

Supporting Information for

Mukaiyama type Aldol Reaction for the Synthesis of *trans*-2,6-Disubstituted Tetrahydropyrans: Synthesis of Diospongin A and B

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^a*Department of Organic Synthesis and Process Chemistry, CSIR-Indian Institute of Chemical Technology, Hyderabad 500007, India*

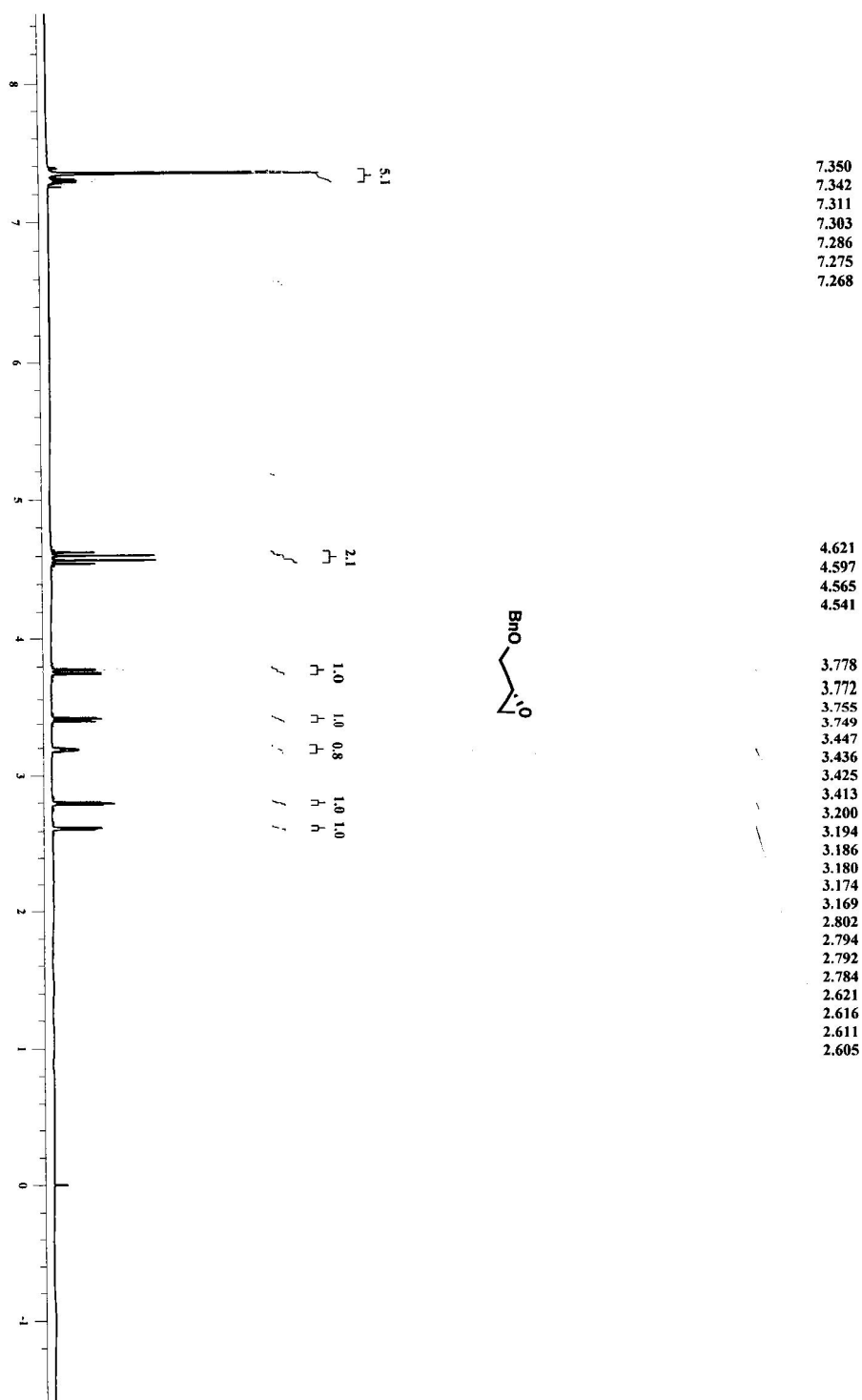
^b*Academy of Scientific and Innovative Research (AcSIR), Mathura Road, New Delhi 110025, India*

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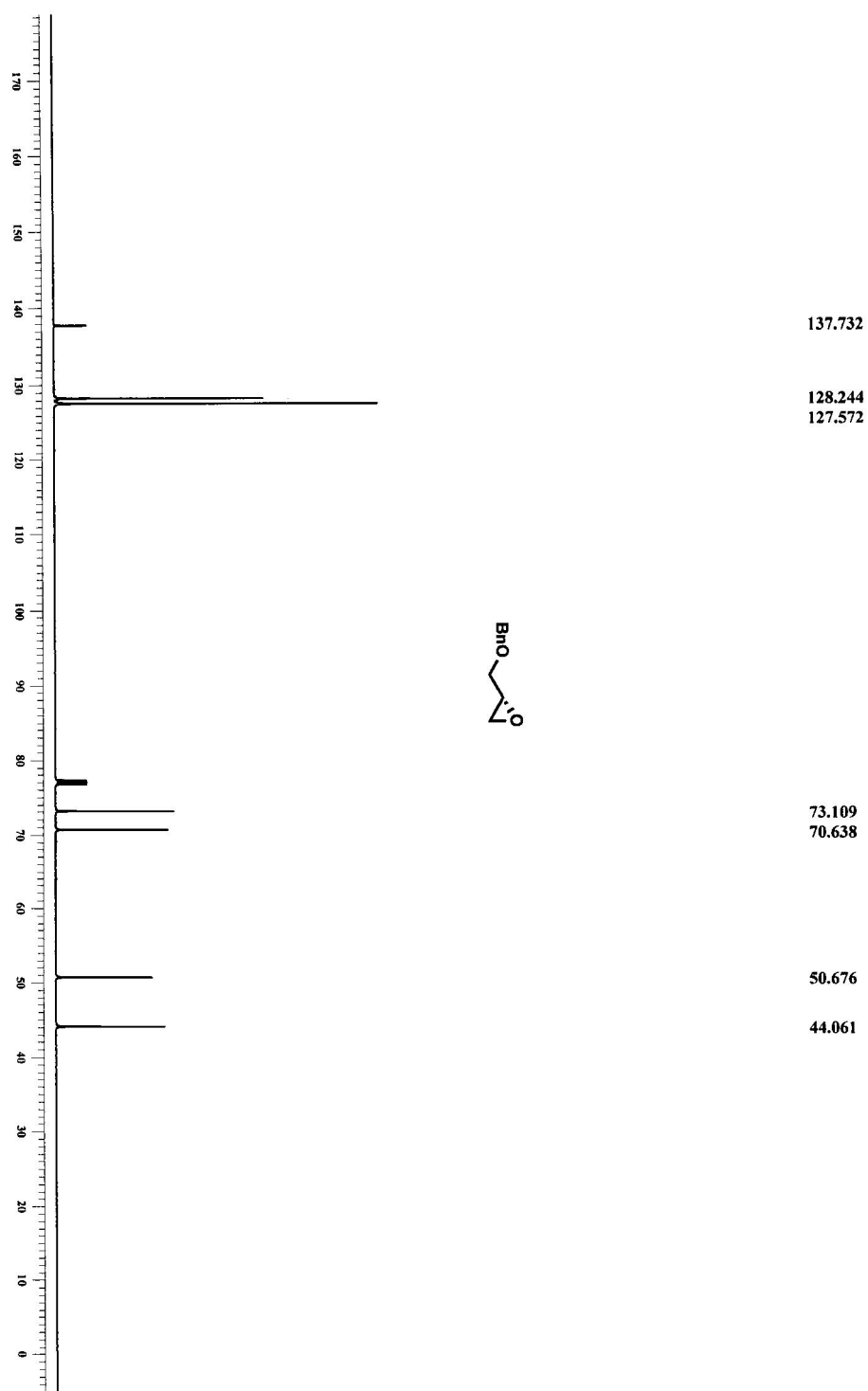
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¹H NMR Spectrum of 9 (500 MHz, CDCl₃)

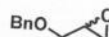


¹³C NMR Spectrum of 9 (125 MHz, CDCl₃)

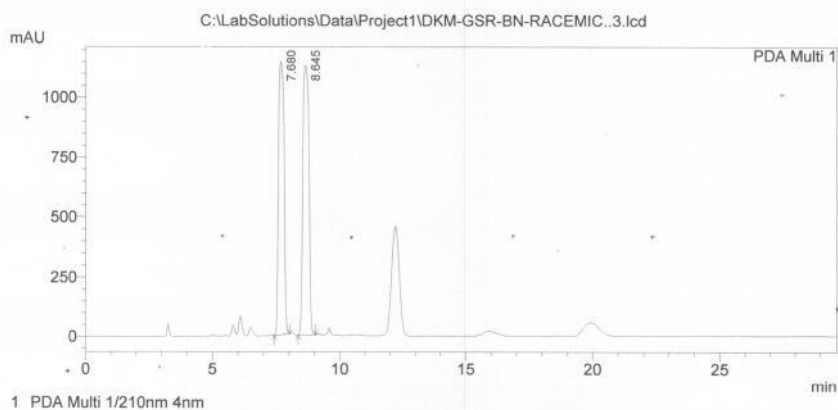
9/15/2016 15:52:48 1 / 1

IICT NPL

Acquired by : Admin
 Sample Name : DKM-GSR-BN-RACEMIC
 Sample ID : DKM-GSR-BN-RACEMIC
 Vial # : 3
 Injection Volume : 20 μ L
 Data File Name : DKM-GSR-BN-RACEMIC..3.lcd
 Method File Name : DKM.lcm
 Batch File Name :
 Report File Name : Report Format.lcr
 Data Acquired : 9/1/2016 10:14:03 AM
 Data Processed : 9/1/2016 10:43:48 AM
 MOBILE PHASE: 10% IPA IN HEXANE
 COLUMN: CHIRAL PAK IC 250X4.6mm 5 μ
 DECTION: 210nm
 FLOW RATE: 1mL/min



<Chromatogram>



PeakTable

Peak#	Ret. Time	Area	Height	Area %	Height %
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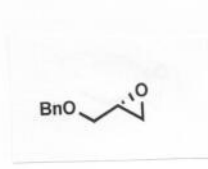
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Chiral HPLC Report of 8

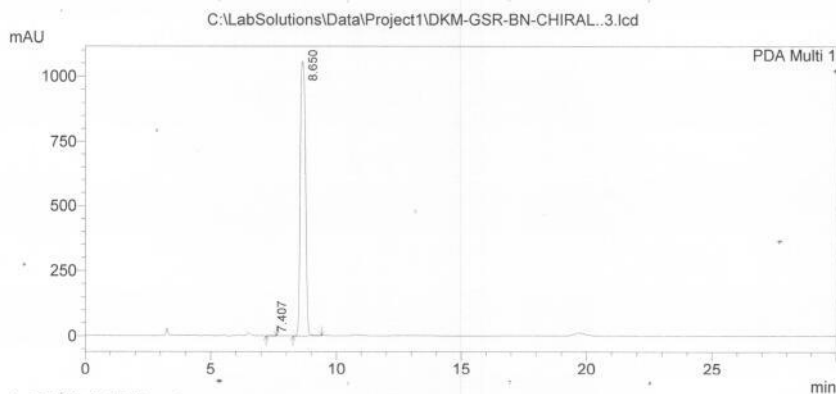
9/15/2016 15:55:29 1 / 1

IICT NPL

Acquired by : Admin
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 Sample ID : DKM-GSR-BN-CHIRAL
 Vial # : 4
 Injection Volume : 20 uL
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 Method File Name : DKM.lcm
 Batch File Name :
 Report File Name : Report Format.lcr
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 Data Processed : 9/1/2016 11:14:54 AM
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 COLUMN: CHIRAL PAK IC 250X4.6mm 5u
 DETECTION: 210nm
 FLOW RATE: 1mL/min



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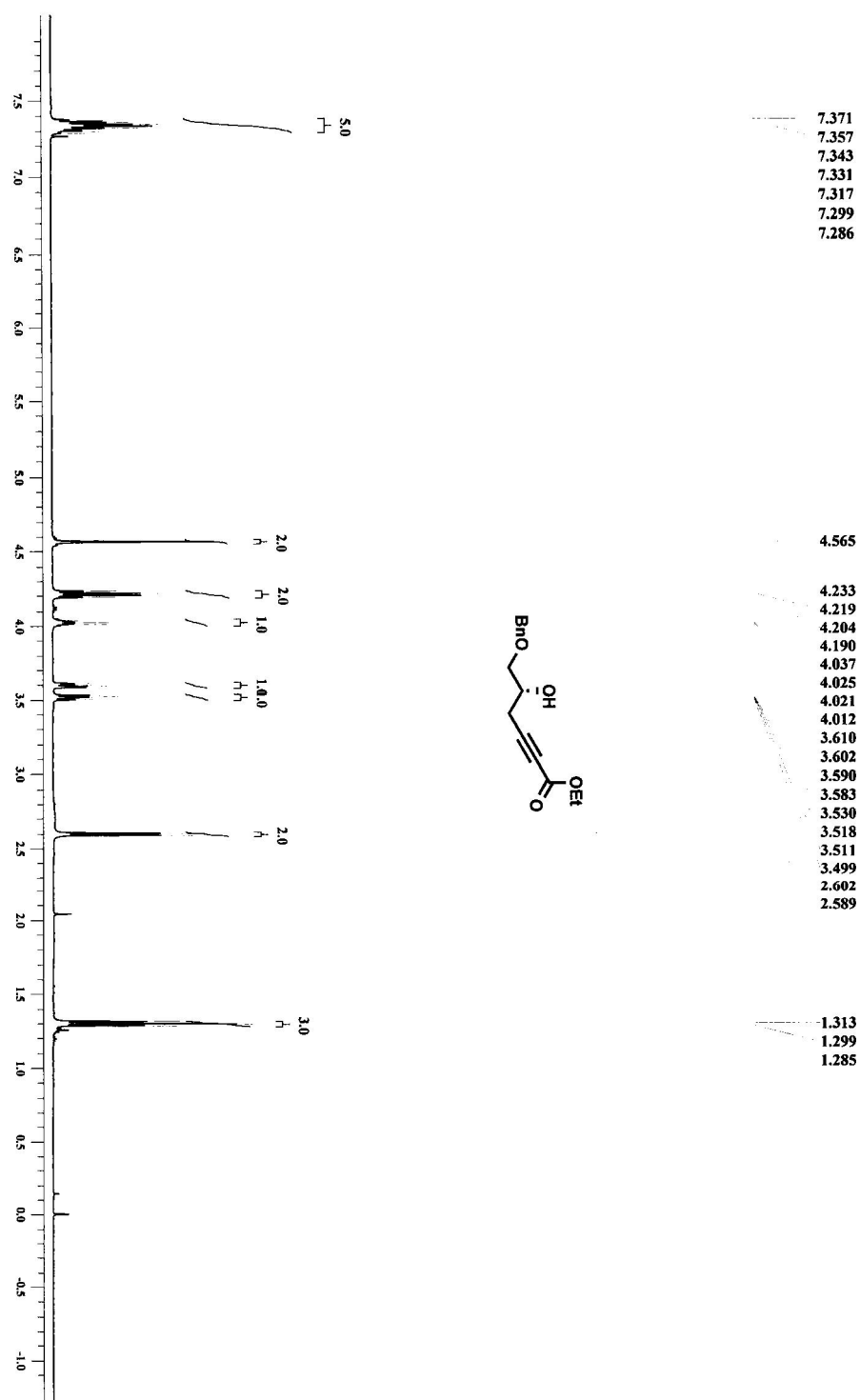


PeakTable

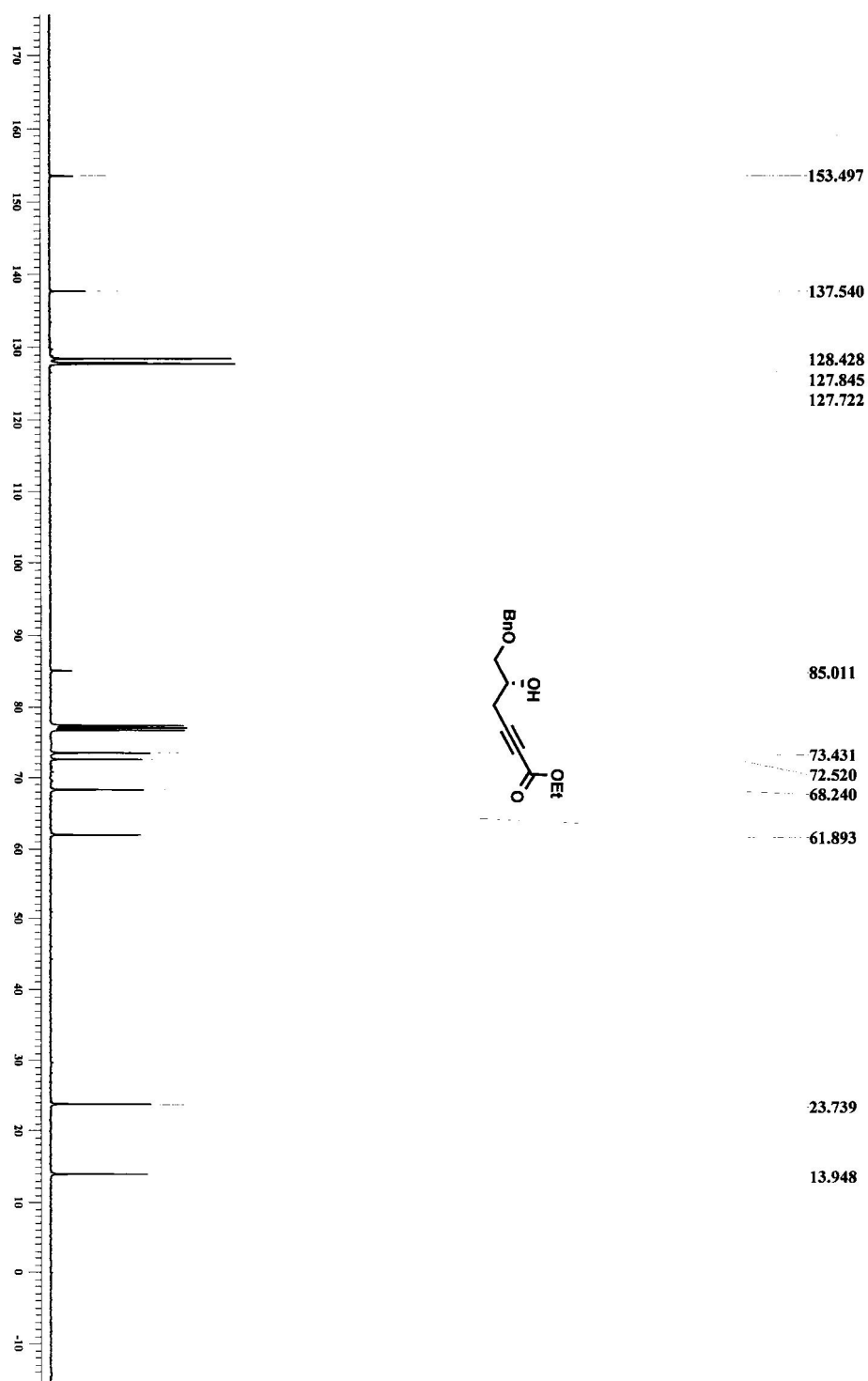
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Total		15147611	1055982	100.000	100.000

C:\LabSolutions\Data\Project1\DKM-GSR-BN-CHIRAL...3.lcd

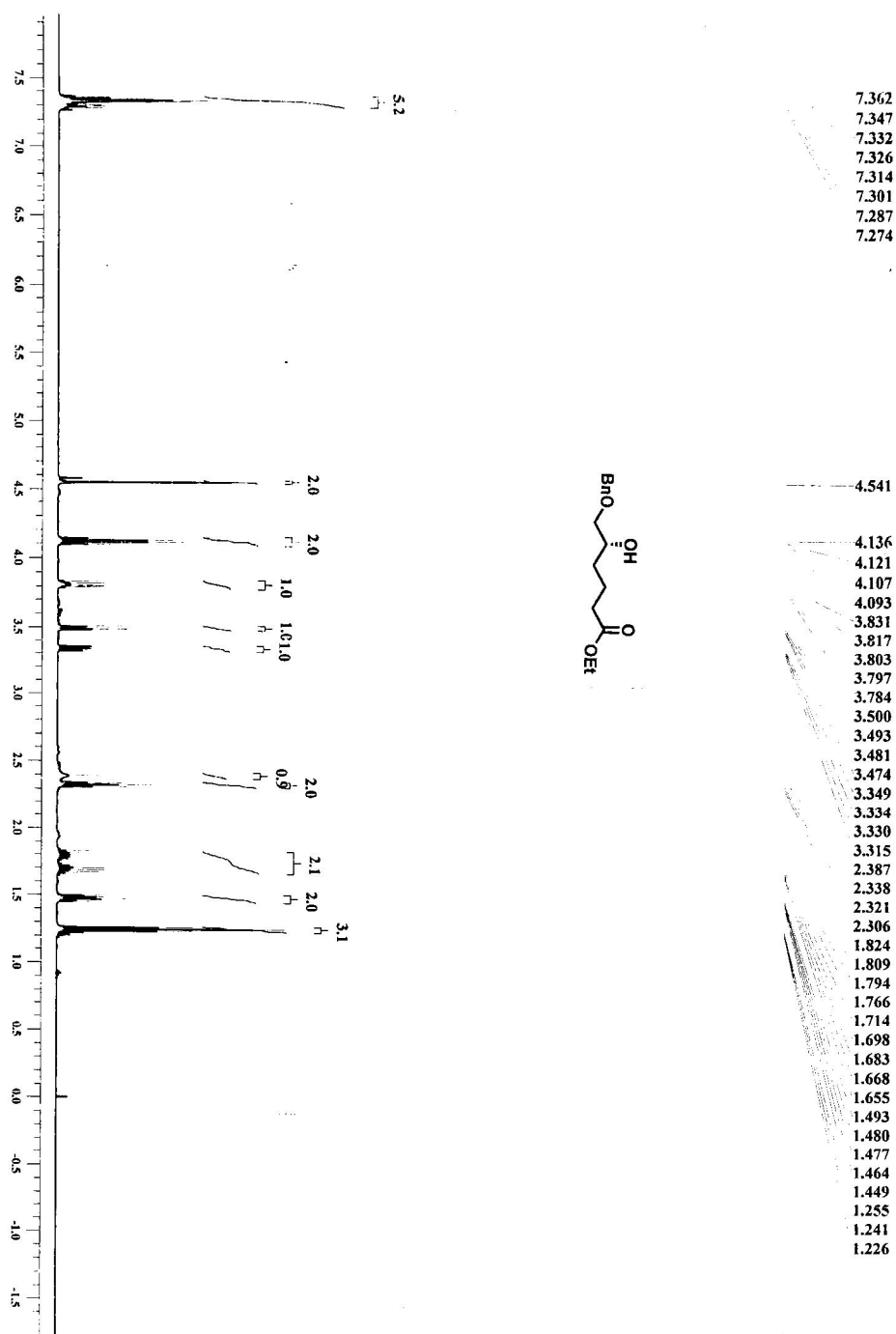
Chiral HPLC Report of 9



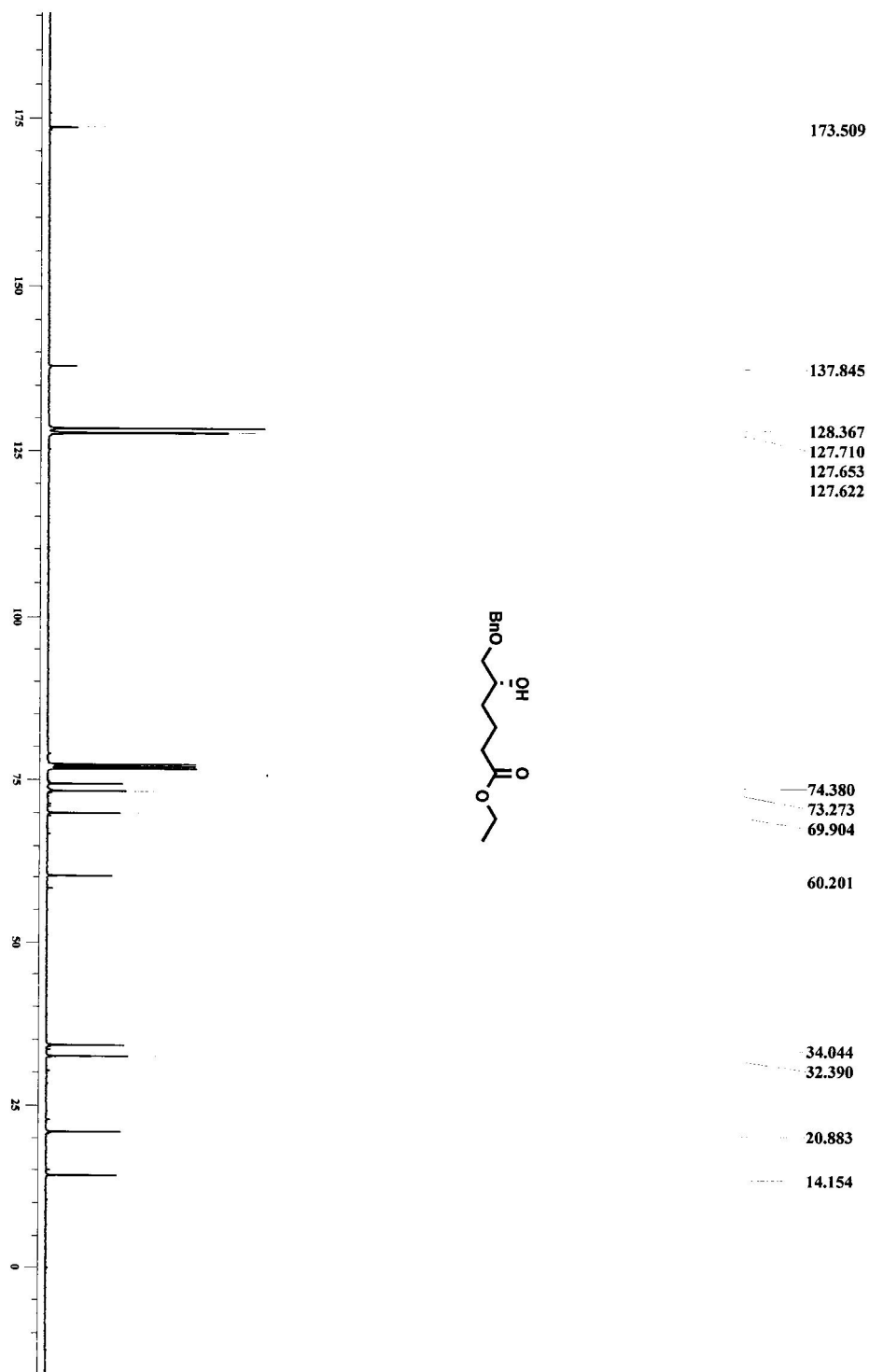
¹H NMR Spectrum of 10 (400 MHz, CDCl₃)



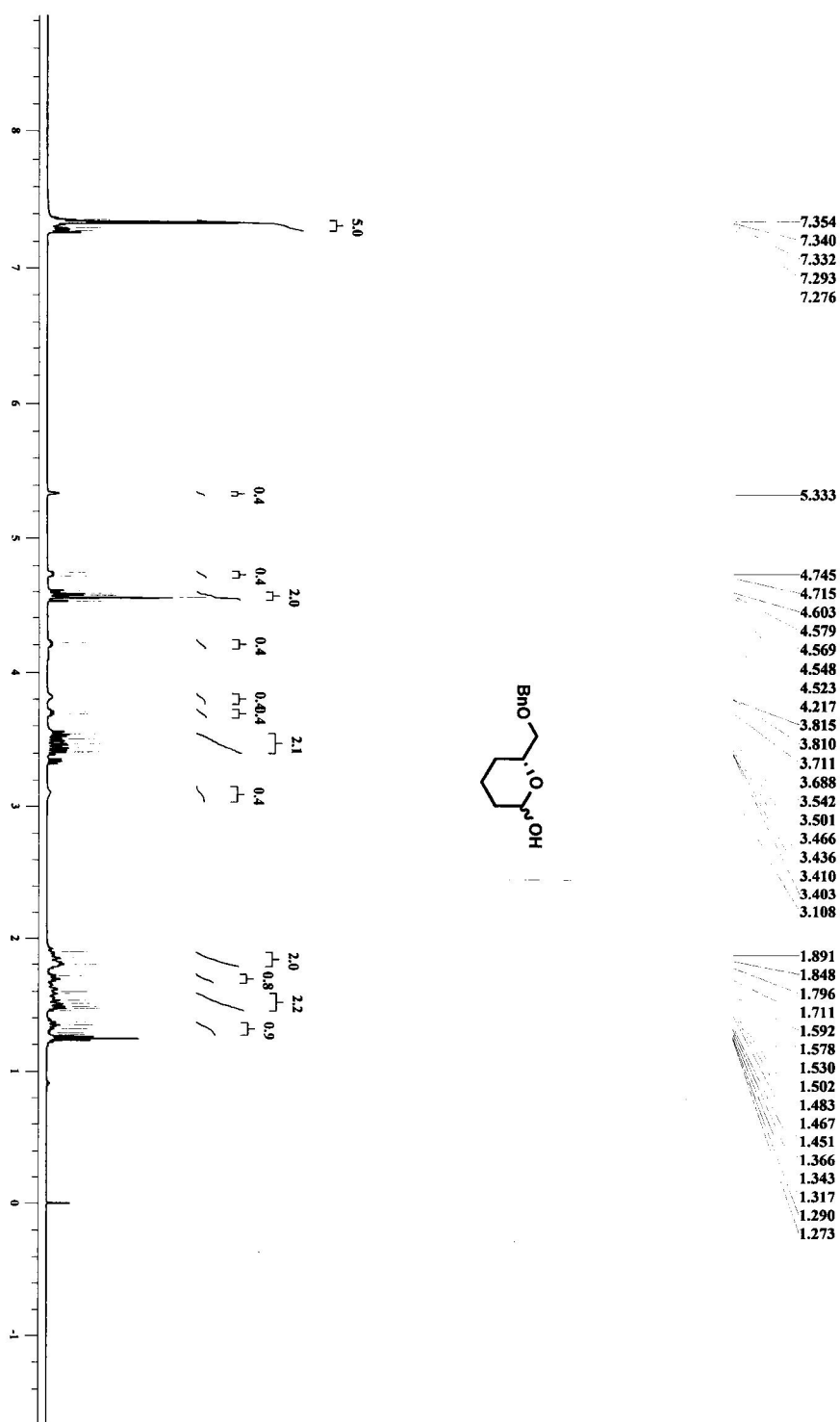
¹³C NMR Spectrum of 10 (125 MHz, CDCl₃)



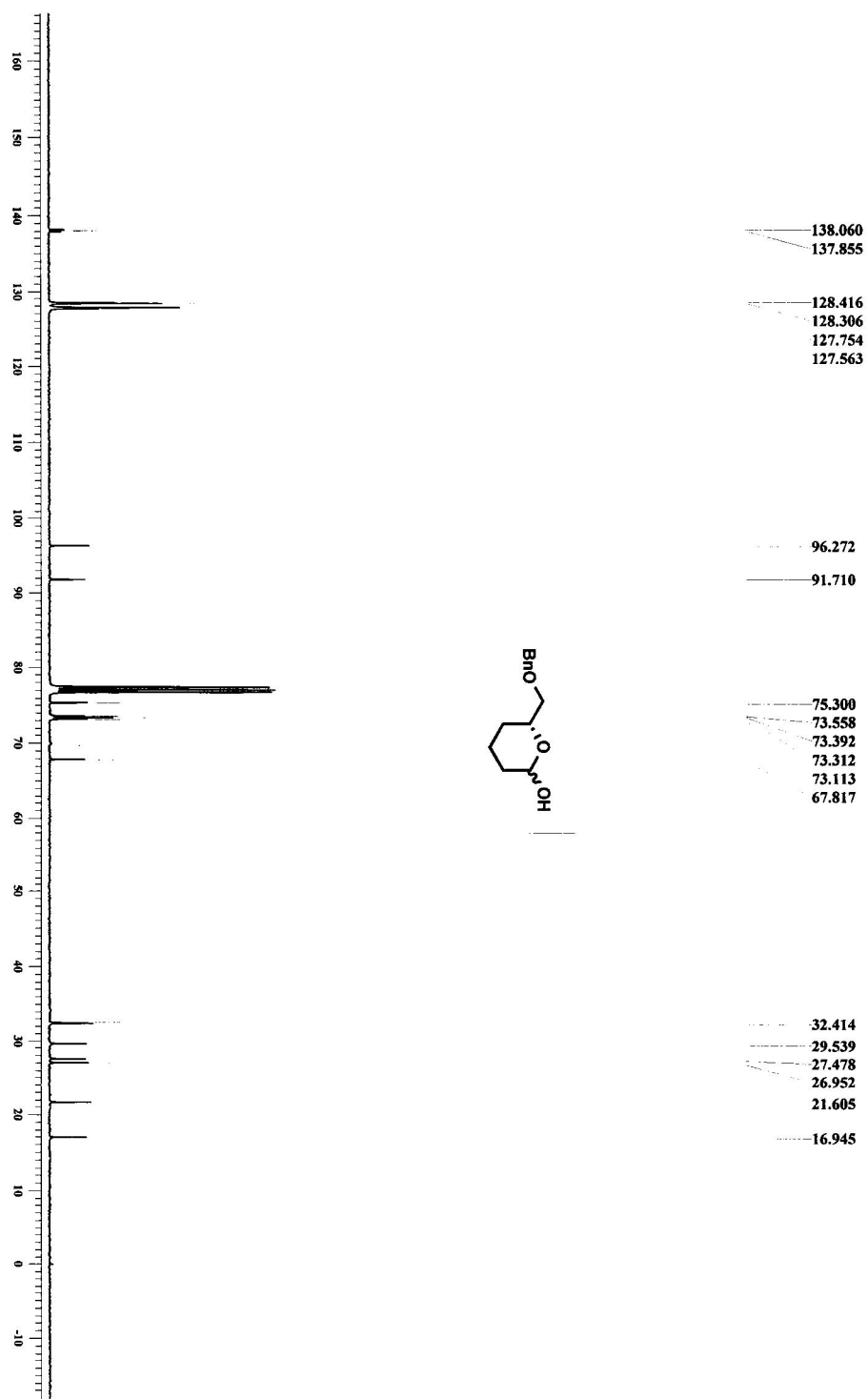
¹H NMR Spectrum of 11 (400 MHz, CDCl₃)



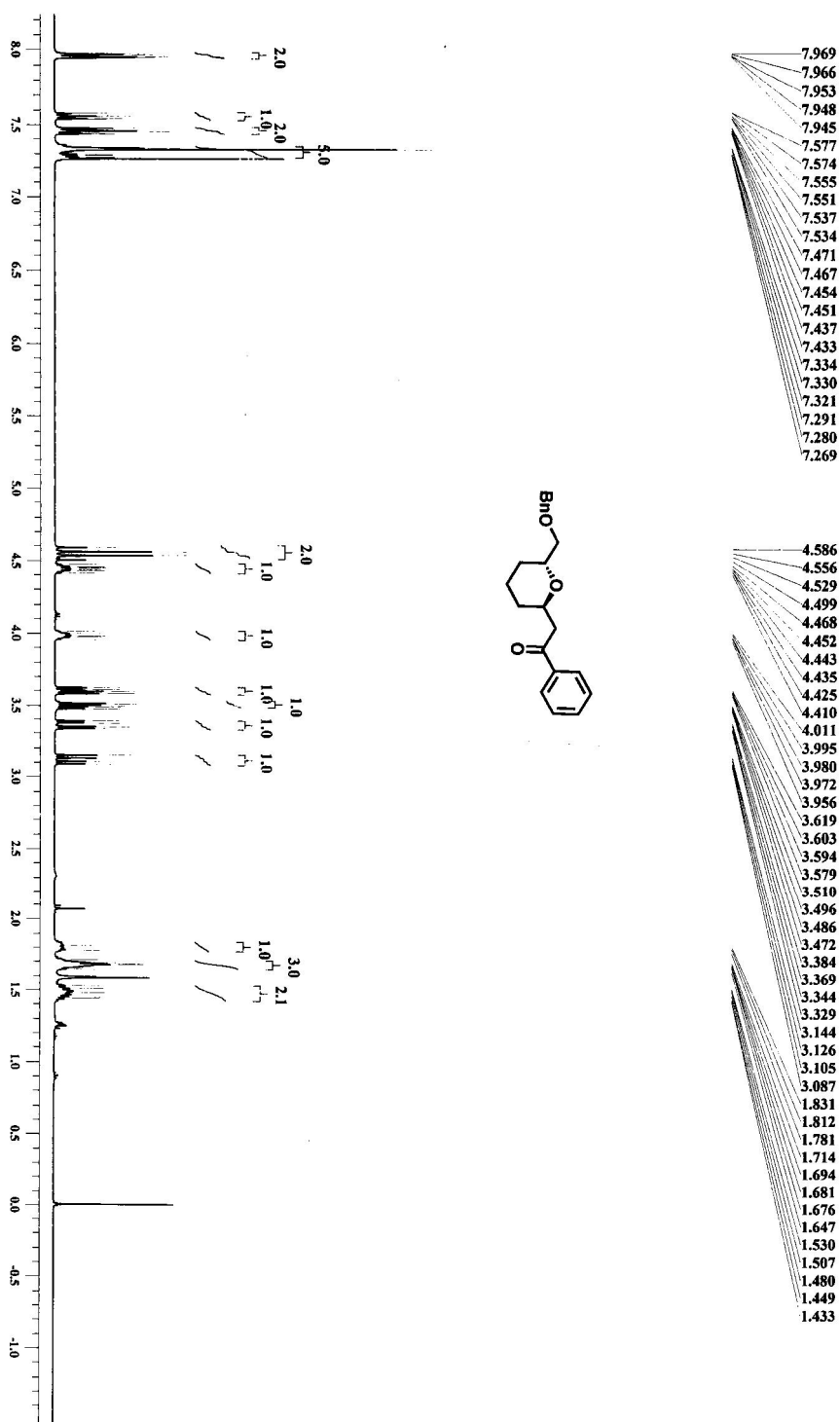
¹³C NMR Spectrum of 11 (125 MHz, CDCl₃)



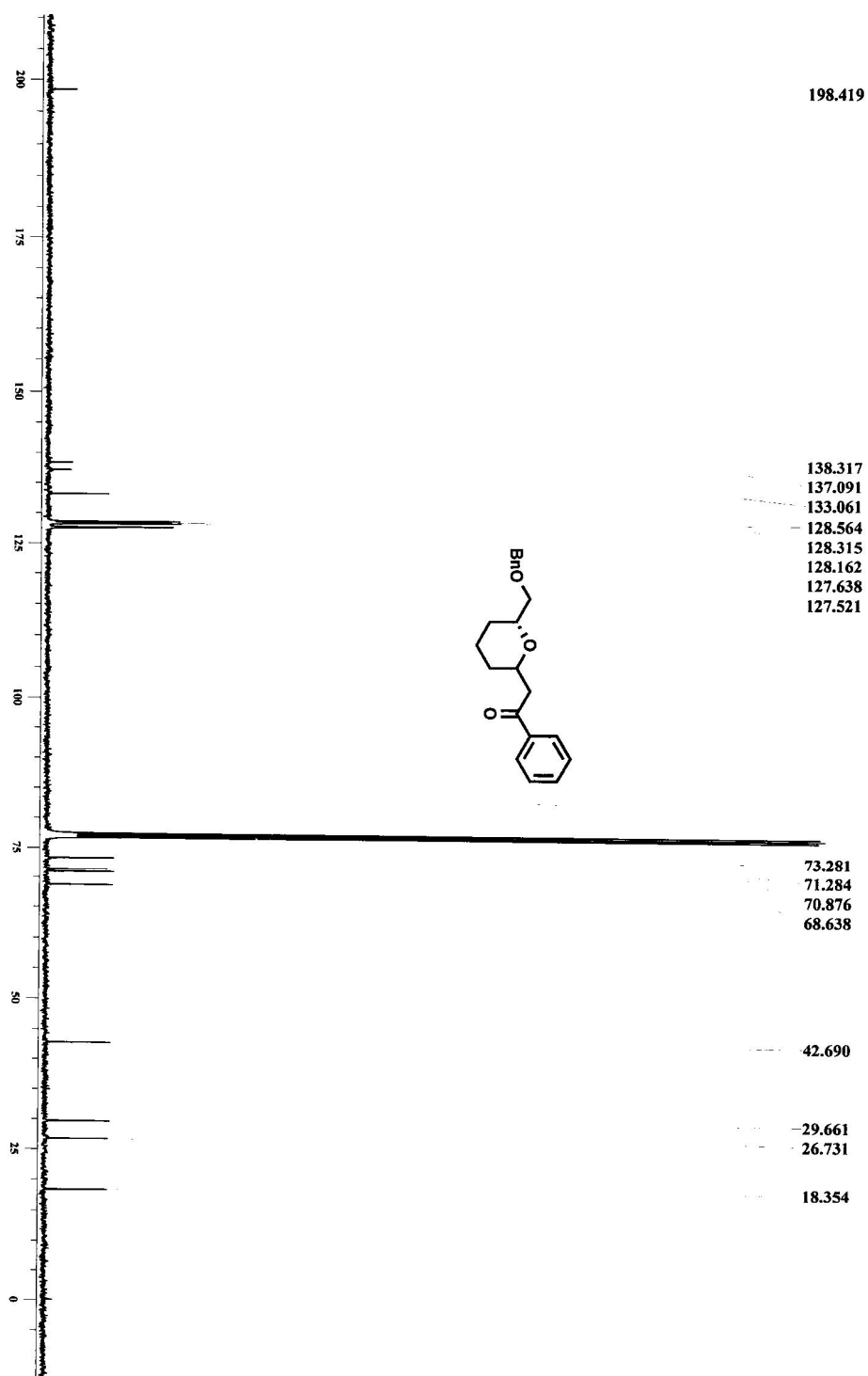
^1H NMR Spectrum of 12a (400 MHz, CDCl_3)



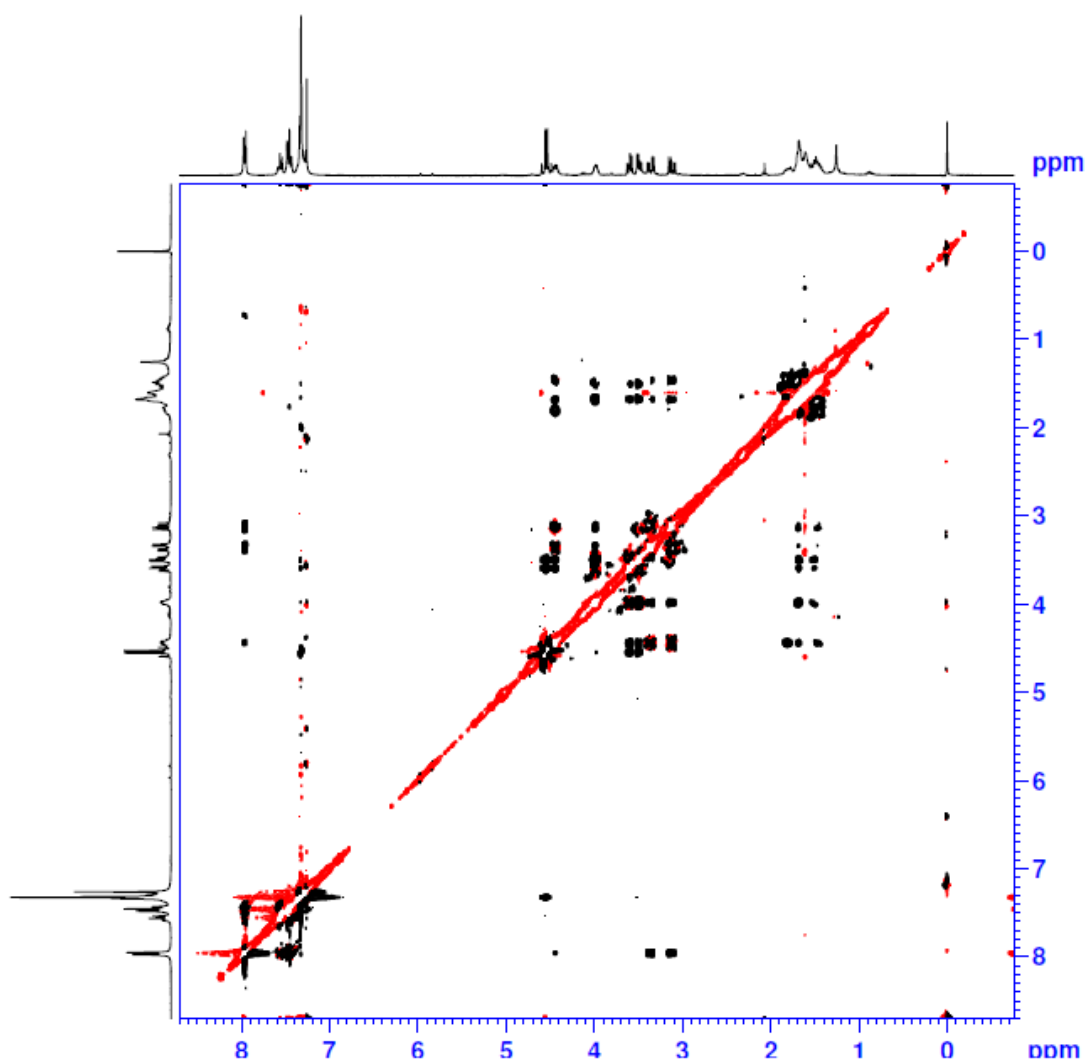
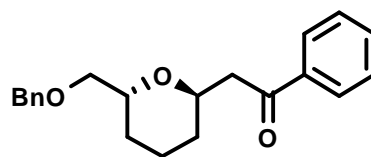
¹³C NMR Spectrum of 12a (75 MHz, CDCl₃)



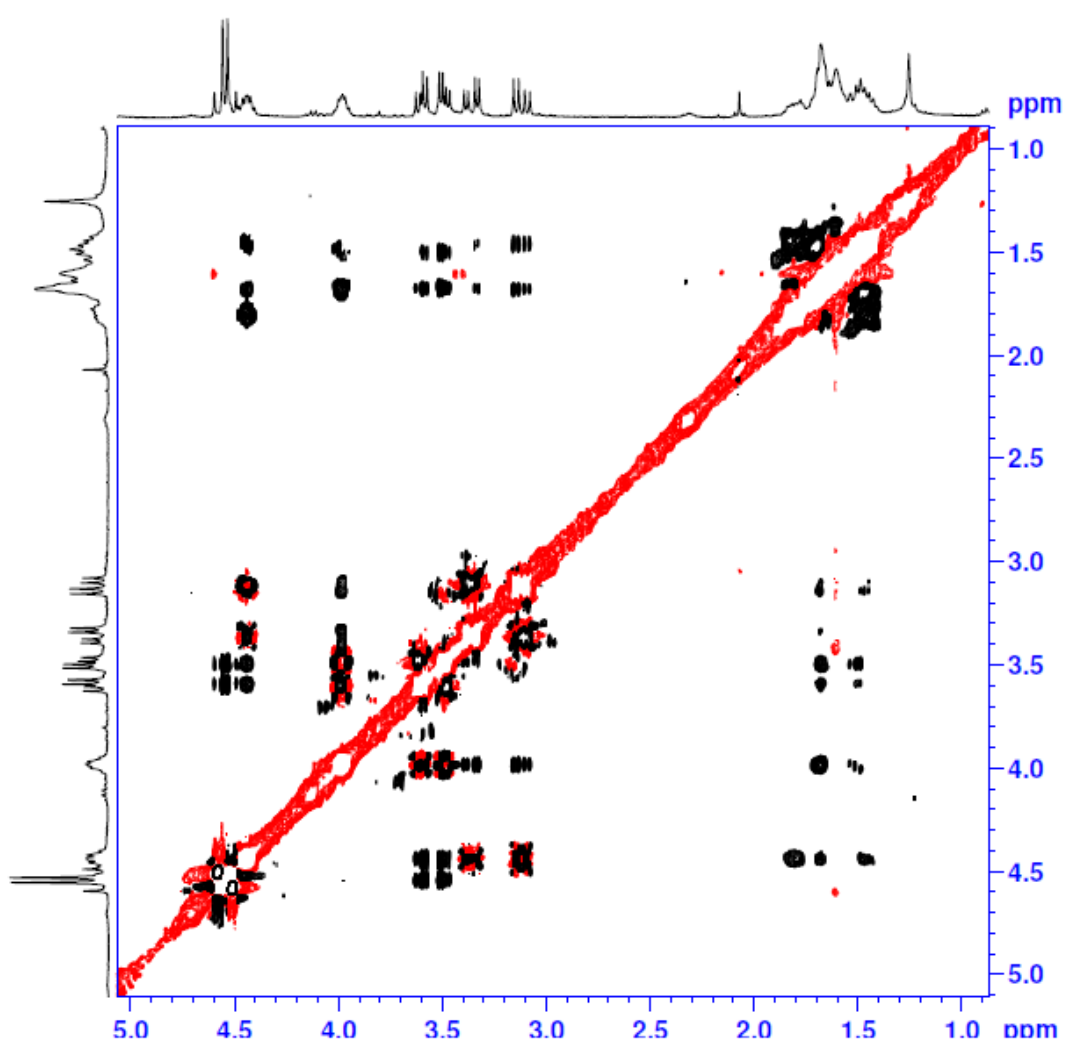
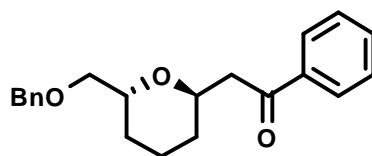
¹H NMR Spectrum of 13a (400 MHz, CDCl₃)



¹³C NMR Spectrum of 13a (100 MHz, CDCl₃)

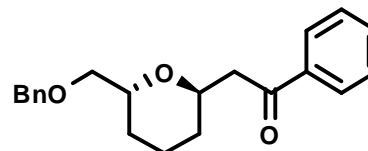


NOESY of Compound 13a (300 MHz, CDCl₃)

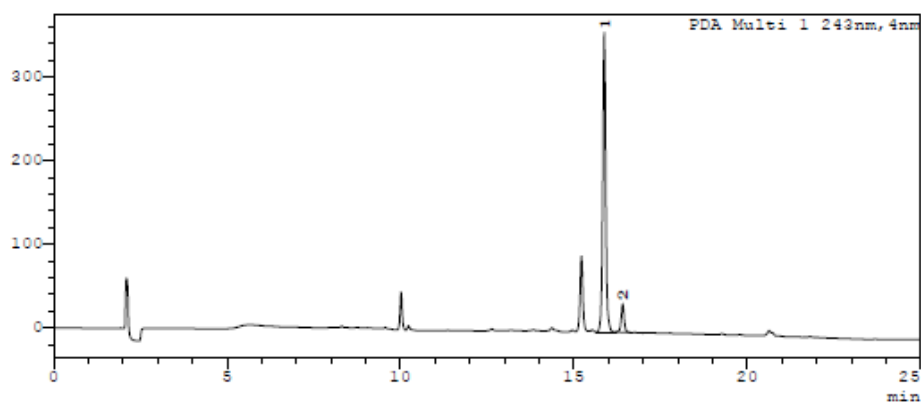


Expanded NOESY of Compound 13a (300 MHz, CDCl₃)

Sample Code : DKM-B-TMS-324
 Data File : 041218.2.1cd
 Method : CD HPLC.lcm
 Injection Volume : 5
 Date Acquired : 12/4/2018 3:38:33 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Chromatographic Conditions :
 Colum: X-SELECT (150 X 4.6mm, 5.0u)
 Mobile Phase:GRADIENT



mAU



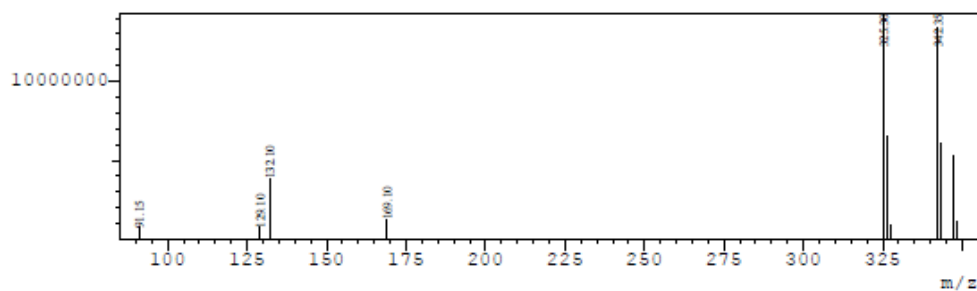
Peak Table

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2	16.412	16.213	16.672	201074	8.581
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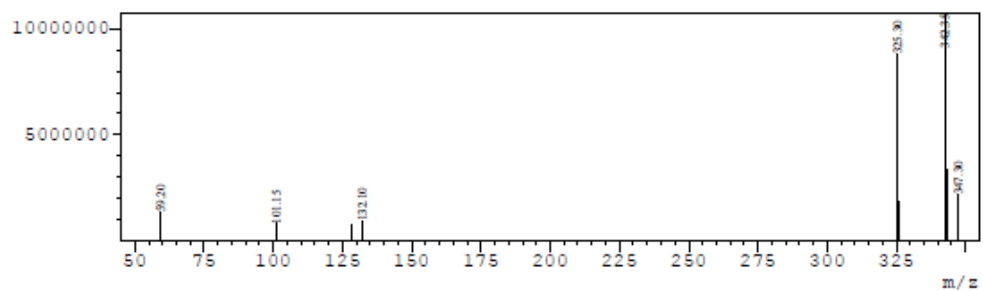
BG Mode:Averaged 0.000-15.472 (1-5627)



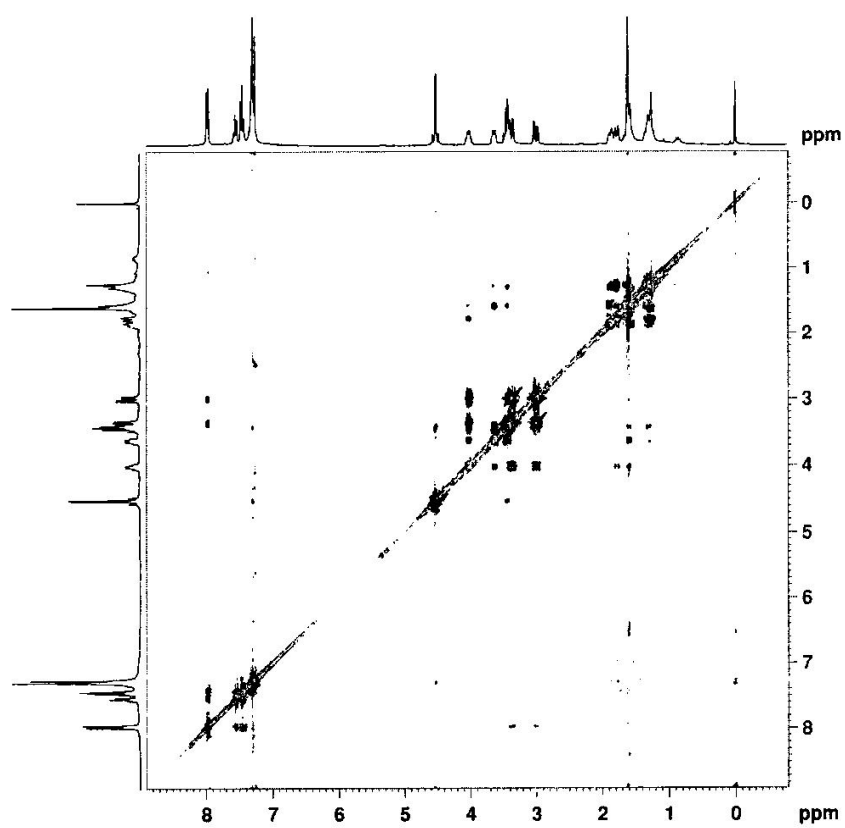
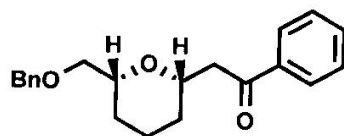
LC-LCMS Chromatogram of 13a

Q1 Scan Positive+
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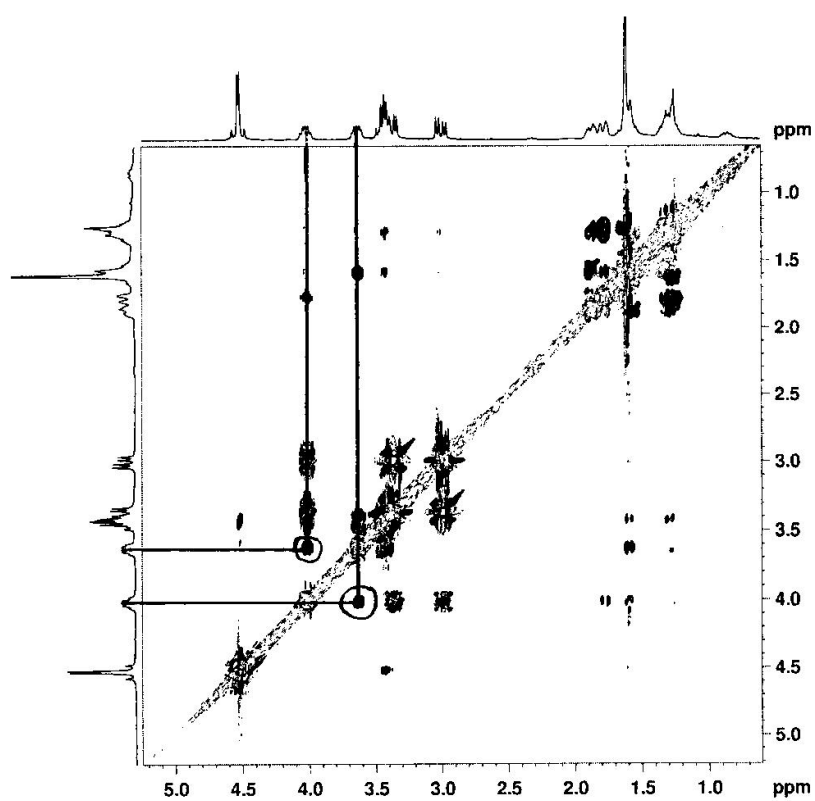
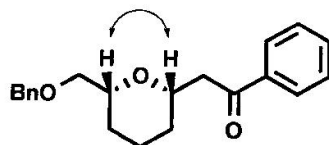
UCT-DNPG



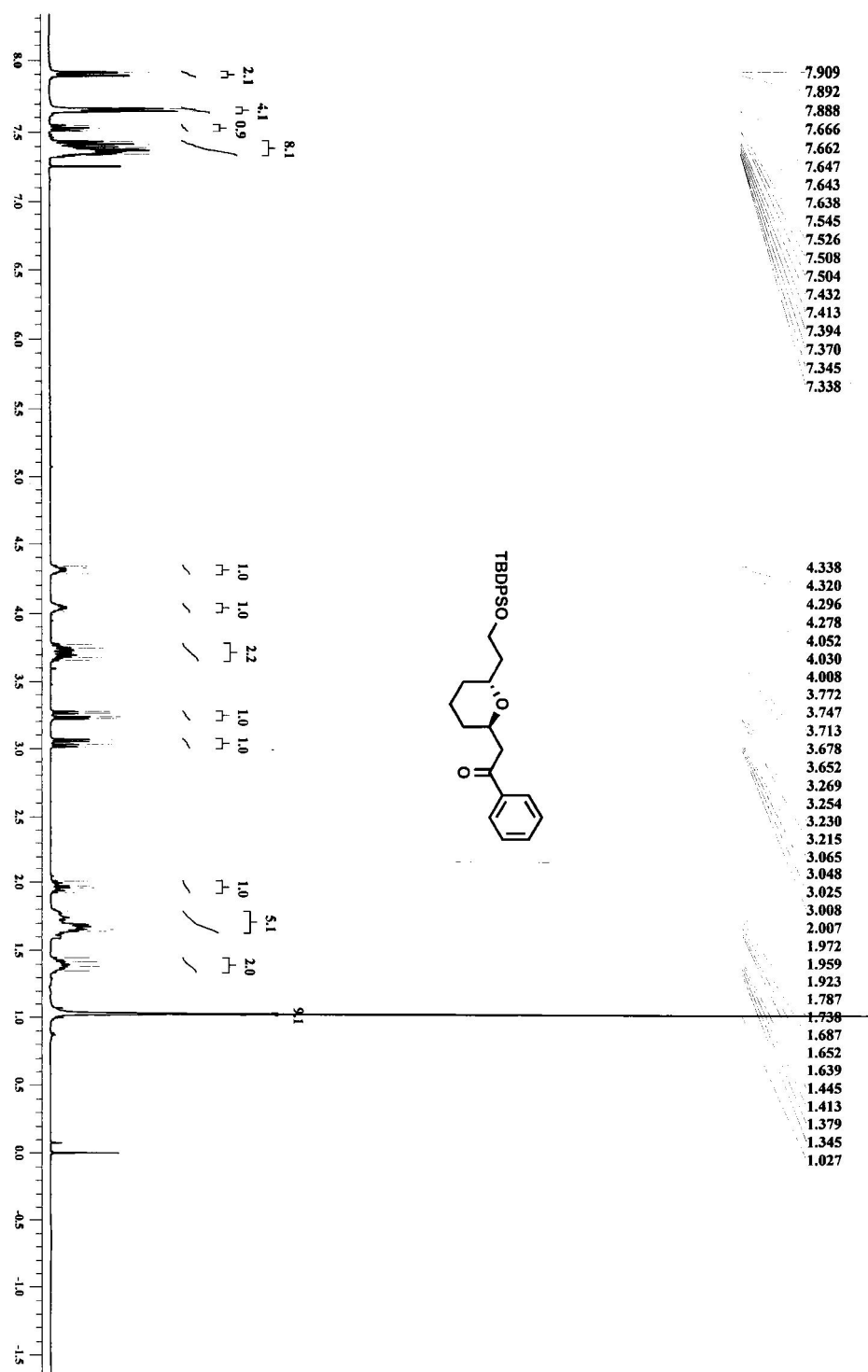
LC-LCMS Chromatogram of 13a

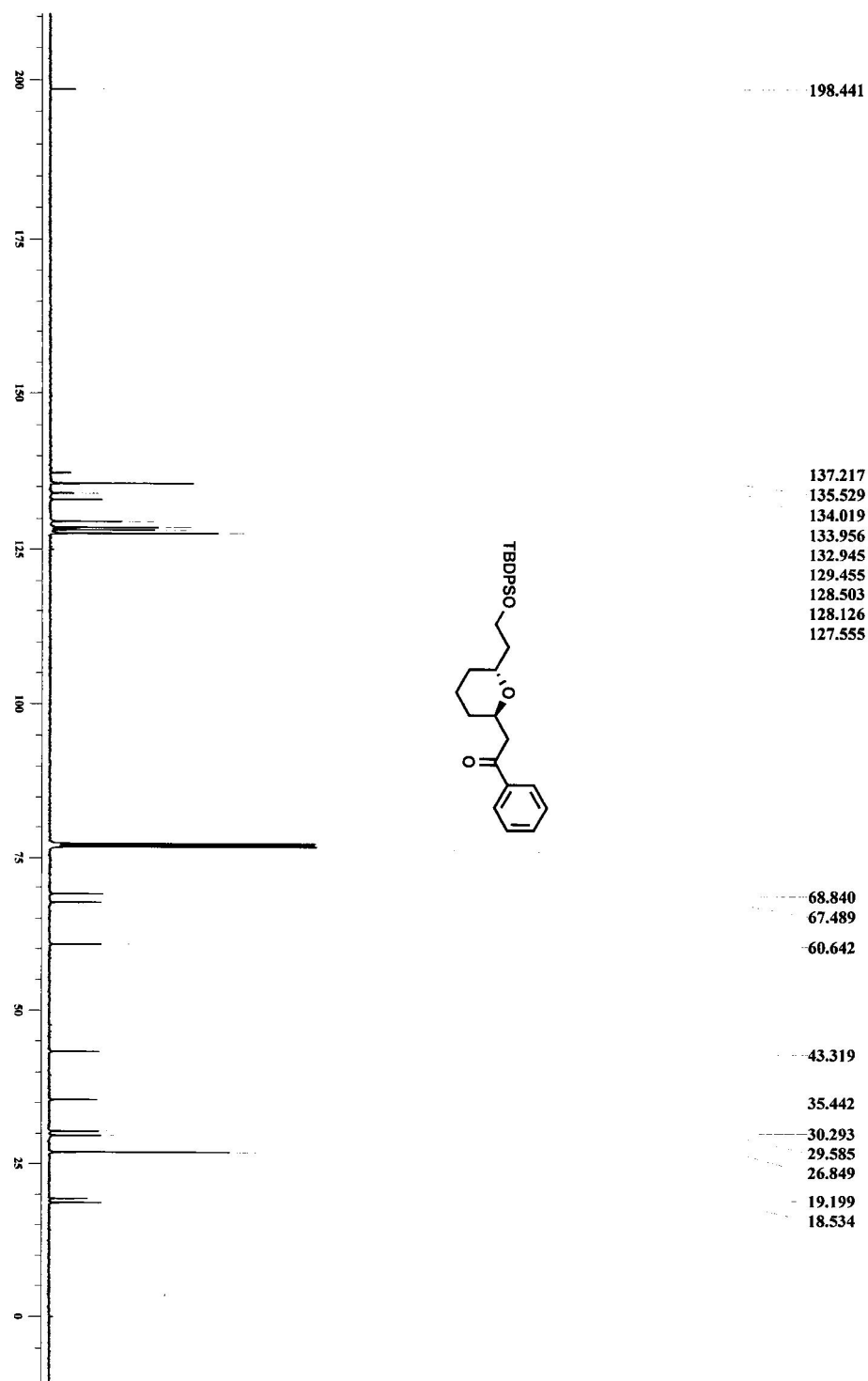


NOESY of Compound 13a' (300 MHz, CDCl₃)

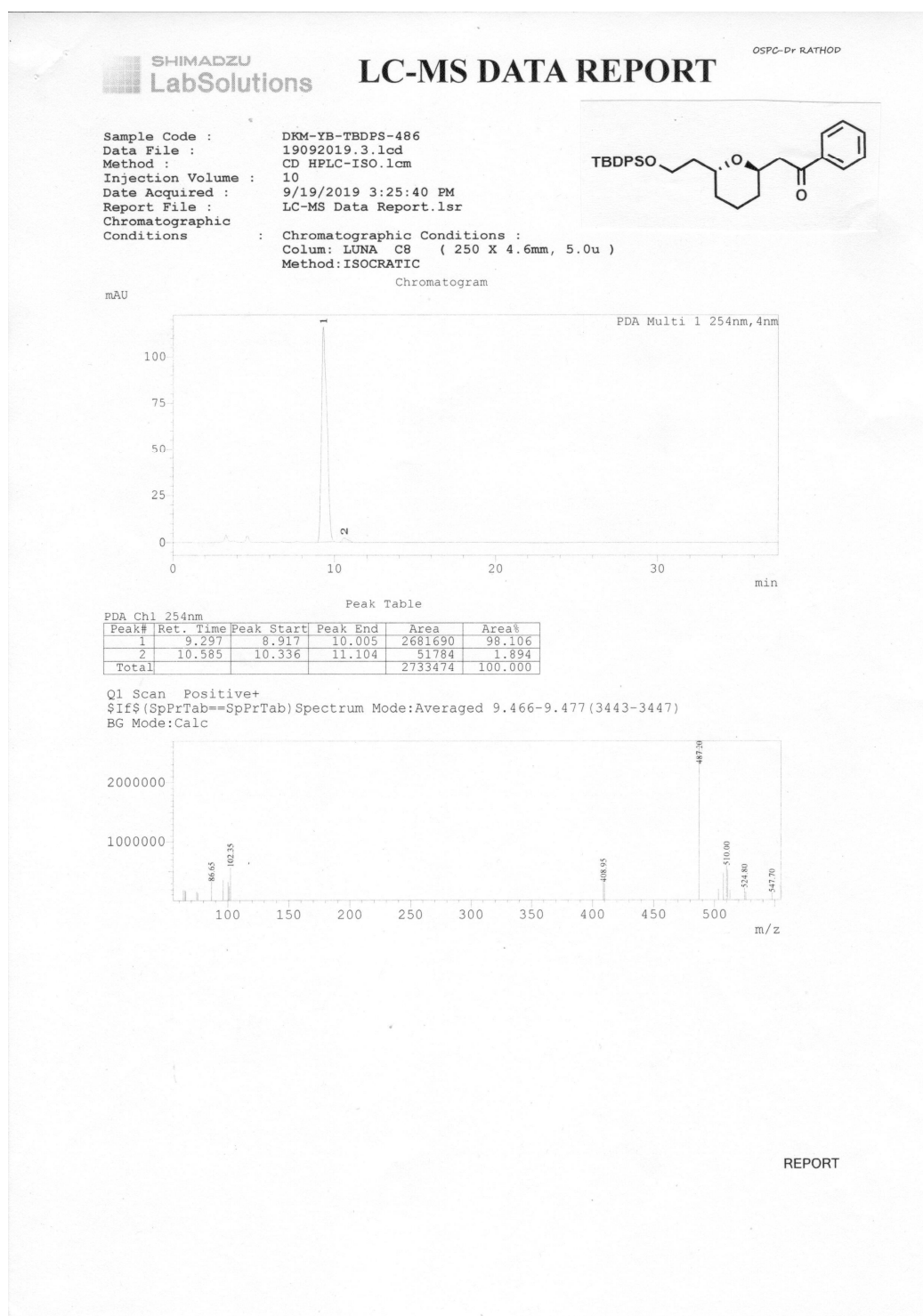


Expanded NOESY of Compound 13a' (300 MHz, CDCl₃)

¹H NMR Spectrum of 13b (500 MHz, CDCl₃)



¹³C NMR Spectrum of 13b (125 MHz, CDCl₃)

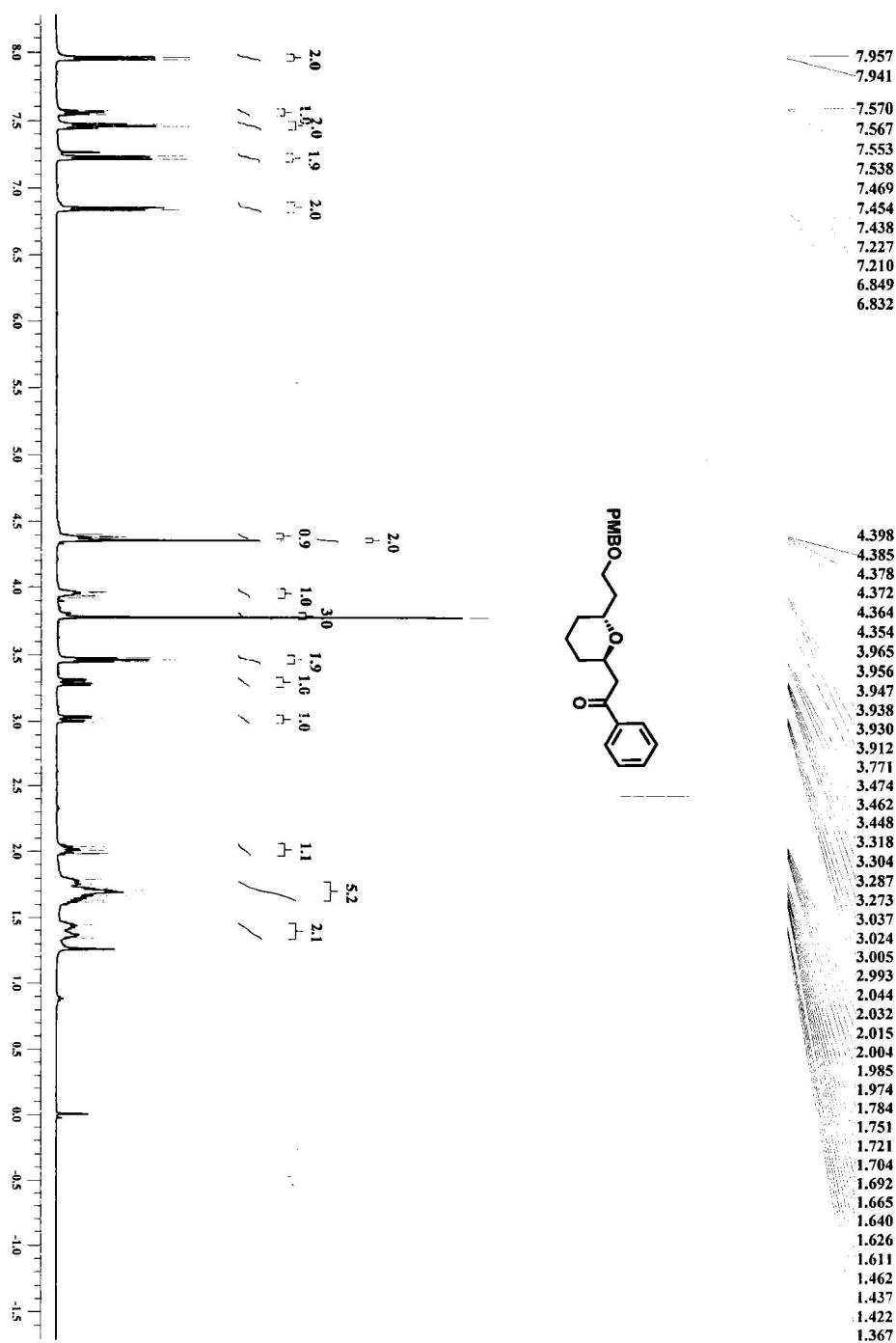


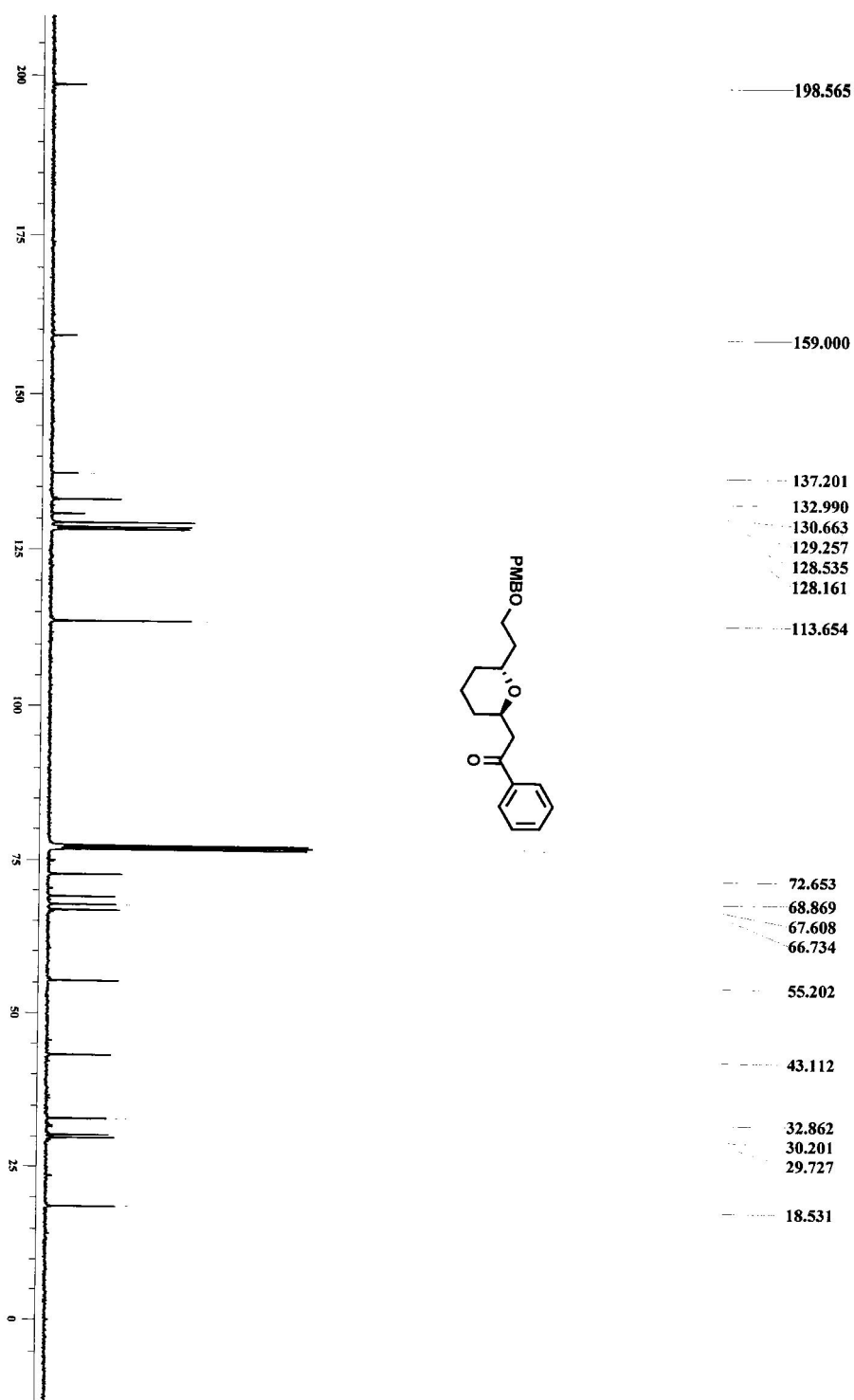
LC-LCMS Chromatogram of 13b



LC-MS REPORT

LC-LCMS Chromatogram of 13b

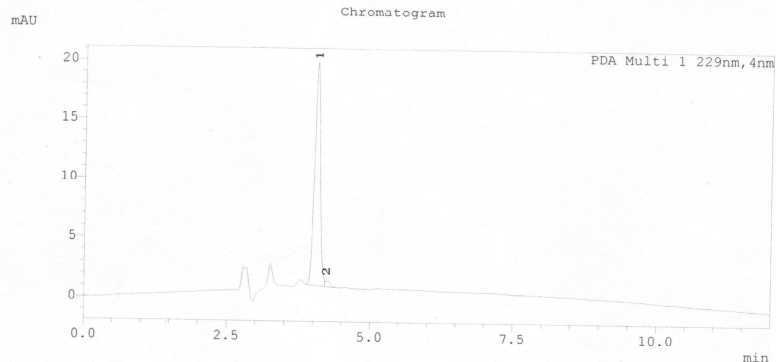
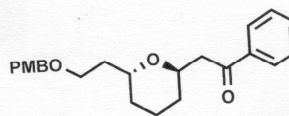
¹H NMR Spectrum of 13c (500 MHz, CDCl₃)



^{13}C NMR Spectrum of 13c (100 MHz, CDCl_3)

Sample Code : DKM-YB-136-368
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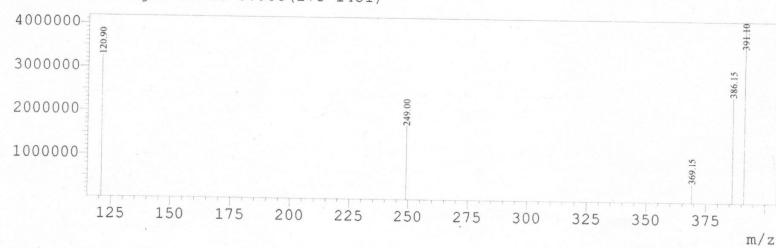
Chromatographic Conditions :
 Column : LUNA C8 (250 X 4.6mm, 5.0u)
 Mobile phase : A : 0.1% Formic acid In 100 ML water
 Mobile phase : B : 90% MEOH
 METHOD : ISOCRATIC



Peak Table

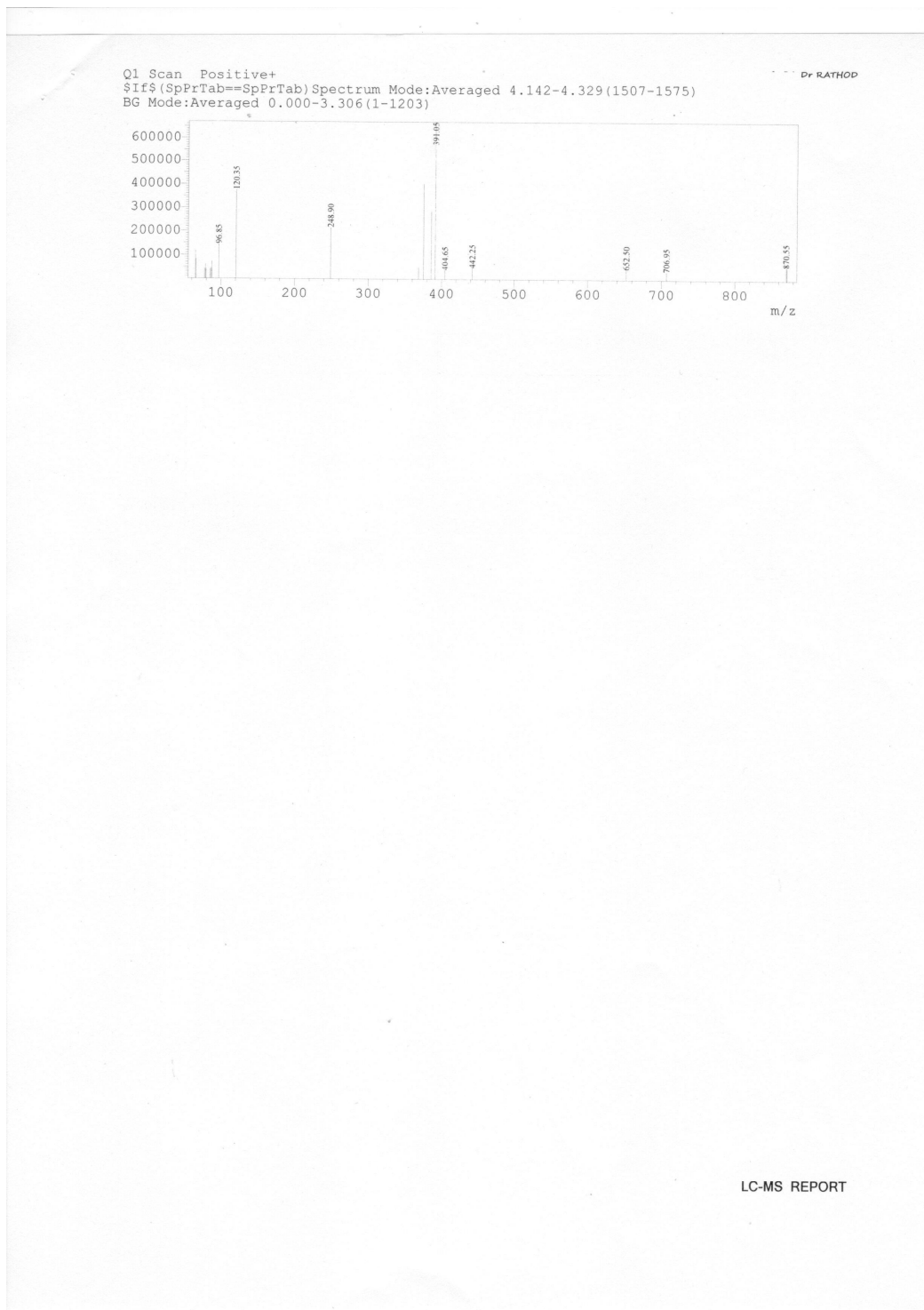
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2	4.219	4.203	4.405	2856	2.136
Total				133690	100.000

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 BG Mode:Averaged 0.748-3.933(273-1431)

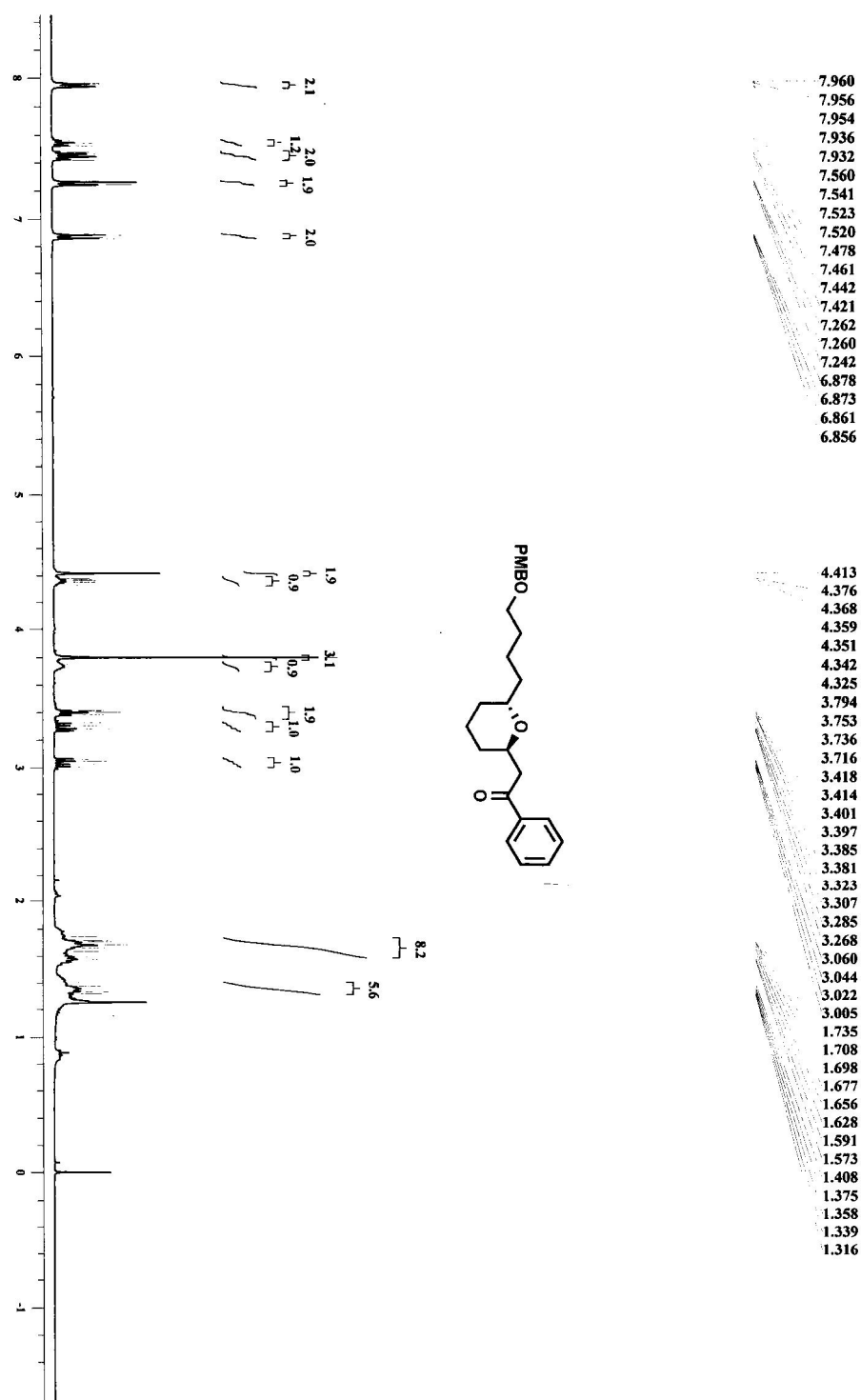


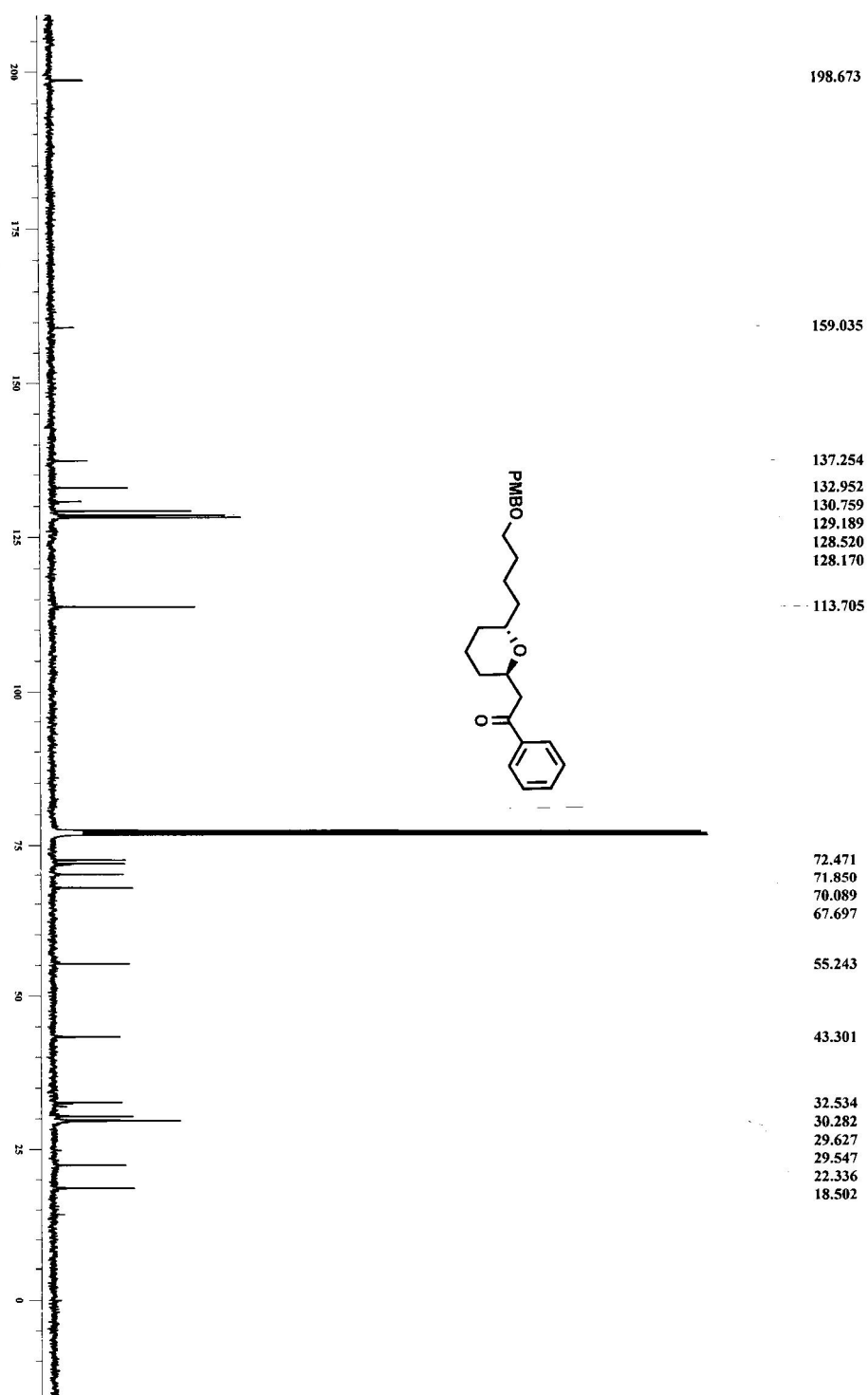
REPORT

LC-LCMS Chromatogram of 13c

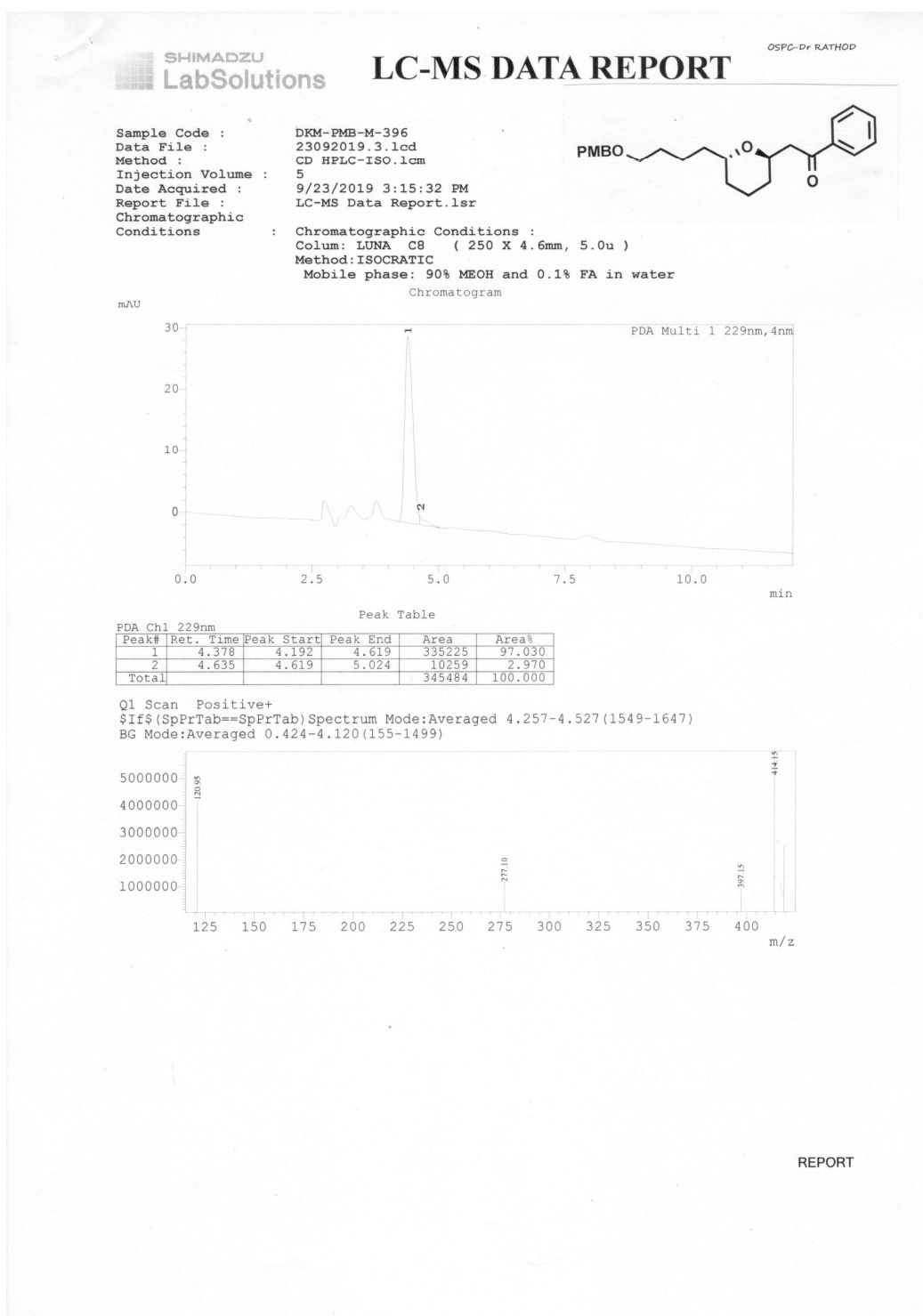


LC-LCMS Chromatogram of 13c

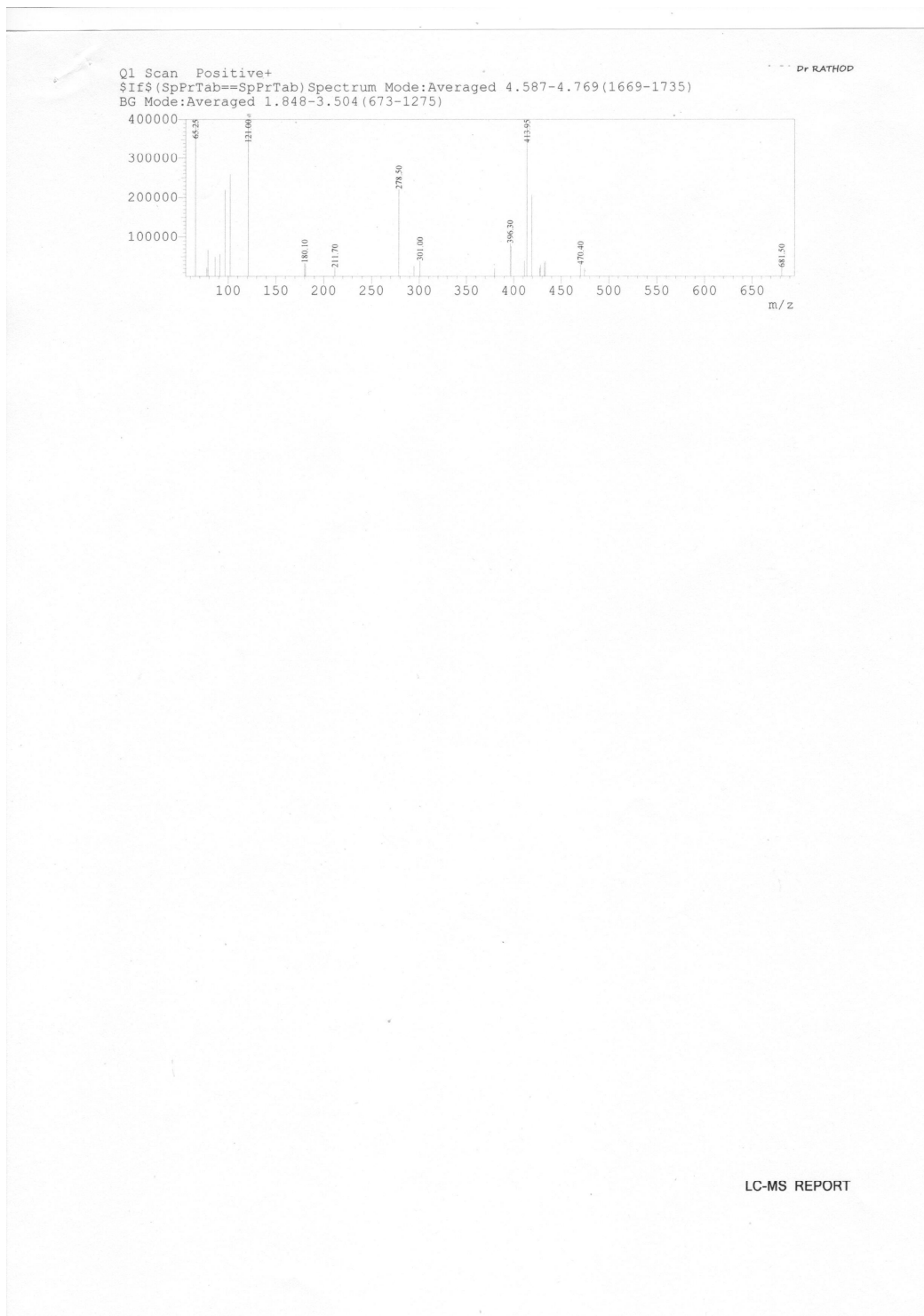
¹H NMR Spectrum of 13d (400 MHz, CDCl₃)



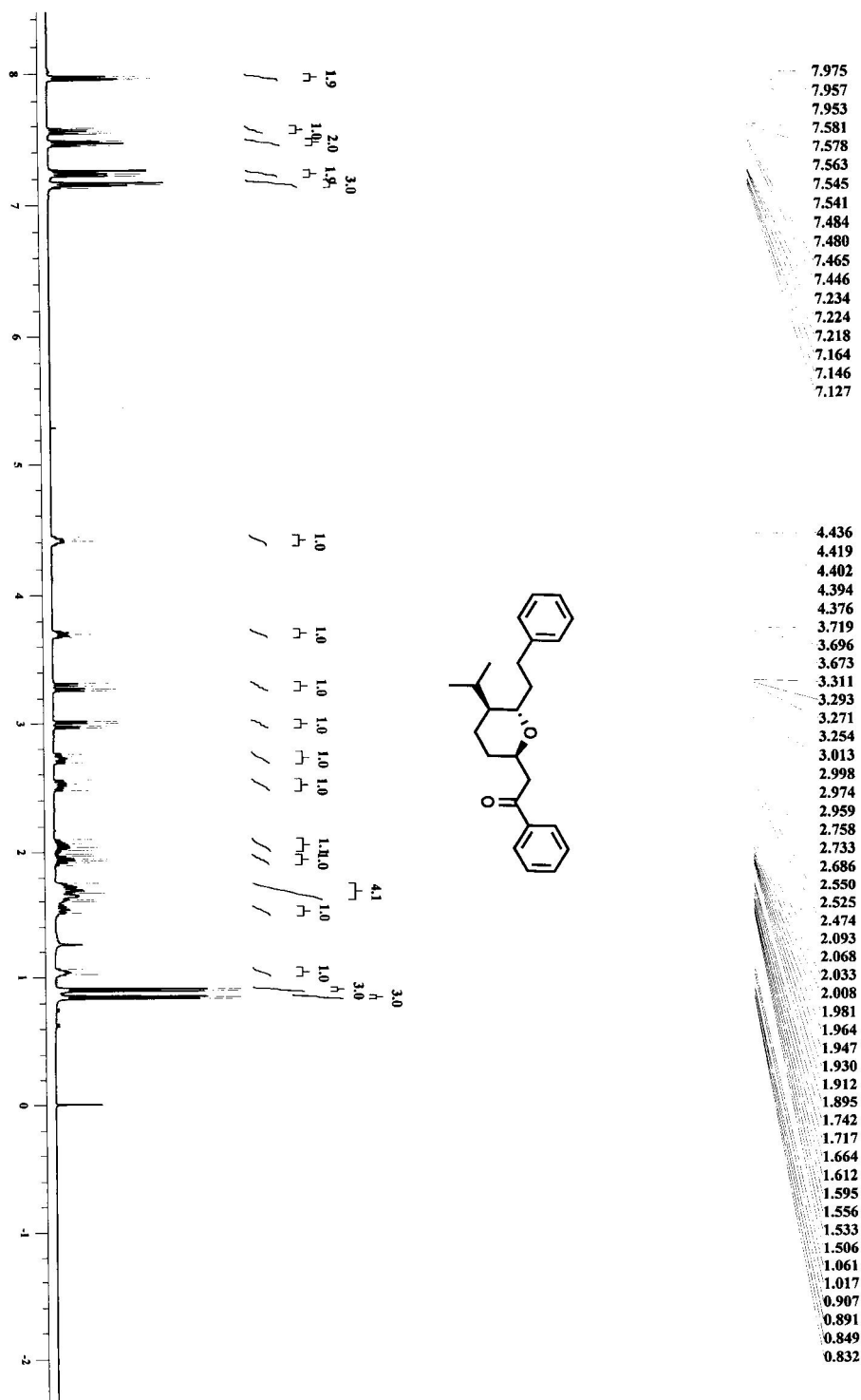
¹³C NMR Spectrum of 13d (100 MHz, CDCl₃)



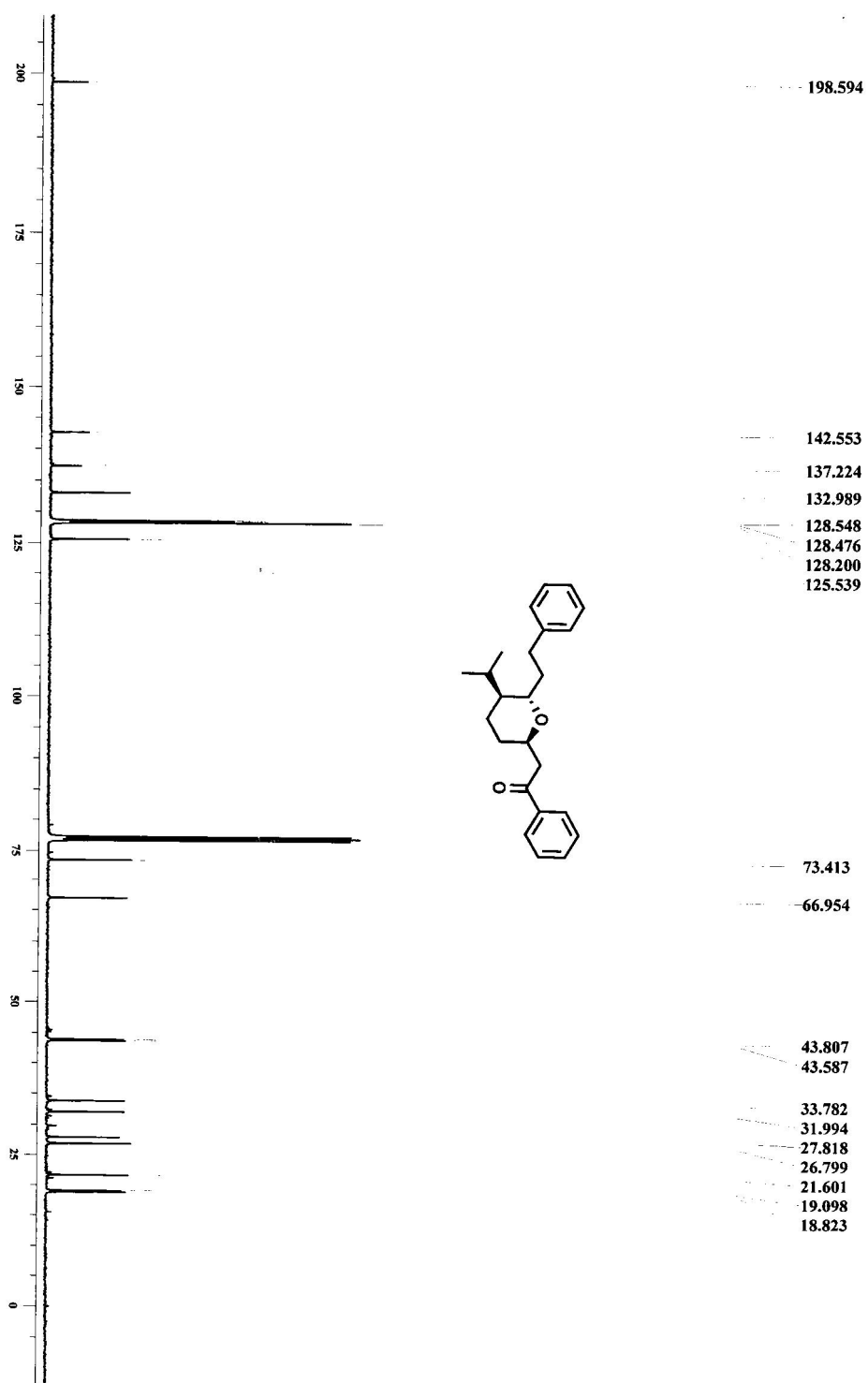
LC-LCMS Chromatogram of 13d



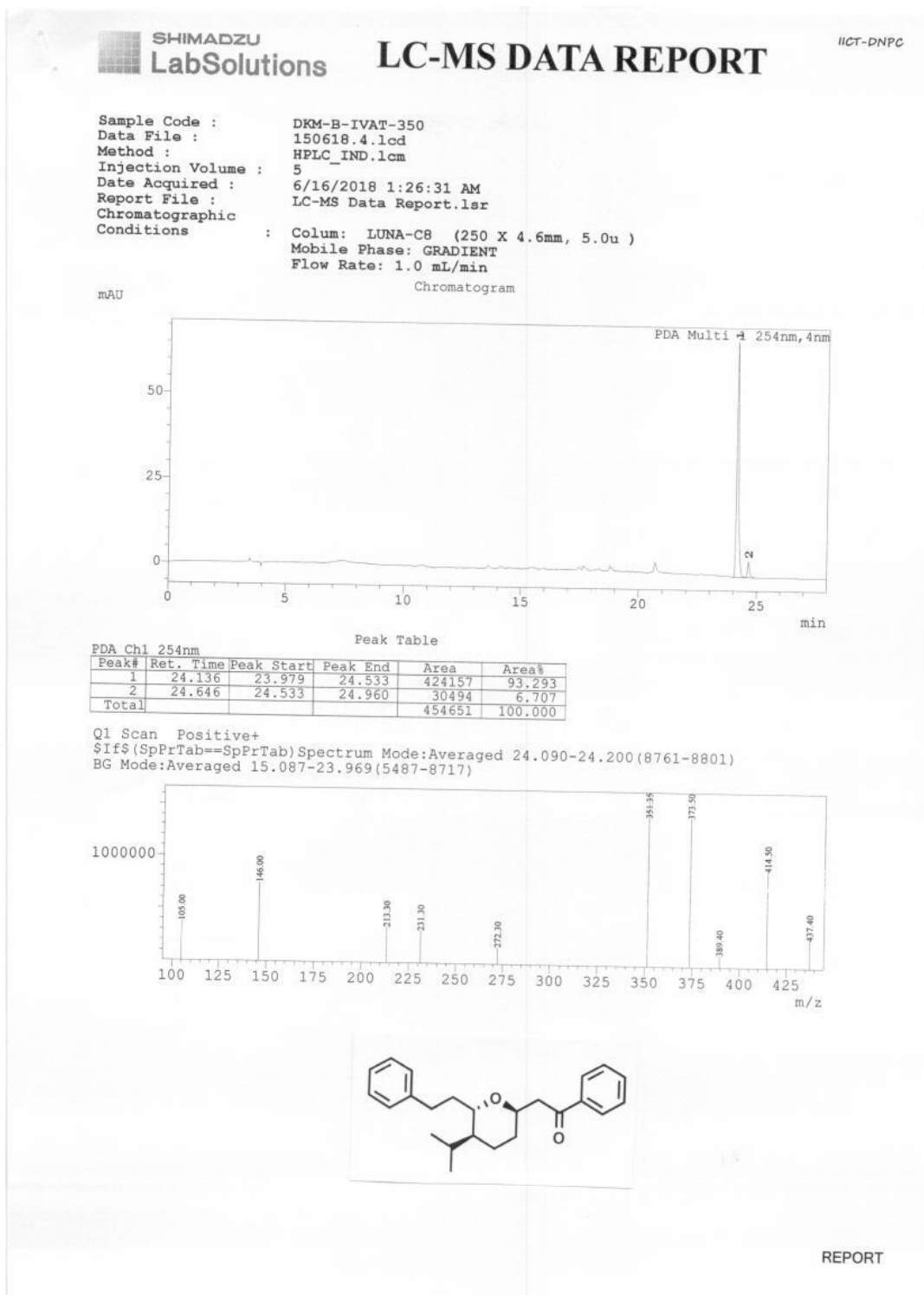
LC-LCMS Chromatogram of 13d



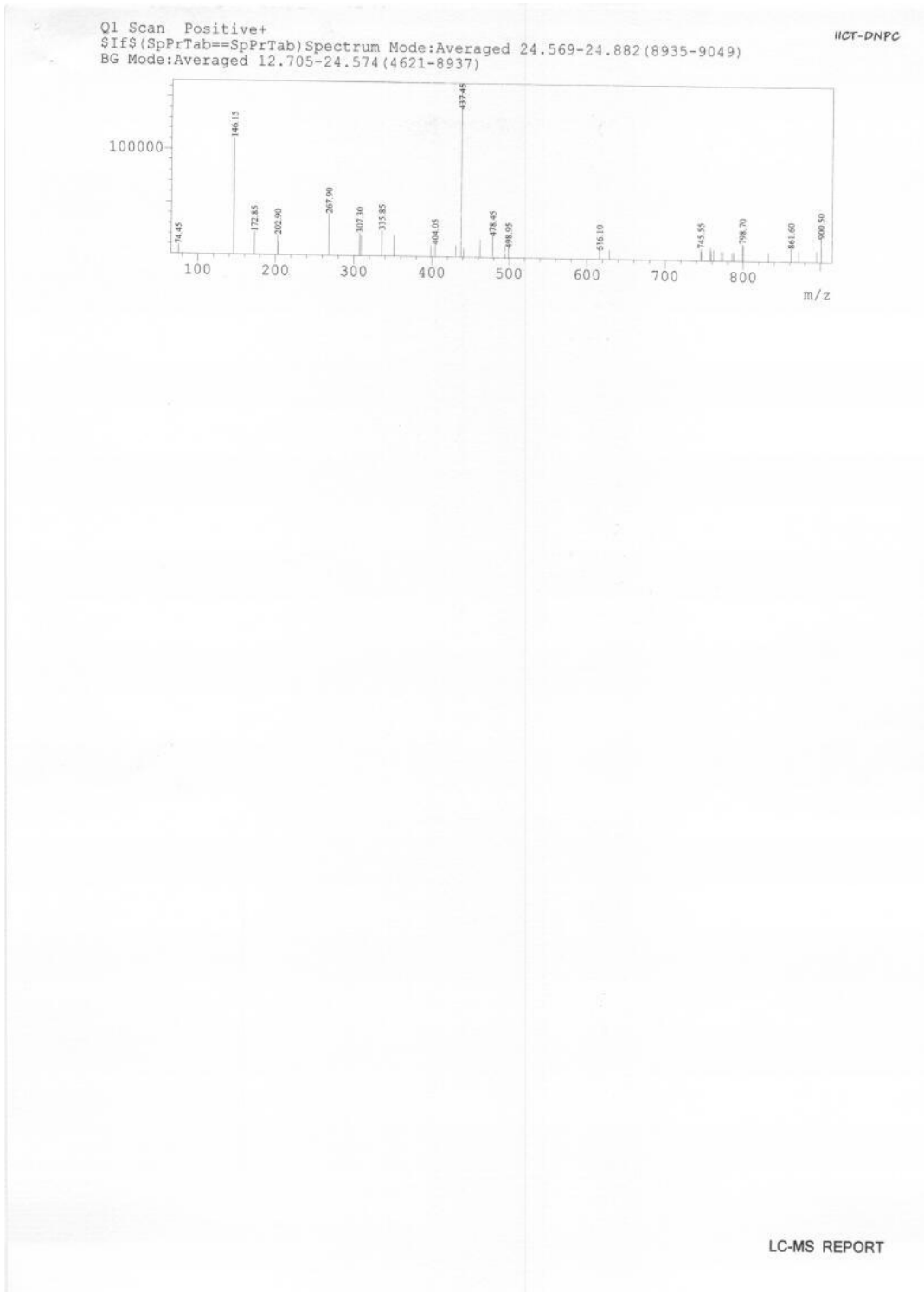
¹H NMR Spectrum of 13e (400 MHz, CDCl₃)



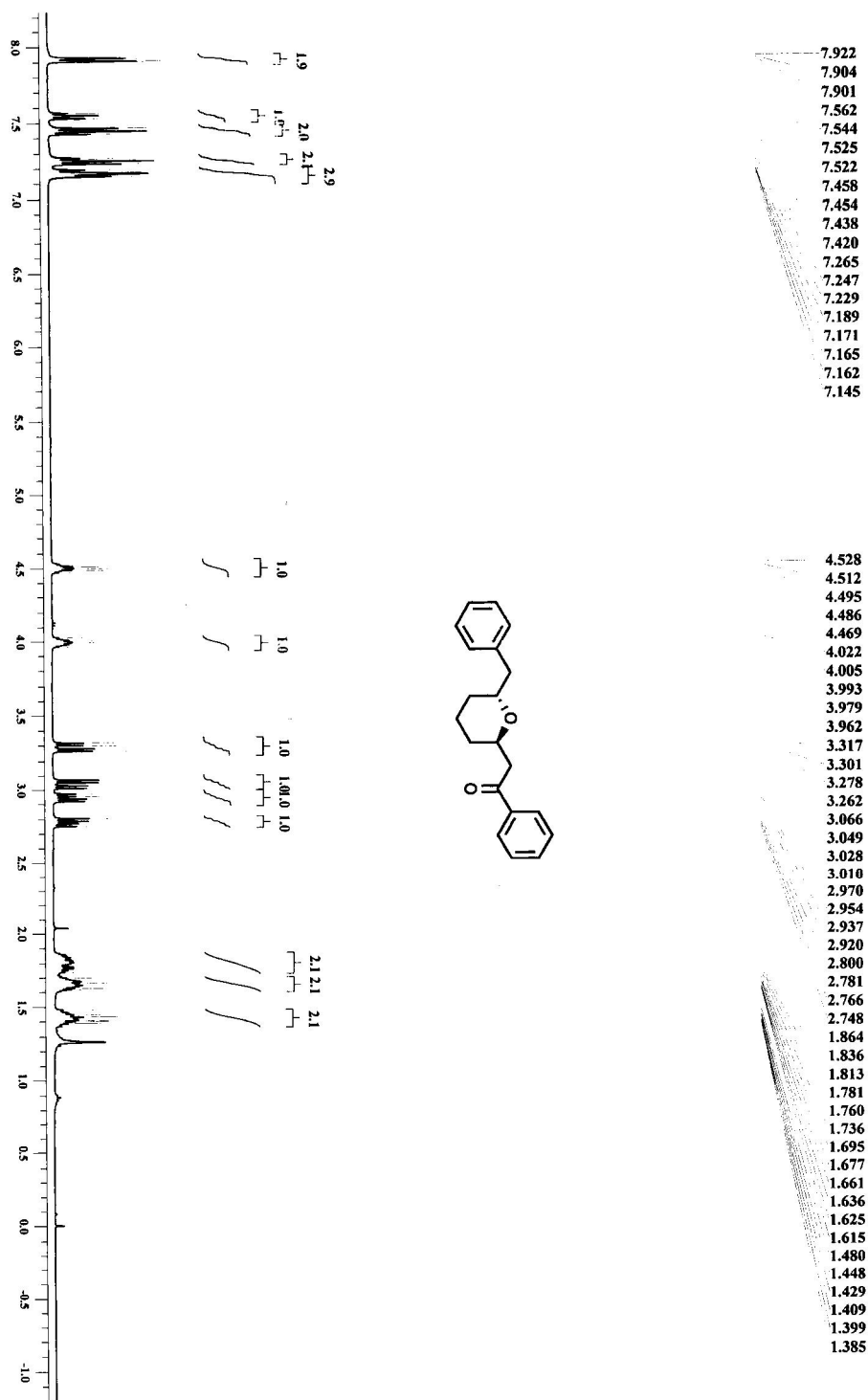
¹³C NMR Spectrum of 13e (100 MHz, CDCl₃)

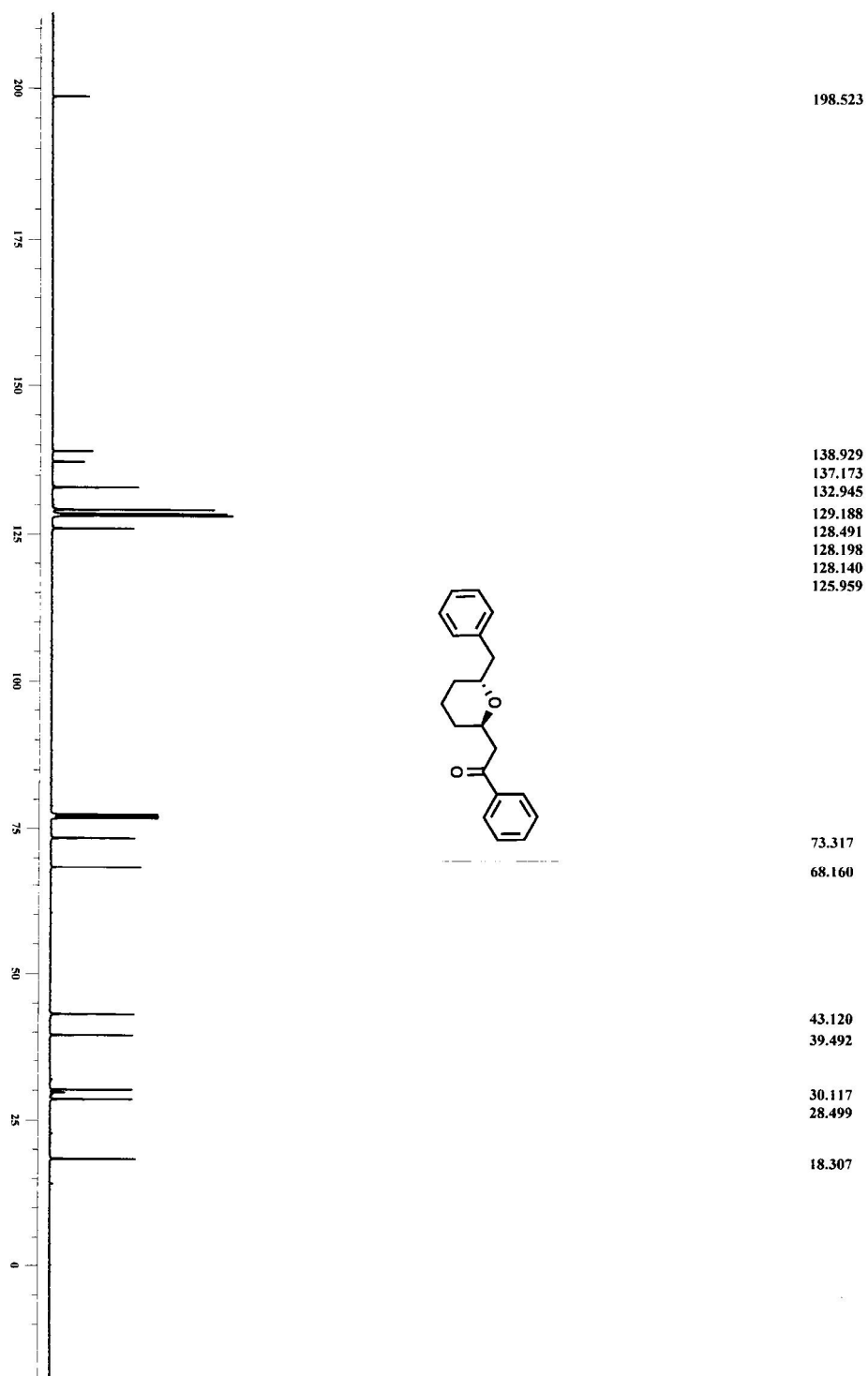


LC-LCMS Chromatogram of 13e



LC-LCMS Chromatogram of 13e

¹H NMR Spectrum of 13f (400 MHz, CDCl₃)



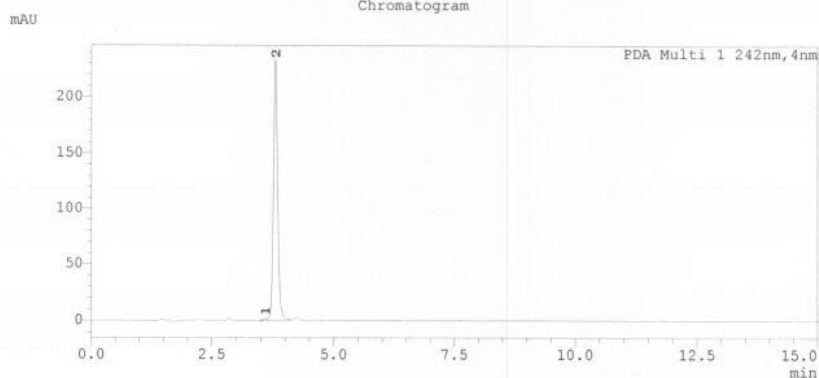
¹³C NMR Spectrum of 13f (100 MHz, CDCl₃)

SHIMADZU
LabSolutions

LC-MS DATA REPORT

11CT-DNPC

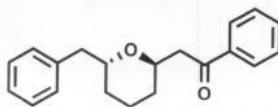
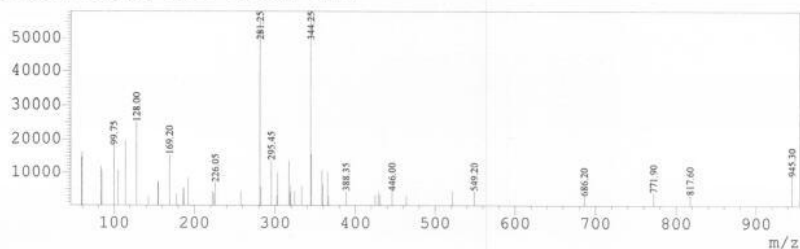
Sample Code : DKM-B-PHAC-294
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 Injection Volume : 3
 Date Acquired : 4/27/2018 3:07:52 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Column: LUNA-C8 (250 X 4.6 mm, 5.0u)
 Mobile Phase: 70% ACN IN 0.1% fa with water
 Flow Rate: 1.0 mL/min



Peak Table

Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	3.594	3.424	3.637	8934	0.639
2	3.805	3.637	4.139	1389251	99.361
Total				1398185	100.000

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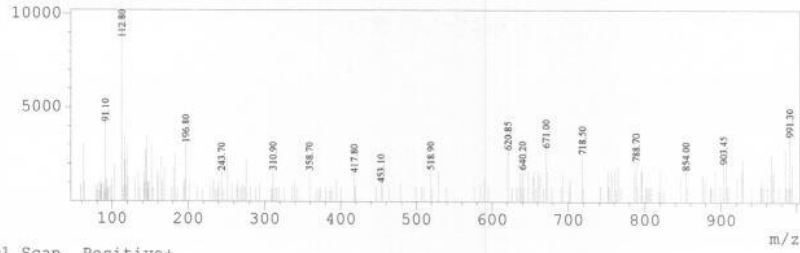


REPORT

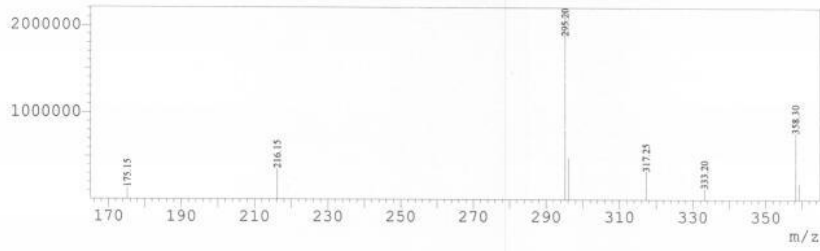
LC-LCMS Chromatogram of 13f

Q1 Scan Negative-
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ICT-DNFC

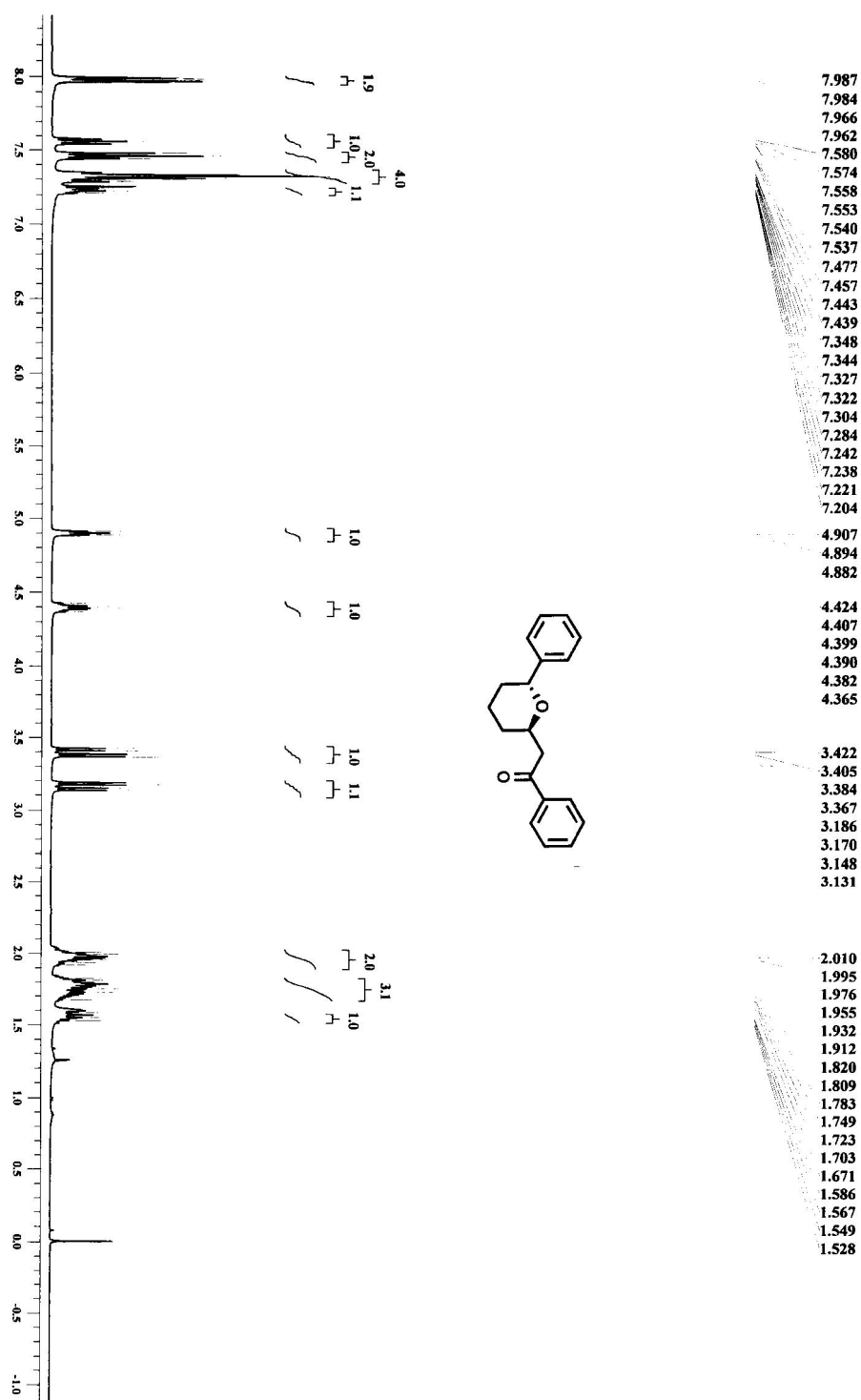


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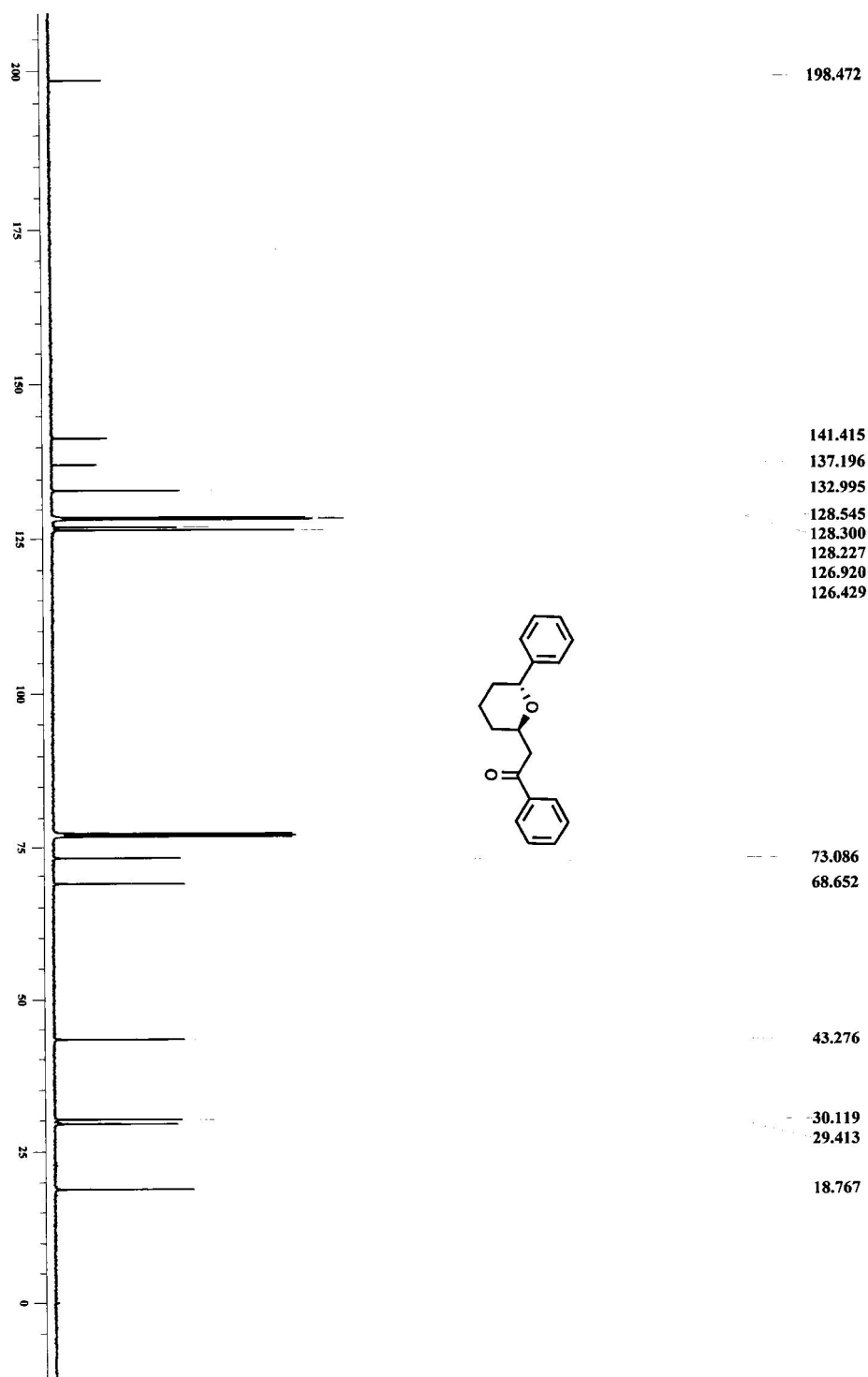


LC-MS REPORT

LC-LCMS Chromatogram of 13f

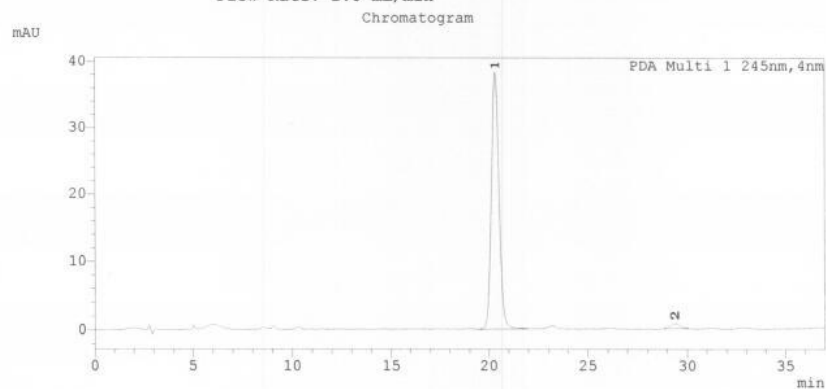


¹H NMR Spectrum of 13g (400 MHz, CDCl₃)



¹³C NMR Spectrum of 13g (125 MHz, CDCl₃)

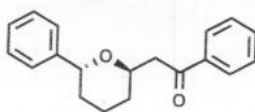
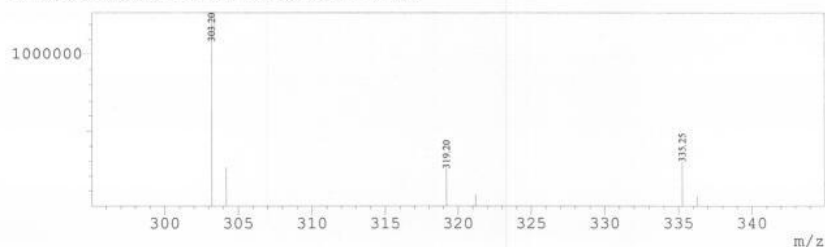
Sample Code : DKM-B-RBACT-280
 Data File : 250418.3.lcd
 Method : HPLC_ISO.lcm
 Injection Volume : 3
 Date Acquired : 4/25/2018 3:30:18 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Column: LUNA-C8 (250 X 4.6 mm, 5.0u)
 Mobile Phase: 60% MEOH IN 0.1% FA
 Flow Rate: 1.0 mL/min



Peak Table

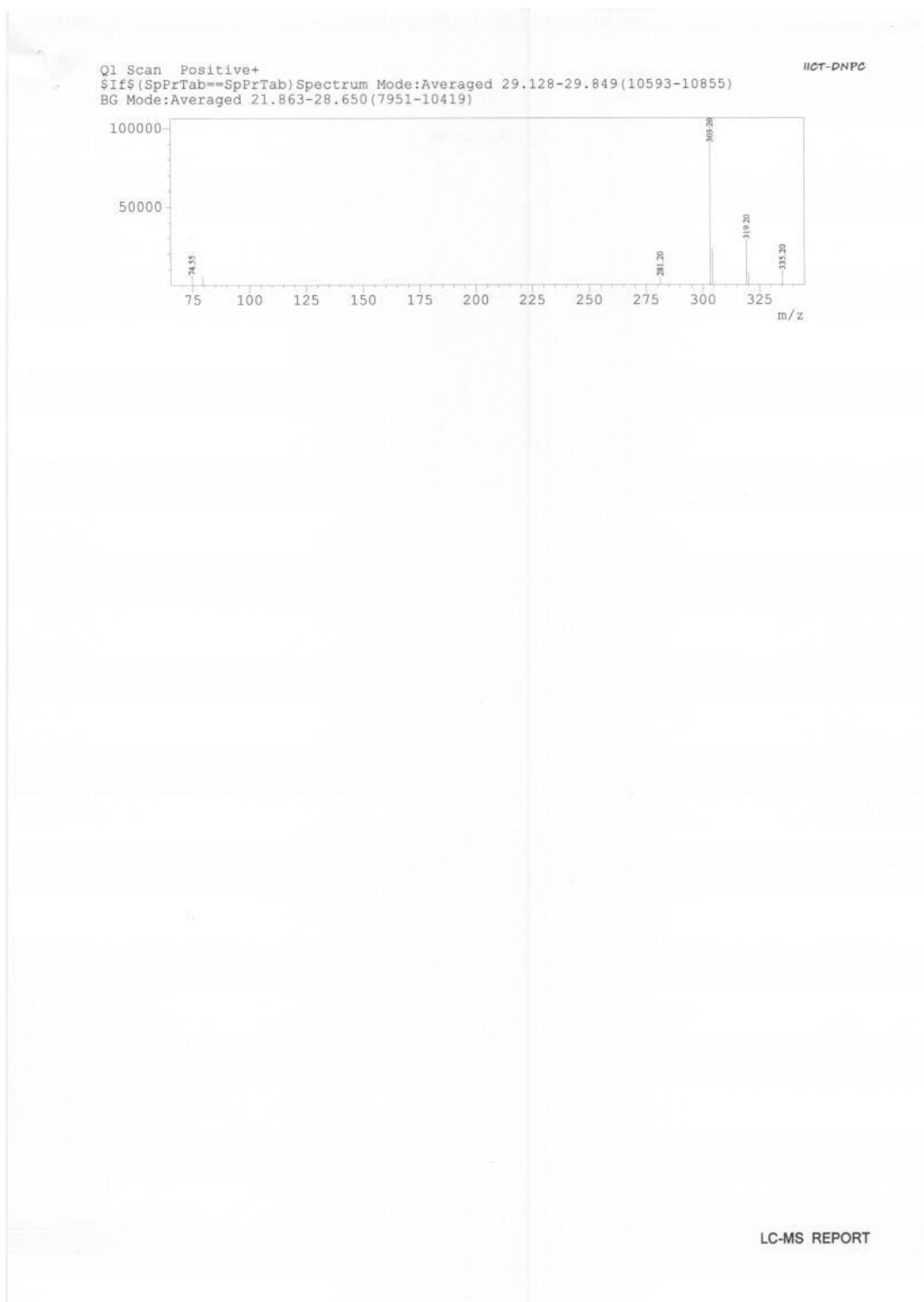
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	20.318	19.403	22.240	1023111	97.660
2	29.366	28.821	30.112	24509	2.340
Total				1047621	100.000

Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 19.916-20.983(7243-7631)
 BG Mode:Averaged 8.168-19.663(2971-7151)

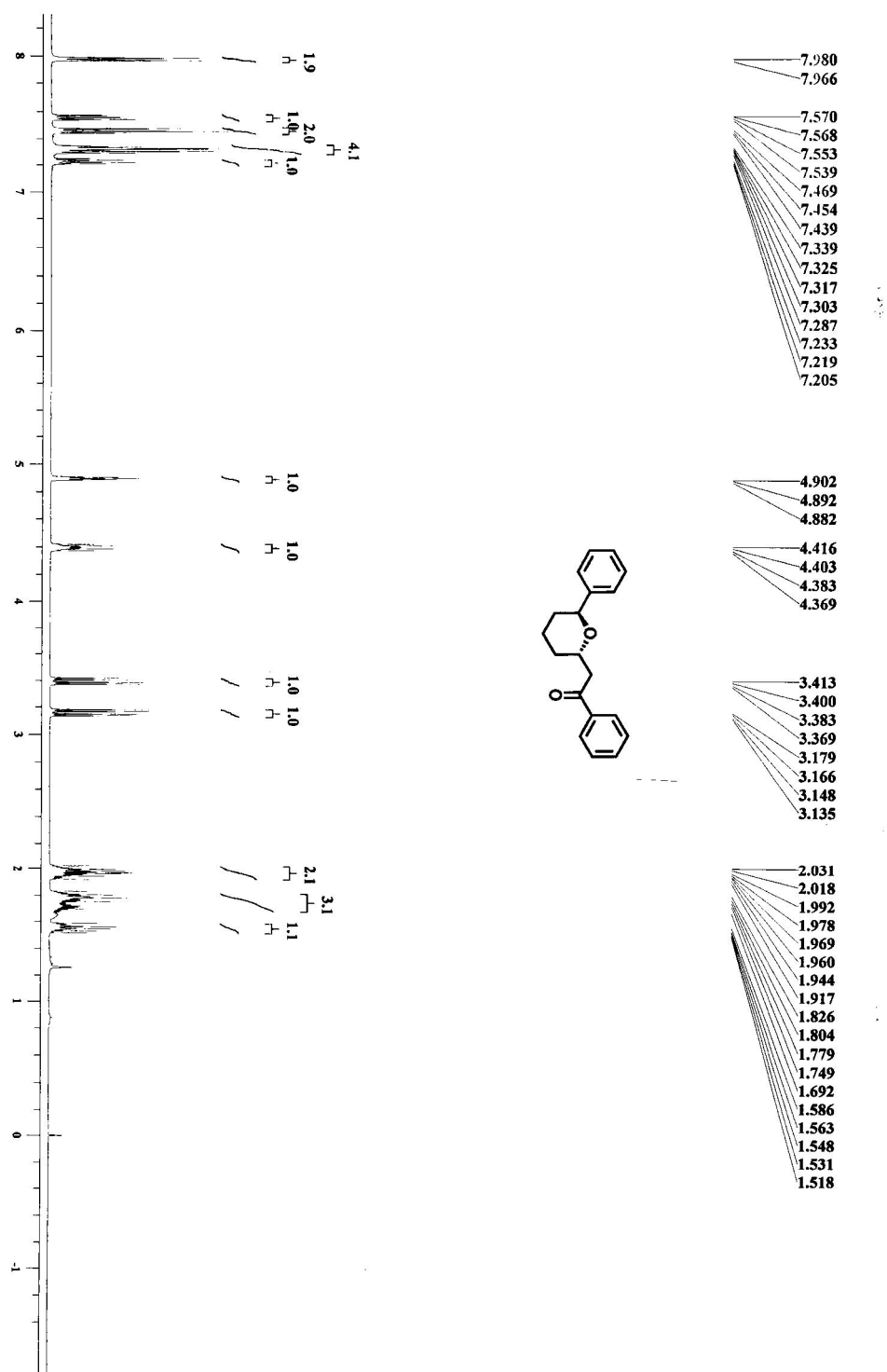


REPORT

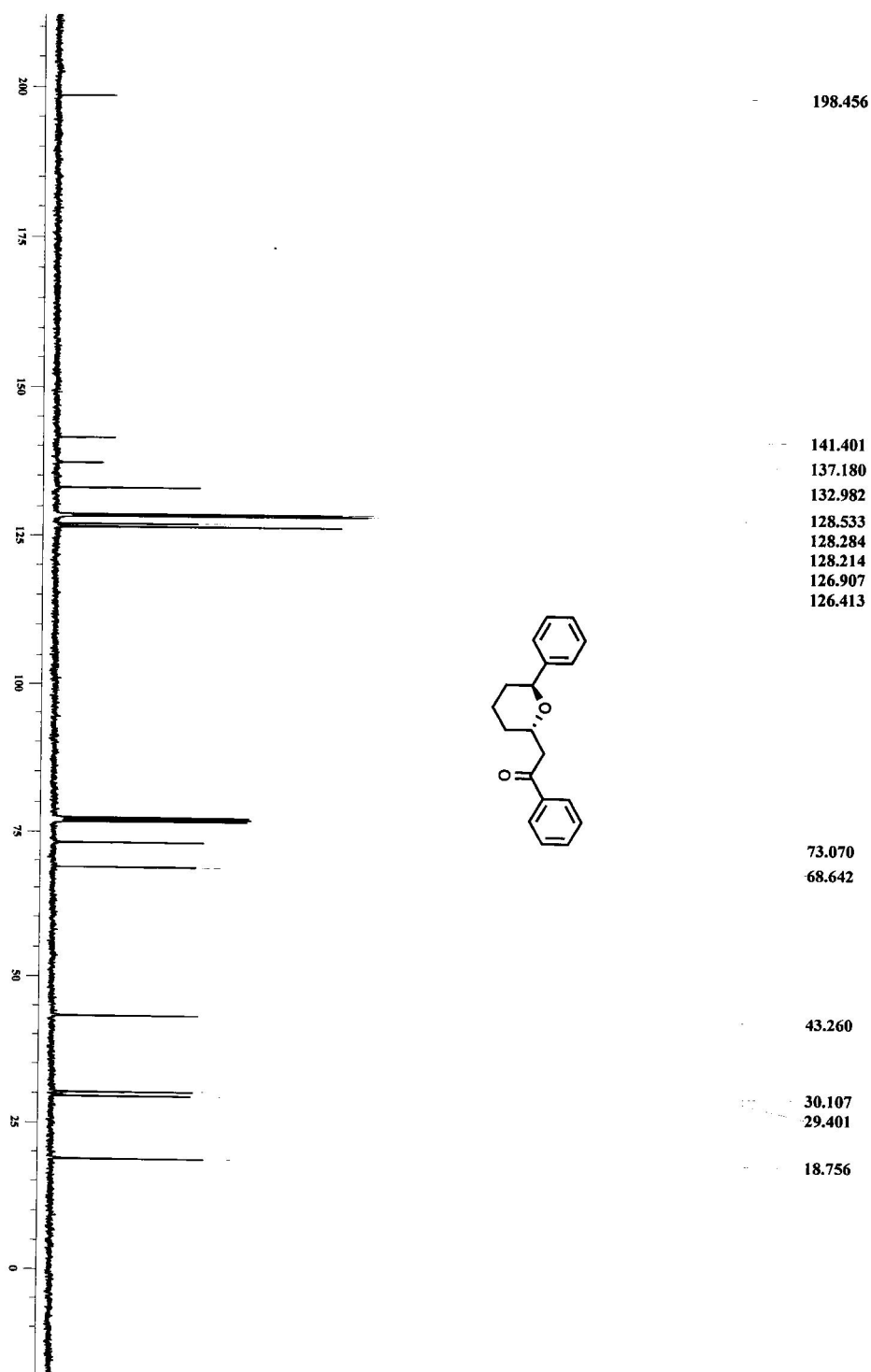
LC-LCMS Chromatogram of 13g



LC-LCMS Chromatogram of 13g

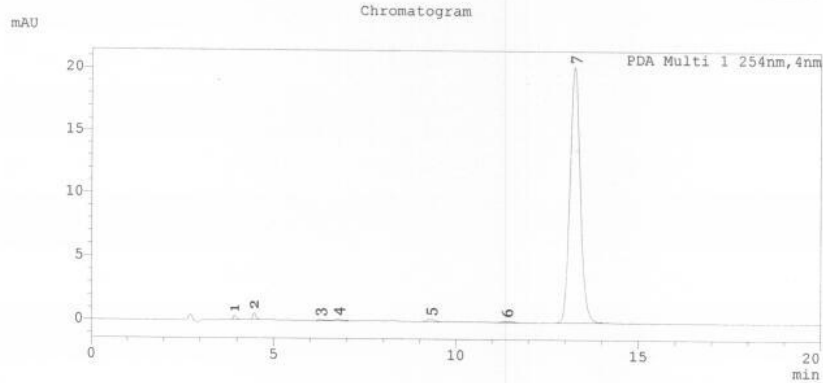
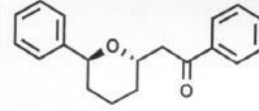


¹H NMR Spectrum of 13h (500 MHz, CDCl₃)



¹³C NMR Spectrum of 13h (100 MHz, CDCl₃)

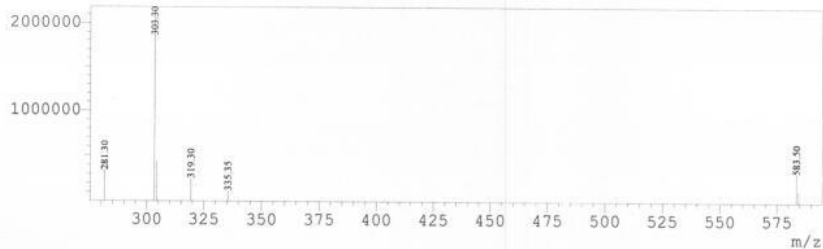
Sample Code : DKM-B-SBACT-280
 Data File : 200417-37.lcd
 Method : LC-MS Lab Method_75.lcm
 Injection Volume : 7
 Date Acquired : 4/20/2017 10:45:34 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : COLUMN: LUNA C18 250*4.6*5
 METHOD: 70 % MECH IN 0.1 % FA



Peak Table

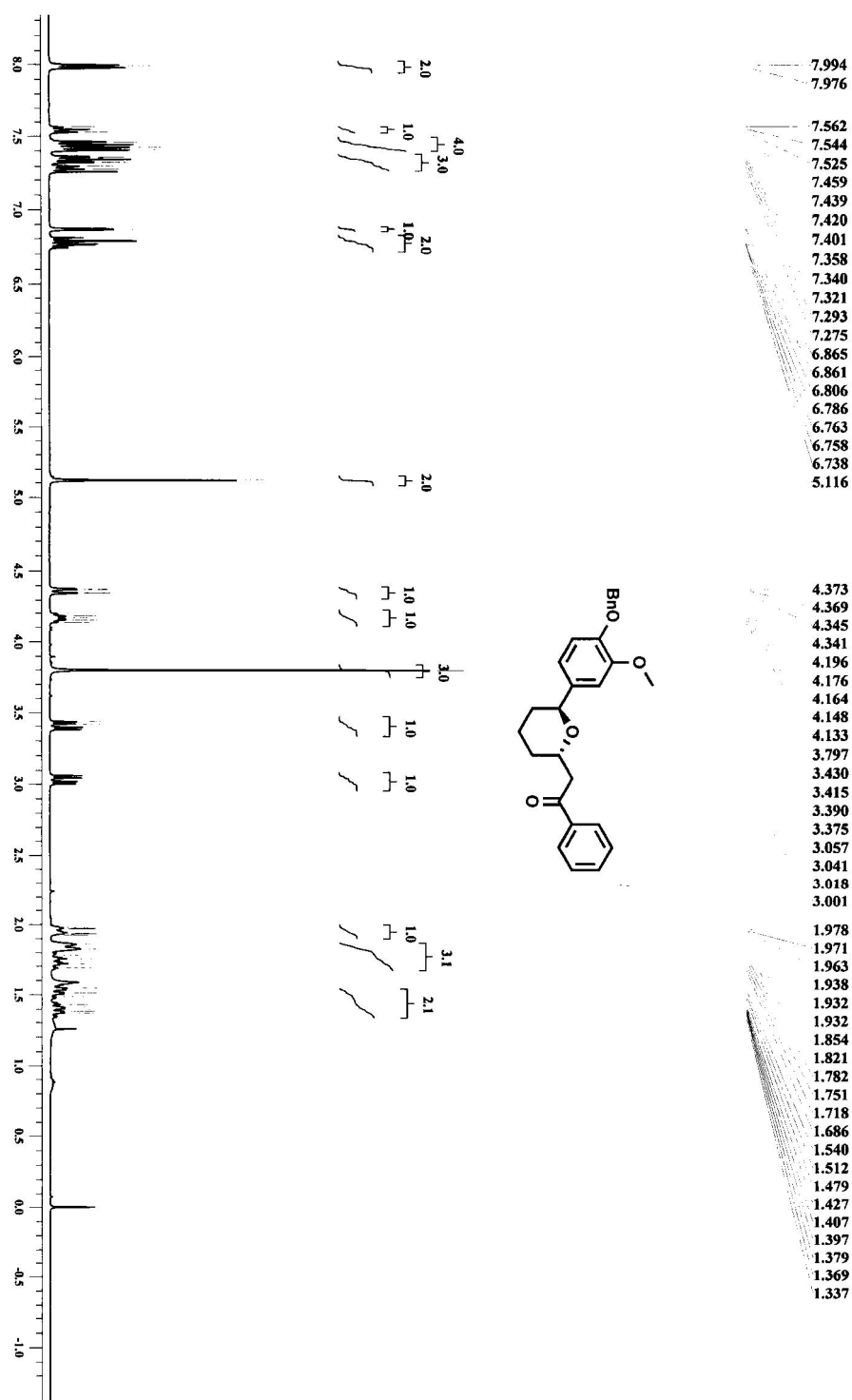
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	3.935	3.840	4.085	2001	0.491
2	4.473	4.363	4.629	3276	0.804
3	6.264	6.155	6.528	928	0.228
4	6.766	6.528	7.093	1657	0.407
5	9.309	9.088	9.579	2503	0.614
6	11.457	11.168	11.712	1217	0.299
7	13.259	12.768	14.197	395980	97.158
Total				407563	100.000

Q1 Scan Positive+
 \$if\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 12.863-13.930(4411-4777)
 BG Mode:Averaged 2.199-12.198(755-4183)

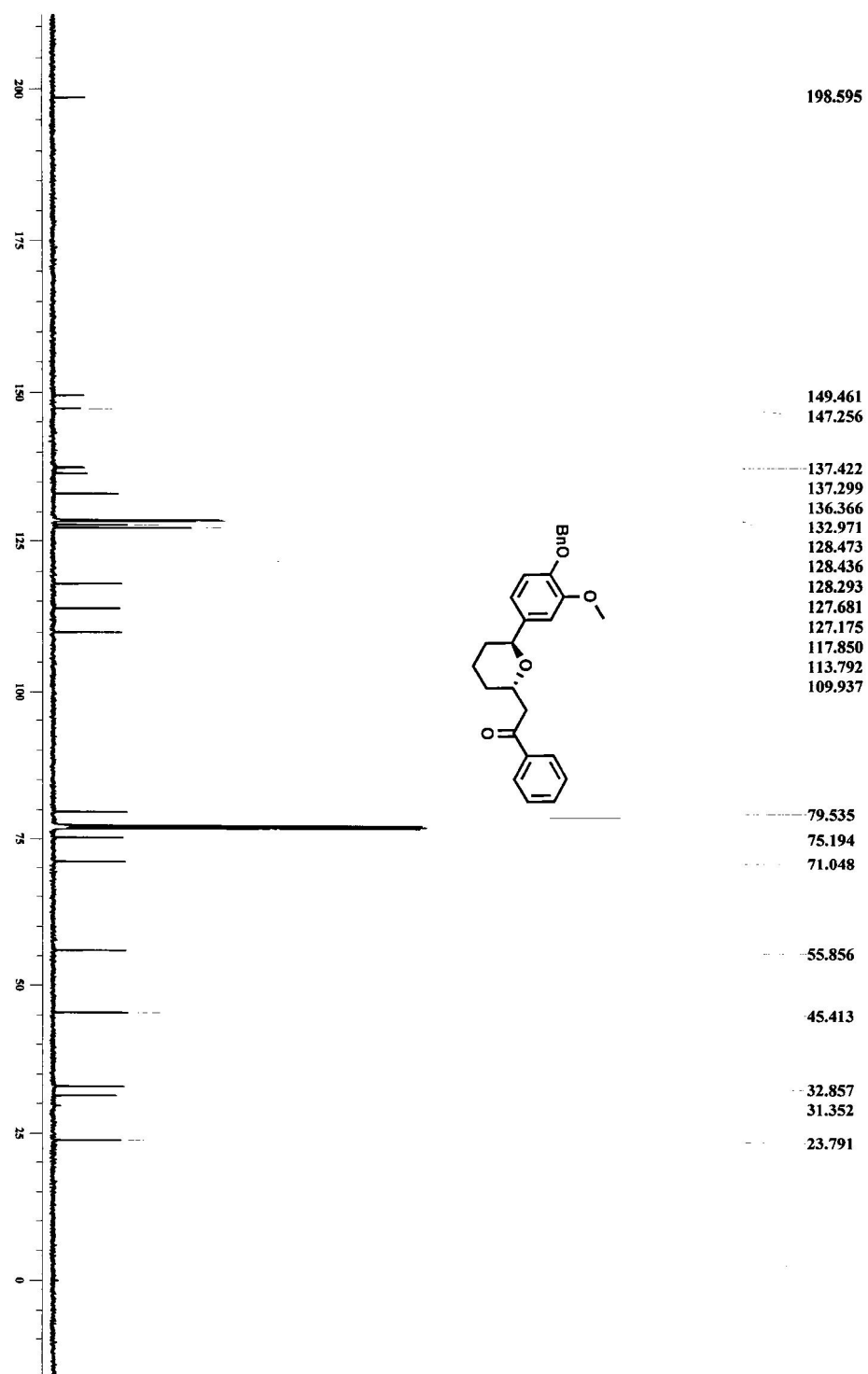


LC-MS REPORT

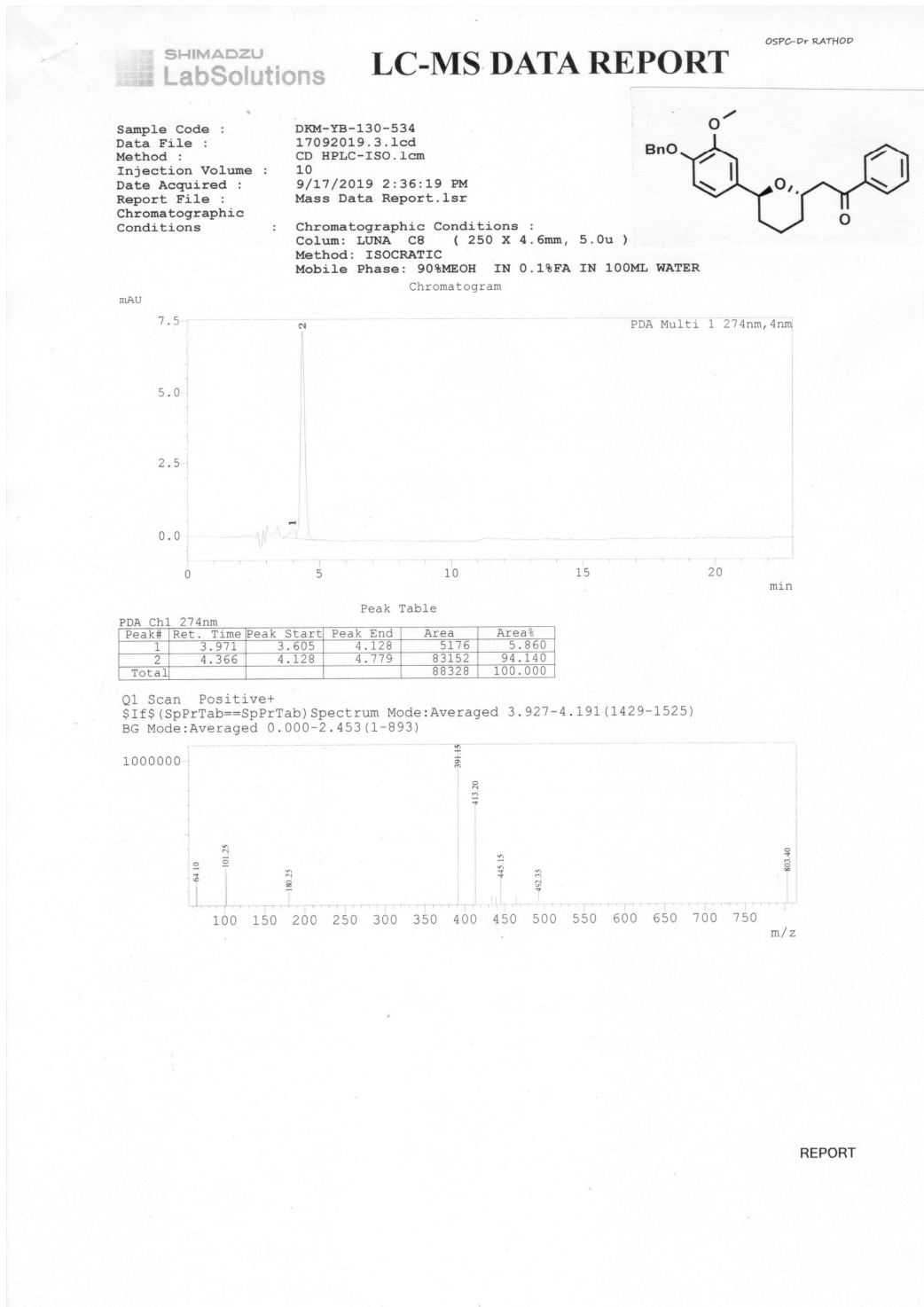
LC-LCMS Chromatogram of 13h



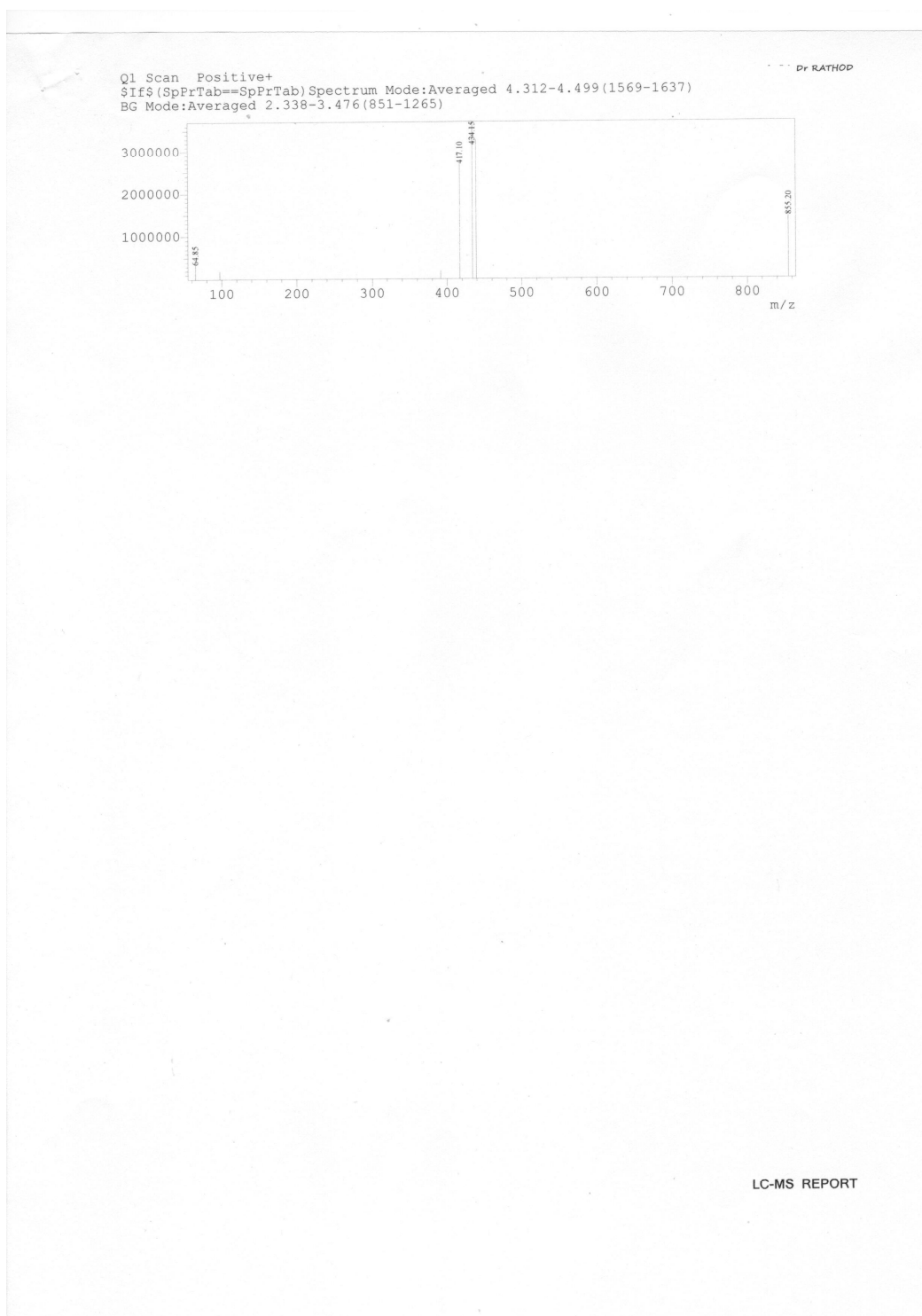
¹H NMR Spectrum of 13i (400 MHz, CDCl₃)



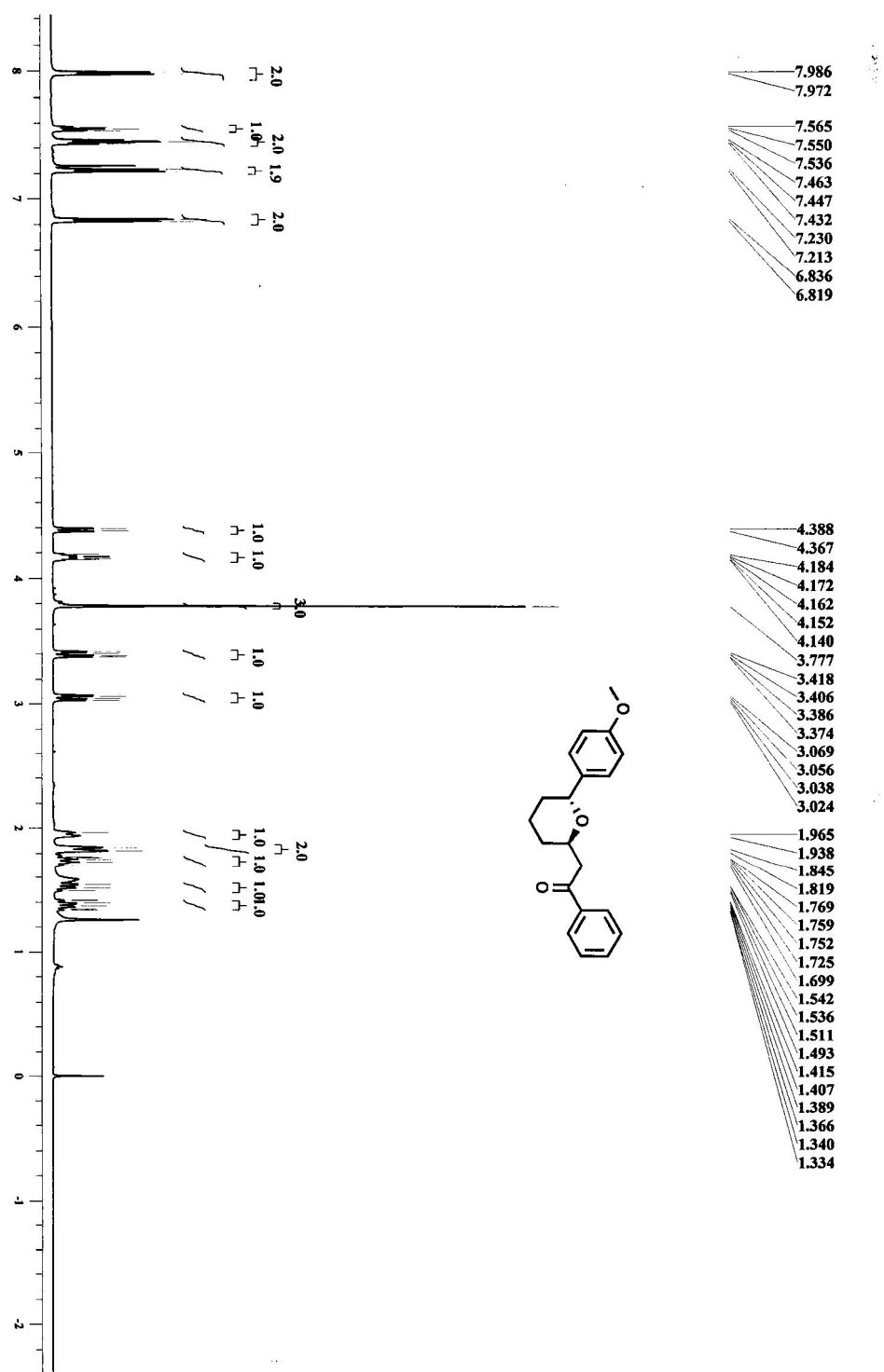
¹³C NMR Spectrum of 13i (125 MHz, CDCl₃)

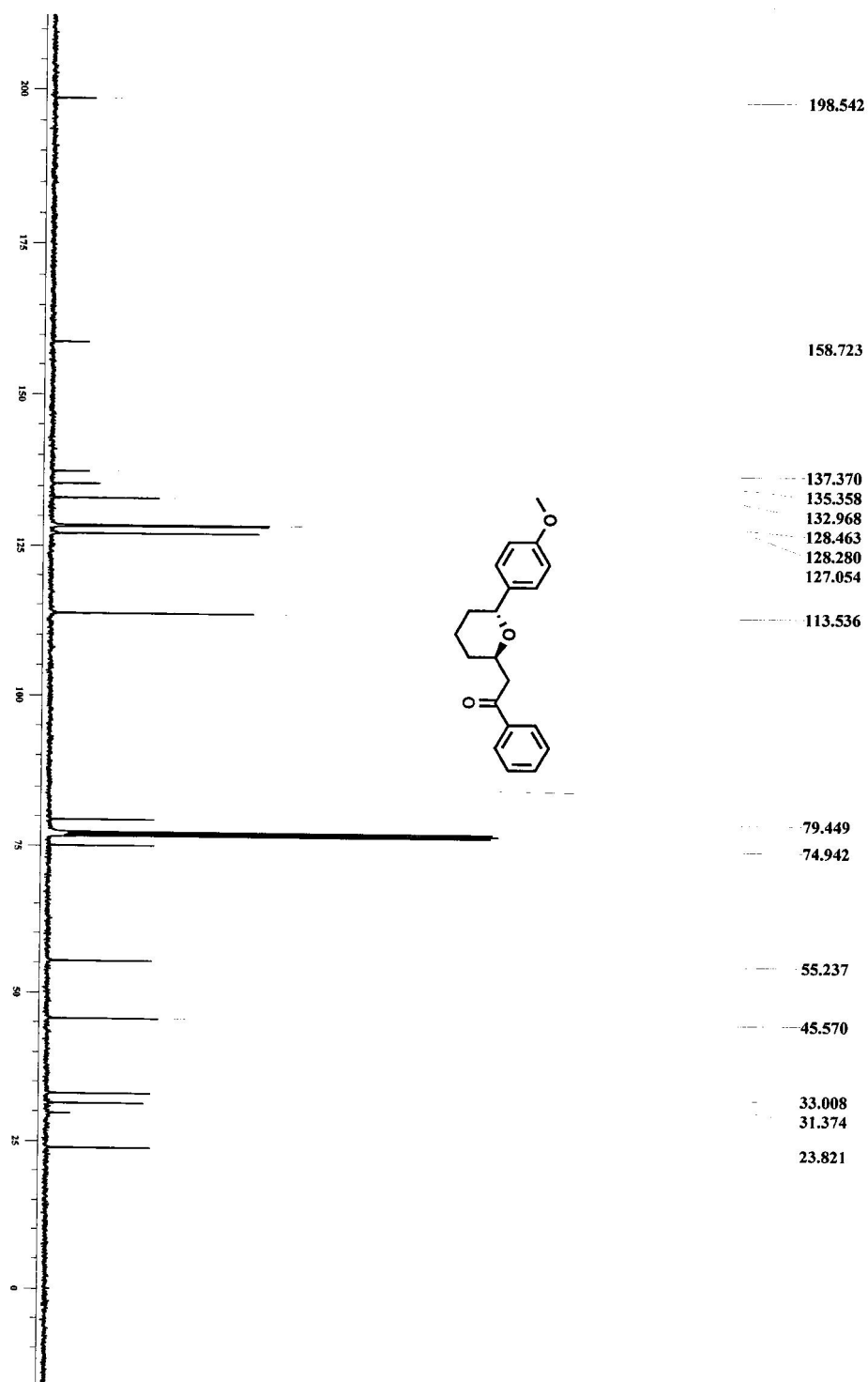


LC-LCMS Chromatogram of 13i



LC-LCMS Chromatogram of 13i

¹H NMR Spectrum of 13j (500 MHz, CDCl₃)



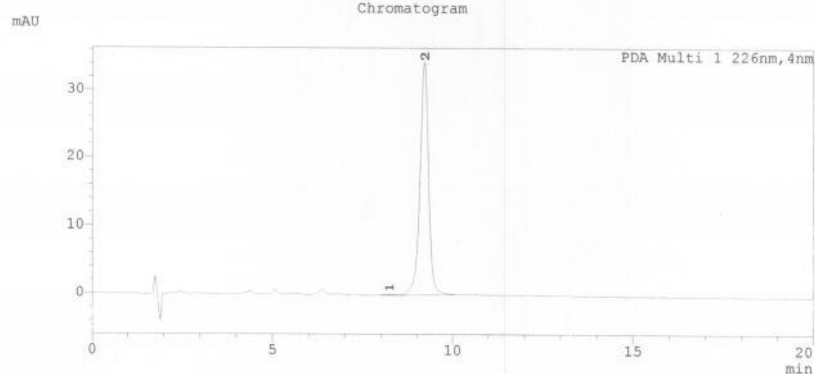
¹³C NMR Spectrum of 13j (100 MHz, CDCl₃)

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LC-MS DATA REPORT

11CT-DNPC

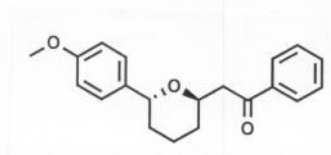
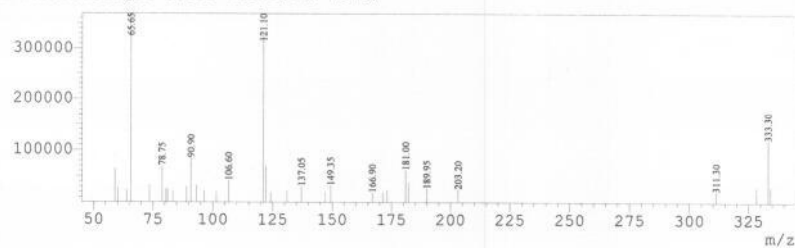
Sample Code : DKM-B-ANACT-310
 Data File : 070518.4.lcd
 Method : HPLC_ISO.lcm
 Injection Volume : 2
 Date Acquired : 5/7/2018 1:42:06 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Column: X-SELECT (150 X 4.6 mm, 5.0u)
 Mobile Phase: 70% MEOH IN 0.1% fa with water
 Flow Rate: 1.0 mL/min



Peak Table

Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	8.232	7.979	8.523	1462	0.254
2	9.210	8.523	10.059	573303	99.746
Total				574765	100.000

Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 8.195-8.311(2981-3023)
 BG Mode:Averaged 2.596-8.179(945-2975)

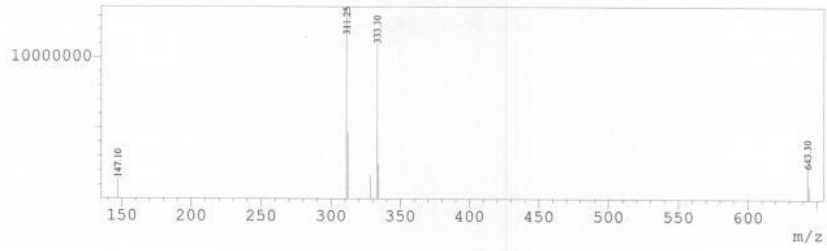


REPORT

LC-LCMS Chromatogram of 13j

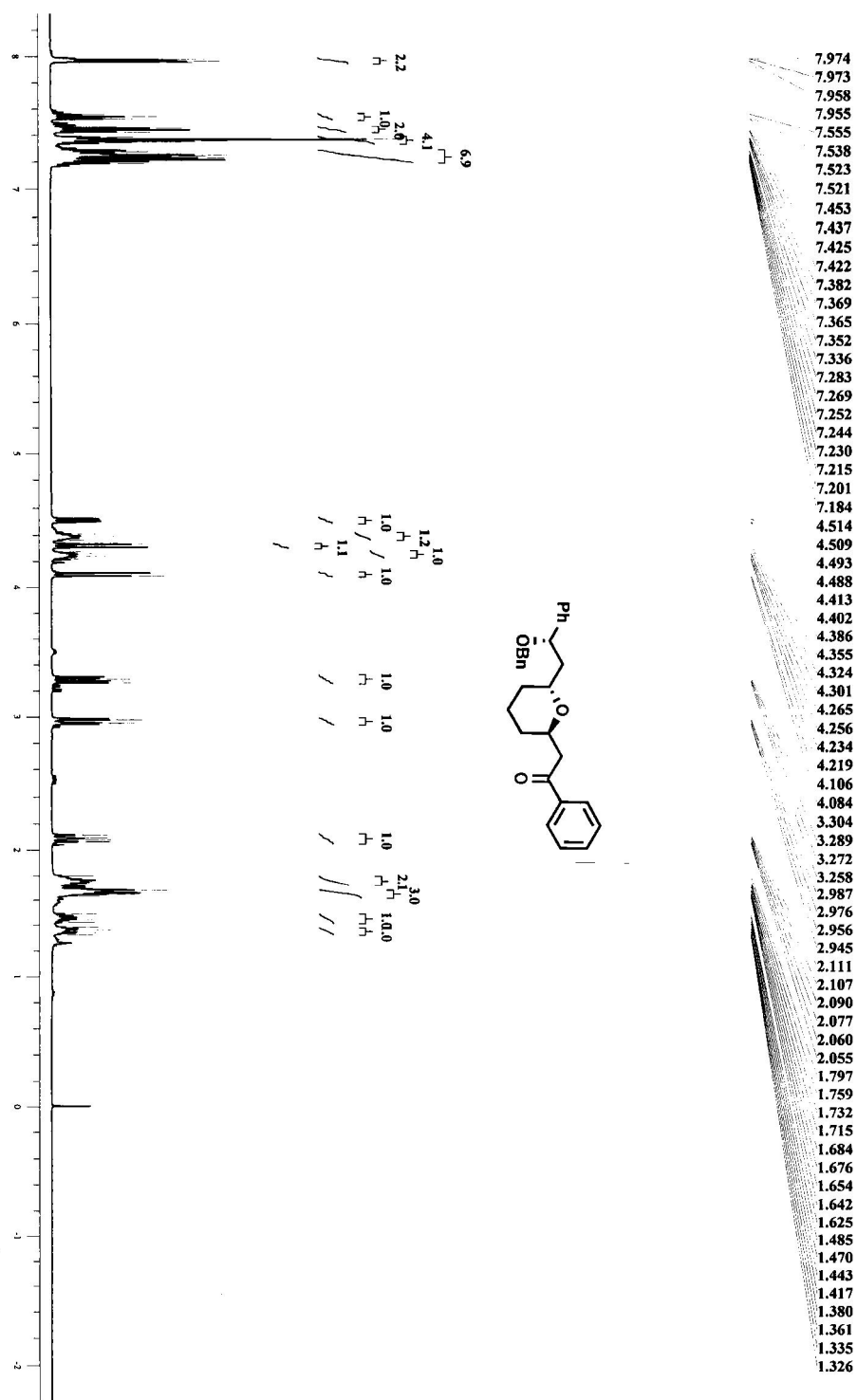
Q1 Scan Positive+
\$If\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 8.949-9.647(3255-3509)
BG Mode:Averaged 2.255-8.690(821-3161)

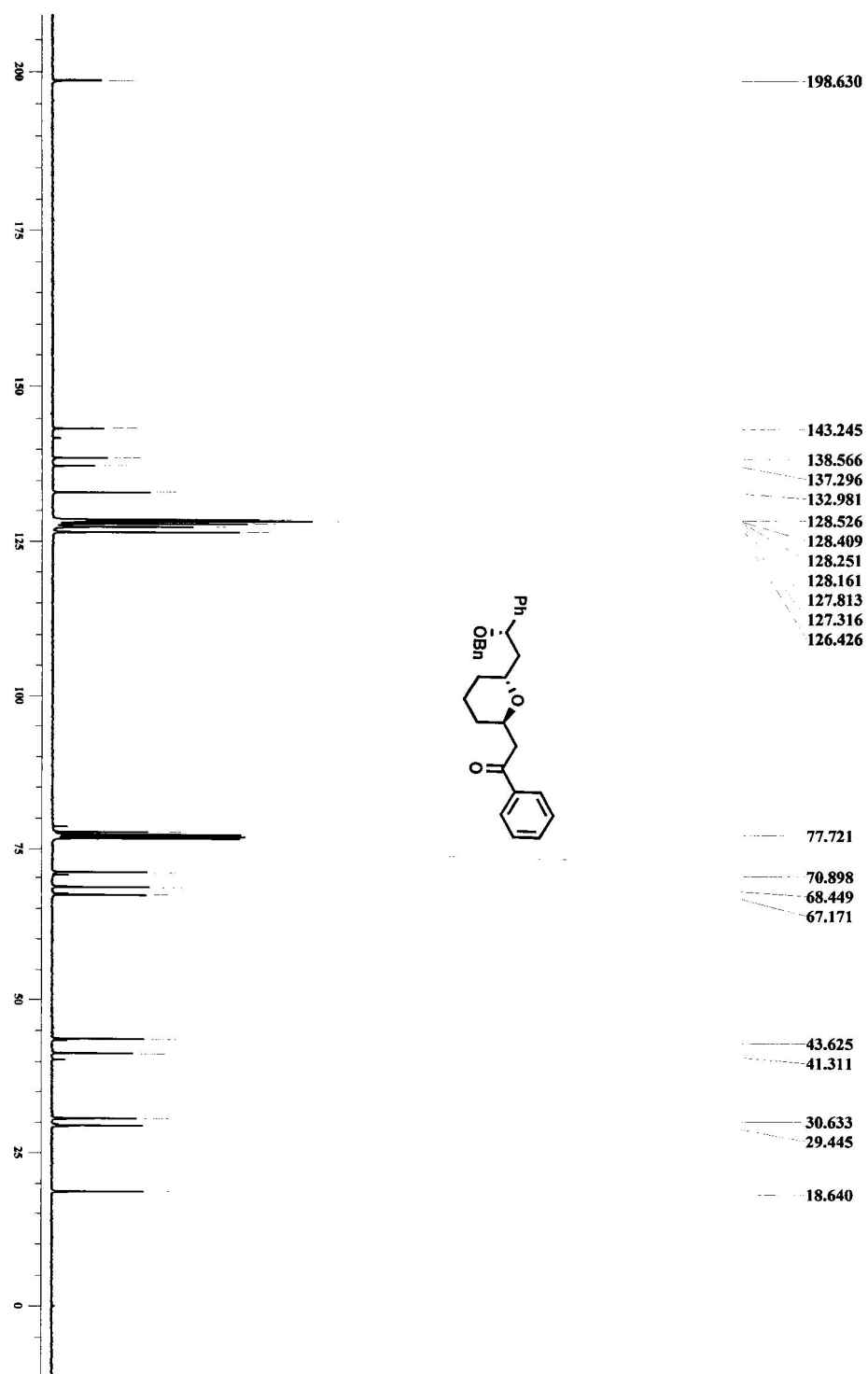
11CT-DNPG



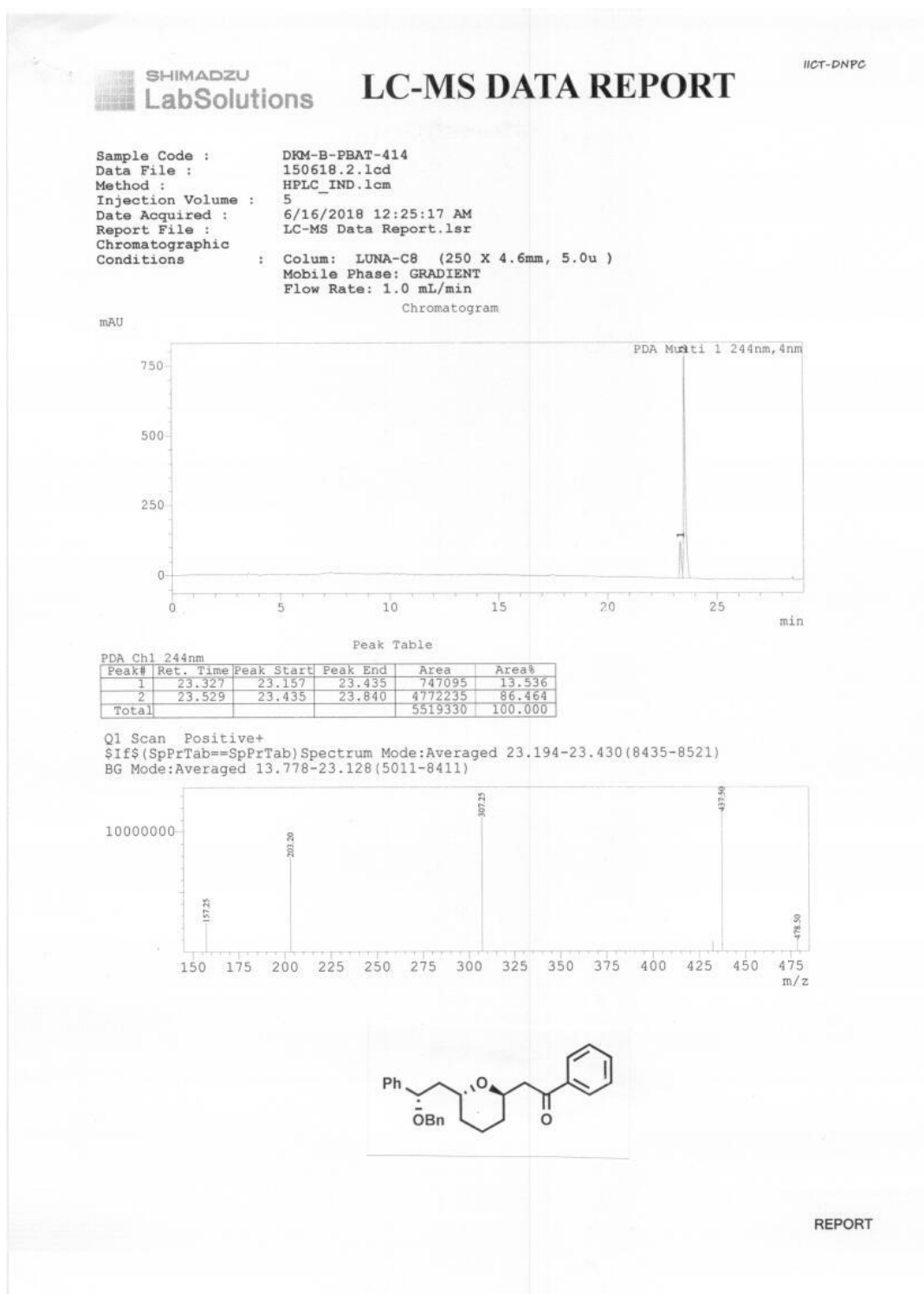
LC-MS REPORT

LC-LCMS Chromatogram of 13j

¹H NMR Spectrum of 13k (500 MHz, CDCl₃)

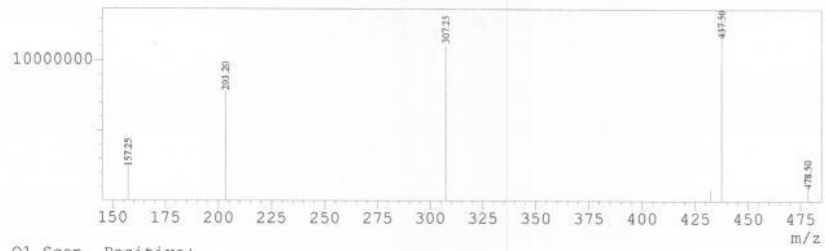


¹³C NMR Spectrum of 13k (100 MHz, CDCl₃)

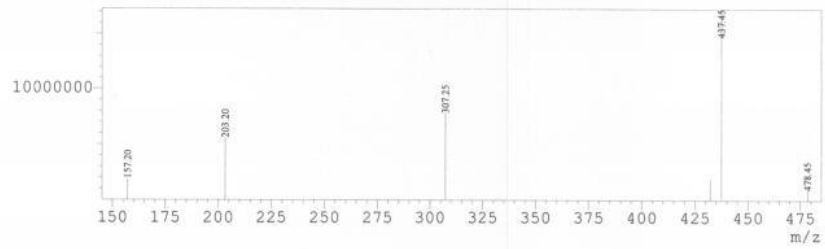


LC-LCMS Chromatogram of 13k

Q1 Scan Positive+
\$If\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 23.194-23.430(8435-8521)
BG Mode:Averaged 13.778-23.128(5011-8411) IIC-T-DNPC

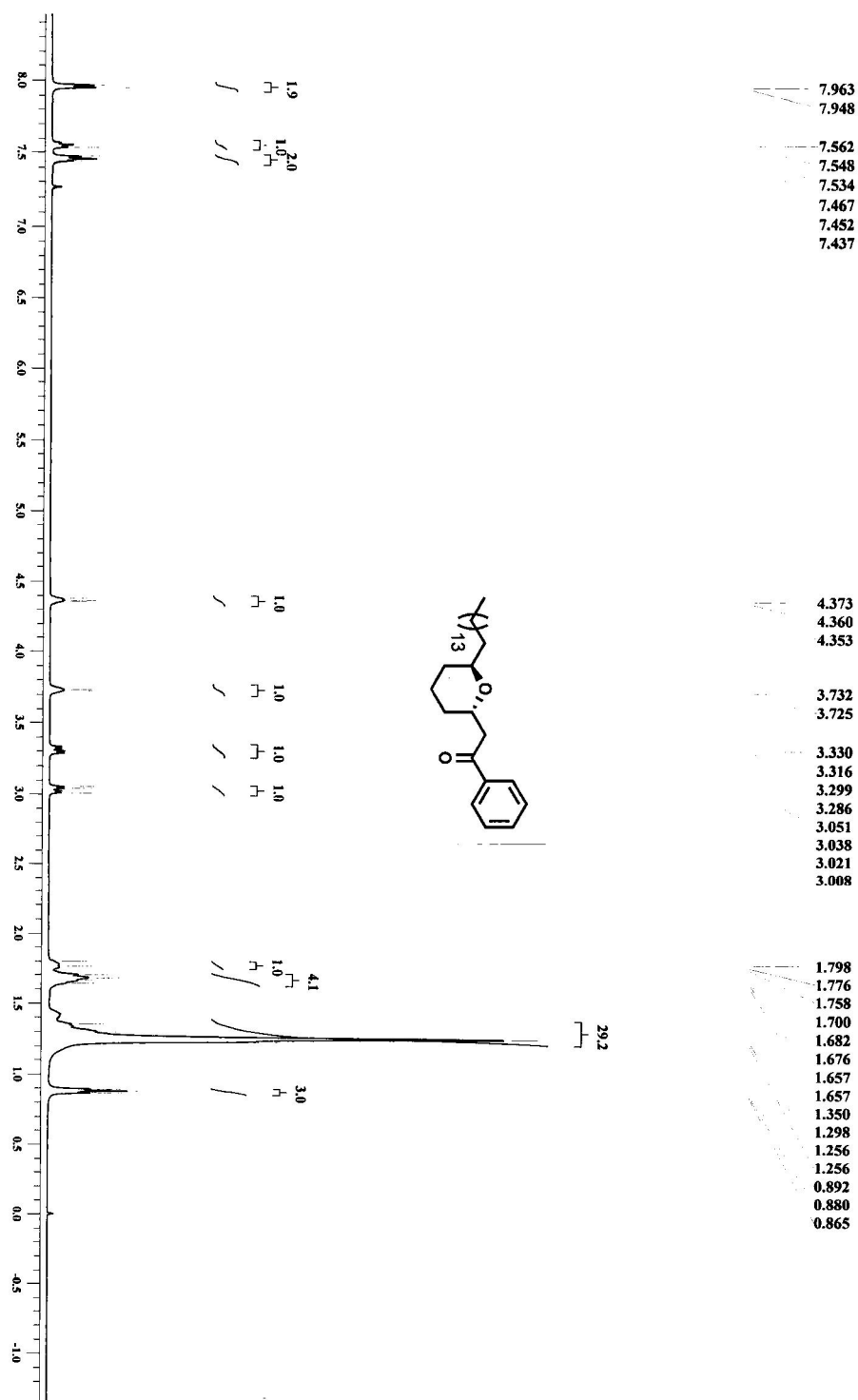


Q1 Scan Positive+
\$If\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 23.474-23.788(8537-8651)
BG Mode:Averaged 12.023-23.419(4373-8517)

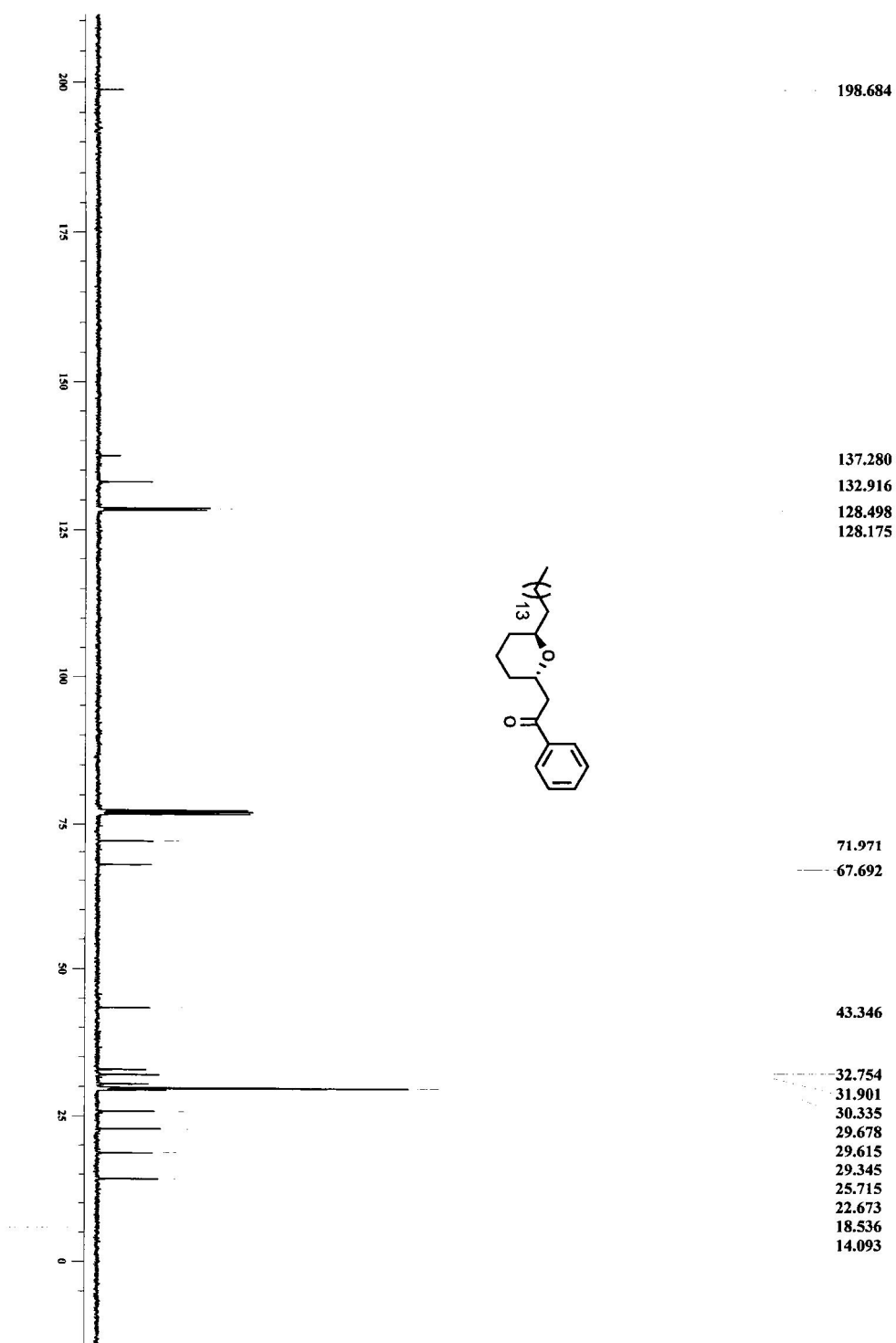


LC-MS REPORT

LC-LCMS Chromatogram of 13k

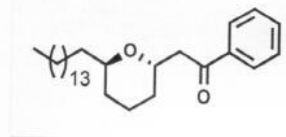


¹H NMR Spectrum of 131 (500 MHz, CDCl₃)

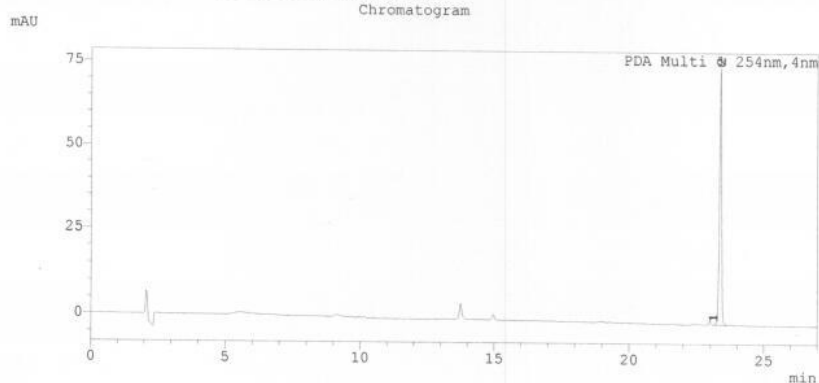


¹³C NMR Spectrum of 13l (100 MHz, CDCl₃)

Sample Code : DKM-B-LCAT-414
 Data File : 110718.2.lcd
 Method : CD HPLC.lcm
 Injection Volume : 5
 Date Acquired : 7/11/2018 10:48:08 AM
 Report File : LC-MS Data Report.lsr



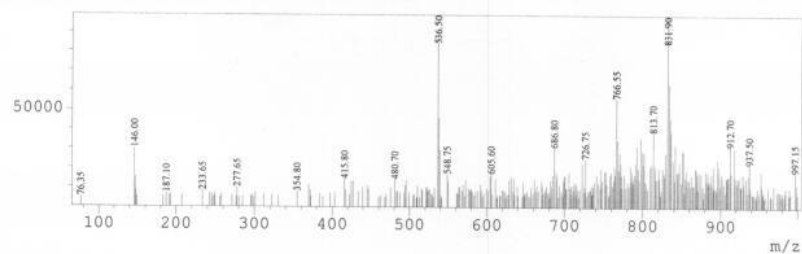
Chromatographic Conditions : Chromatographic
 Conditions : Colum: X-SELECT CSH (250 X 2.1mm, 5.0u)
 Mobile Phase: GRADINET
 Flow Rate: 1.0 mL/min



Peak Table

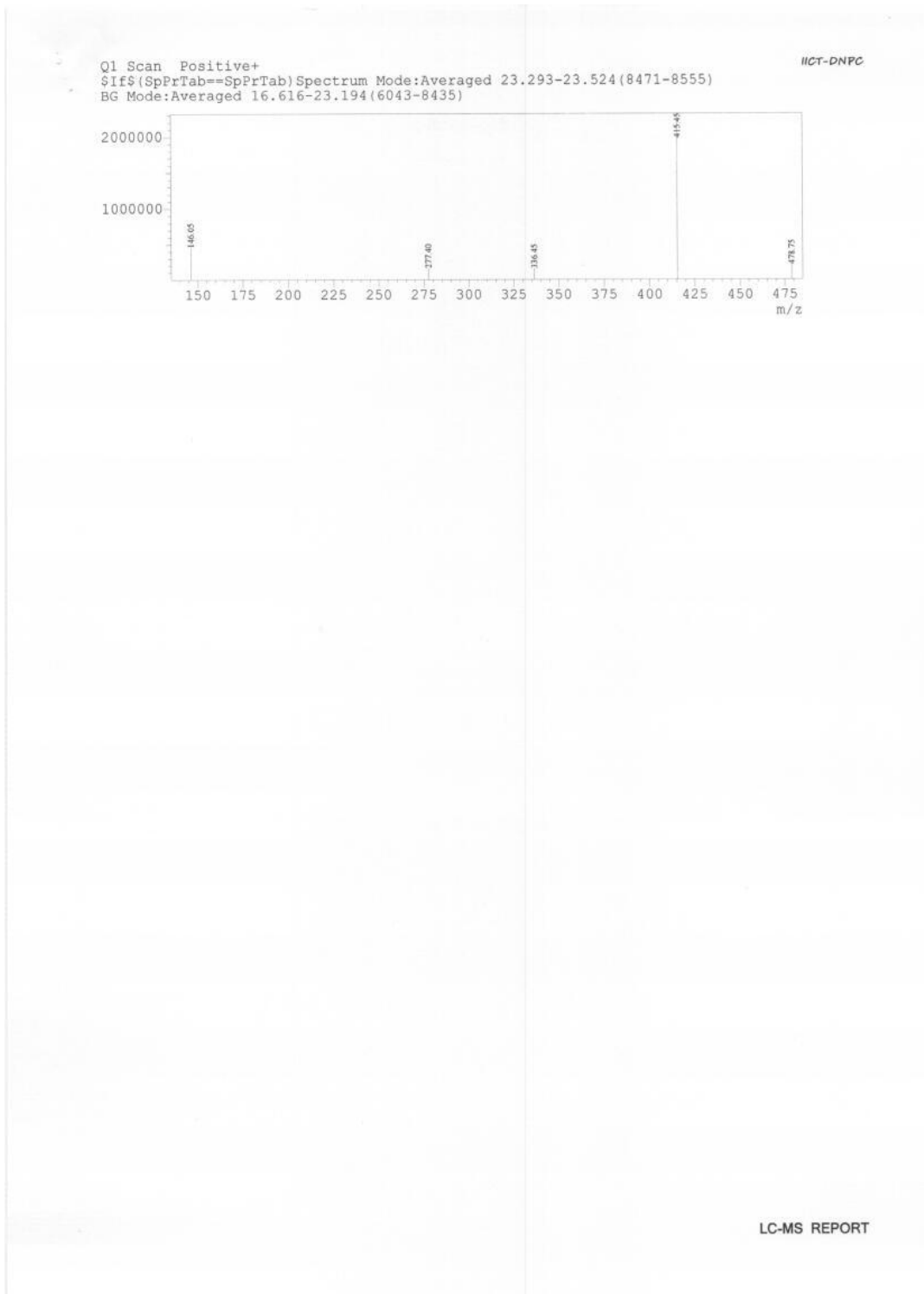
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	23.122	23.093	23.157	79	0.020
2	23.373	23.168	23.627	402188	99.980
Total				402267	100.000

Q1 Scan Positive+
 \$If\$ (SpPrTab==SpPrTab) Spectrum Mode:Averaged 23.166-23.243 (8425-8453)
 BG Mode:Averaged 18.079-22.644 (6575-8235)

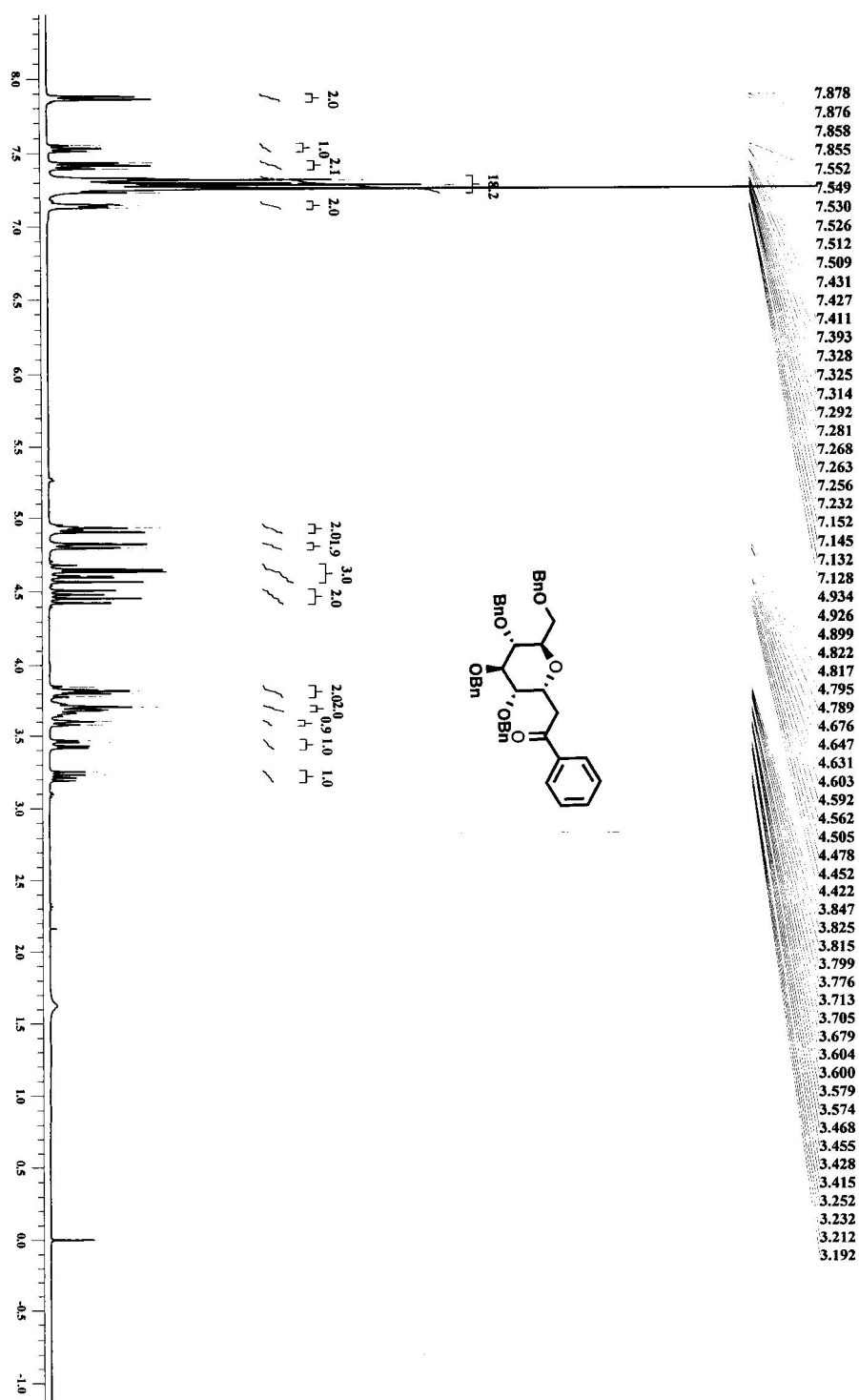


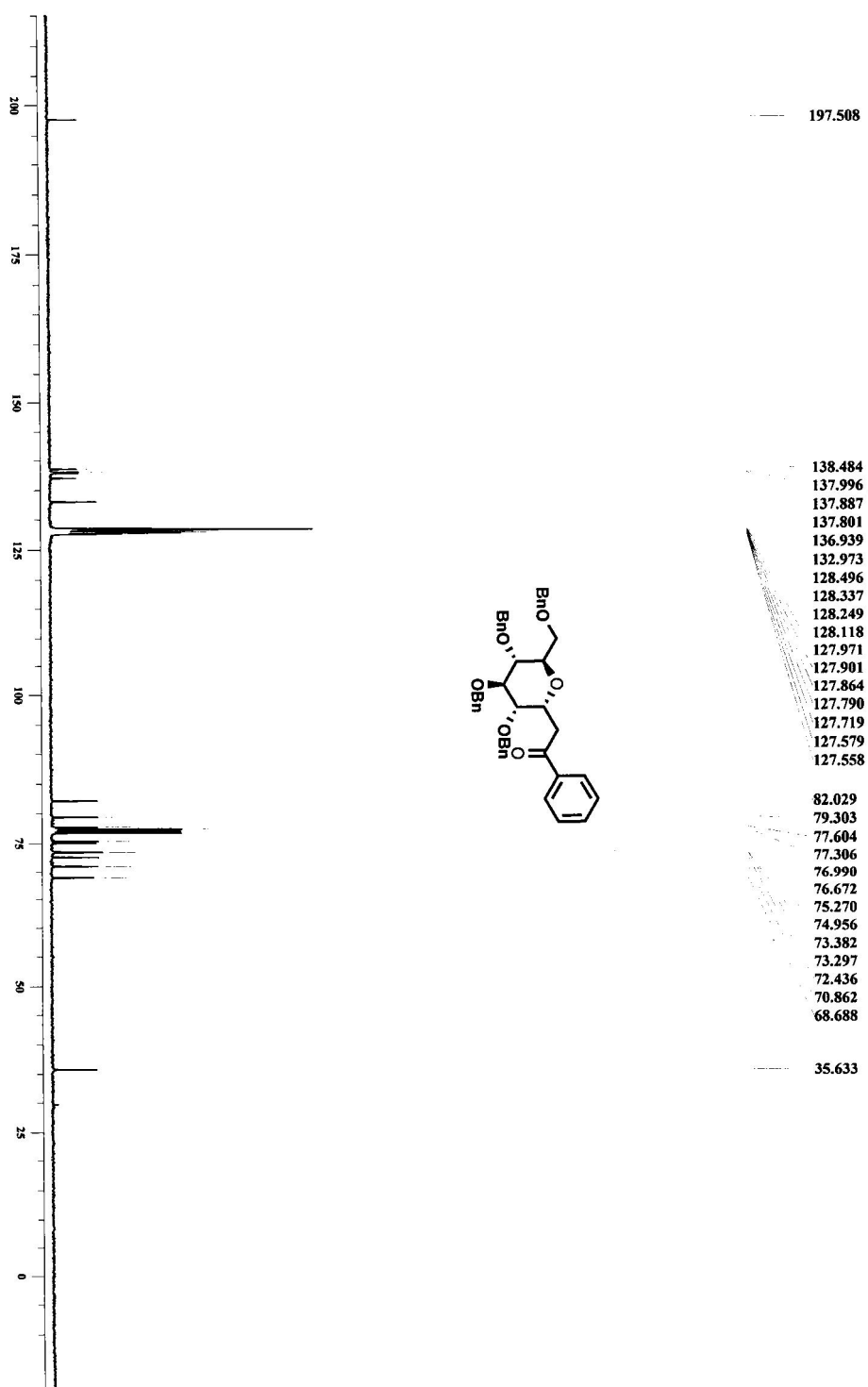
REPORT

LC-LCMS Chromatogram of 13l



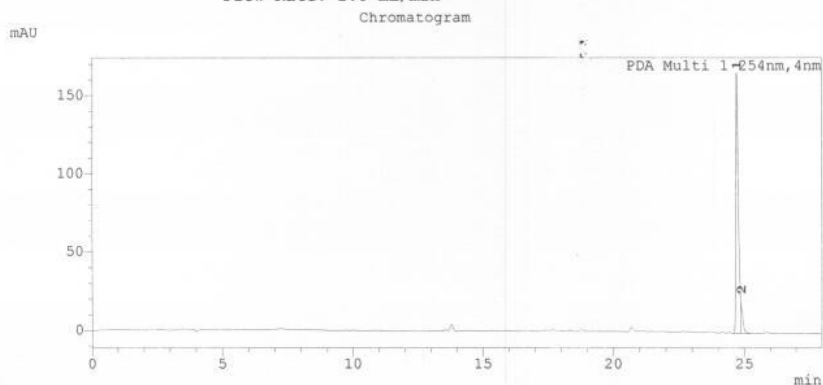
LC-LCMS Chromatogram of 13l

¹H NMR Spectrum of 13m (400 MHz, CDCl₃)



¹³C NMR Spectrum of 13m (100 MHz, CDCl₃)

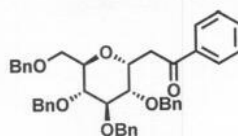
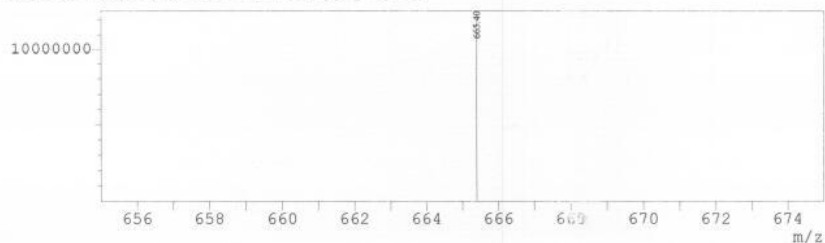
Sample Code : DKM-B-BNAT-642
 Data File : 150618.5.1cd
 Method : HPLC_IND.lcm
 Injection Volume : 5
 Date Acquired : 6/16/2018 1:57:06 AM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Colum: LUNA-C8 (250 X 4.6mm, 5.0µ)
 Mobile Phase: GRADIENT
 Flow Rate: 1.0 mL/min



Peak Table

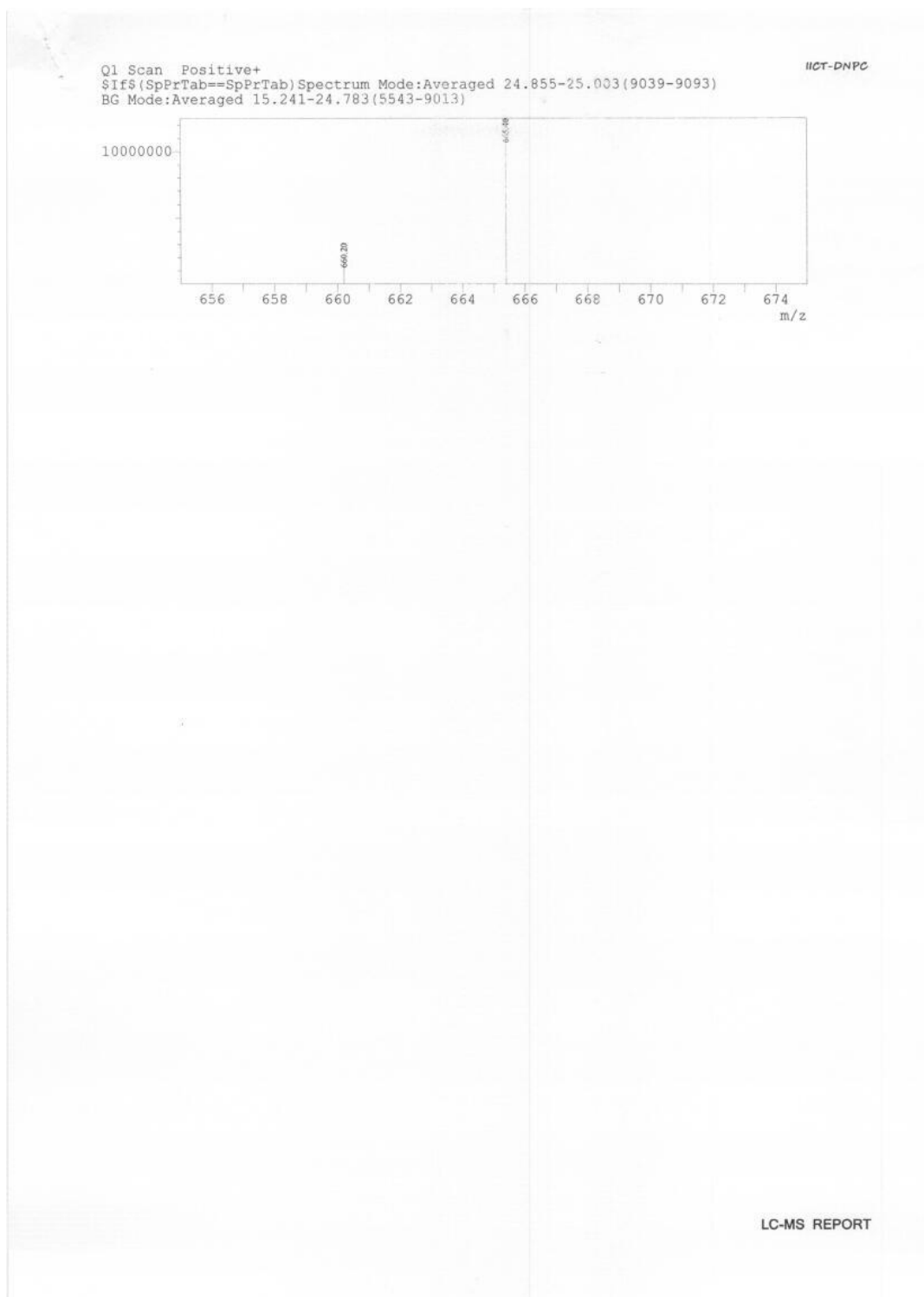
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	24.722	24.576	24.864	1066666	92.150
2	24.880	24.864	25.291	90861	7.850
Total				1157526	100.000

Q1 Scan Positive+
 \$if\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 24.651-24.805 (8965-9021)
 BG Mode:Averaged 10.274-24.591 (3737-8943)

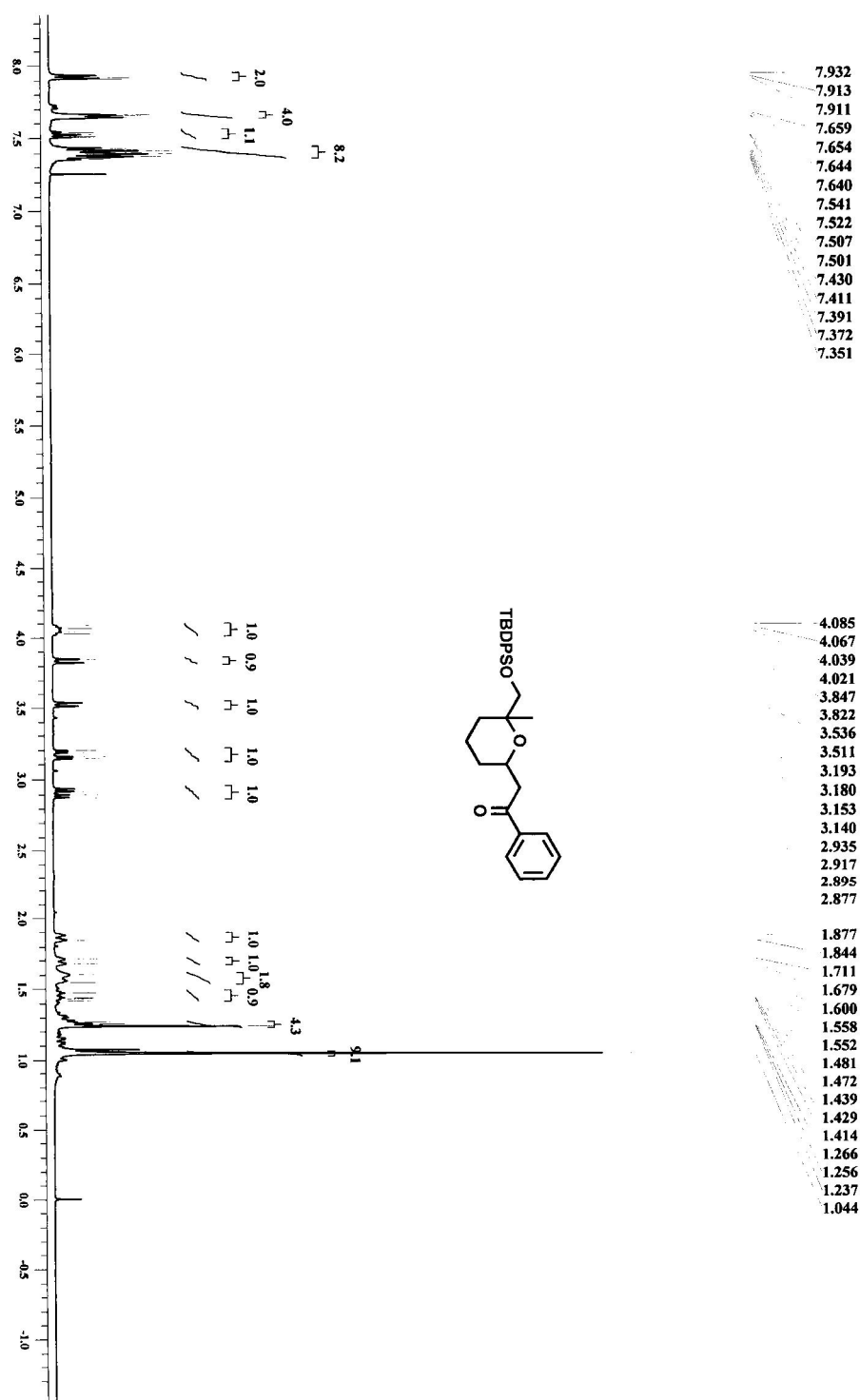


REPORT

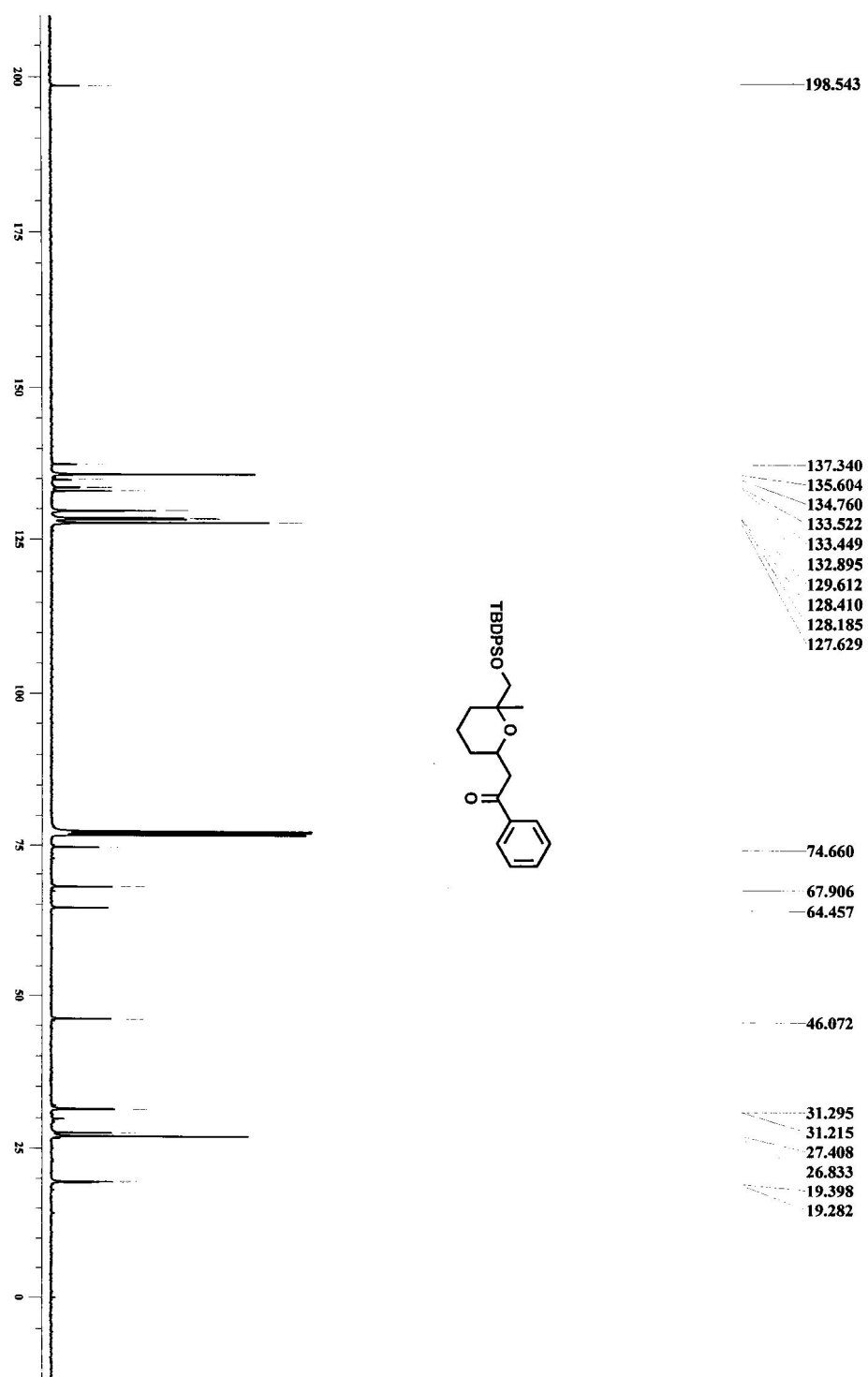
LC-LCMS Chromatogram of 13m



LC-LCMS Chromatogram of 13m



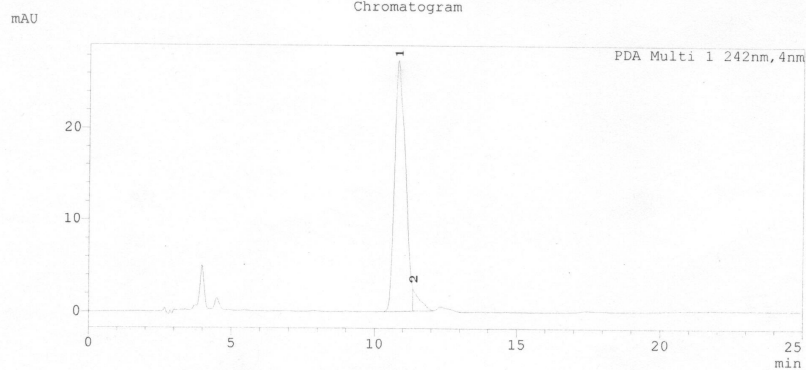
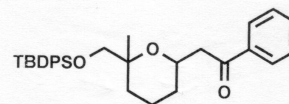
¹H NMR Spectrum of 13n (400 MHz, CDCl₃)



¹³C NMR Spectrum of 13n (100 MHz, CDCl₃)

Sample Code : DKM-YB-13N-486
 Data File : 17092019.6.lcd
 Method : CD HPLC-ISO.lcm
 Injection Volume : 10
 Date Acquired : 9/17/2019 3:53:22 PM
 Report File : Mass Data Report.lsr
 Chromatographic Conditions :

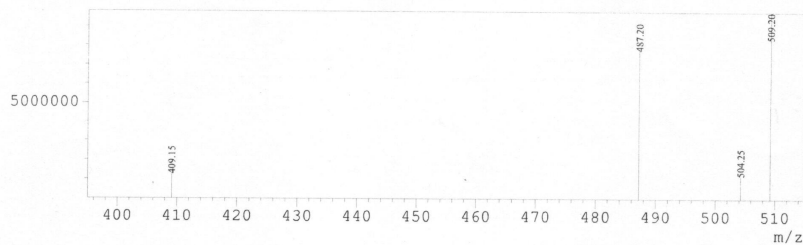
Chromatographic Conditions :
 Colum: LUNA C8 (250 X 4.6mm, 5.0u)
 Method: ISOCRATIC
 Mobile Phase: 90%MEOH IN 0.1%FA IN 100ML WATER



Peak Table

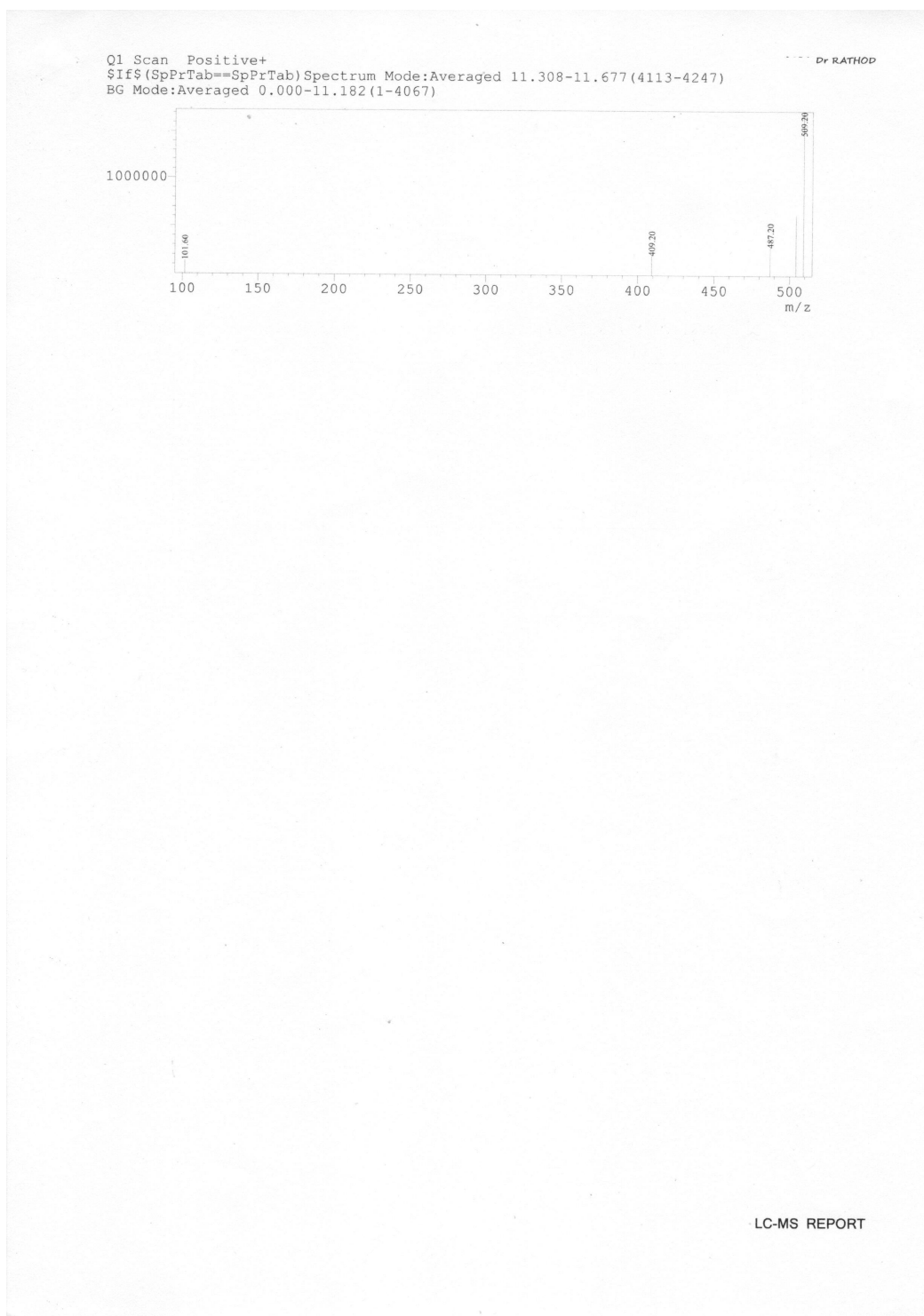
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	10.795	10.315	11.349	731058	94.759
2	11.365	11.349	12.064	40438	5.241
Total				771496	100.000

Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 10.593-11.039(3853-4015)
 BG Mode:Averaged 1.364-10.500(497-3819)



REPORT

LC-LCMS Chromatogram of 13n



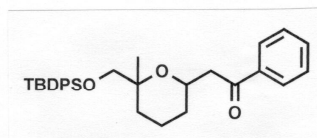
LC-LCMS Chromatogram of 13n

9/20/2019 18:23:04 1 / 1

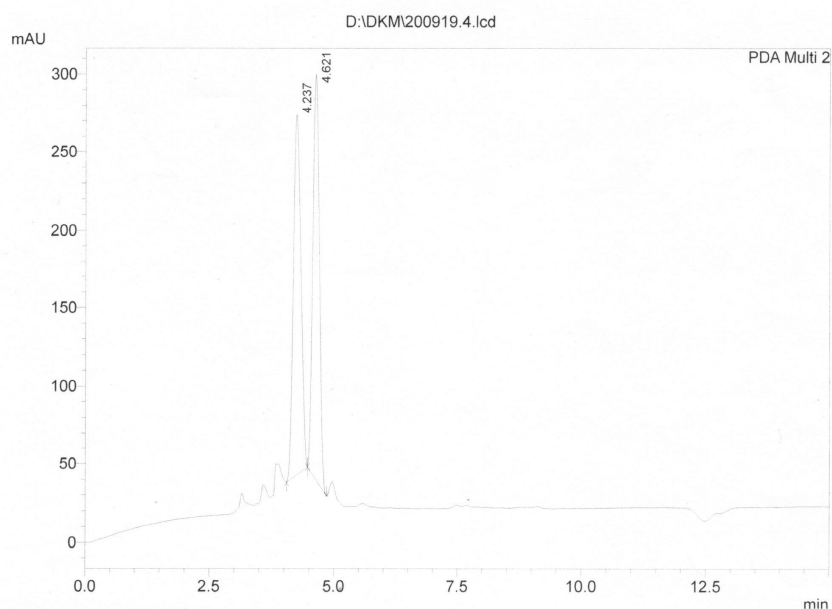
==== Shimadzu LCsolution Analysis Report ====

D:\DKM\200919.4.lcd

Acquired by : Manjula
 Sample Name : DKM-YB-TBDPS
 Sample ID : DKM-YB-TBDPS
 Tray# : 1
 Vial # : 14
 Injection Volume : 10 uL
 Data File Name : 200919.4.lcd
 Method File Name : 200919.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Data Acquired : 9/20/2019 6:03:11 PM
 Data Processed : 9/20/2019 6:18:37 PM
 COLUMN: CHIRAL PAK IA 250 X 4.6mm 5u
 MOBILE PHASE : 2% IPA IN HEXANE
 FLOW RATE: 1ml/min



<Chromatogram>

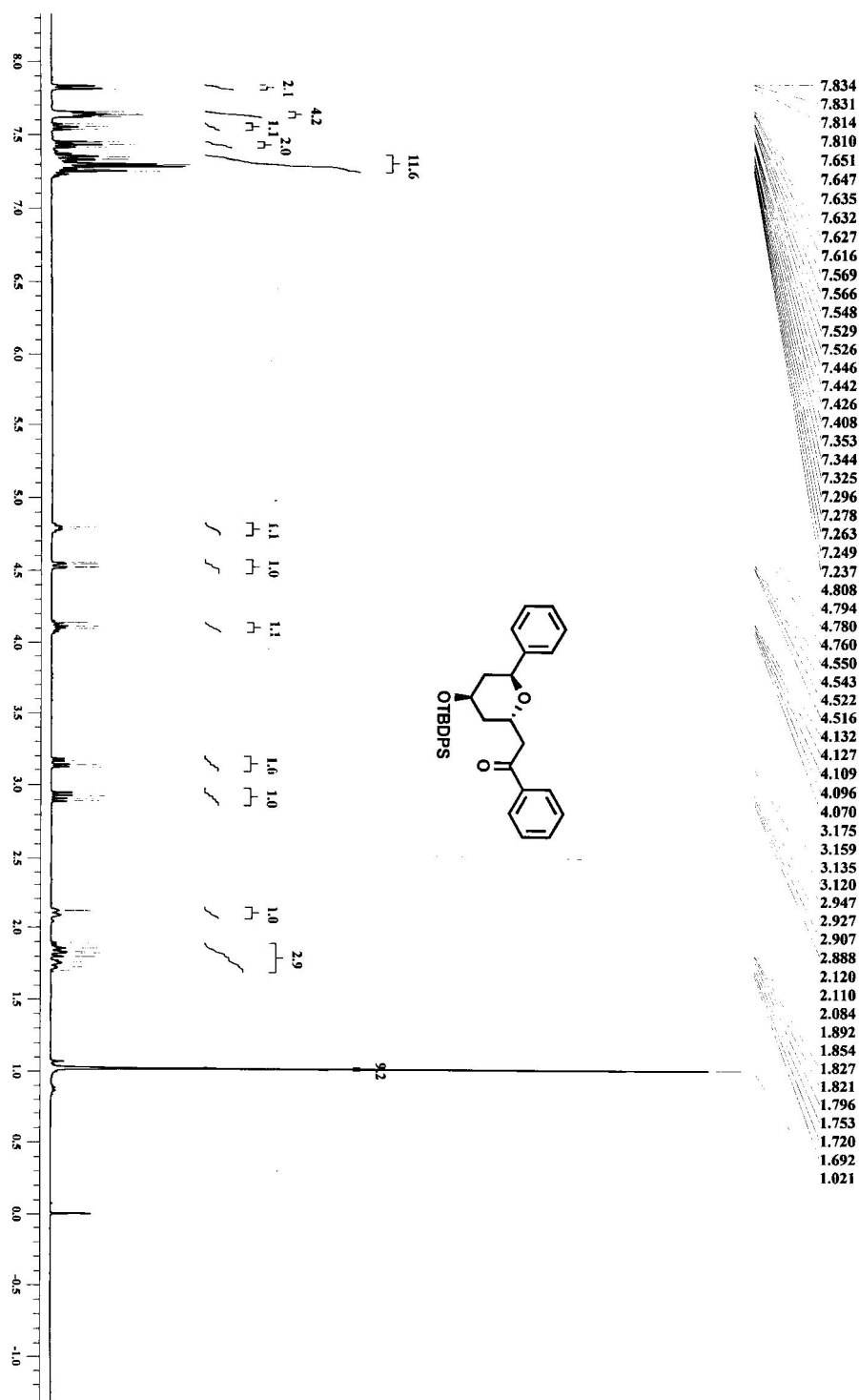


PeakTable

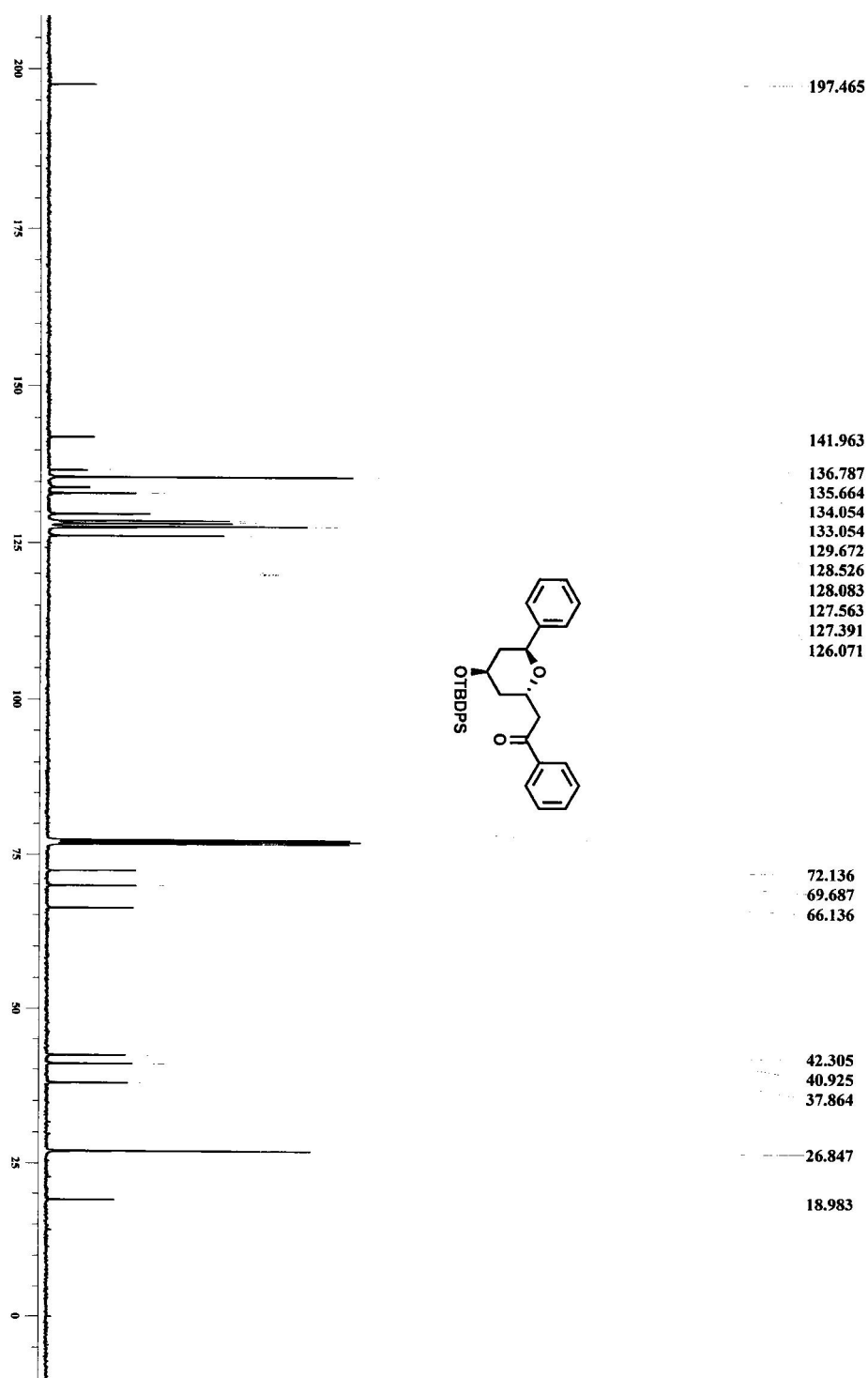
Peak#	Ret. Time	Area	Height	Area %	Height %
1	4.237	2437894	231169	50.557	47.126
2	4.621	2384193	259365	49.443	52.874
Total		4822087	490534	100.000	100.000

D:\DKM\200919.4.lcd

Chiral HPLC compound of 13n



¹H NMR Spectrum of 13o (400 MHz, CDCl₃)

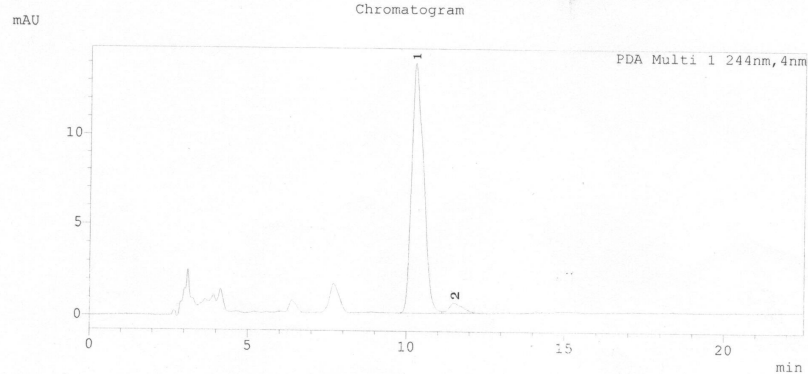
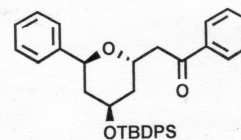


¹³C NMR Spectrum of 13o (100 MHz, CDCl₃)

LC-MS DATA REPORT

OSPC-Dr RATHOD

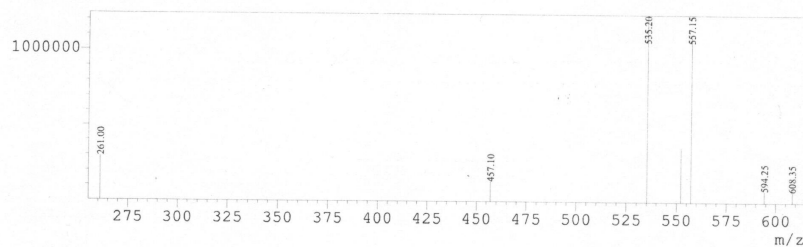
Sample Code : DKM-YB-130-534
 Data File : 17092019.4.lcd
 Method : CD HPLC-ISO.lcm
 Injection Volume : 10
 Date Acquired : 9/17/2019 3:00:56 PM
 Report File : Mass Data Report.lsr
 Chromatographic Conditions : Chromatographic Conditions :
 Colum: LUNA C8 (250 X 4.6mm, 5.0u)
 Method: ISOCRATIC
 Mobile Phase: 90%MEOH IN 0.1%FA IN 100ML WATER



Peak Table

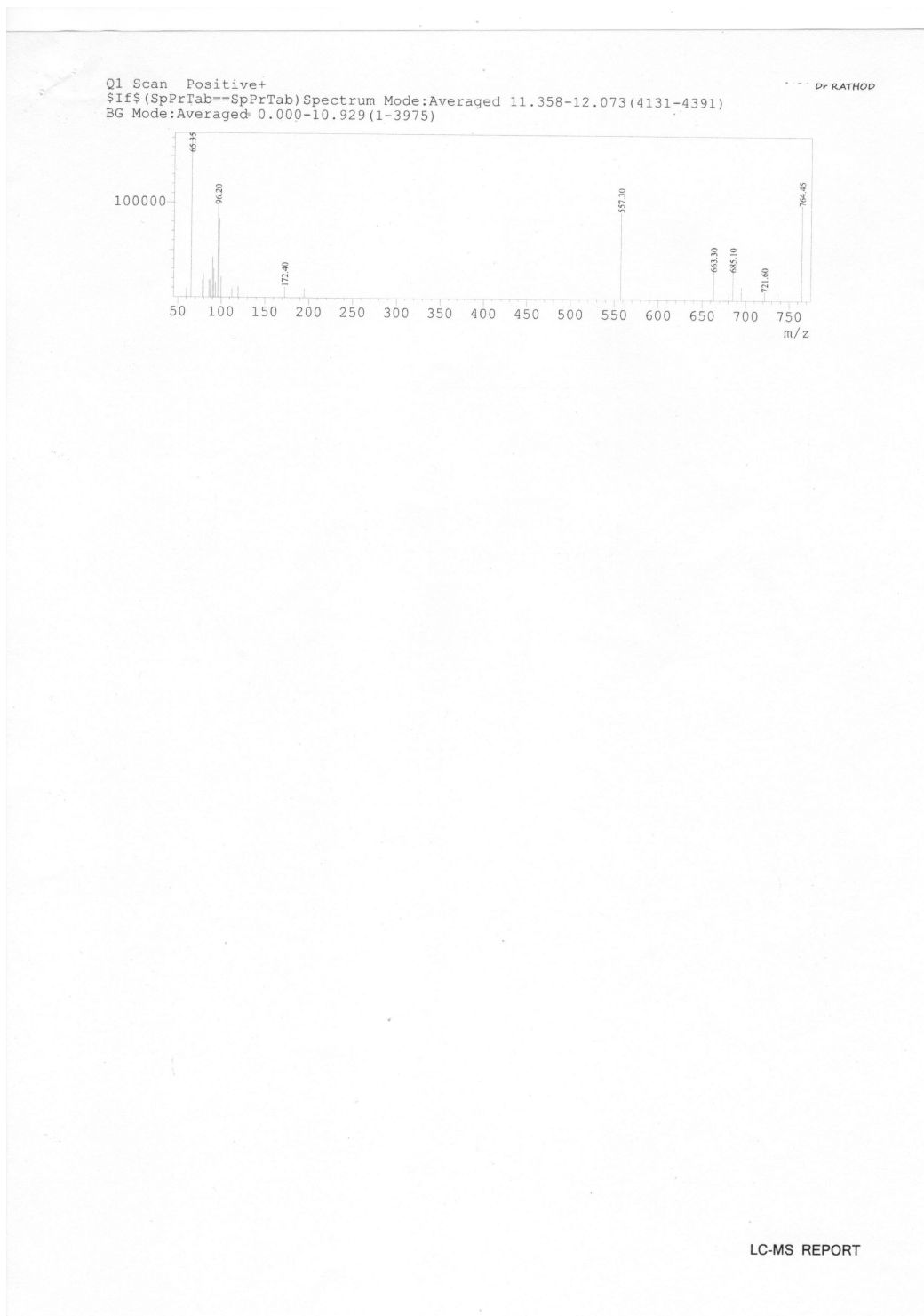
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	10.246	9.792	11.125	362264	96.039
2	11.530	11.125	12.149	14941	3.961
Total				377205	100.000

Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 9.955-10.797(3621-3927)
 BG Mode:Averaged 0.000-9.752(1-3547)

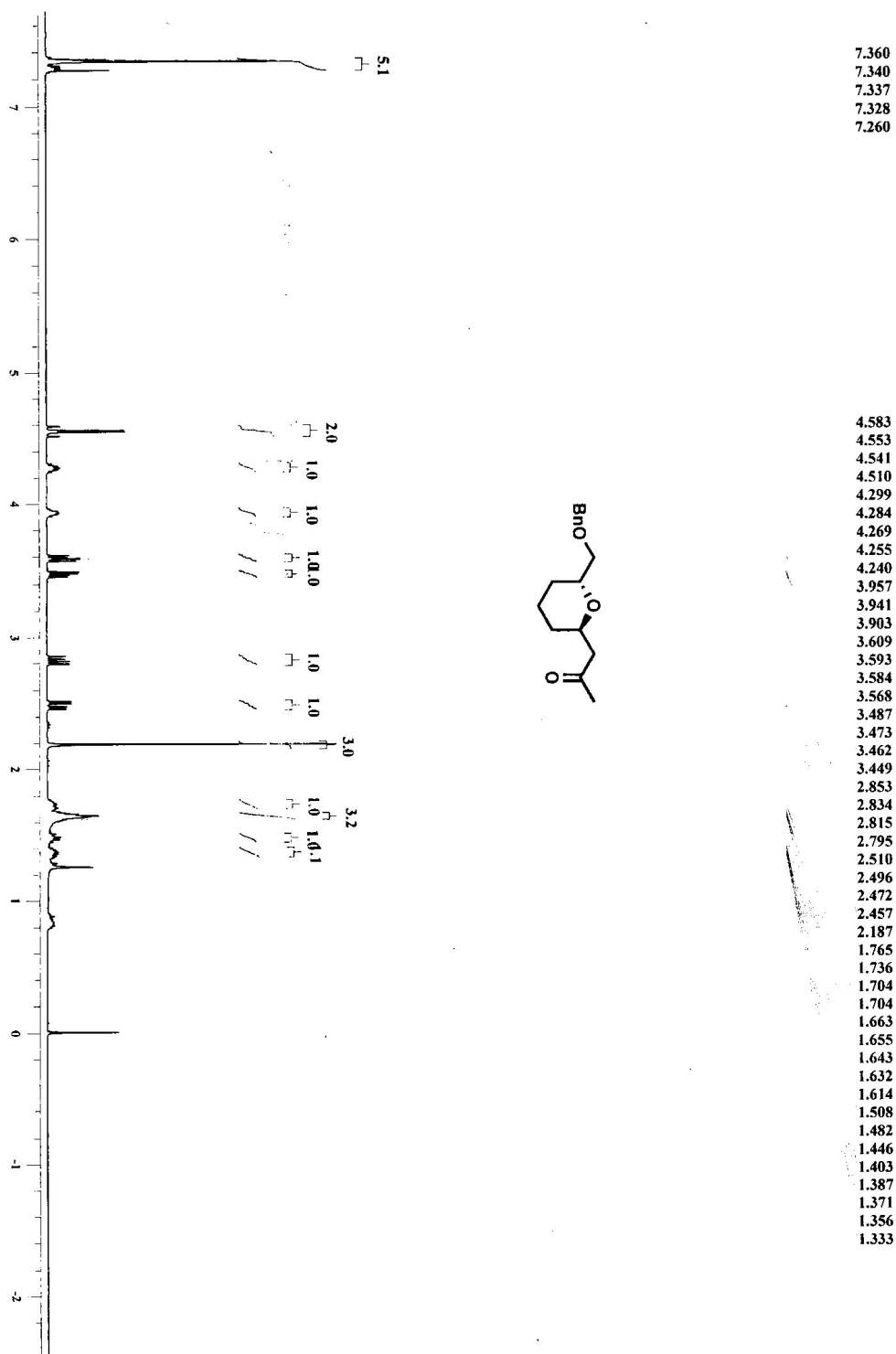


REPORT

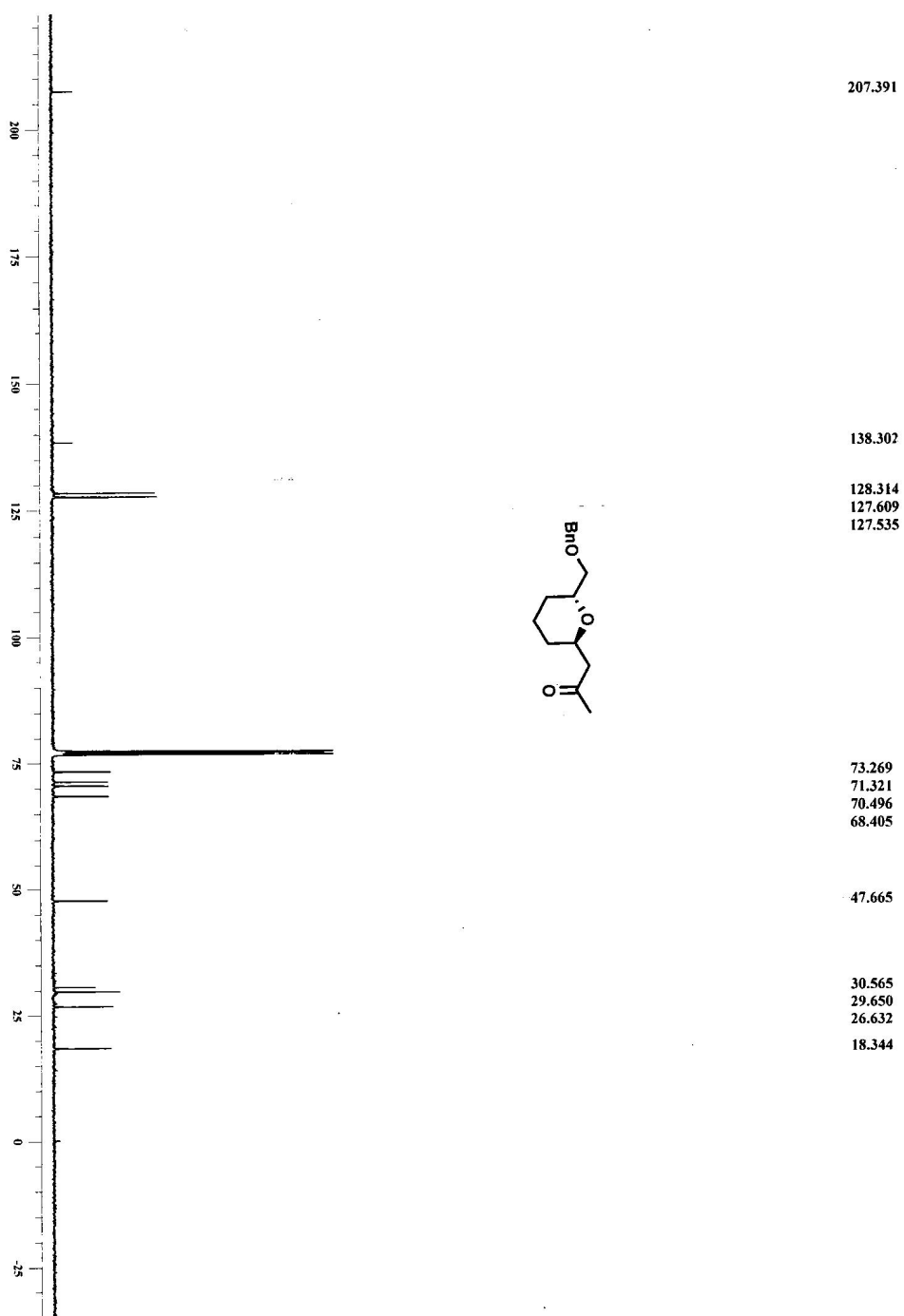
LC-LCMS Chromatogram of 13o



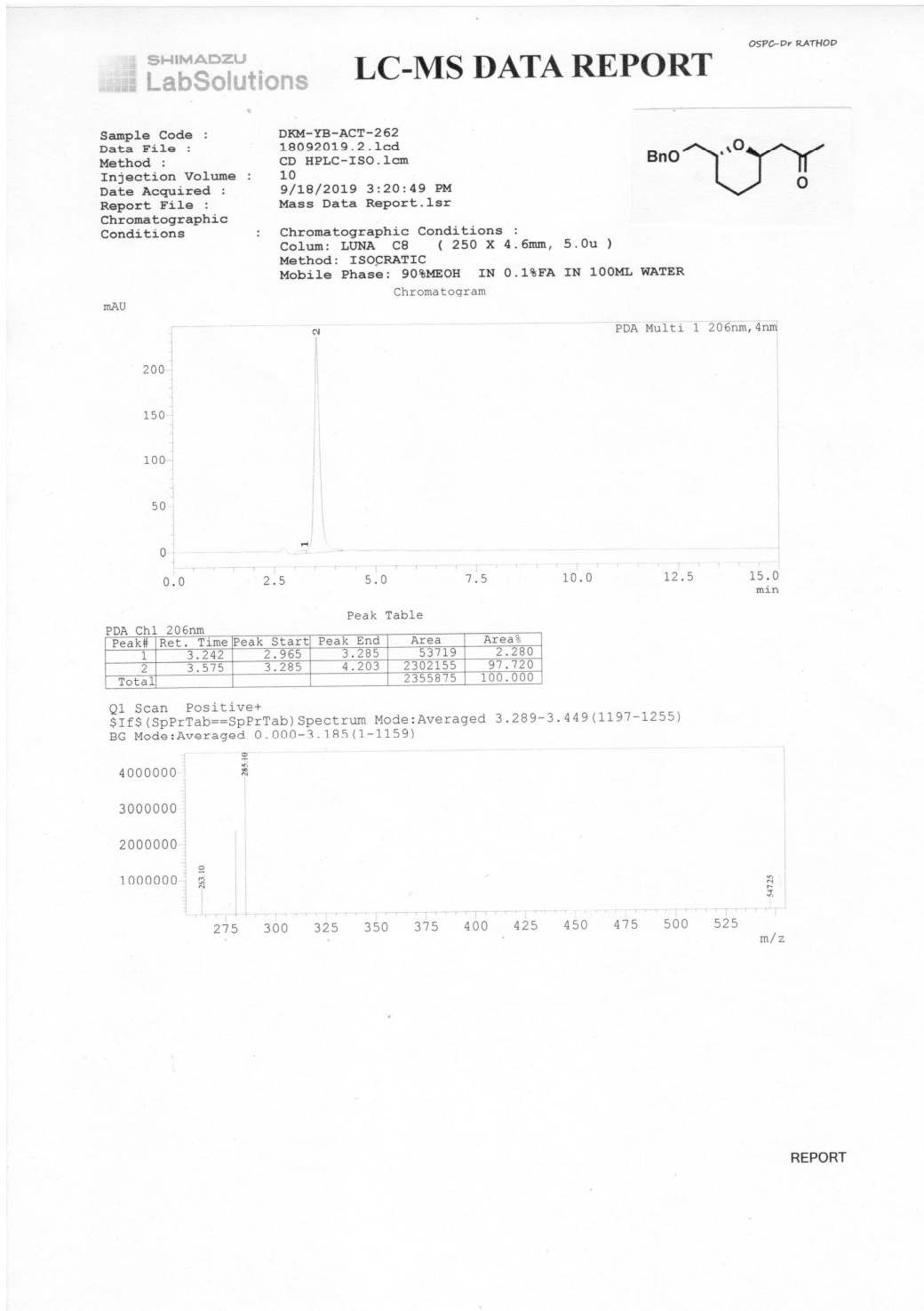
LC-LCMS Chromatogram of 13o



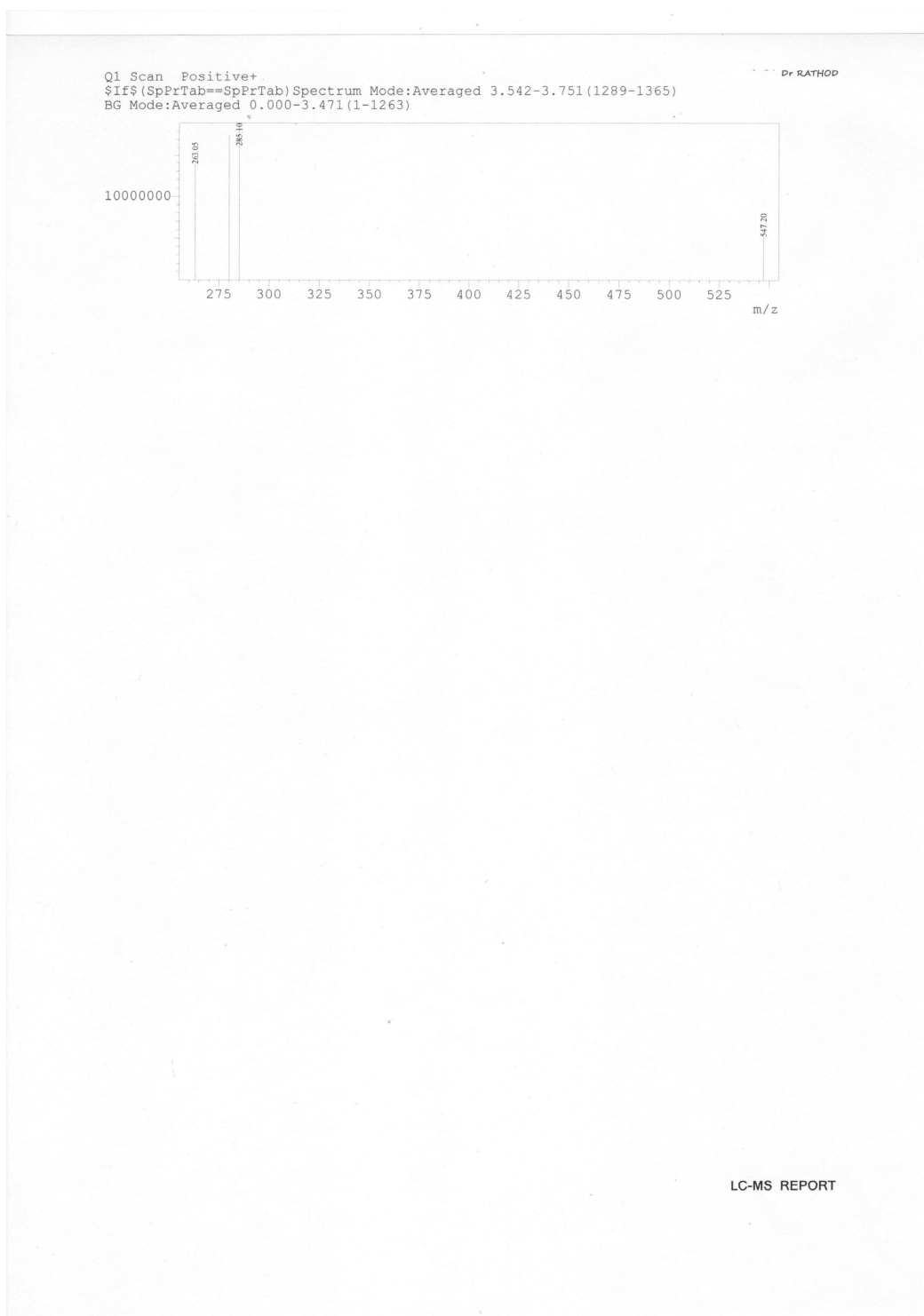
¹H NMR Spectrum of 14 (400 MHz, CDCl₃)



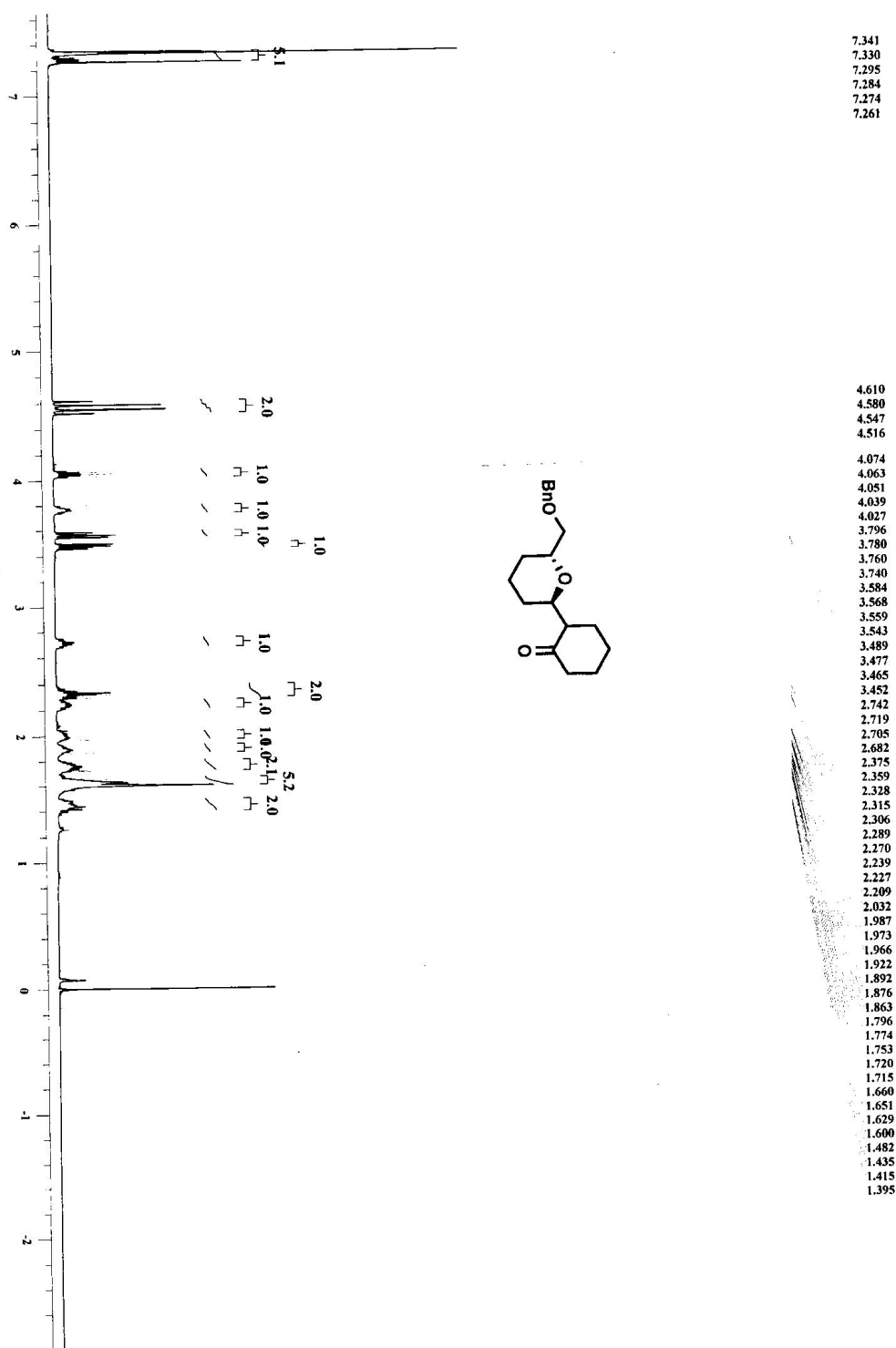
^{13}C NMR Spectrum of 14 (100 MHz, CDCl_3)

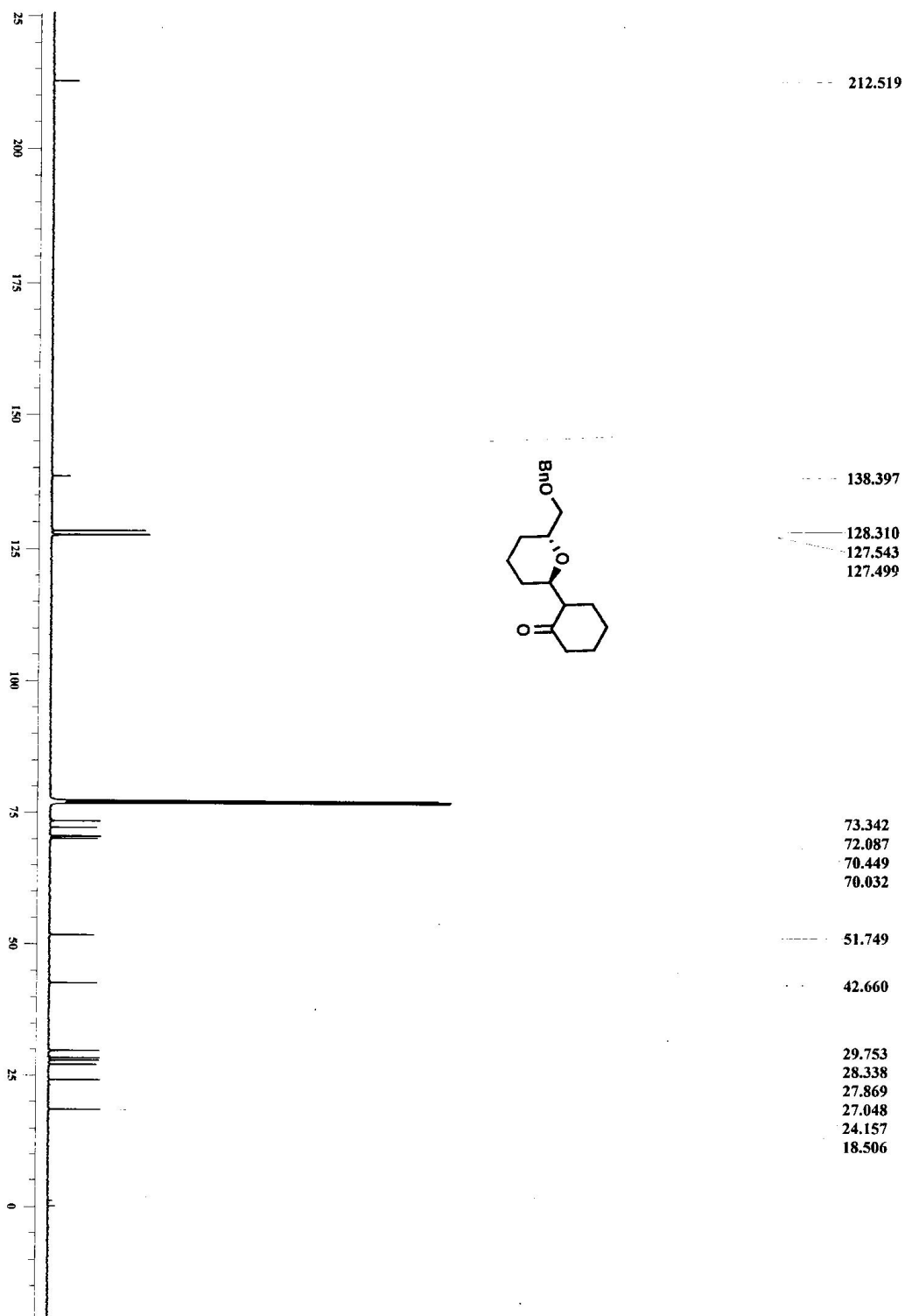


LC-LCMS Chromatogram of 14



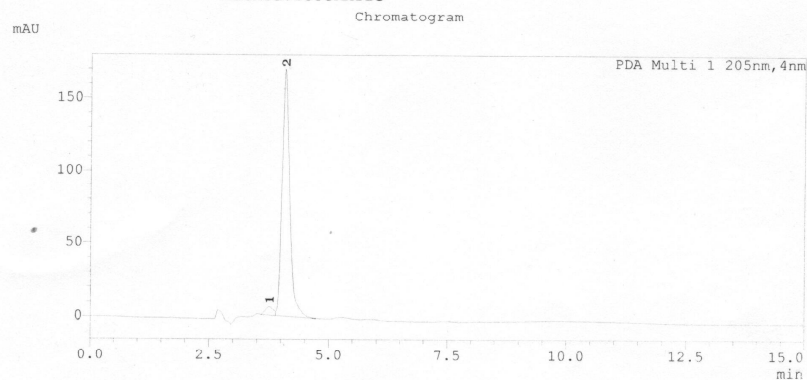
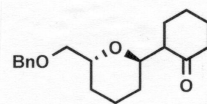
LC-LCMS Chromatogram of 14

¹H NMR Spectrum of 15 (400 MHz, CDCl₃)



¹³C NMR Spectrum of 15 (100 MHz, CDCl₃)

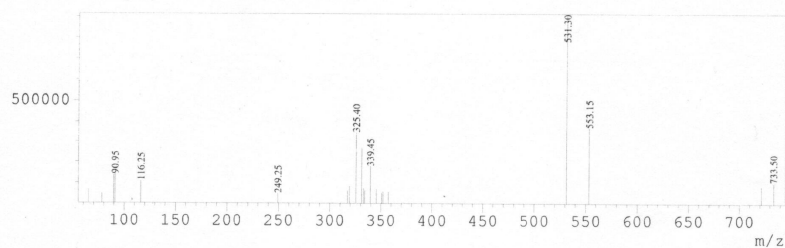
Sample Code : DKM-YB-CYC-302
 Data File : 19092019.2.lcd
 Method : CD HPLC-ISO.lcm
 Injection Volume : 10
 Date Acquired : 9/19/2019 2:59:04 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions : Chromatographic Conditions :
 Colum: LUNA C8 (250 X 4.6mm, 5.0u)
 Method: ISOCRATIC



Peak Table

Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	3.758	3.584	3.893	63081	3.251
2	4.077	3.893	4.768	1877175	96.749
Total				1940256	100.000

Q1 Scan Positive+
 \$Ii\$(SpPrTab==SpPrTab) Spectrum Mode:Averaged 3.746-3.894 (1363-1417)
 BG Mode:Averaged 0.055-3.597 (21-1309)

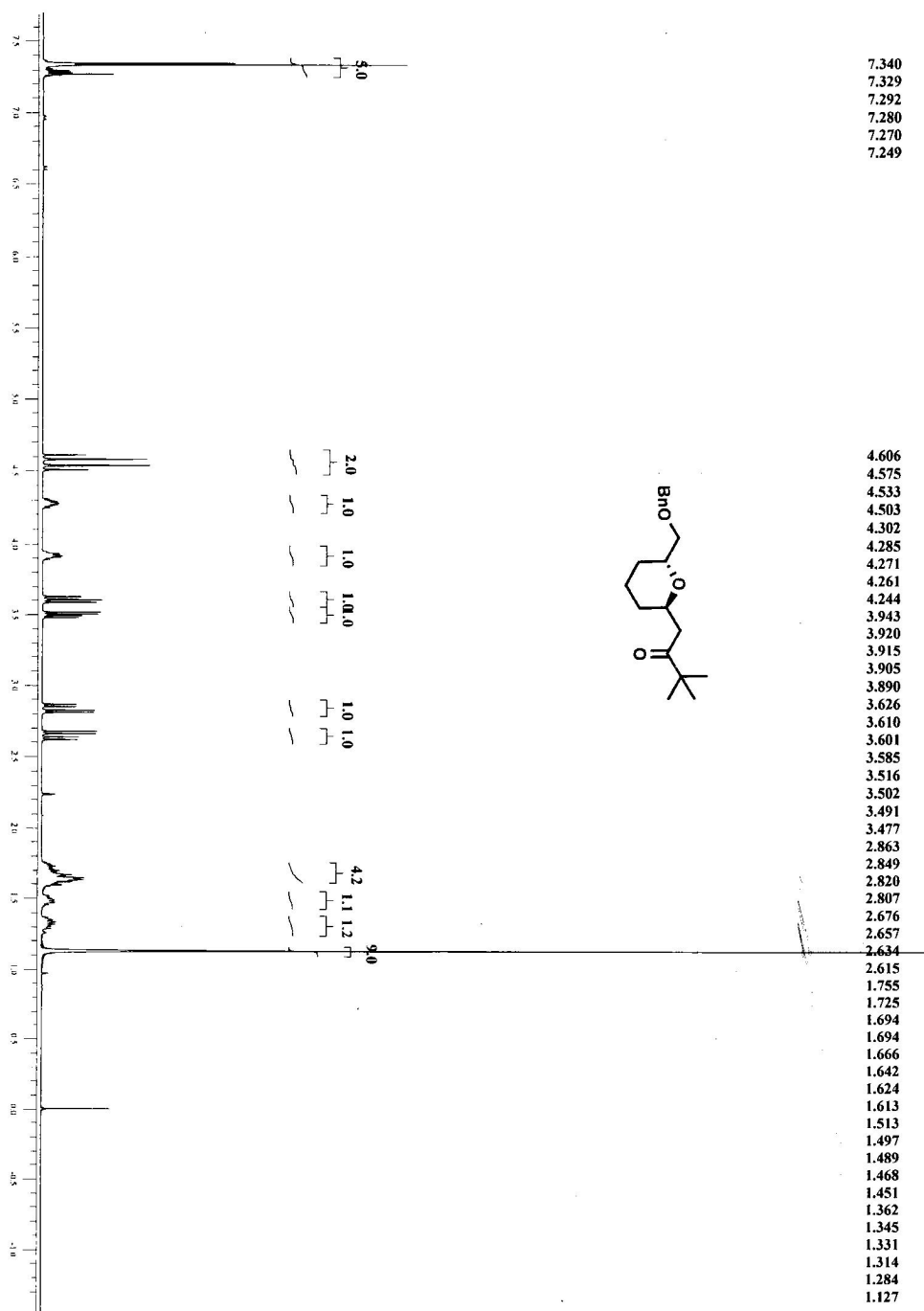


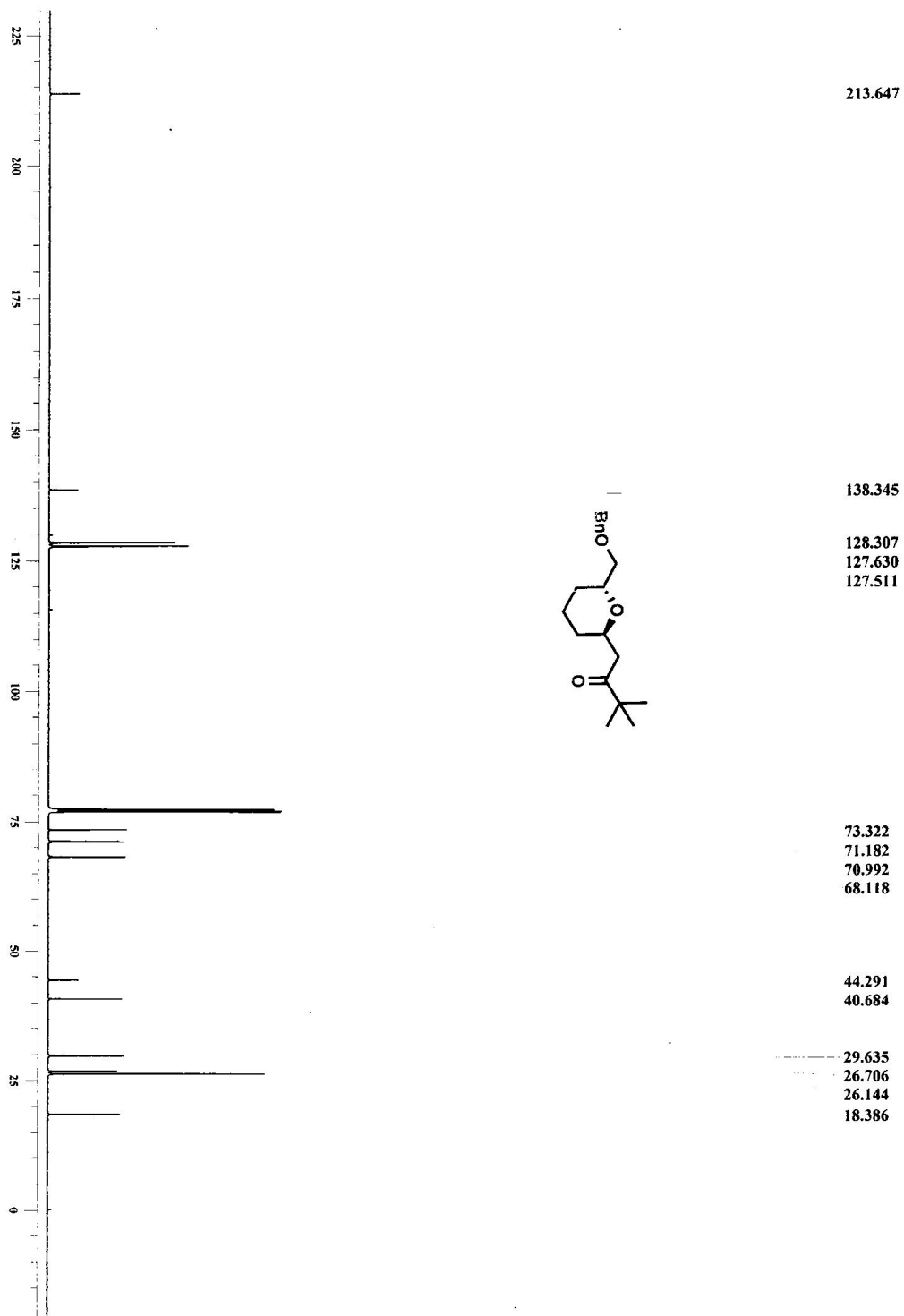
REPORT

LC-LCMS Chromatogram of 15



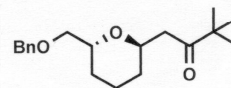
LC-LCMS Chromatogram of 15

 ^1H NMR Spectrum of 16 (400 MHz, CDCl_3)

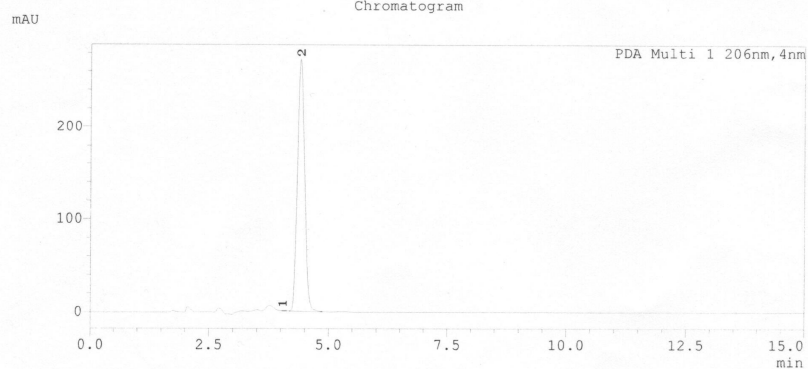


^{13}C NMR Spectrum of 16 (125 MHz, CDCl_3)

Sample Code : DKM-YB-BUT-304
 Data File : 18092019.1.lcd
 Method : CD HPLC-ISO.lcm
 Injection Volume : 10
 Date Acquired : 9/18/2019 3:02:58 PM
 Report File : Mass Data Report.lsr
 Chromatographic Conditions :



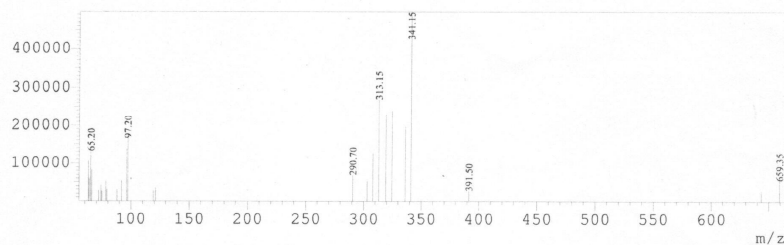
Chromatographic Conditions :
 Colum: LUNA C8 (250 X 4.6mm, 5.0u)
 Method: ISOCRATIC
 Mobile Phase: 90%MEOH IN 0.1%FA IN 100ML WATER



Peak Table

Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	4.038	4.021	4.181	488	0.017
2	4.401	4.181	4.949	2865638	99.983
Total				2866126	100.000

Q1 Scan Positive+
 \$If\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 3.988-4.197(1451-1527)
 BG Mode:Averaged 3.905-3.988(1421-1451)

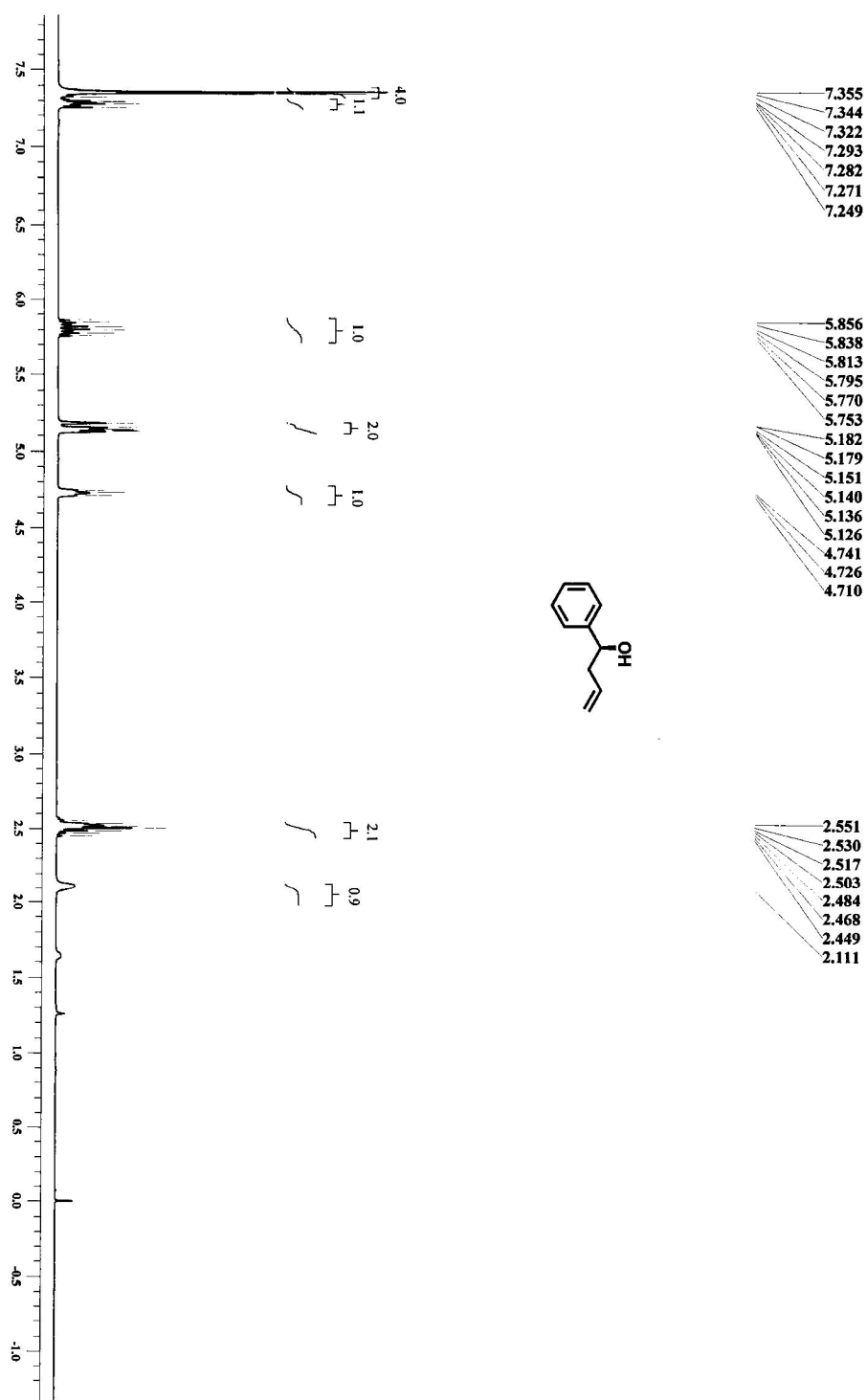


REPORT

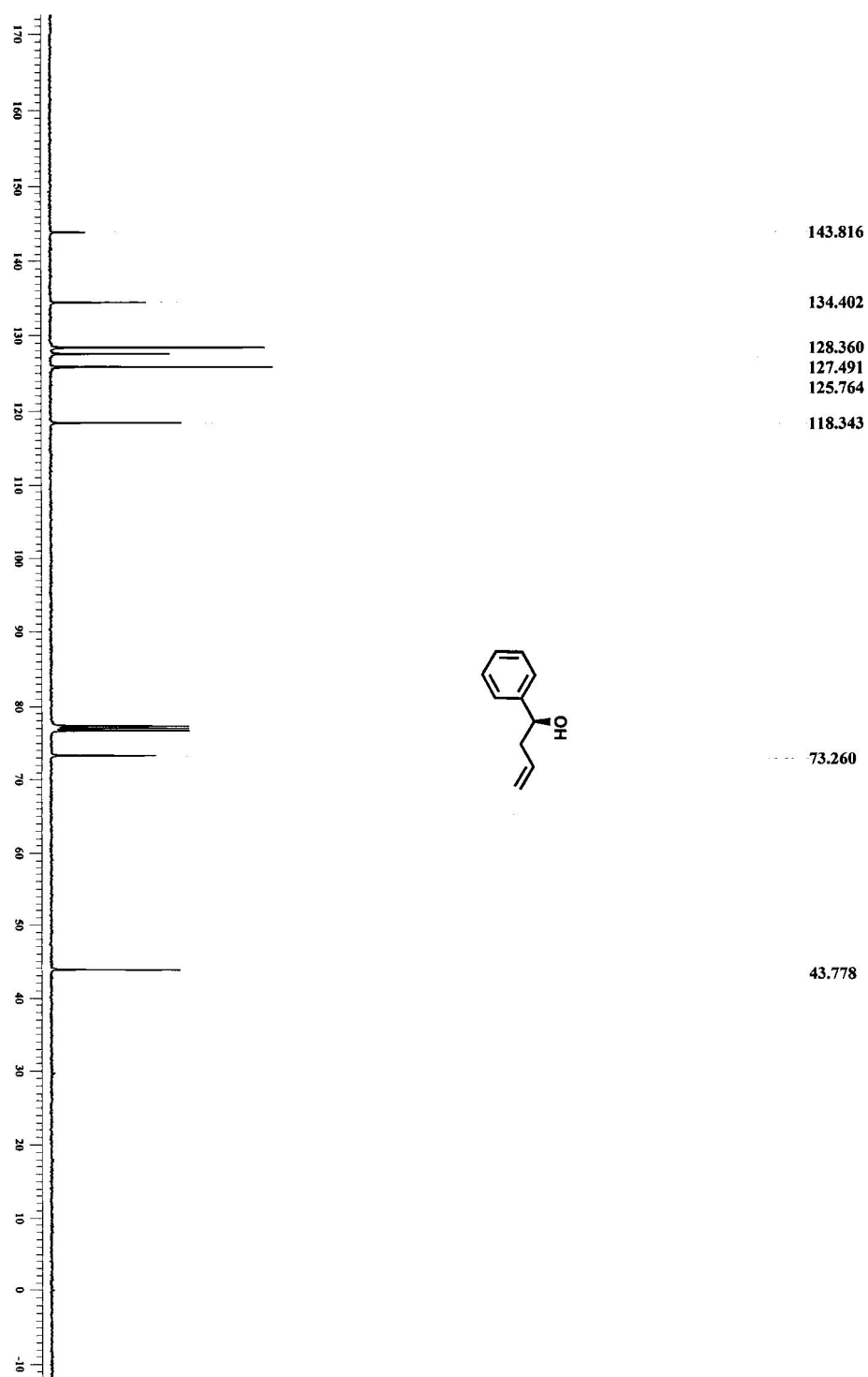
LC-LCMS Chromatogram of 16



LC-LCMS Chromatogram of 16



^1H NMR Spectrum of 20 (400 MHz, CDCl_3)



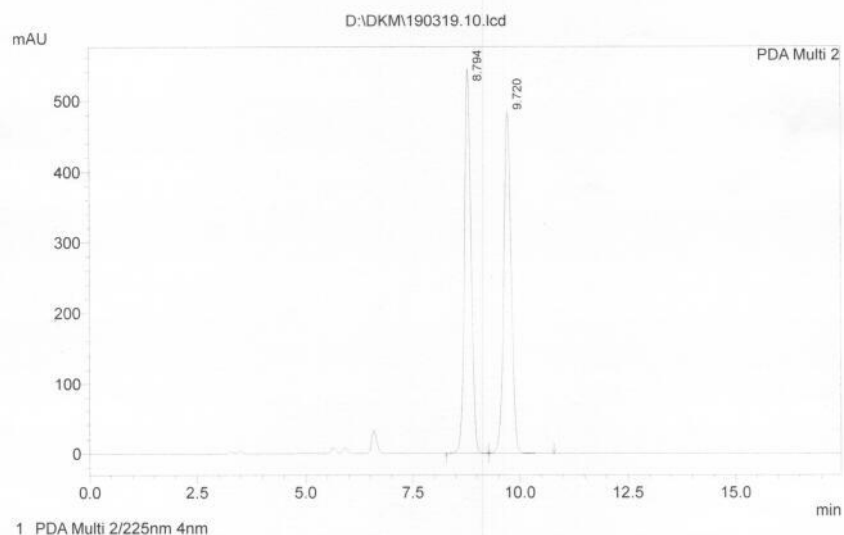
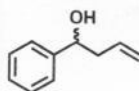
^{13}C NMR Spectrum of 20 (125 MHz, CDCl_3)

3/19/2019 18:08:41 1 / 1

==== Shimadzu LCsolution Analysis Report ====

Acquired by : Manjula
 Sample Name : DKM-YBR-RACEMIC
 Sample ID : DKM-YBR-RACEMIC
 Tray# : 1
 Vial # : 36
 Injection Volume : 5 uL
 Data File Name : 190319.10.lcd
 Method File Name : 190319.lcm
 Batch File Name :
 Report File Name : Default.lcr
 Data Acquired : 3/19/2019 5:11:10 PM
 Data Processed : 3/19/2019 5:28:41 PM
 COLUMN: CHIRAL PAK IC 250X4.6mm 5u
 MOBILE PHASE : 20% IPA IN HEXANE
 FLOW RATE: 1ml/min
 FLOW RATE : 1ml/min

D:\DKM\190319.10.lcd



Peak Table

Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.794	5893732	545000	50.221	53.015
2	9.720	5841783	483017	49.779	46.985
Total		11735515	1028017	100.000	100.000

D:\DKM\190319.10.lcd

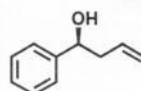
Chiral HPLC Chromatogram for Racemic Compound of 20

3/19/2019 18:10:16 1 / 1

==== Shimadzu LCsolution Analysis Report ====

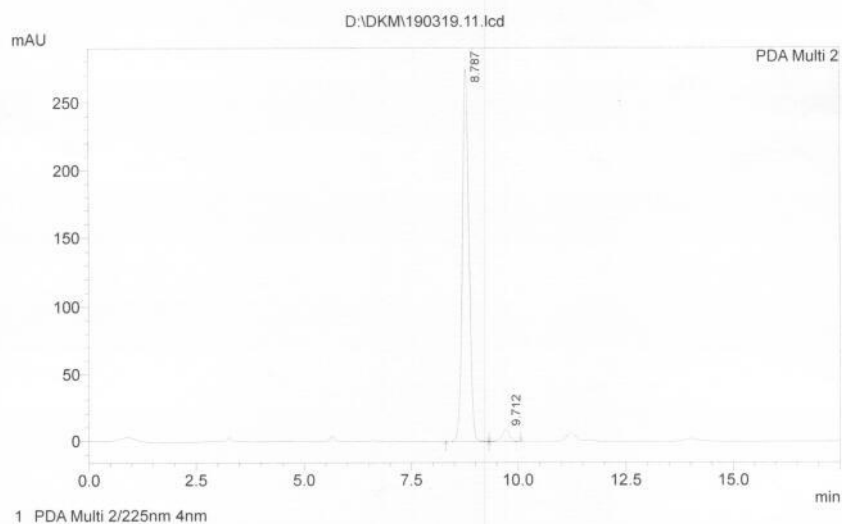
D:\DKM\190319.11.lcd

Acquired by : Manjula
Sample Name : DKM-YBR-1
Sample ID : DKM-YBR-1
Tray# : 1
Vial # : 37
Injection Volume : 5 uL
Data File Name : 190319.11.lcd
Method File Name : 190319.lcm
Batch File Name :
Report File Name : Default.lcr
Data Acquired : 3/19/2019 5:29:44 PM
Data Processed : 3/19/2019 5:47:20 PM
COLUMN: CHIRAL PAK IC 250X4.6mm 5u
MOBILE PHASE : 20% IPA IN HEXANE
FLOW RATE: 1ml/min



FLOW RATE : 1ml/min

<Chromatogram>



1 PDA Multi 2/225nm 4nm

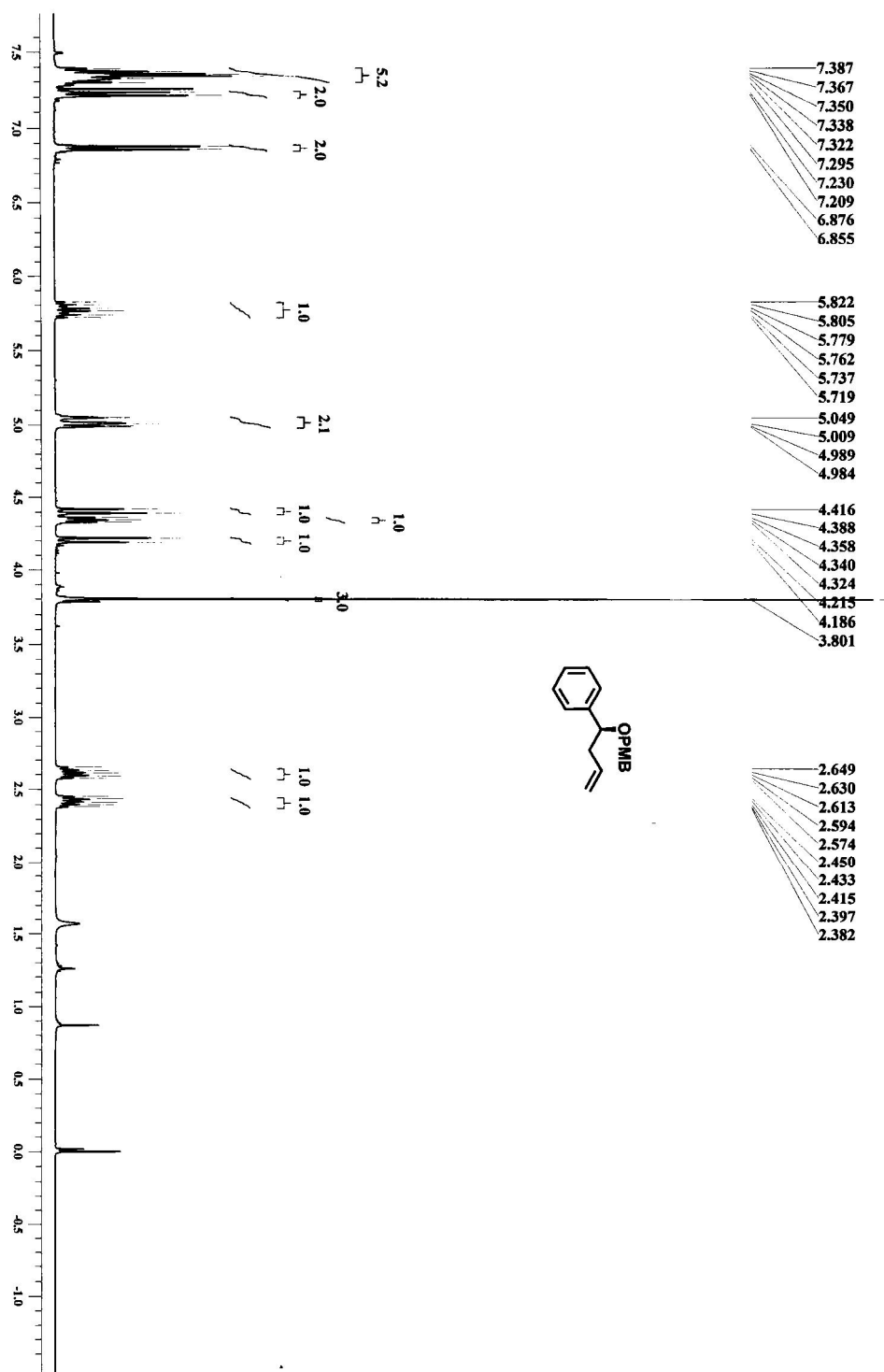
PeakTable

PDA Ch2 225nm 4nm

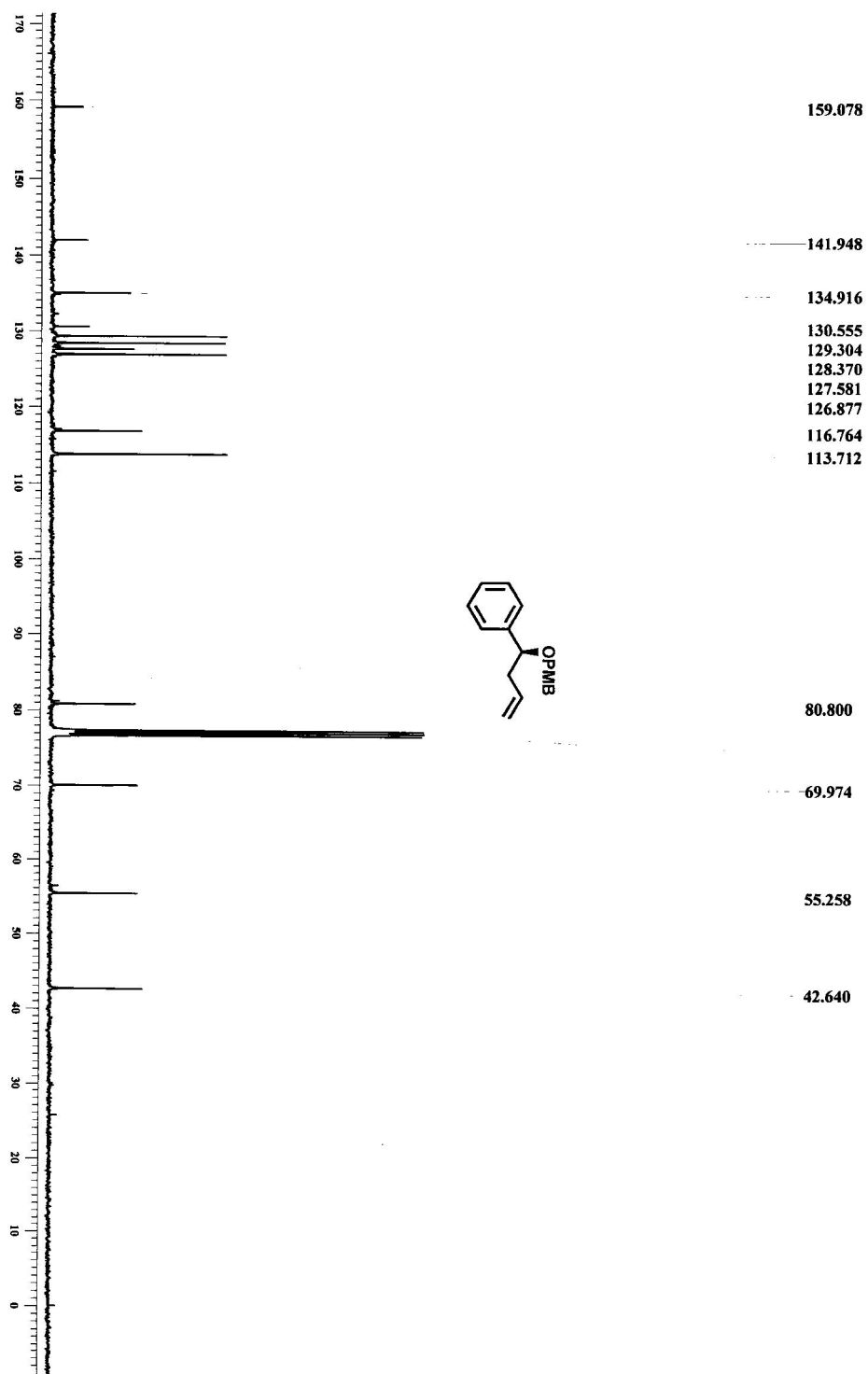
Peak#	Ret. Time	Area	Height	Area %	Height %
1	8.787	2960349	275200	96.438	96.819
2	9.712	109341	9043	3.562	3.181
Total		3069690	284243	100.000	100.000

D:\DKM\190319.11.lcd

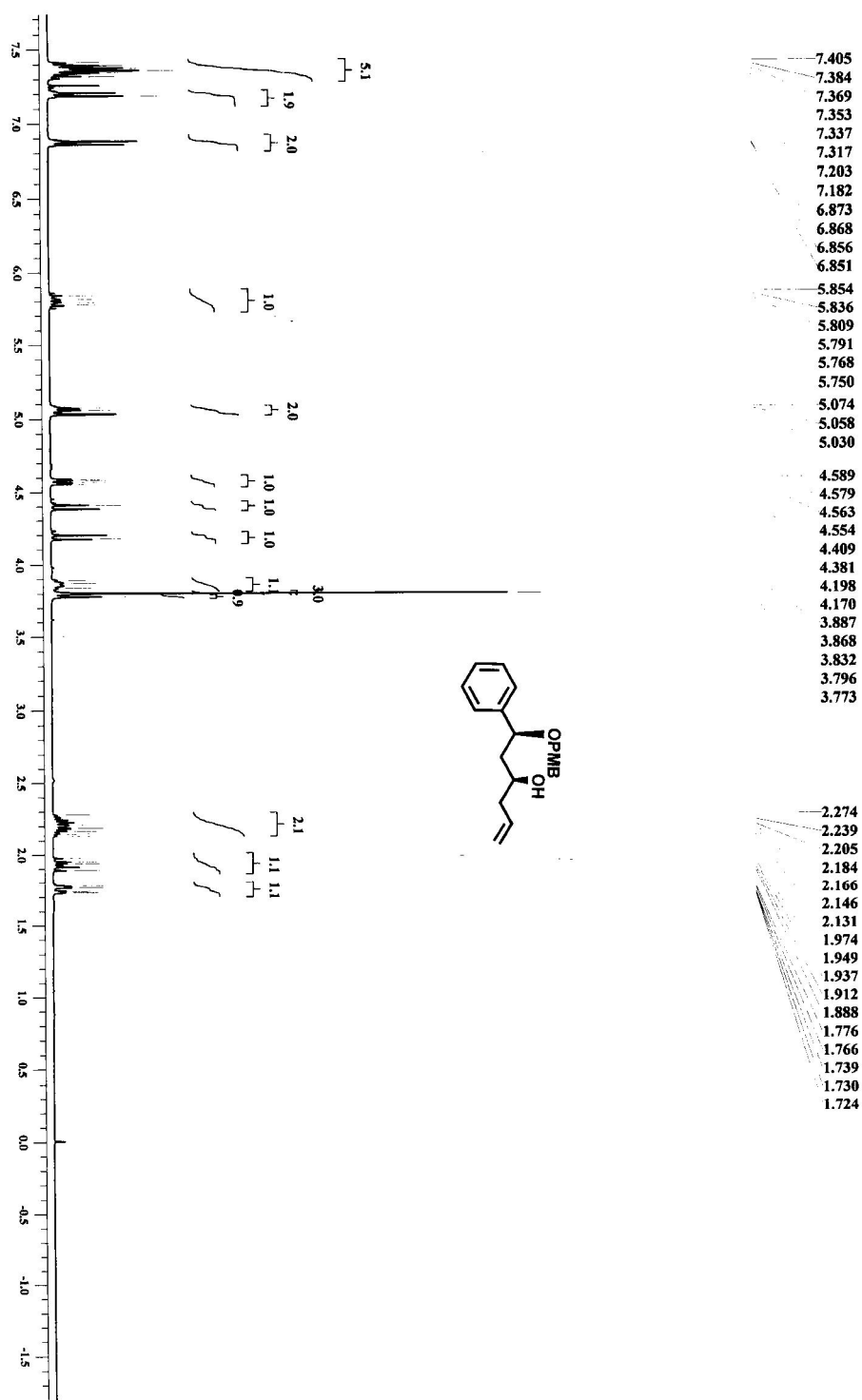
Chiral HPLC Chromatogram of Compound 20



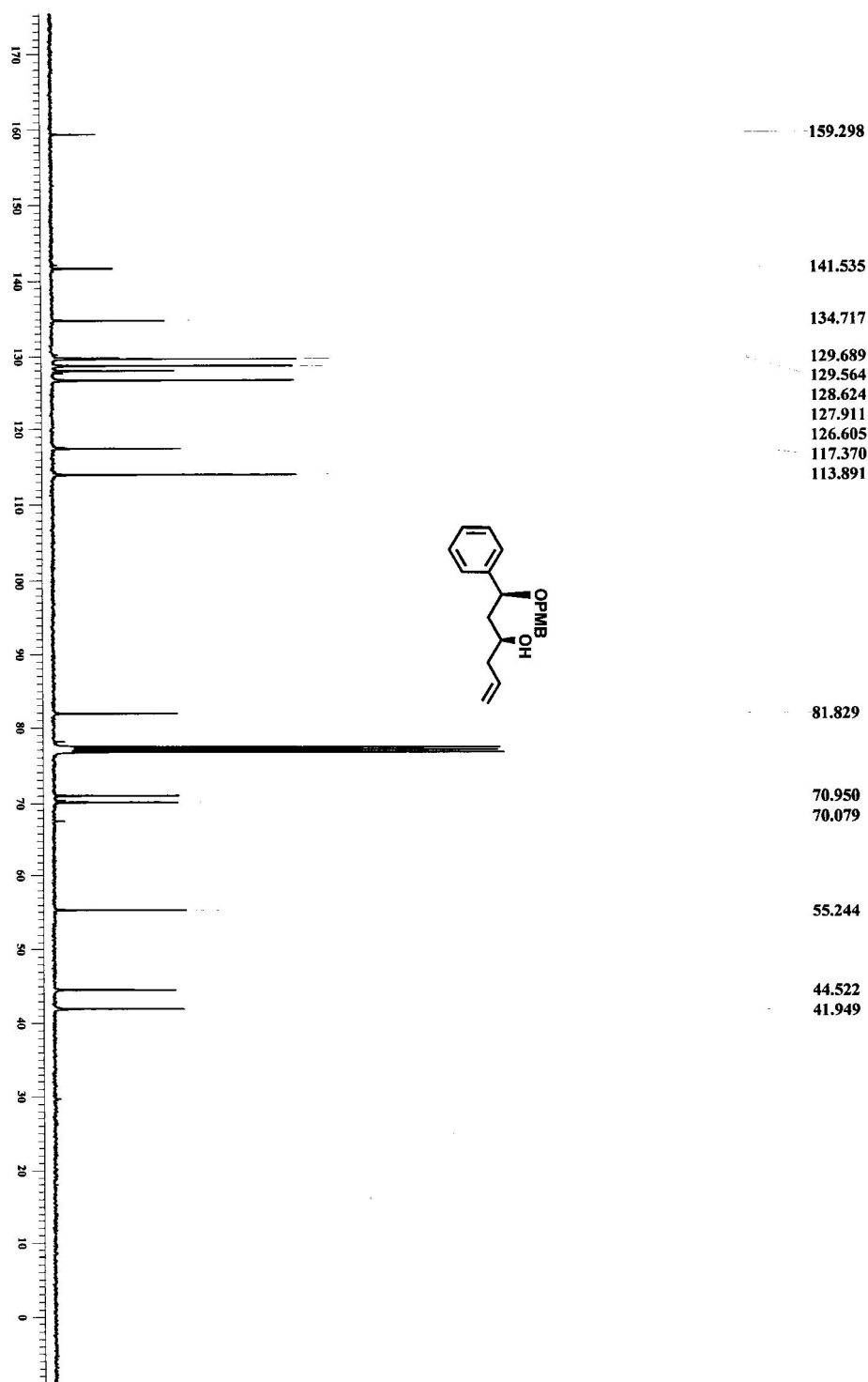
^1H NMR Spectrum of 21 (400 MHz, CDCl_3)



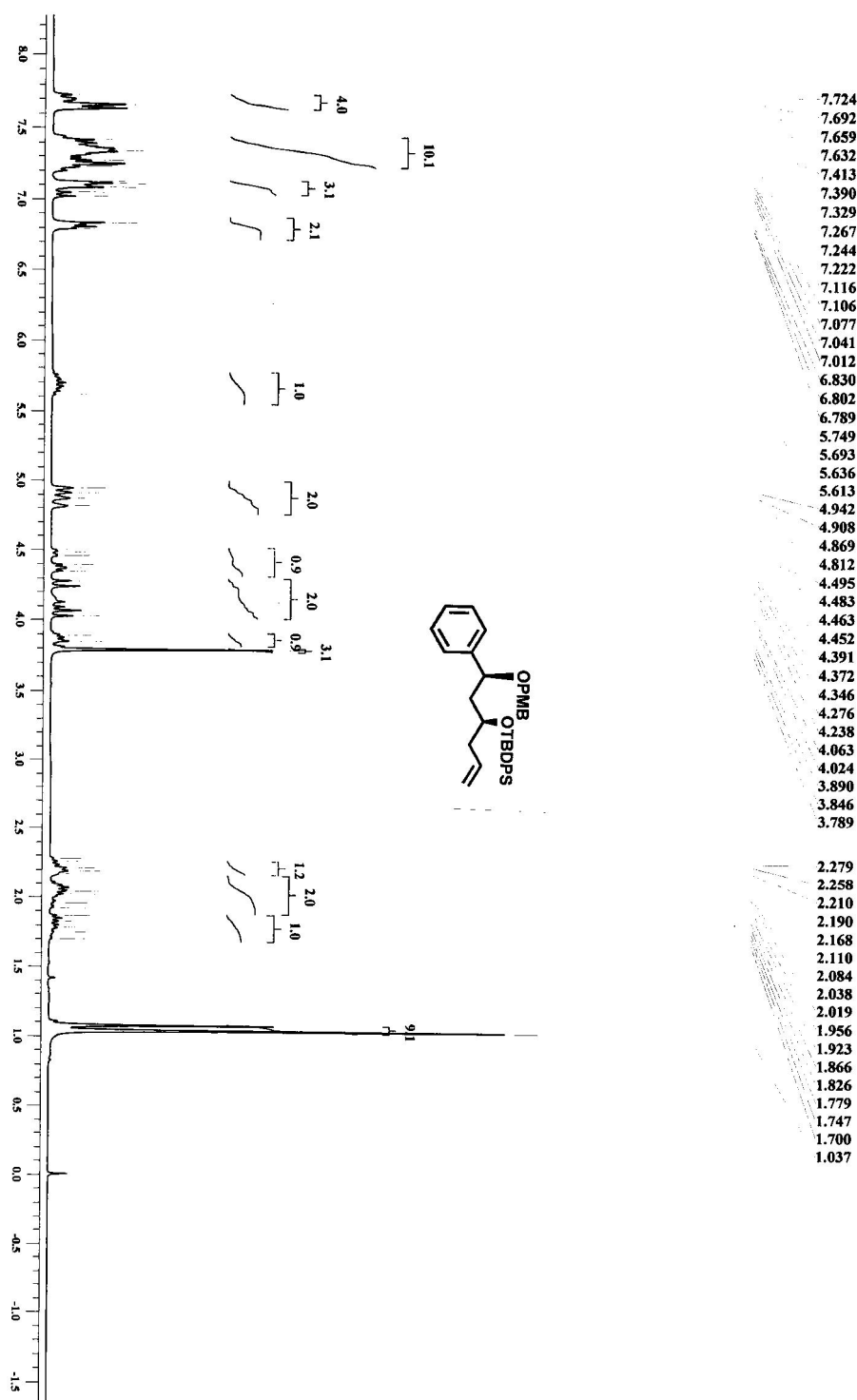
¹³C NMR Spectrum of 21 (100 MHz, CDCl₃)



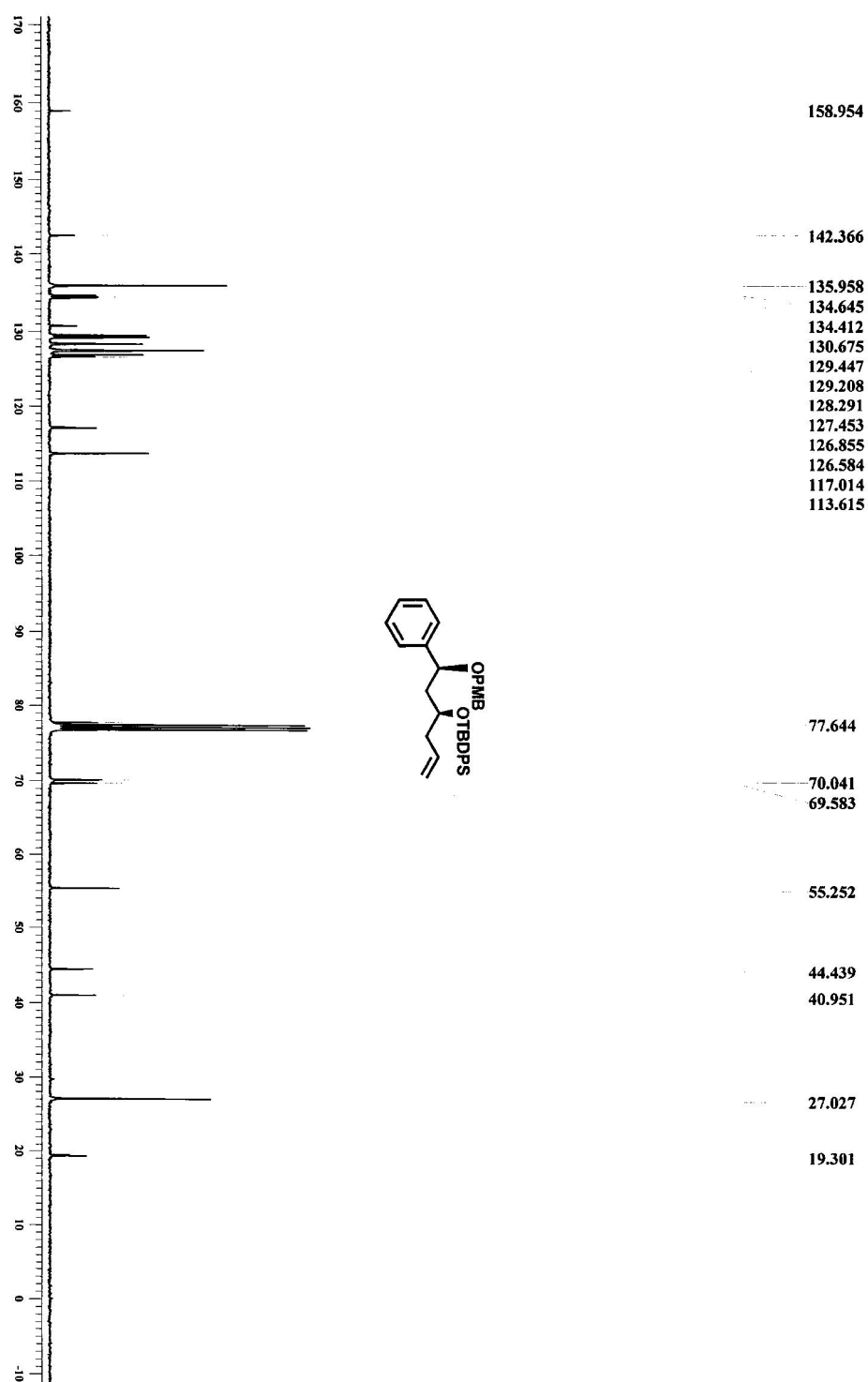
¹H NMR Spectrum of 22 (400 MHz, CDCl₃)



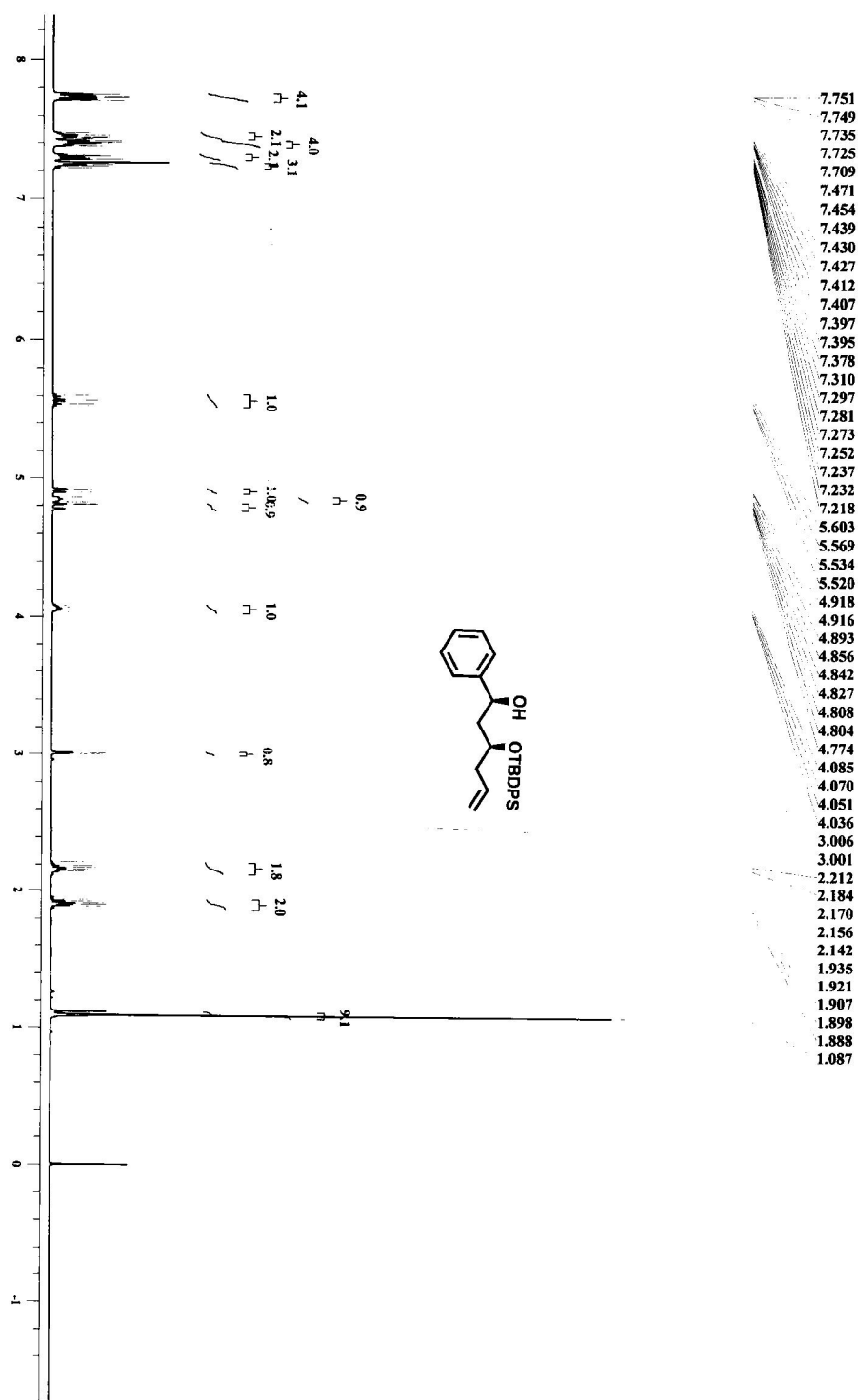
^{13}C NMR Spectrum of 22 (100 MHz, CDCl_3)



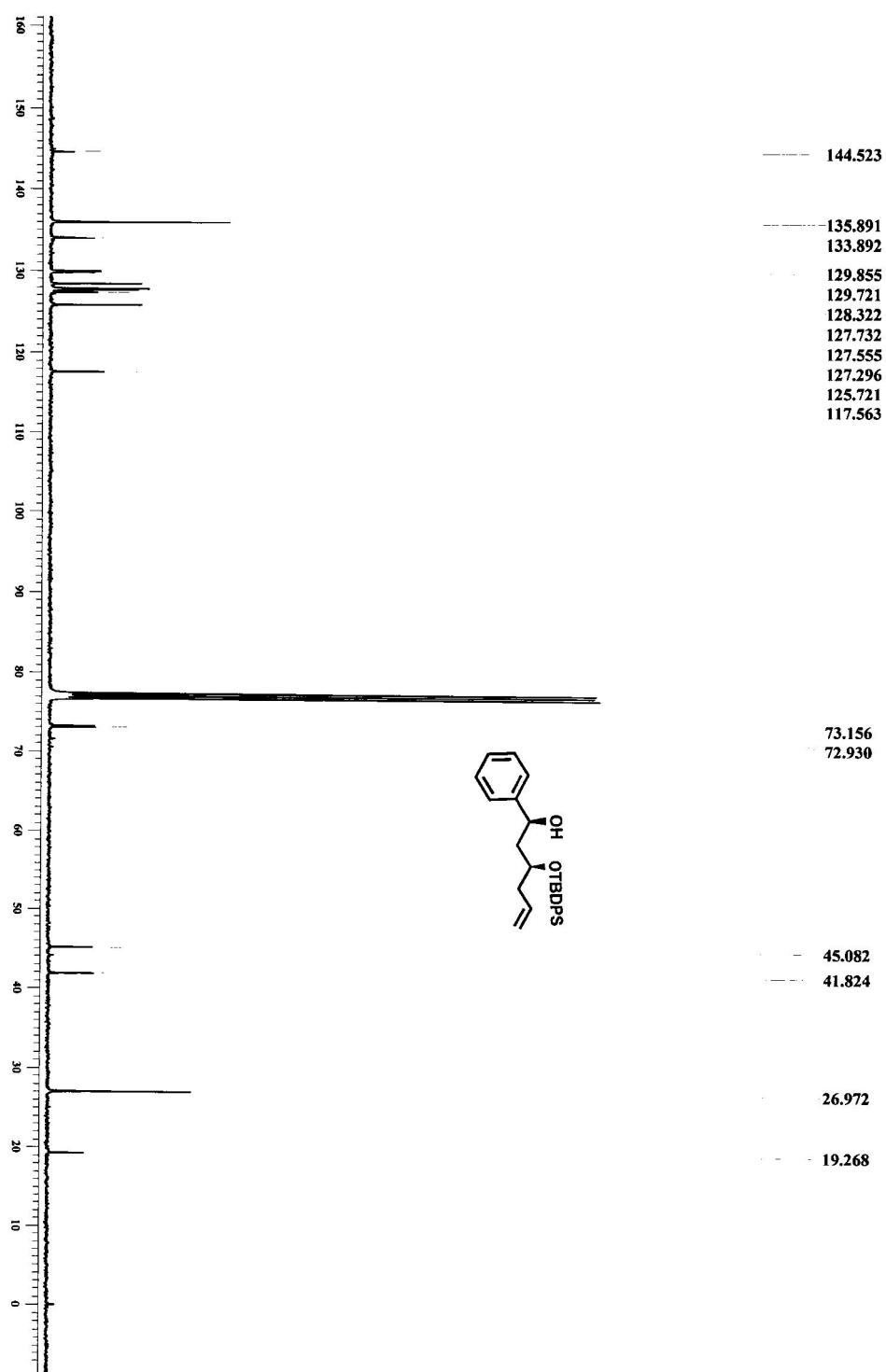
¹H NMR Spectrum of 23 (300 MHz, CDCl₃)



¹³C NMR Spectrum of 23 (100 MHz, CDCl₃)



¹H NMR Spectrum of 24 (500 MHz, CDCl₃)



¹³C NMR Spectrum of 24 (100 MHz, CDCl₃)

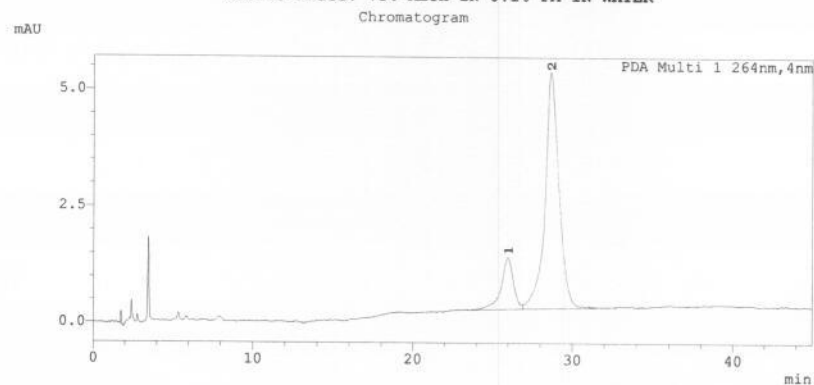


LC-MS DATA REPORT

1107-DNPC

Sample Code : DKM-B-DDQ-430
 Data File : 291118.5.lcd
 Method : CD HPLC-SCS.lcm
 Injection Volume : 5
 Date Acquired : 11/29/2018 2:04:01 PM
 Report File : LC-MS Data Report.lsr

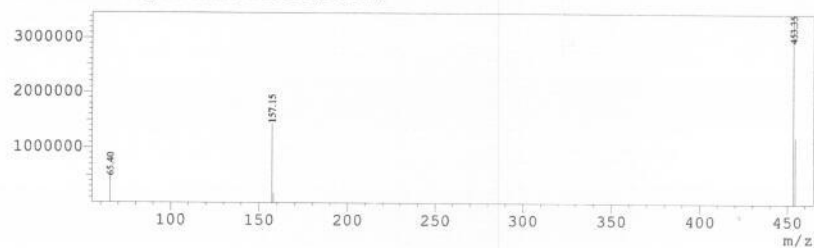
Chromatographic Conditions :
 Colum: X-SELECT (250 X 4.6mm, 5.0u)
 Mobile Phase: 75% MEOH IN 0.1% FA IN WATER



Peak Table

Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	25.973	23.595	26.891	59944	16.192
2	28.643	26.891	31.488	310276	83.808
Total				370220	100.000

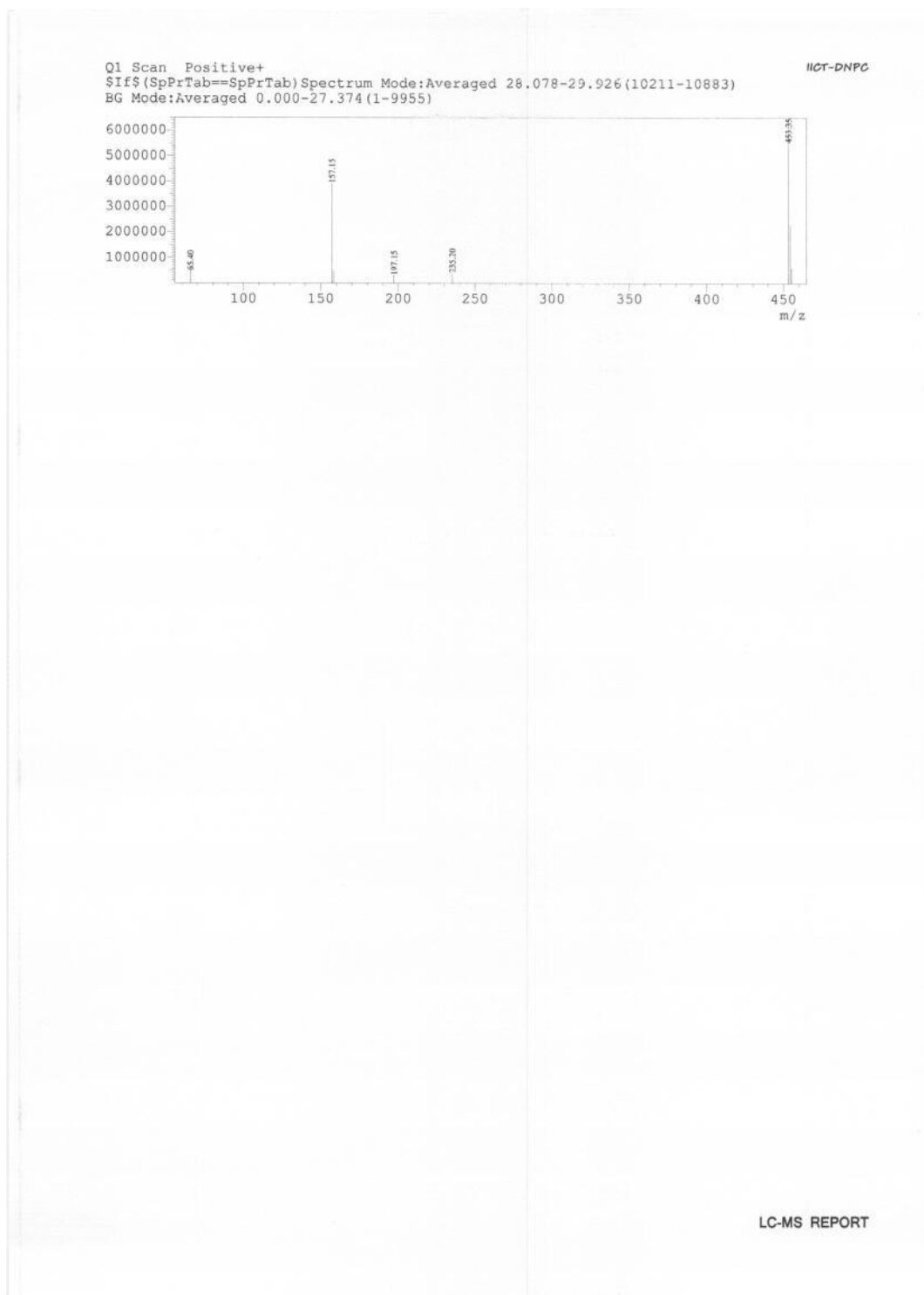
Q1 Scan Positive+
 SIF\$(SpPrTab==SpPrTab)Spectrum Mode:Averaged 25.834-26.362(9395-9587)
 BG Mode:Averaged 0.000-24.624(1-8955)



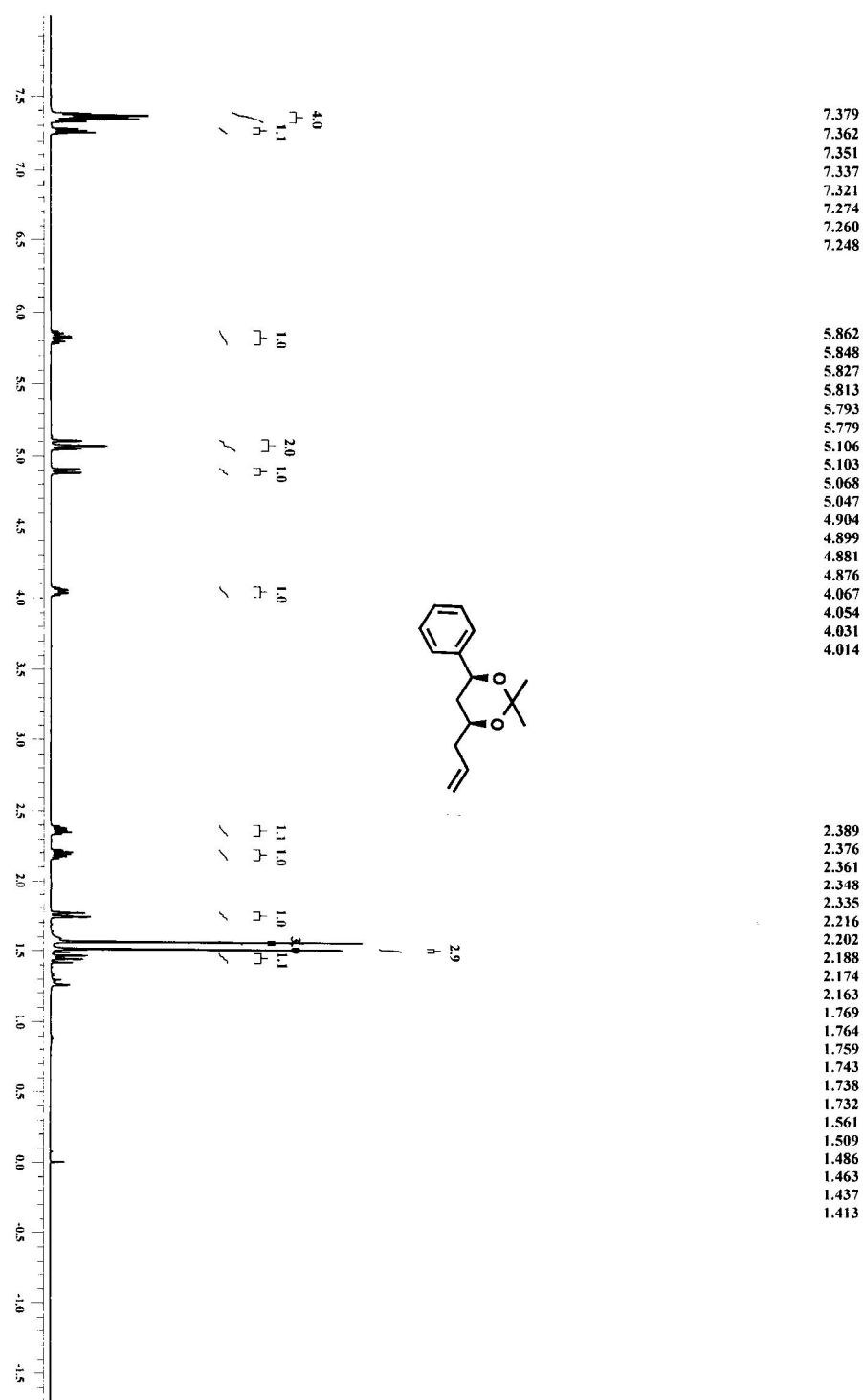
84:16

REPORT

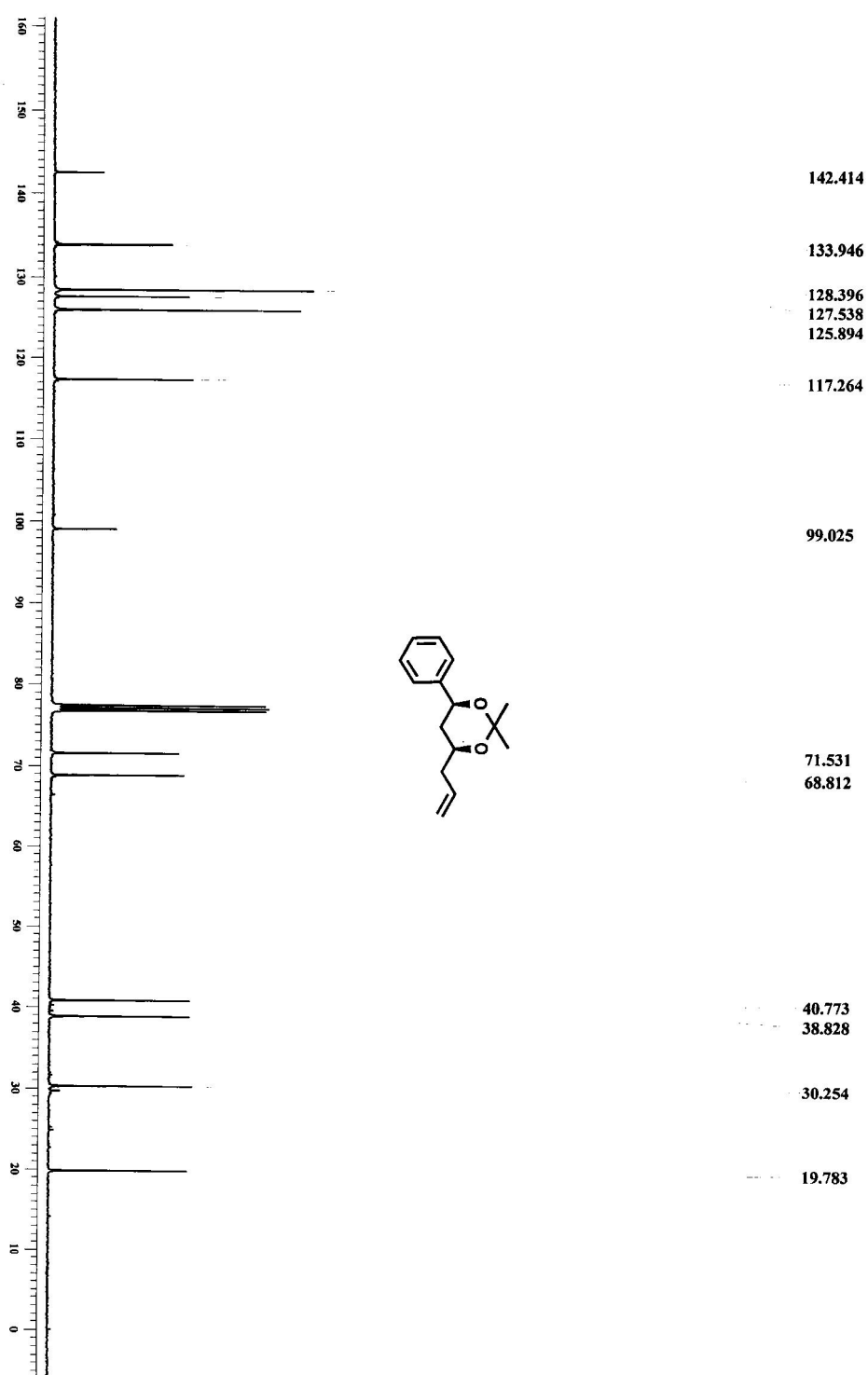
LC-LCMS Chromatogram of 24



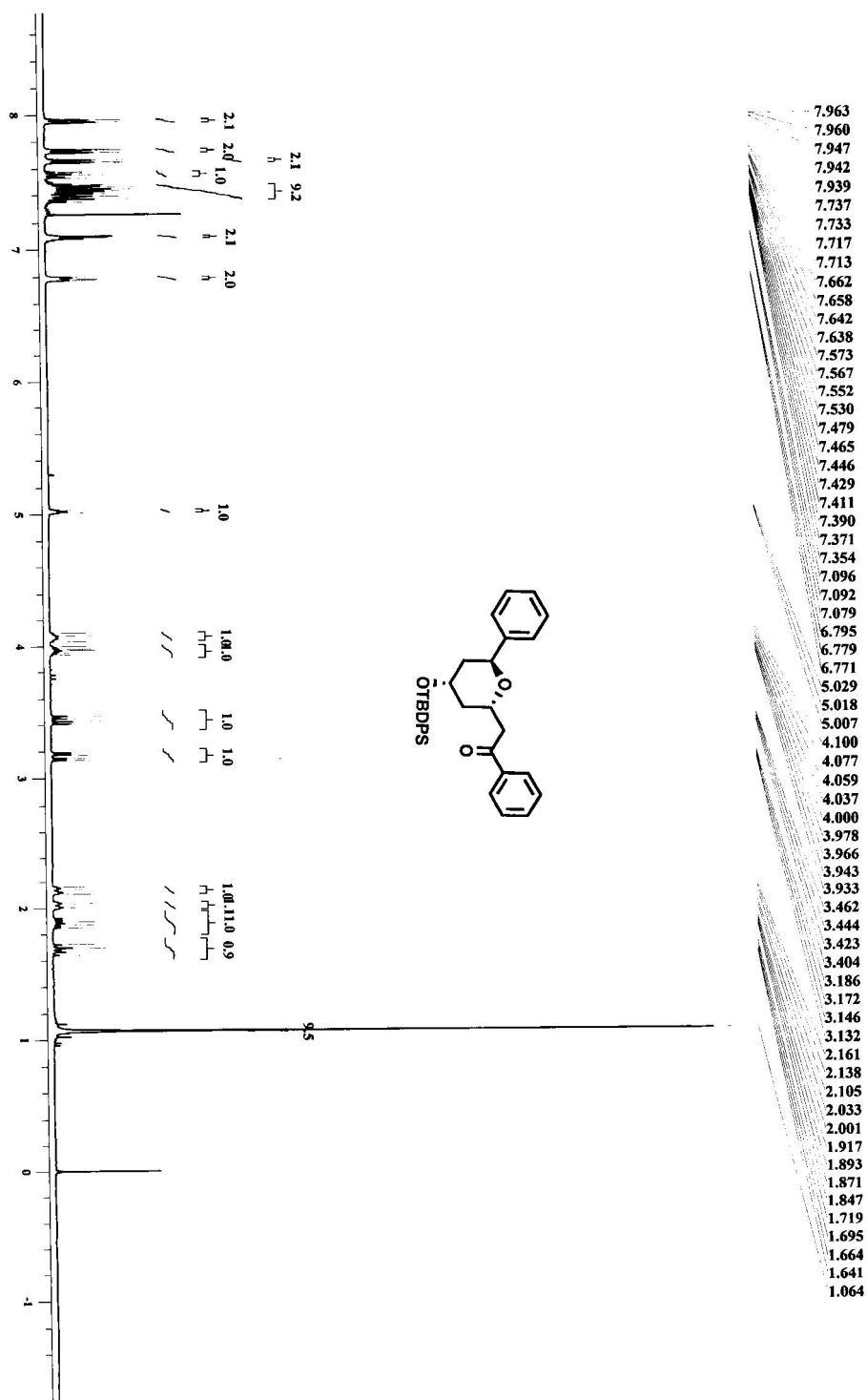
LC-LCMS Chromatogram of 24



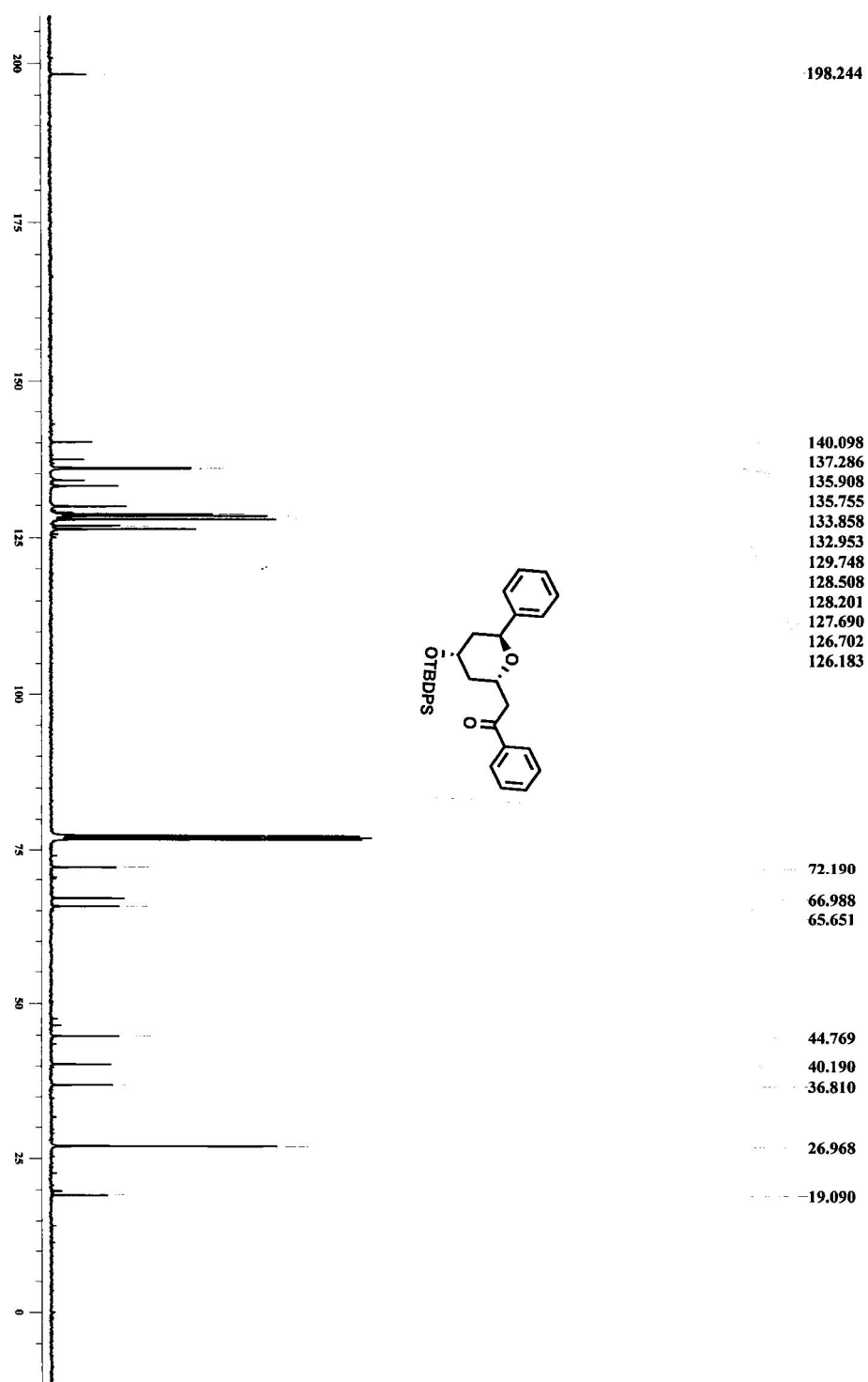
^1H NMR Spectrum of 25 (500 MHz, CDCl_3)



^{13}C NMR Spectrum of 25 (100 MHz, CDCl_3)

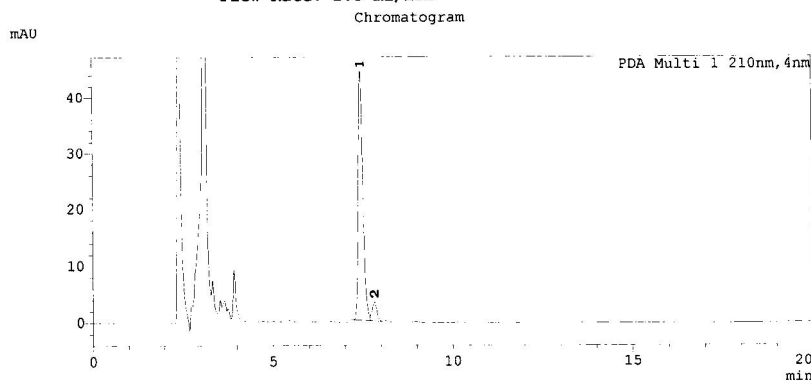


¹H NMR Spectrum of 17 (500 MHz, CDCl₃)



^{13}C NMR Spectrum of 17 (100 MHz, CDCl_3)

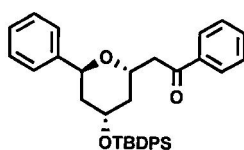
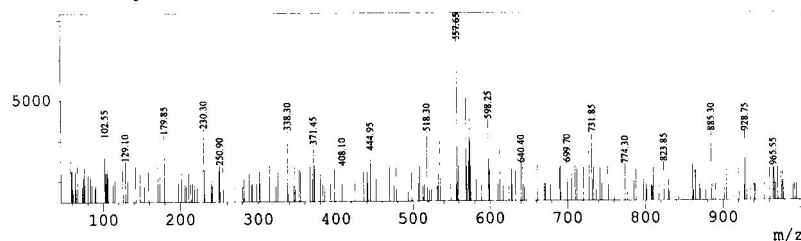
Sample Code : DKM-B-ACT-534
 Data File : 160318.4.lcd
 Method : SCS-RK-173.lcm
 Injection Volume : 5
 Date Acquired : 3/16/2018 1:11:59 PM
 Report File : LC-MS Data Report.lsr
 Chromatographic Conditions :
 : chromatographic
 Conditions : LUNA-C8 (250 X 4.6mm, 5.0u)
 Mobile Phase: 95%ACN IN 10% FA WITH WATER
 Flow Rate: 1.0 mL/min



Peak Table

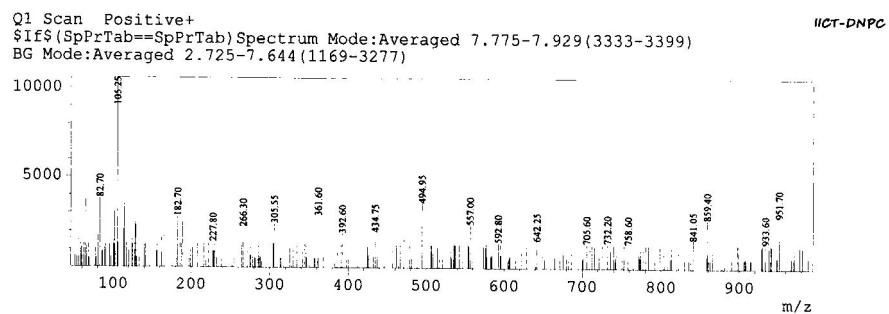
Peak#	Ret. Time	Peak Start	Peak End	Area	Area%
1	7.451	7.232	7.691	394430	93.109
2	7.812	7.691	8.021	29194	6.891
Total				423624	100.000

Q1 Scan Positive+
 \$If\$ (SpPrTab==SpPrTab) Spectrum Mode:Averaged 7.327-7.448 (3141-3193)
 BG Mode:Averaged 4.256-6.986(1825-2995)



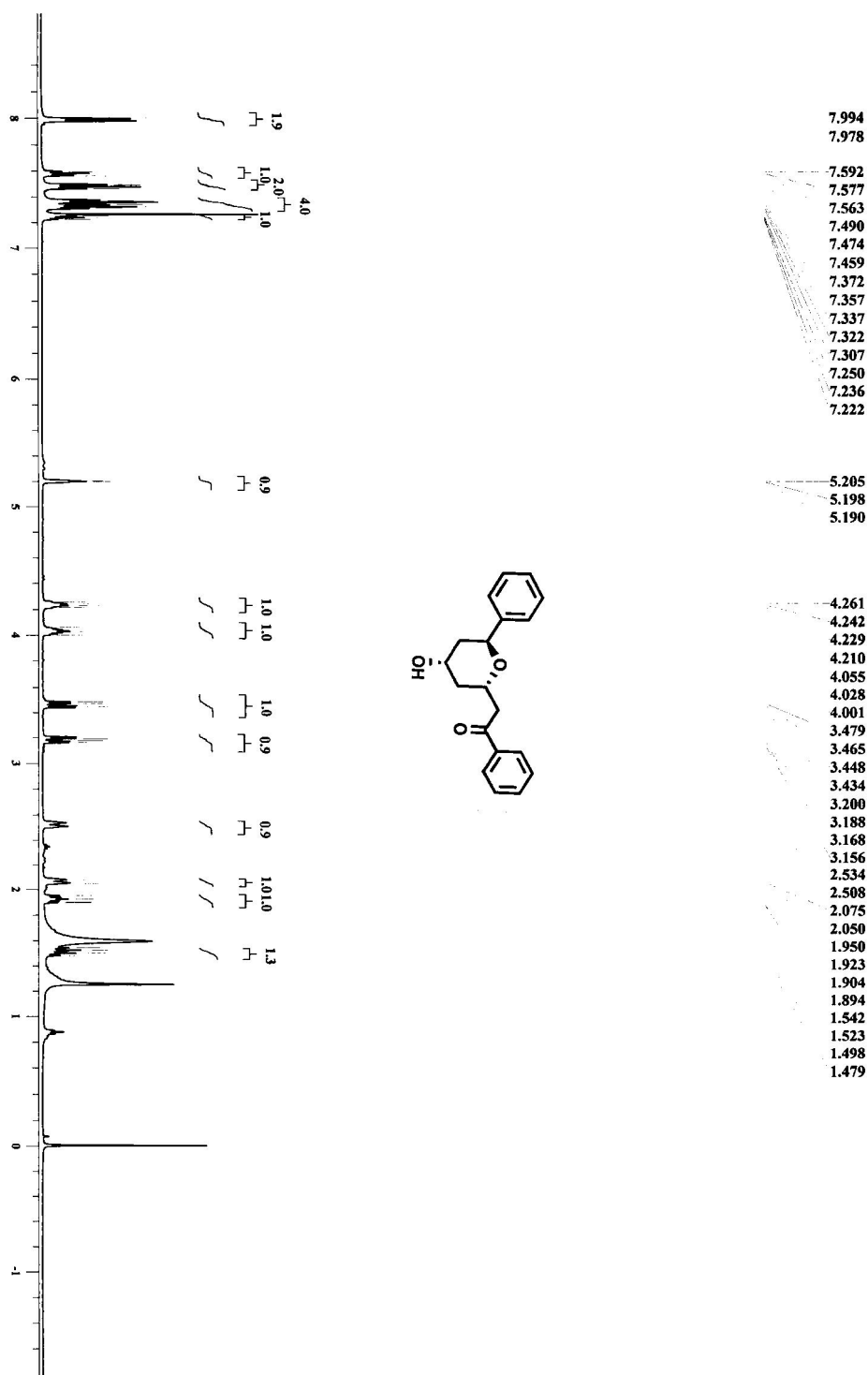
REPORT

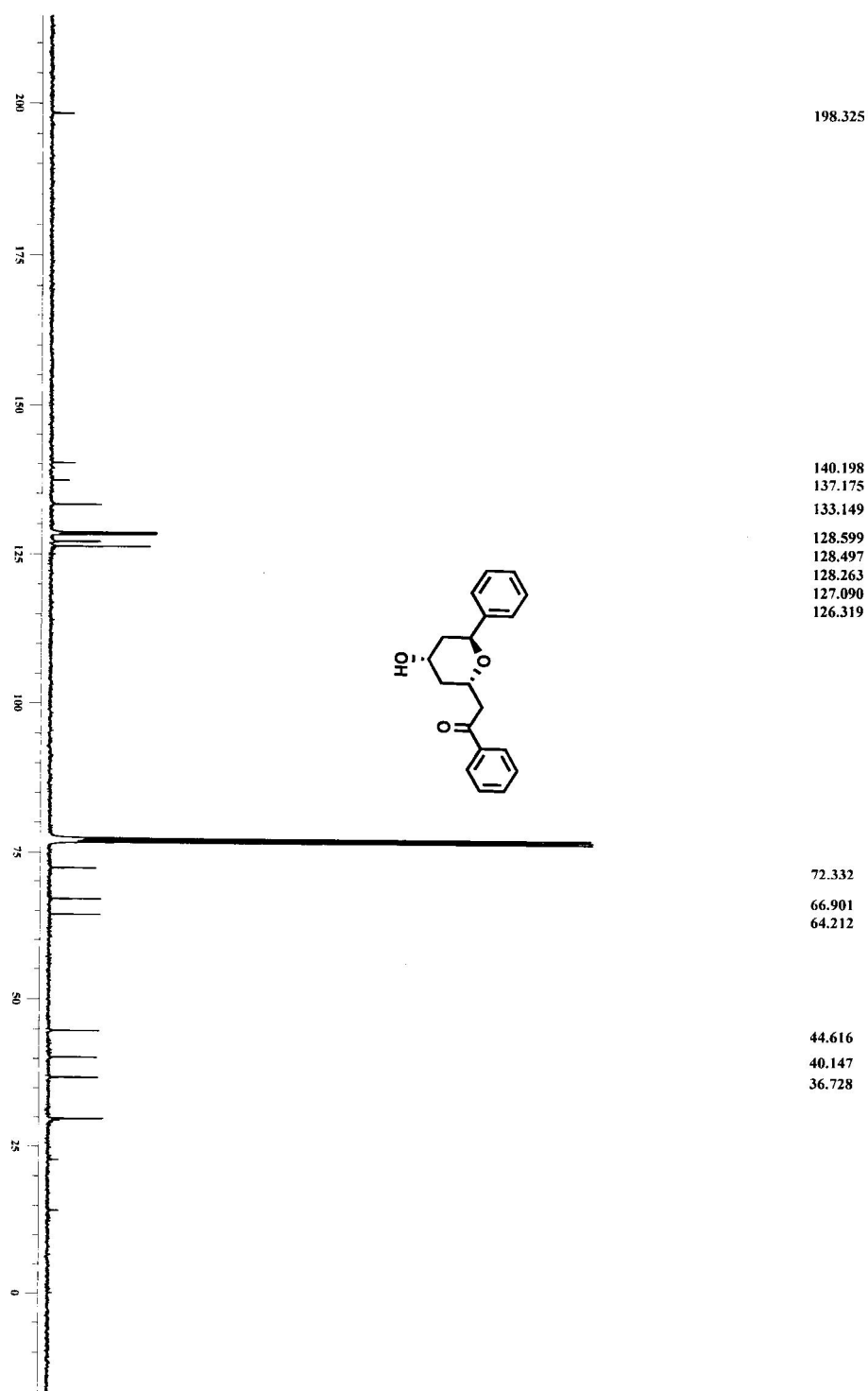
LC-LCMS Chromatogram of 17



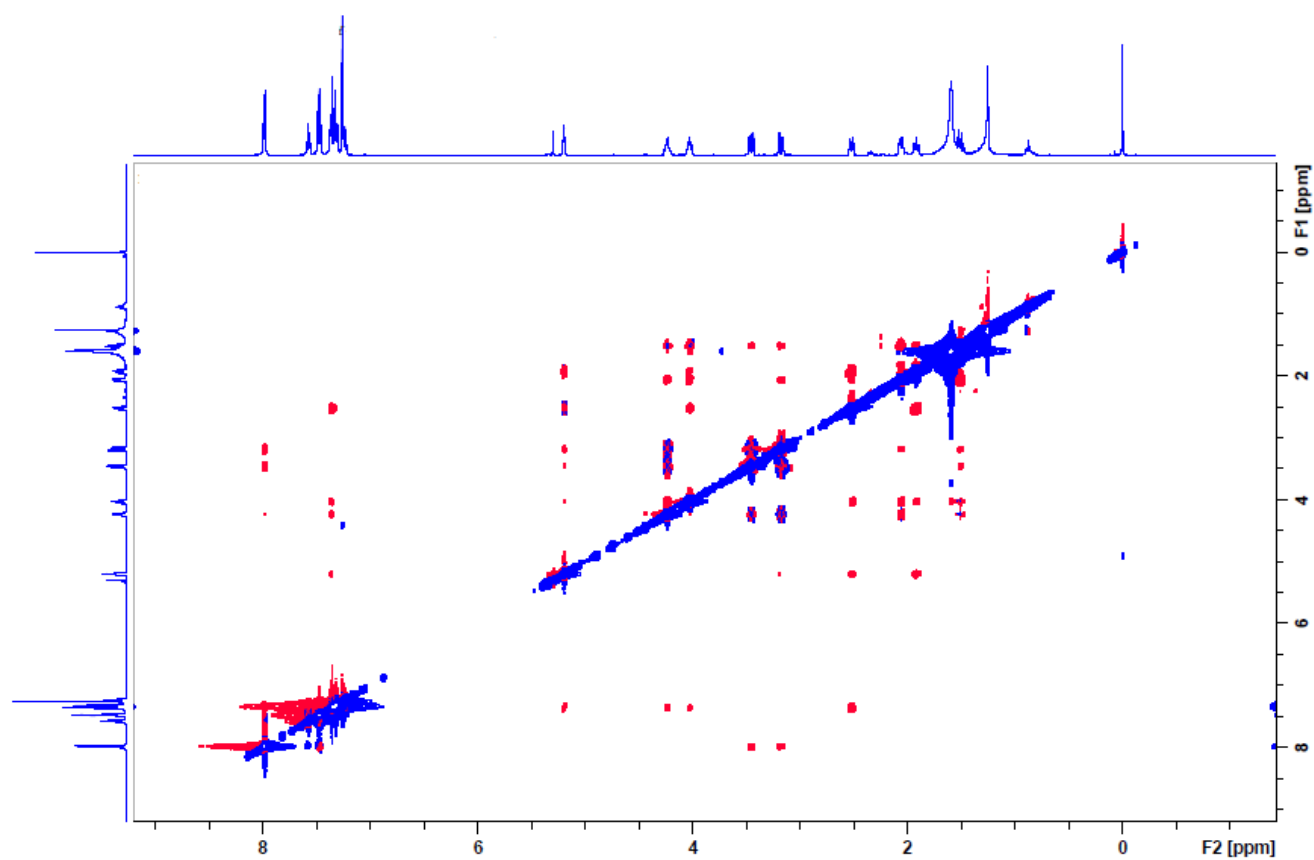
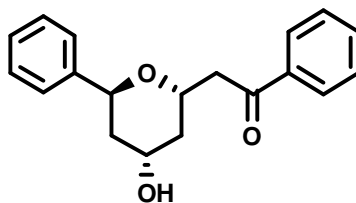
LC-MS REPORT

LC-LCMS Chromatogram of 17

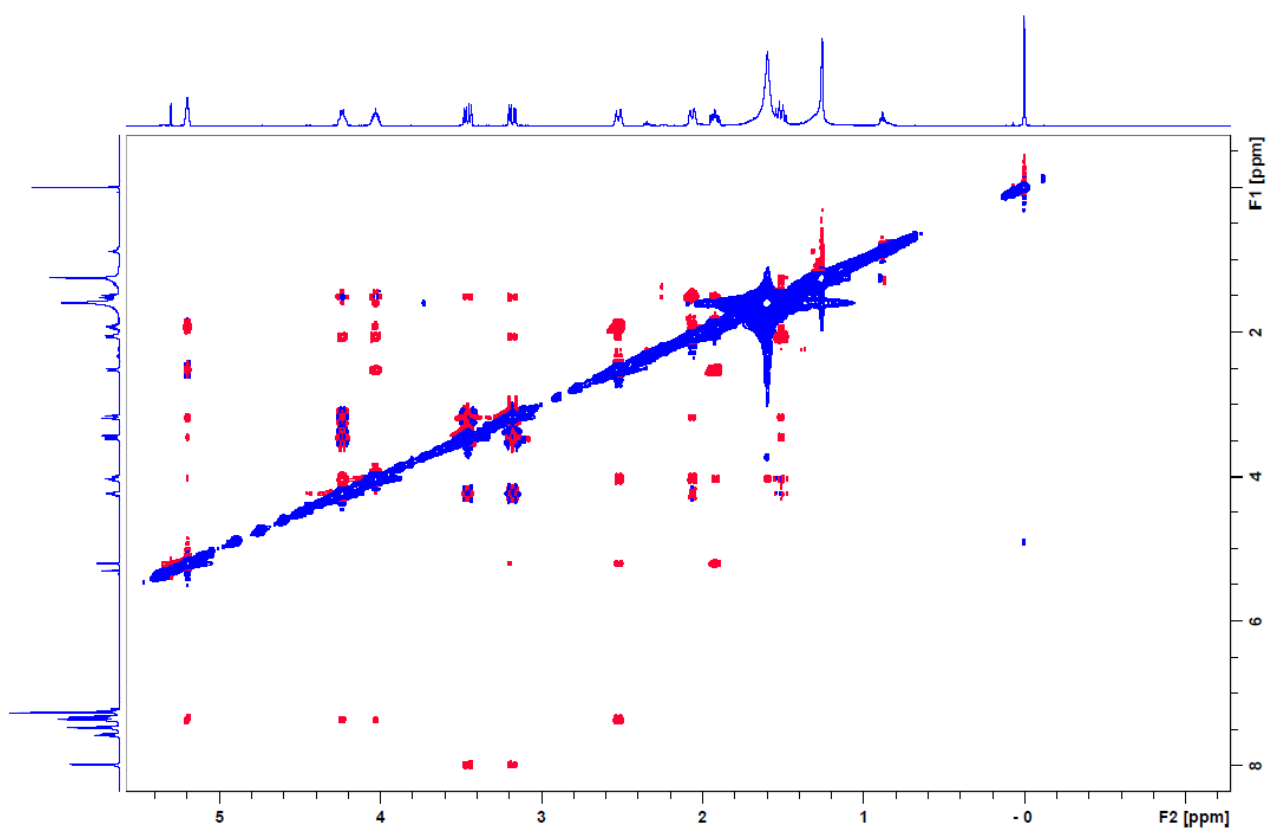
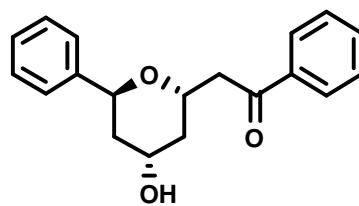
 ^1H NMR Spectrum of 2 (500 MHz, CDCl_3)



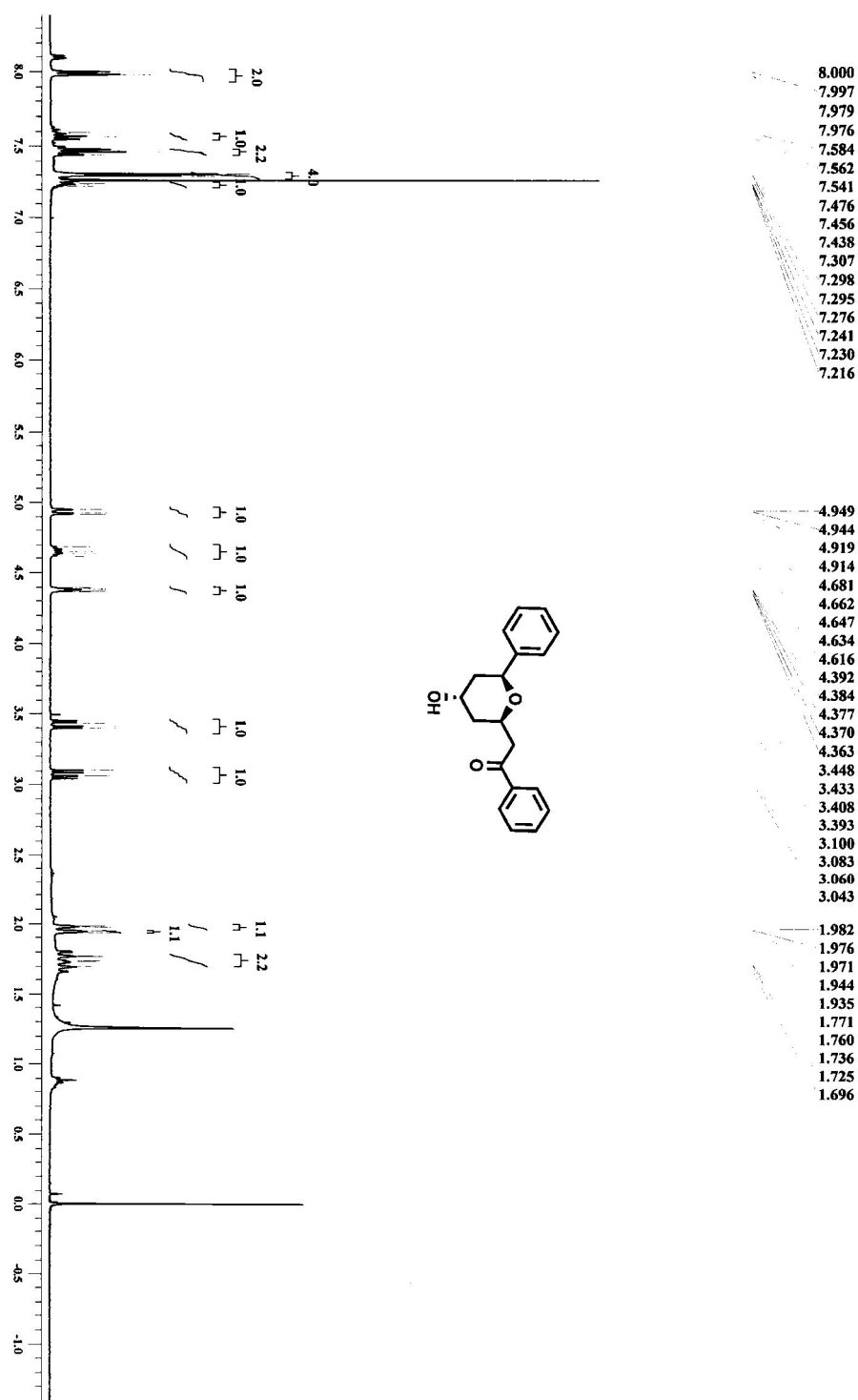
^{13}C NMR Spectrum of 2 (100 MHz, CDCl_3)



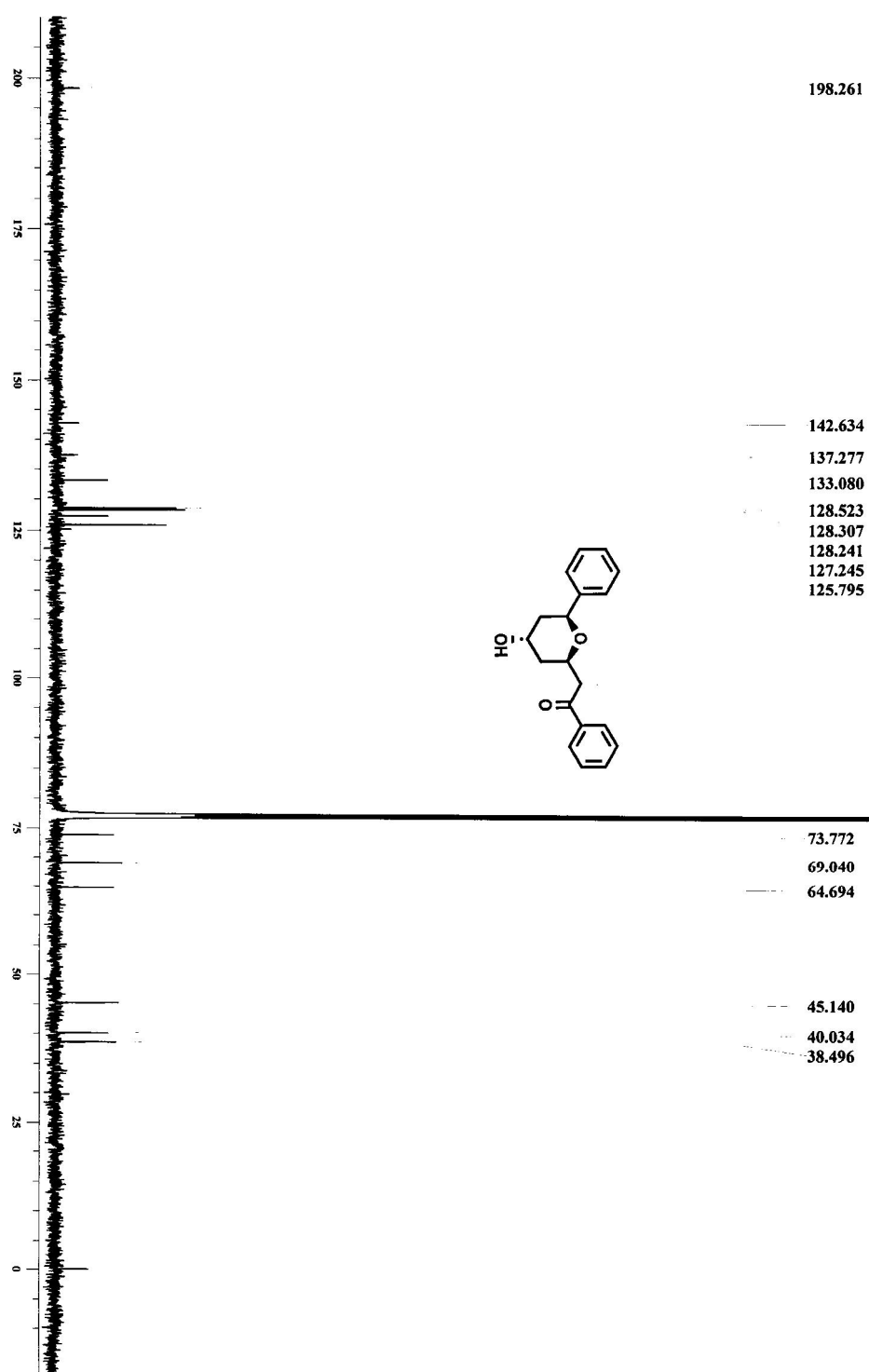
NOESY of Compound 2 (500 MHz, CDCl₃)



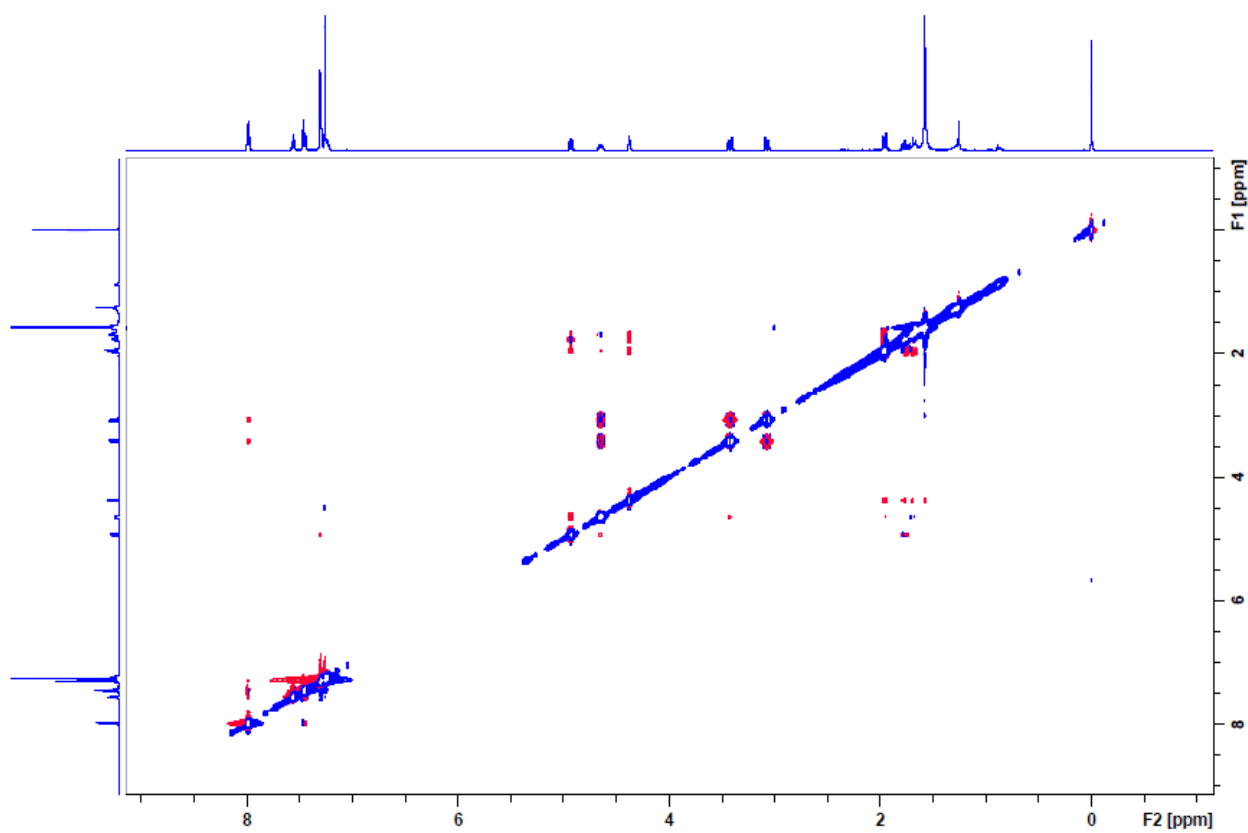
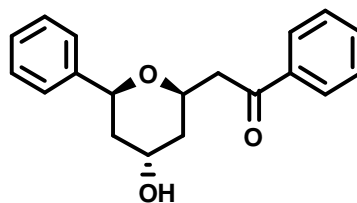
Expanded NOESY of Compound 2 (500 MHz, CDCl₃)



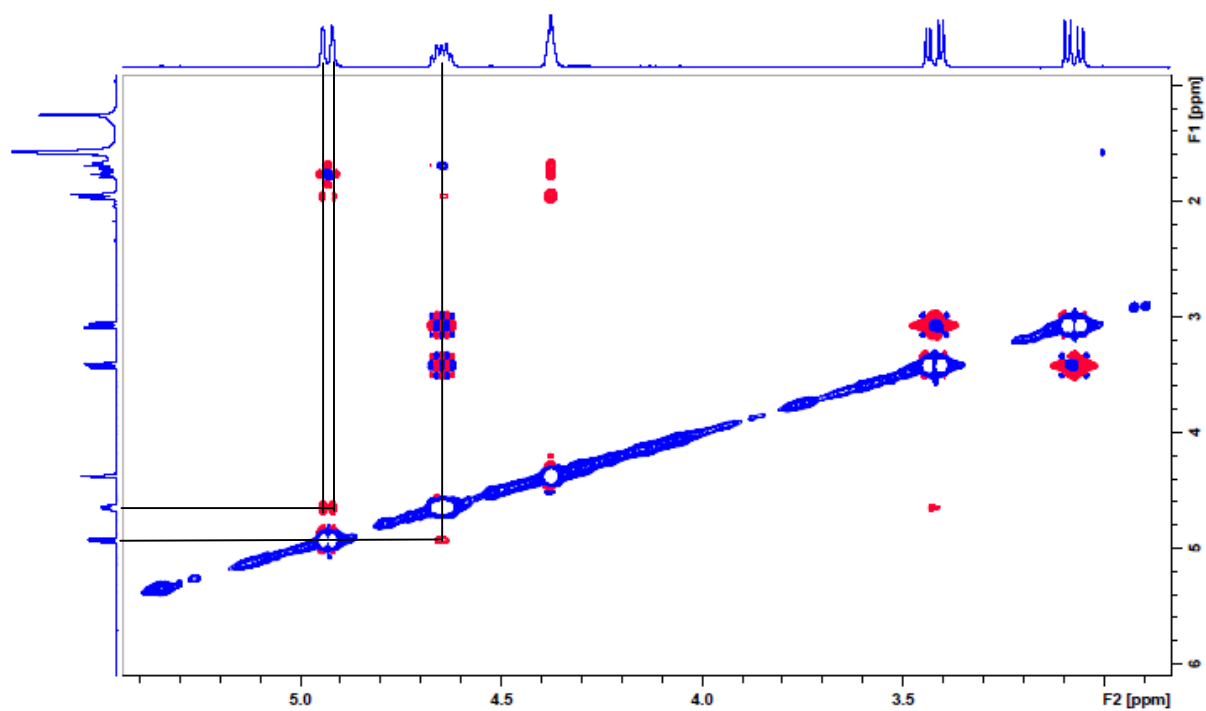
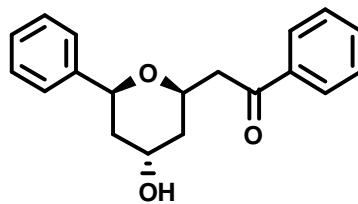
¹H NMR Spectrum of 1 (400 MHz, CDCl₃)



¹³C NMR Spectrum of 1 (100 MHz, CDCl₃)



NOESY of Compound 1 (400 MHz, CDCl₃)



Expanded NOESY of Compound 1 (400 MHz, CDCl₃)