An Enantioselective Strategy for the Total Synthesis of (*S*)-Tylophorine via Catalytic Asymmetric Allylation and One-Pot DMAP-Promoted Isocyanate Formation/Lewis Acid Catalyzed Cyclization Sequence

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

State Key Laboratory of Elemento-Organic Chemistry, Research Institute of Elemento-Organic Chemistry, Nankai University, Tianjin 300071, People's Republic of China

wang98h@263.net; wangqm@nankai.edu.cn

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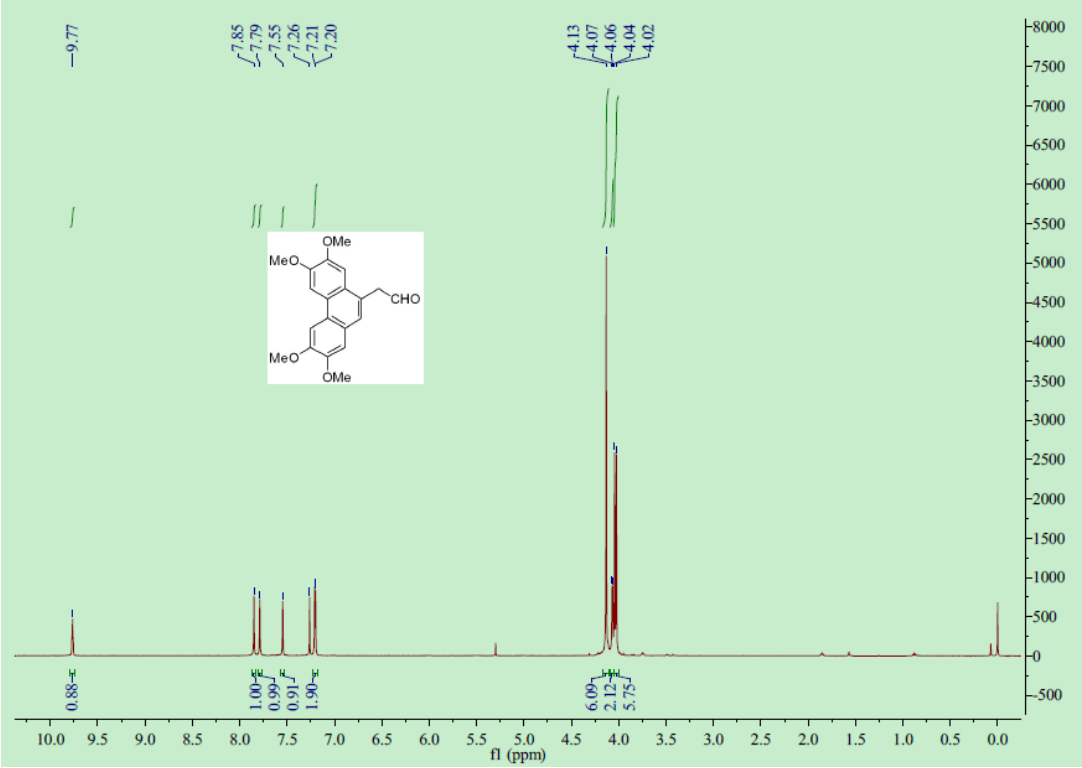
NMR Data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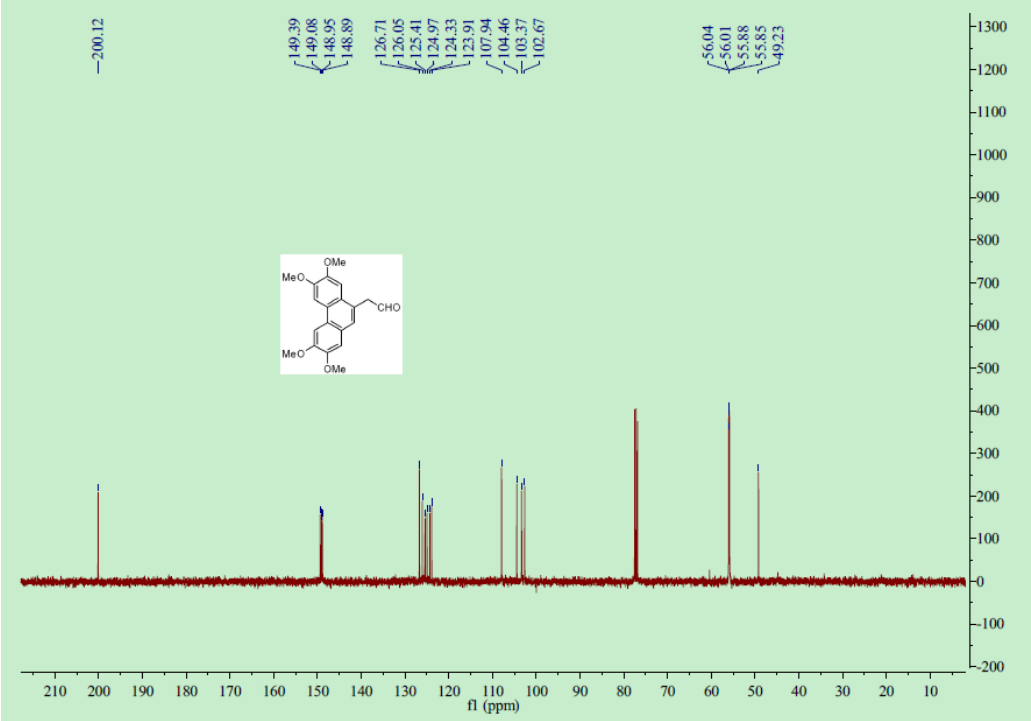
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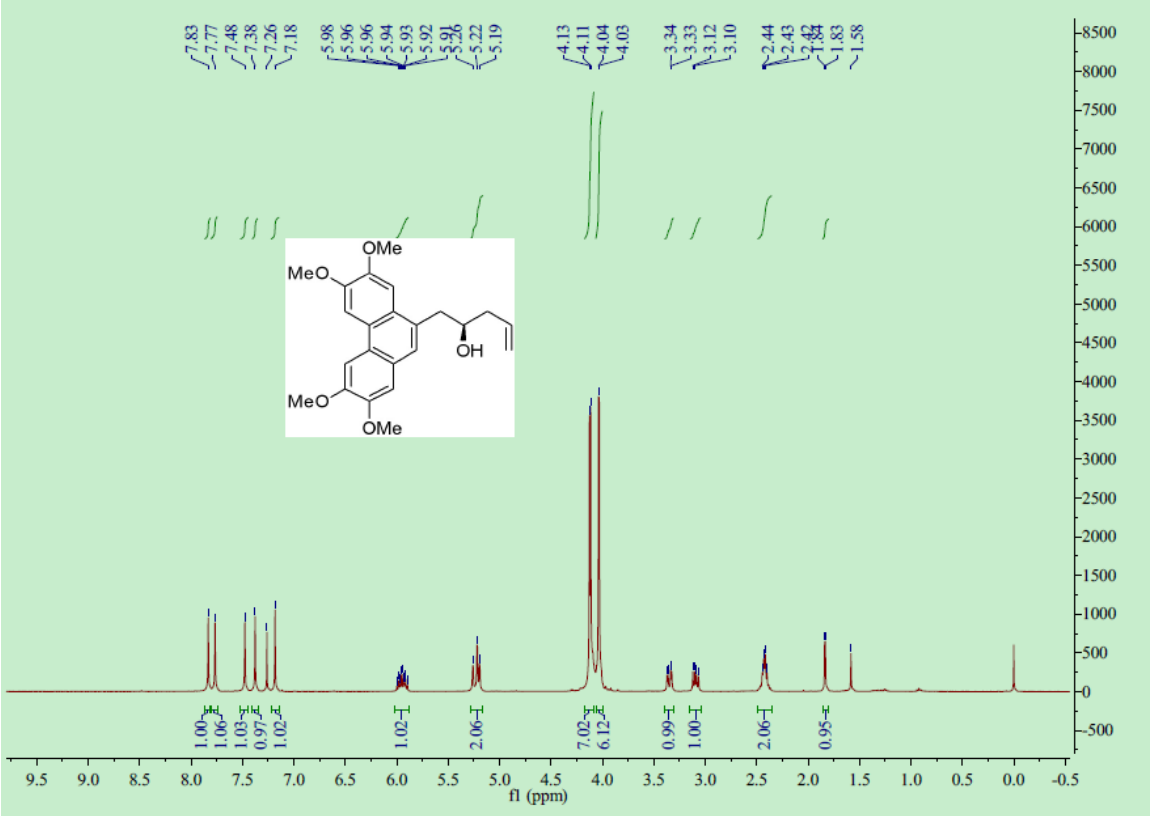
f1 (ppm)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OH
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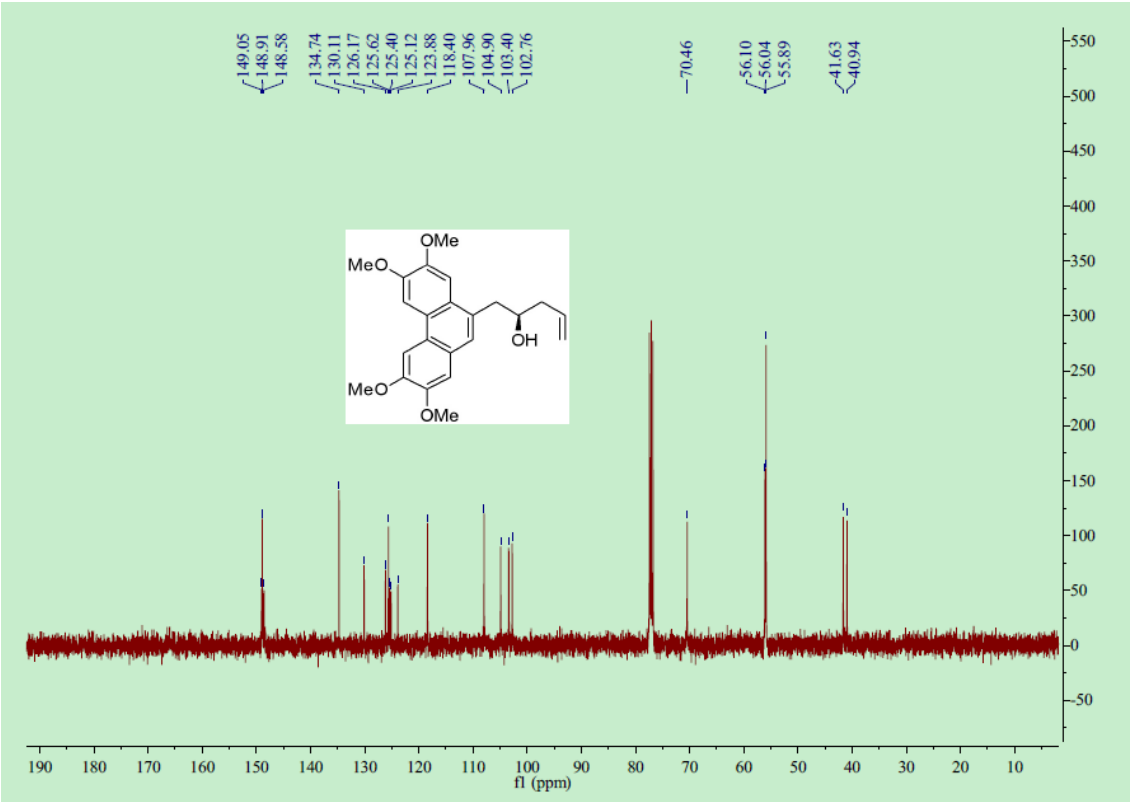
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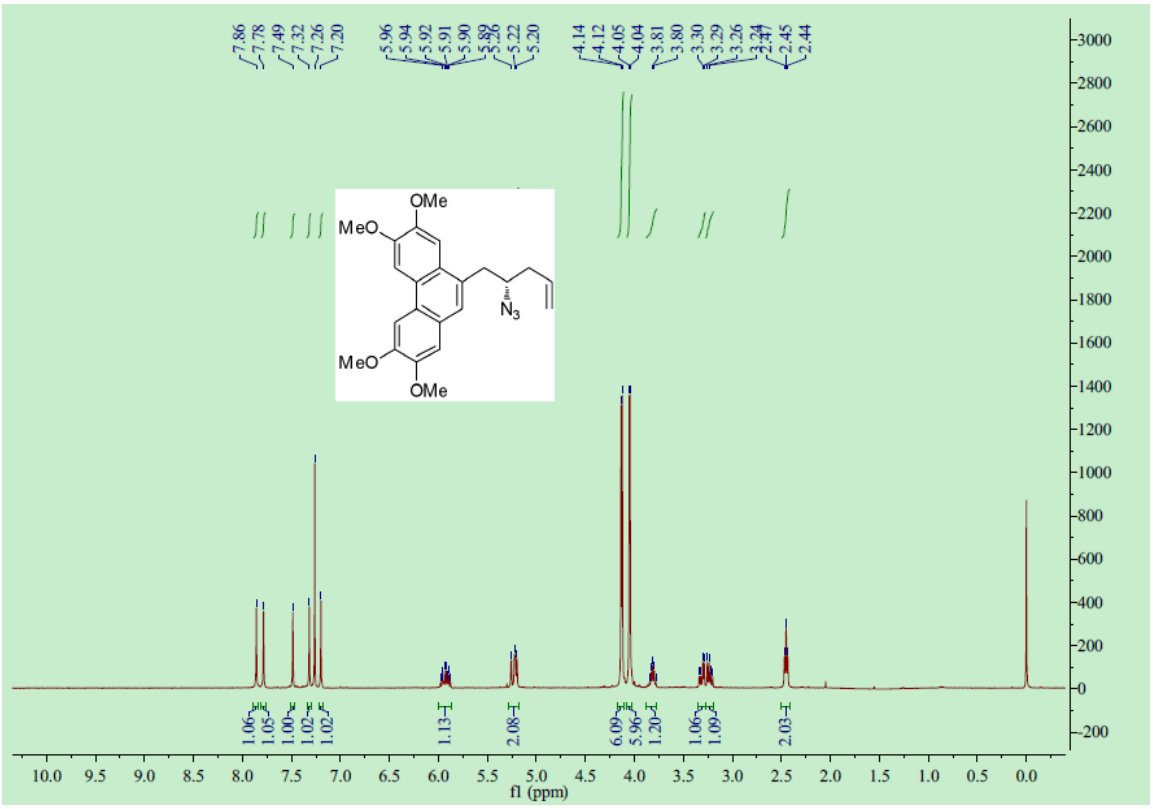
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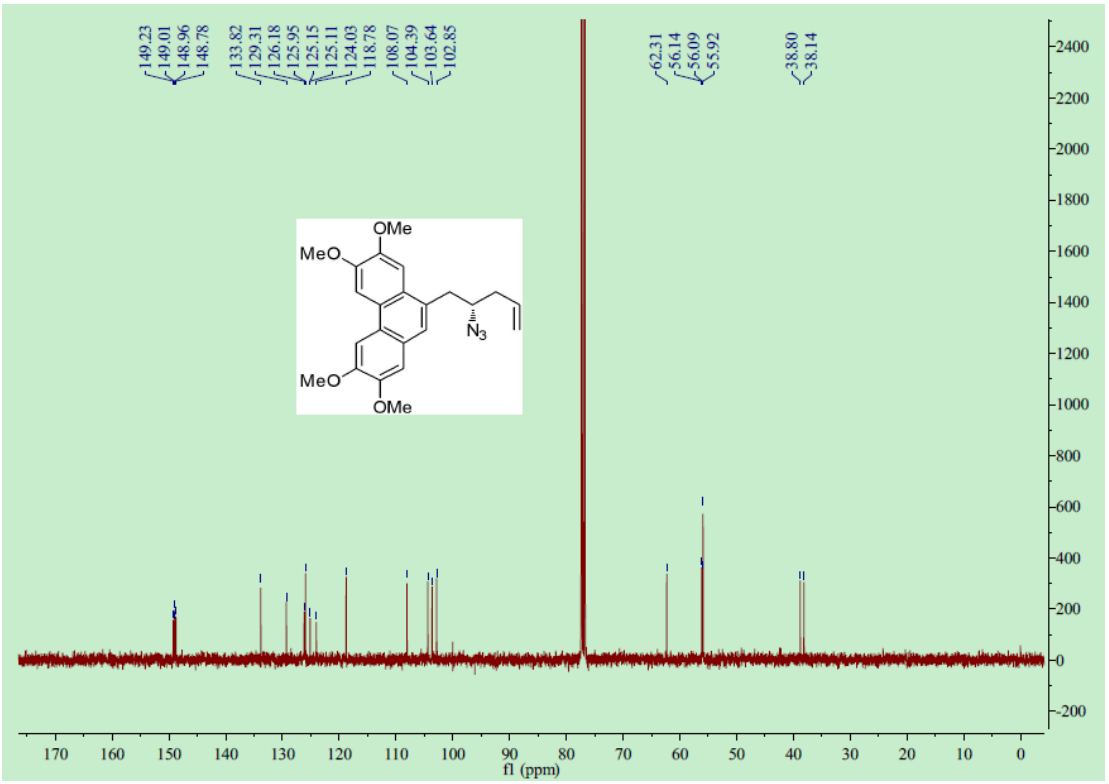
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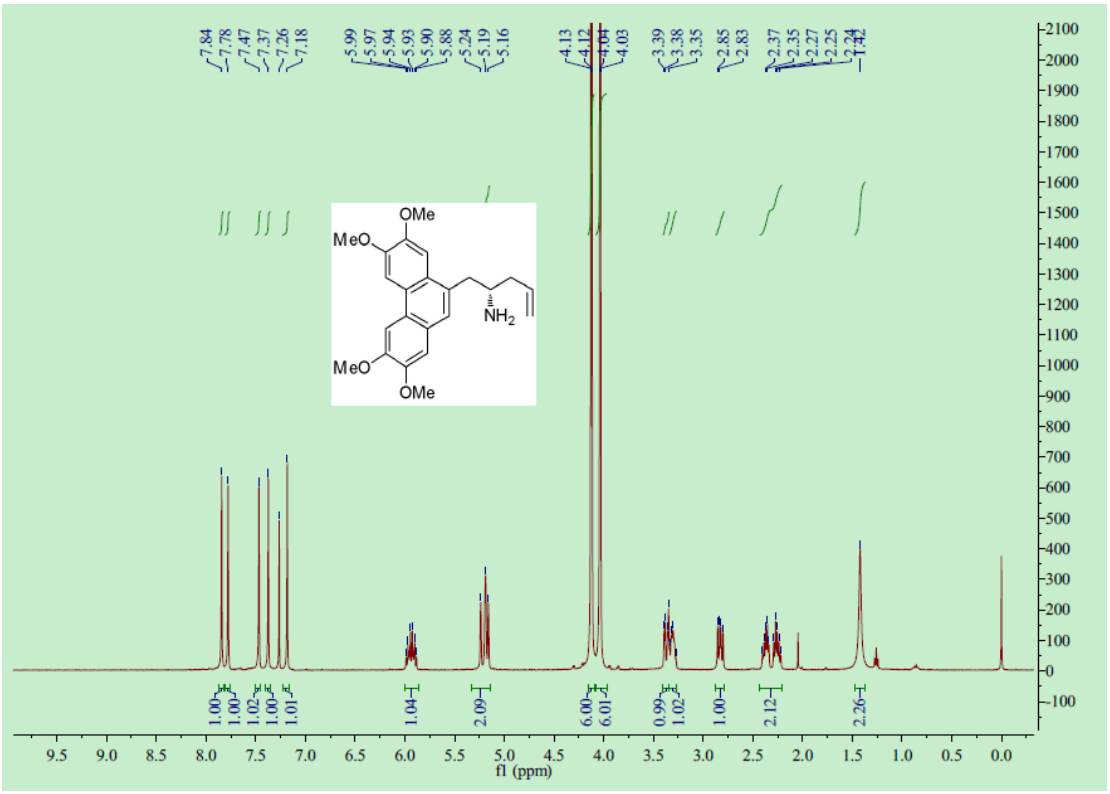
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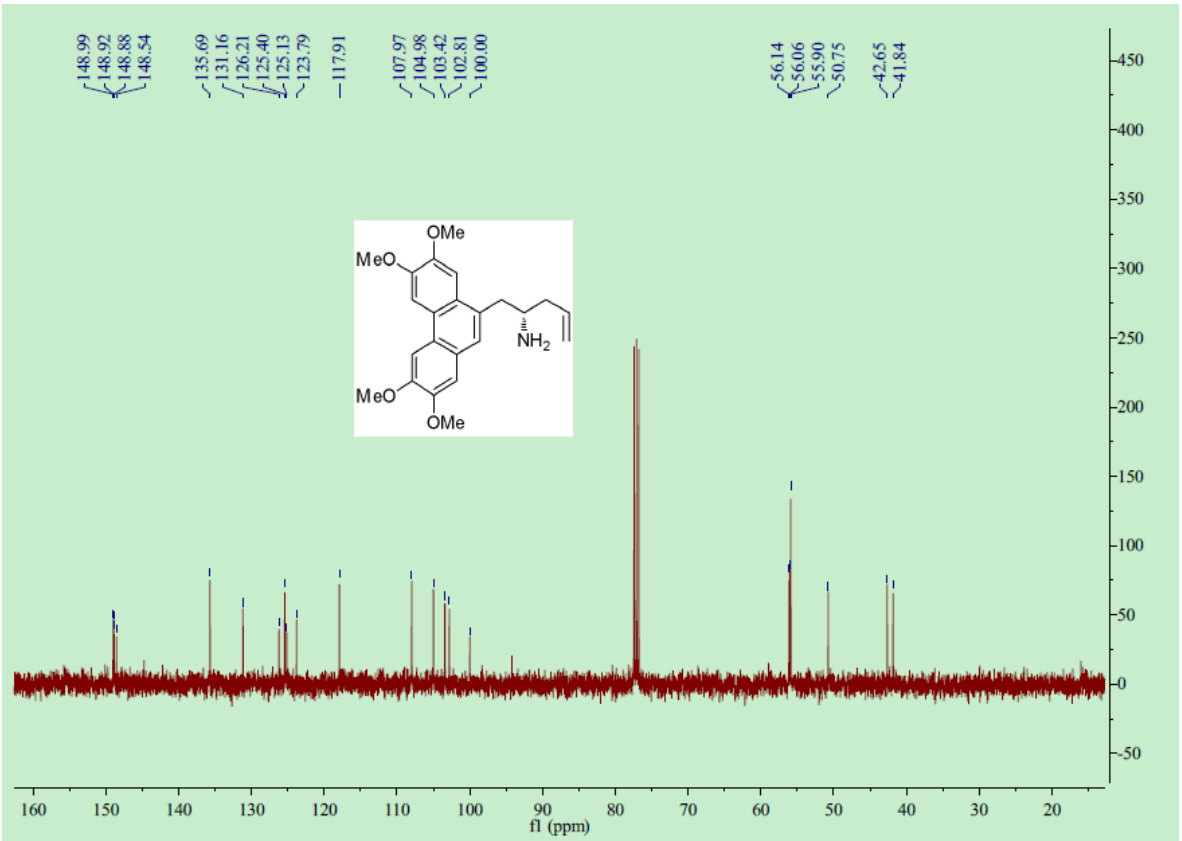
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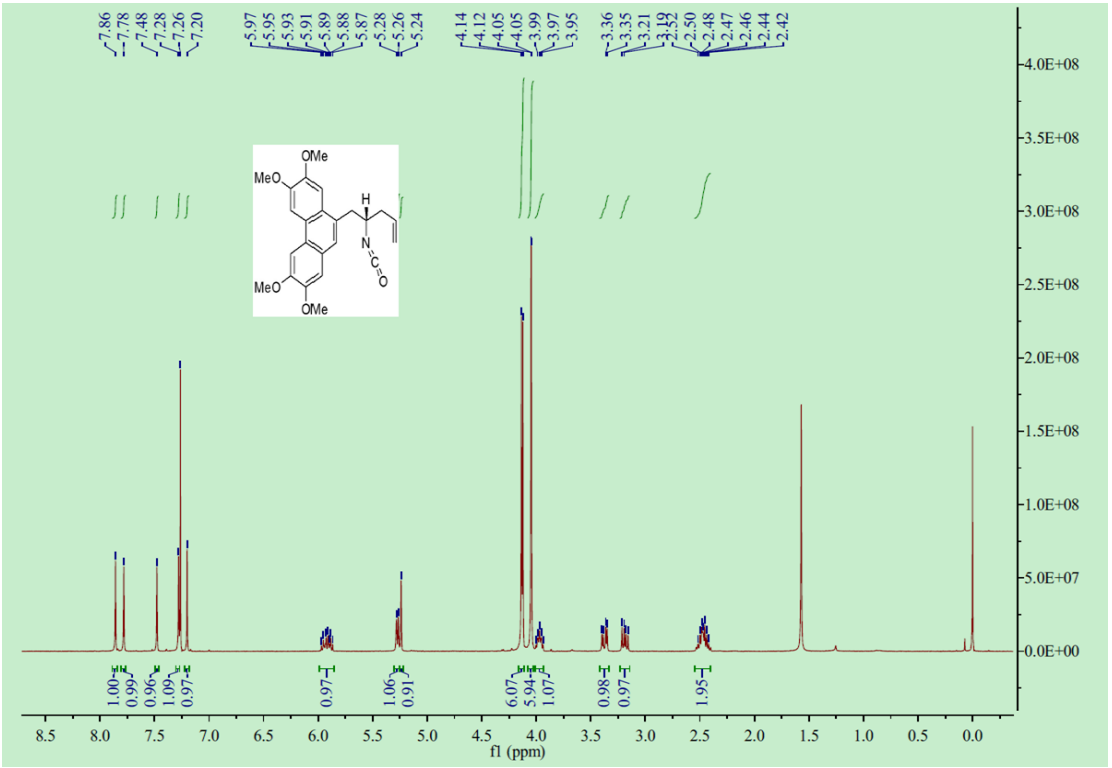
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C
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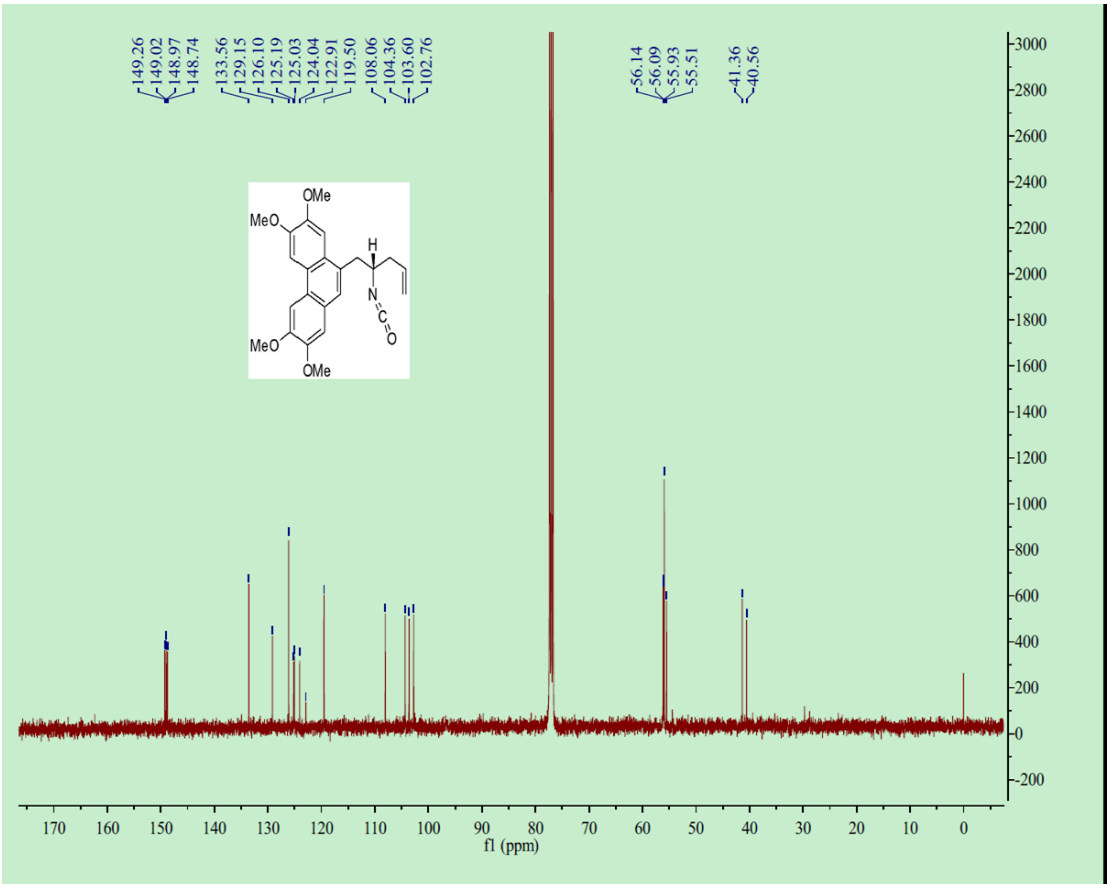
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MeO
MeO
OMe
H
N
C
O
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2400
2200
2000
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400
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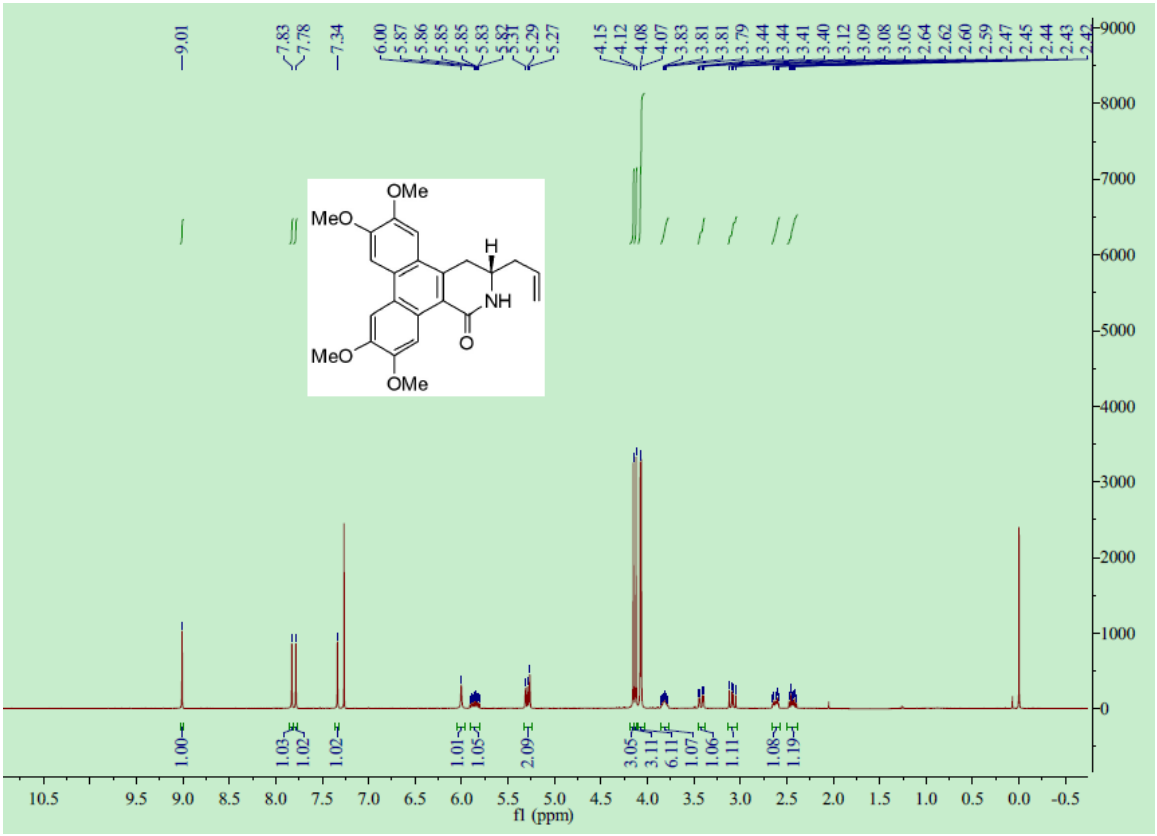
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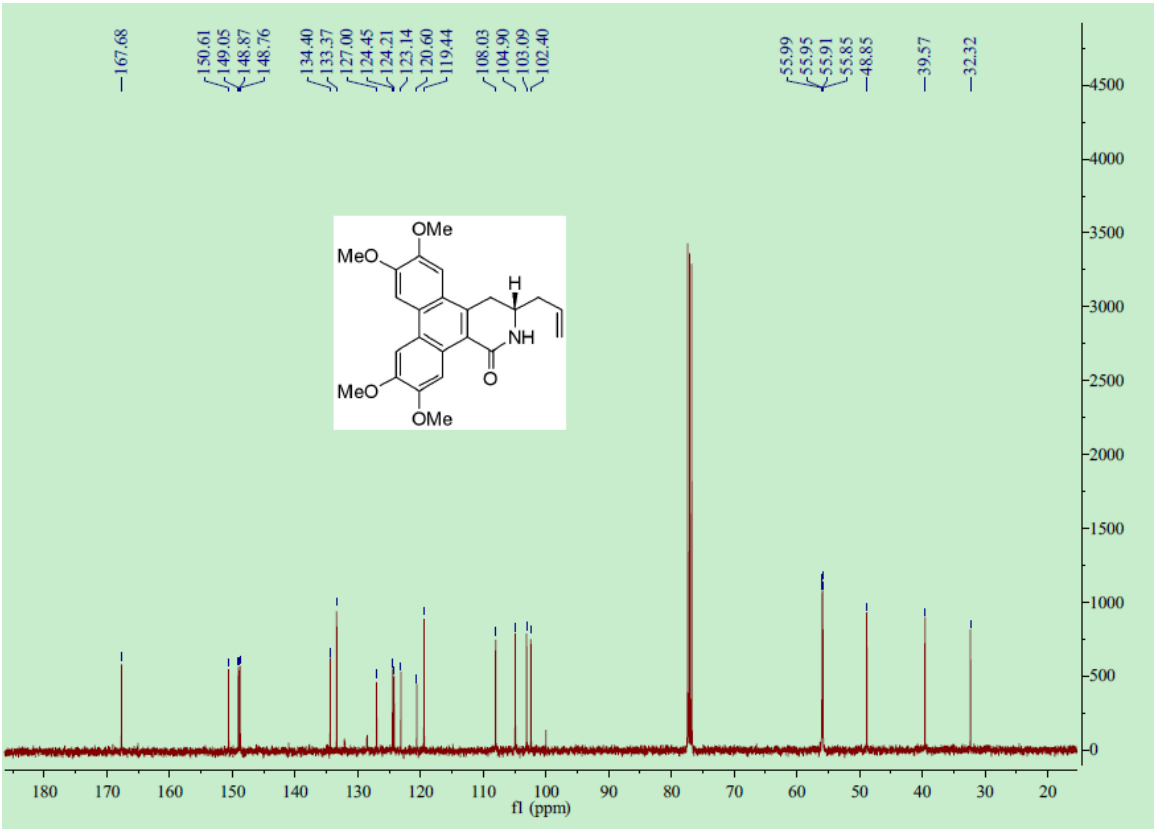
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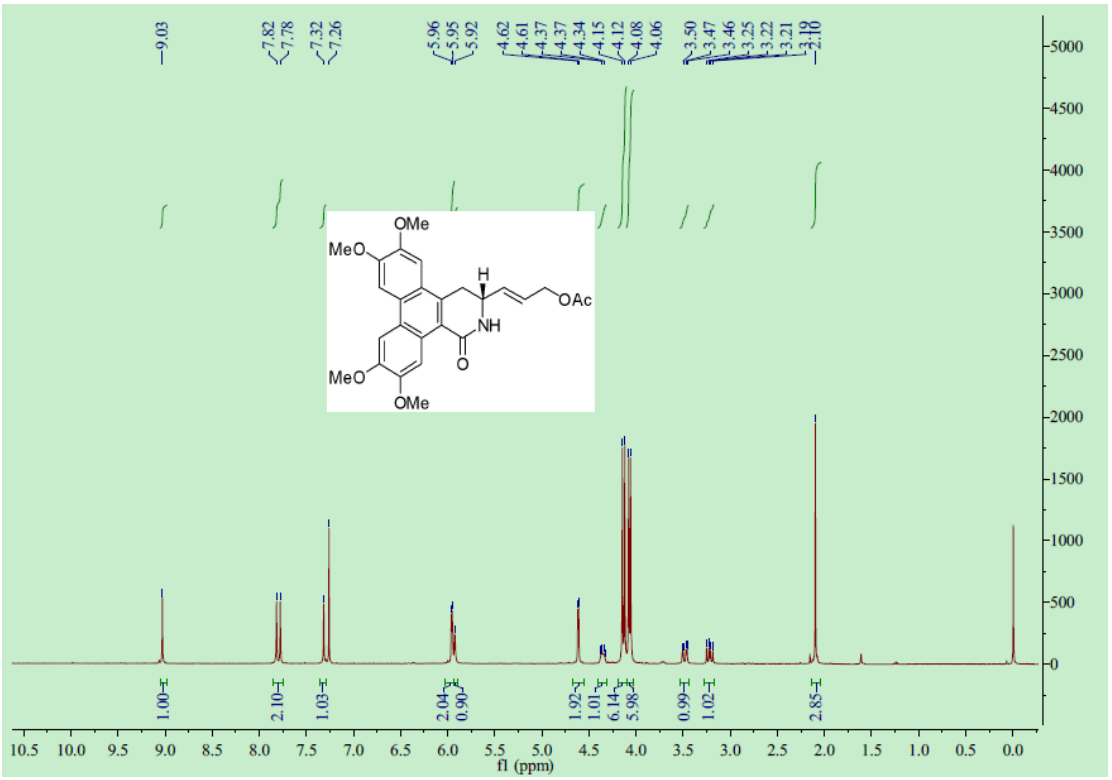
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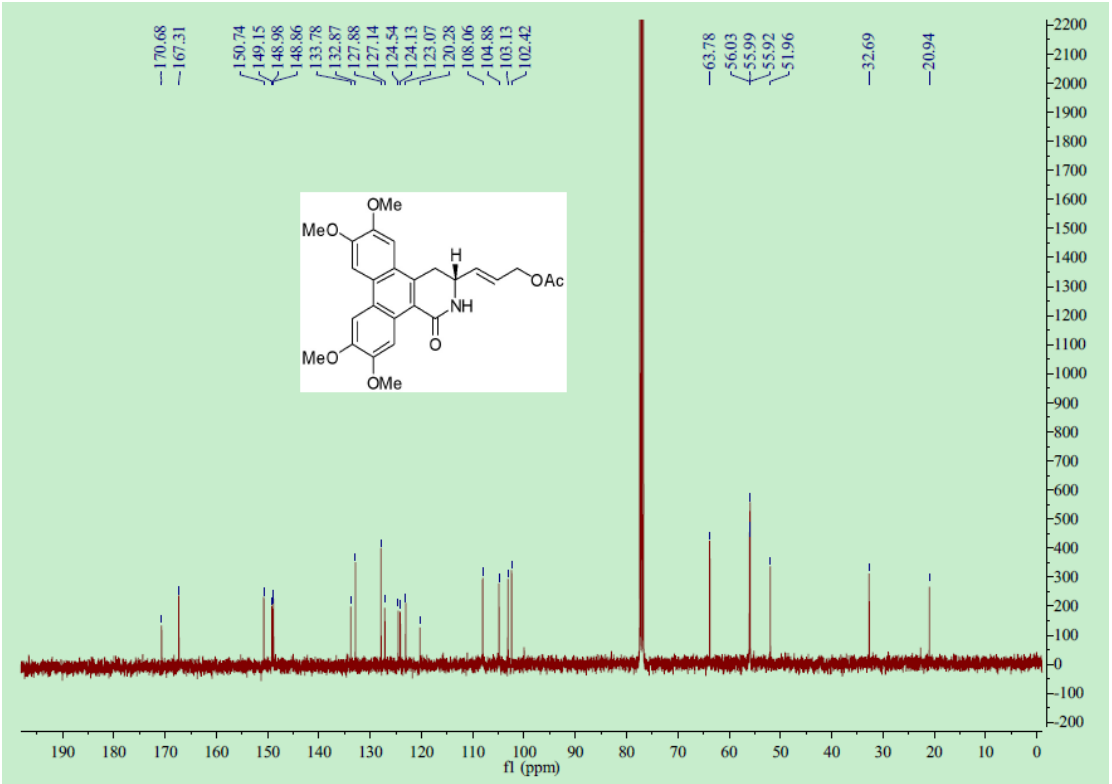
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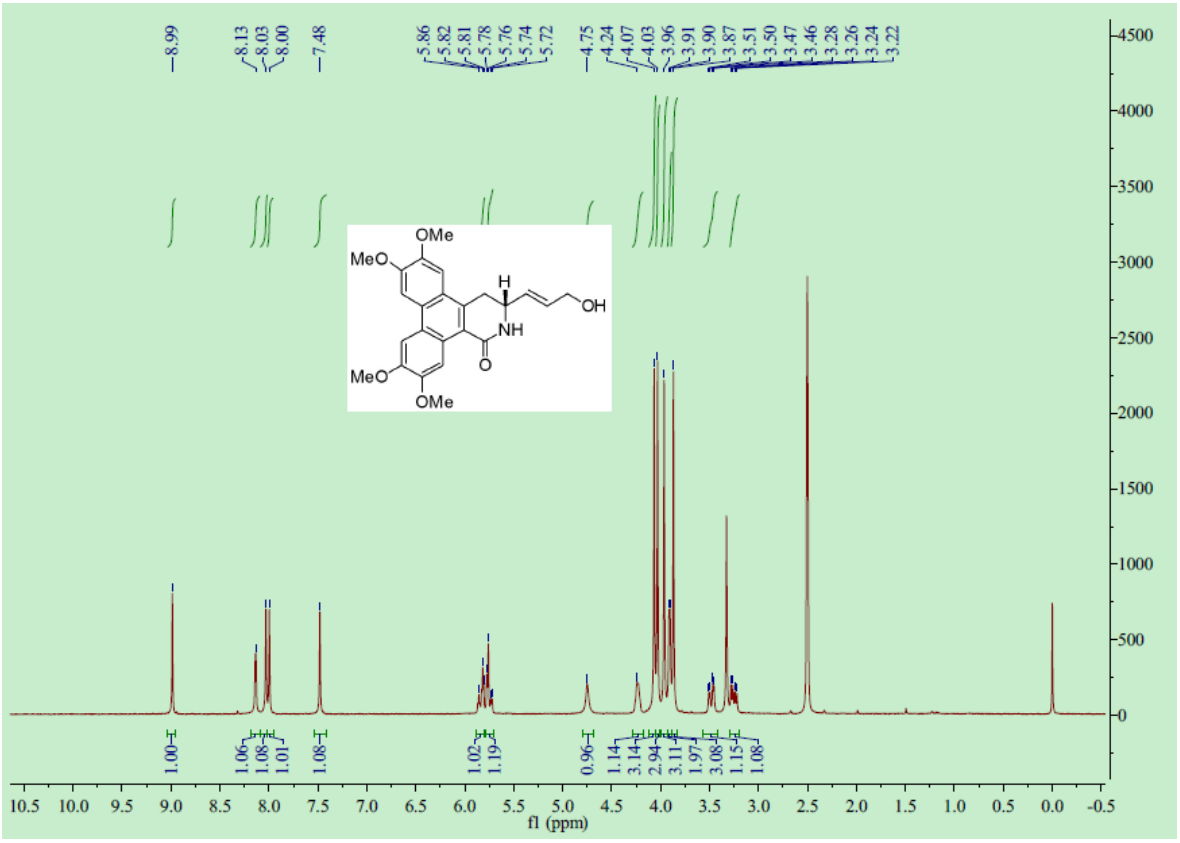
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OH
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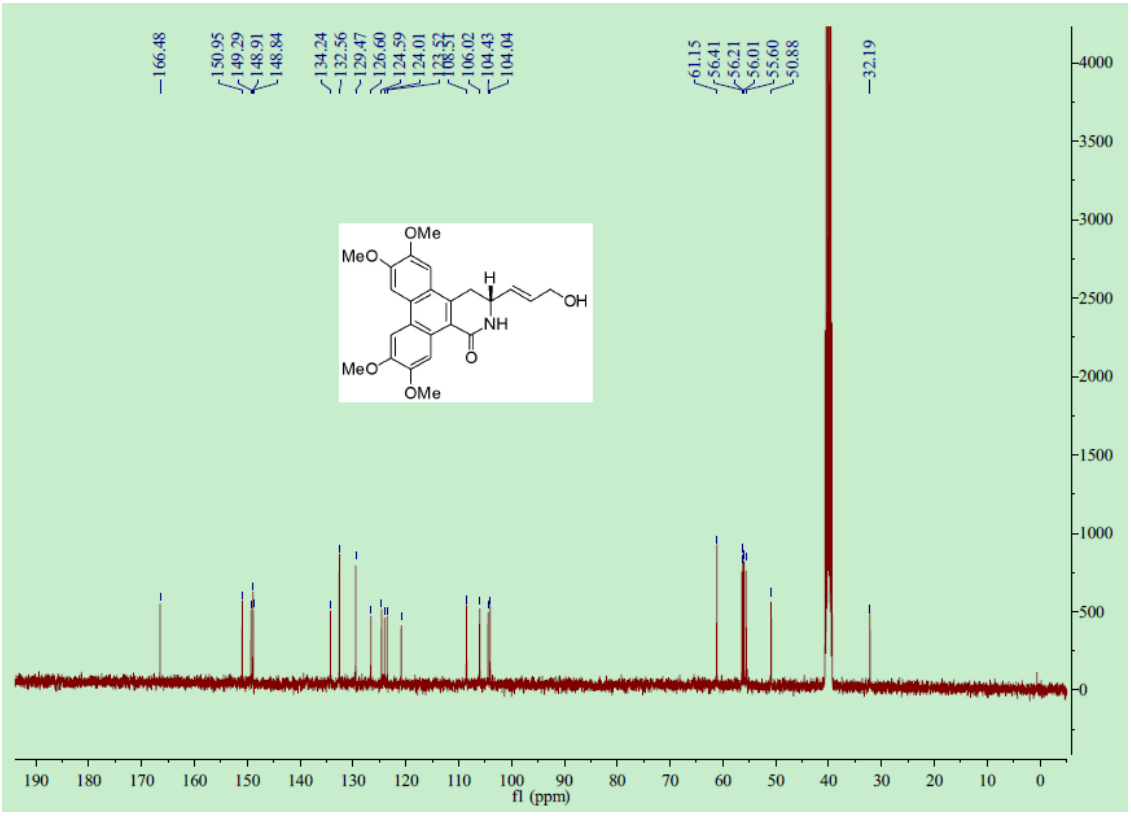
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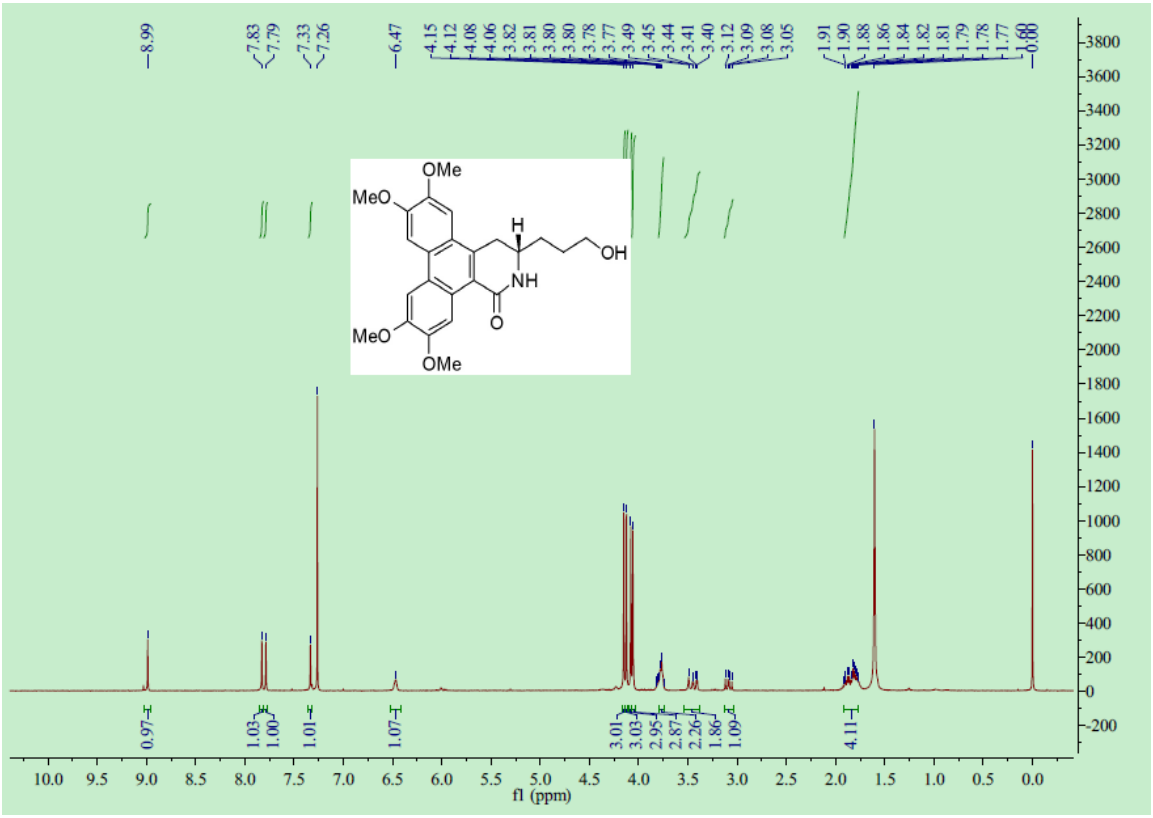
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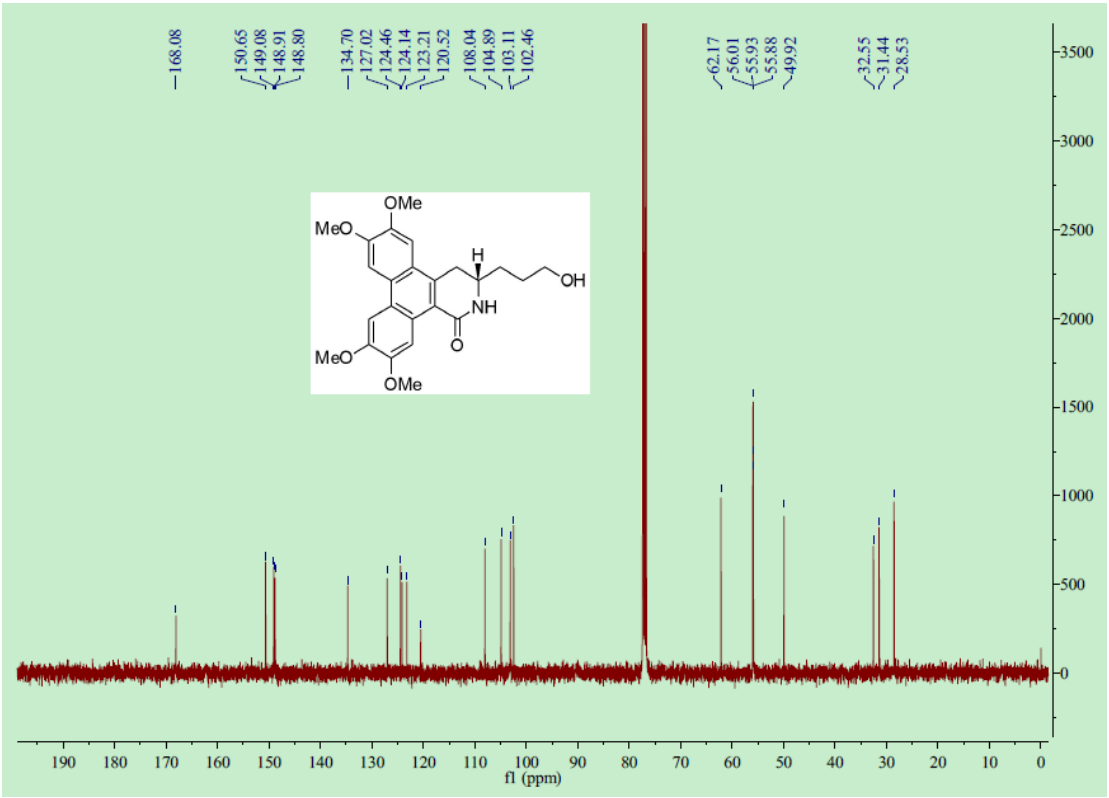
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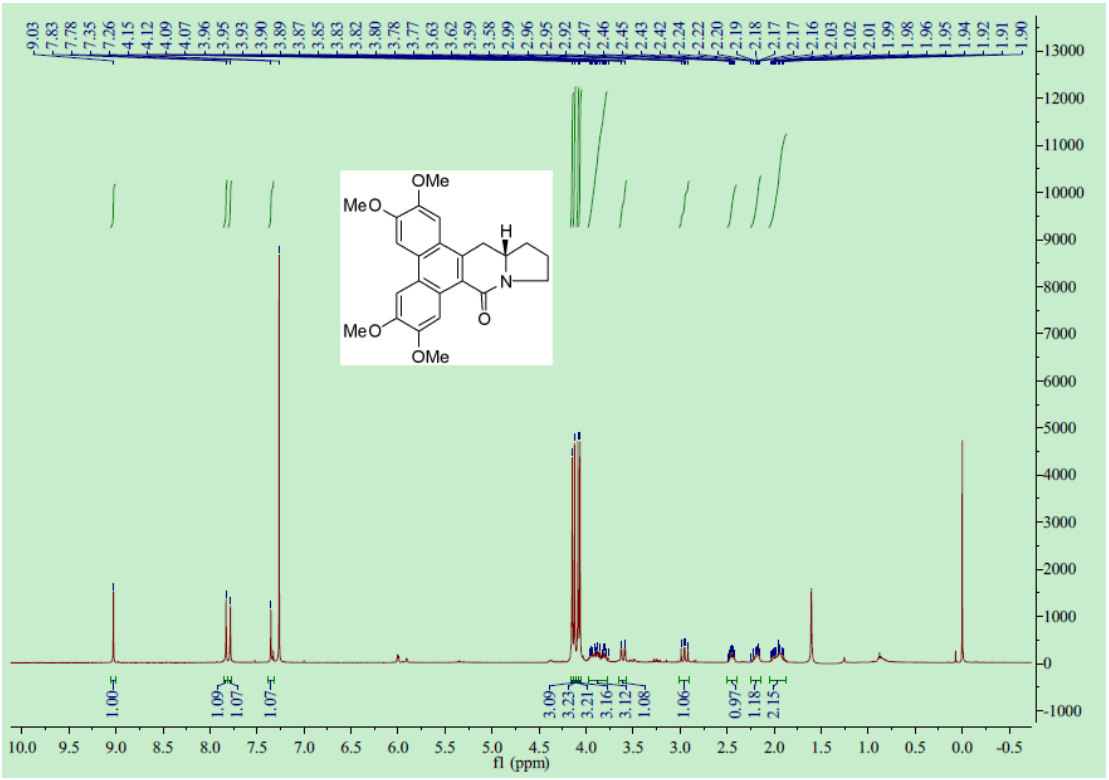
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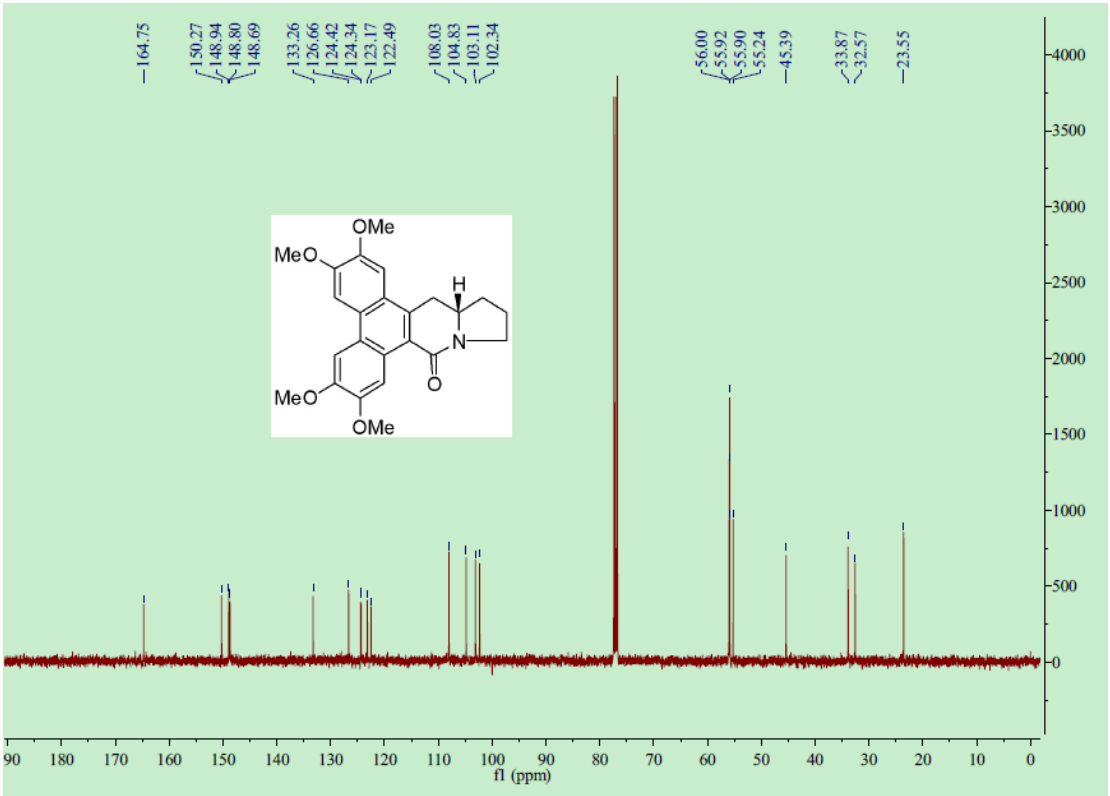
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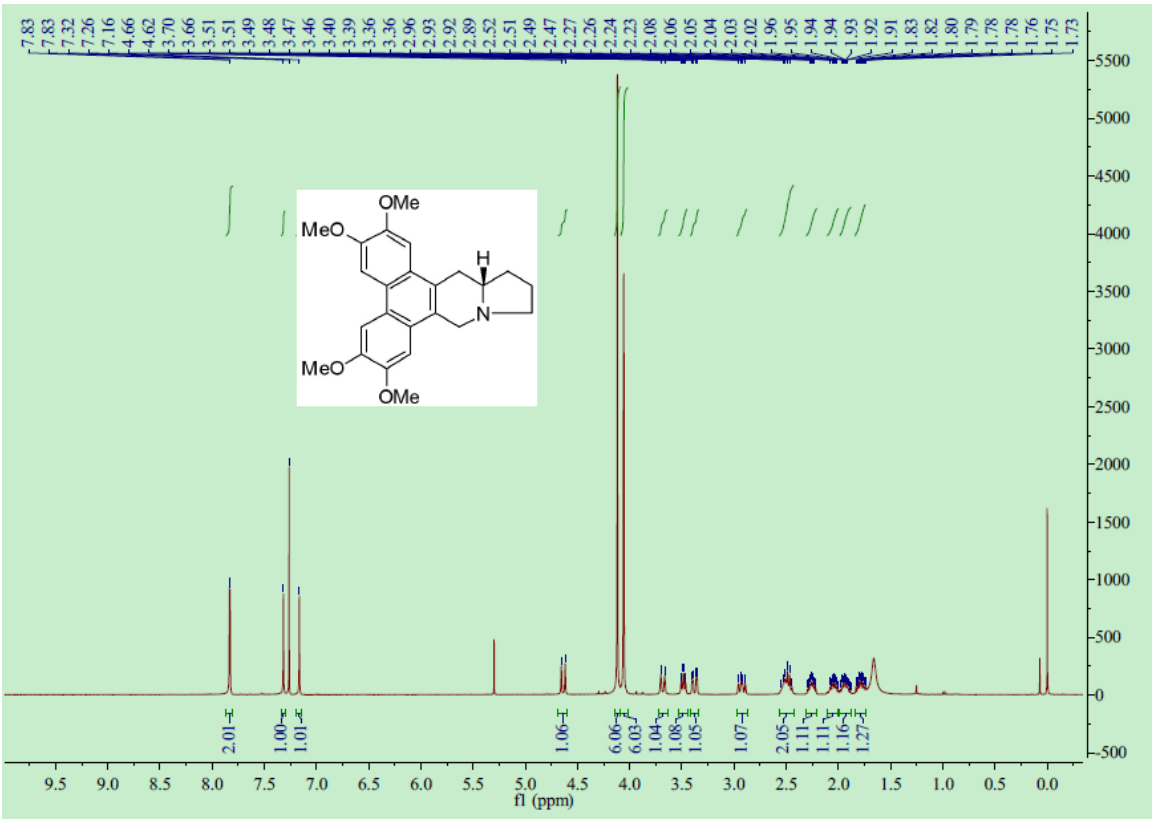
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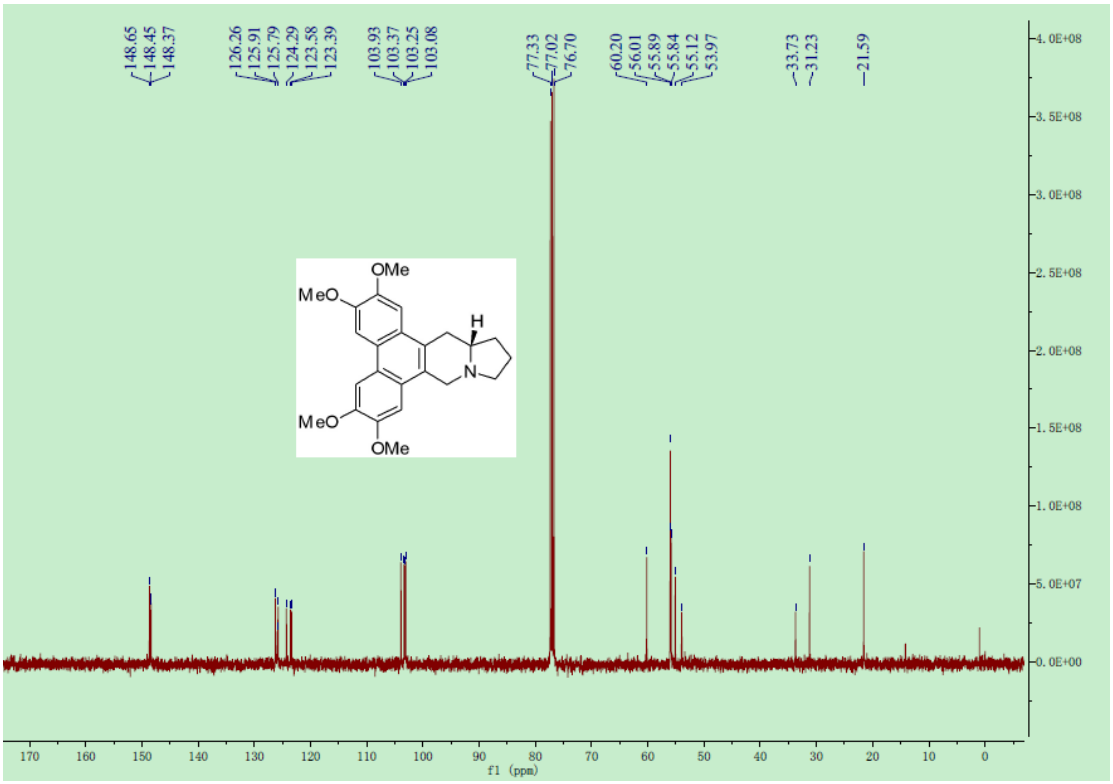

OMe
MeO
H
N
MeO
OMe
f1 (ppm)

HPLC data

Conditions for racemic and enantiopure compound 3:

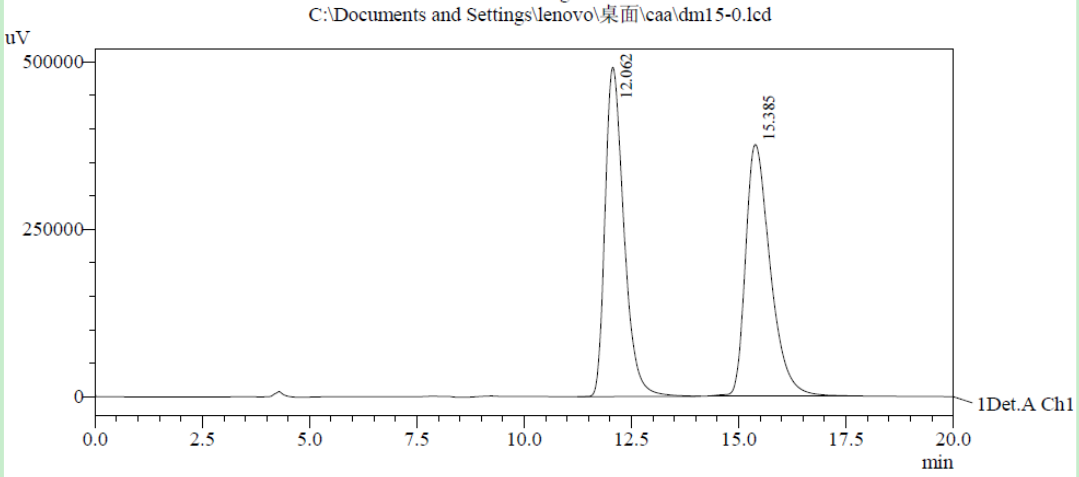
Phenomenex Lux Cellulose-1 column, i-PrOH:Hexane = 30:70, 1.0 mL/min, 254nm。

Sample Information

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail#	:
Injection Volume	: 20 uL
Data Filename	: dm15-0.lcd
Method Filename	: C-1-i-PrOH.lcm
Batch Filename	:
Report Filename	: vvv.lcr
Date Acquired	: 2011-7-21 17:05:37
Data Processed	: 2011-7-21 17:37:08

Chromatogram

C:\Documents and Settings\lenovo\桌面\caa\dm15-0.lcd



1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.062	15019850	491391	49.947	56.699
2	15.385	15051617	375272	50.053	43.301
Total		30071467	866663	100.000	100.000

HPLC data for recemic compound 3

Sample Information

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail#	:
Injection Volume	: 20 uL
Data Filename	: dm16-8.lcd
Method Filename	: C-1-i-PrOH.lcm
Batch Filename	:
Report Filename	: vvv.lcr
Date Acquired	: 2011-7-27 20:54:23
Data Processed	: 2011-7-27 22:37:46

Chromatogram

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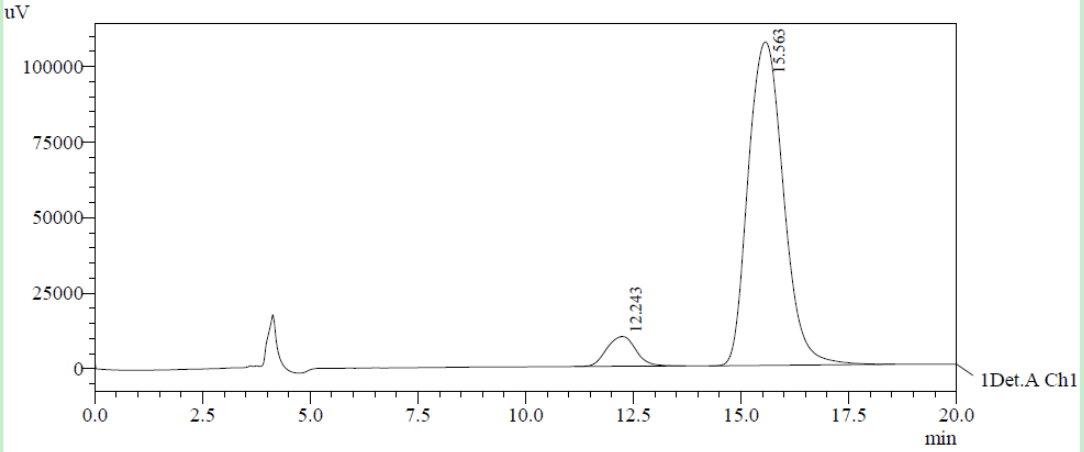

1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	12.243	462456	9849	7.042	8.425
2	15.563	6104875	107055	92.958	91.575
Total		6567331	116905	100.000	100.000

HPLC data for compound (*R*)-3

Conditions for racemic and enantiopure tylophorine:

Chiral AD-H column i-PrOH:Hexane = 30:70 (0.1% Et_3N), 1.0 mL/min, 254nm。

Sample Information

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail#	:
Injection Volume	: 20 uL
Data Filename	: dm16-16-0.lcd
Method Filename	: 100%yijing-1.lcm
Batch Filename	:
Report Filename	: vvv.lcr
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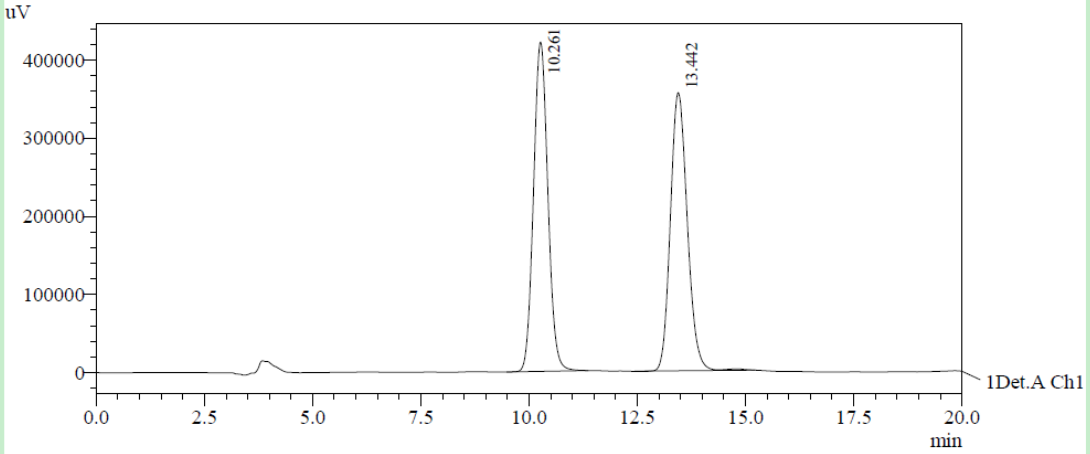
Chromatogram

G:\LC user\dengmeng\data\dm16-16-0.lcd



1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.261	9722999	421426	50.069	54.215
2	13.442	9696050	355897	49.931	45.785
Total		19419049	777323	100.000	100.000

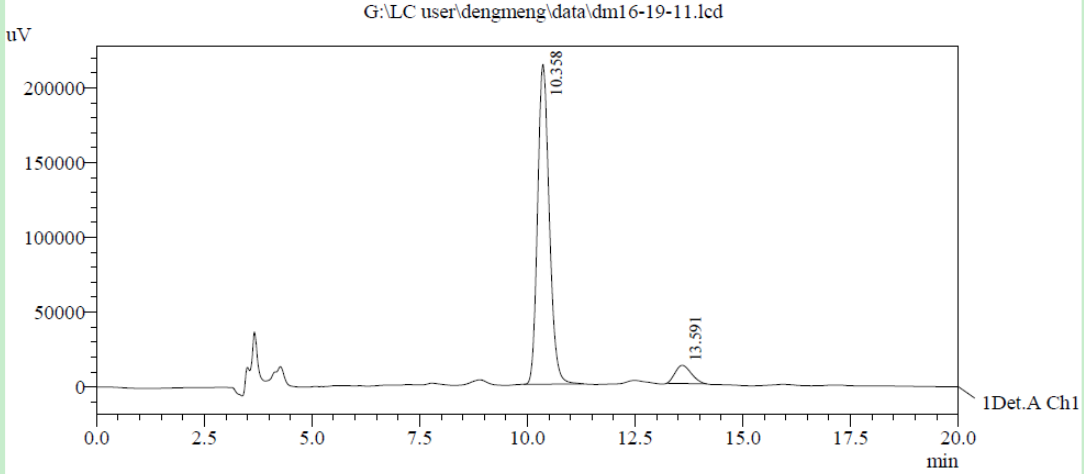
HPLC data for racemic tylophorine

Sample Information

Acquired by	: Admin
Sample Name	:
Sample ID	:
Vail#	:
Injection Volume	: 20 uL
Data Filename	: dm16-19-11.lcd
Method Filename	: 100%yijing-1.lcm
Batch Filename	:
Report Filename	: vvv.lcr
Date Acquired	: 2011-9-23 18:34:31
Data Processed	: 2011-9-23 21:36:28

Chromatogram

G:\LC user\dengmeng\data\dm16-19-11.lcd



1 Det.A Ch1 / 254nm

PeakTable

Detector A Ch1 254nm

Peak#	Ret. Time	Area	Height	Area %	Height %
1	10.358	4043620	213859	92.720	94.625
2	13.591	317488	12149	7.280	5.375
Total		4361108	226008	100.000	100.000

HPLC data for (*S*)-tylophorine