

Supporting Information

for

An oxidative amidation and heterocyclization approach for the synthesis of β -carbolines and dihydroeudistomin Y

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Experimental and analytical data

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Section A: General information

All reagents were used as received from commercial sources without further purification or prepared as described in the literature. Reactions were stirred using Teflon-coated magnetic stirring bars. TLC plates were visualized by ultraviolet light or by treatment with a spray of Pancaldi reagent $\{(\text{NH}_4)_6\text{MoO}_4, \text{Ce}(\text{SO}_4)_2, \text{H}_2\text{SO}_4, \text{H}_2\text{O}\}$. Chromatographic purification of products was carried out by flash column chromatography on silica gel (60–120mesh). Melting points were determined either on DSC-60A, Shimadzu or on an electro thermal melting point apparatus and are uncorrected. NMR spectra were measured in CDCl_3 , and DMSO-d_6 (all with TMS as internal standard) on a Varian Gemini 400 MHz FT and 500 MHz FT magnetic resonance spectrometers. Chemical shifts (δ) are reported in ppm, and coupling constants (J) are in Hz. The following abbreviations were used to explain the multiplicities: s = singlet, d = doublet, t = triplet, q = quartet, m = multiplet. Mass spectra were recorded on an HP-5989A quadrupole mass spectrometer.

Section B: Experimental section:

Analytical data of α -keto-amides (9f–9j).

N-(2-(1*H*-Indol-3-yl)ethyl)-2-(4-methoxyphenyl)-2-oxoacetamide (9f):

55% Yield mp 130-132.4 °C. IR (cm^{-1}): 3246, 2935, 2842, 1668, 1650, 1621, 1598, 1568, 1315, 1264, 1173, 1029, 841, 764, 741. ^1H NMR (400 MHz, CDCl_3): δ_{H} 8.40 (d, 2H, $J = 9.2$ Hz), 8.04 (1H, s, NH), 7.65 (d, 1H, $J = 7.6$ Hz), 7.39 (d, 1H, $J = 8$ Hz), 7.23 to 7.09 (3H ArH), 6.94 (d, 2H, $J = 8.8$ Hz), 3.88 (s, 3H), 3.73 (d, 2H, $J = 6.8$ Hz), 3.09 (t, 2H, $J = 6.8$ Hz). ^{13}C NMR (100 MHz, DMSO d_6): δ_{C} 188.7, 165.0, 164.0, 136.1, 132.2

(2C), 127.1, 125.7, 122.7, 120.8, 118.2 (2C), 114.1 (2C), 111.3 (2C), 55.6, 39.2 and 24.7. Ms, m/z (%) = 323.2 (M+1), 345.2 (M+23).

***N*-(2-(1*H*-Indol-3-yl)ethyl)-2-(2,4-dichlorophenyl)-2-oxoacetamide (9g):**

50% Yield. ^1H NMR (400 MHz, DMSO d^6): δ_{H} 10.84 (s, 1H, NH), 9.14 (s, 1H, NH), 7.77 (s, 1H), 7.62 to 7.53 (3H, ArH), 7.36 (d, 1H, $J = 2.0$ Hz), 7.19 (s, 1H), 6.98 (dd, 1H, $J = 7.6$ Hz), 7.09 (dd, 1H, $J = 7.6$ Hz), 3.52 (d, 2H, $J = 6.8$ Hz), 2.95 (t, 2H, $J = 7.2$ Hz). ^{13}C NMR (100 MHz, DMSO d^6): δ_{C} 189.5, 162.3, 137.5, 136.2, 132.8, 132.6, 129.8 (2C), 127.4, 127.1, 122.7, 120.9, 118.2 (2C), 111.3 (2C), 55.7 and 24.5. Ms, m/z (%) = 361.1 (M+1), 363.1 (M+3), 383.1 (M+23).

***N*-(2-(1*H*-Indol-3-yl)ethyl)-2-(4-(benzyloxy)phenyl)-2-oxoacetamide (9h):**

52% Yield. mp 137.1-138.2 °C. IR (cm^{-1}): 3439, 3251, 3091, 2902, 1681, 1643, 1598, 1568, 1456, 1258, 1225, 1173, 995, 850, 741. ^1H NMR (400 MHz, CDCl_3): δ_{H} 8.39 (d, 2H, $J = 9.2$ Hz), 8.06 (s, 1H, NH), 7.64 (d, 1H, $J = 7.6$ Hz), 7.43- 7.34 (m, 5 ArH), 7.23 – 7.13 (m, 3ArH), 7.11 (s, 1H), 7.02 (d, 2H, $J = 9.2$ Hz), 5.25 (s, 2H), 3.72 (d, 2H, $J = 6.6$ Hz), 3.07 (t, 2H, $J = 6.8$ Hz). ^{13}C NMR (100 MHz, DMSO d^6): δ_{C} 188.6, 164.9, 163.0, 136.1 (2C), 132.1 (2C), 128.3 (2C), 127.9, 127.6 (2C), 127.0, 125.9, 122.6, 120.8, 118.1 (2C), 114.8 (2C), 111.3, 111.2, 69.5, 39.2 and 24.6. Ms, m/z (%) = 399.4 (M+1).

***N*-(2-(1*H*-Indol-3-yl)ethyl)-2-([1,1'-biphenyl]-4-yl)-2-oxoacetamide (9i):**

60% Yield. mp 175.1-177.1 °C. IR (cm^{-1}): 3436, 3239, 3089, 1684, 1645, 1599, 1485, 1457, 1202, 1100, 857, 762, 743. ^1H NMR: (400 MHz, CDCl_3): δ_{H} 8.41 (d, 2H, $J = 6.6$ Hz), 8.06 (s, 1H, NH), 7.70 - 7.63 (m, 5 ArH), 7.47- 7.38 (m, 4 ArH), 7.22 – 7.10 (m, 3 ArH), 3.76 (d, 2H, $J = 6.6$ Hz), 3.10 (t, 2H, $J = 6.8$ Hz). ^{13}C NMR (100 MHz, DMSO d^6):

δ_c 189.9, 164.8, 145.6, 138.6, 136.2, 131.6, 130.4 (2C), 129.1 (2C), 128.6, 127.1 (2C), 127.0 (2C), 126.9, 122.9, 120.9, 118.2 (2C), 111.3 (2C), 39.2 and 24.8. Ms, m/z (%) = 369.2 (M+1), 391.2 (M+23).

***N*-(2-(1*H*-Indol-3-yl)ethyl)-2-oxo-2-(thiophen-2-yl)acetamide (9j):**

42% Yield. mp 147.7-149.9 °C. IR (cm^{-1}): 3394, 3354, 2936, 2916, 2859, 1685, 1654, 1567, 1500, 1404, 1357, 1287, 1093, 1023, 844, 817, 743, 725. ^1H NMR (400 MHz, CDCl_3): δ_H 8.38 (d, 1H, $J = 4$ Hz), 8.08 (s, 1H, NH), 7.81 (d, 1H, $J = 5.5$ Hz), 7.63 (d, 1H, $J = 8.0$ Hz), 7.39 (d, 2H, $J = 8.4$ Hz), 7.23 – 7.11 (m, 2 ArH), 7.06 (d, 1H, $J = 2$ Hz), 3.73 (d, 2H, $J = 6.6$ Hz), 3.07 (t, 2H, $J = 7.2$ Hz). ^{13}C NMR (100 MHz, DMSO d_6): δ_c 179.8, 161.8, 138.8, 137.2, 137.2, 136.2, 128.5, 127.1, 122.6, 120.9, 118.2 (2C), 111.3 (2C), 39.2 and 24.6. Ms, m/z (%) = 299.4 (M+1), 321.4 (M+23).

Analytical data of dihydro-eudistomin (7e–7j).

(2,9-Dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(3-nitrophenyl)methanone (7e):

20% Yield. ^1H NMR (400 MHz, CDCl_3): δ_H 8.33 (s, 1H), 8.14 (d, 1H, $J = 8.0$ Hz), 8.06 (s, 1H), 7.68 (d, 1H, $J = 8.0$ Hz), 7.53 (d, 1H, $J =$ Hz), 7.48 (t, 1H, $J = 8.0$ Hz), 7.41 (d, 1H, $J = 8.0$ Hz), 7.30 - 7.17 (m, 3 ArH), 6.52 (d, 1H, $J = 8.4$ Hz), 5.91 (dd, 1H, $J = 3.2$ & 5.4 Hz), 5.28 (s, 1H), 3.78 (s, 3H). Ms, m/z (%) = 320 (M+1).

(2,9-Dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-methoxyphenyl)methanone (7f):

38% Yield. IR (cm^{-1}): 3222, 3185, 3042, 2937, 2835, 1657, 1624, 1509, 1456, 1249, 1177, 1055, 1032, 848, 810, 770, 742. ^1H NMR (400 MHz, CDCl_3): δ_H 8.17 (s, 1H), 7.67 (d, 1H, $J = 7.2$ Hz), 7.35 (t, 2H, $J = 8.0$ Hz), 7.23 (d, 1H, $J = 7.2$ Hz), 7.20 (d, 3H, $J = 8.0$ Hz), 6.86 (d, 2H, $J = 7.6$ Hz), 6.48 (d, 1H, $J = 8.8$ Hz), 5.94 (dd, 1H, $J = 3.6$ & 5.6 Hz), 5.02 (s, 1H), 3.78 (s, 3H). ^{13}C NMR (100 MHz, $\text{CDCl}_3 + \text{DMSO } d_6$): δ_c 166.8, 157.3,

135.4, 130.1, 129.4, 127.0, 126.7, 124.9, 120.6, 118.4, 117.3, 116.9, 112.5, 110.6, 109.0, 108.8, 105.0, 53.9, 51.5. Ms, m/z (%) = 305 (M+1), 327 (M+23).

(2,4-Dichlorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1H-pyrido[3,4-*b*]indol-1-yl)methanone (8g):

45% Yield. mp 281.1-284.2 °C. IR (cm^{-1}): 3356, 3211, 3086, 2834, 1678, 1581, 1556, 1469, 1385, 1337, 1184, 1110, 1073, 1052, 906, 822, 800, 745. ^1H NMR (400 MHz, DMSO d^6): δ_{H} 10.95 (s, 1H), 8.50 (t, 1H, $J = 6.8$ Hz), 7.64 (s, 1H), 7.43 (d, 2H, $J = 8.4$ Hz), 7.34 (dd, 1H, $J = 2.0, 6.4$ Hz) 7.12 (t, 1H, $J = 7.6$ Hz), 7.02 (t, 1H, $J = 7.6$ Hz), 6.77 (d, 1H, $J = 8.8$ Hz), 6.15 (s, 1H), 3.20 (m, 1H), 3.01 (m, 1H), 2.81 (m, 2H). ^{13}C NMR (100 MHz, DMSO d^6): δ_{C} 171.9, 139.7, 135.1, 133.4, 132.4, 131.6, 130.5, 130.2, 127.9, 127.1, 121.7, 118.7, 118.1, 111.7, 108.6, 74.8, 38.2 and 25.2. Ms, m/z (%) = 343.1 (M+1), 321.4 (M+23). HRMS (EI): calcd. m/z for $\text{C}_{18}\text{H}_{13}\text{N}_2\text{OCl}_2$ $[\text{M}]^+$ 343.0405, found 343.0101.

(4-(Benzyloxy)phenyl)(2,9-dihydro-1H-pyrido[3,4-*b*]indol-1-yl)methanone (7h):

46% Yield. ^1H NMR (400 MHz, CDCl_3): δ_{H} 8.04 (s, 1H), 7.65 (d, 1H, $J = 8.8$ Hz), 7.40-7.32 (m, 5 ArH), 7.23-7.11 (m, 4 ArH), 6.95 (d, 2H, $J = 8.8$ Hz), 6.51 (d, 1H, $J = 8.4$ Hz), 5.95 (dd, 1H, $J = 5.6$ & 3.6 Hz), 5.04 (s, 2H), 5.00 (s, 1H). Ms, m/z (%) = 381.2 (M+1), 403 (M+23). HRMS (EI): calcd. m/z for $\text{C}_{25}\text{H}_{21}\text{N}_2\text{O}_2$ $[\text{M}]^+$ 381.1603, found 381.1583.

[1,1'-Biphenyl]-4-yl(2,9-dihydro-1H-pyrido[3,4-*b*]indol-1-yl)methanone (7i):

35% Yield. mp 224-226 °C. ^1H NMR (400 MHz, CDCl_3): δ_{H} 8.12 (s, 1H), 7.69 (d, 1H, $J = 8.0$ Hz), 7.64 - 7.21 (m, 12 ArH), 6.54 (d, 1H, $J = 8.4$ Hz), 5.97 (dd, 1H, $J = 3.6$ & 4.8 Hz), 5.13 (s, 1H). ^{13}C NMR (100 MHz, DMSO d^6): δ_{C} 173.7, 166.8, 143.0, 139.5, 138.9,

136.2, 135.2, 130.7, 128.8, 127.8, 127.3, 126.9, 126.6, 125.7, 121.8, 119.4, 118.6, 118.0, 111.5, 109.7, 108.5, 105.4, 75.4, 52.8, 25.2 Ms, m/z (%) = 351.2 (M+1).

[1,1'-Biphenyl]-4-yl(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (8i):

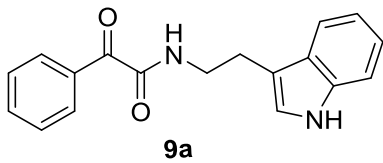
20% Yield. mp 271.1-273.6 °C. ^1H NMR (400 MHz, CDCl_3): δ_{H} 8.81 (s, 1H), 7.55 - 7.27 (m, 11 ArH), 6.31 (t, 1H, $J = 7.0$ Hz, NH), 5.77 (s, 1H, OH), 3.49 (m, 1H), 3.27 (m, 1H), 3.00 (m, 2H). ^{13}C NMR (100 MHz, $\text{DMSO } d^6$): δ_{C} 173.7, 143.0, 139.5, 139.4, 135.0, 130.9, 128.8 (3C), 127.7, 127.5, 126.6 (5), 121.3, 118.4, 117.9, 111.5, 108.5, 75.3, 38.2 and 25.2. Ms, m/z (%) = 351.2 (M+1).

(1-Hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(thiophen-2-yl)methanone (8j):

42% Yield. ^1H NMR (400 MHz, CDCl_3): δ_{H} 8.77 (s, 1H), 7.52 (d, 1H, $J = 8.0$ Hz), 7.44 (m, 2H), 7.17 (t, 2H, $J = 7.6$ Hz), 7.05 (d, 1H, $J = 8.8$ Hz), 6.84 (d, 1H, $J = 8.8$ Hz), 6.15 (brs, 1H, NH), 5.84 (s, 1H, OH), 3.36 – 3.29 (m, 2H), 3.02 (m, 1H), 2.89 (m, 1H). ^{13}C NMR (100 MHz, $\text{DMSO } d^6$): δ_{C} 171.8, 139.6, 135.1, 133.4, 132.4, 131.5, 130.5, 127.8, 127.0, 121.6, 118.0, 111.6, 108.6, 74.7, 38.2 and 25.1. Ms, m/z (%) = 281 (M+1).

Section C: ^1H , ^{13}C , Mass, and IR.

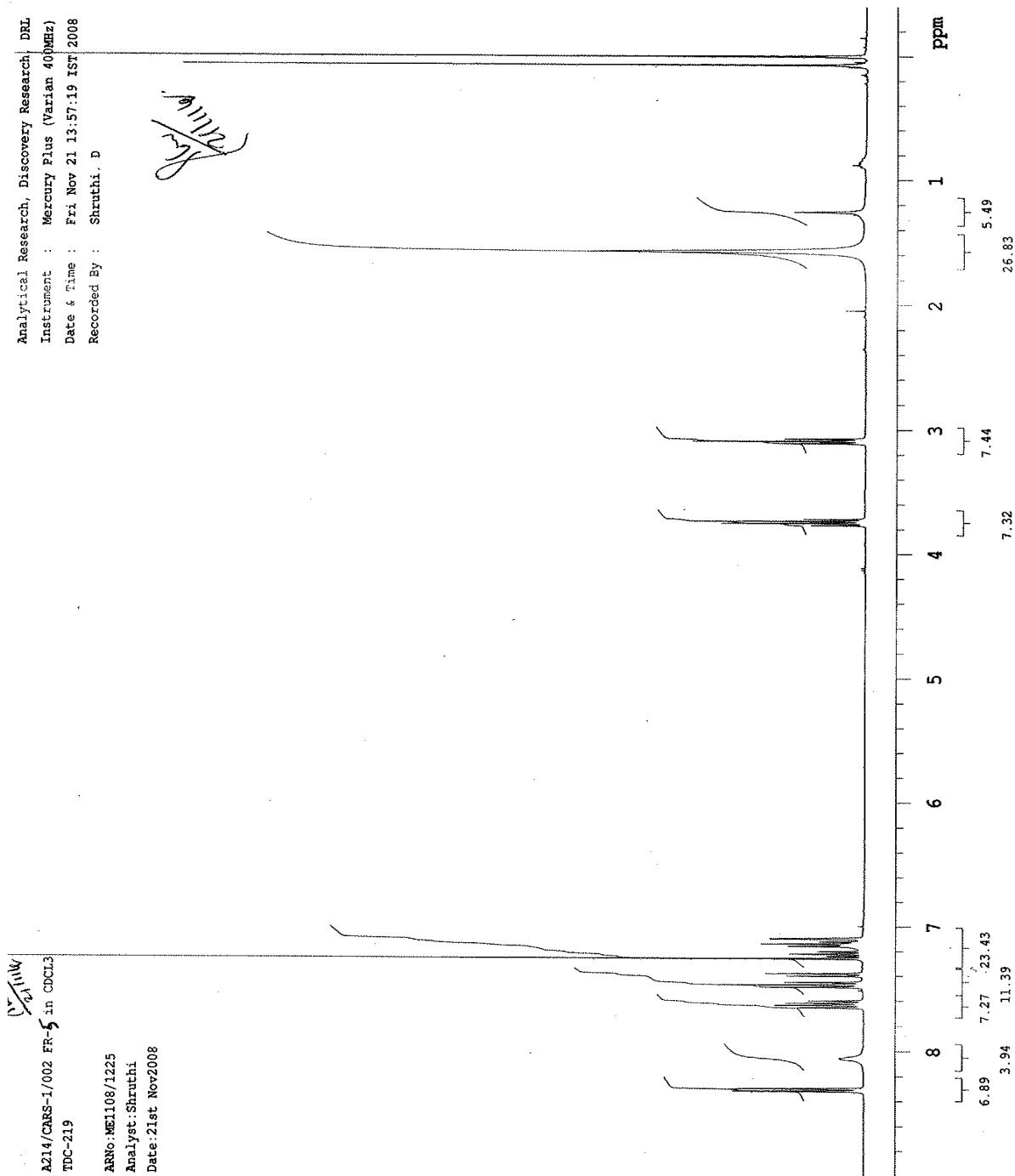
¹H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-phenylacetamide (**9a**):



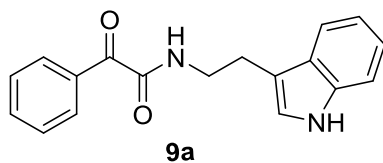
Analytical Research, Discovery Research, DRL
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Date & Time : Fri Nov 21 13:57:19 IST 2008
Recorded By : Shruthi. D

12/11/66
 A214/CARS-1/002 FR-5 in CDCL3
 TDC-219

ARNO:ME1108/1225
Analyst:Shruthi
Date:21st Nov2008



¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-phenylacetamide (**9a**):



Analytical Research, Discovery Research, DRL

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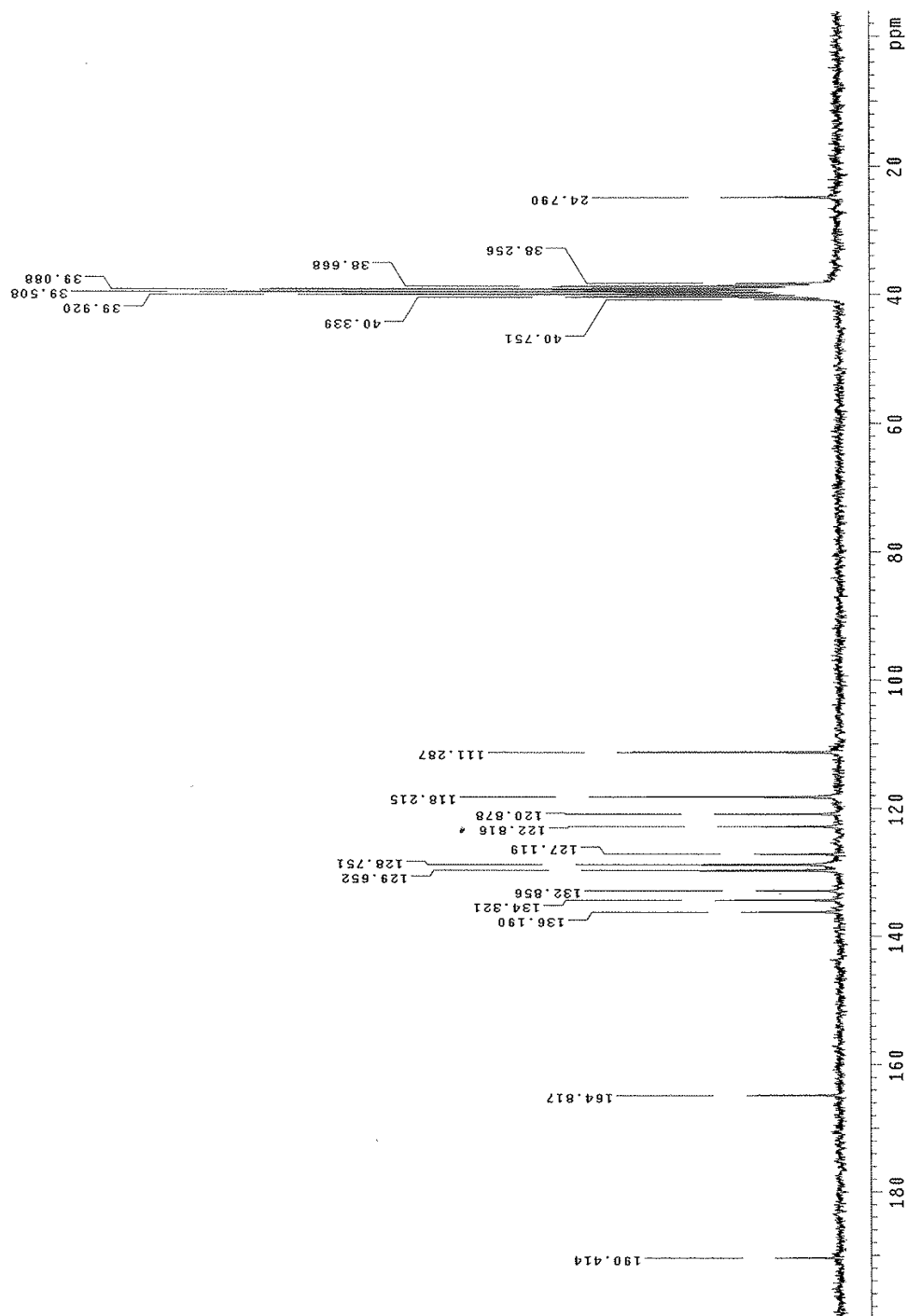
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A214/CARS-1/006 FR2 IN DMSO
TDC-219

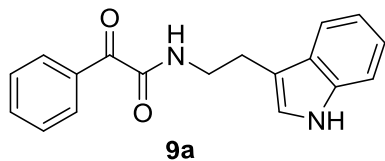
AR No: GE0609/15

Analyst: Seshu

Date: 3rd June 2009



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CPS,MIYAPUR

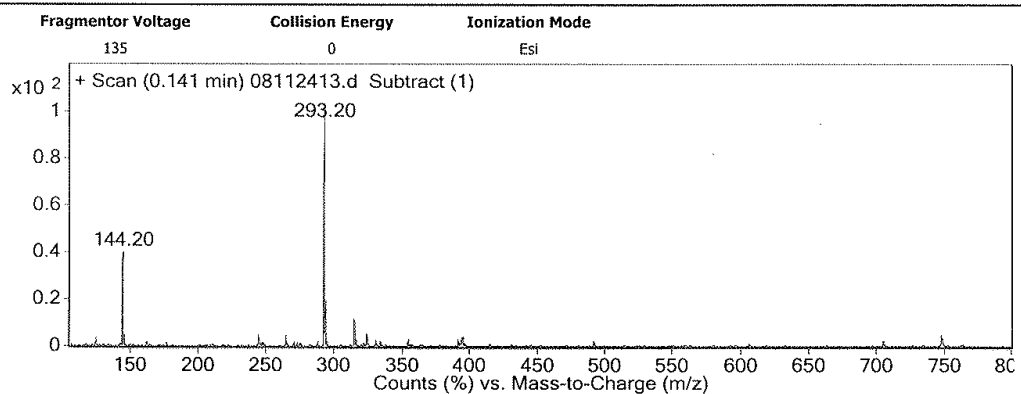
Mass Analysis Report

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DA Method DA.m

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User Name
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Comment

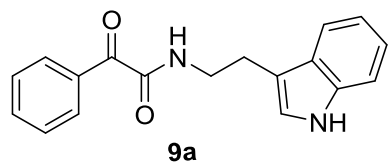
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08/10/08
24/11/08

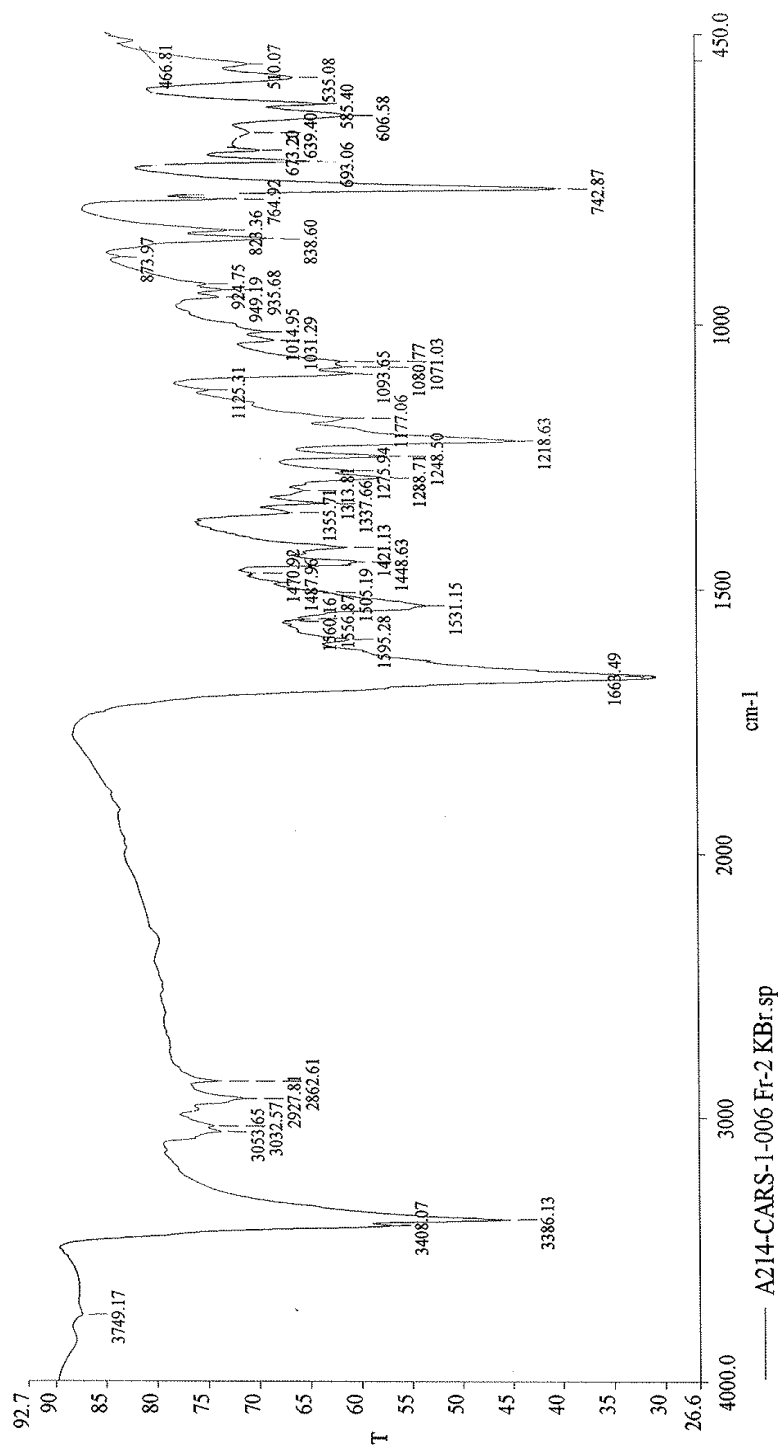
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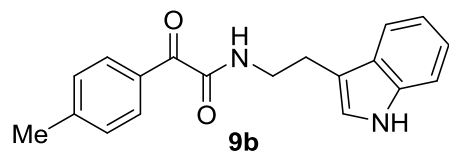
DR. REDDY'S LABORATORIES LIMITED

TDC/CCS-ANALYTICAL RESEARCH.



Analyst: JHANSI
SIGN: *[Signature]*
Date: 6/2/09

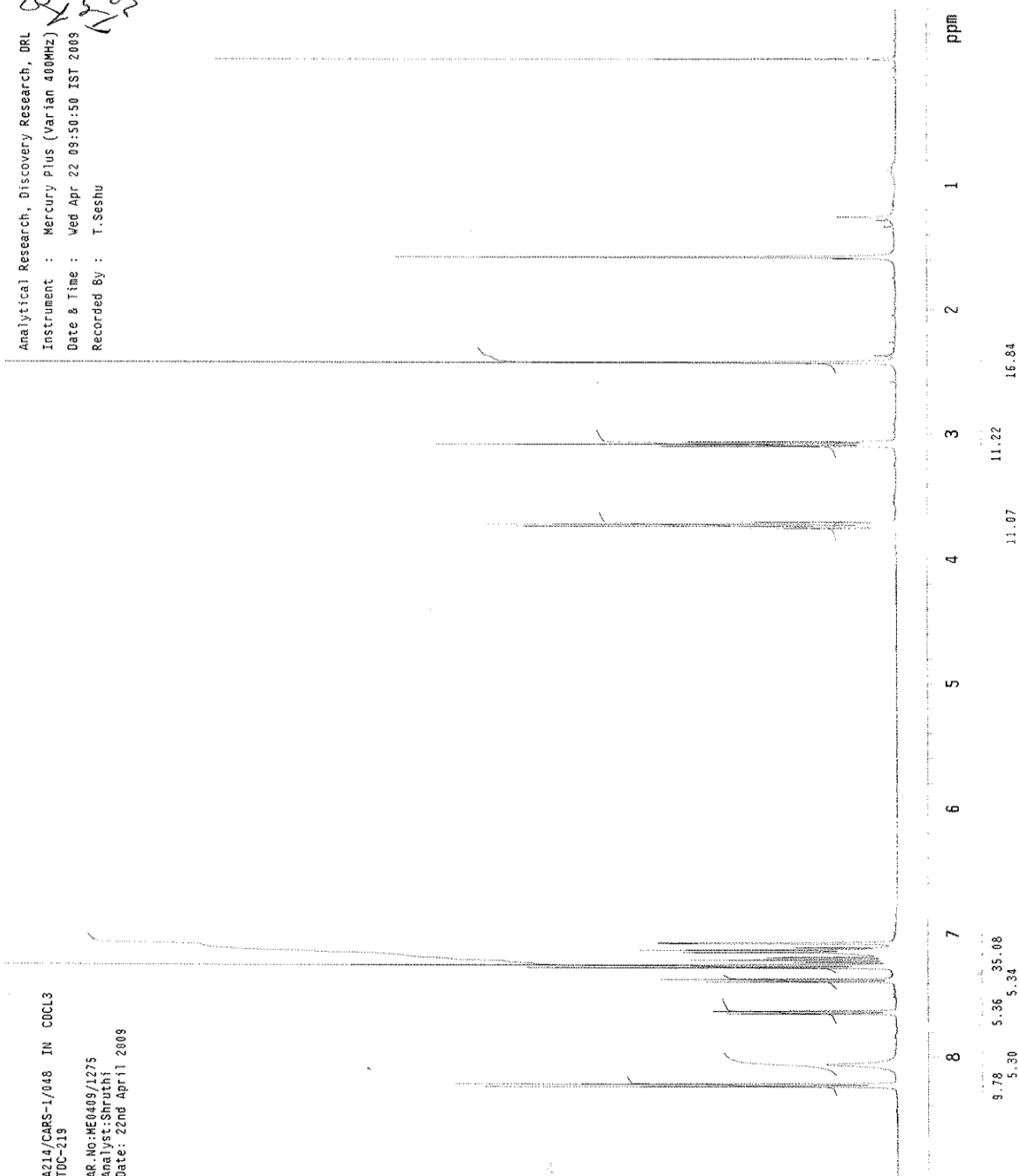
^1H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(*p*-tolyl)acetamide (**9b**):



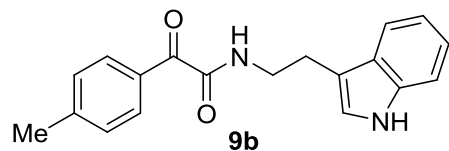
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 Date & Time : Wed Apr 22 09:50:50 IST 2009
 Recorded By : T.Seshu
 10/04/09
 T.S.

4214/CARS-1/048 IN CDCL3
 TDC-213

AR-NO:ME0409/1275
 Analyst:Shruthi
 Date: 22nd April 2009



¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(*p*-tolyl)acetamide (**9b**):



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Analytical Research, Discovery Research, DRL

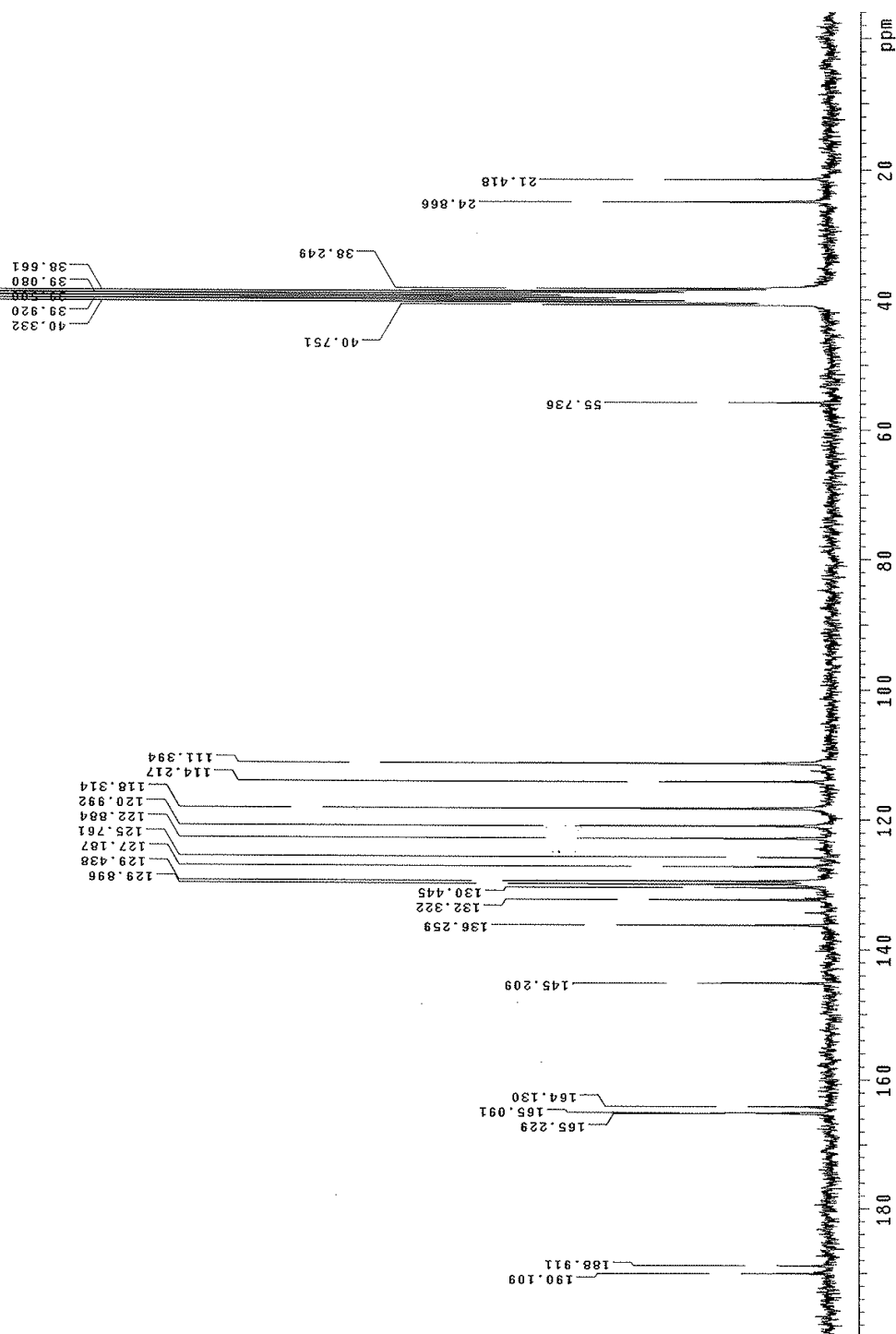
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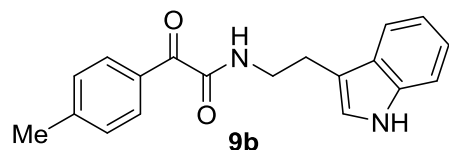
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A214/CARS-1/048 in DMSO
TDC-219

AR-NO:GE0609/42
Analyst: Srikanth.A
Date:10th June 2009



Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(*p*-tolyl)acetamide (**9b**):



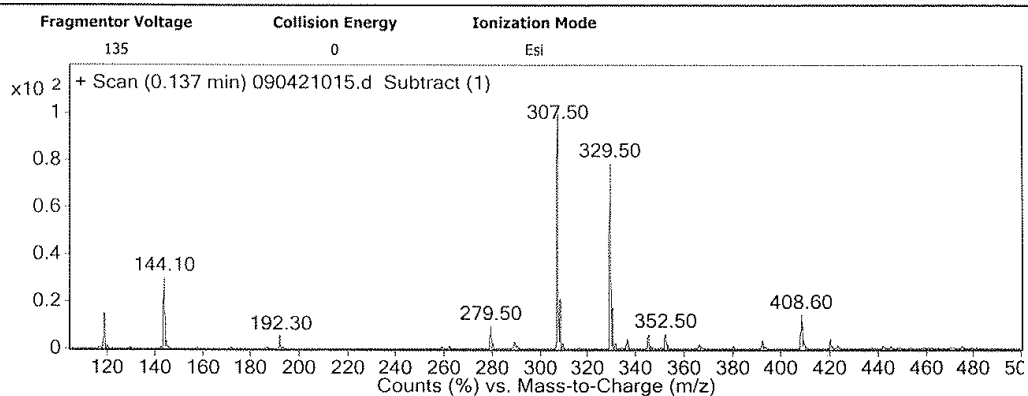
CPS,MIYAPUR

Mass Analysis Report

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DA Method	Quant Process.m	Comment	

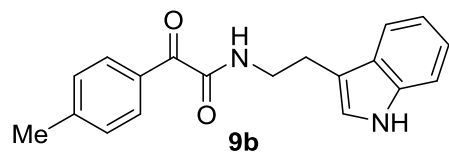
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090421/04/109

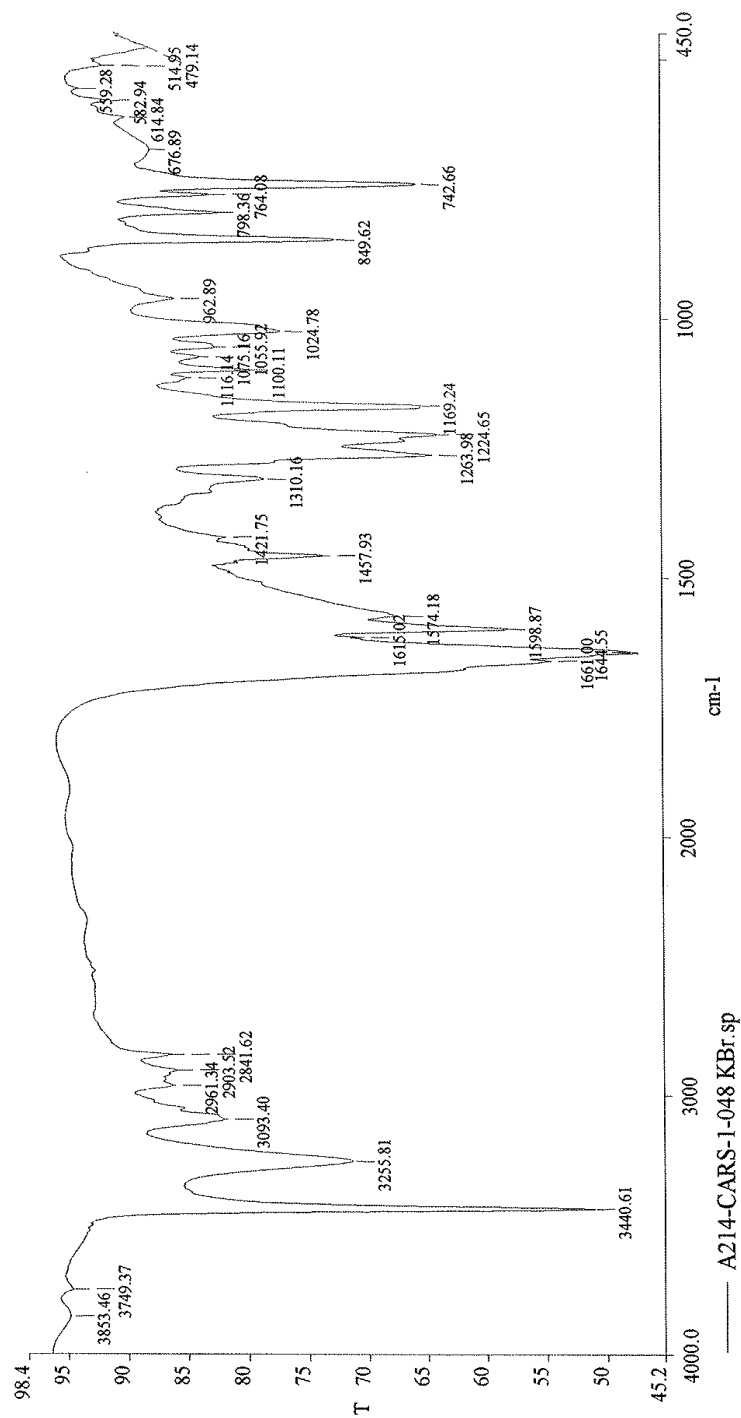
IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(*p*-tolyl)acetamide (**9b**):



Date: 6/2/09
Time: 3:03:37 PM

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TDC/CCS-ANALYTICAL RESEARCH .

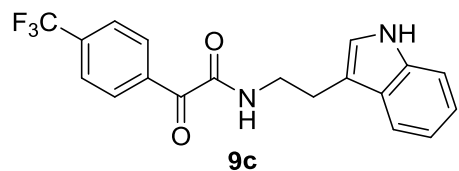


Analyst: JHANSI

SIGN: *[Signature]*

DATE: 06/02/09
TIME: 3:03:37 PM

¹H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(4-(trifluoromethyl)phenyl)acetamide (**9c**):

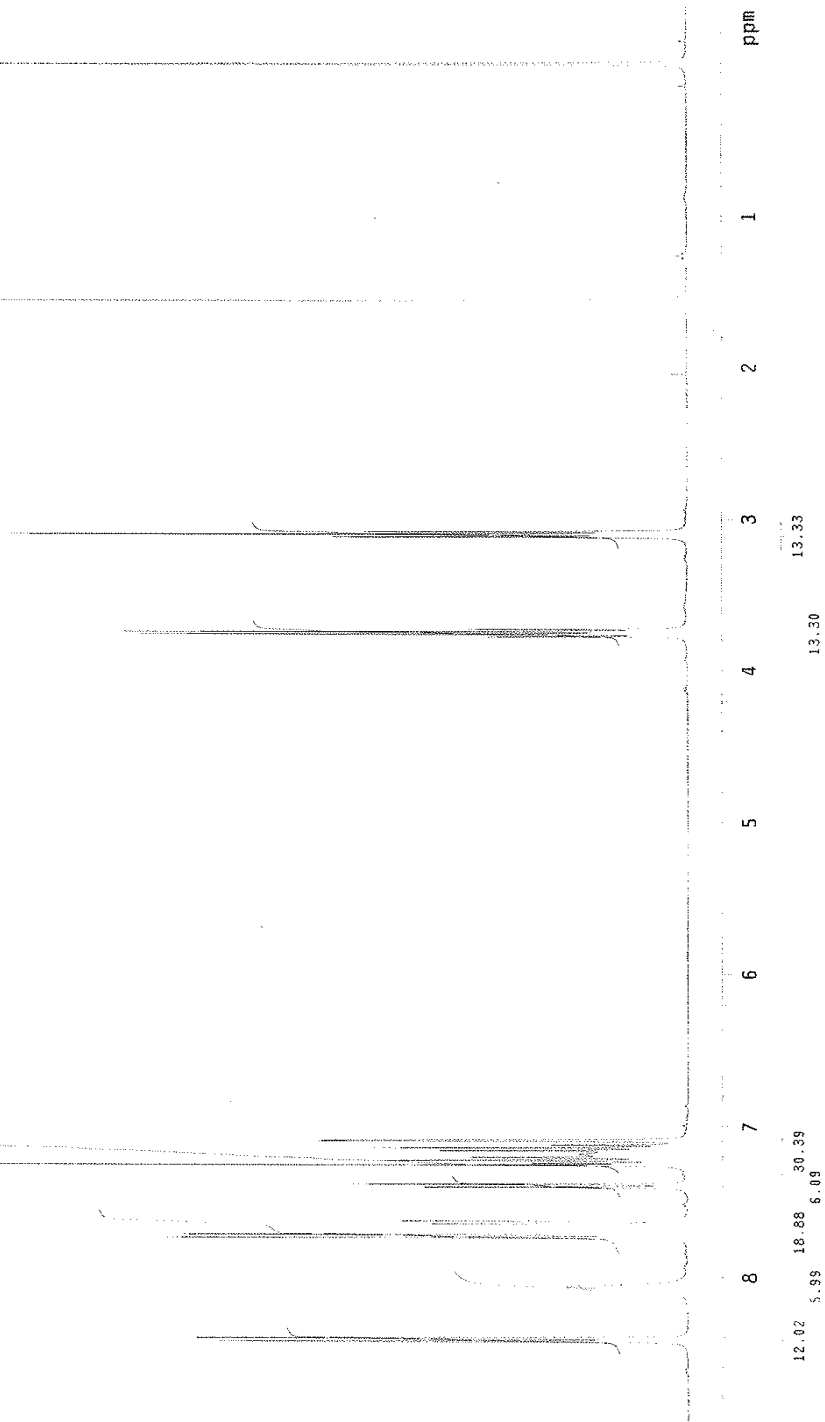


Analytical Research, Discovery Research, DRL
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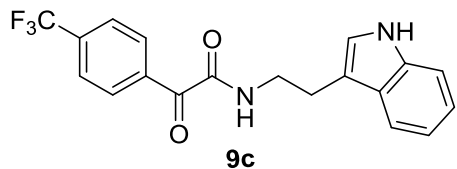
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A214/CARS-1/033 F1-(1) in CDCl3
 TDC-219

AR NO:ME0409/377
 Analyst:Shruthi
 Date:7th April 2009



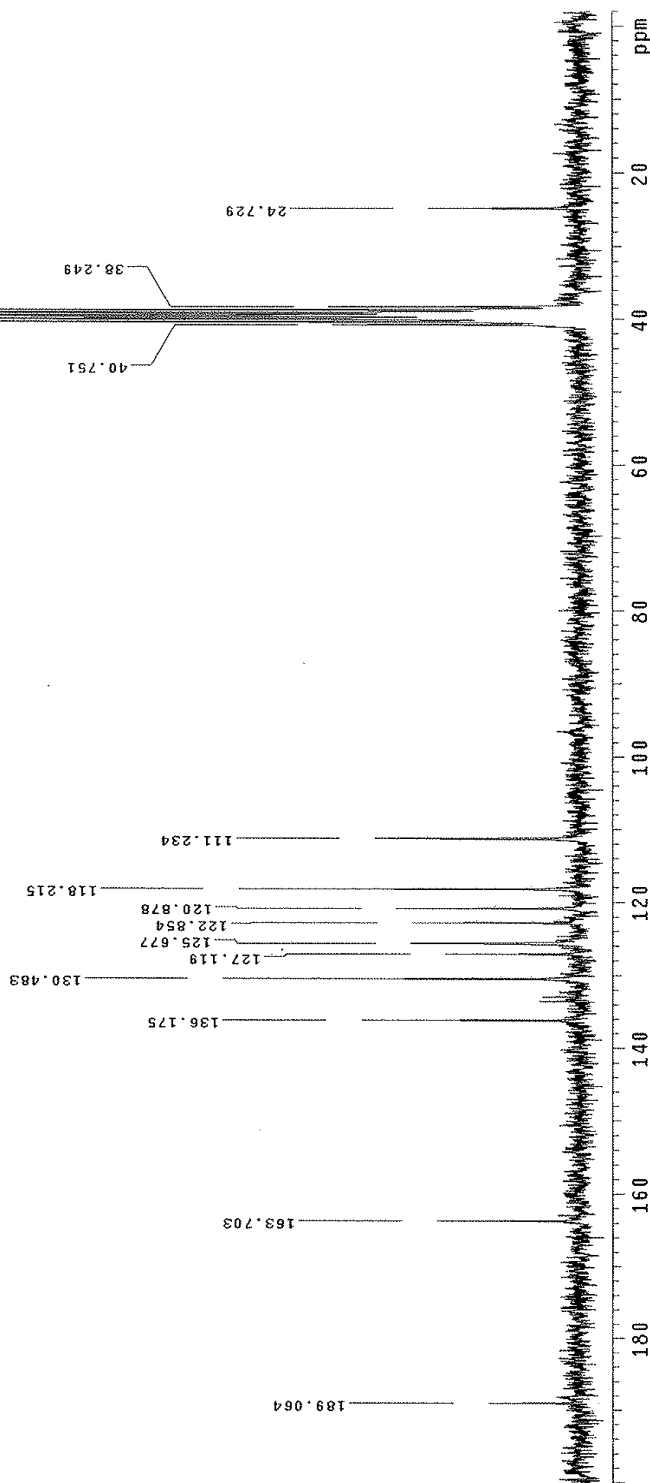
¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(4-(trifluoromethyl)phenyl)acetamide (**9c**):



Analytical Research, Discovery Research, DRL
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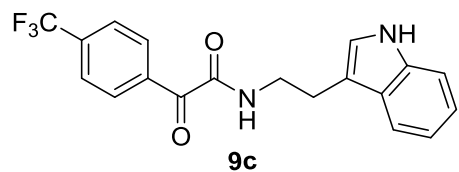
38.668
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Handwritten signature and date: 4/6/09



A214/CARS1/043 in DMSO
 TDC-219
 AR.No: GE0609/19
 Analyst: Srikanth.A
 Date: 4th June 2009

Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(4-(trifluoromethyl)phenyl)acetamide (**9c**):



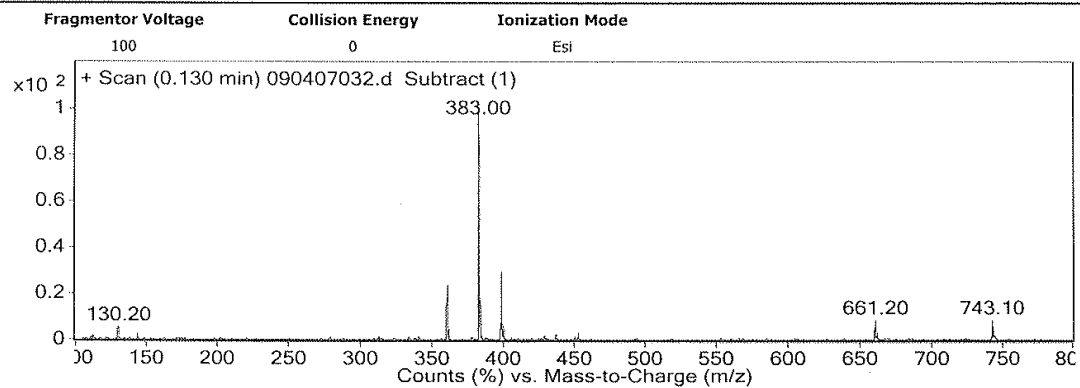
CPS,MIYAPUR

Mass Analysis Report

Y 100.000000

Data Filename	090407032.d	Sample Name	A214/CARS-1/043 FR-1
Sample Type	Sample	Position	Vial 78
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	ESMS.m	Comment	

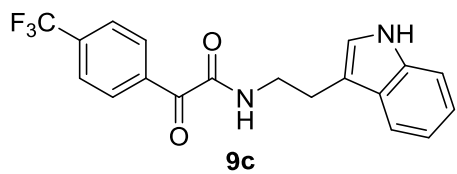
User Spectra



--- End Of Report ---

07/04/09

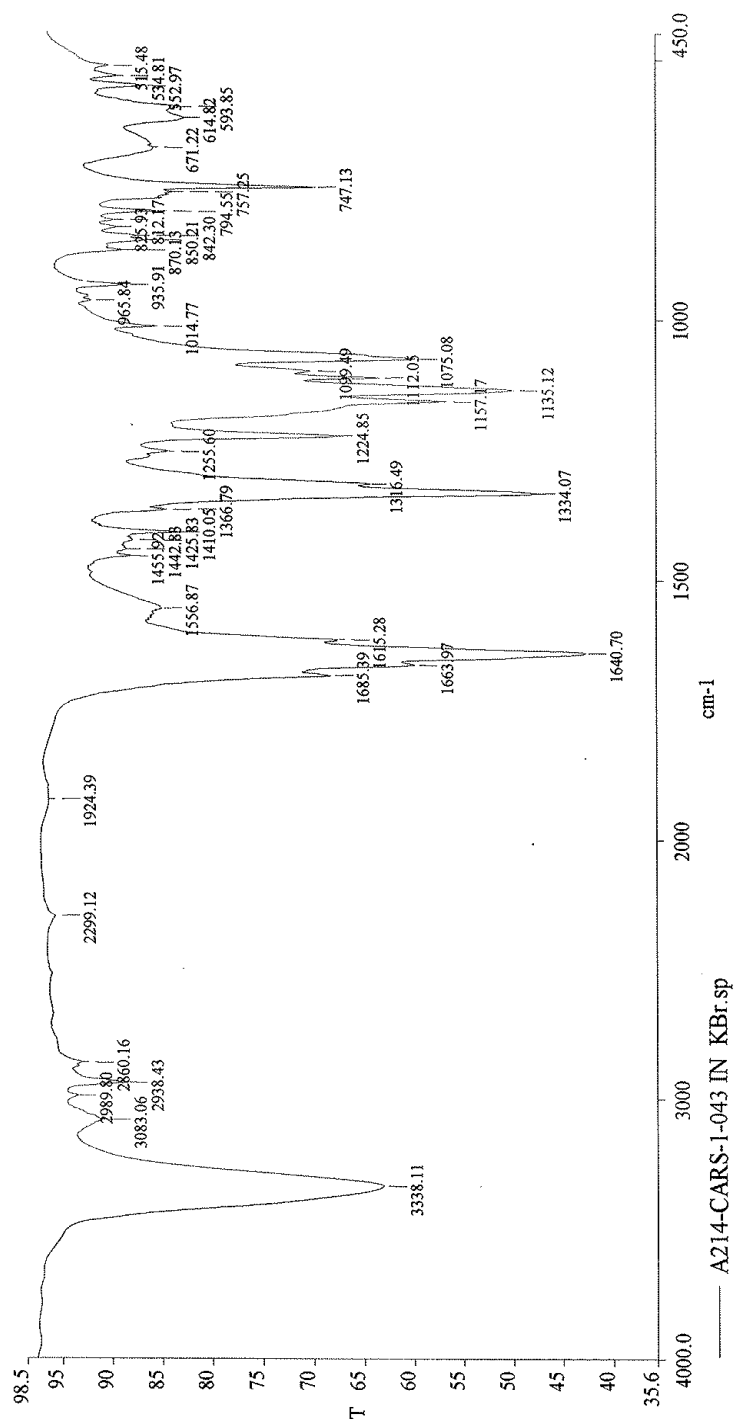
IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(4-(trifluoromethyl)phenyl)acetamide (**9c**):



Date: 6/2/09
Time: 4:02:44 PM

DR.REDDY'S LABORATORIES LIMITED

TDC/CCS-ANALYTICAL RESEARCH.

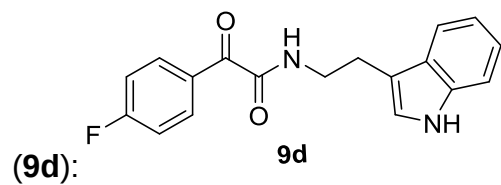


Analyst: JHANSI

SIGN:

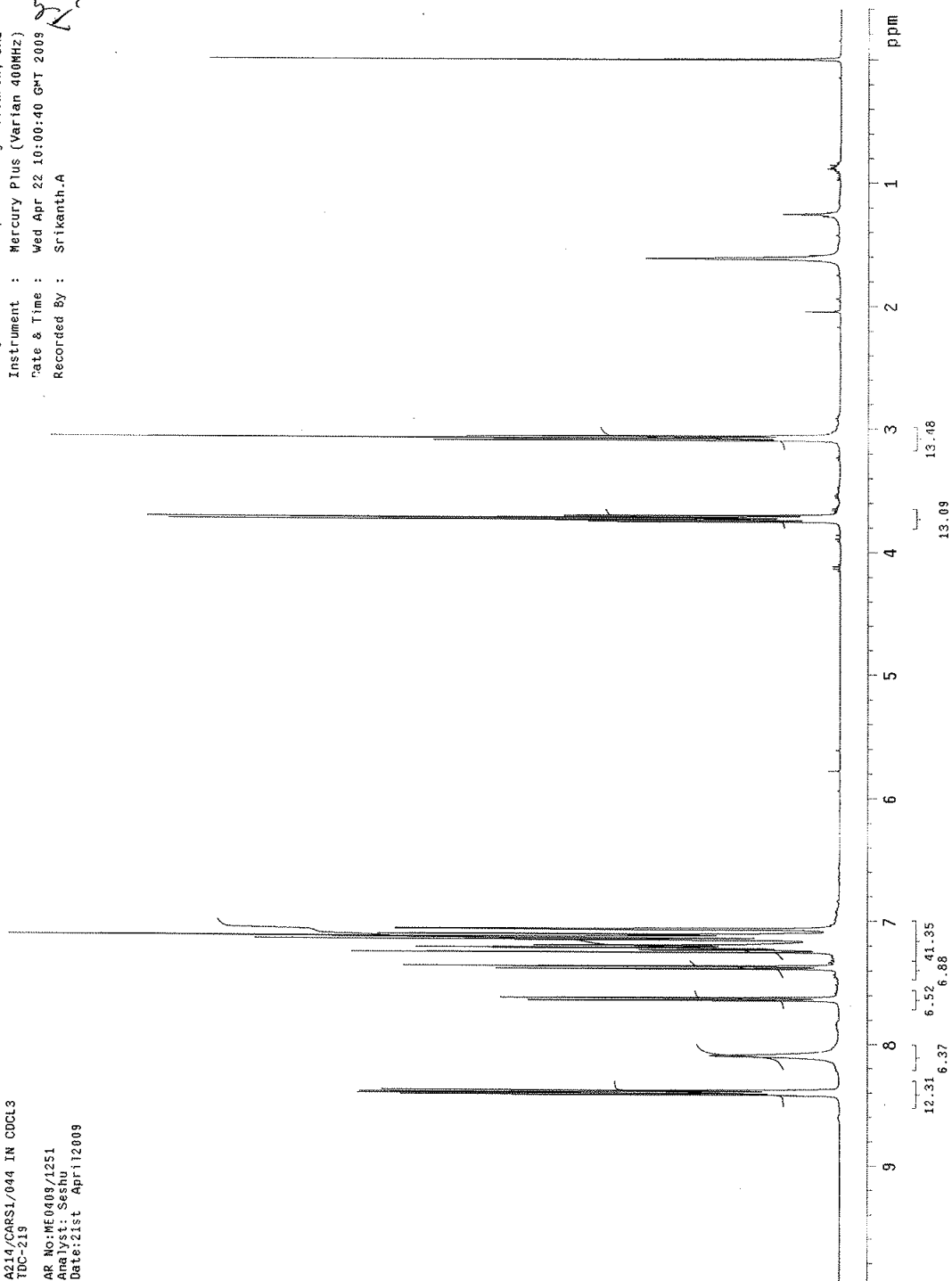
6/2/09
A214-CARS-1-043 IN KBr.sp

¹H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-fluorophenyl)-2-oxoacetamide

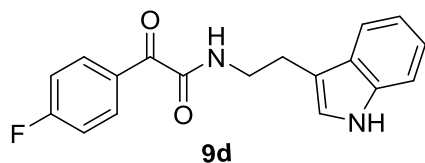


Analytical Research, Discovery Research, DRL
 Instrument : Mercury Plus (Varian 400MHz)
 Date & Time : Wed Apr 22 10:00:40 GMT 2009
 Recorded By : Srikanth.A

A214/CARS1/044 IN CDCl₃
 TDC-219
 AR No: ME0409/1251
 Analyst: Seshu
 Date: 21st April 2009



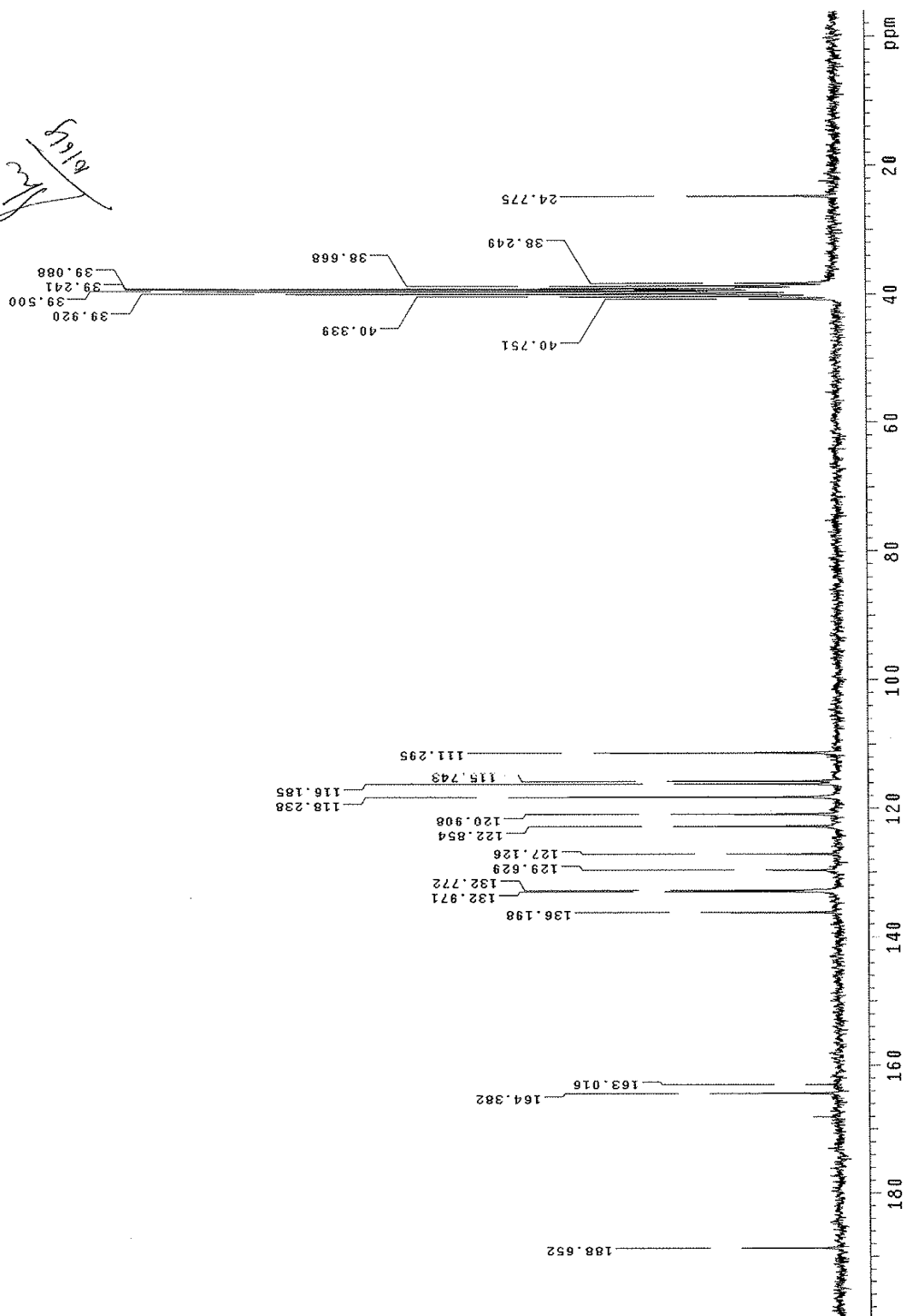
¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-fluorophenyl)-2-oxoacetamide (**9d**):



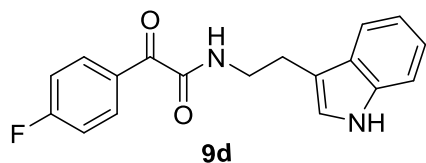
Analytical Research, Discovery Research, DRL
Instrument : Gemini 2000 (Varian 200MHz)
Date & Time : Wed Jun 10 10:07:18 GMT 2009
Recorded By : Shruthi.D

A214/CARS-1/044 in CDCl₃
TDC-219

AR.N0:GE0605/41
Analyst: Shruthi
Date:10th June 2009



Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-fluorophenyl)-2-oxoacetamide (**9d**):



CPS,MIYAPUR

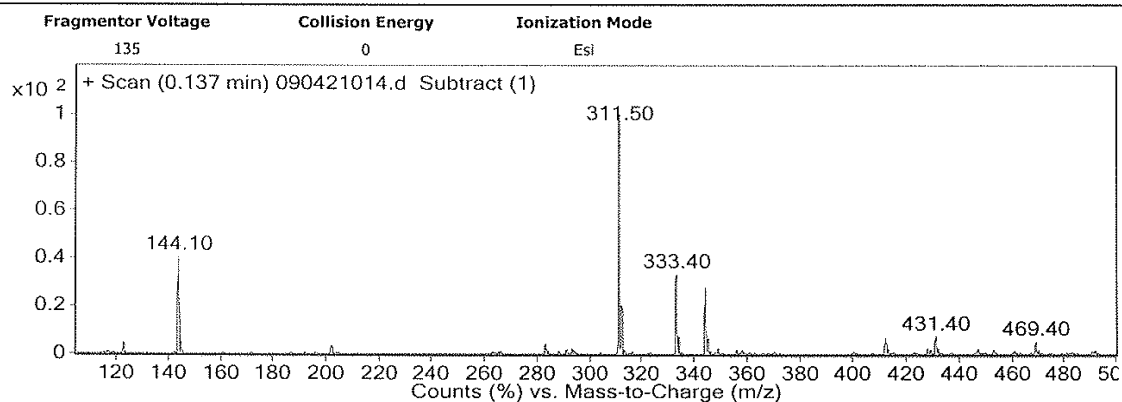
Mass Analysis Report

DATA REPORT

Data Filename 090421014.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method
DA Method Quant Process.m

Sample Name A214/CARS-1/044
Position Vial 53
User Name
IRM Calibration Status **Success**
Comment

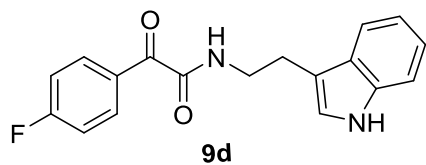
User Spectra



--- End Of Report ---

090421/04/09

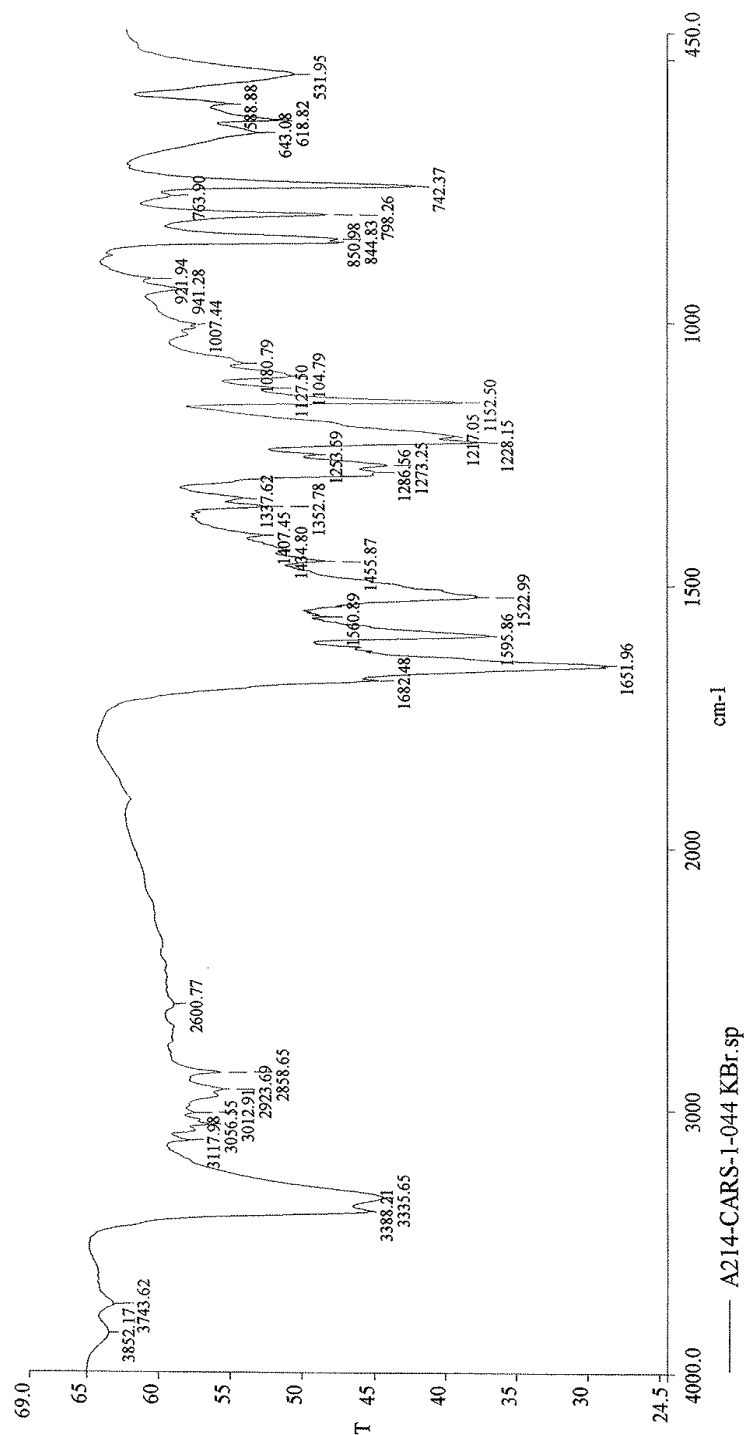
IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-fluorophenyl)-2-oxoacetamide (**9d**):



Date: 6/2/09
Time: 3:10:13 PM

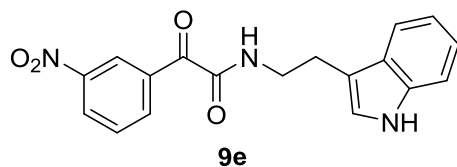
DR.REDDY'S LABORATORIES LIMITED

TDC/CCS-ANALYTICAL RESEARCH .



Analyst: JHANSI
SIGN: *[Signature]* 21/09/2009

¹H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(3-nitrophenyl)-2-oxoacetamide (**9e**):



AR4D, Aurigene Discovery Technologies Ltd, Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Tue Mar 9 14:35:24 IST 2010

Recorded By : Srikanth.A

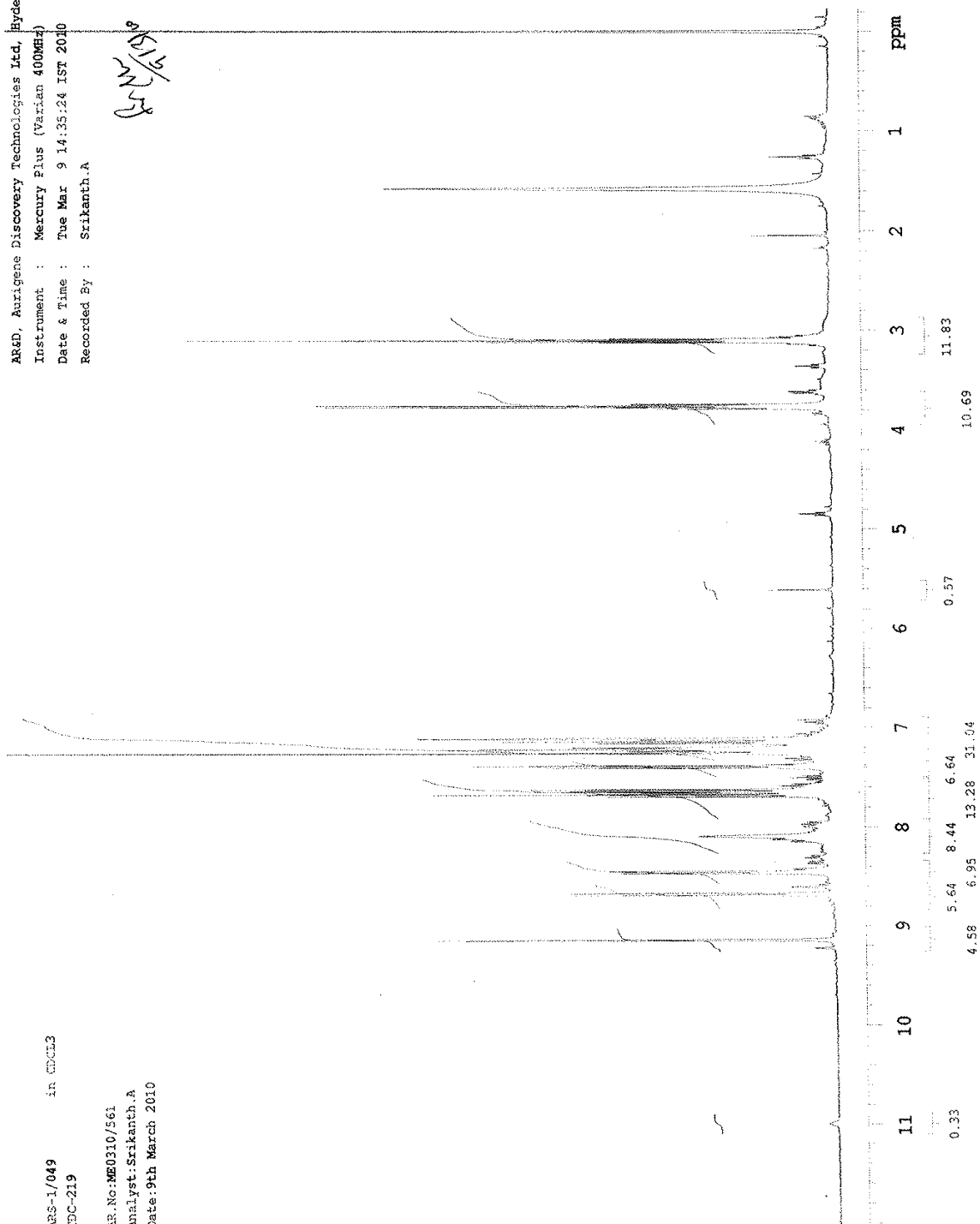
ARS-1/049 in CDCl₃

TDC-219

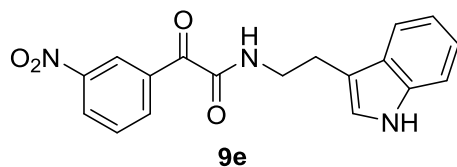
AR.No:ME0310/561

Analyst: Srikanth.A

Date: 9th March 2010

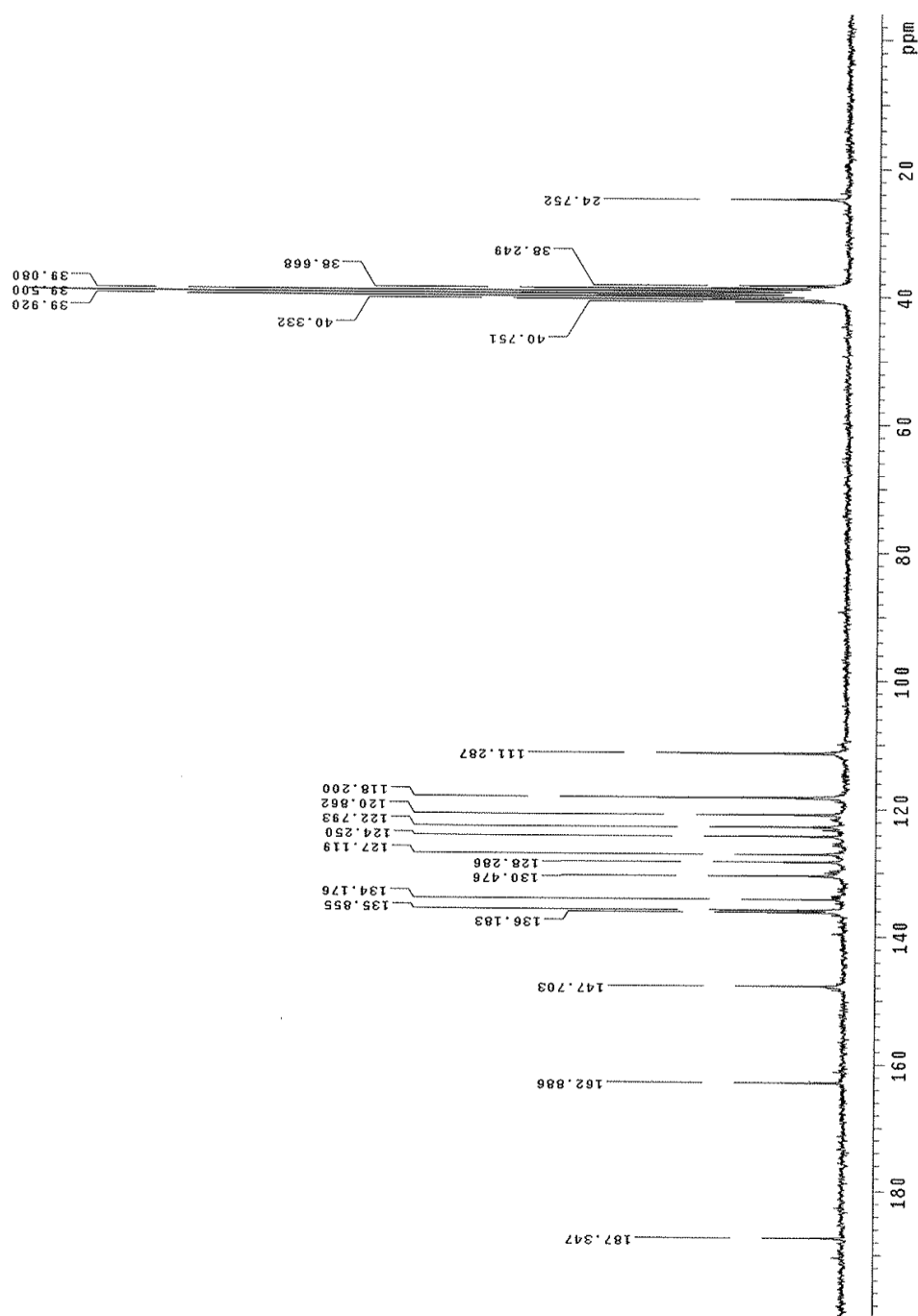


¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(3-nitrophenyl)-2-oxoacetamide (**9e**):



[Handwritten signature]

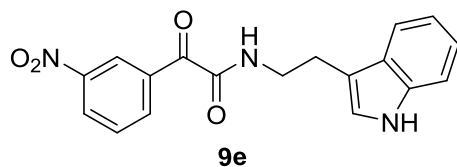
Analytical Research, Discovery Research, DRL
Instrument : Gemini 2000 (Varian 200MHz)
Date & Time : Wed Jun 10 16:46:30 GMT 2009
Recorded By : Shruthi.D



A214/CARS-1/049 in DMSO
TDC-219

AR-NO:GE0603/43
Analyst: Seshu
Date:10th June 2009

Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(3-nitrophenyl)-2-oxoacetamide (**9e**):



CPS,MIYAPUR

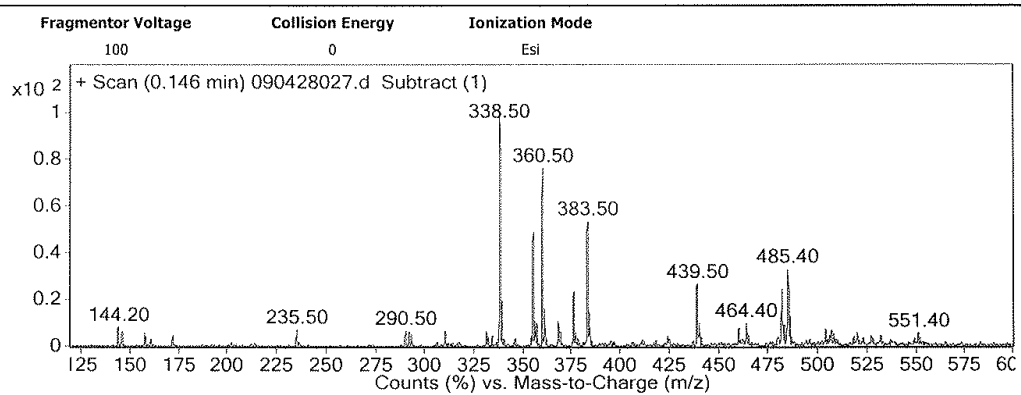
Mass Analysis Report

Y 100.00000

Data Filename 090428027.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method
DA Method Quant Process.m

Sample Name A214/CARS-1/049
Position Vial 82
User Name
IRM Calibration Status Success
Comment

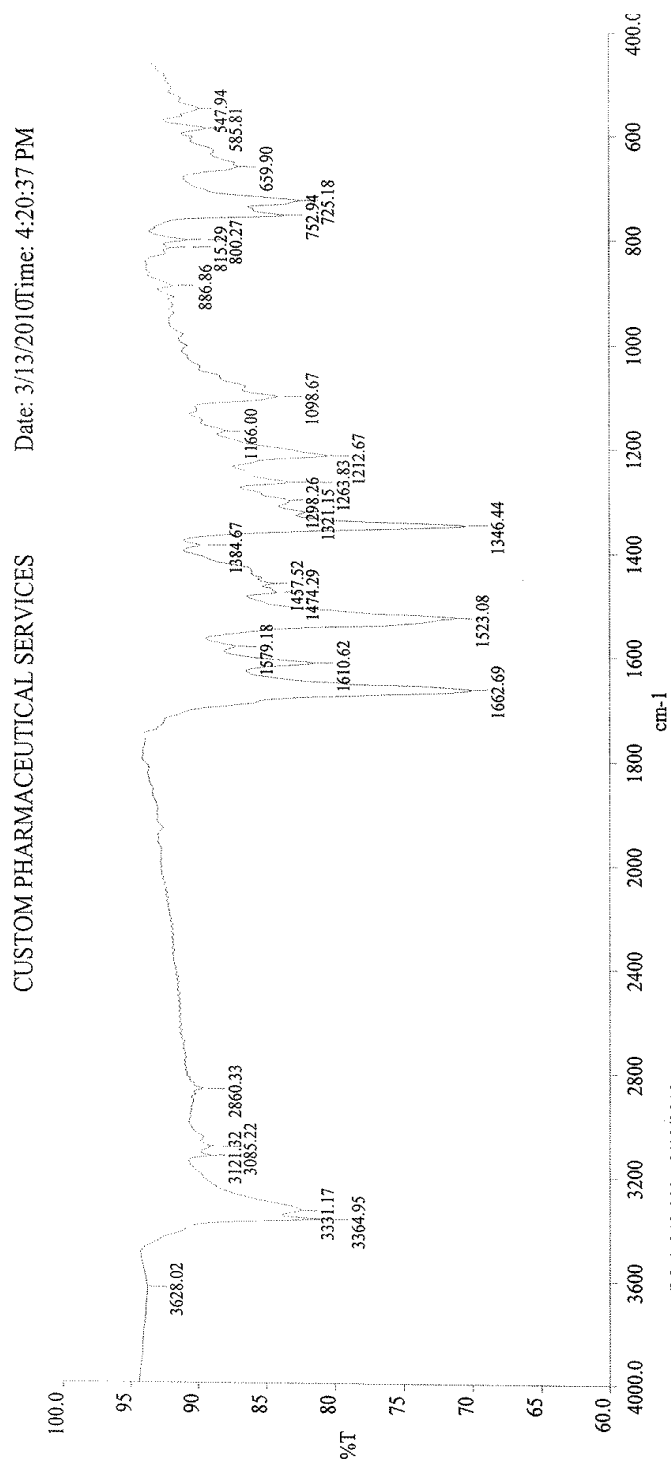
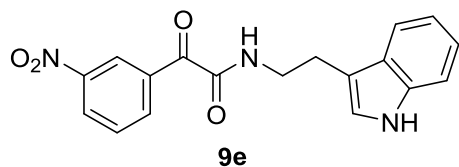
User Spectra



--- End Of Report ---

09/28/09

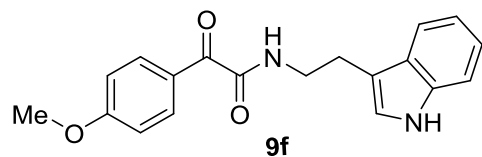
IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(3-nitrophenyl)-2-oxoacetamide (**9e**):



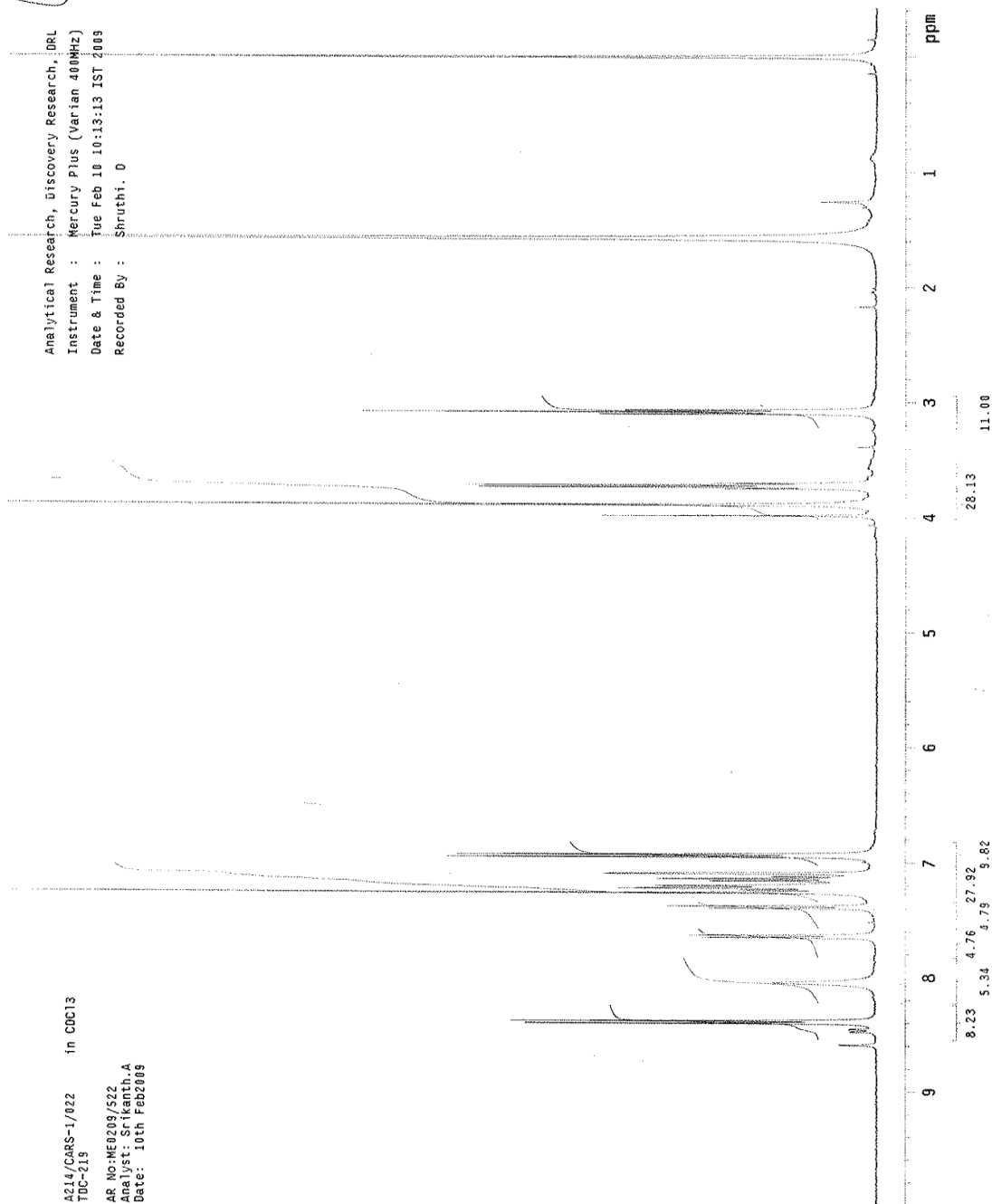
COMPARE REPORT

analyst *[Signature]* 10/3/10

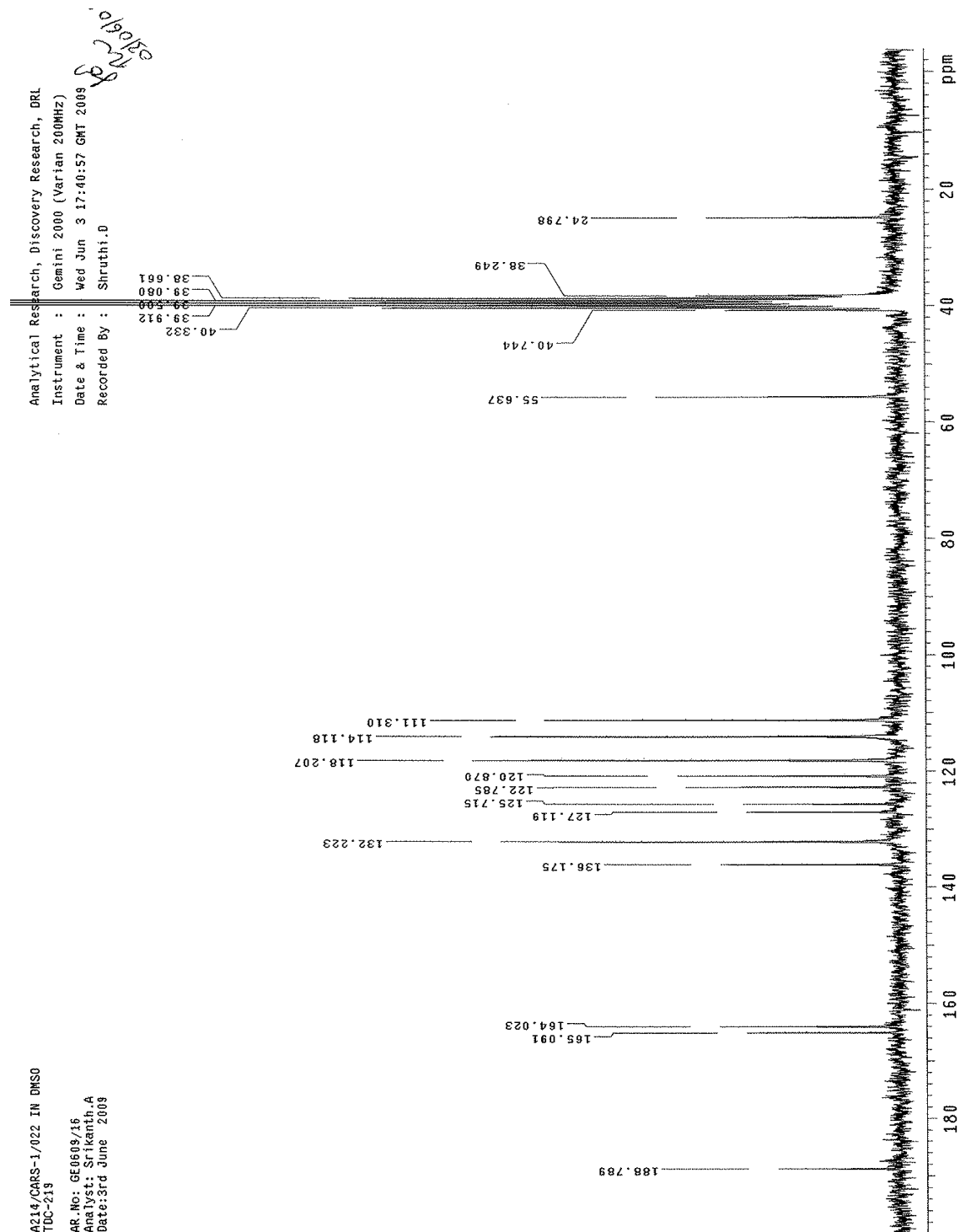
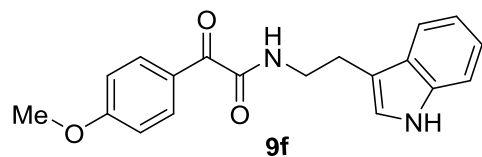
¹H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-methoxyphenyl)-2-oxoacetamide (**9f**):



Handwritten signature

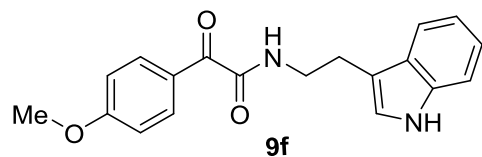


¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-methoxyphenyl)-2-oxoacetamide (**9f**):



A214/CARS-1/022 IN DMSO
 TDC-213
 AR_No: GF0609/16
 Analyst: Srikanth.A
 Date: 3rd June 2009

Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-methoxyphenyl)-2-oxoacetamide (**9f**):



CPS,MIYAPUR

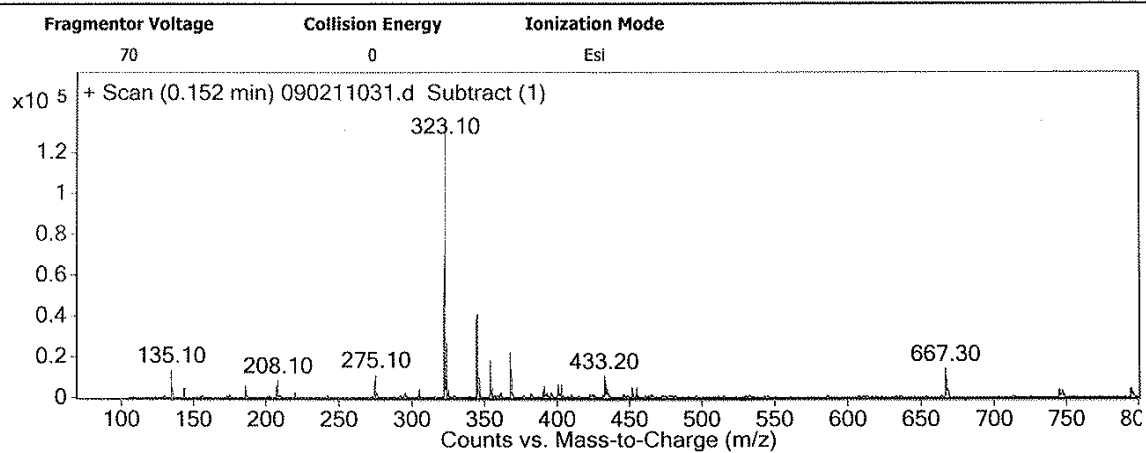
Mass Analysis Report

DR. R. DEEPA

Data Filename 090211031.d
Sample Type Sample
Instrument Name Instrument 1
Acq Method ESI.m
DA Method DA.m

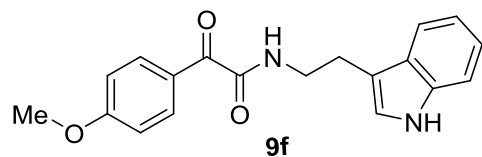
Sample Name A214/CARS/022
Position Vial 30
User Name
IRM Calibration Status Success
Comment

User Spectra



--- End Of Report ---

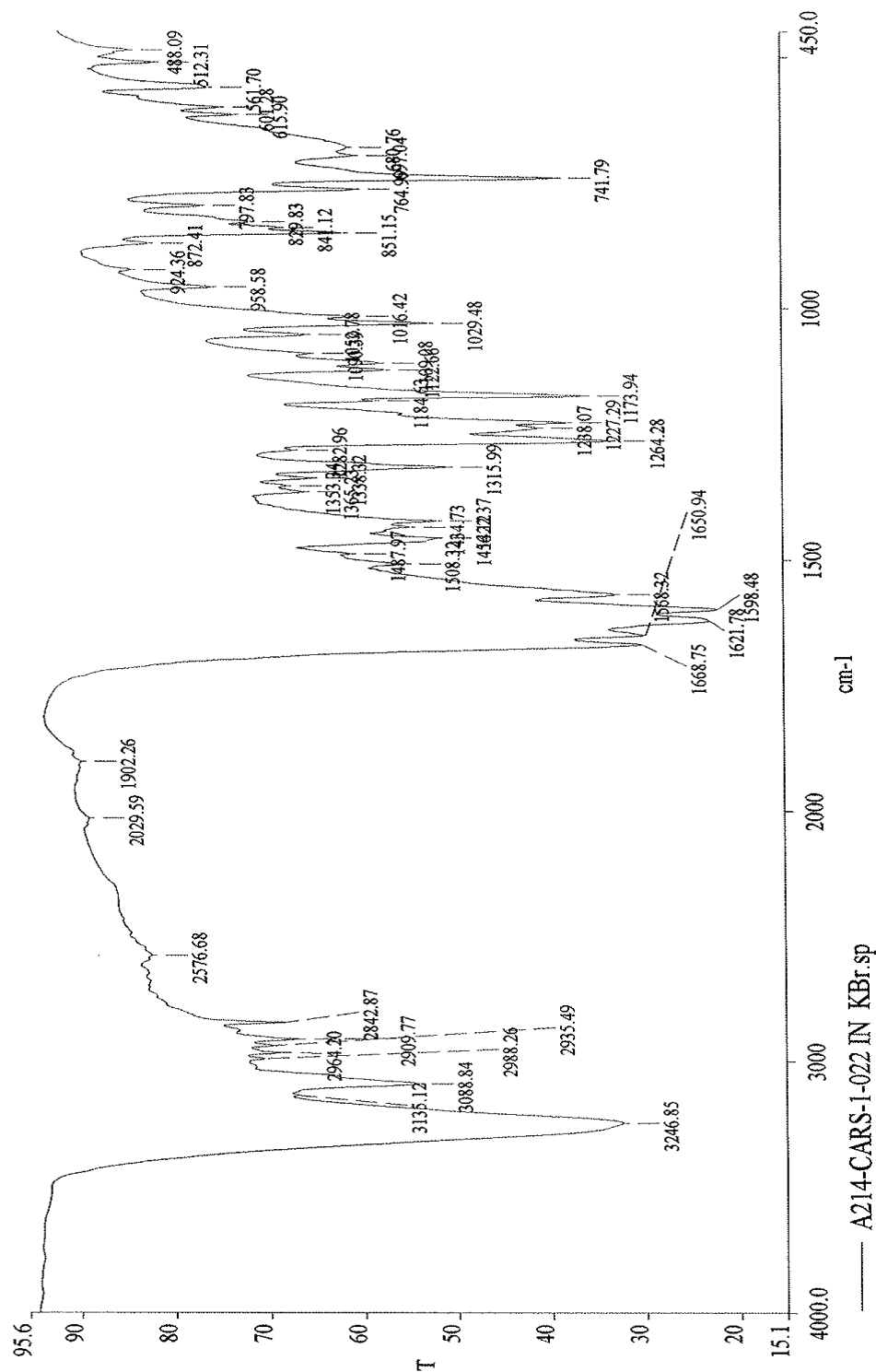
IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-methoxyphenyl)-2-oxoacetamide (**9f**):



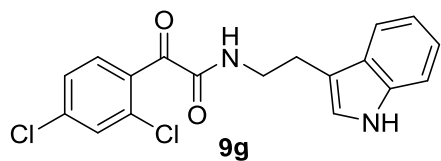
Date: 6/2/09
Time: 3:24:08 PM

DR.REDDY'S LABORATORIES LIMITED

TDC/CCS-ANALYTICAL RESEARCH .



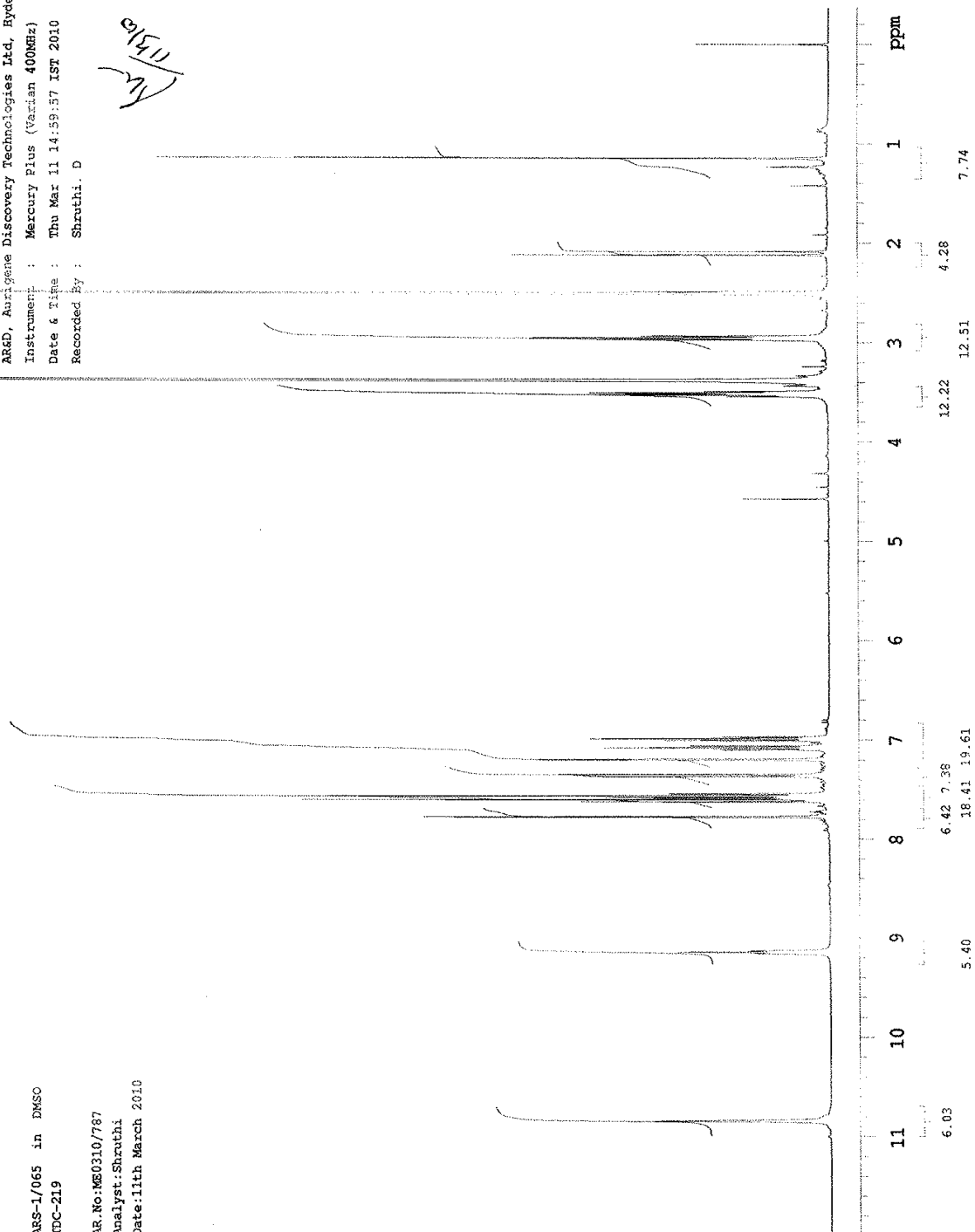
¹H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(2,4-dichlorophenyl)-2-oxoacetamide (**9g**):



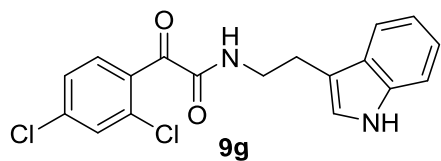
AR&D, Aarigene Discovery Technologies Ltd, Hyderabad
 Instrument : Mercury Plus (Varian 400MHz)
 Date & Time : Thu Mar 11 14:59:57 IST 2010
 Recorded By : Shruthi. D

ARS-1/065 in DMSO
 TDC-219

AR.No:ME0310/787
 Analyst:Shruthi
 Date:11th March 2010



¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(2,4-dichlorophenyl)-2-oxoacetamide (**9g**):



ARAD, Aurigene Discover Technologies Ltd, Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Thu Mar 25 08:54:03 IST 2010

Recorded By : Sridhar

19/3/10
S

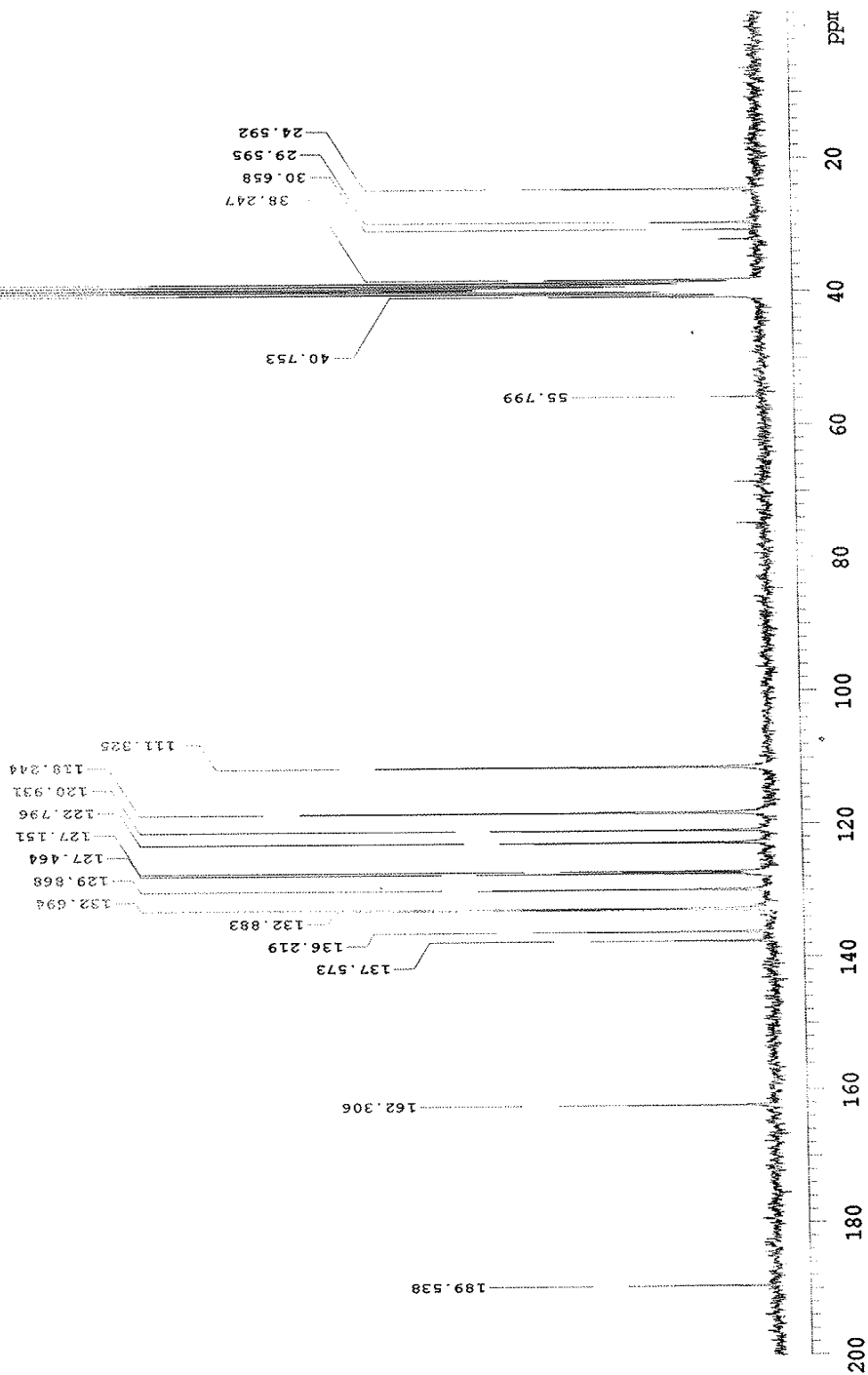
ARS-1/065 in DMSO

TDC-219

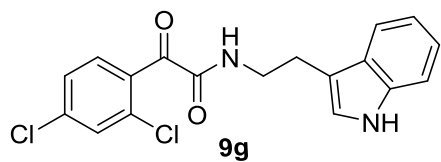
AR.NO:GE0210/67

Analyst: Srikanth.A

Date: 19th March 2010



Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(2,4-dichlorophenyl)-2-oxoacetamide (**9g**):



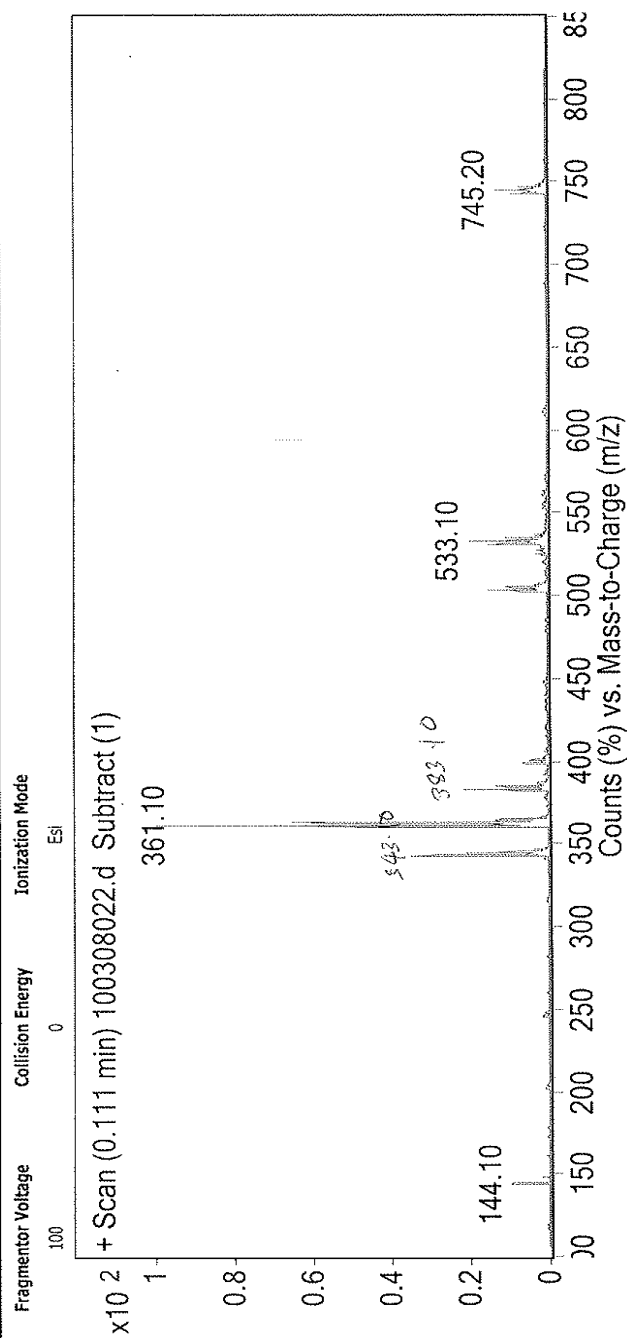
100308022.d

Mass Analysis Report

CPS.MIYAPUR

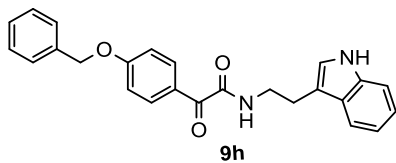
Data Filename	100308022.d	Sample Name	ARS-1/065
Sample Type	Sample	Position	Vial 62
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	default.m	Comment	

User Spectra



--- End Of Report ---

¹H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-(benzyloxy)phenyl)-2-oxoacetamide (**9h**):

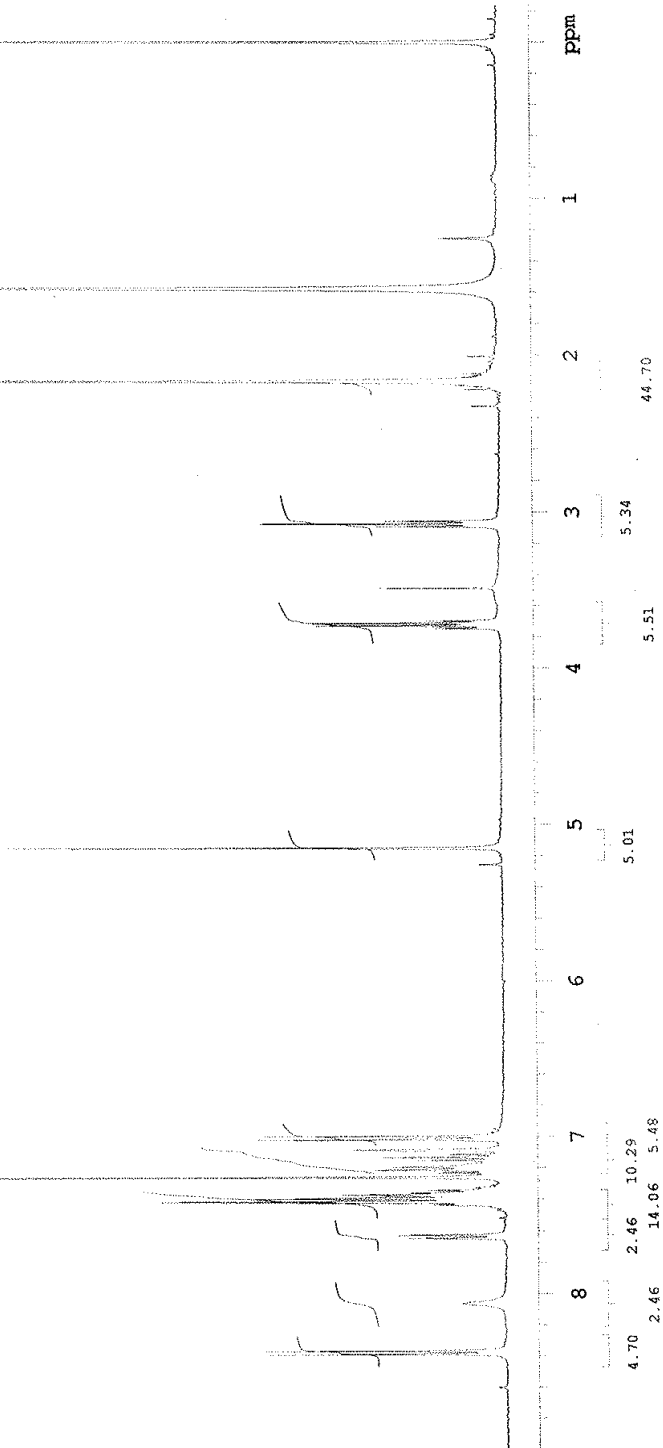


AR&D Aurigene Discovery Technologies Ltd, Hyderabad
 Instrument : Mercury Plus (Varian 400MHz)
 Date & Time : Tue Mar 9 14:13:09 IST 2010
 Recorded By : Srikanth.A

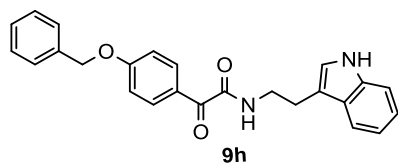
ARS-1/090 in CDCl₃
 TDC-219

AR No: ME0210/558
 Analyst: Srikanth.A
 Date: 9th March 2010

9h



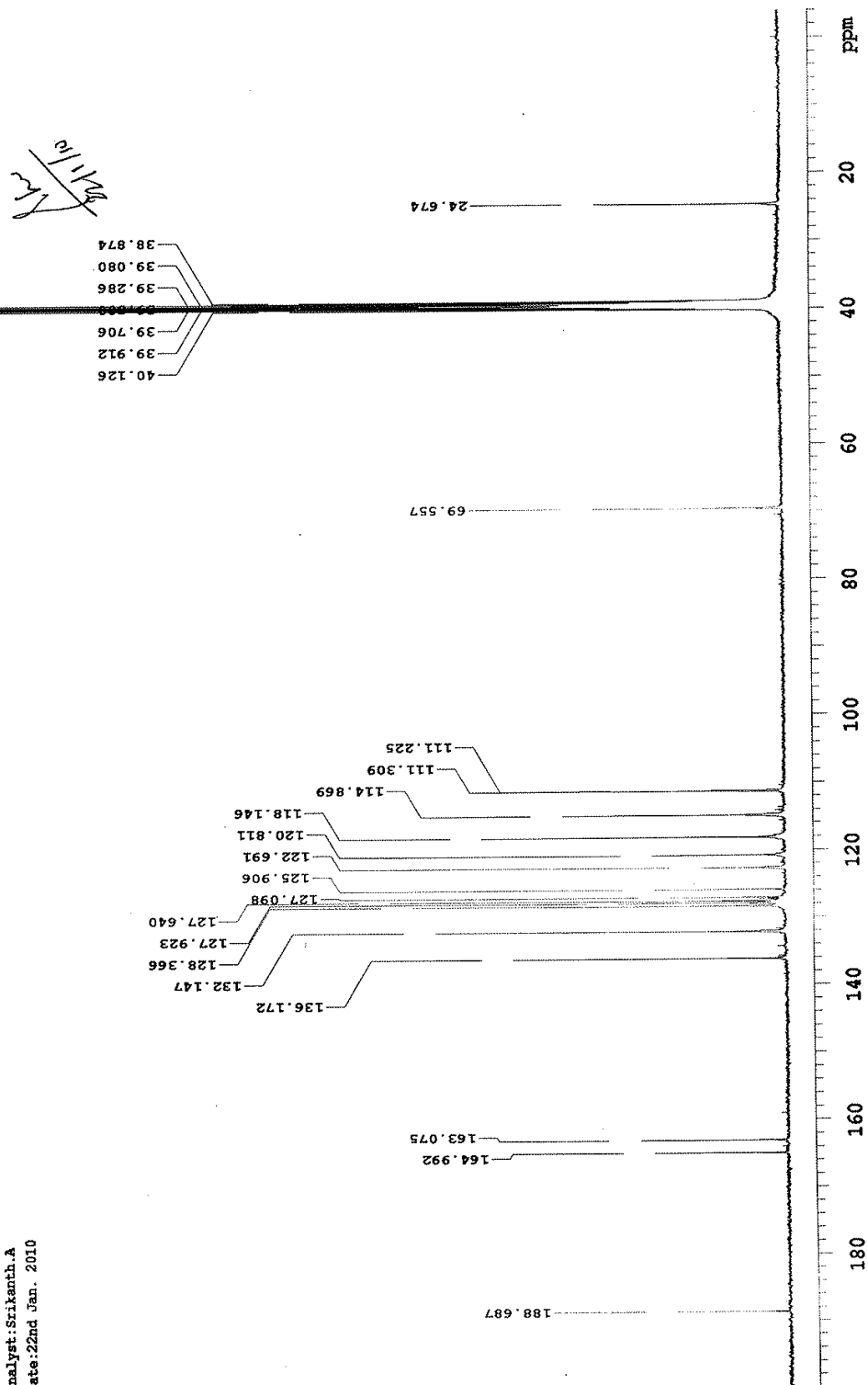
¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-(benzyloxy)phenyl)-2-oxoacetamide (**9h**):



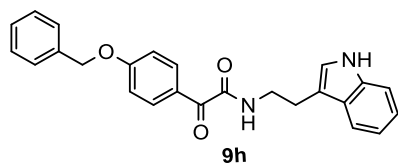
ARd, Aurigene Discovery Technologies Ltd, Hyderabad
 Instrument : Mercury Plus (Varian 400MHz)
 Date & Time : Mon Jan 25 08:49:59 IST 2010
 Recorded By : Srikanth.A

ARS-1/090 Fr2 in DMSO
 TDC-219

AR.No:ME0110/1227
 Analyst:Srikanth.A
 Date:22nd Jan. 2010



Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-(benzyloxy)phenyl)-2-oxoacetamide (**9h**):

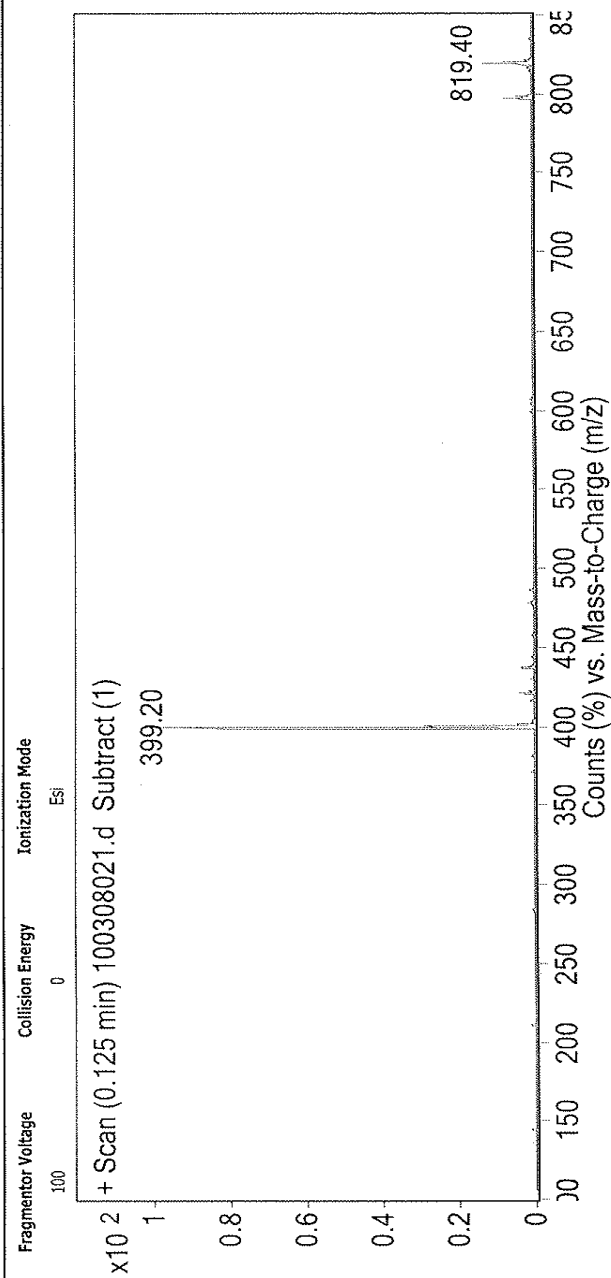


Mass Analysis Report

CPS,MIYAPUR

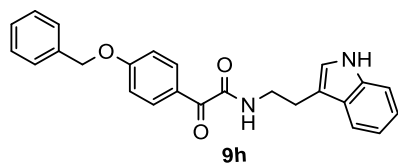
Data Filename	100308021.d	Sample Name	ARS-1/090
Sample Type	Sample	Position	Vial 61
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	default.m	Comment	

User Spectra



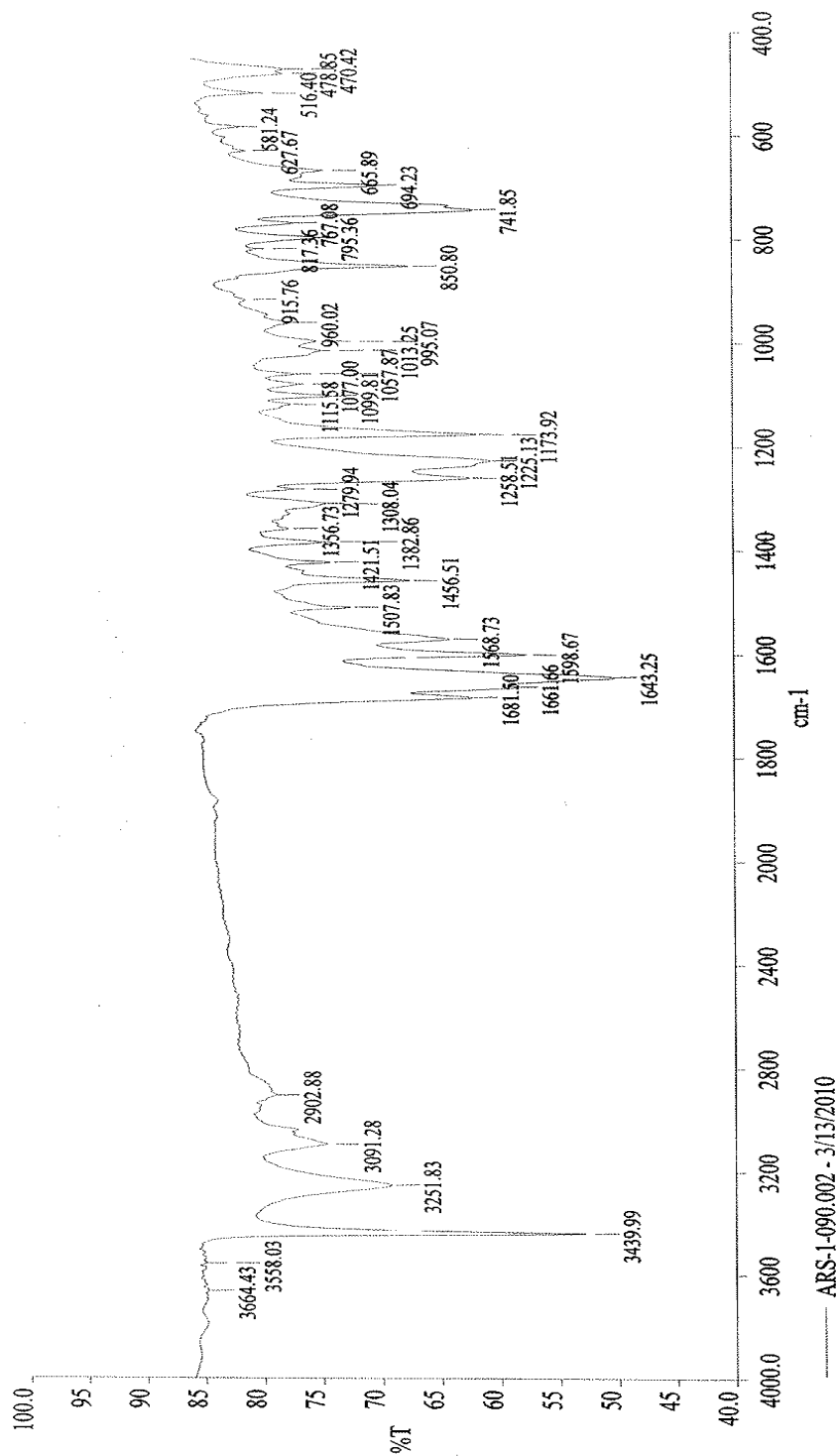
--- End Of Report ---

IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-(benzyloxy)phenyl)-2-oxoacetamide (**9h**):



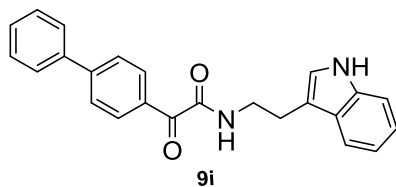
CUSTOM PHARMACEUTICAL SERVICES Date: 3/13/2010 Time: 4:41:54 PM

IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-(4-(benzyloxy)phenyl)-2-oxoacetamide (**9h**):



COMPARE REPORT

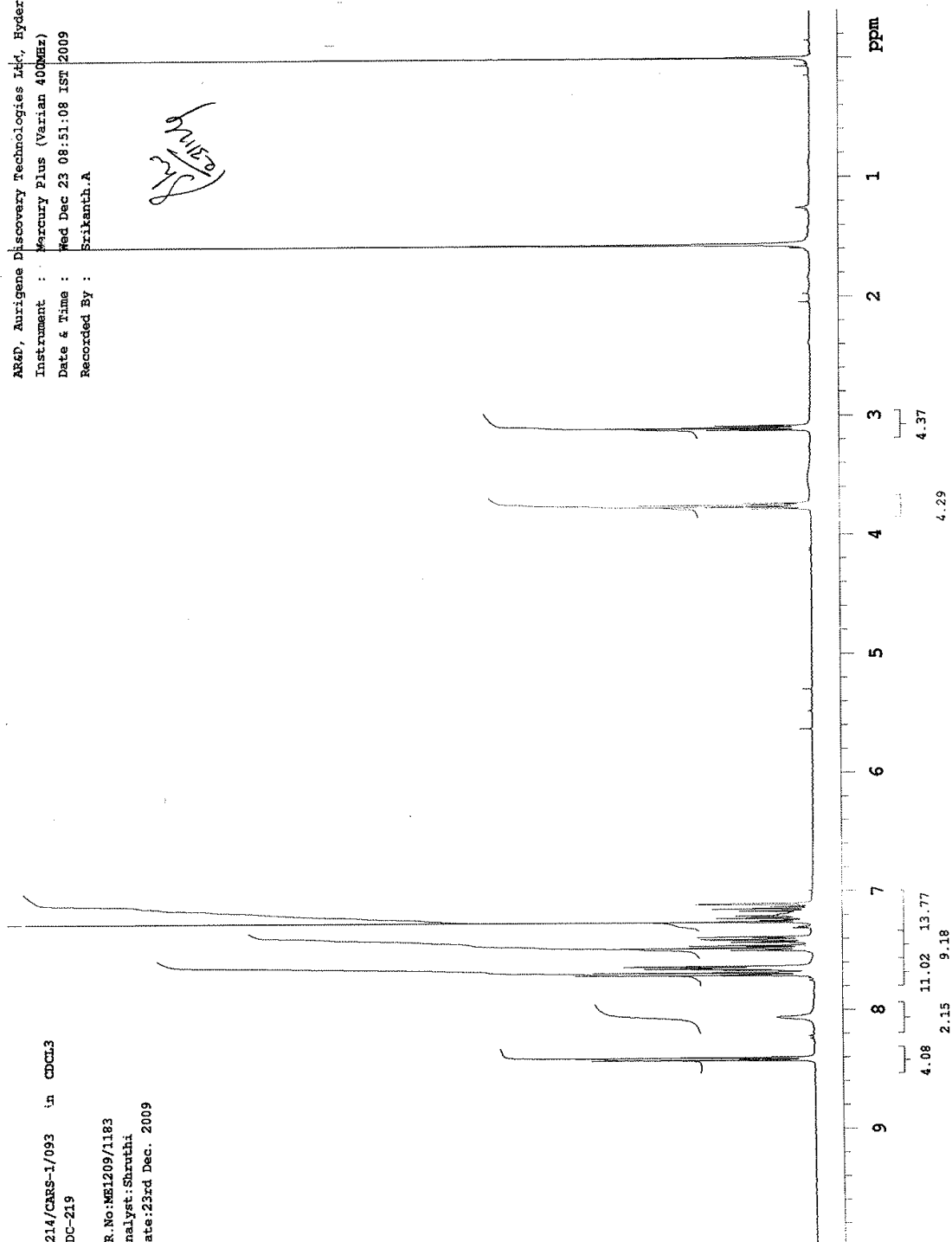
¹H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-([1,1'-biphenyl]-4-yl)-2-oxoacetamide (**9i**):



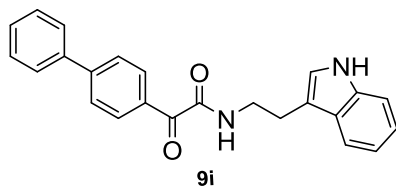
AR&D, Aurigene Discovery Technologies Ltd, Hyderabad
Instrument : Mercury Plus (Varian 400MHz)
Date & Time : Wed Dec 23 08:51:08 IST 2009
Recorded By : Srikanth.A

A214/CARS-1/093 in CDCl₃
TDC-219

AR No: ME1209/1183
Analyst: Shruthi
Date: 23rd Dec. 2009



¹³C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-([1,1'-biphenyl]-4-yl)-2-oxoacetamide (**9i**):



AB&D, Aurigene Discovery Technologies Ltd, Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Thu Mar 25 08:54:29 IST 2010

Recorded By : Shruthi

ARS-1/093 in DMSO

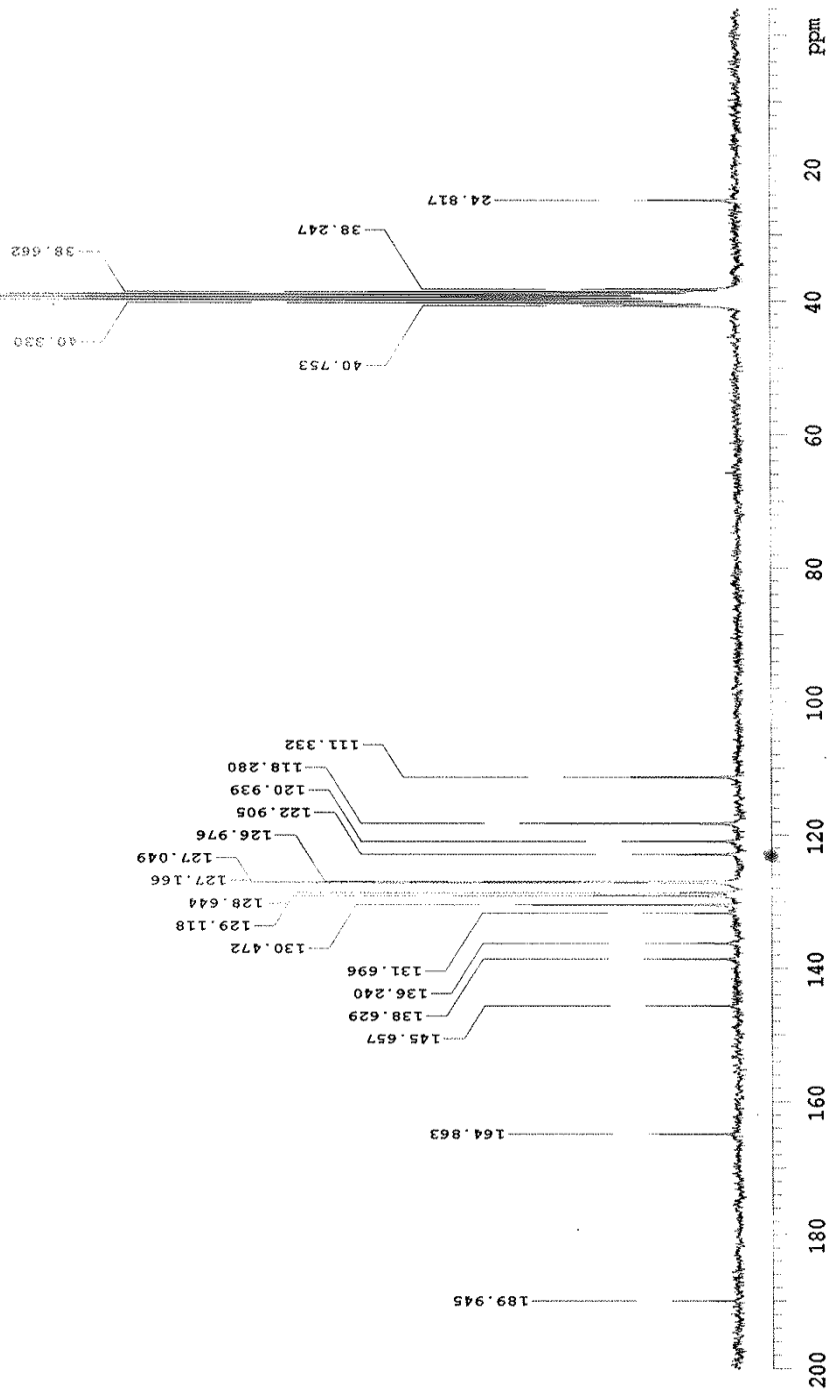
TDC-219

AR NO: GE0210/68

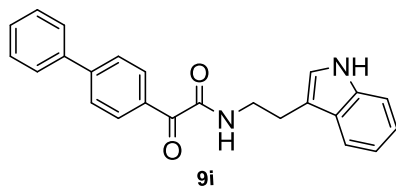
Analyst: Shruthi

Date: 19th March 2010

16



Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-([1,1'-biphenyl]-4-yl)-2-oxoacetamide (**9i**):

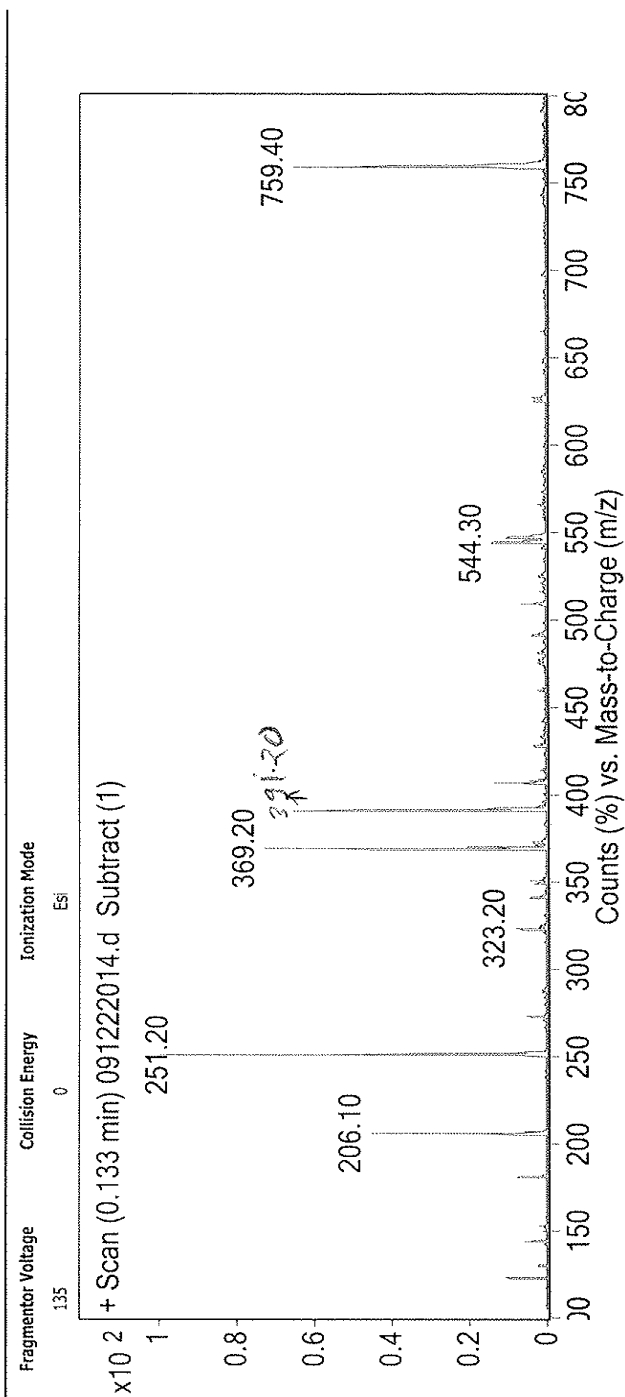


CPS,MIYAPUR

Mass Analysis Report

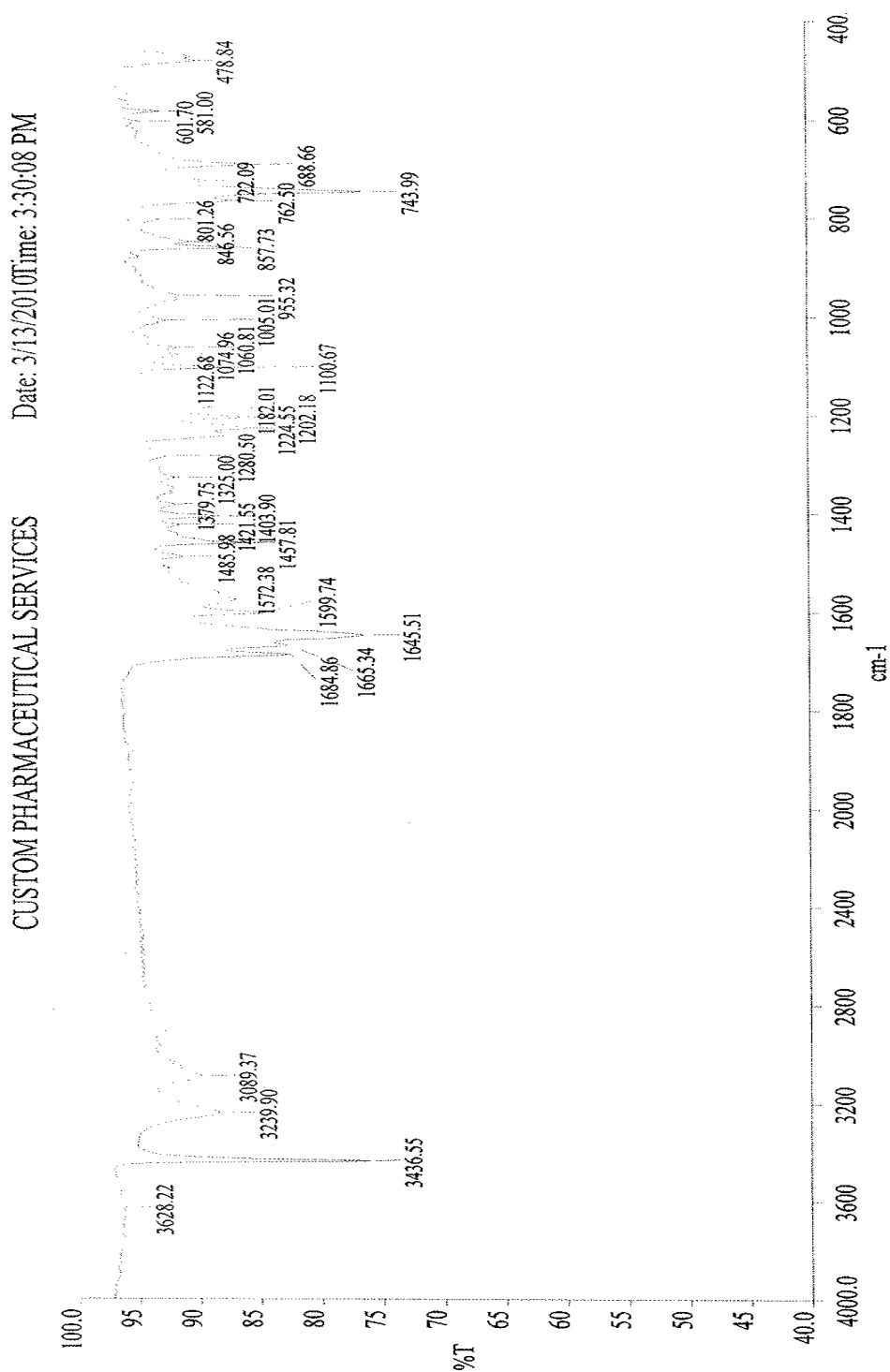
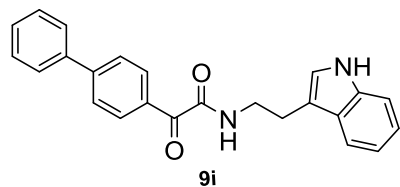
Data Filename	091222014.d	Sample Name	A214/CARS-1/093
Sample Type	Sample	Position	Vial 44
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	default.m	Comment	

User Spectra



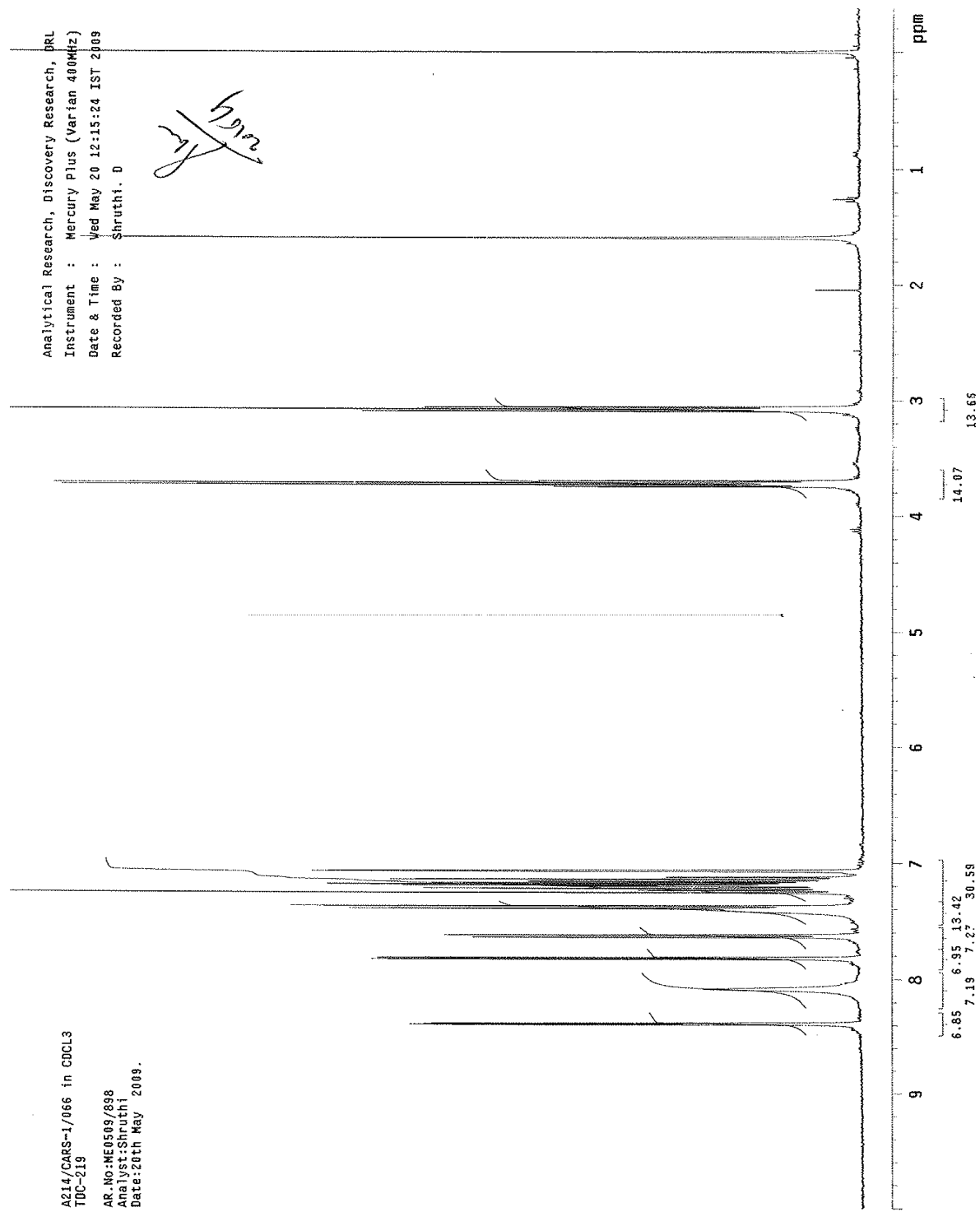
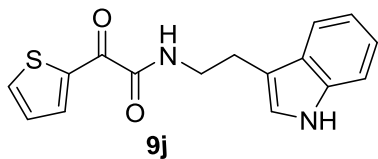
--- End Of Report ---

IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-([1,1'-biphenyl]-4-yl)-2-oxoacetamide (**9i**):



COMPARE REPORT

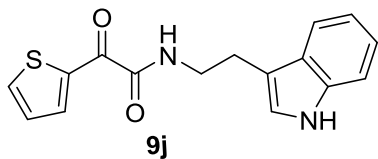
^1H NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(thiophen-2-yl)acetamide (**9j**):



A214/CARS-1/066 in CDCl₃
 TDC-219

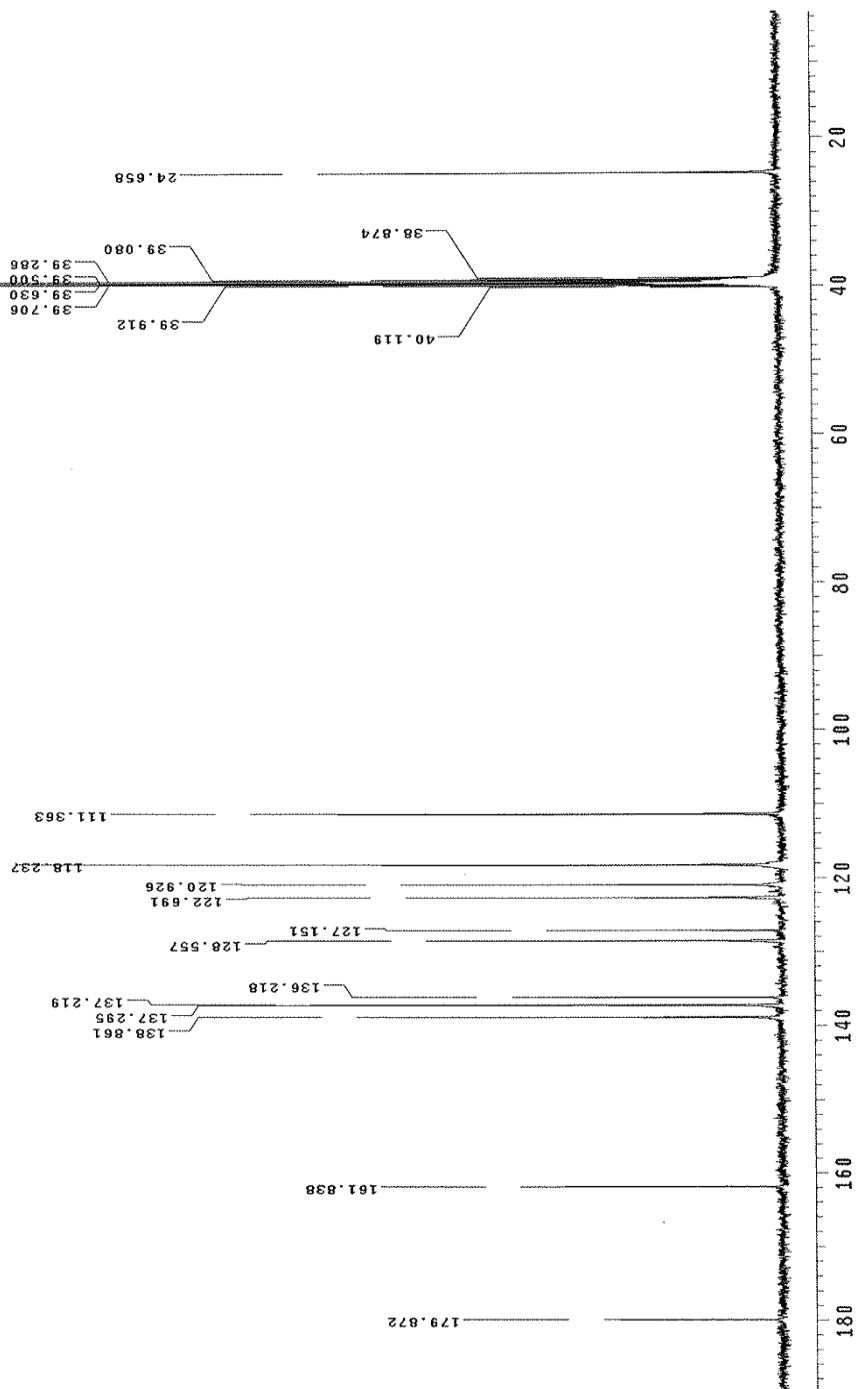
AR-NO-ME0509/898
 Analyst: Shruthi
 Date: 20th May 2009.

^{13}C NMR of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(thiophen-2-yl)acetamide (**9j**):



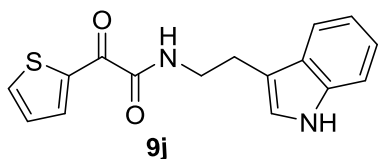
Handwritten signature

Analytical Research, Discovery Research, DRL
Instrument : Mercury Plus (Varian 400MHz)
Date & Time : Wed Jul 15 13:43:42 IST 2009
Recorded By : Shruthi. D



A214/CARS-1/066 in DMSO
TDC-219
AR.No:ME0709/743
Analyst:Shruthi
Date:15th July 2009.

Mass spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(thiophen-2-yl)acetamide (**9j**):



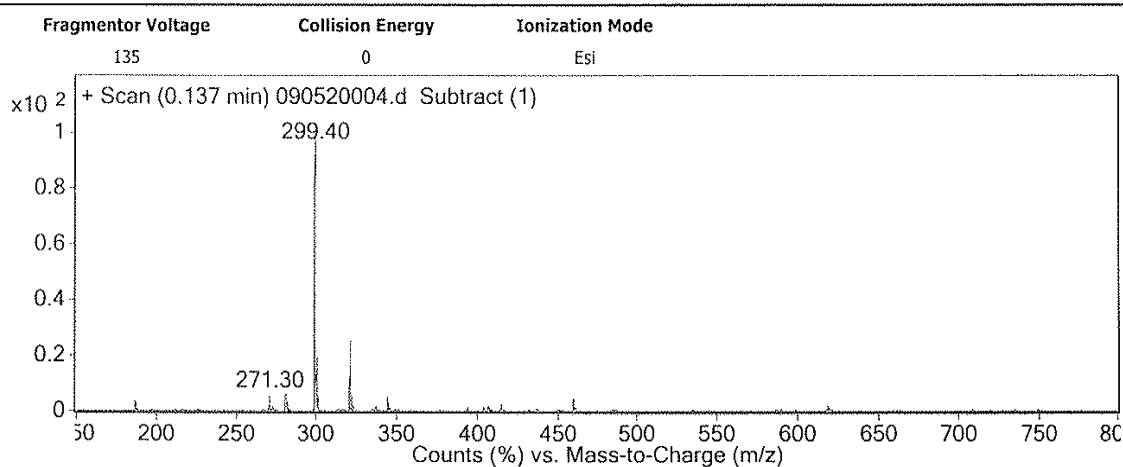
CPS,MIYAPUR

Mass Analysis Report

Y. Du, 2019/11/11

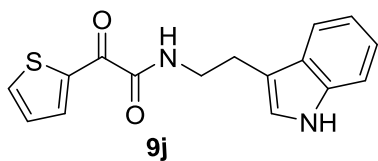
Data Filename	090520004.d	Sample Name	A214/CARS-1/066 FR-1
Sample Type	Sample	Position	Vial 54
Instrument Name	Instrument 1	User Name	
Acq Method		IRM Calibration Status	Success
DA Method	Quant Process.m	Comment	

User Spectra



--- End Of Report ---

IR spectrum of *N*-(2-(1*H*-indol-3-yl)ethyl)-2-oxo-2-(thiophen-2-yl)acetamide (**9j**):

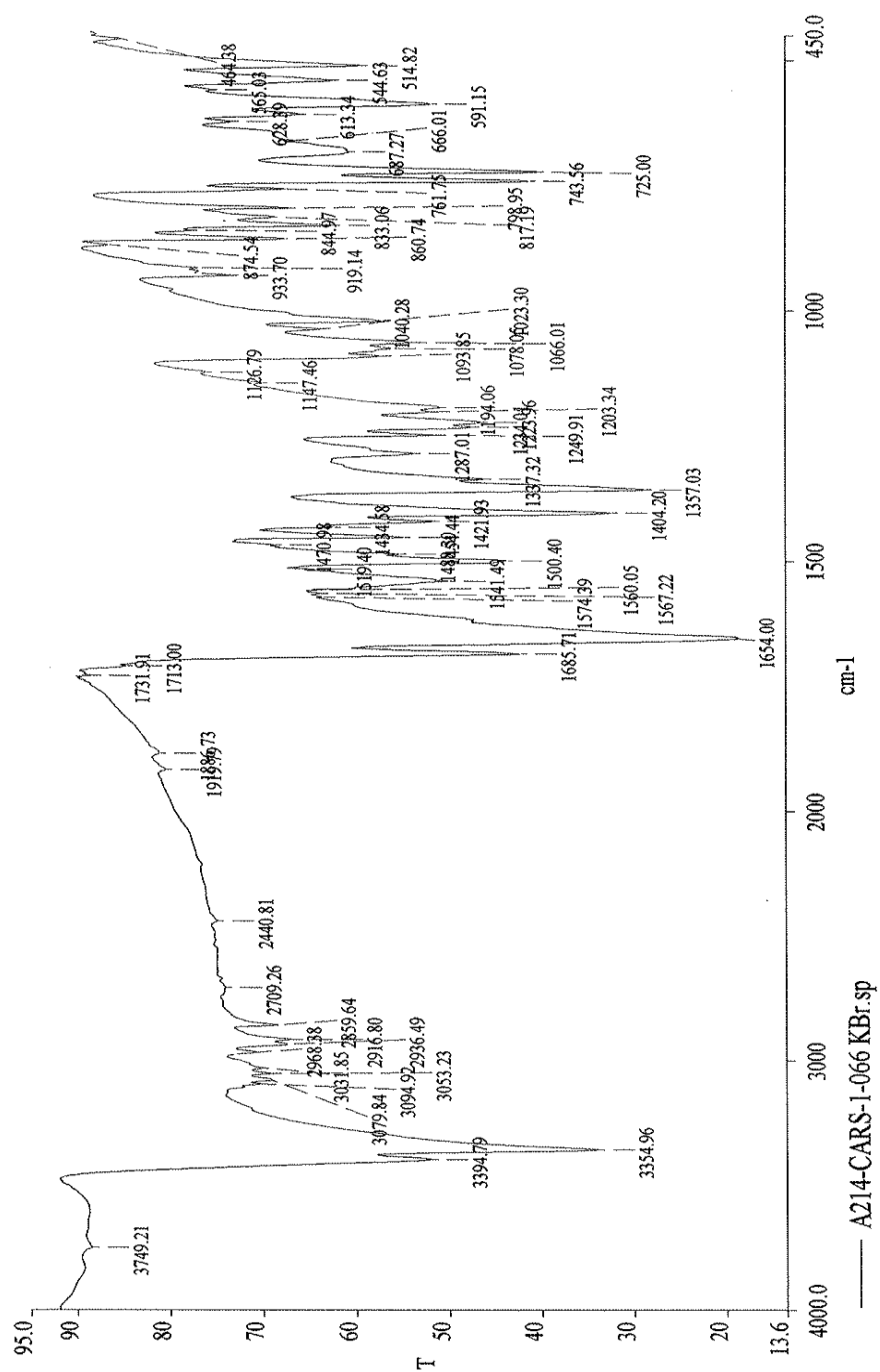


Date: 6/2/09

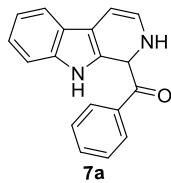
Time: 2:56:27 PM

DR.REDDY'S LABORATORIES LIMITED

TDC/CCS-ANALYTICAL RESEARCH.



¹H NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**7a**):

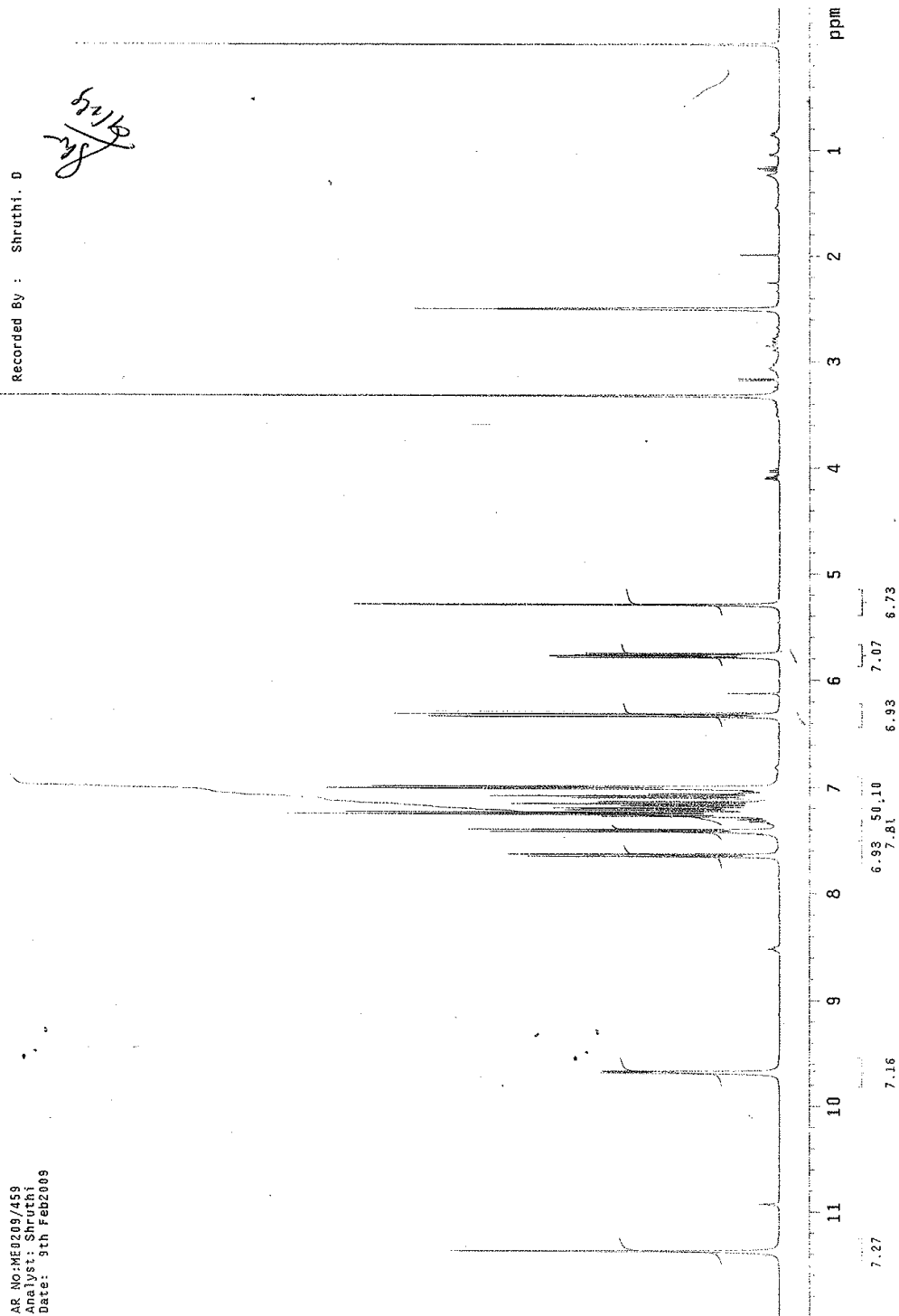


Analytical Research, Discovery Research, DRL
Instrument : Mercury Plus (Varian 400MHz)
Date & Time : Mon Feb 9 12:32:28 IST 2009
Recorded By : Shruthi. D

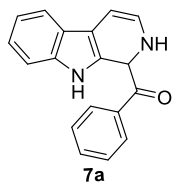
A214/CARS-2/025 in DMSO
T0C-219

AR NO:ME0209/459
Analyst: Shruthi
Date: 9th Feb2009

Shruthi



¹³C NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**7a**):

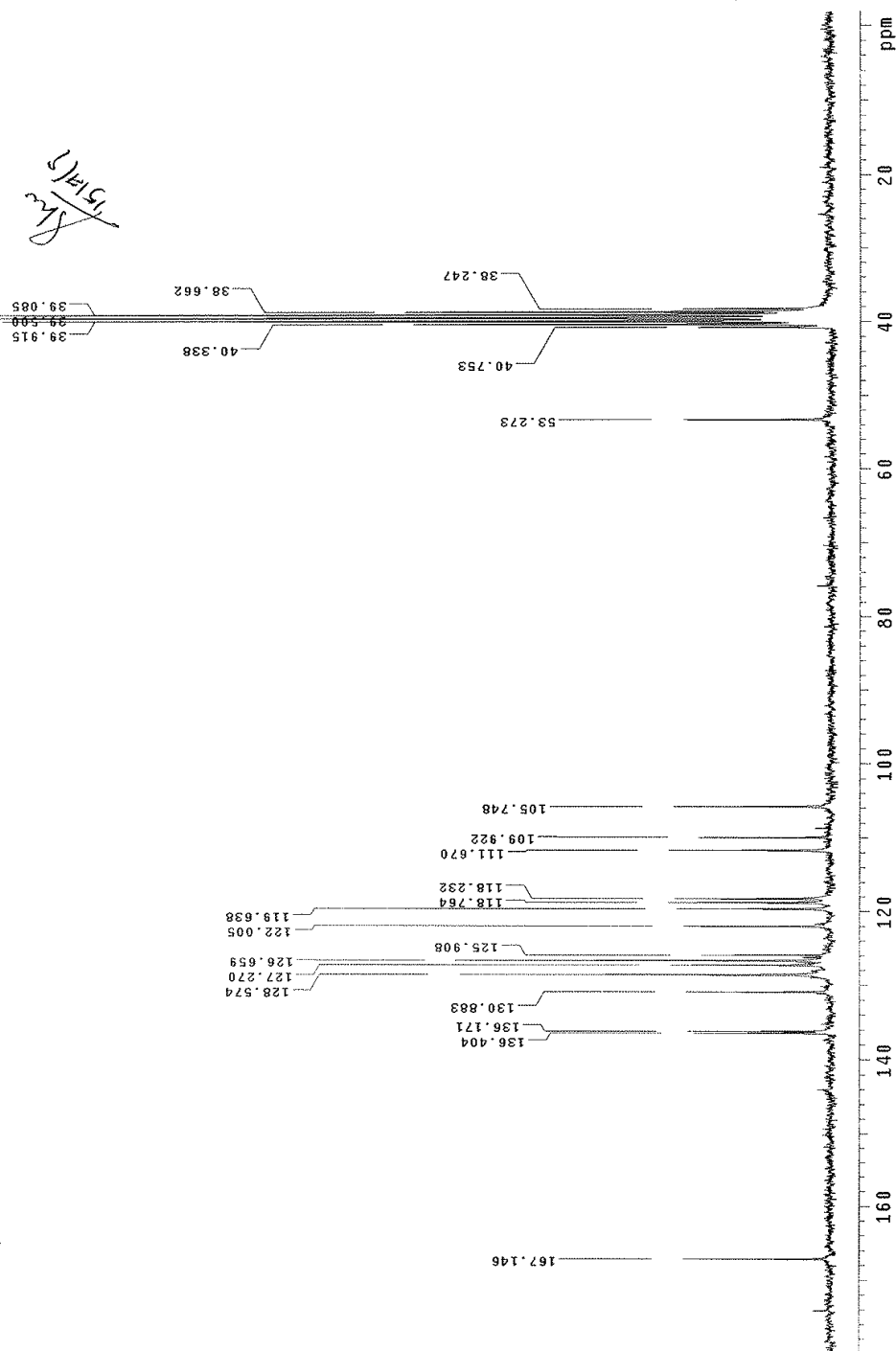


Analytical Research, Discovery Research, DRU
 Instrument : Gemini 2000 (Varian 200MHz)
 Date & Time : Wed Jul 15 10:59:38 GMT 2009
 Recorded By : Shruthi.D

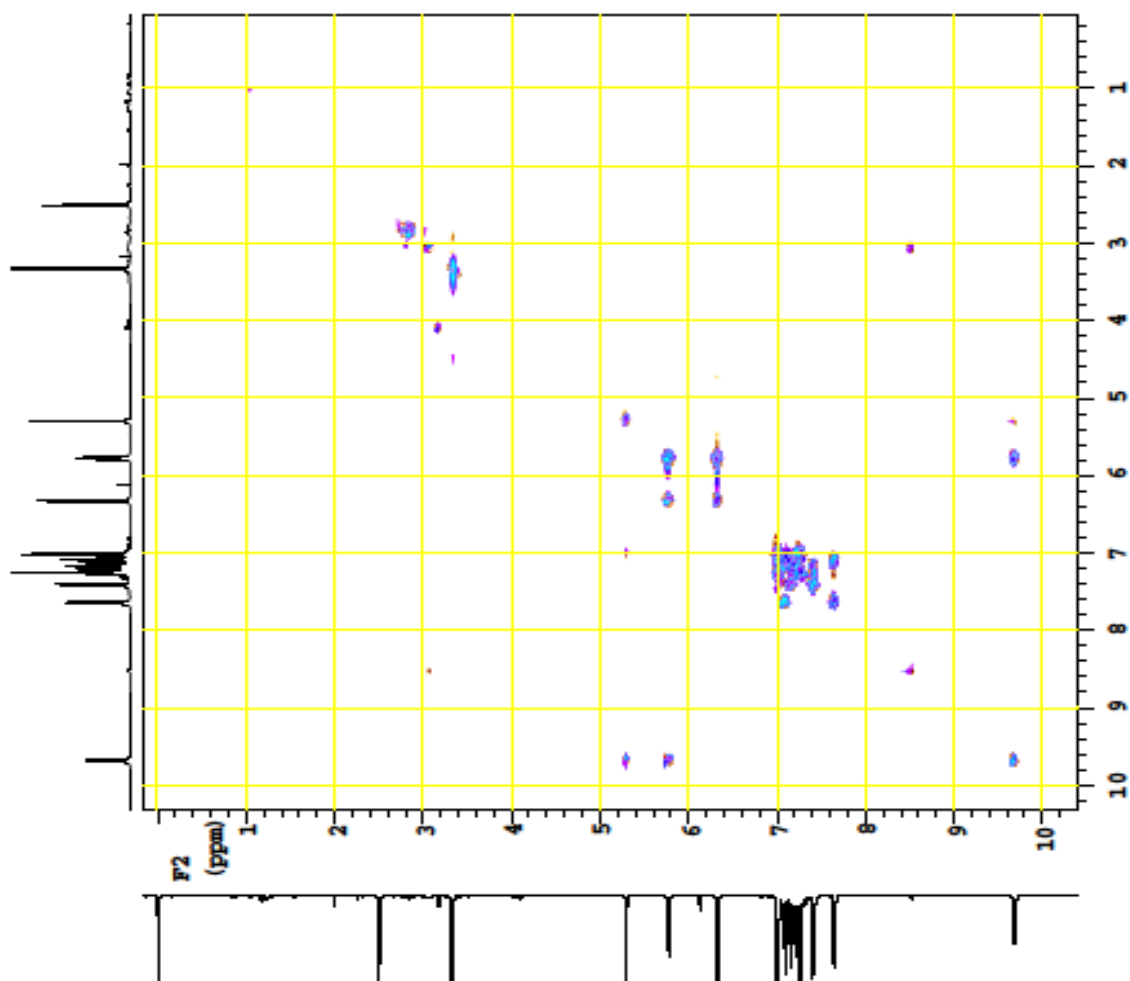
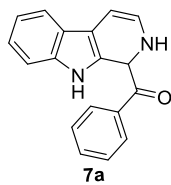
15/7/09
 Shruthi.D

A214/CARS2/025 in DMSO
 TDC-213

AP NO: GF0709/51
 Analyst: Srikanth.A
 Date: 15th July 2009



COSY spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**7a**):



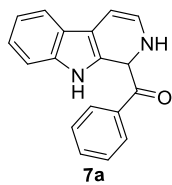
```

AS14/CARB2/025 IN DMSO
TDC-219

MR.NO:MBD309/265
Analyst: Sashu
Date: 6th March 2009

exp2 gq000.fv
=====
SAMPLE          FLAGS
date Mar 6 2009  ha      nn
solvent dmsc      aspl    y
sample          beglv1 1200
=====
ACQUISITION
sw 4950.5 temp 25.0
at 0.150 gain 30
ap 14.00 spin not used
fb not used f2 PROCESSING
sa 32 ab -0.125
dl 1.000 sbs -0.100
nt 32 lsfid -15
=====
2D ACQUISITION fn 2048
sw1 4950.5 f1 PROCESSING
ni 256 gfl 0.025
=====
PREPARATION gfl not used
satosa n procl lp
wet n fnl 2048
=====
TRANSMITTER DISPLAY
tn H1 ap -62.8
sfrc 400.225 wp 4220.5
tof 358.1 ap1 24.2
tpwr 60 wp1 4104.5
pw 8.900 rfl 164.4
=====
GRADIENTS rfp 0
gr1v1e 8.06 rfl1 164.4
gfm 0.002500 rfp1 0
gatab 0.000500 PLOT
=====
DECOUPLER wc 139.3
dn C13 ac 10.0
da nmw wc2 139.3
sc2 0
vs 10577
th ai cdc ph 2
  
```

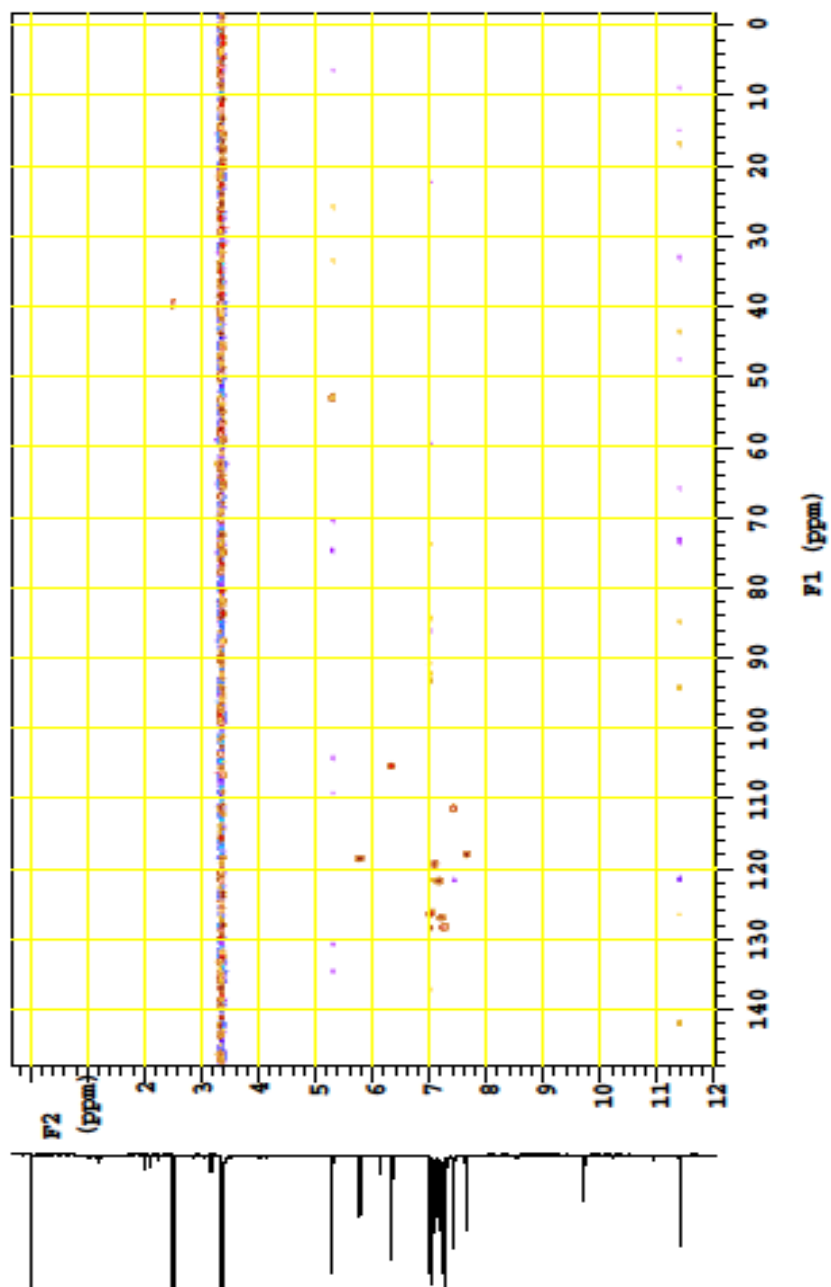
HSQC spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**7a**):



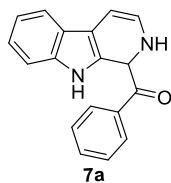
114/CN022/025 IN INDO
PC-219

1. NO INDO309/4.00
solvent: DMSO
date: 10th March 2009

NAME :
ID (MS):
IP :
HSQC



Mass spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**7a**):



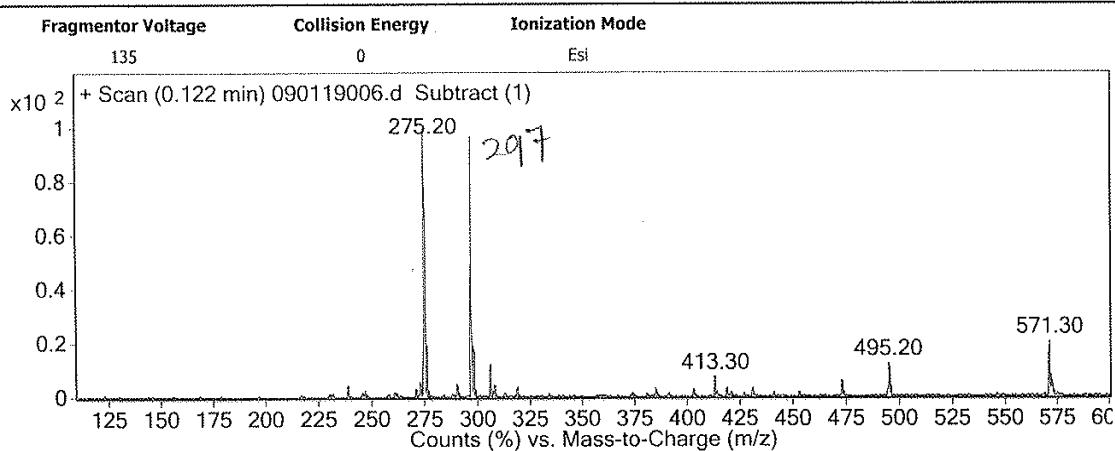
CPS,MIYAPUR

Mass Analysis Report

EXTRAORDINARY

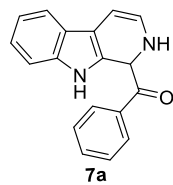
Data Filename	090119006.d	Sample Name	A214/CARS-2/016 RC
Sample Type	Sample	Position	Vial 5
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	DA.m	Comment	

User Spectra



--- End Of Report ---

HRMS of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**7a**):



Page 1

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

96 formula(e) evaluated with 1 results within limits (up to 4 best isotopic matches for each mass)

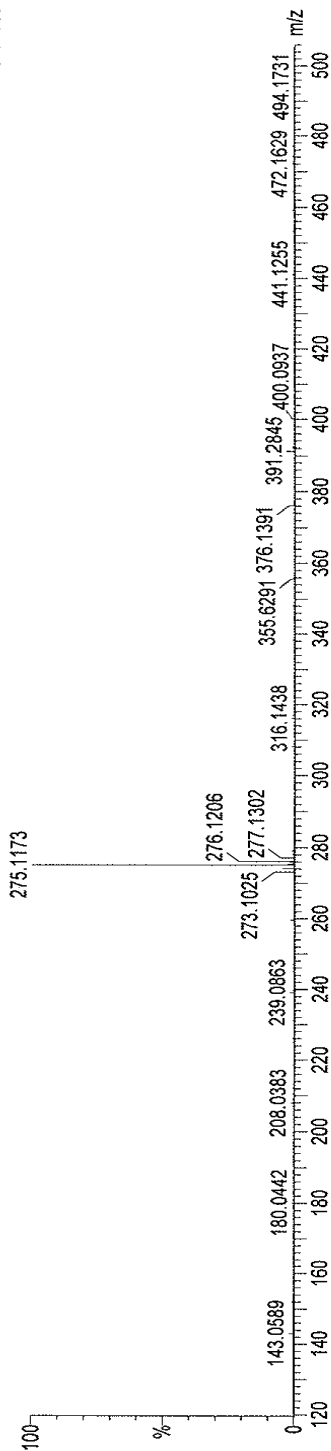
Elements Used:

C: 0-30 H: 0-30 N: 0-5 O: 0-5

9a

UT1212_148 10 (0.366) Cm (10:13-(14+22:28)x0.010)

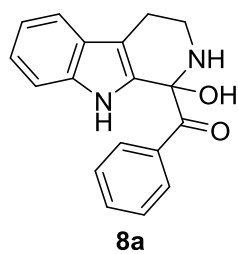
Gajanan
1: TOF MS ES+
7.37e+003



Minimum: -1.5
Maximum: 80.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
275.1173	275.1184	-1.1	-4.0	12.5	54.7	C18 H15 N2 O

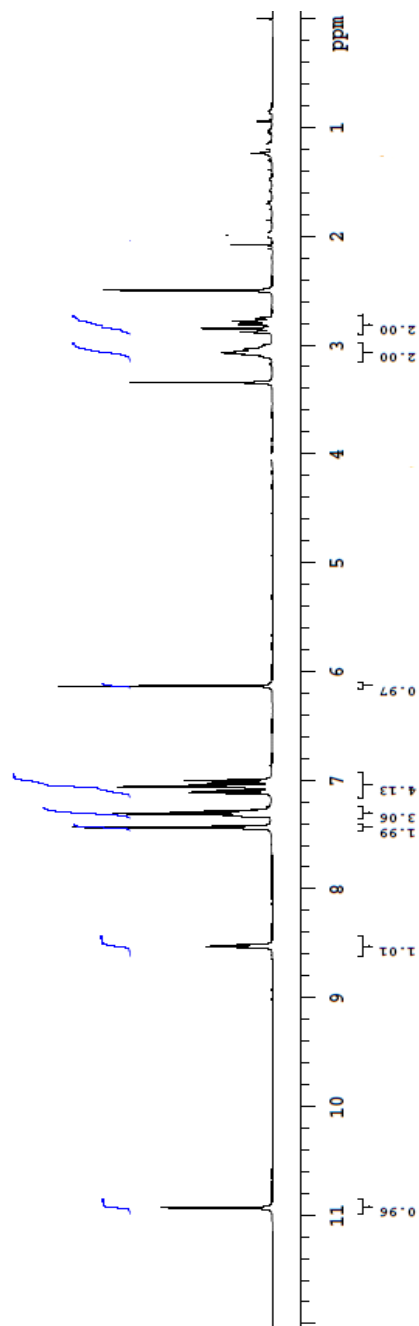
^1H NMR of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**8a**):



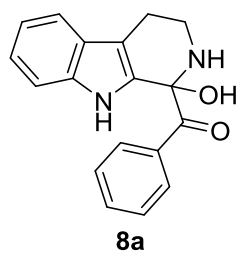
A214/CARS-2/016 Pt-1 in DMSO
TDC-206

RR.No:IN0114/05
Date: 04th Jan.2014
Analyst:Haribabu

NUCLEUS : ^1H
FREQ (MHz): 499.63
EXP : s2pul



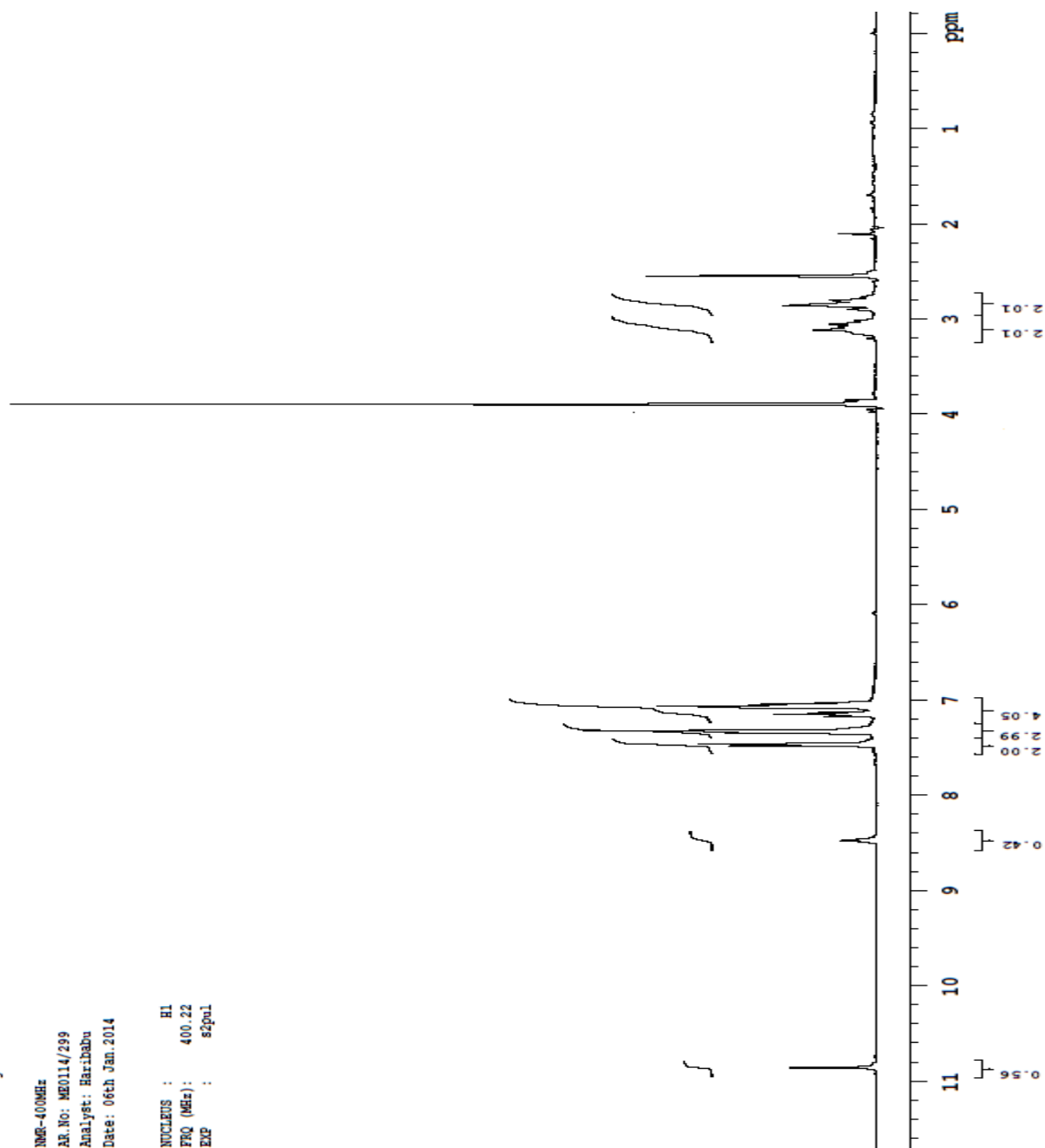
^1H NMR D_2O exchange of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**8a**):



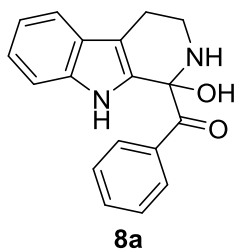
TDC-206 A214/CARS-2/016 Fr-1 in DMSO
 D_2O Exchange

NMR-400MHz
 AR.No: ME0114/299
 Analyst: Haribabu
 Date: 06th Jan.2014

NUCLEUS : ^1H
 FREQ (MHz): 400.22
 EXP : zgpg30



COSY spectrum of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**8a**):

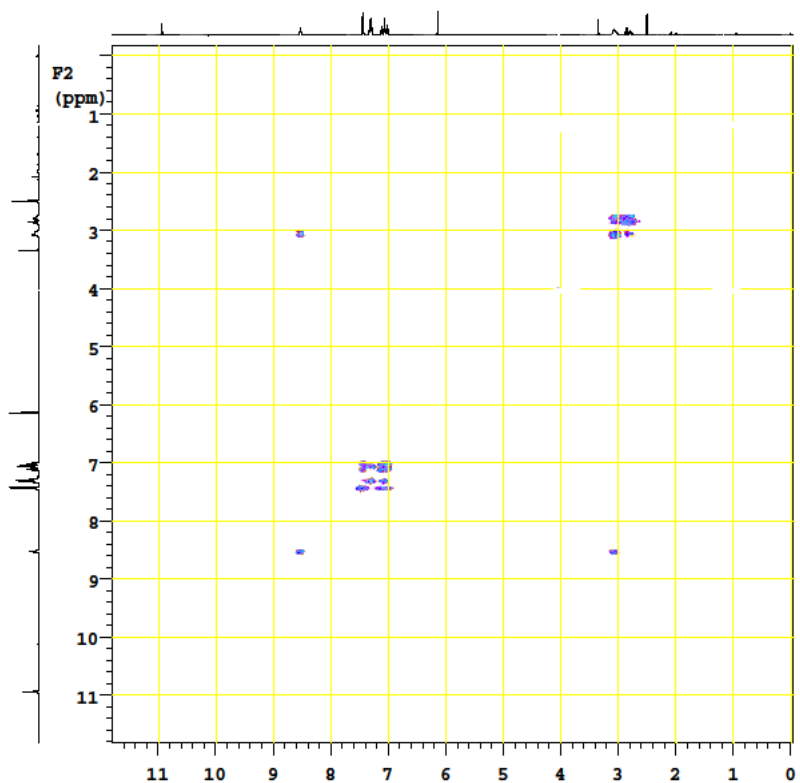


A214/CARS-2/016 Fr-1 in DMSO
TDC-206

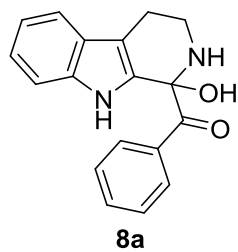
AR.No:IN0114/06
Date: 04th Jan.2014
Analyst:Haribabu

exp2 gDQCOSY

SAMPLE		FLAGS	
date	Jan 4 2014	hs	nn
solvent	DMSO	sspul	y
sample		hsglvt	4237
ACQUISITION		SPECIAL	
sw	7107.3	temp	25.0
at	0.144	gain	16
np	2048	spin	not used
fb	not used	F2 PROCESSING	
ss	32	sb	-0.108
dl	1.000	sbs	-0.072
nt	8	lsfid	-22
2D ACQUISITION		fn	
sw1	7107.3	F1 PROCESSING	
ni	400	ab1	-0.056
TRANSMITTER		abs1	-0.025
tn	H1	gfl	0.024
sfrq	499.629	gfs1	not used
tof	530.1	procl	lp
tpwr	58	fn1	2048
pw	7.200	DISPLAY	
GRADIENTS		sp	-90.5
gslv11	3177.5	wp	5989.9
gt1	0.002500	sp1	-21.1
gslv12	6355	wp1	5913.5
gt2	0.002500	rf1	1776.8
gstab	0.000500	rfl	1249.1
DECOUPLER		rfl1	1776.8
dn	C13	rfp1	1249.1
dm	nnn	PLOT	
		wc	138.9
		sc	10.0
		wc2	138.9
		sc2	0
		vs	66
		th	2
		at	cdh nh



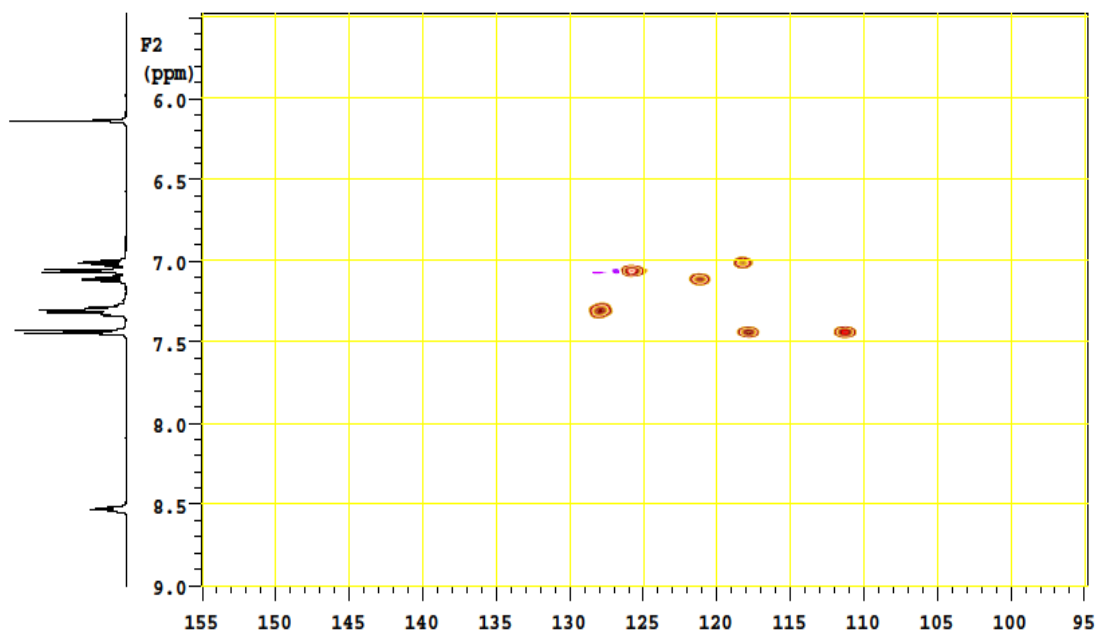
HSQC spectrum of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**8a**):



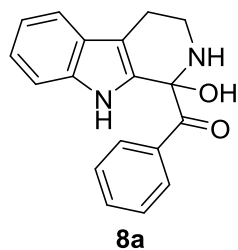
A214/CARS-2/016 Fr-1 in DMSO
TDC-206

AR.No:IN0114/07
Date: 04th Jan.2014
Analyst:Haribabu

NUCLEUS : H1
PRQ (MHz): 499.63
EXP : gHSQC



NOESY spectrum of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**8a**):

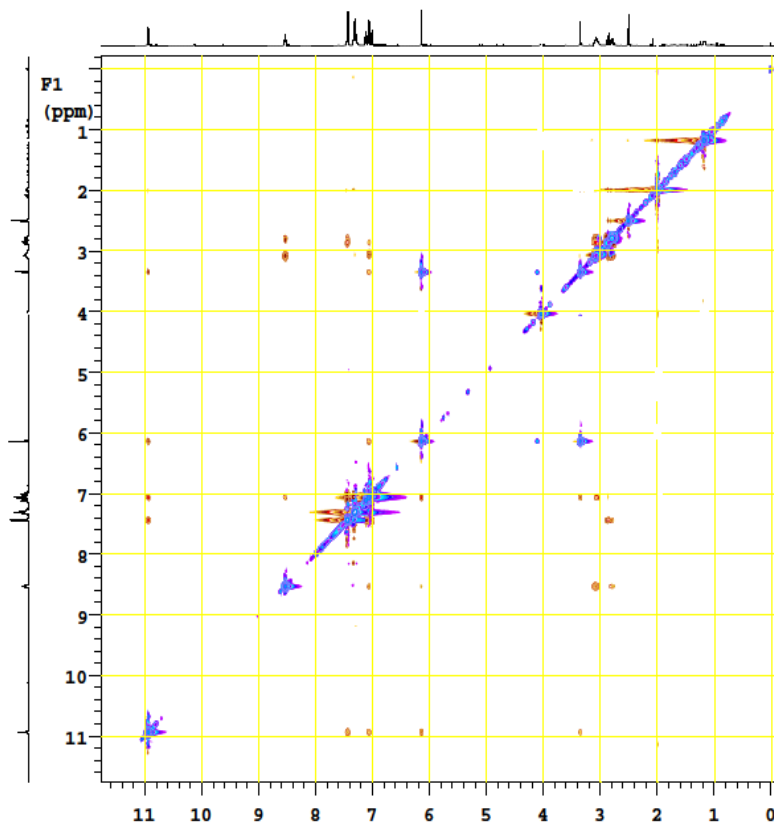


A214/CARS-2/016 Fr-1 in DMSO
TDC-206

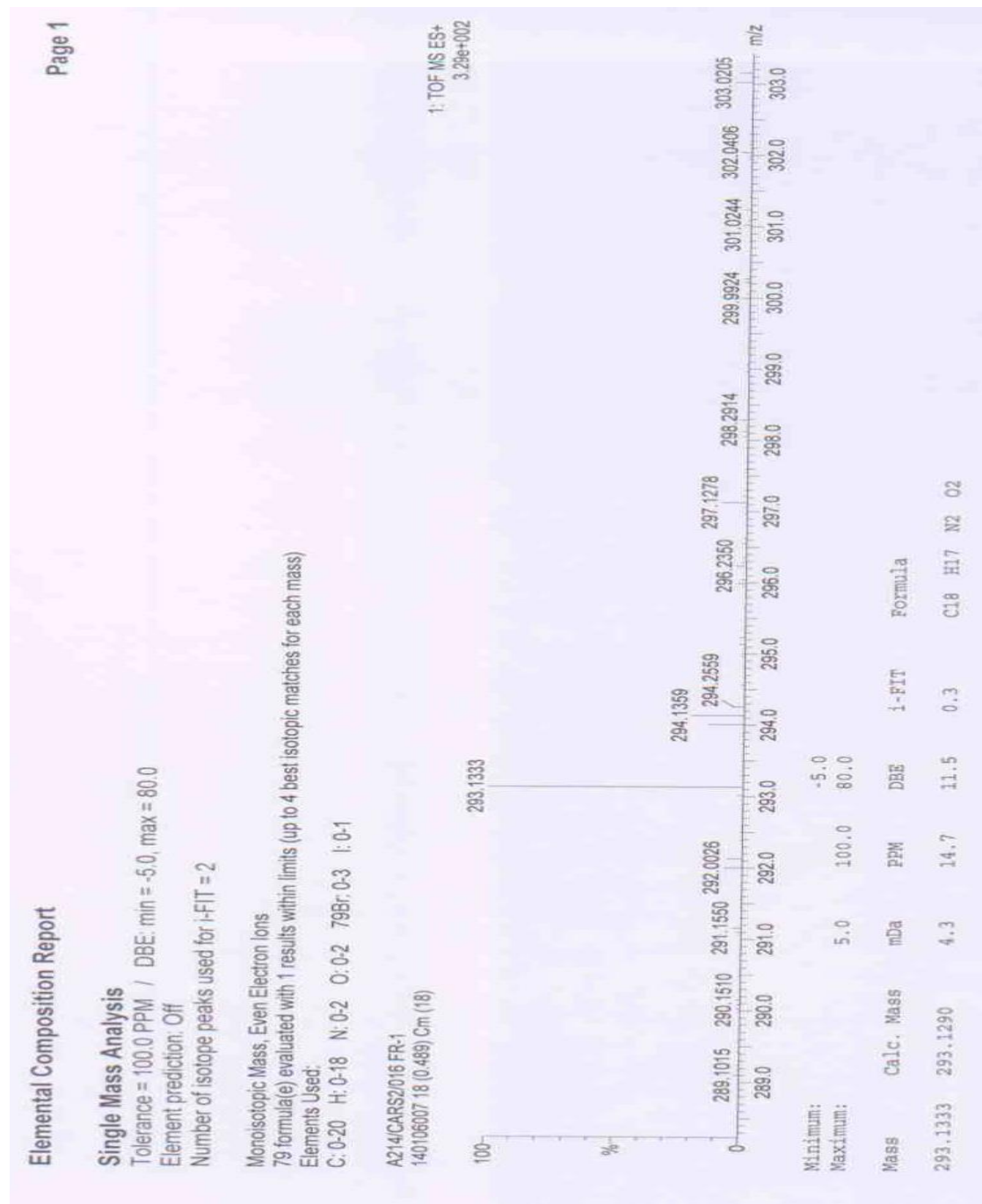
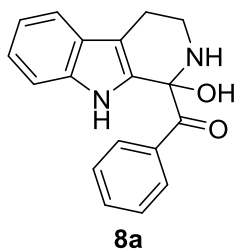
AR.No:IN0114/09
Date: 04th Jan.2014
Analyst:Haribabu

exp2 NOESY

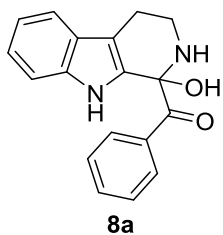
SAMPLE		FLAGS	
date	Jan 4 2014	hs	n
solvent	DMSO	sspul	y
sample		PFGflg	y
ACQUISITION		hsqvlv	4237
sw	7107.3	SPECIAL	
at	0.144	temp	25.0
np	2040	gain	14
fb	not used	spin	not used
ss	32	F2 PROCESSING	
dl	1.000	gf	0.067
nt	16	gfs	not used
2D ACQUISITION		fn	2040
sw1	7107.3	F1 PROCESSING	
ni	400	gfl	0.024
TRANSMITTER		gfs1	not used
tn	H1	procl	lp
sfrq	499.629	fn1	2040
tof	530.1	DISPLAY	
tpwr	50	sp	-55.0
pw	7.200	wp	5934.3
NOESY		sp1	-104.4
mix	0.600	wp1	5976.0
PRESATURATION		rfl	1776.0
satmode	nnnn	rfl	1249.1
satpwr	0	rfl1	1776.0
satdly	0	rfl1	1249.1
satfrq	0	PLOT	
DECOUPLER		wc	139.3
dn	C13	sc	10.0
dm	nnn	wc2	139.3
		sc2	0
		vs	4013
		th	2
	al	ph	



HRMS spectrum of of(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**8a**):



Mass spectrum of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(phenyl)methanone (**8a**):



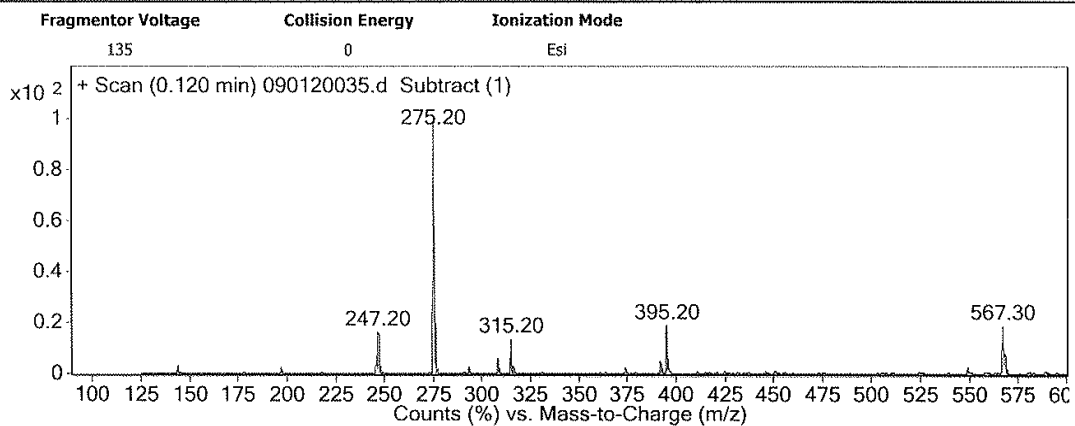
CPS,MIYAPUR

Mass Analysis Report

DAE, R. 2017

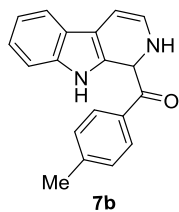
Data Filename	090120035.d	Sample Name	A214/CARS-2/017 C
Sample Type	Sample	Position	Vial 88
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	DA.m	Comment	

User Spectra



--- End Of Report ---

¹H NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(*p*-tolyl)methanone (**7b**):



AKSd, Aurisette Discovery Technologies Ltd, Hyderabad

Instrument

Mercury Plus (Varian 400MHz)

Date & Time

Tue Mar 9 14:21:22 IST 2010

Recorded By

Srikanth.A

AKS-2/051 in DMSO

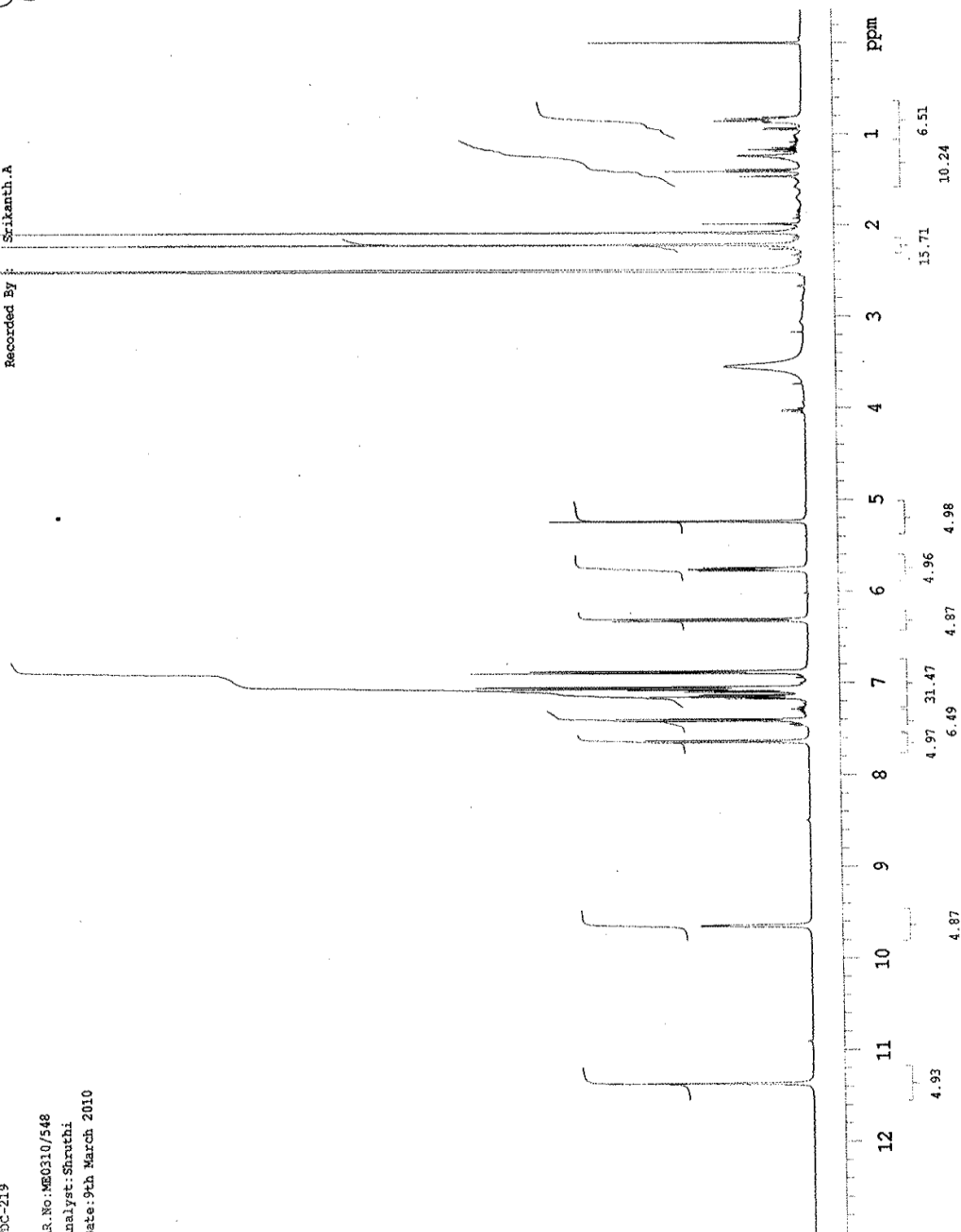
TDC-219

AKS.No:ME0310/548

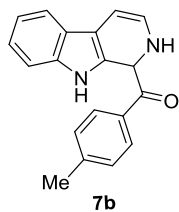
Analyst: Shruthi

Date: 9th March 2010

Handwritten signature and date: 9/3/10



¹³C NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(*p*-tolyl)methanone (**7b**):

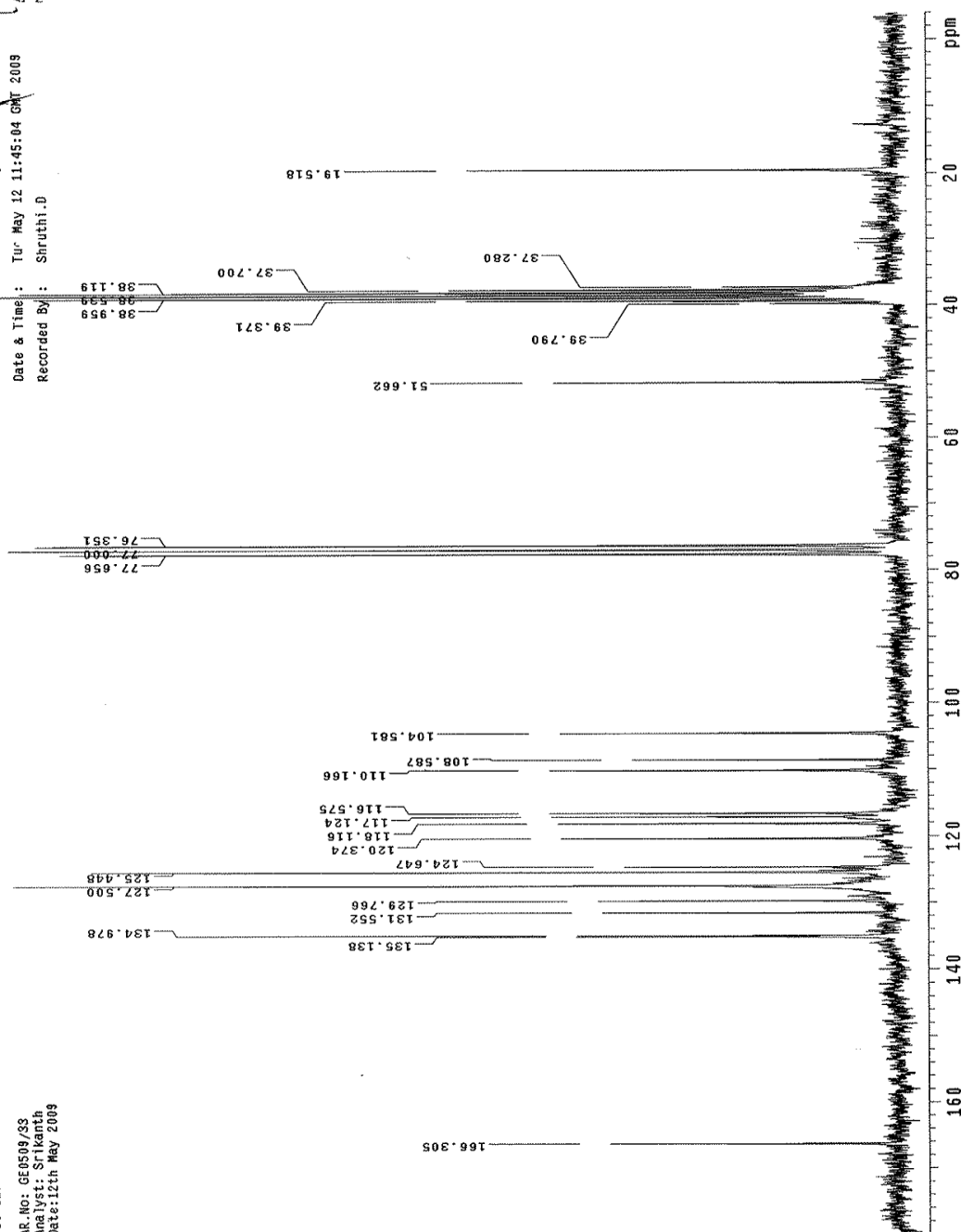


Handwritten signature

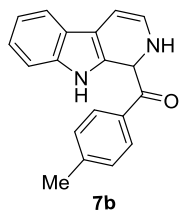
Analytical Research, Discovery Research, DKL
 Instrument : Gemini 2000 (Varian 200MHz)
 Date & Time : Tue May 12 11:45:04 GMT 2009
 Recorded By : Shruthi.D

A214/CARS2/051 Fr2 IN CDCl₃+DMSO
 TDC-219

AR.No: GE0509/53
 Analyst: Srikanth
 Date: 12th May 2009



Mass spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(*p*-tolyl)methanone (**7b**):



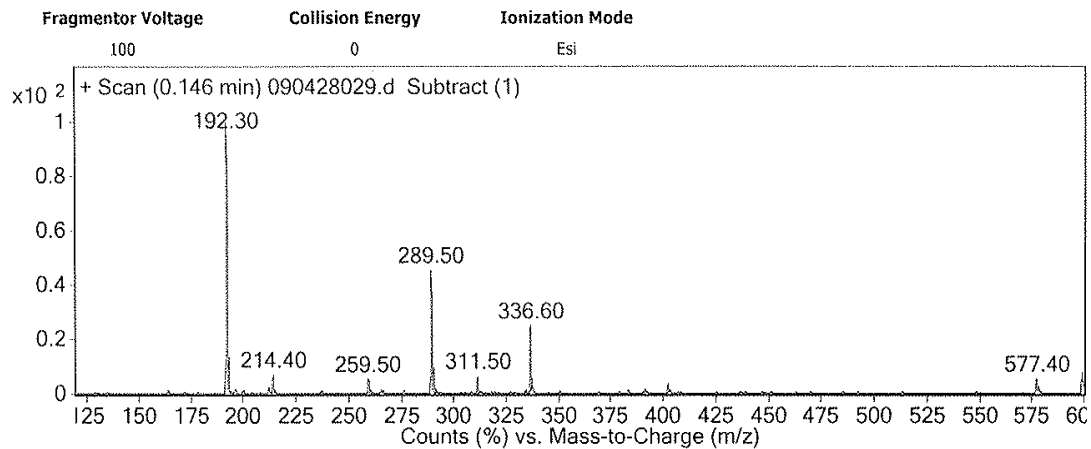
CPS,MIYAPUR

Mass Analysis Report

DocuSign

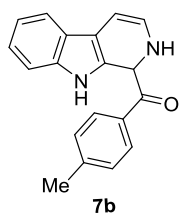
Data Filename	090428029.d	Sample Name	A214/CARS-2/051 FR-2
Sample Type	Sample	Position	Vial 84
Instrument Name	Instrument 1	User Name	
Acq Method		IRM Calibration Status	Success
DA Method	Quant Process.m	Comment	

User Spectra



--- End Of Report ---

HRMS of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(*p*-tolyl)methanone (**7b**):



Page 1

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

58 formula(e) evaluated with 1 results within limits (up to 4 best isotopic matches for each mass)

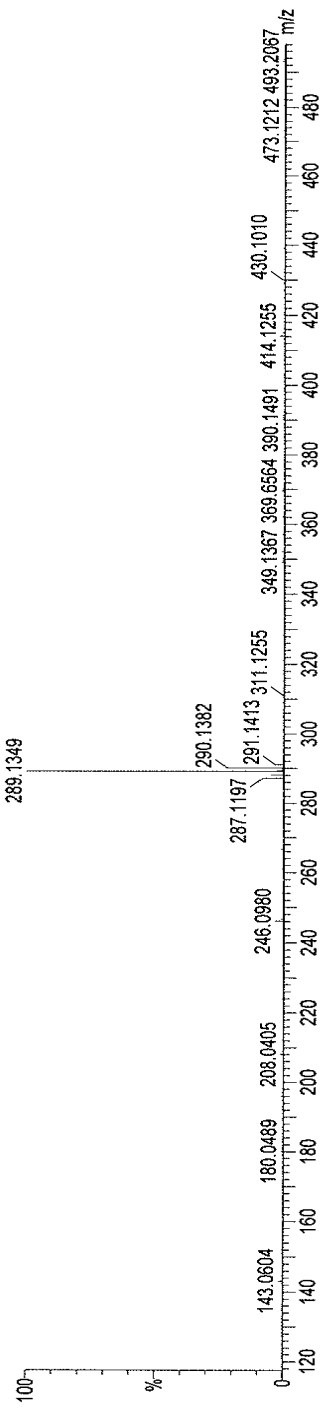
Elements Used:

C: 0-25 H: 0-25 N: 0-4 O: 0-4

9b

UT1212_150.8 (0.278) Cm (8:10)

Galanan
1: TOF MS ES+
8.86e+003



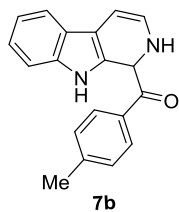
Minimum:
Maximum:

5.0 5.0
-1.5 80.0

Mass Calc. Mass mDa PPM DBE i-FIT Formula

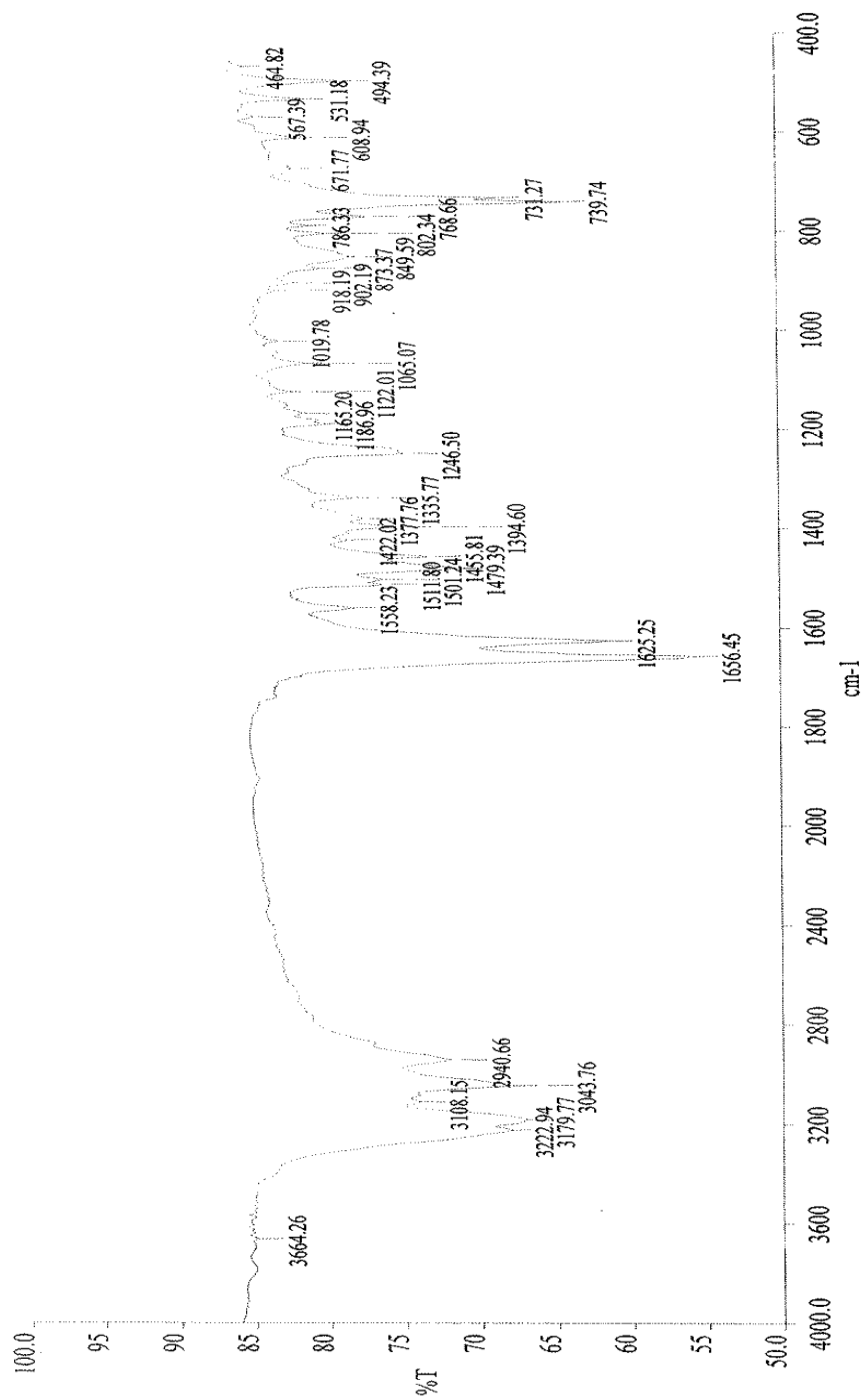
289.1349 289.1341 0.8 2.8 12.5 5.4 C19 H17 N2 O

IR spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(*p*-tolyl)methanone (**7b**):



Date: 3/13/2010 Time: 4:02:54 PM

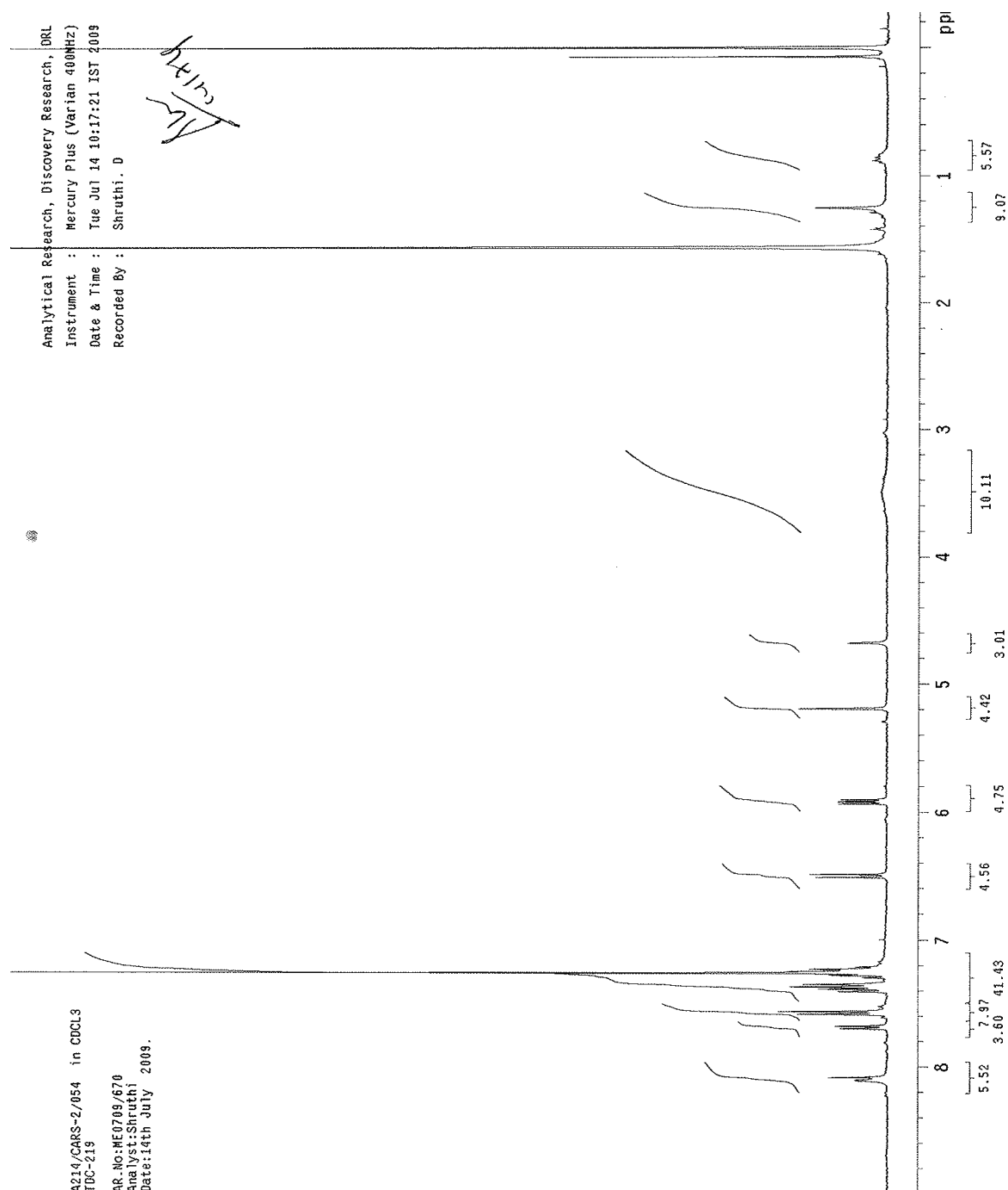
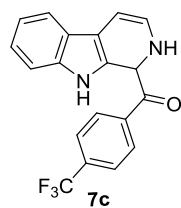
CUSTOM PHARMACEUTICAL SERVICES



— ARS-2-051-fr-2.002 - 3/13/2010

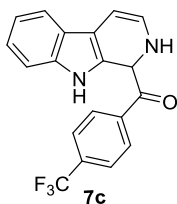
COMPARE REPORT

¹H NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-(trifluoromethyl)phenyl)methanone (**7c**):



A214/CARS-2/054 in CDCl₃
 TDC-219
 AR_No:ME0709/670
 Analyst:Shruthi
 Date:14th July 2009.

^{13}C NMR of (2,9-dihydro-1H-pyrido[3,4-b]indol-1-yl)(4-(trifluoromethyl)phenyl)methanone (**7c**):



AR&D, Aurigene Discovery Technologies Ltd, Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Thu Jan 21 08:57:42 IST 2010

Recorded By : Srikanth.A

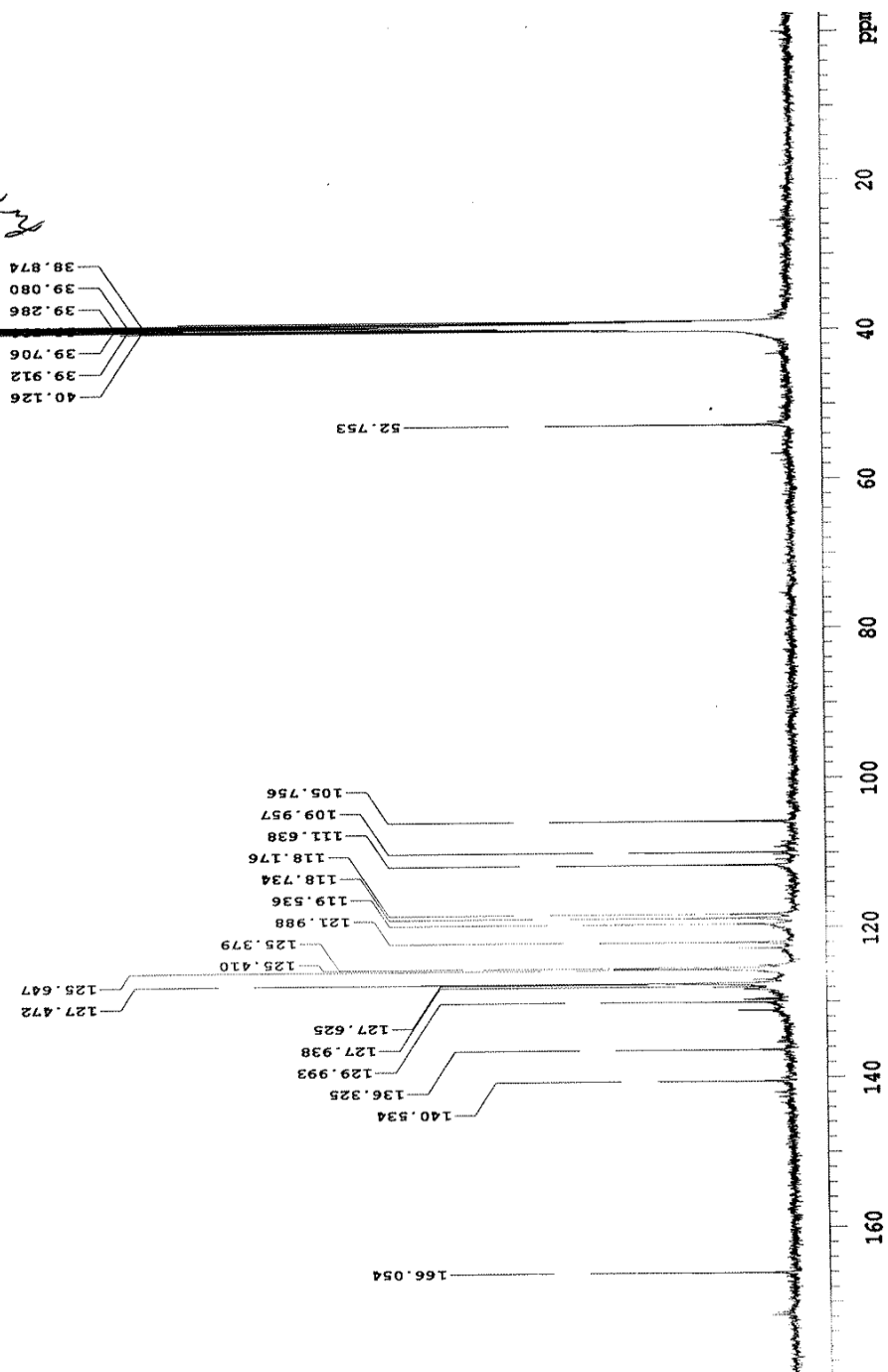
ARS-2/054 in DMSO

TDC-219

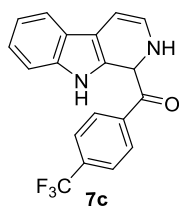
AR.No:ME0110/1068

Analyst:Srikanth.A

Date:20th Jan. 2010



Mass spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-(trifluoromethyl)phenyl)methanone (**7c**):



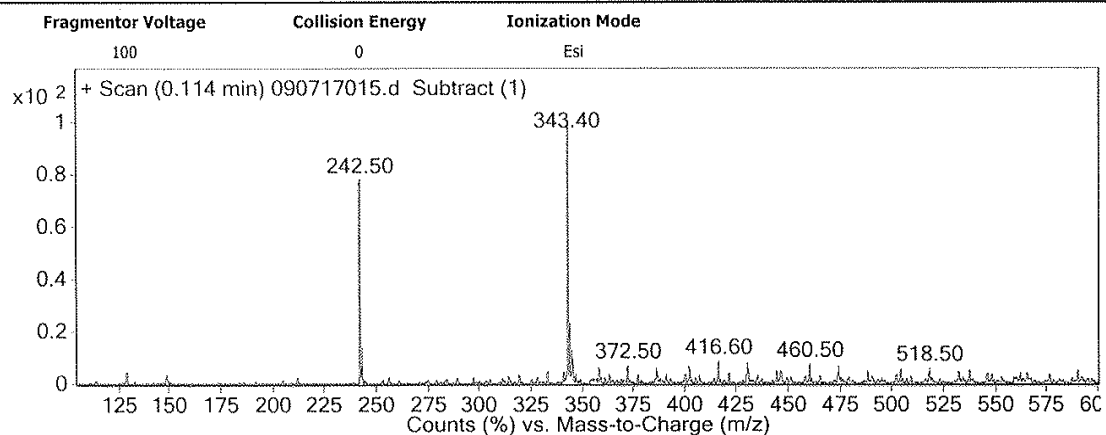
CPS,MIYAPUR

Mass Analysis Report

DATA READY

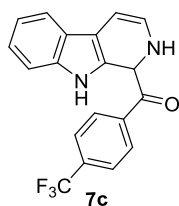
Data Filename	090717015.d	Sample Name	CARS2/054
Sample Type	Sample	Position	Vial 14
Instrument Name	Instrument 1	User Name	
Acq Method		IRM Calibration Status	Success
DA Method	Quant Process.m	Comment	

User Spectra



--- End Of Report ---

HRMS of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-(trifluoromethyl)phenyl)methanone (**7c**):



Page 1

Elemental Composition Report

Single Mass Analysis

Tolerance = 6.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

26 formula(e) evaluated with 1 results within limits (up to 4 best isotopic matches for each mass)

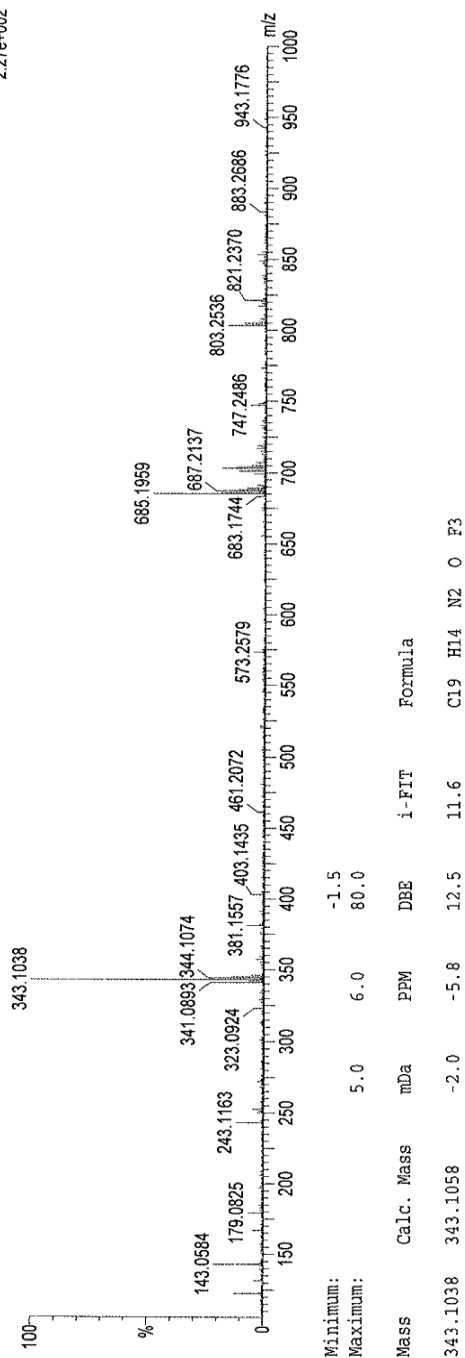
Elements Used:

C: 0-20 H: 0-20 N: 0-2 O: 0-2 F: 0-3

9c

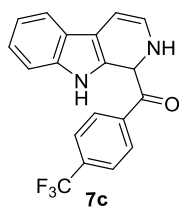
UT1212_179 9 (0.337) Cm (9:10)

Gajanan
1: TOF MS ES+
2.27e+002



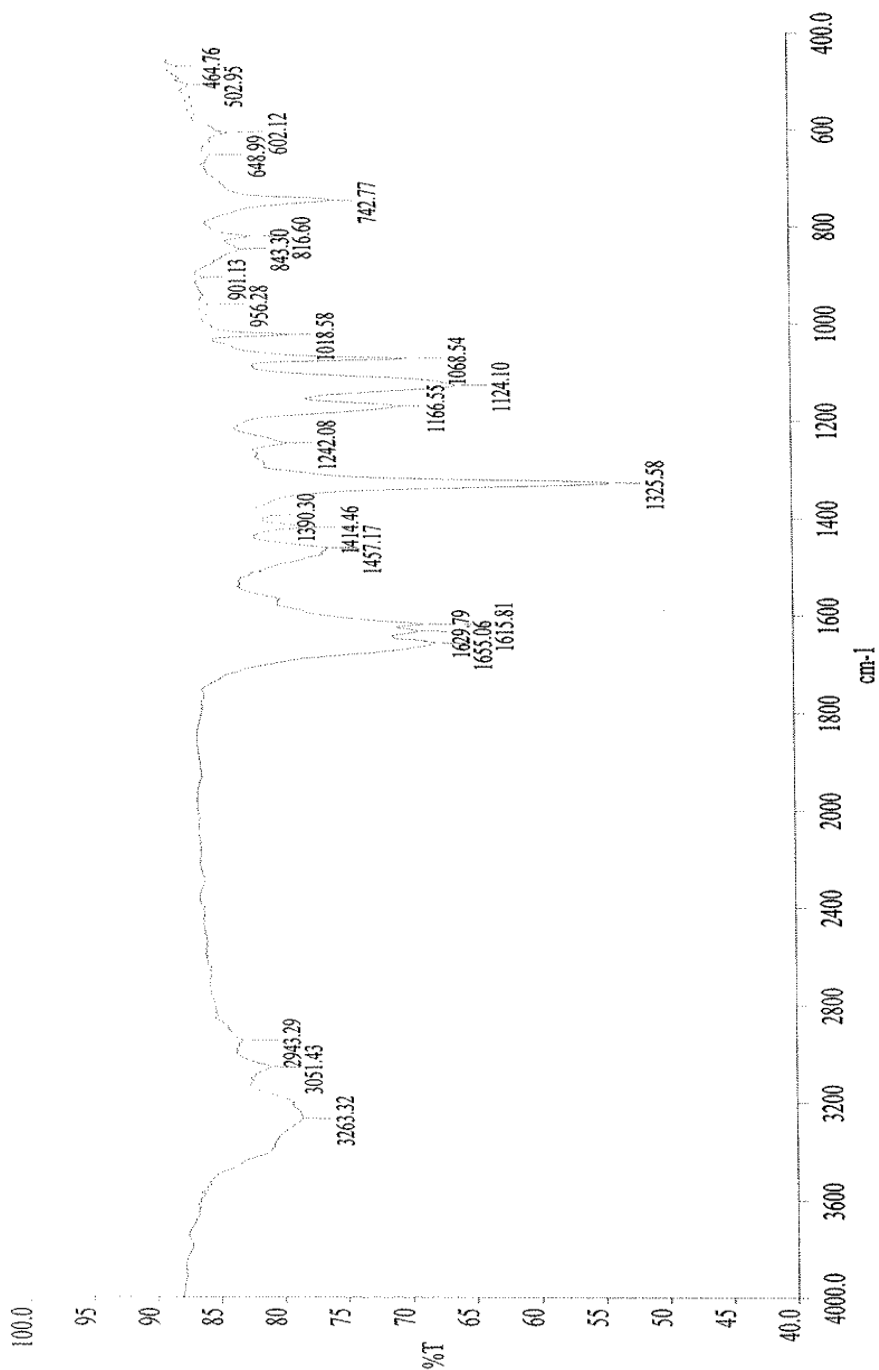
Minimum: -1.5
Maximum: 80.0

IR spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-(trifluoromethyl)phenyl)methanone (**7c**):



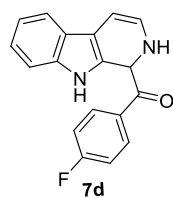
Date: 3/13/2010 Time: 4:35:03 PM

CUSTOM PHARMACEUTICAL SERVICES



COMPARE REPORT

^1H NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-fluorophenyl)methanone (**7d**):



ARSd, Aurigene Discovery Technologies Ltd, Hyderabad

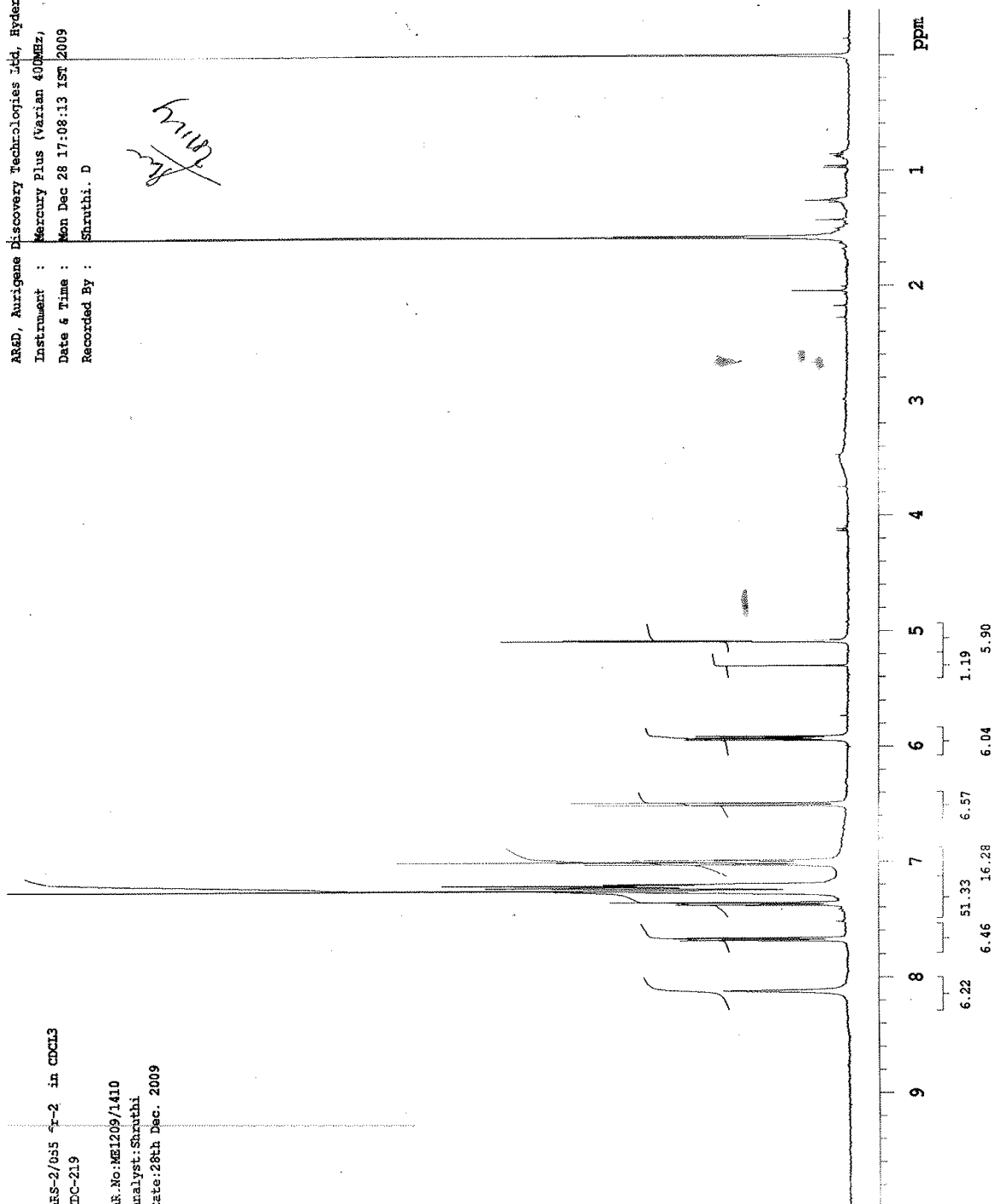
Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Mon Dec 28 17:08:13 IST 2009

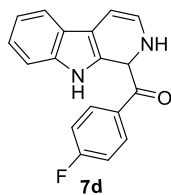
Recorded By : Shruthi. D

ARS-2/035 4T-2 in CDCl₃
TDC-219

AR.No:ME1209/1410
Analyst:Shruthi
Date:28th Dec. 2009



¹³C NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-fluorophenyl)methanone (**7d**):



...d, Anrigene Discovery Technologies Ltd, Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : 26 Jan 26 10:15:14 IST 2010

Recorded By : Srikanth.A

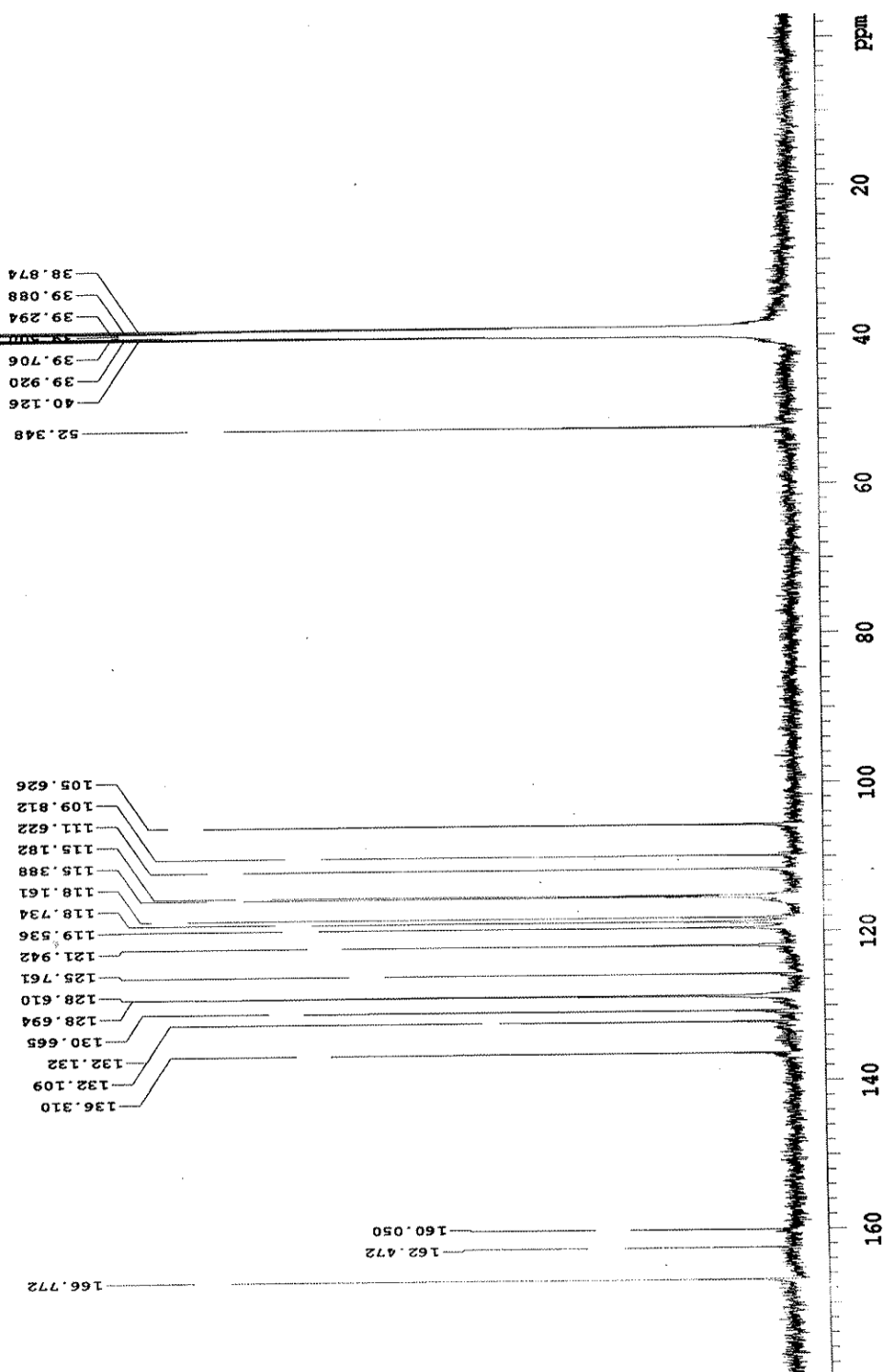
ABS-2/055 FR-2 in DMSO d6

TDC-219

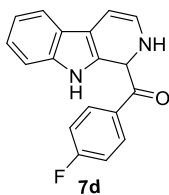
AR.No:ME0110/1323

Analyst: Srikanth.A

Date: 25th Jan. 2010



Mass spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-fluorophenyl)methanone
(7d):

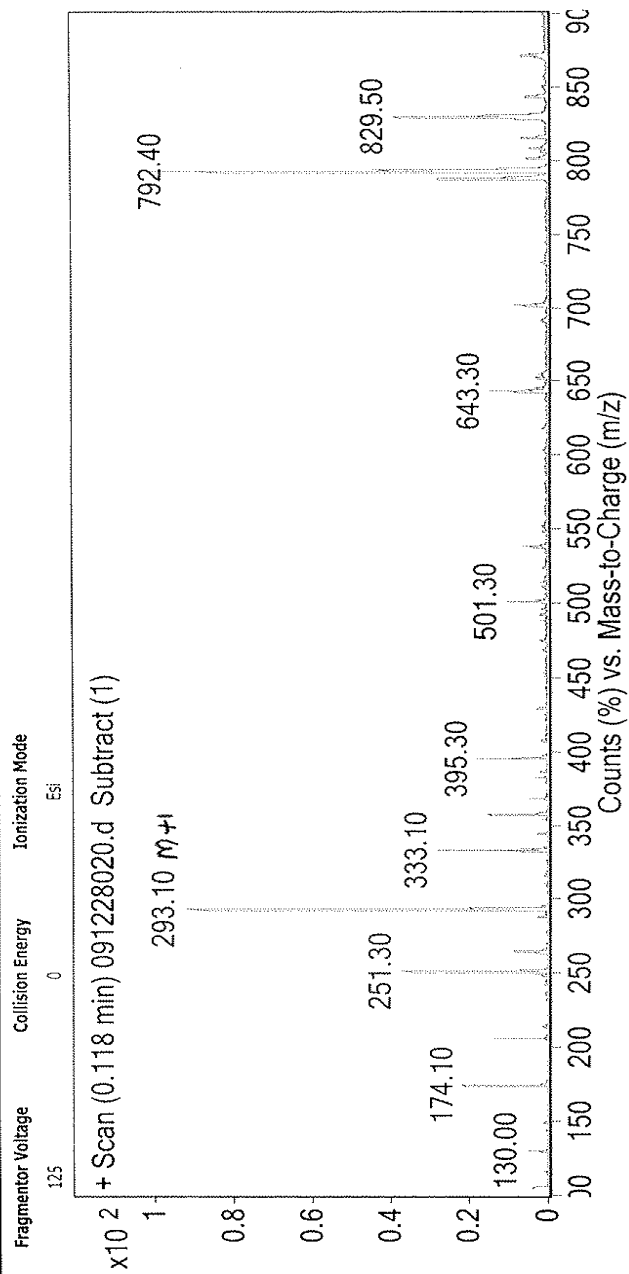


CPS,MIYAPUR

Mass Analysis Report

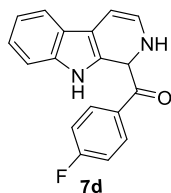
Data Filename	091228020.d	Sample Name	ARS-2/055 FR-2
Sample Type	Sample	Position	Vial 5
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	default.m	Comment	

User Spectra



--- End Of Report ---

HRMS of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-fluorophenyl)methanone (**7d**):



Page 1

Elemental Composition Report

Single Mass Analysis

Tolerance = 6.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

62 formula(e) evaluated with 1 results within limits (up to 4 best isotopic matches for each mass)

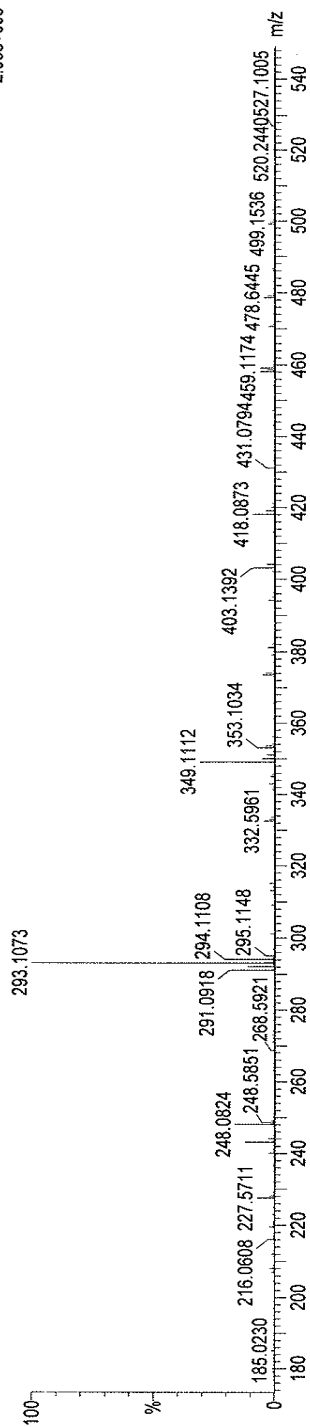
Elements Used:

C: 0-20 H: 0-20 N: 0-6 O: 0-2 F: 0-1

9d

UT1212_181 7 (0.252) Cm (7.8)

Gajanan
1: TOF MS ES+
2.03e+003



Minimum:

Maximum:

-1.5

80.0

5.0

6.0

PPM

DBE

Calc. Mass

mDa

293.1073

293.1090

Mass

Formula

i-FIT

3.9

-5.8

12.5

C18

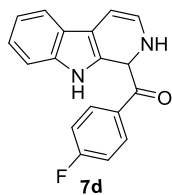
H14

N2

O

F

IR spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-fluorophenyl)methanone (**7d**):



CUSTOM PHARMACEUTICAL SERVICES

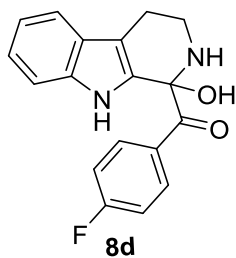
Date: 3/13/2010 Time: 4:27:12 PM



ARS-2-055 002 - 3/13/2010

COMPARE REPORT

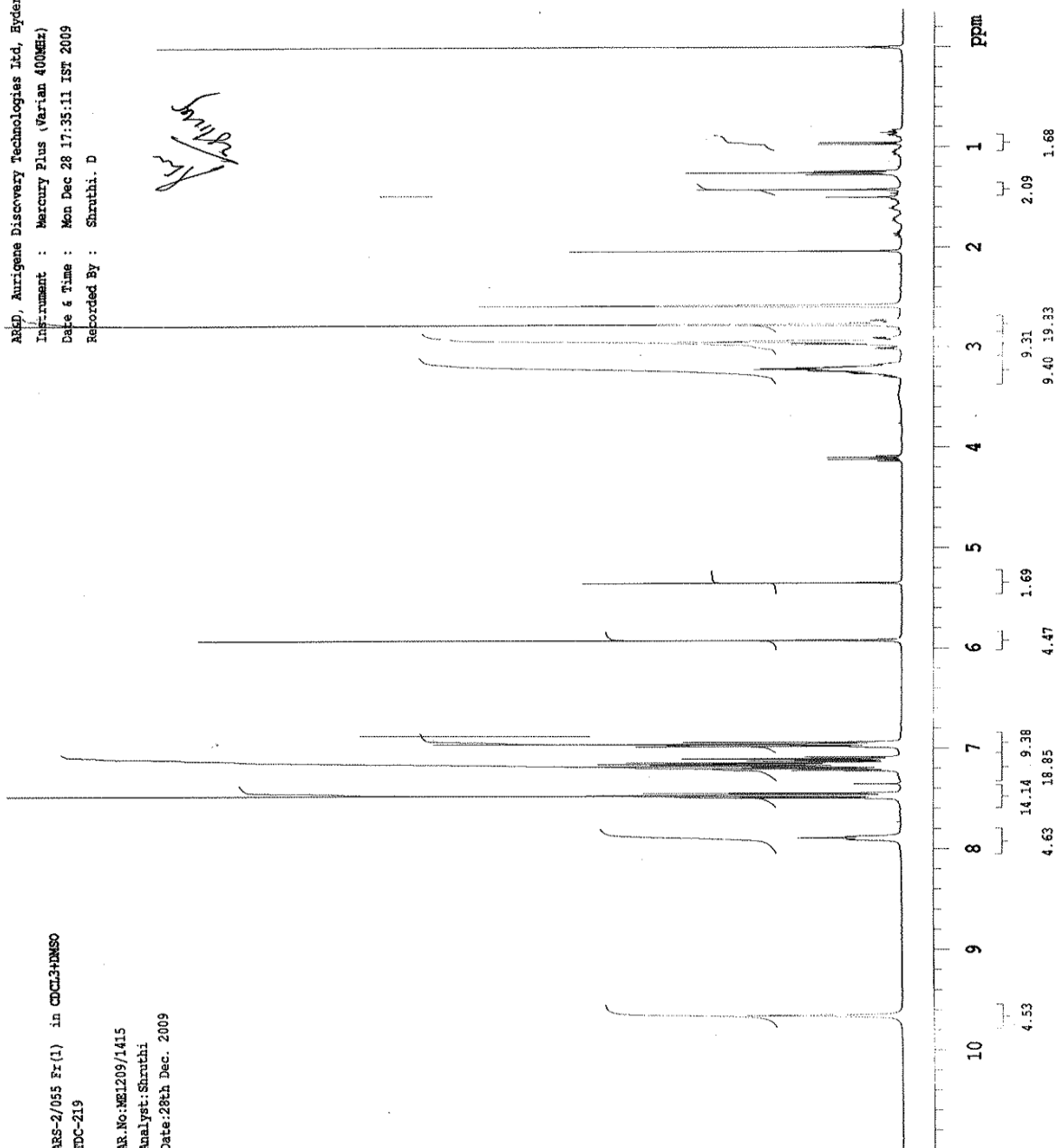
^1H NMR of (4-fluorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1H-pyrido[3,4-b]indol-1-yl)methanone (**8d**):



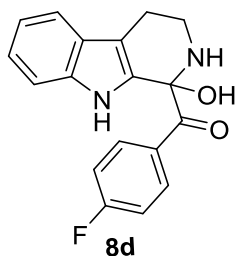
ARUP, Aurigene Discovery Technologies Ltd, Hyderabad
 Instrument : Mercury Plus (Varian 400MHz)
 Date & Time : Mon Dec 28 17:35:11 IST 2009
 Recorded By : Shruthi. D

ARS-2/055 Pr(1) in CDCl₃+DMSO
 TDC-219

AR.No:ME1209/1415
 Analyst:Shruthi
 Date:28th Dec. 2009



^{13}C NMR of (4-fluorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8d**):



AKED, Aurigene Discovery Technologies Ltd, Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Thu Jan 21 08:32:23 IST 2010

Recorded By : Sridhar

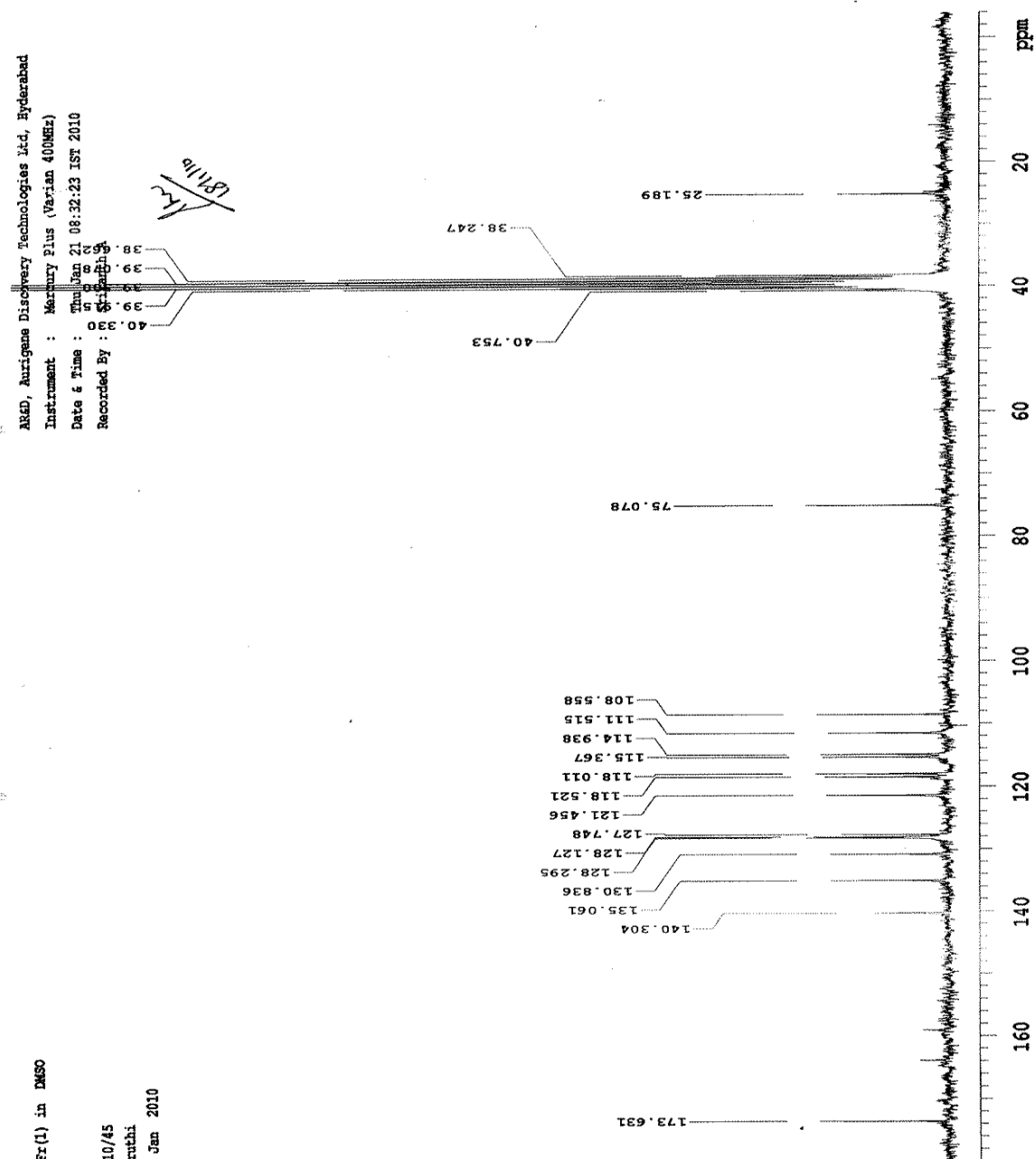
ARS-2/055 Ex (1) in DMSO

TDC-219

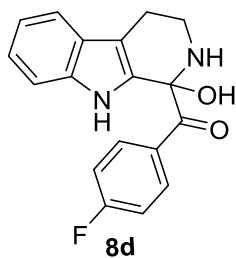
AR.NO:GE0110/45

Analyst:Shruthi

Date:18th Jan 2010



Mass spectrum of (4-fluorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8d**):

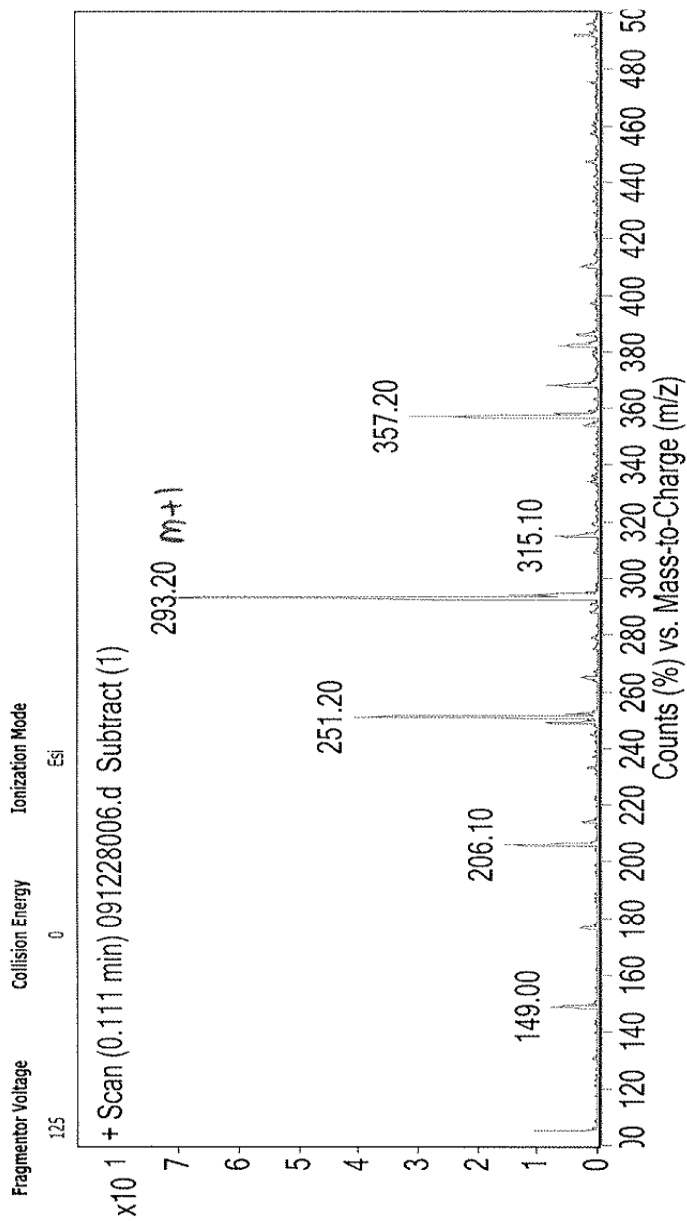


CPS.MIYAPUR

Mass Analysis Report

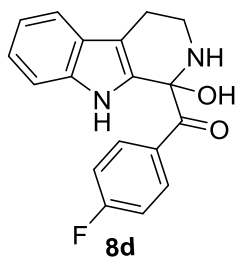
Data Filename	091228006.d	Sample Name	ARS-2/055 FR-1
Sample Type	Sample	Position	Vial 6
Instrument Name	Instrument 1	User Name	
Acq Method	ESL.m	IRM Calibration Status	Success
DA Method	default.m	Comment	

User Spectra



--- End Of Report ---

HRMS of (4-fluorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8d**):



Page 1

Elemental Composition Report

Single Mass Analysis

Tolerance = 7.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass: Even Electron Ions

40 formula(e) evaluated with 2 results within limits (up to 4 best isotopic matches for each mass)

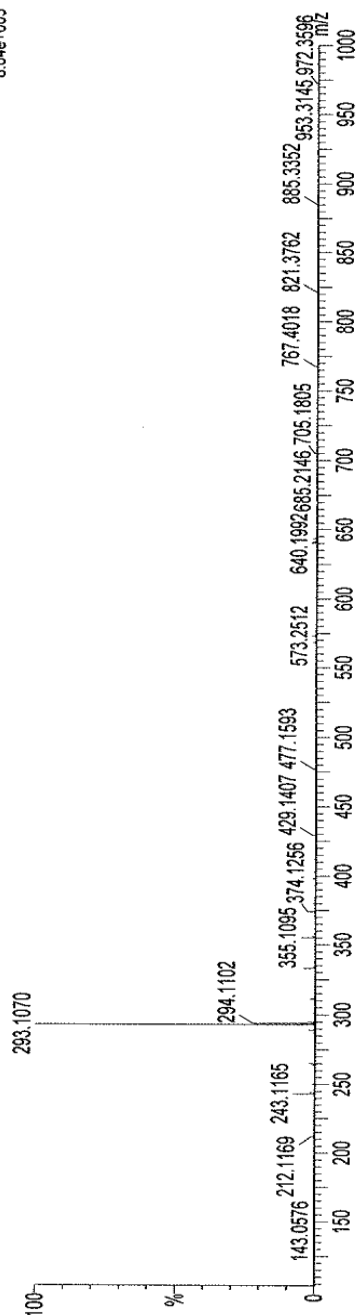
Elements Used:

C: 0-25 H: 0-25 N: 0-2 O: 0-2 F: 0-1

8d

UT1212_175.5 (0.198) Cm (5)

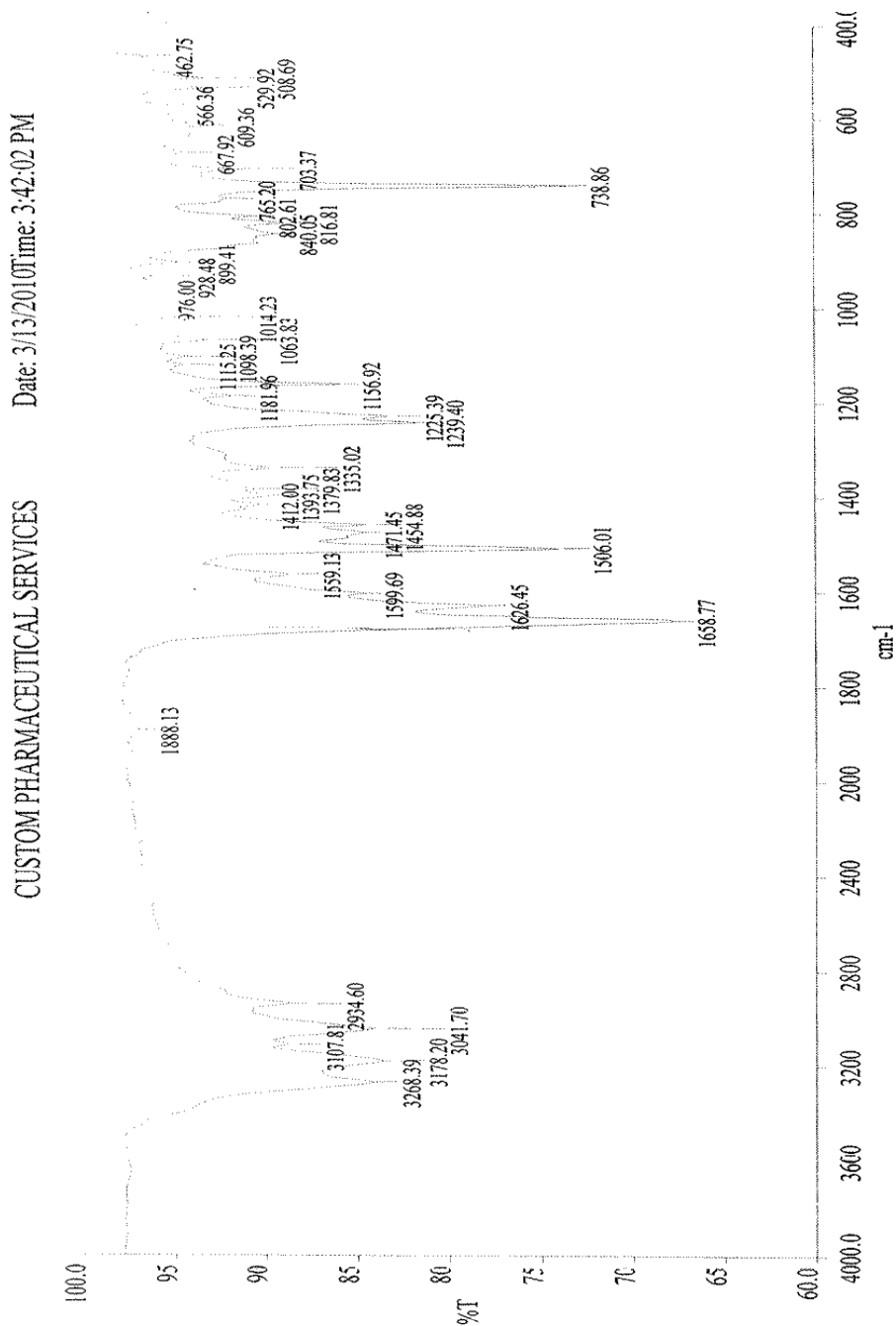
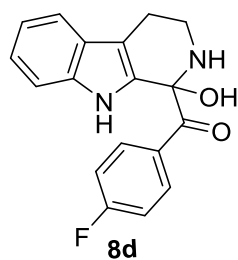
Gajanan
1: TOF MS ES+
8.04e+003



Minimum: -1.5
Maximum: 80.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
293.1070	293.1079	-0.9	-3.1	16.5	6.4	C21 H13 N2
	293.1090	-2.0	-6.8	12.5	7.0	C18 H14 N2 O F

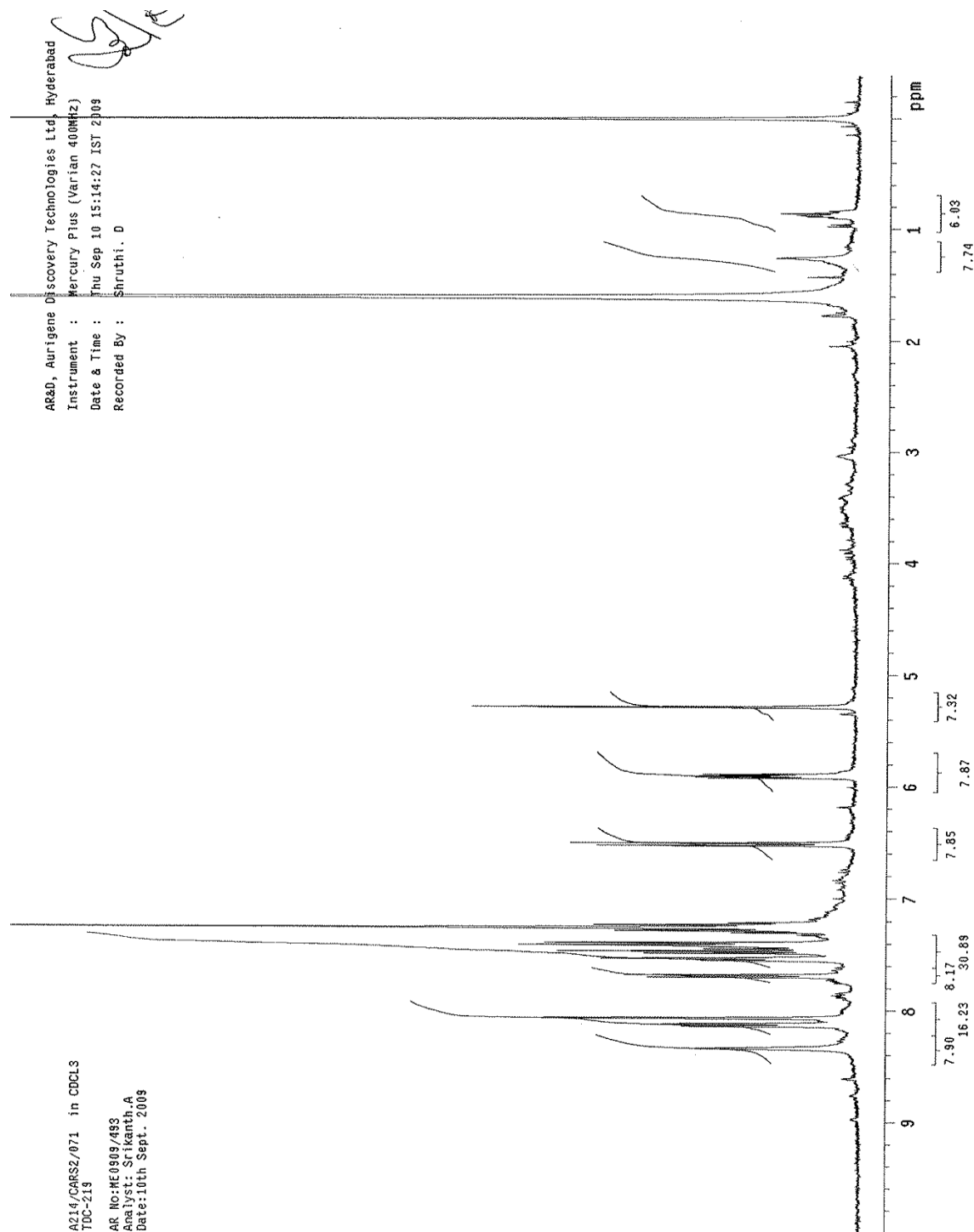
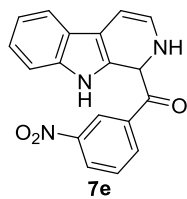
IR spectrum (4-fluorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1H-pyrido[3,4-b]indol-1-yl)methanone (**8d**):



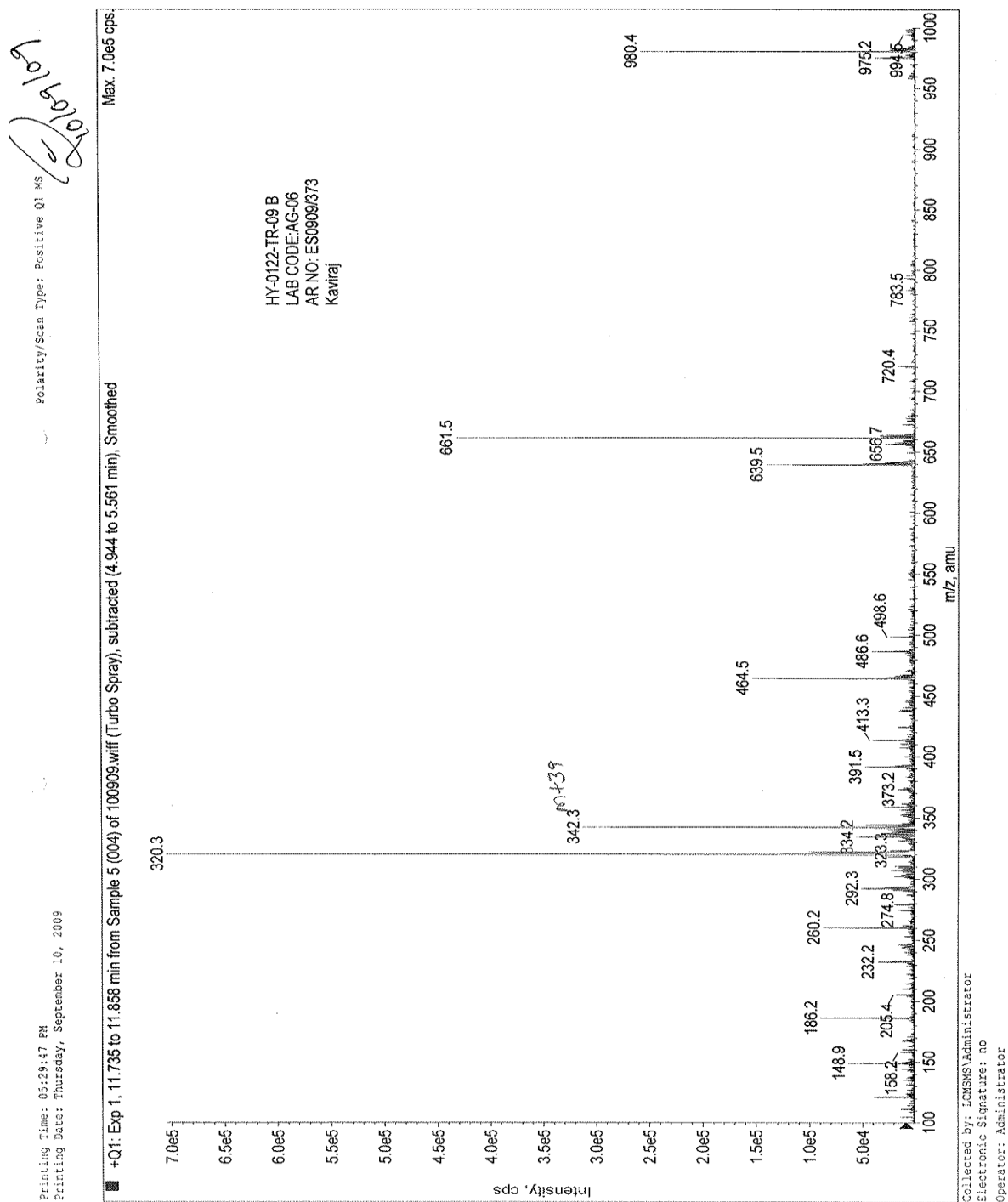
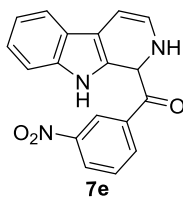
ARS-2-055-fr-2.002 - 3/13/2010

COMPARE REPORT

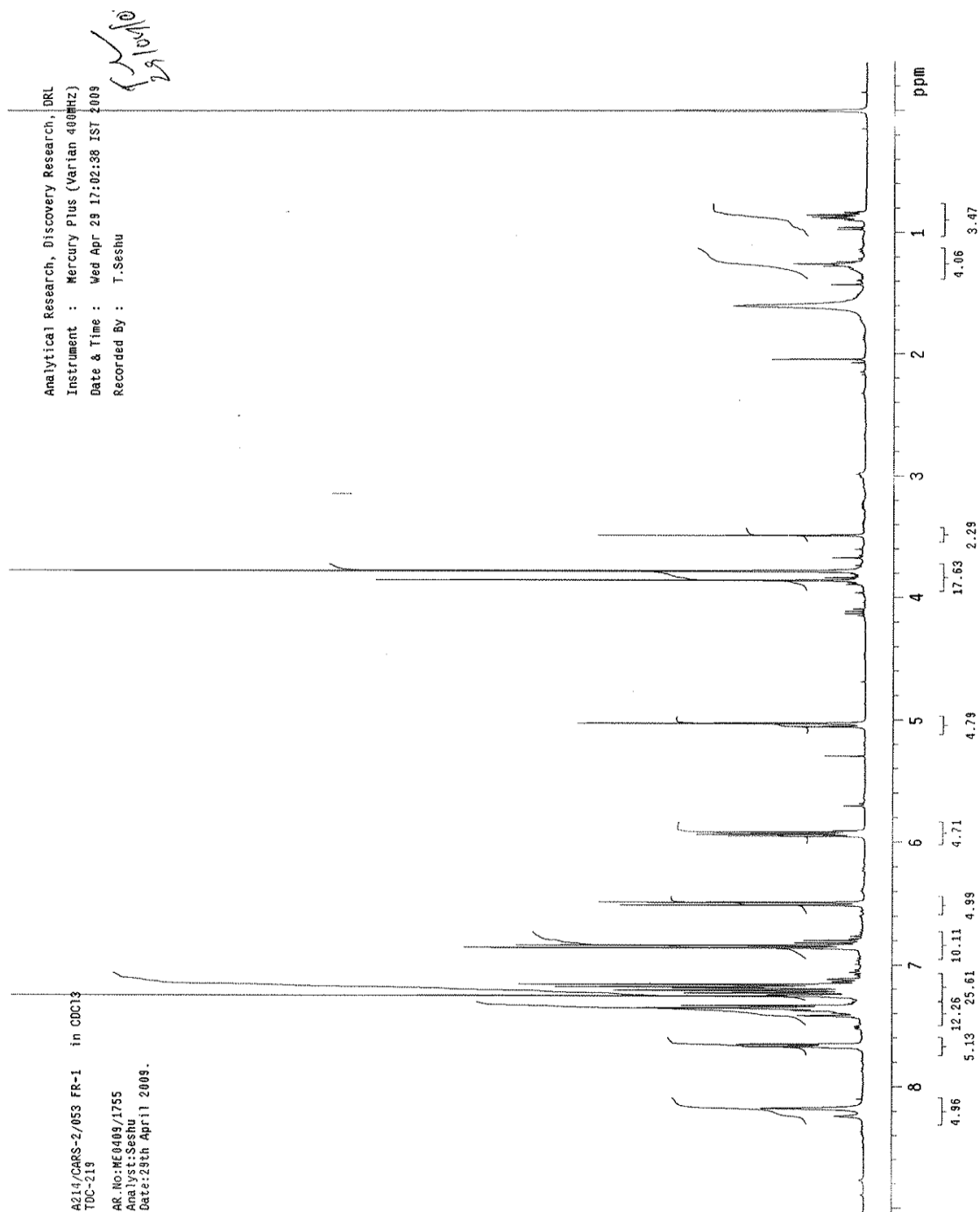
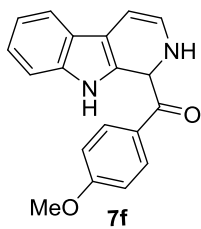
^1H NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(3-nitrophenyl)methanone (**7e**):



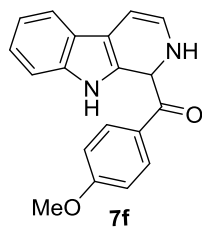
Mass spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(3-nitrophenyl)methanone
(7e):



^1H NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-methoxyphenyl)methanone (**7f**):



¹³C NMR of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-methoxyphenyl)methanone (**7f**):

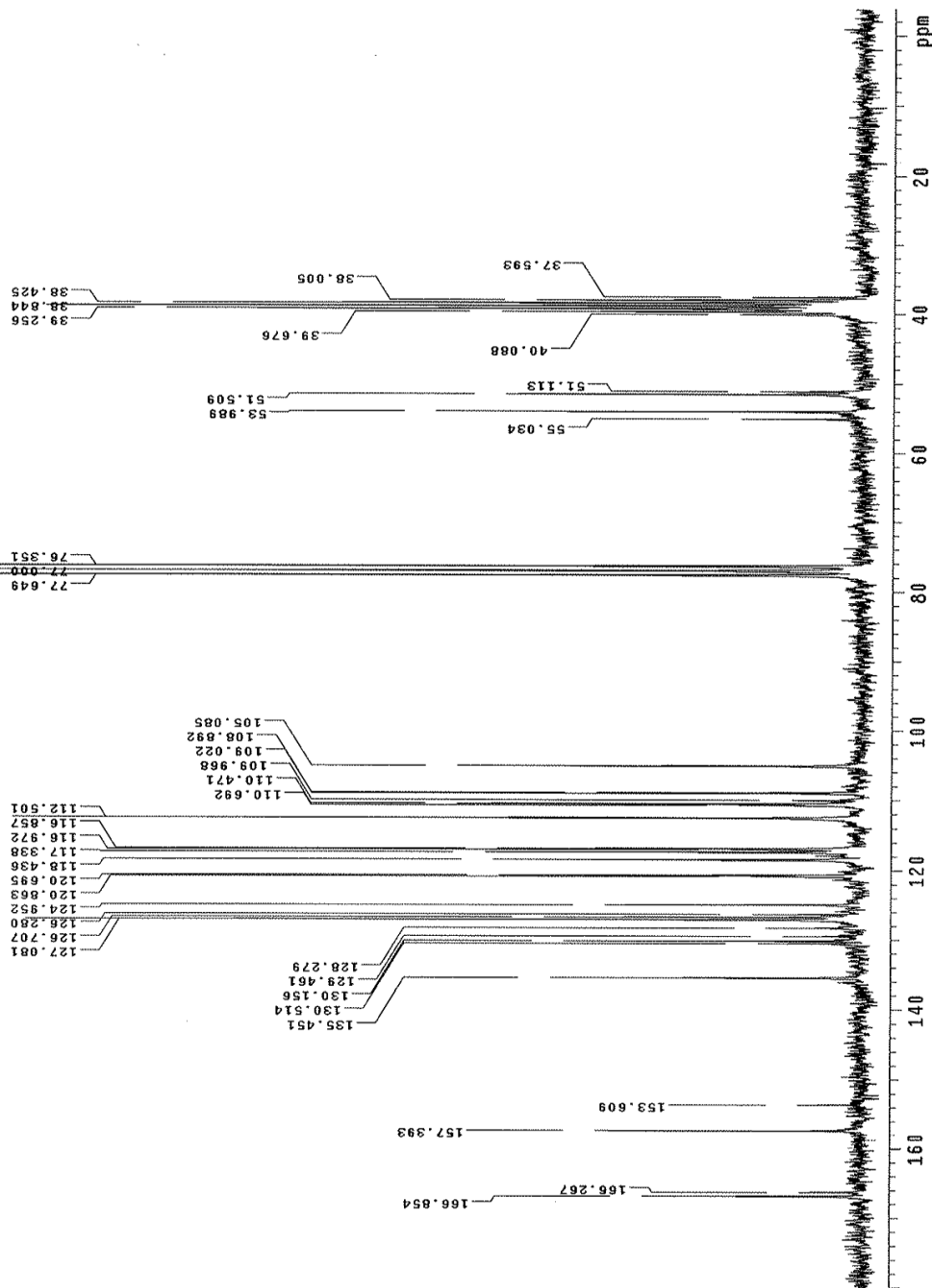


Handwritten signature and date: 12/5/09

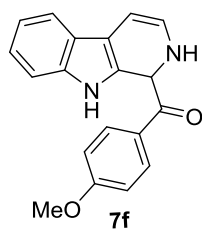
Analytical Research, Discovery Research, DRL
Instrument : Gemini 2000 (Varian 200MHz)
Date & Time : Tue May 12 15:27:37 GMT 2009
Recorded By : Shruthi.D

A214/CARS2/653 Fr1 IN CDCl₃+DMSO
TDC-213

AR-No: GE0509/34
Analyst: Seshu
Date: 12th May 2009



Mass spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-methoxyphenyl)methanone (**7f**):



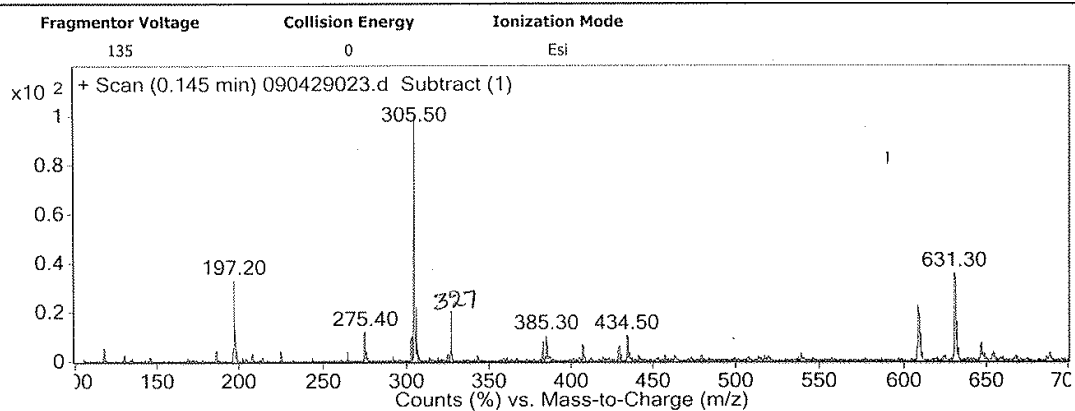
CPS,MIYAPUR

Mass Analysis Report

DAUERHAFT

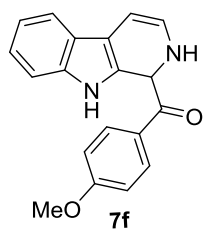
Data Filename	090429023.d	Sample Name	A214/CARS-2/053 FR-1
Sample Type	Sample	Position	Vial 42
Instrument Name	Instrument 1	User Name	
Acq Method		IRM Calibration Status	Success
DA Method	Quant Process.m	Comment	

User Spectra



--- End Of Report ---

IR spectrum of (2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(4-methoxyphenyl)methanone (**7f**):



Date: 3/13/2010 Time: 4:08:42 PM

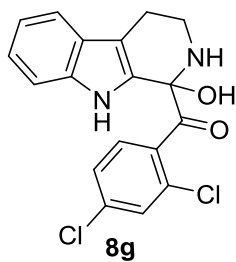
CUSTOM PHARMACEUTICAL SERVICES



ARS-2-053.002 - 3/13/2010

COMPARE REPORT

^1H NMR of (2,4-dichlorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8g**):



ARRIS, Aurisene Discovery Technologies Ltd, Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Mon Jan 4 15:02:31 IST 2010

Recorded By : Srikanth.A

ARS-2/096 Fr2 in DMSO

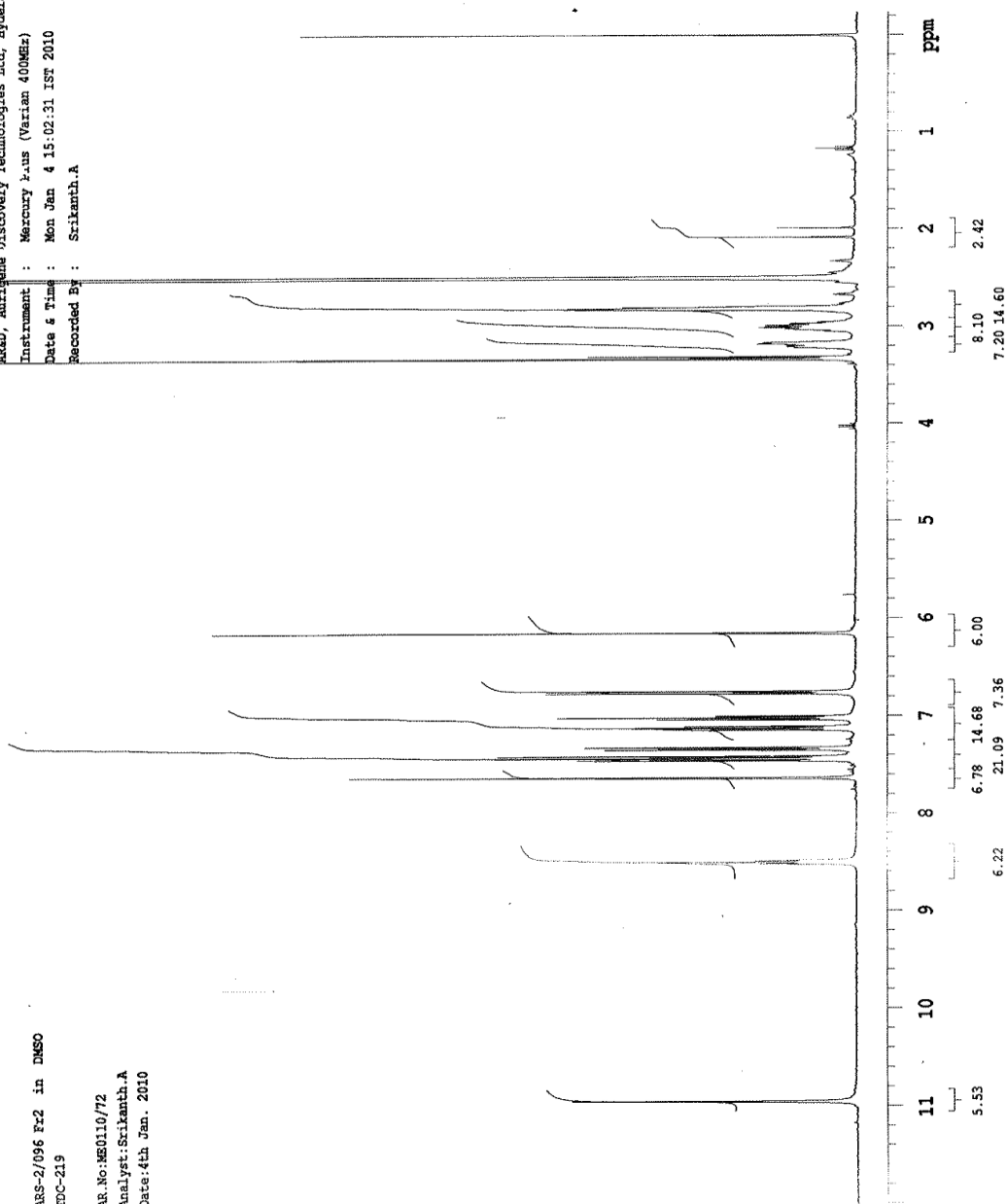
TDC-219

AR.No:ME0110/72

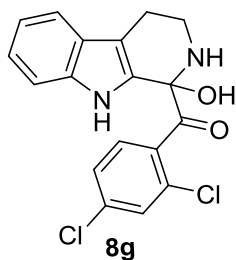
Analyst:Srikanth.A

Date:4th Jan. 2010

(Handwritten signature)



^{13}C NMR of (2,4-dichlorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8g**):



AR&D, Aurigene Discovery Technologies Ltd. Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Fri Jan 22 08:51:46 IST 2010

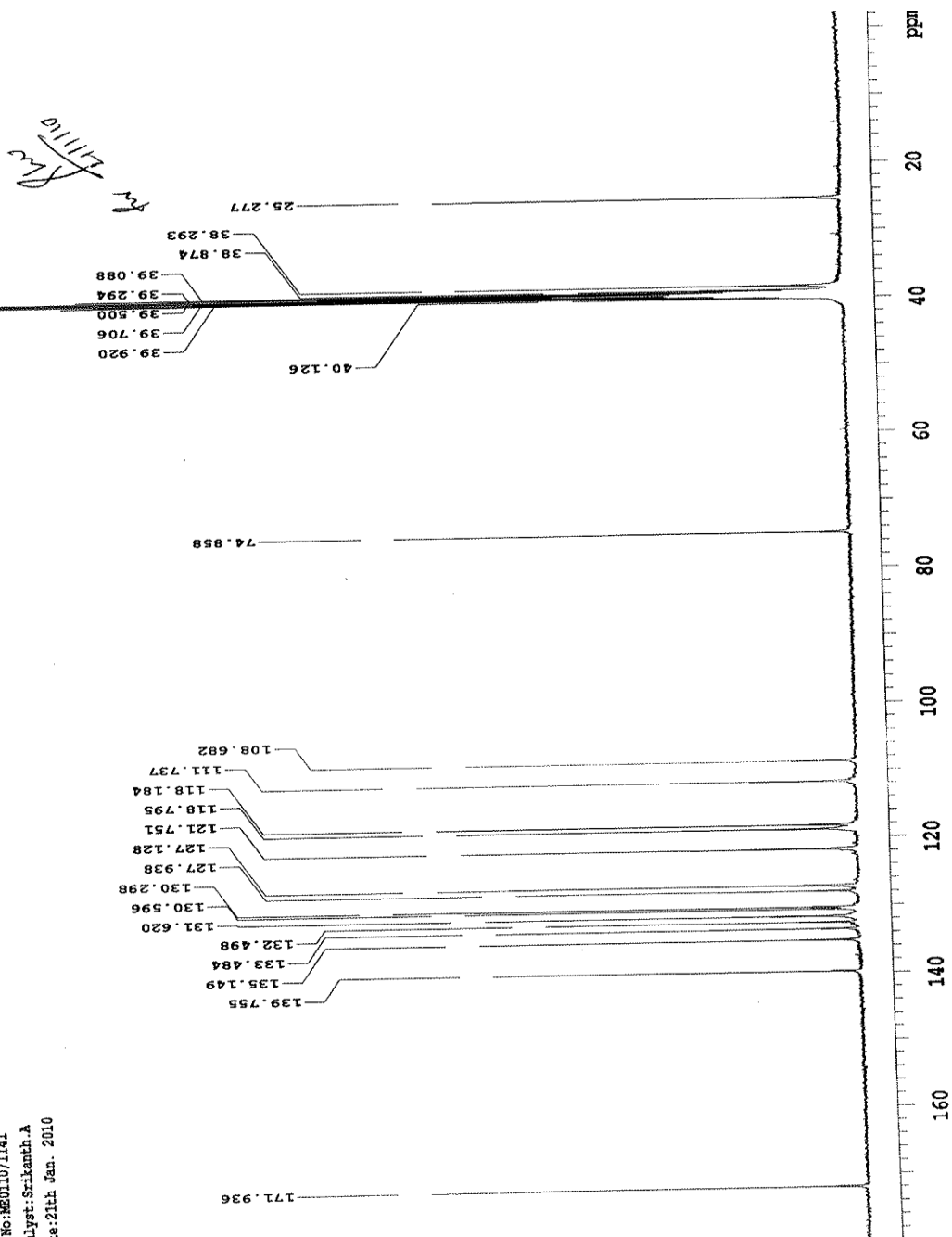
Recorded By : Srikanth.A

MS-2/096 Fr-2 in DMSO
TDC-219

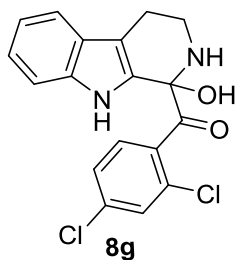
AR.No:ME0110/1141

Analyst: Srikanth.A

Date: 21th Jan. 2010



Mass spectrum (2,4-dichlorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1H-pyrido[3,4-b]indol-1-yl)methanone (**8g**):



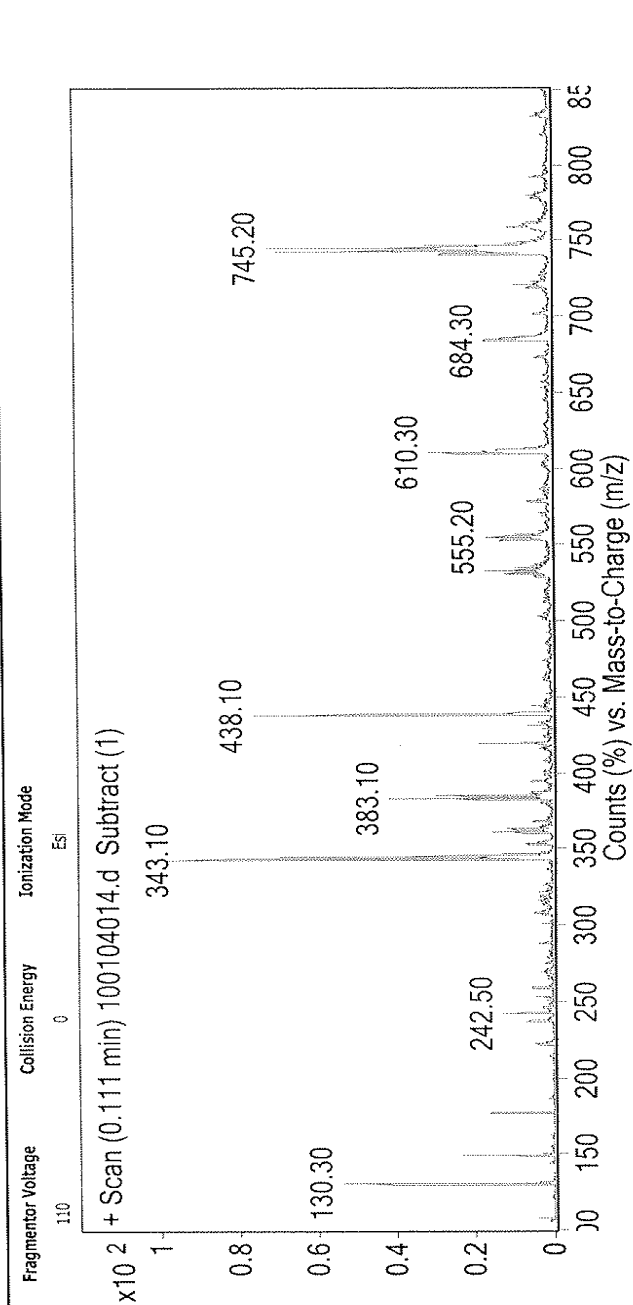
Y. D. 2014.01.14

Mass Analysis Report

CPS,MIYAPUR

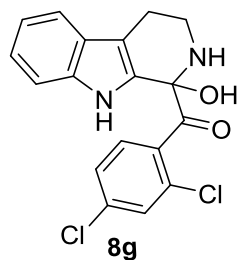
Data Filename	100104014.d	Sample Name	ARS-2/096 FR-2
Sample Type	Sample	Position	Vial 14
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	default.m	Comment	

User Spectra



--- End Of Report ---

HRMS of (2,4-dichlorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8g**):



Page 1

Elemental Composition Report

Single Mass Analysis

Tolerance = 5.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

94 formula(e) evaluated with 2 results within limits (up to 4 best isotopic matches for each mass)

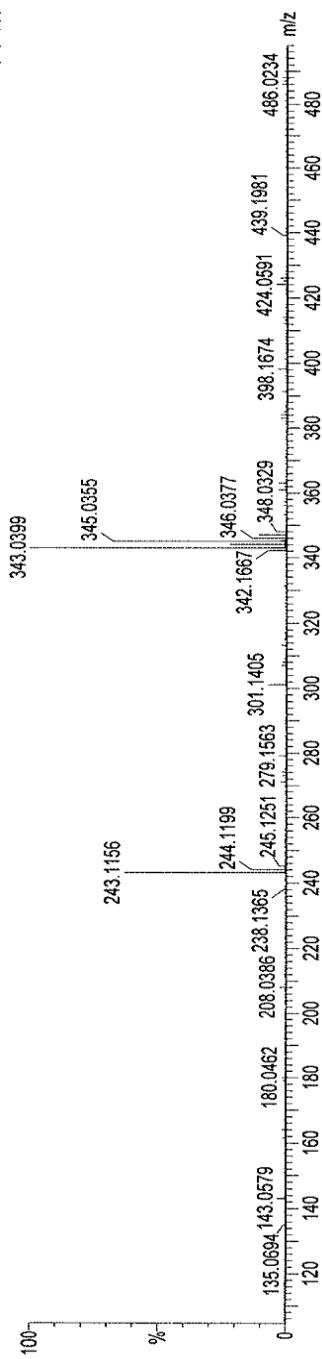
Elements Used:

C: 0-25 H: 0-25 N: 0-4 O: 0-2 Cl: 0-2

9g

UT1212_166 5 (0.197) Cm (5)

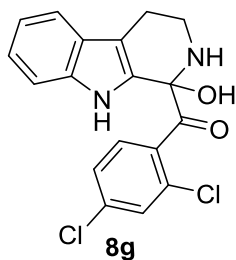
Gajanan
1: TOF MS ES+
1.02e+003



Minimum: -1.5
Maximum: 80.0

Mass	Calc. Mass	mDa	PPM	DBE	i-FIT	Formula
343.0399	343.0405	-0.6	-1.7	12.5	0.5	C18 H13 N2 O Cl2
	343.0387	1.2	3.5	17.5	72.7	C19 H8 N4 O Cl

IR spectrum of (2,4-dichlorophenyl)(1-hydroxy-2,3,4,9-tetrahydro-1H-pyrido[3,4-b]indol-1-yl)methanone (**8g**):



Date: 3/13/2010 Time: 4:14:57 PM

CUSTOM PHARMACEUTICAL SERVICES



ARS-2-096-fr-2.002 - 3/13/2010

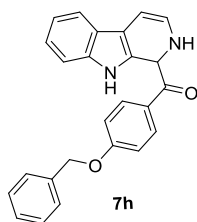
COMPARE REPORT



7h



¹³C NMR of (4-(benzyloxy)phenyl)(2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (7h):

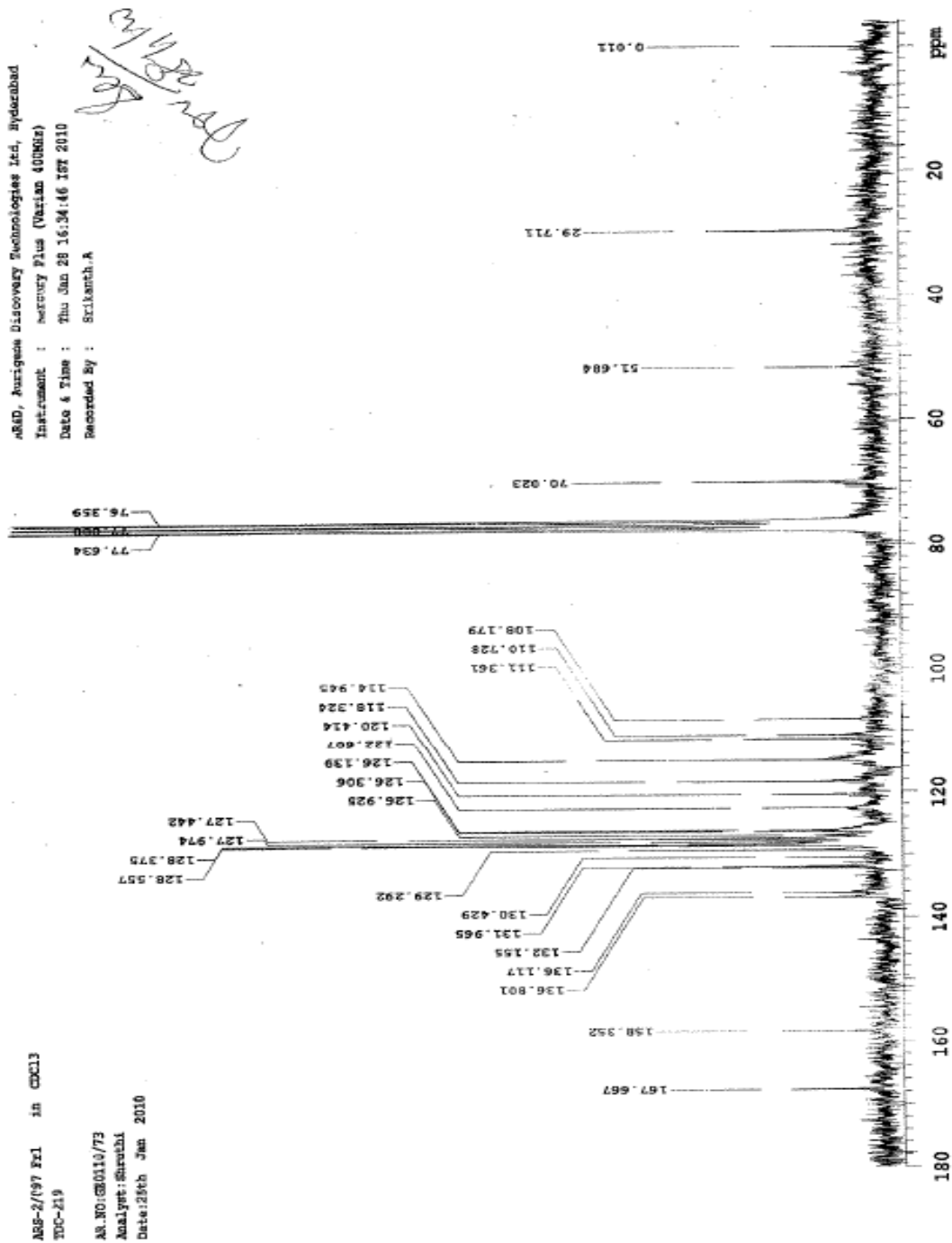


ARSD, Aurigene Discovery Technologies Ltd, Hyderabad

Instrument : Mercury Plus (Varian 400MHz)

Date & Time : Thu Jan 28 16:34:46 IST 2010

Recorded By : Srikanth.A



ASG-2/97 Fr1 in CDCl3

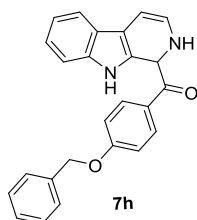
TIC-219

AS NO: GM1010/73

Analyst: Shreshth

Date: 28th Jan 2010

Mass spectrum of (4-(benzyloxy)phenyl)(2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**7h**):



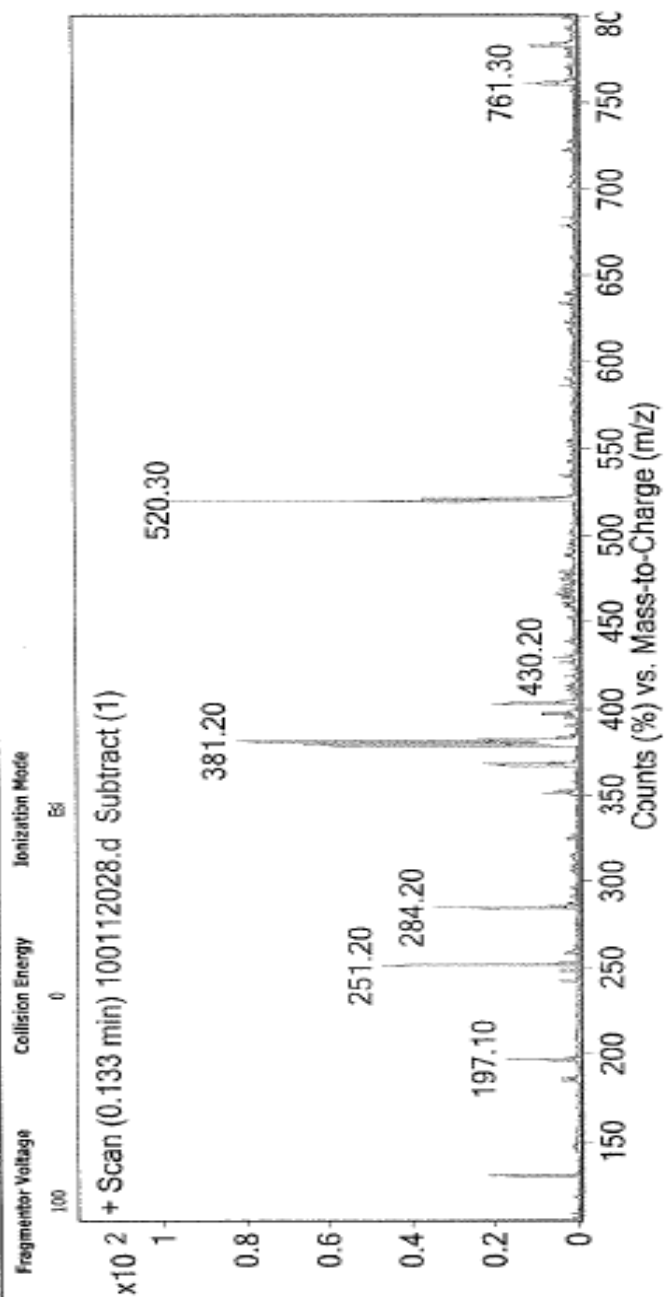
... End Of Report ...

Mass Analysis Report

CPS.MIYAPUR

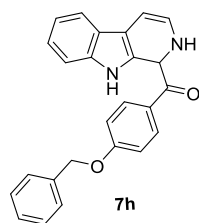
Data Filename	100112028.d	Sample Name	ARS-2/097 fr-1
Sample Type	Sample	Position	Vial 28
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	default.m	Comment	

User Spectra



... End Of Report ...

HRMS of (4-(benzyloxy)phenyl)(2,9-dihydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**7h**):



Page 1

Elemental Composition Report

Single Mass Analysis

Tolerance = 6.0 PPM / DBE: min = -1.5, max = 80.0

Element prediction: Off

Number of isotope peaks used for i-FIT = 3

Monoisotopic Mass, Even Electron Ions

20 formula(e) evaluated with 1 results within limits (up to 4 best isotopic matches for each mass)

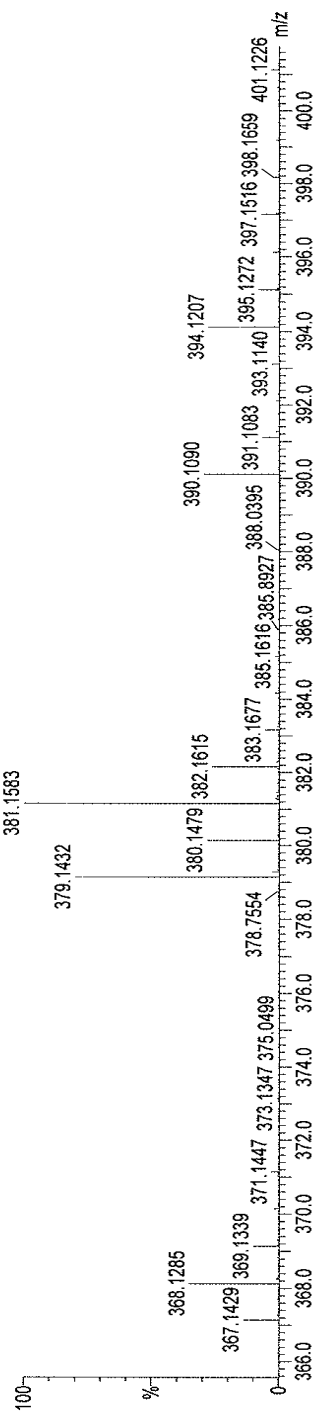
Elements Used:

C: 0-30 H: 0-25 N: 0-2 O: 0-2

9h

UT1212_178.6 (0.224) Cm (6:8)

Gajanan
1: TOF MS ES+
2.61e+003

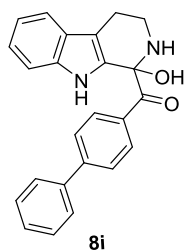


Minimum: -1.5
Maximum: 80.0

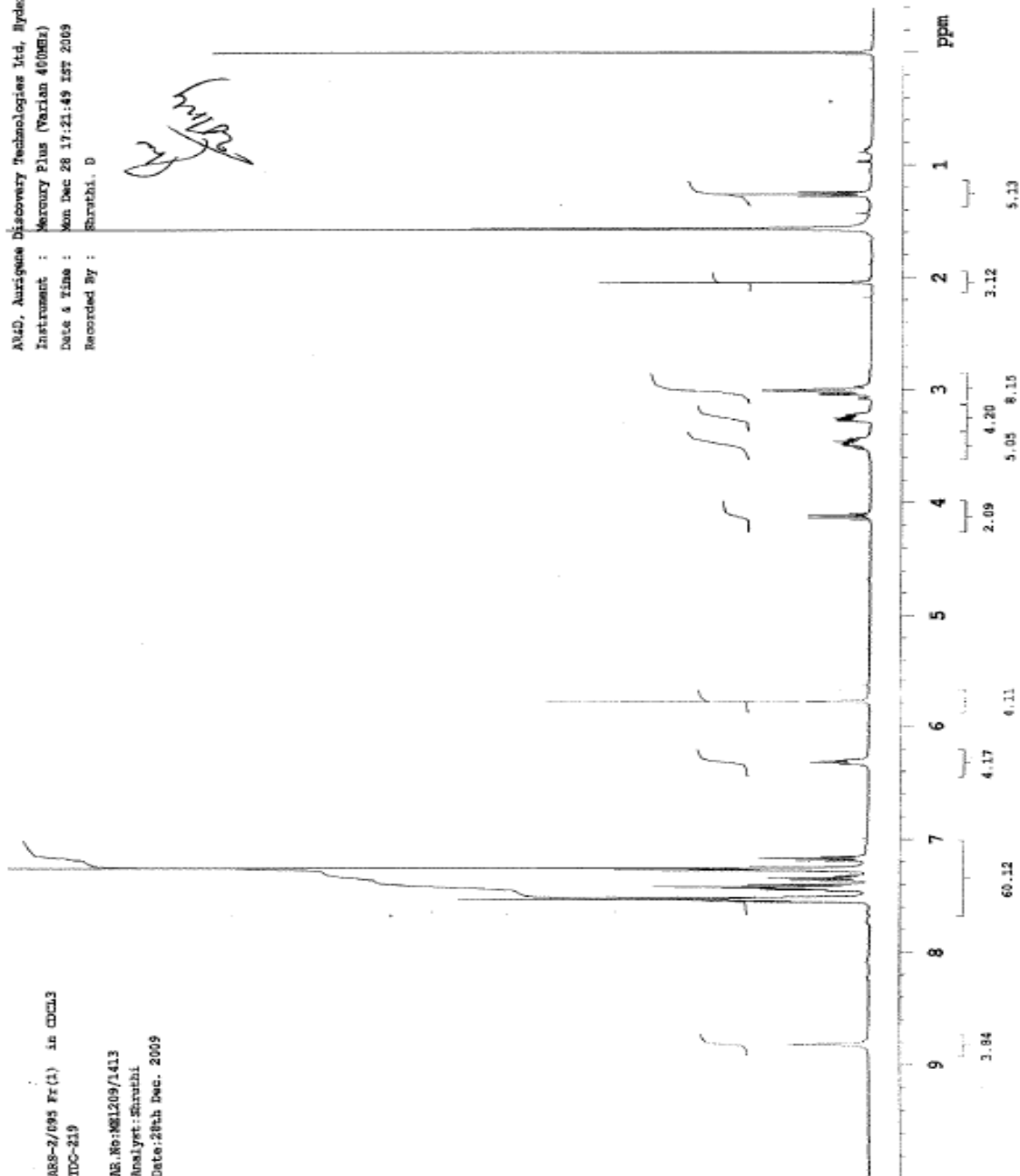
Mass Calc. Mass mDa PPM DBE i-FIT Formula

381.1583 381.1603 -2.0 -5.2 16.5 6.5 C₂₅ H₂₁ N₂ O₂

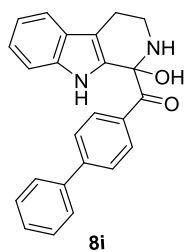
^1H NMR of [1,1'-biphenyl]-4-yl(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8i**):



AS4D, Ausigene Discovery Technologies Ltd, Hyderabad
 Instrument : Mercury Plus (Varian 400MHz)
 Date & Time : Mon Dec 28 17:21:49 IST 2009
 Recorded By : Sharathi. D



Mass spectrum of [1,1'-biphenyl]-4-yl(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8i**):

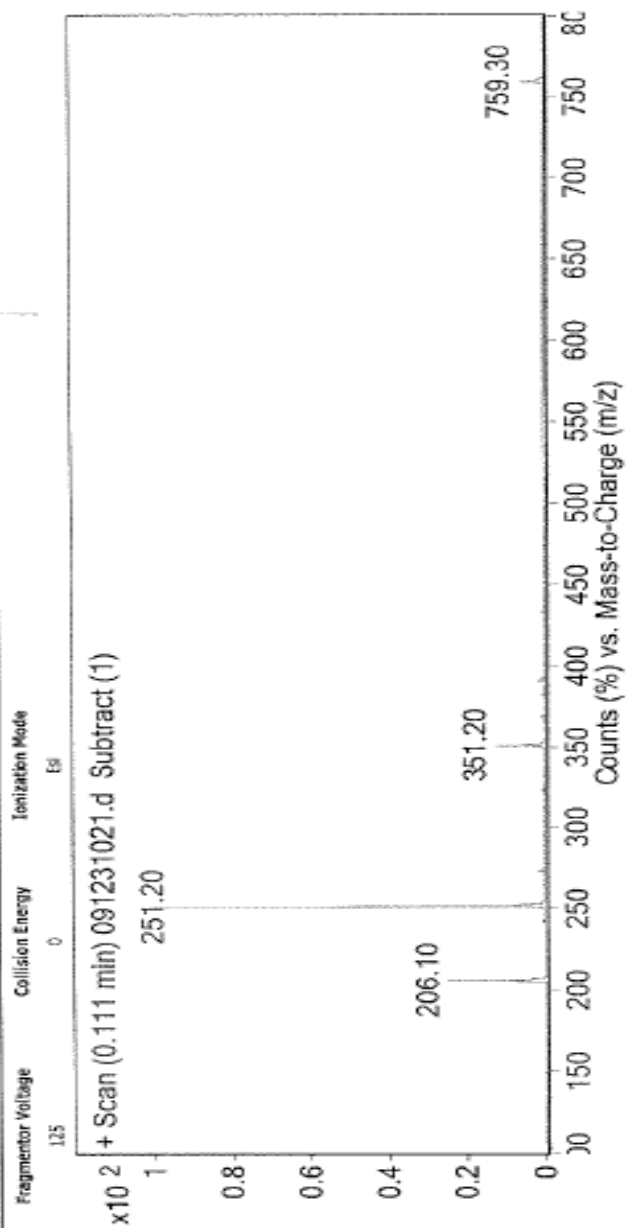


Mass Analysis Report

CPS,MIYAPUR

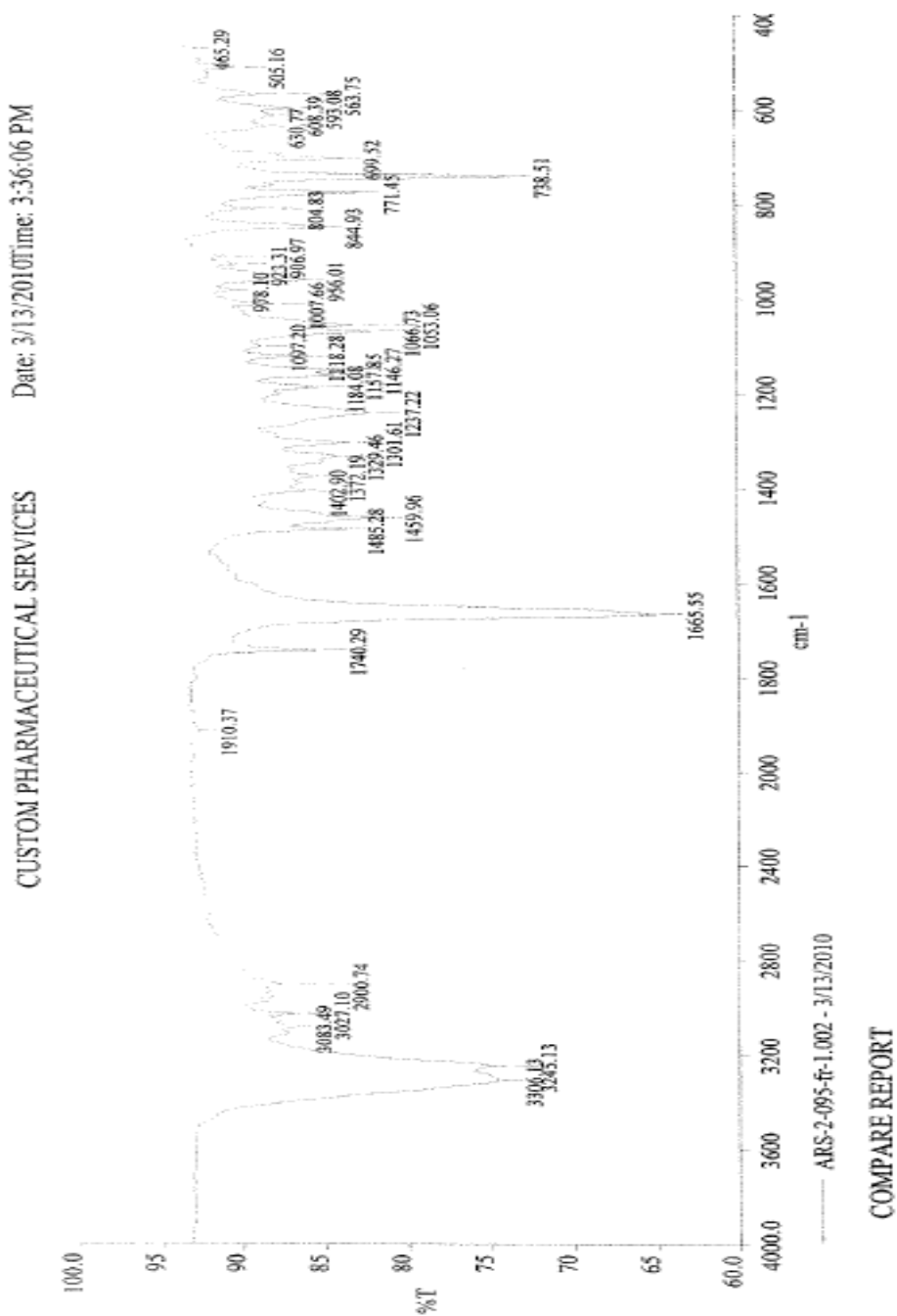
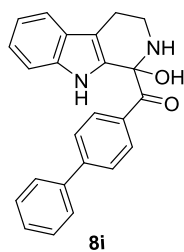
Data Filename: 091231021.d
 Sample Name: AQS-2/095 Fr-1
 Sample Type: Sample
 Instrument Name: Instrument 1
 Acq Method: ESI.m
 DA Method: default.m
 Position: Vial 18
 User Name: Success
 IRM Calibration Status: Success
 Comment:

User Spectra

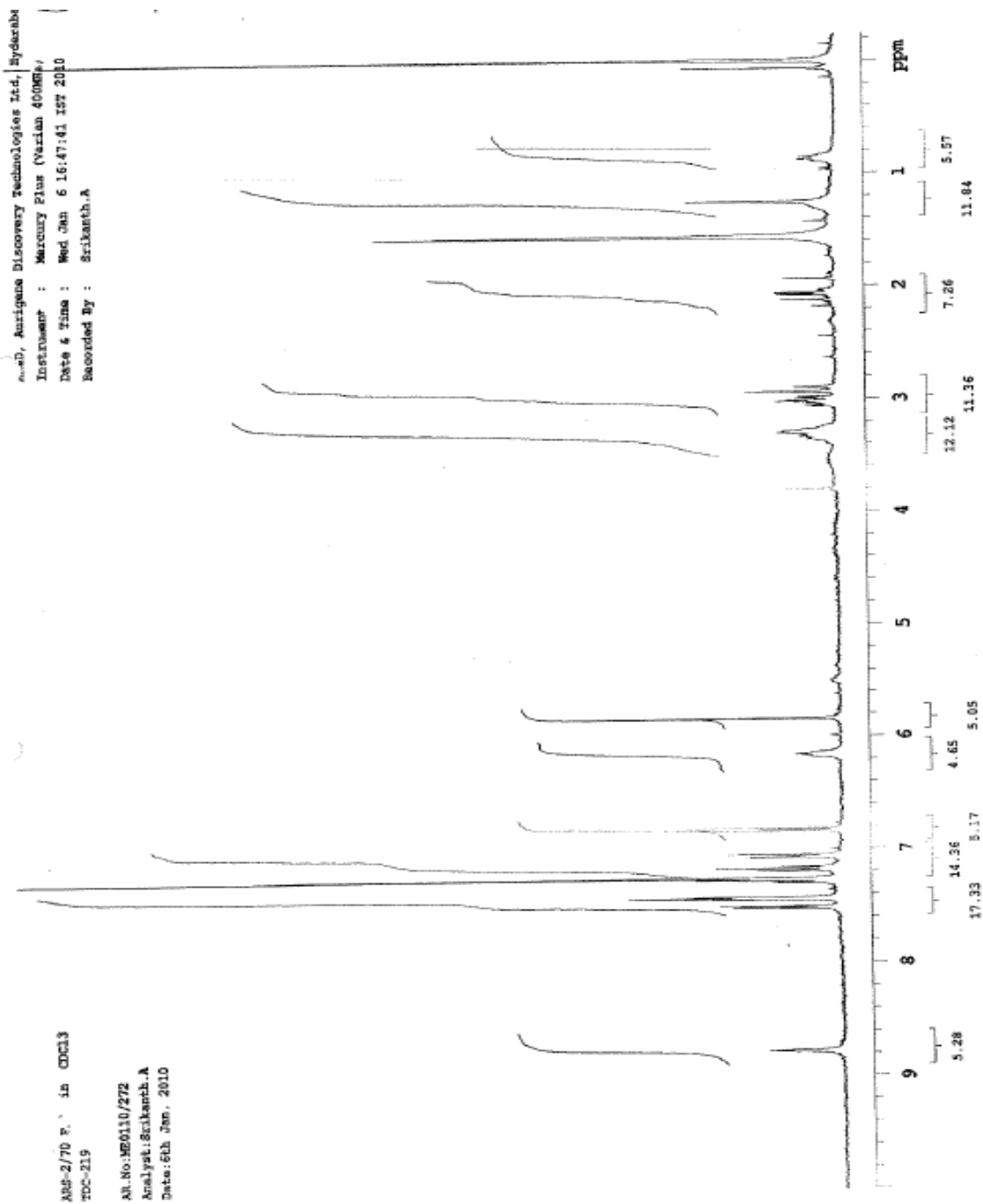
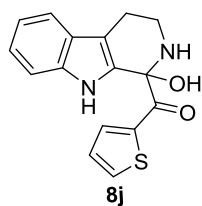


--- End Of Report ---

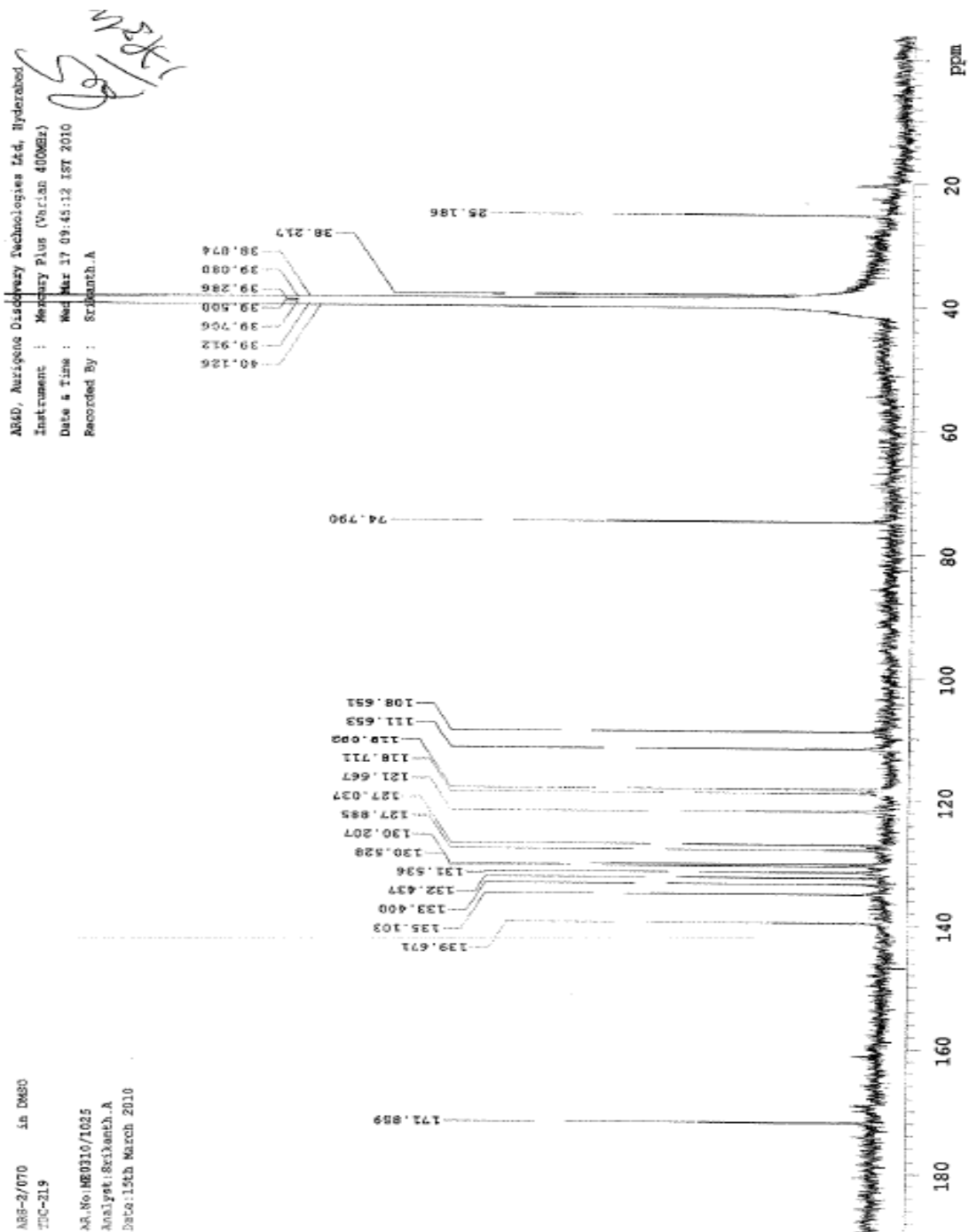
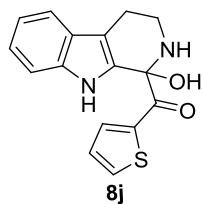
IR spectrum of [1,1'-biphenyl]-4-yl(1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)methanone (**8i**):



^1H NMR of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(thiophen-2-yl)methanone (**8j**):

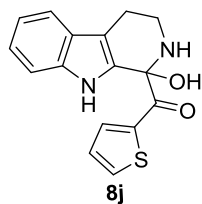


^{13}C NMR of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(thiophen-2-yl)methanone (**8j**):



AB3-2/070 In DMSO
 TPC-219
 Ad.No:ME0310/1025
 Analyst:Srikanth.A
 Date:15th March 2010

Mass spectrum of (1-hydroxy-2,3,4,9-tetrahydro-1*H*-pyrido[3,4-*b*]indol-1-yl)(thiophen-2-yl)methanone (**8j**):

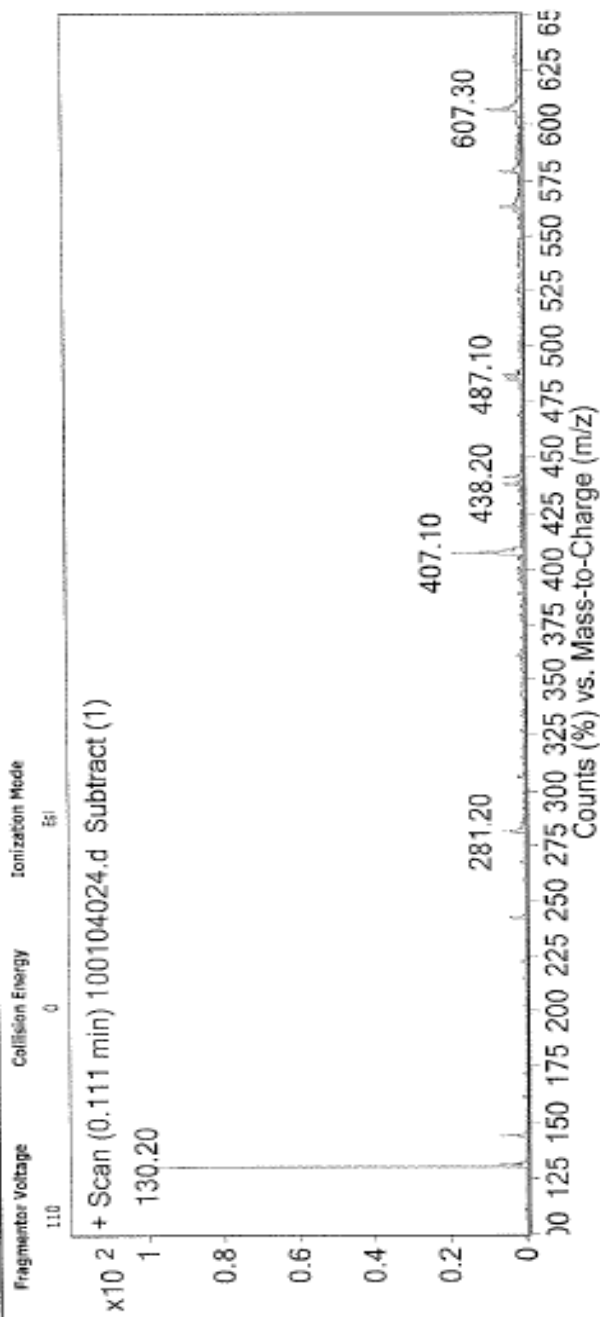


CPS,MIYAPUR

Mass Analysis Report

Data Filename	100104024.d	Sample Name	ARS-2/070
Sample Type	Sample	Position	Vial 21
Instrument Name	Instrument 1	User Name	
Acq Method	ESI.m	IRM Calibration Status	Success
DA Method	default.m	Comment	

User Spectra



... End Of Report ...