Supplementary Material

A novel approach for the synthesis of β-keto esters: one-pot reaction of carboxylic acids with chlorosulfonyl isocyanate

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EXPERIMENTAL

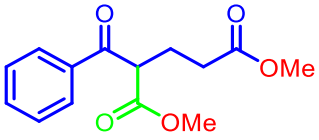
General remarks

Solvents are commercially available and used without further purification. 4- and 5-oxo-carboxylic acid derivatives were synthesized as in the literature. [18] ^{1}H and ^{13}C NMR spectra were recorded a Bruker 400 MHz in $CDCl_3$ with and NMR shifts are presented as δ in ppm. FTIR spectras were mesaured with a Perkin Elmer spectrophotometers in CH_2Cl_2 and by solutions in 0.1mm cells. High resolution mass spectra (HRMS) were obtained with a AB-Sciex 4600 QTOF MS spectrometer.

General procedure Synthesis of *β*-Keto ester:

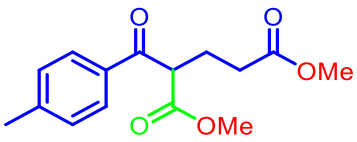
Carboxylic acid (**1a-n**) (1.0 eq) was dissolved in 10 mL DCM. The reaction mixture was added CSI (1.1 eq) and TFA (1.0 eq) and stirred for 2h at room temperature. Then, it was added 2 mL MeOH and stirred for 1h. The reaction mixture was extracted with dichloromethane. The organic extract was dried over sodium sulfate, filtrate and evaporation in vacuo. The resulting residue was purified by thin-layer chromatography (TLC) on silica gel.

Dimethyl 2-benzoylpentanedioate (2a):



Yellowish oil (316 mg, yield 92%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.30-2.35 (m, 2H), 2.46-2.50 (m, 2H), 3.69 (s, 3H), 3.71 (s, 3H), 4.55 (t, J=7.2 Hz, 1H), 7.50-7.53 (m, 2H), 7.60-7.64 (m, 1H), 8.04-8.06 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 24.0, 31.2, 51.7, 52.5, 52.6, 128.7, 128.8, 133.7, 135.9, 170.0, 173.2, 194.9; IR (CH_2Cl_2, cm^{-1}): 3003, 2955, 2849, 1737, 1689, 1596, 1436, 1333, 1272, 1161; HRMS (ESI) calcd. for $C_{14}H_{15}O_5$ $[M - H]^-$ 263.0925; found: 263.0916.

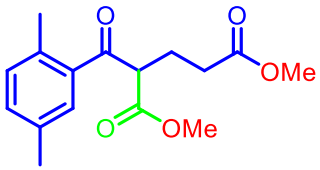
Dimethyl 2-(4-methylbenzoyl)pentanedioate (2b):



Yellowish oil (305 mg, yield 91%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.28-2.32 (m, 2H), 2.42-2.47 (m, 5H), 3.67 (s, 3H), 3.68 (s, 3H), 4.50 (t, J=7.2 Hz, 1H), 7.28 (d, J=8.0, 2H), 7.92 (d, J=8.0 Hz, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 21.7, 24.0, 51.7, 52.4, 52.5, 128.9, 129.5, 133.4, 144.7, 170.1, 173.2, 194.4; IR (CH_2Cl_2, cm^{-1}): 3010, 2942, 2855, 1748, 1690, 1592, 1440, 1328, 128, 1153; HRMS (ESI−)

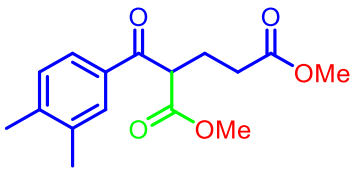
calcd. for $C_{14}H_{15}O_5$ [M − H]− 263,0925; found: 263,0916; HRMS (ESI) calcd. for $C_{15}H_{17}O_5$ [M − H]− 277.1081; found: 277.1088.

Dimethyl 2-(2,5-dimethylbenzoyl)pentanedioate (2c):



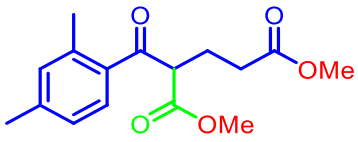
Yellowish oil (294 mg, yield 89%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.25-2.28 (m, 2H), 2.35-2.44 (m, 8H), 3.67 (s, 6H), 4.40 (t, J=7.1 Hz, 1H), 7.12-7.15 (m, 1H), 7.19-7.21 (m, 1H), 7.49-7.51 (m, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 20.6, 23.9, 31.3, 33.1, 51.7, 52.4, 54.9, 129.2, 132.0, 132.6, 135.3, 135.9, 136.7, 170.1, 173.1, 198.4; IR (CH_2Cl_2, cm^{-1}): 2953, 1739, 1652, 1437, 1302, 1203, 1118; HRMS (ESI) calcd. for $C_{16}H_{19}O_5$ [M − H]− 291.1238; found: 291.1240.

Dimethyl 2-(3,4-dimethylbenzoyl)pentanedioate (2d):



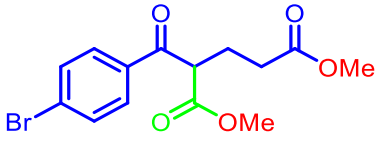
Yellowish oil (297 mg, yield 90%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.26-2.34 (m, 8H), 2.42-2.46 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.50 (t, J=7.2 Hz, 1H), 7.23 (d, J=7.8 Hz, 1H), 7.75 (d, J=7.8 Hz, 1H), 7.79 (s, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 19.8, 20.1, 24.1, 31.3, 51.7, 52.3, 52.5, 126.5, 129.8, 130.0, 133.8, 137.3, 143.5, 170.2, 173.2, 194.7; IR (CH_2Cl_2, cm^{-1}): 2956, 1740, 1686, 1611, 1439, 1286, 1169; HRMS (ESI) calcd. for $C_{16}H_{19}O_5$ [M − H]− 291.1238; found: 291.1224.

Dimethyl 2-(2,4-dimethylbenzoyl)pentanedioate (2e):



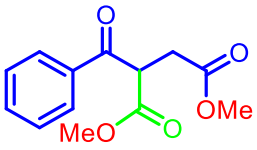
Yellowish oil (292 mg, yield 88%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.25-2.29 (m, 2H), 2.33-2.48 (m, 8H), 3.66 (s, 3H), 3.68 (s, 3H), 4.41 (t, J=7.2 Hz, 1H), 7.08 (s, 1H), 7.10 (s, 1H), 7.66 (d, J=7.8 Hz, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 21.4, 24.0, 51.7, 52.4, 54.6, 126.5, 129.3, 133.1, 133.7, 139.7, 142.7, 170.2, 142.7, 170.3, 173.2, 197.5; IR (CH_2Cl_2, cm^{-1}): 2953, 1742, 1663, 1442, 1328, 1243, 1136; HRMS (ESI) calcd. for $C_{16}H_{19}O_5$ [M − H]− 291.1238; found: 291.1215.

Dimethyl 2-(4-bromobenzoyl)pentanedioate (2f):



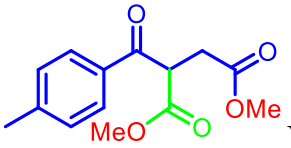
Yellowish oil (275 mg, yield 87%), $_1$H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.26-2.30 (m, 2H), 2.43-2.47 (m, 2H), 3.67 (s, 3H), 3.69 (s, 3H), 4.49 (t, J=7.2 Hz, 1H), 7.63 (d, J=8.5 Hz, 2H), 7.90 (d, J=8.5 Hz, 2H); $_{13}$C-NMR ($CDCl_3$, ppm, 100 MHz): δ 23.4, 31.1, 33.0, 51.7, 52.4, 129.1, 130.2, 132.2, 134.6, 169.8, 173.2, 193.9; IR (CH_2Cl_2, cm^{-1}): 2952, 1735, 1686, 1583, 1435, 1329, 1272, 1171; HRMS (ESI) calcd. for $C_{14}H_{14}BrO_5$ $[M - H]^-$ 341.0030; found: 341.0035.

Dimethyl 2-benzoylsuccinate (2g):



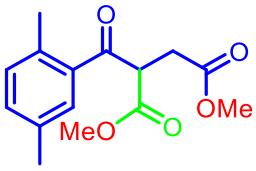
Yellowish oil (327 mg, yield 93%), $_1$H-NMR ($CDCl_3$, ppm, 400 MHz): δ 3.03-3.14 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.89 (t, J=7.2 Hz, 1H), 7.48 (d, J=7.8 Hz, 2H), 7.58-7.61 (m, 1H), 8.04 (d, J=7.3 Hz, 2H); $_{13}$C-NMR ($CDCl_3$, ppm, 100 MHz): δ 33.1, 49.3, 52.1, 52.9, 128.8, 128.9, 133.8, 135.8, 169.2, 171.7, 194.0; IR (CH_2Cl_2, cm^{-1}): 3003, 2955, 2849, 1737, 1689, 1596, 1436, 1333, 1272, 1161; HRMS (ESI) calcd. for $C_{13}H_{14}O_5$ $[M - H]^-$ 250.0847; found: 250.0832.

Dimethyl 2-(4-methylbenzoyl)succinate (2h):



Yellowish oil (309 mg, yield 90%), $_1$H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.42 (s, 3H), 3.05-3.08 (m, 2H), 3.67 (s, 3H), 3.69 (s, 3H), 4.87 (t, J=7.2 Hz, 1H), 7.29 (d, J=8.2 Hz, 2H), 7.93 (d, J=8.2 Hz, 2H); $_{13}$C-NMR ($CDCl_3$, ppm, 100 MHz): δ 28.9, 33.1, 49.2, 52.1, 52.8, 129.1, 129.5, 133.3, 144.8, 169.3, 171.8, 193.5; IR (CH_2Cl_2, cm^{-1}): 2953, 2826, 1737, 682, 1607, 1436, 1255, 1165; HRMS (ESI) calcd. for $C_{14}H_{15}O_5$ $[M - H]^-$ 263.0925; found: 263.0904.

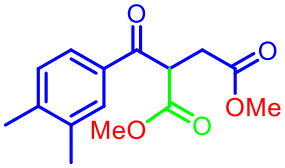
Dimethyl 2-(2,5-dimethylbenzoyl)succinate (2i):



Yellowish oil (293 mg, yield 87%), $_1$H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.37 (s, 3H), 2.41 (s, 3H), 3.01-3.11 (m, 2H), 3.67 (s, 3H), 3.69 (s, 3H), 4.76 (t, J=7.2 Hz, 1H), 7.23-7.25 (m, 1H), 7.76-7.80 (m, 2H); $_{13}$C-NMR ($CDCl_3$, ppm, 100 MHz): δ 20.4, 21.0, 32.9, 51.9, 52.1, 52.7, 129.3, 131.9, 132.6, 135.3, 135.9,

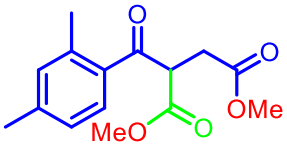
136.7, 169.3, 171.8, 197.5; IR (CH_2Cl_2, cm^{-1}): 2951, 1734, 1680, 1432, 1272, 1170; HRMS (ESI) calcd. for $C_{15}H_{17}O_5$ $[M - H]^-$ 277.1081; found: 277.1090.

Dimethyl 2-(3,4-dimethylbenzoyl)succinate (2j):



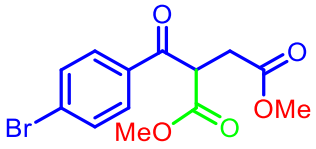
Yellowish oil (290 mg, yield 86%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.32 (s, 6H), 2.98-3.11 (m, 2H), 3.67 (s, 3H), 3.69 (s, 3H), 4.87 (t, J=7.2 Hz, 1H), 7.11-7.22 (m, 2H), 7.58 (s, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 19.8, 20.1, 33.1, 49.1, 52.1, 52.8, 126.7, 129.9, 130.0, 133.6, 137.2, 143.6, 169.4, 171.8, 193.8; IR (CH_2Cl_2, cm^{-1}): 2954, 1738, 1684, 1605, 1438, 1276, 1174; HRMS (ESI) calcd. for $C_{15}H_{17}O_5$ $[M - H]^-$ 277.1081; found: 277.1054.

Dimethyl 2-(2,4-dimethylbenzoyl)succinate (2k):



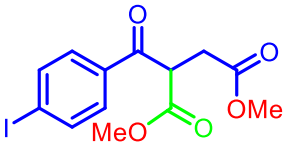
Yellowish oil (291 mg, yield 86%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.35 (s, 3H), 2.46 (s, 3H), 2.94-3.11 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.77 (t, J=6.6 Hz, 1H), 7.08-7.11 (m, 2H), 7.74 (d, J=7.8, Hz, 1H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 21.3, 21.4, 33.1, 51.6, 52.1, 52.7, 126.4, 129.4, 133.0, 133.7, 139.6, 142.7, 169.5, 171.8, 196.6; IR (CH_2Cl_2, cm^{-1}): 2955, 2893, 1741, 1683, 1439, 1203, 1173; HRMS (ESI) calcd. for $C_{15}H_{17}O_5$ $[M - H]^-$ 277.1081; found: 277.1074.

Dimethyl 2-(4-bromobenzoyl)succinate (2l):



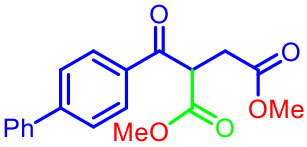
Yellowish oil (279 mg, yield 87%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 3.01-3.17 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.82 (dd, J=6.2, 8.1 Hz, 1H), 7.63-7.65 (m, 2H), 7.89-7.92 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 33.0, 49.1, 52.2, 53.0, 129.1, 130.4, 132.1, 134.6, 168.8, 171.7, 193.1; IR (CH_2Cl_2, cm^{-1}): 2954, 1737, 1688, 1585, 1437, 1331, 1274, 1173; HRMS (ESI) calcd. for $C_{13}H_{12}BrO_5$ $[M - H]^-$ 326.9874; found: 326.9863.

Dimethyl 2-(4-iodobenzoyl)succinate (2m):



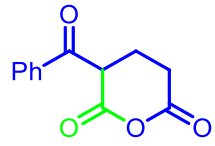
Yellowish oil (260 mg, yield 84%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.99-3.16 (m, 2H), 3.67 (s, 3H), 3.68 (s, 3H), 4.81 (dd, J=6.2 , 8.2 Hz, 1H), 7.73-7.76 (m, 2H), 7.85-7.88 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 33.0, 49.1, 52.2, 53.0, 102.1, 130.2, 135.2, 138.1, 168.8, 171.7, 193.5; IR (CH_2Cl_2, cm^{-1}): 2952, 2833, 1737, 1687, 1581, 1437, 1272, 1071; HRMS (ESI) calcd. for $C_{13}H_{12}IO_5$ $[M - H]^-$ 374.9735; found: 374.9732.

Dimethyl 2-([1,1'-biphenyl]-4-carbonyl)succinate (2n):



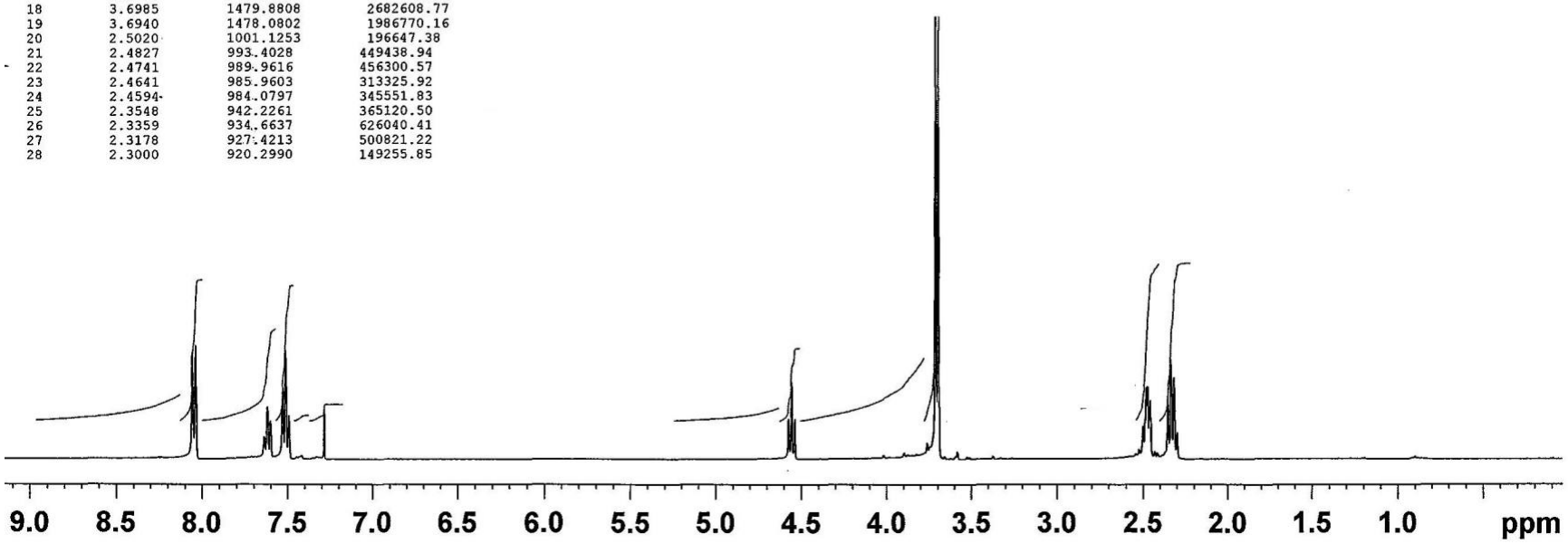
Yellow solid (289 mg, yield 90%), ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 3.03-3.18 (m, 2H), 3.68 (s, 3H), 3.70 (s, 3H), 4.93 (t, J=7.2 Hz, 1H), 7.41-7.50 (m, 3H), 7.62-7.73 (m, 4H), 8.11-8.14 (m, 2H); ^{13}C-NMR ($CDCl_3$, ppm, 100 MHz): δ 33.1, 49.2, 52.2, 52.9, 127.3, 127.4, 128.4, 129.0, 129.6, 134.5, 139.7, 146.5, 169.2, 171.8, 193.6; IR (CH_2Cl_2, cm^{-1}): 2953, 1736, 682, 603, 1436, 1274, 1172; HRMS (ESI) calcd. for $C_{19}H_{17}O_5$ $[M - H]^-$ 325.1081; found: 325.1071.

3-Benzoyldihydro-2H-pyran-2,6(3H)-dione (3):

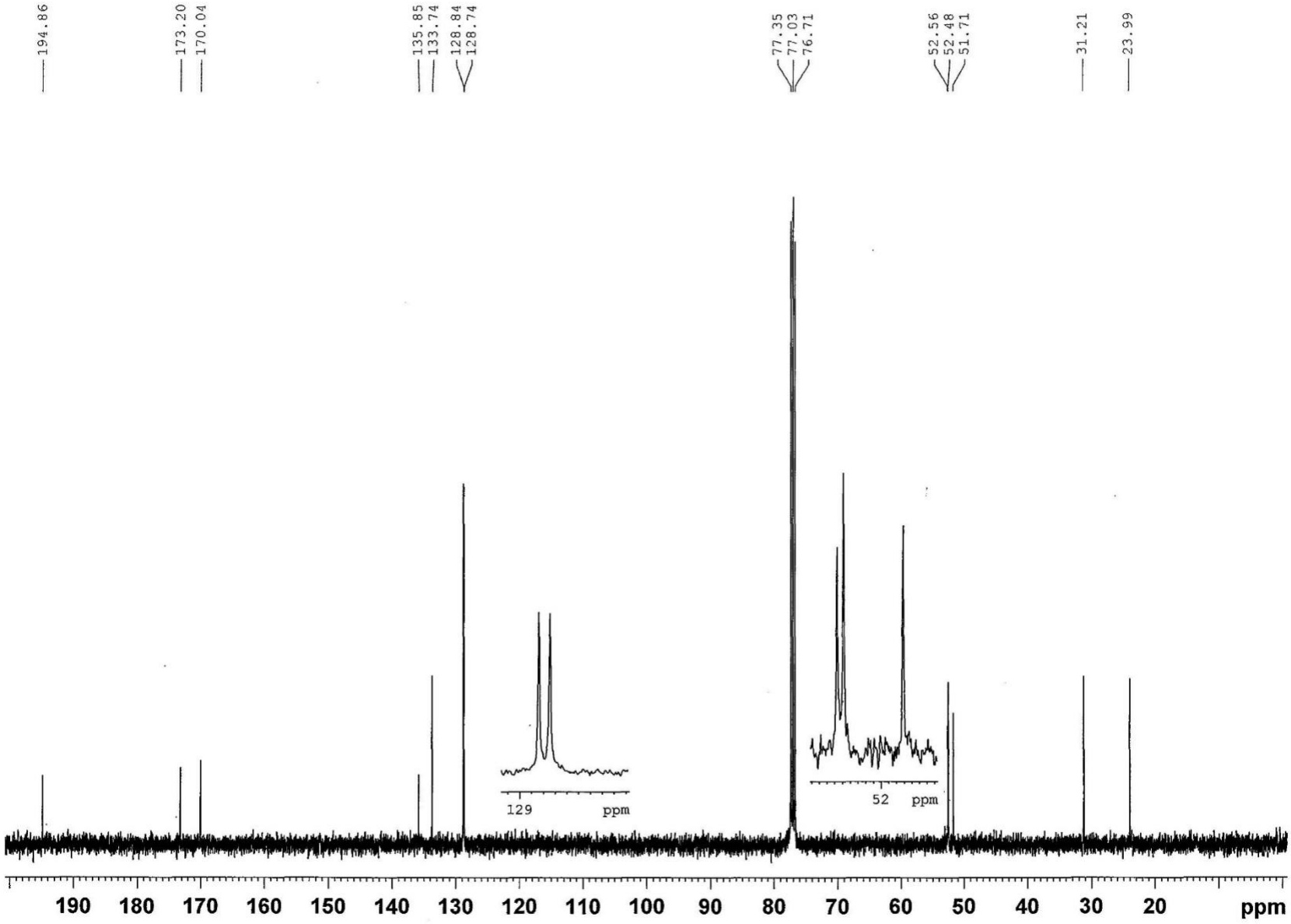


Reaction mixture ^{1}H-NMR ($CDCl_3$, ppm, 400 MHz): δ 2.31-2.47 (m, 2H), 2.93-2.96 (m, 2H), 4.92 (t, *J* 5.3 Hz, 1H), 7.53-7.59 (m, 2H), 7.67-7.73 (m, 1H), 7.96-7.98 (m, 2H).

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	8.0587	3224.5276	665962.95
2	8.0411	3217.4853	577844.88
3	8.0384	3216.4050	712379.53
4	7.6397	3056.8732	137397.72
5	7.6232	3050.2710	324710.65
6	7.6197	3048.8706	301384.48
7	7.6045	3042.7886	236725.80
8	7.6027	3042.0684	225971.75
9	7.5336	3014.4194	412125.99
10	7.5308	3013.2990	443725.77
11	7.5140	3006.5768	678244.06
12	7.4953	2999.0944	274292.61
13	4.5756	1830.8348	246672.24
14	4.5575	1823.5925	490203.91
15	4.5400	1816.5902	253756.27
16	3.7145	1486.2829	2806828.75
17	3.7100	1484.4823	2005019.02
18	3.6985	1479.8808	2682608.77
19	3.6940	1478.0802	1986770.16
20	2.5020	1001.1253	196647.38
21	2.4827	993.4028	449438.94
22	2.4741	989.9616	456300.57
23	2.4641	985.9603	313325.92
24	2.4594	984.0797	345551.83
25	2.3548	942.2261	365120.50
26	2.3359	934.6637	626040.41
27	2.3178	927.4213	500821.22
28	2.3000	920.2990	149255.85

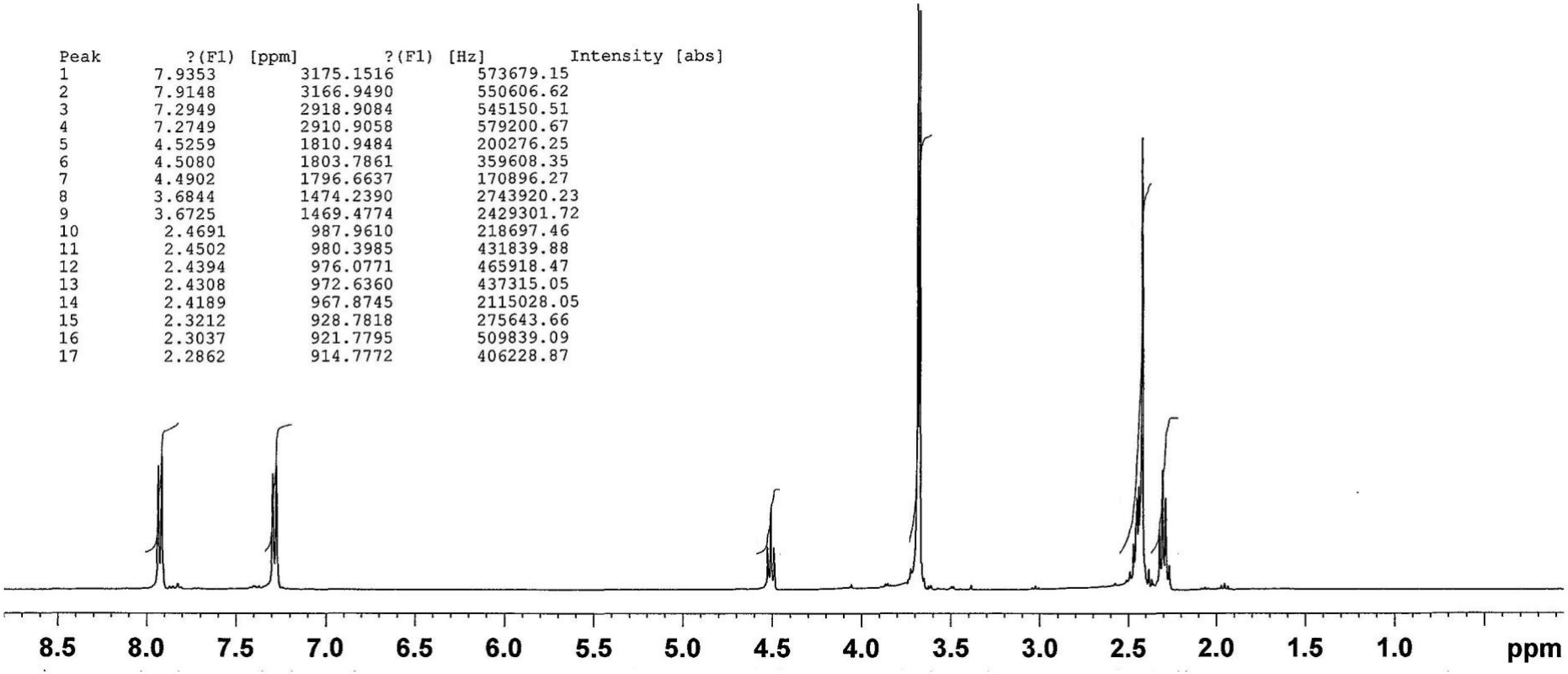


^{1}H NMR Spectrum of Dimethyl 2-benzoylpentanedioate (**2a**)



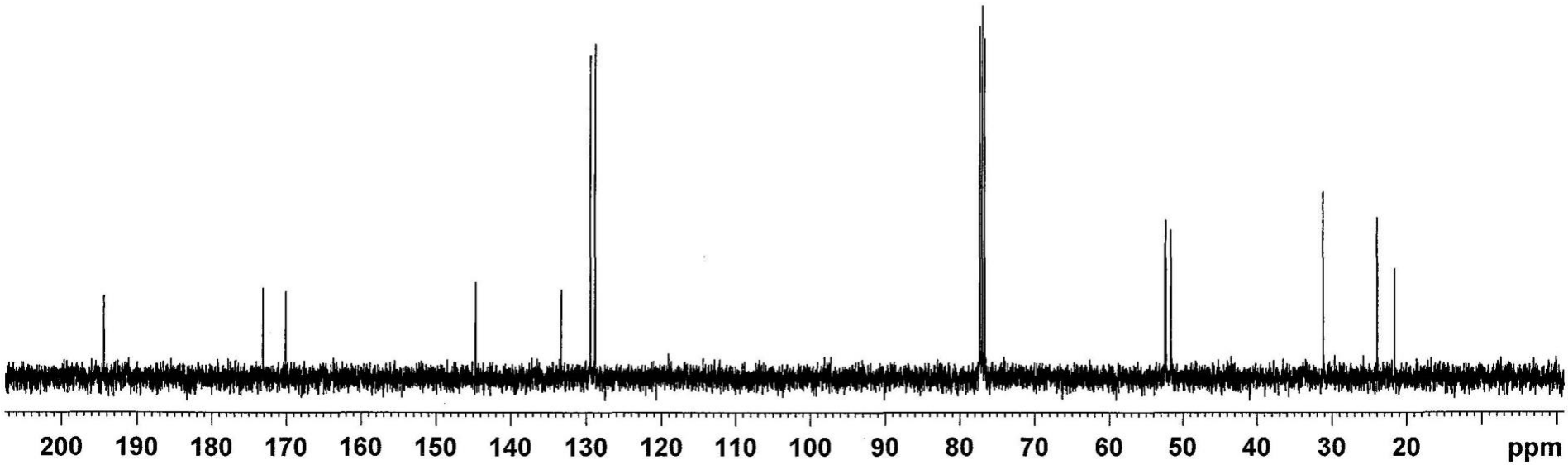
^{13}C NMR Spectrum of Dimethyl 2-benzoylpentanedioate (**2a**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.9353	3175.1516	573679.15
2	7.9148	3166.9490	550606.62
3	7.2949	2918.9084	545150.51
4	7.2749	2910.9058	579200.67
5	4.5259	1810.9484	200276.25
6	4.5080	1803.7861	359608.35
7	4.4902	1796.6637	170896.27
8	3.6844	1474.2390	2743920.23
9	3.6725	1469.4774	2429301.72
10	2.4691	987.9610	218697.46
11	2.4502	980.3985	431839.88
12	2.4394	976.0771	465918.47
13	2.4308	972.6360	437315.05
14	2.4189	967.8745	2115028.05
15	2.3212	928.7818	275643.66
16	2.3037	921.7795	509839.09
17	2.2862	914.7772	406228.87



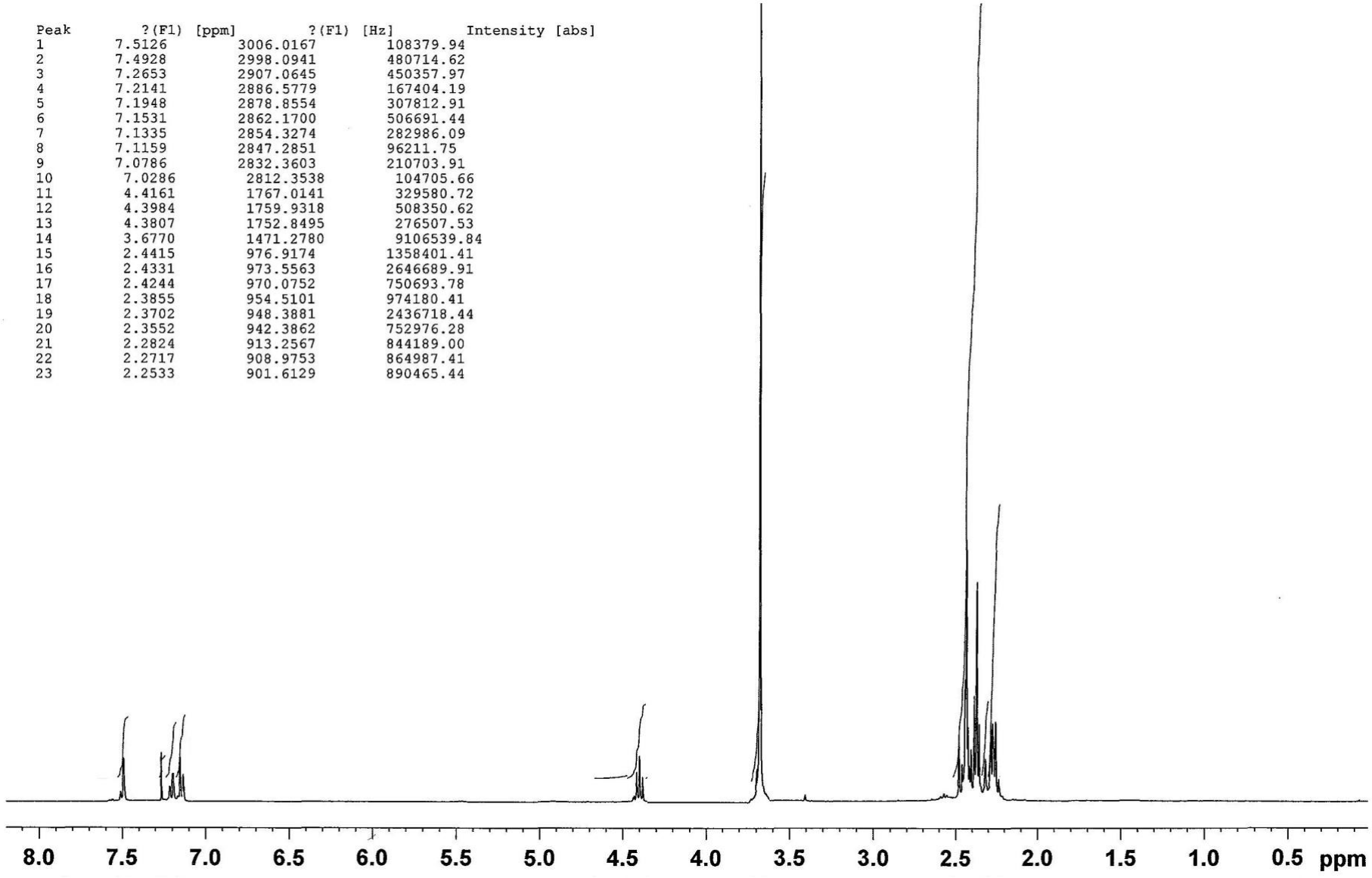
^{1}H NMR Spectrum of Dimethyl 2-(4-methylbenzoyl)pentanedioate (**2b**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	194.4283	19561.9696	41897.45
2	173.2201	17428.1539	38688.65
3	170.1542	17119.6852	44105.22
4	144.7428	14562.9739	48685.72
5	133.3738	13419.1073	40344.44
6	129.5231	13031.6777	127667.45
7	128.8781	12966.7825	169053.86
8	77.3718	7784.5910	177832.85
9	77.0538	7752.5962	150143.19
10	76.7358	7720.6013	169271.29
11	52.5050	5282.6734	53650.02
12	52.3612	5268.2053	78983.27
13	51.6790	5199.5673	75352.91
14	31.2274	3141.8752	94577.07
15	24.0178	2416.4974	61066.51
16	21.6853	2181.8181	47257.47



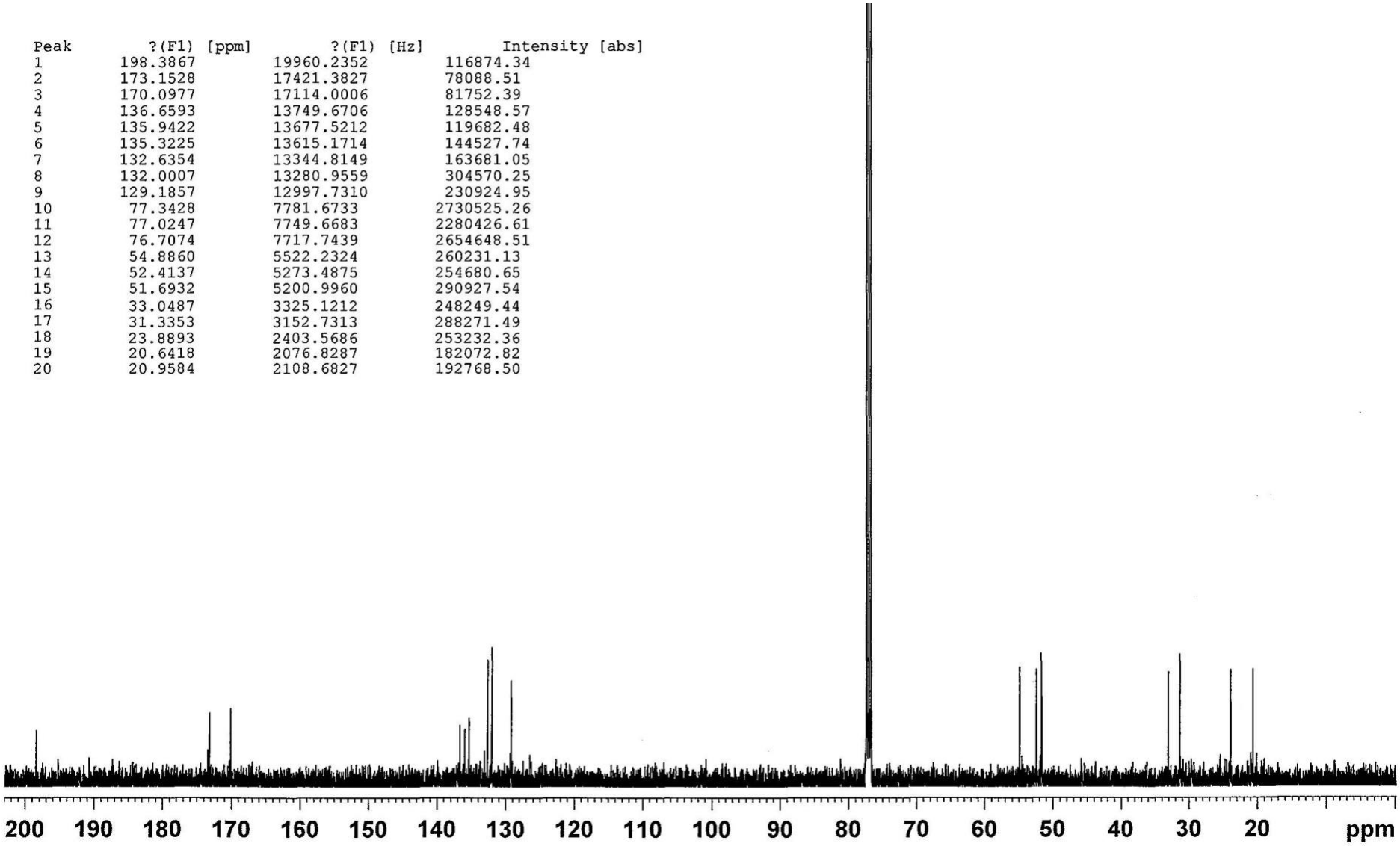
^{13}C NMR Spectrum of Dimethyl 2-(4-methylbenzoyl)pentanedioate (**2b**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.5126	3006.0167	108379.94
2	7.4928	2998.0941	480714.62
3	7.2653	2907.0645	450357.97
4	7.2141	2886.5779	167404.19
5	7.1948	2878.8554	307812.91
6	7.1531	2862.1700	506691.44
7	7.1335	2854.3274	282986.09
8	7.1159	2847.2851	96211.75
9	7.0786	2832.3603	210703.91
10	7.0286	2812.3538	104705.66
11	4.4161	1767.0141	329580.72
12	4.3984	1759.9318	508350.62
13	4.3807	1752.8495	276507.53
14	3.6770	1471.2780	9106539.84
15	2.4415	976.9174	1358401.41
16	2.4331	973.5563	2646689.91
17	2.4244	970.0752	750693.78
18	2.3855	954.5101	974180.41
19	2.3702	948.3881	2436718.44
20	2.3552	942.3862	752976.28
21	2.2824	913.2567	844189.00
22	2.2717	908.9753	864987.41
23	2.2533	901.6129	890465.44



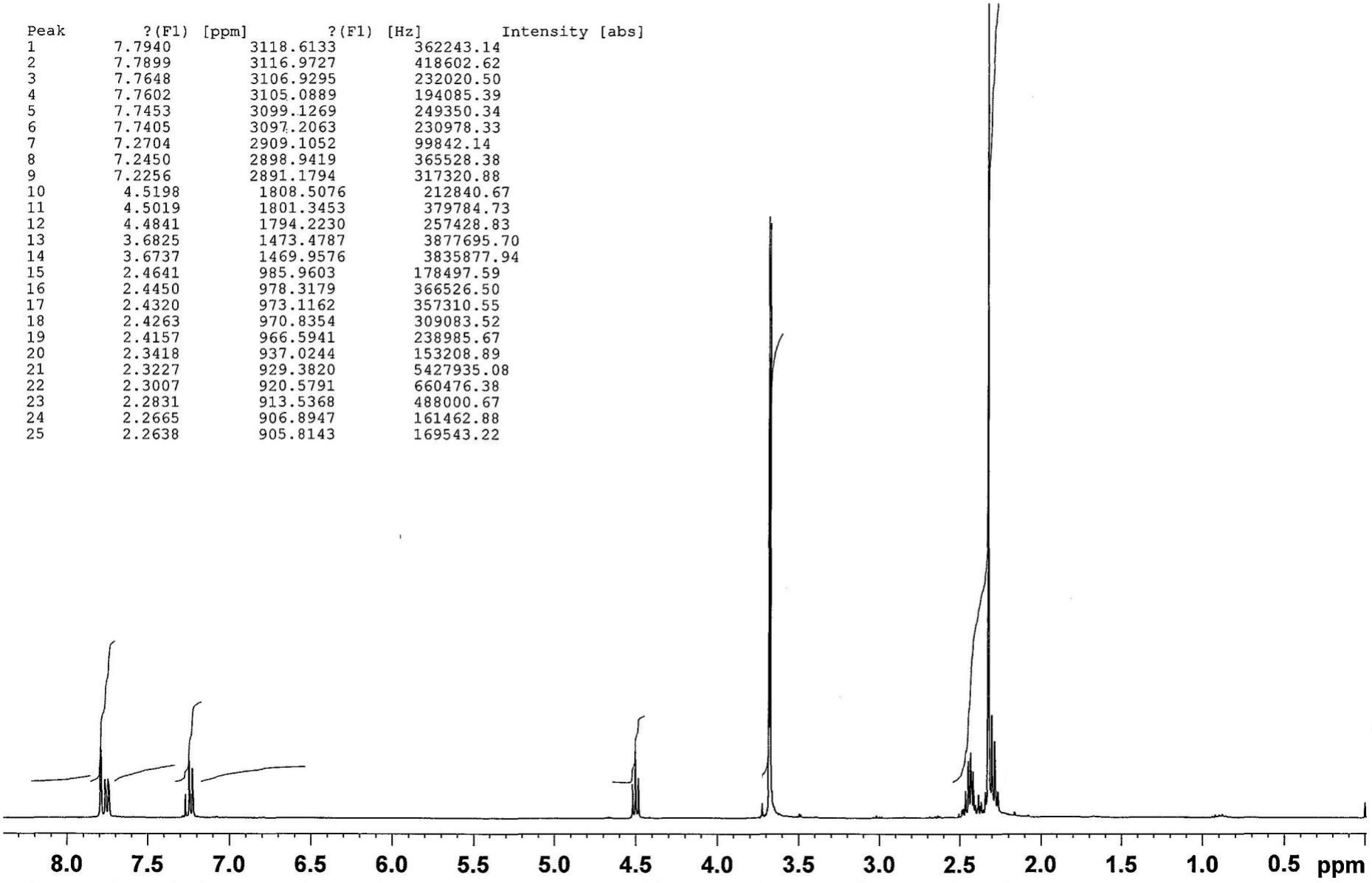
^{1}H NMR Spectrum of Dimethyl 2-(2,5-dimethylbenzoyl)pentanedioate (**2c**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	198.3867	19960.2352	116874.34
2	173.1528	17421.3827	78088.51
3	170.0977	17114.0006	81752.39
4	136.6593	13749.6706	128548.57
5	135.9422	13677.5212	119682.48
6	135.3225	13615.1714	144527.74
7	132.6354	13344.8149	163681.05
8	132.0007	13280.9559	304570.25
9	129.1857	12997.7310	230924.95
10	77.3428	7781.6733	2730525.26
11	77.0247	7749.6683	2280426.61
12	76.7074	7717.7439	2654648.51
13	54.8860	5522.2324	260231.13
14	52.4137	5273.4875	254680.65
15	51.6932	5200.9960	290927.54
16	33.0487	3325.1212	248249.44
17	31.3353	3152.7313	288271.49
18	23.8893	2403.5686	253232.36
19	20.6418	2076.8287	182072.82
20	20.9584	2108.6827	192768.50



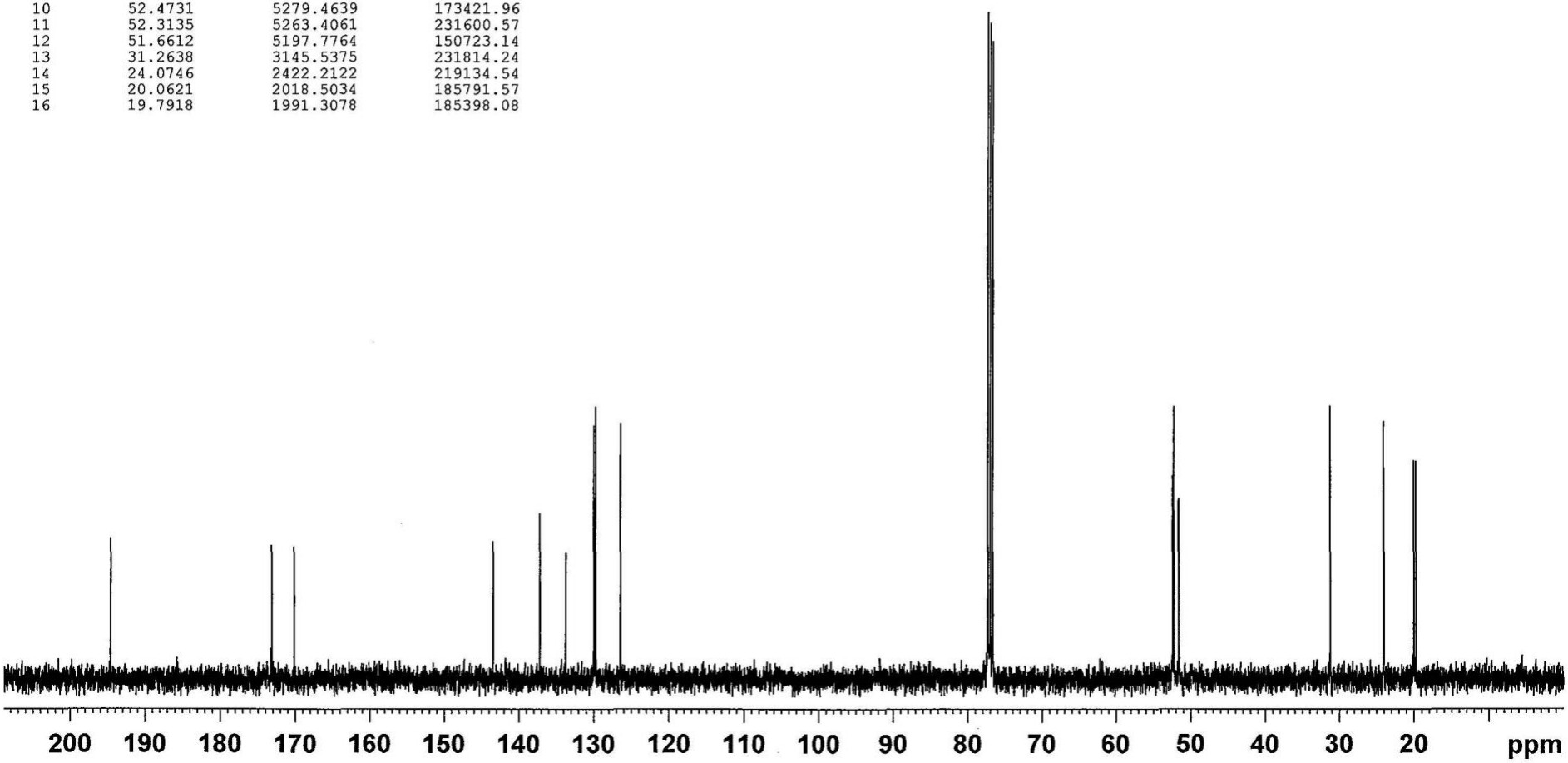
^{13}C NMR Spectrum of Dimethyl 2-(2,5-dimethylbenzoyl)pentanedioate (**2c**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.7940	3118.6133	362243.14
2	7.7899	3116.9727	418602.62
3	7.7648	3106.9295	232020.50
4	7.7602	3105.0889	194085.39
5	7.7453	3099.1269	249350.34
6	7.7405	3097.2063	230978.33
7	7.2704	2909.1052	99842.14
8	7.2450	2898.9419	365528.38
9	7.2256	2891.1794	317320.88
10	4.5198	1808.5076	212840.67
11	4.5019	1801.3453	379784.73
12	4.4841	1794.2230	257428.83
13	3.6825	1473.4787	3877695.70
14	3.6737	1469.9576	3835877.94
15	2.4641	985.9603	178497.59
16	2.4450	978.3179	366526.50
17	2.4320	973.1162	357310.55
18	2.4263	970.8354	309083.52
19	2.4157	966.5941	238985.67
20	2.3418	937.0244	153208.89
21	2.3227	929.3820	5427935.08
22	2.3007	920.5791	660476.38
23	2.2831	913.5368	488000.67
24	2.2665	906.8947	161462.88
25	2.2638	905.8143	169543.22



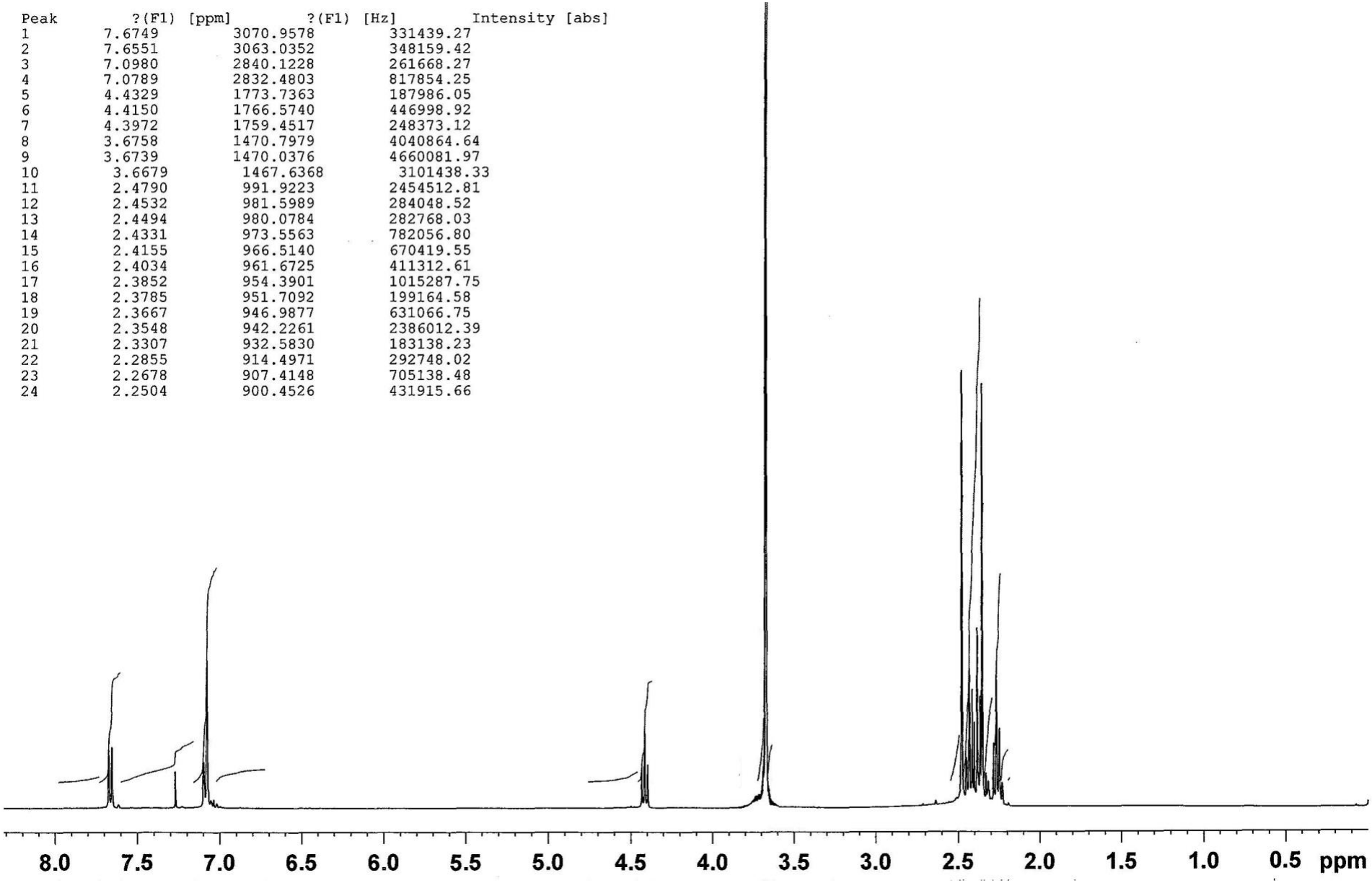
^{1}H NMR Spectrum of Dimethyl 2-(3,4-dimethylbenzoyl)pentanedioate (**2d**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	194.6689	19586.1771	119992.18
2	173.2137	17427.5100	106297.26
3	170.2257	17126.8790	112709.33
4	143.5082	14438.7574	100022.10
5	137.2648	13810.5916	141042.08
6	133.7890	13460.8818	93052.21
7	130.0259	13082.2658	162827.45
8	129.8112	13060.6643	231315.76
9	126.4970	12727.2134	165757.46
10	52.4731	5279.4639	173421.96
11	52.3135	5263.4061	231600.57
12	51.6612	5197.7764	150723.14
13	31.2638	3145.5375	231814.24
14	24.0746	2422.2122	219134.54
15	20.0621	2018.5034	185791.57
16	19.7918	1991.3078	185398.08



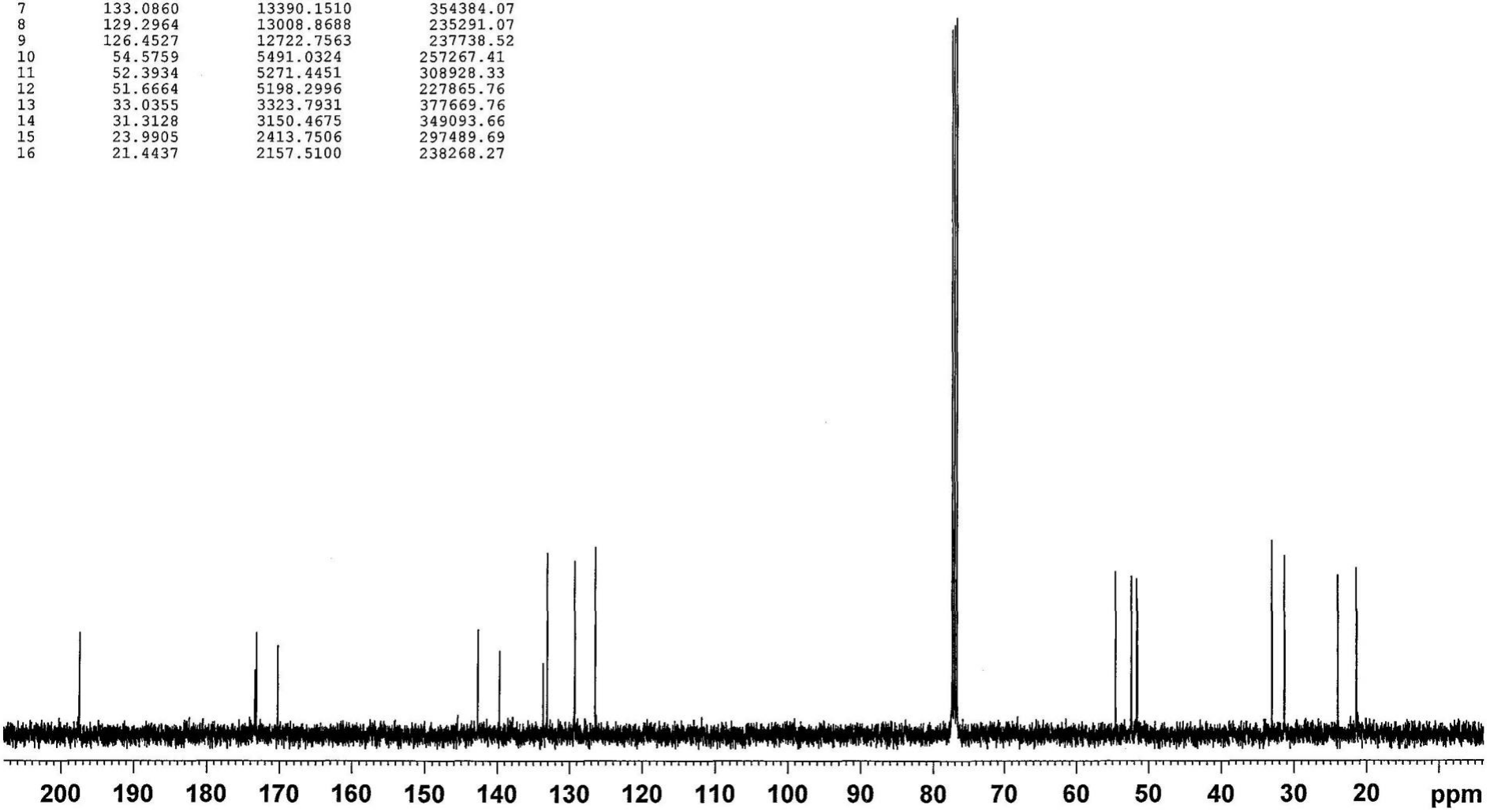
^{13}C NMR Spectrum of Dimethyl 2-(3,4-dimethylbenzoyl)pentanedioate (**2d**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.6749	3070.9578	331439.27
2	7.6551	3063.0352	348159.42
3	7.0980	2840.1228	261668.27
4	7.0789	2832.4803	817854.25
5	4.4329	1773.7363	187986.05
6	4.4150	1766.5740	446998.92
7	4.3972	1759.4517	248373.12
8	3.6758	1470.7979	4040864.64
9	3.6739	1470.0376	4660081.97
10	3.6679	1467.6368	3101438.33
11	2.4790	991.9223	2454512.81
12	2.4532	981.5989	284048.52
13	2.4494	980.0784	282768.03
14	2.4331	973.5563	782056.80
15	2.4155	966.5140	670419.55
16	2.4034	961.6725	411312.61
17	2.3852	954.3901	1015287.75
18	2.3785	951.7092	199164.58
19	2.3667	946.9877	631066.75
20	2.3548	942.2261	2386012.39
21	2.3307	932.5830	183138.23
22	2.2855	914.4971	292748.02
23	2.2678	907.4148	705138.48
24	2.2504	900.4526	431915.66



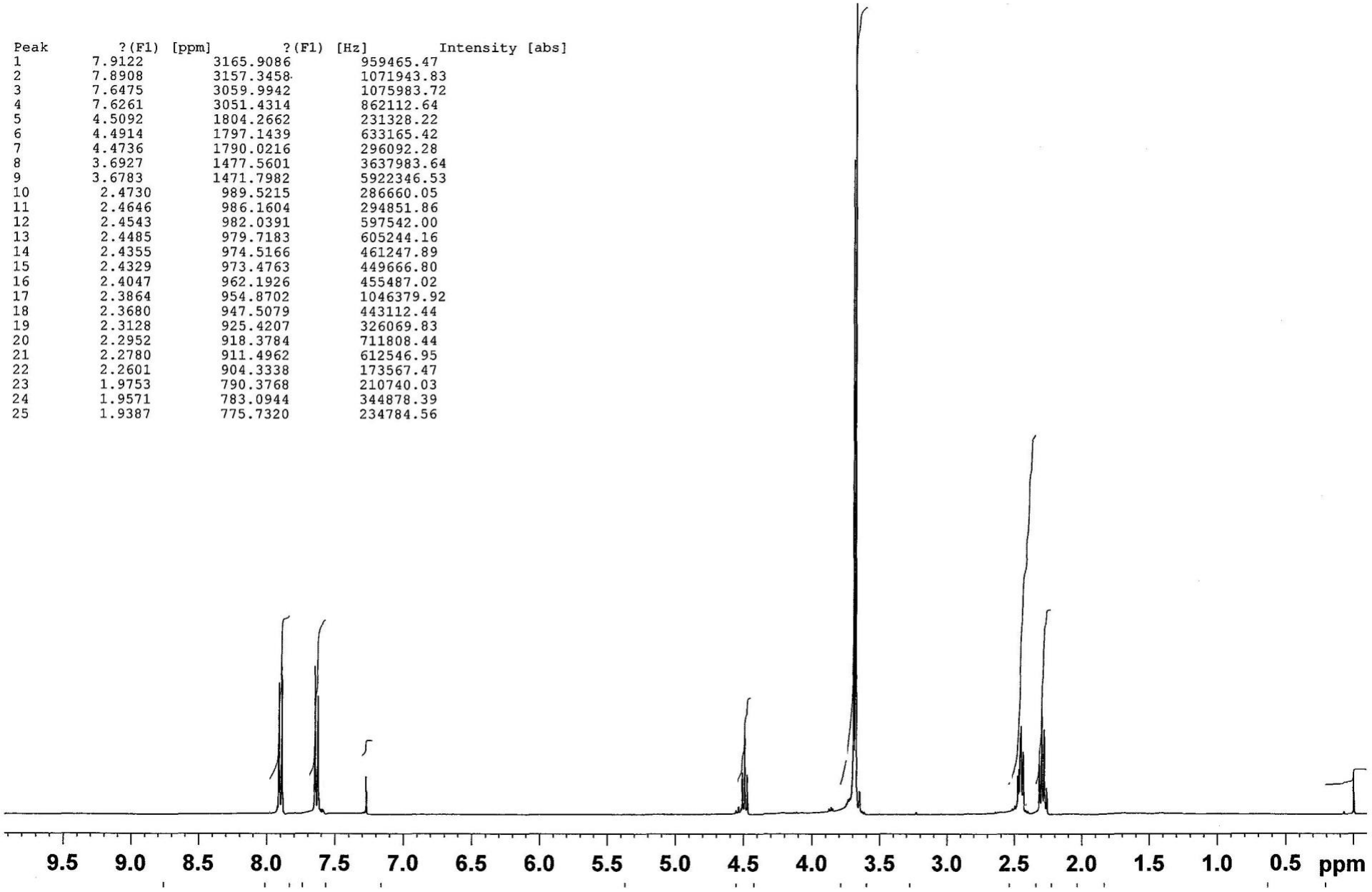
^{1}H NMR Spectrum of Dimethyl 2-(2,4-dimethylbenzoyl)pentanedioate (**2e**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	197.4617	19867.1684	199485.46
2	173.1758	17423.6968	144166.46
3	170.2251	17126.8187	173604.89
4	142.7127	14358.7199	148685.79
5	139.7238	14057.9984	128240.71
6	133.6682	13448.7277	139776.25
7	133.0860	13390.1510	354384.07
8	129.2964	13008.8688	235291.07
9	126.4527	12722.7563	237738.52
10	54.5759	5491.0324	257267.41
11	52.3934	5271.4451	308928.33
12	51.6664	5198.2996	227865.76
13	33.0355	3323.7931	377669.76
14	31.3128	3150.4675	349093.66
15	23.9905	2413.7506	297489.69
16	21.4437	2157.5100	238268.27

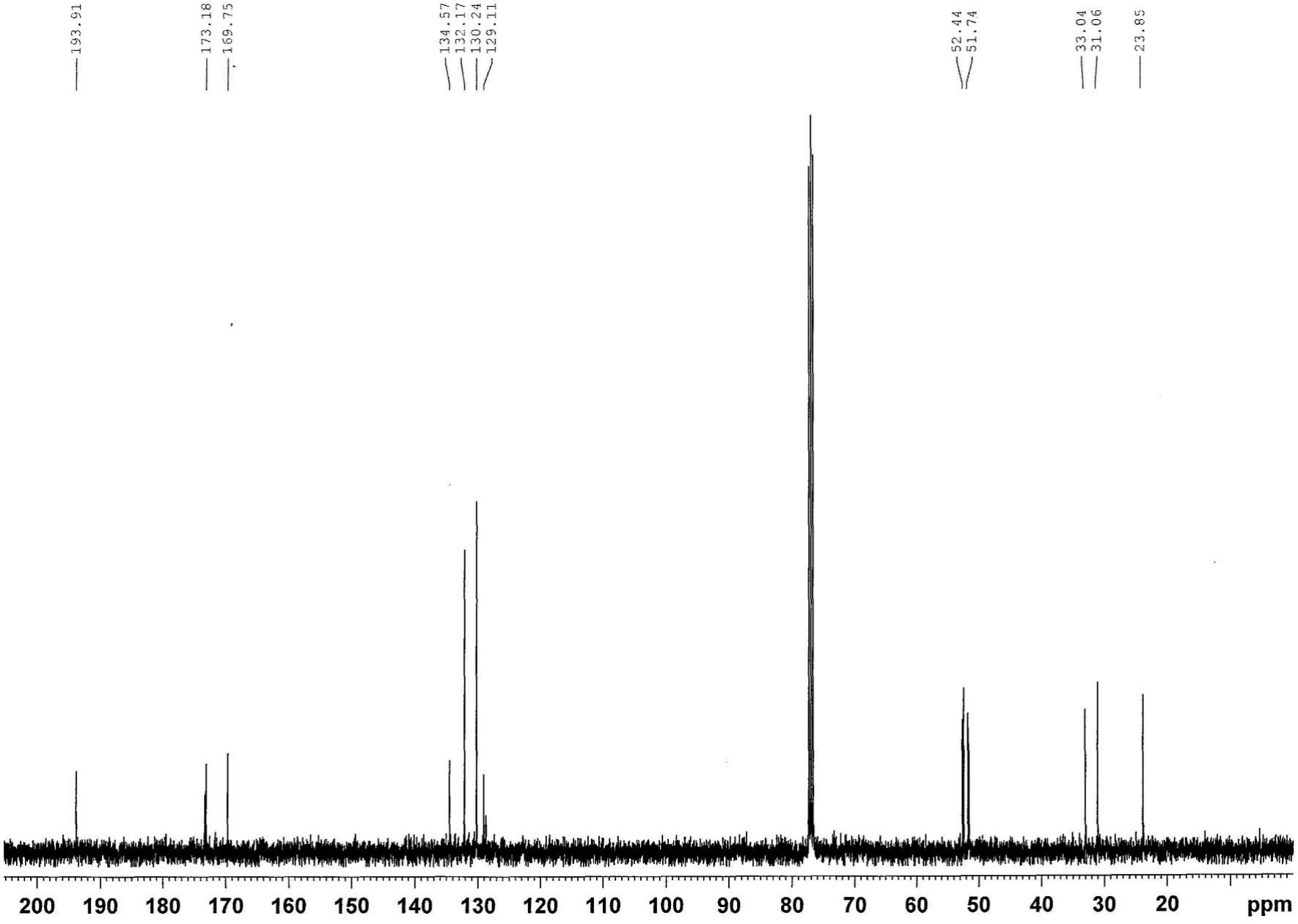


^{13}C NMR Spectrum of Dimethyl 2-(2,4-dimethylbenzoyl)pentanedioate (**2e**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.9122	3165.9086	959465.47
2	7.8908	3157.3458	1071943.83
3	7.6475	3059.9942	1075983.72
4	7.6261	3051.4314	862112.64
5	4.5092	1804.2662	231328.22
6	4.4914	1797.1439	633165.42
7	4.4736	1790.0216	296092.28
8	3.6927	1477.5601	3637983.64
9	3.6783	1471.7982	5922346.53
10	2.4730	989.5215	286660.05
11	2.4646	986.1604	294851.86
12	2.4543	982.0391	597542.00
13	2.4485	979.7183	605244.16
14	2.4355	974.5166	461247.89
15	2.4329	973.4763	449666.80
16	2.4047	962.1926	455487.02
17	2.3864	954.8702	1046379.92
18	2.3680	947.5079	443112.44
19	2.3128	925.4207	326069.83
20	2.2952	918.3784	711808.44
21	2.2780	911.4962	612546.95
22	2.2601	904.3338	173567.47
23	1.9753	790.3768	210740.03
24	1.9571	783.0944	344878.39
25	1.9387	775.7320	234784.56

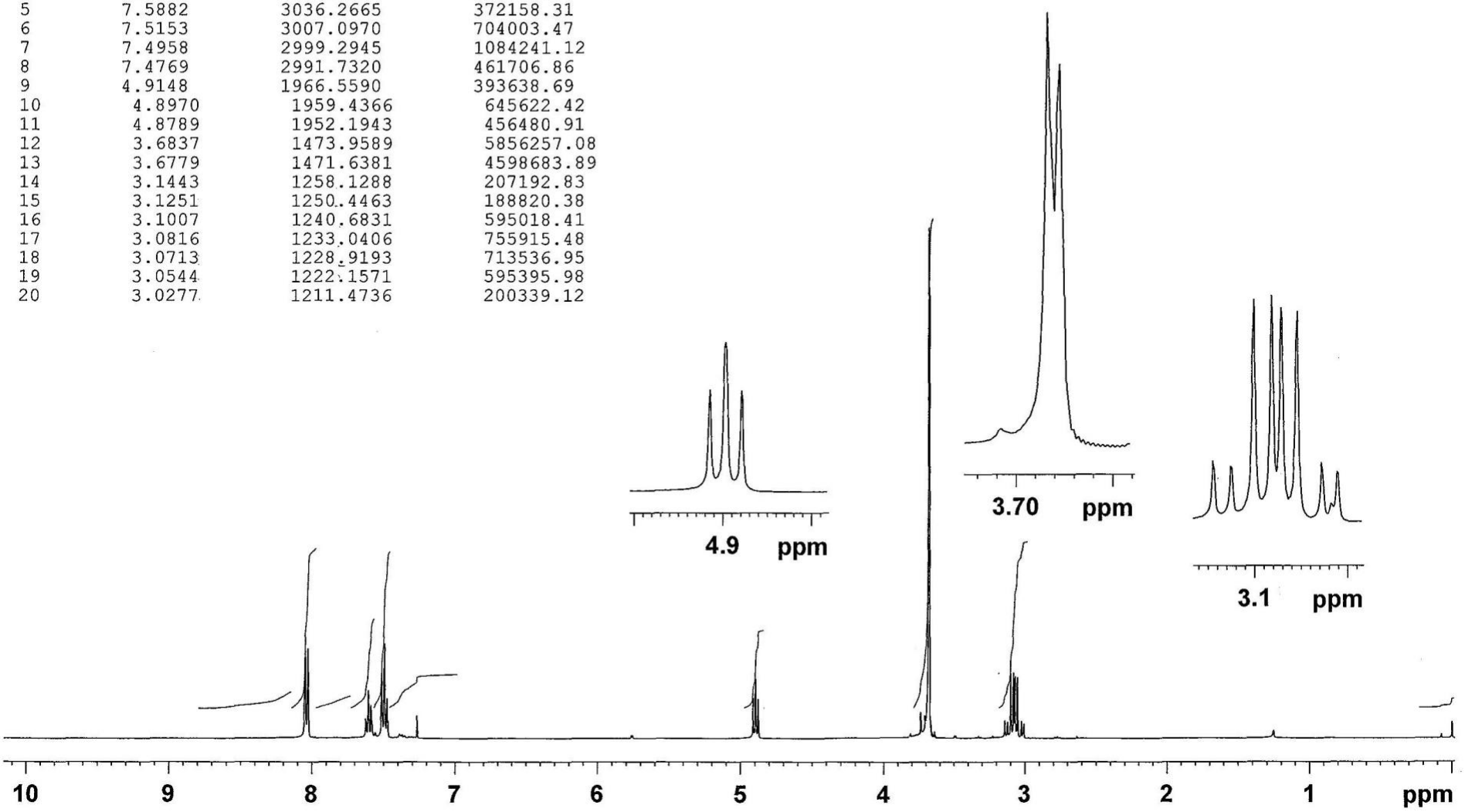


^{1}H NMR Spectrum of Dimethyl 2-(4-bromobenzoyl)pentanedioate (**2f**)



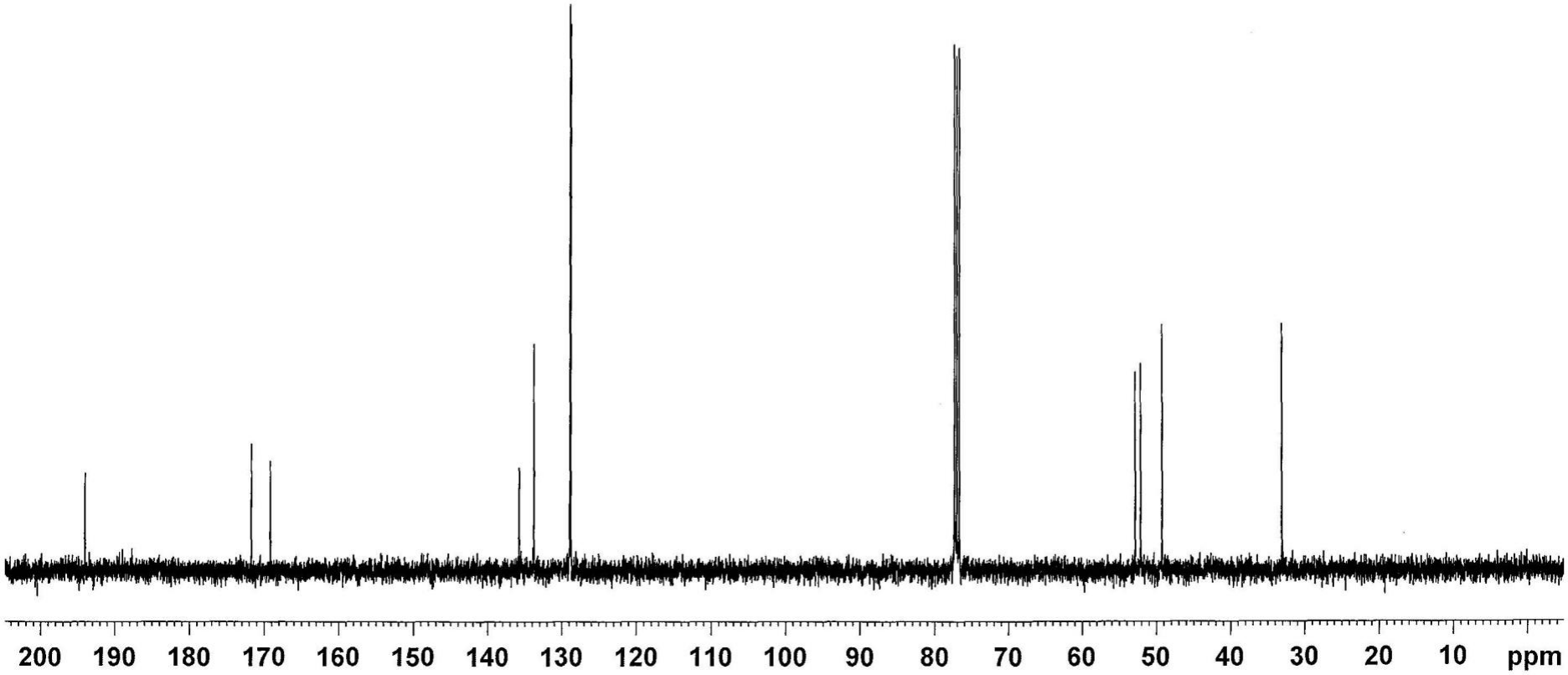
^{13}C NMR Spectrum of Dimethyl 2-(4-bromobenzoyl)pentanedioate (**2f**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	8.0495	3220.8465	920536.23
2	8.0312	3213.5241	1030951.11
3	8.0283	3212.3637	791770.28
4	7.6065	3043.5889	556438.55
5	7.5882	3036.2665	372158.31
6	7.5153	3007.0970	704003.47
7	7.4958	2999.2945	1084241.12
8	7.4769	2991.7320	461706.86
9	4.9148	1966.5590	393638.69
10	4.8970	1959.4366	645622.42
11	4.8789	1952.1943	456480.91
12	3.6837	1473.9589	5856257.08
13	3.6779	1471.6381	4598683.89
14	3.1443	1258.1288	207192.83
15	3.1251	1250.4463	188820.38
16	3.1007	1240.6831	595018.41
17	3.0816	1233.0406	755915.48
18	3.0713	1228.9193	713536.95
19	3.0544	1222.1571	595395.98
20	3.0277	1211.4736	200339.12



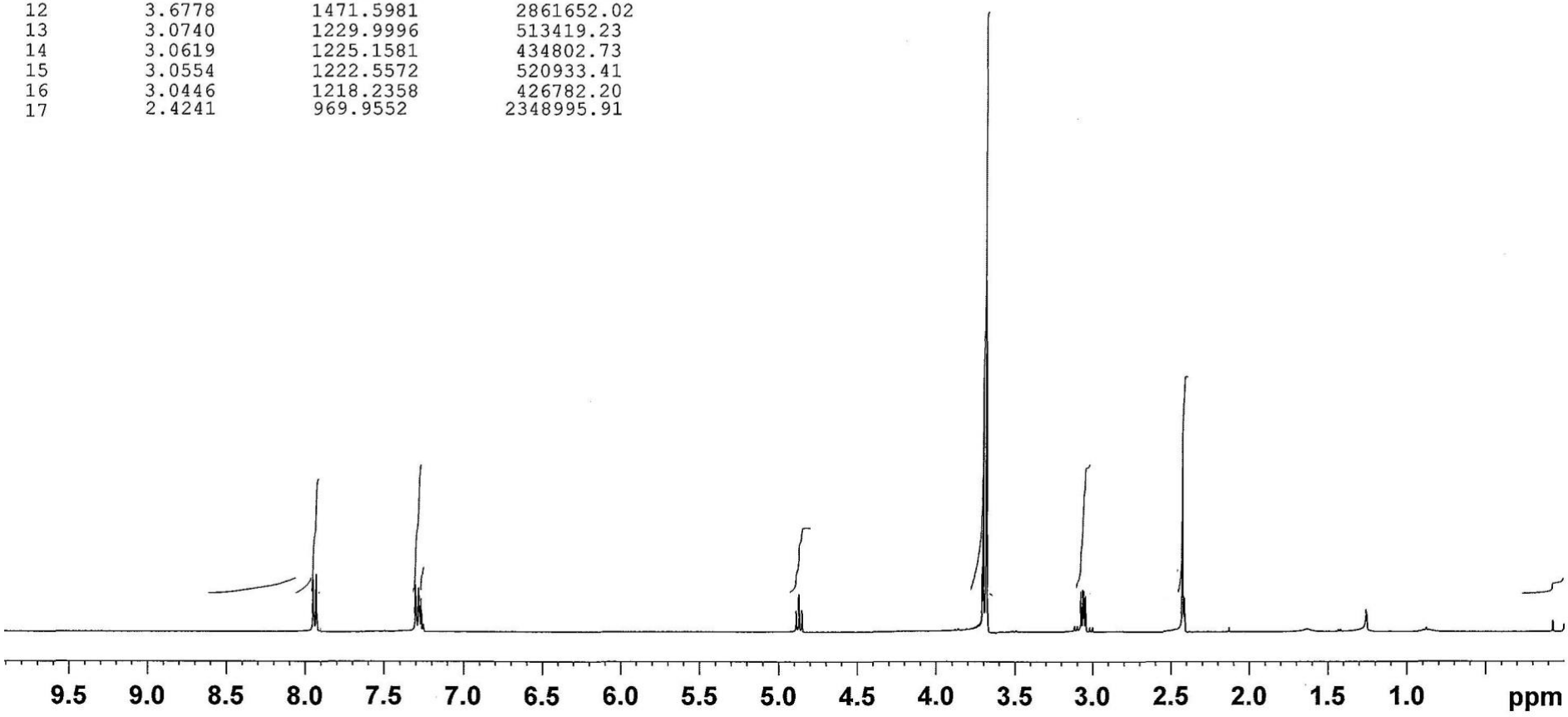
^{1}H NMR Spectrum of Dimethyl 2-benzoylsuccinate (**2g**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	194.0314	19522.0364	9989.85
2	171.7183	17277.0537	10914.92
3	169.1900	17022.6744	11175.69
4	135.7724	13660.4371	9062.09
5	133.7775	13459.7247	22975.87
6	128.9091	12969.9015	57289.85
7	128.7851	12957.4255	55295.71
8	77.3680	7784.2087	46314.24
9	77.0506	7752.2742	51966.80
10	76.7328	7720.2995	52799.36
11	52.8525	5317.6364	20117.89
12	52.1258	5244.5211	16199.99
13	49.2465	4954.8267	21072.11
14	33.0772	3327.9887	24955.12



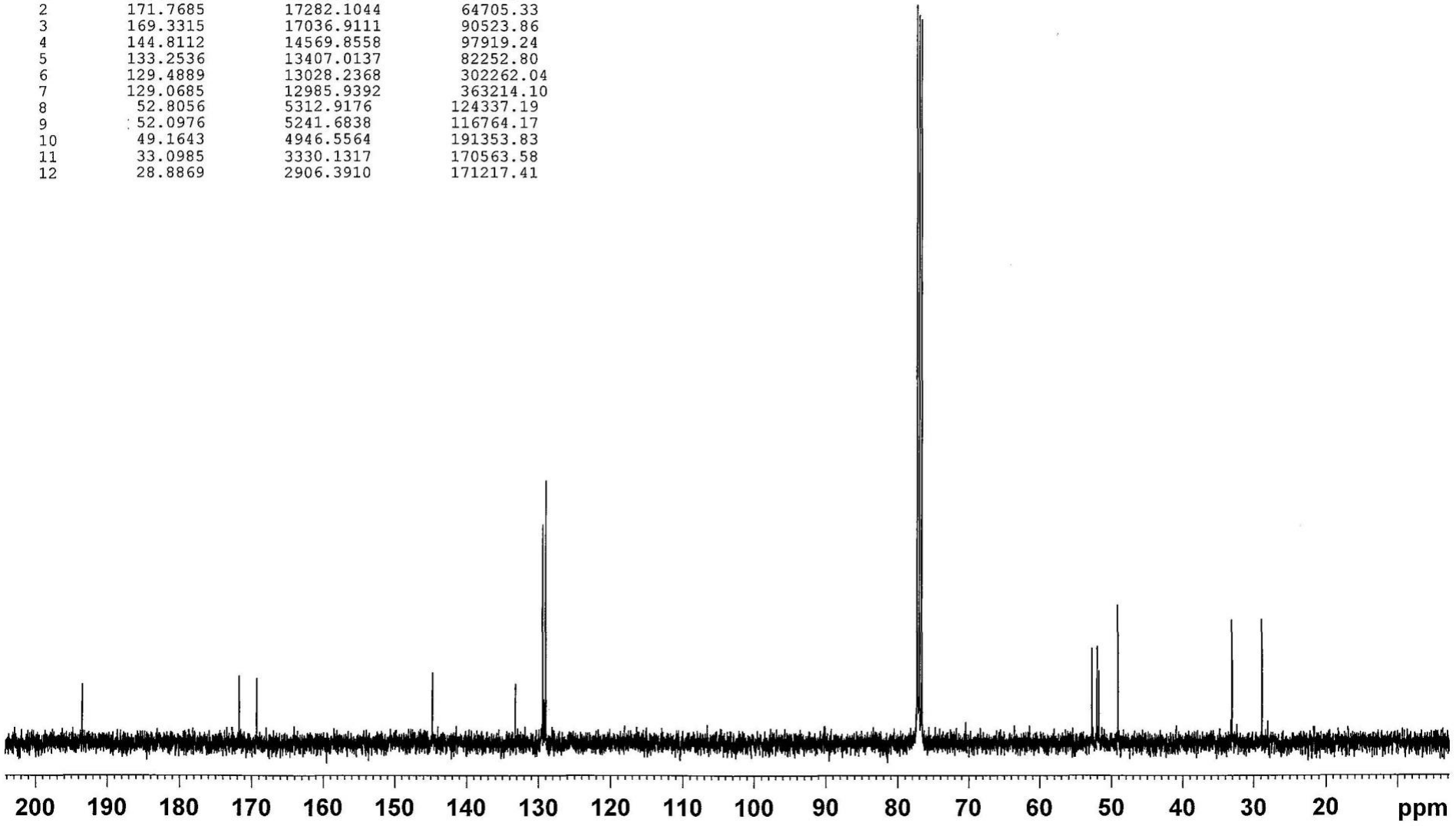
^{13}C NMR Spectrum of Dimethyl 2-benzoylsuccinate (**2g**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.9499	3180.9935	703079.12
2	7.9292	3172.7109	643572.91
3	7.3005	2921.1491	536892.62
4	7.2801	2912.9865	552216.80
5	7.2655	2907.1446	311239.52
6	4.8878	1955.7555	267091.53
7	4.8698	1948.5531	447015.78
8	4.8518	1941.3508	174585.98
9	3.7065	1483.0819	824408.94
10	3.6990	1480.0809	1936230.03
11	3.6814	1473.0386	4407064.14
12	3.6778	1471.5981	2861652.02
13	3.0740	1229.9996	513419.23
14	3.0619	1225.1581	434802.73
15	3.0554	1222.5572	520933.41
16	3.0446	1218.2358	426782.20
17	2.4241	969.9552	2348995.91



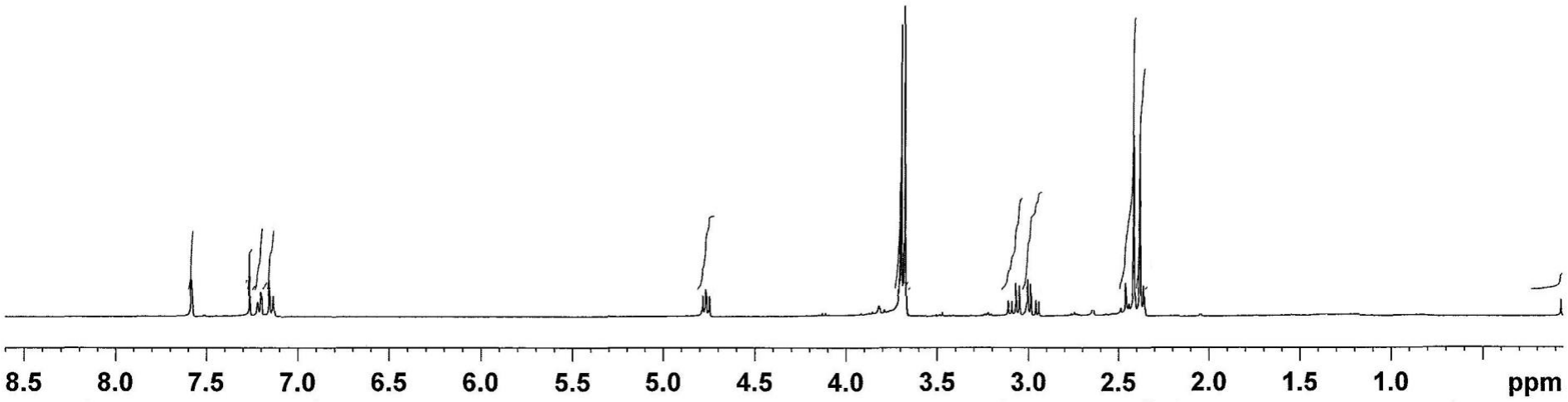
^{1}H NMR Spectrum of Dimethyl 2-(4-methylbenzoyl)succinate (**2h**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	193.5448	19473.0783	81792.94
2	171.7685	17282.1044	64705.33
3	169.3315	17036.9111	90523.86
4	144.8112	14569.8558	97919.24
5	133.2536	13407.0137	82252.80
6	129.4889	13028.2368	302262.04
7	129.0685	12985.9392	363214.10
8	52.8056	5312.9176	124337.19
9	52.0976	5241.6838	116764.17
10	49.1643	4946.5564	191353.83
11	33.0985	3330.1317	170563.58
12	28.8869	2906.3910	171217.41



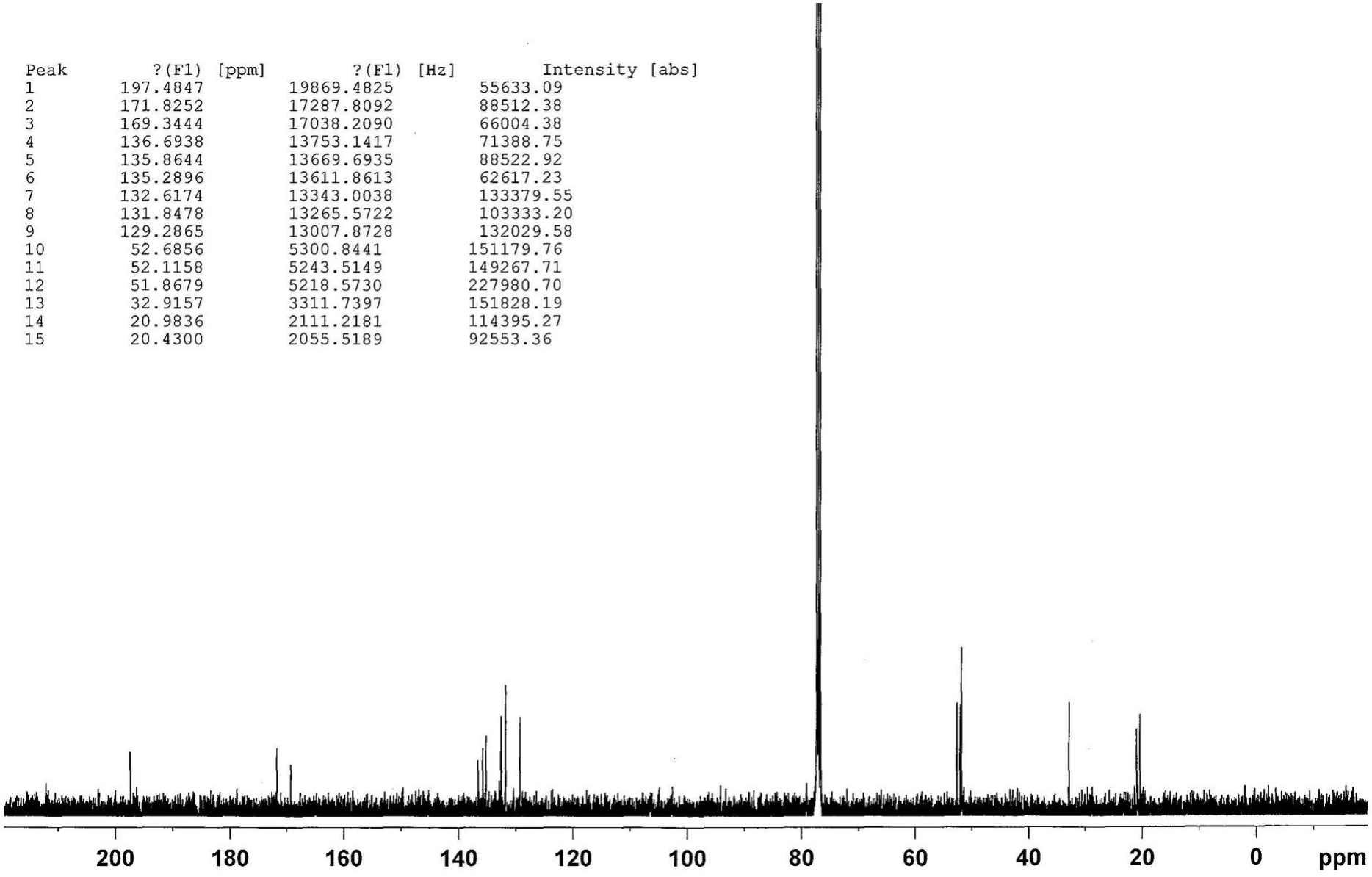
^{13}C NMR Spectrum of Dimethyl 2-(4-methylbenzoyl)succinate (**2h**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.5822	3033.8658	237123.55
2	7.2620	2905.7441	385079.11
3	7.2198	2888.8586	93291.11
4	7.2007	2881.2162	158155.57
5	7.1546	2862.7702	233701.06
6	7.1352	2855.0076	127436.90
7	7.1107	2845.2045	95337.89
8	4.7823	1913.5417	133086.92
9	4.7654	1906.7795	158812.53
10	4.7629	1905.7792	142345.52
11	4.7461	1899.0570	130990.93
12	3.6998	1480.4010	679742.56
13	3.6916	1477.1199	1369519.39
14	3.6724	1469.4374	1976261.50
15	3.1117	1245.0845	102212.07
16	3.0918	1237.1220	99918.58
17	3.0682	1227.6789	182236.19
18	3.0485	1219.7963	195863.12
19	3.0023	1201.3103	237850.45
20	2.9858	1194.7082	207173.43
21	2.9589	1183.9447	75531.09
22	2.9425	1177.3826	98879.96
23	2.4119	965.0736	1152084.85
24	2.3783	951.6292	1003578.64



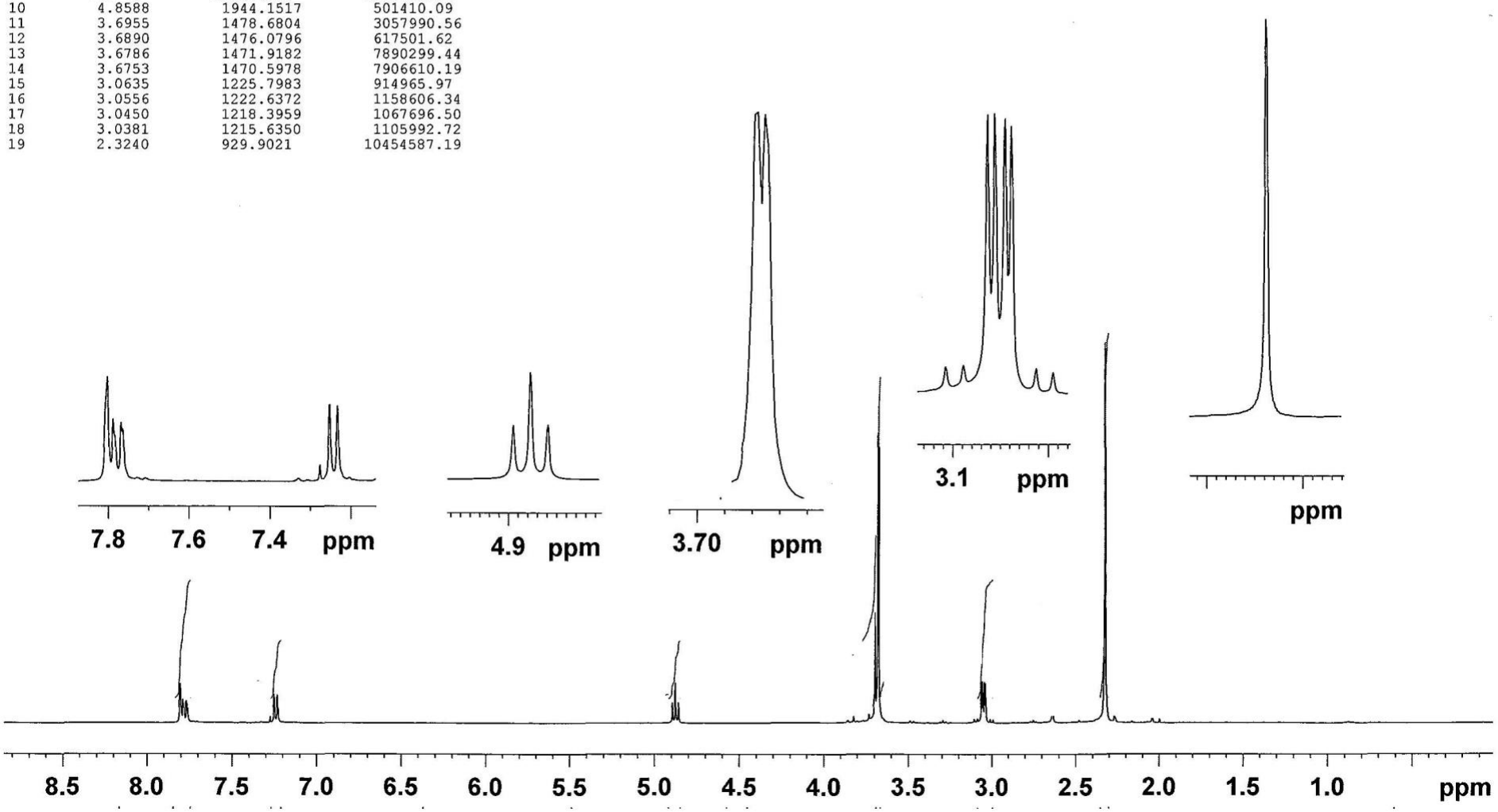
^{1}H NMR Spectrum of Dimethyl 2-(2,5-dimethylbenzoyl)succinate (**2i**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	197.4847	19869.4825	55633.09
2	171.8252	17287.8092	88512.38
3	169.3444	17038.2090	66004.38
4	136.6938	13753.1417	71388.75
5	135.8644	13669.6935	88522.92
6	135.2896	13611.8613	62617.23
7	132.6174	13343.0038	133379.55
8	131.8478	13265.5722	103333.20
9	129.2865	13007.8728	132029.58
10	52.6856	5300.8441	151179.76
11	52.1158	5243.5149	149267.71
12	51.8679	5218.5730	227980.70
13	32.9157	3311.7397	151828.19
14	20.9836	2111.2181	114395.27
15	20.4300	2055.5189	92553.36



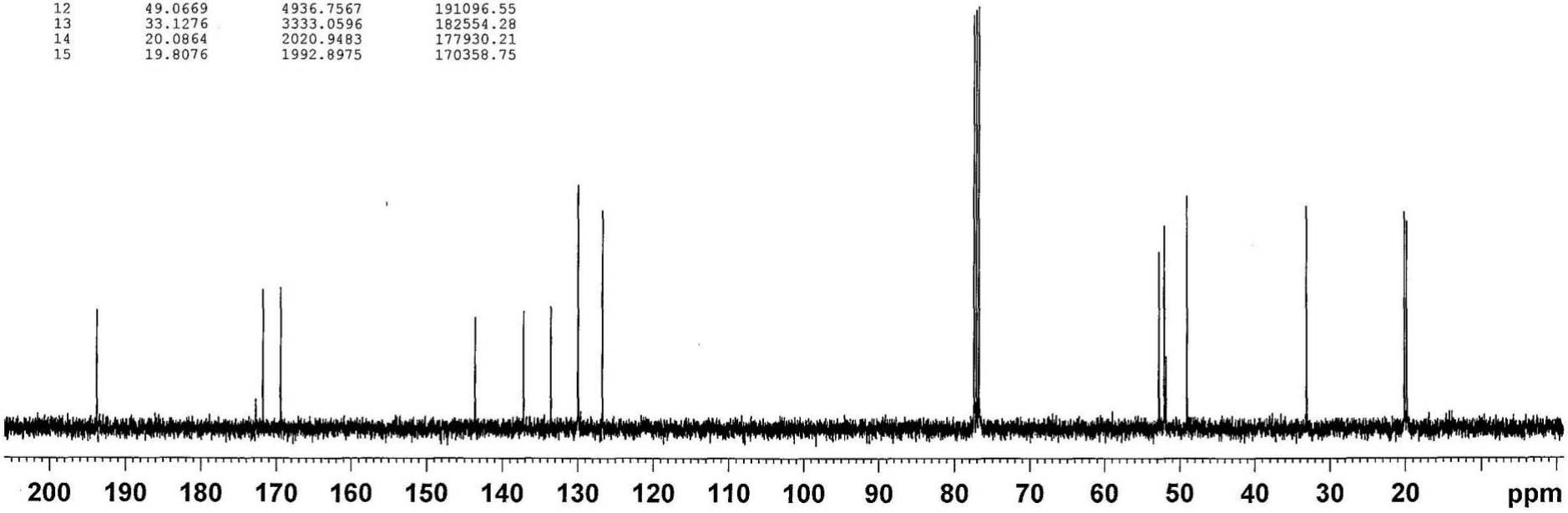
^{13}C NMR Spectrum of Dimethyl 2-(2,5-dimethylbenzoyl)succinate (**2i**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.8036	3122.4545	1084701.38
2	7.7886	3116.4525	646760.88
3	7.7844	3114.7720	471124.19
4	7.7688	3108.5300	563543.62
5	7.7648	3106.9294	542505.97
6	7.2525	2901.9428	790772.56
7	7.2330	2894.1403	783638.78
8	4.8949	1958.5964	563452.72
9	4.8770	1951.4340	1109419.12
10	4.8588	1944.1517	501410.09
11	3.6955	1478.6804	3057990.56
12	3.6890	1476.0796	617501.62
13	3.6786	1471.9182	7890299.44
14	3.6753	1470.5978	7906610.19
15	3.0635	1225.7983	914965.97
16	3.0556	1222.6372	1158606.34
17	3.0450	1218.3959	1067696.50
18	3.0381	1215.6350	1105992.72
19	2.3240	929.9021	10454587.19



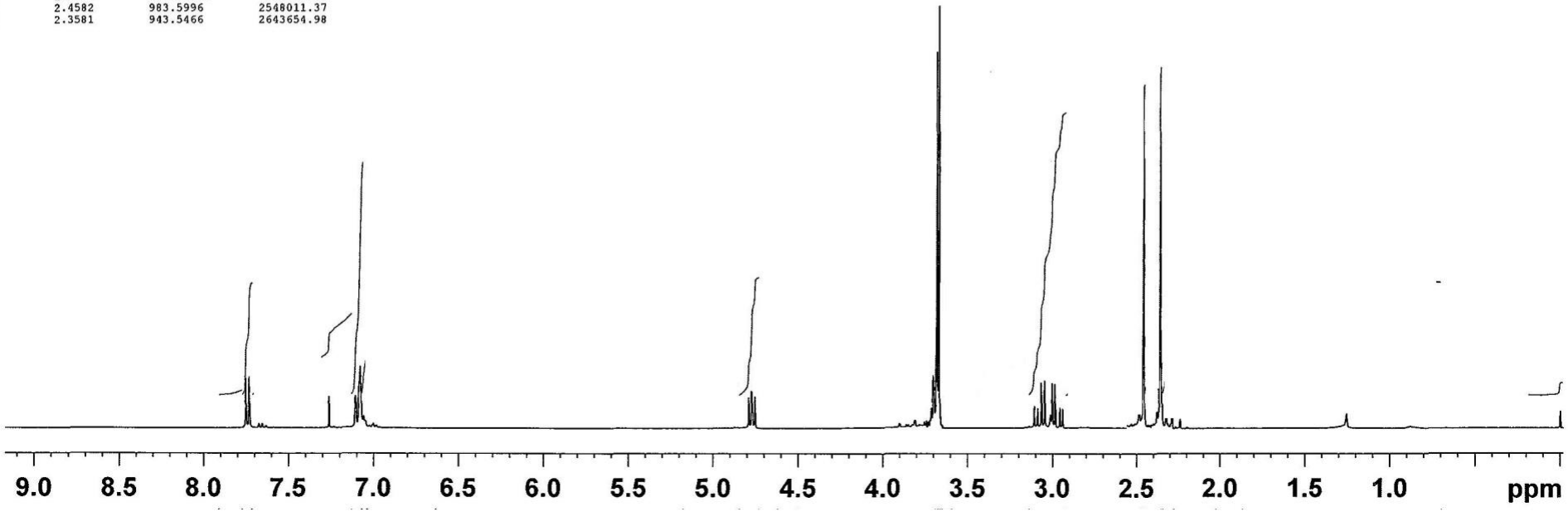
^{1}H NMR Spectrum of Dimethyl 2-(3,4-dimethylbenzoyl)succinate (**2j**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	193.7865	19497.3964	97725.21
2	171.7896	17284.2273	70187.81
3	169.4124	17045.0507	64164.93
4	143.6021	14448.2049	69701.86
5	137.2180	13805.8829	96906.90
6	133.5974	13441.6043	100640.58
7	129.9967	13079.3279	151777.61
8	129.9640	13076.0379	199880.22
9	126.7122	12748.8653	179058.76
10	52.7704	5309.3761	145805.73
11	52.0742	5239.3295	118310.05
12	49.0669	4936.7567	191096.55
13	33.1276	3333.0596	182554.28
14	20.0864	2020.9483	177930.21
15	19.8076	1992.8975	170358.75



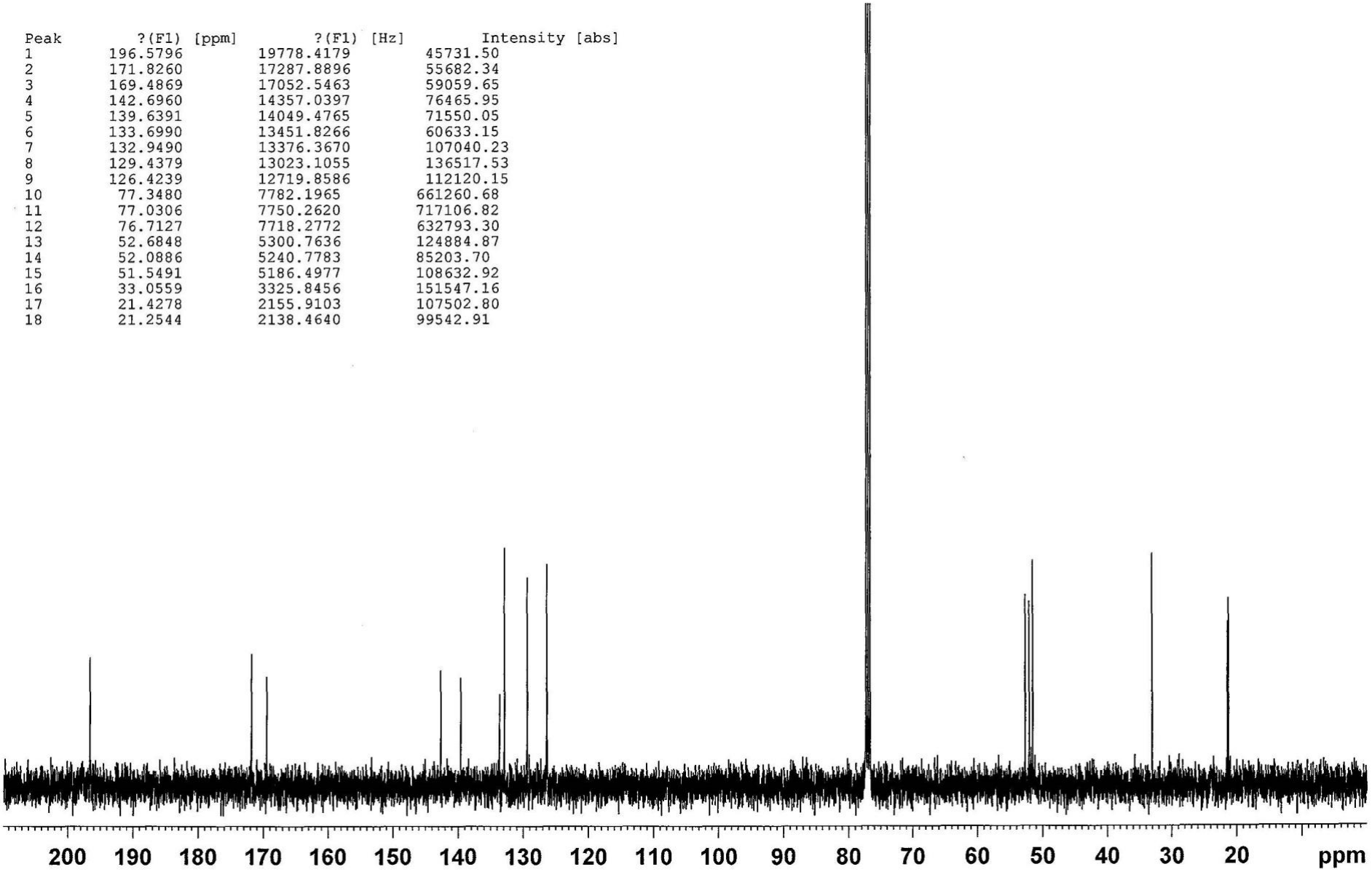
^{13}C NMR Spectrum of Dimethyl 2-(3,4-dimethylbenzoyl)succinate (**2j**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.7519	3101.7678	421249.88
2	7.7323	3093.9253	490486.10
3	7.2640	2906.5444	215804.88
4	7.1073	2843.8440	304389.11
5	7.0854	2835.0812	435432.35
6	7.0799	2832.8804	589579.66
7	4.7899	1916.5827	300800.85
8	4.7733	1909.9406	360916.94
9	4.7705	1908.8202	339236.56
10	4.7537	1902.0980	306543.91
11	3.6805	1472.6785	3606293.70
12	3.6687	1467.9570	2912713.34
13	3.1092	1244.0842	158750.52
14	3.0894	1236.1616	177454.41
15	3.0660	1226.7986	441836.28
16	3.0462	1218.8760	462009.93
17	3.0139	1205.9518	123112.49
18	3.0016	1201.0302	387689.48
19	2.9852	1194.4681	376369.34
20	2.9583	1183.7046	199636.44
21	2.9420	1177.1825	190584.42
22	2.4582	983.5996	2548011.37
23	2.3581	943.5466	2643654.98



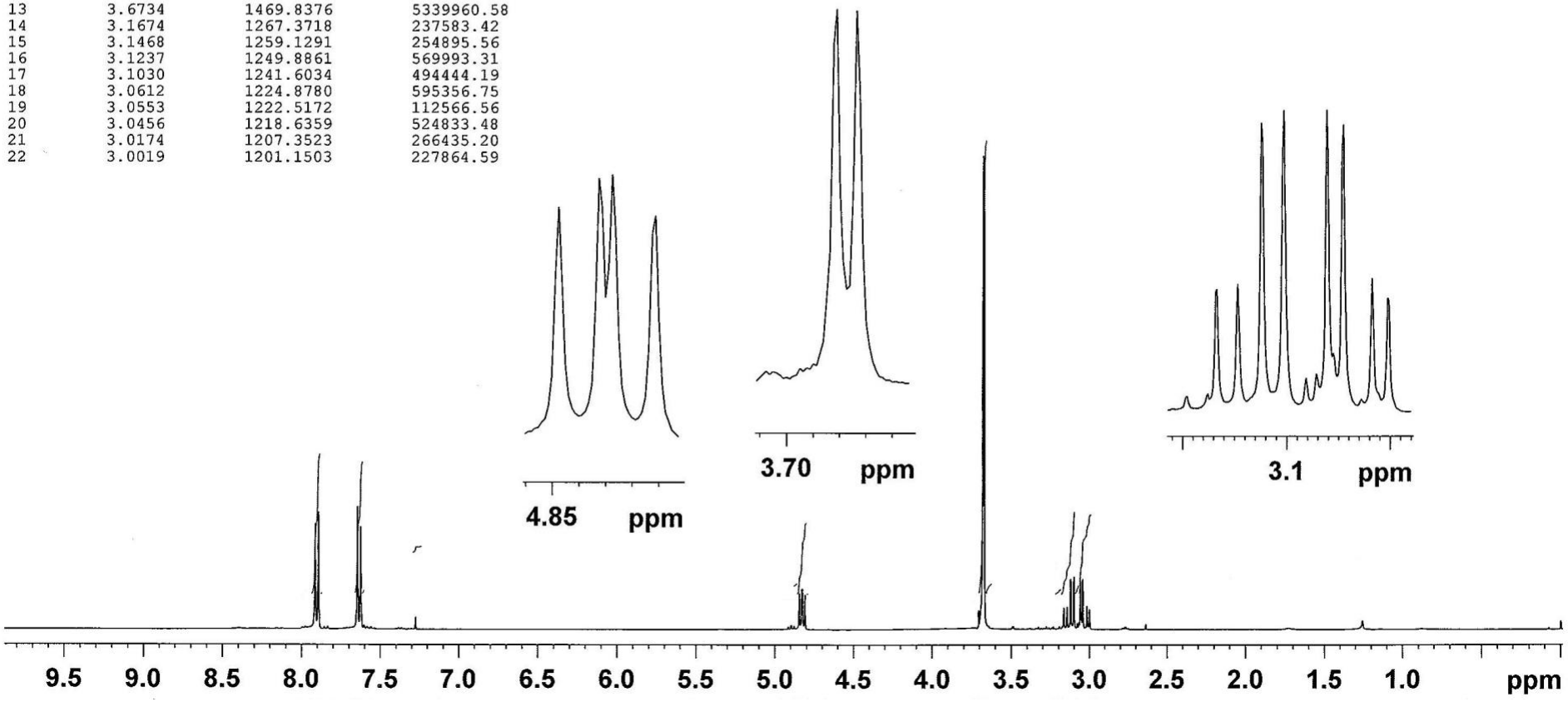
^{1}H NMR Spectrum of Dimethyl 2-(2,4-dimethylbenzoyl)succinate (**2k**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	196.5796	19778.4179	45731.50
2	171.8260	17287.8896	55682.34
3	169.4869	17052.5463	59059.65
4	142.6960	14357.0397	76465.95
5	139.6391	14049.4765	71550.05
6	133.6990	13451.8266	60633.15
7	132.9490	13376.3670	107040.23
8	129.4379	13023.1055	136517.53
9	126.4239	12719.8586	112120.15
10	77.3480	7782.1965	661260.68
11	77.0306	7750.2620	717106.82
12	76.7127	7718.2772	632793.30
13	52.6848	5300.7636	124884.87
14	52.0886	5240.7783	85203.70
15	51.5491	5186.4977	108632.92
16	33.0559	3325.8456	151547.16
17	21.4278	2155.9103	107502.80
18	21.2544	2138.4640	99542.91



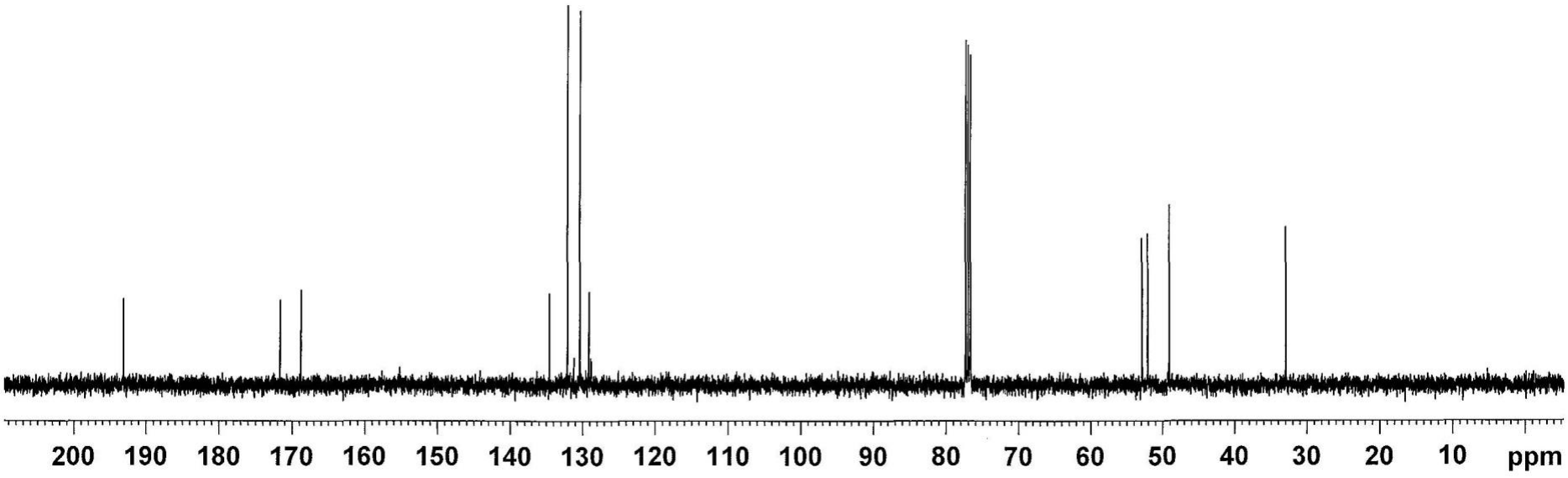
^{13}C NMR Spectrum of Dimethyl 2-(2,4-dimethylbenzoyl)succinate (**2k**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.9170	3167.8292	1188151.08
2	7.9126	3166.0687	395895.05
3	7.8998	3160.9470	438908.56
4	7.8955	3159.2264	1316282.00
5	7.6488	3060.5144	1384480.31
6	7.6442	3058.6738	412314.70
7	7.6274	3051.9516	1157557.77
8	4.8478	1939.7502	406010.89
9	4.8323	1933.5482	455729.02
10	4.8275	1931.6276	462076.38
11	4.8118	1925.3455	361543.36
12	3.6816	1473.1186	4879540.34
13	3.6734	1469.8376	5339960.58
14	3.1674	1267.3718	237583.42
15	3.1468	1259.1291	254895.56
16	3.1237	1249.8861	569993.31
17	3.1030	1241.6034	494444.19
18	3.0612	1224.8780	595356.75
19	3.0553	1222.5172	112566.56
20	3.0456	1218.6359	524833.48
21	3.0174	1207.3523	266435.20
22	3.0019	1201.1503	227864.59



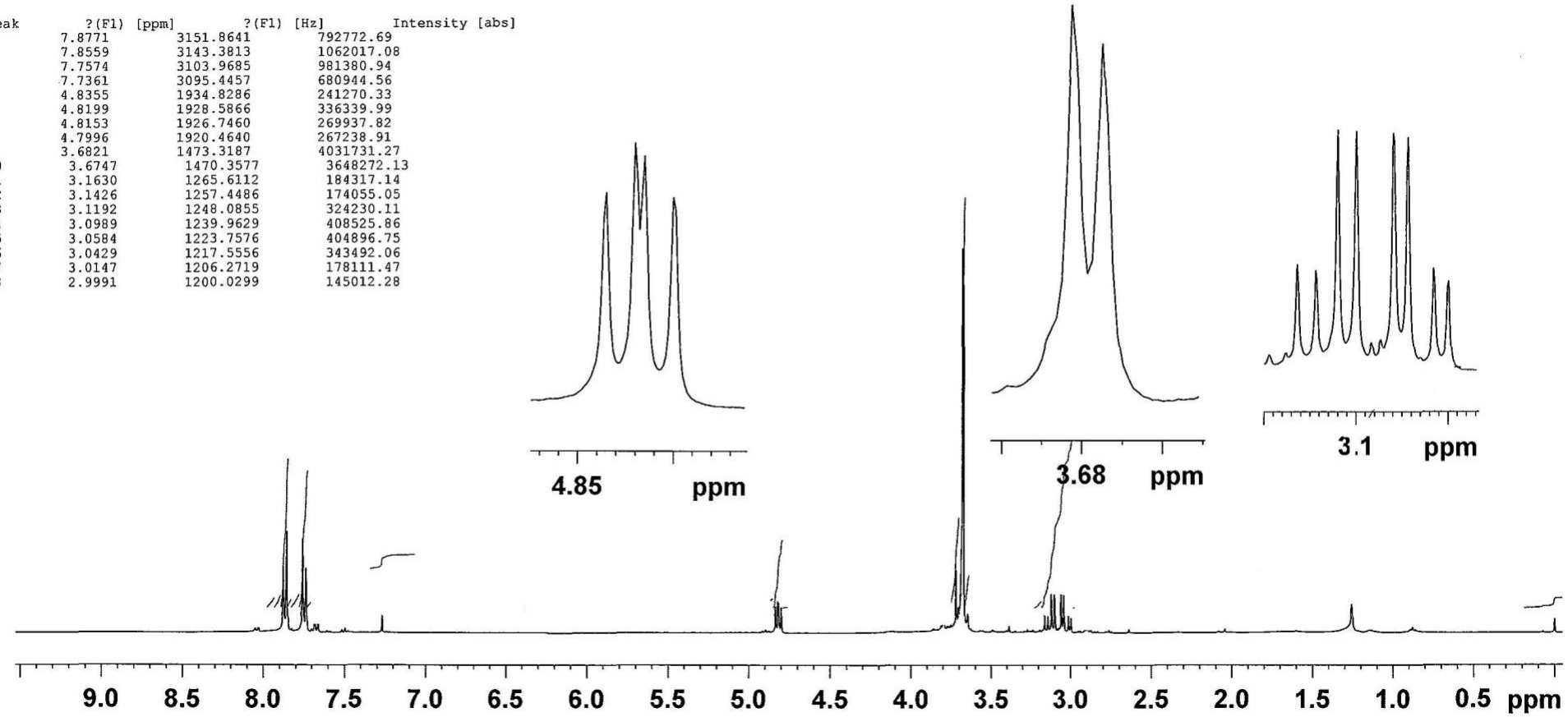
^{1}H NMR Spectrum of Dimethyl 2-(4-bromobenzoyl)succinate (**2l**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	193.1387	19432.2194	29640.21
2	171.6619	17271.3791	47329.34
3	168.8085	16984.2906	39751.62
4	134.6178	13544.2696	51099.61
5	132.1096	13291.9127	209898.61
6	130.4031	13120.2170	206959.04
7	129.1277	12991.8955	51848.63
8	52.9543	5327.8788	81797.16
9	52.1684	5248.8072	84008.63
10	49.1264	4942.7431	99790.74
11	32.9801	3318.2192	80252.42



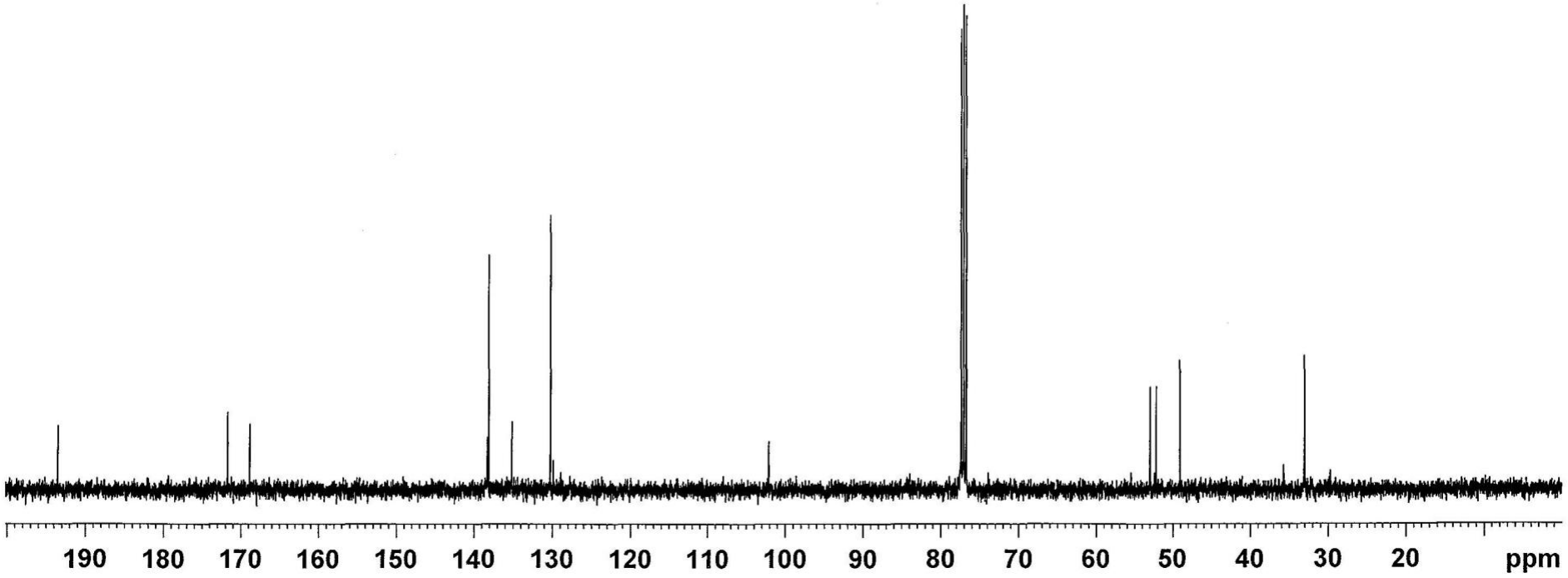
^{13}C NMR Spectrum of Dimethyl 2-(4-bromobenzoyl)succinate (**2l**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	7.8771	3151.8641	792772.69
2	7.8559	3143.3813	1062017.08
3	7.7574	3103.9685	981380.94
4	7.7361	3095.4457	680944.56
5	4.8355	1934.8286	241270.33
6	4.8199	1928.5866	336339.99
7	4.8153	1926.7460	269937.82
8	4.7996	1920.4640	267238.91
9	3.6821	1473.3187	4031731.27
10	3.6747	1470.3577	3648272.13
11	3.1630	1265.6112	184317.14
12	3.1426	1257.4486	174055.05
13	3.1192	1248.0855	324230.11
14	3.0989	1239.9629	408525.86
15	3.0584	1223.7576	404896.75
16	3.0429	1217.5556	343492.06
17	3.0147	1206.2719	178111.47
18	2.9991	1200.0299	145012.28



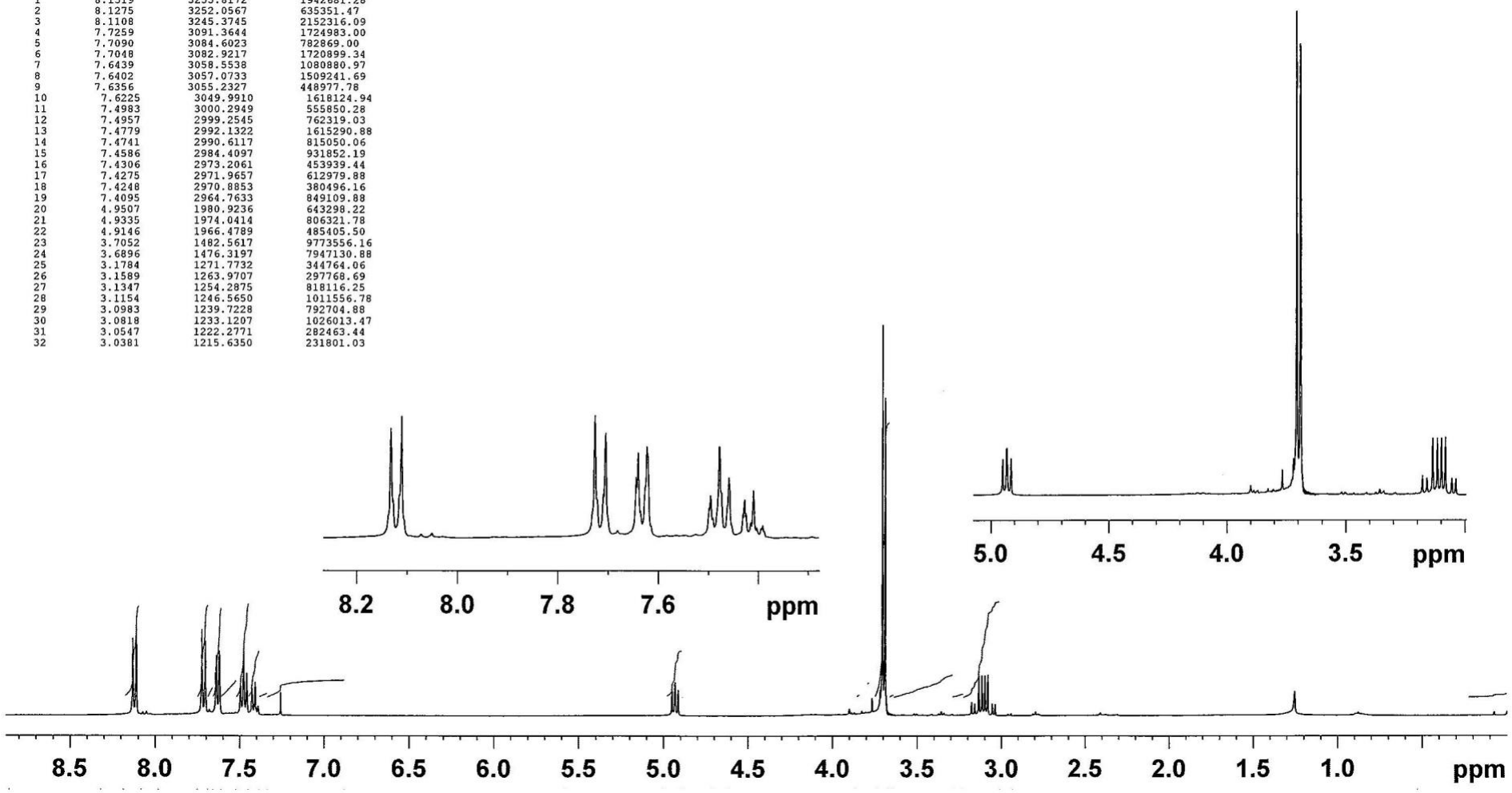
^{1}H NMR Spectrum of Dimethyl 2-(4-iodobenzoyl)succinate (**2m**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	193.4661	19465.1600	59090.57
2	171.6582	17271.0068	39706.53
3	168.8082	16984.2604	40843.71
4	138.1222	13896.8570	214794.55
5	135.1570	13598.5200	62637.25
6	130.2290	13102.7003	144252.82
7	102.0775	10270.2999	27668.41
8	52.9614	5328.5931	94273.69
9	52.1772	5249.6926	76297.94
10	49.0675	4936.8170	118742.27
11	32.9829	3318.5009	122546.42



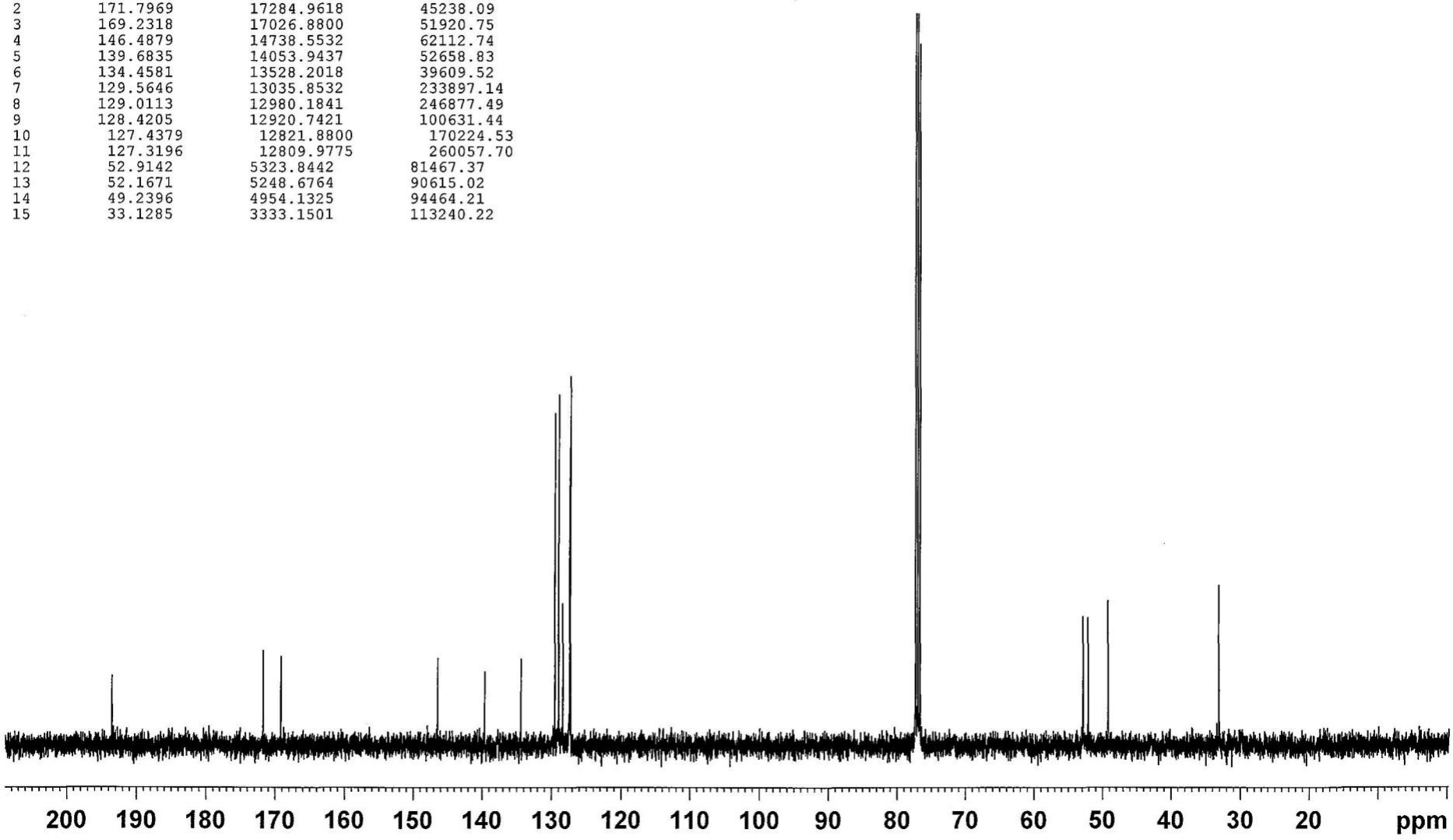
^{13}C NMR Spectrum of Dimethyl 2-(4-iodobenzoyl)succinate (**2m**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	8.1319	3253.8172	1942681.28
2	8.1275	3252.0567	635351.47
3	8.1108	3245.3745	2152316.09
4	7.7259	3091.3644	1724983.00
5	7.7090	3084.6023	782869.00
6	7.7048	3082.9217	1720899.34
7	7.6439	3058.5538	1080880.97
8	7.6402	3057.0733	1509241.69
9	7.6356	3055.2327	448977.78
10	7.6225	3049.9910	1618124.94
11	7.4983	3000.2949	555850.28
12	7.4957	2999.2545	762319.03
13	7.4779	2992.1322	1615290.88
14	7.4741	2990.6117	815050.06
15	7.4586	2984.4097	931852.19
16	7.4306	2973.2061	453939.44
17	7.4275	2971.9657	612979.88
18	7.4248	2970.8853	380496.16
19	7.4095	2964.7633	849109.88
20	4.9507	1980.9236	643298.22
21	4.9335	1974.0414	806321.78
22	4.9146	1966.4789	485405.50
23	3.7052	1482.5617	9773556.16
24	3.6896	1476.3197	7947130.88
25	3.1784	1271.7732	344764.06
26	3.1589	1263.9707	297768.69
27	3.1347	1254.2875	818116.25
28	3.1154	1246.5650	1011556.78
29	3.0983	1239.7228	792704.88
30	3.0818	1233.1207	1026013.47
31	3.0547	1222.2771	282463.44
32	3.0381	1215.6350	231801.03

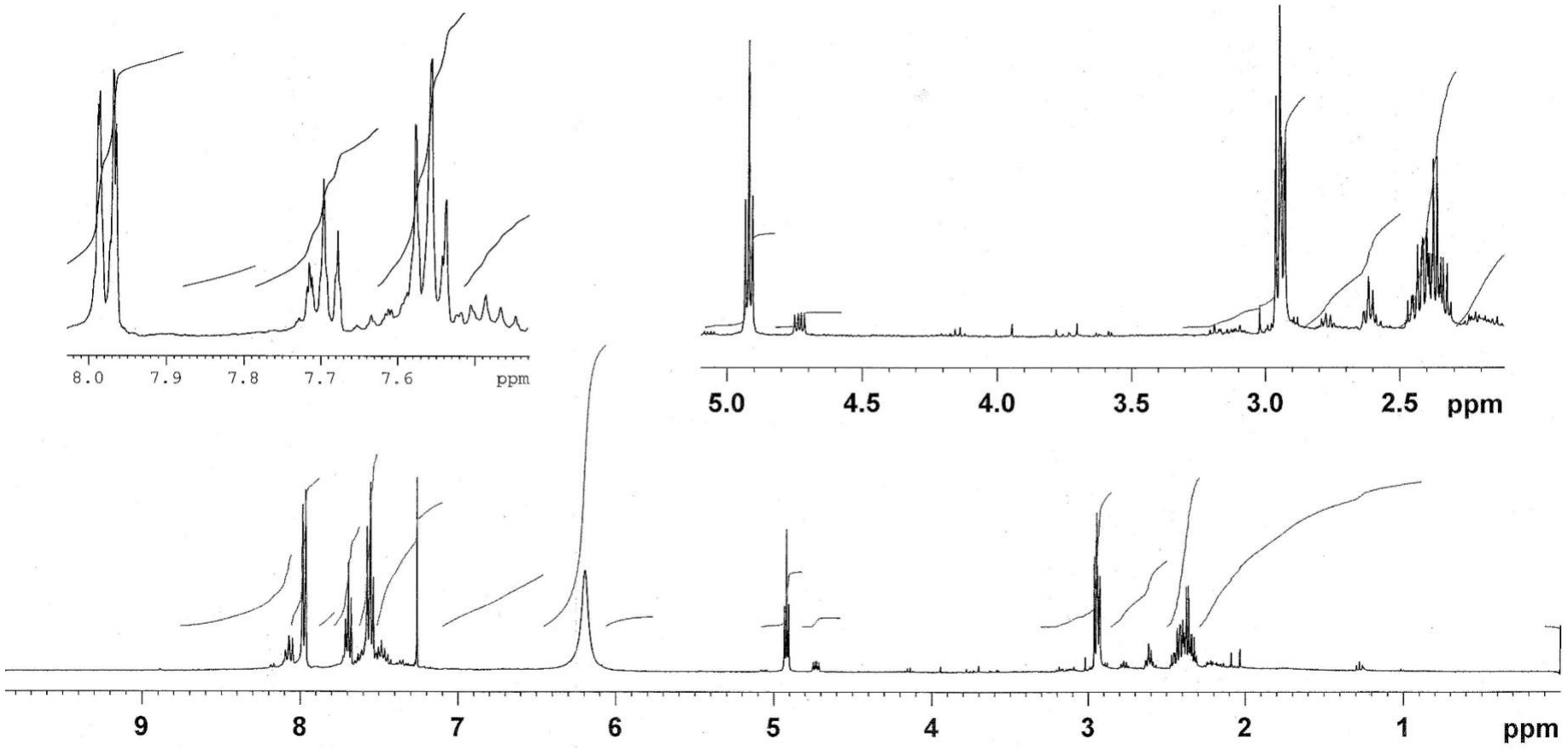


^{1}H NMR Spectrum of Dimethyl 2-([1,1'-biphenyl]-4-carbonyl)succinate (**2n**)

Peak	?(F1) [ppm]	?(F1) [Hz]	Intensity [abs]
1	193.5873	19477.3543	49692.54
2	171.7969	17284.9618	45238.09
3	169.2318	17026.8800	51920.75
4	146.4879	14738.5532	62112.74
5	139.6835	14053.9437	52658.83
6	134.4581	13528.2018	39609.52
7	129.5646	13035.8532	233897.14
8	129.0113	12980.1841	246877.49
9	128.4205	12920.7421	100631.44
10	127.4379	12821.8800	170224.53
11	127.3196	12809.9775	260057.70
12	52.9142	5323.8442	81467.37
13	52.1671	5248.6764	90615.02
14	49.2396	4954.1325	94464.21
15	33.1285	3333.1501	113240.22



^{13}C NMR Spectrum of Dimethyl 2-([1,1'-biphenyl]-4-carbonyl)succinate (**2n**)



^{1}H NMR Spectrum of 3-Benzoyldihydro-2H-pyran-2,6(3H)-dione (**3**) (The reaction mixture)