

Recyclable BINOL-Quinine-Squaramide as Highly Efficient Organocatalyst for α -Amination of 1, 3- Dicarboxyl Compounds and α -Cyanoacetates

Yongzhi Gao,^a Bin Liu,^a Hai-Bing Zhou,^{a,b} Wei Wang,^a Chune Dong*^{a,b}

^a *Key Laboratory of Combinatorial Biosynthesis and Drug Discovery (Wuhan University), Ministry of Education, State Key Laboratory of Virology, Wuhan University School of Pharmaceutical Sciences, Wuhan 430071, China.*

^b *Key Laboratory of Organofluorine Chemistry, Shanghai Institute of Organic Chemistry, Chinese Academy of Sciences, Shanghai, 200032, China.*

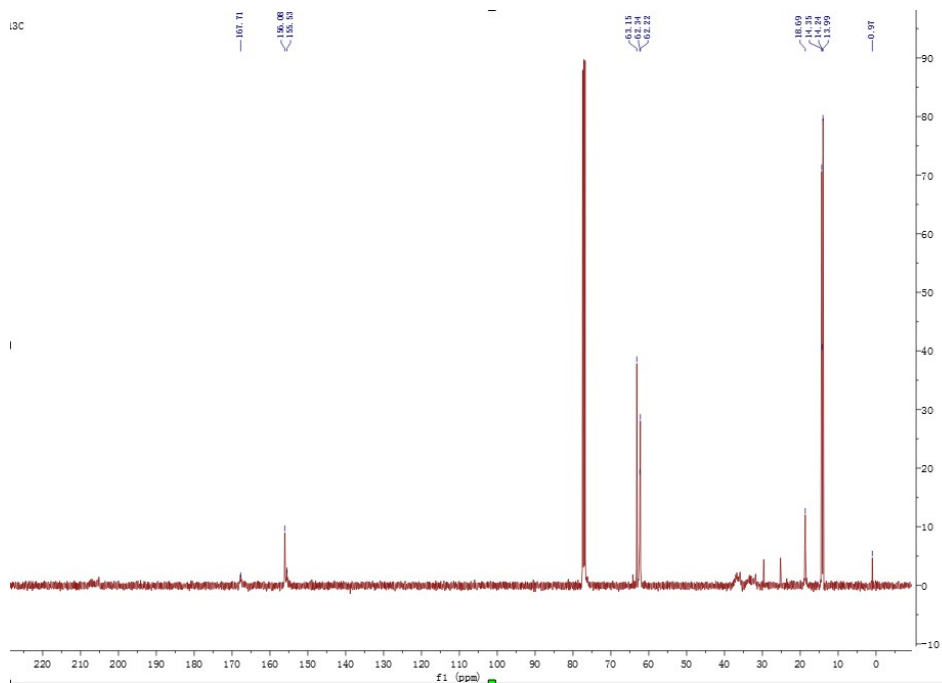
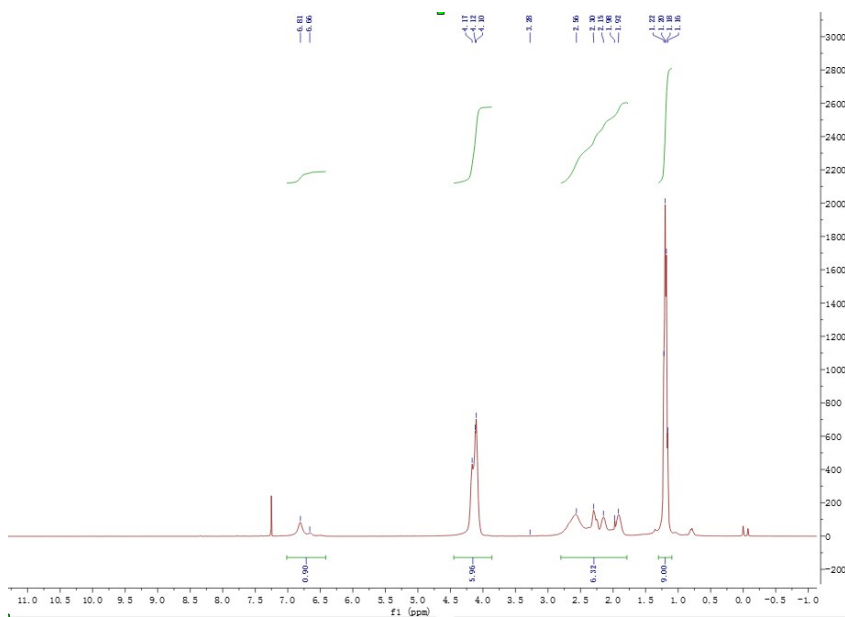
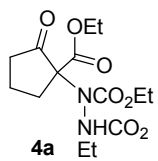
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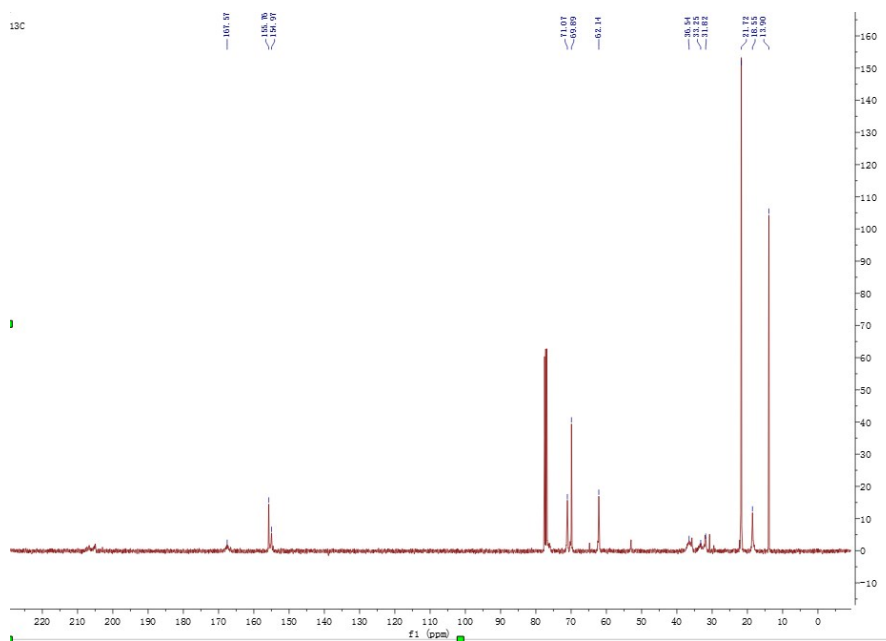
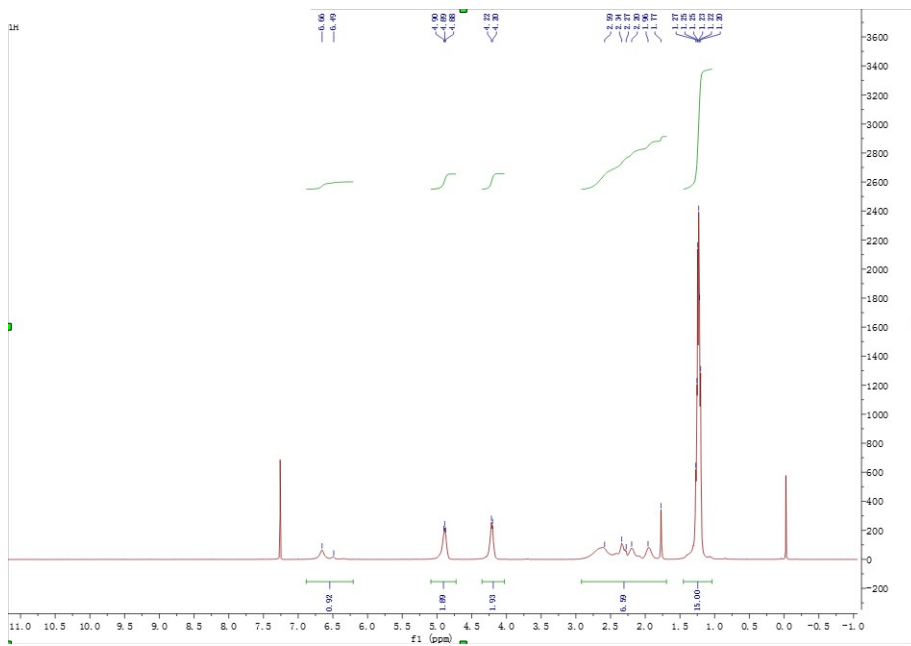
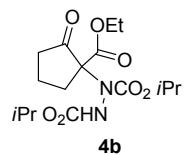
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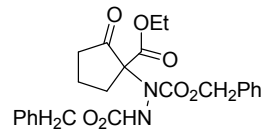
I NMR spectra of 4

II HPLC spectra of 4

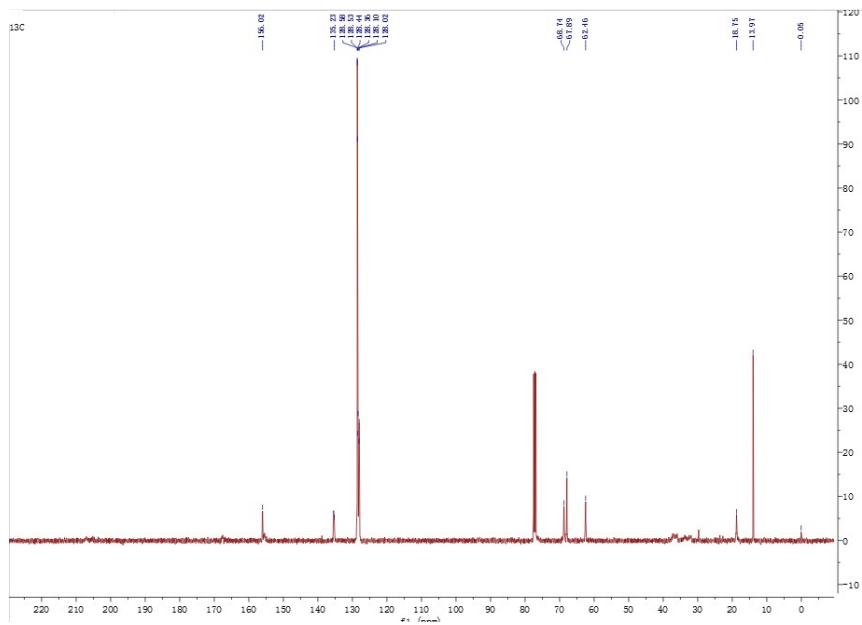
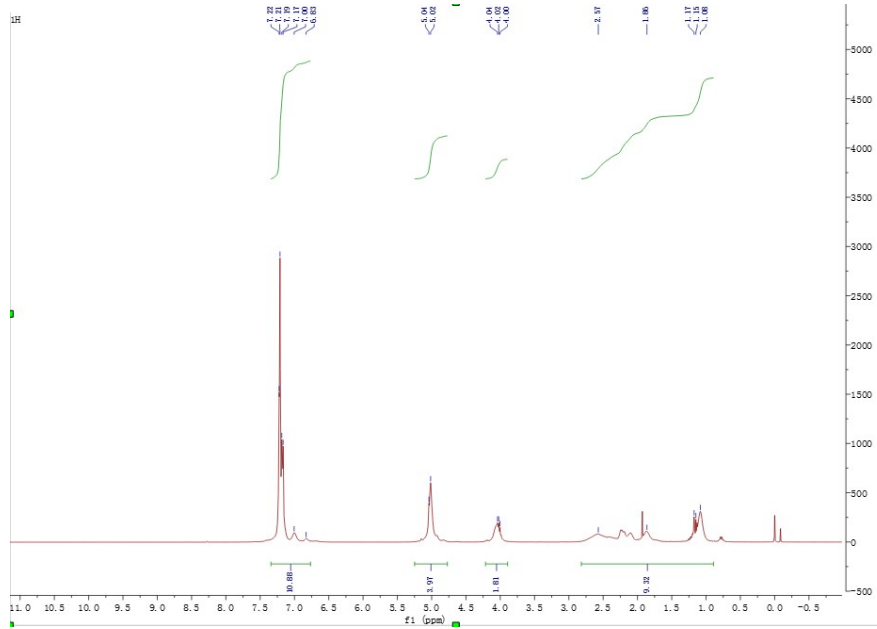
1H NMR spectra of 4

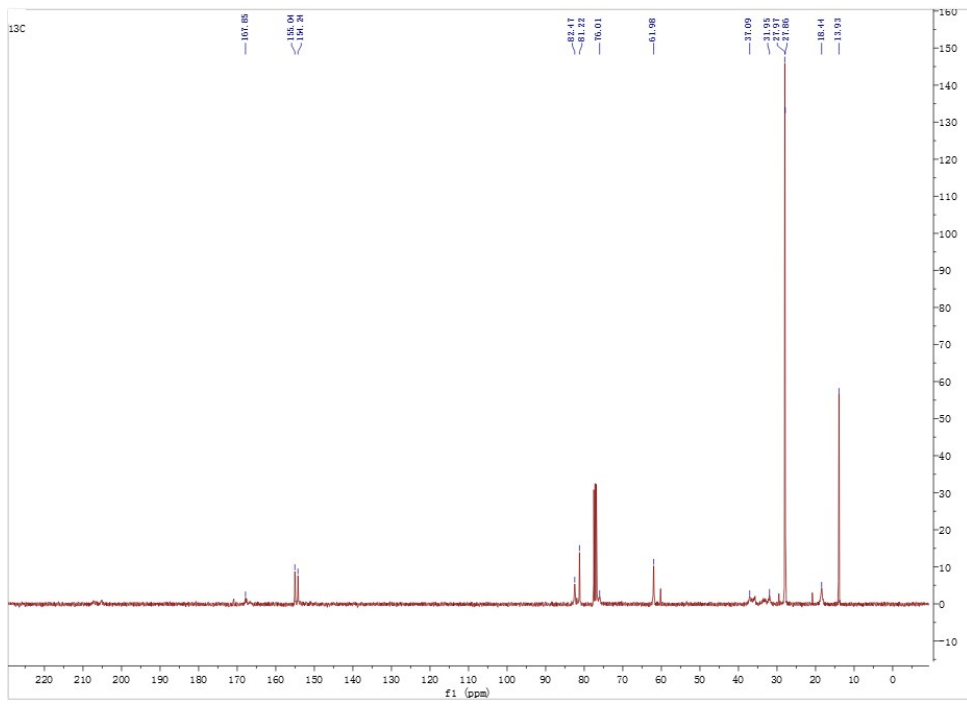
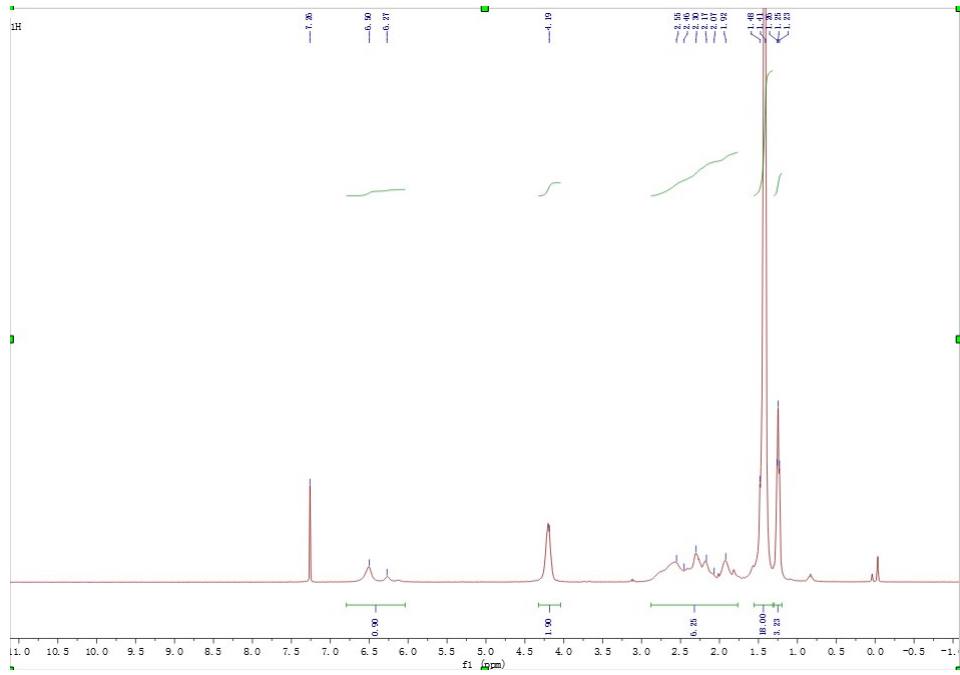
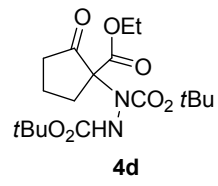


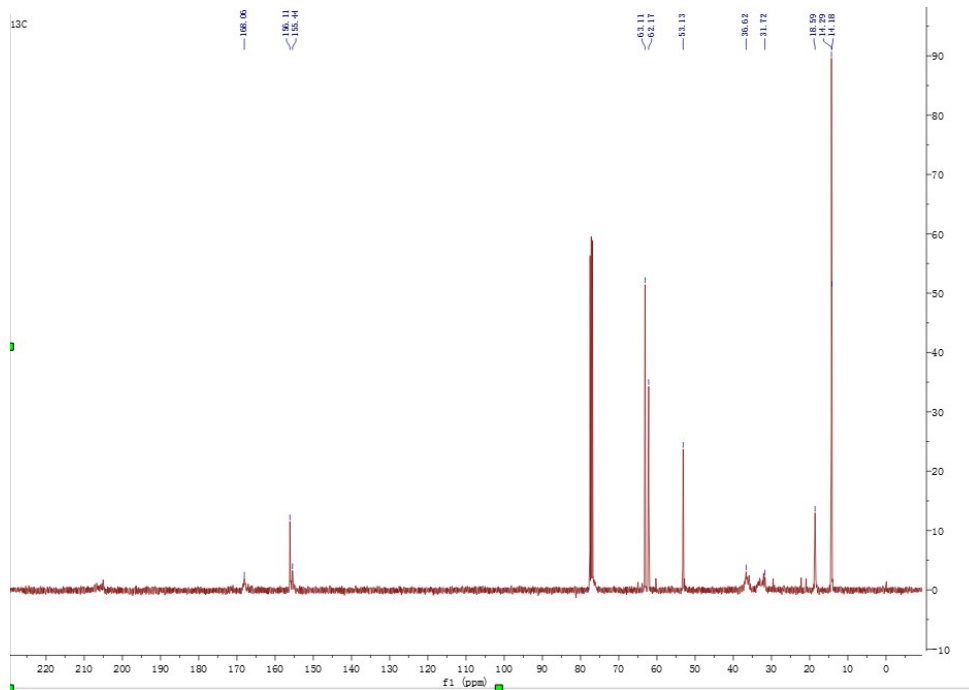
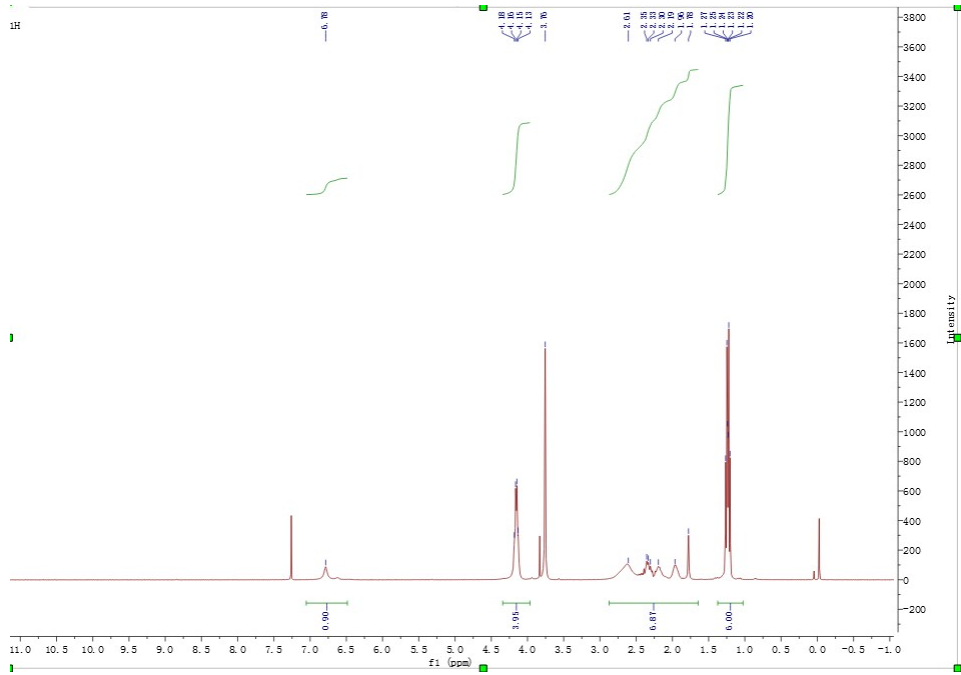
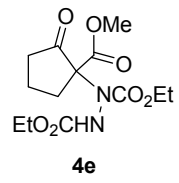


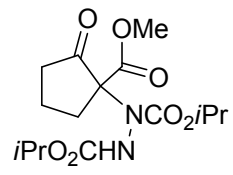


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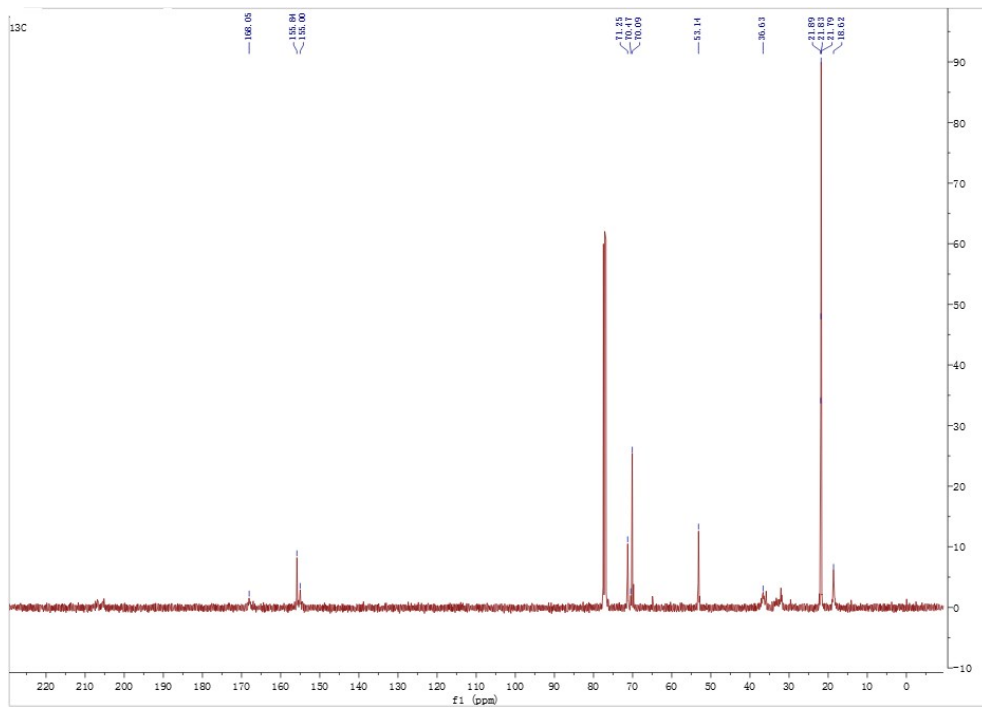
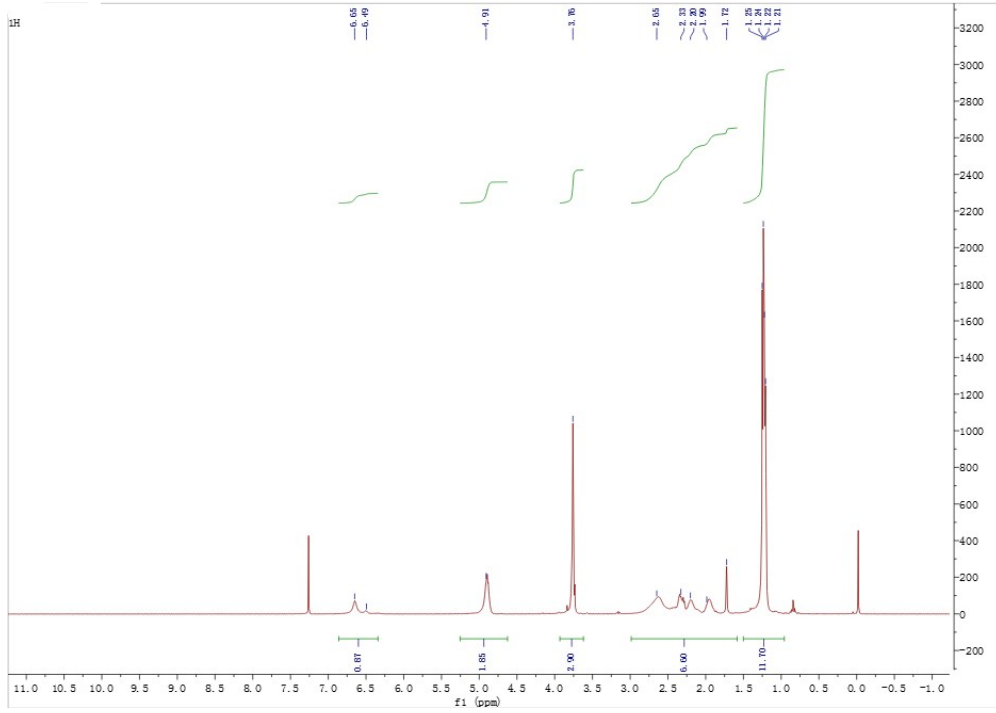


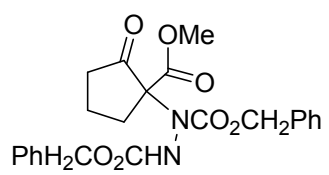




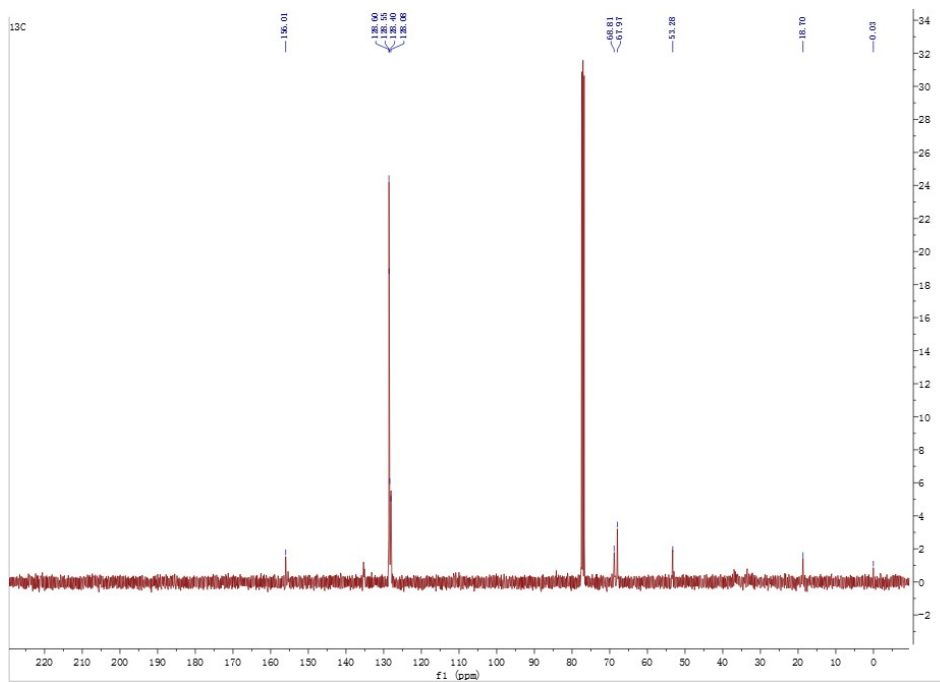
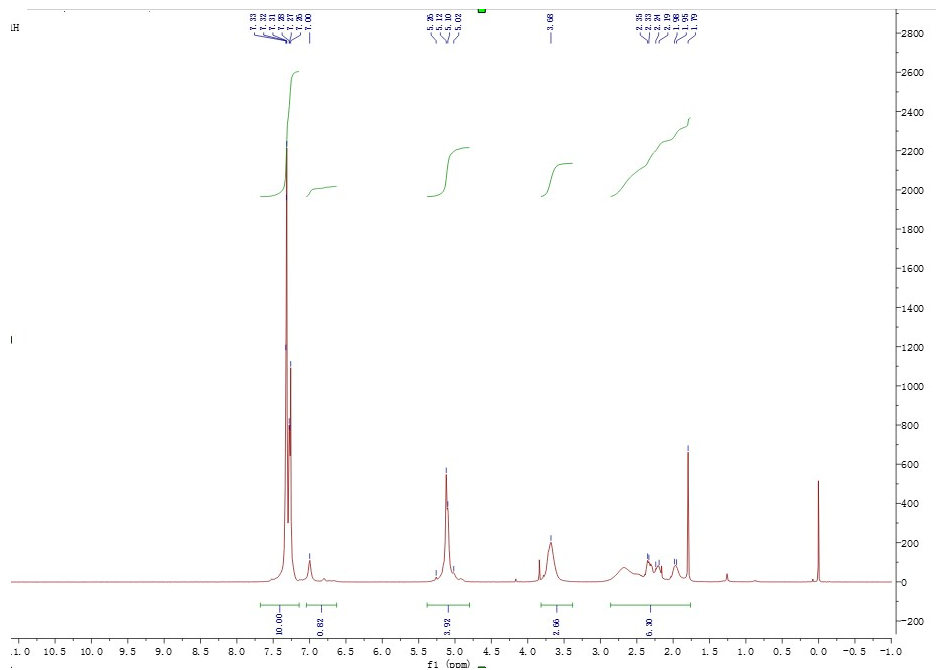


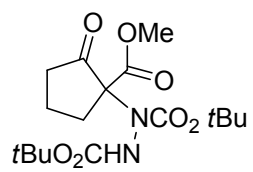
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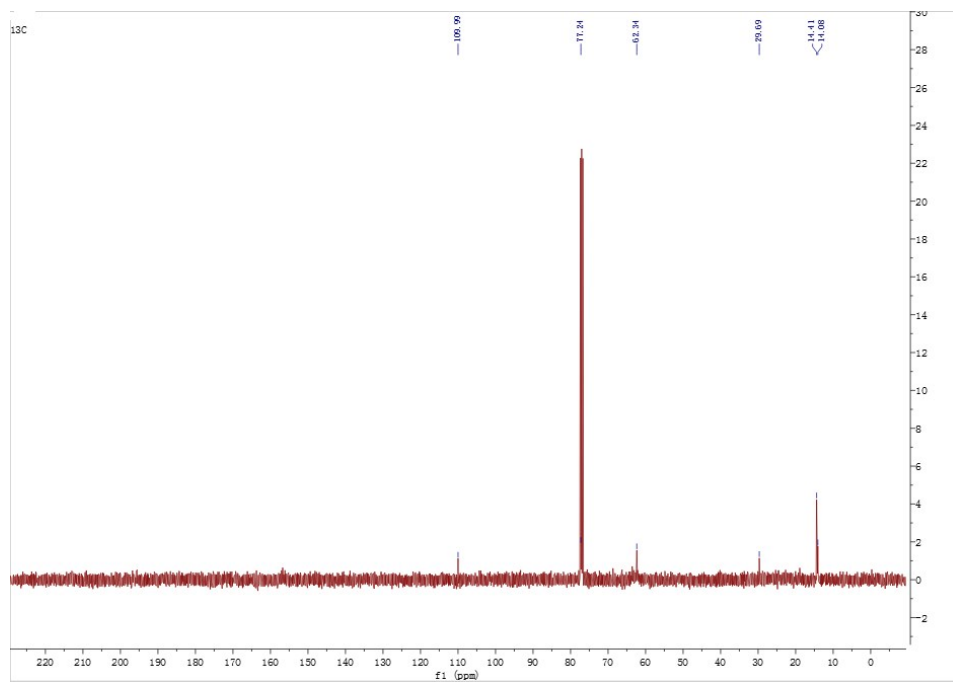
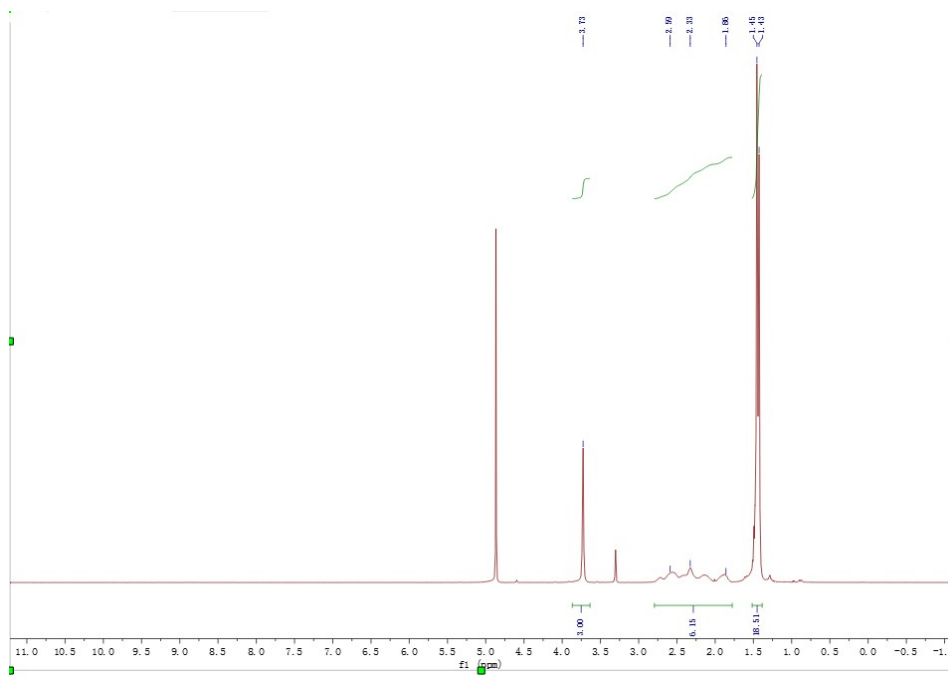


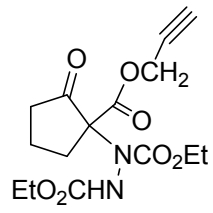
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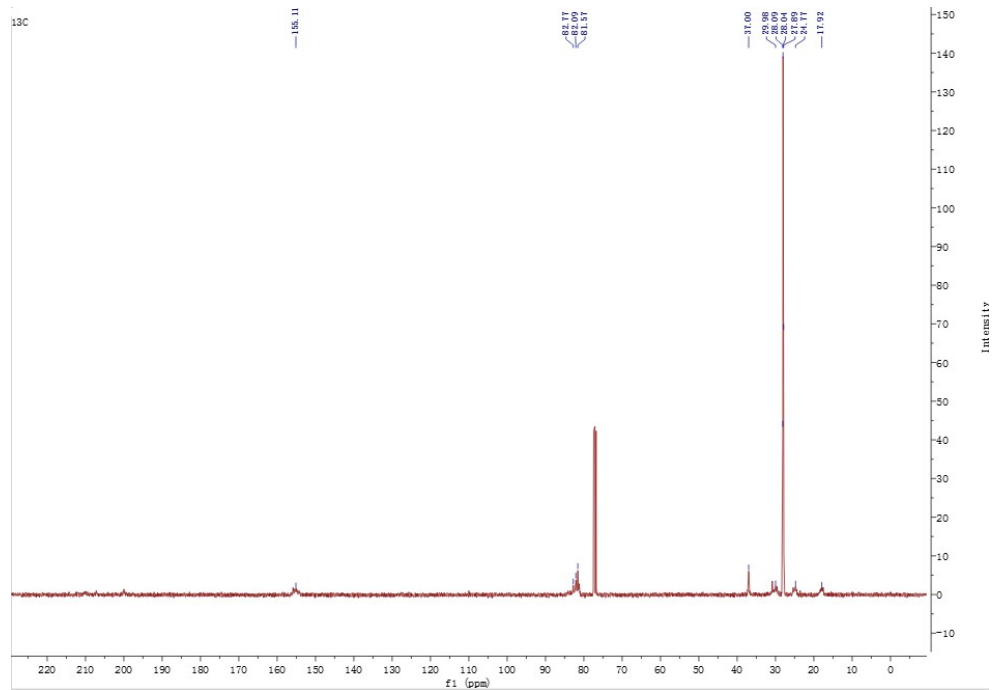
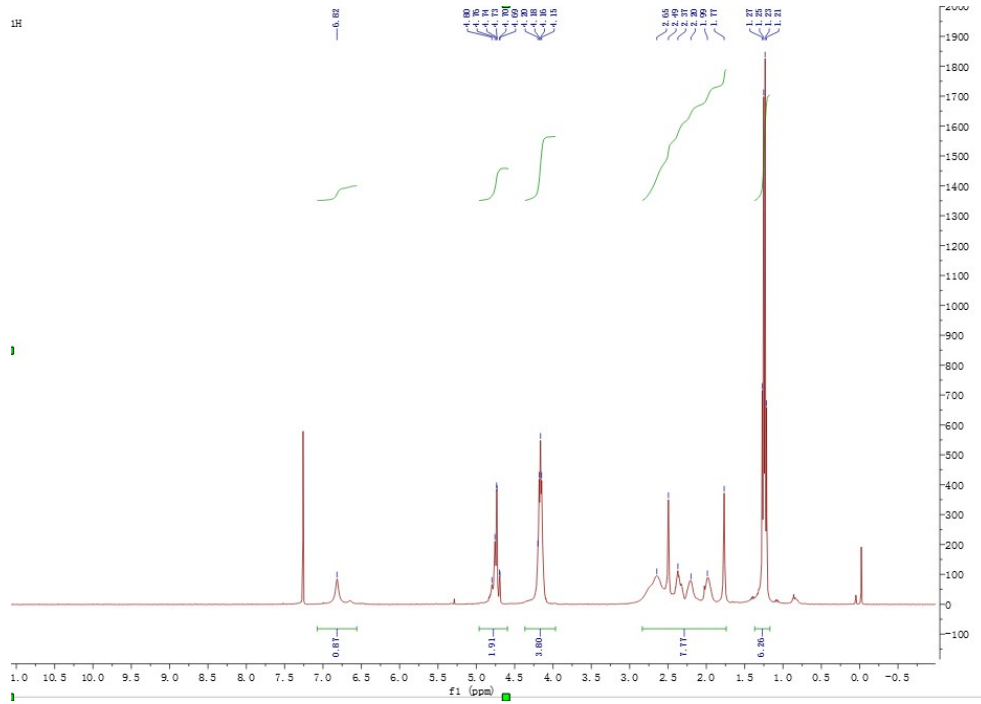


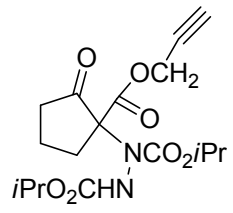
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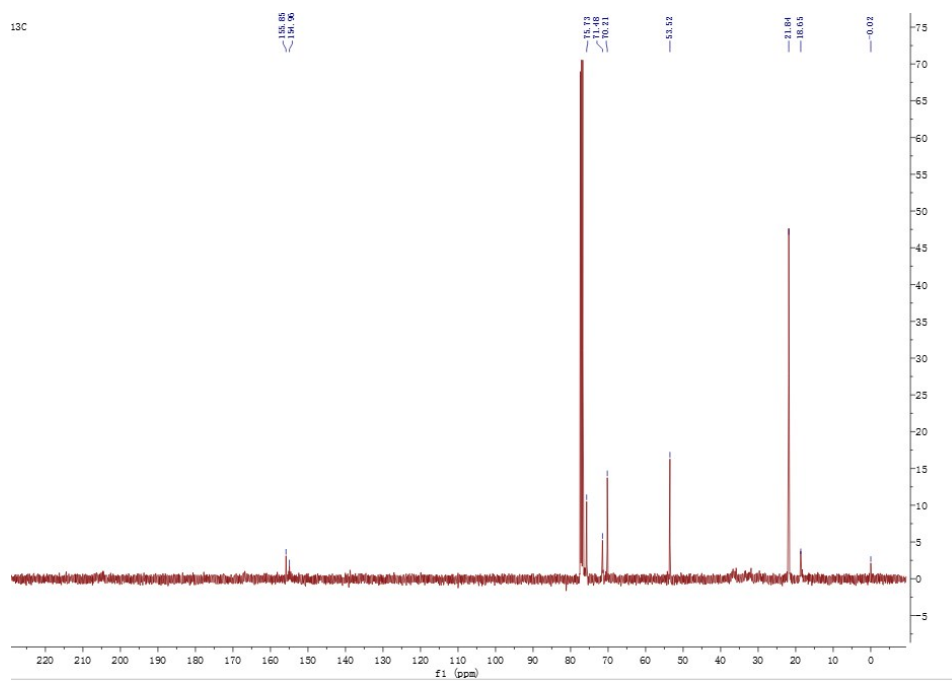
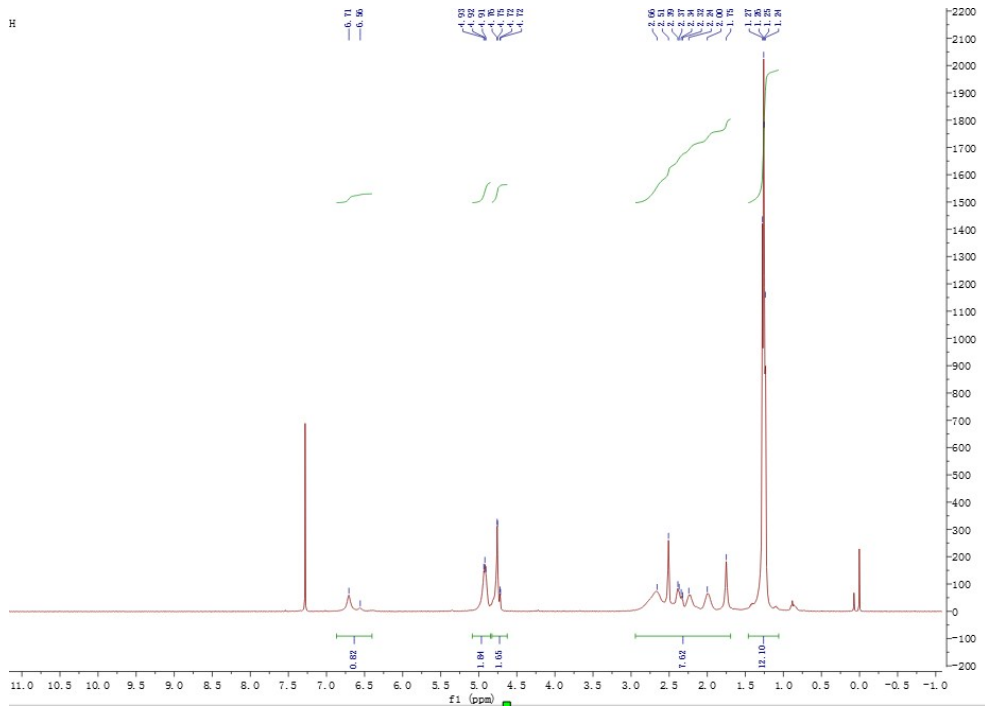


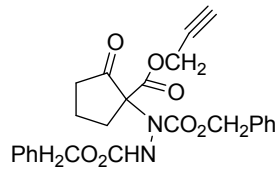
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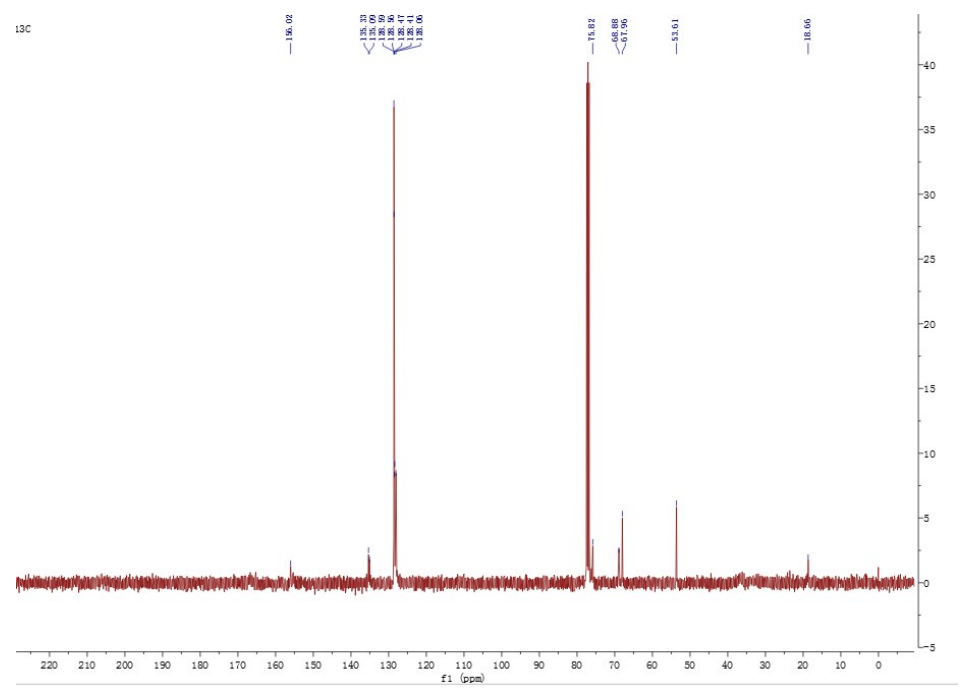
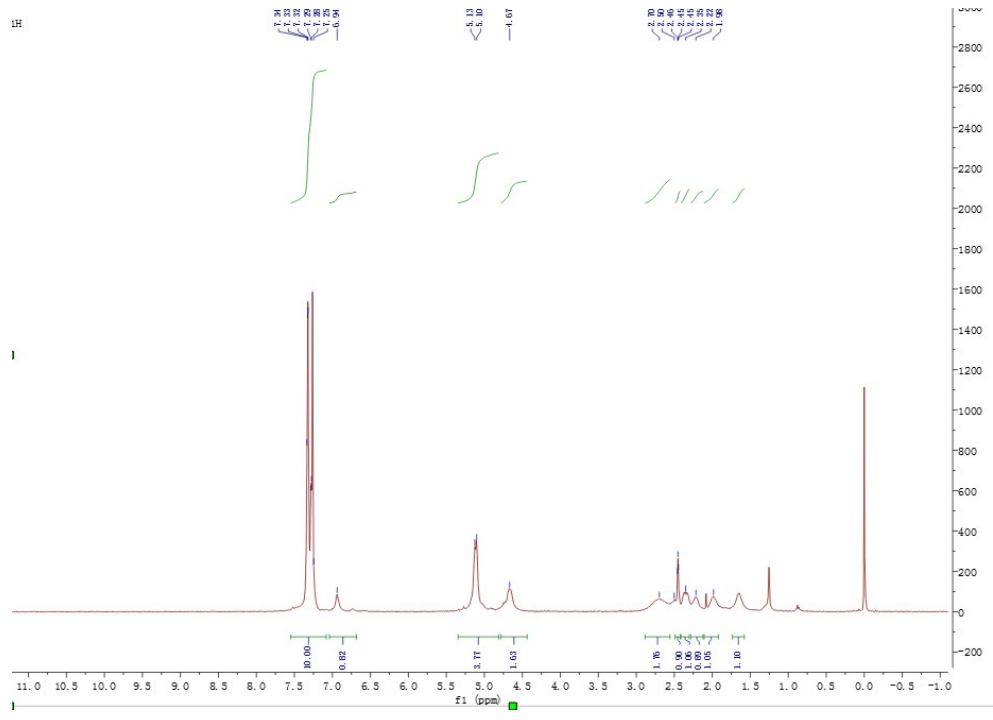


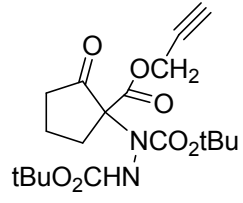
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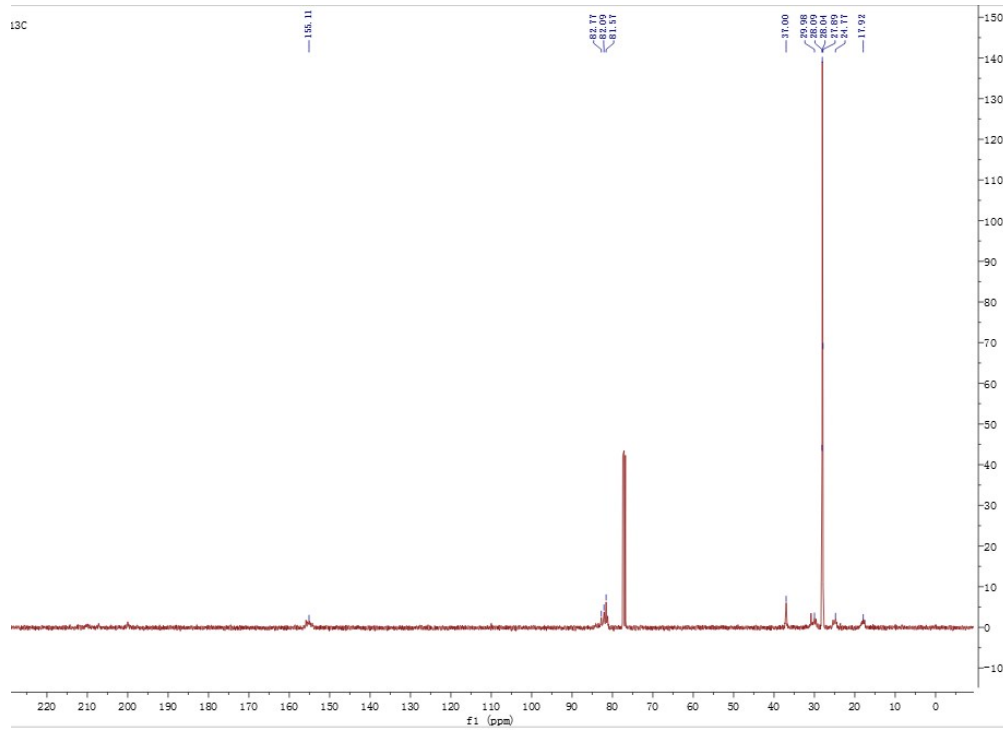
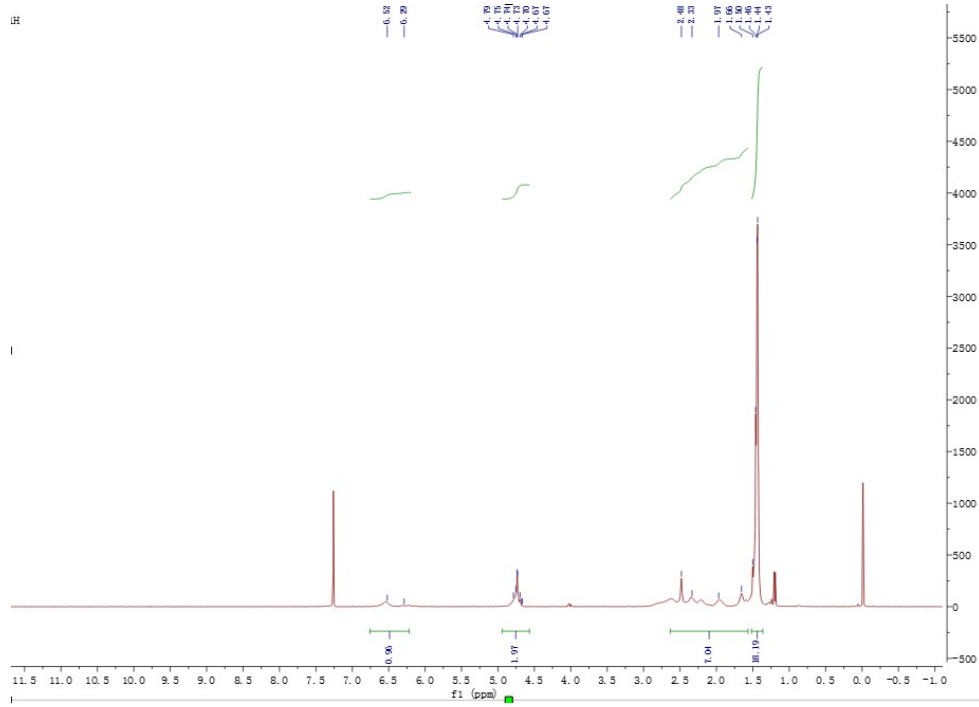


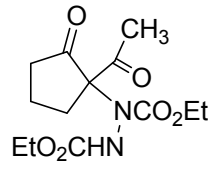
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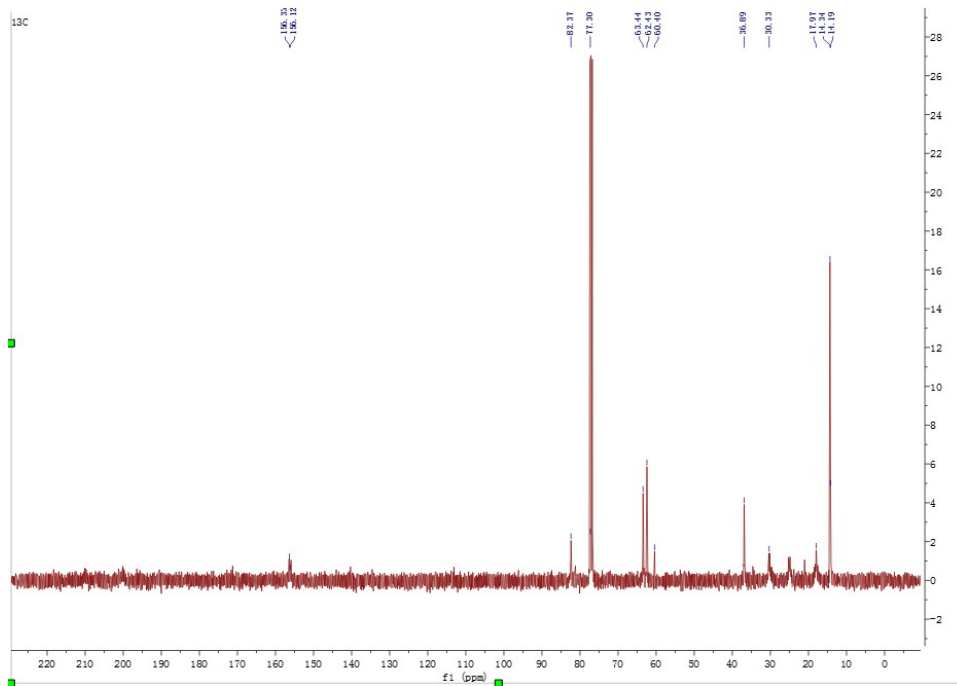
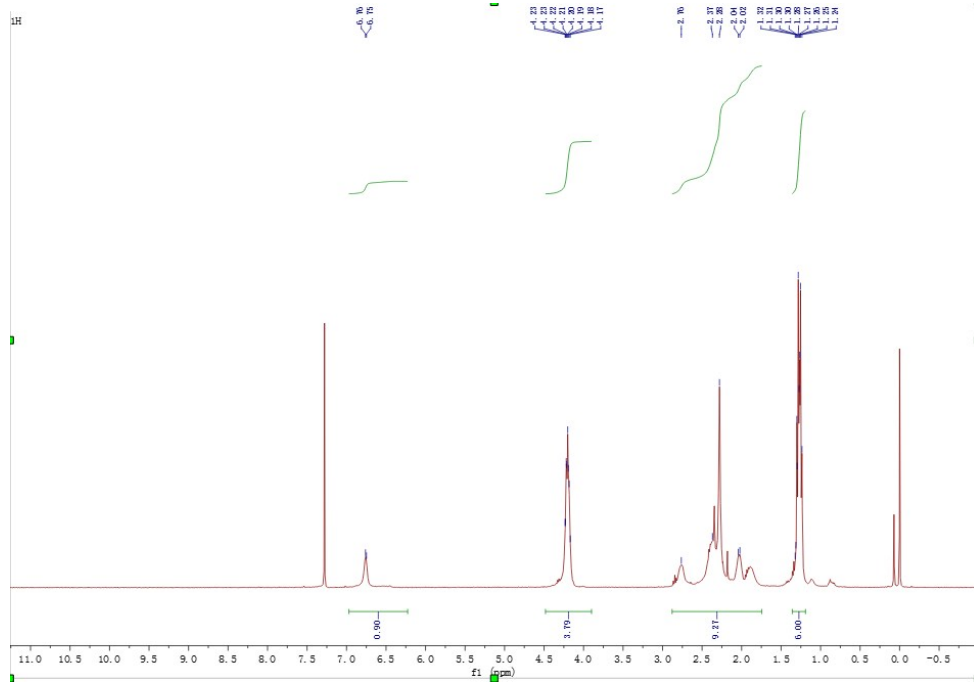


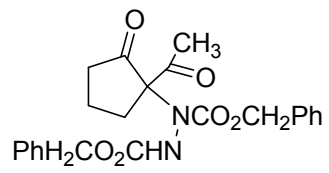
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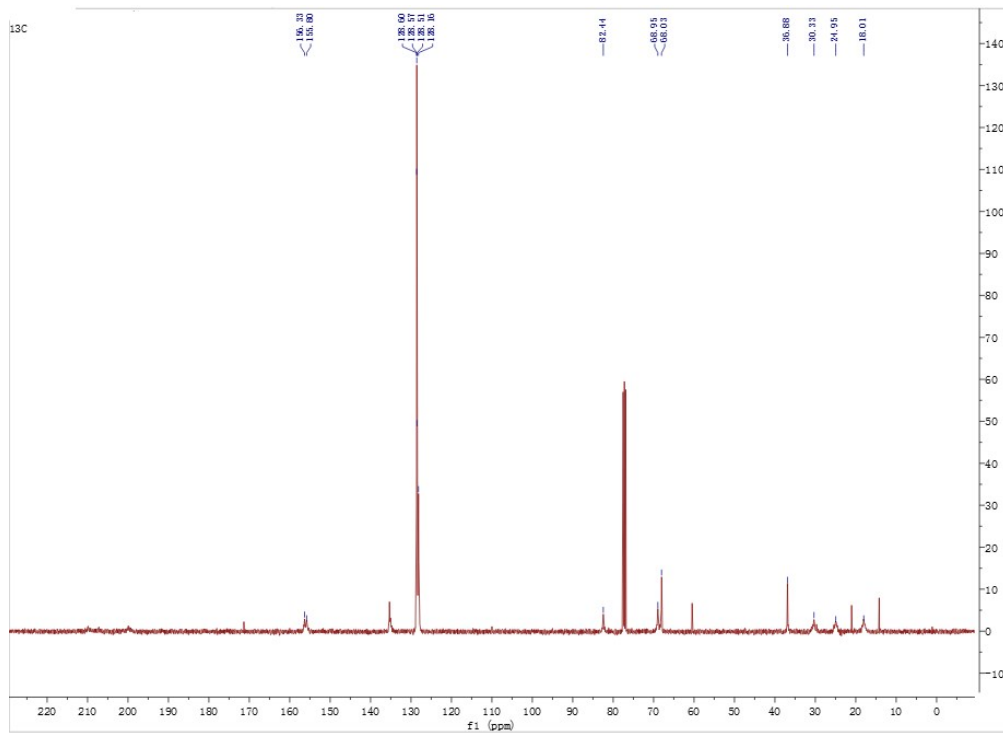
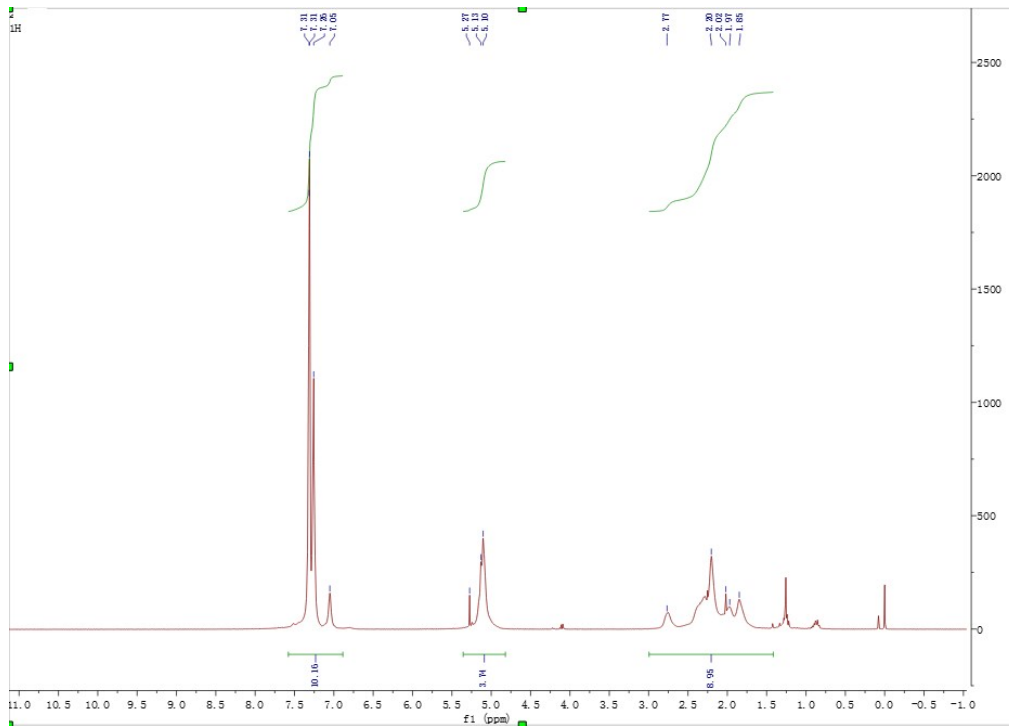


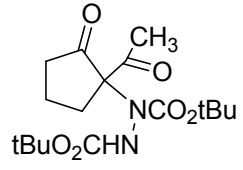
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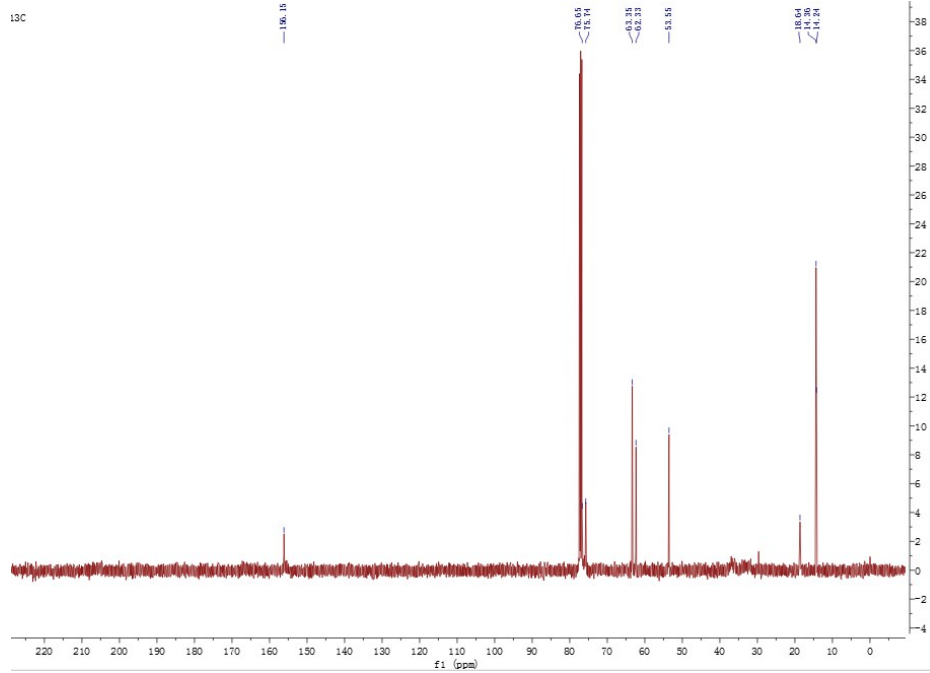
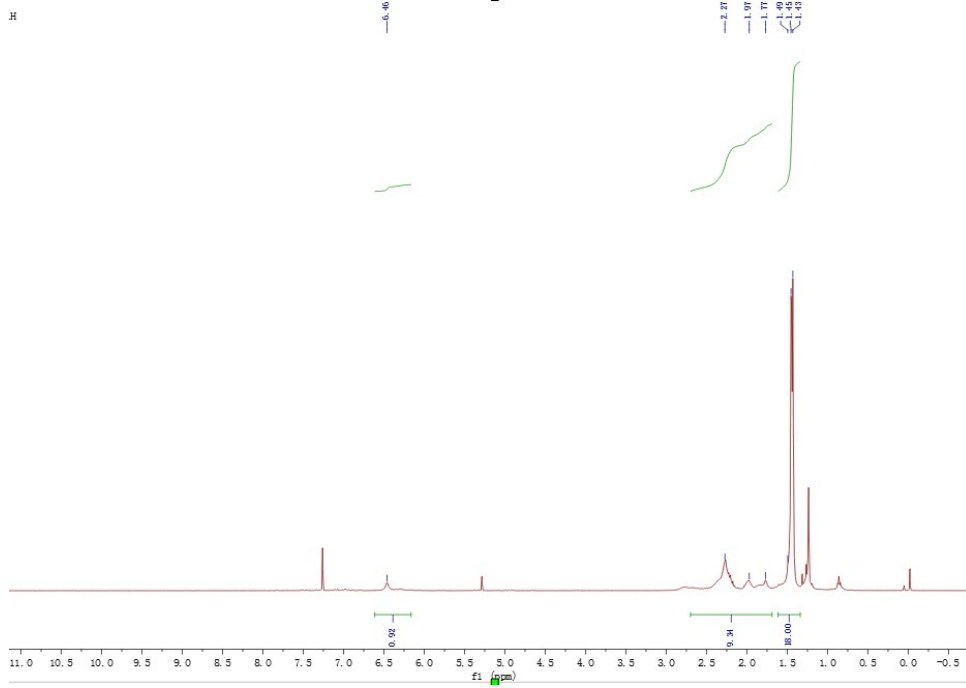


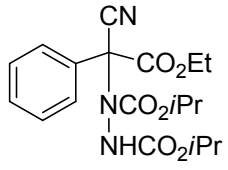
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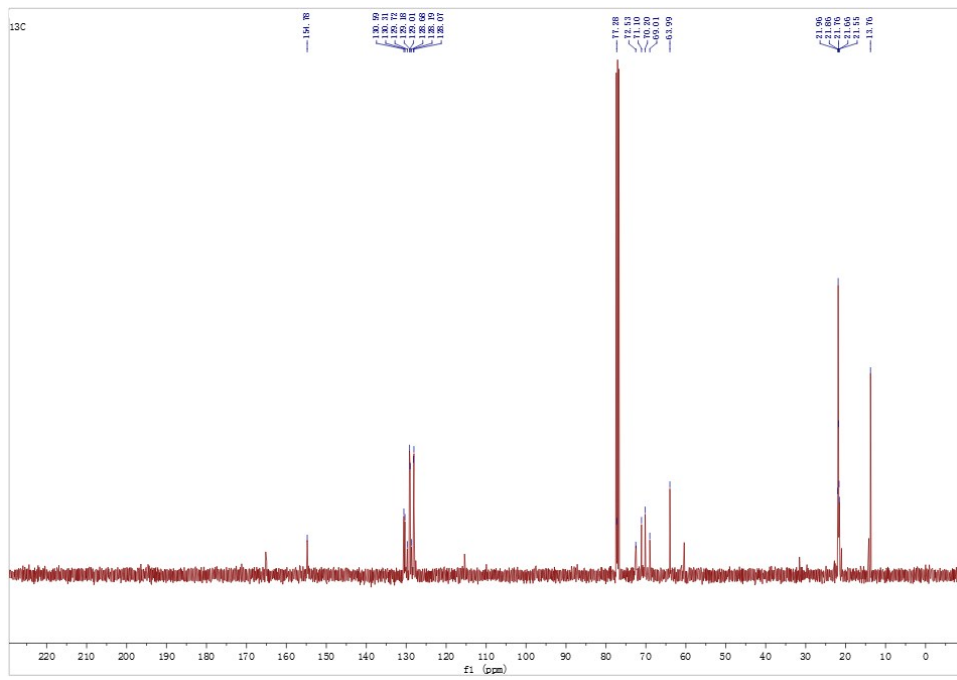
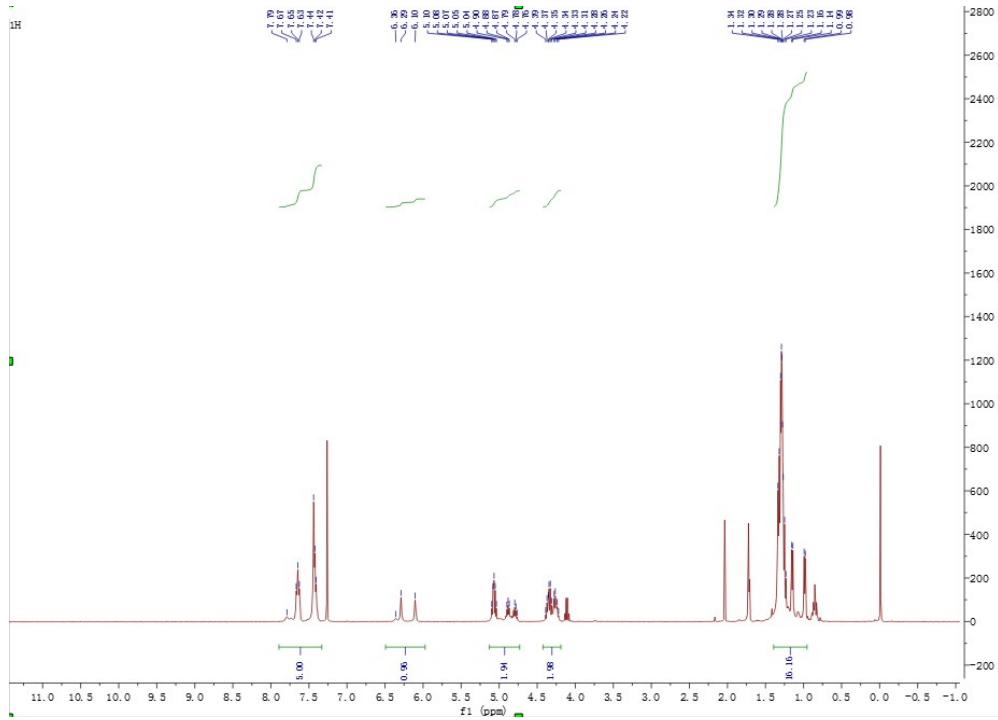


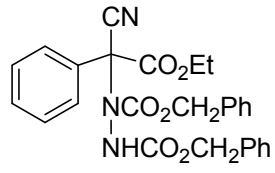
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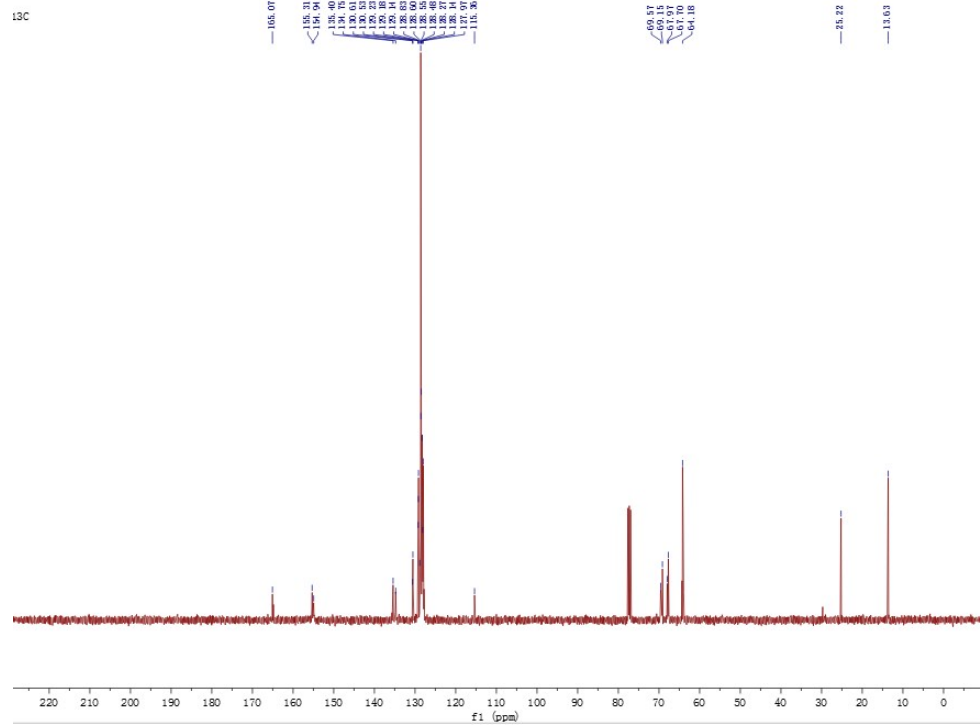
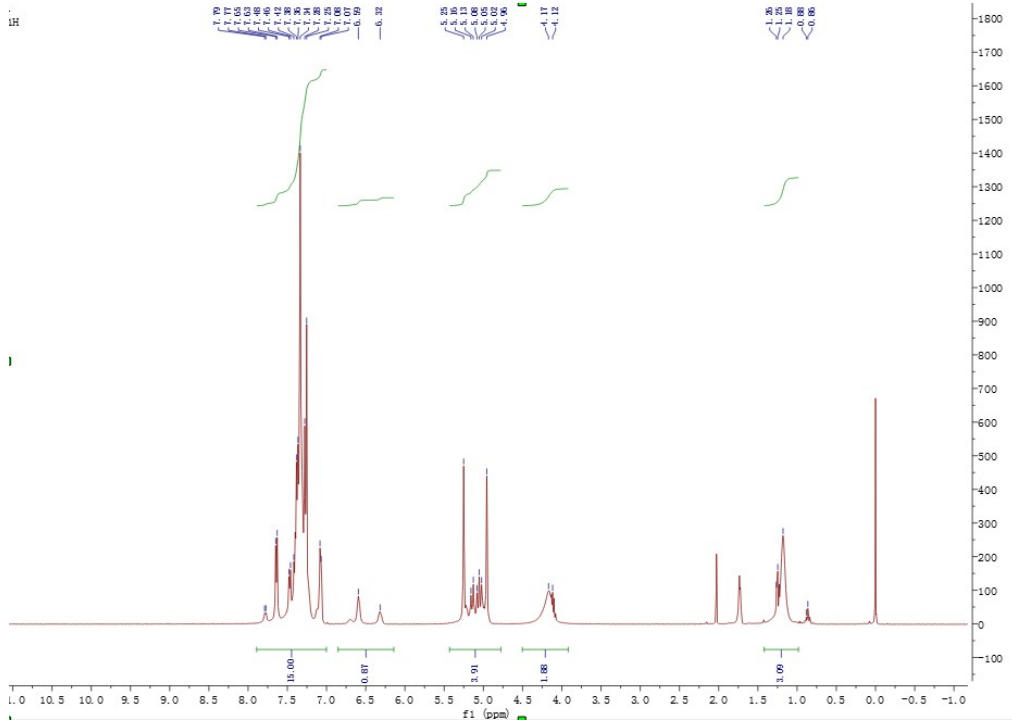


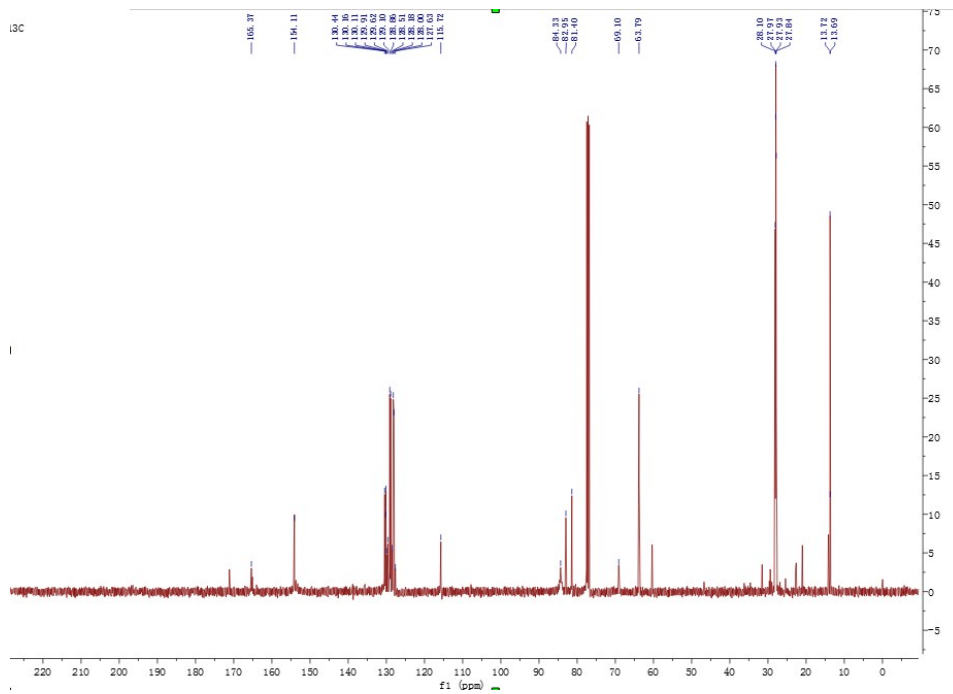
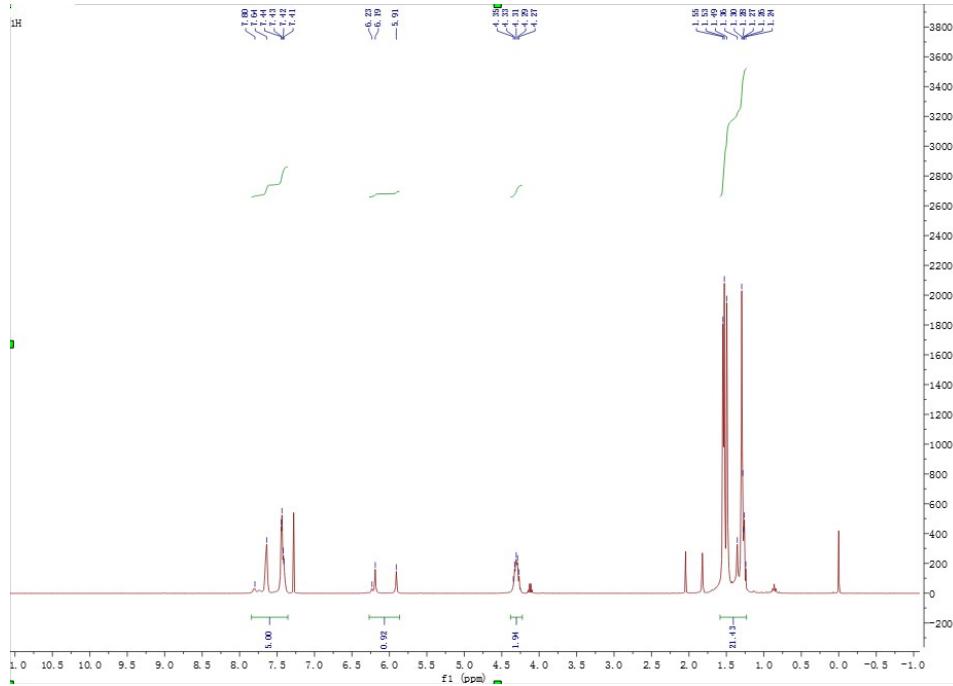
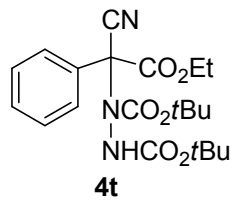
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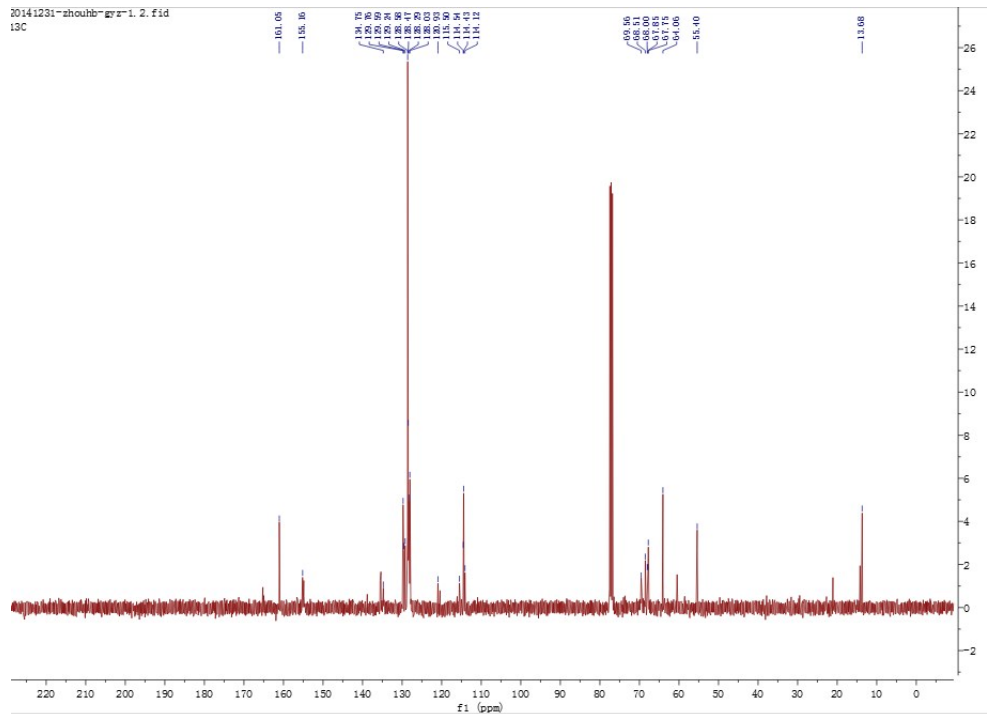
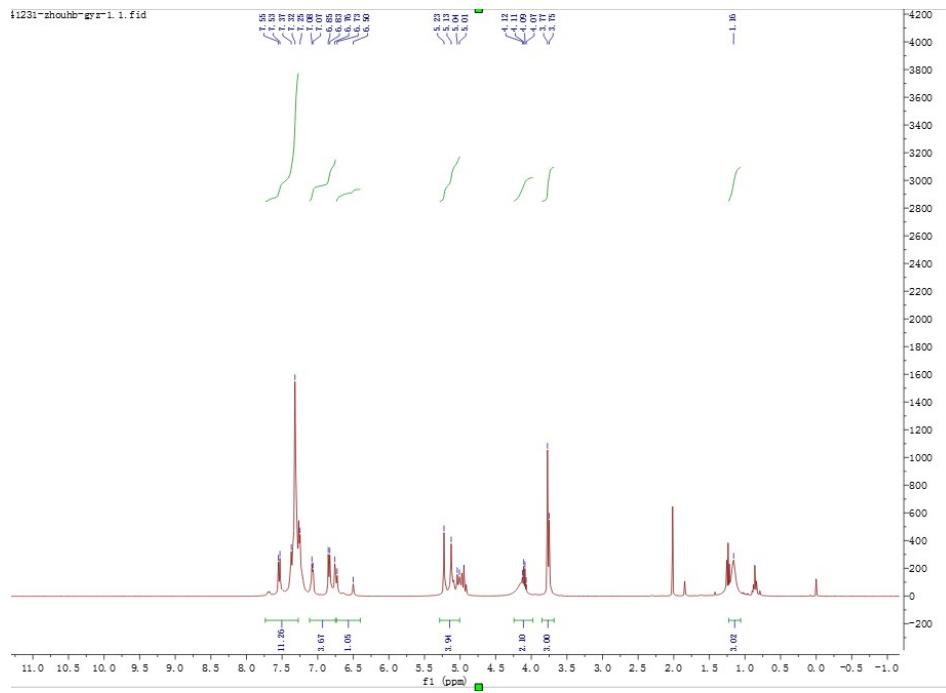
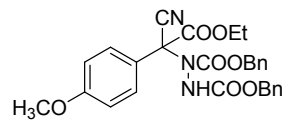


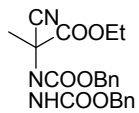


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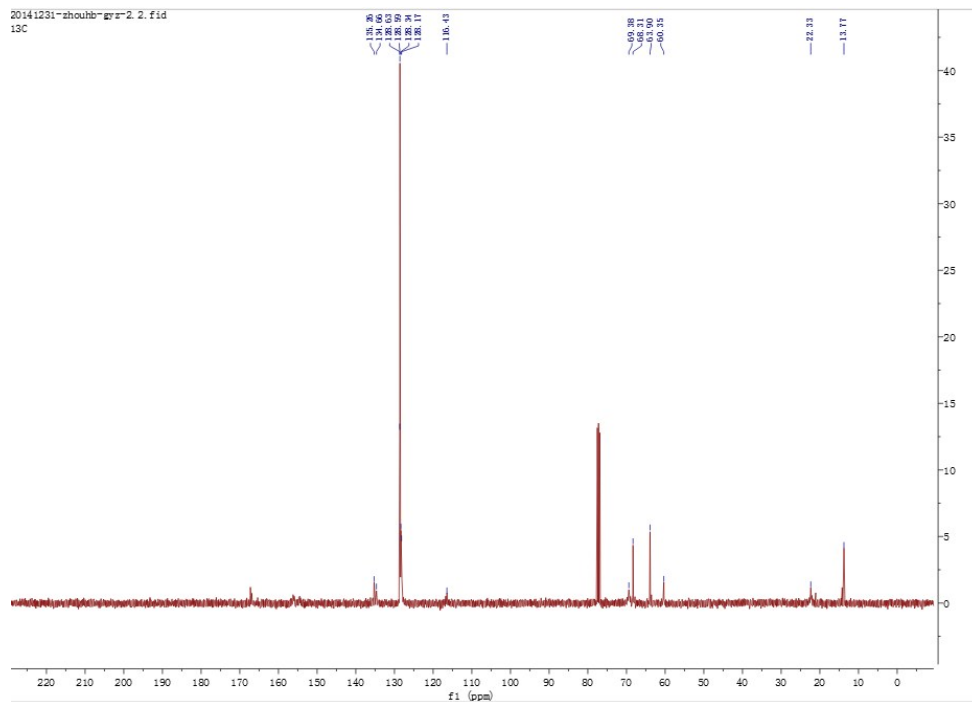
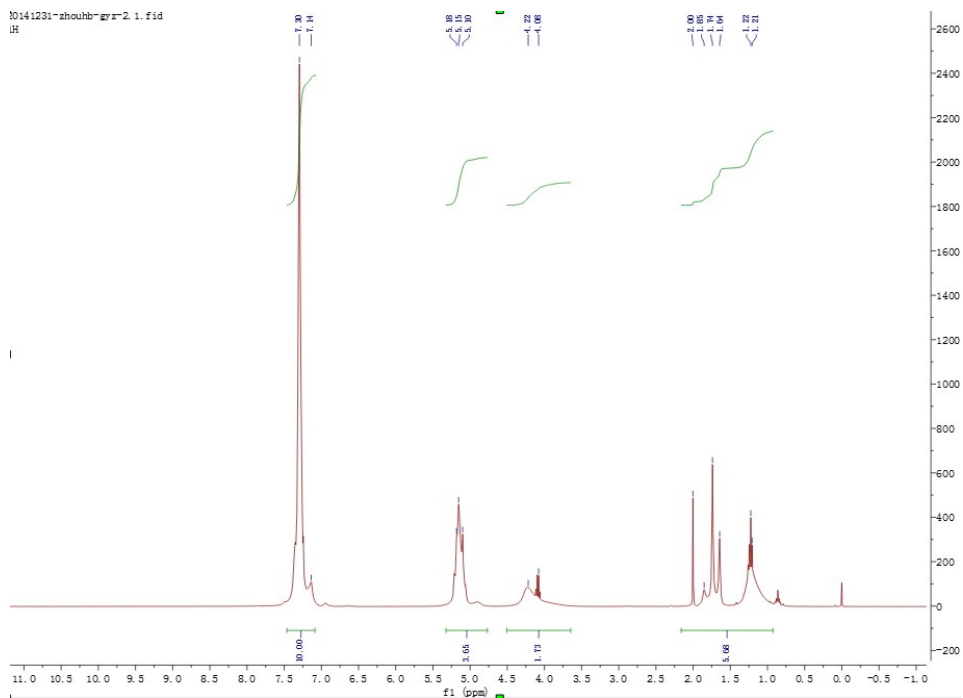


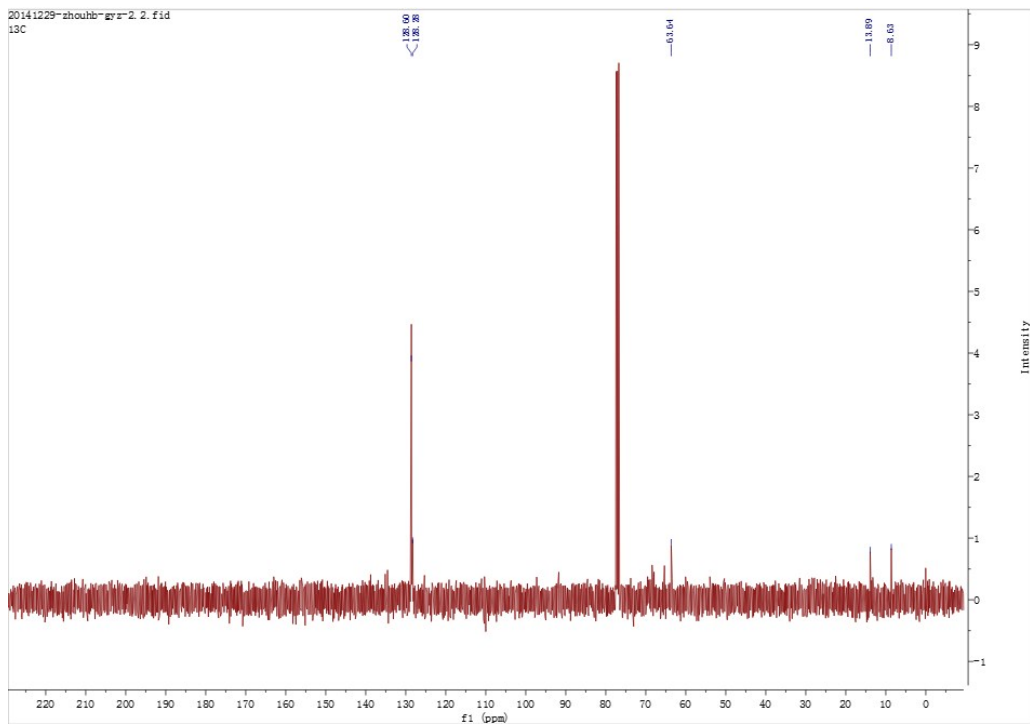
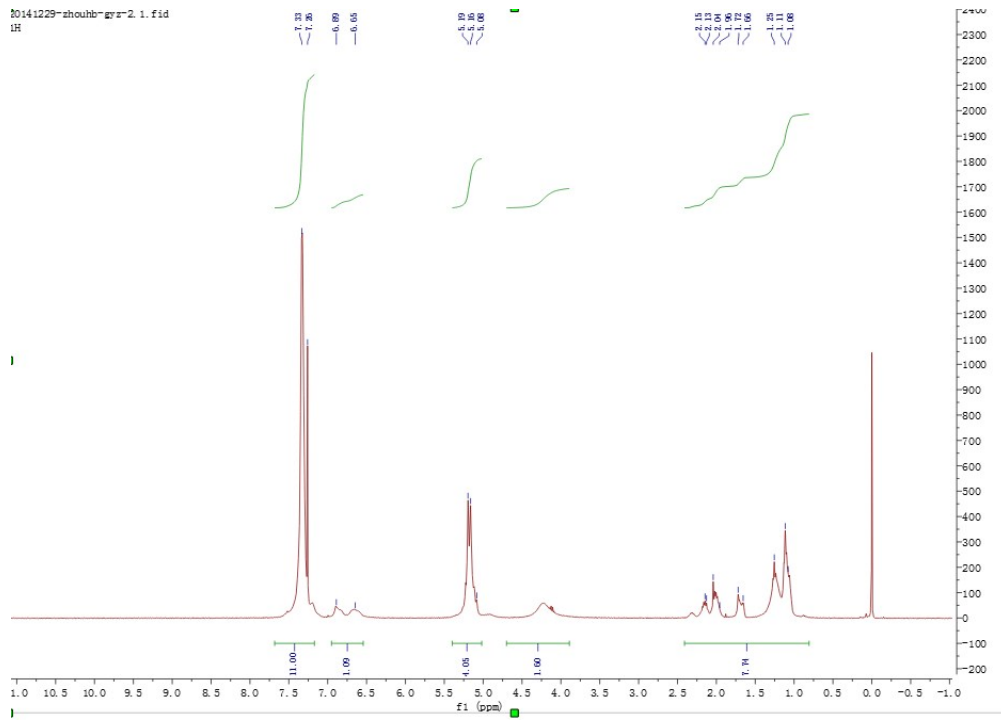
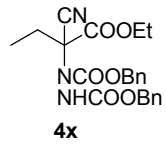


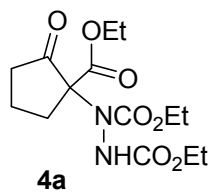




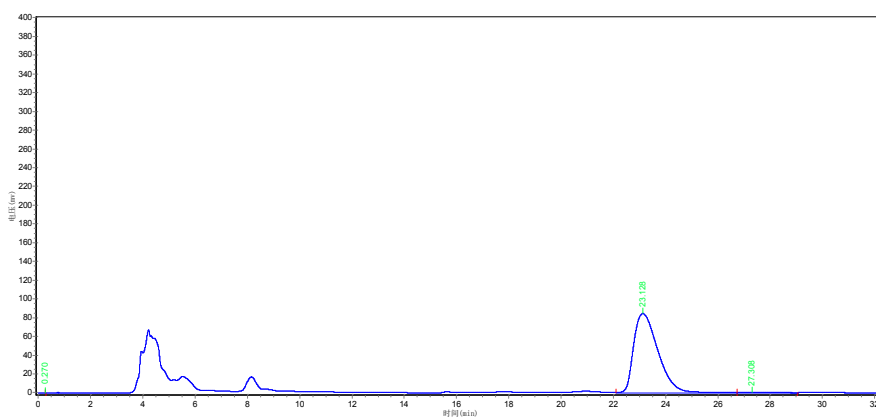
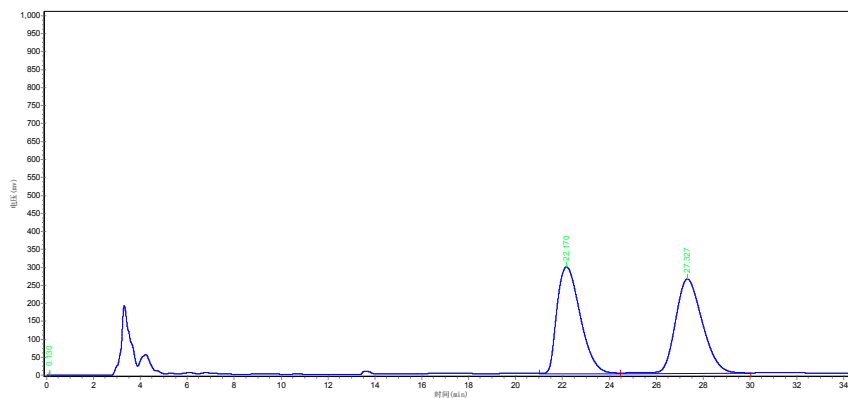
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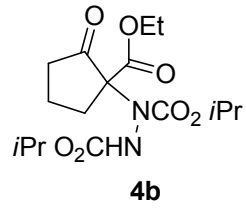




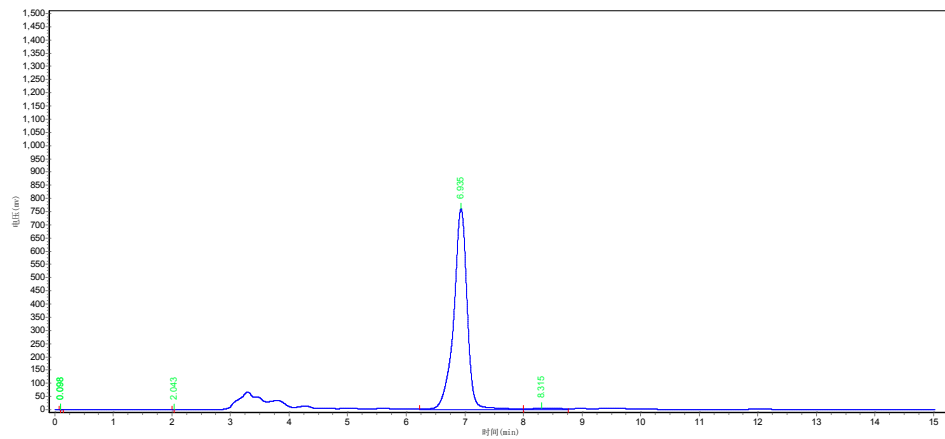
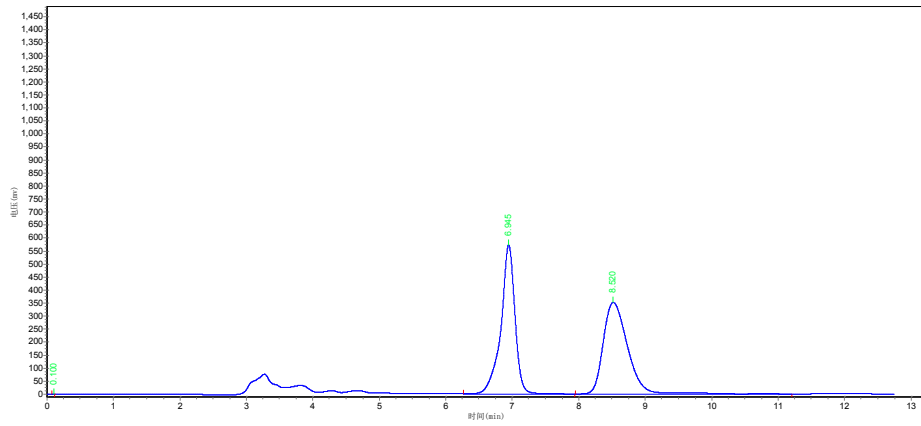
Chiralpak AD-H, 5% iPrOH in hexanes, 1.0 mL/min, 210nm



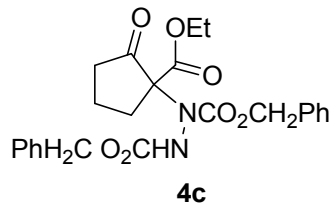
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Total				100



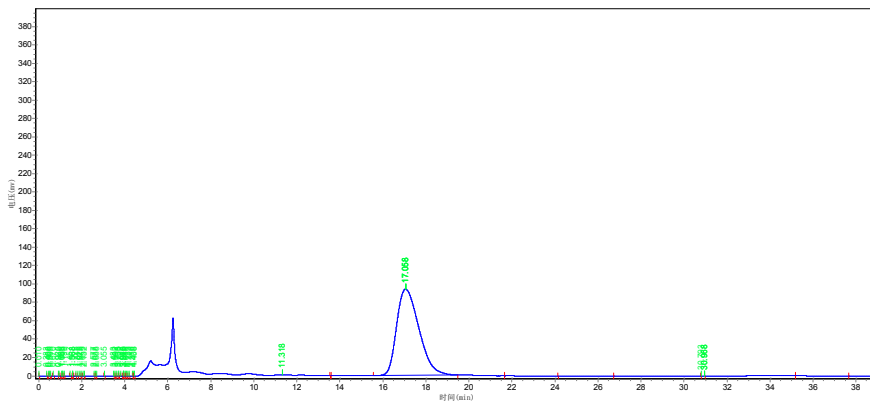
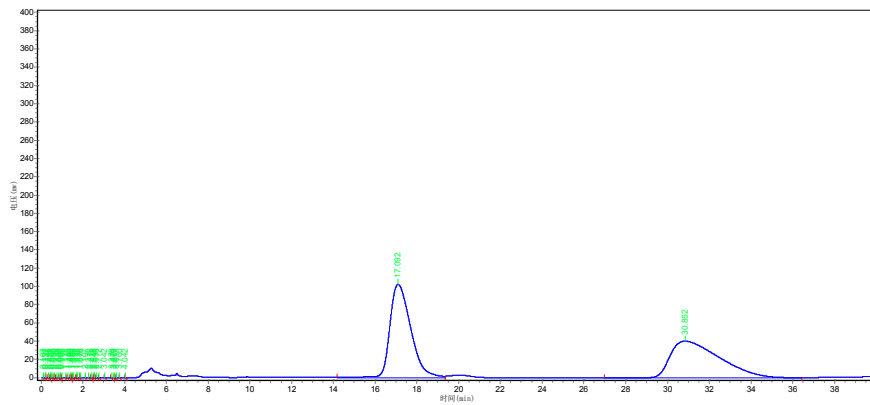
Chiralpak AD-H, 10% iPrOH in hexanes, 1.0 mL/min, 210nm



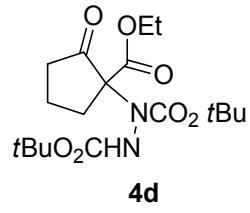
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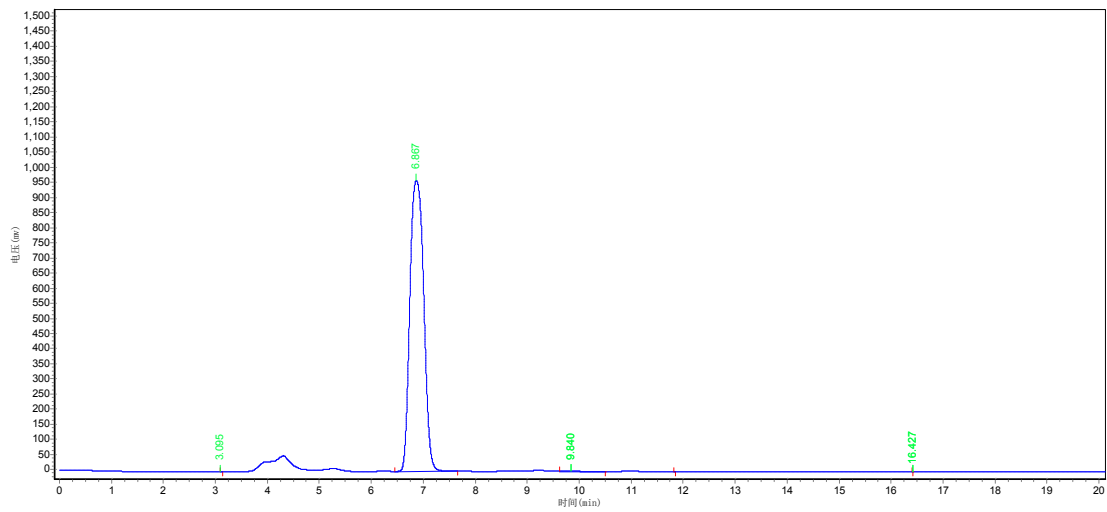
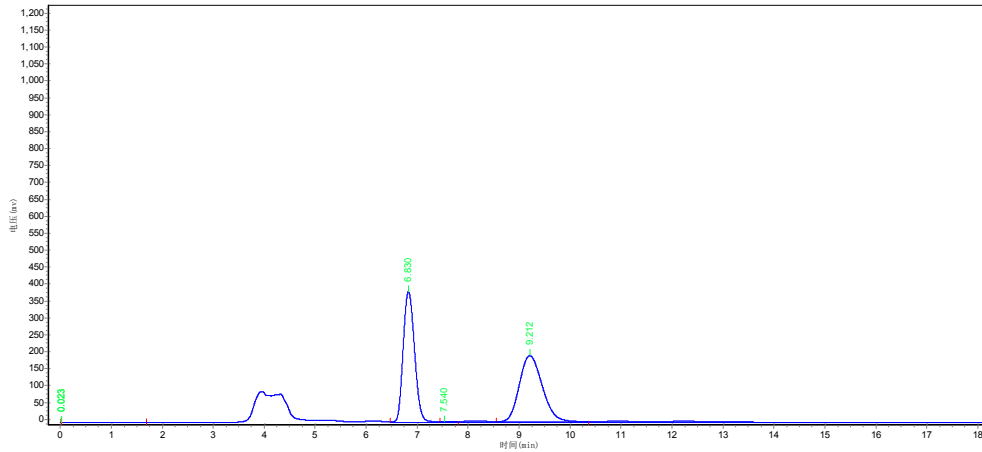
4c Chiralpak AS-H, 10% iPrOH in hexanes, 0.65mL/min, 254nm



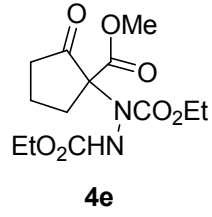
Peak	Ret. Time	Height	Area	Area (%)
1	17.058	93703.883	6739804.500	99.9980
2	30.793	1.692	1.050	0.0020
Totals				100



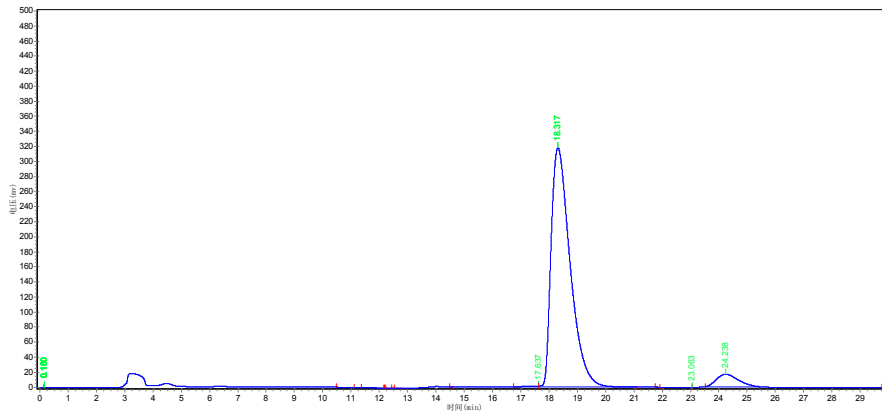
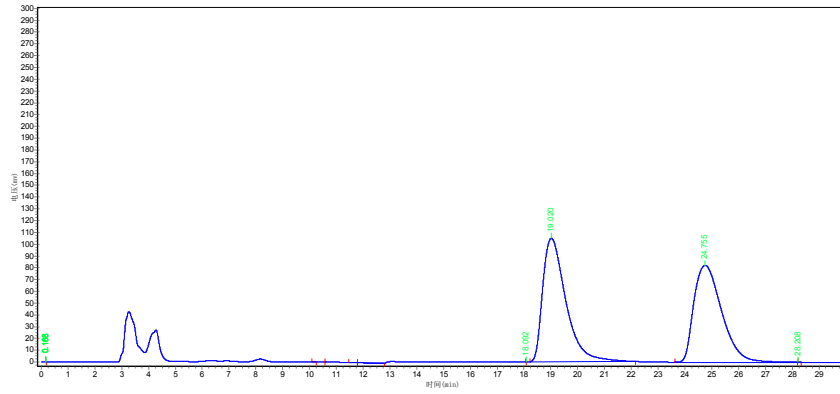
Chiralpak AD-H, 20% *i*PrOH/hexane, 0.8mL/min, 210 nm.



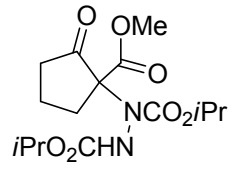
Peak	Ret. Time	Height	Area	Area (%)
1	6.867	961030.125	17737688.000	99.6340
2	9.840	2364.933	64971.461	0.3660
Totals				100



Chiralpak AD-H, 5%*i*PrOH in hexane, 1.0mL/min, 210nm

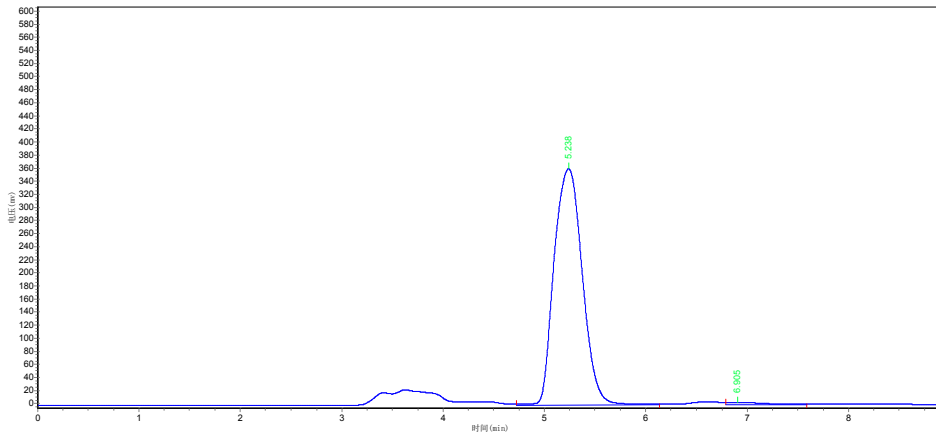
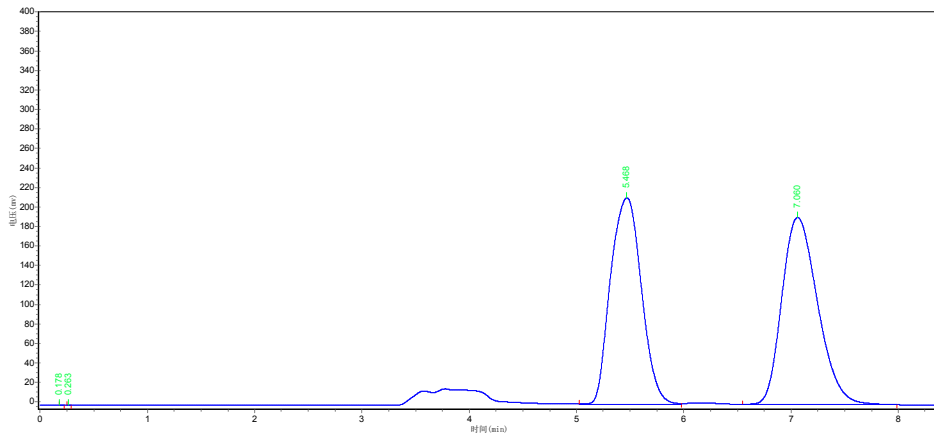


Peak	Ret. Time	Height	Area	Area (%)
1	18.317	317867.219	14792808.000	94.7205
2	24.238	16386.1501	866449.500	5.2795
Totals				100

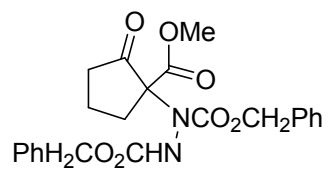


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Chiralpak AD-H, 20% *i*PrOH in hexanes, 0.8 mL/min, 210nm

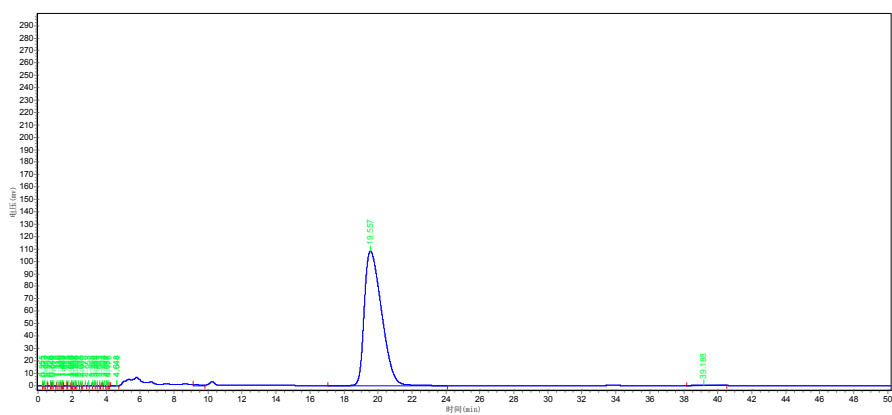
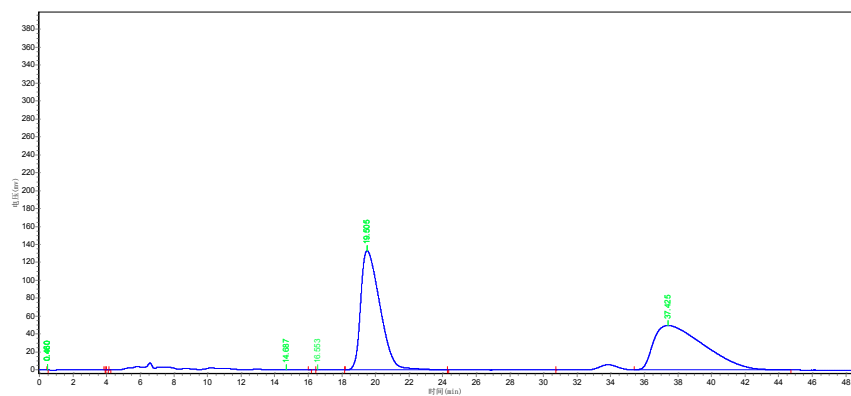


Peak	Ret. Time	Height	Area	Area (%)
1	5.238	361251.250	6997275.000	98.4777
2	6.905	3936.854	108163.383	1.5223
Total				100

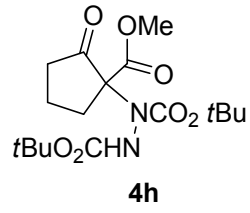


4g

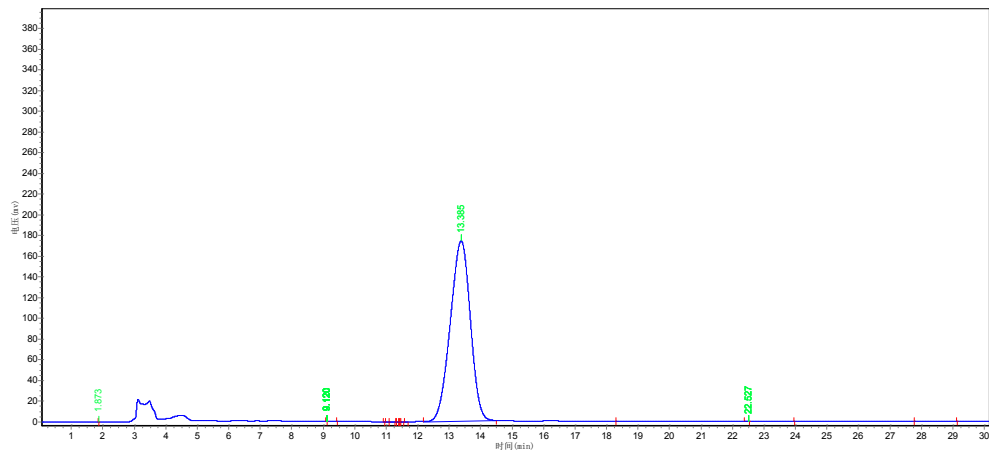
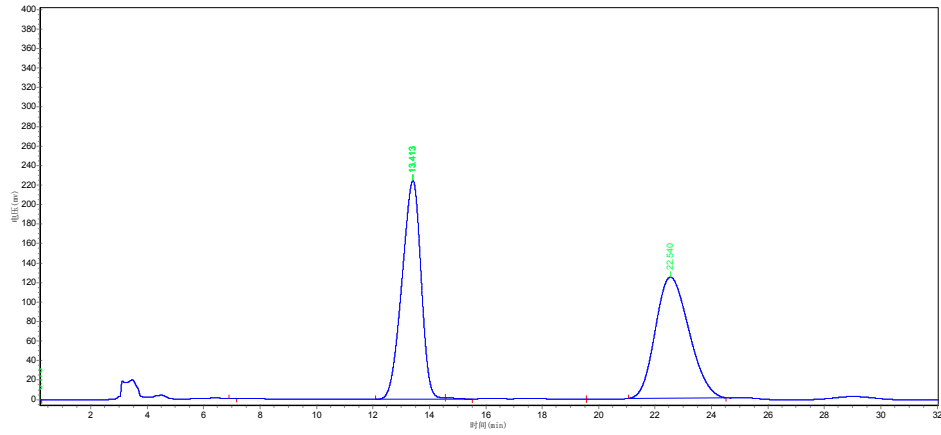
Chiralpak AD-H, 30% *i*PrOH in hexanes, 0.65 mL/min, 254nm



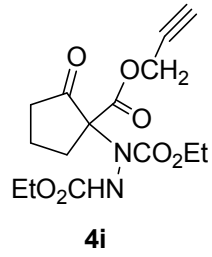
Peak	Ret. Time	Height	Area	Area (%)
1	19.557	108650.281	7728479.500	99.4020
2	39.188	611.017	46490.750	0.5980
Total				100



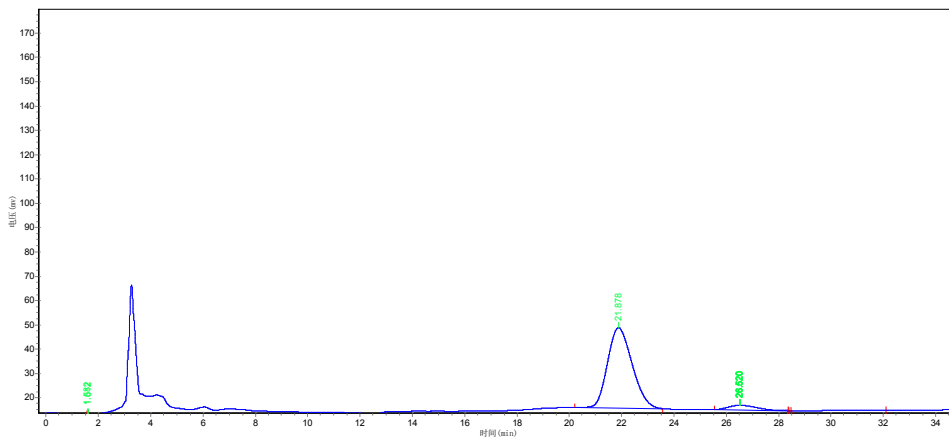
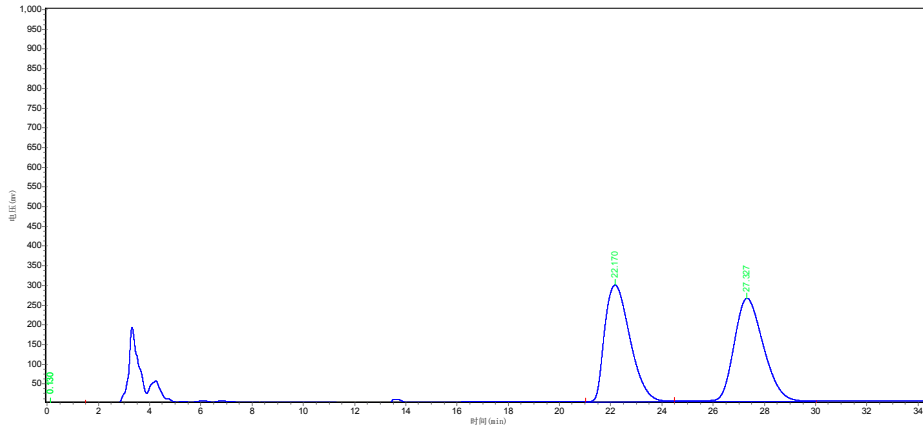
Chiralpak AD-H, 10% *i*PrOH in hexanes, 1.0 mL/min, 210nm



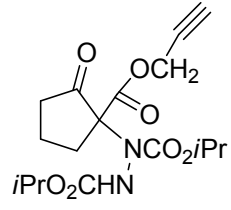
Peak	Ret. Time	Height	Area	Area (%)
1	13.385	174237.031	7703676.000	99.9970
2	22.527	324.245	20.569	0.0030
Totals				100



Chiralpak AD-H, 5%*i*PrOH in hexane, 1.0mL/min, 210nm

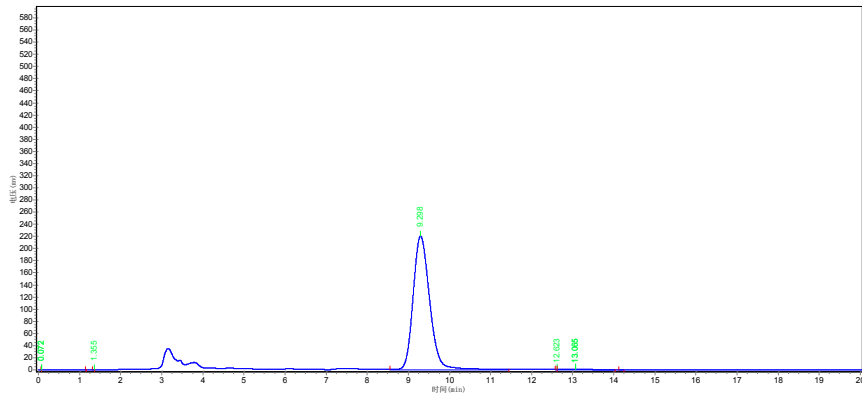
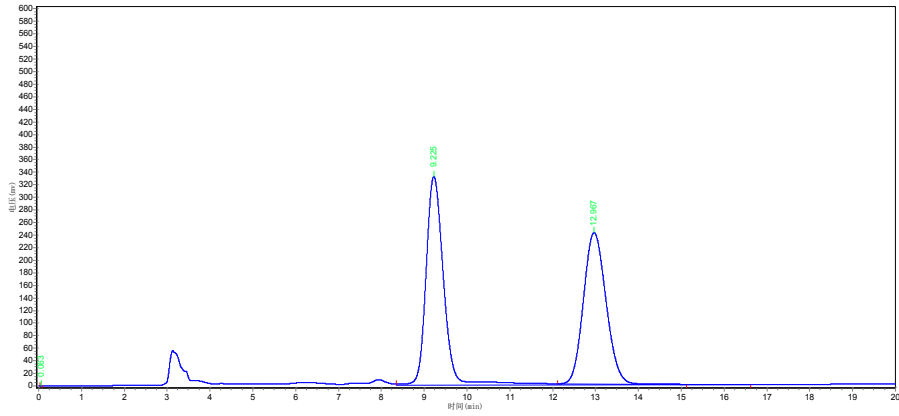


Peak	Ret. Time	Height	Area	Area (%)
1	21.878	33745.617	2273638.250	95.0748
2	26.520	1952.783	143200.672	4.9252
Totals				100

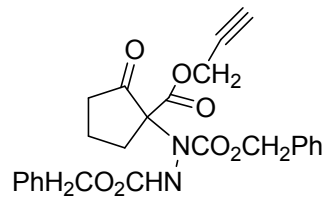


4j

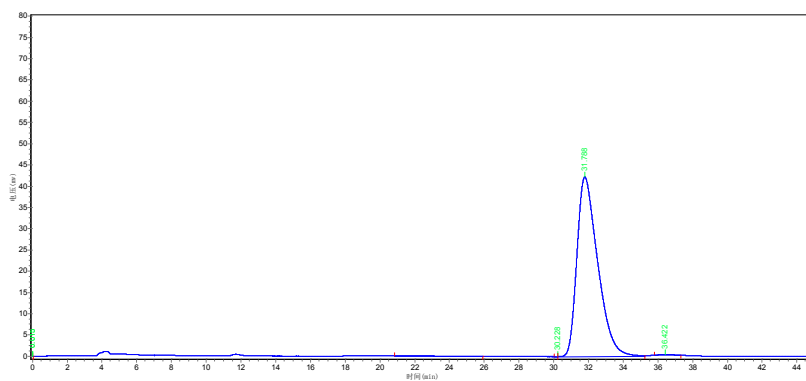
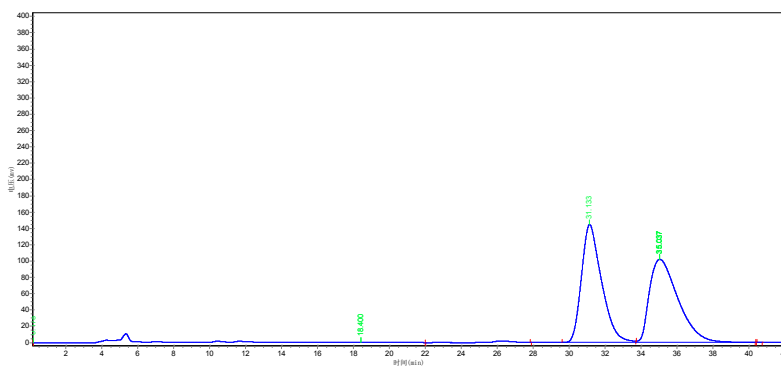
Chiralpak AD-H, 10%*i*PrOH in hexane, 1.0mL/min, 210nm



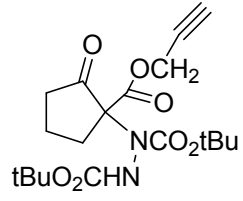
Peak	Ret. Time	Height	Area	Area (%)
1	9.298	220128.219	6158082.000	99.5952
2	13.065	628.000	25028.645	0.4048
Totals				100



Chiralpak AD-H, 10% *i*PrOH in hexanes, 1.0 mL/min, 210nm

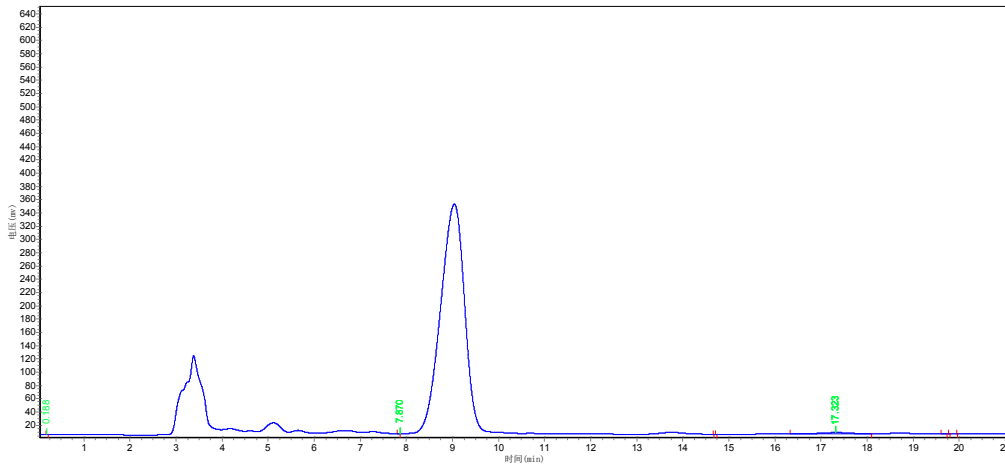
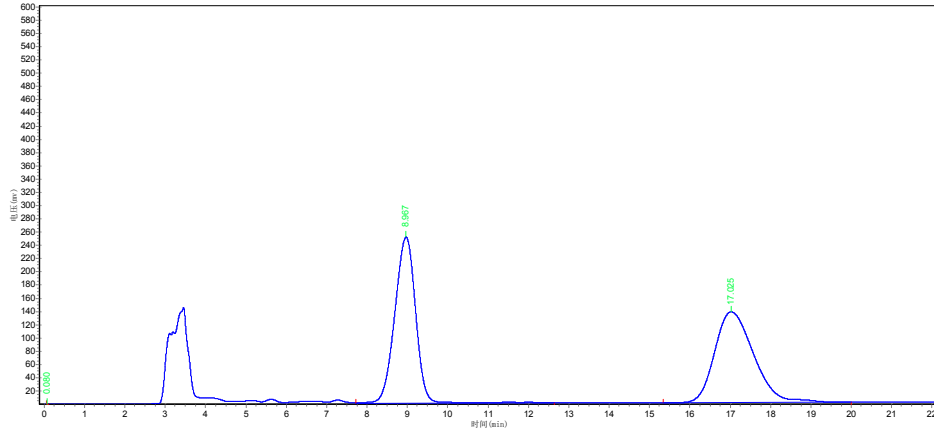


Peak	Ret. Time	Height	Area	Area (%)
1	31.788	42256.422	3509513.750	99.7413
2	36.422	164.674	9098.601	0.2587
Totals				100

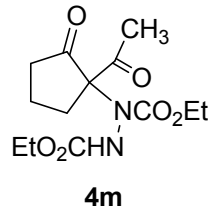


41

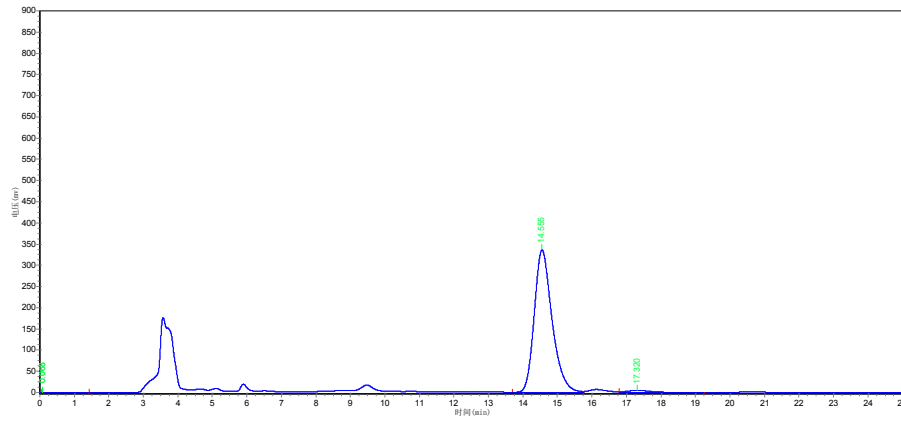
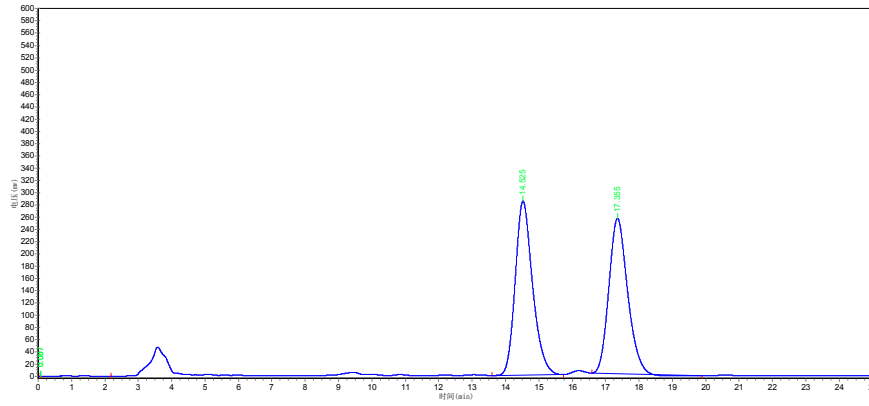
Chiralpak AD-H, 15% *i*PrOH in hexanes, 1.0 mL/min, 210nm



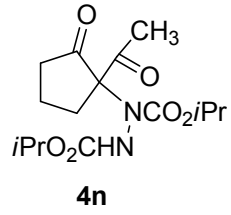
Peak	Ret. Time	Height	Area	Area (%)
1	9.298	220128.219	6158082.000	99.5952
2	13.065	628.000	25028.645	0.4048
Totals				100



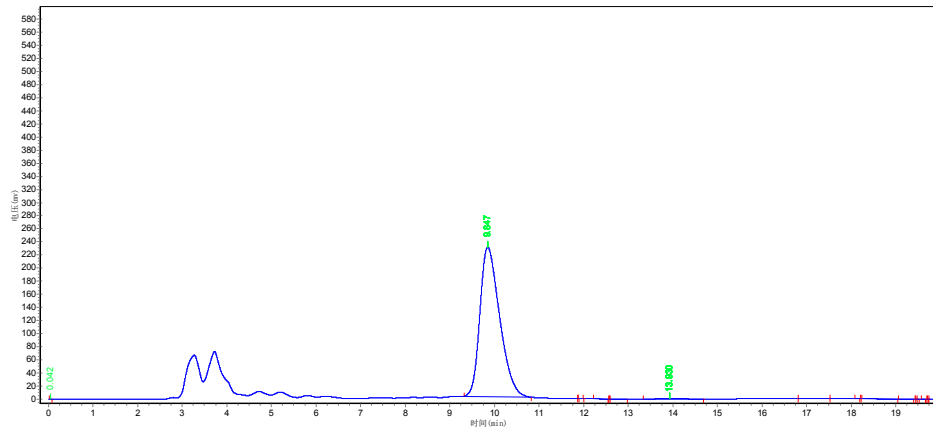
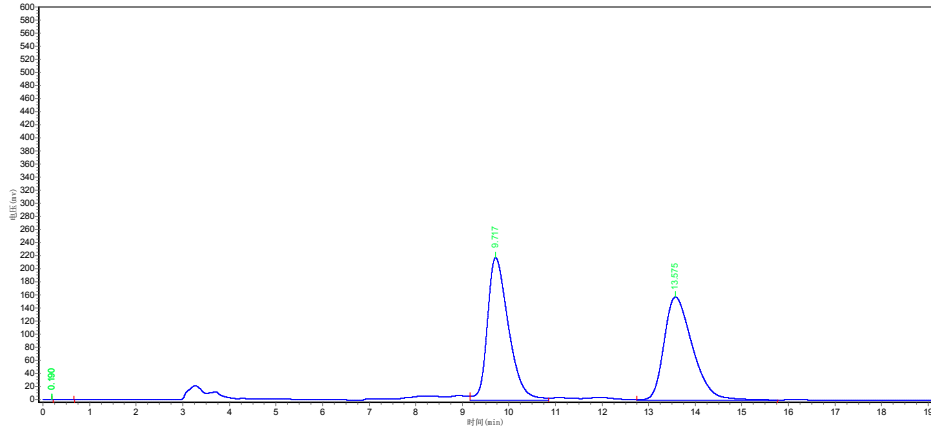
Chiralpak AD-H, 10% *i*PrOH/hexane, 1.0 mL/min, 210 nm.



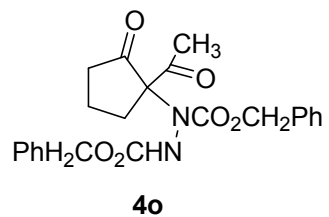
Peak	Ret. Time	Height	Area	Area (%)
1	14.555	336291.156	12100342.000	97.6553
2	17.320	5376.249	290526.781	2.3447
Totals				100



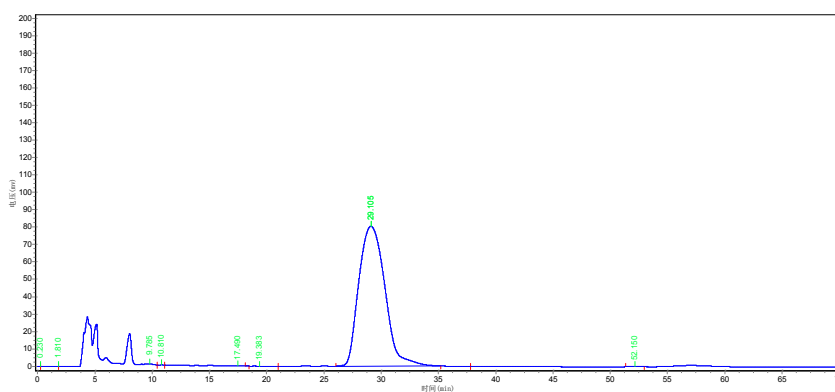
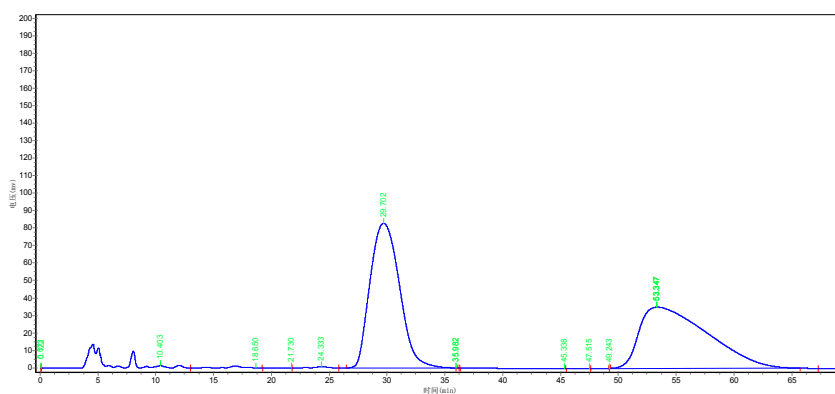
Chiralpak AD-H column, 10% *i*PrOH/hexane; 1.0 mL/min, 210 nm



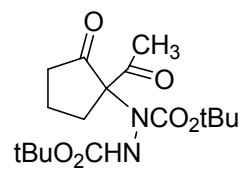
Peak	Ret. Time	Height	Area	Area(%)
1	9.847	227896.219	6987855.000	99.4007
2	13.930	1141.178	41706.027	0.5993



Chiralpak AS-H column, 30% *i*PrOH/hexane; 0.8 mL/min, 254 nm;

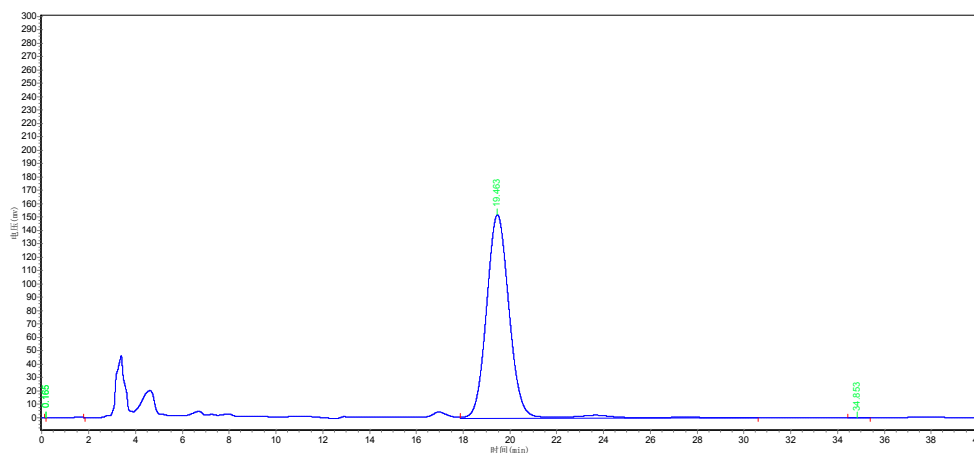
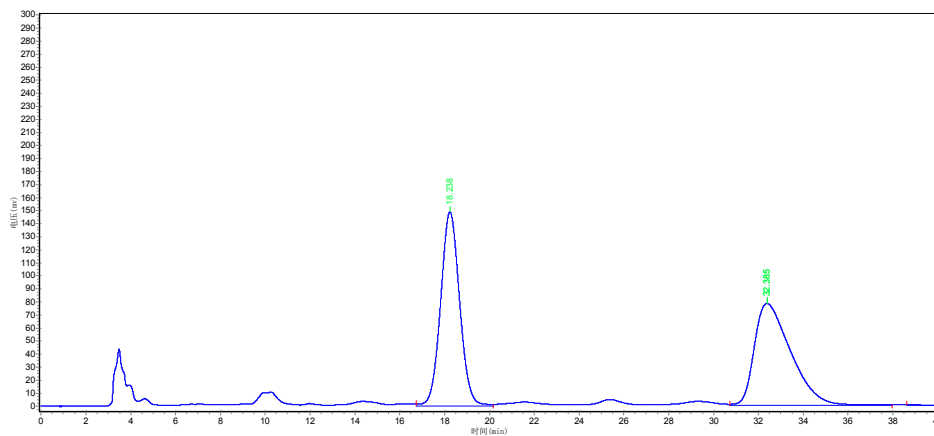


Peak	Ret. Time	Height	Area	Area (%)
1	29.105	80422.070	12738561.000	99.9841
2	52.150	37.029	2040.302	0.0159
Totals				100

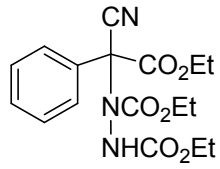


4p

Chiralpak AD-H column, 5% *i*PrOH/hexane; 1.0 mL/min, 210 nm;

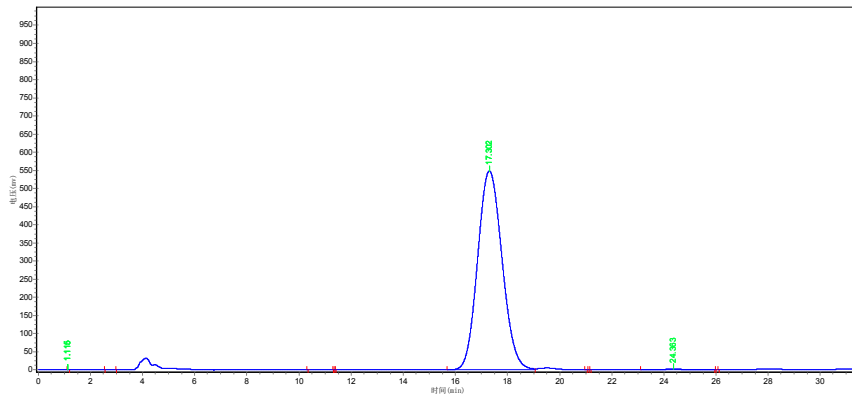
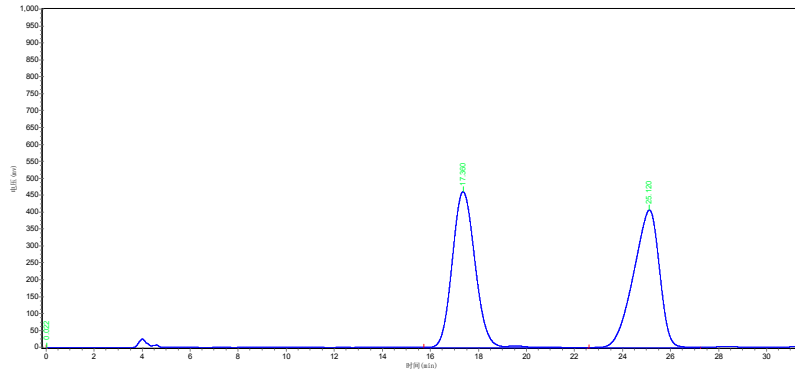


Peak	Ret. Time	Height	Area	Area (%)
1	19.463	152312.531	10426240.000	99.9921
2	34.853	40.518	818.350	0.0079
Totals				100

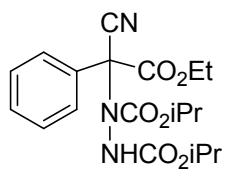


4q

Chiralpak AD-H column, 15% *i*PrOH/hexane; 0.8 mL/min, 210 nm;

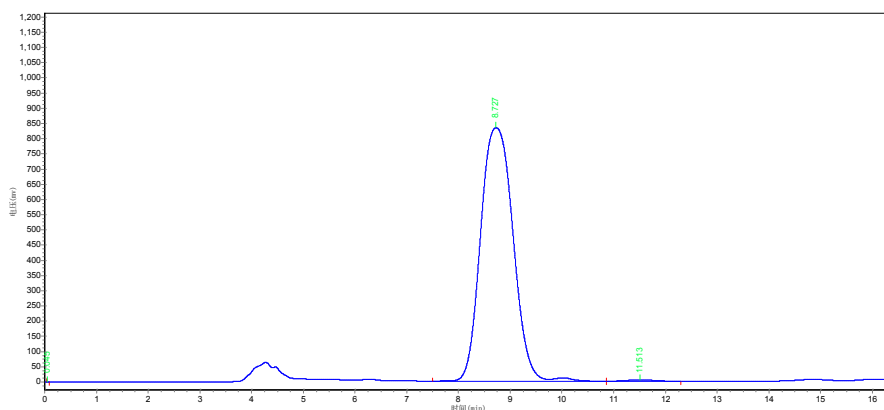
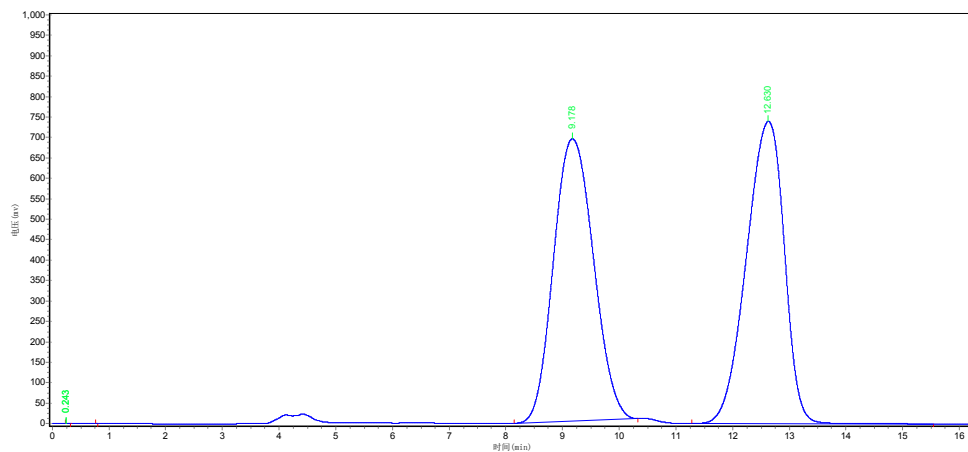


Peak	Ret. Time	Height	Area	Area (%)
1	17.302	548362.813	35705236.000	99.6054
2	24.363	2084.014	141431.109	0.3946
Totals				100

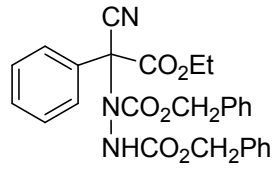


4r

Chiralpak AD-H column, 30% *i*PrOH/hexane; 0.8 mL/min, 210 nm;

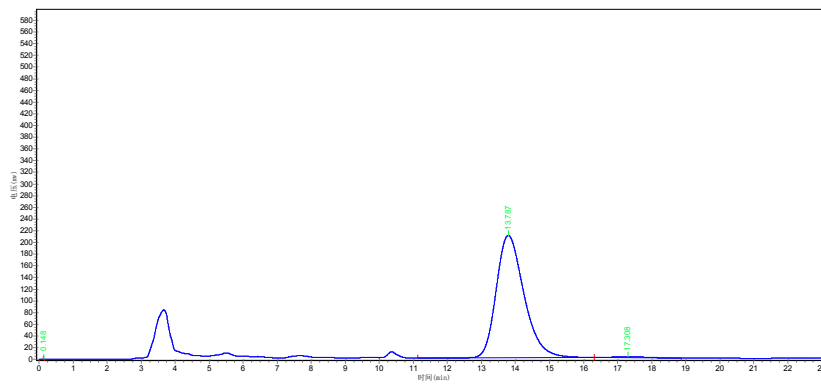
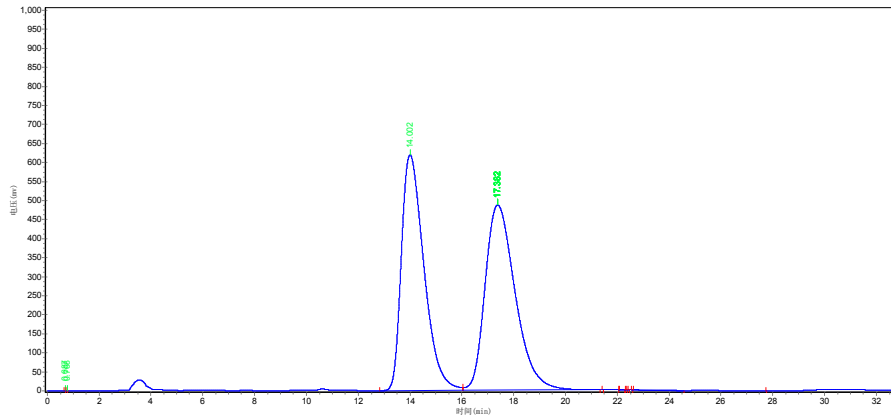


Peak	Ret. Time	Height	Area	Area (%)
1	8.727	834145.250	36441900.000	99.6072
2	11.513	4318.502	143692.422	0.3928
Totals				100

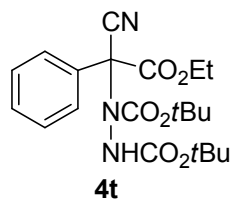


4s

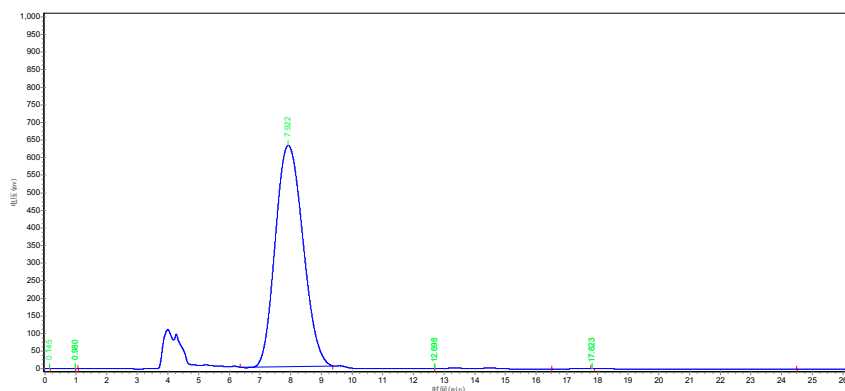
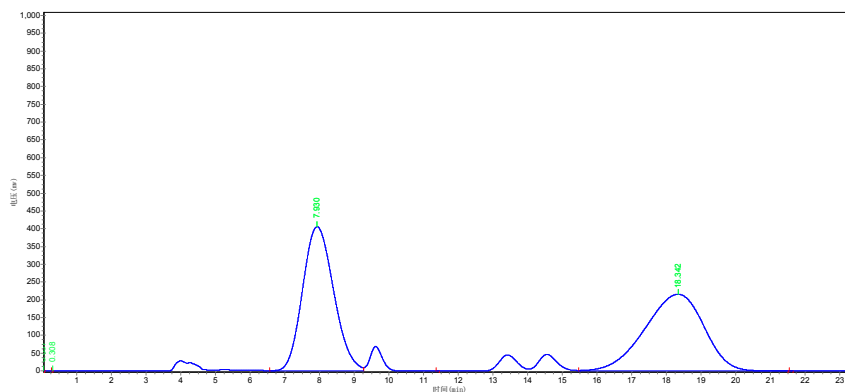
Chiralpak OD-H column, 15% *i*PrOH/hexane; 1.0 mL/min, 210 nm;



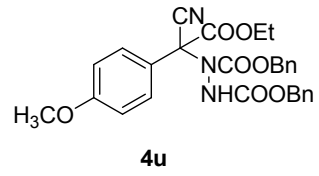
Peak	Ret. Time	Height	Area	Area (%)
1	13.787	209345.875	11879337.000	99.0442
2	17.308	1766.343	114638.984	0.9558
Totals				100



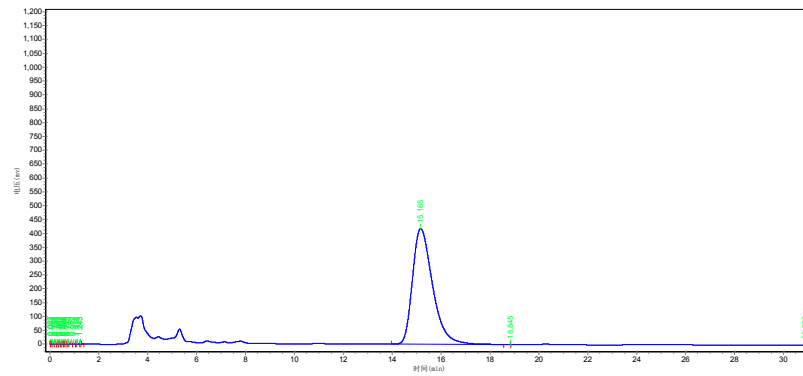
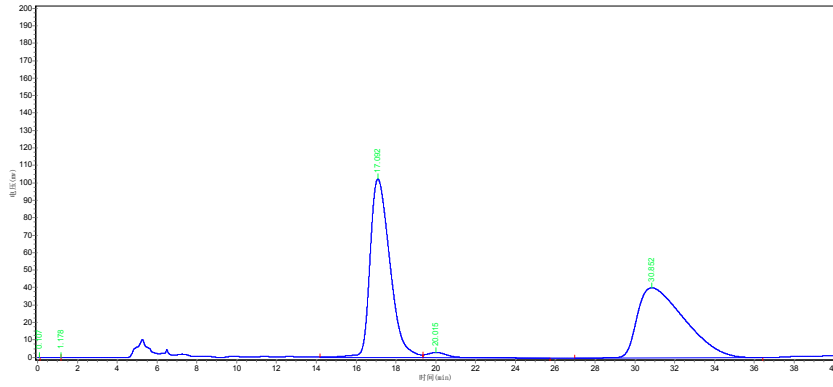
Chiralpak AD-H column, 30% *i*PrOH/hexane; 0.8 mL/min, 210 nm;



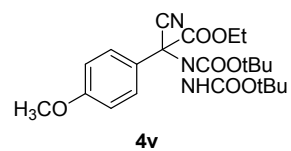
Peak	Ret. Time	Height	Area	Area (%)
1	7.922	628261.063	39305756.000	99.9005
2	17.823	447.651	0.498	0.0995
Totals				100



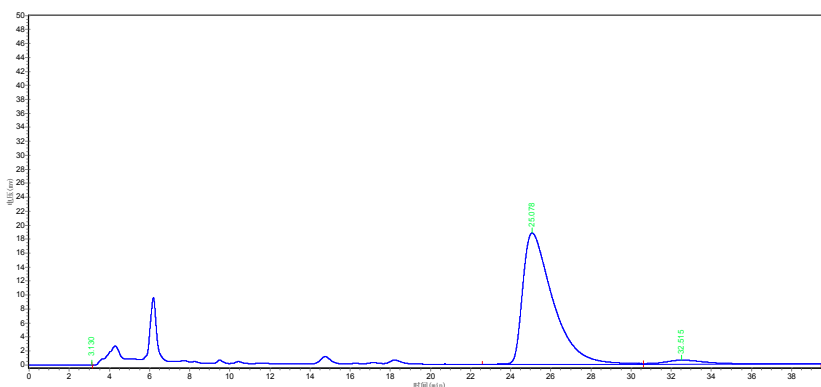
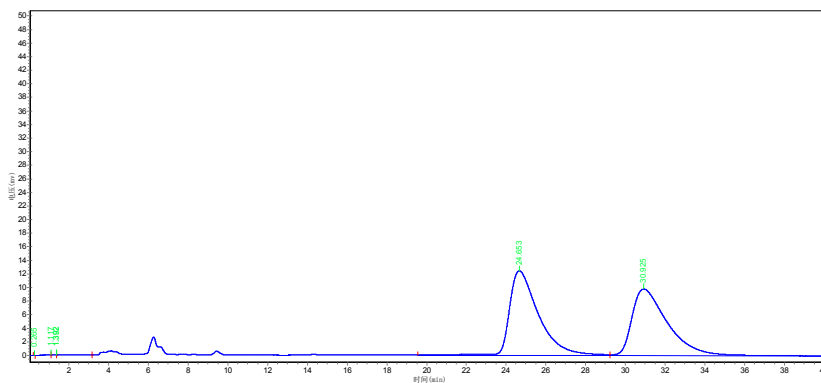
Chiralpak OD-H column, 20% *i*PrOH/hexane; 0.8 mL/min, 254 nm;



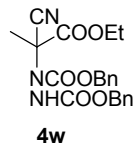
Peak	Ret. Time	Height	Area	Area (%)
1	15.165	417572.906	23856046.000	99.0904
2	18.845	4.4248	4.903	0.9096
Totals				100



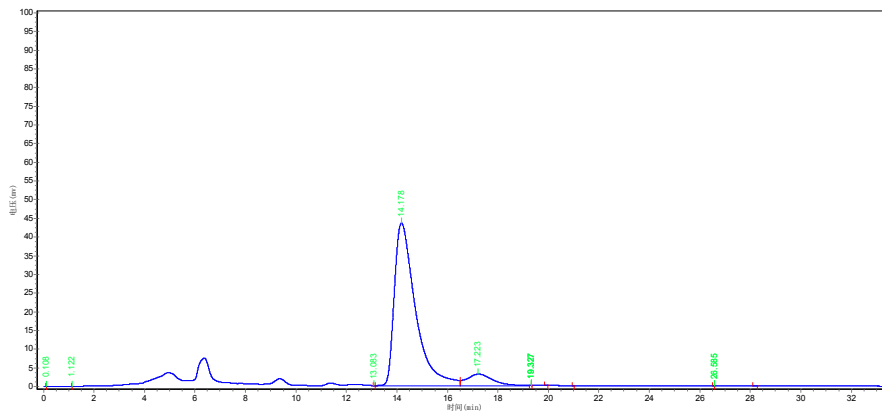
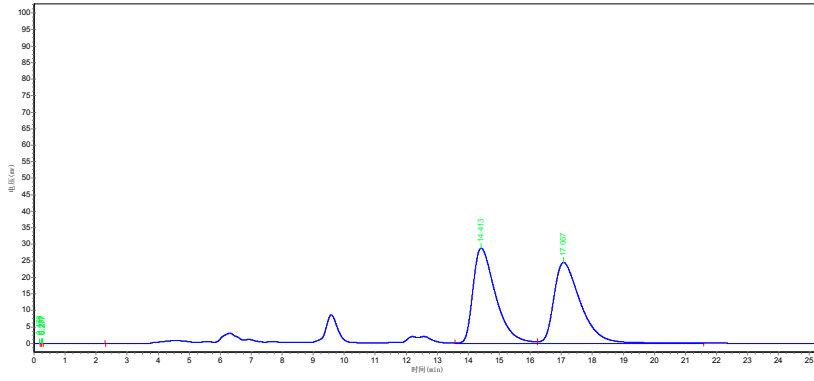
Chiralpak OD-H column, 15% *i*PrOH/hexane; 0.8 mL/min, 254 nm;



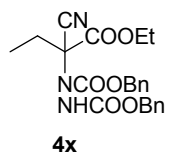
Peak	Ret. Time	Height	Area	Area (%)
1	25.078	18820.006	1970695.500	95.2888
2	32.515	595.434	97450.000	4.71125
Totals				100



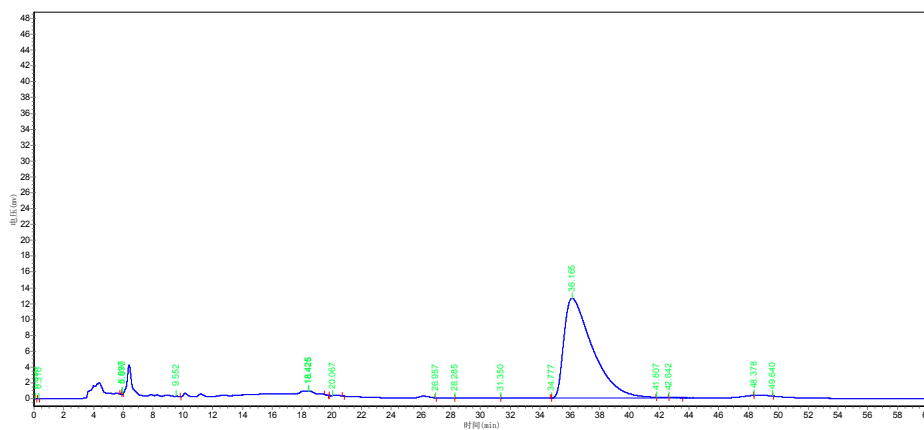
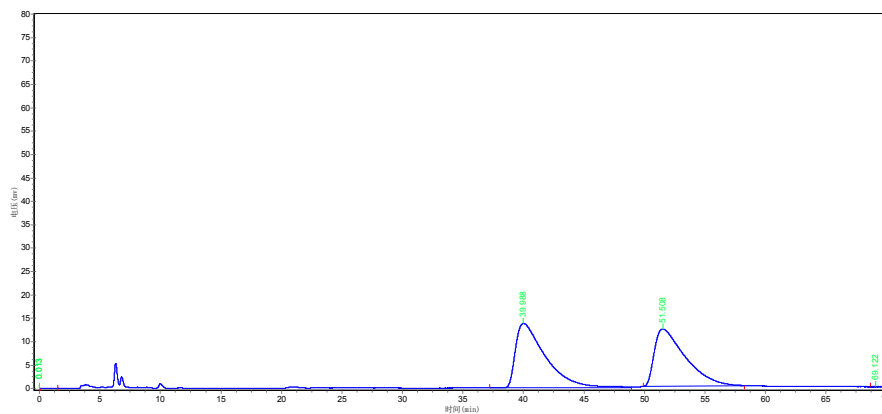
Chiralpak OD-H column, 15% *i*PrOH/hexane; 0.8 mL/min, 254 nm;



Peak	Ret. Time	Height	Area	Area (%)
1	14.178	43580.777	2525939.000	96.0117
2	17.223	2062.153	108321.703	3.9883
Totals				100



Chiralpak AS-H column, 20% *i*PrOH/hexane; 0.8 mL/min, 254 nm;



Peak	Ret. Time	Height	Area	Area (%)
1	36.165	12598.615	1682796.125	99.9827
2	48.025	43.251	290.126	0.0173
Totals				100