

## Supplementary Material

### The synthesis of selected phase II metabolites – *O*-glucuronides and sulfates of drug development candidates

Jens Atzrodt,\* Volker Derdau, Wolfgang Holla and Martin Sandvoss

*Sanofi-Aventis Deutschland GmbH, R&D, SCP Disposition, Safety & Animal Research (DSAR),  
Drug Disposition (DD), Isotope Chemistry & Metabolite Synthesis (ICMS), G876, 65926  
Frankfurt/Höchst, Germany.*

*E-mail: [Jens.Atzrodt@sanofi.com](mailto:Jens.Atzrodt@sanofi.com)*

**Dedicated to Rainer Beckert on the occasion of his 60<sup>th</sup> anniversary**

#### Content

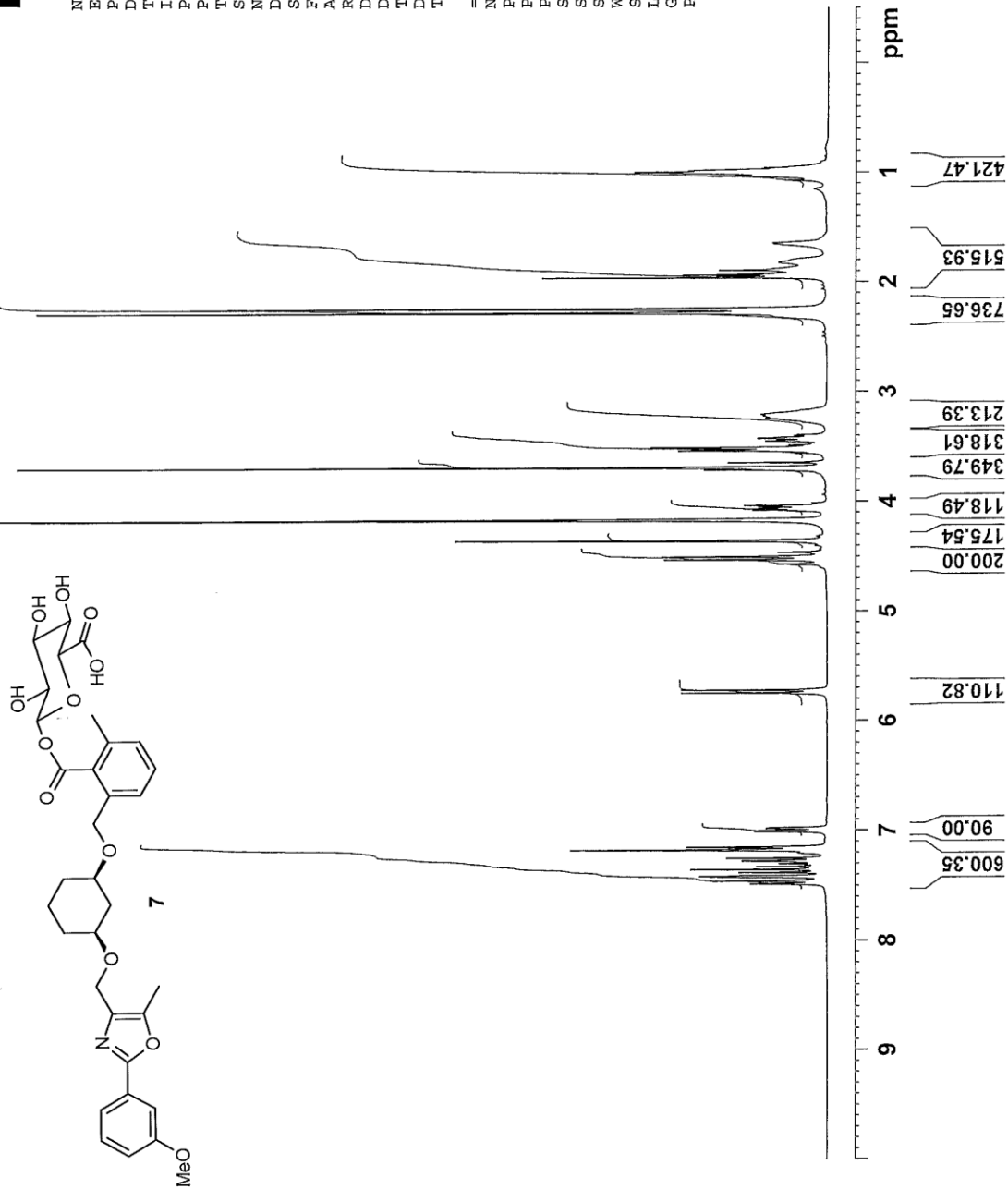
<sup>1</sup> H, <sup>13</sup> C NMR spectra and MS data for <b>7</b>	2
<sup>1</sup> H, <sup>13</sup> C NMR spectra and MS data for <b>21</b>	5
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300MHz 1H/B Dr.Az AVE0897-Acylgluc.(OMe)-13Cd3 17 mg ; ACN-d3 : D2O 1 : 1  
 proton



NAME atzi8334  
 EXPNO 30  
 PROCNO 1  
 Date\_ 20110919  
 Time\_ 14.16  
 INSTRUM spect  
 PROBD 5 mm PABBO BB-  
 PULPROG zg30  
 TD 65536  
 SOLVENT CD3CN  
 NS 128  
 DS 2  
 SWH 6188.119 Hz  
 FIDRES 0.094423 Hz  
 AQ 5.2953587 sec  
 RG 181  
 DW 80.800 usec  
 DE 6.50 usec  
 TE 300.0 K  
 D1 1.0000000 sec  
 TDO 1

==== CHANNEL f1 =====  
 NUC1 1H  
 P1 12.20 usec  
 PL1 0.00 dB  
 PL1W 11.05230045 W  
 SFO1 300.1318534 MHz  
 SI 32768  
 SF 300.1293196 MHz  
 WDW EM  
 SSB 0  
 LB 0.30 Hz  
 GB 0  
 PC 1.00



75MHz 13C/B Dr.Az AVE0897-Acylgluc. (OMe) -13Cd3 17 mg ; ACN-d3 : D2O 1 : 1  
c13cpd

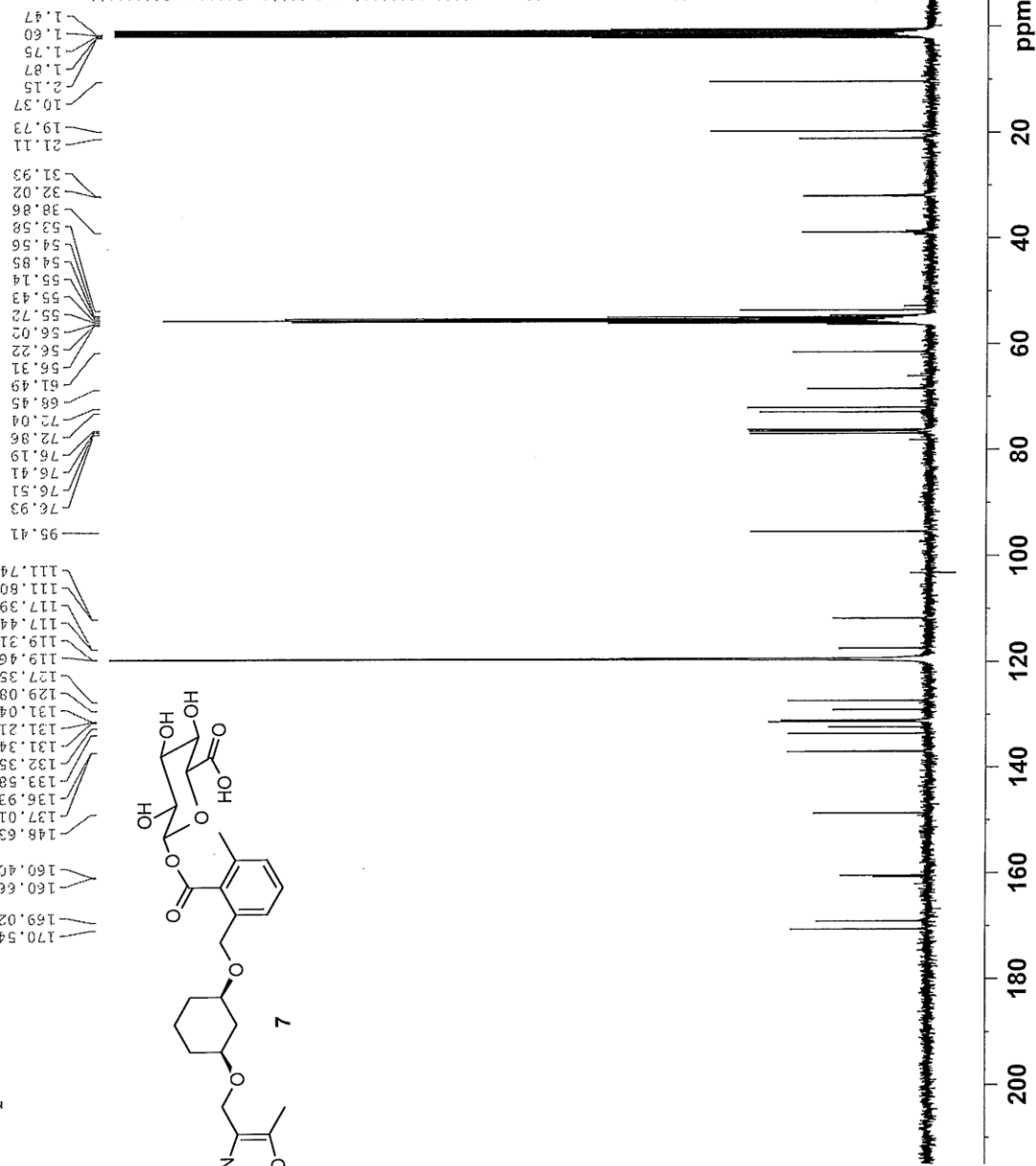


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NAME      atzr8334
EXPNO     32
PROCNO    1
Date_     20110919
Time      21.29
INSTRUM   spect
PROBHD    5 mm PABBO BB-
PULPROG   zgpg30
TD         65536
SOLVENT   CD3CN
NS         6000
DS         4
SWH        18028.846 Hz
FIDRES     0.275098 Hz
AQ         1.8175818 sec
RG         32800
DW         27.733 usec
DE         6.50 usec
TE         300.0 K
D1         2.0000000 sec
D11        0.0300000 sec
TDO        1

===== CHANNEL f1 =====
NUC1       13C
P1         8.00 usec
PL1        -2.00 dB
PL1W       54.54068375 W
SF01       75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2    waltz16
NUC2       1H
PCPD2      80.00 usec
PL2         0.00 dB
PL12       16.00 dB
PL13       16.00 dB
PL2W       11.05230045 W
PL12W      0.27762124 W
PL13W      0.27762124 W
SFO2       300.1312005 MHz
SI         32768
SF         75.4675065 MHz
WDM        EM
SSB         0
LB         0
GB         0
PC         1.40
    
```

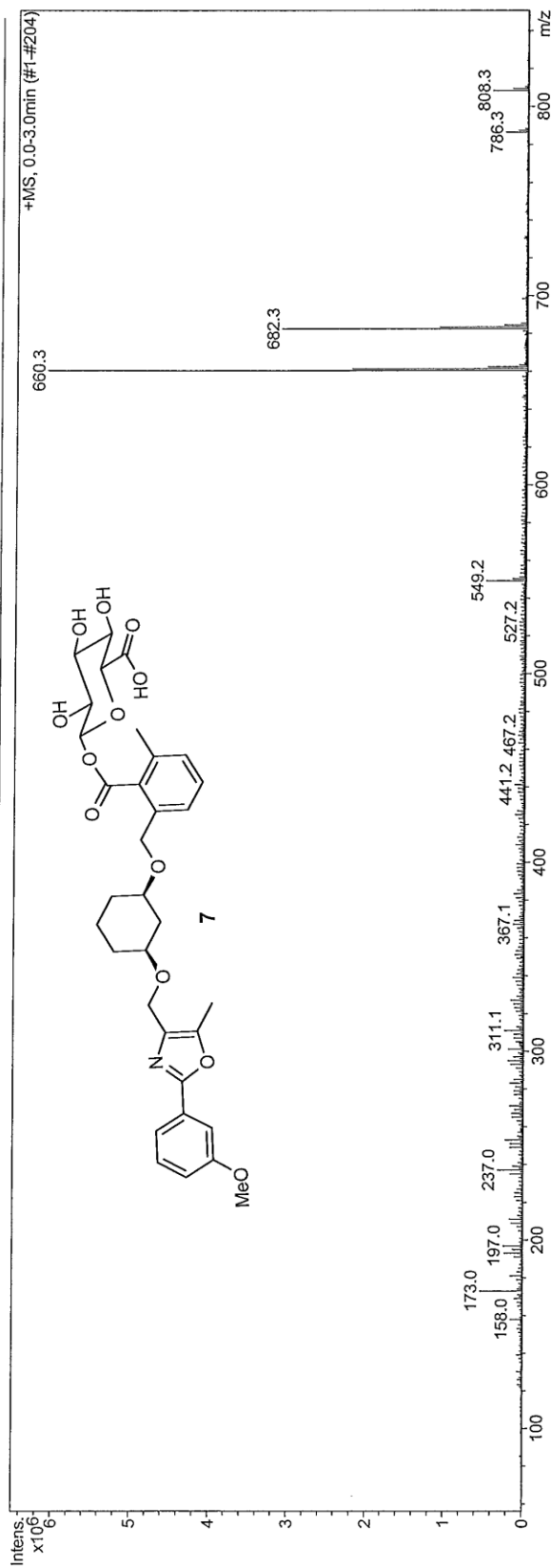


## Analysis Info

Analysis Name 13C00001.d  
 Sample Name AVE0897-gluc-OMe 13C,d3  
 Comment AVE0897-gluc-OMe 13C,d3  
 atzr8334  
 ACN : H2O 1 : 1 + HCOOH  
 Acquisition Date 09/20/11 15:42:34  
 Method Copy of 00 pos.MS  
 Administrator esquire3000  
 Operator Instrument

## MS/MS Parameters

Source/Trap Positive  
 Ion Polarity Positive  
 Scan Begin 50 m/z  
 Scan End 2000 m/z  
 ICC Target 30000

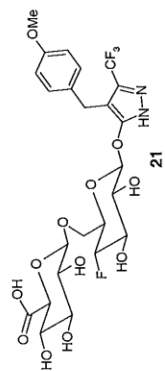


Bruker Daltonics DataAnalysis 3.0

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500MHz 1H/B gs22895 Dr. Ho SAR7226-M2\_Sm\_20/25-1 4.8mg; ACN-d3; D2O 1:1 +DC000  
zg30



```

Current Data Parameters
NAME          R0117460
EXPNO        1
PROCNO       1

F2 - Acquisition Parameters
Date_        20070326
Time         5:22
INSTRUM      spect
PROBHD       5 mm BBO BB-7H
PULPROG      zgpg30
TD           65536
SOLVENT      CD3OD
NS           128
DS           0
SWH          10330.578 Hz
FIDRES       0.157532 Hz
AQ           3.1720407 sec
RG           400
ZM           48.400 usec
DE           6.00 usec
TE           300.0 K
D1           1.50000000 sec
dPREST       0.00000000 sec
NCHRG        0.01500000 sec
***** CHANNEL f1 *****
NUC1         1H
P1           17.15 usec
PL1          0.00 dB
SFO1         500.130884 MHz

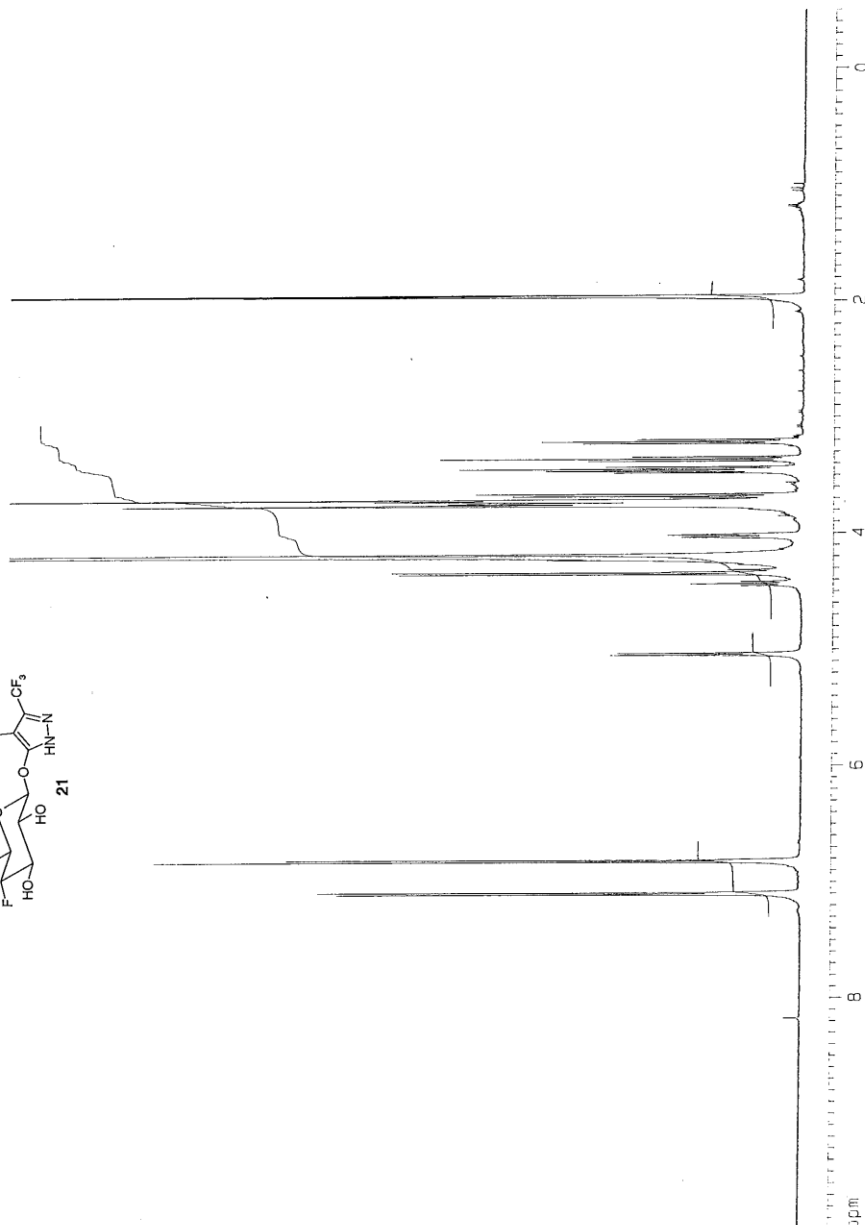
F1 - Acquisition parameters
NUC1         1H
P1           17.15 usec
PL1          0.00 dB
SFO1         500.130884 MHz

F2 - Acquisition parameters
NUC1         1H
P1           17.15 usec
PL1          0.00 dB
SFO1         500.130884 MHz

F1 - Processing parameters
SI           32768
SF           500.1099999 MHz
WDW          EM
SSB          0
LB           0.30 Hz
GB           0
PC           1.00

F1 - Processing parameters
SI           1024
SF           500.1500000 MHz
WDW          rd
SSB          0
LB           0.30 Hz
GB           0.1

1D NMR plot parameters
CX          20.00 cm
CY          20.00 cm
F1P         10.000 ppm
F1          5001.10 Hz
F2P         -0.500 ppm
F2          -250.05 ppm
PRNCHM     0.52300 ppm/Hz
          -0.77%
          250.54777 Hz/Hz
    
```



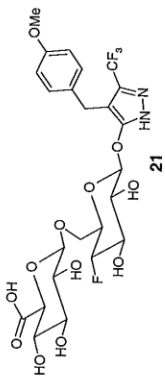
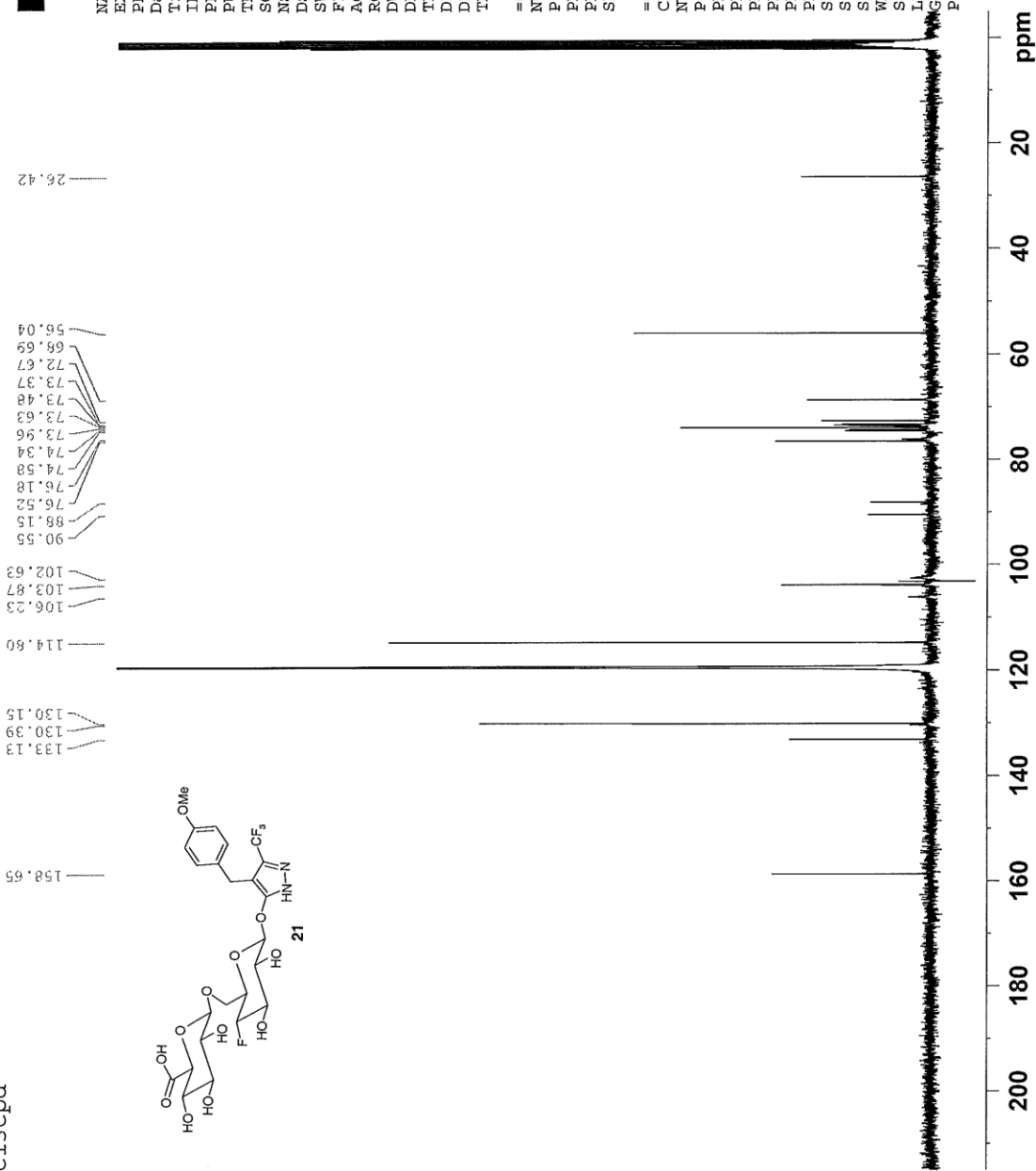
75MHz 13C/B Dr.Ho SAR7226-M2 Sm20/25-3 ; ACN-d3:D2O 1:1  
c13cpd



NAME hol17460  
EXPNO 21  
PROCNO 1  
Date 20110914  
Time 13.07  
INSTRUM spect  
PROBHD 5 mm PABBO BB-  
PULPROG zgpg30  
TD 65536  
SOLVENT CD3CN  
NS 18432  
DS 4  
SWH 18028.846 Hz  
FIDRES 0.275098 Hz  
AQ 1.8175818 sec  
RG 32800  
DW 27.733 usec  
DE 6.50 usec  
TE 300.0 K  
D1 2.0000000 sec  
D11 0.03000000 sec  
TDO 1

==== CHANNEL f1 =====  
NUC1 13C  
P1 8.00 usec  
PL1 -2.00 dB  
PL1W 54.54068375 W  
SF01 75.4752953 MHz

==== CHANNEL f2 =====  
CPDPRG2 waltz16  
NUC2 1H  
PCPD2 80.00 usec  
PL2 0.00 dB  
PL12 16.00 dB  
PL13 16.00 dB  
PL12W 11.05230045 W  
PL13W 0.27762124 W  
SFO2 300.1312005 MHz  
SI 32768  
SF 75.4675066 MHz  
WDW EM  
SSB 0  
LB 1.00 Hz  
GB 0  
PC 1.40



## Analysis Info

Analysis Name 25x10001.d  
 Sample Name SAR7226-M2 Sm 20/25-1  
 Comment SAR7226-M2 Sm 20/25-1  
 hol17460  
 ACN : H2O 1 : 1 + HCOOH

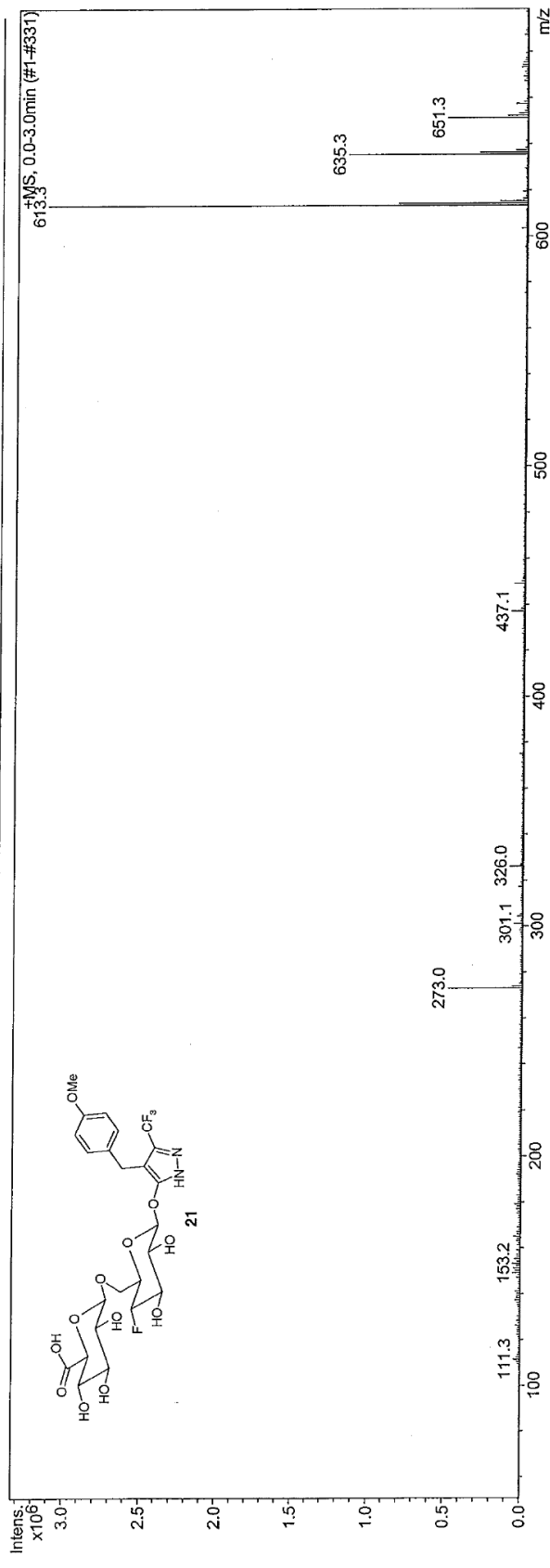
Acquisition Date 03/27/07 14:22:10  
 Method Copy of SAR7226+Fr3.MS

Operator  
 Instrument

Administrator  
 esquire3000

## MS/MS Parameters

Source/Trap Positive  
 Ion Polarity Positive  
 Scan Begin 50 m/z  
 Scan End 1000 m/z  
 ICC Target 30000

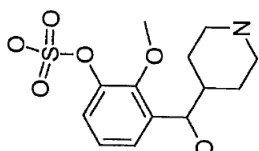


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500MHz 1H NMR 9s24833 Dr. De HDL407292 Wb 37.07 (M38007) 0.4mg. DMSO-d<sub>6</sub> 2930



33

```

Current Data Parameters
NAME      der07735
EXPNO    1
PROCNO   1

F2 - Acquisition Parameters
Date_    20080325
Time     14:48
INSTRUM  spect
PROBHD   5 mm BBO BB-1H
PULPROG  zgpg30
TD        65536
SOLVENT  DMSO
VS        1024
DS        2
SMA      10330.573 Hz
FIDRES   0.157632 Hz
AQ        3.1720007 SEC
RG        600
DM        48.400 usec
DE        6.00 usec
TE        300.0 K
D1        1.50000000 SEC
WDEXT     0.00000000 SEC
WCHRG     0.01500000 SEC

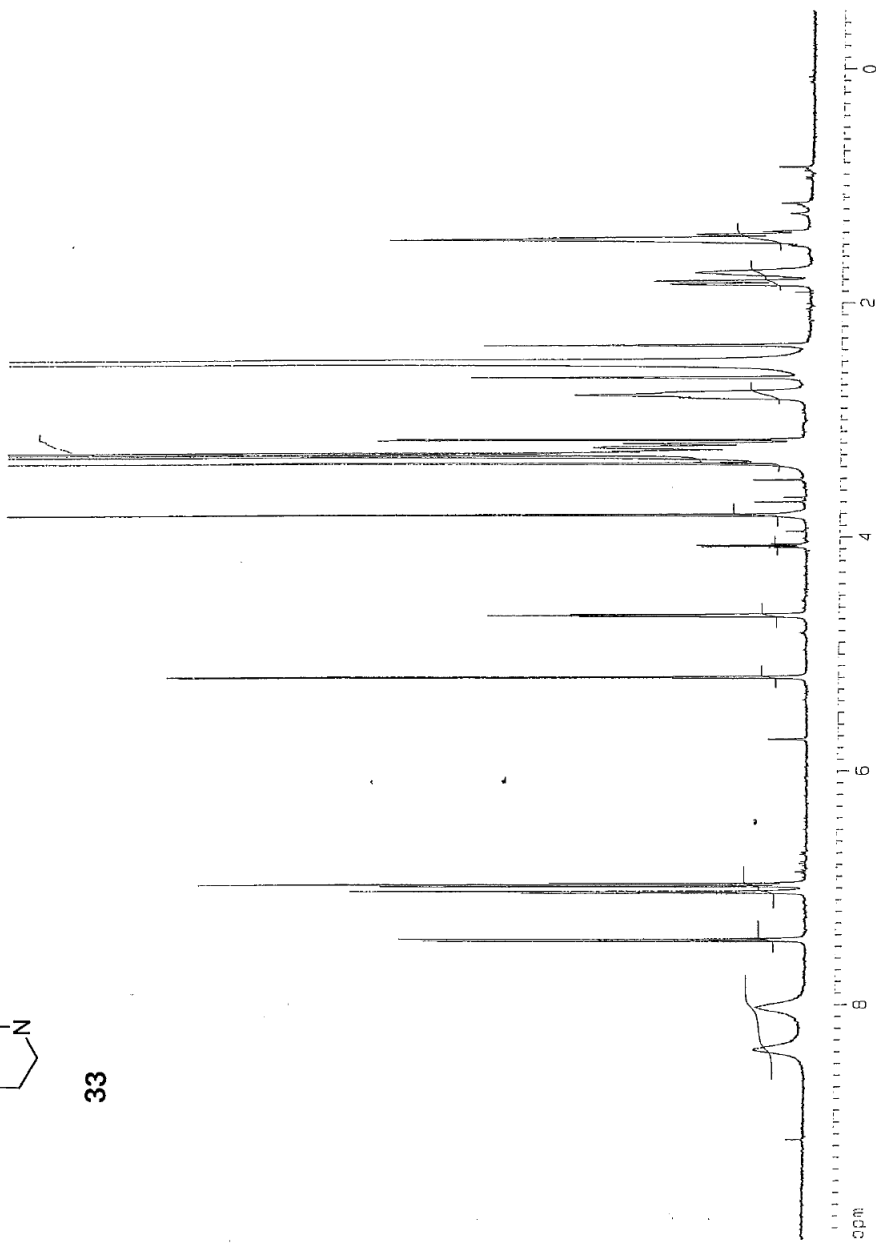
***** CHANNEL f1 *****
NUC1      1H
P1        17.15 usec
PL1       0.00 dB
SFO1      500.130884 MHz

F1 - Acquisition parameters
NUC2      13C
P2        12.00 usec
PL2       0.00 dB
SFO2      125.761170 MHz

F2 - Processing parameters
SI        32768
SF        500.1099985 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

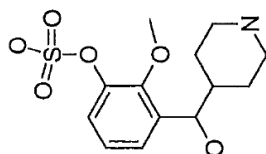
F1 - Processing parameters
SI        1024
NUC2      13C
P1        12.00 usec
PL1       0.00 dB
SFO1      125.761170 MHz
WDW       EM
SSB       0
LB        0.30 Hz
GB        0
PC        1.00

1D NMR plot parameters
CX        20.00 cm
CY        1000.00 cm
F1        10.000 ppm
F2        5001.10 Hz
ZP        -0.500 ppm
Z2        -250.05 Hz
SFOCH     0.52500 ppm/cm
AQCH      385.85777 sec/cm
  
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MDL07292 Compound 33  
c13cpd



33

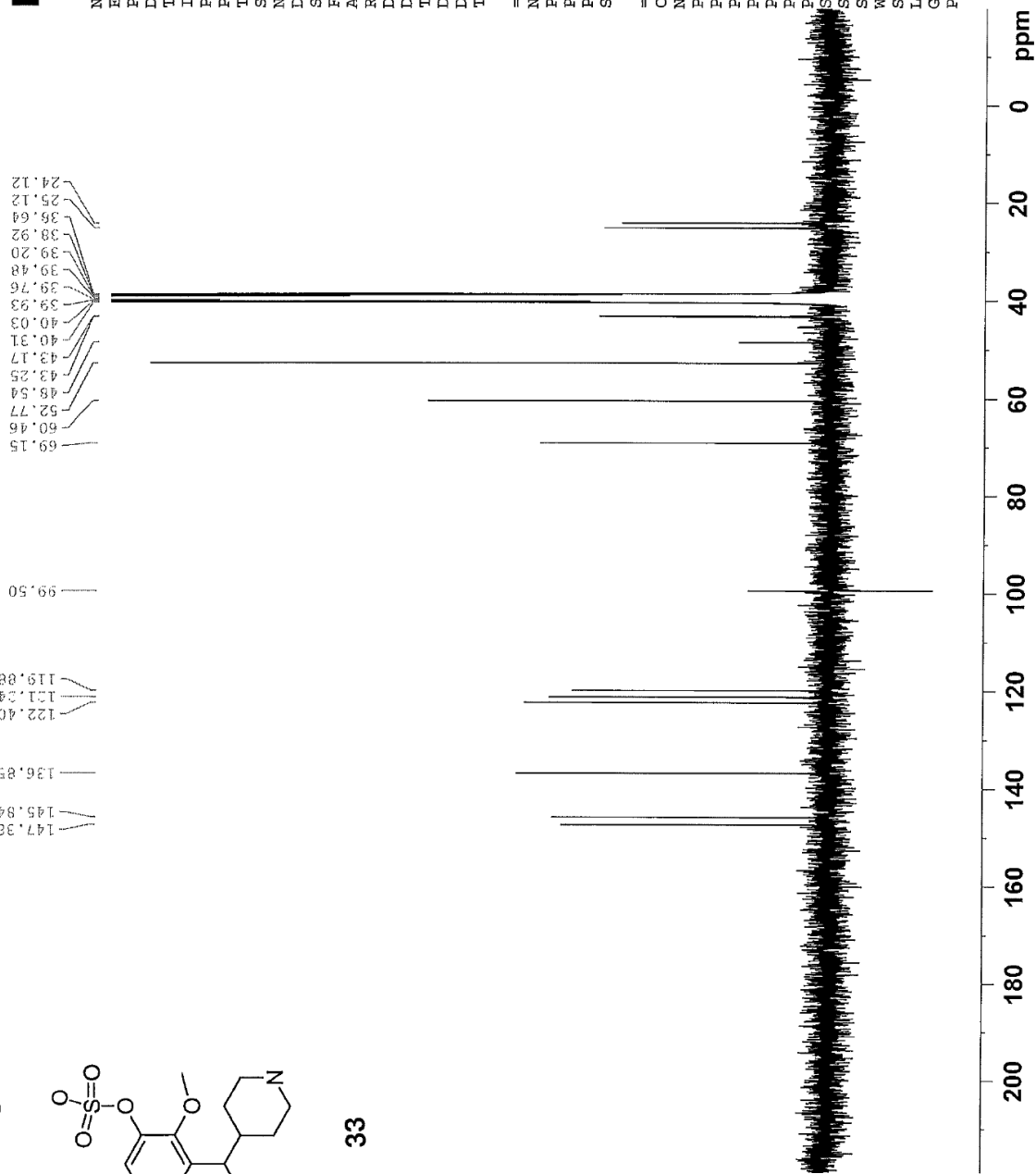


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NAME derd8345
EXPNO 11
PROCNO 1
Date_ 20111020
Time_ 16.48
INSTRUM spect
PROBED 5 mm PABBO BB-
PULPROG zgpg30
TD 65536
SOLVENT DMSO
NS 12288
DS 4
SWH 18028.846 Hz
FIDRES 0.275098 Hz
AQ 1.8175818 sec
RG 32800
DW 27.733 usec
DE 6.50 usec
TE 300.0 K
D1 2.00000000 sec
D11 0.03000000 sec
TDO 1

===== CHANNEL f1 =====
NUC1 13C
P1 8.00 usec
PL1 -2.00 dB
PL1W 54.54068375 W
SFO1 75.4752953 MHz

===== CHANNEL f2 =====
CPDPRG2 waltz16
NUC2 1H
PCPD2 80.00 usec
PL2 0.00 dB
PL12 16.00 dB
PL13 16.00 dB
PL12W 11.05230045 W
PL12W 0.27762124 W
PL13W 0.27762124 W
SFO2 300.1312005 MHz
SI 32768
SF 75.4677867 MHz
WDW EM
SSB 0
LB 1.00 Hz
GB 0
PC 1.40
    
```



**Analysis Info**

Analysis Name 37x07001.d  
 Sample Name MDL107292 M38007  
 Comment MDL107292 Wb 37107 M38007  
 derd7735  
 H2O/ACN 1:1 + HCOOH  
 Acquisition Date 03/26/08 14:04:19  
 Method Copy of MDL107292-.MS  
 Operator Administrator  
 Instrument esquire3000

**MS/MS Parameters**

Source/Trap Ion Polarity Negative  
 Scan Begin 50 m/z  
 Scan End 2000 m/z  
 ICC Target 10000

