

One-Pot Synthesis of Enantioenriched α -Amino Lactones, Lactams and Polycyclic Derivatives with Multiple Stereogenic Centers

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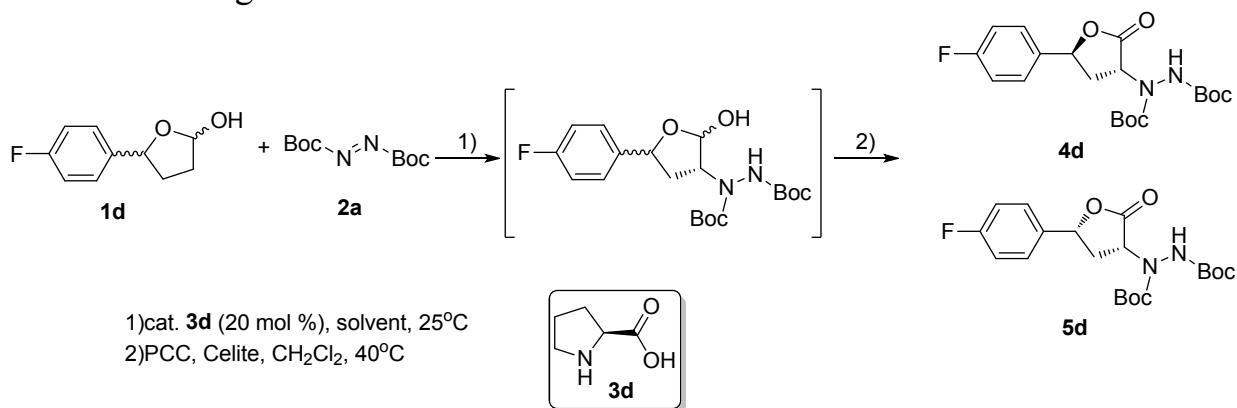
Supporting Information

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1. Details of Reaction Conditions Study

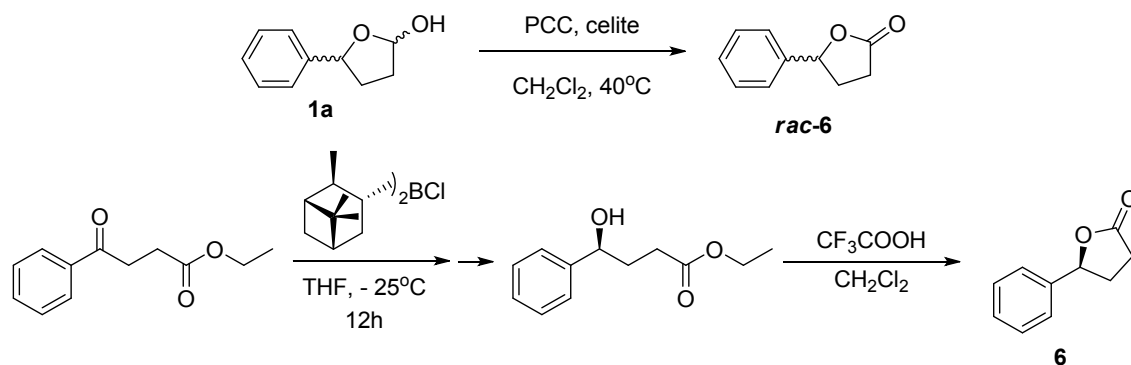
Table 1. Screening of solvents^a



Entry	Solvent	Time[h] ^b	ee[%] ^c 4d	Yield[%] ^d total
1	CH ₃ OH	6	17	78.3
2	CH ₂ Cl ₂	24	88	81.2
3	DMSO	2	30	73.4
4	DMF	6	80	70
5	CH ₃ CN	Overnight	93	87.1
6^e	CH ₃ CN/H ₂ O (9:1)	--	--	--
7^f	Et ₂ O	--	--	--

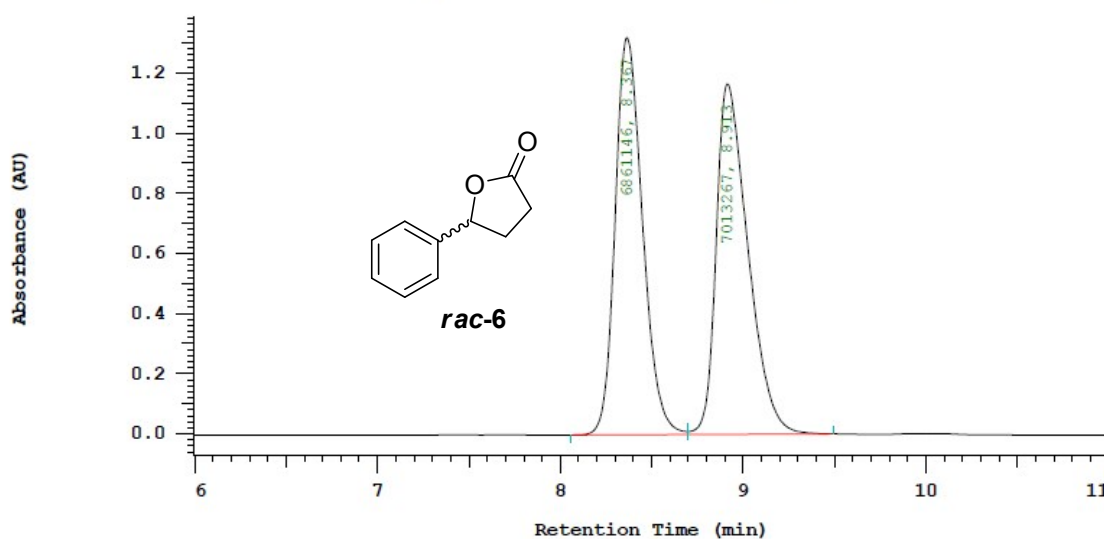
[a] Unless otherwise specified, all reactions were carried out using **1d** (0.10 mmol, 1.0 equiv), **2a** (0.12 mmol, 1.2 equiv) in solvent (0.2 mL) with cat. **3d** (20 mol %) at 25 °C. After workup, the crude product was oxidized by PCC to afford **4d** and **5d** after purified by flash chromatography on silica gel. [b] For the first step. [c] Determined by Chiral HPLC analysis. [d] Total yield of **4d** and **5d**. [e] The reaction was too slow. [f] No reaction.

2. The kinetic resolution experiment

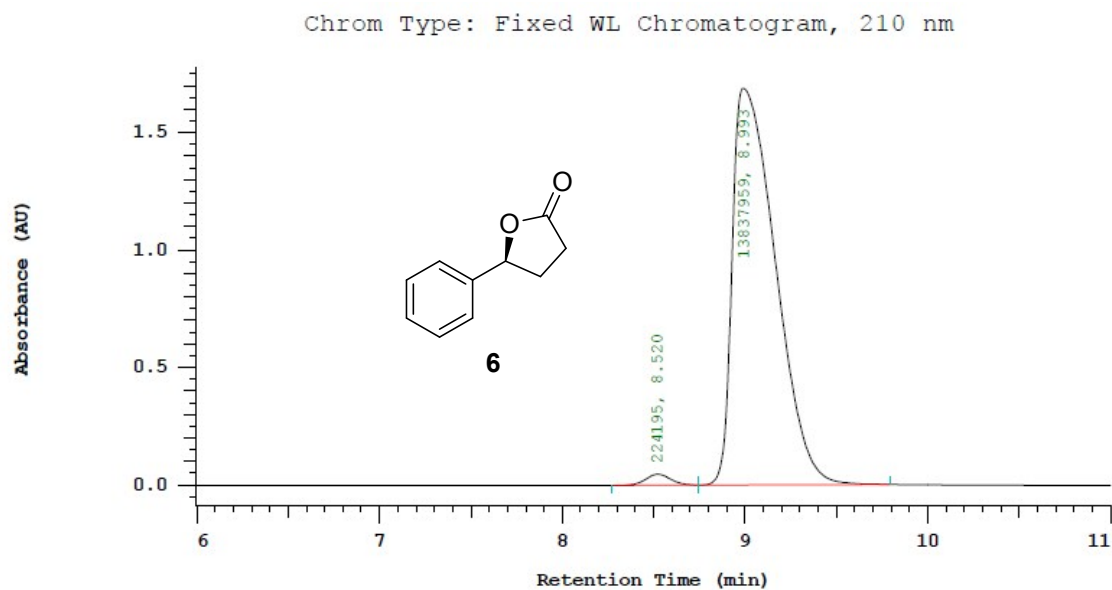


In order to explore if there was kinetic resolution in this reaction process. First, we got the racemic and chiral product of **6**. The racemic modification of **6** (*rac*-6) was obtained by PCC oxidation of **1a**. The chiral product of **6** whose ee is 97% was obtained through the literature method.¹ And their HPLC Traces are shown below.

Chrom Type: Fixed WL Chromatogram, 210 nm

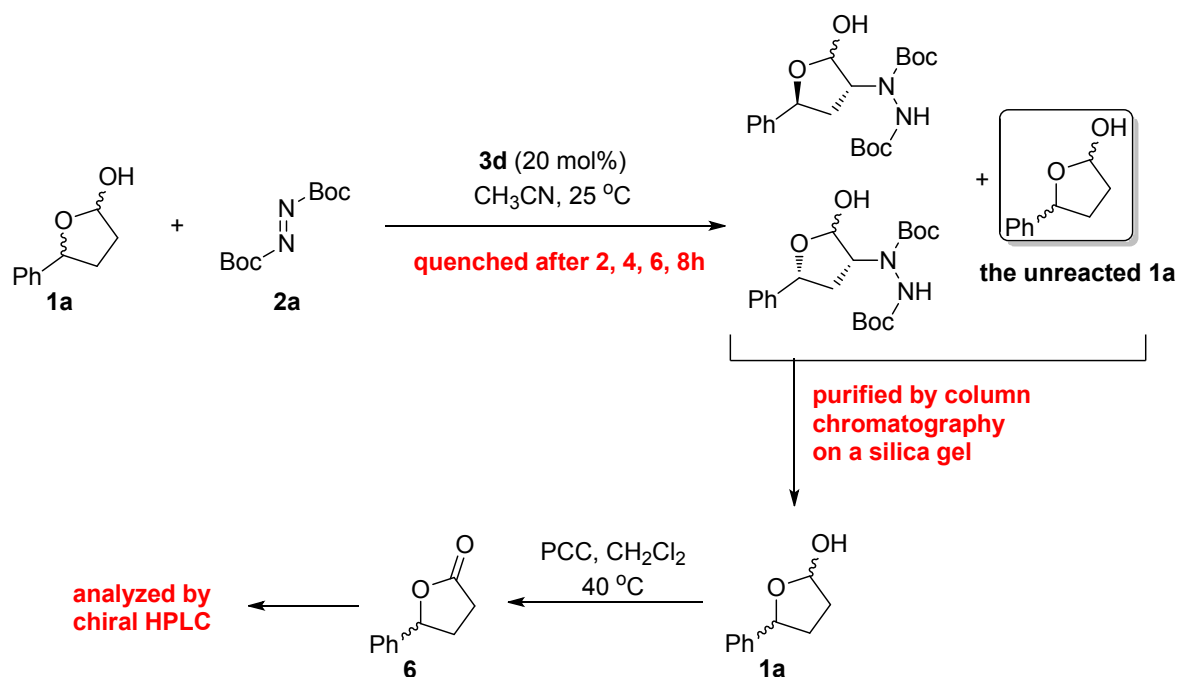


1. P. V. Ramachandran, S. Pitre, H. C. Brown, *J. Org. Chem.* **2002**, 67, 5315-5319.



Chrom Type: Fixed WL Chromatogram, 210 nm
 Peak Quantitation: AREA
 Calculation Method: AREA%

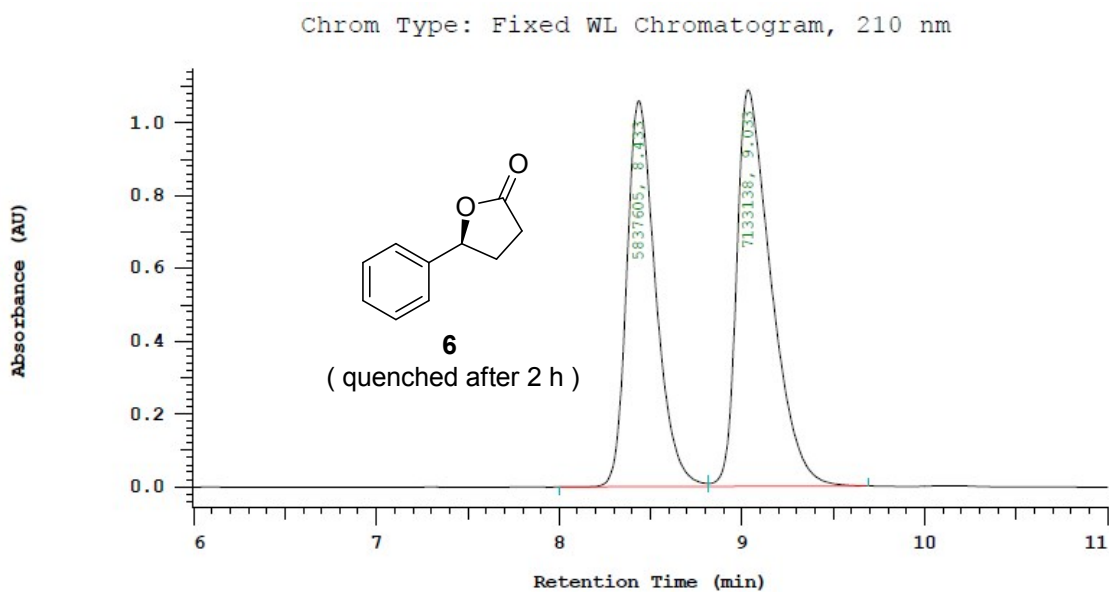
No.	RT	Area	Area %	BC
1	8.520	224195	1.594	BV
2	8.993	13837959	98.406	VB
		14062154	100.000	



We carried out Several parallel reactions between **1a** (16.4mg, 0.1mmol) and **2a** (27.6mg, 0.12mmol) in CH_3CN in the presence of **3d** (2.3mg, 20 mol%). As the reaction was completed after 9 hours, we quenched these reactions after 2 h, 4 h, 6 h, and 8 h, respectively, and the unreacted **1a** remained in the reaction solution was isolated and directly oxidized into lactone **6** with PCC. After purified by flash chromatography on silica gel, the product was

analyzed by chiral HPLC. Notable kinetic resolution was achieved and the enantiomeric excess of **6** was increased along with longer reaction time until the reaction was complete. The enantiomeric excess and yield of **6** are shown below.

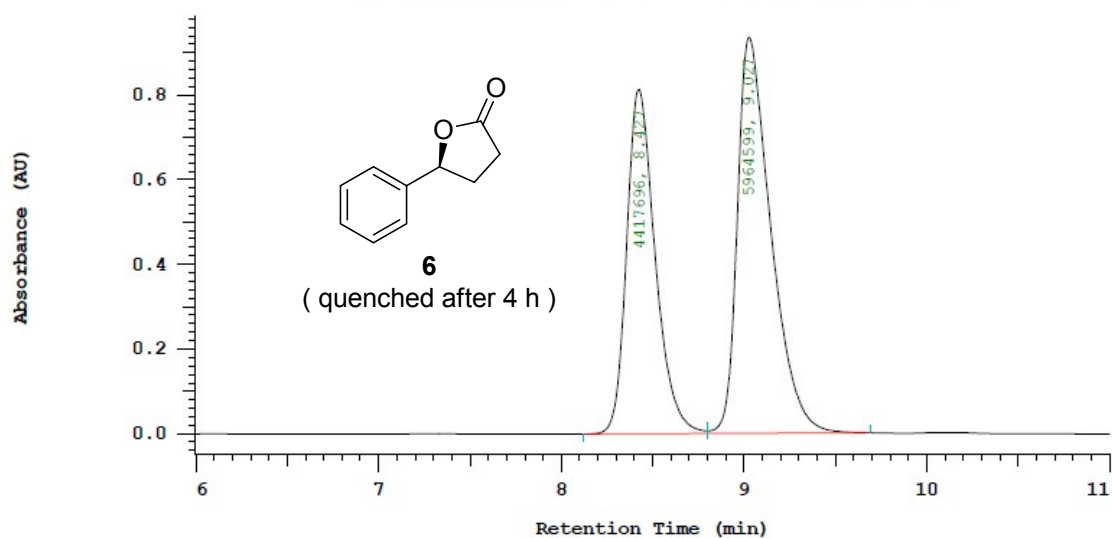
Time	2 h	4 h	6 h	8 h
ee [%]	10	15	25	34
Yield of 6	49.4% (8.0 mg)	34.6% (5.6 mg)	19.8% (3.2 mg)	<6.2% (<1 mg)



Chrom Type: Fixed WL Chromatogram, 210 nm
 Peak Quantitation: AREA
 Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.433	5837605	45.006	BV
2	9.033	7133138	54.994	VB
		12970743	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



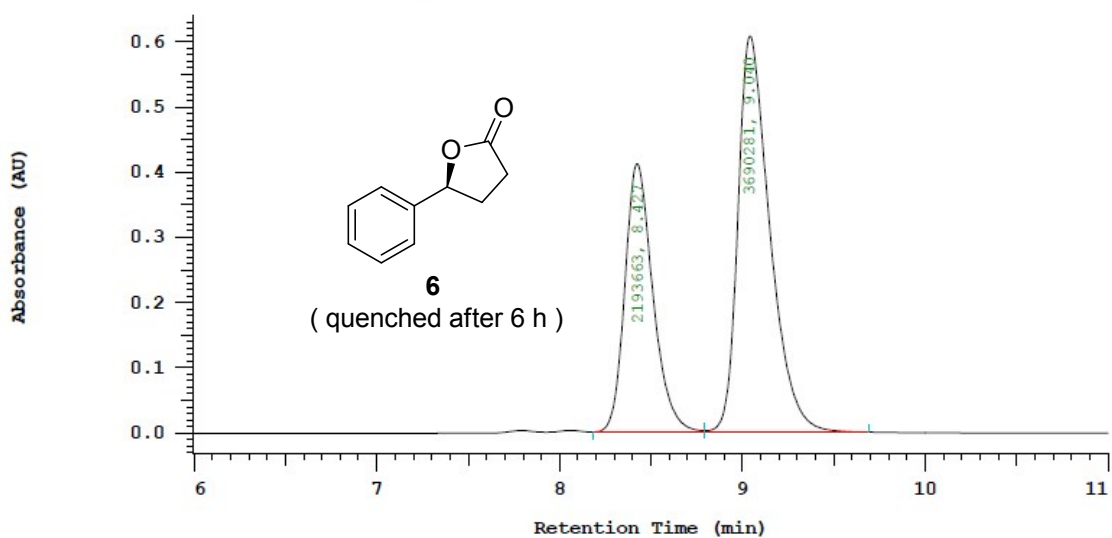
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.427	4417696	42.550	BV
2	9.027	5964599	57.450	VB
10382295			100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



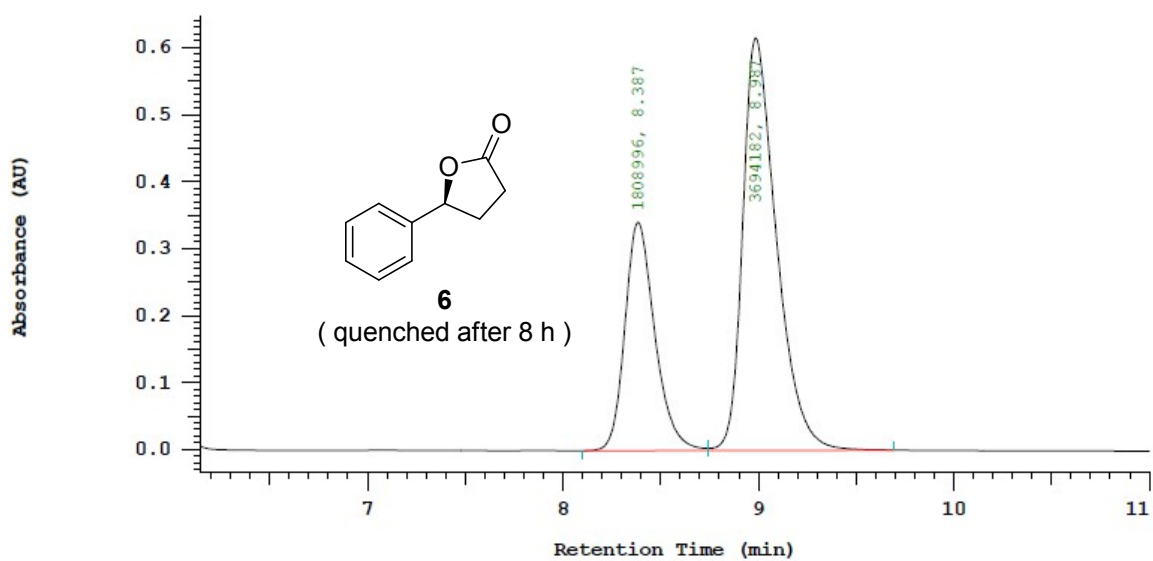
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.427	2193663	37.282	BV
2	9.040	3690281	62.718	VB
5883944			100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm

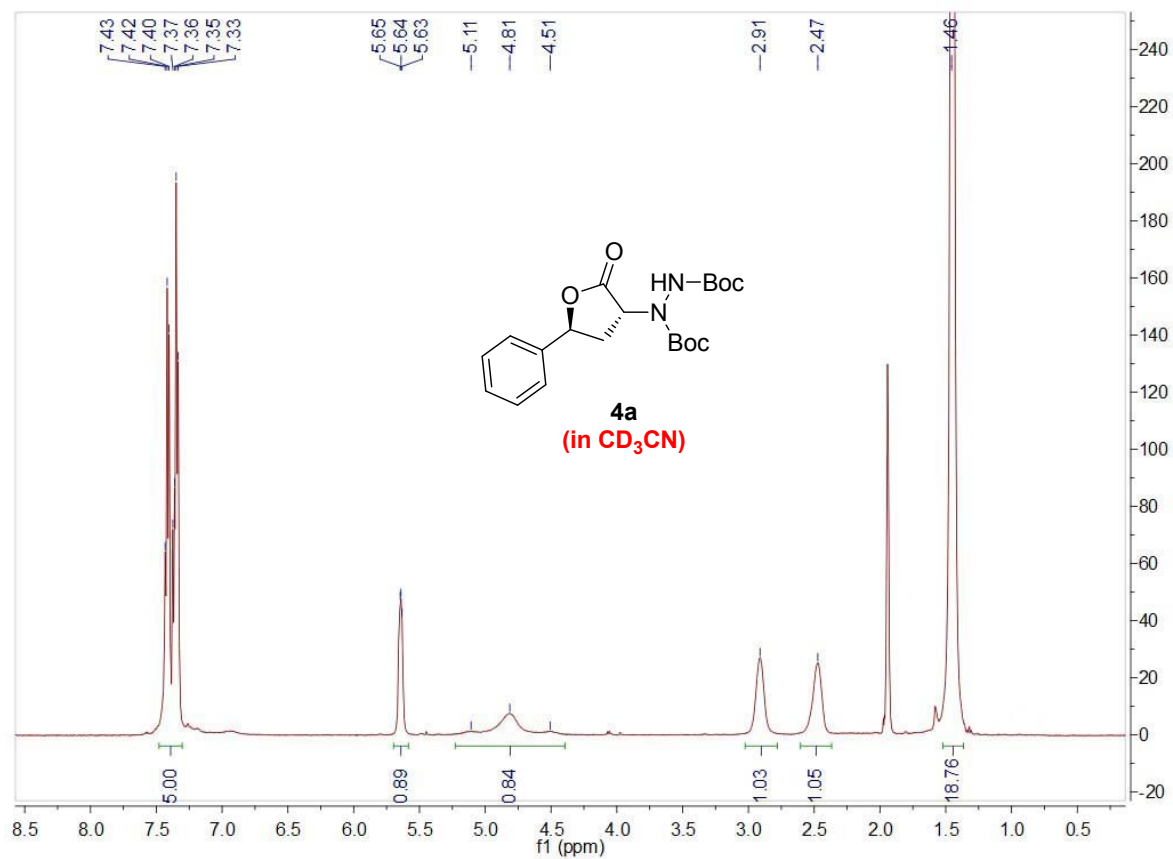
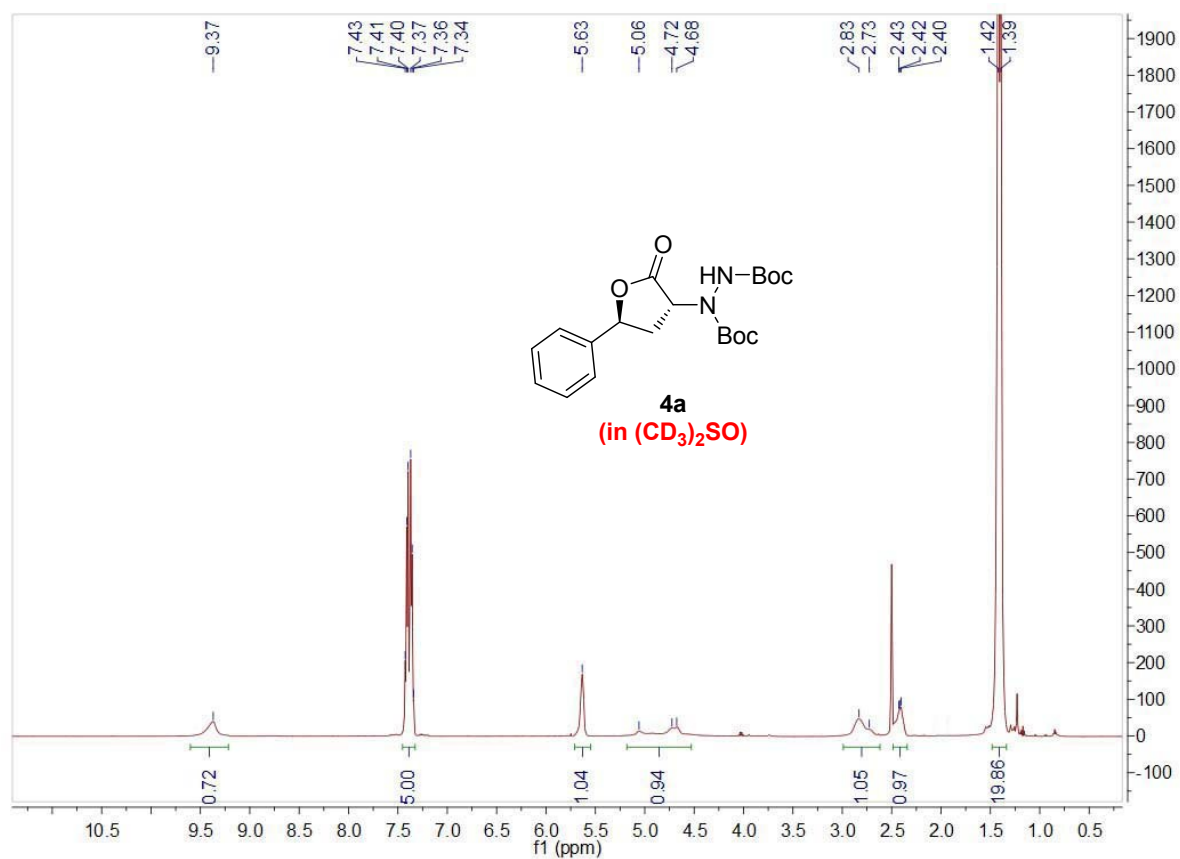
Peak Quantitation: AREA

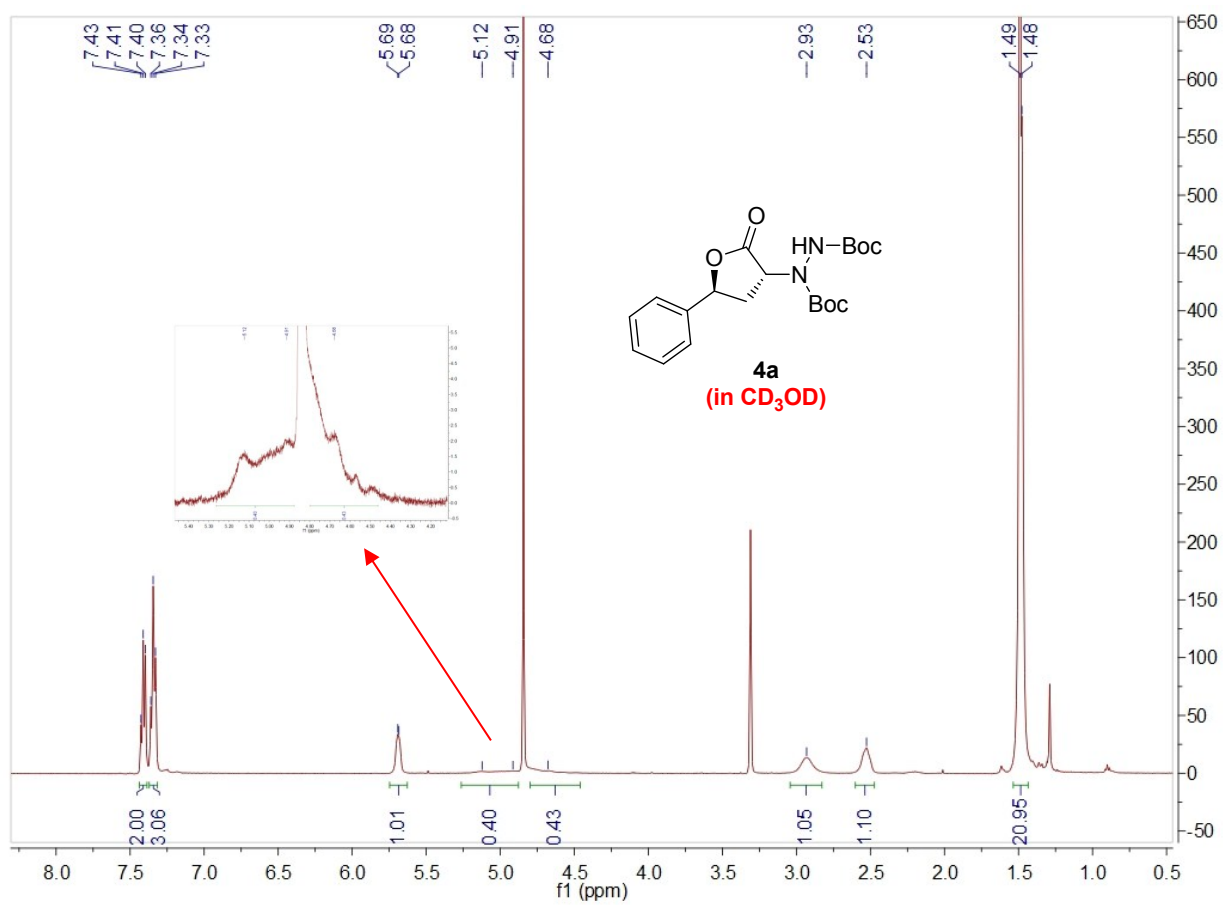
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.387	1808996	32.872	BV
2	8.987	3694182	67.128	VB
		5503178	100.000	

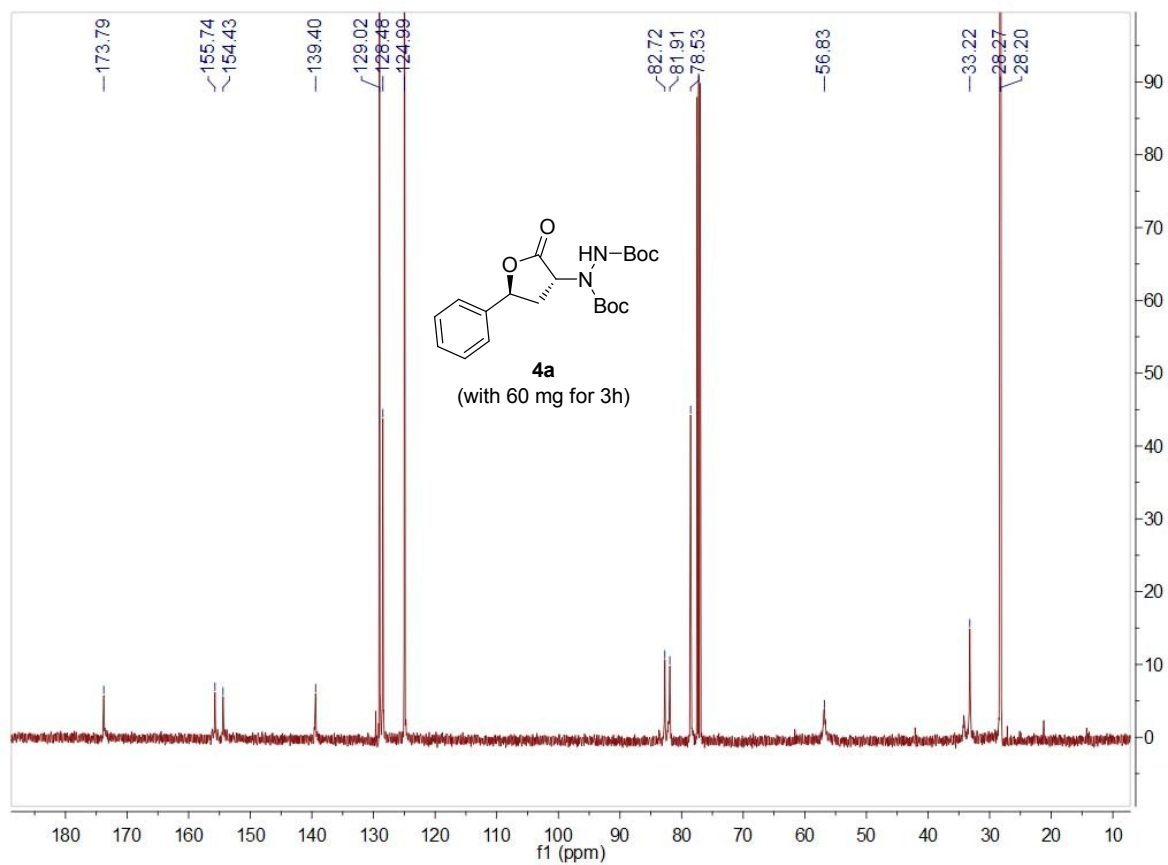
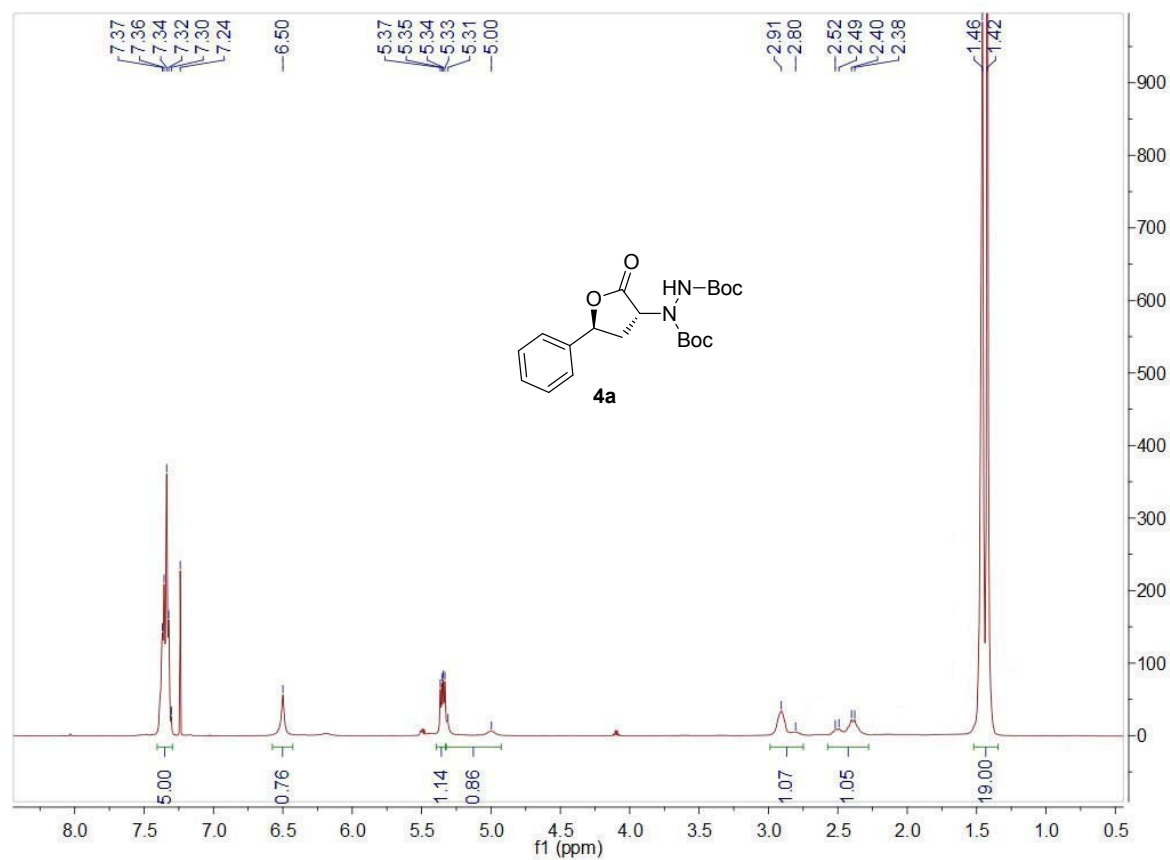
3. ^1H NMR spectra of 4a in other D-Substituted Solvents

As we found the splitting in the ^1H NMR spectra of **4** and **5** was not showing well when CDCl_3 was used as the solvent. We then tried other D-substituted solvents, such as $(\text{CD}_3)_2\text{SO}$ @ 2.50 ppm ^1H NMR, CD_3CN @ 1.94 ppm ^1H NMR, $(\text{CD}_3)_2\text{CO}$ @ 2.05 ppm ^1H NMR, CD_3OD @ 3.31 ppm ^1H NMR, however, the similar results were observed.

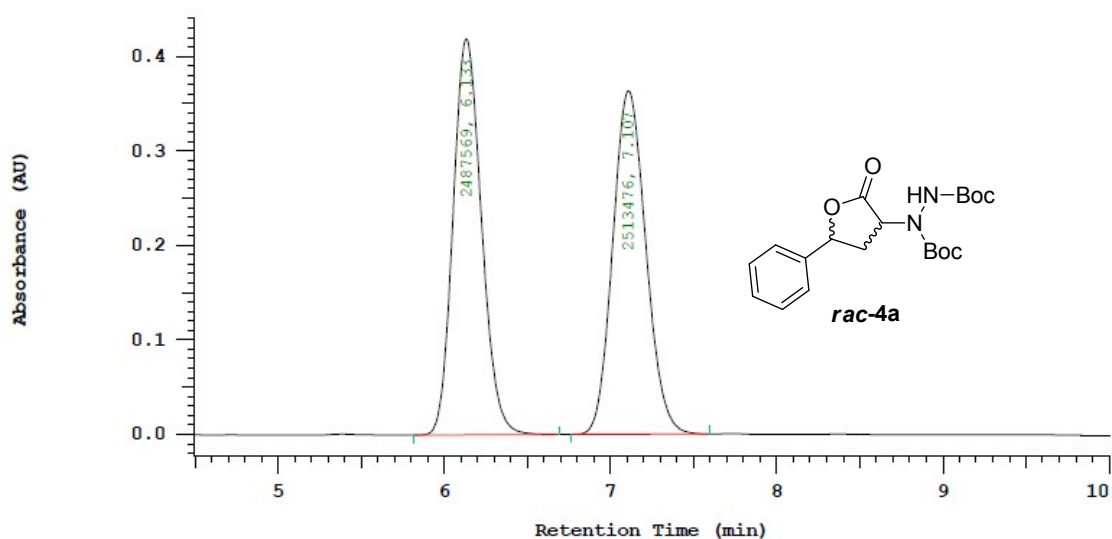




4. NMR spectra and HPLC Traces



Chrom Type: Fixed WL Chromatogram, 210 nm



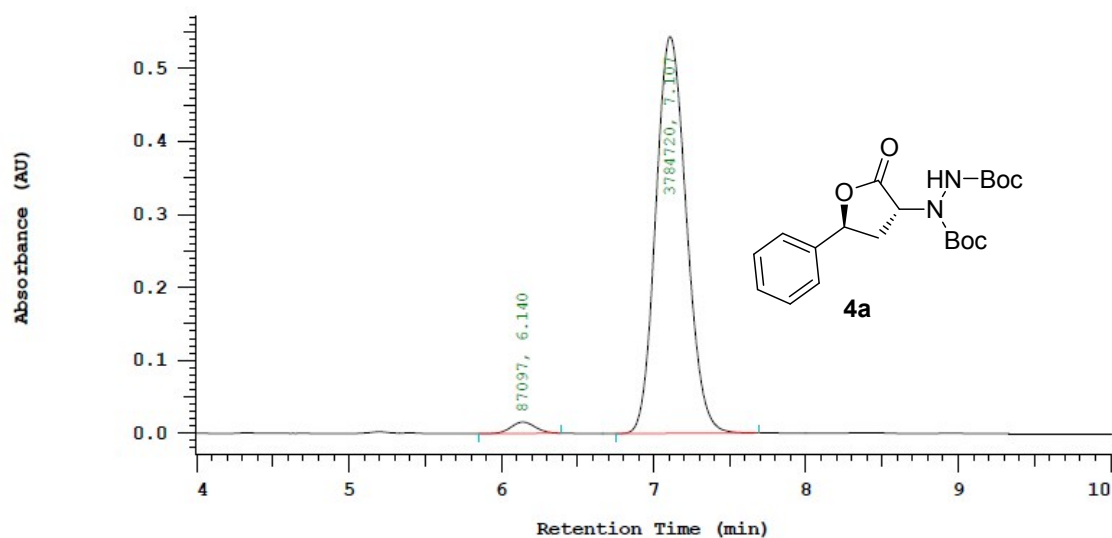
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.133	2487569	49.741	BB
2	7.107	2513476	50.259	BB
5001045			100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



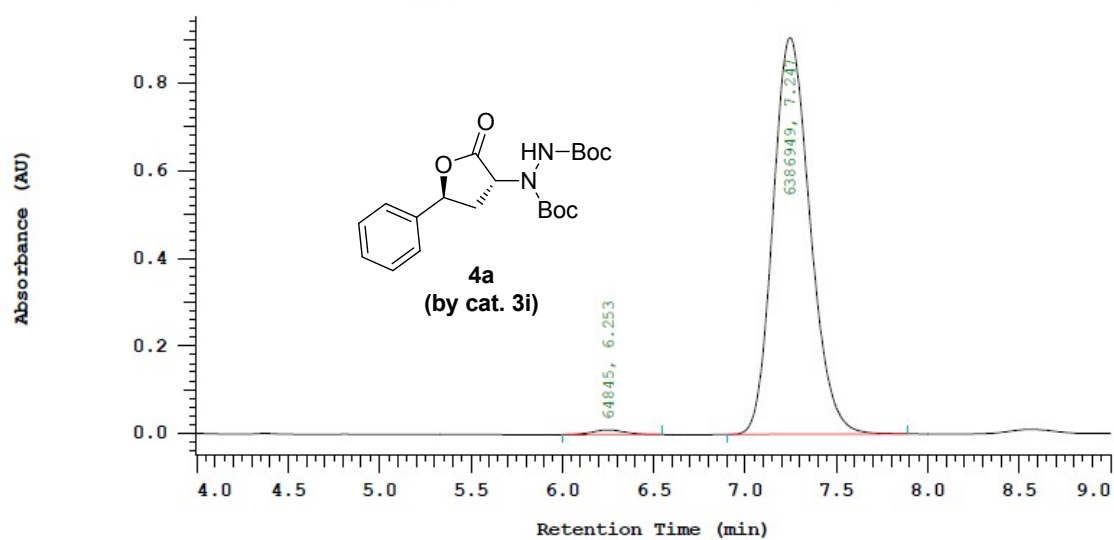
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.140	87097	2.250	BB
2	7.107	3784720	97.750	BB
3871817			100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

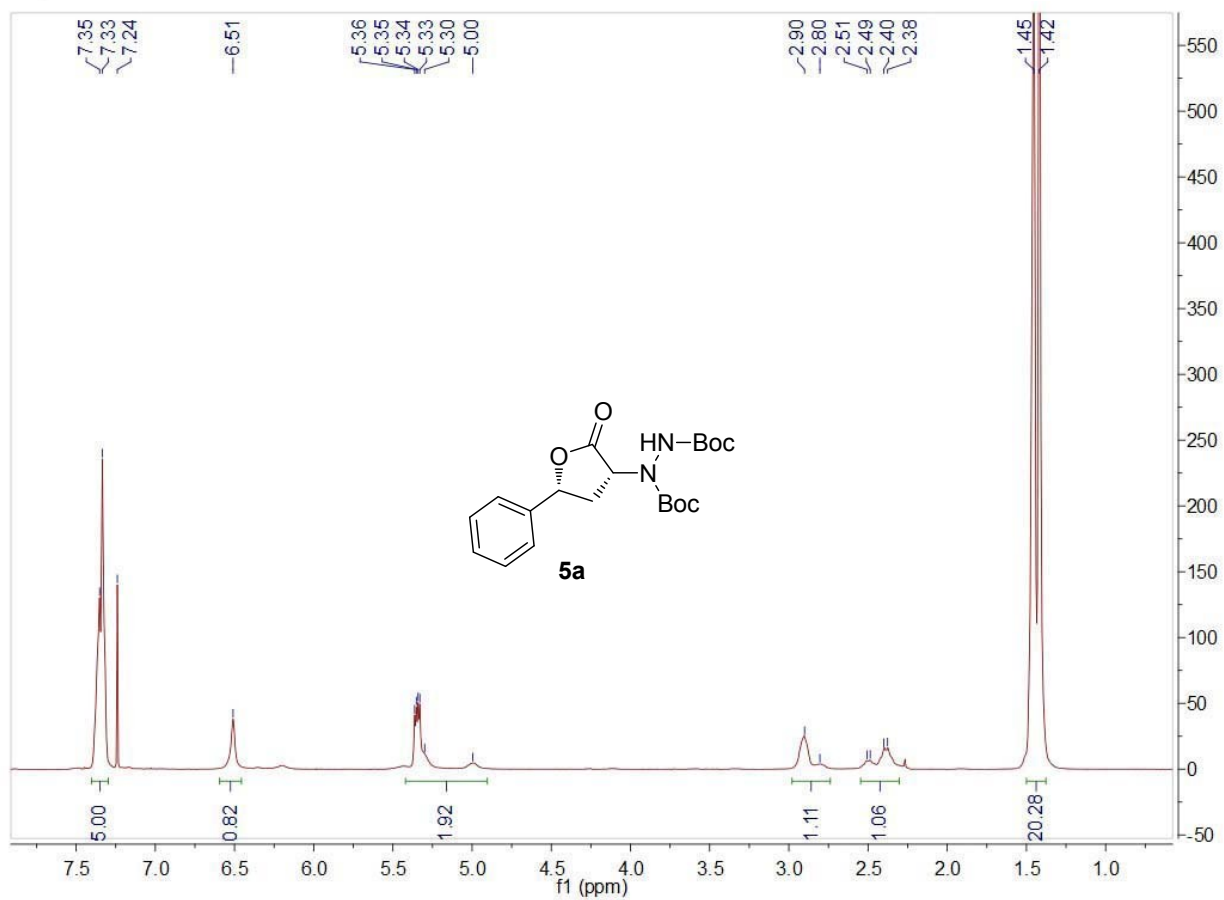


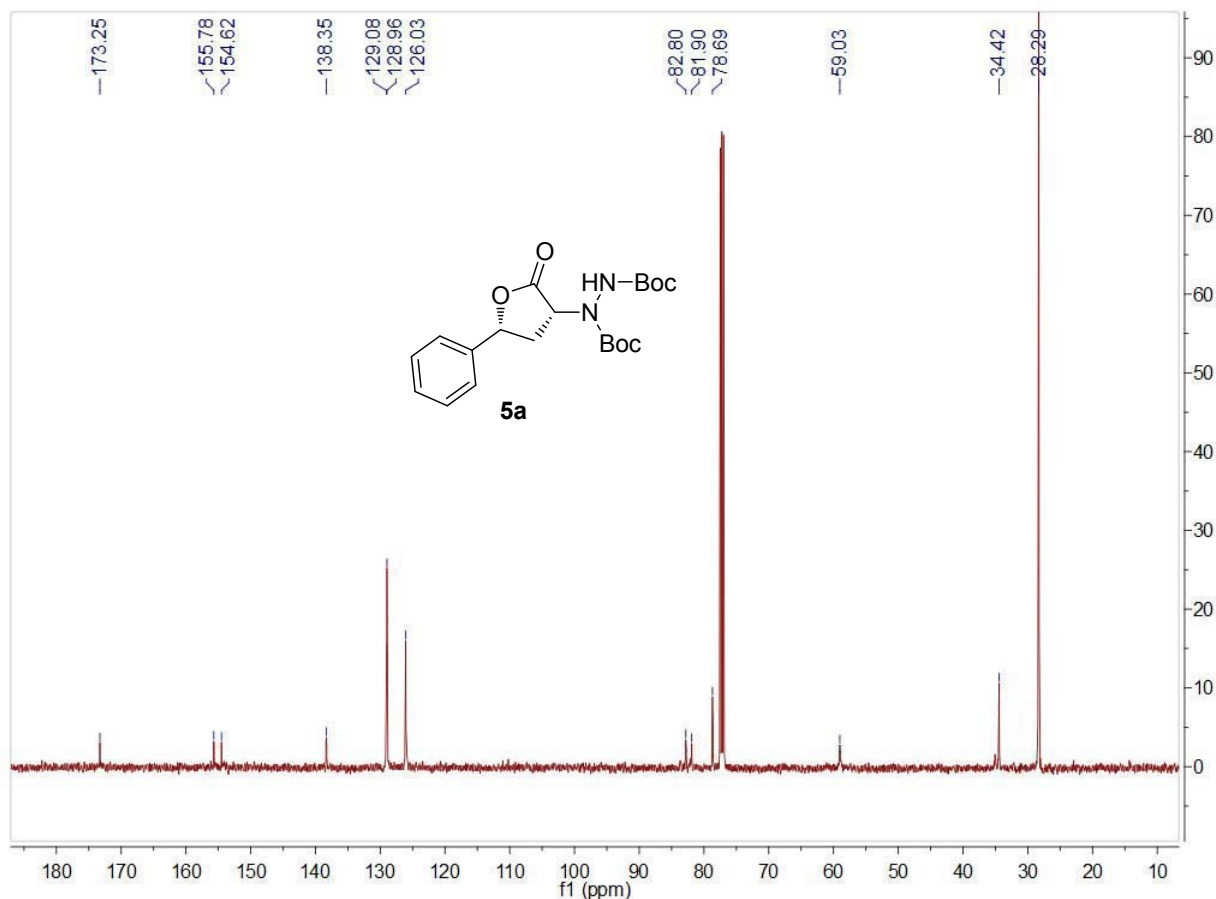
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

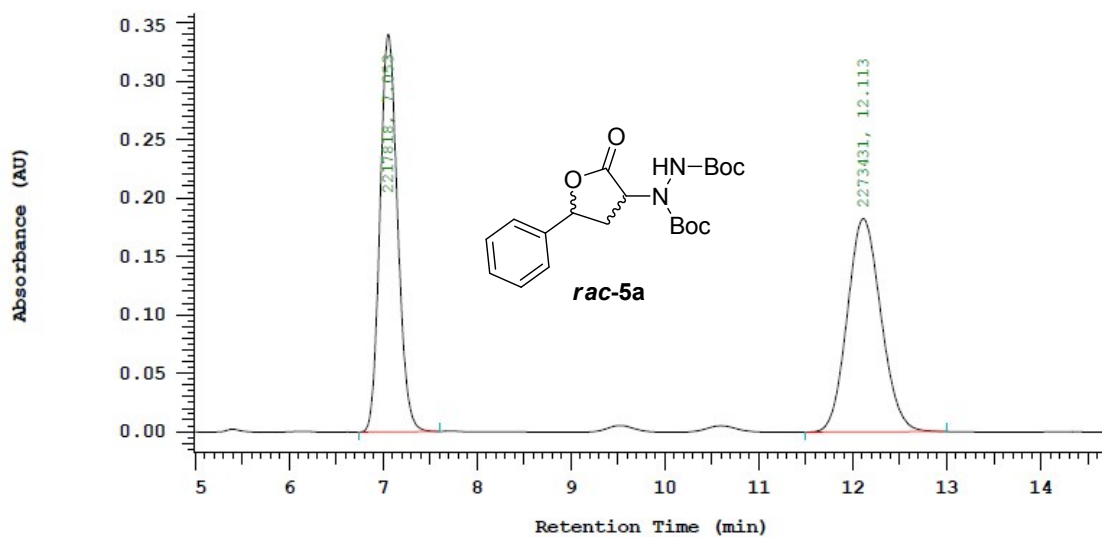
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.253	64845	1.005	BB
2	7.247	6386949	98.995	BB
		6451794	100.000	





Chrom Type: Fixed WL Chromatogram, 210 nm

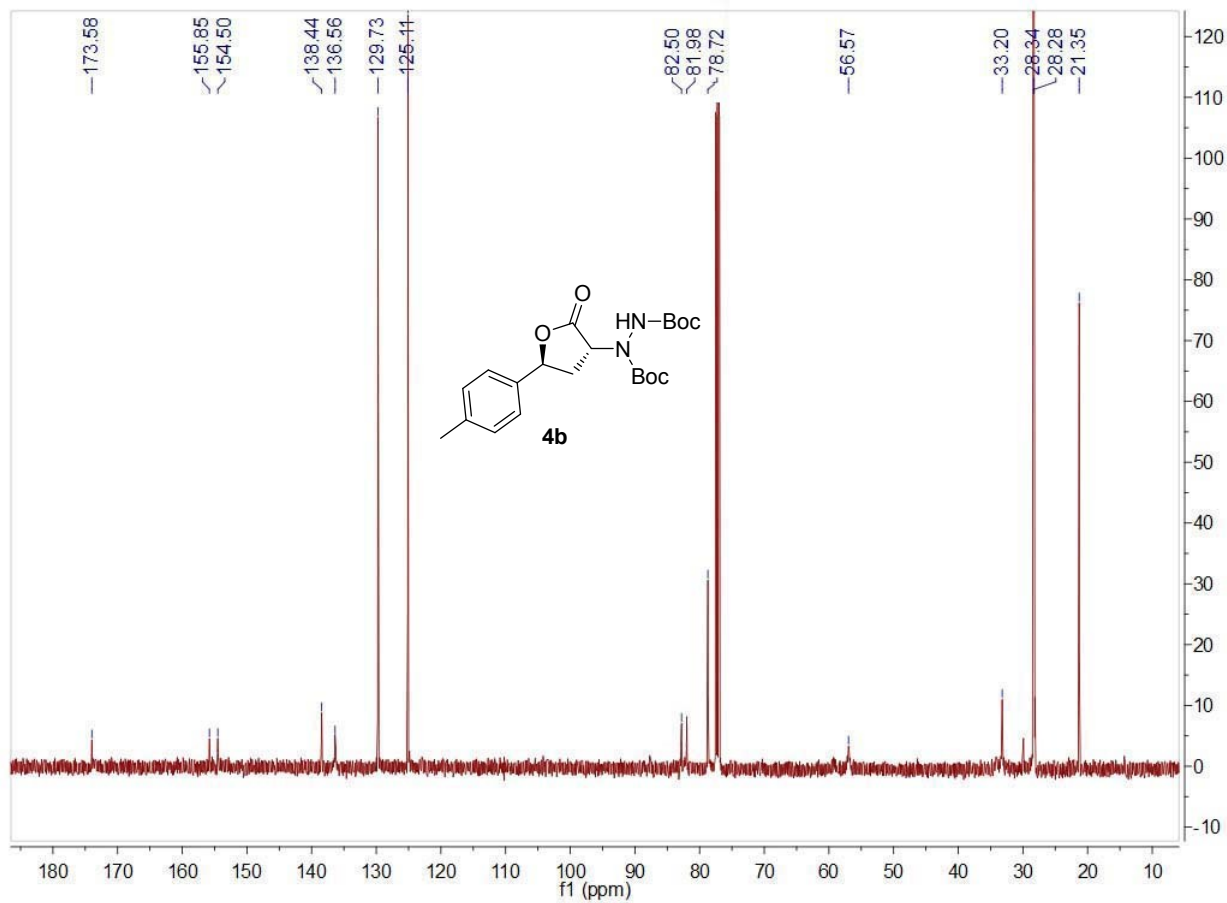
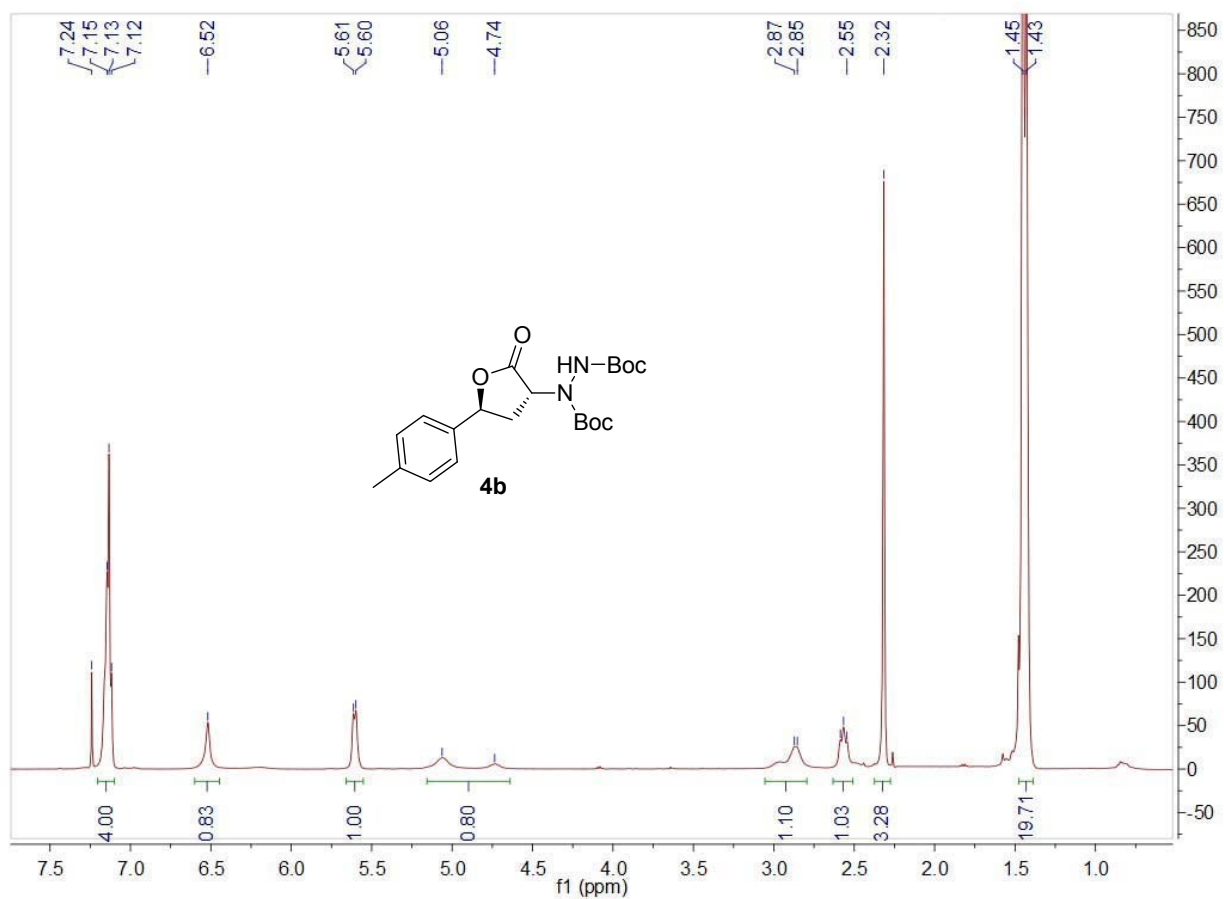


Chrom Type: Fixed WL Chromatogram, 210 nm

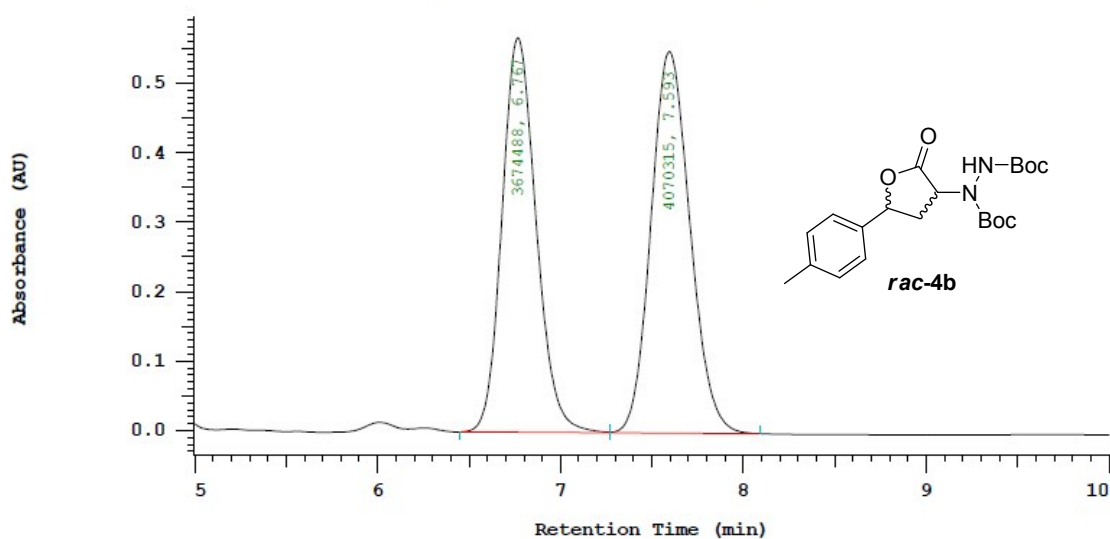
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.053	2217818	49.381	BB
2	12.113	2273431	50.619	BB
		4491249	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



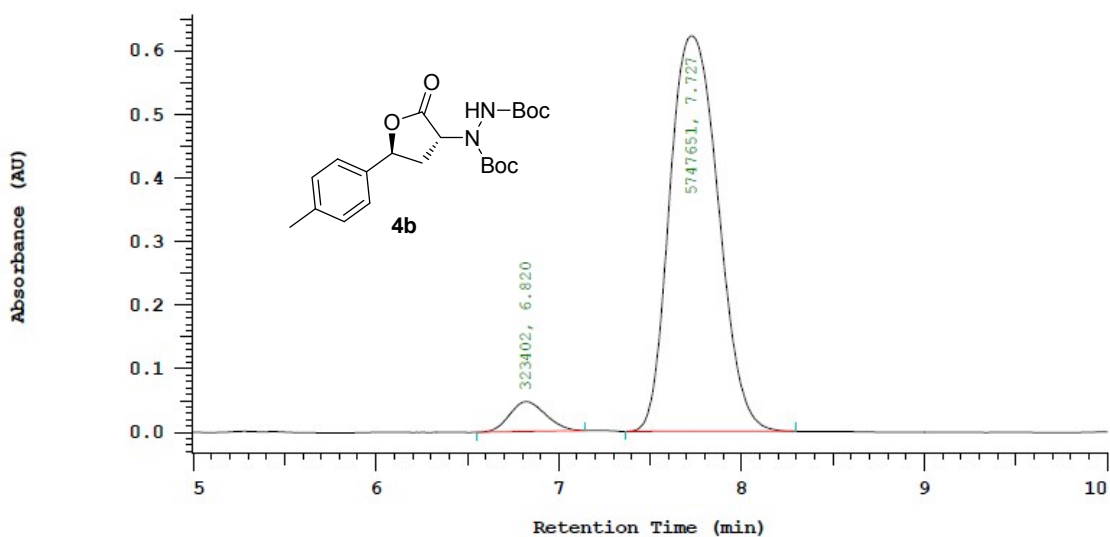
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.767	3674488	47.445	BV
2	7.593	4070315	52.555	VB
		7744803	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



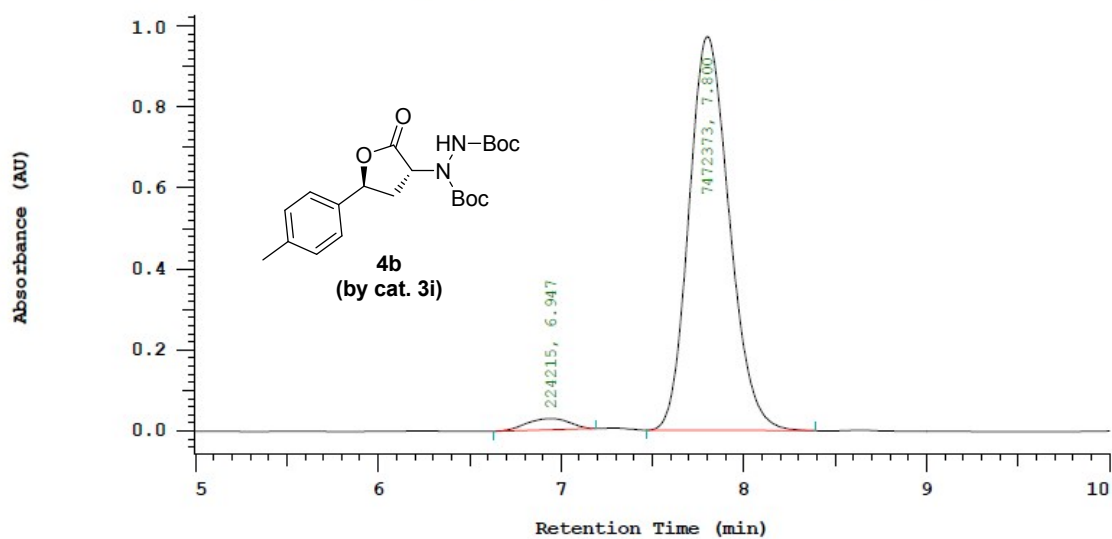
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.820	323402	5.327	BB
2	7.727	5747651	94.673	BB
		6071053	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

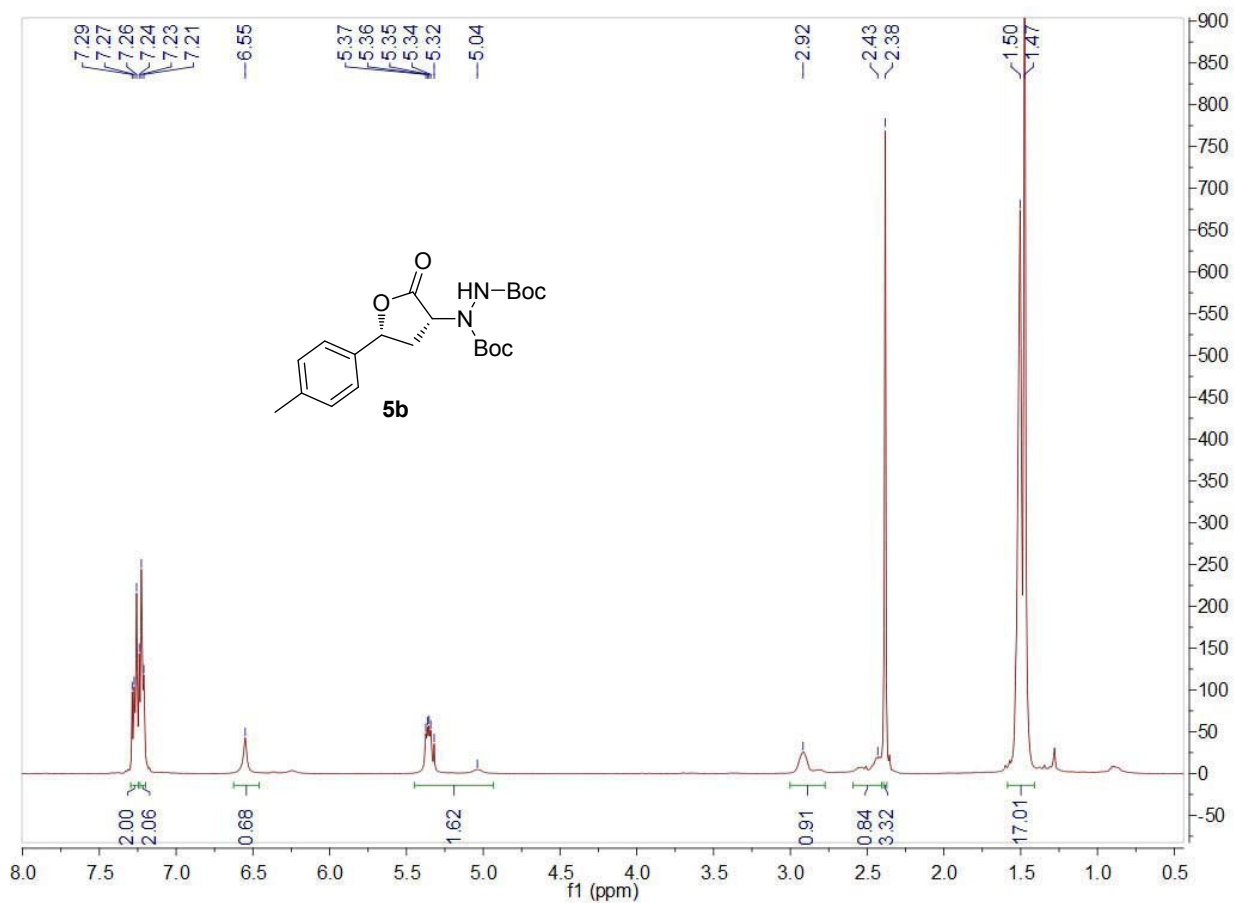


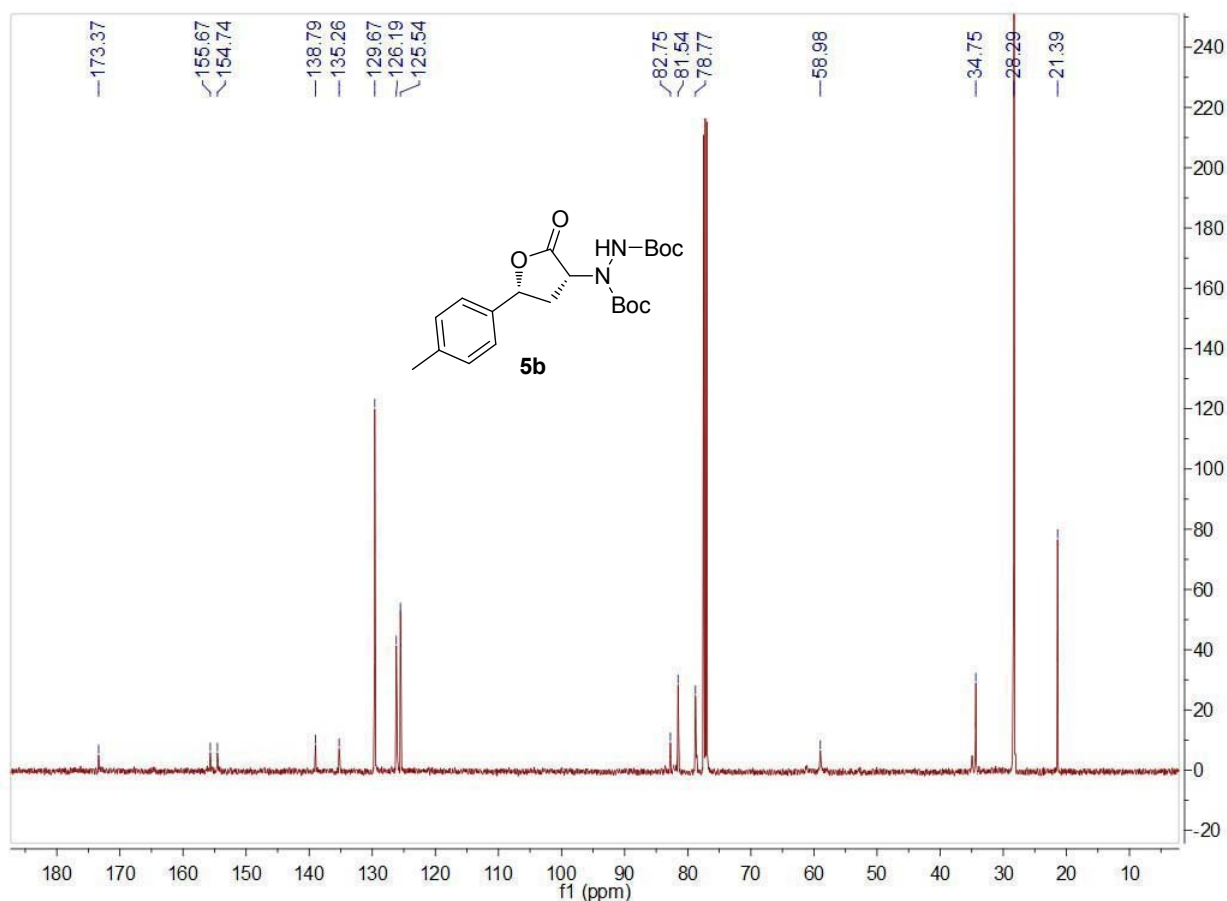
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

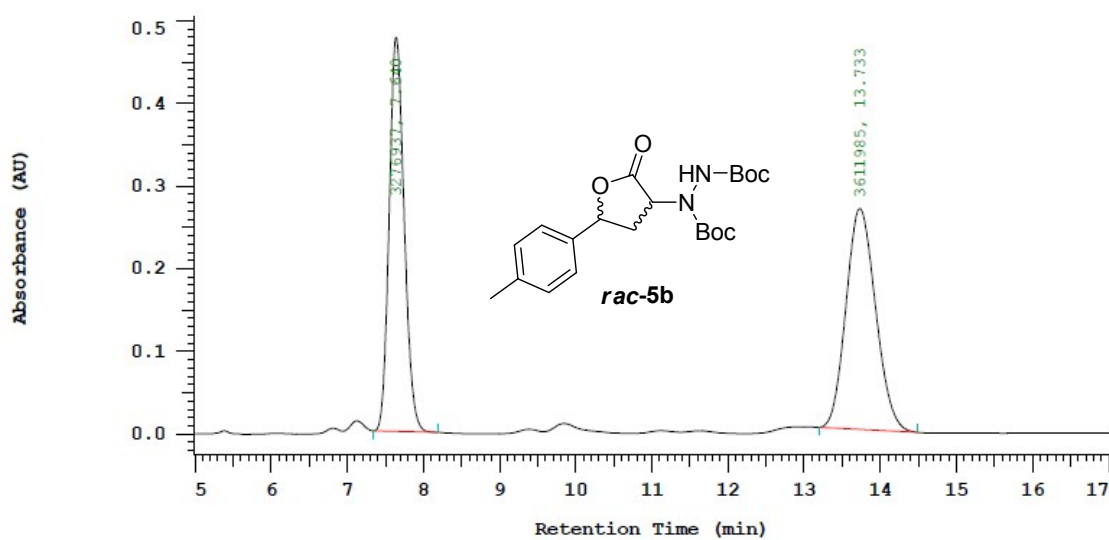
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.947	224215	2.913	BB
2	7.800	7472373	97.087	BB
		7696588	100.000	





Chrom Type: Fixed WL Chromatogram, 210 nm

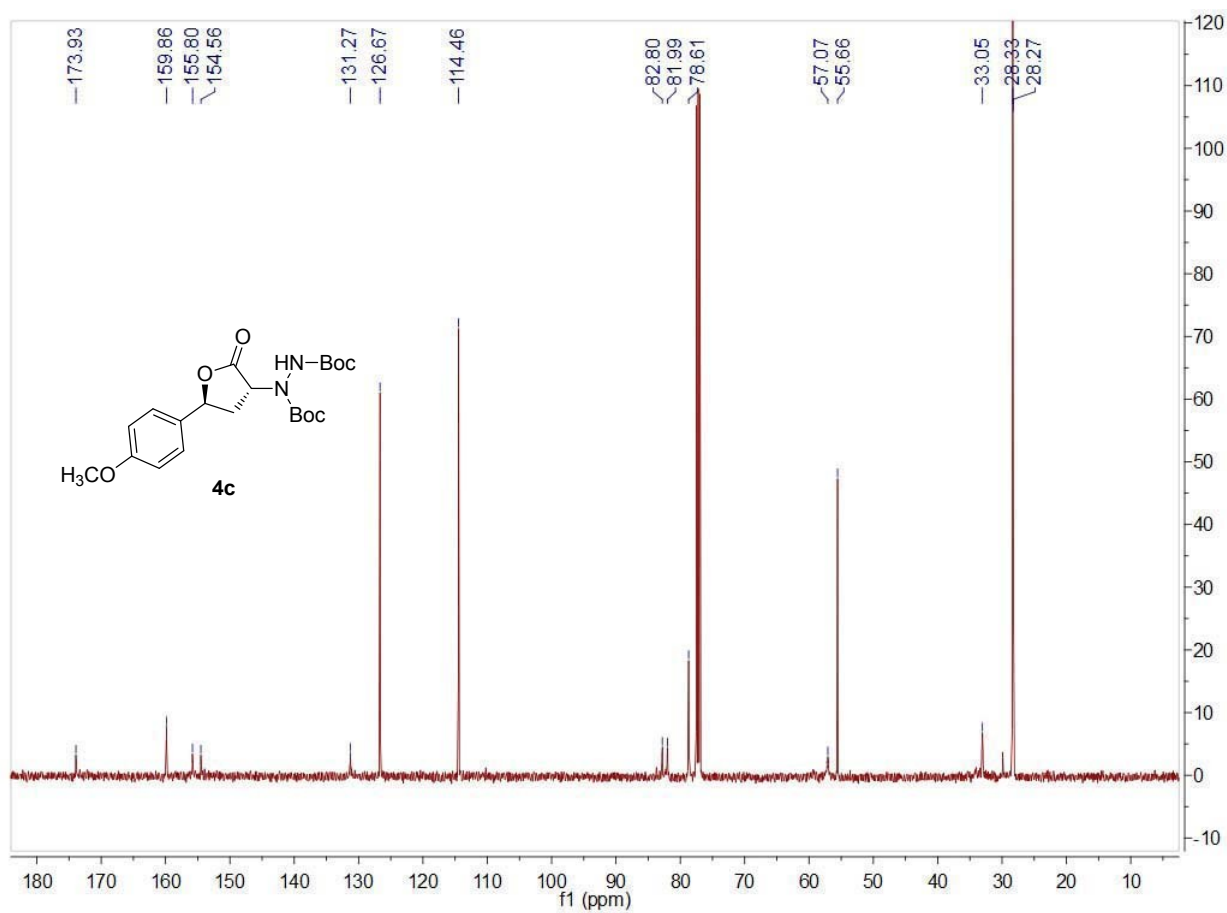


Chrom Type: Fixed WL Chromatogram, 210 nm

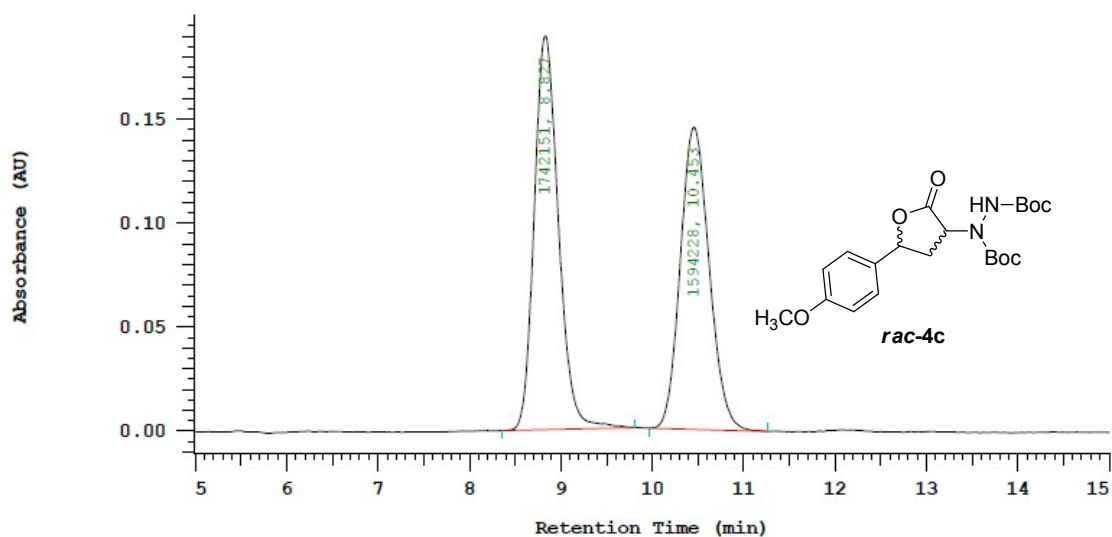
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.640	3276937	47.568	BB
2	13.733	3611985	52.432	BB
		6888922	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



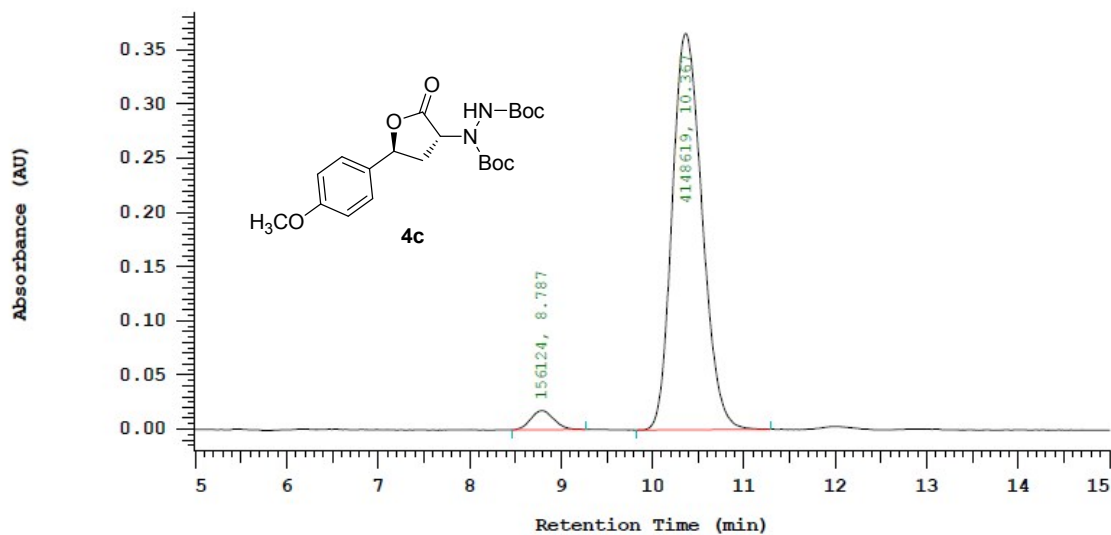
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.827	1742151	52.217	BB
2	10.453	1594228	47.783	BB
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Chrom Type: Fixed WL Chromatogram, 210 nm

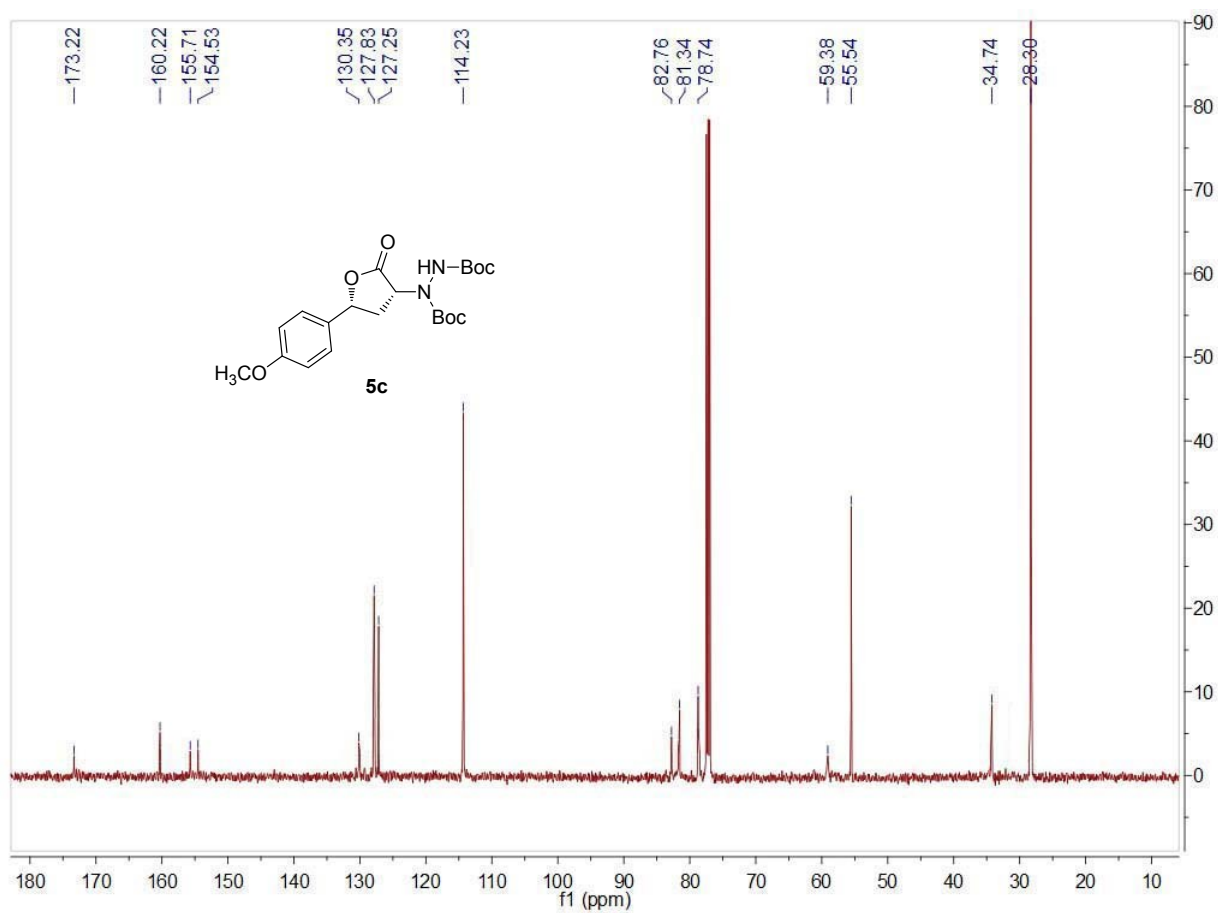
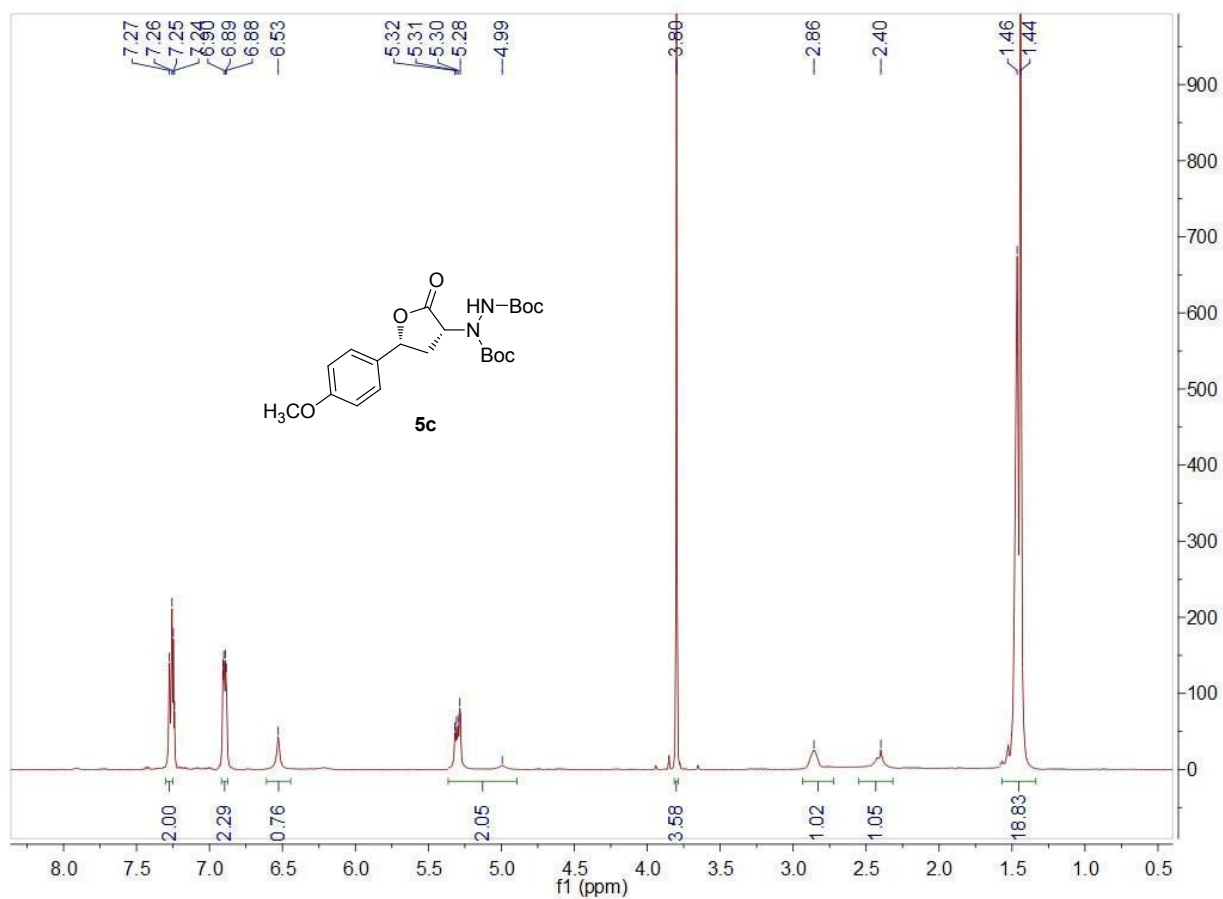


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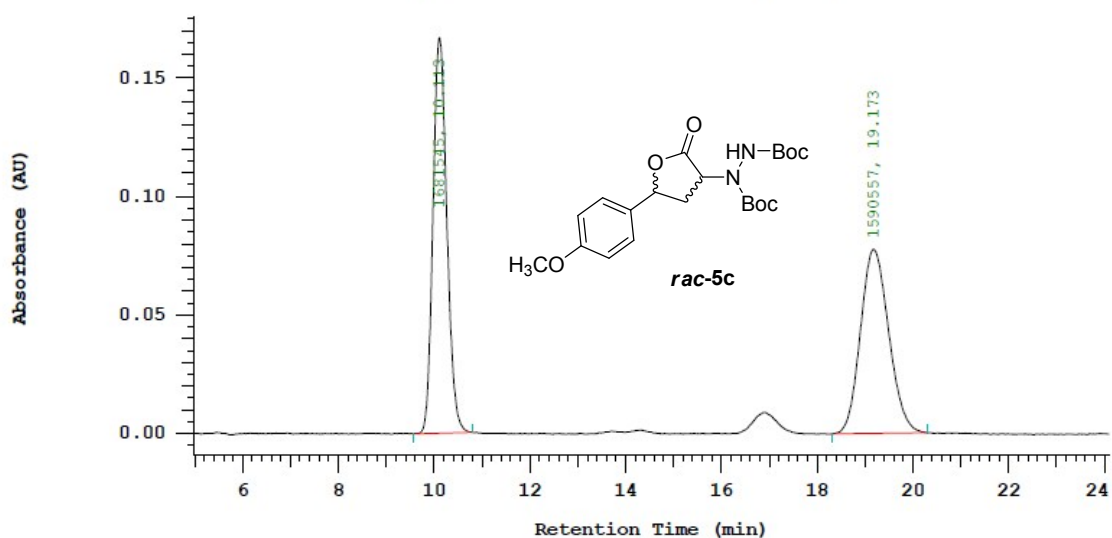
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.787	156124	3.627	BB
2	10.367	4148619	96.373	BB
		4304743	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm

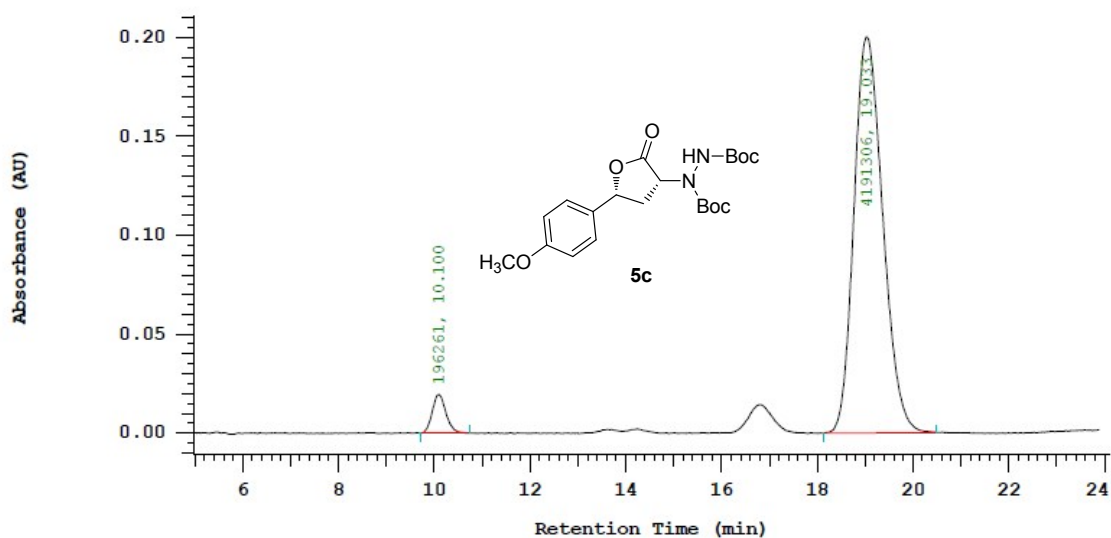


Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	10.113	1681545	51.390	BB
2	19.173	1590557	48.610	BB
		3272102	100.000	

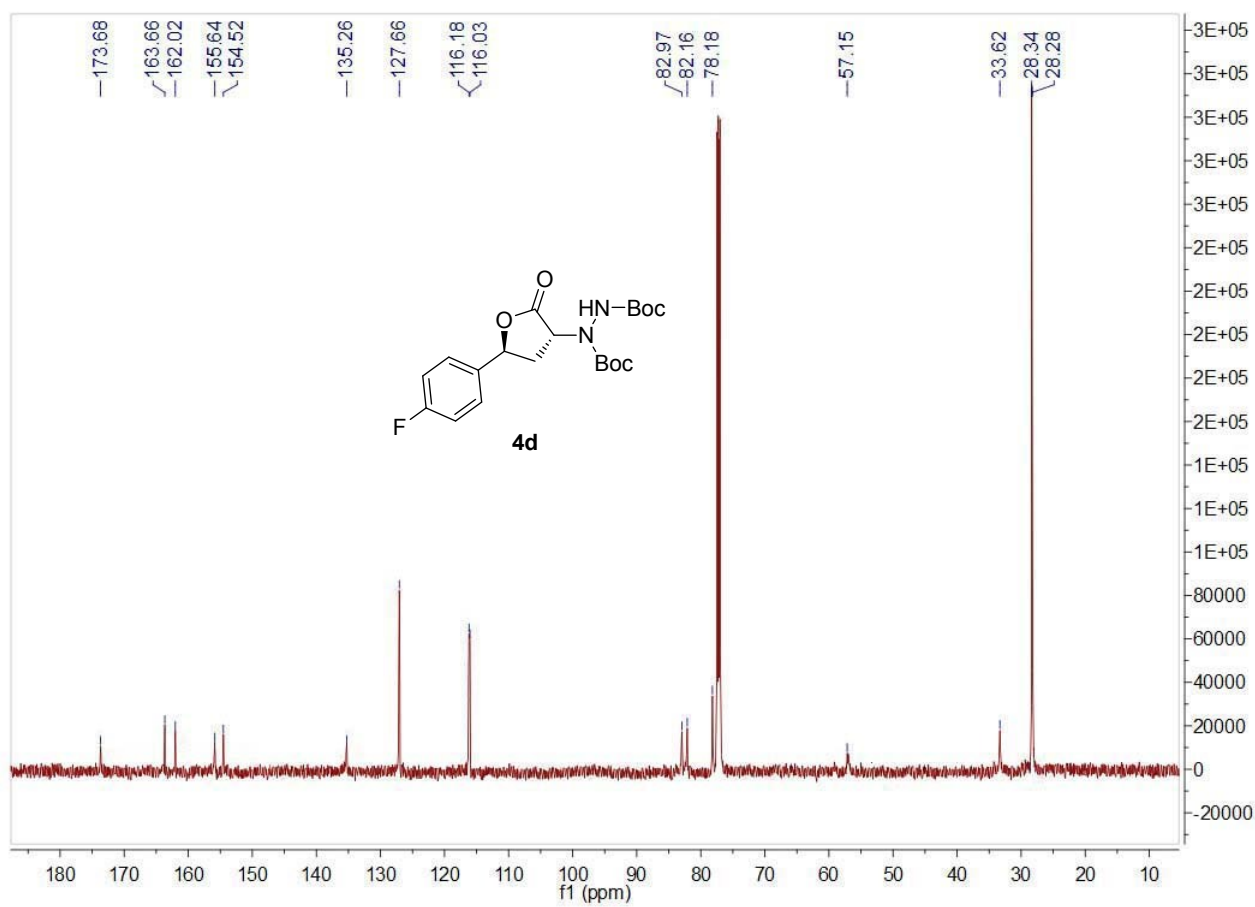
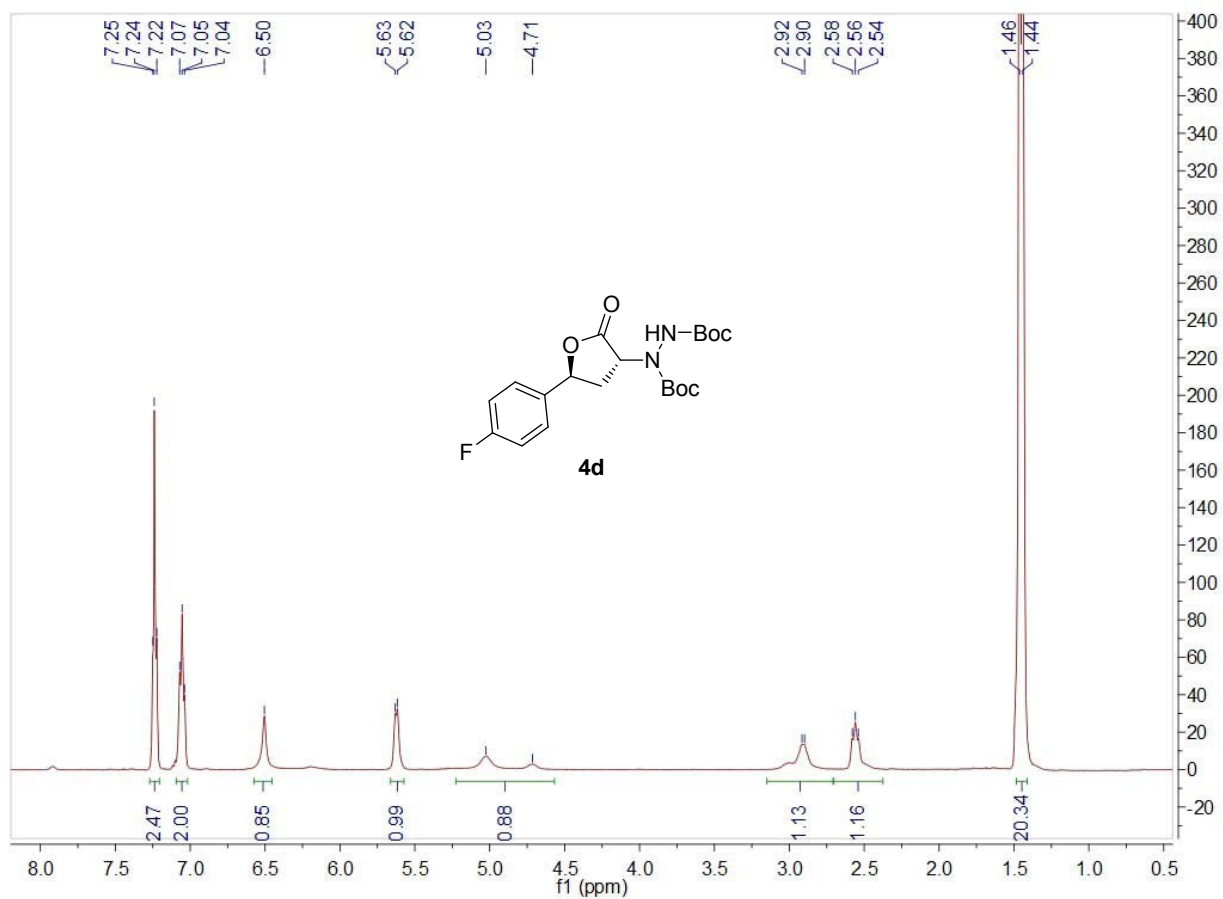
Chrom Type: Fixed WL Chromatogram, 210 nm



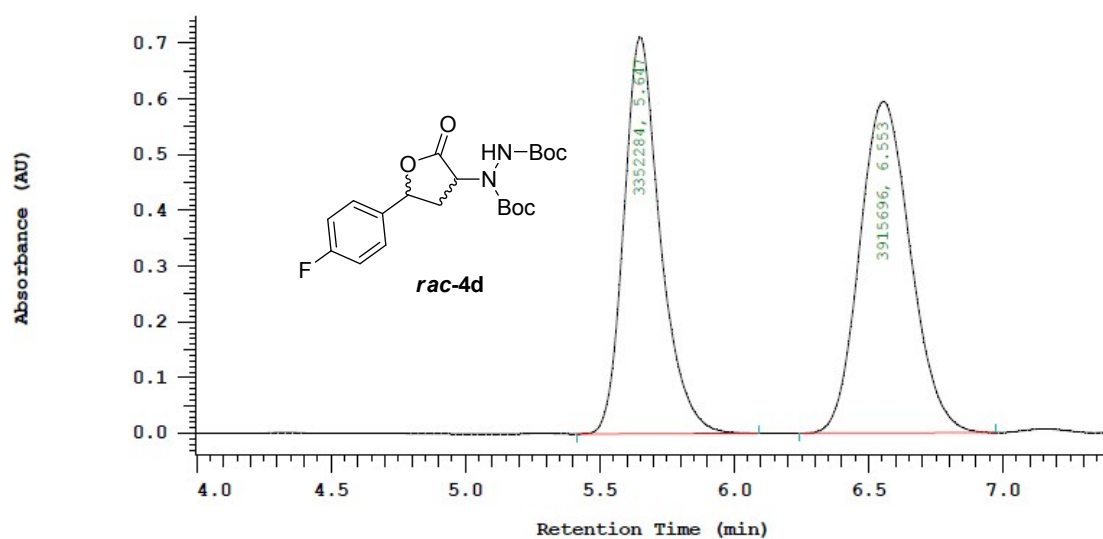
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	10.100	196261	4.473	BB
2	19.033	4191306	95.527	BB
		4387567	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



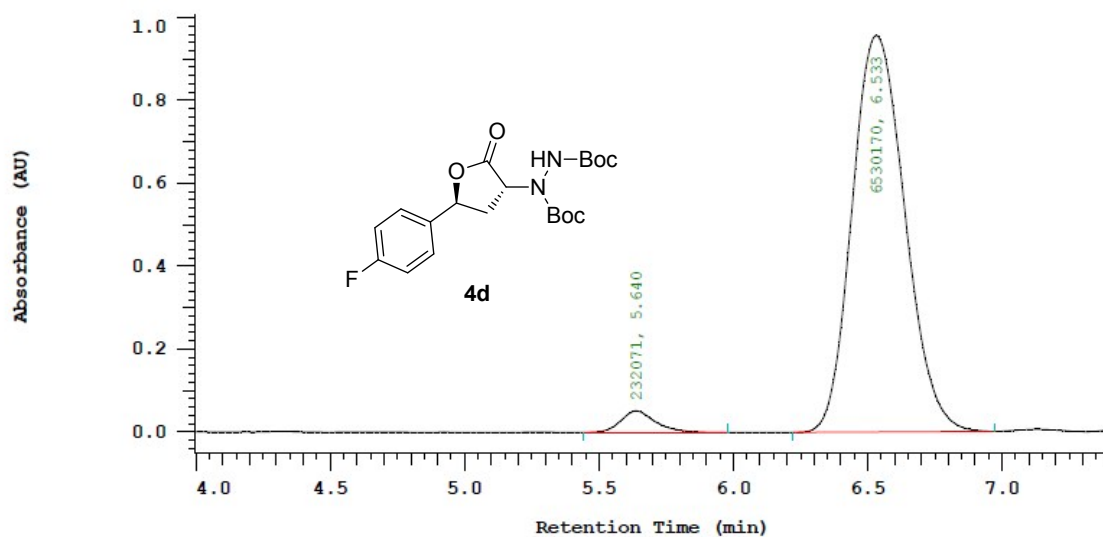
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	5.647	3352284	46.124	BB
2	6.553	3915696	53.876	BB
		7267980	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

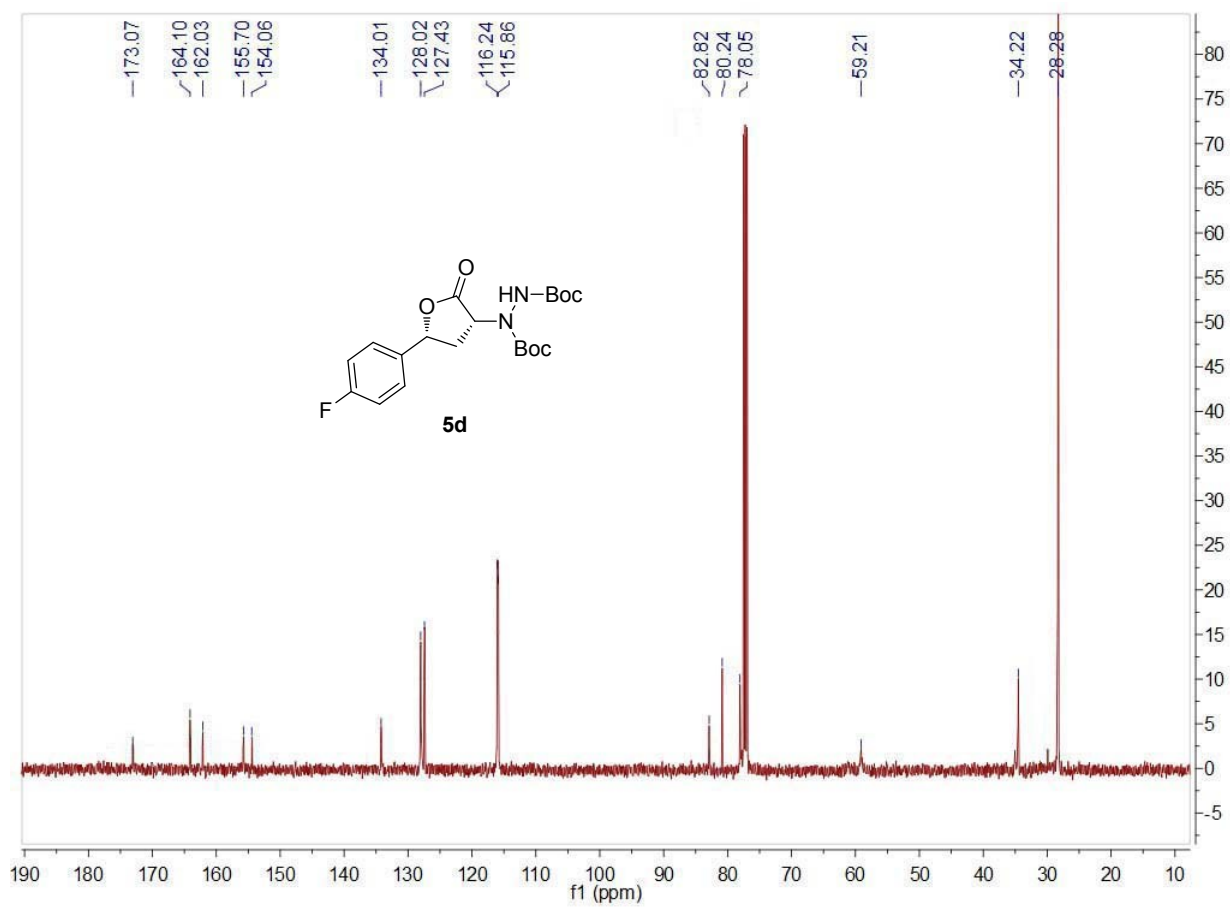
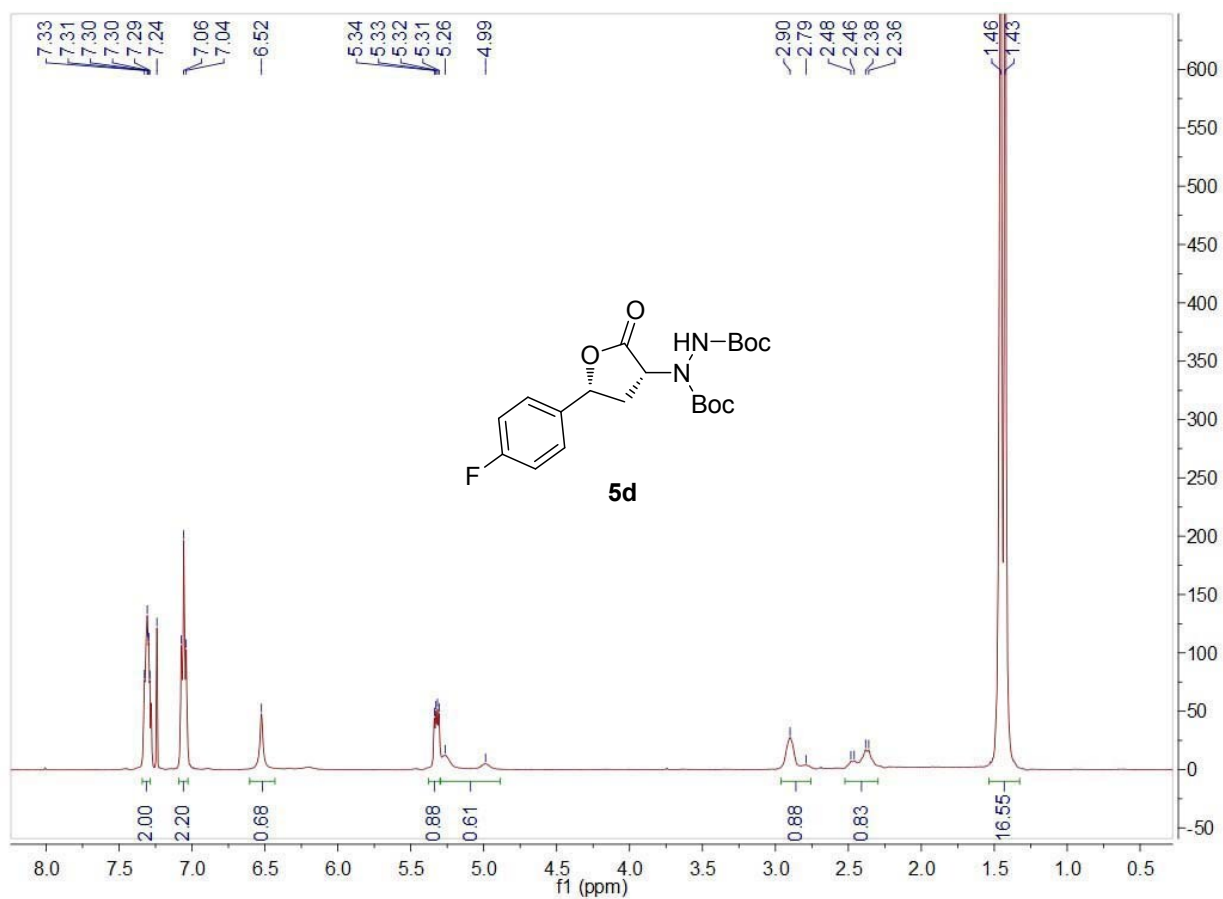


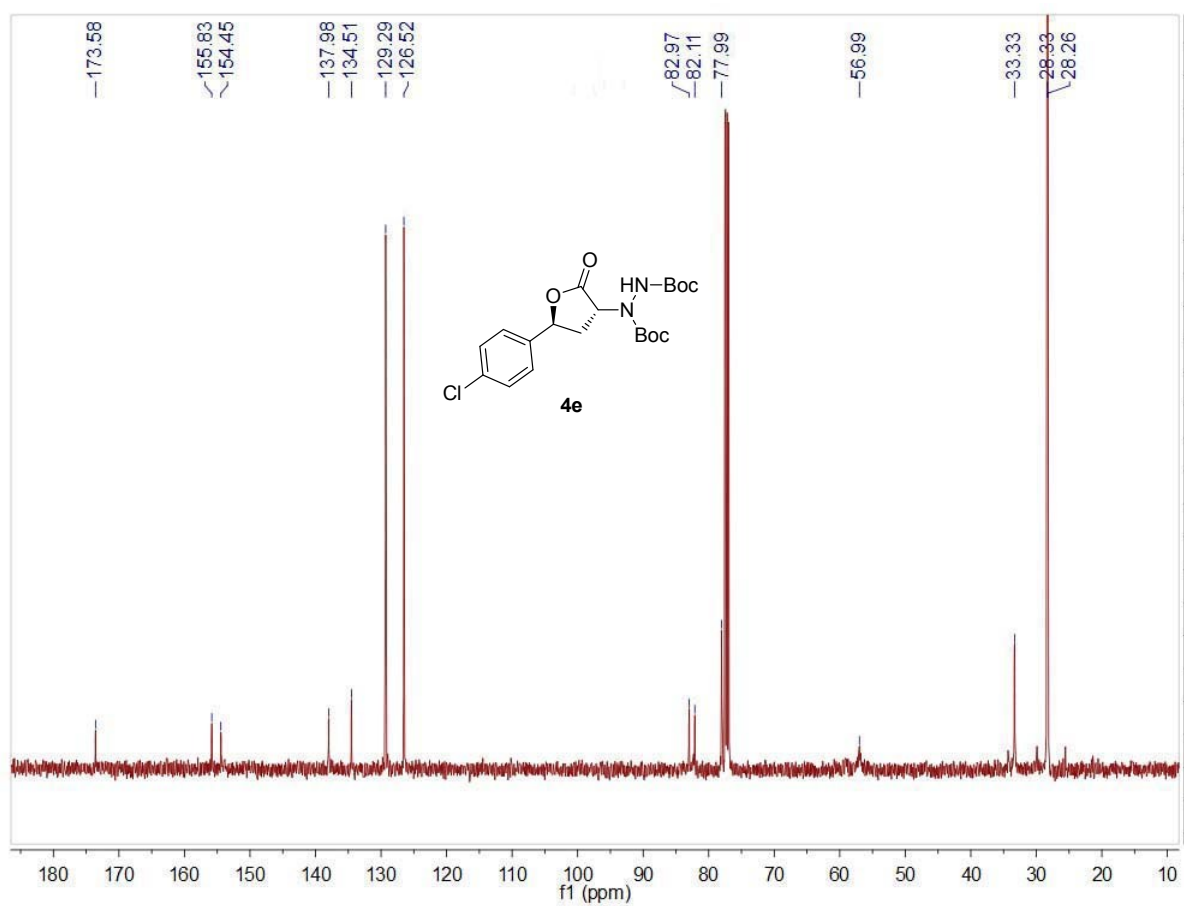
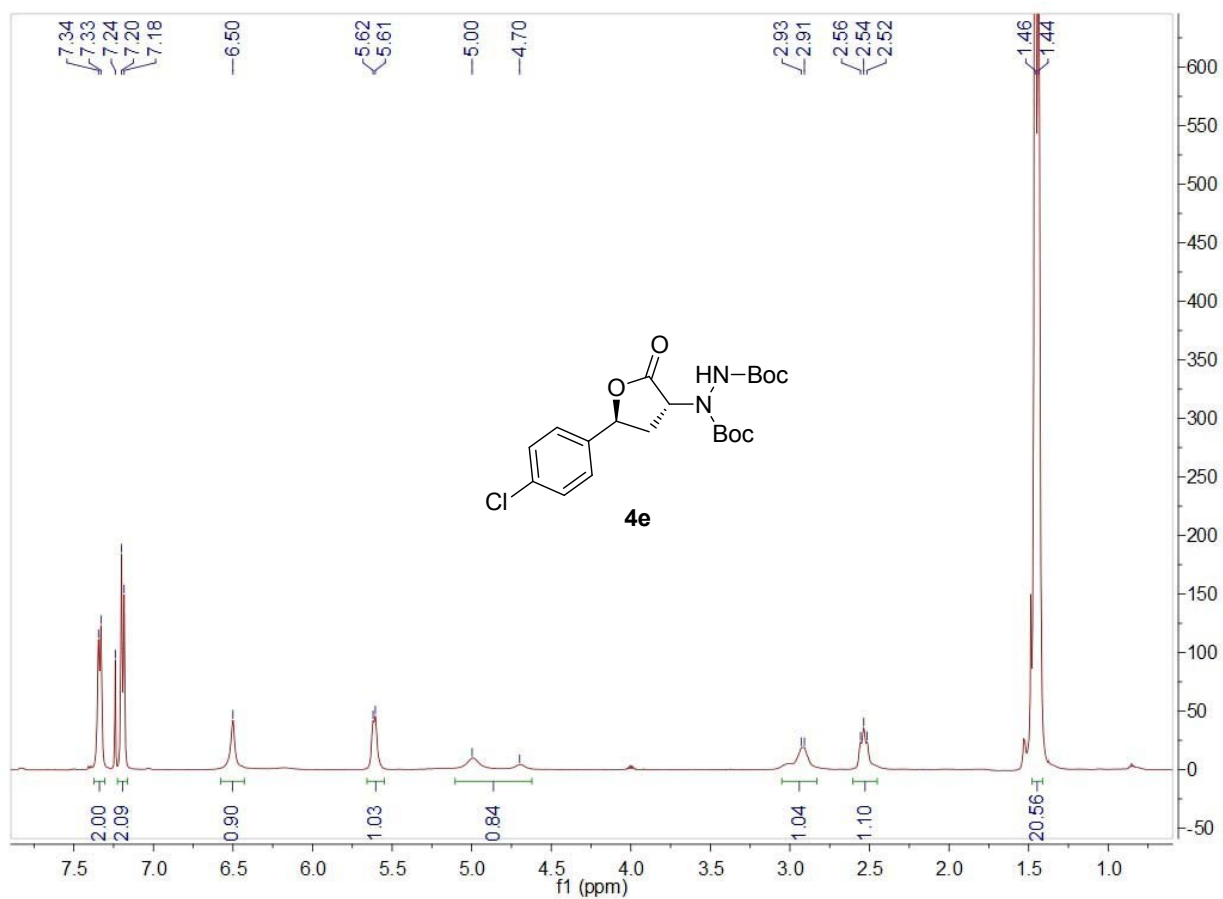
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

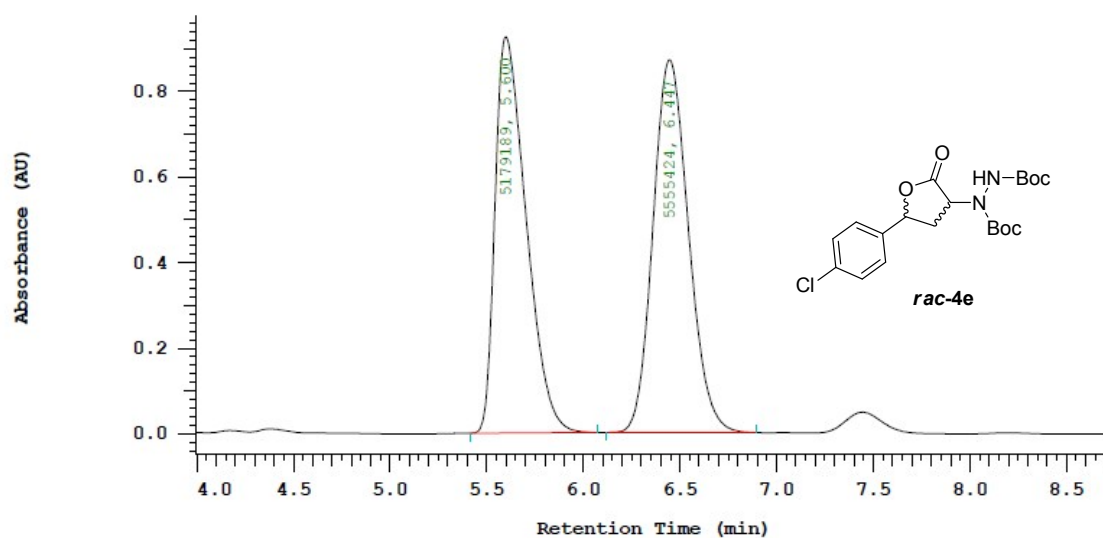
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	5.640	232071	3.432	BB
2	6.533	6530170	96.568	BB
		6762241	100.000	





Chrom Type: Fixed WL Chromatogram, 210 nm



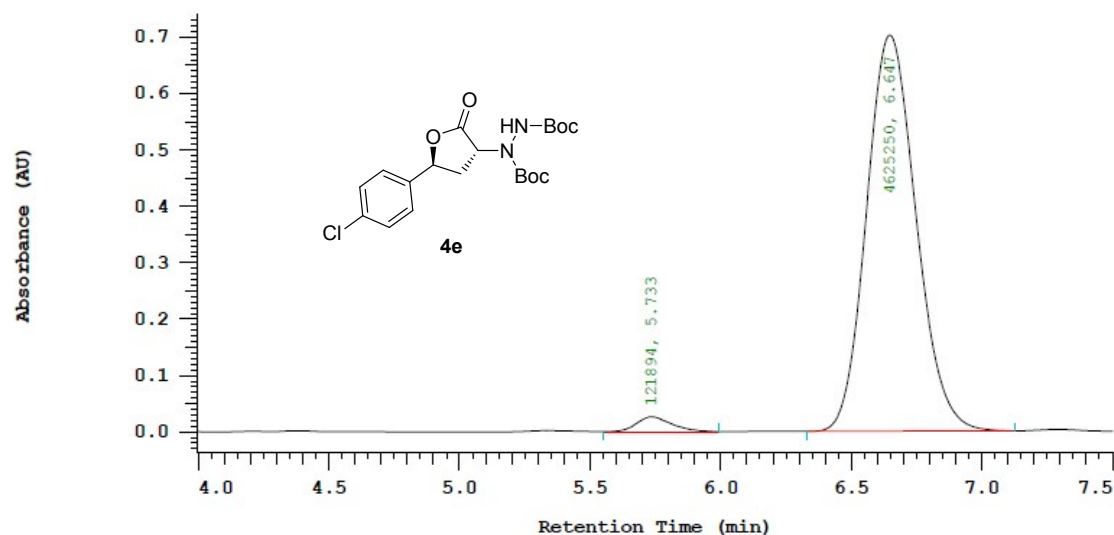
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	5.600	5179189	48.248	BB
2	6.447	5555424	51.752	BB
10734613			100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

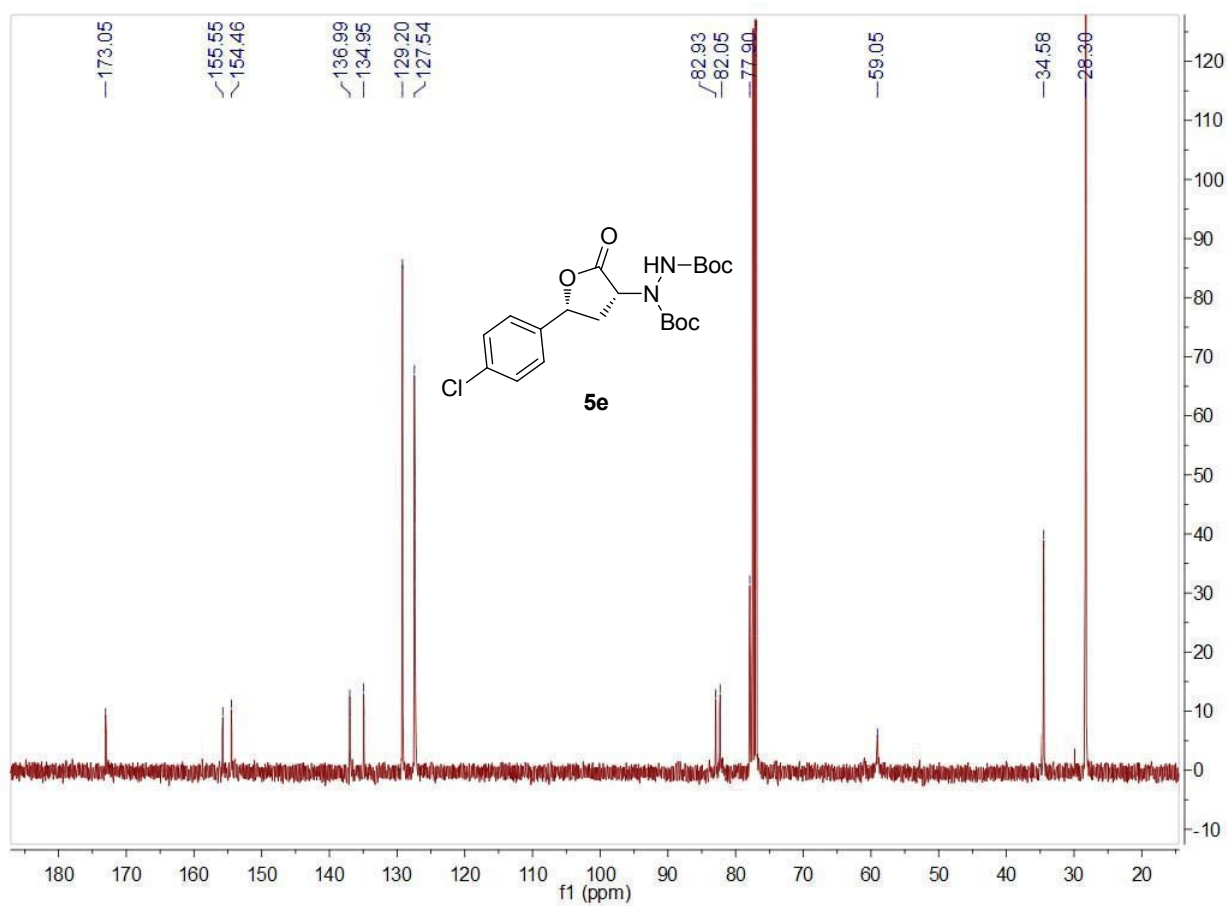
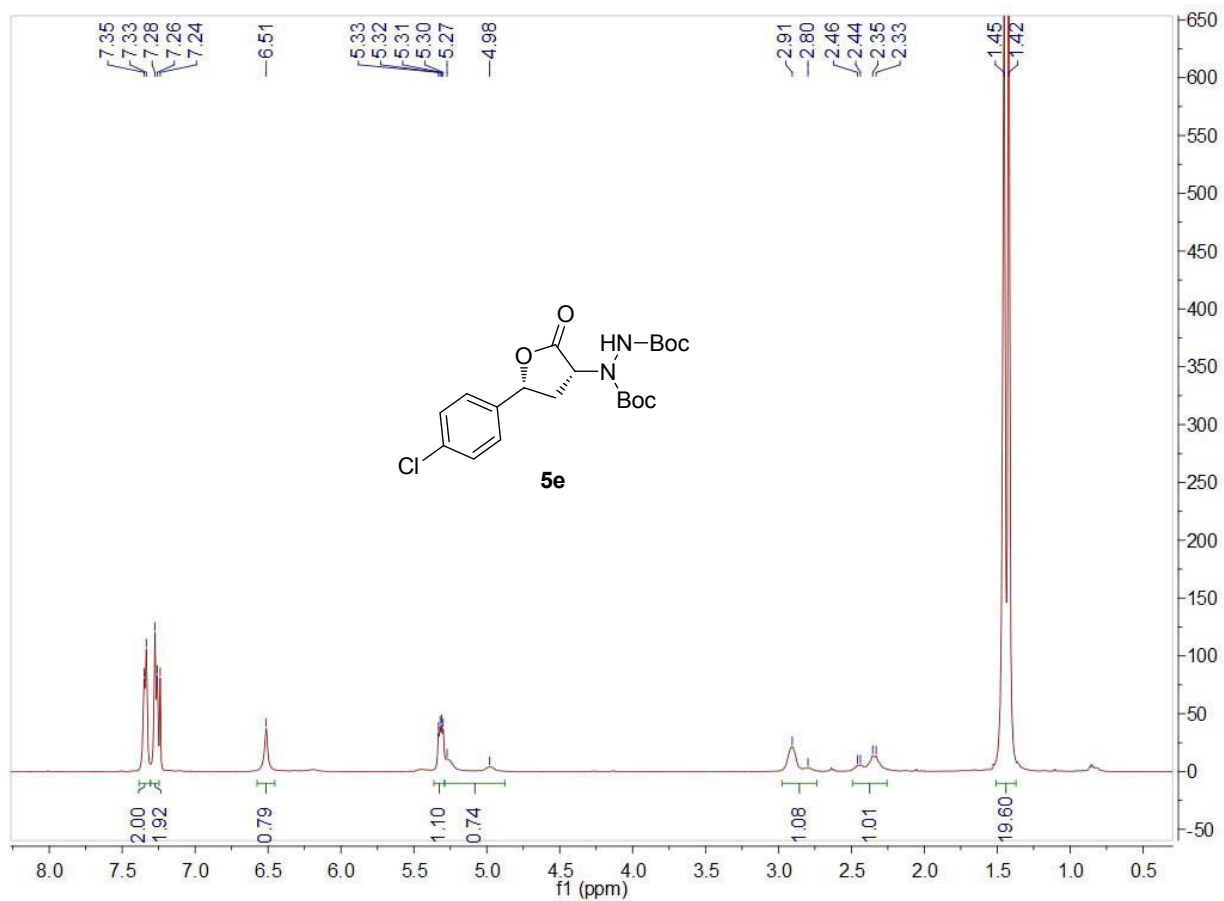


Chrom Type: Fixed WL Chromatogram, 210 nm

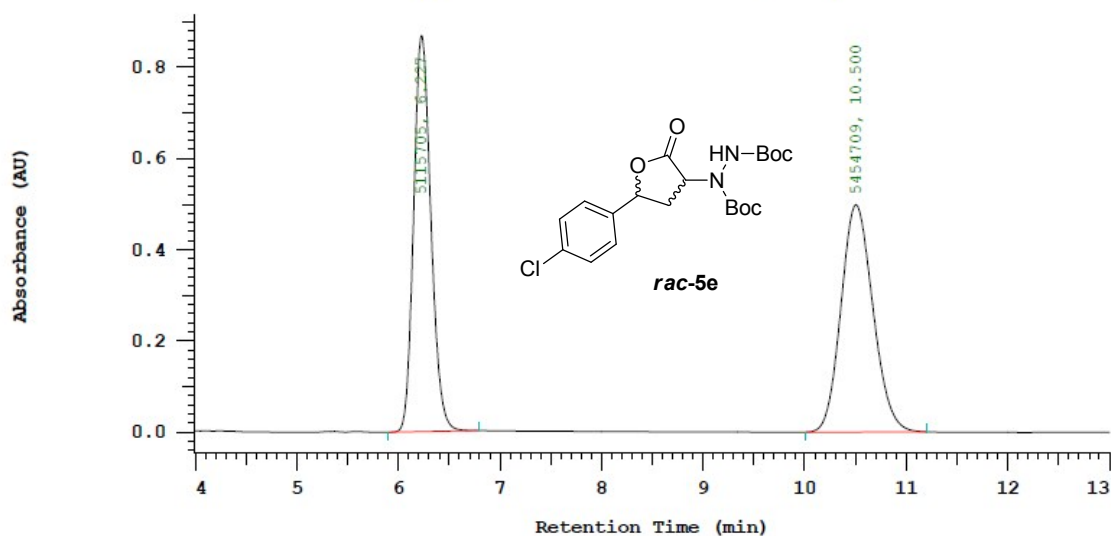
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	5.733	121894	2.568	BB
2	6.647	4625250	97.432	BB
4747144			100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm

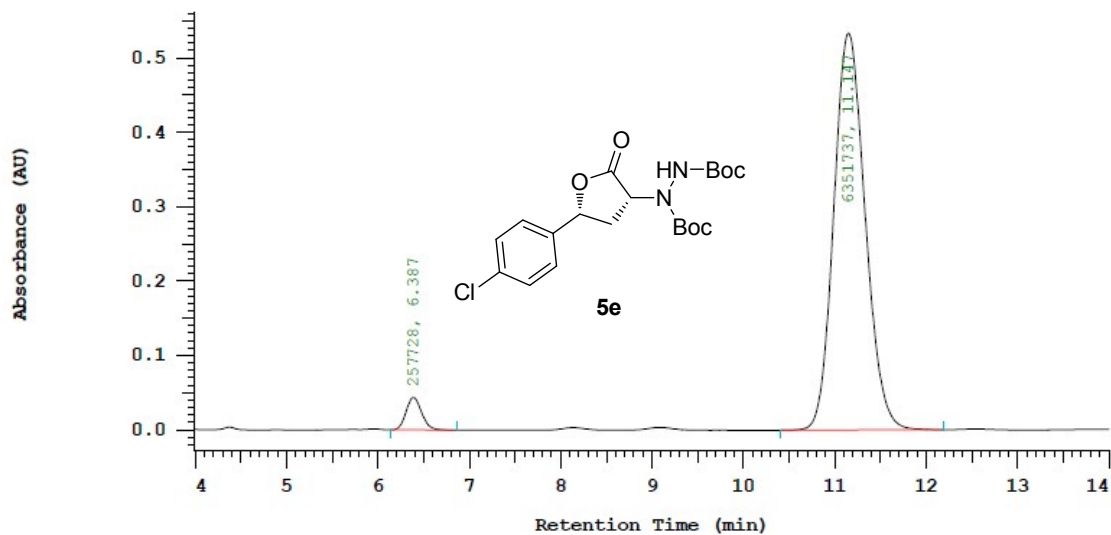


Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.227	5115705	48.396	BB
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		10570414	100.000	

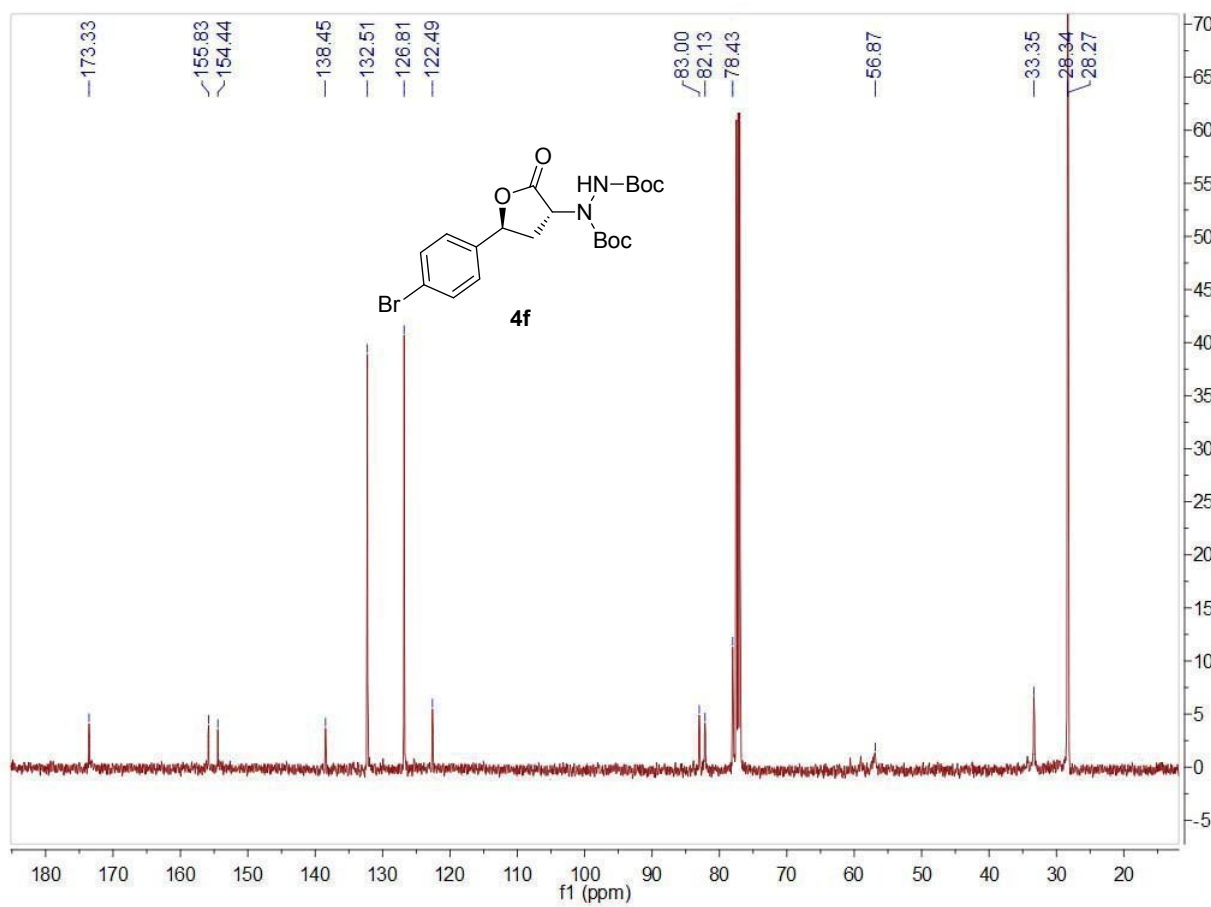
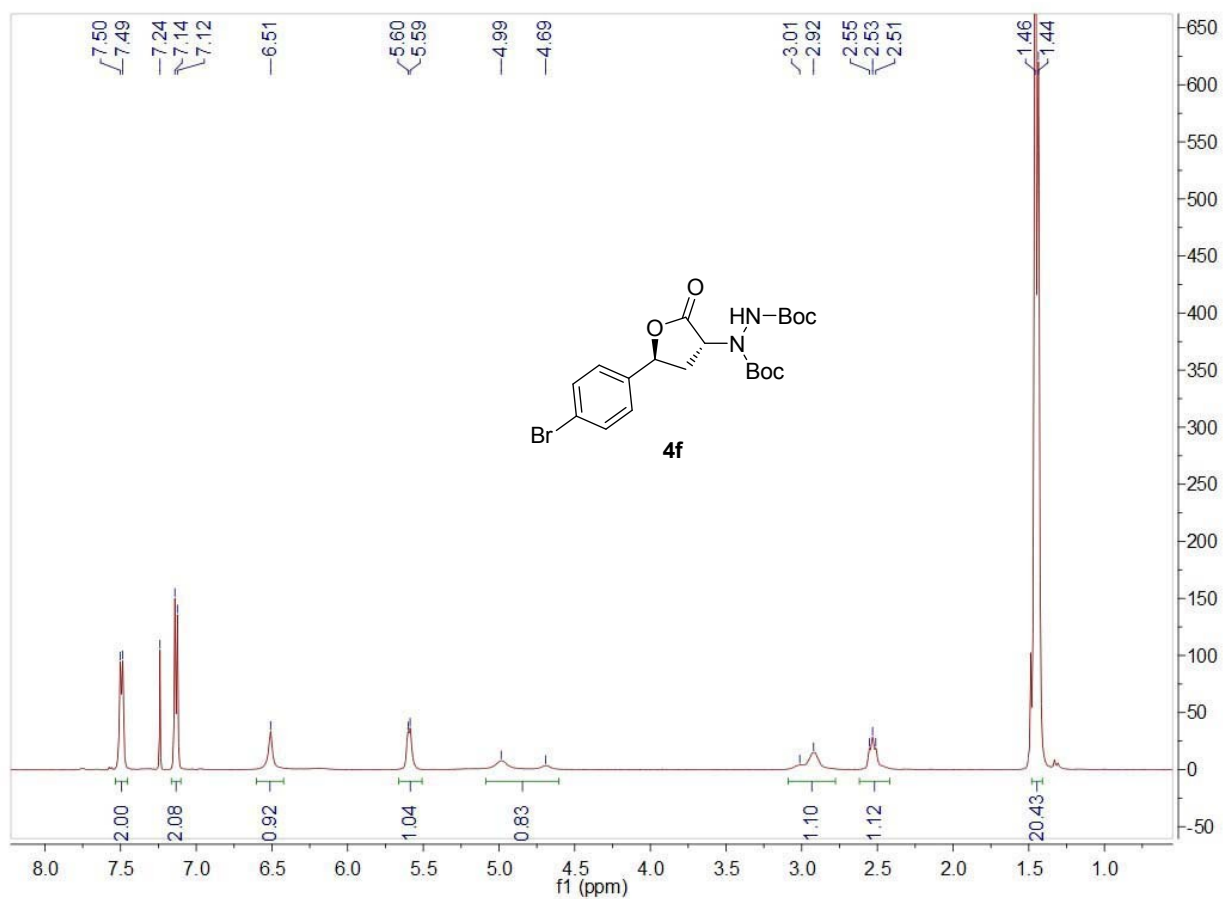
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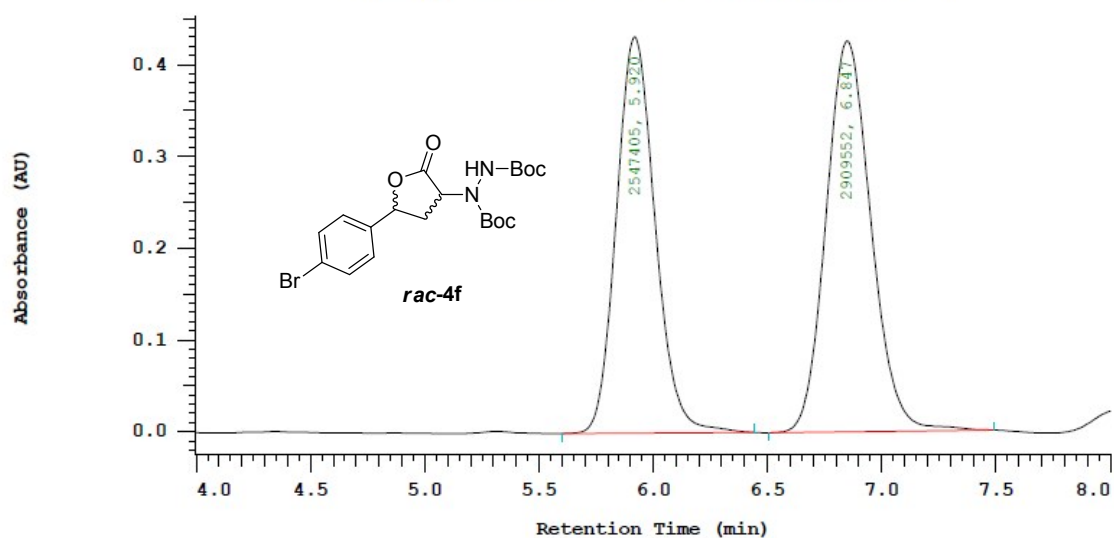
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.387	257728	3.899	BB
2	11.147	6351737	96.101	BB
		6609465	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



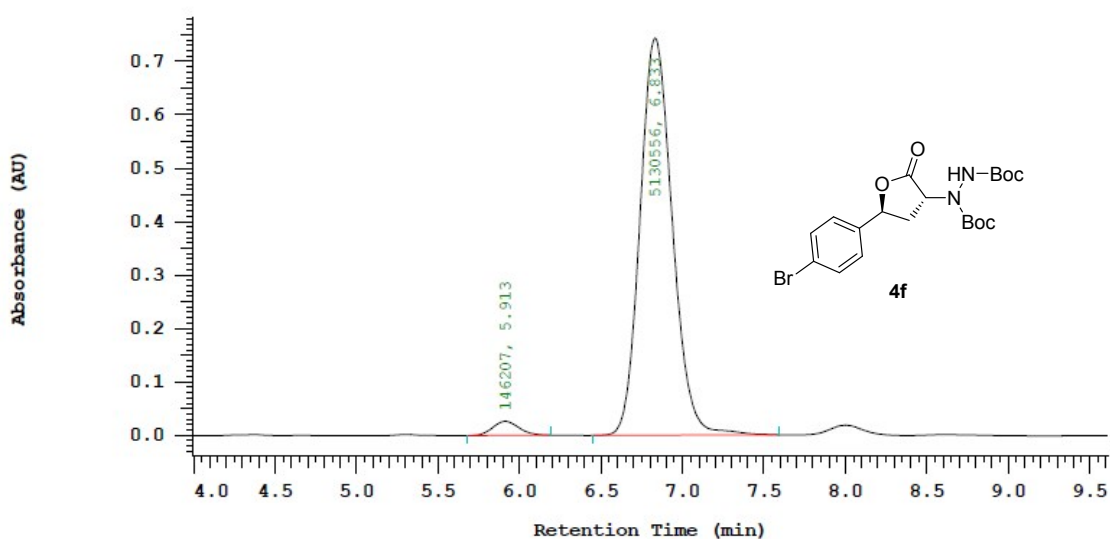
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	5.920	2547405	46.682	BB
2	6.847	2909552	53.318	BB
		5456957	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

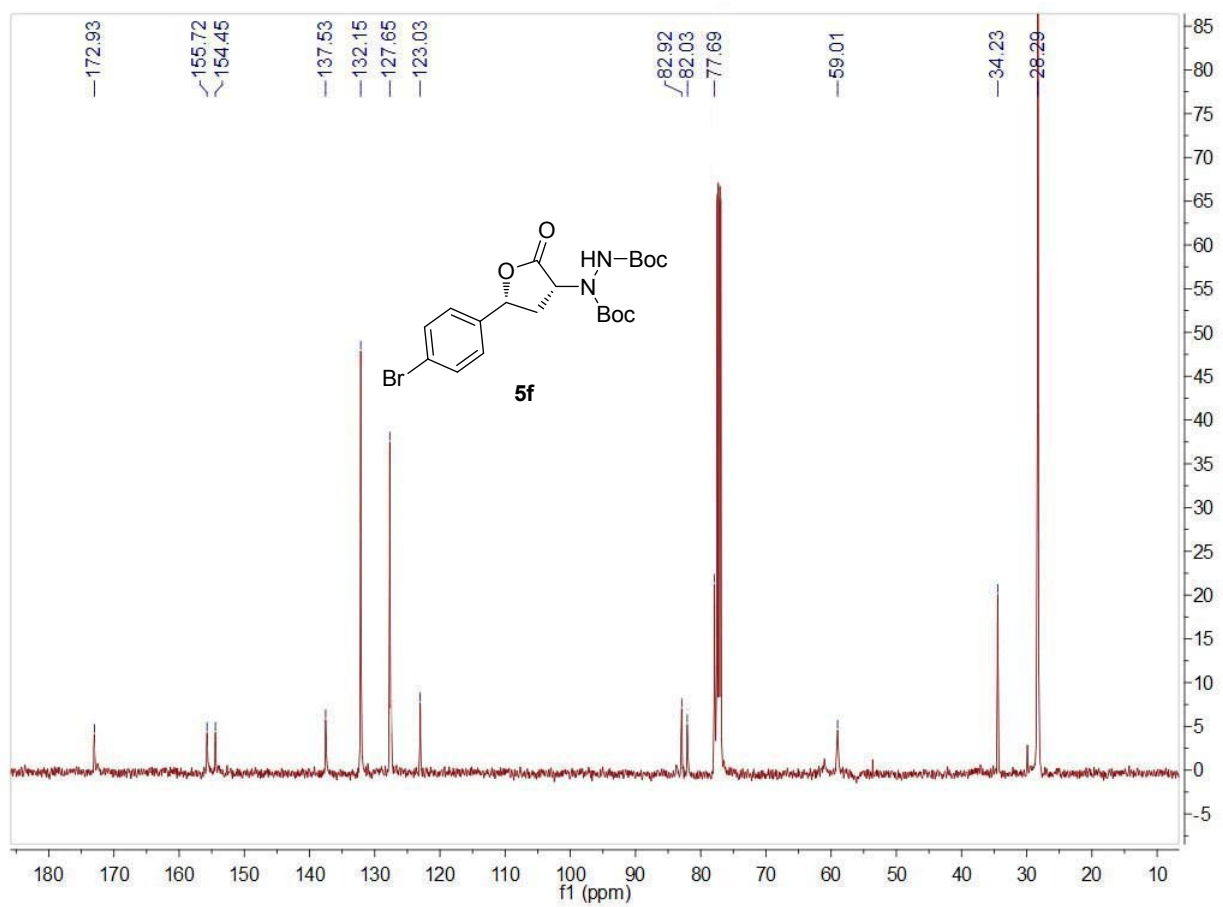
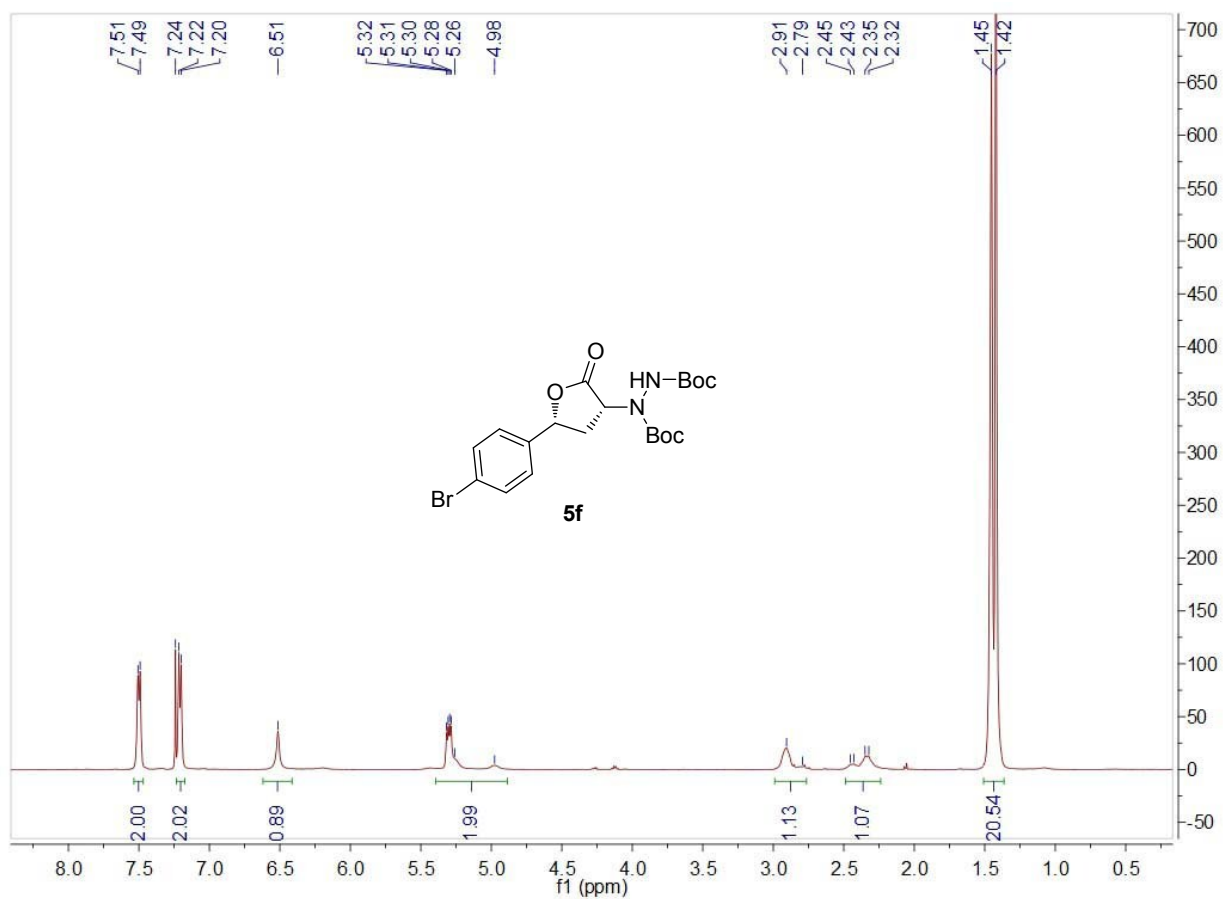


Chrom Type: Fixed WL Chromatogram, 210 nm

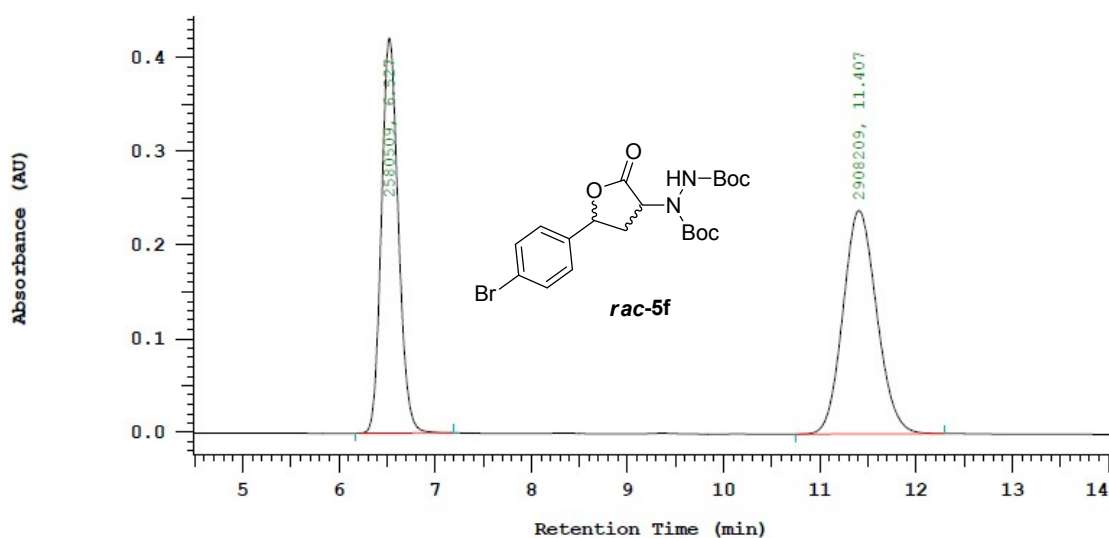
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	5.913	146207	2.771	BB
2	6.833	5130556	97.229	BB
		5276763	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



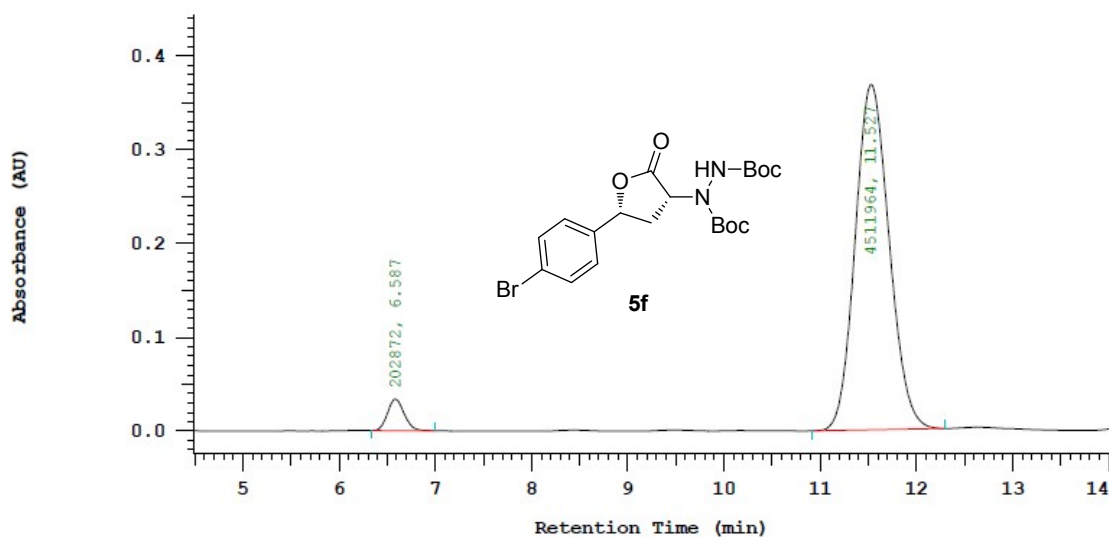
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.527	2580509	47.015	BB
2	11.407	2908209	52.985	BB
		5488718	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

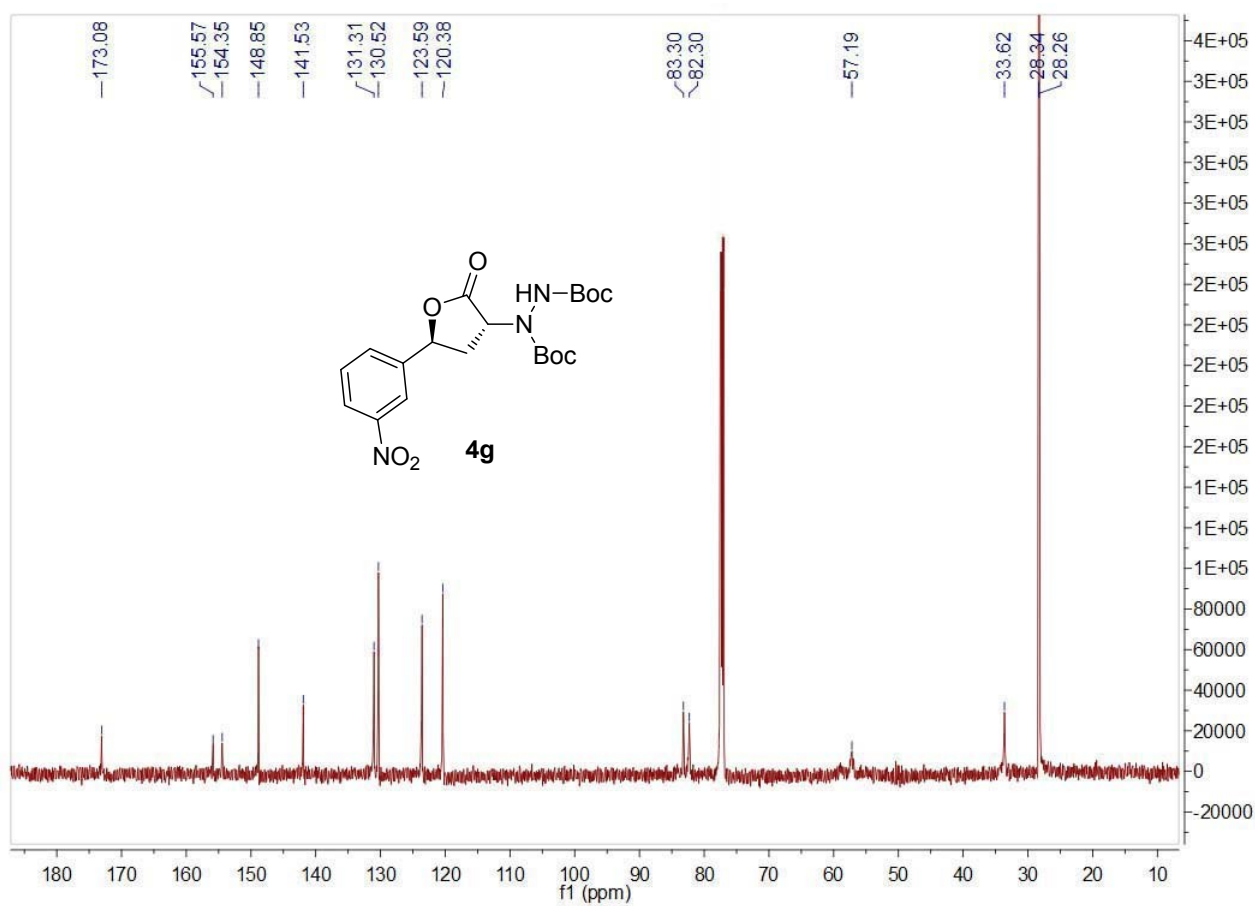
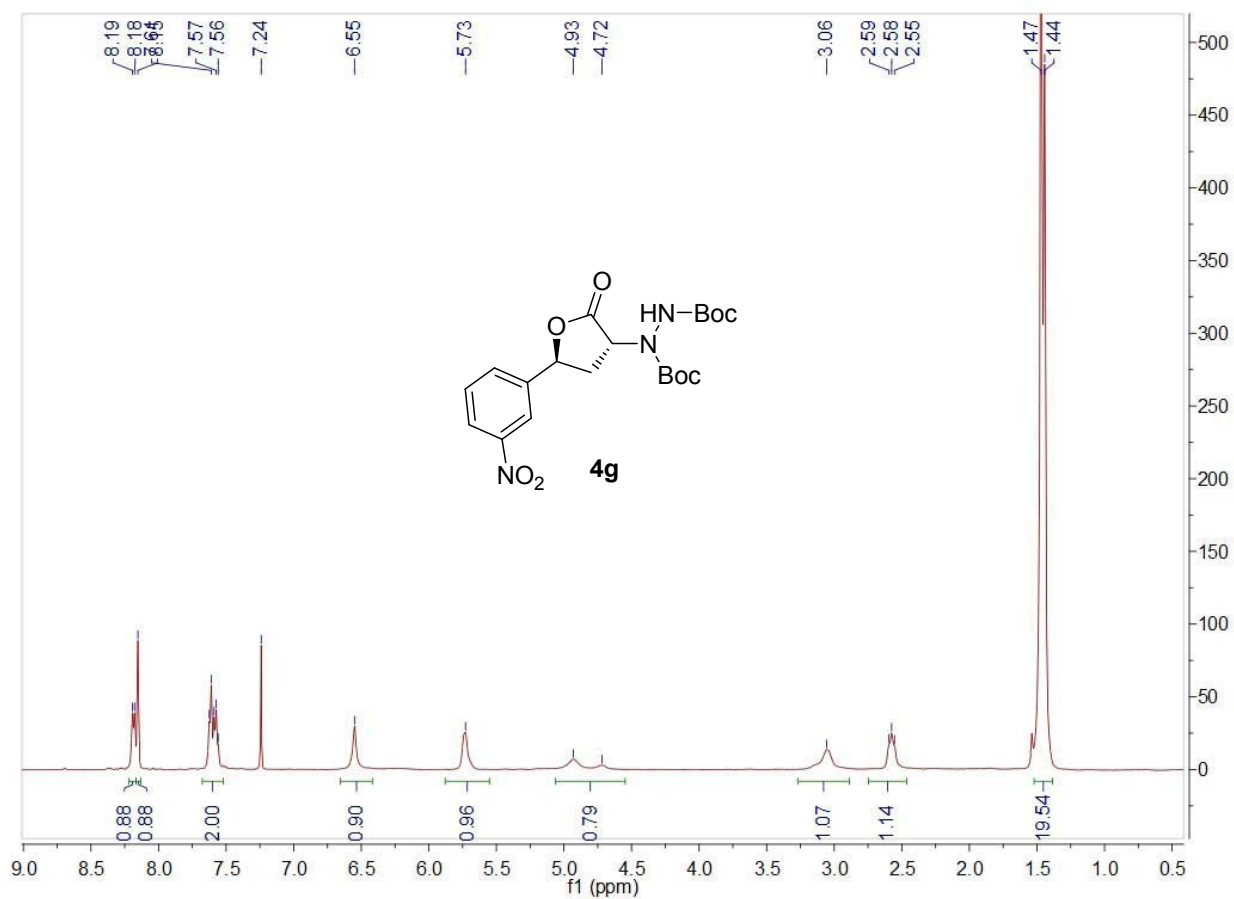


Chrom Type: Fixed WL Chromatogram, 210 nm

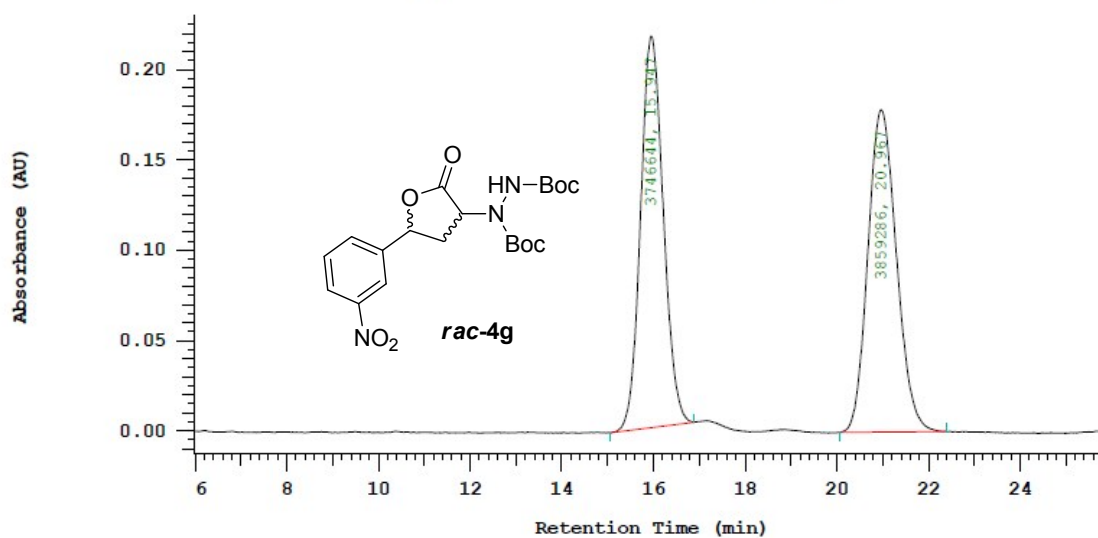
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.587	202872	4.303	BB
2	11.527	4511964	95.697	BB
		4714836	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm

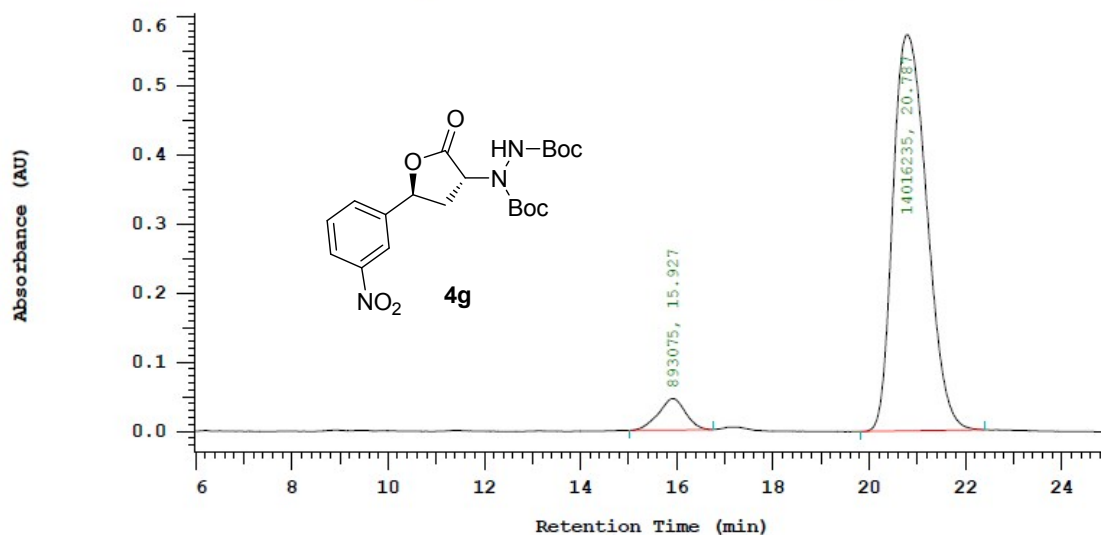


Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	15.947	3746644	49.260	BB
2	20.967	3859286	50.740	BB
		7605930	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

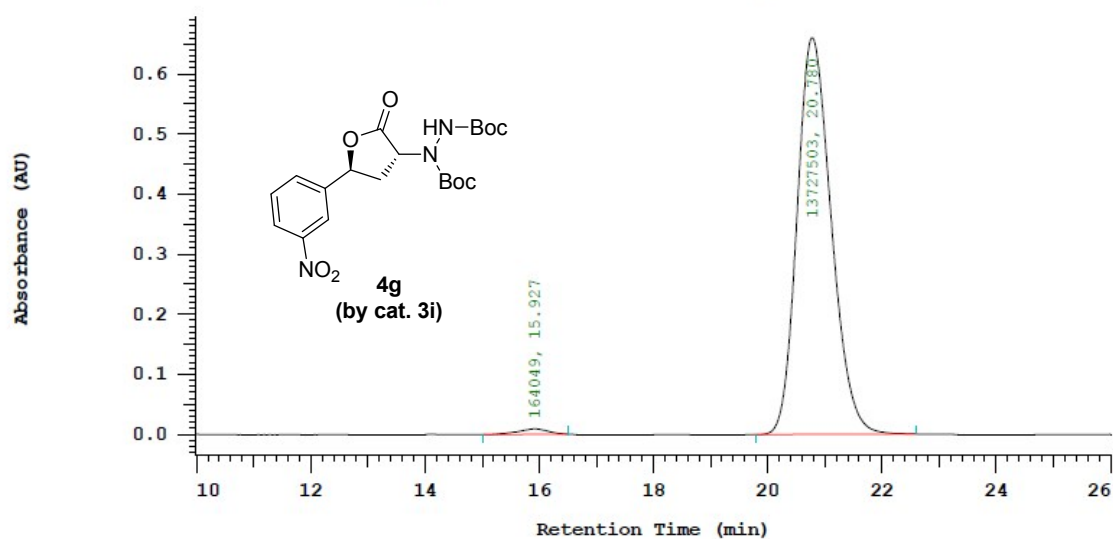


Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	15.927	893075	5.990	BB
2	20.787	14016235	94.010	BB
		14909310	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

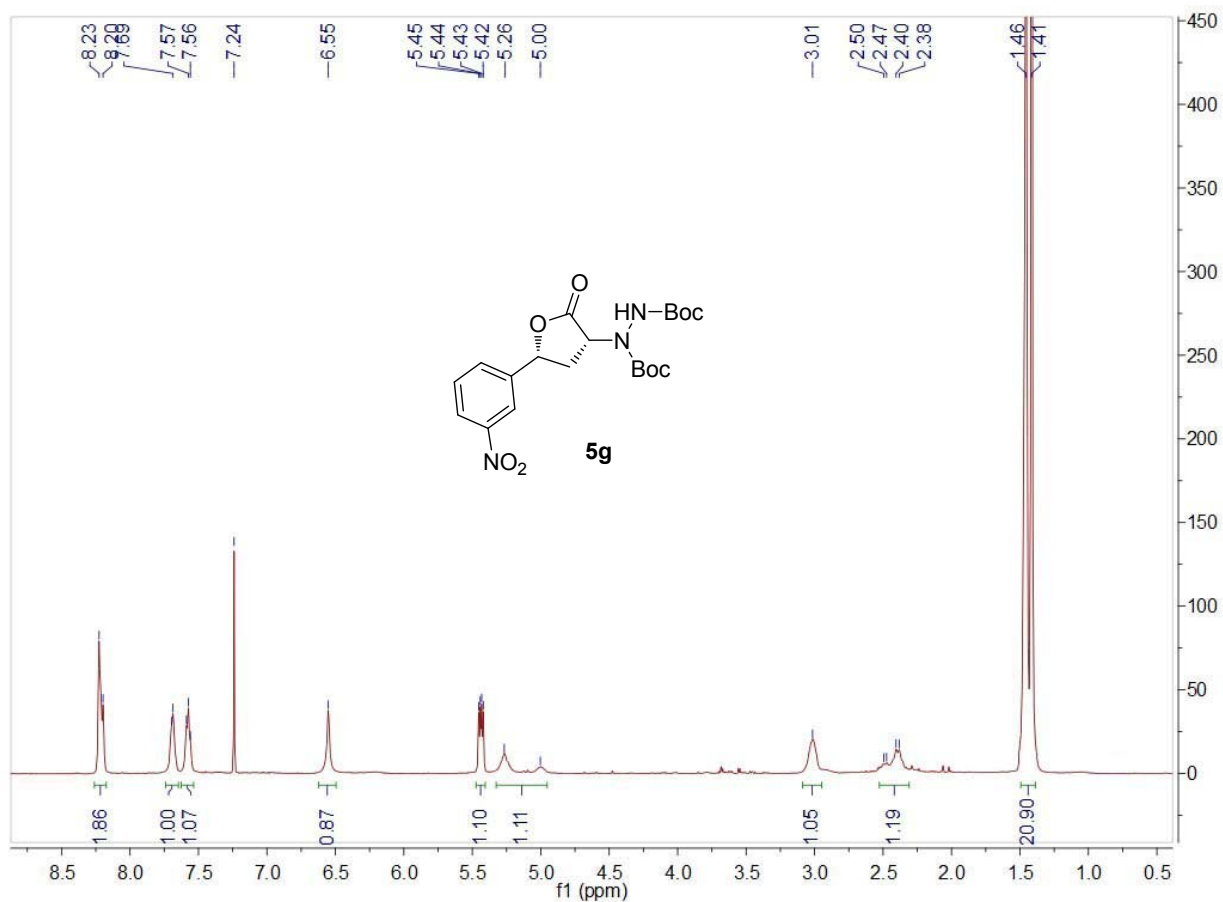


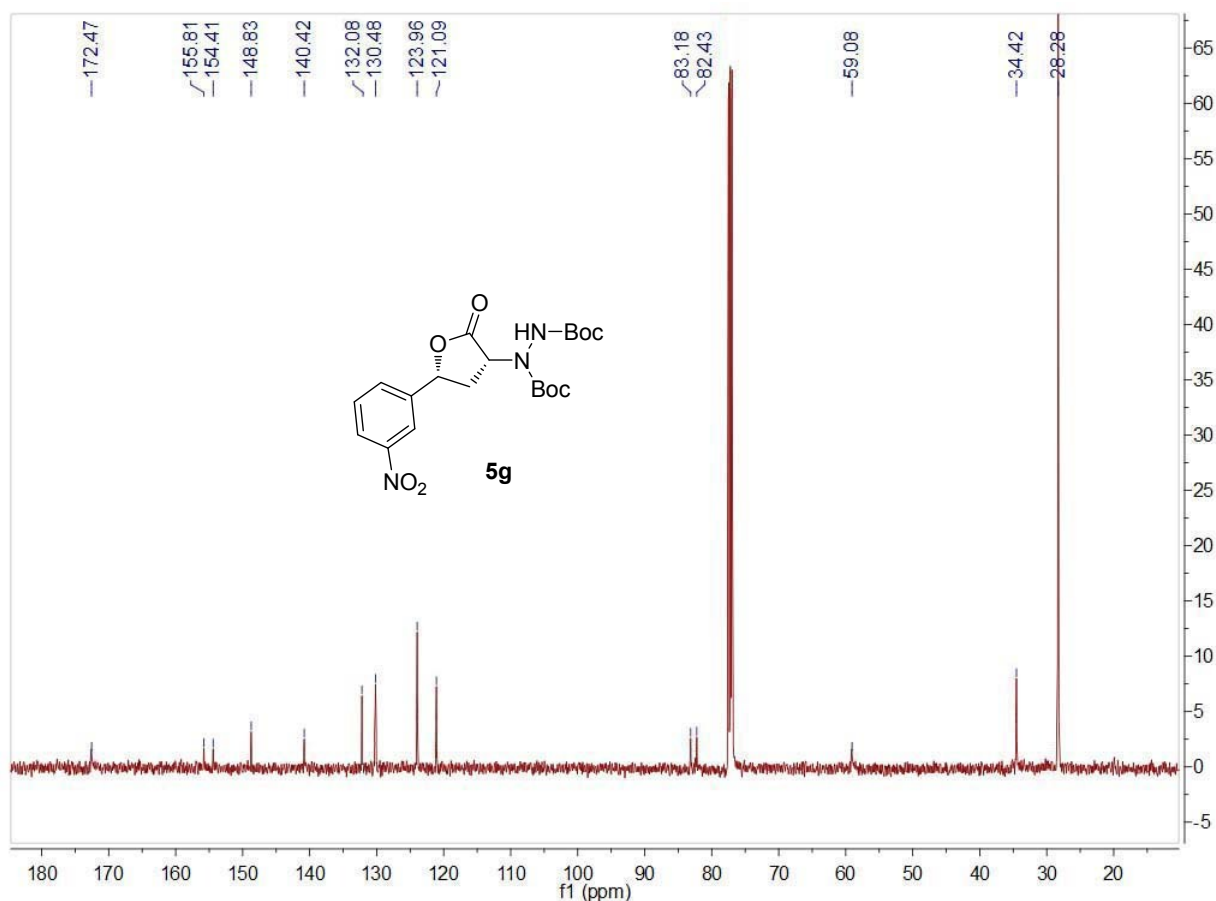
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

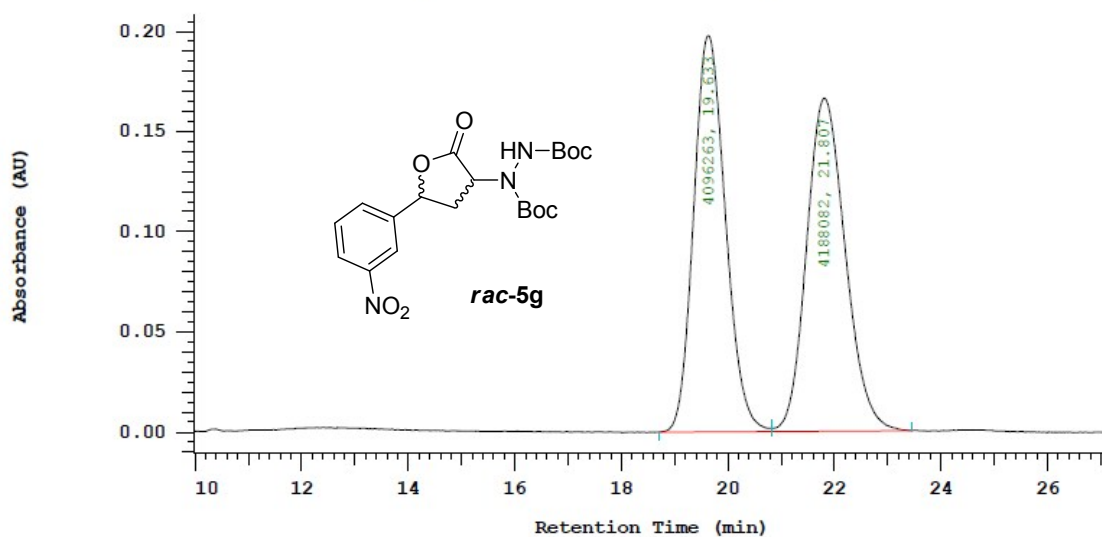
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	15.927	164049	1.181	BB
2	20.780	13727503	98.819	BB
		13891552	100.000	





Chrom Type: Fixed WL Chromatogram, 210 nm



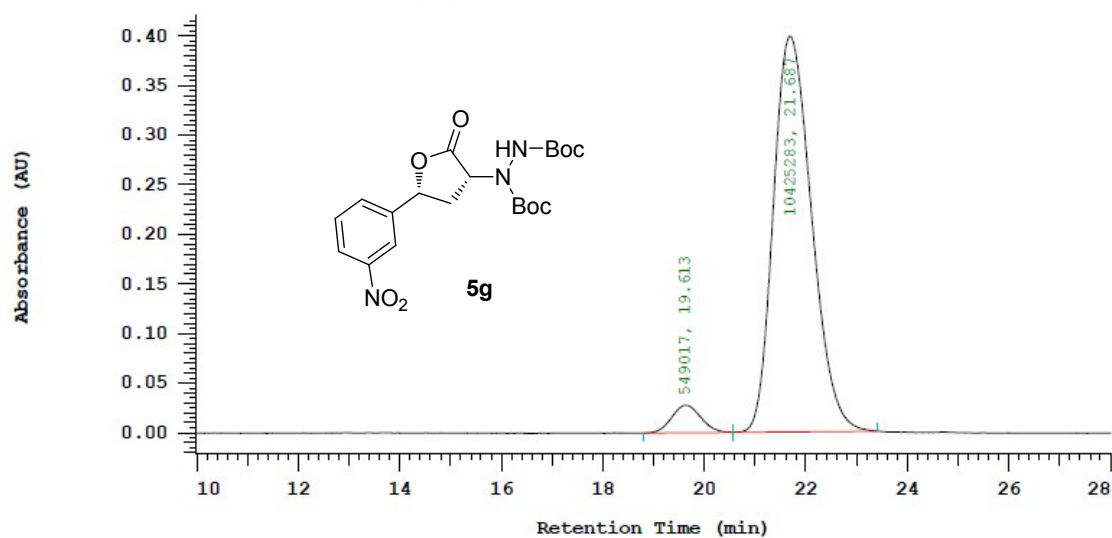
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	19.633	4096263	49.446	BV
2	21.807	4188082	50.554	VB
		8284345	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



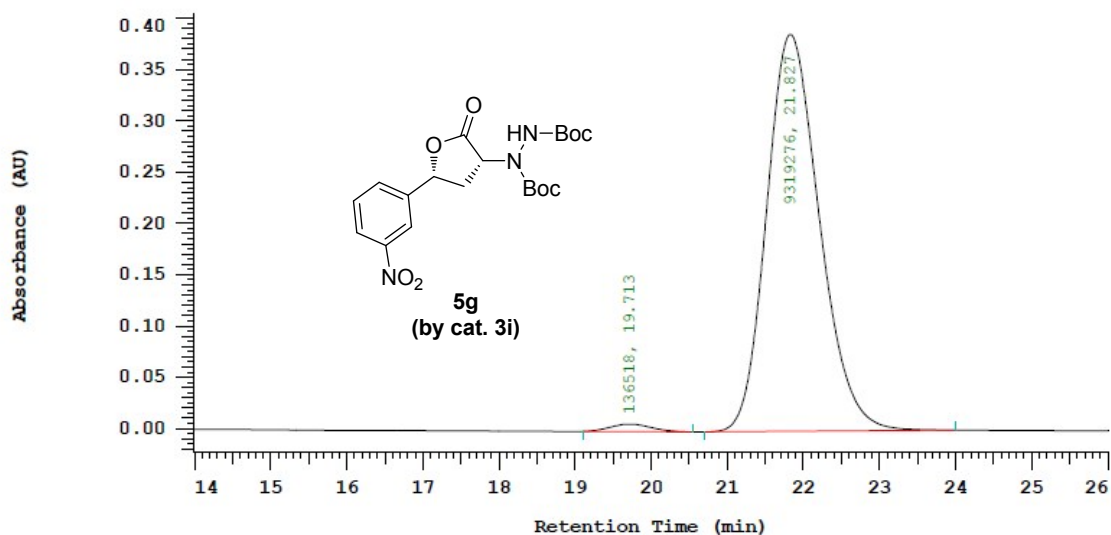
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	19.613	549017	5.003	BB
2	21.687	10425283	94.997	BB
		10974300	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

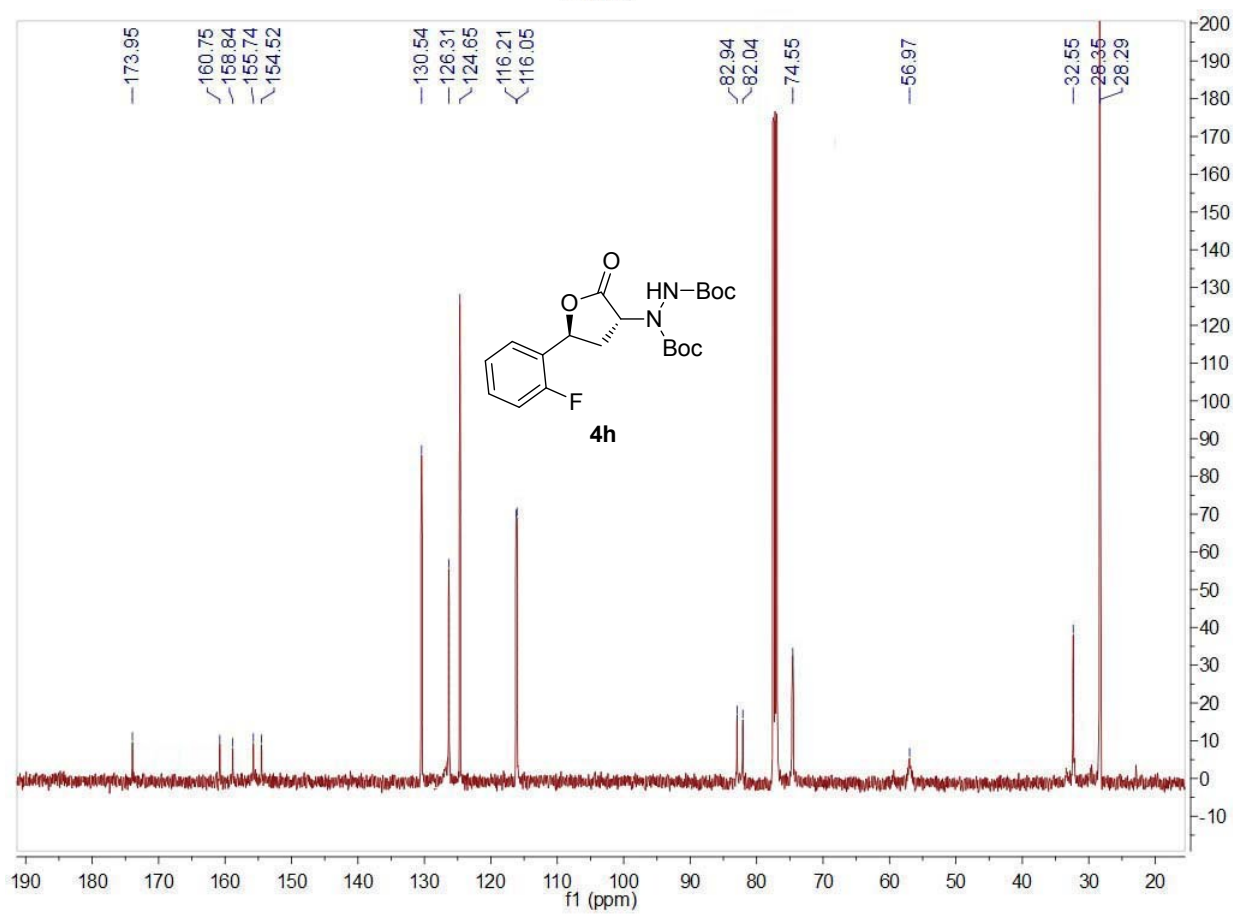
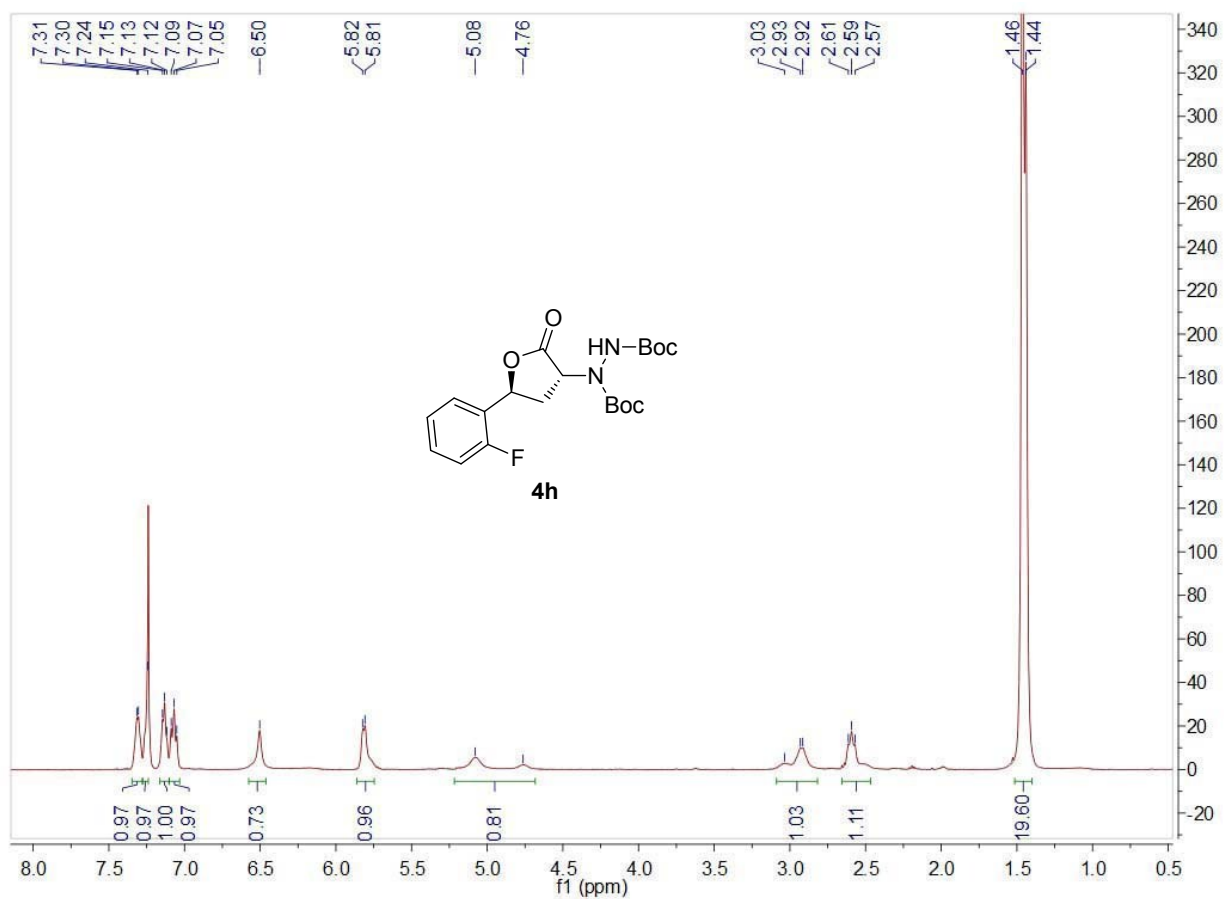


Chrom Type: Fixed WL Chromatogram, 210 nm

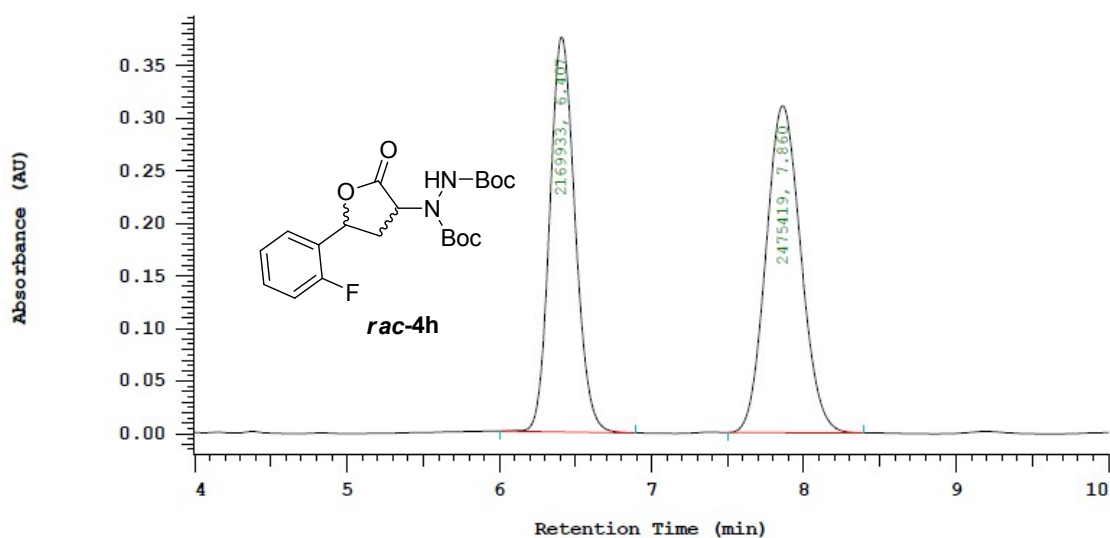
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	19.713	136518	1.444	BB
2	21.827	9319276	98.556	BB
		9455794	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



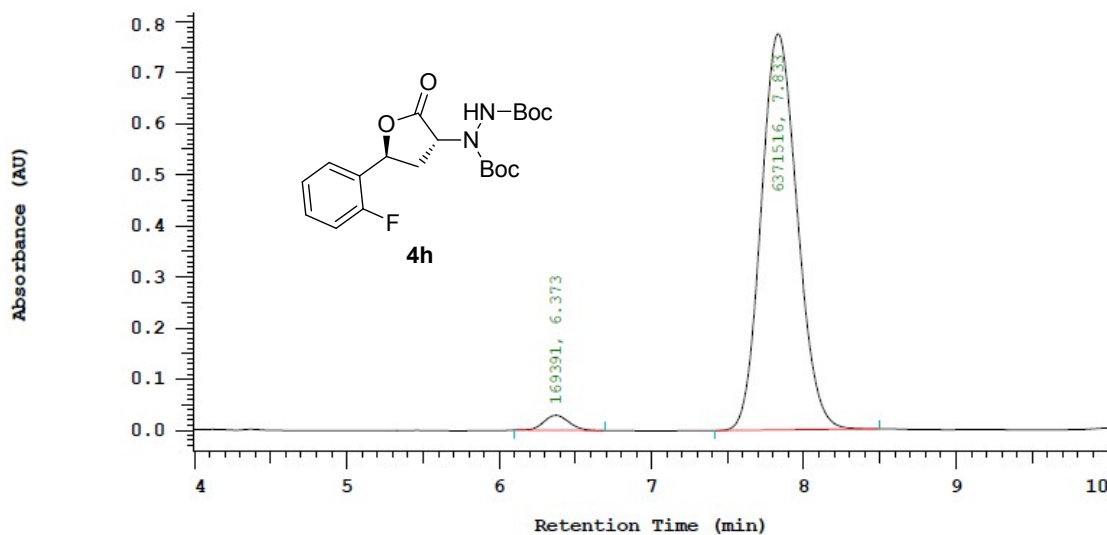
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.407	2169933	46.712	BB
2	7.860	2475419	53.288	BB
		4645352	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

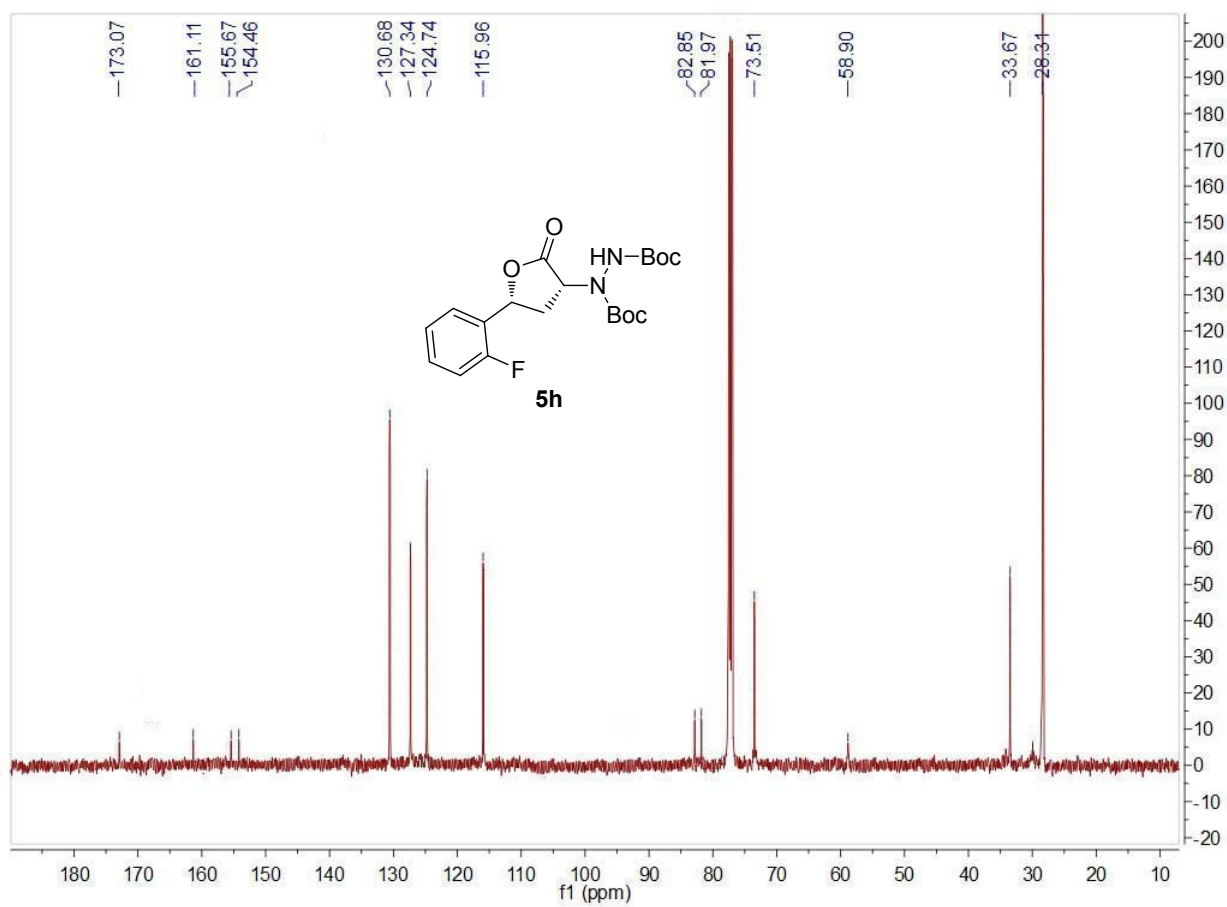
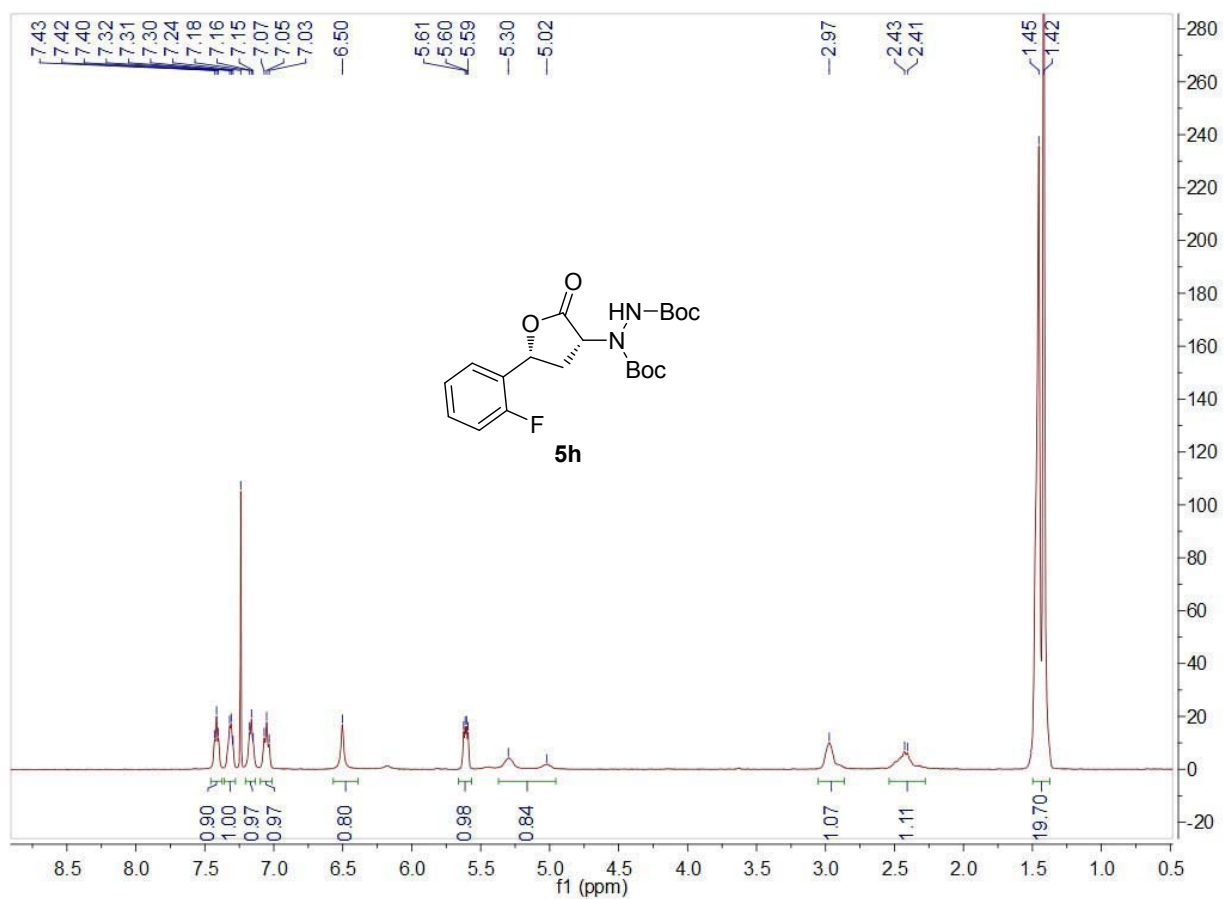


Chrom Type: Fixed WL Chromatogram, 210 nm

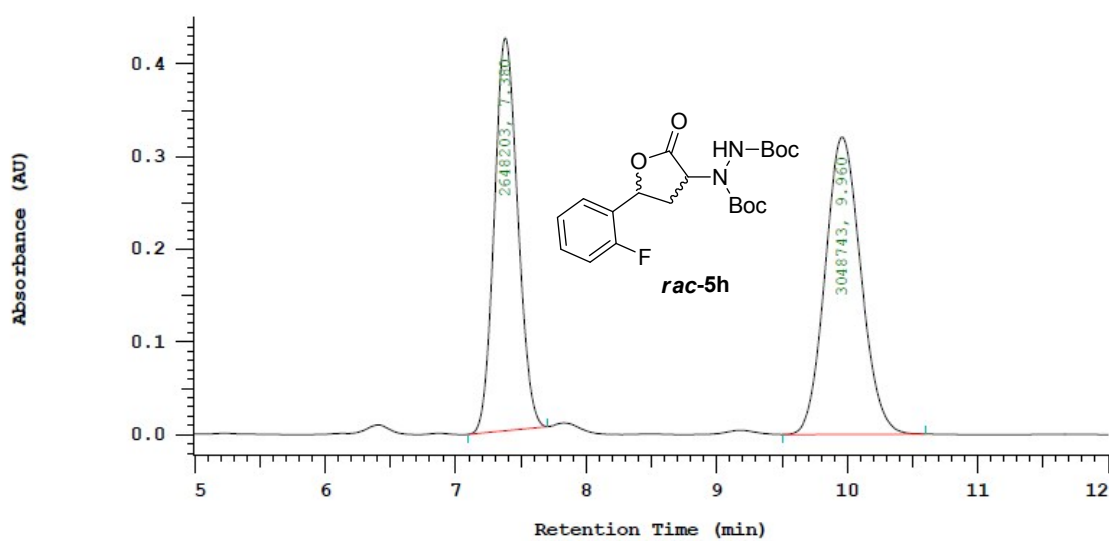
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.373	169391	2.590	BB
2	7.833	6371516	97.410	BB
		6540907	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



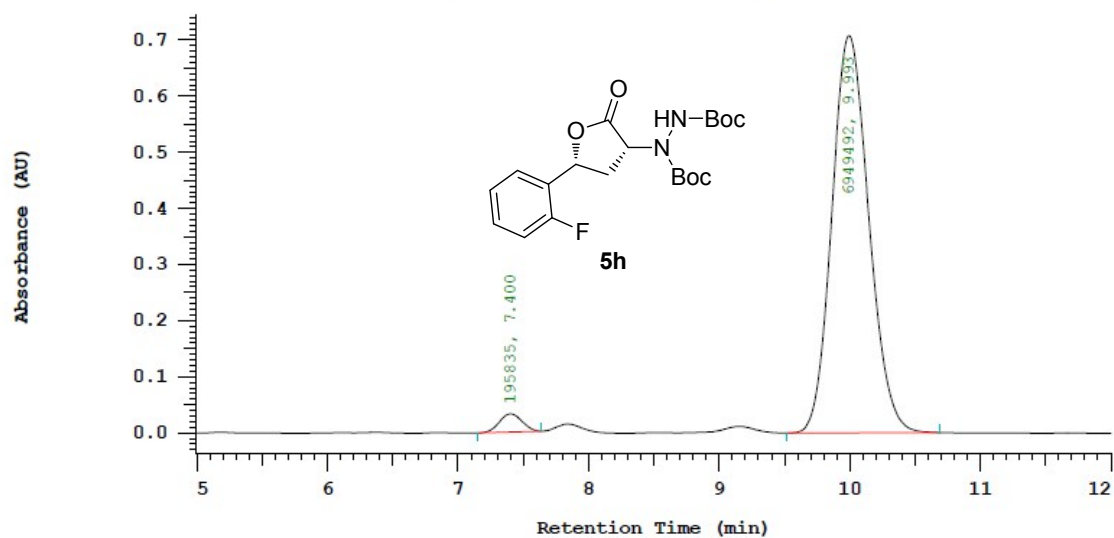
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.380	2648203	46.485	BB
2	9.960	3048743	53.515	BB
		5696946	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

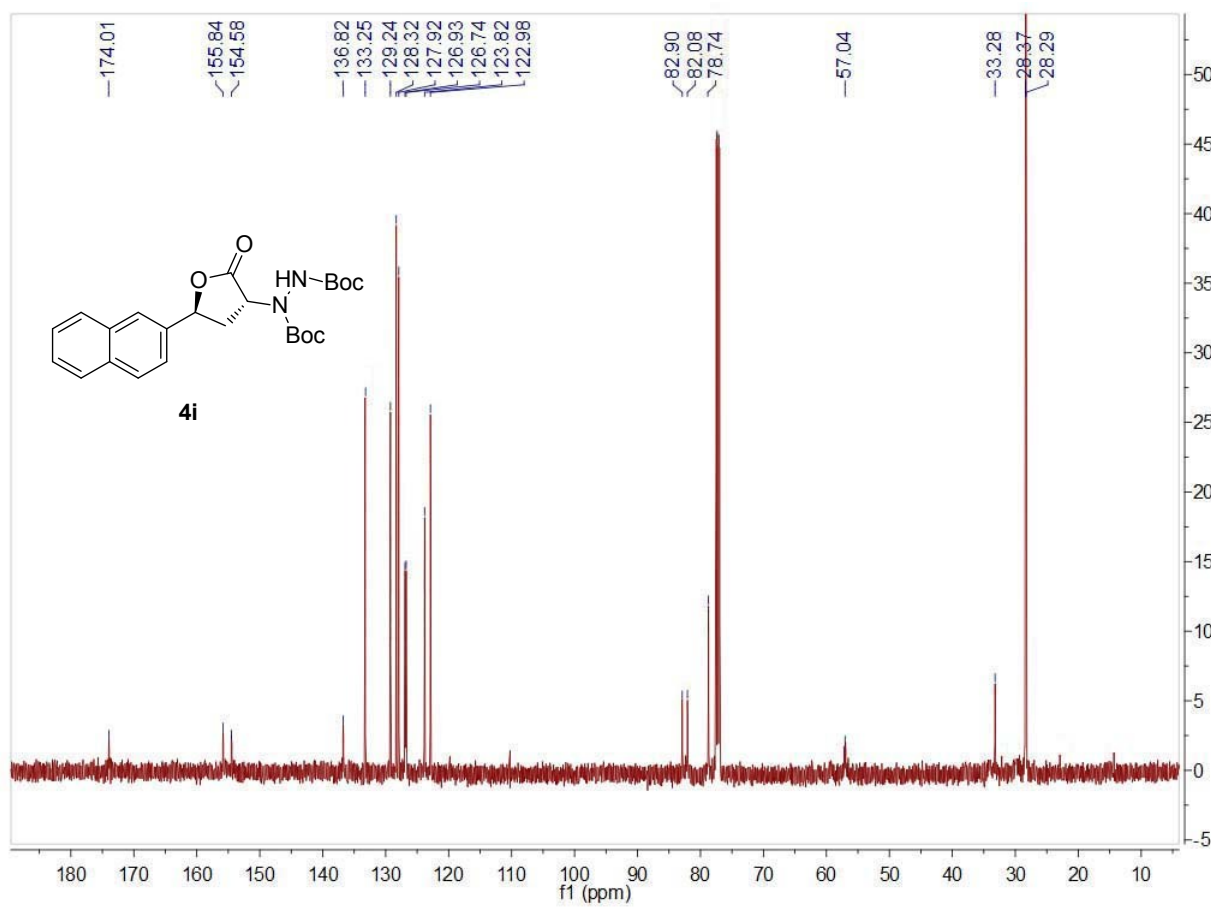
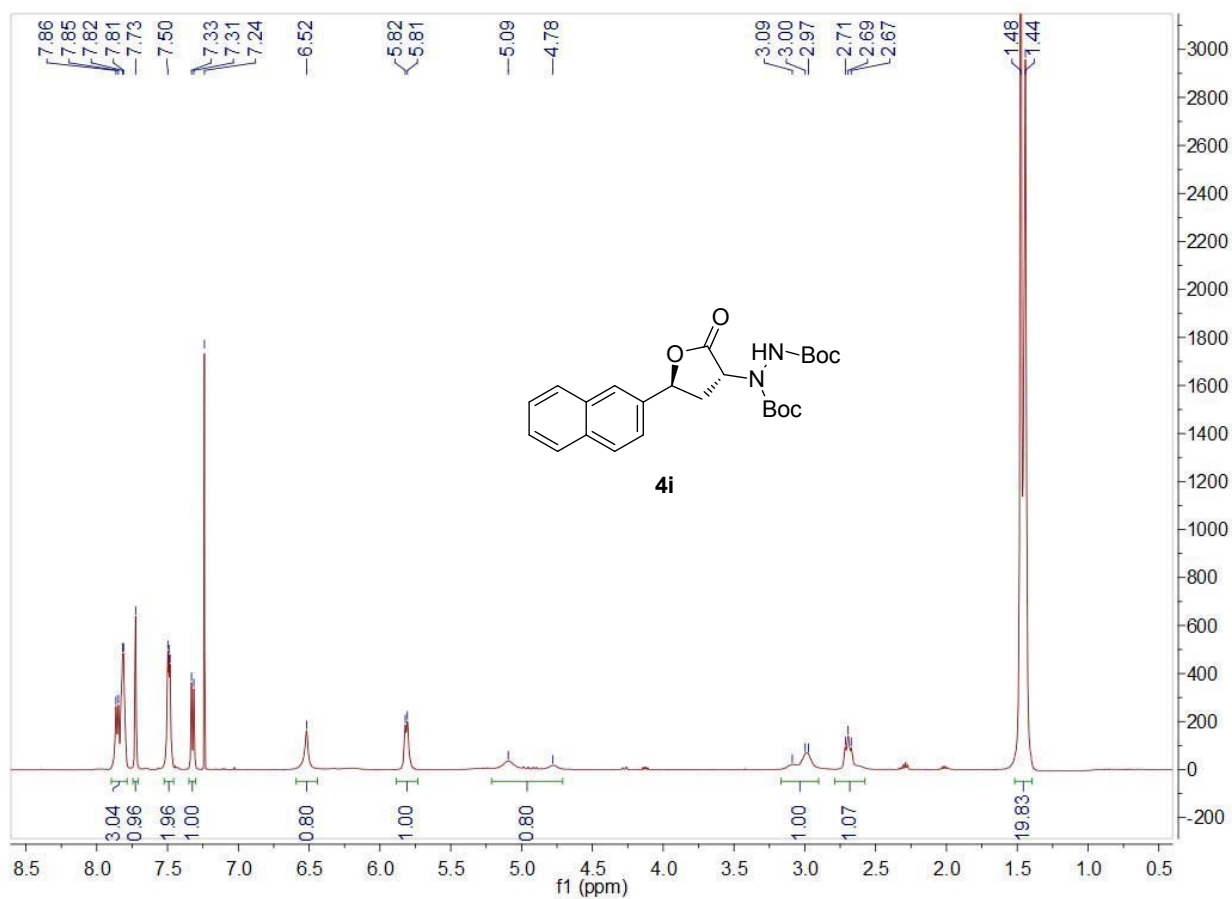


Chrom Type: Fixed WL Chromatogram, 210 nm

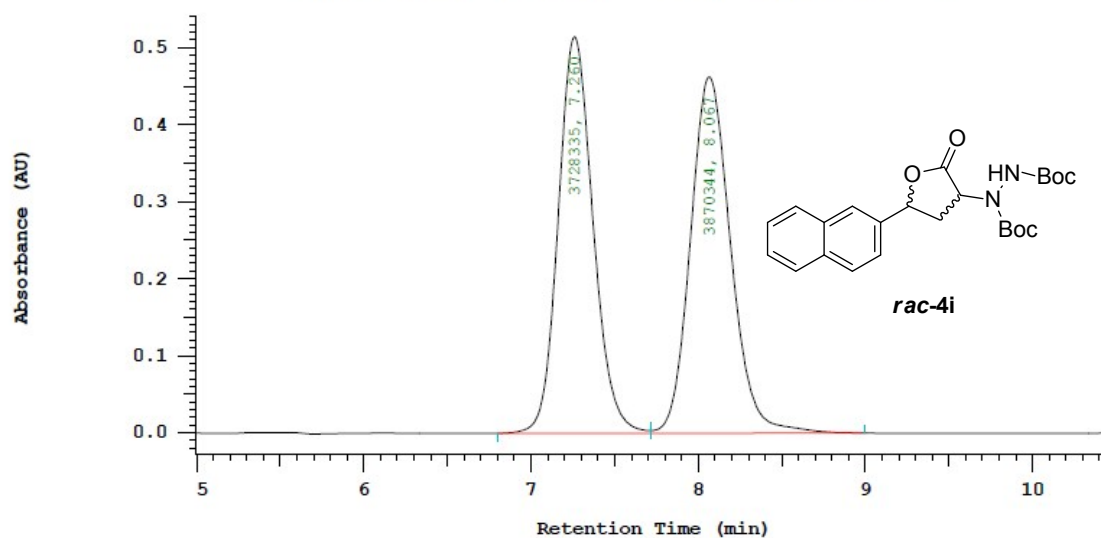
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.400	195835	2.741	BB
2	9.993	6949492	97.259	BB
		7145327	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



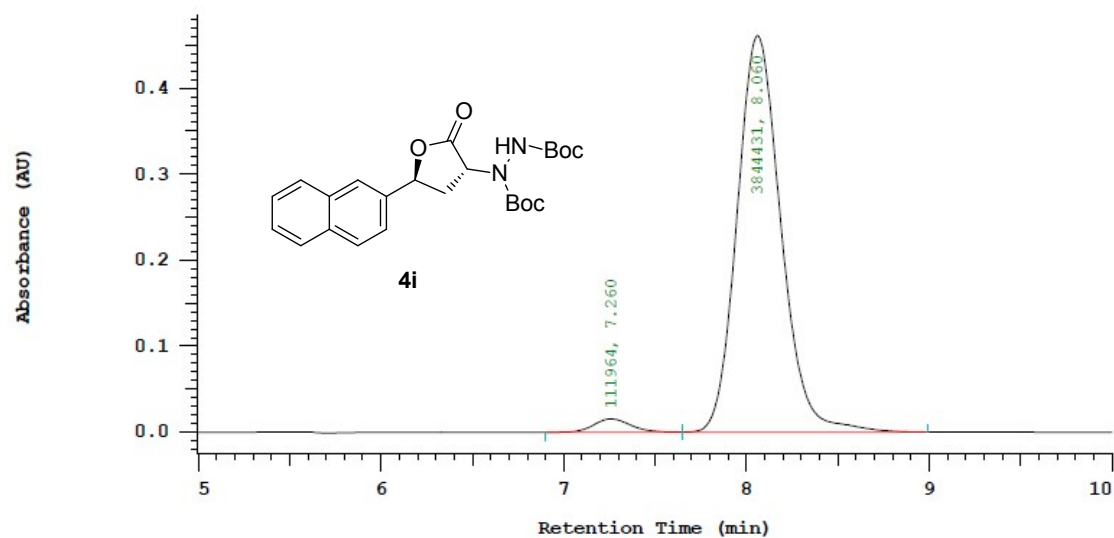
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.260	3728335	49.066	BV
2	8.067	3870344	50.934	VB
		7598679	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

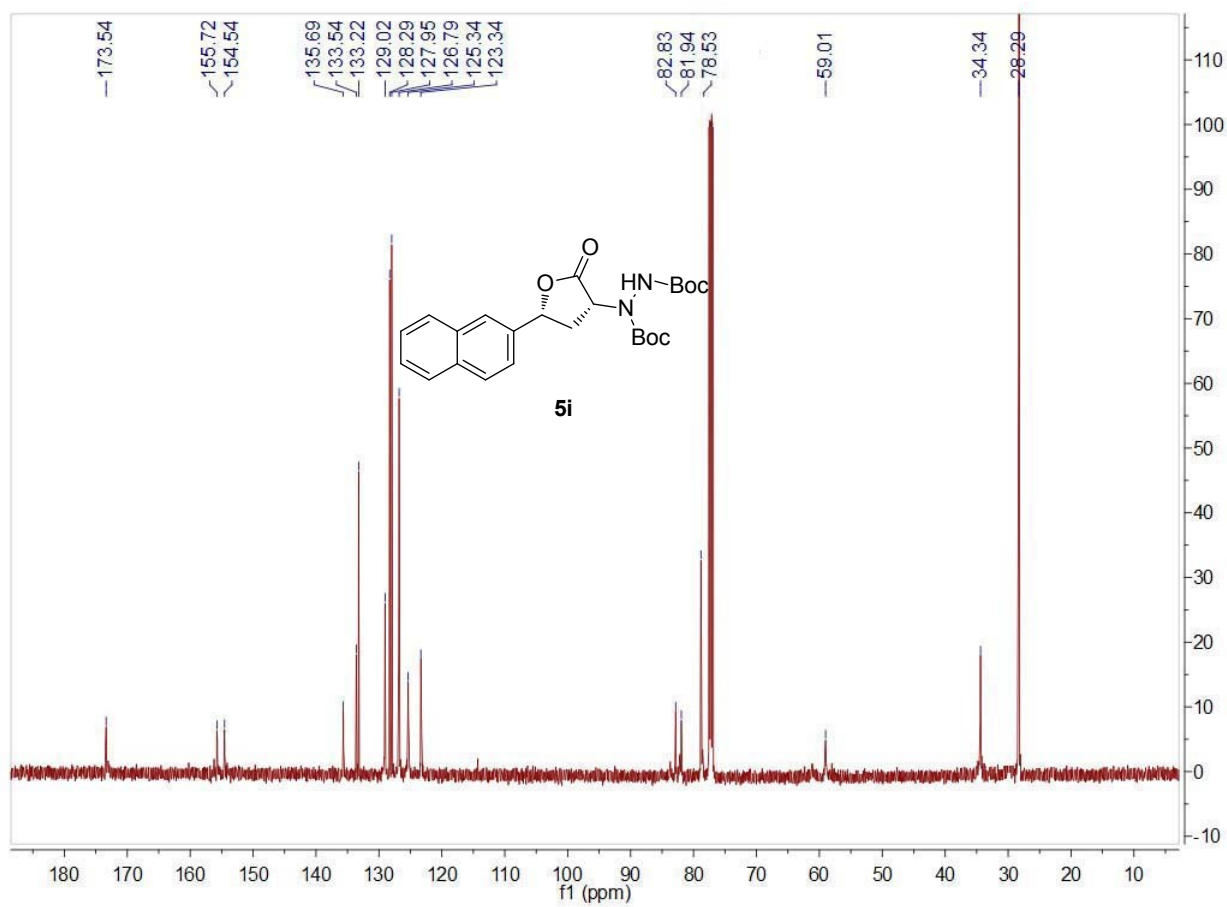
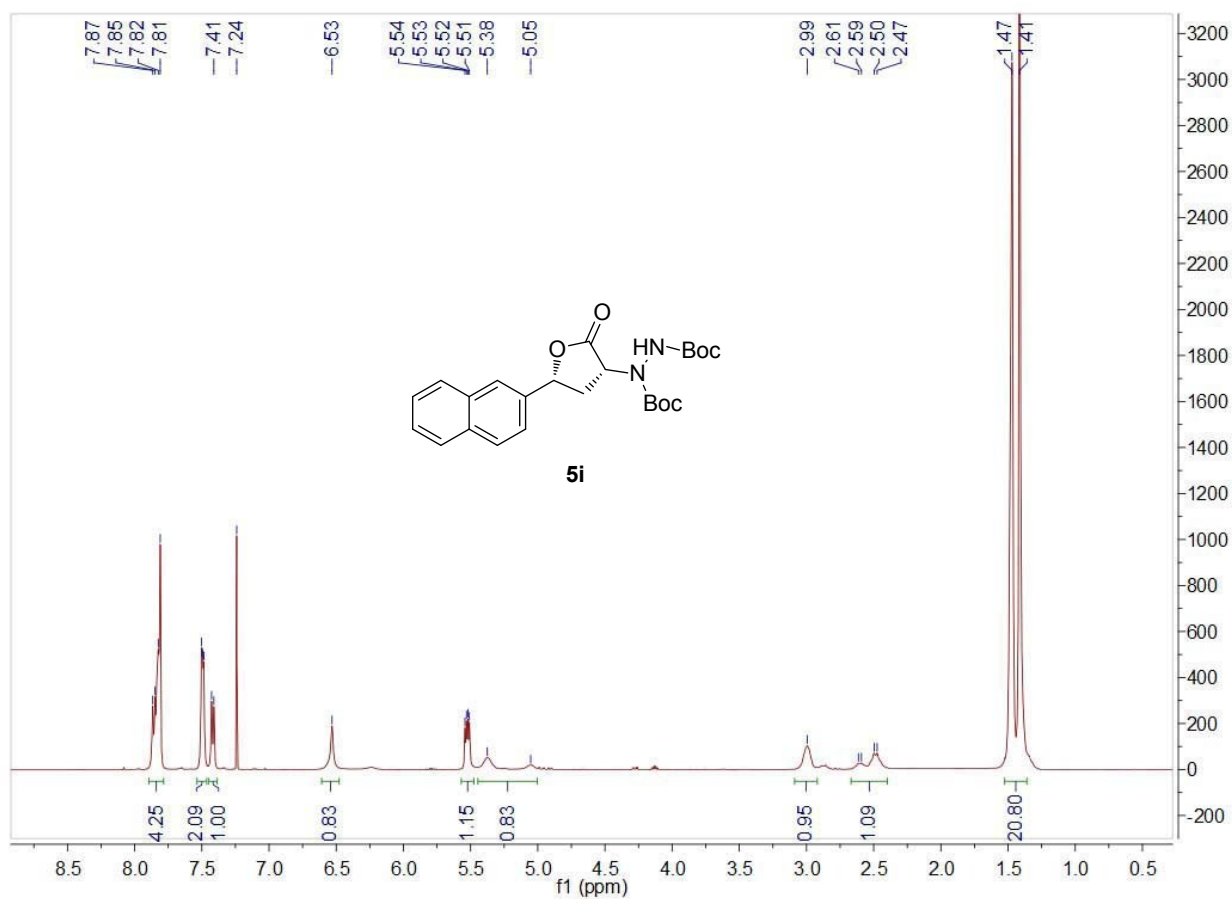


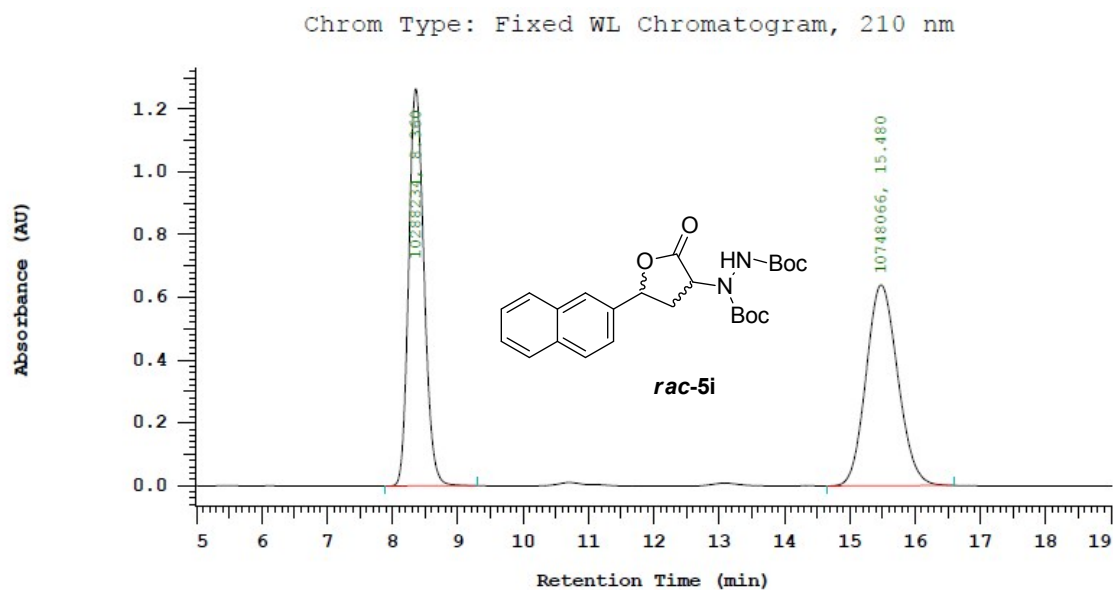
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

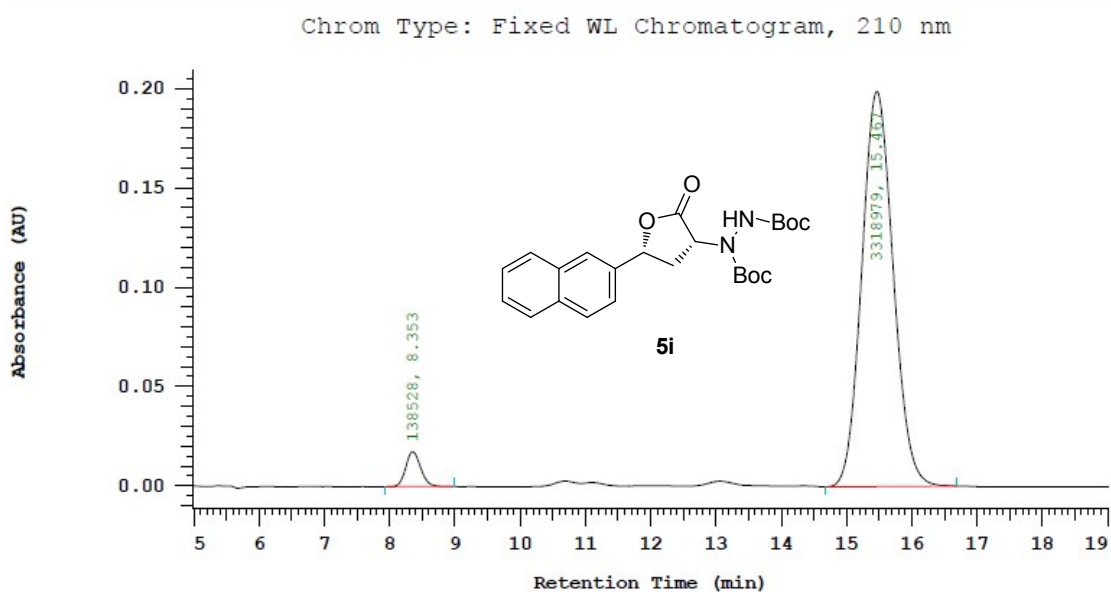
No.	RT	Area	Area %	BC
1	7.260	111964	2.830	BV
2	8.060	3844431	97.170	VB
		3956395	100.000	





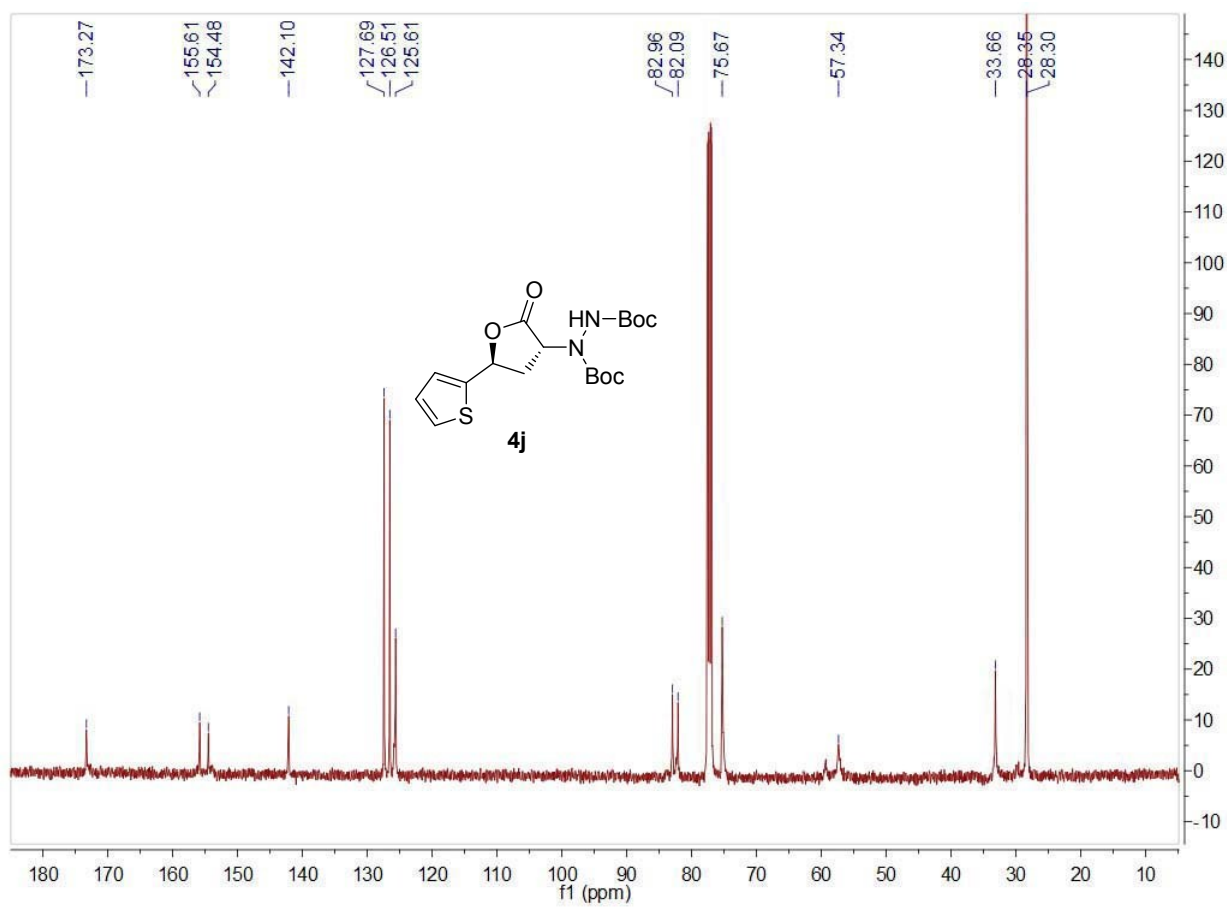
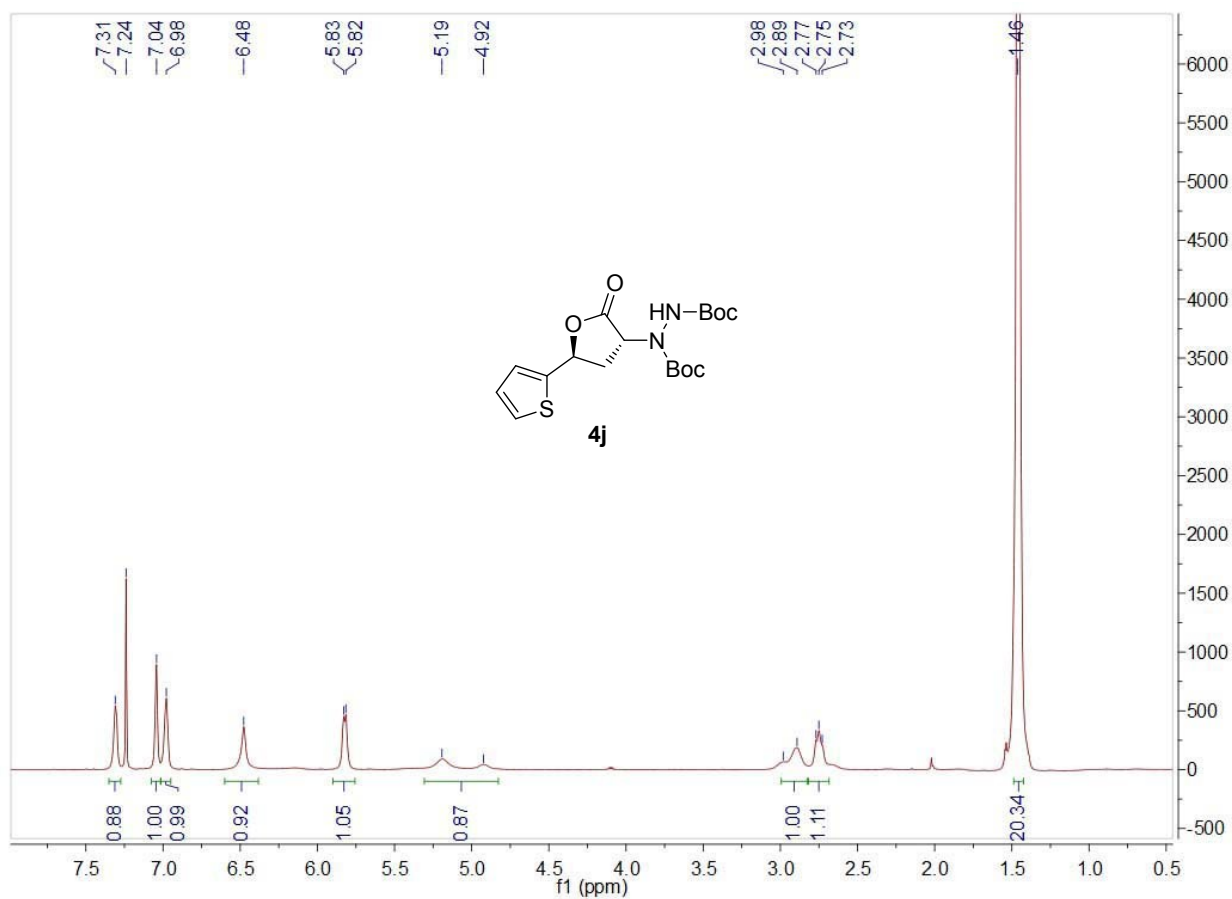
Chrom Type: Fixed WL Chromatogram, 210 nm
 Peak Quantitation: AREA
 Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.360	10288234	48.907	BB
2	15.480	10748066	51.093	BB
		21036300	100.000	

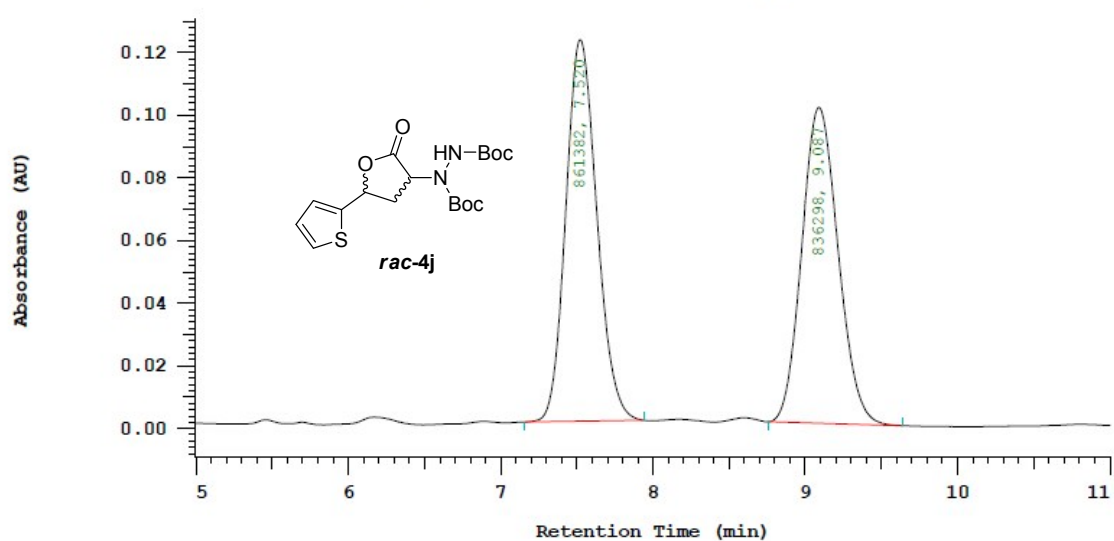


Chrom Type: Fixed WL Chromatogram, 210 nm
 Peak Quantitation: AREA
 Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.353	138528	4.007	BB
2	15.467	3318979	95.993	BB
		3457507	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



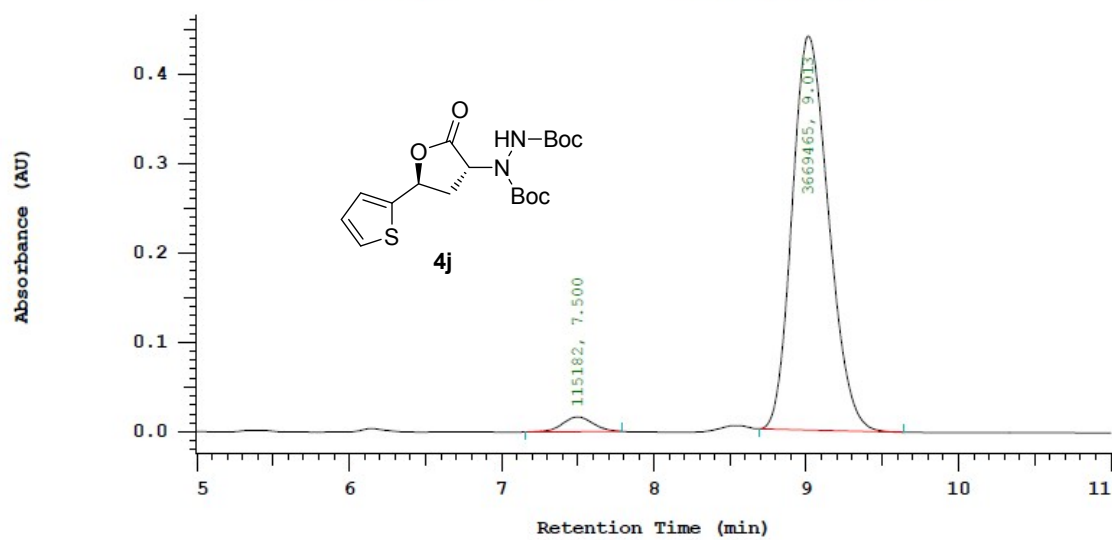
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.520	861382	50.739	BB
2	9.087	836298	49.261	BB
1697680			100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

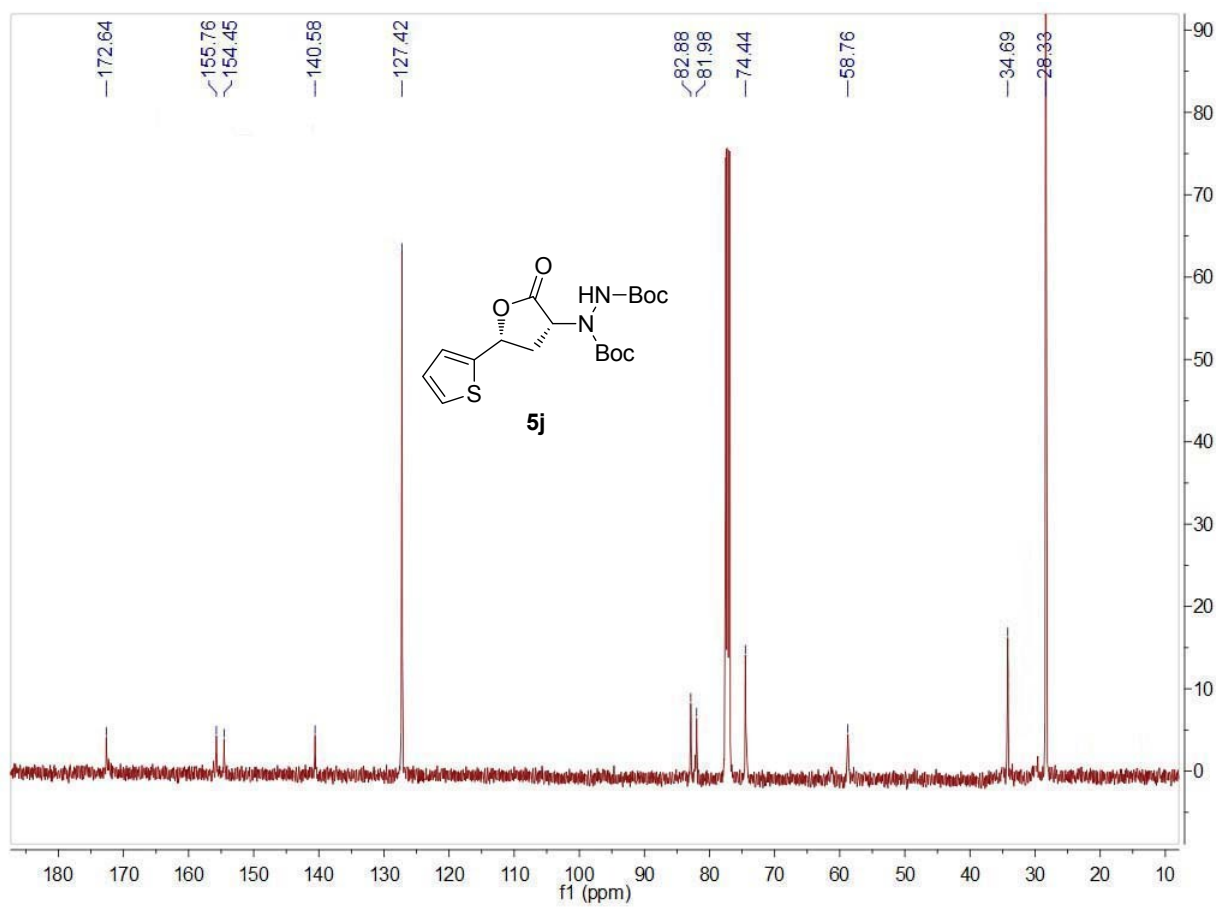
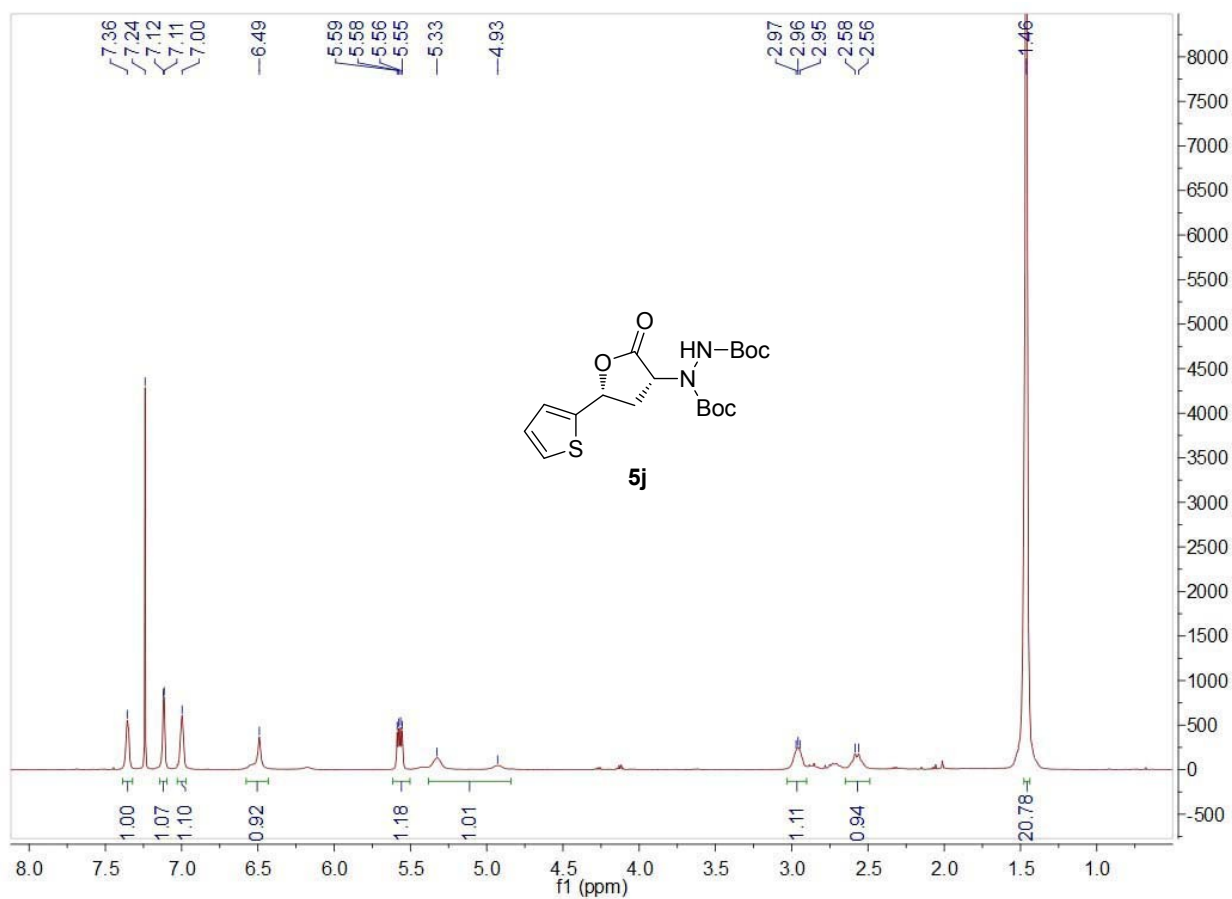


Chrom Type: Fixed WL Chromatogram, 210 nm

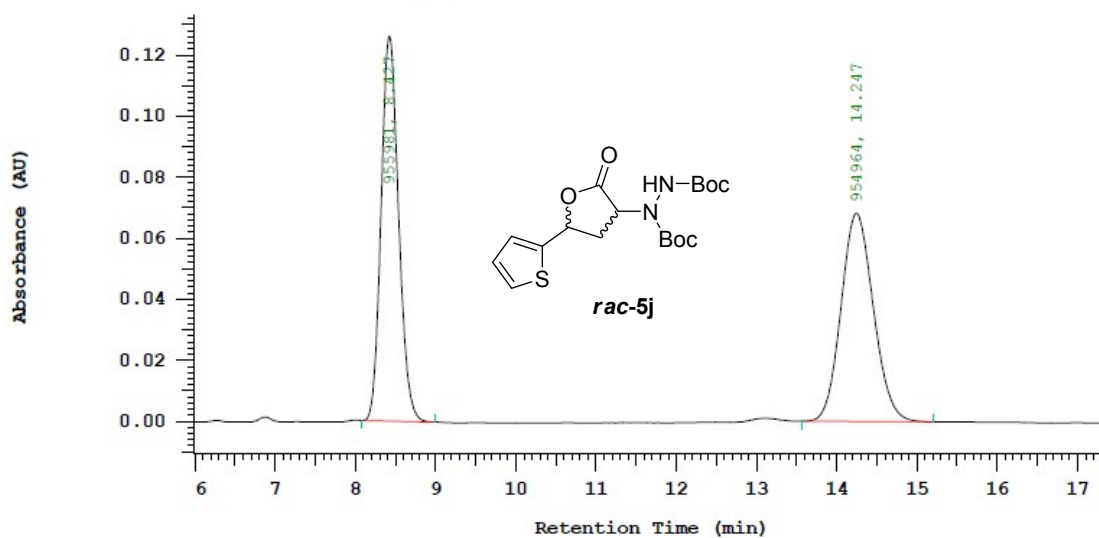
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.500	115182	3.043	BB
2	9.013	3669465	96.957	BB
3784647			100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



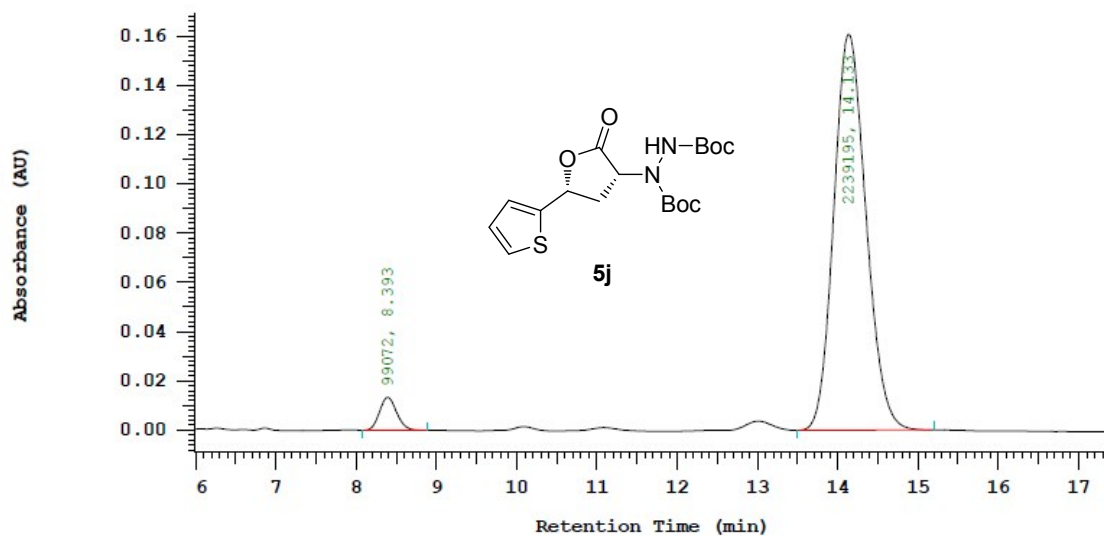
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.427	955981	50.027	BB
2	14.247	954964	49.973	BB
		1910945	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

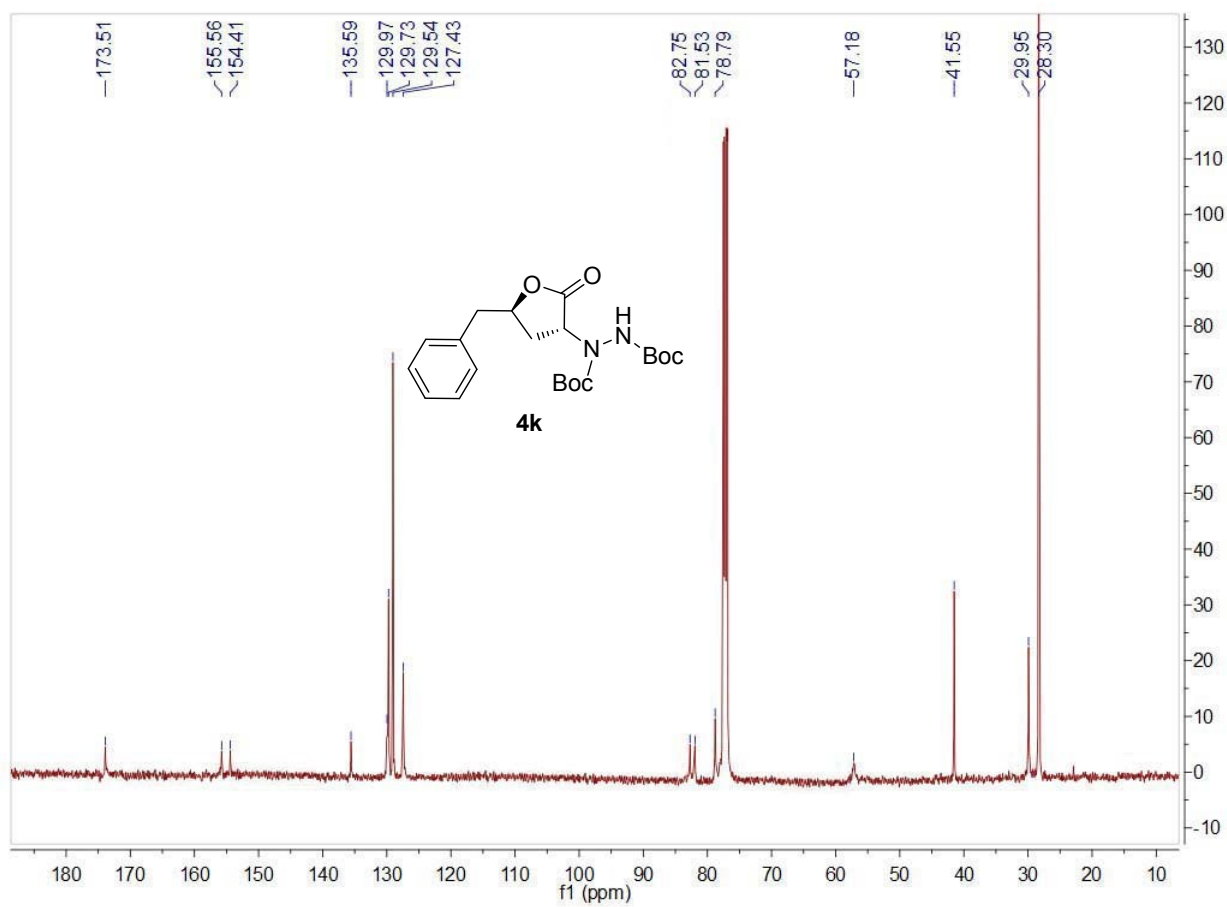
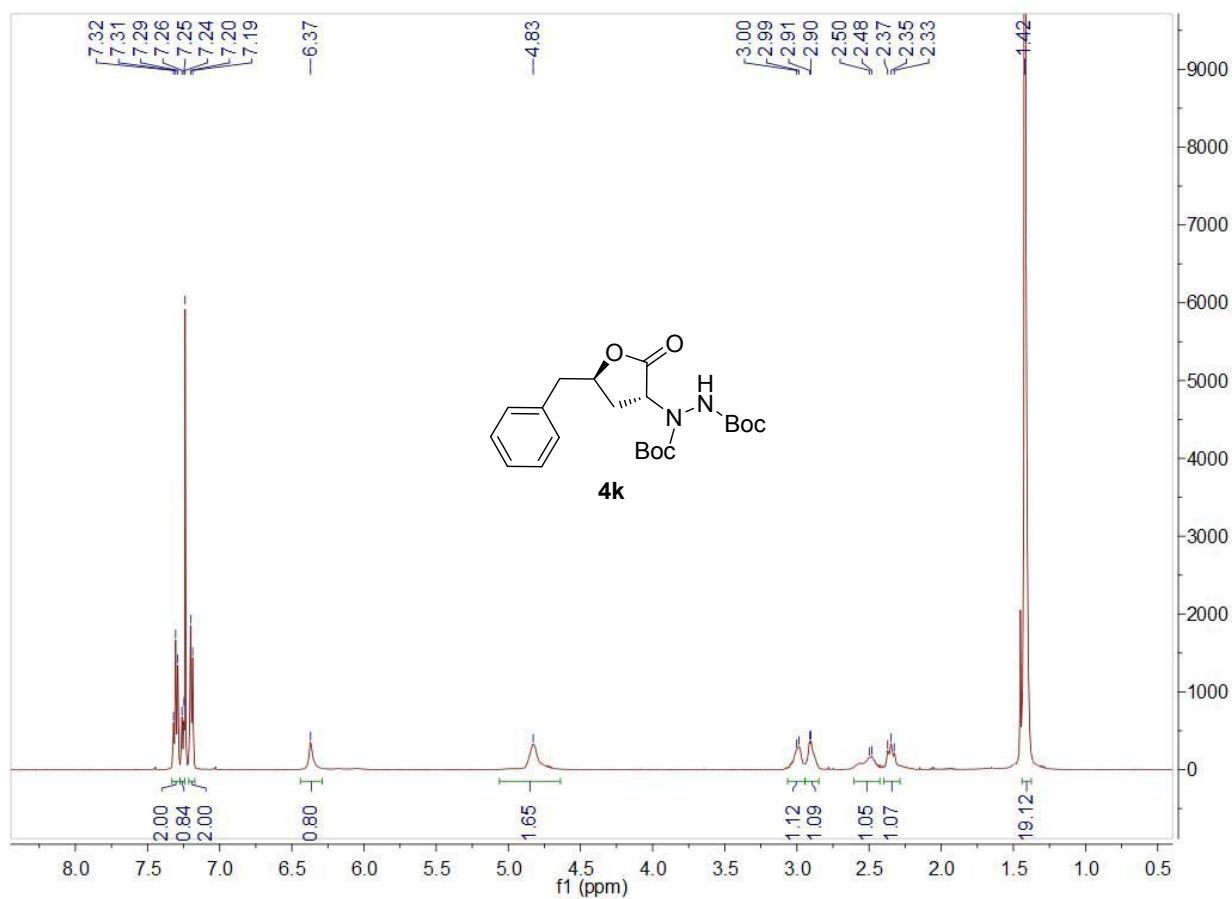


Chrom Type: Fixed WL Chromatogram, 210 nm

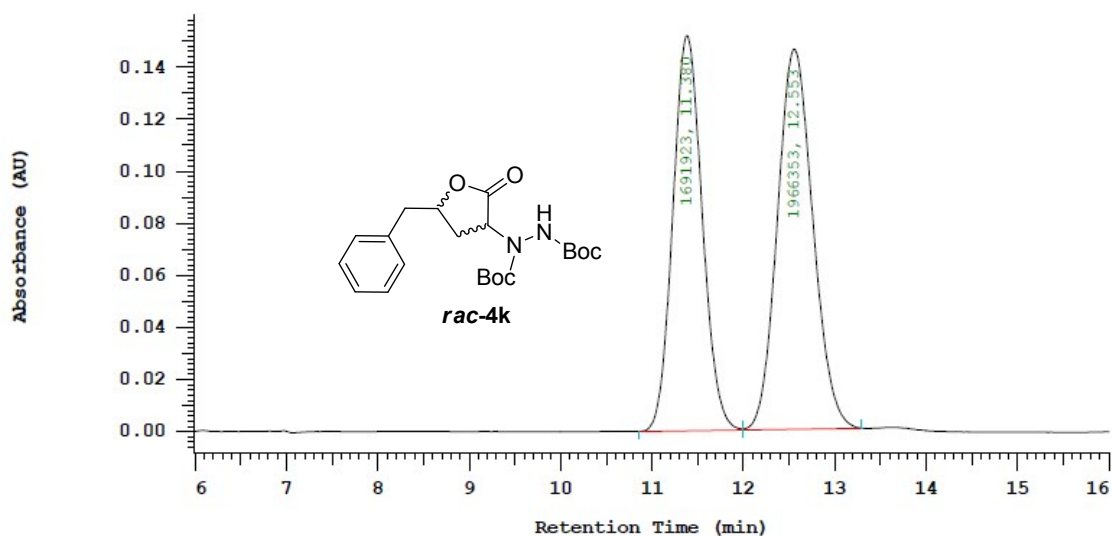
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.393	99072	4.237	BB
2	14.133	2239195	95.763	BB
		2338267	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



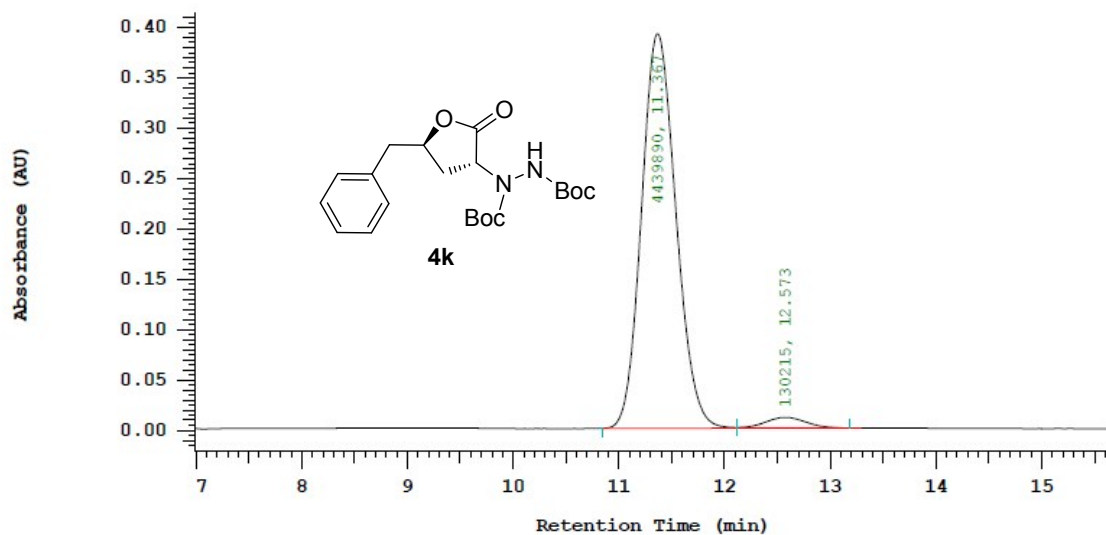
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.380	1691923	46.249	BV
2	12.553	1966353	53.751	VB
		3658276	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

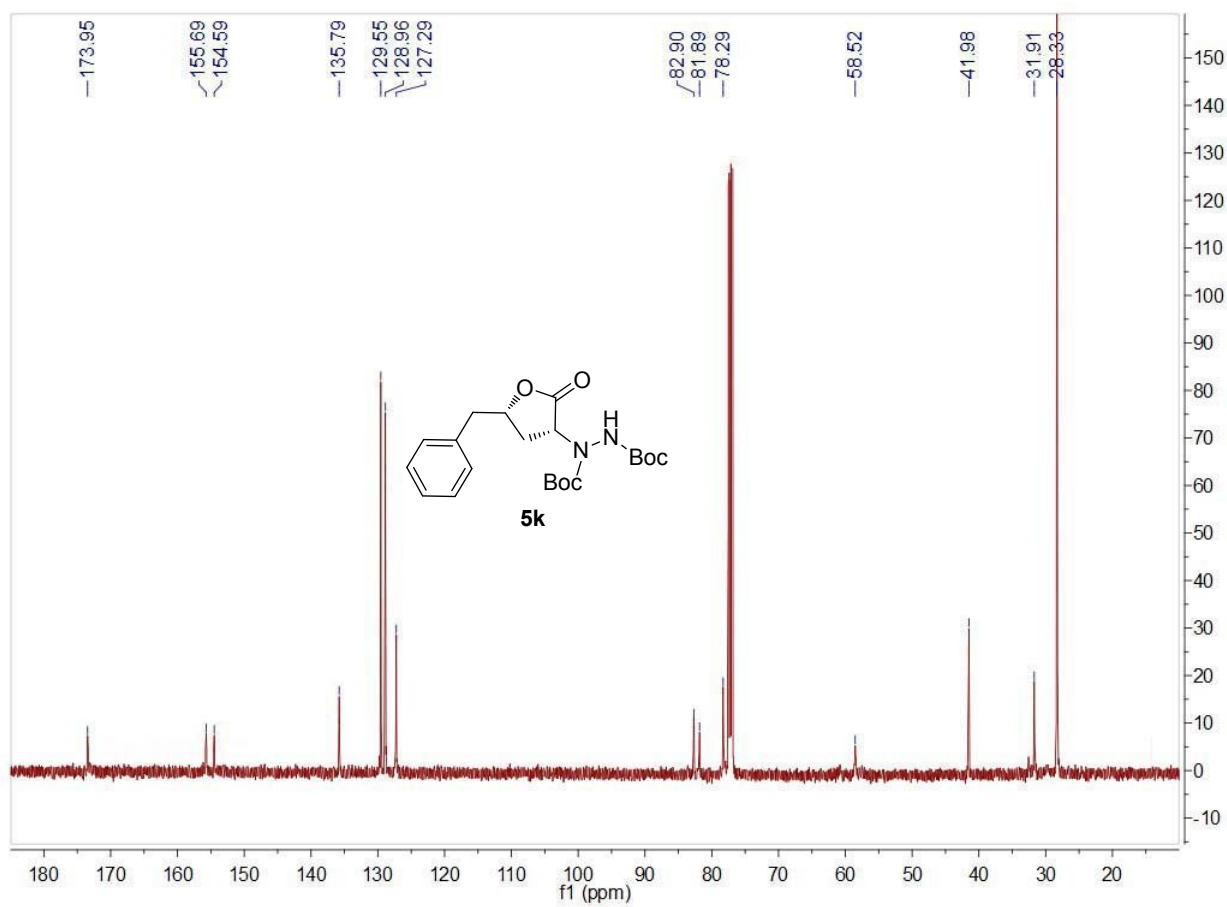
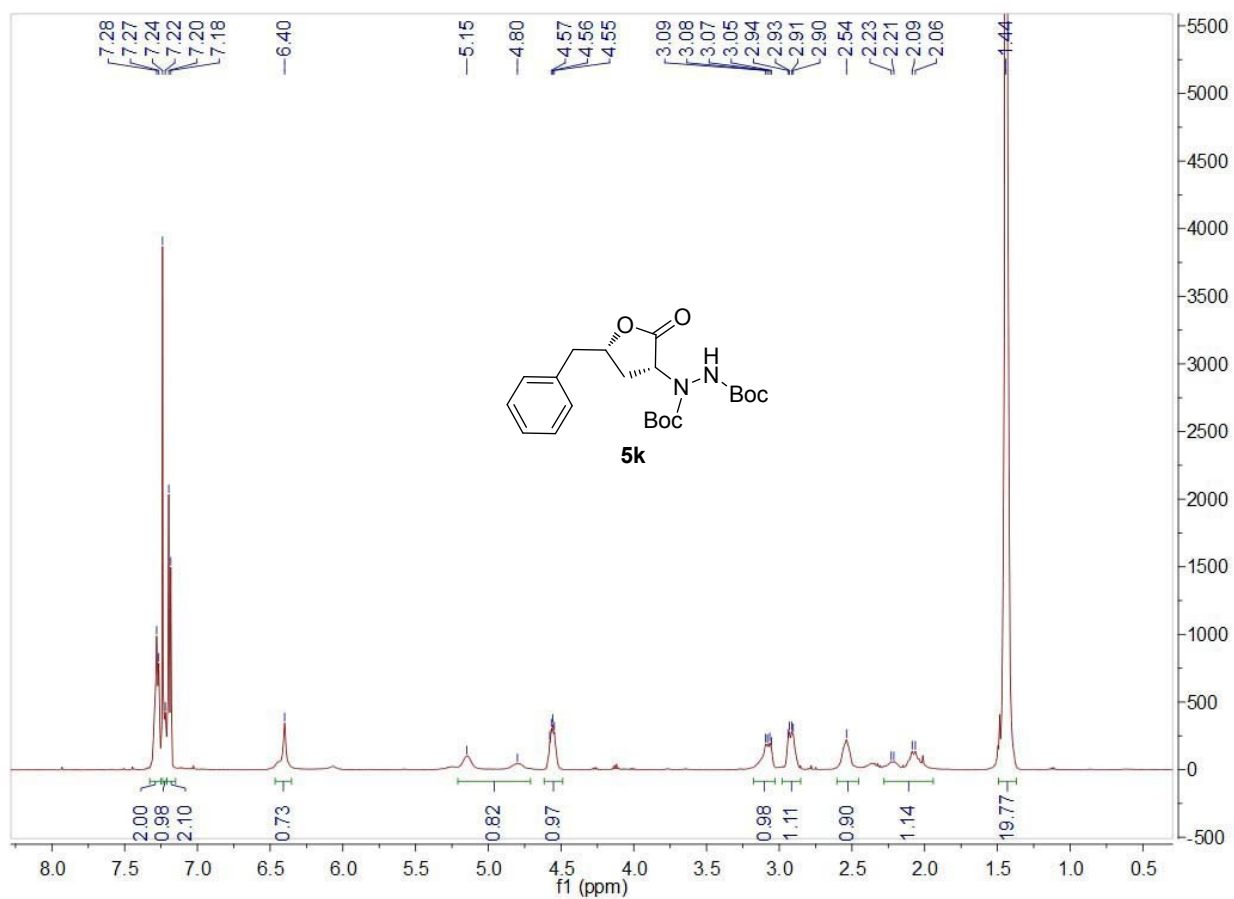


Chrom Type: Fixed WL Chromatogram, 210 nm

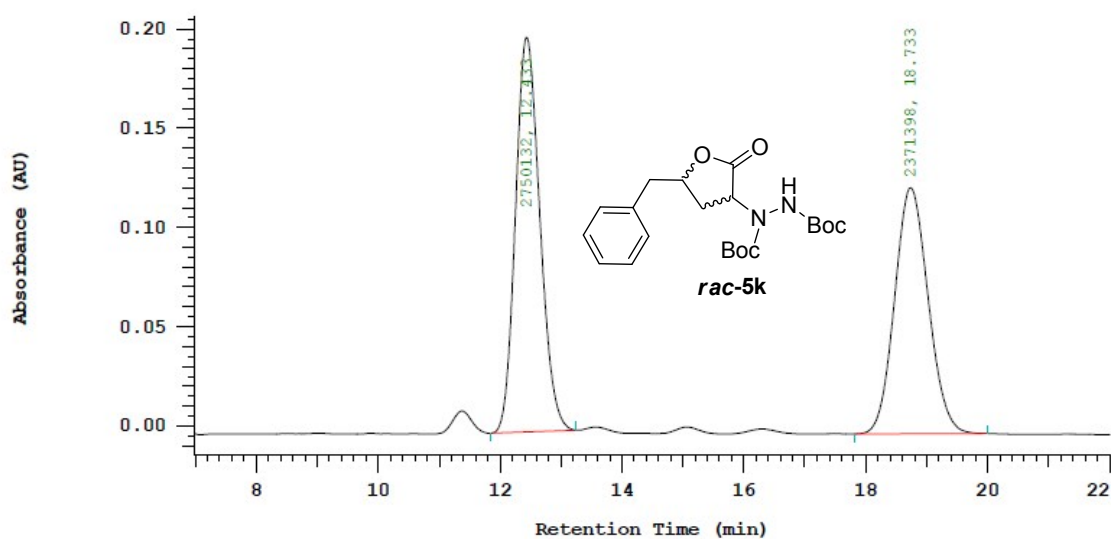
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.367	4439890	97.151	BV
2	12.573	130215	2.849	TBB
		4570105	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



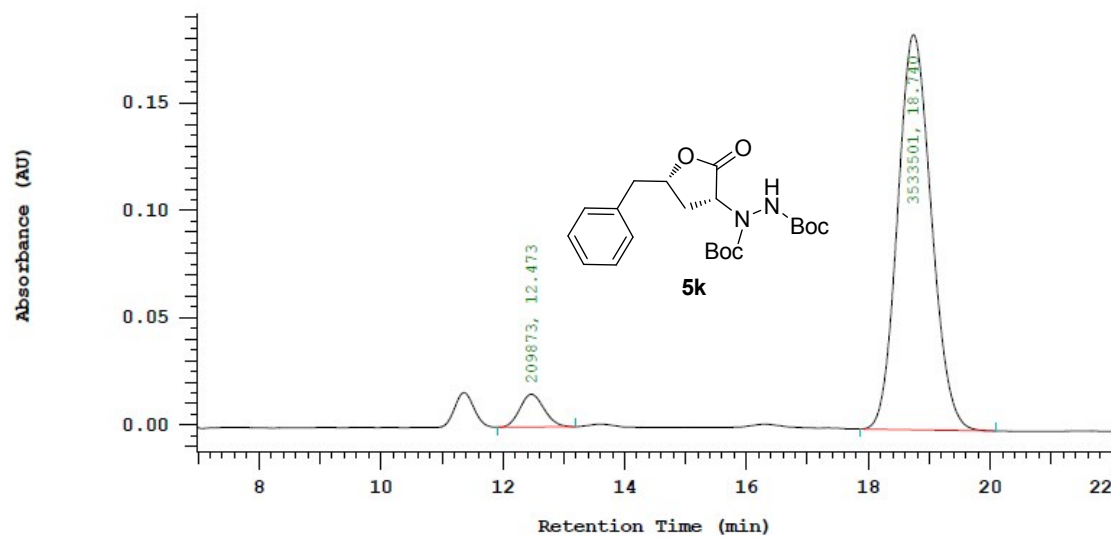
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	12.433	2750132	53.697	BB
2	18.733	2371398	46.303	BB
		5121530	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

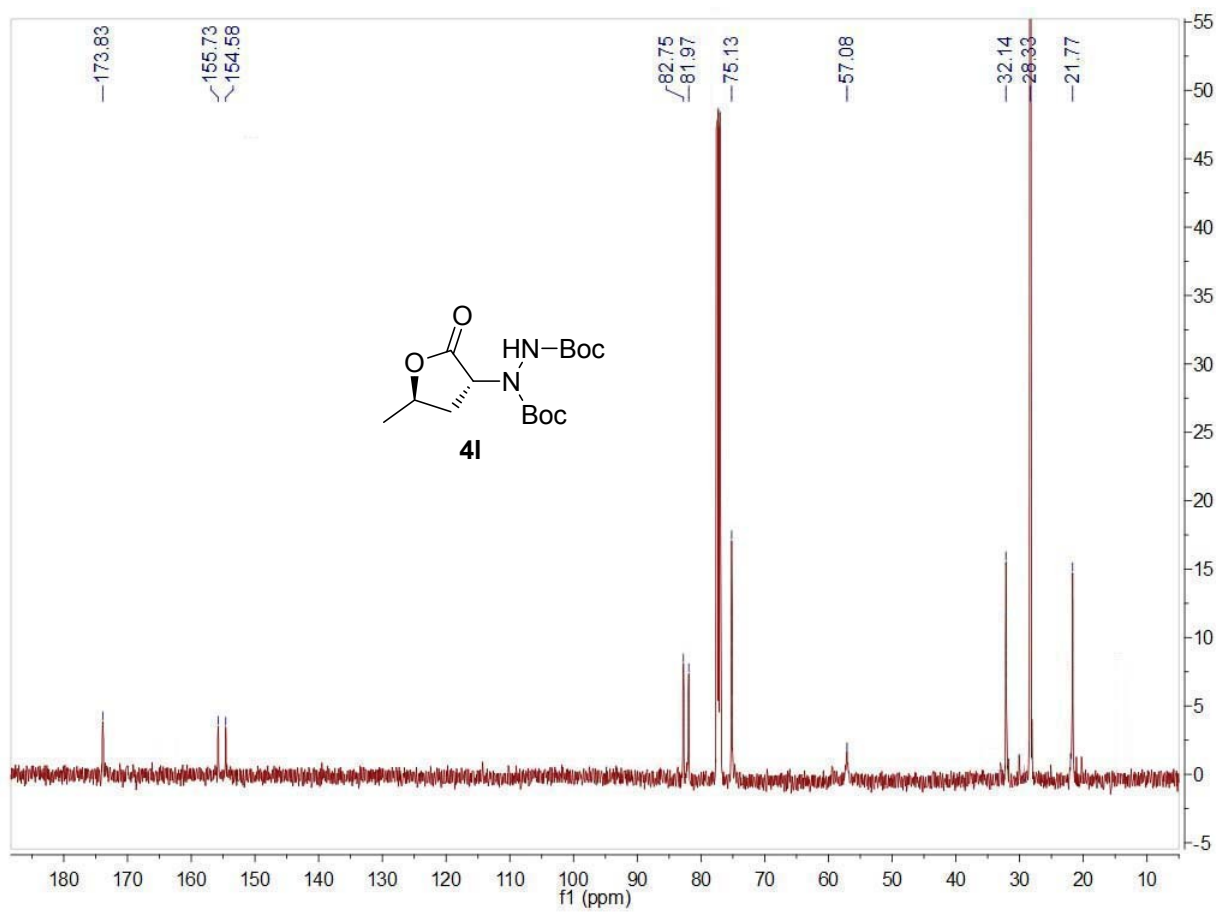
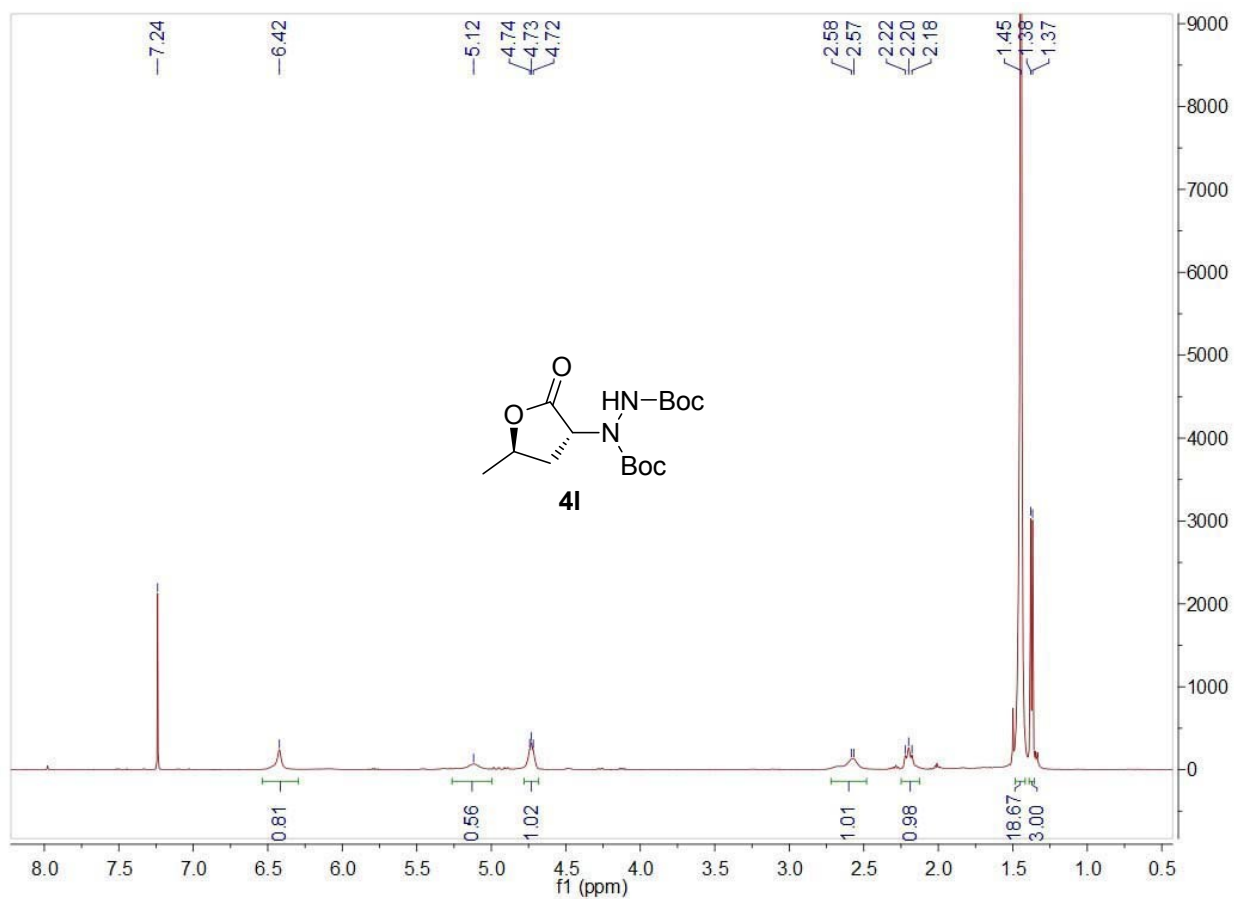


Chrom Type: Fixed WL Chromatogram, 210 nm

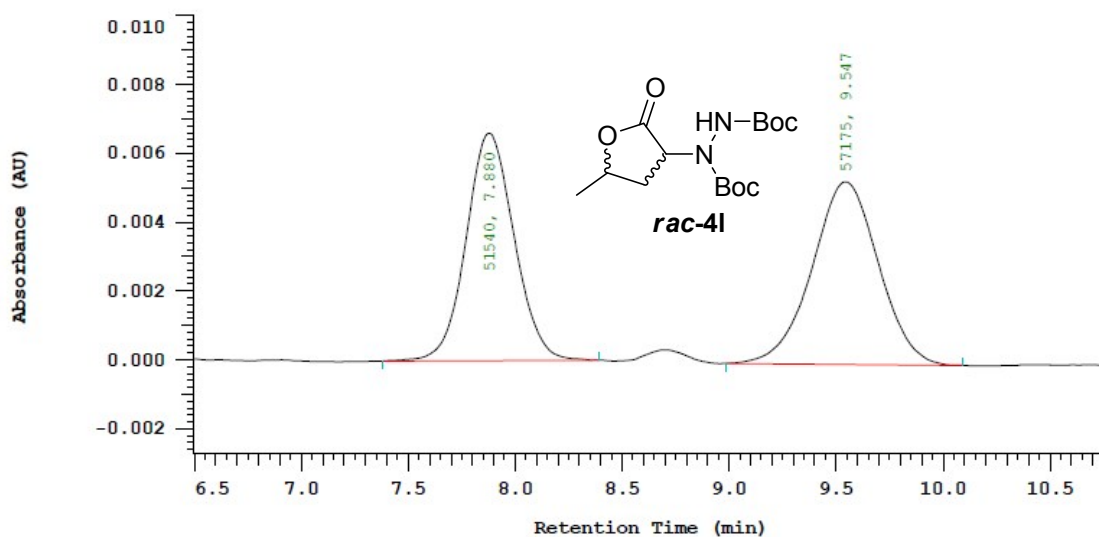
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	12.473	209873	5.607	BB
2	18.740	3533501	94.393	BB
		3743374	100.000	



Chrom Type: Fixed WL Chromatogram, 230 nm



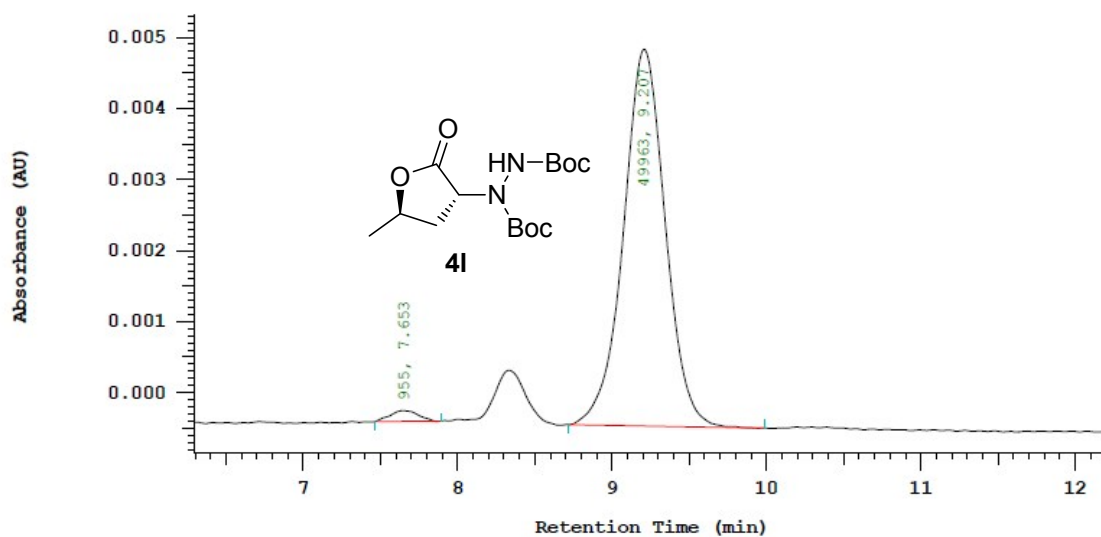
Chrom Type: Fixed WL Chromatogram, 230 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.880	51540	47.408	BB
2	9.547	57175	52.592	BB
		108715	100.000	

Chrom Type: Fixed WL Chromatogram, 230 nm

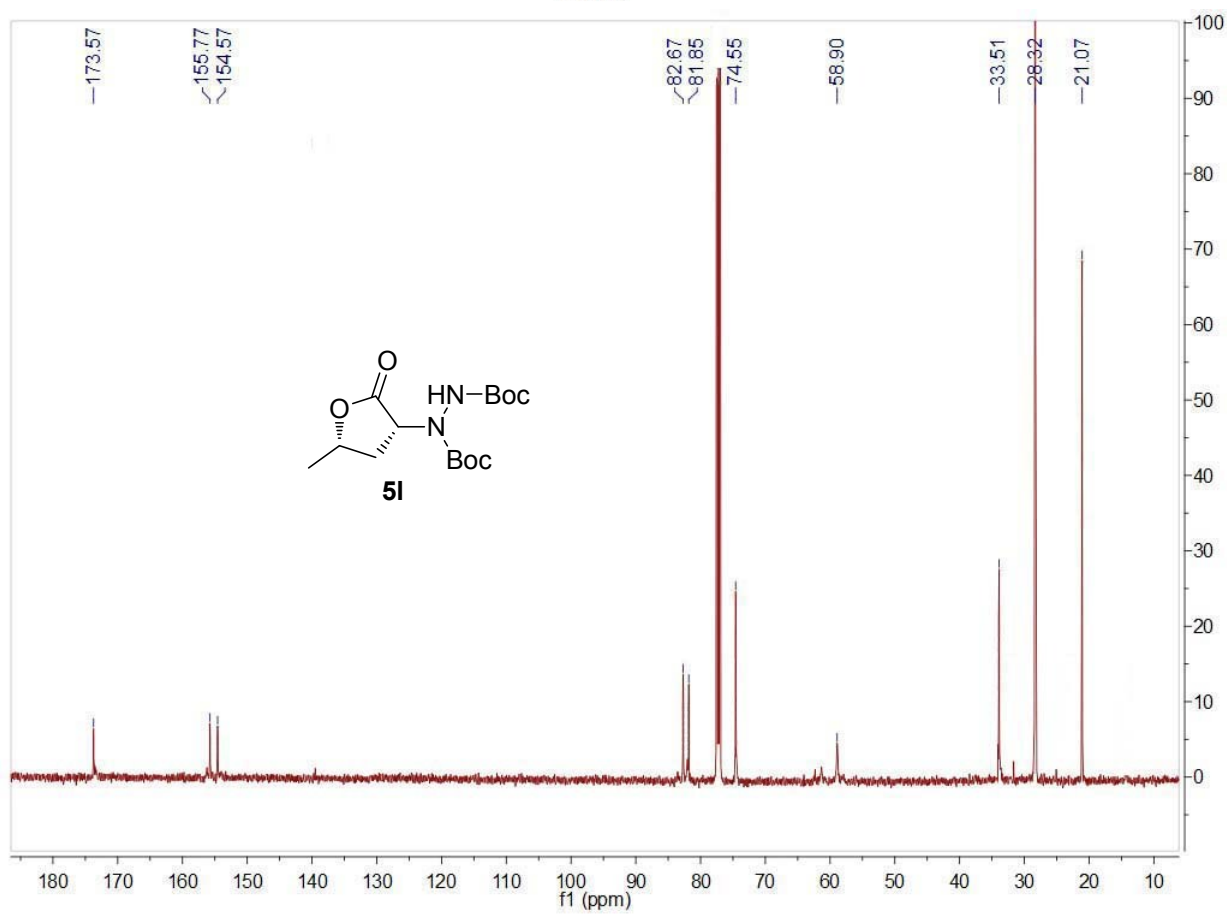
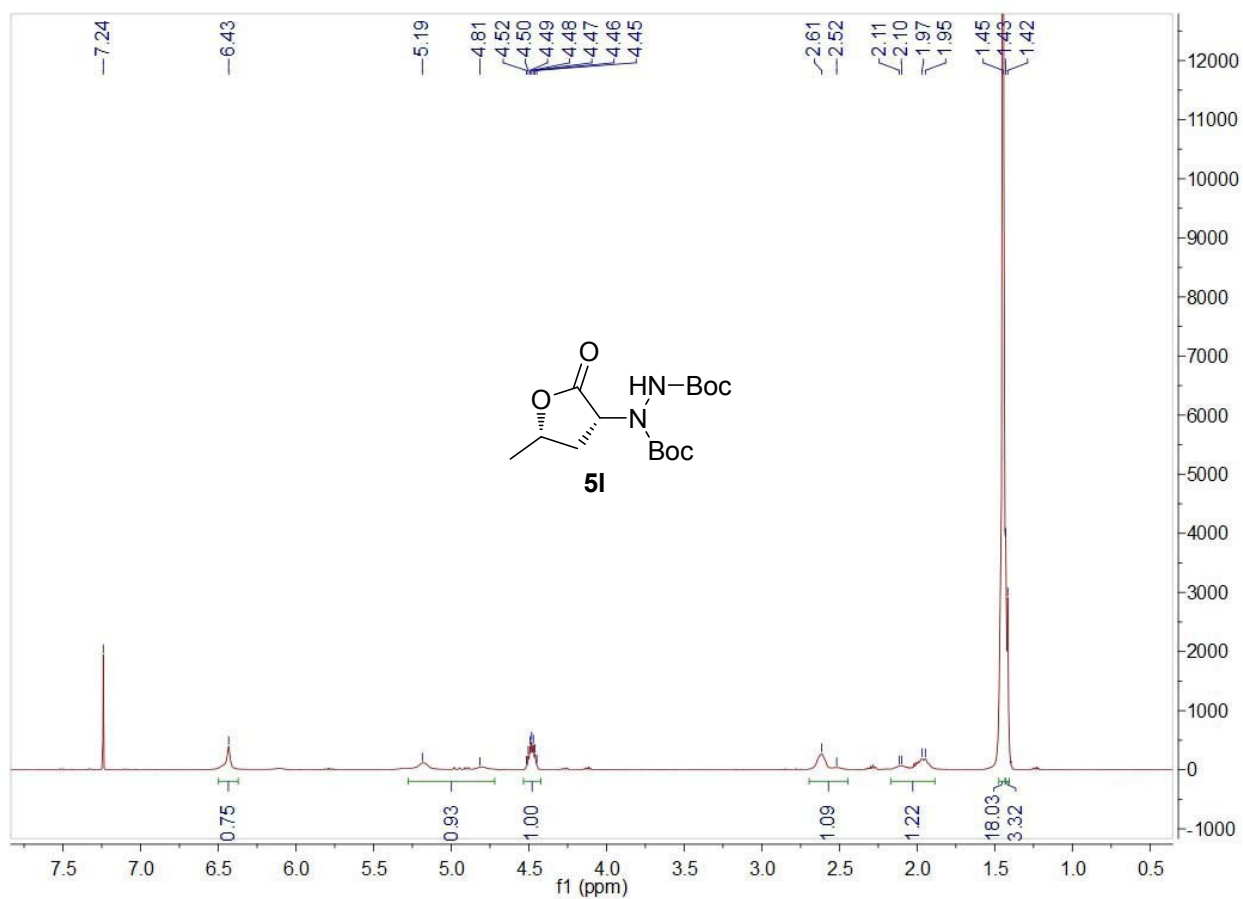


Chrom Type: Fixed WL Chromatogram, 230 nm

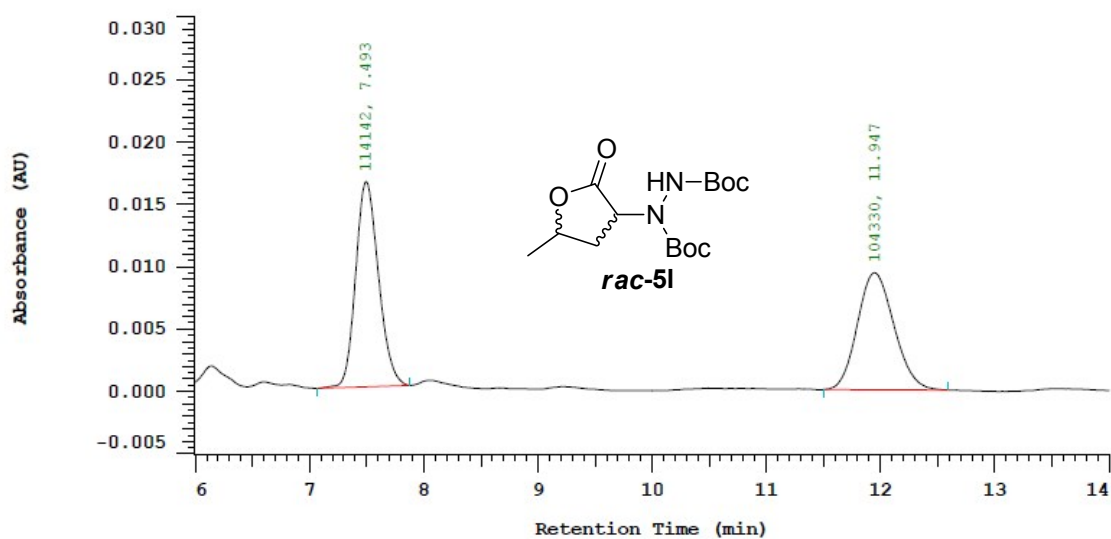
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.653	955	1.877	BB
2	9.207	49963	98.123	BB
		50918	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



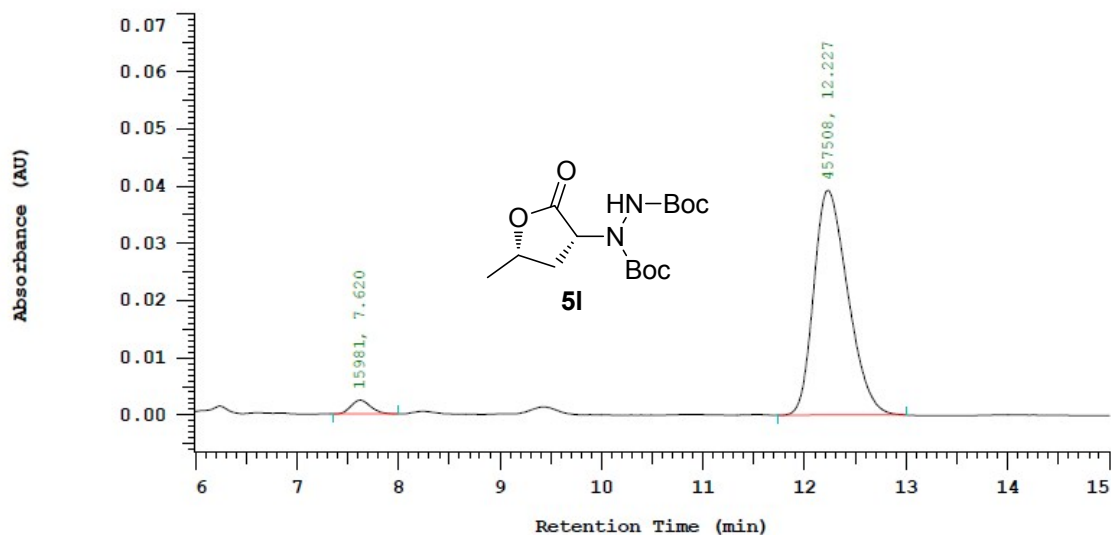
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.493	114142	52.246	BB
2	11.947	104330	47.754	BB
		218472	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

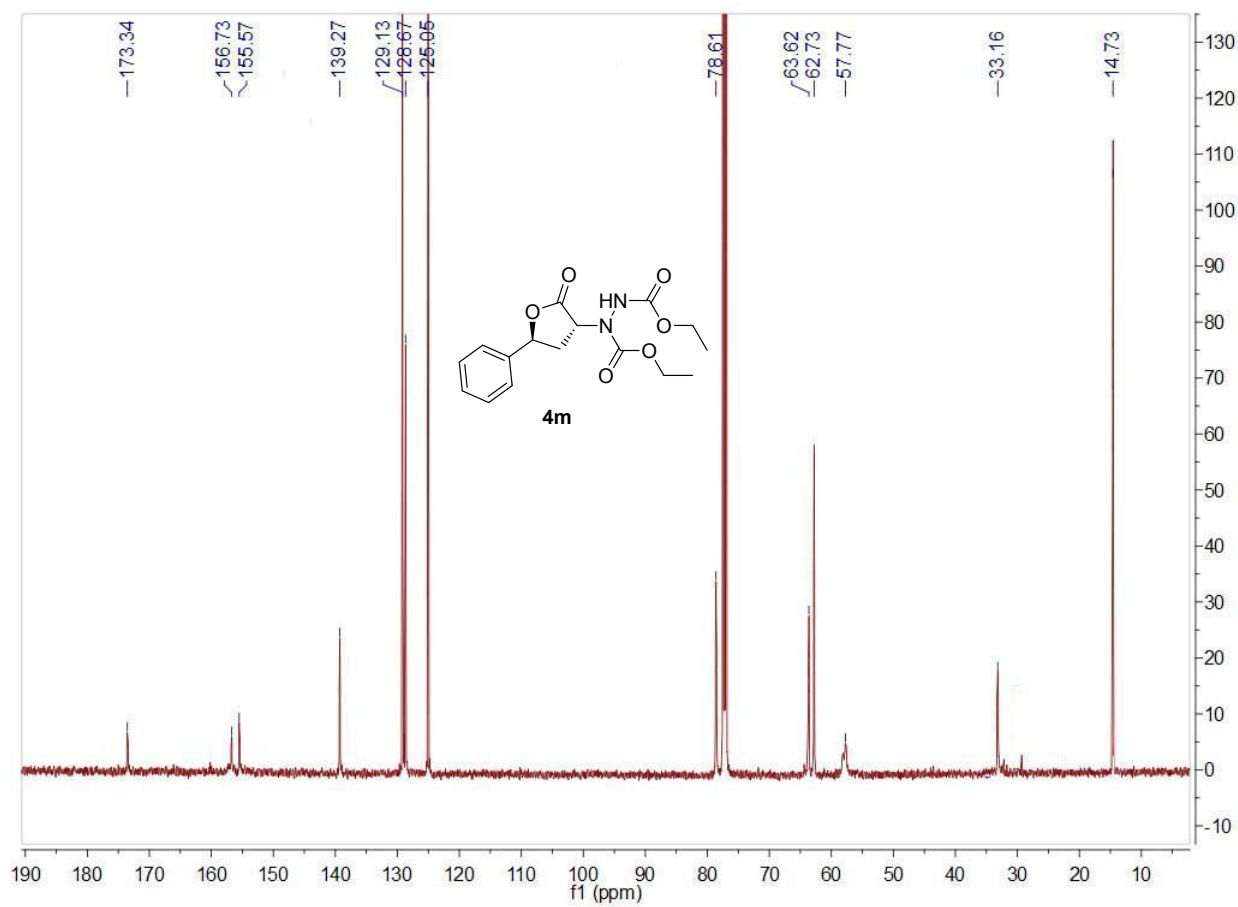
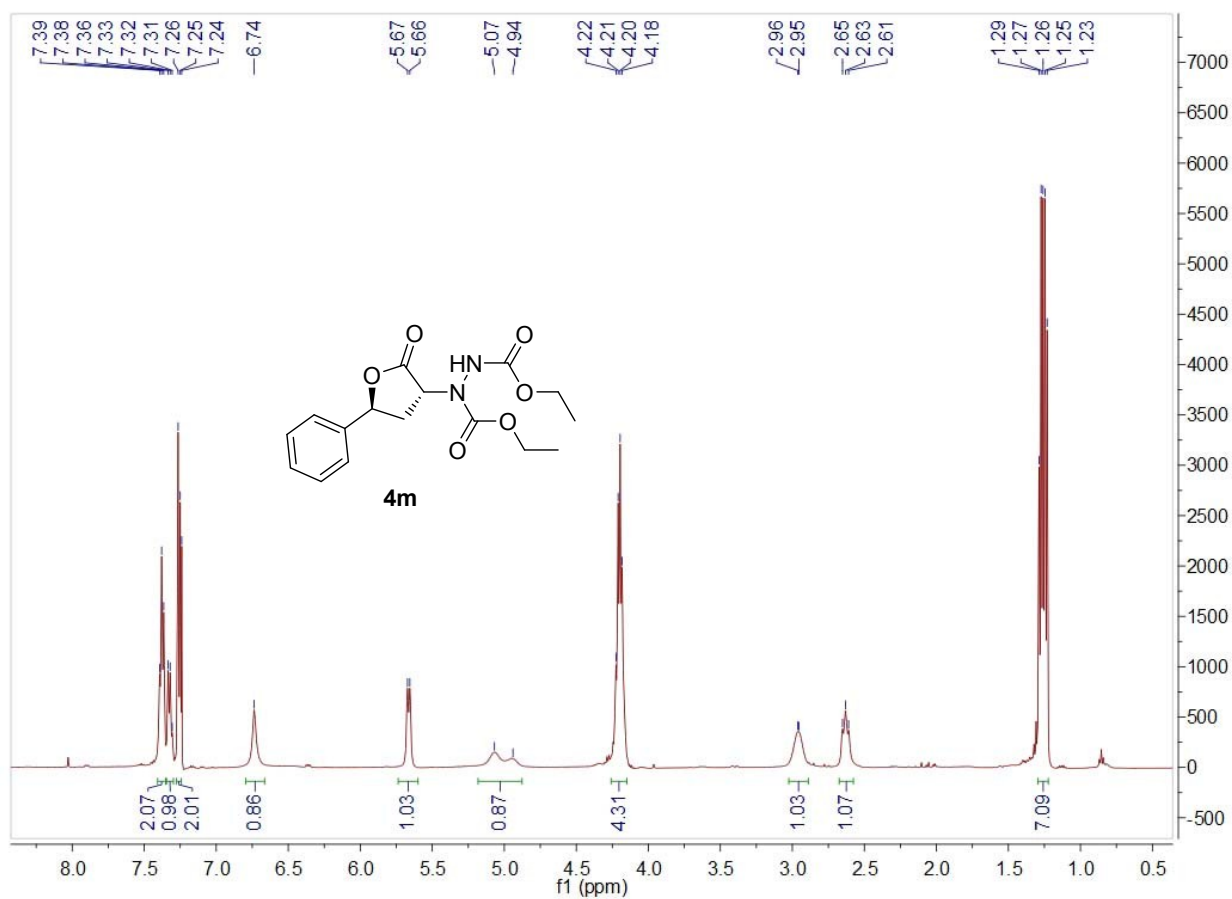


Chrom Type: Fixed WL Chromatogram, 210 nm

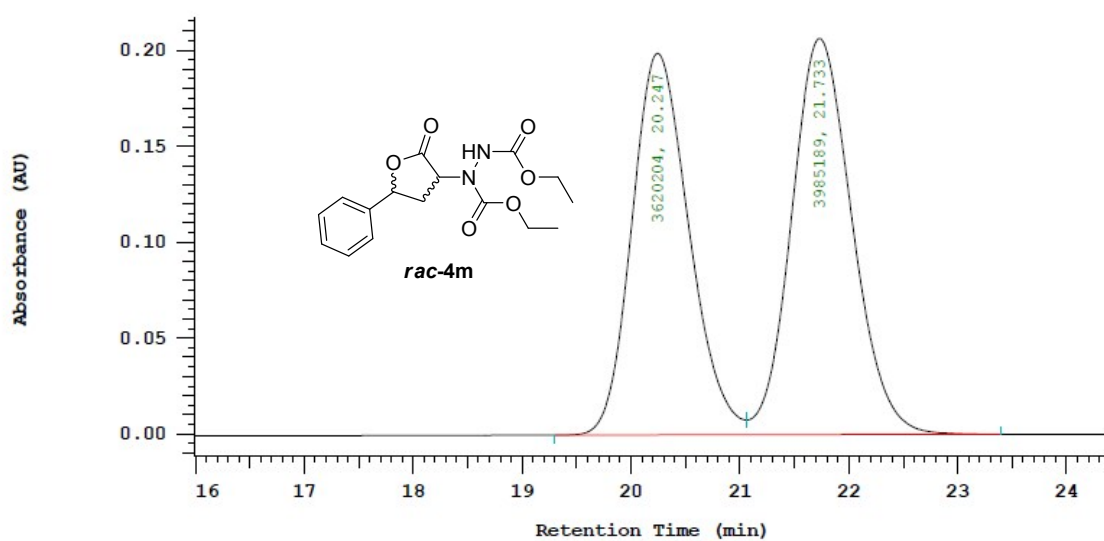
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.620	15981	3.375	BB
2	12.227	457508	96.625	BB
		473489	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



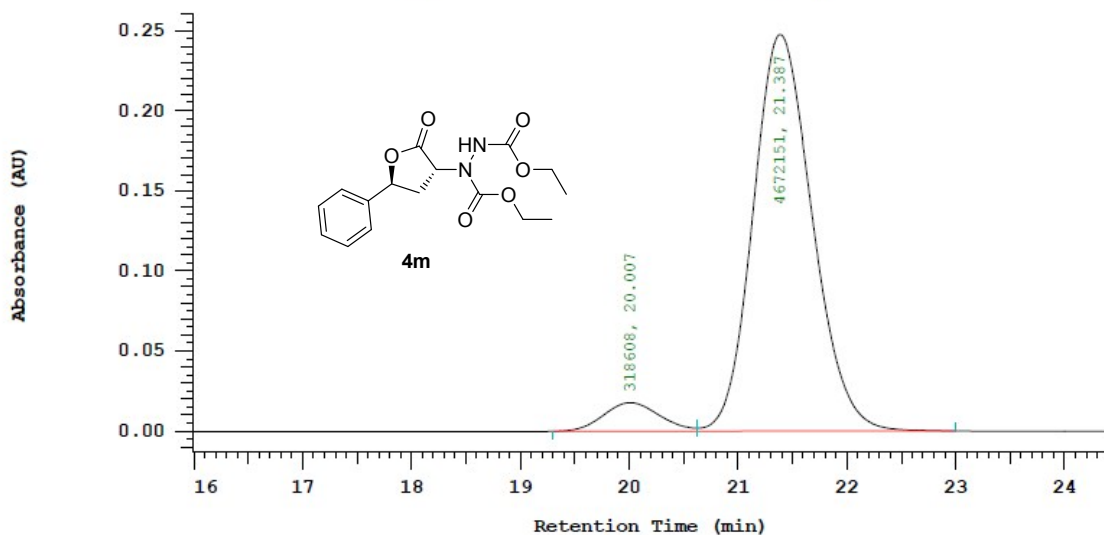
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	20.247	3620204	47.600	BV
2	21.733	3985189	52.400	VB
		7605393	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



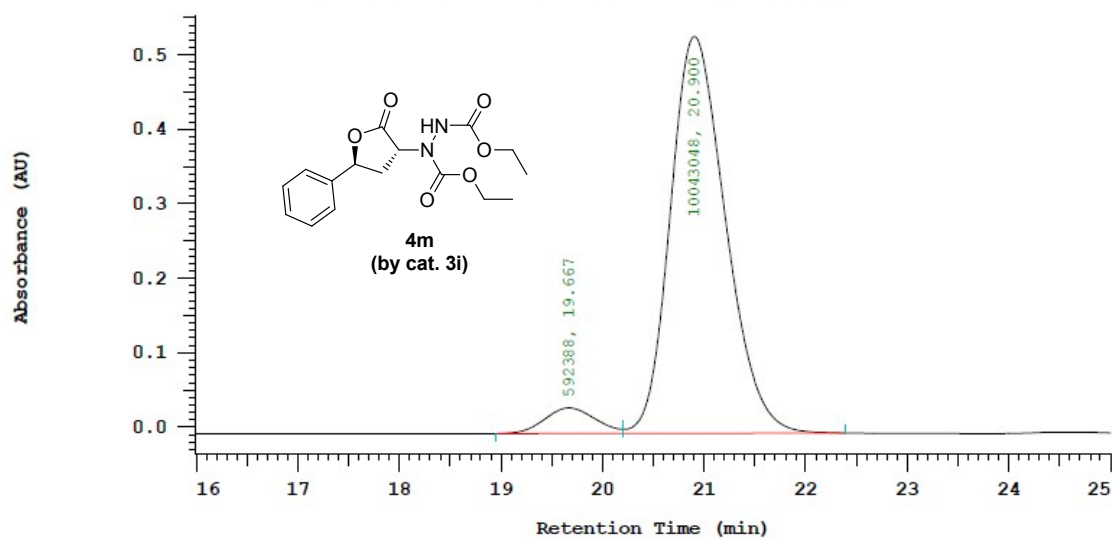
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	20.007	318608	6.384	BV
2	21.387	4672151	93.616	VB
		4990759	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

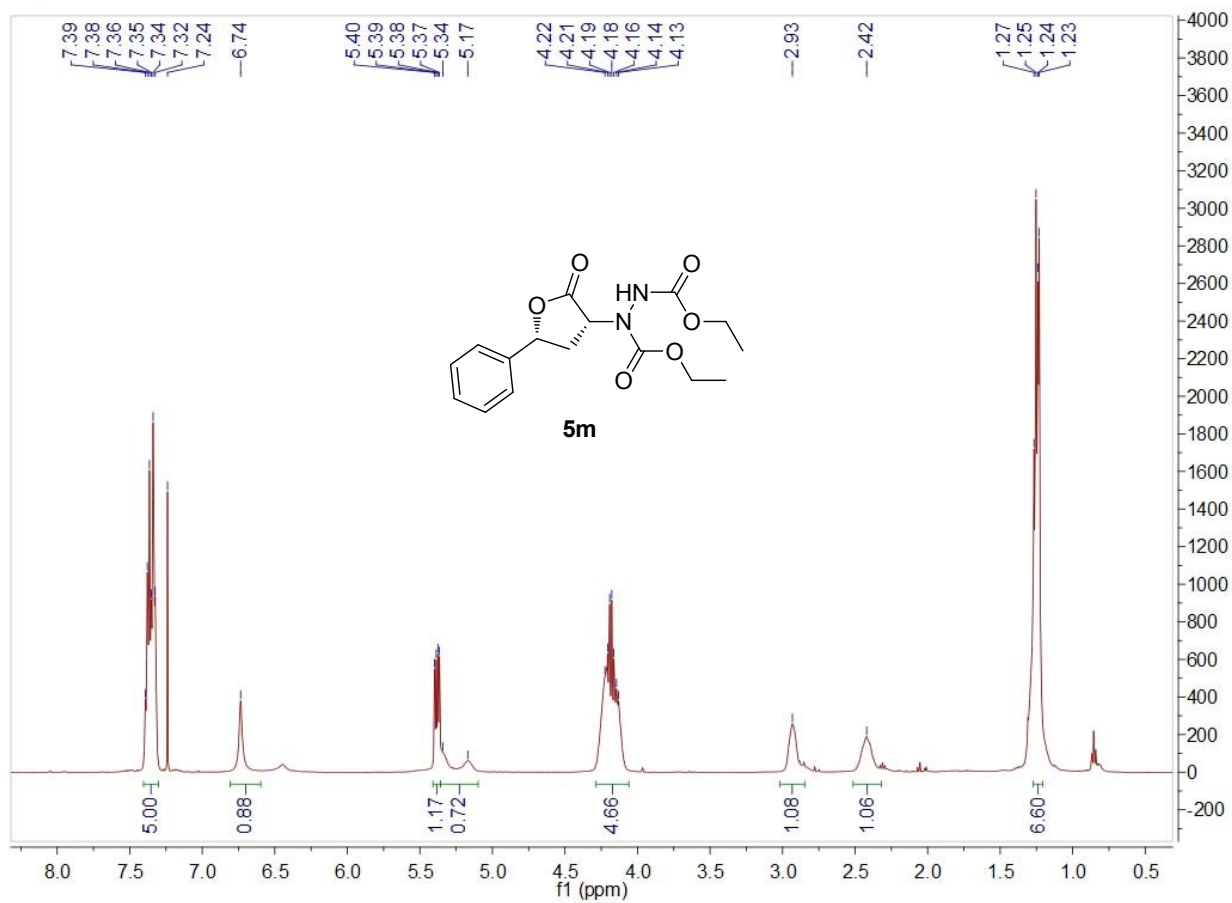


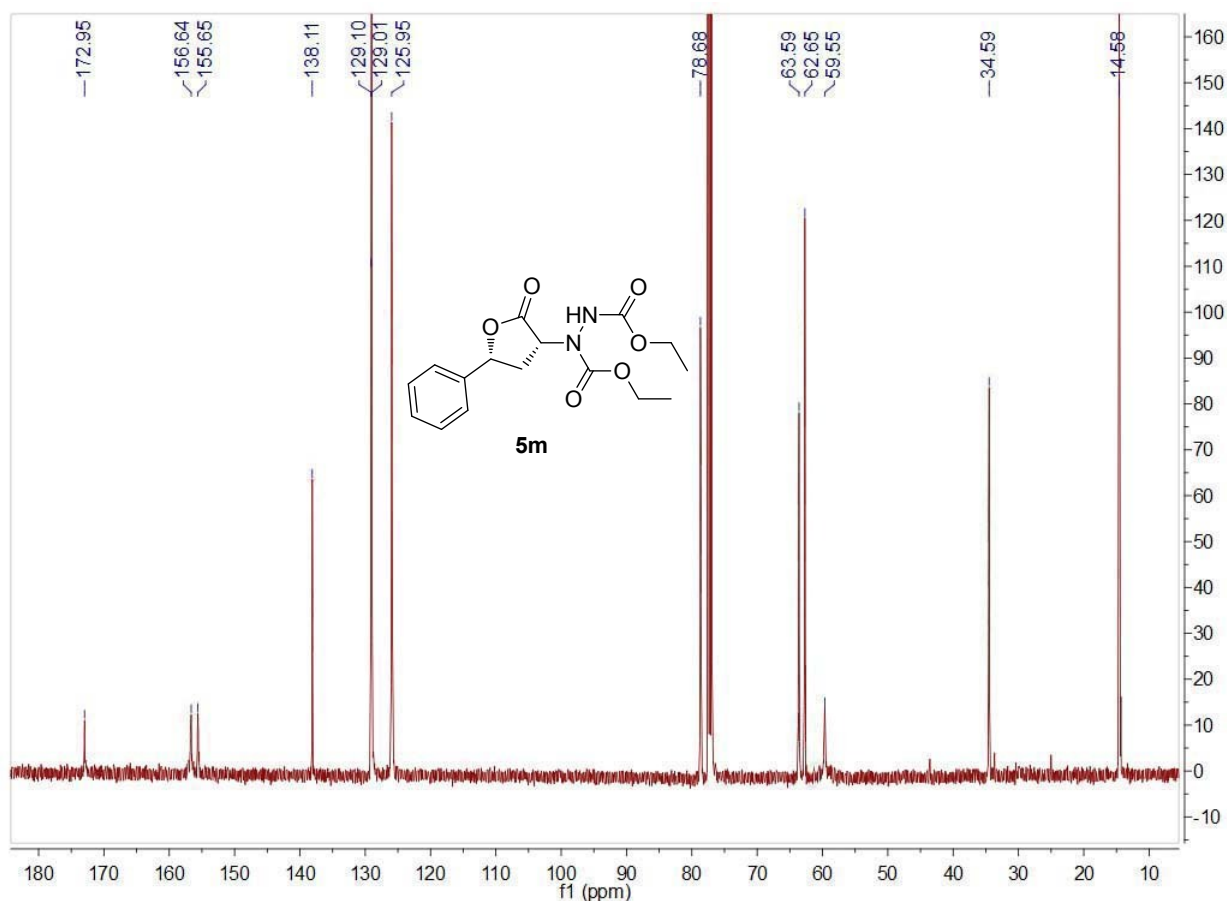
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

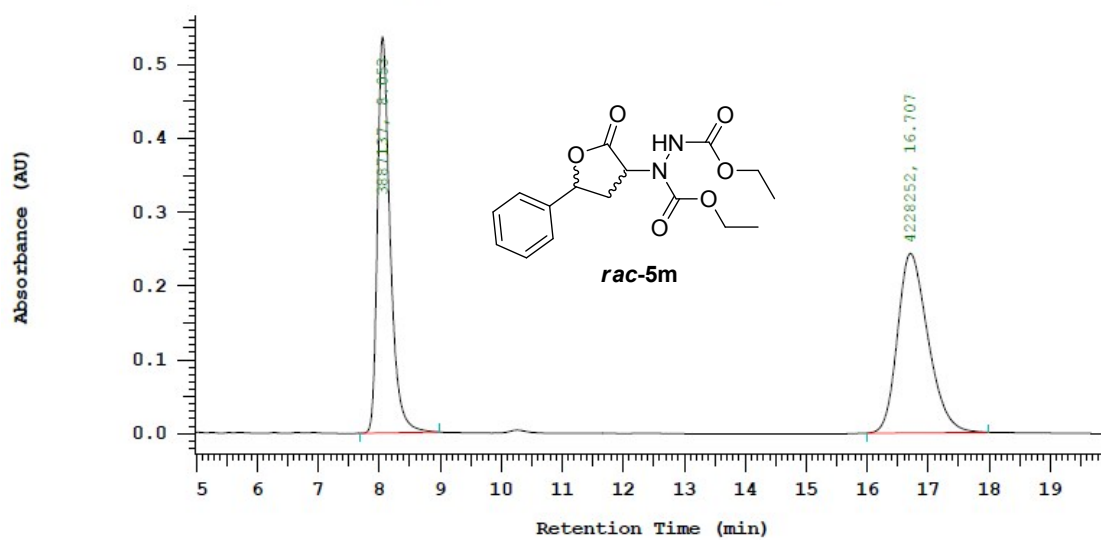
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	19.667	592388	5.570	BV
2	20.900	10043048	94.430	VB
		10635436	100.000	





Chrom Type: Fixed WL Chromatogram, 210 nm



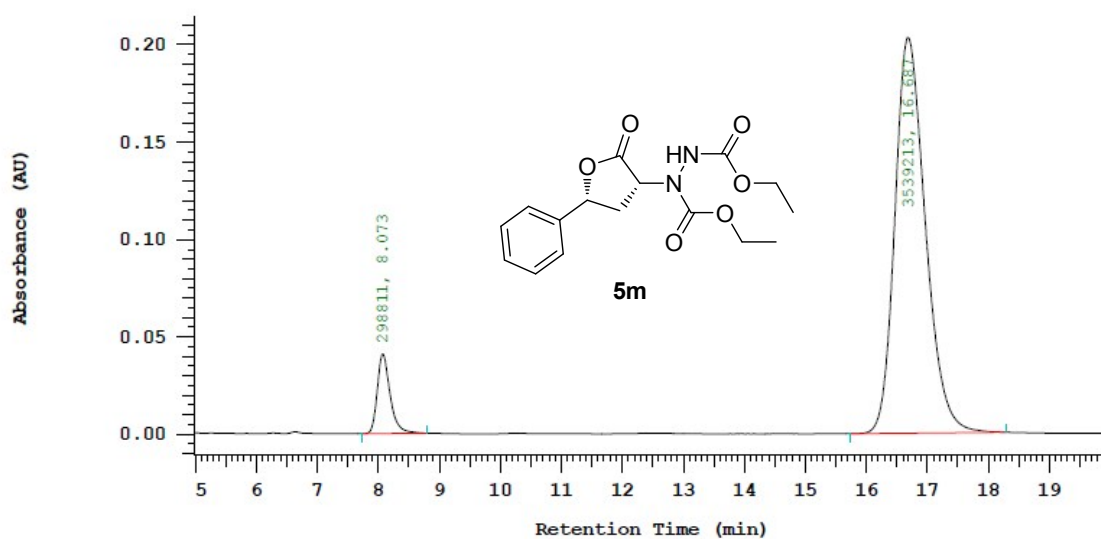
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.053	3887137	47.898	BB
2	16.707	4228252	52.102	BB
		8115389	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



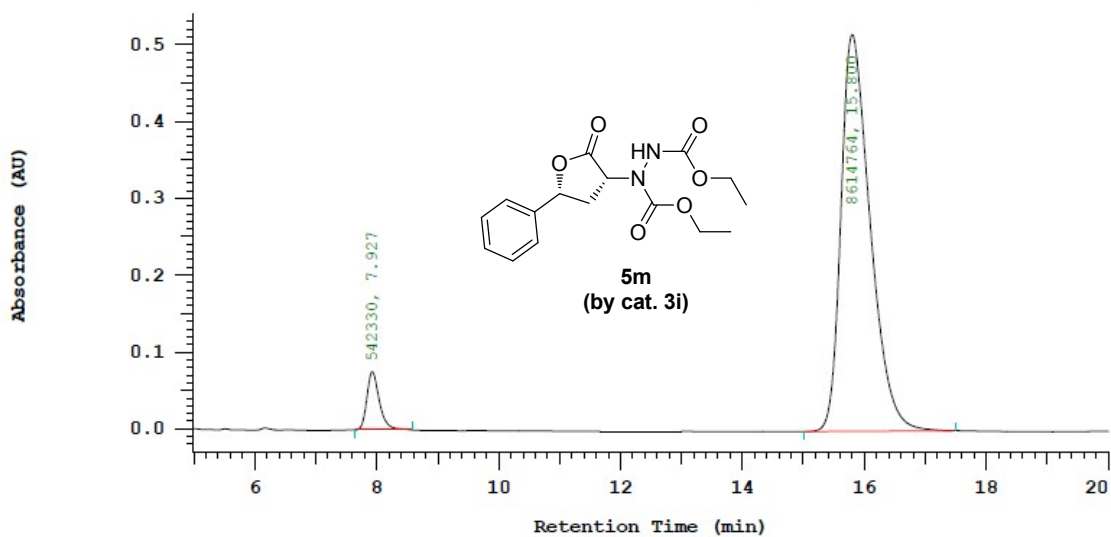
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.073	298811	7.786	BB
2	16.687	3539213	92.214	BB
		3838024	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

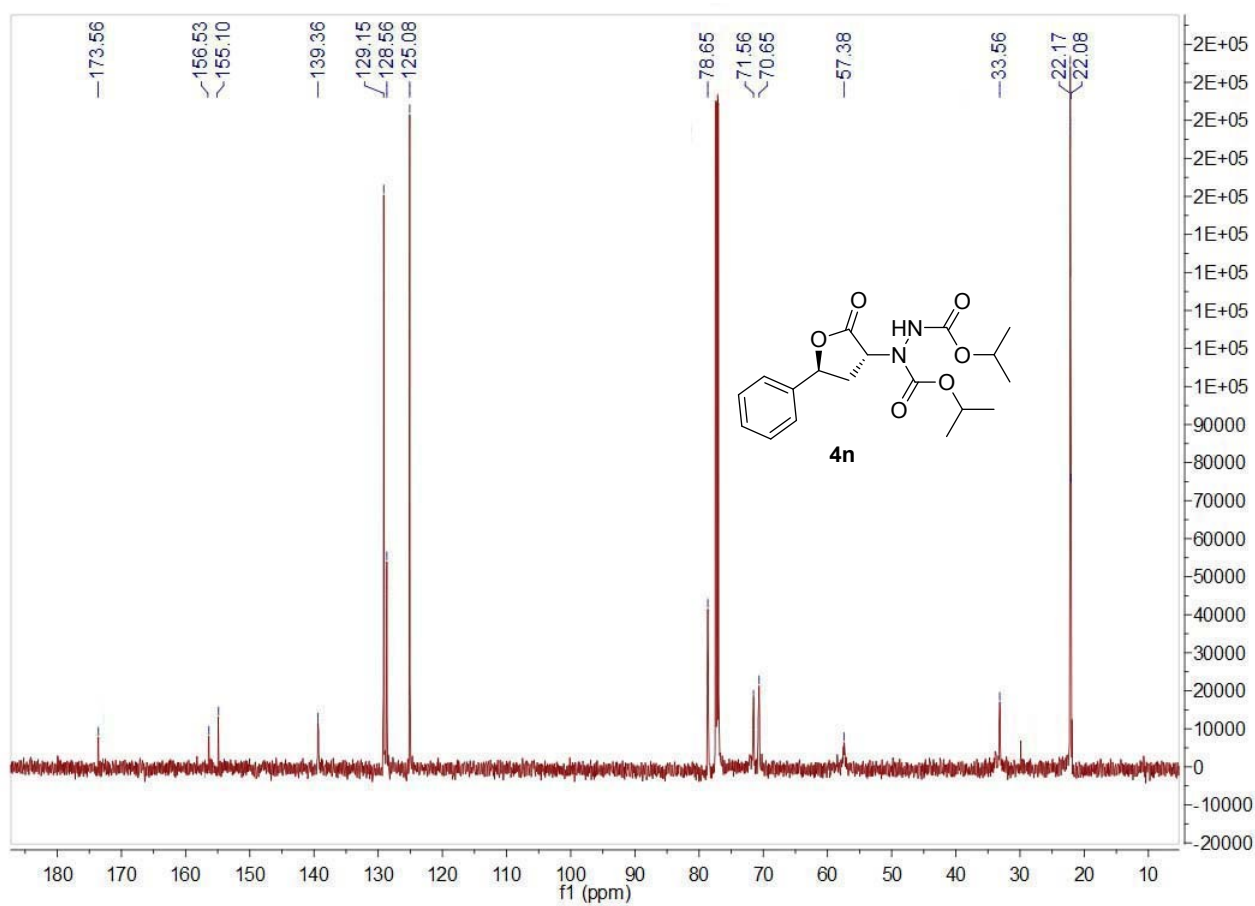
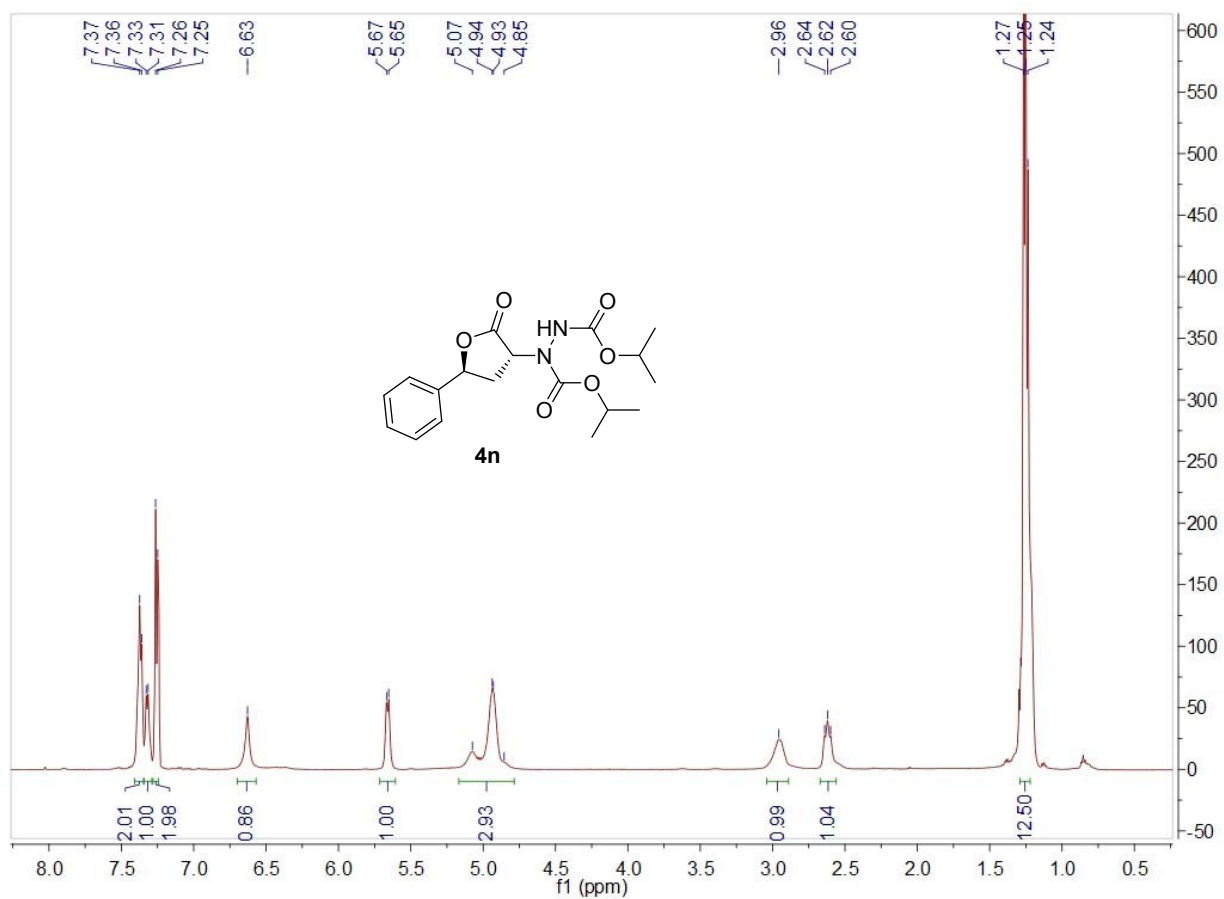


Chrom Type: Fixed WL Chromatogram, 210 nm

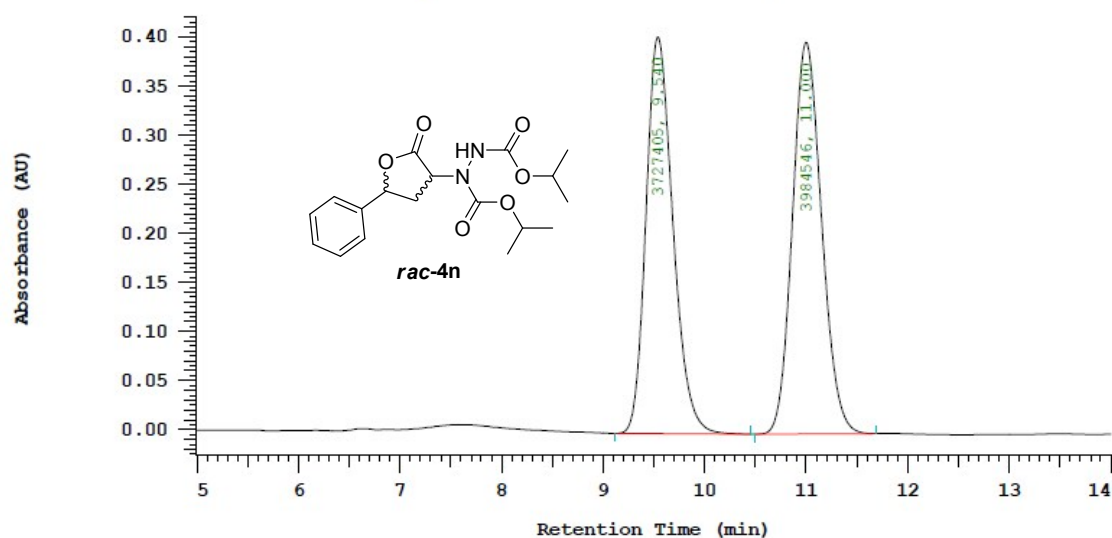
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.927	542330	5.923	BB
2	15.800	8614764	94.077	BB
		9157094	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



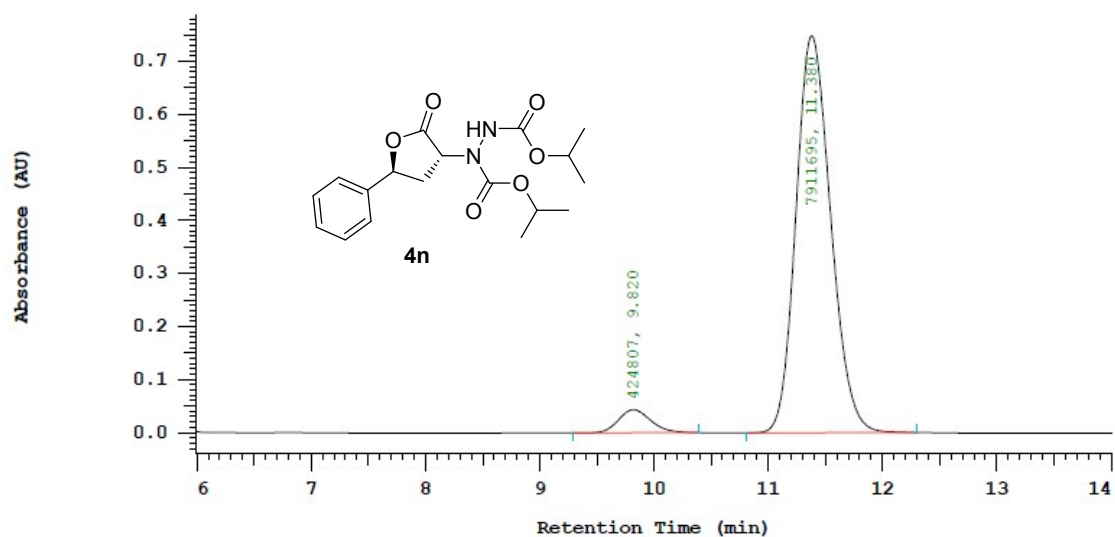
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	9.540	3727405	48.333	BB
2	11.000	3984546	51.667	BB
		7711951	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



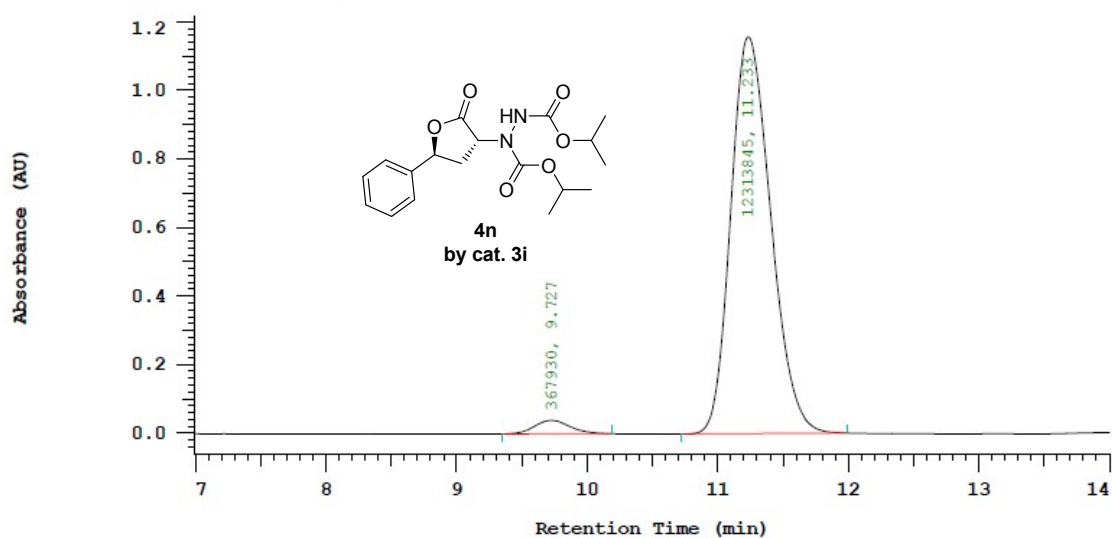
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	9.820	424807	5.096	BB
2	11.380	7911695	94.904	BB
		8336502	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

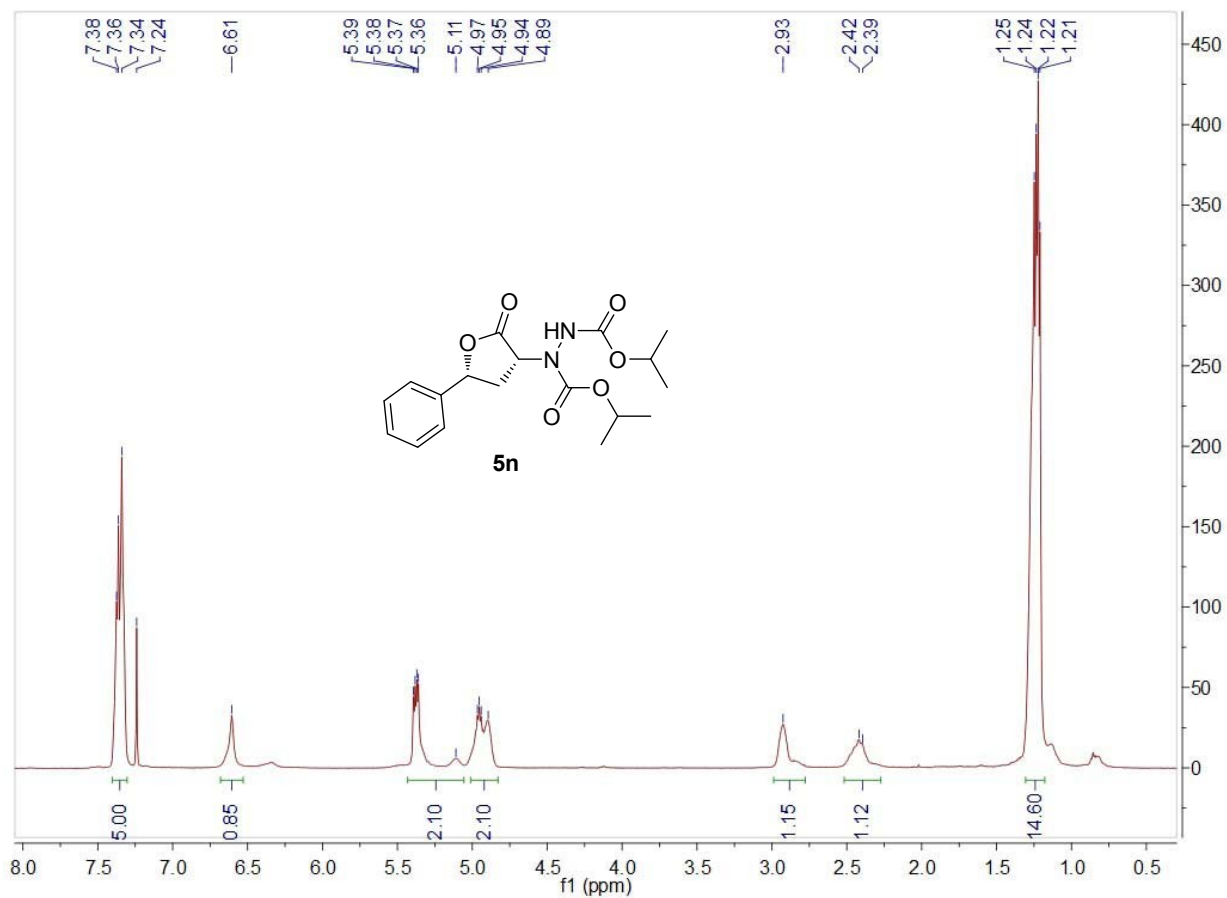


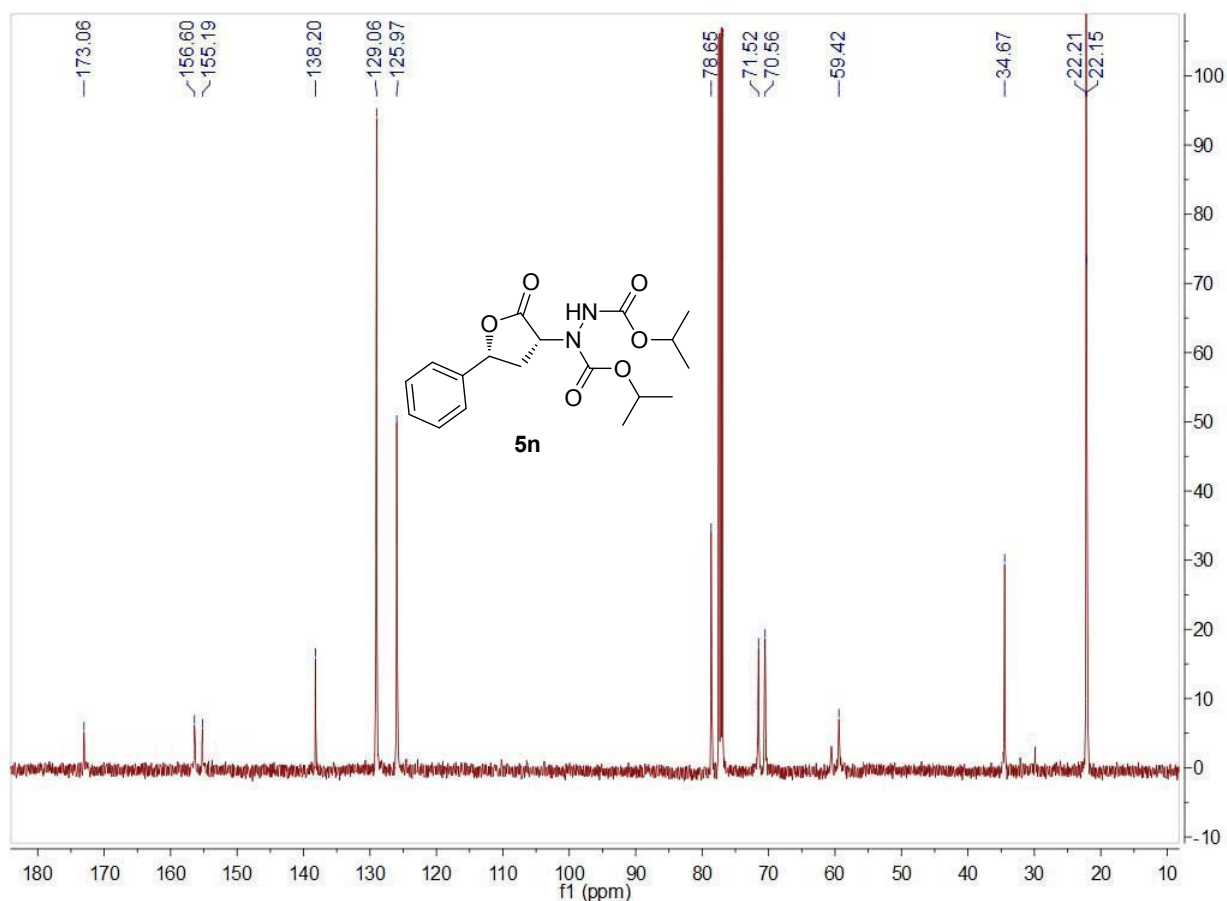
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

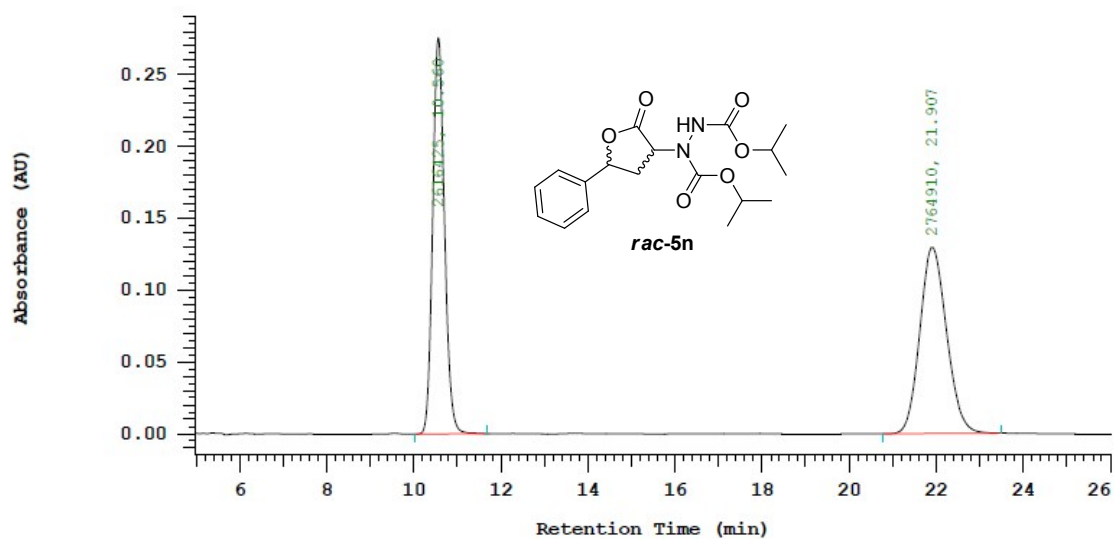
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	9.727	367930	2.901	BB
2	11.233	12313845	97.099	BB
		12681775	100.000	





Chrom Type: Fixed WL Chromatogram, 210 nm



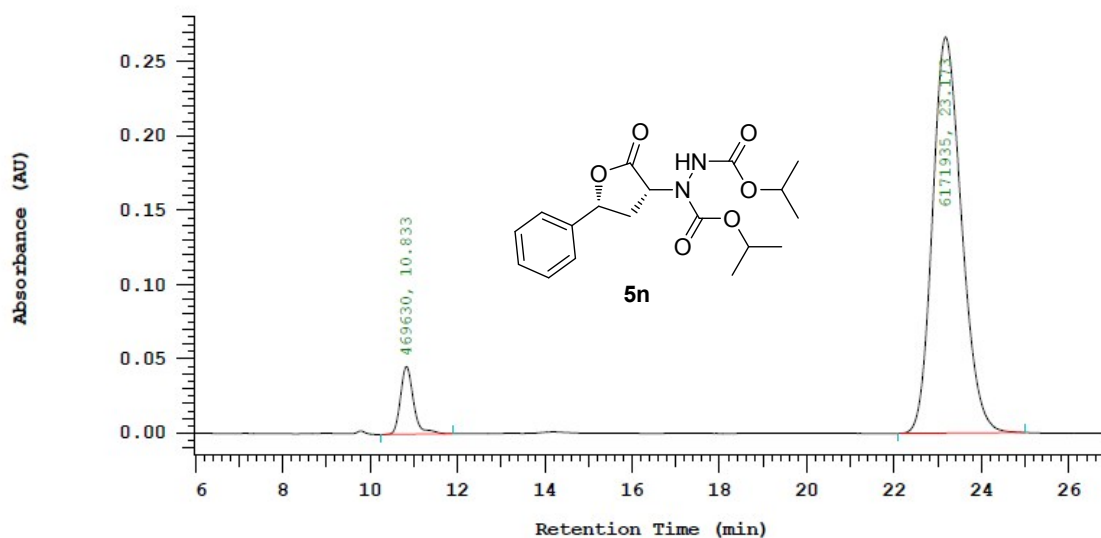
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	10.560	2616425	48.620	BB
2	21.907	2764910	51.380	BB
		5381335	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



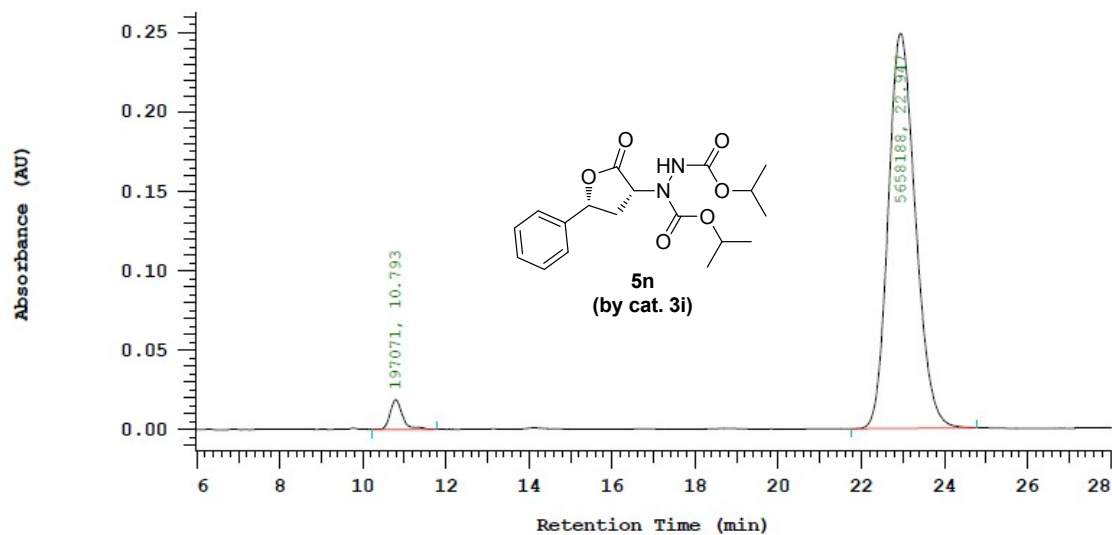
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	10.833	469630	7.071	BB
2	23.173	6171935	92.929	BB
		6641565	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

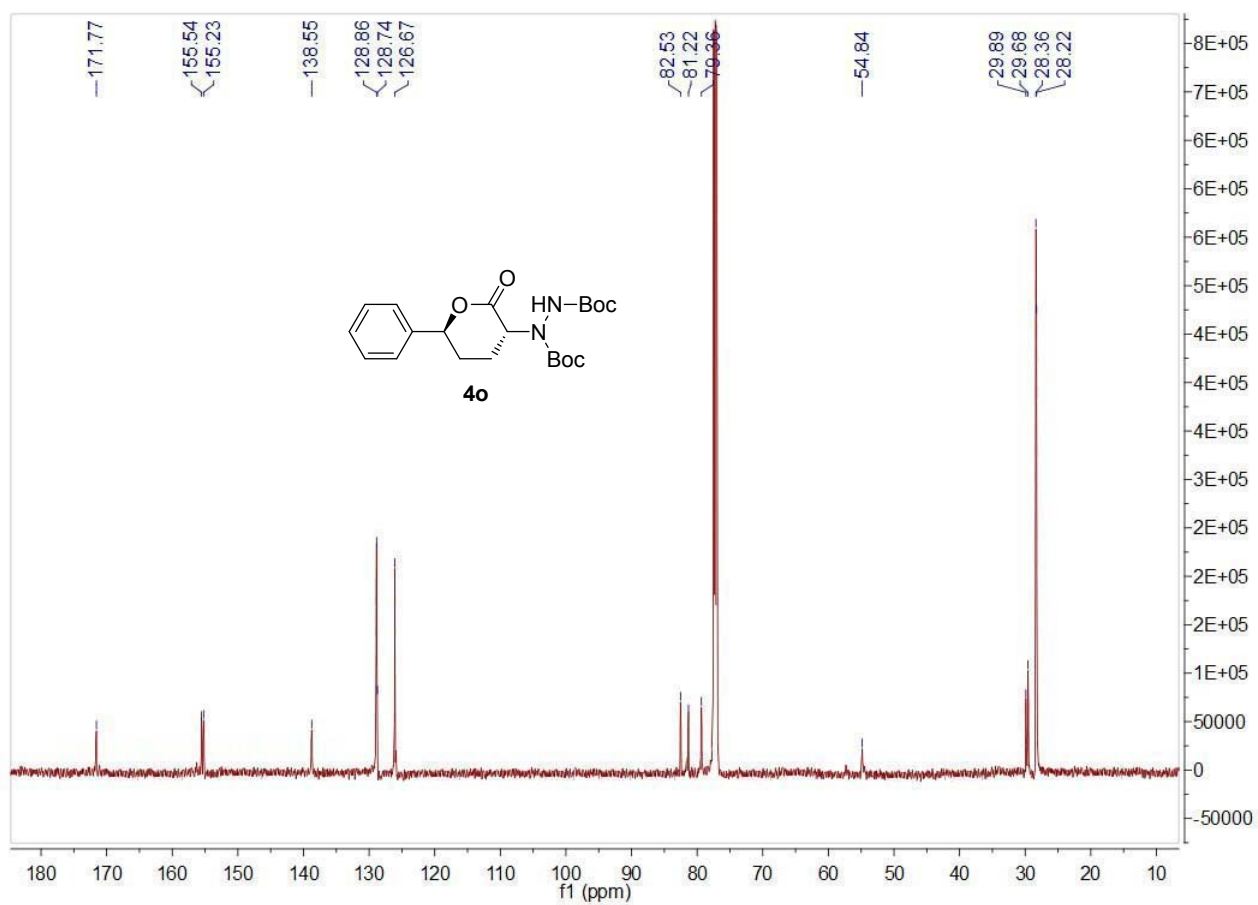
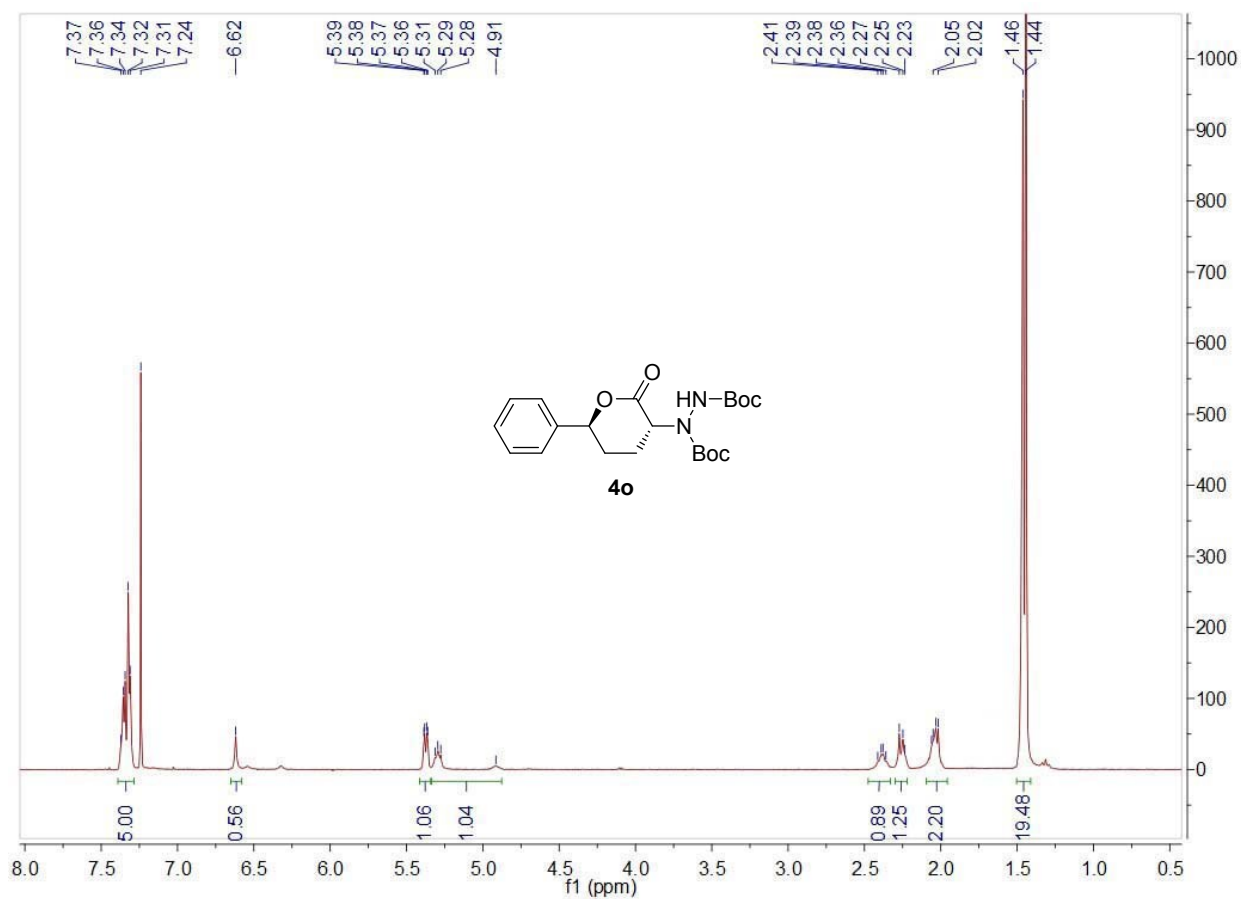


Chrom Type: Fixed WL Chromatogram, 210 nm

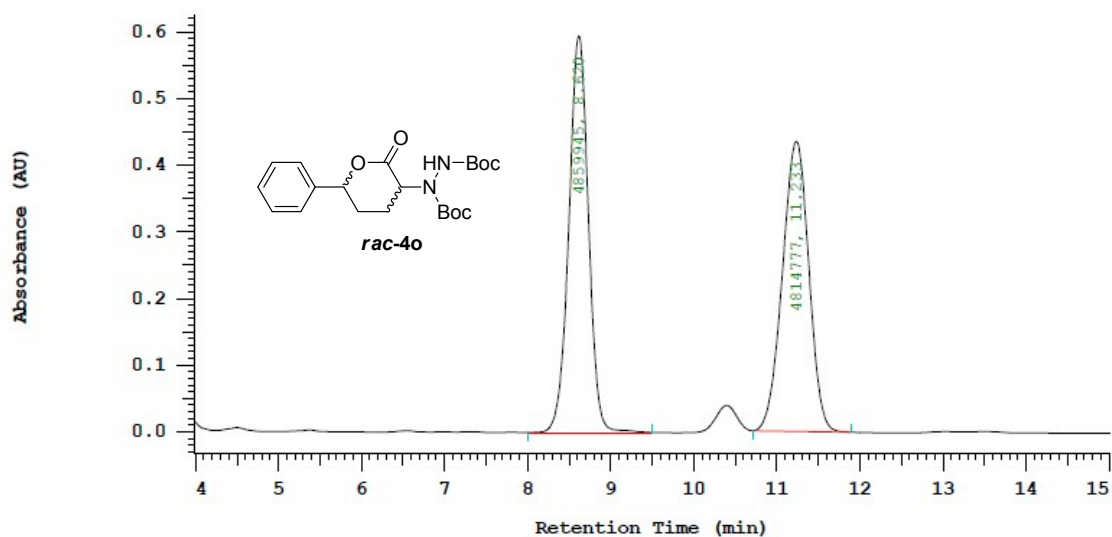
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	10.793	197071	3.366	BB
2	22.947	5658188	96.634	BB
		5855259	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



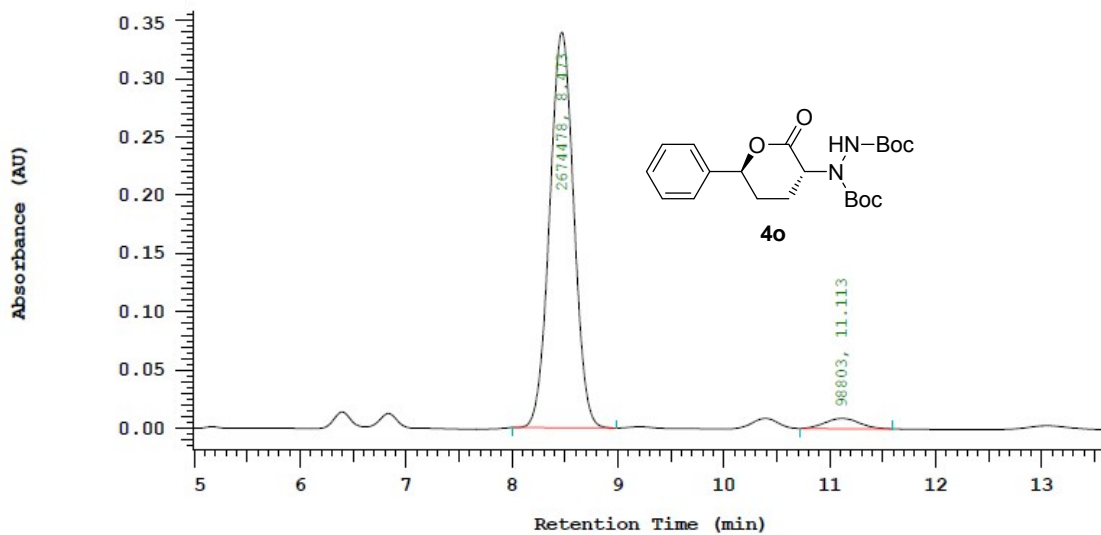
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.620	4859945	50.233	BB
2	11.233	4814777	49.767	BB
		9674722	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

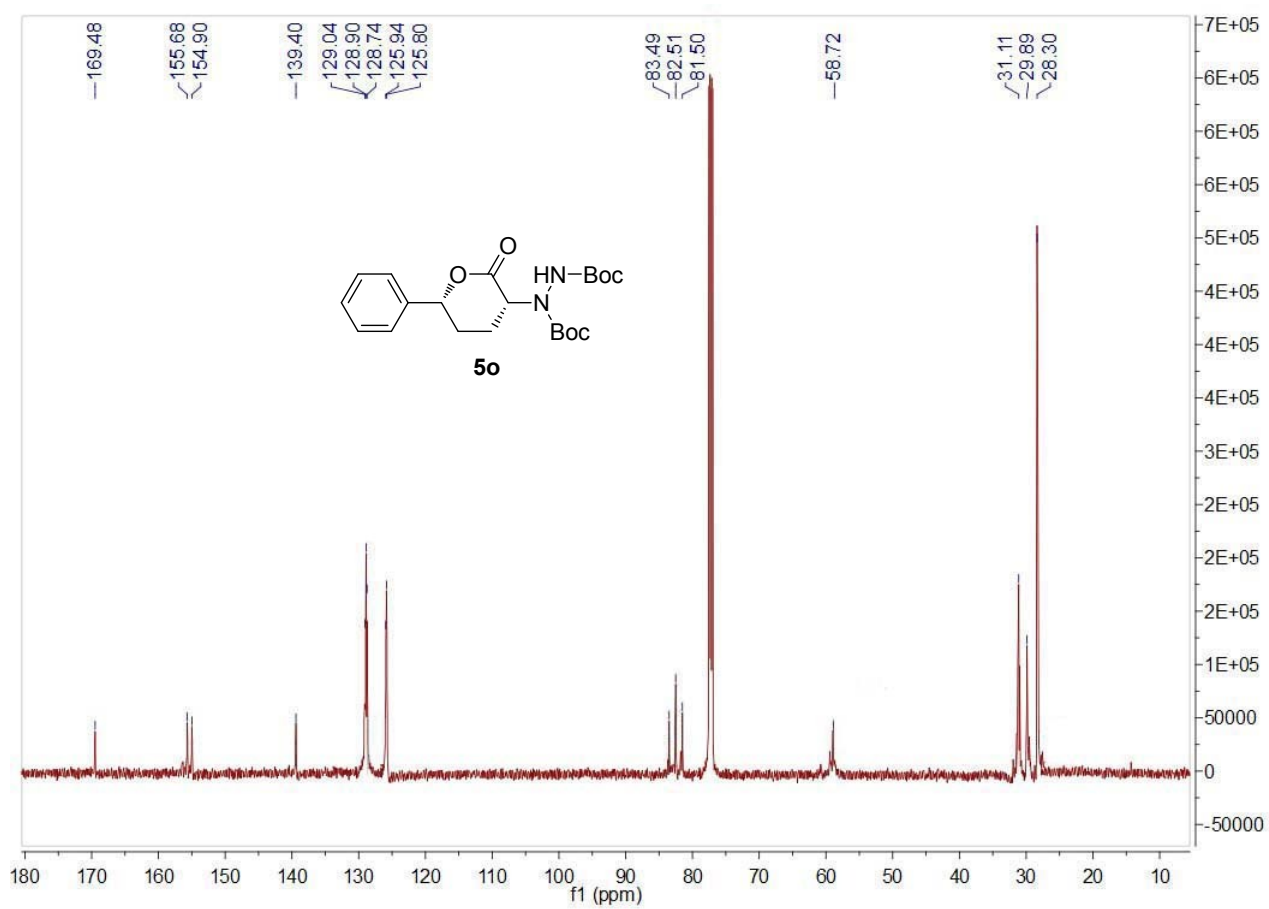
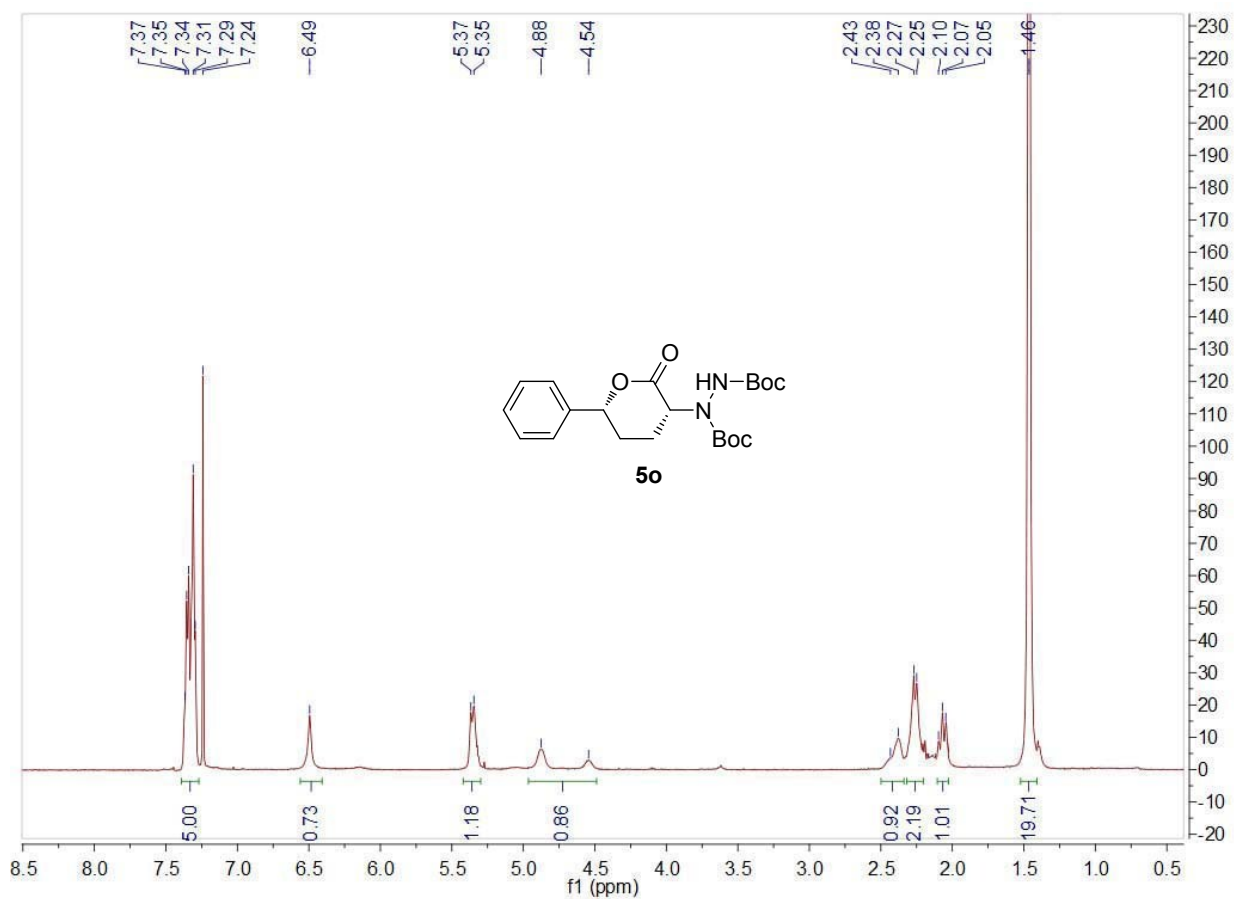


Chrom Type: Fixed WL Chromatogram, 210 nm

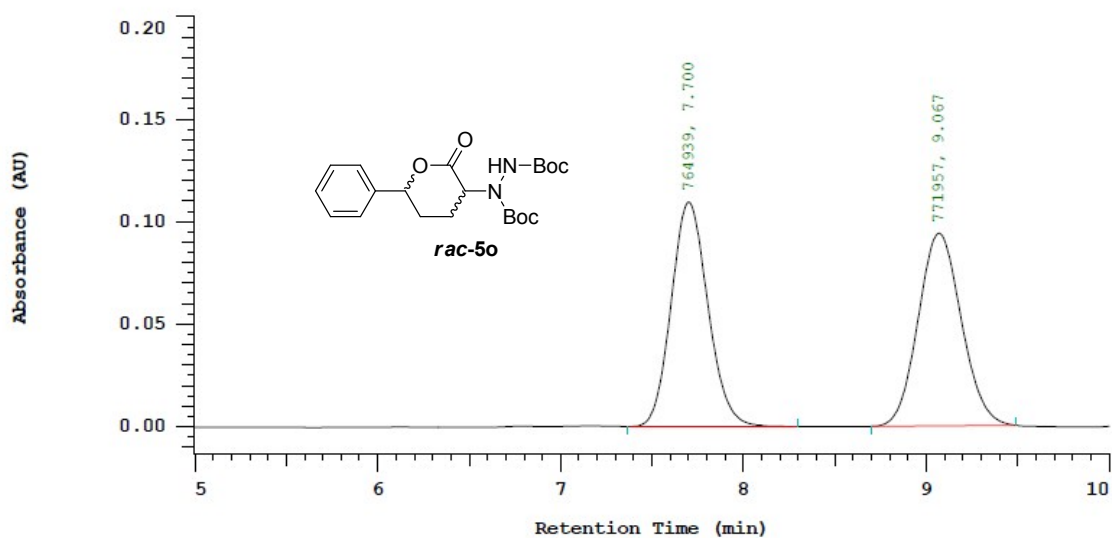
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.473	2674478	96.437	BB
2	11.113	98803	3.563	BB
		2773281	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



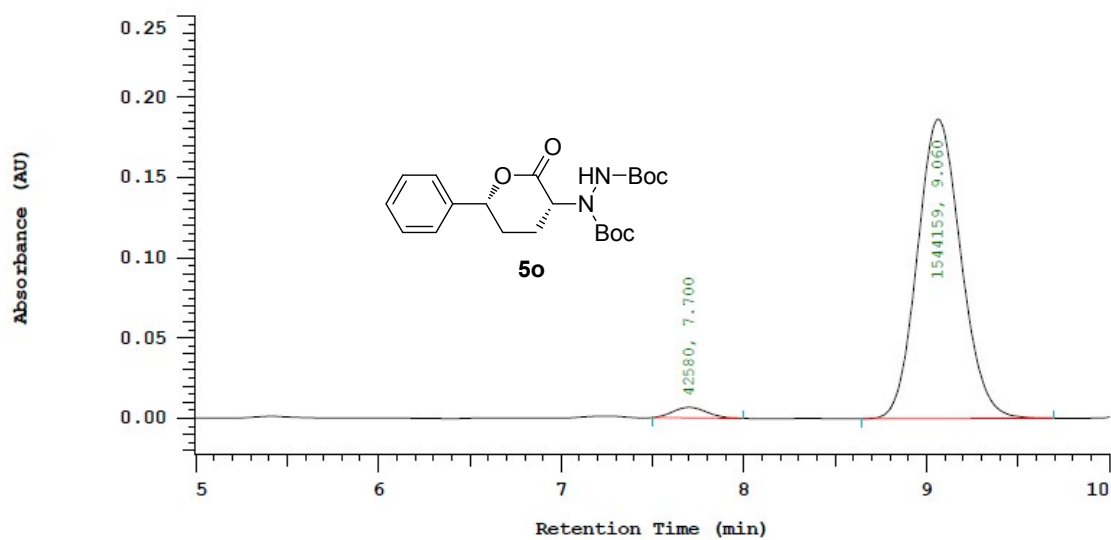
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.700	764939	49.772	BB
2	9.067	771957	50.228	BB
		1536896	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

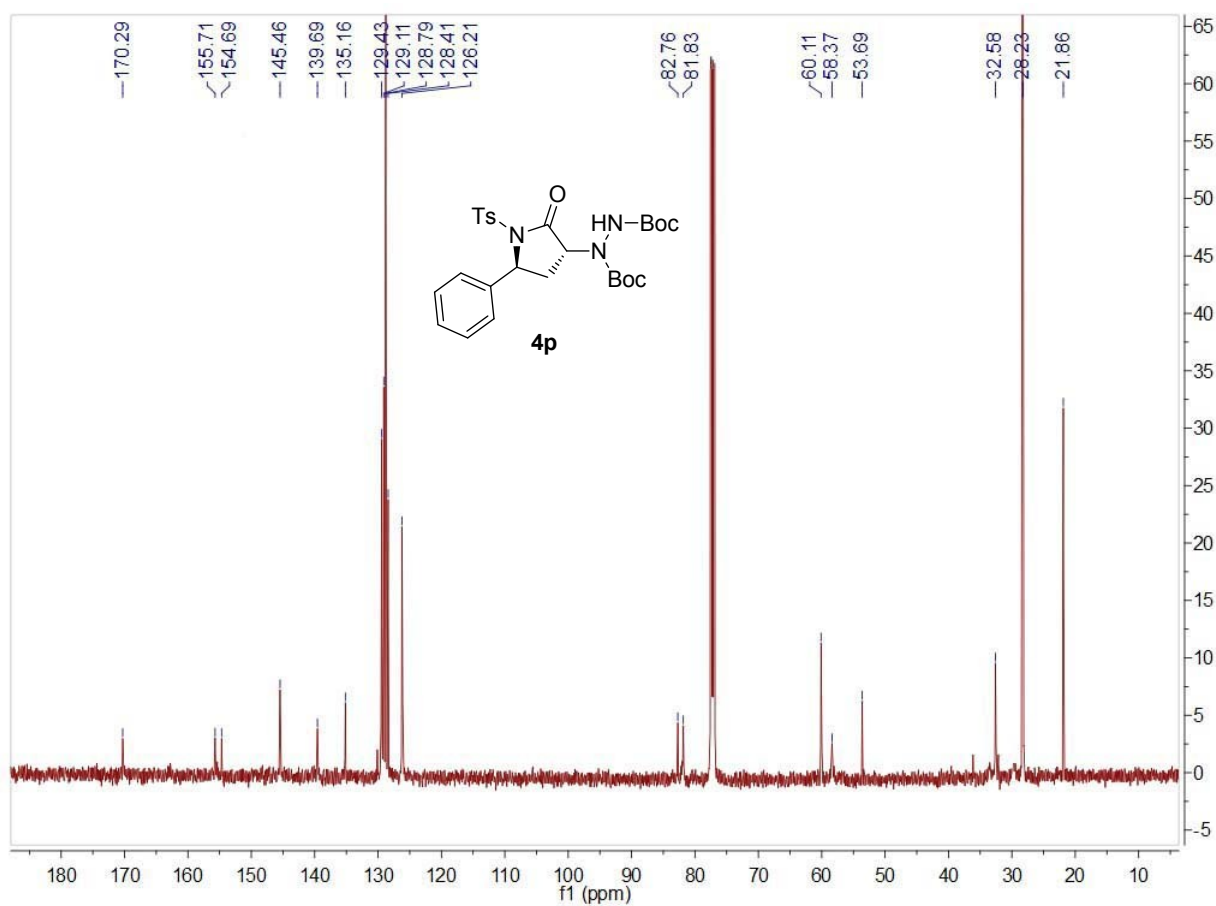
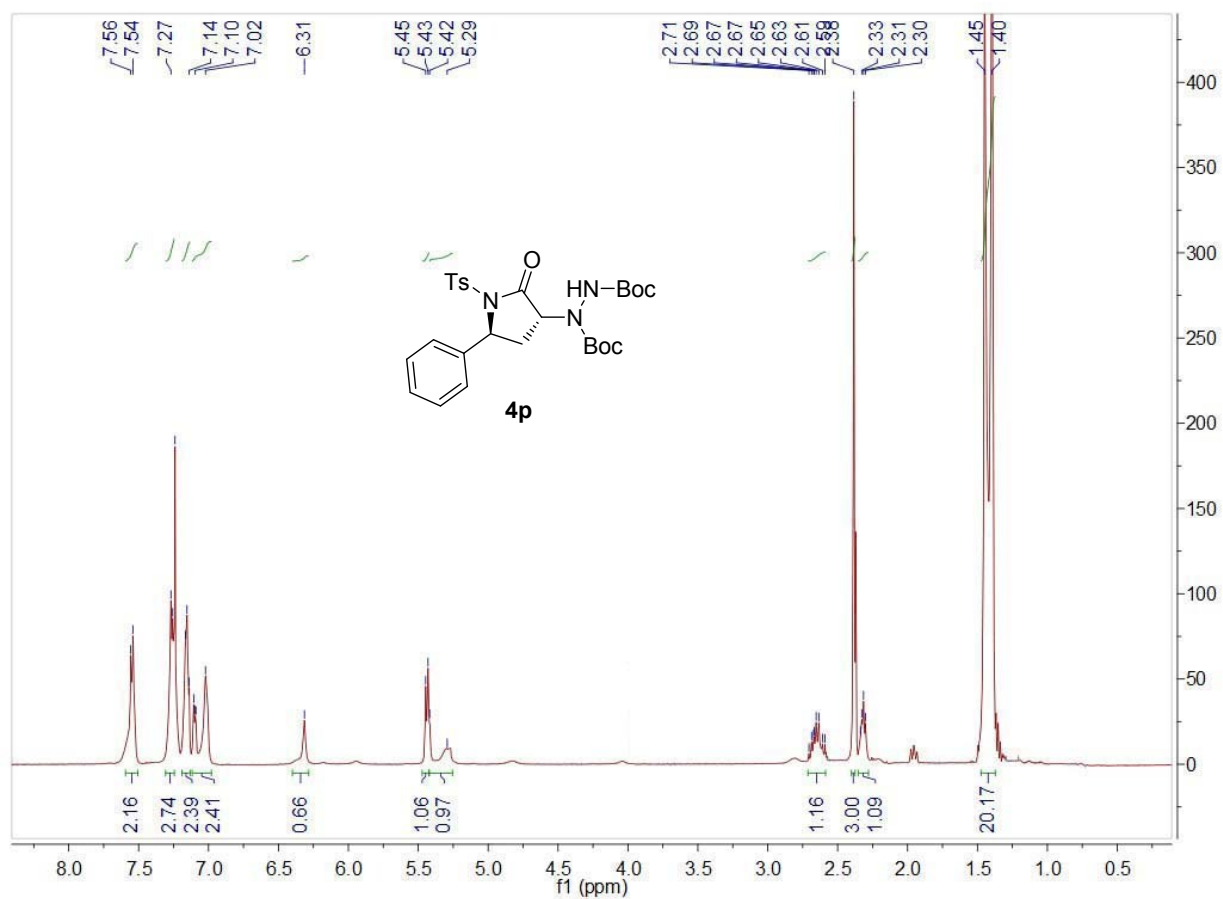


Chrom Type: Fixed WL Chromatogram, 210 nm

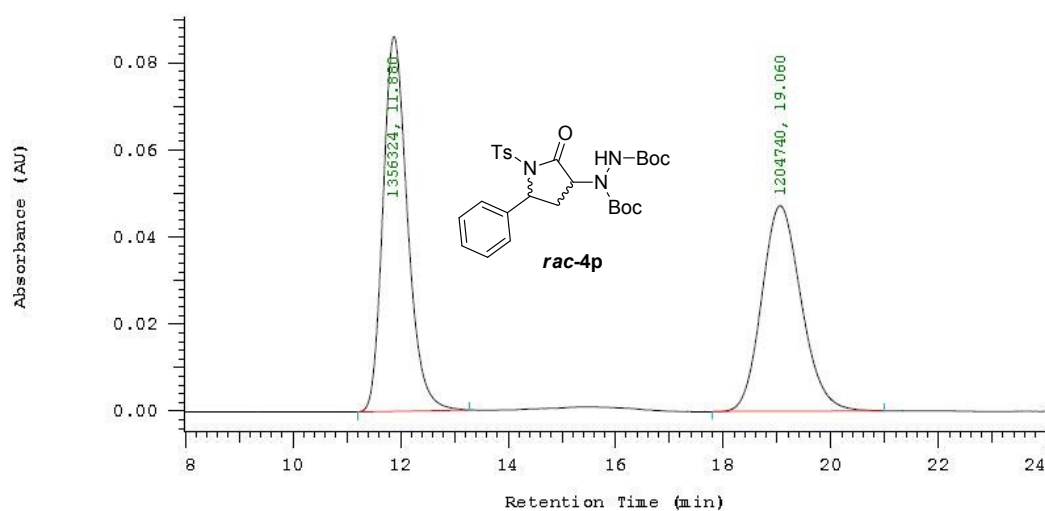
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.700	42580	2.683	BB
2	9.060	1544159	97.317	BB
		1586739	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm

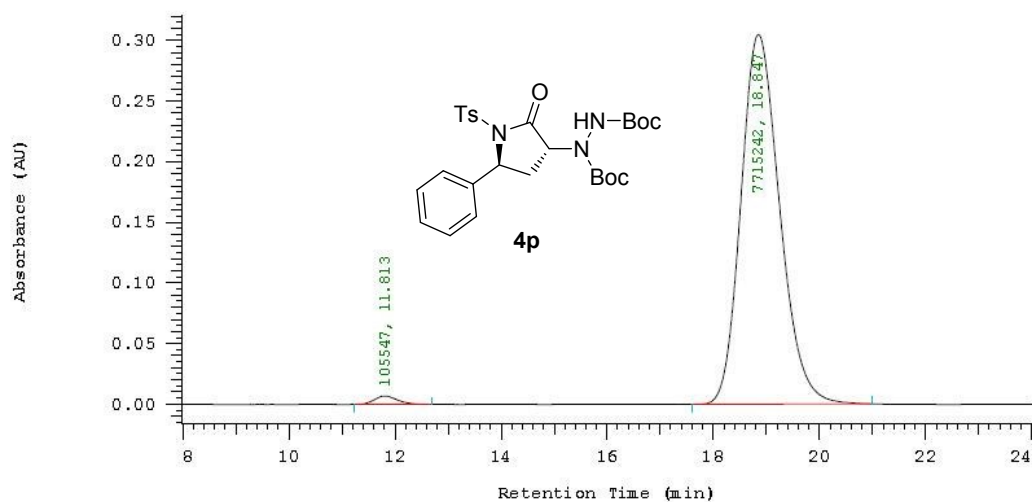


Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.880	1356324	52.959	BB
2	19.060	1204740	47.041	BB
		2561064	100.000	

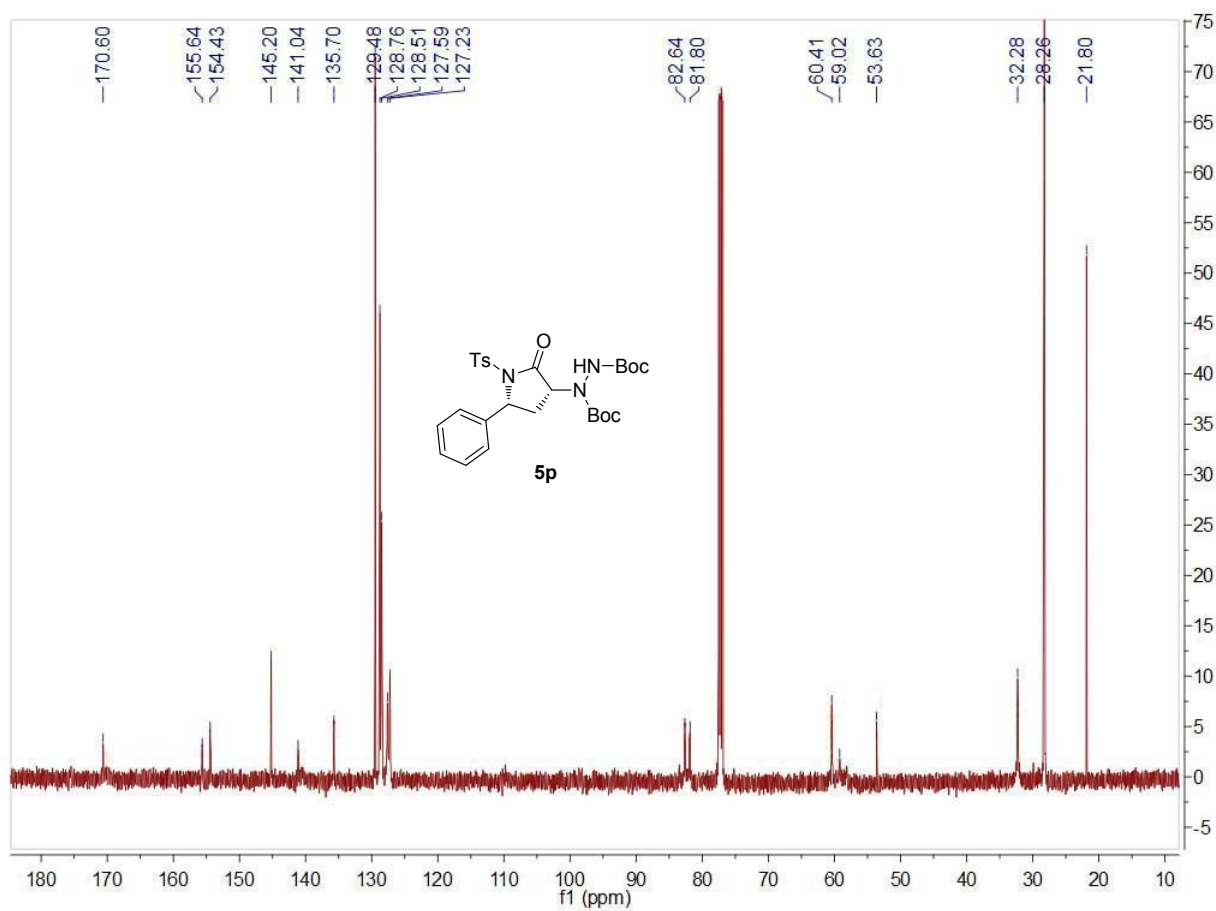
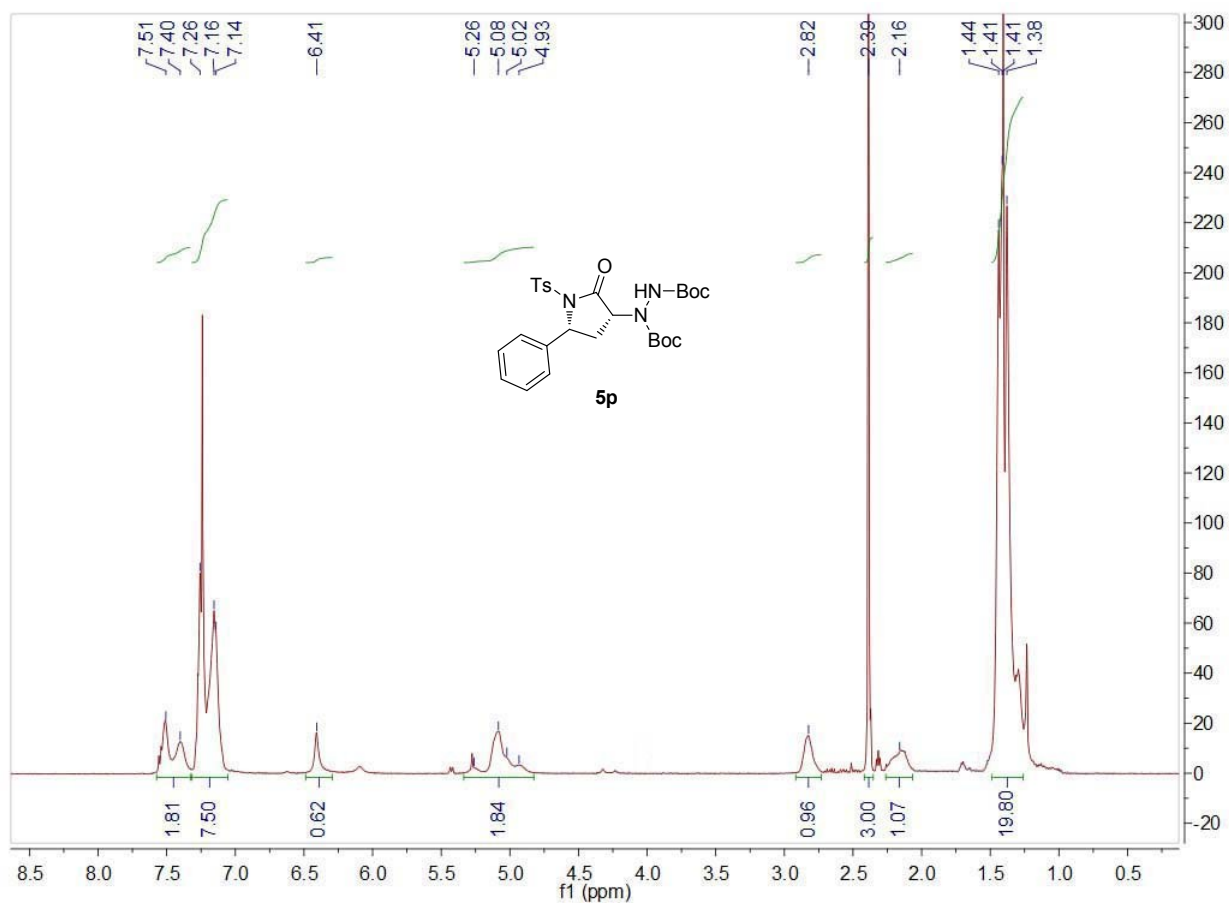
Chrom Type: Fixed WL Chromatogram, 210 nm



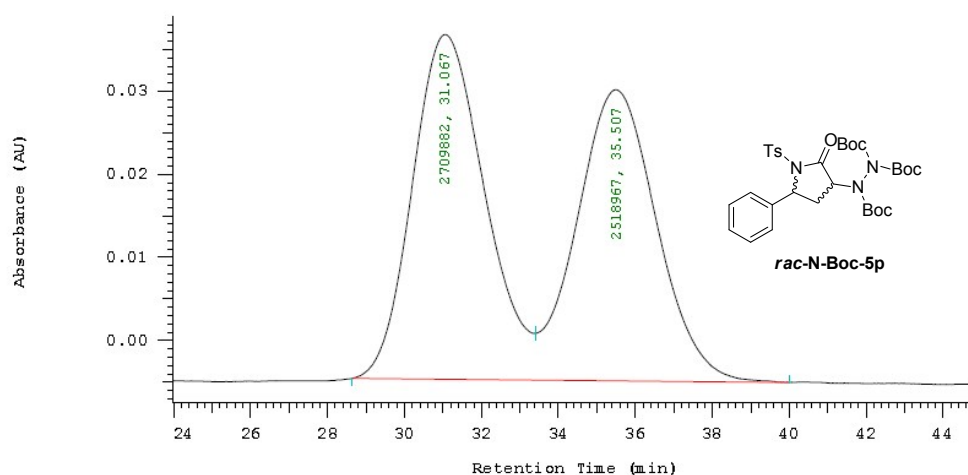
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	11.813	105547	1.350	BB
2	18.847	7715242	98.650	BB
		7820789	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm

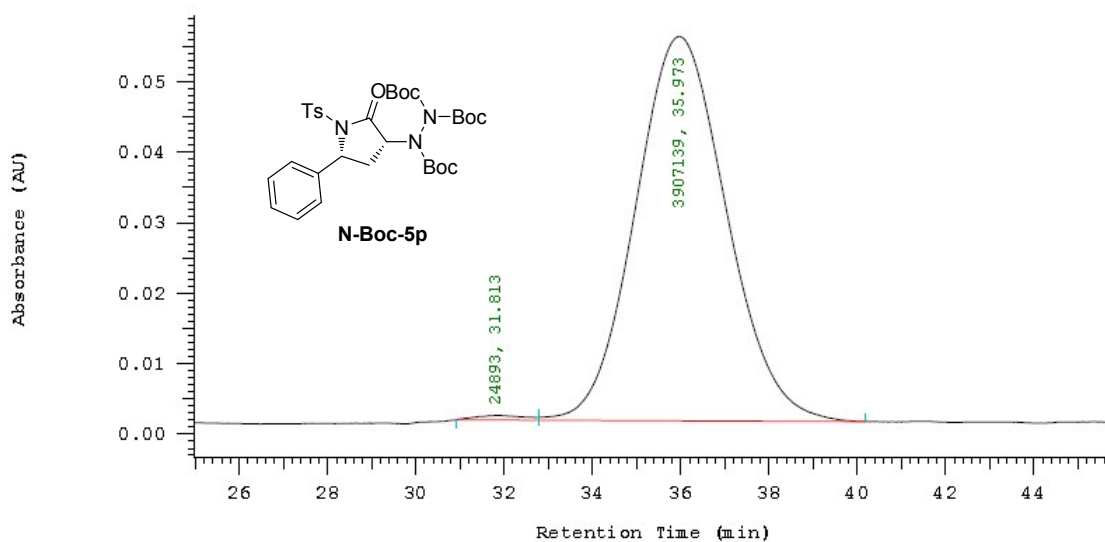


Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	31.067	2709882	51.826	BV
2	35.507	2518967	48.174	VB
		5228849	100.000	

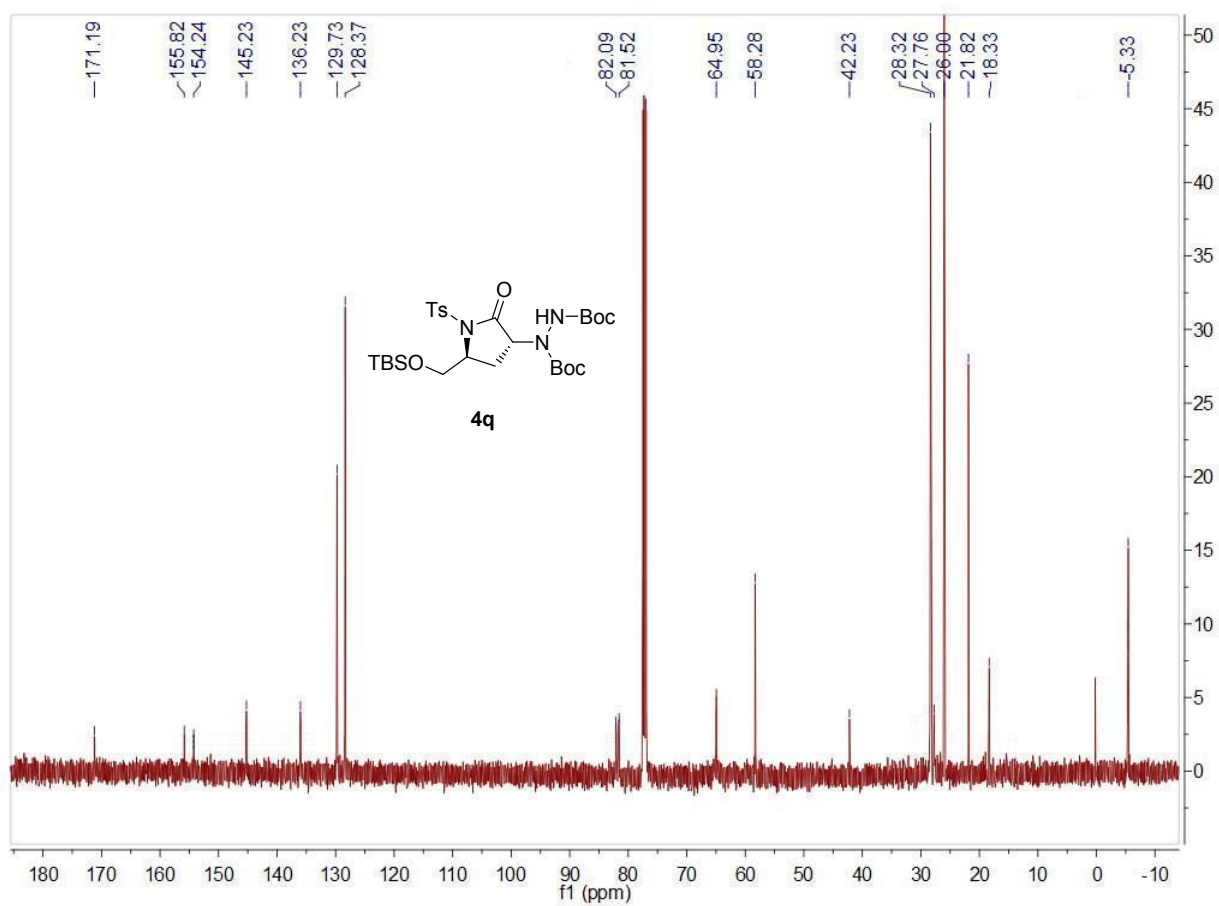
Chrom Type: Fixed WL Chromatogram, 210 nm



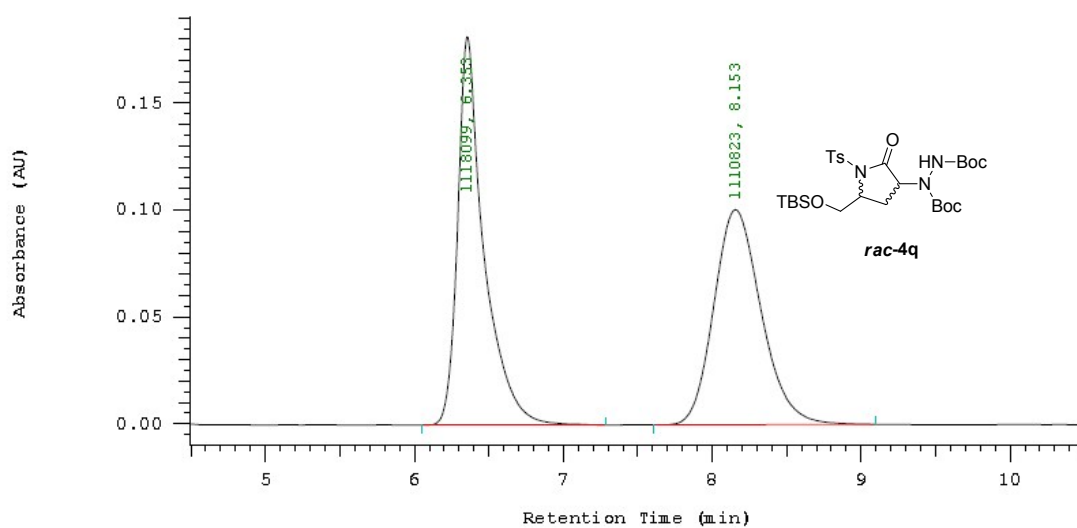
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	31.813	24893	0.633	BV
2	35.973	3907139	99.367	VB
		3932032	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



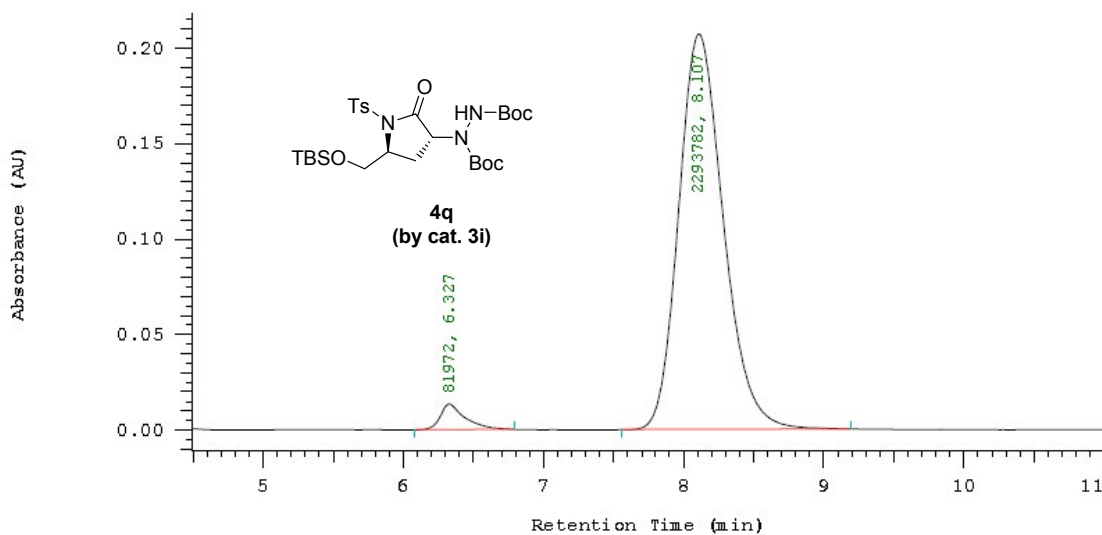
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.353	1118099	50.163	BB
2	8.153	1110823	49.837	BB
		2228922	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

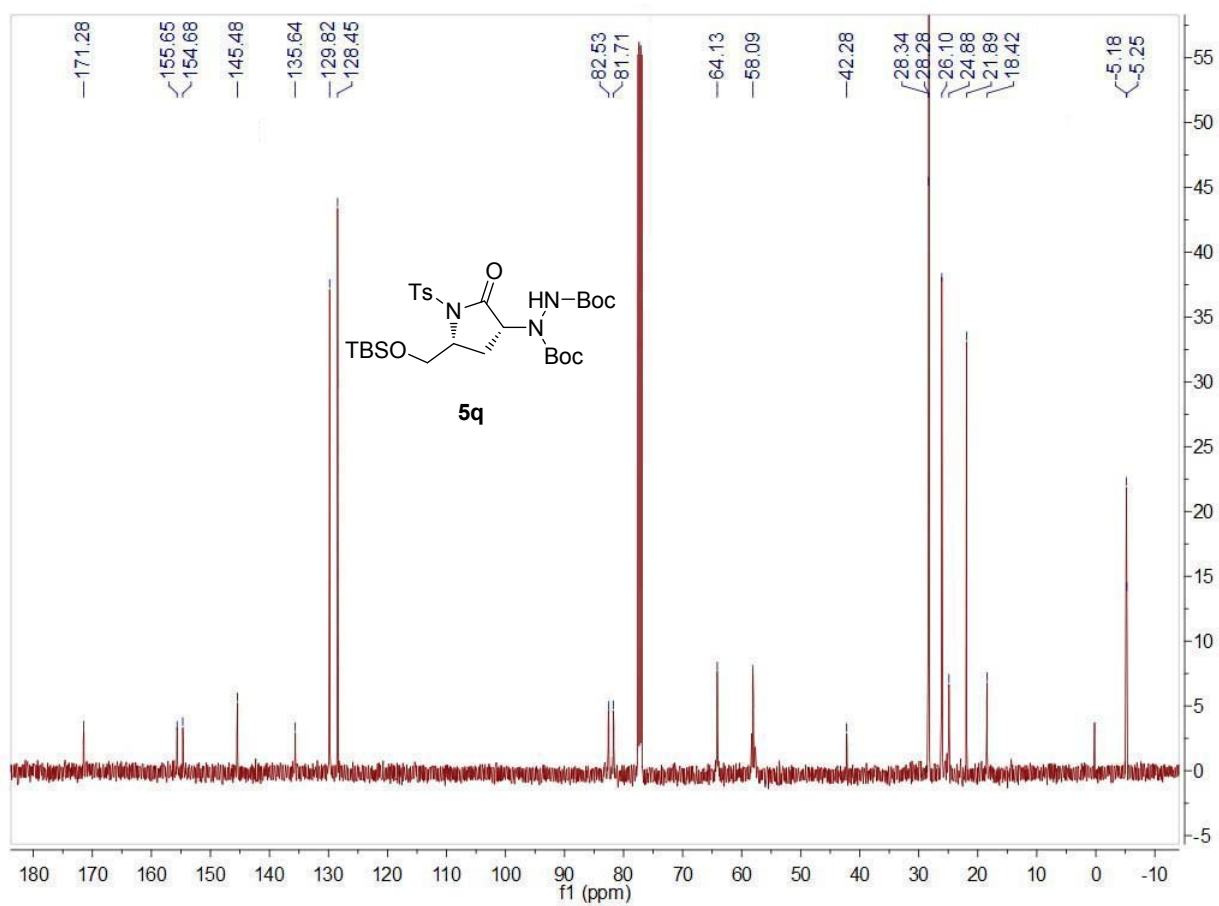
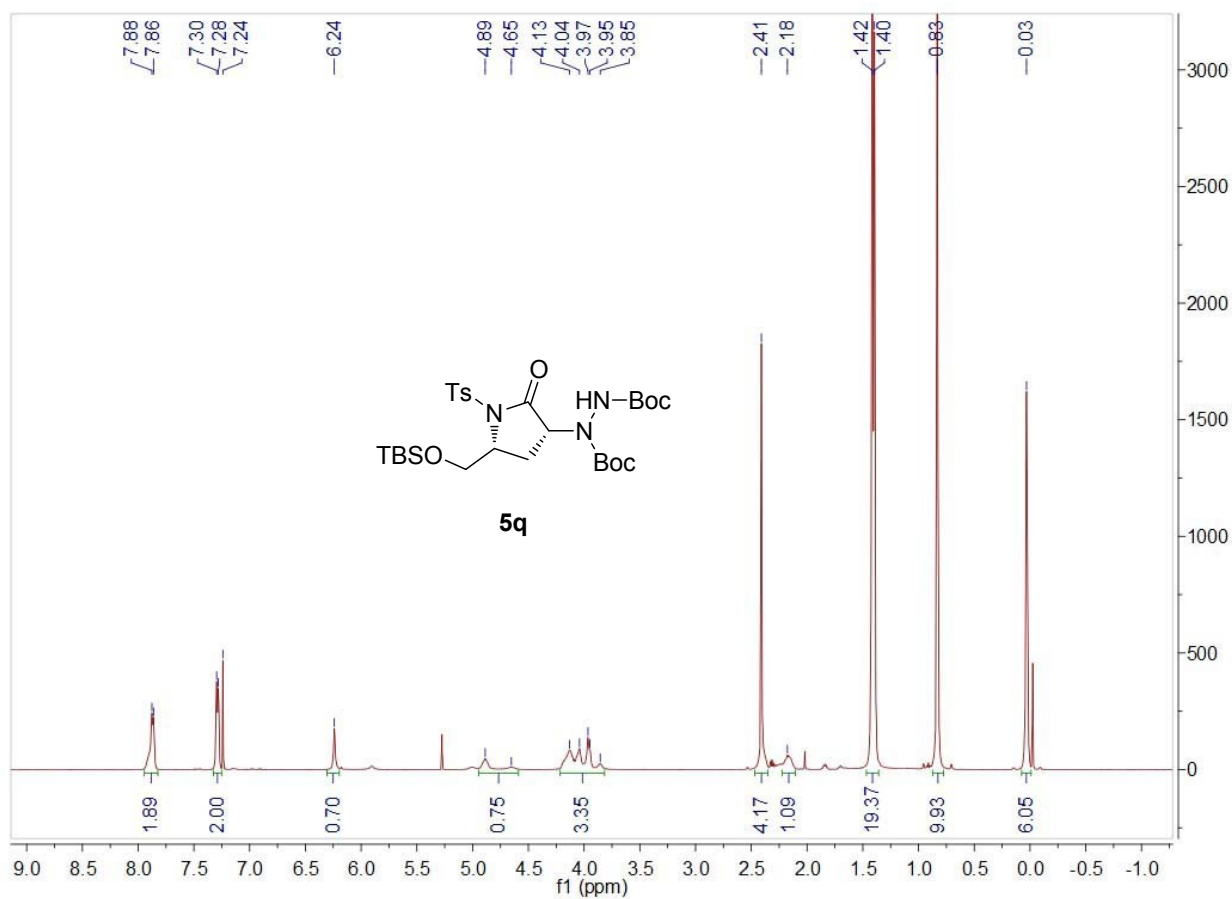


Chrom Type: Fixed WL Chromatogram, 210 nm

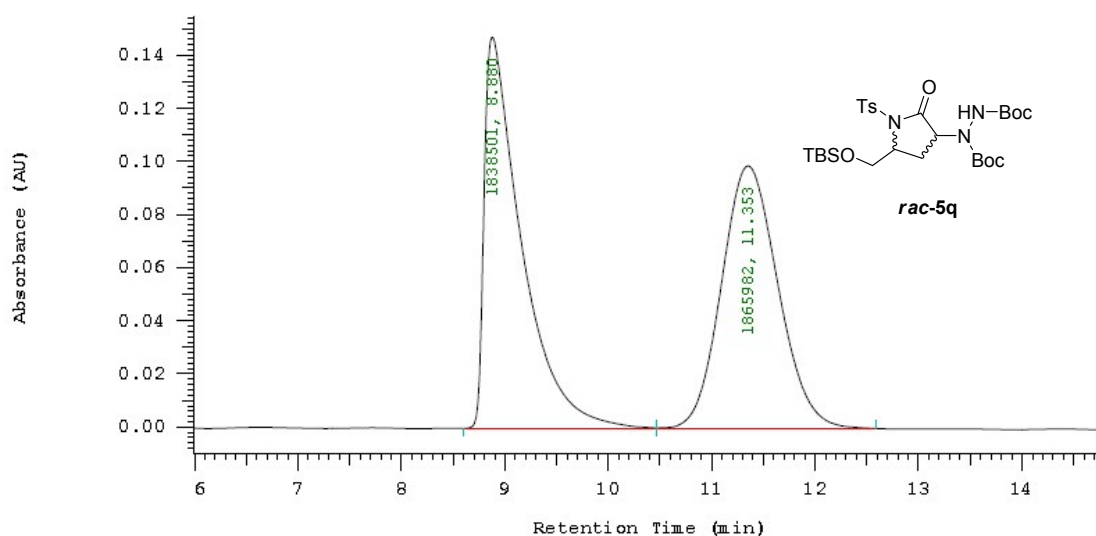
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.327	81972	3.450	BB
2	8.107	2293782	96.550	BB
		2375754	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm

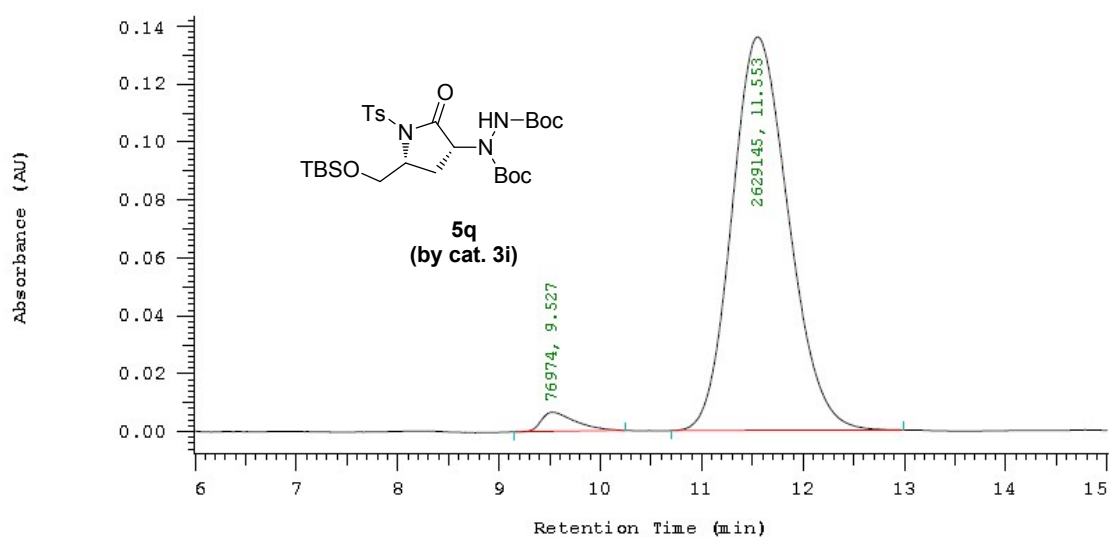


Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	8.880	1838501	49.629	BV
2	11.353	1865982	50.371	VB
		3704483	100.000	

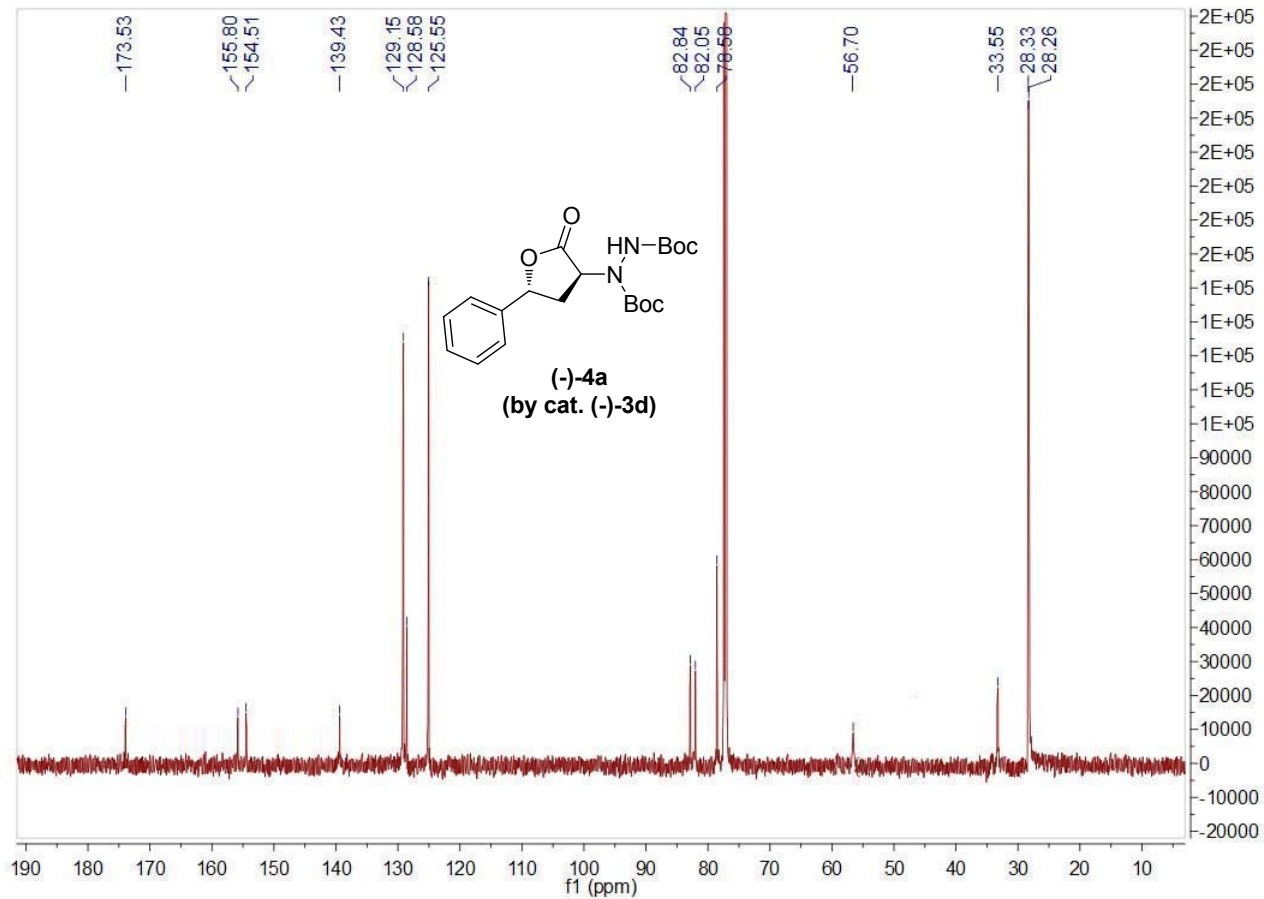
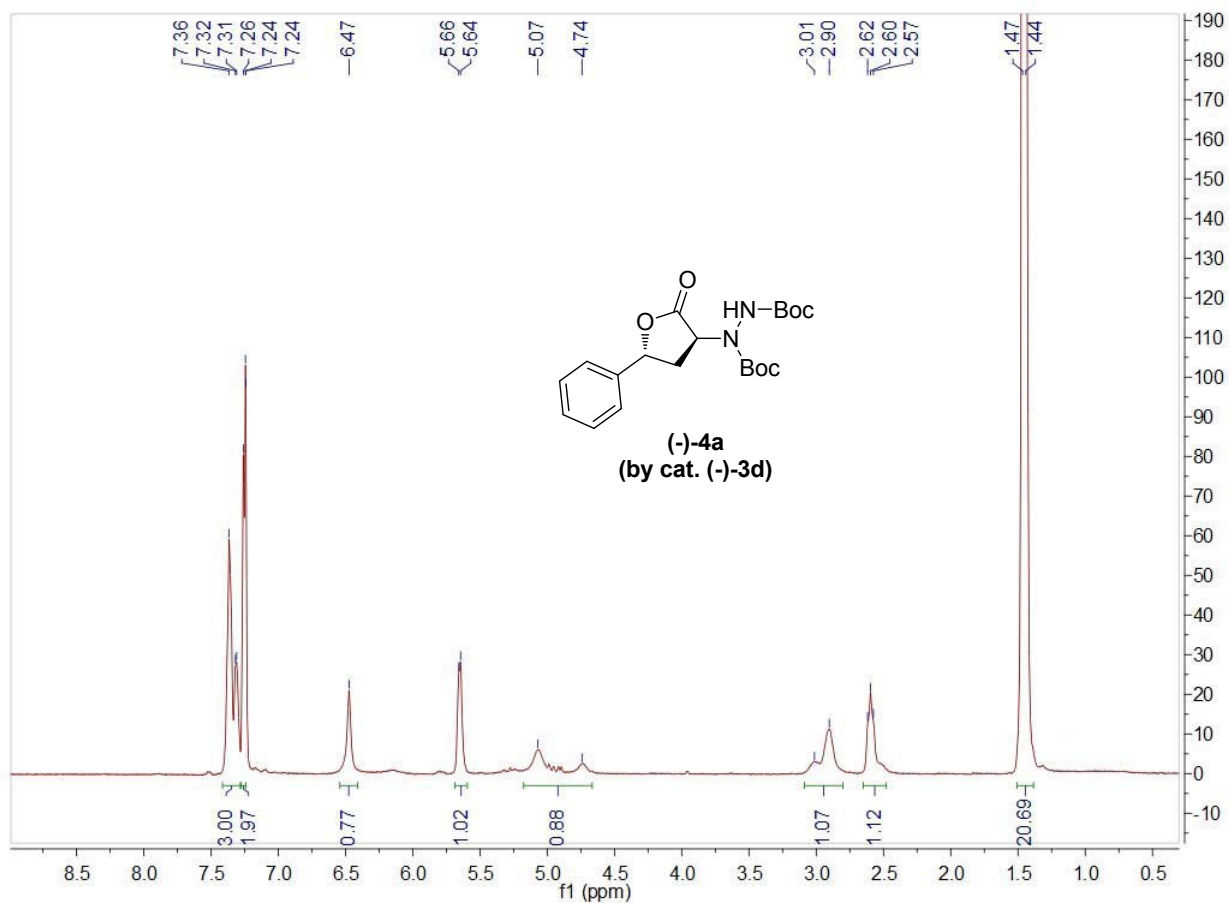
Chrom Type: Fixed WL Chromatogram, 210 nm

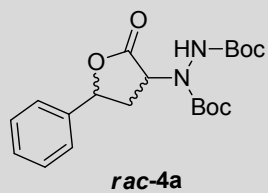


Chrom Type: Fixed WL Chromatogram, 210 nm

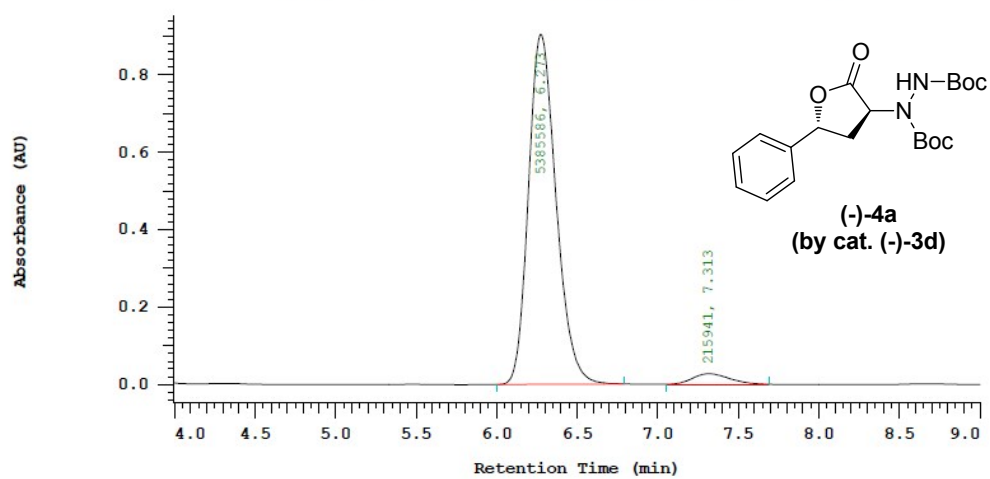
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	9.527	76974	2.844	BB
2	11.553	2629145	97.156	BB
		2706119	100.000	





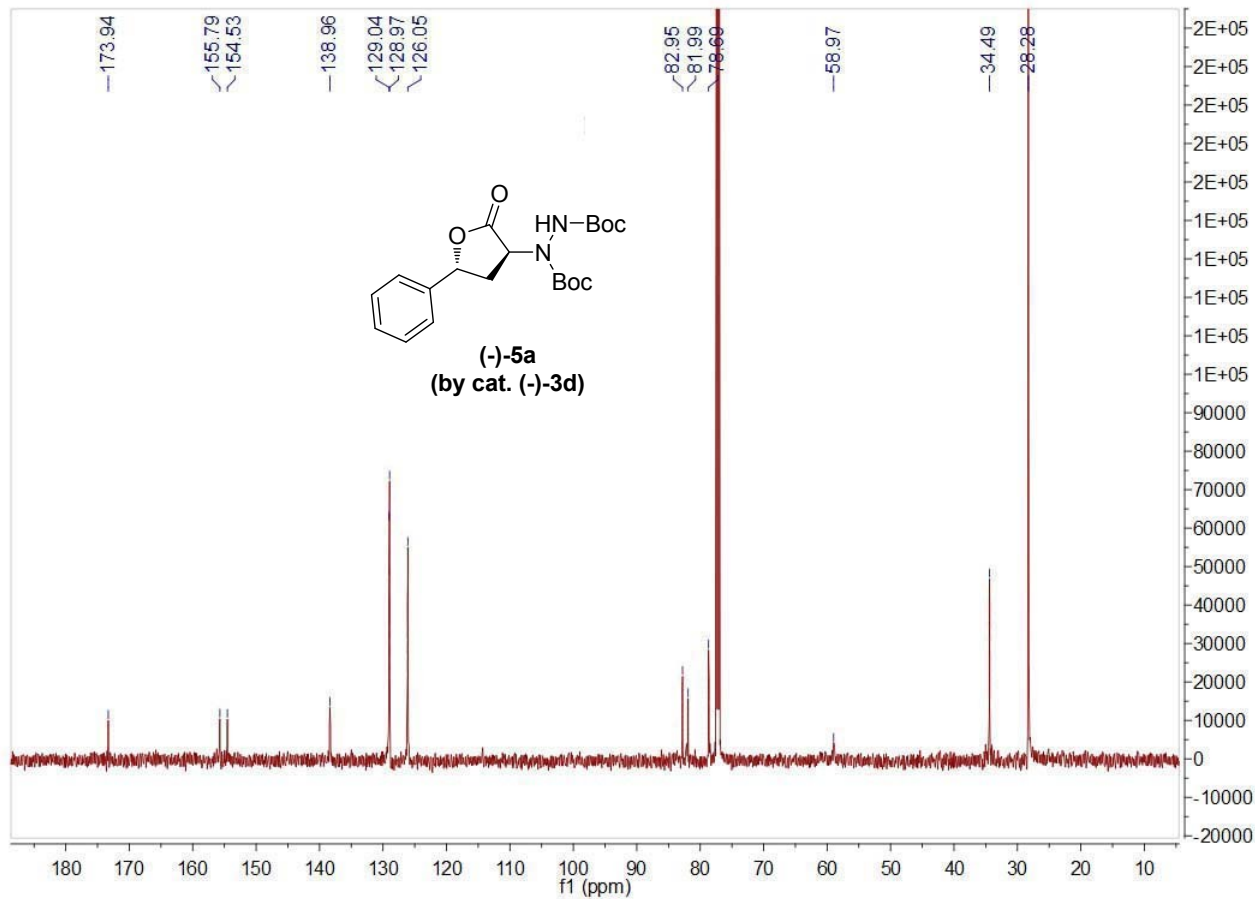
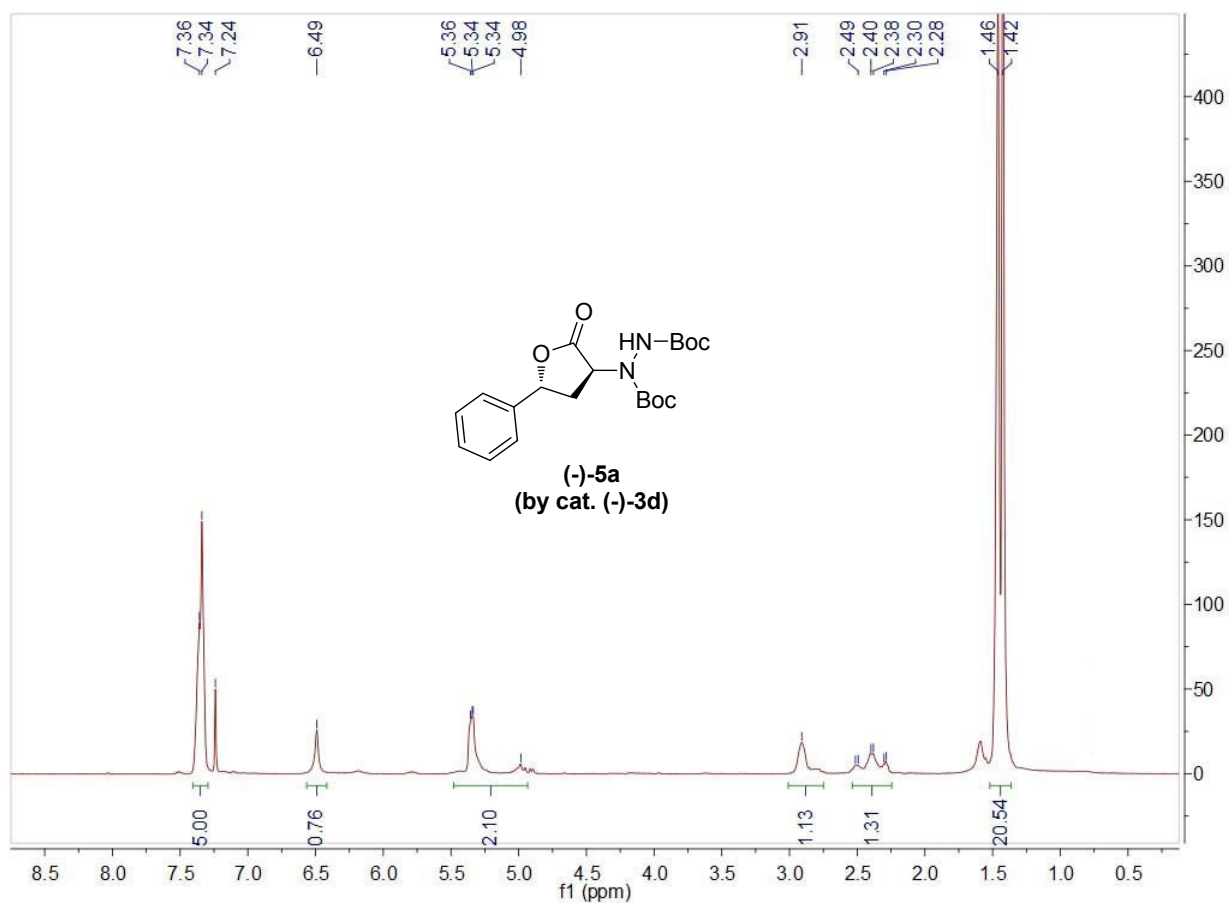
Chrom Type: Fixed WL Chromatogram, 210 nm

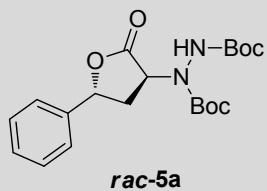


Chrom Type: Fixed WL Chromatogram, 210 nm

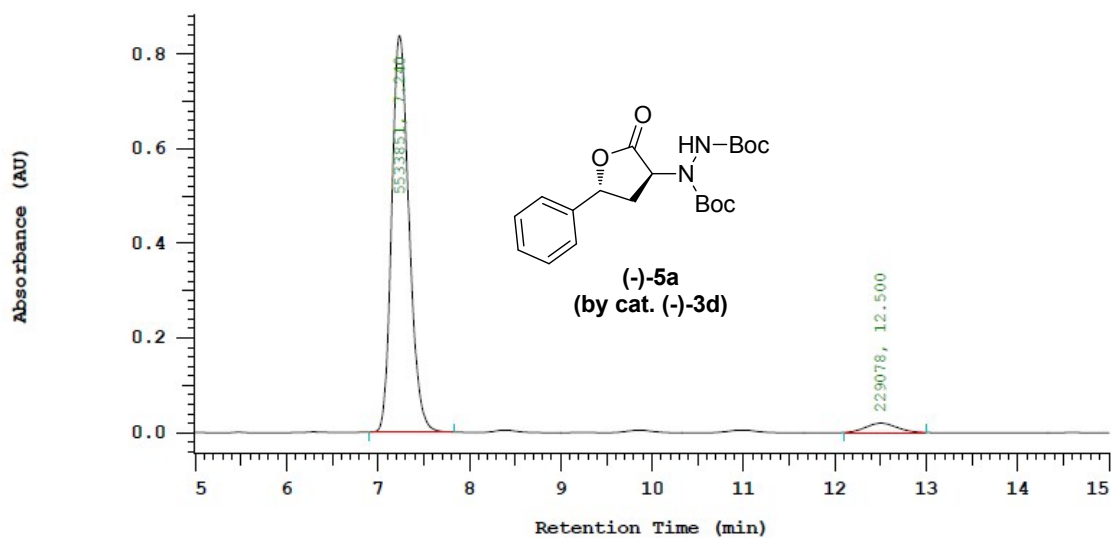
Peak Quantitation: AREA
Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.273	5385586	96.145	BB
2	7.313	215941	3.855	BB
		5601527	100.000	





Chrom Type: Fixed WL Chromatogram, 210 nm

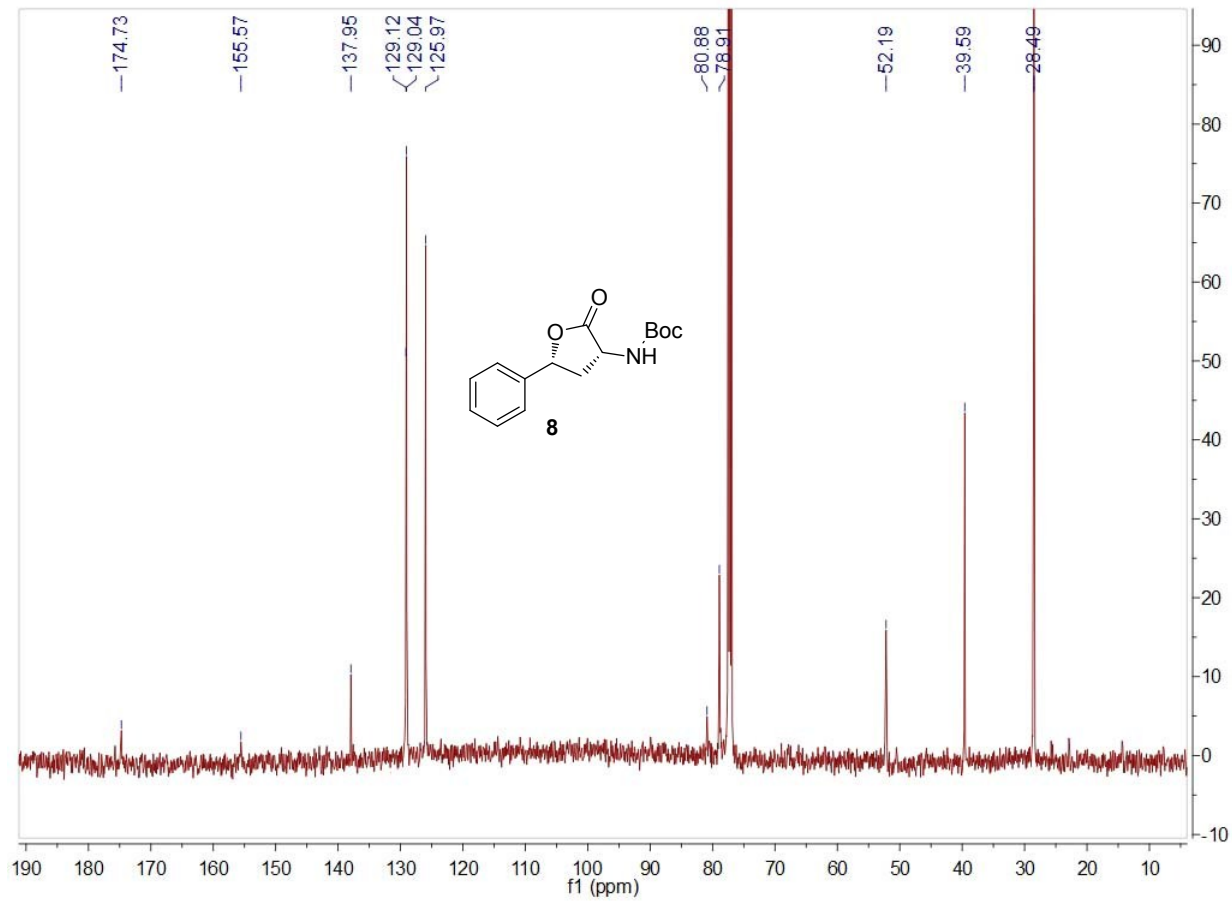
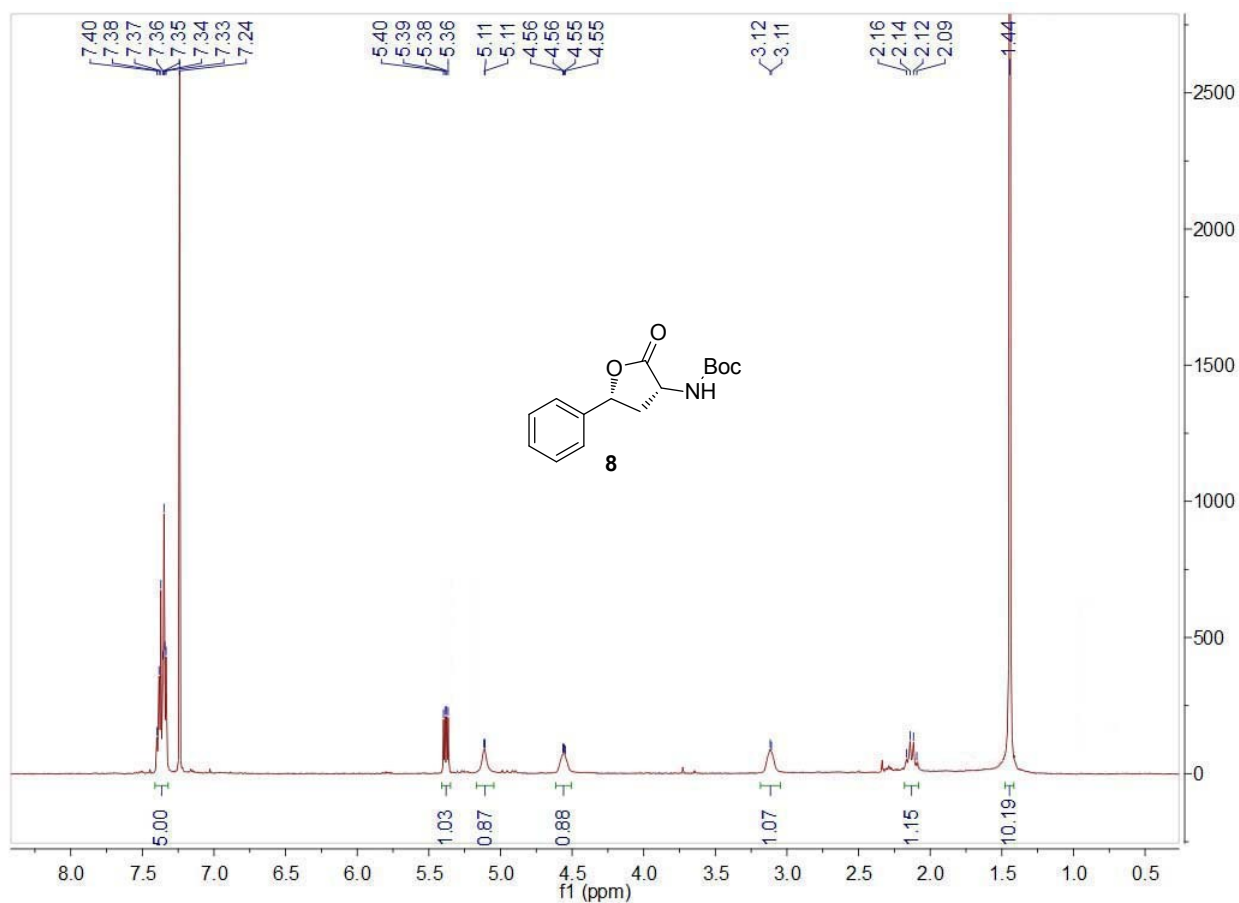


Chrom Type: Fixed WL Chromatogram, 210 nm

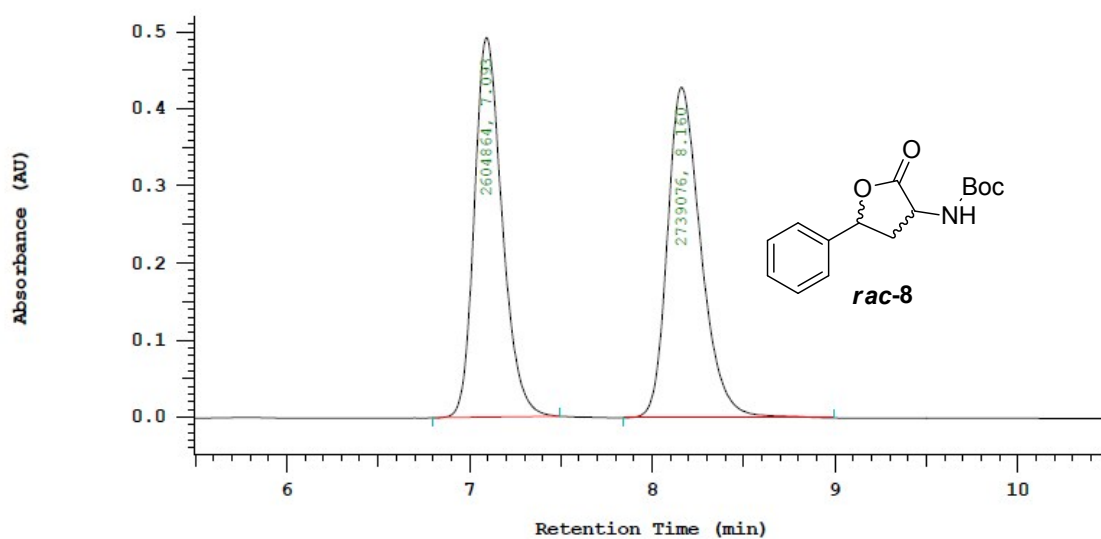
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.240	5533851	96.025	BB
2	12.500	229078	3.975	BB
		5762929	100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



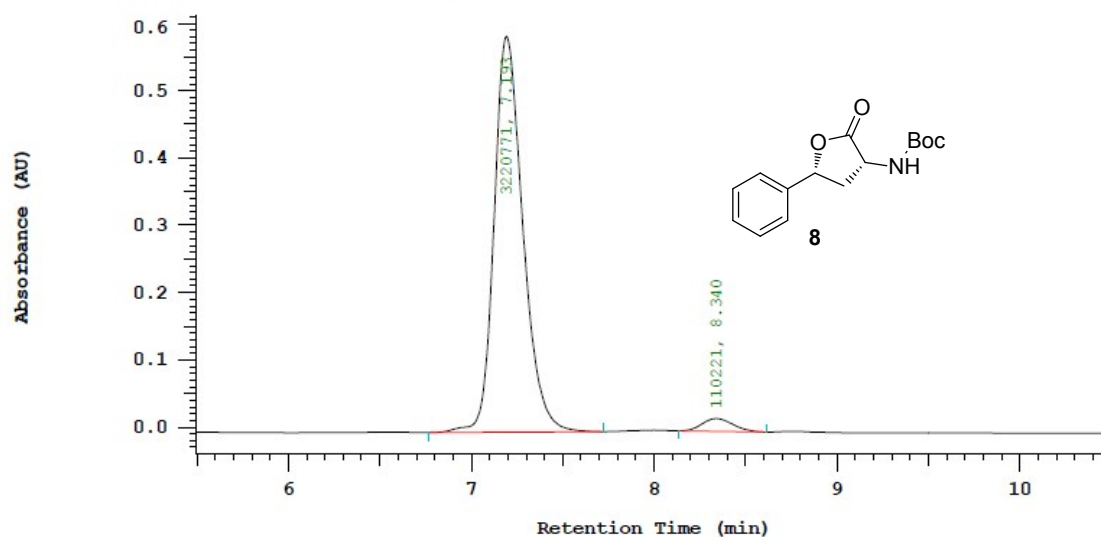
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	7.093	2604864	48.744	BB
2	8.160	2739076	51.256	BB
5343940			100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

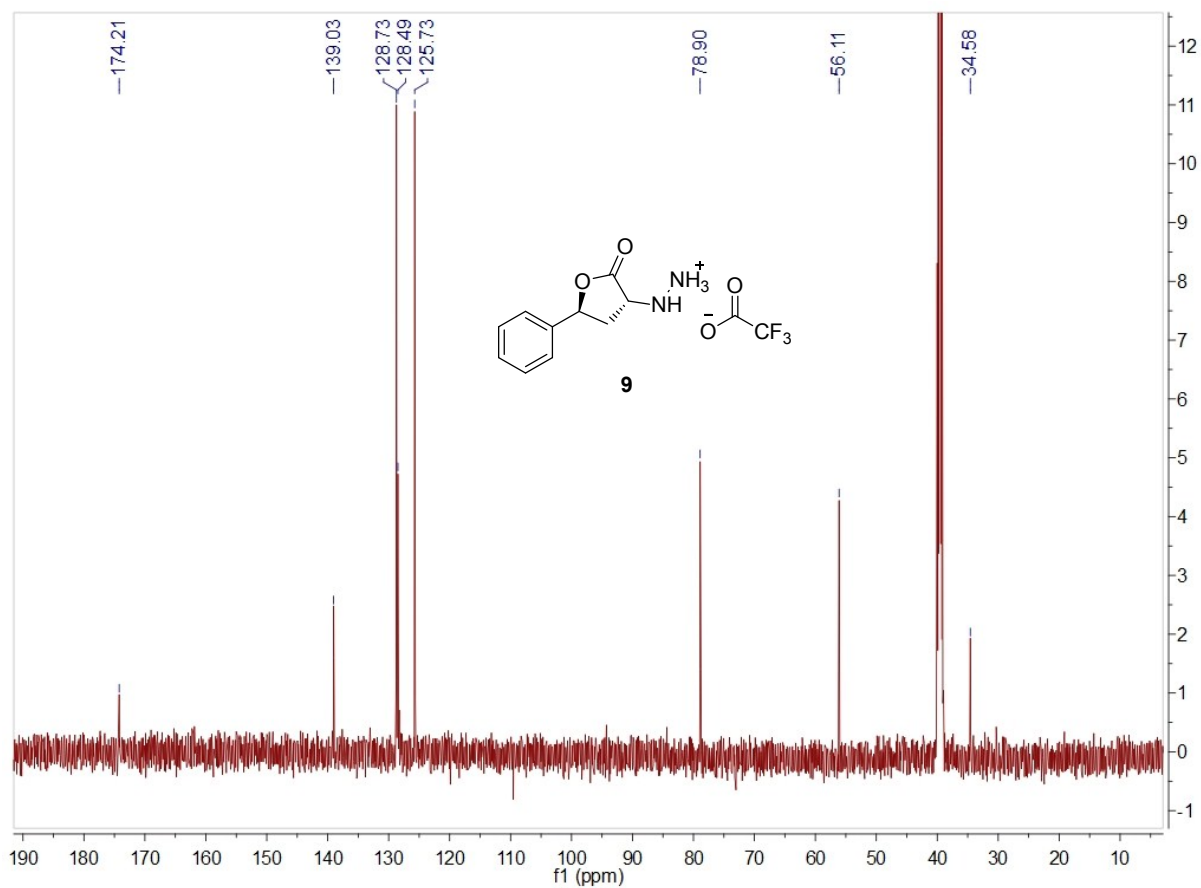
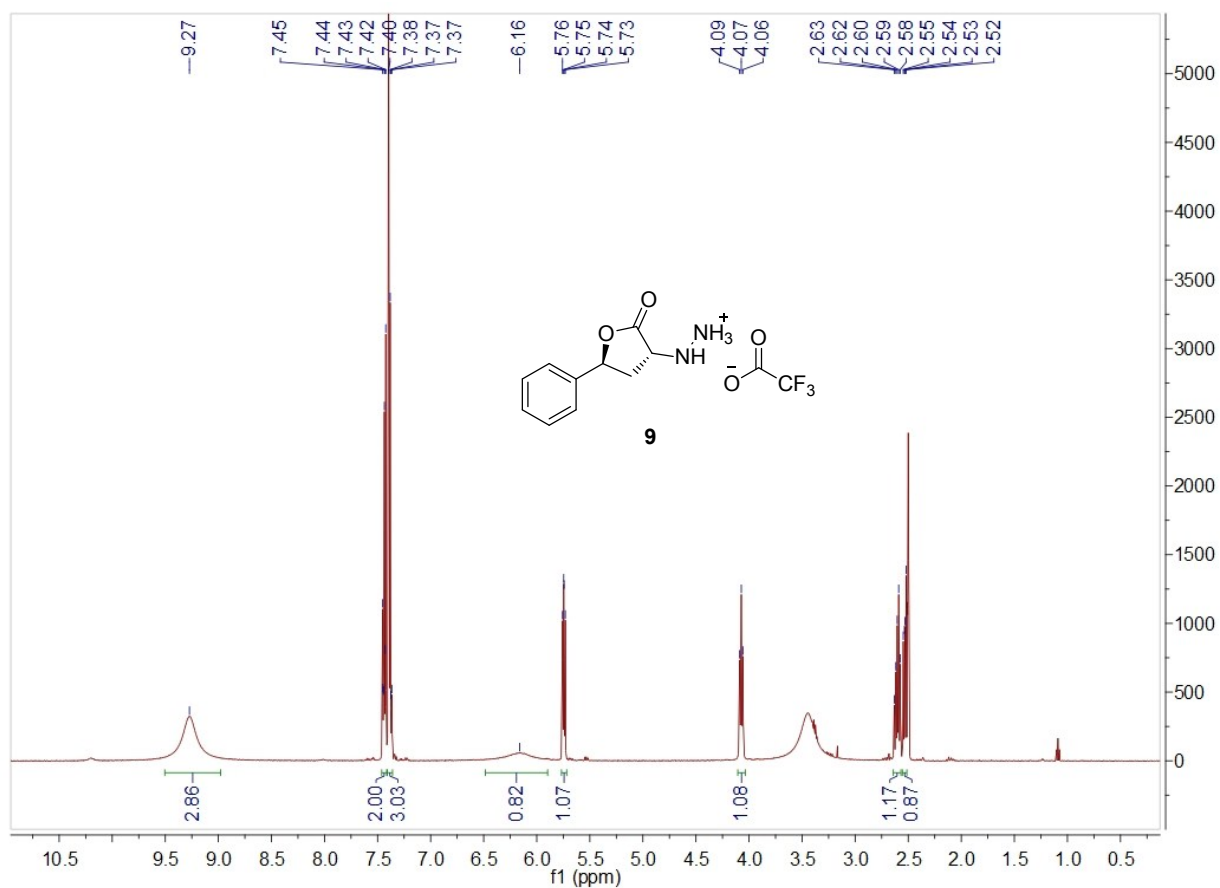


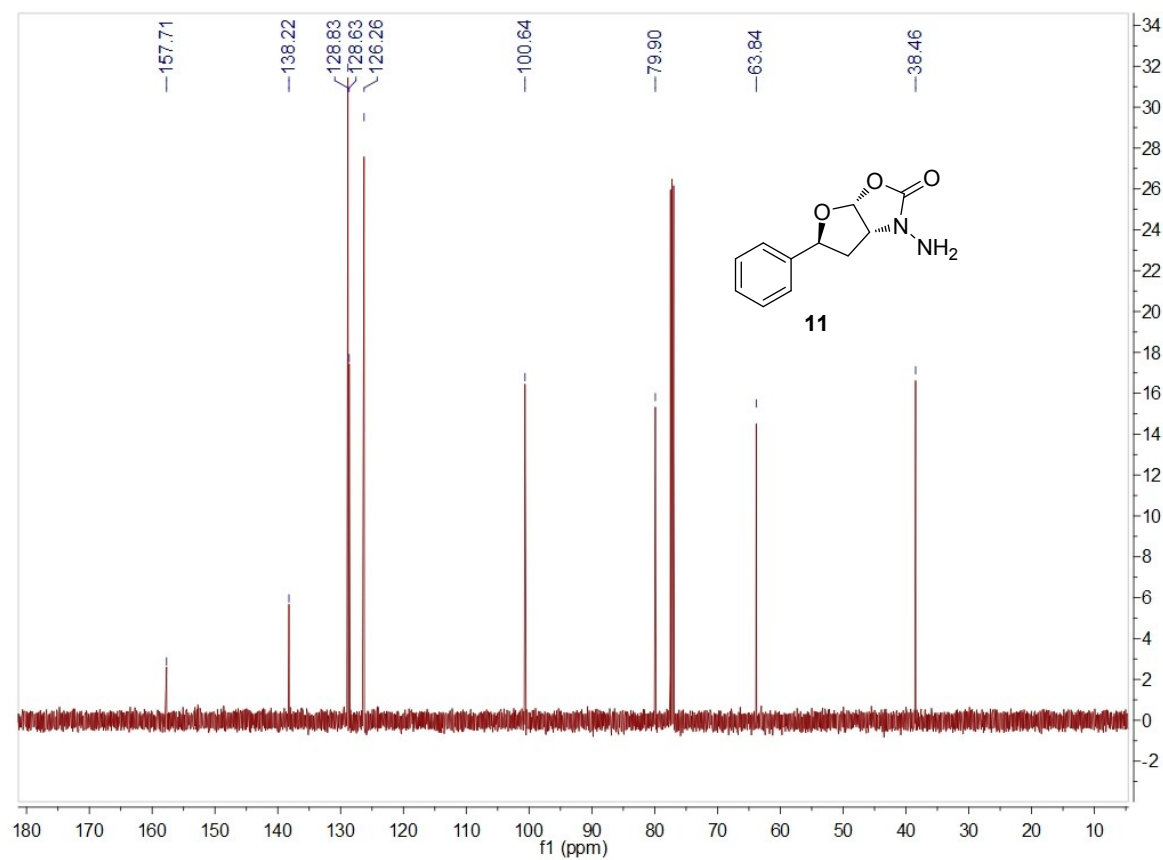
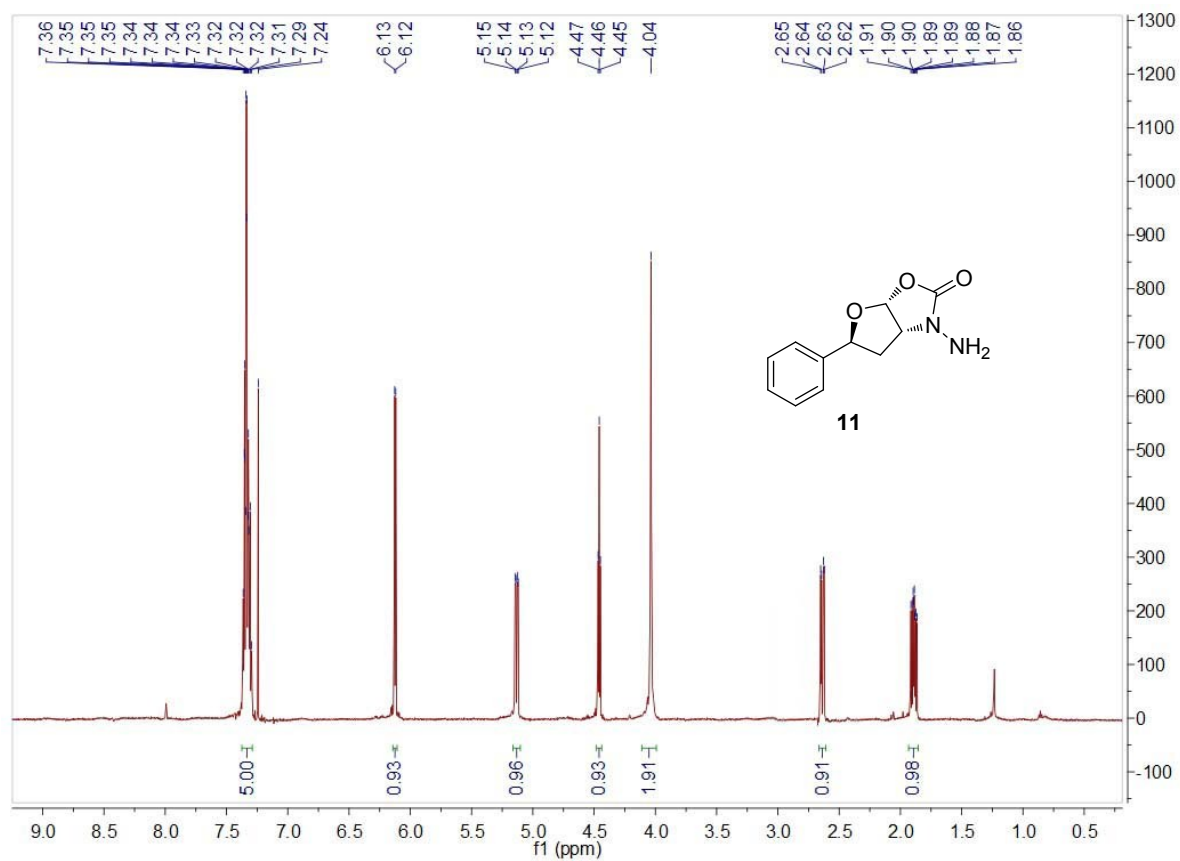
Chrom Type: Fixed WL Chromatogram, 210 nm

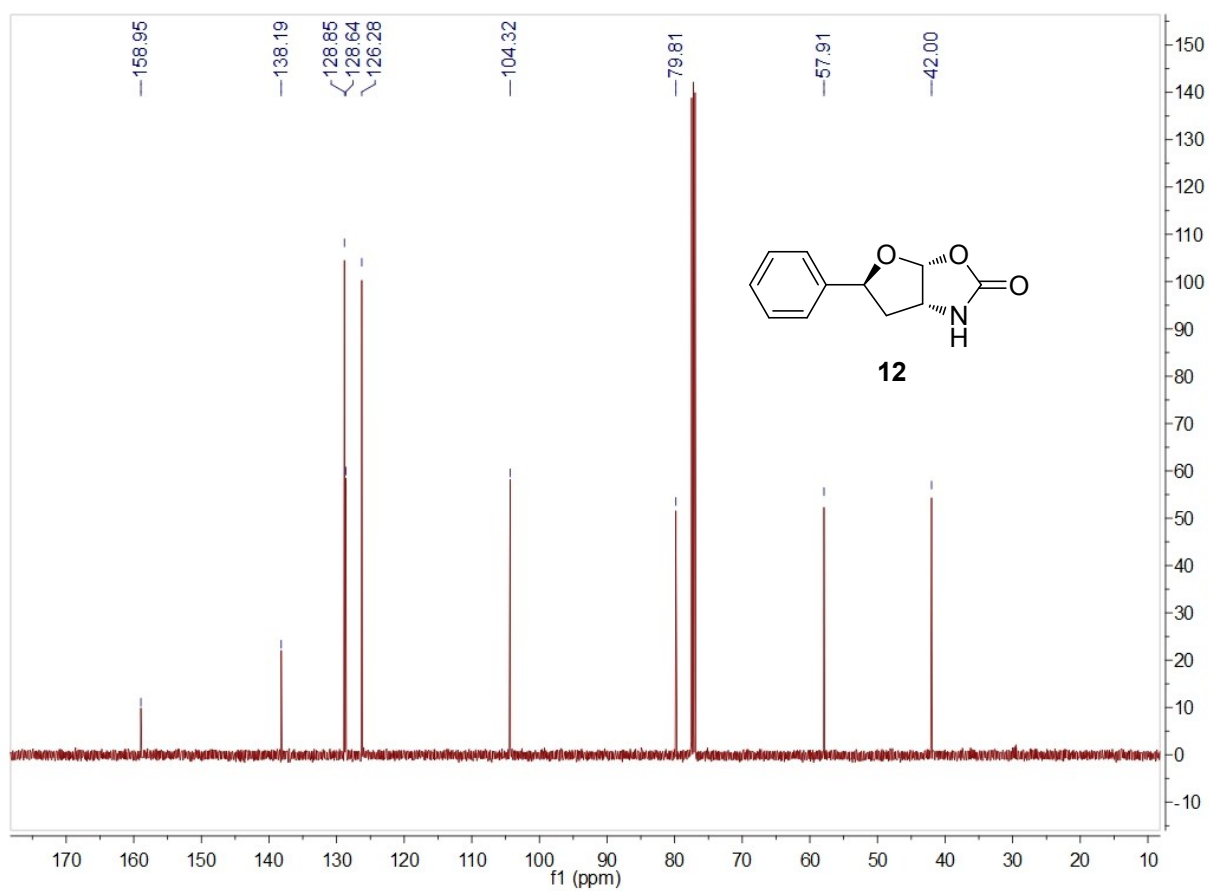
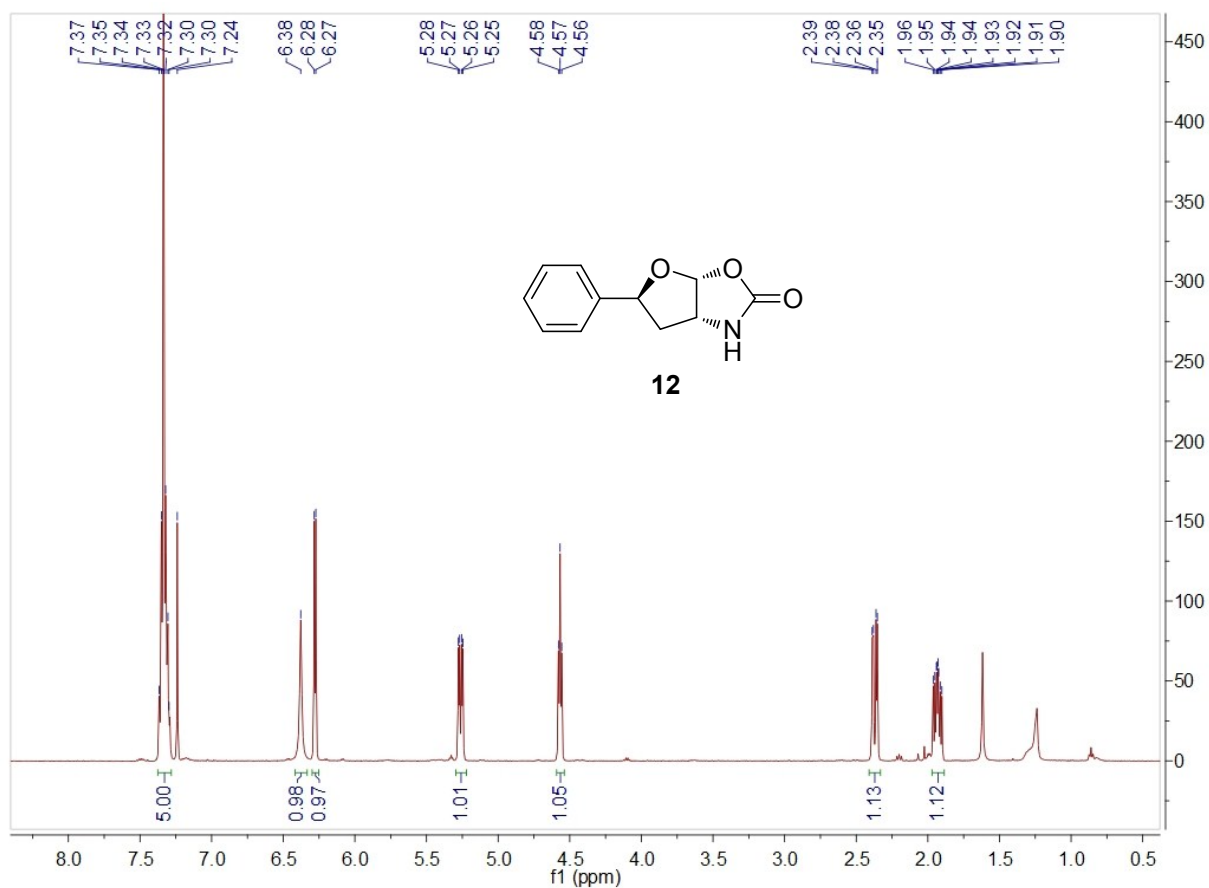
Peak Quantitation: AREA

Calculation Method: AREA%

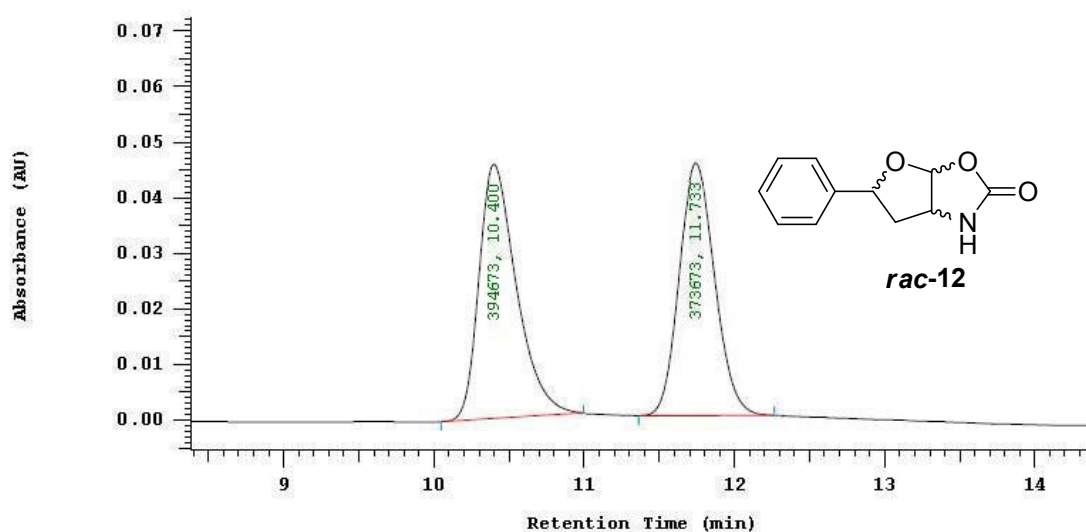
No.	RT	Area	Area %	BC
1	7.193	3220771	96.691	BB
2	8.340	110221	3.309	BB
3330992			100.000	







Chrom Type: Fixed WL Chromatogram, 210 nm



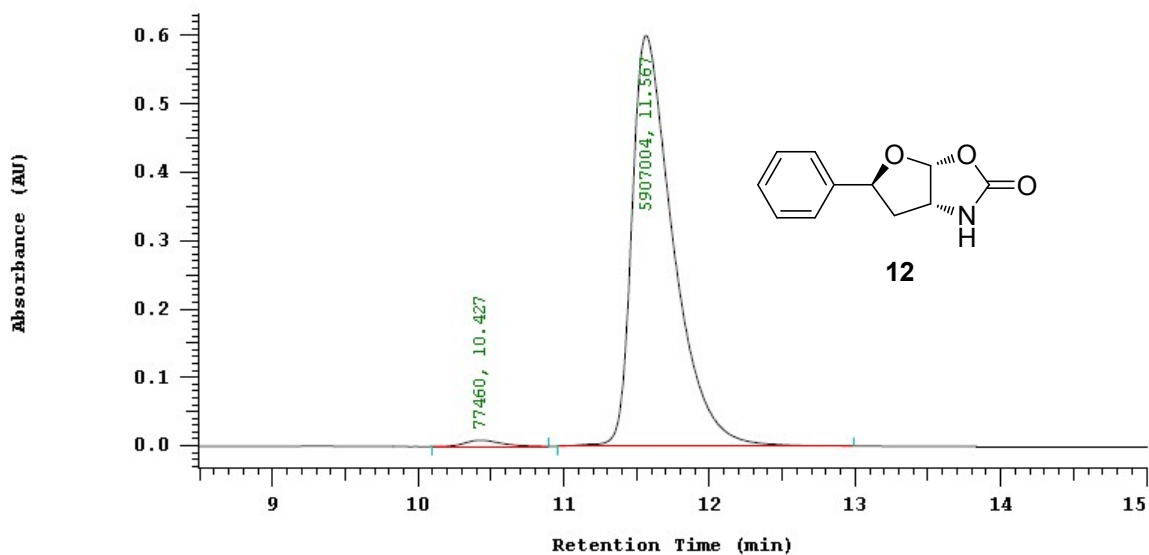
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	10.400	394673	51.367	BB
2	11.733	373673	48.633	BB
768346			100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm

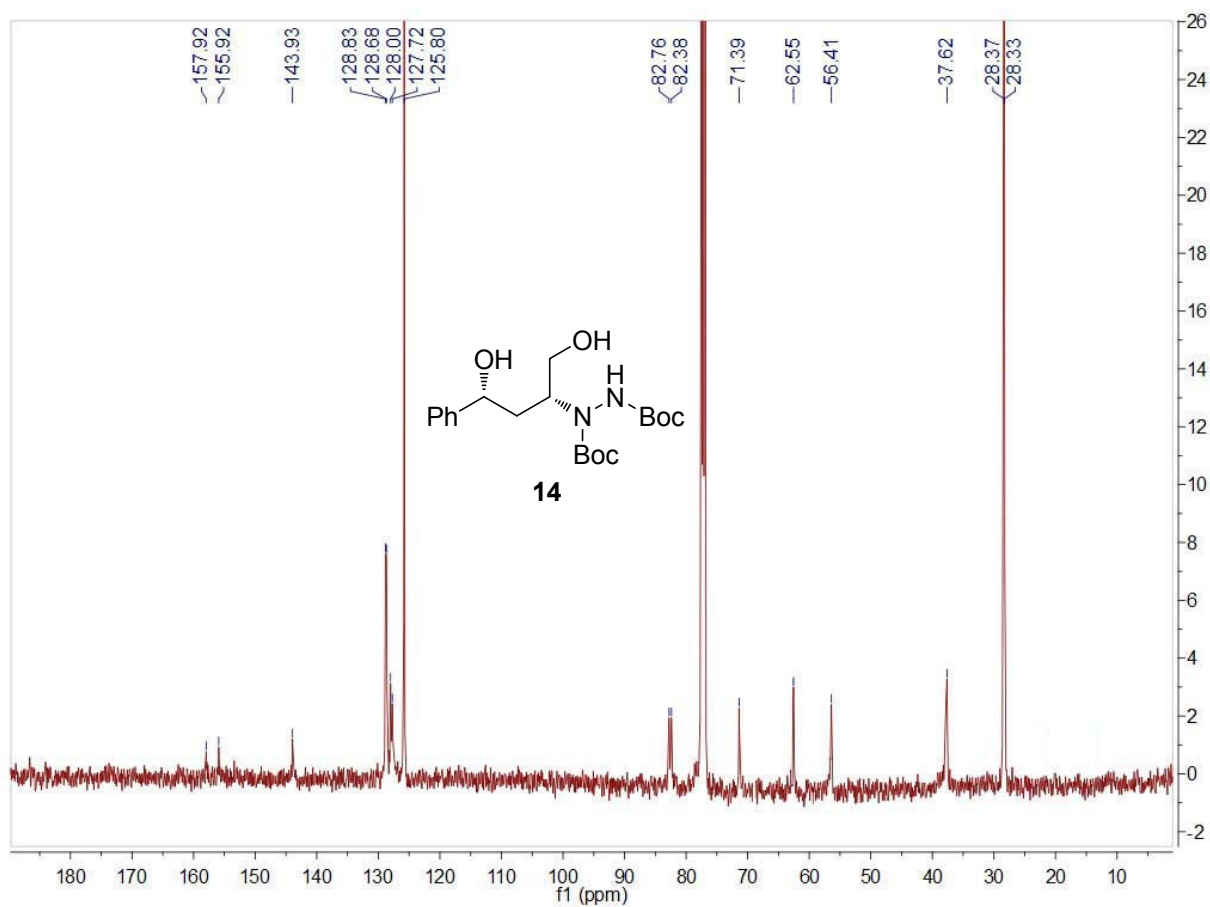
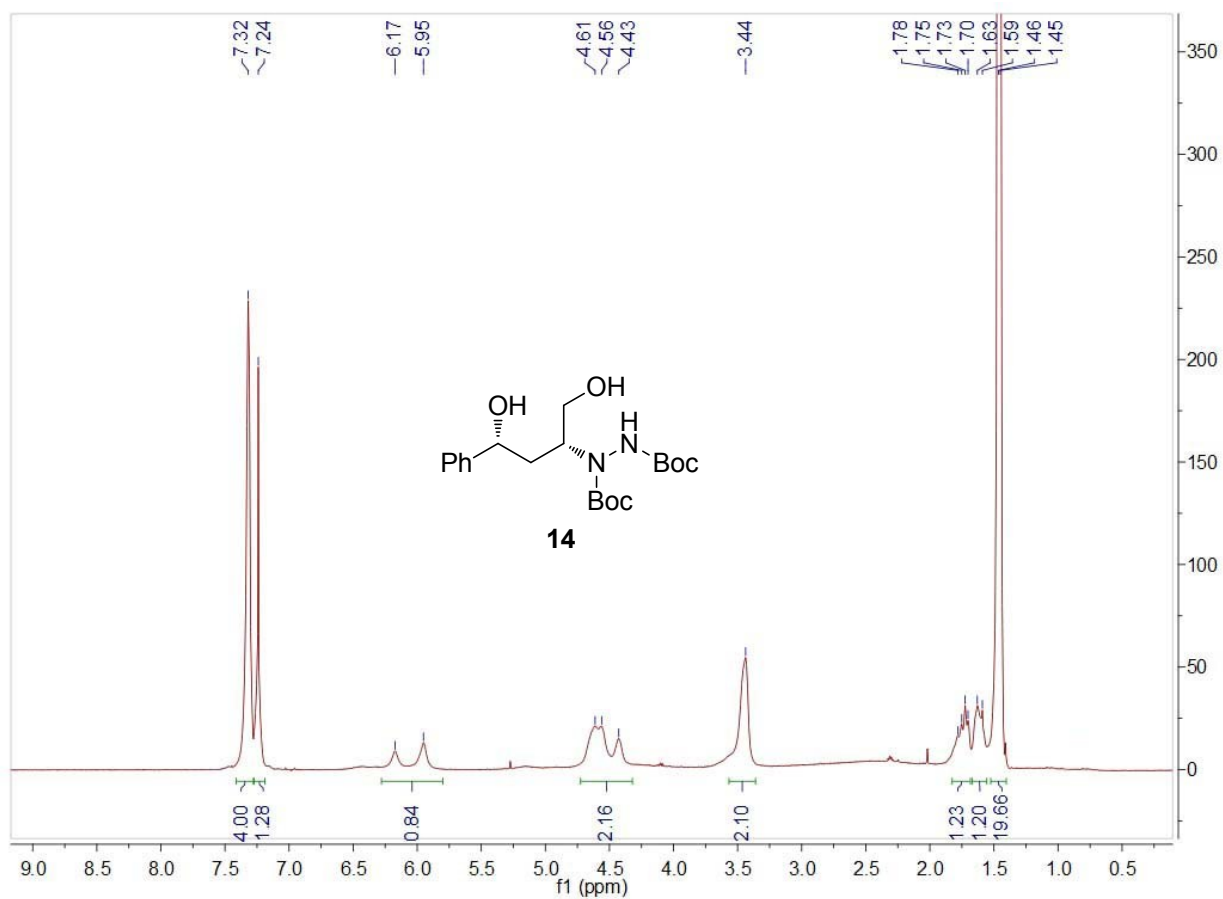


Chrom Type: Fixed WL Chromatogram, 210 nm

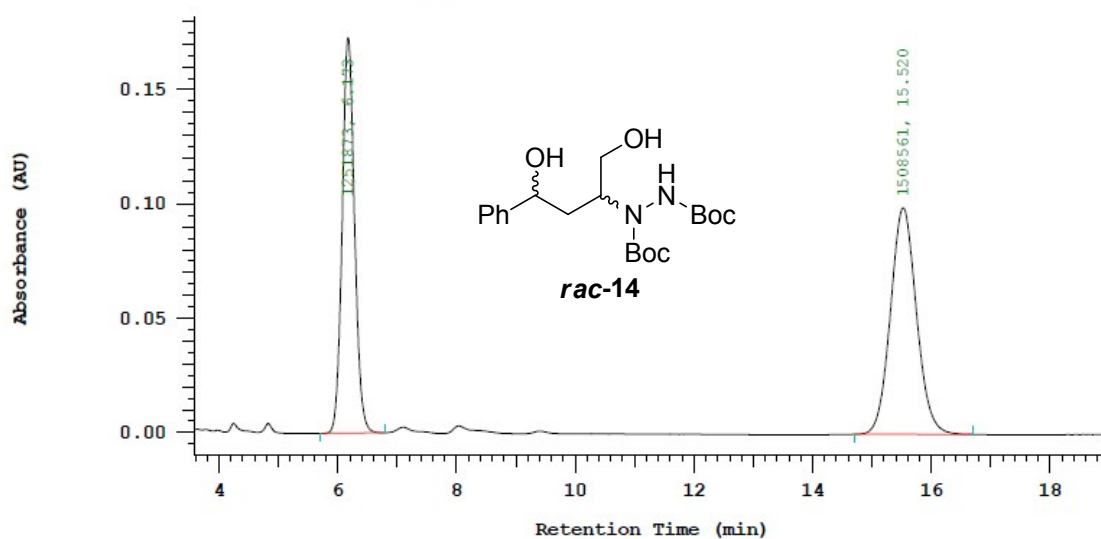
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	10.427	77460	1.294	BB
2	11.567	5907004	98.706	BB
5984464			100.000	



Chrom Type: Fixed WL Chromatogram, 210 nm



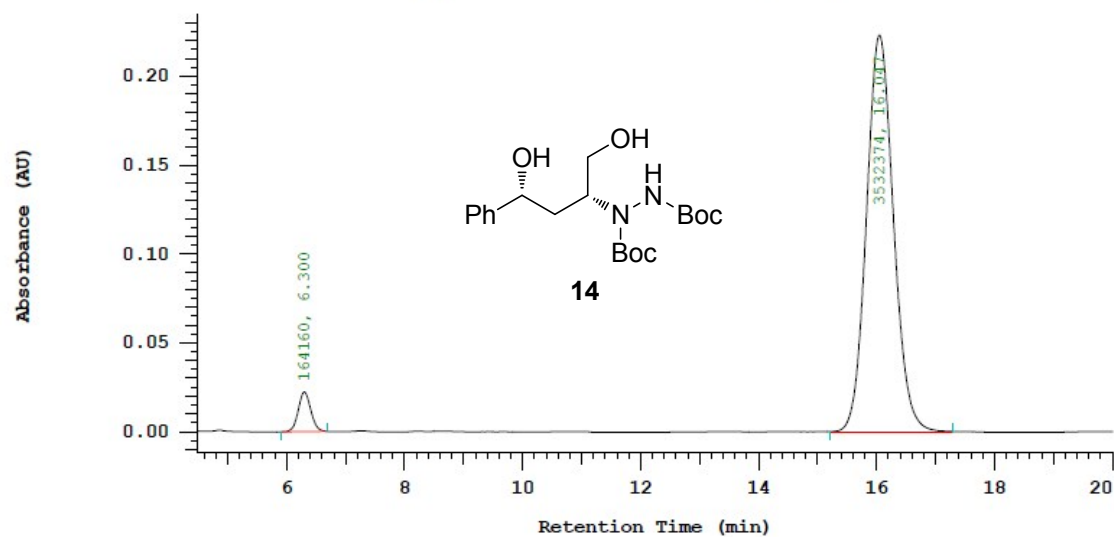
Chrom Type: Fixed WL Chromatogram, 210 nm

Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.173	1251873	45.351	BB
2	15.520	1508561	54.649	BB
		2760434	100.000	

Chrom Type: Fixed WL Chromatogram, 210 nm



Chrom Type: Fixed WL Chromatogram, 210 nm

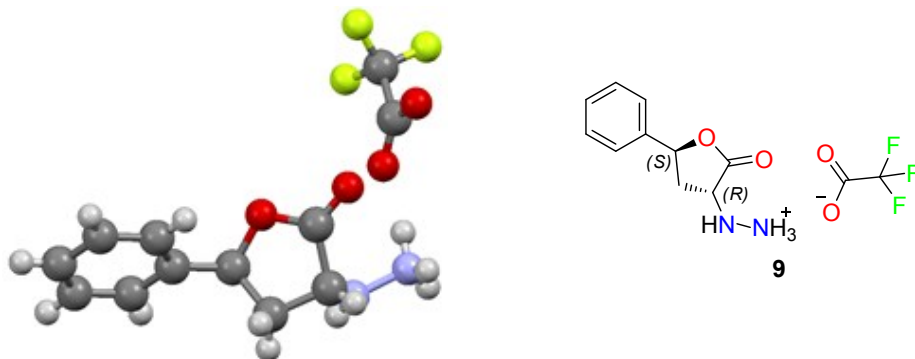
Peak Quantitation: AREA

Calculation Method: AREA%

No.	RT	Area	Area %	BC
1	6.300	164160	4.441	BB
2	16.047	3532374	95.559	BB
		3696534	100.000	

5. Single Crystal X-Ray Diffraction Data

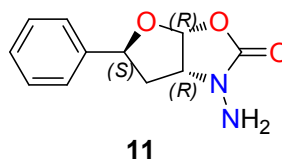
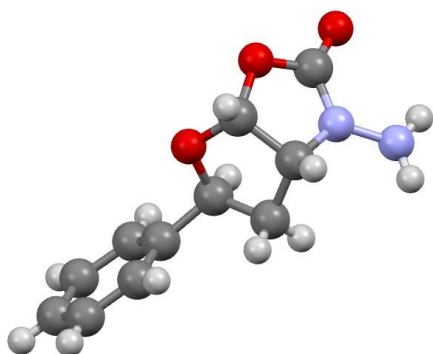
Single crystal X-ray structure (ORTEP) of **9**. X-ray crystallographic data have been deposited in the Cambridge Crystallographic Data Centre database (http://www.ccdc.cam.ac.uk/data_request/cif.) under accession code CCDC 1436994.



Crystal data and structure refinement for **9**

Bond precision:	C-C = 0.0060 Å	Wavelength=1.54184
Cell:	a=8.1994(5) b=5.9821(4) c=13.9212(9)	
	alpha=90 beta=93.572(6) gamma=90	
Temperature: 293 K		
	Calculated	Reported
Volume	681.50(8)	681.50(7)
Space group	P 21	P21
Hall group	P 2yb	?
Moiety formula	C10 H13 N2 O2, C2 F3 O2	?
Sum formula	C12 H13 F3 N2 O4	C12 H13 F3 N2 O4
Mr	306.24	306.24
Dx, g cm ⁻³	1.492	1.492
Z	2	2
Mu (mm ⁻¹)	1.216	1.216
F000	316.0	316.0
F000'	317.34	
h,k,lmax	9,7,16	9,7,16
Nref	2427[1340]	2310
Tmin,Tmax	0.864,0.875	0.868,0.878
Tmin'	0.864	
Correction method= # Reported T Limits: Tmin=0.868 Tmax=0.878 AbsCorr =		
MULTI-SCAN		
Data completeness= 1.72/0.95	Theta(max)= 67.230	
R(reflections)= 0.0547(1962)	wR2(reflections)= 0.1662(2310)	
S = 1.046	Npar= 192	

Single crystal X-ray structure (ORTEP) of **11**. X-ray crystallographic data have been deposited in the Cambridge Crystallographic Data Centre database (http://www.ccdc.cam.ac.uk/data_request/cif.) under accession code CCDC 1437043.



Crystal data and structure refinement for 11

Bond precision:	C-C = 0.0043 Å	Wavelength=1.54184
Cell:	a=5.4617(3) b=8.2109(5)	c=23.4585(11)
	alpha=90 beta=90	gamma=90
Temperature: 293 K		
	Calculated	Reported
Volume	1052.01(10)	1052.01(10)
Space group	P 21 21 21	P212121
Hall group	P 2ac 2ab	?
Moiety formula	C11 H12 N2 O3	?
Sum formula	C11 H12 N2 O3	C11 H12 N2 O3
Mr	220.23	220.23
Dx, g cm ⁻³	1.390	1.390
Z	4	4
Mu (mm ⁻¹)	0.857	0.857
F000	464.0	464.0
F000'	465.55	
h,k,lmax	6,9,28	6,9,28
Nref	1892[1140]	1882
Tmin,Tmax	0.934,0.942	0.935,0.942
Tmin'	0.934	
Correction method= # Reported T Limits: Tmin=0.935 Tmax=0.942 AbsCorr =		
MULTI-SCAN		
Data completeness= 1.65/0.99	Theta(max)= 67.240	
R(reflections)= 0.0457(1531)	wR2(reflections)= 0.1229(1882)	
S = 1.046	Npar= 146	