### supplementary information

# Amino acid-linked porphyrin-nitroimidazole antibiotics targeting *Porphyromonas gingivalis*†

Simon A. Dingsdag, <sup>A,a,b</sup> Benjamin C-M. Yap, <sup>A,a,c</sup> Neil Hunter<sup>a,b</sup> and Maxwell J. Crossley<sup>\*</sup>

#### Bacteriology



Supplementary figure 1: Growth inhibitory activity of the mixture of 12a and 12b for P. gingivalis.



Supplementary figure 2: Growth inhibitory activity of the mixture of 14a and 14b for P. gingivalis.



Supplementary figure 3: Growth inhibitory activity of the mixture of 17a and 17b for *P. gingivalis*.



Supplementary figure 4: Growth inhibitory activity of the mixture of 22a and 22b for P. gingivalis.



Supplementary figure 5: Growth inhibitory activity of the mixture of 24a and 24b for P. gingivalis.



Supplementary figure 6: Growth activity of control mixture of 29a and 29b for P gingivalis.

#### **Enzyme activity studies**

The substrate lysine-para-nitroanilide (KpNA) was used to establish the activity of the lysine-specific gingipain (Kgp, 1  $\mu$ M). It was shown that 1  $\mu$ L of Kgp is able to hydrolyse 200  $\mu$ L of 0.5 mM KpNA with a velocity of 20.2 mOD/min. Kgp (1  $\mu$ L) was incubated with the DPIX-Lys(Boc)-Pro-Nim adducts **22a** and **22b** (200  $\mu$ M), with a lysine-proline preferred site of cleavage, and analysis by HPLC-MS showed the complete and successful cleavage of the lysine-proline linkage in the presence of cysteine (1  $\mu$ M, 1  $\mu$ L) after 1 h at 37 °C (**Fig. S7c**). The products of the reaction were monitored by HPLC-MS using a solvent system of CH<sub>3</sub>CN:H<sub>2</sub>O:CF<sub>3</sub>CO<sub>2</sub>H (10:90:0.5) over a gradient to CH<sub>3</sub>CN:H<sub>2</sub>O:CF<sub>3</sub>CO<sub>2</sub>H (60:40:0.5). A control experiment whereby the DPIX-Pro-Mtz adducts **24a** and **24b** were used in place of the DPIX-Lys(Boc)-Pro-Nim adducts **22a** and **22b** (200  $\mu$ M) in the presence of cysteine (1  $\mu$ M) for 30 min before incubation with DPIX-Lys(Boc)-Pro-Nim adducts **22a** and **22b** (200  $\mu$ M) in the presence of cysteine (1  $\mu$ M) for 1 h at 37 °C. HPLC-MS analysis showed no cleavage preference for a proline experiment in these adducts (**Figs. S7a and S7b**). This confirms Kgp has a cleavage preference for a proline next to a lysine and that the Kgp is responsible for the cleavage as Kgp inactivation prevented cleavage of the adducts.

These results indicate the possibility of attaching these amino-acid linked porphyrin-antibiotic adducts to antibodies which are selective to an antigen selectively expressed by the epithelial lining in disease sites (for instance CD24),<sup>14,15</sup> to form depots to increase the availability of porphyrin adduct. The localisation of adduct on epithelial cells would allow for controlled local release of porphyrin adduct for uptake by *P. gingivalis* present within and attached to epithelial cells, following Kgp-mediated cleavage of a lysine-proline linker.

![](_page_3_Figure_1.jpeg)

**Supplementary figure 7a)** LC-MS analysis of DPIX-Lys(Boc)-Pro-Nim adducts **22a** and **22b** initially and with prior inactivation of Kgp with TLCK; **b**) Mass spectrum of product in main peak of TLC chromatogram that eluted at ~23 min; **c**) LC-MS analysis of DPIX-Lys(Boc)-Pro-Nim adducts **22a** and **22b** after incubation with cysteine activated Kgp for 1 h at 37 °C.

![](_page_4_Figure_0.jpeg)

 $\begin{array}{l} Fmoc-Lys(Boc)-Met\ amine\\ Chemical\ Formula:\ C_{32}H_{40}N_6O_7\\ Molecular\ Weight:\ 620.69600 \end{array}$ 

![](_page_4_Picture_2.jpeg)

## <sup>13</sup>C NMR:

√**MS**:

Hi Res MS:

UV:

IR:

mp:

Yield:

Experimental:

![](_page_5_Figure_0.jpeg)

28/06/2007 3:49:45 PM

C:\Data\Keith\C28\_070628154945

F-M-L(BOC)-M-A

![](_page_6_Figure_0.jpeg)

ć Ë

THU THUM

CHS: 115186-31-7

R

ar un ar un ar de

L

| aine                                  | ers:<br>HHZ<br>HHZ<br>SSC<br>SSC<br>SSC<br>SSC<br>SSC<br>SSC  | usec<br>dB<br>MHz                                  | ers<br>MHz<br>Hz  | ст<br>ст<br>Ррт<br>Н2<br>Н2<br>H2/ст<br>H2/ст  |
|---------------------------------------|---|--|---|--|
| nt Data Parameters<br>FrocLysBocMetAm | Acquistion Parameti<br>20070615<br>17.1615<br>17.1615<br>17.1615<br>17.1615<br>10.122061<br>10.122266<br>10.122266<br>10.122266<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.50000000<br>11.50000000000<br>11.50000000<br>11.50000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.5000000<br>11.50000000<br>11.50000000<br>11.500000000000000000000000000000000000   | <pre>cHANNEL f1 ==== 1H 6.35 1.00 700 10007 </pre> | Processing parameter<br>16384<br>200 1300071 N<br>EM<br>0<br>0<br>10 H<br>0<br>1 00 | <pre>R plot parameters 20 00 6 40 00 6 8 800 6 1761 14 1 1761 14 1 -0 500 6 -0 500 6 93 06045 1 93 06045 1</pre> |
| Curre<br>VAME<br>EXPNO                | - 21<br>Instead<br>of the second second second second<br>of the second sec | NUCI<br>PI<br>SF01                                 |   | 1D NM<br>CX<br>CX<br>CX<br>  |

![](_page_7_Figure_2.jpeg)

wdd

8

| Current Data Parameters<br>NAME FmocLysBocMetAmine<br>EXPNO 1<br>DACCNO 1<br>PROCLYSBOCMetAmine<br>F2 - Acquisition Parameters<br>Date 17.16<br>INSTRUM spect<br>PROBHD 5 mm PHDUL 13C<br>PULPPG 5 mm PHDUL 13C<br>PULPPG 72568<br>SOLVENT 223<br>D3 S<br>SOLVENT 4006.410 Hz<br>SMH 4006.410 Hz   | A0         4.0454056         5.00         1.05         1.01           A6         1.034466         5.00         usec         5.00         usec           A6         1.24.800         usec         5.00         usec         5.00         usec           D1         1.50000000         5.00         usec         295.0 K         0.0000000         50           MCREST         0.00000000         50         0.00000000         50         14           NUC1         1.1         1.6         5.35         usec           P1         6.00         1.000000         50         50           P1         2.00         1.0007         MHZ         7         55           F0         200.1310007         MHZ         55         50         56         56           SF         200.130007         MHZ         58         5         50         55         5         5           SSB         0         0         0         0         0         5         5 | UB 0.10 HZ<br>0 0 0<br>0 1.00<br>C 1.00<br>CX 12.00 cm<br>CY 12.00 cm<br>7 12.00 cm<br>7 12.00 cm<br>7 2P 0.278 ppm<br>7 2P 0.16.16 HZ<br>7 2P 0.16.16 HZ<br>7 2P 0.1016.16 HZ<br>7 2P 0.0016.10 HZ<br>7 2P 0.0016.10 HZ<br>7 2P 0.0016.16 HZ<br>7 2P 0.0016.10 HZ<br>7 2P 0.0016.16 HZ<br>7 2P 0.0016 |
|--|---|--|
| ۲۲۲۶   |   |  |
|  |   |  |
| 65538 · 9  |   |  |
| 91892 '2<br>00972 '2<br>730311<br>71102 '2<br>72207 '2<br>72207 '2<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>90372<br>907<br>907<br>907<br>907<br>907<br>907<br>907<br>907 |   |  |
| 24500.8<br>21529.7<br>27637.7  |   |  |

3 . .

 $\frac{1}{2}$ 

1

ພປປ

0 19611 ppm/cm 37 14579 Hz/cm 4.0894966 sec 181 124 800 usec 6 00 usec 1H 6 35 usec 1 00 dB 200 1310007 MHz 1D NMH plot parameters CX 20 00 cm F1P 972 34 H2 F1 972 34 H2 F2P 272 34 H2 F2P 272 34 H2 F2P 272 34 H2 F2P 272 34 H2 F2 99PMCM 97 136 H2/Em 1.5000000 sec 0.00000000 sec 0.01500000 sec F2 - Processing parameters SI 16384 SF 200 1300071 MHz WDW EM SSB 0 10 Hz C 10 Hz CB 0 10 Pz CB 1 00 Current Data Parameters NAME FmocLysBocMetAmine EXPNO 1 ====== CHANNEL f1 ====== 4006.410 Hz 0.122266 Hz F2 - Acquisition Parameters 295 O K Date\_\_\_\_\_\_ Time PROBHD PULPROG PULPROG SULVENT NS SOLVENT NS SULVENT NS SULVENT DT DM DE DM DE DD DE DD DE DD DE DD DE PROCNO MCWRK NUC1 PL1 SF01 -1.22446 772955.1-10846.1--1.42182 Zor ÷ 72818.1---₽89¤0`2-e8571.S-------2. 47785 62828 2-----96996 2...... - <u>..</u> m ESE78.E---\_ -3.60282 57780.4--4.10350 568E1.4-78E71.4-⊅966I`⊅--4 S3186 /GILV V-----80172 2------4.71723 000 wdd

| sta Parameters<br>imocLysBocMetAmine<br>1 | <pre>isition Parameters 20070615 17.16 spect spect spect 3 mm PHOUL 13C 29 32768 32768 32768 32768 32768 4006.410 Hz 0 4006.410 Hz 0.122266 Hz 4006.410 Hz 124.800 usec 5.00 K 1.50000000 sec 0.0000000 sec 0.01500000 sec 0.001500000 sec 0.001500000 sec 0.001500000 sec 0.001500000 sec 0.001500000 sec 0.0000000 sec 0.001500000 sec 0.0015000000 sec 0.001500000000 sec 0.0015000000 sec 0.0015000000 sec 0.001500000 sec 0.001500000 sec 0.0015000000 sec 0.0015000000 sec 0.0015000000000000000000000000000000000</pre>   | HANNEL f1 ======<br>1H<br>6.35 usec<br>1.00 dB<br>200.1310007 MHz<br>ssing parameters<br>16394<br>200 1300071 MHz<br>EM<br>0<br>0<br>1.0 Hz<br>0<br>0 | t parameters<br>20.00 cm<br>10.00 cm<br>8.415 ppm<br>1684.02 HZ<br>4.944 ppm<br>989 52 HZ<br>0 17351 ppm/cm<br>34 72490 Hz/cm |
|---|--|---|---|
| Current D.<br>NAME I<br>EXHNO<br>PROCNO   | 22 - Argu<br>Date<br>Instrum<br>PROBHD<br>PROBHD<br>PROBHD<br>PROBHD<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOL | **************************************  | 10 NMR pic<br>CX<br>CY<br>71P<br>71P<br>72P<br>2PMCN<br>42CM  |

69638.8-----

22124 S-----

 2400.8

 21529.7

 21529.7

 21529.7

 21529.7

 21529.7

wdd

![](_page_10_Figure_4.jpeg)

(eut-ru) D

11

H

------

![](_page_11_Figure_0.jpeg)

| 10/ 66 42 |
|-----------|
|-----------|

![](_page_12_Figure_1.jpeg)

| Current Data Parameters<br>NAME FmocLysBocMetAmine<br>EXPNO 1<br>PROCNO 1  | F2 - Acquisition Parameters<br>Date20070615<br>Time17.16<br>INSTRUM spect<br>PROBHD 5 mm PHDUL_13C<br>PULRPNG 5 mm PHDUL_13C<br>PULRPNG 29<br>TD 232768<br>SOLVENT CDC13 | NS 32<br>DS 32<br>DS 0<br>SWH 4006.410 Hz<br>FIDRES 0.122266 Hz<br>A0 4.089466 sec<br>RG 12.12266 Hz<br>A0 122266 Hz<br>A0 122265 Hz<br>A0 122265 Hz<br>A0 122256 Hz<br>DW 1225 0 Usec<br>DE 6.00 Usec<br>D1 1.5000000 sec  | MCREST         0.0000000 sec           MCWRK         0.01500000 sec           ======         CHANNEL f1 ========           =====         CHANNEL f1 ========           ====         CHANNEL f1 ========           ====         CHANNEL f1 =======           NUC1         11           P1         6         35 usec           PL1         200.1310007 MHz | F2 - Processing parameters<br>SI 16384<br>SF 200.1300071 MH2<br>WDW EM EM<br>SSB 0<br>LB 0<br>LB 0<br>PC 10 H2<br>PC 1 00 | CX         C3         C0         Cm           CY         20         00         cm           CY         10         00         cm           F1P         5         085         ppm           F1         101         62         Hz           F2P         0         05         ppm           F2P         0         65         ppm           PNICM         6         22145         ppm/cm | HZCM 44 35665 Nizion                         |
|--|--|---|--|---|---|--|
| 5815A [1-<br>2625]<br>772957]<br>77265<br>77262<br>77264<br>77254<br>7<br>77254<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7<br>7 |  | ting to<br>start the second secon |  | 2<br>2<br>2<br>2<br>3   |   | 7659 (                                       |
| 08571.5-<br>⊳88⊳0.5-<br>78818.1-   |  |   |  | 2000<br>2000<br>2000<br>2000<br>2000<br>2000<br>2000<br>200   |   | 9222 - 2<br>9292 - 2<br>9292 - 2<br>9657 - 6 |
| 28774.5-   |  |   |  |   |   |  |
| 62828 2-<br>96996 2-<br>81920 8-<br>90718-<br>90718-   |  |   | ~  |   |   | 1907 ()<br>0/31 ()<br>7 200 ()<br>10         |
|  |  |   |  |   |   | <u>E155 3</u> a                              |
| -4,19954<br>20861.4-<br>03201.4  |  |   |  |   | Marth   |  |
| 29117 7-<br>Heirr 7-   |  |   |  |   |   |  |
| wdd  |  |   |  |   | рарост-<br>   | (e.dero) — =                                 |

![](_page_14_Figure_1.jpeg)

 $\begin{array}{l} {\sf Fmoc-Lys(Met\ acid)-Met\ amine}\\ {\sf Chemical\ Formula:\ C_{33}H_{37}N_9O_8}\\ {\sf Molecular\ Weight.\ 687.70238} \end{array}$ 

![](_page_14_Picture_3.jpeg)

<sup>13</sup>C NMR:

MS:

Hi Res MS:

UV:

IR:

mp:

Yield:

Experimental:

![](_page_15_Figure_0.jpeg)

![](_page_16_Figure_0.jpeg)

.

| Fmoc-Lys(Netacid)-Metamine<br>1 1<br>40 Acquisition Parameters<br>200725 | 10.32<br>10.5 mm PHDuL 13C<br>10.5 mm PHDuL 13C<br>29<br>10.5 16.384<br>10.00C13<br>22<br>20<br>22<br>22<br>22<br>22<br>22<br>22<br>22<br>22 | 2.044731 sec<br>161.3<br>161.3<br>164.800 usec<br>6.00 usec<br>6.00 usec<br>1.5000000 sec<br>1.0000000 sec<br>6 0.01500000 sec   | CHANNEL f1<br>1H<br>6.35 usec<br>1.00 dB<br>200.1310007 MH2 | Processing parameters<br>16384<br>200.1300075 MH2<br>EN<br>0<br>0.10 H2<br>0<br>1.00  | <pre>49 plot parameters<br/>20.00 cm<br/>12.000 cm<br/>5.966 ppm<br/>1197.33 H2<br/>2.776 ppm<br/>5.52 A2<br/>5.62 A2<br/>9.1551 H2/cm<br/>32.11551 H2/cm</pre> |                      |               |
|--|--|--|---|---|---|----------------------|---------------|
| NAME<br>EXPNO<br>PROCNO<br>F2 - J  | 1 JME<br>INSTRI<br>PHOBHG<br>PHOBHG<br>PULPRO<br>PULPRO<br>SOLVEP<br>SWH<br>SSWH<br>SSWH<br>FIDRE  | A0<br>A0<br>A0<br>AC<br>AC<br>AC<br>AC<br>AC<br>AC<br>AC<br>AC<br>AC<br>AC<br>AC<br>AC<br>AC   | NUC1<br>PL1<br>PL1<br>SF01                                  | F SS<br>NOW<br>SSI<br>SSB<br>F C<br>SSB<br>F C<br>SSB<br>F C<br>SSB<br>F C<br>SSB<br>F C<br>SSB<br>F C<br>SSB<br>F C<br>SSB<br>F C<br>SSB<br>F C<br>SS<br>F C<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS | 10 NM<br>C X<br>C X<br>C Y<br>F 1<br>F 2<br>F<br>F 2<br>F<br>F 2<br>F<br>F 2<br>F<br>F 2<br>F<br>F 2<br>F<br>F 2<br>F<br>F 2<br>F<br>F 2<br>F<br>F 2<br>F 2     |                      |               |
|  |  |  |   |   |   |                      |               |
|  |  |  |   |   |   | J                    |               |
|  |  |  |   |   |   | $\overline{\langle}$ | BIEP          |
| 82782.5  |  | NY WERE DISCONSISTENT OF THE SECOND SEC |   |   |   |                      |               |
|  |  |  |   |   |   | $\langle \rangle$    | 2019          |
|  |  |  |   |   |   |                      |               |
|  |  |  |   |   |   |                      | 0015          |
| 4.21488  |  |  |   |   |   | X                    | 1067          |
| 10014.4  |  |  |   |   | ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~   |                      | E438.         |
| 1901   |  |  |   |   |   |                      |               |
|  |  |  |   |   | N   |                      | $\overline{}$ |
| 4.84236  |  |  |   |   |   |                      | ₽SSE          |
|  |  |  |   |   |   |                      |               |
|  |  |  |   |   |   |                      |               |
|  |  |  |   |   |   |                      | _             |
| 9  |  |  |   |   |   |                      | 9711          |
|  |  |  |   |   |   | 1                    |               |
| wdd  |  |  |   |   |   |                      | (enge)        |

| ld)-Metamin                             | .ers<br>Hz<br>sec  | uusec<br>vusec<br>sec<br>vuses<br>dbsc<br>mH2<br>MH2  | rrs<br>MHz<br>Hz   | cm<br>cm<br>ppm<br>ppm<br>Hz<br>Hz/cm<br>Hz/cm   |
|---|--|---|--|--|
| Data Parameters<br>Fmoc-Lys (metac<br>1 | auisition Parame'<br>20070725<br>15.32<br>15.32<br>spect<br>25<br>26<br>1634<br>1634<br>2013<br>22<br>2444732<br>2444732<br>2444732<br>2613<br>22                          | 124.800<br>6.00<br>6.00<br>300.4<br>300.4<br>300.4<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.01500000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.0150000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.015000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.015000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.015000000<br>0.0150000000<br>0.015000000000<br>0.0150000000<br>0.0150000000<br>0.015000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.015000000<br>0.015000000<br>0.0150000000<br>0.015000000<br>0.015000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000<br>0.0150000000000 | DC essing paramete<br>16384<br>16384<br>16384<br>200.1300075<br>200.1300076<br>0<br>0.10<br>0.10 | 2010t parameters<br>20.00<br>12.00<br>2035<br>2035<br>2035<br>557.65<br>-0.335<br>-78.66<br>0.16148<br>0.16148 |
| Current<br>NAME<br>EXPNO<br>PROCNO      | F2 - AC<br>Date<br>Time<br>INSTRUM<br>PROBHD<br>PULPROG<br>TD<br>SULPROG<br>SOLVENT<br>NS<br>SOLVENT<br>SWH<br>SWH<br>SWH<br>SWH<br>SWH<br>SWH<br>SWH<br>SWH<br>SWH<br>SWH | DW<br>DE<br>D1<br>MCREST<br>MCREST<br>MCREST<br>NUCH<br>NUC1<br>P1<br>P1<br>SF01<br>SF01  | - 55<br>- 72<br>- 72<br>- 72<br>- 72<br>- 72<br>- 72<br>- 72<br>- 72                             | 10 NMR<br>CCY<br>CY<br>F1<br>F2<br>PPMCM<br>HZCM<br>HZCM   |

00000 0------

61

85416.0-----

| STELLI      |
|-------------|
| 78Eb1.1-    |
| E1881.1-//- |
| 1.19837     |
| E8815.1     |
| EAES.1-     |
| 87852.1-    |
| -1.26669    |
| 1.38214     |
| 52262.1     |
| 95025.1     |
| 28113.1     |
| 96059.1     |
|             |

9965.4

1.1

Land Land

\_\_\_\_\_C

1.0028

6555.4

. .

វេ

L

-

> с Ч

tenpesni\_\_\_\_\_c E

6829.6

wdd

15524.5-

11a and 11b.

![](_page_19_Figure_1.jpeg)

![](_page_20_Figure_0.jpeg)

![](_page_21_Figure_0.jpeg)

![](_page_22_Figure_0.jpeg)

| Current Data Parameters<br>NAME BCMY-03-65monoLysmonoMe<br>EXPNO 1<br>PPOCNO 1<br>PPOCNO 1<br>PPOCNO 1<br>PPOCNO 2<br>Date 20070917<br>Time 20.47<br>Inme 20.47<br>Inme 20.47<br>Inme 20.47<br>Inme 20.47<br>Inme 20.47<br>Inme 20.47<br>PULPPOG 29<br>DULPPOG 29<br>SOLVENT COC13<br>NS SOLVENT COC13<br>NS SOLVENT COC13<br>NS SOLVENT COC13<br>NS SOLVENT COC13 | FIDRES         0.122266 Hz           AG         0.122266 Hz           AG         1.22266 Hz           AG         1.22266 Hz           AG         1.22266 Hz           AG         1.23266 Hz           AG         1.23266 Hz           AG         1.23266 Hz           AG         1.23200 Hz           AG         1.2300 usec           BG         0.0000000 sec           MCHK         0.01500000 sec           MUC1         1.4           P1         6.36 usec | SF01         200.1308005 MHz           F2         - Processing parameters           SI         15384           SF         200.1300083 MHz           MDW         EM           MDW         EM           MDW         EM           MDW         EM           MDW         EM           MDW         EM           CB         0           CB         0           CB         0           CB         0           CB         0           CC         100           CC         20.00 cm           CY         10           F1P         2020 65 Hz           F2P         7 983 ppm           F2         1597 56 Hz | РРАСМ 0.10570 рлл/ст<br> |
|--|---|--|--------------------------|
|  |   |  |                          |
|  |   |  |                          |
| 05828.8  |   |  |                          |
|  |   |  |                          |
| 15484.9  |   |  |                          |
| 01768.9<br>87762.9<br>87762.9<br>87762.9<br>87762.9<br>9<br>87762.9<br>9<br>9<br>7<br>8<br>7<br>8<br>7<br>8<br>7<br>8<br>7<br>8<br>7<br>8<br>7<br>8<br>7<br>8<br>7   |   |  |                          |
| waq  |   |  | [6709171] (<br>- e<br>   |

1D NMR plot parameters CX 20 00 cm F1P 8.135 ppm F1 1628 16 Hz 6.276 ppm F2P 0.09296 ppm/cm HZCM 18.60483 Hz/cm Current Data Parameters NAME BCMY-03-65monolysmonoMe EXPNO 1 PAOCNO 1 71.8 71.8 6.00 usec 300.6 K 1.5000000 sec 0.0000000 sec 0.01500000 sec F2 - Acquisition Parameters Date\_\_\_\_\_\_20070917 Time 20070917 INSTRUM spect PROBHD 5 mm PHDUL 13C PULPROG 29 DULPROG 29 SOLVENT 2013 SOLVENT 2013 SOLVENT 20123 SOLVENT 2012266 Hz A0 4006.410 Hz FIDRES 0.122266 Hz A0 1224.600 usec BG 124.600 usec BG 124.600 usec BG 124.600 usec BC 6.00 usec BC 201 1.5000000 sec MCREST 0.0150000 sec 1H 6.35 usec 1.00 dB 200.1308005 MHz F2 - Processing parameters SI 16384 SF 200.1300083 MHz WDW EM SSB 0.10 H2 CB 0.10 H2 CB 0.10 H2 CB 0.10 H2 16384 200.1300083 MHz EM 0.10 Hz 1.00 8054 0 97548.8-11610.9-0 4502 r ſ 6909S.7-, t Ę r í Ŧ 98818.7----[690920[ wdd

| ,y smonoMe                              | ທ<br>ເ<br>ຍ  | Hz<br>Hz<br>sec<br>usec<br>K<br>Sec   | sec<br>sec<br>usec<br>MHz  | rrs<br>Hz  | cm<br>cm<br>h2<br>h2<br>h2<br>h2<br>cm<br>h2/cm<br>h2/cm                                     |
|---|--|---|--|--|--|
| a Parameters<br>4Y-03-65monol<br>1      | 11100 Paramet<br>20070917<br>20.47<br>20.47<br>spect<br>nm PHDUL 13C | 32768<br>CDC13<br>32<br>32<br>4006.410<br>0.122266<br>4.0894966<br>4.0894966<br>1.18<br>124.800<br>124.800<br>124.800<br>300.6<br>1<br>50000000 | 0.0000000<br>0.01500000<br>0.01500000<br>1H<br>6.35<br>6.35<br>1.00<br>1.00<br>200.1308005 | aing paramete<br>16384<br>200.1300083<br>EM<br>0<br>0<br>0.10<br>0<br>1.00 | parameters<br>20.00<br>10.00<br>6.429<br>1286.65<br>4.493<br>8.99226<br>8992678<br>0.0992678 |
| urrent Date<br>AME BCA<br>XPNO<br>ROCNO | 2 - Acquis:<br>ate   | 0<br>S<br>S<br>MW<br>MH<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G<br>G                               | CHESI<br>CWRK<br>L<br>L<br>L<br>F01<br>F01   | , Process<br>DT<br>SB<br>BB<br>BB<br>CC<br>BB                              | D NMR plot<br>X X<br>1P<br>2P<br>2P<br>2CM<br>ZCM  |

![](_page_25_Figure_1.jpeg)

U .

α ~

α ---υ

 $\subset$ 

ά

0 ų

6760.0 **2650:0** 0 0005

2900 0

0.0223

(enterni \_ E

÷

£

| 12078.2    |  |
|------------|--|
| 2×9296 - 2 |  |
| 62910.3    |  |

97286.4-

6E0E1'9-

62681 · S---

05907 '9----

12002.2---

75252----67285 . 67749

vEEG1.9-----

wdd

÷ -

| LysmanoM                                 | ters<br>HA2<br>Sec<br>Uusec<br>Sec  | sec<br>usec<br>MHz<br>MHz<br>HZ<br>HZ   | cm<br>cm<br>ppm<br>Hz<br>hz<br>Hz<br>Hz/cm<br>Hz/cm   |
|--|---|---|---|
| ta Parameters<br>CMY-03-65mono<br>1<br>1 | sittion Parame<br>20070917<br>20147<br>20141 150<br>20141 150<br>201410<br>201410<br>201512266<br>41006.410<br>4006.410<br>4006.410<br>124.800<br>122266<br>41.084366<br>41.084366<br>41.084360<br>200000000<br>0.000000000   | 2.01200000<br>144NVEL f1 ====<br>6.35<br>1.00<br>200.1308005<br>1.308061<br>15384<br>200.130806<br>0<br>0<br>0<br>1.00                          | : parameters<br>20 00<br>10.00<br>4 546<br>929.84<br>2 889<br>2 899<br>2 893<br>2 893<br>17.58539<br>17.58539 |
| Current Da<br>NAME B<br>EXPNO<br>PROCNO  | 52 - Acquu<br>Date<br>I Ime<br>PROBHDM<br>5<br>PHUPAGG<br>7D<br>PULPAGG<br>7D<br>NS<br>SCUVEN<br>7D<br>NS<br>SCUVEN<br>7<br>10<br>8<br>8<br>40<br>0<br>7<br>10<br>7<br>10<br>7<br>10<br>8<br>10<br>10<br>10<br>10<br>10<br>10<br>10<br>10<br>10<br>10<br>10<br>10<br>10 | NUCI<br>SF01<br>P1<br>P1<br>P1<br>P1<br>SF01<br>SF01<br>SF2<br>SF<br>S5<br>S5<br>S5<br>S5<br>S5<br>S5<br>S5<br>S5<br>S5<br>S5<br>S5<br>S5<br>S5 | 10 NMA plot<br>CC<br>C7<br>F1<br>F7<br>PPMCM<br>H2CM  |

![](_page_26_Figure_1.jpeg)

∪ ₽

 $\subset P$ 

A A

lengezni 0 E

7.257 S

4.15088

~ .

wdd

**د** م

| Current Data Parameters<br>NAME BENV-03-EEmocol VERNOVA | NAME DUMITOJEDMUNOLYSMUNOME<br>EXPNO 1<br>DANCAN 1 | •      | F2 - Acquisition Parameters<br>Date20070917 | Time 20.47 | PROBHD 5 mm PHDUL 13C | 5Z 5G | 10 32768<br>SALVENT COC13 | NS 32 | 0 SC | SWH 4006.410 Hz | ► 1UHES 0.122266 H2<br>AD A DBAAGES SEC | RG 4.0034300 SEL | DW 124.800 usec   | DE 6.00 usec | TE 300.6 K | D1 1.5000000 sec | MCWRK 0.01500000 sec | restrate CHANNEL 11 arerest | NUC1 1H | P1 6.35 usec | PL1 1.00 dB | SFUI CUUBULIJUUS | 50 - Drorassino nanamatars | SI 16384 | SF 200.1300083 MHz | WDW EM | SSB | LB 0.10 Hz |   | PC 1 00 | 10 NMR plot parameters | CX 20.00 Cm | F1P 2 991 00m | F1 539.52 Hz | F2P 1.335 ppm | F2 267.21 H2 | HZCM 0 00676 ppm/cm<br>HZCM 16.56594 HZ/Cm |           |                              |   |   |               |     |          |            |         |            |  |
|---|--|--------|---|------------|-----------------------|-------|---------------------------|-------|------|-----------------|---|------------------|---|--------------|------------|------------------|----------------------|-----------------------------|---------|--------------|-------------|------------------|----------------------------|----------|--------------------|--------|-----|------------|---|---------|------------------------|-------------|---------------|--------------|---------------|--------------|--|-----------|------------------------------|---|---|---------------|-----|----------|------------|---------|------------|--|
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      | <br>                        |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              |   |   |               |     |          |            | -       |            |  |
|   | ₽96I   | \$`I   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      | <br>                        |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        | _           | $\geq$        | 2            |               |              |  |           |                              |   |   |               | >   | IG       | 82         | 0       |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           | <u> </u>                     |   |   |               |     |          |            | **      |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              | }                                       | 2 |               |     |          |            | ~       |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              | 3                                       |   |               |     |          |            |         |            |  |
|   | 2962   | a : t- |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  | _         | ~                            | ſ                                       |   |               |     |          |            |         | _ u<br>_ • |  |
|   | 0000   |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  | 2         |                              | $\overline{\}$                          |   |               |     |          |            | ~       |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        | ,           |               |              |               |              |  |           |                              |   |   |               |     |          |            | <i></i> |            |  |
|   | 775E   | 7.1-   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      | <br>                        |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               | ~            |               |              |  |           | _                            |   |   |               |     | 968      | 2          | 0       |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              | 7                                       |   |               |     |          |            |         | .α         |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              | 1                                       |   |               |     |          |            |         | *          |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              | 5                                       |   |               |     |          |            |         |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              | ~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~~ |   |               |     |          |            | -       |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              | Ţ                                       |   |               |     |          |            | **      | ~          |  |
|   | 5181   | -5:0   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           | $\langle$                    | )                                       |   |               |     |          |            |         | - c        |  |
|   | ¢909   | -5.0   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           | $\langle$                    |   |   |               |     |          |            | -       |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           | ]                            | }                                       |   |               |     |          |            |         |            |  |
|   | 2095   | 1.S-   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              | ~  | $\langle$ | <                            | r                                       |   |               |     | 190      | e Li i     | ō-      |            |  |
|   | 69108  | 5.5-   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               | -            |               |              |  | $\geq$    |                              |   | - | · · · · · ·   |     | 628      | 1          |         | 0          |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  | ~         | Y.                           |   | - |               |     |          |            | -       | ()         |  |
|   | 18179  | -5`5-  |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           | $\left\langle \right\rangle$ |   |   |               |     |          |            |         |            |  |
| 1   | 66528  | -5.3   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  | V         | $\leq$                       |   |   |               |     |          |            |         |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           | {                            |   |   |               |     |          |            | -       | V          |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              | _  |           |                              |   |   |               |     |          |            |         | 'n         |  |
|   | 0006+  |        |   |            |                       |       |                           |       |      |                 |   | 1                | _   | _            |            |                  | _                    |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        | /           |               |              |               |              |  |           |                              |   | - | $\overline{}$ |     |          |            | -       |            |  |
| ţ   | 99619  | 5.S-   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  | <                    | <br>                        |         | >            | -           | $\geq$           | 2                          |          |                    |        |     |            |   |         |                        |             |               |              |               |              |  |           |                              |   |   |               |     | 990      | Z          | ī       |            |  |
| ç   | 91209  | S      |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  | 2                          |          |                    | ····   |     |            |   |         |                        |             |               |              |               |              |  |           |                              |   |   | /             | /   |          |            |         |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             | ~~            | -            |               |              | `  |           |                              |   | ~ |               |     |          |            |         | ເ          |  |
|   | 21999  | -5.1   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            | - |         |                        |             |               |              |               |              | \$   |           |                              |   |   |               |     |          |            | -       |            |  |
| 4   | 44269  | ).S-   |   |            |                       |       |                           |       |      |                 |   | -                | in the second |              |            | <u> </u>         |                      | <br>                        |         |              |             |                  |                            |          |                    |        |     |            |   |         | >-<                    | 2           |               |              |               |              |  |           |                              |   |   |               |     | 568      | <u>ç (</u> | 5       |            |  |
| ç   | 96627  | . S-   |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      | <br>                        |         |              | ~~~~~       |                  |                            |          |                    |        |     | >          |   |         |                        |             |               |              |               |              |  |           | _                            |   | - | ~~            | /~/ | 50E      | 7.1        | 2       |            |  |
| 6   | 29992<br>2696/                                     | -5     |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          | <                  | _      | 2   |            |   | ~       |                        |             |               |              |               |              |  | ~         |                              |   |   |               |     |          |            |         | α          |  |
| c   |  |        |   | -          |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   | ς       | ~                      |             |               | _            |               |              |  |           |                              |   |   |               |     |          |            |         | n,         |  |
| Ĺ   | 15238  | -51    |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               | 2            | _             | _            |  |           |                              |   |   |               |     |          |            | -       |            |  |
|   |  |        |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              | S  | 2         |                              |   |   |               |     |          |            |         |            |  |
|   |  | d      |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               |              | J  |           |                              |   |   |               |     |          |            |         | шc         |  |
|   | ωu   | 4      |   |            |                       |       |                           |       |      |                 |   |                  |   |              |            |                  |                      |                             |         |              |             |                  |                            |          |                    |        |     |            |   |         |                        |             |               |              |               | ~            | 1  |           |                              |   |   |               |     | (ខ្មុរព័ | ala        | 1       | č          |  |

6.^ 6.

د ۲

| Current D<br>VAME | ata Parameters<br>BCMY-03-65monoL    | ysmonoMe         |
|-------------------|--------------------------------------|------------------|
| EXPNO<br>PROCNO   | *** ***                              |                  |
| ≈2 - Acqu<br>Date | isıtıon Paramet<br>20070917<br>20.47 | ers              |
| INSTRUM<br>PROBHD | 5 mm PHDUL 13C                       |                  |
| PULPROG<br>TD     | 2g<br>32768                          |                  |
| SOL VENT          | CDC13                                |                  |
| SN SC             | 32                                   |                  |
| SWH               | 4006 410                             | H2               |
| FIORES            | 0.122266                             | Hz               |
| A()               | 4.0894966<br>71 A                    | sec              |
| MO                | 124.800                              | usec             |
| ЭС                | 6.00                                 | usec             |
| 巴 :               | 300.6                                | ×                |
| 01<br>            | 1.5000000                            | Sec              |
| MCWRK             | 0.0150000                            | sec              |
| *******           | CHANNEL f1 ====                      | n<br>H<br>H<br>H |
| NUCI              | 11                                   |                  |
| P1                | 6.35                                 | nsec             |
| PL1               | 1.00                                 | dB               |
| SF01              | 200.1308005                          | MHz              |
| F2 - Proc         | essing paramete                      | rs               |
| 1.0               | 40000L7 000                          | - 10             |
| NDW               | Eduvuci VV2                          | 71114            |
| SSB               | 0                                    |                  |
| В                 | 0.10                                 | HZ               |
| 68                | 0                                    |                  |
| с<br>С            | 1.00                                 |                  |
| 1D NMA DI         | ot parameters                        |                  |
| cx                | 20.00                                | C B              |
| cγ                | 10.00                                | ۳.<br>م          |
| LIP               | 1.564                                | mqq              |
|                   | 313 08                               | HZ               |
|                   | -0.142                               | mqq              |
| r 2<br>DDMCM      | 65.85-<br>CERDO O                    | H2<br>nnm/rm     |
| HZCM              | 17 07567                             | HZ/Cm            |
|                   |                                      |                  |

54060.0-----

.....

20985.0------

51158.0---

Þ961Þ.1-----

wdd

4

![](_page_28_Figure_6.jpeg)

C C

c c

¢

น ว

0 C

<

С

.

~

EGG0.E

6767.12

769912-

1992 1

5086.0

9214.0.

1269.0

1796 6

0 4184

[engesni\_\_\_\_\_

| ent Data Parameters<br>BCMY-03-65monoLysmongMe<br>0 1<br>1 | Acquisition Parameters<br>20.47<br>20.47<br>20.47<br>20.47<br>20.47<br>20.47<br>20.47<br>20.47<br>20.47<br>20.47<br>20.47<br>20.0113<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>22768<br>20000000<br>2000<br>2000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>2000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>20000000<br>200000000 |   | A plot parameters<br>20.00 cm<br>10.00 cm<br>-3.6.75 Hz<br>-726.75 Hz<br>-1205.89 Hz<br>-1205.89 Hz<br>0 11971 ppm/cm<br>23 95690 Hz/cm |
|--|---|---|---|
| Current<br>NAME<br>EXPNO<br>PROCNO                         | F2 - Acca<br>Date<br>Time<br>PROBIDUA<br>PRUPNIC<br>PRUPNIC<br>TD<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>DS<br>DS<br>DS<br>DS<br>DM<br>DD<br>MCREST<br>MCREST<br>MCREST<br>MCREST  | NUC1<br>NUC1<br>PL1<br>SF01<br>S55<br>S55<br>S56<br>W0W<br>W0W<br>S58<br>S56<br>S56<br>S56<br>S56<br>S56<br>S56<br>S56<br>S56<br>S56<br>S56 | 10 NMR 01<br>CC<br>CY<br>F1<br>F1<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>CM   |

![](_page_29_Figure_1.jpeg)

\_\_\_\_

-450

-4.00 -4.25

wdd

4

![](_page_30_Figure_0.jpeg)

Experimental:

![](_page_31_Figure_0.jpeg)

![](_page_32_Figure_0.jpeg)

....cters 15384 300.1299835 MHz EM EM 0 0 0.10 Hz 1.00 10 NWR plot parameters CX 20.00 cm CY 11.98 cm F1P 12.500 ppm F1 3751.52 Hz F2P -4.500 ppm F2 -1350.59 Hz F2 -1350.59 Hz PPMCM 255.11047 Hz/Cm BCMY~03~75monoLysmonoH 5995.204 Hz 0.182959 Hz 2.7329011 sec 3322.5 usec 83.400 usec 6.00 usec 1.50000000 sec 0.01500000 sec ====== CHANNEL f1 ======== NUC1 1H P1 5.80 usec P1 -3.00 dB F01 300.1315007 MHZ 1H 5.80 usec -3.00 dB 300.1315007 MHz F2 - Processing parameters SI 16384 SF 300.1299833 MHz WDW EM SSB 0.10 Hz B 0.10 Hz BB 0.10 Hz BB 0.10 Hz F2 - Acquisition Parameters 20071019 15.12 15.12 15.12 15.12 5 mm PHGNP Swi 29 2768 0MSD 32768 0MSD Current Data Parameters NAME BCMY-03-75monoL PROBHD PULPROG T 10 NS NS SWH SSWH AQ DS DG DM MCREST MCREST MCREST INSTRUM PROCNO Date\_ EXPNO Time - 7 068.1 0100.4-----လု C ⊅SEB'0 -1976.0 2996 0 679.7 1.2827 5 13 625 1 3265 1.582 1.8525 9286.1 5.1399 - 3.1555 -- 3.0748 - 3.0748 15,336 575.21 3.2544 1425.E 7 14.403 - 3.2997 - 3.3700 < 1.219 < 4.029 E976.-£907.E ----5597.E -1877.E -0108.6 -0118.5 3978.E u 4.4232 694.0 E8E.0 2168.7 15.231 α

176.1

4.000

16109301 828.0

\_ <u><</u>

![](_page_33_Figure_1.jpeg)

10.3044 10.3158 10.3158

wdd

| s<br>bLysmonoH<br>1                | ters<br>Rec c c c c c c c c c c c c c c c c c c  | HT H   | ZHM  | cm<br>cm<br>ppm<br>ppm<br>ppm/cm<br>Hz/cm  |
|------------------------------------|--|--|--|--|
| Data Parameter<br>BCMY-03-75monu   | ULISITION Parame<br>25.13<br>55.13<br>56.15<br>56.0071015<br>27.25<br>2275<br>000000<br>5995.20<br>5995.20<br>5995.20<br>6.00<br>5.00<br>5.00<br>5.00<br>5.00<br>6.00<br>0.01500000<br>0.015000000 | CHANNEL f1 ===<br>1H<br>5.80<br>-3.00<br>300.1315007<br>300.1315007<br>cessing paramet | EM3<br>EM3<br>0.10<br>0.10<br>0.11<br>0.10<br>0.11 | lot parameters<br>20.00<br>11.98<br>12.50<br>6.000<br>1800.78<br>6.000<br>1800.78<br>97.5424 |
| Current<br>NAME<br>EXPNO<br>PROCNO | F2 - Acc<br>Date -<br>Time<br>PROBRUM<br>PROBRUM<br>PROBRUM<br>PULPROG<br>TD<br>SSULPROS<br>SWH<br>SSULPROS<br>SWH<br>SSULPROS<br>DM<br>AG<br>DM<br>MCREST<br>TE<br>D1<br>MCREST<br>MCREST         | NUC1<br>P1<br>PL1<br>SF01<br>SF01<br>SF01<br>SI<br>SI                                  | MOW B B B B B B B B B B B B B B B B B B B          | 10 NMR p<br>CCX<br>F1P<br>F1<br>F2P<br>PPMCM<br>HZCM   |

1019.7

2966 6 -----

2876.01 7765.01 8215.01 10.3044

![](_page_34_Figure_4.jpeg)

wdd

a singe

t c

![](_page_35_Figure_0.jpeg)
| ita Parameters<br>ICMY-03-75monoLysmonoH<br>1<br>1 | sition Parameters<br>20071019<br>15.12<br>15.12<br>95ect<br>28<br>2768<br>0MS0<br>32<br>0<br>32<br>0<br>5995.204 Hz<br>0.182959 Hz<br>2.7329011 sec<br>322.5 | B3.400 usec<br>6.00 usec<br>6.00 usec<br>300.0 K<br>1.5000000 sec<br>0.01500000 sec<br>0.01500000 sec<br>1.1<br>1.1<br>5.60 usec<br>-3.00 dB<br>300.1315007 MHz | ssing parameters<br>16394<br>300.1299035 MHz<br>EM<br>0<br>0<br>0<br>10 Hz<br>0<br>1.00 | t parameters<br>20.00 cm<br>11.98 cm<br>2.400 ppm<br>720.31 H2<br>-4.500 ppm<br>-1350.58 Hz<br>0.34500 ppm/cm<br>103.544B5 Hz/cm |
|--|--|---|---|--|
| Current Da<br>NAME E<br>EXPNO<br>PROCNO            | F2 - Acqui<br>Date_<br>Time<br>PROBHD E<br>PULPROG<br>TO SOLVENT<br>NS<br>SWH<br>FIDRES<br>AG<br>FIDRES  | DW<br>DE<br>TE<br>D1<br>MCREST<br>MCREST<br>MCREST<br>MCREST<br>MCREST<br>P1<br>P1<br>P1<br>SF01  | F2 - Proce<br>Sf<br>SSB<br>SSB<br>LB<br>CB<br>PC  | 10 NWR Plo<br>CX<br>F1<br>F1<br>F2<br>PPMCM<br>H2CM  |

\* \*

068.1 - 7

- ~

- î

- 1



0100.5-----

139/136



Chemical Formula: C<sub>43</sub>H<sub>52</sub>N<sub>10</sub>O<sub>6</sub> Exact Mass: 804.40713 Molecular Weight: 804.93638 m/z: 804.40713 (100.0%), 805.41048 (46.5%), 806.41384 (10.6%), 805.40416 (3.7%), 806.40752 (1.7%), 807.41719 (1.6%), 806.41138 (1.2%) Elemental Analysis: C, 64.16; H, 6.51; N, 17.40; O, 11.93



149/145

# Generic Display Report

### Analysis Info

| Analysis Info<br>Analysis Name<br>Method<br>Sample Name<br>Comment | D:\Data\K220808-ESI\KF_000<br>1MW Positive ESI<br>BY-04-79-5<br>RB5-3 | 0012.d            | Acquisition Date<br>Operator<br>Instrument | 8/22/2008 10:30:52 AM<br>Administrator<br>apex-Qe |
|--|---|-------------------|--|---|
| Intens.<br>x10 <sup>6</sup><br>1.50-                               | 791 40043   |                   | 1  |   |
| 1.25-  |   | -NI<br>-NI        | HN J                                       |   |
| 1.00-  |   | O=<br>WH          | a<br>NH2<br>Hb                             | [4  |
| 0.75-  |   | 792.40349 In 5td. |  |   |
| 0.50-  |   | Ę.                |  |   |
| 0.25   | MMMMMM  | WWWWW             | MAMM                                       | MMMMM   |
| 0.00 / / / / / / / / / / / / / / / / / /                           | 791.0 791.5 792.0   | 792.5 793.0 79    | )3.5 794.0                                 | 794.5 795.0 m/z                                   |
| +MS  |   |                   |  |   |

Bruker Daltonics DataAnalysis 3.4

#### printed: 8/22/2008 10:32:31 AM



0.90000 ppm/cm 70 11700 Hz/cm 10 NWR plot parameters CX 20.00 cm 21 120.00 cm 13.000 ppm 13.000 ppm 13.000 ppm 13.000 ppm 13.000 ppm 15.00 5 Hz 22 1500 55 Hz 22 1500 50 Hz 22 25 25 11700 Hz/cm 5.80 usec -3.00 dB 300.1315007 MHz F2 - Processing parameters SI 16384 SF 300 1300000 MHz WDW no SSB 0 B3.400 usec 6.00 usec F2 - Acquisition Parameters 2.7329011 sec 0.0000000 sec 5995.204 Hz 1.50000000 sec 0.01500000 sec ====== CHANNEL f1 ======= 0.00 Hz 0.182959 Hz 300.0 K 18.03 32768 Current Data Parameters BY-04-79 20080508 spect 0SMO 128 C Τ 1.00 52 0 5 mm PHONP Swi 256 TD SOL VENT NS DS SWH F TDRES Time INSTRUM PR08H0 PUL PROG NAME EXPNO PROCNO Jate\_ AO DW DW DE DE MCREST MCREST NUCI P1 PL1 SF01 882



PR010N

5.80 usec -3.00 dB 300.1315007 MHz 256 83.400 usec 5.00 usec 300.0 K 5995.204 Hz 0.182959 Hz 2.7329011 sec 1.50000000 sec 0.00000000 sec 0.01500000 sec F2 - Processing parameters SI 15384 SF 300.1300000 MHz WDW n0 SSB 0 LB 0 00 Hz GB 0 PC 1 00 F2 - Acquisition Parameters ====== CHANNEL f1 ====== 128 0 18.03 23 32768 DMSO 20080508 spect 5 mm PHONP Swi H PROCNO

 
 1D NMR plot parameters

 CX
 20 00 0

 CY
 300.00 0

 F1P
 13 006 p

 F1
 3903 54 H
 INSTRUM PROBHD PULPROG TD SOL VENT Date\_ F I DRES MCREST Time MCWRK F2P F2 PPMCM H2CM NUC1 P1 PL1 SF01 DS SWH ŝ Q ģ ð Ж ш 5

H H H

H4 , H4

HO-

0.21517 00m/cm 64\_57760 Hz/cm

0998 1

0000 v

0280 1

8 703 ppm 3903 54 Hz 2611 gg Hz

20 00 cm 300.00 cm 13 006 ppm

-10.3505 -10.3505 -10.3524 -10.3524

wdd

Current Data Parameters

BY-04-79

EXPNO

6'5925 6'3535 6'3365

\_

----

NAME

PR010N

|       | -7895-11   |   |  |   |
|-------|--|---|--|---|
|       | Current Dala Parameters<br>NAME BY-04-79<br>EXPNO 1<br>PROCNO 1<br>PROCNO 1<br>PROCNO 1<br>F2 - Acquisition Parameters<br>Date 20080508<br>Time 18.03<br>Time 18.03<br>Time 18.03<br>Time 20<br>PROBHD 5 mm PHONP Swi<br>PULPROG 5 mm PHONP Swi<br>PULPROG 5 mm PHONP Swi<br>PULPROG 12<br>DNS 1 | SWH         5995         204         Hz           FIDRES         0.182959         Hz           AQ         2<7329011 | PL1     -3.00 dB       F2     Processing parameters       Sf     300 1315007 MHz       Sf     300 1315007 MHz       Sf     300 100000 MHz       MDW     no       MDW     no       SSB     0       LB     0       LB     0       LB     0       CB     100       H2     100       MR     101 parameters       CX     20 | CY         300.00 cm           F1P         B 141 ppm           F1         2443 41 Hz           F2P         6 145 ppm           F2         1844 59 Hz           PPMCN         0 03936 ppm/cm           HZCM         29 94099 Hz/cm |
|       |  | •   | H I K  | GIC9 (<br>//599 ()  |
| ROTON | 99v78.ð  | ·   |  |   |
|       | 7 1 2 1 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2 2  |   | N HZ C   | 2 1841  |
|       | 20443.7<br>20443.7<br>20443.7  |   | ŧ  | <i>pl62</i> C   |
|       | 90262 2<br>P 9018 2<br>19099 2<br>P 2228 2<br>21588 2<br>61268 2<br>18116 2  |   | Z Z H K  | 6779 F  |
|       | шdo  |   |  |   |

0.09908 ppm/cm 29.73573 Hz/cm 256 83.400 usec 6.00 usec 5.80 usec -3.00 dB 300.1315007 MHz 5995.204 Hz 0.182959°Hz 2.7329011 sec 1.50000000 sec 0.00000000 sec 0.01500000 sec F2 - Processing parameters SI 16384 SF 300.130000 MHz WDW no SSB 0 1.00 Hz SB 0.00 Hz SB 0.00 Hz SB 0.00 Hz 20.00 cm 300.00 cm 4.660 ppm 1398.53 Hz 2.678 ppm 803 81 Hz 22 - Acquisition Parameters EXTERNEL (1 ====== 300.0 K 18.03 spect 5 mm PHONP Swi Current Data Parameters NAME BY-04-79 EXPNO 1 29 32768 DMS0 1D NMR plot parameters 2X 20.00 0 CY 300.00 0 71P 4.660 p 128 20080508 0 1 H INSTRUM 0H80Ha SOL VENT PULPROG PROCNO Date FIDRES MCRES1 lıme MCWRK M:)Mdc NUC1 Р1 РL1 SF01 SWH HZCM 01 d2 : ŝ OS Å0 В ð В Щ 5 ũ с. Ц 5.69299 5.72910 -2.79080 09908 . S 25.63035 06558.5 15759.5 2.99283 62E00 E 65190'E 808 5 Ť 10280 E E3 67601 E-212057E CH1 926 1 8855S.E 71825.E-19198 8-----3.65910 -3.62806 -3.57800 PR010N 92999.6 266-61 10517.E-3.72980 75527.E 756627 第三 T \$0020 P Eri 29260 % CF2 1742E.4 12526.4 64E wdd (610310)-

|  | ດ<br>ບ   | HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>HZ<br>H  | 12 212 6C<br>213 6C<br>14 2, 35 6C<br>14 2, 35 6C                           | m<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>C<br>D<br>D<br>C<br>D<br>D<br>C<br>D<br>D<br>C<br>D<br>D<br>C<br>D<br>D<br>C<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D<br>D   |
|--|--|--|---|--|
|  | ters<br>4-79<br>1<br>1<br>0508<br>8:03<br>8:03<br>8:03<br>8:03<br>8:03   | 2768<br>2768<br>128<br>128<br>128<br>128<br>128<br>128<br>128<br>128<br>128<br>12  | =====<br>1H<br>1H<br>5.80 L<br>5.80 L<br>6007 M<br>6007 M<br>100 M<br>100 M | 000 c<br>314 p<br>77 H p<br>57 B p<br>77 B p   |
|  | BY-0<br>BY-0<br>2008<br>11<br>51<br>51<br>9H0NP  | 3;<br>5995.<br>0.182<br>0.183<br>83.<br>83.<br>50000<br>50000<br>50000<br>000000<br>000000<br>0000000  | L f1<br>5<br>-3<br>1315<br>Para<br>Para<br>15<br>15<br>15<br>0              | 1<br>200<br>3.00<br>994<br>0.6<br>180<br>0.180<br>0.180  |
|  | ata P<br>isitio  | -00  | HANNE<br>300<br>55 ing<br>300   | , par  |
|  | Acquiring and acquiring actions and acquiring actions acquiring actions action | S S F  | ے ۔<br>۲۰۵۲ دو۔   |  |
|  | NAME<br>EXPNO<br>PROCN<br>Date -<br>INSTF<br>PROBH   | SOLVE<br>SOLVE<br>SSOLVE<br>SSWH<br>AQ<br>DS<br>SWH<br>AQ<br>DS<br>CVE<br>SCME<br>SOLVE<br>SCME<br>SCME<br>SCME<br>SCME<br>SCME<br>SCME<br>SCME<br>SCM |   | ZCM<br>ZCM   |
| 9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0<br>9/51/0   |  |  |   |  |
| 9/65/0     5/7       9/60/0     5/7       9/60/0     5/7       9/60/0     5/7       9/60/0     5/7       9/60/0     5/7       9/60/0     5/7       9/60/0     5/7       9/60/0     5/7       9   |  |  |   |  |
| 92684 0     77 8       97528 0     87 8       97529 0     87 8       97529 0     87 8       97529 0     97 8  |  |  |   |  |
| P2123 0<br>P2123 0<br>P21   | 92652 0  |  | ਰ   | E E E  |
| NSN0 0     C       27280 1     C       27280 1     C       27281 2     C <tr< td=""><td>84568 0<br/>84568 0</td><td></td><td>.38</td><td>to the second se</td></tr<> | 84568 0<br>84568 0   |  | .38   | to the second se |
| 27/2001     39/21       69/21     39/21       19/201     39/21  | 61100.1  |  |   |  |
| G6061     G6061       G6061 </td <td>27/90 1</td> <td></td> <td></td> <td><u>}</u></td>  | 27/90 1  |  |   | <u>}</u>   |
| EVEN 1<br>ECC2 1<br>1955 1<br>1955 1<br>1955 1<br>1955 1<br>1955 1<br>1955 1<br>1955 1<br>1955 1<br>1050 1<br>1955 1<br>1050 1   | 862/11   |  |   |  |
| BOES   | 865v2 · I  |  |   | $\sim$   |
| Bellov 1<br>Bellov   | 02922 1  |  |   |  |
| 2010/1 1<br>2000 2<br>2000   |  |  |   |  |
| U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101<br>U101   |  |  | · · ·   | ~ ~ ~  |
| und     Under the second  |  |  |   |  |
| W00     Itel:       100     Itel: <t< td=""><td></td><td>،<br/>د</td><td></td><td></td></t<>   |  | ،<br>د   |   |  |
| mu0     11675       11625     1167       11655     1167       11   | \$8187 ;   | to it  |   |  |
| LP981 2-<br>LP981 2-<br>LP981 2-<br>L1022  | 99519.1  | <u>X</u>   |   | B/AL P   |
| Lr991 2<br>ELr/2 2<br>ELr/2 2<br>GCBC 2<br>CGCC 2<br>CGCCC 2<br>CGCC 2<br>CG   |  | a'   |   |  |
| L/991 2<br>EL/2  |  |  |   |  |
| ELVL2'2<br>BCBEC 2<br>CEBEC 2<br>CEBEC 2<br>CECED 2<br>CEC   | 2×18647  |  |   |  |
| 00000 C C C C C C C C C C C C C C C C C  | 5.27473  | 0  |   | , second se   |
| 66259 / 2       1162 1         166259 / 2       1662 1         166259 / 2       162 1         166259 / 2       162 1         166259 / 2       162 1         166259 / 2       162 1         166259 / 2       162 1         167 1       167 1         167 2       167 1         167 2       167 1         167 2       167 1         167 2       167 1         167 2       167 1         167 3       167 1<   |  | w B  |   |  |
| 46605.5       66269.5       6700.6       6700.6       6700.6       6700.7       700.7       700.7       700.7       700.7       700.7       700.7 </td <td>5,43429</td> <td>0</td> <td></td> <td></td>   | 5,43429  | 0  |   |  |
| mdd  | 50332  | X  |   |  |
| mqq     (e36)at       mqq     (e36)at  | -2.69299   |  |   |  |
| mgq     Isolar       mgq     Isolar  | 0908/2   |  |   | $\langle \rangle$  |
| E9866'2<br>62 00 'E<br>62 00 'E<br>10990 'E<br>10990 'E<br>10990 'E<br>10970 '   | -5.82280   |  |   | 3  |
| 65190°C<br>10580°C<br>28161°C<br>28161°C<br>20071°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°C<br>21802°  |  |  |   |  |
| udo<br>wdo<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatutor)<br>(estatut   | 10580.E-   |  |   |  |
| wdo  |  |  |   | 1876 5-  |
| 2152812<br>wdo   | 21502'E  |  |   | $\sum$   |
|  | 3.25817  |  |   | <u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u><u></u></u>   |
|  |  |  |   | (euɓa)u!~  |

PR010N

| Current Data Parameters<br>MAMF BY-04-79 | E E PROCNO | F2 - Acquisition Parameters<br>Date20080508<br>Time18.03<br>INSTRUM spect | PROBHD 5 mm PHONP Sw1<br>PULPROG 29<br>TD 32768<br>SOLVENT DMS0<br>NS<br>DS 0<br>DS 0 | SWH         5995.204 Hz           FICHER         0.180911 SEC           AQ         2.7329011 SEC           RG         2.700 USEC           DE         300.0 K           MCREST         0.00000000 SEC           MCWRK         0.01500000 SEC | <pre>======= CHANNEL f1 =========<br/>NUC1 1H<br/>F1 5.80 USEC<br/>P1 300.1315007 MHZ<br/>SF01 300.1315007 MHZ</pre> | F2 - Processing parameters<br>SI 16384<br>SF 300.1300000 MHz<br>MDW no<br>SSB 0<br>LB 0<br>LB 0<br>C0 Hz<br>CB 1.00 | 10 NMR plot parameters<br>CX 20.00 cm<br>CY 300.00 cm<br>F1P -1545 ppm<br>F1 -493 75 H2<br>-4707 num | F2 - 1412 R5 H7<br>эрмСм 0.15312 рр.//с<br>H2CM 45.95522 H2/cr                            |
|--|------------|---|---|---|--|---|--|---|
|  |            |   |   |   |  | J-Z-I-  |  | station (1998)<br>1999 - Station (1998)<br>1999 - Station (1998)<br>1999 - Station (1998) |
|  | 2550.4-    |   |   |   |  | . www.  |  |   |
| Z  |            |   |   |   |  |   |  |   |
|  |            |   |   |   |  |   |  |   |
|  |            |   |   |   |  |   |  |   |
|  |            |   |   |   |  |   |  |   |
|  | យបីរ       | đ   |   | 47  |  |   |  | tergant.  |

•.

.

:. •



Experimental:

<sup>1</sup>H NMR:

<sup>13</sup>C NMR:

MS:

UV:

IR:

mp:

Yield:



;

ł

ž

179/176

avelet = 1 - 1 - 1



Chemical Formula: C<sub>48</sub>H<sub>55</sub>N<sub>13</sub>O<sub>9</sub> Molecular Weight: 958.03200

 $\mathcal{I}_{\text{HNMR}}$ 

## <sup>13</sup>C NMR:

MS:

✓ Hi Res MS:

UV:

IR:

mp:

Yield:

Experimental:







|   | ters<br>Hz<br>Hz<br>sec   | usec<br>sec<br>sec<br>sec  | usec<br>dB<br>MHz<br>ers  | ZHM ZH   | ст<br>ст<br>ррт<br>Н2<br>ррт<br>H2<br>H2/ст   |
|---|---|--|---|--|---|
| - Parameters<br>BCMY-03-103<br>2        | tion Parame<br>20071123<br>11.26<br>spect<br>m PHQNP Swi<br>29<br>32768<br>0MS0<br>64<br>64<br>64<br>0.18295204<br>0.182951204    | B3.400<br>6.00<br>300.0<br>1.5000000<br>0.00000000<br>0.01500000 | NNEL f1 ===<br>1H<br>5.80<br>-3.00<br>300.1315007<br>ing parametu | 16384<br>300.1300000<br>EM<br>0<br>0.10<br>0.10<br>0 | parameters<br>20.00<br>30.00<br>11.500<br>3451.50<br>-4.500<br>-1350.58<br>0.80000<br>240.10400 |
| Current Data<br>NAME<br>EXPNO<br>PROCNO | F2 - Acquisi<br>Date_<br>INSTRUM<br>PROBHD 5 m<br>PULPROG<br>TD<br>SULVENT<br>NS<br>SWH<br>SWH<br>SWH<br>SMH<br>SMH<br>SMH<br>SMH | DW<br>DE<br>1 TE<br>MCREST<br>MCWRK                              | ====== CHA<br>NUC1<br>P1<br>PL1<br>SF01<br>F2 - Process           | SI<br>MDW<br>FC<br>GB<br>BB<br>BB<br>PC<br>C         | 10 NMR plot -<br>CX<br>F1P<br>F1<br>F2<br>F2<br>PPMCM<br>H2CM                                   |



|  | د<br>د   | Hz<br>Hz<br>Hz<br>Sec<br>Sec<br>Sec<br>Sec   | u usec<br>dB<br>MHz<br>RTS   | MHZ<br>HZ                              | сл<br>сл<br>ррт<br>Н2<br>Н2<br>ррт/сл<br>Н2/ст  |
|--|--|--|--|--|---|
| ta Parameters<br>BCMY-03-103<br>2<br>1 | sition Parame<br>20071123<br>11.26<br>spect<br>mm PHQNP Swi<br>29<br>32768<br>DMSO<br>64 | 0<br>5995.204<br>0.182959<br>2.7329011<br>362<br>83.400<br>8.00<br>6.00<br>0.0000000<br>0.01500000<br>0.01500000 | AANNEL f1 ===:<br>1H<br>5.80<br>-3.00<br>300.1315007<br>351ng paramett | 300.1300000<br>EM<br>0<br>0.10<br>1.00 | : parameters<br>20.00<br>80.00<br>8.558<br>2568.59<br>6.851<br>2056.25<br>0.08535<br>25.61711<br>25.61711 |
| Current Da<br>NAME<br>EXPNO<br>PROCNO  | F2 - Acqui<br>Date<br>T1me<br>INSTRUM<br>PROBHD 5<br>PULPROG<br>TD<br>SOLVENT<br>NS      | DS<br>SWH<br>A AQ<br>DW<br>DG<br>DT<br>T TE<br>MCREST<br>MCREST  | ====== CH<br>NUC1<br>P1<br>PL1<br>SF01<br>SF01<br>S1<br>S1             | PC E B B B C K                         | 10 NMR Ploi<br>CX<br>CY<br>F1P<br>F1<br>F2P<br>F2P<br>F2P<br>PPMCM<br>H2CM                                |

۲/162'6 <u>-----</u>

2275.01 2275.01 2445

7487.01-----

wdd

ŝ



|  | H H Z K R S S S S S S S S S S S S S S S S S S  | sec<br>usec<br>MHz   | A MHZ   | cm<br>cm<br>hz<br>ppm<br>Hz<br>Hz<br>Hz/cm<br>Hz/cm   |
|--|--|--|---|---|
| Jata Parameters<br>BCMY-03-103<br>2<br>1 | 11211100 Parame<br>20071123<br>11.26<br>spect<br>5 mm PHQNP Swi<br>22<br>2758<br>00000<br>5995.204<br>0.182959<br>2.7329011<br>2.7329011<br>2.7329011<br>1.5000000<br>6.00 | 0.01500000<br>0.01500000<br>14<br>5.80<br>-3.00<br>300.1315007 | essing paramete<br>15384<br>300.1300000<br>EM<br>EM<br>0.10<br>0.10<br>1.00 | ot parameters<br>20.00<br>80.00<br>5.145<br>5.145<br>1544.21<br>2.005<br>601.77<br>0.15701<br>47.12214    |
| Current (<br>NAME<br>EXPNO<br>PROCNO     | F2 - Acqu<br>Date_<br>INSTRUM<br>PROBHD<br>PULPROG<br>T0<br>PULPROG<br>T0<br>S0LVENT<br>NS<br>SWH<br>SSUH<br>AQ<br>SWH<br>AQ<br>BM<br>AQ<br>DW<br>DM<br>DC<br>DM           | MCWRK<br>MCWRK<br>NUC1<br>P1<br>PL1<br>SF01                    | F2 - Proc<br>SF<br>WDW<br>SSB<br>SSB<br>CB<br>GB<br>FC                      | 10 NMR pl<br>CX<br>CV<br>F1<br>F1<br>F7<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>CM |



| 10700.7-      |
|---------------|
| 7.02999       |
| E8540.7.04283 |
| 95550 . 7     |
| 67820'Z       |

| V-7.42984 |
|-----------|
| 20124.7   |
| 85574.7   |
| 8E784.7   |
| 26060 2   |
| 7990S'L   |
| 57532.7   |
| E5725'2   |
| S6065'2-  |

E1010.7-----

83824.8-----

udd

ŝ



wdd

£,

57

lengarni –

ł

|                                     | ى<br>v  | Hz<br>HZ<br>Sec<br>Sec<br>Sec<br>Sec<br>Sec  | usec<br>MHz<br>Hz<br>Hz  | cm<br>cm<br>ppm<br>Hz<br>ppm<br>Hz<br>rm<br>rz/cm  |
|-------------------------------------|---|--|--|--|
| Parameters<br>BCMY-03-103<br>2<br>1 | .tion Parame!<br>20071123<br>11.26<br>spect<br>m PHQNP Swi<br>32768<br>DMSO | 5995.204<br>0.182959<br>2.7329011<br>362<br>83.400<br>83.400<br>5.00<br>5.00<br>300.0<br>1.5000000<br>0.01500000 | NNEL f1 ===:<br>14<br>5.80<br>-3.00<br>-3.00<br>-3.00<br>-3.00<br>16384<br>16384<br>16384<br>16384<br>16384<br>100000<br>00<br>00<br>10010 | parameters<br>20.00<br>80.00<br>-2.402<br>-720.82<br>-4.803<br>-1441.51<br>0.12005<br>36.03459 |
| rrent Data<br>ME<br>PNO<br>OCNO     | - Acquisi<br>te   | H<br>DRES<br>REST<br>WRK   | C C C C C C C C C C C C C C C C C C C  | NMR Plot<br>PCM  |
| PR NA CU                            | P P P P P P P P P P P P P P P P P P P                                       |  | PLLBSSI SFICE  | 6222 <u>55555</u> 5555   |



Ļ

| PNO<br>DCNO<br>- Acq<br>te_<br>STRUM | bemr-us-lus<br>2<br>1<br>1<br>2<br>1<br>20071123<br>11.26<br>11.26<br>spect  |
|--------------------------------------|--|
| CENT<br>VENT                         | 5 mm PHQNP 5wi<br>29<br>32768<br>DMSO<br>64<br>64<br>5995.204 Hz   |
| EST TEST                             | 0.182959 Hz<br>2.7329011 sec<br>352<br>83.400 usec<br>6.00 usec<br>6.00 usec<br>0.0500000 sec<br>0.01500000 sec  |
|                                      | CHANNEL f1 =======<br>1H<br>5.80 usec<br>-3.00 dB<br>300.1315007 MHz   |
| Pro                                  | cessing parameters<br>15384<br>300.1300000 MHz<br>EM<br>0<br>0.10 Hz<br>0.10 Hz<br>1.00  |
| d W                                  | <pre>10t parameters     20.00 cm     80.00 cm     -2.402 ppm     -720.82 Hz     -4.803 ppm     -1441.51 Hz     0.12006 ppm/cm     36.03459 Hz/cm</pre> |





Ľ.



|                                     | ອ<br>ບ<br>ນ  | HZ<br>HZ<br>K<br>K<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S<br>S    | usec<br>dB<br>MHz                                  | ers<br>Hz   | ст<br>ст<br>ррт<br>ррт<br>Нг<br>ррт/ст<br>H2/ст   |
|-------------------------------------|--|--|--|---|---|
| Parameters<br>BY04-45               | ion Parame<br>20080331<br>23.55<br>13.55<br>spect<br>spect<br>1<br>22<br>22<br>22<br>22<br>22<br>22<br>22<br>22<br>22<br>22<br>22<br>22<br>2 | 5,4657526<br>5,4657526<br>5,4657526<br>11.5<br>11.5<br>166.800<br>6.00<br>5.00<br>0.00000000<br>0.01500000 | INEL f1 ===<br>1F<br>10.1C<br>-3.0C<br>100.1315007 | ng paramet<br>16384<br>100.13000000<br>EV<br>EV<br>0.10<br>0.10<br>1.00 | arameters<br>20.00<br>11.96<br>5.345<br>1604.27<br>0.906<br>272.00<br>0.22195<br>66.61337 |
| urrent Data<br>AME<br>XPNO<br>ROCNO | 2 - Acquisit<br>ate<br>ime<br>NSTRUM<br>NSTRUM<br>NSTRUM<br>ULPROG<br>D<br>OLVENT<br>c<br>c  |  | ===== CHAN<br>UC1<br>1<br>L1<br>F01 3              | 2 - Processi<br>DF - 2<br>DF<br>B<br>B<br>B<br>C<br>C                   | D NMR Plot P<br>4<br>11P<br>22P<br>22P<br>22M<br>22M<br>22M                               |



004 Ŧ

| Jata Parameters<br>BY-04-45<br>2     | usition Parameters<br>20080331<br>13.55<br>13.55<br>spect<br>5 mm PHQNP Swi<br>29<br>32768<br>CDC13<br>15 | 2997.602 Hz<br>0.091480 Hz<br>5.4657526 sec<br>11.3<br>166.800 usec<br>6.00 usec<br>300.0 K<br>1.5000000 sec<br>0.0000000 sec<br>0.01500000 sec | CHANNEL f1 =======<br>1H<br>10.10 Usec<br>-3.00 dB<br>300.1315007 MHz | :essing parameters<br>16384<br>300.1300000 MHz<br>EM<br>0<br>0.10 Hz<br>1.00 | .ot parameters<br>20.00 cm<br>11.98 cm<br>2.781 ppm<br>834.52 Hz<br>0.931 ppm<br>279.40 Hz<br>0 09248 ppm/cm<br>27,75557 Hz/cm |
|--------------------------------------|---|---|---|--|--|
| Current  <br>NAME<br>EXPNO<br>PROCNO | F2 - Acqu<br>Date _<br>Time<br>INSTRUM<br>PROBHD<br>PULPROG<br>TD<br>SOLVENT<br>NS                        | Sum<br>Sum<br>FIDRES<br>AQ<br>DM<br>DE<br>DM<br>CMEST<br>MCREST   | NUC1<br>P1<br>PL1<br>SF01   | F2 - Proc<br>SI<br>WDW<br>SSB<br>SSB<br>SSB<br>CB<br>GB<br>FC                | 10 NMR D.<br>CC<br>CC<br>CC<br>F1<br>F1<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>CM<br>H2CM                                |

29152°E 18822°E E2178°E 19798°E

4.44103

2'00420 2'00302 2'02302 2'15422 2'15552

wdd



.

| urrent C<br>AME<br>XPNO<br>ROCNO                            | iata Parameters<br>BY-04-45<br>2<br>1   |
|---|---|
| 2 - Acqu<br>ime<br>NSTRUM<br>ROBHD<br>ULPROG<br>DLVENT<br>S | 15:11:00 Parameters<br>20080331<br>13.55<br>5 mm PHQNP Swi<br>29<br>32768<br>CDC13<br>15<br>15                                |
| AH<br>IDRES<br>3  | 0<br>2997.602 Hz<br>0.091480 Hz<br>5.4557526 sec<br>11.3  |
| CAREST<br>CWRK  | 156.800 usec<br>6.00 usec<br>300.0 K<br>1.5000000 sec<br>0.0000000 sec<br>0.01500000 sec                                      |
|   | CHANNEL f1 =======<br>1H<br>10.10 usec<br>-3.00 dB<br>300.1315007 MHz   |
| с<br>- желесс<br>- желесс                                   | essing parameters<br>15384<br>300.1300000 MHz<br>EM<br>0<br>0.10 Hz<br>1.00   |
| NMA P P P P P P P P P P P P P P P P P P P                   | ot parameters<br>20.00 cm<br>11.98 cm<br>2.781 ppm<br>834.52 Hz<br>0.931 ppm<br>279.40 Hz<br>0 09248 ppm/cm<br>27.75557 Hz/cm |







# Generic Display Report

## Analysis Info

| Analysis Name | D:\Data |
|---------------|---------|
| Method        | 1MW F   |
| Sample Name   | BY-04-  |
| Comment       |         |

D:\Data\K270808a-ESI\KF\_000005.d 1MW Positive ESI BY-04-67

## Acquisition Date 8/27/2008 10:38:35 AM

Operator Instrument Administrator

apex-Qe



Bruker Daltonics DataAnalysis 3.4

printed: 8/27/2008 10:40:05 AM

Page 1 of 1

28a /286

|     | Current Data Parameters<br>NAME by-04-49<br>EXPNO 2<br>PROCNO 1   | F2 - Acquisition Parameters<br>Date20080418<br>Time17.48<br>INSTRUM spect | PHUBHU 5 mm BBI 1H-BB<br>PULPROG 22768<br>32768<br>SOLVENT 20C13<br>NS 8<br>DS 0 | SWH 8012.820 Hz<br>FIDRES 0.244532 Hz<br>AO 2.0447731 sec<br>RG 25.400 usec<br>DW 62.400 usec | DE         6.00 usec           TE         300.0 K           D1         1.00000000 sec           MCHEST         0.00000000 sec           MCMRK         0.01500000 sec | ======= CHANNEL f1 ========<br>NUC1 1H<br>P1 8.20 usec<br>PL1 -3.00 dB<br>SF01 400.1320007 MHz | F2 - Processing parameters<br>SI 32768<br>SF 400.1300100 MHz<br>WDW no<br>SSB 0 | LB 0.00 Hz<br>GB 0<br>PC 1.00  | CX 12.50 Cm<br>CY 12.50 Cm<br>F1P 10.822 ppm<br>F1 4330.31 Hz<br>F2P -4.748 ppm<br>F2P -1899.90 HZ<br>PPMCM 01.77852 ppm/cm<br>H7CM 311.51062 Hz/Cm   |     |
|-----|---|---|--|---|--|--|---|--|---|-----|
|     | 1841.   | -   | A LAND   | L'ELL   | t of to  | °,∕<≂  |   |  | <u>LET.</u>   |     |
|     | , 1536<br>, 1536<br>, 2580<br>, 2580<br>, 2580<br>, 2580<br>, 2580<br>, 2580<br>, 2680<br>, 26800<br>, 26800<br>, 26800<br>, 26800<br>, 26800<br>, 26800<br>, 26800<br>, 26800<br>, 26800 |   | Y  | <b>`</b>  | τö   |  |   |  | 2:0.327<br>3.902<br>3.902<br>1.826<br>1.826<br>1.826<br>1.826<br>3.902<br>1.826<br>1.826<br>3.902<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.827<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826<br>1.826  | 2 0 |
| 171 | 8008<br>9088<br>1007<br>1007<br>1007<br>1007<br>1007<br>1007<br>1007<br>1   |   |  |   |  |  |   | Contraction of the second seco | 2.361<br>0.084<br>0.092<br>0.092<br>2.060<br>3.233<br>6.150<br>0.092<br>2.439<br>0.092<br>3.233<br>0.390<br>0.433<br>0.390<br>0.433<br>0.390<br>0.433<br>0.390<br>0.433<br>0.390<br>0.433<br>0.390<br>0.433<br>0.390<br>0.433<br>0.390<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.433<br>0.4330<br>0.4330<br>0.4330<br>0.4330<br>0.4330<br>0.4330<br>0.4330<br>0.4330000000000 | 6 4 |
|     | . 9501<br>. 9233<br>. 9048<br>. 0290<br>. 2599<br>. 9876  |   |  |   |  |  |   |  | 799.1<br>865.0<br>865.0<br>637.0<br>867.0   |     |
|     | .0234<br>.0234<br>0832  | 6<br>OT<br>d  |  |   |  |  |   |  | [670937]<br>540,4   | ppm |

PROTON

23+1.5+5.+1.6

, A

3

×



wdd

68

.

|   | .ers<br>HZ<br>Sec<br>sec<br>sec<br>sec<br>sec   | usec<br>dB<br>MHz                                 | ers<br>MHz<br>Hz   | ст<br>ст<br>ррт<br>Hz<br>Ppm<br>Hz<br>Hz/ст<br>Hz/ст   |
|---|---|---|--|--|
| Parameters<br>by-04-49<br>2             | tion Paramet<br>20080418<br>17.48<br>spect<br>spect<br>22<br>32768<br>8012.820<br>0.244532<br>2.0447731<br>2.0447731<br>25.44<br>62.400<br>6.00<br>6.00<br>6.0000000<br>0.01500000  | NNEL f1 ===<br>1H<br>8.20<br>-3.00<br>400.1320007 | ing paramett<br>32768<br>400.1300100<br>00<br>0.00<br>1.00 | parameters<br>20.00<br>12.50<br>6.657<br>6.653.57<br>4.763<br>1905.96<br>0.09467<br>37.88041 |
| Current Data<br>NAME<br>EXPNO<br>PROCNO | F2 - Acquisit<br>Date -<br>Time<br>INSTRUM<br>PULPROBHD 5 mm<br>PULPROG<br>50LVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOLVENT<br>SOL<br>DE<br>FIDRES<br>AG<br>DM<br>D1<br>DE<br>DE<br>DE<br>DE<br>DE<br>DE<br>DE<br>DE<br>DE<br>DE<br>DE<br>DE<br>DE | ====== CHAN<br>NUC1<br>P1<br>PL1<br>SF01          | F2 - Processi<br>S1<br>SF<br>WDW<br>SSB<br>LB<br>CG<br>PC  | 10 NMR Plot F<br>CX<br>CY<br>F1P<br>F1<br>F2P<br>F2P<br>F2P<br>PPMCM<br>H2CM                 |

4,98865 43856 43856 438662

AE10.0 ļ Alt र्ड इ.स τ 2.3613 5.0 AND VE · X Hr Z 0.7643 ນ ເງ 5.4 CH an DWA HA 5.6 1F. Z JWJ 6622.0 2.8 6.0 8795.0 6.2 Ş 5516.0 H) S HN 6.4

lengərni

mdd

¥

PROTON sw: 20ppm 17--3



| ita Parameters<br>by-04-49<br>2<br>1 | <pre>:sition Parameters     20080418     17.48     spect     spect     smm BBI 1H-BB     zg     32768     COC13     B     0     B     0     B     0     B     0     B     0     COC133     B     cO     32758     cond0731     sec     25.4     62.400 usec     6.00 usec     6.00 usec     6.00 usec     0.0000000 sec     0.00000000 sec     0.0000000 sec     0.0000000 sec     0</pre> | CHANNEL f1 =======<br>1H<br>8.20 usec<br>-3.00 dB<br>-3.00 dB<br>32768<br>400.13200100 MHz<br>n0<br>0<br>0<br>0<br>1.00<br>1.00 | ot parameters<br>20.00 cm<br>12.50 cm<br>3.000 ppm<br>1200.39 Hz<br>1.400 ppm<br>560.18 Hz<br>0.08000 ppm/cm<br>32.01040 Hz/cm |
|--------------------------------------|--|---|--|
| Current D<br>NAME<br>EXPNO<br>PROCNO | F2 - Acqu<br>Date<br>Time<br>INSTRUM<br>PROBHD<br>PULPROG<br>PULPROG<br>PULPROG<br>SOLVENT<br>NS<br>SOLVENT<br>NS<br>SOLVENT<br>NS<br>SOLVENT<br>NS<br>PID<br>FIDRES<br>SWH<br>SWH<br>DM<br>CREST<br>MCREST<br>MCMRK   | REFERENCE<br>NUCL<br>PL<br>PL<br>FZ - Proc<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS        | 10 NMR p1<br>CX<br>CY<br>F1<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2<br>F2                      |



s

|                                       | ers<br>HZ<br>HZ<br>Sec<br>Sec<br>Sec<br>Sec   | uusec<br>dB<br>Brs<br>MHz<br>Hz<br>Hz  | cm<br>cm<br>ppm<br>Hz<br>ppm<br>Hz<br>Hz/cm                                     |
|---------------------------------------|---|--|---|
| ata Parameters<br>by-04-49<br>2<br>1  | isition Paramet<br>20080418<br>17.48<br>spect<br>spect<br>29<br>32768<br>32768<br>60<br>32768<br>8012.820<br>0.244532<br>2.0447731<br>2.0447731<br>2.0447731<br>25.0447731<br>25.44532<br>2.0447731<br>25.44532<br>2.0447731<br>1.00000000<br>6.00<br>6.00<br>6.000<br>0.00000000<br>0.01500000 | CHANNEL f1 ===:<br>1H<br>8.20<br>-3.00<br>-3.00<br>-3.00<br>-3.00<br>-3.00<br>-3.00<br>-3.00<br>-3.00<br>-3.00<br>-3.00<br>-0.00<br>-0.00<br>-0.00 | <pre>pt parameters 20.00 12.50 1.400 560.18 -0.000 -0.00 0.07000 28.00910</pre> |
| Current Di<br>NAME<br>EXPNO<br>PROCNO | F2 - Acqu<br>Date _<br>INSTRUM<br>PROBHD _<br>PULPROG<br>PULPROG<br>TD<br>SOLVENT<br>NS<br>SWH<br>NS<br>SWH<br>NS<br>SWH<br>NS<br>SWH<br>NG<br>RG<br>DM<br>DG<br>DM<br>CWRK<br>MCREST<br>MCWRK  |  | 10 NWR pl.<br>CX<br>CY<br>F1P<br>F1<br>F2P<br>F2P<br>PPMCM<br>HZCM              |



ş

قرد


| ameters<br>y-04-49<br>2<br>1                   | Parameters<br>0080418<br>17.48<br>spect<br>1 H-BB<br>2768<br>32768<br>CDC13     | B<br>012.820 Hz<br>.244532 Hz<br>0417731 sec<br>25.4<br>6.00 usec<br>5.00 usec<br>300.0 K<br>0000000 sec<br>1500000 sec | f1 ========<br>1H<br>8.20 usec<br>-3.00 dB<br>1320007 MHz<br>1320007 MHz<br>32768<br>1300100 MHz<br>0<br>0 0.00 Hz<br>0<br>1.00 | meters<br>20.00 cm<br>12.50 cm<br>-2.476 ppm<br>-990.77 Hz<br>-4.815 ppm<br>-1926.64 Hz<br>0.11695 ppm/cm |
|--|---|---|---|---|
| Current Data Par<br>NAME br<br>EXPNO<br>PROCNO | F2 - Acquisition<br>Date2<br>Time<br>INSTRUM<br>PROBHD 5 mm BB<br>PULPHOG<br>TD | NS<br>SWH<br>SWH<br>FIDRES<br>AG<br>AG<br>AG<br>AG<br>AG<br>DM<br>DE<br>D1<br>D1<br>MCMRK<br>0.0                        | ======= CHANNEL<br>NUC1<br>P1<br>PL1 400.<br>SF01 400.<br>SF2 - Processing<br>SI<br>SSB<br>MDW<br>SSB<br>LB<br>GB               | 1D NMR plot para<br>CX<br>CY<br>F1P<br>F1<br>F2<br>F2P<br>F2<br>PPMCM                                     |

HULANI

РRОТОN sw: 20ppm 17--3

ú

mqq

lengerni

٤.











|                                  | м<br>L   | HZ<br>HZ<br>Sec<br>Sec<br>Sec<br>Sec   | ====<br>usec<br>dB<br>MHz                        | HZ HZ  | cm<br>cm<br>ppm<br>Hz<br>Hz<br>Ppm/cm<br>Hz/cm   |
|----------------------------------|--|--|--|--|--|
| arameters<br>BY-04-75a<br>1<br>1 | ton Parameti<br>20080508<br>12.20<br>spect<br>BBI 1H-BB<br>32768<br>MeOD<br>32 | 0<br>8012.820<br>0.244532<br>2.0447731<br>50.8<br>62.400<br>62.400<br>62.00<br>6.00<br>6.00<br>0.0000000<br>0.00000000 | NEL f1 ====<br>1H<br>8.20<br>-3.00<br>00.1320007 | ng paramete<br>32768<br>00.1300000<br>00<br>0<br>0.00<br>1.00                    | arameters<br>20.00<br>300.00<br>10.379<br>4152.93<br>5.675<br>2270.85<br>0.23518<br>94.10406 |
| ent Data F<br>10<br>10           | - Acquisit)<br>  | S 3F   | ===== CHAN<br>1<br>1 4                           | - Processi<br>4  | NMA Plot P   |
| CULL<br>NAME<br>EXPN<br>PROC     | F2 -<br>Time<br>Time<br>PULP<br>FULP<br>NS<br>NS<br>NS                         | DS<br>FIDF<br>AQ<br>DE<br>DE<br>MCRI<br>MCRI   | PL1<br>SF0                                       | PC<br>CB<br>CB<br>CB<br>CB<br>CB<br>CB<br>CB<br>CB<br>CB<br>CB<br>CB<br>CB<br>CB | 10<br>11<br>12<br>12<br>12<br>12<br>12<br>12<br>12<br>12<br>12                               |

75163.8----6.67742

07978.7----

РЯОТОN sw: 20ppm 17--3



G19E9.9-E7068.e--9.73212 88887.6-





|   | ers<br>ssec<br>ssec<br>ssec<br>ssec<br>ssec<br>ssec  | usec<br>dB<br>MHz<br>MHz<br>Hz  | см<br>см<br>Ррм<br>Нz<br>Ррм<br>Нz<br>Hz/см   |
|---|--|---|---|
| a Parameters<br>BY-04-75a<br>1          | ition Paramet<br>20080508<br>12.20<br>spect<br>mm BBI 1H-BB<br>72<br>32768<br>MeOD<br>8012.820<br>0.244552<br>2.0447731<br>50.8<br>52.400<br>6.00<br>6.00<br>0.0000000<br>0.01500000<br>0.01500000 | ANNEL f1 ====<br>1H<br>8.20<br>-3.00<br>400.1320007<br>sing paramete<br>32768<br>400.1300000<br>0<br>0.00<br>0              | parameters<br>20.00<br>100.000<br>4.657<br>1863.21<br>2.681<br>1072.72<br>0.09878<br>39.52457 |
| Current Data<br>NAME<br>EXPNO<br>PROCNO | F2 - Acquis<br>Date<br>INSTRUM<br>PROBHO 5 1<br>PULPROG<br>TD PULPROG<br>TD PULPROG<br>SWH<br>SSWH<br>SWH<br>SWH<br>SWH<br>SWH<br>CDE<br>DE<br>DE<br>DE<br>DI<br>MCREST<br>MCREST                  | ======= CH<br>NUC1<br>P1<br>P1<br>PL1<br>SF01<br>SF<br>SF<br>SF<br>SF<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS<br>SS | 1D NMR plot<br>CX<br>CY<br>F1P<br>F1<br>F2<br>F2P<br>PPMCM<br>HZCM                            |



РRОТОN sw: 20ррт 17--3

| 1913<br>1913<br>1913<br>1914<br>1924<br>1924<br>1929<br>1929<br>1929<br>1929<br>1929 | 400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>40000000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>400000<br>4000000<br>4000000<br>40000000<br>4000000<br>40000000<br>400000000 |
|--|--|
|--|--|

PROTON sw: 20ppm 17--3

۲. ۲

| ata Parameters<br>BY-04-75a<br>1    | isition Parameters<br>20080508<br>12.20<br>spect<br>5 mm BBI 1H-BB<br>29<br>32768<br>MeOD<br>327 | 0<br>8012.820 Hz<br>0.244532 Hz<br>2.0447731 sec<br>50.8<br>62.400 usec<br>6.00 usec<br>300.0 K<br>1.00000000 sec<br>0.01500000 sec | CHANNEL f1 =======<br>1H<br>8.20 usec<br>-3.00 dB<br>400.1320007 MHz | :essing parameters<br>32768<br>400.1300000 MHz<br>no<br>0<br>0.00 Hz<br>1.00 | .ot parameters<br>20.00 cm<br>30.00 cm<br>3.451 ppm<br>1380.75 ppm<br>1315.24 Hz<br>0.00819 ppm/cm<br>3.27544 Hz/cm |
|-------------------------------------|--|---|--|--|---|
| urrent Data<br>AME<br>XPNO<br>ROCNO | 2 - Acquis<br>ste<br>ustRUM<br>vSTRUM<br>30BHD 5 -<br>JLVENT<br>5 -<br>JLVENT                    | S<br>24<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1<br>1                                   | LC1<br>LC1<br>L1<br>C1<br>C1   |  | ) NMR plot<br>11<br>22P<br>22P<br>22CM<br>22CM  |



81

ц. Р