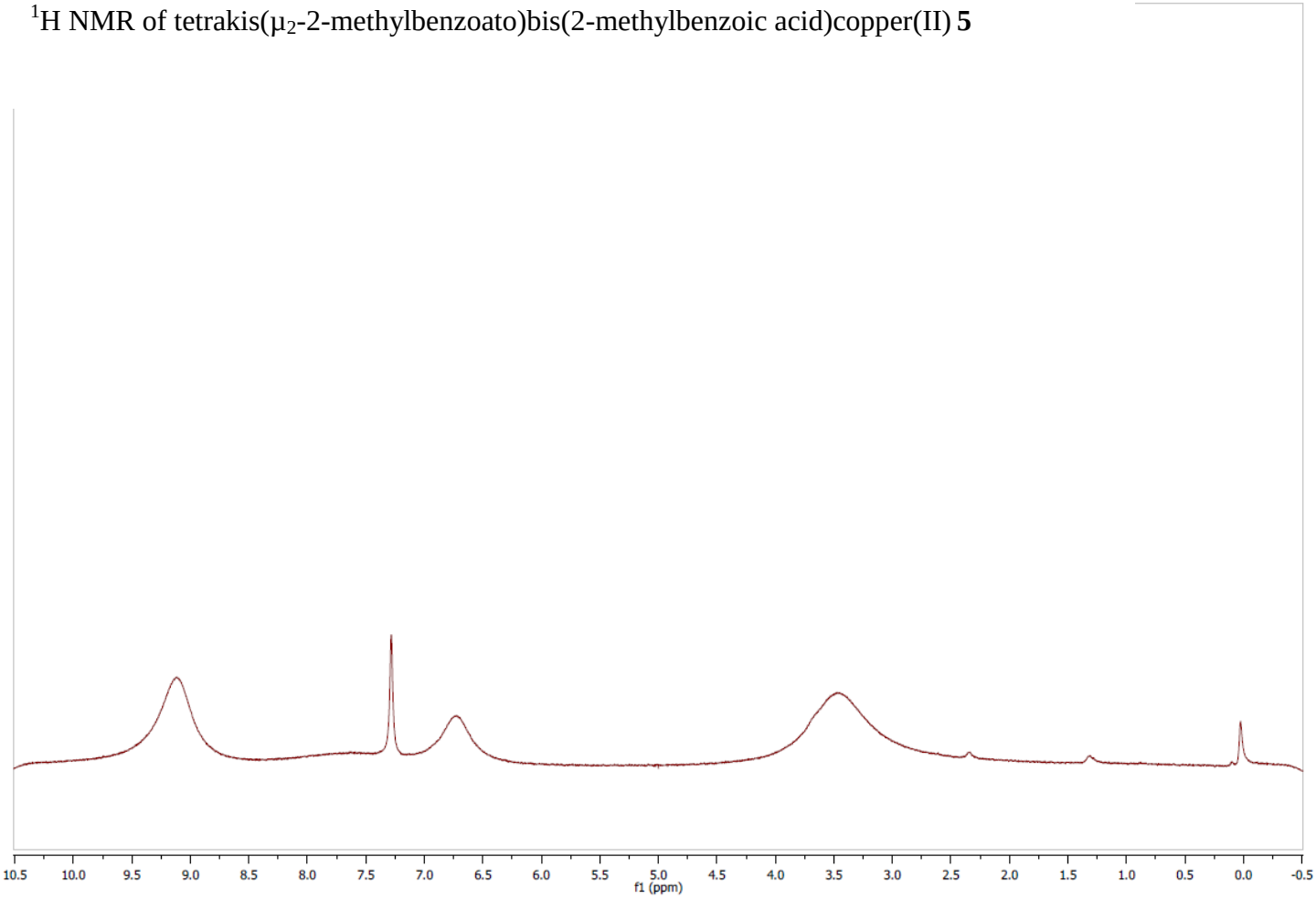
Electronic Supplementary Material (ESI) for Catalysis Science & Technology

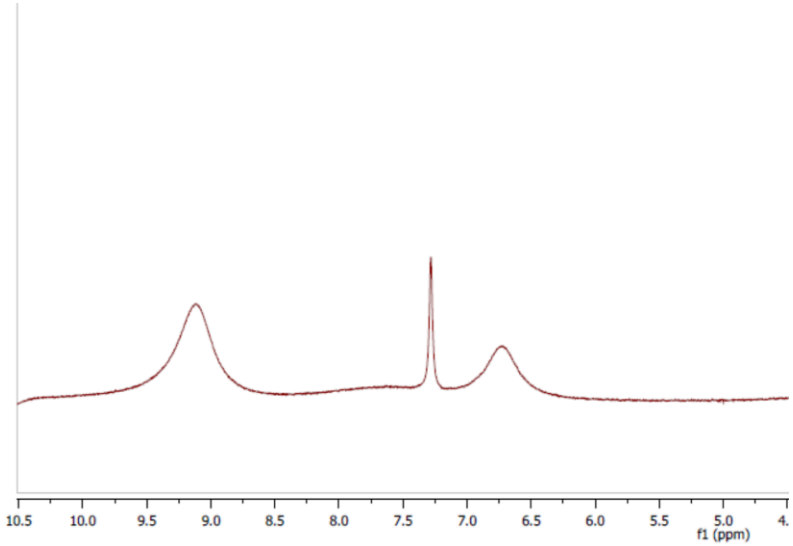
Thermo-analytical determination of intermediates in the copper catalysed rearrangement of *o*-toluic acid to *meta*-cresol

Abraham C. Sunil, Ernst H.G. Langner, Charlene Marais, and Barend C.B. Bezuidenhoudt[a*]

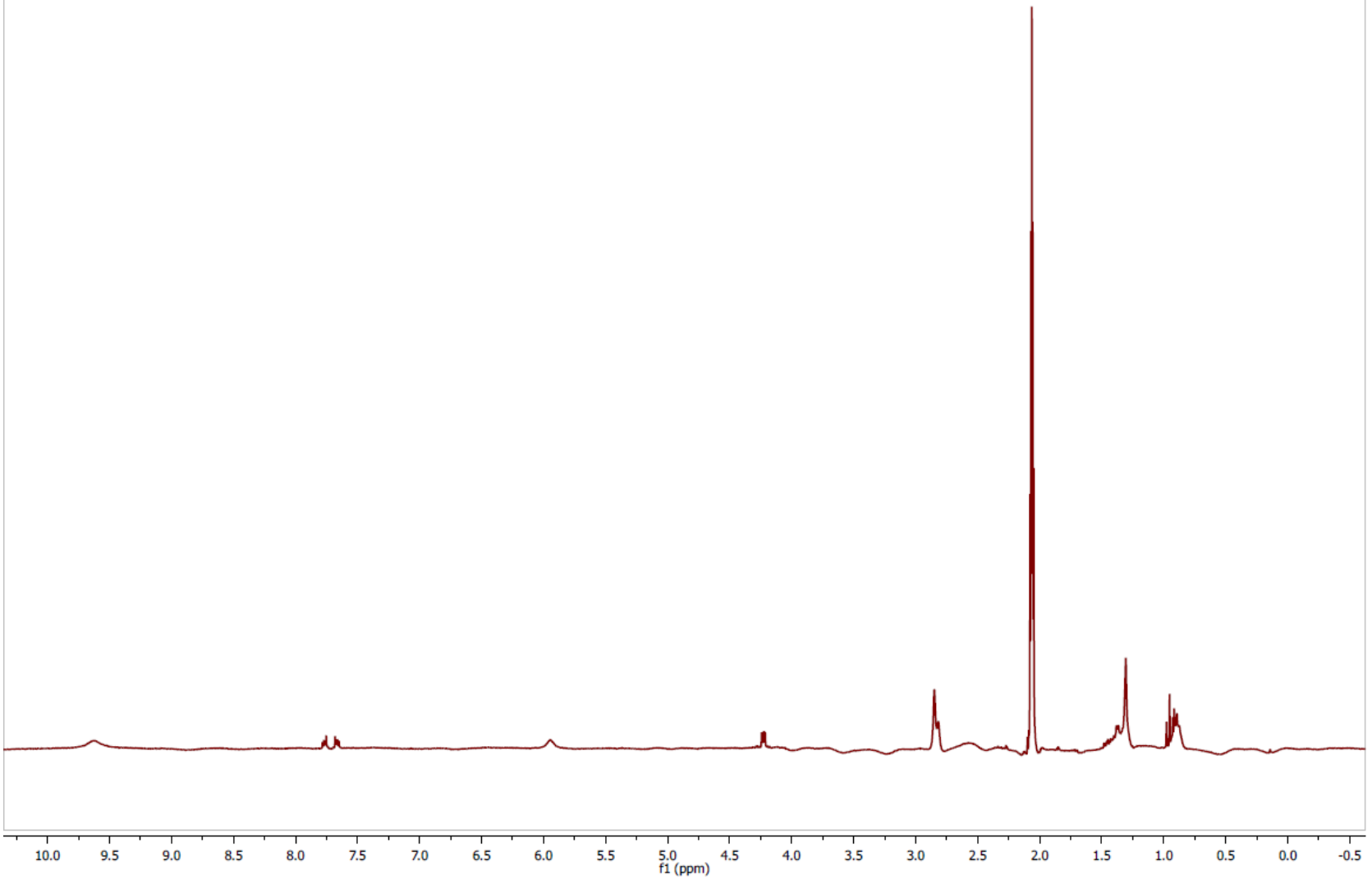
^{1}H NMR of tetrakis(μ$_2$-2-methylbenzoato)bis(2-methylbenzoic acid)copper(II) **5**



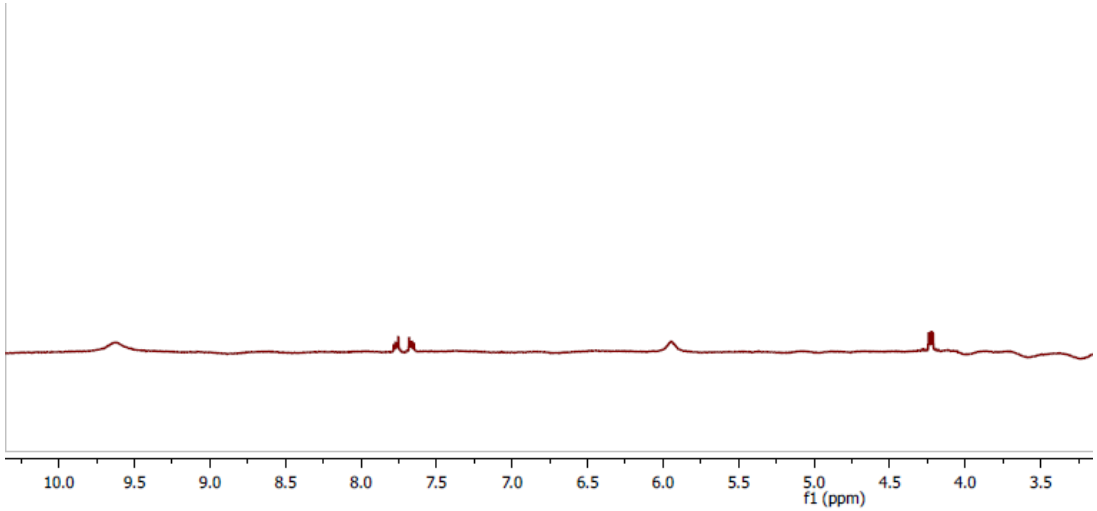
^{1}H NMR of tetrakis(μ_2-2-methylbenzoato)bis(2-methylbenzoic acid)copper(II) **5**

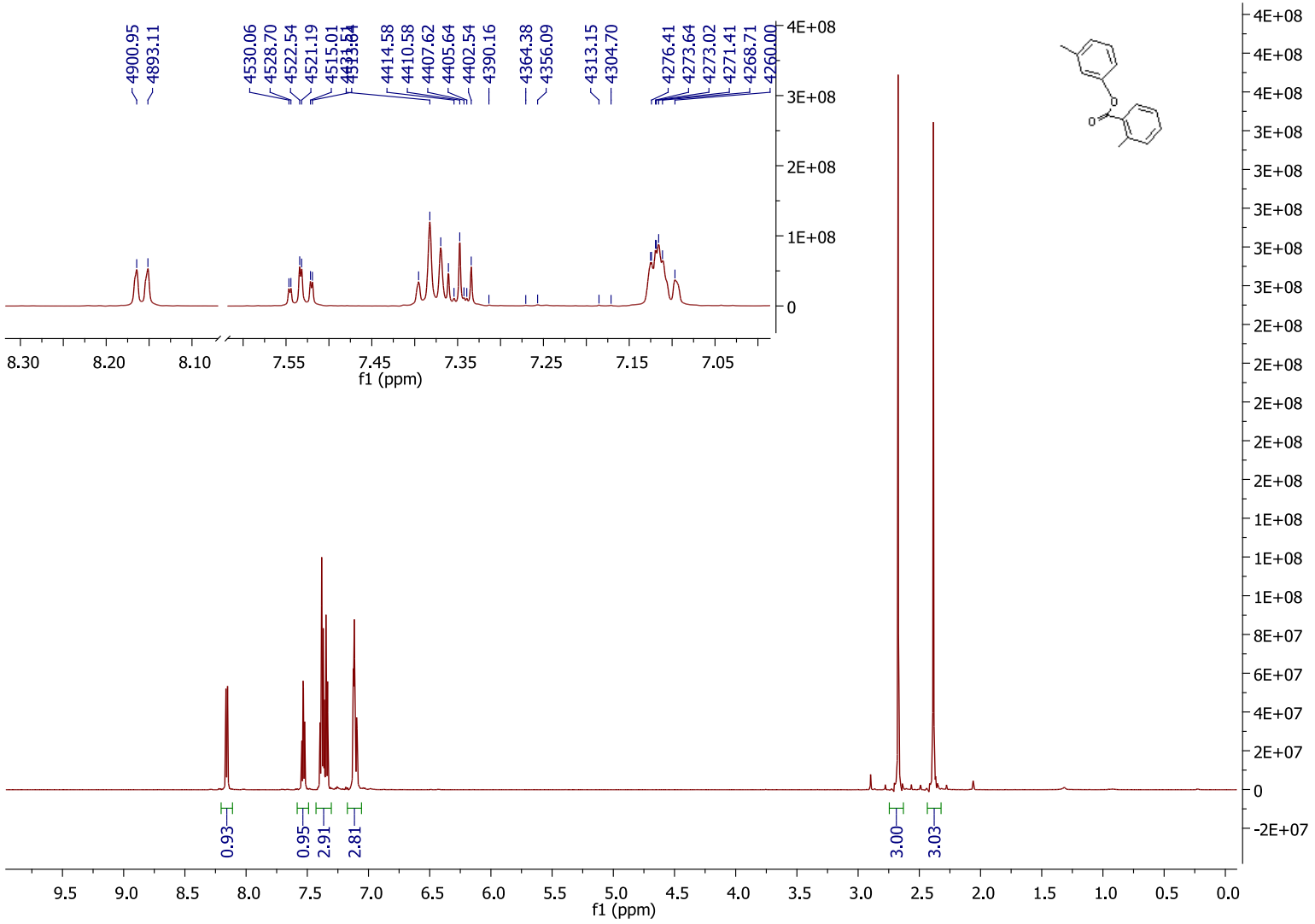


^{1}H NMR of DSC product tetrakis(μ_2-2-methylbenzoato) copper(II) **6**

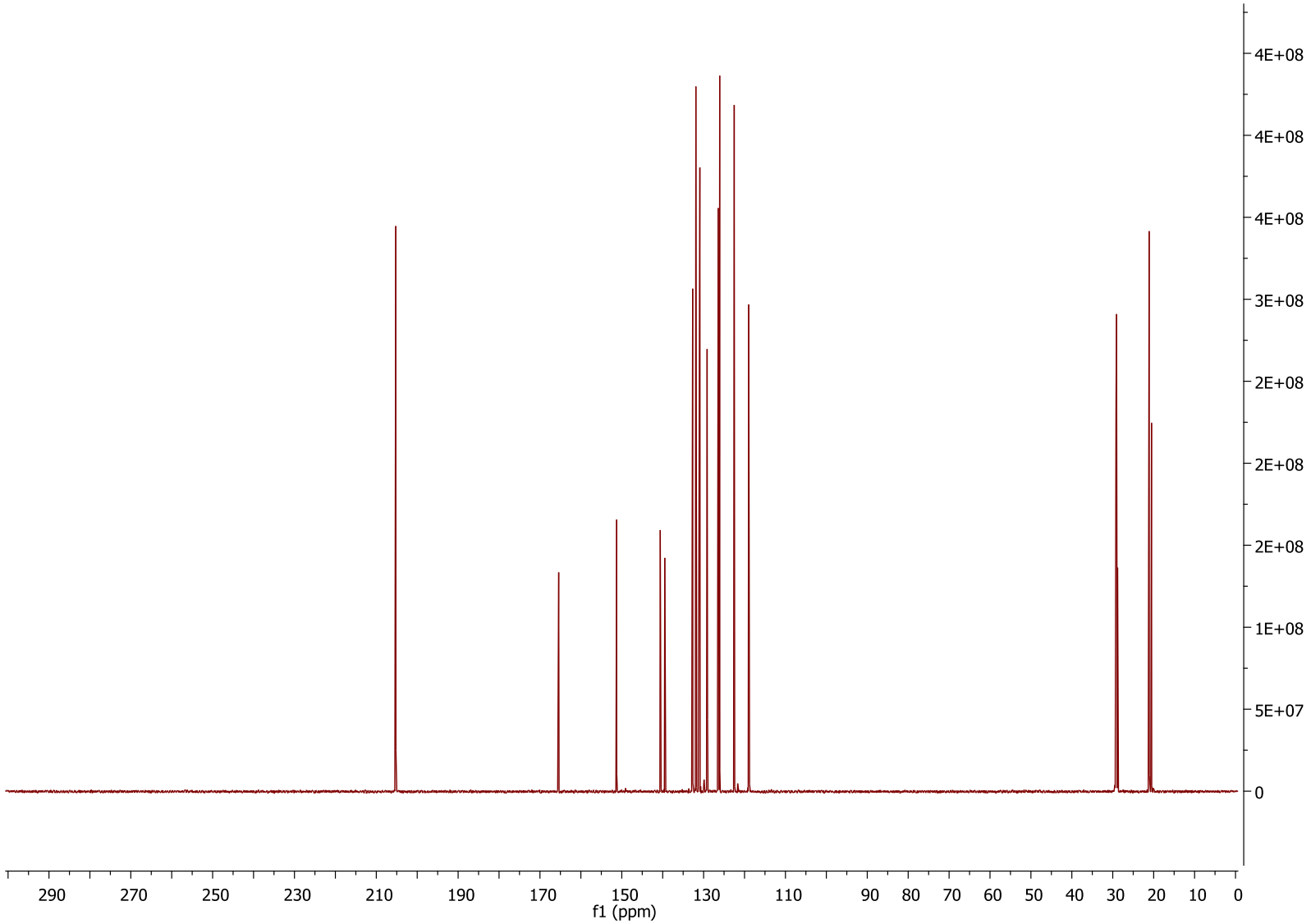


^{1}H NMR of DSC product tetrakis(μ_2-2-methylbenzoato) copper(II) **6**

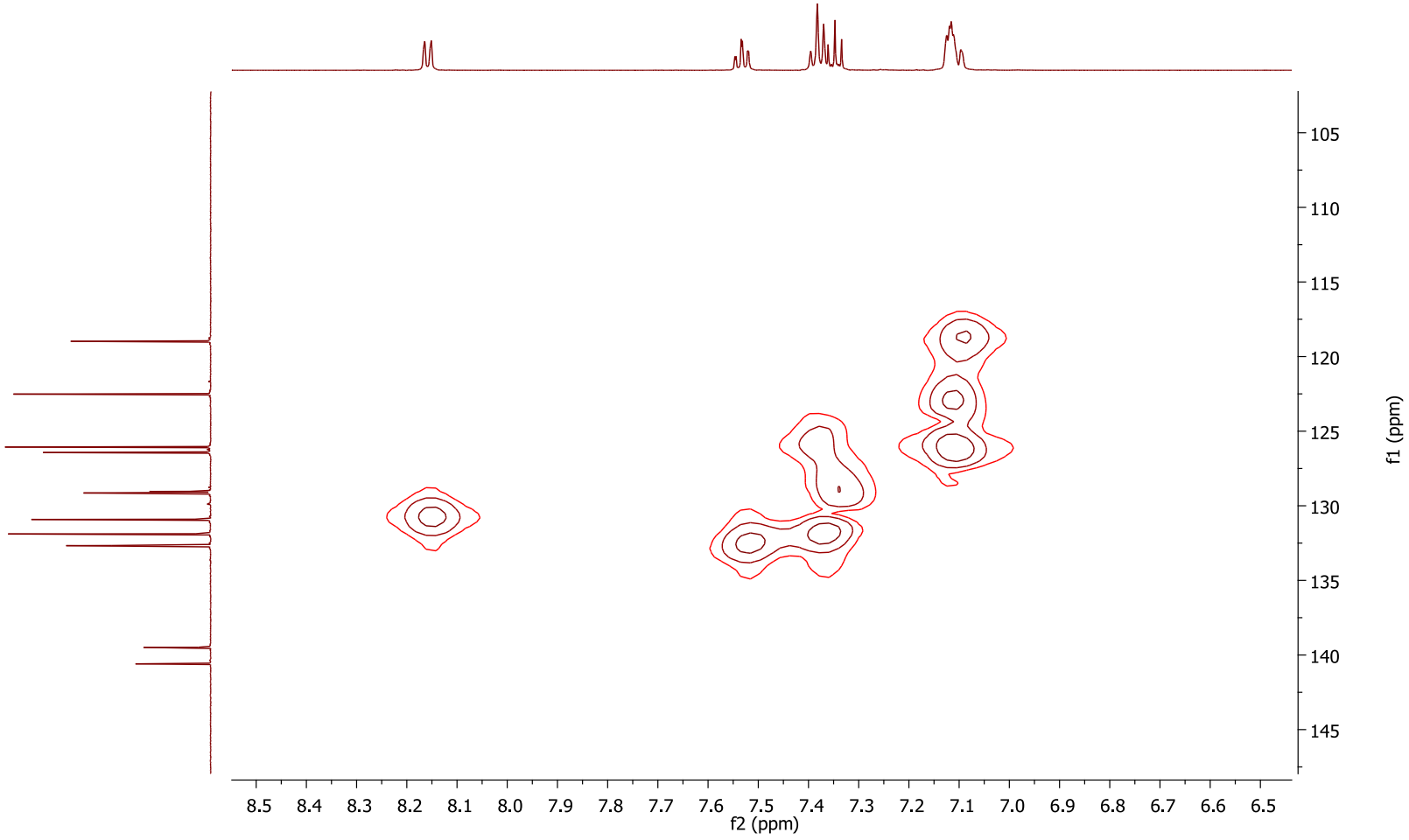




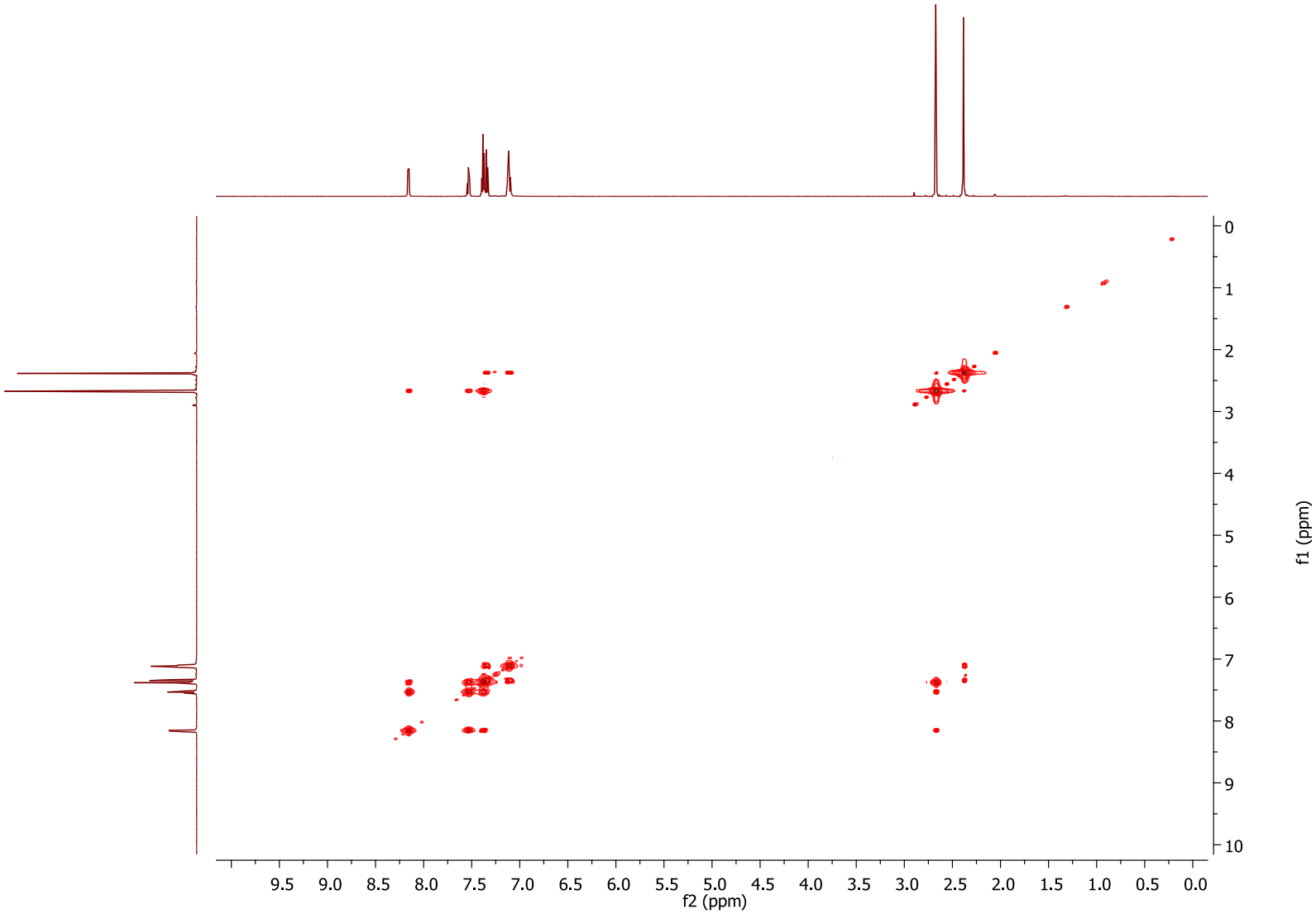
^{1}H NMR of 3-methylphenyl 2-methylbenzoate **4**



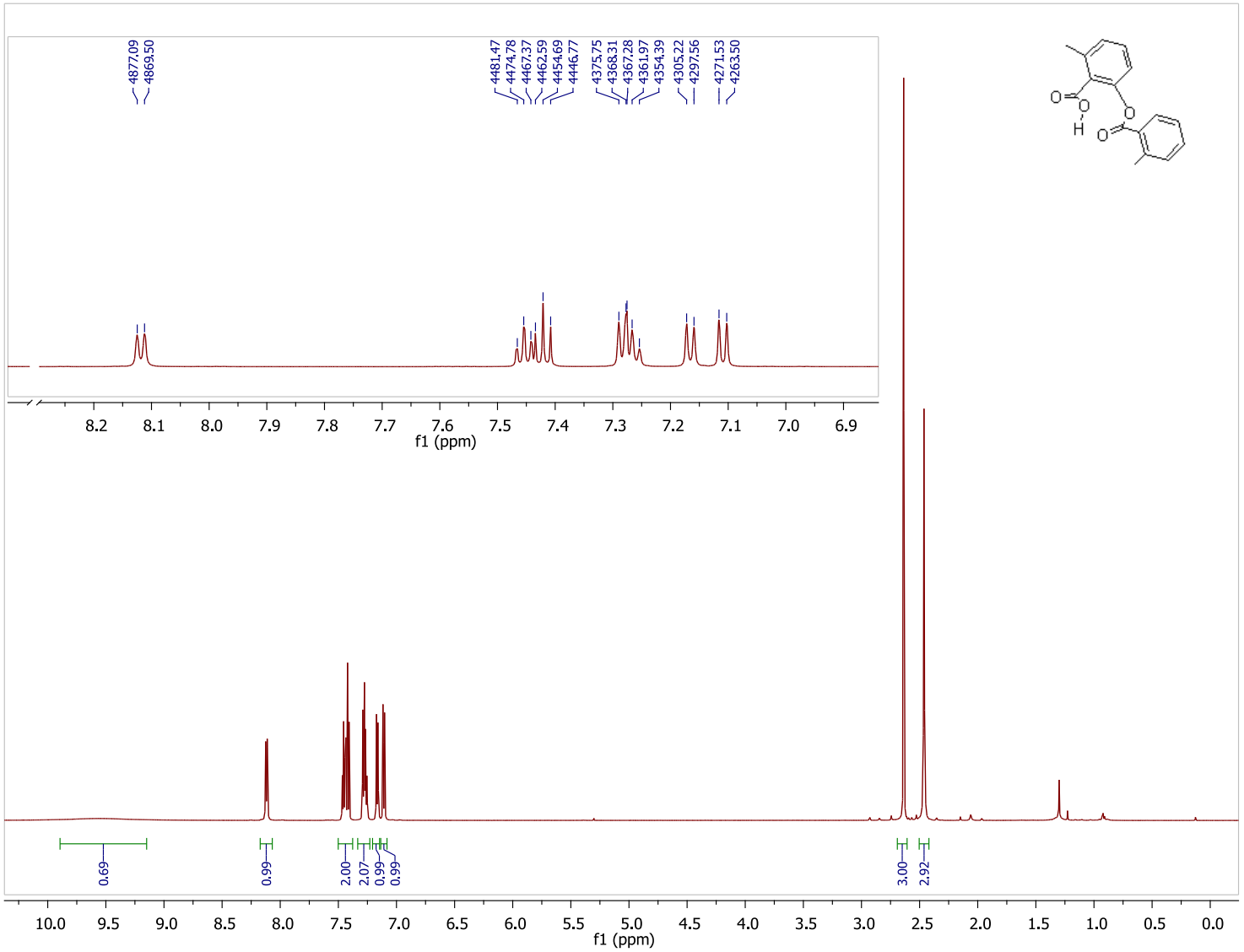
^{13}C NMR of 3-methylphenyl 2-methylbenzoate **4**



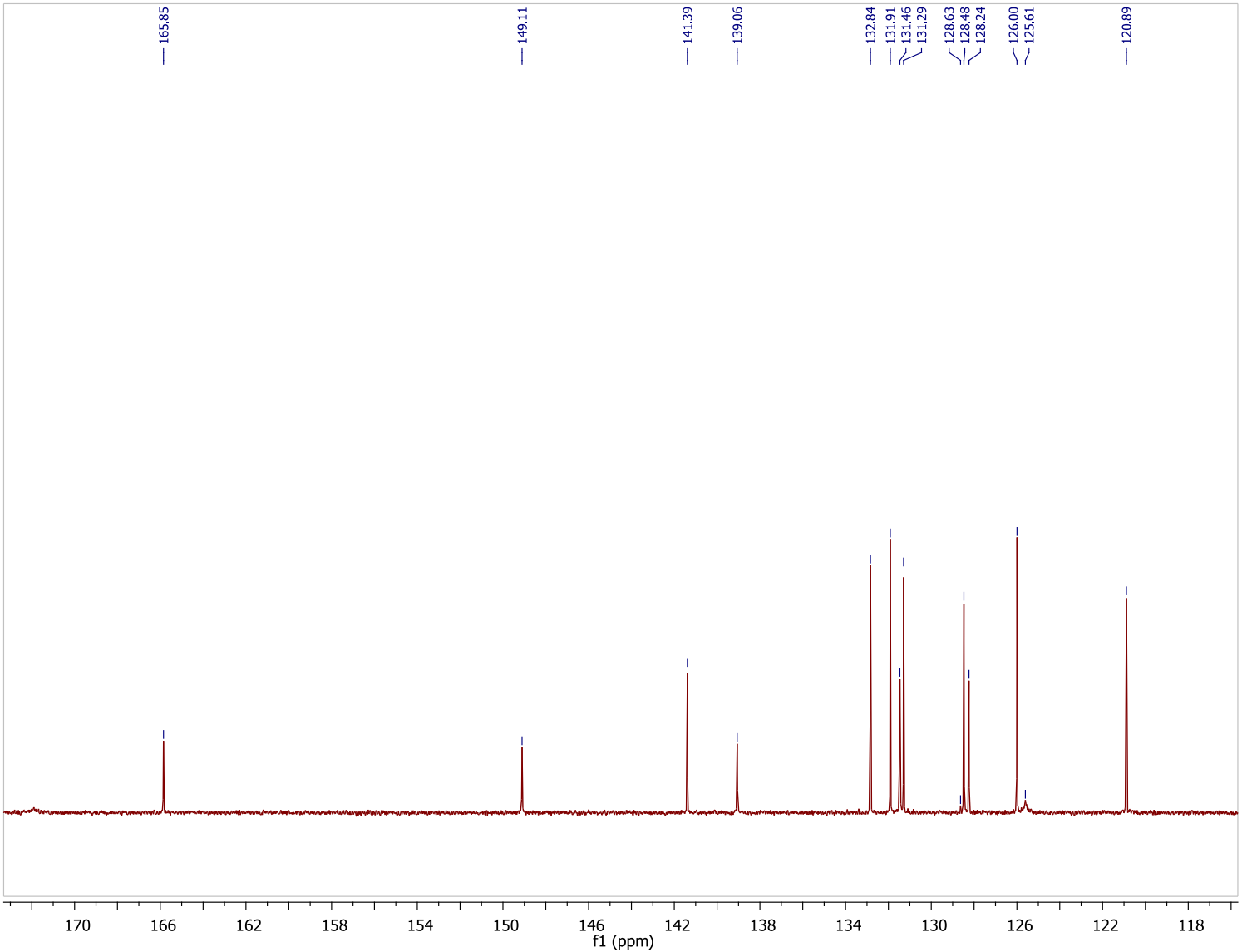
HSQC of 3-methylphenyl 2-methylbenzoate **4**



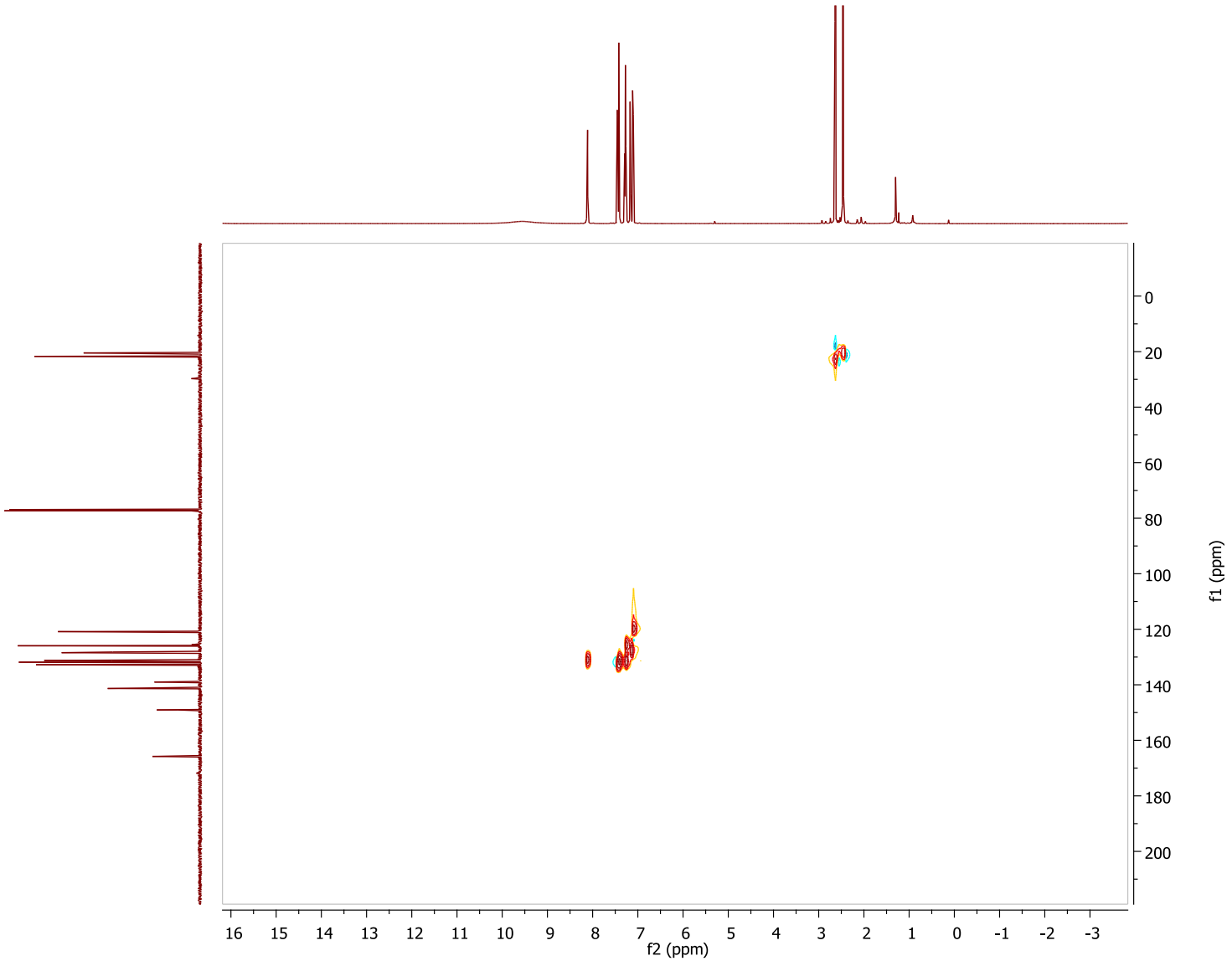
HMBC of 3-methylphenyl 2-methylbenzoate **4**



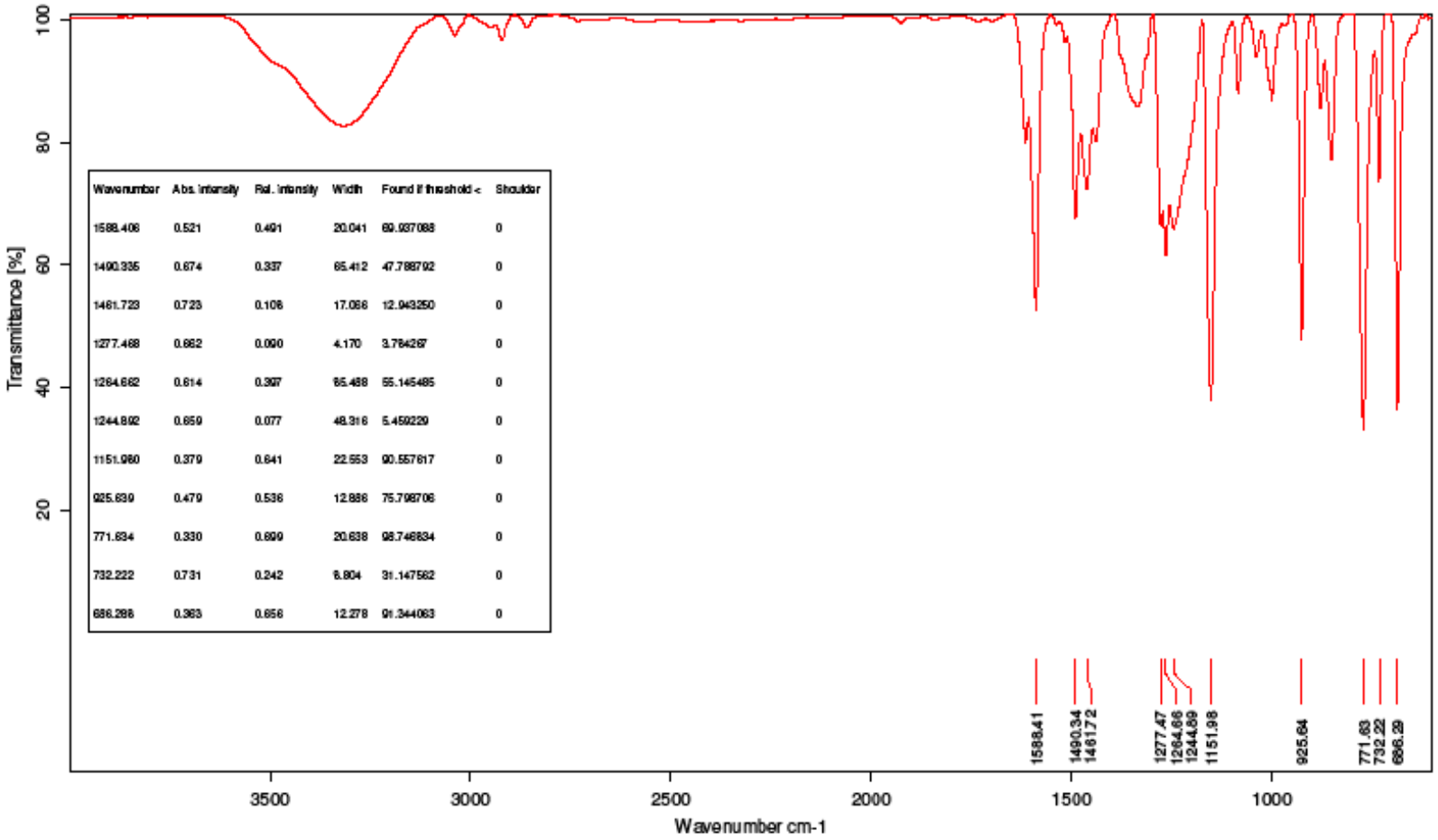
^{1}H NMR of 2-methyl-6-{[(2-methylphenyl)carbonyl]oxy}benzoic acid **3**



^{13}C NMR of 2-methyl-6-{[(2-methylphenyl)carbonyl]oxy}benzoic acid **3**

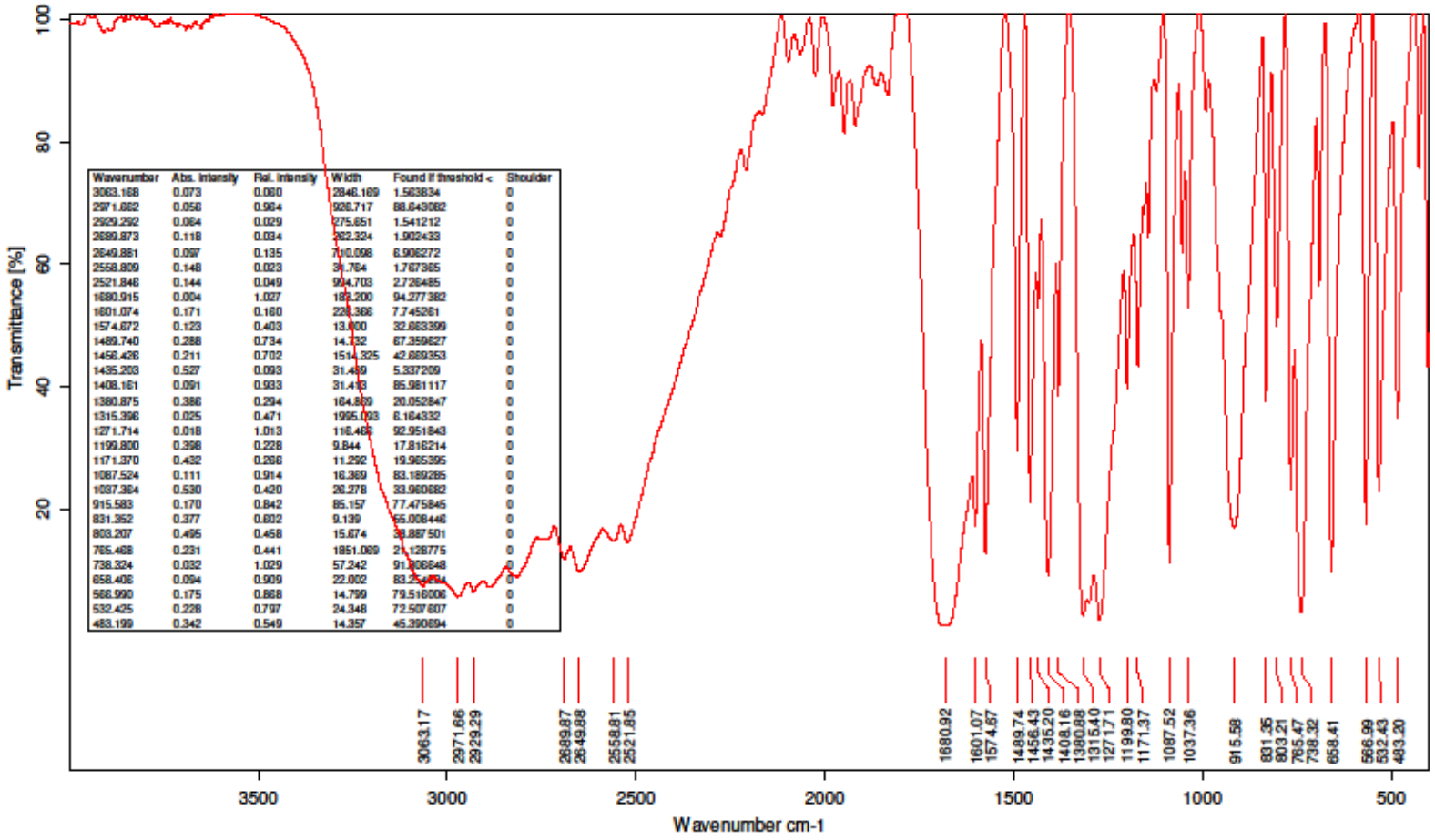


HSQC of 2-methyl-6-{[(2-methylphenyl)carbonyl]oxy}benzoic acid **3**



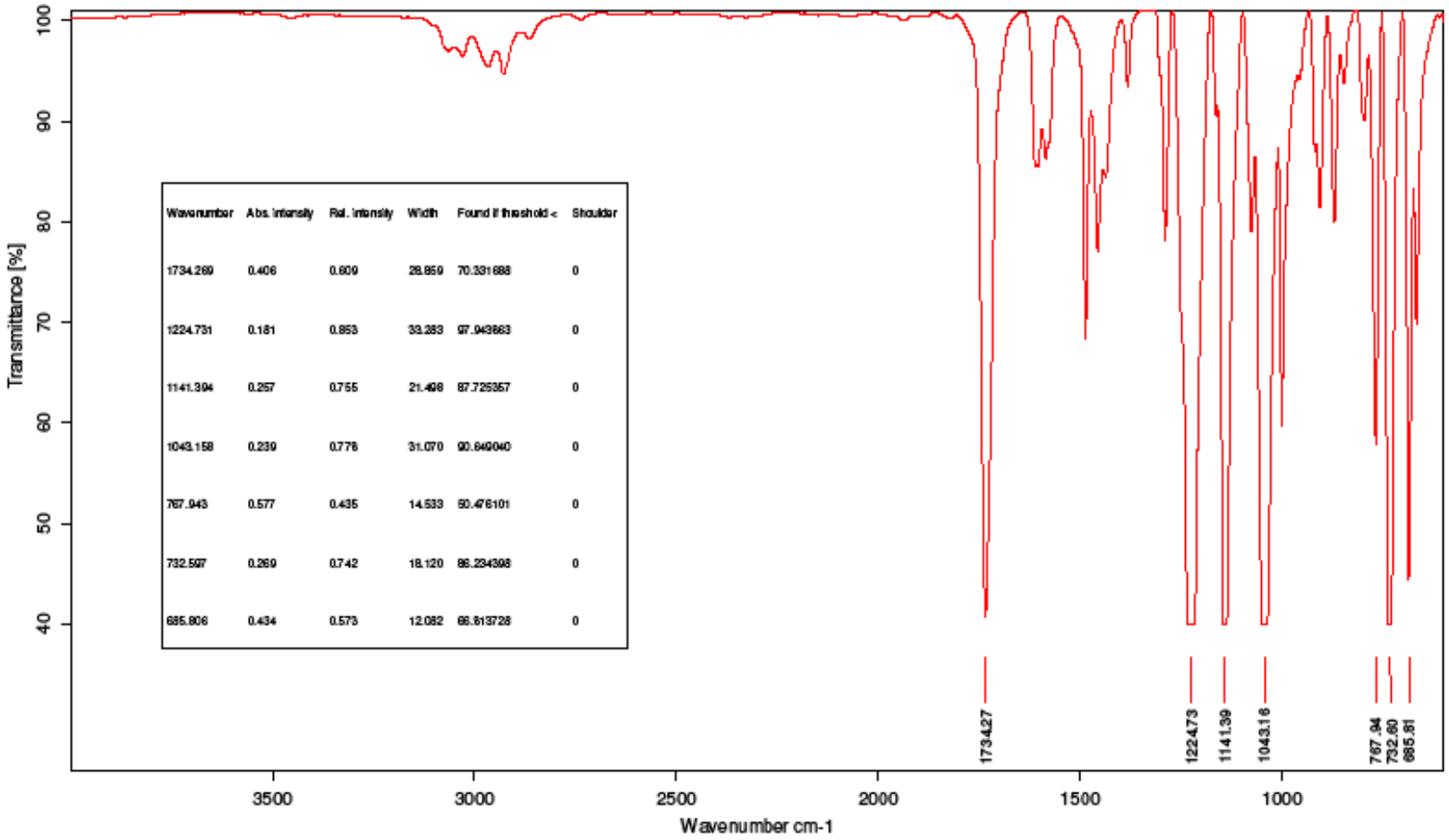
Wavenumber	Abs. Intensity	Rel. Intensity	Width	Found if threshold <	Shoulder
1588.406	0.521	0.491	20.041	69.937088	0
1490.335	0.674	0.337	65.412	47.788792	0
1461.723	0.723	0.106	17.066	12.943250	0
1277.468	0.882	0.090	4.170	3.784267	0
1264.662	0.814	0.397	65.488	55.145485	0
1244.892	0.859	0.077	48.316	5.459229	0
1151.980	0.379	0.641	22.553	90.557617	0
925.639	0.479	0.536	12.886	75.798706	0
771.634	0.330	0.899	20.638	98.746834	0
732.222	0.731	0.242	6.804	31.147582	0
686.286	0.383	0.656	12.278	91.344063	0

IR of *m*-cresol **1**



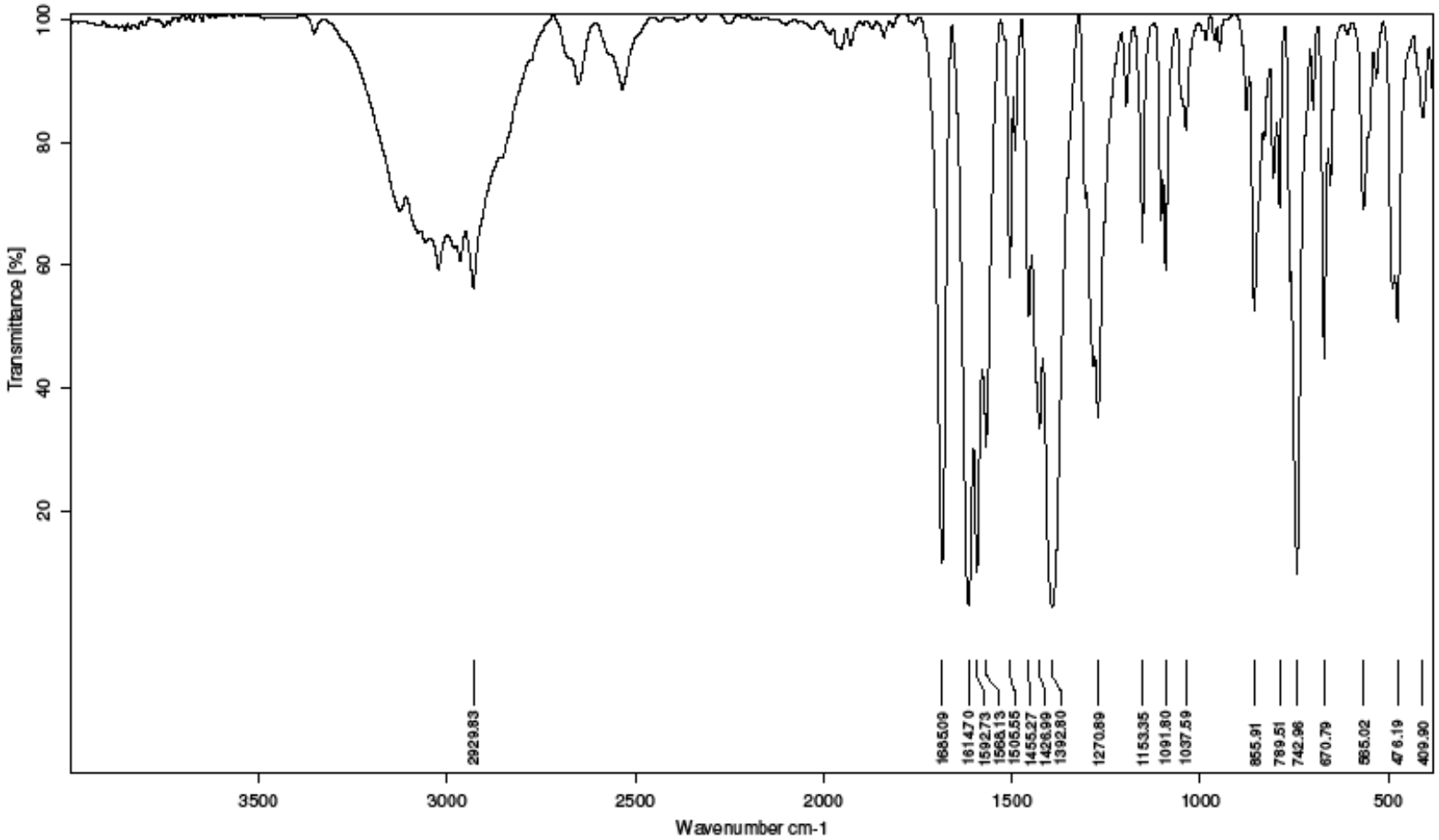
Wavenumber	Abs. Intensity	Rel. Intensity	Width	Found if threshold <	Shoulder
3063.168	0.073	0.060	2846.169	1.563834	0
2971.662	0.056	0.964	926.717	88.643082	0
2929.292	0.064	0.029	275.651	1.541212	0
2689.873	0.118	0.034	282.324	1.902433	0
2649.881	0.097	0.135	710.098	6.906272	0
2558.809	0.148	0.023	31.764	1.767365	0
2521.846	0.144	0.049	994.703	2.726485	0
1680.915	0.004	1.027	183.200	94.277382	0
1601.074	0.171	0.160	226.368	7.745261	0
1574.672	0.123	0.403	13.900	32.883399	0
1489.740	0.288	0.734	14.732	67.359627	0
1456.428	0.211	0.702	1514.325	42.689353	0
1435.203	0.527	0.093	31.469	5.337209	0
1408.161	0.091	0.933	31.413	85.981117	0
1380.875	0.386	0.294	164.859	20.052847	0
1315.396	0.025	0.471	1995.093	6.164332	0
1271.714	0.018	1.013	116.483	92.951843	0
1199.800	0.398	0.228	9.844	17.818214	0
1171.370	0.432	0.268	11.292	19.965395	0
1087.524	0.111	0.914	16.369	83.189285	0
1037.364	0.530	0.420	26.278	33.960682	0
915.583	0.170	0.842	85.157	77.475845	0
831.352	0.377	0.602	9.139	55.008446	0
803.207	0.495	0.458	15.674	38.887501	0
765.468	0.231	0.441	1851.069	21.128775	0
738.324	0.032	1.029	57.242	91.206648	0
658.408	0.094	0.909	22.002	83.254784	0
566.990	0.175	0.868	14.799	79.518006	0
532.425	0.228	0.797	24.348	72.507607	0
483.199	0.342	0.549	14.357	45.390694	0

IR of *o*-toluic acid **2**

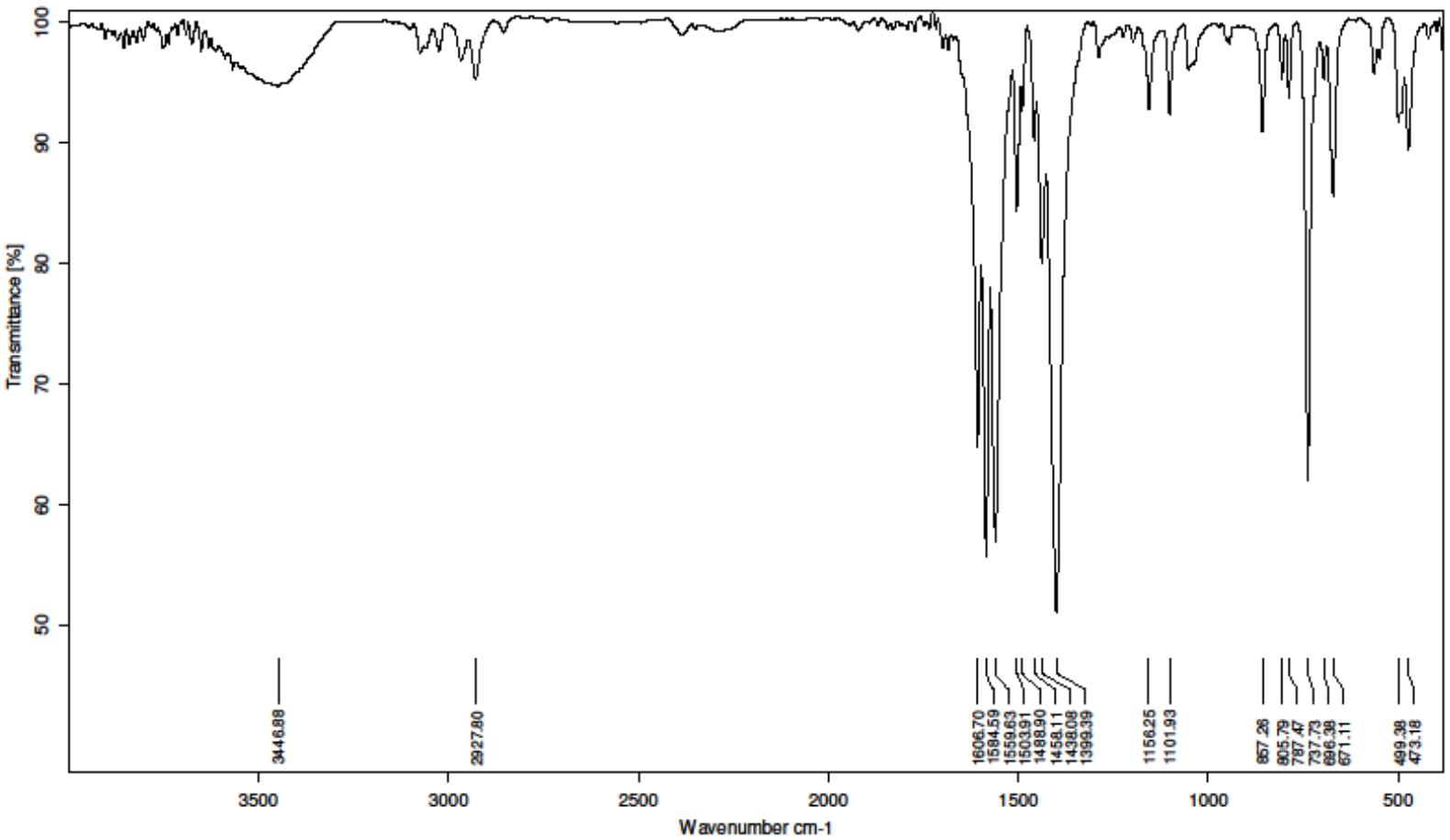


Wavenumber	Abs. Intensity	Rel. Intensity	Width	Found if threshold <	Shoulder
1734.269	0.406	0.609	28.859	70.331688	0
1224.731	0.181	0.853	33.283	97.043663	0
1141.394	0.257	0.755	21.498	87.725357	0
1043.158	0.239	0.776	31.070	90.849040	0
767.943	0.577	0.435	14.533	50.476101	0
732.597	0.269	0.742	18.120	86.234398	0
685.806	0.434	0.573	12.082	66.813728	0

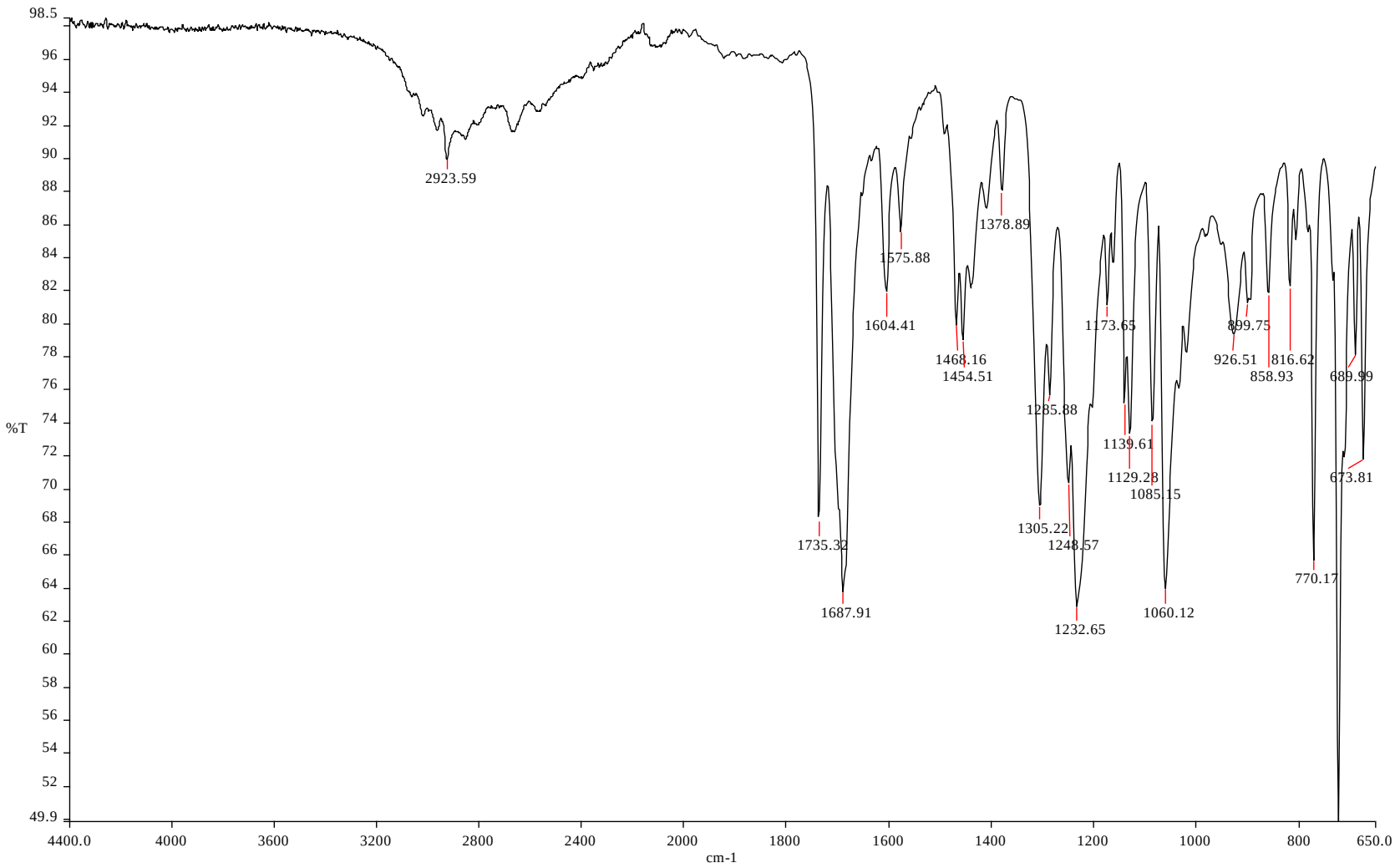
IR of 3-methylphenyl 2-methylbenzoate **4**



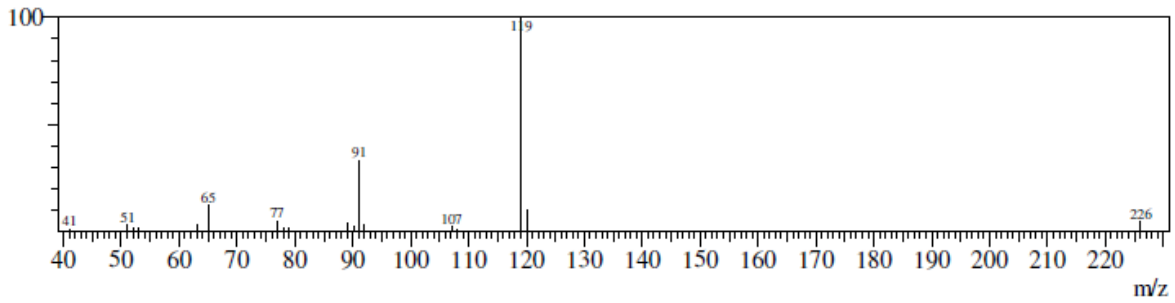
IR of tetrakis(μ_2-2-methylbenzoato)bis(2-methylbenzoic acid)copper(II) **5**



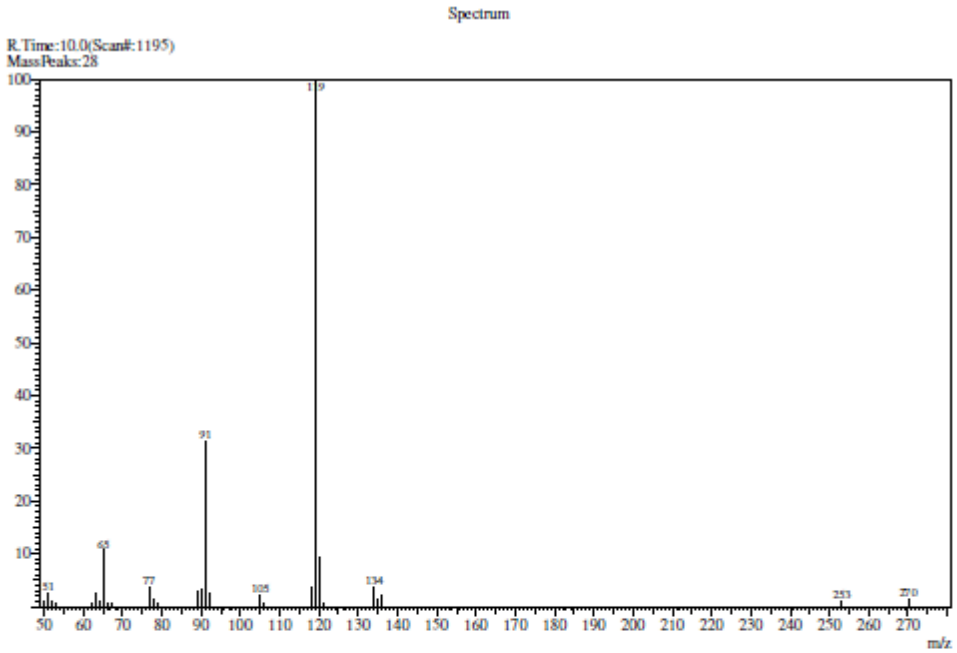
IR of tetrakis(μ_2-2-methylbenzoato) copper(II) **6**



IR of 2-methyl-6-{[(2-methylphenyl)carbonyl]oxy}benzoic acid **3**



Mass spectrum of 3-methylphenyl 2-methylbenzoate **4**



Mass Table
R.Time:10.0(Scan#:1195)
MassPeaks:28

#	m/z	Abs. Int.	Rel. Int.
1	50.05	19698	0.85
2	51.05	59297	2.56
3	52.05	25939	1.12
4	53.05	17812	0.77
5	62.05	12501	0.54
6	63.00	52938	2.29
7	64.05	19457	0.84
8	65.05	246276	10.65
9	66.05	17056	0.74
10	67.05	9204	0.40
11	77.05	83975	3.63
12	78.05	35738	1.54
13	79.05	16865	0.73
14	89.05	69694	3.01
15	90.10	79047	3.42
16	91.10	724305	31.31
17	92.05	58992	2.55
18	105.05	46788	2.02
19	106.05	17230	0.74
20	118.15	84204	3.64
21	119.10	2313482	100.00
22	120.10	215420	9.31

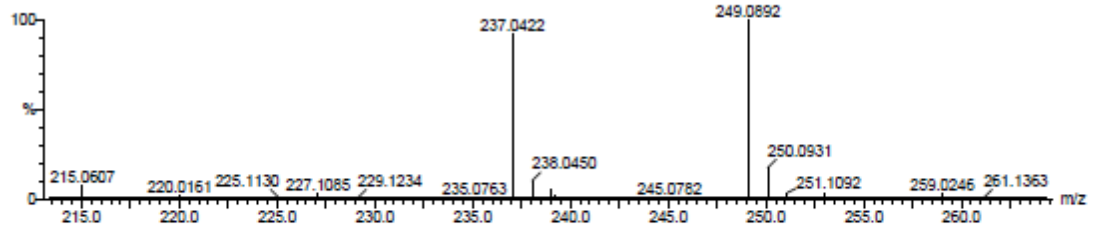
Mass spectrum of 2-methyl-6-{[(2-methylphenyl)carbonyl]oxy}benzoic acid **3**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions
17 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 10-20 H: 10-20 O: 0-10 Na: 0-1
UFS
SCA 3,2-Benzoate 85 2 (0.017) Cm (2:59)

TOF MS ES+
2.17e+005



Minimum:				-1.5			
Maximum:		5.0	5.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
249.0892	249.0891	0.1	0.4	8.5	692.4	0.0	C15 H14 O2 Na

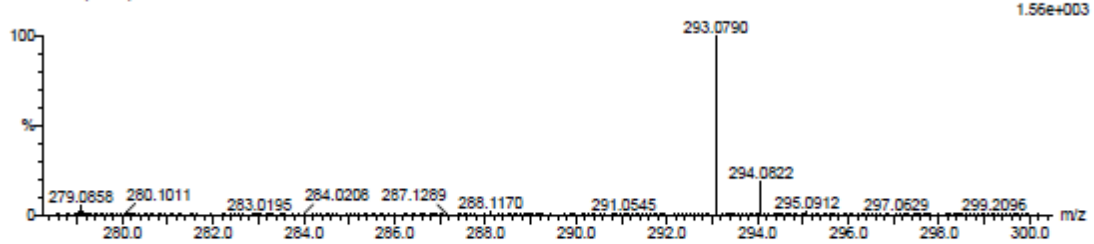
HR-MS of 3-methylphenyl 2-methylbenzoate **4**

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 5.0 PPM / DBE: min = -1.5, max = 50.0
Element prediction: Off
Number of isotope peaks used for i-FIT = 3

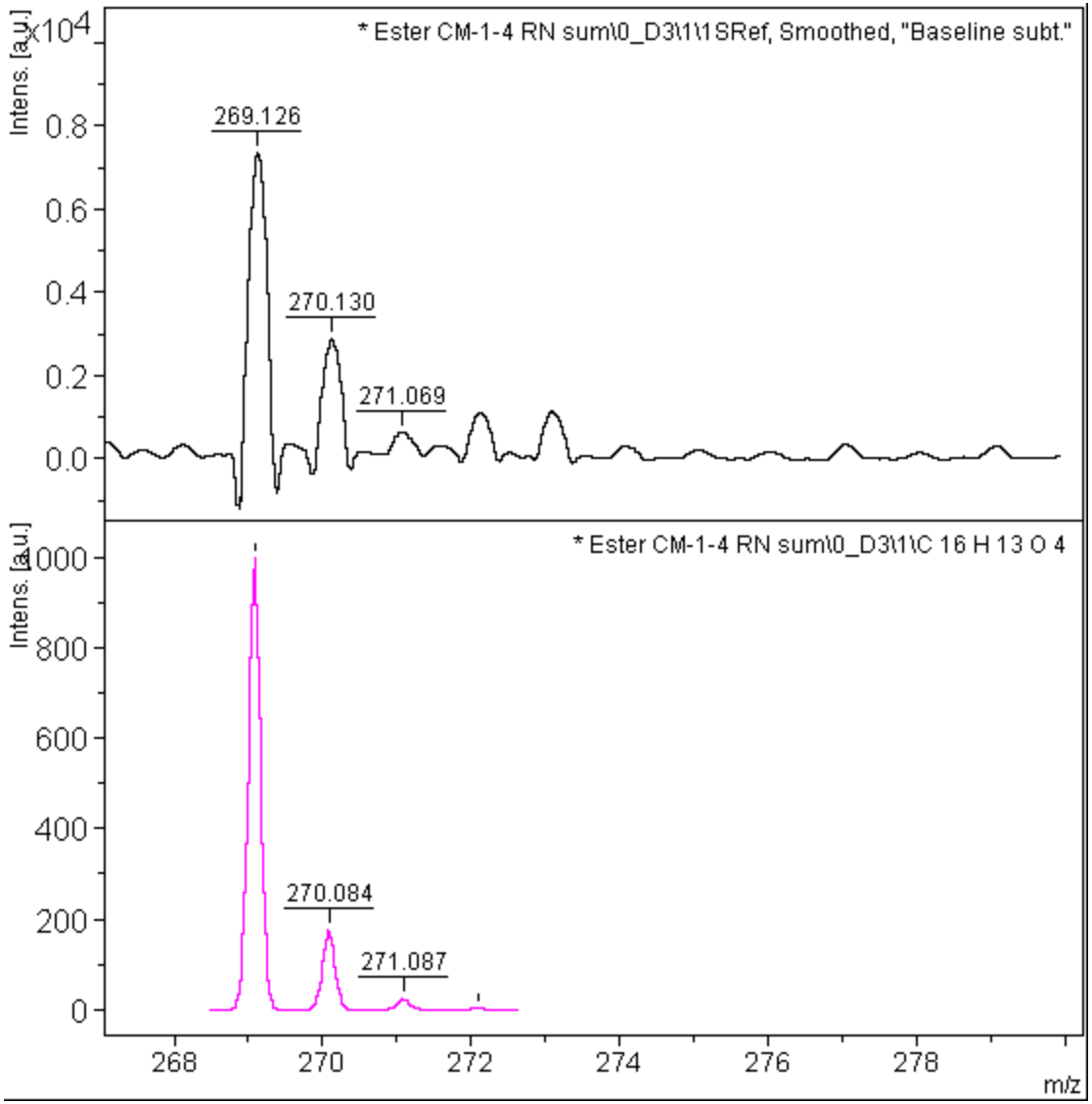
Monoisotopic Mass, Even Electron Ions
7 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)
Elements Used:
C: 15-20 H: 10-20 O: 0-5 Na: 0-1
Cherlene Marais
CM-1-4 12 (0.188)

TOF MS ES+
1.56e+003

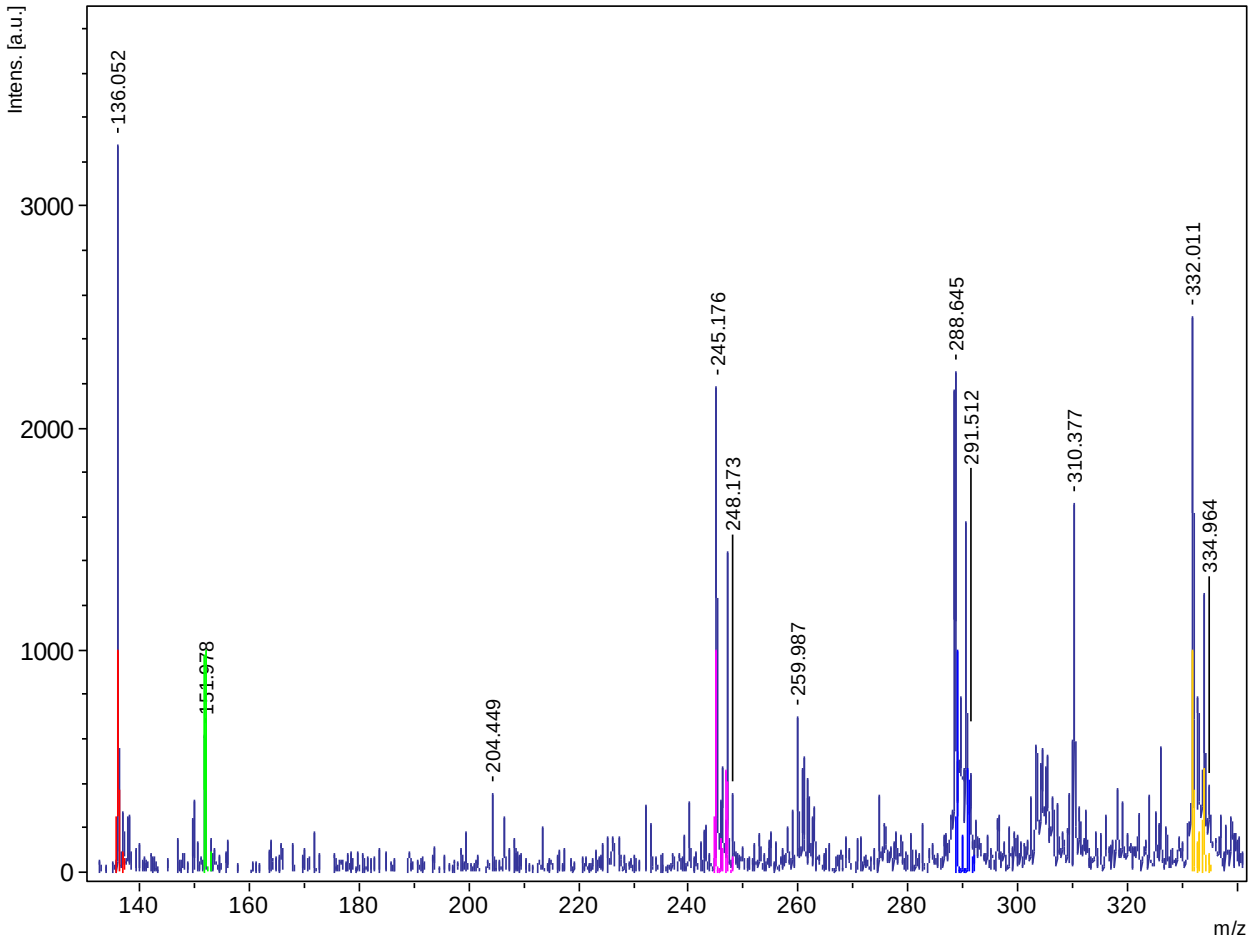


Minimum:				-1.5			
Maximum:		5.0	5.0	50.0			
Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	i-FIT (Norm)	Formula
293.0790	293.0790	0.0	0.0	9.5	111.0	0.0	C16 H14 O4 Na

HR-MS of 2-methyl-6-{[(2-methylphenyl)carbonyl]oxy}benzoic acid **3**



MALDI-TOF MS (-ve) of 2-methyl-6-{[(2-methylphenyl)carbonyl]oxy}benzoic acid **3**



MALDI-TOF MS analysis (-ve) of the reaction mixture after the second DSC cycle on tetrakis(μ_2-2-methylbenzoato)bis(2-methylbenzoic acid)copper(II) **5**