

Supporting information

Evaluation of sodium acetate trihydrate-urea DES as a benign reaction media for the Biginelli reaction. Unexpected synthesis of methylenebis(3-hydroxy-5,5-dimethylcyclohex-2-enones), hexahydroxanthene-1,8-diones and hexahydroacridine-1,8-diones

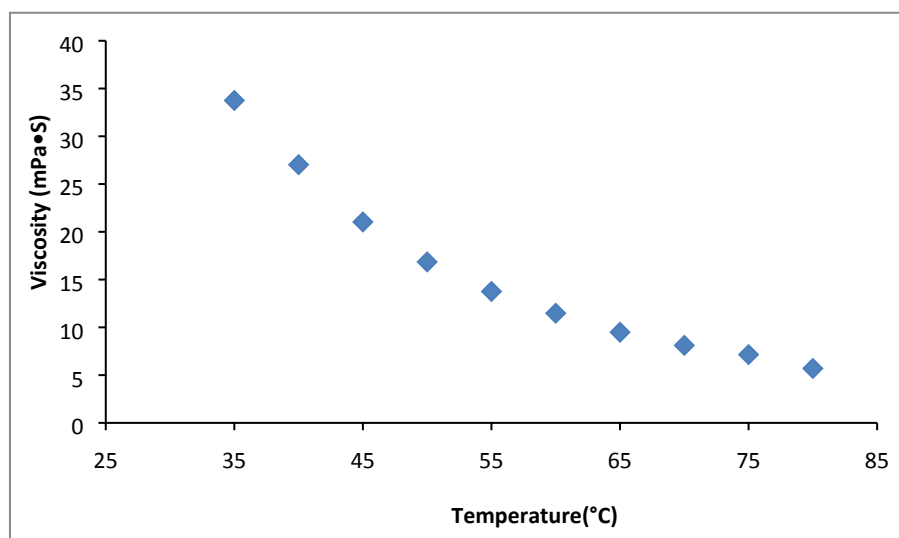
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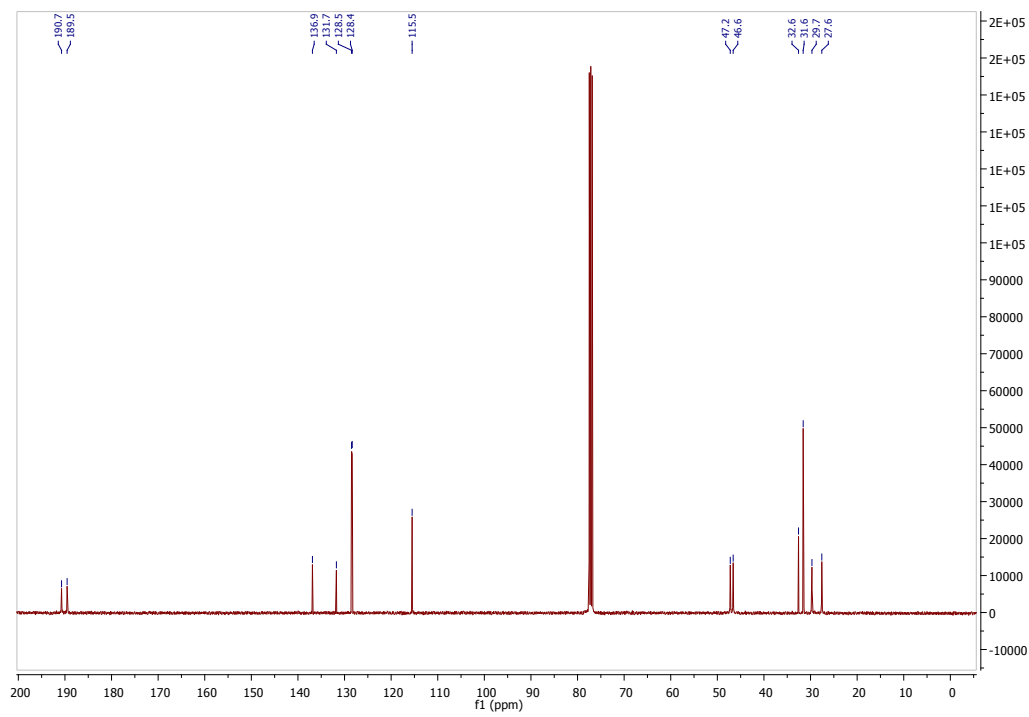
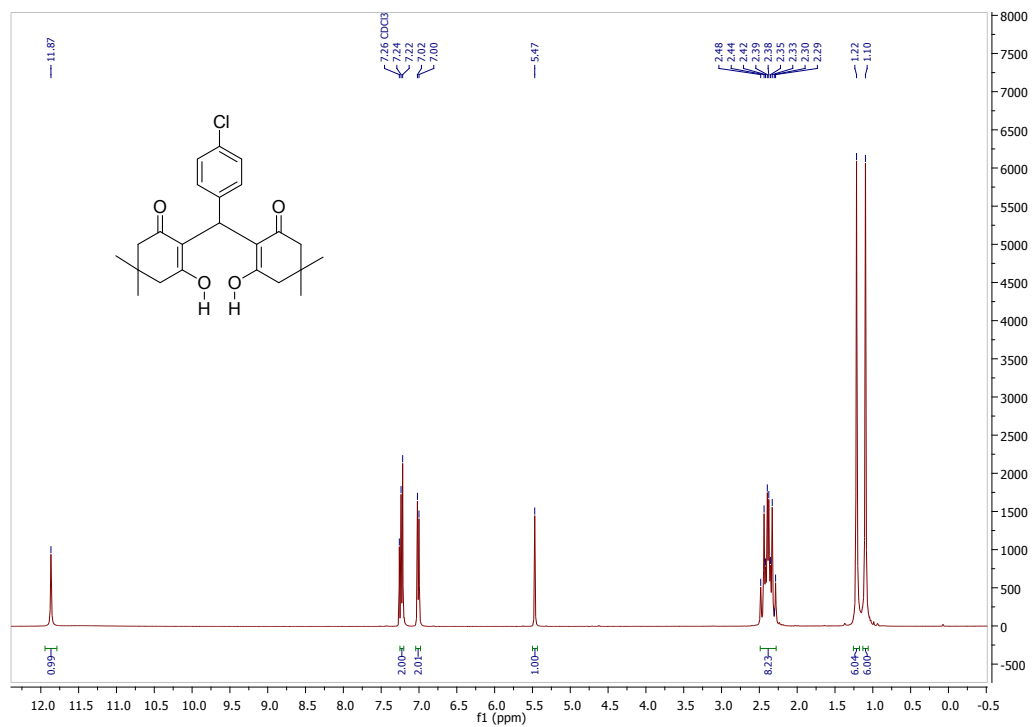
Table of contents

Viscosity of the DES as a function of temperature	S2
¹ H and ¹³ C spectra for 6a-6l and organic elemental analysis for 6a , 6k and 6l	S3-S17
¹ H and ¹³ C spectra for 5a-5l and organic elemental analysis for 5a , 5k and 5l	S18-S32
References	S33

Viscosity of the DES as a function of temperature

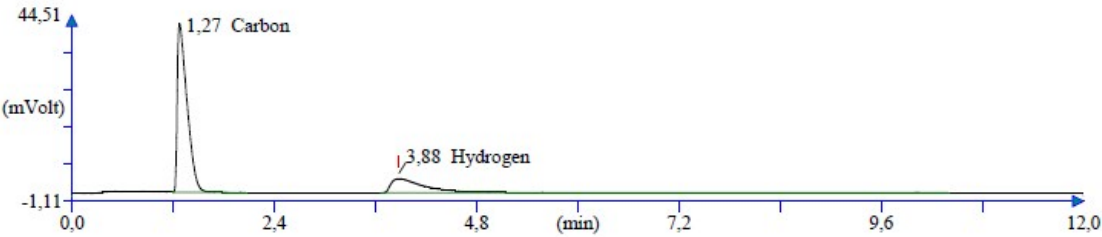


^1H and ^{13}C spectra for 2,2'-((4-chlorophenyl)methylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6a**).¹

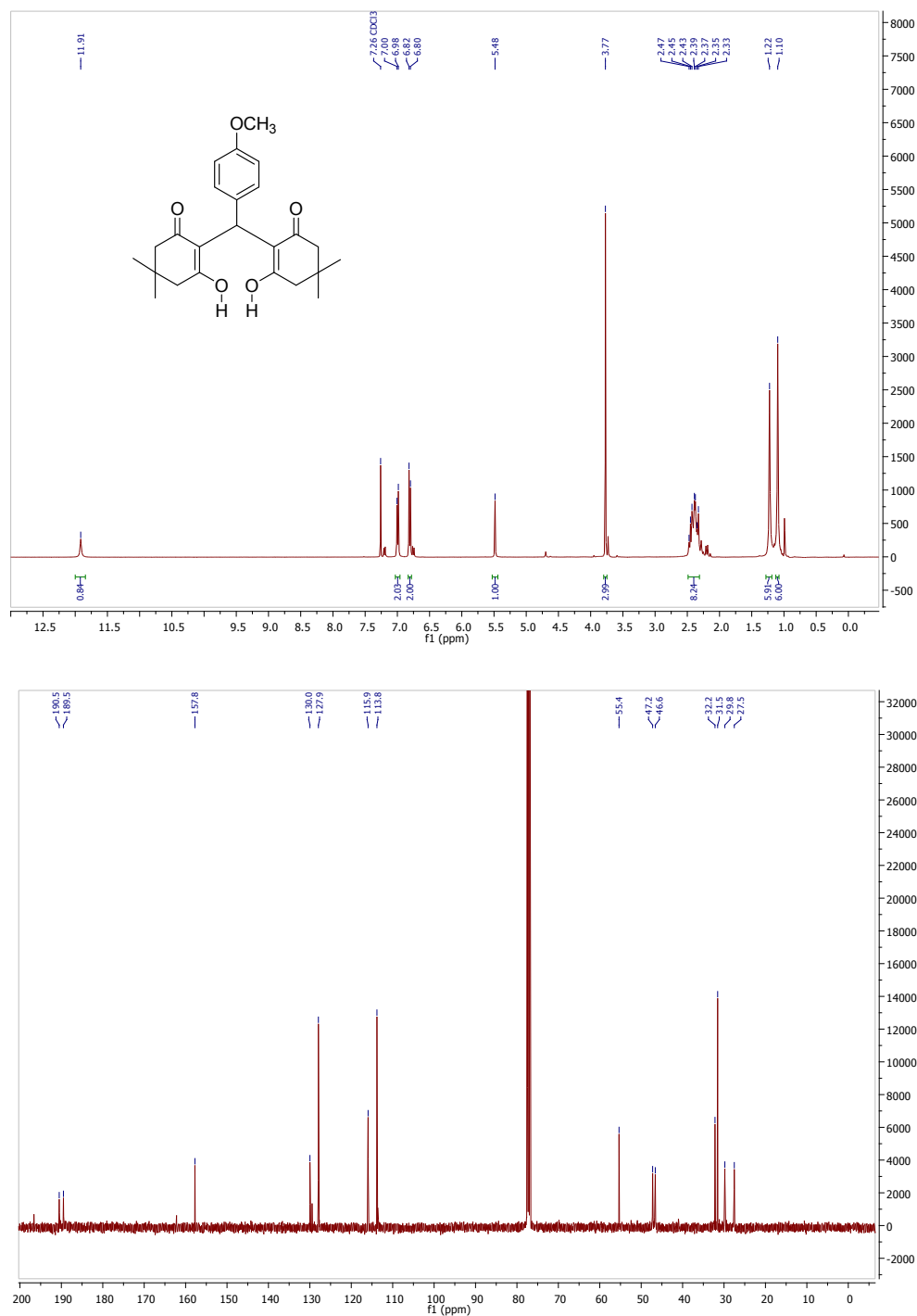


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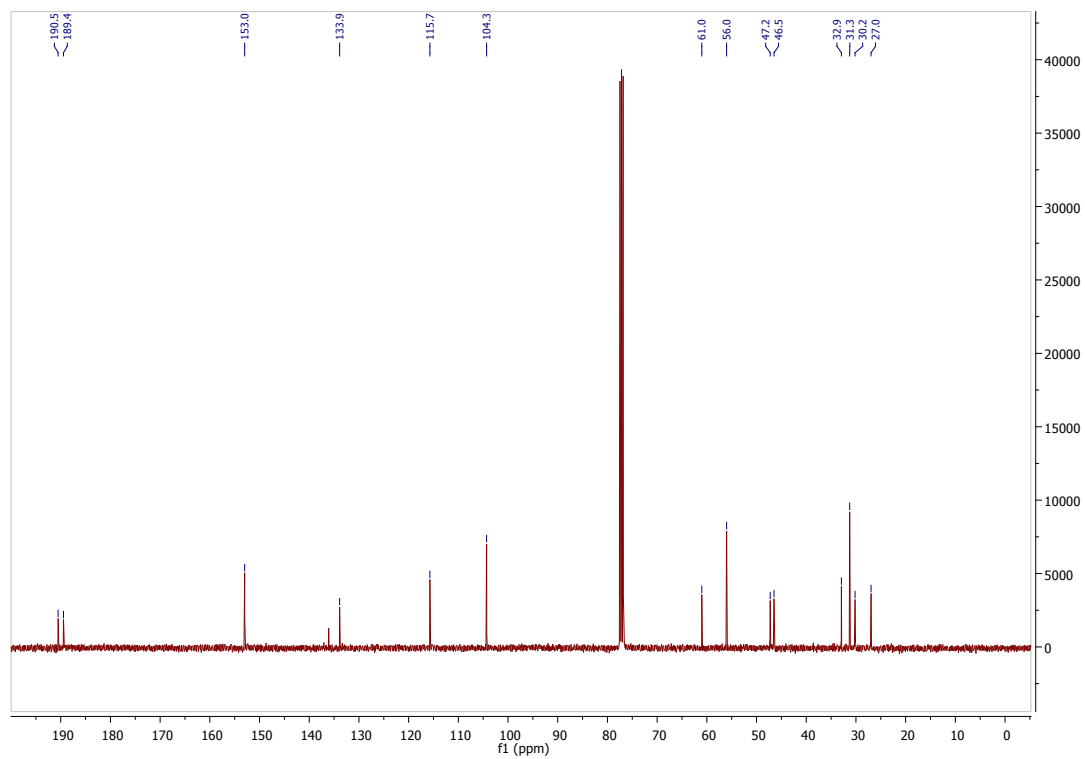
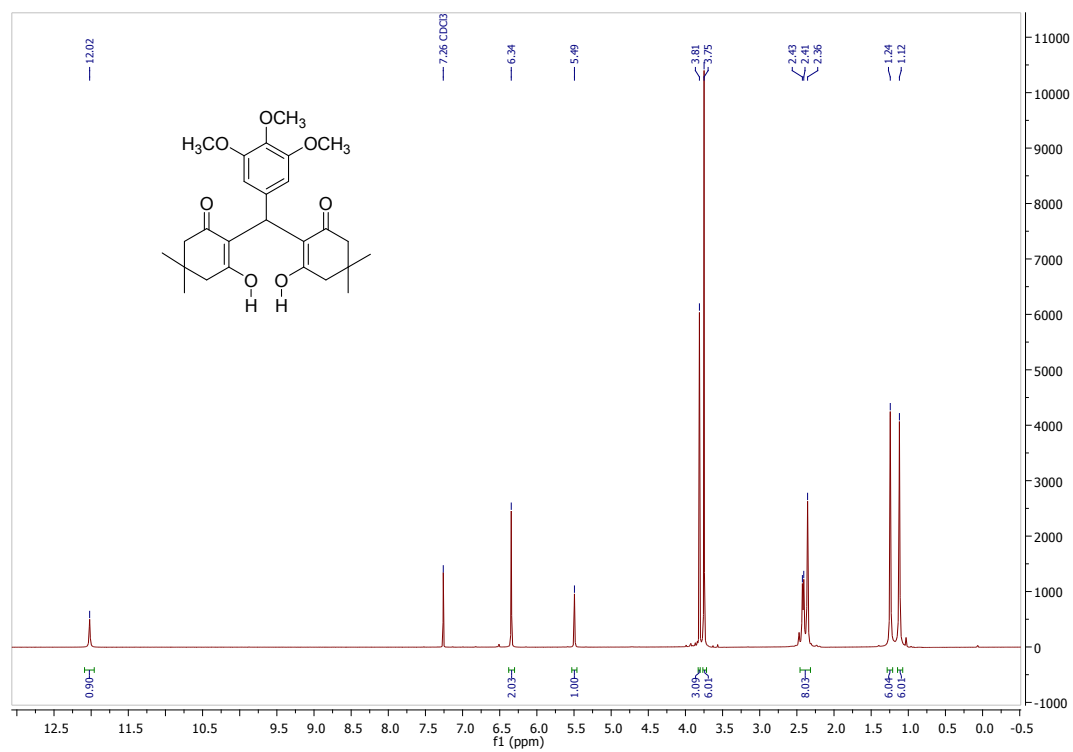
Element Name	Ret. Time	Area	BC	Area ratio	K factor
Carbon	68.8383	77	3235806	mi	1.000000 .464422E+07
Hydrogen	6.7621	233	1014477	mi	3.189630 .147901E+08
Totals	75.6004		4250283		



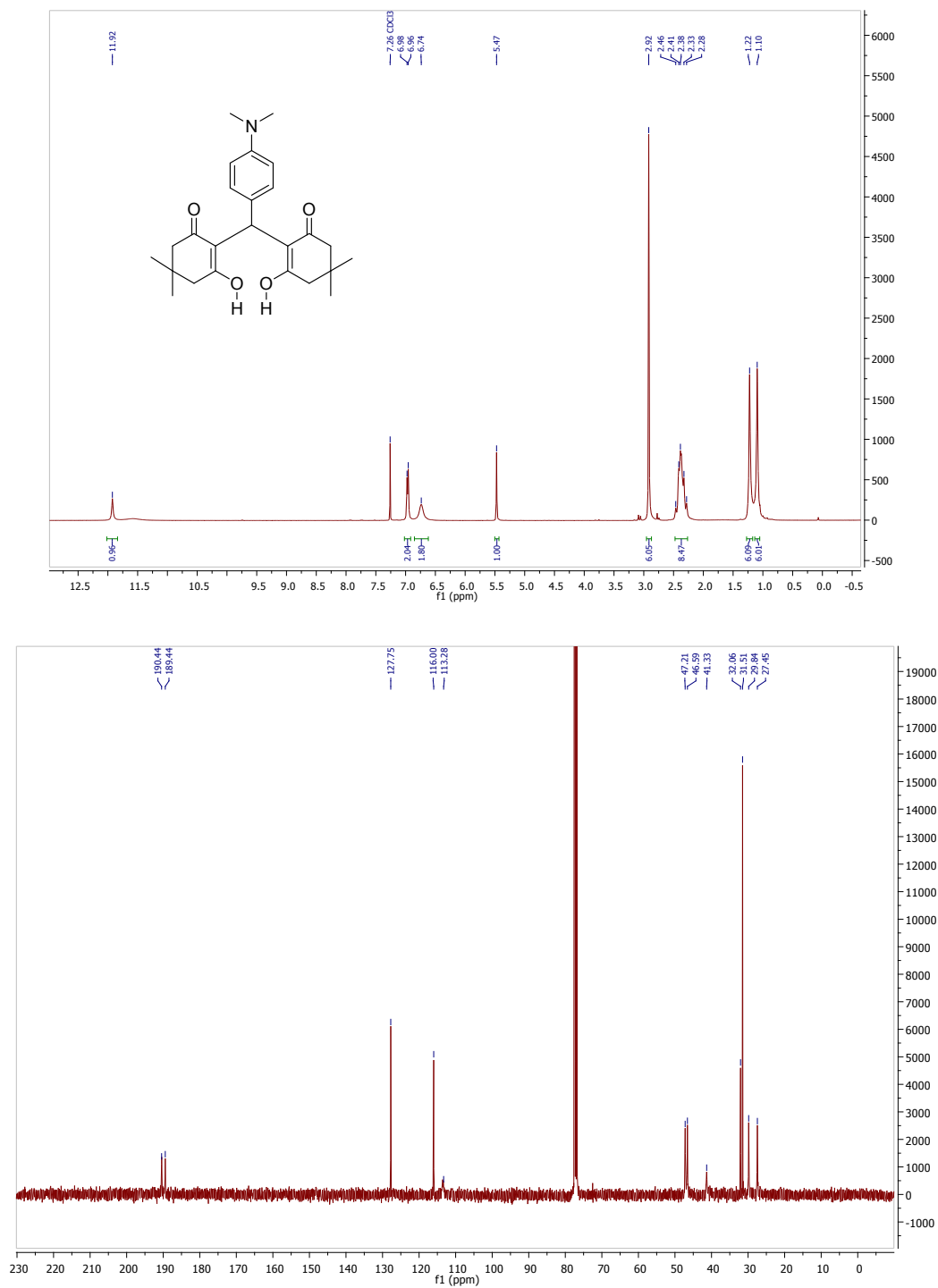
^1H and ^{13}C spectra for 2,2'-((4-methoxyphenyl)methylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6b**).²



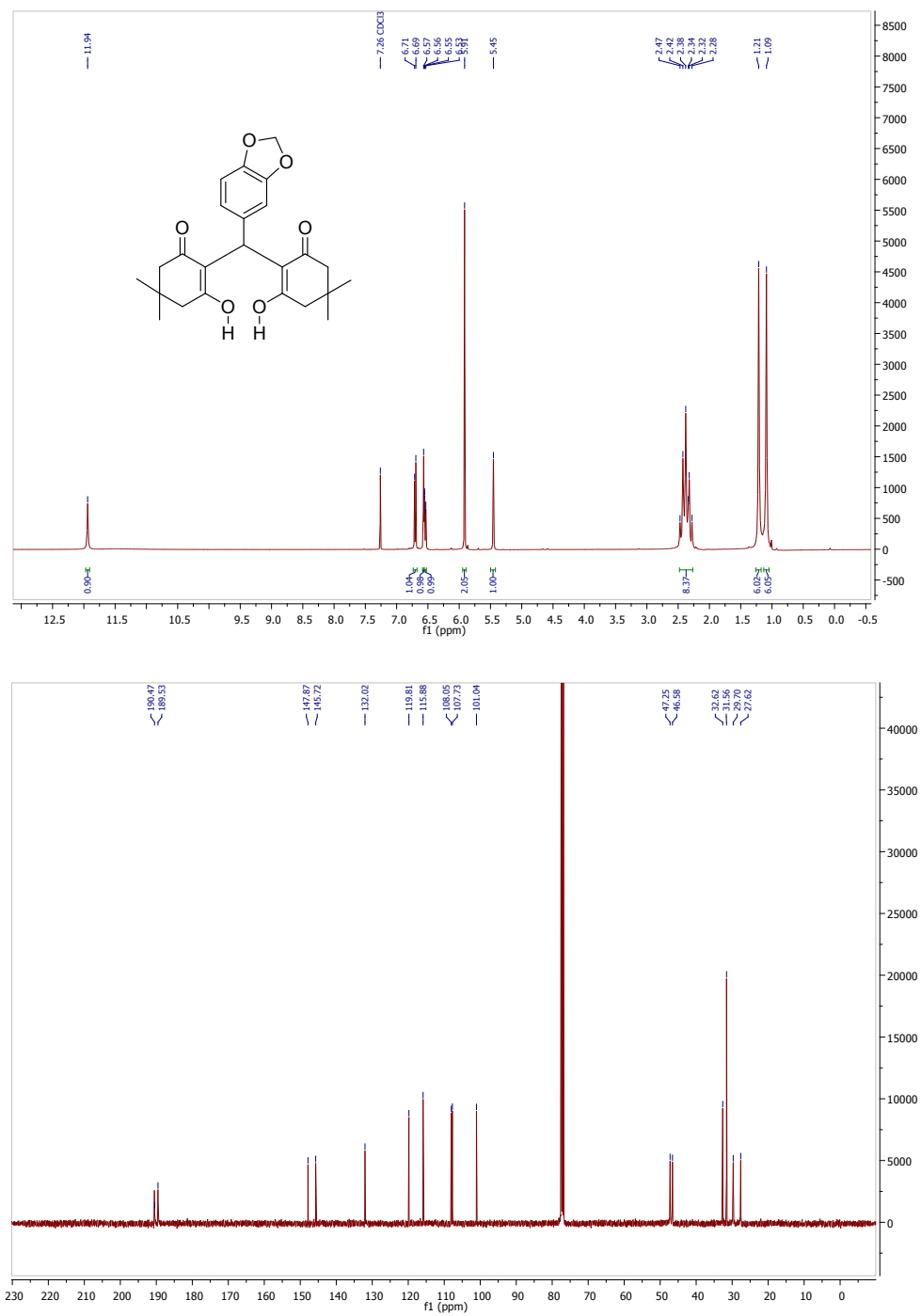
^1H and ^{13}C spectra for 2,2'-((3,4,5-trimethoxyphenyl)methylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6c**).³



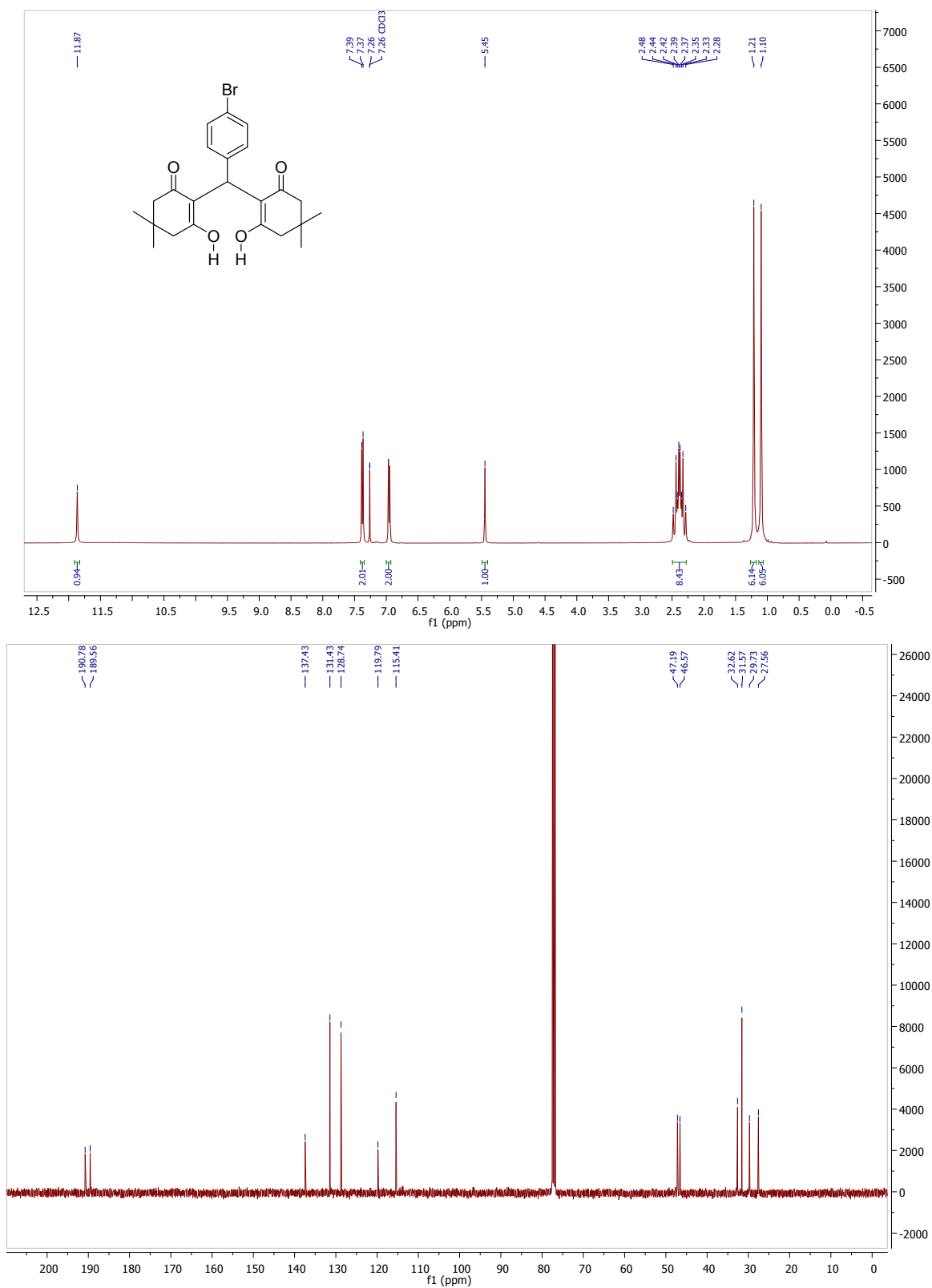
^1H and ^{13}C spectra for 2,2'-((4-(dimethylamino)phenyl)methylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6d**).³



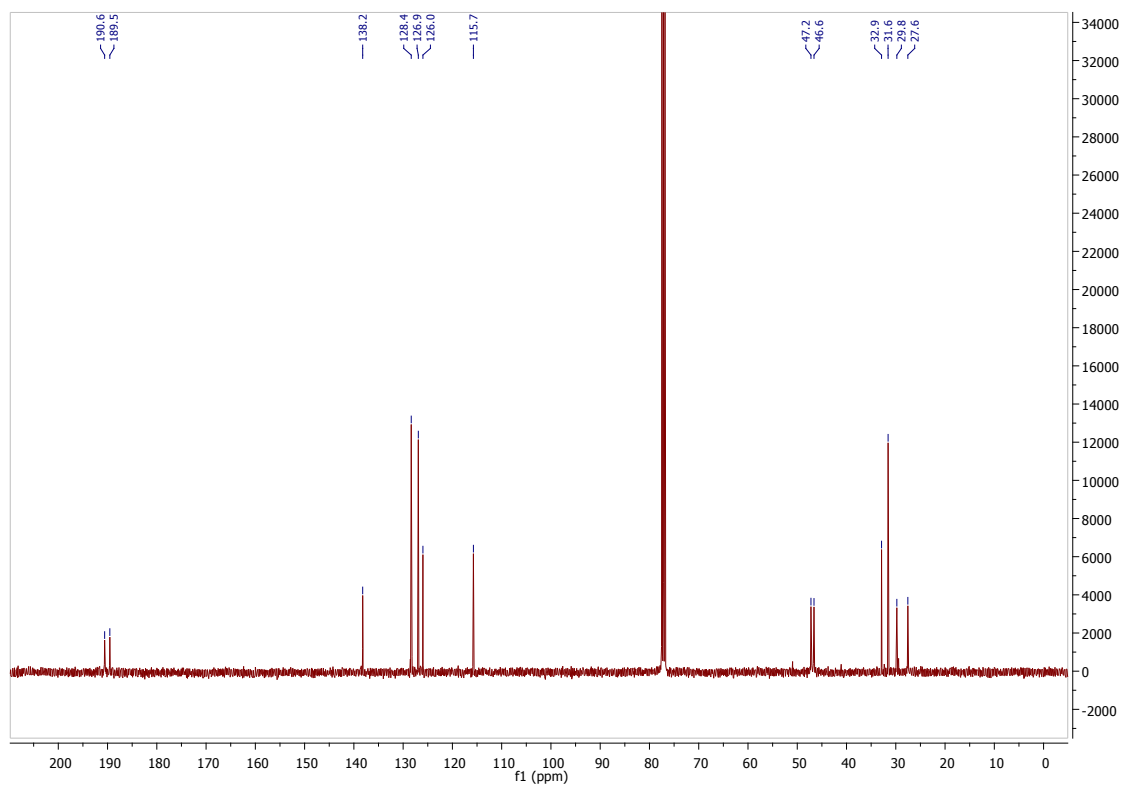
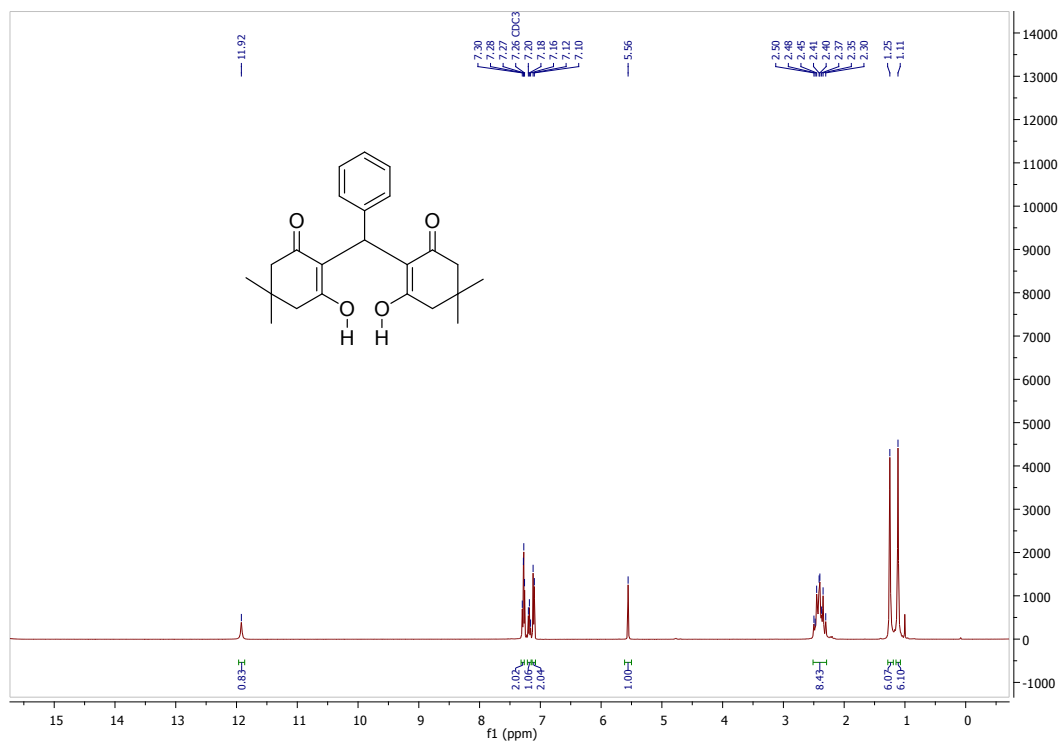
^1H and ^{13}C spectra for 2,2'-(benzo[d][1,3]dioxol-5-ylmethylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6e**).⁴



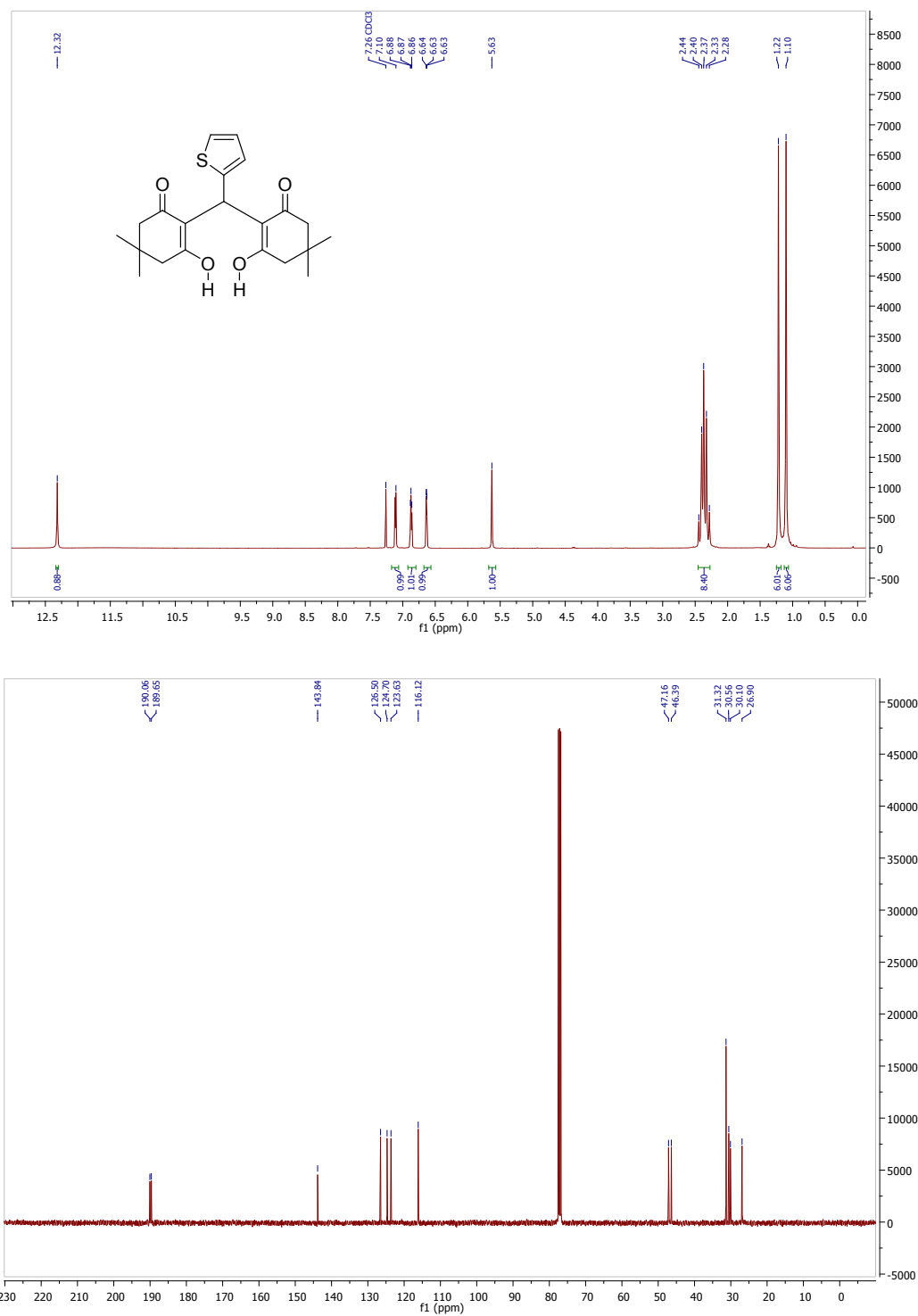
^1H and ^{13}C spectra for 2,2'-((4-bromophenyl)methylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6f**).³



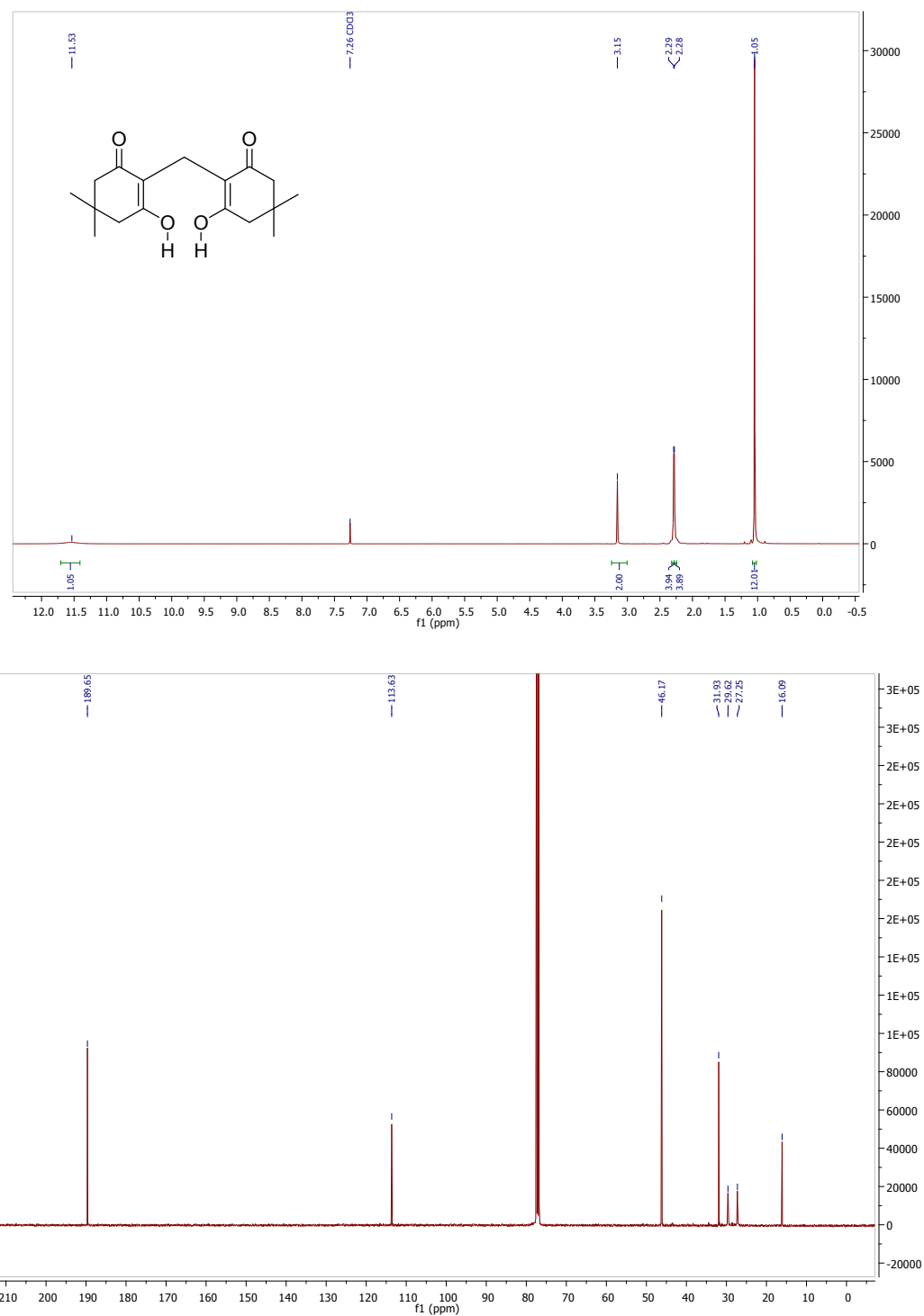
^1H and ^{13}C spectra for 2,2'-(phenylmethylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6g**).²



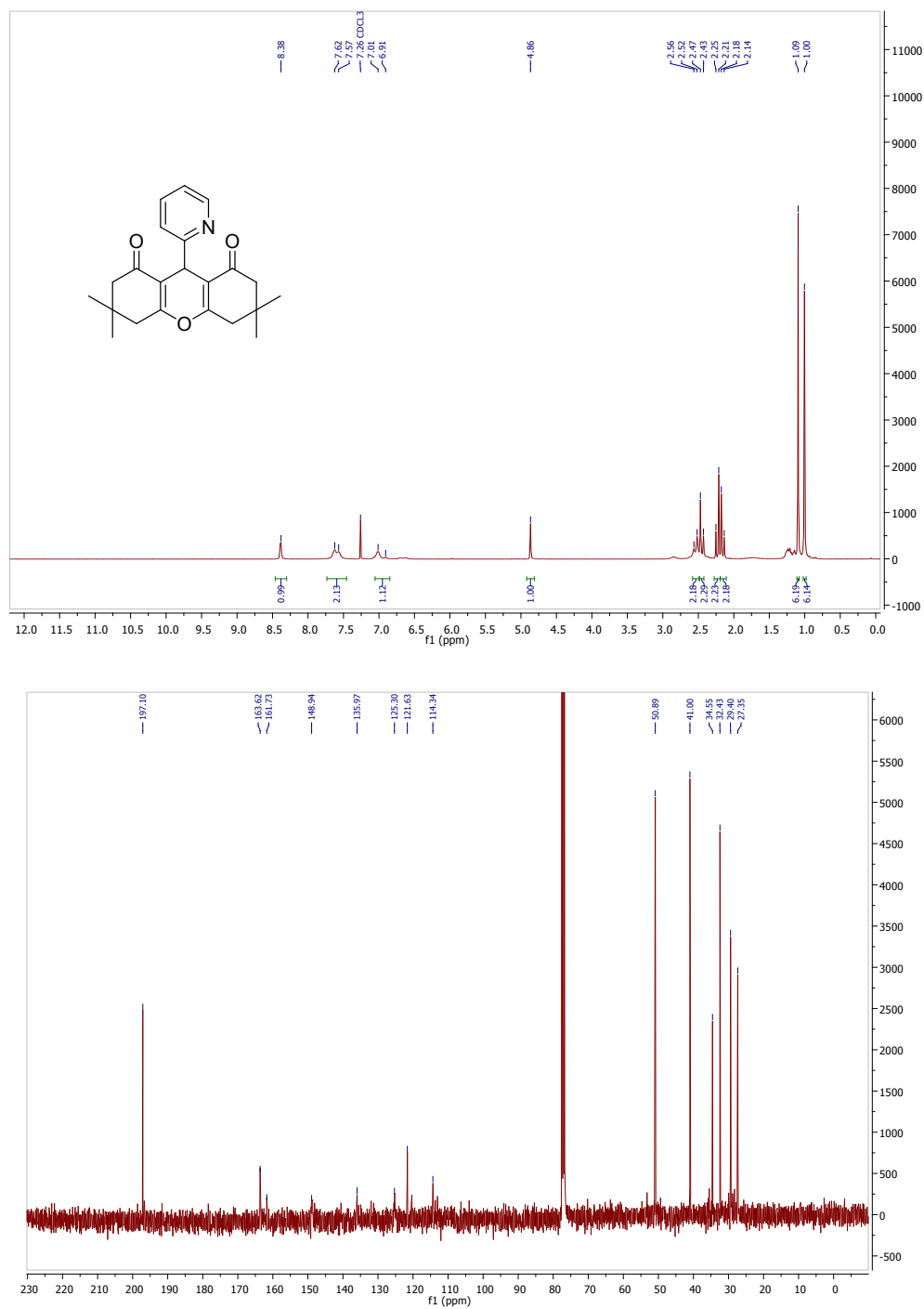
^1H and ^{13}C spectra for 2,2'-(thiophen-2-ylmethylene)bis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6h**).⁵



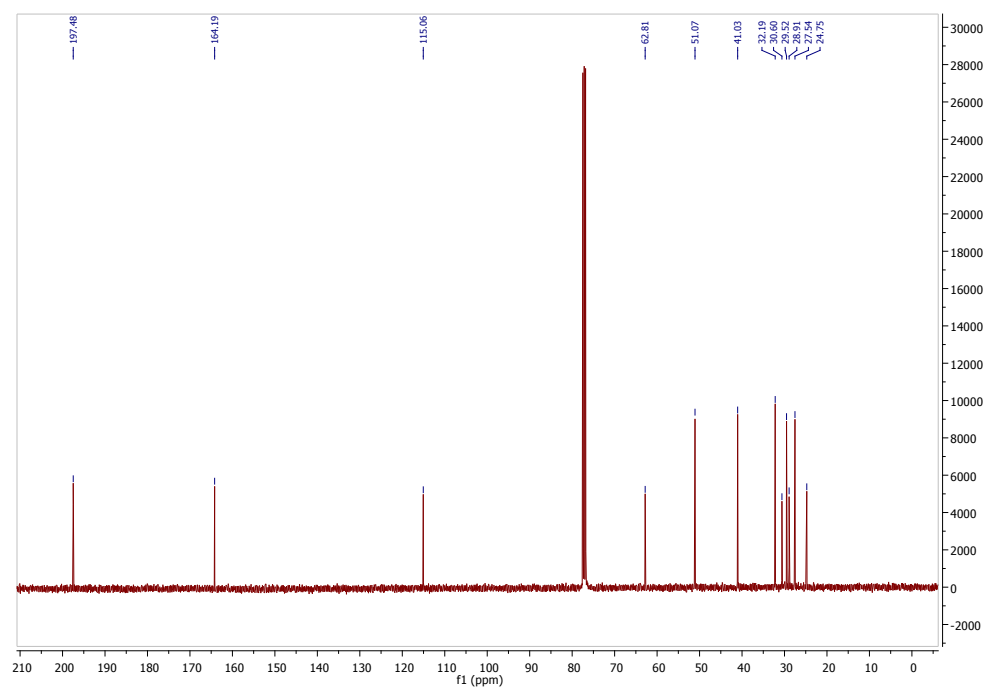
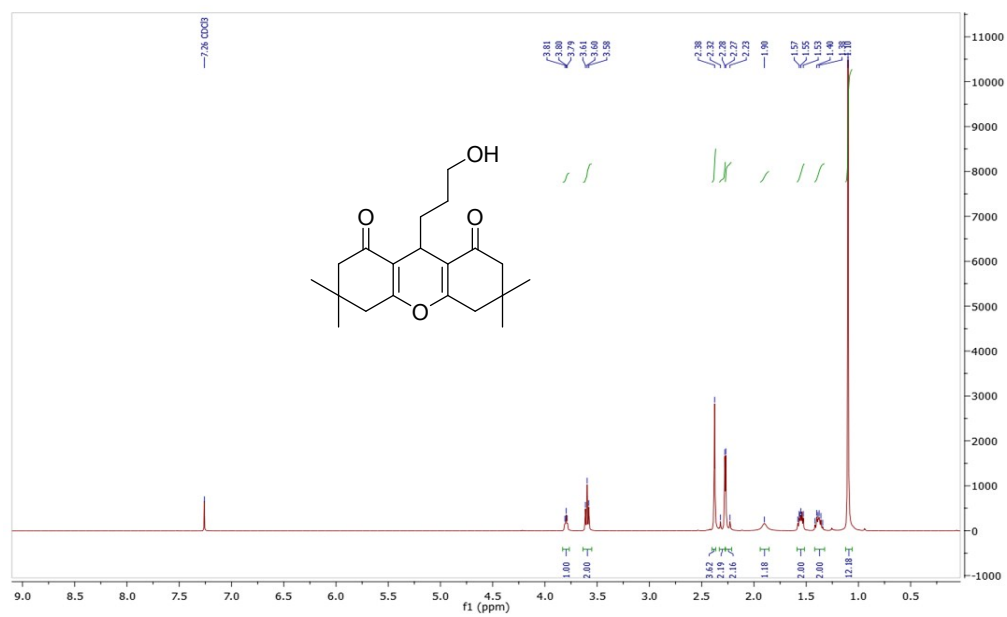
^1H and ^{13}C spectra for 2,2'-methylenebis(3-hydroxy-5,5-dimethylcyclohex-2-enone) (**6i**).⁵



¹H and ¹³C spectra for 3,3,6,6-tetramethyl-9-(pyridin-2-yl)-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione (**6j**).⁶



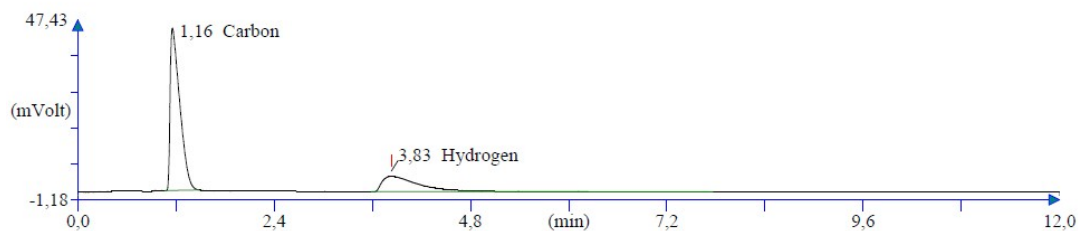
^1H and ^{13}C spectra and organic elemental analysis for 9-(3-hydroxypropyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione (**6k**).



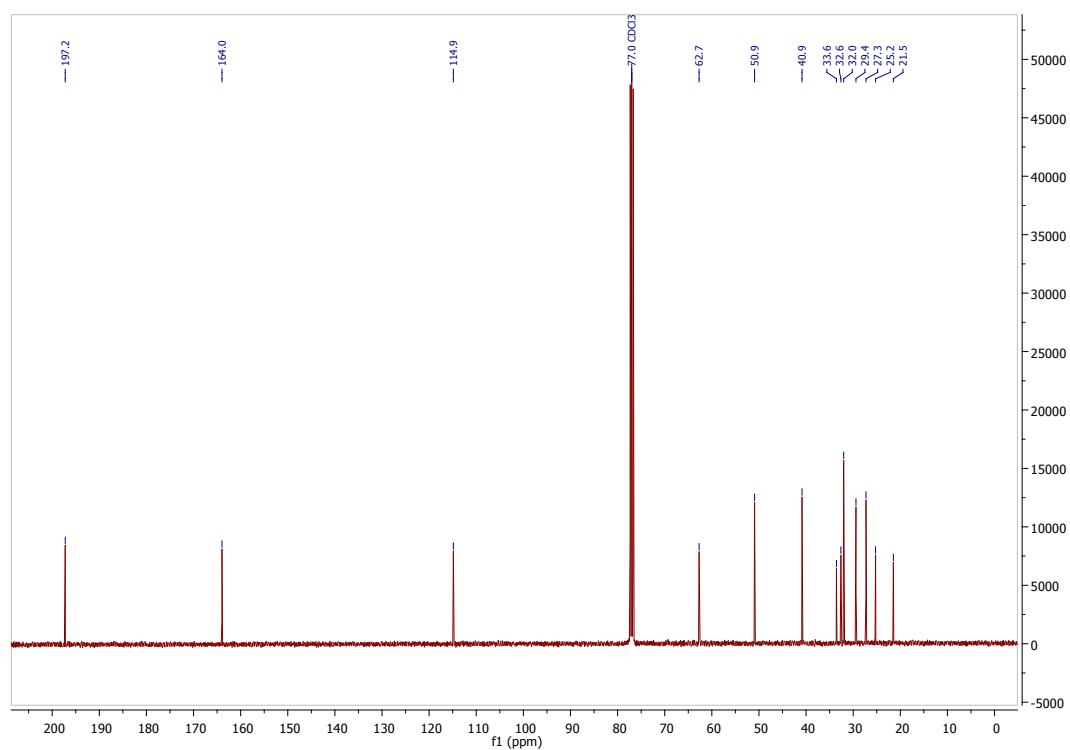
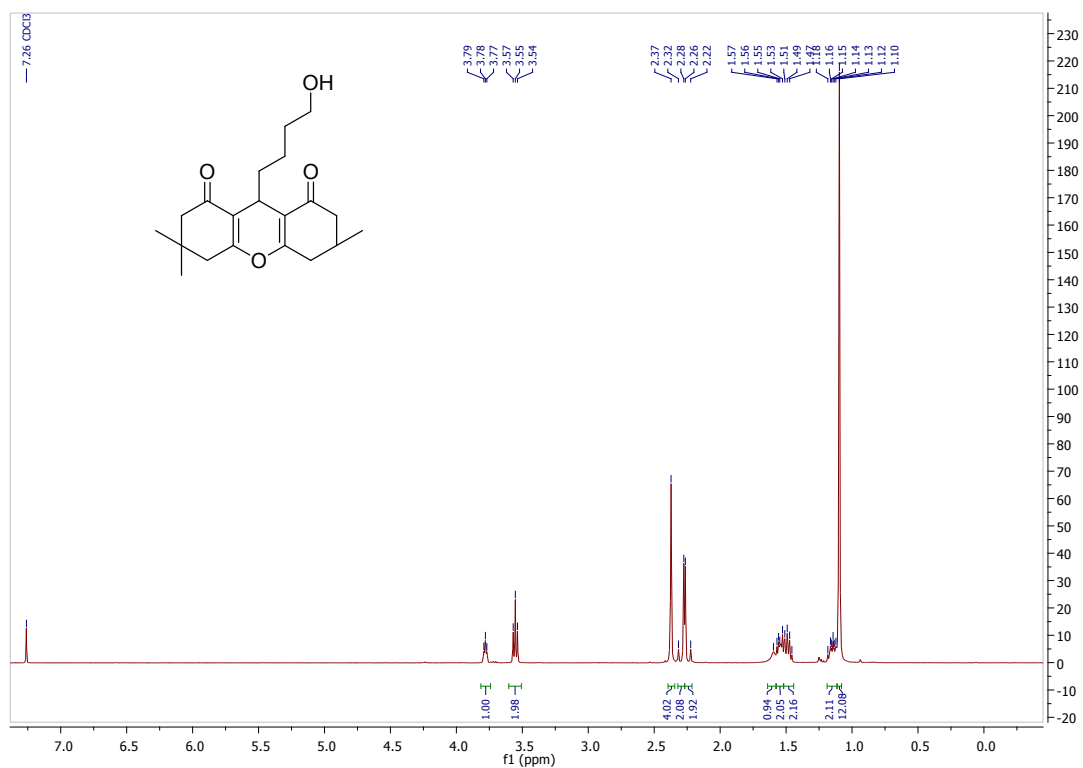
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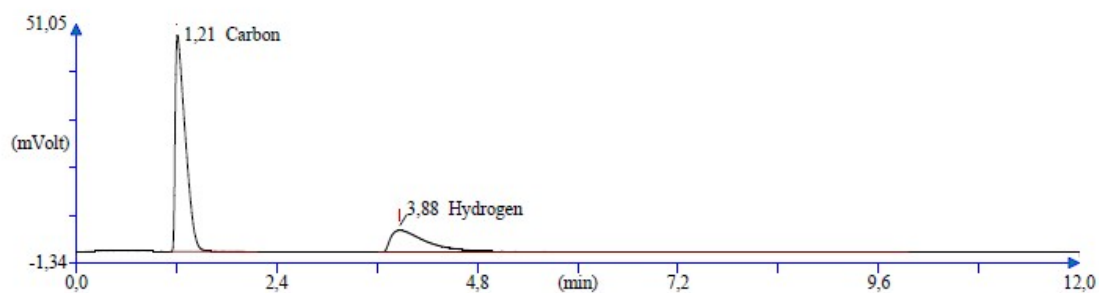
Element Name	Ret. Time	Area	BC	Area ratio	K factor	
Carbon	72.6140	70	3584727	mi	1.000000	.464422E+07
Hydrogen	8.4968	230	1337443	mi	2.680284	.147901E+08
Totals	81.1108		4922170			



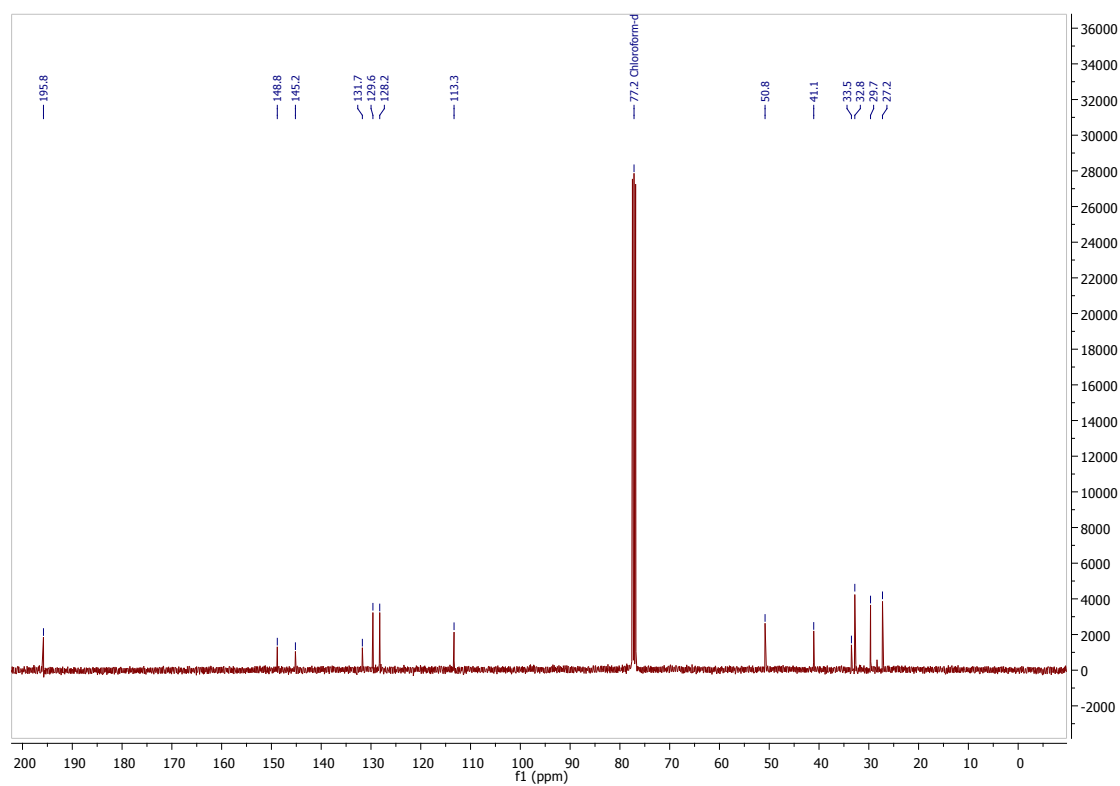
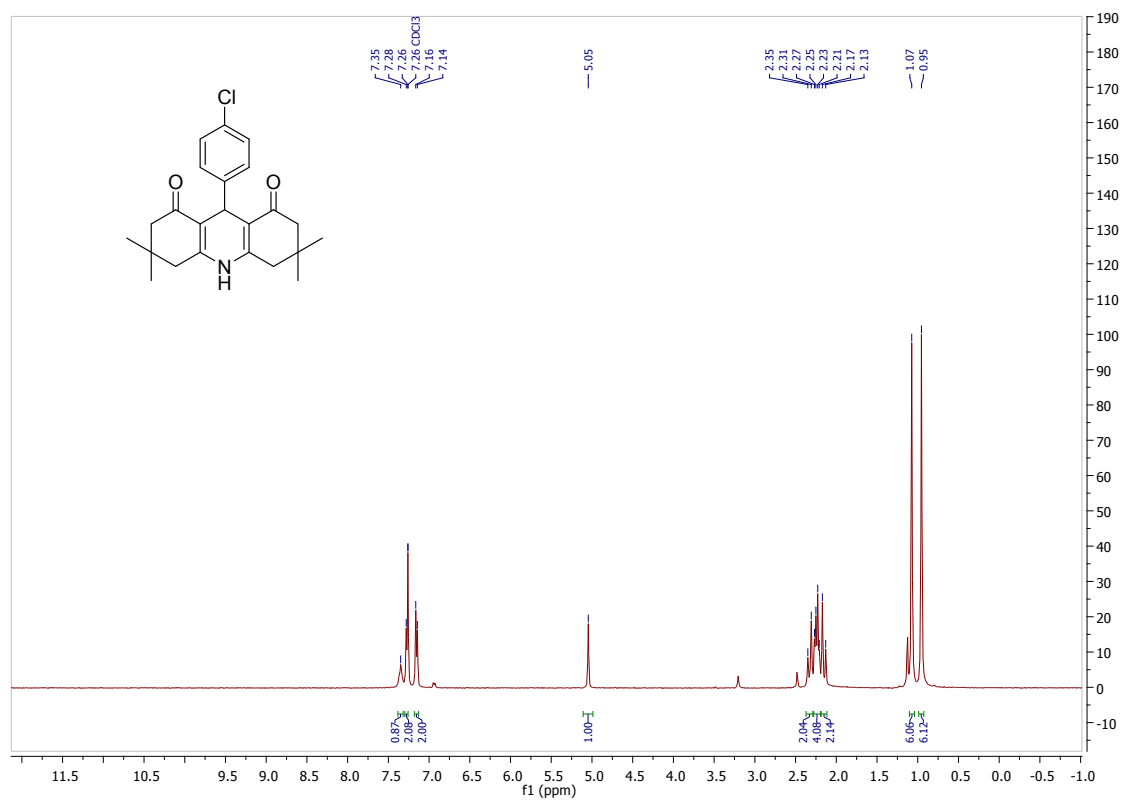
^1H and ^{13}C spectra and organic elemental analysis for 9-(4-hydroxybutyl)-3,3,6,6-tetramethyl-3,4,5,6,7,9-hexahydro-1*H*-xanthene-1,8(2*H*)-dione (**6I**).



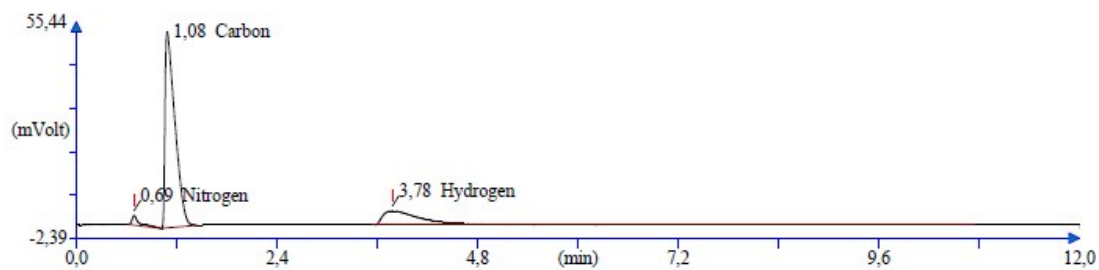
Element Name	% peso	Ret. Time	Area	BC	Area ratio	K factor
Carbon	72.5373	73	3982816	RS	1.000000	
Hydrogen	8.6833	233	1492913	RS	2.667815	
Totals	81.2206		5475729			



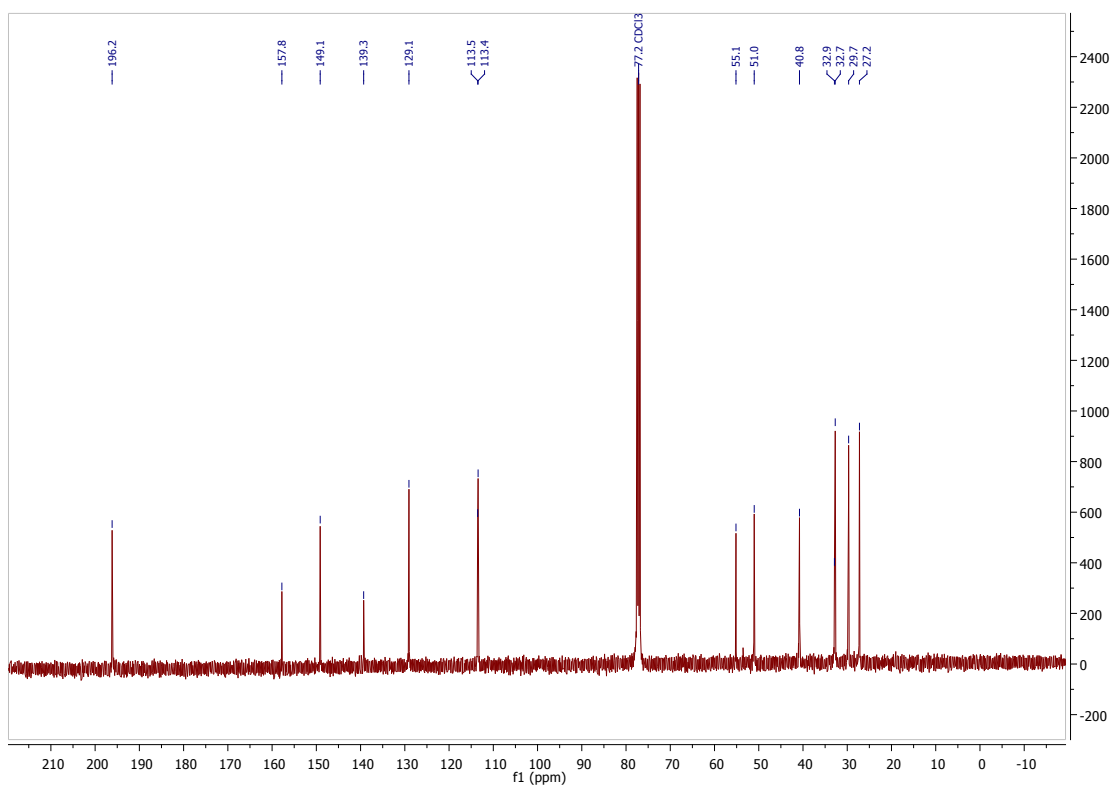
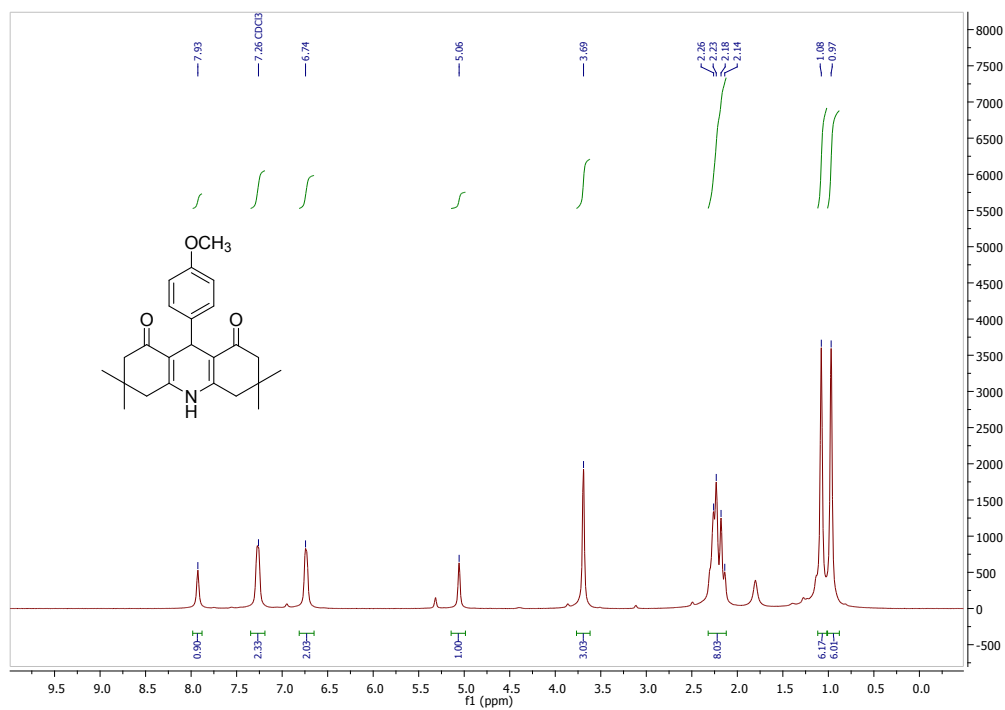
^1H and ^{13}C spectra for 9-(4-chlorophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5a**).^{1, 7}



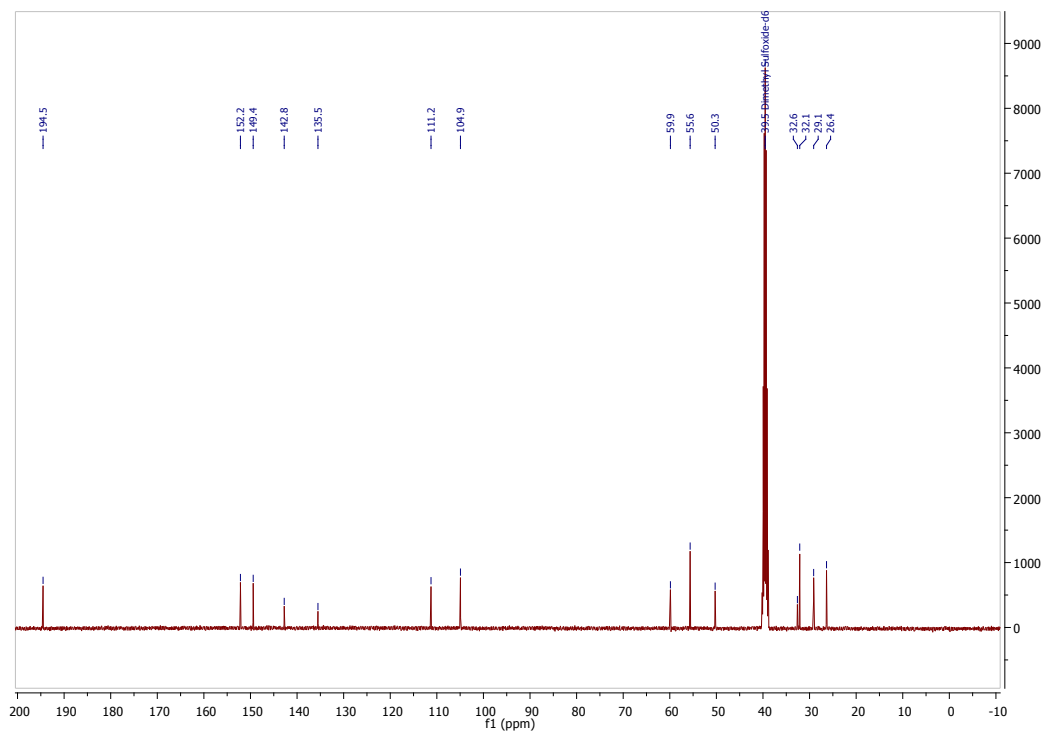
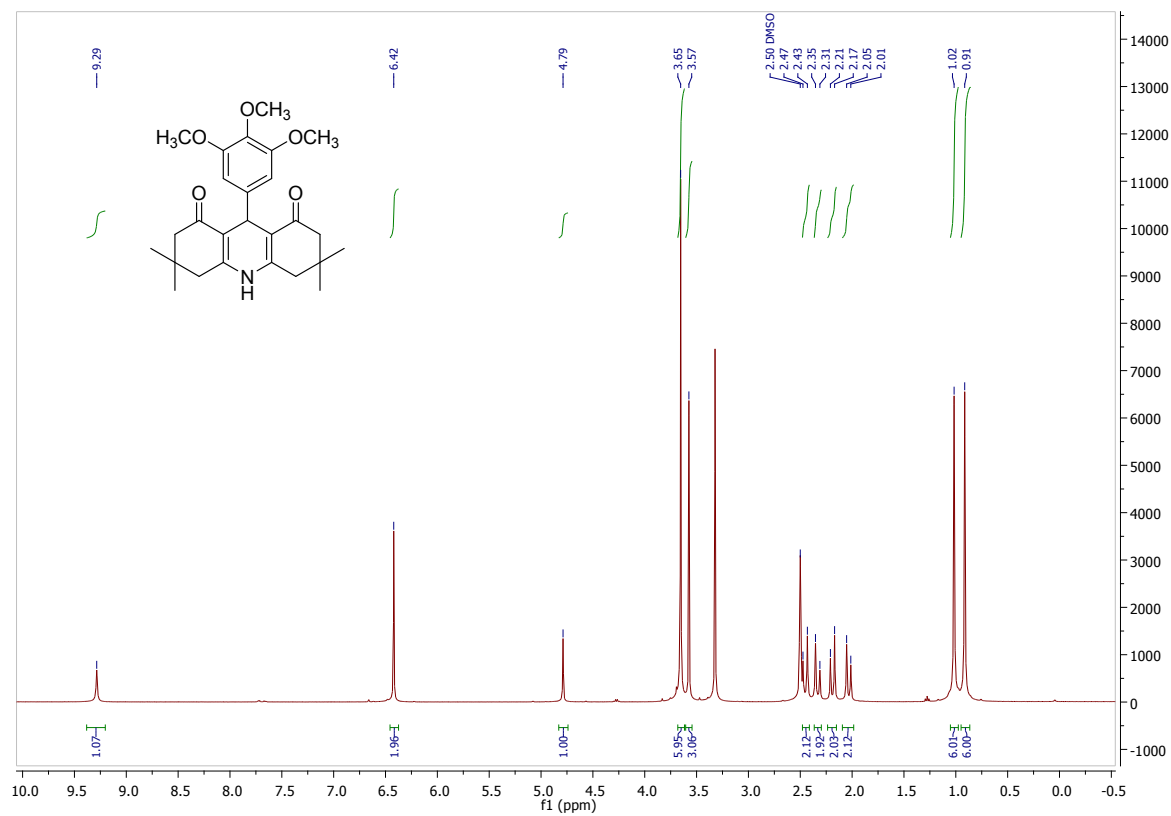
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Nitrogen	3.6352	42	153917	RS	27.979890 .187129E+07
Carbon	71.9872	65	4308580	RS	1.000000 .449478E+07
Hydrogen	6.8093	227	1332464	RS	3.232042 .147351E+08
Totals	82.4318		5792961		



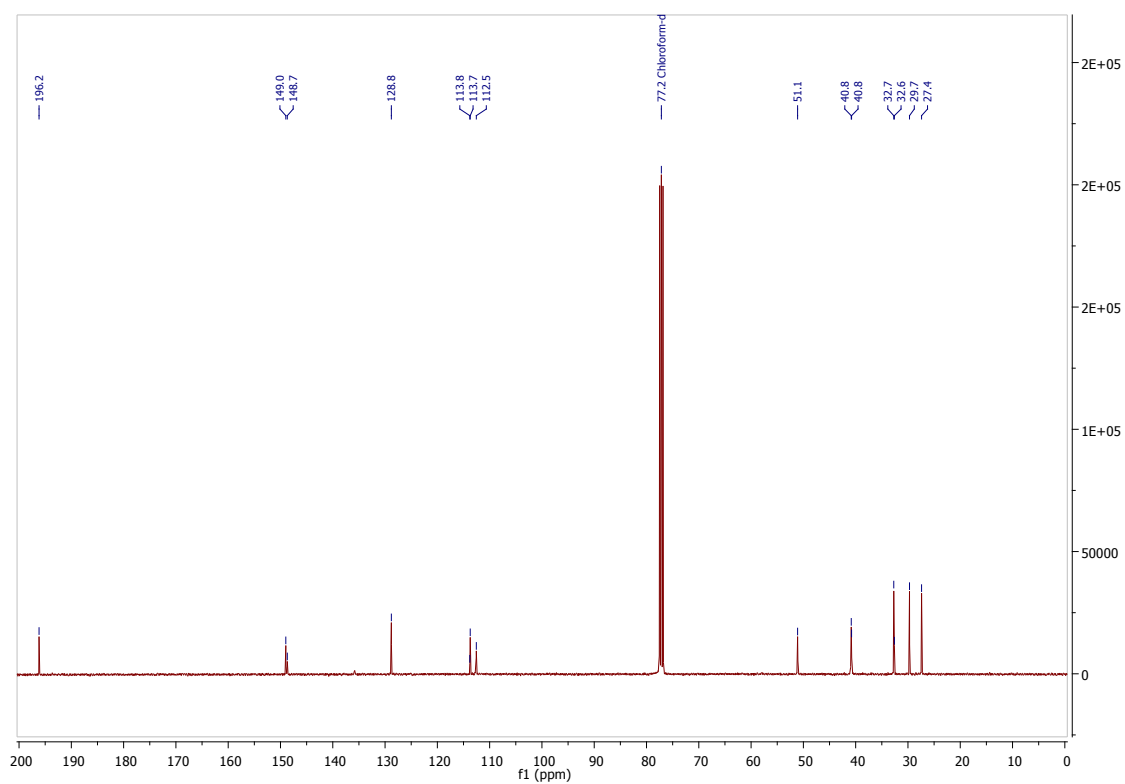
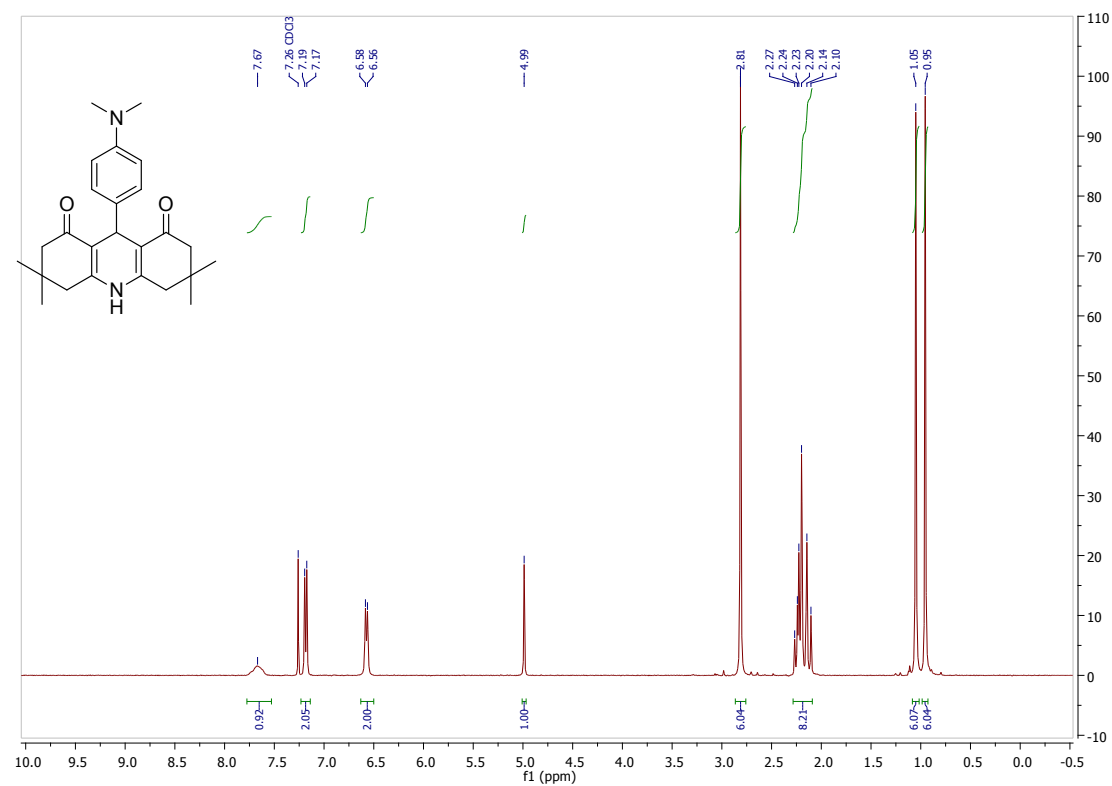
^1H and ^{13}C spectra for 9-(4-methoxyphenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5b**).⁷



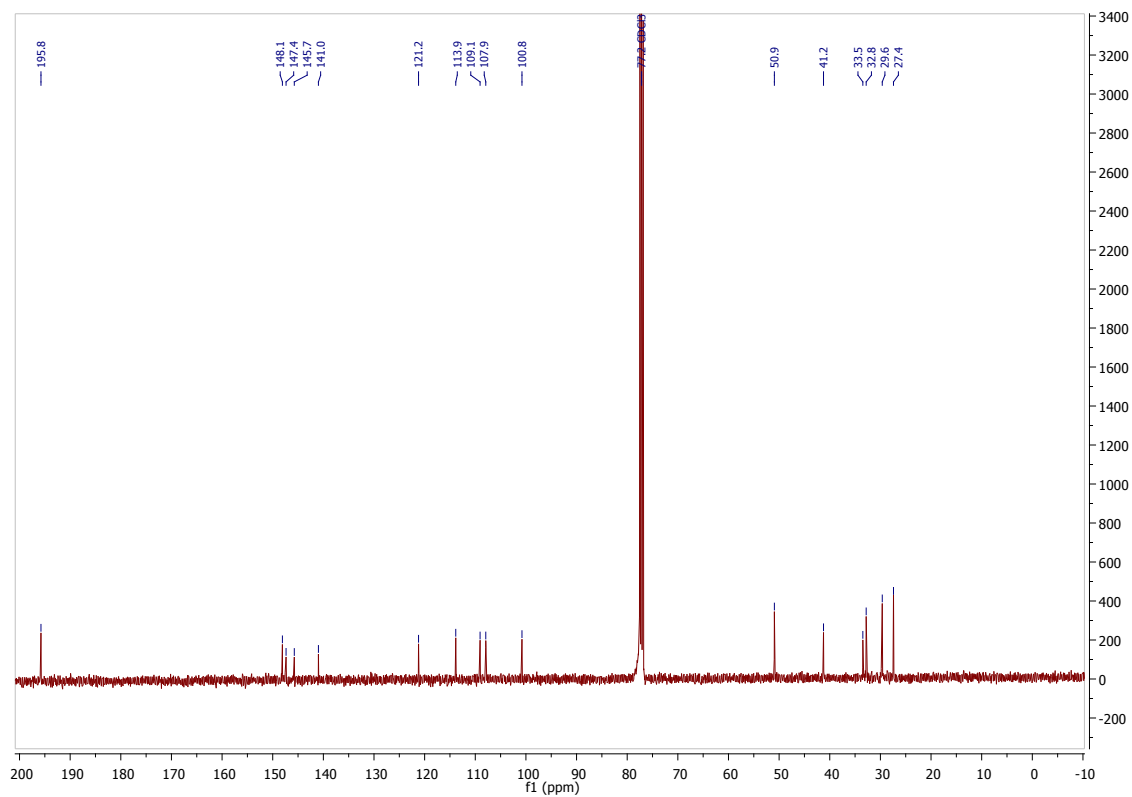
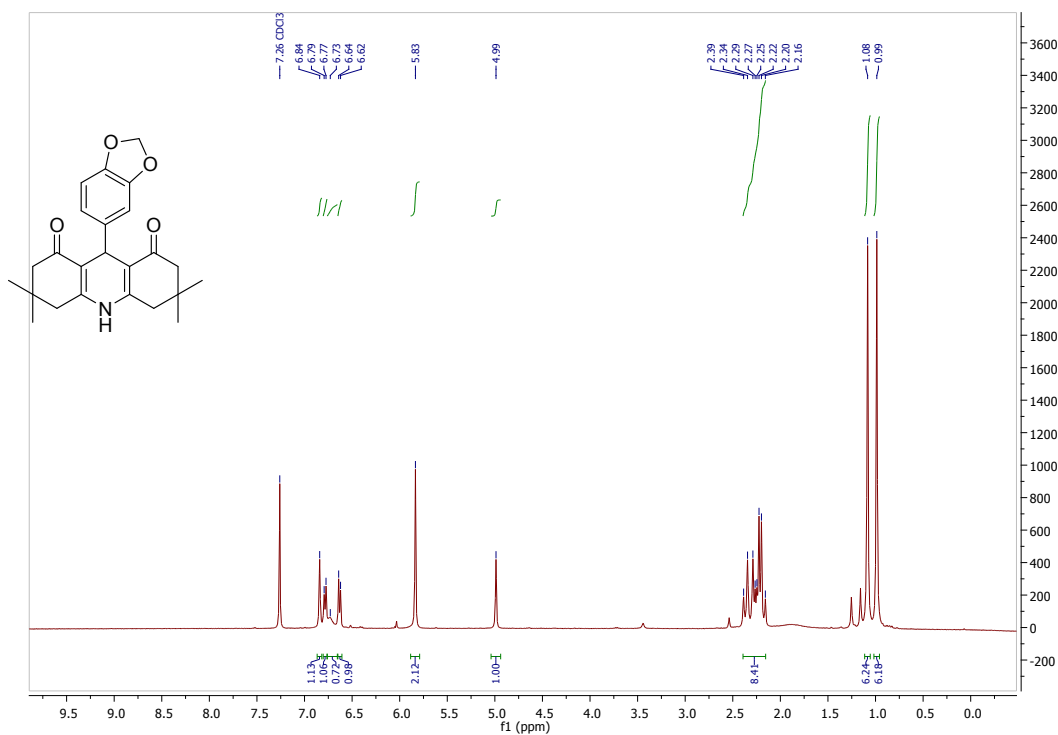
^1H and ^{13}C spectra for 3,3,6,6-tetramethyl-9-(3,4,5-trimethoxyphenyl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5c**).^{8,9}



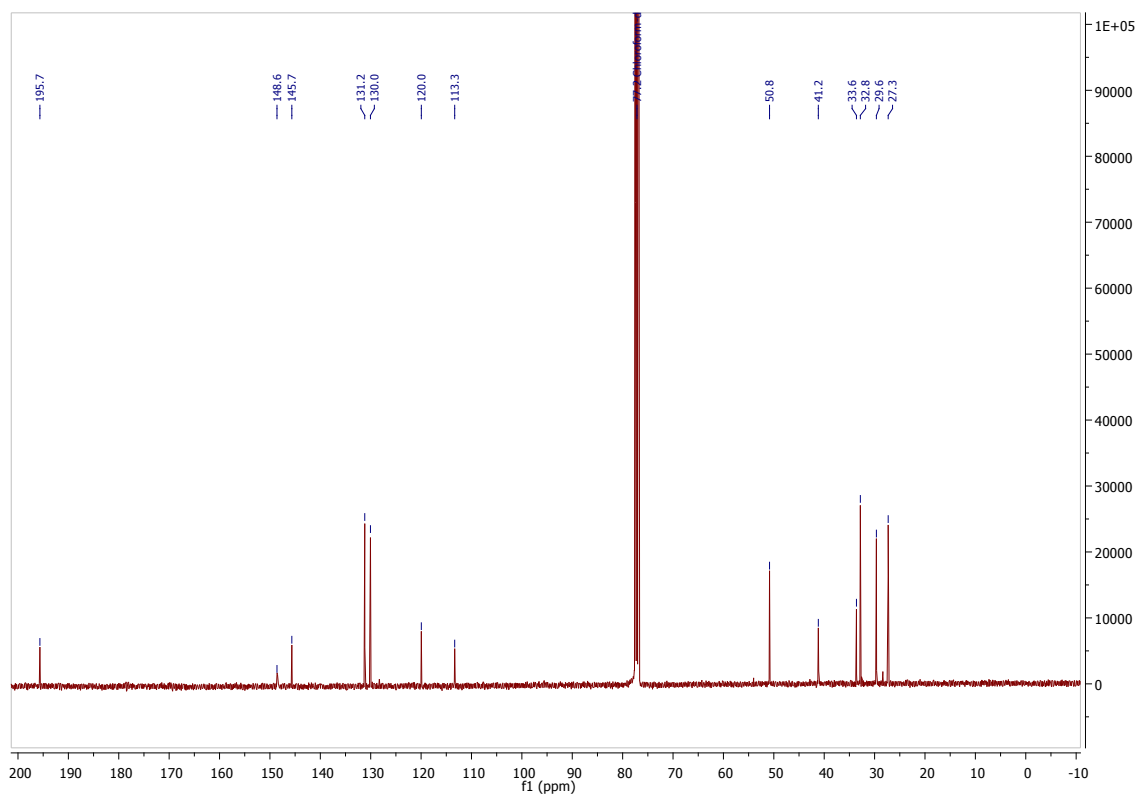
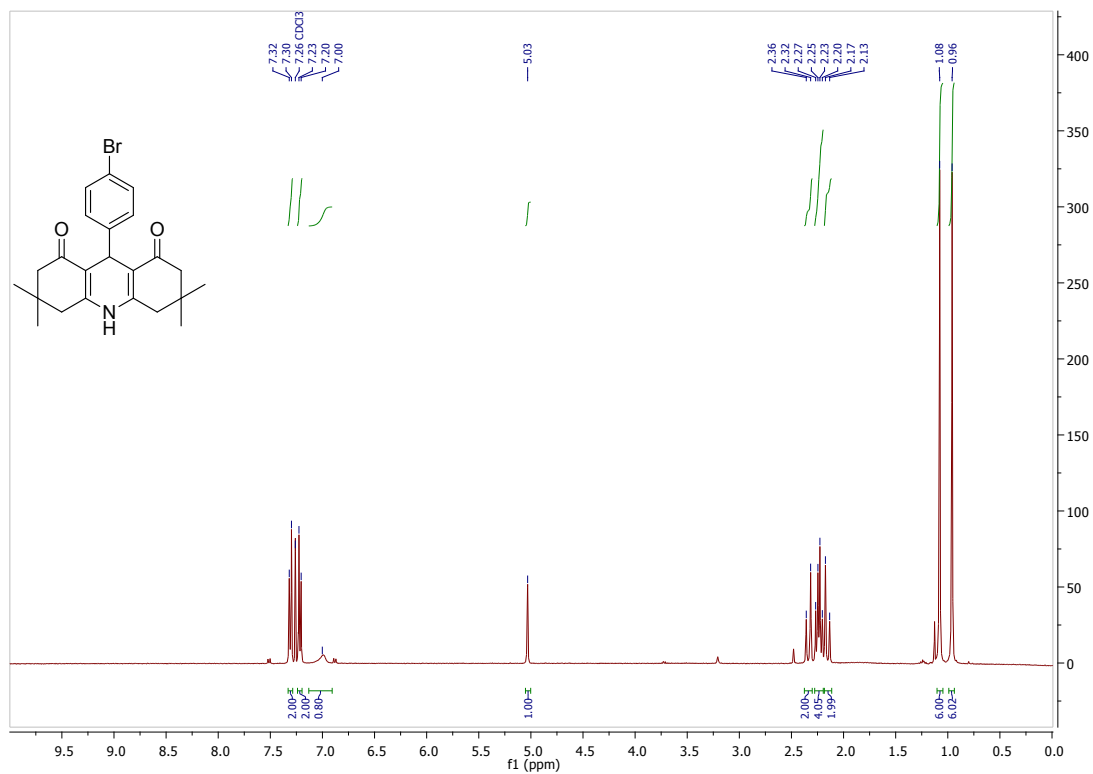
^1H and ^{13}C spectra for 9-(4-(dimethylamino)phenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5d**).⁷



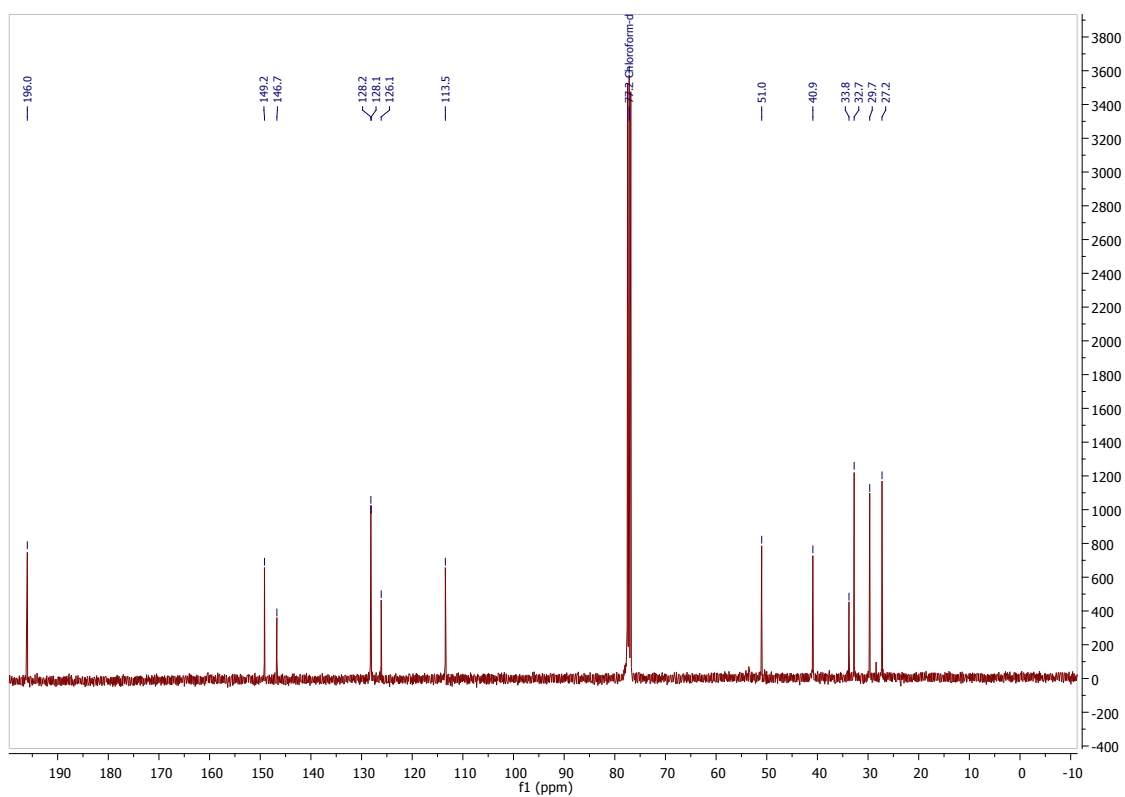
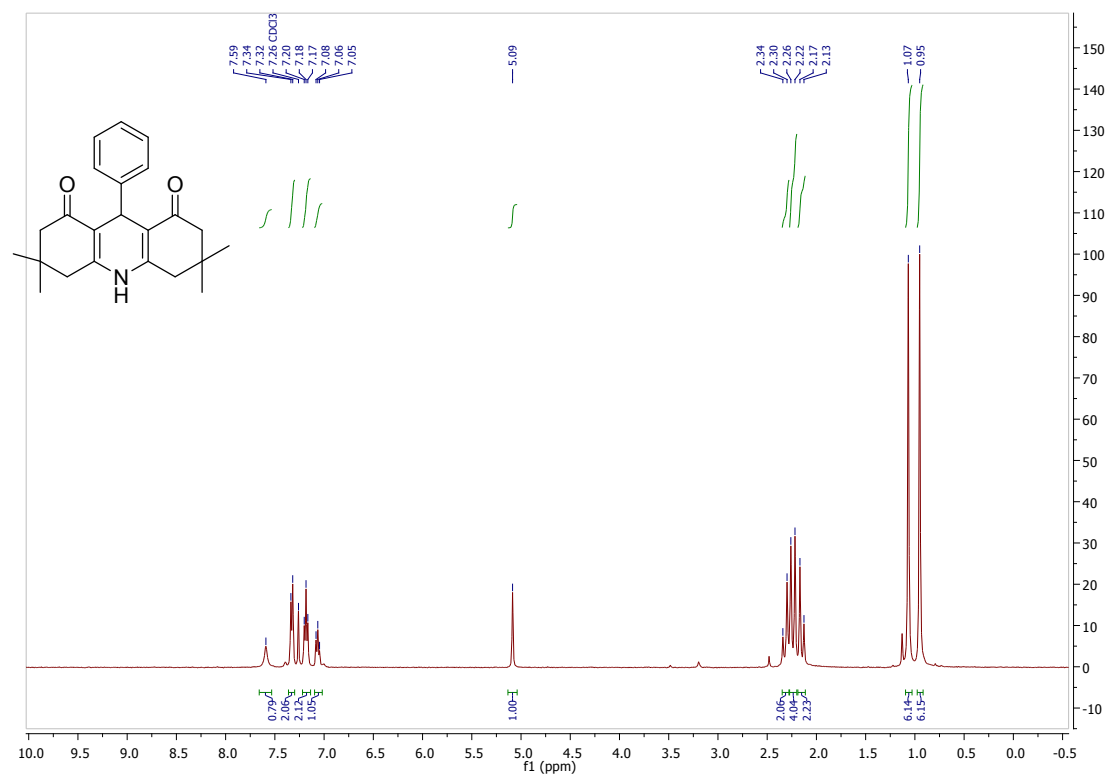
^1H and ^{13}C spectra for 9-(benzo[d][1,3]dioxol-5-yl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5e**).⁷



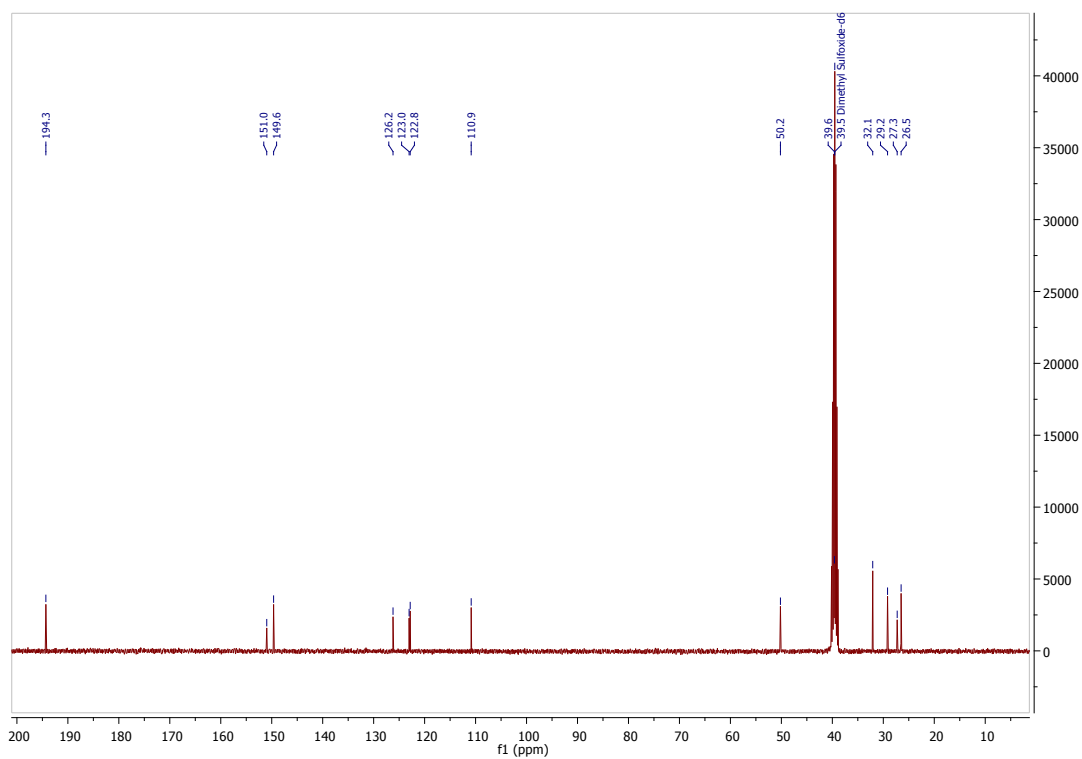
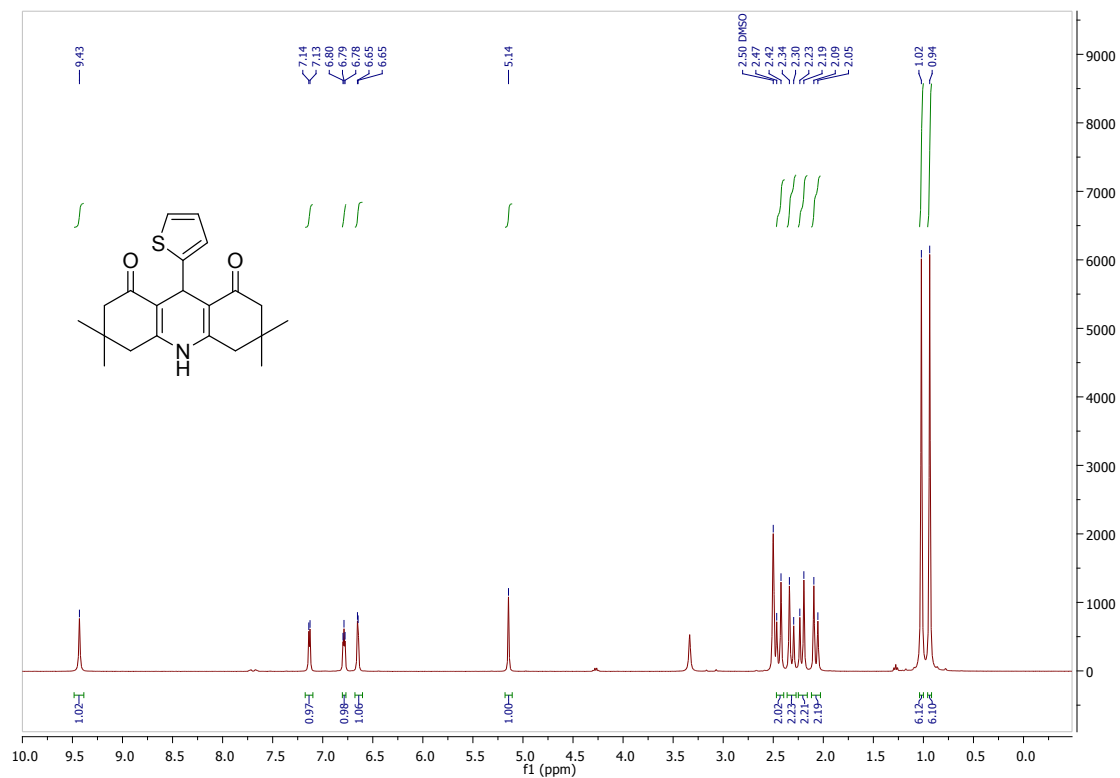
^1H and ^{13}C spectra for 9-(4-bromophenyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5f**).⁷



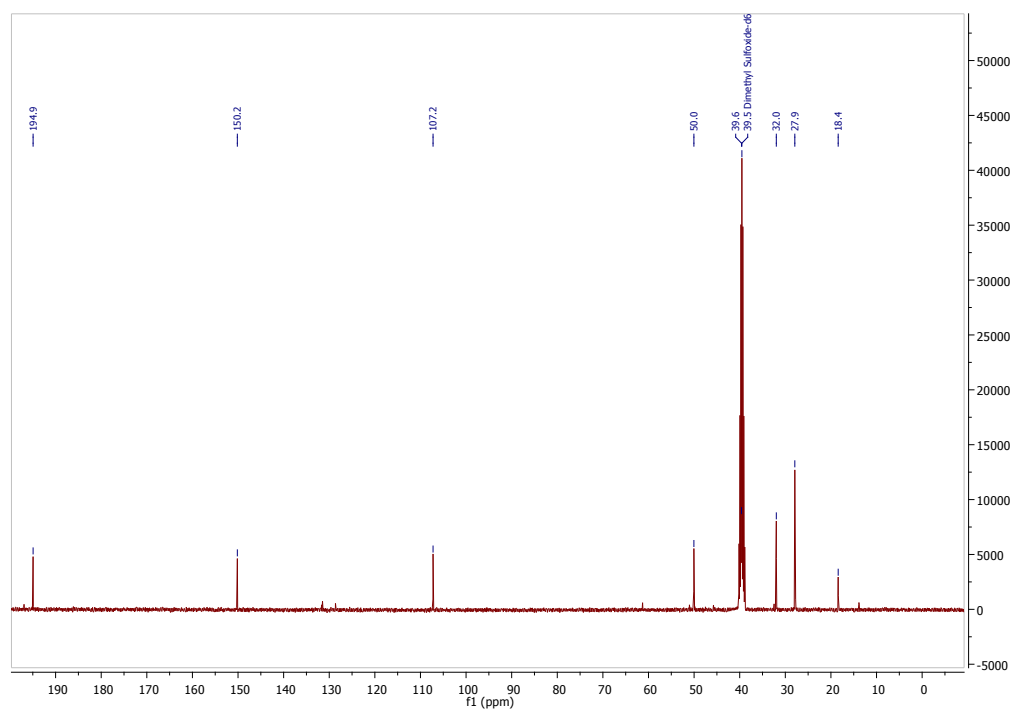
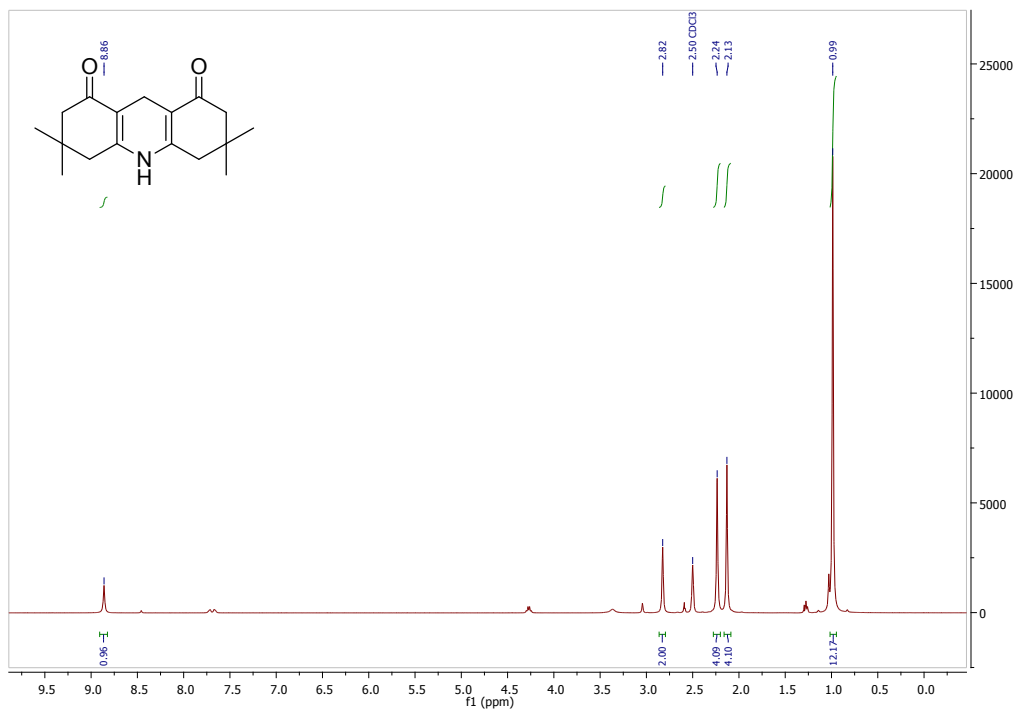
¹H and ¹³C spectra for 3,3,6,6-tetramethyl-9-phenyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5g**).⁷



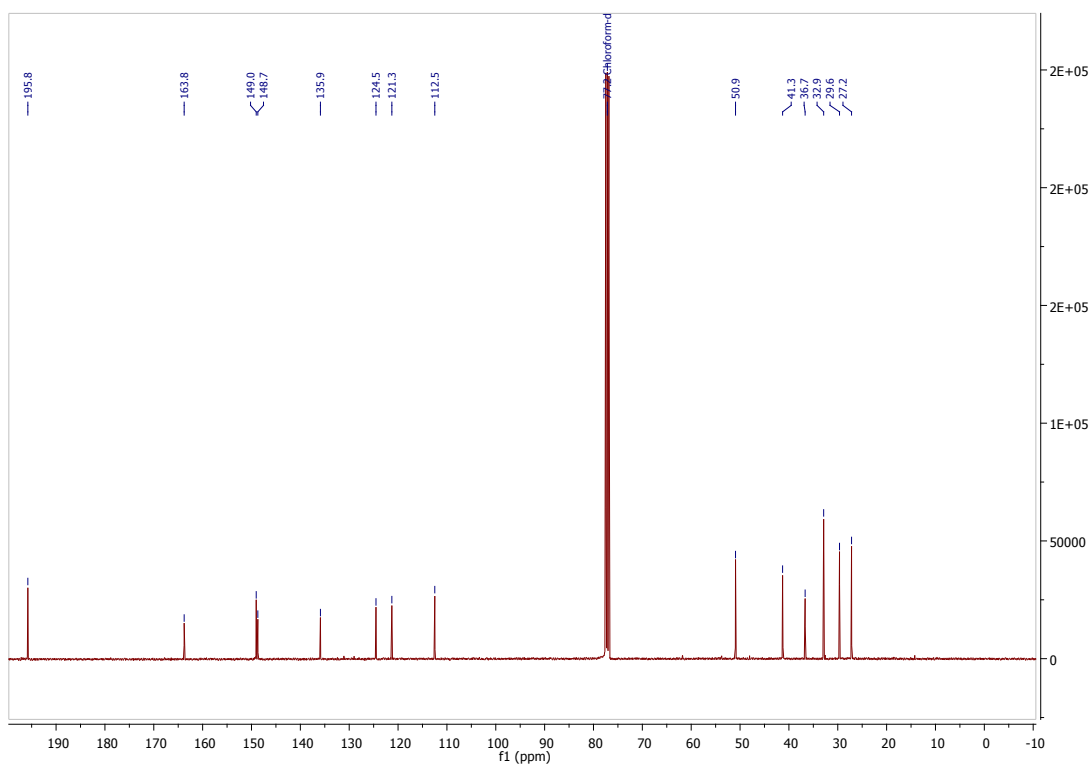
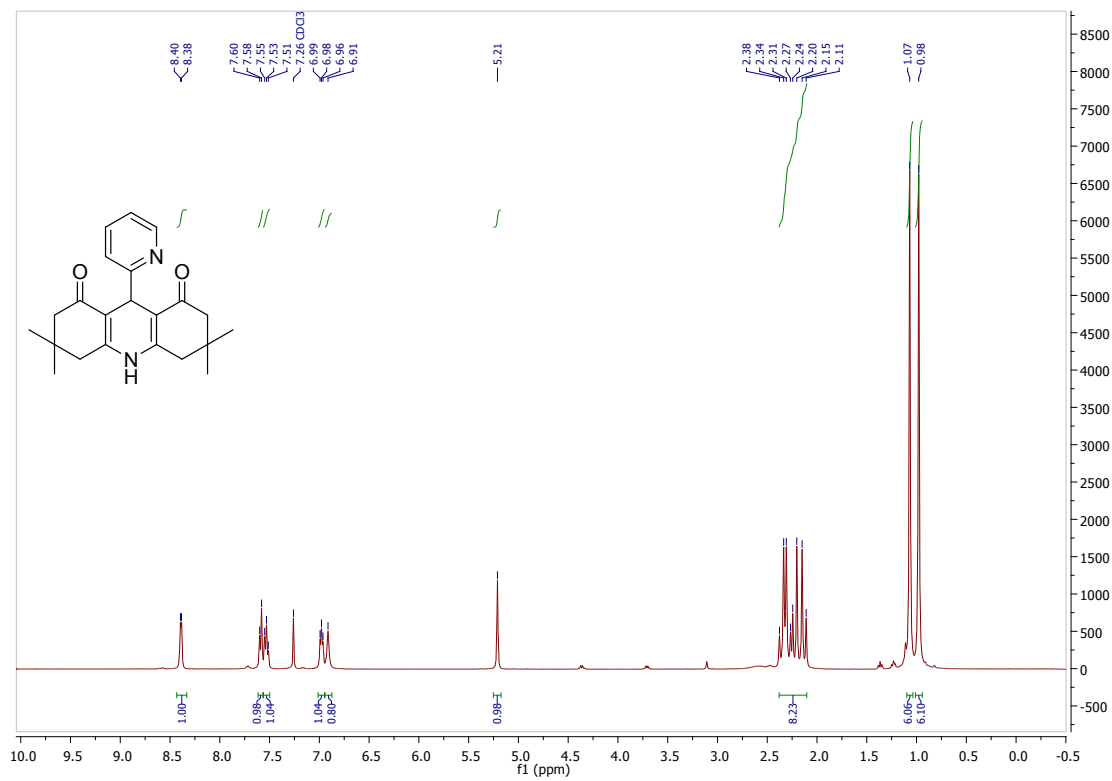
^1H and ^{13}C spectra for 3,3,6,6-tetramethyl-9-(thiophen-2-yl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5h**).⁹



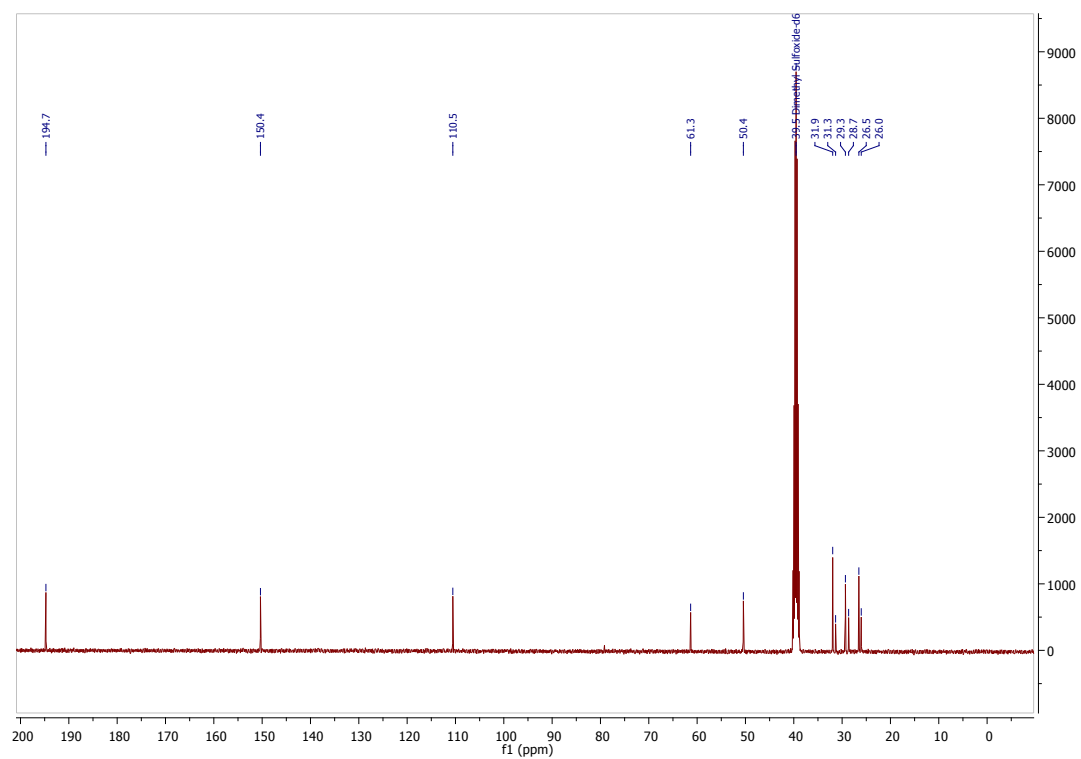
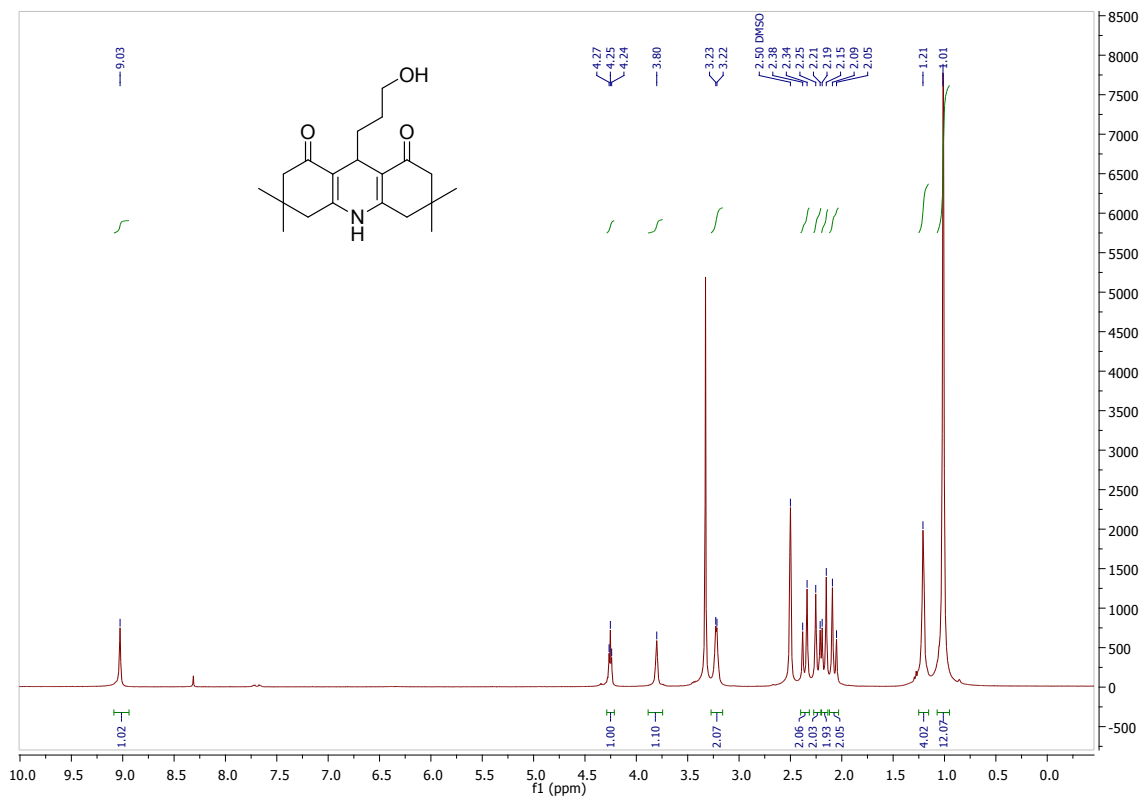
^1H and ^{13}C spectra for 3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5i**).¹⁰



¹H and ¹³C spectra for 3,3,6,6-tetramethyl-9-(pyridin-2-yl)-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5j**).¹

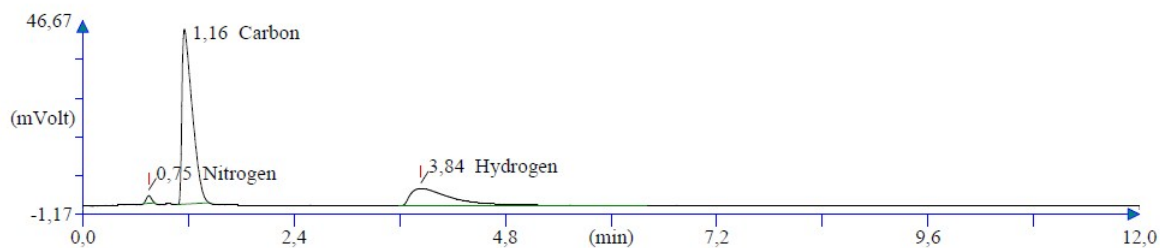


¹H and ¹³C spectra and organic elemental analysis for 9-(3-hydroxypropyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5k**).

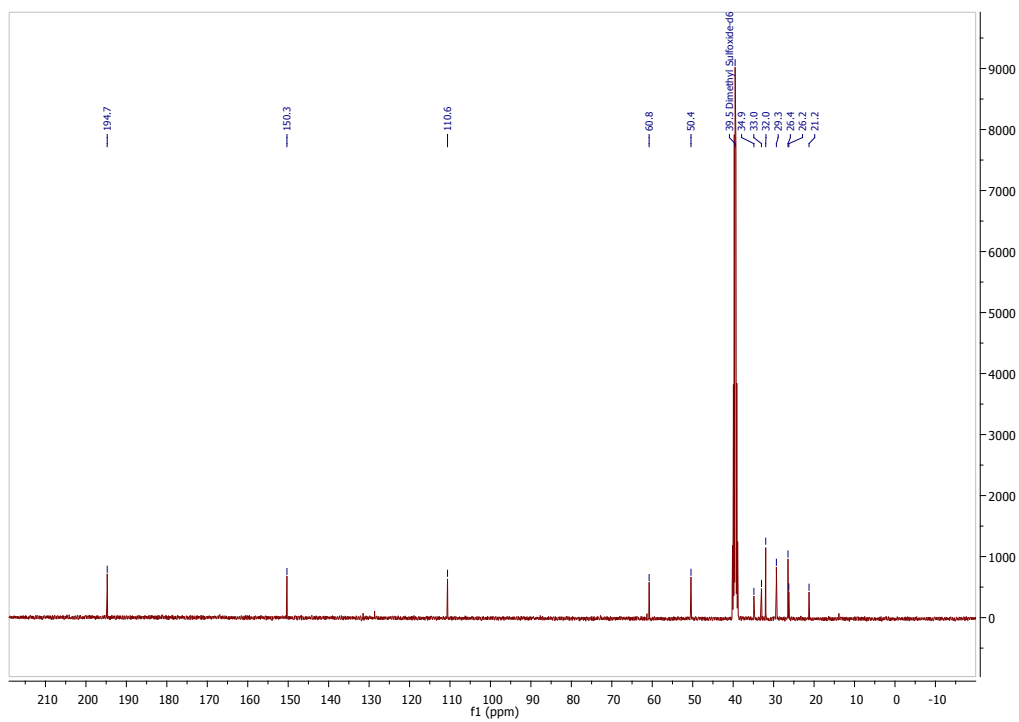
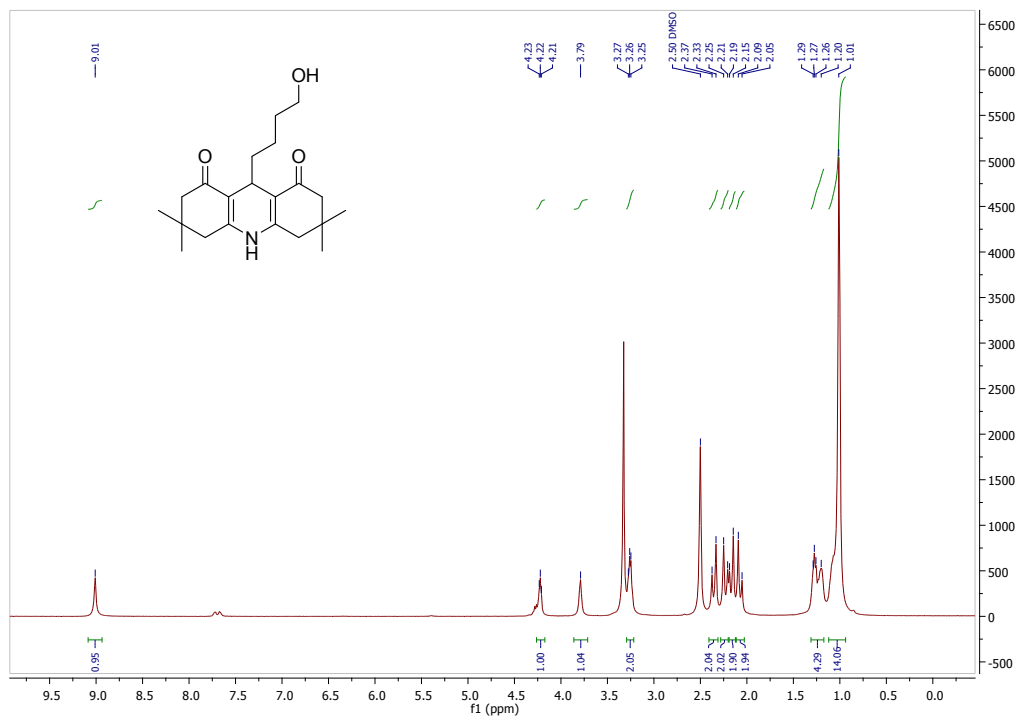


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Element Name		Ret. Time	Area	BC	Area ratio	K factor
Nitrogen	4.1417	45	75921	mi	46.053710	.177283E+07
Carbon	72.6015	70	3496444	mi	1.000000	.464422E+07
Hydrogen	8.8400	231	1357251	mi	2.576122	.147901E+08
Totals	85.5832		4929616			

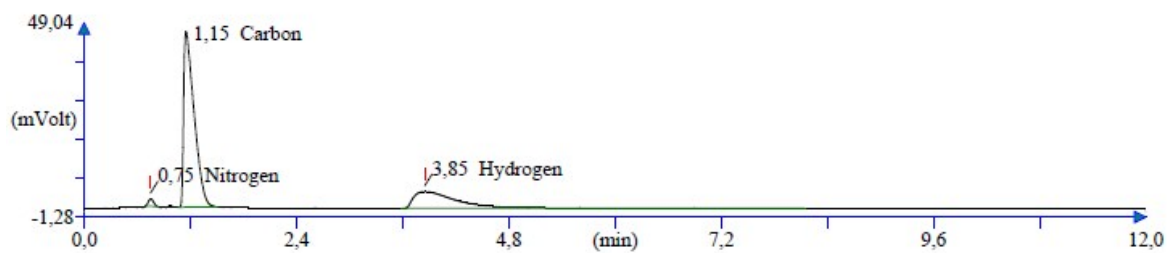


^1H and ^{13}C spectra and organic elemental analysis for 9-(4-hydroxybutyl)-3,3,6,6-tetramethyl-3,4,6,7,9,10-hexahydroacridine-1,8(2*H*,5*H*)-dione (**5I**).



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Element Name	Ret. Time	Area	BC	Area ratio	K factor
Nitrogen	3.9768	45	77269	mi	48.425910 .177283E+07
Carbon	73.3152	69	3741822	mi	1.000000 .464422E+07
Hydrogen	9.1084	231	1481815	mi	2.525161 .147901E+08
Totals	86.4003		5300906		



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