

Journal of
Medicinal Chemistry

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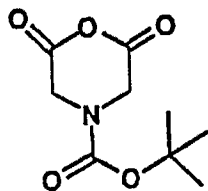


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JM 950043J

M-1608-1



$C_9H_{13}NO_5$
Exact Mass: 215.0793
Mol. Wt.: 215.2054
C, 50.23; H, 6.09; N, 6.51; O, 37.17

☒ CHN Requested

	Theoretical
% C	50.23
% H	6.09
% N	6.51

CHN Analyst ULB

Found

49.996.206.67

Date

12-27-95

4.462

49.3

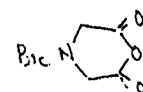
110.4

OBSERVE
Nucleus 1.000 Freq 300 MHz
Spec. Width 4500.5 Hz Offset 0 Hz
Acq. Time 3.335 sec Delay 1.000 sec
Pulse Width 14.0 μ sec Transients 32

RECEIVE
Nucleus 1.000 Offset -450.0 Hz
Mode NNN Power 1900.0 db
Module: Mode C Freq 200 Hz
Pulse Width μ sec Power Mode 1.0

PLOT/PROCESSING
FN 64 K RE sec CD sec
LB Hz AF sec CCD
Width 3000.9 Hz/ppm Start -59.9 Hz/ppm
Reference

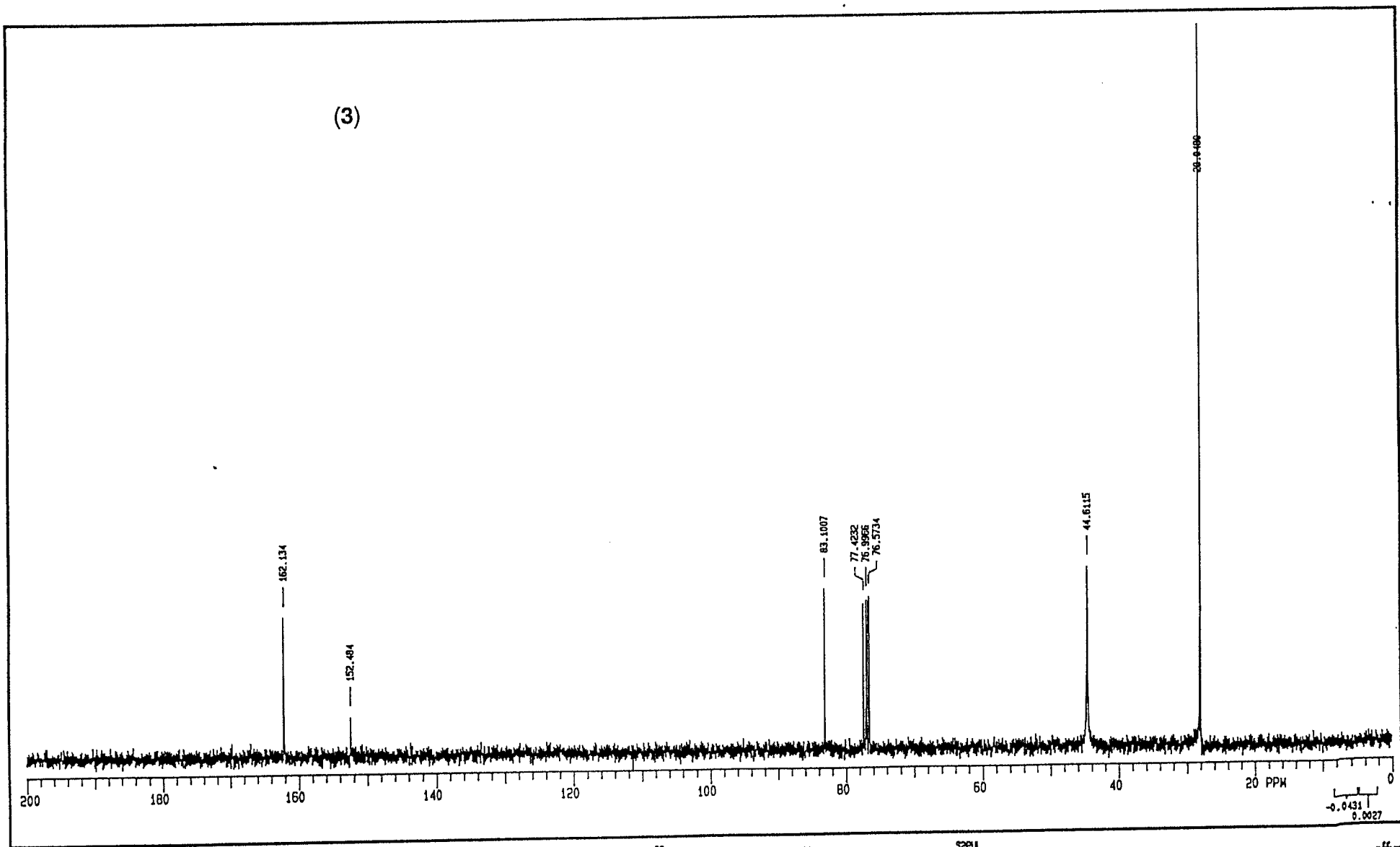
EXPERIMENT
Pulse Sequence SPUL
Tube OD mm
Temp °C
Solvent CDCL3

SAMPLE
BOC-ANHYDRIDE in CDCL3

Number H
File 09-26-94
Date 12-27-95
Time 5:52:45
XL 300

affymax

M-1608-d



RESERVE

Nucleus	13.000	Freq	75	MHz	
Spec Width	18761.7	Hz	Offset	0	Hz
Acq Time	0.800	sec	Delay	0	sec
Pulse Width	13.3	μsec	Transients	320	

DECOUPLE

Nucleus	1.000	Offset	-450.0	Hz	
Mode	YYY	Power	20.0	dB	
Modulation Mode	S	Freq	9656	Hz	
Pulse Width	15.8	μsec	Power Mode	1.0	

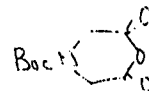
PLOT/PROCESSING

FN	32	K	RE	sec	CD	sec
LB	1.000	Hz	AF	sec	CCD	sec
Width	15092.5	Hz/ppm	Start	0	Hz/ppm	
Reference						

EXPERIMENT

Pulse Sequence	S2FUL
Tube OD	mm
Temp	°C
Solvent	CDCl3

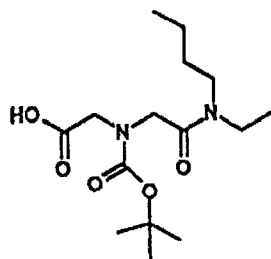
SAMPLE
PROTON PARAMETERS
VARIAN BENINI-300



Number C
File 09-26-94
Date 14 50 41
Time BM 300
XL

affymax

M-1608-3

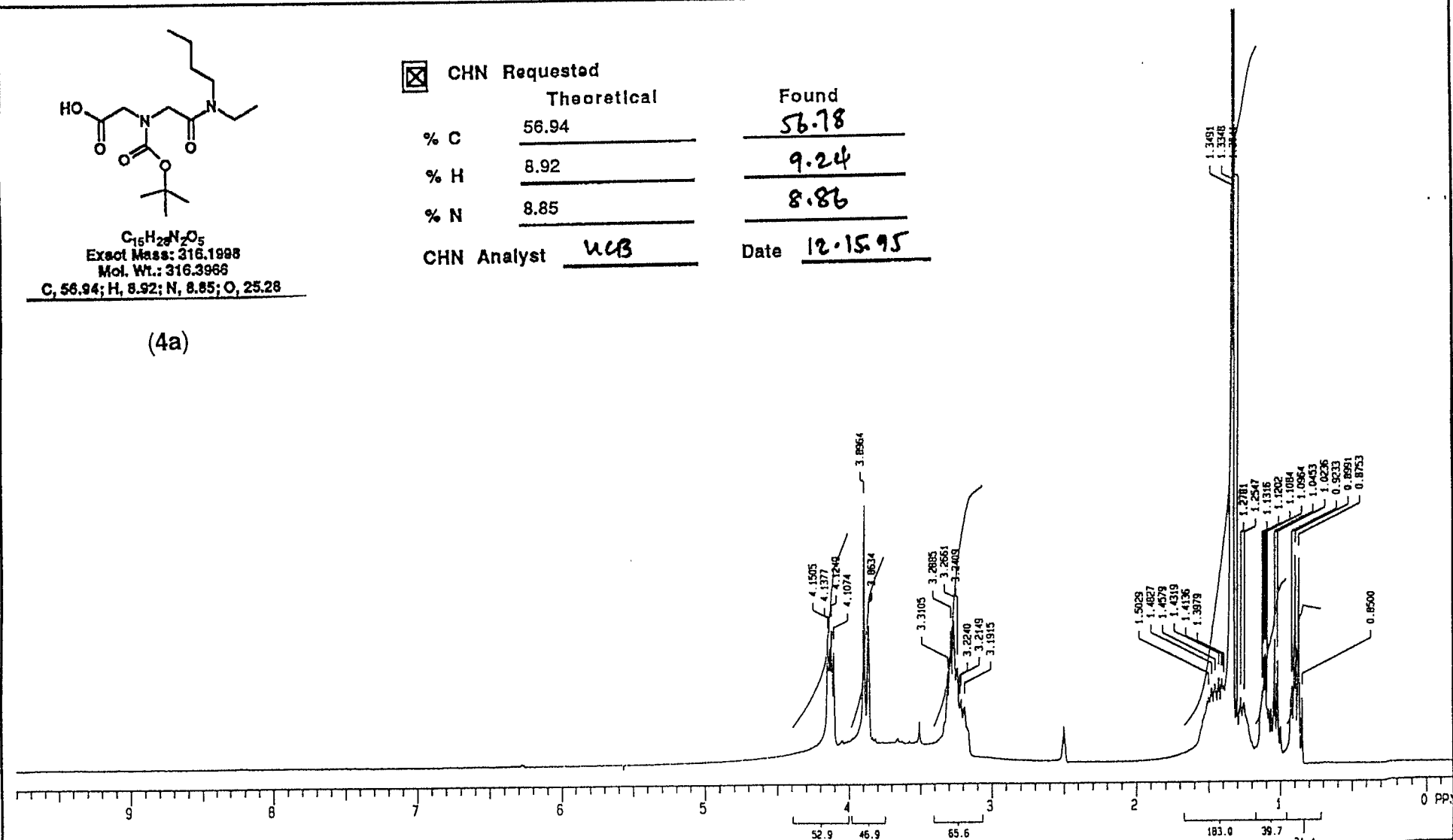


$C_{15}H_{22}N_2O_5$
 Exact Mass: 316.1998
 Mol. Wt.: 316.3966
 C, 56.94; H, 8.92; N, 8.85; O, 25.28

(4a)

☒ CHN Requested

	Theoretical	Found
% C	56.94	56.78
% H	8.92	9.24
% N	8.85	8.86

CHN Analyst UCBDate 12.15.95

OBSERVE
 Nucleus 1.000 Freq 300 MHz
 Spec. Width 4500.5 Hz Offset 0 Hz
 Acq. Time 1.778 sec Delay 1.000 sec
 Pulse Width 12.0 μ sec Transvers 32

RECEIVE
 Nucleus 1.000 Offset -450.0 Hz
 Mode HNH Power 20.0 db
 Modulation Mode C Freq 200 Hz
 Pulse Width 1.0 μ sec Power Mode 1.0

PLOT/PROCESSING
 FN 16 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD --- sec
 Width 3001.0 Hz/turn Start -58.9 Hz/ppm
 Reference ---

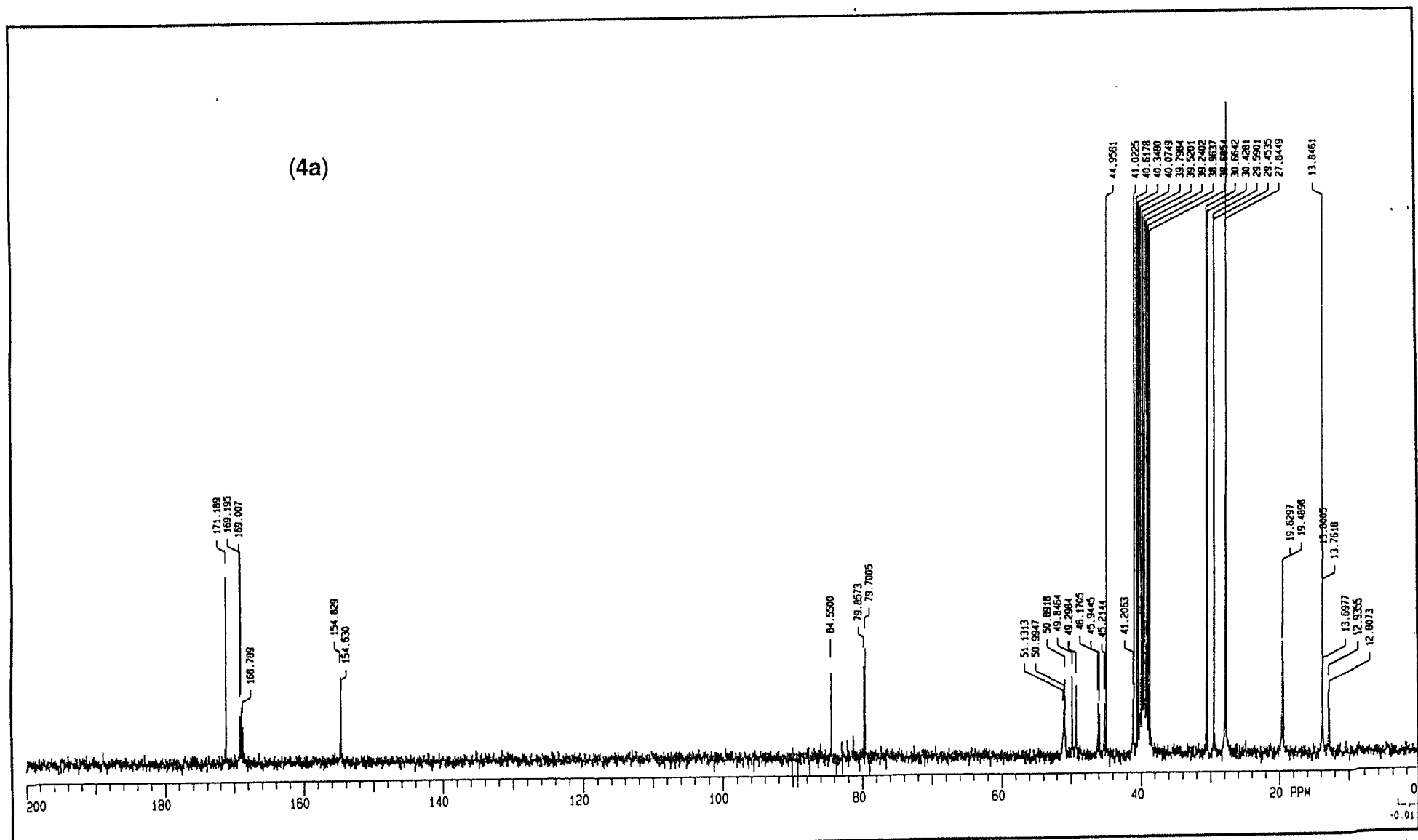
EXPERIMENT
 Pulse Sequence SZBLU
 Tube O.D. --- mm
 Temp --- °C
 Solvent DMSO

SAMPLE
 BOC-EBU IN DMSO

Number ---
 File ---
 Date 12-07-95
 Time 13:08:02
 XL GEN 300

affymax

M-1608-4



RESERVE

Nucleus	13.000	Freq	75	MHz
Spec. Width	8761.7	Hz	Offset	0
Acq. Time	0.800	sec	Delay	1.000
Pulse Width	13.3	μsec	Transmits	640

RECEIVED

Nucleus	1.000	Offset	-450.0	Hz
Mode	YYY	Power	20.0	db
Modulation Mode	S	Freq	9656	Hz
Pulse Width	15.8	μsec	Power Mode	1.0

PLBT/PROCESSING

FN	32	K	RE	---	sec	CD	---	sec
LB	1.000	Hz	AF	---	sec	CCD	---	sec
Width	15092.5	Hz/ppm	Start	0	Hz/ppm			
Reference								

EXPERIMENT

Pulse Sequence	S2PL1	
Tube OD	mm	
Temp	---	°C
Solvent	DMSO	

SAMPLE

BOC-EBU IN DMSO

Number

File

Date

Time

XL

affymax

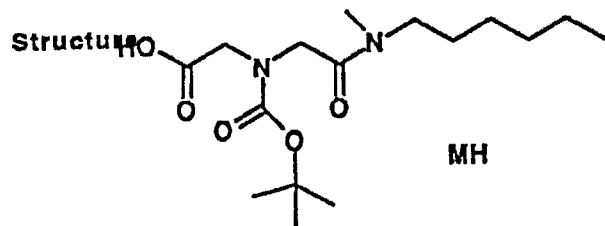
C13

12-07-96

13:15:39

GEN 300

M-1608-5

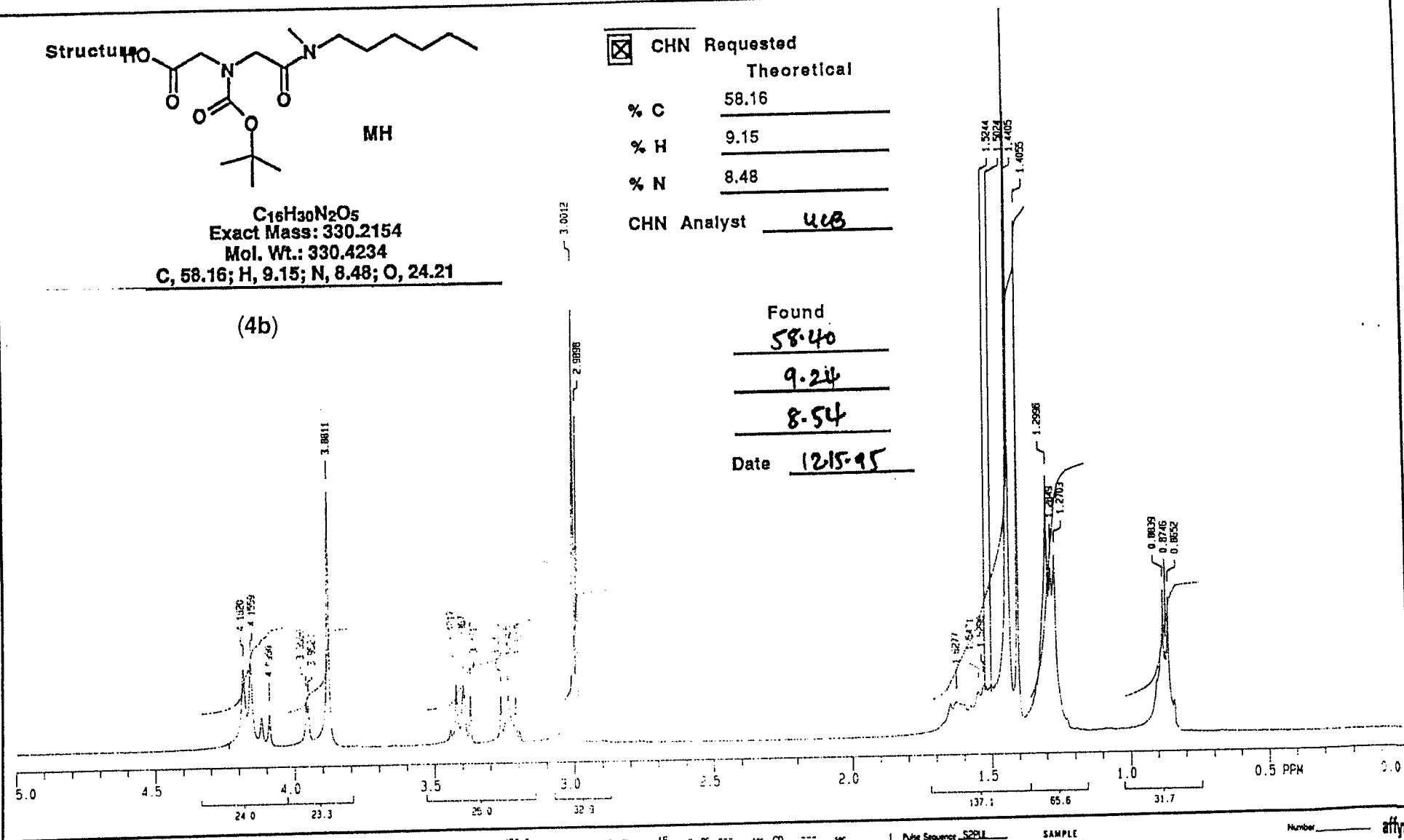


$C_{16}H_{30}N_2O_5$
 Exact Mass: 330.2154
 Mol. Wt.: 330.4234
 C, 58.16; H, 9.15; N, 8.48; O, 24.21

(4b)

☒ CHN Requested
 Theoretical
 % C 58.16
 % H 9.15
 % N 8.48
 CHN Analyst UJB

Found
58.40
9.24
8.54
 Date 12-15-95



OBSERVE
 Nucleus 1.000 Freq 300 MHz
 Spec. Width 4500.5 Hz Offset 0 Hz
 Acq. Time 1.778 sec Delay 1.000 sec
 Pulse Width 12.0 µsec Transmits 32

RECEIVE
 Nucleus 1.000 Offset -450.0 Hz
 Mode NNN Power 20.0 db
 Modulation Mode C Freq 200 Hz
 Pulse Width µsec Power Mode 1.0

PLOT/PROCESSING
 F1 16 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD ---
 Width 1500.5 Hz ppm Start 0 Hz ppm
 Reference

EXPERIMENT
 Pulse Sequence S2P1
 Tube OD --- mm
 Temp --- °C
 Solvent CDCl3

SAMPLE
BOC-MH

Number affym
 File H
 Date 12-05-95
 Time 12:45:14
 XL GEN 300

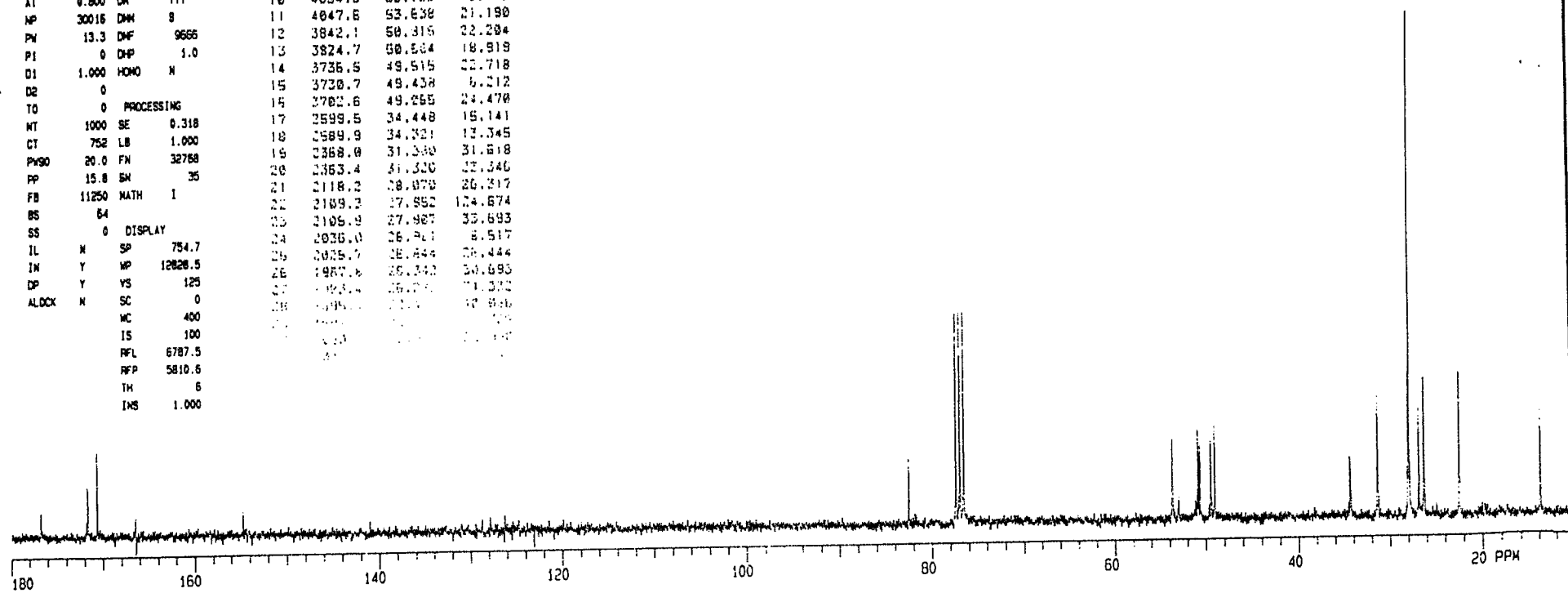
M-1608-6

SPECTRAL LINES FOR 13C 5.02
RFL 6787.5 RFP 5810.6EXP3 PULSE SEQUENCE: SPPUL
DATE 12-06-95 13:02:57
SOLVENT CDCL3
FILE C13

ACQUISITION		DEC. & VT	
TM	13.000	DN	1.000
SM	18761.7	DO	-450.0
AT	0.800	DM	YYY
MP	30016	DW	8
PM	13.3	DHF	9666
PI	0	DHP	1.0
DI	1.000	HOMO	N
D2	0		
TO	0	PROCESSING	
MT	1000	SE	0.318
CT	752	LB	1.000
PM90	20.0	FN	32768
PP	15.8	SW	35
FB	11250	MATH	1
BS	64		
SS	0	DISPLAY	
IL	N	SP	754.7
IN	Y	MP	12626.5
DP	Y	VS	125
ALOCK	N	SC	0
		MC	400
		IS	100
		RFL	6787.5
		RFP	5810.6
		TH	6
		INS	1.000

INDEX	FREQ	PPM	INTENSITY
01	12341.7	176.620	5.185
02	12459.9	171.741	12.827
03	12957.8	171.112	9.650
04	12878.9	170.691	21.487
05	11078.7	154.762	7.584
06	6227.6	82.523	16.244
07	5844.1	77.444	52.416
08	5812.0	77.019	55.188
09	5779.4	76.594	54.102
10	4054.0	53.733	16.113
11	4047.6	53.638	21.190
12	3842.1	50.315	22.204
13	3824.7	50.264	18.919
14	3736.5	49.515	22.718
15	3730.7	49.438	6.212
16	3702.6	49.255	24.478
17	2599.5	34.448	15.141
18	2589.9	34.321	13.345
19	2368.0	31.330	31.618
20	2363.4	31.320	12.340
21	2118.2	28.070	26.317
22	2109.3	27.852	124.674
23	2105.9	27.987	33.693
24	2036.0	26.821	8.517
25	2025.7	26.644	26.444
26	1987.6	25.342	34.693
27	1923.4	25.074	71.332
28	1895.1	24.714	17.866
29	1877.1	24.444	17.866
30	1877.1	24.444	17.866

(4b)



OBSERVE
 Nucleus _____ Freq _____ MHz
 Spec. Width _____ Hz Offset _____ Hz
 Acq. Time _____ sec Delay _____ sec
 Pulse Width _____ μ sec Transients _____

RECEIVED
 Nucleus _____ Offset _____ Hz
 Mode _____ Power _____ db
 Modulation Mode _____ Freq _____ Hz
 Pulse Width _____ μ sec Power Mode _____

PLOT/PROCESSING
 FM _____ K RE _____ sec CD _____ sec
 LB _____ Hz AF _____ sec CCD _____
 Width _____ Hz/ppm Start _____ Hz/ppm
 Reference _____

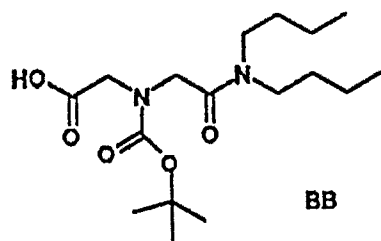
EXPERIMENT
 Pulse Sequence _____
 Tube O.D. _____ mm
 Temp _____ °C
 Solvent _____

SAMPLE

Boc-MH

Number _____ affym
 File _____
 Date _____
 Time _____
 XL _____

M-1608-7



$C_{17}H_{32}N_2O_5$
 Exact Mass: 344.2311
 Mol. Wt.: 344.4502
 C, 59.28; H, 9.36; N, 8.13; O, 23.22

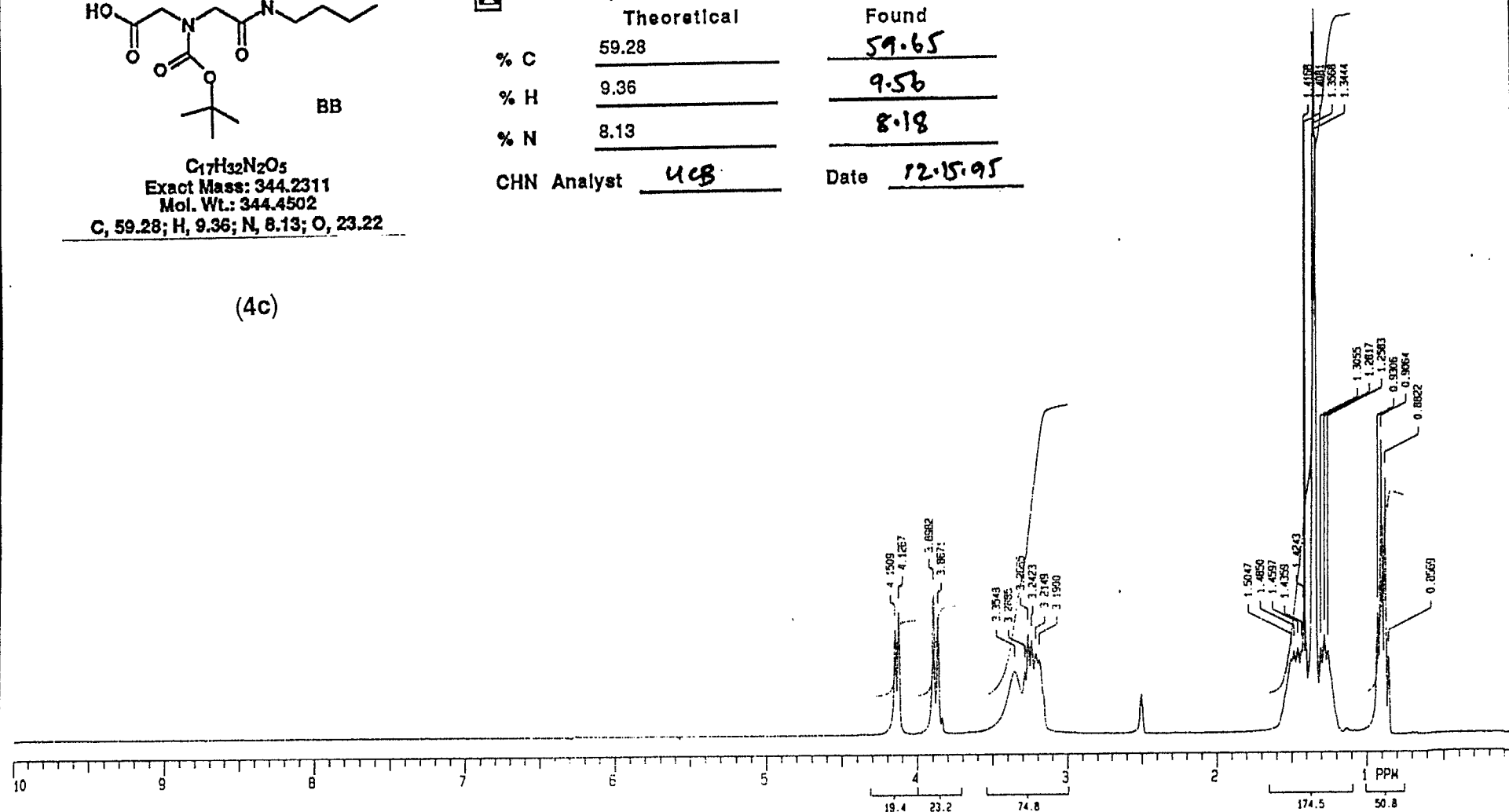
(4c)

☒ CHN Requested

	Theoretical
% C	59.28
% H	9.36
% N	8.13

CHN Analyst UCB

Found
59.65
9.56
8.18

Date 12-15-95

Observe
 Nucleus 1.000 Freq 300 MHz
 Spec Width 4500.5 Hz Offset 0 Hz
 Acq Time 1.778 sec Delay 1.000 sec
 Pulse Width 12.0 μ sec Transients 32

RECUPIE
 Nucleus 1.000 Offset -450.0 Hz
 Mode MW Power 20.0 dB
 Modulation Mode C Freq 200 Hz
 Pulse Width 1.0 μ sec Power Mode 1.0

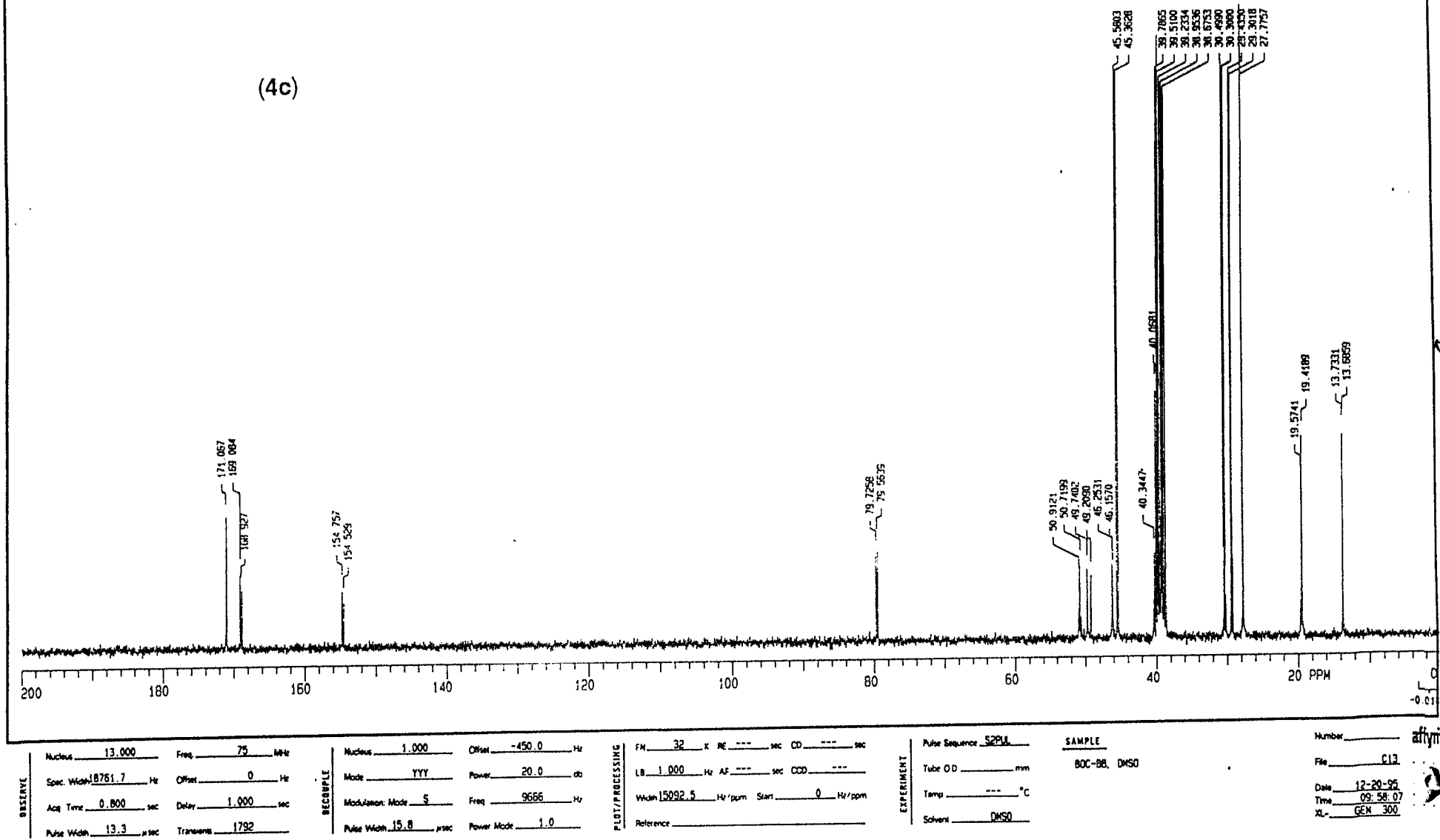
PLOT/PROCESSING
 F1 16 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD ---
 Width 3001.0 Hz/ppm Start 0 Hz/ppm
 Reference ---

EXPERIMENT
 Pulse Sequence S2PUL
 Tube OD --- mm
 Temp --- °C
 Solvent DMSO

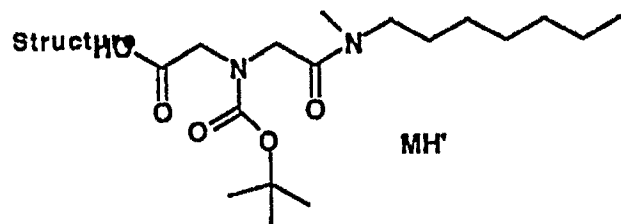
SAMPLE
 BOC-BB IN DMSO

Number --- affyma
 File --- H
 Date 12-20-95
 Time 09:52:41
 XL GEN 300

M-1608-8



M-1608-9



$C_{17}H_{32}N_2O_5$
 Exact Mass: 344.2311
 Mol. Wt.: 344.4502
 C, 59.28; H, 9.36; N, 8.13; O, 23.22

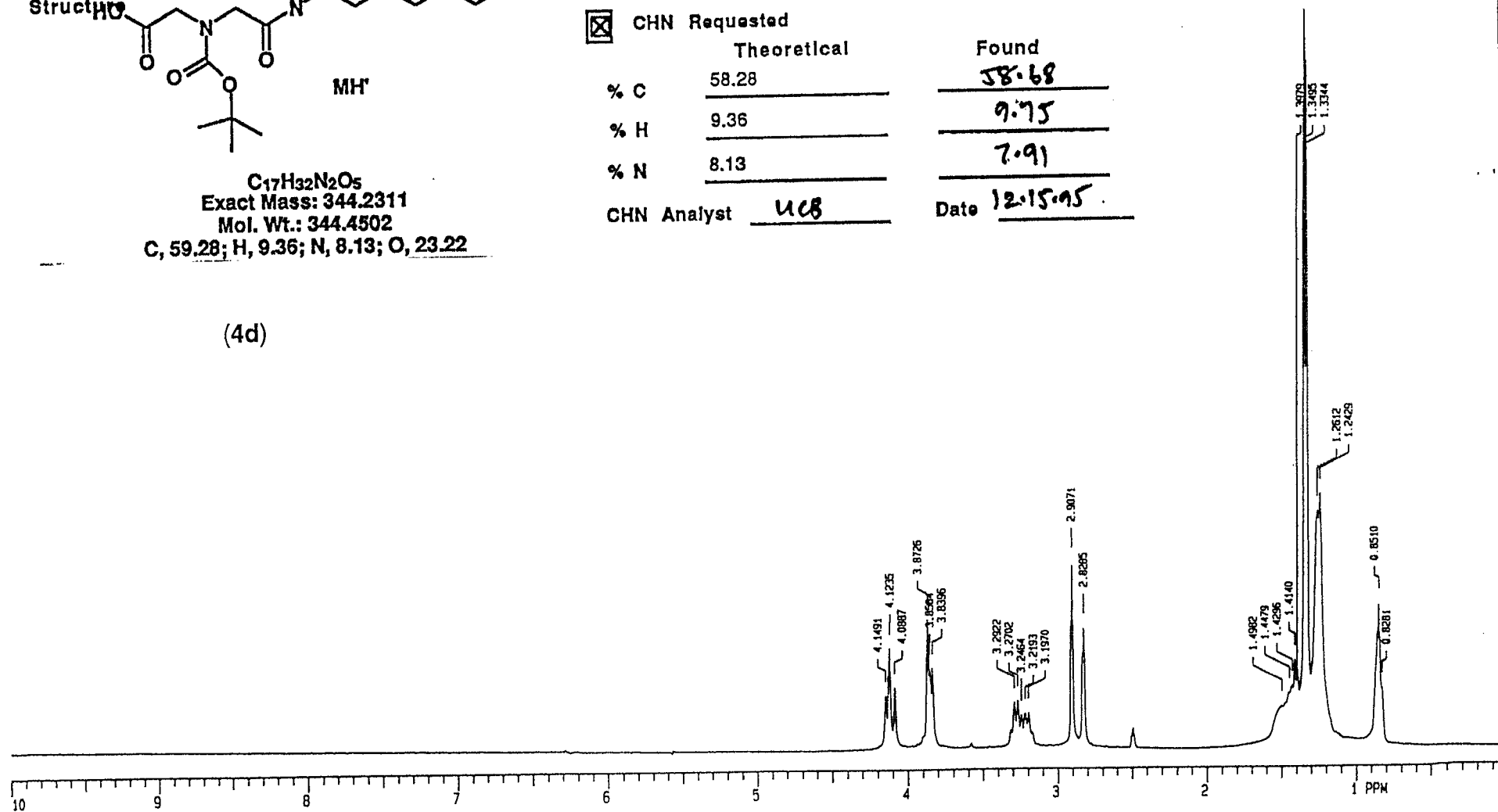
(4d)

☒ CHN Requested

	Theoretical	Found
% C	58.28	58.68
% H	9.36	9.75
% N	8.13	7.91

CHN Analyst UCB

Date 12-15-95



Observe
 Nucleus 1.000 Freq 300 MHz
 Spec Width 4500.5 Hz
 Acq Time 1.778 sec
 Pulse Width 12.0 μ sec
 Offset 0 Hz
 Delay 1.000 sec
 Transients 32

RECEIVE
 Nucleus 1.000 Offset -450.0 Hz
 Mode NNN Power 20.0 dB
 Modulation Mode C Freq 200 Hz
 Pulse Width 1.0 μ sec Power Mode 1.0

PLOT/PROCESSING
 F1 16 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD ---
 Width 3001.0 Hz/ppm Start 0 Hz/ppm
 Reference ---

EXPERIMENT
 Pulse Sequence SPUL
 Tube O.D. --- mm
 Temp --- °C
 Solvent DMSO

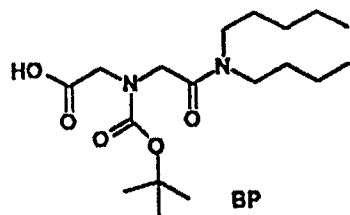
SAMPLE
 NAME Boc-MH'
 IN DMSO

Number --- affymax
 File H
 Date 12-08-95
 Time 08:24:44
 XL GEN 300

10.



M-1608-11



$C_{28}H_{44}N_2O_5$
Exact Mass: 358.2467
Mol. Wt.: 358.4770
C, 60.31; H, 9.56; N, 7.81; O, 22.32

(4e)

☒ CHN Requested
Theoretical

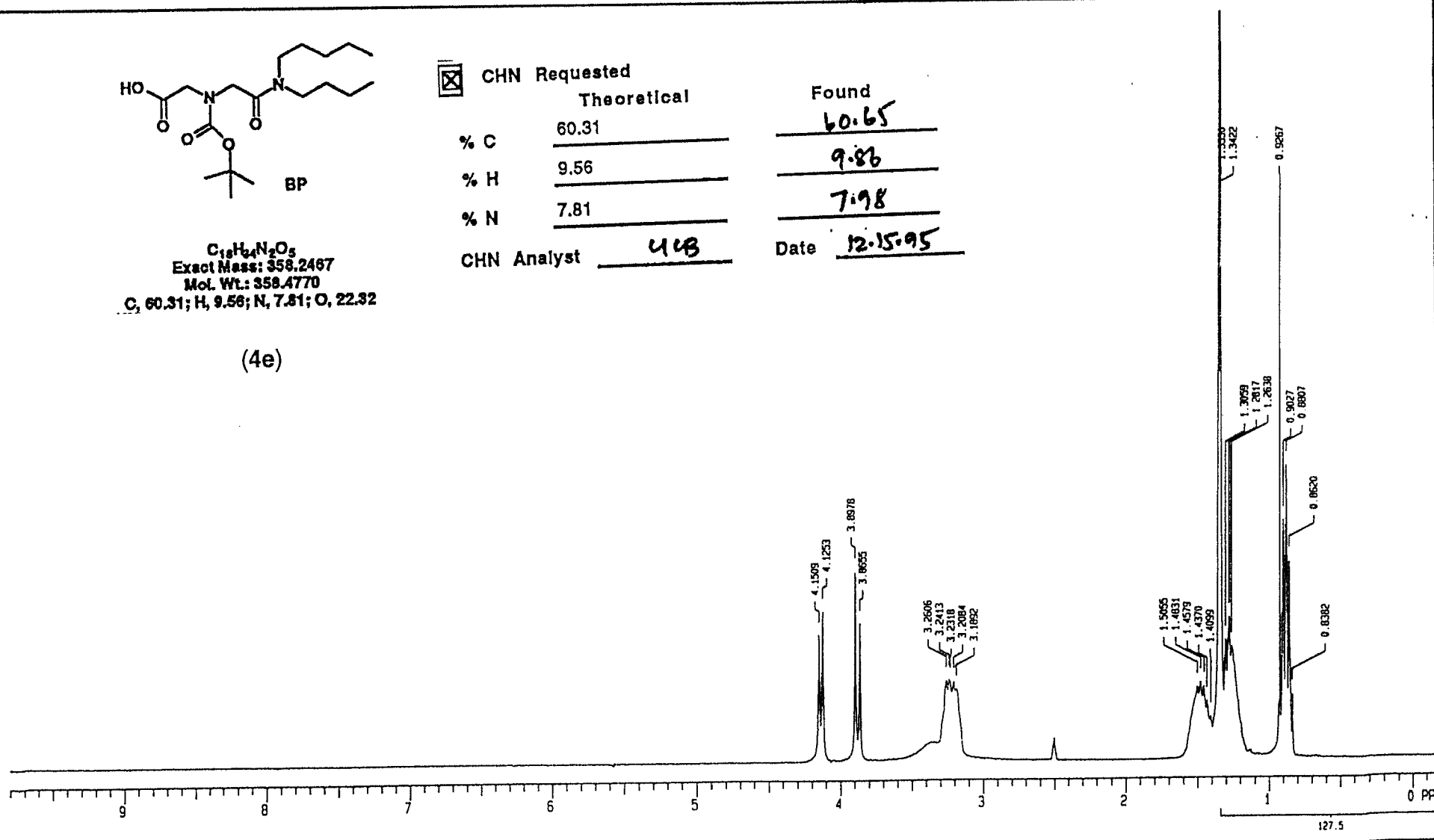
% C 60.31
% H 9.56
% N 7.81

CHN Analyst 418

Found

60.65
9.86
7.98

Date 12-15-95



RESERVE
Nucleus 1.000 Freq. 300 MHz
Spec. Width 4500.5 Hz Offset 0 Hz
Acq. Time 1.778 sec Delay 1.000 sec
Pulse Width 12.0 μsec Transmits 32

RECEIVE
Nucleus 1.000 Offset -450.0 Hz
Mode NNN Power 20.0 db
Modulation Mode C Freq. 200 MHz
Pulse Width μsec Power Mode 1.0

PLBT/PROCESSING
FN 16 K RE --- sec CD --- sec
LB --- Hz AF --- sec CCD ---
Width 3001.0 Hz/ppm Start -59.9 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence SPUL
Tube OD mm
Temp --- °C
Solvent DMSO

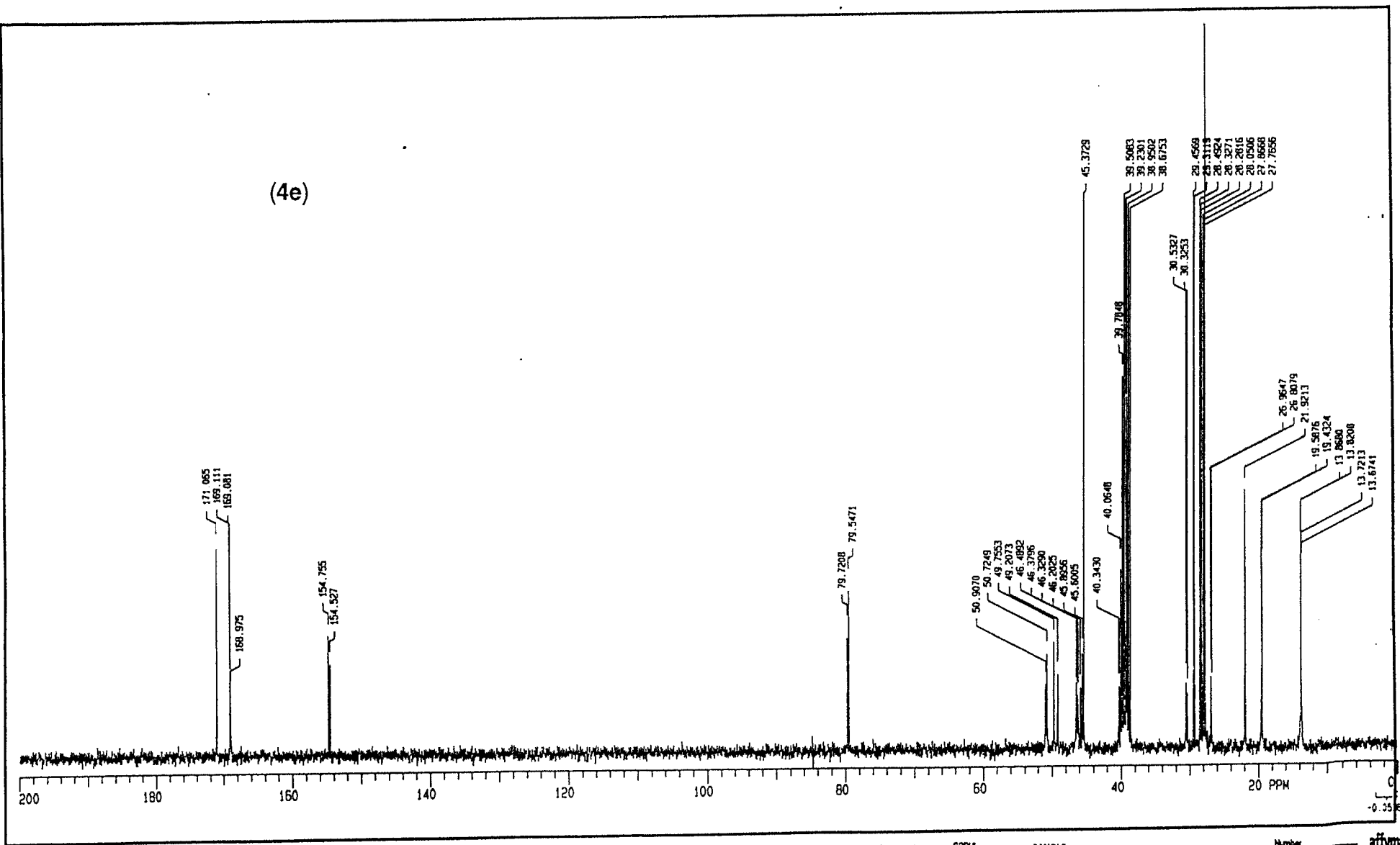
SAMPLE
BOC-C4-C5 IN DMSO

Number affymax
File H
Date 12-08-95
Time 11:24:01
XL GEM 300

12.

M-1608-12

(4e)



Nucleus 13.000 Freq 75 MHz
 Spec. Width 18761.7 Hz Offset 0 Hz
 Acq. Time 0.800 sec Delay 1.000 sec
 Pulse Width 13.3 µsec Transmits 448

Nucleus 1.000 Offset -450.0 Hz
 Mode YYY Power 20.0 dB
 Modulation Mode S Freq 9666 Hz
 Pulse Width 15.8 µsec Power Mode 1.0

PLOT/PROCESSING
 FN 32 K RE --- sec CD --- sec
 LB 1.000 Hz AF --- sec CCD ---
 Width 15092.5 Hz/ppm Start 0 Hz/ppm
 Reference ---

EXPERIMENT
 Pulse Sequence S2PL1
 Tube O.D. --- mm
 Temp --- °C
 Solvent DMSO

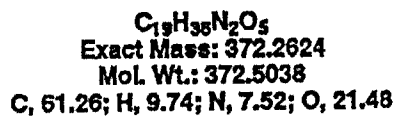
SAMPLE
 BOC-METHYLENE IN DMSO
 4-5

Number ---
 File C13
 Date 12-08-95
 Time 11:26:09
 XL GEH 300

affymax

A

13



☒ CHN Requested

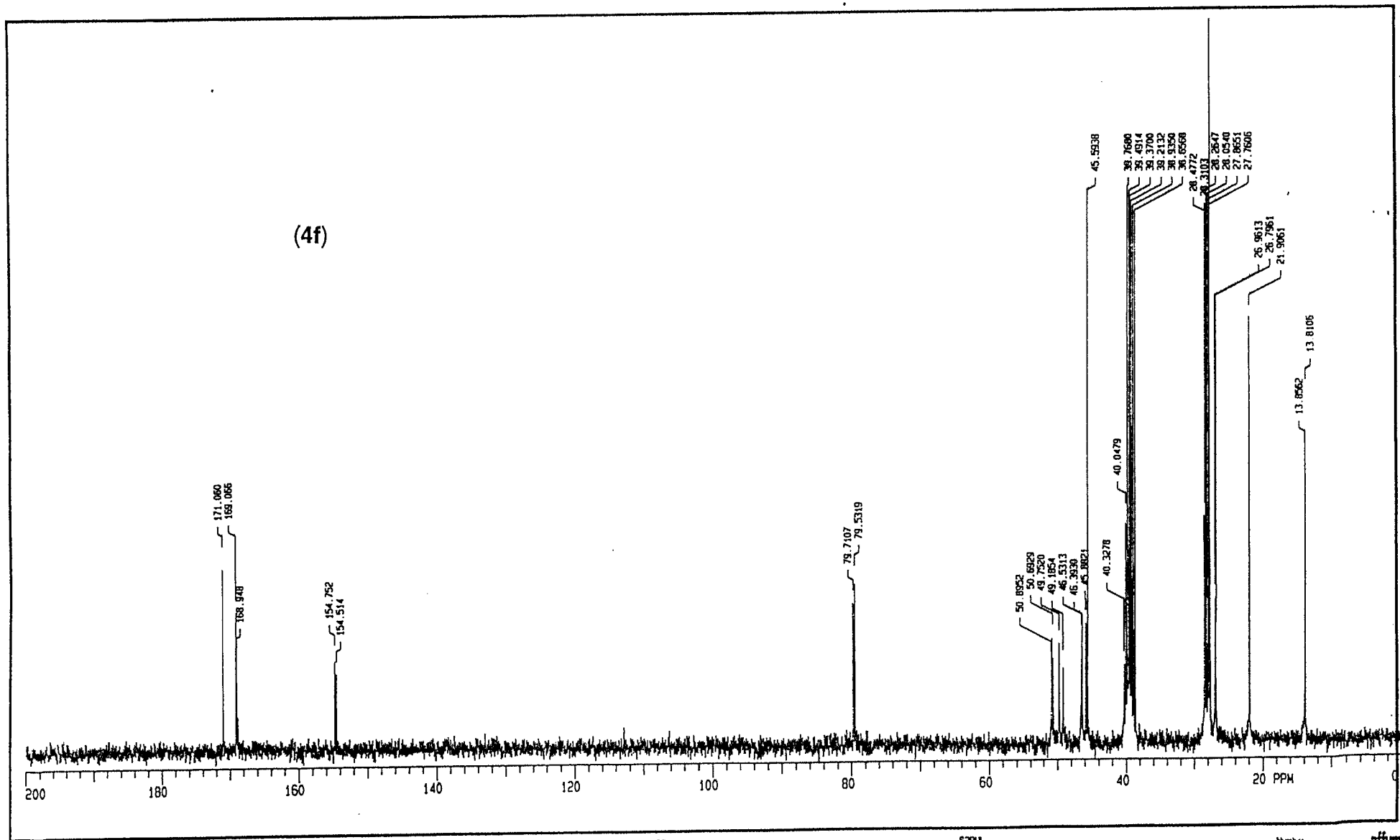
CHN Analyst 418

Date 12-15-95



Number _____
File _____ H
Date 12-07-95
Time 19:04:23
XL- GEM 300

M-1608-14



OBSERVE
Nucleus 13.000 Freq 75 MHz
Spec. Width 18761.7 Hz Offset 0 Hz
Acq. Time 0.800 sec Delay 1.000 sec
Pulse Width 13.3 µsec Transmits 448

RECEIVE
Nucleus 1.000 Offset -450.0 Hz
Mode YYY Power 20.0 db
Modulation Mode S Freq 9666 Hz
Pulse Width 15.8 µsec Power Mode 1.0

PLOT/PROCESSING
FN 32 K RE --- sec CD --- sec
LB 1.000 Hz AF --- sec CCD --- sec
Width 15092.5 Hz/ppm Start 0 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence S2PUL
Tube O.D. mm
Temp --- °C
Solvent DMSO

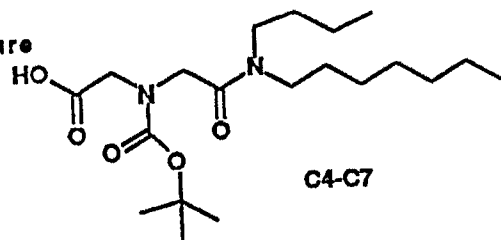
SAMPLE
BOC-PP, DMSO

Number
File C13
Date 12-07-95
Time 19:12:31
XL GEN 300

affymax

15.

Structure



C4-C7

$C_{20}H_{35}N_2O_5$
Exact Mass: 386.2780
Mol. Wt.: 386.5306

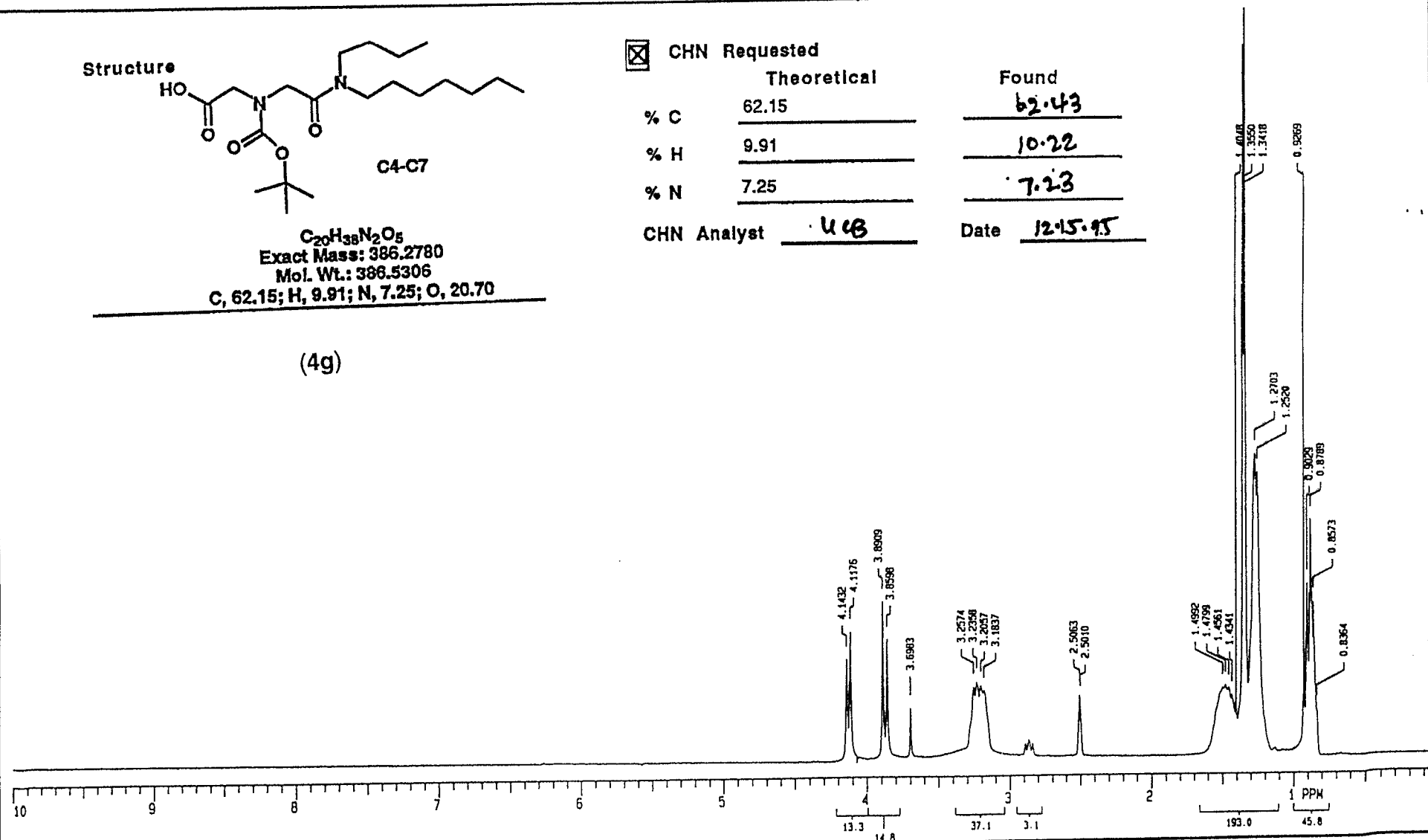
C, 62.15; H, 9.91; N, 7.25; O, 20.70

(4g)



CHN Requested

	Theoretical	Found
% C	62.15	62.43
% H	9.91	10.22
% N	7.25	7.23
CHN Analyst	UCB	Date 12-15-95



RESERVE
Nucleus 1.000 Freq 300 MHz
Spec Width 4500.5 Hz
Acq Time 1.778 sec
Pulse Width 12.0 µsec
Offset 0 Hz
Delay 1.000 sec
Transients 32

RECOUPLE

Nucleus 1.000 Offset -450.0 Hz
Mode NNN Power 20.0 dB
Modulation Mode C Freq 200 Hz
Pulse Width µsec Power Mode 1.0

PLOT/PROCESSING

FN 16 K RE --- sec CD --- sec
LB --- Hz AF --- sec CCD ---
Width 3001.0 Hz/ppm Start 0 Hz/ppm
Reference

EXPERIMENT

Pulse Sequence SPB1
Tube OD --- mm
Temp --- °C
Solvent DMSO

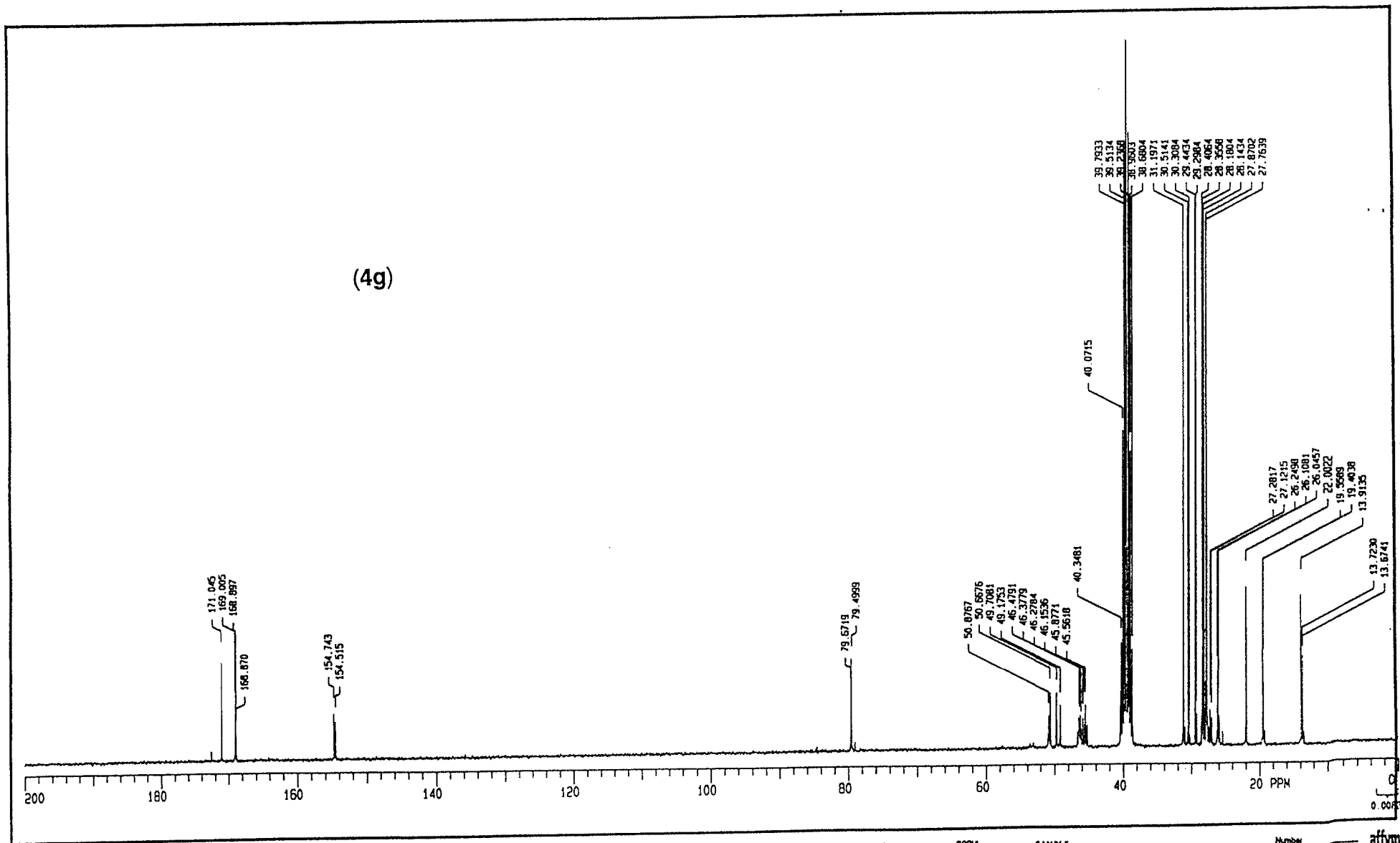
SAMPLE

80C-C4-C7 IN DMSO

Number
File
Date 12-08-95
Time 20:40:03
XL GEN 300

affymax

M-1608-16



OBSERVE

Nucleus 13.000 Freq 75 MHz
 Spec. Width 18761.7 Hz Offset 0 Hz
 Acq. Time 0.800 sec Delay 1.000 sec
 Pulse Width 13.3 μ sec Transients 23360

RECEIVE

Nucleus 1.000 Offset -450.0 Hz
 Mode YYY Power 20.0 dB
 Modulation Mode S Freq 9666 Hz
 Pulse Width 15.8 μ sec Power Mode 1.0

PLOT/PROCESSING

FM 32 K RE --- sec CD --- sec
 LB 1.000 Hz AF --- sec CCD ---
 Width 15092.5 Hz/ppm Start 0 Hz/ppm
 Reference ---

EXPERIMENT

Pulse Sequence S2P11
 Tube OD --- mm
 Temp --- °C
 Solvent DMSO

SAMPLE

BOC-C4-C7, DMSO

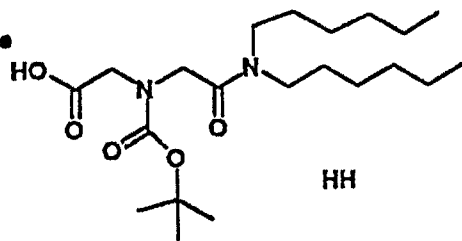
Number ---
 File C13
 Date 12-08-95
 Time 20:45:27
 XL GFH 300

affymax

17.

M-1608-17

Structure



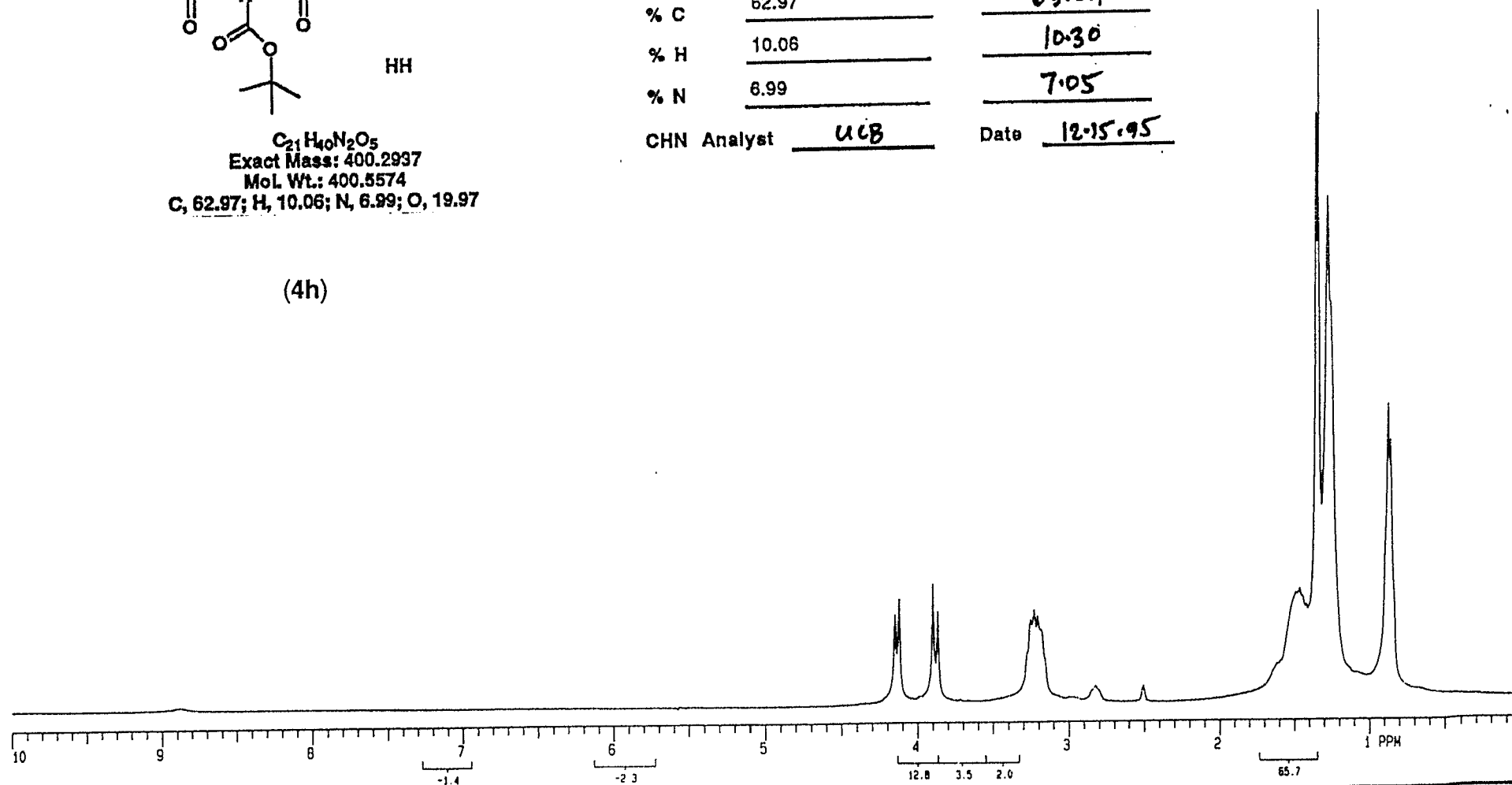
$C_{21}H_{40}N_2O_5$
 Exact Mass: 400.2937
 Mol. Wt.: 400.5574
 C, 62.97; H, 10.06; N, 6.99; O, 19.97

(4h)



CHN Requested

	Theoretical	Found
% C	62.97	63.04
% H	10.06	10.30
% N	6.99	7.05
CHN Analyst	UCB	Date 12-15-95



OBSERVE
 Nucleus 1.000 Freq 300 MHz
 Spec. Width 4500.5 Hz Offset 0 Hz
 Acq. Time 1.778 sec Delay 1.000 sec
 Pulse Width 12.0 µsec Transients 32

RECEIVE
 Nucleus 1.000 Offset -450.0 Hz
 Mode NMR Power 20.0 db
 Modulation Mode C Freq 200 Hz
 Pulse Width µsec Power Mode 1.0

PLT/PROCESSING
 F1 16 K RE --- sec CD --- sec
 L1 --- Hz AF --- sec CCD ---
 Width 3001.0 Hz/ppm Start 0 Hz/ppm
 Reference

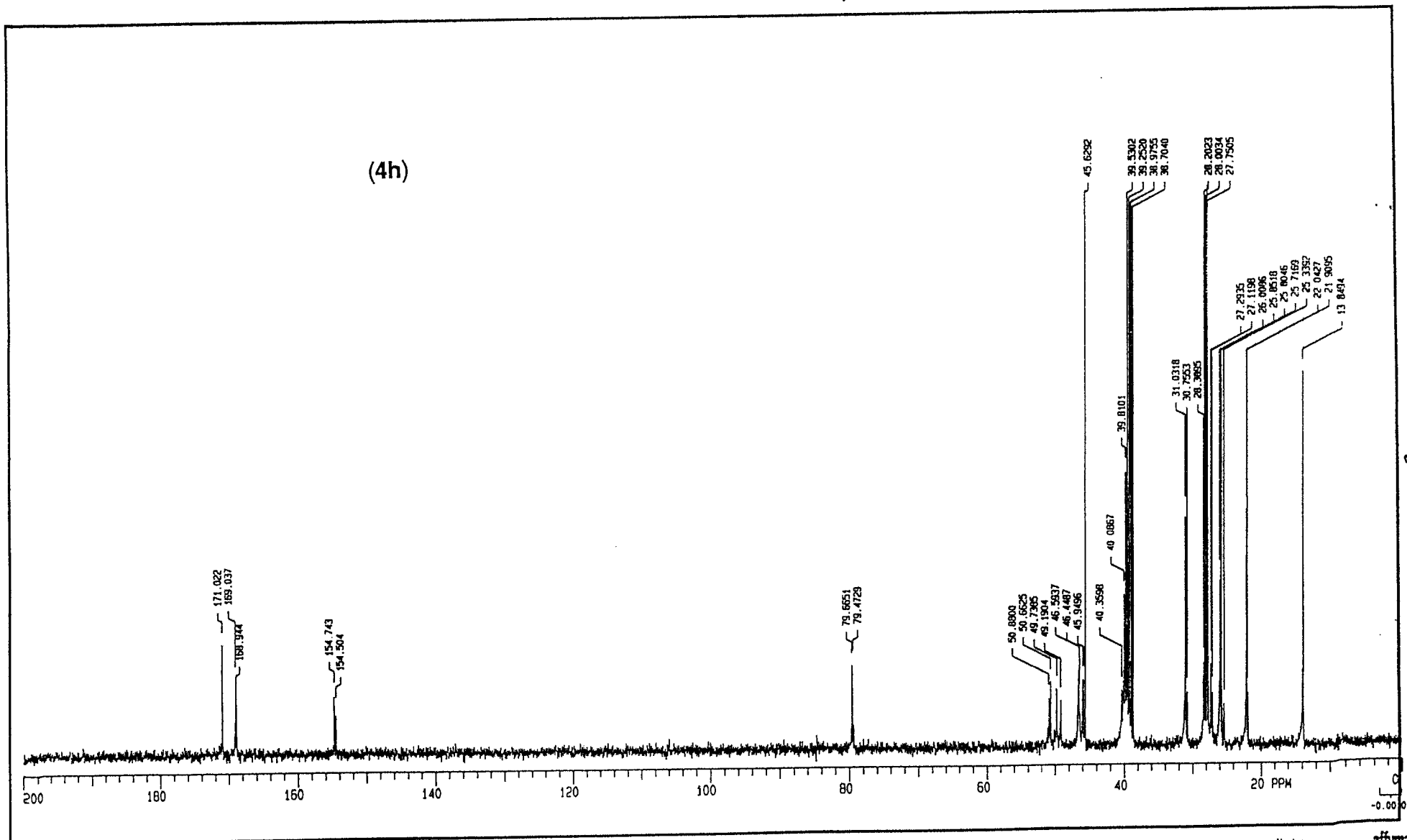
EXPERIMENT
 Pulse Sequence SPM
 Tube O.D. mm
 Temp --- °C
 Solvent DMSO

SAMPLE
 BOC-HH IN DMSO

Number affymax
 File H
 Date 12-08-95
 Time 13:11:30
 XL- GEN 300

18

M-1608-18



RESERVE

Nucleus	13.000	Freq	75	MHz	
Spec Width	18761.7	Hz	Offset	0	Hz
Acq Time	0.800	sec	Delay	1.000	sec
Pulse Width	13.3	μsec	Transmit	448	

RECEIVED

Nucleus	1.000	Offset	-450.0	Hz	
Mode	YYY	Power	20.0	db	
Mod/Spec Mode	S	Freq	9666	Hz	
Pulse Width	15.8	μsec	Power Mode	1.0	

PLOT/PROCESSING

FN	32	K	RE	---	sec	CD	---	sec
LS	1.000	Hz	AF	---	sec	OCD	---	sec
Width	15092.5	Hz/ppm	Start	0	Hz/ppm			
Reference								

EXPERIMENT

Pulse Sequence	S2PUL	
Tube O.D.	mm	
Temp	---	°C
Solvent	DMSO	

SAMPLE

BOC-HI IN DMSO

Number

File C13

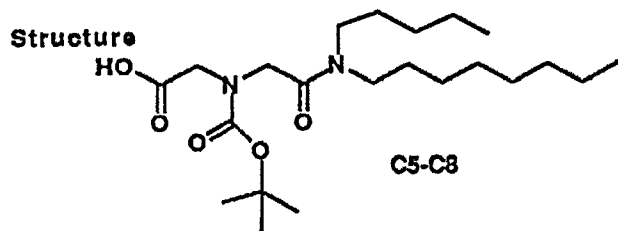
Date 12-08-95

Time 13:13:58

XL GEM 300

affymax

M-1608-19



$C_{22}H_{42}N_2O_5$
 Exact Mass: 414.3093
 Mol. Wt.: 414.5842
 C, 63.74; H, 10.21; N, 6.76; O, 19.30

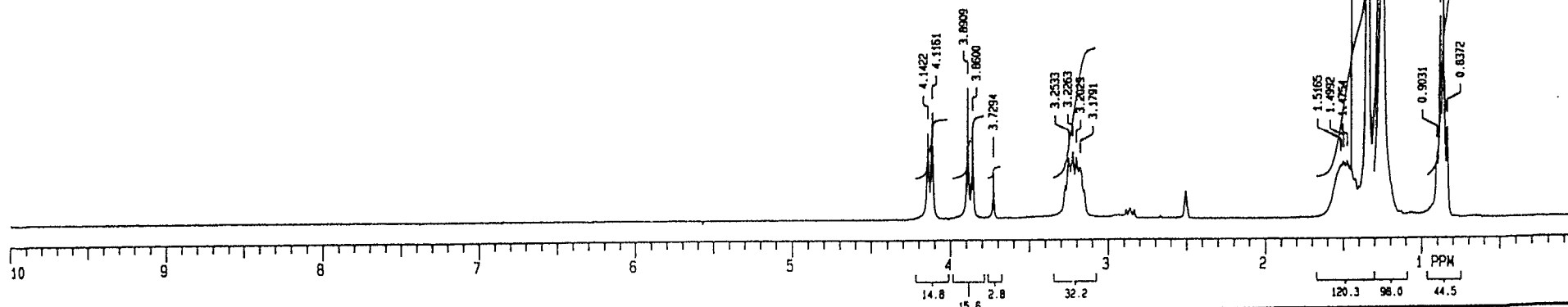
(4i)

☒ CHN Requested

	Theoretical	Found
% C	63.74	63.74
% H	10.21	10.57
% N	6.76	6.64

CHN Analyst u13

Found
 63.74
 10.57
 6.64
 Date 10.15.95



Observe
 Nucleus 1.000 Freq 300 MHz
 Spec. Width 4500.5 Hz Offset 0 Hz
 Acq. Time 1.778 sec Delay 1.000 sec
 Pulse Width 12.0 μsec Transmits 32

RECEIVE
 Nucleus 1.000 Offset -450.0 Hz
 Mode NMR Power 20.0 dB
 Modulation Mode F Freq 200 Hz
 Pulse Width μsec Power Mode 1.0

PLDT/PROCESSING
 FN 15 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD ---
 Width 3001.0 Hz/ppm Start 0 Hz/ppm
 Reference

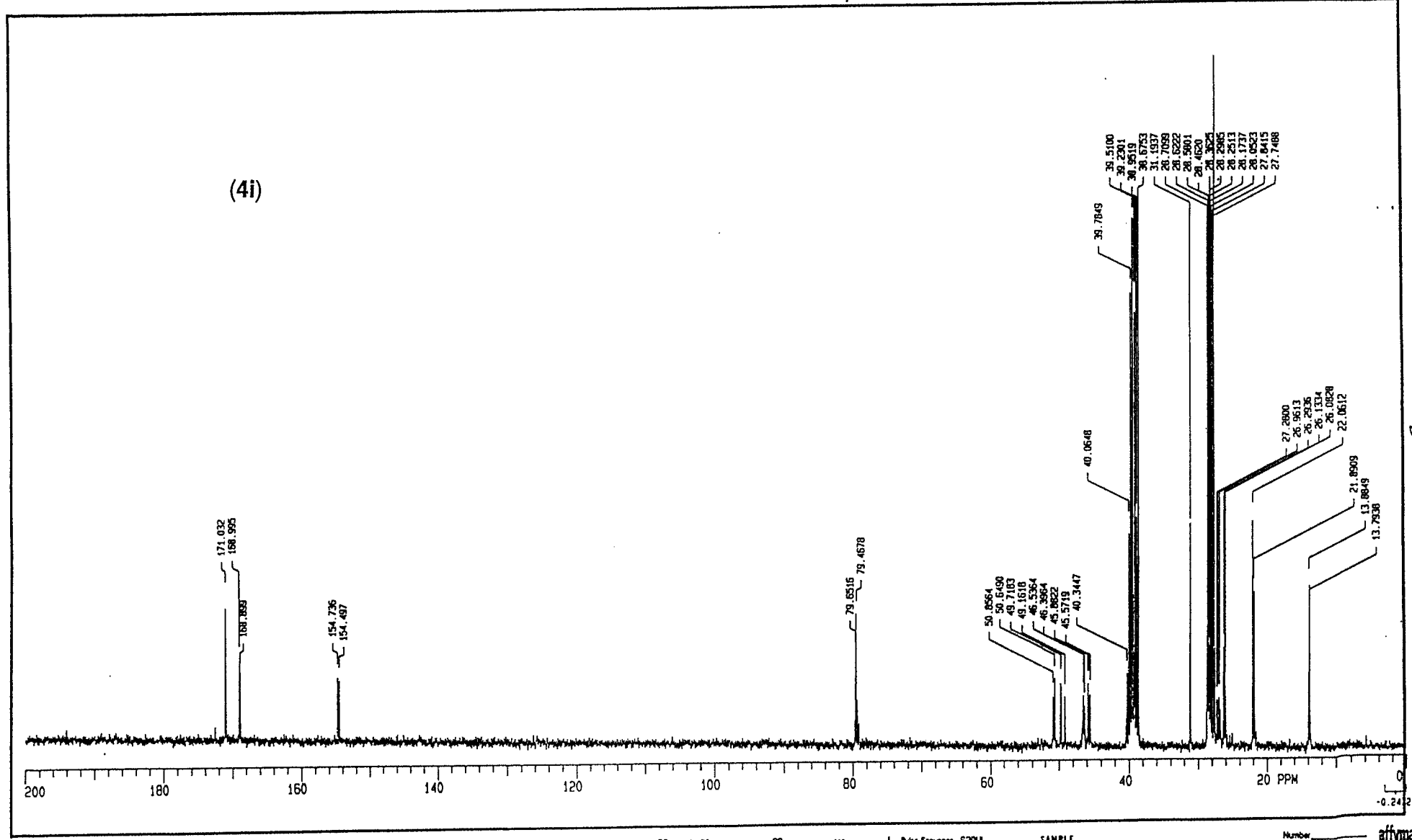
EXPERIMENT
 Pulse Sequence 52911
 Tube O.D. mm
 Temp --- °C
 Solvent DMSO

SAMPLE
 BOC-C5-C8 DMSO

Number affymax
 File H
 Date 12-11-95
 Time 08:35:17
 XL GEN 300

20.

M-1608-20



OBSERVE
 Nucleus 13.000 Freq 75 MHz
 Spec. Width 18751.7 Hz
 Acq. Time 0.800 sec
 Pulse Width 13.3 μ sec
 Transients 1344
 Nucleus 1.000 Offset -450.0 Hz
 Mode YYY Power 20.0 dB
 Modulation Mode S Freq 9566 Hz
 Pulse Width 15.8 μ sec Power Mode 1.0
 PLOT/PROCESSING
 FN 32 K RE --- sec CD --- sec
 LB 1.000 Hz AF --- sec CCD ---
 Width 15092.5 Hz/ppm Start 0 Hz/ppm
 Reference ---

EXPERIMENT
 Pulse Sequence S2BLL
 Tube OD --- mm
 Temp --- °C
 Solvent DMSO

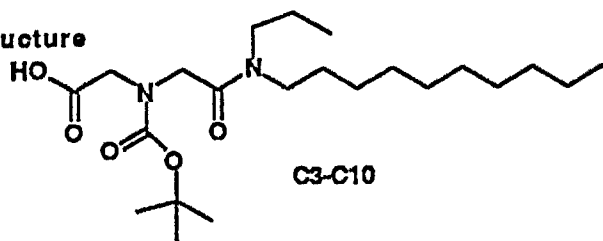
SAMPLE
 BOC-C5-C8, DMSO

Number ---
 File C13
 Date 12-11-95
 Time 08:39:49
 XL GEW 300

affymax

M-1608-21

Structure



C₂₂H₄₂N₂O₅
 Exact Mass: 414.3093
 Mol. Wt.: 414.5842
 C, 63.74; H, 10.21; N, 6.76; O, 19.30

(4j)



CHN Requested

Theoretical

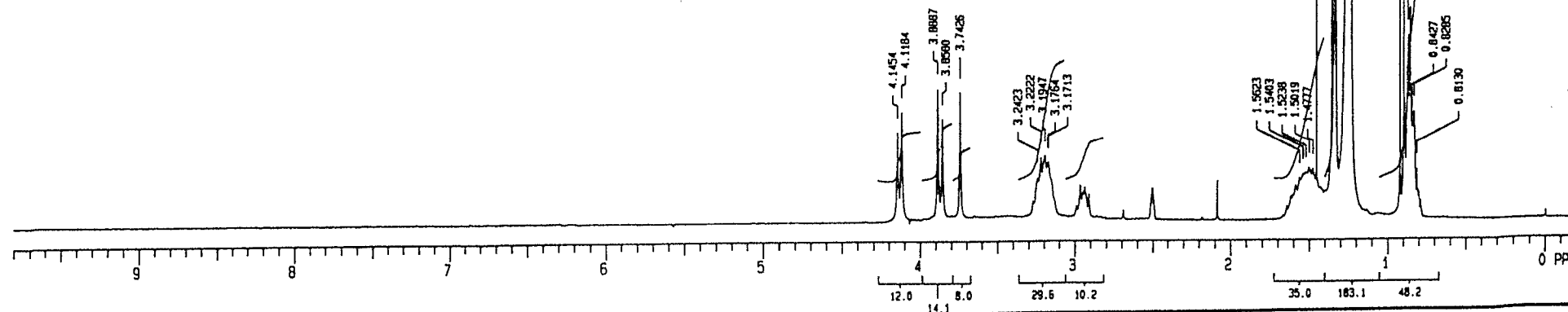
% C 63.74

% H 10.21

% N 6.76

CHN Analyst 4CB

Found

63.7010.556.83Date 12.15.95

RESERVE
 Nucleus 1.000 Freq. 300 MHz
 Spec. Width 4500.5 Hz Offset 0 Hz
 Acq. Time 1.778 sec Delay 1.000 sec
 Pulse Width 12.0 µsec Transients 32

RECEIVED
 Nucleus 1.000 Offset -450.0 Hz
 Mode MHW Power 20.0 db
 Modulation Mode C Freq. 200 Hz
 Pulse Width µsec Power Mode 1.0

PLOT/PROCESSING
 F1 15 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD ---
 Width 3001.0 Hz/ppm Start -59.9 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence S2PUL
 Tube OD mm
 Temp --- °C
 Solvent DMSO

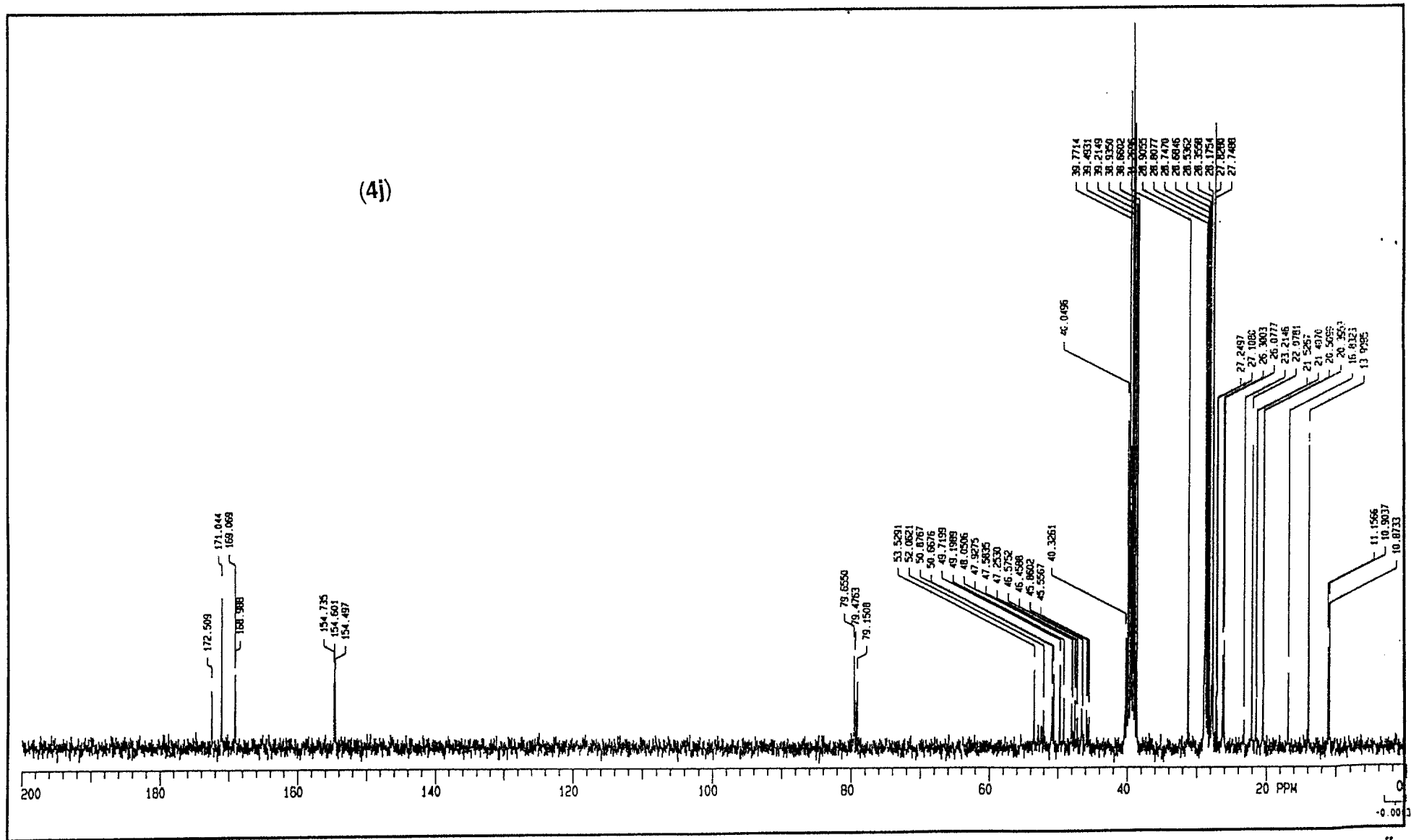
SAMPLE
 BOC-C3-C10 IN DMSO

Number
 File H
 Date 12-11-95
 Time 13:49:27
 XL GEN 300

affymax

27

M-1608-dd



Nucleus 13.000 Freq 75 MHz
 Spec Width 18761.7 Hz Offset 0 Hz
 Acq Time 0.800 sec Delay 1.000 sec
 Pulse Width 13.3 μ sec Transm 0.32

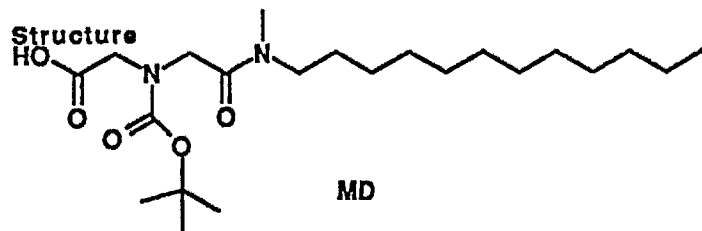
Nucleus 1.000 Offset -450.0 Hz
 Mode YYY Power 20.0 db
 Modulation Mode S Freq 9666 Hz
 Pulse Width 15.8 μ sec Power Mode 1.0

FM 32 K RE --- sec CD --- sec
 LB 1.000 Hz AF --- sec CCD ---
 Width 15092.5 Hz/ppm Start 0 Hz/ppm
 Reference ---

Pulse Sequence S2911
 Tube O.D. --- mm
 Temp --- °C
 Solvent DMSO

SAMPLE
 BOC-C3-C10 DMSO

Number --- affymax
 File C13
 Date 12-11-95
 Time 13:54:29
 XL GFH 300



C₂₂H₄₂N₂O₅
Exact Mass: 414.3093
Mol. Wt.: 414.5842
C, 63.74; H, 10.21; N, 6.76; O, 19.30

(4k)

☒ CHN Requested

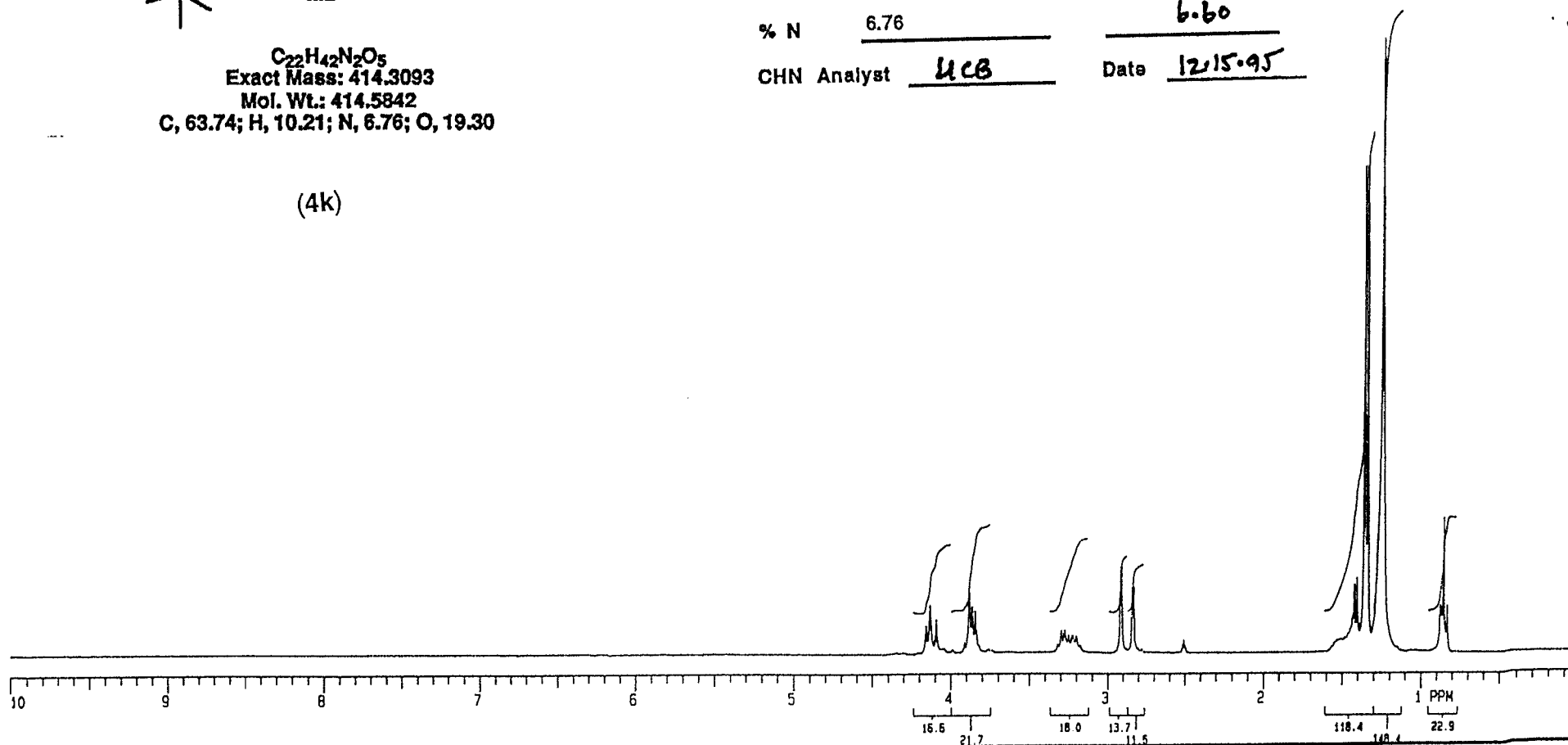
	Theoretical
% C	63.74
% H	10.21
% N	6.76

CHN Analyst UCB

Found

$$\begin{array}{r} 64.11 \\ 10.58 \\ \hline 6.60 \end{array}$$

Date 12/15/95



Nucleus 1.000 Freq. 300 MHz
 Spec. Width 4500.5 Hz Offset 0 Hz
 Acq. Time 1.778 sec Delay 1.000 sec
 Pulse Width 12.0 μ sec Transients 32

RECEIVER

Nucleus	1.000	Offset	-450.0	Hz
Mode	NNH	Power	20.0	dB
Modulation Mode	C	Freq	200	Hz
Pulse Width	μsec	Power Mode	1.0	

FN 16 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD ---
 Width 3001.0 Hz/ppm Start 0 Hz/ppm
 Reference _____

EXPERIMENT

Pulse Sequence S2PU

Tube OD _____ mm

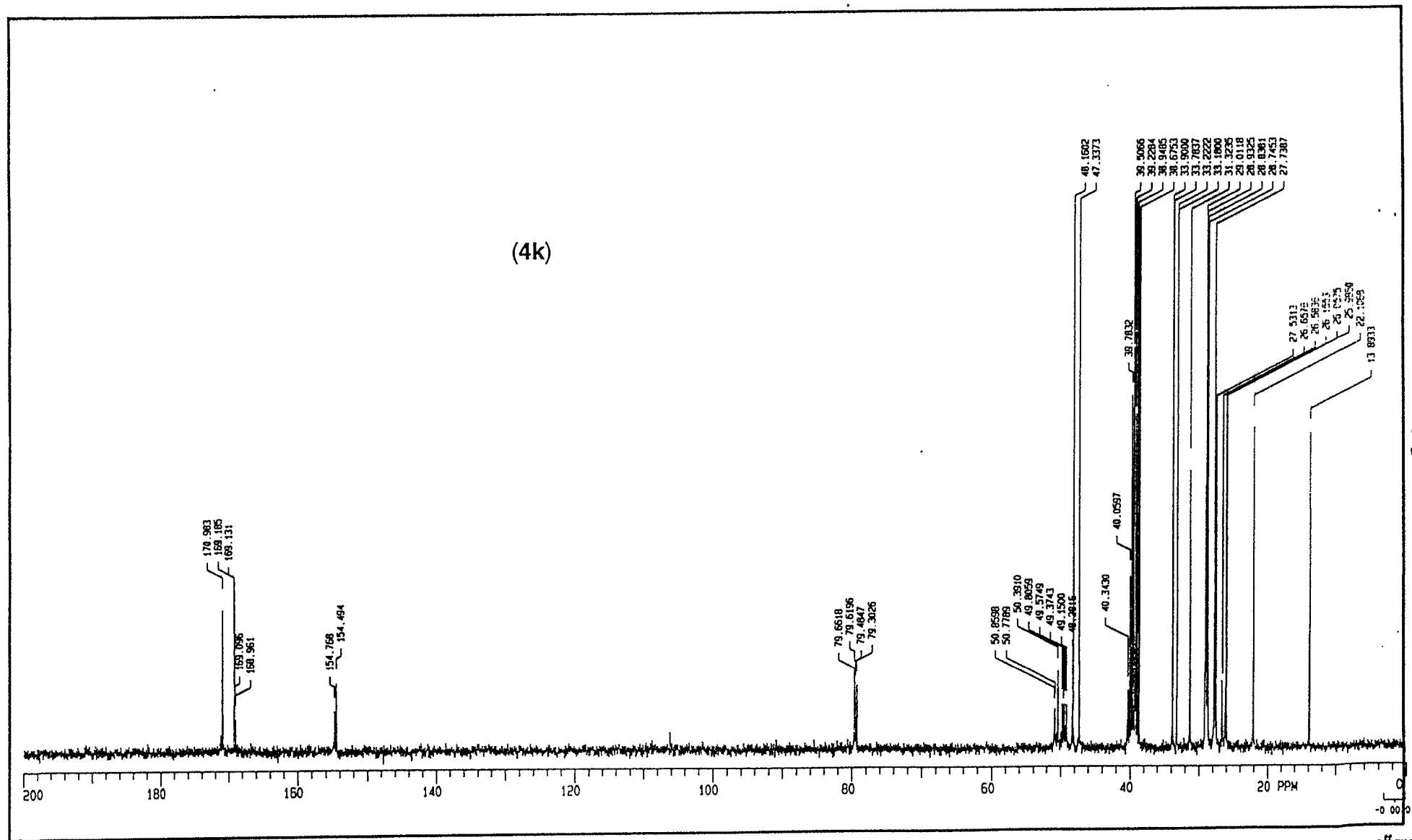
Temp _____ °C

Solvent DMSO

SAMPLE
BOC-ND IN DMSO

Number _____
File _____ H
Date 12-08-95
Time 14:31:08
XL- GEM 300

M-1608-24



Nucleus 13.000 Freq 75 MHz
 Spec. Widen 18761.7 Hz Offset 0 Hz
 Acq. Time 0.500 sec Delay 1.000 sec
 Pulse Width 13.3 μ sec Transmits 512

Nucleus 1.000 Offset -450.0 Hz
 Mode YYY Power 20.0 db
 Modulation Mode S Freq 9666 Hz
 Pulse Width 15.8 μ sec Power Mode 1.0

FM 32 K RE --- sec CD --- sec
 LB 1.000 Hz AF --- sec CCD --- sec
 Width 15092.5 Hz/ppm Start 0 Hz/ppm
 Reference ---

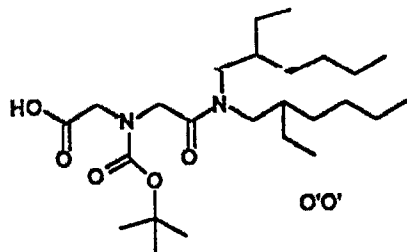
Pulse Sequence S2PUL
 Tube O.D. --- mm
 Temp --- °C
 Solvent DMSO

SAMPLE
 BOC-HO IN DMSO

Number affymax
 File C13
 Date 12-08-95
 Time 14:33:18
 XL GEM 300

25

M-1608-25



$C_{25}H_{43}N_2O_5$
Exact Mass: 456.3563
Mol. Wt.: 456.6646
C, 65.75; H, 10.59; N, 6.13; O, 17.52

(4I)

☒ CHN Requested

Theoretical

% C

65.75

% H

10.59

% N

6.13

CHN Analyst

u18

Found

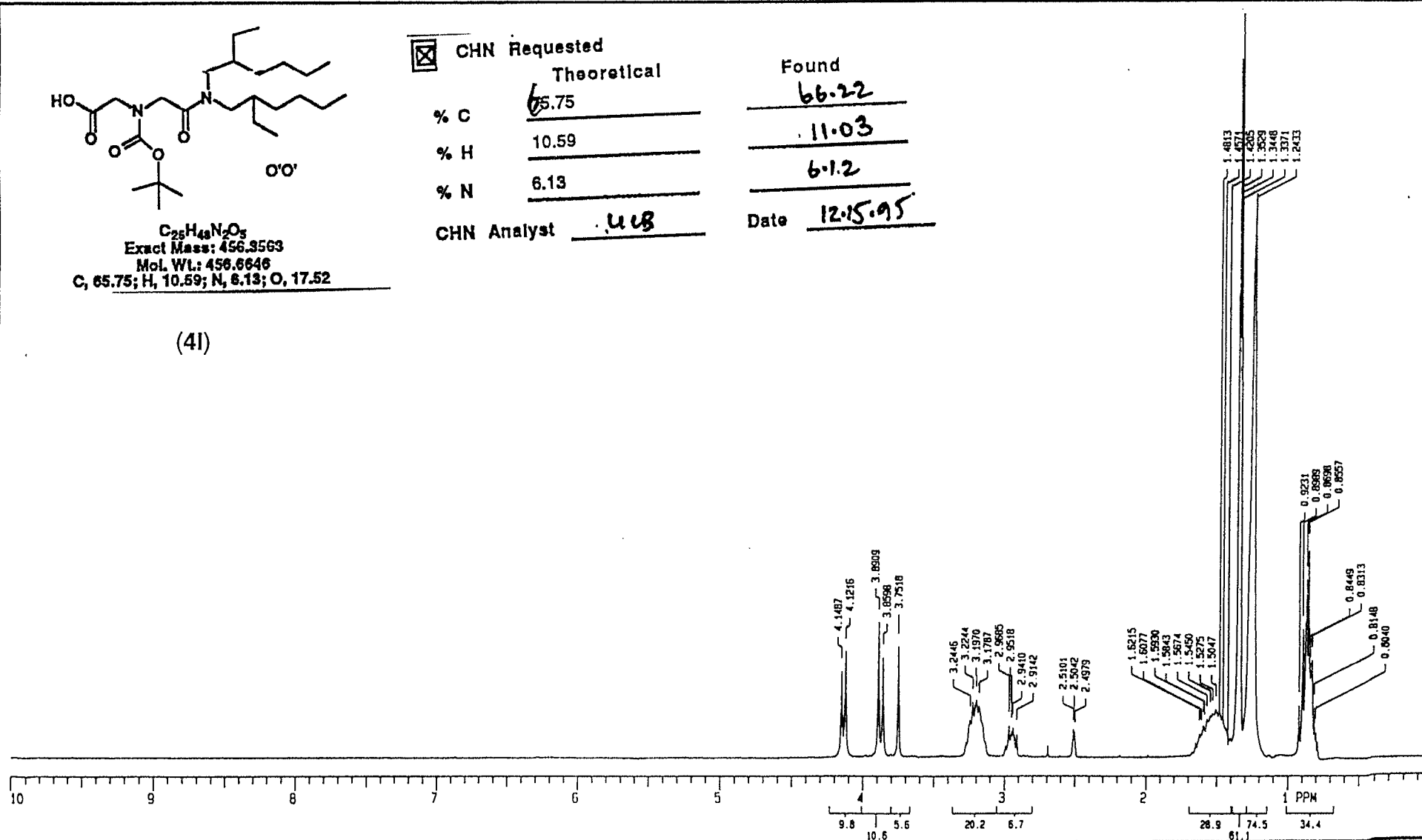
66.22

11.03

6.12

Date

12.15.95



Observe
Nucleus 1.000 Freq 300 MHz
Solv. Wdth 4500.5 Hz
Acq. Time 1.778 sec
Pulse Width 12.0 μ sec
Transients 32

RECEIVED

Nucleus 1.000 Offset -450.0 Hz
Mode NNN Power 20.0 dB
Modulation Mode C Freq 200 MHz
Pulse Width 12.0 μ sec Power Mode 1.0

PLOT/PROCESSING

FM 16 K RE --- sec CD --- sec
LB --- Hz AF --- sec COD ---
Width 3001.0 Hz/ppm Start 0 Hz/ppm
Reference ---

EXPERIMENT

Pulse Sequence S2PL
Tube OD --- mm
Temp --- °C
Solvent DMSO

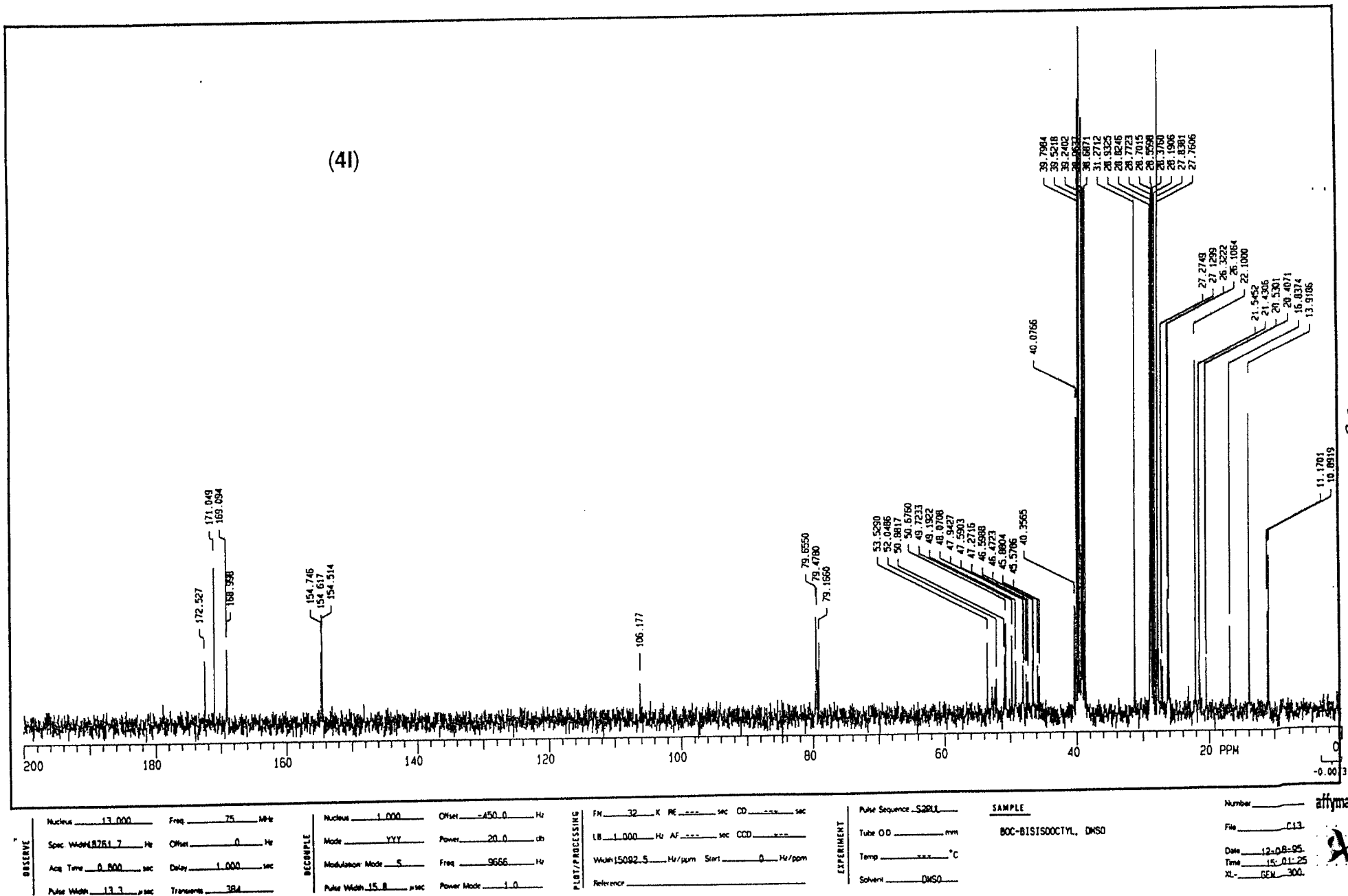
SAMPLE

BOC-BISISOOCTYL, DMSO

Number --- affymax
File --- H
Date 12-08-95
Time 14:56:38
XL GEH 300

26.

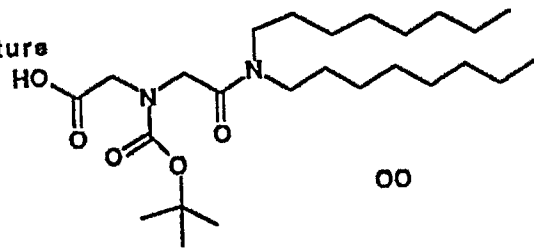
M-1608-26



27

M-1608-27

Structure



$C_{25}H_{48}N_2O_5$
 Exact Mass: 456.3563
 Mol. Wt.: 456.6646
 C, 65.75; H, 10.59; N, 6.13; O, 17.52

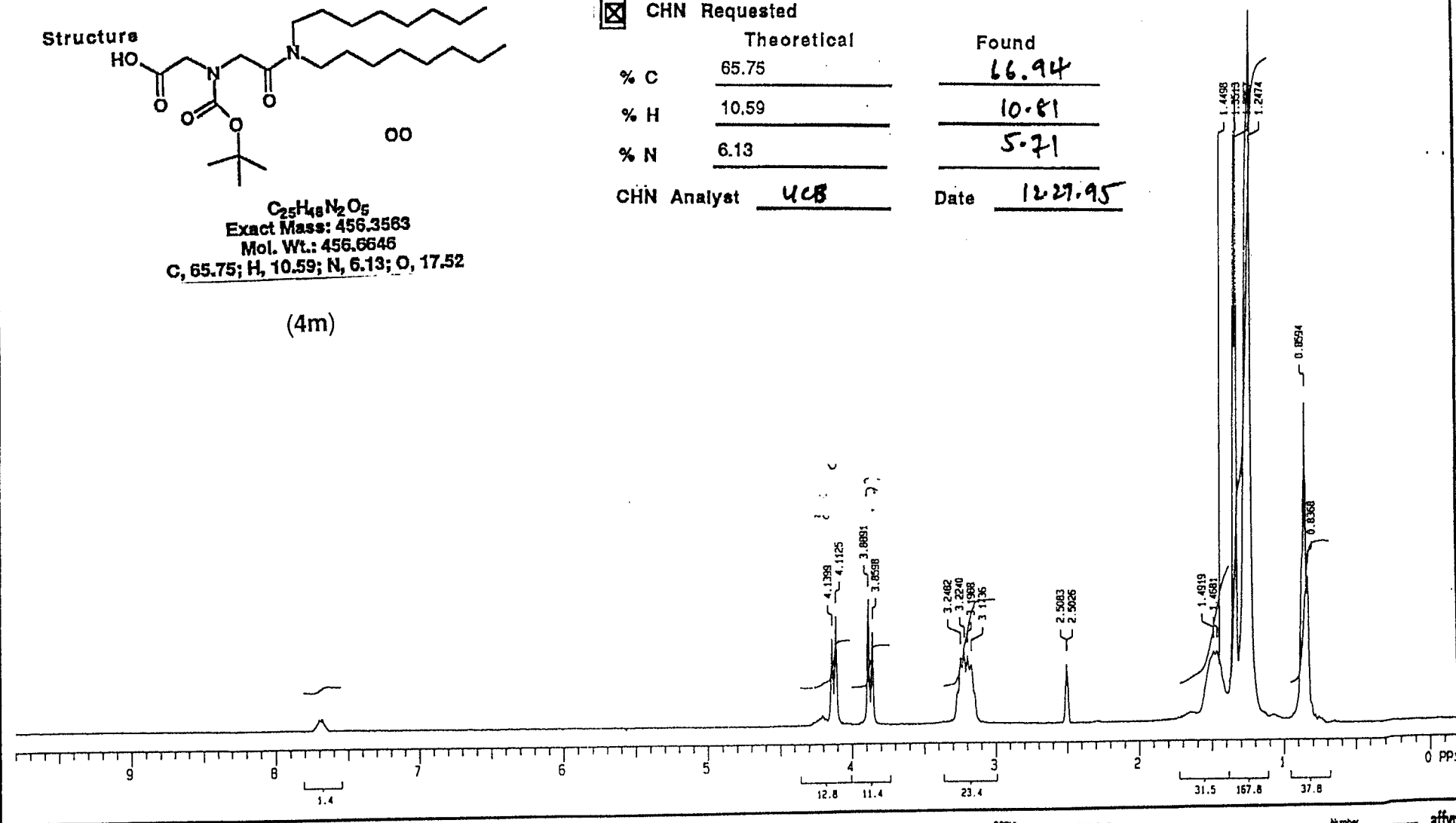
(4m)

☒ CHN Requested

	Theoretical
% C	65.75
% H	10.59
% N	6.13

Found

% C	66.94
% H	10.81
% N	5.71

CHN Analyst UCBDate 12-27-95

OBSERVE
 Nucleus 1.000 Freq 300 MHz
 Spec. Width 2500.5 Hz
 Acq. Time 1.778 sec
 Pulse Width 12.0 µsec
 Offset 0 Hz
 Delay 1.000 sec
 Transmits 32

RECEIVE
 Nucleus 1.000 Offset -450.0 Hz
 Mode HNH Power 20.0 dB
 Modulation Mode C Freq 200 Hz
 Pulse Width 1.0 µsec
 Power Mode 1.0

PLOT/PROCESSING
 F1 16 K RE --- sec CD --- sec
 L1 --- Hz AF --- sec CCD --- sec
 Width 3001.0 Hz/ppm Start -59.9 Hz/ppm
 Reference ---

EXPERIMENT
 Pulse Sequence S2P1
 Tube OD --- mm
 Temp --- °C
 Solvent DMSO

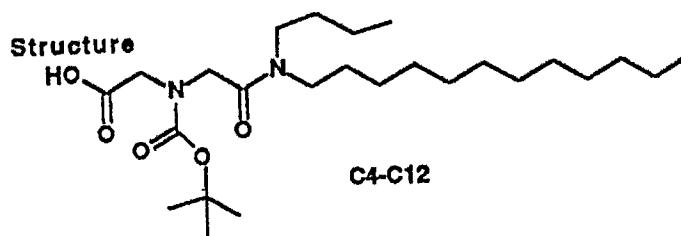
SAMPLE
 BOC-DO IN DMSO

Number --- affymax
 File H
 Date 12-07-95
 Time 10:54:55
 XL GEN 300

28.



M-1608-29



$C_{25}H_{48}N_2O_5$
 Exact Mass: 456.3563
 Mol. Wt.: 456.6646
 C, 65.75; H, 10.59; N, 6.13; O, 17.52

(4n)

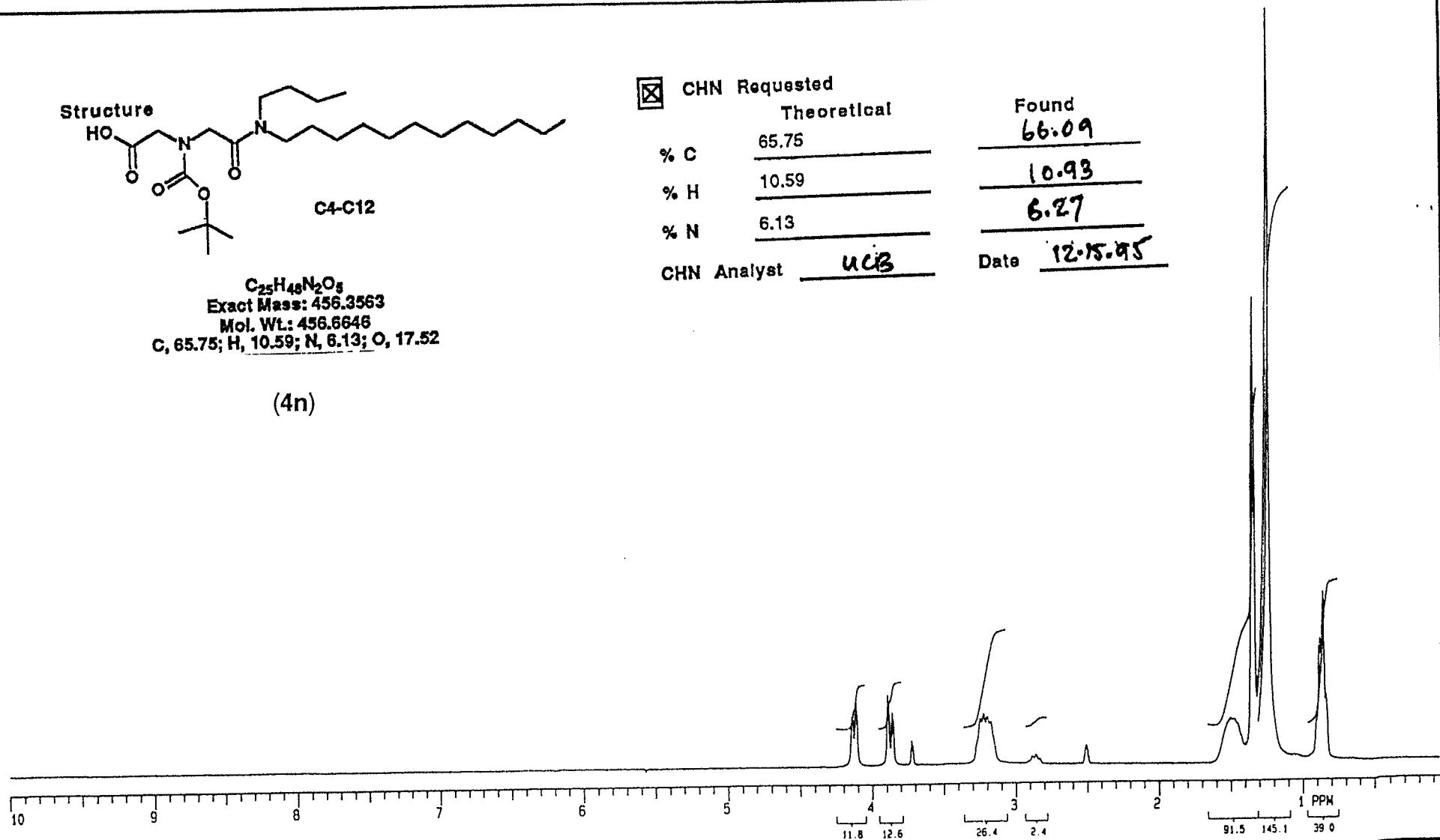
☒ CHN Requested

	Theoretical
% C	65.75
% H	10.59
% N	6.13

Found
66.09

10.93

6.27

CHN Analyst UCBDate 12-15-95

RESERVE

Nucleus 1.000 Freq 300 MHz
 Spec. Width 4500.5 Hz
 Acq. Time 1.778 sec
 Pulse Width 12.9 μ sec

Offset 0 Hz
 Delay 1.000 sec
 Transients 32

DECOUPLE

Nucleus 1.000 Offset -450.0 Hz
 Mode NNH Power 20.0 dB
 Modulation Mode C Freq 200 Hz
 Pulse Width 1.0 μ sec Power Mode 1.0

PL1/PROCESSING

FN 16 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD ---
 Width 3001.0 Hz/ppm Start 0 Hz/ppm
 Reference ---

EXPERIMENT

Pulse Sequence SQPM
 Tube O.D. --- mm
 Temp --- °C
 Solvent DMSO

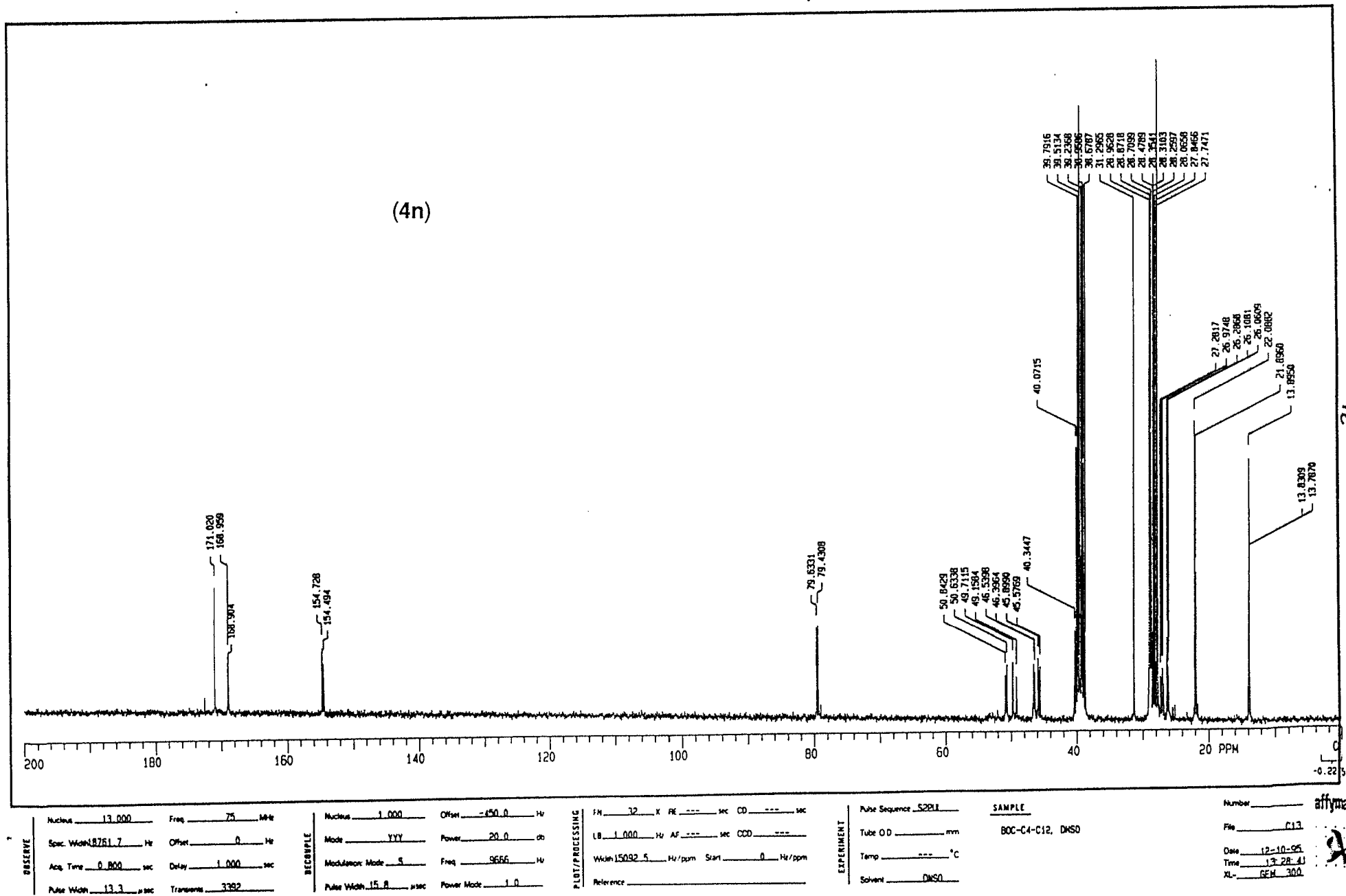
SAMPLE

BOC-C4-C12 IN DMSO

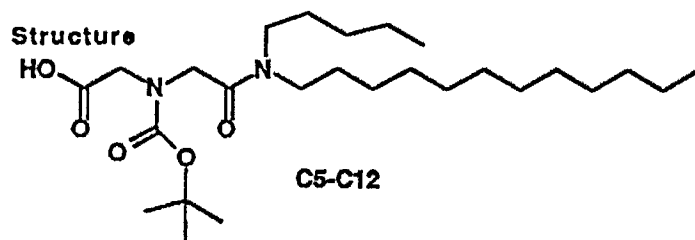
Number --- affymax
 File H
 Date 12-09-95
 Time 11:48:24
 XL GEN 300

30.

M-1608-30



M-1608-31

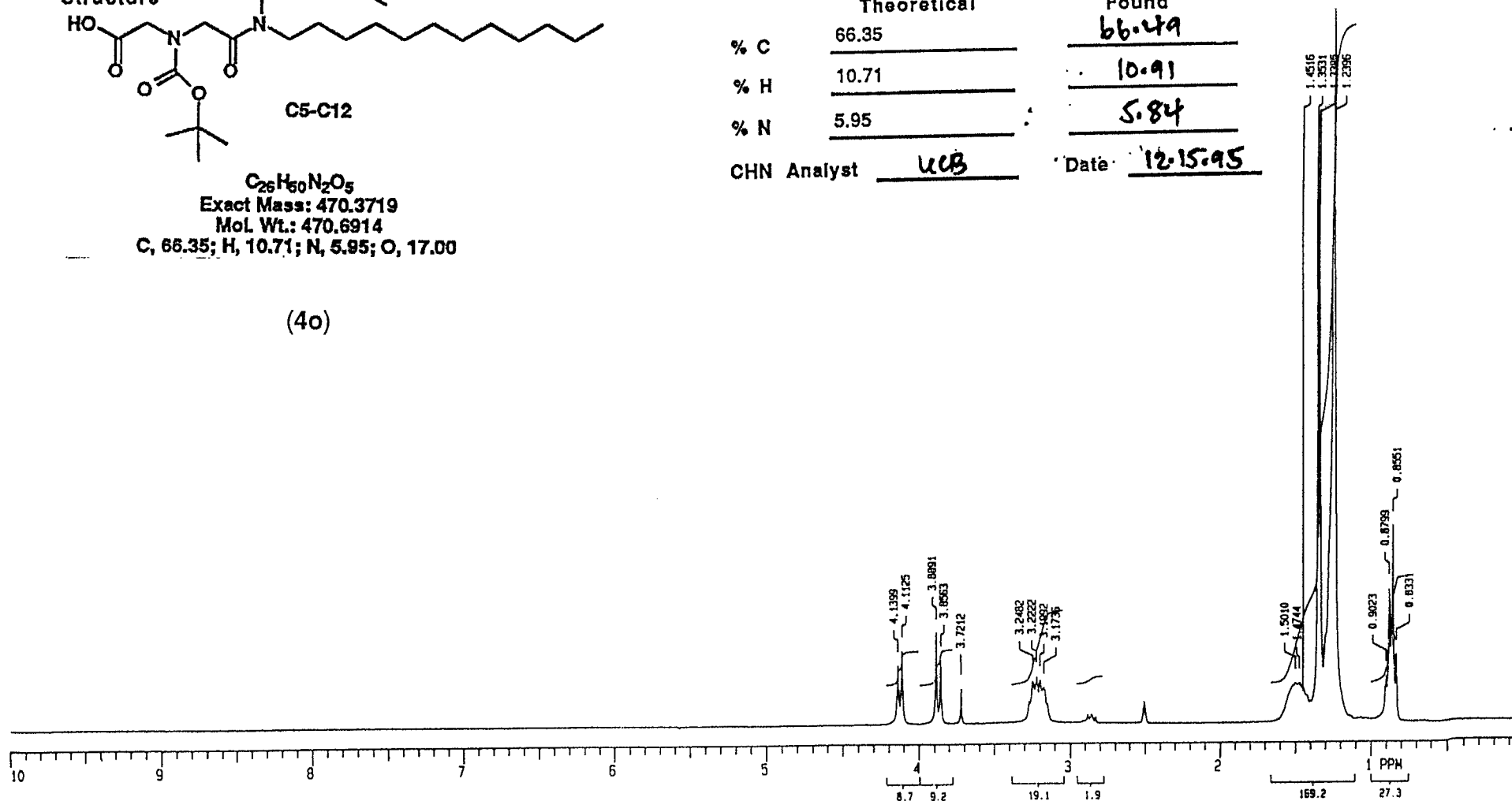


$C_{26}H_{50}N_2O_5$
 Exact Mass: 470.3719
 Mol. Wt.: 470.6914
 C, 66.35; H, 10.71; N, 5.95; O, 17.00

(4o)

☒ CHN Requested

	Theoretical	Found
% C	66.35	66.49
% H	10.71	10.91
% N	5.95	5.84
CHN Analyst	UCB	Date: 12-15-95



Observe
 Nucleus 1.000 Freq 300 MHz
 Spec. Width 4500.5 Hz Offset 0 Hz
 Acq. Time 1.778 sec Delay 1.000 sec
 Pulse Width 12.0 µsec Transmits 32

Decouple
 Nucleus 1.000 Offset -450.0 Hz
 Mode NNN Power 20.0 db
 Modulation Mode C Freq 200 Hz
 Pulse Width µsec Power Mode 1.0

Plot/Processing
 FN 16 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CCD ---
 Width 3001.0 Hz/ppm Start 0 Hz/ppm
 Reference

Experiment
 Pulse Sequence S2P11
 Tube O.D. mm
 Temp °C
 Solvent DMSO

SAMPLE
 BOC-C5-C12 IN DMSO

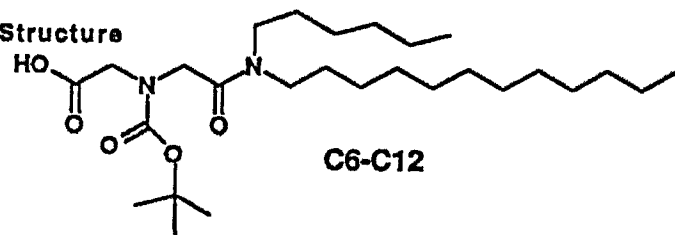
Number affymax
 File H
 Date 12-09-95
 Time 10:49:37
 XL GEN 302

32.



M-1608-33

Structure



C6-C12

$C_{27}H_{52}N_2O_5$
 Exact Mass: 484.3876
 Mol. Wt.: 484.7182
 C, 66.90; H, 10.81; N, 5.78; O, 16.50

(4p)

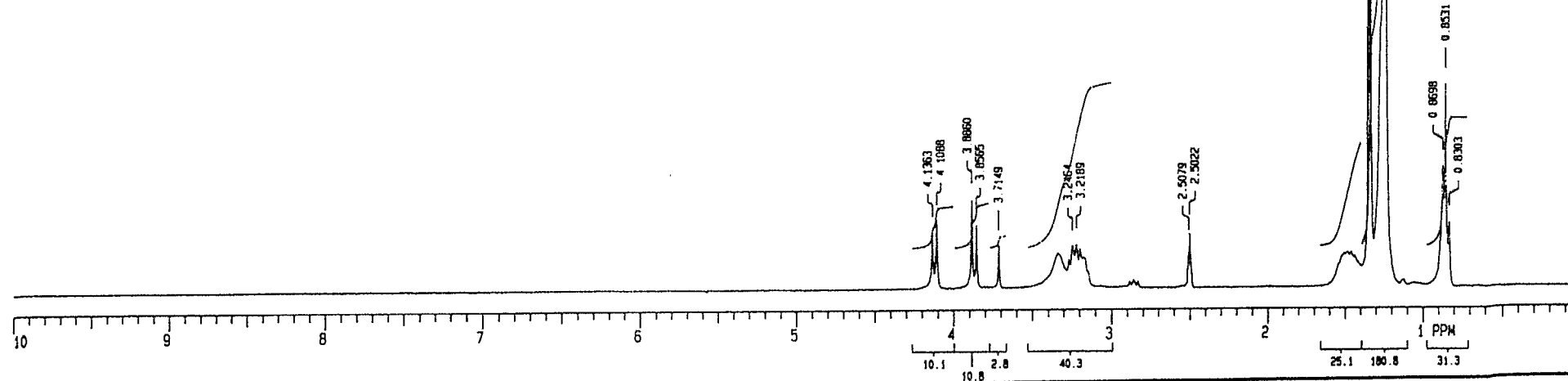
☒ CHN Requested

	Theoretical
% C	66.90
% H	10.81
% N	5.78

CHN Analyst uub

Found

67.02
10.87
5.57

Date 12-15-95

Observe
 Nucleus 1.000 Freq 300 MHz
 Spec. Width 4500.5 Hz
 Acq. Time 1.778 sec
 Pulse Width 12.0 μ sec
 Offset 0 Hz
 Delay 1.000 sec
 Transients 32

Recouple
 Nucleus 1.000 Offset -450.0 Hz
 Mode MW Power 20.0 dB
 Modulation Mode C Freq 200 Hz
 Pulse Width 1.0 μ sec
 Power Mode 1.0

Plot/Processing
 F1 16 K RE --- sec CD --- sec
 LB --- Hz AF --- sec CDD --- sec
 Width 3001.0 Hz/ppm Start 0 Hz/ppm
 Reference ---

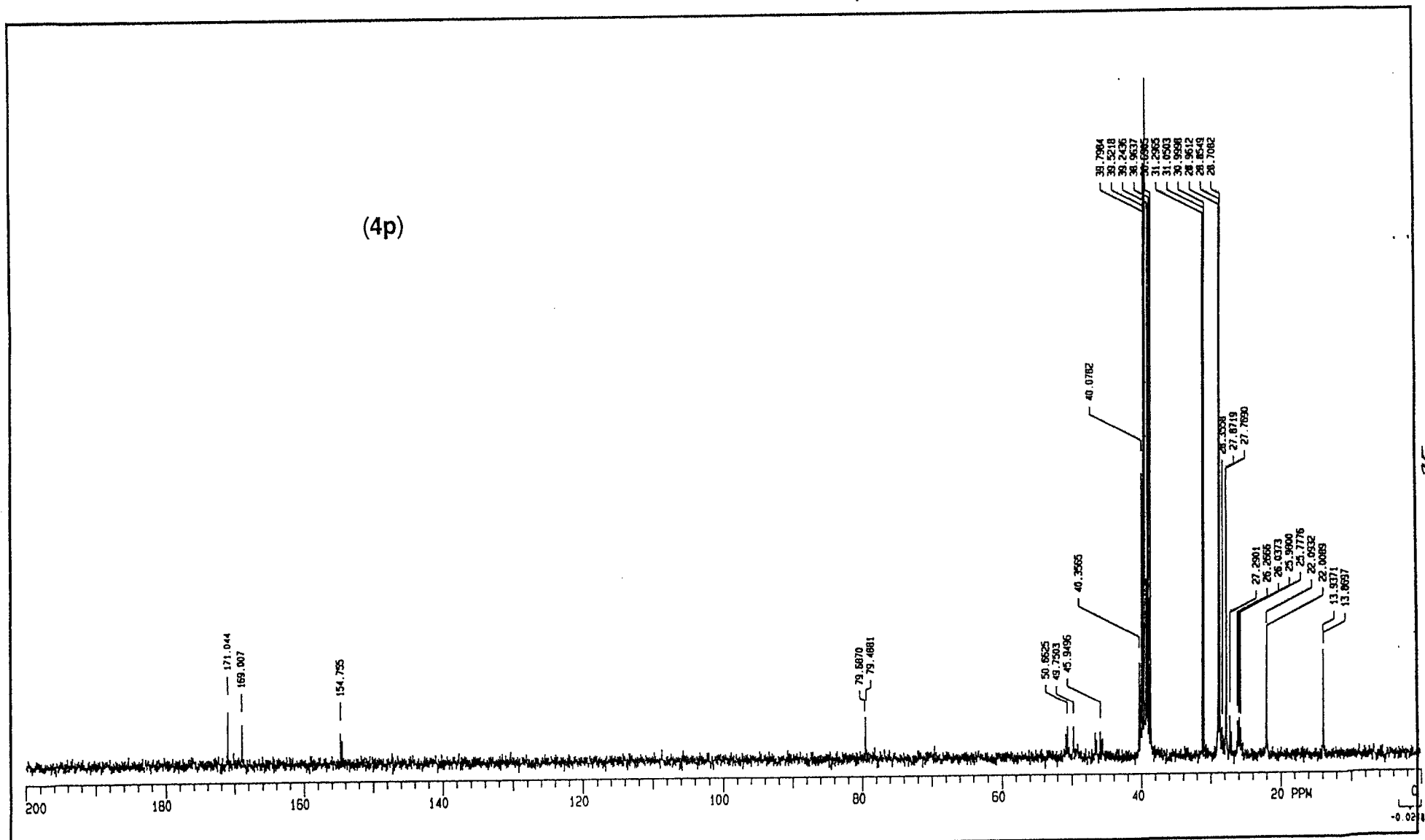
Experiment
 Pulse Sequence SZEM
 Tube O.D. --- mm
 Temp --- $^{\circ}$ C
 Solvent DMSO

Sample
 BOC-C6-C12, DMSO

Number --- affymax
 File --- H
 Date 12-27-95
 Time 08:43:15
 XL GEN 300

211

M-1608-34



OBSERVE
Nucleus 13.000 Freq 75 MHz
Spec Width 18761.7 Hz
Acq Time 0.800 sec Delay 1.000 sec
Pulse Width 13.3 µsec Transients 1280

RECEIVE
Nucleus 1.000 Offset -450.0 Hz
Mode YYY Power 20.0 db
Modulation Mode S Freq 9666 Hz
Pulse Width 15.8 µsec Power Mode 1.0

PLBT/PROCESSING
F1 32 K RE --- sec CD --- sec
LB 1.000 Hz AF --- sec CCD --- sec
Width 15092.5 Hz/ppm Start 0 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence SOPUL
Tube OD --- mm
Temp --- °C
Solvent DMSO

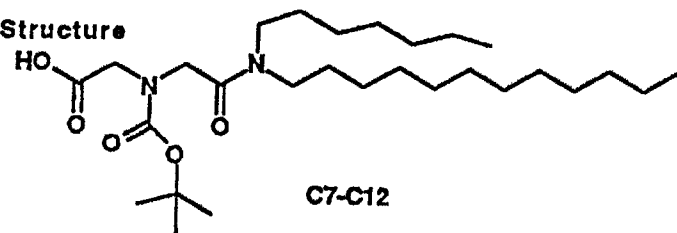
SAMPLE
BOC-C5-C12

Number
File C13
Date 12-27-95
Time 08:46:59
XL GEM 300

affymax

35.

Structure



C7-C12

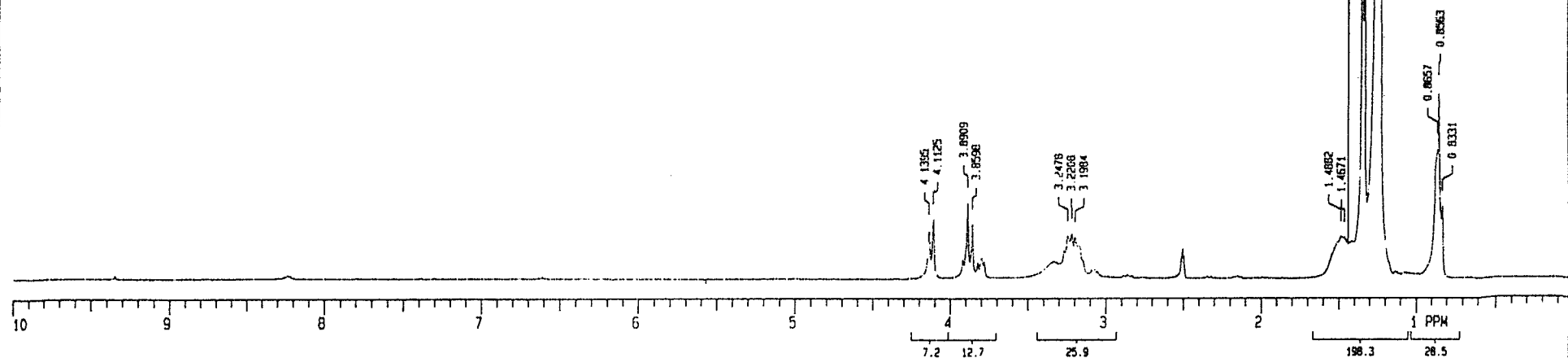
$C_{25}H_{54}N_2O_5$
Exact Mass: 498.4032
Mol. Wt.: 498.7450

C, 67.43; H, 10.91; N, 5.62; O, 16.04

(4q)

☒ CHN Requested

	Theoretical	Found
% C	67.43	66.89
% H	10.91	11.22
% N	5.62	5.54
CHN Analyst	UCB	Date 12.15.95



RESERVE
Nucleus 1.000 Freq 300 MHz
Spec. Width 4500.5 Hz Offset 0 Hz
Acq. Time 1.778 sec Delay 1.000 sec
Pulse Width 12.0 µsec Transients 32

DECOUPLE
Nucleus 1.000 Offset -450.0 Hz
Mode MNH Power 20.0 dB
Modulation Mode C Freq 200 Hz
Pulse Width µsec Power Mode 1.0

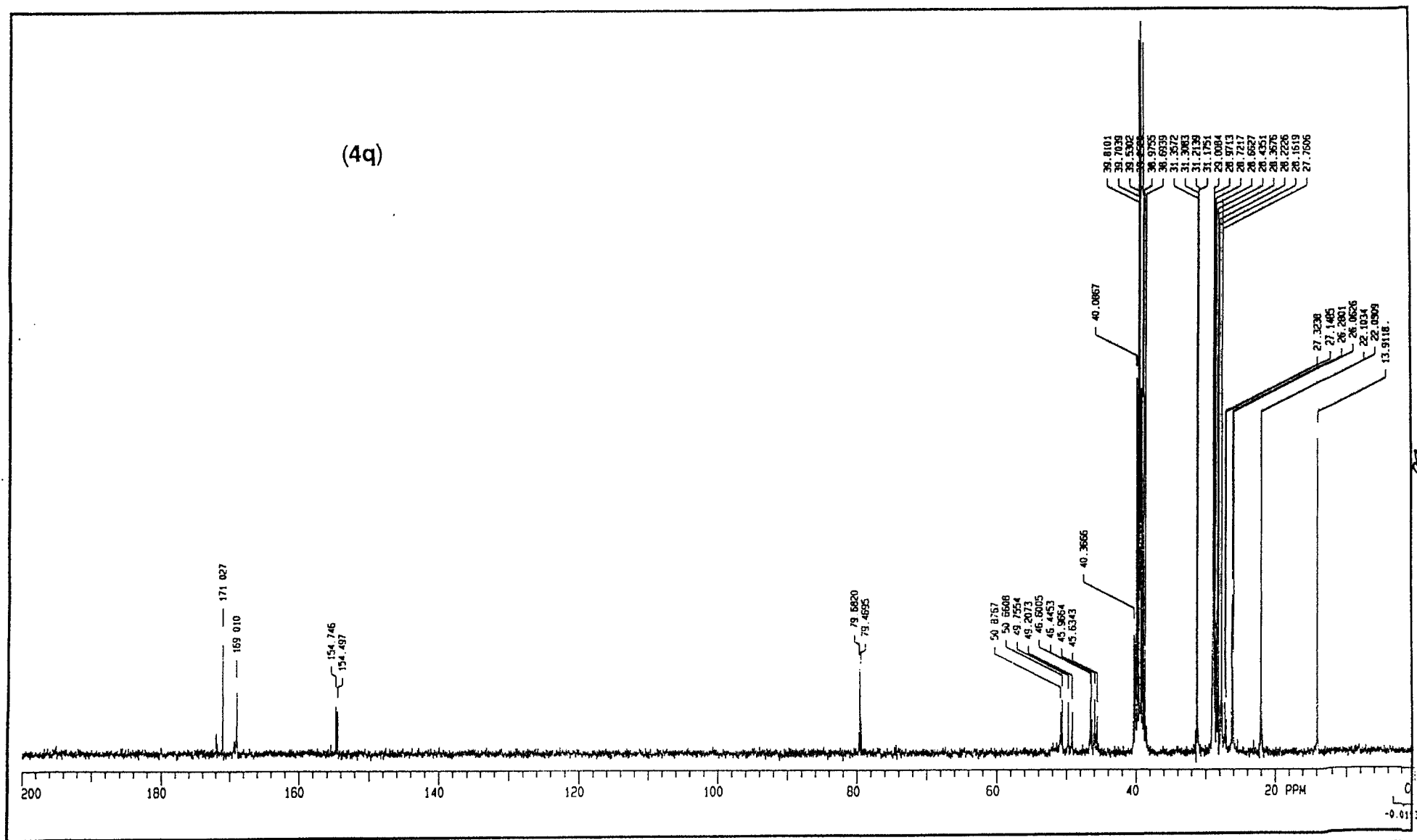
PLOT/PROCESSING
F1 16 K RE --- sec CD --- sec
LB --- Hz AF --- sec CCD ---
Width 3001.0 Hz/ppm Start 0 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence S2PUL
Tube OD mm
Temp --- °C
Solvent DMSO

SAMPLE
BOC-C7-C12 IN DMSO

affymax
Number ---
File H
Date 12-20-95
Time 17:08:28
XL GEN 300

M-1608-36



OBSERVE
Nucleus 13.000 Freq 75 MHz
Spec. Width 18761.7 Hz Offset 0 Hz
Acq. Time 0.800 sec Delay 1.000 sec
Pulse Width 13.3 µsec Transients 4160

RECUPLE
Nucleus 1.000 Offset -450.0 Hz
Mode YYY Power 20.0 dB
Modulation Mode S Freq 9566 Hz
Pulse Width 15.8 µsec Power Mode 1.0

PLBT/PROCESSING
FN 32 K RE --- sec CD --- sec
LB 1.000 Hz AF --- sec CCD ---
Width 15092.5 Hz/ppm Start 0 Hz/ppm
Reference

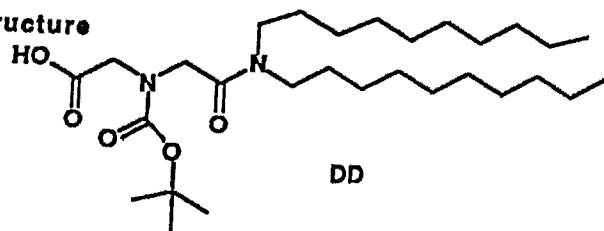
EXPERIMENT
Pulse Sequence S2PUL
Tube OD mm
Temp --- °C
Solvent DMSO

SAMPLE
BOC-C7-C12, DMSO

Number affyma
File C13
Date 12-20-95
Time 17:15:20
XL GEN 300

M-1608-37

Structure



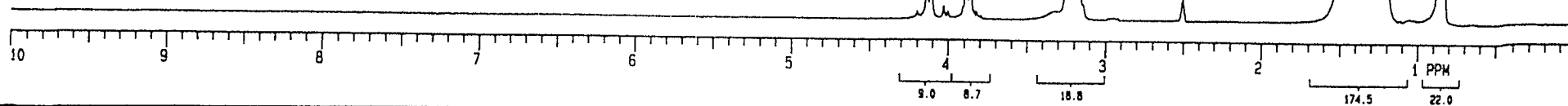
$C_{23}H_{56}N_2O_5$
Exact Mass: 512.4189
Mol. Wt.: 512.7718
C, 67.93; H, 11.01; N, 5.46; O, 15.60

(4r)



CHN Requested

	Theoretical	Found
% C	67.93	68.12
% H	11.01	11.35
% N	5.46	5.28
CHN Analyst	UCB	Date 12/15/95



Observe
Nucleus 1.000 Freq 300 MHz
Spec. Width 500.5 Hz Offset 0 Hz
Acq. Time 1.778 sec Delay 1.000 sec
Pulse Width 12.0 µsec Transmits 32

Decouple
Nucleus 1.000 Offset -450.0 Hz
Mode NMR Power 20.0 db
Modulation Mode C Freq 200 Hz
Pulse Width µsec Power Mode 1.0

PLOT/PROCESSING

FW 16 K RE --- sec CD --- sec
LB --- Hz AF --- sec CCD ---
Width 3001.0 Hz/ppm Start 0 Hz/ppm
Reference

EXPERIMENT

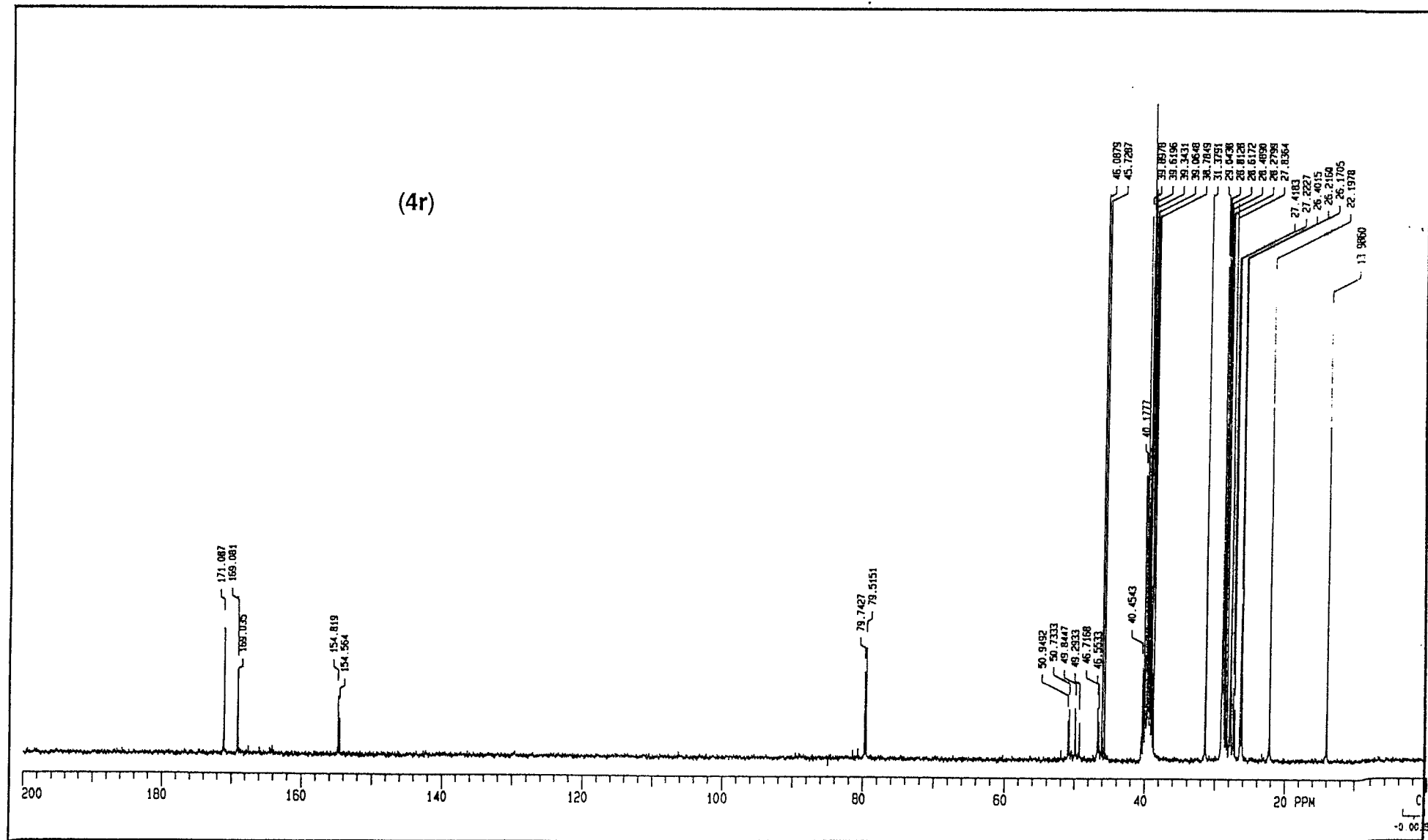
Pulse Sequence S2B1X
Tube 00 mm
Temp --- °C
Solvent DMSO

SAMPLE

800-DD, DMSO

Number affymax
File H
Date 12-08-95
Time 17:30:22
XL GEN 300

38.



OBSEV
Nucleus 13.000 Freq 75 MHz
Spec. Width 18761.7 Hz
Acq. Time 0.800 sec Delay 1.000 sec
Pulse Width 13.3 µsec Transmits 4864

RECSMPLE
Nucleus 1.000 Offset -450.0 Hz
Mode YYY Power 20.0 db
Modulation Mode S Freq 9656 Hz
Pulse Width 15.8 µsec Power Mode 1.0

PLBT/PROCESSING
F1 32 K RE --- sec CD --- sec
LB 1.000 Hz AF --- sec CCD ---
Width 15092.5 Hz/ppm Start 0 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence S2911
Tube OD mm
Temp --- °C
Solvent DMSO

SAMPLE
BOC-DO, DMSO

Number affymax
File C13
Date 12-08-95
Time 17:36:05
XL GEM 300