Supplementary Material

Protecting-group directed stereospecific organocatalytic [3+2] cycloadditions: a facile access to chiral spirocyclic oxindoles

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Dedicated to Professor Pierre Vogel on the occasion of his 70th birthday

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X-ray structure (3c)

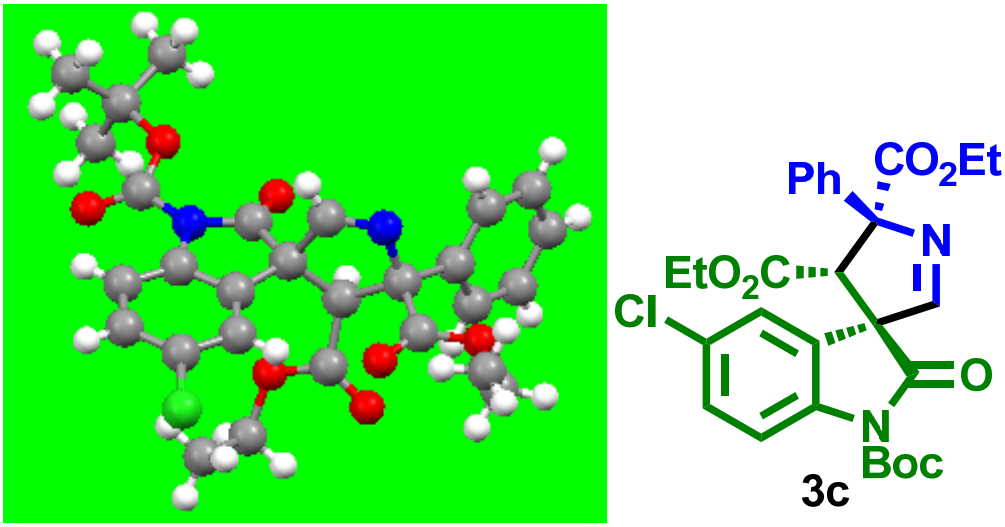


Table 1. Crystal data and structure refinement for zgf73s.

Identification code	zgf73s	
Empirical formula	C28 H29 Cl N2 O7	
Formula weight	540.98	
Temperature	103(2) K	
Wavelength	0.71073 Å	
Crystal system	Orthorhombic	
Space group	P2(1)2(1)2(1)	
Unit cell dimensions	a = 11.9680(3) Å	α= 90°.
	b = 13.1292(3) Å	β= 90°.
	c = 17.2482(3) Å	γ = 90°.
Volume	2710.21(10) $Å^3$	
Z	4	
Density (calculated)	1.326 Mg/m^3	
Absorption coefficient	0.190 mm^{-1}	
F(000)	1136	
Crystal size	0.40 x 0.40 x 0.32 mm^3	
Theta range for data collection	1.95 to 32.50°.	
Index ranges	-15<=h<=17, -16<=k<=19, -13<=l<=26	
Reflections collected	34101	
Independent reflections	9729 [R(int) = 0.0334]	
Completeness to theta = 32.50°	99.7 %	

Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9418 and 0.9280
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	9729 / 0 / 348
Goodness-of-fit on F^2	1.085
Final R indices [I>2sigma(I)]	R1 = 0.0365, wR2 = 0.0964
R indices (all data)	R1 = 0.0436, wR2 = 0.1079
Absolute structure parameter	0.00(4)
Largest diff. peak and hole	0.458 and -0.485 e.Å^{-3}

X-ray structure of the Bn-protected substrate

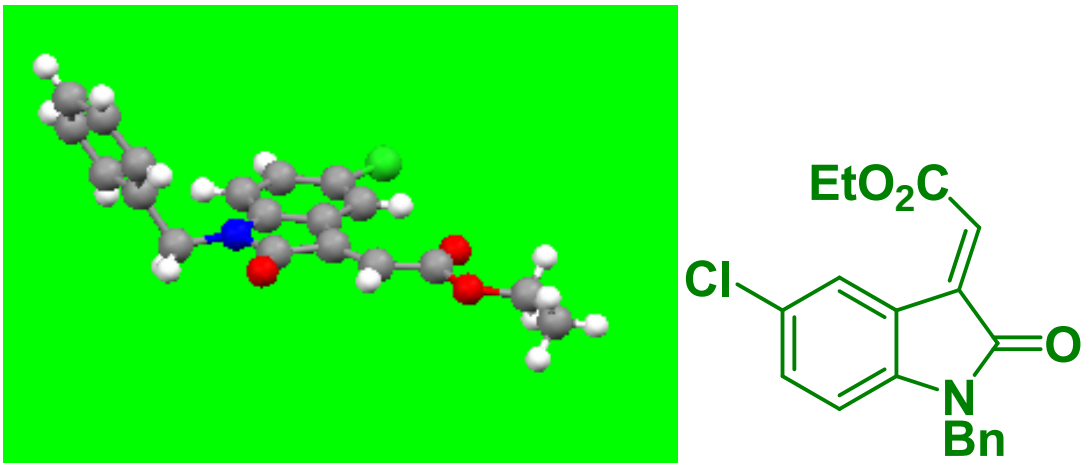
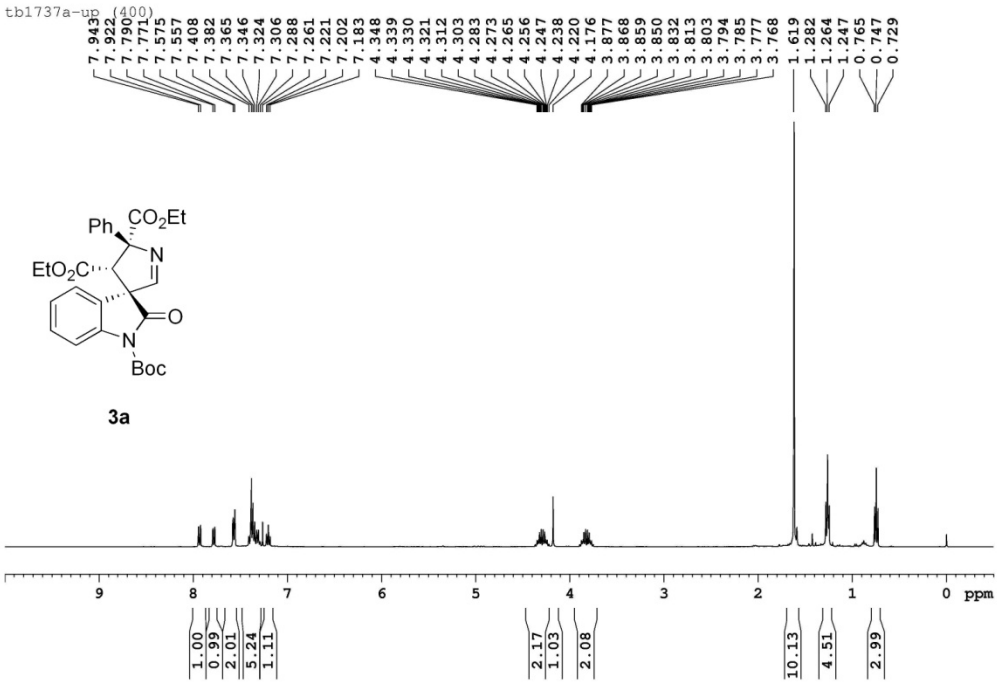


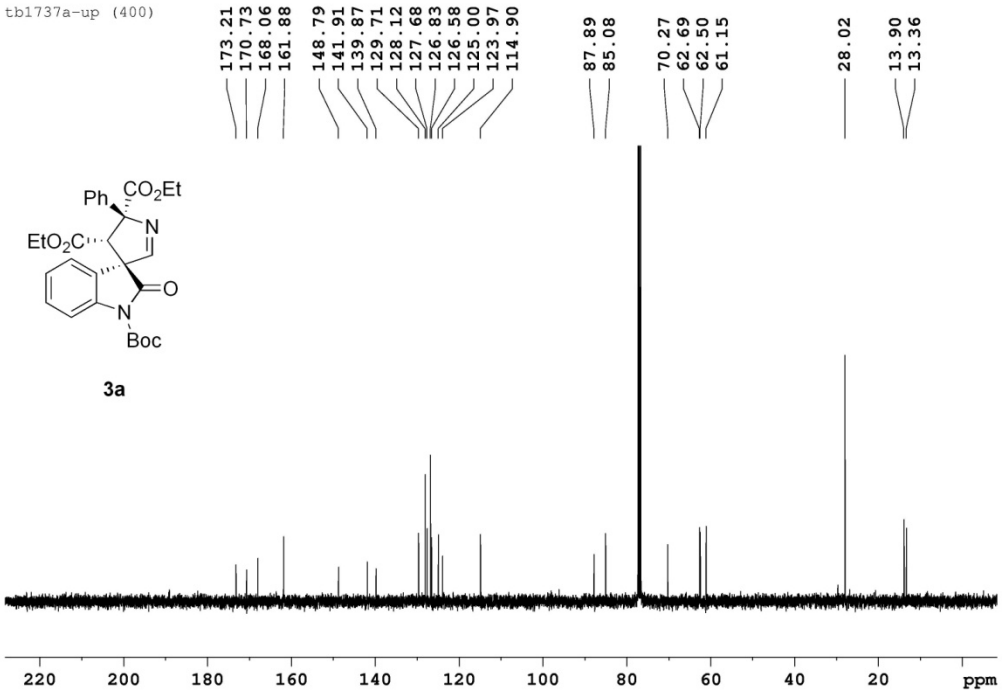
Table 2. Crystal data and structure refinement for zgf77.

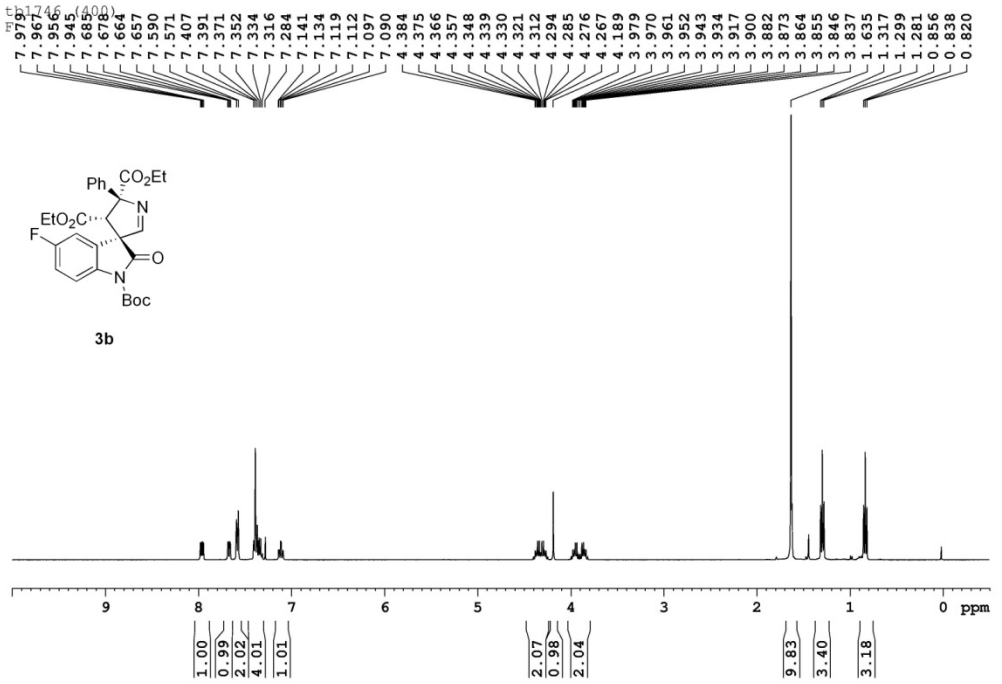
Identification code	zgf77	
Empirical formula	C19 H16 Cl N O3	
Formula weight	341.78	
Temperature	103(2) K	
Wavelength	0.71073 Å	
Crystal system	Monoclinic	
Space group	P2(1)	
Unit cell dimensions	a = 7.3254(7) Å	α= 90°.
	b = 6.0096(5) Å	β= 95.261(5)°.
	c = 18.1224(17) Å	γ = 90°.
Volume	794.44(13) Å^3	

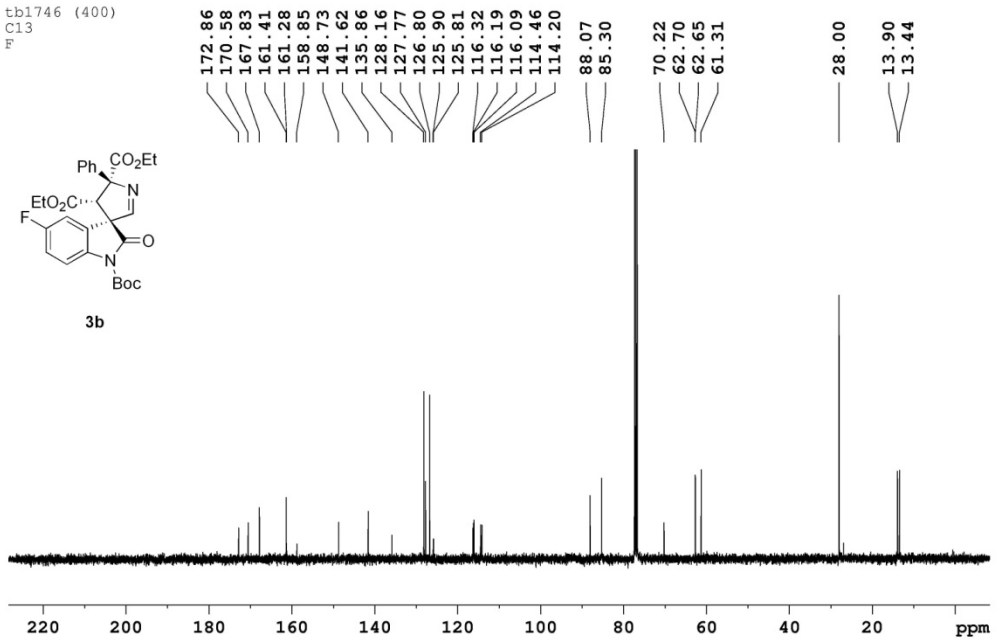
Z	2
Density (calculated)	1.429 Mg/m^3
Absorption coefficient	0.258 mm^{-1}
F(000)	356
Crystal size	0.40 x 0.20 x 0.02 mm^3
Theta range for data collection	2.26 to 34.22°.
Index ranges	-11<=h<=11, -8<=k<=9, -28<=l<=28
Reflections collected	20373
Independent reflections	6273 [R(int) = 0.0792]
Completeness to theta = 34.22°	98.9 %
Absorption correction	Semi-empirical from equivalents
Max. and min. transmission	0.9949 and 0.9039
Refinement method	Full-matrix least-squares on F^2
Data / restraints / parameters	6273 / 1 / 218
Goodness-of-fit on F^2	1.080
Final R indices [I>2sigma(I)]	R1 = 0.0636, wR2 = 0.1423
R indices (all data)	R1 = 0.0945, wR2 = 0.1630
Absolute structure parameter	-0.03(7)
Largest diff. peak and hole	0.888 and -0.559 $e.Å^{-3}$

NMR Spectra

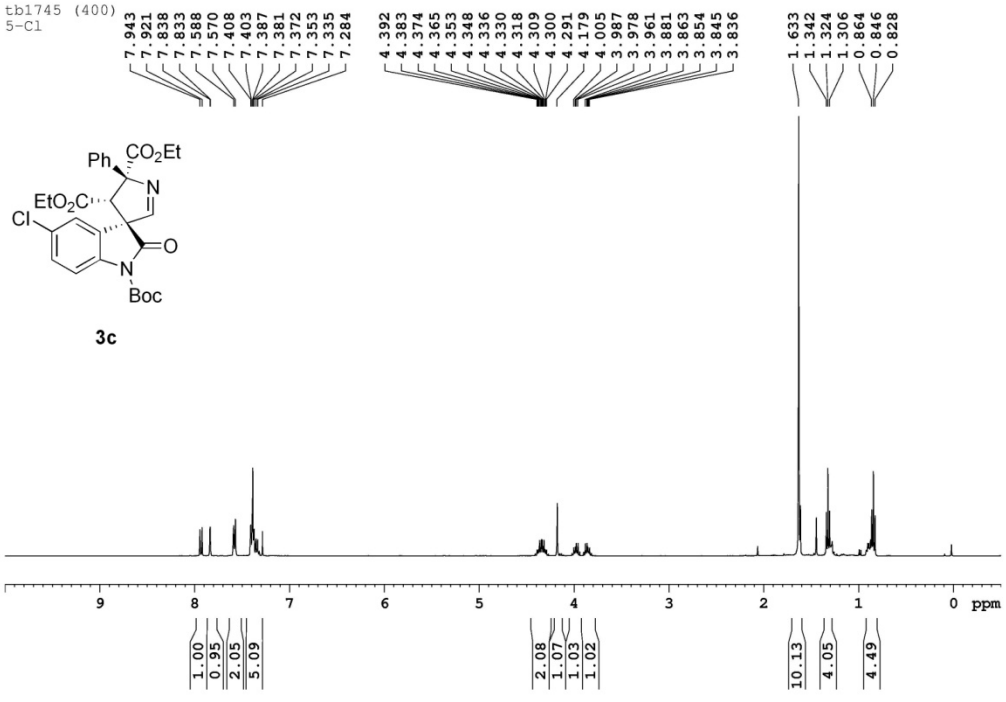








3b



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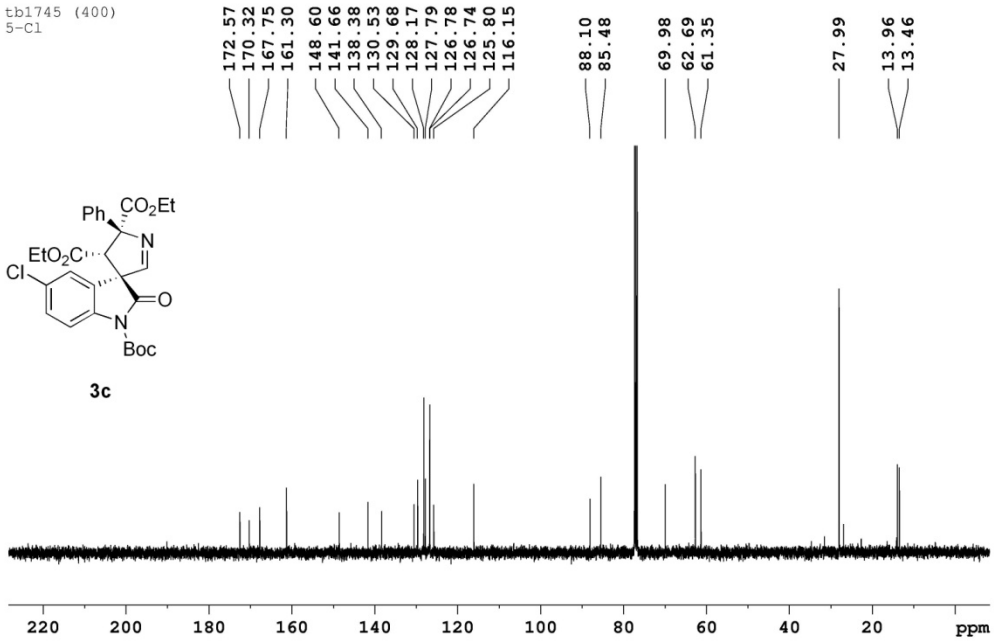

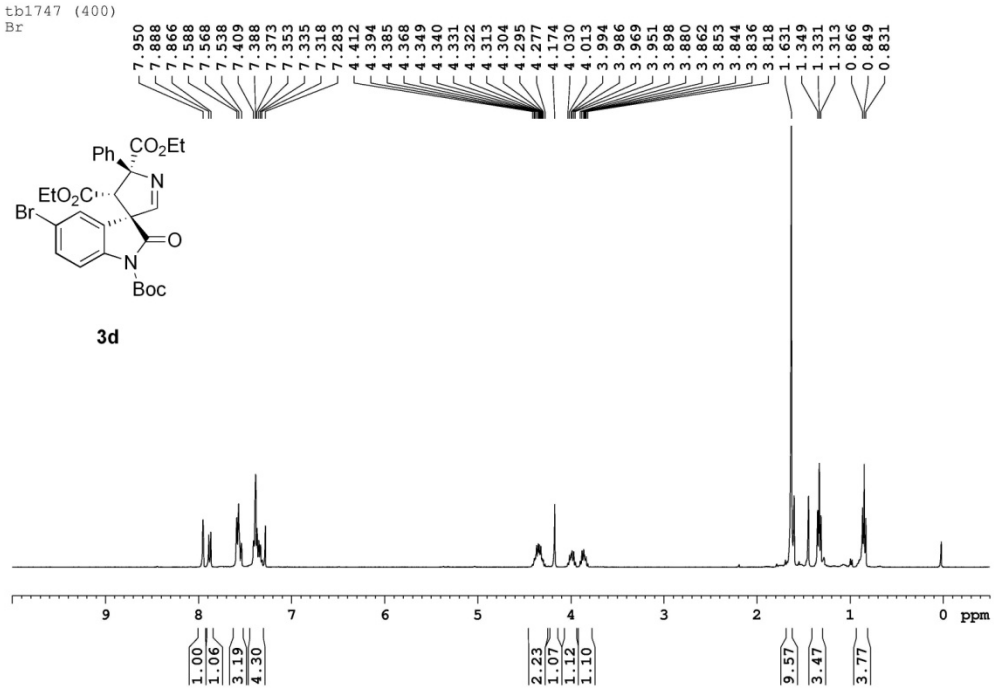

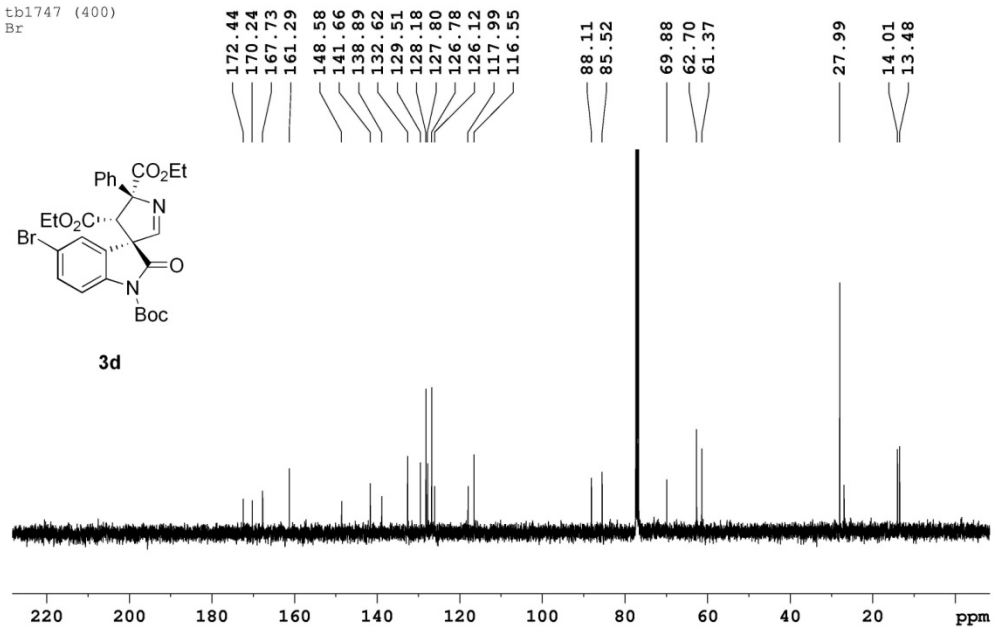

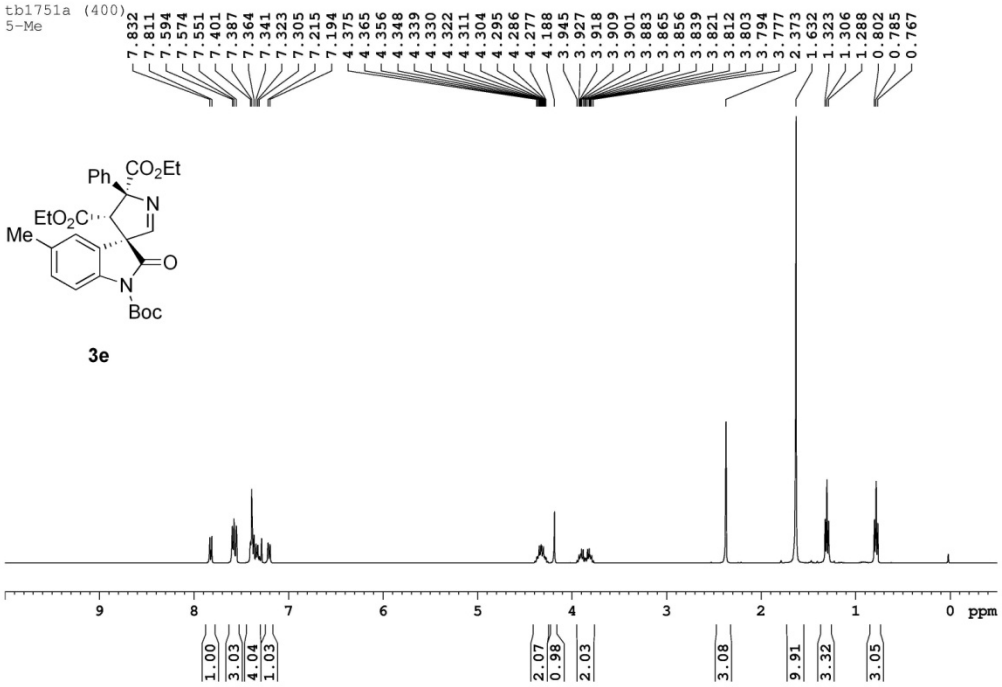

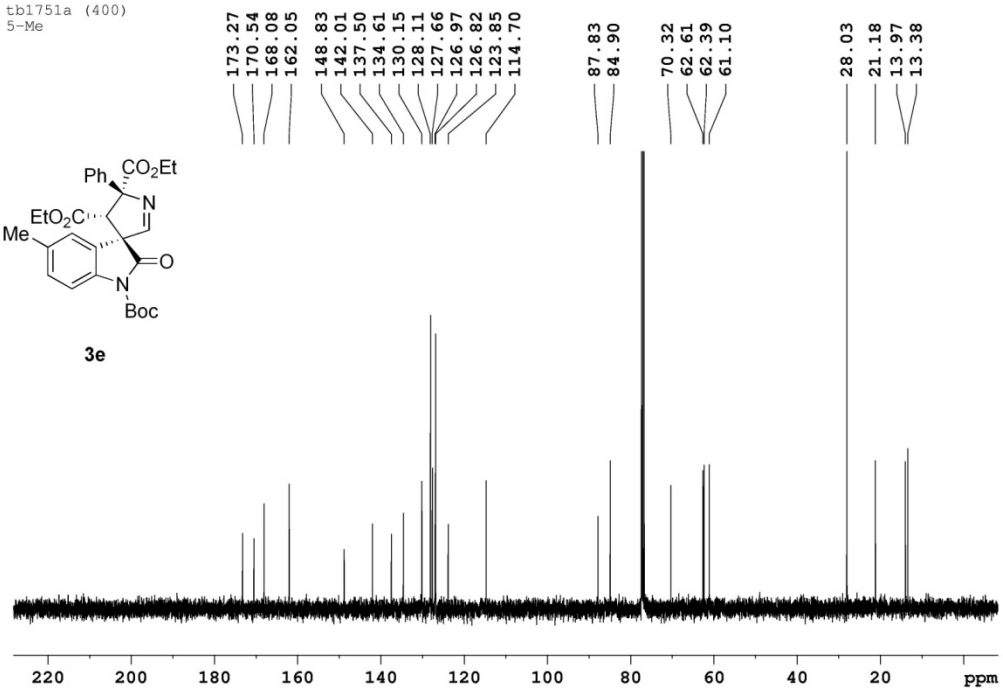

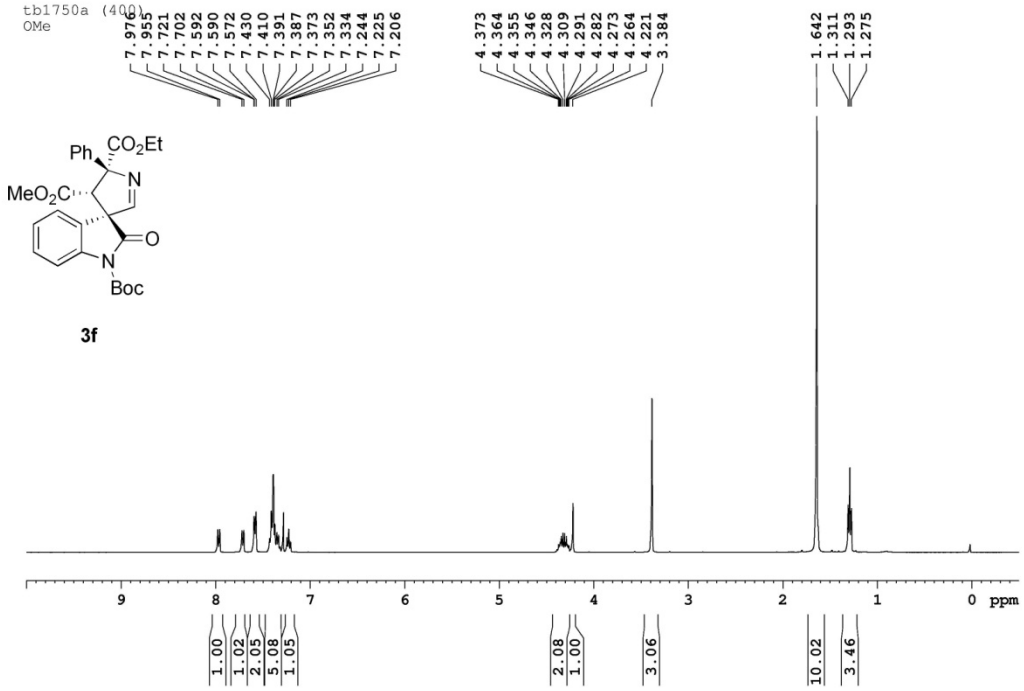

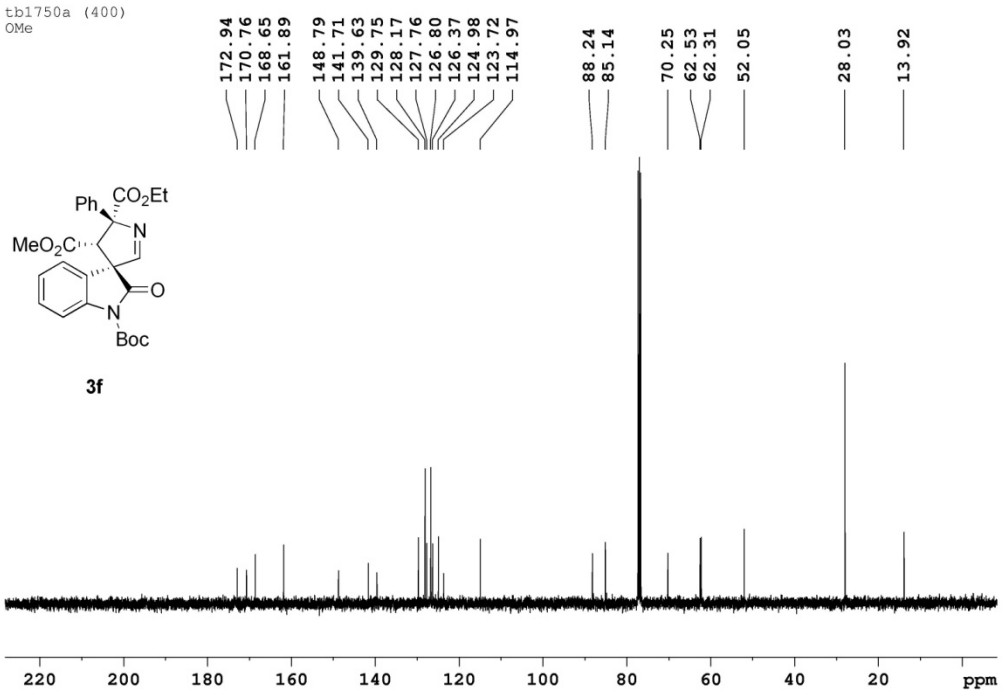

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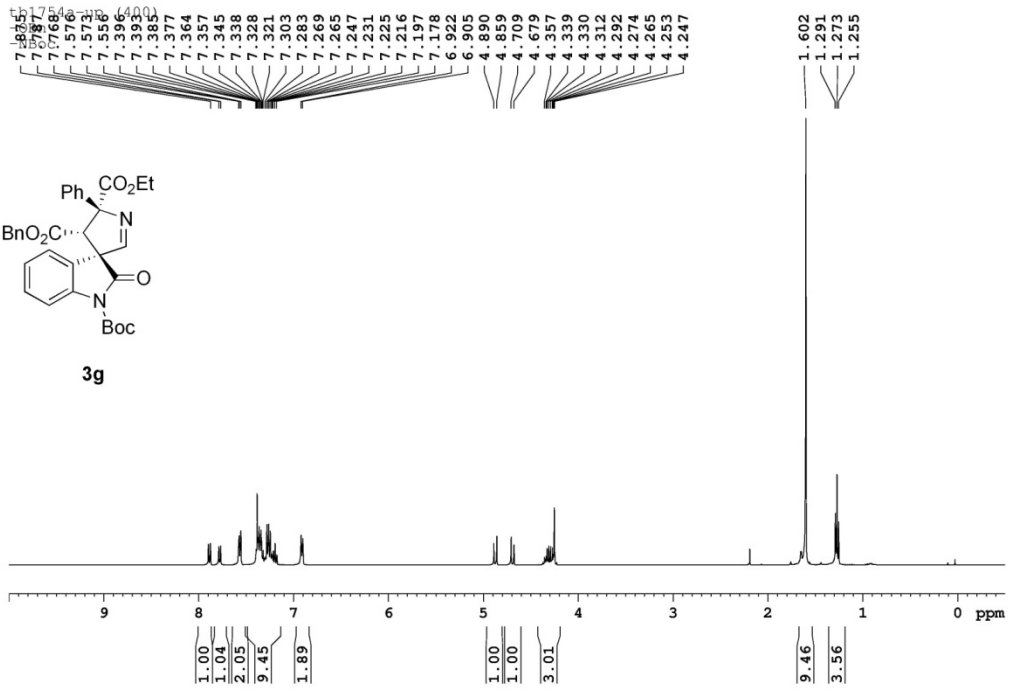

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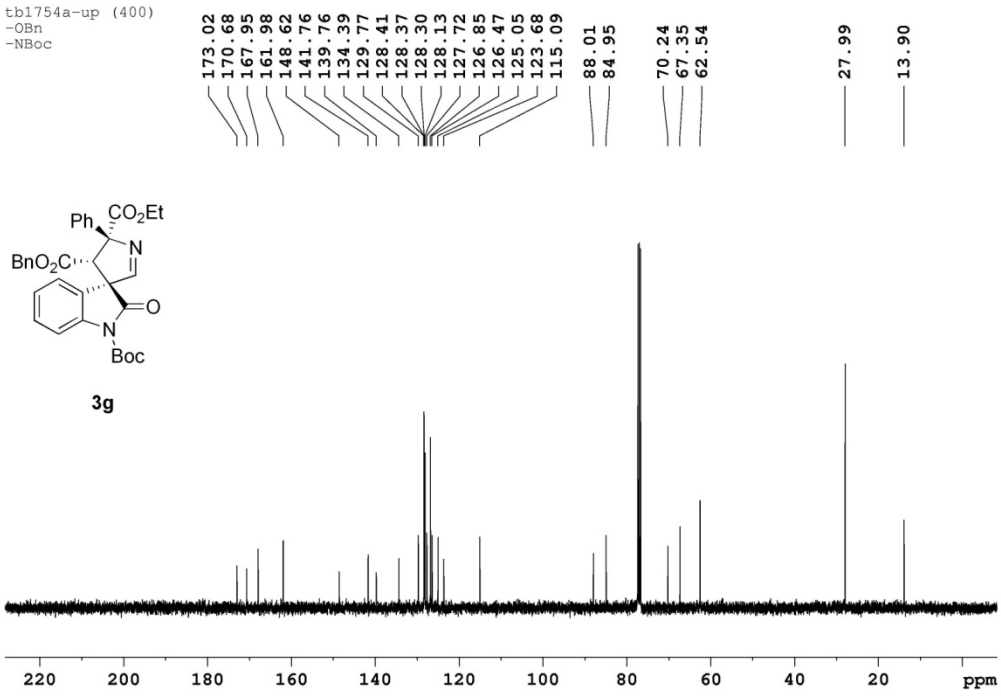

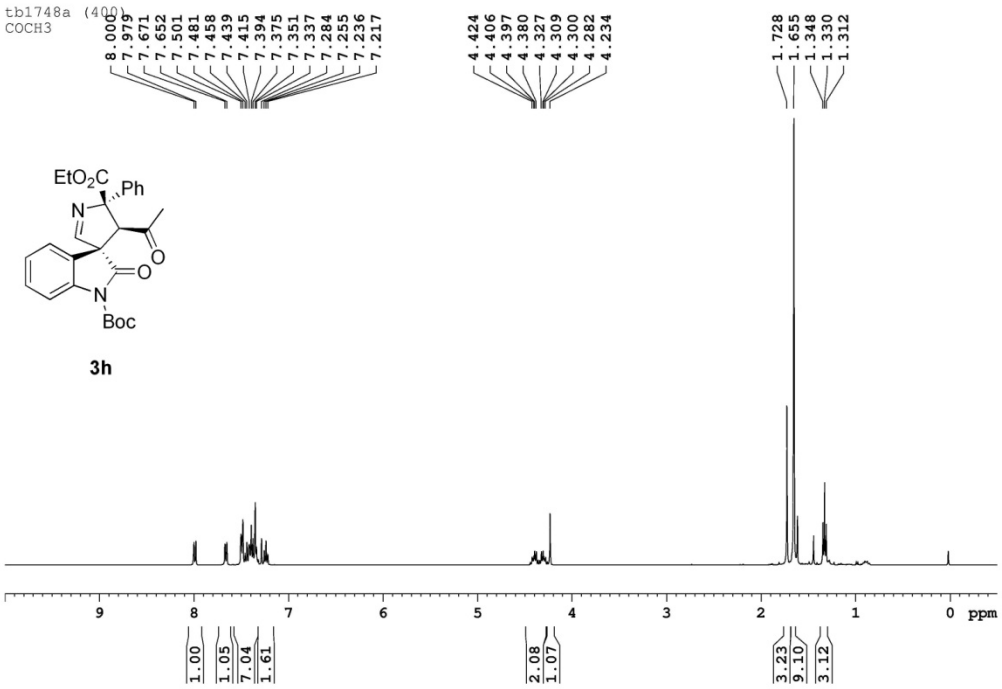

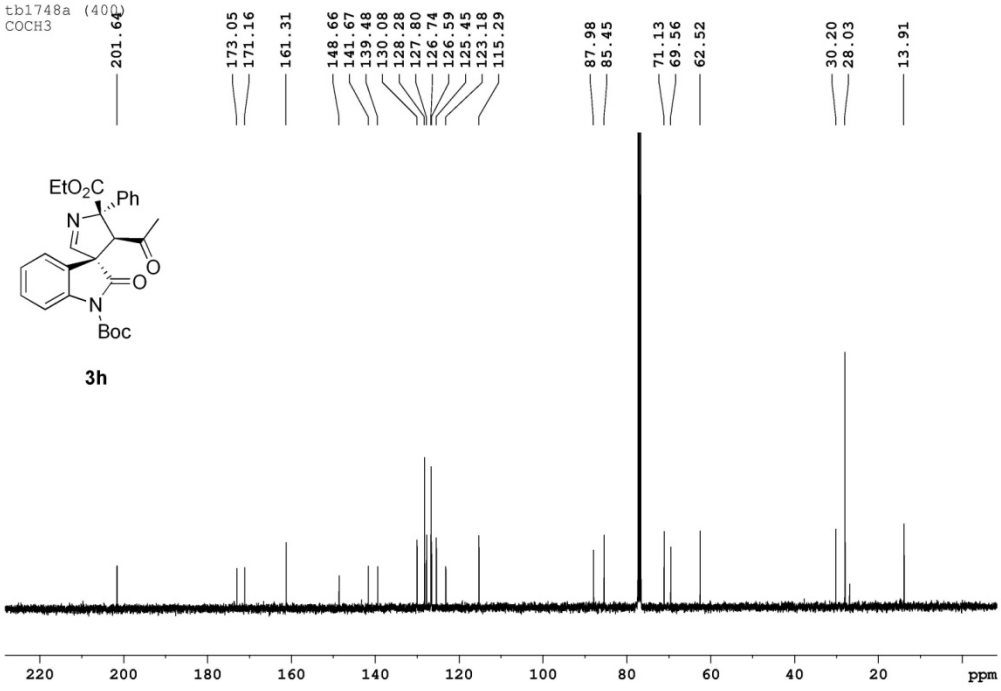

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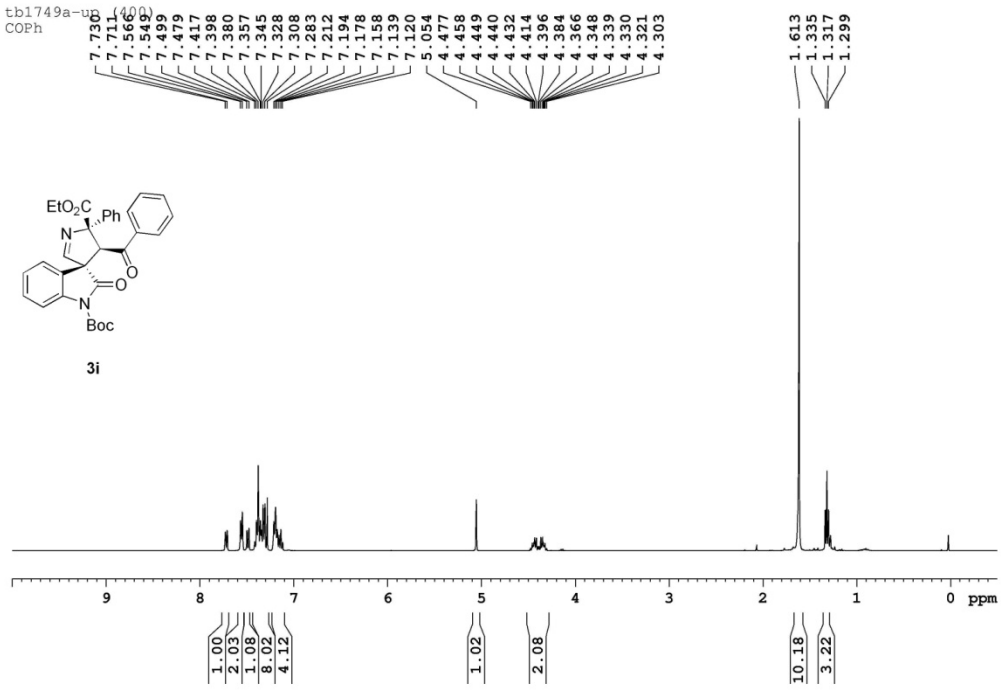

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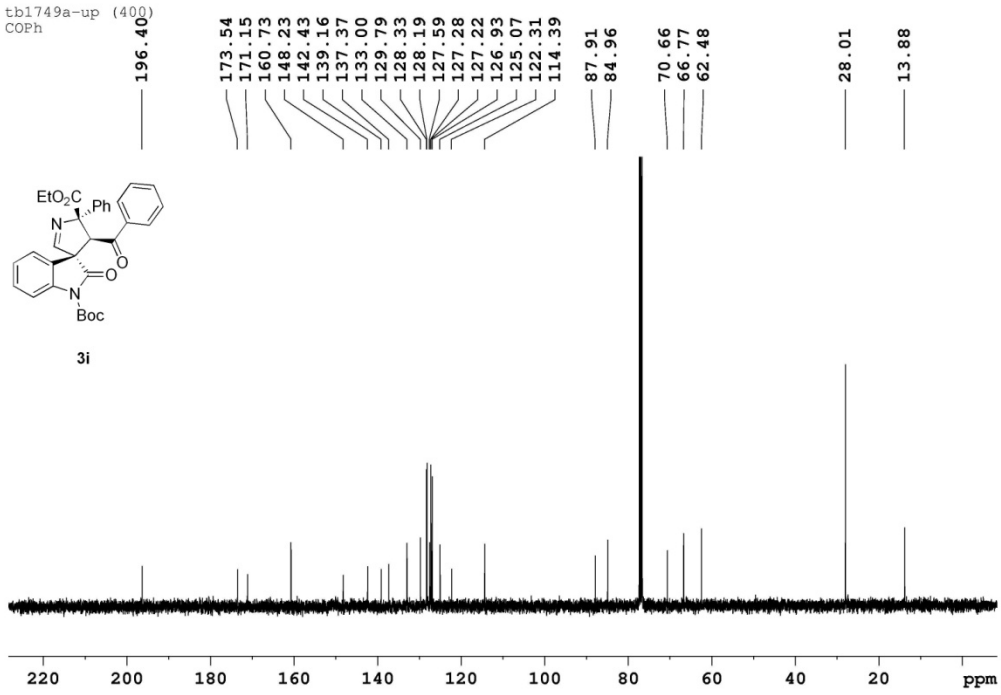
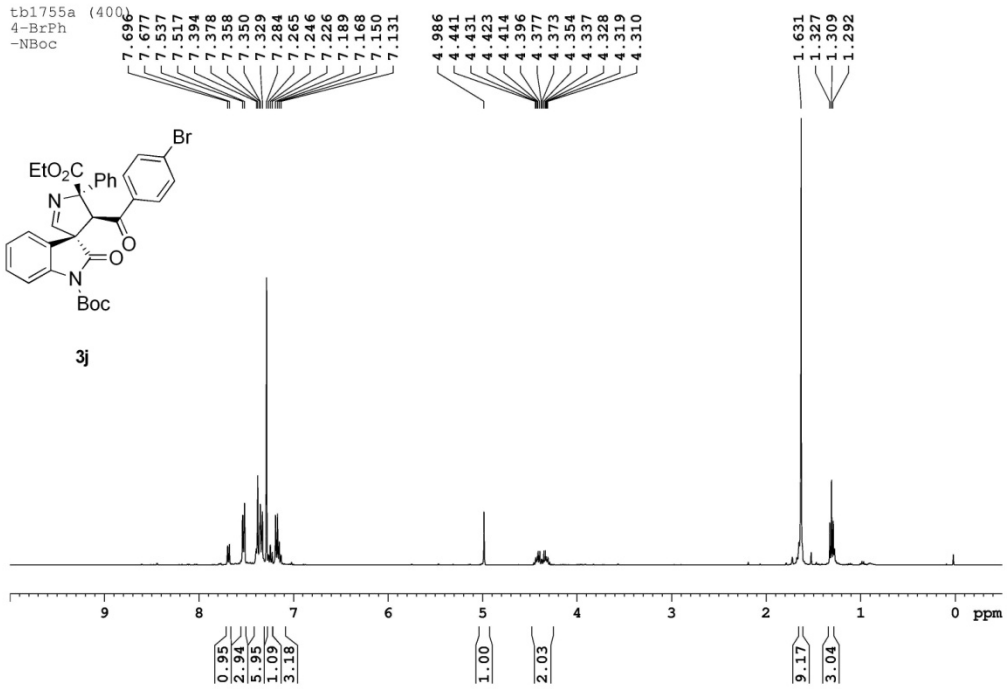
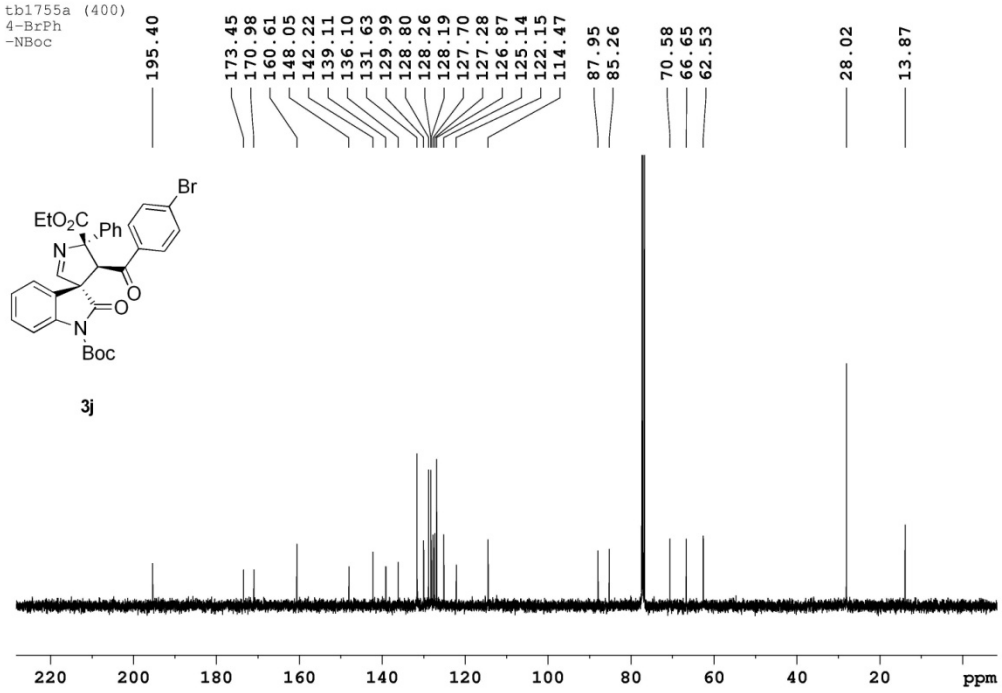
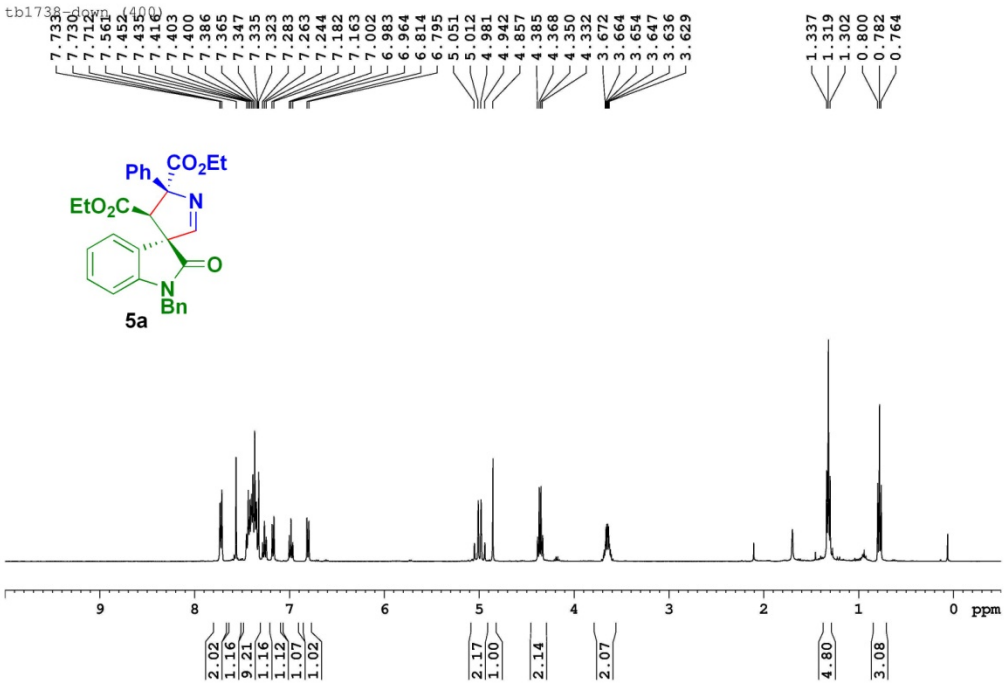
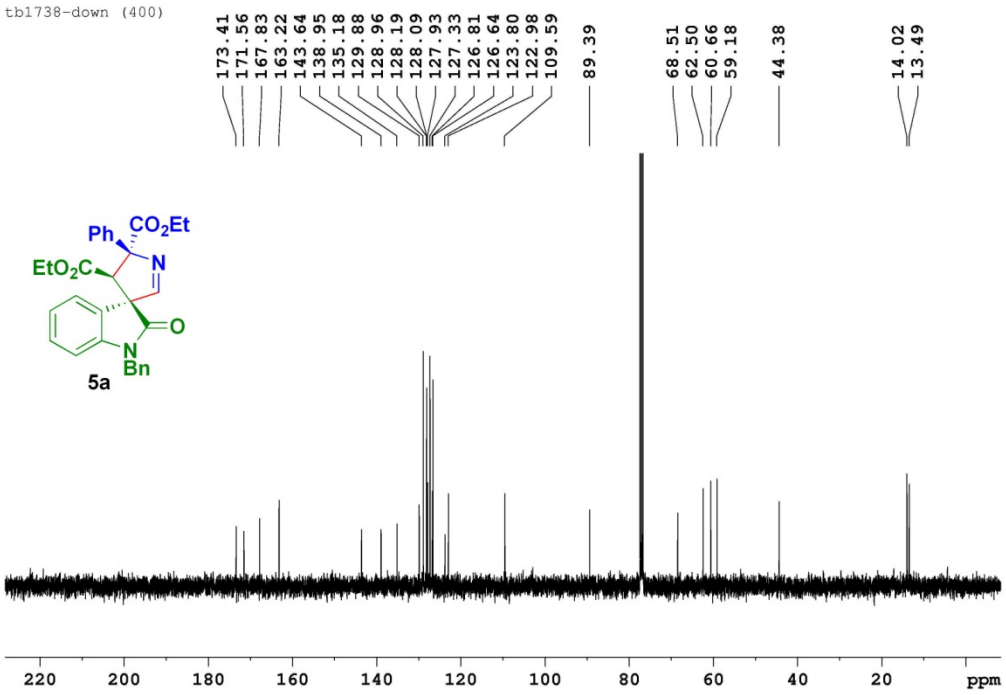
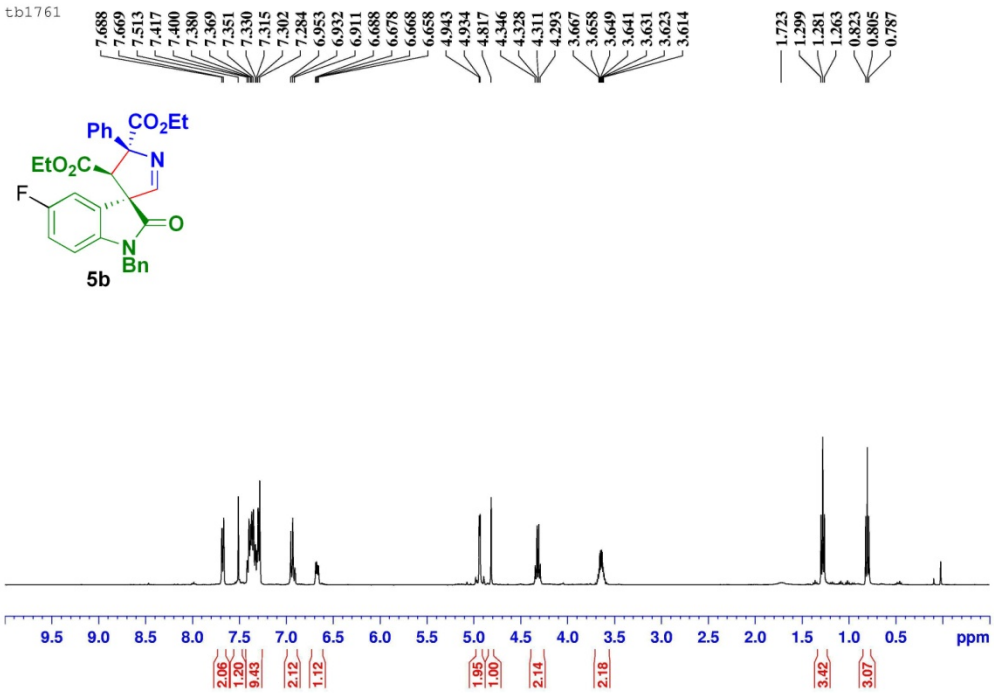


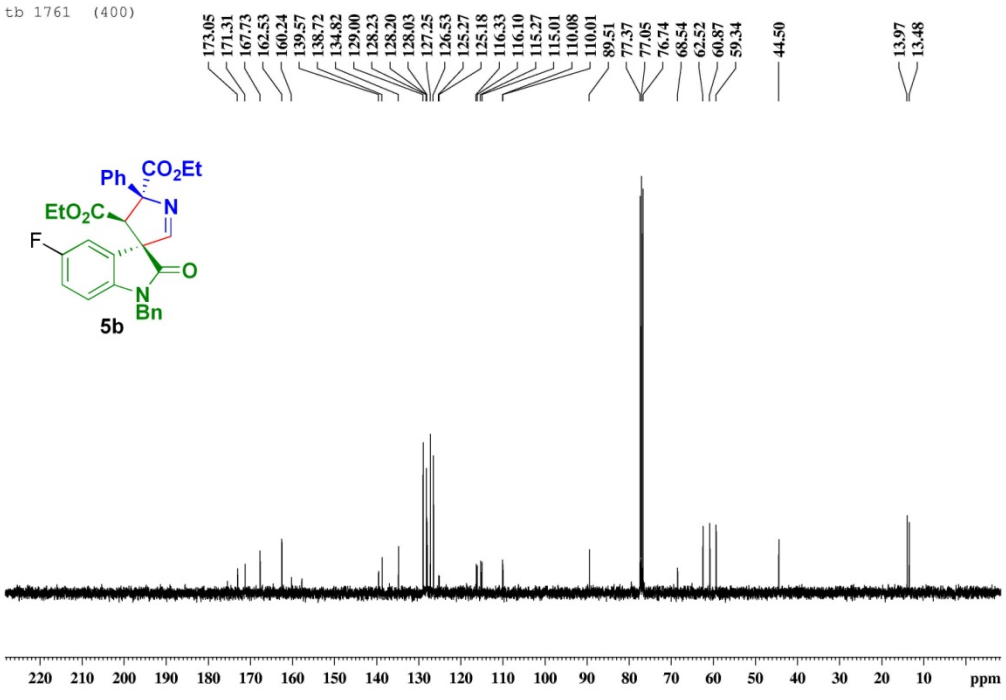


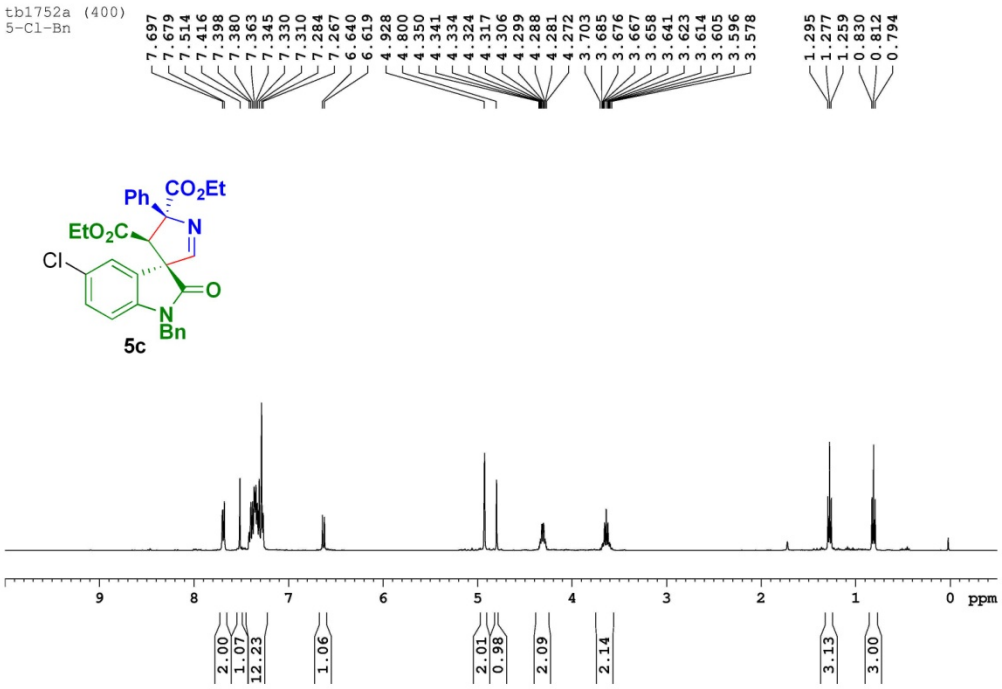


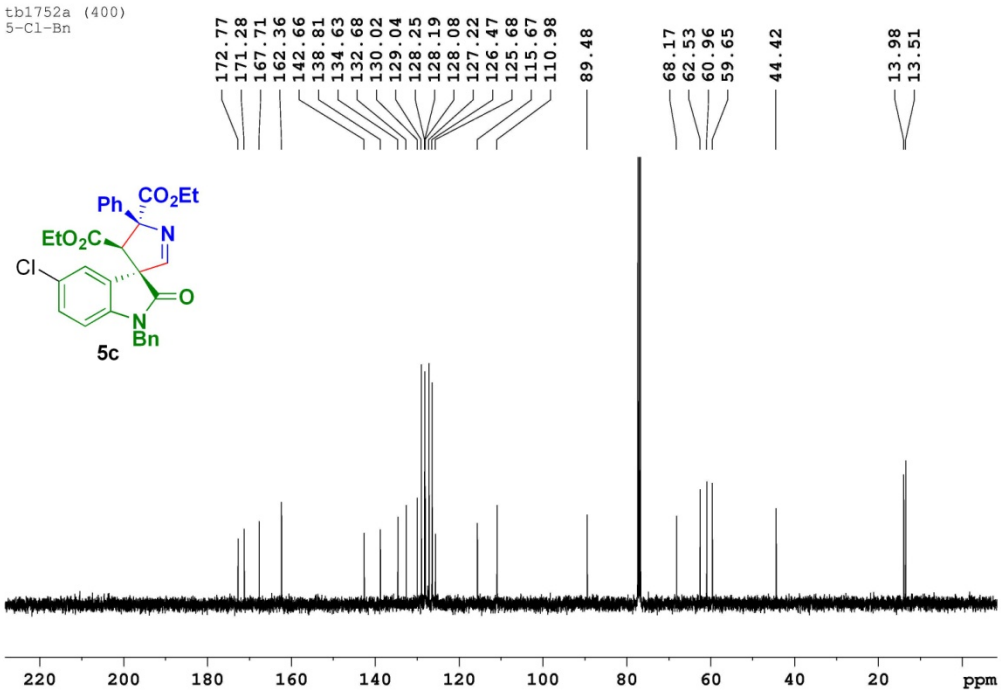


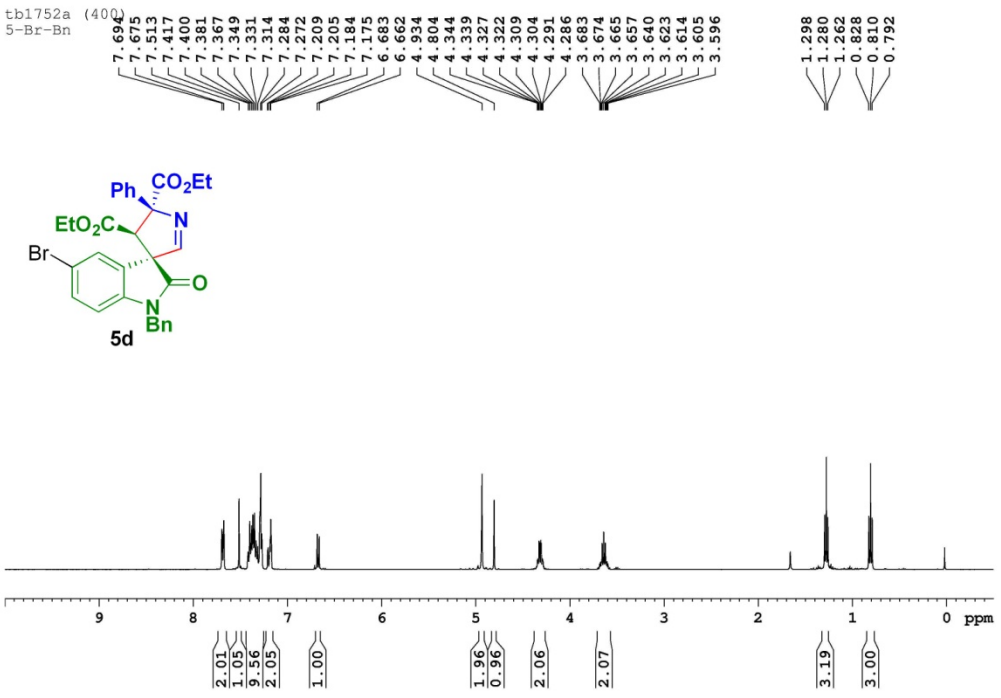


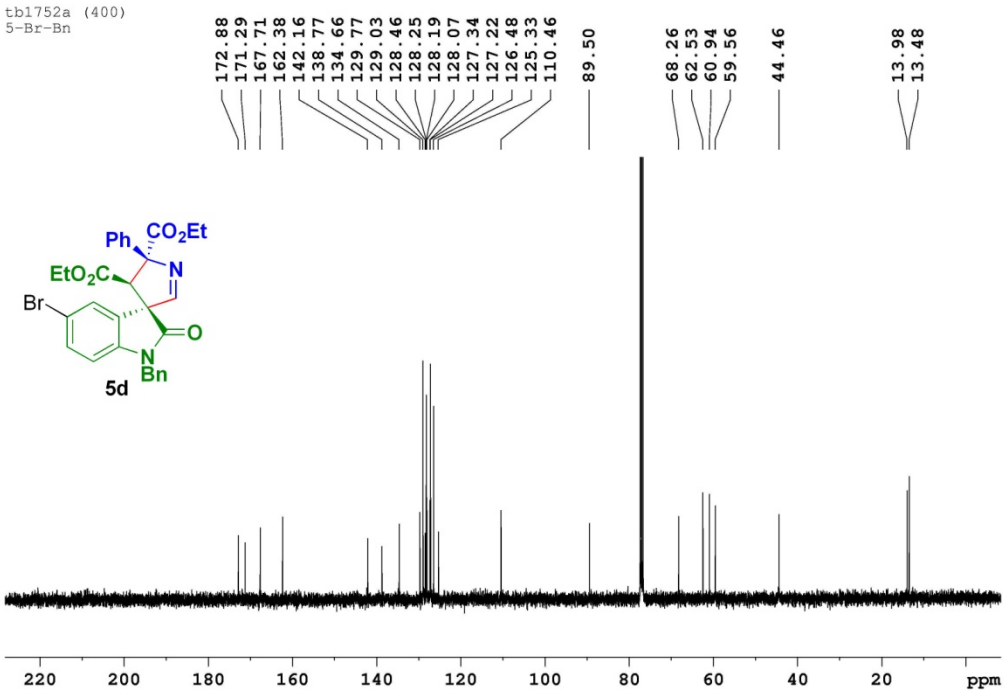


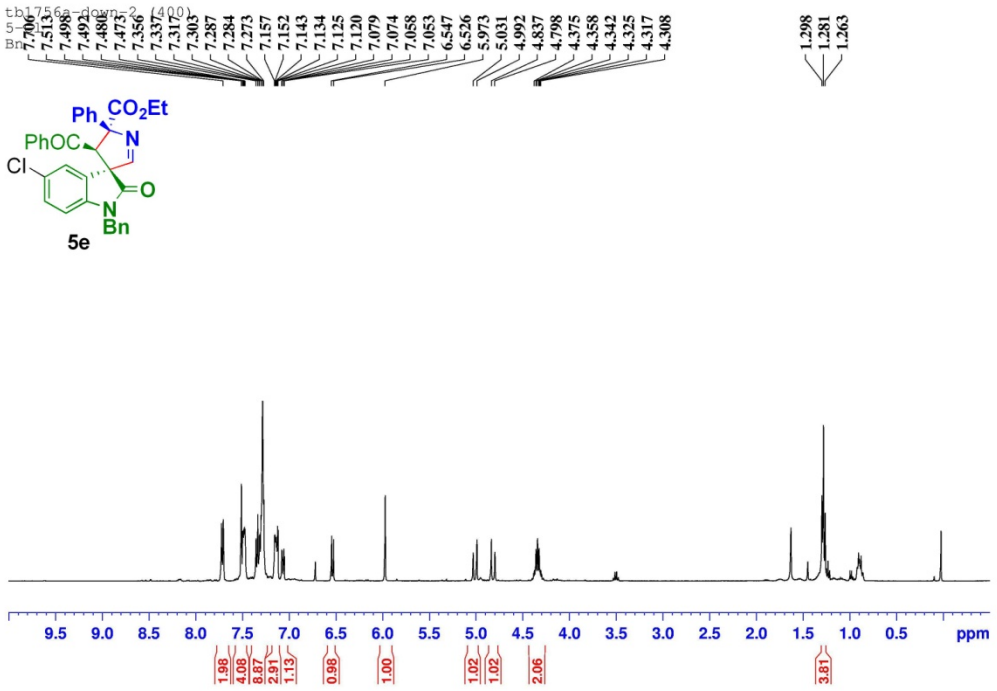


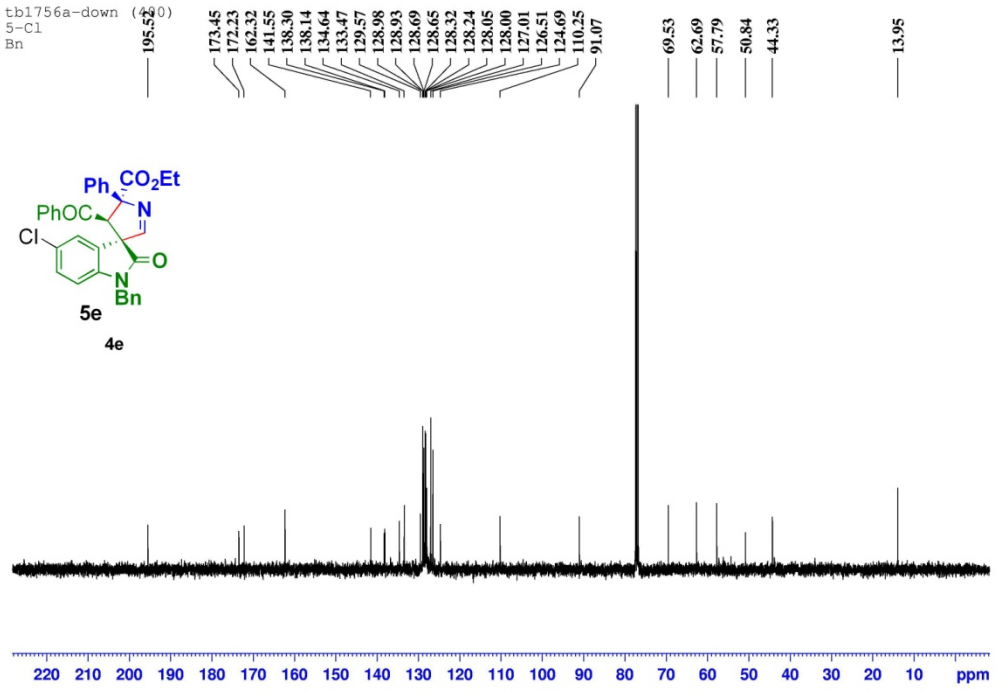




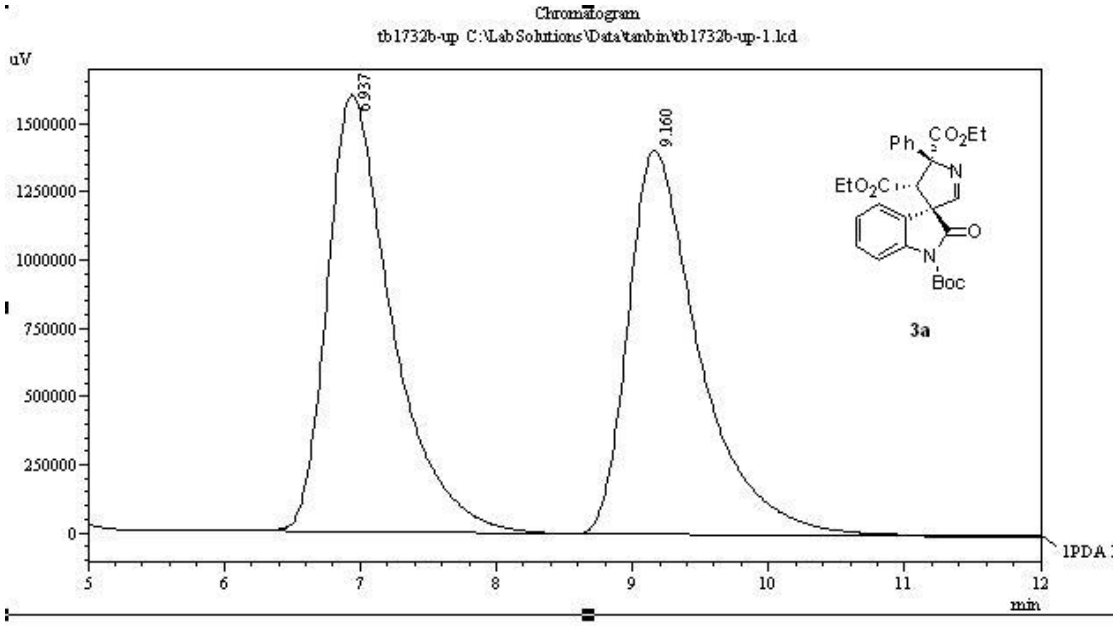








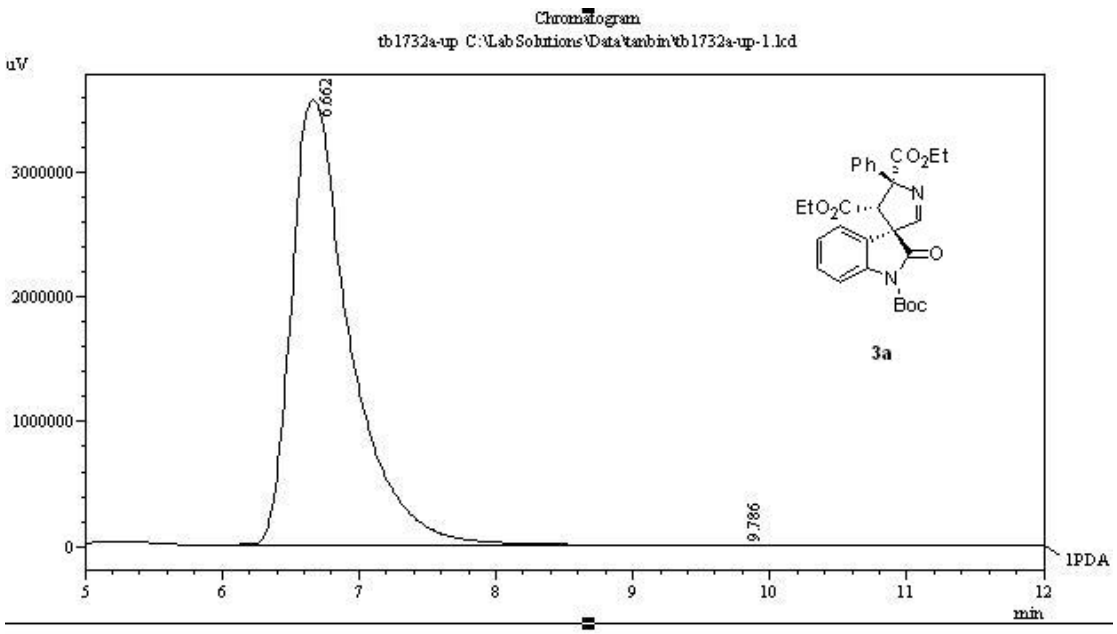
HPLC Spectra



PeakTable

PDA Ch1 220nm 4nm

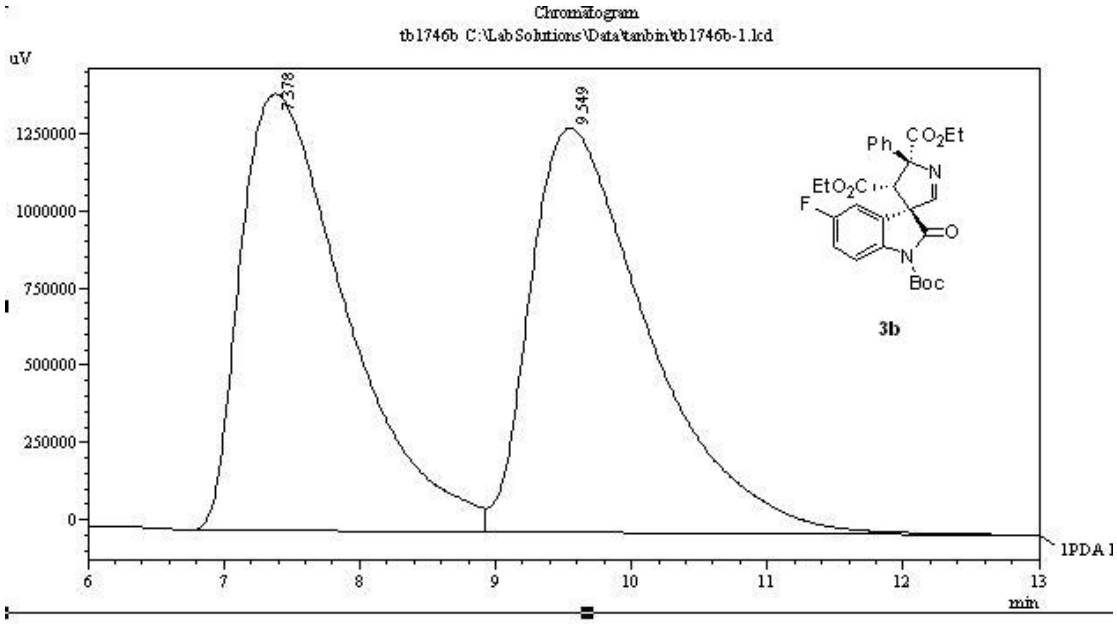
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.937	53187219	1600677	50.772	53.229
2	9.160	51570222	1406500	49.228	46.771
Total		104757441	3007178	100.000	100.000



PeakTable

PDA Ch1 220nm 4nm

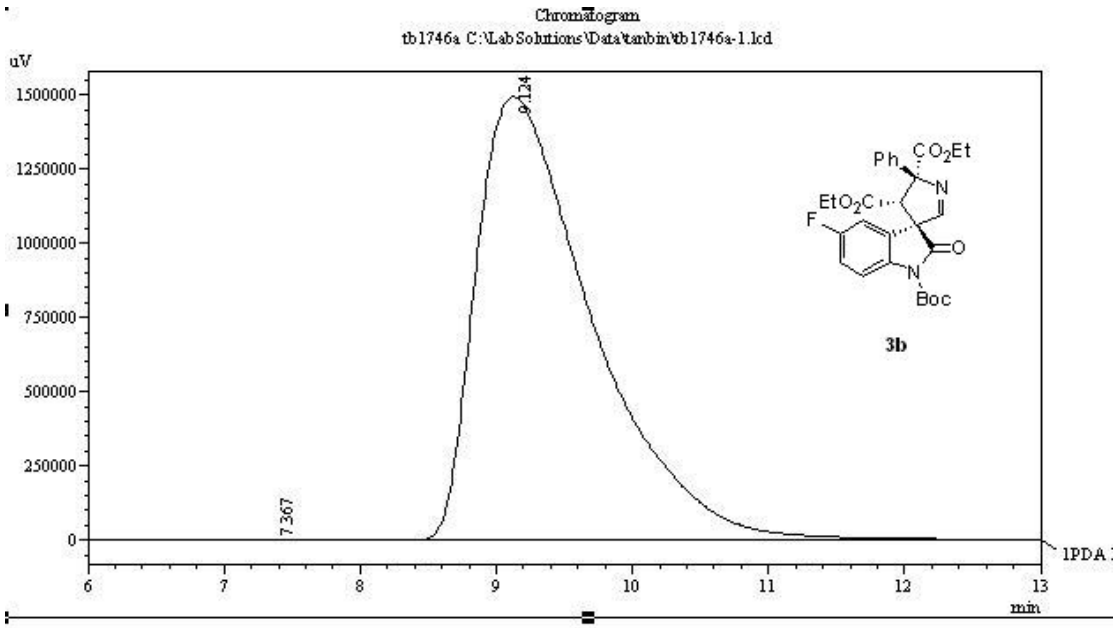
Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.662	106631961	3574722	99.983	99.973
2	9.786	18259	967	0.017	0.027
Total		106650220	3575689	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

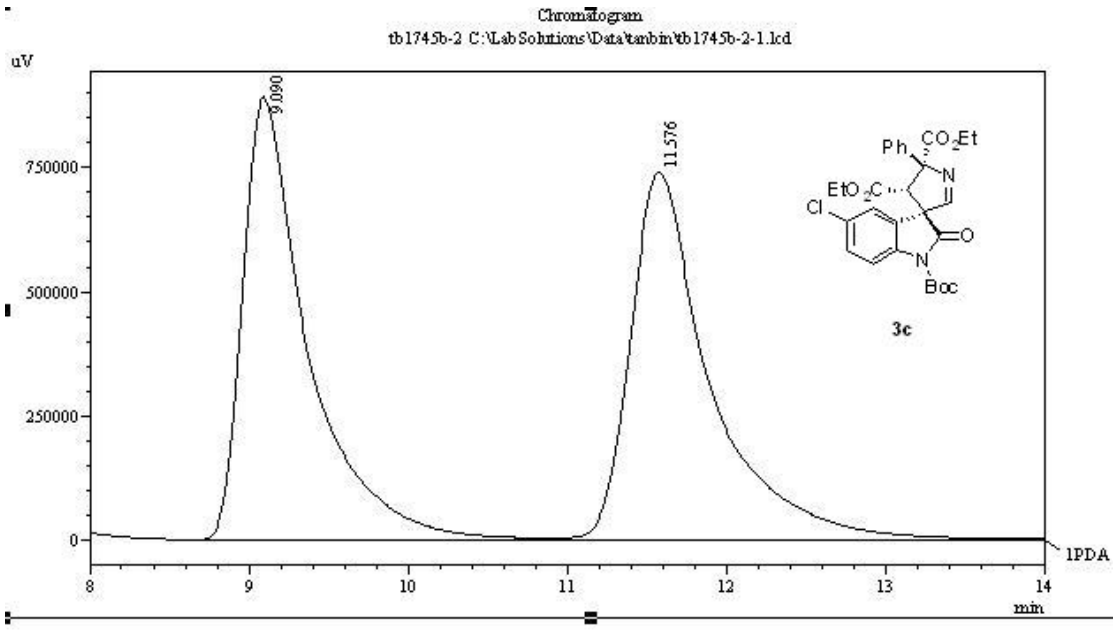
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.378	76579825	1410848	49.111	51.923
2	9.549	79353302	1306342	50.889	48.077
Total		155933127	2717190	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

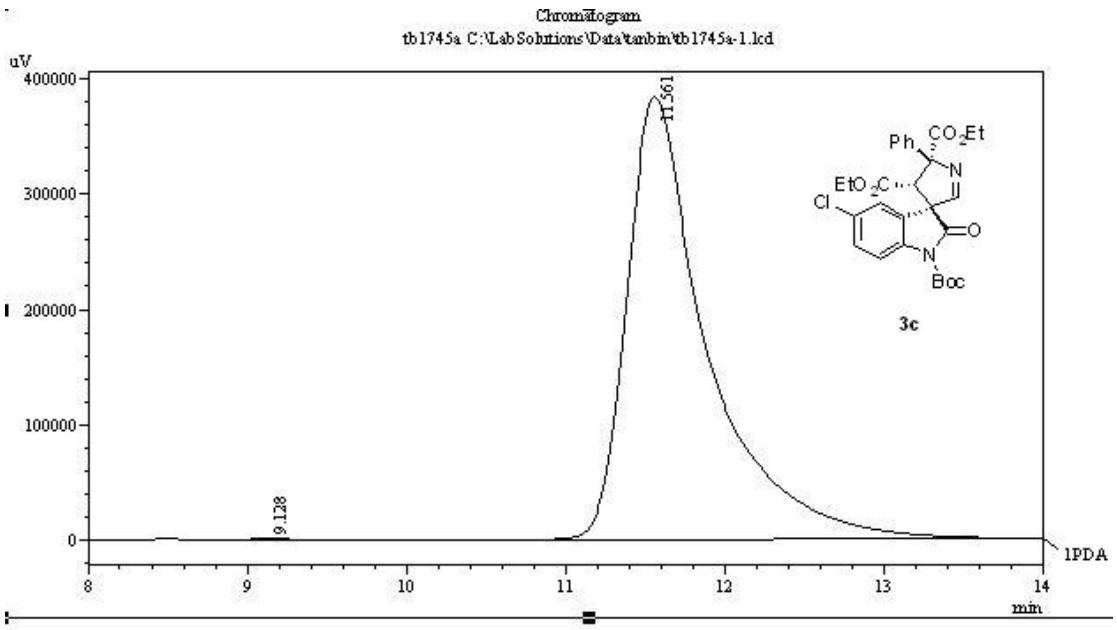
Peak#	Ret. Time	Area	Height	Area %	Height %
1	7.367	6147	32	0.007	0.002
2	9.124	89301982	1493791	99.993	99.998
Total		89308129	1493824	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

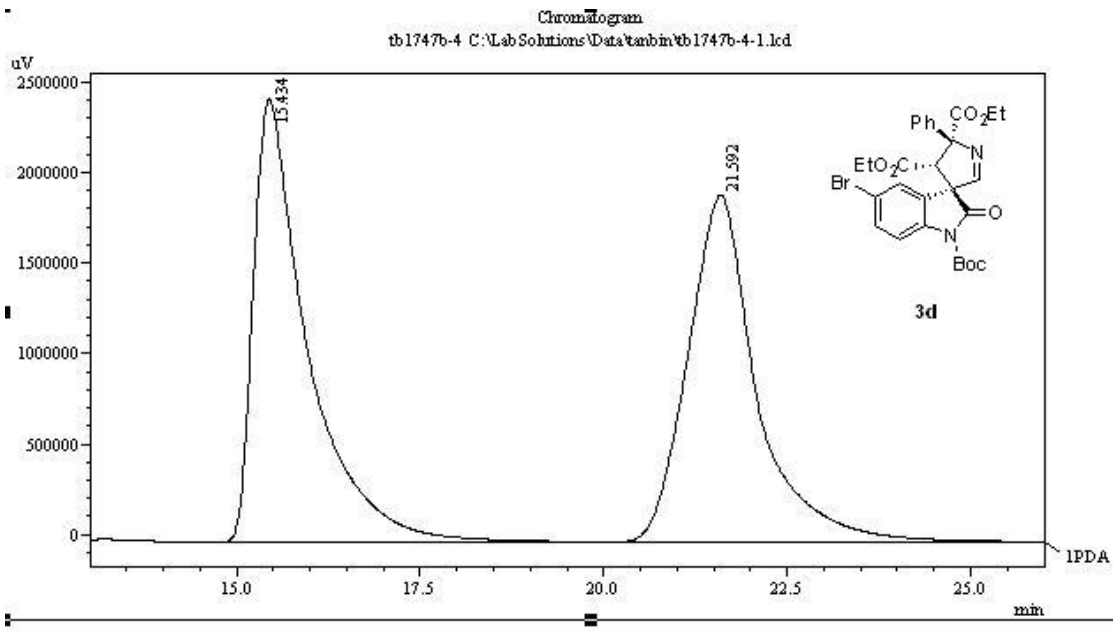
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.090	25377908	890197	49.394	54.651
2	11.576	26000747	738665	50.606	45.349
Total		51378655	1628862	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

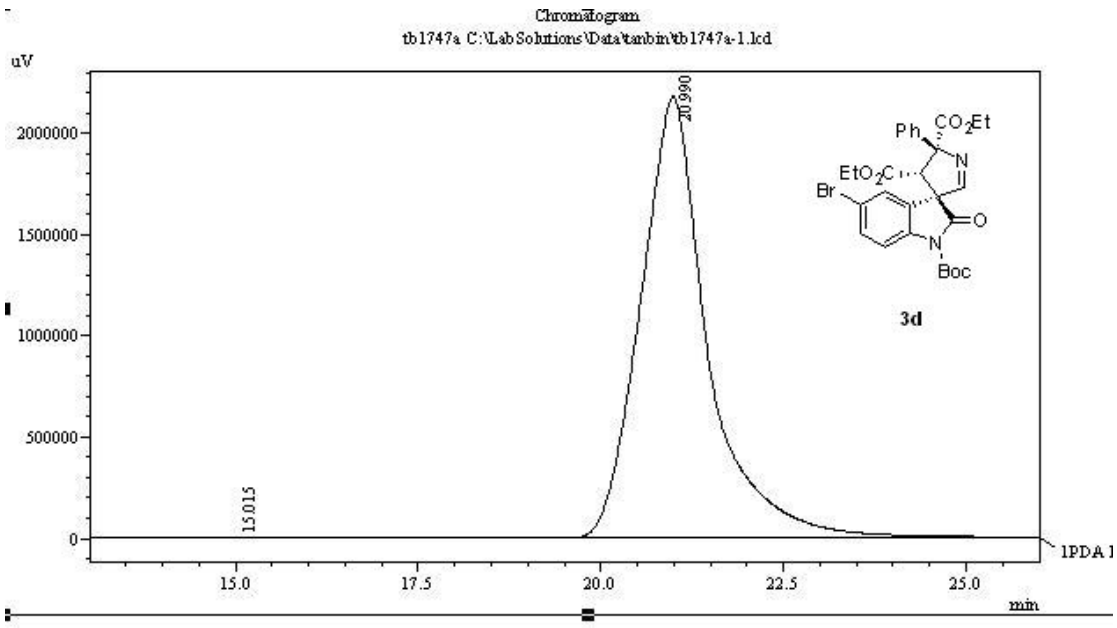
Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.128	12017	776	0.089	0.202
2	11.561	13455663	382868	99.911	99.798
Total		13467680	383644	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

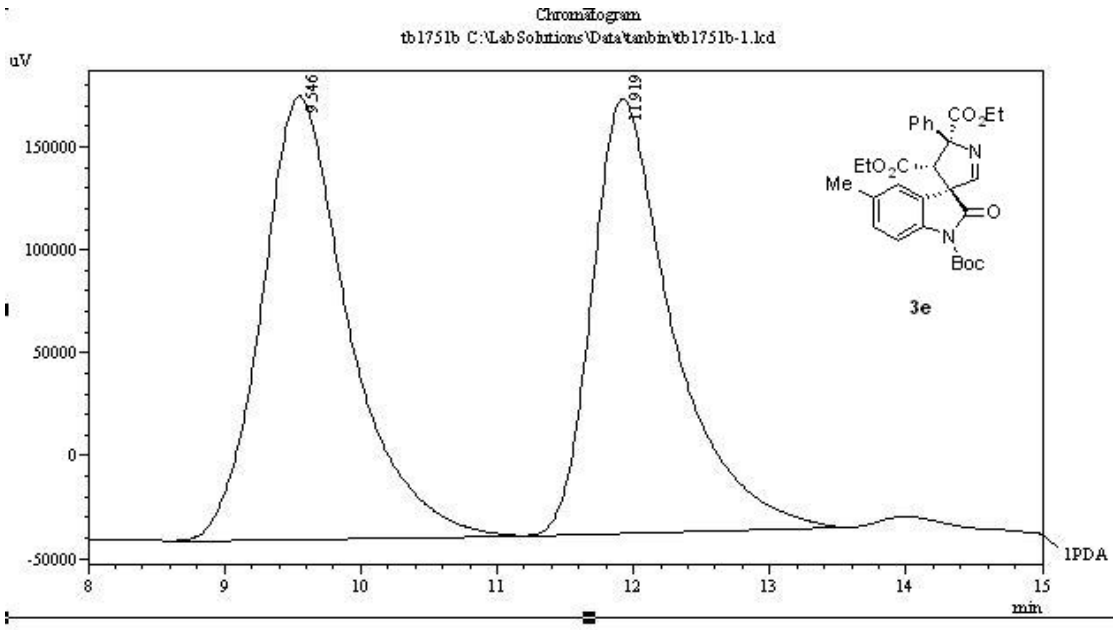
Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.434	126130906	2449870	49.963	56.082
2	21.592	126317980	1918517	50.037	43.918
Total		252448886	4368387	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	15.015	5498	200	0.004	0.009
2	20.990	140044770	2177912	99.996	99.991
Total		140050268	2178113	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	9.546	9520229	215165	52.305	50.473
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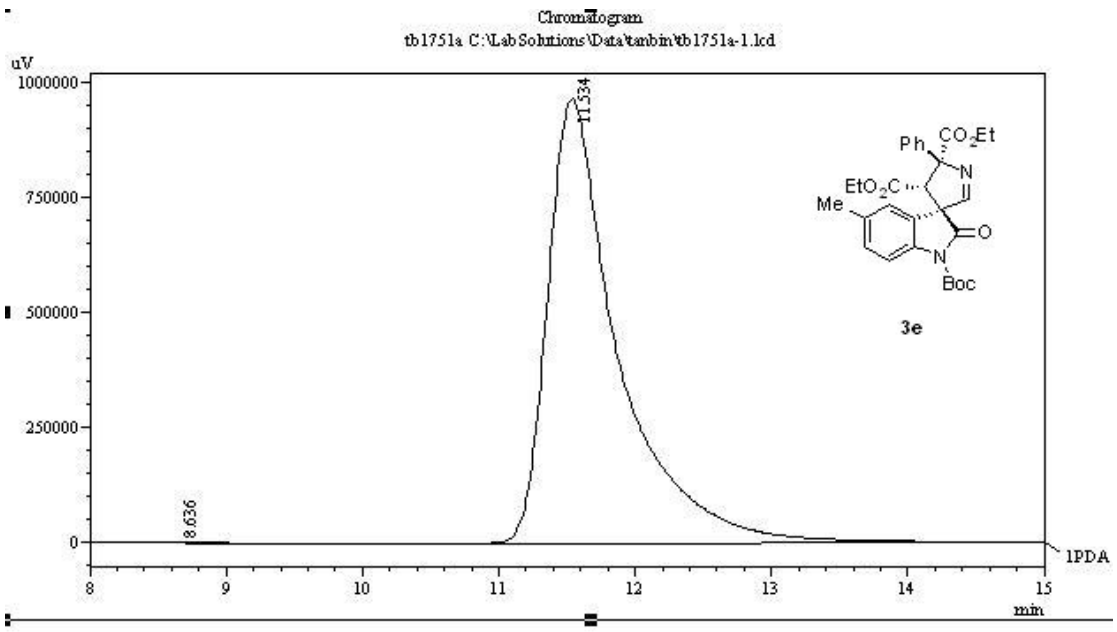
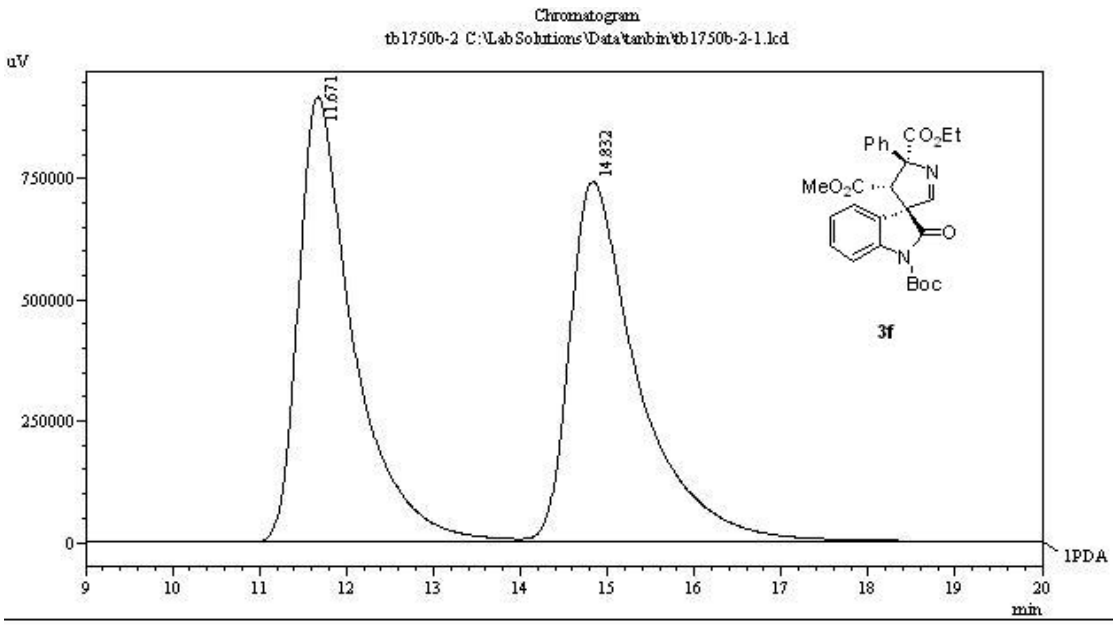
Total		18201462	426298	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

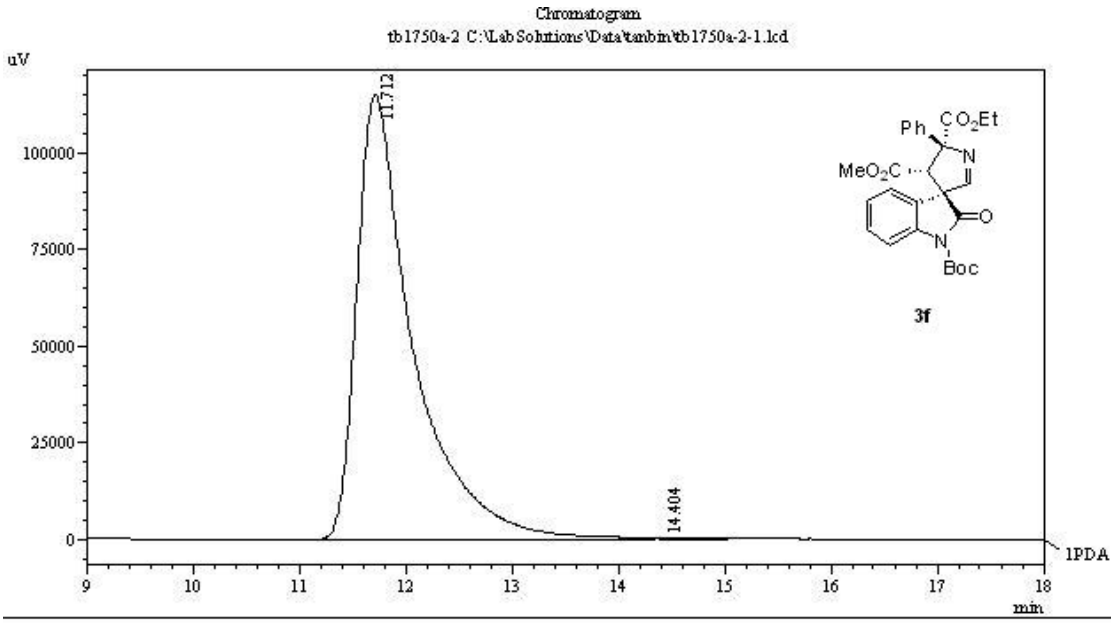
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.636	8896	433	0.025	0.045
2	11.534	34951958	965106	99.975	99.955
Total		34960854	965538	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

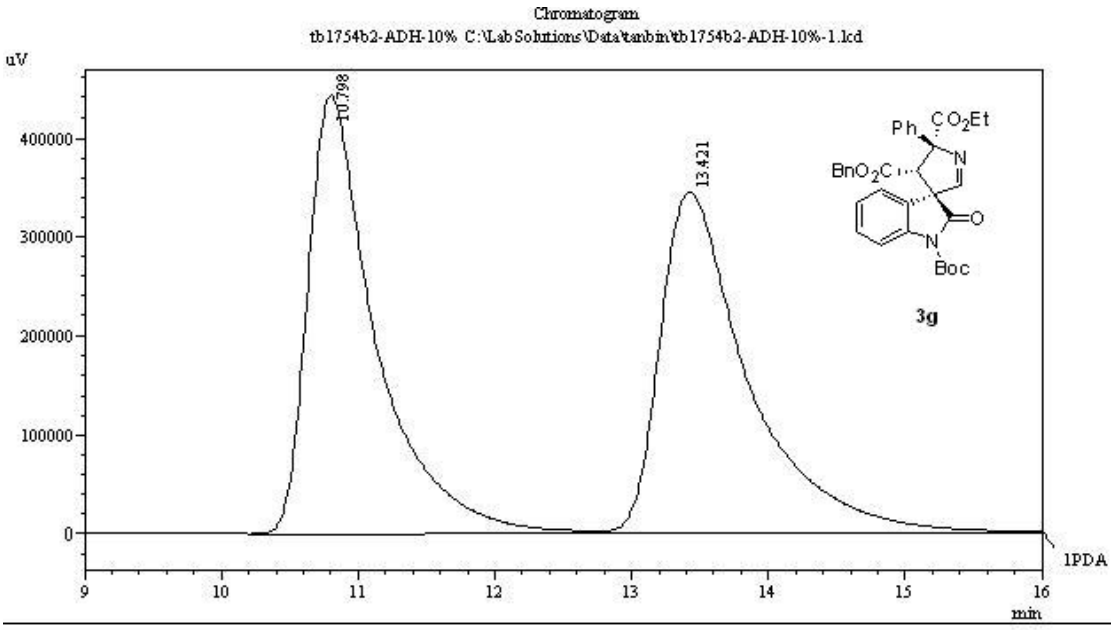
Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.671	40406276	917242	49.477	55.239
2	14.832	41260604	743261	50.523	44.761
Total		81666880	1660502	100.000	100.000



PeakTable

PDA Ch3 254nm 4nm

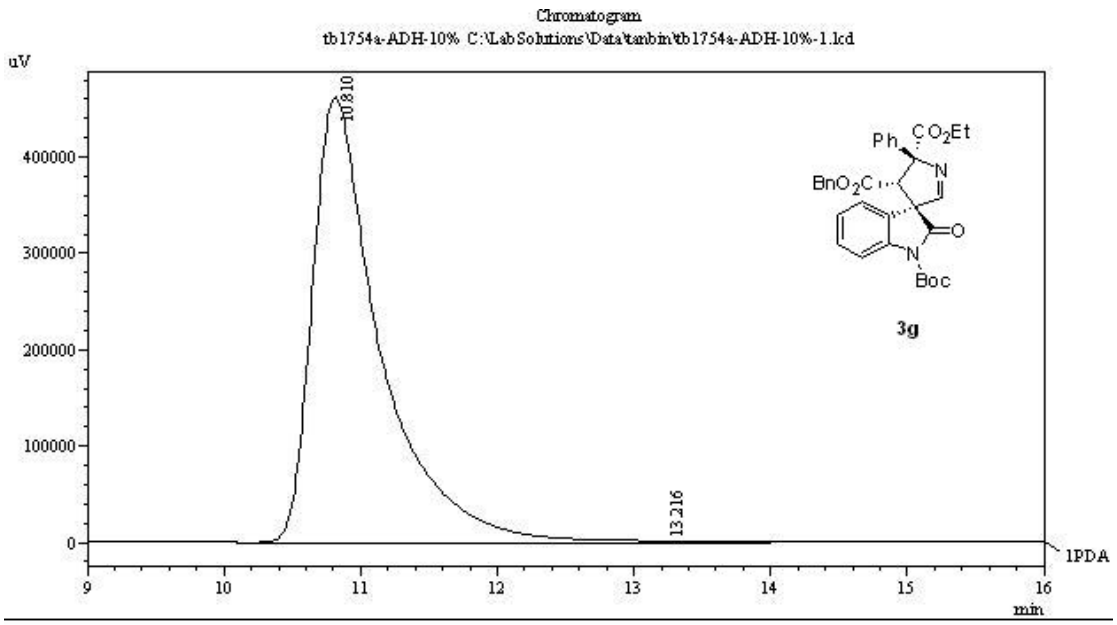
Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.712	4252560	114845	99.682	99.634
2	14.404	13579	422	0.318	0.366
Total		4266139	115267	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

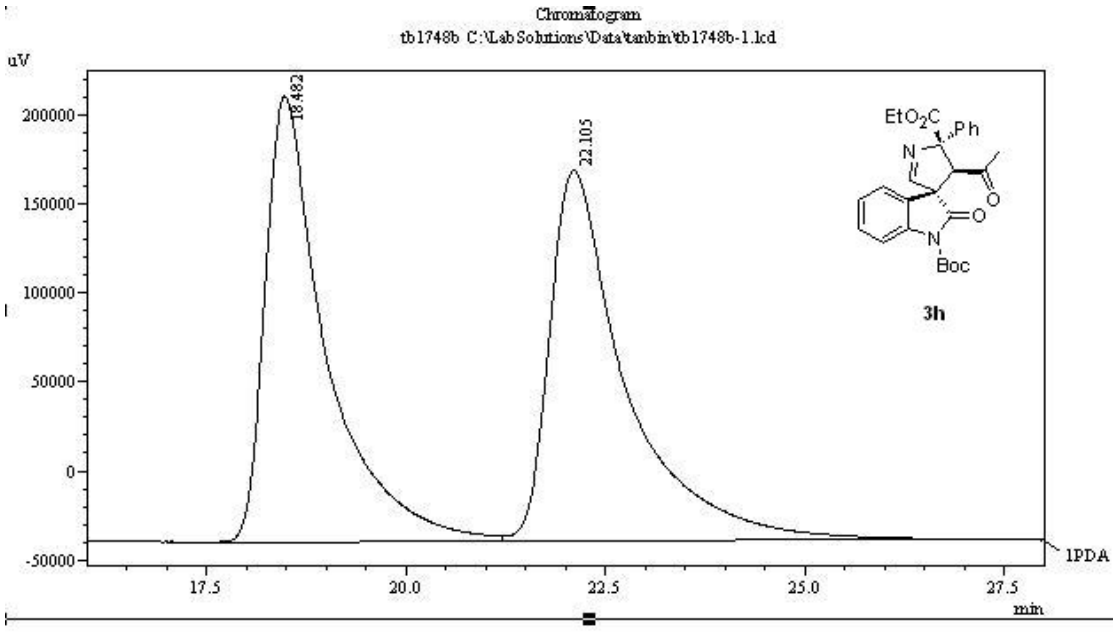
Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.798	15395472	442350	49.626	56.162
2	13.421	15627600	345278	50.374	43.838
Total		31023071	787628	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

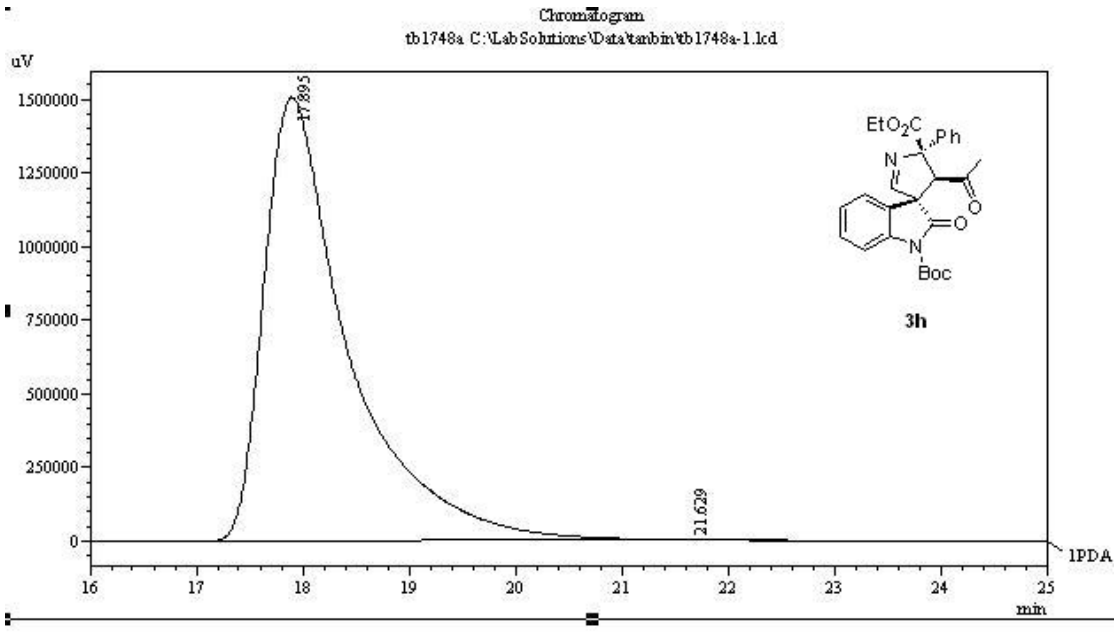
Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.810	15974890	461914	99.651	99.662
2	13.216	56005	1568	0.349	0.338
Total		16030895	463482	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

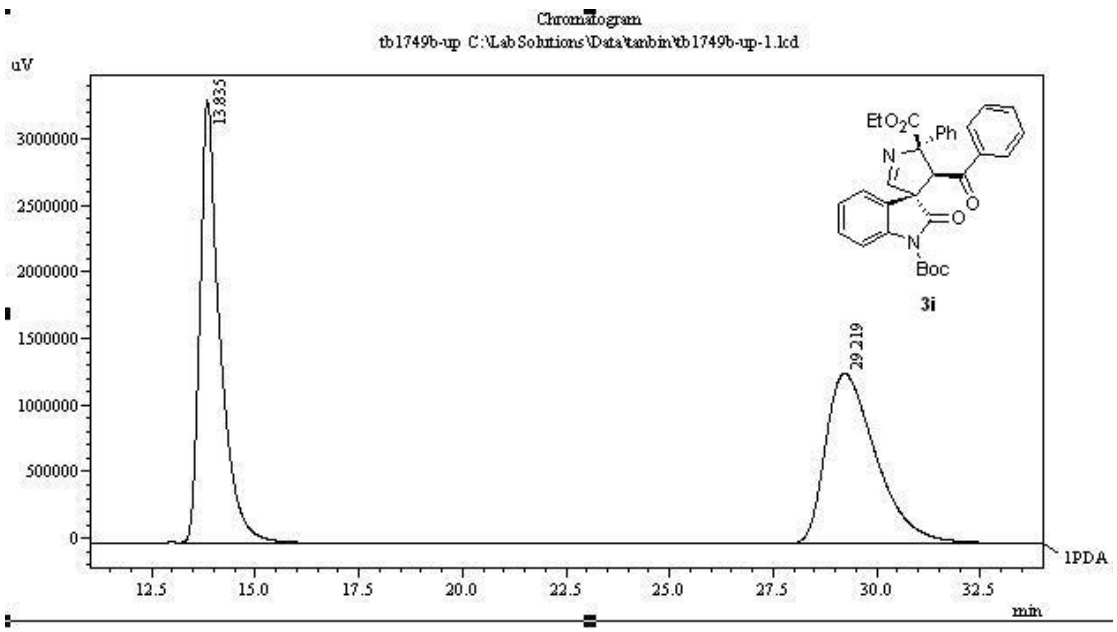
Peak#	Ret. Time	Area	Height	Area %	Height %
1	18.482	13306874	250257	49.001	54.594
2	22.105	13849214	208140	50.999	45.406
Total		27156088	458397	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

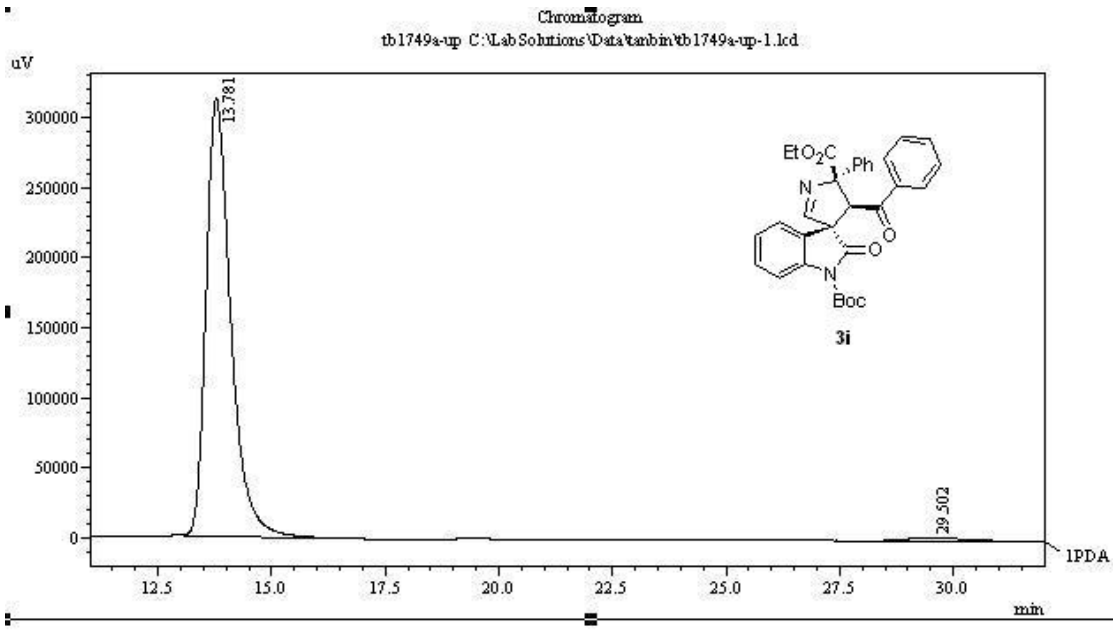
Peak#	Ret. Time	Area	Height	Area %	Height %
1	17.895	83063582	1507071	99.864	99.830
2	21.629	113327	2561	0.136	0.170
Total		83176909	1509632	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

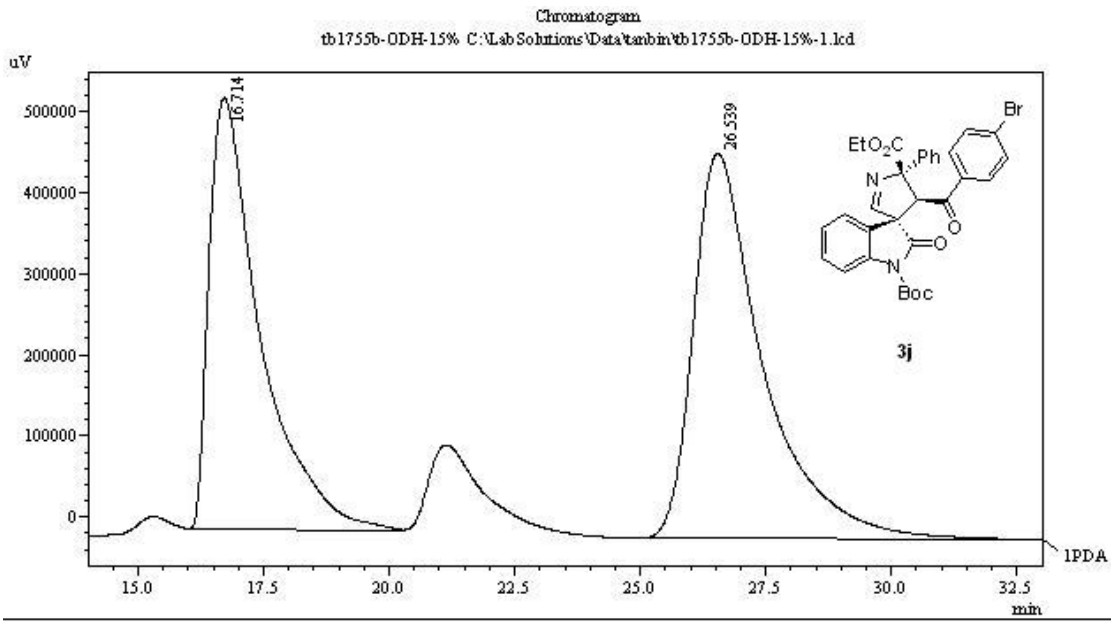
Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.835	112266621	3322978	50.540	72.197
2	29.219	109866801	1279688	49.460	27.803
Total		222133423	4602665	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

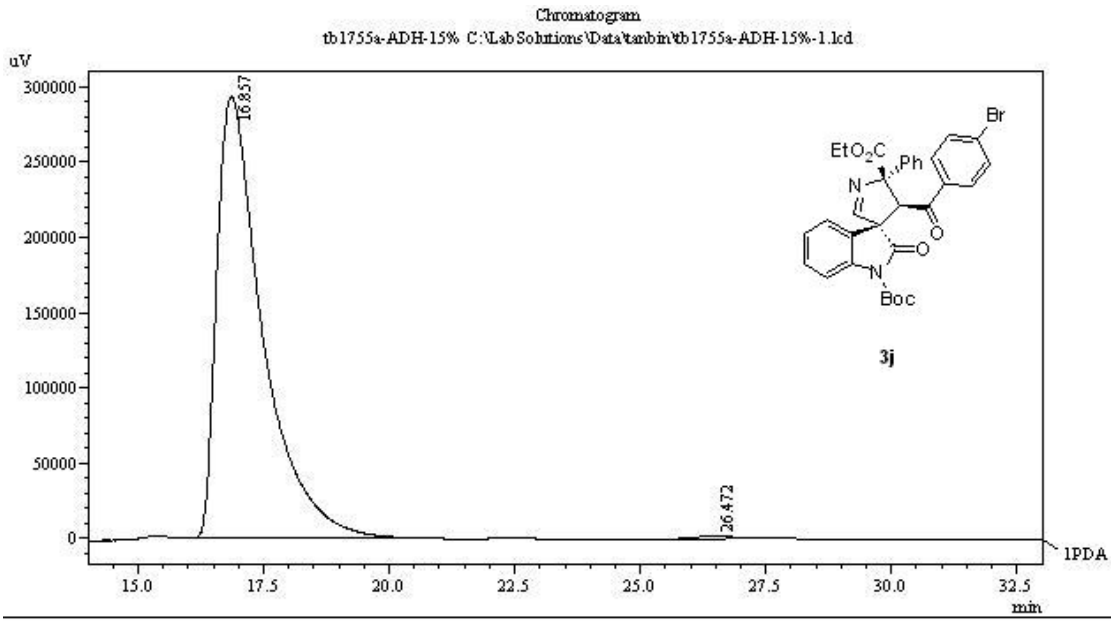
Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.781	11921095	313115	98.389	99.196
2	29.502	195155	2539	1.611	0.804
Total		12116250	315654	100.000	100.000



PeakTable

PDA Ch1 220nm 4nm

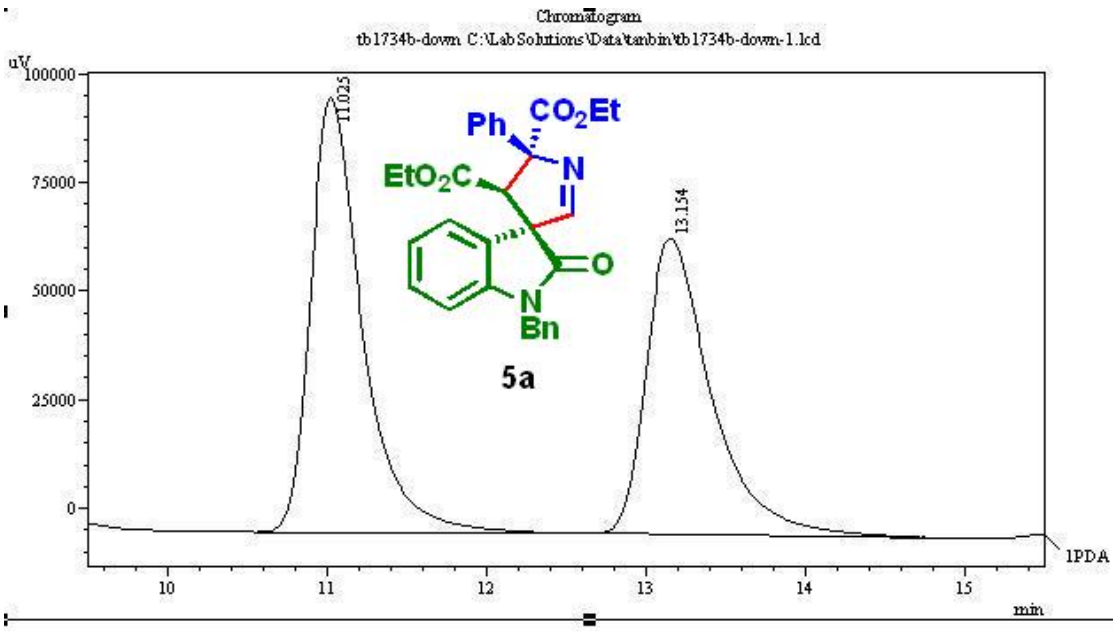
Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.714	37521948	531325	45.003	52.850
2	26.539	45854081	474027	54.997	47.150
Total		83376030	1005353	100.000	100.000



PeakTable

PDA Ch1 220nm 4nm

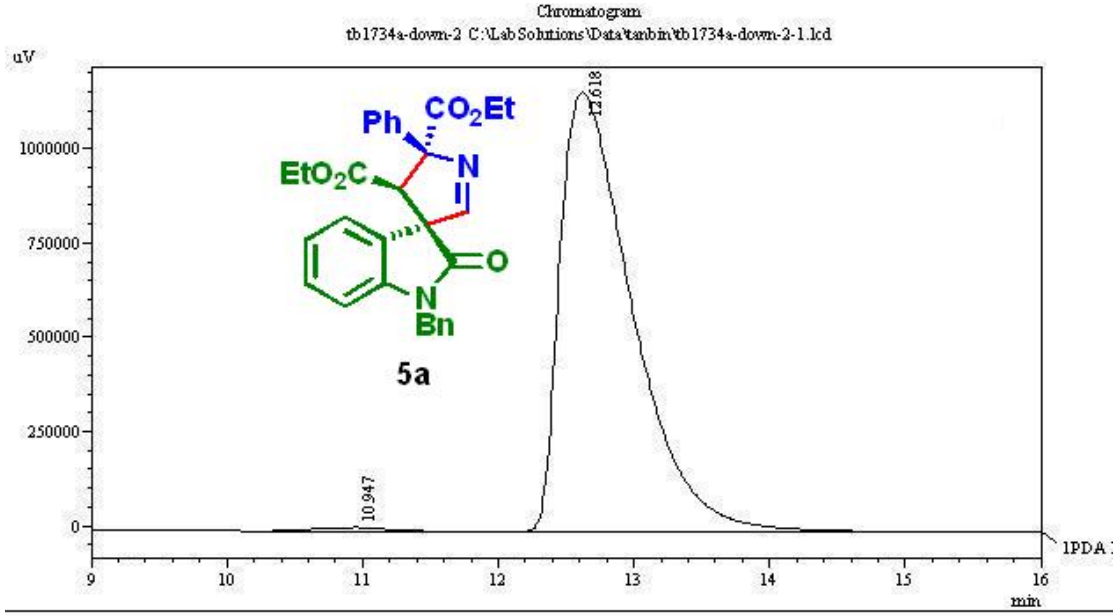
Peak#	Ret. Time	Area	Height	Area %	Height %
1	16.857	18685289	293519	99.330	99.398
2	26.472	126024	1779	0.670	0.602
Total		18811313	295298	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

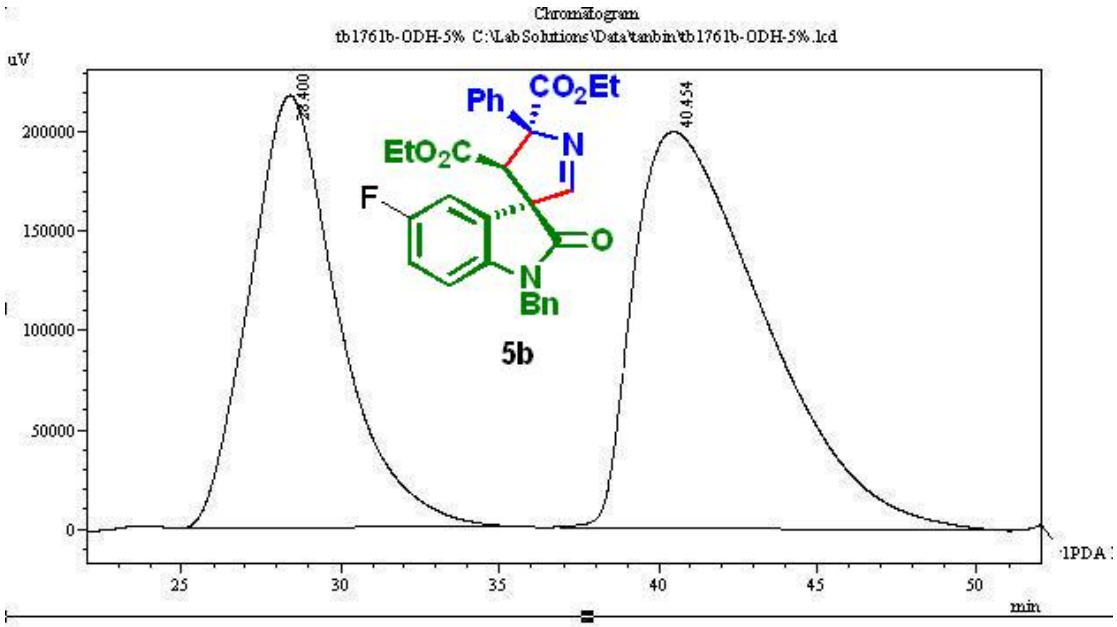
Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.025	2269298	99990	53.786	59.434
2	13.154	1949821	68248	46.214	40.566
Total		4219119	168238	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

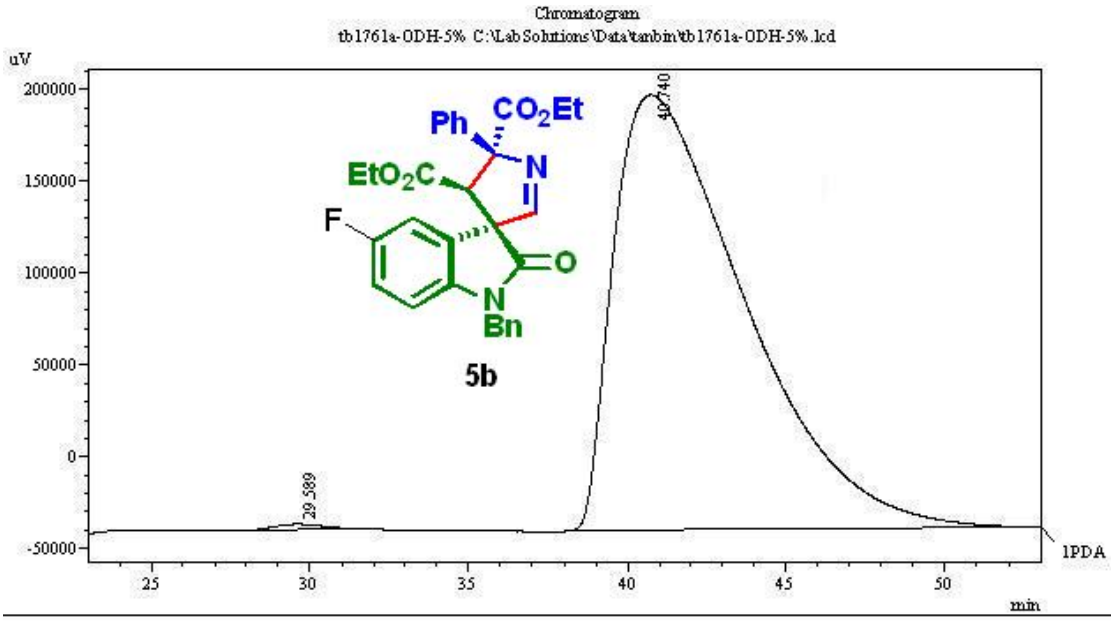
Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.947	359310	10080	0.827	0.859
2	12.618	43081784	1163090	99.173	99.141
Total		43441094	1173170	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

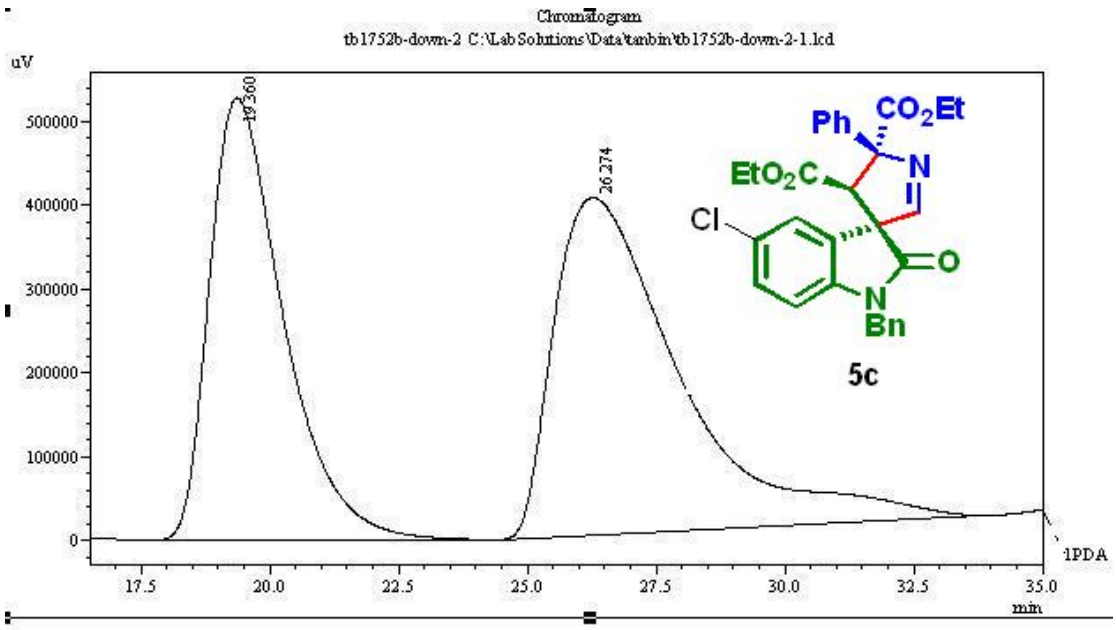
Peak#	Ret. Time	Area	Height	Area %	Height %
1	28.400	42382820	218002	42.570	52.204
2	40.454	57178135	199596	57.430	47.796
Total		99560955	417598	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

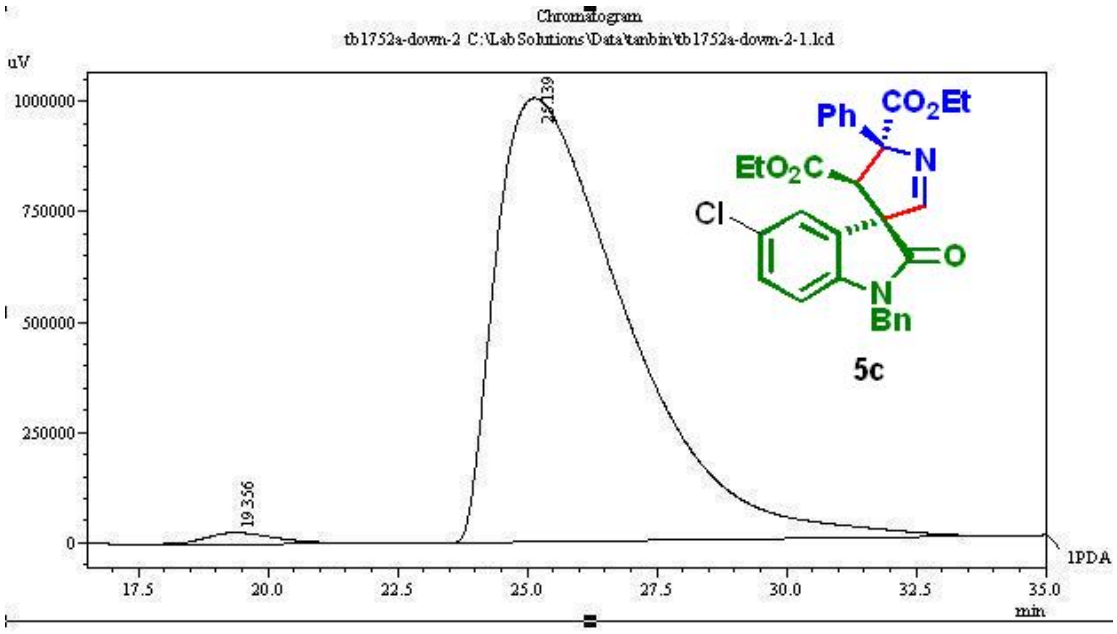
Peak#	Ret. Time	Area	Height	Area %	Height %
1	29.589	368770	3273	0.535	1.360
2	40.740	68514072	237361	99.465	98.640
Total		68882843	240634	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

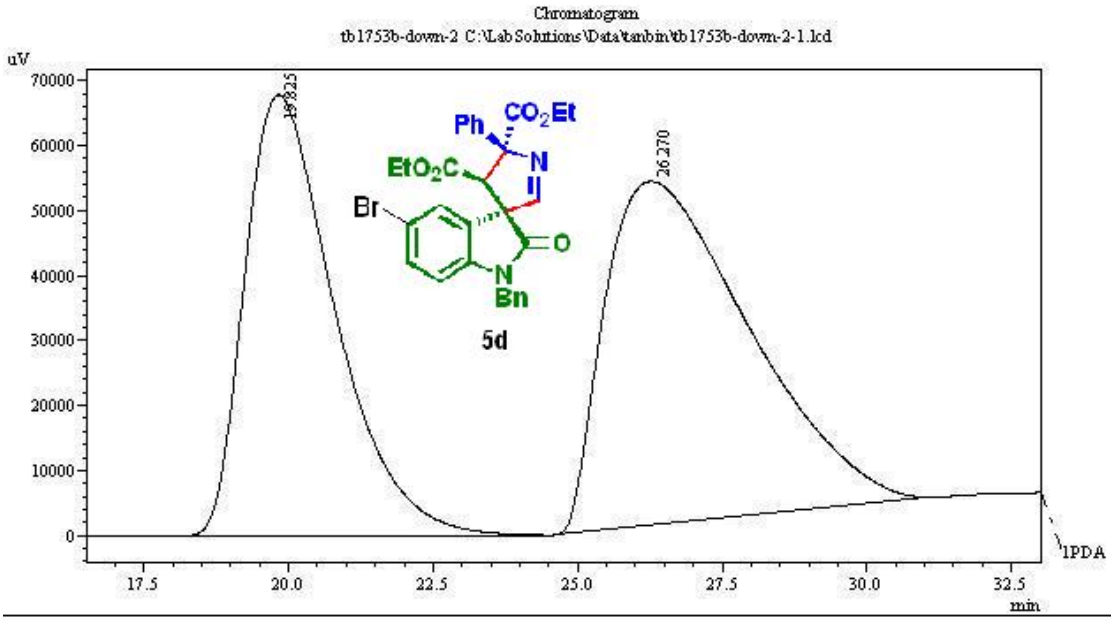
Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.360	52705615	527132	43.419	56.734
2	26.274	68682712	401999	56.581	43.266
Total		121388327	929130	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

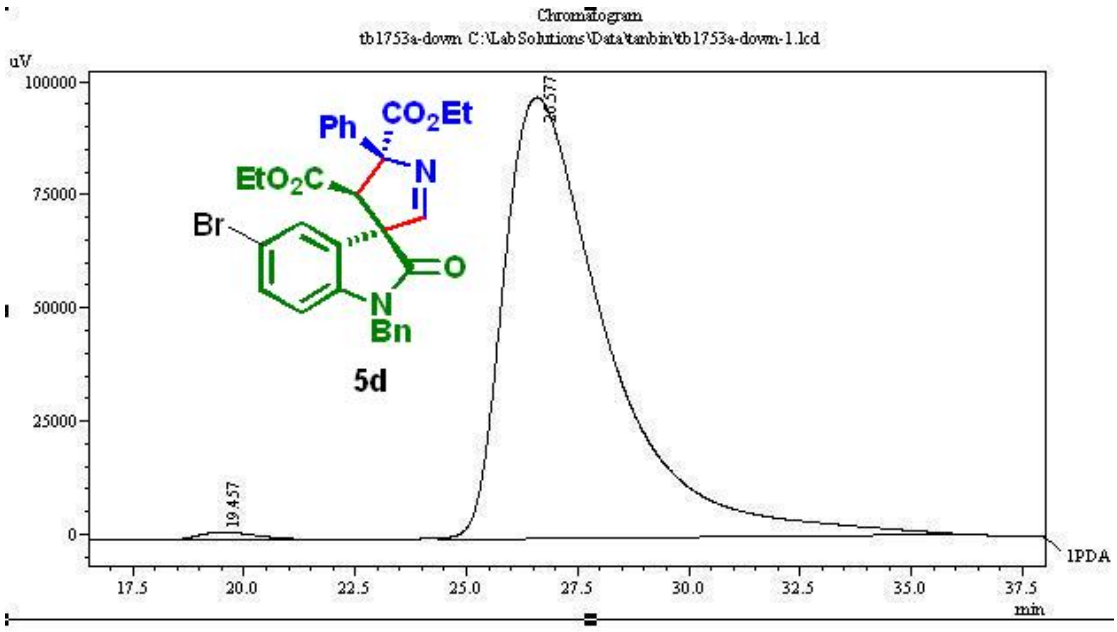
Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.356	2451259	25488	1.363	2.471
2	25.139	177420662	1005919	98.637	97.529
Total		179871920	1031407	100.000	100.000



PeakTable

PDA Ch5 280nm 4nm

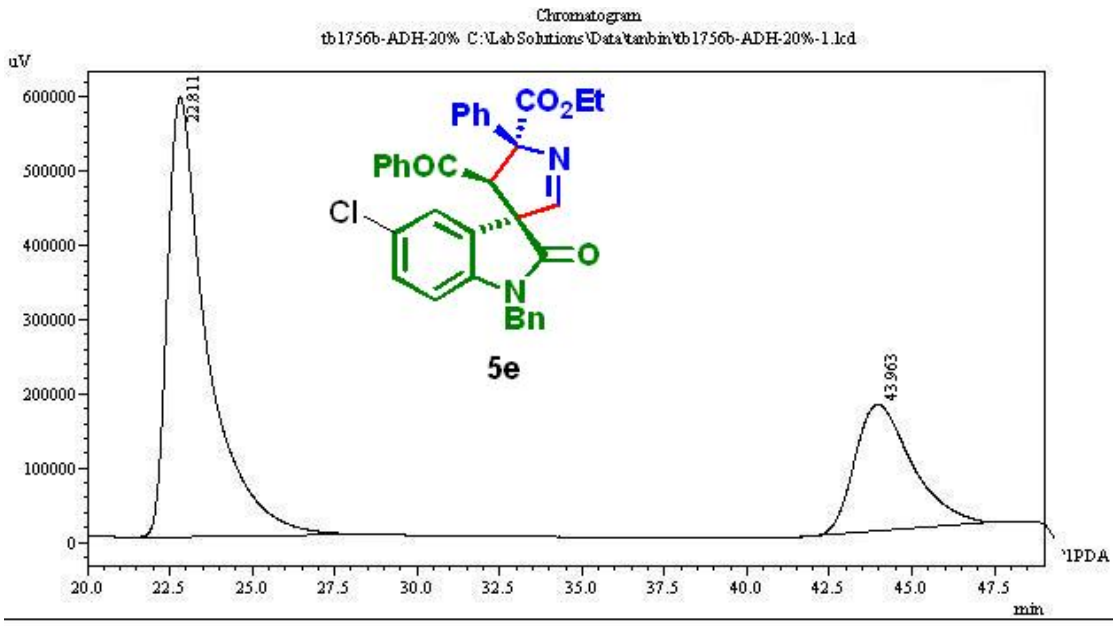
Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.825	7356739	67896	44.480	56.198
2	26.270	9182585	52920	55.520	43.802
Total		16539324	120815	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

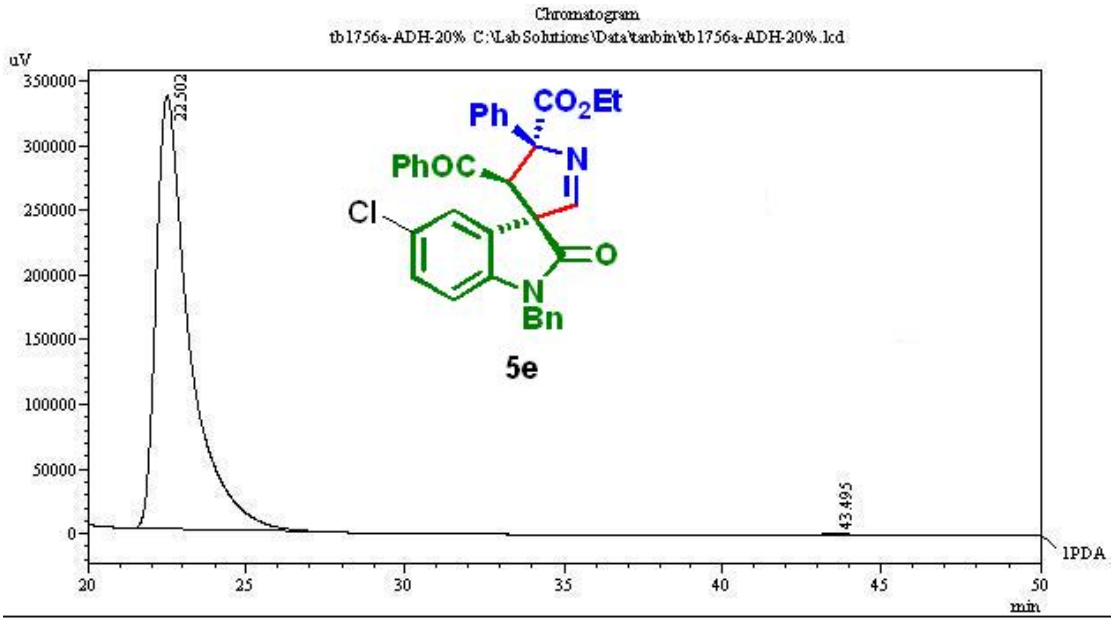
Peak#	Ret. Time	Area	Height	Area %	Height %
1	19.457	156956	1714	0.989	1.730
2	26.577	15714567	97350	99.011	98.270
Total		15871523	99064	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.811	49710146	592364	70.522	77.600
2	43.963	20779069	170994	29.478	22.400
Total		70489215	763359	100.000	100.000



PeakTable

PDA Ch1 210nm 4nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	22.502	24537581	334912	99.801	99.860
2	43.495	48810	468	0.199	0.140
Total		24586392	335380	100.000	100.000

