Supporting information for:

# Selection and Development of a Route for Cholesterol Absorption Inhibitor AZD4121

*Staffan Karlsson and J. Henrik Sörensen**

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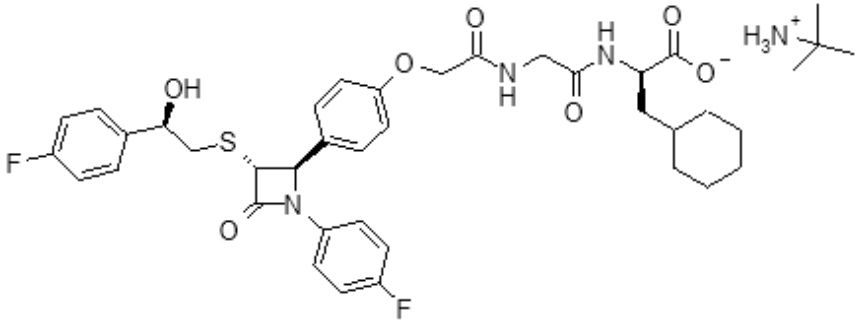
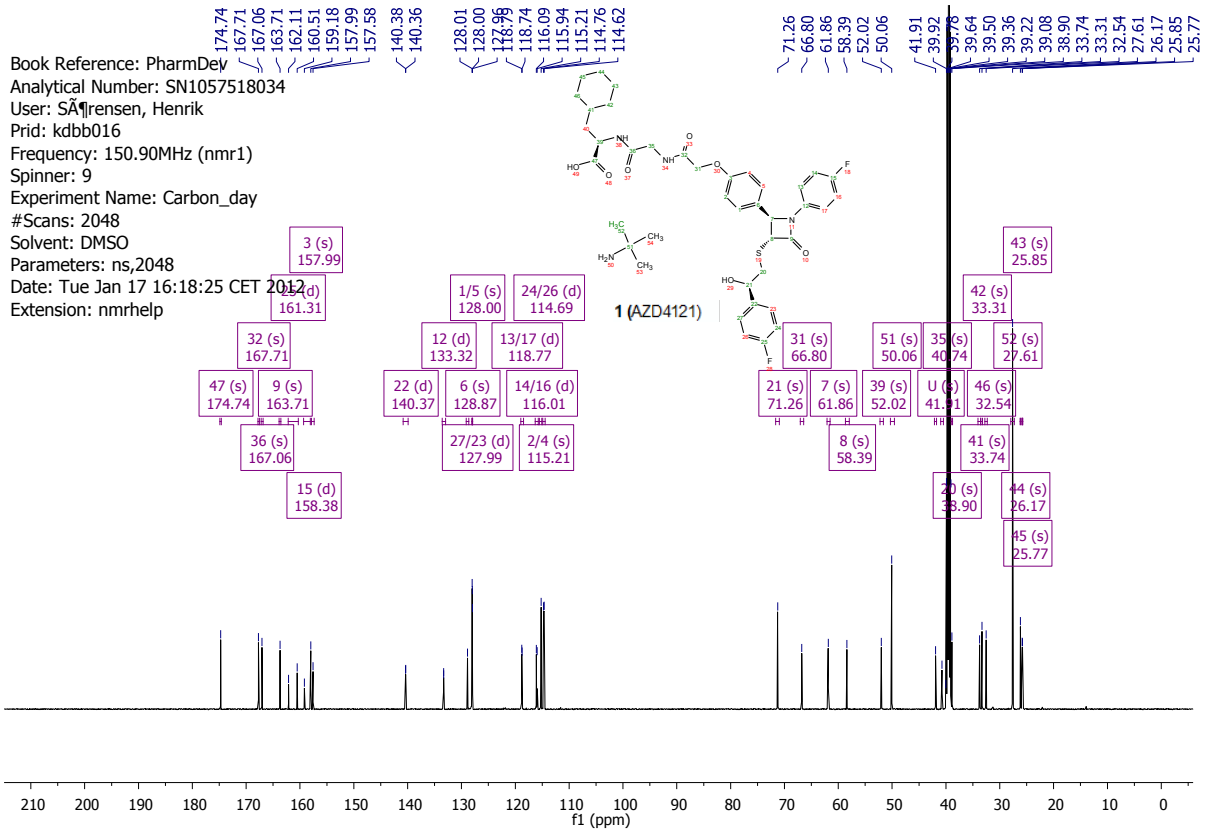
Contents:

Data File Name / / samba-hpc/ nmrdata/ HREG/ h0800_0899/ h0869/ H_086959.016_h1nmr_1.fid/ fid
Owner
Solvent DMSO
Acquisition Date 2005-10-04T11:33:51
Temperature 25.0
Number of Scans 16
Filename
Sample_ID
EN
experiment h1nmr
operator
chemist
Sample# 0
prid
Date Oct 4 2005
solvent DMSO
NMR Frequency 500
probe id500
temperature 25
nt 16
site AstraZeneca R&D Mölndal
Text
Comment H_086959.016_h1nmr_1.fid
Spectrometer inova
Author
Experiment 1D
Pulse Width 0.0000
Acquisition Time 4.0000






| Name | Shift | Class | J's | 2/ 4 (s) | 115.21 | s | |
|---|---|---|---|---|---|---|---|
| 45 (s) | 25.77 | s | | 14/ 16 (d) | 116.01 | d | 22.75 |
| 43 (s) | 25.85 | s | | 13/ 17 (d) | 118.77 | d | 8.09 |
| 44 (s) | 26.17 | s | | 27/ 23 (d) | 127.99 | d | 7.89 |
| 52 (s) | 27.61 | s | | 1/ 5 (s) | 128 | s | |
| 46 (s) | 32.54 | s | | 6 (s) | 128.87 | s | |
| 42 (s) | 33.31 | s | | 12 (d) | 133.32 | d | 2.3 |
| 41 (s) | 33.74 | s | | 22 (d) | 140.37 | d | 2.82 |
| 20 (s) | 38.9 | s | | 3 (s) | 157.99 | s | |
| 35 (s) | 40.74 | s | | 15 (d) | 158.38 | d | 241.19 |
| U (s) | 41.91 | s | | 25 (d) | 161.31 | d | 242.59 |
| 51 (s) | 50.06 | s | | 9 (s) | 163.71 | s | |
| 39 (s) | 52.02 | s | | 36 (s) | 167.06 | s | |
| 8 (s) | 58.39 | s | | 32 (s) | 167.71 | s | |
| 7 (s) | 61.86 | s | | 47 (s) | 174.74 | s | |
| 31 (s) | 66.8 | s | | | | | |
| 21 (s) | 71.26 | s | | | | | |
| 24/ 26 (d) | 114.69 | d | 21.11 | | | | |

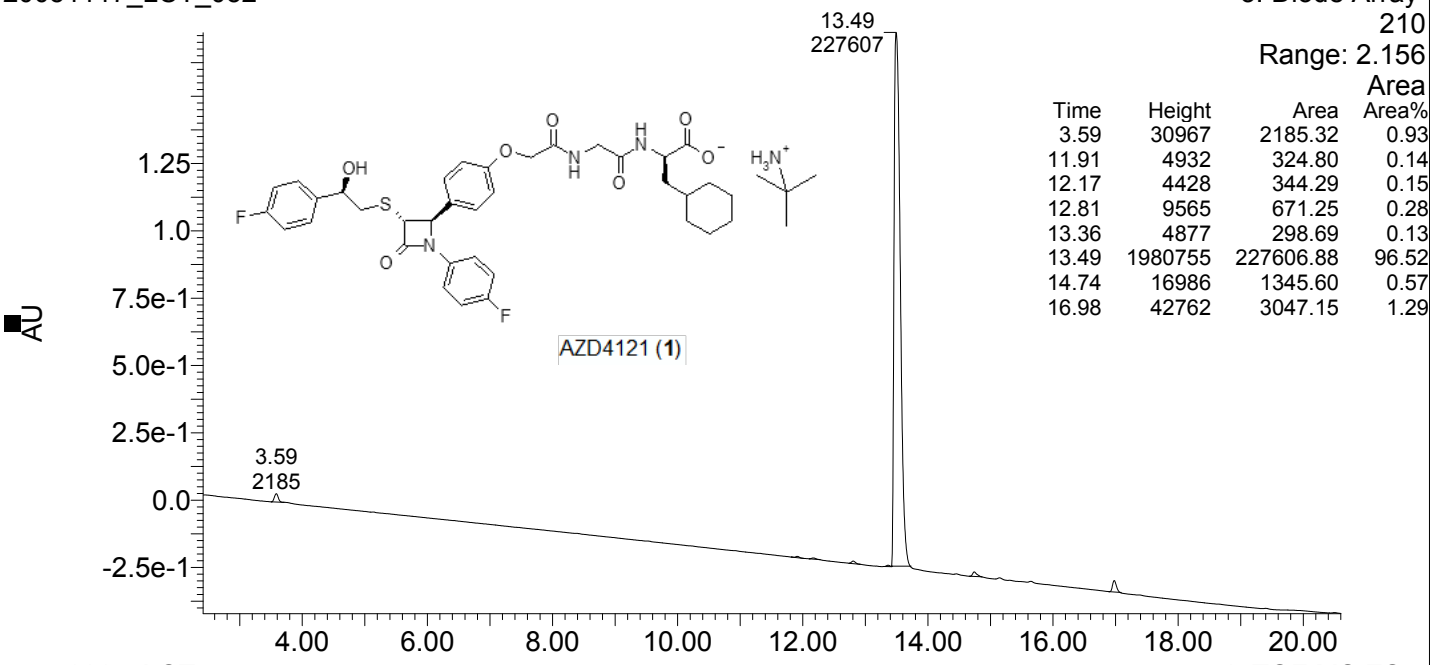
Shifts relative to DMSO-d6 at 39.5 ppm, temp 25deg C. Doublets due to 19F-13C coupling J(Hz)

**Ar-H086959:016** **Ar-H086959:016** **Henrik Sörensen**

20051117_LCT_052

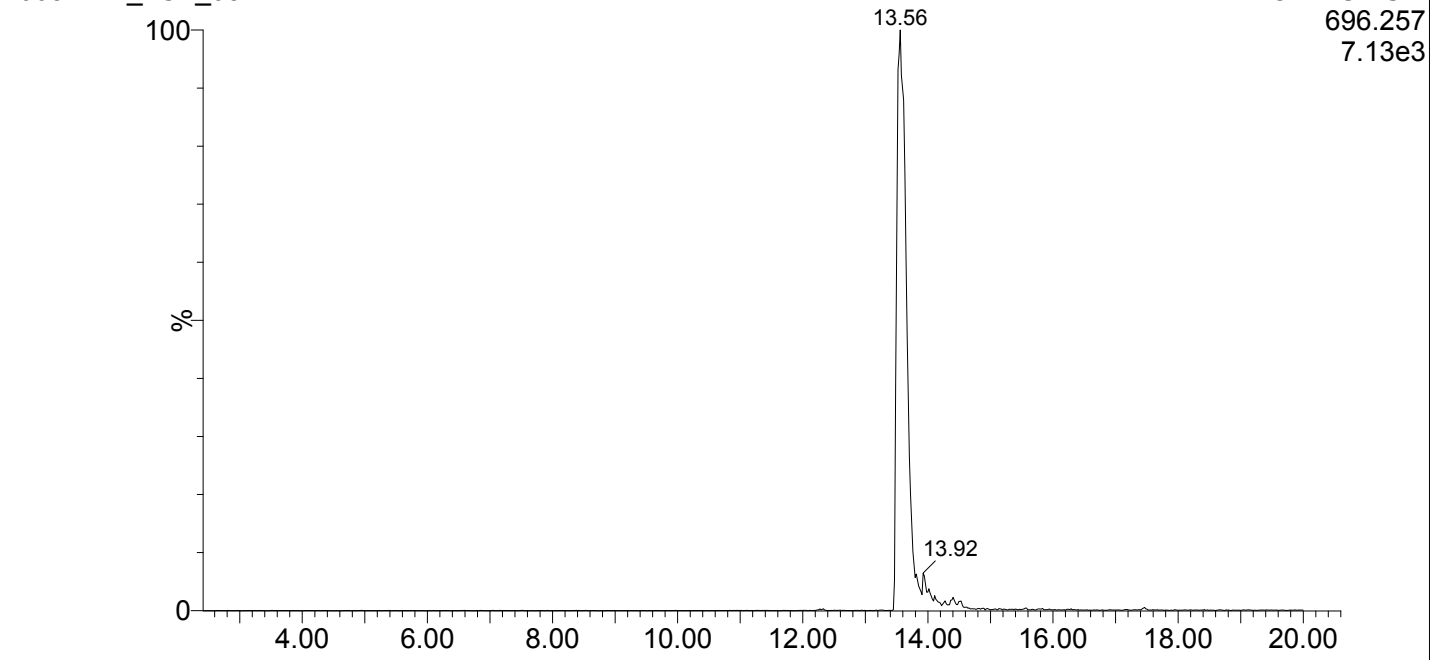
3: Diode Array
210
Range: 2.156
Area

| Time | Height | Area | Area% |
|---|---|---|---|
| 3.59 | 30967 | 2185.32 | 0.93 |
| 11.91 | 4932 | 324.80 | 0.14 |
| 12.17 | 4428 | 344.29 | 0.15 |
| 12.81 | 9565 | 671.25 | 0.28 |
| 13.36 | 4877 | 298.69 | 0.13 |
| 13.49 | 1980755 | 227606.88 | 96.52 |
| 14.74 | 16986 | 1345.60 | 0.57 |
| 16.98 | 42762 | 3047.15 | 1.29 |



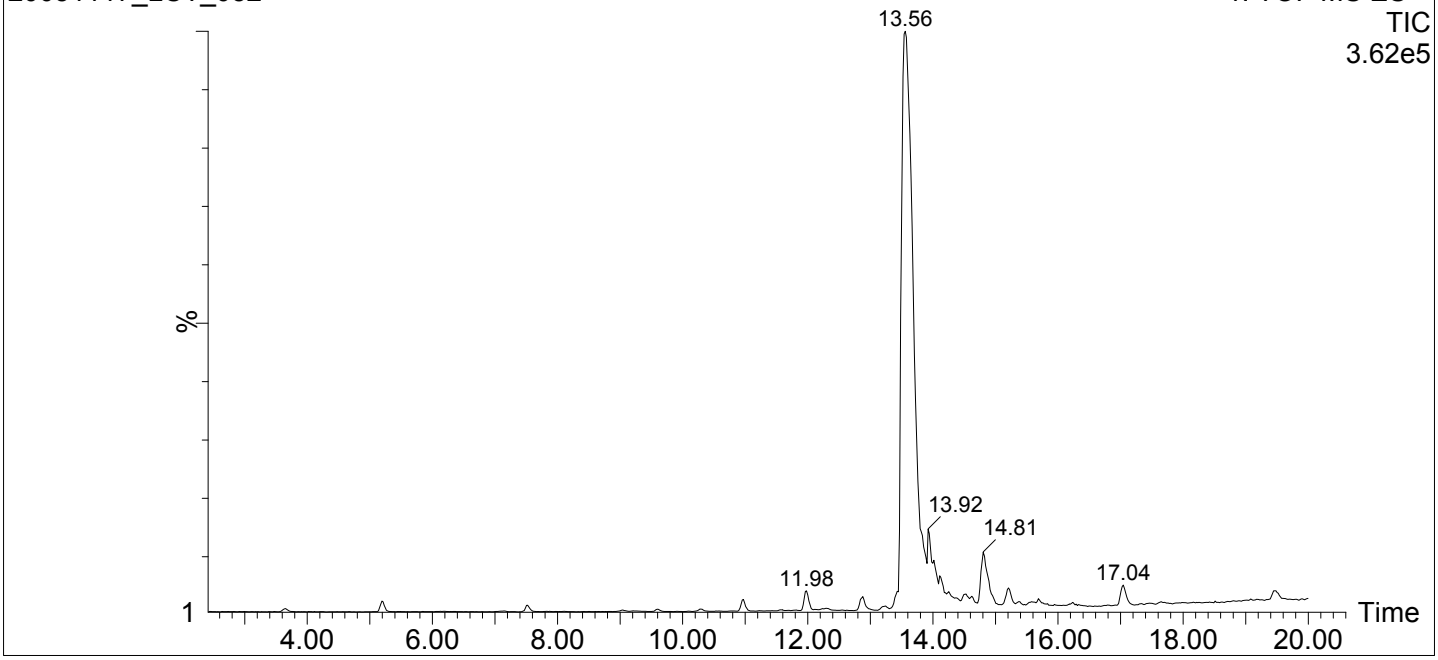

20051117_LCT_052

1: TOF MS ES+
696.257
7.13e3




20051117_LCT_052

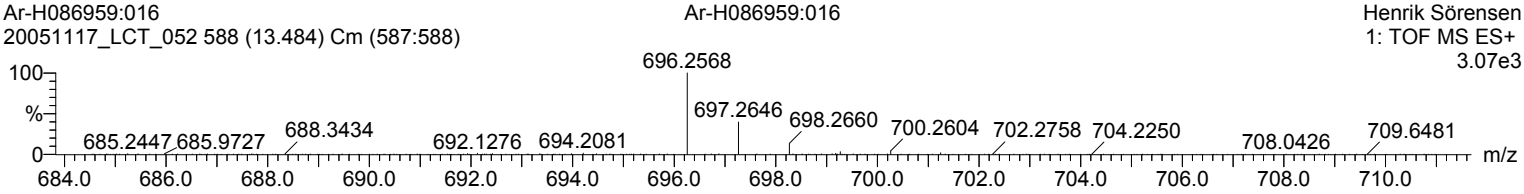
1: TOF MS ES+
TIC
3.62e5



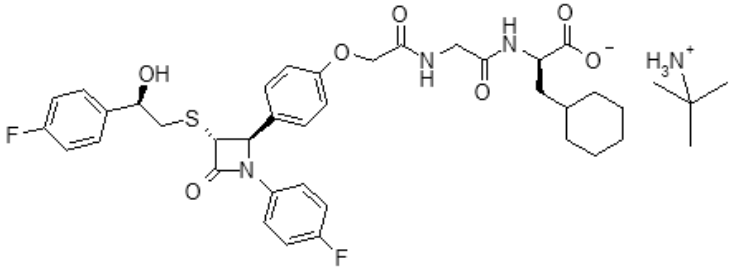
## Single Mass Analysis

Tolerance = 15.0 mDa / DBE: min = -1.5, max = 50.0
Selected filters: None

Monoisotopic Mass, Even Electron Ions
3 formula(e) evaluated with 1 results within limits (up to 50 best isotopic matches for each mass)
Elements Used:
C: 36-40 H: 35-40 N: 3-6 O: 7-7 F: 2-2 23Na: 0-1 S: 1-1

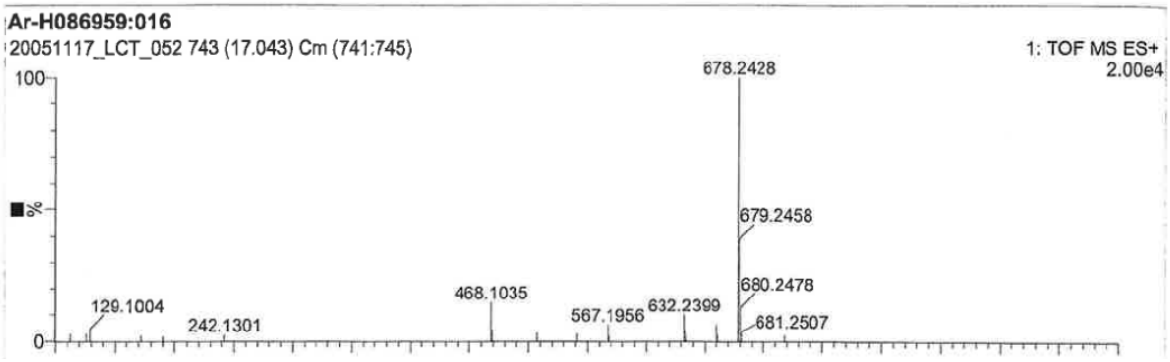


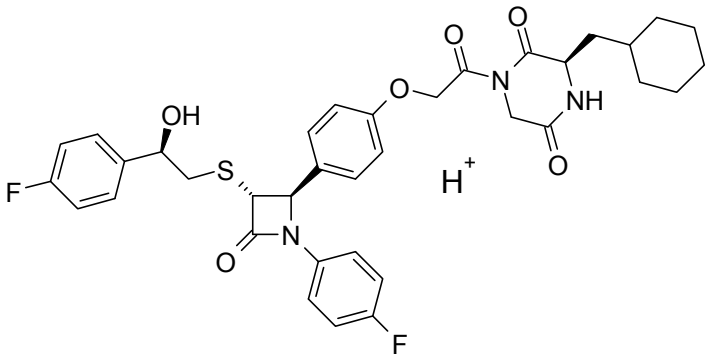

| | | | | | | |
|---|---|---|---|---|---|---|
| Minimum: | | | | -1.5 | | |
| Maximum: | | 15.0 | 10.0 | 50.0 | | |
| Mass | Calc. Mass | mDa | PPM | DBE | i-FIT | Formula |
| 696.2568 | 696.2555 | 1.3 | 1.9 | 17.5 | 4.6 | C36 H40 N3 O7 F2 S |




AZD4121 (**1**)

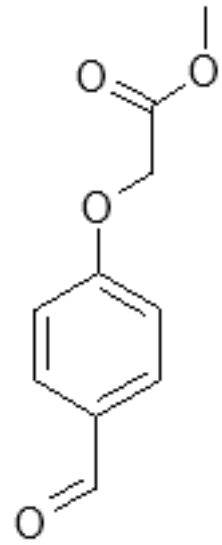
**HRMS-Proposed structure for the major contaminant in the API**



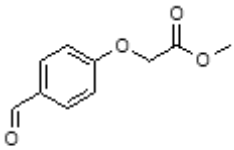




Calc. Mass 678.2449

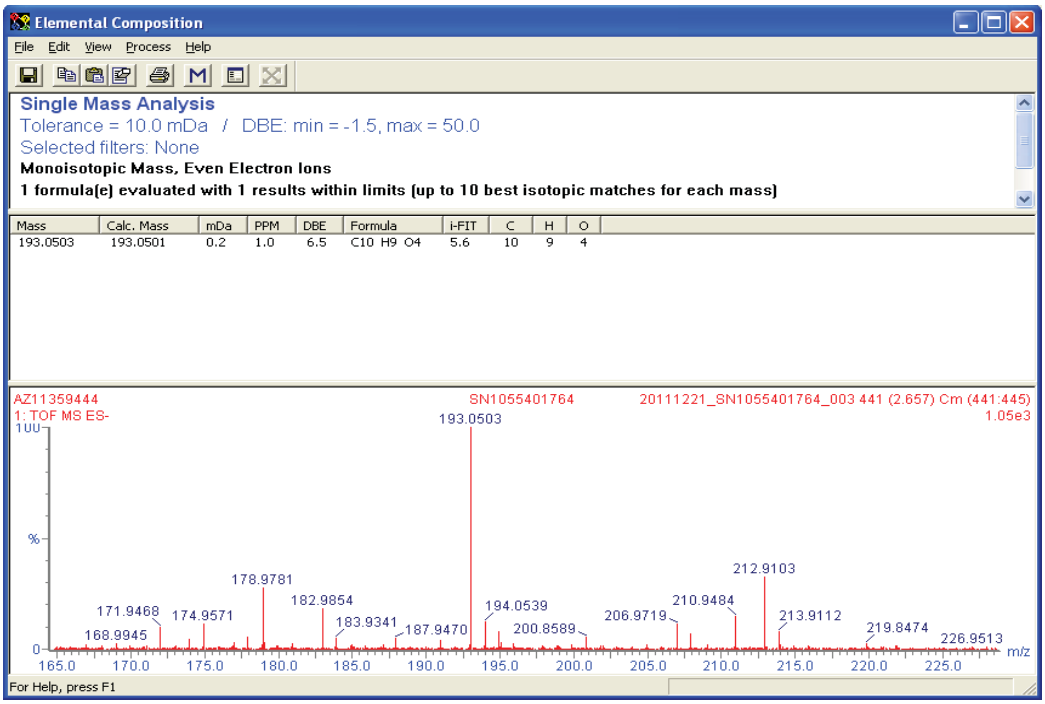
| | |
|---|---|
| Data File Name | / / samba-hpc/ nmrdata/ HREG/ h1000_1099/ h1032/ H_103214.001_h1nmr_1.fid/ fid |
| Owner | medkem |
| Solvent | CDCL3 |
| Acquisition Date | 2005-06-14T15:52:37 |
| Temperature | 25.0 |
| Number of Scans | 16 |
| Filename | |
| Sample_ID | |
| EN | |
| experiment | h1nmr |
| operator | |
| chemist | |
| Sample# | 83 |
| prid | |
| Date | Jun 14 2005 |
| solvent | CDCL3 |
| NMR Frequency | 400 |
| probe | dbg400 |
| temperature | 25 |
| nt | 16 |
| site | AstraZeneca R&D Mölndal |
| Text | |
| Comment | H_103214.001_h1nmr_1.fid |
| Spectrometer | inova |
| Author | medkem |
| Experiment | 1D |
| Pulse Width | 0.0000 |
| Acquisition Time | 3.9993 |

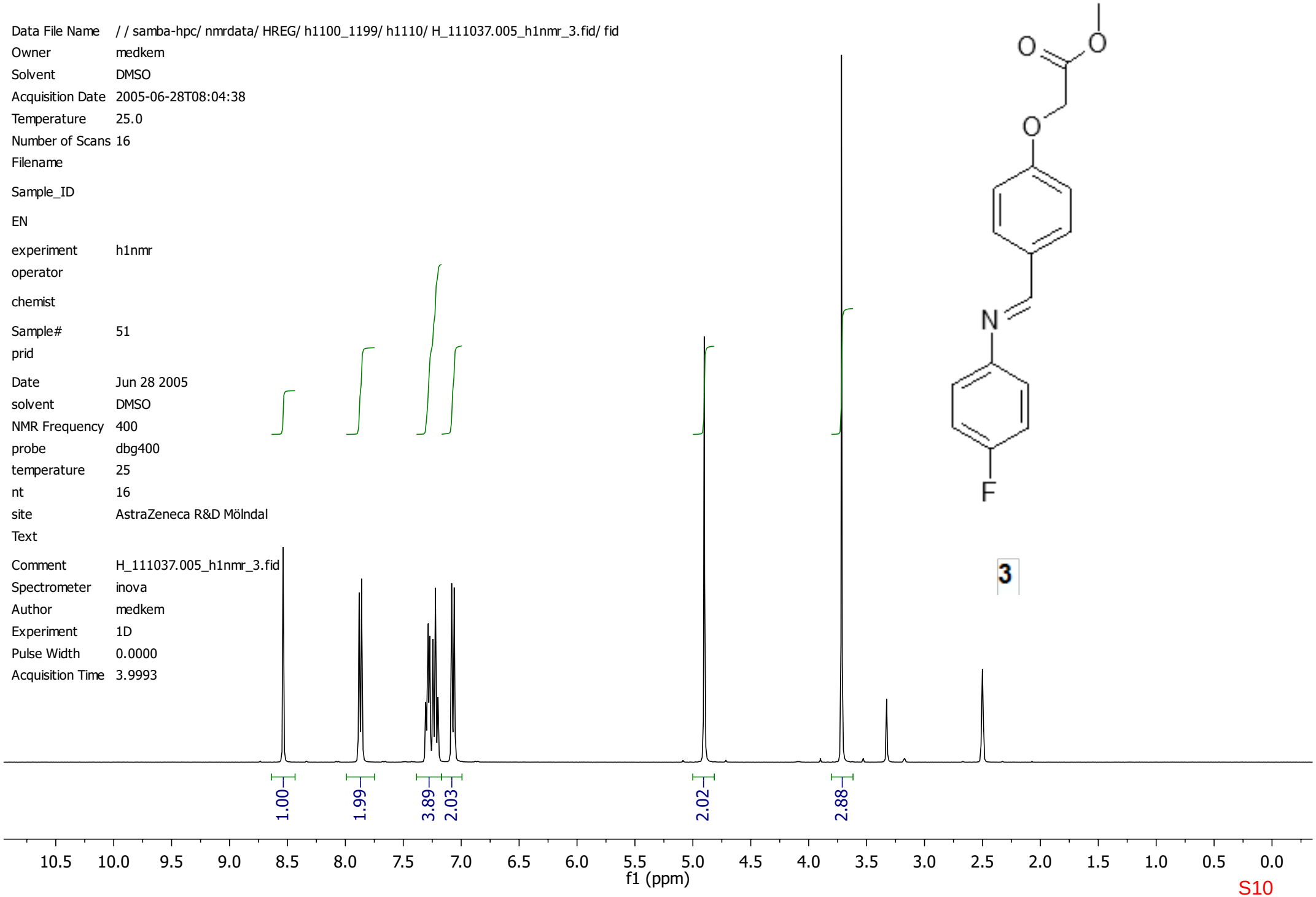



**2**



**2**


Elemental Composition
File Edit View Process Help
Single Mass Analysis
Tolerance = 10.0 mDa / DBE: min = -1.5, max = 50.0
Selected filters: None
Monoisotopic Mass, Even Electron Ions
1 formula(e) evaluated with 1 results within limits (up to 10 best isotopic matches for each mass)
Mass Calc. Mass mDa PPM DBE Formula i-FIT C H O
193.0503 193.0501 0.2 1.0 6.5 C10 H9 O4 5.6 10 9 4
AZ11359444
SN1055401764
20111221_SN1055401764_003 441 (2.657) Cm (441:445)
1: TOF MS ES-
1.05e3
193.0503
212.9103
178.9781
182.9854
171.9468
174.9571
183.9341
187.9470
194.0539
200.8589
206.9719
210.9484
213.9112
219.8474
168.9945
226.9513
m/z
For Help, press F1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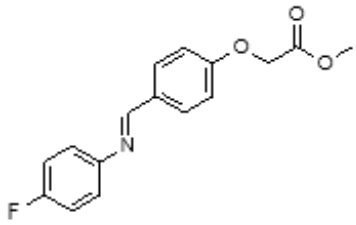
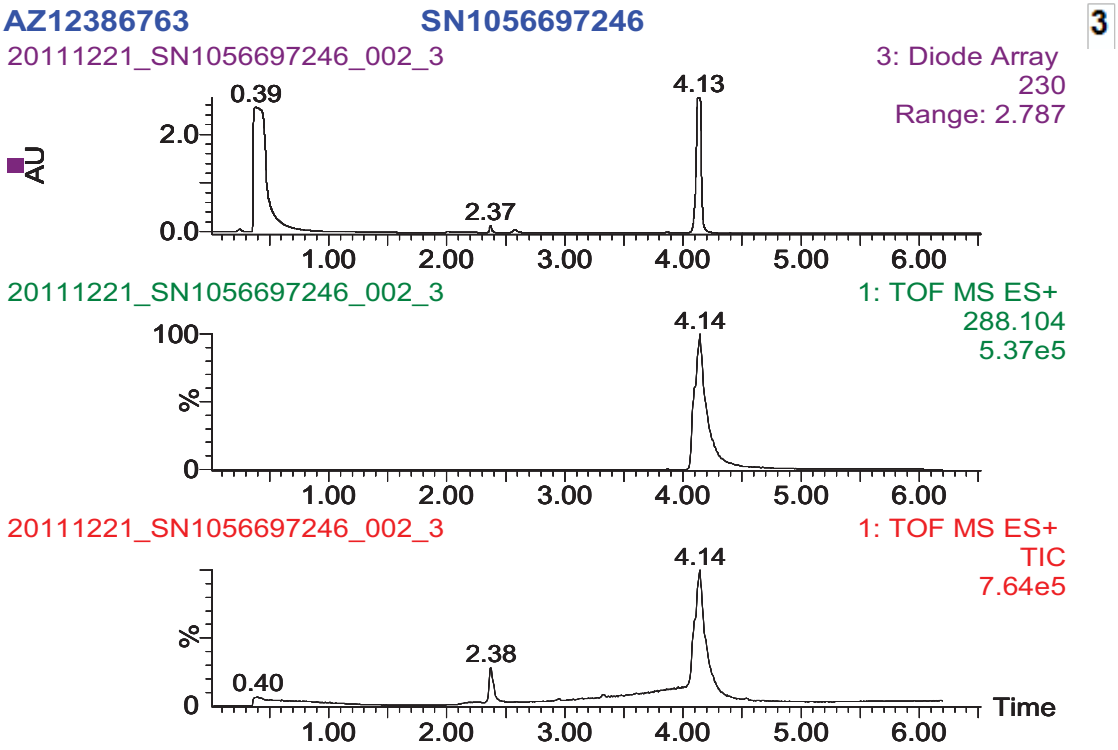
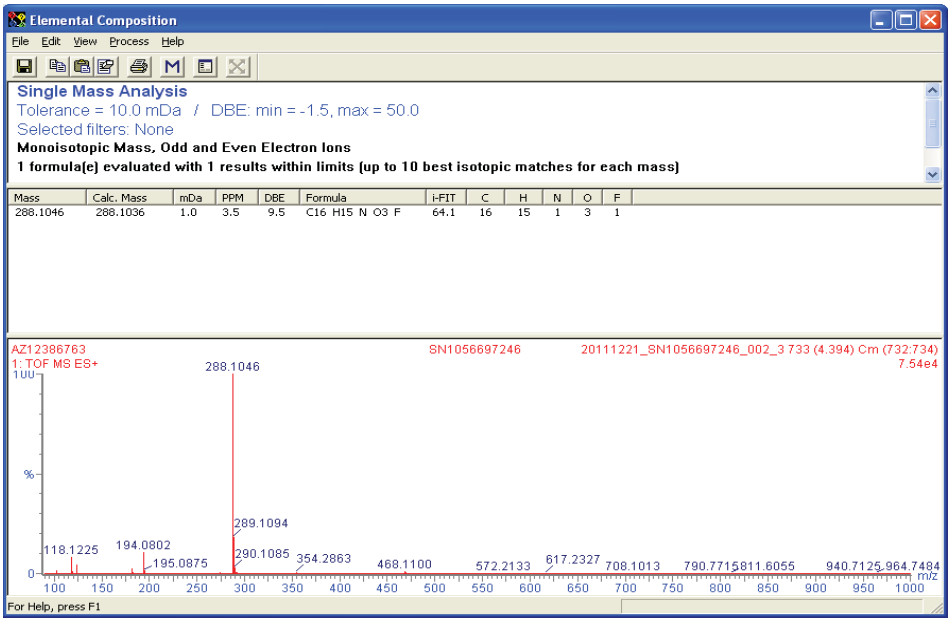
Owner medkem
Solvent DMSO
Acquisition Date 2005-06-28T08:04:38
Temperature 25.0
Number of Scans 16
Filename
Sample_ID
EN
experiment h1nmr
operator
chemist
Sample# 51
prid
Date Jun 28 2005
solvent DMSO
NMR Frequency 400
probe dbg400
temperature 25
nt 16
site AstraZeneca R&D Mölndal
Text
Comment H_111037.005_h1nmr_3.fid
Spectrometer inova
Author medkem
Experiment 1D
Pulse Width 0.0000
Acquisition Time 3.9993
O
O
O
N
F
3
1.00
1.99
3.89
2.03
2.02
2.88
10.5 10.0 9.5 9.0 8.5 8.0 7.5 7.0 6.5 6.0 5.5 5.0 4.5 4.0 3.5 3.0 2.5 2.0 1.5 1.0 0.5 0.0
f1 (ppm)

**Cars4305_SN1056697246**

Accurate mass determination





Data File Name / / samba-hpc/ nmrdata/ HREG/ h1000_1099/ h1088/ H_108817.023_h1nmr_1.fid/ fid
Owner

Solvent DMSO
Acquisition Date 2005-08-11T08:53:52
Temperature 25.0
Number of Scans 16
Filename

Sample_ID

EN

experiment h1nmr
operator

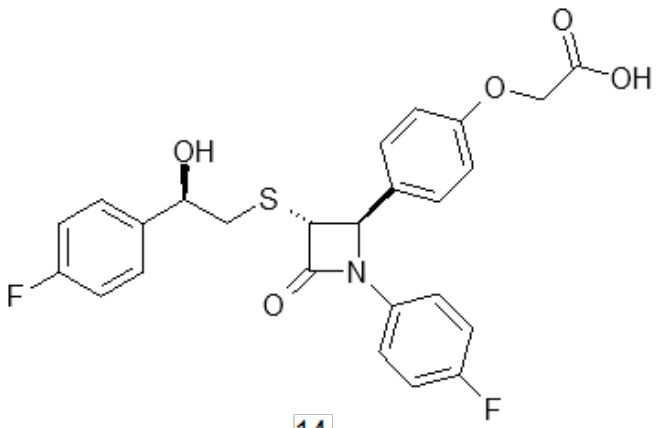
chemist

Sample# 0
prid

Date Aug 11 2005
solvent DMSO
NMR Frequency 600
probe tr600
temperature 25
nt 16
site AstraZeneca R&D Mölndal
Text

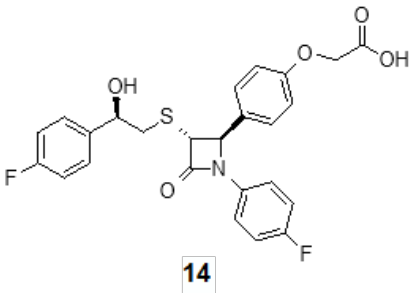
Comment H_108817.023_h1nmr_1.fid
Spectrometer inova
Author

Experiment 1D
Pulse Width 0.0000
Acquisition Time 4.0000




14

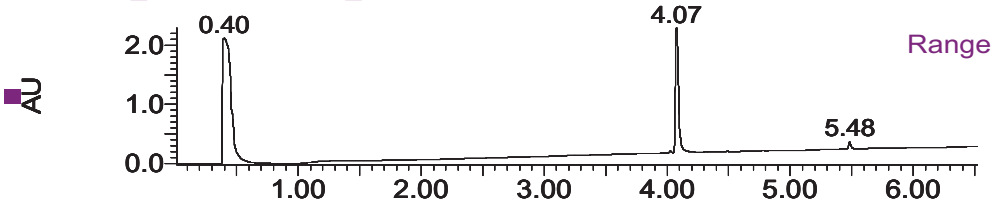
Cars4307_SN1054736493
Accurate mass determination



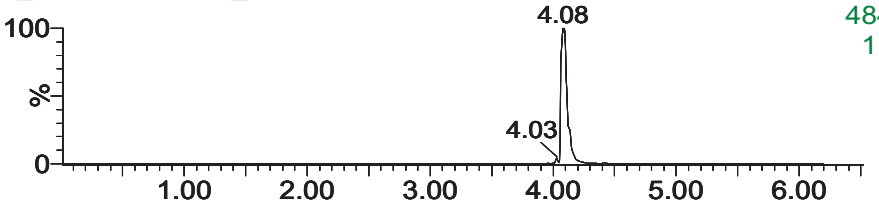

14

**AZ12447465** **SN1054736493**

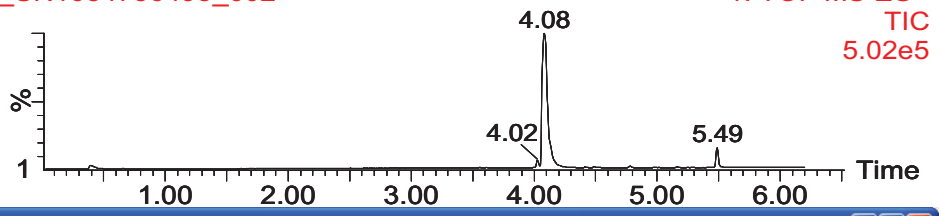
20111221_SN1054736493_002 3: Diode Array
230
Range: 2.29

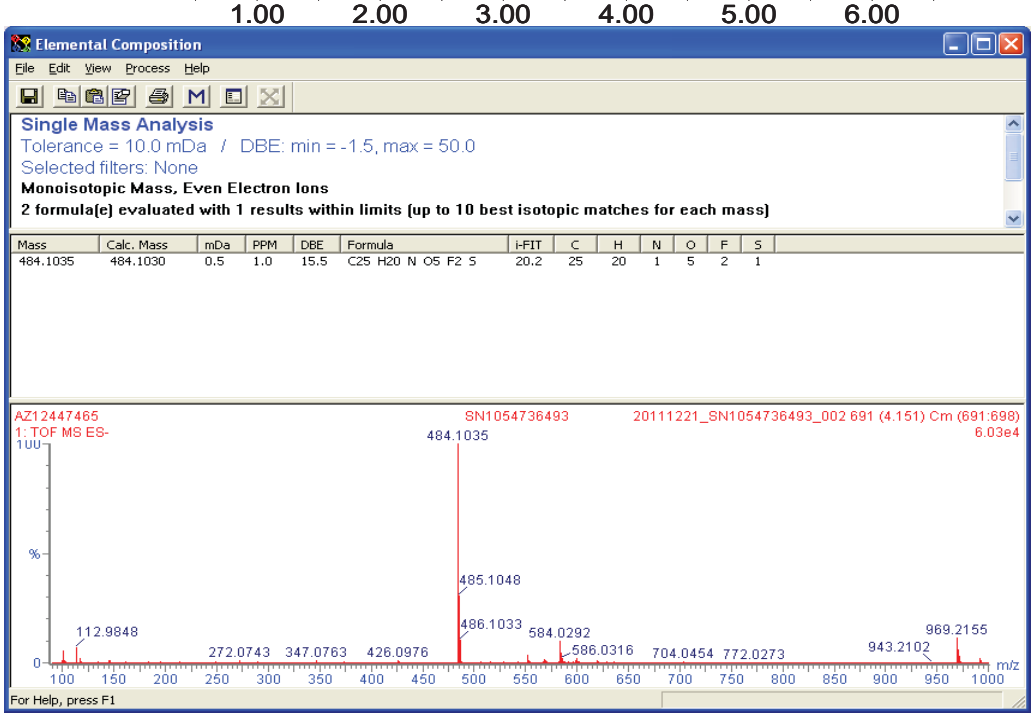



20111221_SN1054736493_002 1: TOF MS ES-
484.103
1.28e5

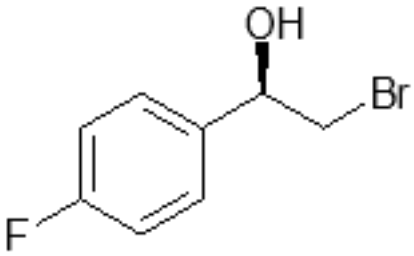



20111221_SN1054736493_002 1: TOF MS ES-
TIC
5.02e5



| | |
|---|---|
| Data File Name | / / samba-hpc/ nmrdata/ HREG/ h1000_1099/ h1070/ H_107004.012_h1nmr_1.fid/ fid |
| Owner | medkem |
| Solvent | CDCL3 |
| Acquisition Date | 2005-07-12T13:00:36 |
| Temperature | 25.0 |
| Number of Scans | 16 |
| Filename | |
| Sample_ID | |
| EN | |
| experiment | h1nmr |
| operator | |
| chemist | |
| Sample# | 63 |
| prid | |
| Date | Jul 12 2005 |
| solvent | CDCL3 |
| NMR Frequency | 400 |
| probe | dbg400 |
| temperature | 25 |
| nt | 16 |
| site | AstraZeneca R&D Mölndal |
| Text | |
| Comment | H_107004.012_h1nmr_1.fid |
| Spectrometer | inova |
| Author | medkem |
| Experiment | 1D |
| Pulse Width | 0.0000 |
| Acquisition Time | 3.9993 |




**15**

## Sample Information

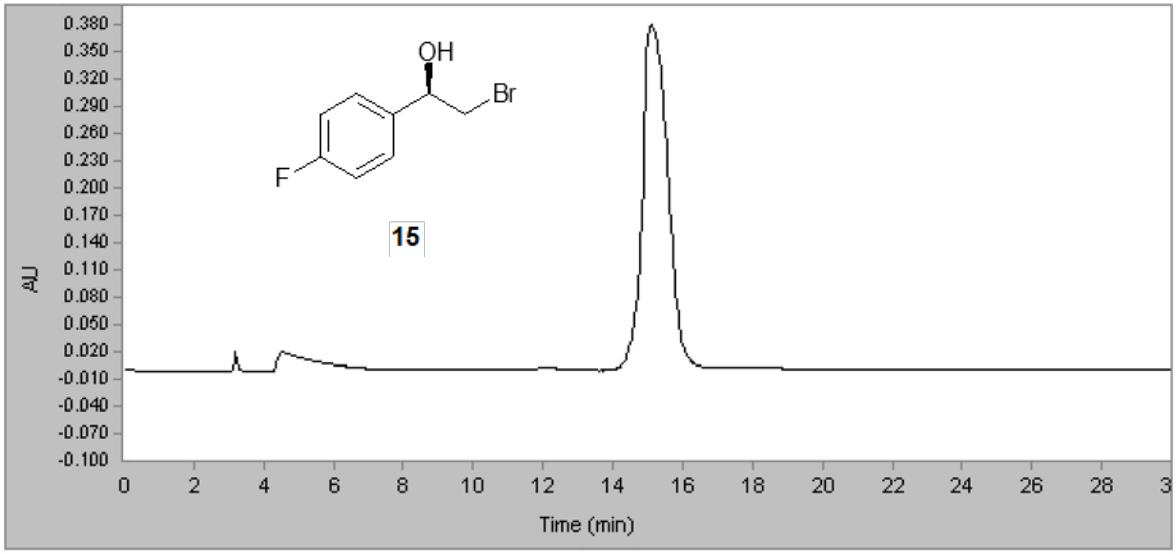
| Sample ID | Compound name | Project name |
|---|---|---|
| SN1056079599 | AZ12372917 | CAI |

## Analytical Conditions

| Column | Dimensions (mm) | Particle Size (µm) |
|---|---|---|
| (R,R) Whelk-O1 | 4.6 * 250 | 10 |
| **Mobile Phase** | | |
| Hep/IPA 99/1 | | |
| **Flow (ml/min)** | **Detection (nm)** | **Temperature (C)** |
| 1 ml/min | PDA 265.0 nm | rt |
| **Comment** | | |
| | | |

## Analytical Result

**UV Chromatogram**




| Retention Time | Area | Area % | K' | N | USP Resolution |
|---|---|---|---|---|---|
| 3.203 | 103308 | 0.54 | 0.000 | 10424 | |
| 12.084 | 97900 | 0.51 | 2.773 | 1799 | 14.62 |
| 15.101 | 18991644 | 98.95 | 3.714 | 2557 | 2.63 |

**ee: 99.0%**

**Chemist Signature**

**Print Date**
2011-12-19

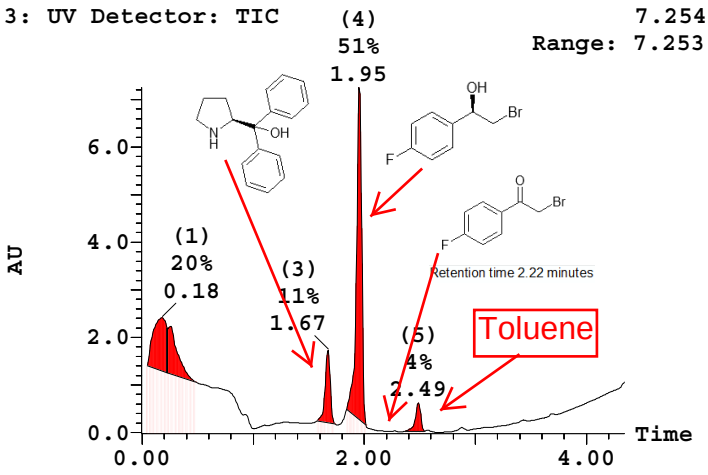
**Openlynx Report walk-up LC/MS**

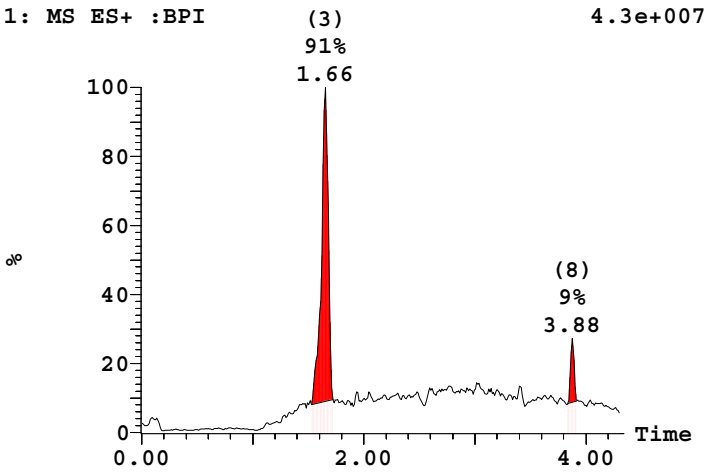
JobCode (batch):klcc7601562 | Sample: 1 | Date:16-Dec-2011
UserName:klcc760 | Sample ID:klcc7601562-1 | Description:efternatt
Vial:2,1:67 | Instrument:W3100#C07310020W | Location: KJ208
Method:C:\MassLynx\IS_5min standard.olp | pH: 7 | Column: Sunfire C18 5µ 3x50 mm

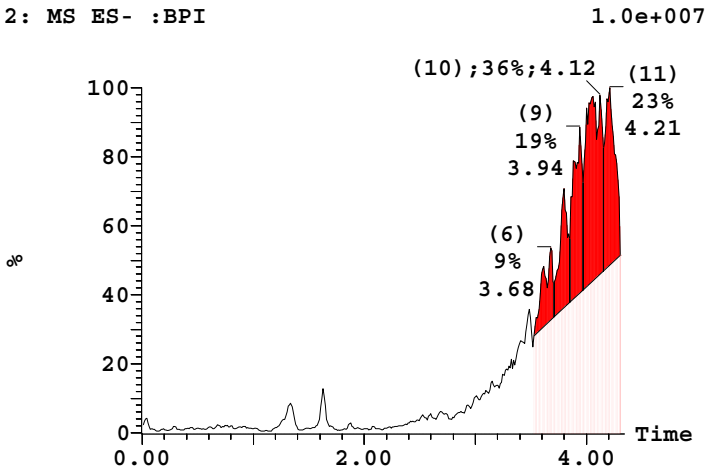
Printed: Fri Dec 16 08:05:58 2011

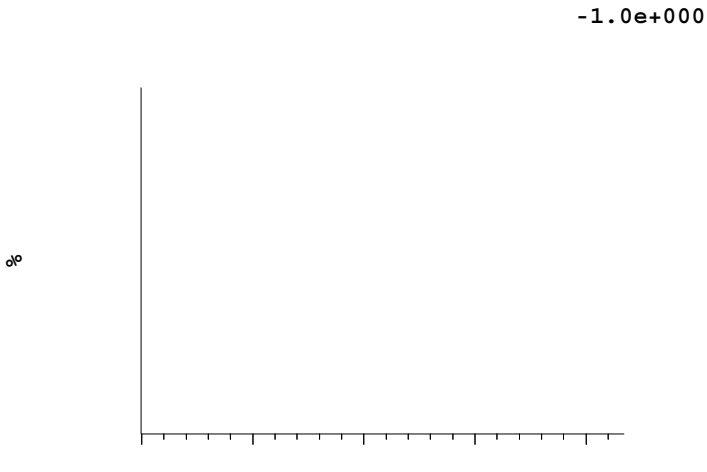
**Sample Report:**

**Sample ID klcc7601562-1 Description efternatt**

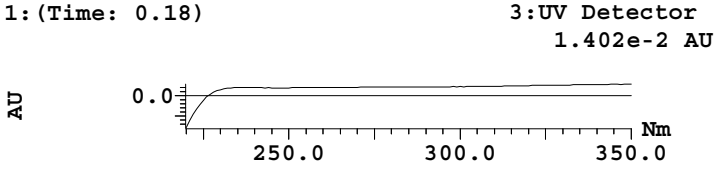


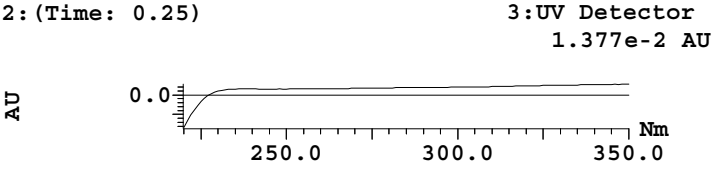



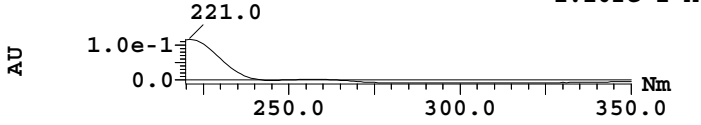

| Peak ID | Time | Mass Found |
|---|---|---|
| 1 | 0.18 | |



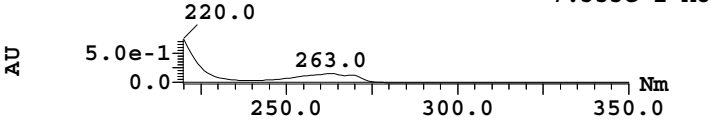

| Peak ID | Time | Mass Found |
|---|---|---|
| 2 | 0.25 | |




| Peak ID | Time | Mass Found |
|---|---|---|
| 3 | 1.66 | |




| Peak ID | Time | Mass Found |
|---|---|---|
| 4 | 1.95 | |



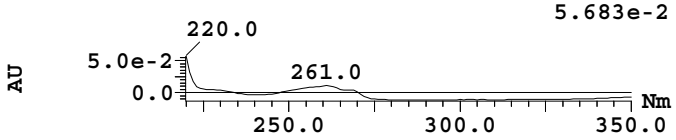
JobCode (batch):klcc7601562 Sample: 1 Date:16-Dec-2011
UserName:klcc760 Sample ID:klcc7601562-1 Description:efternatt
Vial:2,1:67 Instrument:W3100#C07310020W Location: KJ208
Method:C:\MassLynx\IS_5min standard.olp pH: 7 Column: Sunfire C18 5µ 3x50 mm

Printed: Fri Dec 16 08:05:58 2011

**Sample Report (continued):**

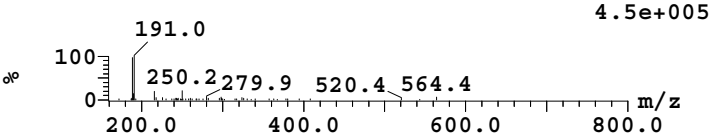
| Peak ID | Time |
|---|---|
| 5 | 2.49 |

5:(Time: 2.49) 3:UV Detector 5.683e-2 AU



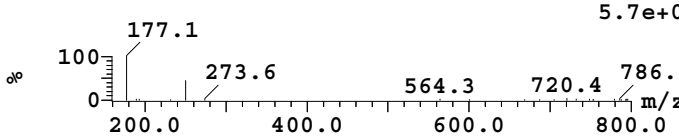

| Peak ID | Time |
|---|---|
| 1 | 0.18 |

1:(Time: 0.18) 1:MS ES+ 4.5e+005



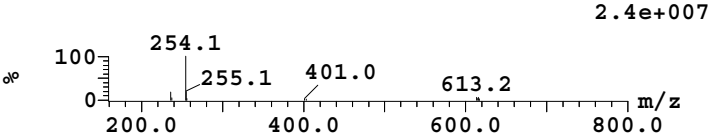

| Peak ID | Time |
|---|---|
| 2 | 0.25 |

2:(Time: 0.25) 1:MS ES+ 5.7e+004



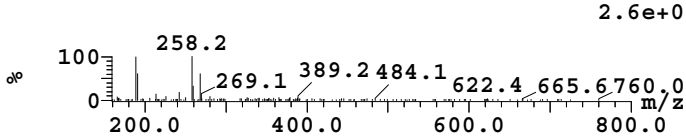

| Peak ID | Time |
|---|---|
| 3 | 1.66 |

3:(Time: 1.66) 1:MS ES+ 2.4e+007



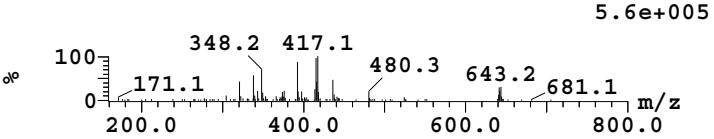

| Peak ID | Time |
|---|---|
| 4 | 1.95 |

4:(Time: 1.95) 1:MS ES+ 2.6e+005



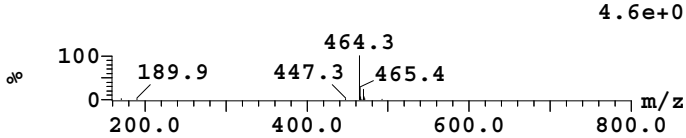

| Peak ID | Time |
|---|---|
| 5 | 2.49 |

5:(Time: 2.49) 1:MS ES+ 5.6e+005



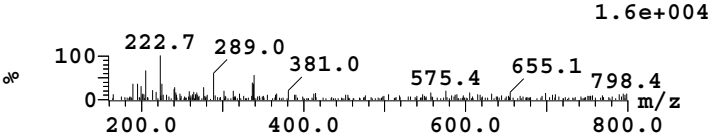

| Peak ID | Time |
|---|---|
| 8 | 3.88 |

8:(Time: 3.88) 1:MS ES+ 4.6e+006



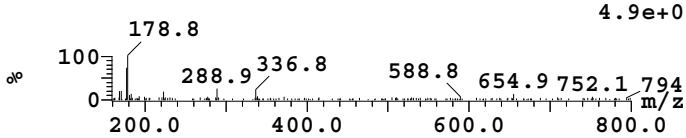

| Peak ID | Time |
|---|---|
| 1 | 0.18 |

1:(Time: 0.18) 2:MS ES- 1.6e+004



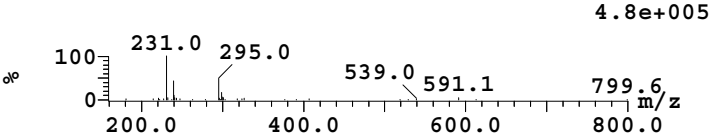

| Peak ID | Time |
|---|---|
| 2 | 0.25 |

2:(Time: 0.25) 2:MS ES- 4.9e+004



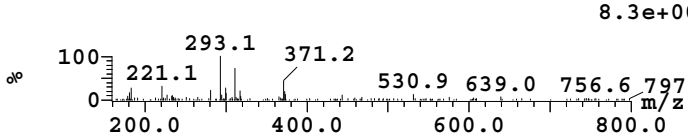

| Peak ID | Time |
|---|---|
| 3 | 1.66 |

3:(Time: 1.67) 2:MS ES- 4.8e+005



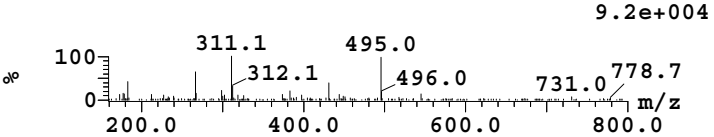

| Peak ID | Time |
|---|---|
| 4 | 1.95 |

4:(Time: 1.95) 2:MS ES- 8.3e+004




| Peak ID | Time |
|---|---|
| 5 | 2.49 |

5:(Time: 2.49) 2:MS ES- 9.2e+004



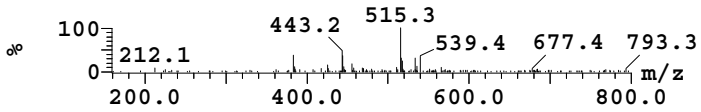
JobCode (batch):klcc7601562 Sample: 1 Date:16-Dec-2011
UserName:klcc760 Sample ID:klcc7601562-1 Description:efternatt
Vial:2,1:67 Instrument:W3100#C07310020W Location: KJ208
Method:C:\MassLynx\IS_5min standard.olp pH: 7 Column: Sunfire C18 5µ 3x50 mm

Printed: Fri Dec 16 08:05:58 2011

**Sample Report (continued):**

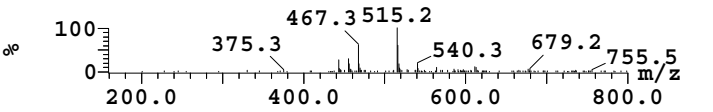
| Peak ID | Time | Mass Found |
|---|---|---|
| 6 | 3.68 | |

6:(Time: 3.68) 2:MS ES- 2.1e+005



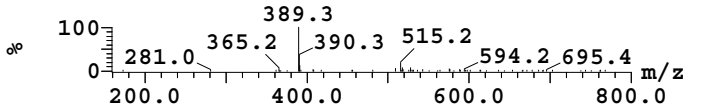

| Peak ID | Time | Mass Found |
|---|---|---|
| 7 | 3.80 | |

7:(Time: 3.80) 2:MS ES- 3.7e+005



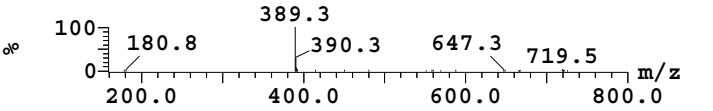

| Peak ID | Time | Mass Found |
|---|---|---|
| 9 | 3.94 | |

9:(Time: 3.94) 2:MS ES- 9.1e+005



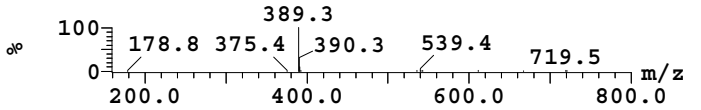

| Peak ID | Time | Mass Found |
|---|---|---|
| 10 | 4.12 | |

10:(Time: 4.12) 2:MS ES- 2.3e+006




| Peak ID | Time | Mass Found |
|---|---|---|
| 11 | 4.21 | |

11:(Time: 4.21) 2:MS ES- 3.8e+006



Data File Name / / samba-hpc/ nmrdata/ HREG/ h1100_1199/ h1110/ H_111043.008_h1nmr_2.fid/ fid
Owner

Solvent CDCl3
Acquisition Date 2005-07-15T08:24:15
Temperature 25.0
Number of Scans 16
Filename

Sample_ID

EN

experiment h1nmr
operator

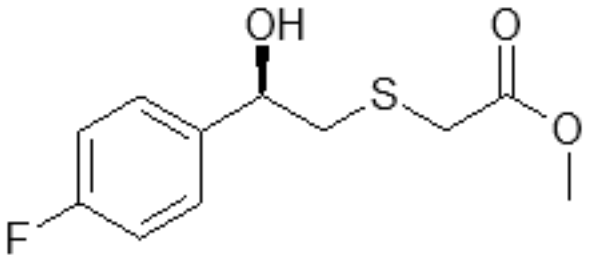
chemist

Sample# 2
prid

Date Jul 15 2005
solvent CDCl3
NMR Frequency 600
probe tr600
temperature 25
nt 16
site AstraZeneca R&D Mölndal
Text

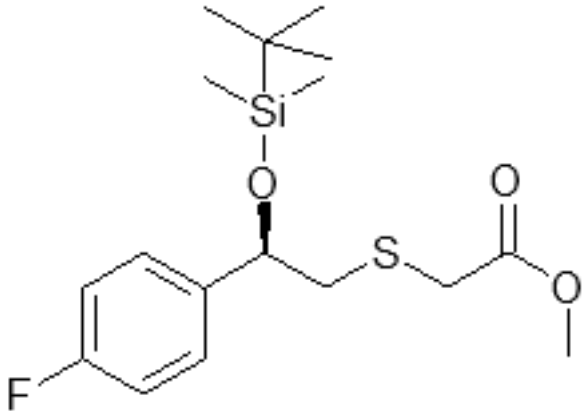
Comment H_111043.008_h1nmr_2.fid
Spectrometer inova
Author

Experiment 1D
Pulse Width 0.0000
Acquisition Time 4.0000



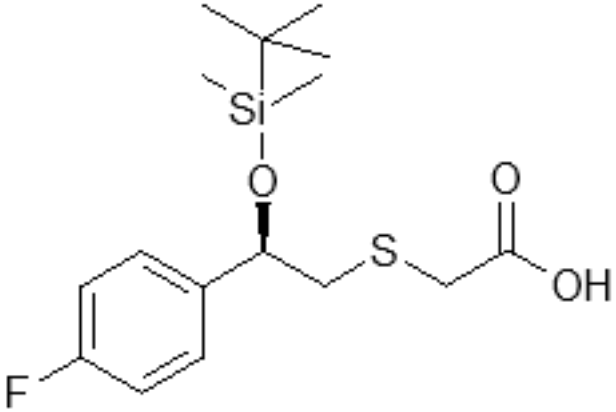

**16**

| | |
|---|---|
| Data File Name | / / samba-hpc/ nmrdata/ HREG/ h1000_1099/ h1032/ H_103209.003_h1nmr_1.fid/ fid |
| Owner | medkem |
| Solvent | CDCL3 |
| Acquisition Date | 2005-07-18T14:29:22 |
| Temperature | 25.0 |
| Number of Scans | 16 |
| Filename | |
| Sample_ID | |
| EN | |
| experiment | h1nmr |
| operator | |
| chemist | |
| Sample# | 9 |
| prid | |
| Date | Jul 18 2005 |
| solvent | CDCL3 |
| NMR Frequency | 400 |
| probe | dbg400 |
| temperature | 25 |
| nt | 16 |
| site | AstraZeneca R&D Mölndal |
| Text | |
| Comment | H_103209.003_h1nmr_1.fid<br>TBDMSOH present as one major impurity |
| Spectrometer | inova |
| Author | medkem |
| Experiment | 1D |
| Pulse Width | 0.0000 |
| Acquisition Time | 3.9993 |




**17**

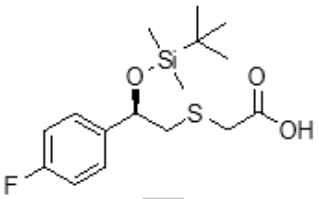
| | |
|---|---|
| Data File Name | / / samba-hpc/ nmrdata/ HREG/ h1100_1199/ h1110/ H_111047.010_h1nmr_1.fid/ fid |
| Owner | medkem |
| Solvent | CDCL3 |
| Acquisition Date | 2005-07-20T08:29:18 |
| Temperature | 25.0 |
| Number of Scans | 16 |
| Filename | |
| Sample_ID | |
| EN | |
| experiment | h1nmr |
| operator | |
| chemist | |
| Sample# | 7 |
| prid | |
| Date | Jul 20 2005 |
| solvent | CDCL3 |
| NMR Frequency | 400 |
| probe | dbg400 |
| temperature | 25 |
| nt | 16 |
| site | AstraZeneca R&D Mölndal |
| Text | |
| Comment | H_111047.010_h1nmr_1.fid |
| Spectrometer | inova |
| Author | medkem |
| Experiment | 1D |
| Pulse Width | 0.0000 |
| Acquisition Time | 3.9993 |



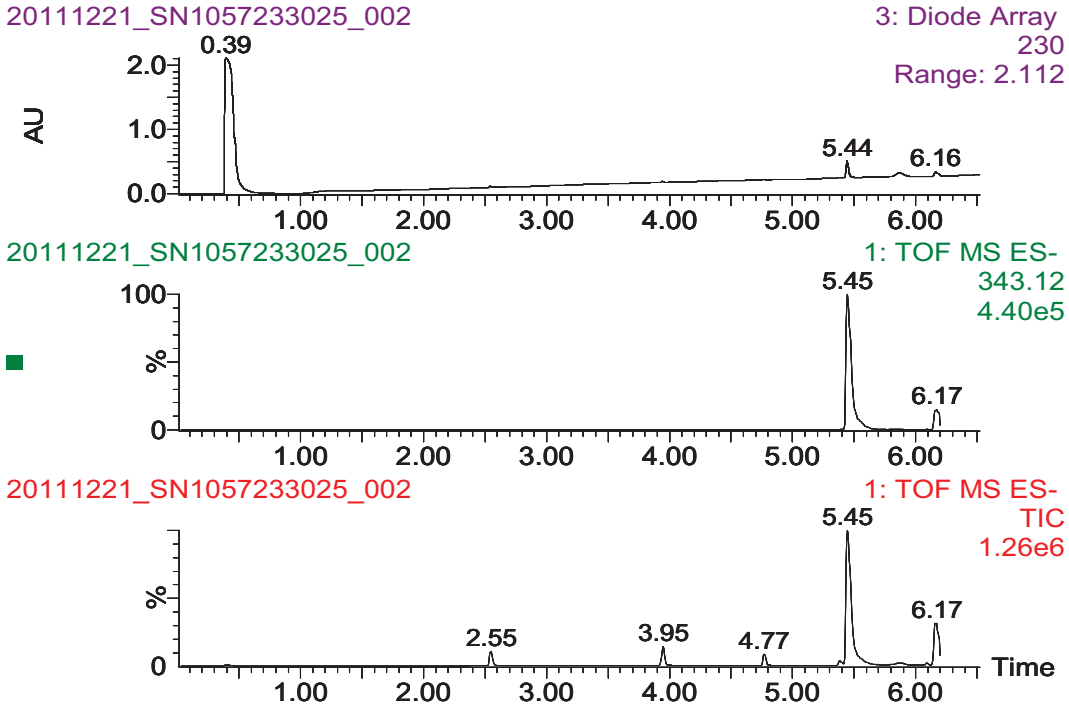

**18**

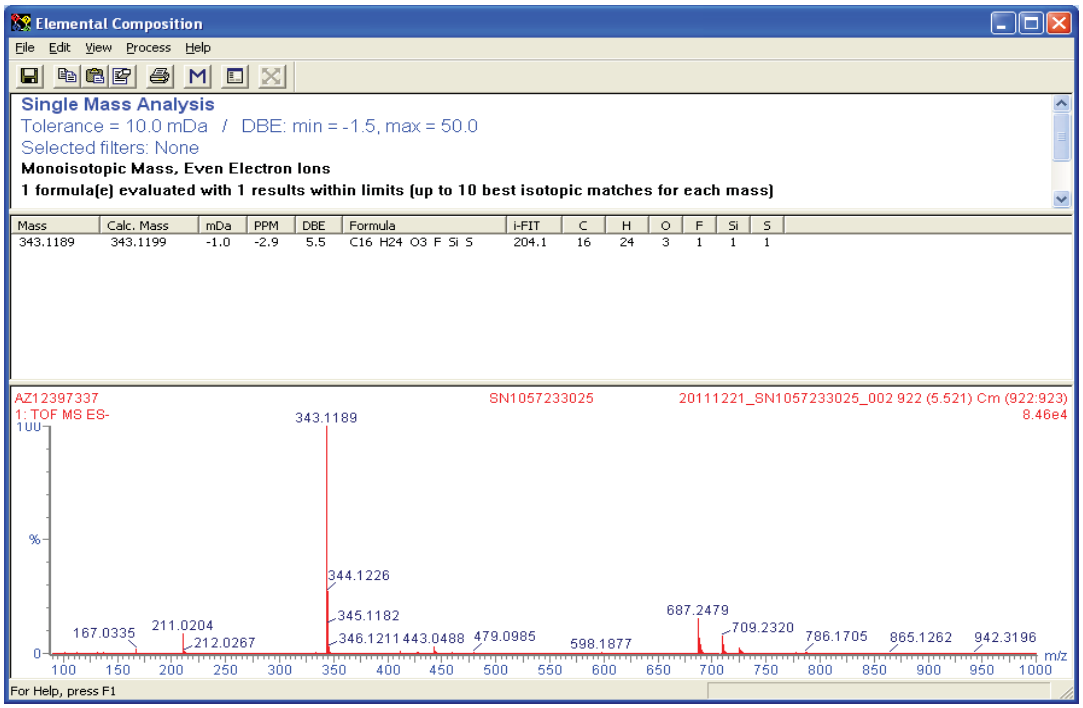
**Cars4309_SN1057233025**

Accurate mass determination

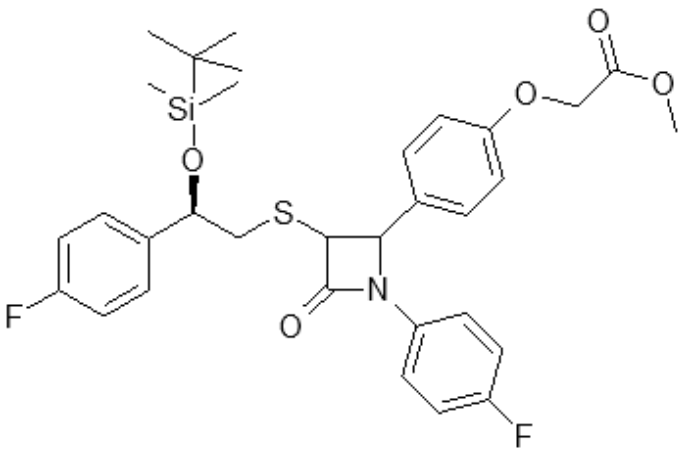



18

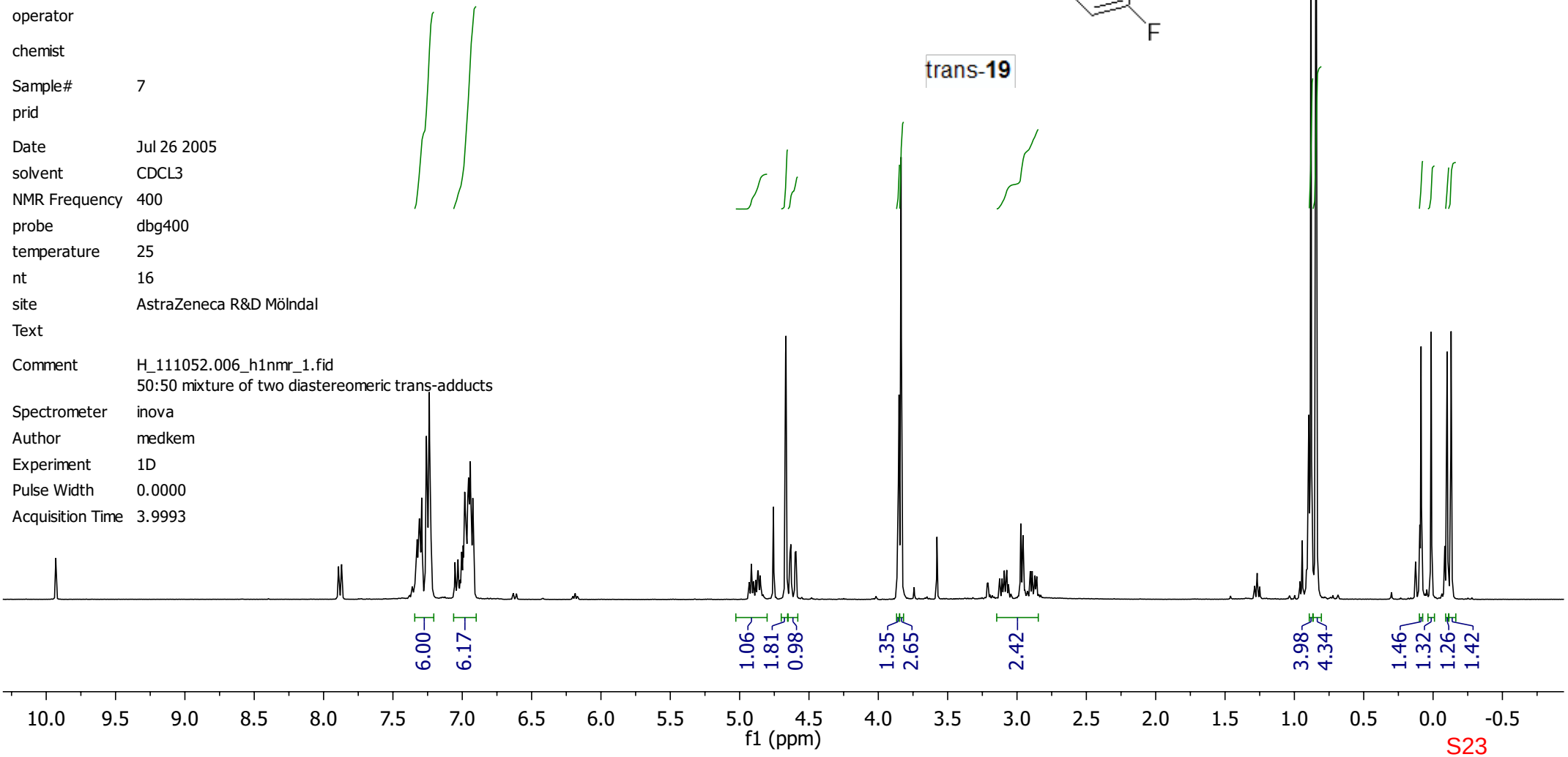


| | |
|---|---|
| Data File Name | / / samba-hpc/ nmrdata/ HREG/ h1100_1199/ h1110/ H_111052.006_h1nmr_1.fid/ fid |
| Owner | medkem |
| Solvent | CDCL3 |
| Acquisition Date | 2005-07-26T08:22:06 |
| Temperature | 25.0 |
| Number of Scans | 16 |
| Filename | |
| Sample_ID | |
| EN | |
| experiment | h1nmr |
| operator | |
| chemist | |
| Sample# | 7 |
| prid | |
| Date | Jul 26 2005 |
| solvent | CDCL3 |
| NMR Frequency | 400 |
| probe | dbg400 |
| temperature | 25 |
| nt | 16 |
| site | AstraZeneca R&D Mölndal |
| Text | |
| Comment | H_111052.006_h1nmr_1.fid<br>50:50 mixture of two diastereomeric trans-adducts |
| Spectrometer | inova |
| Author | medkem |
| Experiment | 1D |
| Pulse Width | 0.0000 |
| Acquisition Time | 3.9993 |




trans-**19**



**Openlynx Report LC/MS KJ2 (ZQ-LAA155) hs**

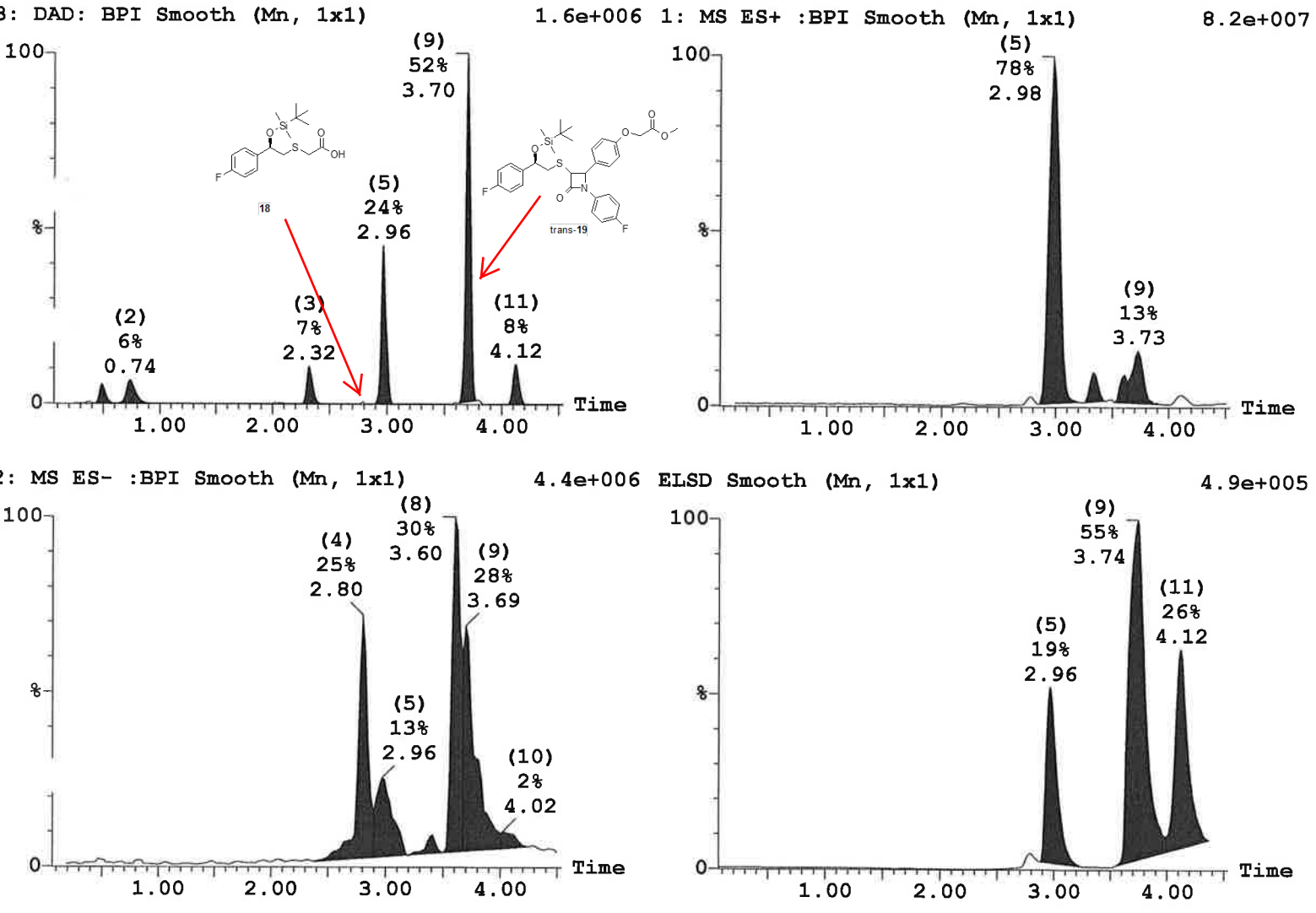
Sample: 1
File:hs290-1
Description:111052
UserName:hs

Vial:2,1:50
Date:05-Jul-2005
Method:C:\MassLynx\5min_steep.olp

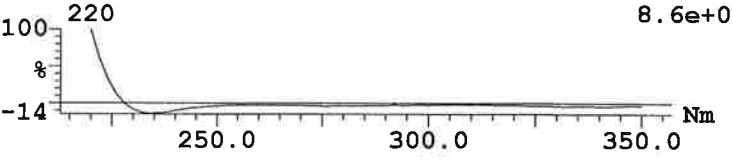
ID:hs290-1
Time:15:50:15
JobCode:hs290

Printed: Tue Jul 05 15:55:51 2005

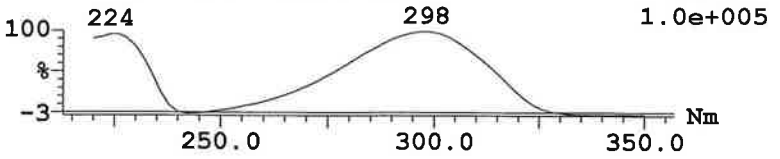
**Sample Report:** IPC, Staudinger reaction, lactam formation




| Peak ID | Time | Mass Found |
|---|---|---|
| 1 | 0.49 | |




| Peak ID | Time | Mass Found |
|---|---|---|
| 2 | 0.74 | |



**Openlynx Report LC/MS KJ2 (ZQ-LAA155) hs**

Sample: 1
File:hs291-1
Description:run
UserName:hs

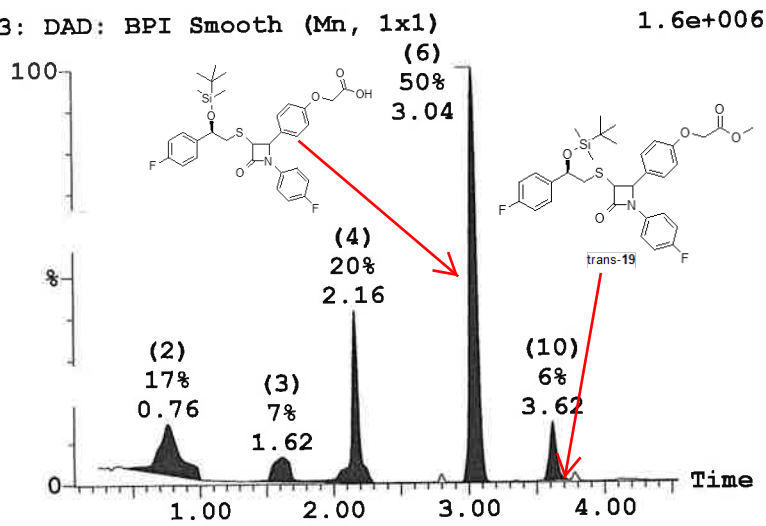
Vial:2,1:53
Date:06-Jul-2005
Method:C:\MassLynx\5min_steep.olp

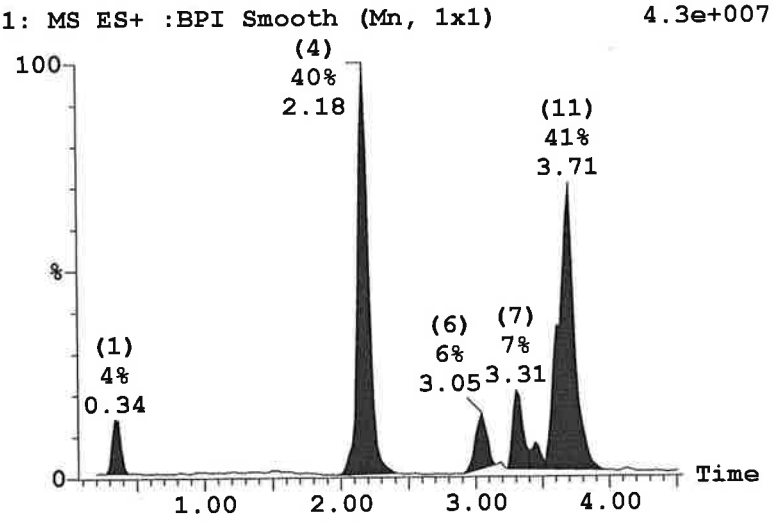
ID:hs291-1
Time:06:51:05
JobCode:hs291

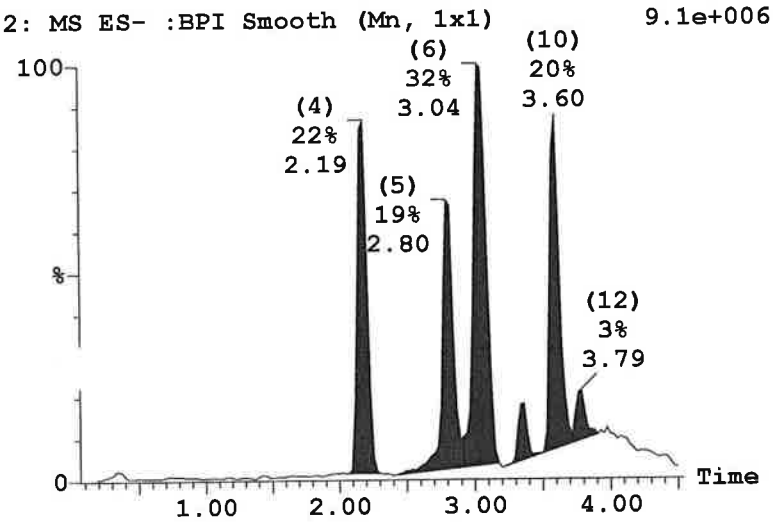
Printed: Wed Jul 06 06:56:41 2005

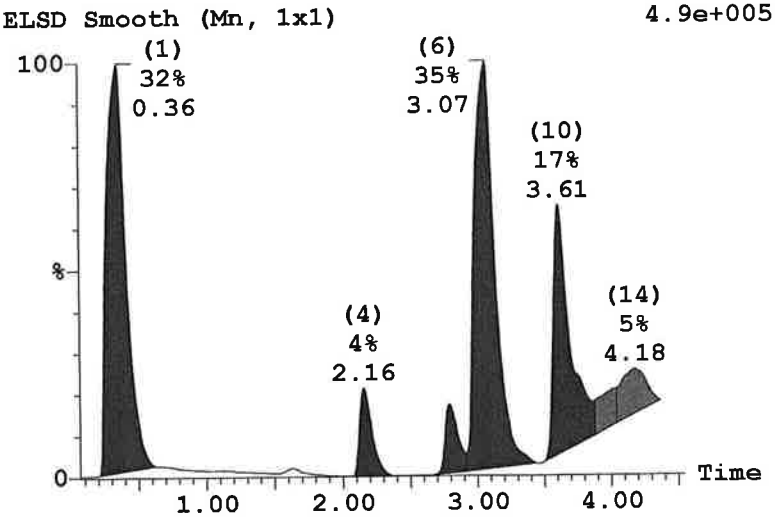
**Sample Report:**

IPC, hydrolysis

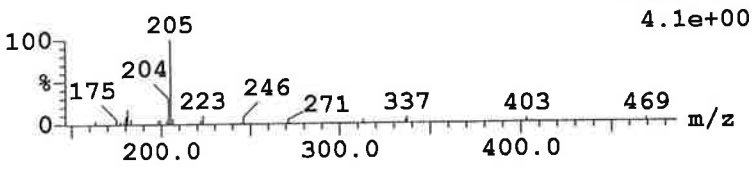


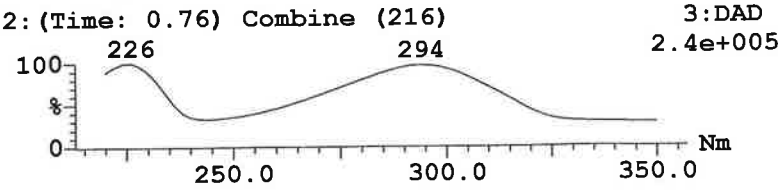



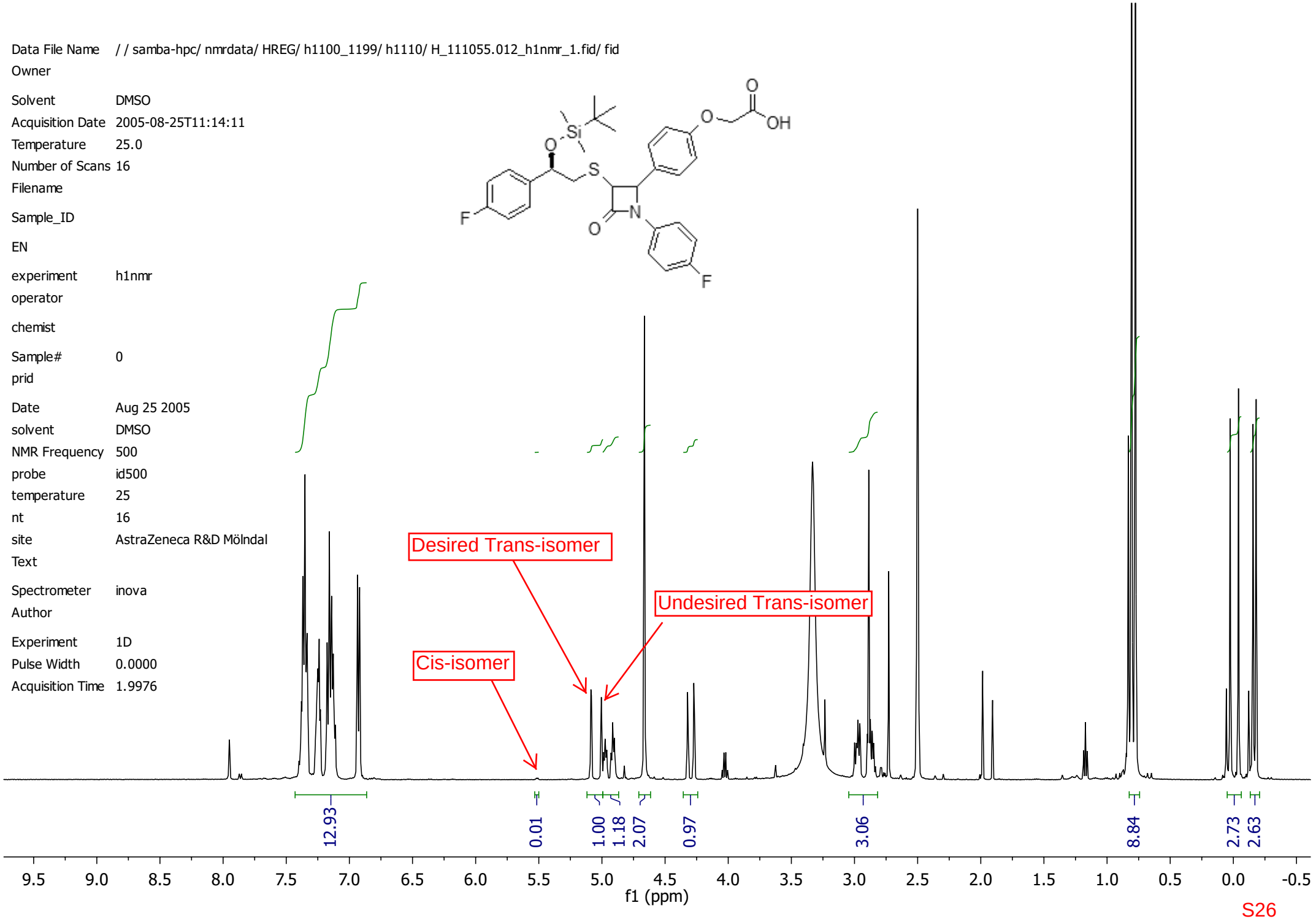



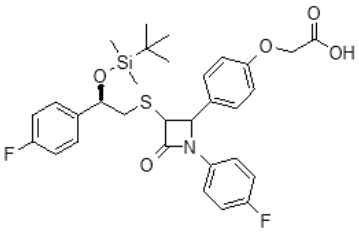

**Peak ID** 1 **Time** 0.34 **Mass Found**



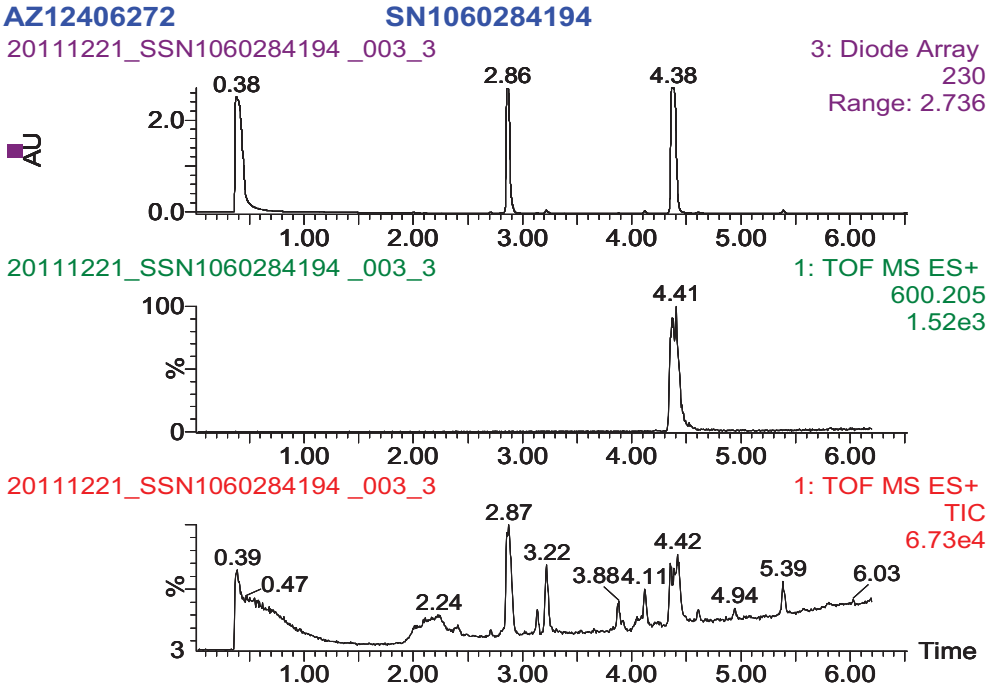

**Peak ID** 2 **Time** 0.76 **Mass Found**

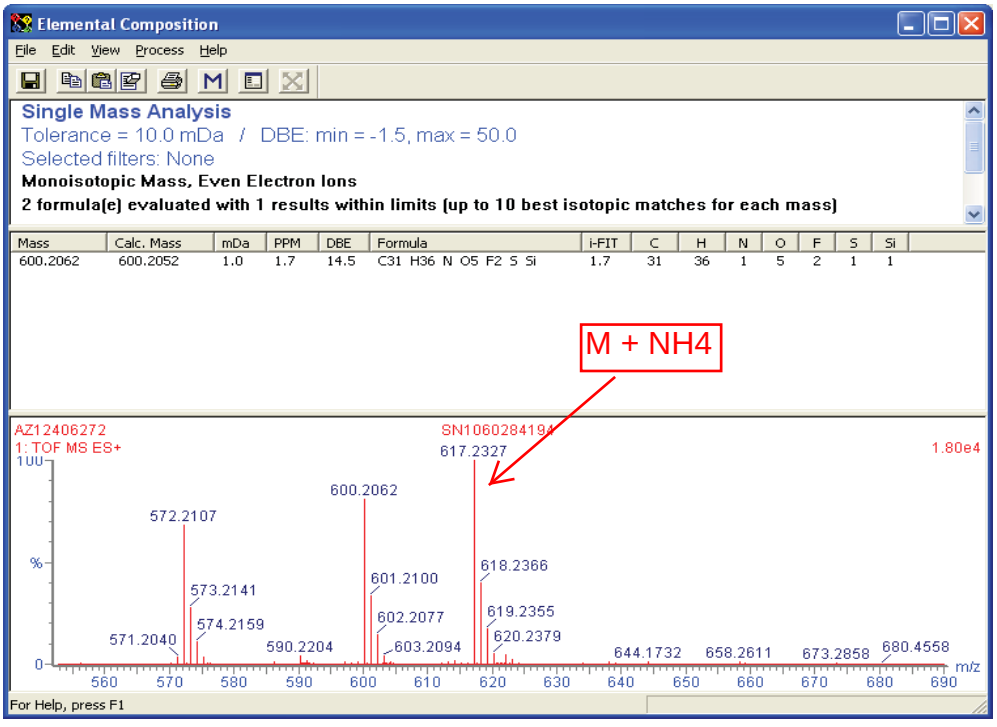



Data File Name / / samba-hpc/ nmrdata/ HREG/ h1100_1199/ h1110/ H_111055.012_h1nmr_1.fid/ fid
Owner
Solvent DMSO
Acquisition Date 2005-08-25T11:14:11
Temperature 25.0
Number of Scans 16
Filename
Sample_ID
EN
experiment h1nmr
operator
chemist
Sample# 0
prid
Date Aug 25 2005
solvent DMSO
NMR Frequency 500
probe id500
temperature 25
nt 16
site AstraZeneca R&D Mölndal
Text
Spectrometer inova
Author
Experiment 1D
Pulse Width 0.0000
Acquisition Time 1.9976
Desired Trans-isomer
Undesired Trans-isomer
Cis-isomer
12.93
0.01
1.00
1.18
2.07
0.97
3.06
8.84
2.73
2.63
f1 (ppm)

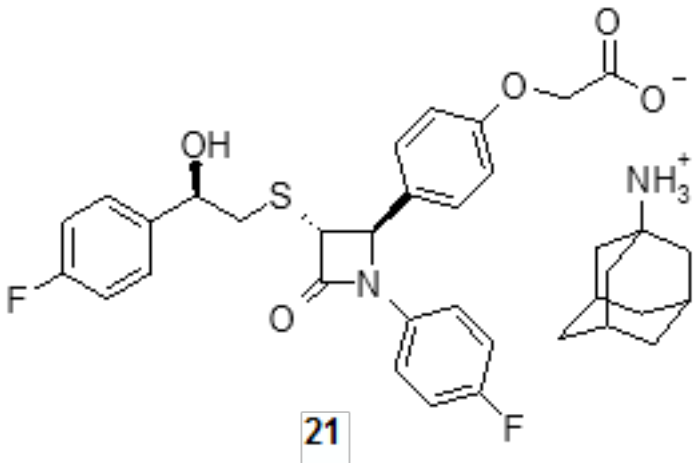


Cars4308_SN1060284194
Accurate mass determination



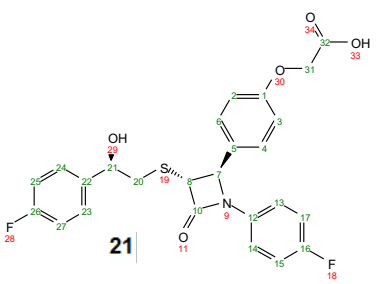



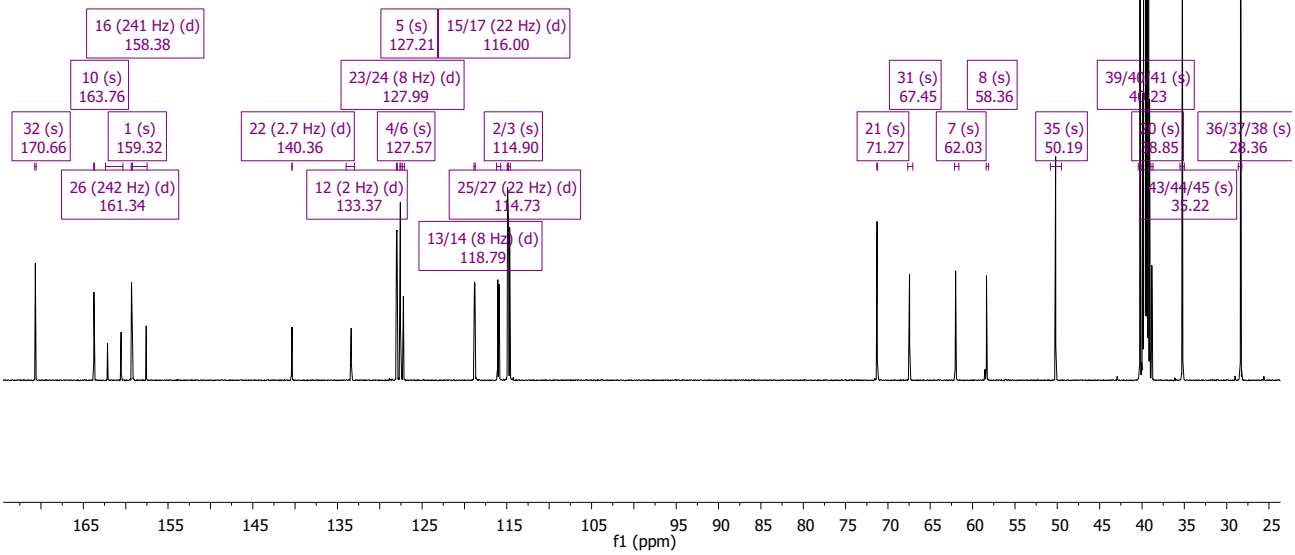
Data File Name / / samba-hpc/ nmrdata/ HREG/ h1000_1099/ h1088/ H_108817.032_h1nmr_1.fid/ fid
Owner
Solvent DMSO
Acquisition Date 2005-09-15T07:39:56
Temperature 25.0
Number of Scans 16
Filename
Sample_ID
EN
experiment h1nmr
operator
chemist
Sample# 0
prid
Date Sep 15 2005
solvent DMSO
NMR Frequency 500
probe id500
temperature 25
nt 16
site AstraZeneca R&D Mölndal
Text
Comment H_108817.032_h1nmr_1.fid
Spectrometer inova
Author
Experiment 1D
Pulse Width 0.0000
Acquisition Time 1.9976




21

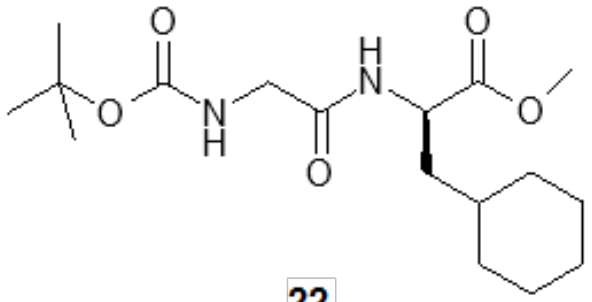
Book Reference: PharmDev
Analytical Number: SN1057248118
User: Karlsson, Staffan PO
Prid: klcc760
Frequency: 150.90MHz (nmr1)
Spinner: 5
Experiment Name: Carbon_night
#Scans: 4000
Solvent: DMSO
Parameters:
Date: Wed Jan 11 10:18:31 CET 2012
Extension: NMRhelp



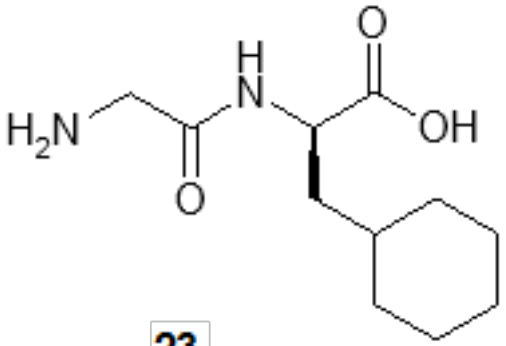

| Name | Shift | Class |
|---|---|---|
| 31 (s) | 67.45 | s |
| 16 (241 Hz) (d) | 158.38 | d |
| 32 (s) | 170.66 | s |
| 15/ 17 (22 Hz) (d) | 116 | d |
| 12 (2 Hz) (d) | 133.37 | d |
| 25/ 27 (22 Hz) (d) | 114.73 | d |
| 35 (s) | 50.19 | s |
| 26 (242 Hz) (d) | 161.34 | d |
| 10 (s) | 163.76 | s |
| 5 (s) | 127.21 | s |
| 1 (s) | 159.32 | s |
| 22 (2.7 Hz) (d) | 140.36 | d |
| 20 (s) | 38.85 | s |
| 8 (s) | 58.36 | s |
| 21 (s) | 71.27 | s |
| 43/ 44/ 45 (s) | 35.22 | s |
| 4/ 6 (s) | 127.57 | s |
| 7 (s) | 62.03 | s |
| 23/ 24 (8 Hz) (d) | 127.99 | d |
| 13/ 14 (8 Hz) (d) | 118.79 | d |
| 2/ 3 (s) | 114.9 | s |
| 39/ 40/ 41 (s) | 40.23 | s |
| 36/ 37/ 38 (s) | 28.36 | s |

Data File Name / / samba-hpc/ nmrdata/ HREG/ h1000_1099/ h1088/ H_108811.001_h1nmr_1.fid/ fid
Owner
Solvent CDCl3
Acquisition Date 2005-02-02T11:03:47
Temperature 25.0
Number of Scans 16
Filename
Sample_ID
EN
experiment h1nmr
operator
chemist
Sample# 51
prid
Date Feb 2 2005
solvent CDCl3
NMR Frequency 300
probe 4nuc300
temperature 25
nt 16
site AstraZeneca R&D Mölndal
Text
Comment H_108811.001_h1nmr_1.fid
boc-glycin-D-cyclohexylalanin-metylester
Spectrometer mercury
Author
Experiment 1D
Pulse Width 0.0000
Acquisition Time 3.0001

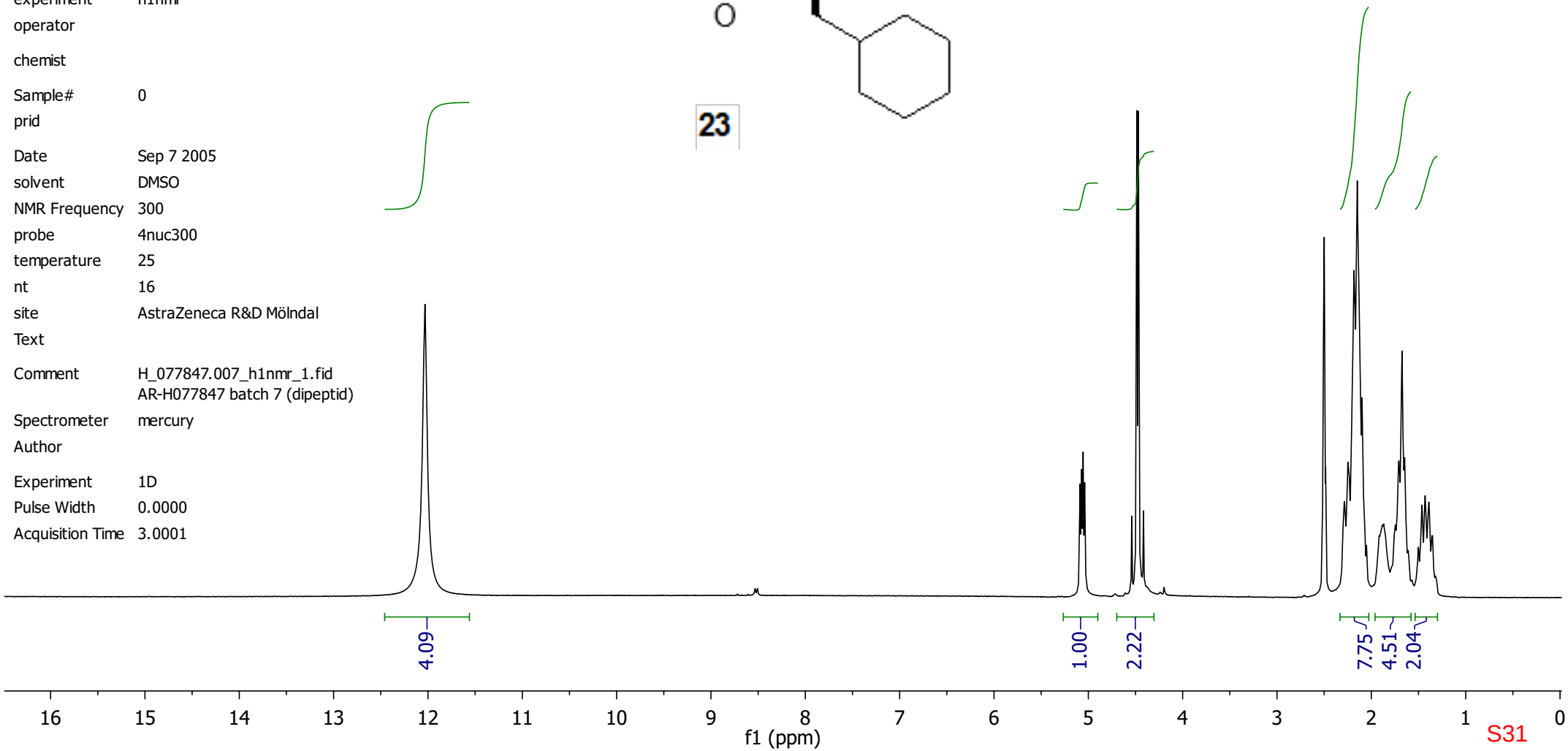



22

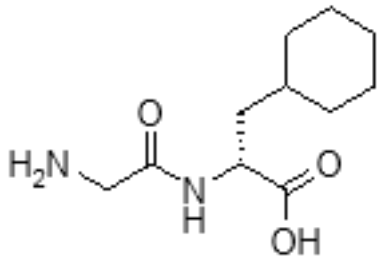
| | |
|---|---|
| Data File Name | / / samba-hpc/ nmrdata/ HREG/ h0700_0799/ h0778/ H_077847.007_h1nmr_1.fid/ fid |
| Owner | |
| Solvent | DMSO |
| Acquisition Date | 2005-09-07T16:10:44 |
| Temperature | 25.0 |
| Number of Scans | 16 |
| Filename | |
| Sample_ID | |
| EN | |
| experiment | h1nmr |
| operator | |
| chemist | |
| Sample# | 0 |
| prid | |
| Date | Sep 7 2005 |
| solvent | DMSO |
| NMR Frequency | 300 |
| probe | 4nuc300 |
| temperature | 25 |
| nt | 16 |
| site | AstraZeneca R&D Mölndal |
| Text | |
| Comment | H_077847.007_h1nmr_1.fid<br>AR-H077847 batch 7 (dipeptid) |
| Spectrometer | mercury |
| Author | |
| Experiment | 1D |
| Pulse Width | 0.0000 |
| Acquisition Time | 3.0001 |




**23**

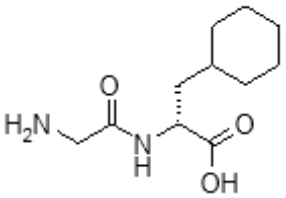


Book Reference: EN05880-03-001
Analytical Number: SN1039204675
User: Karlsson, Staffan PO
Prid: klcc760
Frequency: 125.76MHz (nmr3)
Spinner: 37
Experiment Name: Carbon_day
#Scans: 296
Solvent: Acetic
Parameters:
Date: Wed Dec 21 11:46:51 CET 2011
Extension: SN1053393552




**23**

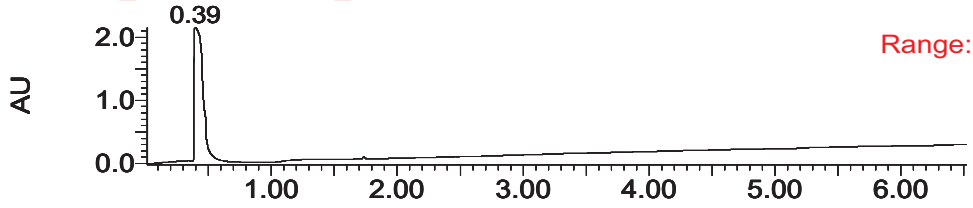
**Cars4304_SN1053393552**
Accurate mass determination
C11H20N2O3



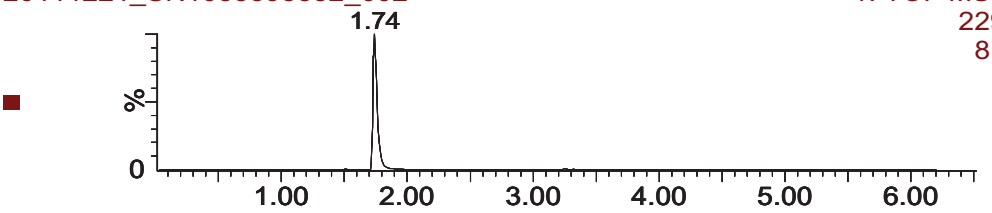
**23**

**AZ12304455** **SN1053393552**

20111221_SN1053393552_002 3: Diode Array
230
Range: 2.148




20111221_SN1053393552_002 1: TOF MS ES+
229.155
8.50e4




20111221_SN1053393552_002 1: TOF MS ES+
TIC
2.45e5