

Supporting Information

Catalytic Asymmetric Tamura Cycloaddition of Homophthalic Anhydrides with 2-Arylidene-1,3-diones

Han Xu, Feng Sha, Qiong Li, Xin-Yan Wu*

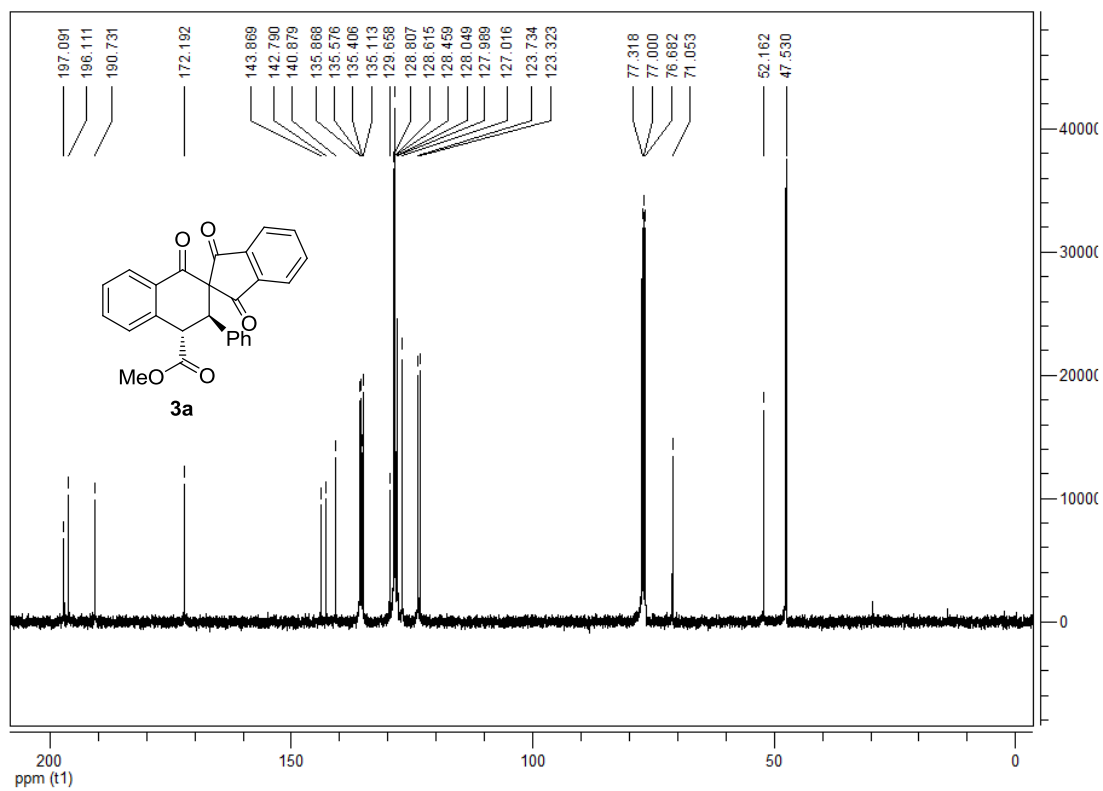
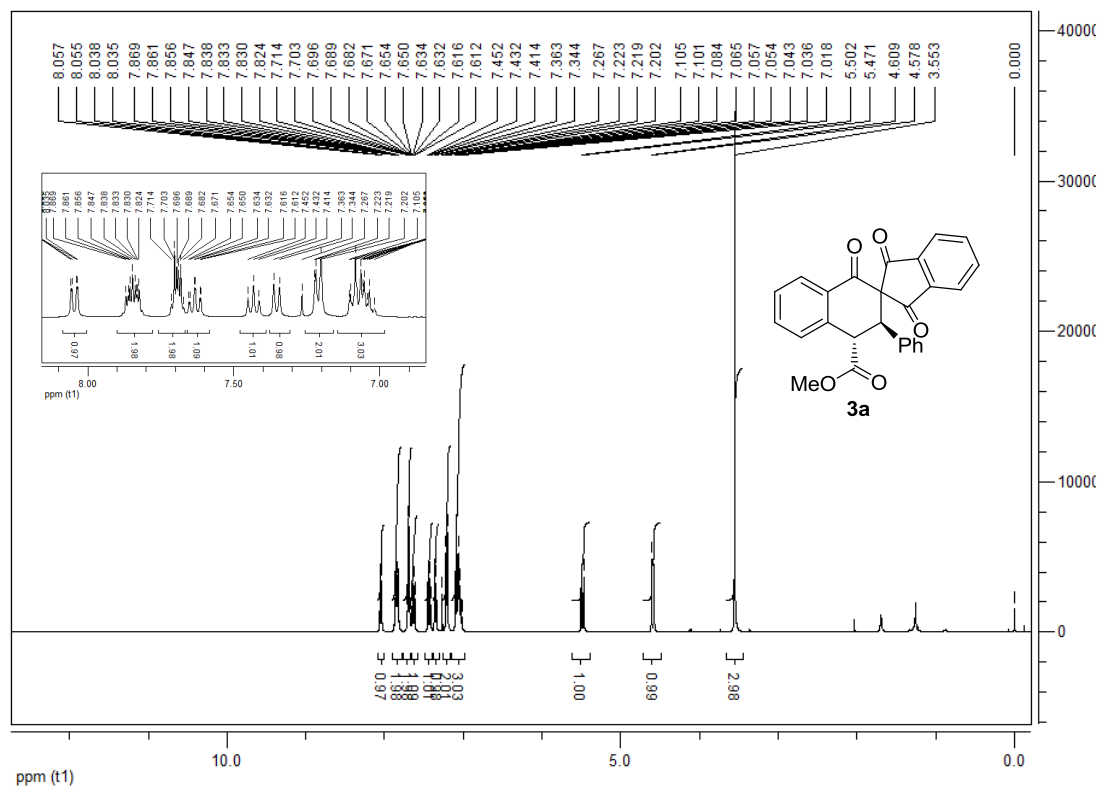
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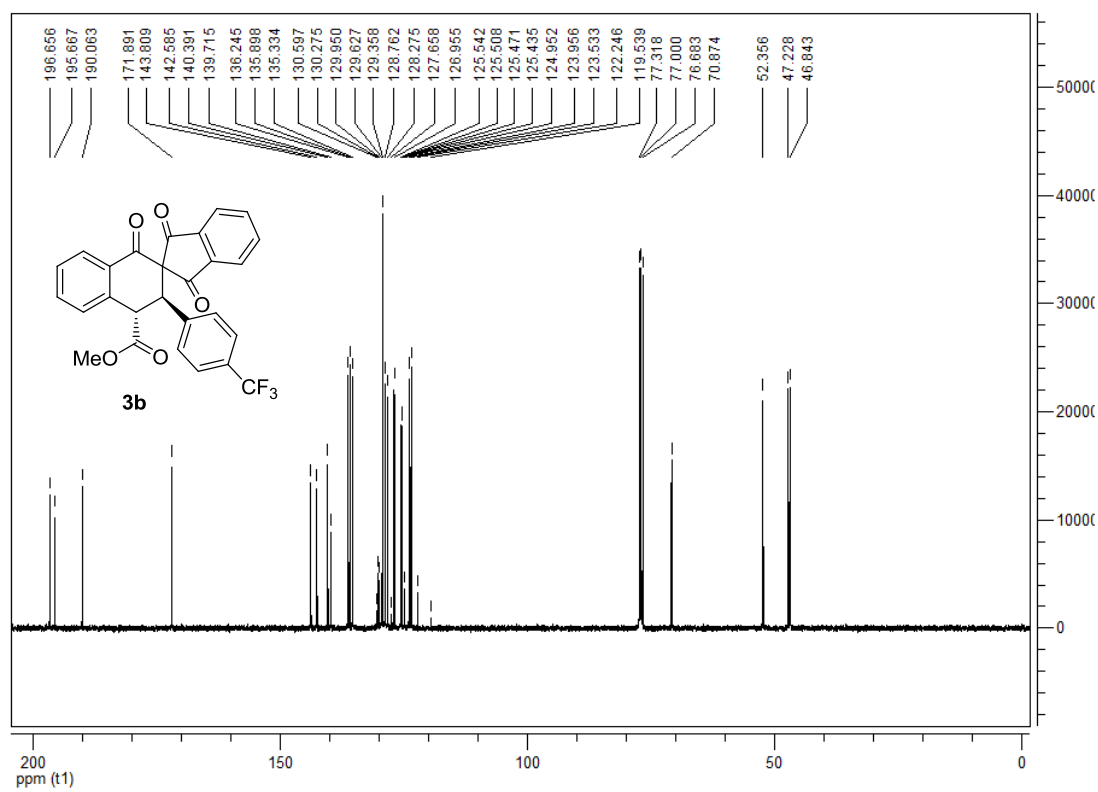
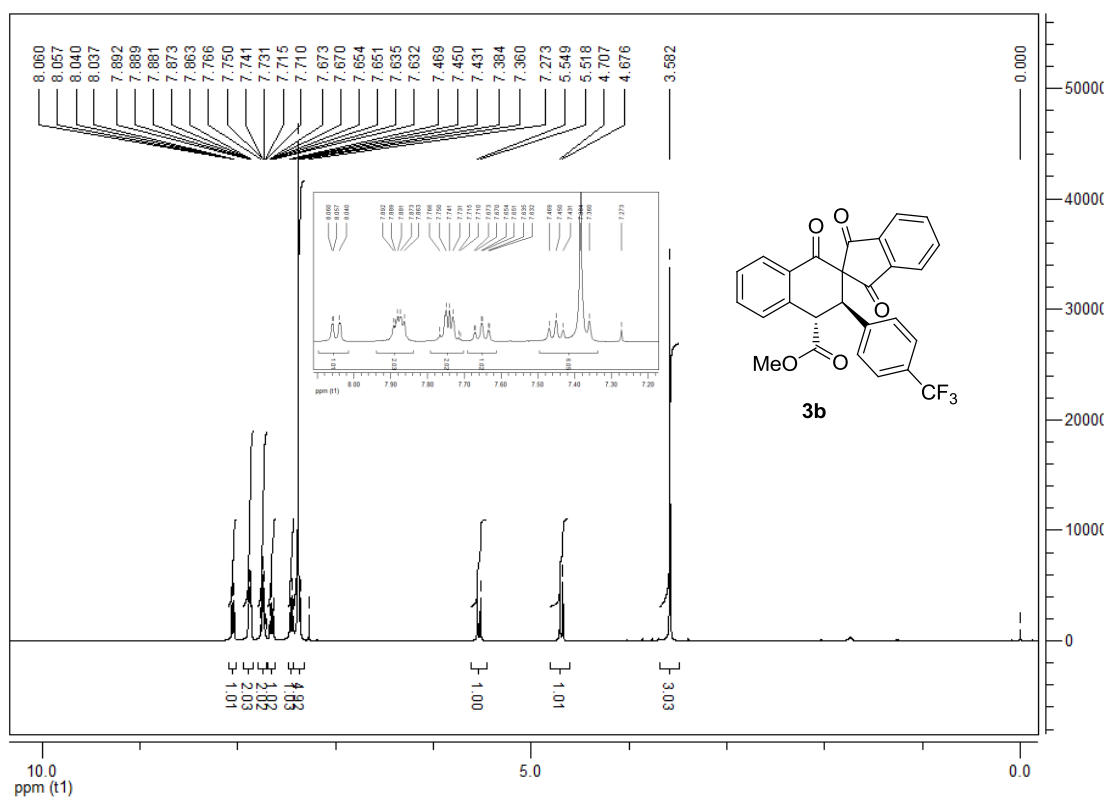
xinyanwu@ecust.edu.cn

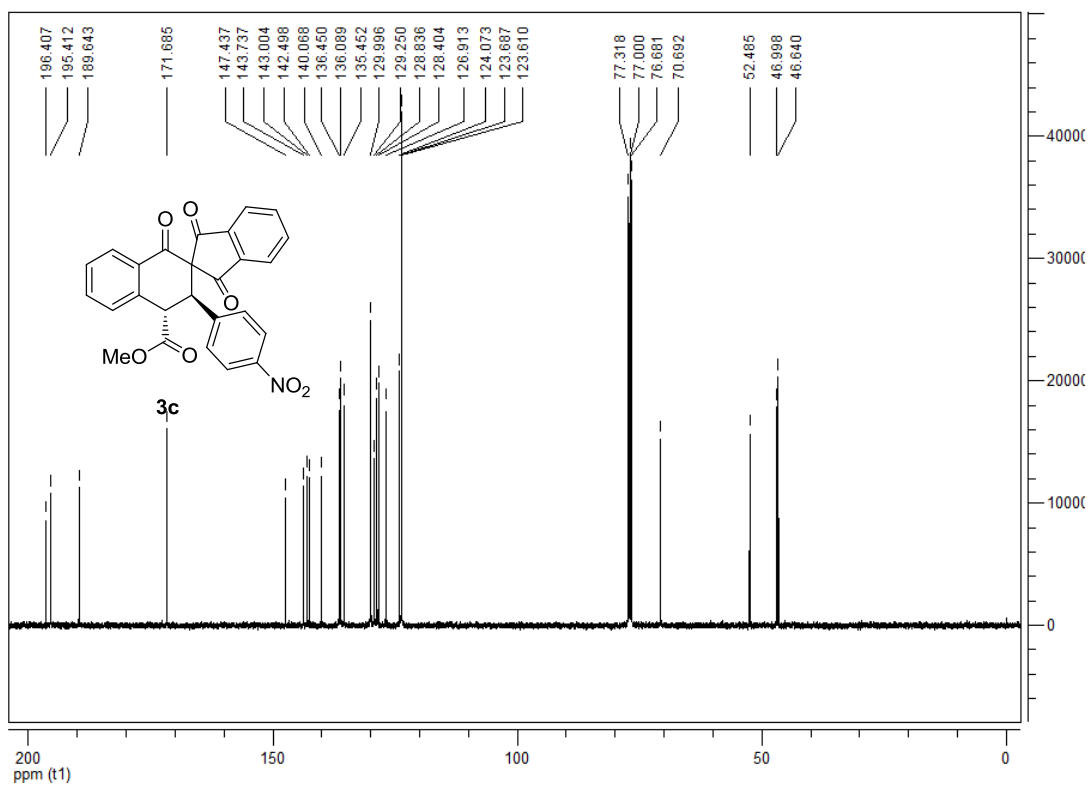
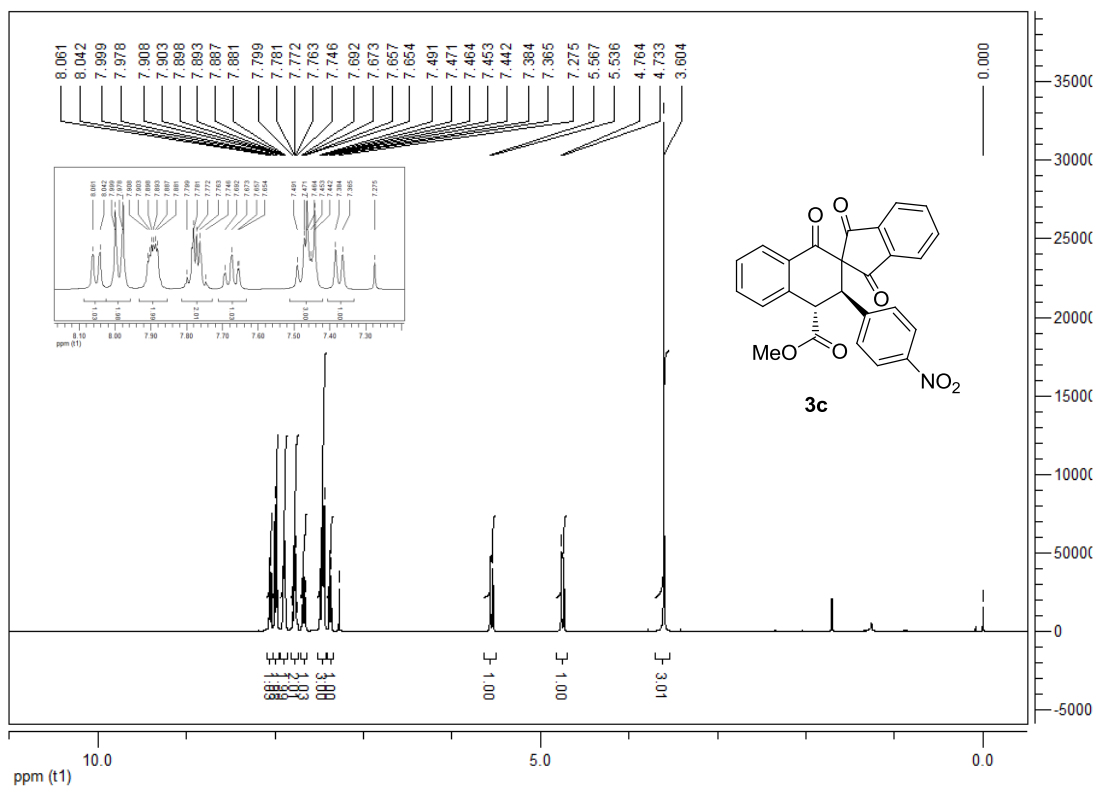
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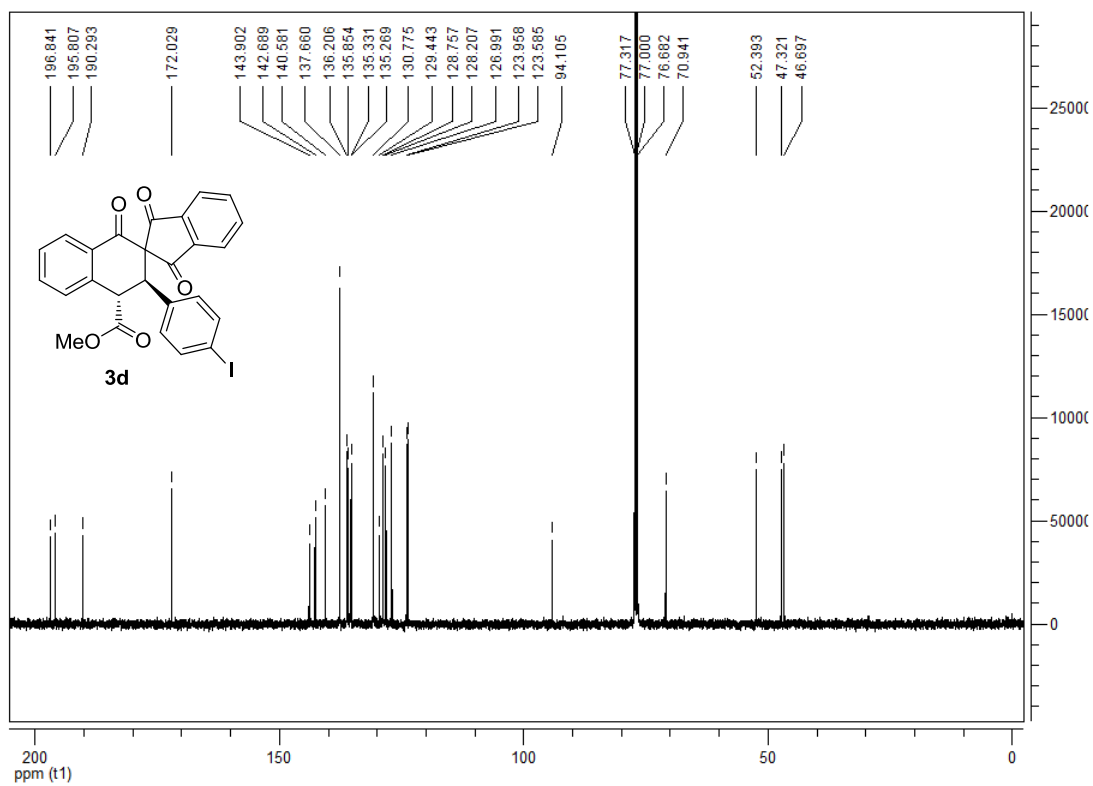
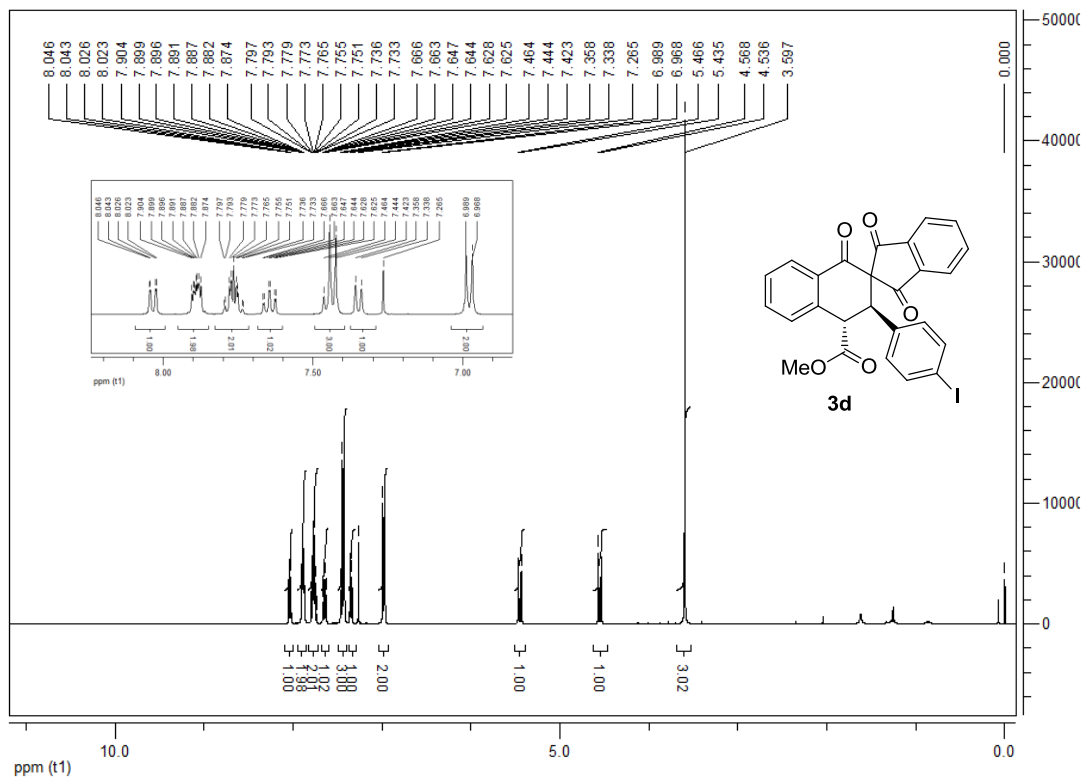
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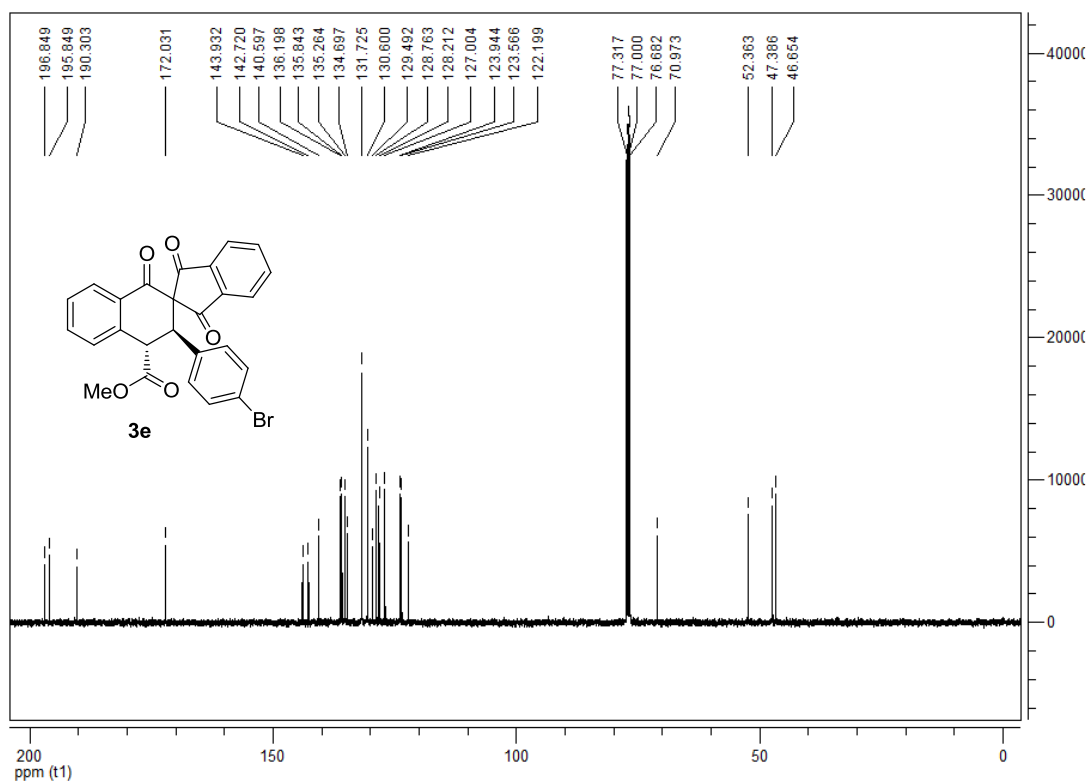
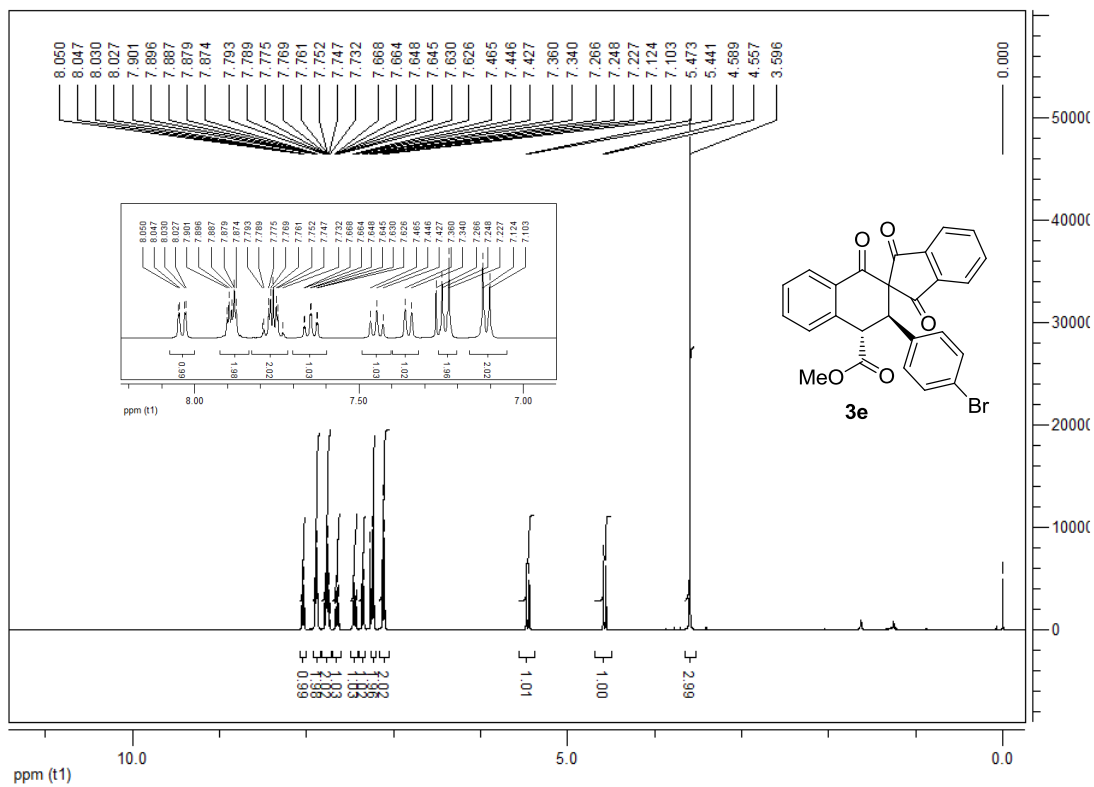
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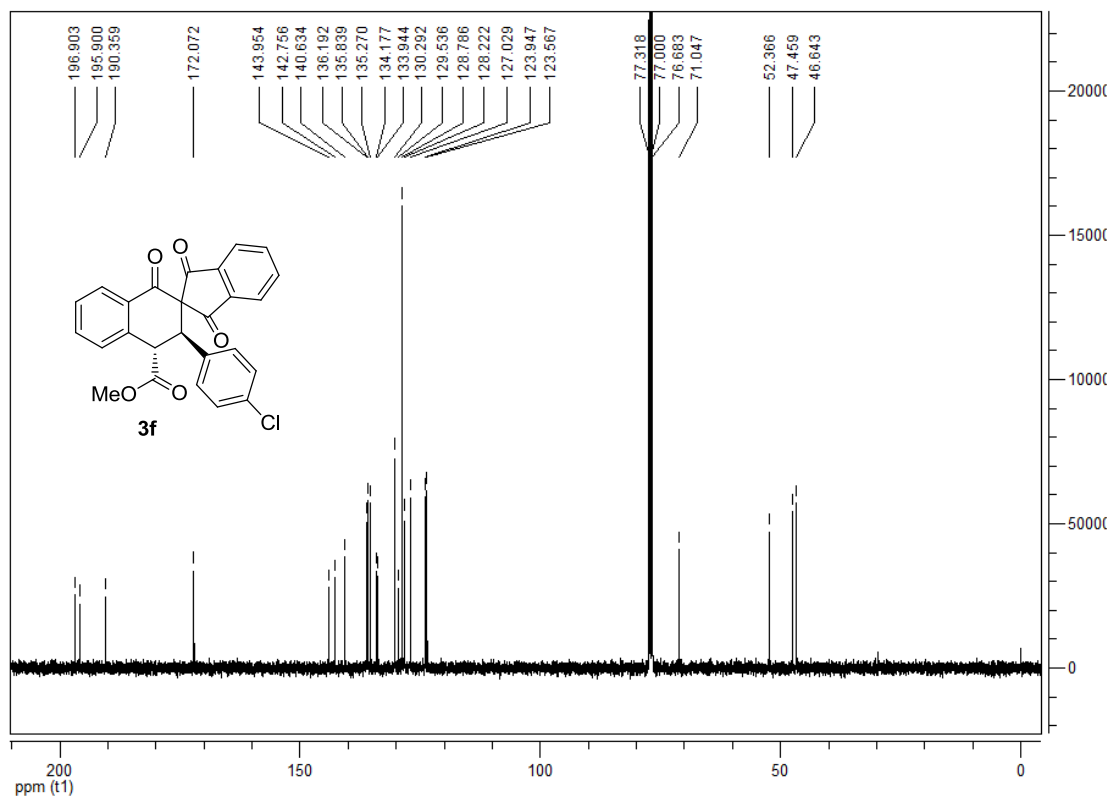
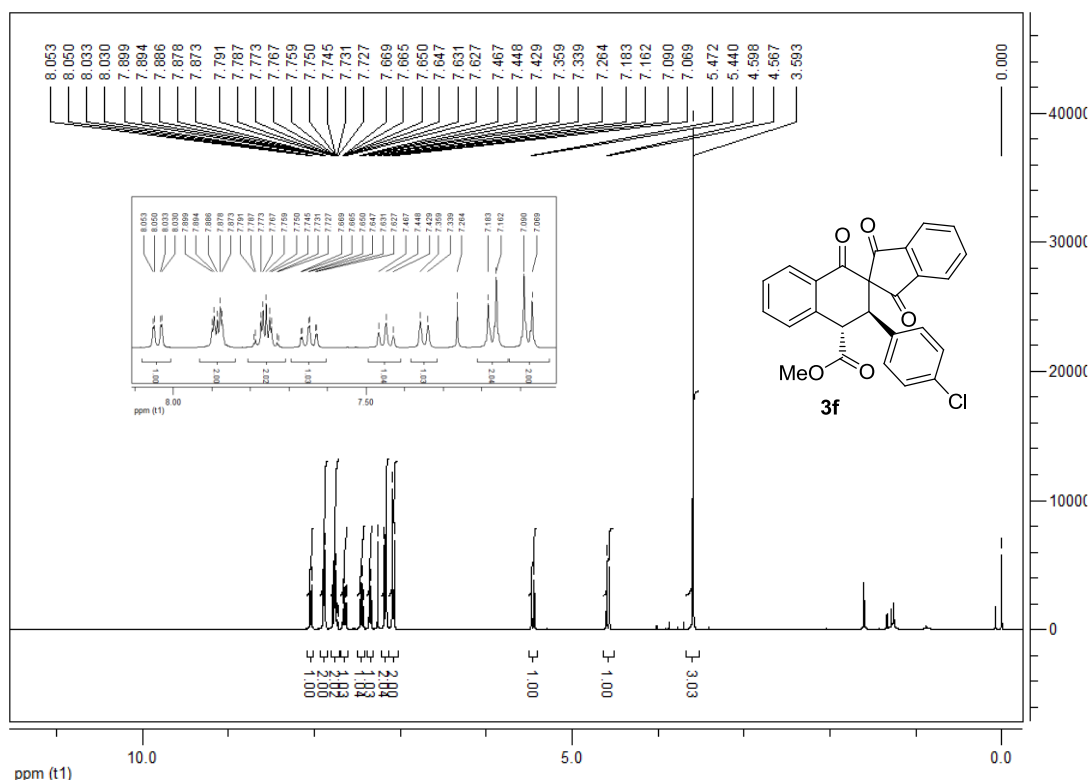


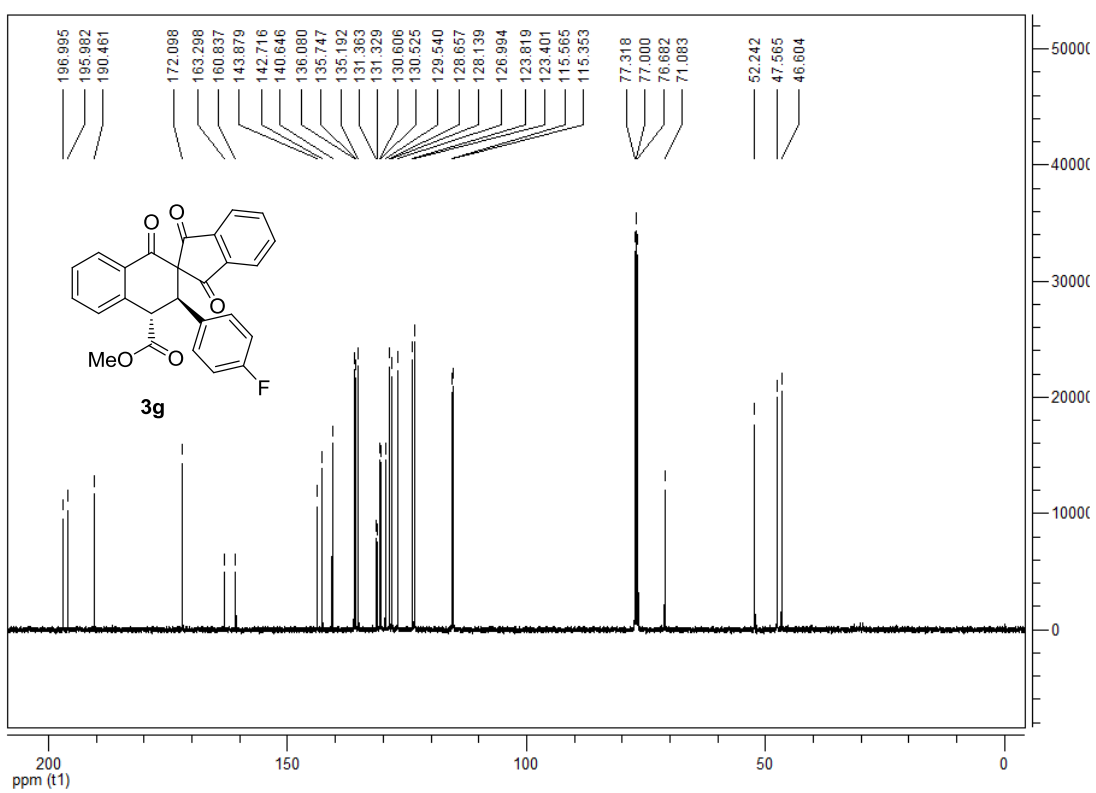
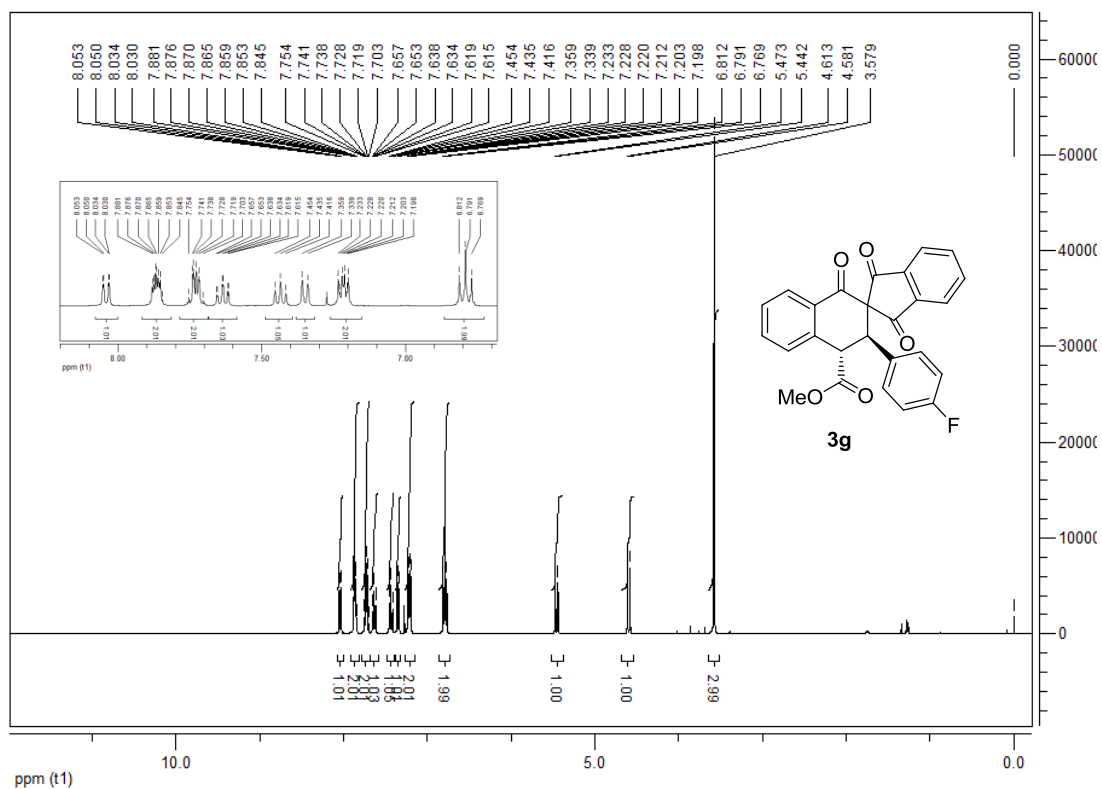


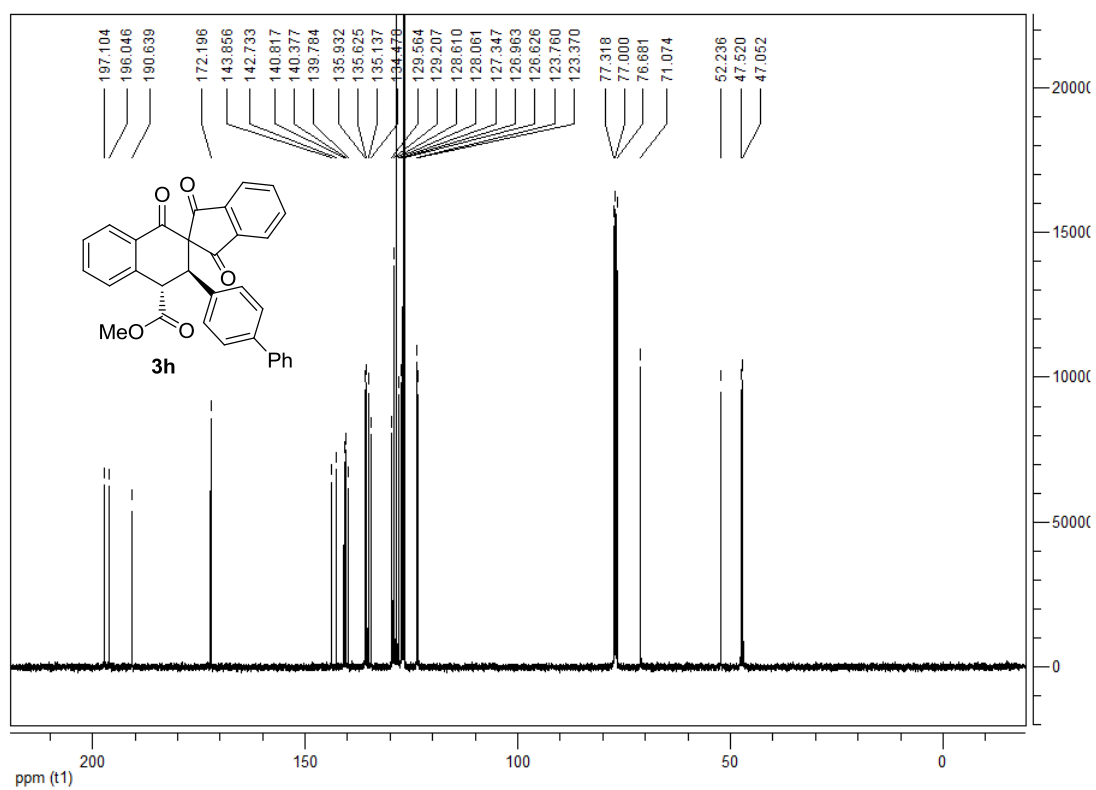
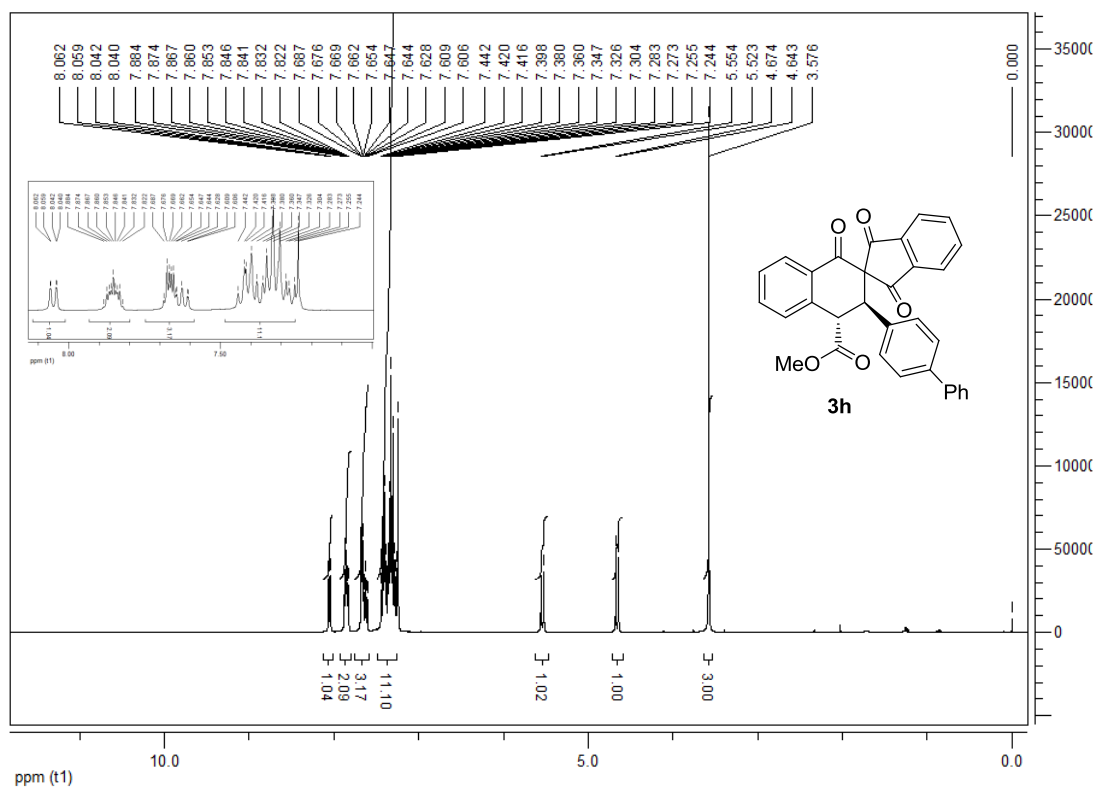


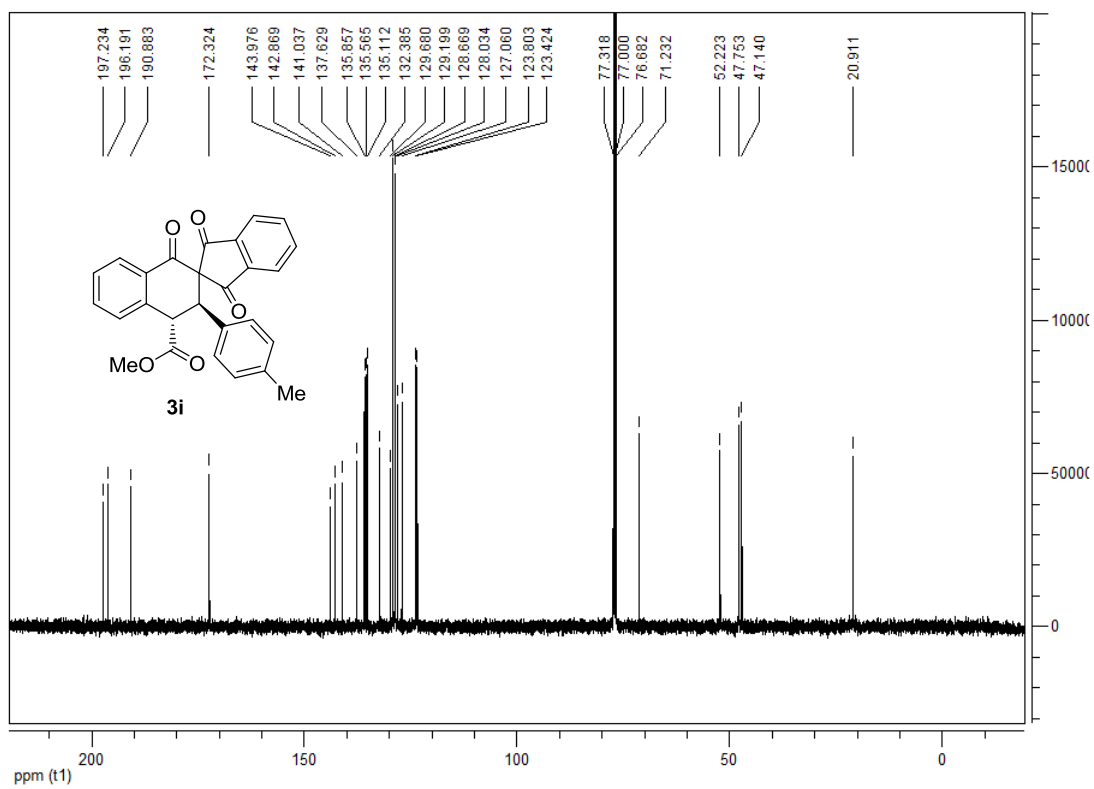
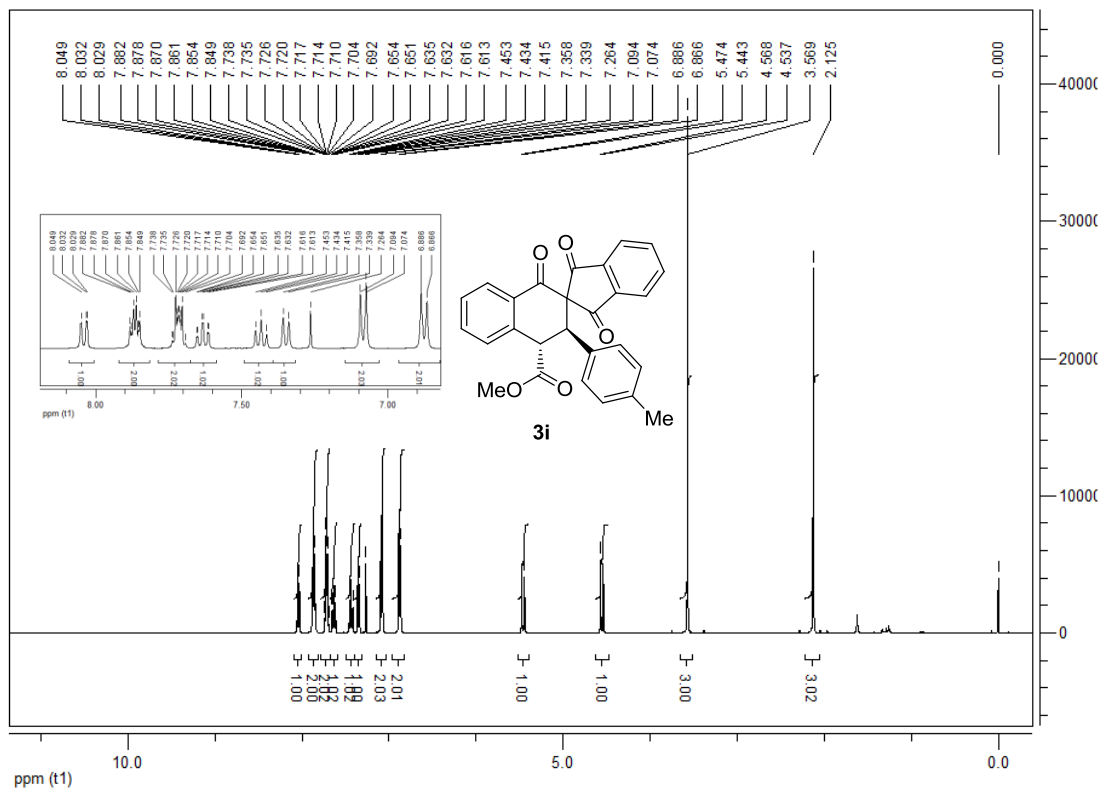


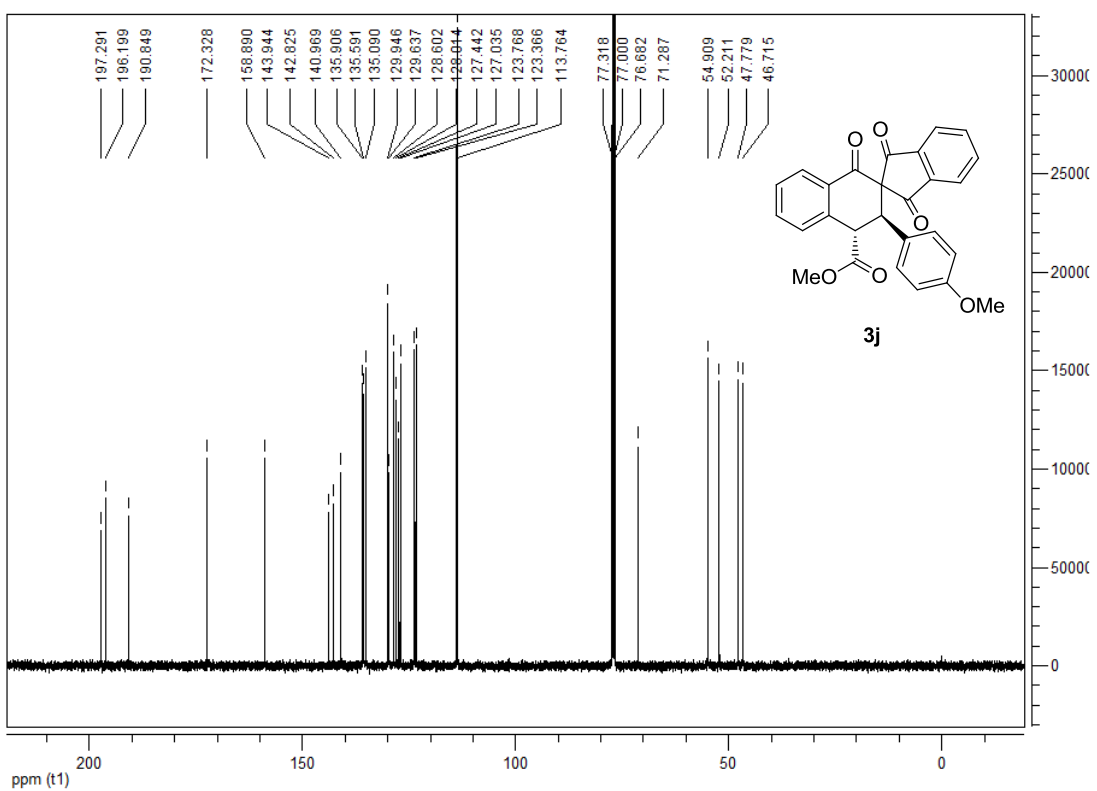
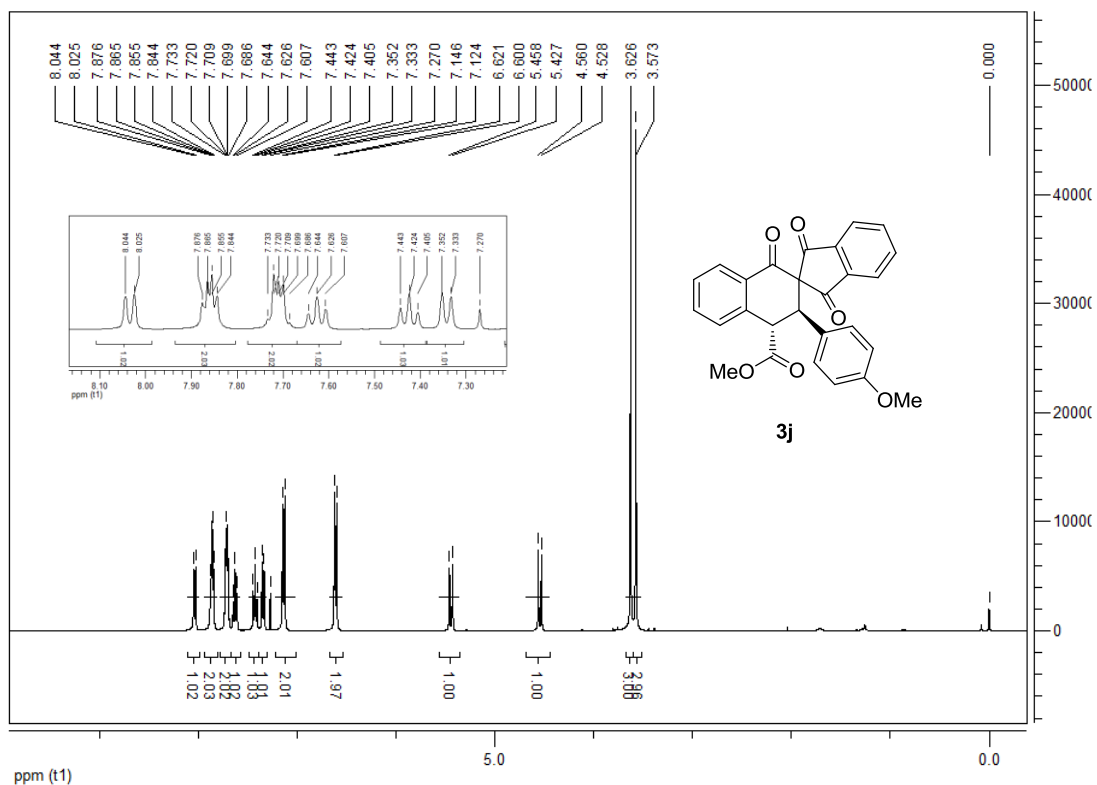


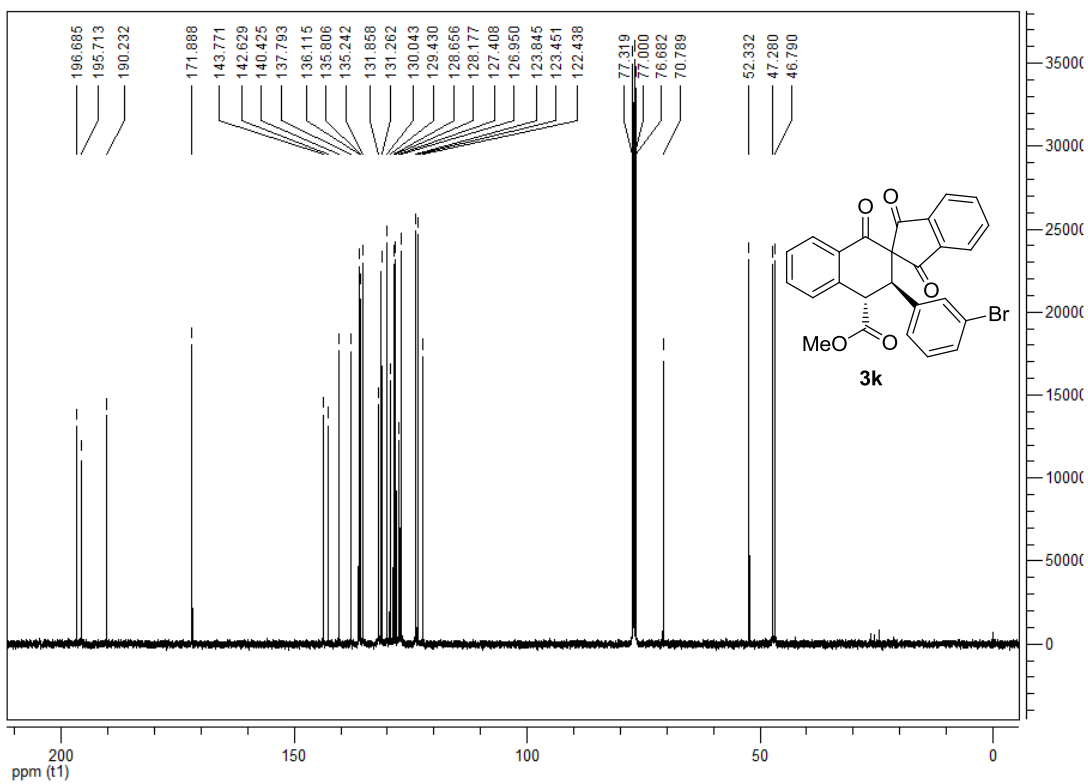
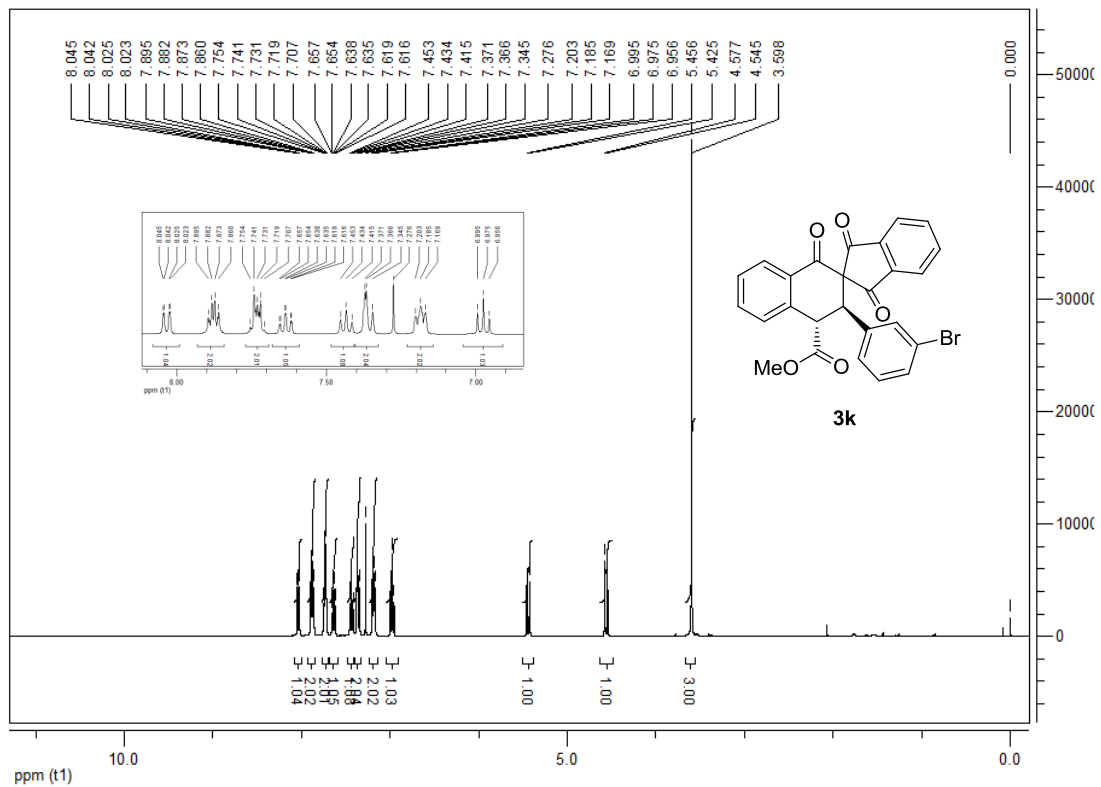


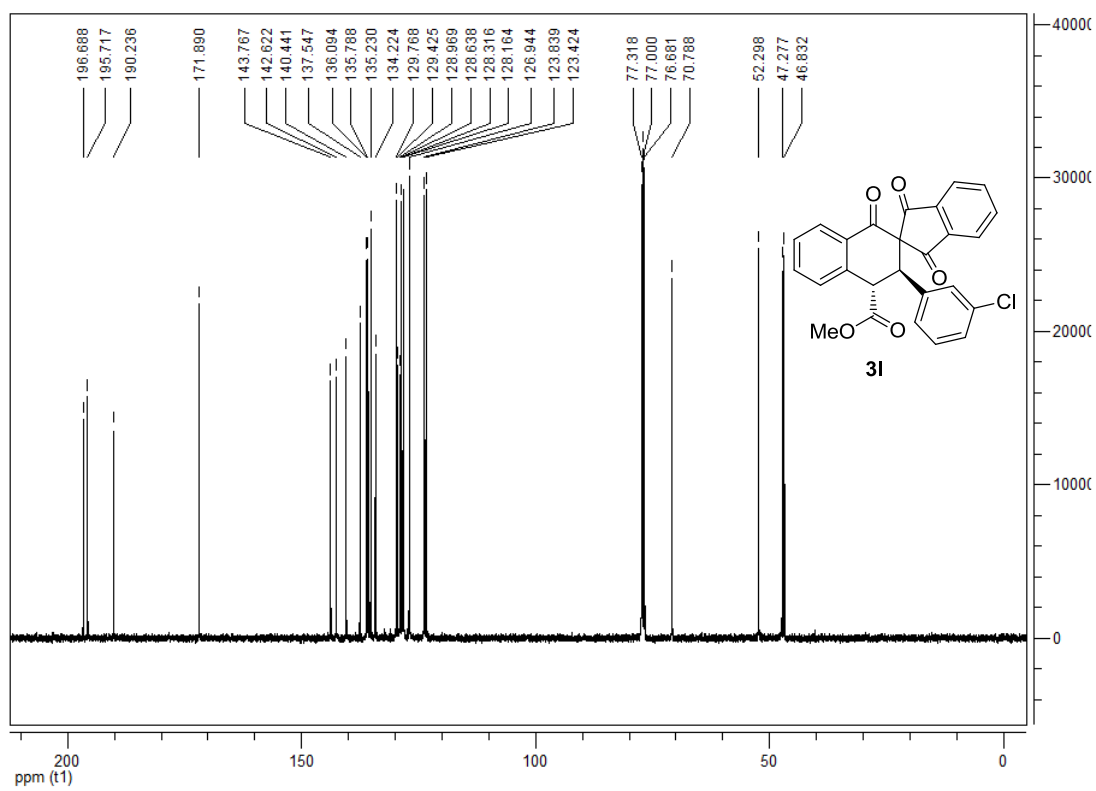
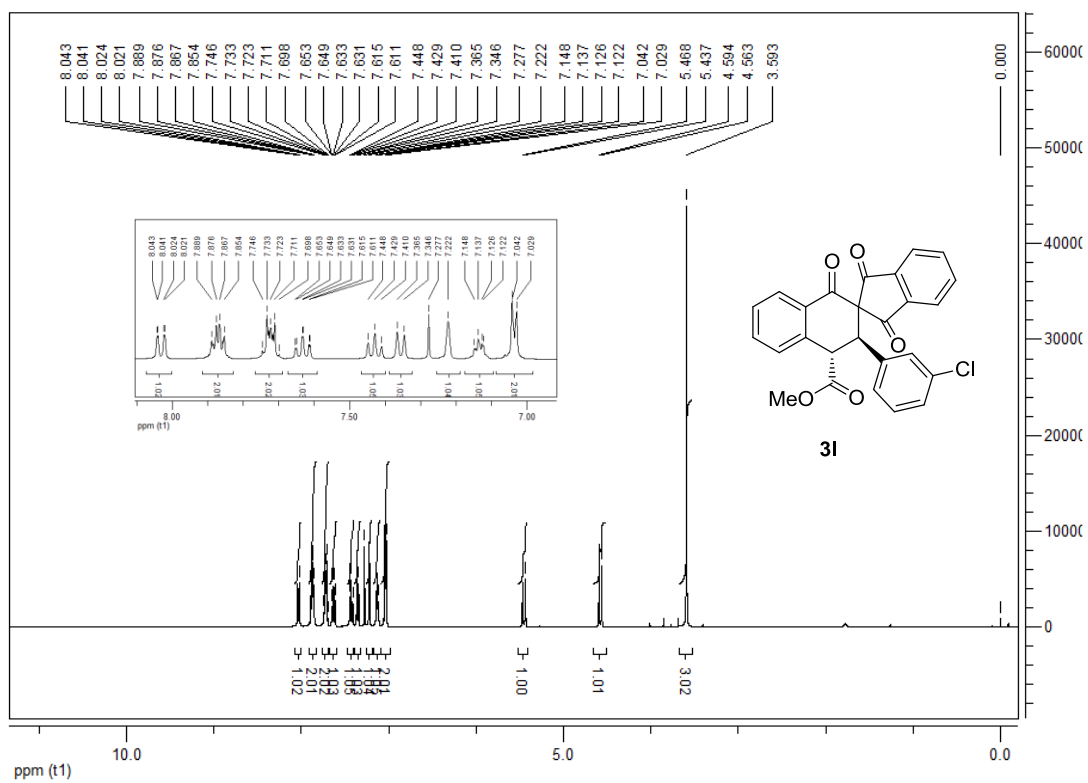


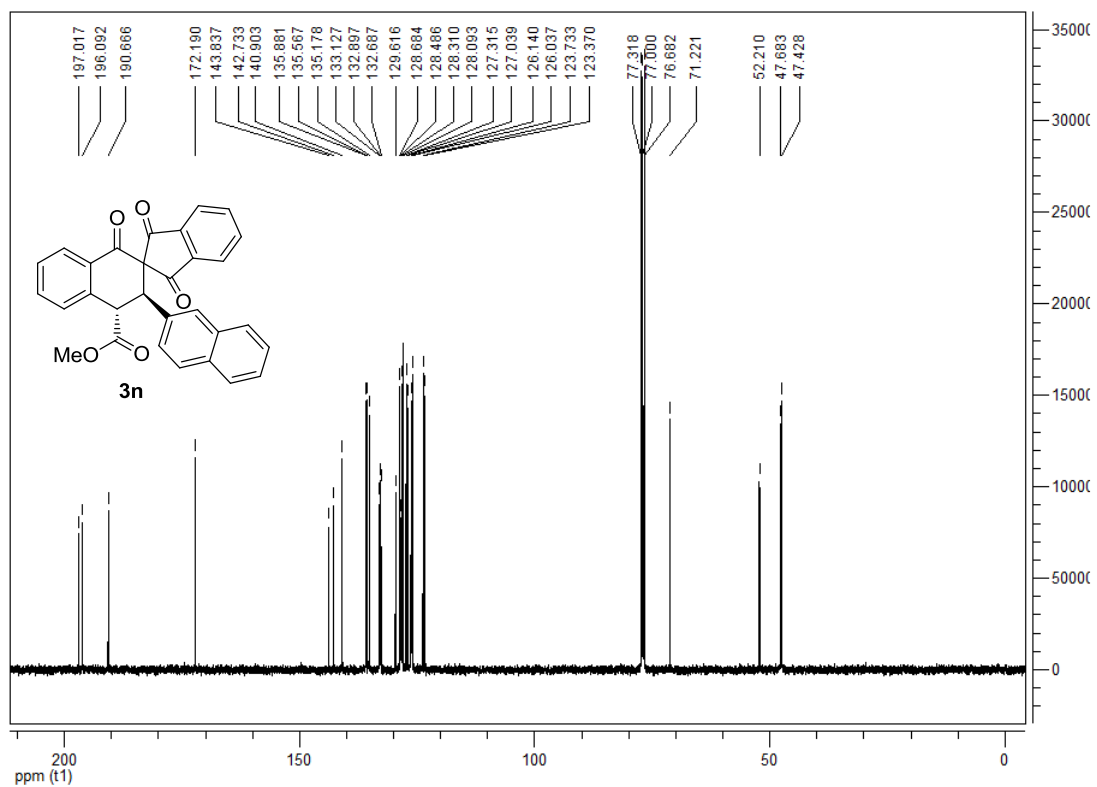
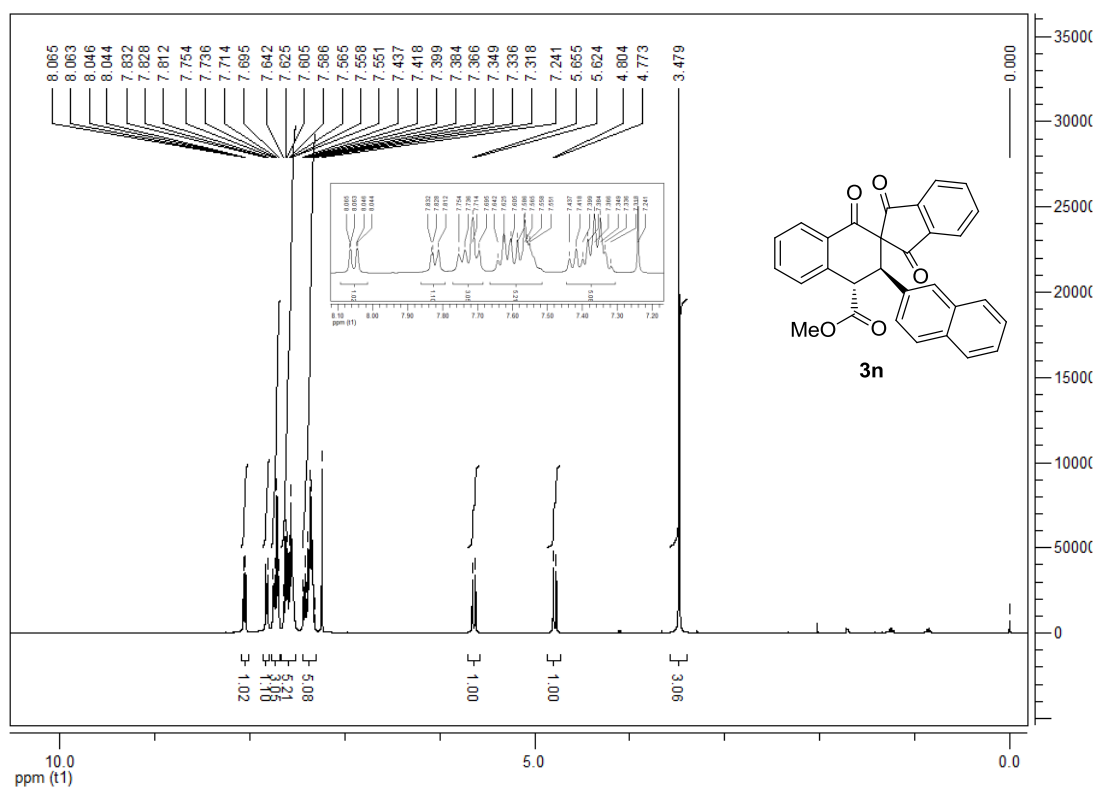


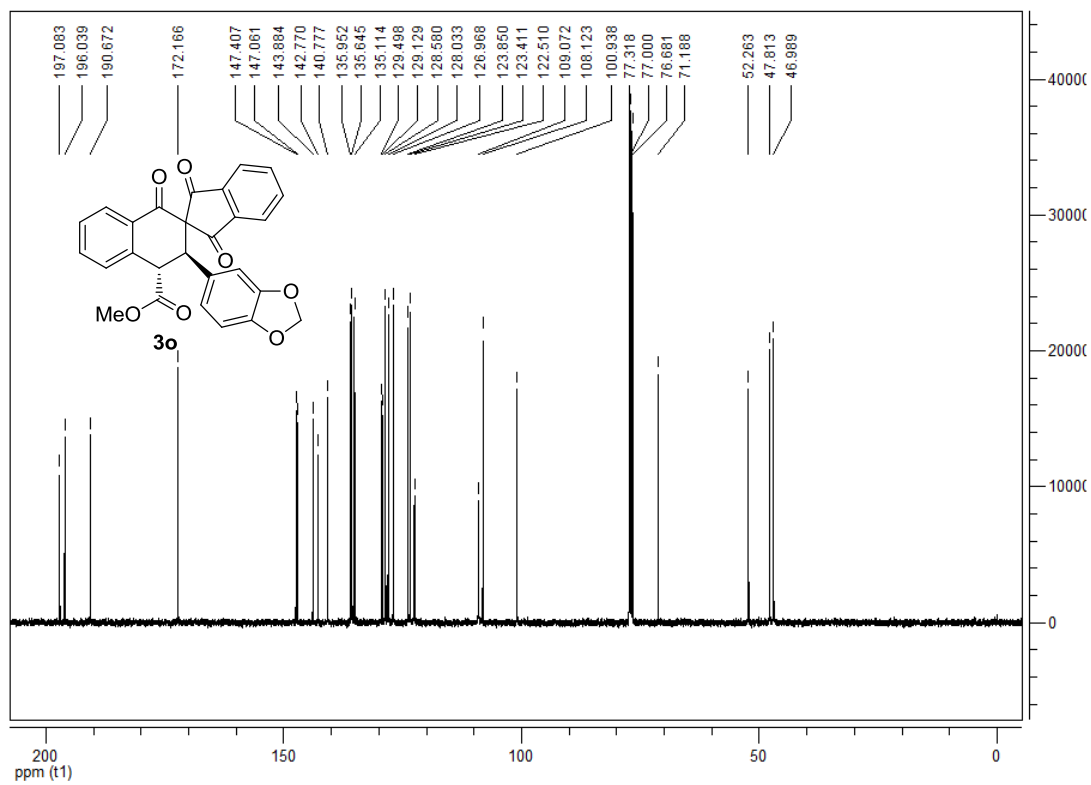
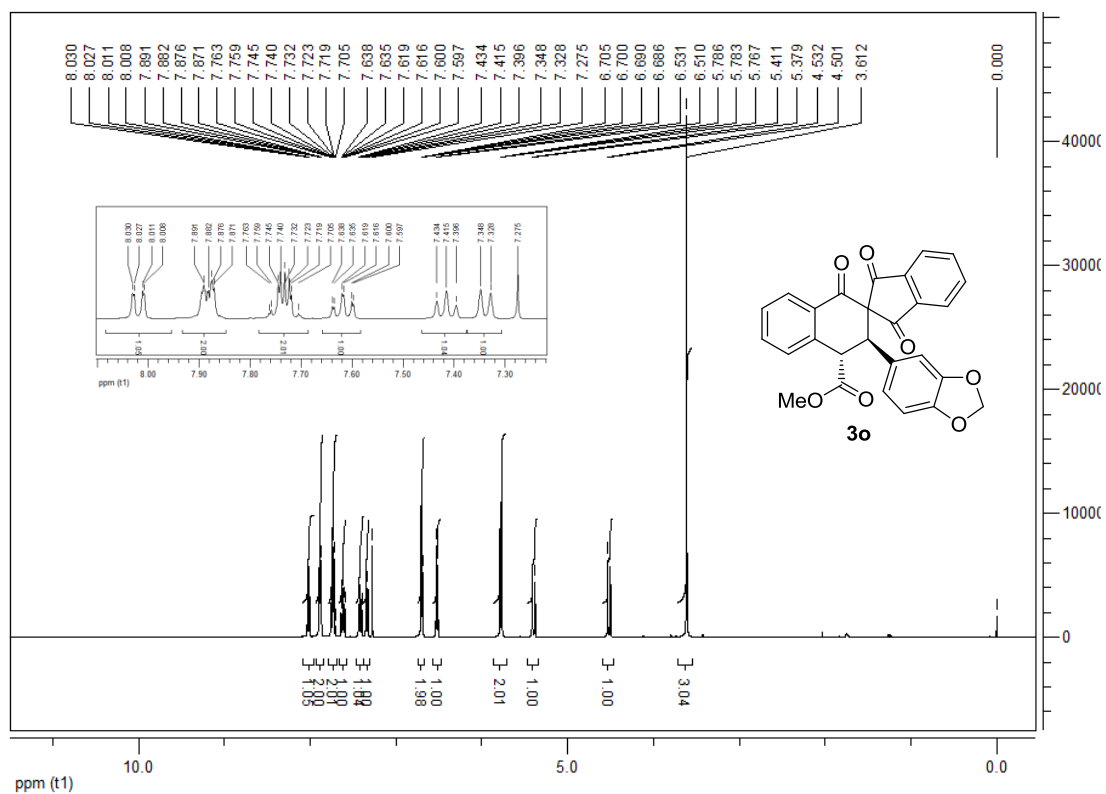


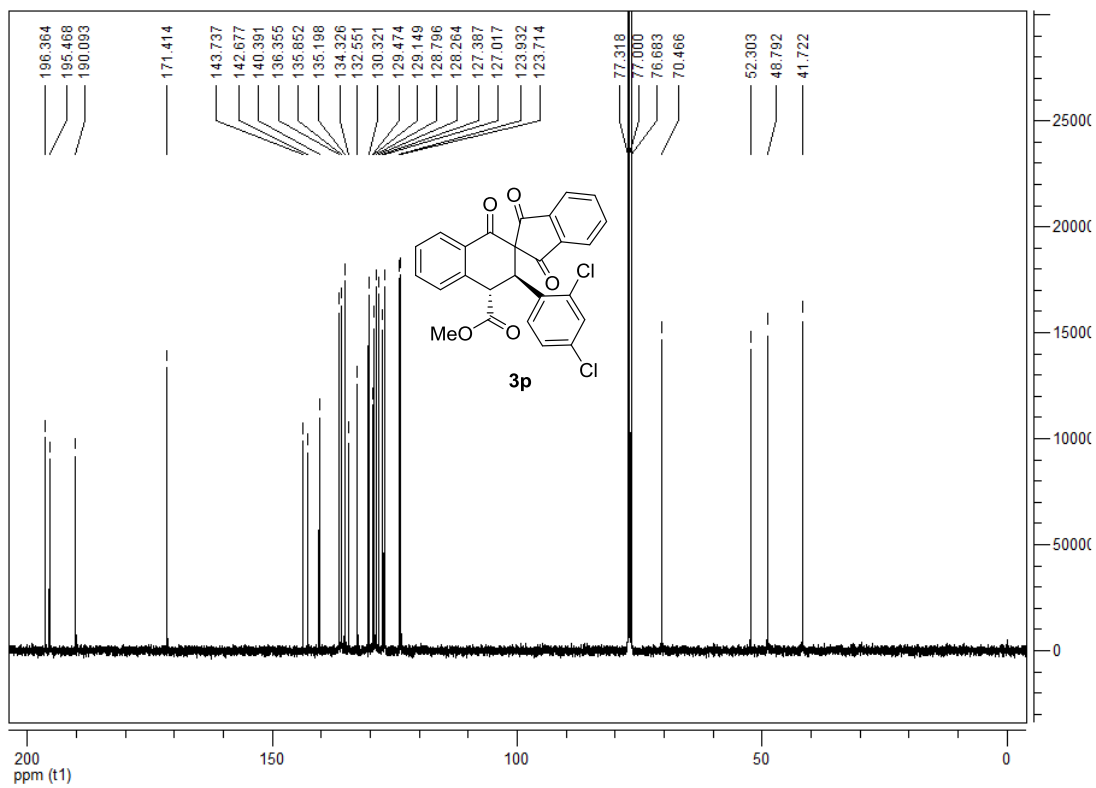
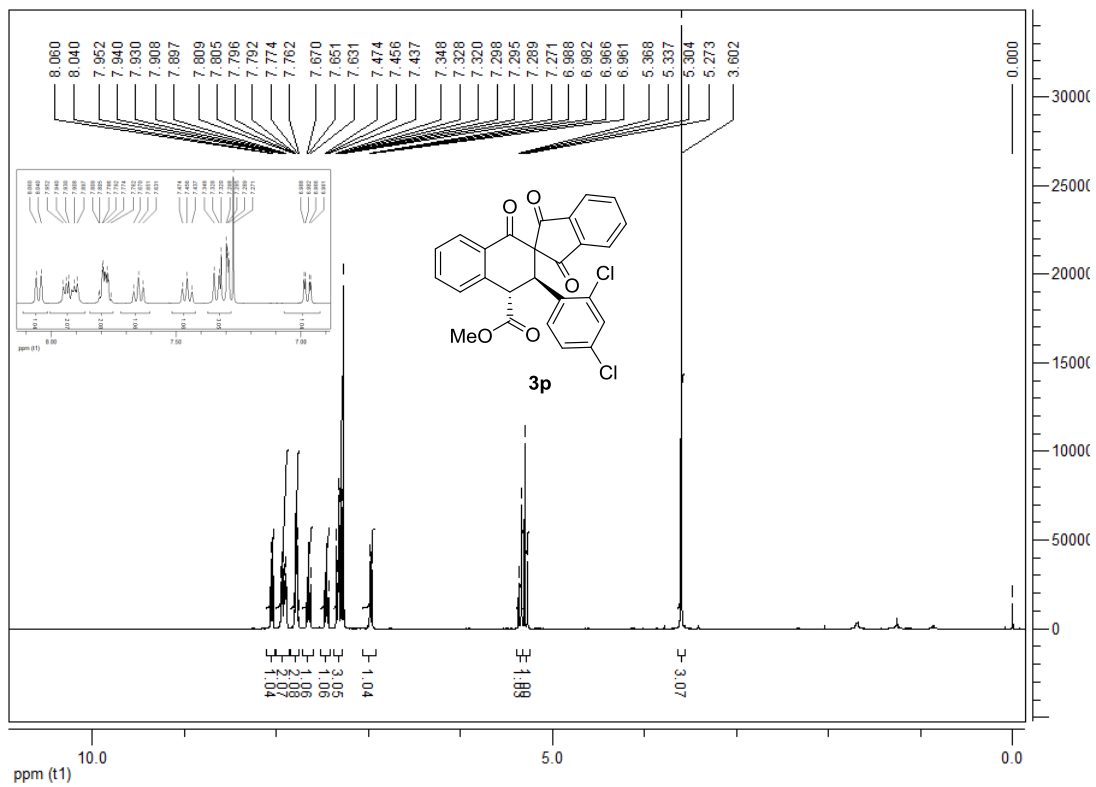


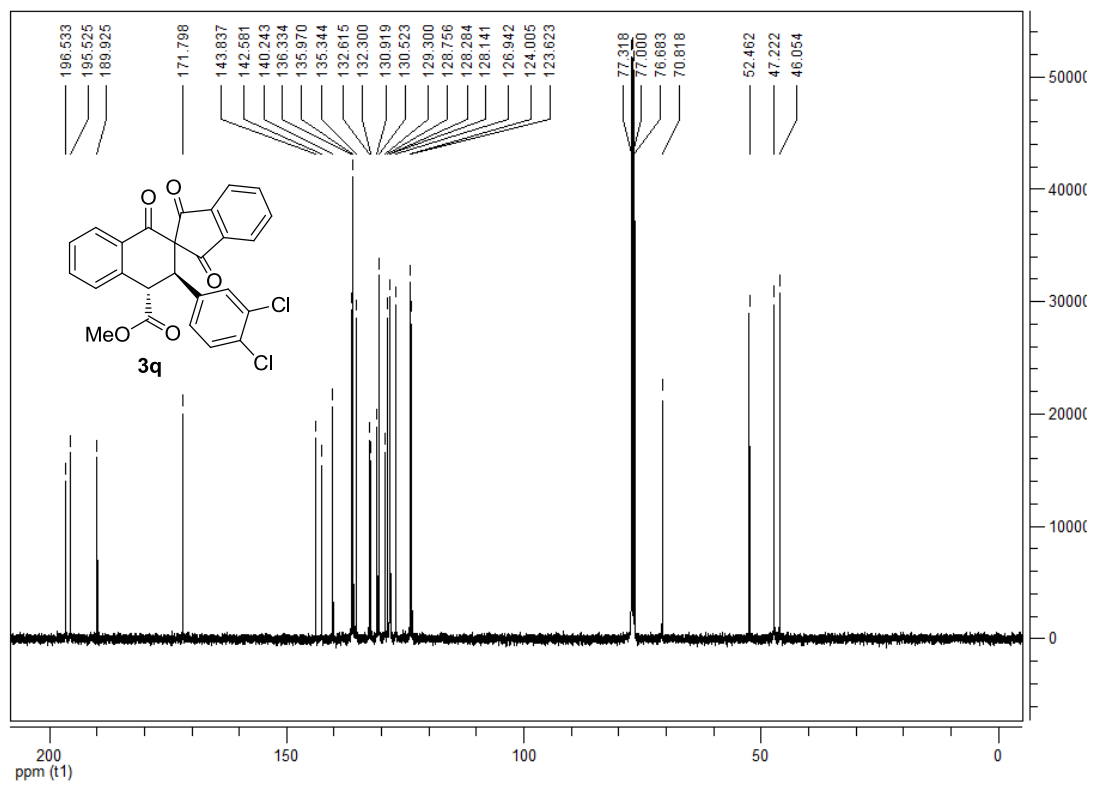
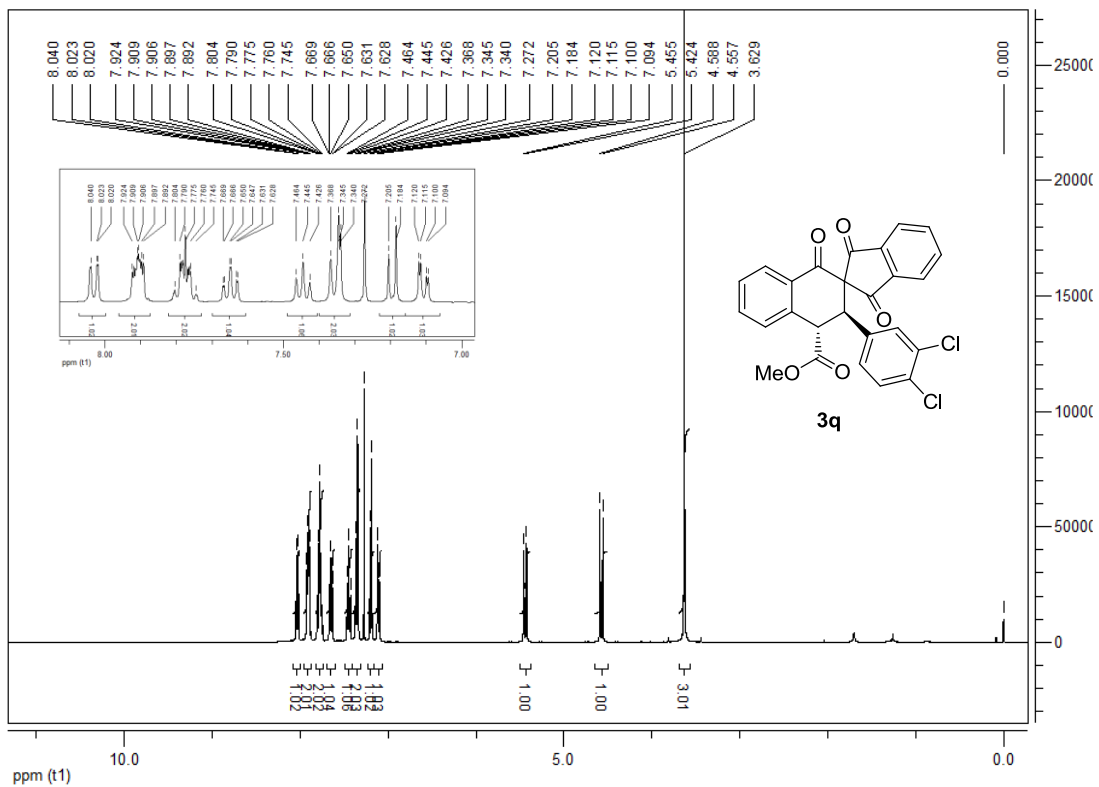


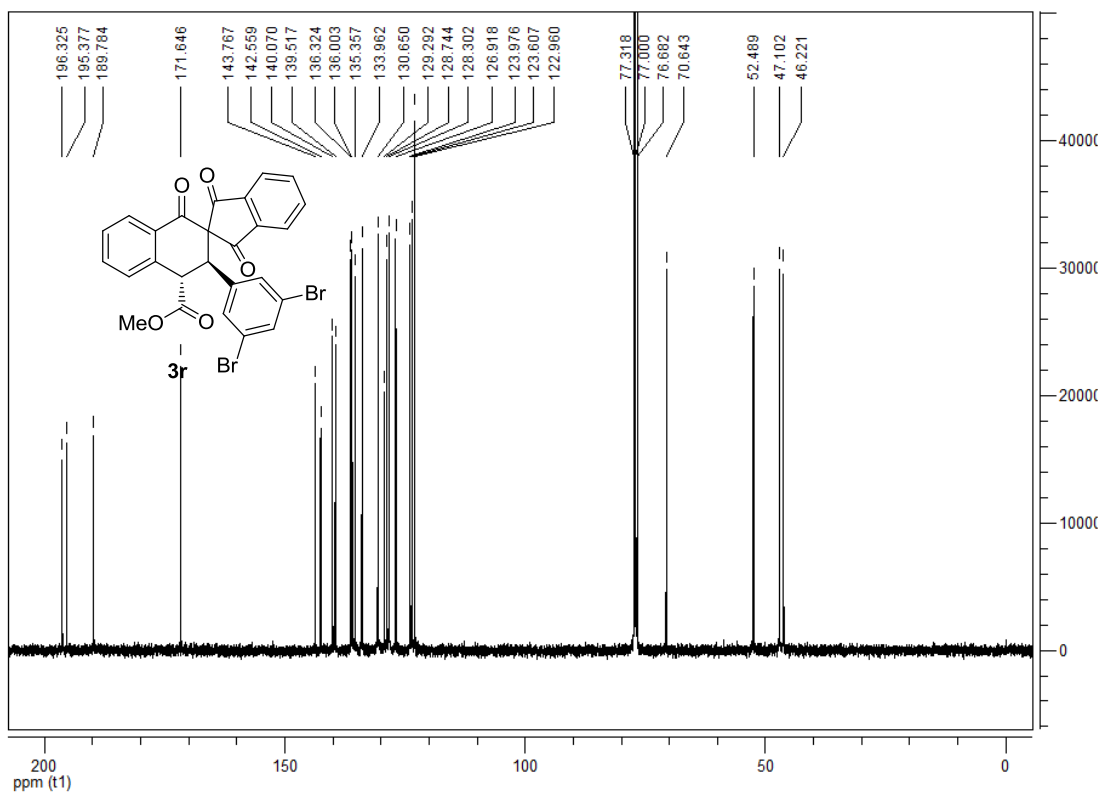
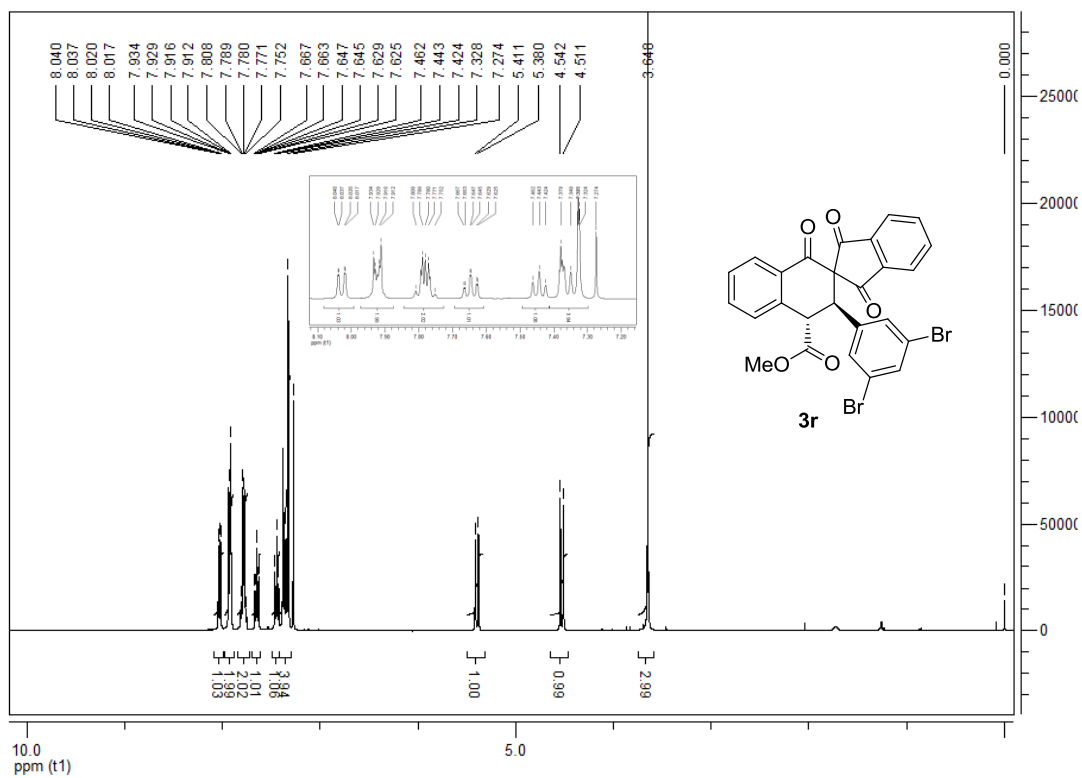


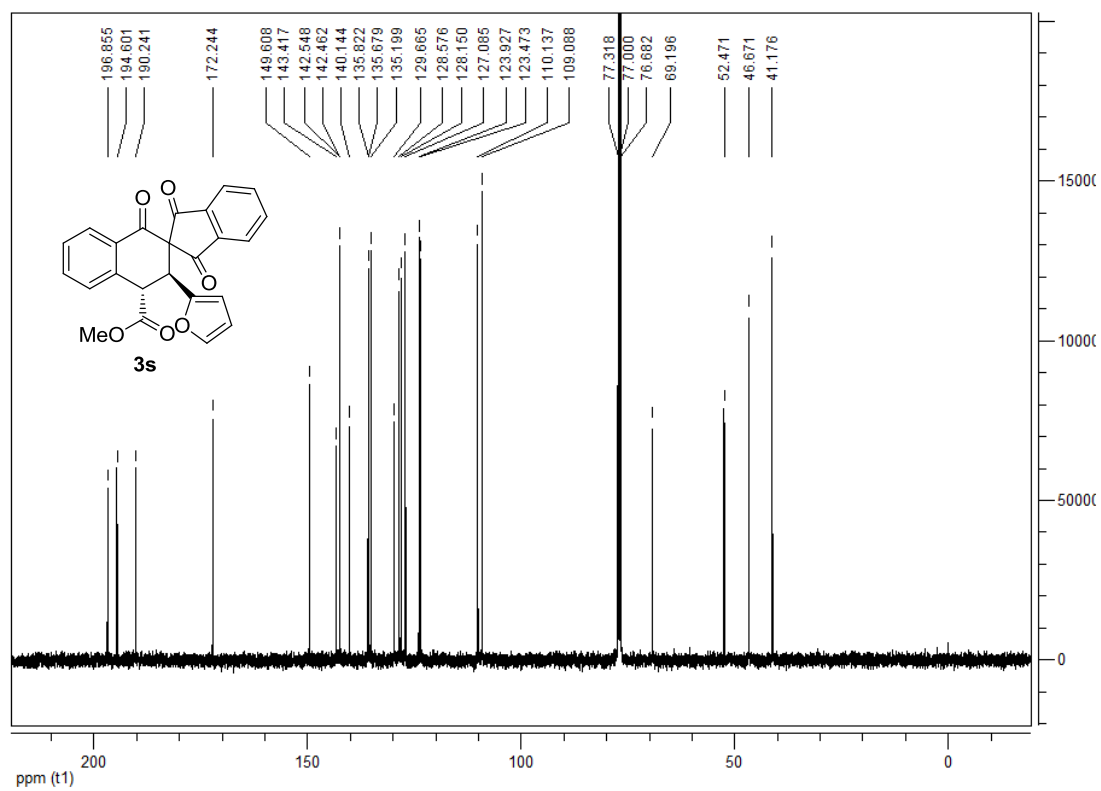
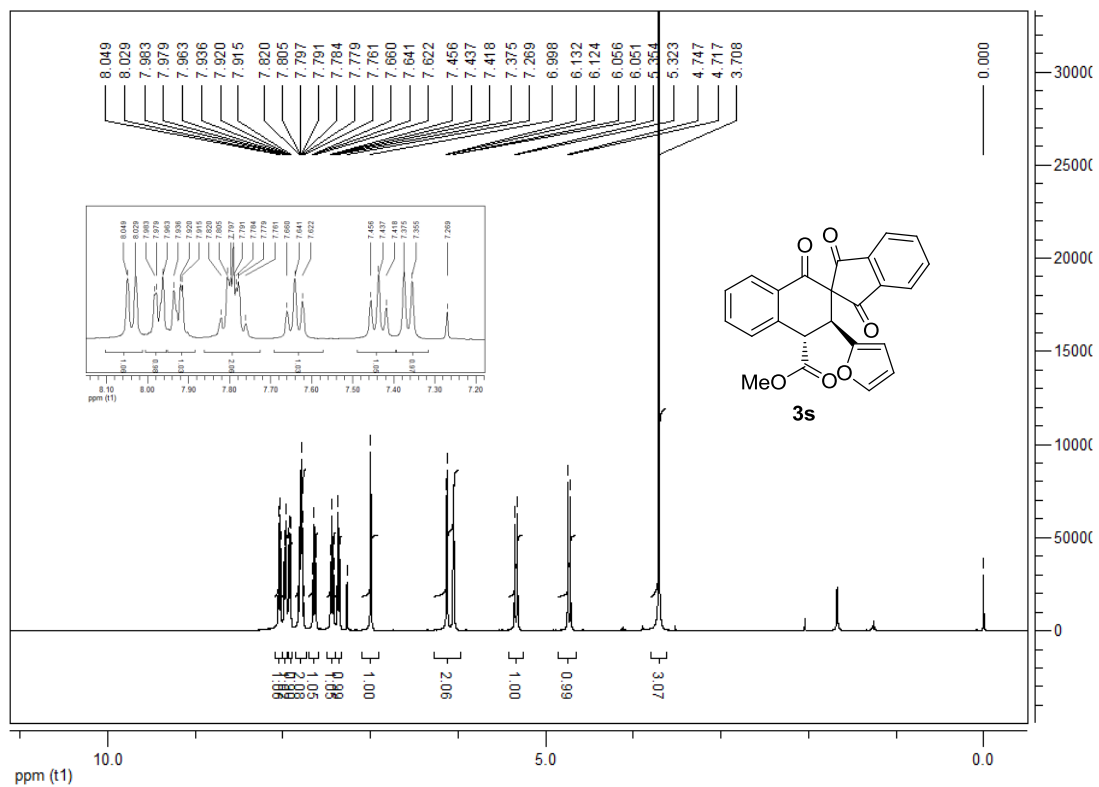


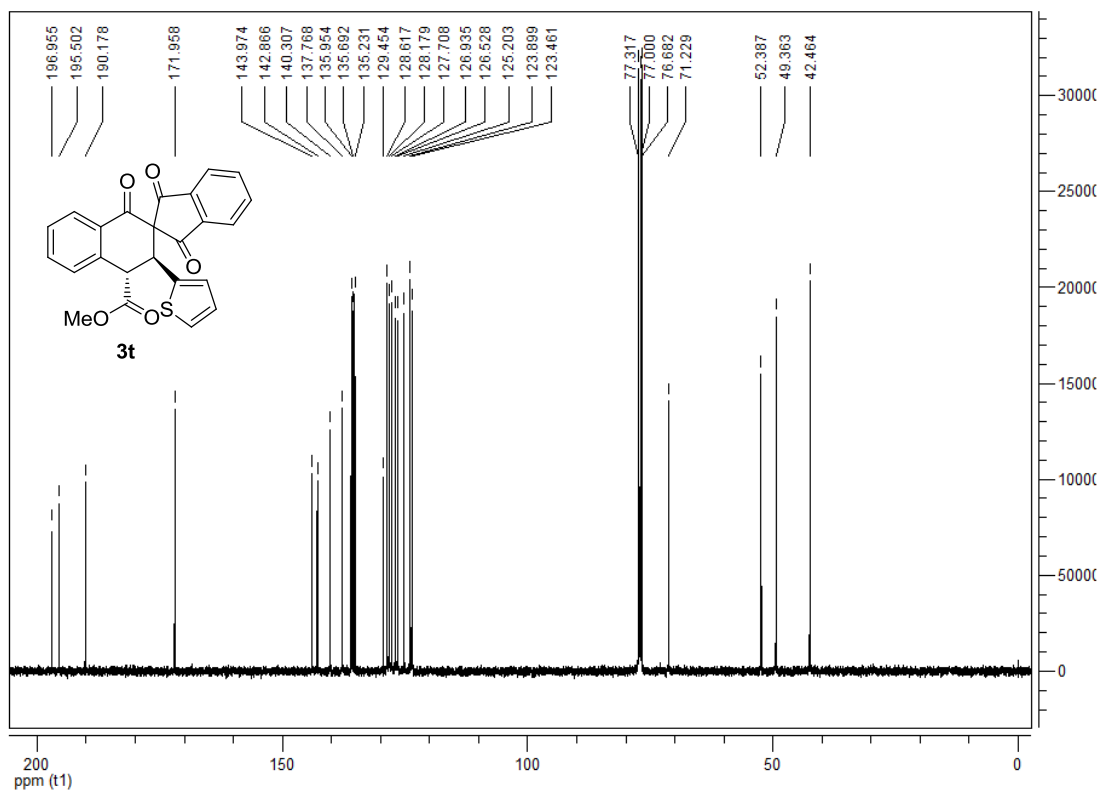
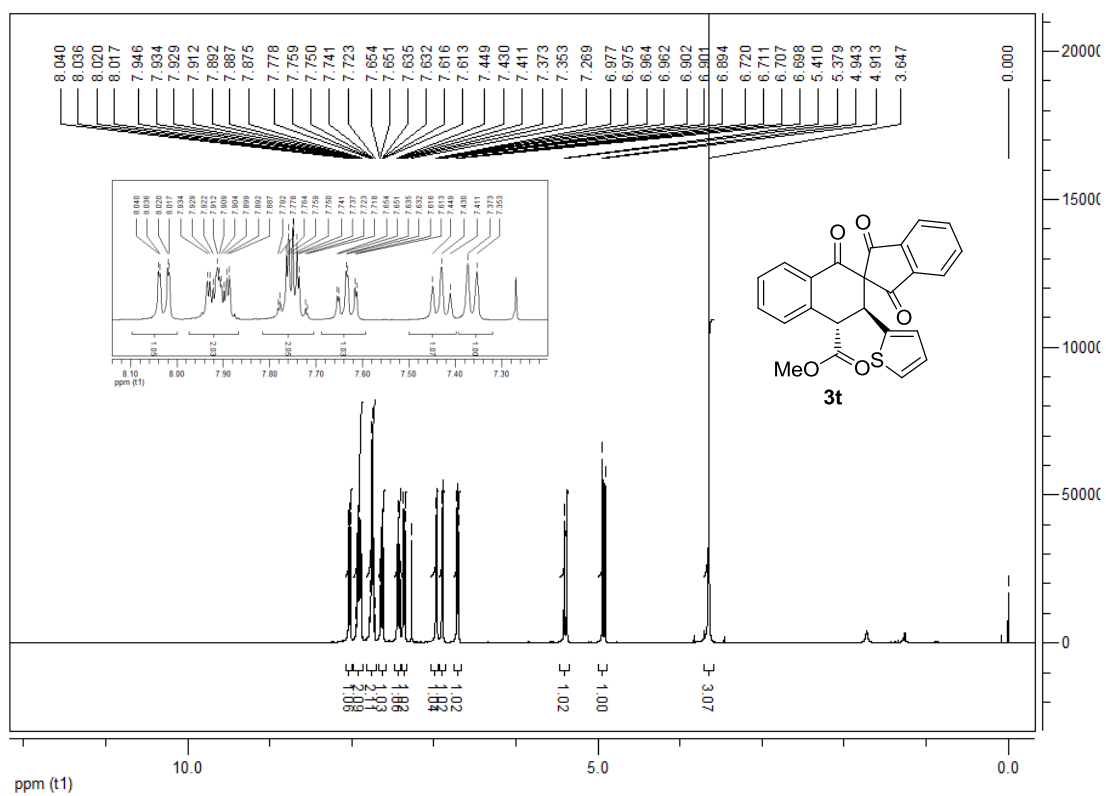


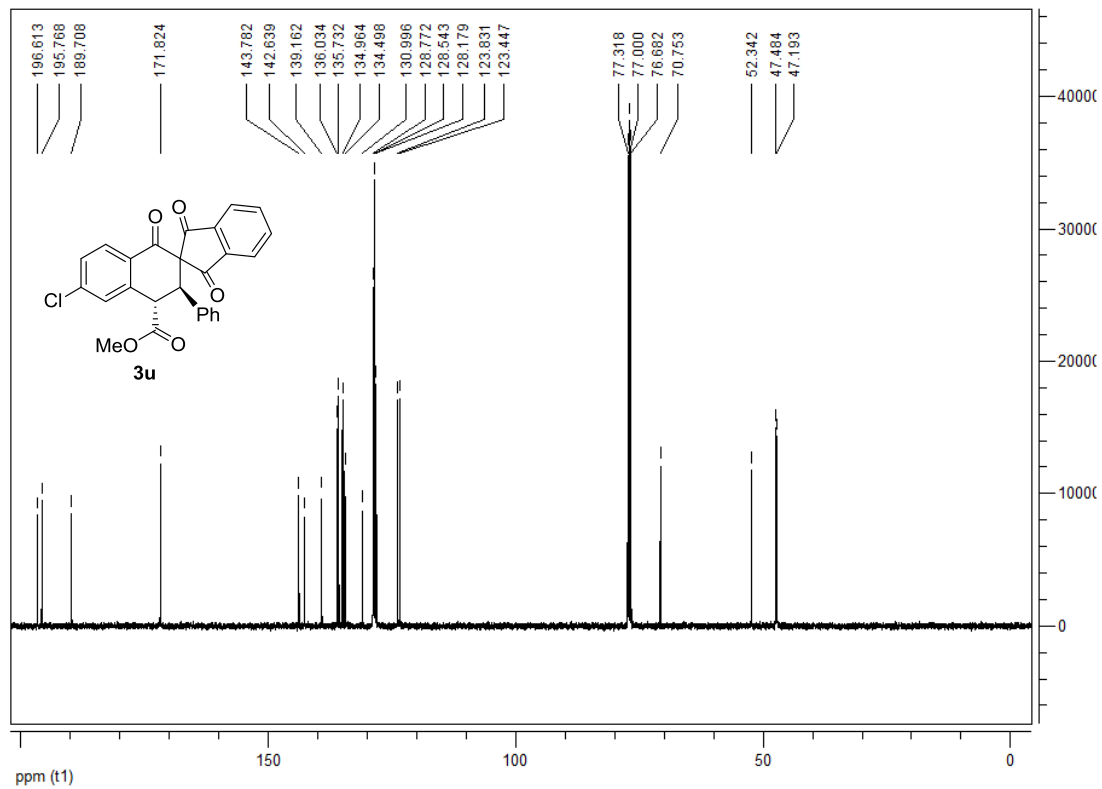
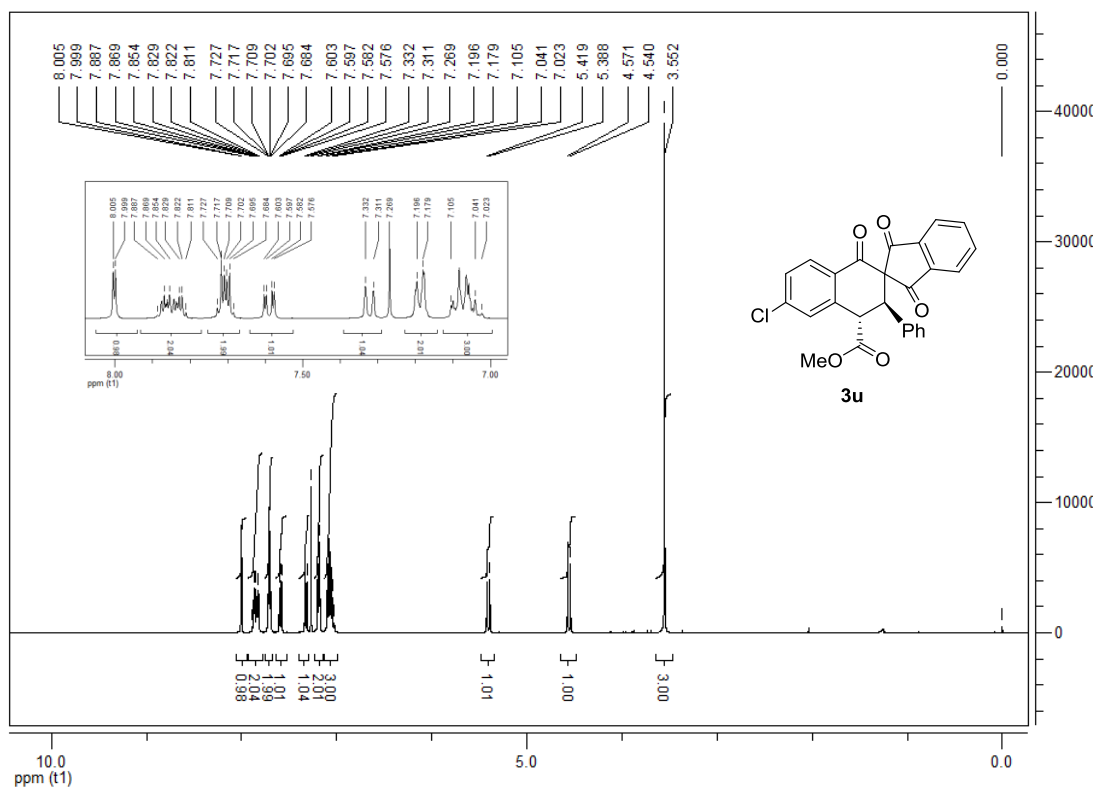


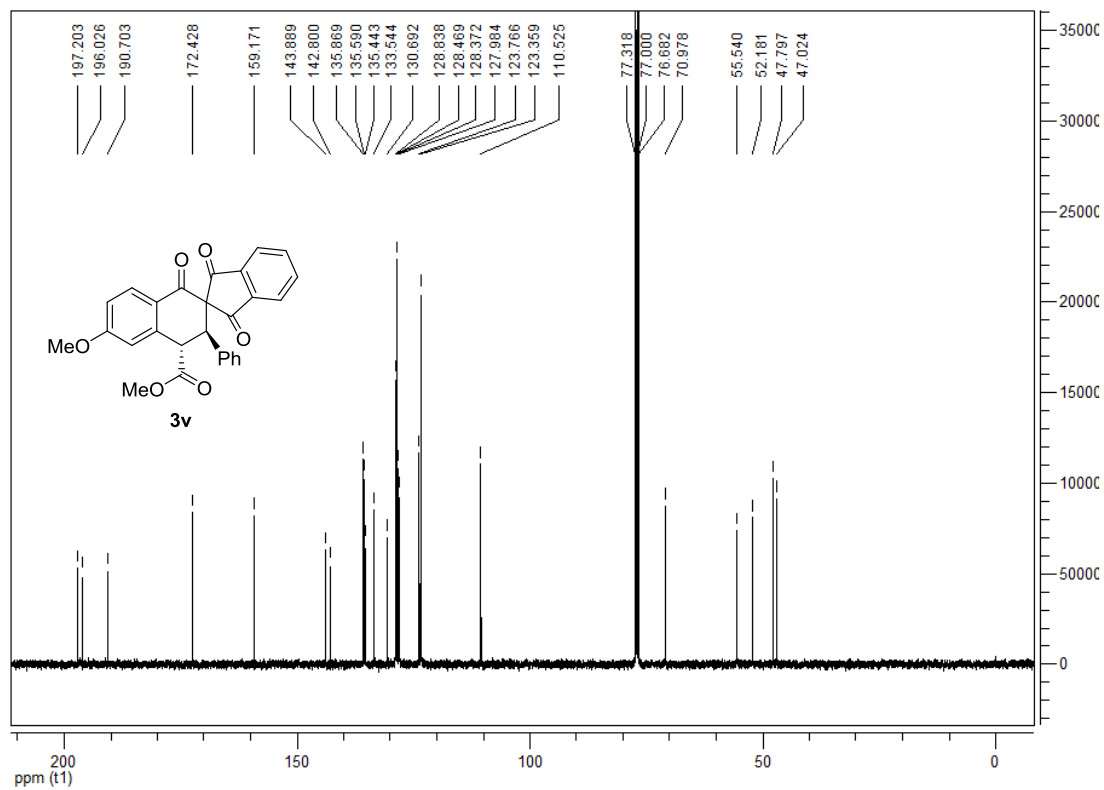
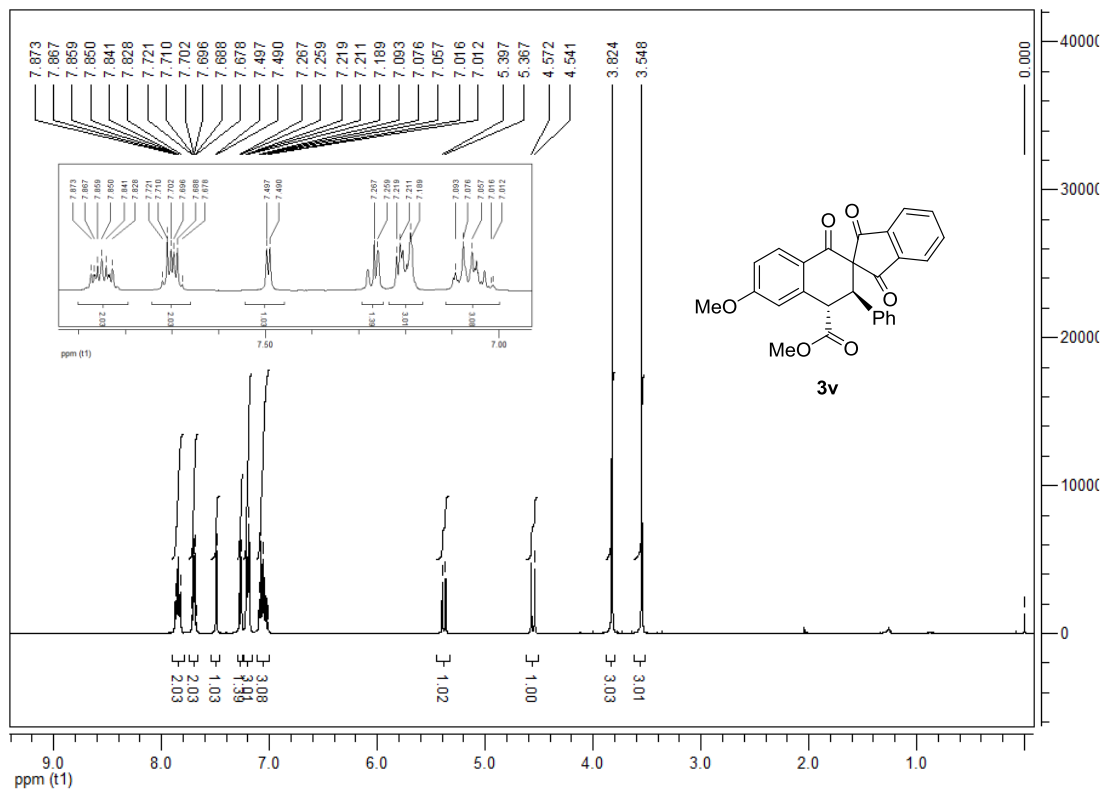




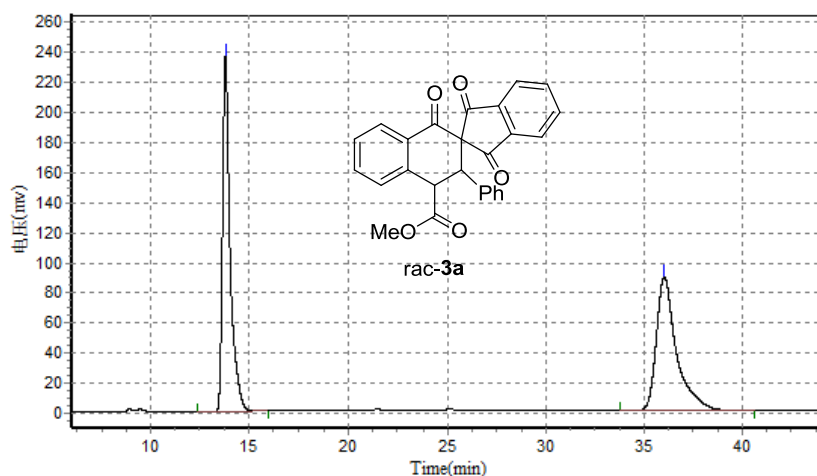






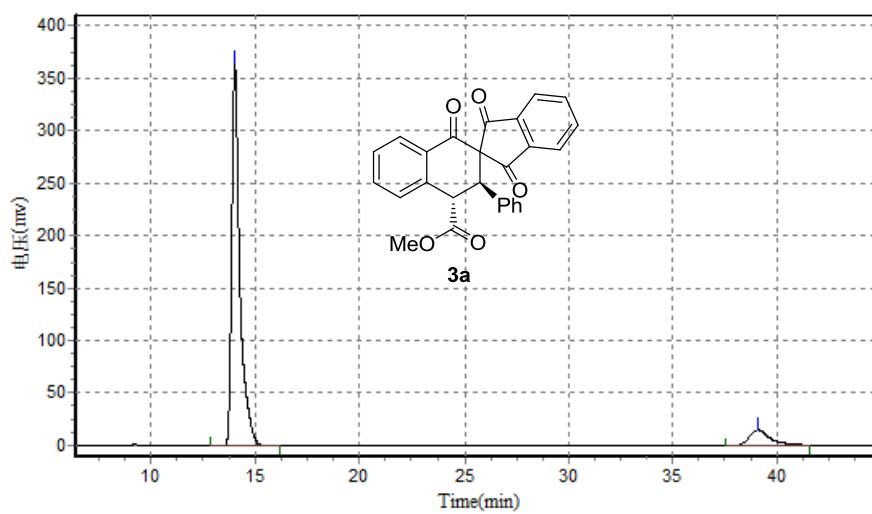


2. Copies of HPLC Spectra of the Products



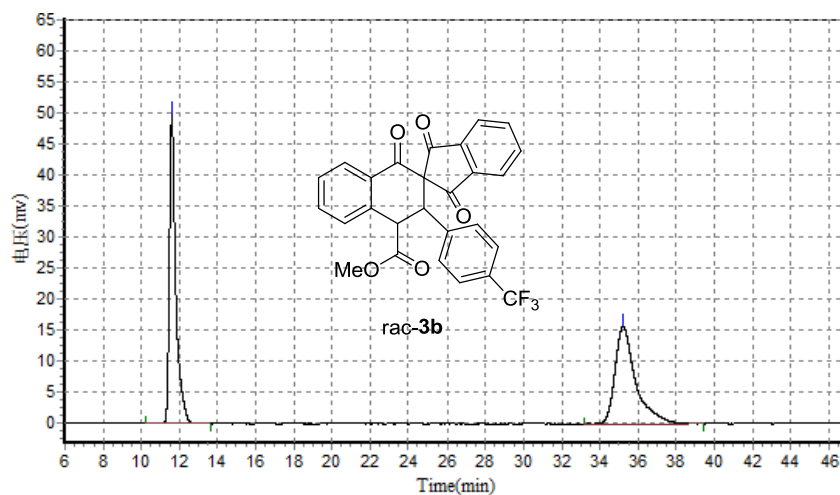
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		13.785	234925.891	6264482.000	50.0986
2		37.028	88574.148	6239822.500	49.9014
Total			323500.039	12504304.500	100.0000



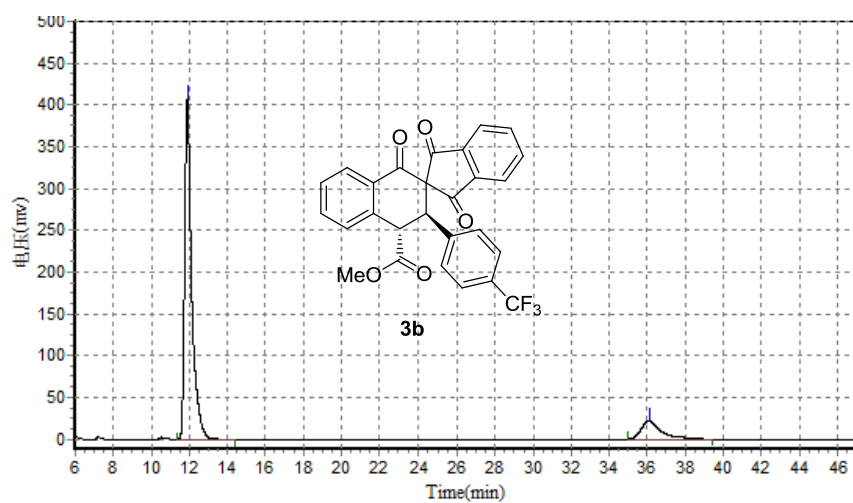
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.032	363205.938	9808152.000	90.1017
2		39.068	14880.192	1077498.250	9.8983
Total			378086.130	10885650.250	100.0000



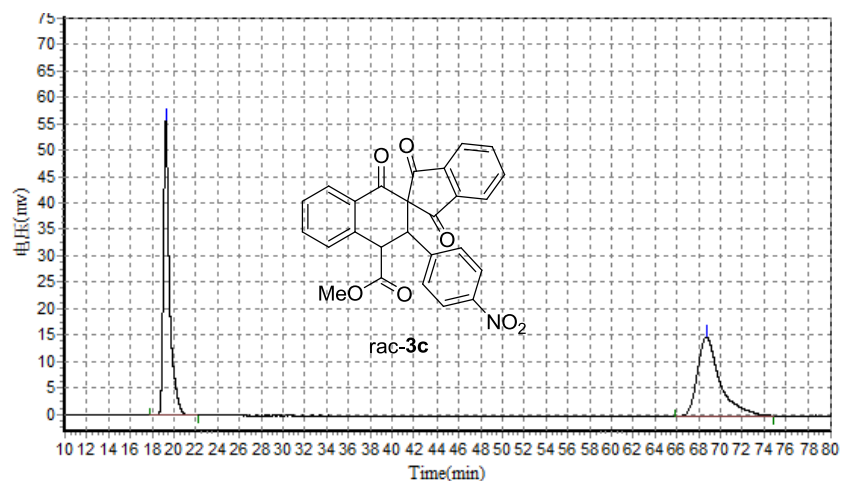
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.603	49802.445	1162055.000	49.7636
2		35.213	15746.078	1173093.750	50.2364
Total			65548.523	2335148.750	100.0000



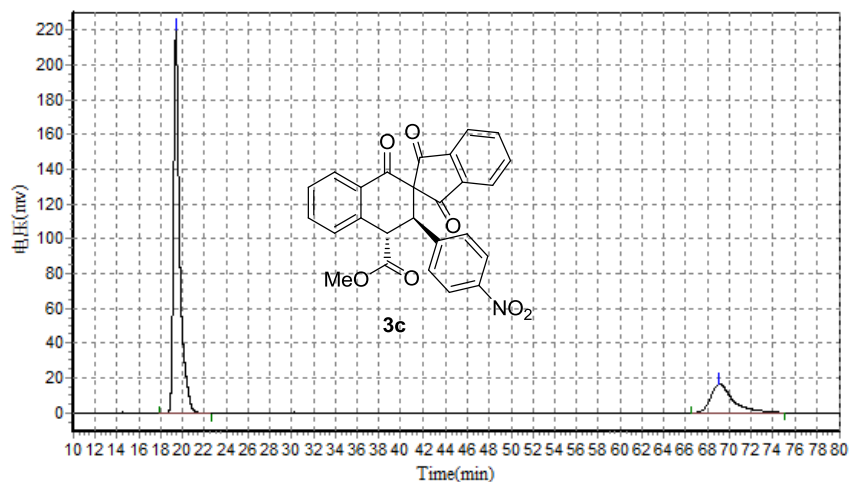
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.888	407253.406	10411565.000	87.9658
2		36.107	20937.467	1424358.375	12.0342
Total			428190.873	11835923.375	100.0000



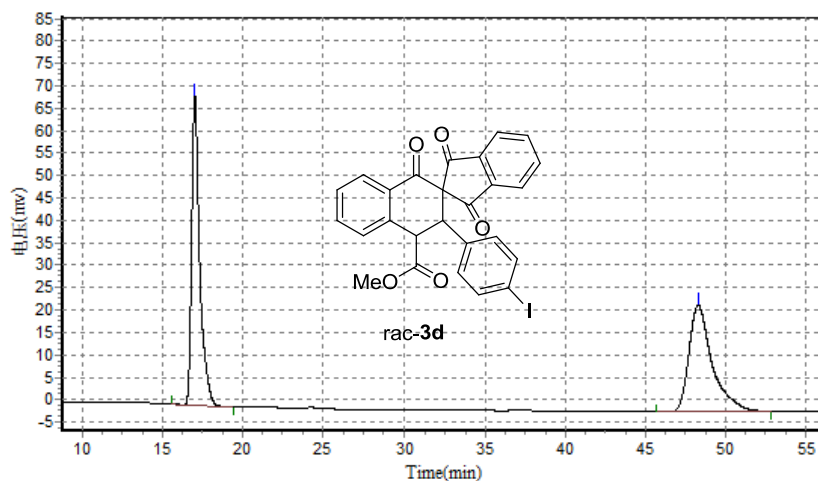
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		19.242	55791.668	2169731.250	50.2956
2		68.697	15111.603	2144227.500	49.7044
Total			70903.271	4313958.750	100.0000



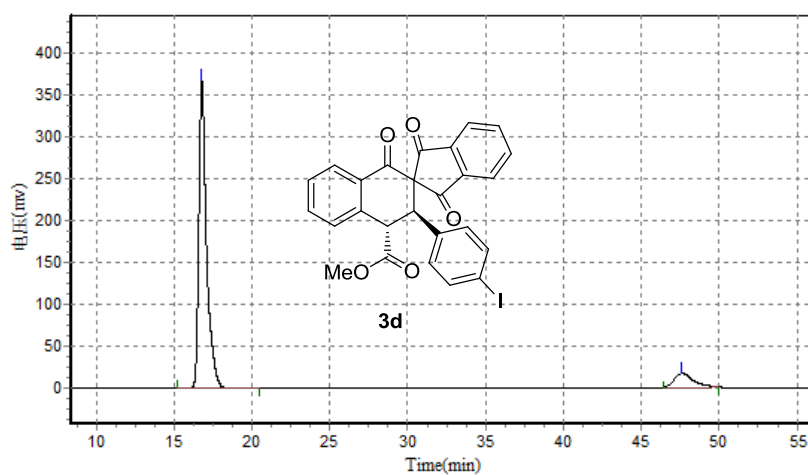
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		19.390	219065.922	8652199.000	79.8809
2		69.087	16255.794	2179180.750	20.1191
Total			235321.716	10831379.750	100.0000



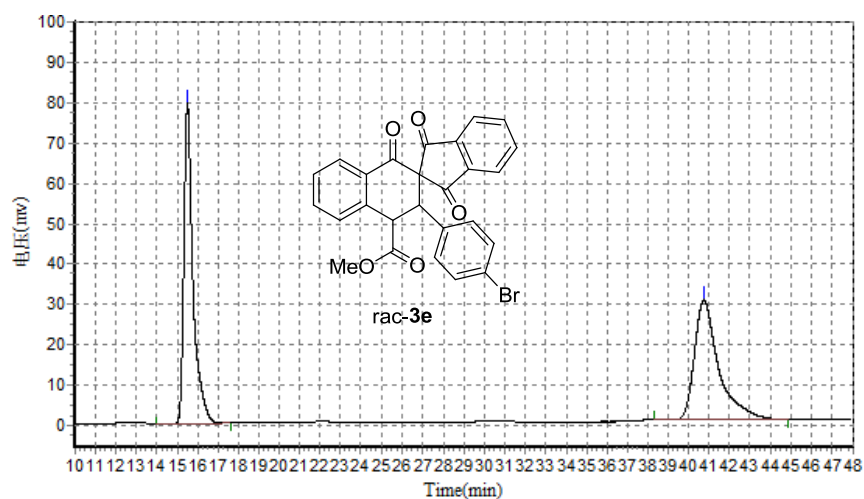
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.992	69001.328	2399230.500	50.1751
2		48.255	23740.859	2382488.250	49.8249
Total			92742.188	4781718.750	100.0000



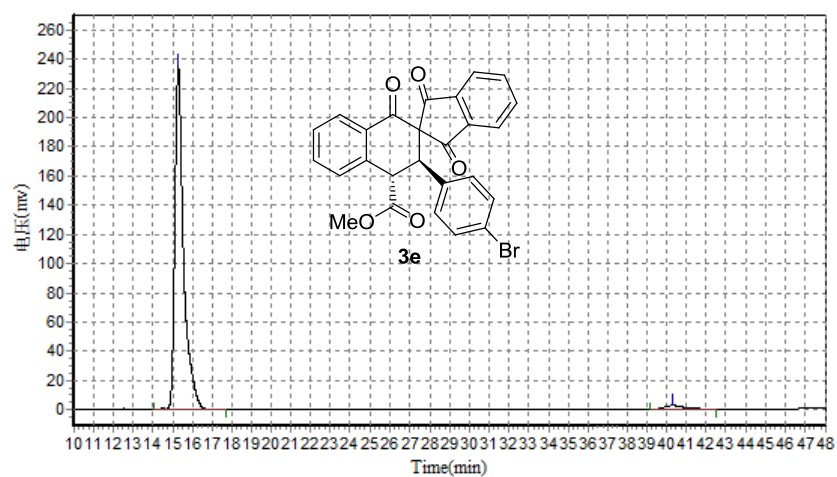
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		16.795	365810.563	12652640.000	89.6596
2		47.588	16700.760	1459225.375	10.3404
Total			382511.322	14111865.375	100.0000



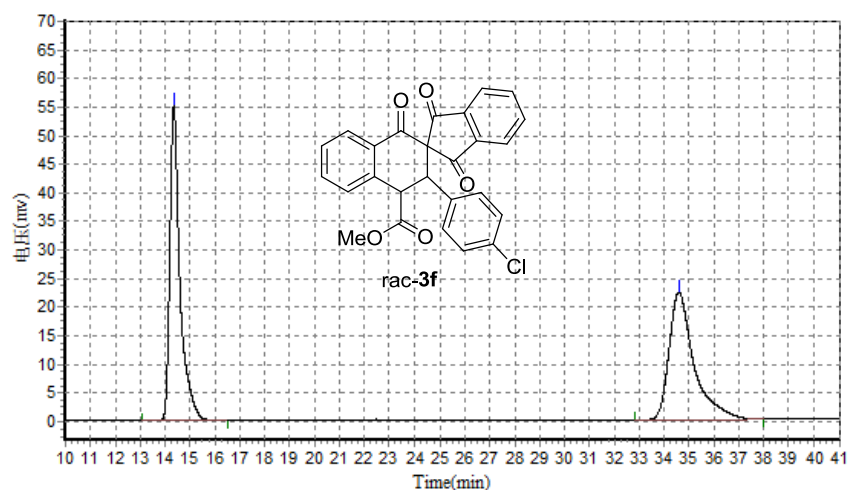
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		15.500	79406.125	2447675.250	50.0397
2		40.747	29628.045	2443791.500	49.9603
Total			109034.170	4891466.750	100.0000



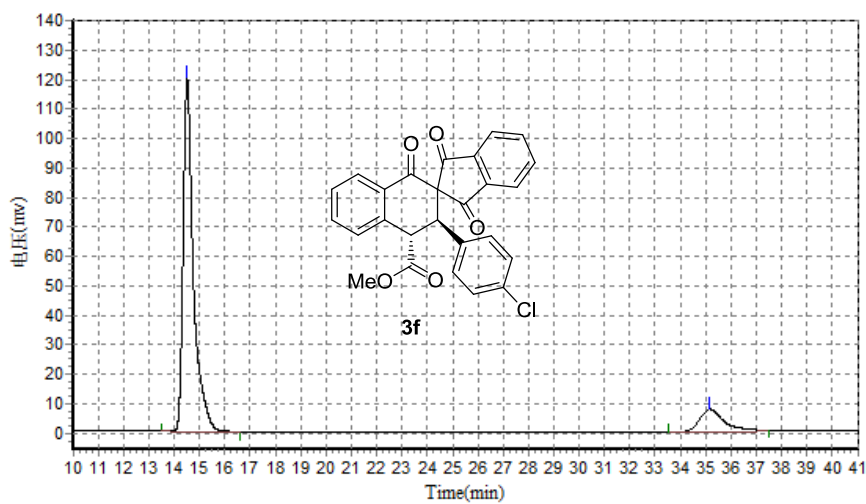
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		15.280	234500.578	7268099.500	97.4306
2		40.318	2577.626	191672.906	2.5694
Total			237078.204	7459772.406	100.0000



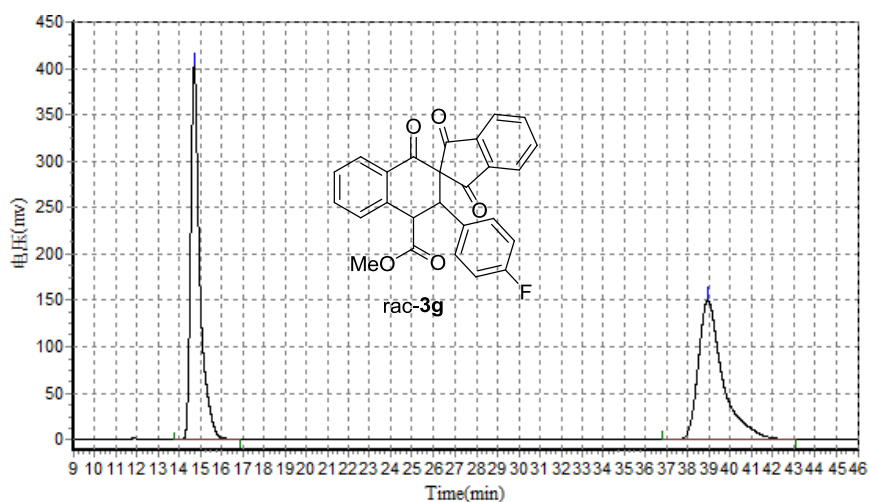
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.348	54913.785	1543037.375	49.9484
2		34.587	22289.498	1546226.750	50.0516
Total			77203.283	3089264.125	100.0000



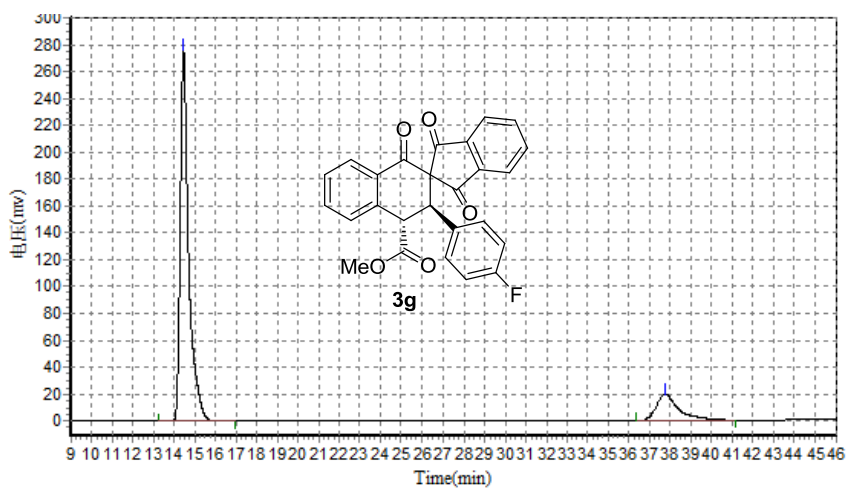
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.503	119437.602	3363848.250	87.6387
2		35.147	7414.307	474465.688	12.3613
Total			126851.909	3838313.938	100.0000



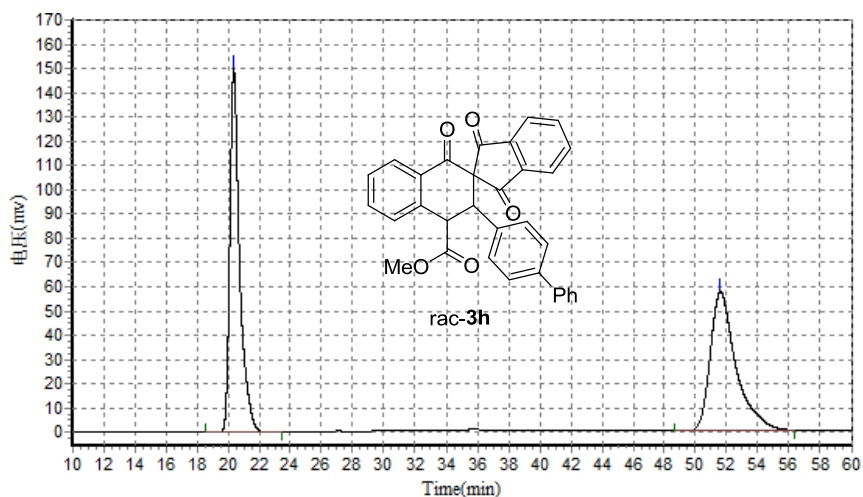
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.703	403176.813	12102807.000	50.1380
2		38.942	149986.047	12036195.000	49.8620
Total			553162.859	24139002.000	100.0000



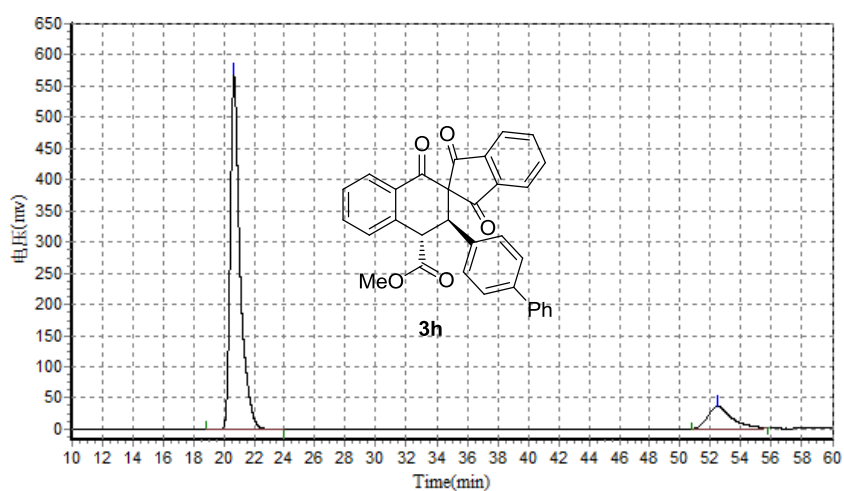
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.447	274636.000	7821081.500	84.8055
2		37.740	19252.969	1401288.625	15.1945
Total			293888.969	9222370.125	100.0000



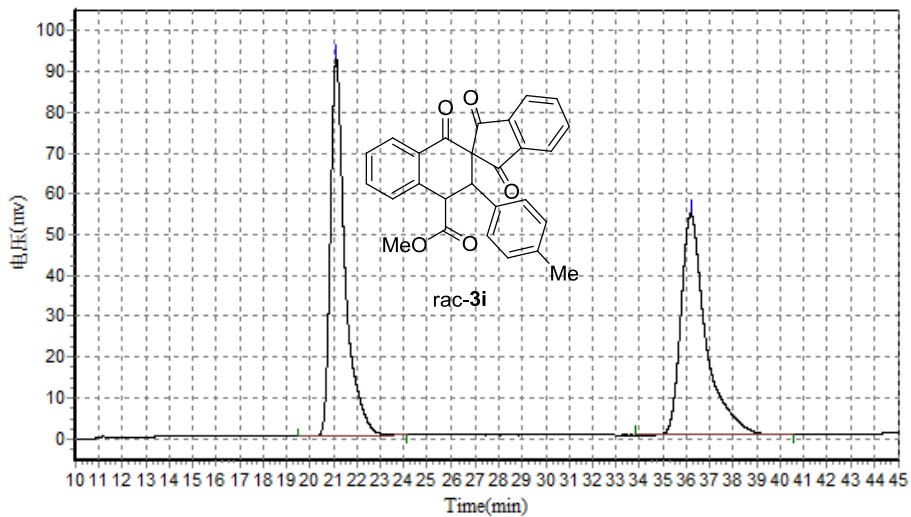
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		20.357	149957.578	6597570.000	50.2015
2		51.610	56975.645	6544610.500	49.7985
Total			206933.223	13142180.500	100.0000



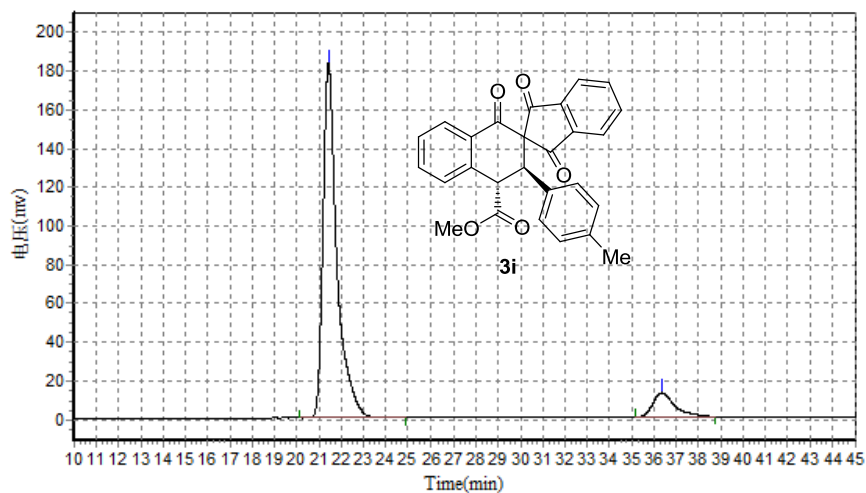
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		20.678	565879.500	25471310.000	86.6673
2		52.465	35826.254	3918439.000	13.3327
Total			601705.754	29389749.000	100.0000



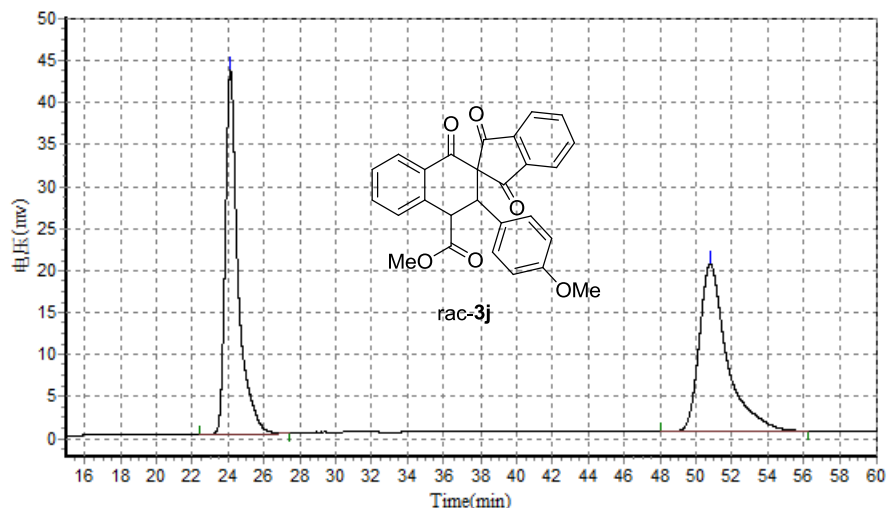
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		21.120	92482.352	4028971.750	50.0392
2		36.182	54291.777	4022654.000	49.9608
Total			146774.129	8051625.750	100.0000



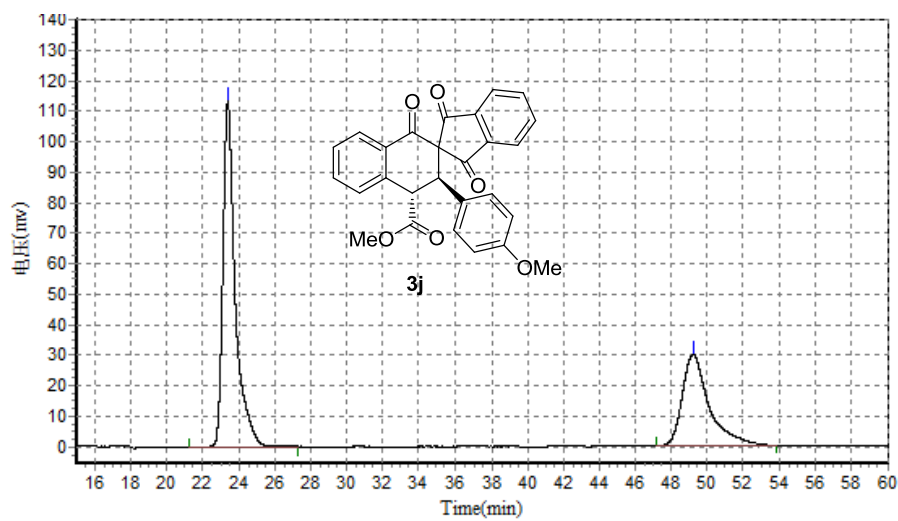
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		21.430	183230.094	7988642.500	90.4015
2		36.355	12528.111	848205.625	9.5985
Total			195758.205	8836848.125	100.0000



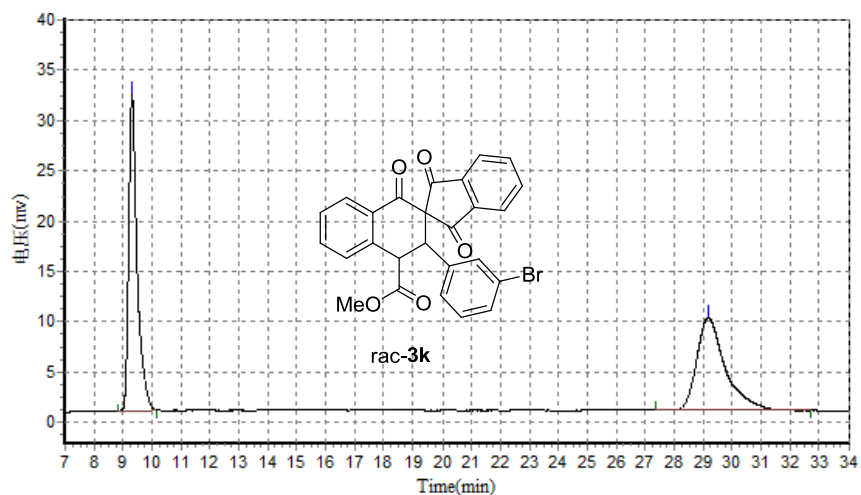
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		24.123	43280.566	2130183.750	49.8557
2		50.793	19936.539	2142513.000	50.1443
Total			63217.105	4272696.750	100.0000



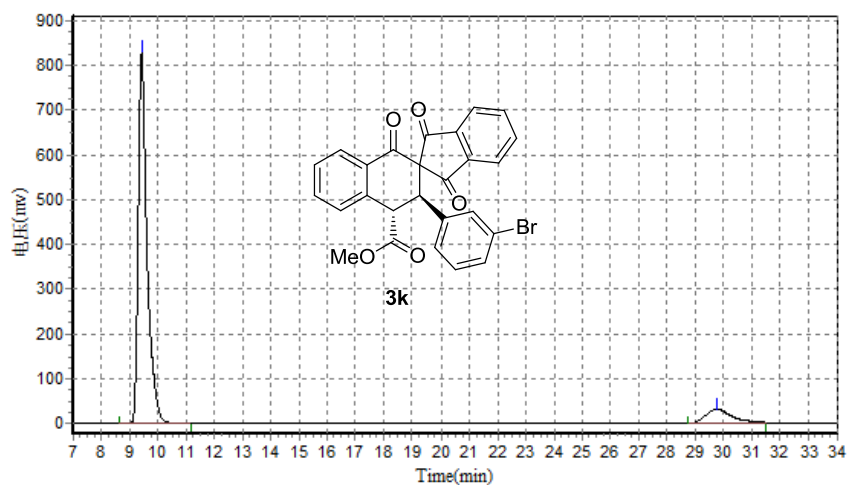
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		23.392	113054.180	5364233.000	63.8465
2		49.205	29823.295	3037529.500	36.1535
Total			142877.475	8401762.500	100.0000



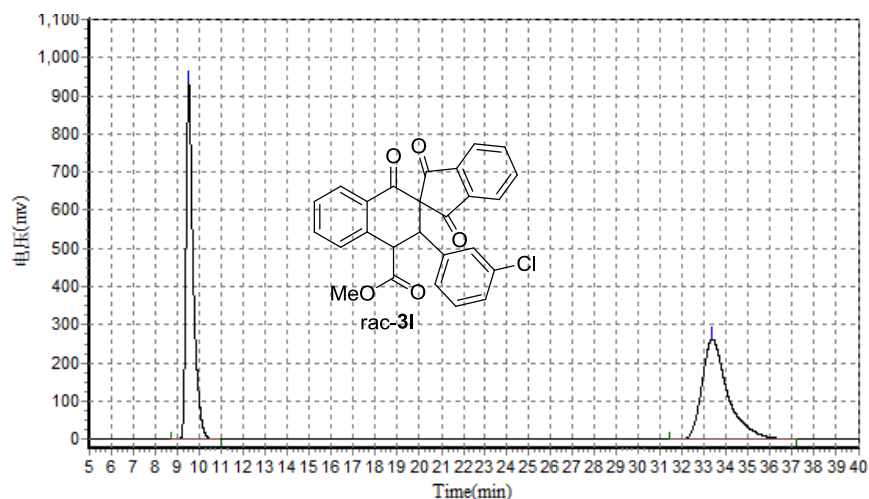
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.308	31420.369	634894.438	50.3948
2		29.178	9195.264	624946.313	49.6052
Total			40615.633	1259840.750	100.0000



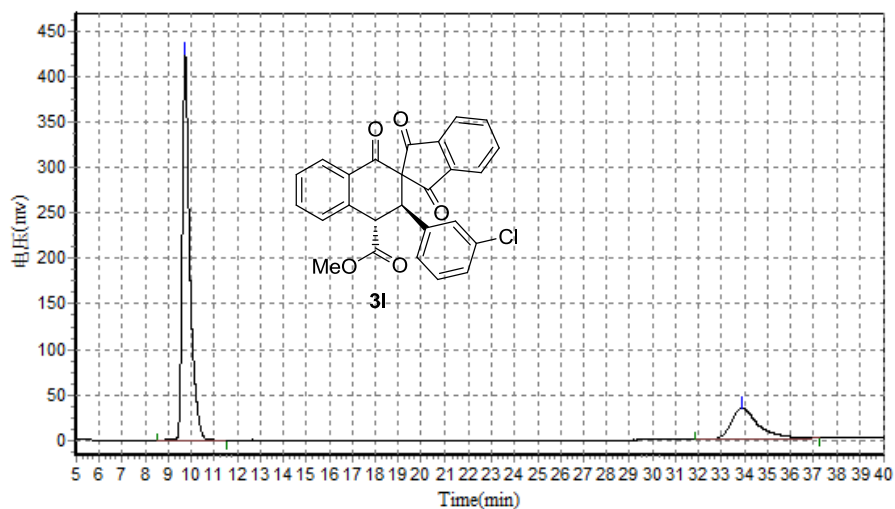
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.418	826235.438	17994558.000	89.8941
2		29.748	31525.049	2022956.625	10.1059
Total			857760.486	20017514.625	100.0000



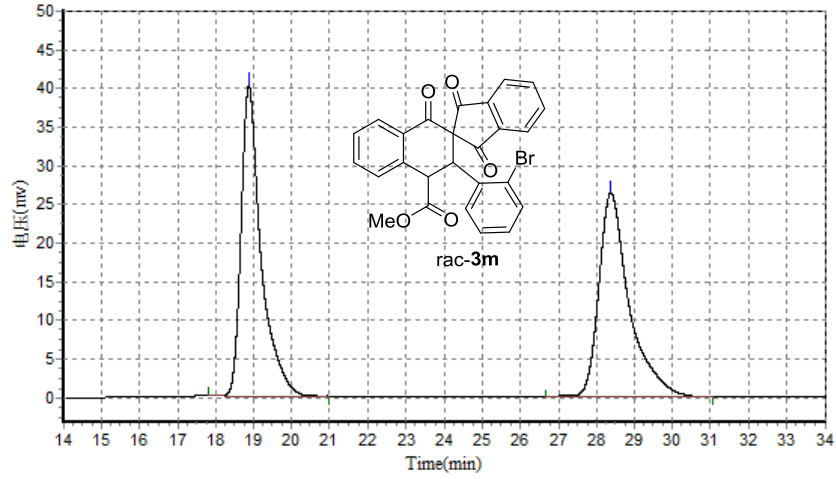
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.532	930646.188	21201748.000	49.7691
2		33.370	261600.984	21398432.000	50.2309
Total			1192247.172	42600180.000	100.0000



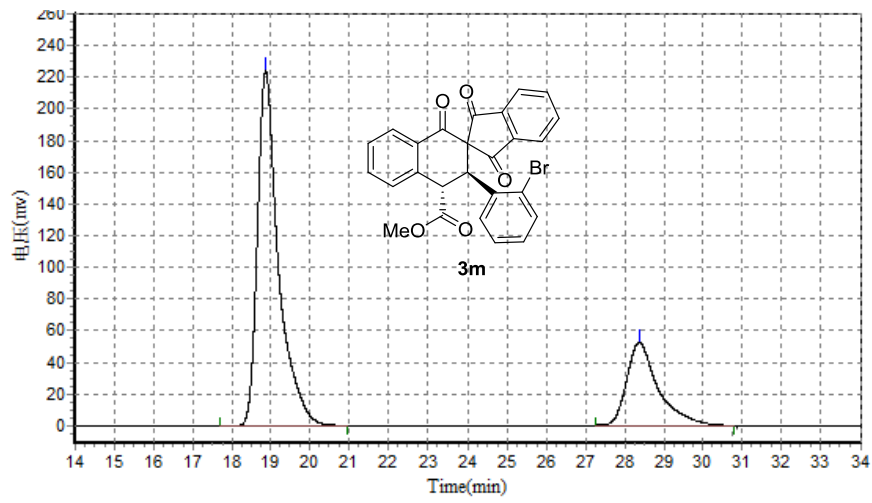
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.733	422902.156	9649351.000	77.4473
2		33.888	34277.703	2809903.000	22.5527
Total			457179.859	12459254.000	100.0000



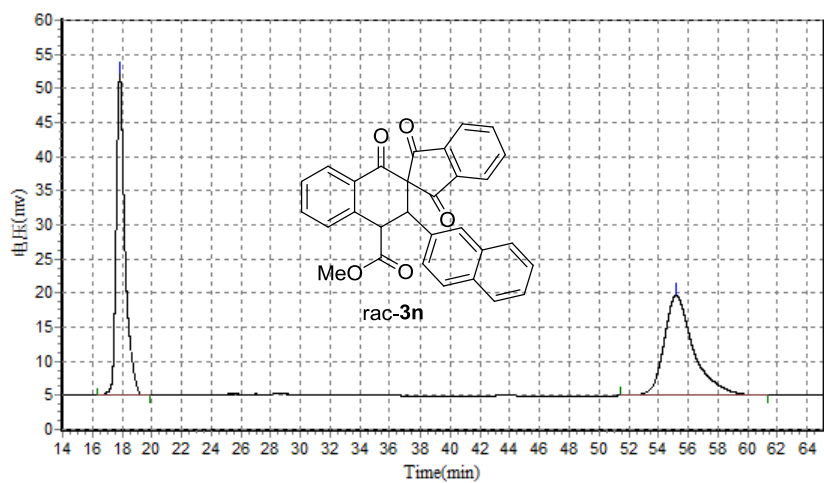
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		18.875	40157.469	1462333.000	50.0005
2		28.377	26360.756	1462304.500	49.9995
Total			66518.225	2924637.500	100.0000



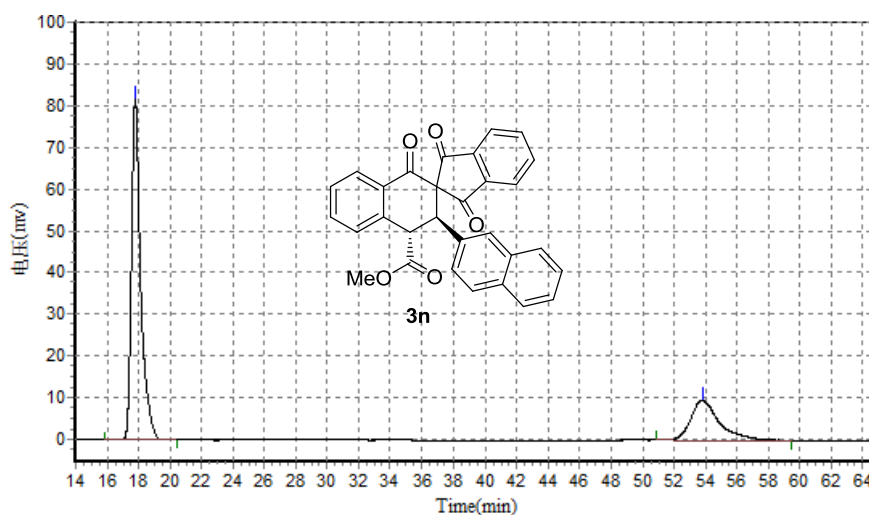
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		18.863	223519.297	8315795.500	75.1127
2		28.365	51428.359	2755297.000	24.8873
Total			274947.656	11071092.500	100.0000



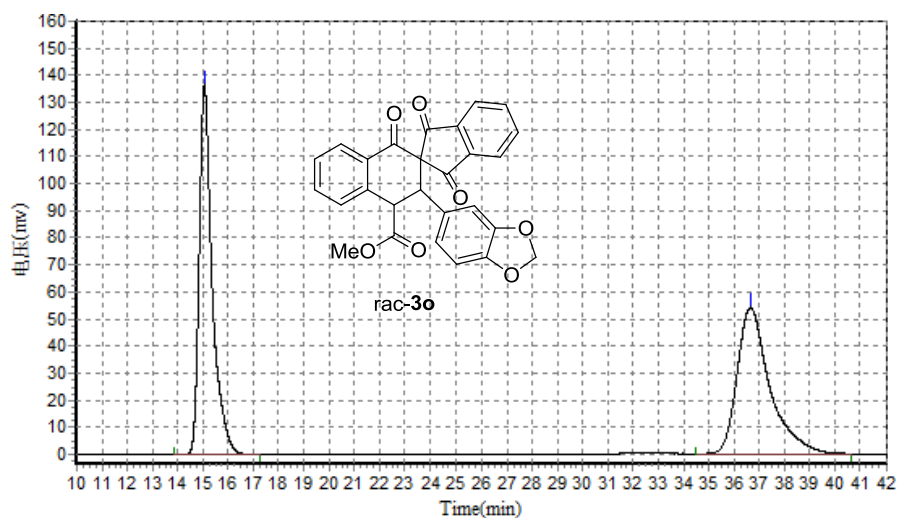
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.845	47813.891	2011220.625	50.1185
2		55.188	14642.953	2001706.500	49.8815
Total			62456.844	4012927.125	100.0000



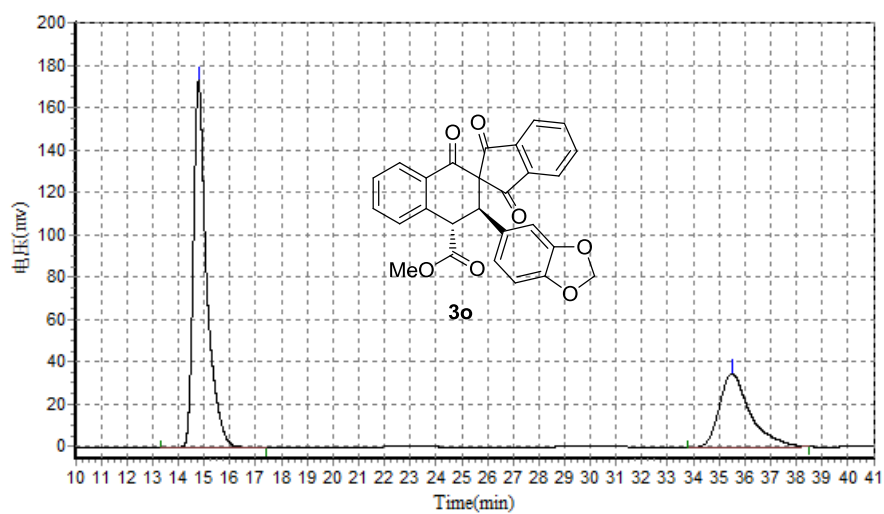
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		17.787	81683.664	3345266.500	73.4752
2		53.817	9546.980	1207650.875	26.5248
Total			91230.645	4552917.375	100.0000



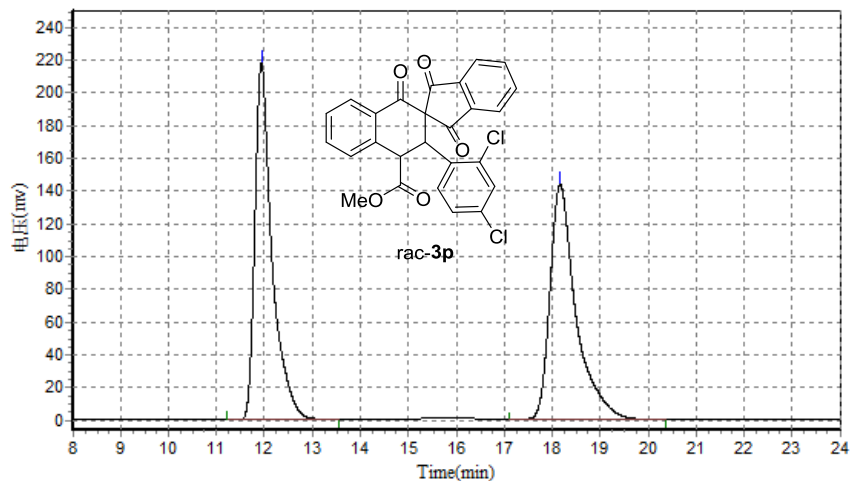
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		15.058	136497.406	4865779.000	50.1383
2		36.650	53963.191	4838931.000	49.8617
Total			190460.598	9704710.000	100.0000



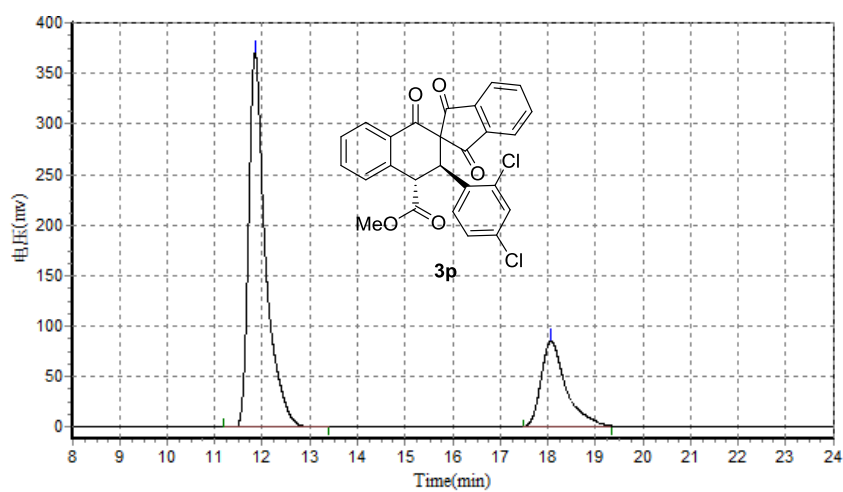
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.775	172868.156	6204607.000	68.1617
2		35.503	34850.316	2898169.250	31.8383
Total			207718.473	9102776.250	100.0000



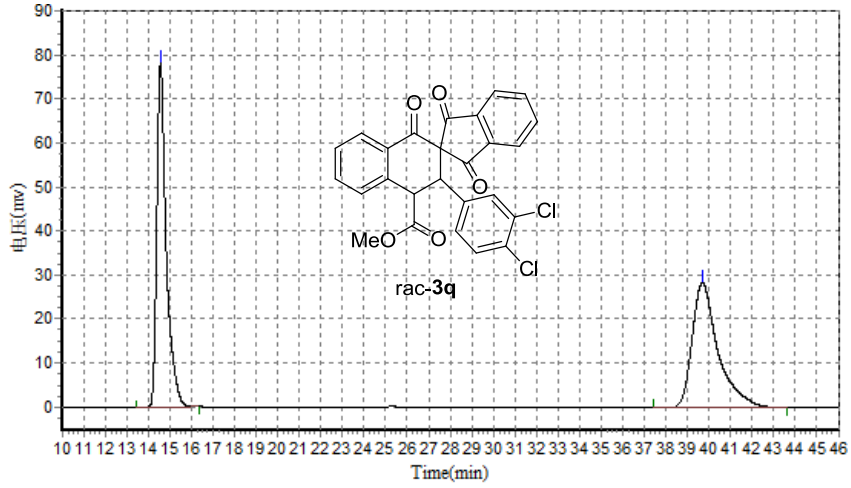
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.937	217454.844	5405534.500	50.1190
2		18.158	143143.828	5379861.500	49.8810
Total			360598.672	10785396.000	100.0000



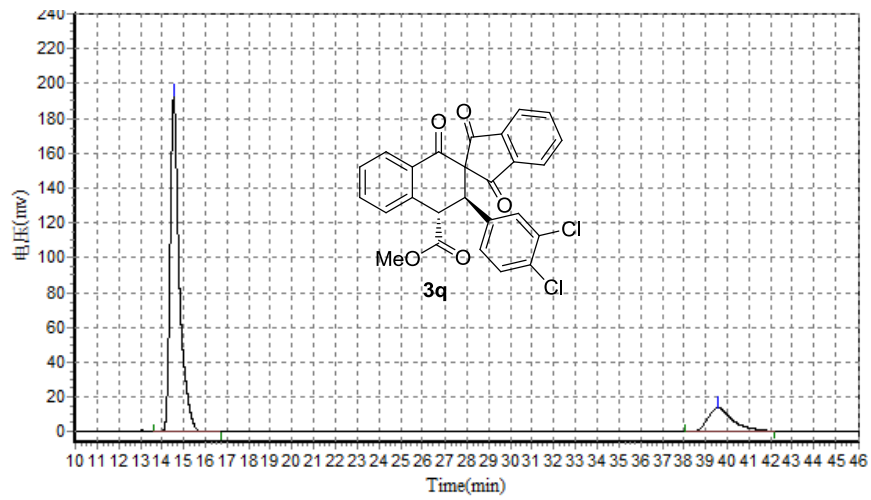
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.847	370588.906	9136808.000	74.7392
2		18.060	84606.188	3088111.250	25.2608
Total			455195.094	12224919.250	100.0000



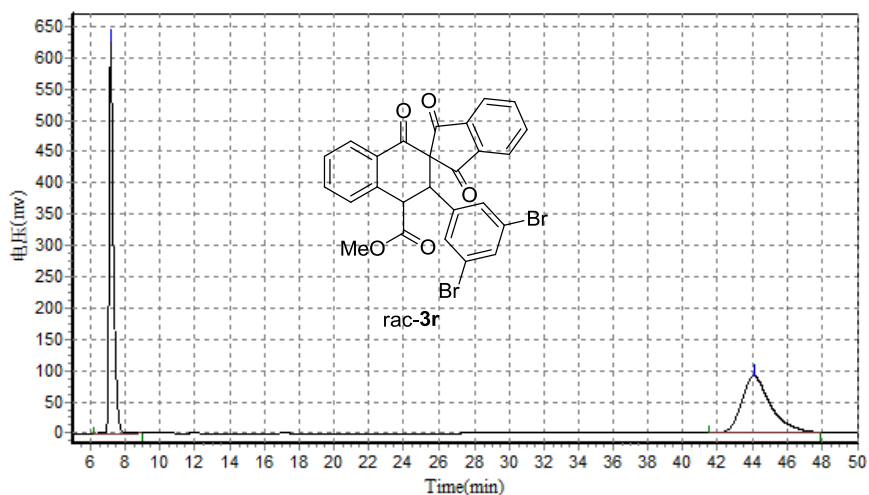
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.557	78111.055	2371497.500	49.8680
2		39.702	28330.479	2384049.500	50.1320
Total			106441.533	4755547.000	100.0000



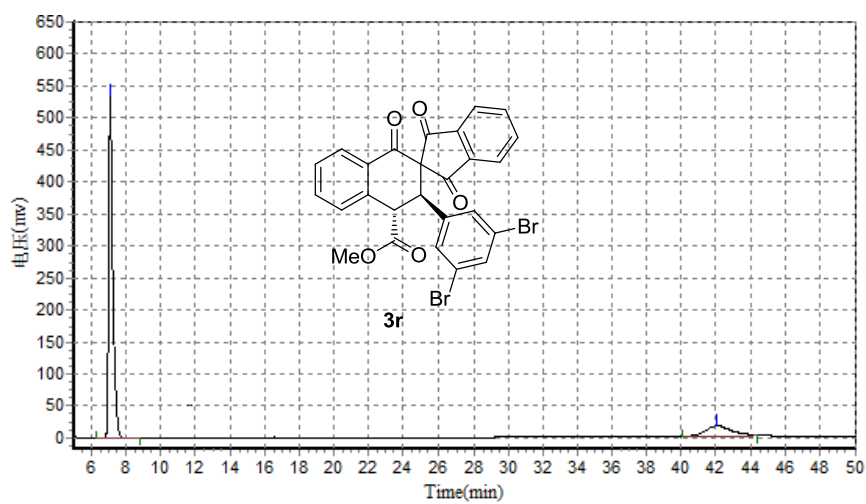
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.538	191760.063	5686325.000	84.9506
2		39.562	13553.083	1007356.750	15.0494
Total			205313.146	6693681.750	100.0000



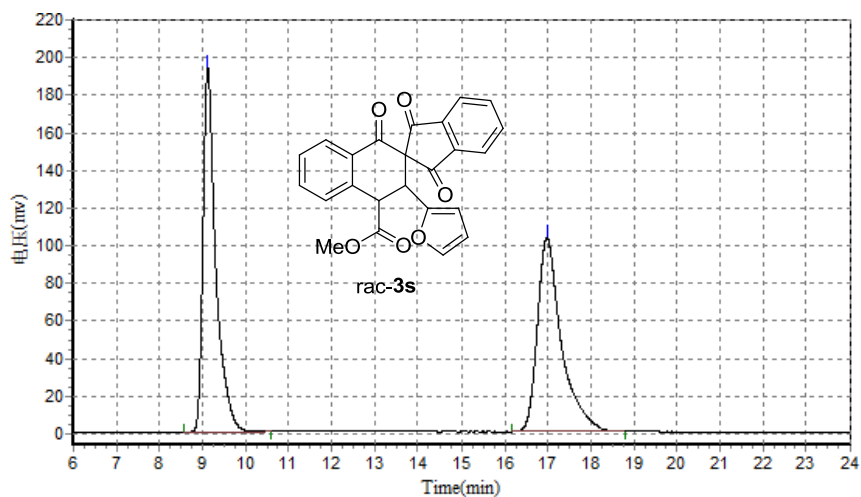
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.157	623774.875	10567547.000	50.3207
2		44.083	90327.930	10432833.000	49.6793
Total			714102.805	21000380.000	100.0000



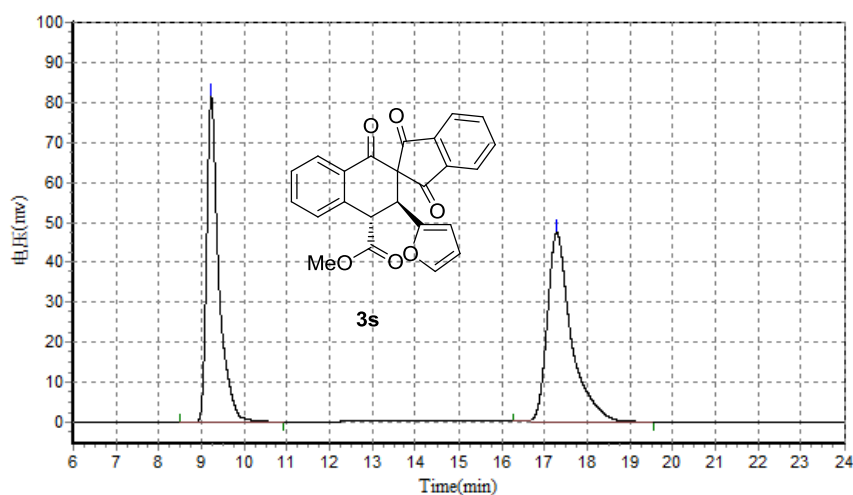
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		7.073	532768.063	8702543.000	84.8395
2		42.065	15551.573	1555110.750	15.1605
Total			548319.636	10257653.750	100.0000



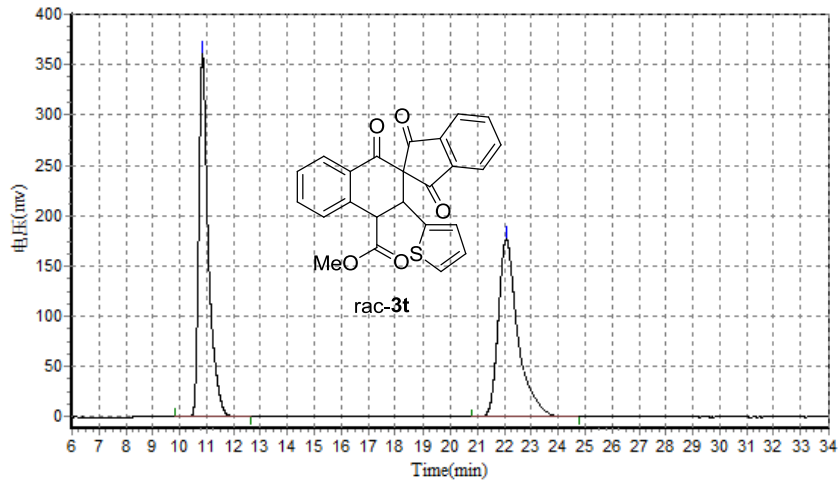
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.122	193014.828	3981055.000	49.8393
2		16.973	102974.742	4006725.250	50.1607
Total			295989.570	7987780.250	100.0000



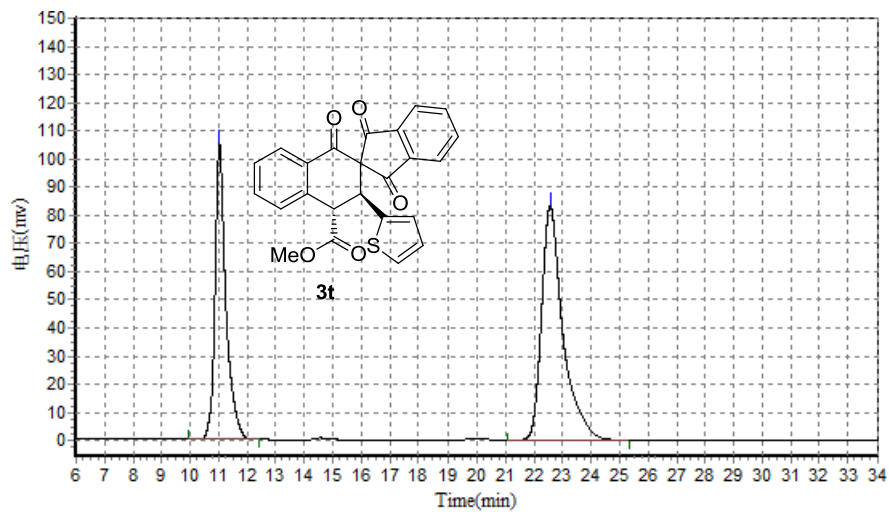
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		9.228	81345.203	1629736.000	47.1761
2		17.283	47179.543	1824841.125	52.8239
Total			128524.746	3454577.125	100.0000



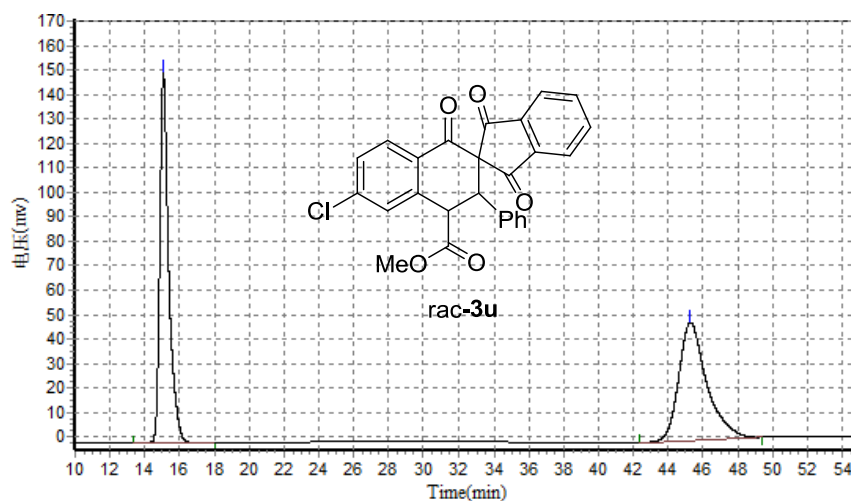
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		10.843	360366.906	8941688.000	49.7549
2		22.070	176462.703	9029800.000	50.2451
Total			536829.609	17971488.000	100.0000



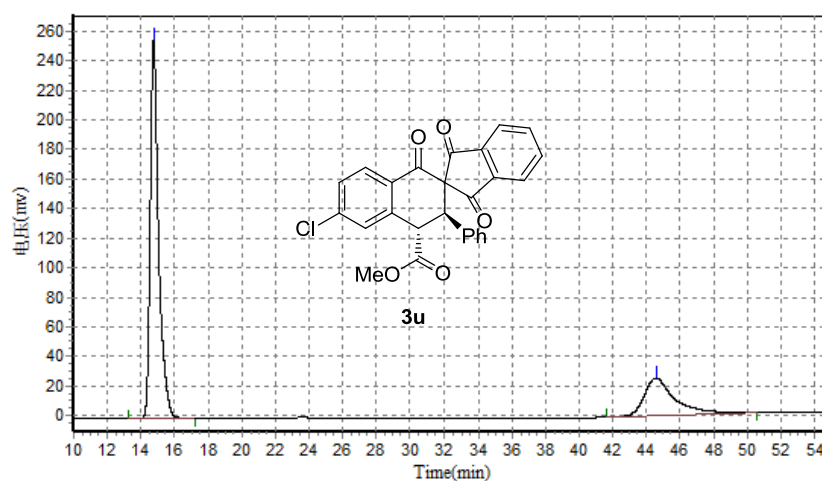
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.022	104758.867	2764435.250	39.0313
2		22.563	83085.281	4318175.000	60.9687
Total			187844.148	7082610.250	100.0000



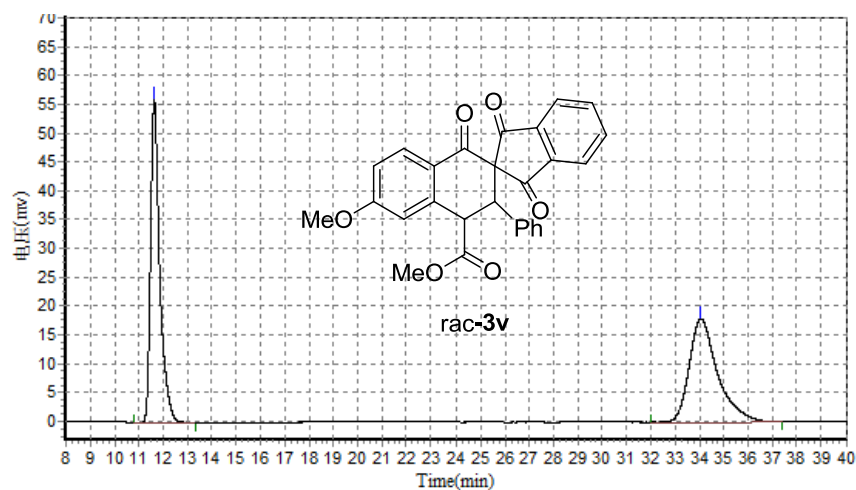
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		15.072	150856.266	5358462.000	49.8926
2		45.280	48175.836	5381528.500	50.1074
Total			199032.102	10739990.500	100.0000



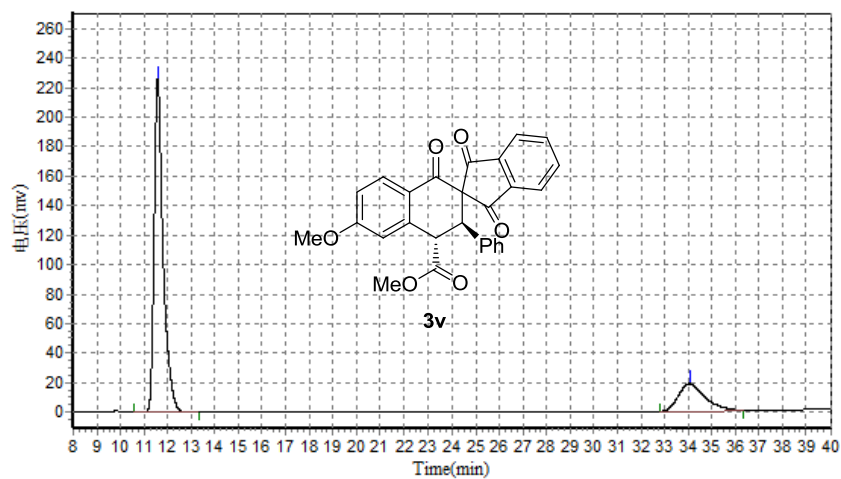
Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		14.753	255260.391	8801070.000	72.9211
2		44.563	25479.232	3268232.000	27.0789
Total			280739.623	12069302.000	100.0000



Results

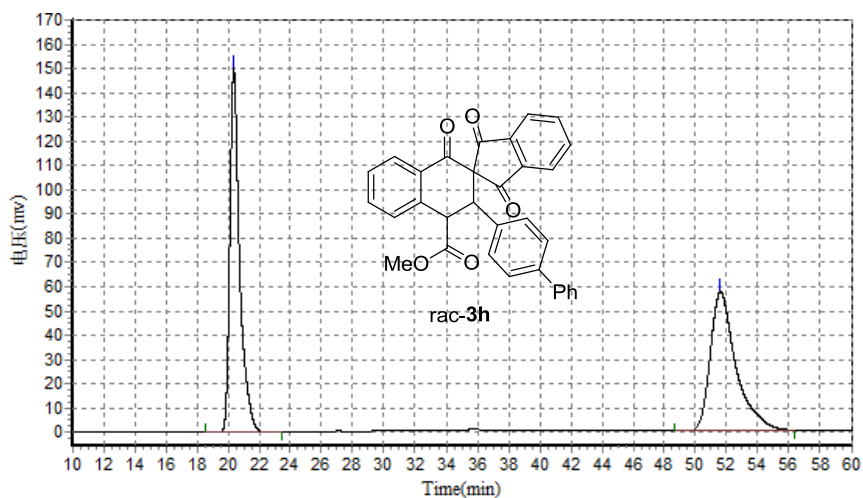
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.630	56085.117	1460984.125	50.1382
2		34.058	17955.318	1452932.375	49.8618
Total			74040.436	2913916.500	100.0000



Results

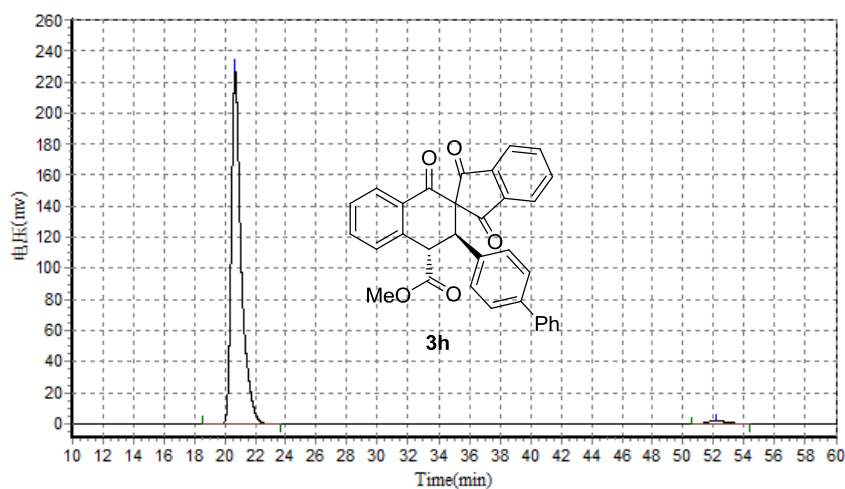
Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		11.578	226267.219	5971486.500	80.0734
2		34.063	18759.893	1486025.625	19.9266
Total			245027.111	7457512.125	100.0000

Compound **3h** transformed from product **3e**



Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		20.357	149957.578	6597570.000	50.2015
2		51.610	56975.645	6544610.500	49.7985
Total			206933.223	13142180.500	100.0000



Results

Peak No.	Peak ID	Ret Time	Height	Area	Conc.
1		20.531	229462.416	6746210.000	97.3685
2		52.288	2311.835	151472.000	2.6315
Total			231774.251	6897682.000	100.0000

3. X-ray Analysis of Product 3e

The crystal data of compound **3e** have been deposited in CCDC with number 1584009.

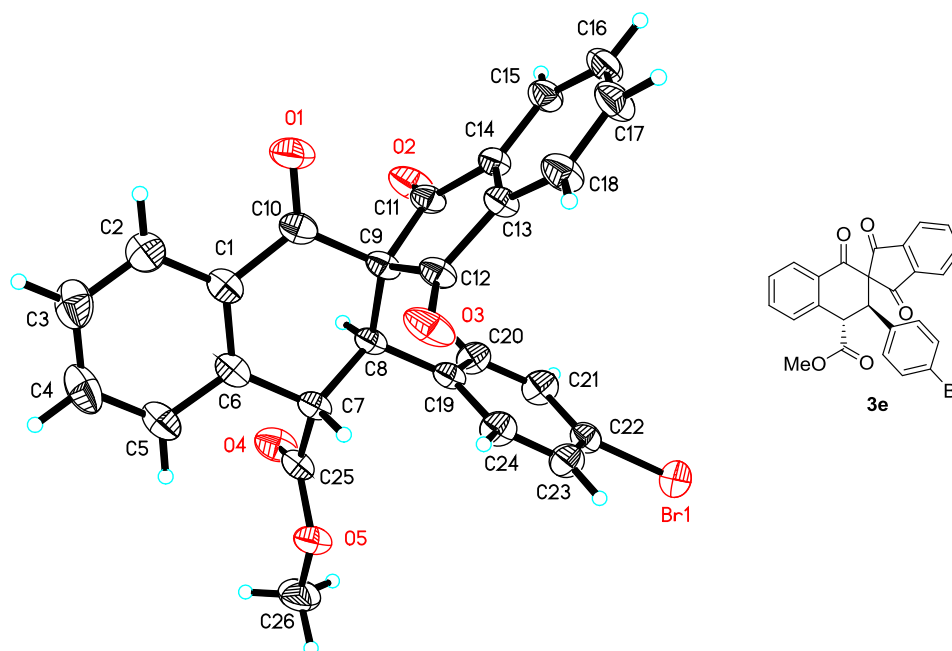


Table 1. Crystal data and structure refinement for compound **3e**.

Identification code	d8v17497	
Empirical formula	C ₂₆ H ₁₇ Br O ₅	
Formula weight	489.30	
Temperature	296(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P 21 21 21	
Unit cell dimensions	a = 8.3403(2) Å	α = 90 °
	b = 15.6568(4) Å	β = 90 °
	c = 16.7849(4) Å	γ = 90 °
Volume	2191.81(9) Å ³	
Z	4	
Density (calculated)	1.483 Mg/m ³	
Absorption coefficient	1.911 mm ⁻¹	
F(000)	992	
Crystal size	0.200 x 0.170 x 0.120 mm ³	
Theta range for data collection	2.754 to 26.000 °	
Index ranges	-10 ≤ h ≤ 10, -19 ≤ k ≤ 19, -20 ≤ l ≤ 20	
Reflections collected	36594	

Independent reflections	4270 [R(int) = 0.0405]
Completeness to theta = 25.242 °	99.1 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.7456 and 0.5252
Refinement method	Full-matrix least-squares on F ²
Data / restraints / parameters	4270 / 0 / 291
Goodness-of-fit on F ²	1.022
Final R indices [I>2sigma(I)]	R1 = 0.0276, wR2 = 0.0669
R indices (all data)	R1 = 0.0327, wR2 = 0.0694
Absolute structure parameter	0.011(3)
Extinction coefficient	0.025(2)
Largest diff. peak and hole	0.311 and -0.347 e.Å ⁻³