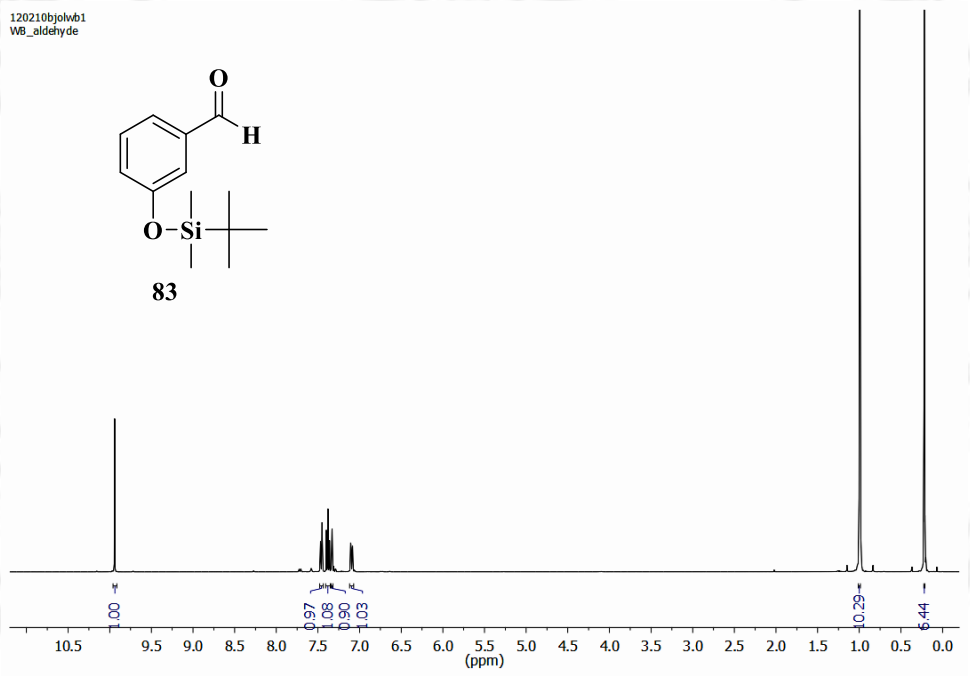
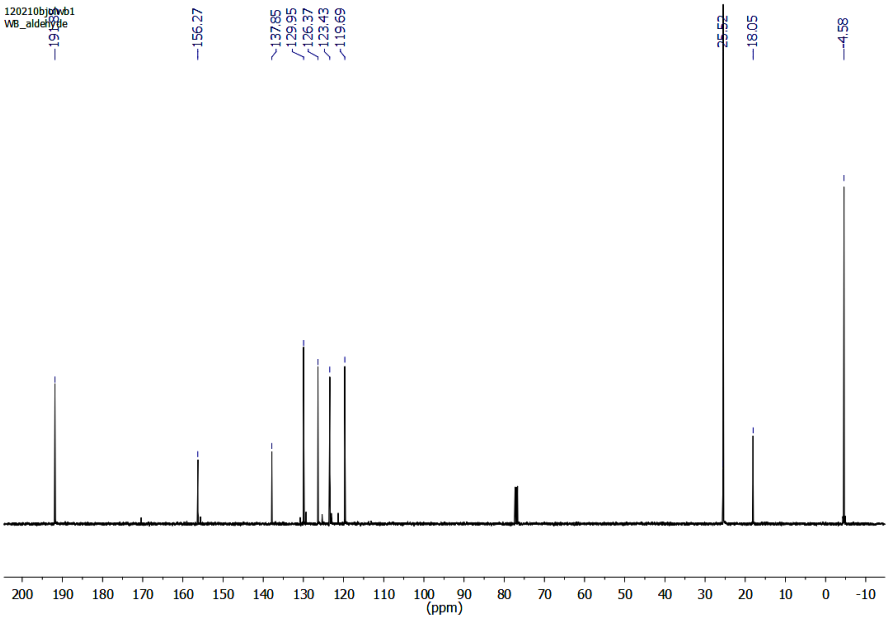
APPENDIX

(¹H-NMR 400 MHz, ¹³C-NMR 100 MHz, IR and HRMS)

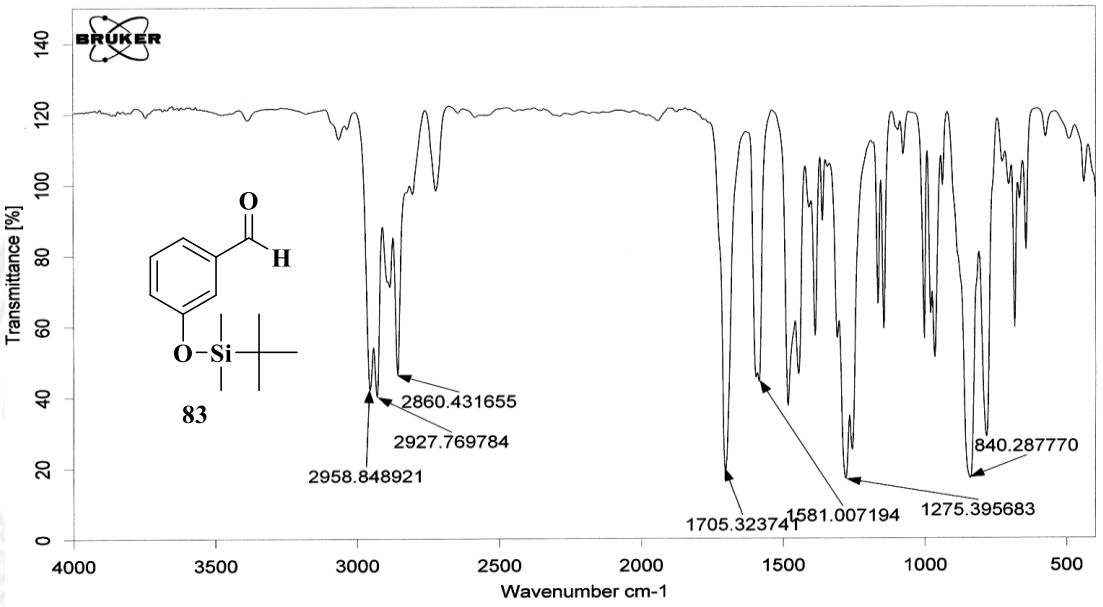
¹H-NMR in $CDCl_3$



¹³C-NMR in $CDCl_3$



FT-IR (neat)



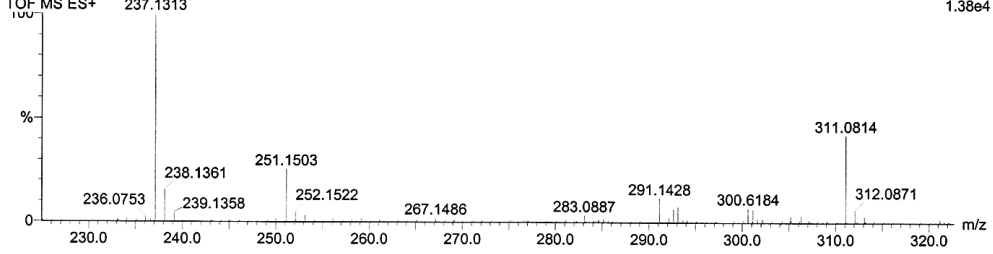
HRMS

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 250.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

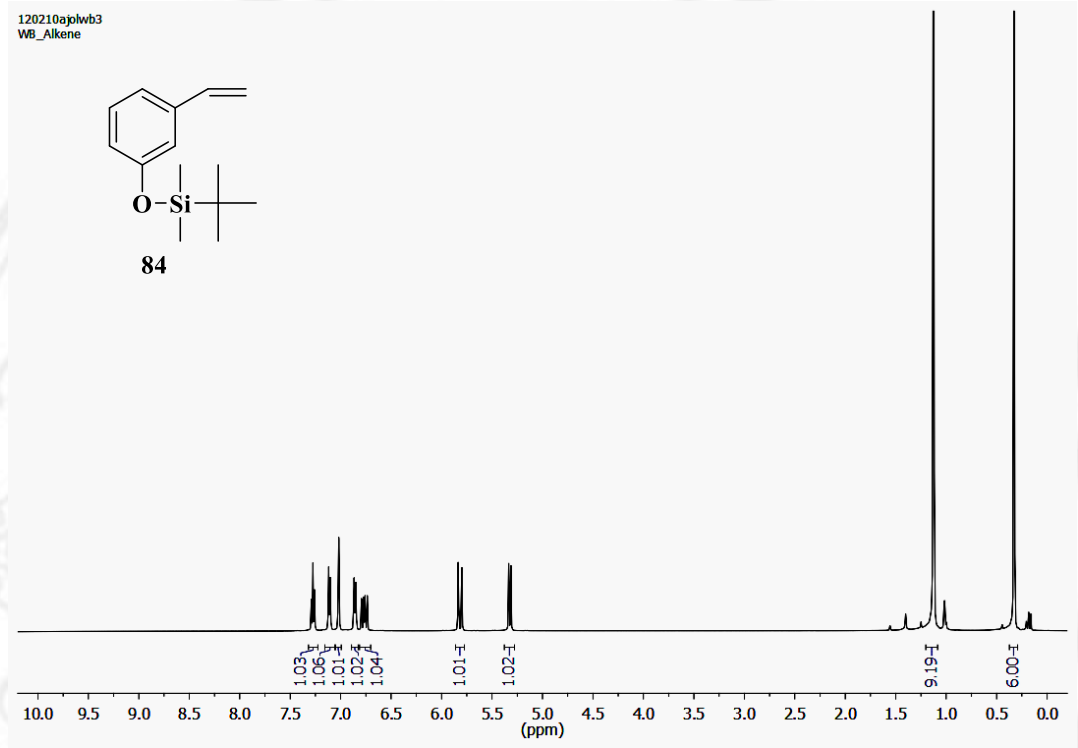
Monoisotopic Mass, Odd and Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

TS_S164_236_SULFADIMETHOXINE 421 (7.810) AM (Cen,4, 80.00, Ar,10000.0,311.08,0.00); Cm (397:434)
TOF MS ES+ 237.1313 1.38e4

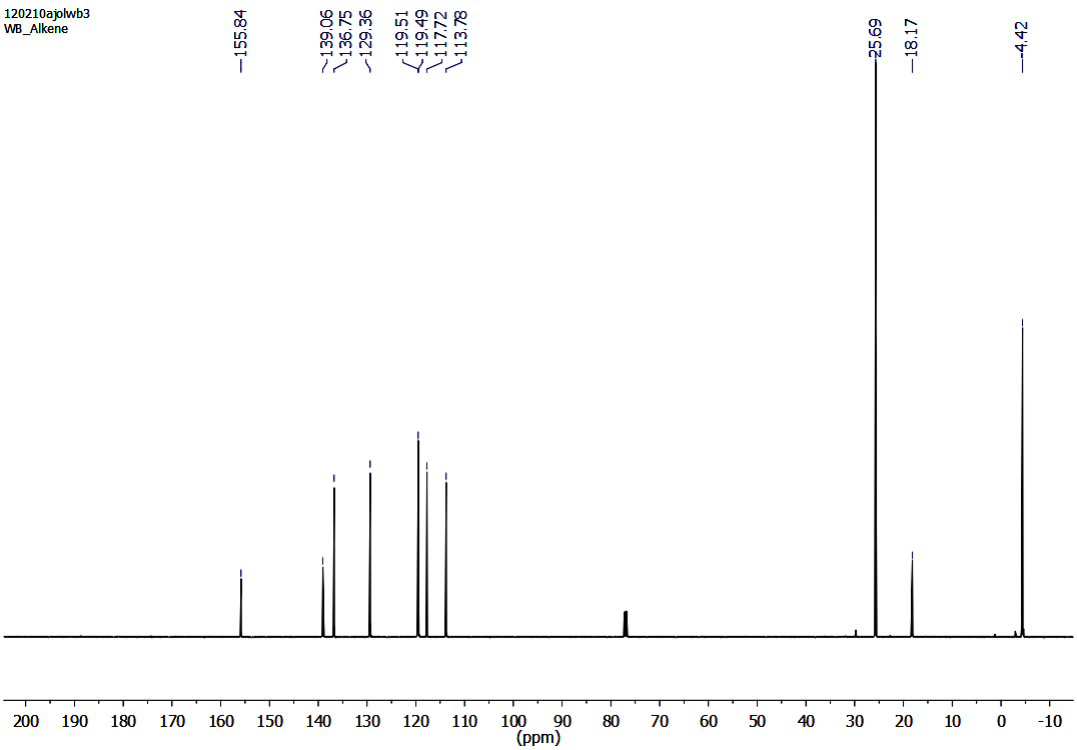


Minimum:				-1.5		
Maximum:		200.0	250.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
237.1313	237.1311	0.2	0.9	4.5	1	C13 H21 O2 Si

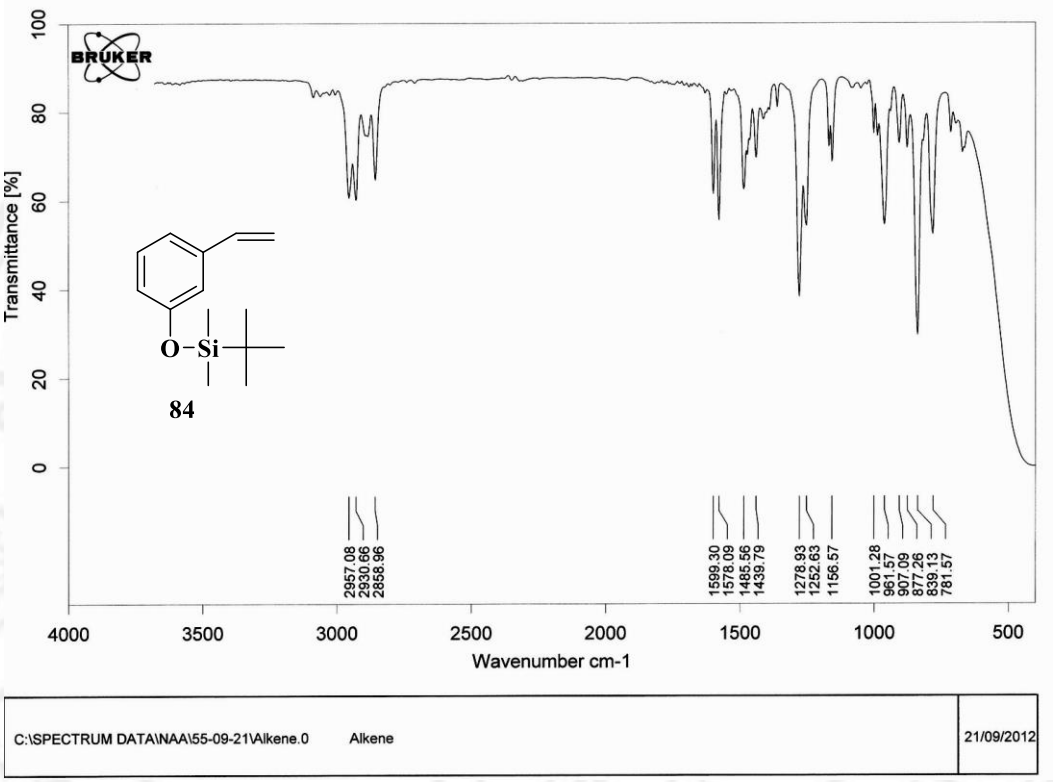
^{1}H-NMR in $CDCl_3$



^{13}C-NMR in $CDCl_3$



FT-IR (neat)



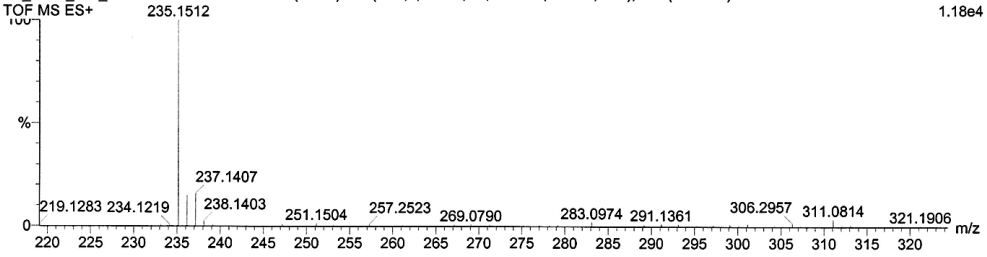
HRMS

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 250.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

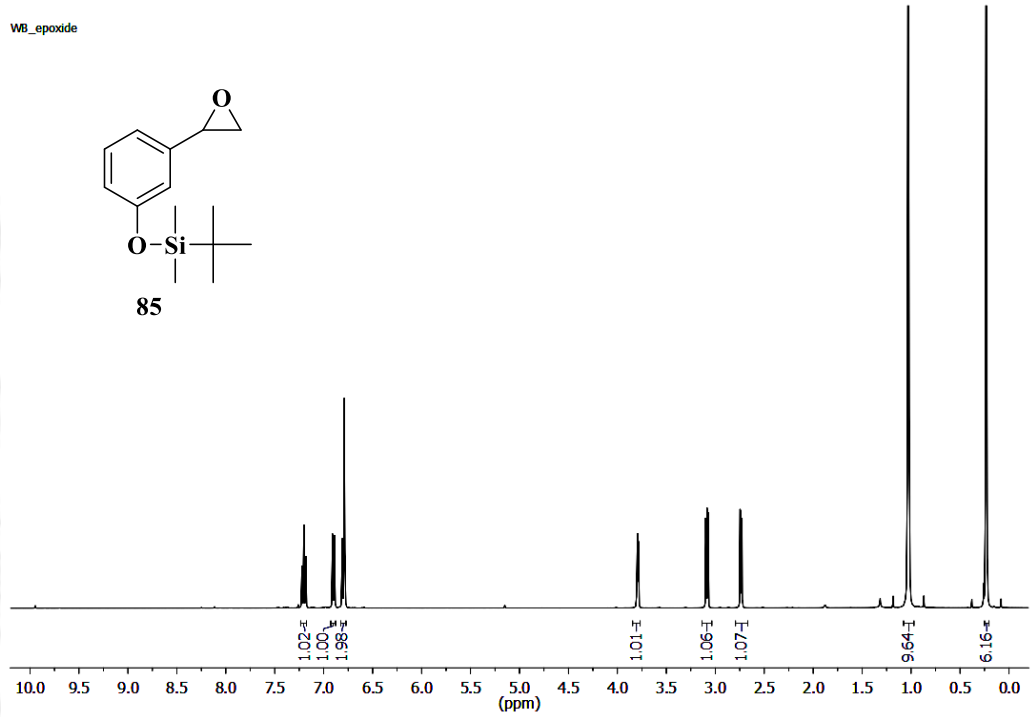
Monoisotopic Mass, Odd and Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

TS_S165_235_SULFADIMETHOXINE 298 (5.530) AM (Cen,4, 80.00, Ar,10000.0,311.08,0.00); Cm (278:308)
TOF MS ES+ 235.1512 1.18e4

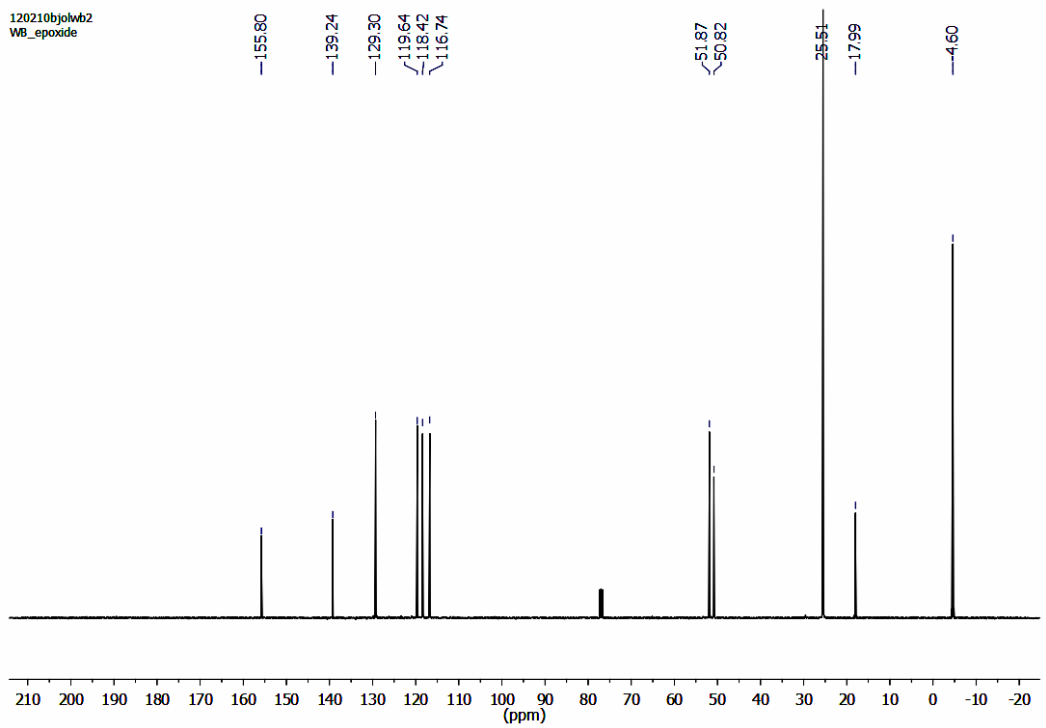


Minimum:				-1.5		
Maximum:		200.0	250.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
235.1512	235.1518	-0.6	-2.6	4.5	1	C14 H23 O Si

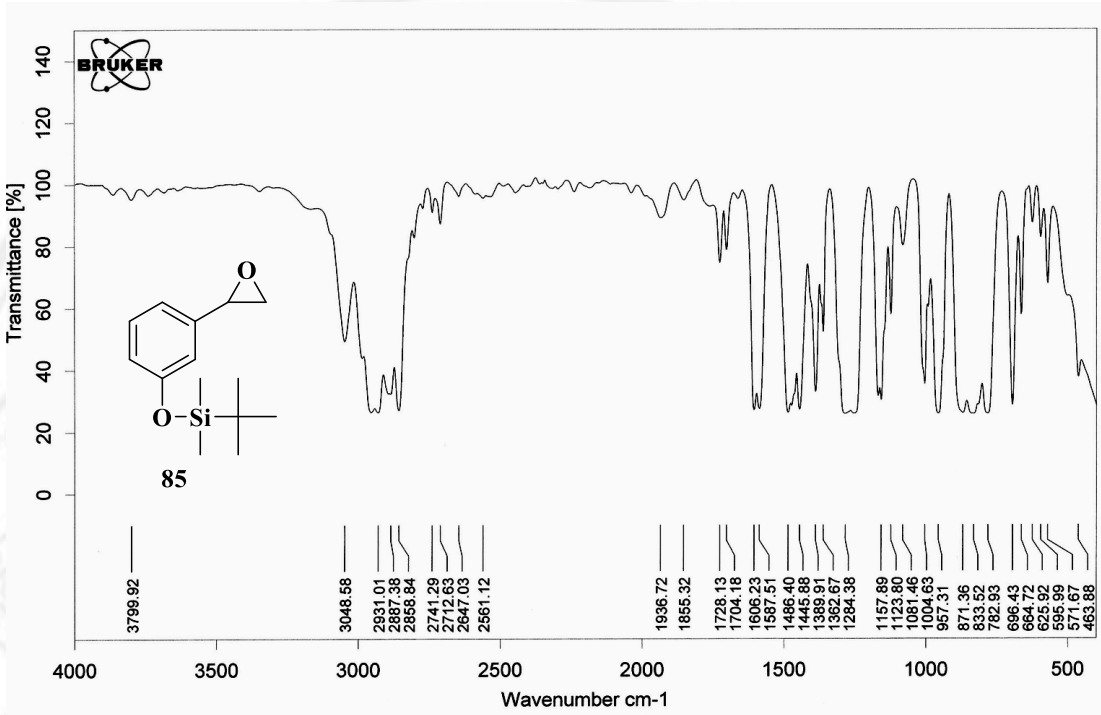
^{1}H-NMR in $CDCl_3$



^{13}C-NMR in $CDCl_3$



FT-IR (neat)



C:\spectrum data\NAA\EPOXIDE.0	EPOXIDE	27/10/2010

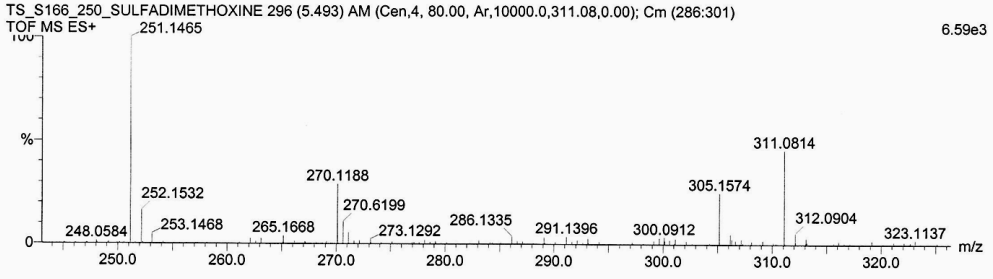
HRMS

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 250.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

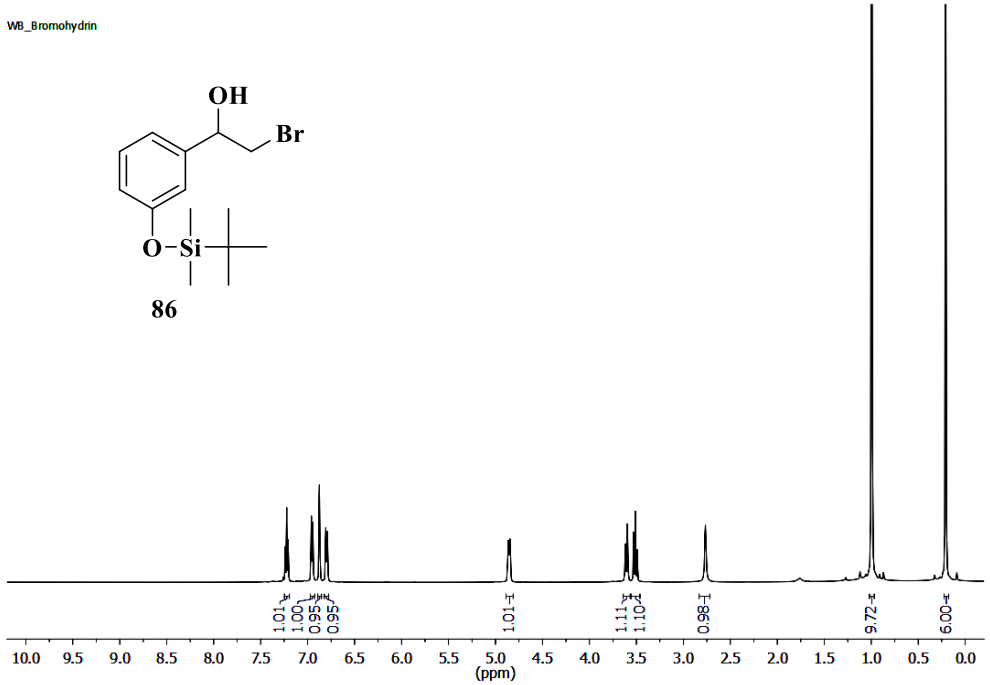
Monoisotopic Mass, Odd and Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

TS_S166_250_SULFADIMETHOXINE 296 (5.493) AM (Cen,4, 80.00, Ar,10000.0,311.08,0.00); Cm (286:301)
TOF MS ES+ 6.59e3

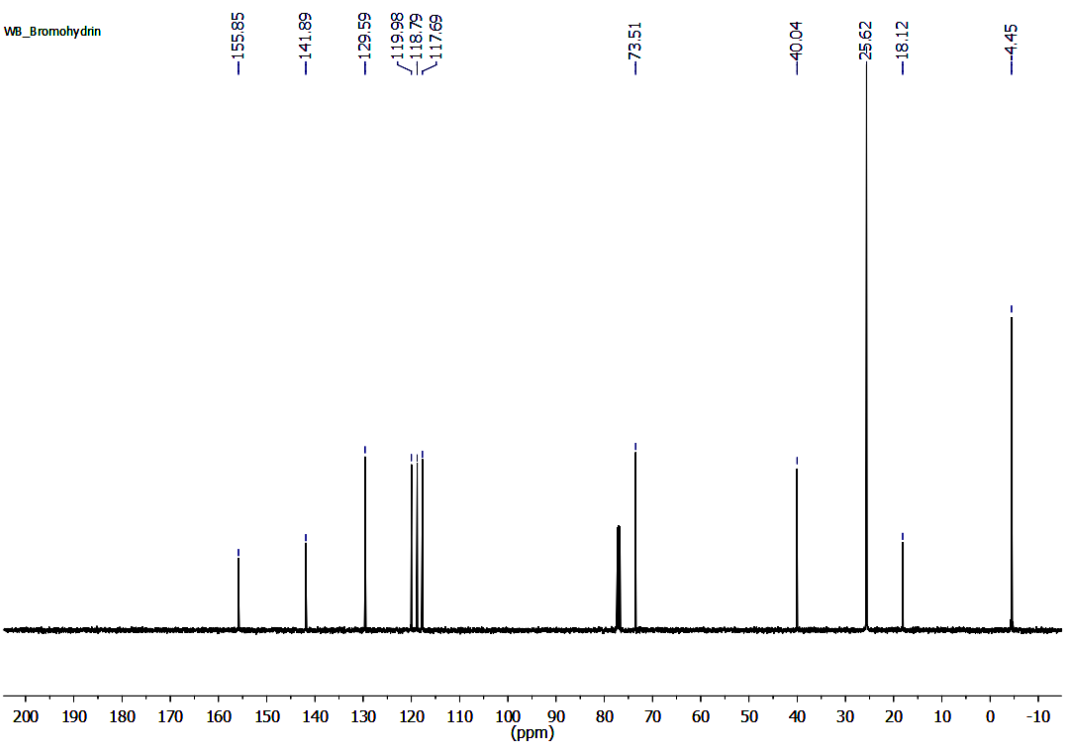


Minimum:				-1.5		
Maximum:		200.0	250.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
251.1465	251.1467	-0.2	-0.9	4.5	1	C14 H23 O2 Si

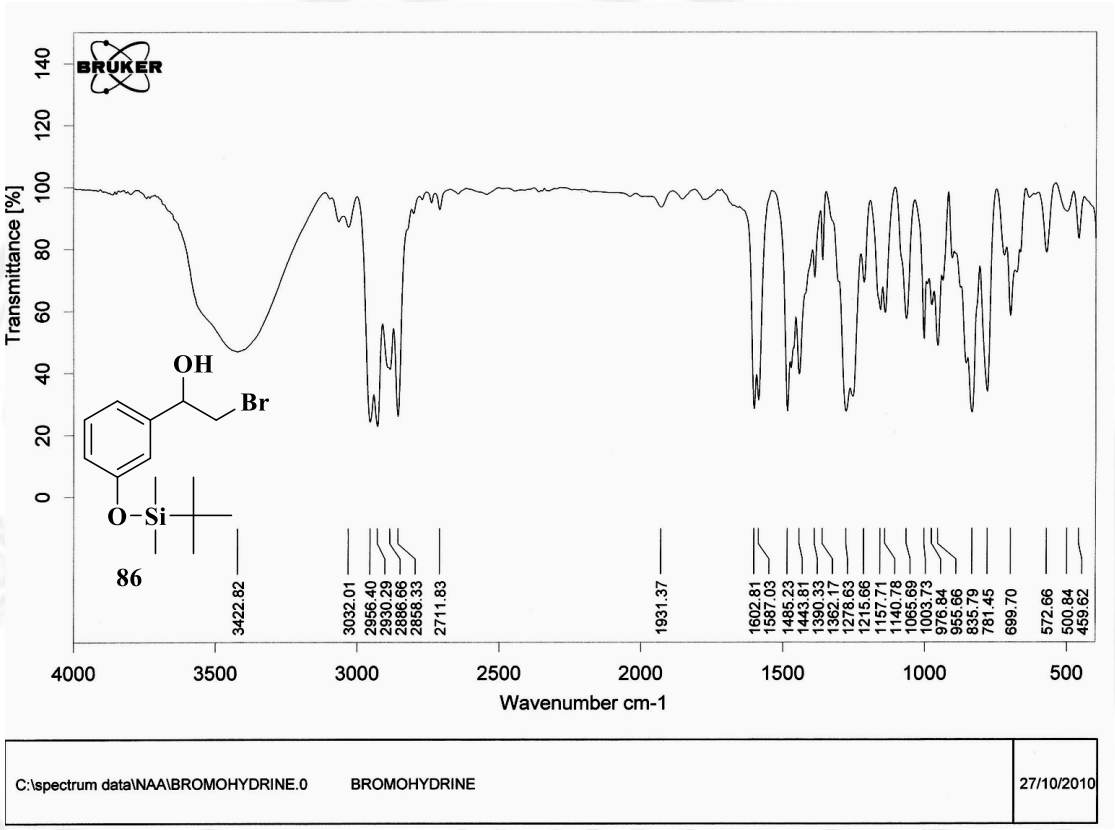
^{1}H-NMR in $CDCl_3$



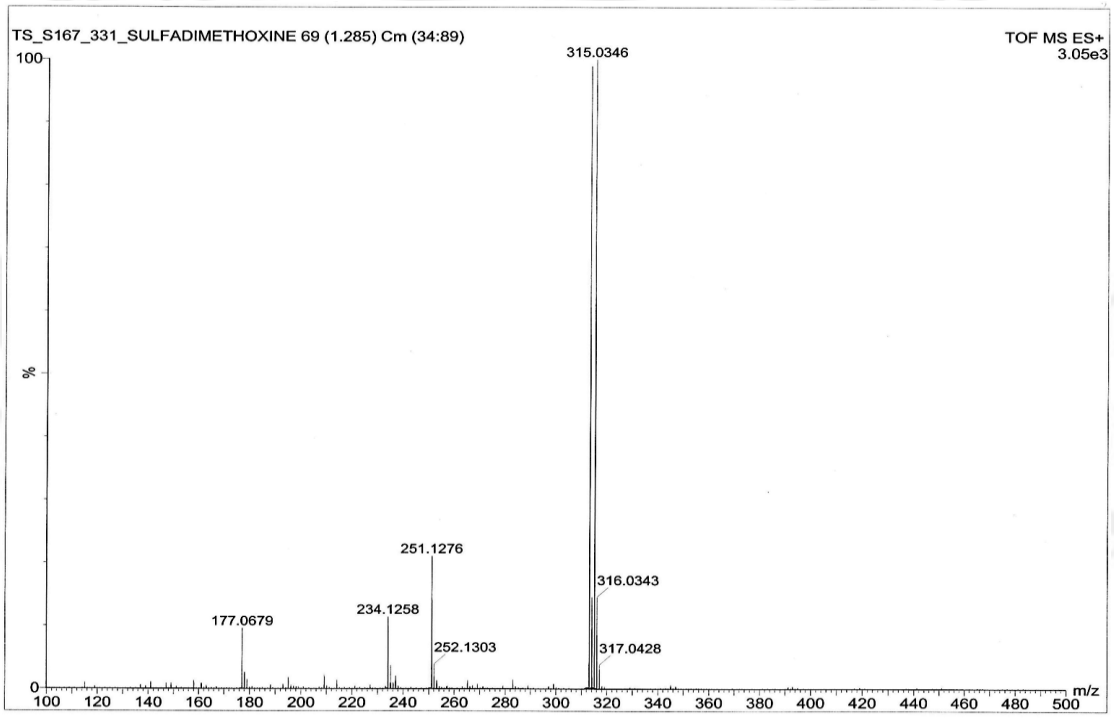
^{13}C-NMR in $CDCl_3$



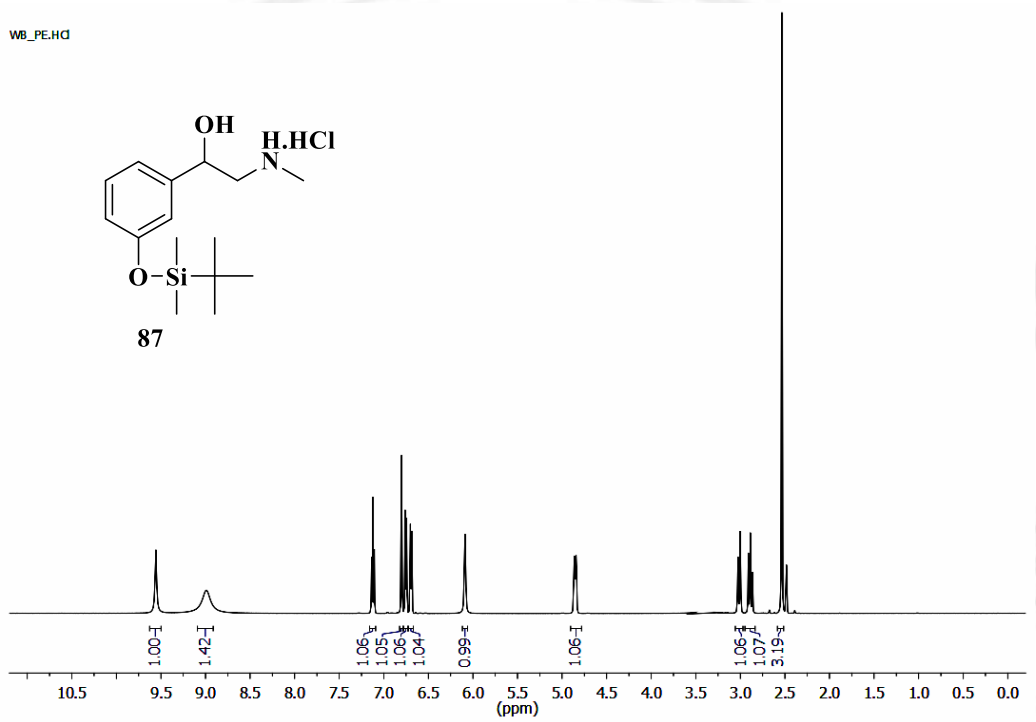
FT-IR (neat)



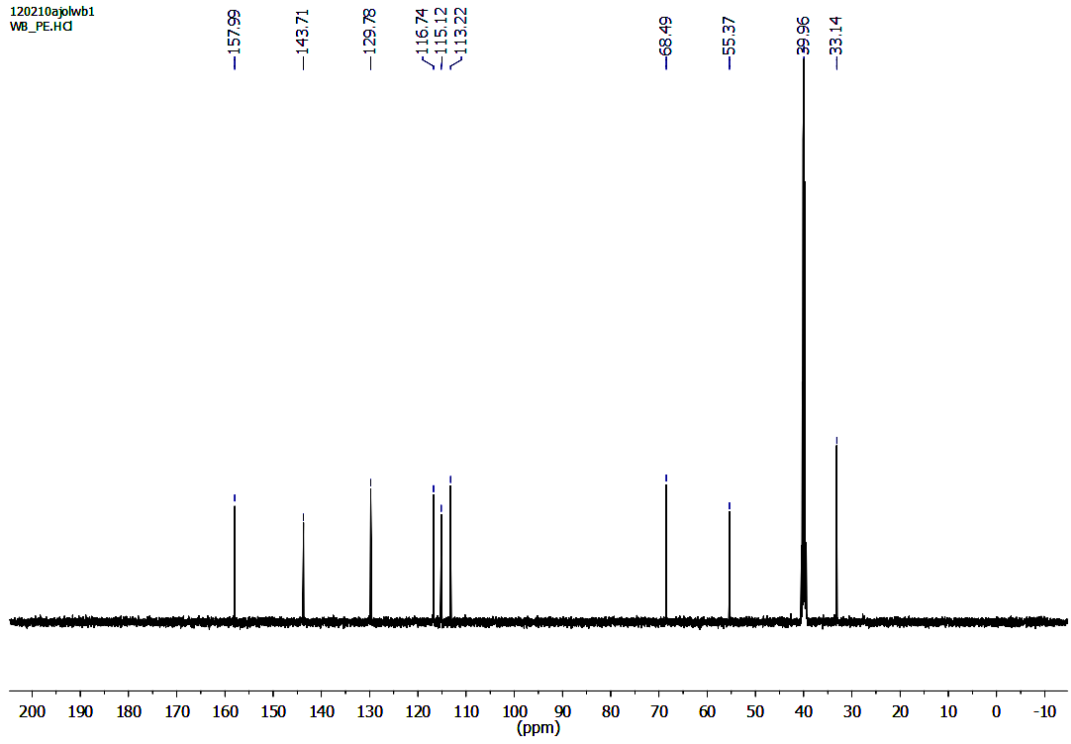
HRMS



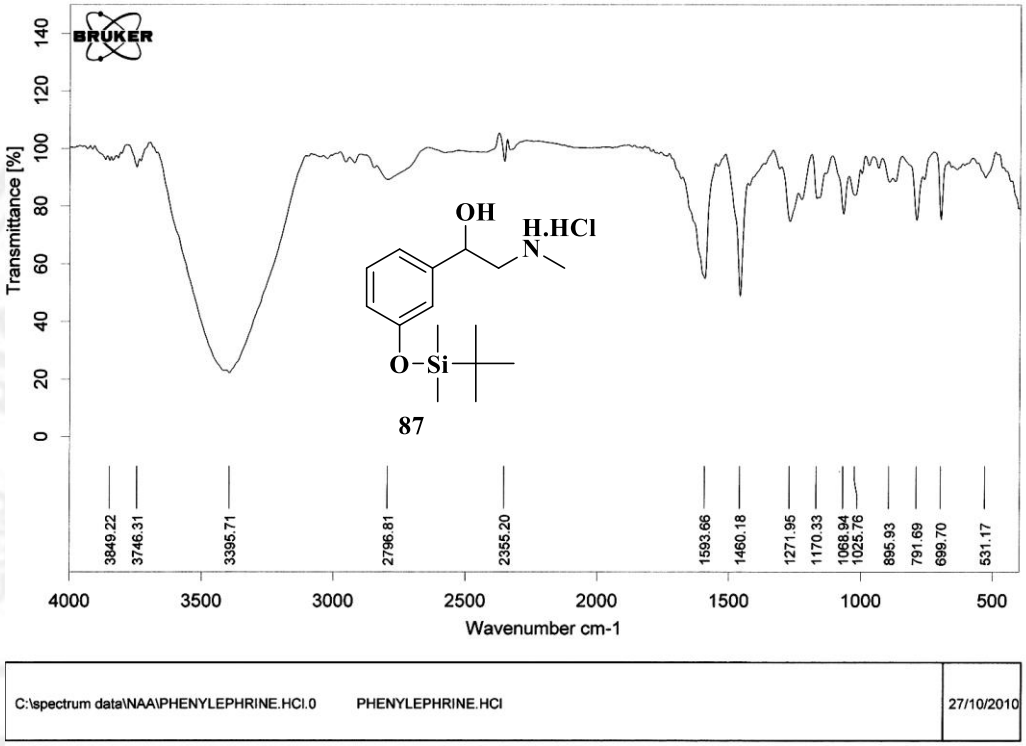
^{1}H-NMR in DMSO-d_6



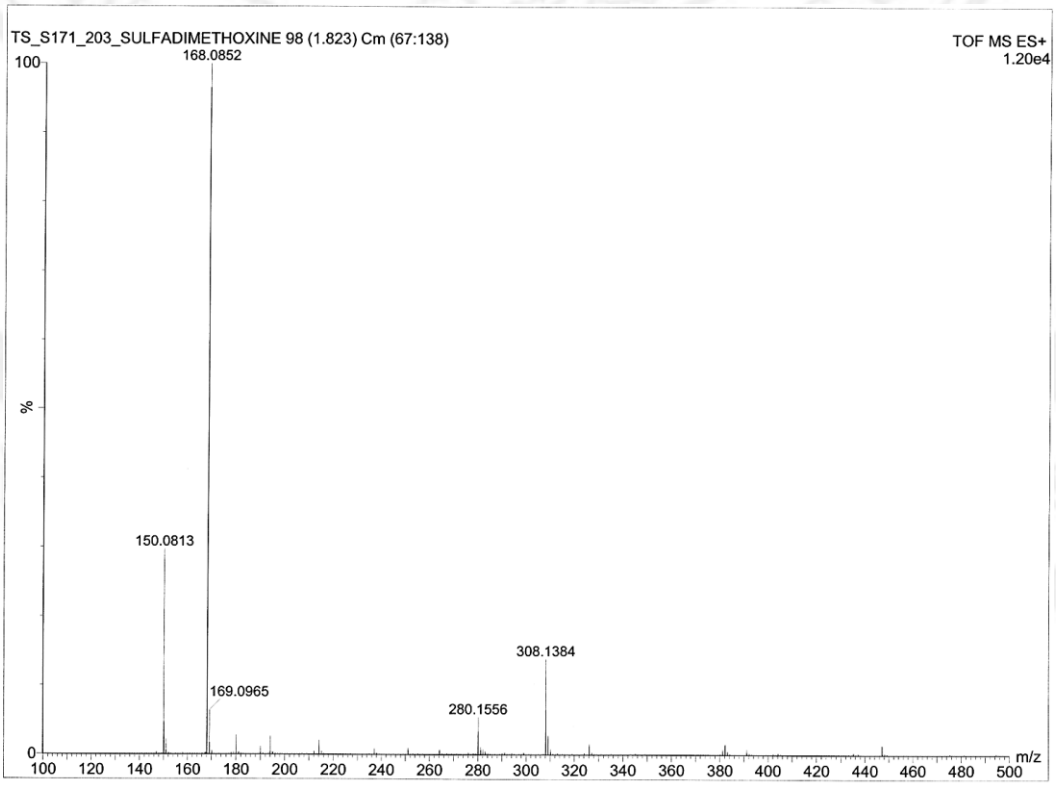
^{13}C-NMR in DMSO-d_6



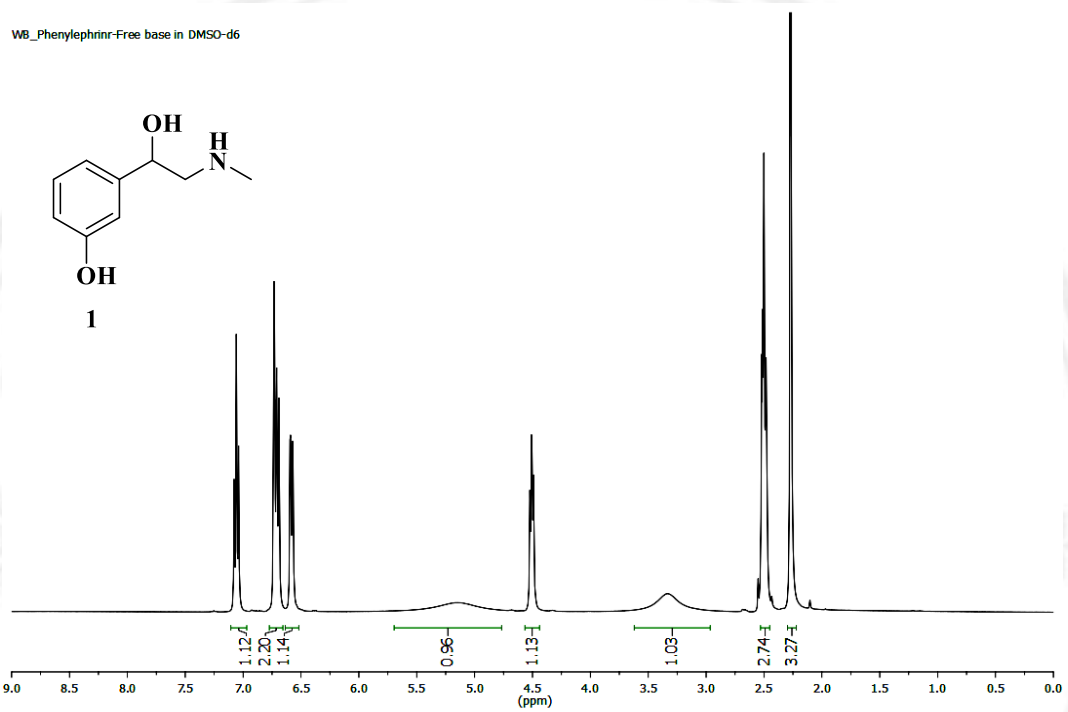
FT-IR (KBr)



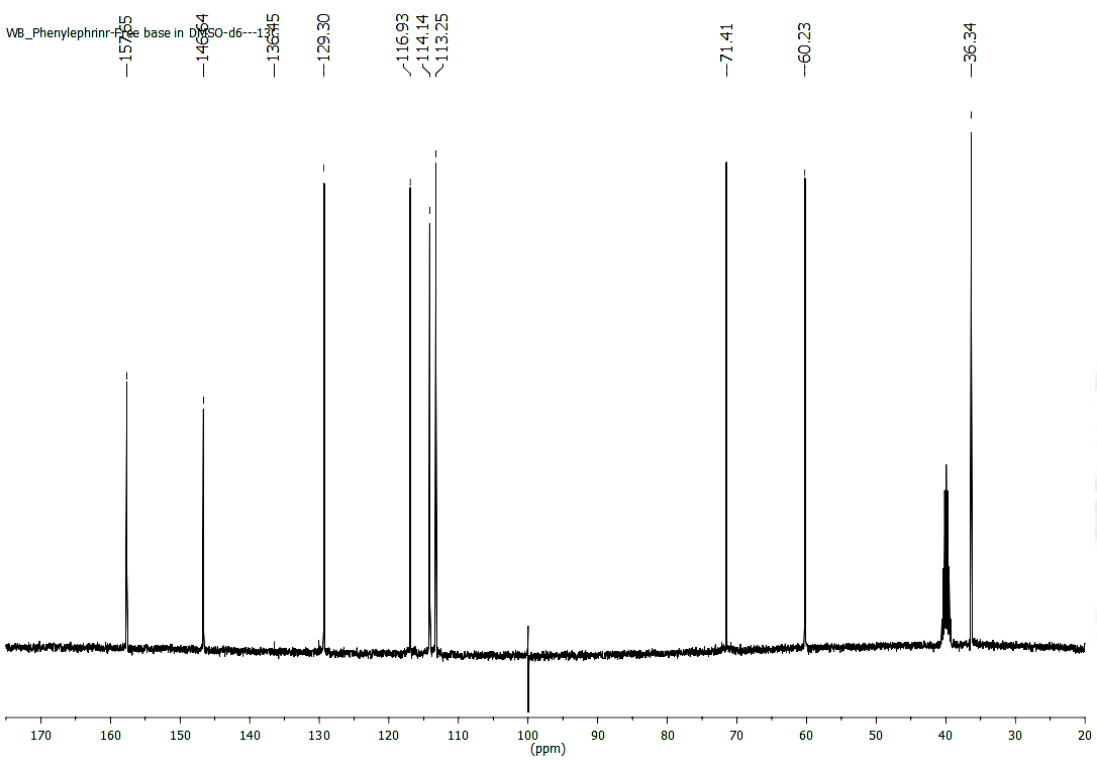
HRMS



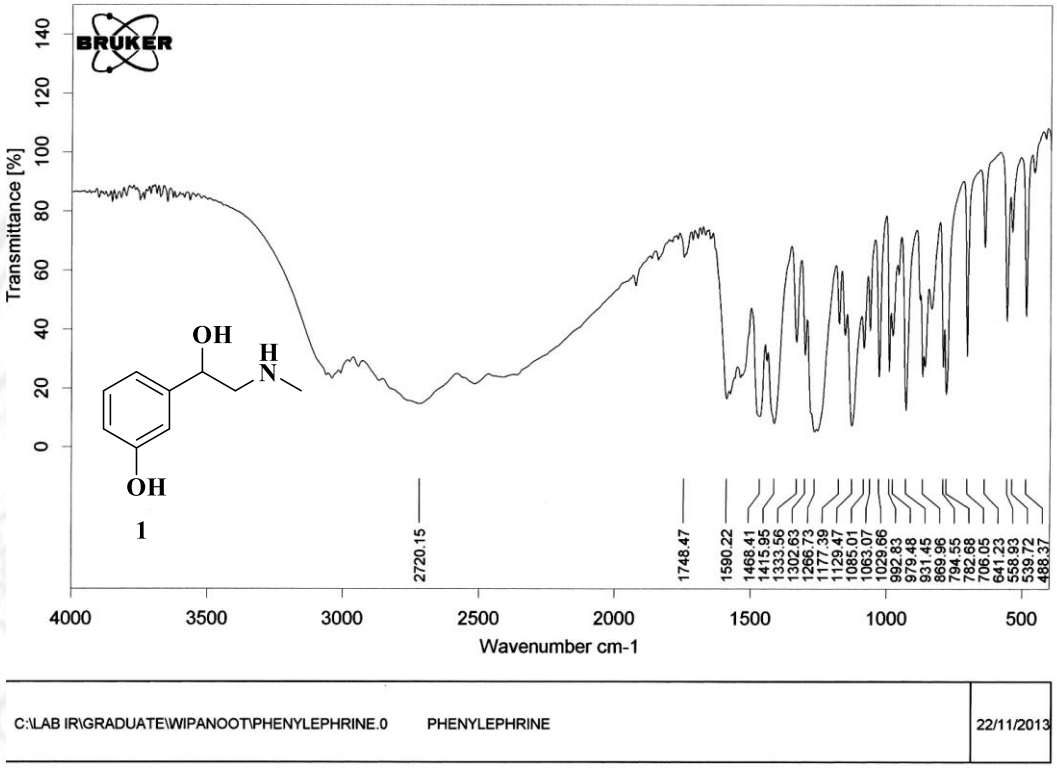
^{1}H-NMR in DMSO-*d*$_6$



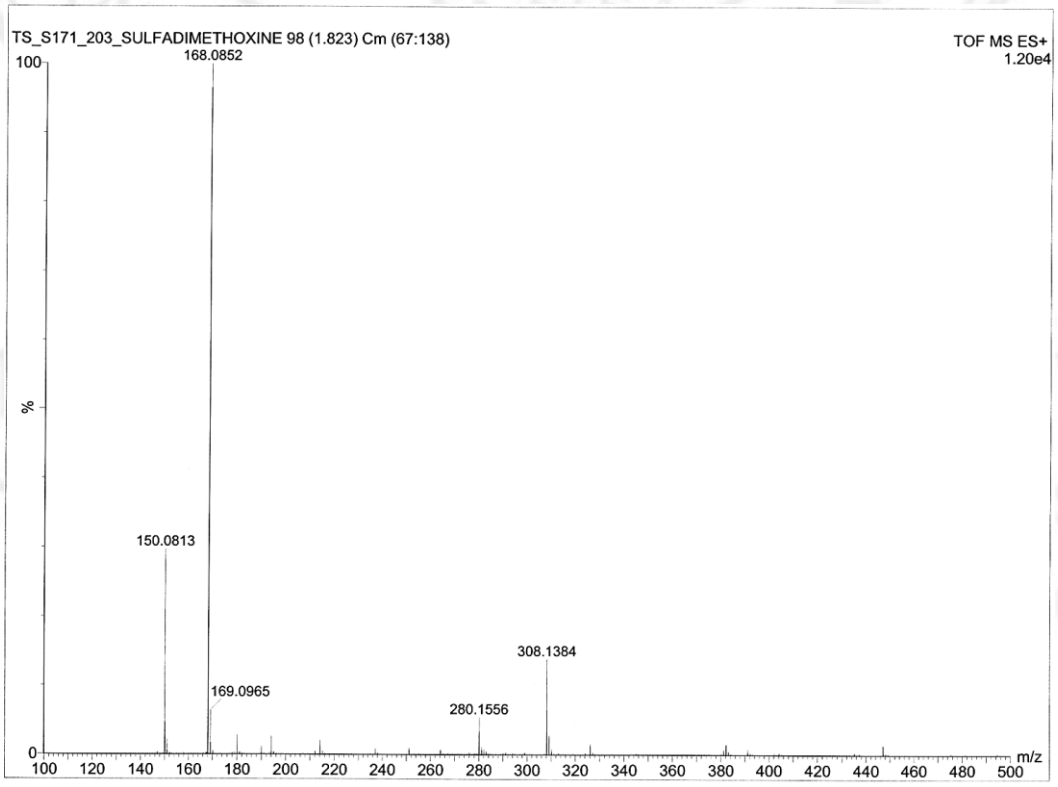
^{13}C-NMR in DMSO-*d*$_6$



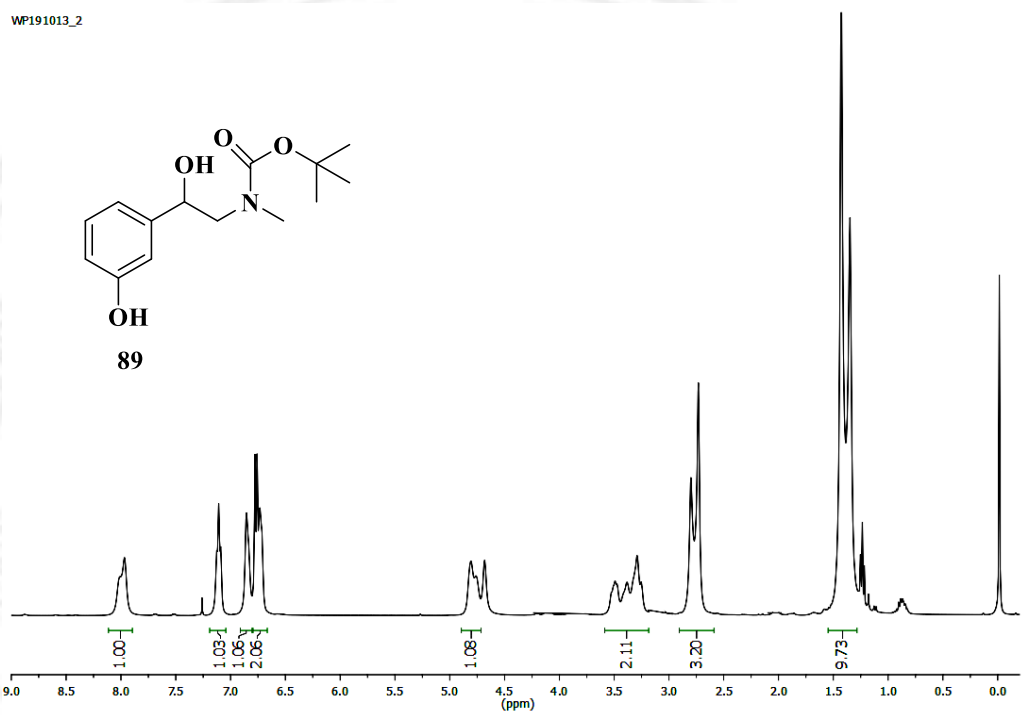
FT-IR (KBr)



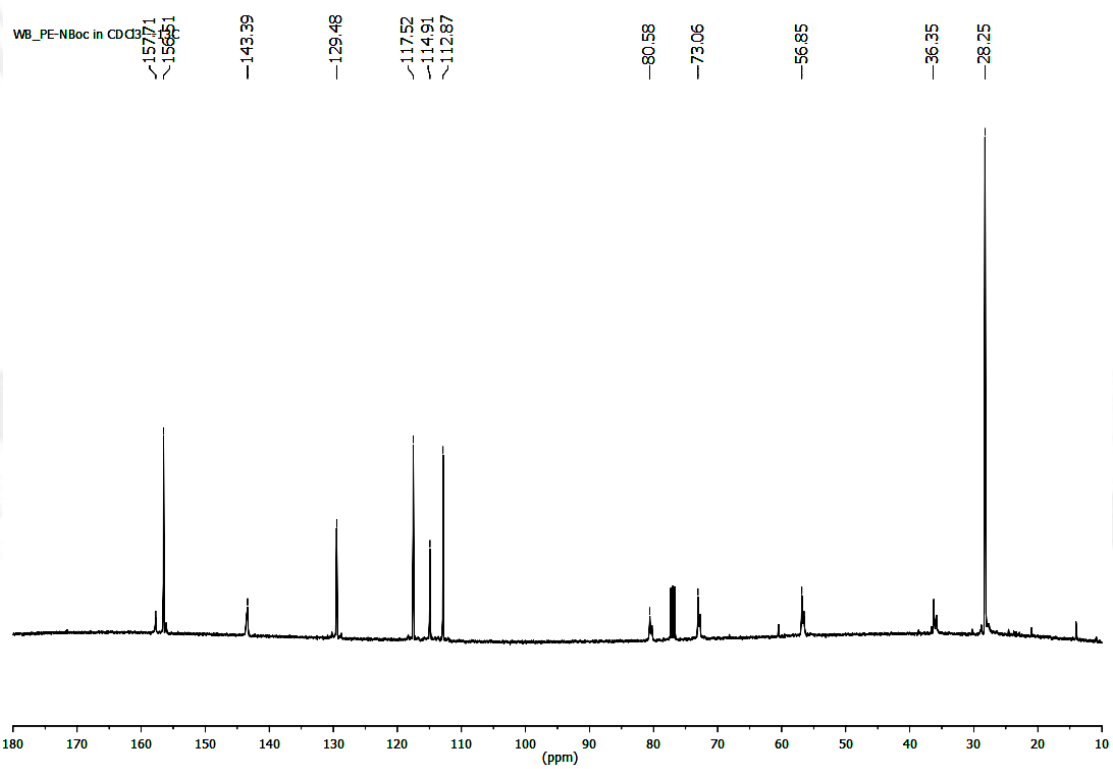
HRMS



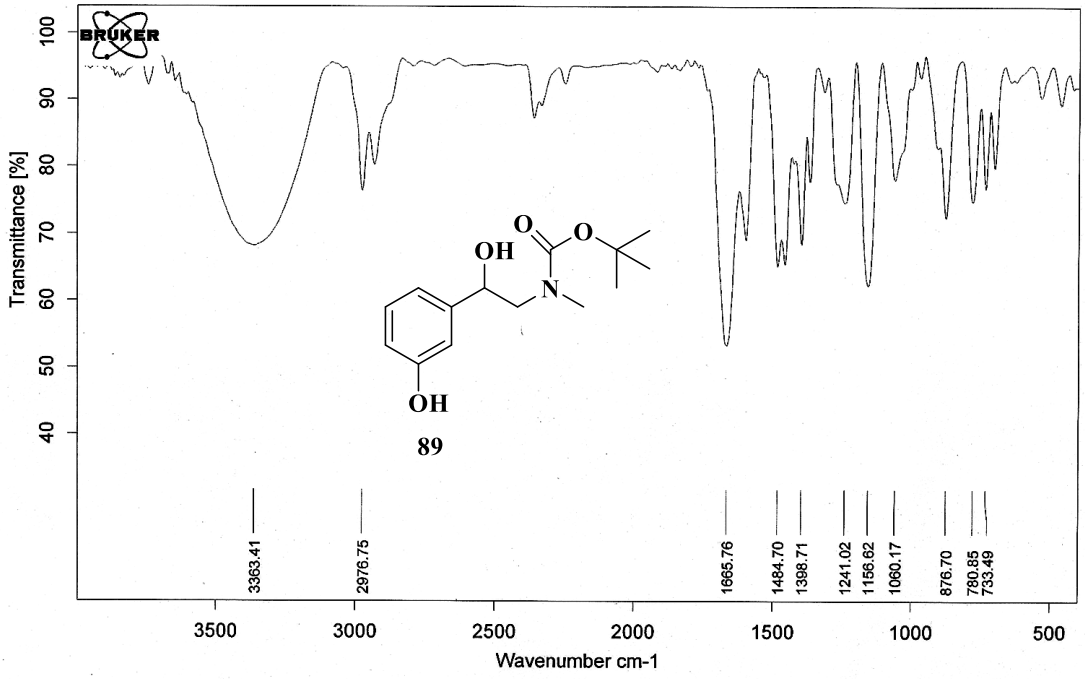
^{1}H-NMR in $CDCl_3$



^{13}C-NMR in $CDCl_3$



FT-IR (neat)



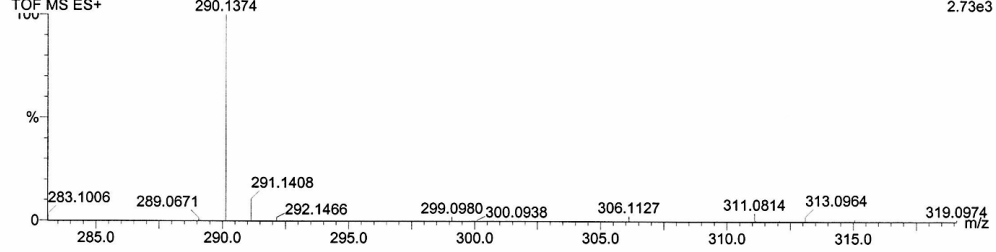
HRMS

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 250.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

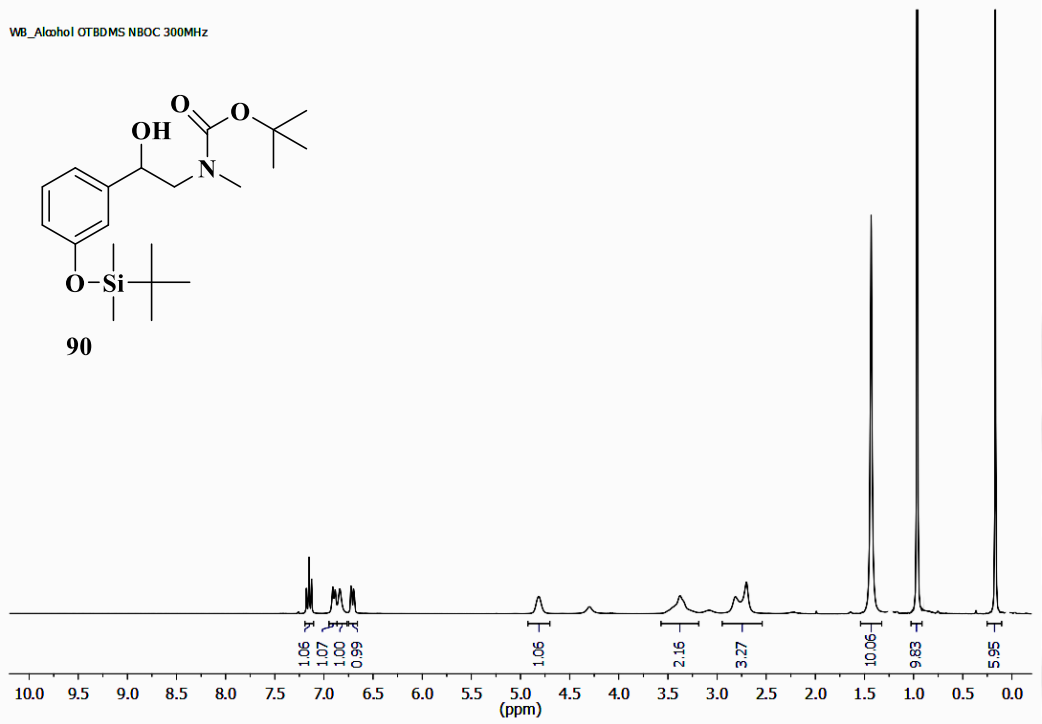
Monoisotopic Mass, Odd and Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

TS_S168_267_SULFADIMETHOXINE 425 (7.884) AM (Cen,4, 80.00, Ar,10000.0,311.08,0.00); Cm (416:426)
TOF MS ES+ 290.1374 2.73e3



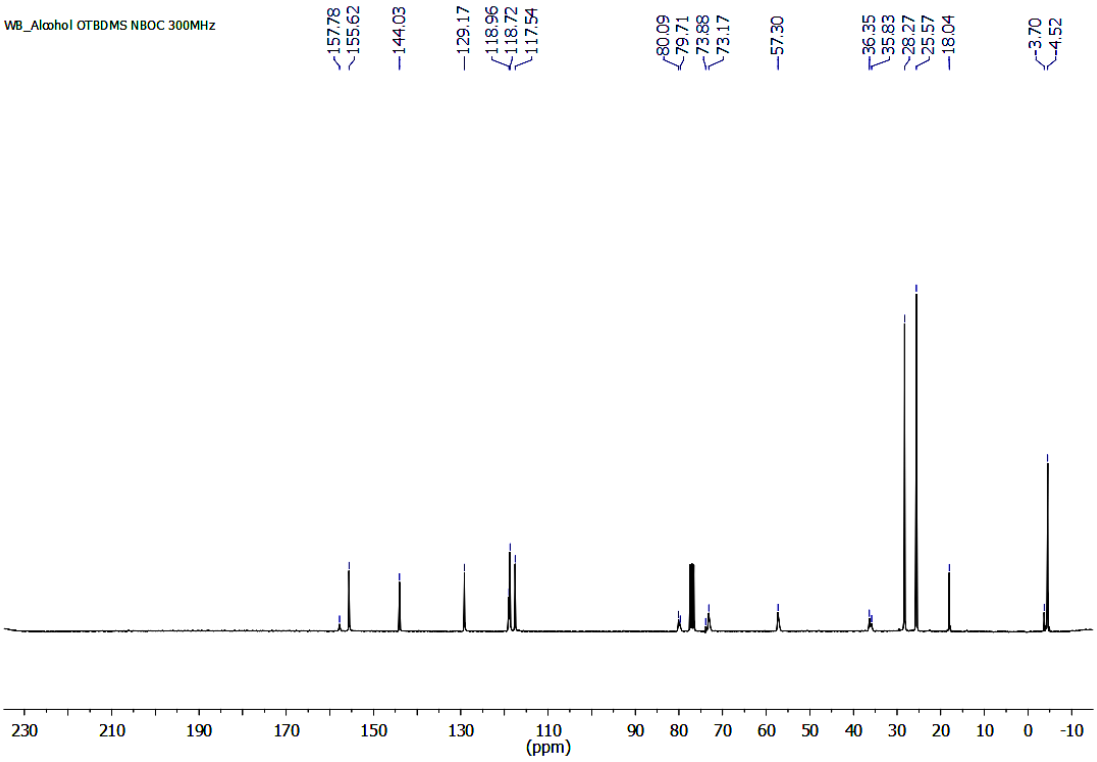
Minimum:				-1.5		
Maximum:		200.0	250.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
290.1374	290.1368	0.6	2.0	4.5	1	C14 H21 N O4 Na

^{1}H-NMR in $CDCl_3$

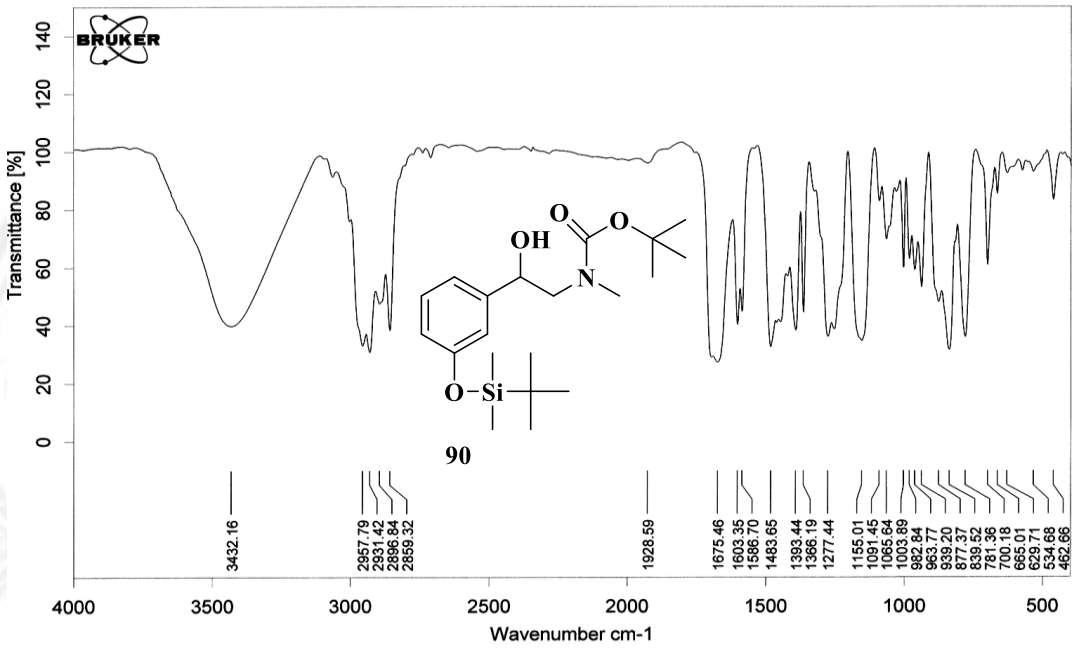


90

^{13}C-NMR in $CDCl_3$



FT-IR (KBr)



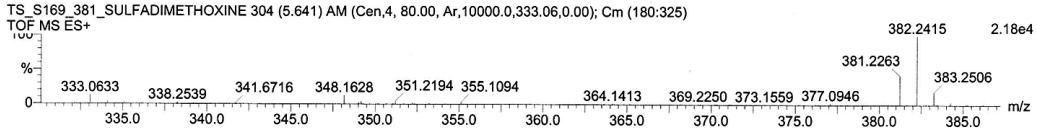
HRMS

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 250.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

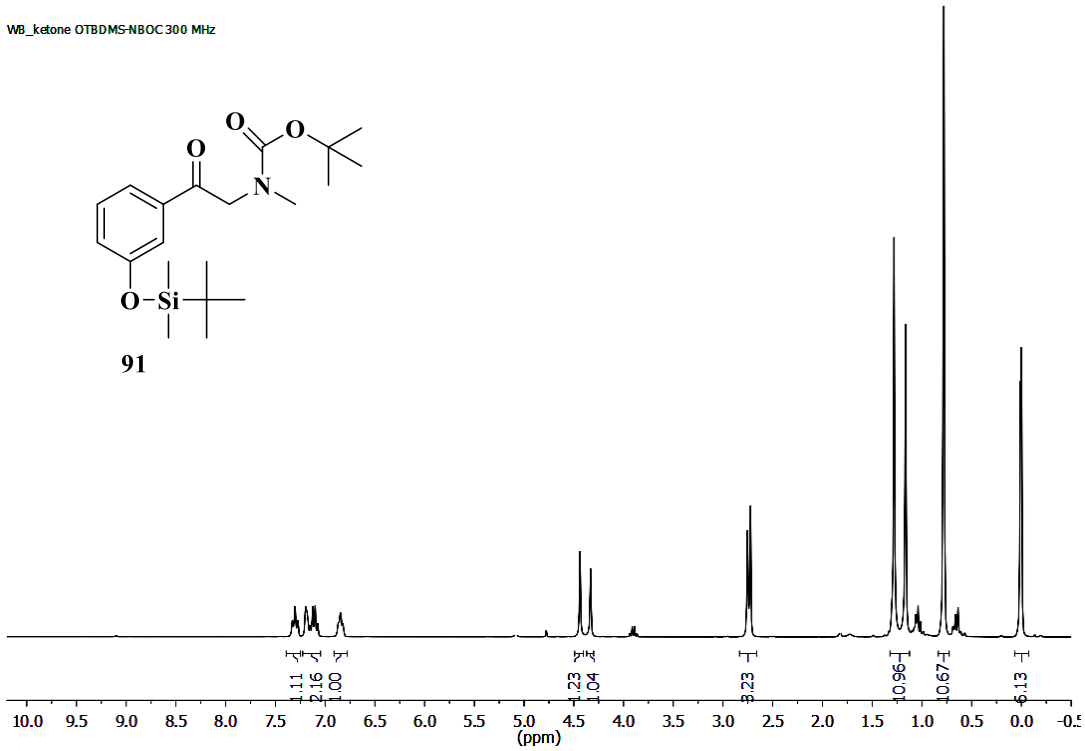
Monoisotopic Mass, Odd and Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

TS_S169_381_SULFADIMETHOXINE 304 (5.641) AM (Cen,4, 80.00, Ar,10000.0,333.06,0.00); Cm (180:325)
TOF MS ES+

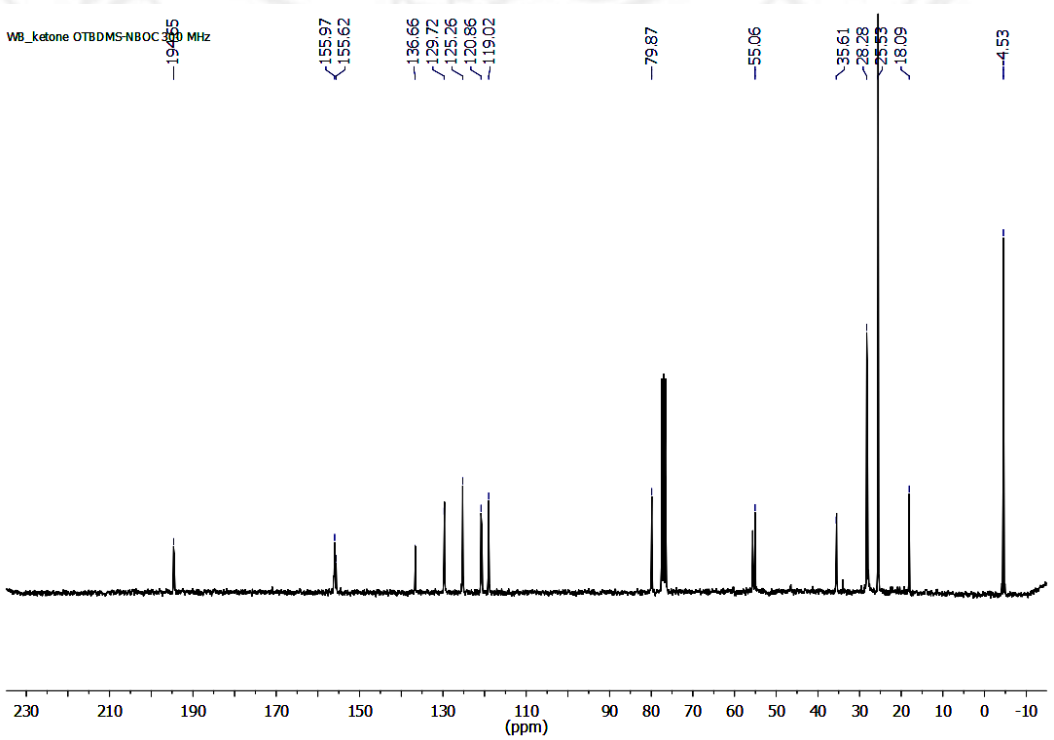


Minimum:				-1.5		
Maximum:		200.0	250.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
382.2415	382.2414	0.1	0.4	4.5	1	C20 H36 N O4 Si

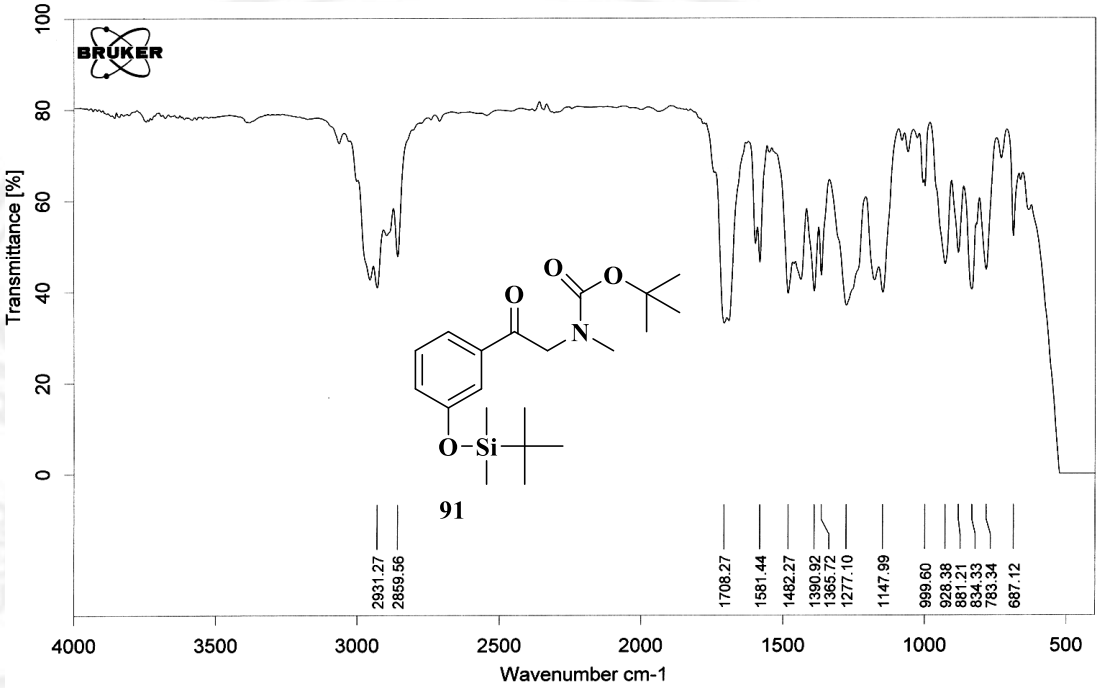
^{1}H-NMR in $CDCl_3$



^{13}C-NMR in $CDCl_3$



FT-IR (neat)



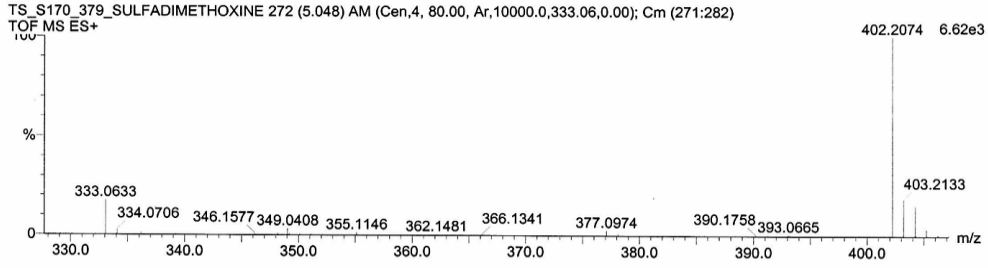
HRMS

Elemental Composition Report **Page 1**

Single Mass Analysis
Tolerance = 250.0 PPM / DBE: min = -1.5, max = 50.0
Isotope cluster parameters: Separation = 1.0 Abundance = 1.0%

Monoisotopic Mass, Odd and Even Electron Ions
3 formula(e) evaluated with 1 results within limits (all results (up to 1000) for each mass)

TS_S170_379_SULFADIMETHOXINE 272 (5.048) AM (Cen,4, 80.00, Ar,10000.0,333.06,0.00); Cm (271:282)
TOF MS ES+



Minimum:				-1.5		
Maximum:		200.0	250.0	50.0		
Mass	Calc. Mass	mDa	PPM	DBE	Score	Formula
402.2074	402.2077	-0.3	-0.6	5.5	1	C20 H33 N O4 Na Si

Chromatogram of chiral HPLC

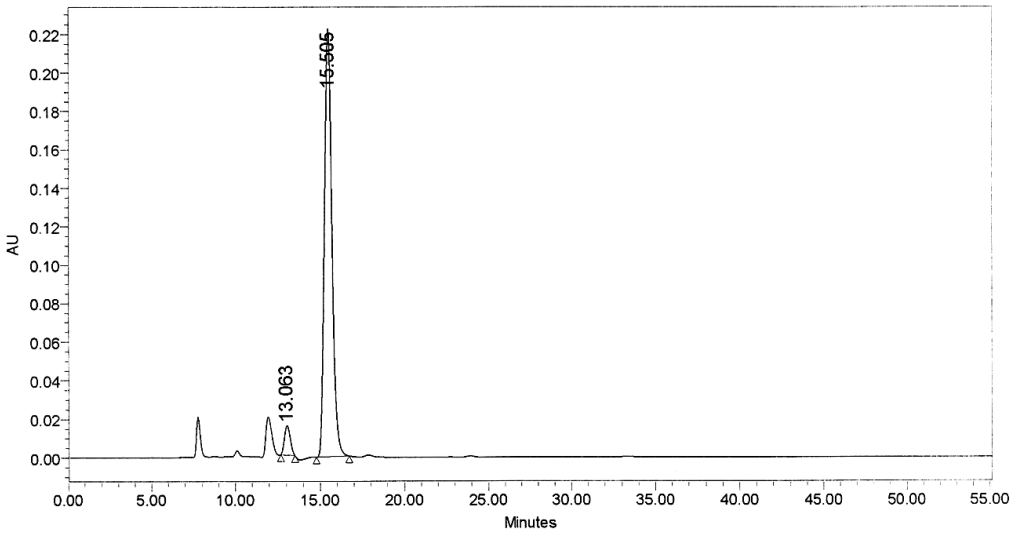
Asymmetric reduction of ketone (91) with borane-THF and *(R)*-2-methyl-CBS-oxazaborolidine (MeCBS) 1.0 eq. at room temperature

Empower

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_R-Mosher 1egMeCBS 86-3	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	10/12/2011 12:21:54 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	3	Date Processed:	10/12/2011 1:32:22 PM EST
Injection Volume:	10.00 ul	Channel Name:	2487Channel 2
Run Time:	60.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	13.063	364669	5.04	15347
2	15.505	6872576	94.96	222209

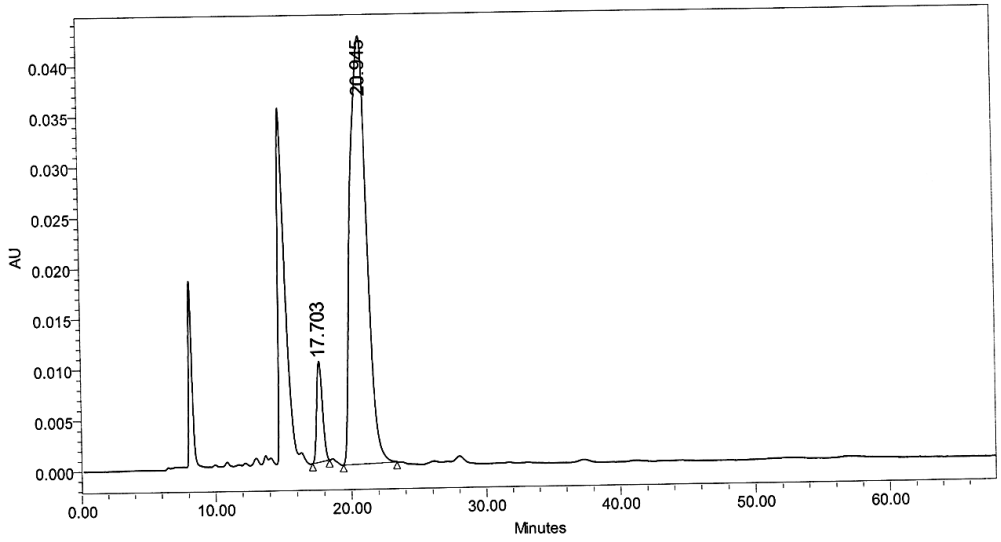
Asymmetric reduction of ketone (91) with borane-THF and *(R)*-2-methyl-CBS-oxazaborolidine (MeCBS) 0.5 eq. at room temperature

Empower software

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_Mosher 0.5eq MeCBS	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	2/11/2011 12:26:51 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	1	Date Processed:	2/11/2011 1:36:19 PM EST
Injection Volume:	5.00 ul	Channel Name:	2487Channel 2
Run Time:	120.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	17.703	292940	7.67	9913
2	20.945	3527271	92.33	42277

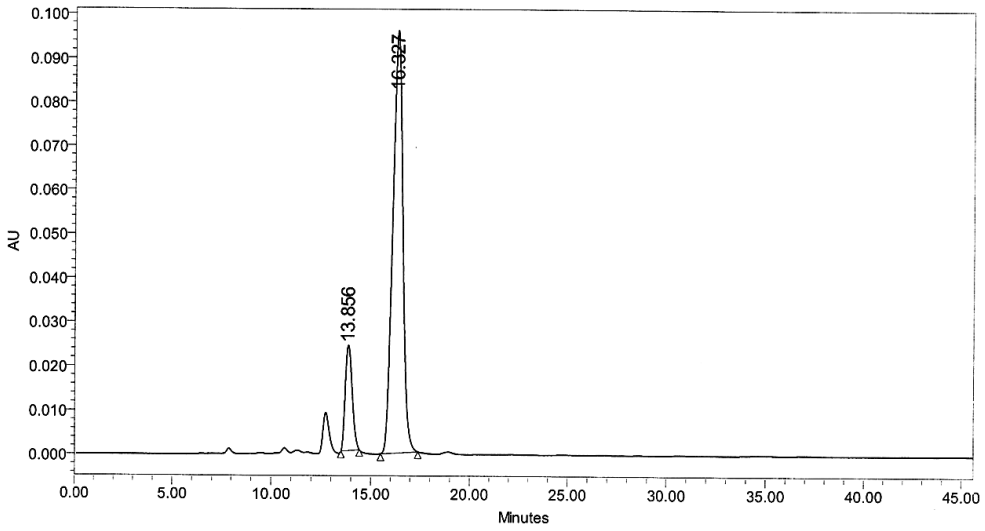
Asymmetric reduction of ketone (91) with borane-THF and *(R)*-2-methyl-CBS-oxazaborolidine (MeCBS) 0.25 eq. at room temperature

Empower software

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_R-Mosher-0.25 eq MeCBS	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	11/11/2011 1:00:45 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	7	Date Processed:	11/11/2011 1:48:06 PM EST
Injection Volume:	5.00 ul	Channel Name:	2487Channel 2
Run Time:	60.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	13.856	575905	14.57	23843
2	16.327	3376725	85.43	95975

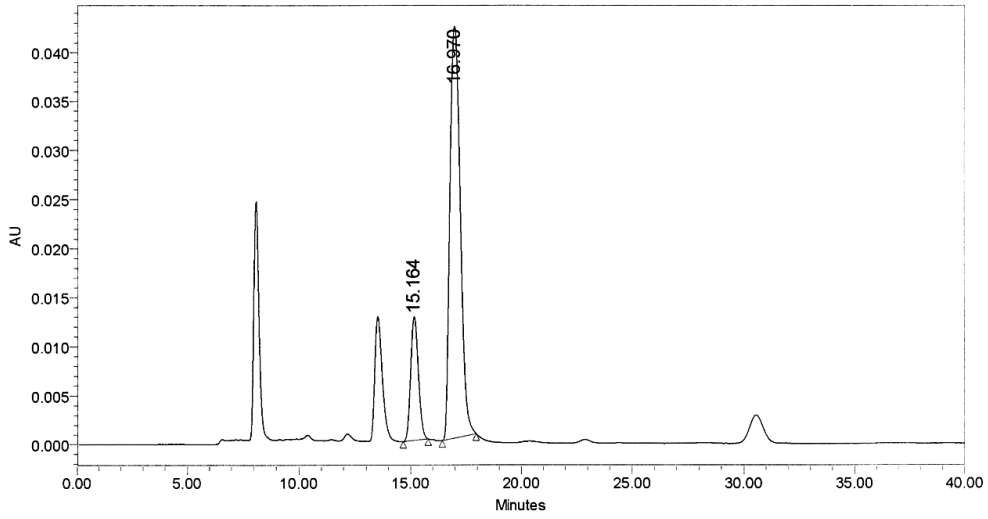
Asymmetric reduction of ketone (91) with borane-THF and *(R)*-2-methyl-CBS-oxazaborolidine (MeCBS) 0.10 eq. at room temperature

Empower SOFTWARE

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_PE-(S)-Mosher ~~61+22~~ 62+21 rt 3	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	9/16/2011 3:31:02 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	6	Date Processed:	9/16/2011 4:12:36 PM EST
Injection Volume:	10.00 ul	Channel Name:	2487Channel 2
Run Time:	45.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	15.164	304050	18.20	12644
2	16.970	1366586	81.80	42008

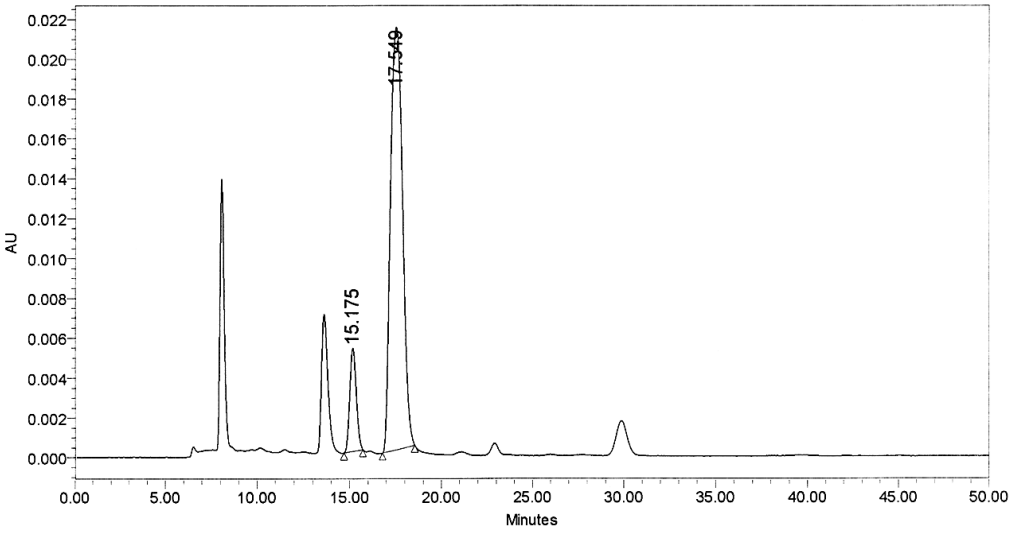
Asymmetric reduction of ketone (91) with borane-THF and *(R)*-2-methyl-CBS-oxazaborolidine (MeCBS) 0.10 eq. at 0 ºC

Empower

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_PE-(S)-Mosher 60+21 61+20	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	9/16/2011 12:05:39 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	2	Date Processed:	9/16/2011 12:59:56 PM EST
Injection Volume:	10.00 ul	Channel Name:	2487Channel 2
Run Time:	60.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	15.175	128317	11.73	5184
2	17.549	966012	88.27	21224

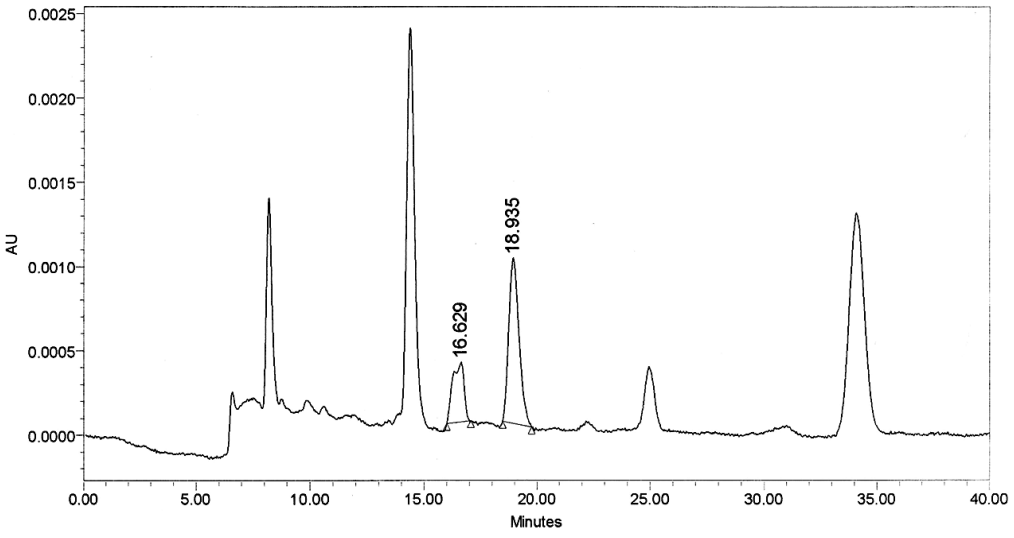
Asymmetric reduction of ketone (91) with borane-THF and *(R)*-2-methyl-CBS-oxazaborolidine (MeCBS) 0.10 eq. at -20 ºC

Empower

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_PE-(S)-Mosher 57+17	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	9/16/2011 4:12:07 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	7	Date Processed:	9/16/2011 4:54:18 PM EST
Injection Volume:	10.00 ul	Channel Name:	2487Channel 2
Run Time:	40.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	16.629	12331	28.24	353
2	18.935	31335	71.76	985

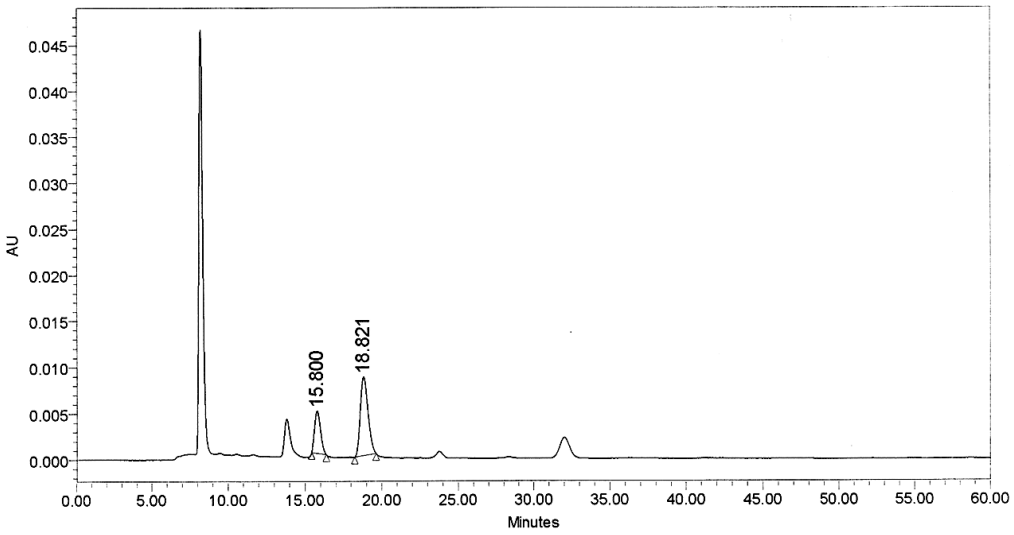
Asymmetric reduction of ketone (91) with borane-THF and *(R)*-2-methyl-CBS-oxazaborolidine (MeCBS) 0.10 eq. at -40 ºC

Empower

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_PE-(S)-Mosher 58+18	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	9/9/2011 11:30:11 AM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	2	Date Processed:	9/9/2011 1:41:00 PM EST
Injection Volume:	10.00 ul	Channel Name:	2487Channel 2
Run Time:	60.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	15.800	123565	29.45	4609
2	18.821	296004	70.55	8520

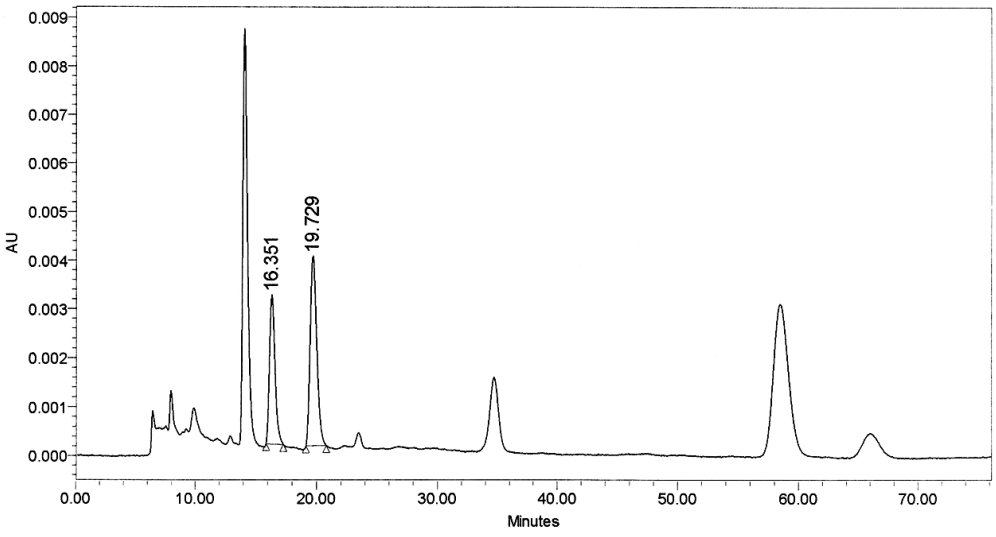
Asymmetric reduction of ketone (91) with borane-THF and *(R)*-2-methyl-CBS-oxazaborolidine (MeCBS) 0.10 eq. at -78 ºC

Empower

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_PE-(S)-Mosher 76 -78oC	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	10/4/2011 1:23:35 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	3	Date Processed:	10/4/2011 3:19:17 PM EST
Injection Volume:	20.00 ul	Channel Name:	2487Channel 2
Run Time:	90.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	16.351	95480	39.31	3056
2	19.729	147400	60.69	3896

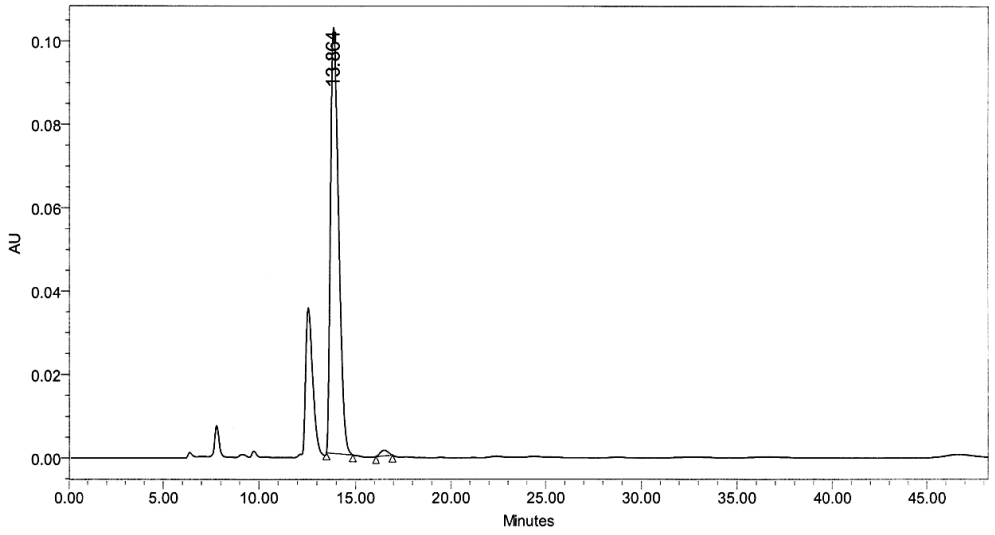
Asymmetric reduction of ketone (91) with *(+)-B*-chlorodiisopinocampheyl borane

Empower software

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_(+) DIP new	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	7/02/2012 1:17:01 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	4	Date Processed:	7/02/2012 3:21:30 PM EST
Injection Volume:	5.00 ul	Channel Name:	2487Channel 2
Run Time:	90.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



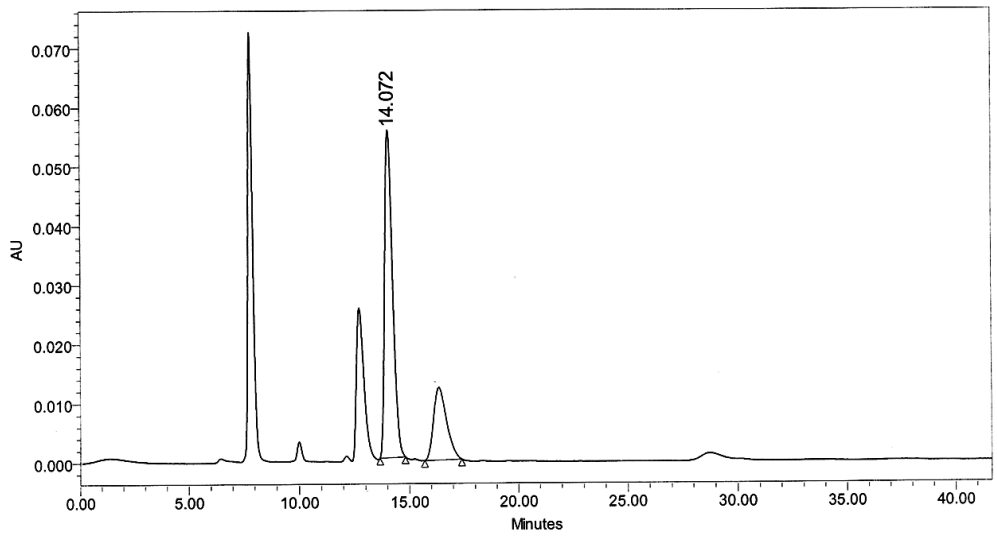
Asymmetric reduction of ketone (91) with *(R)*-1,1-binaphthol and lithium aluminium hydride

Empower software

The University of Sydney, School of Chemistry
Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_LiAlH4+BINOL	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	7/02/2012 3:41:36 PM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	7	Date Processed:	7/02/2012 5:02:50 PM EST
Injection Volume:	10.00 ul	Channel Name:	2487Channel 2
Run Time:	90.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



Asymmetric reduction of ketone (91) with sodium borohydride and L-tartaric acid

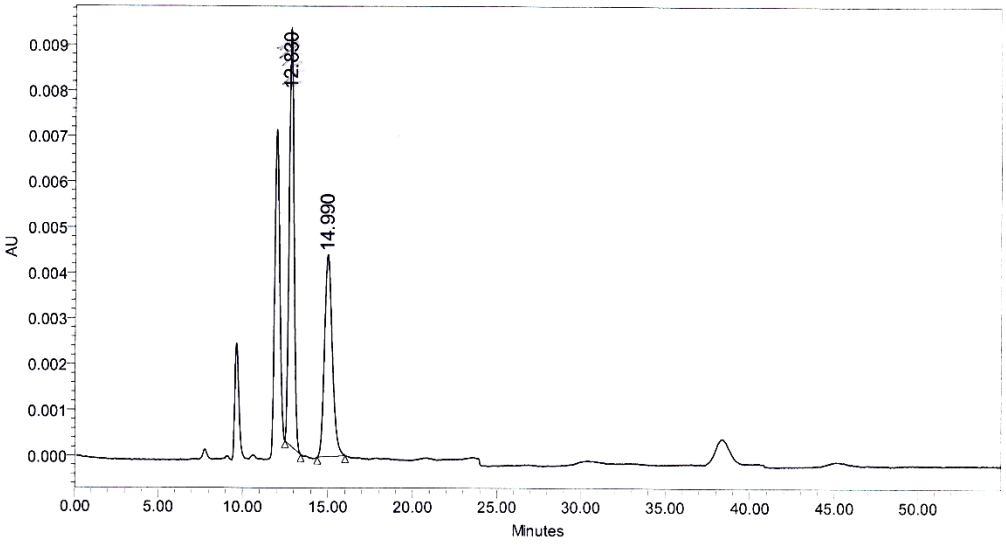
Empower software

The University of Sydney, School of Chemistry

Single Channel Report

SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_L-tartaric+NaBH4	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	13/02/2012 11:05:44 AM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	1	Date Processed:	13/02/2012 12:03:50 PM EST
Injection Volume:	10.00 ul	Channel Name:	2487Channel 2
Run Time:	90.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	12.830	181127	56.25	9154
2	14.990	140886	43.75	4426

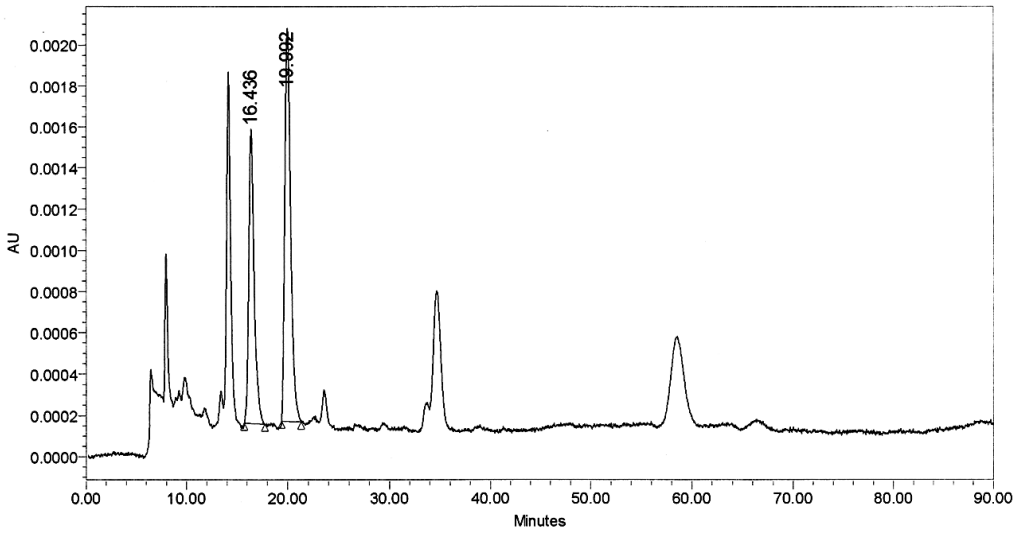
Asymmetric reduction of ketone (91) with sodium borohydride and *β*-cyclodextrins

Empower

The University of Sydney, School of Chemistry
Single Channel Report

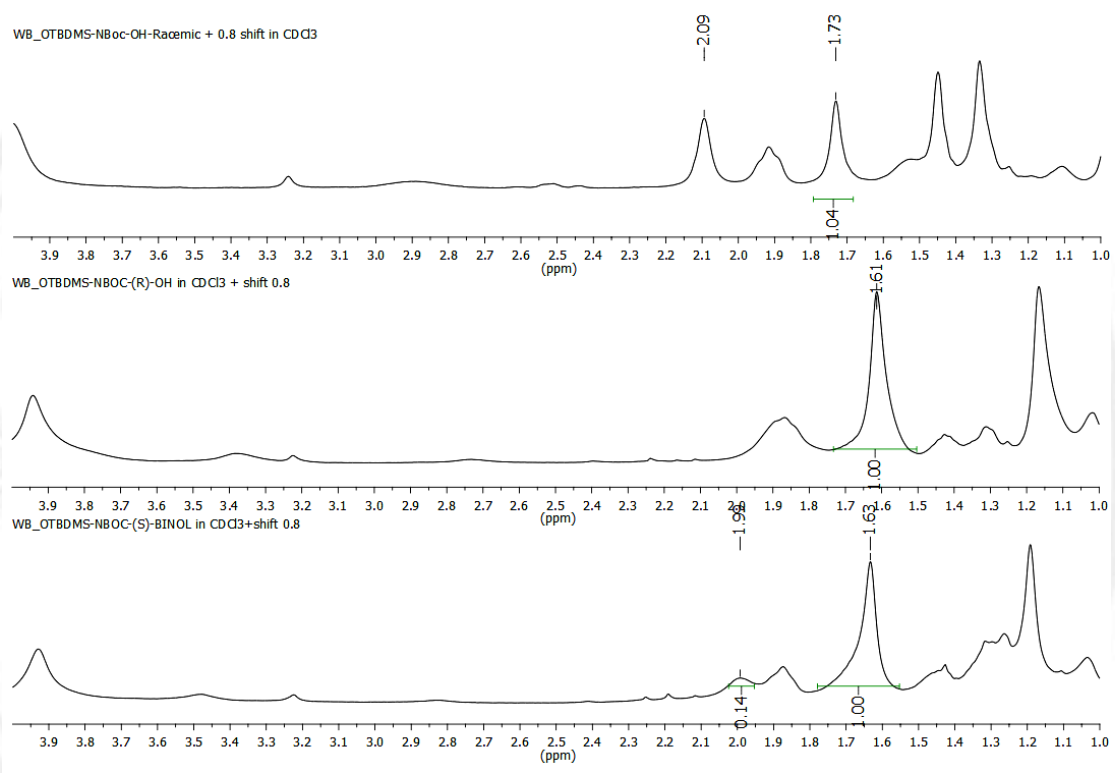
SAMPLE INFORMATION

Project Name:	Jolliffe	Acq. Method Set:	Iso_Anal_JC_UV254_270 05mL
Sample Name:	WB_PE-(S)-Mosher	Sample Set Name:	
Sample Type:	Unknown	Date Acquired:	10/4/2011 11:49:21 AM EST
Vial:	1	Proc. Method:	WB_rac3
Injection #:	2	Date Processed:	10/4/2011 3:18:45 PM EST
Injection Volume:	20.00 ul	Channel Name:	2487Channel 2
Run Time:	90.0 Minutes	Proc. Chnl. Descr.:	270
Chromatogram_Info:			



	RT	Area	% Area	Height
1	16.436	52562	40.74	1430
2	19.992	76469	59.26	1911

Asymmetric reduction of ketone (91) with *(S)*-1,1-binaphthol and lithium aluminium hydride



VITA

Name Miss Wipanoot Baison

Date of Birth January 26, 1982

Academic Status

- B.Sc. (Chemistry), Chiang Mai University, 2004
- M.Sc. (Chemistry), Chiang Mai University, 2007
- Ph.D. (Chemistry), Chiang Mai University, 2013

Experiences

- 2007: Lecturer of Chiang Mai Rajabhat University, Chiang Mai, Thailand
- 2011-2012: Research at The School of Chemistry, University of Sydney, Australia

Scholarships

- Development and Promotion of Science and Technology Talents Project (DPST)

Current Process Chemistry
June 02 - 03, 2011, Philadelphia, PA

A Novel Synthesis of Racemic Phenylephrine Hydrochloride

Wipanoot Baison

Wipanoot Baison[1,2]; Aphiwat Teerawutgulrag[1]; Pakawan Puangsombat[1]; Nuansri Rakariyatham [1]

Chiang Mai University, Department of Chemistry, Faculty of Science, Chiang Mai, 50200 Thailand

[1] Chiang Mai University, Department of Chemistry, Faculty of Science, Chiang Mai, Thailand; [2] University of Sydney, School of Chemistry, Sydney, Australia

Two new synthetic pathways of racemic phenylephrine hydrochloride were explored. m-Hydroxybenzaldehyde was used as a starting material. Wittig reaction yielded the alkene which could go through either epoxidation or bromohydrine formation. The overall yield of the desired product were 71% and 66%, respectively. All reactions were performed at room temperature and short reaction time with good to excellent yields.