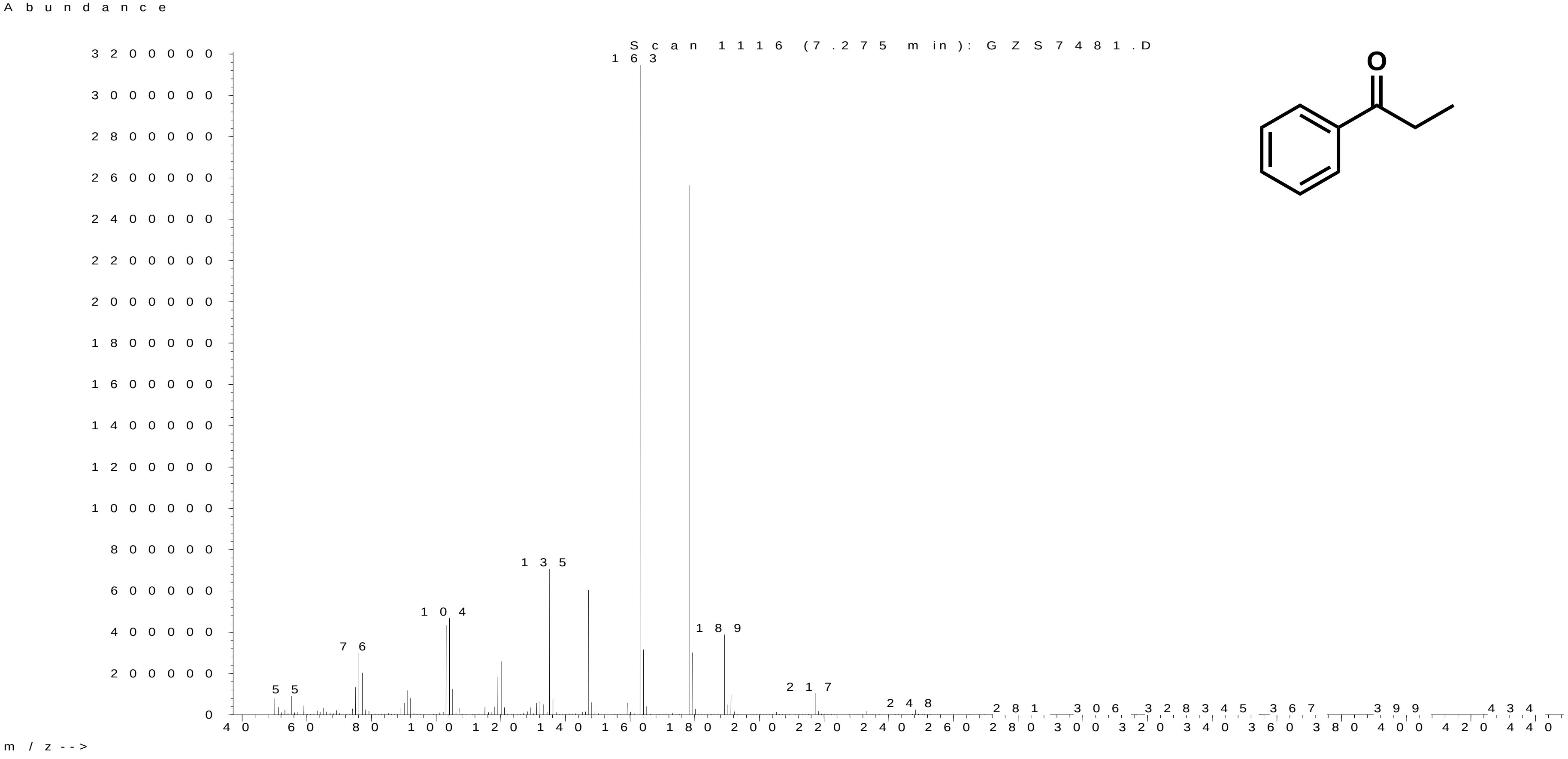
(E)-methyl 3-(9-oxo-9*H*-fluoren-2-yl)acrylate (2h)



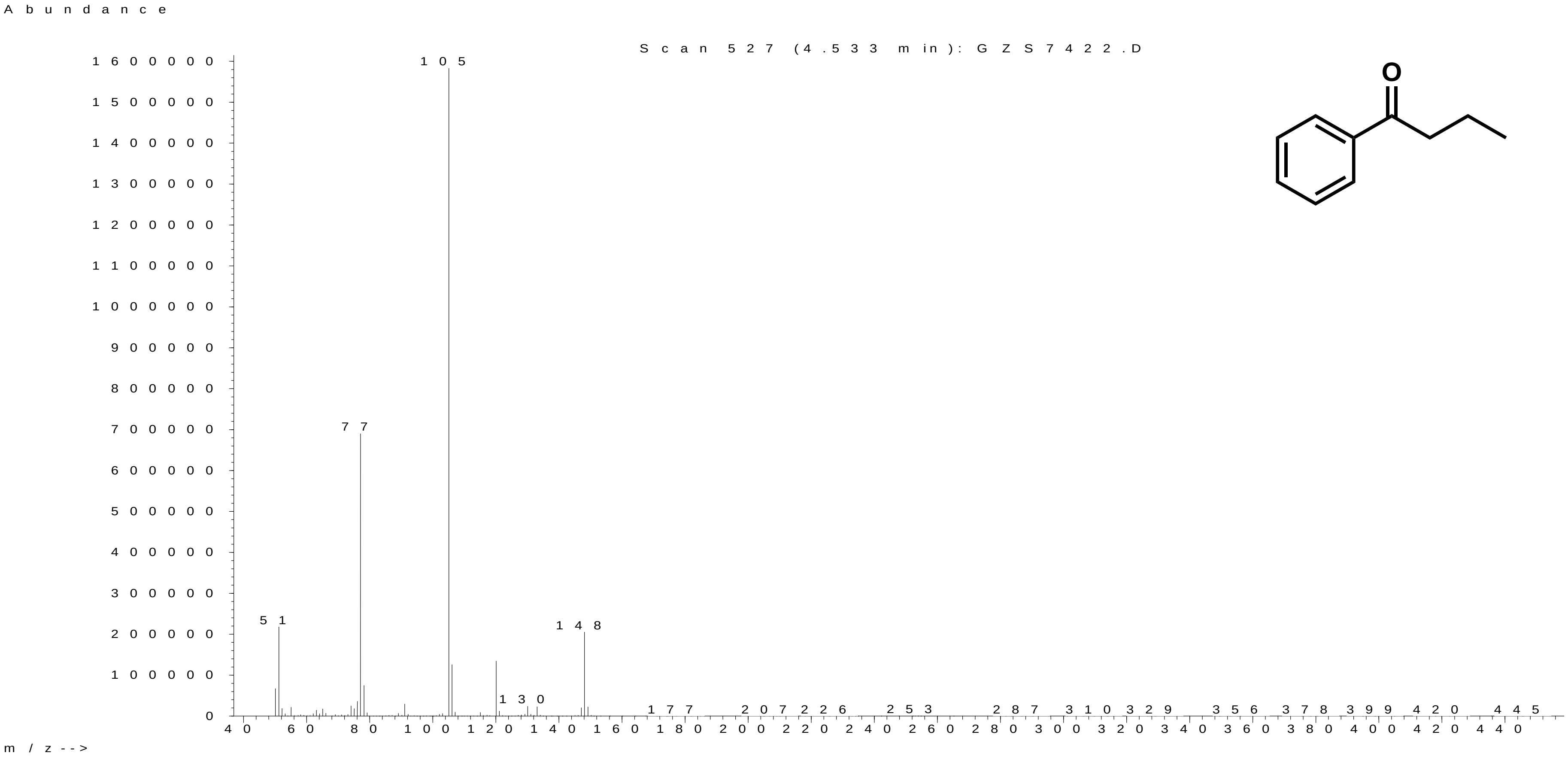
Acetophenone (2i)[3]



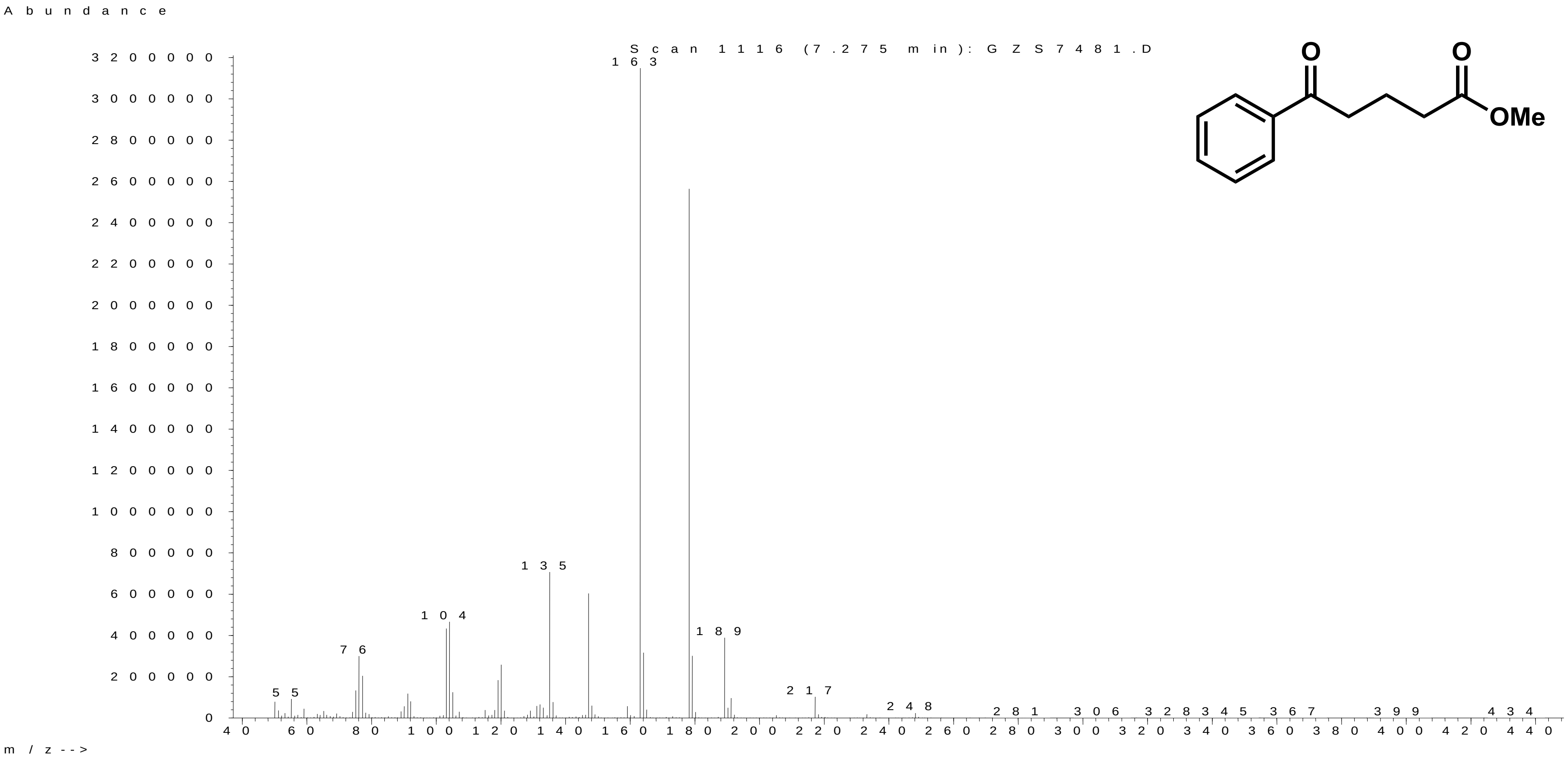
Propiophenone (2j)[8]



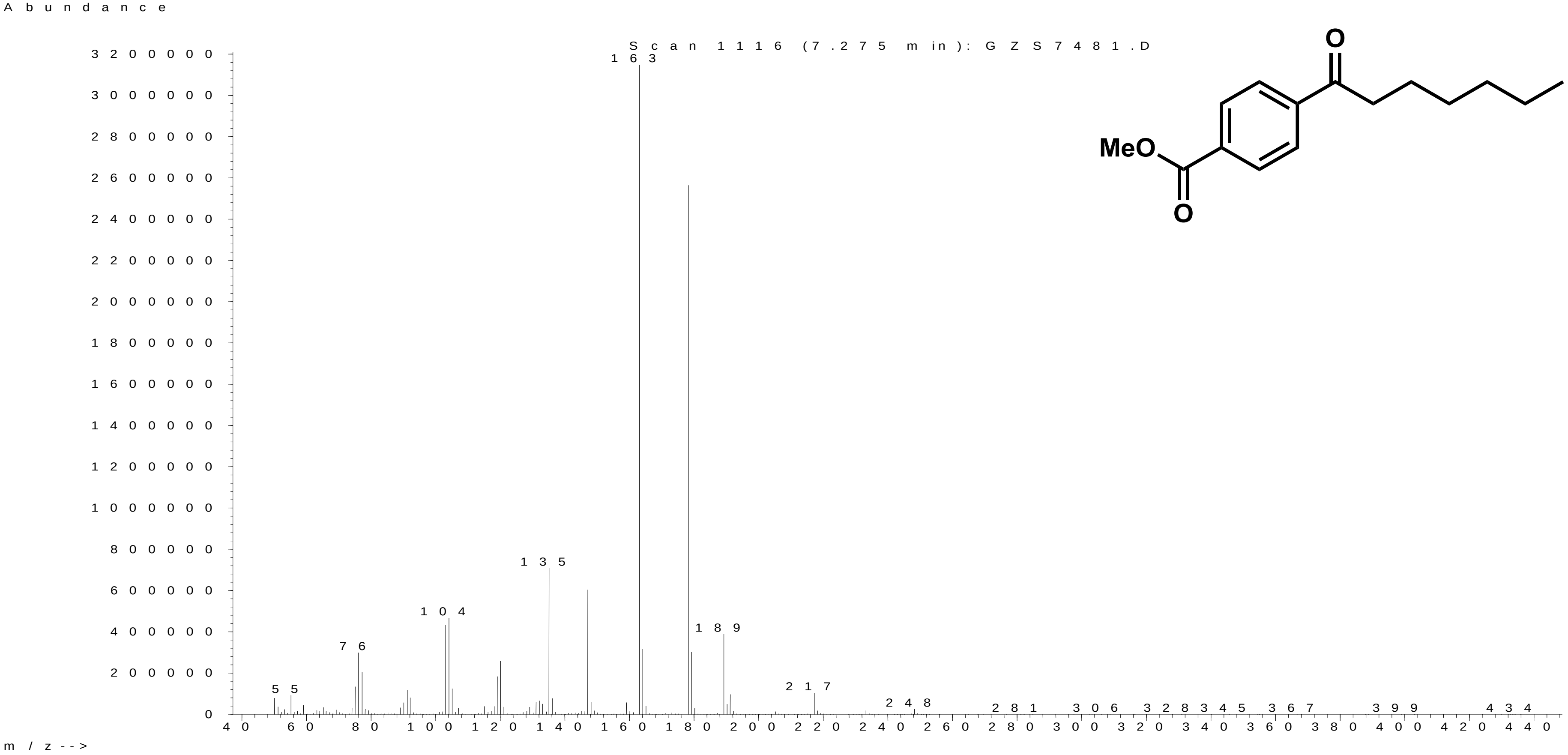
Butirophenone (2k)[9]



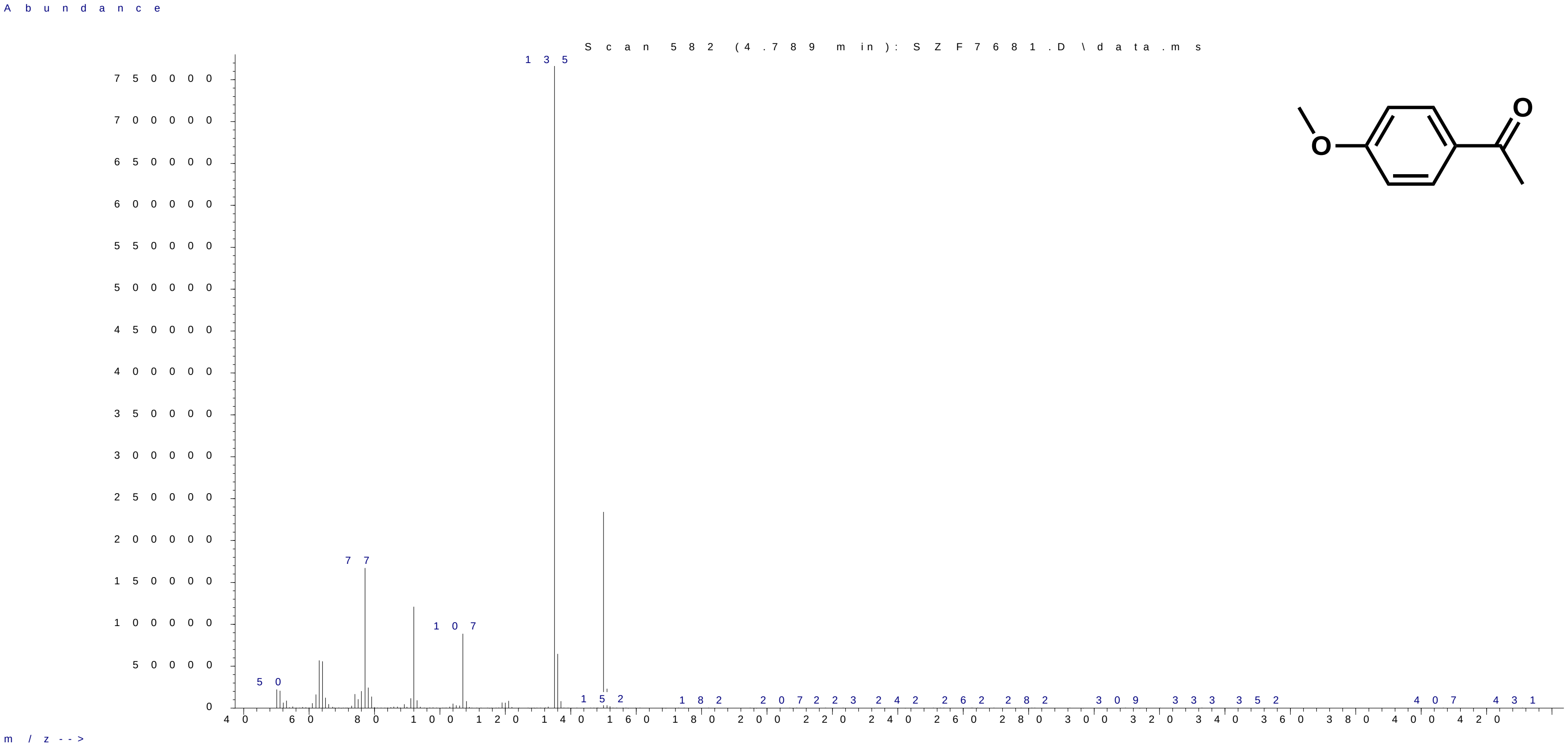
Methyl 5-oxo-5-phenylpentanoate (2l)[10]



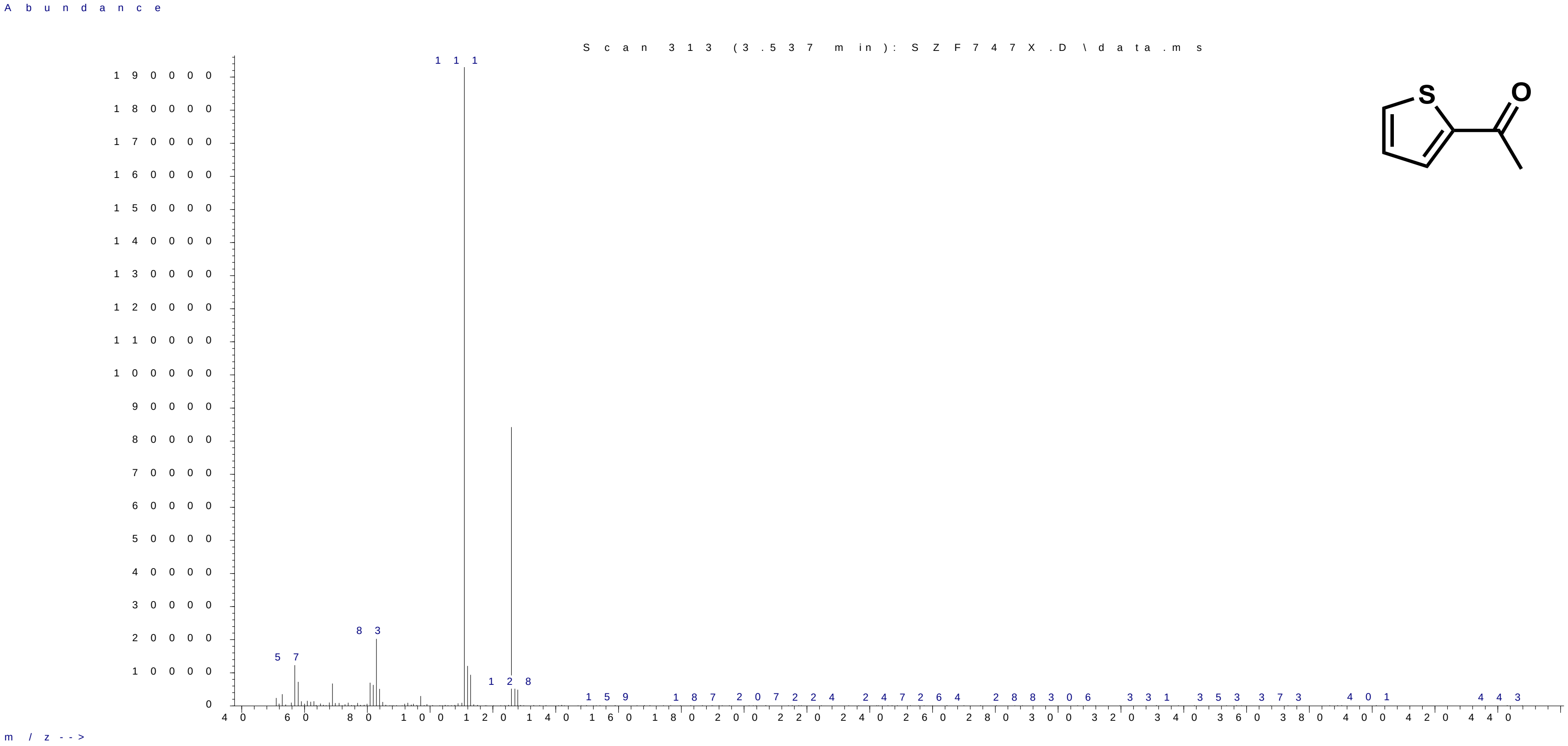
Methyl 4-heptanoylbenzoate (2m)[11]



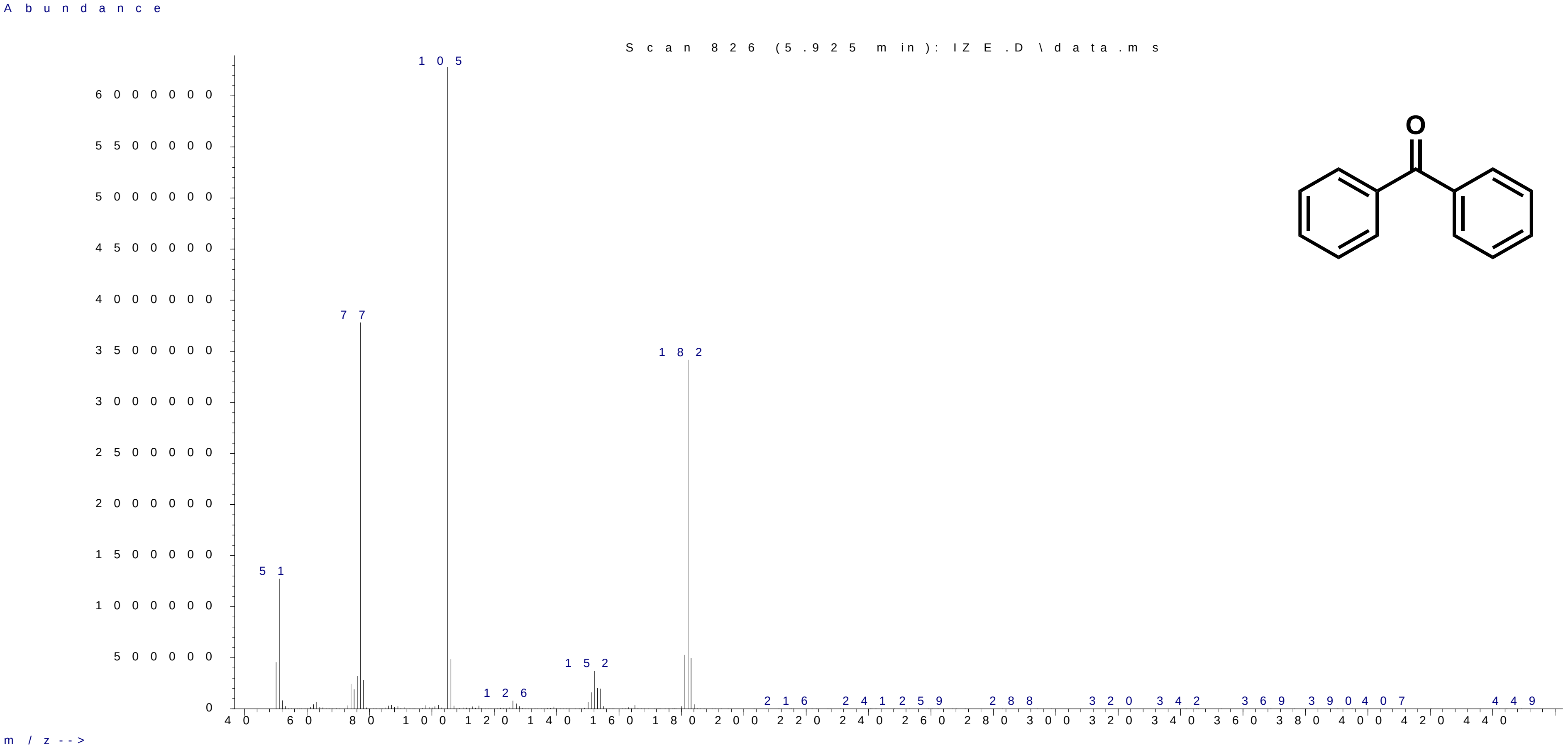
1-(4-methoxyphenyl)ethanone (2n)[12]



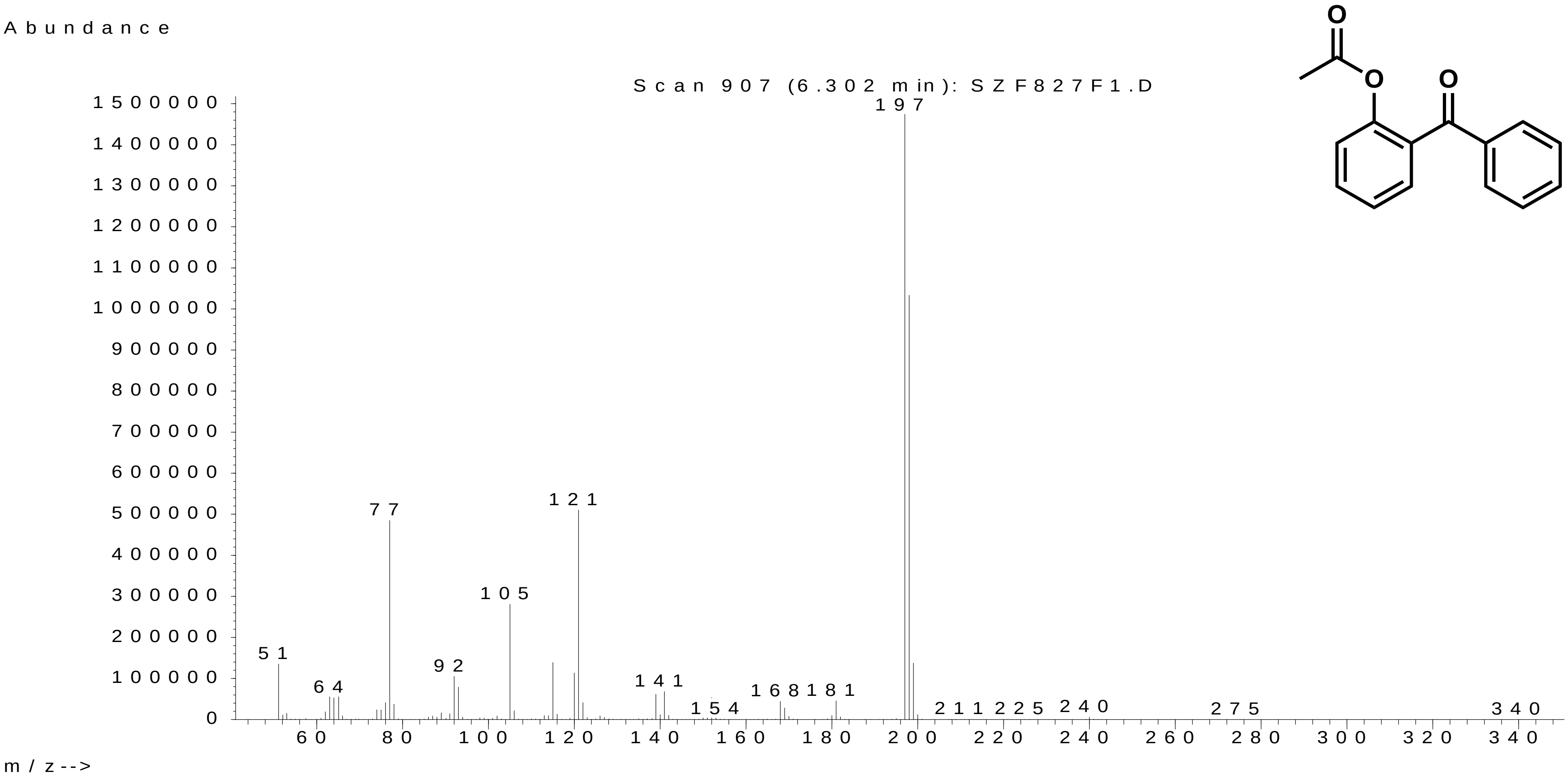
1-(thiophen-2-yl)ethanone (2p)[13]



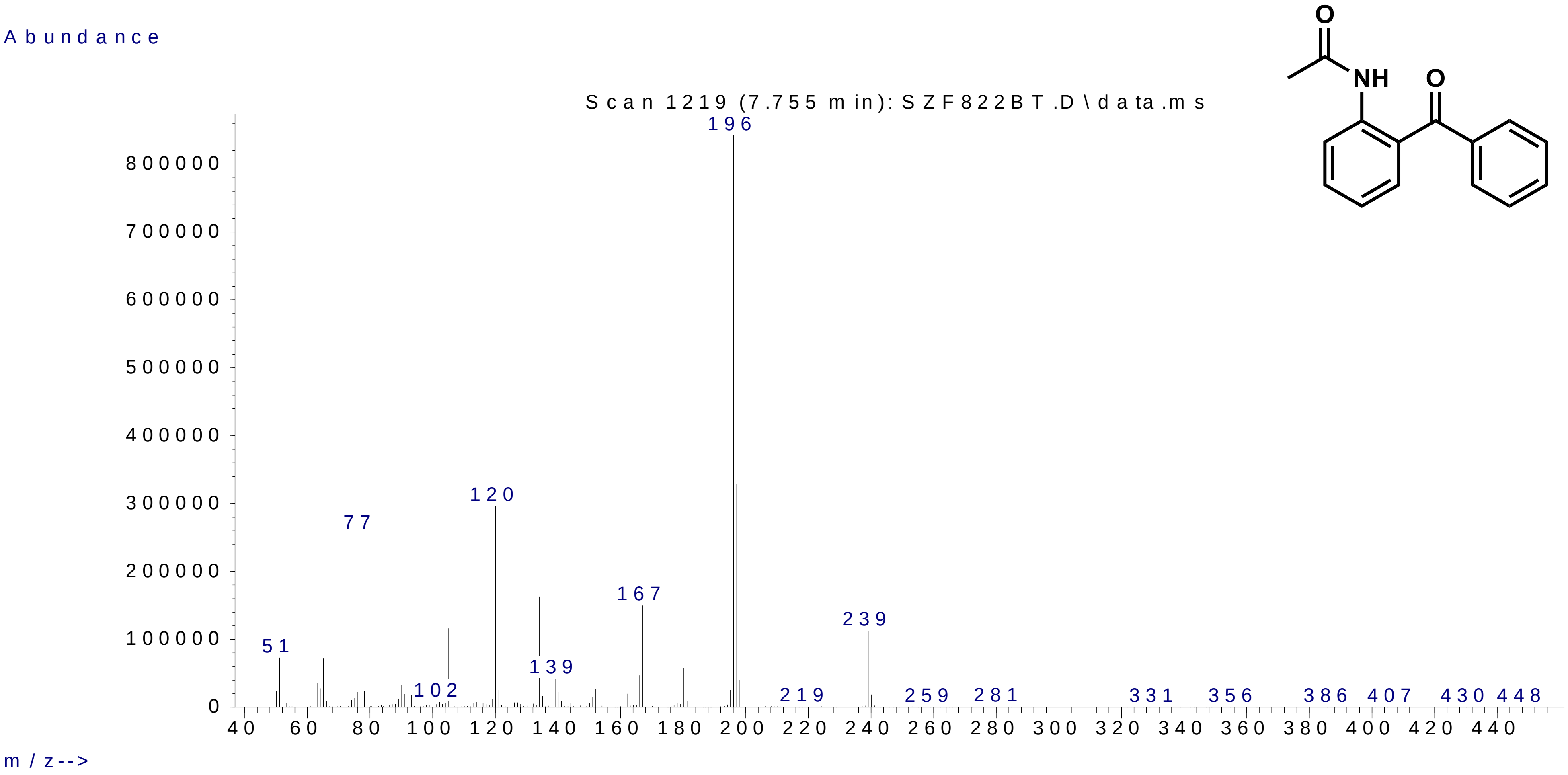
Benzophenone (2q)[3]



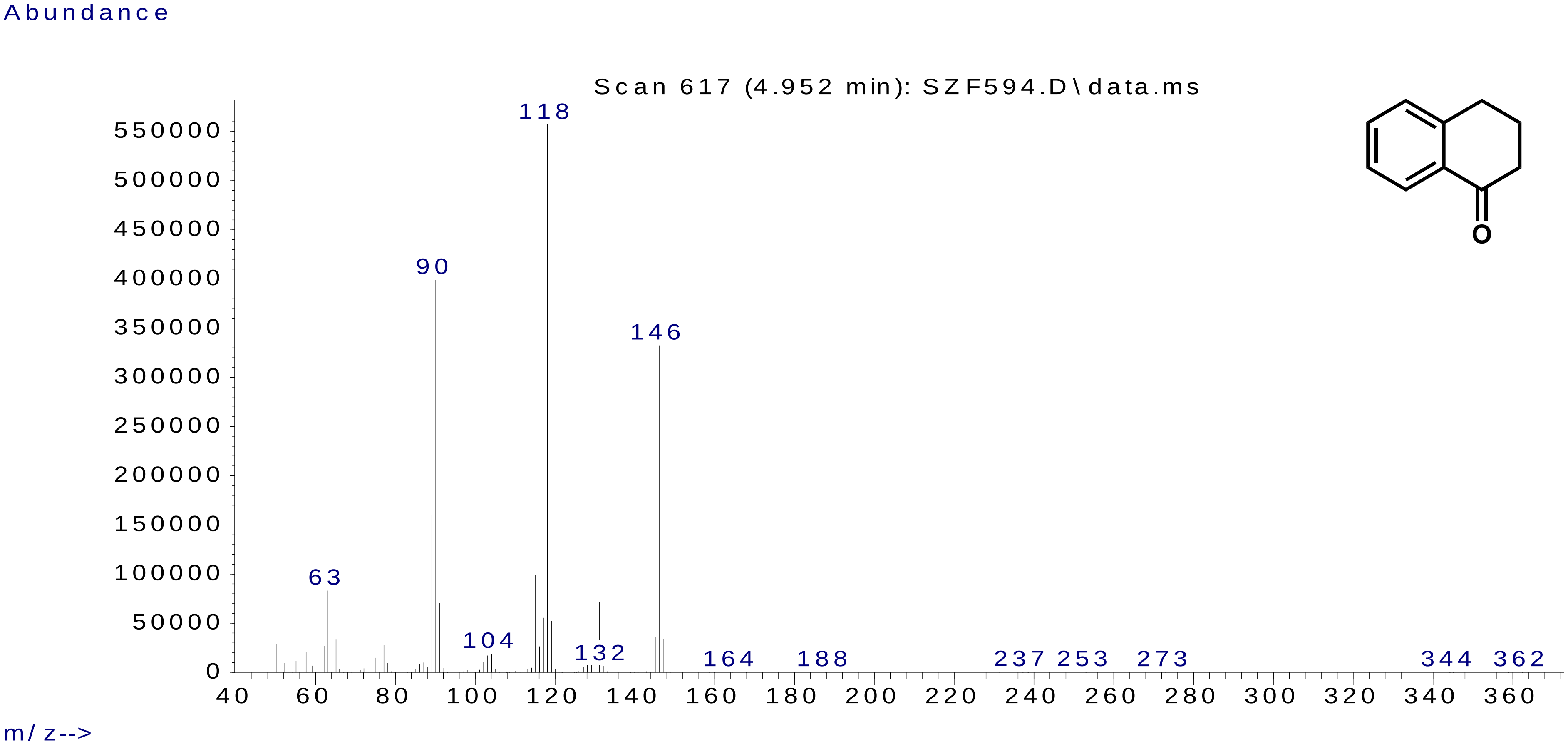
2-benzoylphenyl acetate (2r)[14]



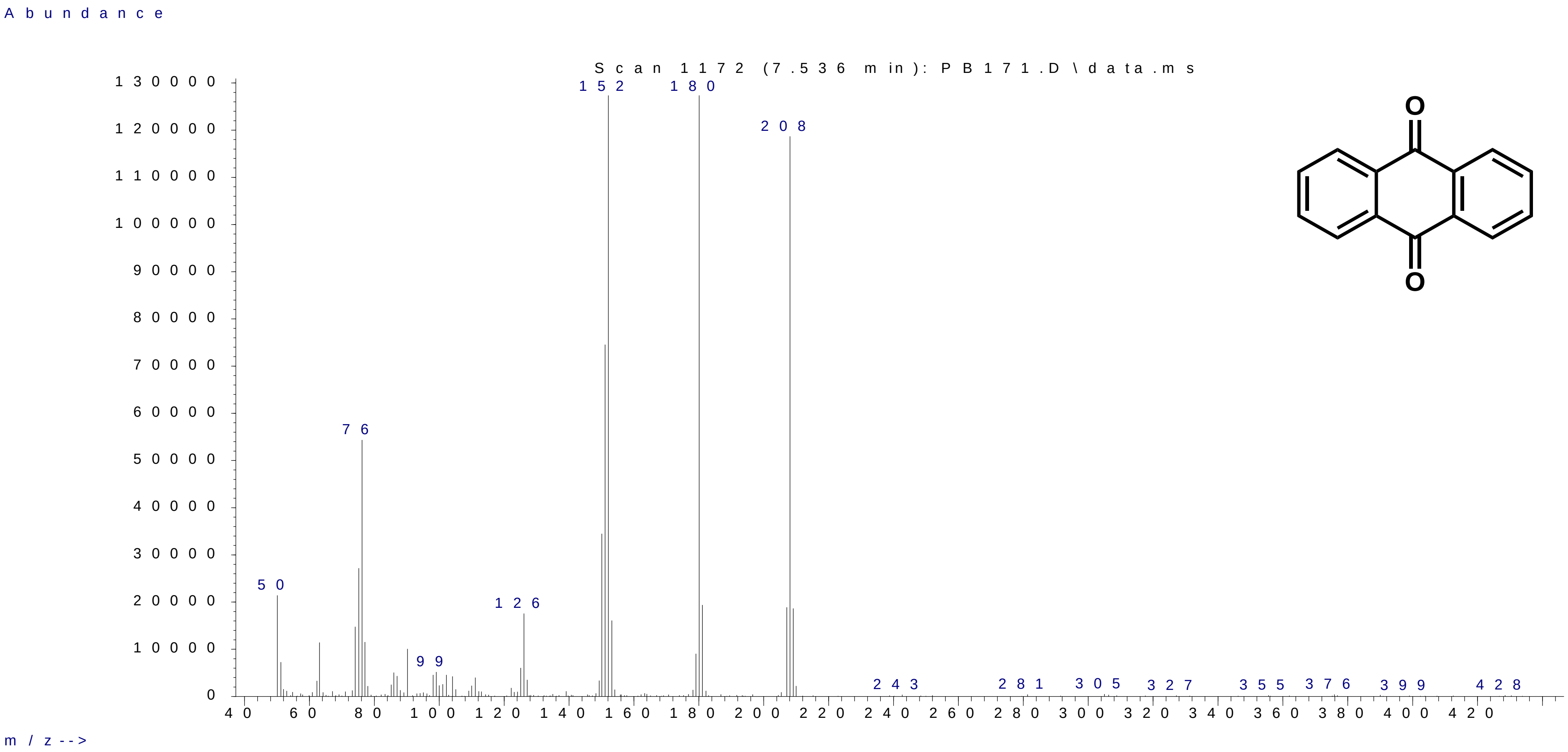
N-(2-benzoylphenyl)acetamide (2s)[15]



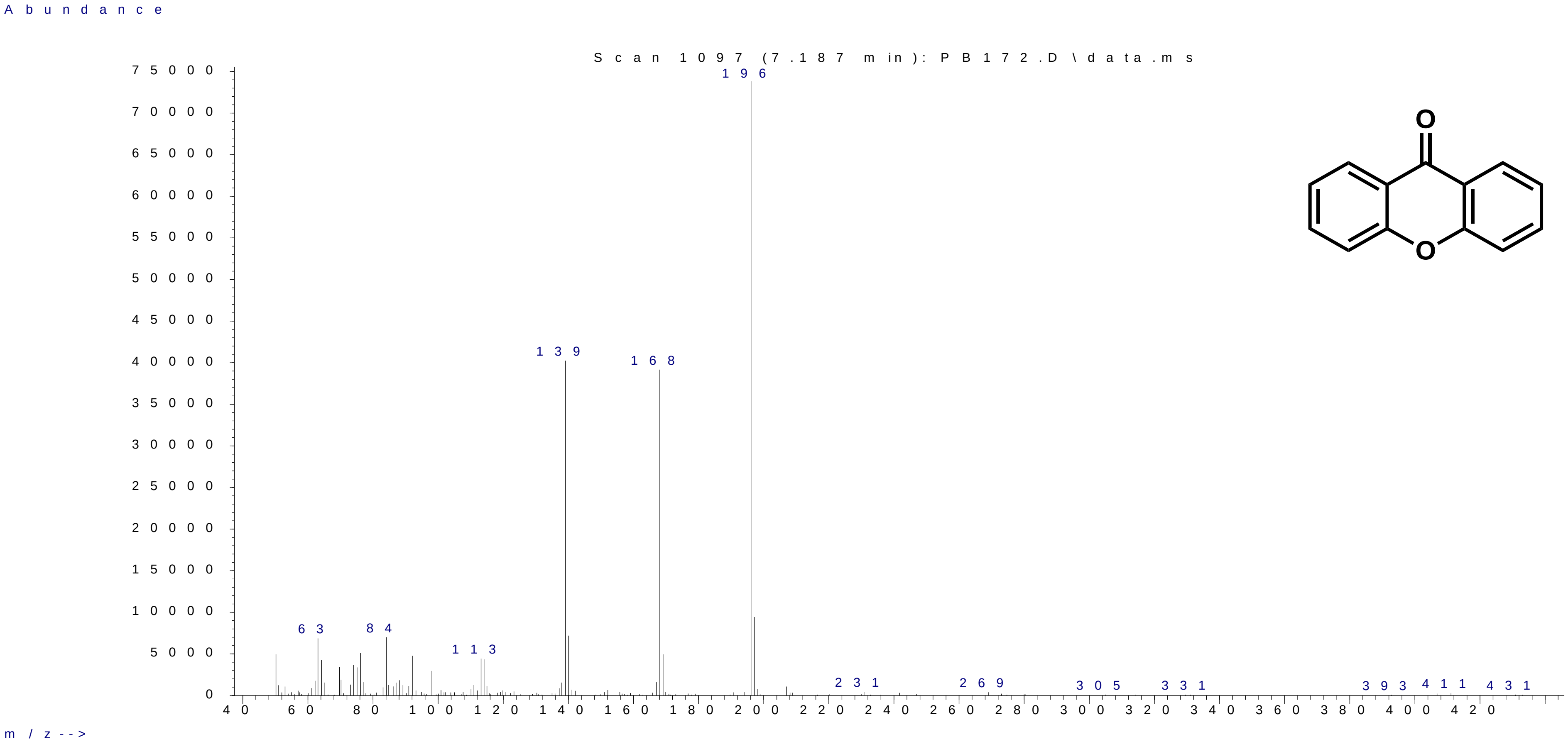
3,4-dihydronaphthalen-1(2*H*)-one (2t)[3]



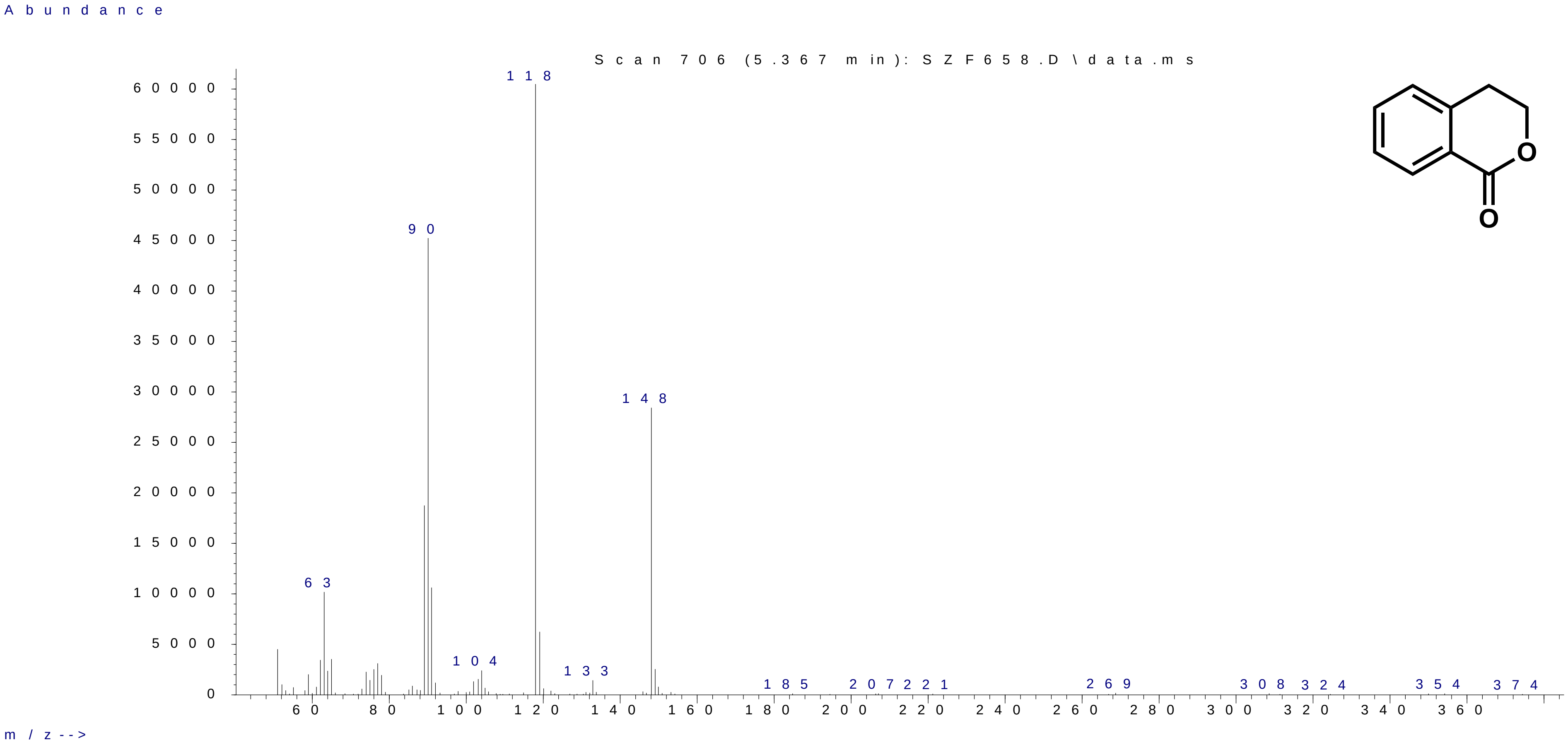
Anthracene-9,10-dione (2v)[16]



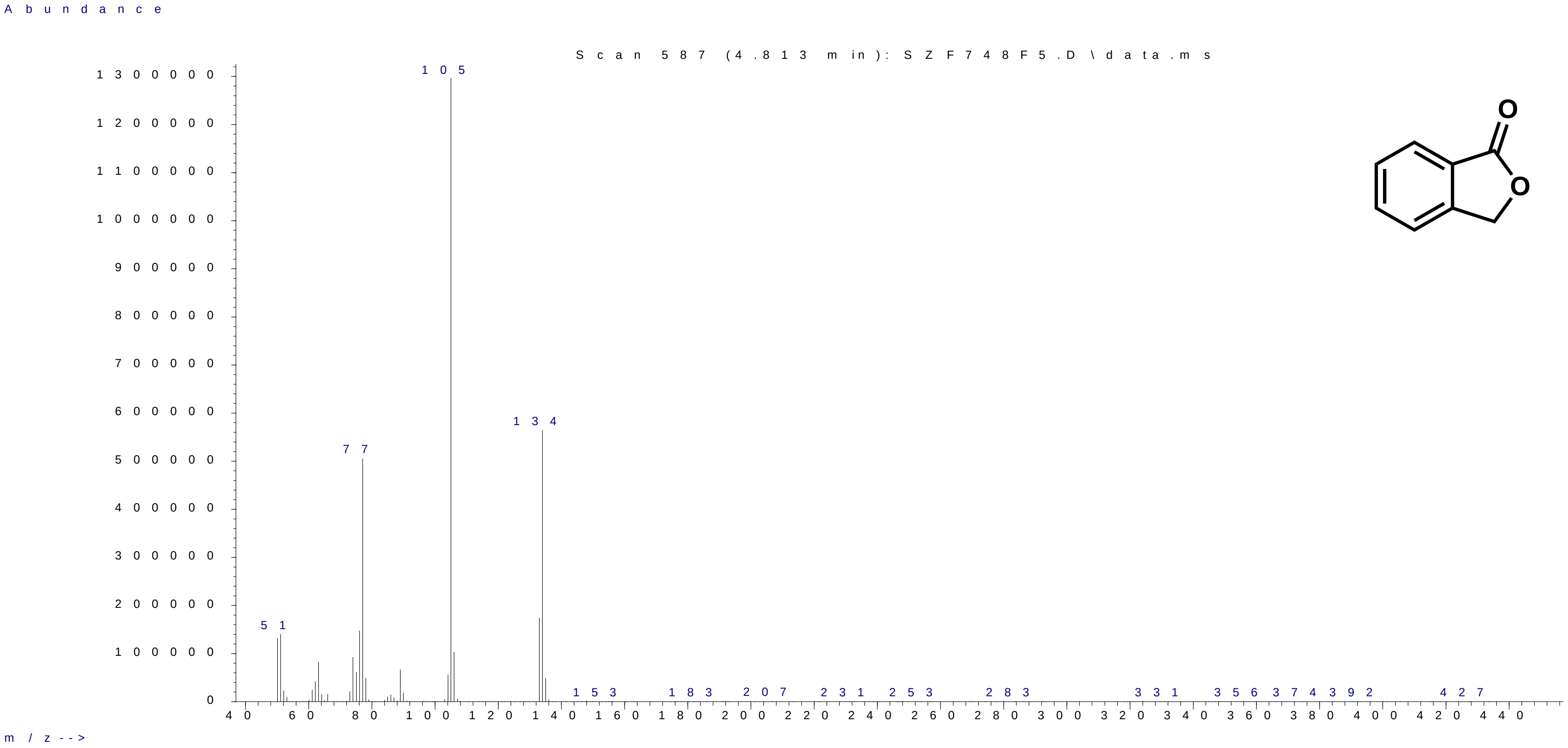
9H-xanthen-9-one (2w)[3]



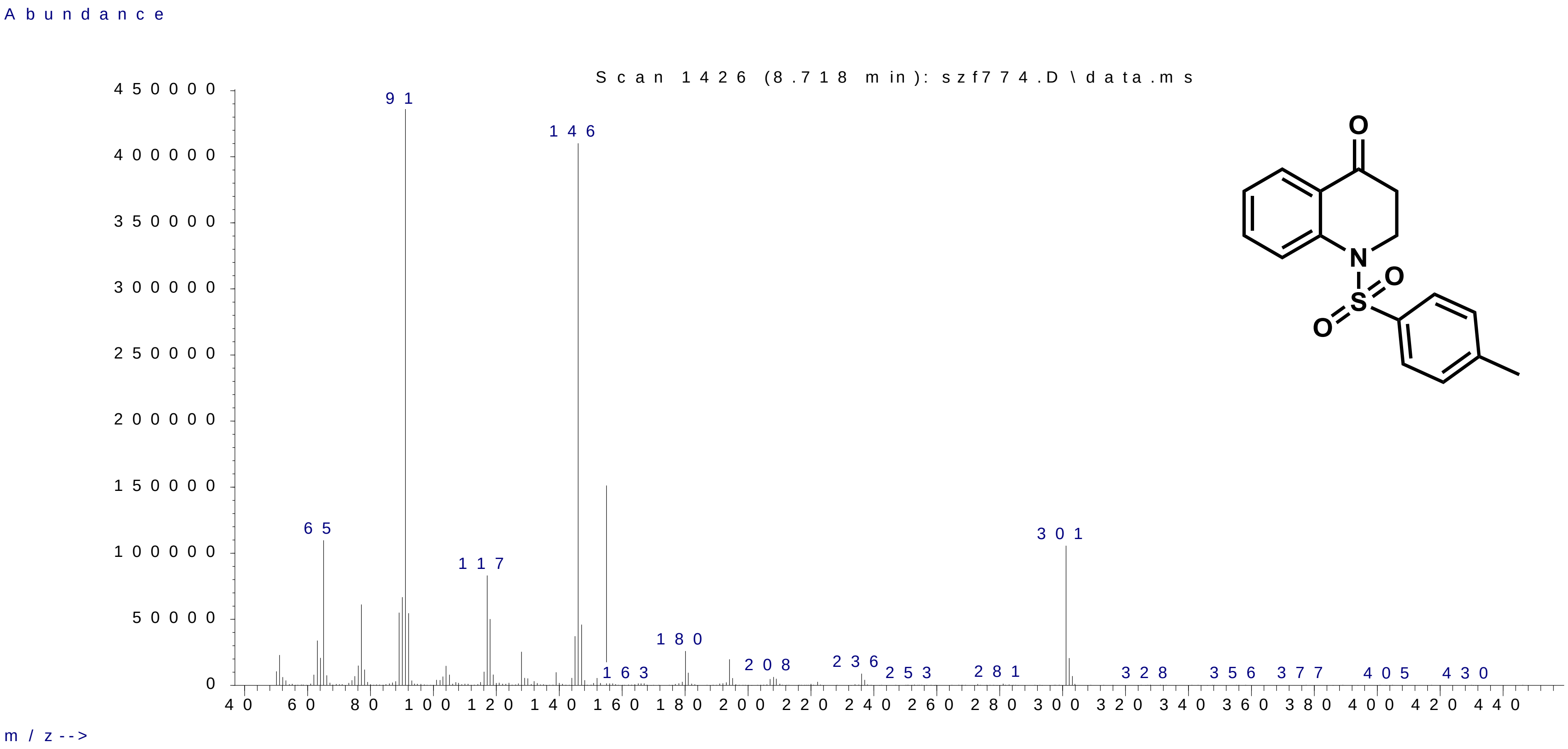
Isochroman-1-one (2x)[3]



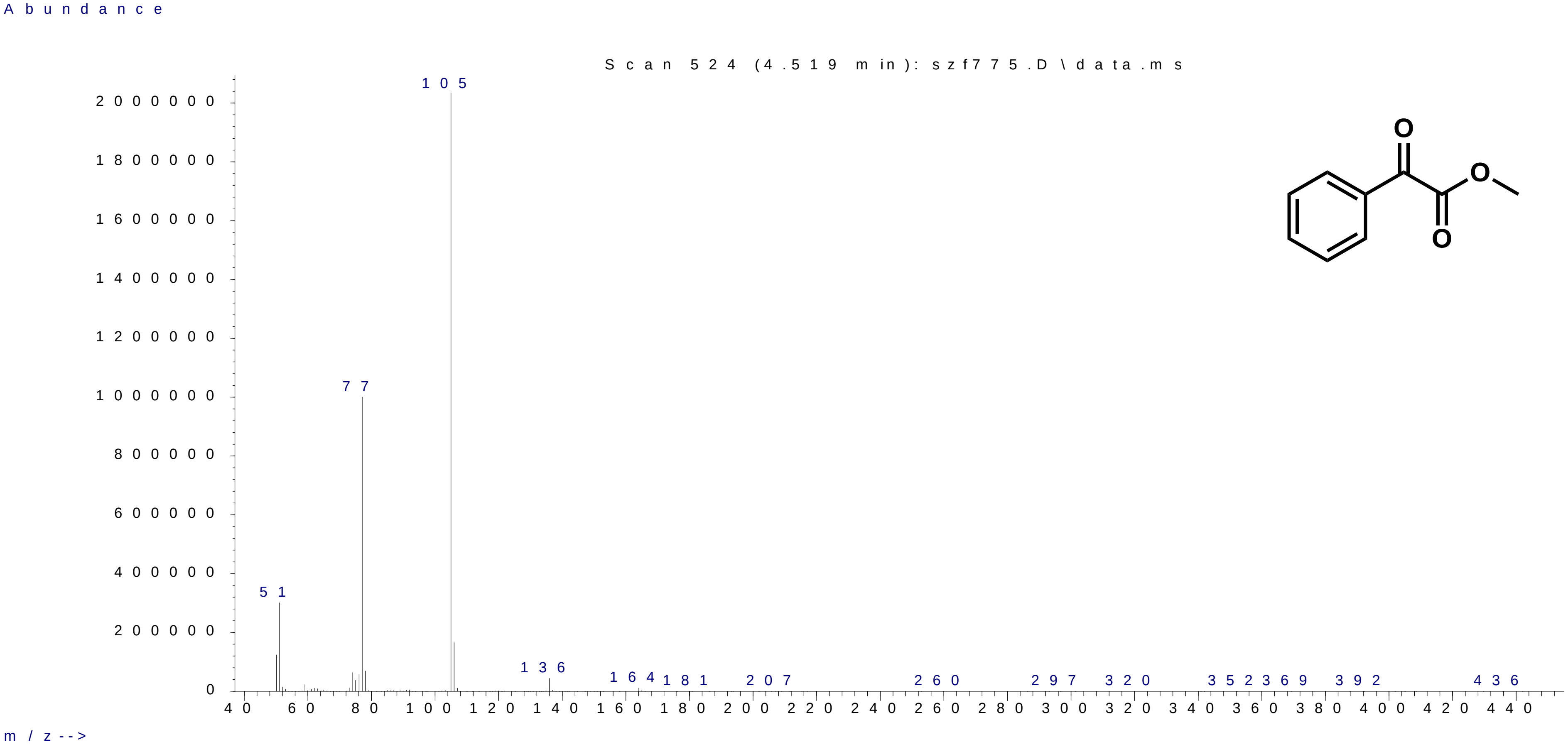
Isobenzofuran-1(3*H*)-one (2y)[3]



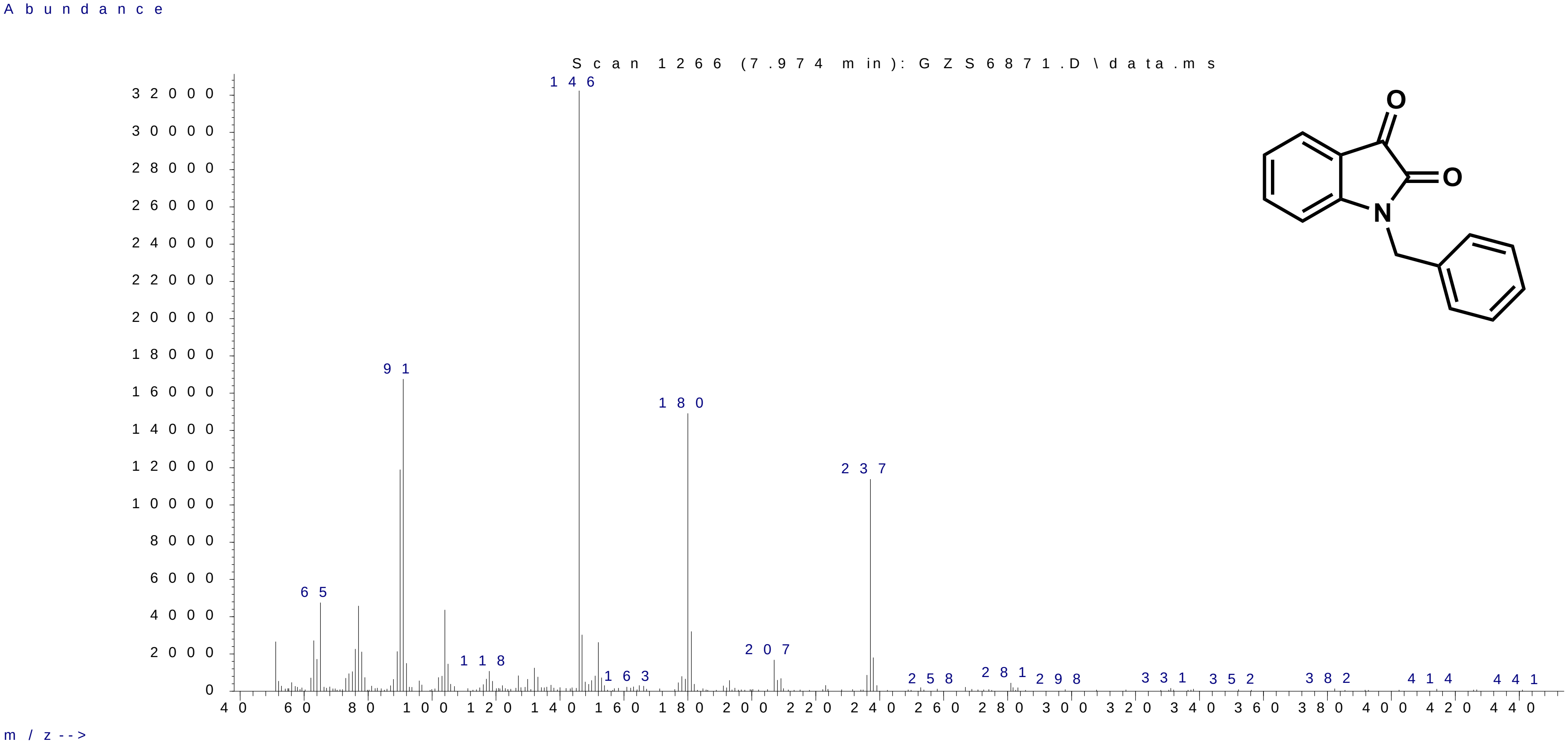
1-tosyl-2,3-dihydroquinolin-4(1*H*)-one (2x)[17]



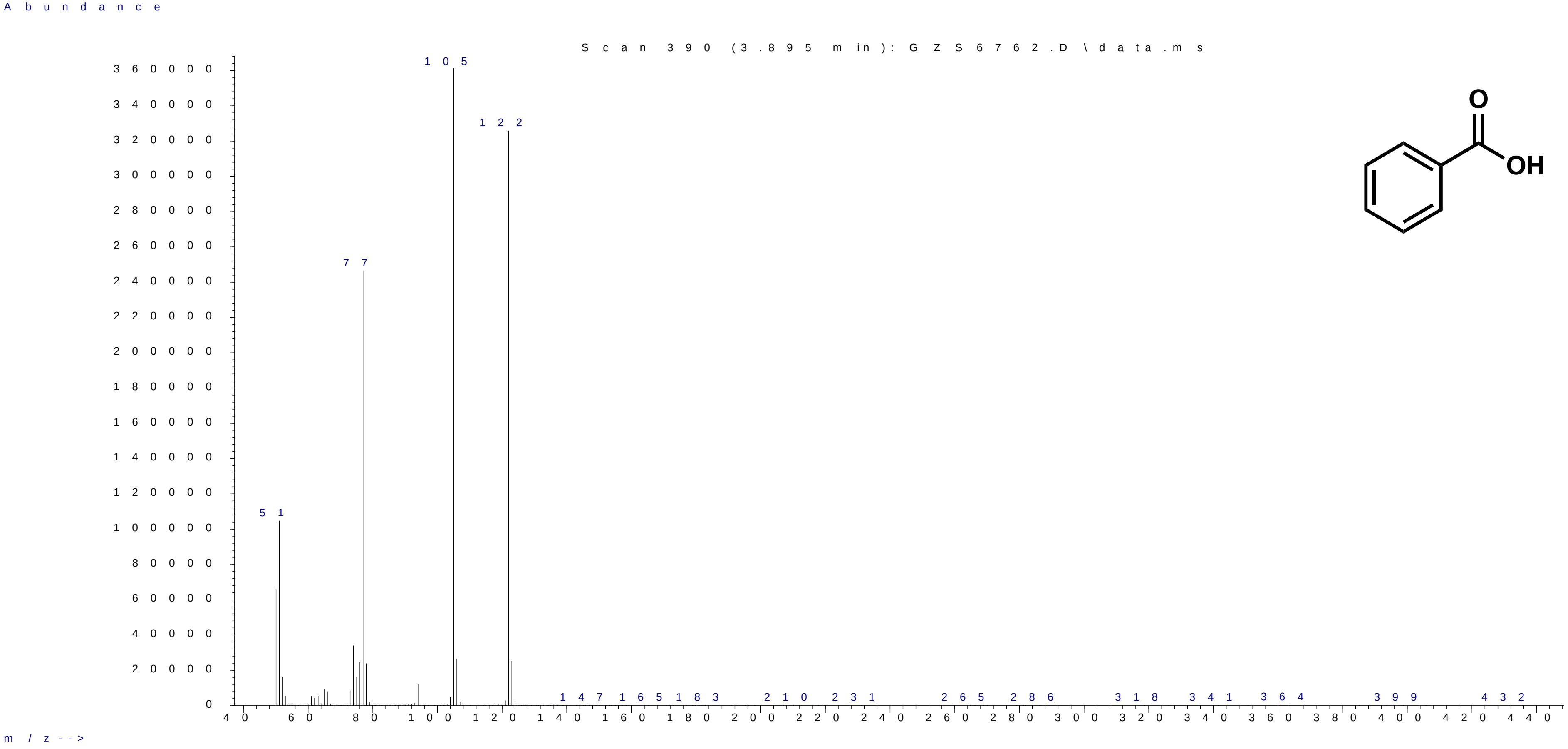
Methyl 2-oxo-2-phenylacetate (2aa)[18]



1-benzyl isatin (2bb)[19]



Benzoic acid (2cc)[20]



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