

Organocatalyzed Asymmetric Aldol Reactions of Ketones and β,γ -Unsaturated α -Ketoesters and Phenylglyoxal Hydrates

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Supporting Information

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X-ray Crystallography and Data Solution

The data were collected at 98(2) K using a Rigaku AFC12/Saturn 724 CCD fitted with Mo K α radiation ($k = 0.71073 \text{ \AA}$). Data collection and unit cell refinement were performed using CRYSTAL CLEAR software.¹⁷ The total number of data were measured in the range $2.90^\circ < \theta < 25.5^\circ$ and $2.35^\circ < \theta < 27.5^\circ$ using ω scans for structures **3f** [methyl (*R,E*)-4-(4-bromophenyl)-2-hydroxy-2-(2-oxo-2-phenylethyl)but-3-enoate] and **22d** {(*R*)-2-[(*S*)-2-(4-bromophenyl)-1-hydroxy-2-oxoethyl]cyclohexanone}, respectively. Data processing and absorption correction, giving minimum and maximum transmission factors, were accomplished with CRYSTAL CLEAR and ABSCOR,¹⁸ respectively. The structure, using SHELXL-97, was solved by direct methods and refined (on F^2) using full-matrix, least-squares techniques.^{19,20} All non-hydrogen atoms, for all structures, were refined with anisotropic displacement parameters. The following hydrogen atoms: H2A, H3A, H5A, H6A, H9A and H2B, for structure **22d**, were located on a difference Fourier map. The carbon bound hydrogen atoms were refined with $U_{\text{iso}}(\text{H}) = 1.2 U_{\text{eq}}(\text{C})$ and oxygen bound hydrogen atoms were refined with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$. All remaining carbon bound hydrogen atoms, on structure **22d**, were determined by geometry and refined by a riding model. For structure **3f**, all carbon bound hydrogen atoms were determined by geometry and refined by a riding model. The oxygen bound hydrogen atom was located in a difference Fourier map and refined with $U_{\text{iso}}(\text{H}) = 1.5 U_{\text{eq}}(\text{O})$.

Table S-1. Crystal Data and Structure Refinement for Compounds 3f and 22d

Compound	3f	22d
Empirical formula	C ₁₉ H ₁₇ BrO ₄	C ₁₄ H ₁₅ BrO ₃
Formula weight	389.24	311.17
Crystal system	orthorhombic	orthorhombic
Space group	P 2 ₁ 2 ₁ 2 ₁	P 2 ₁ 2 ₁ 2 ₁
<i>a</i> , Å	5.7652(15)	7.422(2)
<i>b</i> , Å	14.062(4)	10.122(3)
<i>c</i> , Å	20.949(5)	16.852(5)
<i>α</i> , deg	90.00	90.00
<i>β</i> , deg	90.00	90.00
<i>γ</i> , deg	90.00	90.00
vol., Å ³	1698.3(8)	1266.0(6)
<i>Z</i>	4	4
D _(cal) , g/cm ³	1.522	1.633
μ(M _o -K _α , mm ⁻¹)	2.440	3.244
<i>F</i> (000)	792	632
2θ range for data collection (°)	5.80 to 51.00	4.70 to 55.00
Index Ranges	-5 ≤ <i>h</i> ≤ 6, -17 ≤ <i>k</i> ≤ 15, -25 ≤ <i>l</i> ≤ 11	-9 ≤ <i>h</i> ≤ 9, -6 ≤ <i>k</i> ≤ 13, -21 ≤ <i>l</i> ≤ 15
Reflections collected	4955	4580
Independent reflections	3090[R(int) = 0.1001]	2857[R(int) = 0.1235]
Max. and Min. transmission	1.000, 0.528	1.000, 0.633
Data/Restraints/Parameters	3090/220/1	2857/181/0
<i>wR</i> (<i>F</i> ² all data)	R1 = 0.0627, <i>wR</i> 2 = 0.1328	R1 = 0.0594, <i>wR</i> 2 = 0.1356
<i>R</i> (<i>F</i> obsd data) [<i>I</i> > 2σ(<i>I</i>)]	R1 = 0.0566, <i>wR</i> 2 = 0.1283	R1 = 0.0568, <i>wR</i> 2 = 0.1337
goodness-of-fit on <i>F</i> ²	1.003	

$$wR_2 = \{ \sum [w(F_o^2 - F_c^2)^2] / \sum [w(F_o^2)^2] \}^{1/2}$$

$$R_1 = \sum ||F_o| - |F_c|| / \sum |F_o|$$

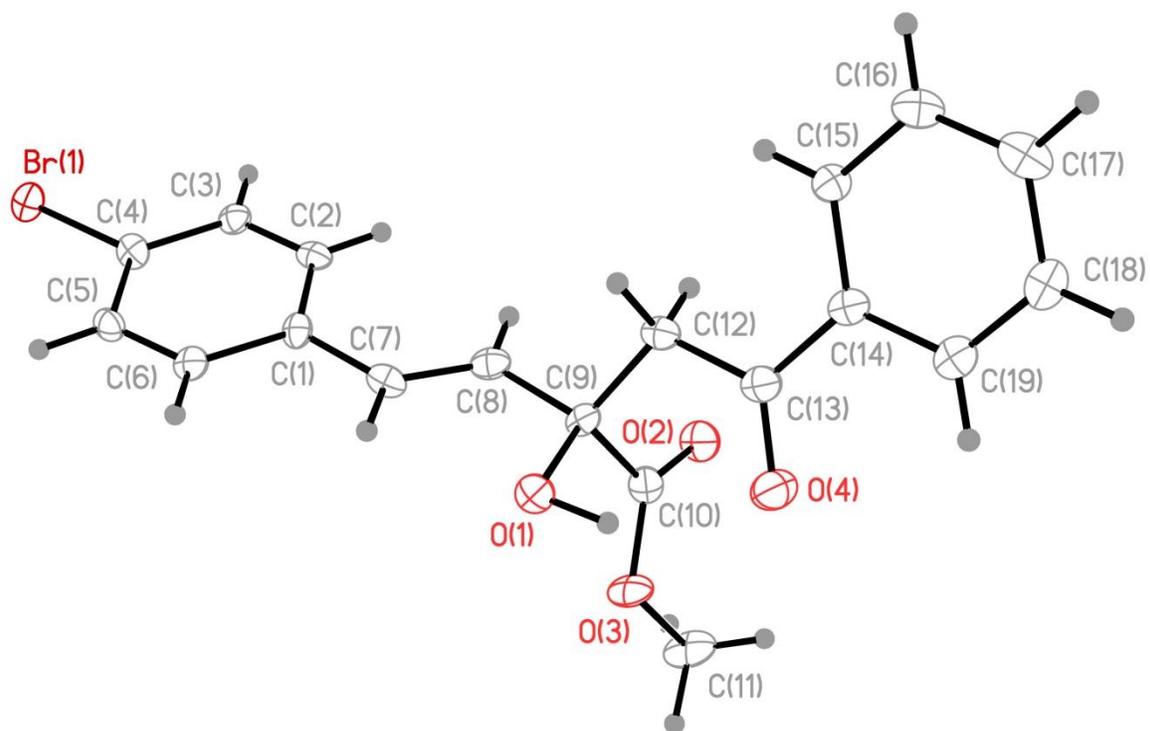


Figure S-1. Thermal ellipsoid plot of the asymmetric unit of compound **3f**

Table S-2. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2) for structure 3f

	x	y	z	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.54064 (10)	0.15278 (4)	0.17098 (3)	0.02231 (18)
O1	1.4637 (7)	0.1008 (3)	-0.16407 (19)	0.0202 (9)
O3	1.5614 (7)	-0.0790 (3)	-0.1602 (2)	0.0236 (10)
C9	1.2694 (9)	0.0405 (4)	-0.1762 (3)	0.0157 (11)
C7	1.1421 (10)	0.1031 (4)	-0.0701 (3)	0.0176 (12)
H7A	1.2800	0.1374	-0.0701	0.021*
C4	0.7213 (10)	0.1388 (4)	0.0957 (3)	0.0174 (12)
C5	0.9377 (11)	0.1817 (4)	0.0914 (3)	0.0178 (12)
H5A	0.9950	0.2174	0.1253	0.021*
C19	1.3590 (11)	0.0801 (4)	-0.4123 (3)	0.0207 (13)
H19A	1.5023	0.0504	-0.4079	0.025*
C14	1.2170 (10)	0.0929 (4)	-0.3585 (3)	0.0171 (12)
C10	1.3442 (10)	-0.0652 (4)	-0.1791 (3)	0.0198 (13)
C15	0.9988 (9)	0.1365 (4)	-0.3665 (3)	0.0182 (13)
H15A	0.9007	0.1443	-0.3316	0.022*
C3	0.6386 (10)	0.0828 (4)	0.0452 (3)	0.0173 (12)
H3A	0.4953	0.0528	0.0484	0.021*
O2	1.2142 (7)	-0.1273 (3)	-0.1970 (2)	0.0254 (10)
C6	1.0660 (11)	0.1710 (4)	0.0367 (3)	0.0183 (12)
H6A	1.2082	0.2018	0.0333	0.022*
C1	0.9880 (10)	0.1150 (4)	-0.0139 (3)	0.0165 (12)
C8	1.1022 (10)	0.0489 (4)	-0.1199 (3)	0.0194 (13)
H8A	0.9656	0.0138	-0.1209	0.023*
C16	0.9305 (11)	0.1677 (4)	-0.4266 (3)	0.0235 (13)
H16A	0.7865	0.1966	-0.4315	0.028*
O4	1.5041 (7)	0.0340 (3)	-0.2895 (2)	0.0279 (10)
C17	1.0707 (10)	0.1567 (5)	-0.4786 (3)	0.0254 (13)
H17A	1.0230	0.1790	-0.5182	0.030*
C2	0.7698 (10)	0.0727 (4)	-0.0087 (3)	0.0171 (12)
H2A	0.7125	0.0369	-0.0426	0.021*
C13	1.3039 (10)	0.0629 (5)	-0.2952 (3)	0.0200 (13)
C12	1.1481 (11)	0.0676 (4)	-0.2377 (3)	0.0181 (13)
H12A	1.0174	0.0252	-0.2442	0.022*
H12B	1.0879	0.1317	-0.2336	0.022*
C18	1.2869 (11)	0.1117 (4)	-0.4723 (3)	0.0217 (14)
H18A	1.3813	0.1030	-0.5078	0.026*
C11	1.6415 (11)	-0.1776 (4)	-0.1649 (4)	0.0306 (15)
H11A	1.7987	-0.1817	-0.1501	0.046*
H11B	1.6338	-0.1980	-0.2086	0.046*
H11C	1.5444	-0.2176	-0.1392	0.046*
H1A	1.550 (12)	0.091 (6)	-0.205 (2)	0.046*

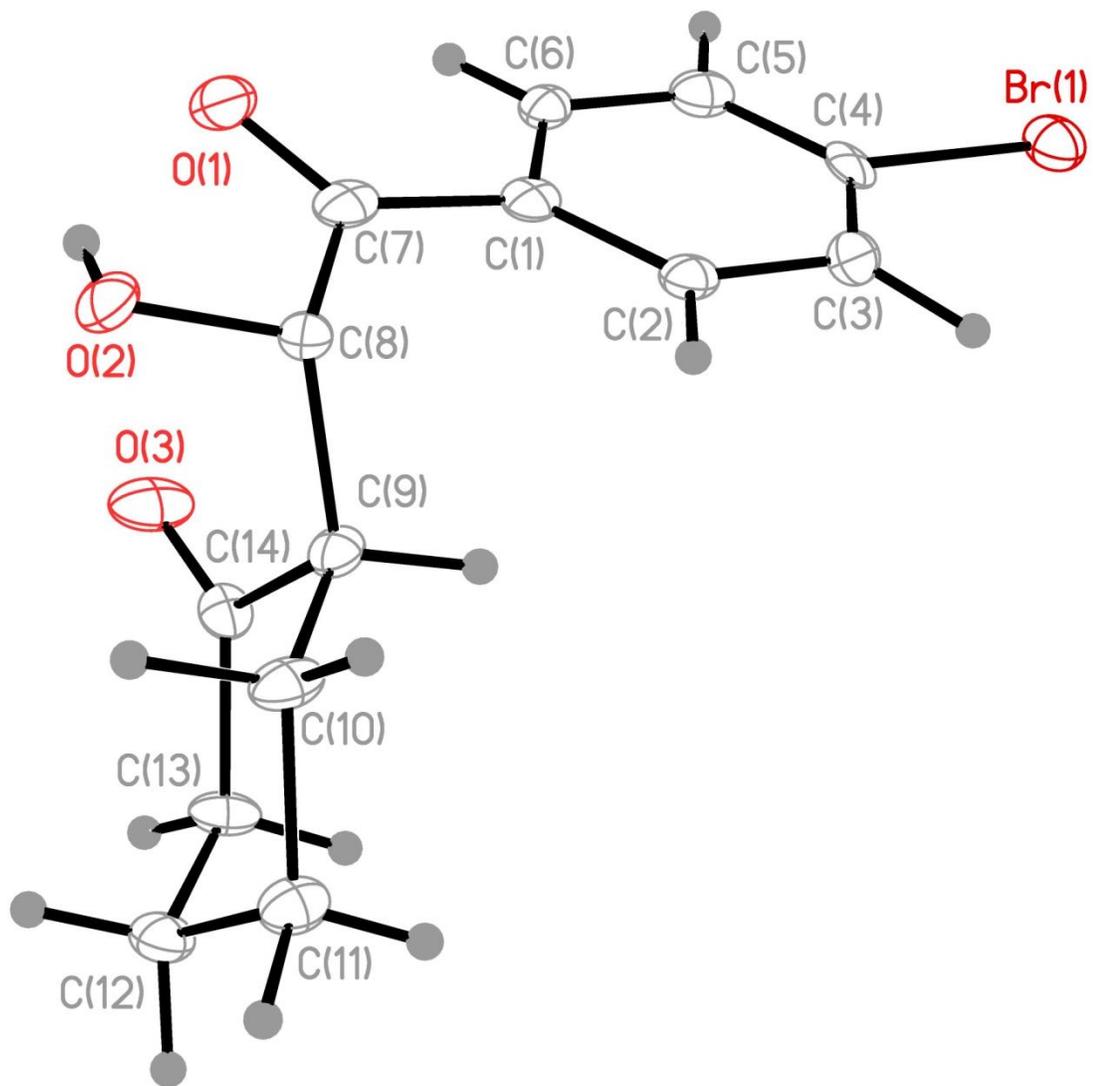


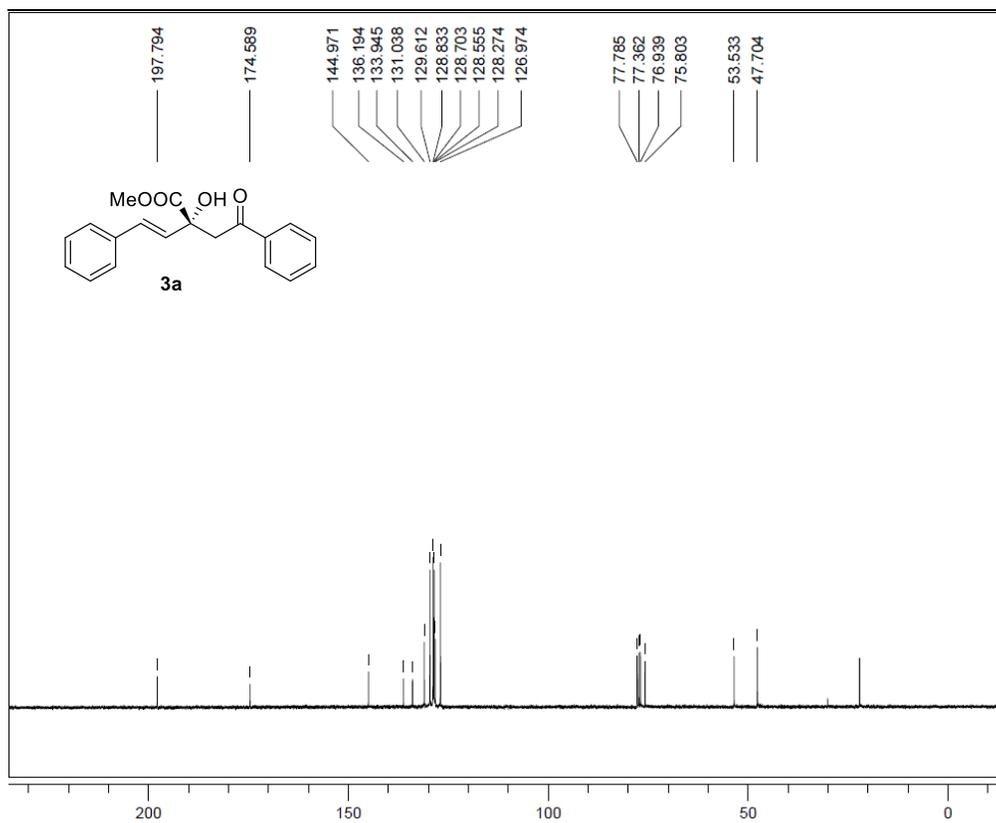
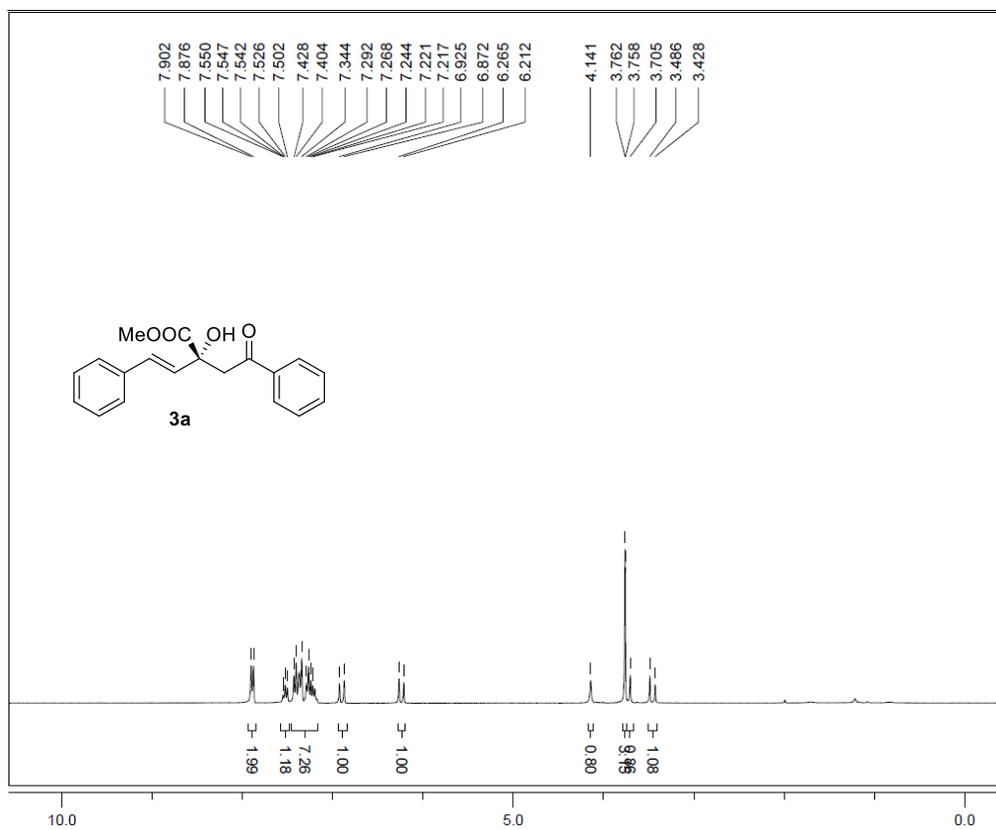
Figure S-2. Thermal ellipsoid plot of the asymmetric unit of compound **22d**

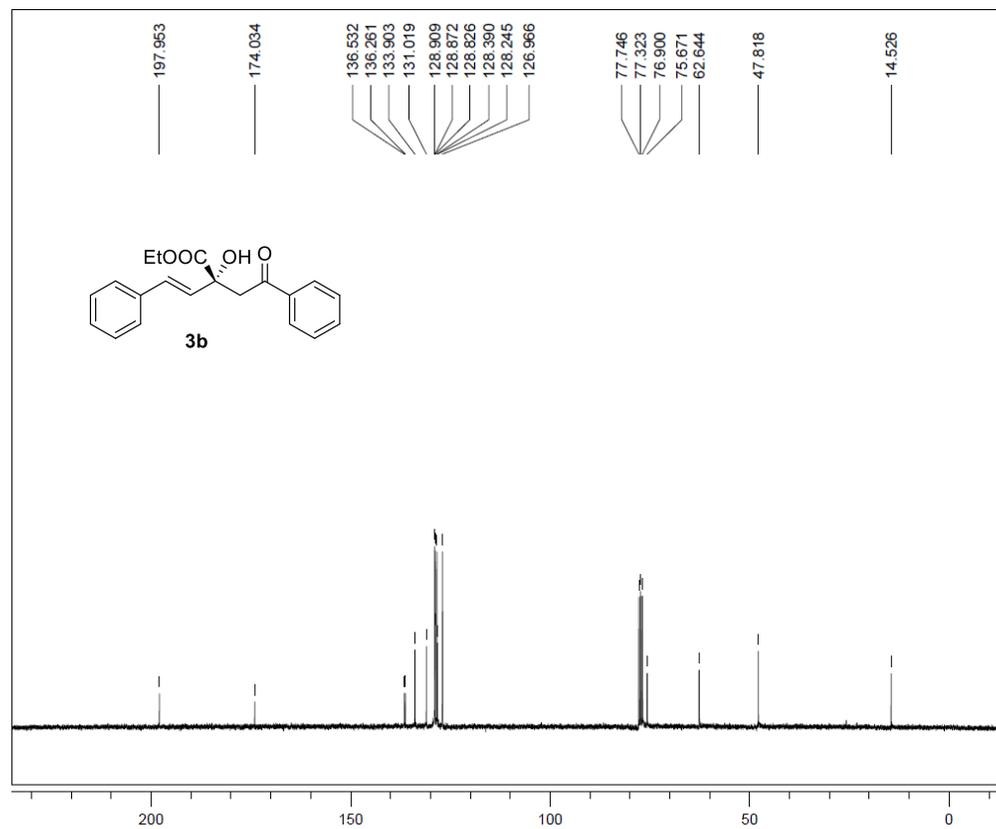
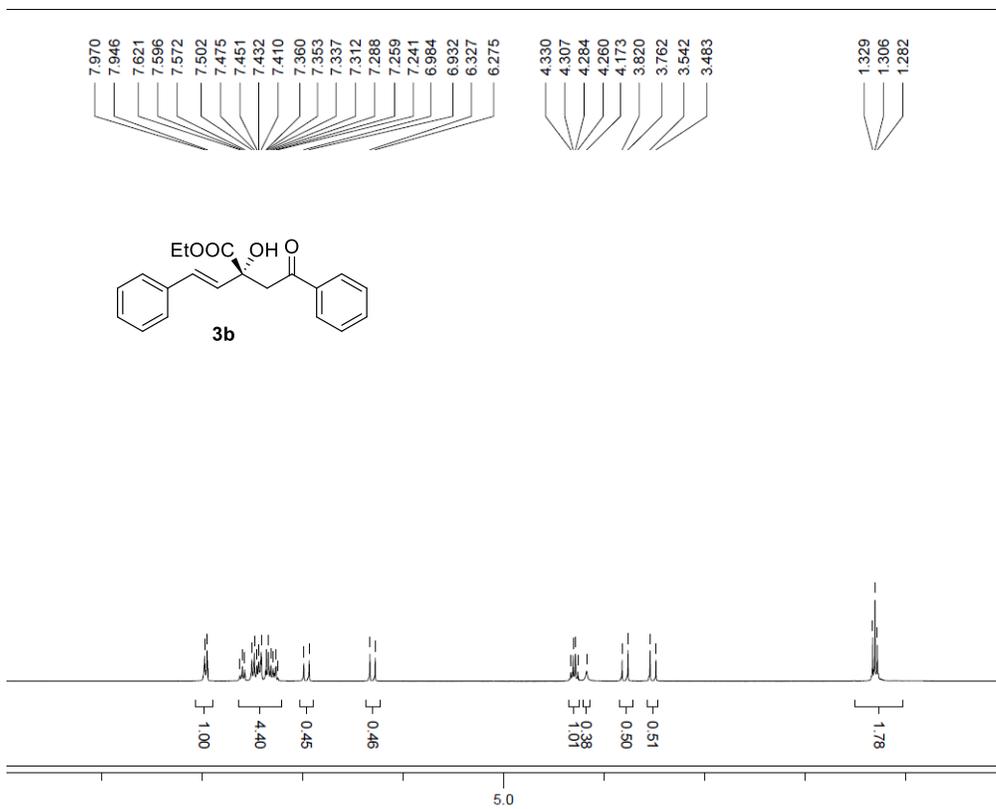
Table S-3. Fractional atomic coordinates and isotropic or equivalent isotropic displacement parameters (\AA^2) for structure 22d

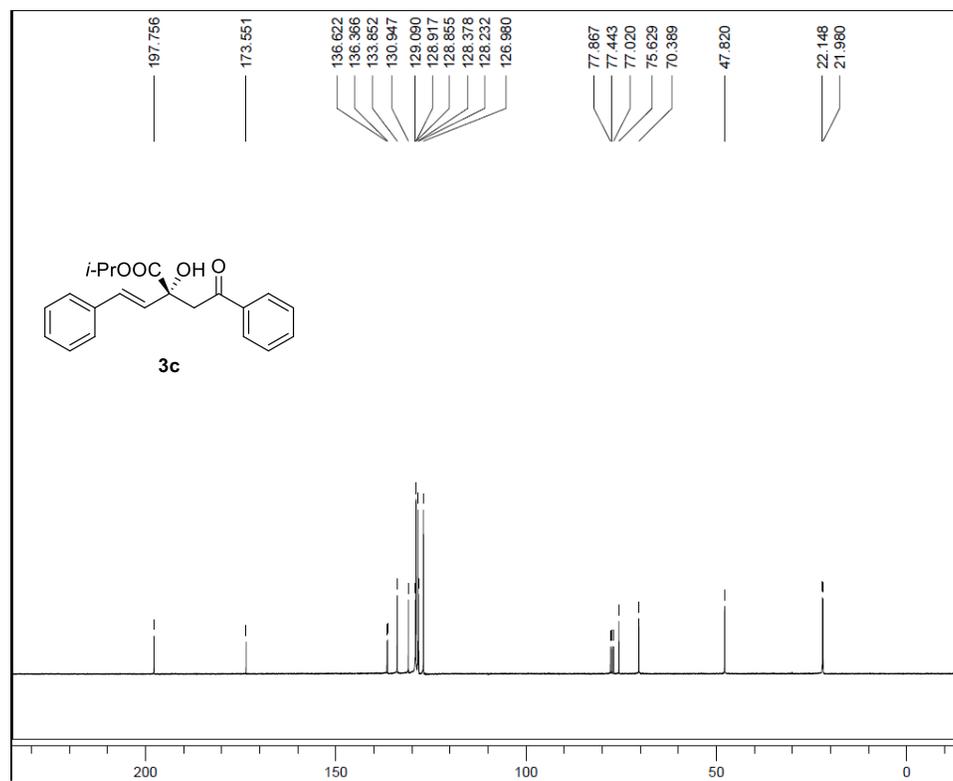
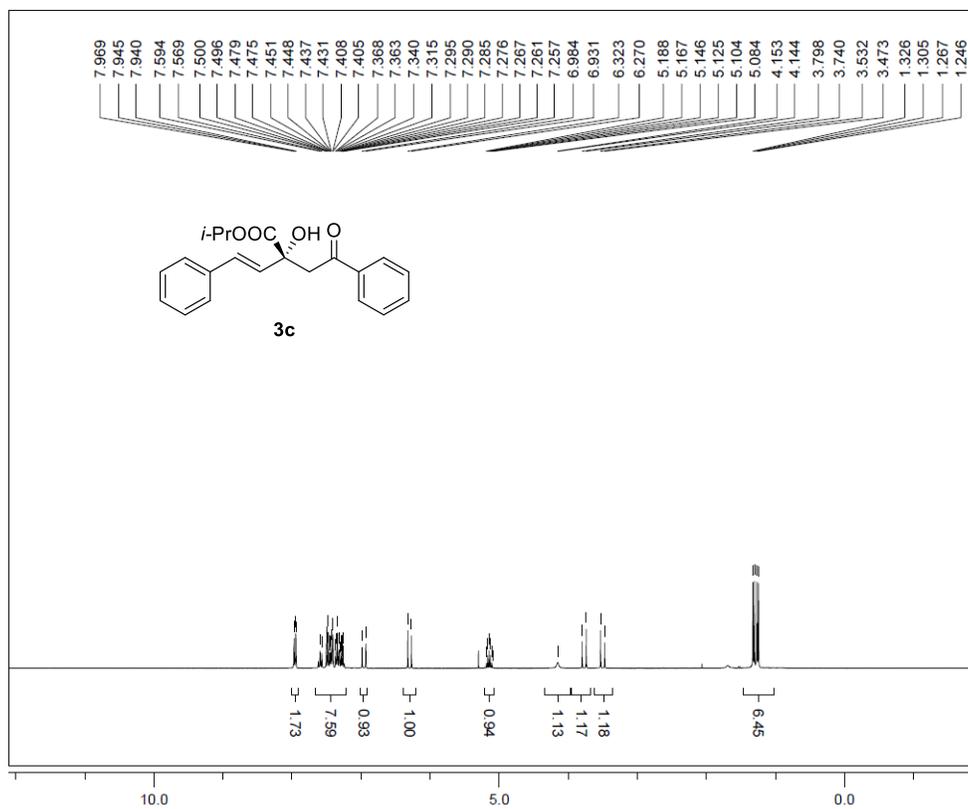
	<i>x</i>	<i>y</i>	<i>z</i>	$U_{\text{iso}}^*/U_{\text{eq}}$
Br1	0.35211 (8)	0.84310 (6)	0.10576 (3)	0.02330 (17)
C4	0.3550 (8)	0.7555 (5)	0.0064 (3)	0.0163 (10)
C3	0.3509 (8)	0.8310 (5)	-0.0622 (3)	0.0184 (9)
C2	0.3538 (8)	0.7669 (5)	-0.1357 (3)	0.0168 (10)
C8	0.3456 (8)	0.6276 (5)	-0.2949 (3)	0.0167 (9)
H8A	0.4342	0.6993	-0.2977	0.020*
C7	0.3770 (7)	0.5562 (5)	-0.2159 (3)	0.0185 (10)
O2	0.3849 (5)	0.5397 (4)	-0.3583 (2)	0.0206 (8)
C1	0.3611 (7)	0.6290 (5)	-0.1394 (3)	0.0161 (9)
C6	0.3678 (8)	0.5562 (5)	-0.0688 (3)	0.0171 (10)
O1	0.4227 (6)	0.4406 (4)	-0.2177 (3)	0.0231 (8)
C9	0.1587 (8)	0.6908 (5)	-0.3075 (3)	0.0159 (9)
C10	0.1502 (8)	0.7583 (5)	-0.3894 (3)	0.0194 (10)
H10A	0.2349	0.8313	-0.3902	0.023*
H10B	0.1873	0.6955	-0.4296	0.023*
C14	0.0068 (7)	0.5910 (5)	-0.2979 (3)	0.0153 (11)
C12	-0.1763 (7)	0.6987 (5)	-0.4051 (3)	0.0198 (11)
H12A	-0.1461	0.6306	-0.4433	0.024*
H12B	-0.2951	0.7324	-0.4180	0.024*
C5	0.3625 (9)	0.6189 (5)	0.0046 (3)	0.0203 (11)
C13	-0.1789 (7)	0.6393 (6)	-0.3216 (3)	0.0190 (11)
H13A	-0.2195	0.7055	-0.2840	0.023*
H13B	-0.2632	0.5661	-0.3200	0.023*
C11	-0.0386 (7)	0.8107 (5)	-0.4103 (3)	0.0192 (11)
H11A	-0.0716	0.8808	-0.3739	0.023*
H11B	-0.0378	0.8469	-0.4636	0.023*
O3	0.0306 (5)	0.4809 (4)	-0.2716 (3)	0.0268 (9)
H6A	0.374 (10)	0.463 (7)	-0.071 (4)	0.032*
H5A	0.370 (11)	0.566 (7)	0.051 (4)	0.032*
H2A	0.339 (11)	0.827 (7)	-0.187 (4)	0.032*
H3A	0.332 (11)	0.932 (7)	-0.059 (4)	0.032*
H2B	0.381 (12)	0.473 (8)	-0.330 (5)	0.040*
H9A	0.140 (11)	0.761 (7)	-0.262 (4)	0.032*

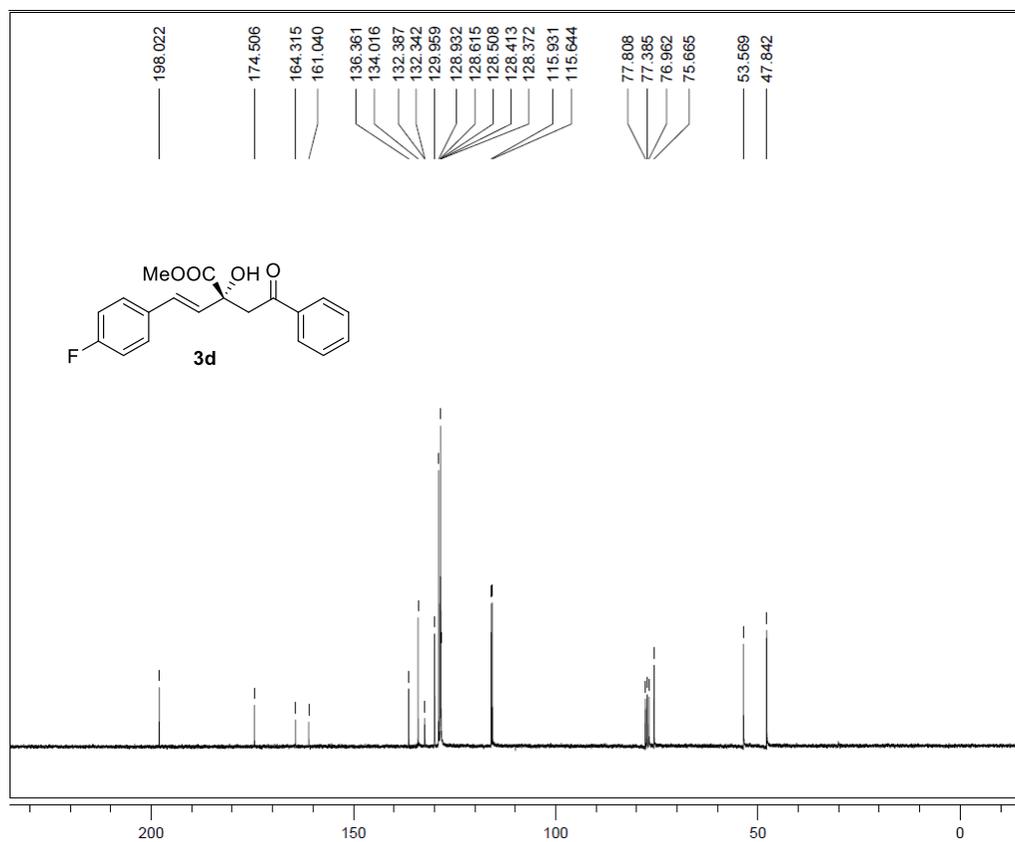
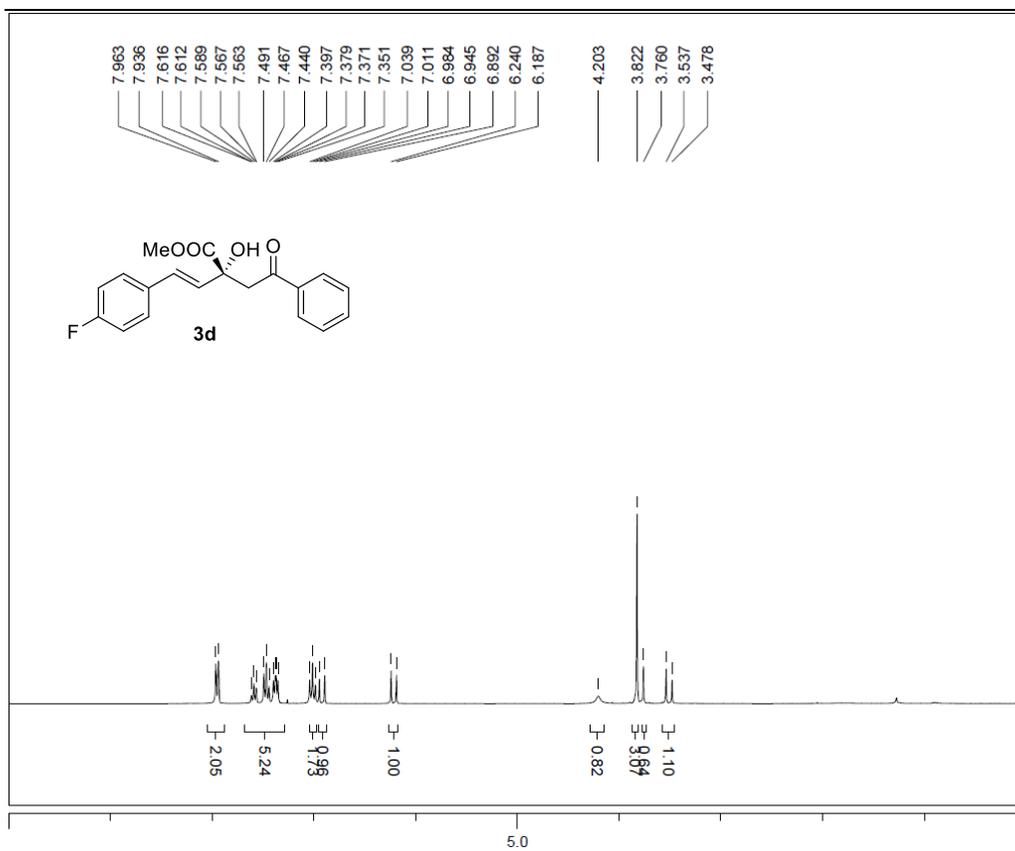
Additional References

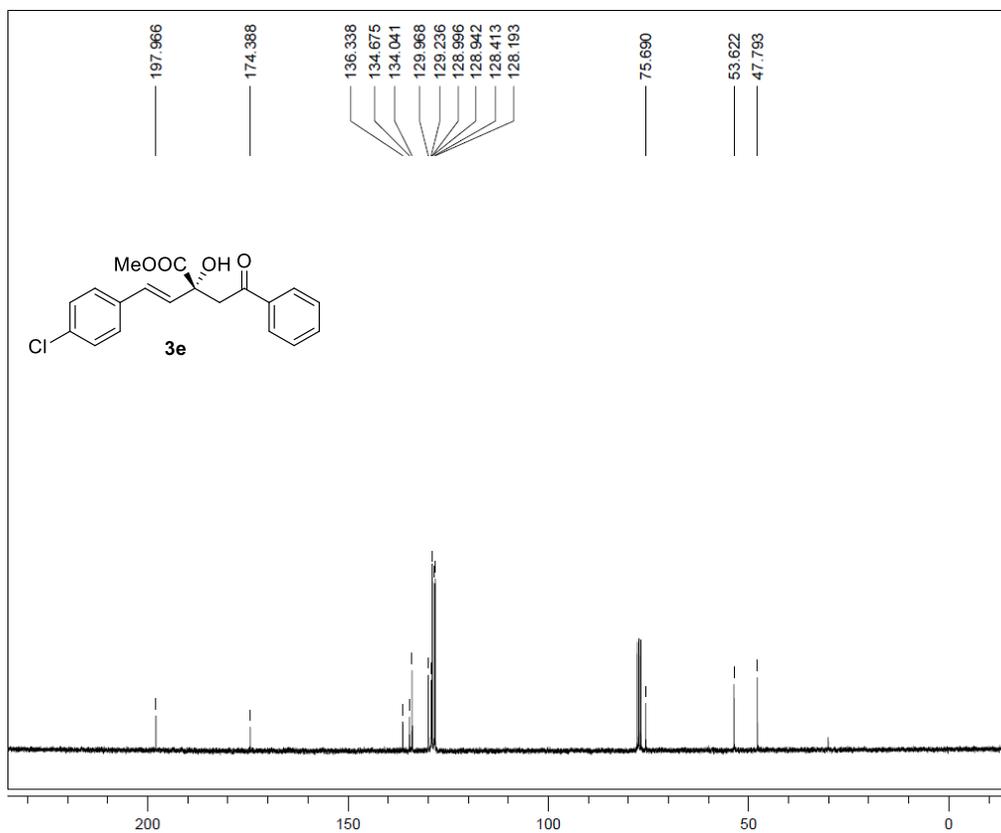
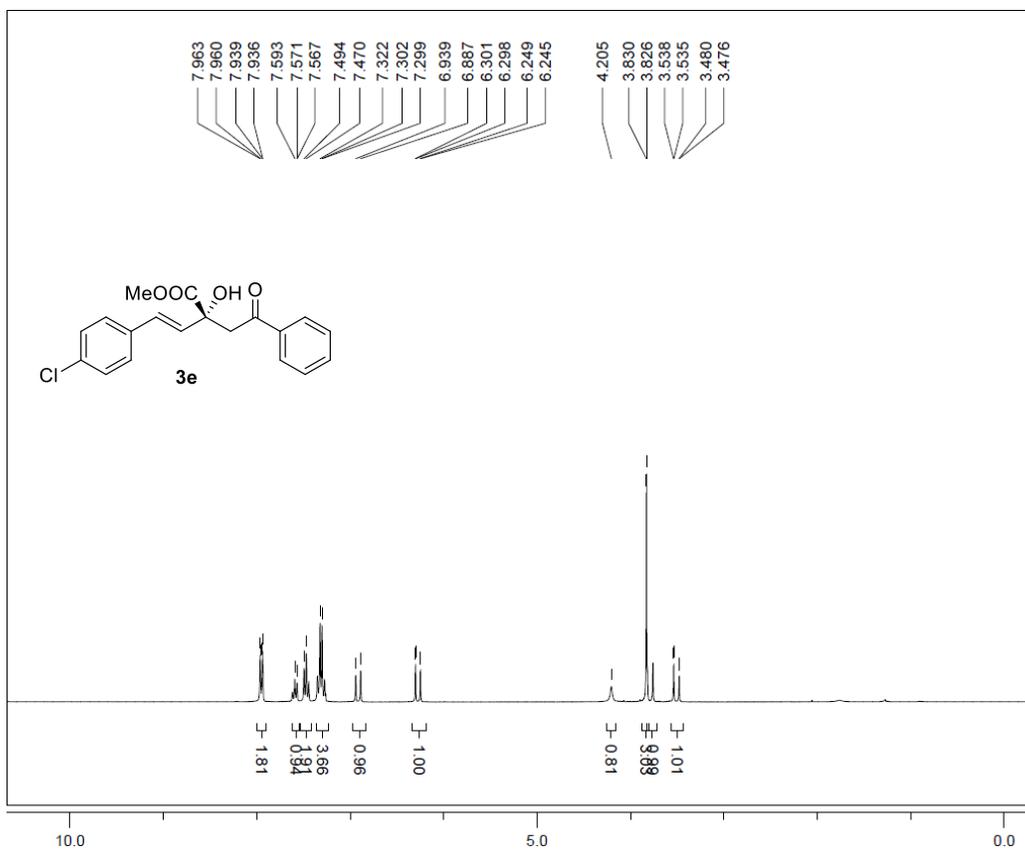
17. *CrystalClear*, User Manual. Rigaku/MSI Inc., Rigaku Corporation, The Woodlands, TX, 2005.
18. Higashi, *ABSCOR*, Rigaku Corporation, Tokyo, Japan, 1995.
19. Sheldrick, G. M. *SHELXS97*. Program for the Solution of Crystal Structures. University of Göttingen, Germany, 1997.
20. Sheldrick, G. M. *SHELXL97*, Program for crystal structure analysis. University of Göttingen, Germany, 1997.

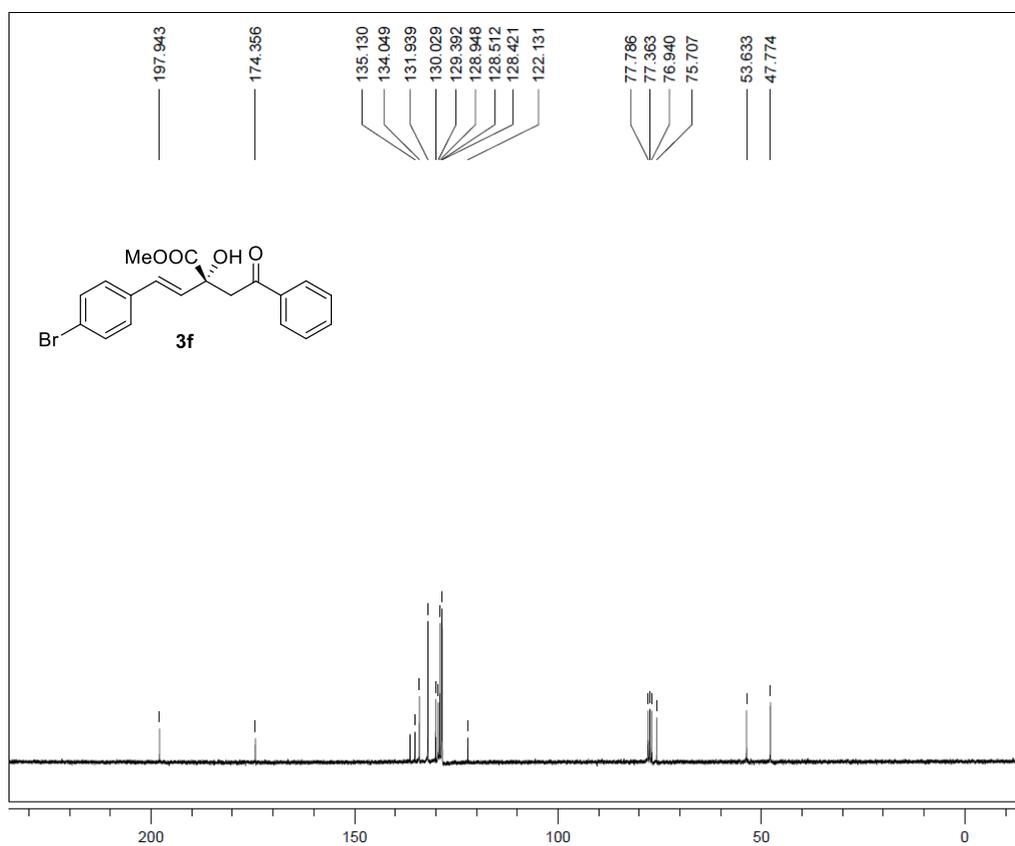
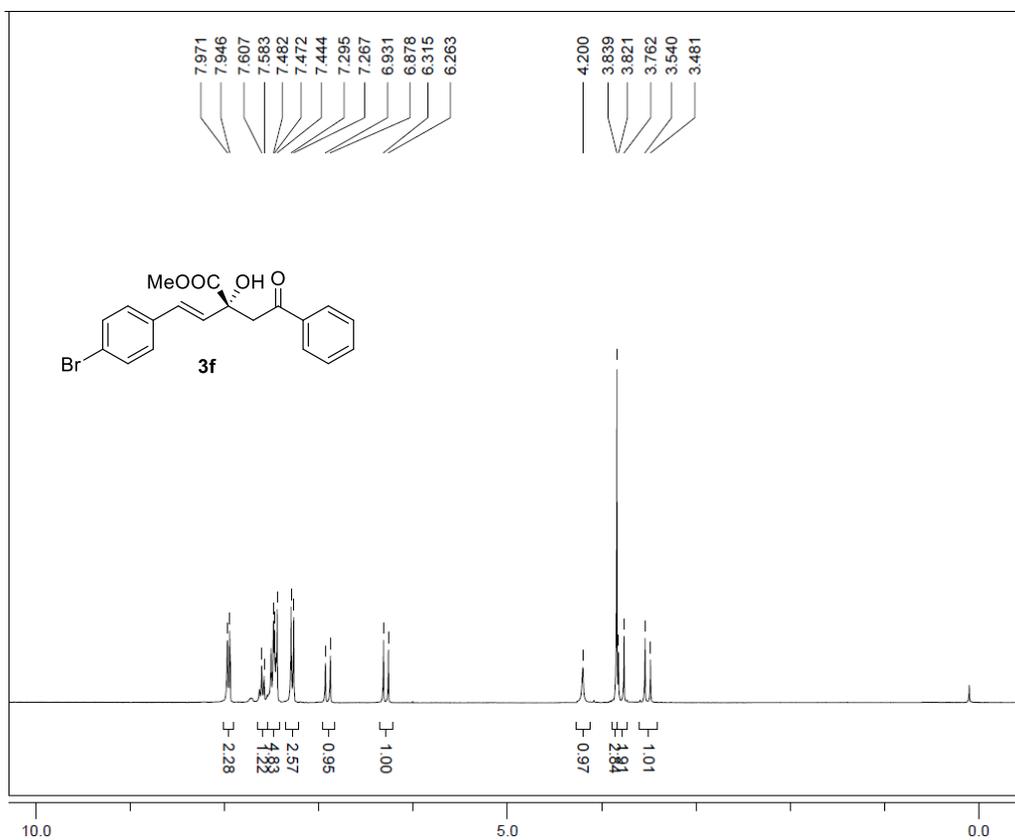


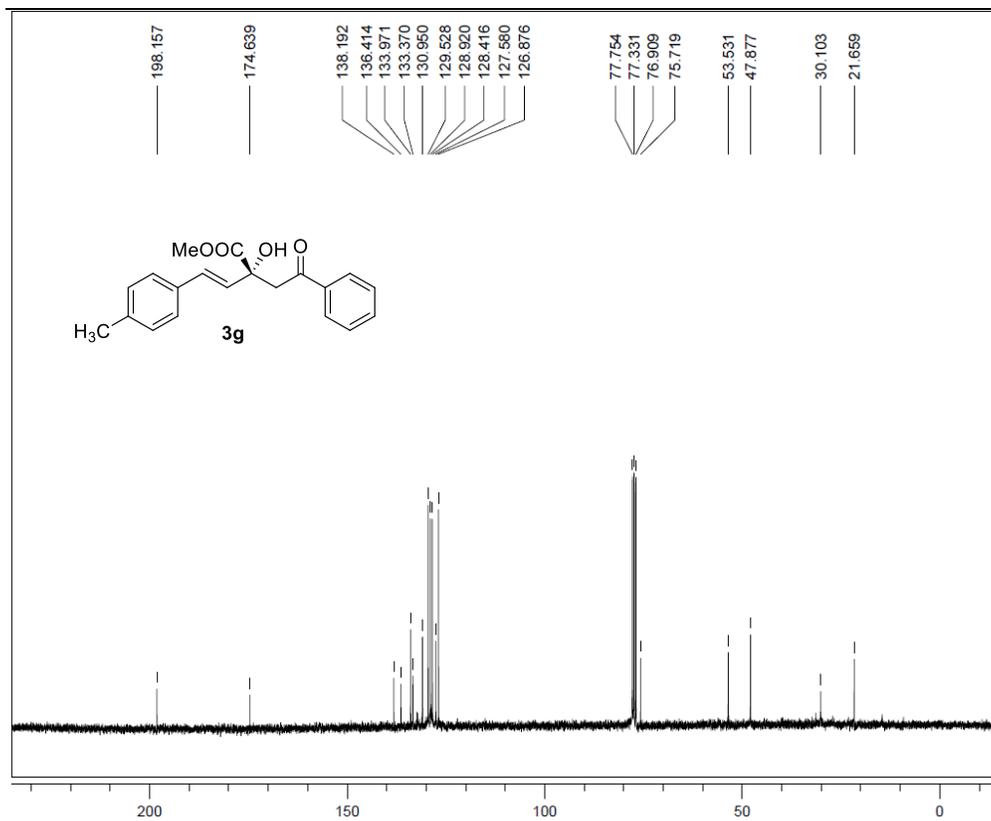
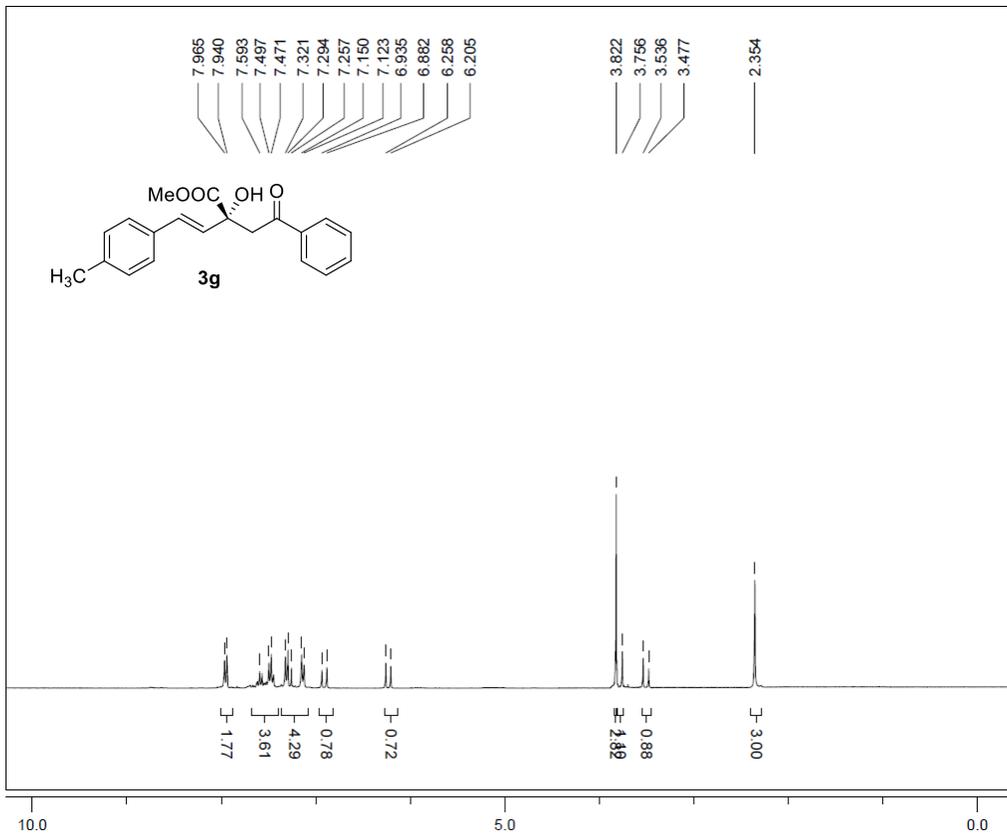


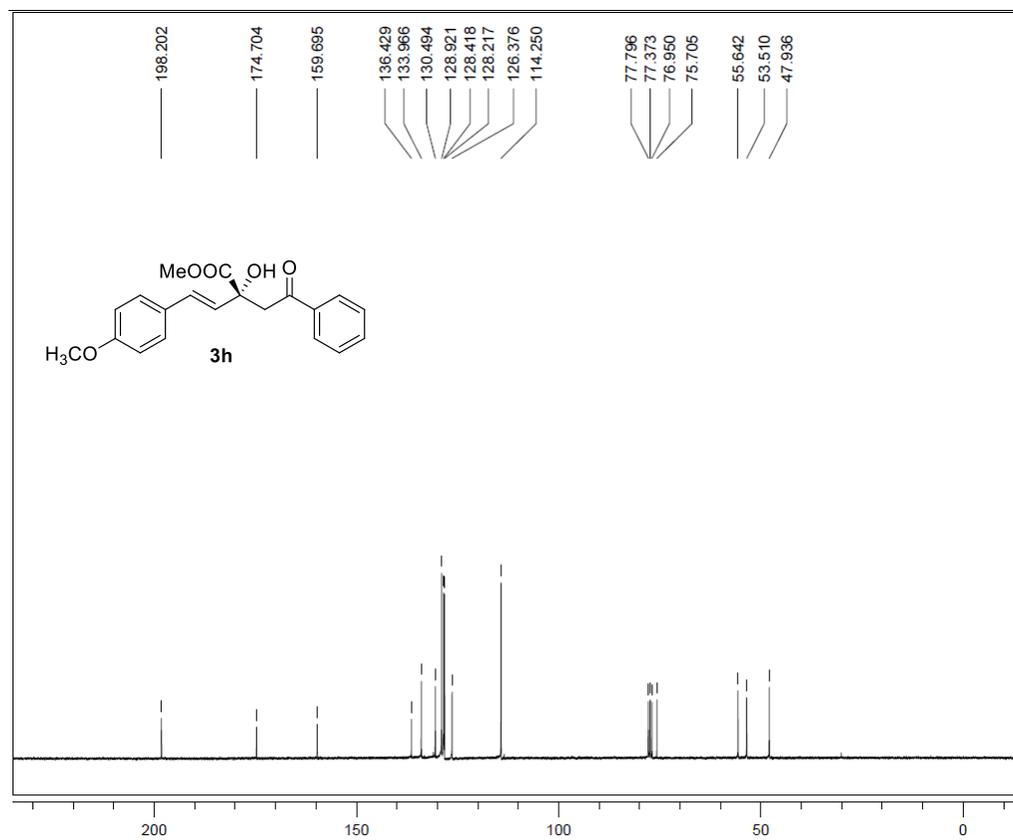
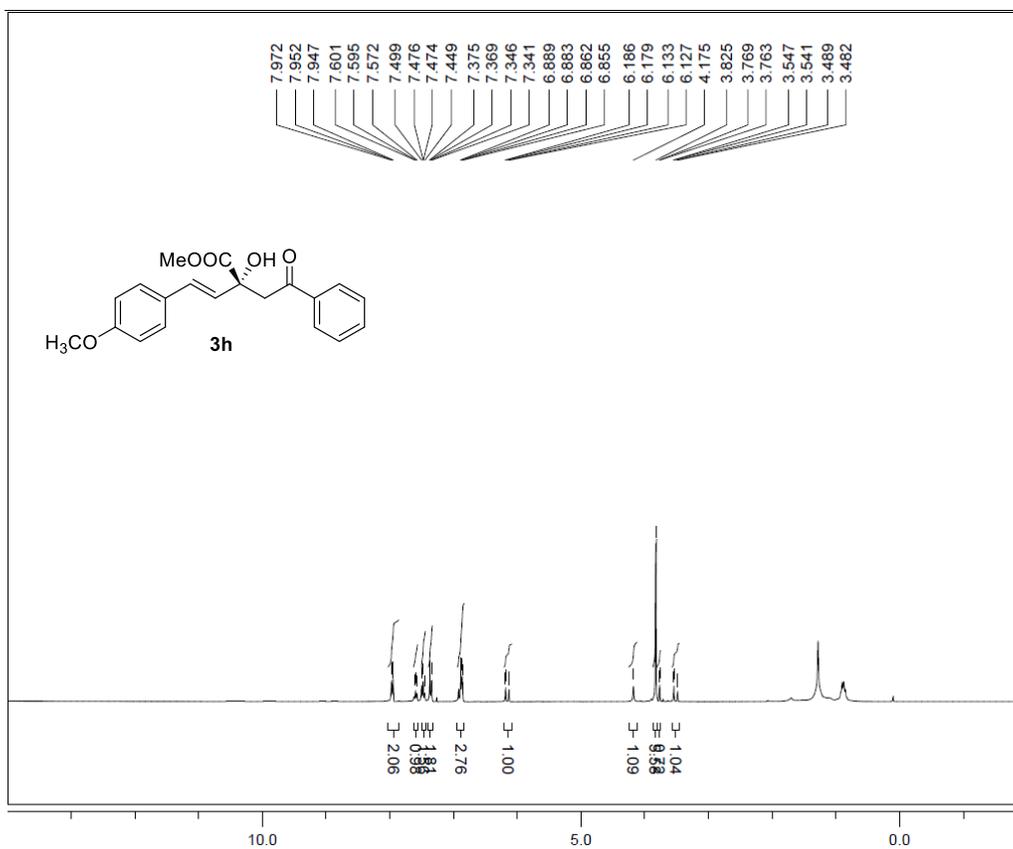


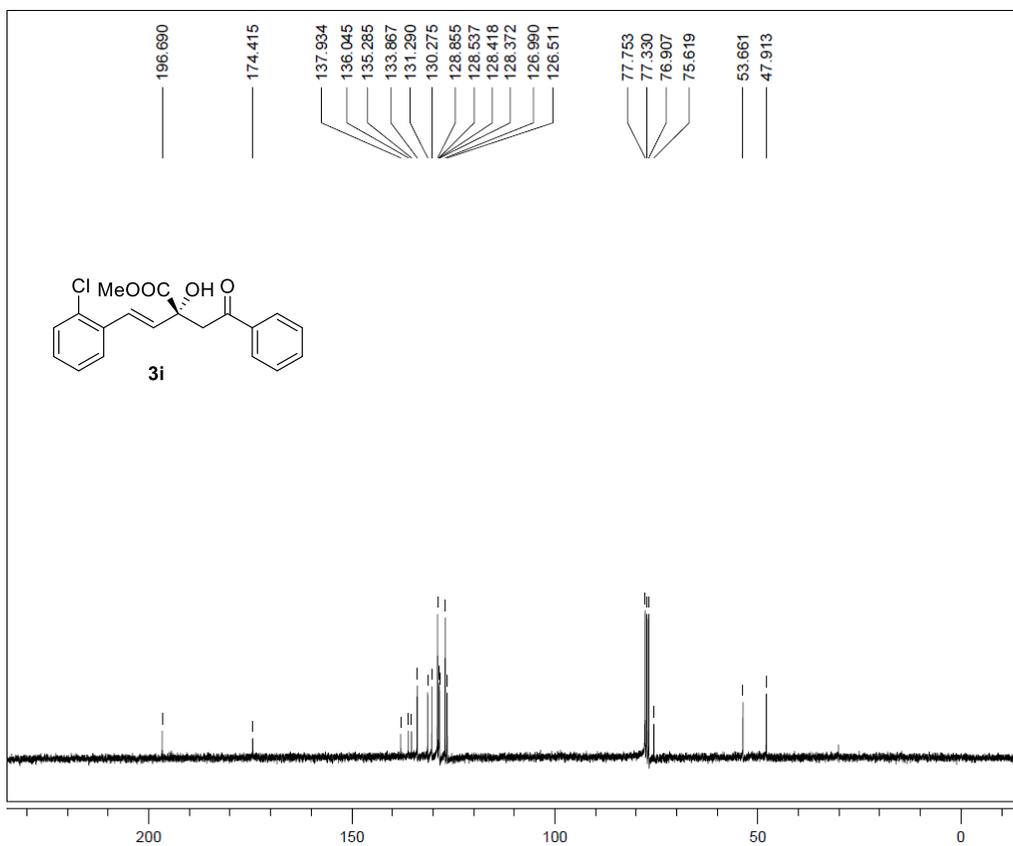
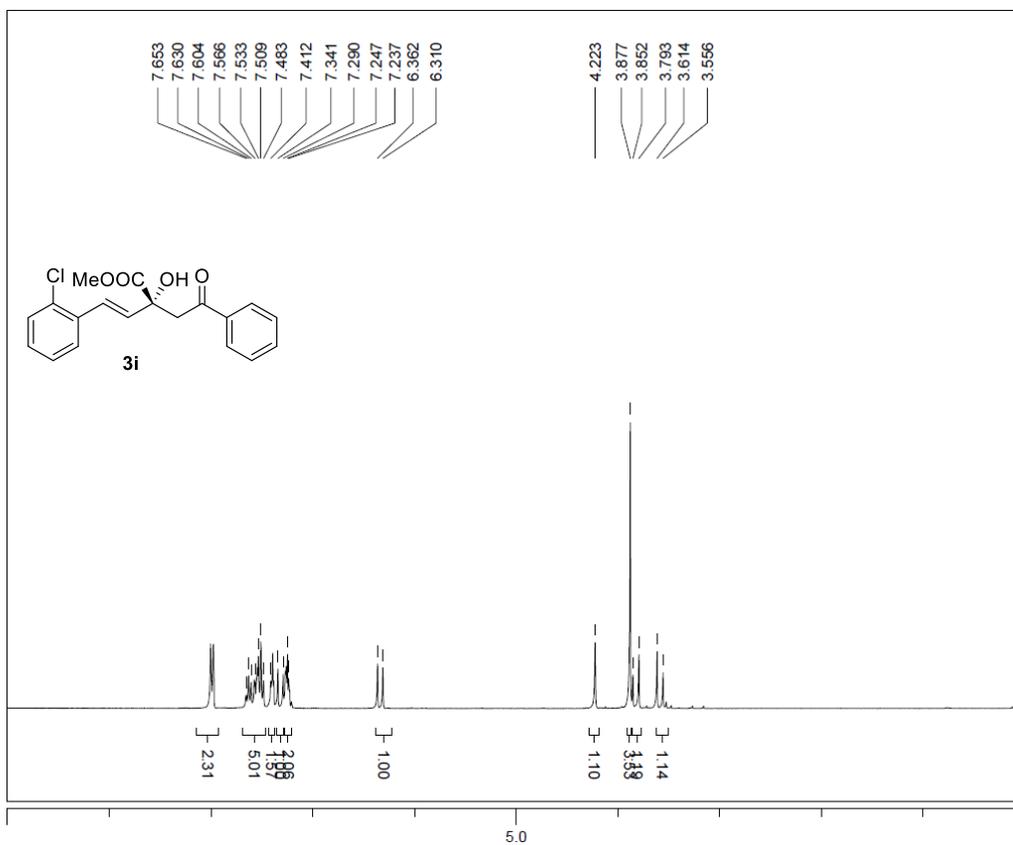


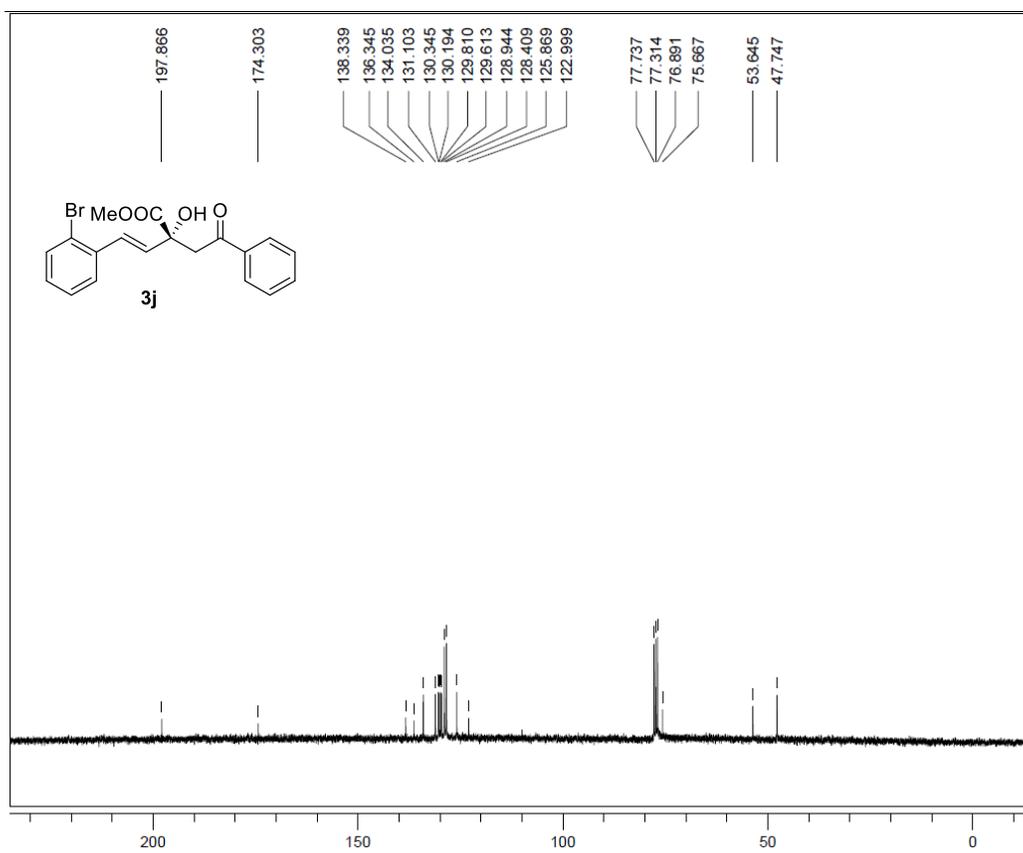
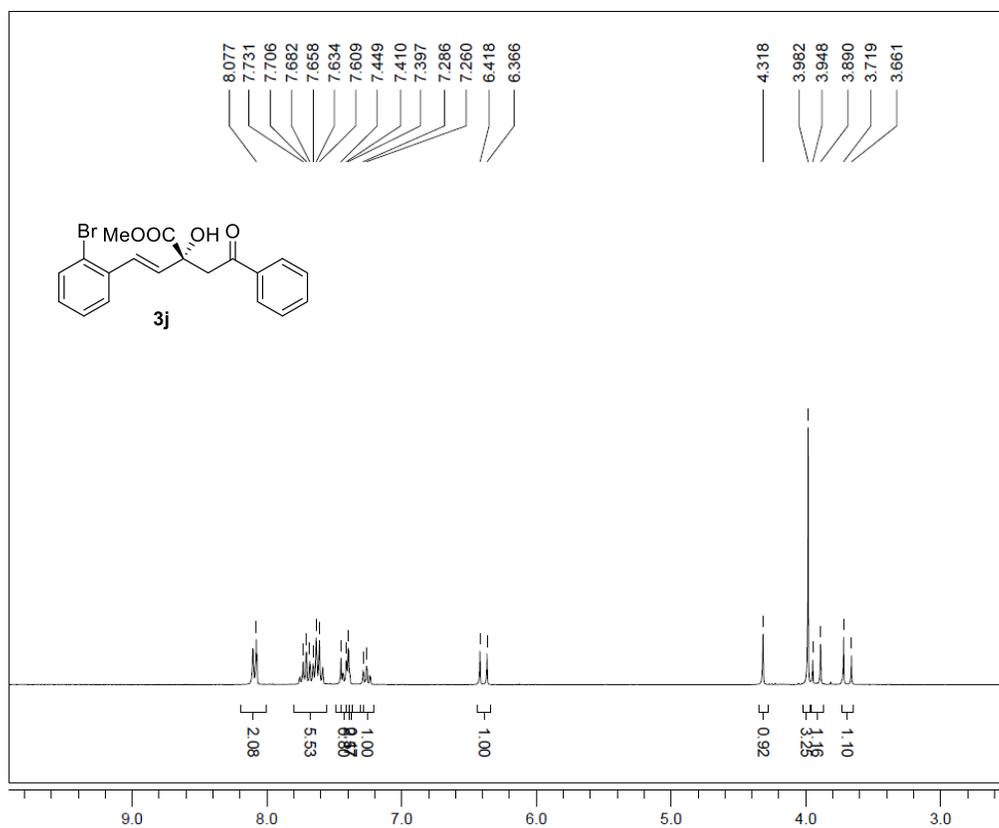


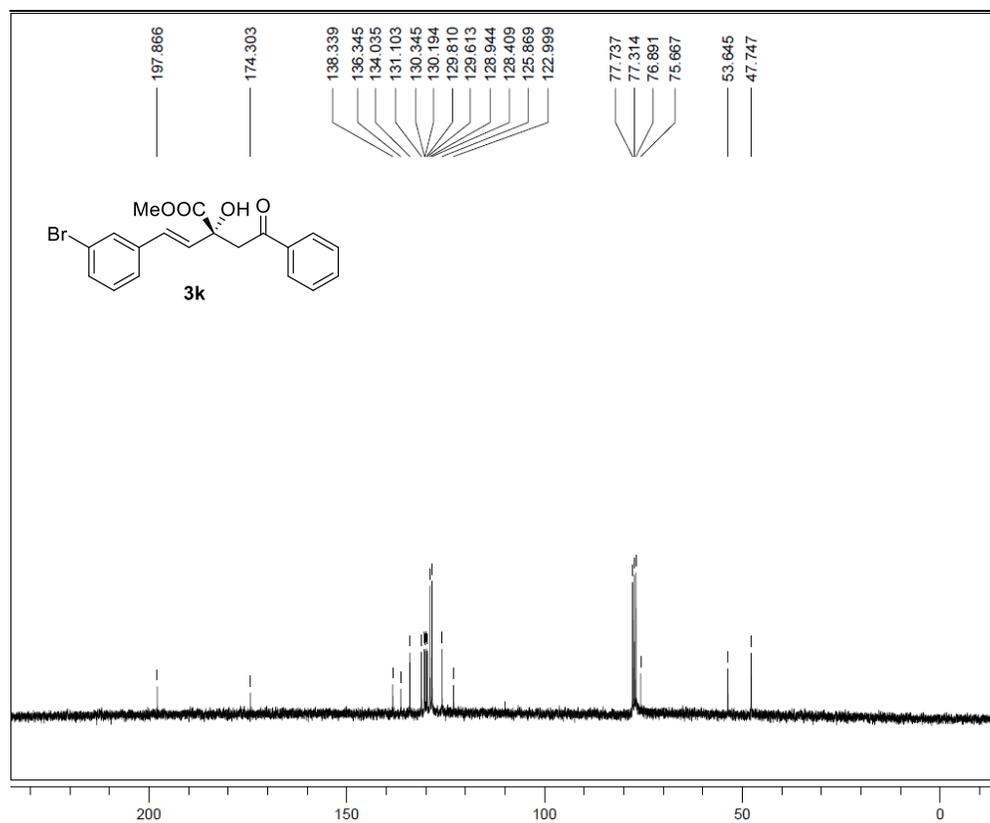
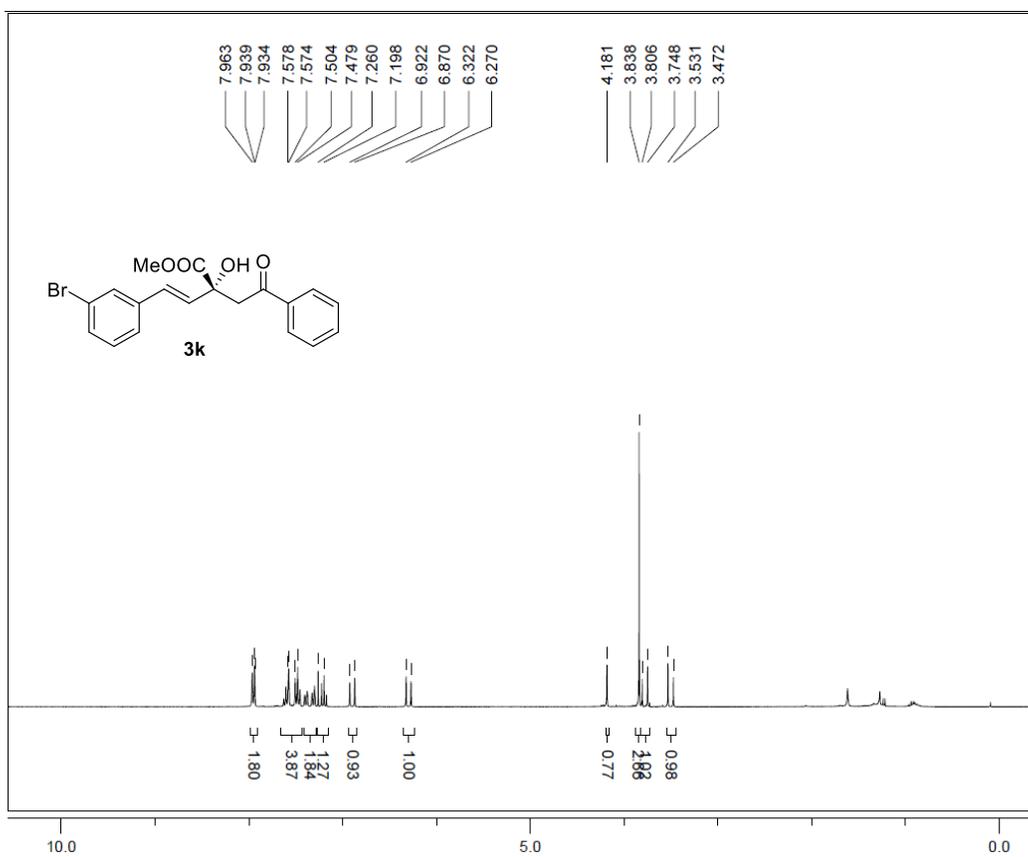


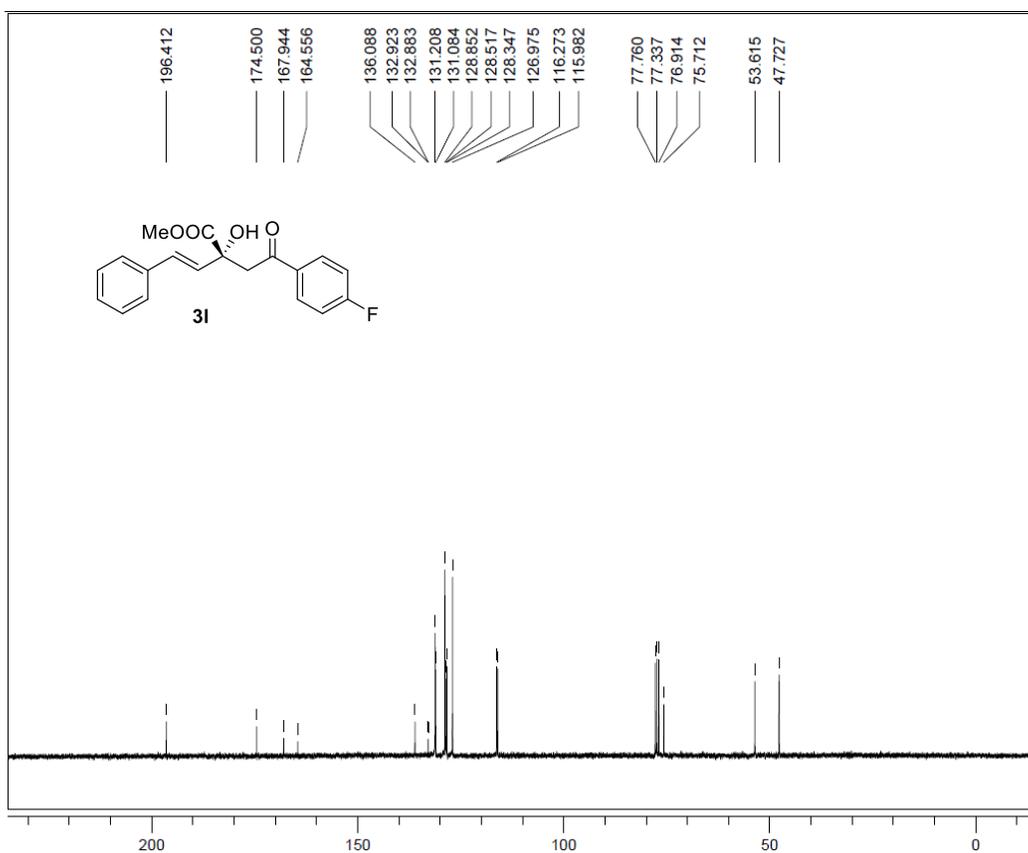
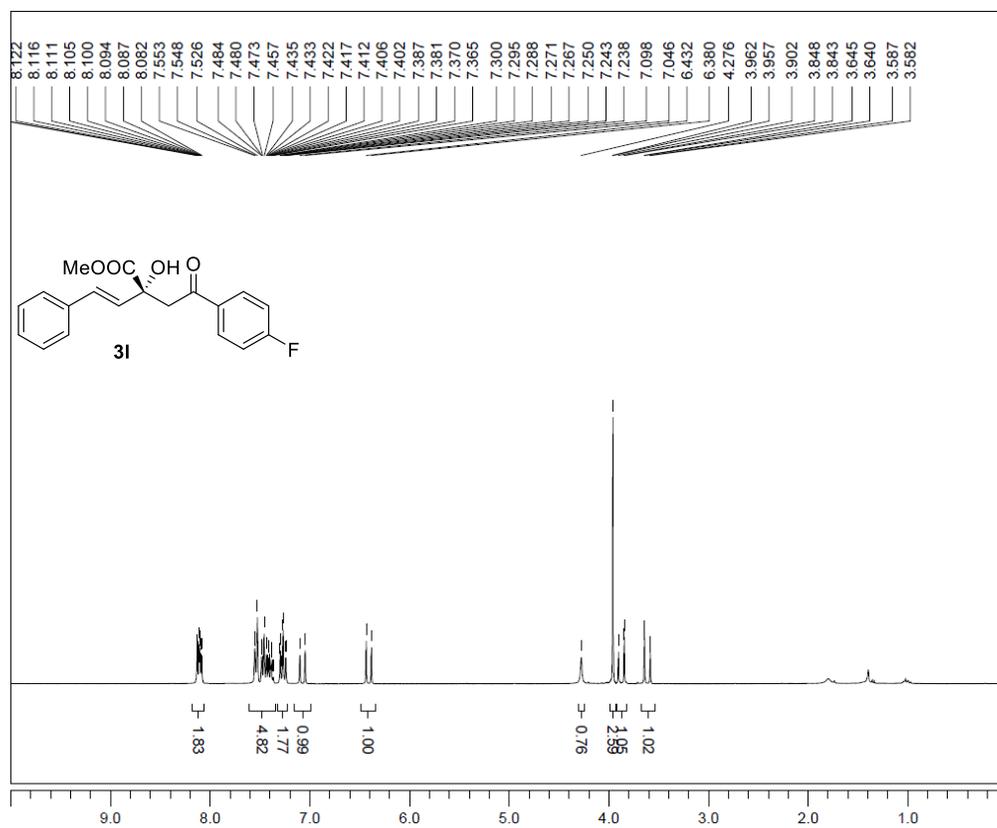


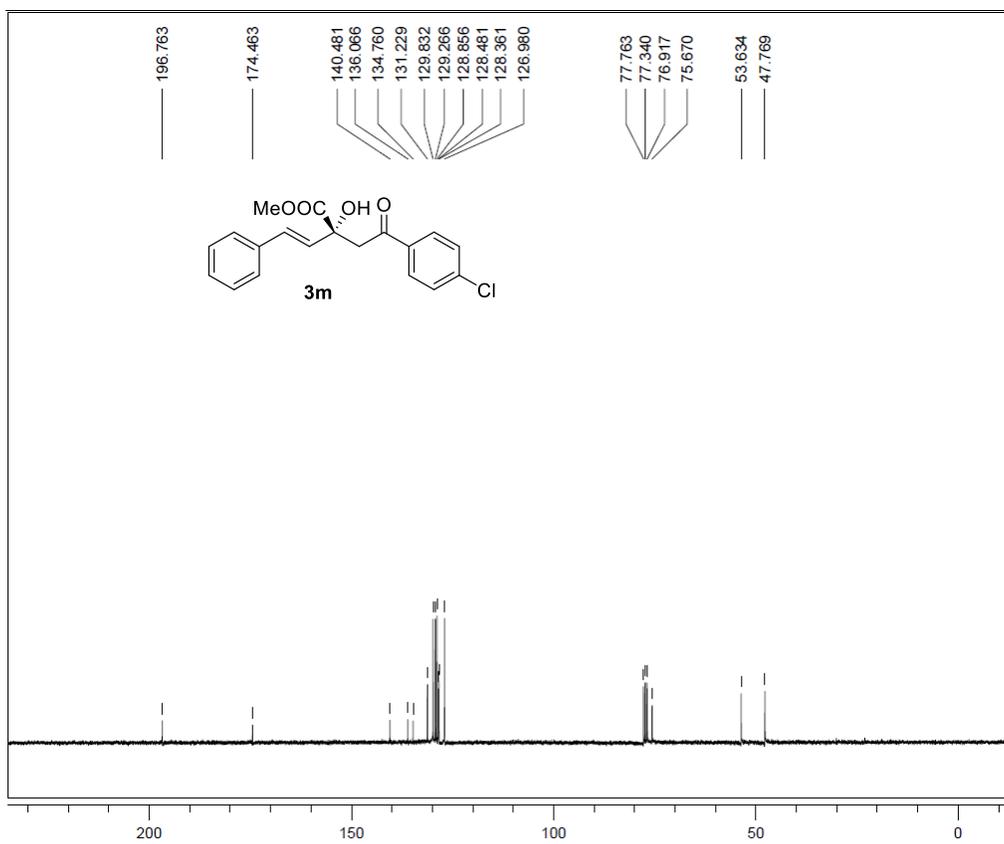
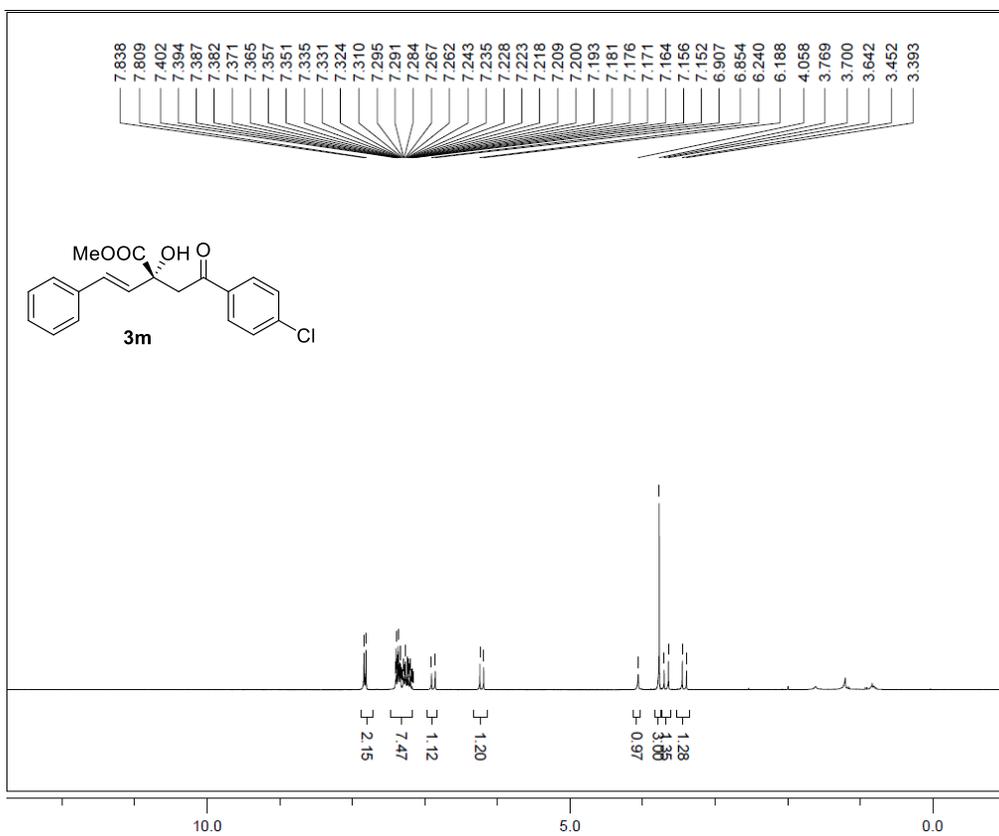


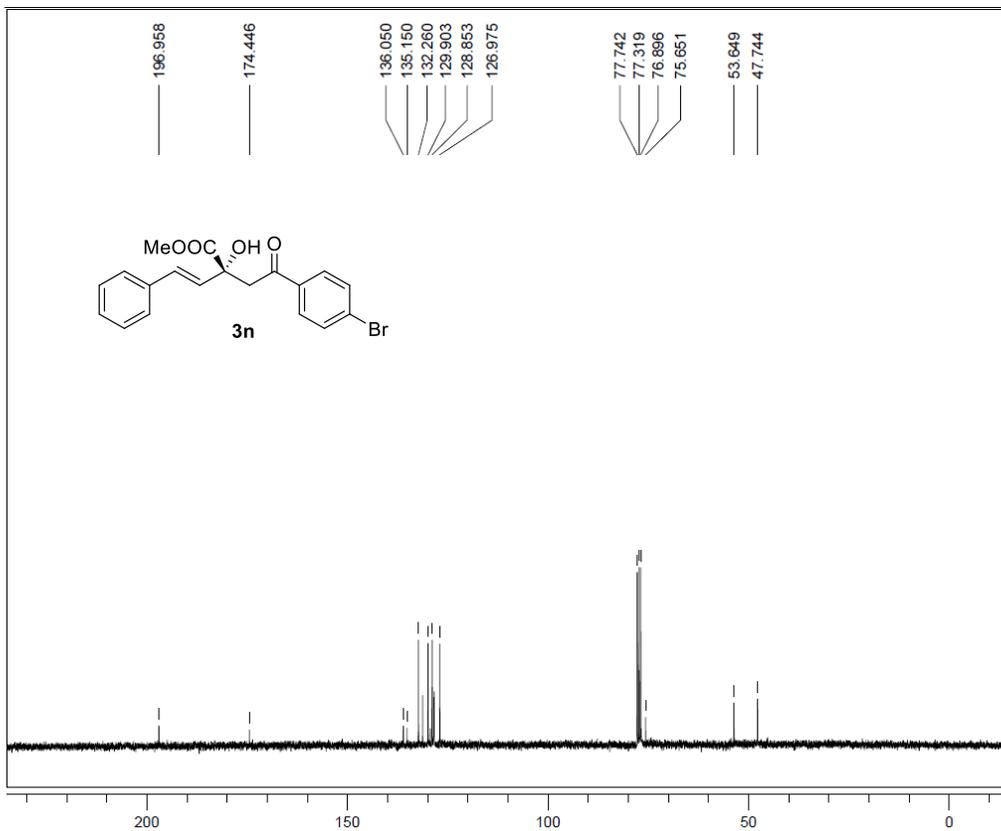
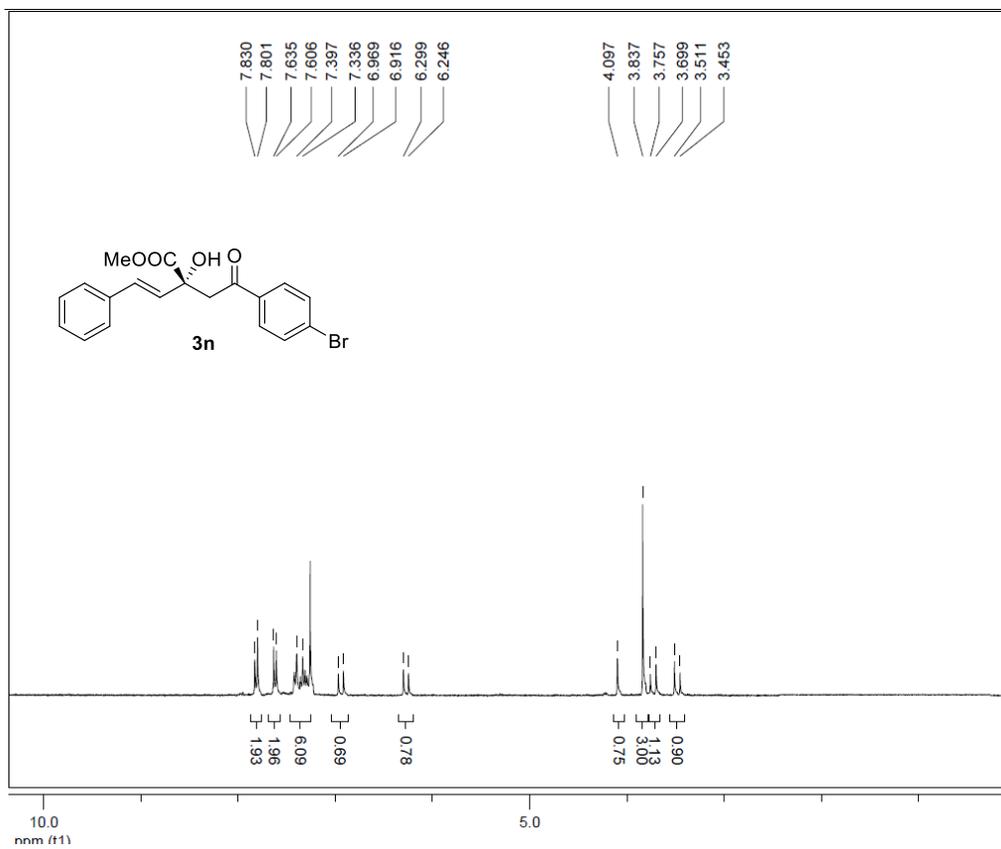


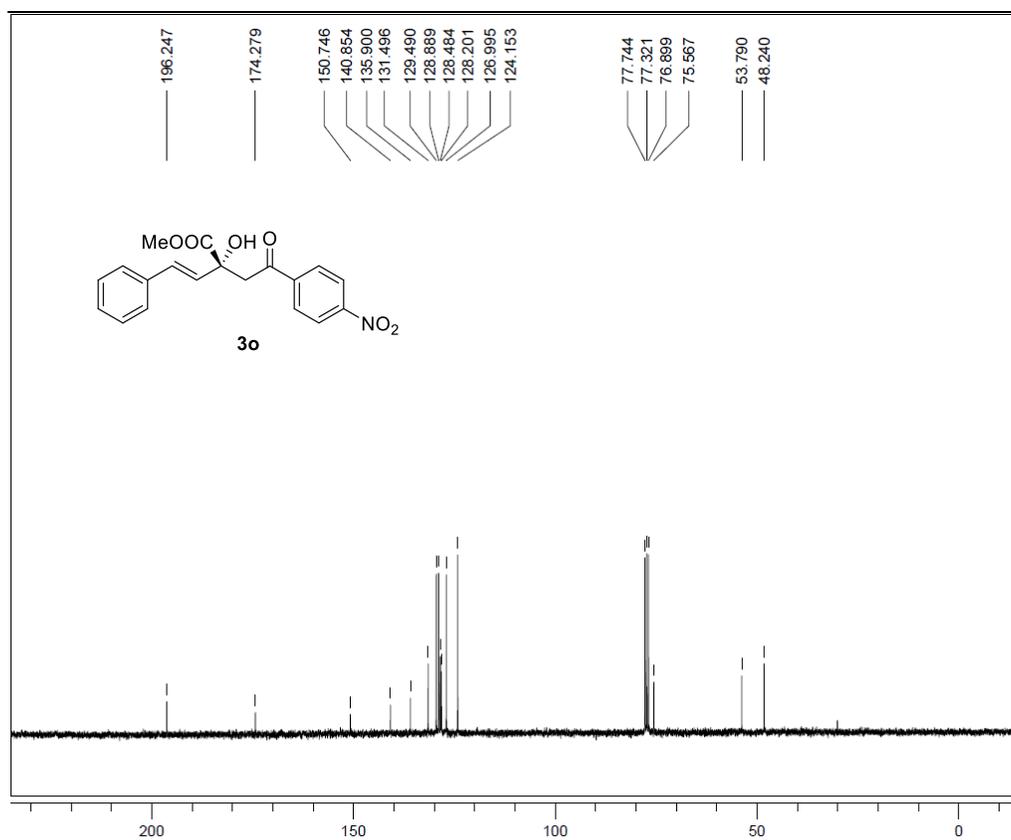
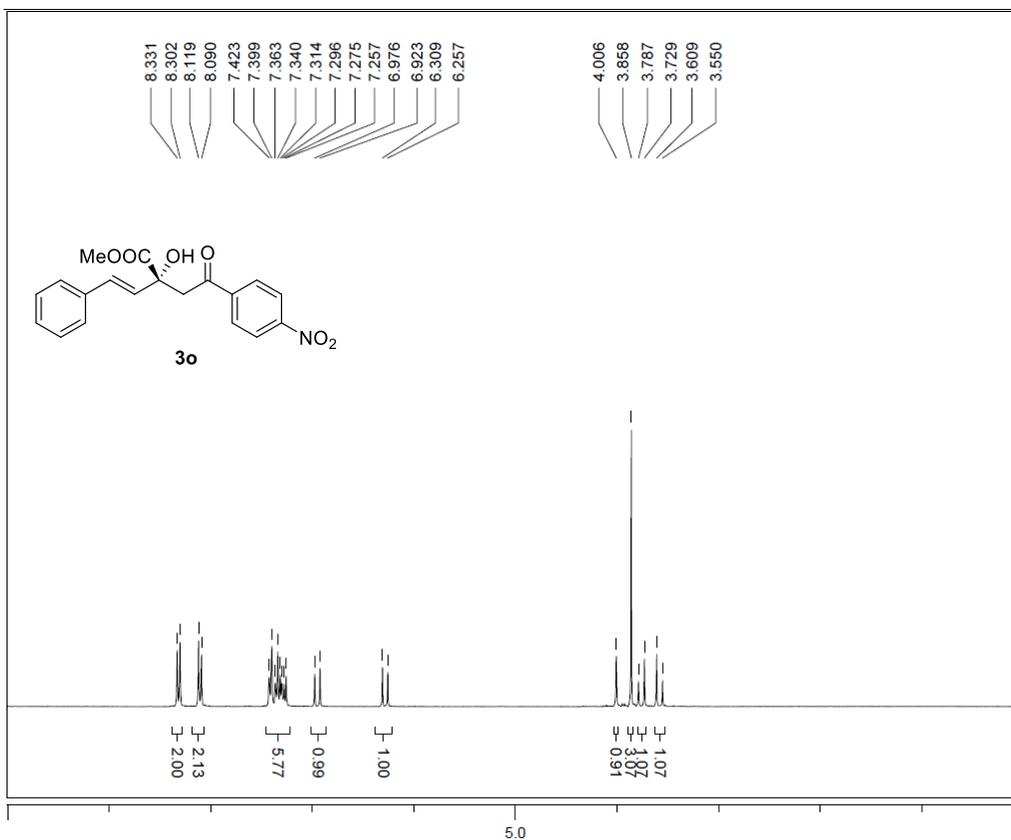


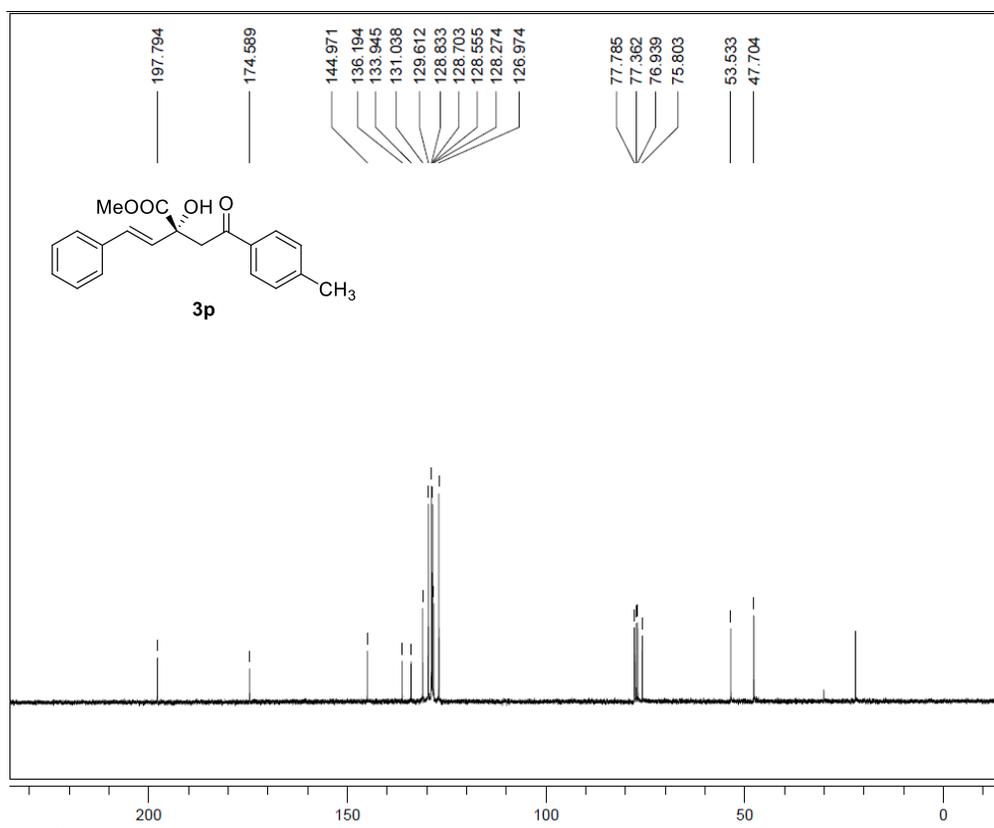
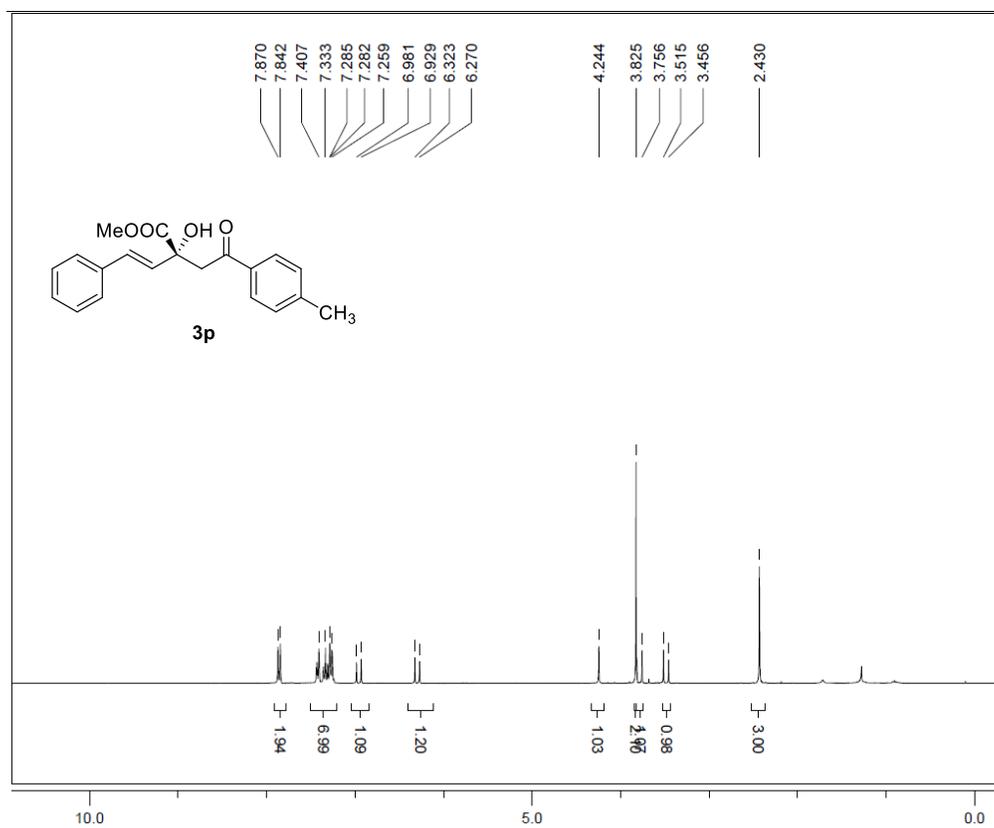


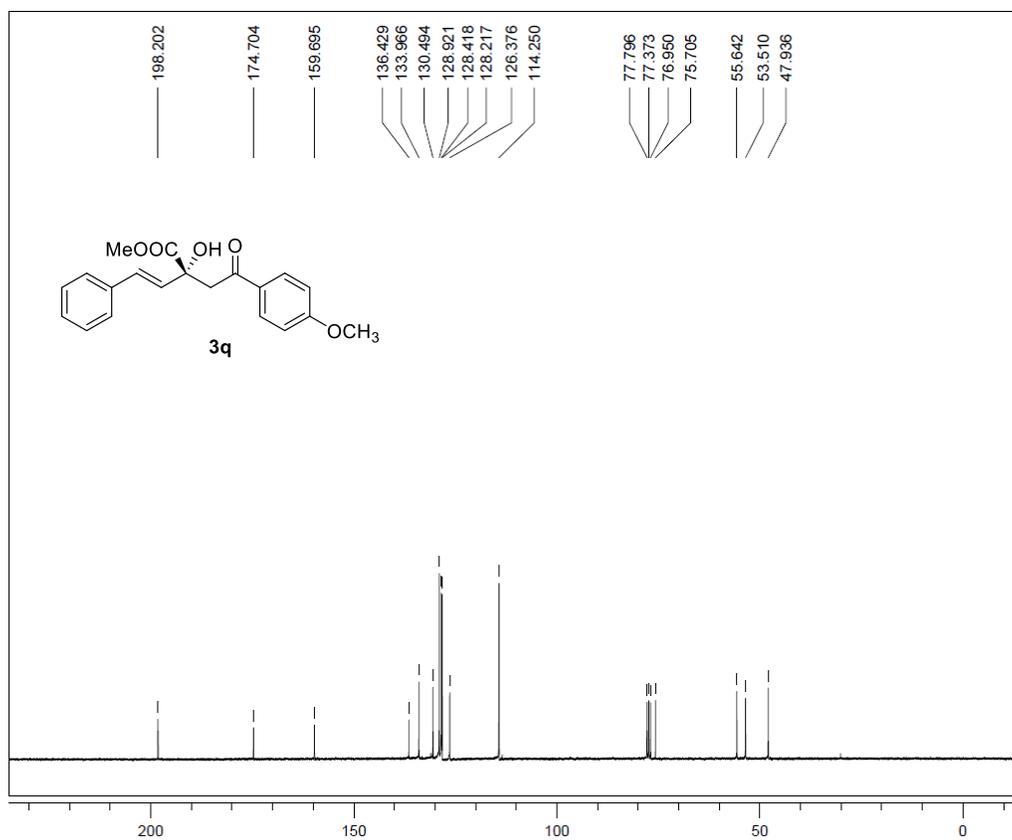
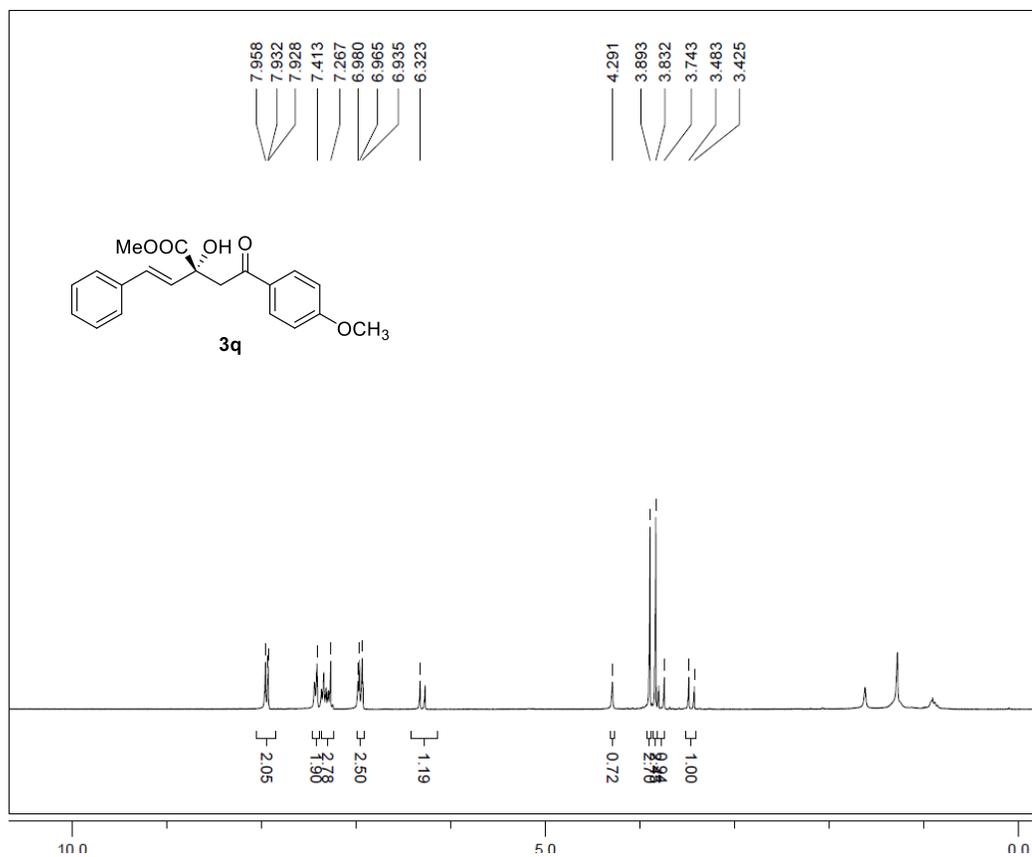


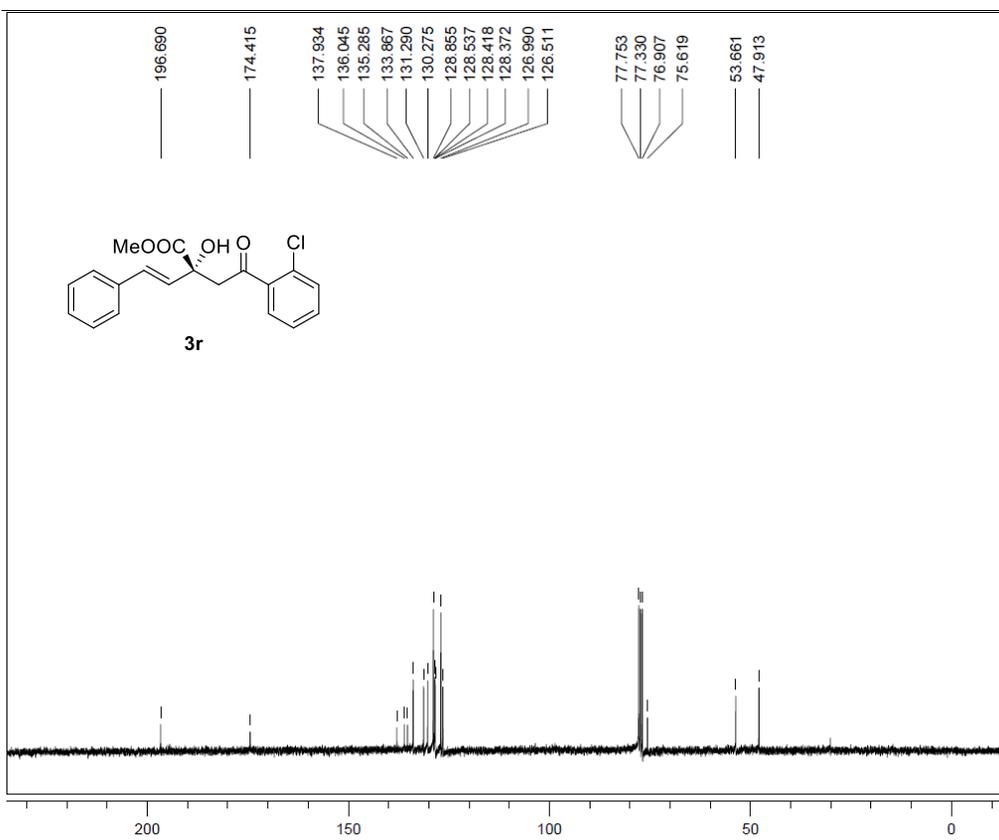
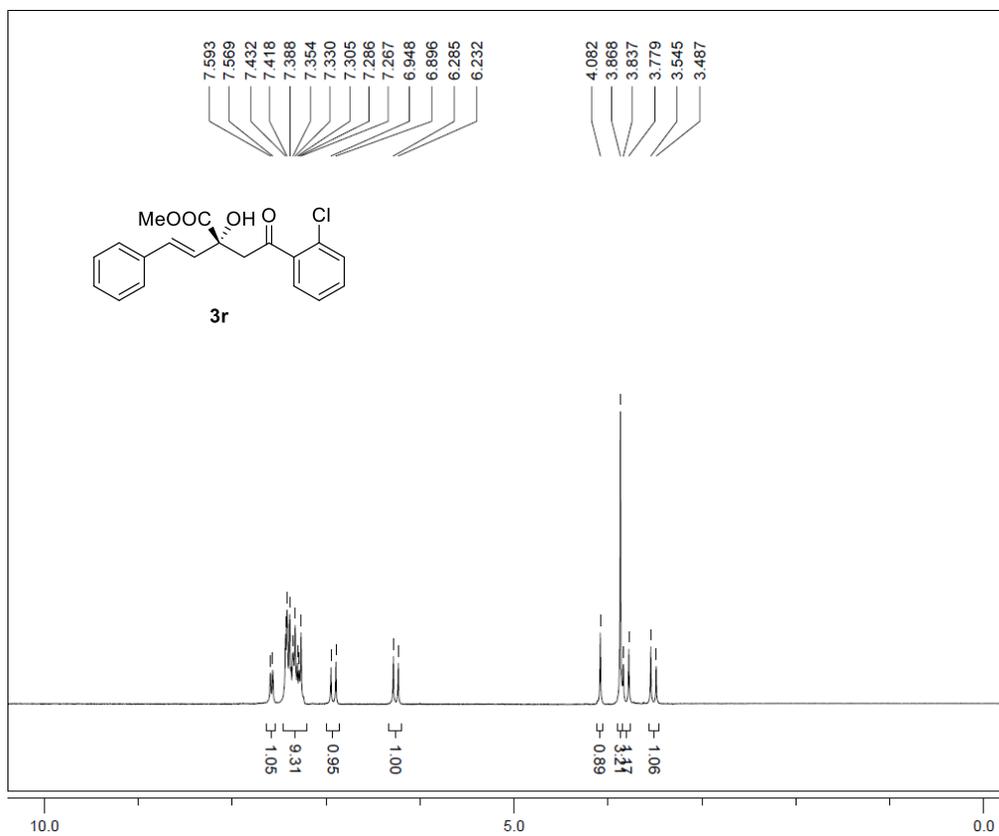


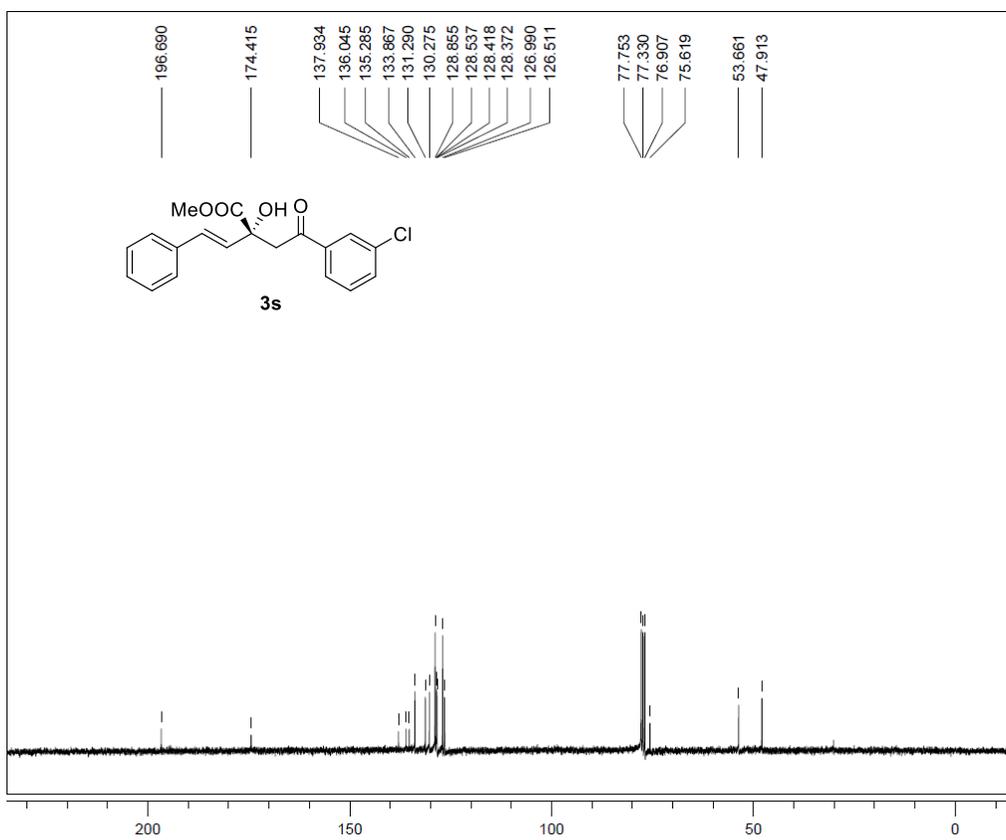
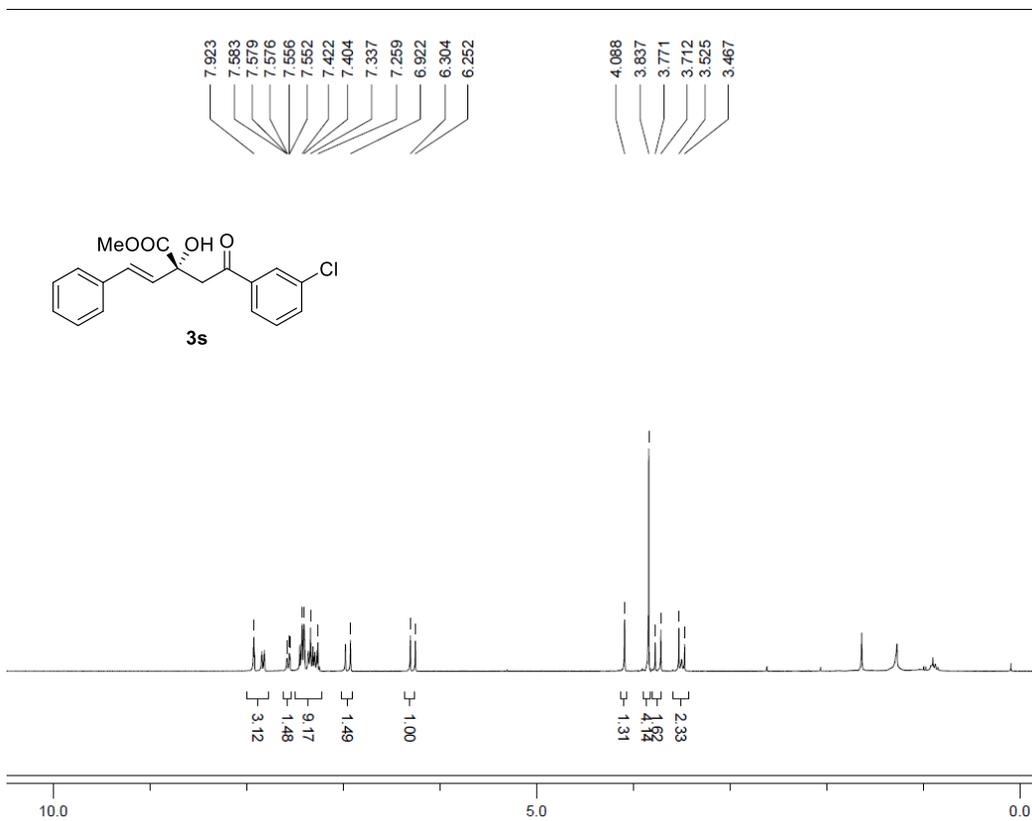




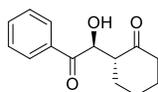




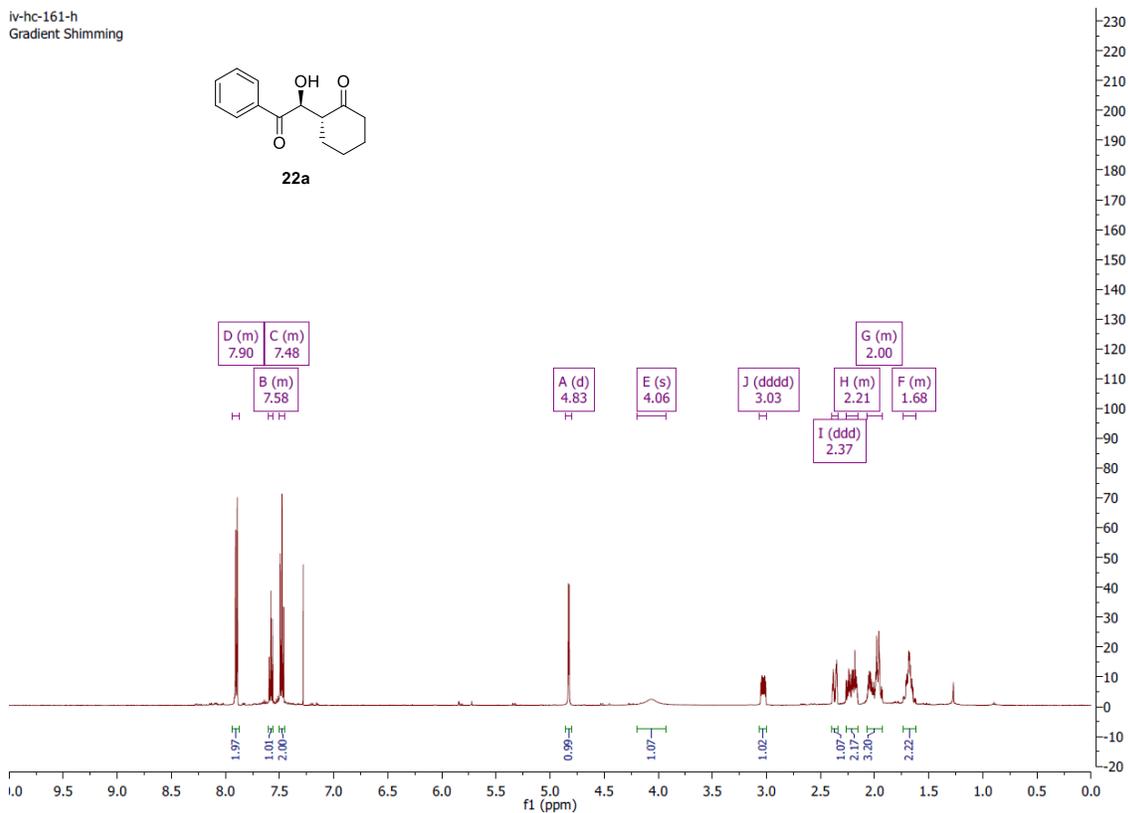




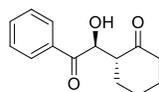
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Gradient Shimming



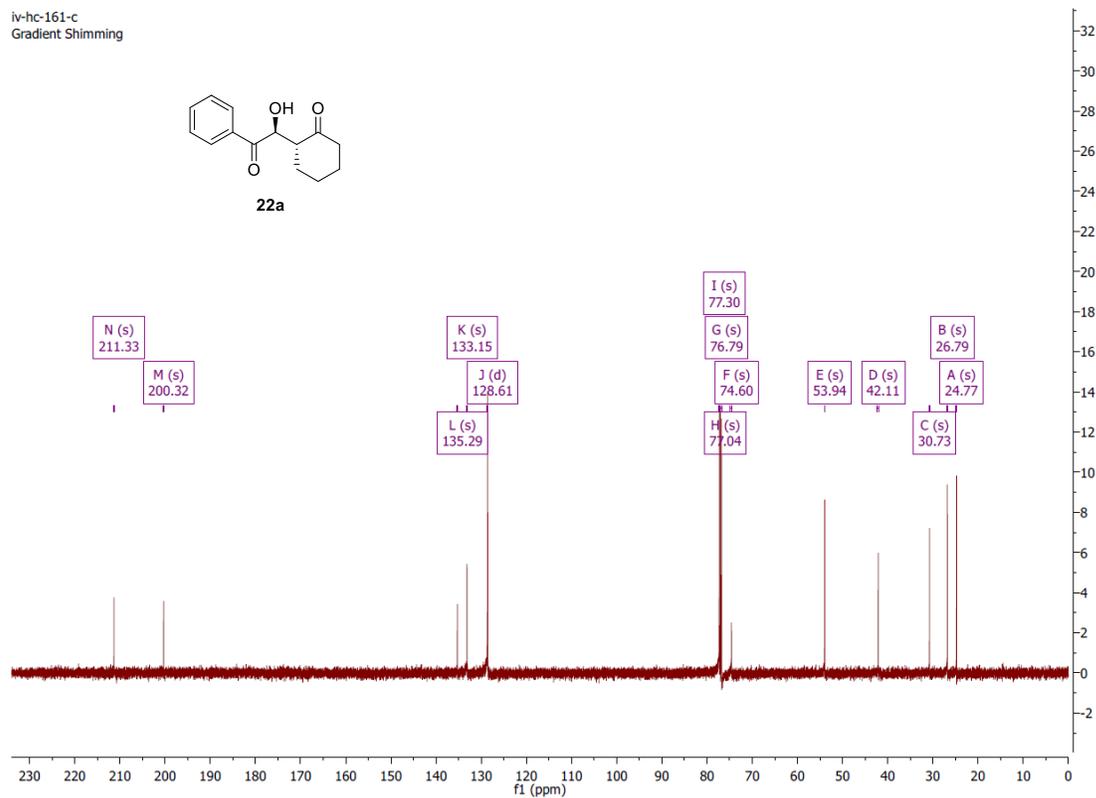
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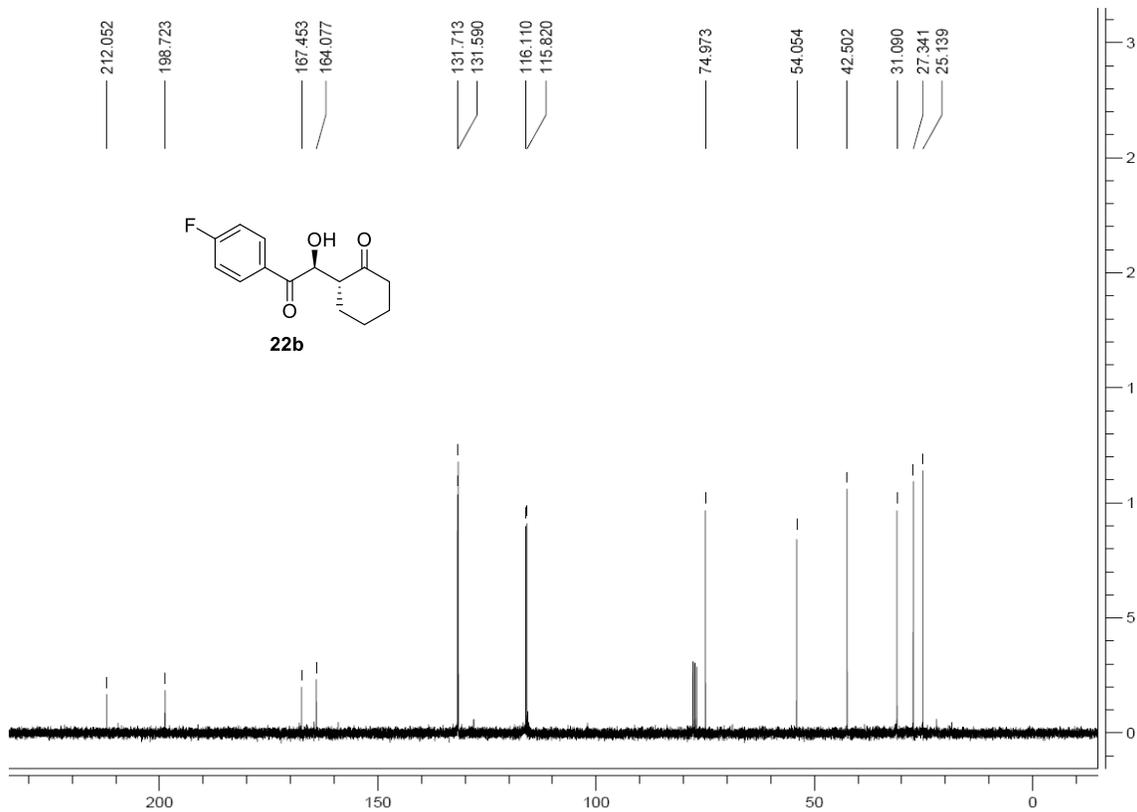
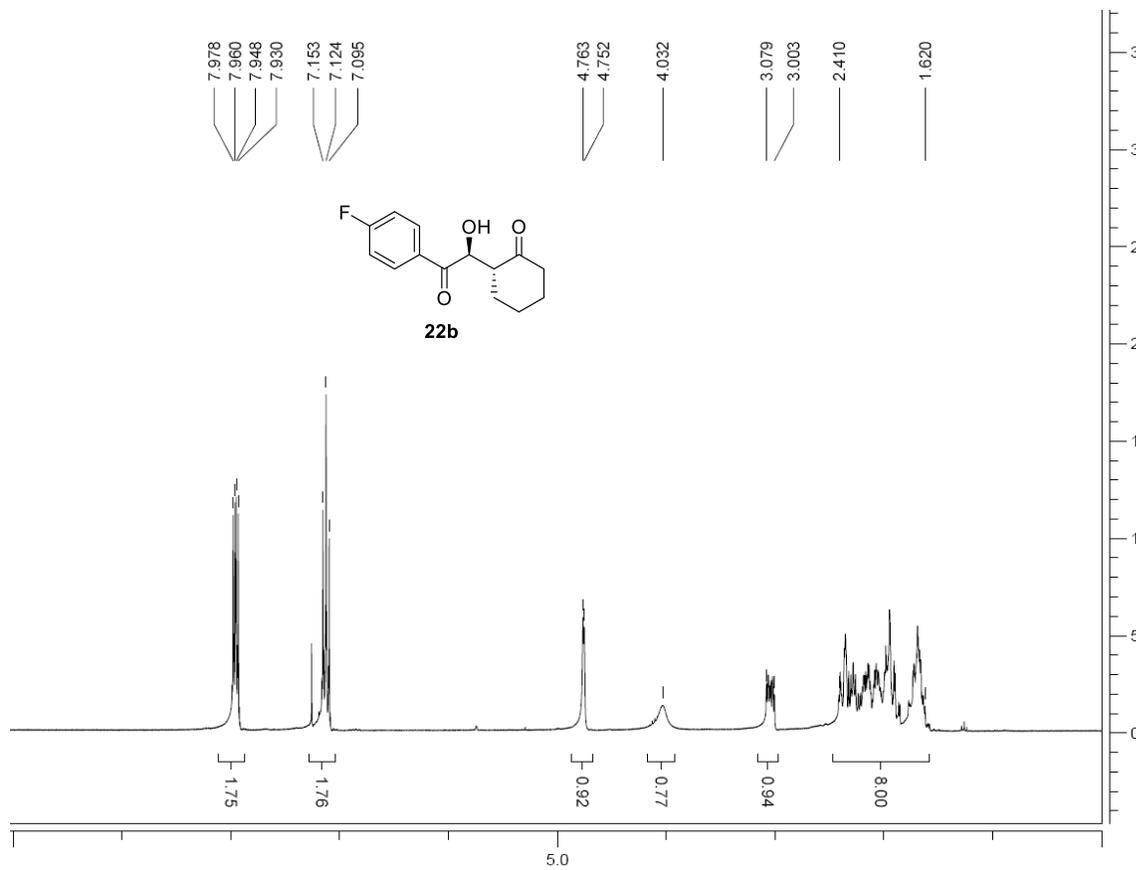


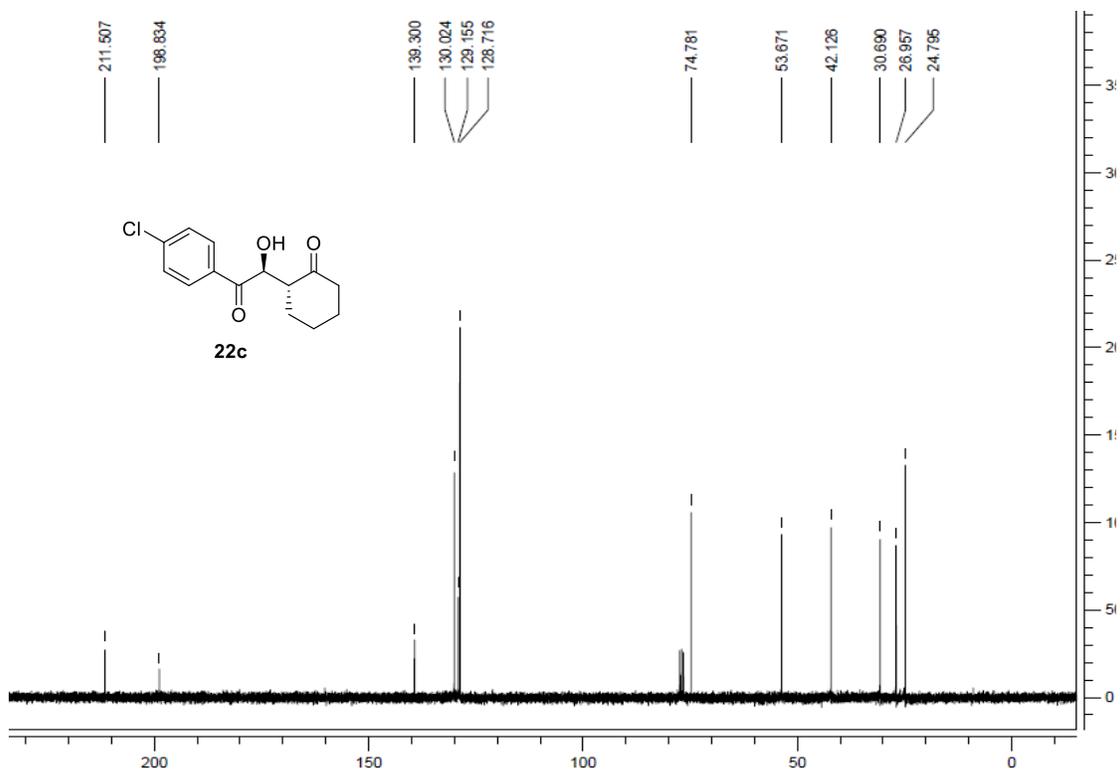
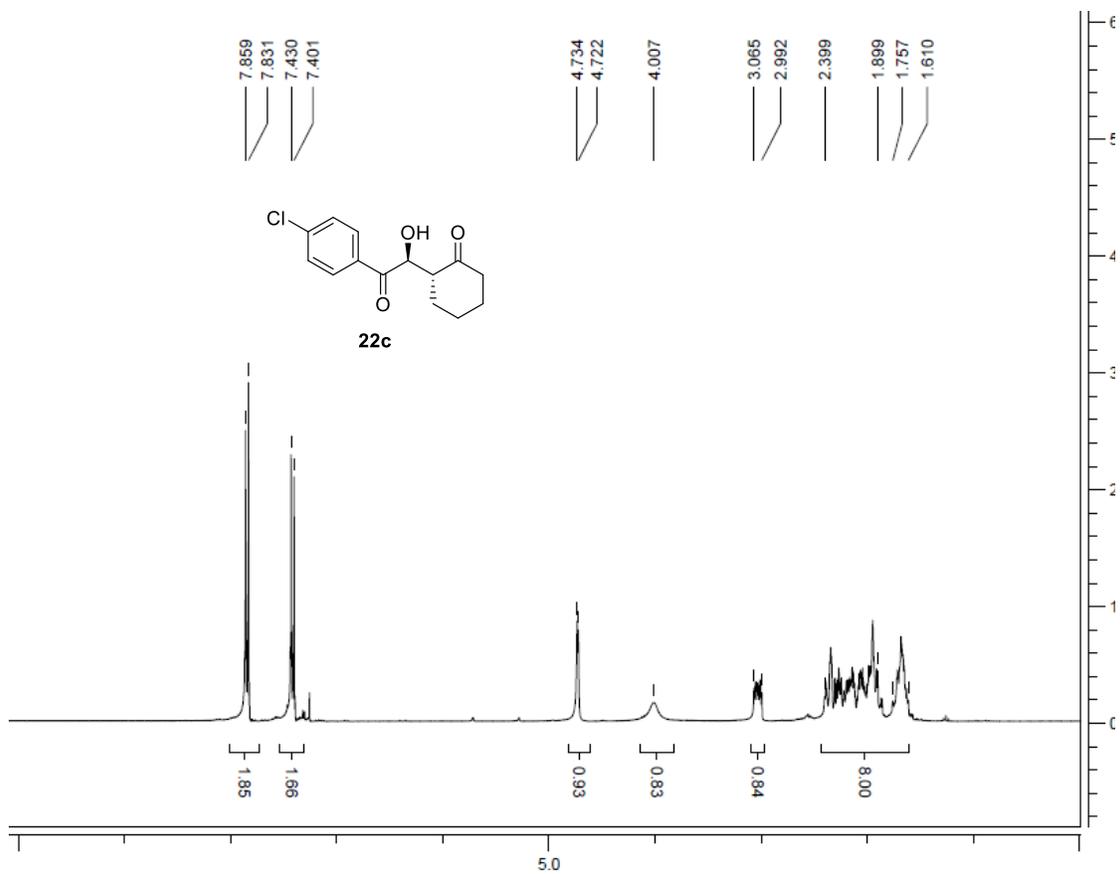
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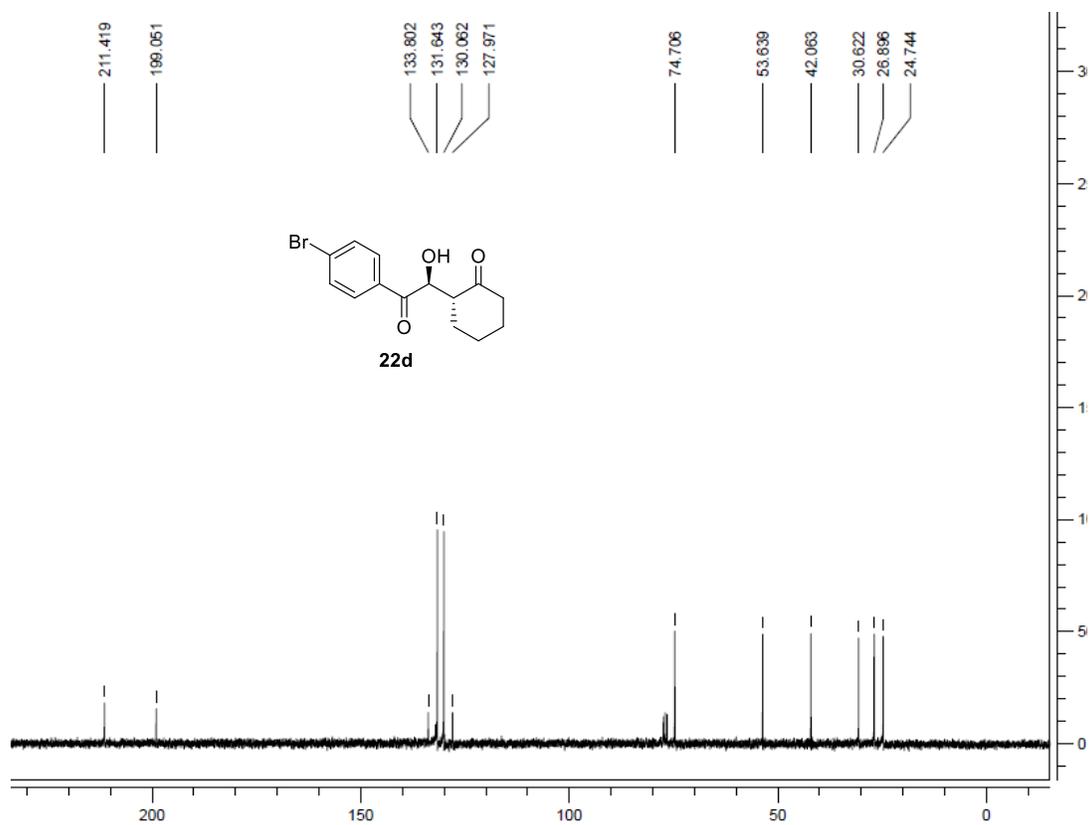
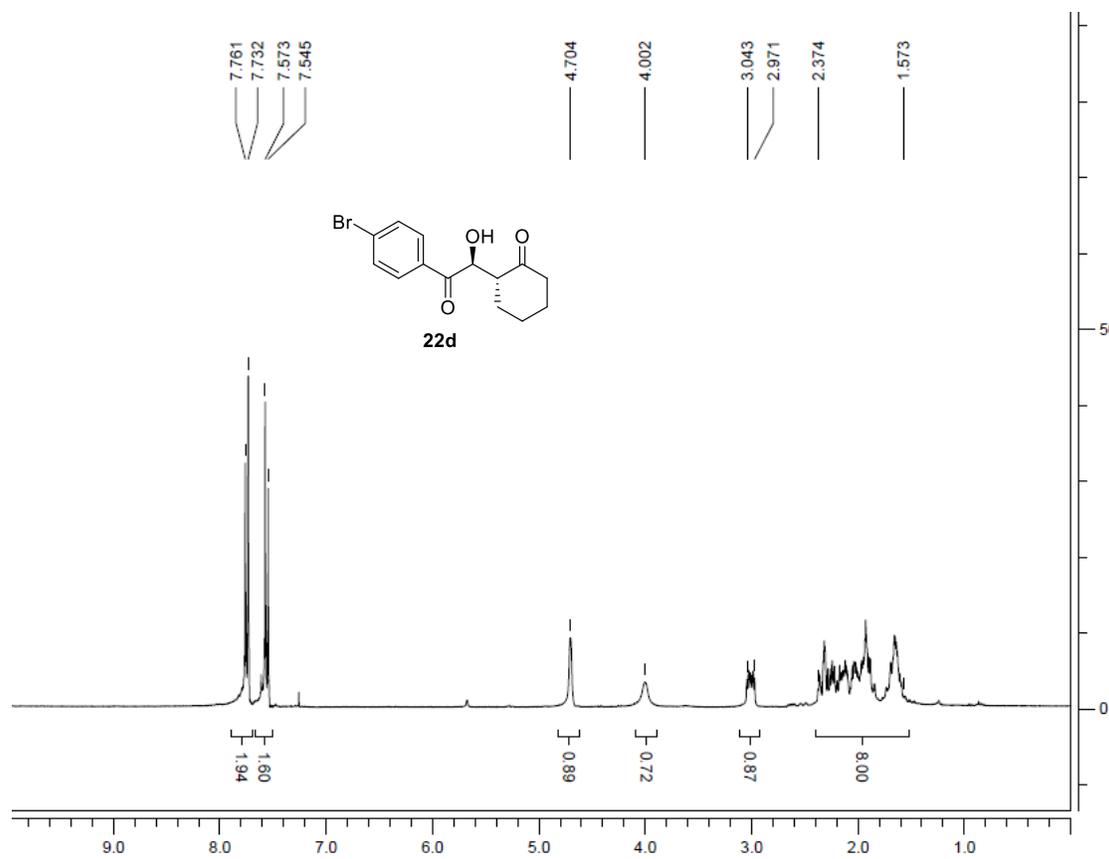


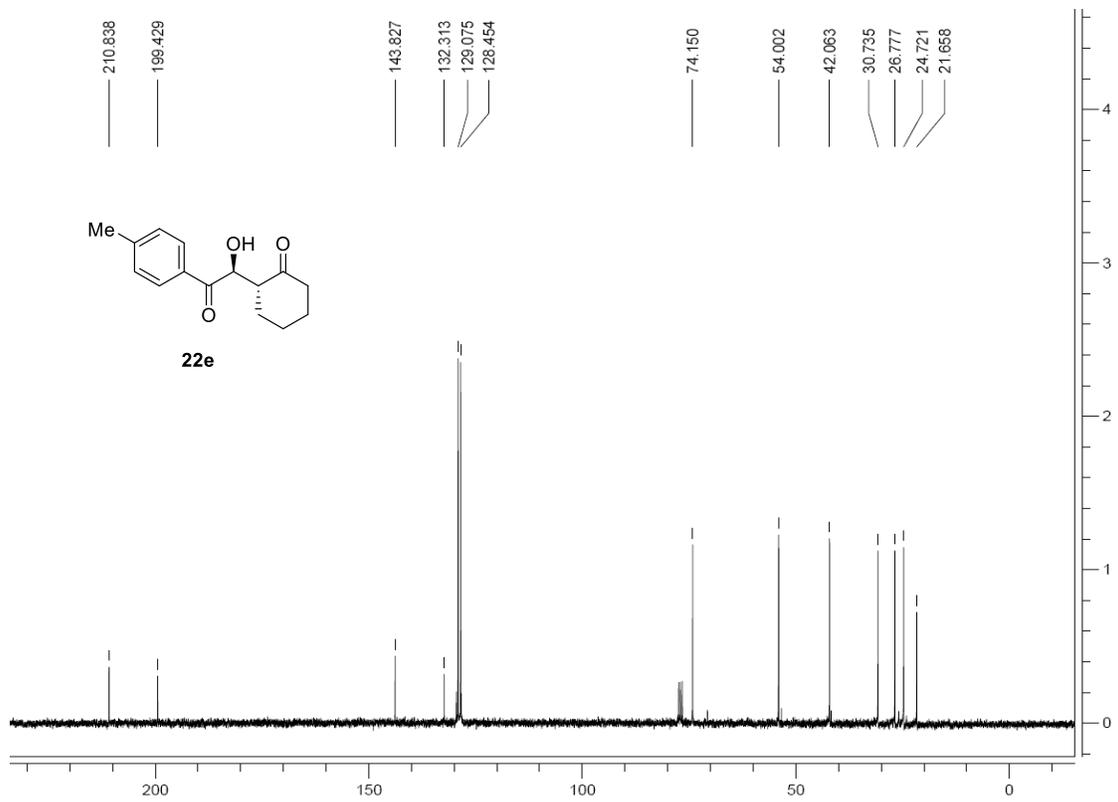
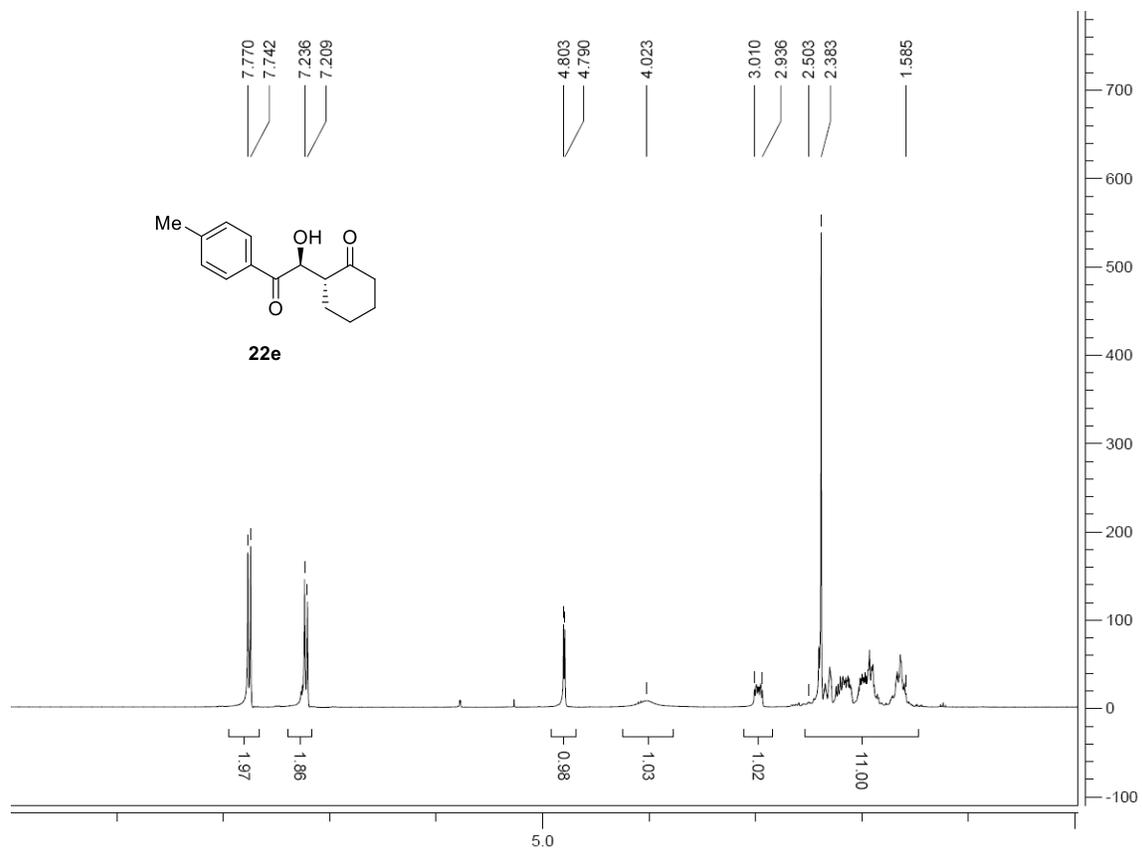
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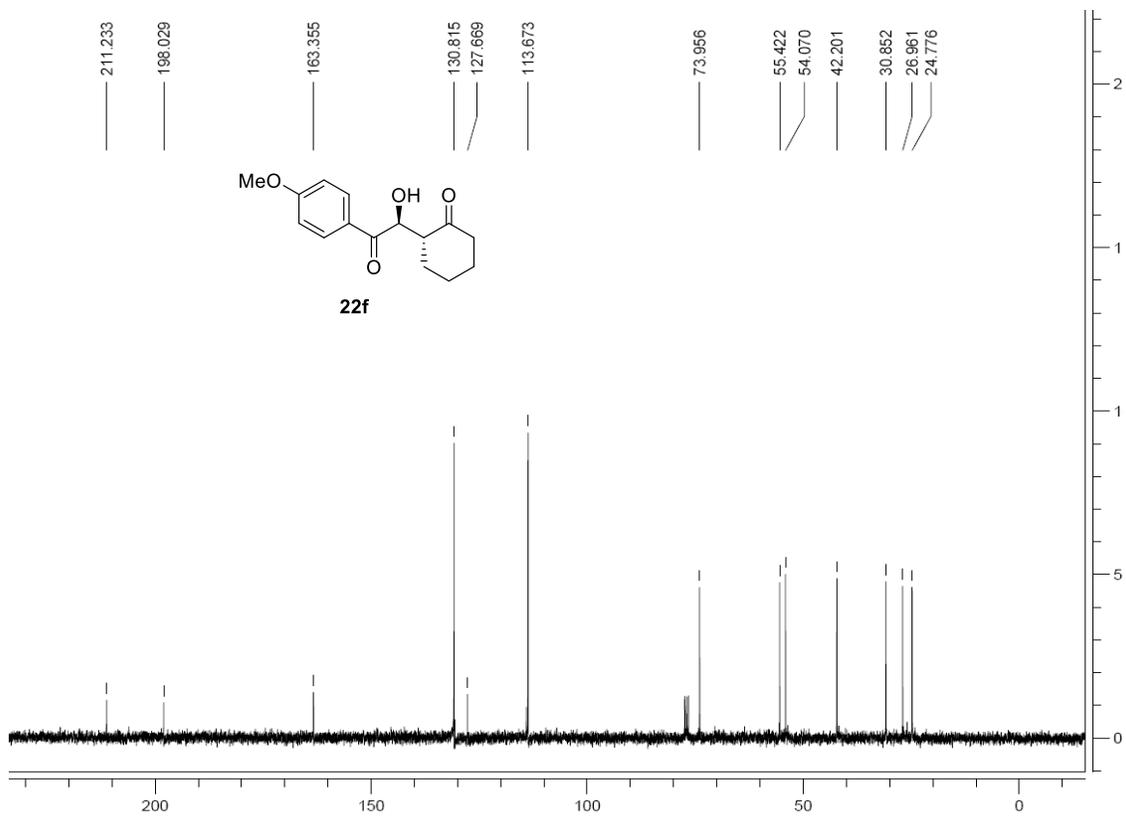
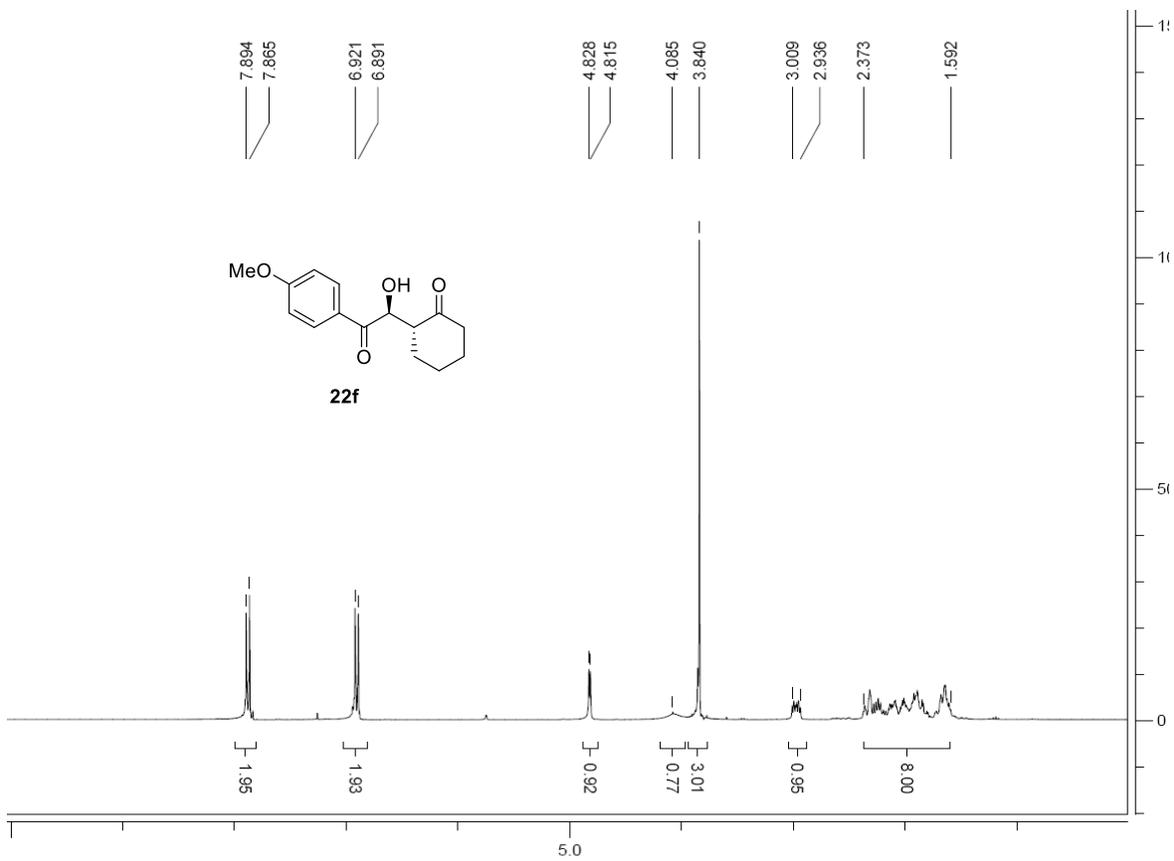


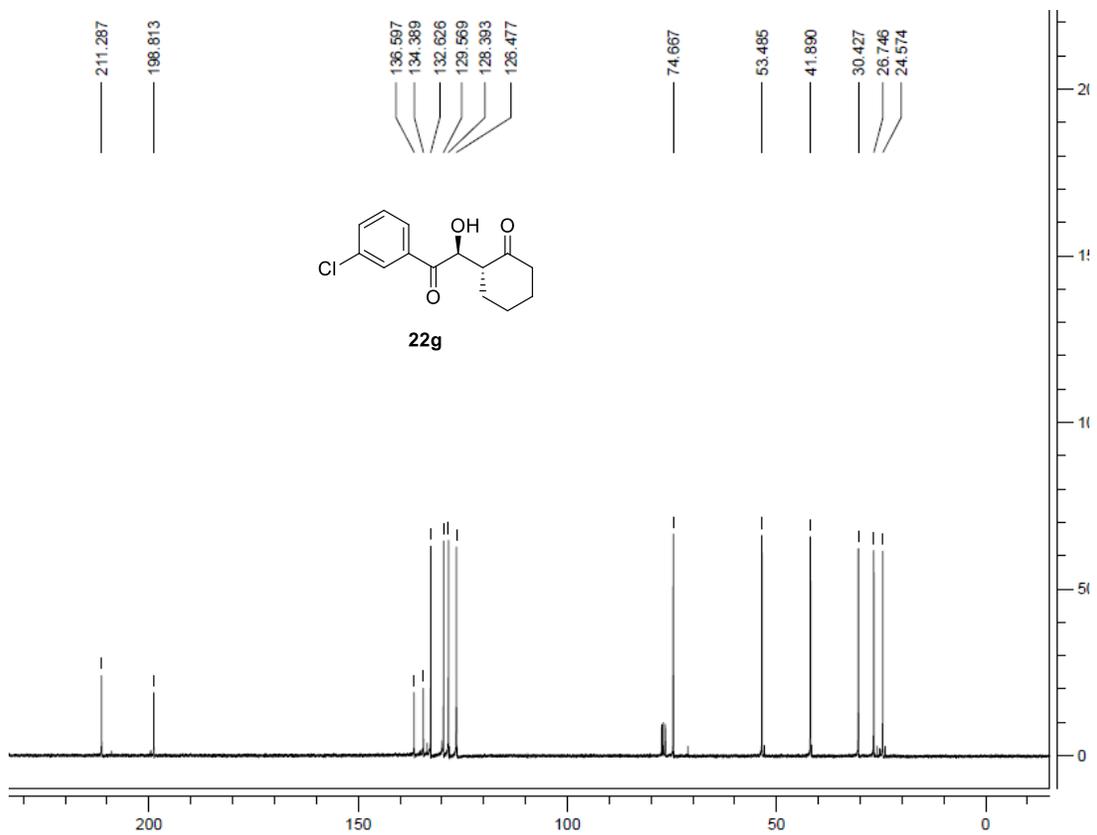
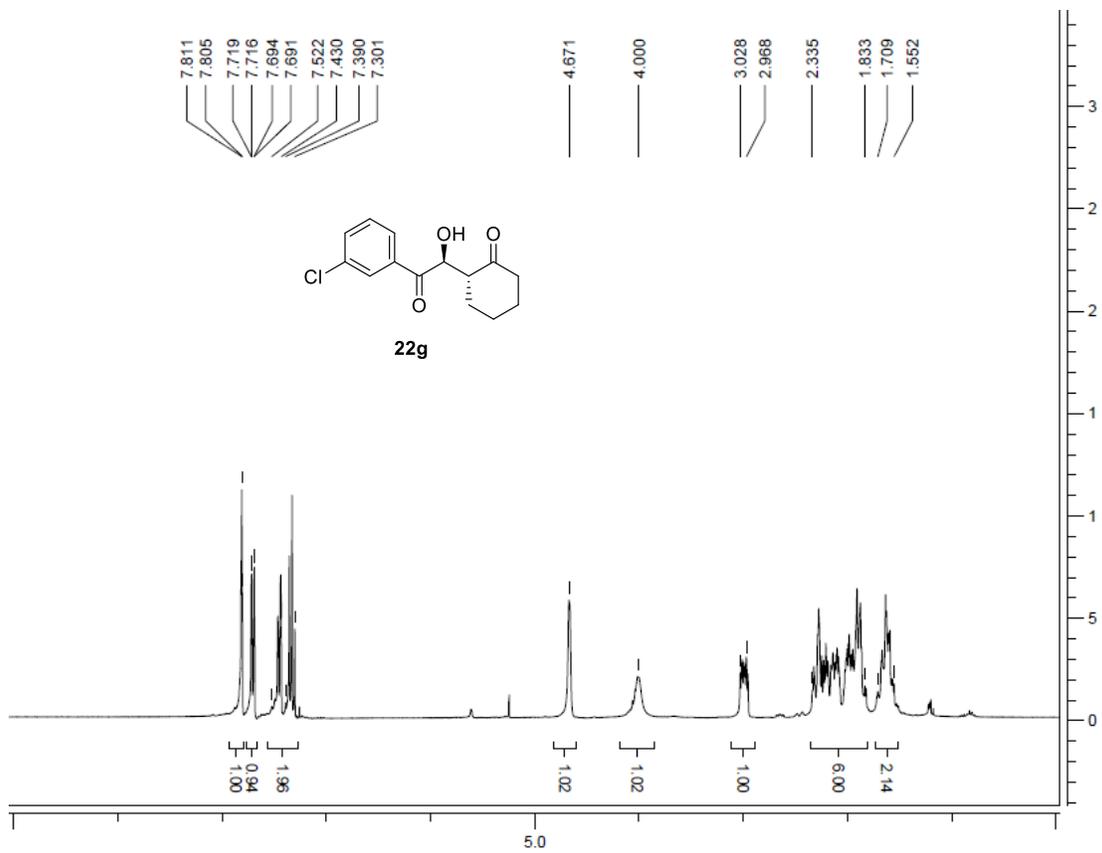


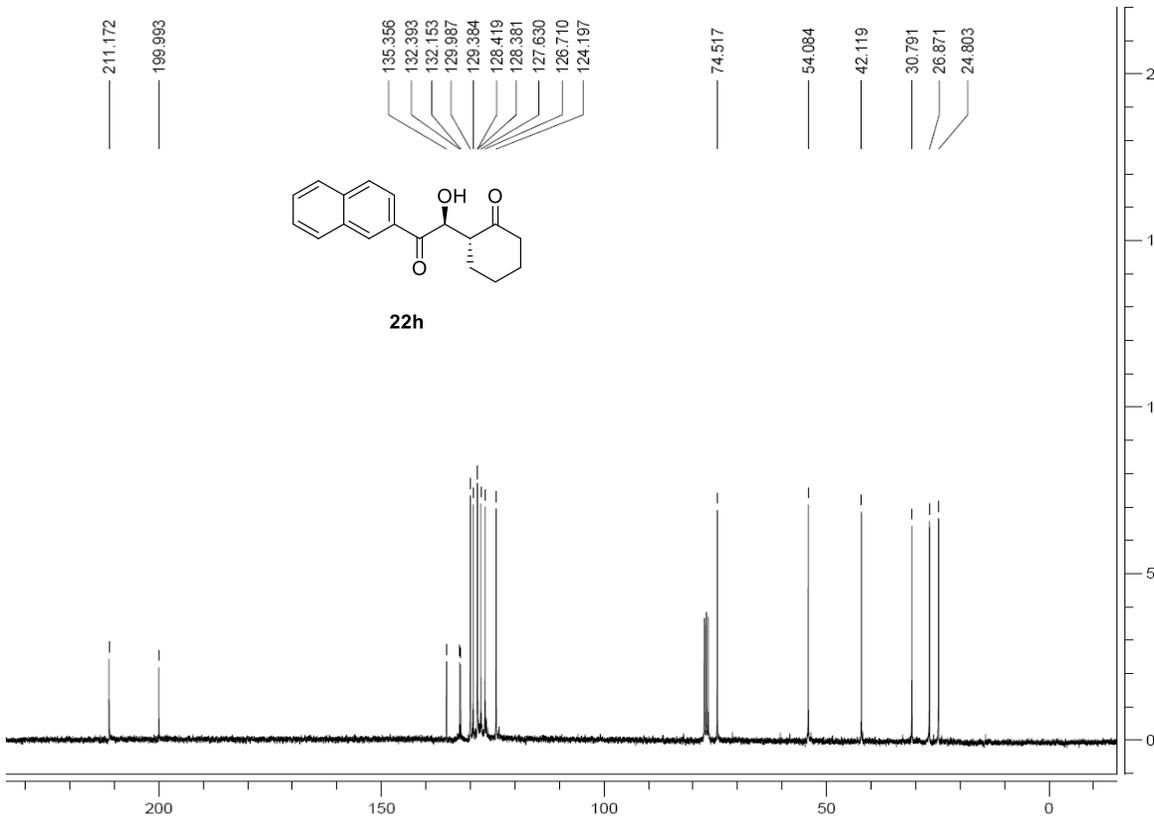
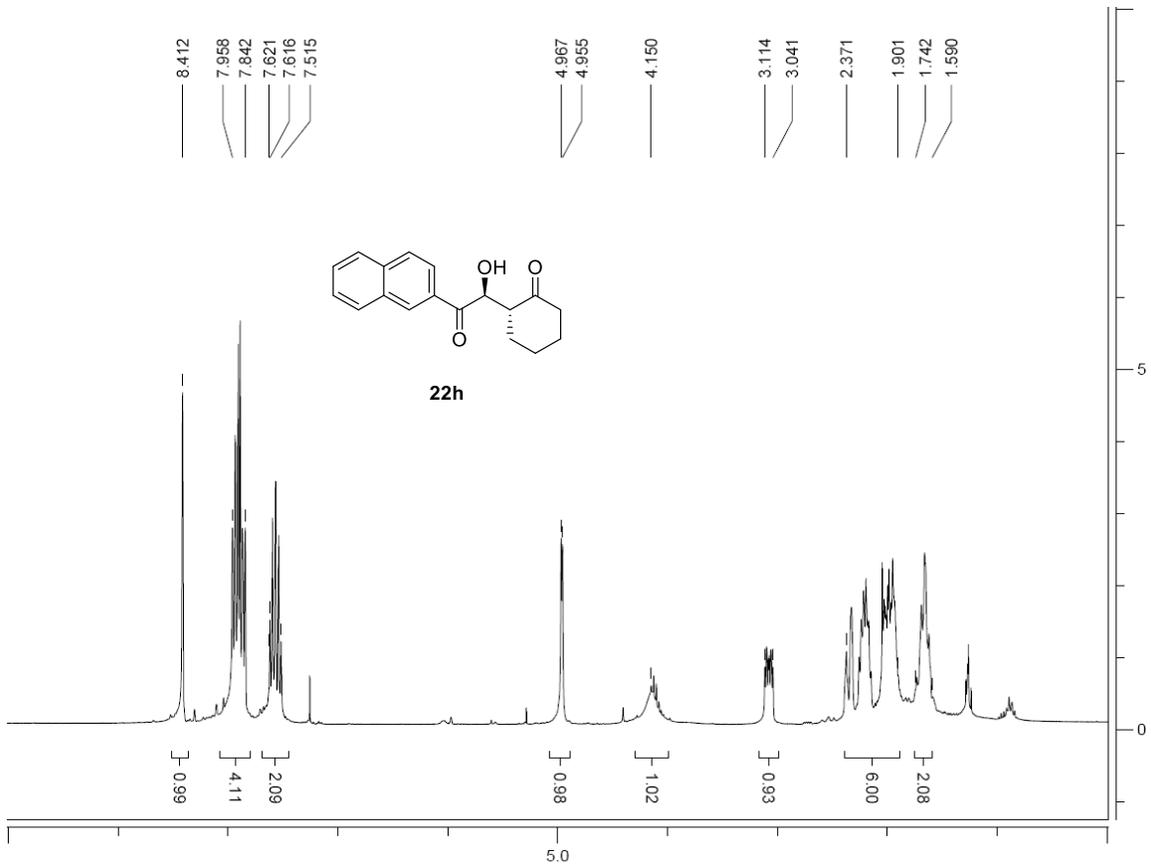


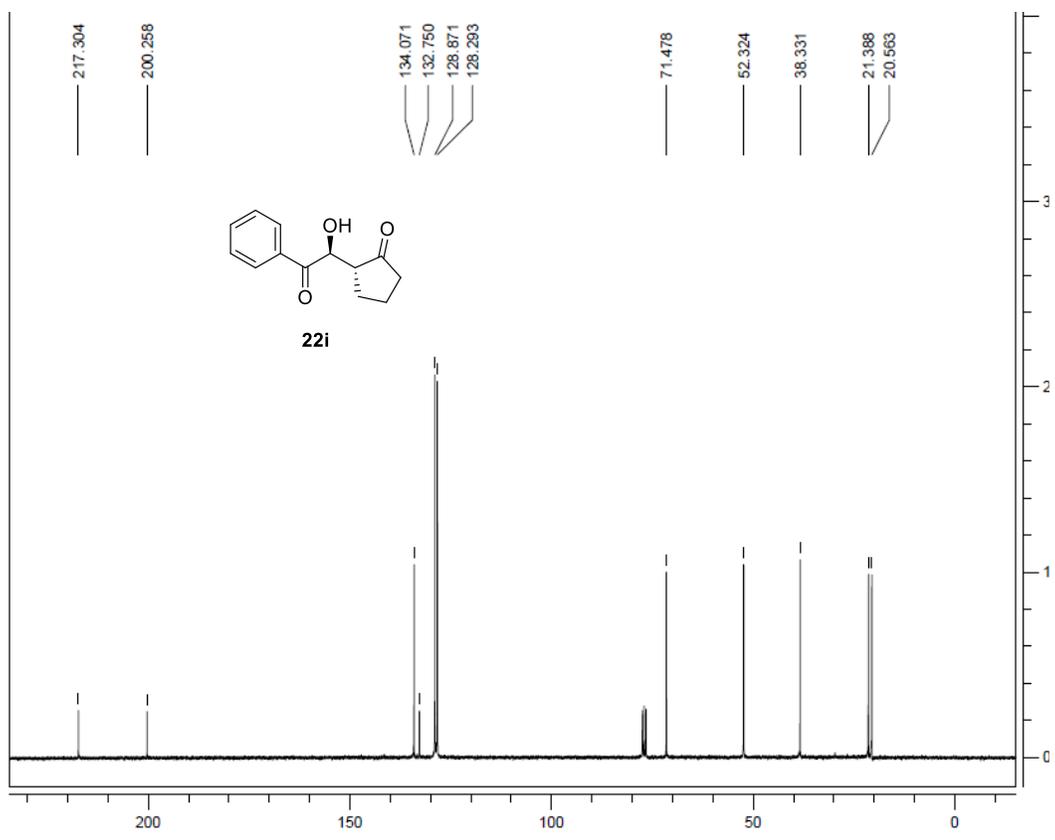
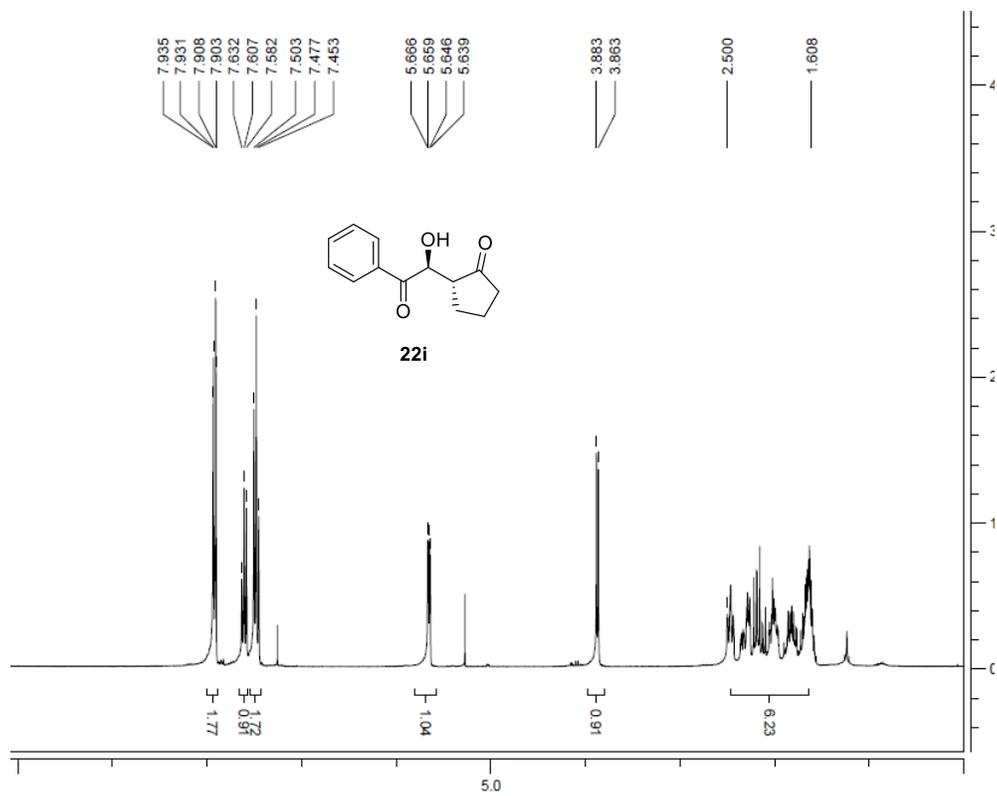


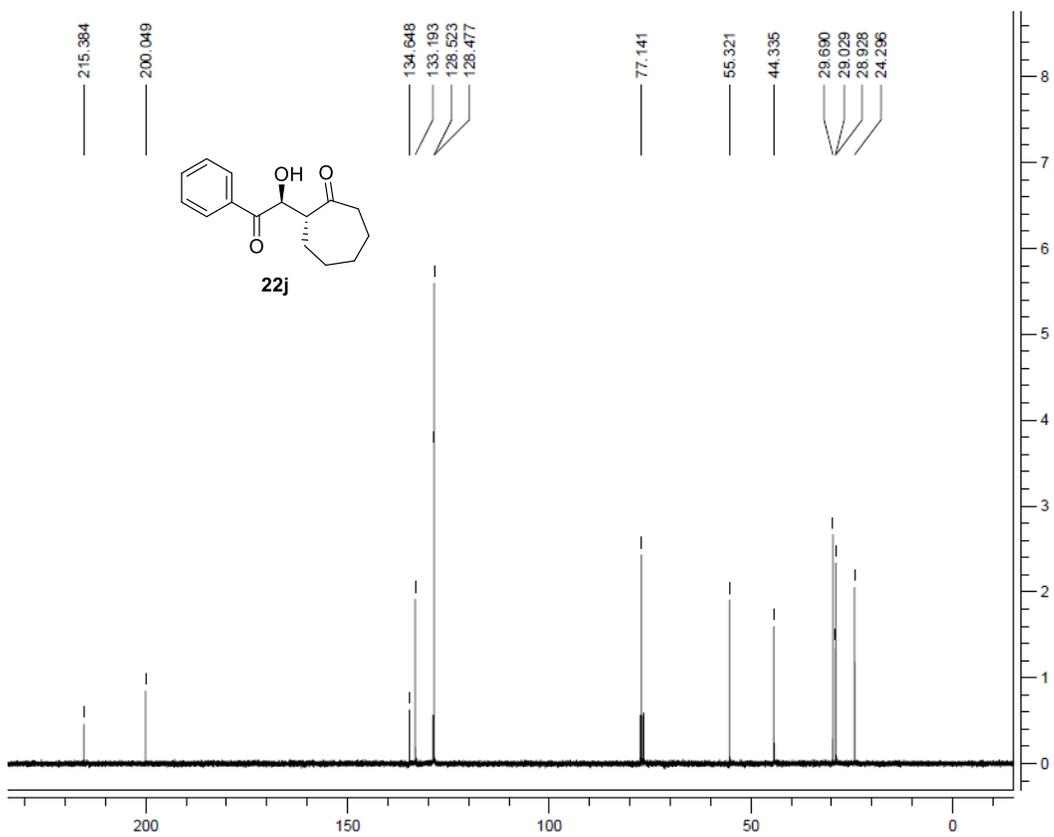
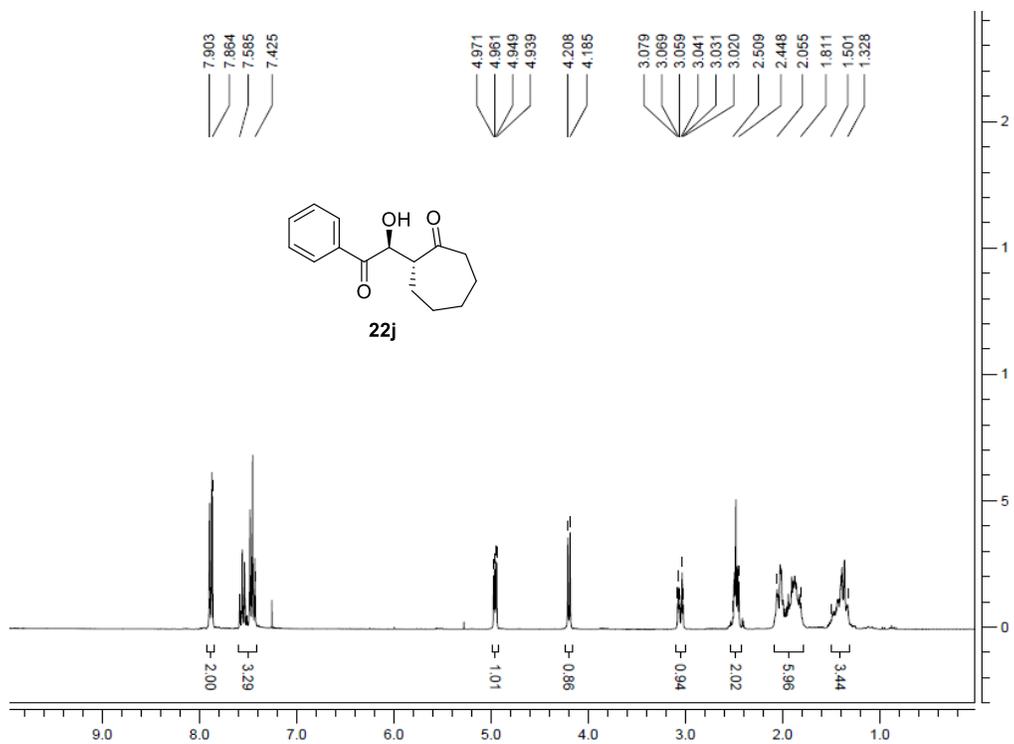


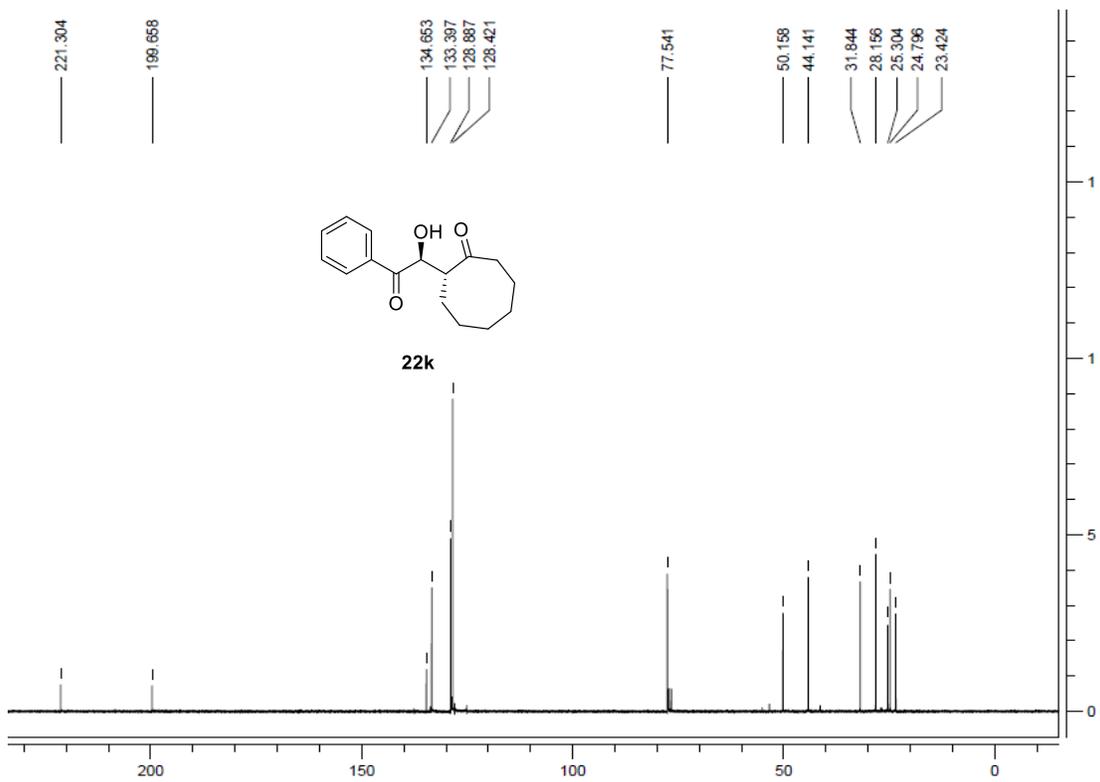
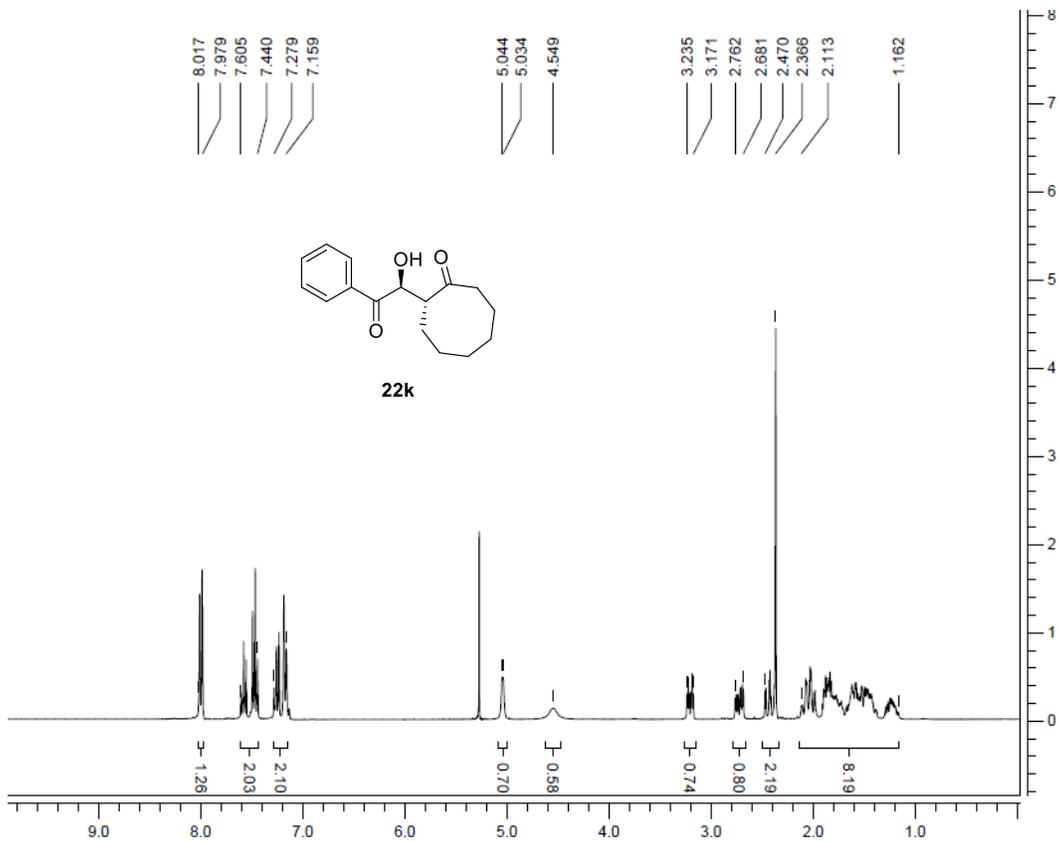






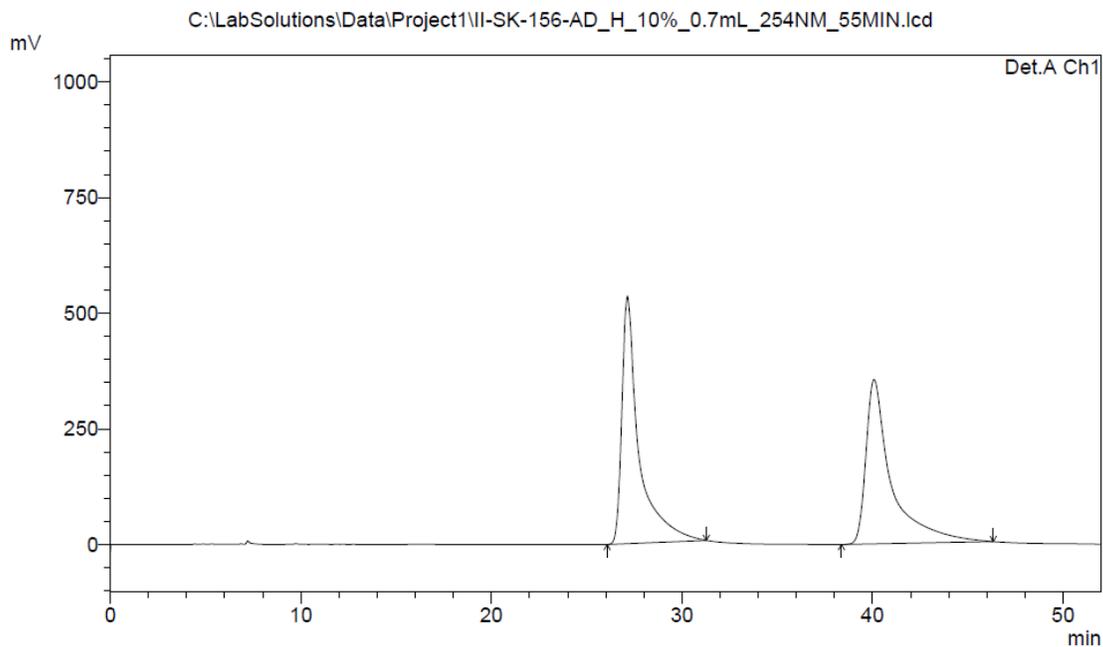






==== Shimadzu LCsolution LC Data Image ====

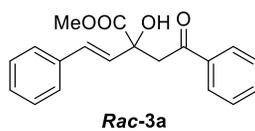
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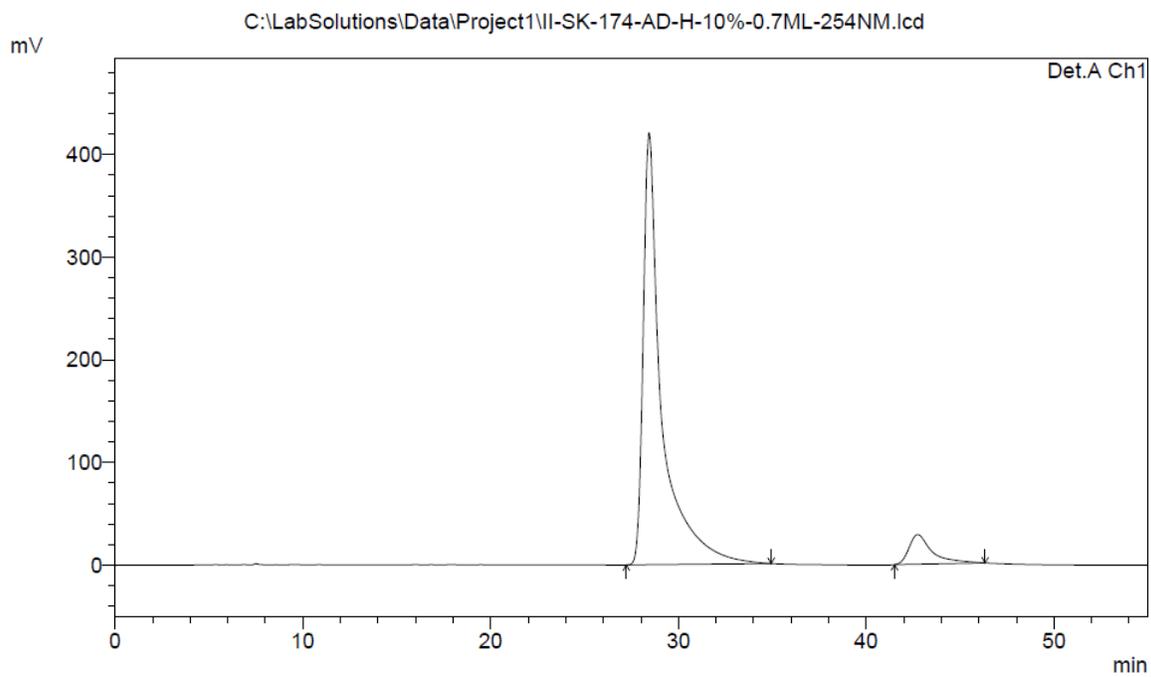


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.131	33259828	535295	50.185	60.107
2	40.073	33014018	355281	49.815	39.893
Total		66273847	890575	100.000	100.000

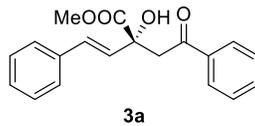




PeakTable

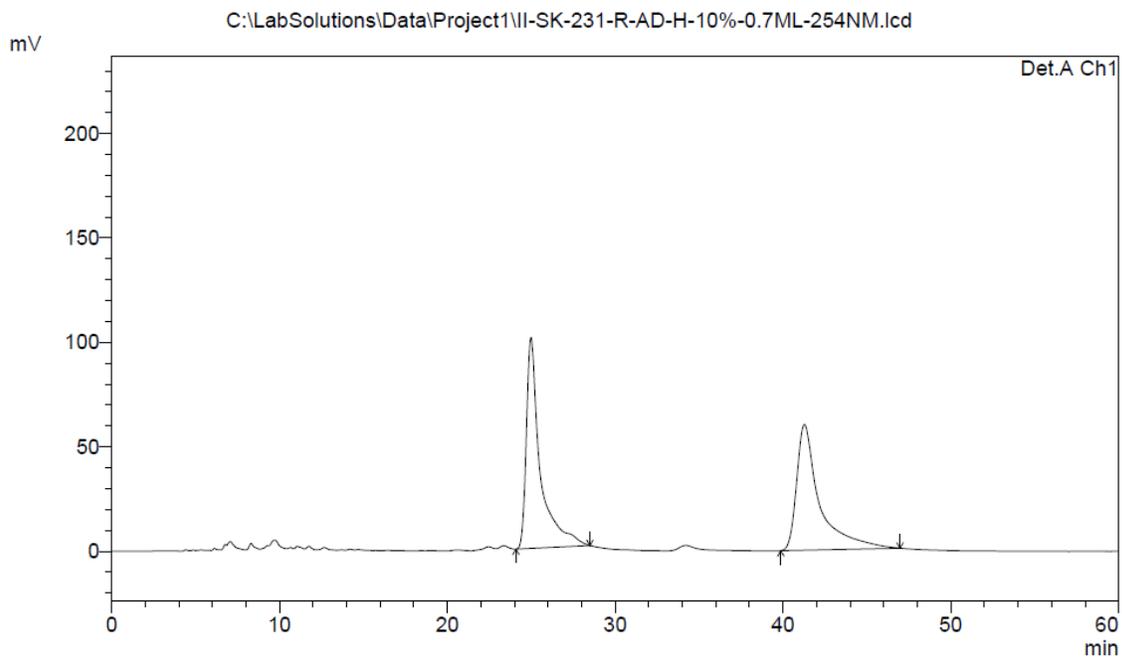
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.434	28894483	421105	92.092	93.570
2	42.733	2481260	28937	7.908	6.430
Total		31375744	450042	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

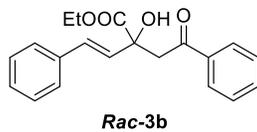
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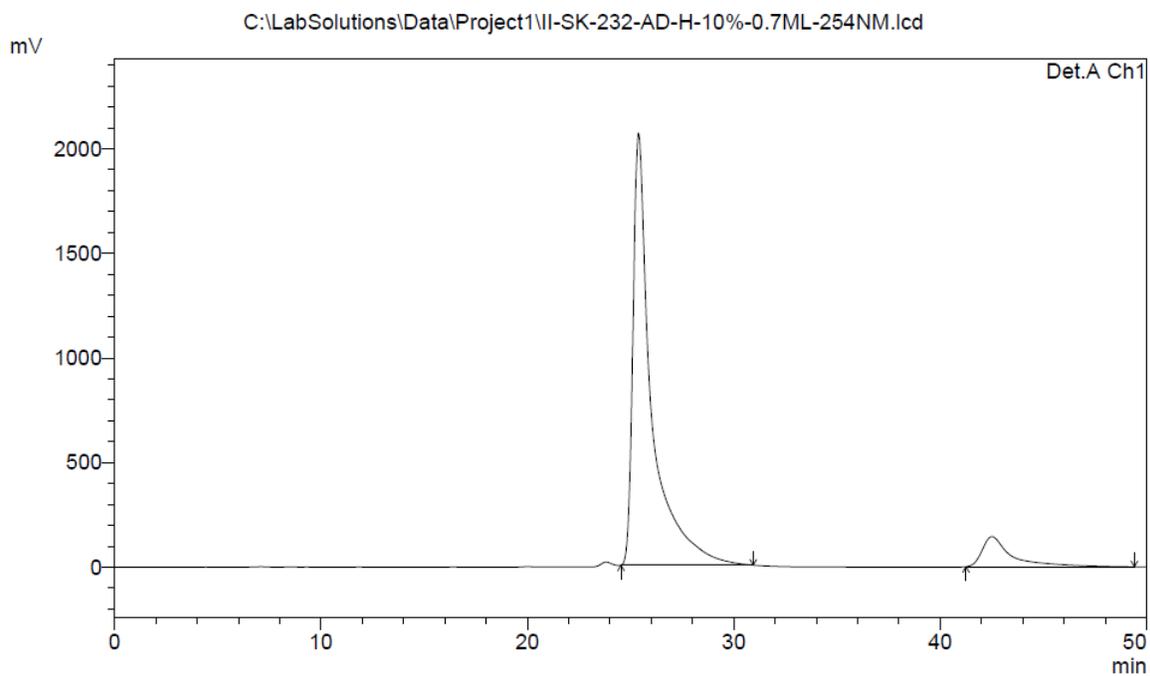


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.980	5826921	101042	50.468	62.663
2	41.272	5718757	60205	49.532	37.337
Total		11545679	161247	100.000	100.000

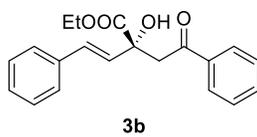




PeakTable

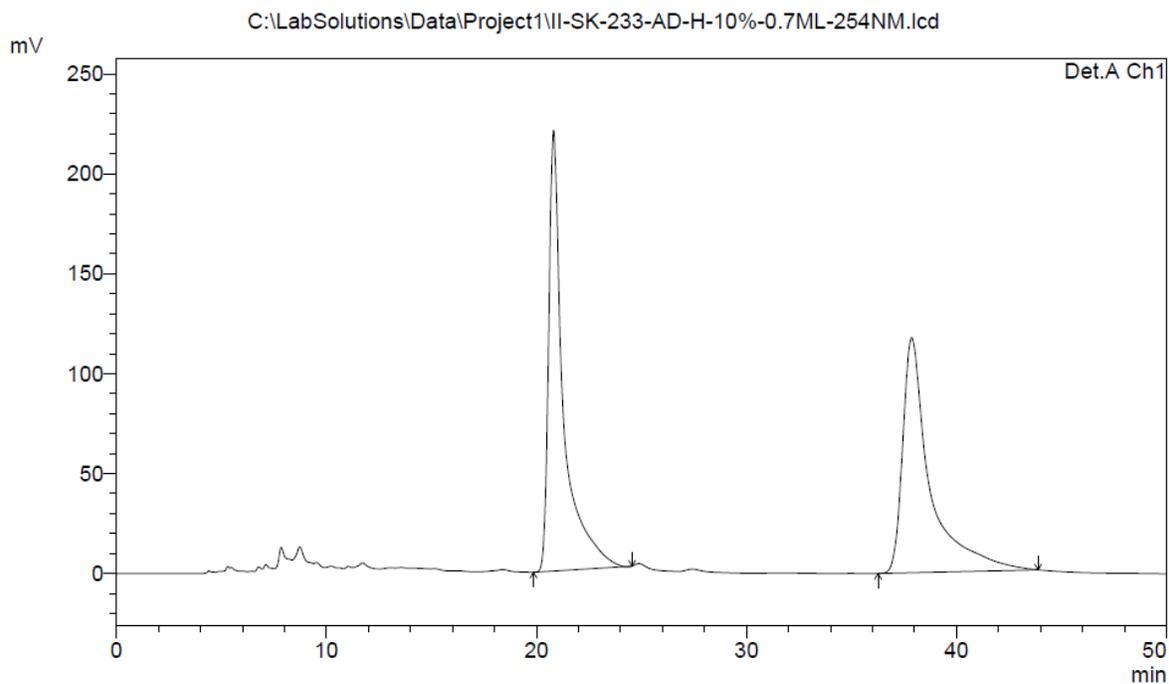
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	25.377	129352950	2064528	89.951	93.464
2	42.496	14450198	144376	10.049	6.536
Total		143803149	2208904	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

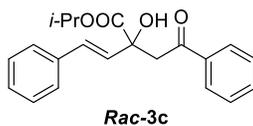
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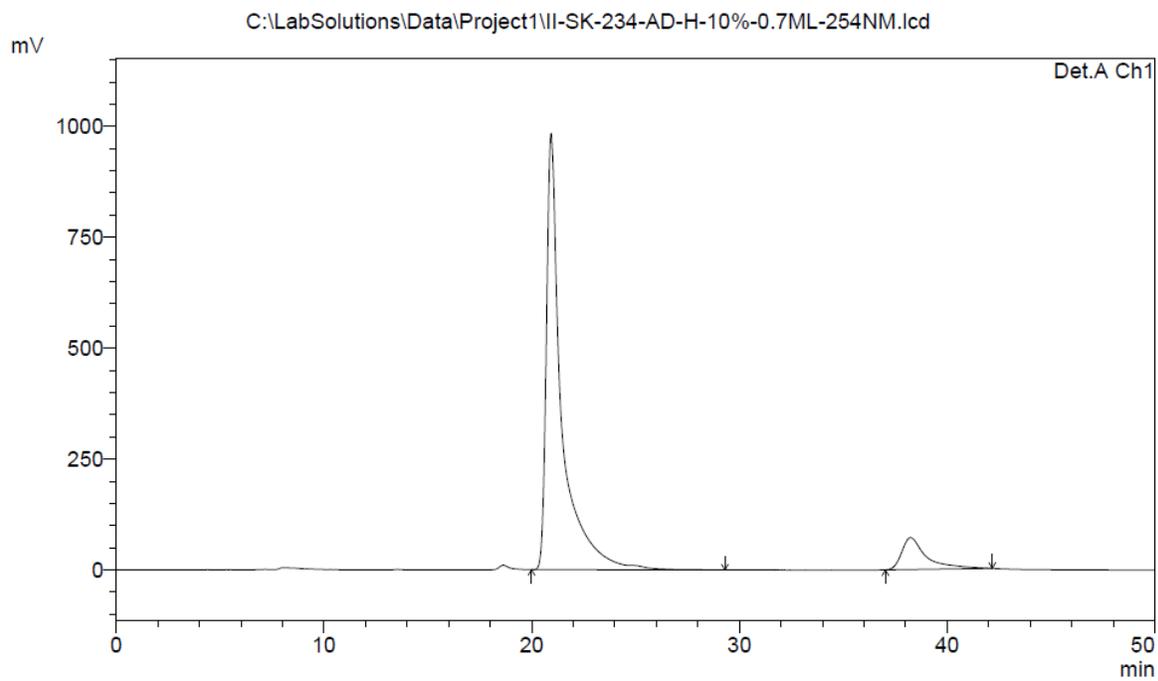


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.809	10792721	220575	50.289	65.207
2	37.856	10668643	117693	49.711	34.793
Total		21461365	338268	100.000	100.000

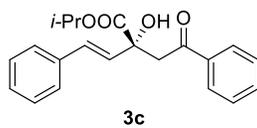




PeakTable

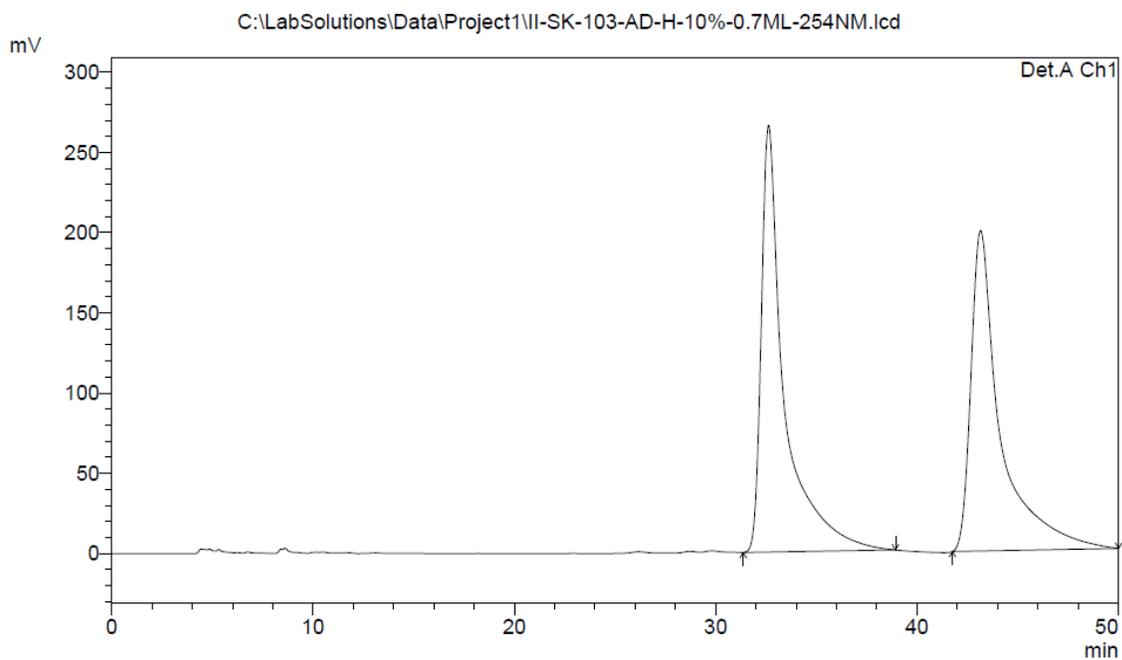
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.931	50298361	983999	89.410	93.181
2	38.230	5957340	72012	10.590	6.819
Total		56255701	1056011	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

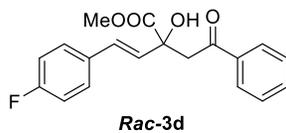
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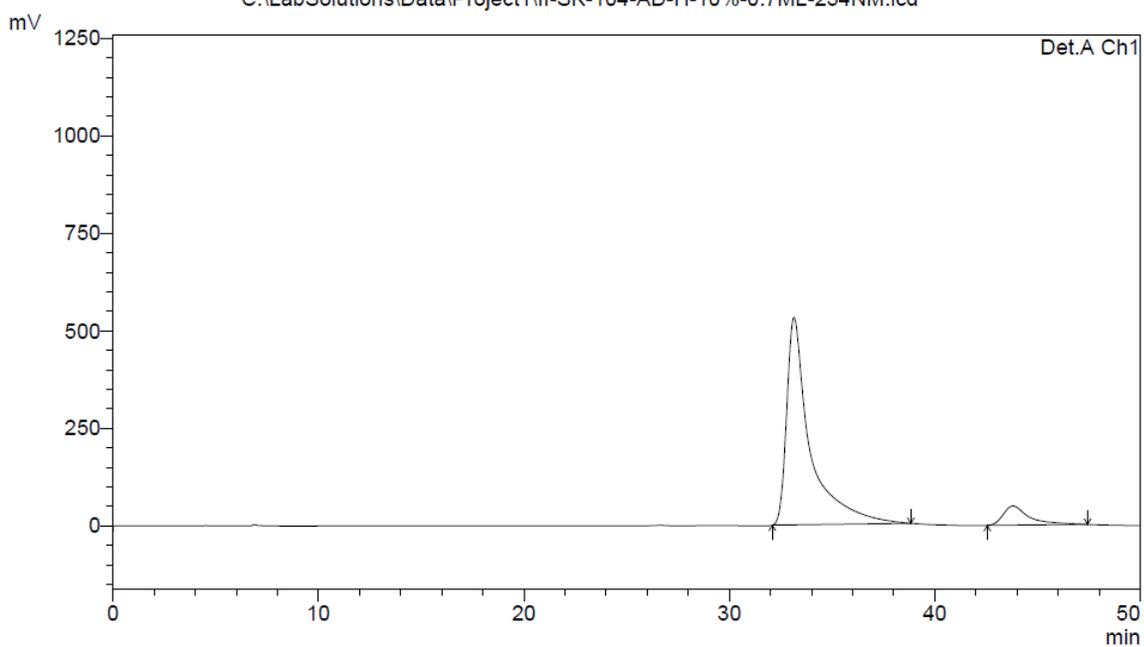
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	32.617	21038101	266037	50.948	57.112
2	43.146	20255578	199776	49.052	42.888
Total		41293680	465813	100.000	100.000



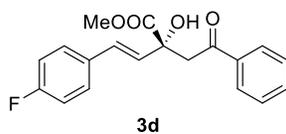
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PeakTable

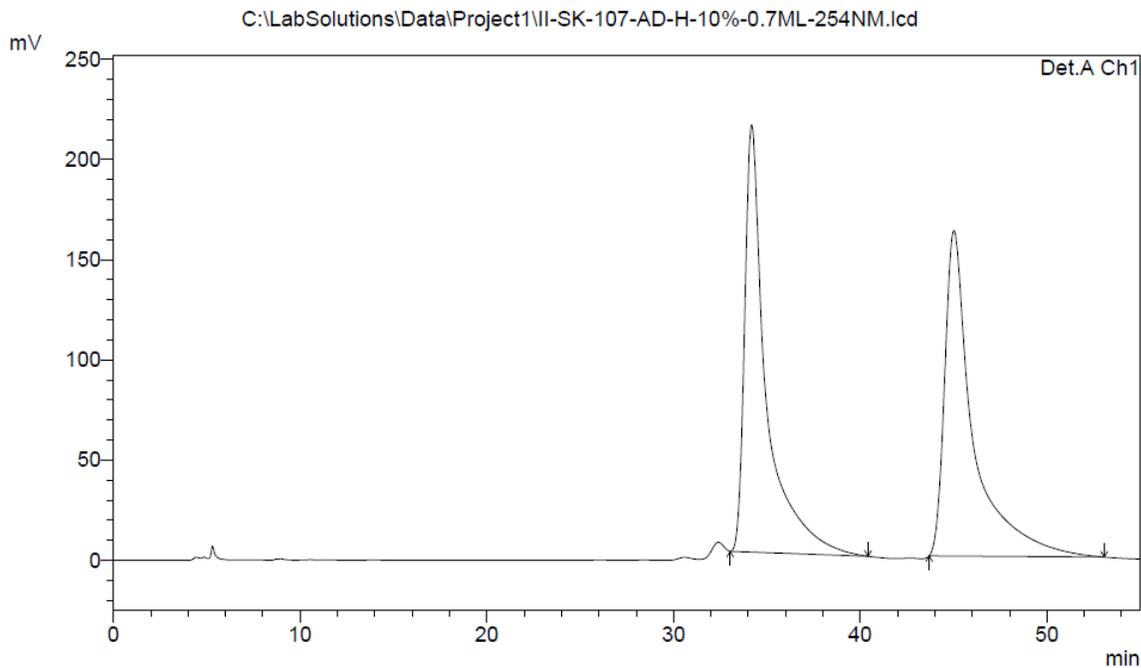
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	33.137	42249633	532036	90.716	91.546
2	43.792	4324105	49131	9.284	8.454
Total		46573737	581167	100.000	100.000



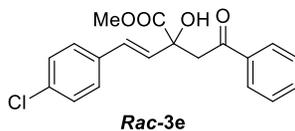
==== Shimadzu LCsolution LC Data Image ====

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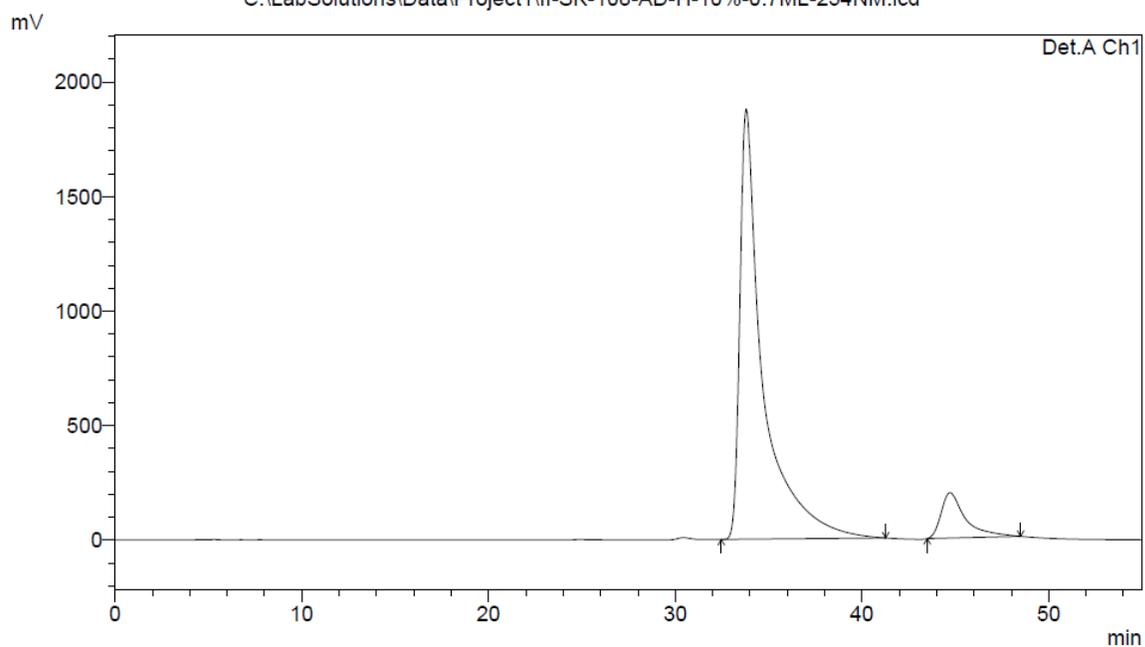


PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	34.180	17123432	213272	49.883	56.765
2	45.011	17203986	162439	50.117	43.235
Total		34327419	375711	100.000	100.000



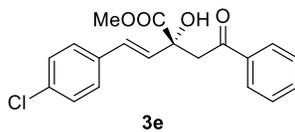
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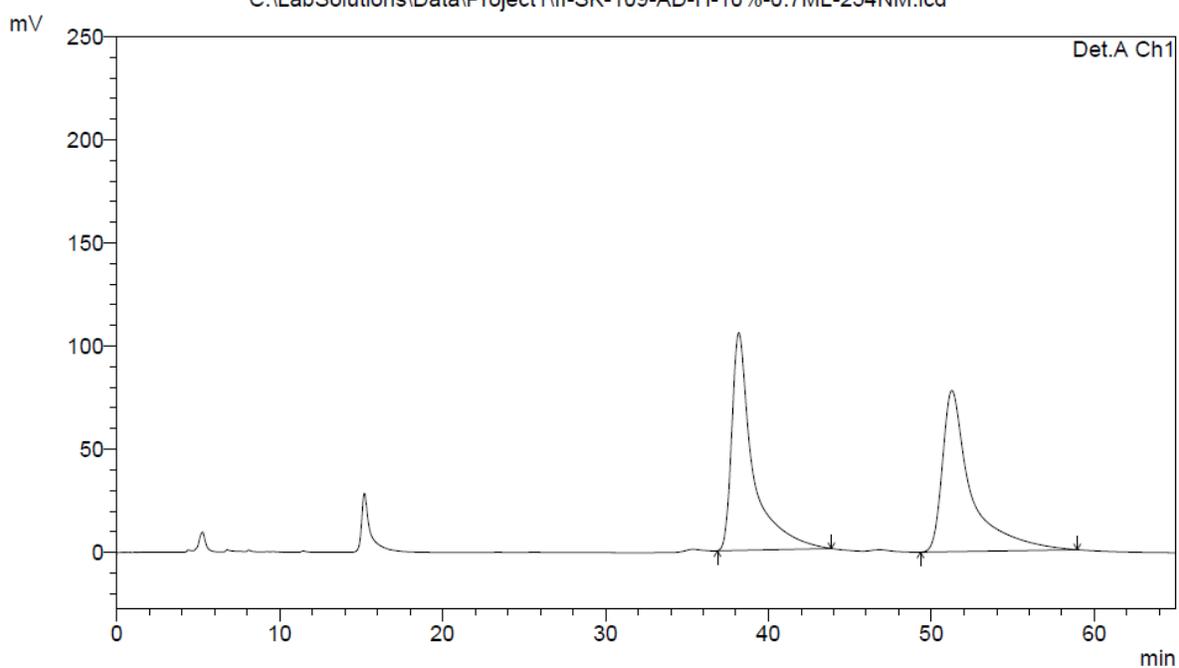
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	33.794	160738564	1880193	89.951	90.454
2	44.710	17956343	198415	10.049	9.546
Total		178694908	2078607	100.000	100.000



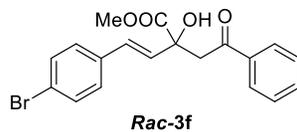
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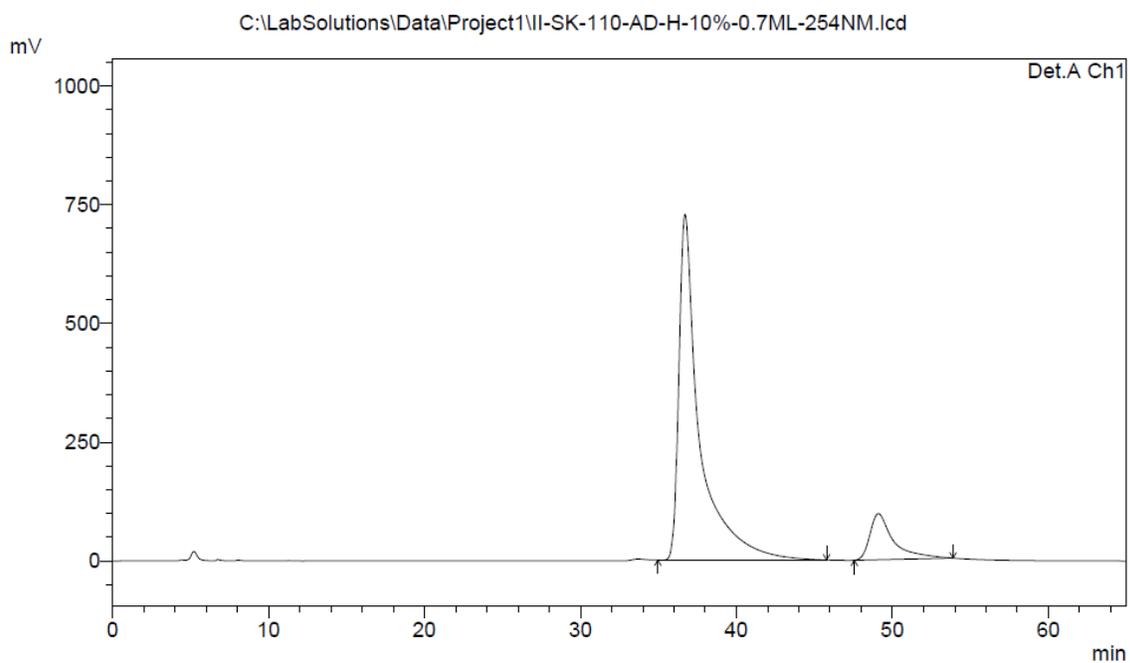


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	38.175	9485540	105543	49.855	57.470
2	51.252	9540652	78106	50.145	42.530
Total		19026192	183649	100.000	100.000

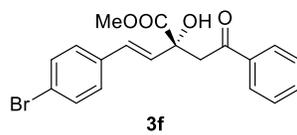




PeakTable

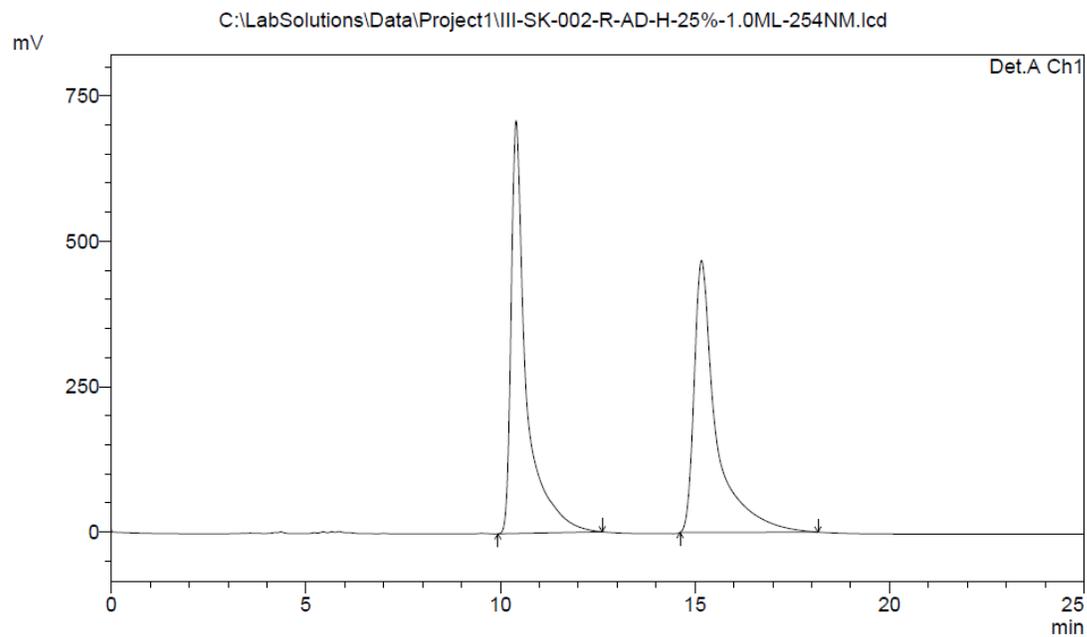
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	36.700	66628030	728412	86.728	88.242
2	49.096	10196007	97063	13.272	11.758
Total		76824037	825475	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

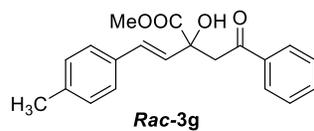
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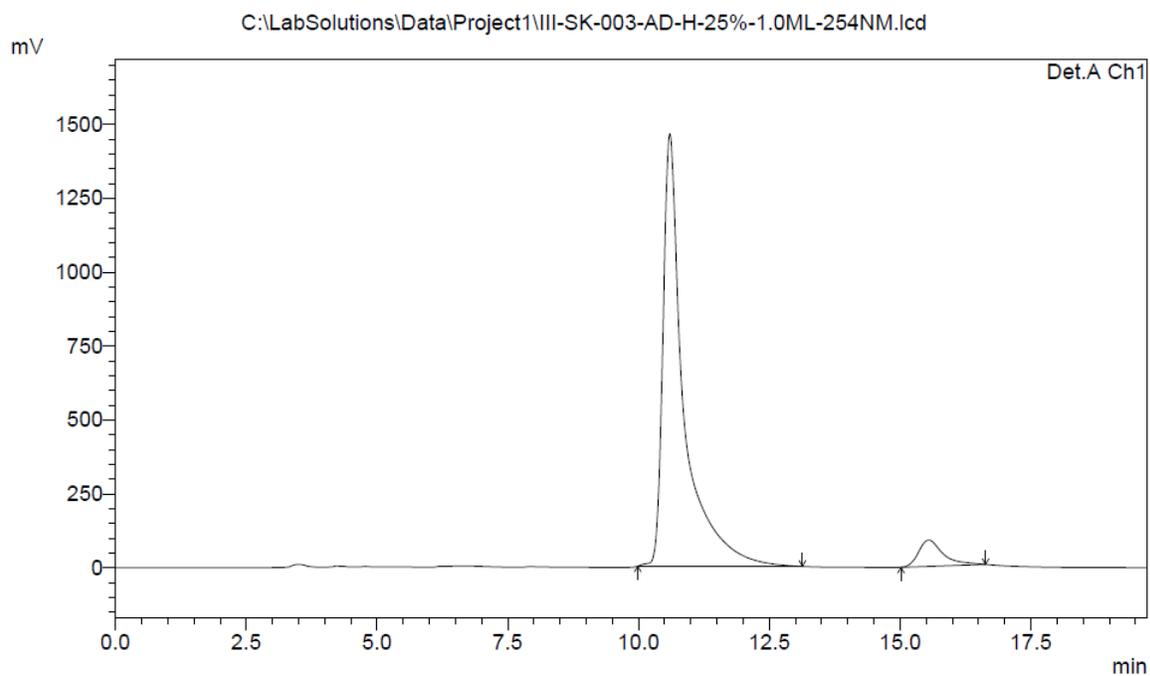


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.399	19078923	709920	50.854	60.275
2	15.164	18438446	467891	49.146	39.725
Total		37517369	1177811	100.000	100.000

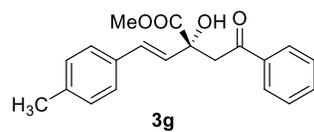




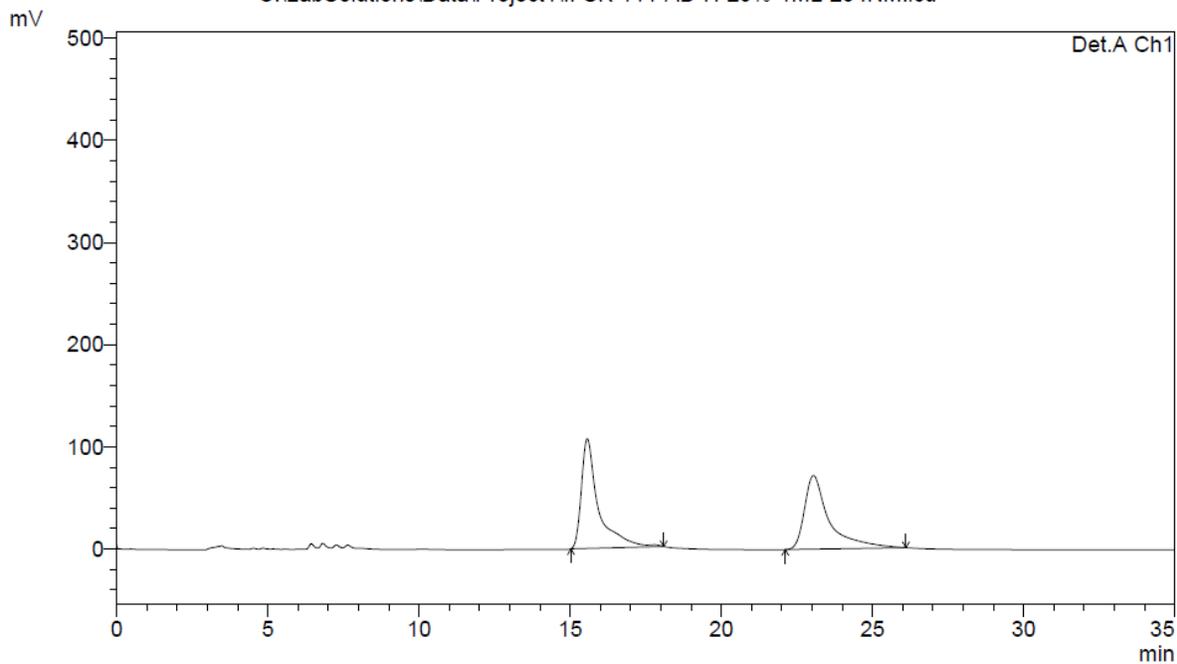
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.593	40599136	1462872	93.306	94.272
2	15.543	2912570	88885	6.694	5.728
Total		43511706	1551757	100.000	100.000



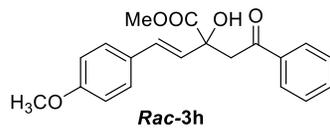
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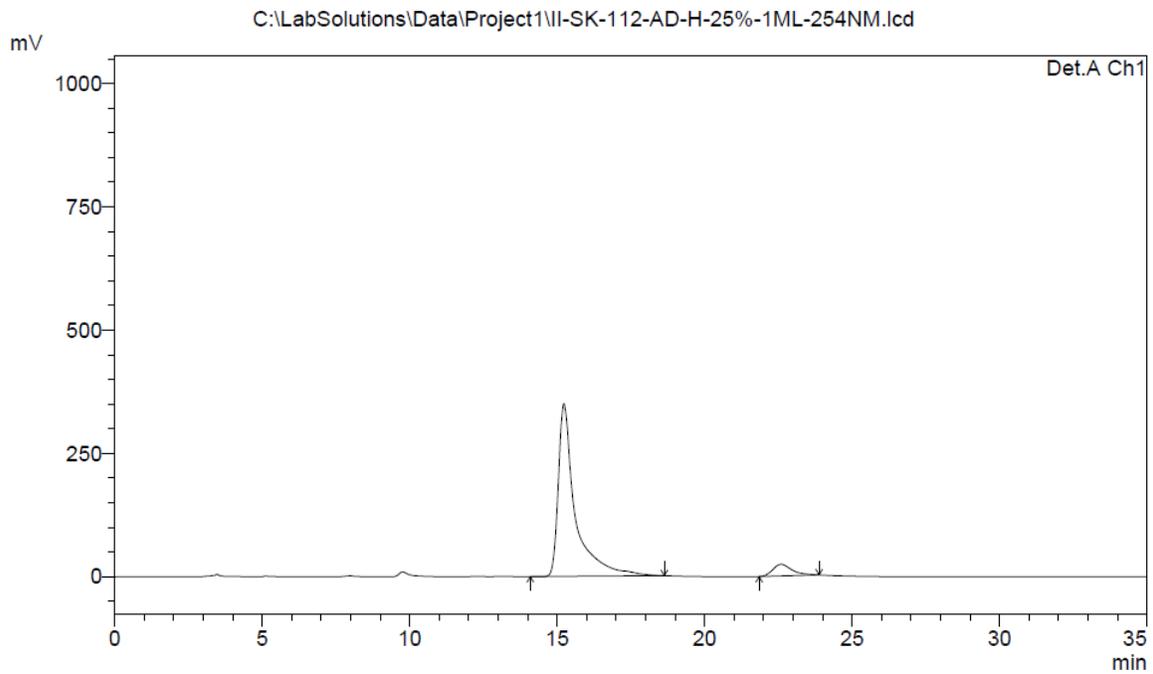


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.561	4237474	107410	50.736	59.806
2	23.042	4114554	72187	49.264	40.194
Total		8352028	179597	100.000	100.000

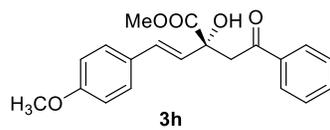




PeakTable

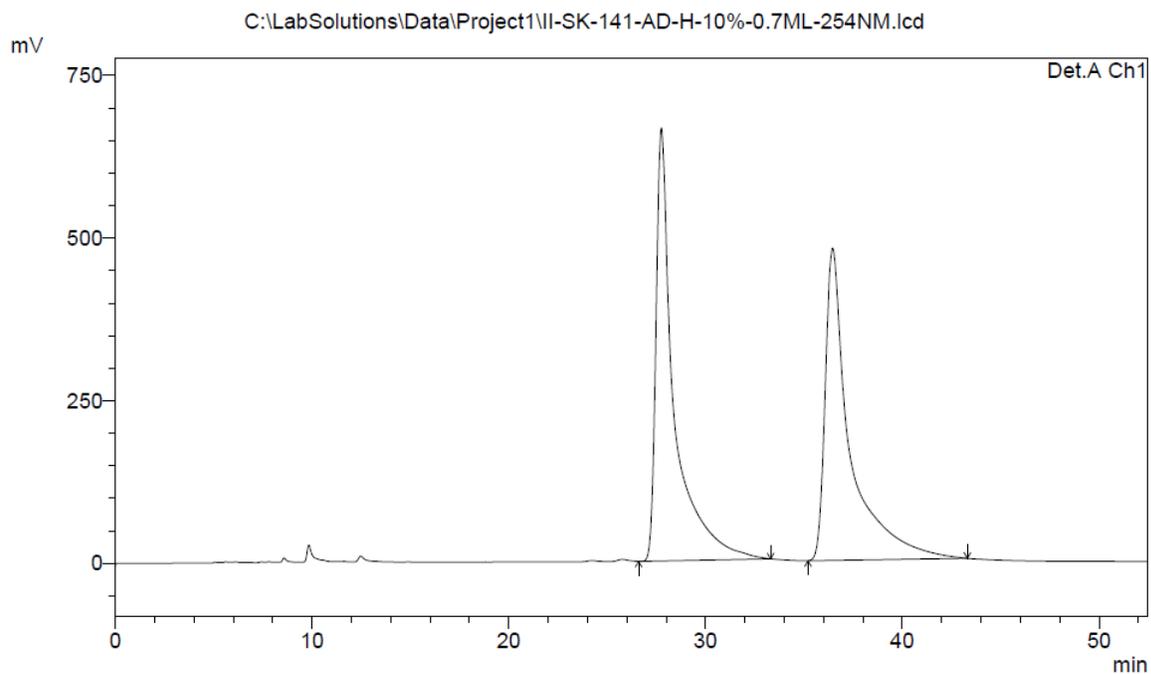
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.229	14057536	350468	92.910	93.670
2	22.591	1072786	23685	7.090	6.330
Total		15130322	374153	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

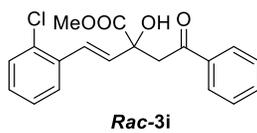
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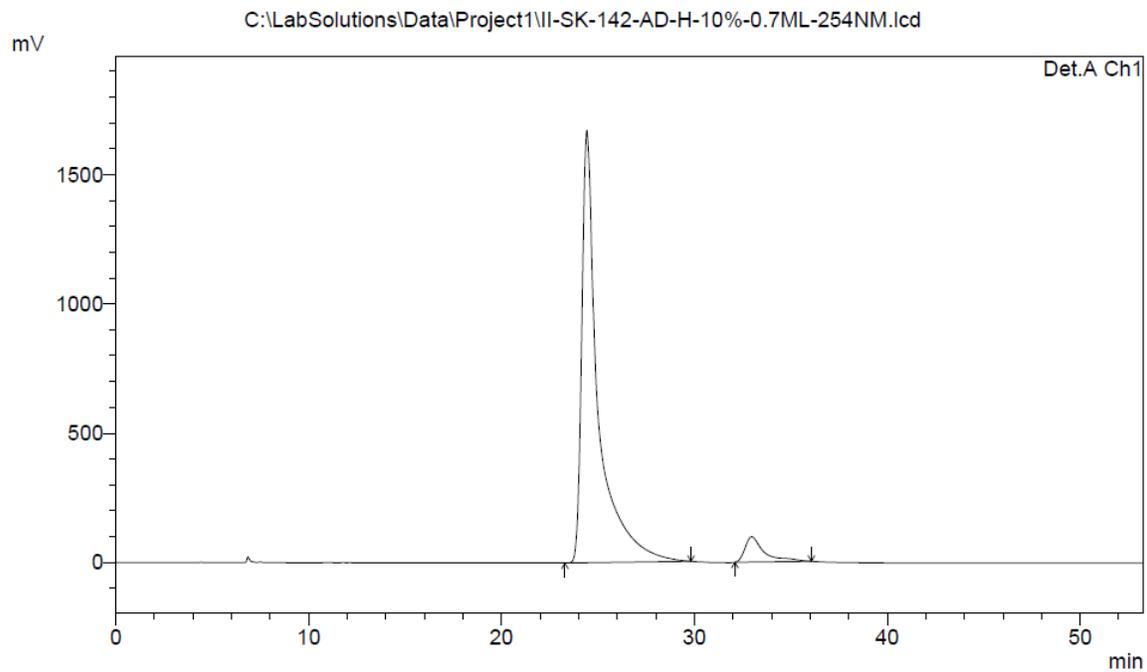


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.751	42582150	665580	50.319	58.094
2	36.449	42041530	480105	49.681	41.906
Total		84623680	1145685	100.000	100.000

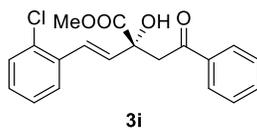




PeakTable

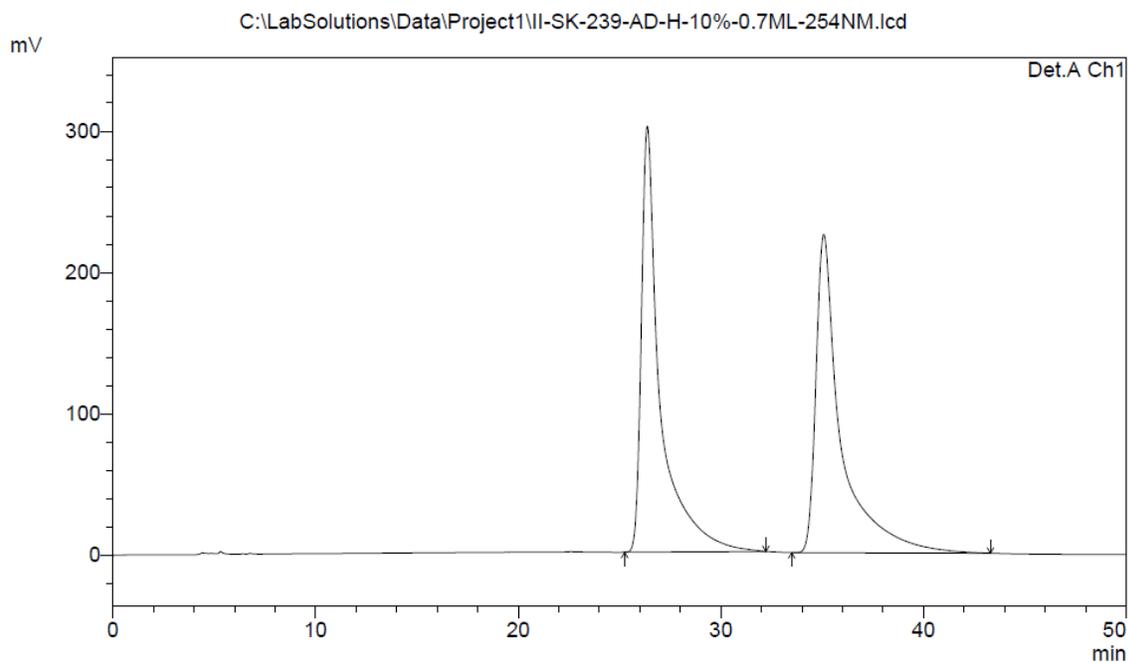
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	24.422	99121256	1671555	93.589	94.453
2	32.969	6789581	98167	6.411	5.547
Total		105910837	1769722	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

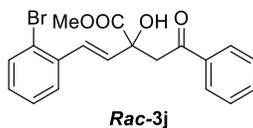
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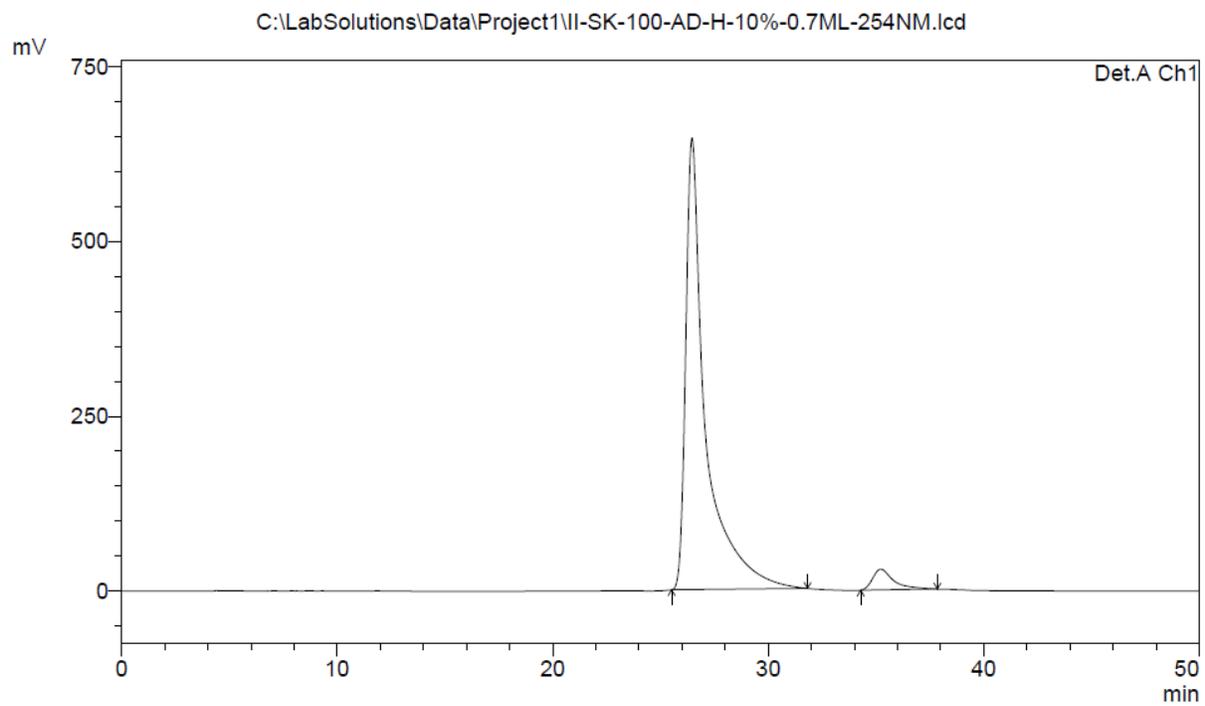


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.363	19473294	301611	50.129	57.229
2	35.065	19372978	225413	49.871	42.771
Total		38846272	527024	100.000	100.000

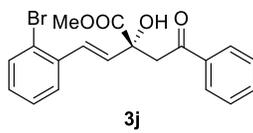




PeakTable

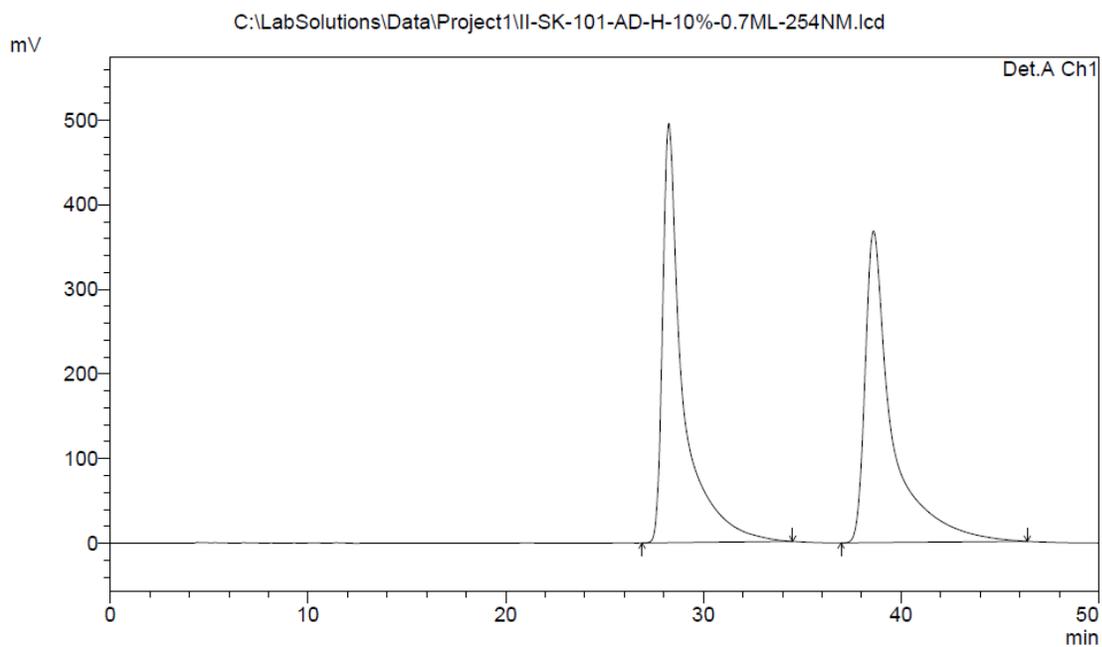
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	26.456	41189605	645959	95.352	95.605
2	35.214	2007660	29693	4.648	4.395
Total		43197265	675653	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

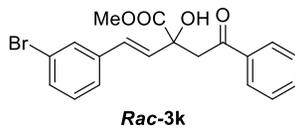
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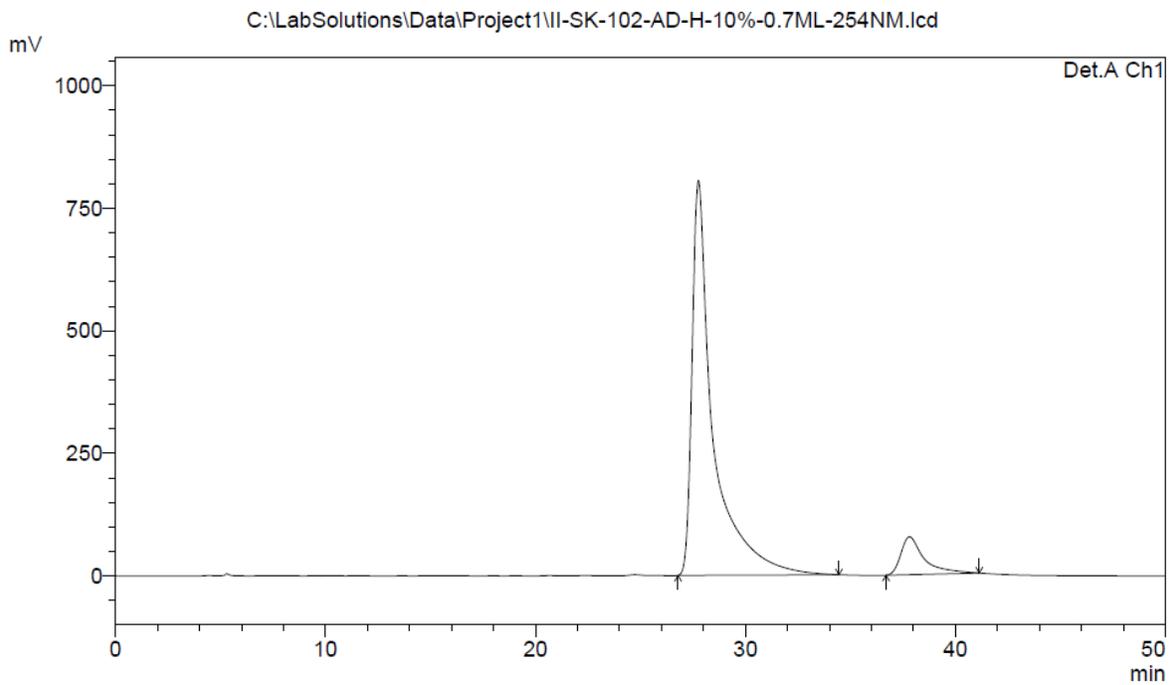


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.243	35162654	495874	50.267	57.376
2	38.594	34789392	368377	49.733	42.624
Total		69952046	864251	100.000	100.000

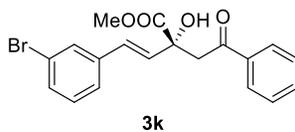




PeakTable

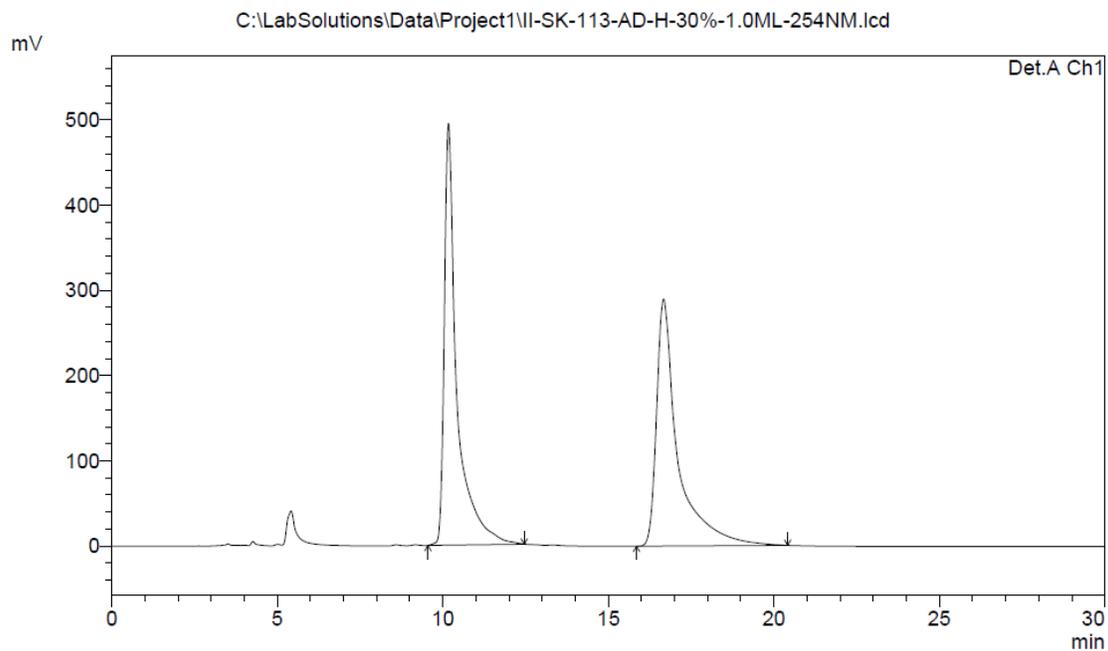
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	27.751	55963214	805155	90.334	91.238
2	37.801	5987922	77320	9.666	8.762
Total		61951136	882474	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

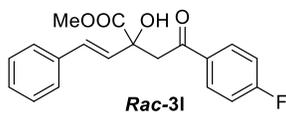
<Chromatogram>

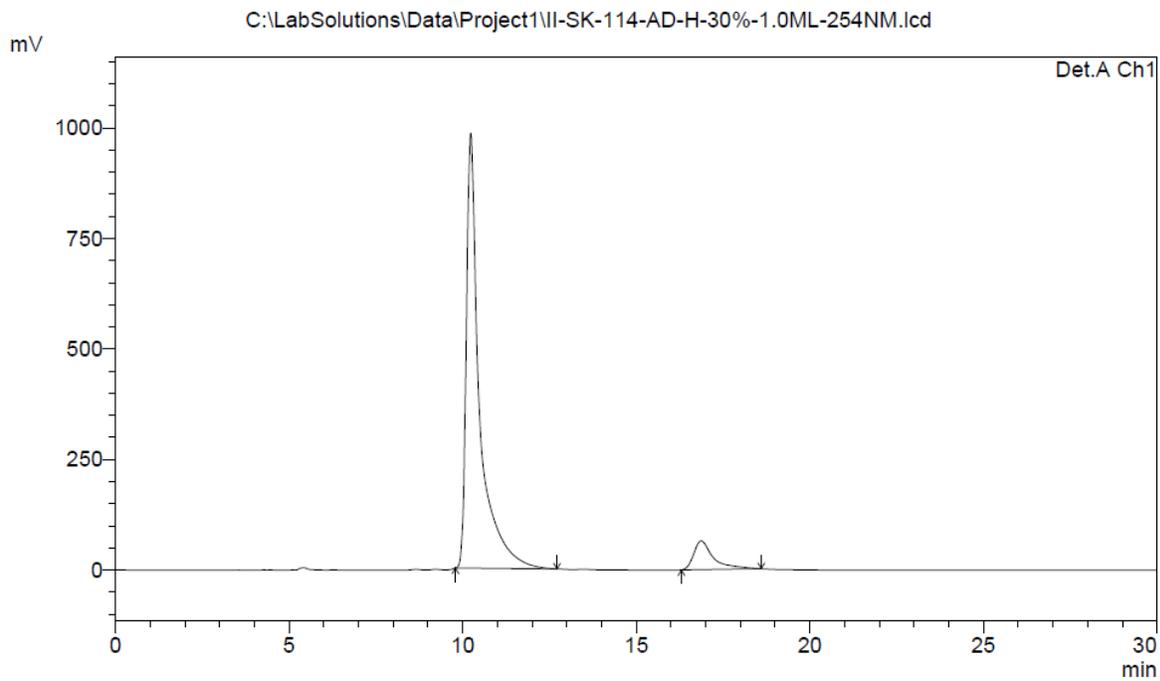


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.167	13040226	494559	50.249	63.058
2	16.666	12911202	289730	49.751	36.942
Total		25951428	784289	100.000	100.000

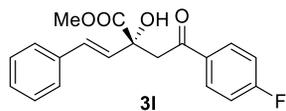




PeakTable

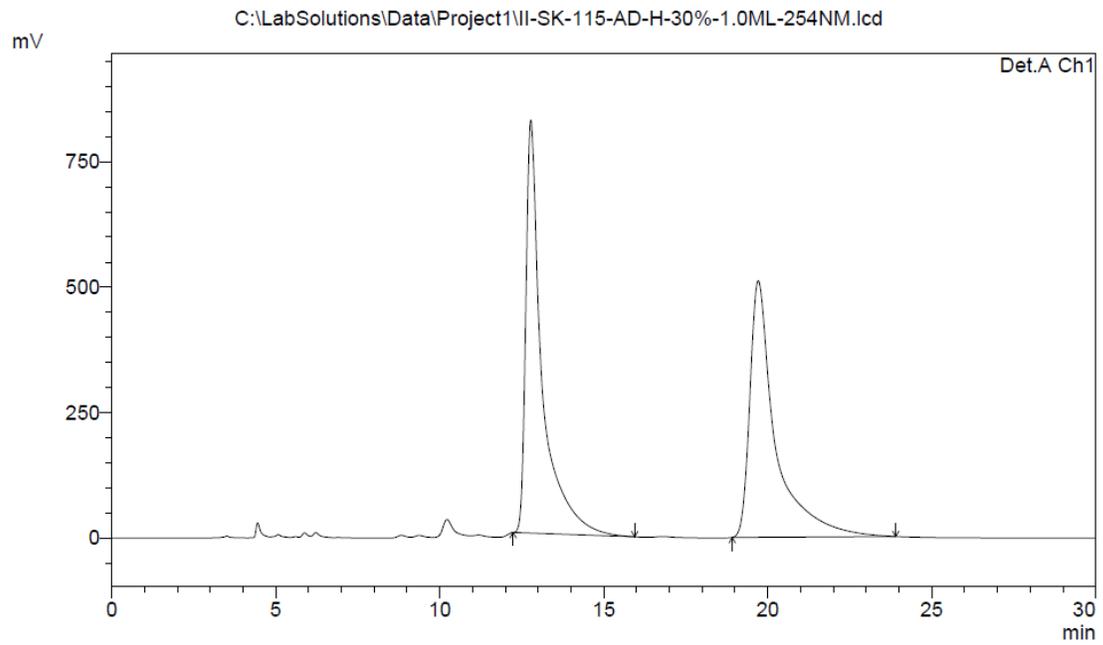
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.228	26054206	983411	91.035	93.805
2	16.867	2565710	64948	8.965	6.195
Total		28619916	1048359	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

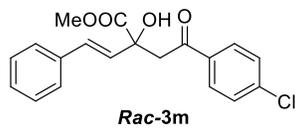
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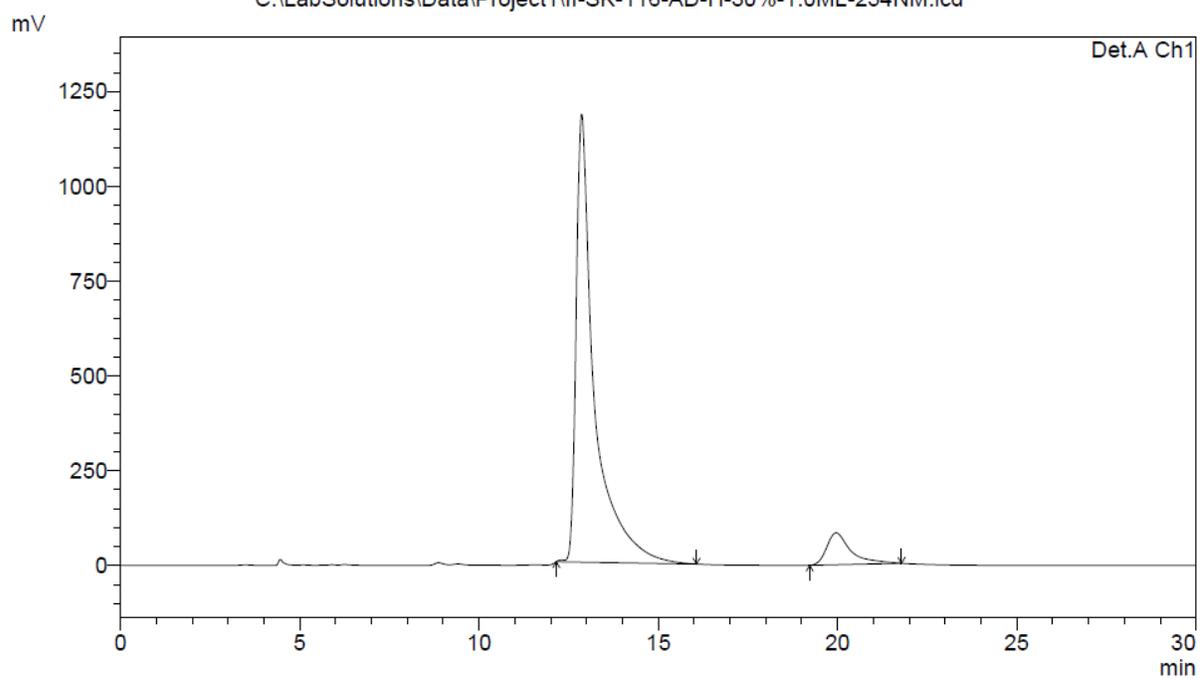
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.773	28336034	823058	50.587	61.668
2	19.706	27677898	511593	49.413	38.332
Total		56013933	1334651	100.000	100.000



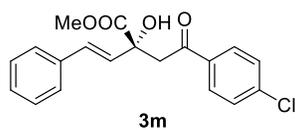
C:\LabSolutions\Data\Project1\II-SK-116-AD-H-30%-1.0ML-254NM.lcd



PeakTable

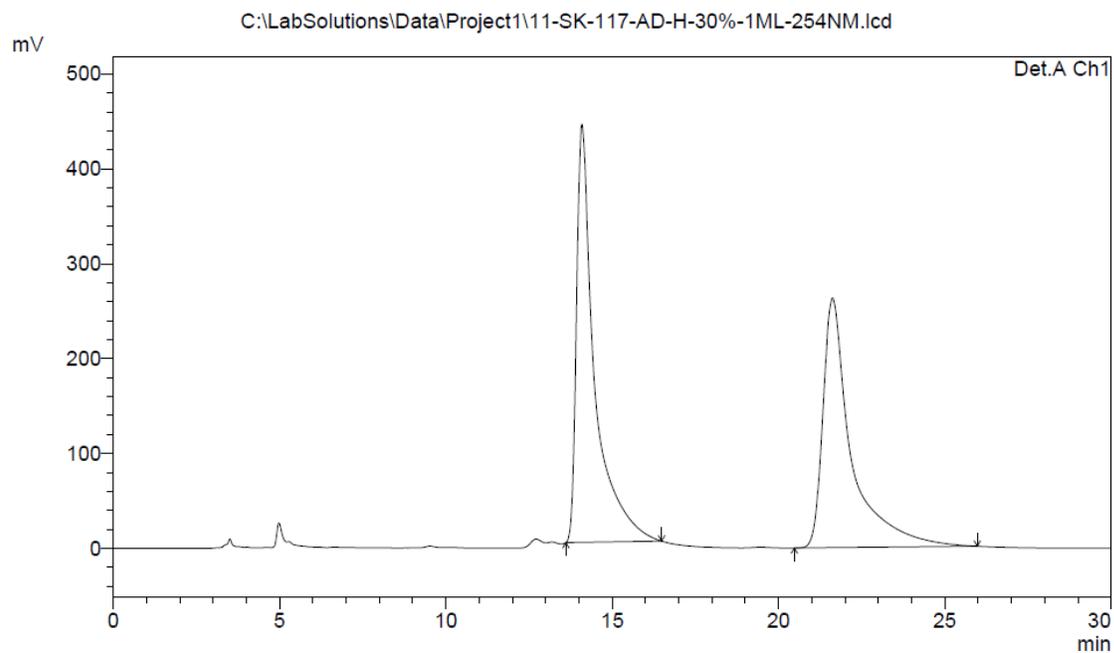
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.859	41673610	1181174	91.411	93.362
2	19.956	3915510	83986	8.589	6.638
Total		45589120	1265160	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

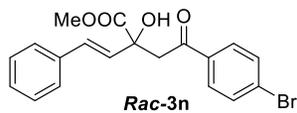
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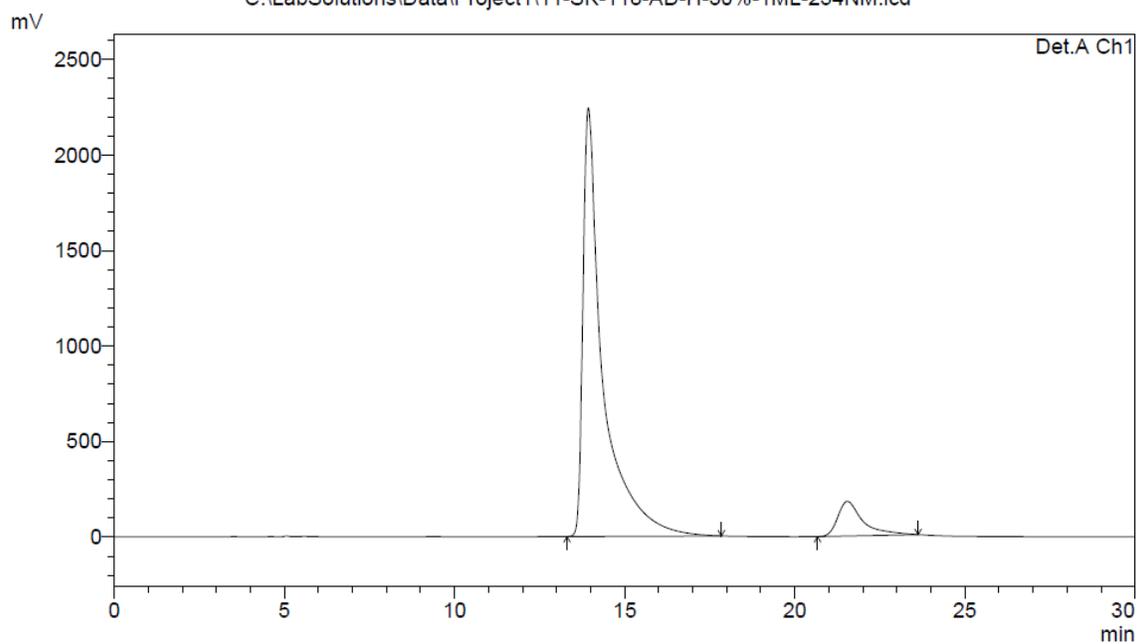
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	14.087	16651856	440740	51.658	62.616
2	21.619	15583116	263134	48.342	37.384
Total		32234972	703874	100.000	100.000



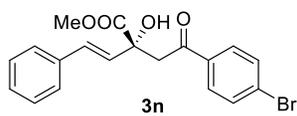
C:\LabSolutions\Data\Project1\11-SK-118-AD-H-30%-1ML-254NM.lcd



PeakTable

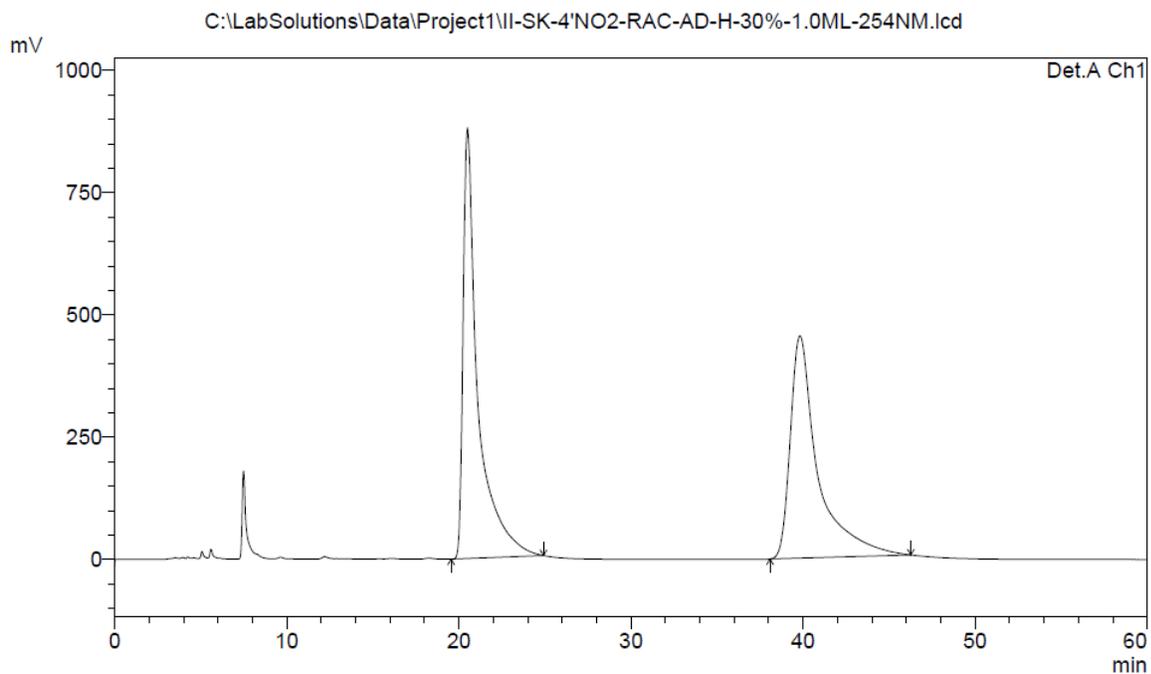
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.930	91153359	2245505	90.731	92.520
2	21.542	9312103	181548	9.269	7.480
Total		100465462	2427053	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

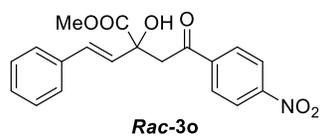
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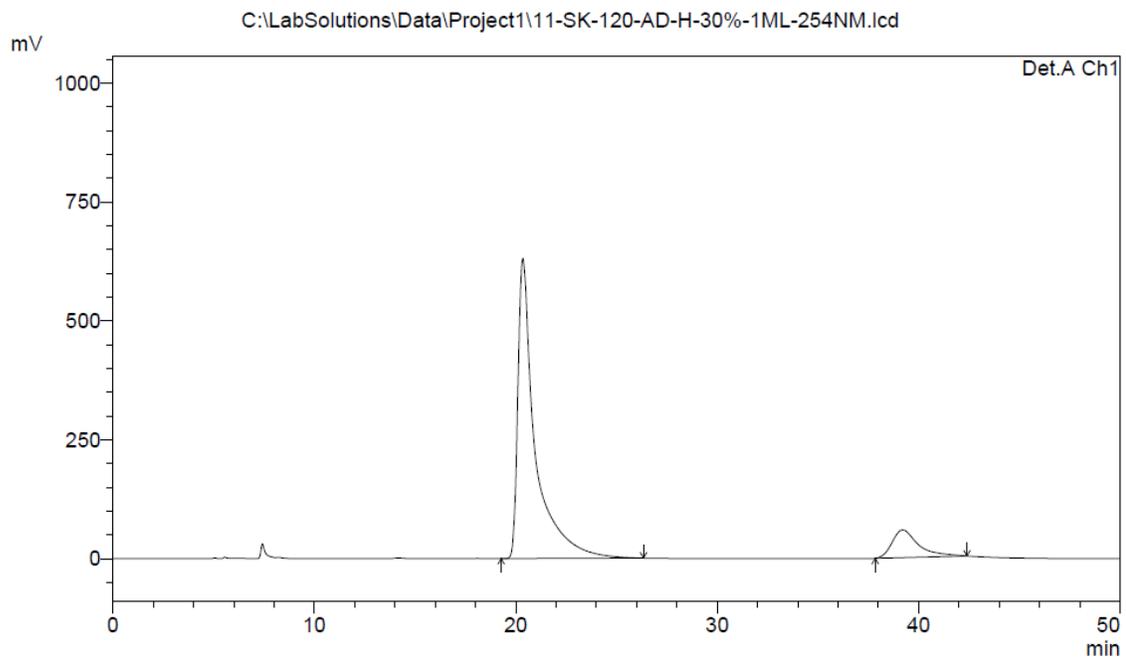


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.496	52439806	880786	50.958	65.947
2	39.815	50468991	454813	49.042	34.053
Total		102908798	1335599	100.000	100.000

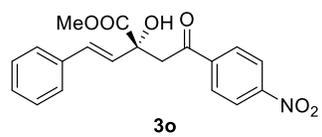


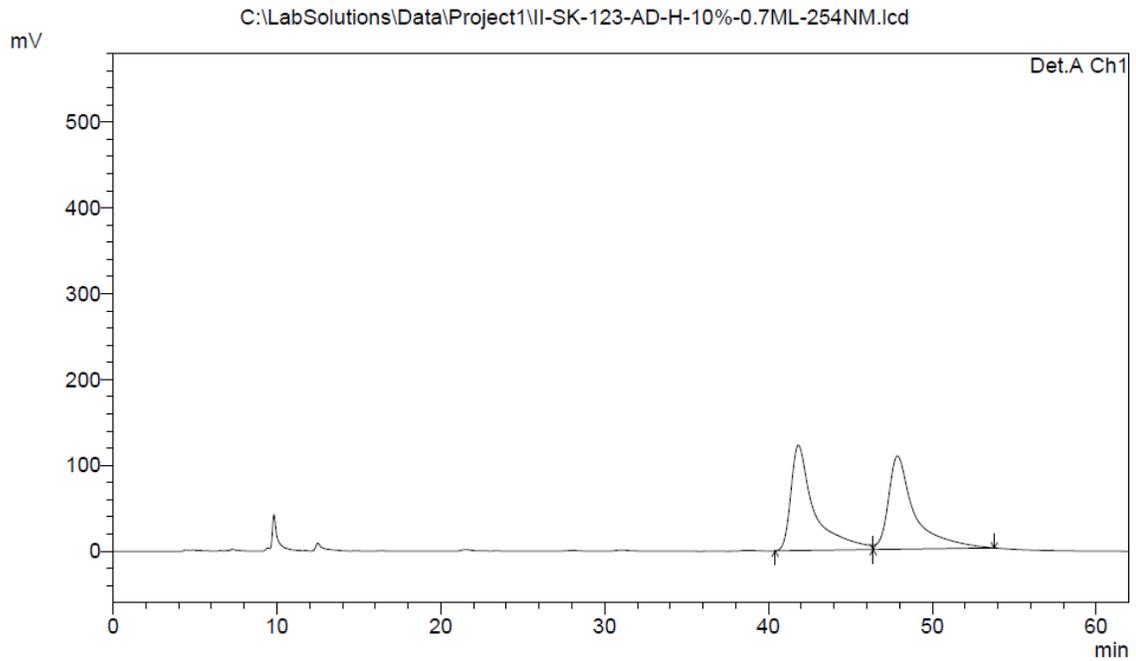


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	20.348	37640379	631344	87.656	91.545
2	39.197	5300408	58307	12.344	8.455
Total		42940786	689651	100.000	100.000

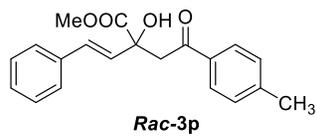


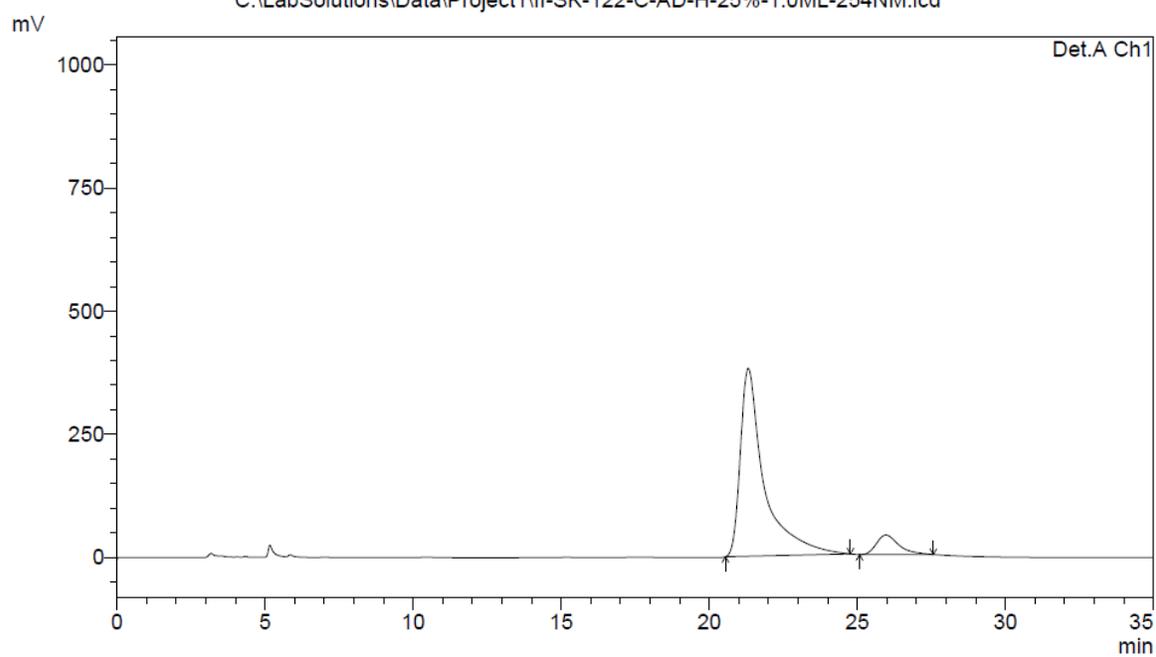


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	41.820	12194738	122825	50.276	53.045
2	47.870	12061020	108723	49.724	46.955
Total		24255758	231549	100.000	100.000

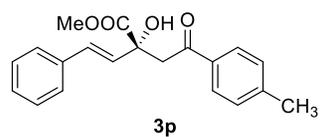


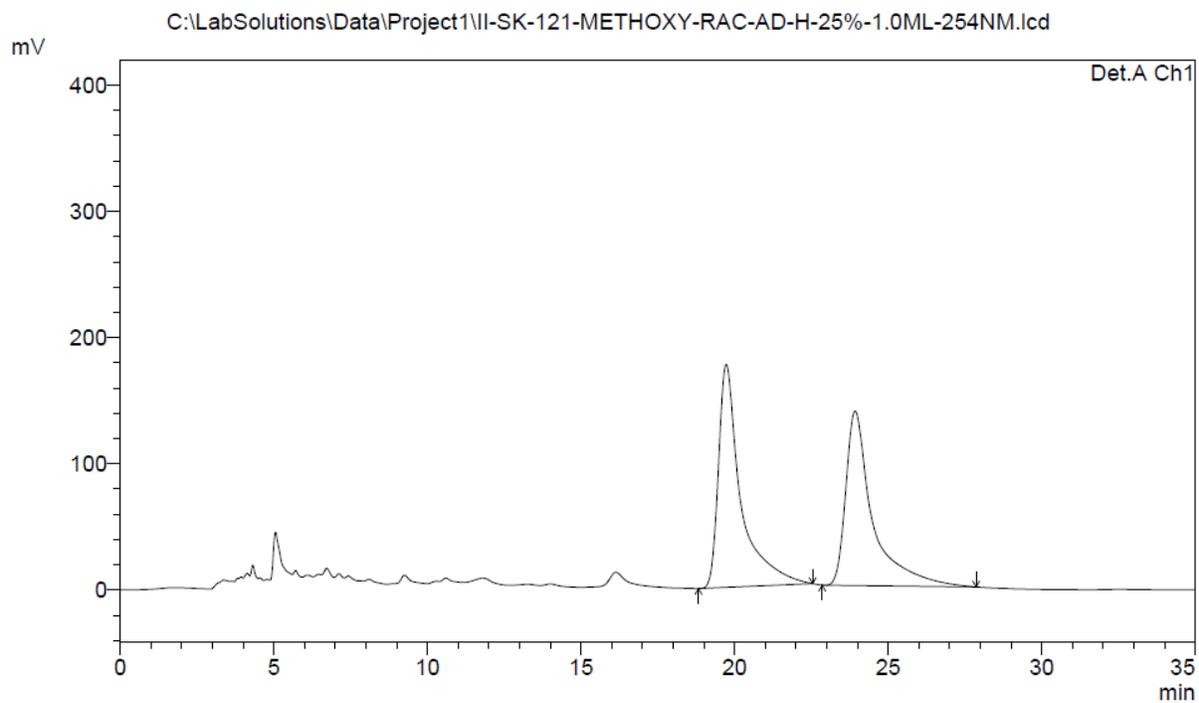


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.314	21366531	381562	91.060	90.569
2	25.963	2097626	39730	8.940	9.431
Total		23464157	421292	100.000	100.000

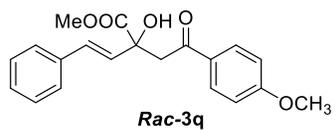


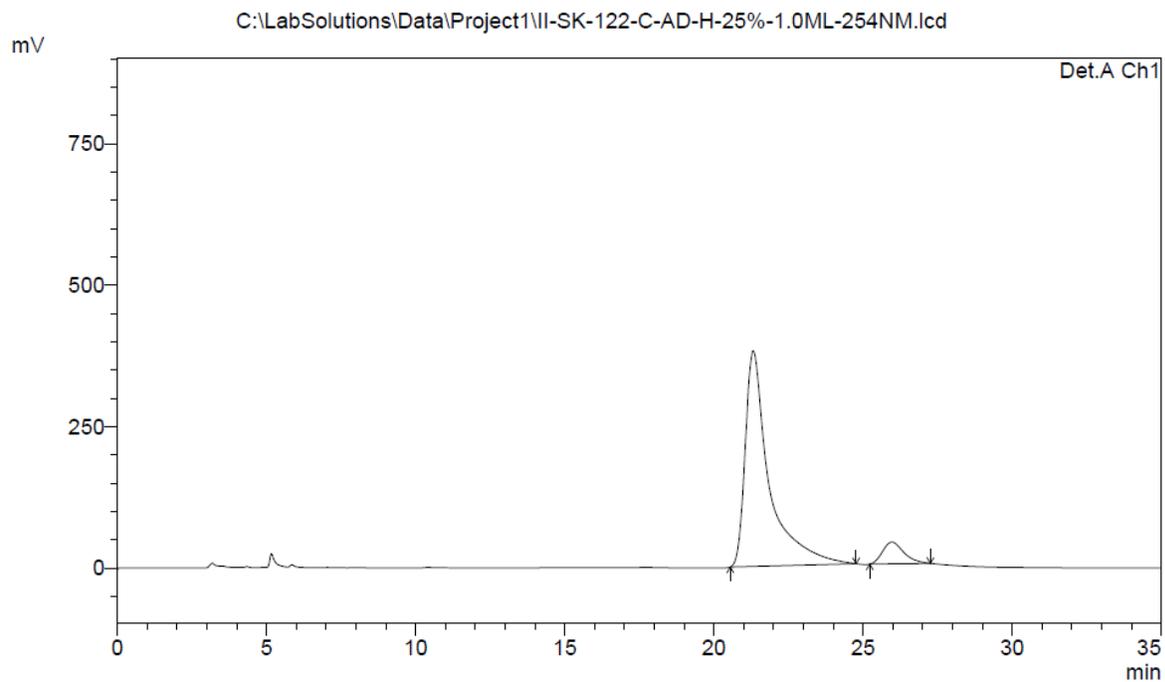


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.724	8932818	176737	51.503	56.083
2	23.920	8411507	138397	48.497	43.917
Total		17344324	315133	100.000	100.000

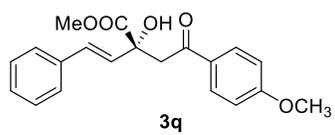




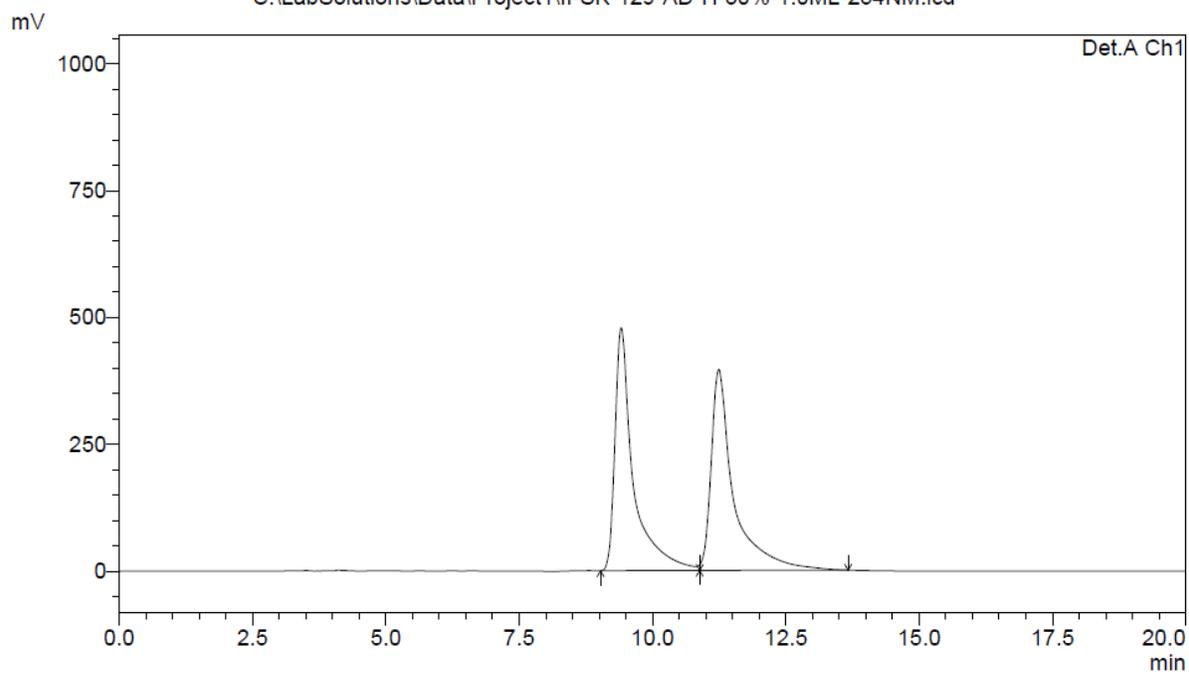
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	21.314	21366531	381562	91.753	90.820
2	25.963	1920389	38567	8.247	9.180
Total		23286920	420129	100.000	100.000



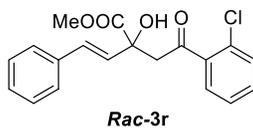
C:\LabSolutions\Data\Project1\II-SK-129-AD-H-30%-1.0ML-254NM.lcd

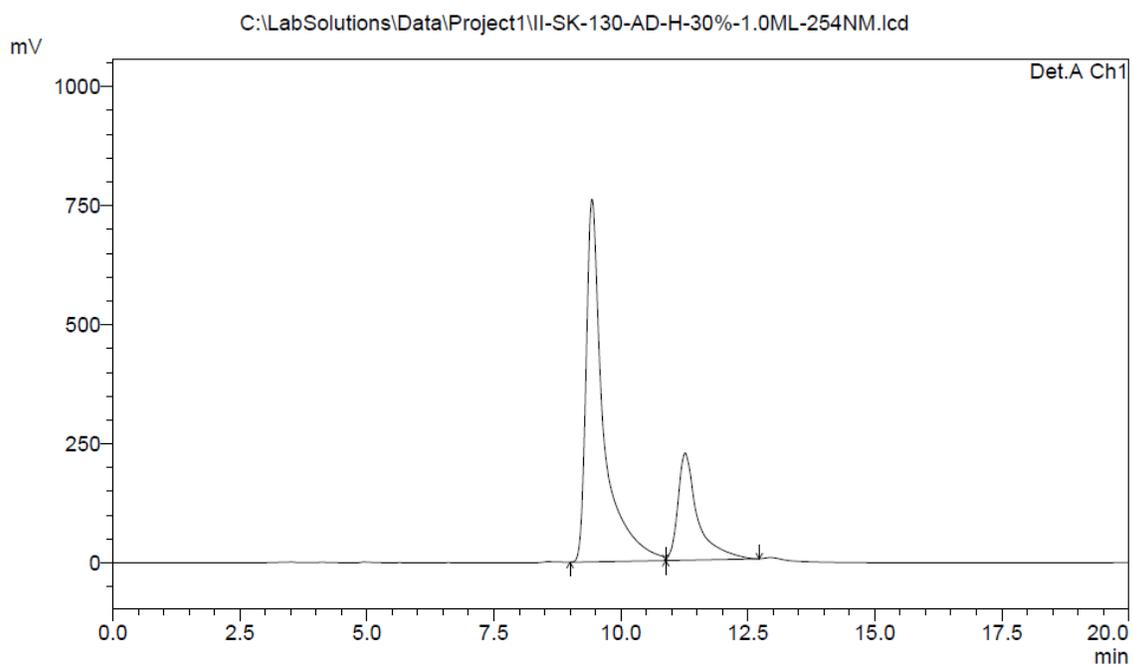


PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.408	11476812	478768	49.689	54.715
2	11.238	11620321	396250	50.311	45.285
Total		23097133	875019	100.000	100.000

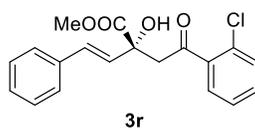




PeakTable

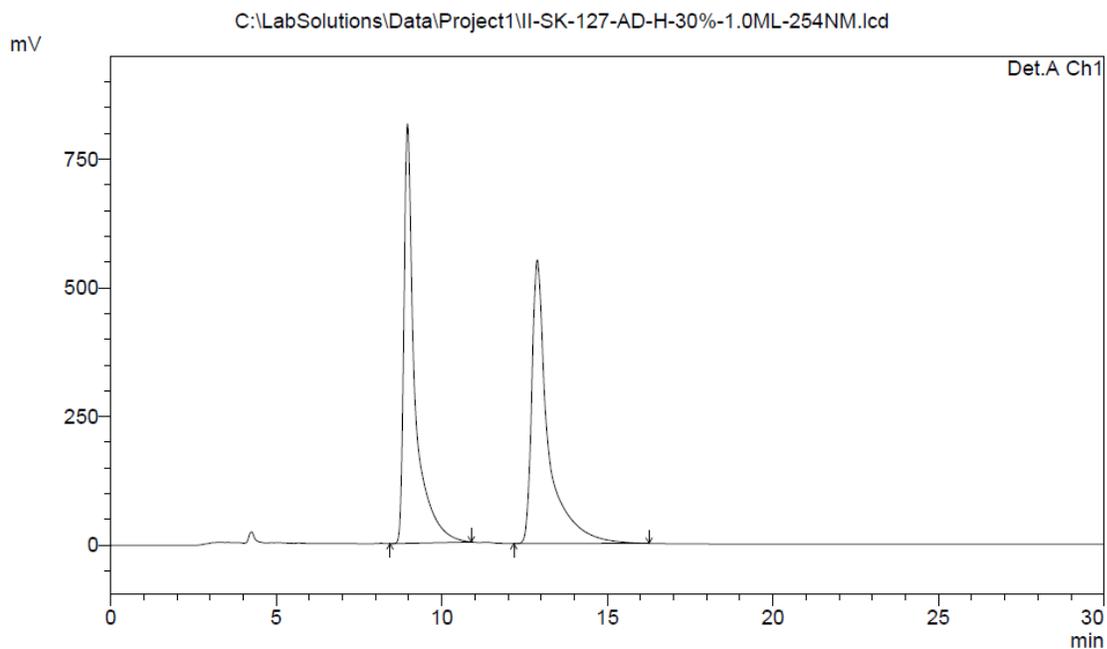
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.428	18162472	761899	74.873	77.203
2	11.262	6095382	224982	25.127	22.797
Total		24257854	986881	100.000	100.000



==== Shimadzu LCsolution LC Data Image ====

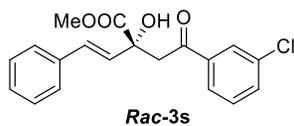
<Chromatogram>



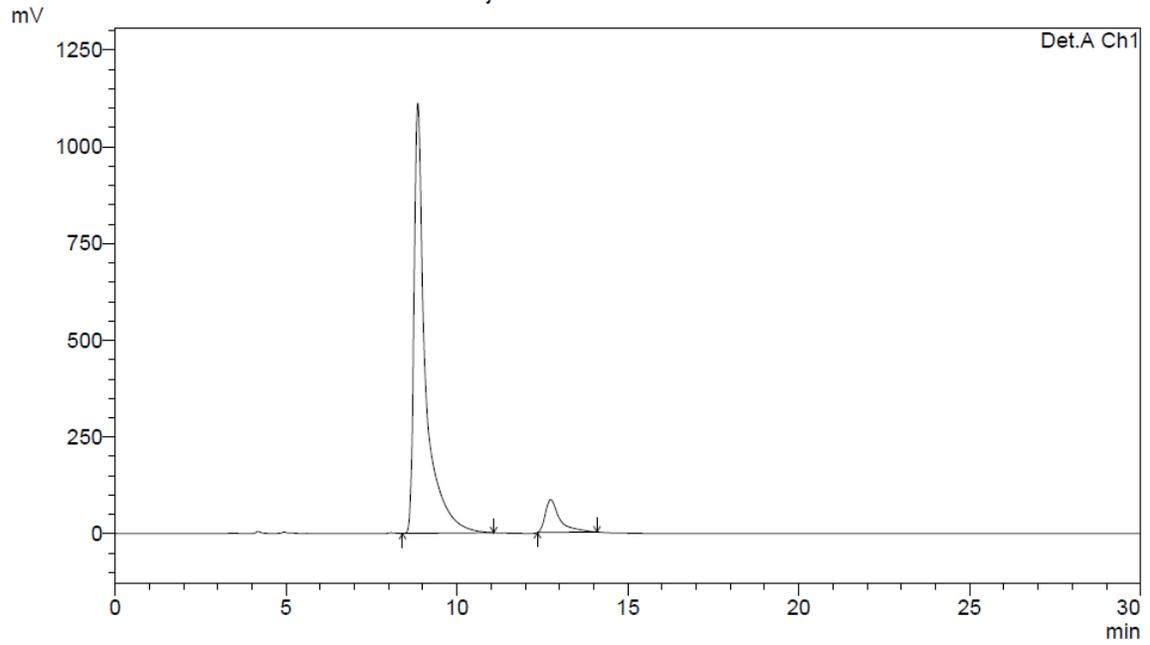
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.960	18836885	815168	49.920	59.664
2	12.878	18897561	551106	50.080	40.336
Total		37734445	1366274	100.000	100.000



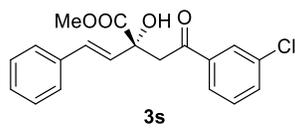
C:\LabSolutions\Data\Project1\II-SK-128-AD-H-30%-1.0ML-254NM.lcd



PeakTable

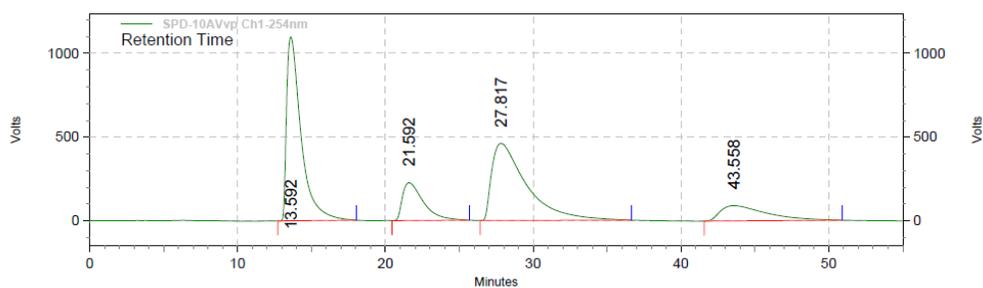
Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.850	25856861	1111664	91.119	92.880
2	12.739	2520285	85223	8.881	7.120
Total		28377146	1196887	100.000	100.000



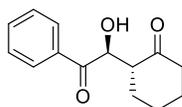
Area % Report

Method Name: C:\Documents and Settings\Administrator\Desktop\murali\Styrene oxide,0.1ml,1%IPA,254nm.met
 Data: C:\EZStart\Projects\Default\Data\IV-GQS-190-AS-10%-1ML-RAC
 User: System
 Acquired: 8/18/2010 3:27:03 PM
 Printed: 10/9/2012 8:27:21 PM



SPD-10AVvp
 Ch1-254nm
 Results

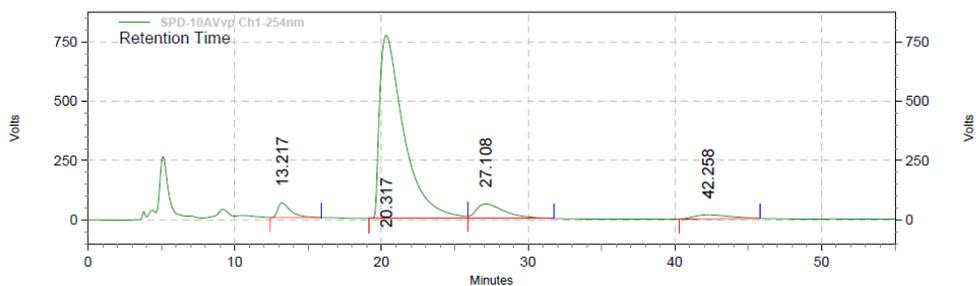
Retention Time	Area	Area %	Height	Height %
13.592	75021152	39.00	1097379	58.52
21.592	22108915	11.49	225514	12.03
27.817	75548552	39.27	461008	24.59
43.558	19689736	10.24	91212	4.86
Totals	192368355	100.00	1875113	100.00



Rac-22a

Area % Report

Method Name: C:\Documents and Settings\Administrator\Desktop\murali\Styrene oxide,0.1ml,1%IPA,254nm.met
 Data: C:\EZStart\Projects\Default\Data\V-GQS-59-AS-10%-1ML-CHIRAL
 User: System
 Acquired: 9/19/2010 1:37:53 PM
 Printed: 10/9/2012 8:29:39 PM

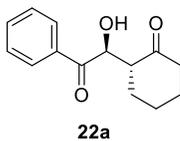


SPD-10AVvp

Ch1-254nm

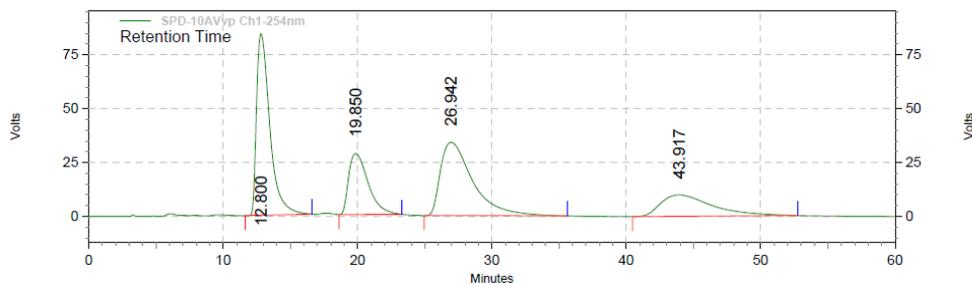
Results

Retention Time	Area	Area %	Height	Height %
13.217	3786473	3.67	61991	6.80
20.317	88234416	85.62	771447	84.66
27.108	8249529	8.00	61060	6.70
42.258	2785207	2.70	16703	1.83
Totals	103055625	100.00	911201	100.00



Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
 Data: C:\EZStart\Projects\Default\Data\VI-GQS-134-AS-10%-1ML-RAC
 User: System
 Acquired: 1/25/2011 12:41:38 PM
 Printed: 1/25/2011 1:45:15 PM

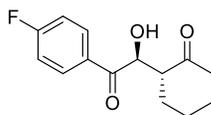


SPD-10AVvp

Ch1-254nm

Results

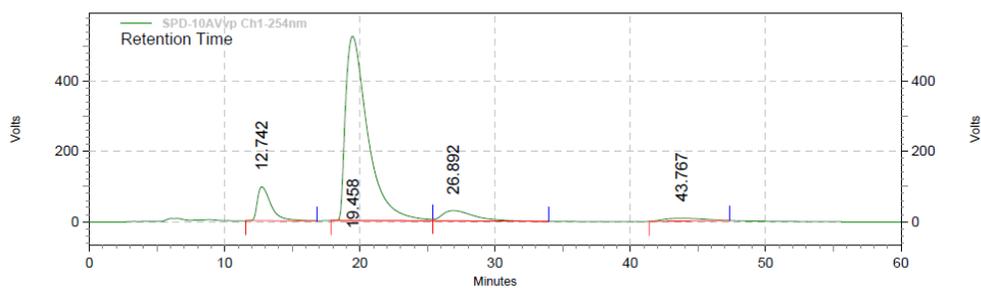
Retention Time	Area	Area %	Height	Height %
12.800	5815540	34.16	84207	53.83
19.850	2844804	16.71	28269	18.07
26.942	5687792	33.41	33993	21.73
43.917	2674931	15.71	9961	6.37
Totals	17023067	100.00	156430	100.00



Rac-22b

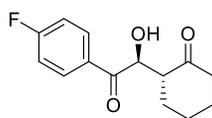
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
Data: C:\EZStart\Projects\Default\Data\VI-GQS-134-AS-10%-1ML-CHIRAL
User: System
Acquired: 1/25/2011 1:47:03 PM
Printed: 1/25/2011 2:59:21 PM



SPD-10AVvp
Ch1-254nm
Results

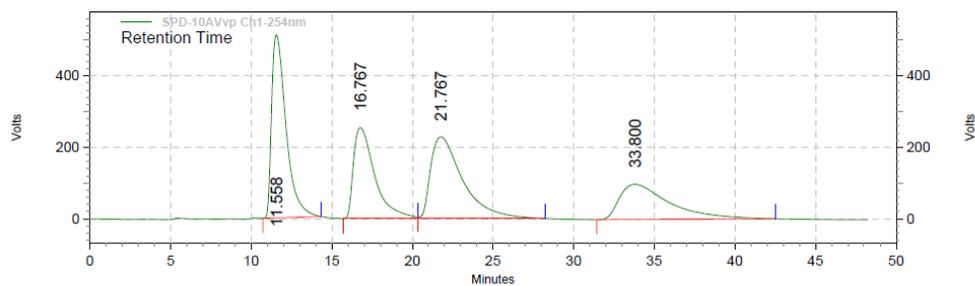
Retention Time	Area	Area %	Height	Height %
12.742	6687241	9.18	96366	14.63
19.458	59675097	81.88	524763	79.66
26.892	4902981	6.73	29526	4.48
43.767	1619245	2.22	8138	1.24
Totals	72884564	100.00	658793	100.00



22b

Area % Report

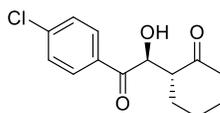
Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
 Data: C:\EZStart\Projects\Default\Data\VI-GQS-123-AS-10%-1ML-RAC1
 User: System
 Acquired: 1/19/2011 2:07:03 PM
 Printed: 1/19/2011 2:55:36 PM



SPD-10AVvp
Ch1-254nm

Results

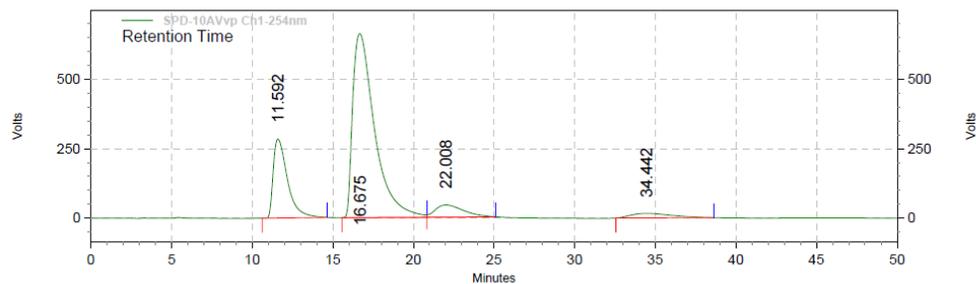
Retention Time	Area	Area %	Height	Height %
11.558	31881526	29.58	511661	46.84
16.767	23221802	21.55	254167	23.27
21.767	31522953	29.25	228467	20.91
33.800	21153631	19.63	98103	8.98
Totals	107779912	100.00	1092398	100.00



Rac-22c

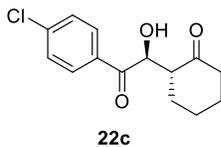
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
Data: C:\EZStart\Projects\Default\Data\VI-GQS-123-AS-10%-1ML-CHIRAL
User: System
Acquired: 1/19/2011 2:56:26 PM
Printed: 1/19/2011 4:20:51 PM



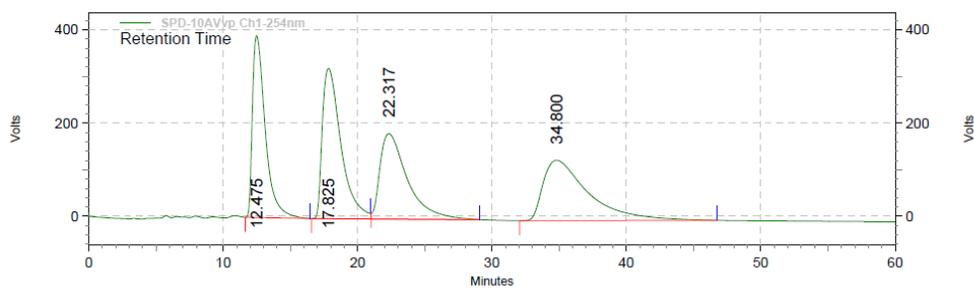
SPD-10AVvp
Ch1-254nm
Results

Retention Time	Area	Area %	Height	Height %
11.592	17434636	19.80	283971	28.21
16.675	62383345	70.84	662698	65.83
22.008	5366264	6.09	43586	4.33
34.442	2881857	3.27	16395	1.63
Totals	88066102	100.00	1006650	100.00



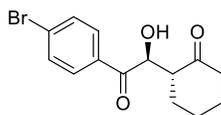
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
 Data: C:\EZStart\Projects\Default\Data\VI-GQS-120-AS-10%-1ML-RAC1
 User: System
 Acquired: 1/19/2011 11:03:06 AM
 Printed: 1/19/2011 12:06:54 PM



SPD-10AVvp
 Ch1-254nm
 Results

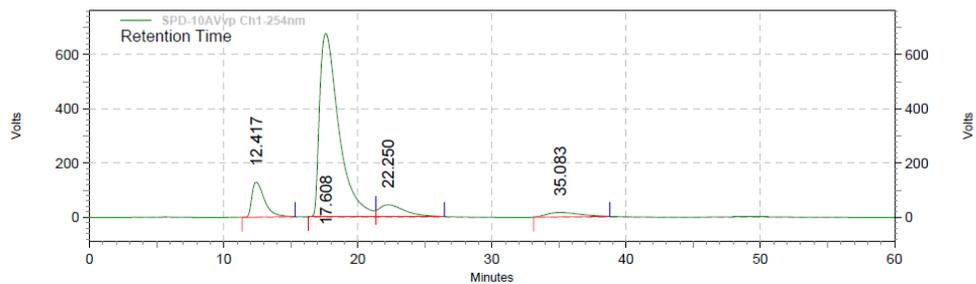
Retention Time	Area	Area %	Height	Height %
12.475	26009230	22.89	389820	38.02
17.825	31220566	27.48	322372	31.44
22.317	26018386	22.90	183335	17.88
34.800	30371878	26.73	129818	12.66
Totals	113620060	100.00	1025345	100.00



Rac-22d

Area % Report

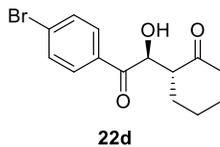
Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
 Data: C:\EZStart\Projects\Default\Data\VI-GQS-120-AS-10%-1ML-CHIRAL
 User: System
 Acquired: 1/19/2011 12:18:15 PM
 Printed: 1/19/2011 1:21:41 PM



SPD-10AVvp
 Ch1-254nm

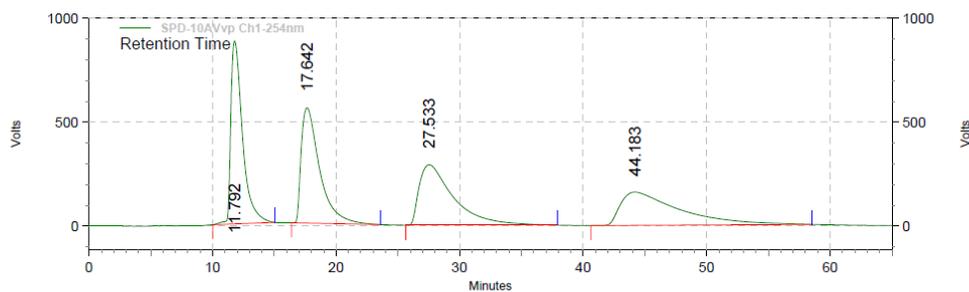
Results

Retention Time	Area	Area %	Height	Height %
12.417	8374138	9.91	128825	14.92
17.608	67565456	79.98	675306	78.19
22.250	5774506	6.84	43404	5.03
35.083	2767200	3.28	16116	1.87
Totals	84481300	100.00	863651	100.00



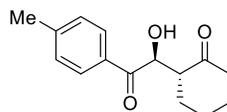
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
Data: C:\EZStart\Projects\Default\Data\VI-GQS-125-AS-10%-1ML-RAC
User: System
Acquired: 1/19/2011 4:25:14 PM
Printed: 1/19/2011 5:41:34 PM



SPD-10AVvp
Ch1-254nm
Results

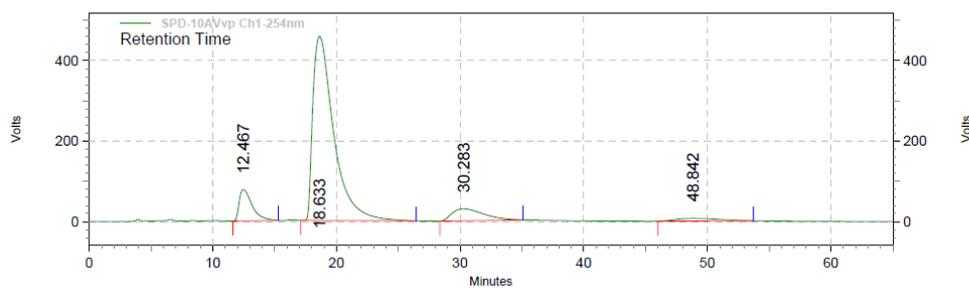
Retention Time	Area	Area %	Height	Height %
11.792	58123439	25.89	879464	46.66
17.642	57372653	25.55	554277	29.41
27.533	56298250	25.07	291061	15.44
44.183	52741483	23.49	159978	8.49
Totals	224535825	100.00	1884780	100.00



Rac-22e

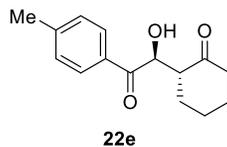
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
 Data: C:\EZStart\Projects\Default\Data\VI-GQS-125-AS-10%-1ML-CHIRAL
 User: System
 Acquired: 1/20/2011 11:03:30 AM
 Printed: 1/20/2011 12:13:14 PM



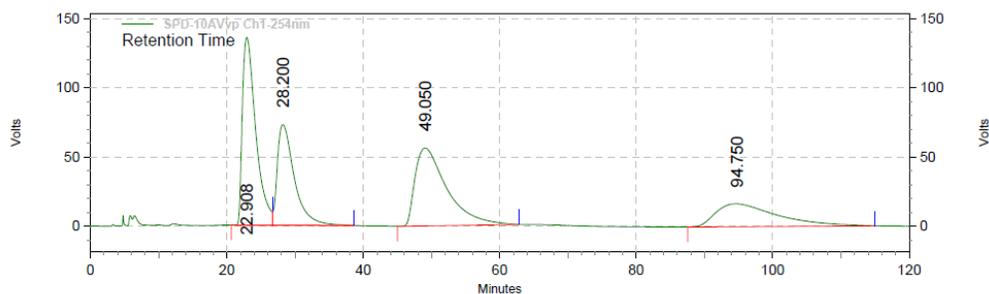
SPD-10AVvp
 Ch1-254nm
 Results

Retention Time	Area	Area %	Height	Height %
12.467	5647691	8.52	77954	13.64
18.633	53762790	81.10	457432	80.03
30.283	5318625	8.02	29644	5.19
48.842	1565507	2.36	6536	1.14
Totals	66294613	100.00	571566	100.00



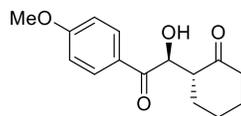
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
 Data: C:\EZStart\Projects\Default\Data\VI-GQS-35-AS-10%-1ML-RAC
 User: System
 Acquired: 12/10/2010 12:45:53 PM
 Printed: 12/10/2010 5:05:21 PM



SPD-10AVvp
 Ch1-254nm
 Results

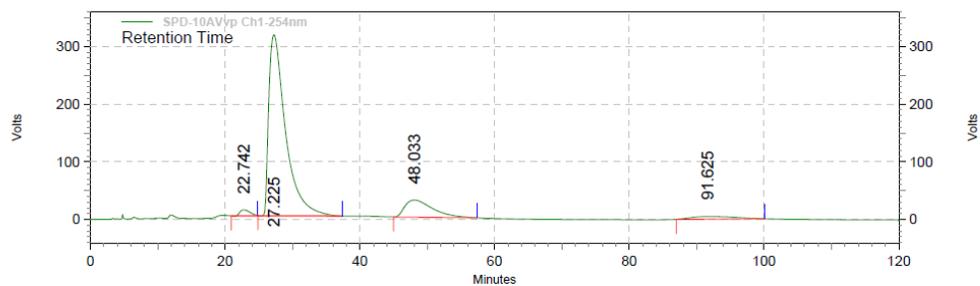
Retention Time	Area	Area %	Height	Height %
22.908	18475840	30.97	135576	48.25
28.200	12611654	21.14	72621	25.85
49.050	18218074	30.53	56208	20.01
94.750	10360415	17.36	16562	5.89
Totals	59665983	100.00	280967	100.00



Rac-22f

Area % Report

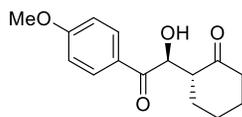
Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
Data: C:\EZStart\Projects\Default\Data\VI-GQS-35-AS-10%-1ML-CHIRAL
User: System
Acquired: 12/10/2010 10:38:33 AM
Printed: 12/10/2010 12:43:53 PM



SPD-10AVvp
Ch1-254nm

Results

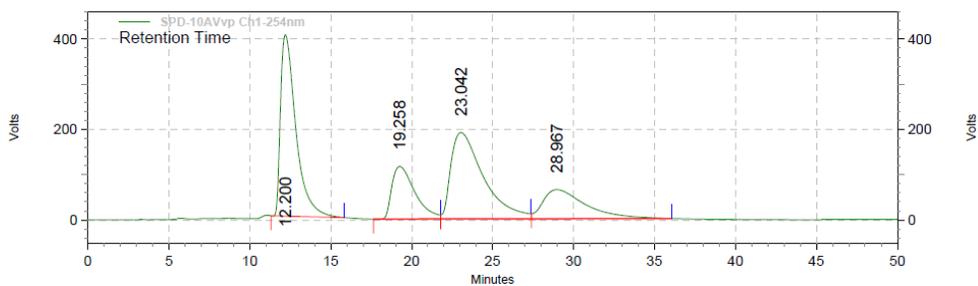
Retention Time	Area	Area %	Height	Height %
22.742	1083824	1.64	10553	2.94
27.225	54574902	82.37	314407	87.49
48.033	8544517	12.90	29700	8.26
91.625	2050079	3.09	4701	1.31
Totals	66253322	100.00	359361	100.00



22f

Area % Report

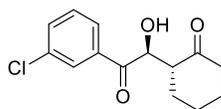
Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
 Data: C:\EZStart\Projects\Default\Data\VI-GQS-136-AS-10%-1ML-RAC
 User: System
 Acquired: 1/25/2011 3:02:14 PM
 Printed: 1/25/2011 3:56:41 PM



SPD-10AVvp Ch1-254nm

Results

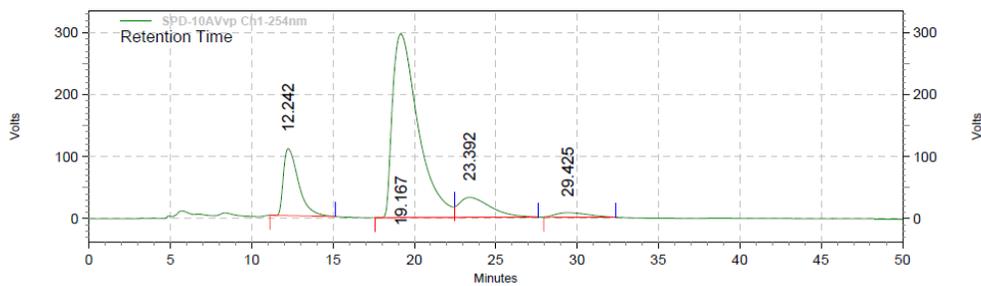
Retention Time	Area	Area %	Height	Height %
12.200	26481461	34.59	400938	51.89
19.258	11456812	14.97	116218	15.04
23.042	26774809	34.98	191062	24.73
28.967	11839529	15.47	64428	8.34
Totals	76552611	100.00	772646	100.00



Rac-22g

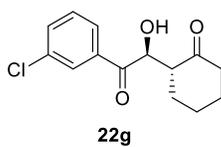
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
Data: C:\EZStart\Projects\Default\Data\VI-GQS-136-AS-10%-1ML-CHIRAL
User: System
Acquired: 1/25/2011 3:58:21 PM
Printed: 1/25/2011 4:48:44 PM



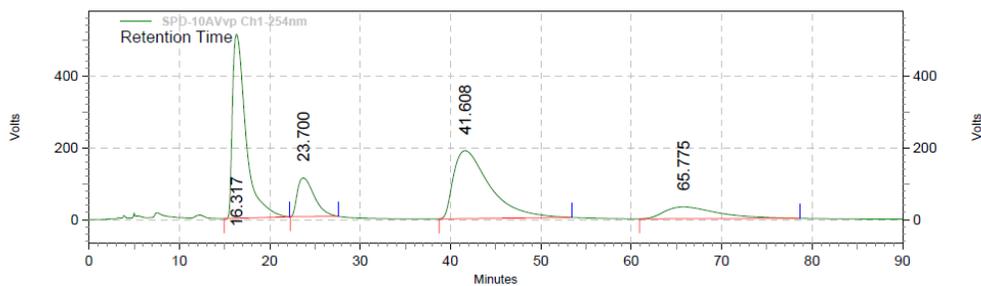
SPD-10AVvp Ch1-254nm Results

Retention Time	Area	Area %	Height	Height %
12.242	6940040	15.51	107911	24.34
19.167	32518056	72.66	296217	66.81
23.392	4318154	9.65	32137	7.25
29.425	978434	2.19	7101	1.60
Totals	44754684	100.00	443366	100.00



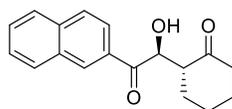
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
Data: C:\EZStart\Projects\Default\Data\VI-GQS-39-AS-10%-1ML-RAC
User: System
Acquired: 12/12/2010 4:32:28 PM
Printed: 12/12/2010 6:02:54 PM



SPD-10AVvp
Ch1-254nm
Results

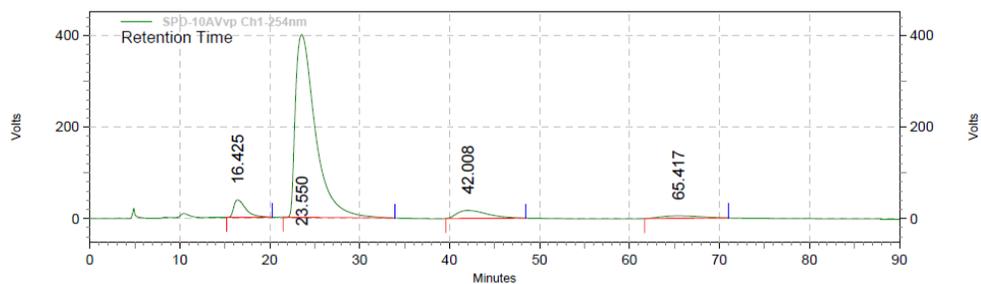
Retention Time	Area	Area %	Height	Height %
16.317	53146258	39.90	511183	60.86
23.700	13954087	10.48	107304	12.77
41.608	52692540	39.56	188812	22.48
65.775	13399599	10.06	32664	3.89
Totals	133192484	100.00	839963	100.00



Rac-22h

Area % Report

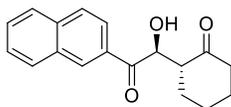
Method Name: C:\EZStart\Projects\Default\Methods\AS_10%_1mL.met
 Data: C:\EZStart\Projects\Default\Data\VI-GQS-39-AS-10%-1ML-CHIRAL
 User: System
 Acquired: 12/12/2010 6:03:58 PM
 Printed: 12/12/2010 7:35:14 PM



SPD-10AVvp
 Ch1-254nm

Results

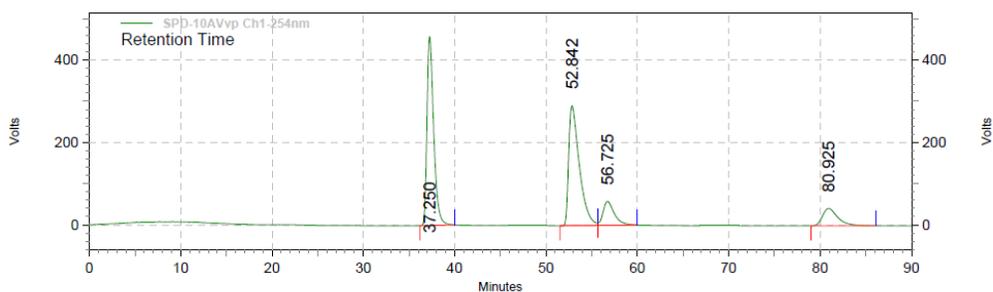
Retention Time	Area	Area %	Height	Height %
16.425	3558587	5.04	38092	8.29
23.550	61413767	86.91	399106	86.81
42.008	4089137	5.79	17337	3.77
65.417	1599352	2.26	5215	1.13
Totals	70660843	100.00	459750	100.00



22h

Area % Report

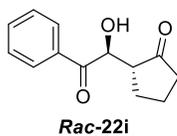
Method Name: C:\EZStart\Projects\Default\Methods\IC-10%_1ML_254NM.met
 Data: C:\Documents and
 Settings\Administrator\Desktop\murali\XI-GQS-182-IC-10%-1ML-RAC
 User: System
 Acquired: 9/23/2012 4:45:41 PM
 Printed: 9/23/2012 6:16:29 PM



SPD-10AVvp
 Ch1-254nm

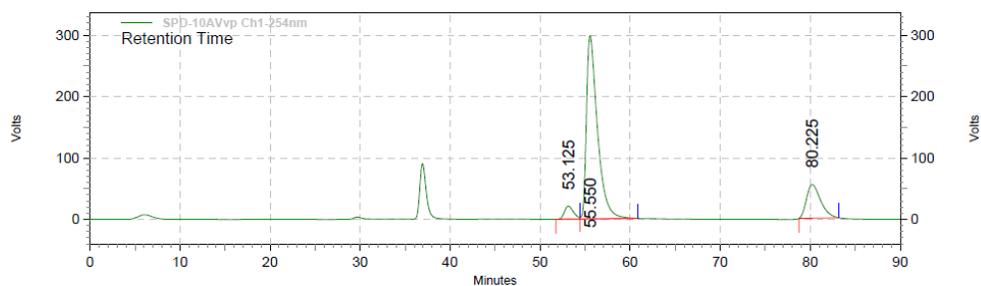
Results

Retention Time	Area	Area %	Height	Height %
37.250	23237132	41.10	458381	53.98
52.842	23466165	41.51	290491	34.21
56.725	4946338	8.75	58231	6.86
80.925	4883311	8.64	42061	4.95
Totals	56532946	100.00	849164	100.00



Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\IC-10%_1ML_254NM.met
 Data: C:\Documents and
 Settings\Administrator\Desktop\murali\XI-GQS-182-IC-10%-1ML-CHIR
 User: System
 Acquired: 9/23/2012 6:17:34 PM
 Printed: 9/23/2012 7:47:46 PM

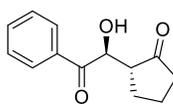


SPD-10AVvp

Ch1-254nm

Results

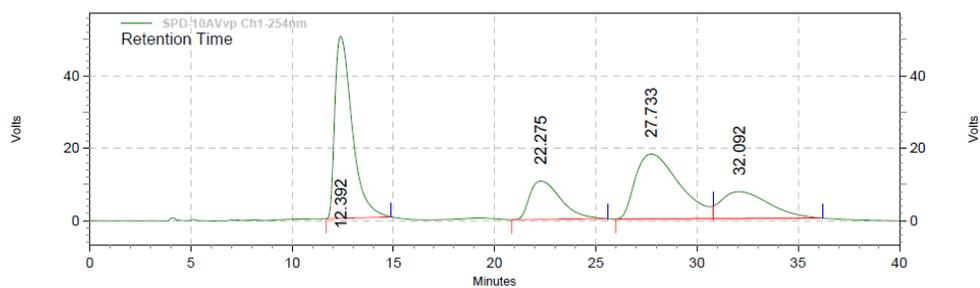
Retention Time	Area	Area %	Height	Height %
53.125	1477247	4.52	21247	5.67
55.550	25296525	77.32	298567	79.69
80.225	5944026	18.17	54839	14.64
Totals	32717798	100.00	374653	100.00



22i

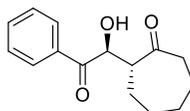
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\AS_8.5%_0.8mL.met
 Data: C:\EZStart\Projects\Default\Data\V-GQS-122-AS-8.5%-0.8ML-RAC
 User: System
 Acquired: 10/22/2010 11:53:03 AM
 Printed: 10/22/2010 12:37:42 PM



SPD-10AVvp
 Ch1-254nm
 Results

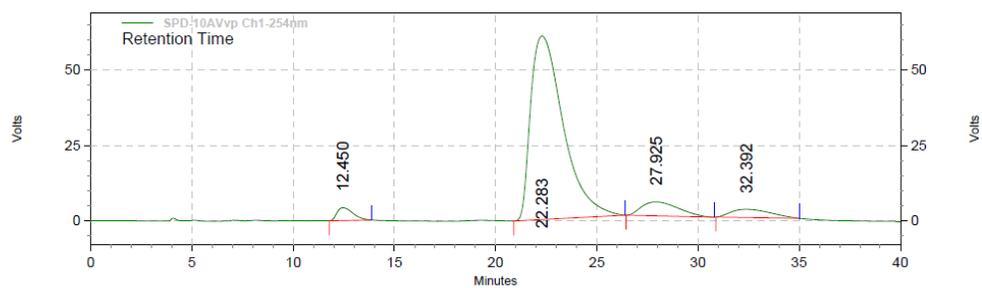
Retention Time	Area	Area %	Height	Height %
12.392	3000016	37.27	50196	58.38
22.275	1123442	13.96	10529	12.25
27.733	2707452	33.64	17876	20.79
32.092	1218162	15.13	7382	8.59
Totals	8049072	100.00	85983	100.00



Rac-22j

Area % Report

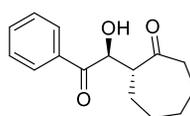
Method Name: C:\EZStart\Projects\Default\Methods\AS_8.5%_0.8mL.met
Data: C:\EZStart\Projects\Default\Data\V-GQS-123-AS-8.5%-0.8ML-CHIRAL
User: System
Acquired: 10/22/2010 12:38:54 PM
Printed: 10/22/2010 1:59:37 PM



SPD-10AVvp
Ch1-254nm

Results

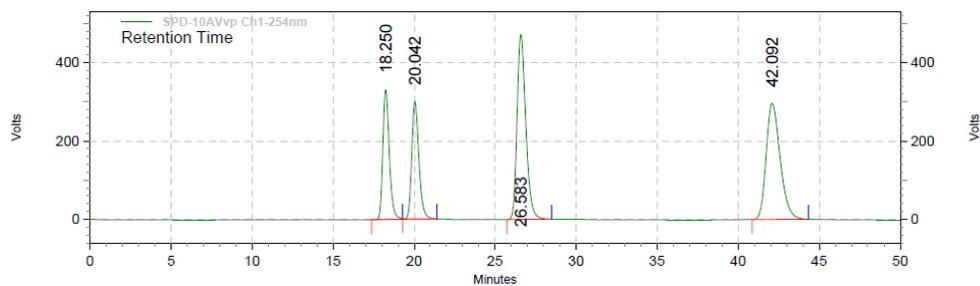
Retention Time	Area	Area %	Height	Height %
12.450	233946	2.92	4219	5.84
22.283	6821897	85.11	60710	84.01
27.925	585359	7.30	4605	6.37
32.392	374007	4.67	2732	3.78
Totals	8015209	100.00	72266	100.00



22j

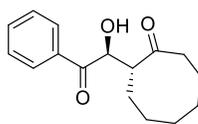
Area % Report

Method Name: C:\EZStart\Projects\Default\Methods\IC-10%-1ML_254NM.met
 Data: C:\Documents and
 Settings\Administrator\Desktop\SANDUN2\XI-GQS-180-IC-10%-1ML-RAC
 User: System
 Acquired: 9/18/2012 3:52:11 PM
 Printed: 9/18/2012 4:43:25 PM



SPD-10AVvp
 Ch1-254nm
 Results

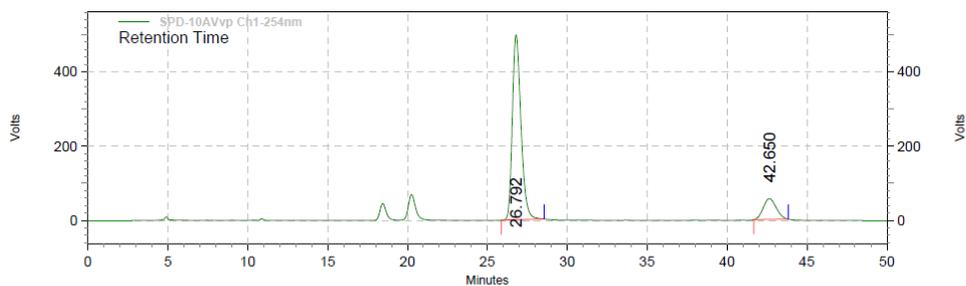
Retention Time	Area	Area %	Height	Height %
18.250	9166409	16.99	331236	23.68
20.042	9235169	17.11	300206	21.46
26.583	17764460	32.92	471218	33.68
42.092	17798419	32.98	296356	21.18
Totals	53964457	100.00	1399016	100.00



Rac-22k

Area % Report

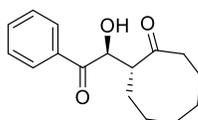
Method Name: C:\EZStart\Projects\Default\Methods\IC-10%_1ML_254NM.met
 Data: C:\Documents and Settings\Administrator\Desktop\SANDUN2\XI-GQS-180-IC-10%-1ML-CHIRA
 User: System
 Acquired: 9/18/2012 2:59:02 PM
 Printed: 9/18/2012 3:50:28 PM



SPD-10AVvp
 Ch1-254nm

Results

Retention Time	Area	Area %	Height	Height %
26.792	18693172	85.56	498123	89.87
42.650	3155298	14.44	56138	10.13
Totals	21848470	100.00	554261	100.00



22k