

Supplementary Material (ESI) for RSC Advances

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**Total Synthesis of Phenanthroindolizidine Alkaloids via Asymmetric
Deprotonation of *N*-Boc-pyrrolidine**

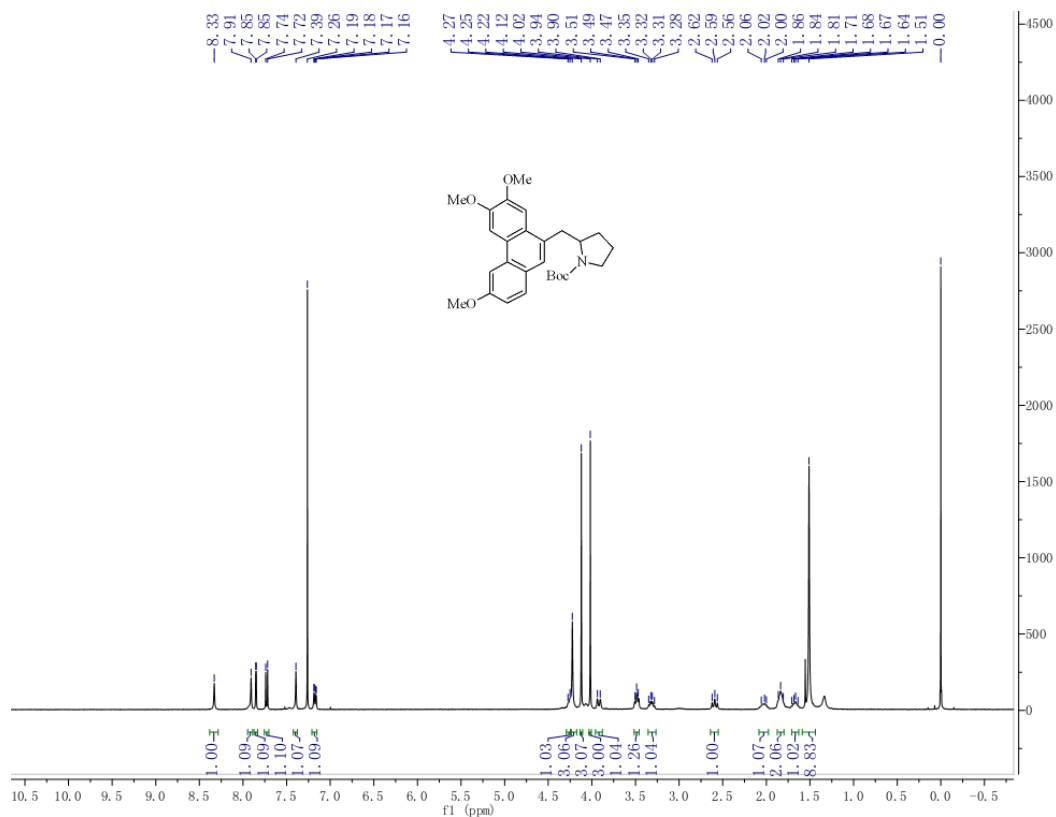
Meng Deng, Bo Su, Hui Zhang, Yuxiu Liu, and Qingmin Wang*

*State Key Laboratory of Elemento-Organic Chemistry, Research Institute of Elemento-Organic Chemistry, Nankai
University, Collaborative Innovation Center of Chemical Science and Engineering(Tianjin), Tianjin 300071,
People's Republic of China
wang98h@263.net; wangqm@nankai.edu.cn*

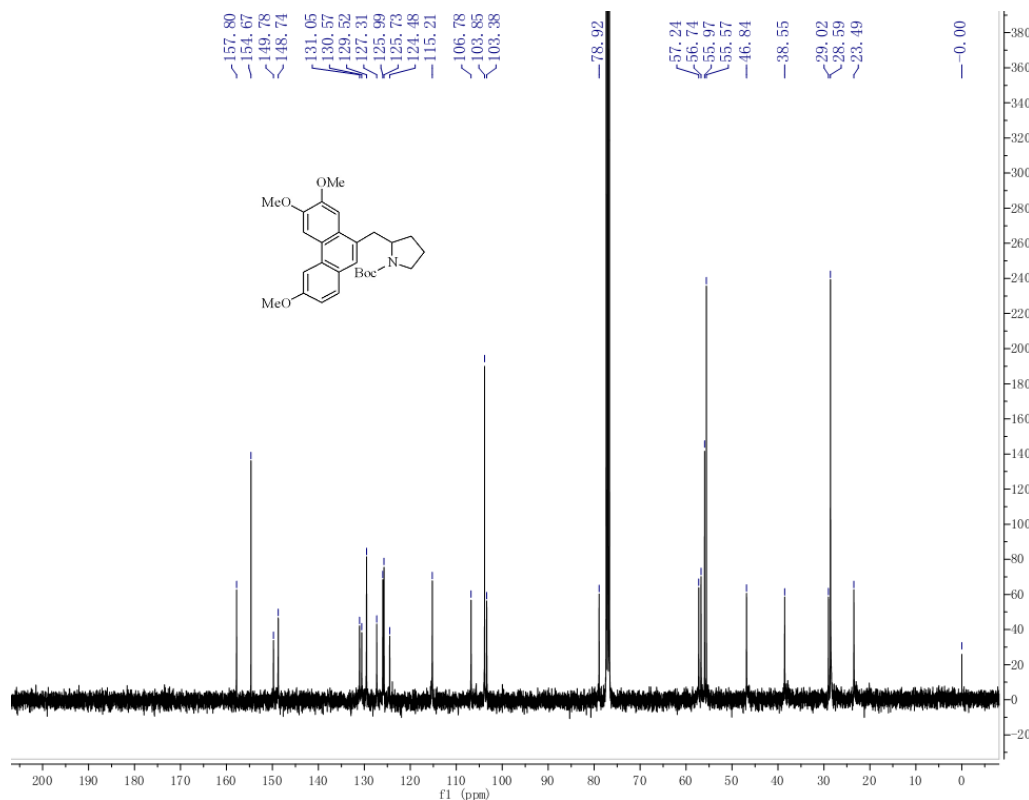
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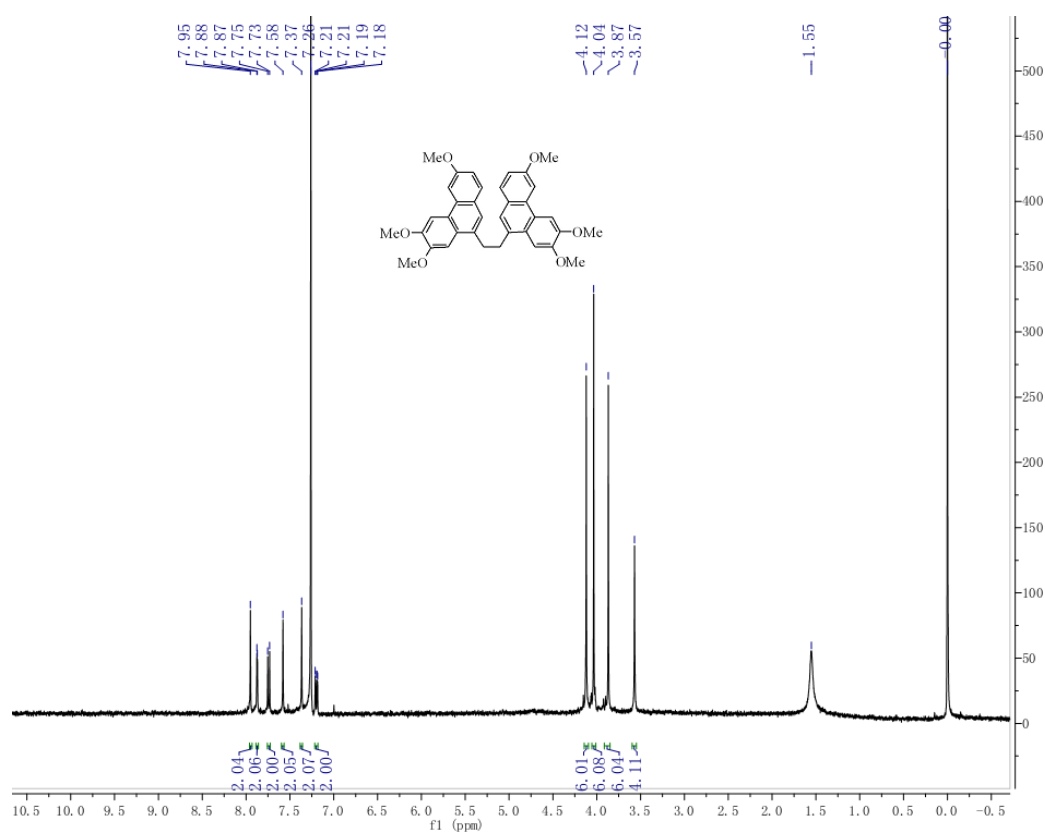
NMR spectra (400 MHz, CDCl₃, 25 °C)



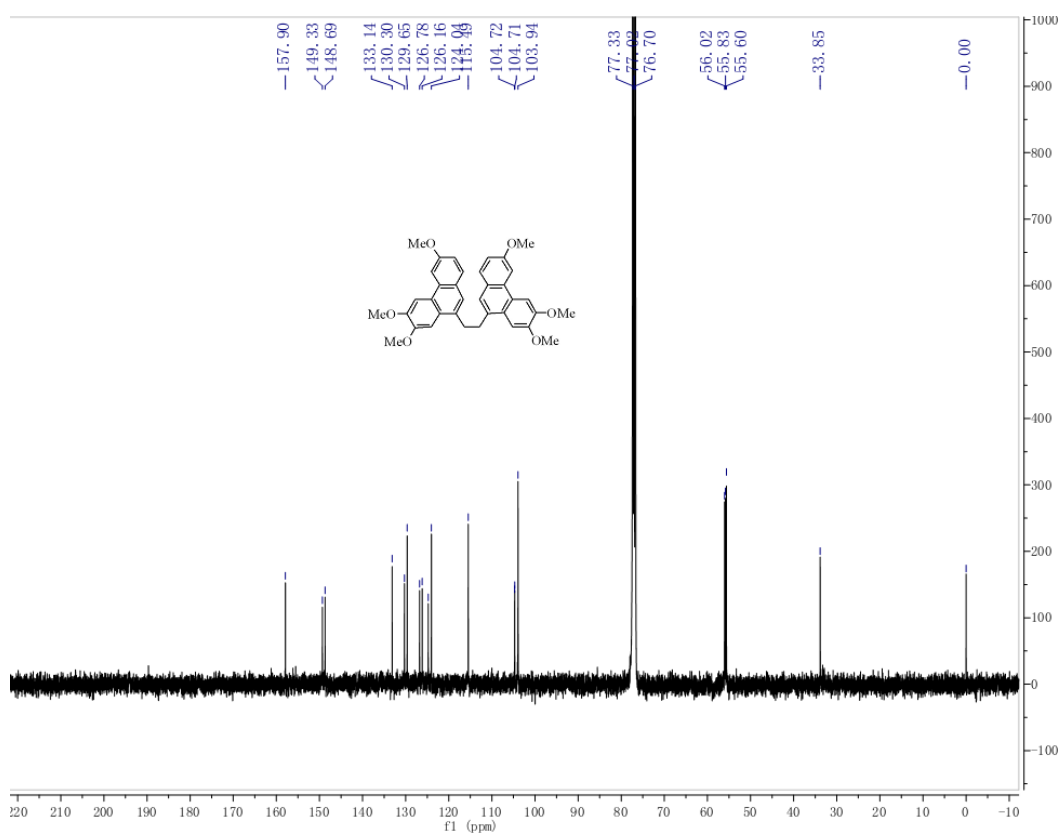
¹H NMR of compound 5



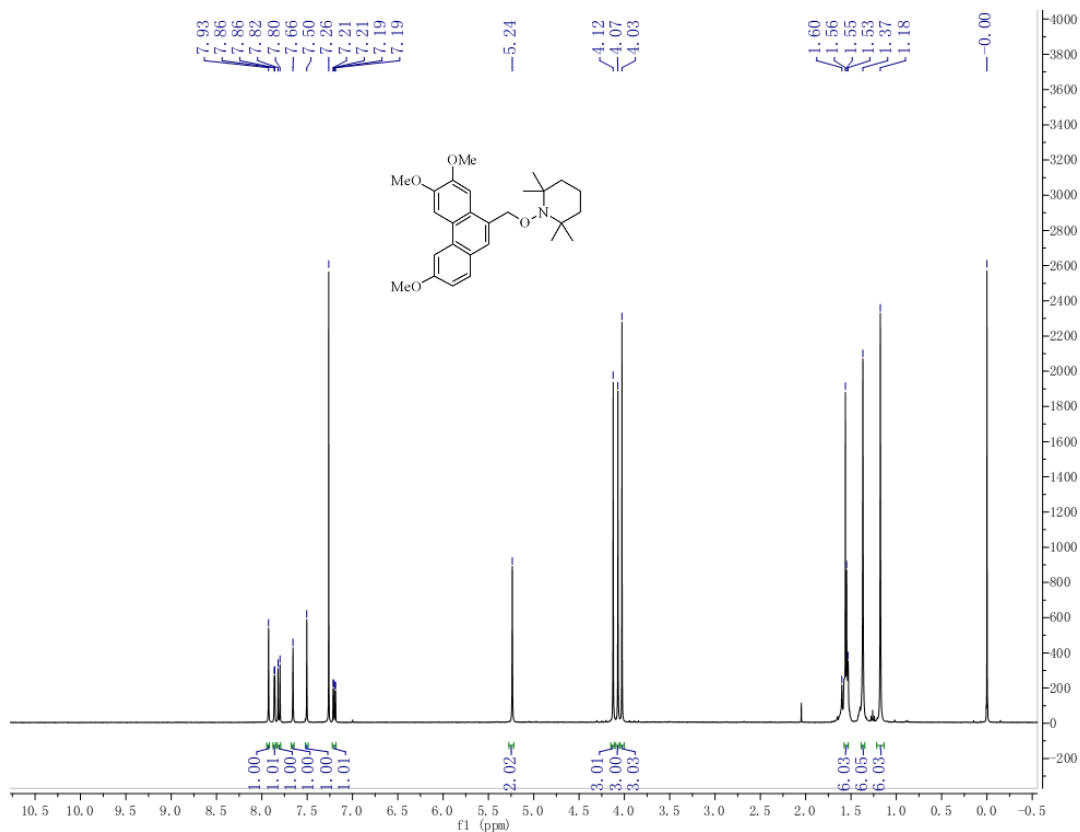
¹³C NMR of compound 5



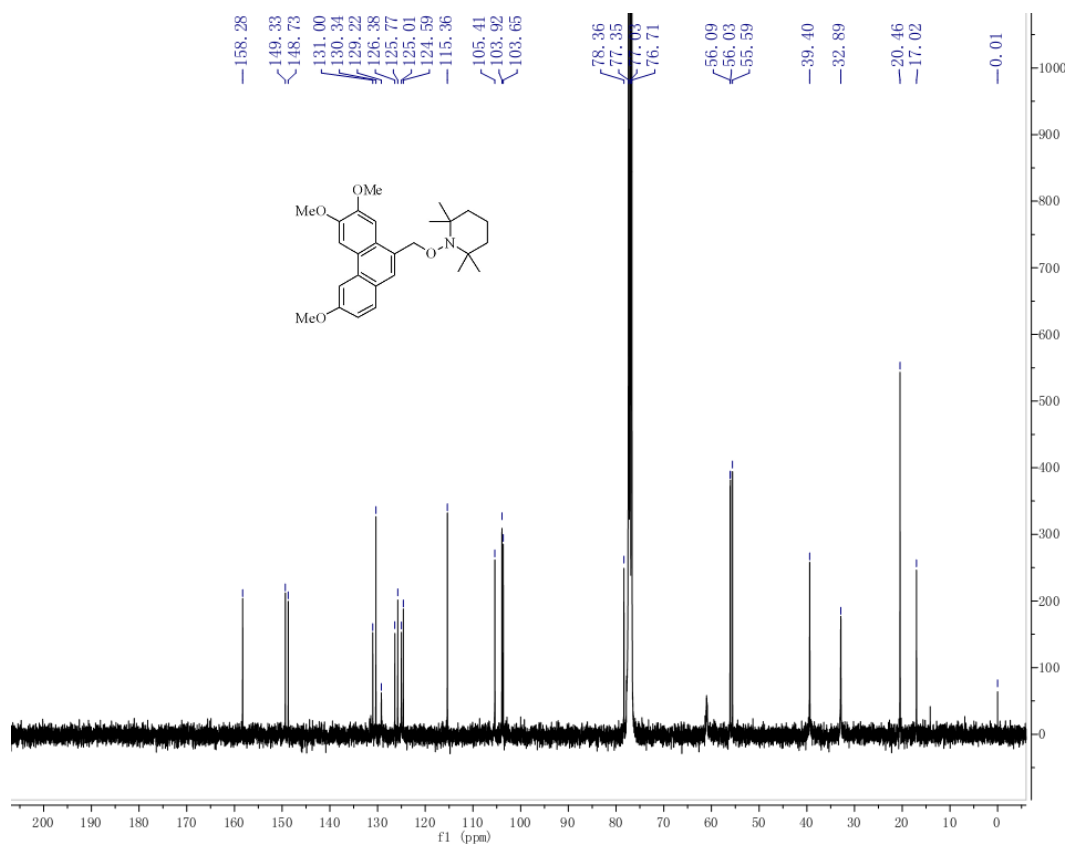
¹H NMR of compound 5a



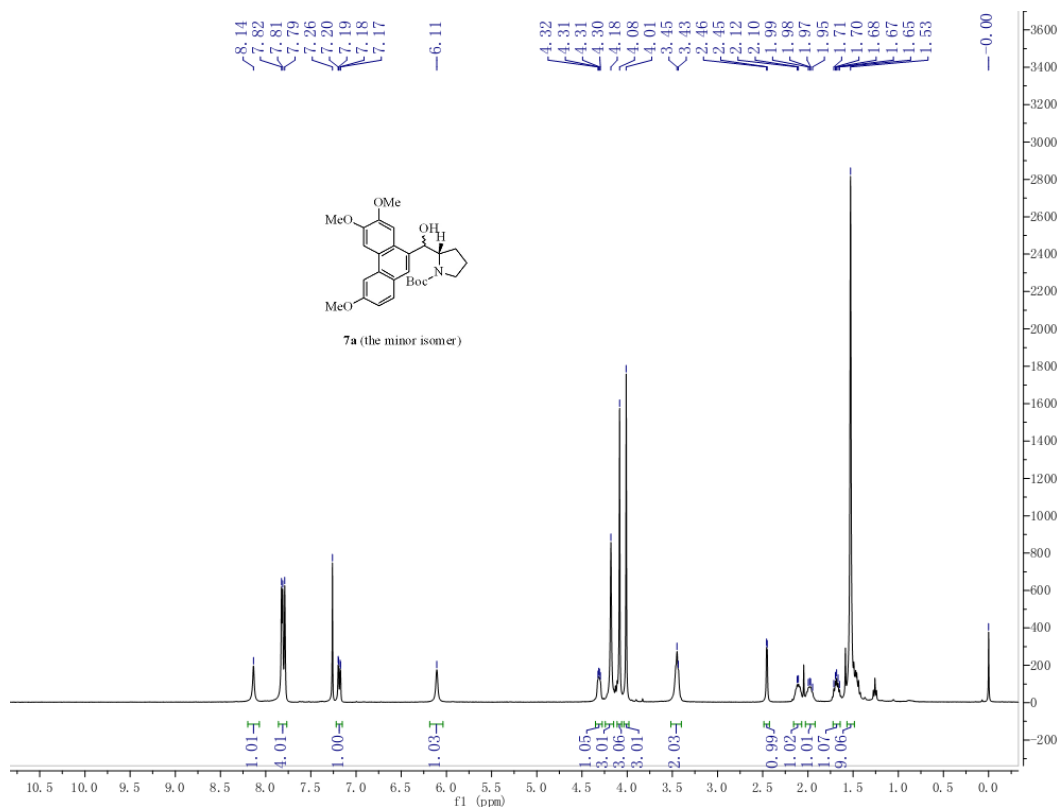
¹³C NMR of compound 5a



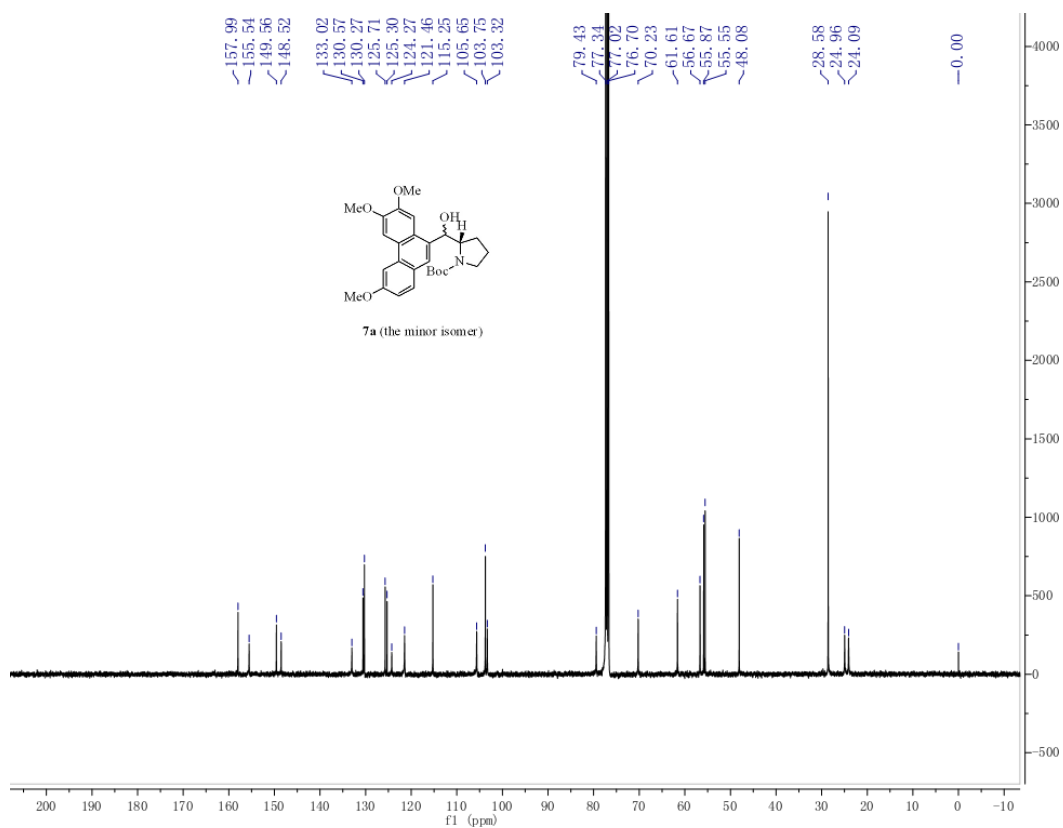
¹H NMR of compound 4c



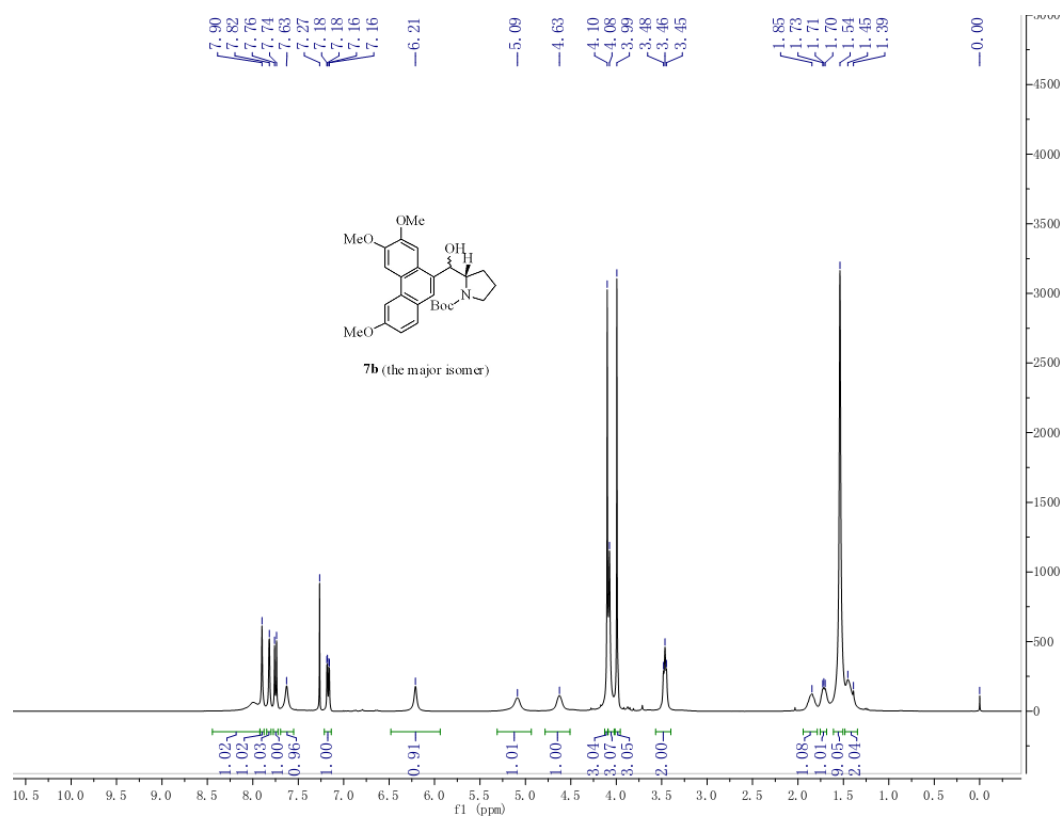
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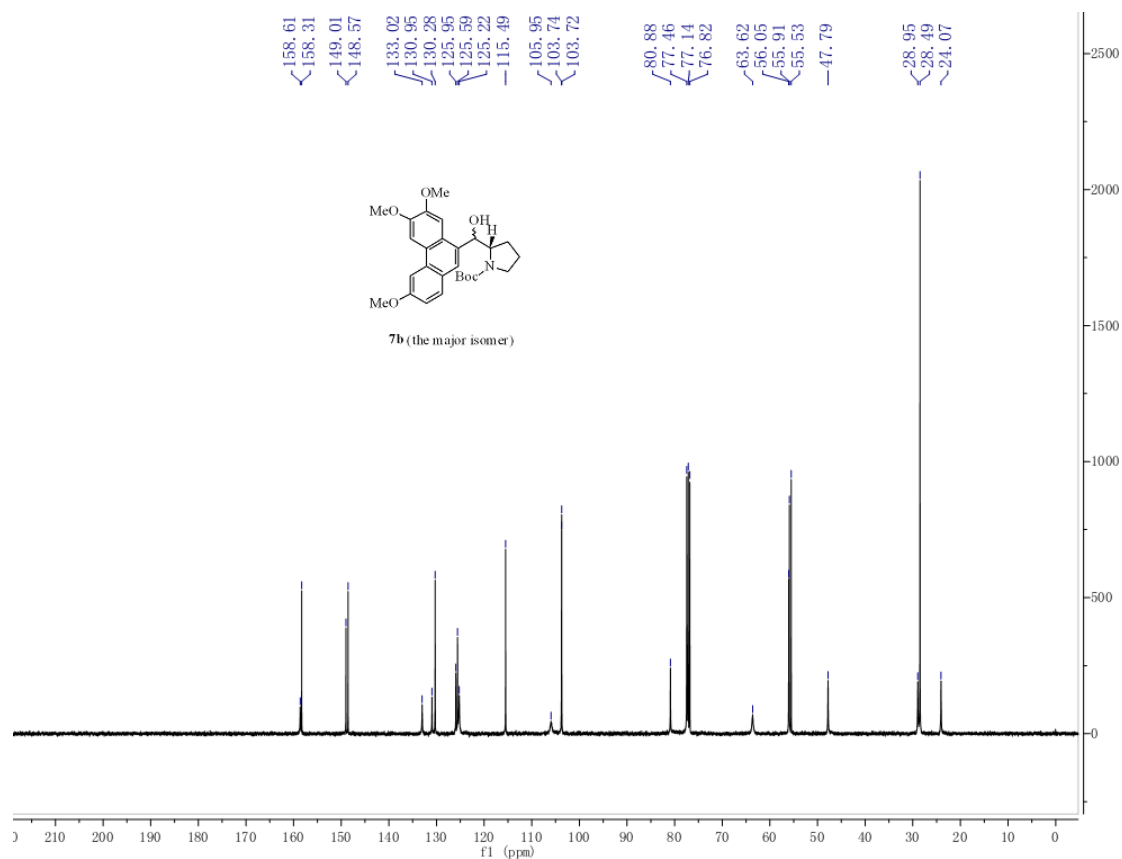
¹H NMR of compound 7a



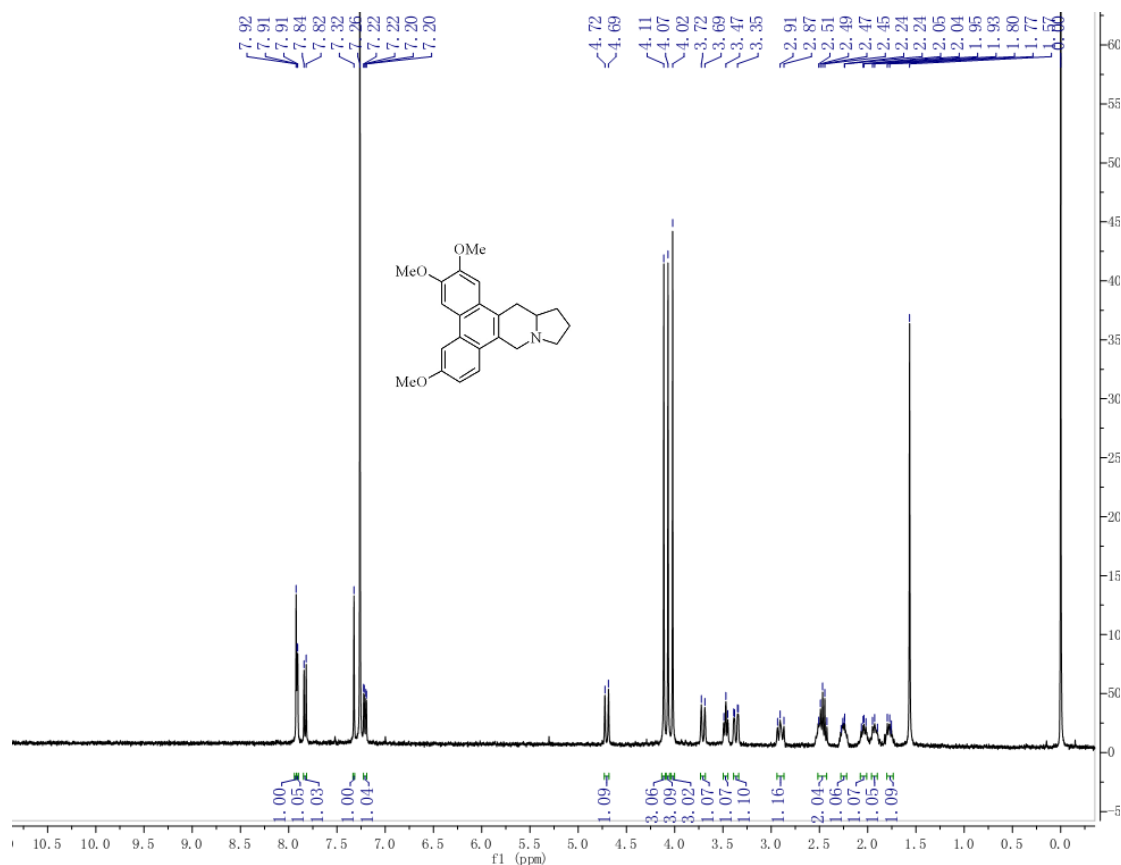
¹³C NMR of compound 7a



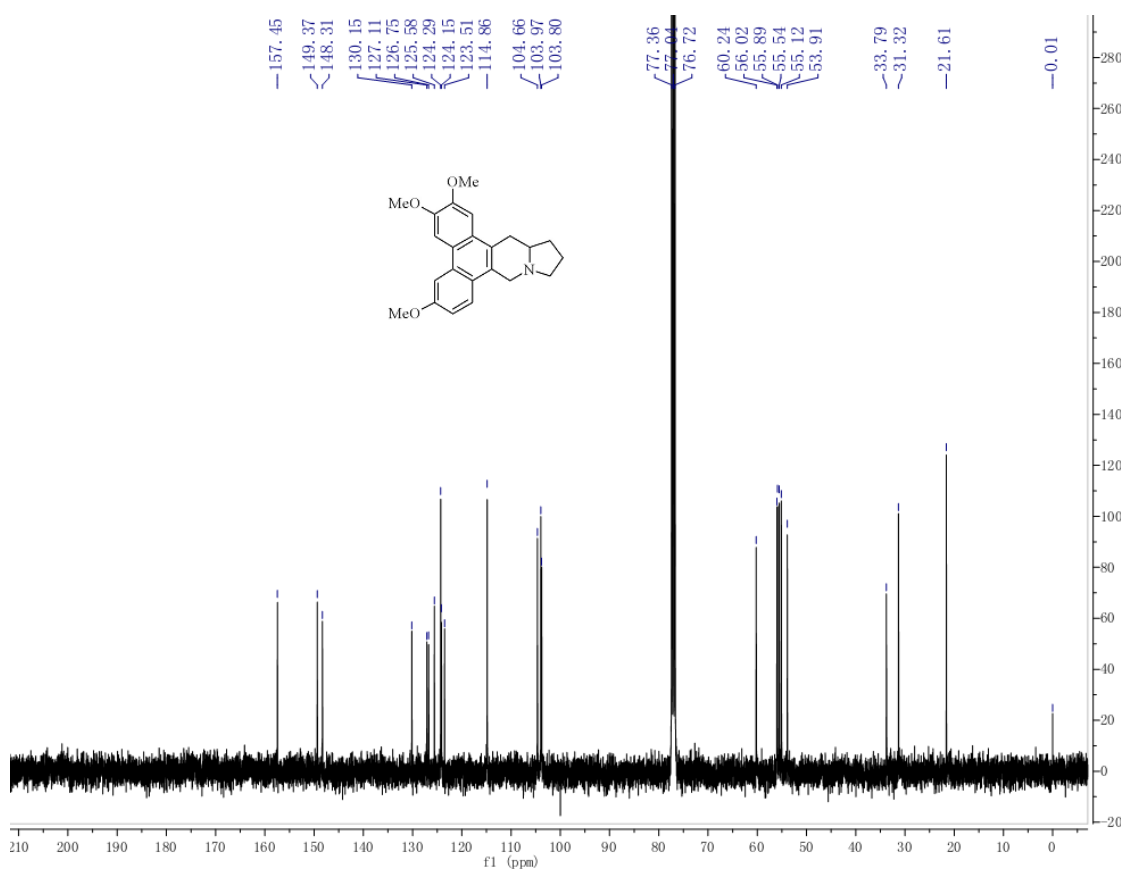
¹H NMR of compound 7b



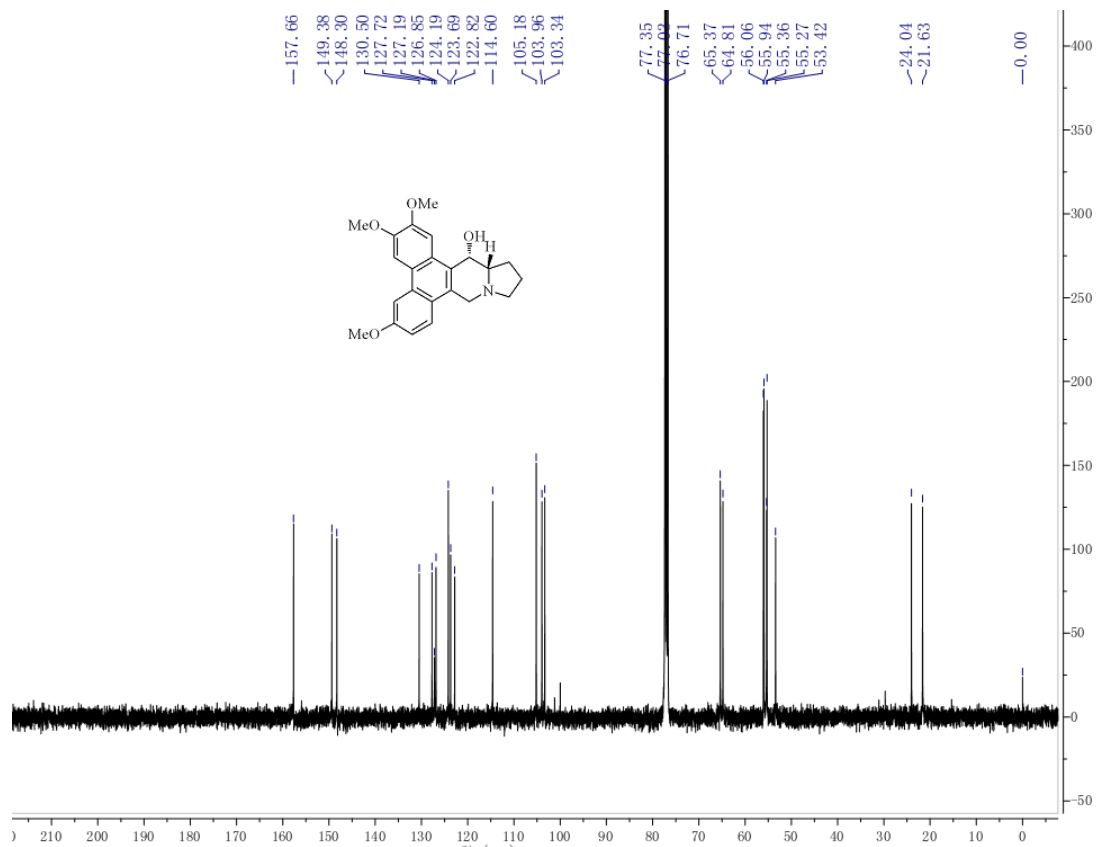
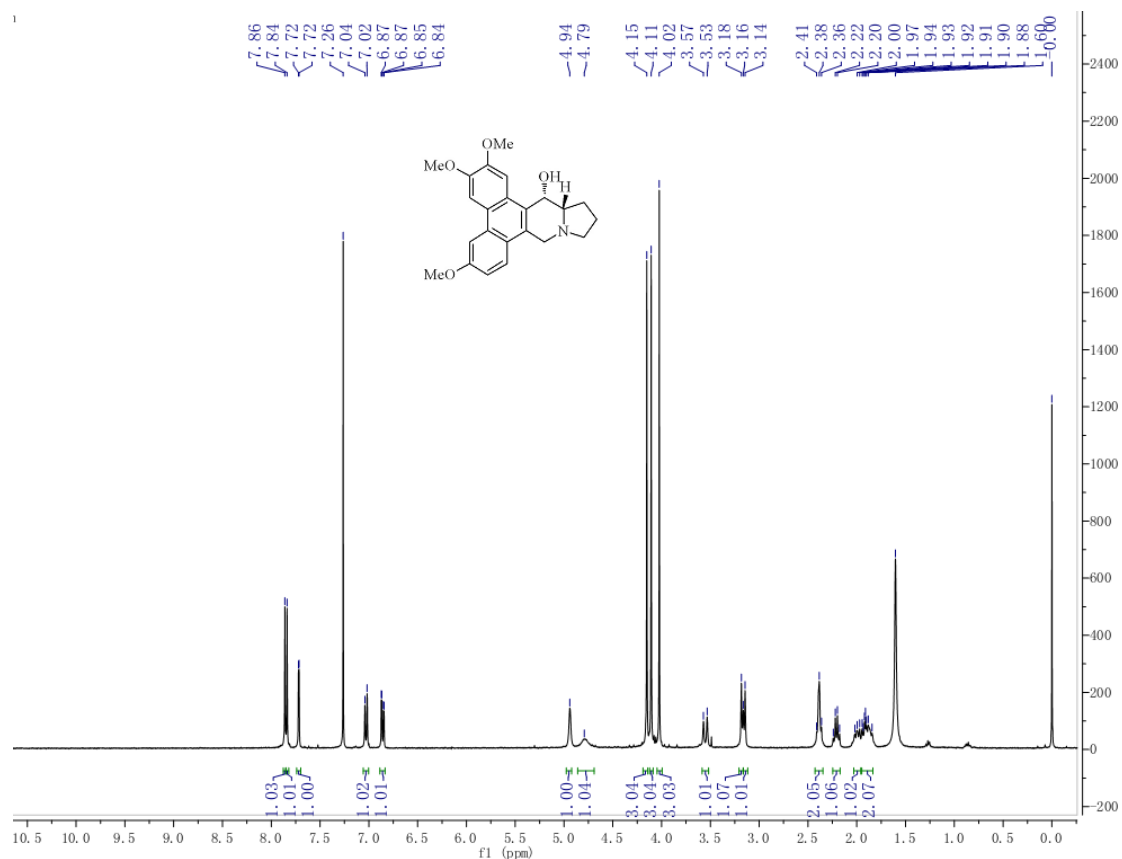
¹³C NMR of compound 7b



¹H NMR of compound 1a

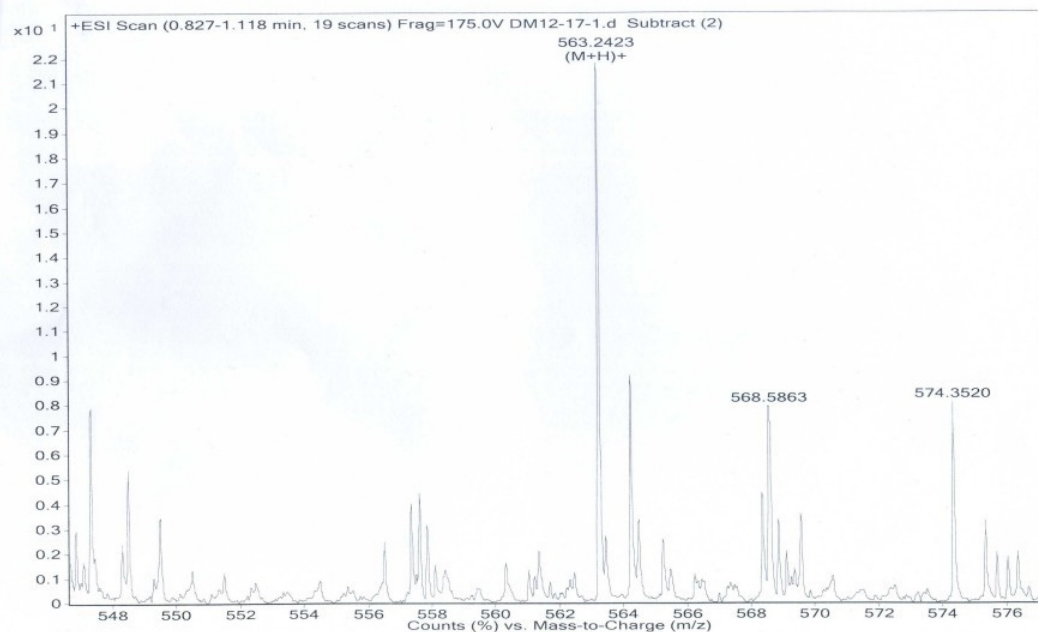


¹³C NMR of compound 1a



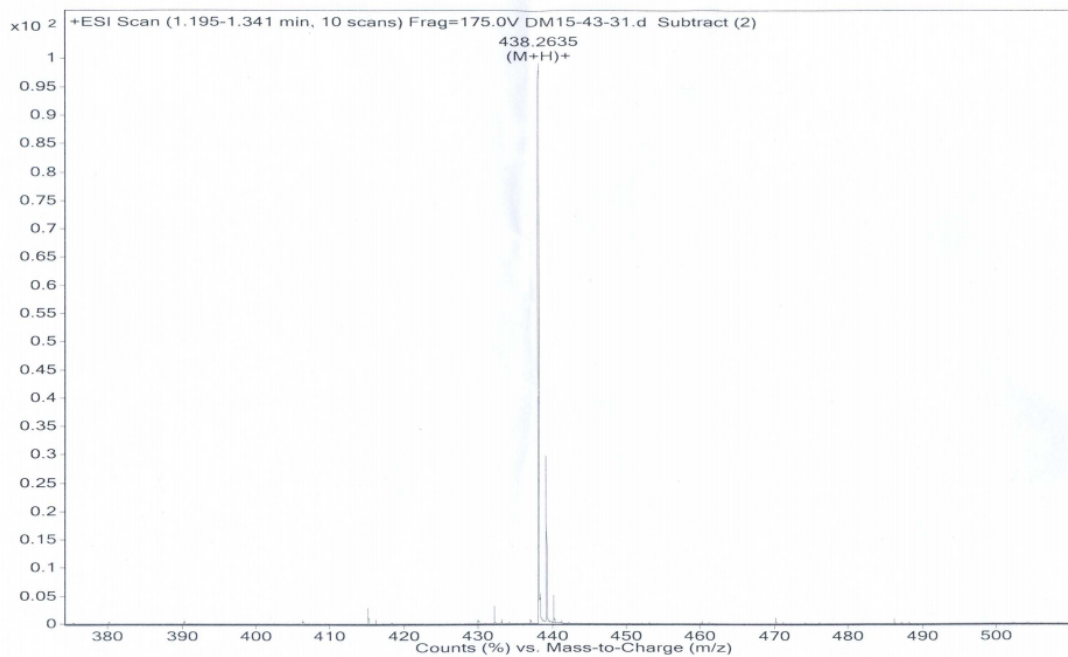
Copies of HRMS (ESI)

Sample Name	lc/ms	Position	P1-A1	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	DM12-17-1.d	ACQ Method	chen-ms.m	Comment		Acquired Time	8/14/2013 9:08:55 AM



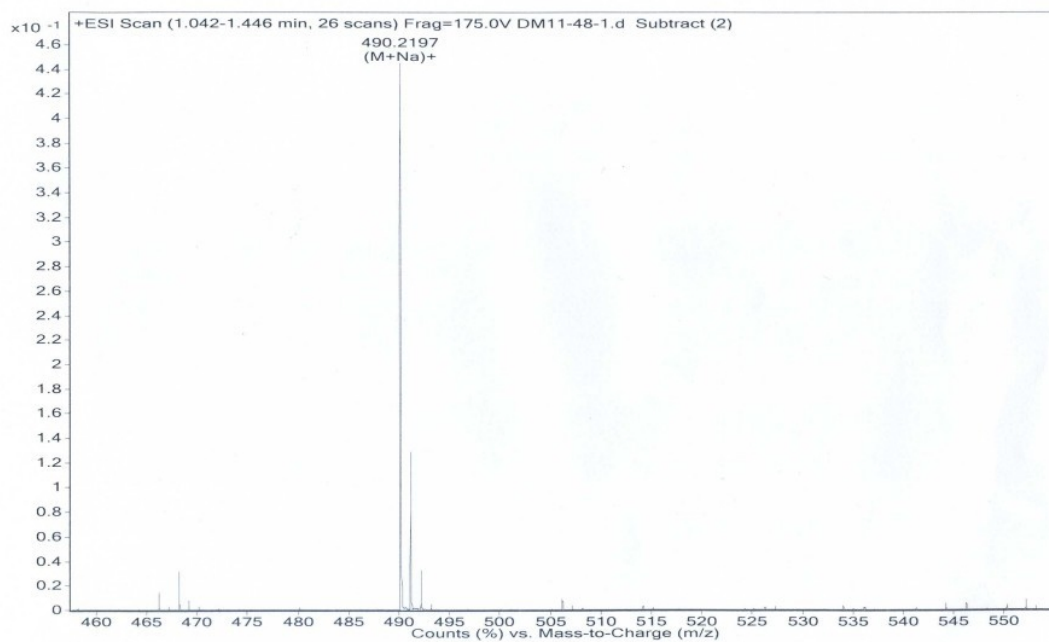
HRMS of compound 5a

Sample Name	lc/ms	Position	P1-A7	Instrument Name	Instrument 1	User Name	
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Data Filename	DM15-43-31.d	ACQ Method	chen-ms.m	Comment		Acquired Time	8/30/2013 11:48:32 AM



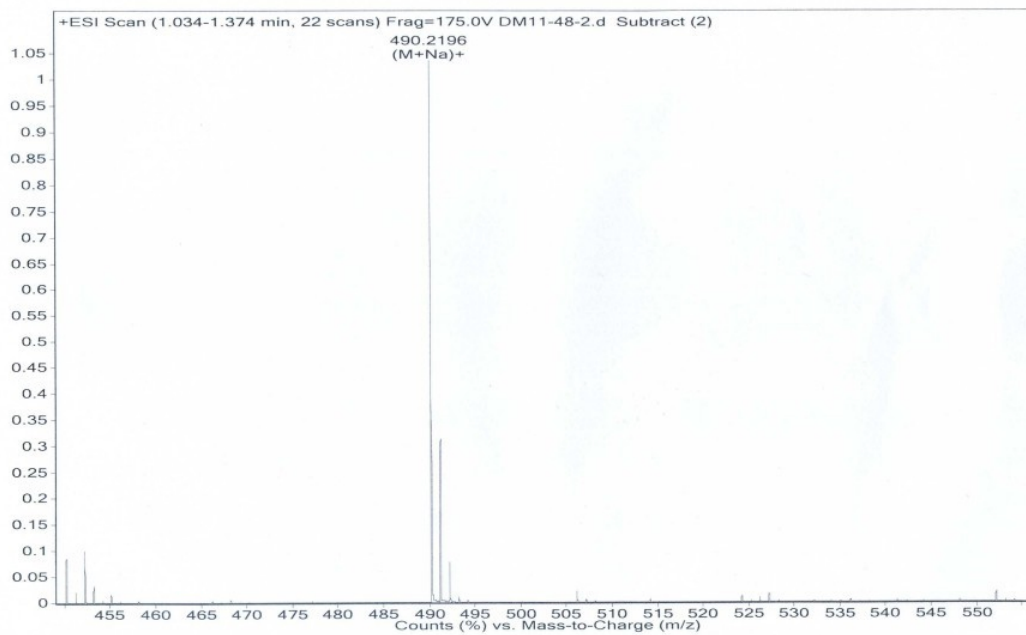
HRMS of compound 4c

Sample Name	LC/MS	Position	P1-A3	Instrument Name	Instrument 1	User Name	
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Data Filename	DM11-48-1.d	ACQ Method	chen-ms.m	Comment		Acquired Time	3/1/2013 11:52:35 AM



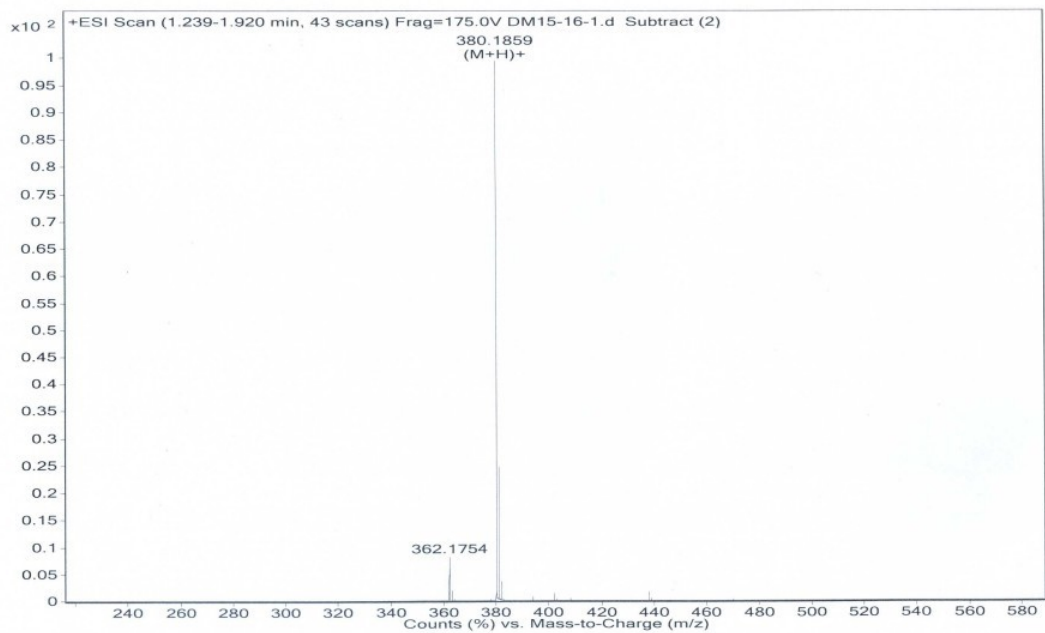
HRMS of compound 7a

Sample Name	LC/MS	Position	P1-A4	Instrument Name	Instrument 1	User Name	
Inj Vol	3	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
Data Filename	DM11-48-2.d	ACQ Method	chen-ms.m	Comment		Acquired Time	3/1/2013 11:56:25 AM



HRMS of compound 7b

Sample Name	lc/ms	Position	P1-A4	Instrument Name	Instrument 1	User Name	
Inj Vol	2	InjPosition		SampleType	Sample	IRM Calibration Status	Some Ions Missed
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HRMS of compound 2

Chiral-HPLC chromatogram

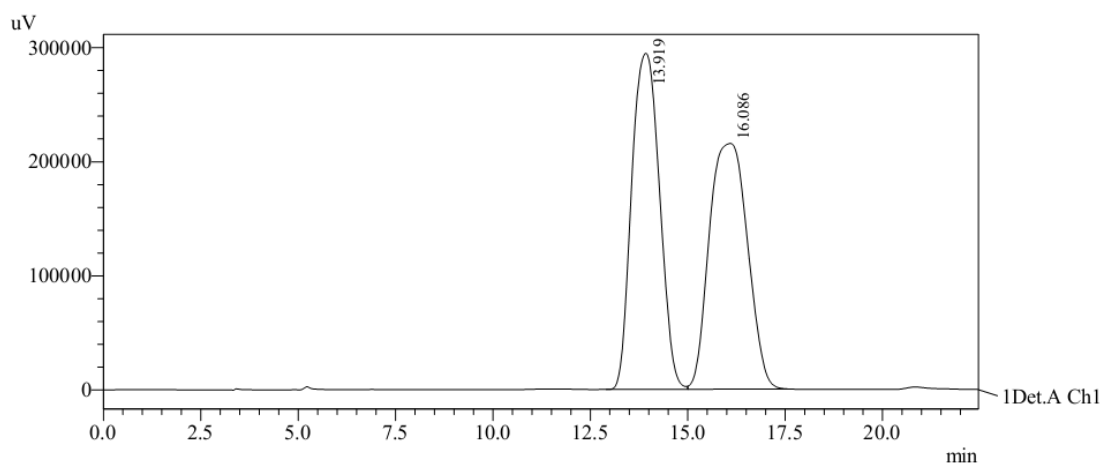
The enantiomeric excesses of (+)-**5** and racemic **5** were determined by HPLC with a Chiralcel AD-H column using Agilent 1100 instrument.

Conditions:

wave length: 254 nm

flow rate: 1.0 mL/ min

mobile phase: i-PrOH : hexane = 10:90



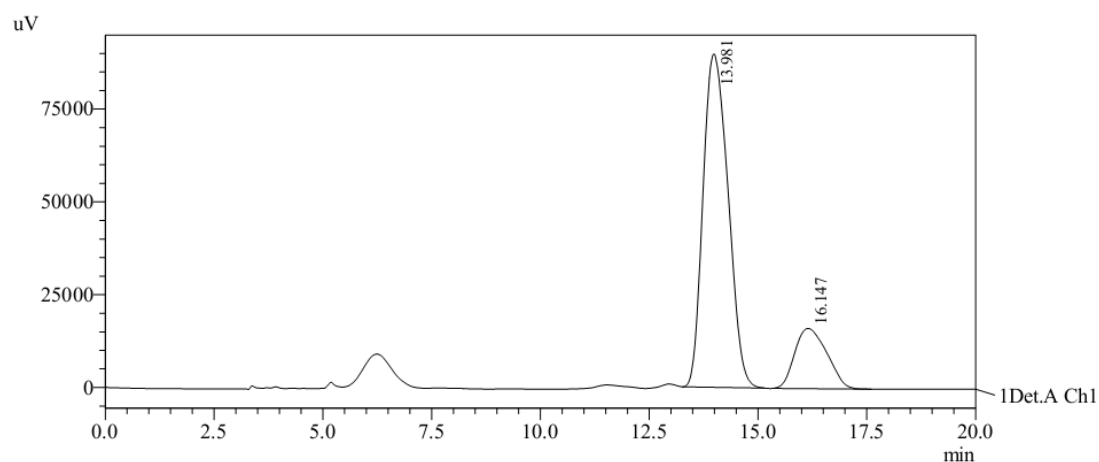
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.919	14685487	294591	49.963	57.781
2	16.086	14707168	215251	50.037	42.219
Total		29392656	509842	100.000	100.000

HPLC chromatogram of racemic **5**



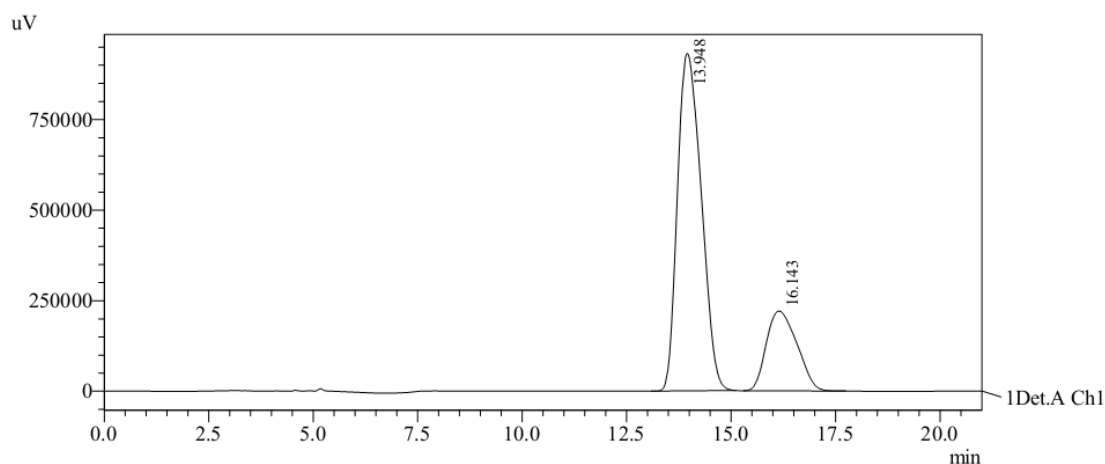
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.981	3596305	89826	81.222	84.711
2	16.147	831453	16213	18.778	15.289
Total		4427758	106039	100.000	100.000

HPLC chromatogram of (+)-**5** (Table 1, Entry 3)



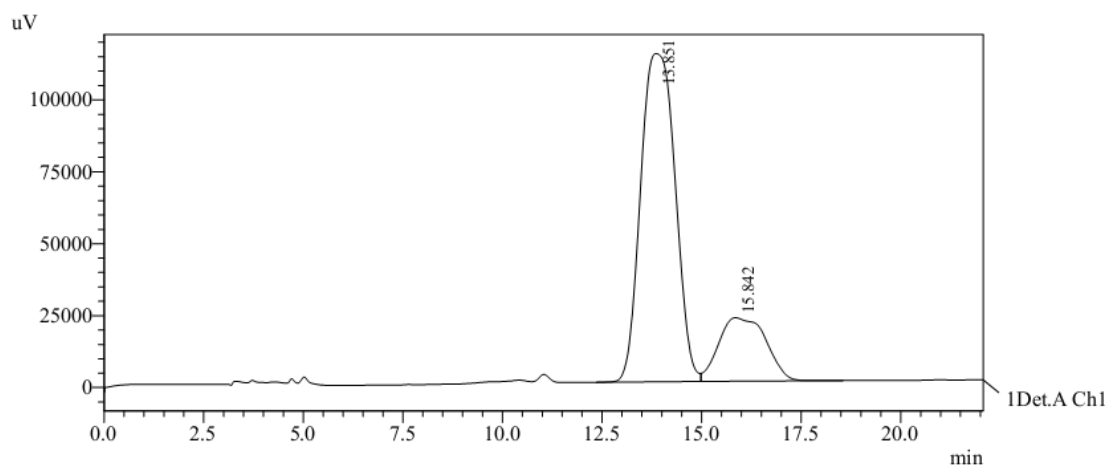
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.948	37055412	931789	76.735	80.867
2	16.143	11234992	220457	23.265	19.133
Total		48290404	1152245	100.000	100.000

HPLC chromatogram of (+)-5 (Table 1, Entry 4)



1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	13.851	6879961	114103	79.064	83.763
2	15.842	1821846	22118	20.936	16.237
Total		8701806	136221	100.000	100.000

HPLC chromatogram of (+)-5 (Table 1, Entry 5)

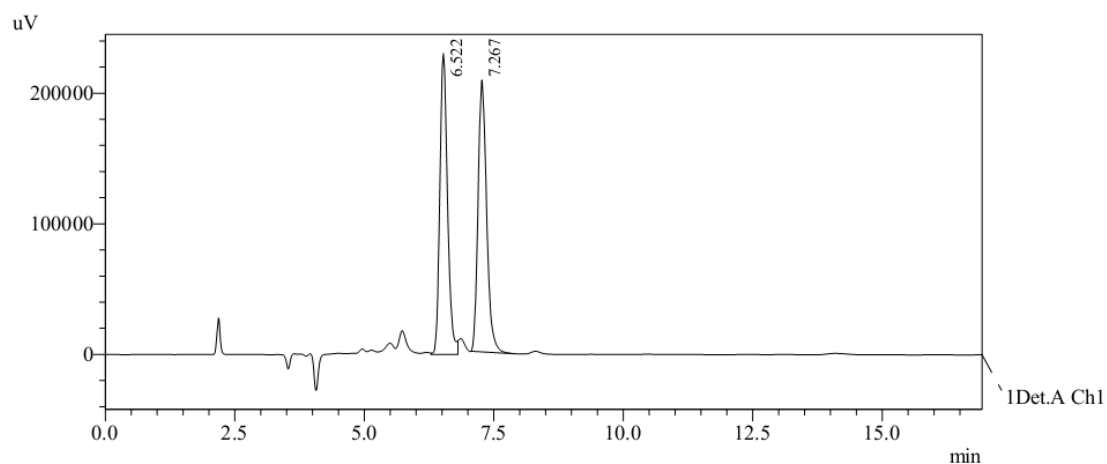
The enantiomeric excesses of **2** and racemic **2** were determined by HPLC with a Phenomenex Lux Cellulose-1 column using Agilent 1100 instrument.

Conditions:

wave length: 254 nm

flow rate: 1.0 mL/ min

mobile phase: i-PrOH: CH₃CN(0.1% Et₃N) = 5:95

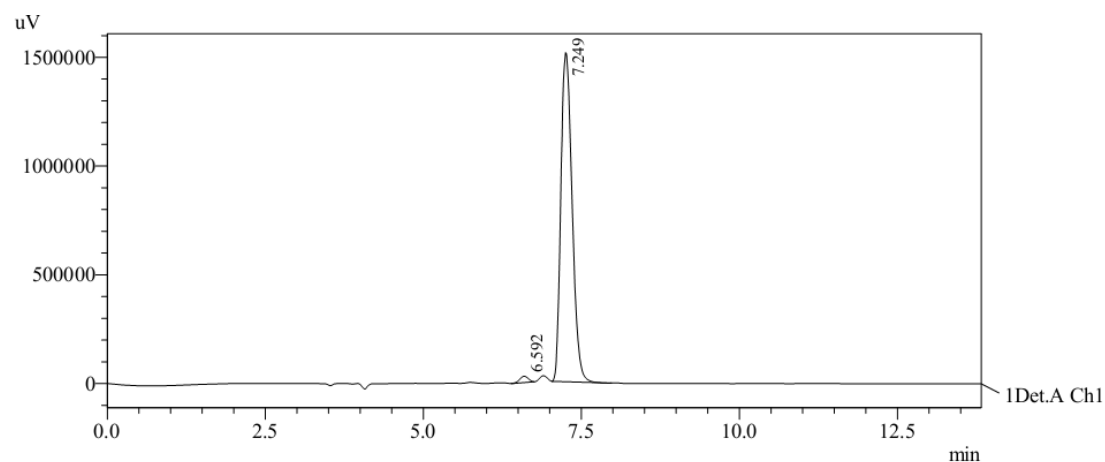


1 Det.A Ch1 / 254nm

PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.522	2378666	230634	49.813	52.575
2	7.267	2396478	208044	50.187	47.425
Total		4775144	438678	100.000	100.000

HPLC chromatogram of racemic 2

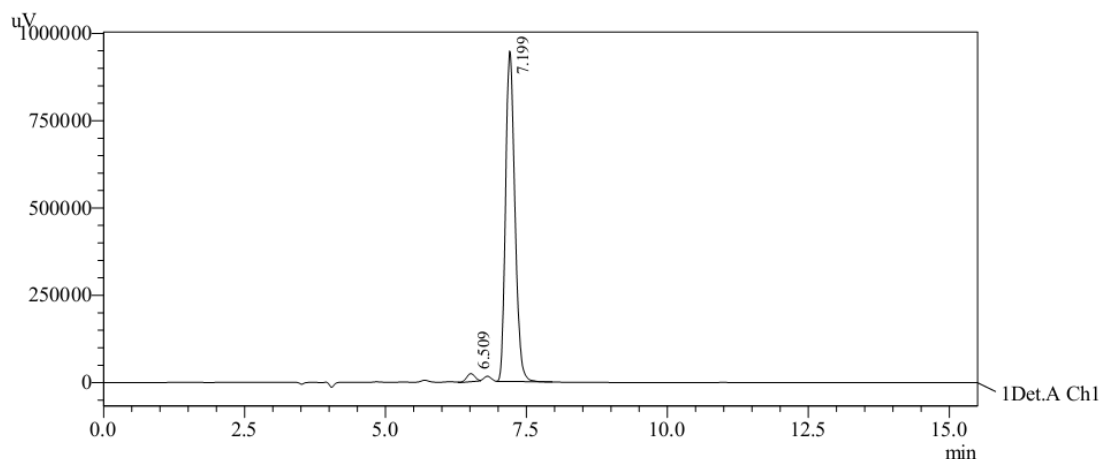


1 Det.A Ch1 / 254nm

PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.592	281123	29584	1.480	1.917
2	7.249	18708501	1513497	98.520	98.083
Total		18989624	1543081	100.000	100.000

HPLC chromatogram of 2 (produced by 7a)



1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	6.509	243496	23273	2.191	2.399
2	7.199	10870780	946845	97.809	97.601
Total		11114276	970118	100.000	100.000

HPLC chromatogram of 2 (produced by 7b)

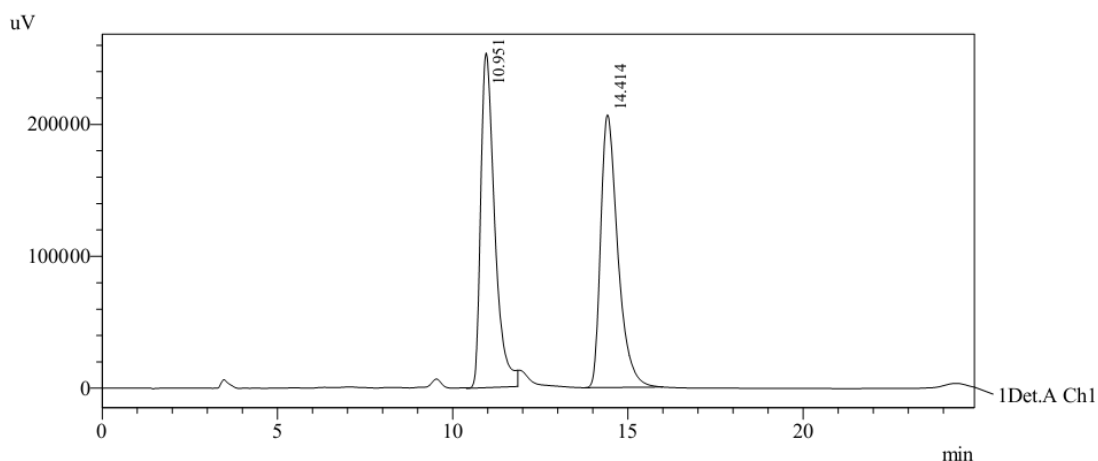
The enantiomeric excesses of 1a and racemic 1a were determined by HPLC with a Chiralcel AD-H column using Agilent 1100 instrument.

Conditions:

wave length: 254 nm

flow rate: 1.0 mL/ min

mobile phase: i-PrOH: hexane = 50:50



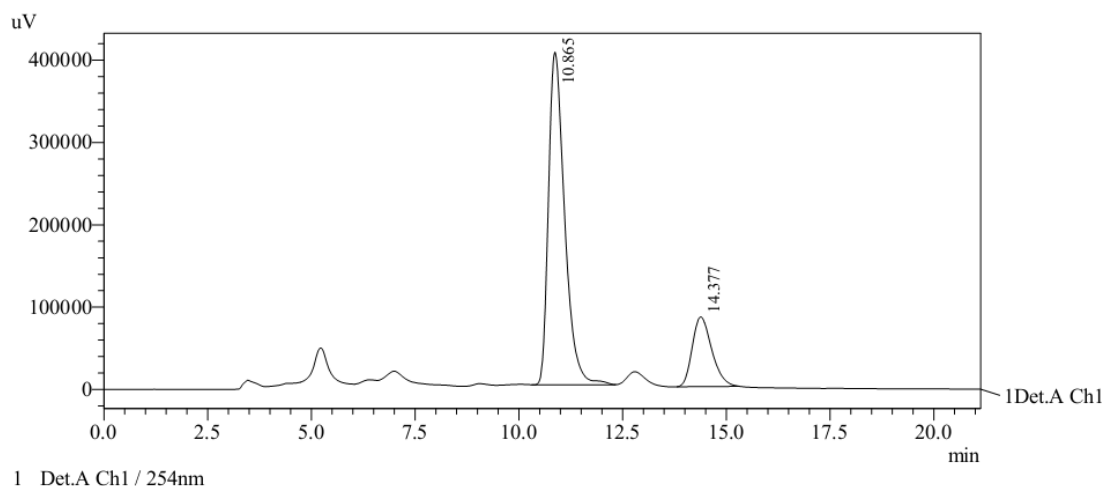
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.951	7005371	253739	49.973	55.106
2	14.414	7012943	206715	50.027	44.894
Total		14018314	460454	100.000	100.000

HPLC chromatogram of racemic 1a



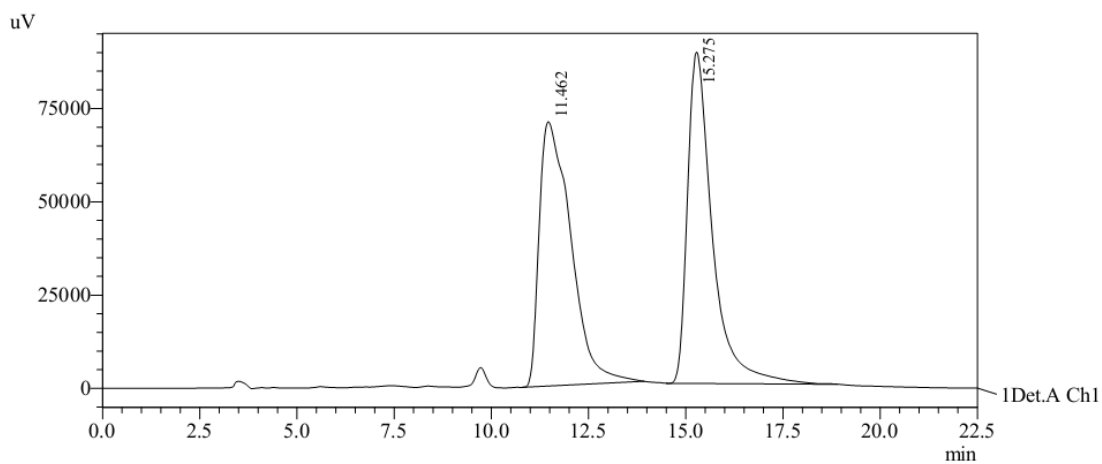
1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.865	10924037	404119	79.767	82.667
2	14.377	2770899	84731	20.233	17.333
Total		13694937	488850	100.000	100.000

HPLC chromatogram of 1a [produced by 5 (Table 1, Entry 3)]



1 Det.A Ch1 / 254nm

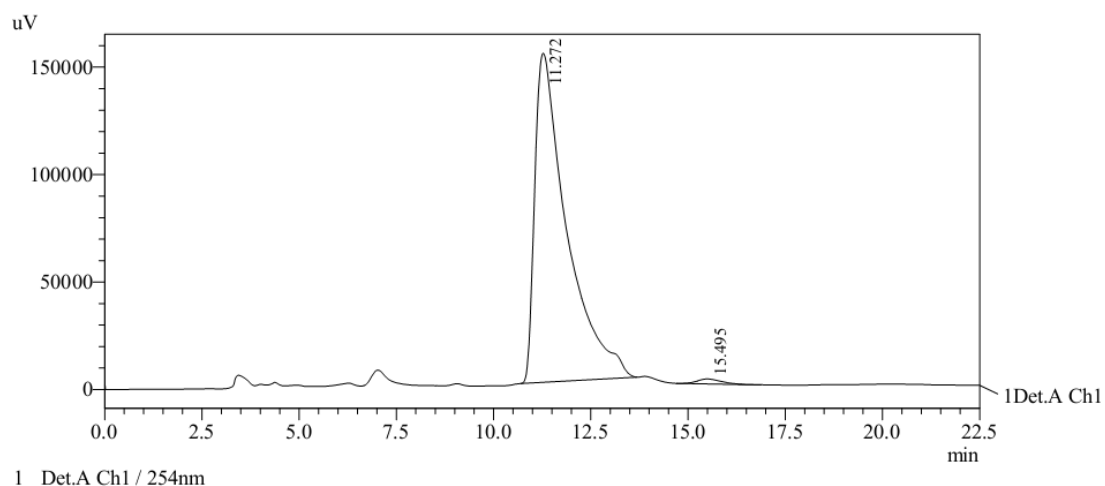
PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.462	4002097	70785	50.281	44.355
2	15.275	3957302	88803	49.719	55.645
Total		7959399	159588	100.000	100.000

HPLC chromatogram of racemic 1a

(the drift in contrast with above HPLC chromatogram was due to different testing batches)



PeakTable

Detector A, Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	11.272	8763385	153208	98.870	98.512
2	15.495	100185	2315	1.130	1.488
Total		8863570	155523	100.000	100.000

HPLC chromatogram of 1a (produced by 2)