Organic Process Research & Development

Improved Process for the Preparation of 1-Benzhydrylazetidin-3-ol: Development of an Efficient Synthesis and Identification of Process related Impurities and/or Intermediates

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Supporting Information

1-Benzhydrylazetidin-3-ol, 1 (Purity by HPLC):

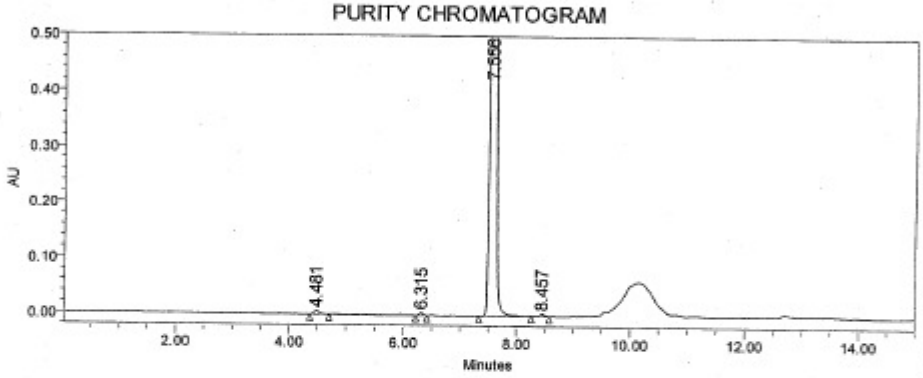
[redacted]

INOGENT LABORATORIES PRIVATE LIMITED
ANALYTICAL DEVELOPMENT LABORATORY

Sample Name : DEN/BHA/001/07-08
Acq. Method Set : CCS
Date Acquired : 3/12/2008 11:21:16 PM IST
Acquired By : System
Injection volume : 10µL
Run time : 15.00 Minutes
Vial no : Vial 31

System Name ADEQP 001 PDA
COLUMN: EclipseXDB-C18,100x4.6,3.5 µ
Processed Channel Descr. PDA 220.0 nm

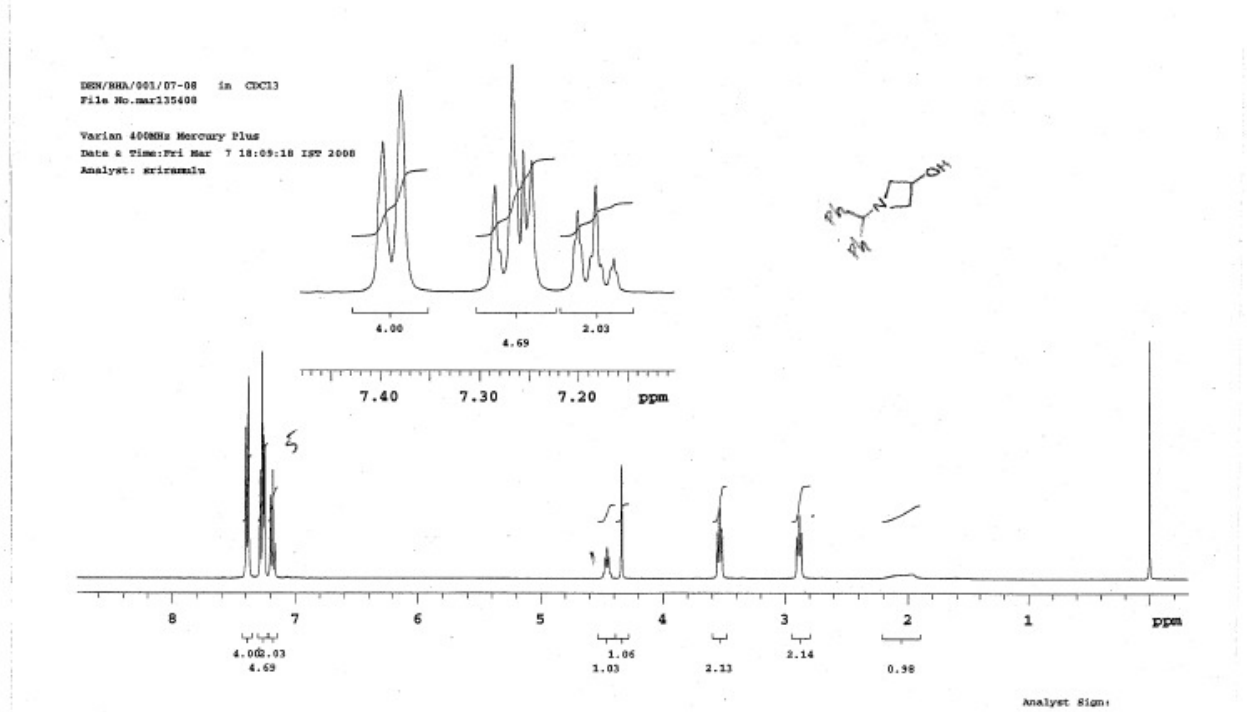
PURITY CHROMATOGRAM



	RT	Area	% Area
1	4.481	39753	0.31
2	6.315	22662	0.17
3	7.556	12873842	99.32
4	8.457	25379	0.20

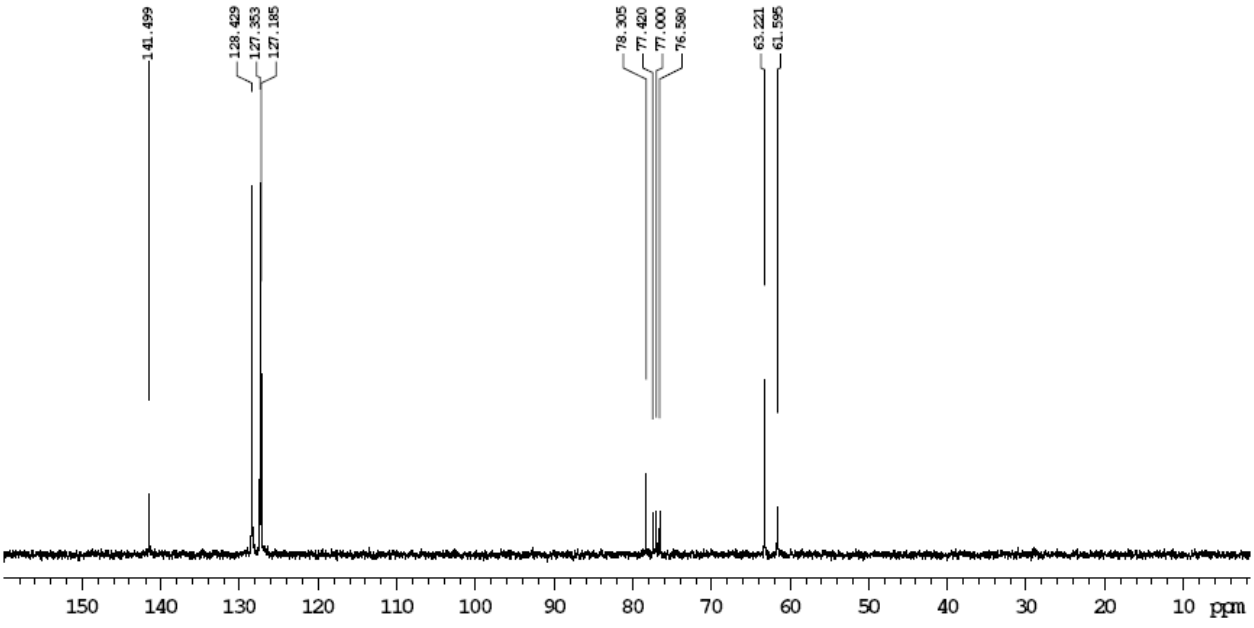
10/3/08

1-Benzhydrylazetidin-3-ol, 1 (^{1}H NMR):

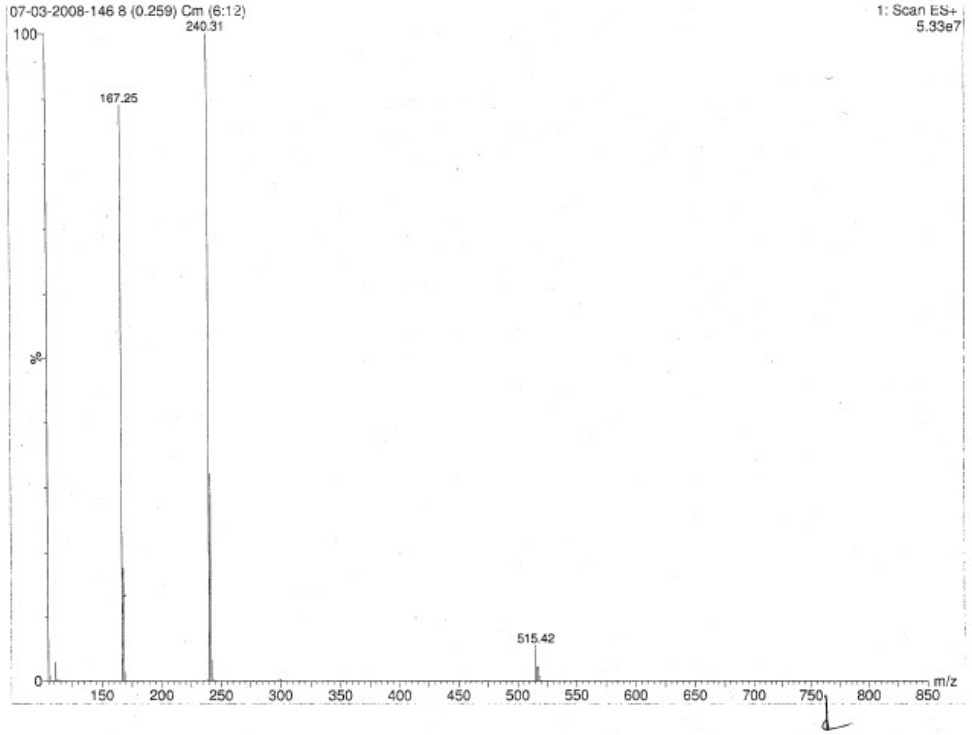


1-Benzhydrylazetidin-3-ol, 1 (^{13}C NMR):

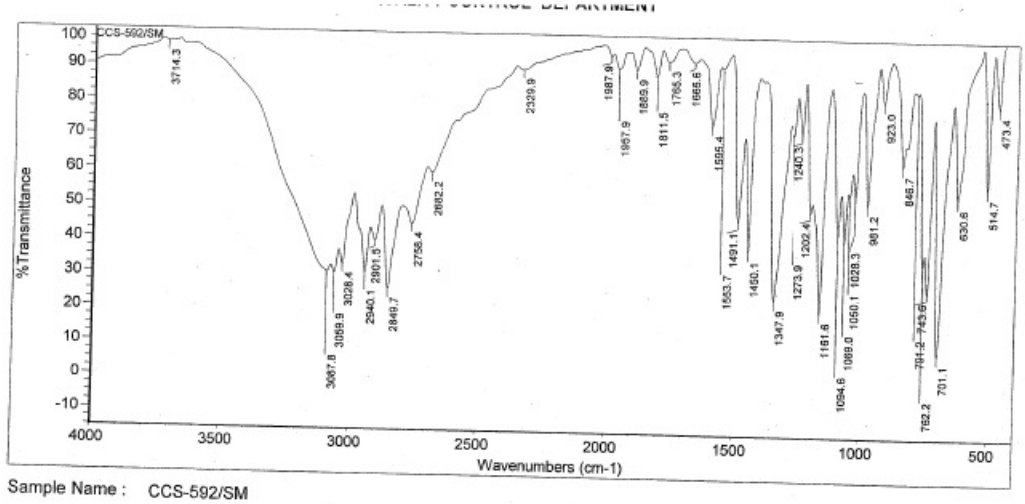
CCS-668-B346-BHA in CDCl3
13C Expt
File no:marin10410



1-Benzhydrylazetidin-3-ol, 1 (ESI-MS):



1-Benzhydrylazetidin-3-ol, 1 (IR):



Sample Name : CCS-592/SM

Collection time: Thu Mar 13 18:21:09 2008 (GMT+05:30)

Batch No: DEN/BHA/001/07-08

Number of sample scans: 16
Number of background scans: 16
Resolution: 4.000
Sample gain: 8.0
Mirror velocity: 0.6329
Aperture: 100.00

Analysed By:
Date: 18/3/08

Checked By:
Date: 18/3/08

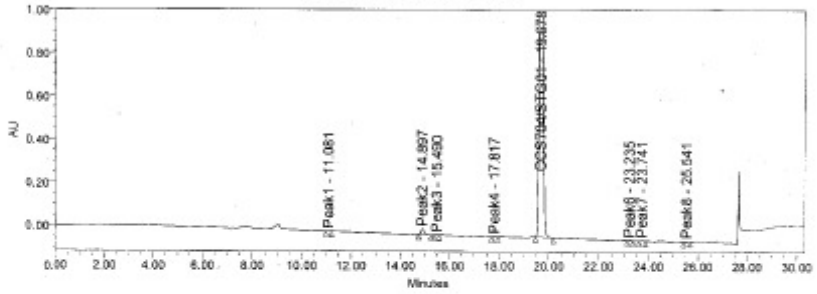
1-Benzhydrylazetidin-3-yl methanesulfonate, 8 (Purity by HPLC):

INOGENT LABORATORIES PRIVATE LIMITED
ANALYTICAL DEVELOPMENT LABORATORY

Sample Name : CCS704/STG-01/00209(R)
System Name ADEQP 002
Aq.Method Set : CCS704_INST
Data Acquired : 7/2/2009 11:13:16 AM IST
Processed Channel Descr. A1100 AU 210 nm
Injection volume : 10.00 uL
COLUMN INERTSILODS 150 x 4.6mm,5µm
Vial no : Vial 22
Run time : 30.00 Minutes

Mobile phase -A: 0.02M NH4HCO3 PH 7.0mobile phasese-B: ACN Flow : 1.0ml/min
Gradient programme: Time/%B:0.01/10,25/90,25.01/10,30/10

PURITY CHROMATOGRAM

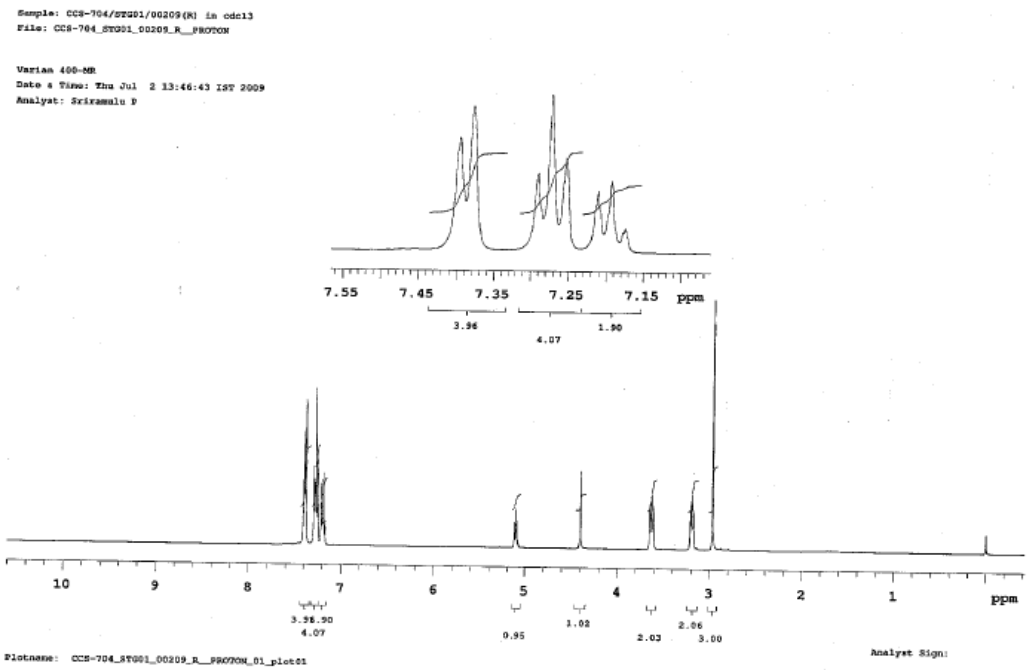


	RT	Area	% Area	RT Ratio	Name
1	11.081	5101	0.02	0.56	Peak1
2	14.897	208922	0.63	0.76	Peak2
3	15.490	3170	0.01	0.79	Peak3
4	17.817	13952	0.04	0.91	Peak4
5	19.678	32882865	99.21	1.00	CCS704/STG01
6	23.235	6795	0.02	1.18	Peak6
7	23.741	9265	0.03	1.21	Peak7
8	25.541	14857	0.04	1.30	Peak8

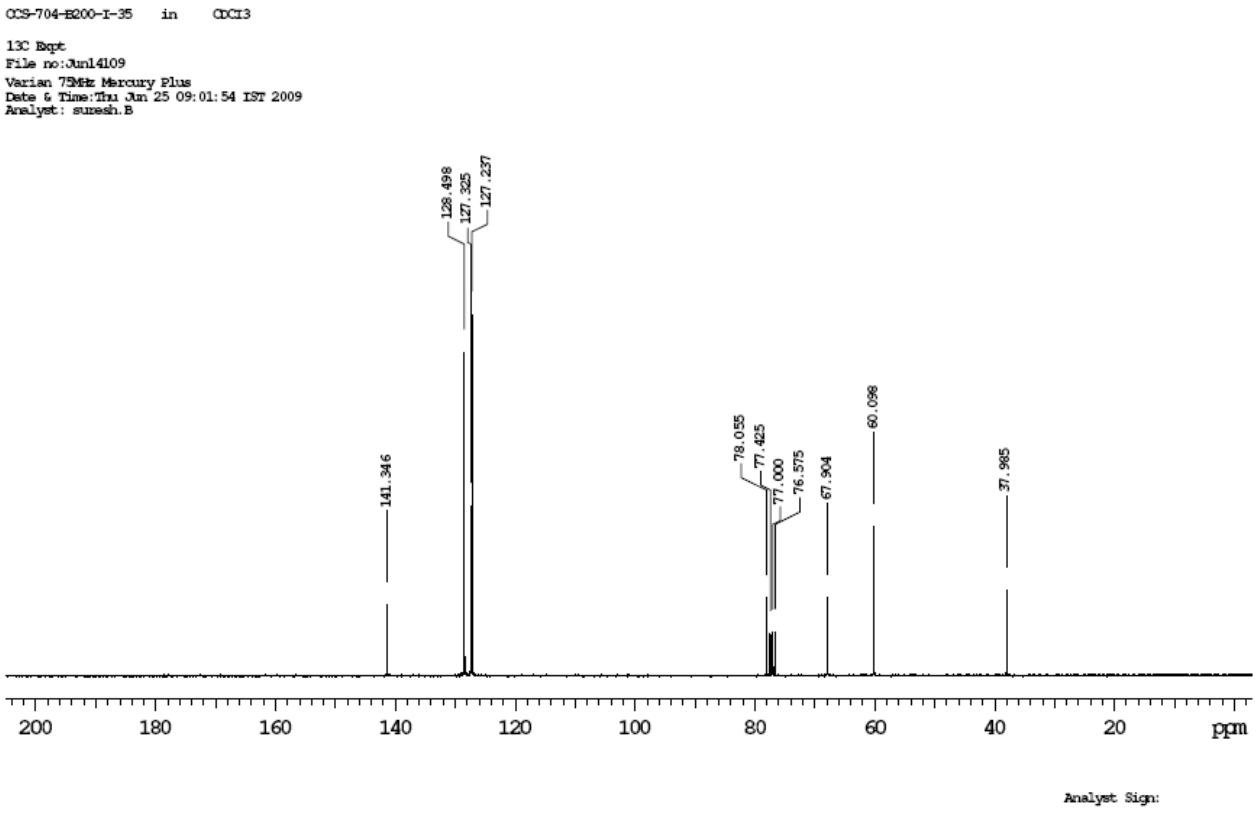
Tested by : 03/07/2009
Checked by 02/09/09

Report Method: CCS704_RPT
Page: 1 of 1

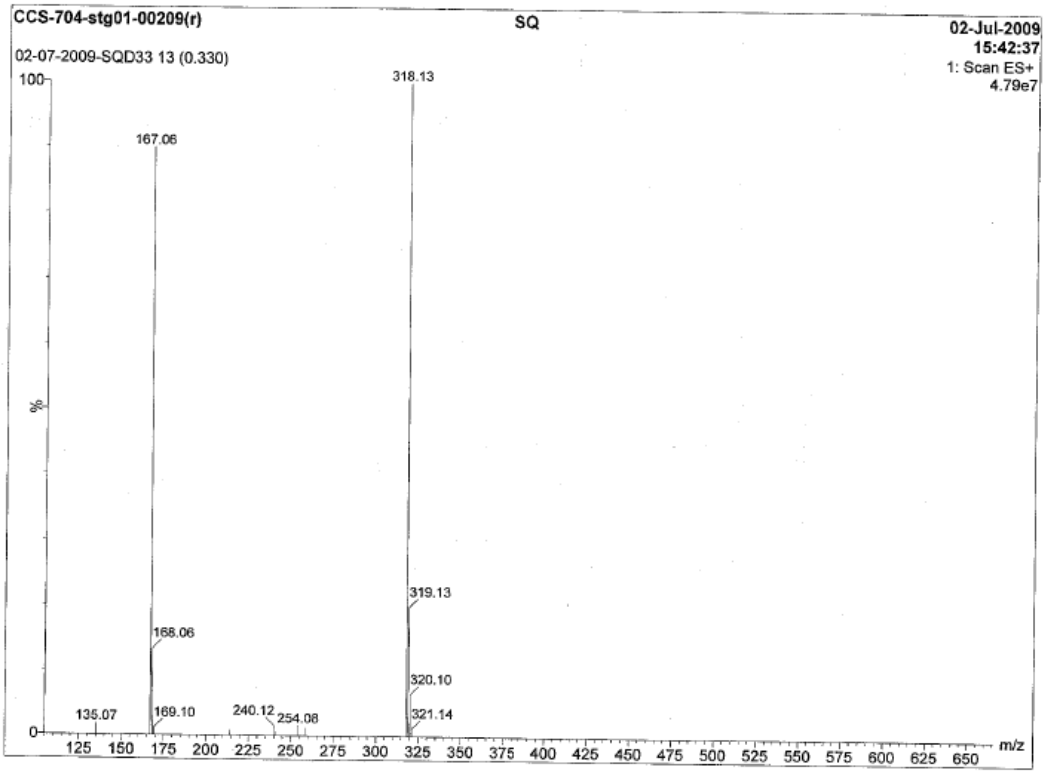
1-Benzhydrylazetidin-3-yl methanesulfonate, 8 (^{1}H NMR):



1-Benzhydrylazetidin-3-yl methanesulfonate, 8 (^{13}C NMR):



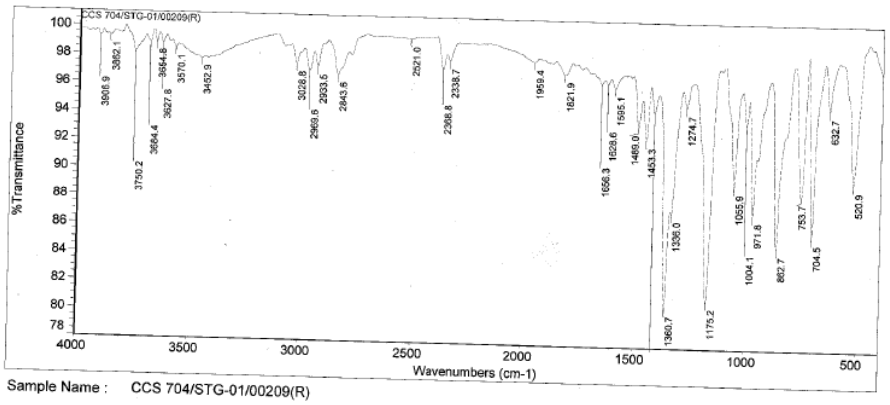
1-Benzhydrylazetidin-3-yl methanesulfonate, 8 (ESI-MS):



1-Benzhydrylazetidin-3-yl methanesulfonate, 8 (IR):

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Sample Name : CCS 704/STG-01/00209(R)

Collection time: Thu Jul 02 11:48:00 2009 (GMT+05:30)

Standard :

Analysed By :

Date : 02/07/09

Number of sample scans: 32
Number of background scans: 32
Resolution: 4.000
Sample gain: 4.0
Optical velocity: 0.6329
Aperture: 100.00

Checked By :

Date : 02/07/2009

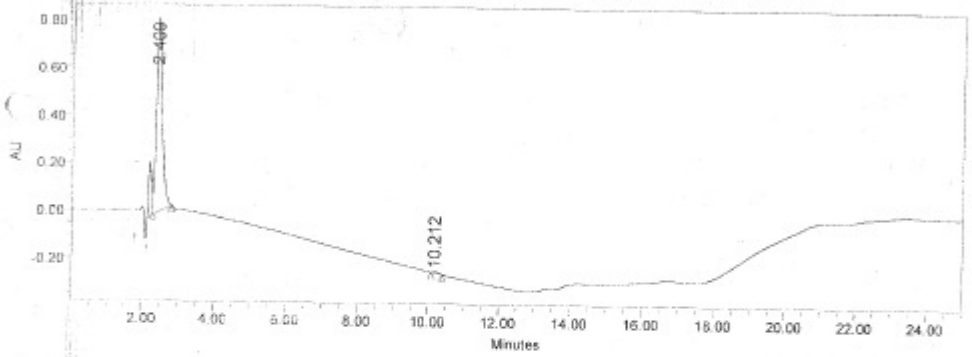
Azetidin-3-ol hydrochloride, 9 (Purity by HPLC):

INOGENT LABORATORIES PRIVATE LIMITED
ANALYTICAL DEVELOPMENT LABORATORY

Sample Name	: CCS-668/B210/IA HCl/04	System Name	ADEQP016_PDA
Aq Method Set	: CCS668_INST		
Data Acquired	: 2/9/2009 11:52:10 AM IST	Processed Channel Descr.	PDA 200.0 nm
Injection volume	: 10.00 uL	COLUMN	Inertsil ODS, 250 x 4.6mm,5µm
Vial no	: Vial 4	Run time	: 25.00 Minutes

Mobile phase -A: 0.02M NH4HCO3 PH 7.0mobile phasese-B: ACN Flow :1.0ml/min
Gradient programme: Time/%B:0.01/30,10/70,15/70,20/30,25/30

PURITY CHROMATOGRAM



	RT (min)	Area (µV*sec)	% Area
1	2.409	9486931	99.31
2	10.212	65785	0.69

Tested by 18.02.09

Checked by 18/02/09

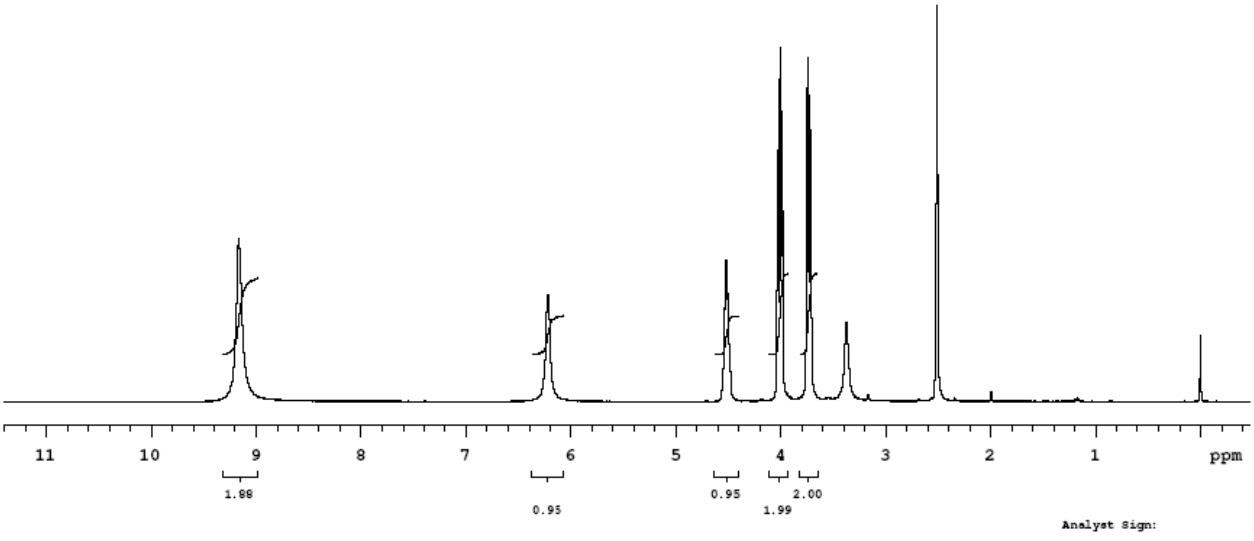
Report Method: CCS668_RPT

Page: 1 of 1

Azetidin-3-ol hydrochloride, 9 (^{1}H NMR):

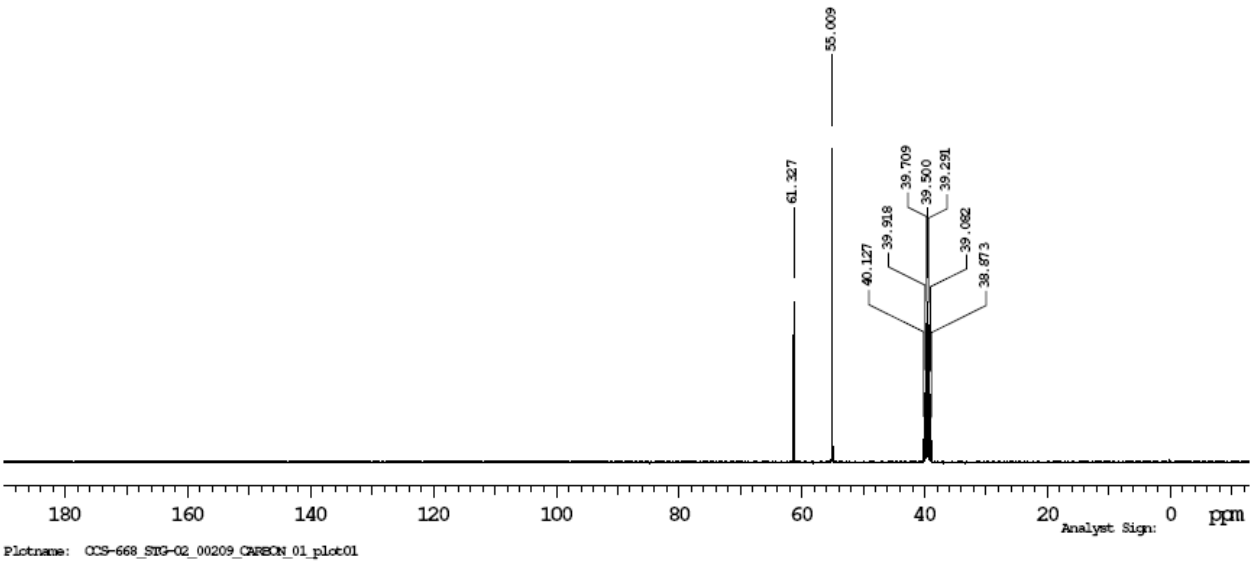
Sample: CCS-668/STG-02/00209 in dmso
File: CCS-668_STG-02_00209_PROTON

Varian 400-MR
Date & Time: Tue Jun 9 15:05:42 IST 2009
Analyst: Sriramulu P



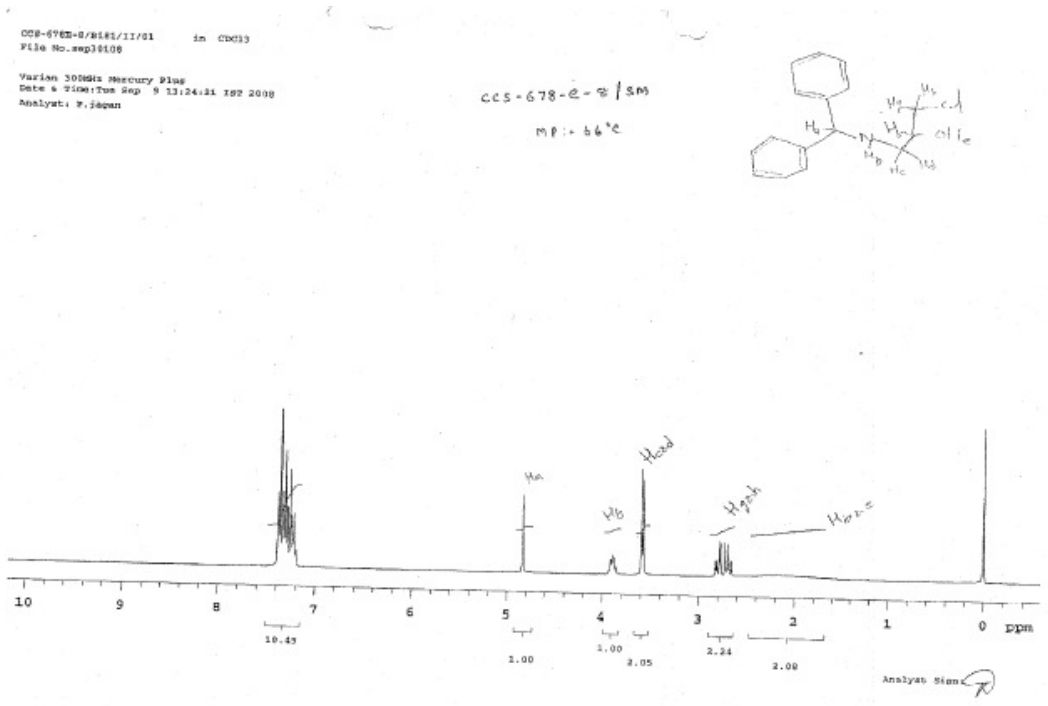
Plotname: CCS-668_STG-02_00209_PROTON_01_plot01

Azetidin-3-ol hydrochloride, 9 (^{13}C NMR):

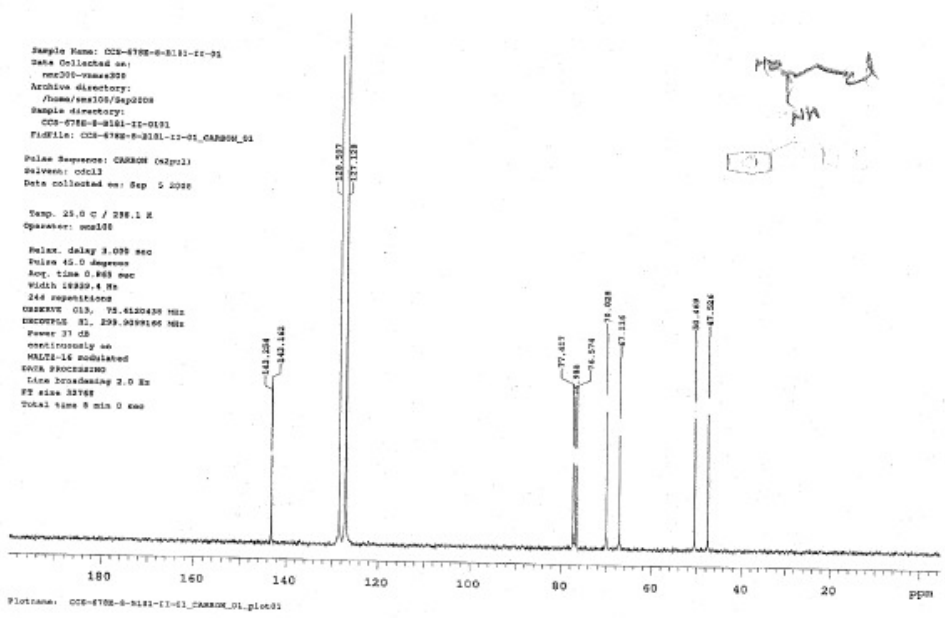


Plotname: CCS-668_STG-02_00209_CARBON_01_plot01

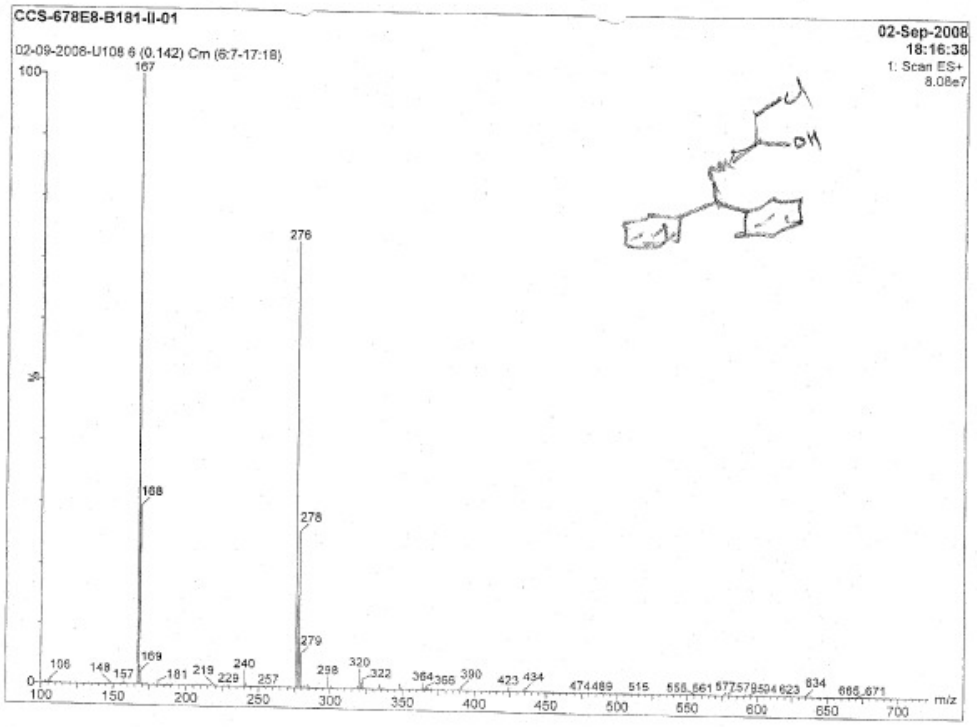
3-Chloro-1-diphenylmethyl amino-2-hydroxypropane, 4a (^{1}H NMR):



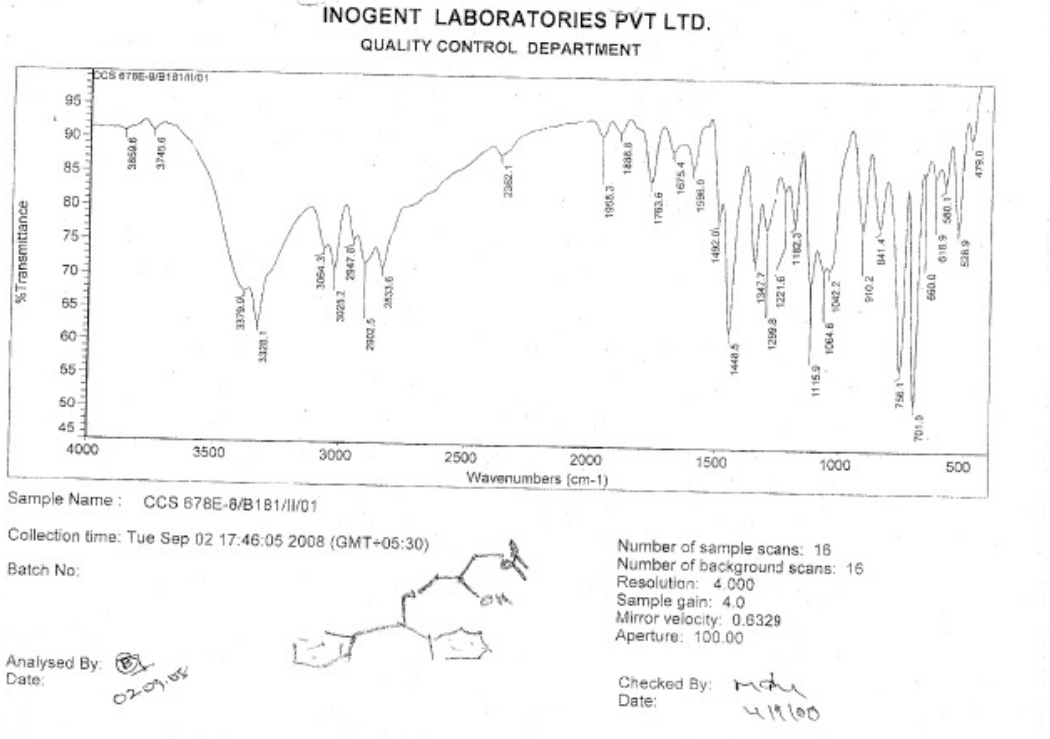
3-Chloro-1-diphenylmethyl amino-2-hydroxypropane, 4a (^{13}C NMR):



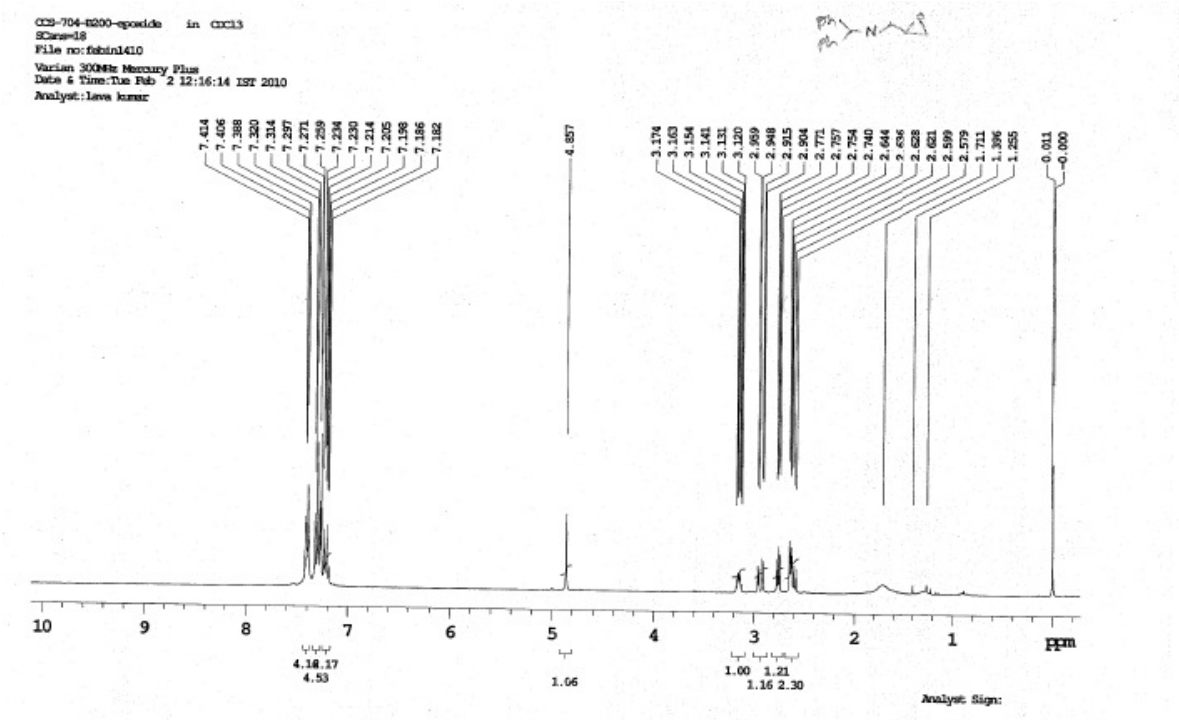
3-Chloro-1-diphenylmethyl amino-2-hydroxypropane, 4a (ESI-MS):



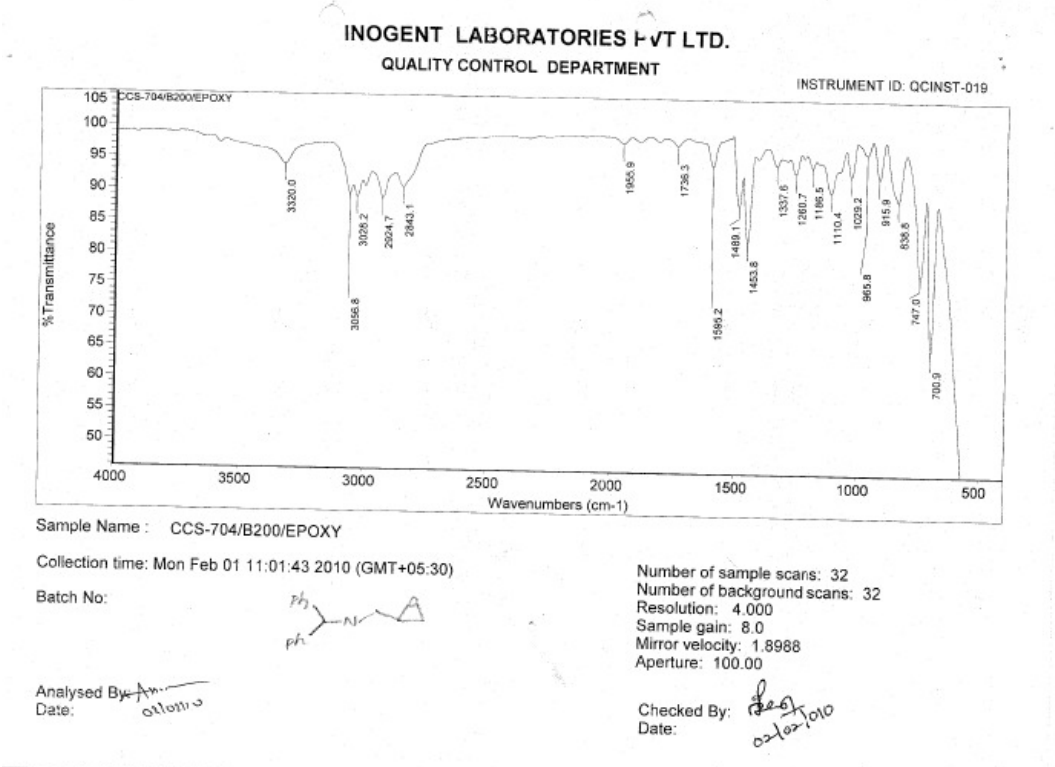
3-Chloro-1-diphenylmethyl amino-2-hydroxypropane, 4a (IR):



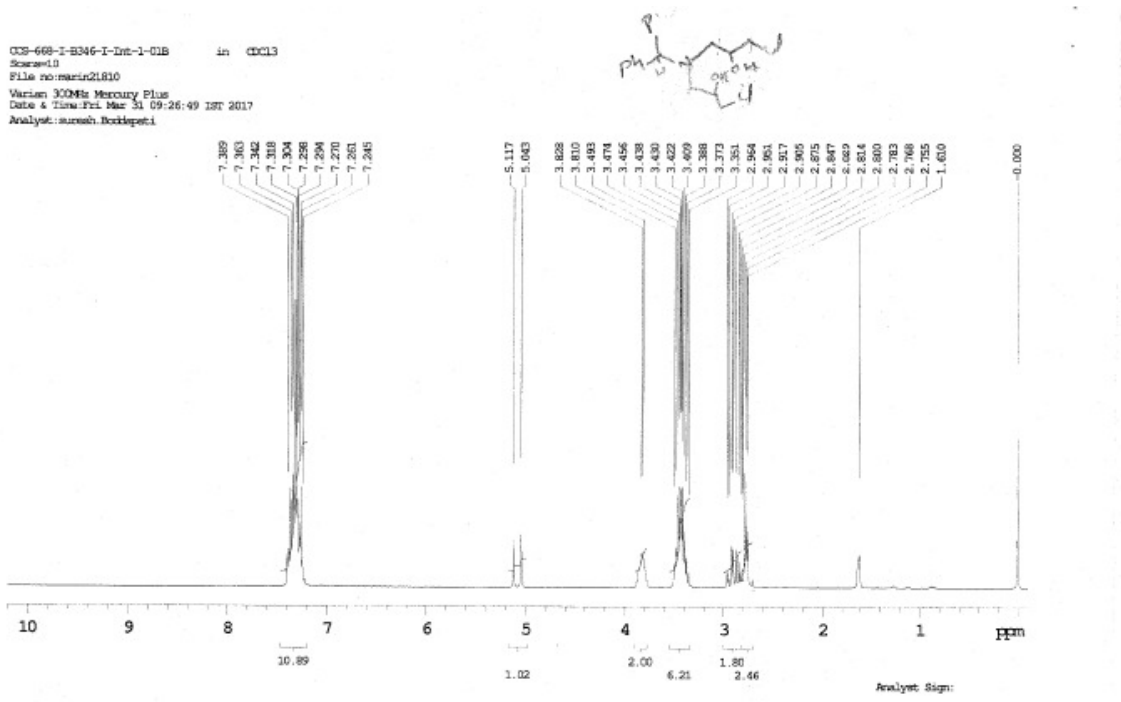
N-((Oxiran-2-yl)methyl)diphenylmethanamine, 5a (^{1}H NMR):



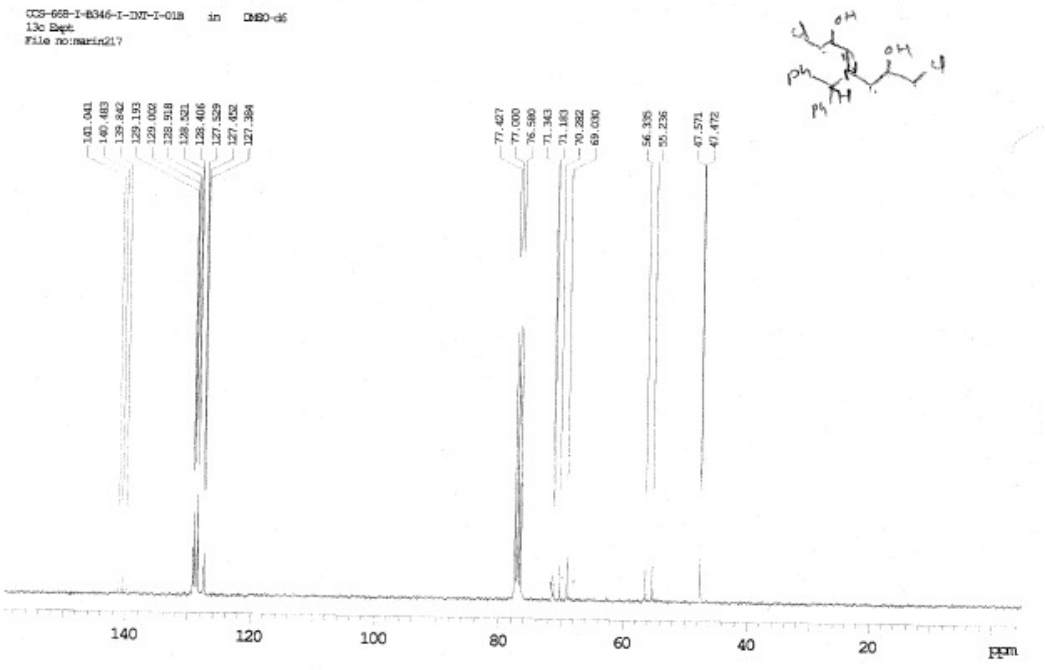
N-((Oxiran-2-yl)methyl)diphenylmethanamine, 5a (IR):



Bis impurity, 6b (^{1}H NMR):



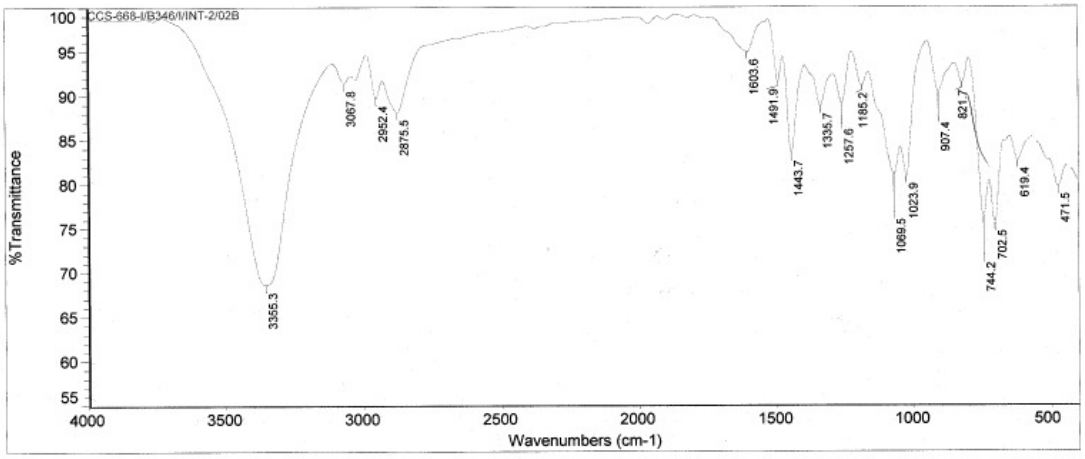
Bis impurity, 6b (^{13}C NMR):



Bis impurity, 6b (IR):

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ANALYTICAL DEVELOPMENT LABORATORY



Sample Name : CCS-668-I/B346/I/INT-2/02B

Collection time: Wed Apr 14 14:35:20 2010 (GMT+05:30)

Standard :

Analysed By :

Date : 14/04/10

Number of sample scans: 32
Number of background scans: 32
Resolution: 4.000
Sample gain: 2.0
Optical velocity: 0.6329
Aperture: 100.00

Checked By :

Date :

Bis impurity, 6b (ESI-MS):

