

Supplemental material for:

**Highly chemoselective synthesis of dimeric 2-oxindoles with a C-3/C-5'
via Friedel-Crafts alkylations of 2-oxindoles with 3-hydroxy-2-
oxindoles†**

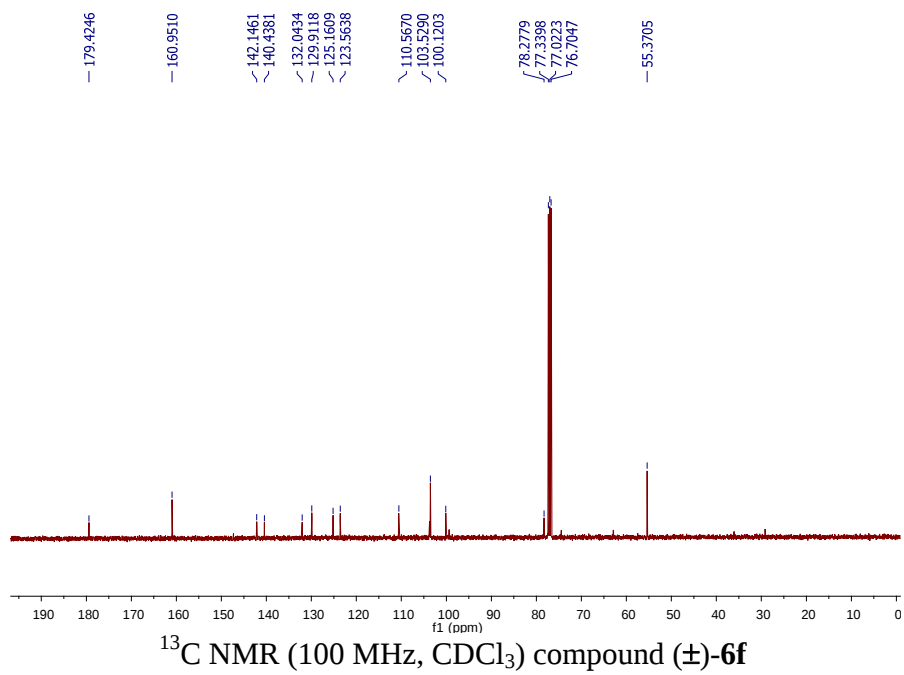
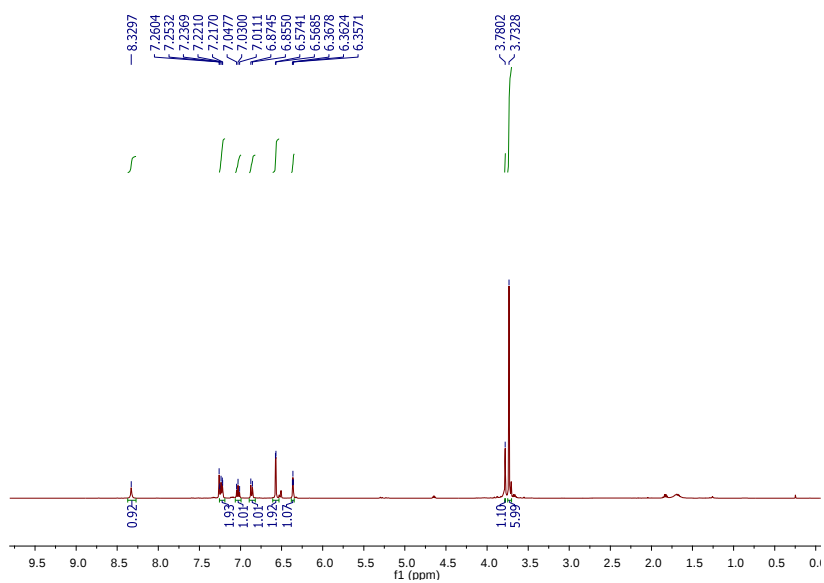
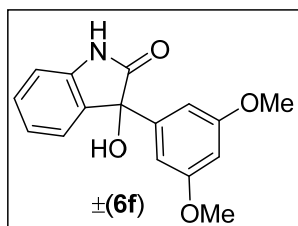
K. Naresh Babu,[§] Lakshmana K. Kinthada,[§] Santanu Ghosh, and Alakesh Bisai*

*Department of Chemistry, Indian Institute of Science Education and
Research Bhopal, Bhopal - 462 066, MP, India
E-Mail: alakesh@iiserb.ac.in*

Spectral Graphics

Spectroscopic analysis of all new compounds

S2 - S79



Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JULY-2015\29-JULY\Dr.A.Bisai-AB-LK-03-046_1-A,5_01_3223.d
Method HRLCMS-20 Sept.m
Sample Name Dr.A.Bisai-AB-LK-03-046
Comment

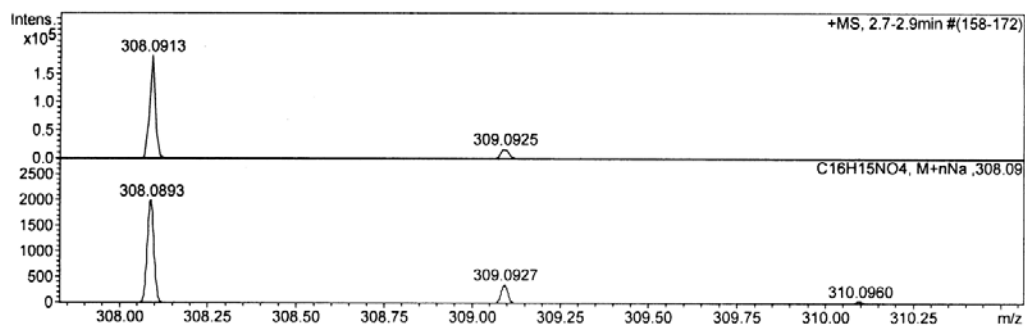
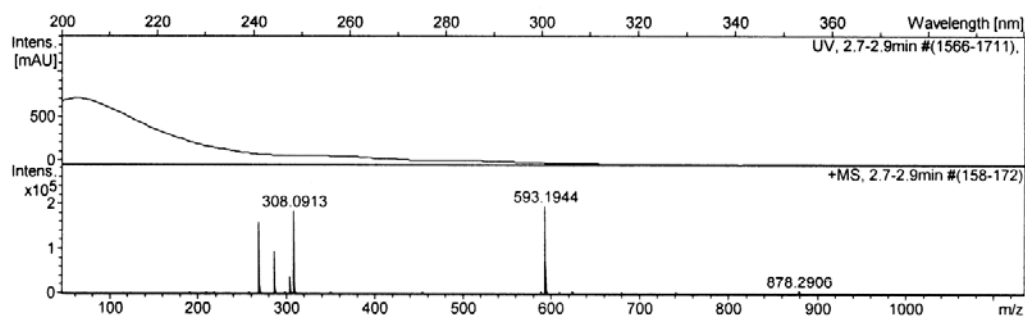
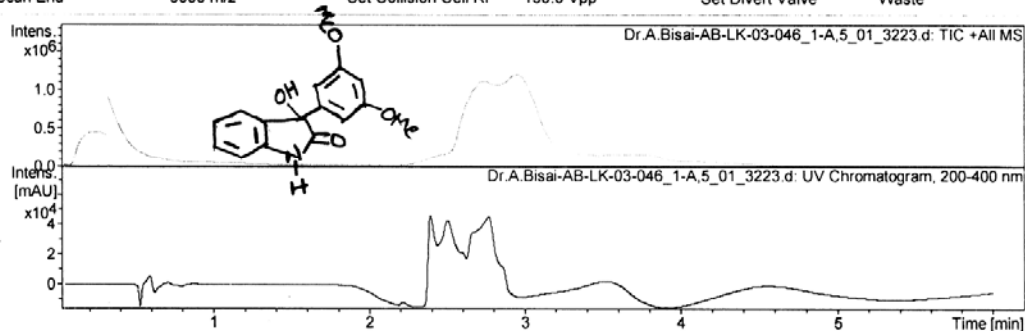
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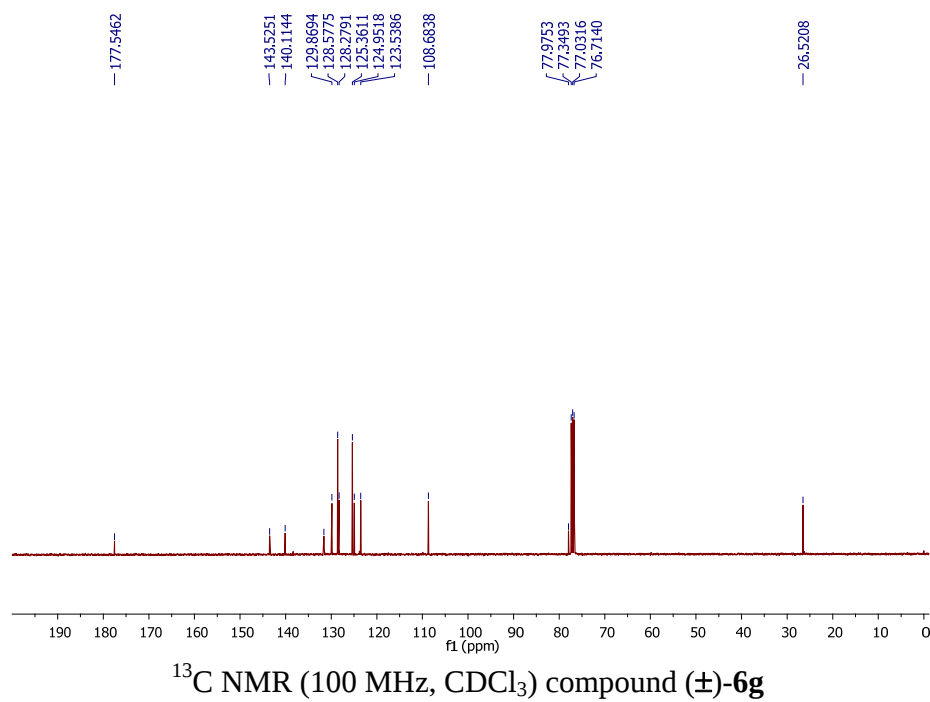
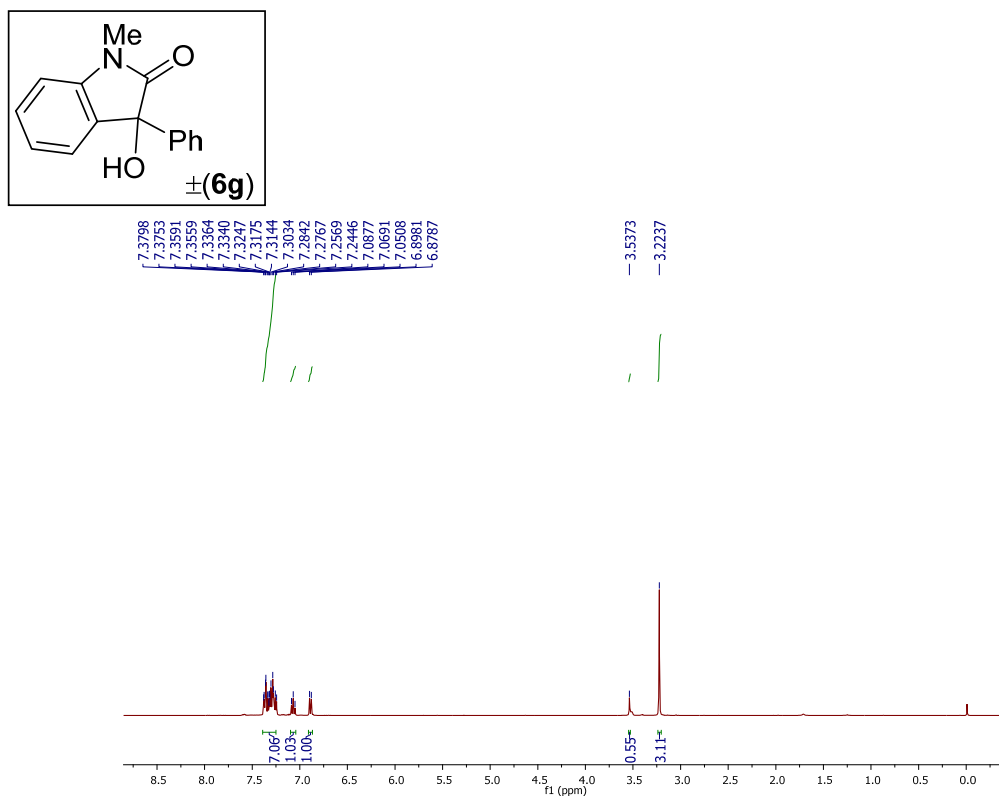
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Scanned copy of mass spectrum of (±)-6f



Display Report

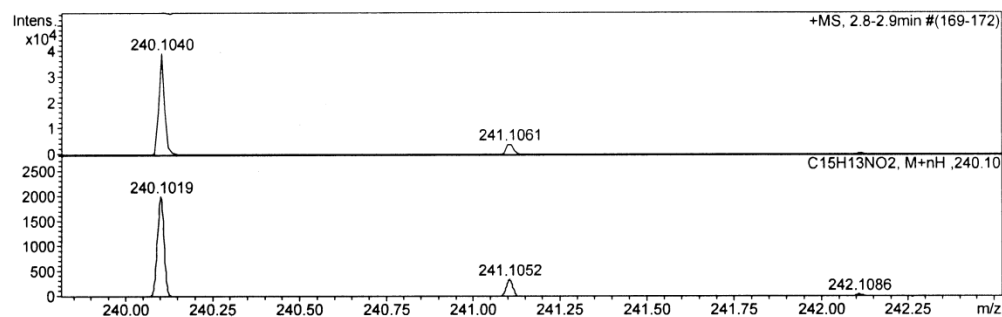
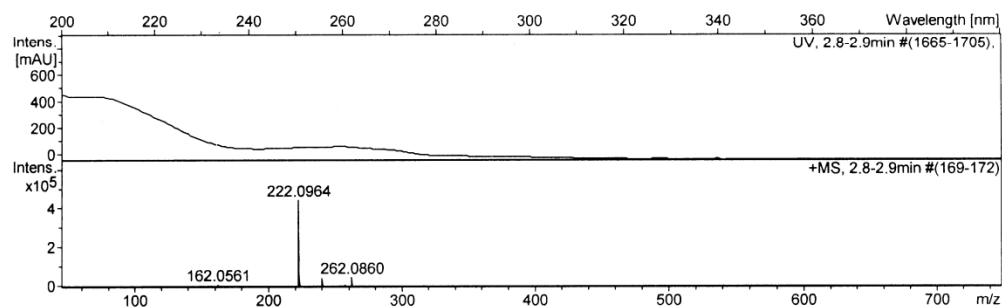
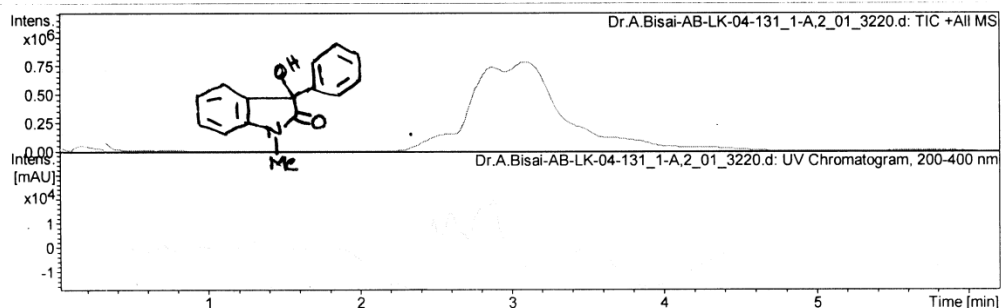
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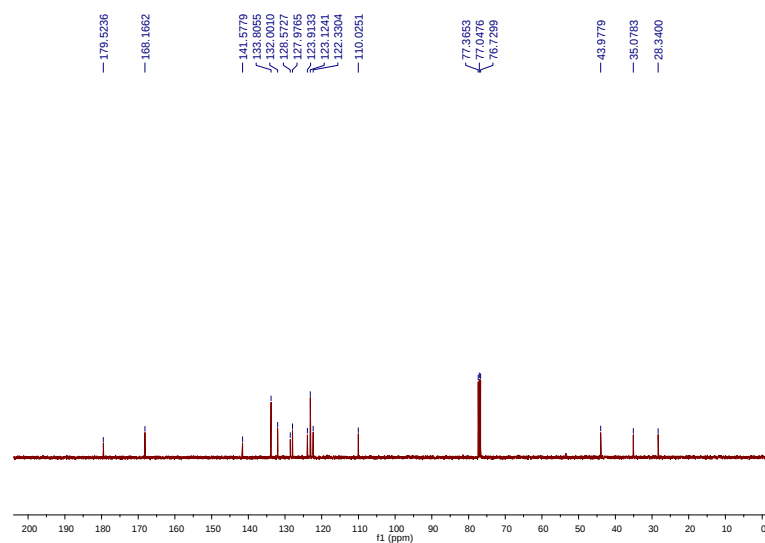
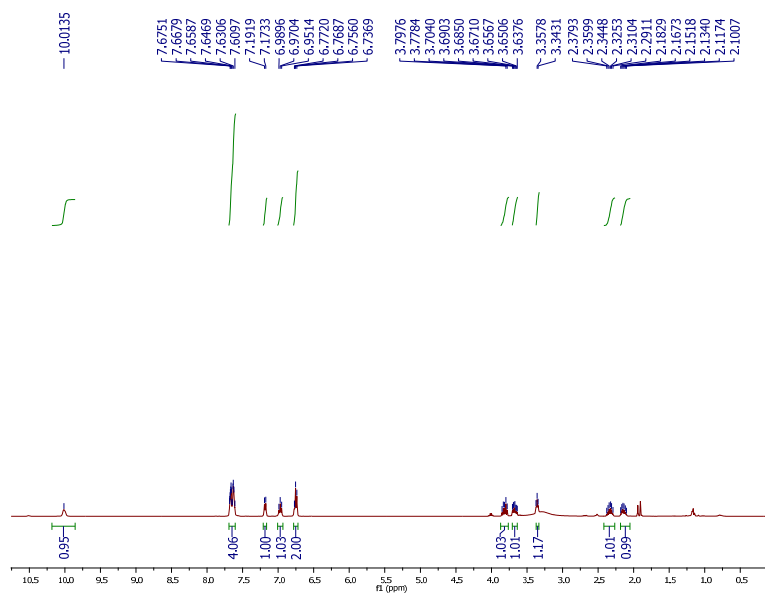
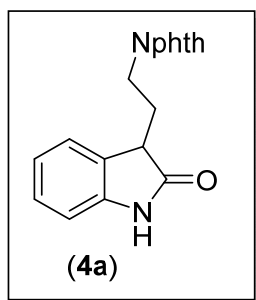
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Scanned copy of mass spectrum of (±)-6g



Display Report

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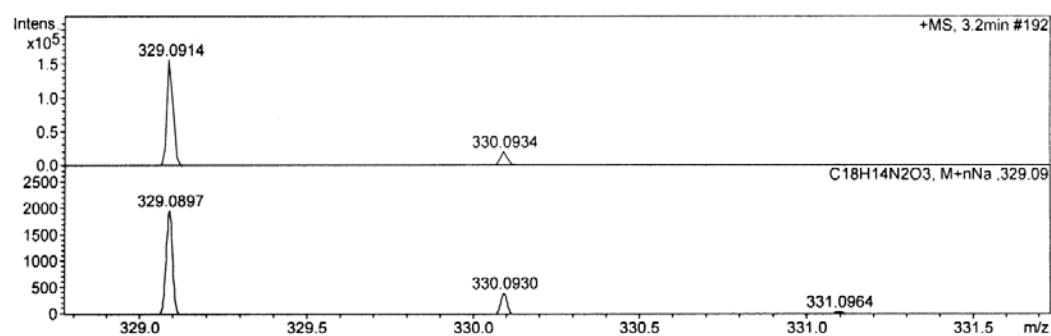
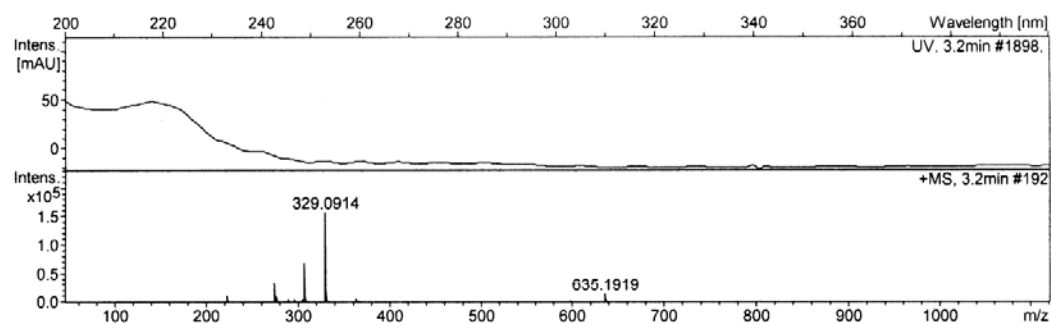
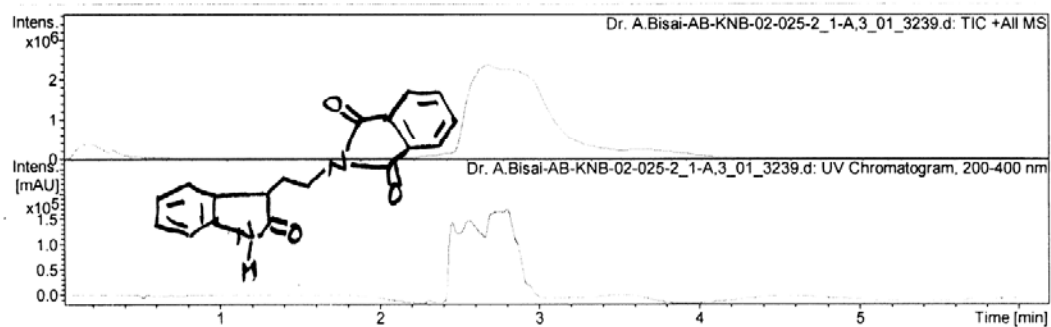
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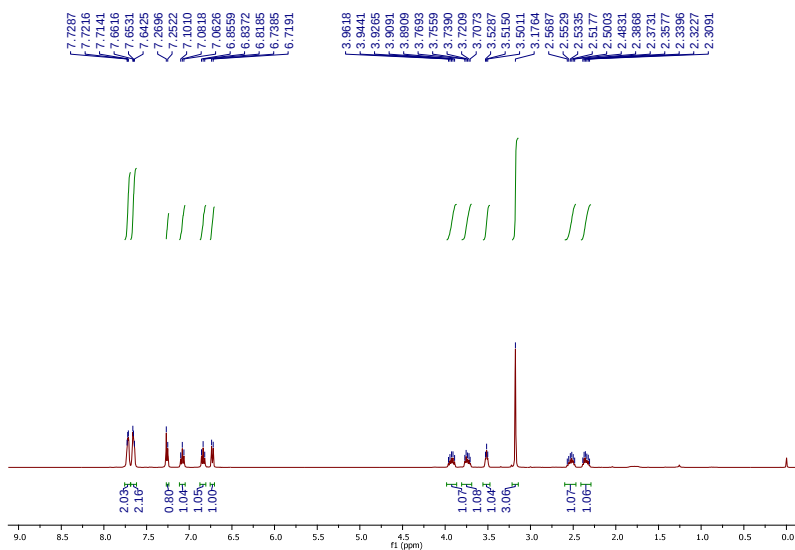
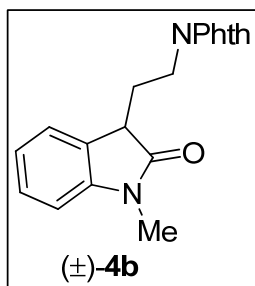
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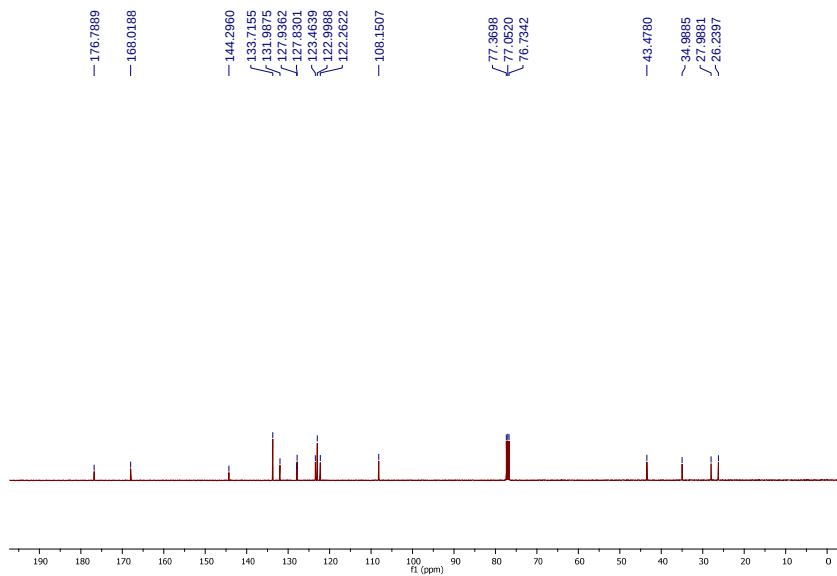
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Scanned copy of mass spectrum of (±)-4a



¹H NMR (400 MHz, CDCl₃) compound (±)-4b



¹³C NMR (100 MHz, CDCl₃) compound (±)-4b

Display Report

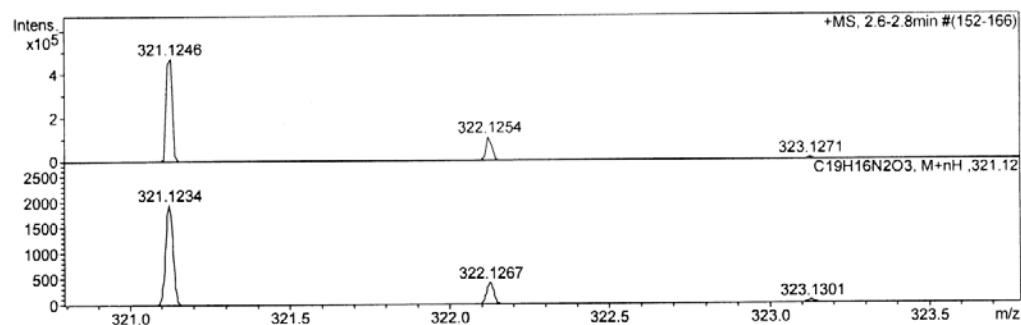
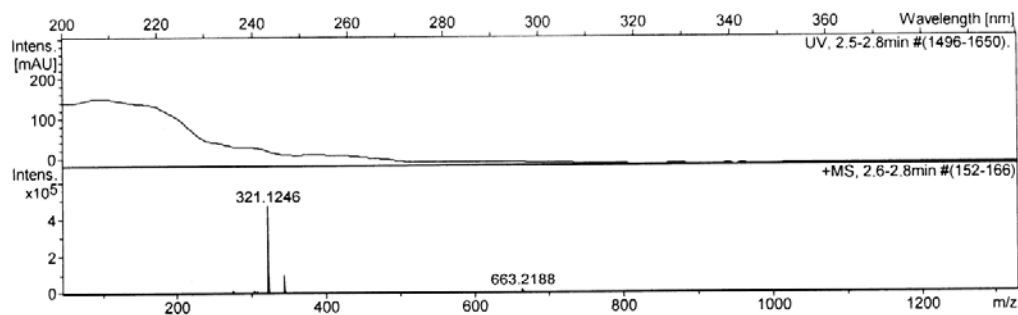
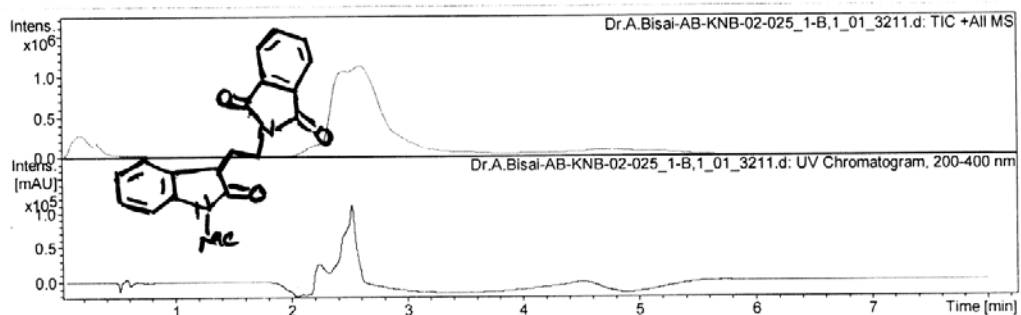
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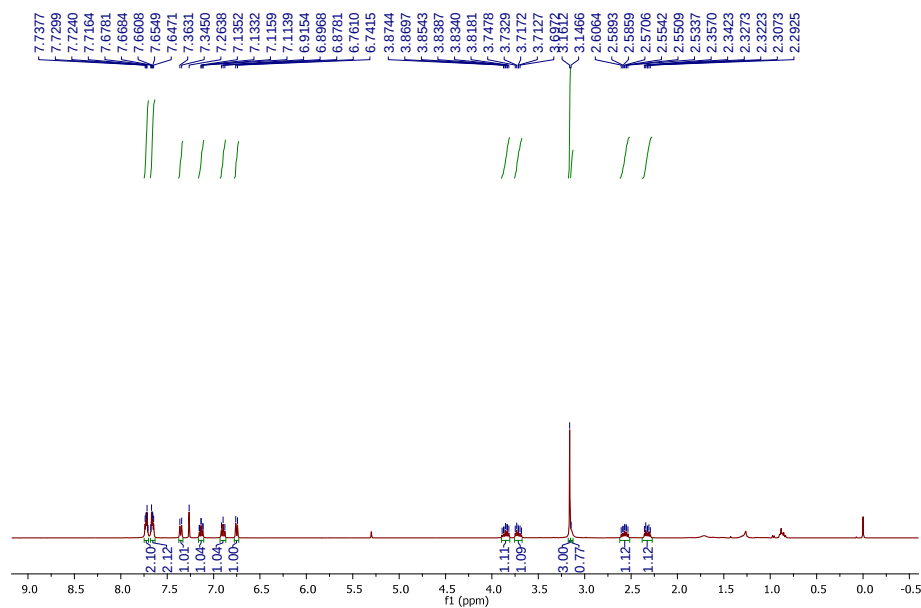
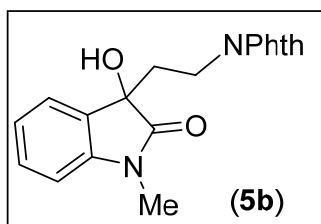
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Instrument micrOTOF-Q II 10330

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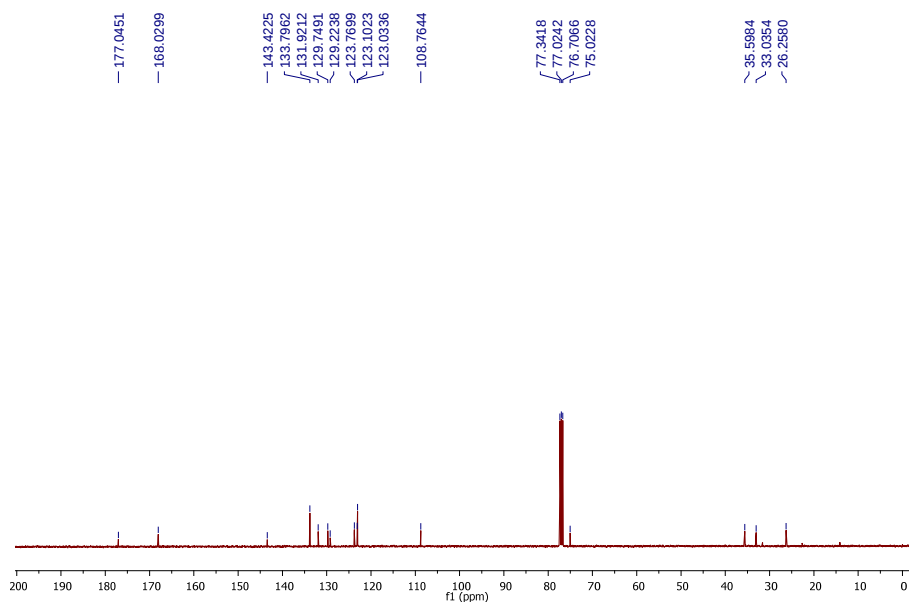
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-4b



^1H NMR (400 MHz, CDCl_3) compound (±)-5b



^{13}C NMR (100 MHz, CDCl_3) compound (±)-5b

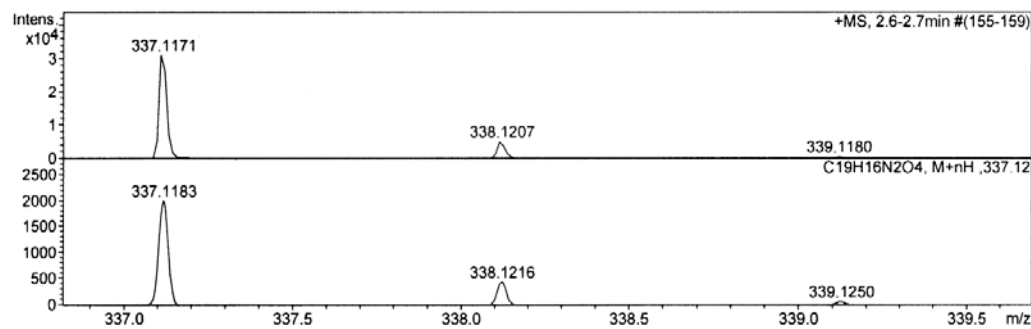
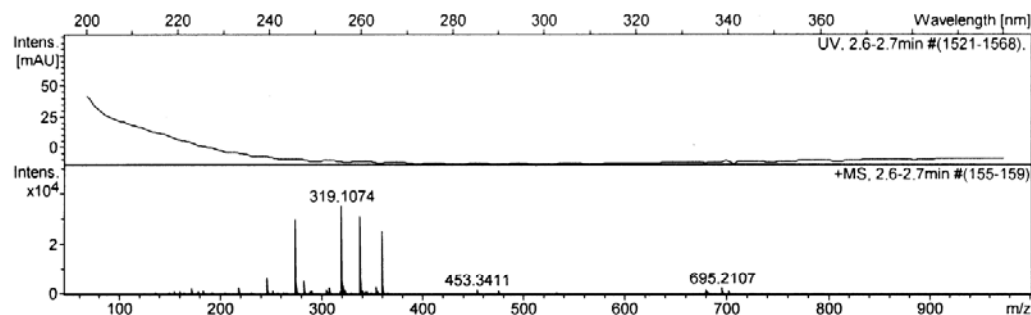
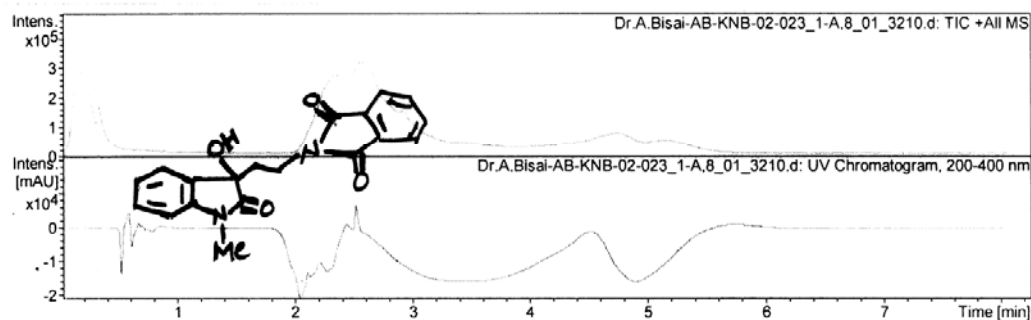
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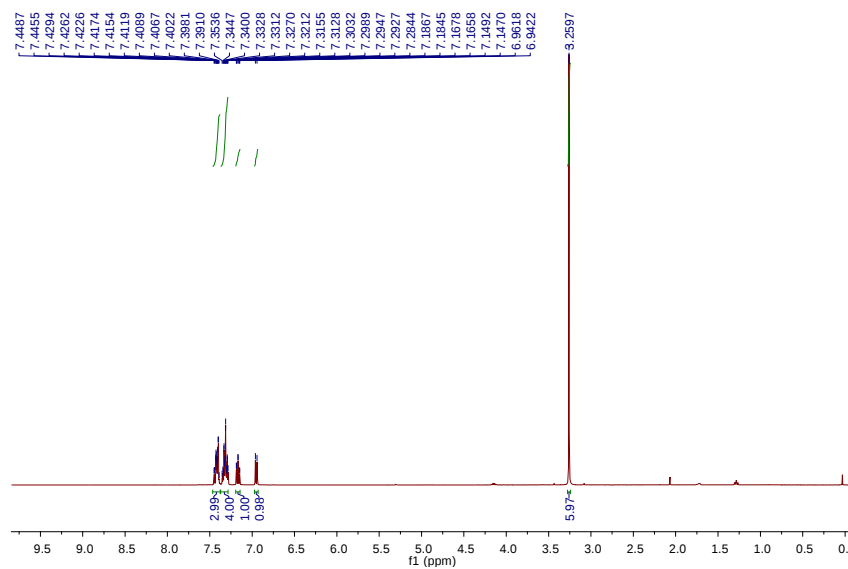
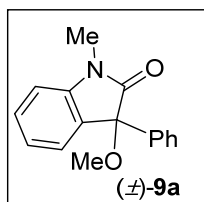
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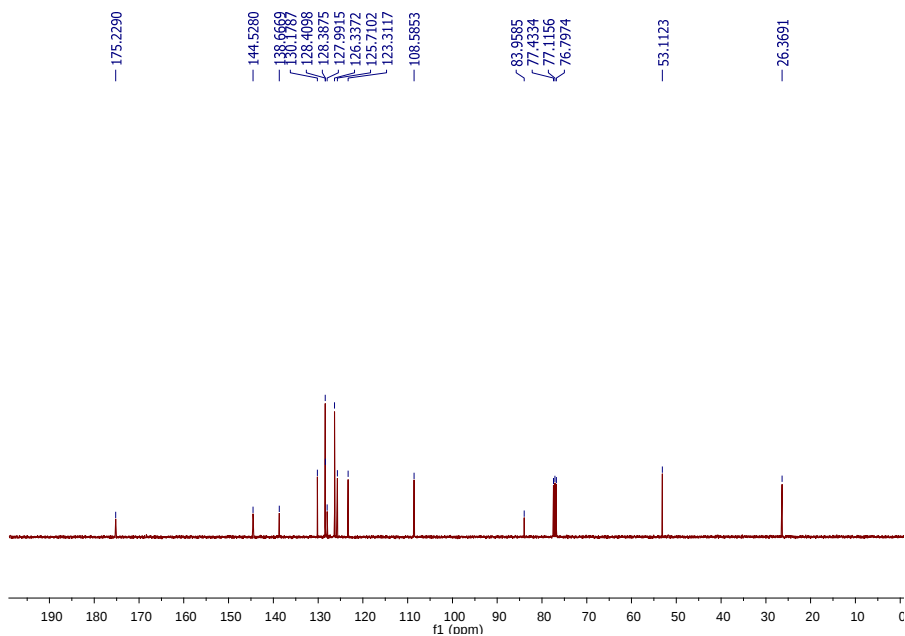
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Scanned copy of mass spectrum of (±)-5b



¹H NMR (400 MHz, CDCl₃) compound (±)-**9a**



¹³C NMR (100 MHz, CDCl₃) compound (±)-**9a**

Display Report

Analysis Info

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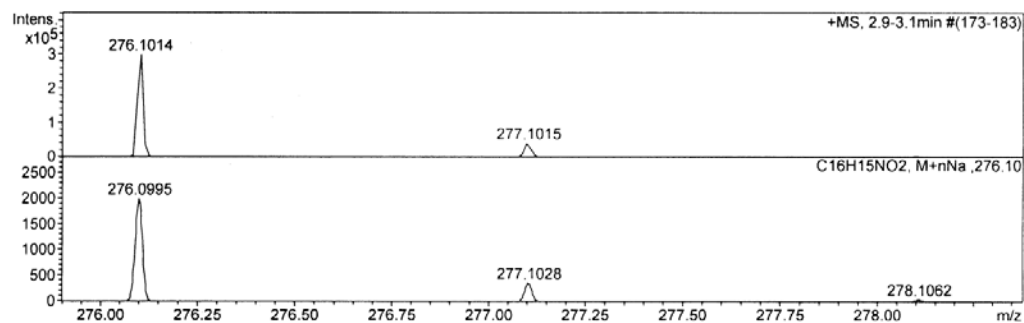
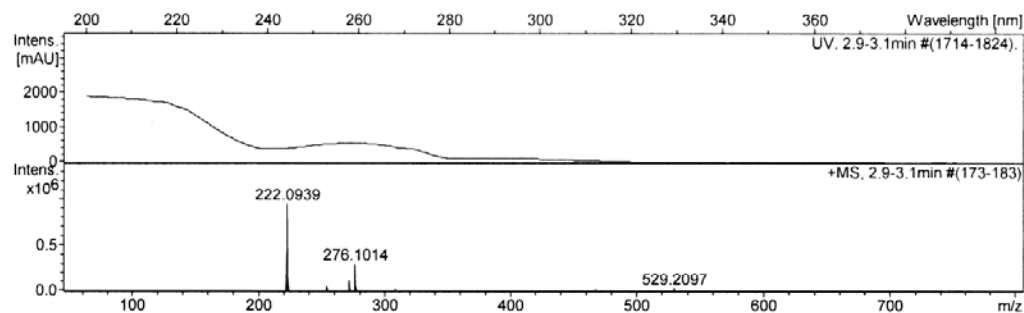
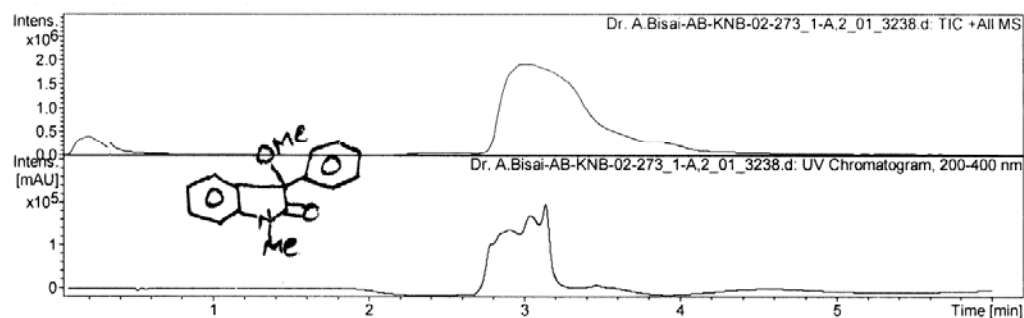
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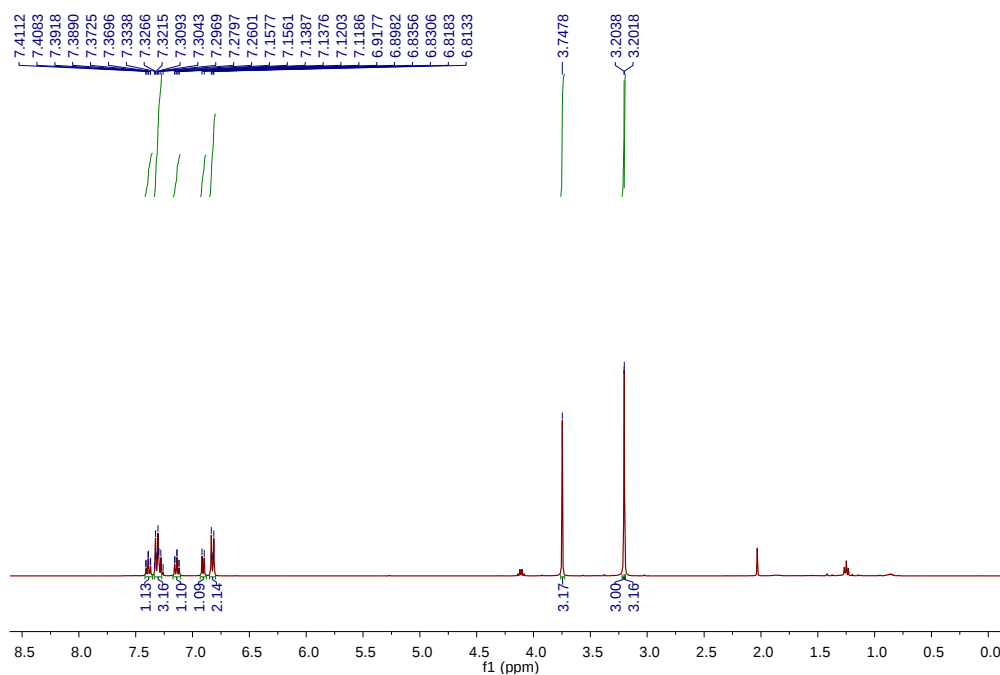
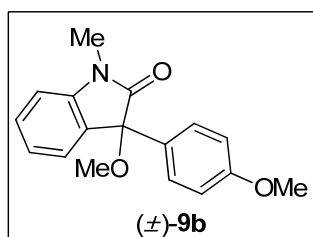
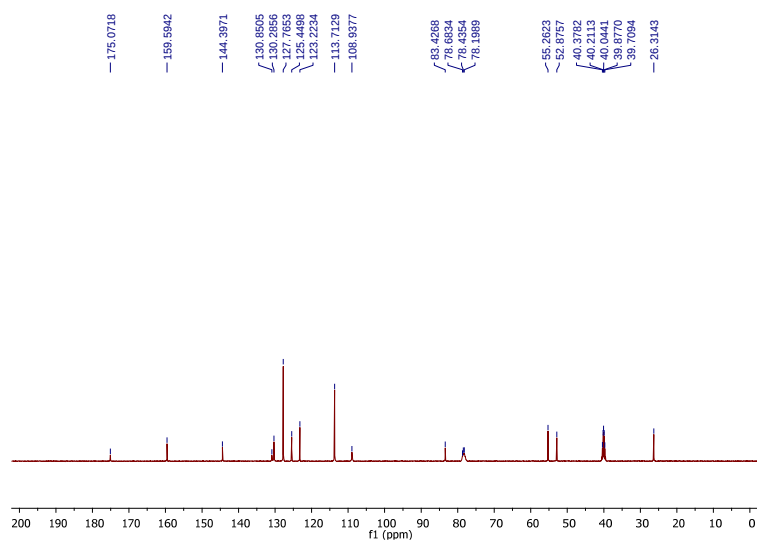
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Scanned copy of mass spectrum of (±)-9a

¹H NMR (400 MHz, CDCl₃) compound (±)-**9b**¹³C NMR (100 MHz, CDCl₃) compound (±)-**9b**

Display Report

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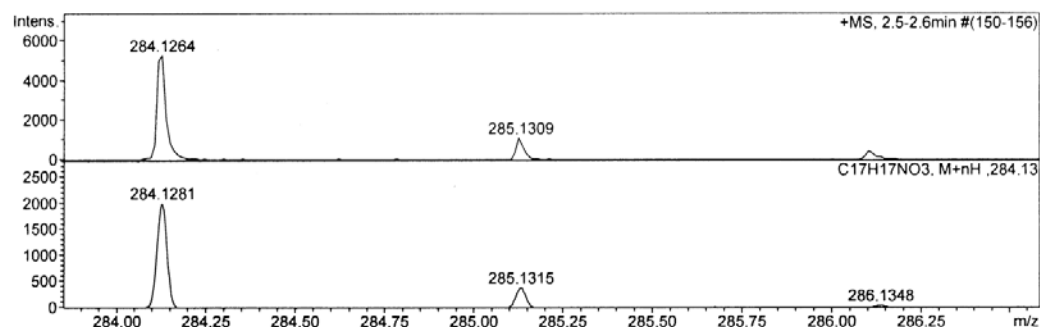
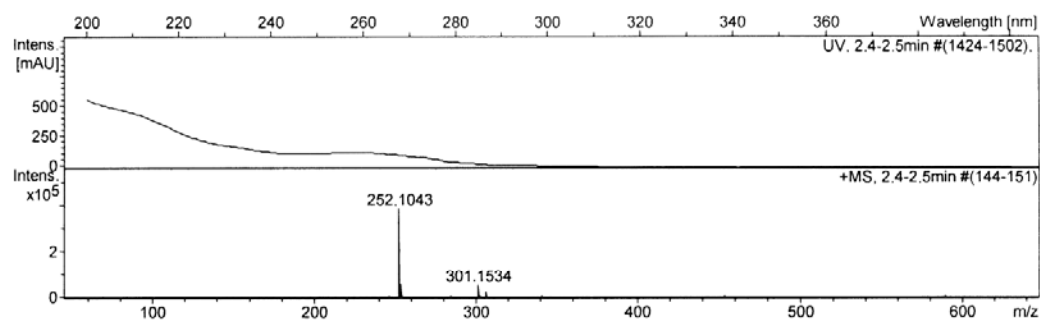
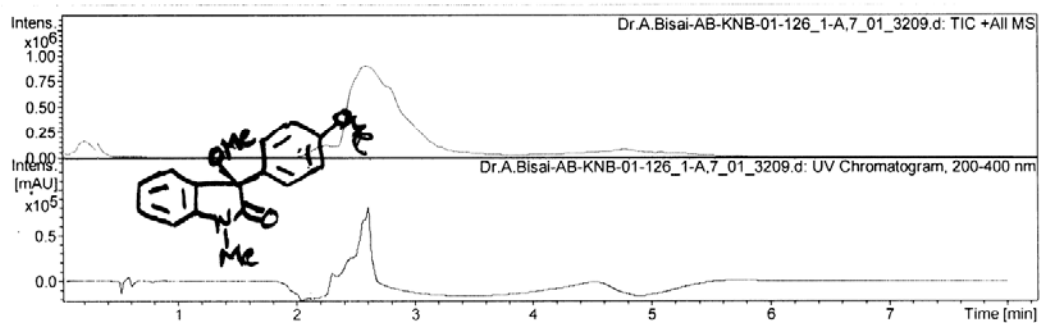
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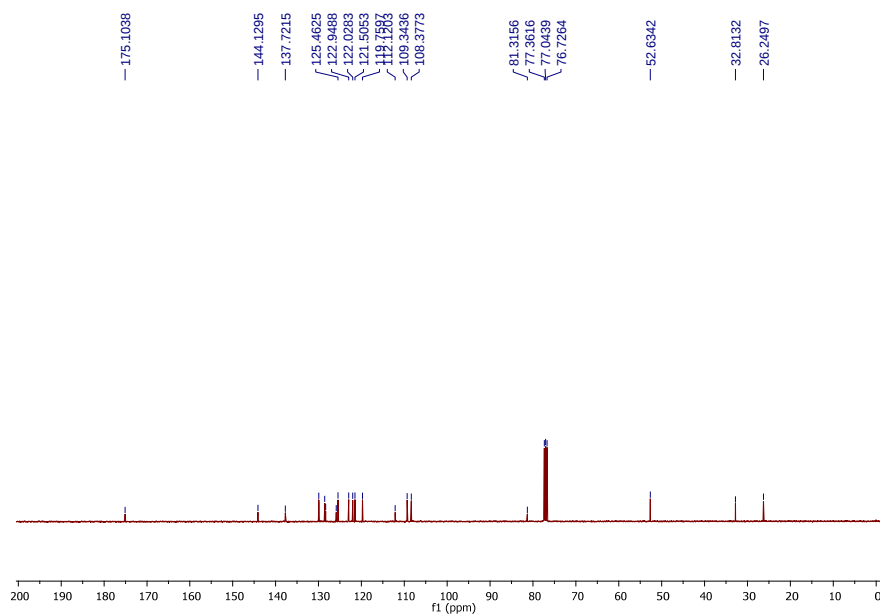
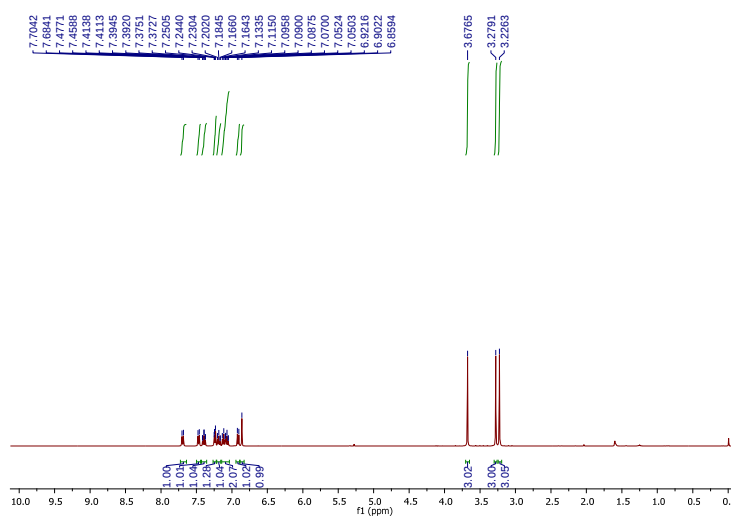
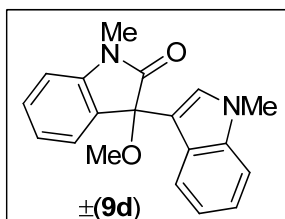
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Scanned copy of mass spectrum of (±)-9b



Display Report

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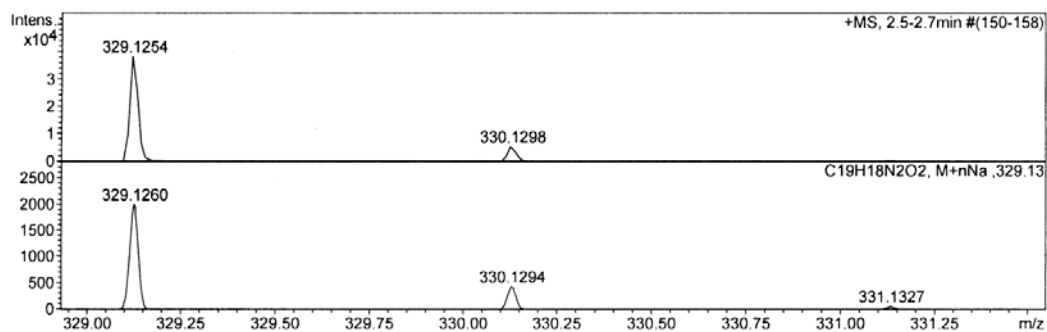
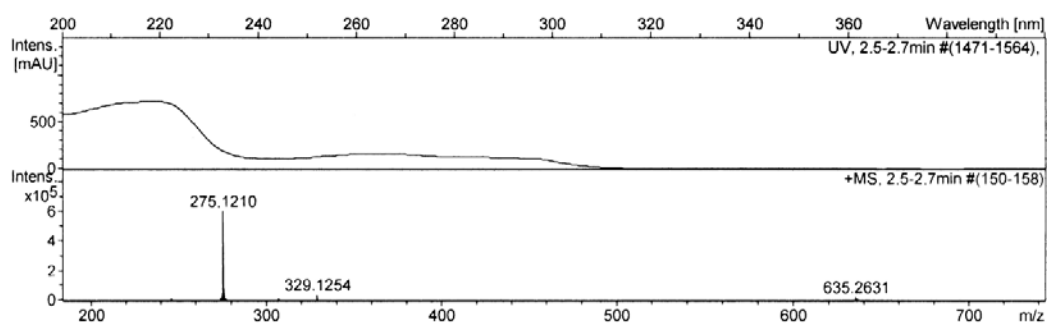
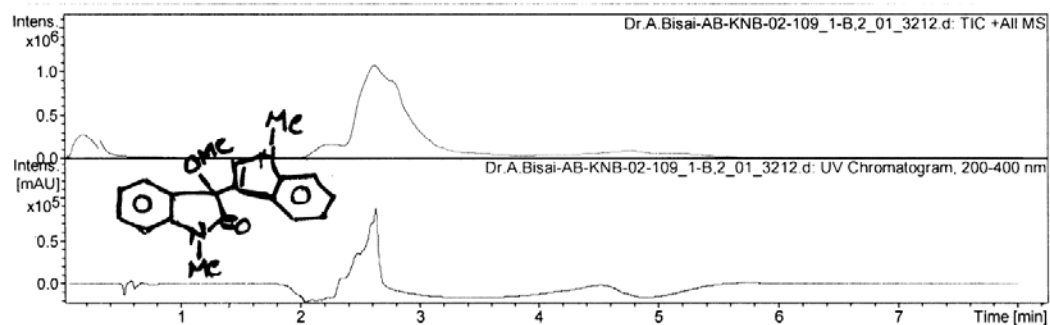
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Operator RUCHI

Instrument microTOF-Q II 10330

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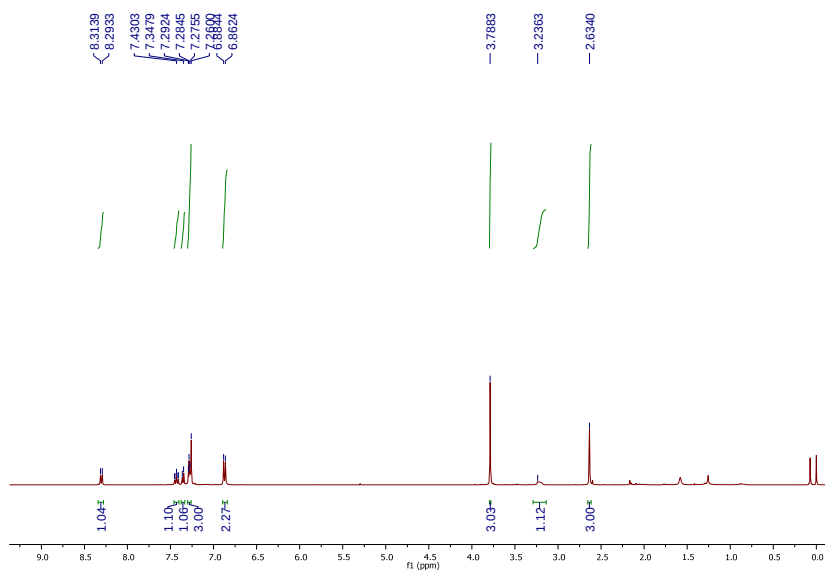
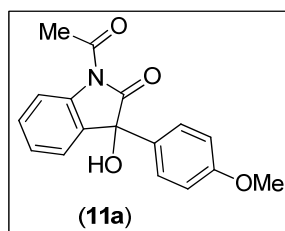


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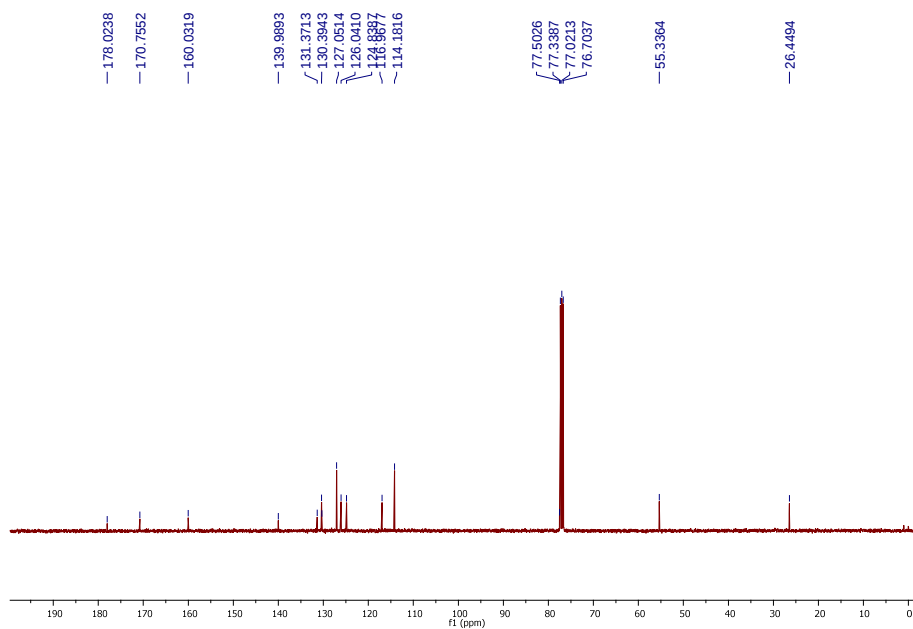
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Page 1 of 1

Scanned copy of mass spectrum of (±)-9d



¹H NMR (400 MHz, CDCl₃) compound (±)-**11a**



¹³C NMR (100 MHz, CDCl₃) compound (±)-**11a**

Display Report

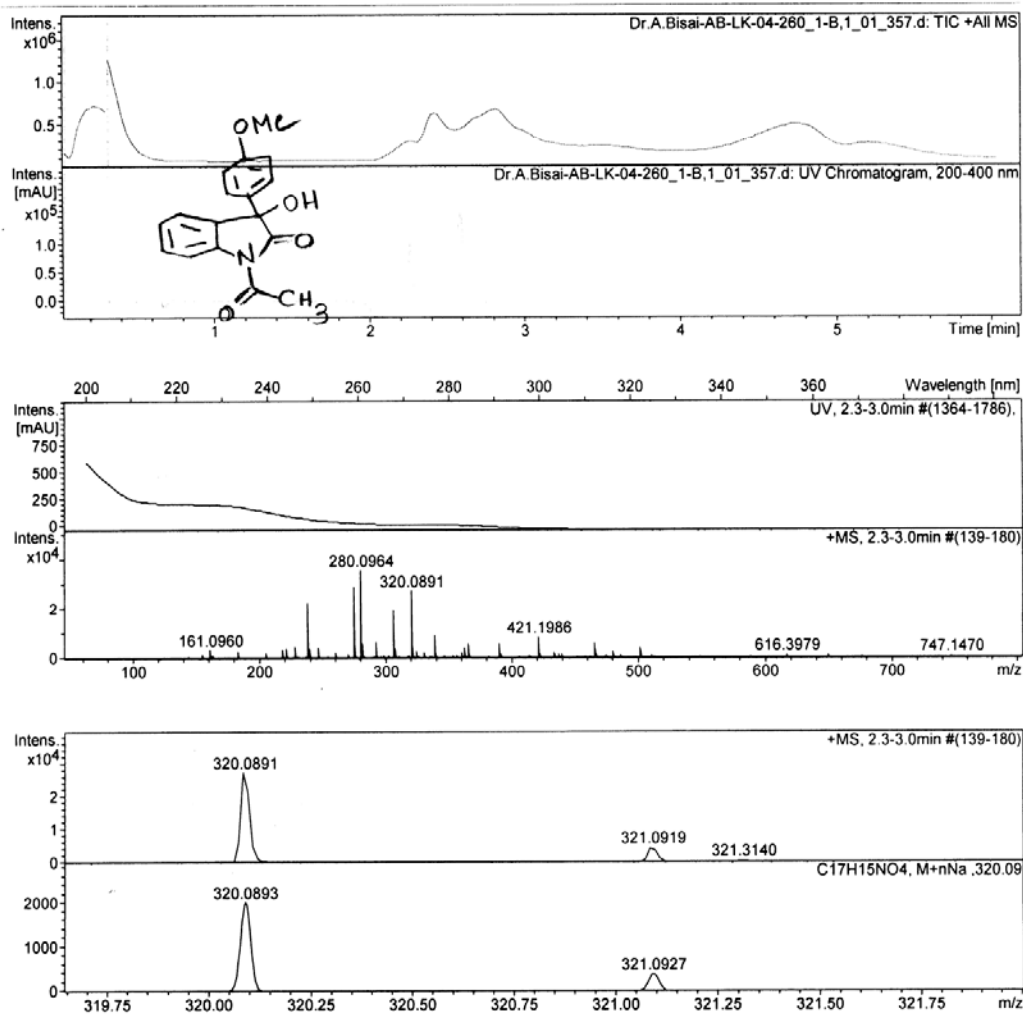
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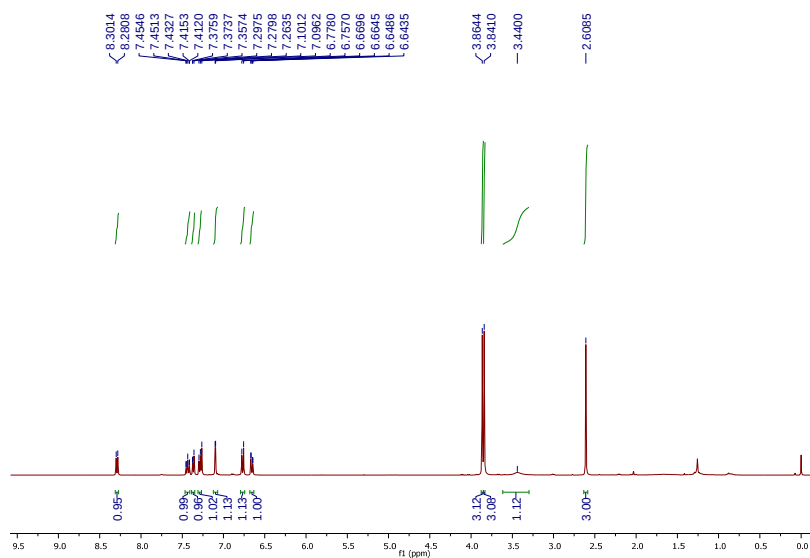
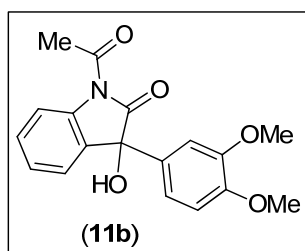
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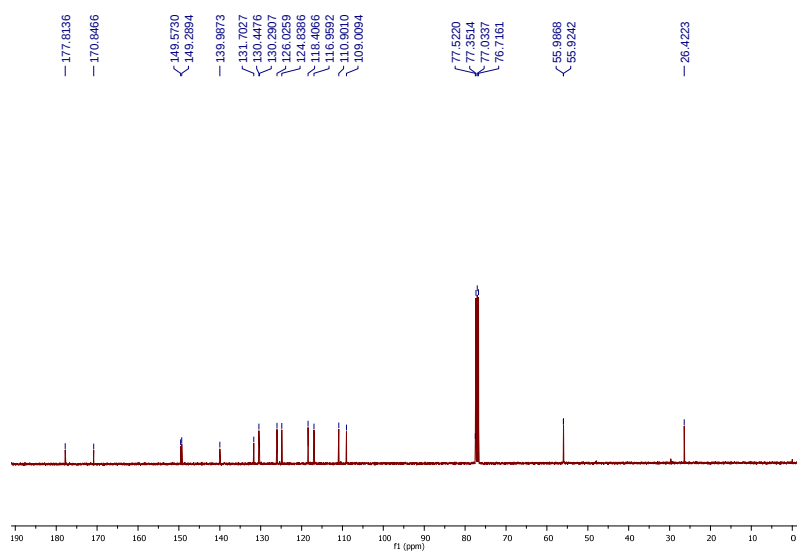
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Scanned copy of mass spectrum of (±)-11a



¹H NMR (400 MHz, CDCl₃) compound (±)-**11b**



¹³C NMR (100 MHz, CDCl₃) compound (±)-**11b**

Display Report

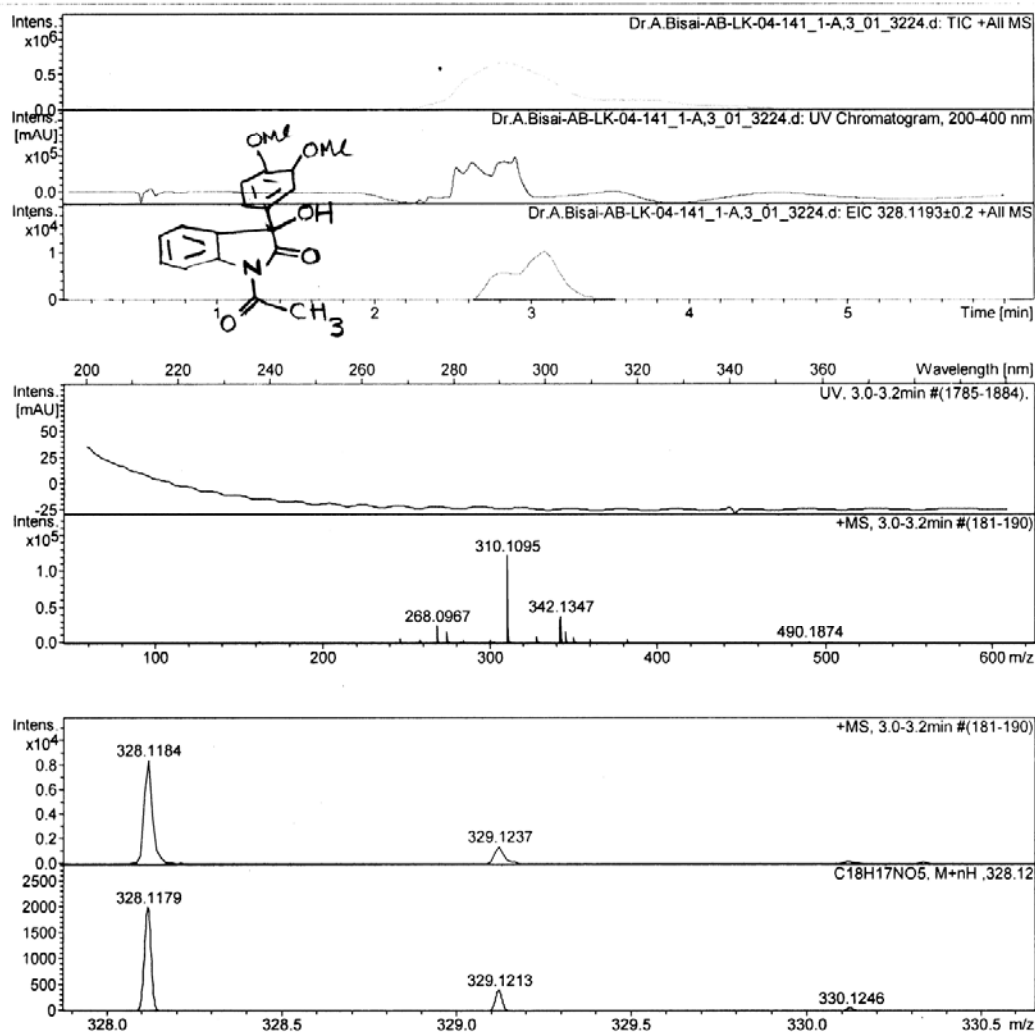
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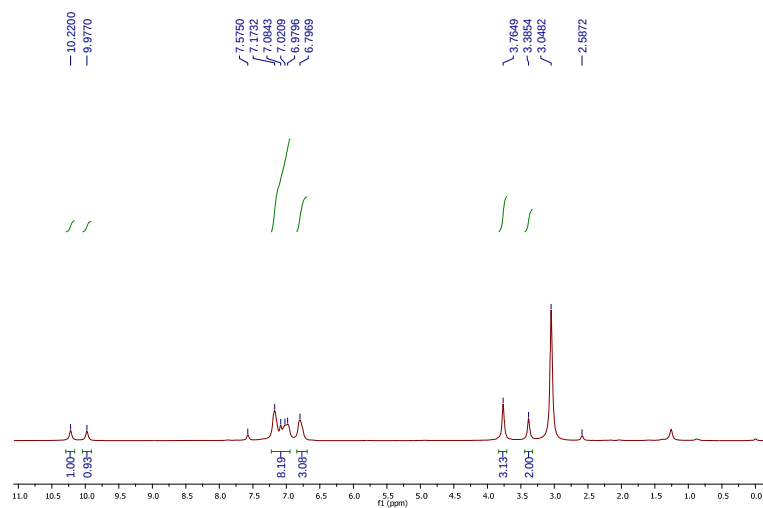
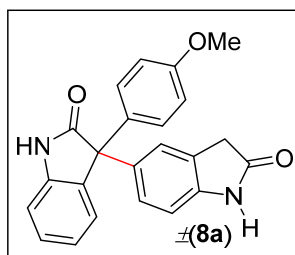
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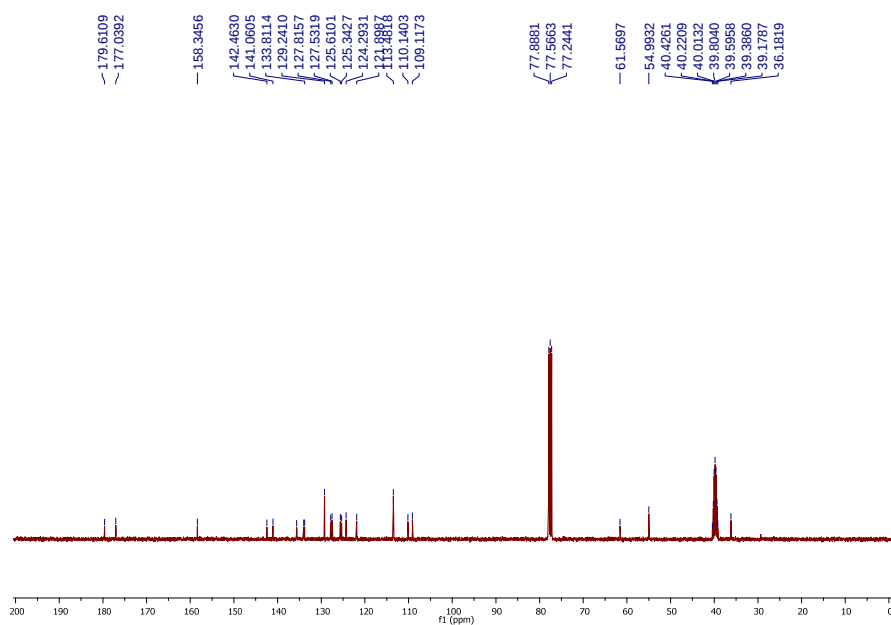
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Scanned copy of mass spectrum of (±)-11b



¹H NMR (400 MHz, 0.5 mL CDCl₃, 0.1 mL DMSO-D₆) of compound (±)-**8a**



¹³C NMR (100 MHz, 0.5 mL CDCl₃, 0.1 mL DMSO-D₆) of compound (±)-**8a**

Display Report

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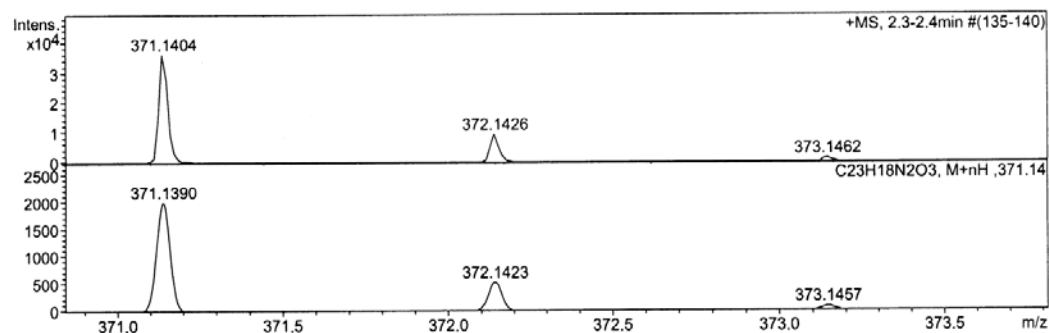
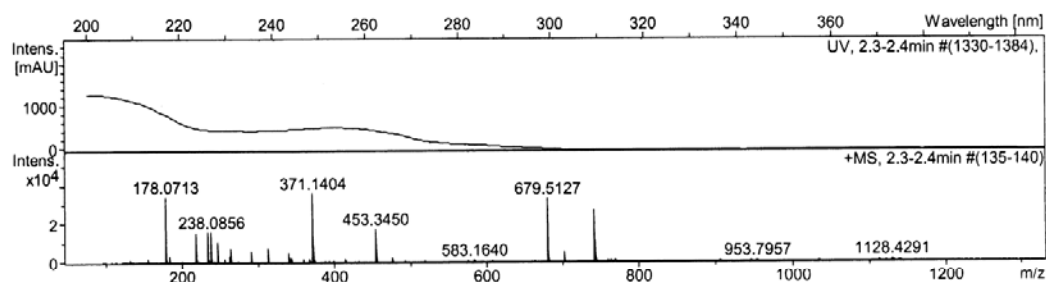
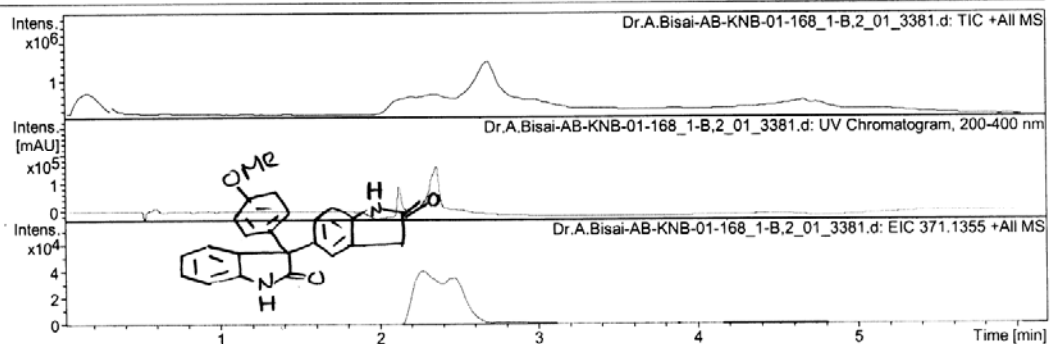
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Operator Ravindra

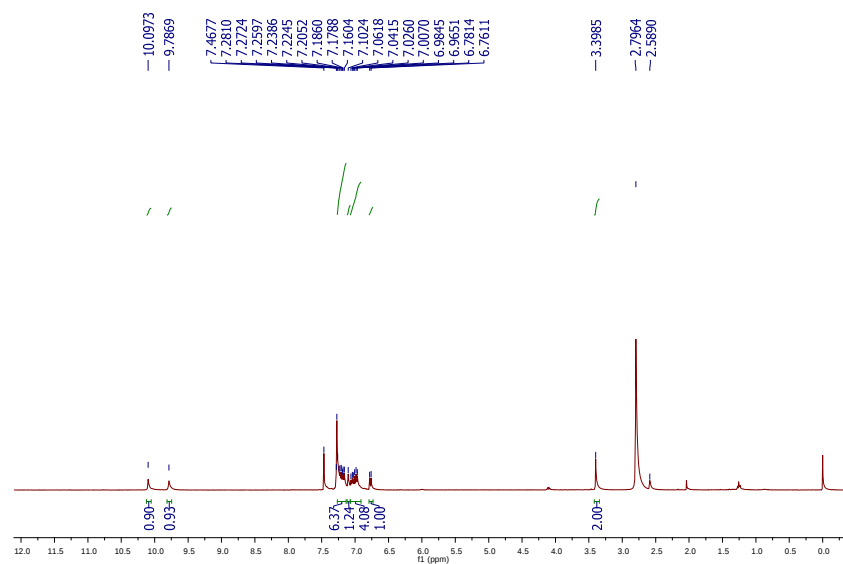
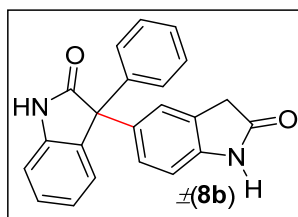
Instrument micrOTOF-Q II 10330

Acquisition Parameter

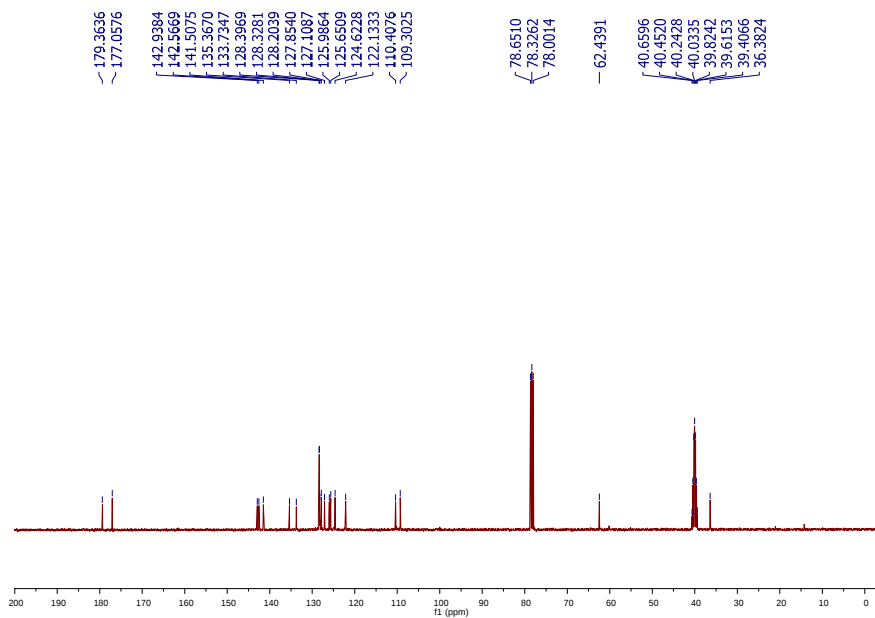
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8a



^1H NMR (400 MHz, 0.3 mL CDCl_3 , 0.2 mL $\text{DMSO}-\text{D}_6$) of compound (±)-**8b**



^{13}C NMR (100 MHz, 0.3 mL CDCl_3 , 0.2 mL $\text{DMSO}-\text{D}_6$) of compound (±)-**8b**

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\31-JAN-2015\Dr.A.Bisai-AB-LK-04-179.d
Method tune_wide.m
Sample Name AB-LK-04-179
Comment

Acquisition Date 1/31/2015 12:36:37 PM

Operator

RUCHI

Instrument

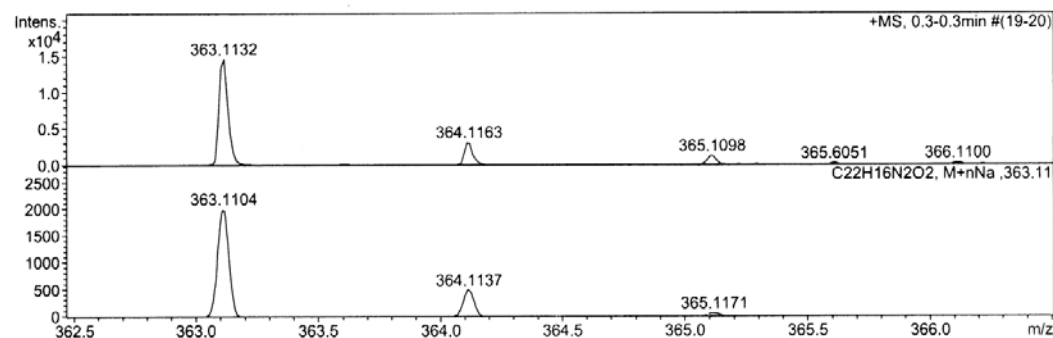
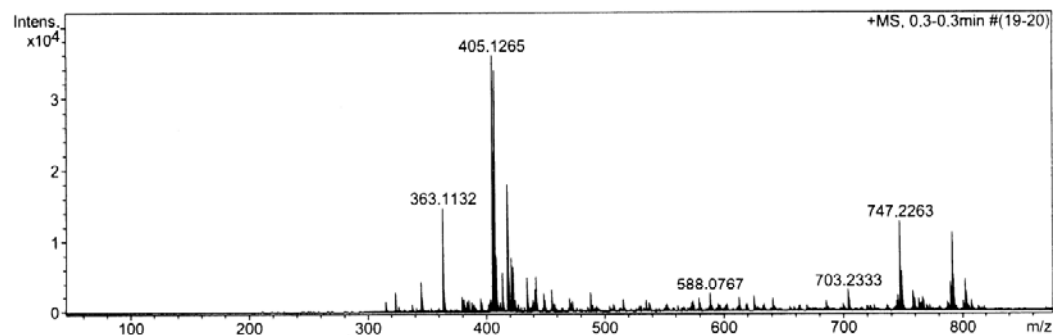
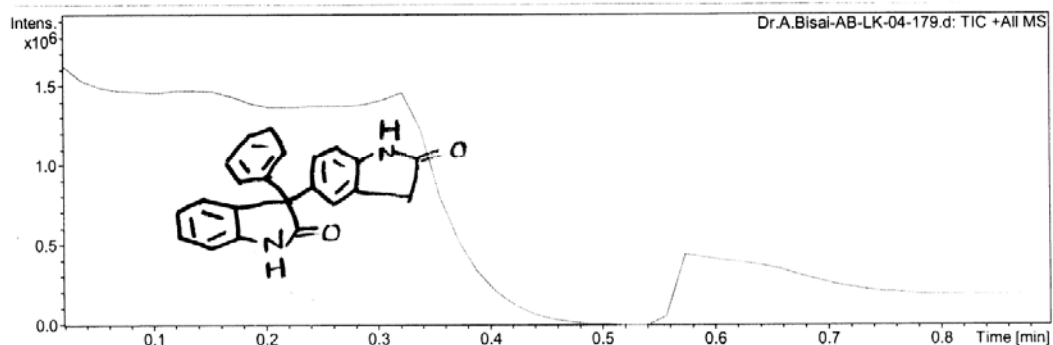
micrOTOF-Q II 10330

Acquisition Parameter

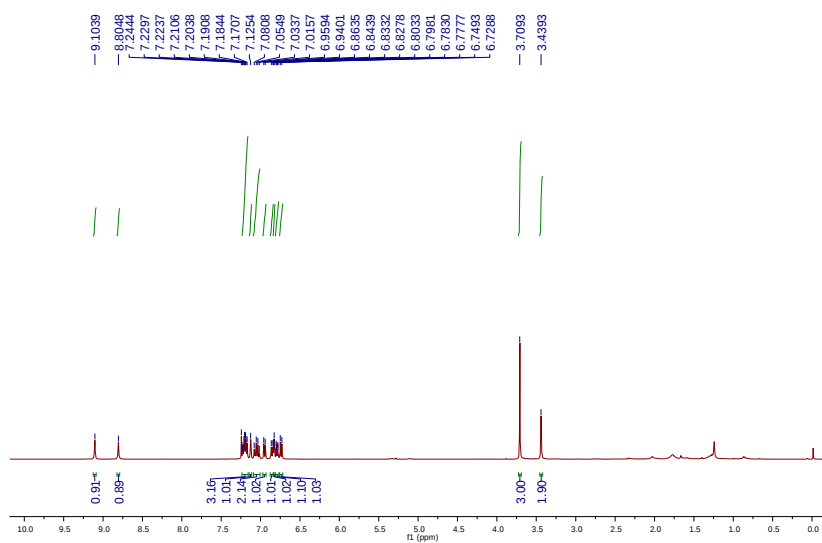
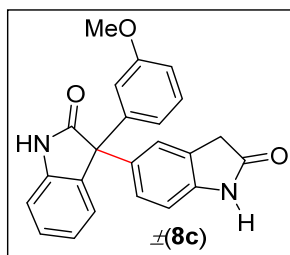
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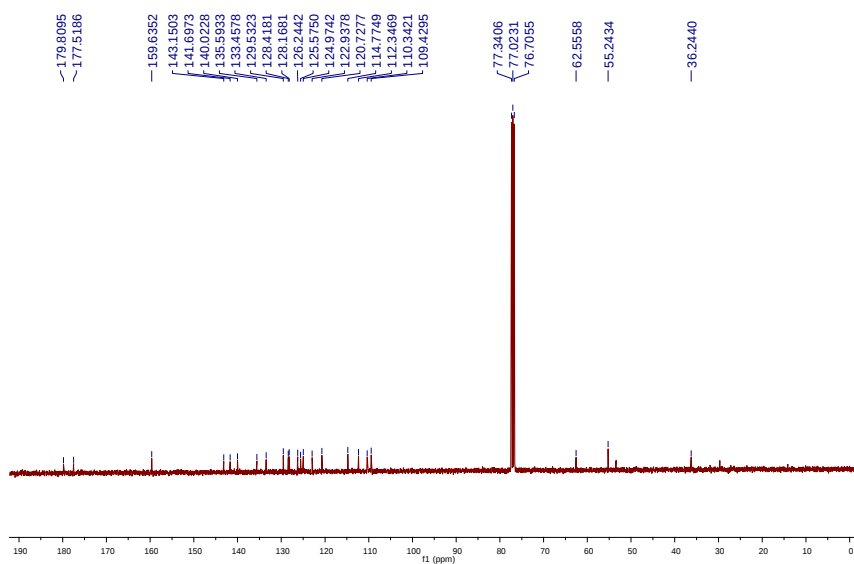
Set Nebulizer 0.4 Bar
Set Dry Heater 180 °C
Set Dry Gas 4.0 l/min
Set Divert Valve Waste



Scanned copy of mass spectrum of (±)-8b



¹H NMR (400 MHz, CDCl₃) of compound (±)-8c



¹³C NMR (100 MHz, CDCl₃) of compound (±)-8c

Display Report

Analysis Info

Analysis Name D:\Data\user data\2014\OCTMBER\09 oct\Dr.A.Bisai-AB-LK-04-248_1-A,6_01_337.d
 Method HRLCMS-20 Sept.m
 Sample Name Dr.A.Bisai-AB-LK-04-248
 Comment

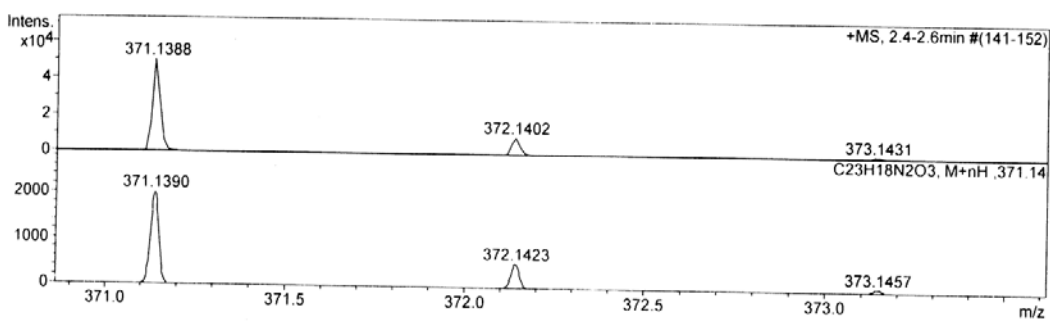
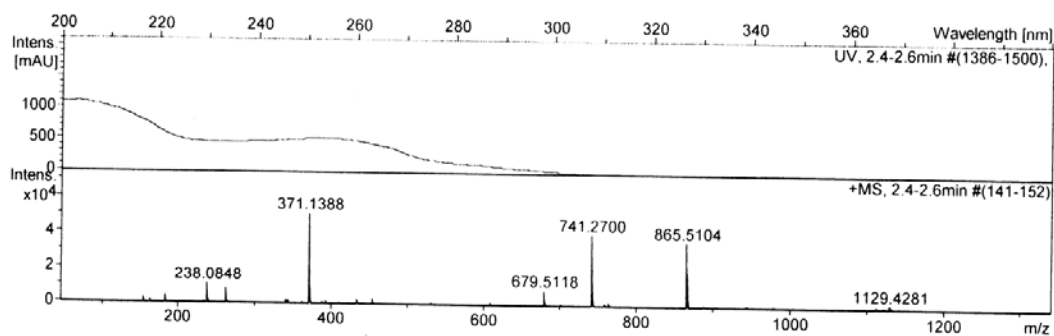
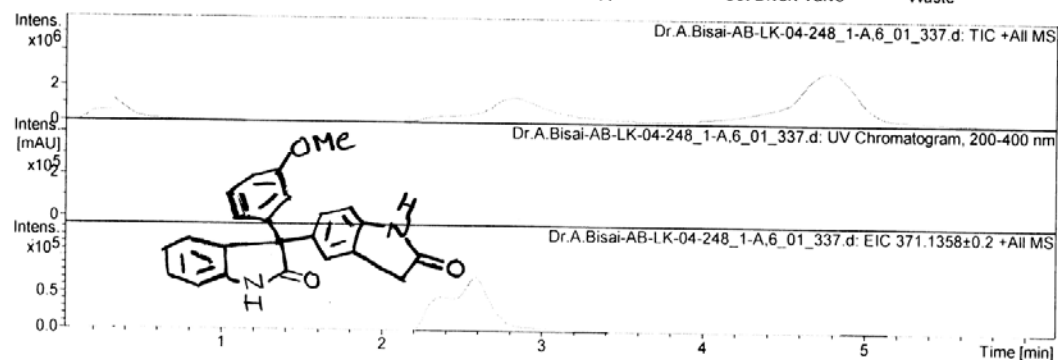
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Operator RUCHI

Instrument micrOTOF-Q II 10330

Acquisition Parameter

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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

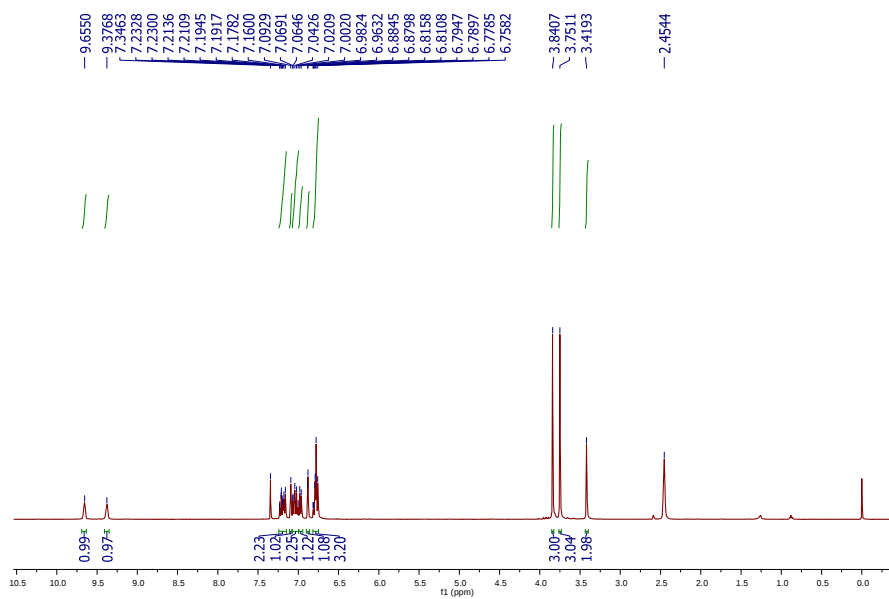
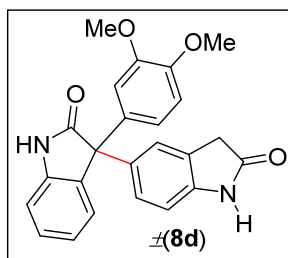


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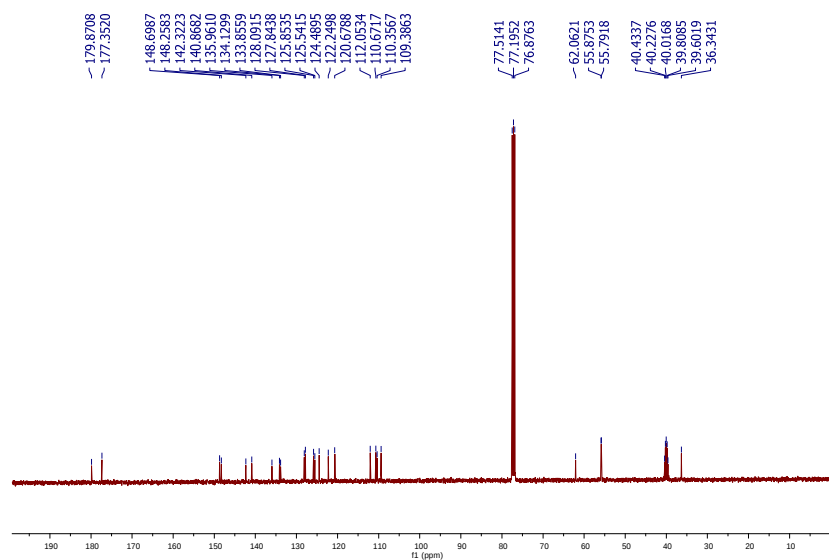
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Page 1 of 1

Scanned copy of mass spectrum of (±)-8c



¹H NMR (400 MHz, 0.5 mL CDCl₃, 0.1 mL DMSO-D₆) of compound (±)-8d



¹³C NMR (100 MHz, 0.5 mL CDCl₃, 0.1 mL DMSO-D₆) of compound (±)-8d

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\29-JAN-2015\Dr.A.Bisai-AB-LK-04179_1-A.3_01_1506.d
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 Sample Name Dr.A.Bisai-AB-LK-04179
 Comment

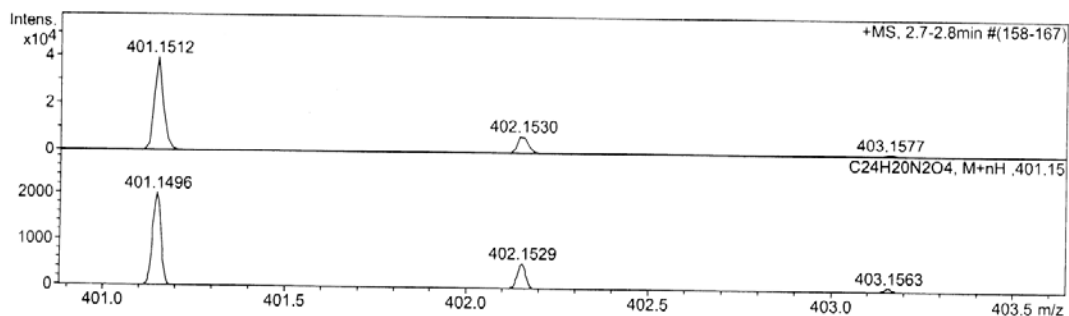
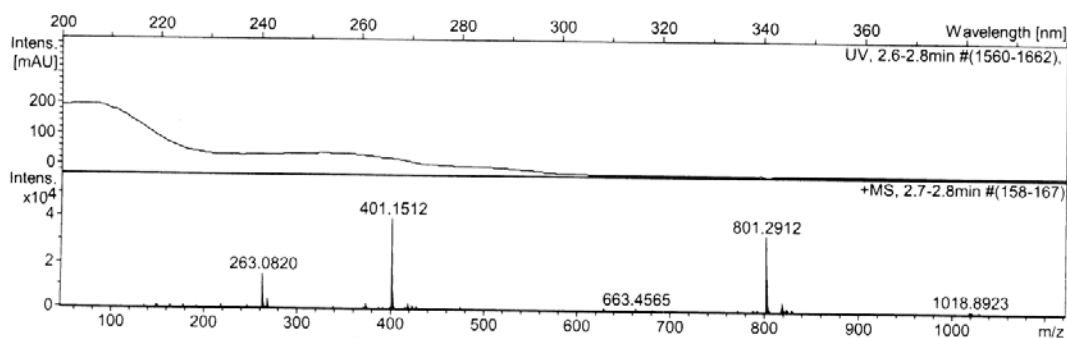
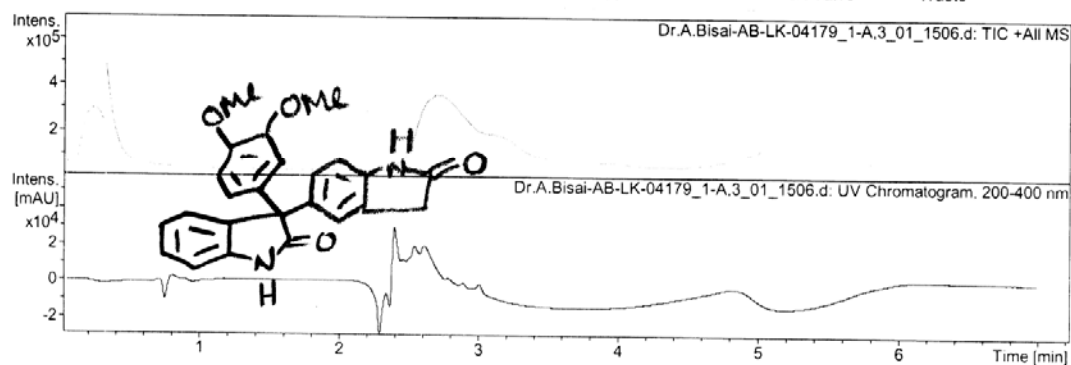
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Operator RUCHI

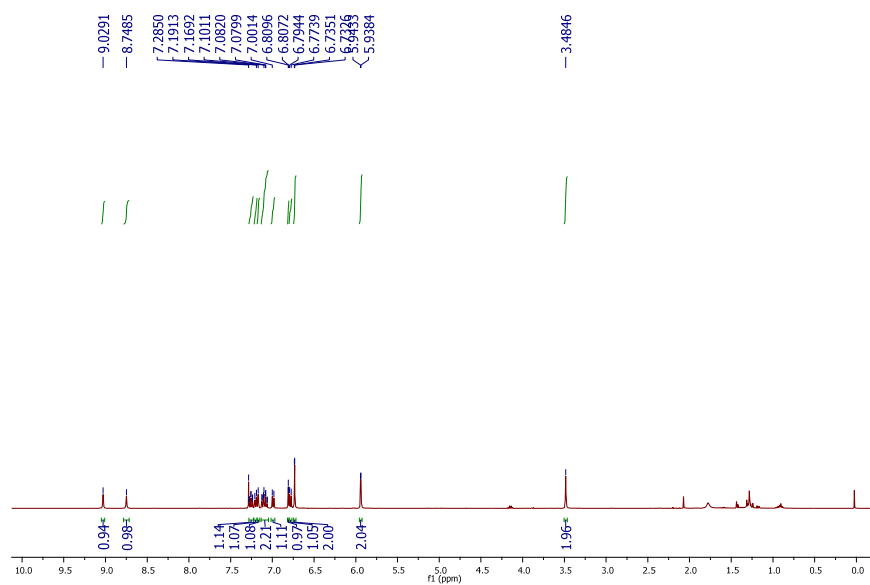
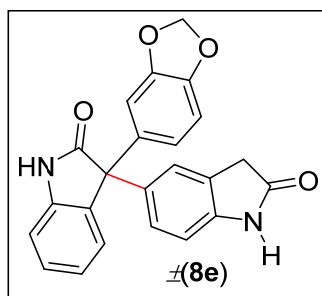
Instrument micrOTOF-Q II 10330

Acquisition Parameter

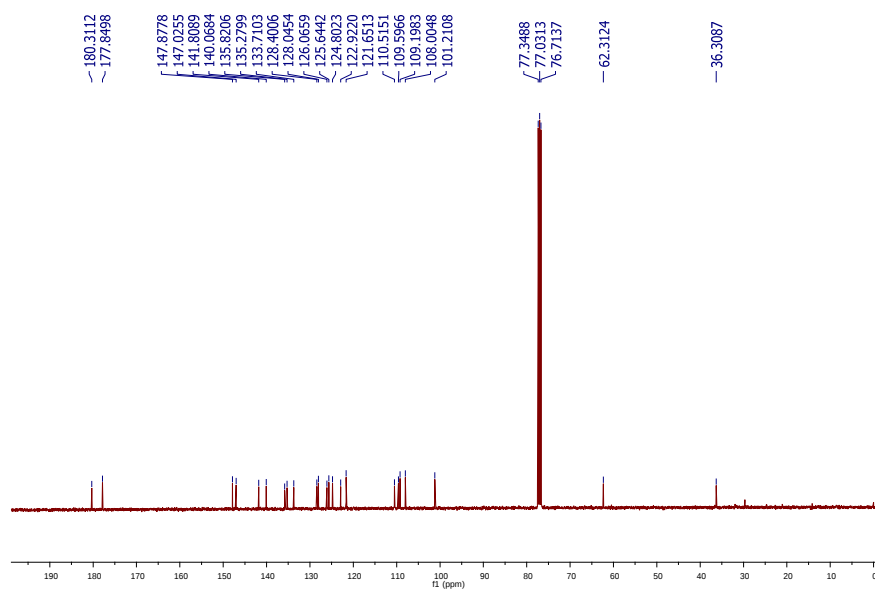
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8d



¹H NMR (400 MHz, CDCl₃) of compound (±)-8e



¹³C NMR (100 MHz, CDCl₃) of compound (±)-8e

Display Report

Analysis Info

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Method tune_wide.m
Sample Name AB-LK-04-191-1
Comment

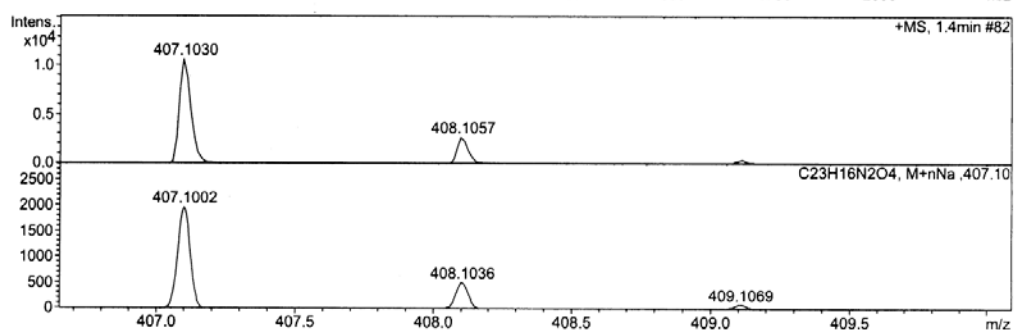
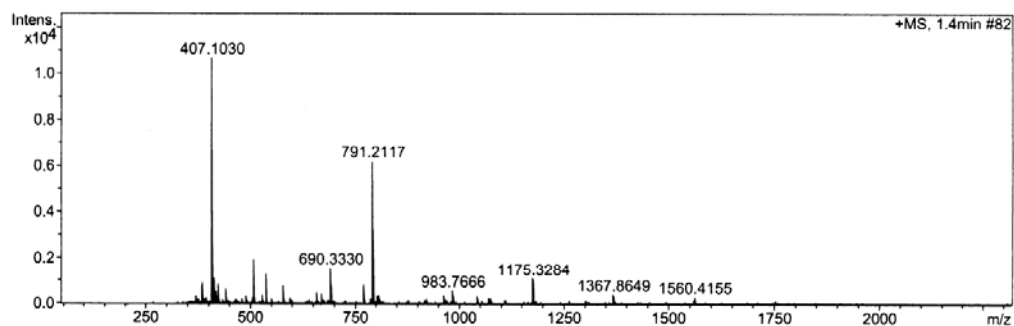
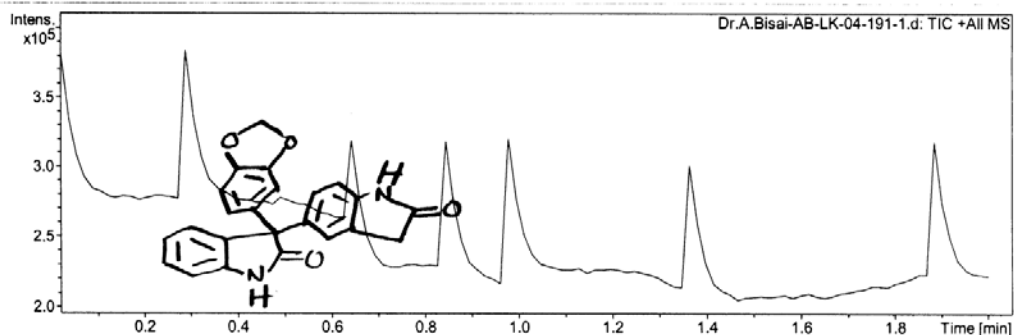
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Operator RUCHI

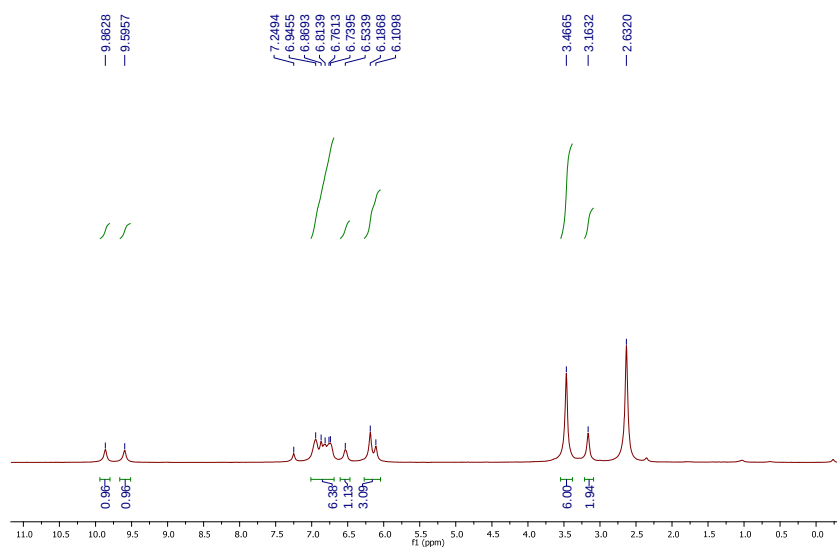
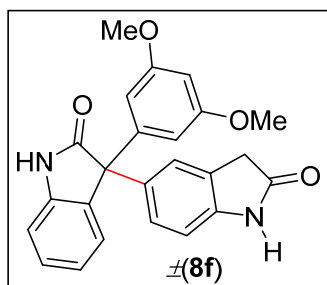
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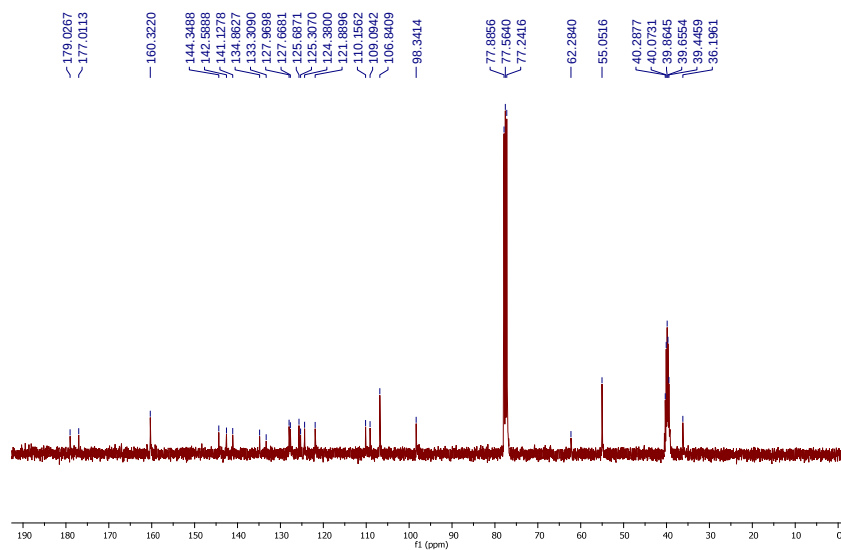
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Scan End	3000 m/z	Set Collision Cell RF	700.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8e



¹H NMR (400 MHz, 0.2 mL CDCl₃, 0.3 mL DMSO-D₆) of compound (±)-8f



¹³C NMR (100 MHz, 0.5 mL CDCl₃, 0.1 mL DMSO-D₆) of compound (±)-8f

Display Report

Analysis Info

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 Sample Name Dr.A.Bisai-AB-knb-01-255
 Comment

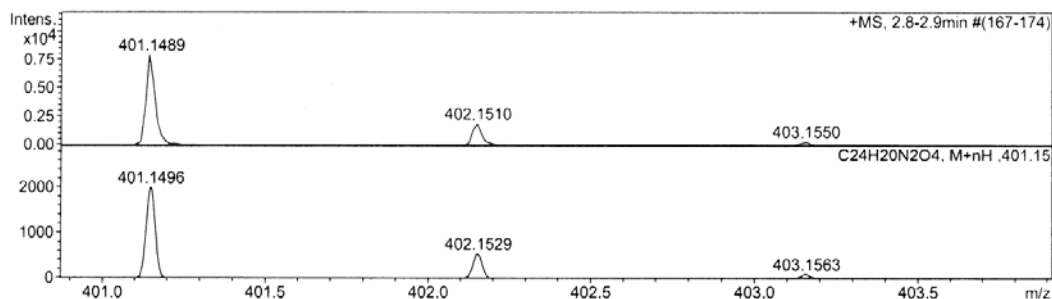
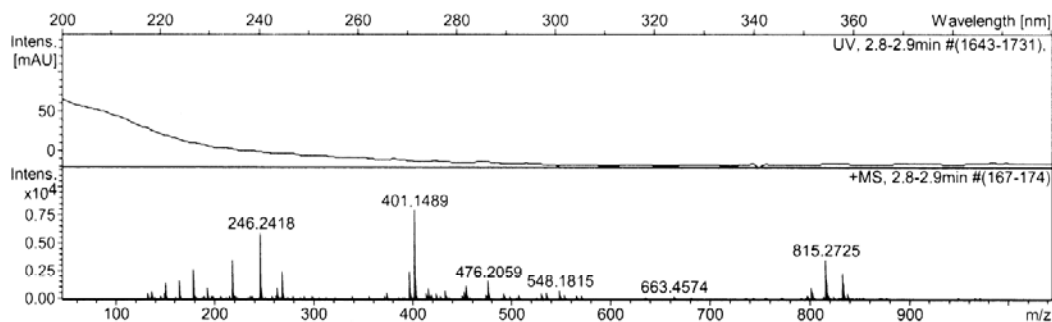
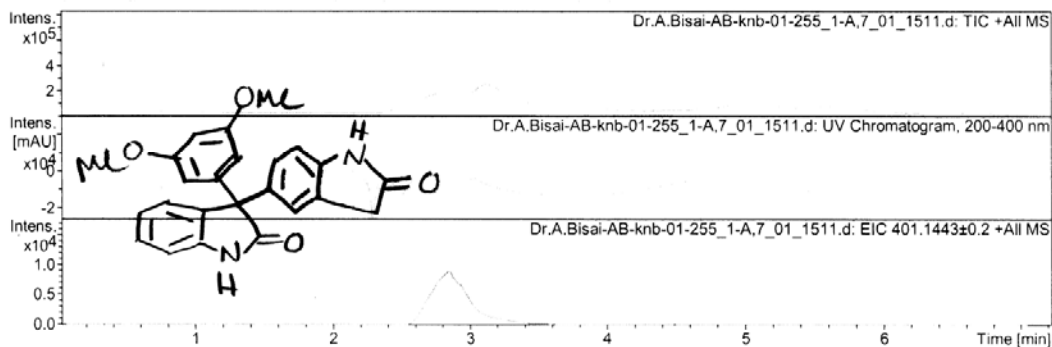
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Operator RUCHI

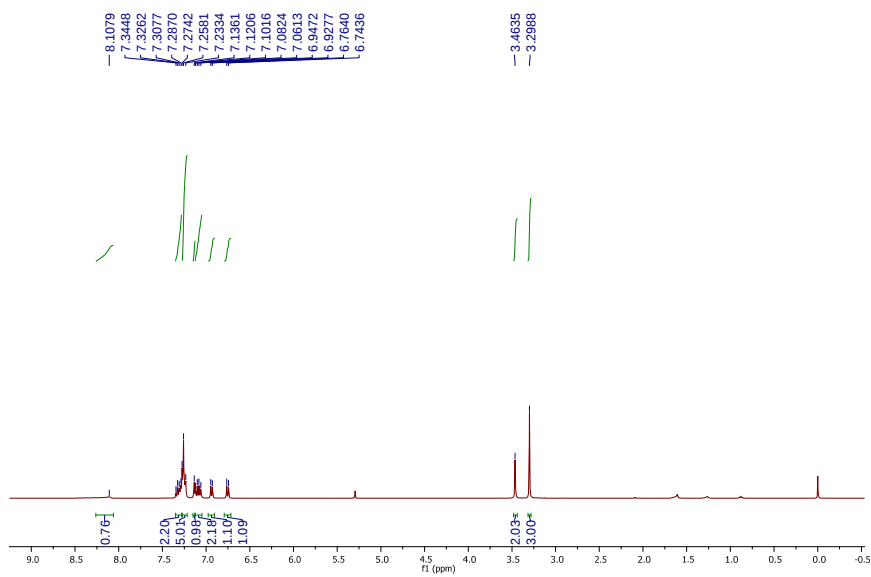
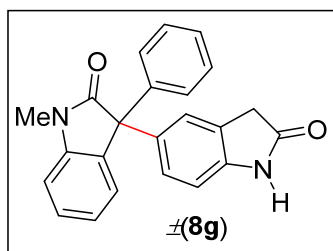
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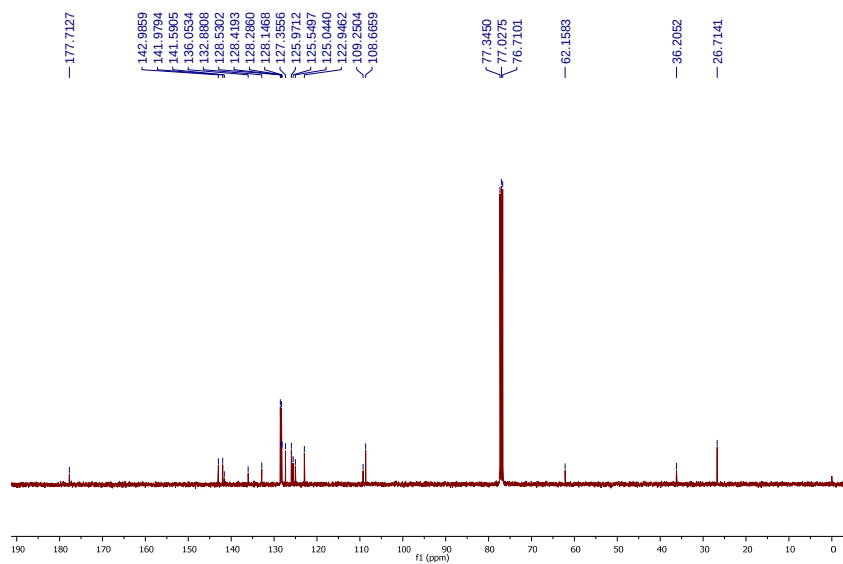
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8f



¹H NMR (400 MHz, CDCl₃) of compound (±)-8g



¹³C NMR (100 MHz, CDCl₃) of compound (±)-8g

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\27-JAN-2015\Dr.A.Bisai-AB-LK-04-273_1-B,3_01_1488.d
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Sample Name Dr.A.Bisai-AB-LK-04-273
Comment

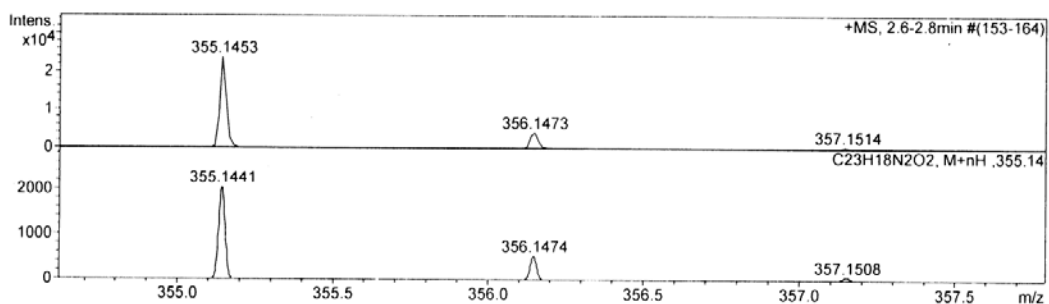
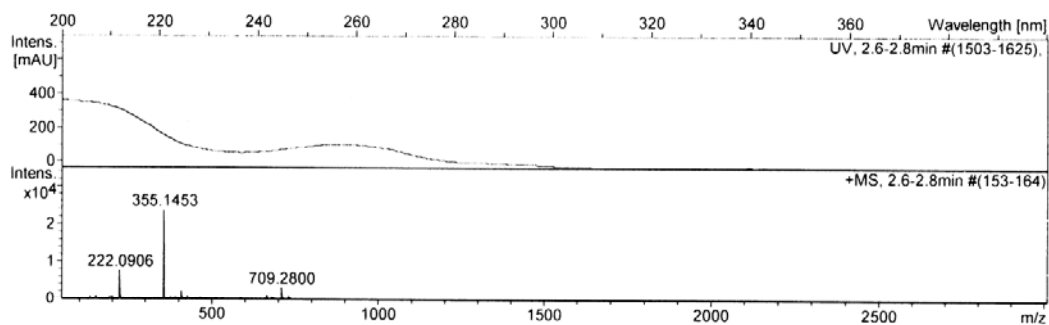
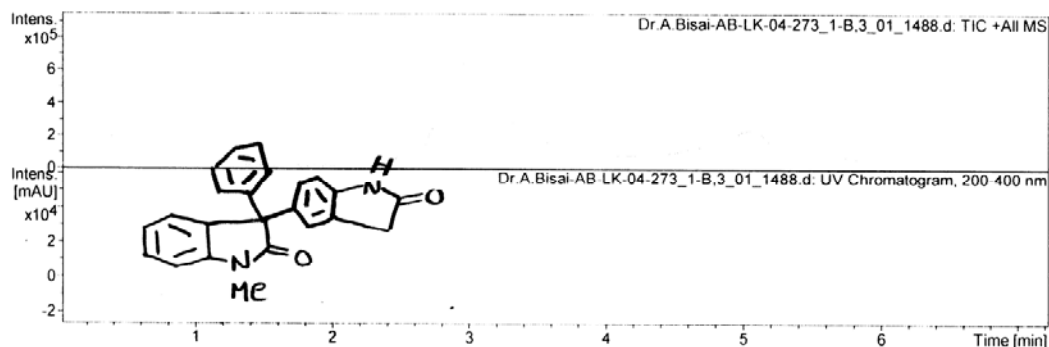
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Operator RUCHI

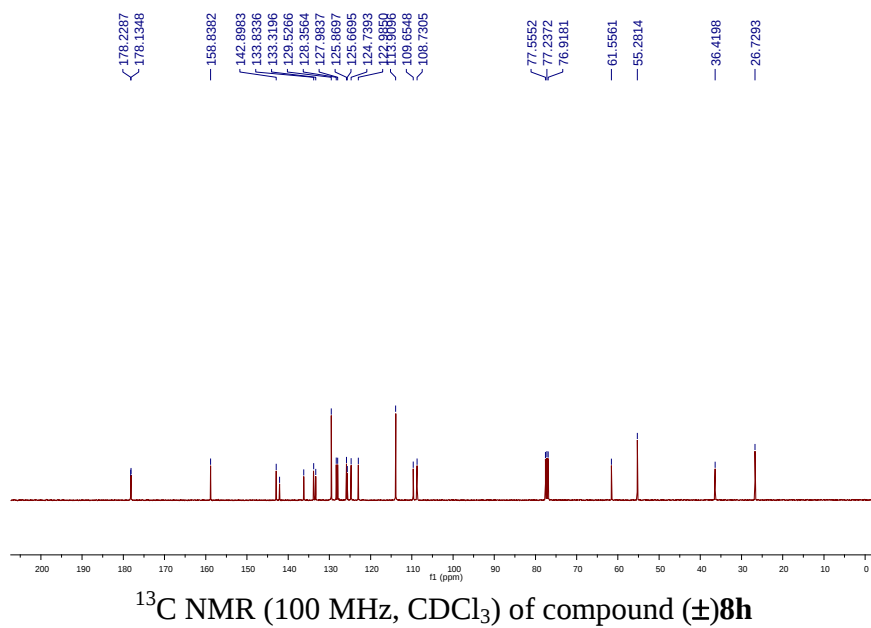
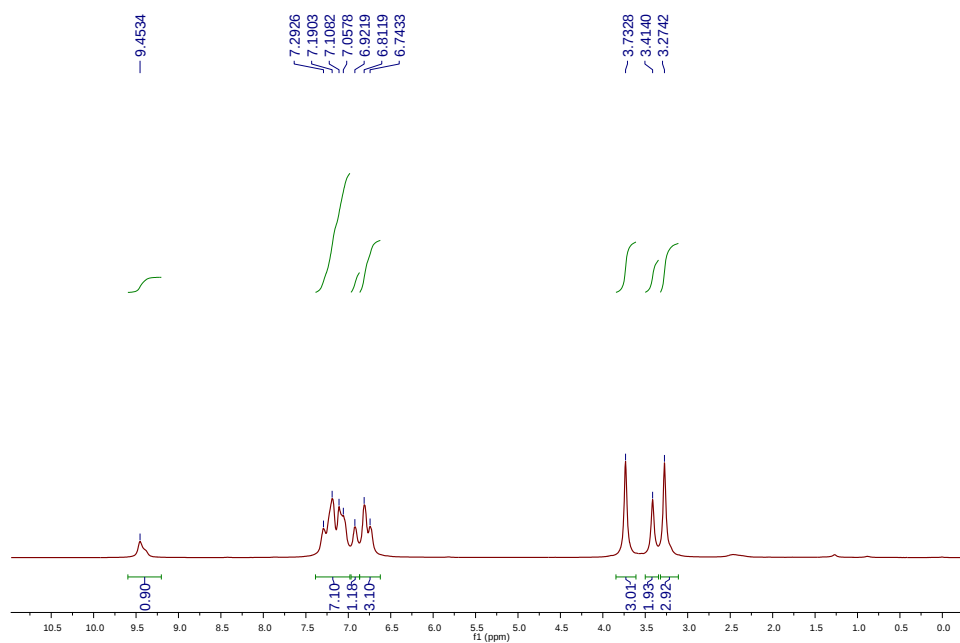
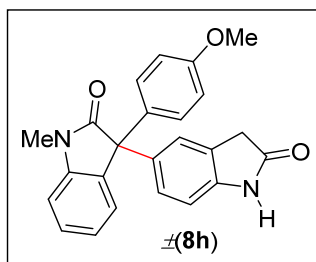
Instrument microTOF-Q II 10330

Acquisition Parameter

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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8g



Display Report

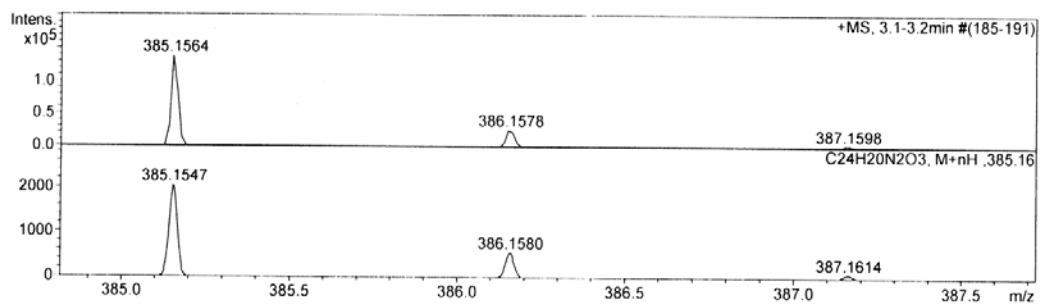
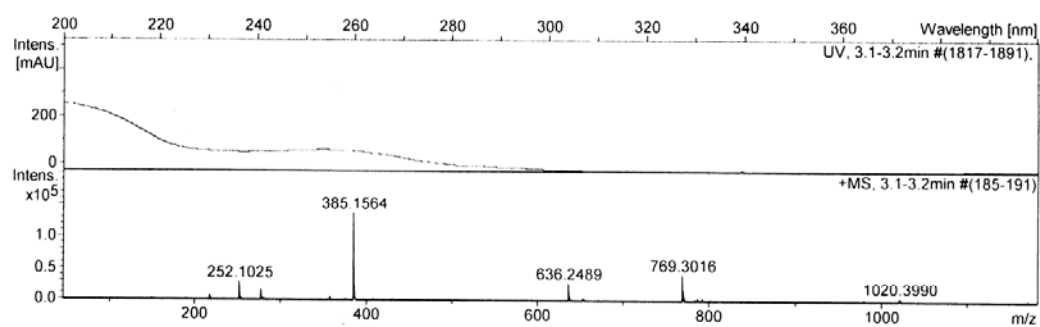
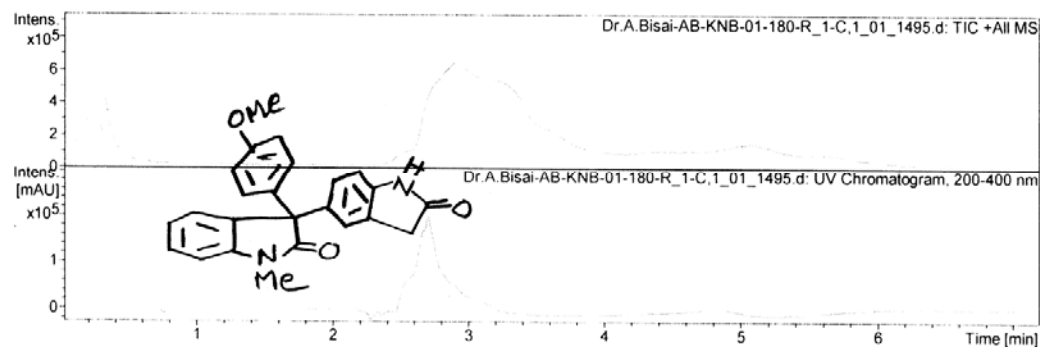
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 Sample Name Dr.A Bisai-AB-KNB-01-180-R
 Comment

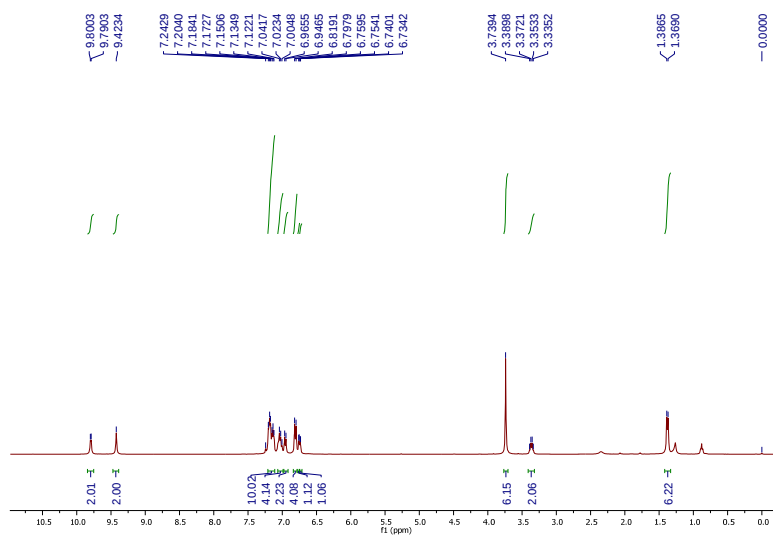
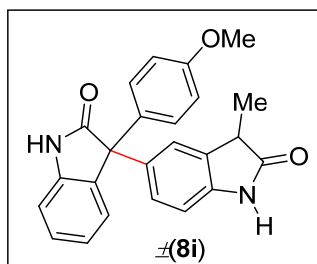
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 Operator RUCHI
 Instrument micrOTOF-Q II 10330

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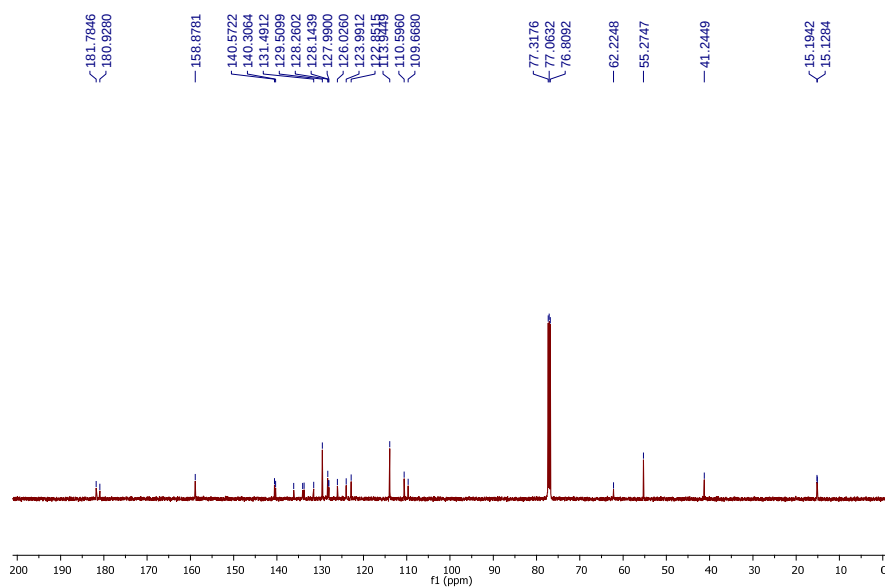
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8h



¹H NMR (400 MHz, CDCl₃) of compound (±)-8i



¹³C NMR (125 MHz, CDCl₃) of compound (±)-8i

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\FEB-2015\03-FEB-2015\Dr.A.Bisai-AB-LK-04-207_1-C.8_01_1569.d
 Method HRLCMS-20 Sept tune wide.m
 Sample Name Dr.A.Bisai-AB-LK-04-207
 Comment

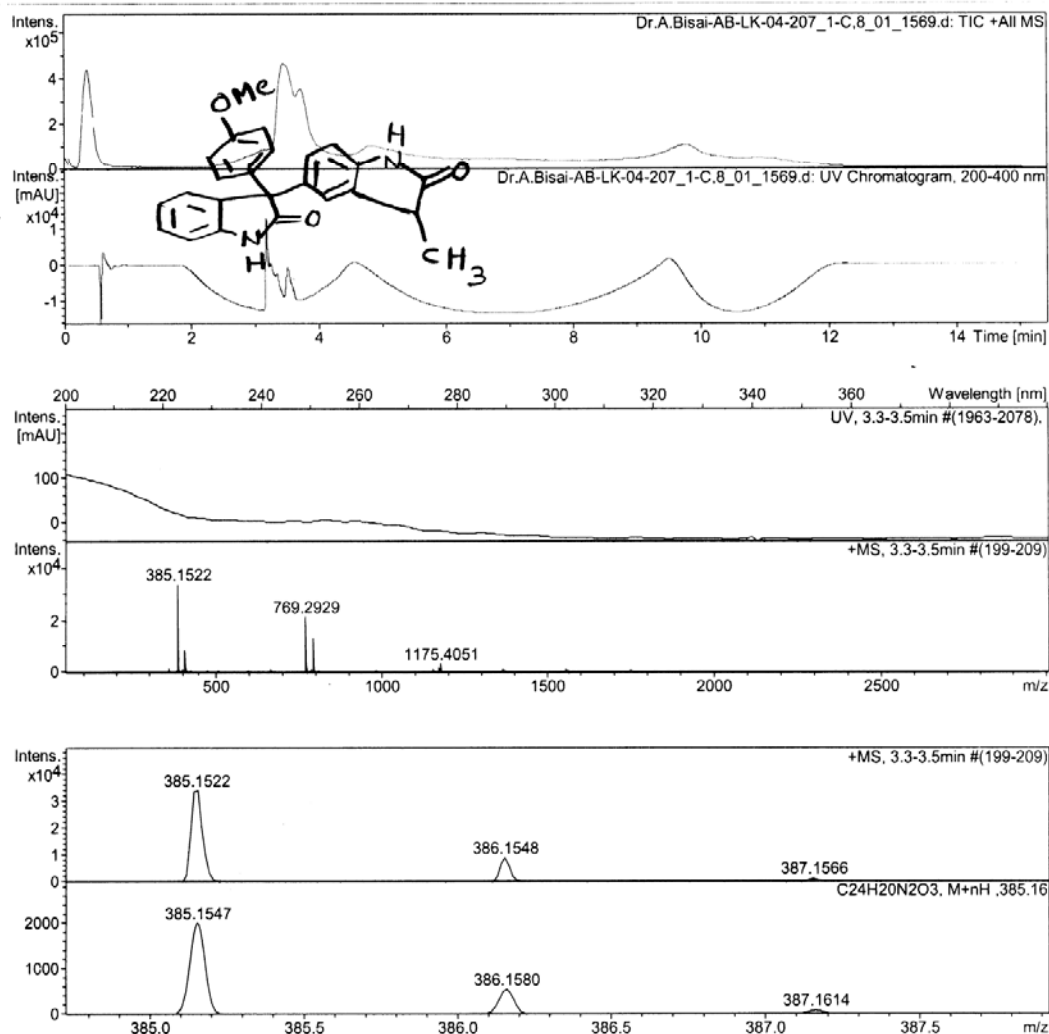
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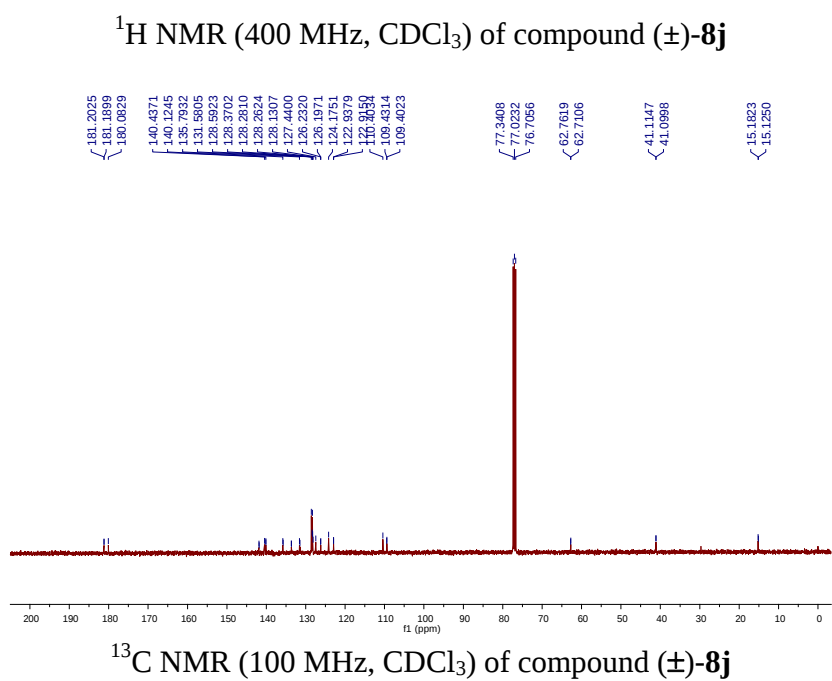
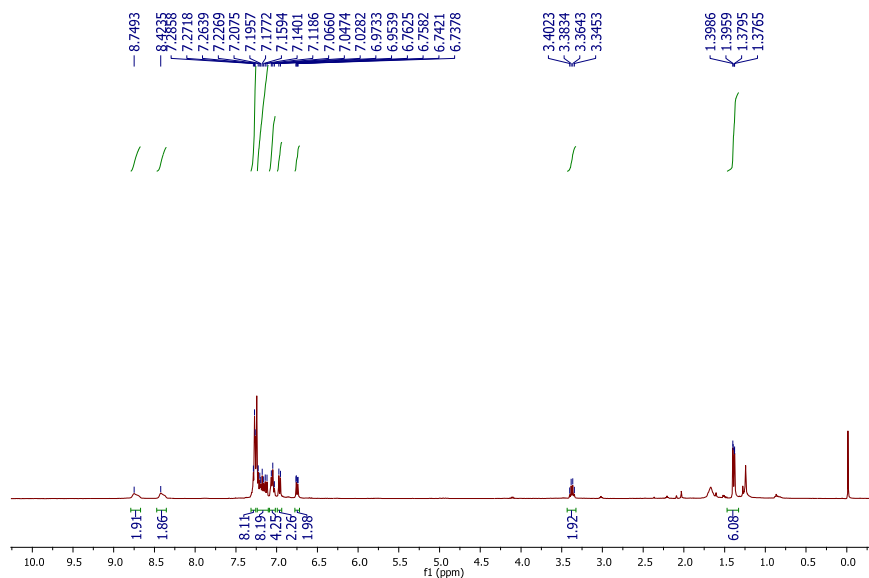
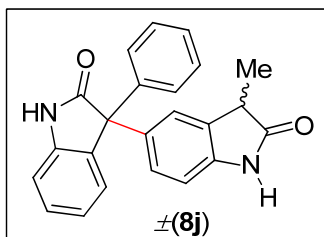
Operator RUCHI

Instrument micrOTOF-Q II 10330

Acquisition Parameter

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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of (±)-**8i**



Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\29-JAN-2015\Dr.A.Bisai-AB-LK-04-202_1-B,7_01_1521.d
Method HRLCMS-20 Sept tune wide.m
Sample Name Dr.A.Bisai-AB-LK-04-202
Comment

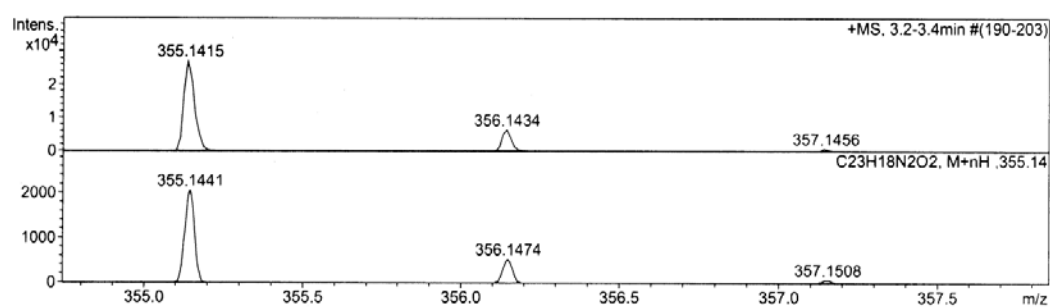
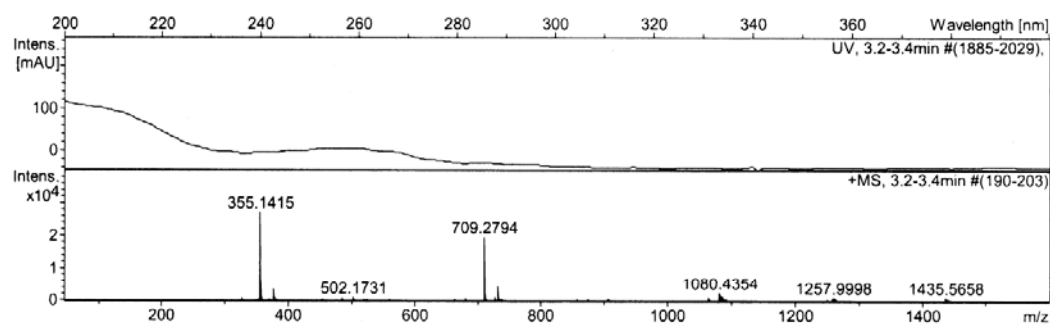
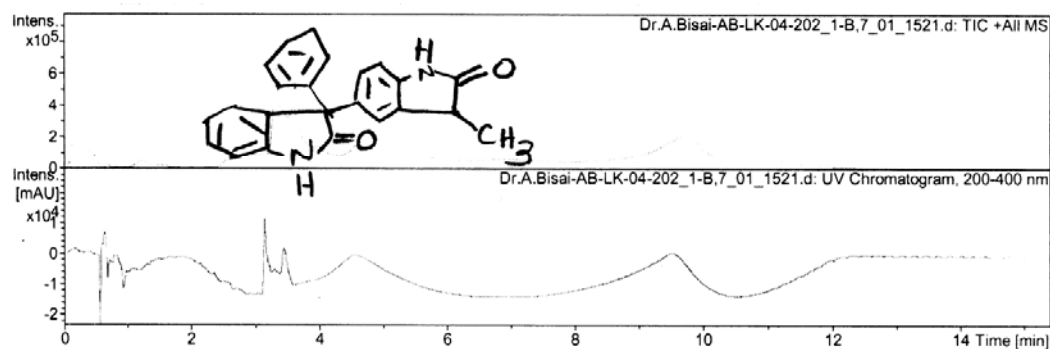
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Operator RUCHI

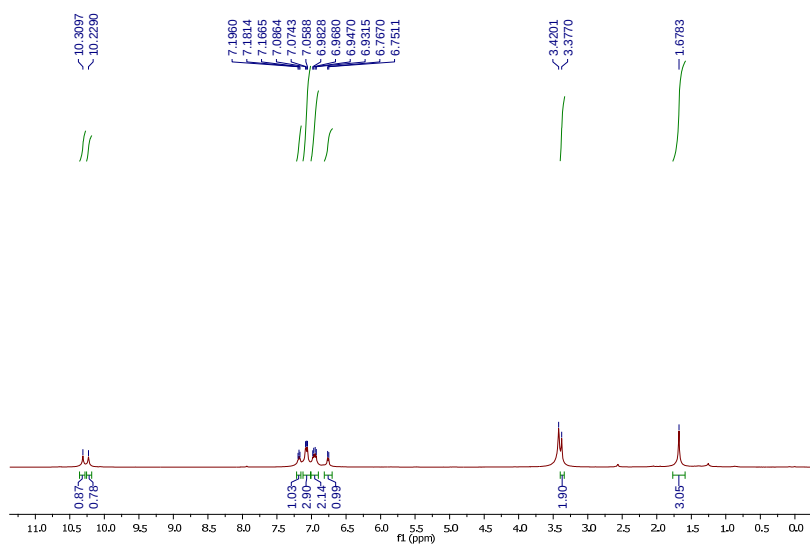
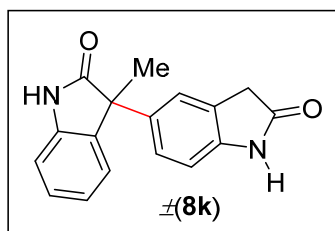
Instrument micrOTOF-Q II 10330

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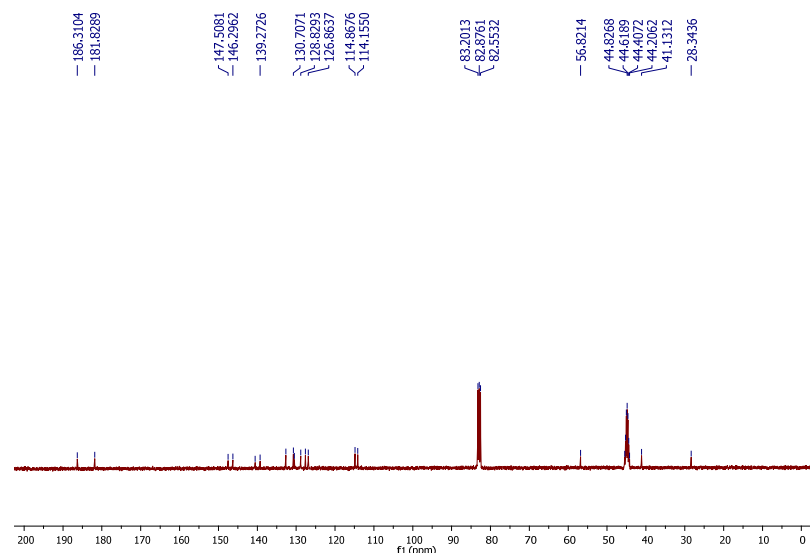
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Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8j



¹H NMR (400 MHz, 0.3 mL CDCl₃, 0.25 mL DMSO-D₆) of compound (±)-8k



¹³C NMR (100 MHz, 0.3 mL CDCl₃, 0.25 mL DMSO-D₆) of compound (±)-8k

Display Report

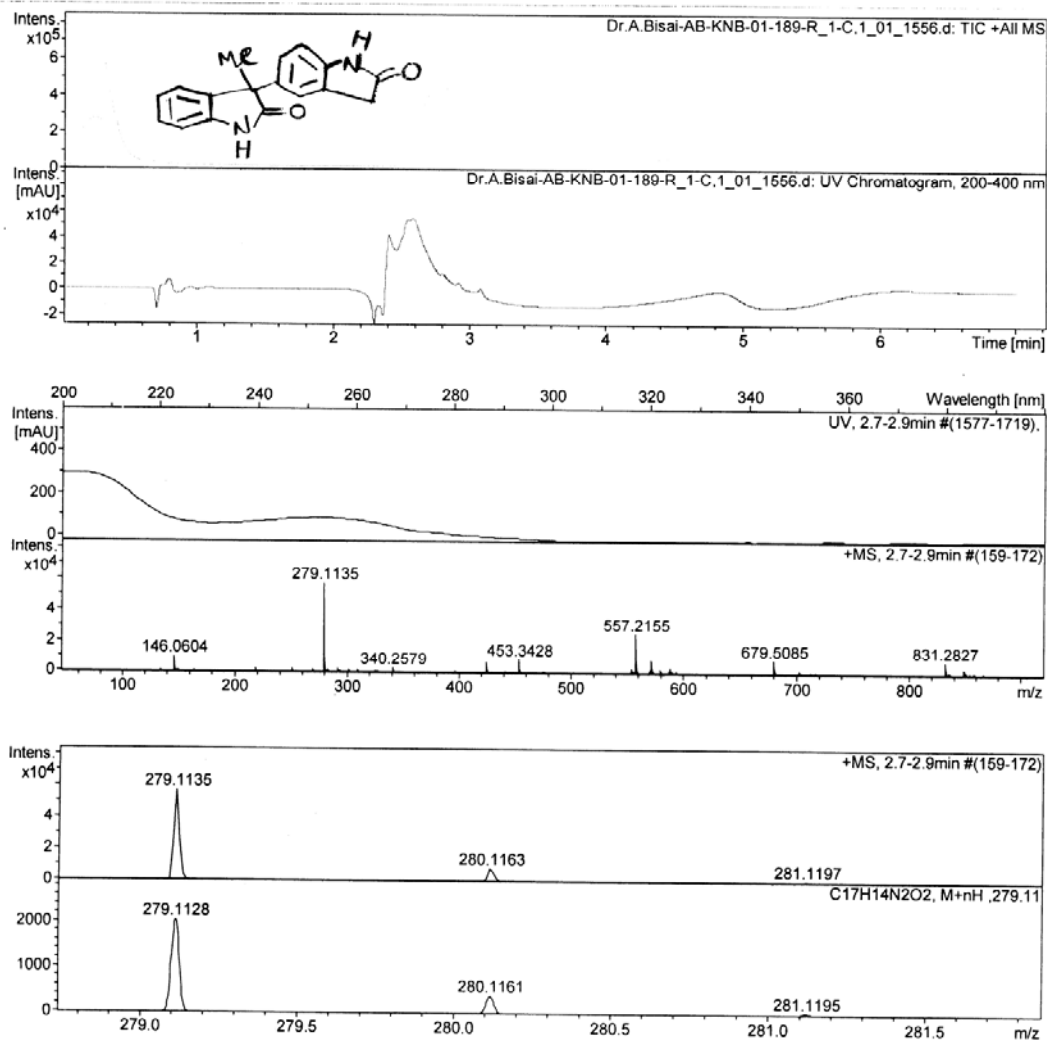
Analysis Info

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Method HRLCMS-20 Sept.m
Sample Name Dr.A.Bisai-AB-KNB-01-189-R
Comment

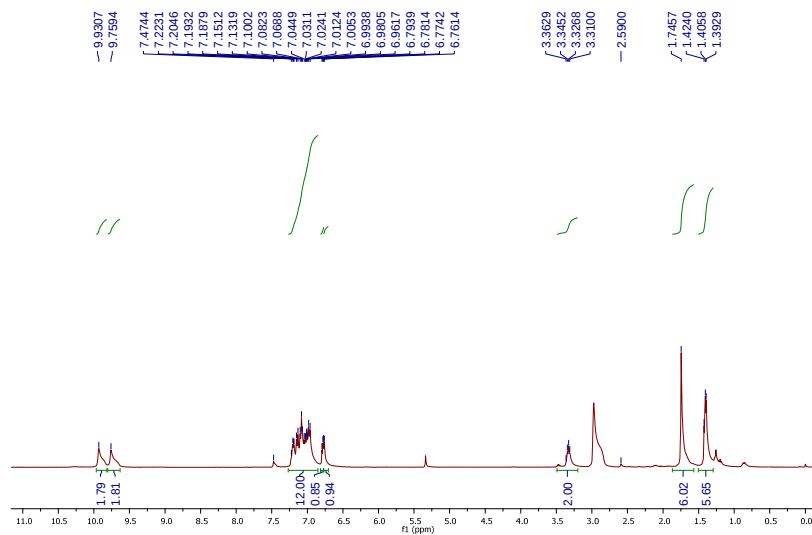
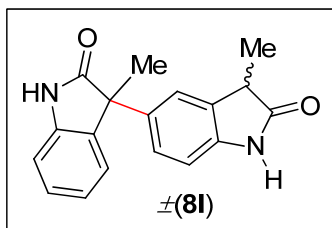
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Operator RUCHI
Instrument micrOTOF-Q II 10330

Acquisition Parameter

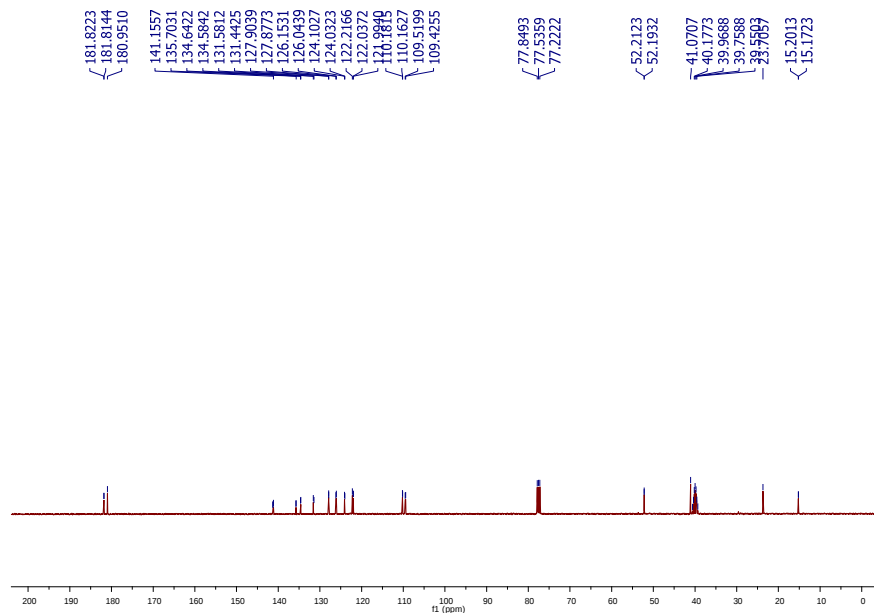
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8k



¹H NMR (400 MHz, 0.3 mL CDCl₃, 0.25 mL DMSO-D₆) of compound (±)-8I



¹³C NMR (100 MHz, 0.3 mL CDCl₃, 0.25 mL DMSO-D₆) of compound (±)-8I

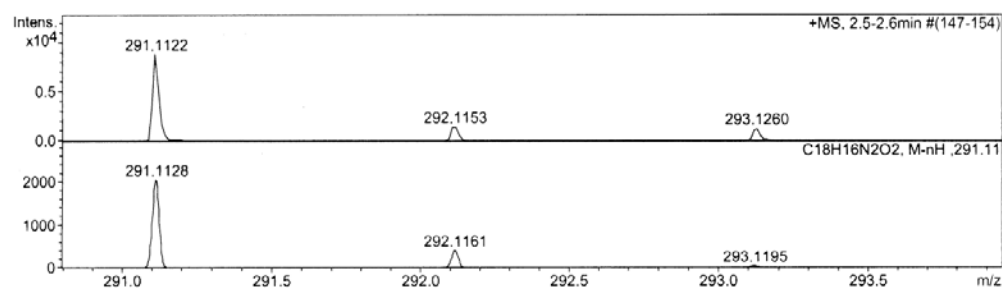
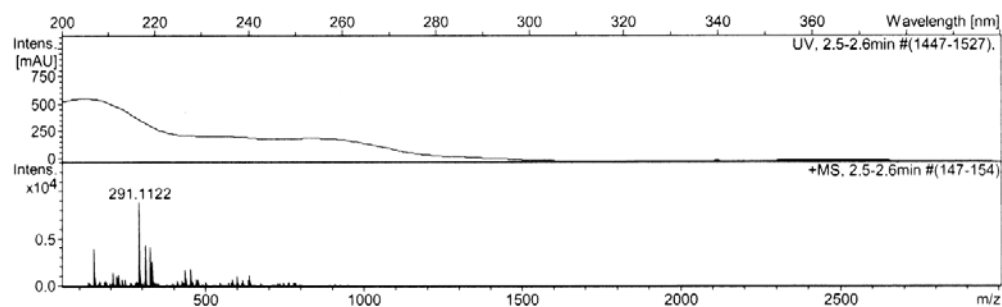
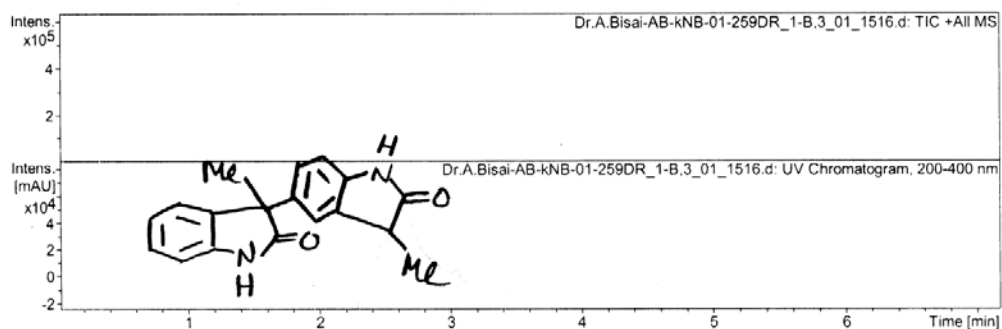
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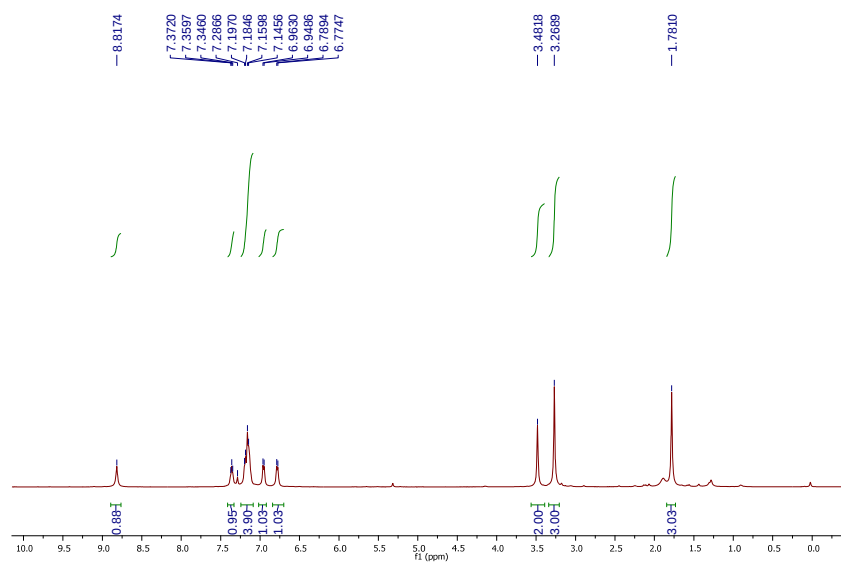
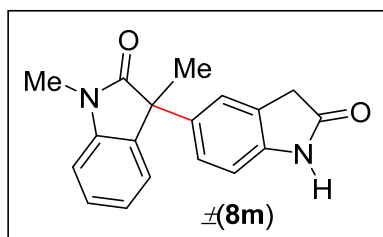
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Method HRLCMS-20 Sept.m
Sample Name Dr.A.Bisai-AB-kNB-01-259DR
Comment
Acquisition Date 1/29/2015 1:35:23 PM
Operator RUCHI
Instrument micrOTOF-Q II 10330

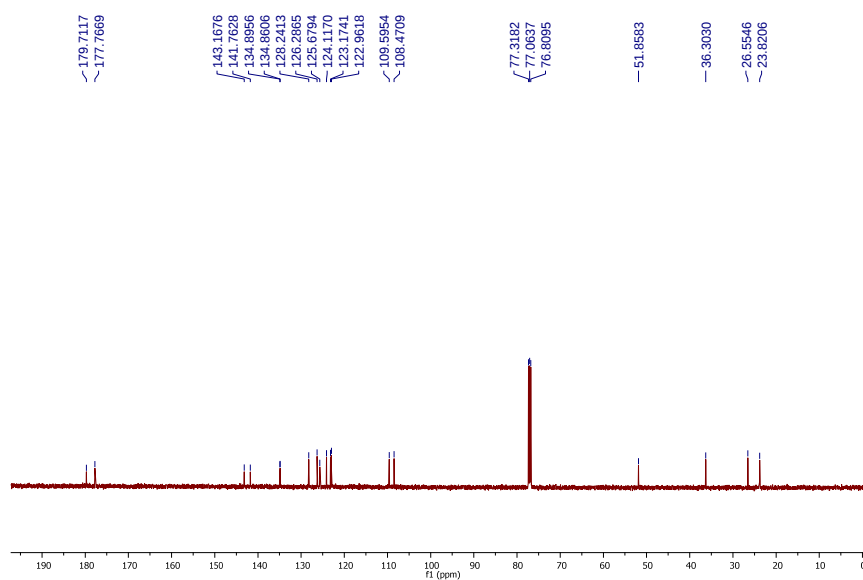
Acquisition Parameter

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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of ($\pm$)-**8l**



¹H NMR (500 MHz, CDCl₃) of compound (±)-**8m**



¹³C NMR (125 MHz, CDCl₃) of compound (±)-**8m**

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\27-JAN-2015\Dr.A Bisai-AB-KNB-01-241-PS_1-B.8_01_1494.d
Method HRLCMS-20 Sept.m
Sample Name Dr.A Bisai-AB-KNB-01-241-PS
Comment

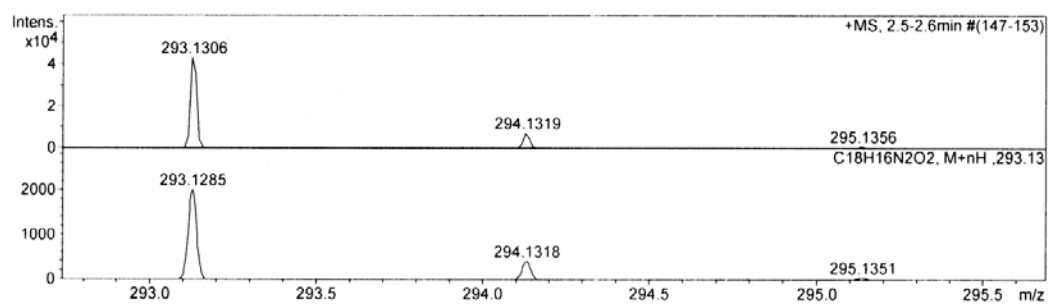
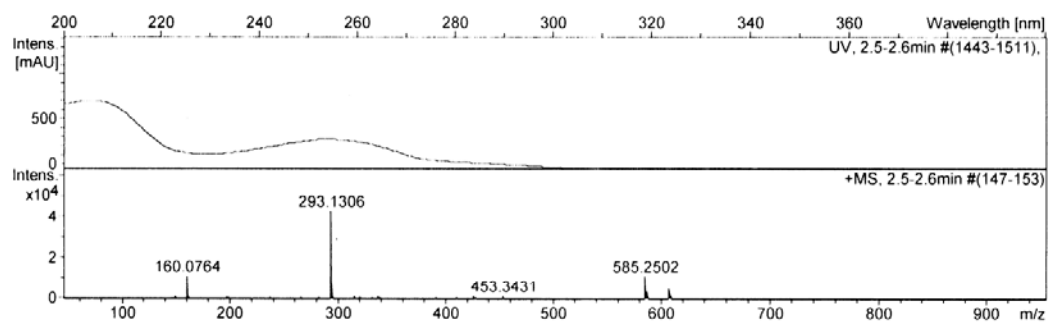
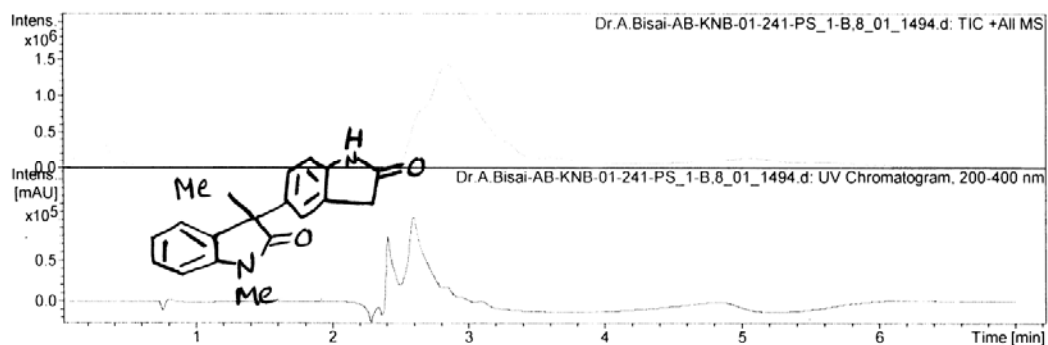
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Operator RUCHI

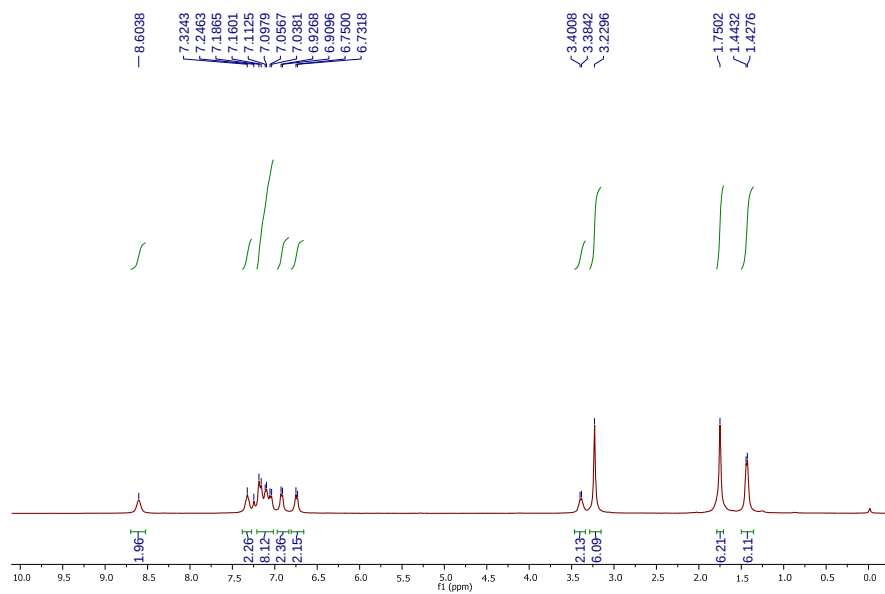
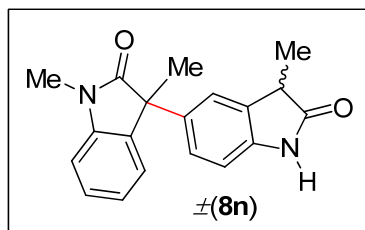
Instrument micrOTOF-Q II 10330

Acquisition Parameter

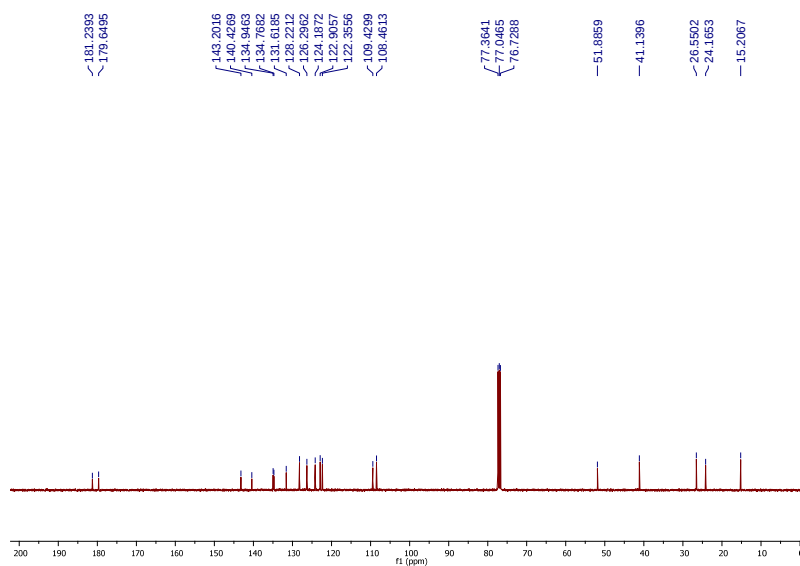
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8m



¹H NMR (400 MHz, CDCl₃) of compound ($\pm$)-**8n**



¹³C NMR (125 MHz, CDCl₃) of compound ($\pm$)-**8n**

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\29-JAN-2015\Dr.A.Bisai-AB-kNB-01-265-DR_1-B,4_01_1517.d
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 Sample Name Dr.A.Bisai-AB-kNB-01-265-DR
 Comment

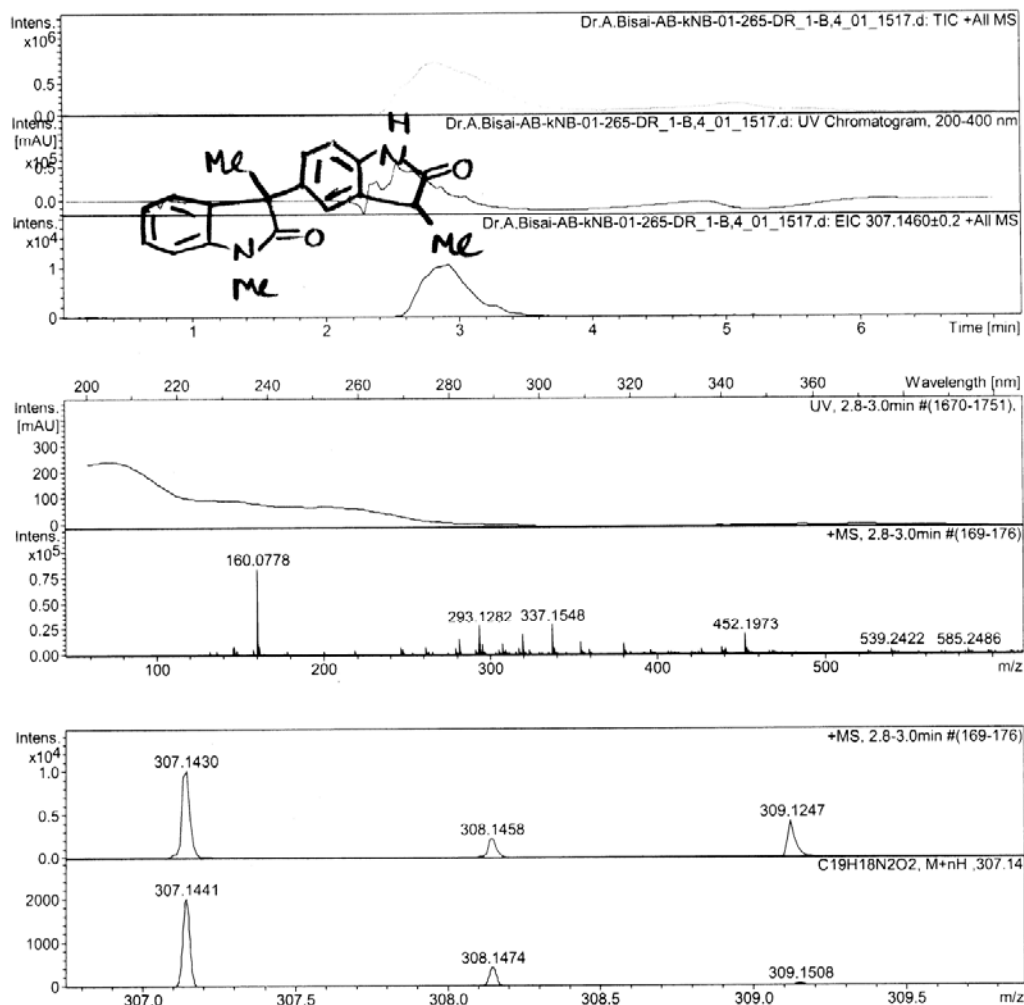
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Operator RUCHI

Instrument microTOF-Q II 10330

Acquisition Parameter

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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

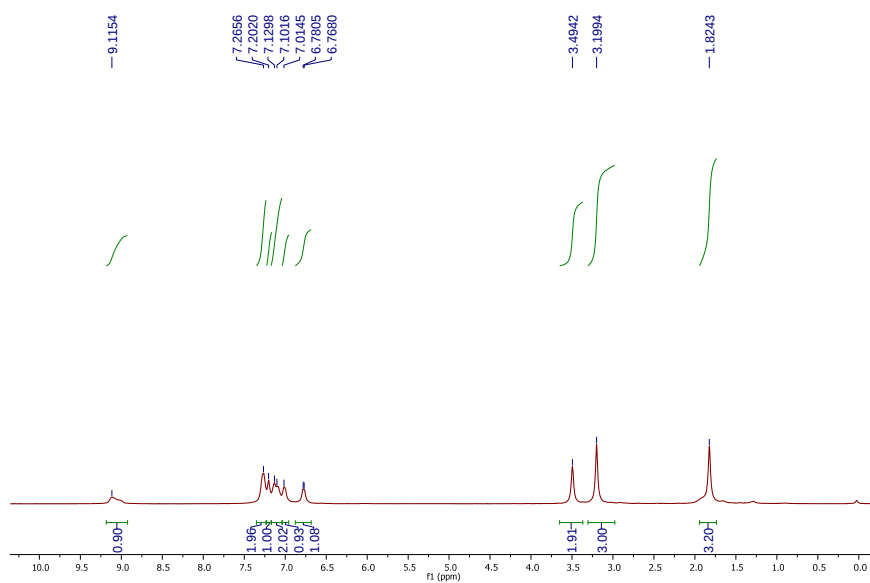
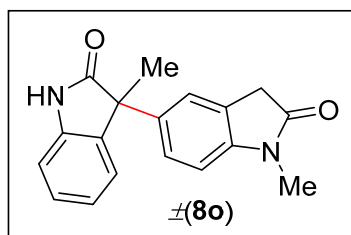


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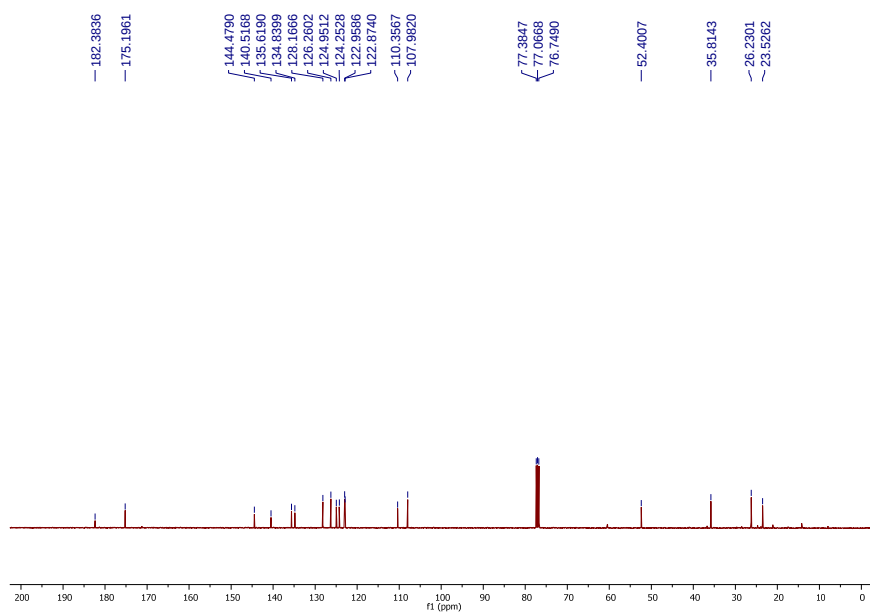
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Page 1 of 1

Scanned copy of mass spectrum of (±)-8n



¹H NMR (400 MHz, CDCl₃) of compound (±)-8o



¹³C NMR (100 MHz, CDCl₃) of compound (±)-8o

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\29-JAN-2015\Dr.A.Bisai-AB-kNB-01-309-R_1-B_2_01_1515.d
Method HRLCMS-20 Sept.m
Sample Name Dr.A.Bisai-AB-kNB-01-309-R
Comment

Acquisition Date 1/29/2015 1:27:12 PM

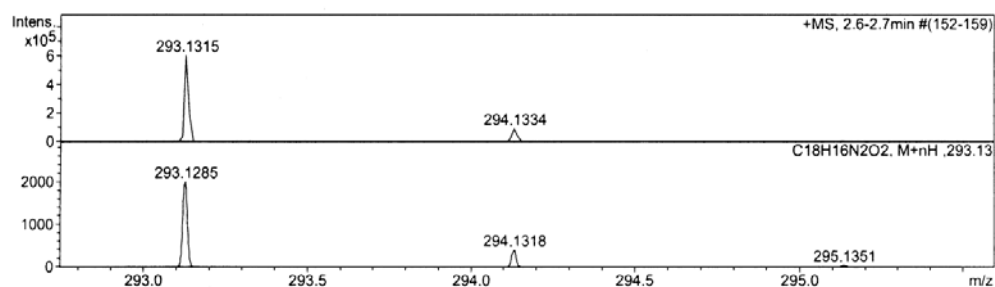
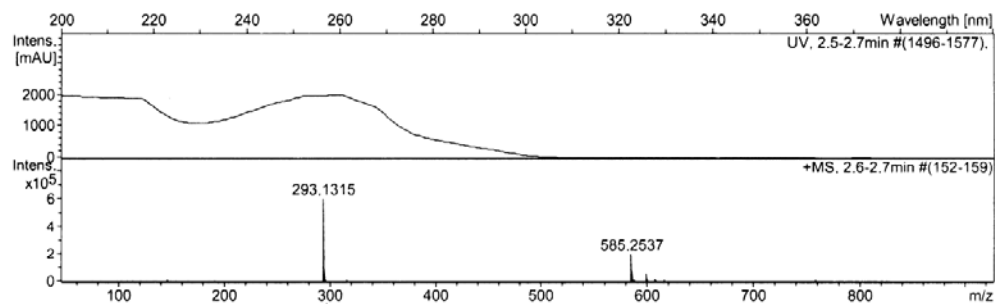
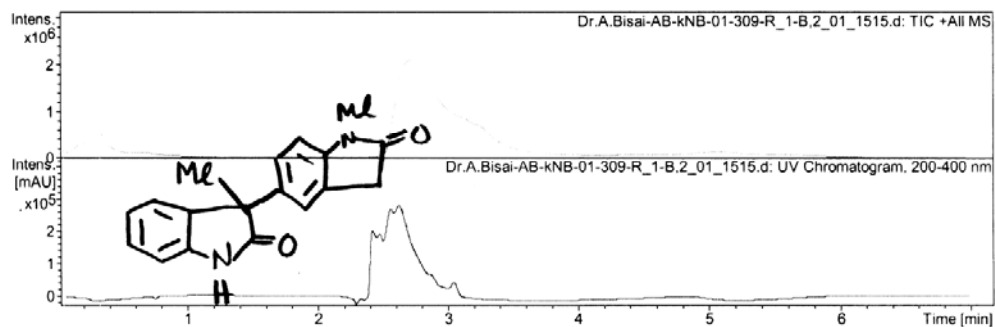
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Operator RUCHI

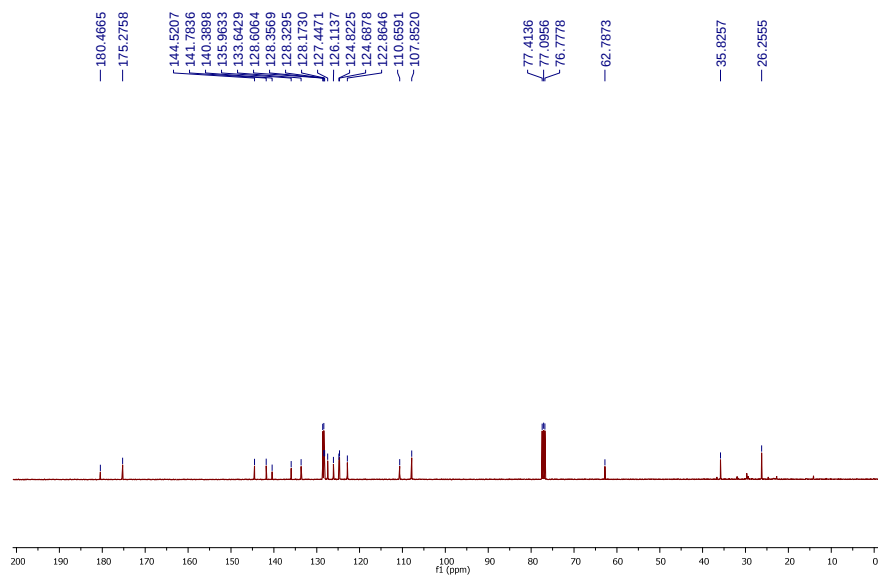
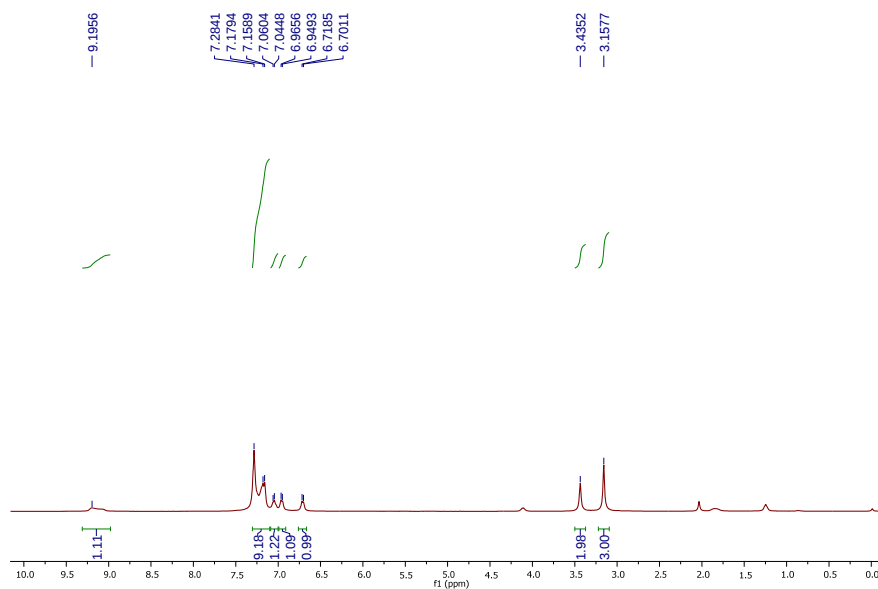
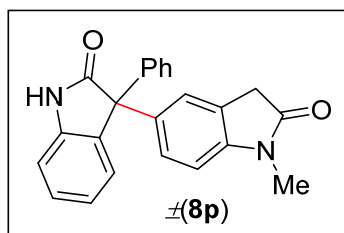
Instrument microTOF-Q II 10330

Acquisition Parameter

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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8o



Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\29-JAN-2015\Dr.A.Bisai-AB-Lknb-01-316_1-A,5_01_1509.d
 Method HRLCMS-20 Sept.m
 Sample Name Dr.A.Bisai-AB-Lknb-01-316
 Comment

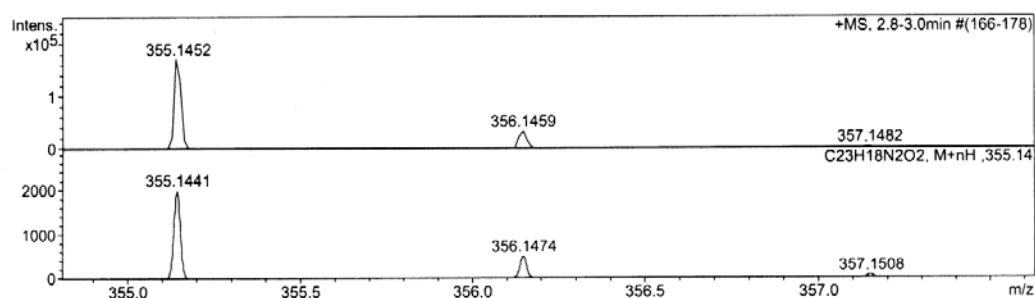
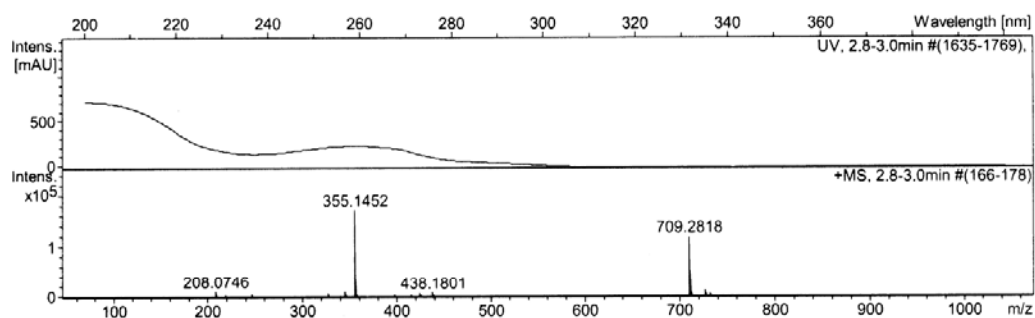
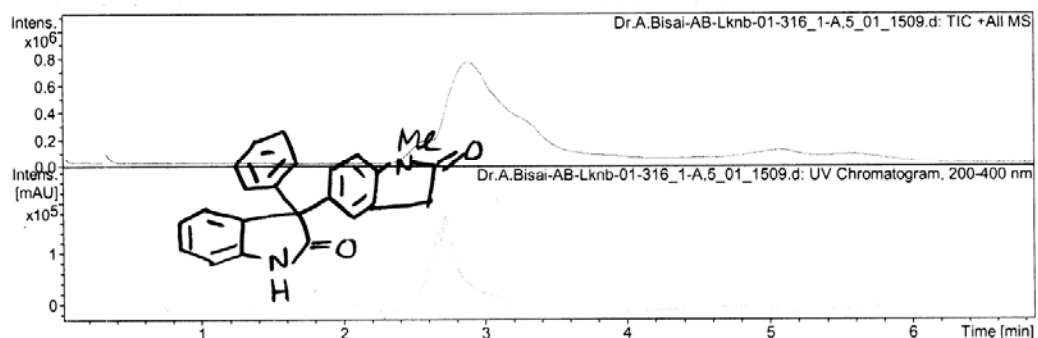
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Operator RUCHI

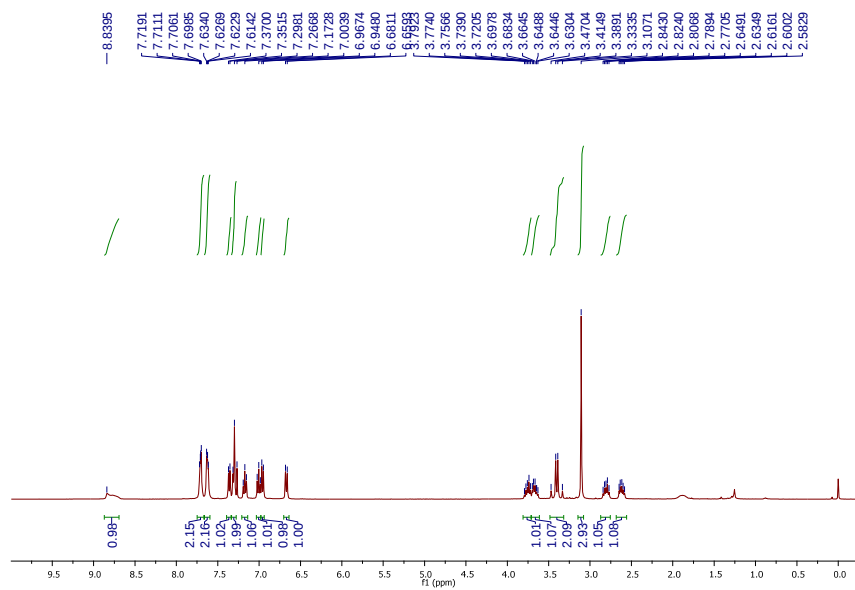
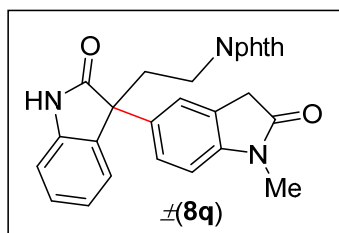
Instrument micrOTOF-Q II 10330

Acquisition Parameter

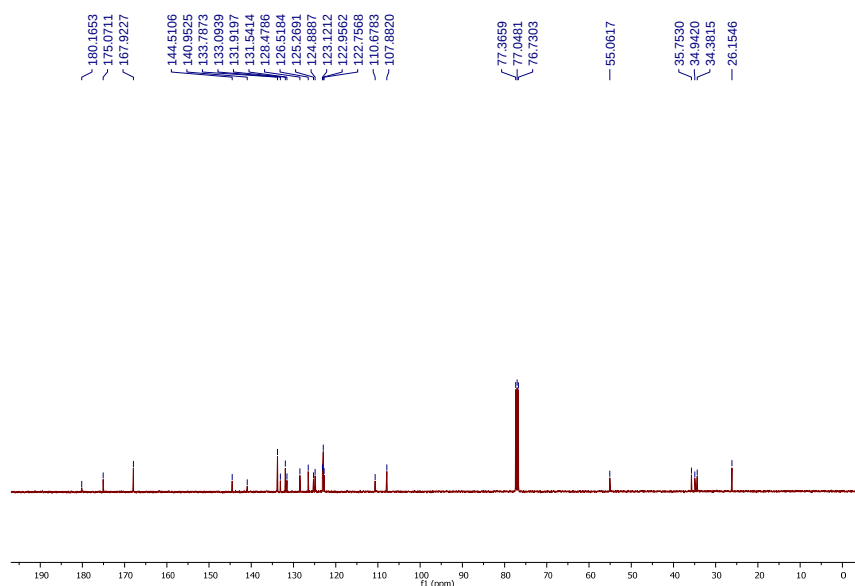
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8p



¹H NMR (400 MHz, CDCl₃) of compound (±)-8q



¹³C NMR (100 MHz, CDCl₃) of compound (±)-8q

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\29-JAN-2015\Dr.A.Bisai-AB-knb-01-318_1-A.6_01_1510.d
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 Sample Name Dr.A.Bisai-AB-knb-01-318
 Comment

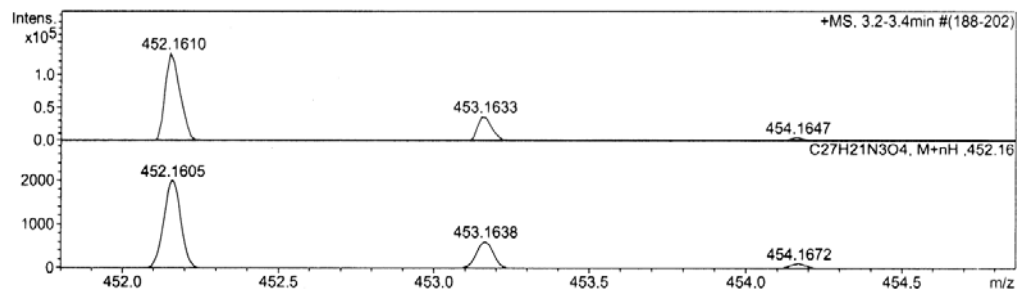
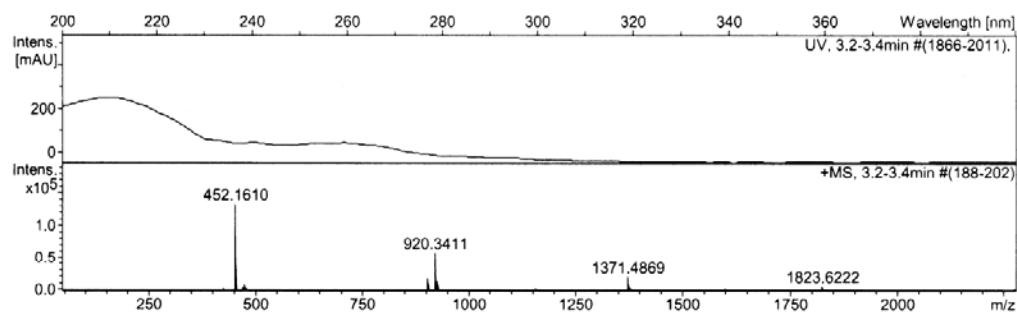
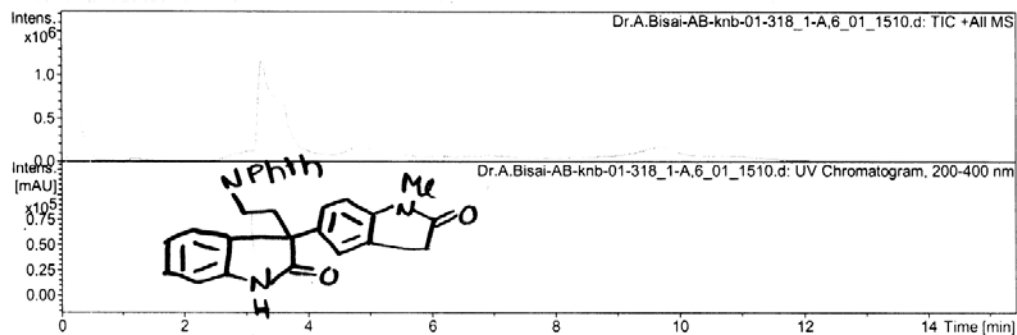
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Operator RUCHI

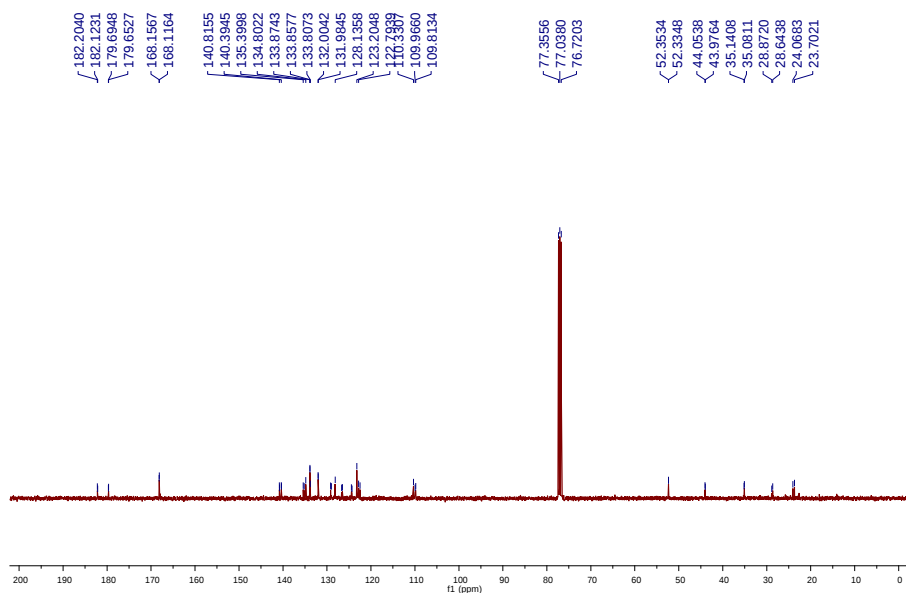
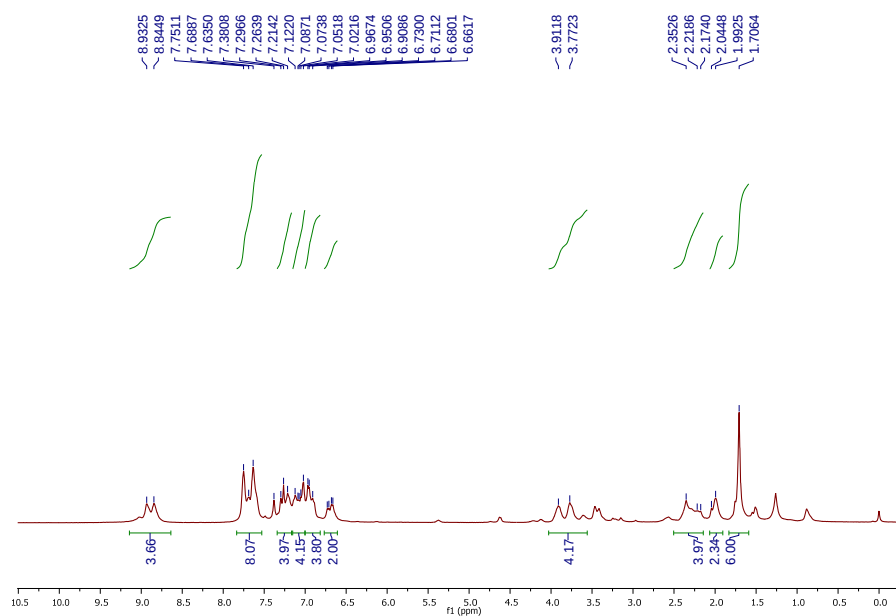
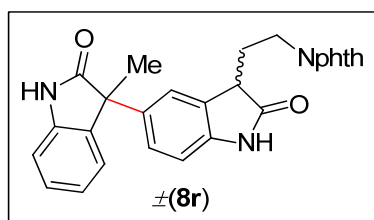
Instrument micrOTOF-Q II 10330

Acquisition Parameter

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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8q



Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\29-JAN-2015\Dr.A.Bisai-AB-KNB-01-285_1-B,1_01_1514.d
Method HRLCMS-20 Sept tune wide.m
Sample Name Dr.A.Bisai-AB-KNB-01-285
Comment

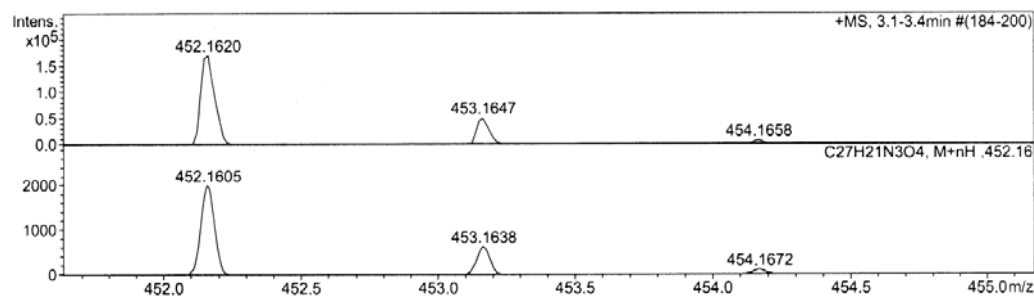
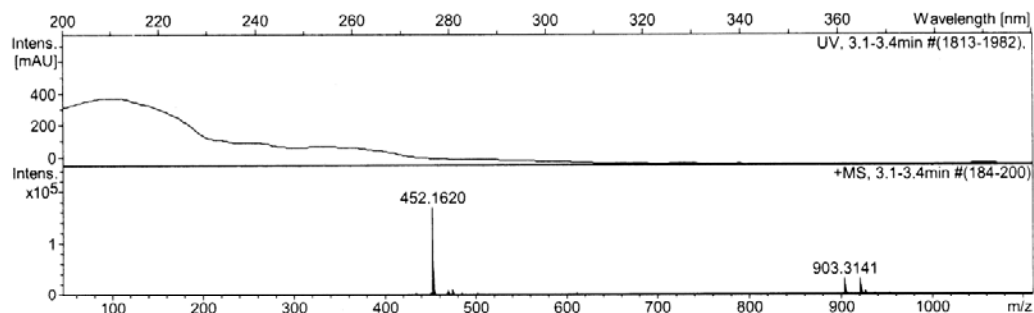
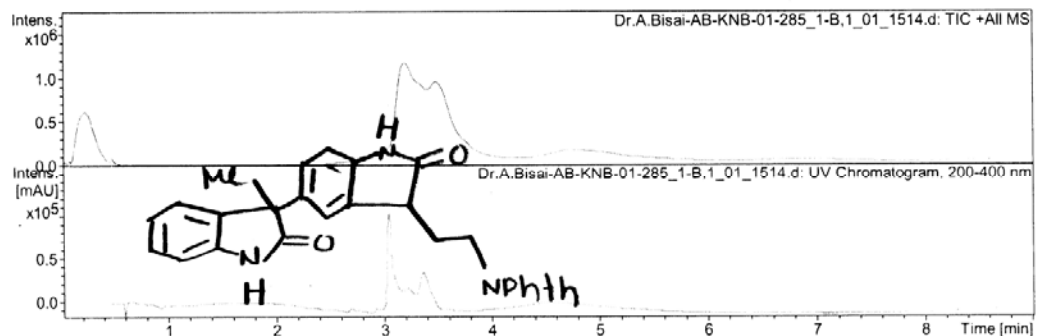
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Operator RUCHI

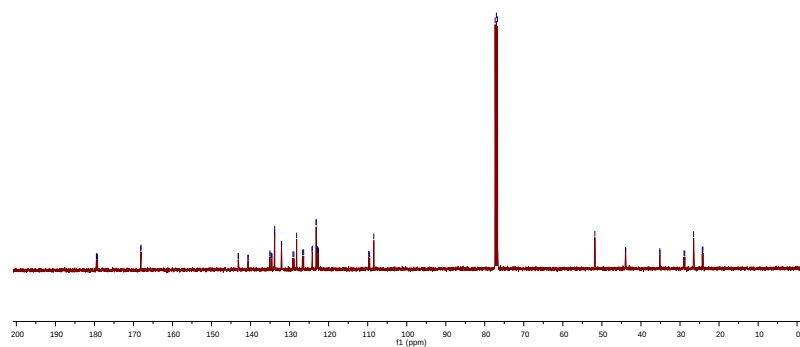
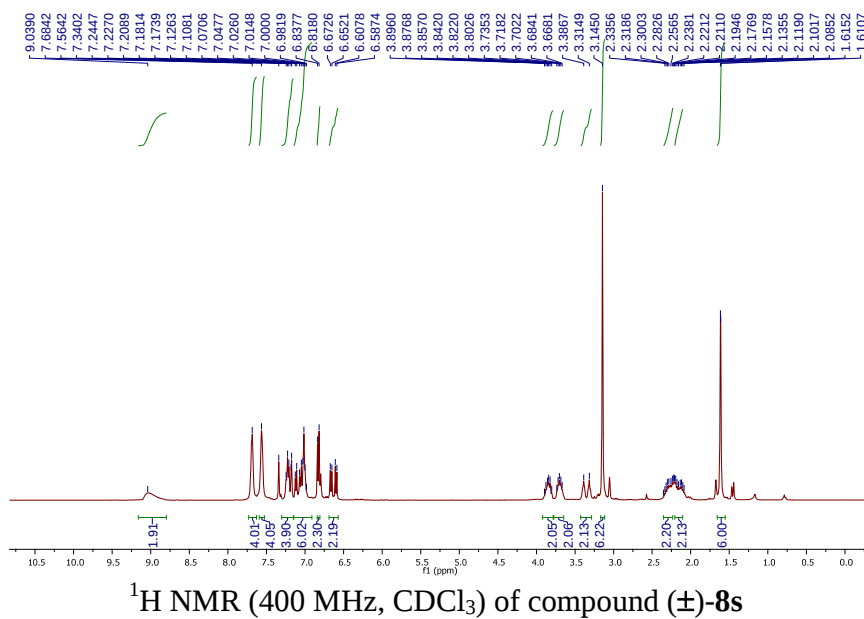
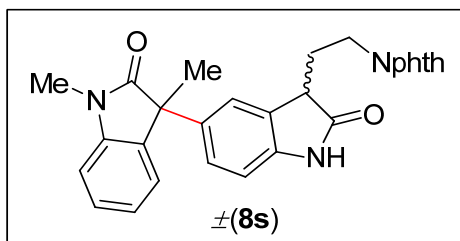
Instrument micrOTOF-Q II 10330

Acquisition Parameter

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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8r



Display Report

Analysis Info

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Sample Name AB-KNB-01-278
Comment

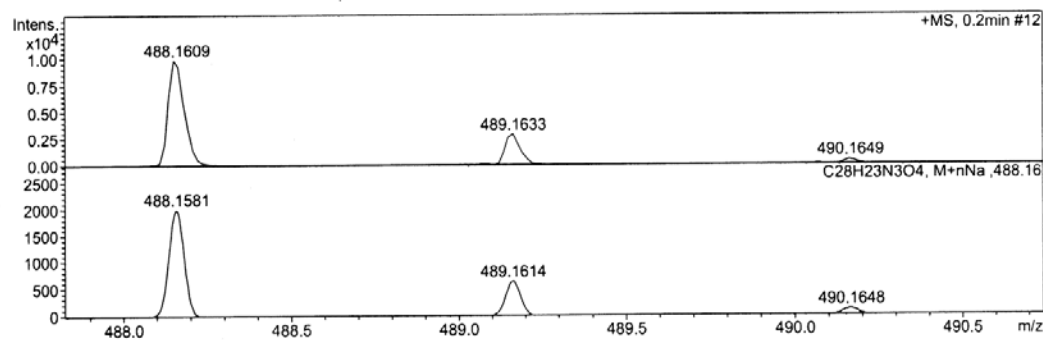
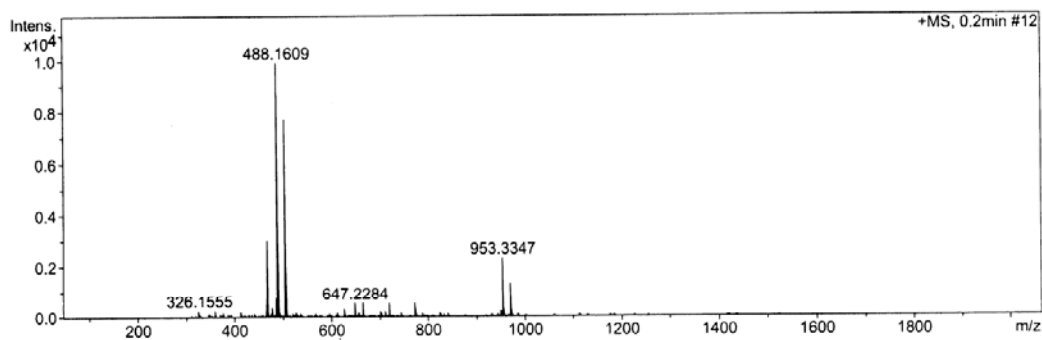
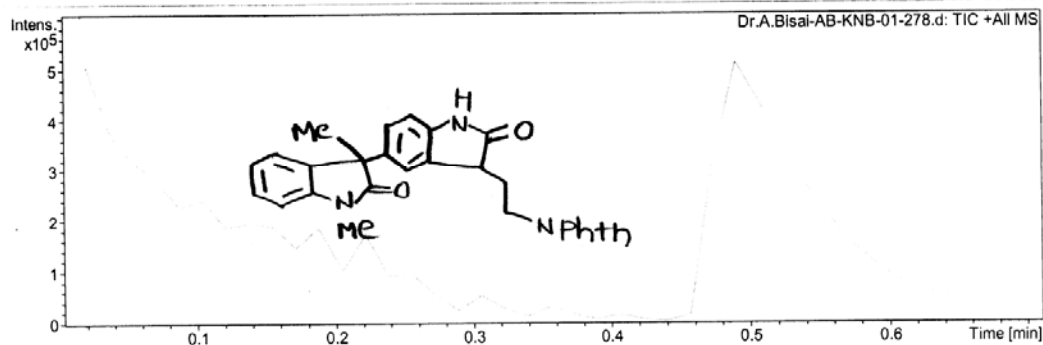
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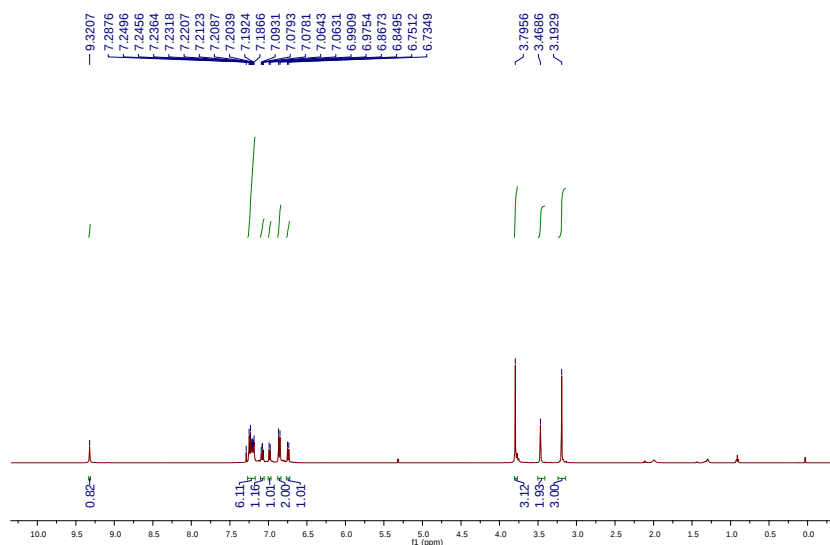
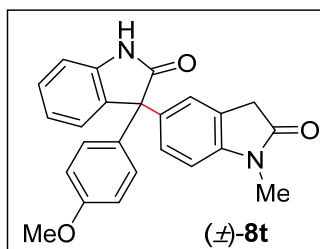
Operator RUCHI

Instrument micrOTOF-Q II 10330

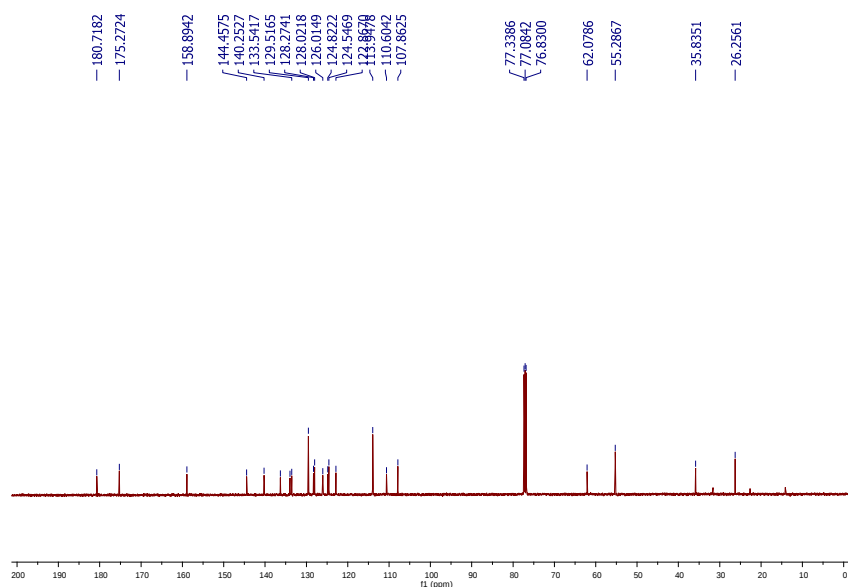
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Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste

Scanned copy of mass spectrum of ($\pm$)-8s



¹H NMR (400 MHz, CDCl₃) of compound (±)-8t



¹³C NMR (100 MHz, CDCl₃) of compound (±)-8t

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\27-JAN-2015\Dr.A Bisai-AB-LK-04-268_1-B.6_01_1491.d
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 Sample Name Dr.A.Bisai-AB-LK-04-268
 Comment

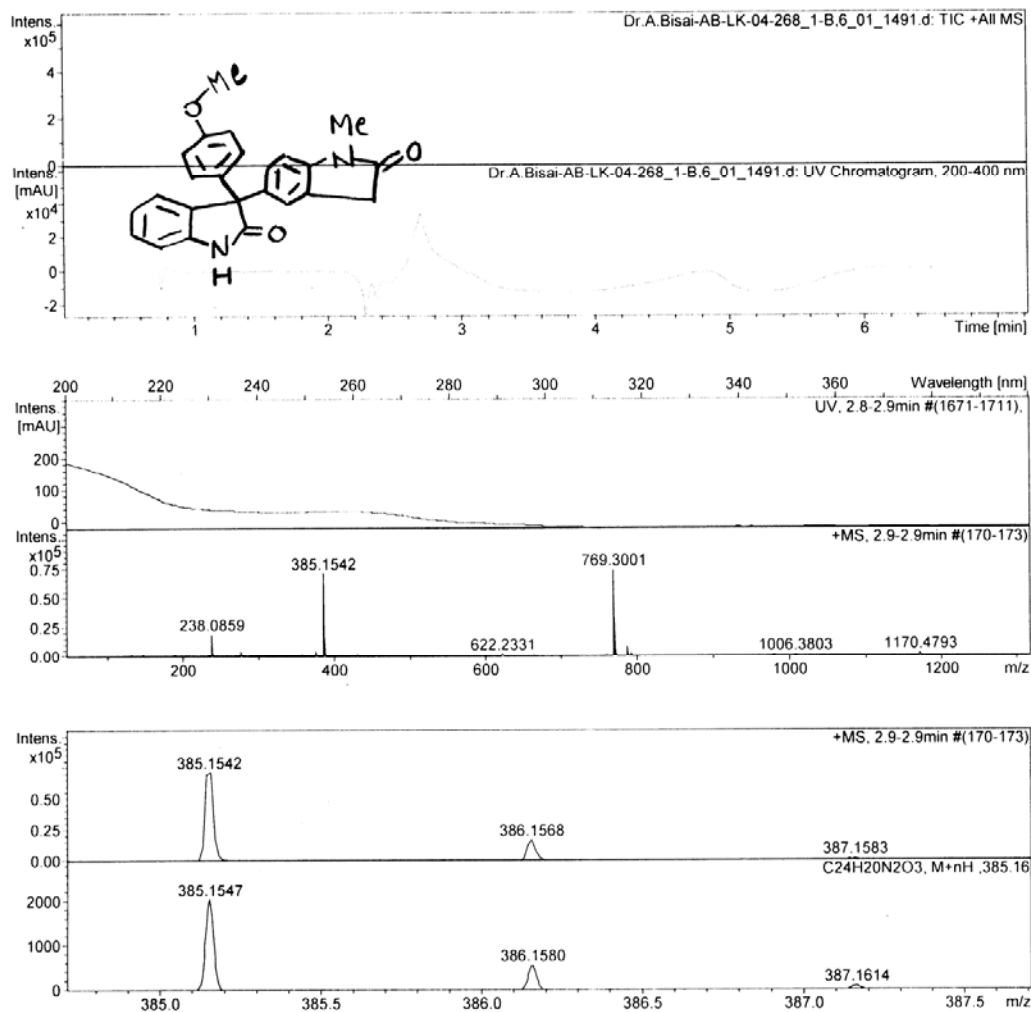
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Operator RUCHI

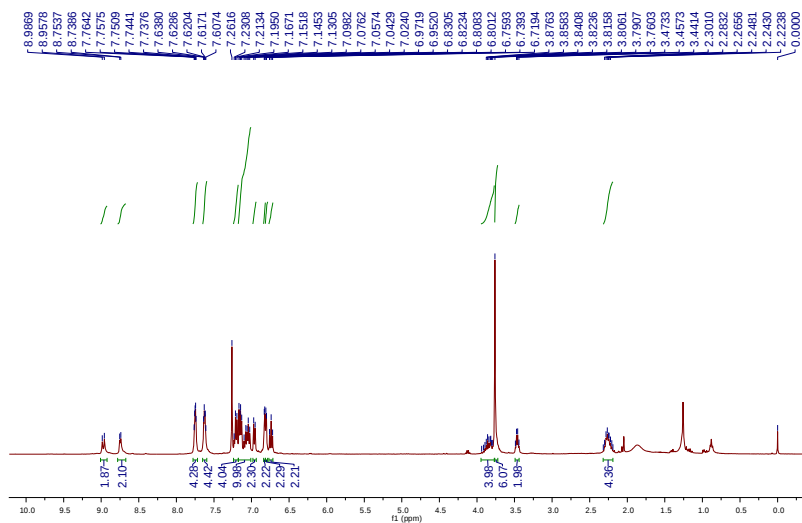
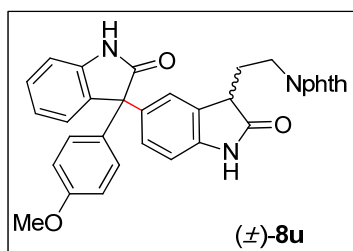
Instrument micrOTOF-Q II 10330

Acquisition Parameter

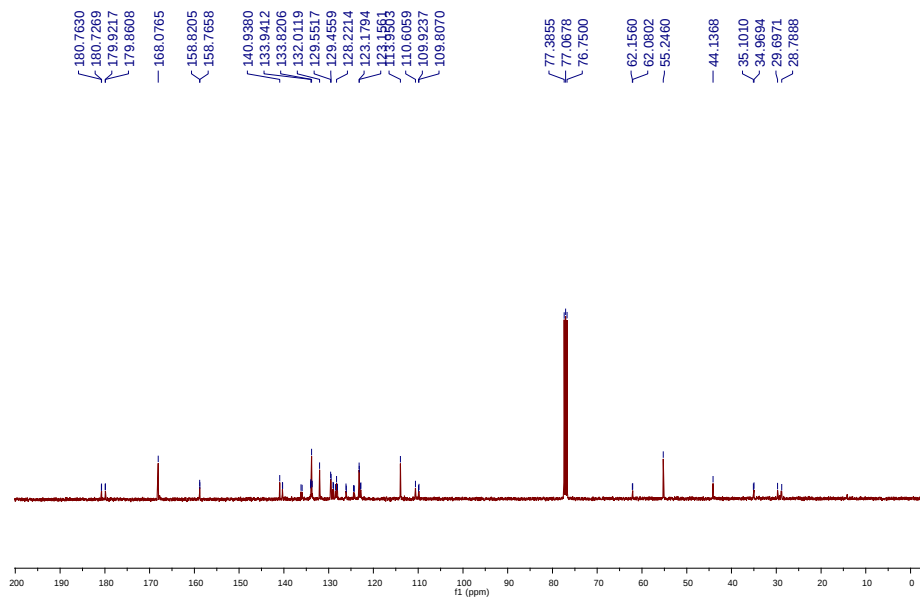
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Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8t



¹H NMR (400 MHz, CDCl₃) compound (±)-**8u**



¹³C NMR (100 MHz, CDCl₃) compound (±)-**8u**

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\29-JAN-2015\Dr.A.Bisai-AB-knb-01-252-R_1-A,8_01_1512.d
 Method HRLCMS-20 Sept tune wide.m
 Sample Name Dr.A.Bisai-AB-knb-01-252-R
 Comment

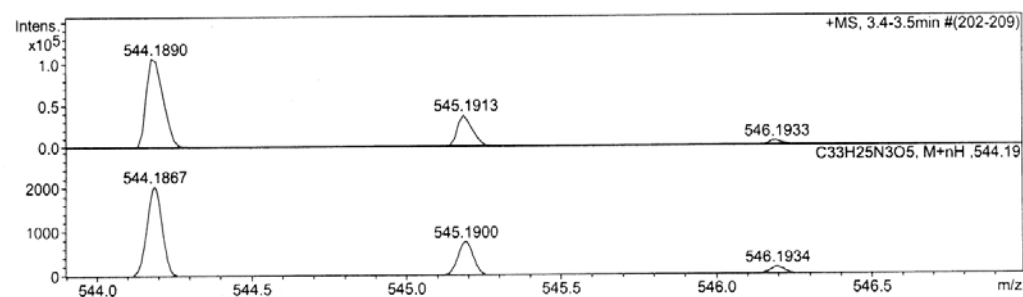
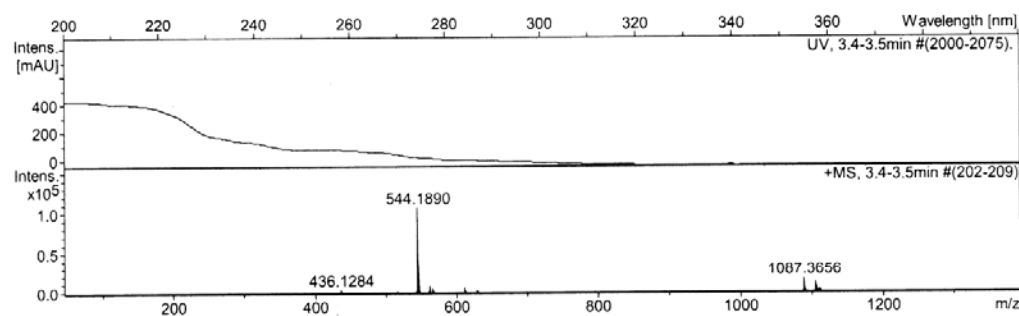
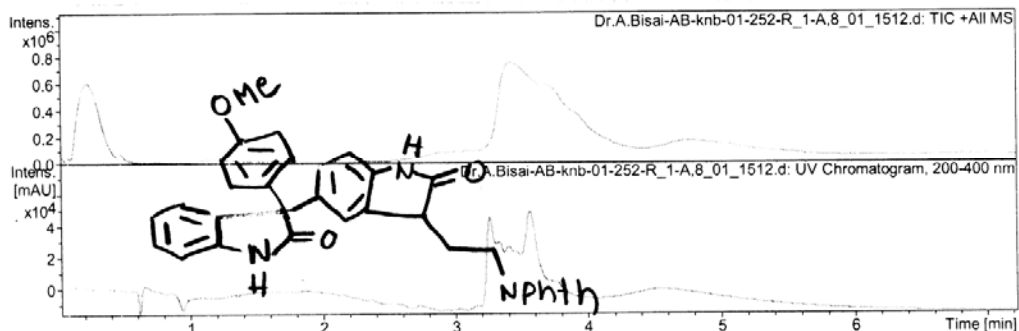
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Operator RUCHI

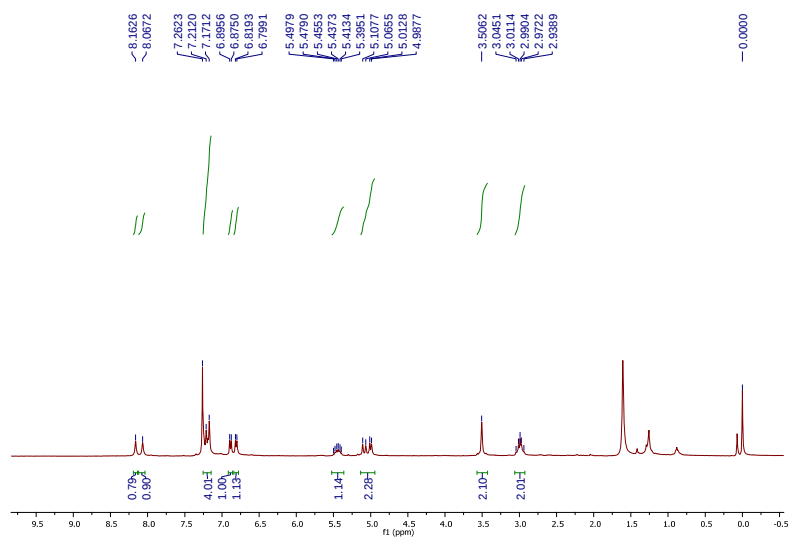
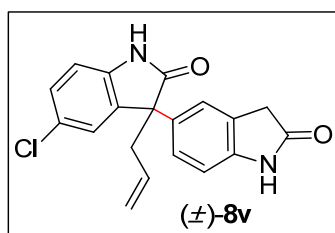
Instrument micrOTOF-Q II 10330

Acquisition Parameter

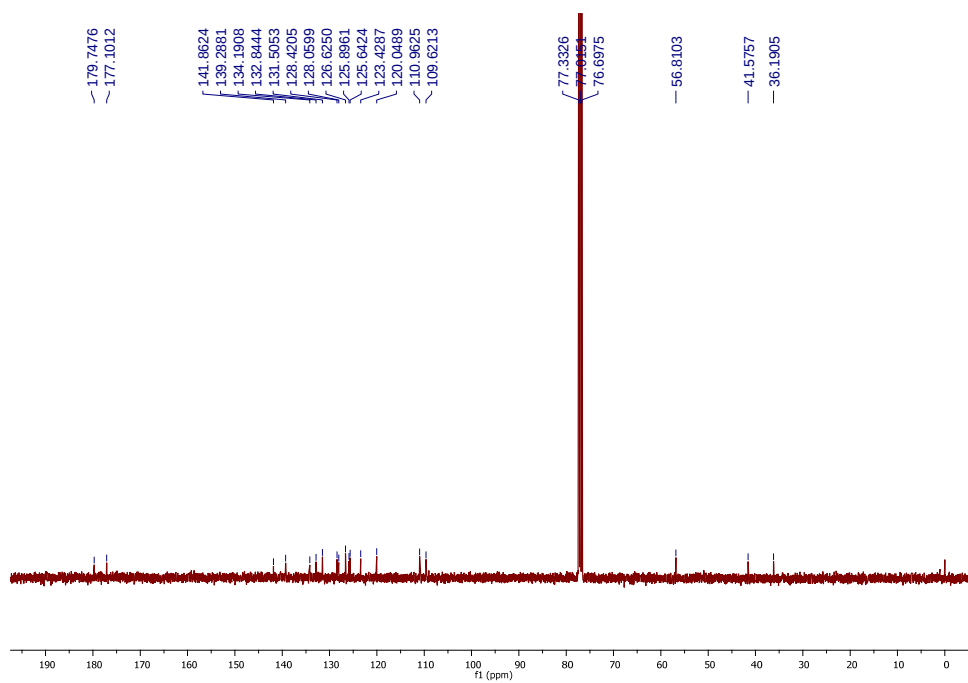
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Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8u



¹H NMR (400 MHz, CDCl₃) compound (±)-**8v**



¹³C NMR (100 MHz, CDCl₃) compound (±)-**8v**

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\27-JAN-2015\Dr.A Bisai-AB-KNB-01-226_1-C_2_01_1496.d
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 Sample Name Dr.A Bisai-AB-KNB-01-226
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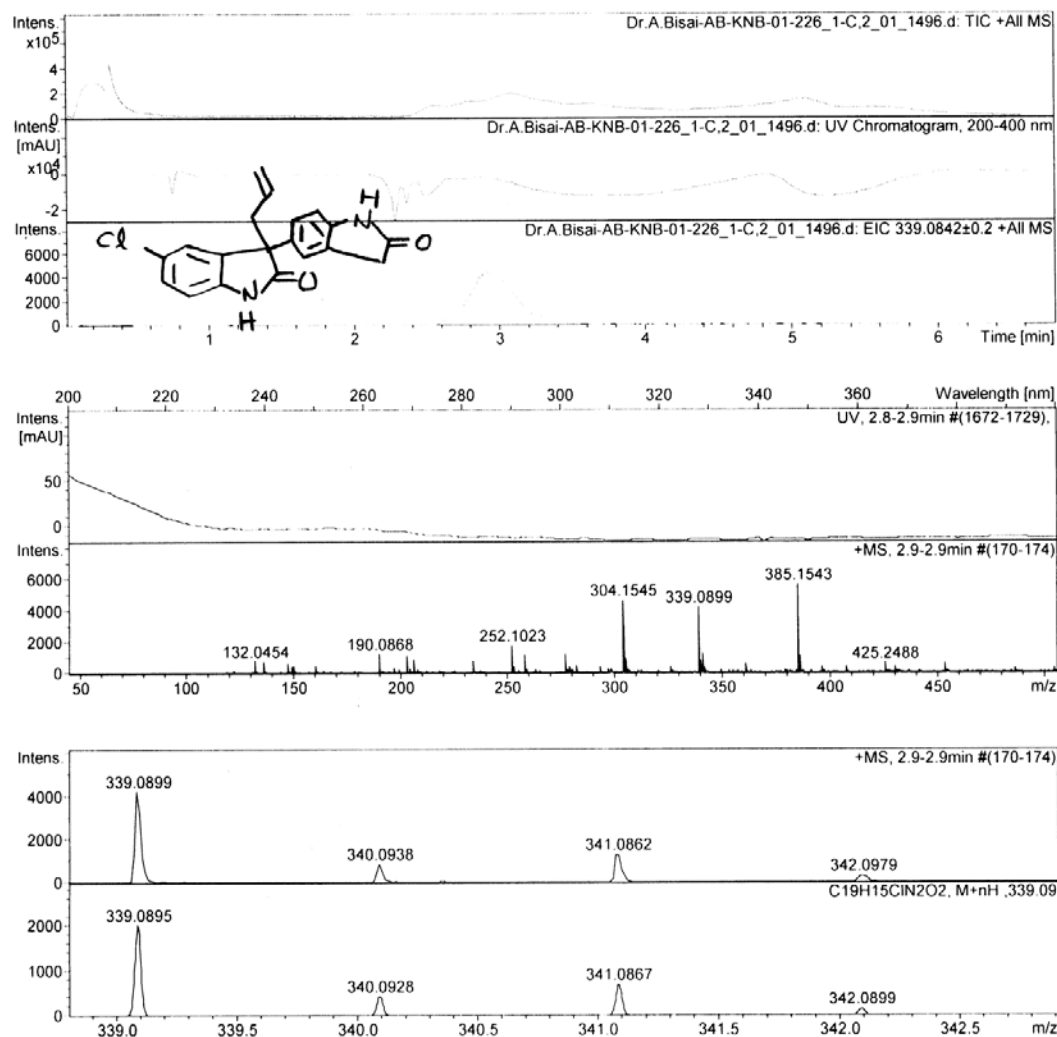
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Operator RUCHI

Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste

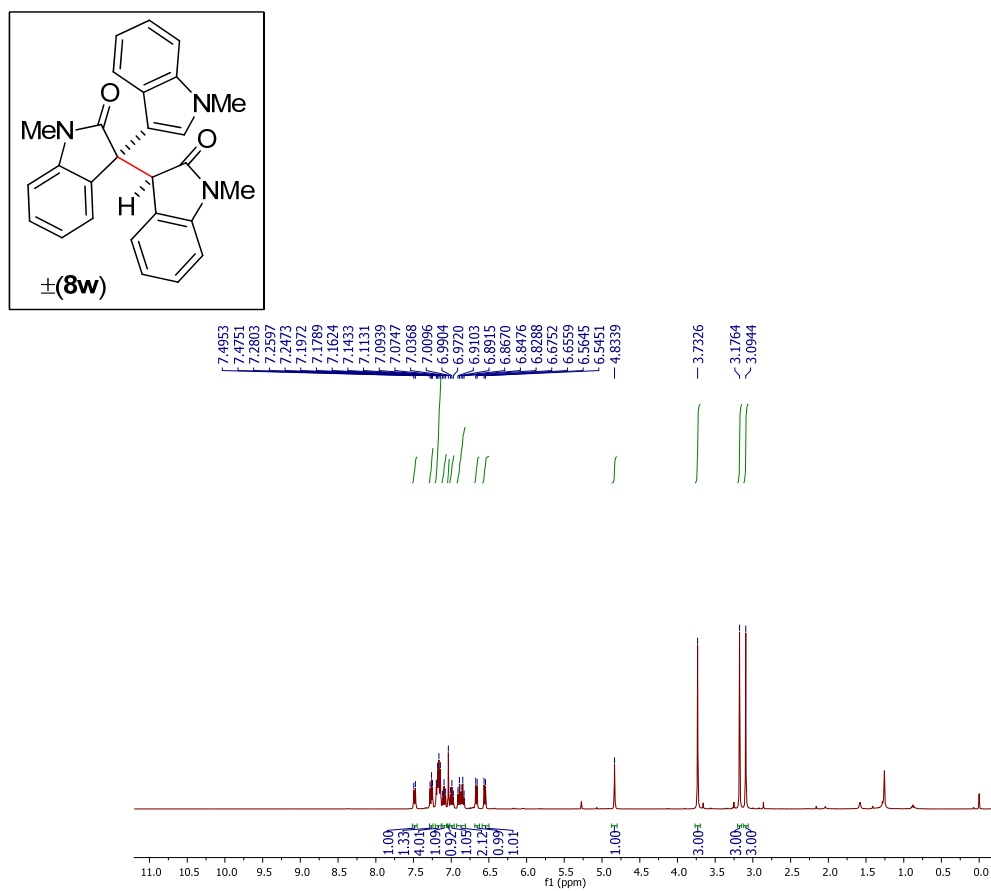


Bruker Compass DataAnalysis 4.0

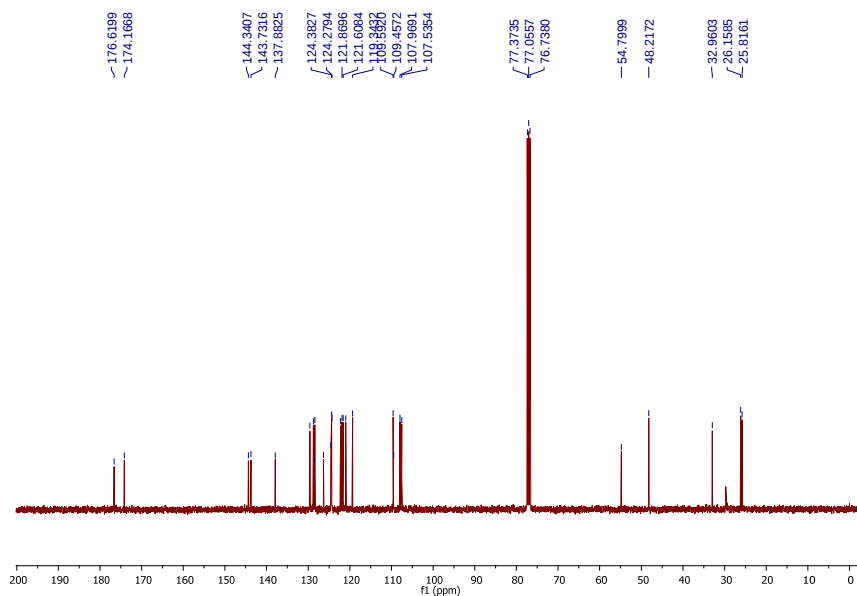
printed: 1/27/2015 3:37:43 PM

Page 1 of 1

Scanned copy of mass spectrum of (±)-8v



^1H NMR (400 MHz, CDCl_3) of compound ($\pm$)-8w (Major diastereomer)



^{13}C NMR (100 MHz, CDCl_3) of compound ($\pm$)-8w (Major diastereomer)

Display Report

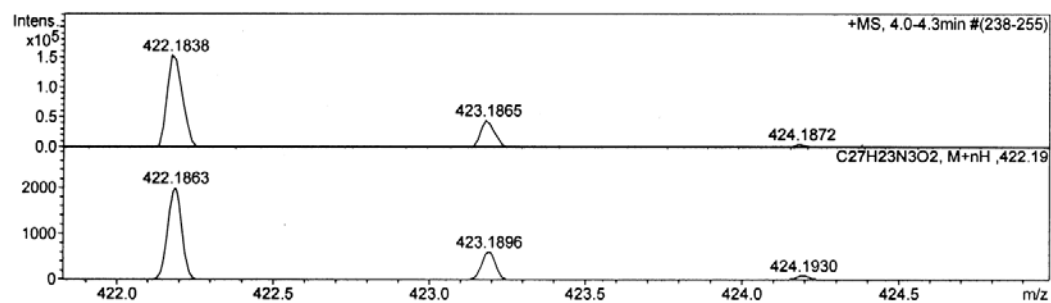
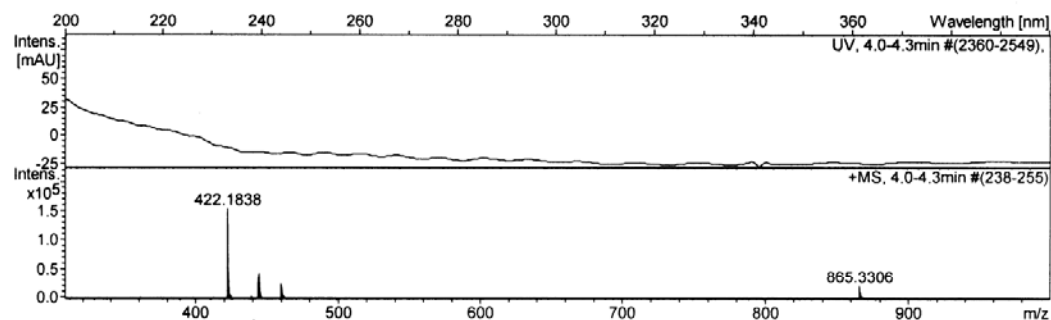
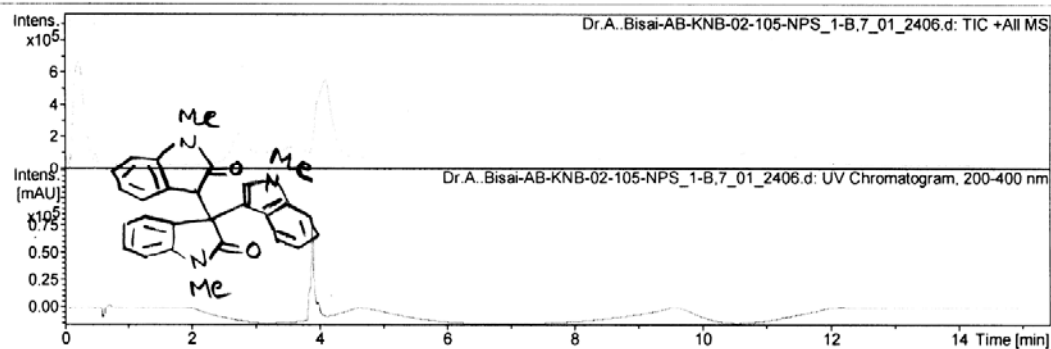
Analysis Info

Analysis Name D:\Data\user data\2015\MAY-2015\07-MAY-2015\Dr.A..Bisai-AB-KNB-02-105-NPS_1-B,7_01_2406.d
 Method HRLCMS-20 Sept tune wide.m
 Sample Name Dr.A..Bisai-AB-KNB-02-105-NPS
 Comment

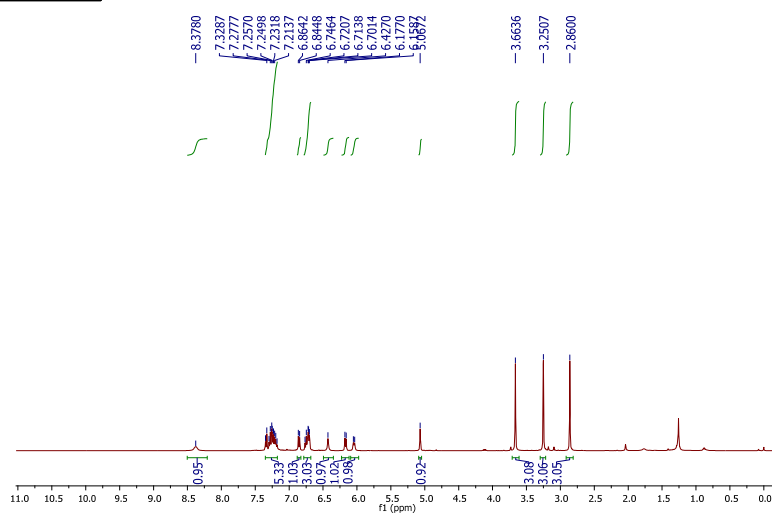
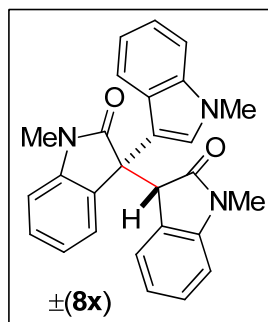
Acquisition Date 5/7/2015 1:50:25 PM
 Operator RUCHI
 Instrument micrOTOF-Q II 10330

Acquisition Parameter

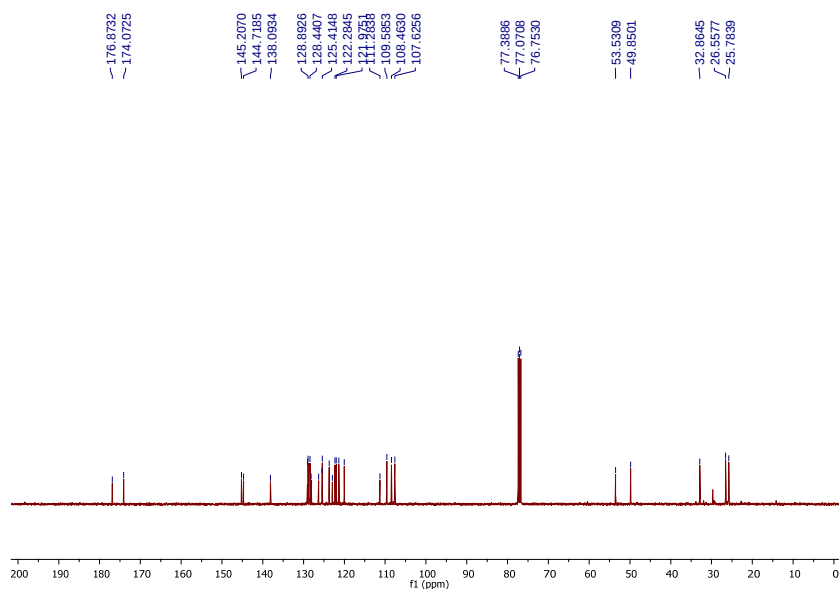
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Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8w (Major diastereomer)



^1H NMR (400 MHz, CDCl_3) of compound $(\pm)\text{-}8x$ (Minor diastereomer)



^{13}C NMR (100 MHz, CDCl_3) of compound $(\pm)\text{-}8x$ (Minor diastereomer)

Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\MAY-2015\07-MAY-2015\Dr.A..Bisai-AB-KNB-02-105-MSR_1-B,8_01_2407.d
 Method HRLCMS-20 Sept tune wide.m
 Sample Name Dr.A..Bisai-AB-KNB-02-105-MSR
 Comment

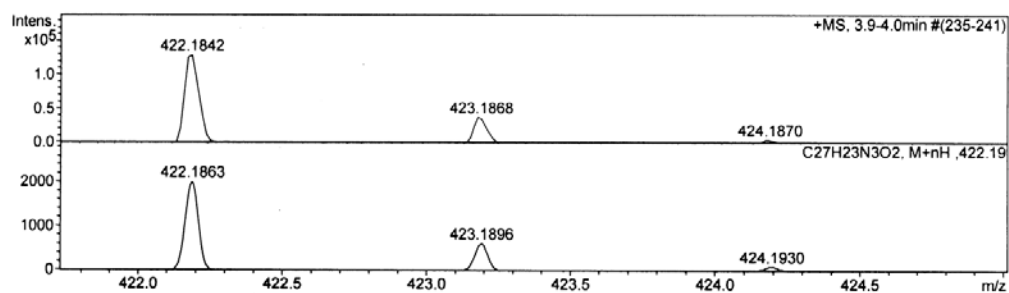
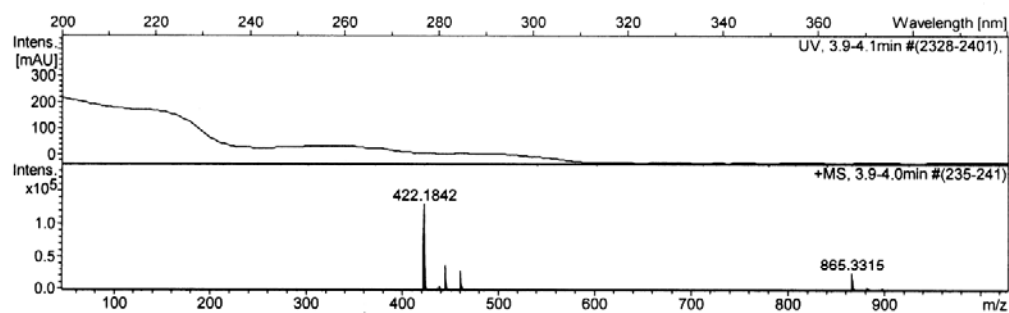
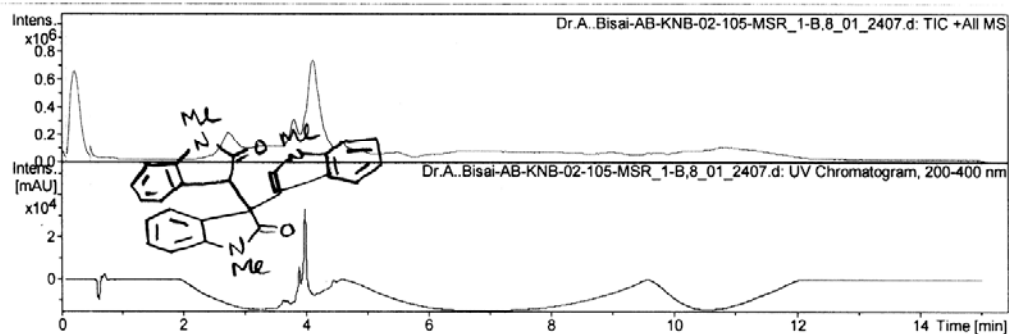
Acquisition Date 5/7/2015 2:06:36 PM

Operator RUCHI

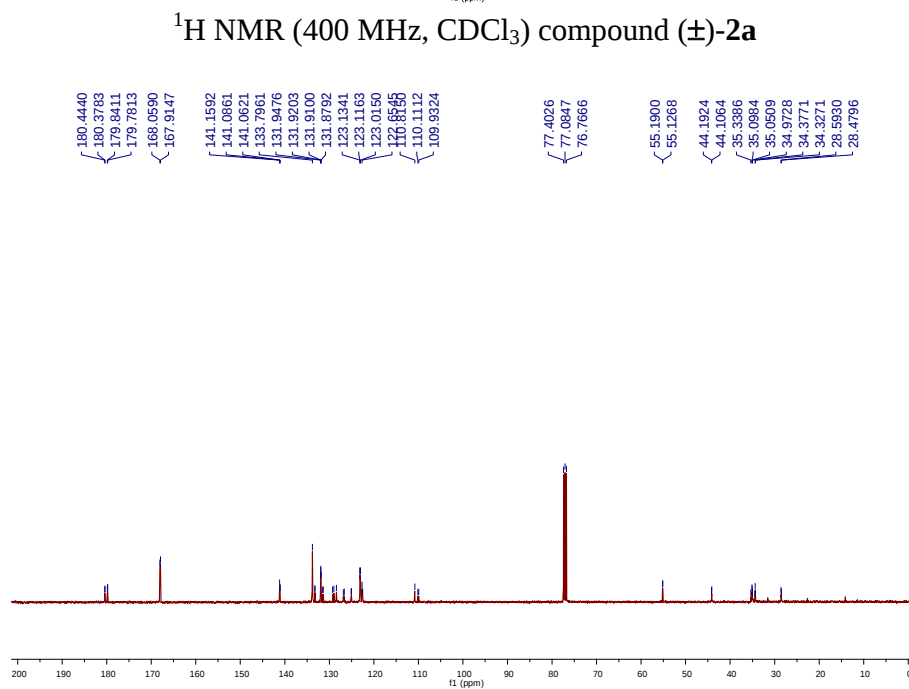
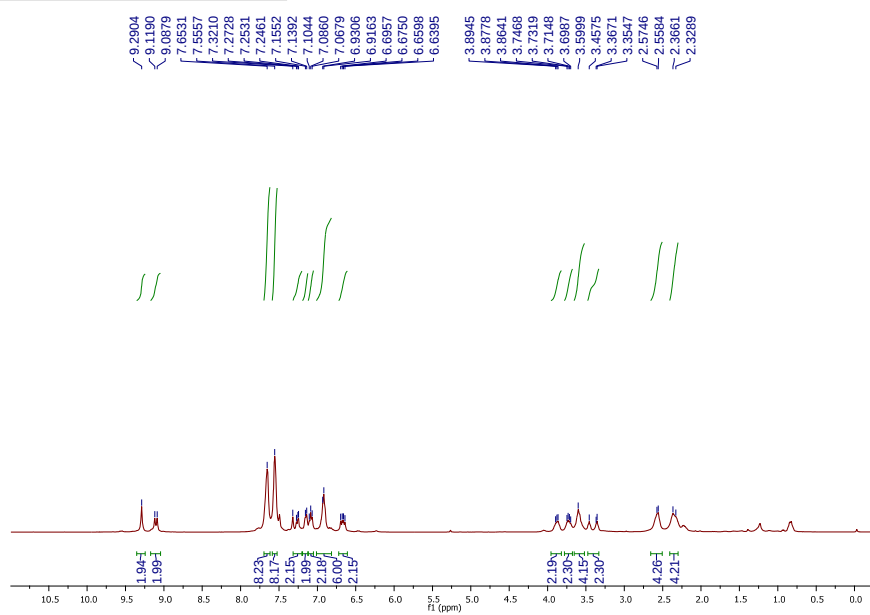
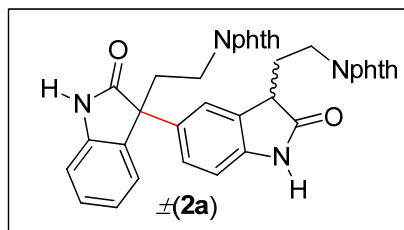
Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type	ESI	Ion Polarity	Positive	Set Nebulizer	1.2 Bar
Focus	Not active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	600.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-8x (Minor diastereomer)



Display Report

Analysis Info

Analysis Name D:\Data\user data\2015\JAN-2015\27-JAN-2015\Dr.A.Bisai-AB-LK-04-269_1-B,5_01_1490.d
 Method HRLCMS-20 Sept.m
 Sample Name Dr.A.Bisai-AB-LK-04-269
 Comment

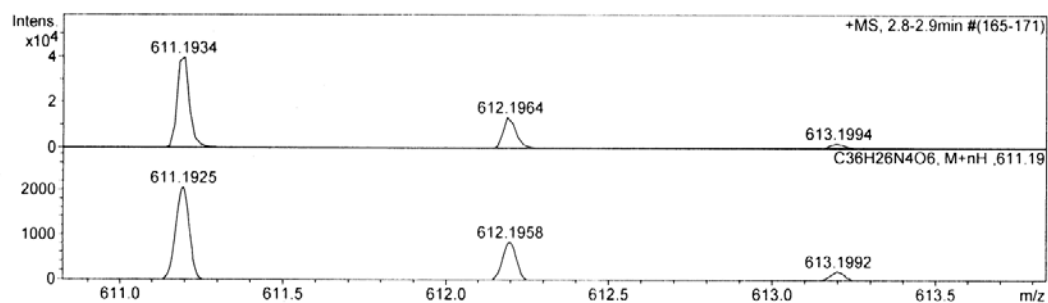
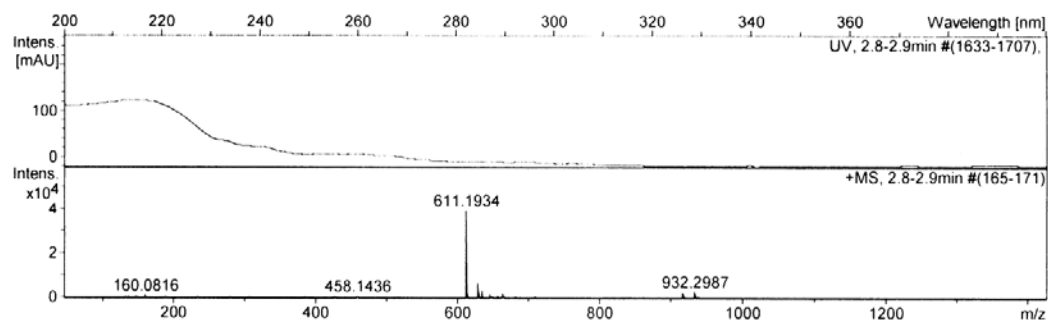
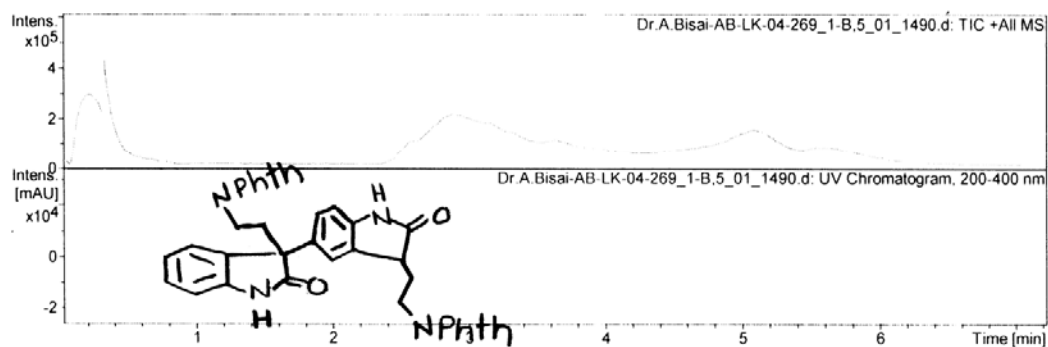
Acquisition Date 1/27/2015 1:52:44 PM

Operator RUCHI

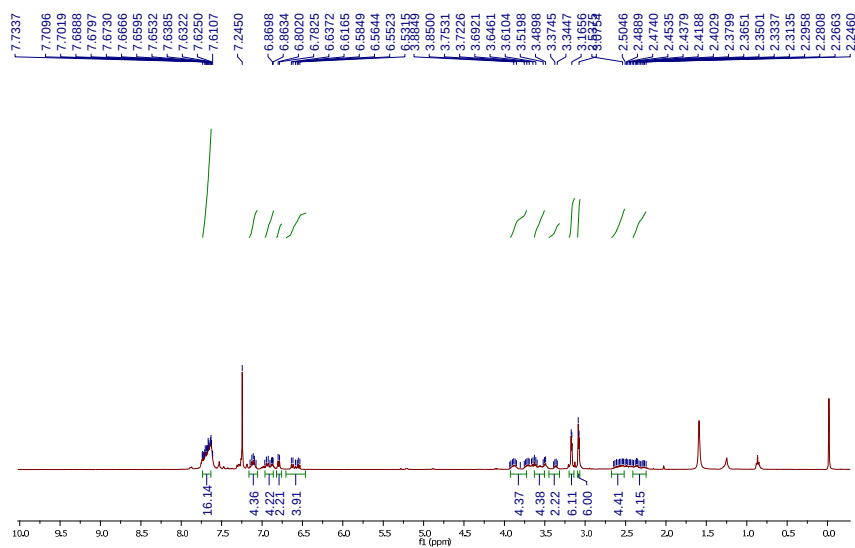
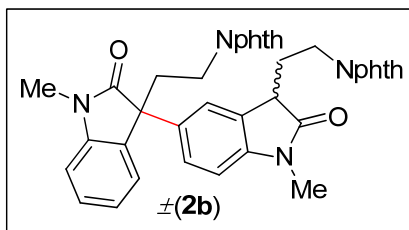
Instrument micrOTOF-Q II 10330

Acquisition Parameter

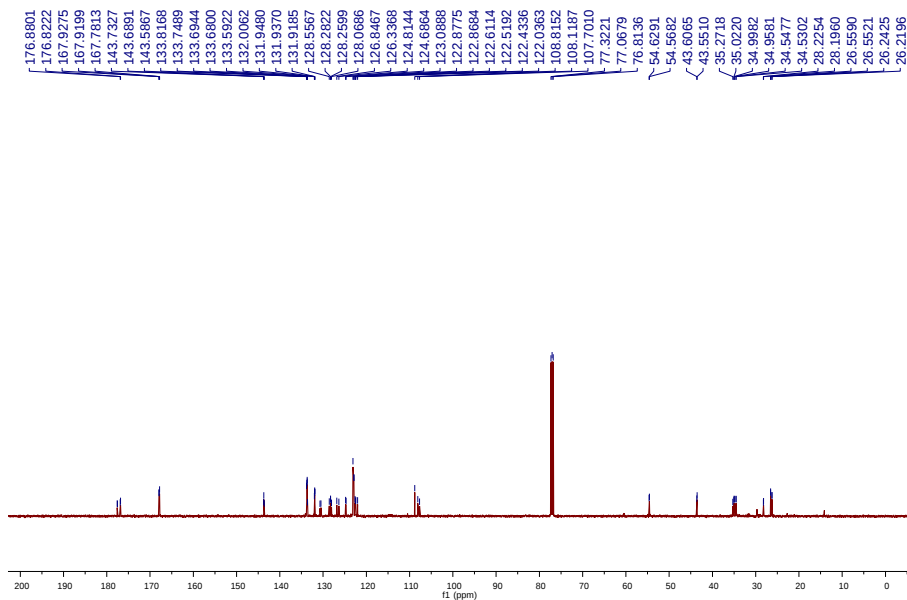
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Focus	Active	Set Capillary	4500 V	Set Dry Heater	200 °C
Scan Begin	50 m/z	Set End Plate Offset	-500 V	Set Dry Gas	7.0 l/min
Scan End	3000 m/z	Set Collision Cell RF	130.0 Vpp	Set Divert Valve	Waste



Scanned copy of mass spectrum of (±)-2a



¹H NMR (400 MHz, CDCl₃) of compound (±)-2b



¹³C NMR (100 MHz, CDCl₃) of compound (±)-2b

Display Report

Analysis Info

Analysis Name
Method
Sample Name
Comment

D:\Data\user data\2015\JAN-2015\19-JAN-2015\Dr.A.Bisai-AB-LK-05-94_1-A,2_01_1445.d
HRLCMS-20 Sept tune wide.m
Dr.A.Bisai-AB-LK-05-94

Acquisition Date 1/10/2015 11:37:40 PM

Operator RUCHI

Instrument micrOTOF-Q II 10330

Acquisition Parameter

Source Type
Focus
Scan Begin
Scan End

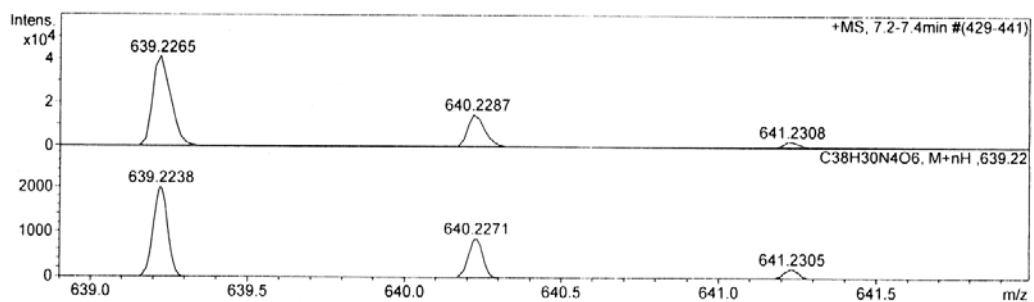
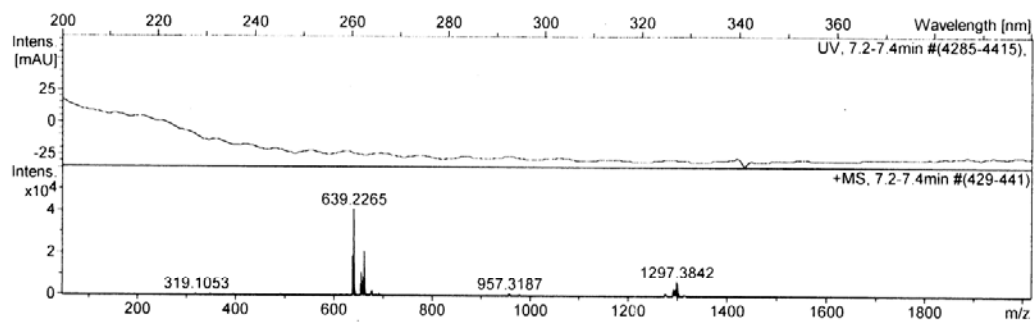
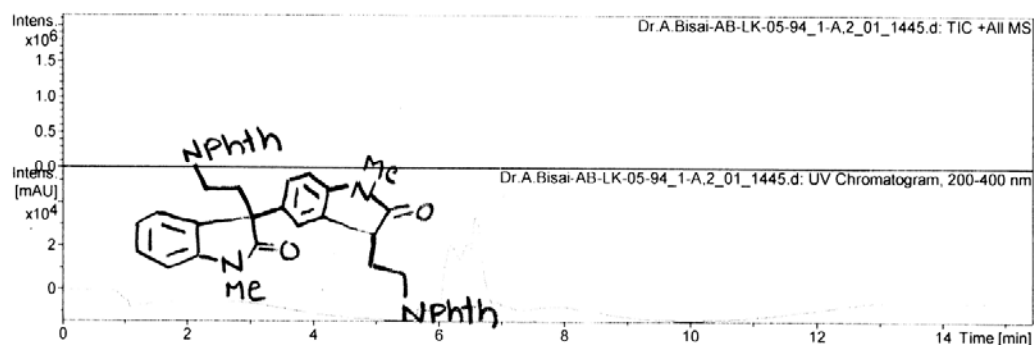
ESI
Not active
50 m/z
3000 m/z

Ion Polarity
Set Capillary
Set End Plate Offset
Set Collision Cell RF

Positive
4500 V
-500 V
600.0 Vpp

Set Nebulizer
Set Dry Heater
Set Dry Gas
Set Divert Valve

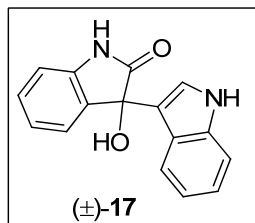
1.2 Bar
200 °C
7.0 l/min
Waste



Scanned copy of mass spectrum of (±)-2b

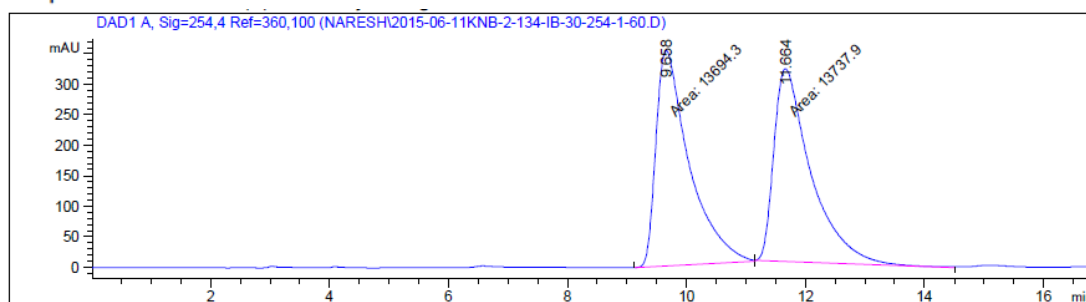
HPLC data

HPLC data of (17) (Racemic)



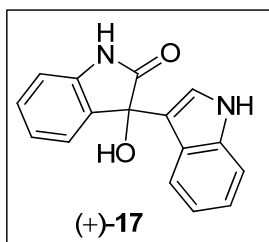
Data File C:\CHEM32\1\DATA\NARESH\2015-06-11KNB-2-134-IB-30-254-1-60.D

Sample Name: KNB-2-134-IB-30-254-1-60



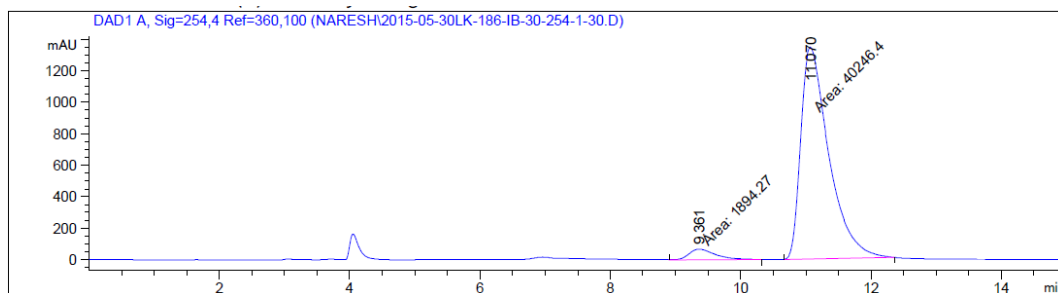
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.658	MM	0.6458	1.36943e4	353.43991	49.9204
2	11.664	MM	0.7239	1.37379e4	316.31293	50.0796
Totals :				2.74322e4	669.75284	

HPLC data of (+)-17:



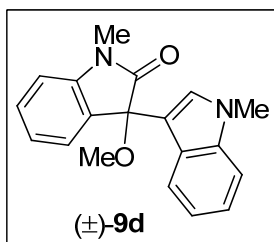
Data File C:\CHEM32\1\DATA\NARESH\2015-05-30LK-186-IB-30-254-1-30.D

Sample Name: LK-186-IB-30-254-1-30



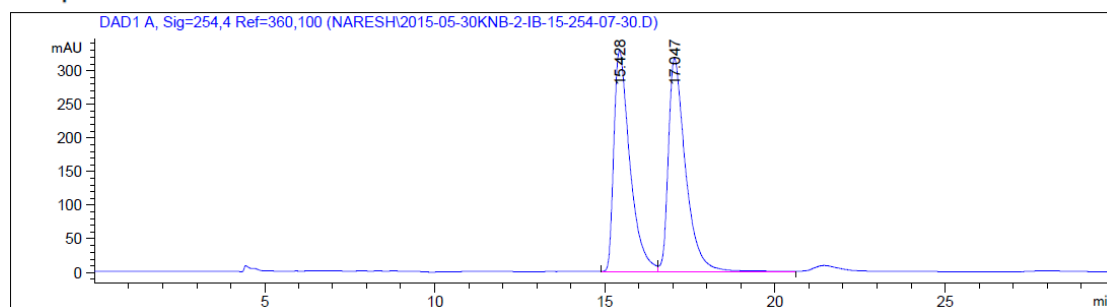
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.361	MM	0.4656	1894.26733	67.81317	4.4951
2	11.070	MM	0.4983	4.02464e4	1346.06982	95.5049

Totals : 4.21407e4 1413.88300

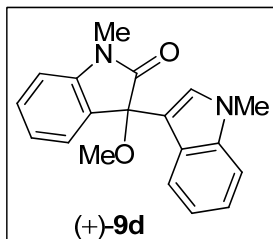
HPLC data of (9d) (Racemic):

Data File C:\CHEM32\1\DATA\NARESH\2015-05-30\KNB-2-IB-15-254-07-30.D

Sample Name: KNB-2-IB-15-254-07-30

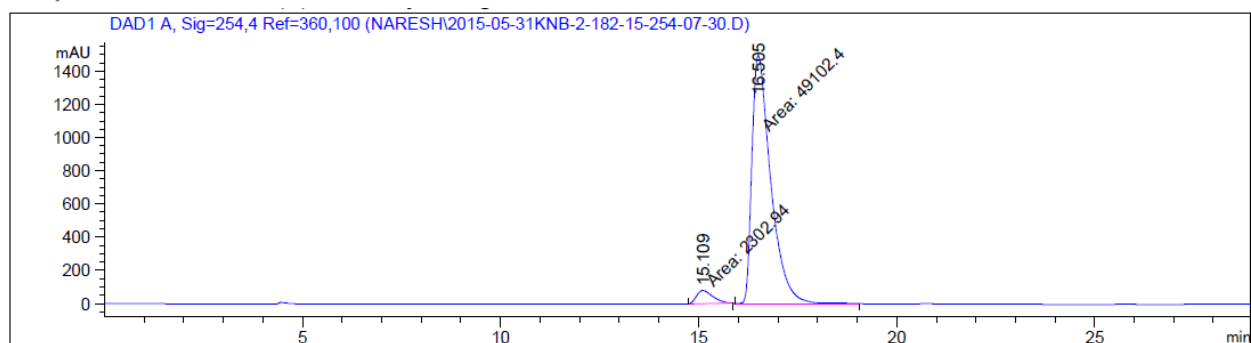


Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.428	BV	0.4892	1.07345e4	328.56503	49.2501
2	17.047	VB	0.5194	1.10613e4	316.73373	50.7499
Totals :				2.17958e4	645.29877	

HPLC data of (+)-9d:

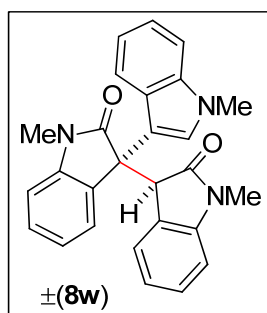
Data File C:\CHEM32\1\DATA\NARESH\2015-05-31KNB-2-182-15-254-07-30.D

Sample Name: KNB-2-182-15-254-07-30



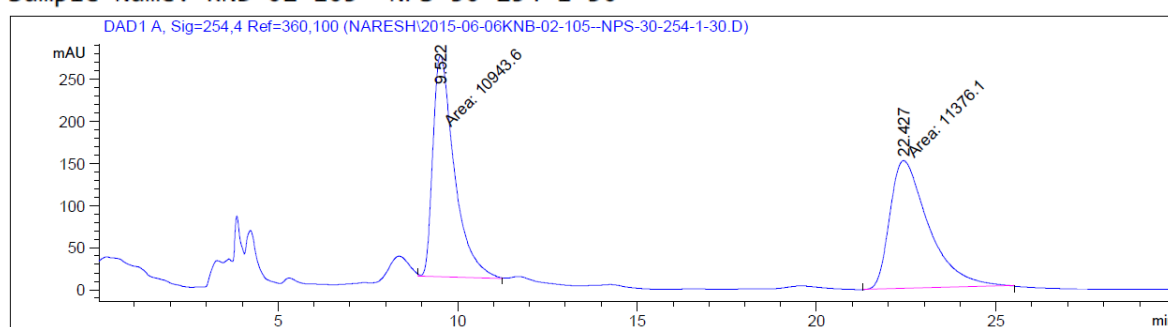
Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	15.109	MM	0.4845	2302.93848	79.21637	4.4800
2	16.505	MM	0.5471	4.91024e4	1495.94312	95.5200

Totals : 5.14053e4 1575.15948

HPLC data of (8w) (Racemic):

Data File C:\CHEM32\1\DATA\NARESH\2015-06-06KNB-02-105--NPS-30-254-1-30.D

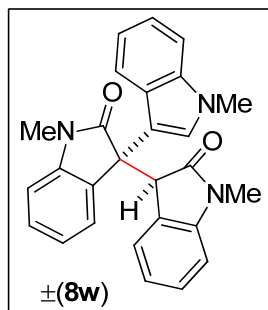
Sample Name: KNB-02-105--NPS-30-254-1-30



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	9.522	MM	0.6942	1.09436e4	262.72665	49.0310
2	22.427	MM	1.2508	1.13761e4	151.59036	50.9690

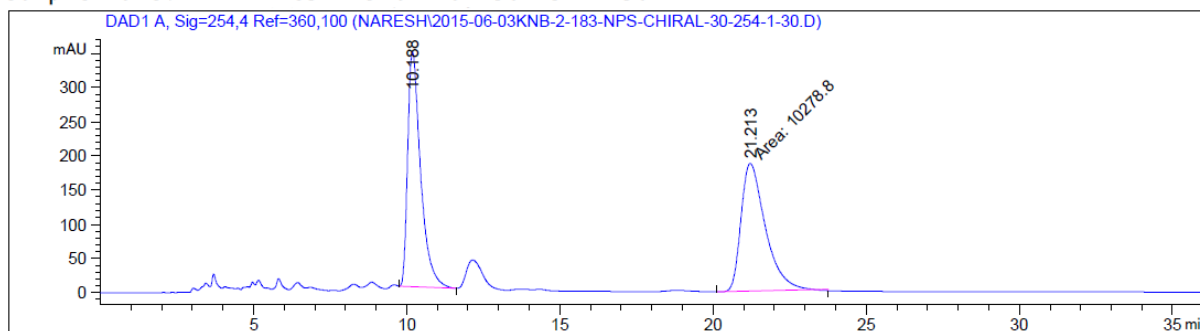
Totals : 2.23197e4 414.31702

HPLC data of (8w) synthesized from enantioenriched starting material (+)-9d (ee = 91%):



Data File C:\CHEM32\1\DATA\NARESH\2015-06-03KNB-2-183-NPS-CHIRAL-30-254-1-30.D

Sample Name: KNB-2-183-NPS-chiral-30-254-1-30



Peak #	RetTime [min]	Type	Width [min]	Area [mAU*s]	Height [mAU]	Area %
1	10.188	BB	0.4426	1.00984e4	343.85004	49.5573
2	21.213	MM	0.9179	1.02788e4	186.62915	50.4427
Totals :				2.03772e4	530.47919	