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Supporting Information: Experimental procedures and characterization of **16c** and **17a-c**, and ^1H NMR data for all compounds (40 pages).

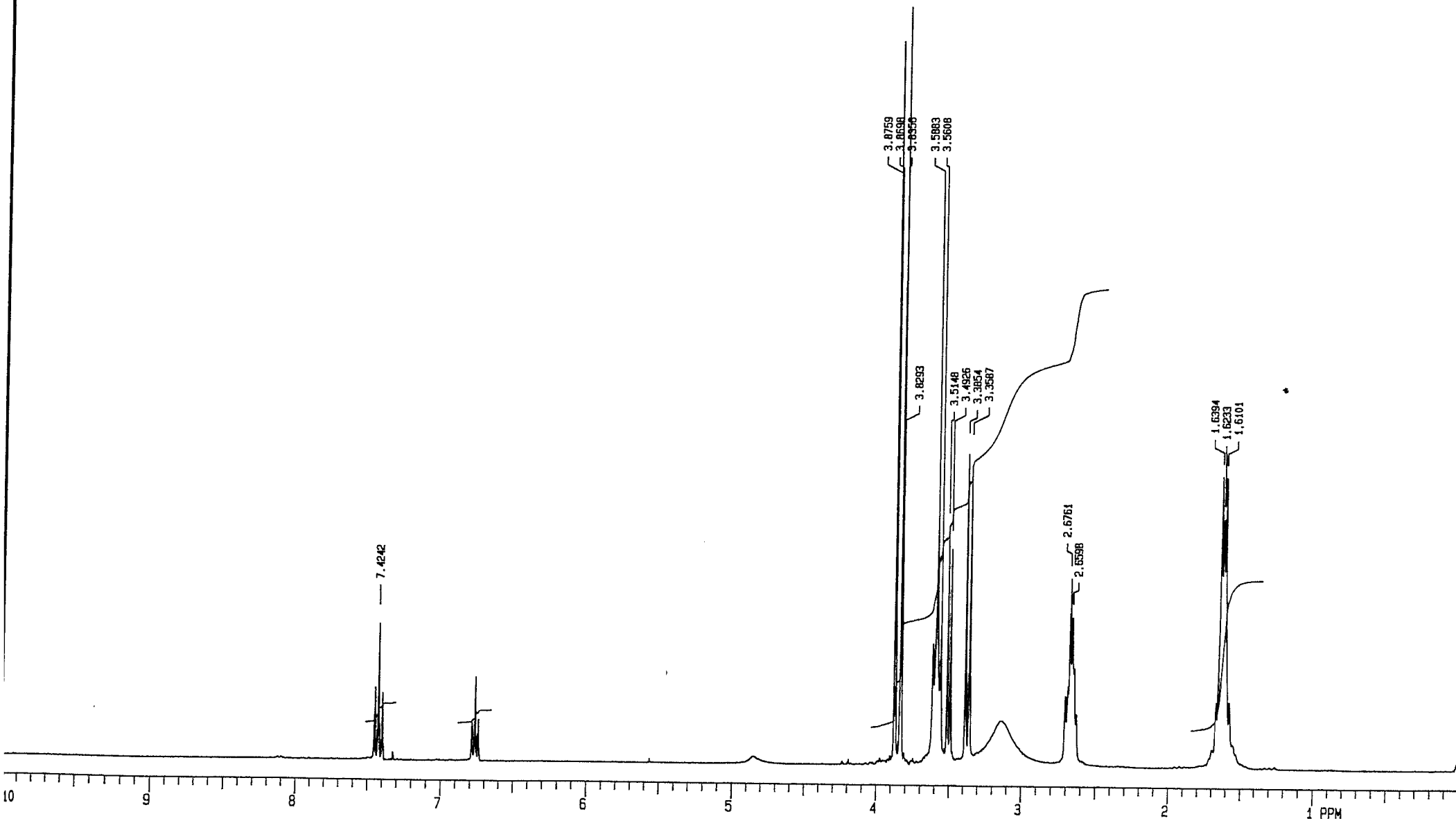
1,8-Dimethoxy-3-methyl-9,10-anthracenedione (16c). To a solution of chrysophanic acid **15c** (300 mg, 1.2 mmol) in acetone (236 mL) were added K_2CO_3 (7.1 g, 51.4 mmol) and Me_2SO_4 (0.59 mL, 6.2 mmol) under a nitrogen atmosphere at rt. The reaction mixture was heated at reflux for 5h. After the solvent was evaporated at reduced pressure, the residue was diluted with water and CH_2Cl_2 . The layers were separated and the aqueous phase was extracted with CH_2Cl_2 . The combined organic phase was dried over MgSO_4 and concentrated at reduced pressure. Purification of the residue by flash column chromatography (AcOEt/hexane 1:1) afforded **16c** (333 mg, quant.) as yellow crystals. mp 202-203 °C (AcOEt). IR (CHCl_3) 1668 (ArCOAr) cm^{-1} . ^1H NMR (CDCl_3) δ 7.83 (1H, d, $J=8$ Hz), 7.65 (1H, quint, $J=1$ Hz), 7.62 (1H, t, $J=8$ Hz), 7.29 (1H, d, $J=8$ Hz), 7.10 (1H, br s), 4.00 (3H, s), 3.99 (3H, s), 2.47 (3H, d, $J=1$ Hz). ^{13}C NMR (CDCl_3) δ 184.4, 182.7, 159.7, 159.5, 145.1, 134.9, 134.6, 133.7, 124.1, 121.7, 119.6, 118.9, 118.7, 118.1, 56.5, 56.5, 22.2. HRMS: Calcd for $\text{C}_{17}\text{H}_{14}\text{O}_4$ (M^+) : 282.0891, Found : 282.0885. Anal. Calcd for $\text{C}_{17}\text{H}_{14}\text{O}_4$: C, 72.33; H, 5.00, Found : C, 72.14; H, 5.05.

1,8,9,10-Tetramethoxy-3-methylanthracene (17c). To a solution of **16c** (765 mg, 2.7 mmol) and tetrabutylammonium bromide (174 mg, 0.54 mmol) in THF (236 mL) was added a solution of $\text{Na}_2\text{S}_2\text{O}_4$ (2.83 g, 16.3 mmol) in H_2O (24.7 mL) under a nitrogen atmosphere at rt. After stirring at the same temperature for 15 min, 6N KOH (10.3 mL) was added to the reaction mixture. After stirring at the same temperature 5 min, Me_2SO_4 (1.4 mL, 14.8 mmol) was added to the reaction mixture. The mixture was stirred at the same temperature for 12 h and concentrated at reduced pressure. The aqueous residue was extracted with CH_2Cl_2 and the organic phase was

dried over MgSO_4 and concentrated at reduced pressure. Purification of the residue by medium-pressure column chromatography (AcOEt/hexane 1:3) afforded **17c** (718 mg, 85%) as yellow crystals. mp 83-84 °C (AcOEt/hexane). IR (CHCl_3) 3014, 2931, 2840 (Me) cm^{-1} . ^1H NMR (CDCl_3) δ 7.82 (1H, dd, $J=8.5$, 1 Hz), 7.61 (1H, quint, $J=1.5$ Hz), 7.36 (1H, dd, $J=8.5$, 7.5 Hz), 6.74 (1H, dd, $J=7.5$, 1 Hz), 6.61 (1H, d, $J=1.5$ Hz), 4.03 (9H, s), 3.89 (3H, s), 2.52 (3H, d, $J=1.5$ Hz). ^{13}C NMR (CDCl_3) δ 157.6, 157.3, 151.0, 146.7, 135.7, 127.68, 127.65, 125.7, 118.5, 117.7, 114.5, 113.0, 106.9, 103.8, 63.6, 62.3, 56.30, 56.29, 22.5. HRMS: Calcd for $\text{C}_{19}\text{H}_{20}\text{O}_4$ (M^+) : 312.1360, Found : 312.1370. Anal. Calcd for $\text{C}_{19}\text{H}_{20}\text{O}_4$: C, 73.06; H, 6.45, Found : C, 72.77; H, 6.48.

1,8,9,10-Tetramethoxyanthracene (17a). According to the literature,²⁸ compound **17a** was obtained from **15a** in 70% yield as a yellow solid. IR (CHCl_3) 3016, 2957, 2936, 2837 (Me) cm^{-1} . ^1H NMR (CDCl_3) δ 7.85 (2H, dd, $J=8.7$, 1 Hz), 7.38 (2H, t, $J=7.6$ Hz), 6.78 (2H, d, $J=7.3$ Hz), 4.04 (9H, s), 3.90 (3H, s). HRMS: Calcd for $\text{C}_{18}\text{H}_{18}\text{O}_4$ (M^+) : 298.1204, Found : 298.1220.

1,4,9,10-Tetramethoxyanthracene (17b). According to the literature,²⁸ compound **17b** was obtained from **15b** in 60% yield as yellow crystals. mp 154-157 °C (AcOEt/hexane). IR (CHCl_3) 2936, 2837 (Me) cm^{-1} . ^1H NMR (CDCl_3) δ 8.35 (2H, AA'BB', $J_{\text{AB}}=10$ Hz, $J_{\text{AB}'}=3.2$ Hz), 7.51 (2H, AA'BB', $J_{\text{BA}}=10$ Hz, $J_{\text{BA}'}=3.2$ Hz), 6.68 (2H, s), 4.01 (6H, s), 3.99 (6H, s). HRMS: Calcd for $\text{C}_{18}\text{H}_{18}\text{O}_4$ (M^+) : 298.1204, Found : 298.1205. Anal. Calcd for $\text{C}_{18}\text{H}_{18}\text{O}_4$: C, 72.47; H, 6.08, Found : C, 72.43; H, 6.12.



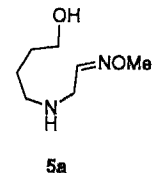
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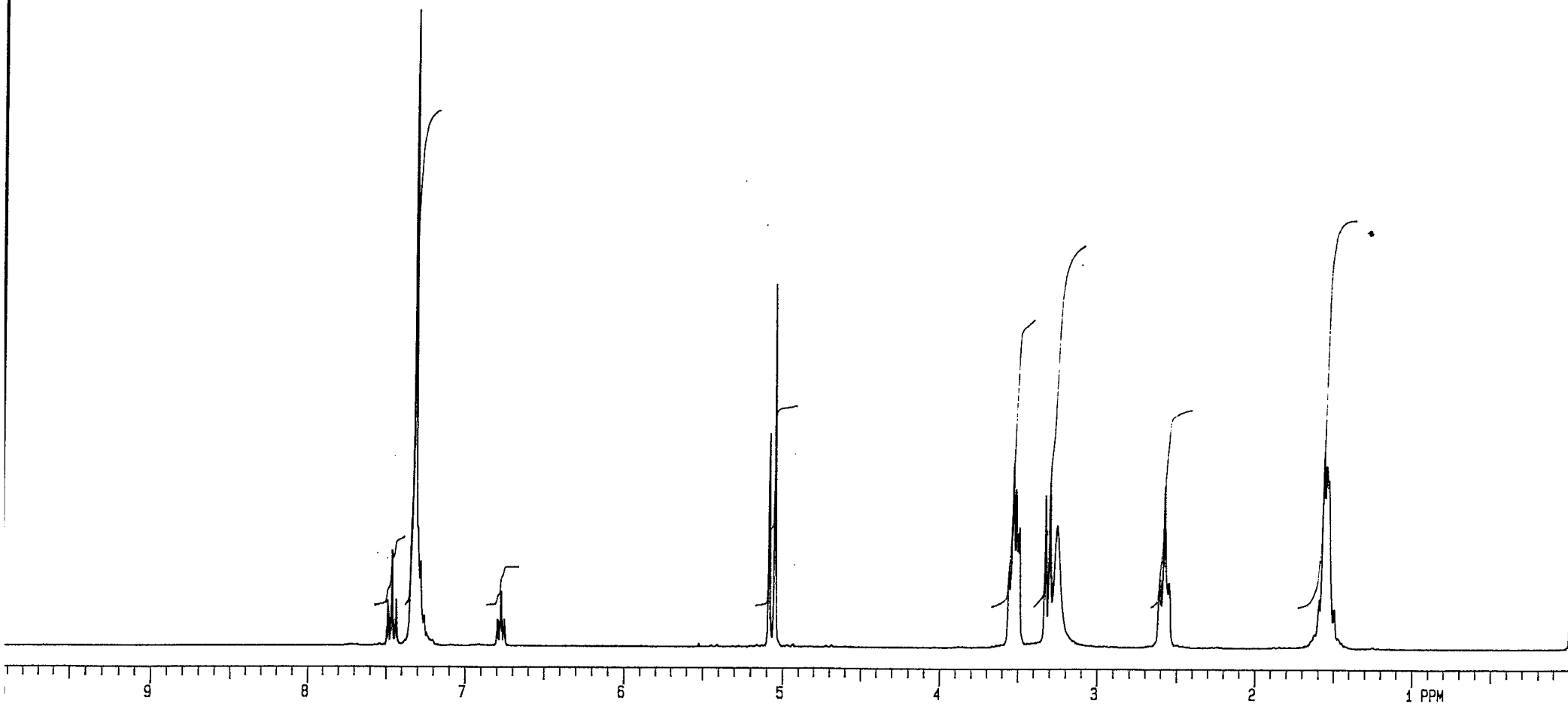
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 Temp °C
 Solvent CDCl3

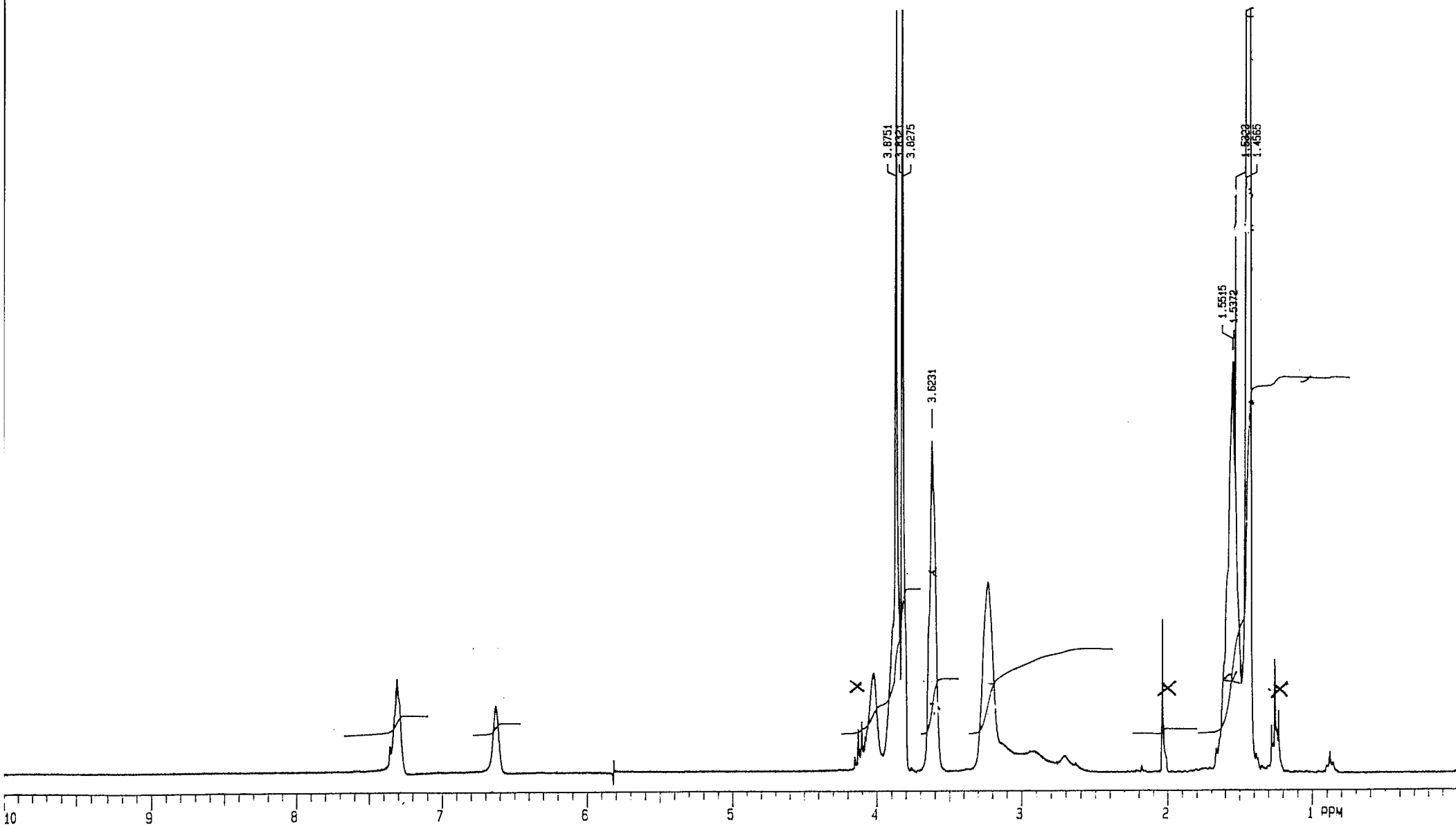
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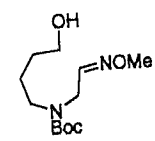
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PLOT/PROCESSING
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Temp. °C
Solvent CDCl3

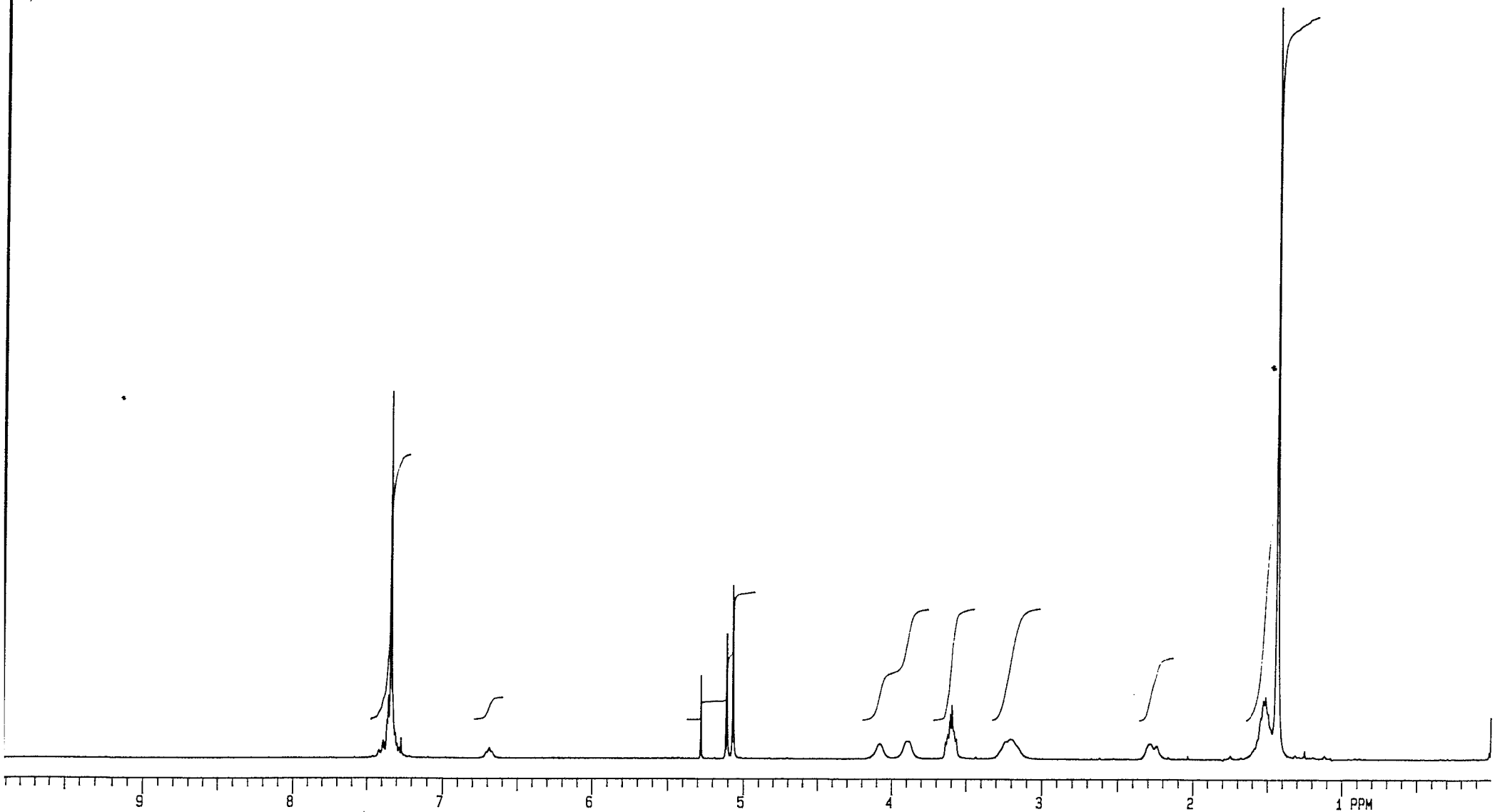
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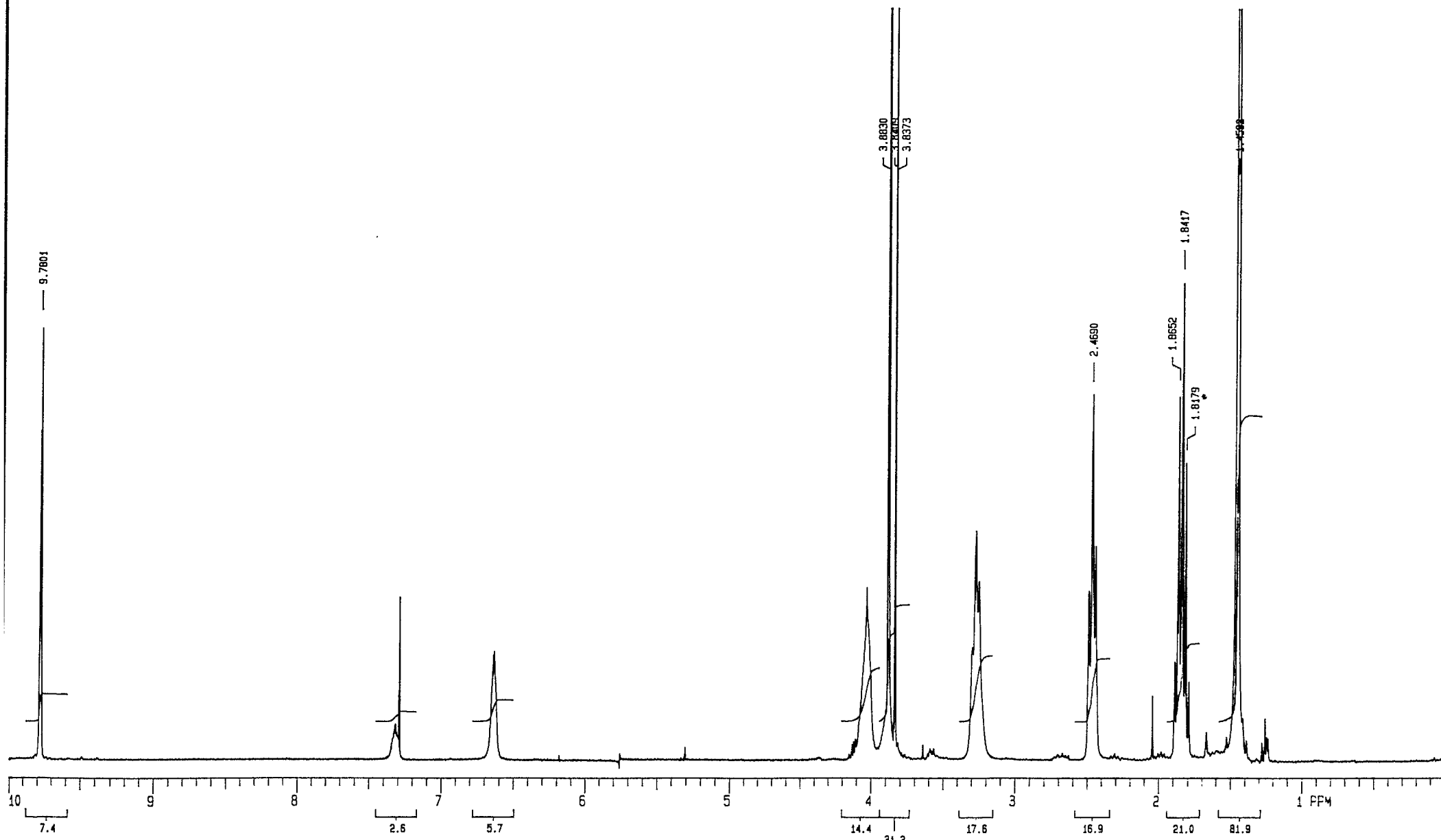
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Varian Japan Ltd. (W)



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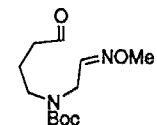
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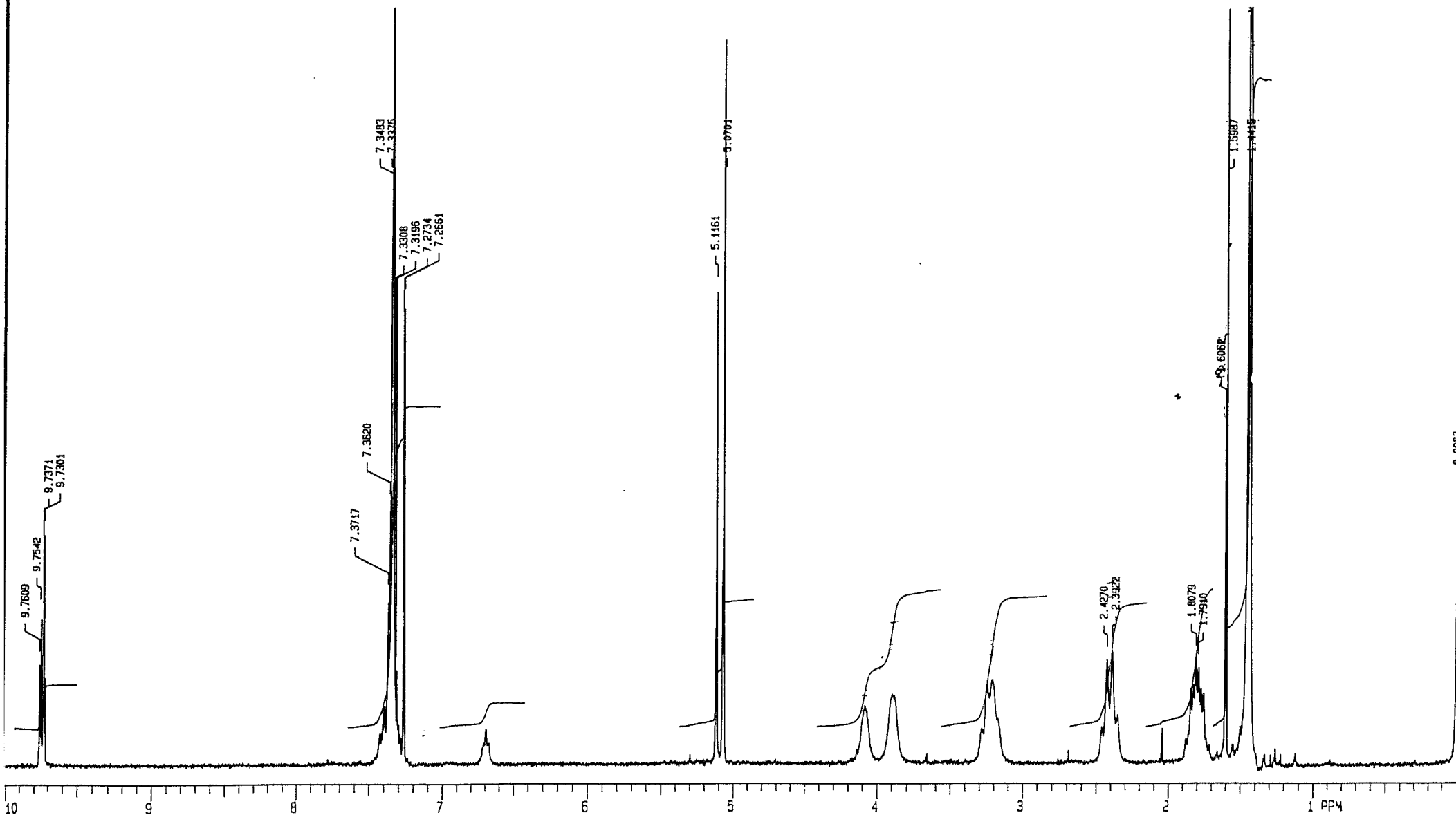
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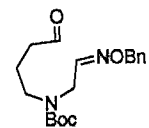
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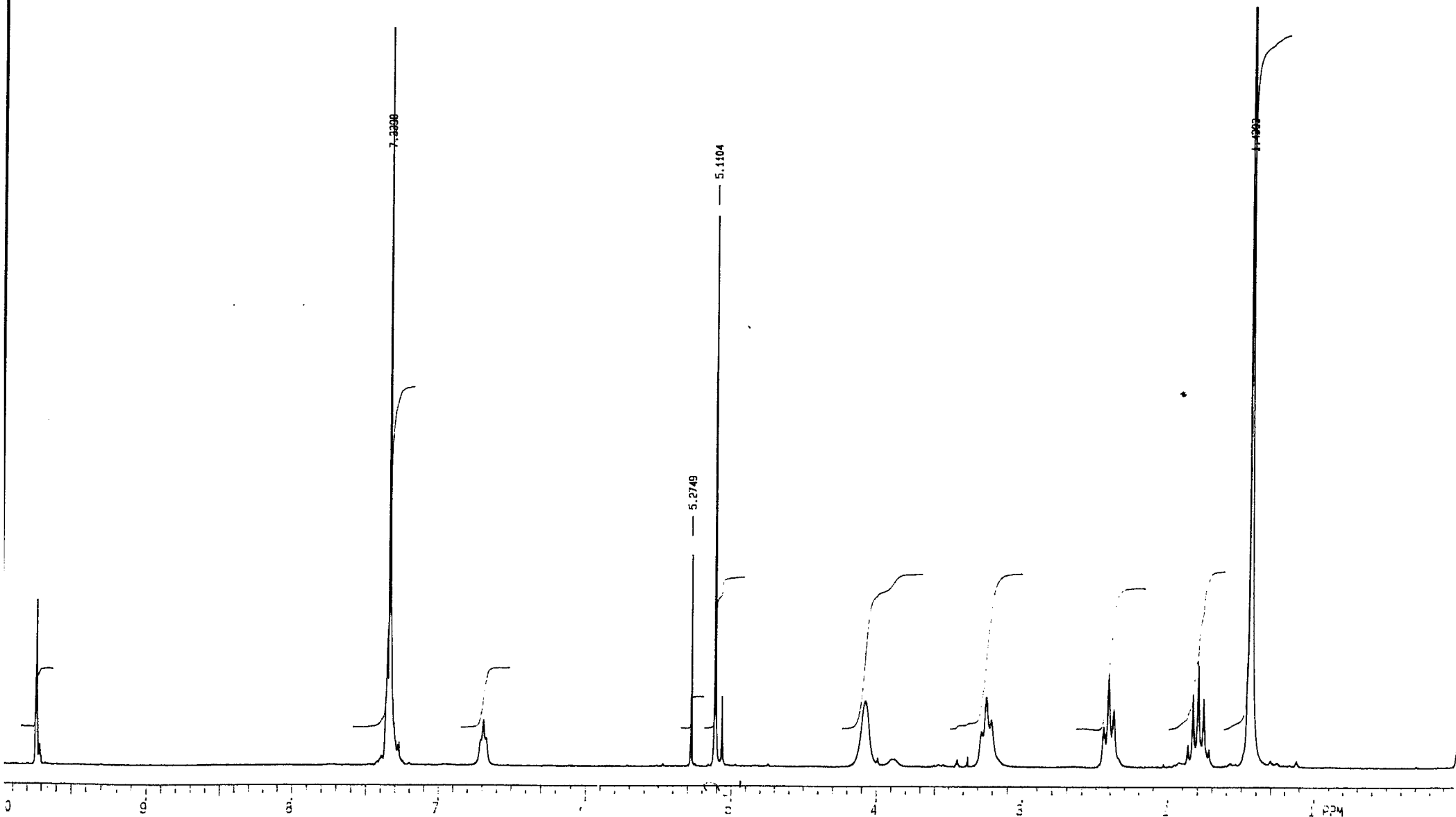
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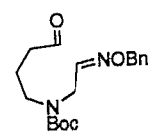
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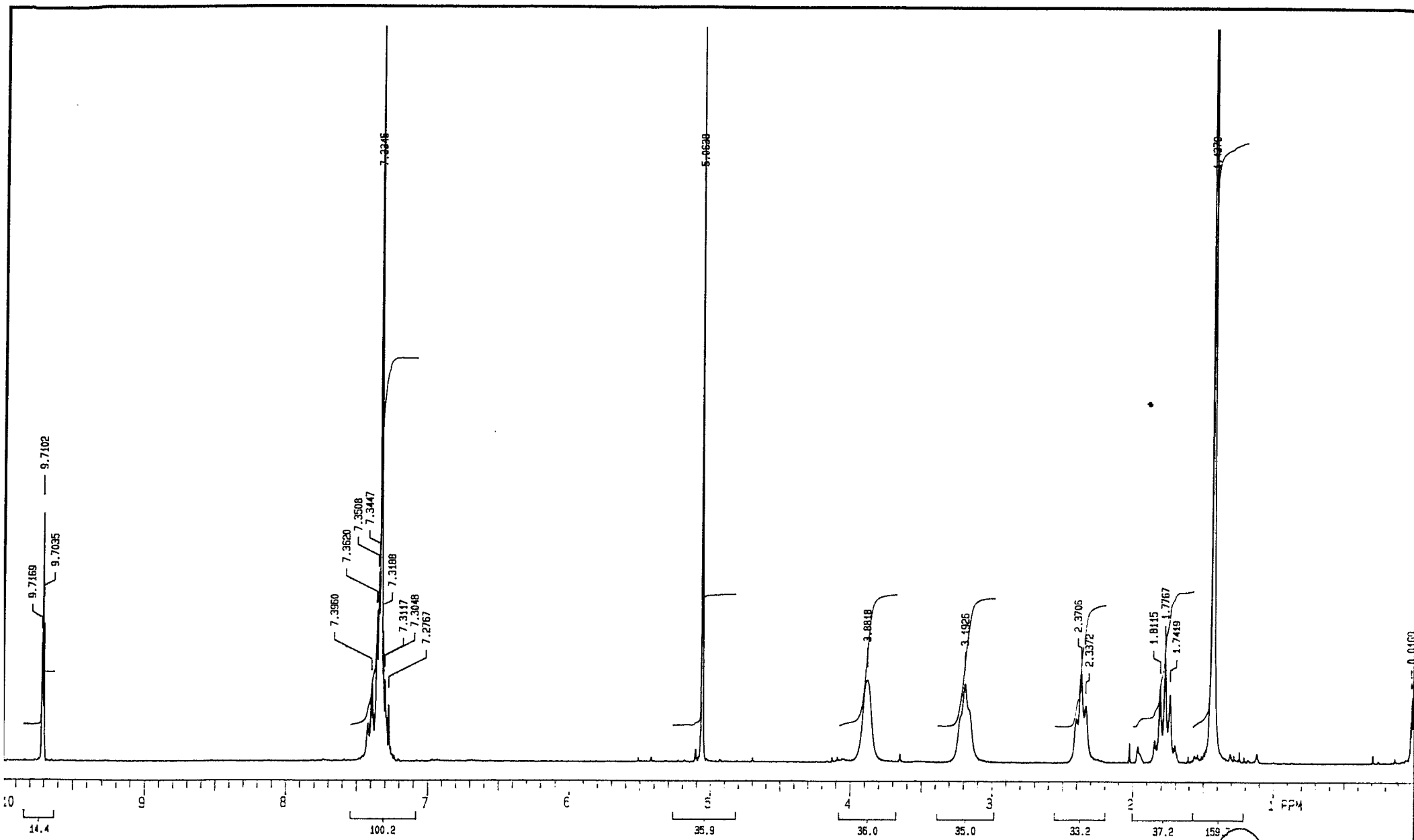
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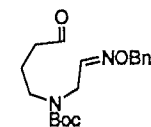
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PLT/PROCESSING
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Temp. °C
Solvent CDCl₃

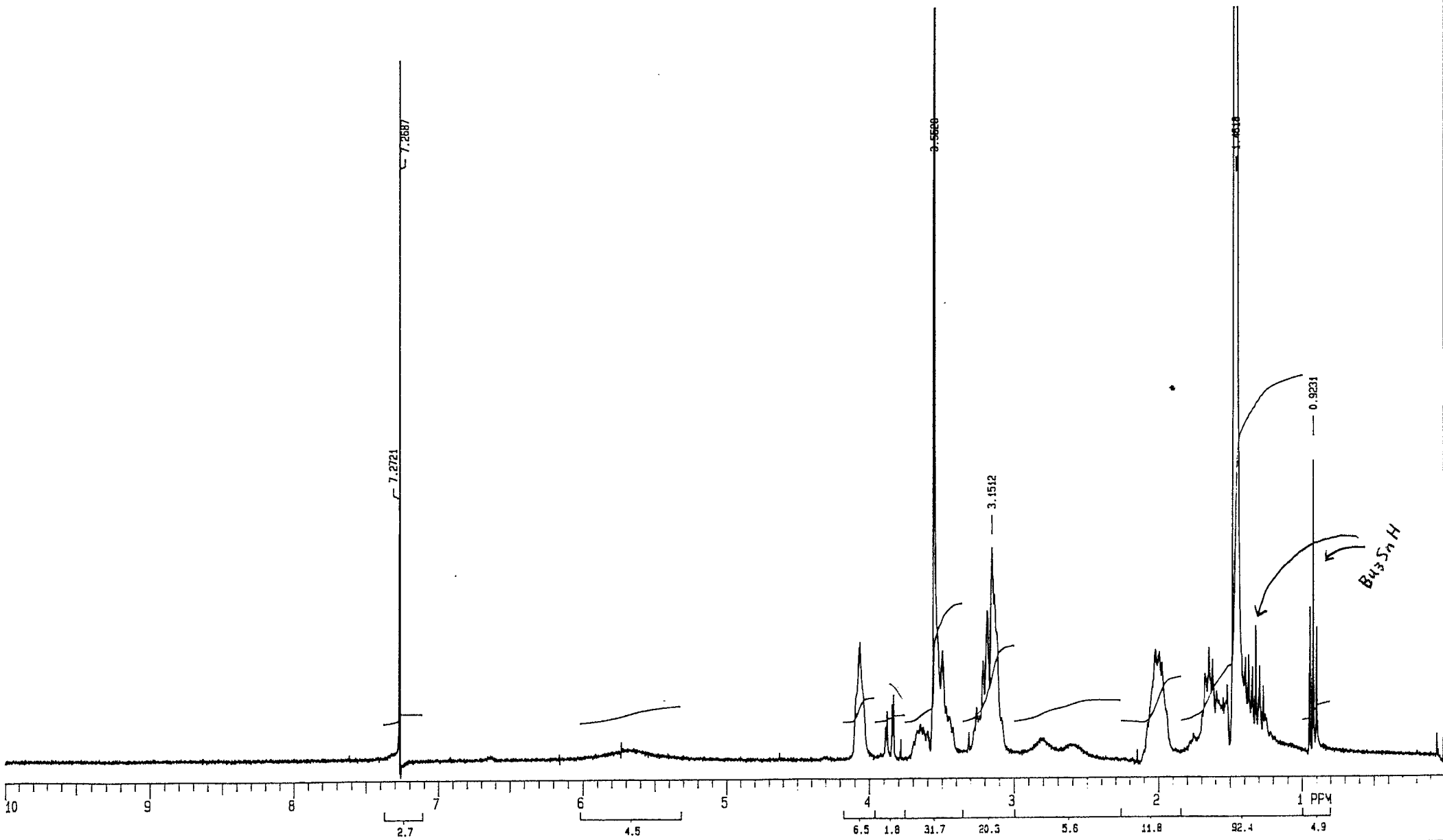
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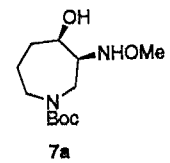
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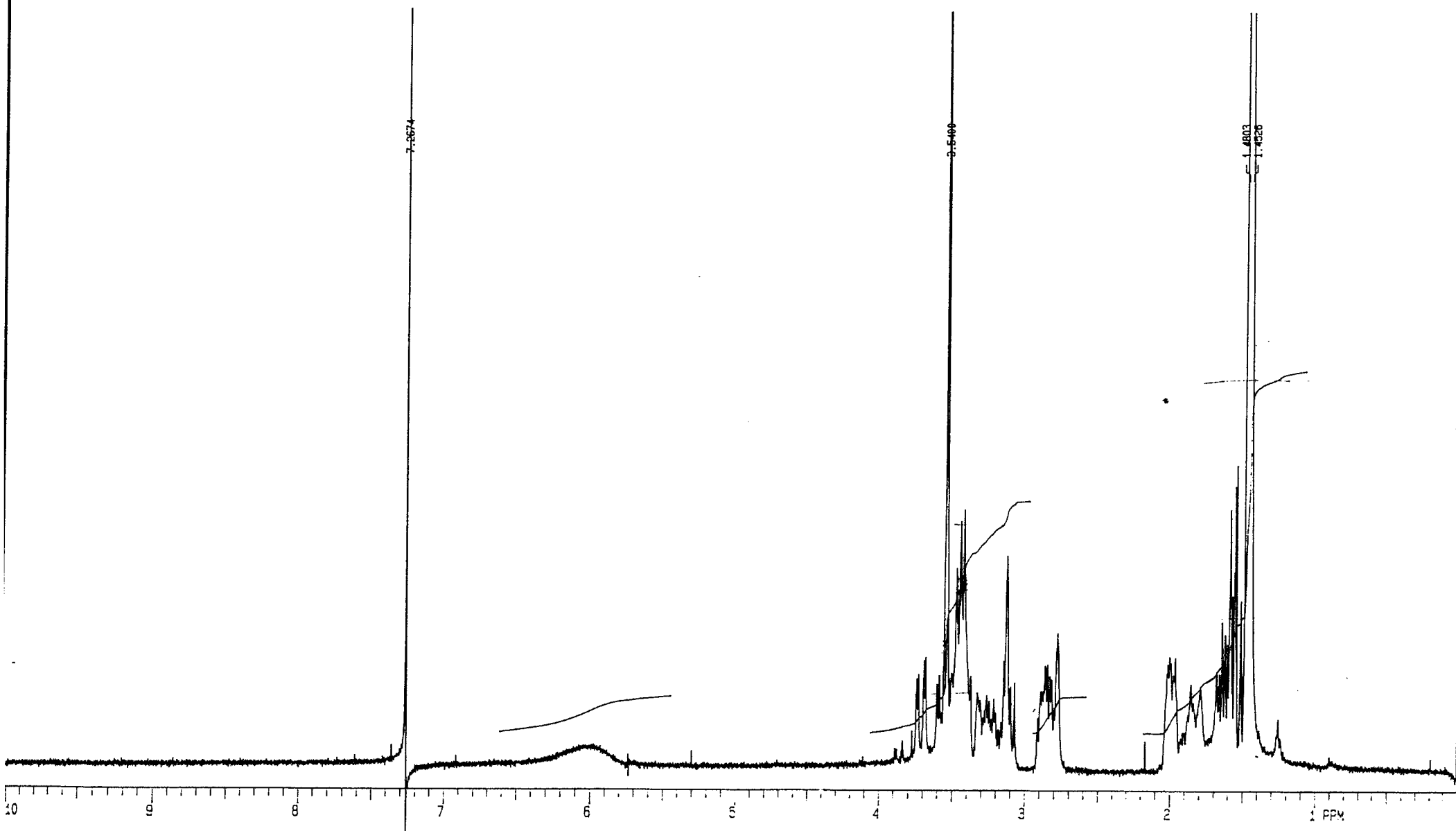
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Varian Japan Ltd. (V)



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OBSERVE

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Pulse Width 9.4 μ sec Transients 16

DECOUPLE

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Pulse Width μ sec Power Mode 1.0

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Reference

EXPERIMENT

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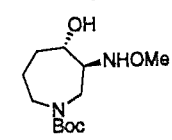
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Temp. °C

Solvent CDCl3

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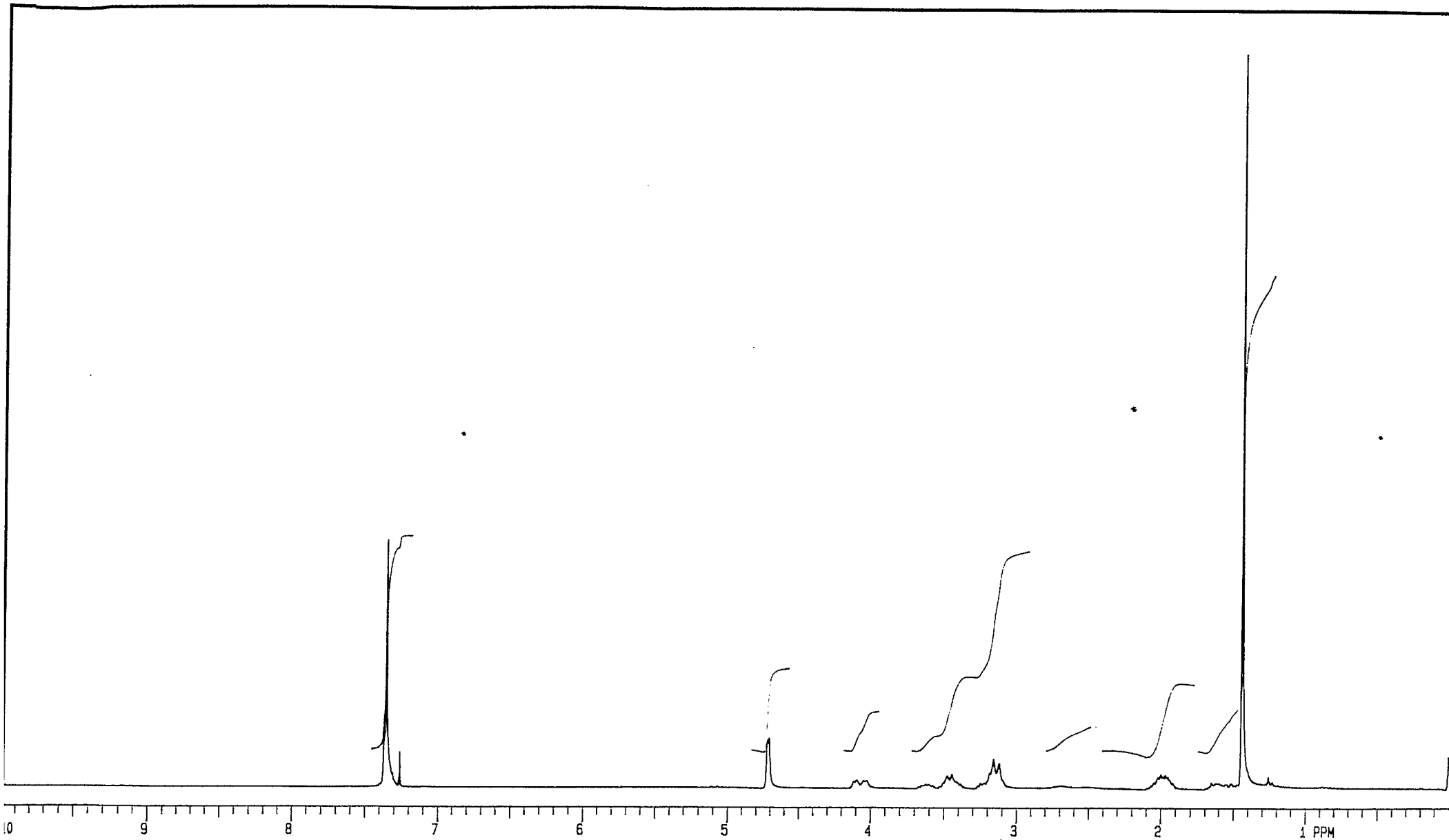
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XL 6CH 300

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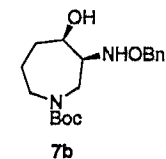
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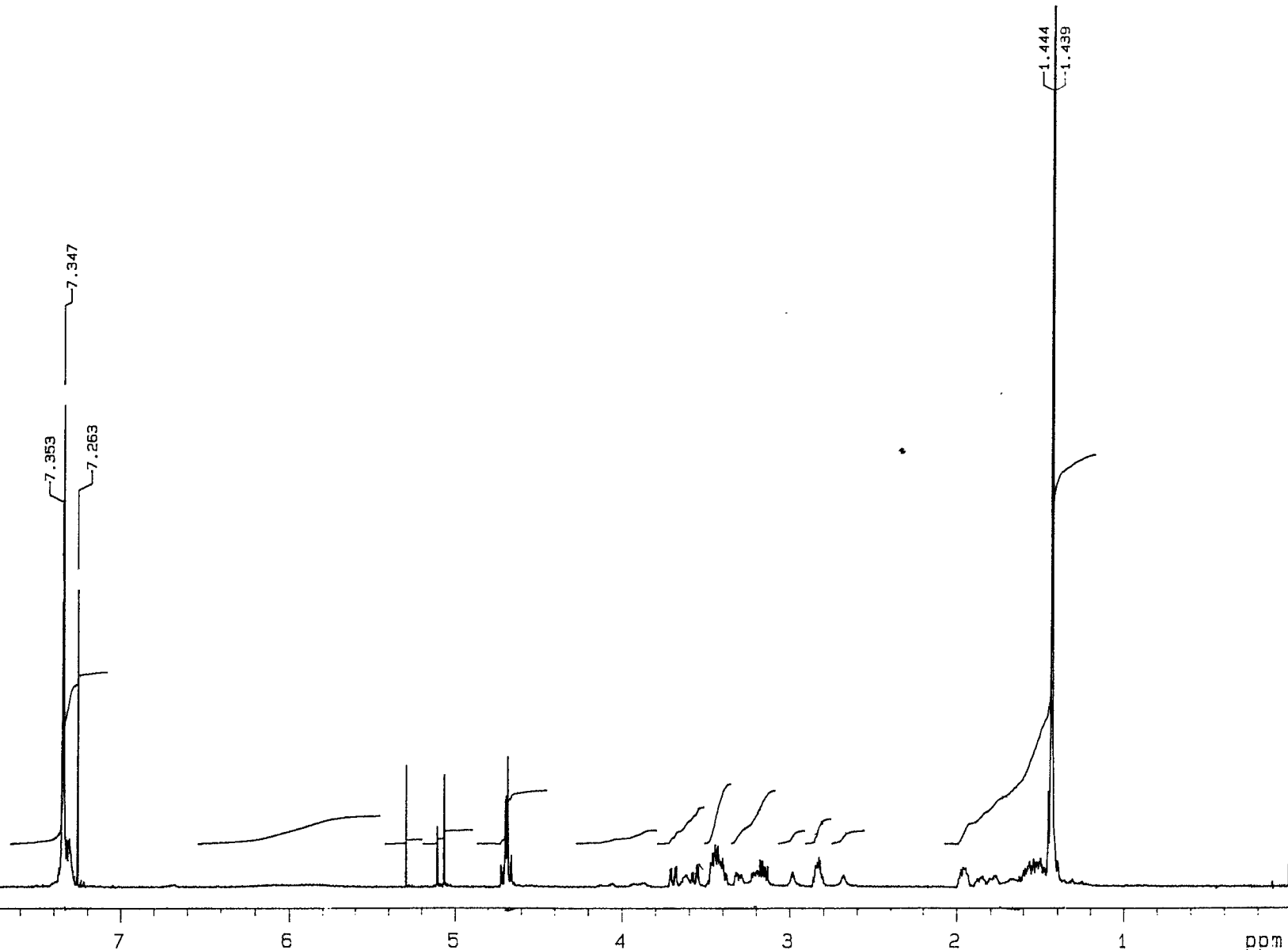
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H-1

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 fb 4400 fn not used
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 tpwr 61
 pw 7.0 werr
 d1 0 wexp wft
 tof 1000.0 wbs wft
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 gain 9 wp 4999.2
 FLAGS vs 326
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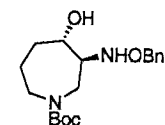
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 KPN-3689
 H-1



8b

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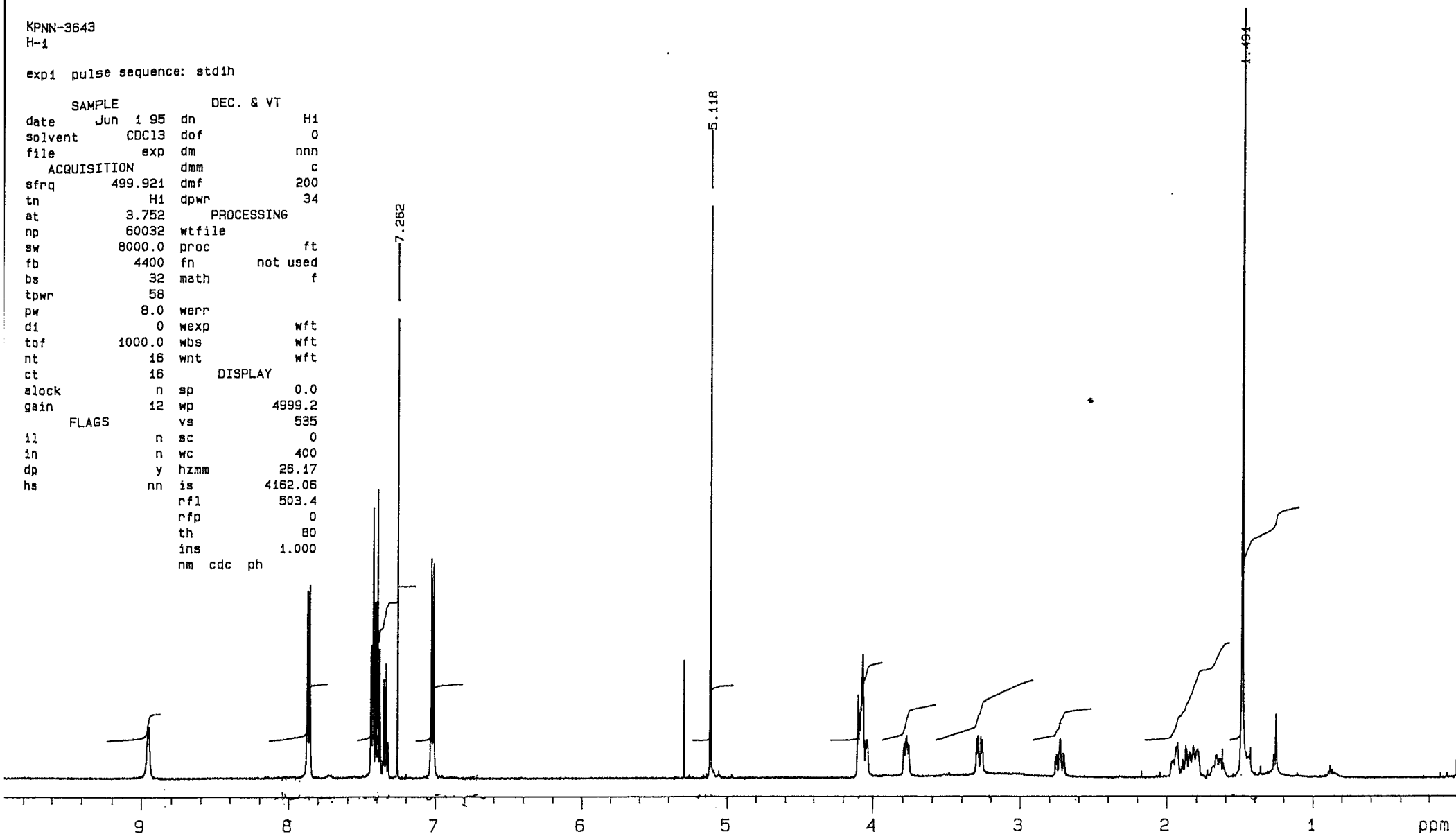
Varian Japan Ltd. (VA)

KPNN-3643

H-1

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tpwr	58		
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di	0	wexp	wft
tof	1000.0	wbs	wft
nt	16	wnt	wft
ct	16	DISPLAY	
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il	n	sc	0
in	n	wc	400
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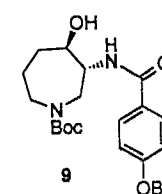
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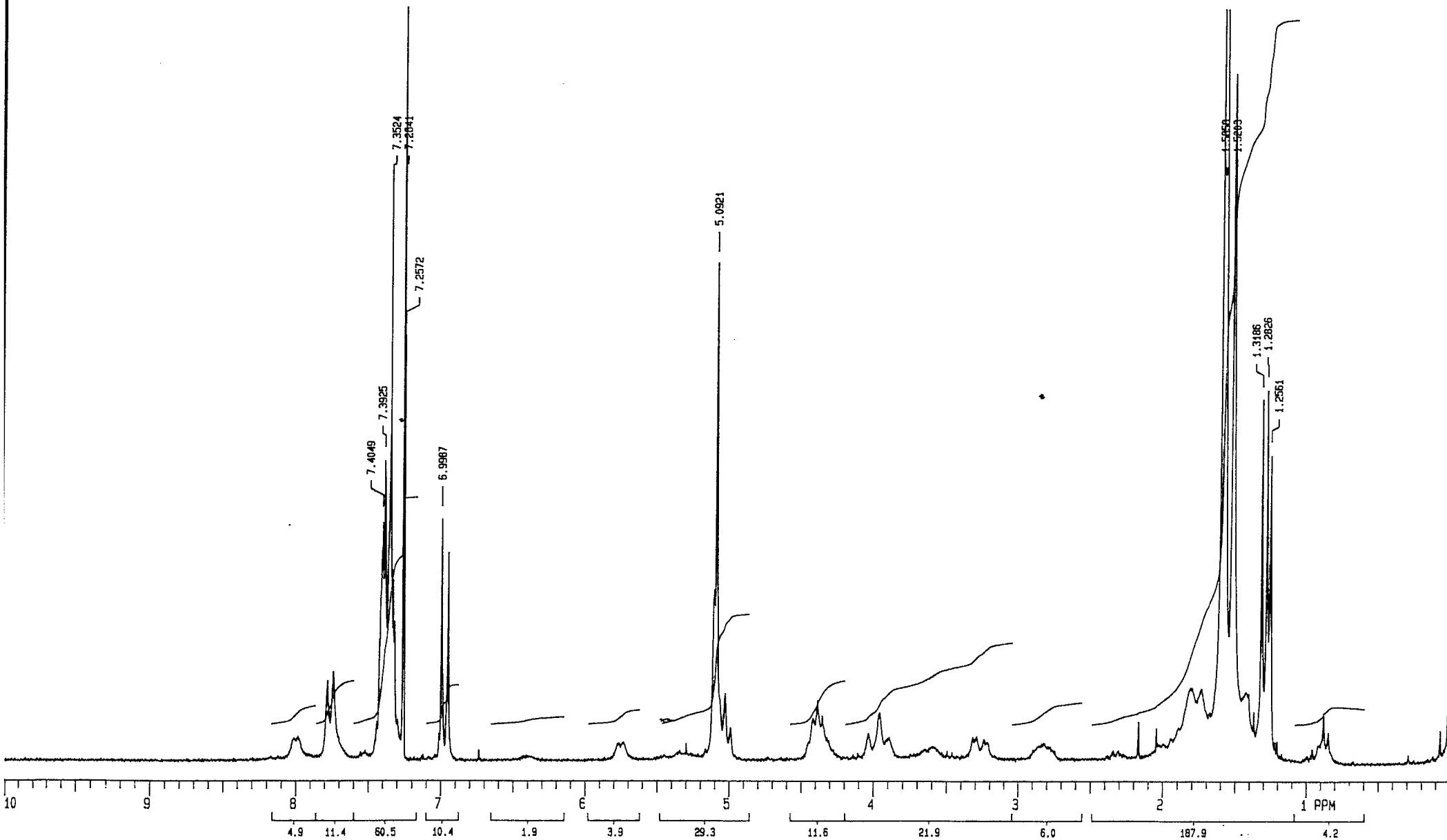
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Varian Japan Ltd. (V)



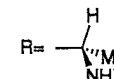
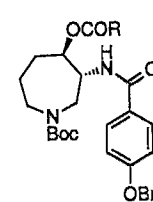
Nucleus 1.000 Freq. 200 MHz
 Spec. Width 3000.3 Hz Offset 0 Hz
 Acq. Time 2.866 sec Delay 0 sec
 Pulse Width 11.5 μ sec Transients 608

Nucleus 1.000 Offset -300.0 Hz
 Mode NNN Power 1750.0 db
 Modulation Mode CCC Freq. 200 Hz
 Pulse Width μ sec Power Mode 1.0

PLOT/PROCESSING
 FN 16 K RE sec CD sec
 LB Hz AF sec CCD
 Width 2000.0 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence S2PUL
 Tube O.D. mm
 Temp. °C
 Solvent CDCl3

SAMPLE 1



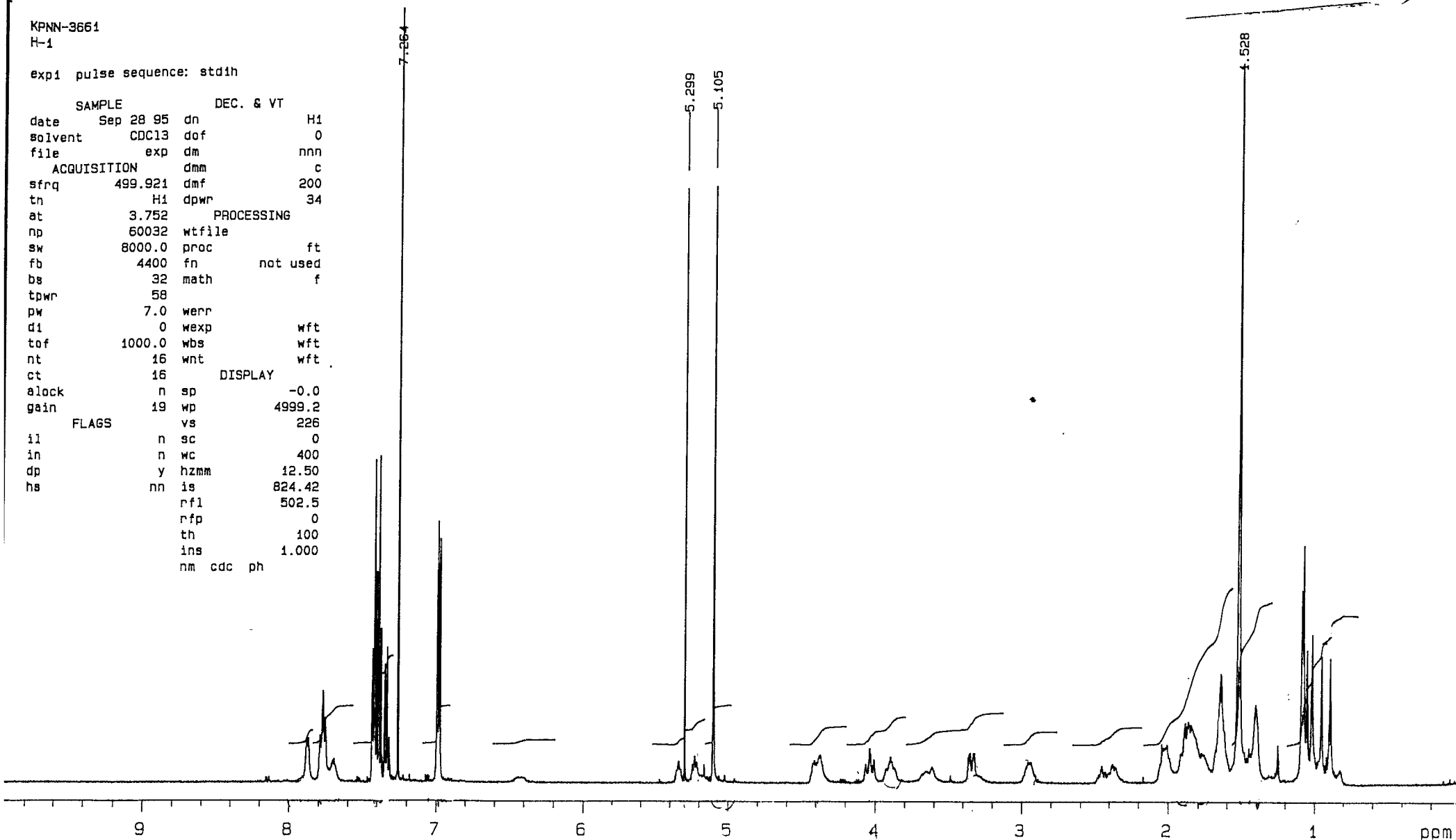
rber 6-47
 10-02-95
 15:06:33
 SEM 200

KPNN-3661

H-1

exp1 pulse sequence: std1h

SAMPLE		DEC. & VT	
date	Sep 28 95	dn	H1
solvent	CDC13	dof	0
file	exp	dm	nnn
ACQUISITION			
sfrq	499.921	dmm	c
th	H1	dmf	200
at	3.752	dpwr	34
PROCESSING			
np	60032	wtfile	
sw	8000.0	proc	ft
fb	4400	fn	not used
bs	32	math	f
tpwr	58		
pw	7.0	werr	
d1	0	wexp	wft
tof	1000.0	wbs	wft
nt	16	wnt	wft
ct	16	DISPLAY	
alock	n	sp	-0.0
gain	19	wp	4999.2
FLAGS			
il	n	sc	0
in	n	wc	400
dp	y	hzmm	12.50
hs	nn	is	824.42
		rfl	502.5
		rfp	0
		th	100
		ins	1.000
		nm	cdc ph

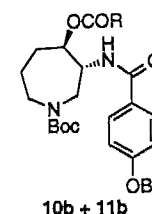


Nucleus H1 Freq. 499.9214 MHz
 Spec. Width 8000.0 Hz Offset 1000.0 Hz
 Acq. Time 3.752 sec Delay 0 sec
 Pulse Width 7.0 μ sec Transients 16

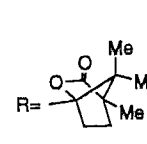
Nucleus H1 Offset 0 Hz
 Mode nnn Power not used db
 Modulation Mode c Freq. 200 Hz
 Pulse Width --- μ sec Power Mode 34

PLOT/PROCESSING
 F1 not used k RE --- sec CD --- sec
 LR not used Hz not used sec CD ---
 Width 4999.2 Hz/ppm Start -0.0 Hz/ppm
 Reference ---

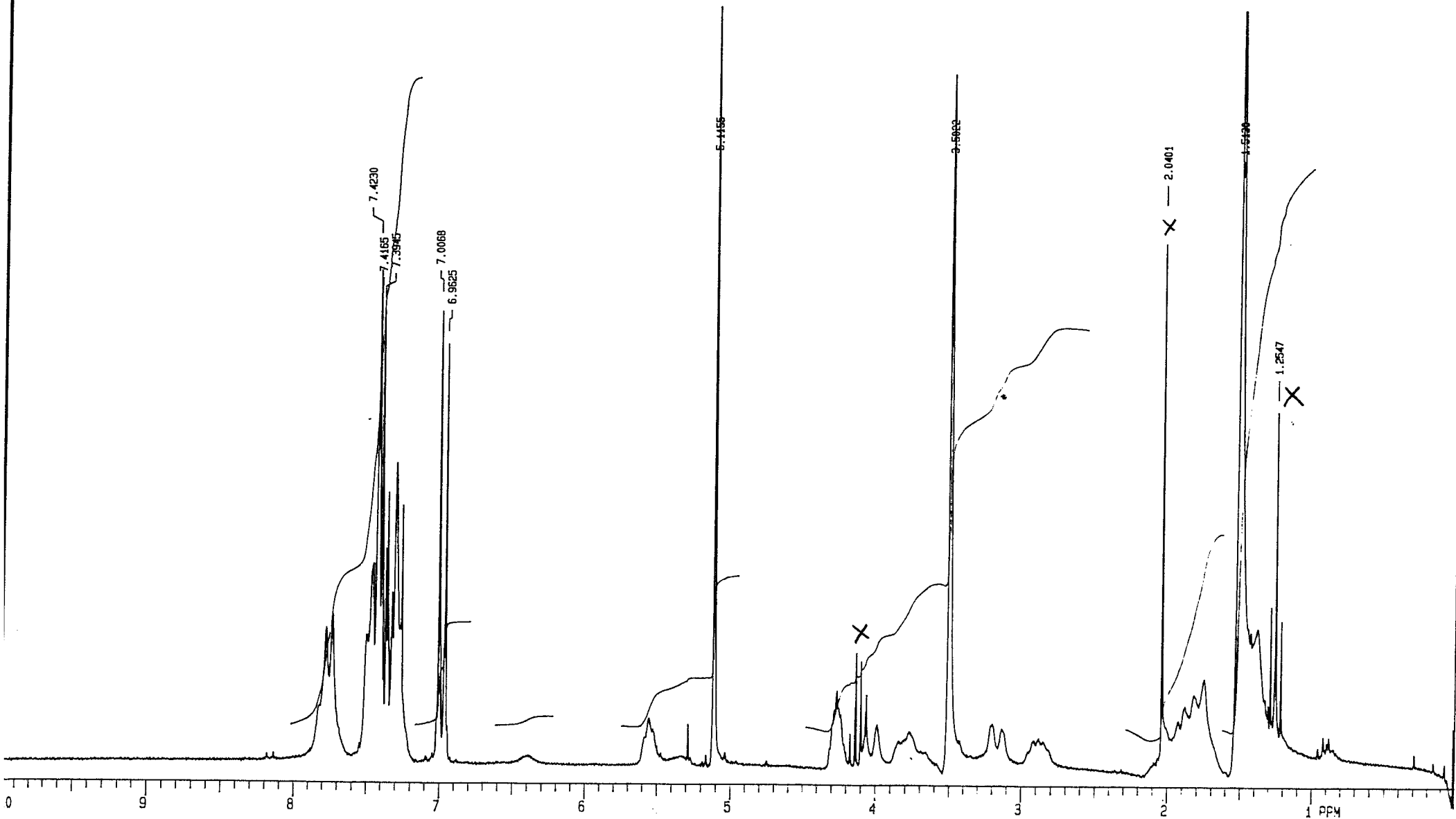
EXPERIMENT
 Pulse Sequence std1
 Tube O.D. --- mm
 Temp. not used °C
 Solvent CDC13



10b + 11b



Number 0-57
 File EXP
 Date Sep 28 95
 XL 500

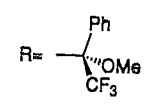
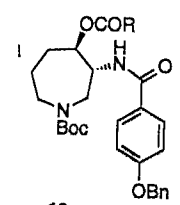


Nucleus 1.000 Freq. 200 MHz
 Spec. Width 2000.3 Hz Offset 0 Hz
 Acq. Time 2.656 sec Delay 0 sec
 Pulse Width 11.5 µsec Transients 16

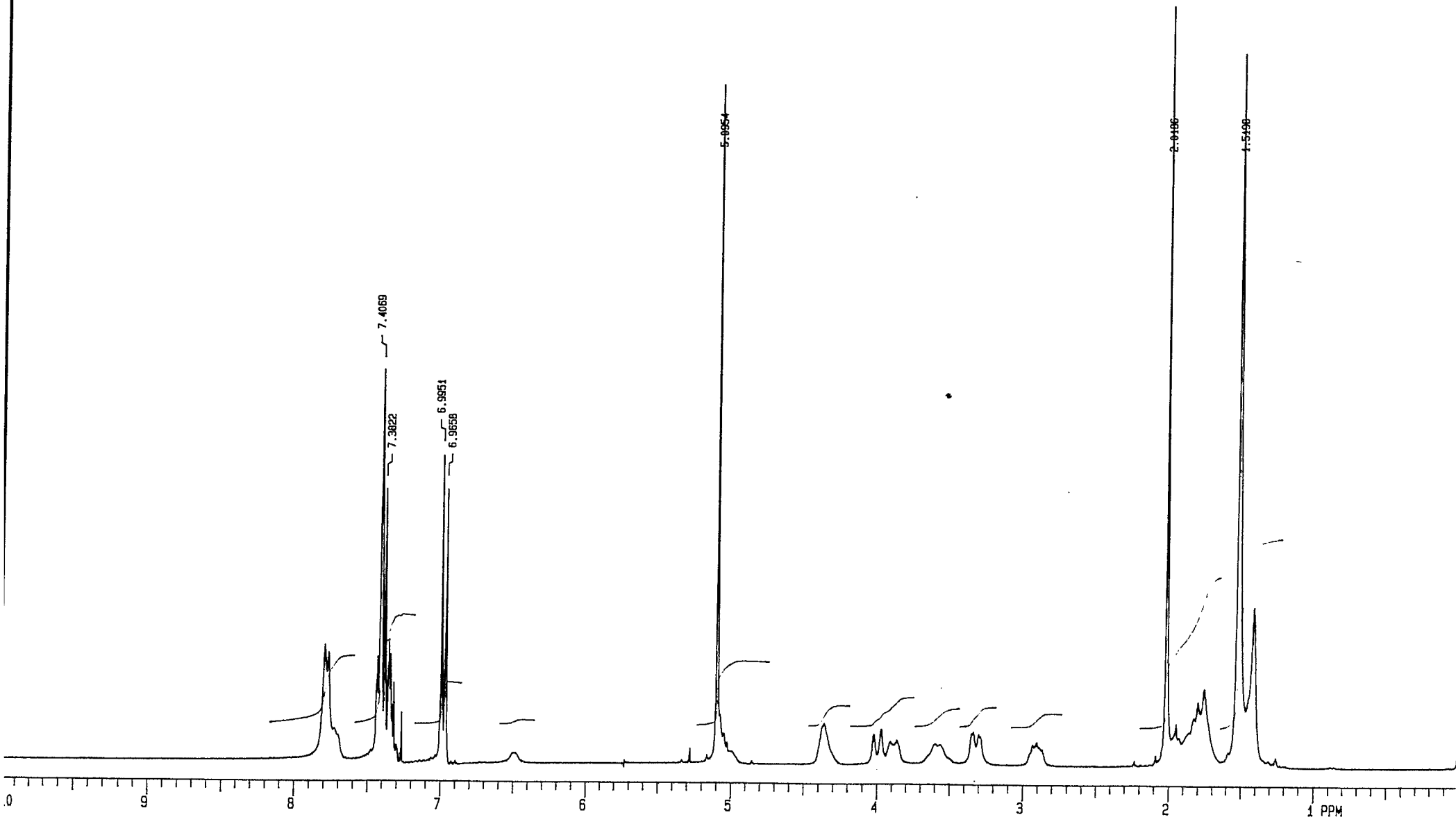
Nucleus 1.000 Offset -300.0 Hz
 Mode NNN Power 1750.0 db
 Modulation Mode CCC Freq. 200 Hz
 Pulse Width µsec Power Mode 1.0

PLOT/PROCESSING
 FN 16 K RE sec CD sec
 LB Hz AF sec CCD
 Width 2000.0 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence SPUL
 Tube O.D. mm
 Temp °C
 Solvent CDCl3



Number E-72
 File H
 Date 11-16-95
 18:39:37
 XL-SEM 200



Nucleus 1.000 Freq. 300 MHz
 Spec. Width 500.5 Hz Offset 0 Hz
 Acq. Time 3.335 sec Delay 1.670 sec
 Pulse Width 9.4 μsec Transients 16

Nucleus 1.000 Offset -450.0 Hz
 Mode NNN Power 1700.0 db
 Modulation Mode C Freq. 200 Hz
 Pulse Width μsec Power Mode 1.0

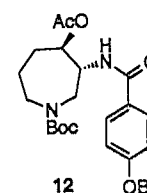
PLT/PROCESSING

FN 32 K RE sec CD sec
 LB Hz AF sec CCD
 Width 3000.9 Hz/ppm Start 0 Hz/ppm
 Reference

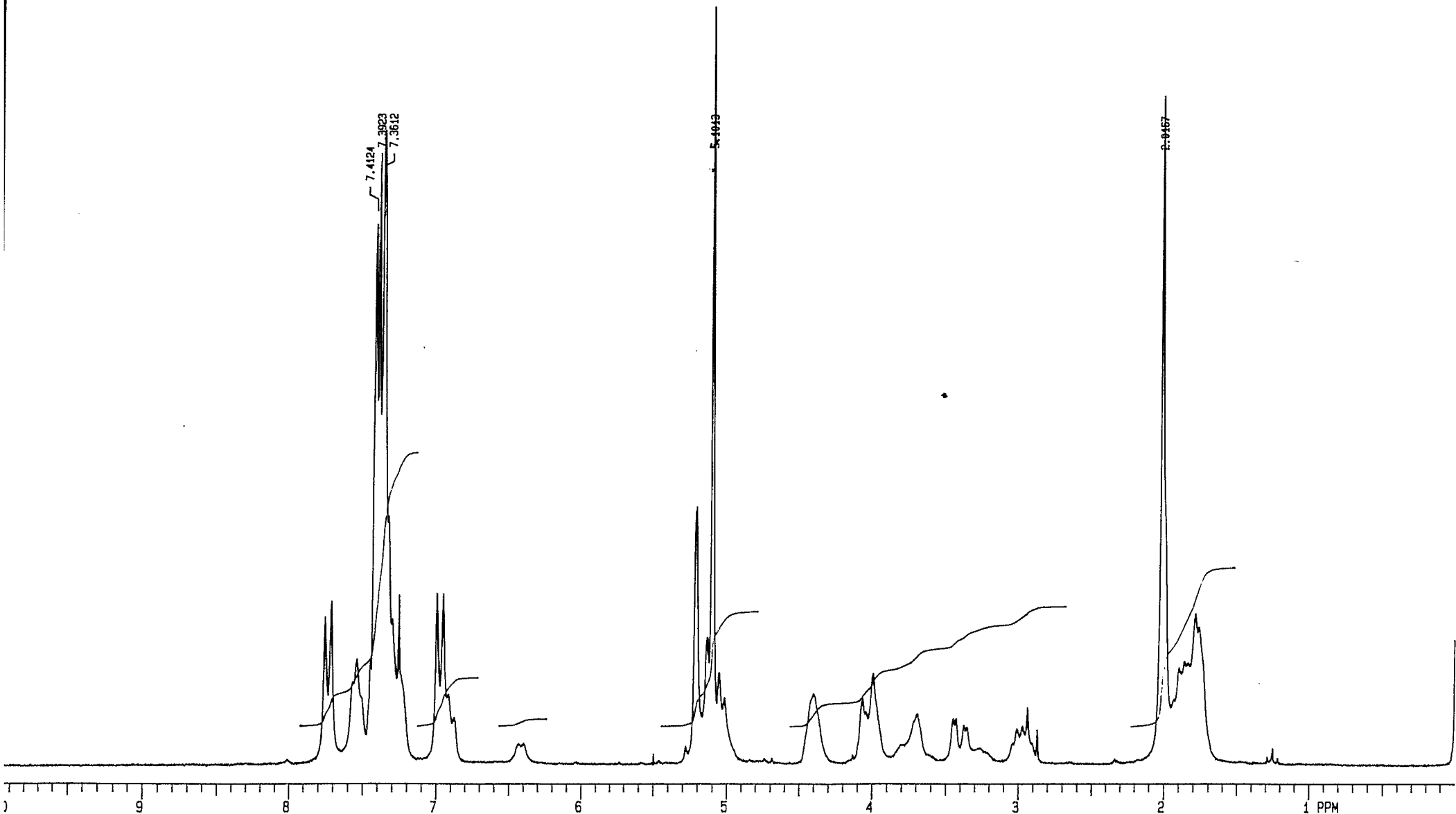
EXPERIMENT

Pulse Sequence SPUL
 Tube O.D. mm
 Temp °C
 Solvent CDCl₃

SAMPLE
 AC LIOPASE



Number
 File H
 Date 01-30-97
 09:42:25
 XL GEM 300



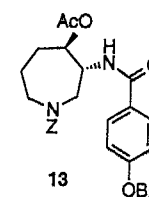
Nucleus 1.000 Freq. 200 MHz
 Spec. Width 3000.3 Hz
 Acq. Time 2.556 sec
 Pulse Width 11.5 µsec

Nucleus 1.000 Offset -300.0 Hz
 Mode NNN Power 1750.0 db
 Modulation Mode CCC Freq. 200 Hz
 Pulse Width µsec Power Mode 1.0

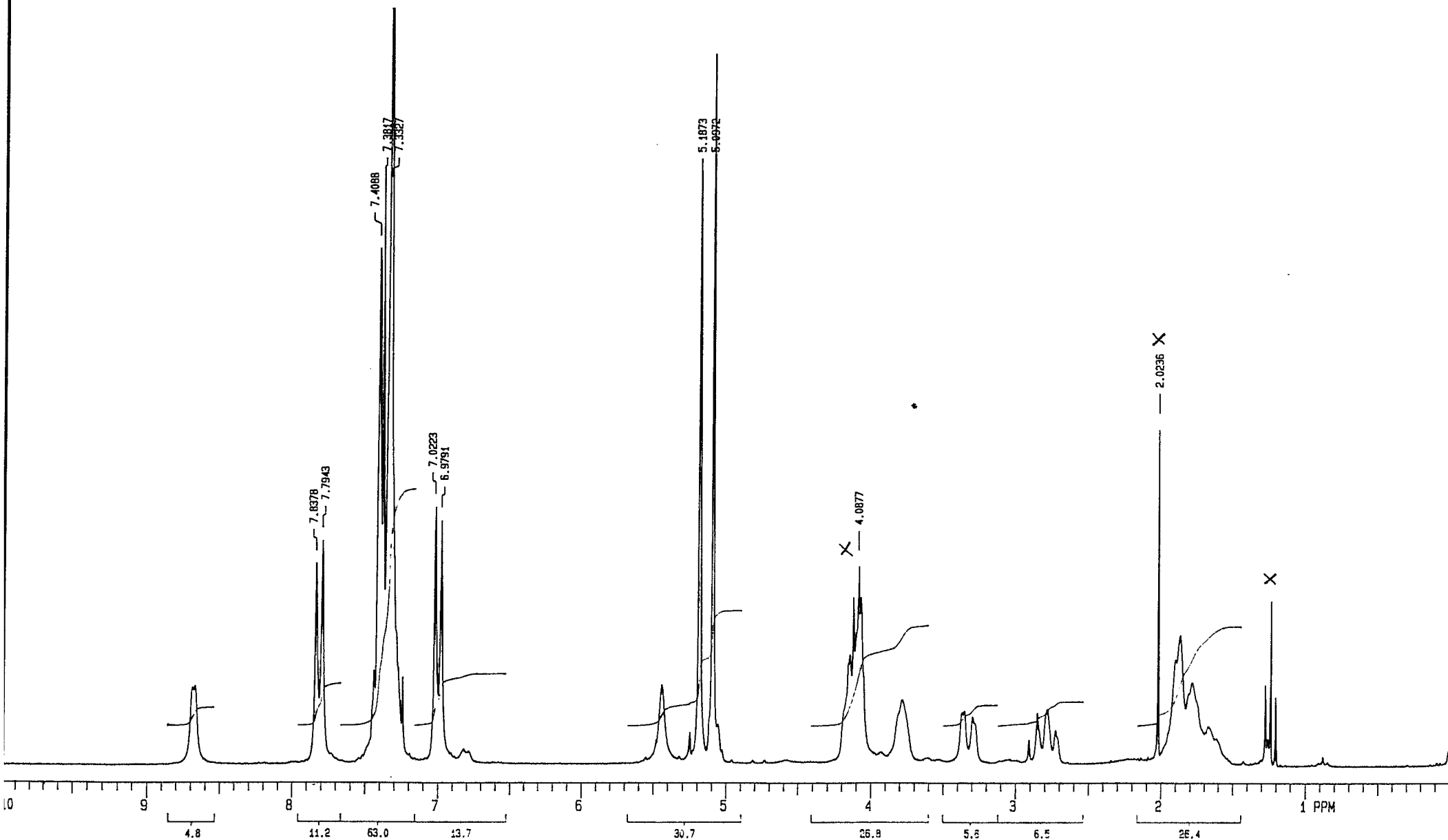
PLOT/PROCESSING
 FN 15 K RE sec CD sec
 LB. Hz AF sec CCD
 Width 2000.0 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence S2PUL
 Tube O.D. mm
 Temp °C
 Solvent CDCl3

SAMPLE
 Z AC



Number
 File H
 Date 01-09-96
 06:07:08
 GEM 200
 XL



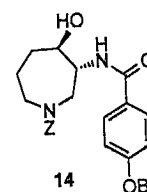
OBSERVE
 Nucleus 1.000 Freq 200 MHz
 Spec. Width 3000.3 Hz
 Acq. Time 2.566 sec
 Pulse Width 10.0 µsec
 Delay 0 sec
 Transients 16

DECOUPLE
 Nucleus 1.000 Offset -300.0 Hz
 Mode NMN Power 1750.0 db
 Modulation: CCC 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 2000.0 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence S2PUL
 Tube O.D. mm
 Temp °C
 Solvent CDCL3

SAMPLE
 KASSEITAI Z PURE



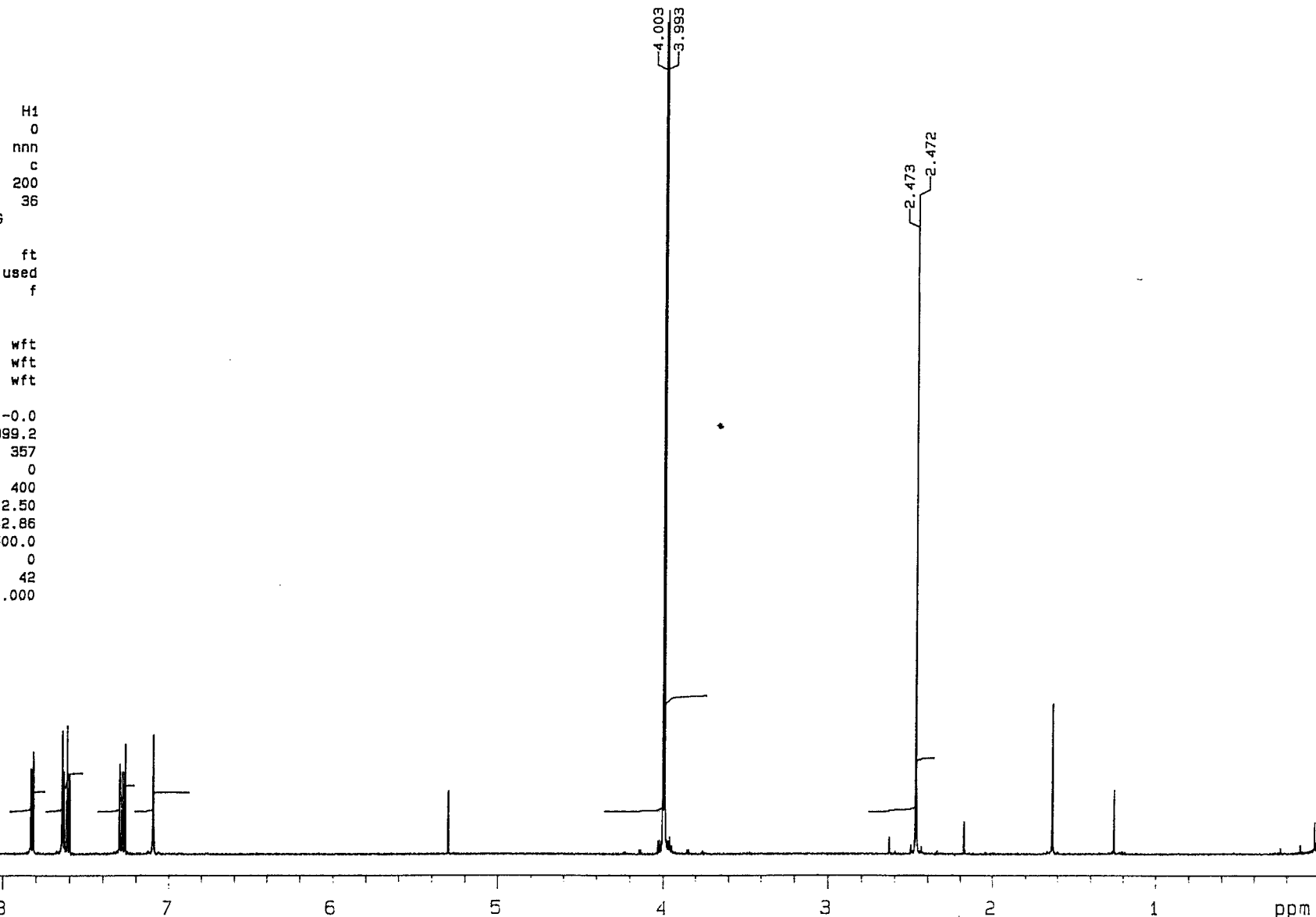
Number
 File H
 Date 12-20-96
 12:36:56
 XL GEH 200

in Japan Ltd. (VW)

KPNN-3732
H-1

exp5 pulse sequence: stdih

SAMPLE		DEC. & VT	
date	Jun 6 96	dn	H1
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	499.921	dmf	200
tn	H1	dpwr	36
at	3.752	PROCESSING	
np	60032	wtfile	
sw	8000.0	proc	ft
fb	4400	fn	not used
bs	32	math	f
tpwr	61		
pw	8.0	werr	
d1	0	wexp	wft
tof	1000.0	wbs	wft
nt	4	wnt	wft
ct	4	DISPLAY	
alock	n	sp	-0.0
gain	13	wp	4999.2
FLAGS		vs	357
il	n	sc	0
in	n	wc	400
dp	y	hzmm	12.50
hs	nn	is	1942.86
		rfl	500.0
		rfp	0
		th	42
		ins	1.000
		nm	cdc ph



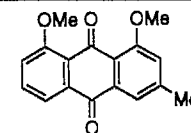
Nucleus H1 Freq. 499.9214 MHz
Spec. Width 8000.0 Hz Offset 1000.0 Hz
Acq. Time 3.752 sec Delay 0 sec
Pulse Width 8.0 μ sec Transients 4

Nucleus H1 Offset 0 Hz
Mode nnn Power not used db
Modulation: Mode C Freq. 200 Hz
Pulse Width — μ sec Power Mode 36

FW not used K RE — sec CD — sec
LB not used Hz HAT not used sec CCD —
Width 4999.2 Hz/ppm Start -0.0 Hz/ppm
Reference —

Pulse Sequence stdih
Tube O.D. — mm
Temp. not used °C
Solvent CDCl3

SAMPLE
KPNN-3732
H-1



16c

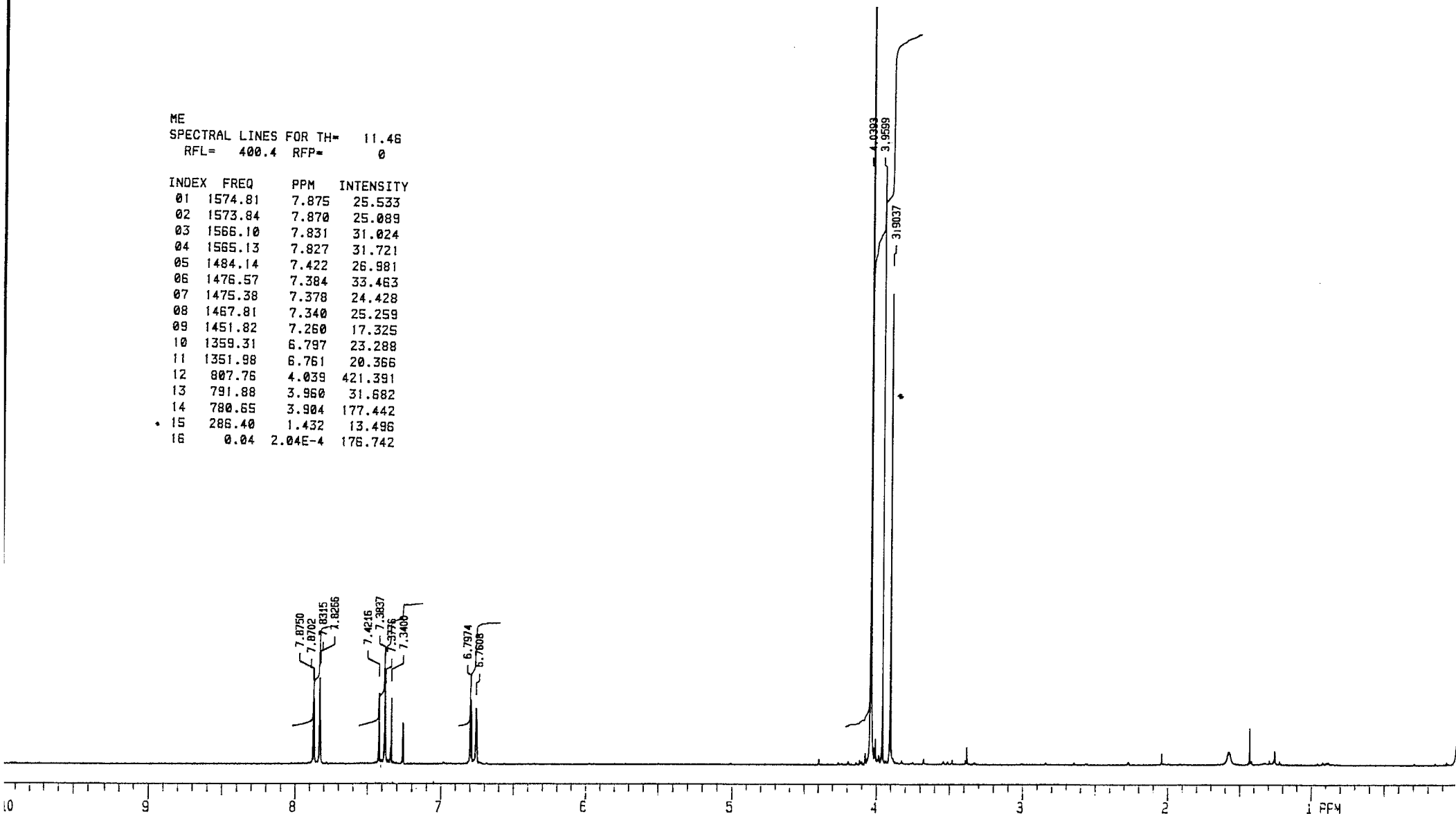
NO 997260 J

Number —
File exp
Date Jun 6 96
XL 500

Varian Japan Ltd. (V)

ME
SPECTRAL LINES FOR TH= 11.46
RFL= 400.4 RFP= 0

INDEX	FREQ	PPM	INTENSITY
01	1574.81	7.875	25.533
02	1573.84	7.870	25.089
03	1566.10	7.831	31.024
04	1565.13	7.827	31.721
05	1484.14	7.422	26.981
06	1476.57	7.384	33.463
07	1475.38	7.378	24.428
08	1467.81	7.340	25.259
09	1451.82	7.260	17.325
10	1359.31	6.797	23.288
11	1351.98	6.761	20.366
12	807.76	4.039	421.391
13	791.88	3.960	31.682
14	780.65	3.904	177.442
15	286.40	1.432	13.496
16	0.04	2.04E-4	176.742



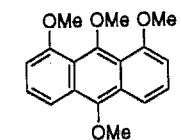
OBSERVE
Nucleus 1.000 Freq. 200 MHz
Spec. Width 2000.3 Hz
Acq. Time 2.656 sec
Pulse Width 11.5 μsec

DECOUPLE
Nucleus 1.000 Offset -300.0 Hz
Mode NNN Power 1750.0 db
Modulation Mode CCC Freq. 200 Hz
Pulse Width μsec Power Mode 1.0

PLOT/PROCESSING
P1 15 K RE sec CD sec
LB Hz AF sec CCD
Width 2000.0 Hz/ppm Start 0 Hz/ppm
Reference

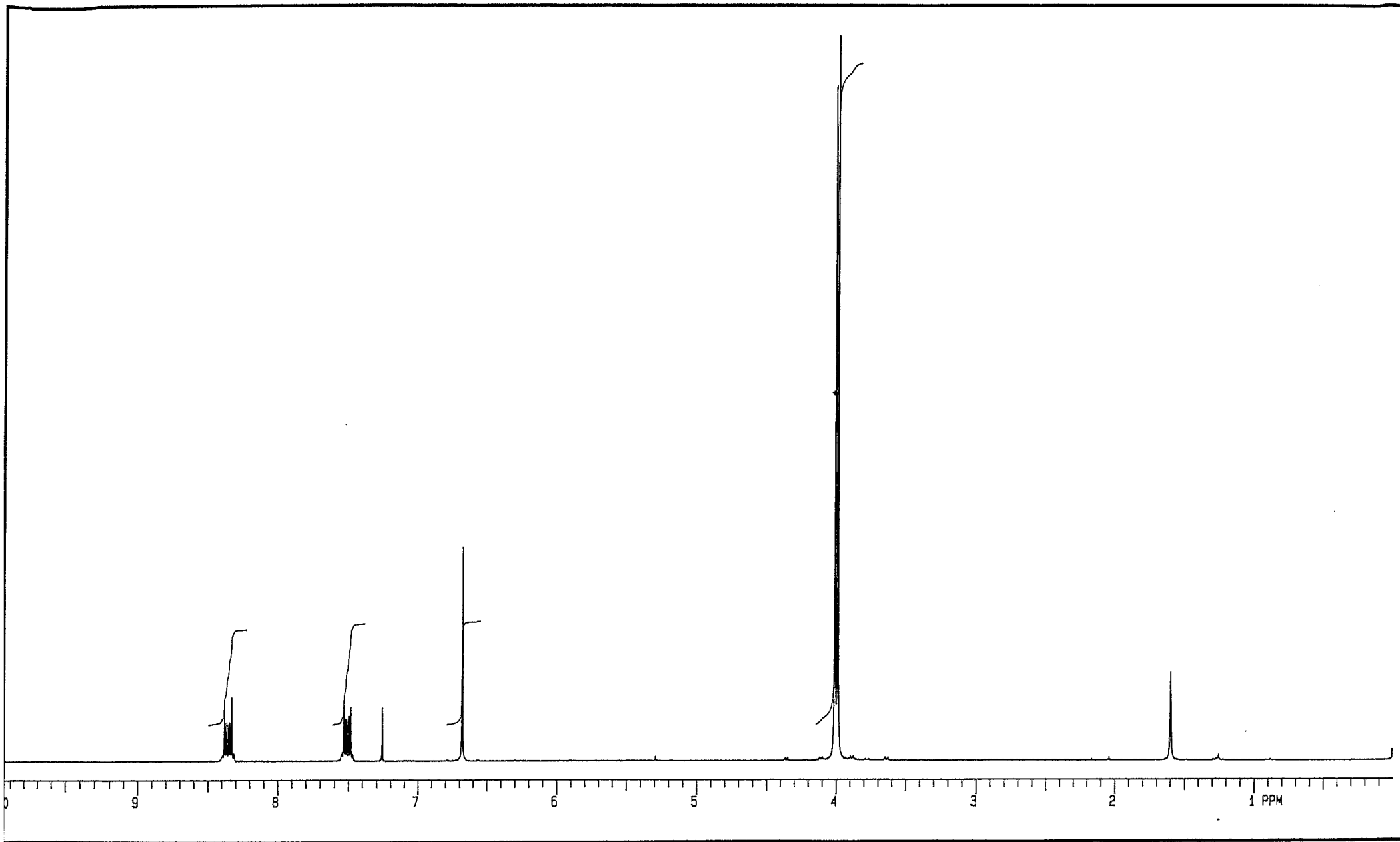
EXPERIMENT
Pulse Sequence S2PUL
Tube O.D. mm
Temp. °C
Solvent CDCL3

SAMPLE
ME

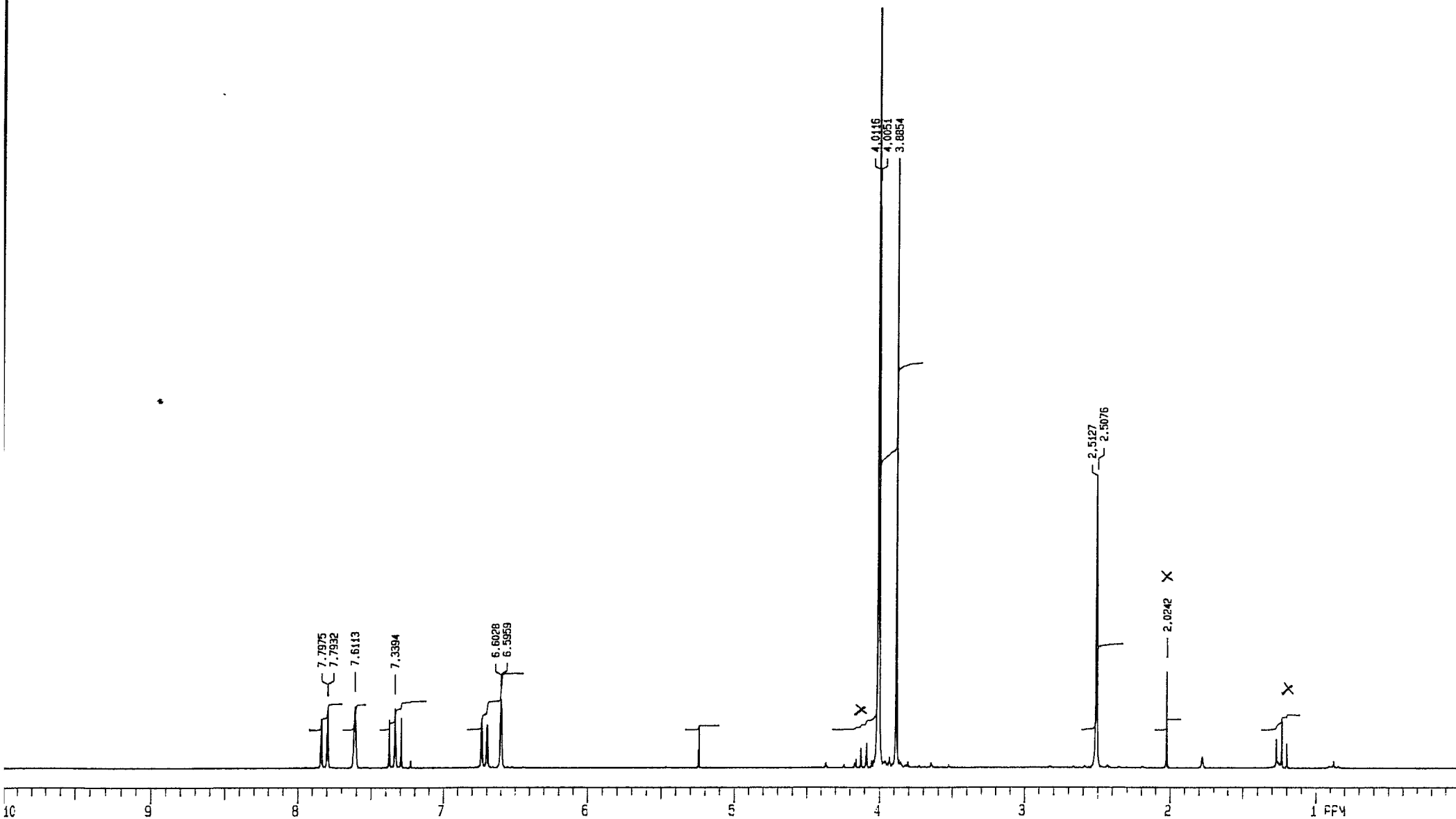


17a
NO 997260 J

Number
File H
Date 05-17-96
13:54:01
XL GEM 200



OBSERVE Nucleus <u>1.000</u> Freq <u>200</u> MHz Spec. Width <u>0.3</u> Hz Offset <u>0</u> Hz Acq. Time <u>2.555</u> sec Delay <u>0</u> sec Pulse Width <u>1.5</u> µsec Transients <u>15</u>	DECOUPLE Nucleus <u>1.000</u> Offset <u>300.0</u> Hz Mode <u>MMN</u> Power <u>1750.0</u> db Modulation: <u>MSSC</u> Freq <u>200</u> Hz Pulse Width <u> </u> µsec Power Mode <u>1.0</u>	PLOT/PROCESSING FN <u>15</u> K RE <u> </u> sec CD <u> </u> sec LB <u> </u> Hz AF <u> </u> sec CCD <u> </u> Width <u>0.0</u> Hz/ppm Start <u>0</u> Hz/ppm Reference <u> </u>	EXPERIMENT Pulse Sequence <u>zgpg30</u> Tube O.D. <u> </u> mm Temp. <u> </u> °C Solvent <u>CDCl3</u>	SAMPLE 1. 4	 17b NO 997280 J	Number <u> </u> File <u> </u> Date <u>10-07</u> Time <u>13:00:19</u> XGEN <u>200</u>
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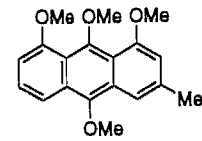
Nucleus 1.000 Freq. 200 MHz
 Spec. Width 3000.3 Hz Offset 0 Hz
 Acq. Time 2.666 sec Delay 0 sec
 Pulse Width 11.5 µsec Transients 16

Nucleus 1.000 Offset -300.0 Hz
 Mode NNN Power 1750.0 db
 Modulation: Mod CCC 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 2000.0 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence S2PUL
 Tube O.D. mm
 Temp. °C
 Solvent CDCl3

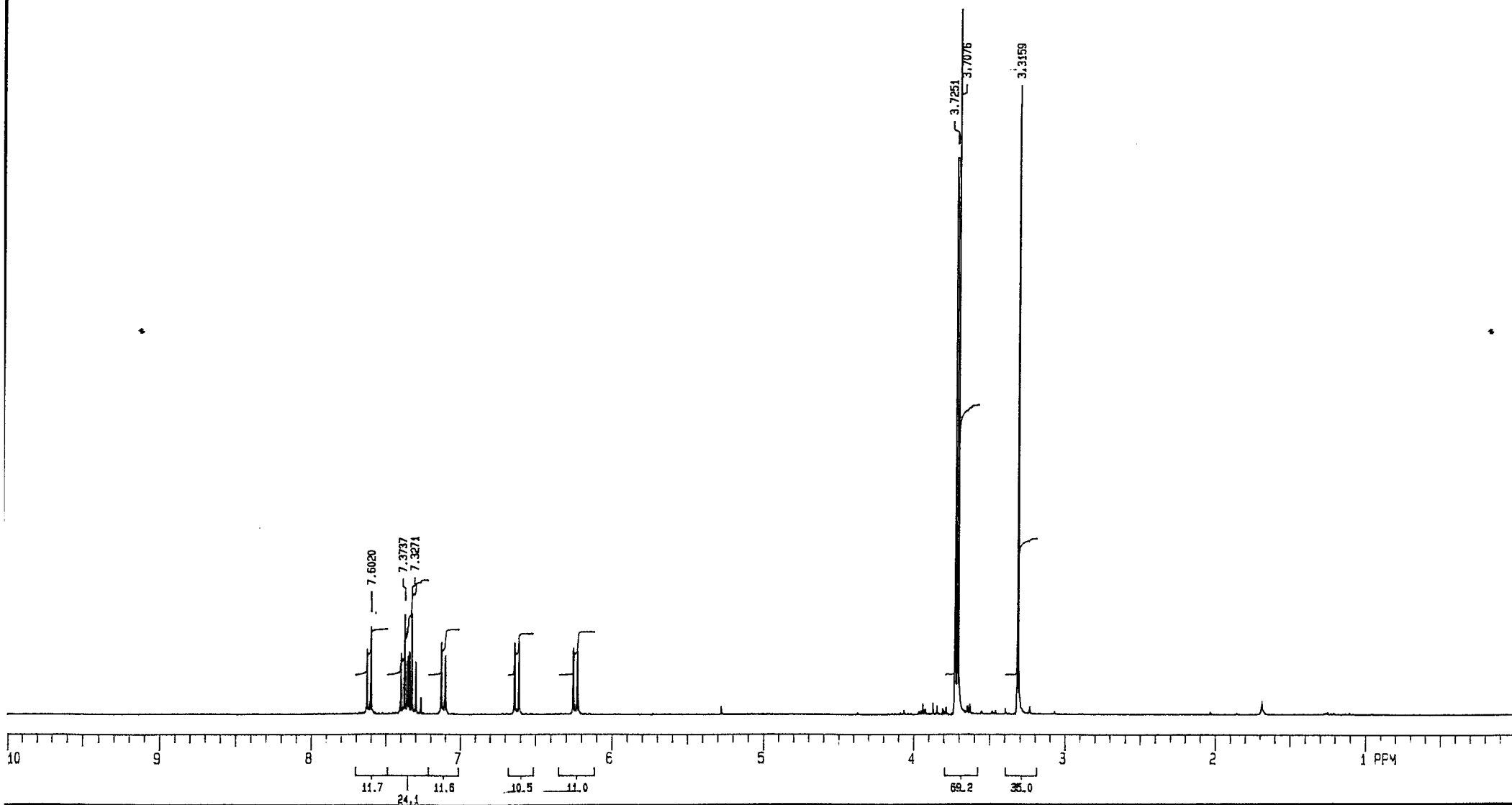
SAMPLE
 6GATUINITII FR4



17c
 NO 987280 J

Number
 File H
 Date 06-03-96
 OS: 48: 07
 XL: GEM 200

Varian Japan Ltd. (VA)



OBSERVE

Nucleus 1.000 Freq. 300 MHz
 Spec. Width 4500.5 Hz Offset 0 Hz
 Acq. Time 3.335 sec Delay 1.670 sec
 Pulse Width 9.4 μ sec Transients 16

DECOUPLE

Nucleus 1.000 Offset -450.0 Hz
 Mode HNH Power 1700.0 db
 Modulation: Mode C Freq. 200 Hz
 Pulse Width μ sec Power Mode 1.0

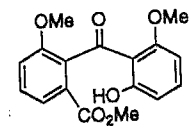
PLOT/PROCESSING

FN 32 K RE sec CD sec
 LB Hz AF sec CCD
 Width 3000.9 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT

Pulse Sequence S2PUL
 Tube O.D. mm
 Temp. °C
 Solvent CDCl3

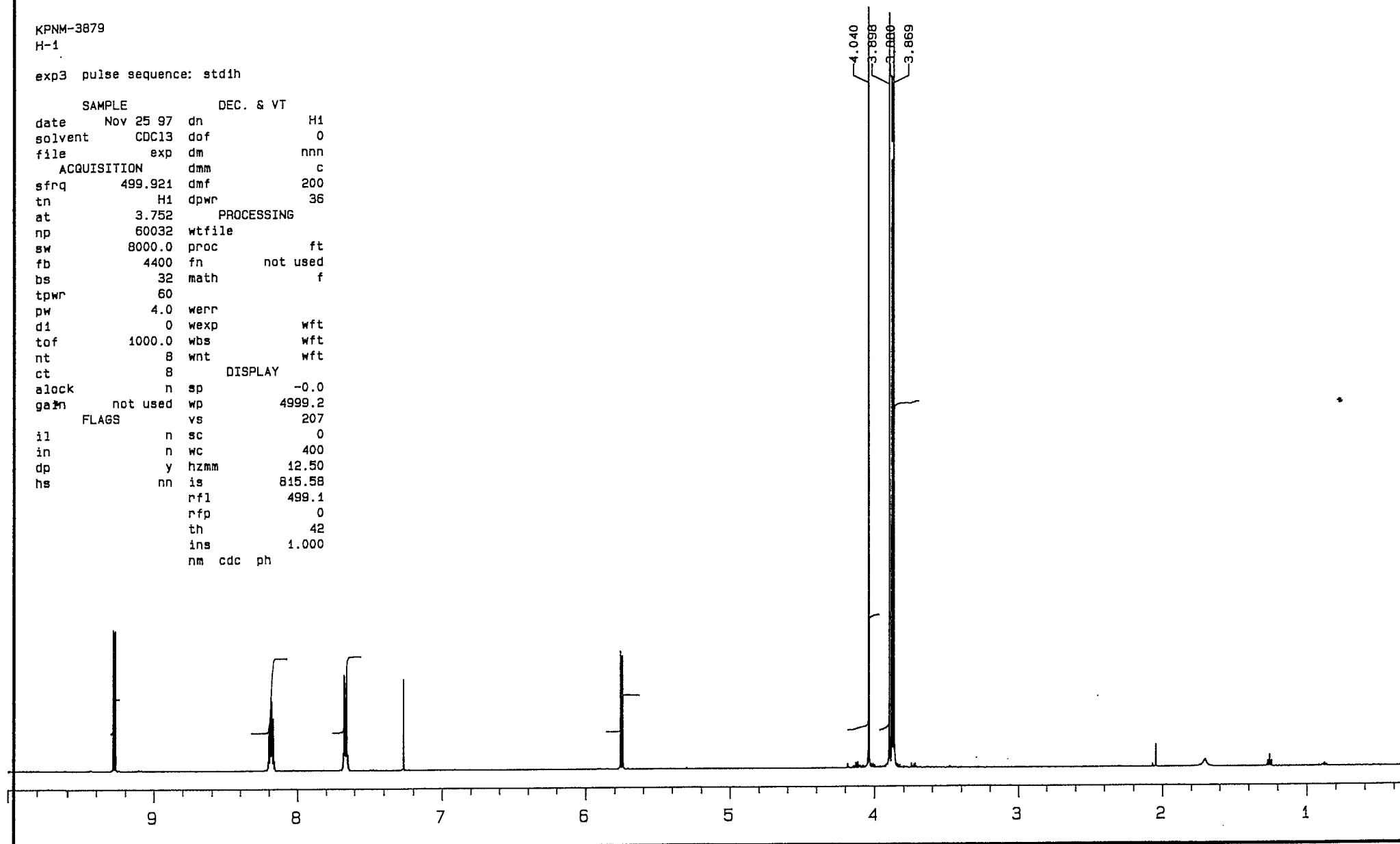
SAMPLE
 HIKARI SANIN FR3



Number 58
 File H
 Date 05-31-96
 10:50:06
 XL GEN 300

H-1

SAMPLE			DEC. & VT	
date	Nov 25 97	dn		H1
solvent	CDC13	dof		0
file	exp	dm		nnn
ACQUISITION		dmm		c
sfrq	499.921	dmf		200
tn	H1	dpwr		36
at	3.752	PROCESSING		
np	60032	wtfile		
sw	8000.0	proc		ft
fb	4400	fn	not used	
bs	32	math		f
tpwr	60			
pw	4.0	werr		
d1	0	wexp		wft
tof	1000.0	wbs		wft
nt	8	wnt		wft
ct	8	DISPLAY		
alock	n	sp		-0.0
gain	not used	wp		4999.2
FLAGS		vs		207
il	n	sc		0
in	n	wc		400
dp	y	hzmm		12.50
hs	nn	is		815.58
		rfl		499.1
		rfp		0
		th		42
		ins		1.000
		nm	cdc ph	



RESERVE

Nucleus H1 Freq. 499.9214 MHz
Spec. Width 8000.0 Hz Offset 1000.0 Hz
Acq. Time 3.752 sec Delay 0 sec
Pulse Width 4.0 μ sec Transients 8

5. CONCLUSIONS

Nucleus H1 Offset 0 Hz
Mode 000 Power not used dB
Modulation: Mode C Freq. 200 Hz
Pulse Width μ sec Power Mode 36

PLOT/PROCESSING

```

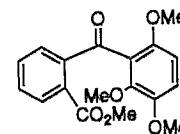
FNdnot used K RE_____sec CD_____sec
LBnot used H7NF used_____sec CCD_____
Width 4999.2 Hz/ppm Start_____ -0.0 Hz/ppm
Reference_____

```

EXPERIMENT

Pulse Sequence 2pu1
Tube O.D. _____ mm
Temp. not used °C
Solvent CDC13

SAMPLE
RPN-3879
H-1



20
NO 997260 J

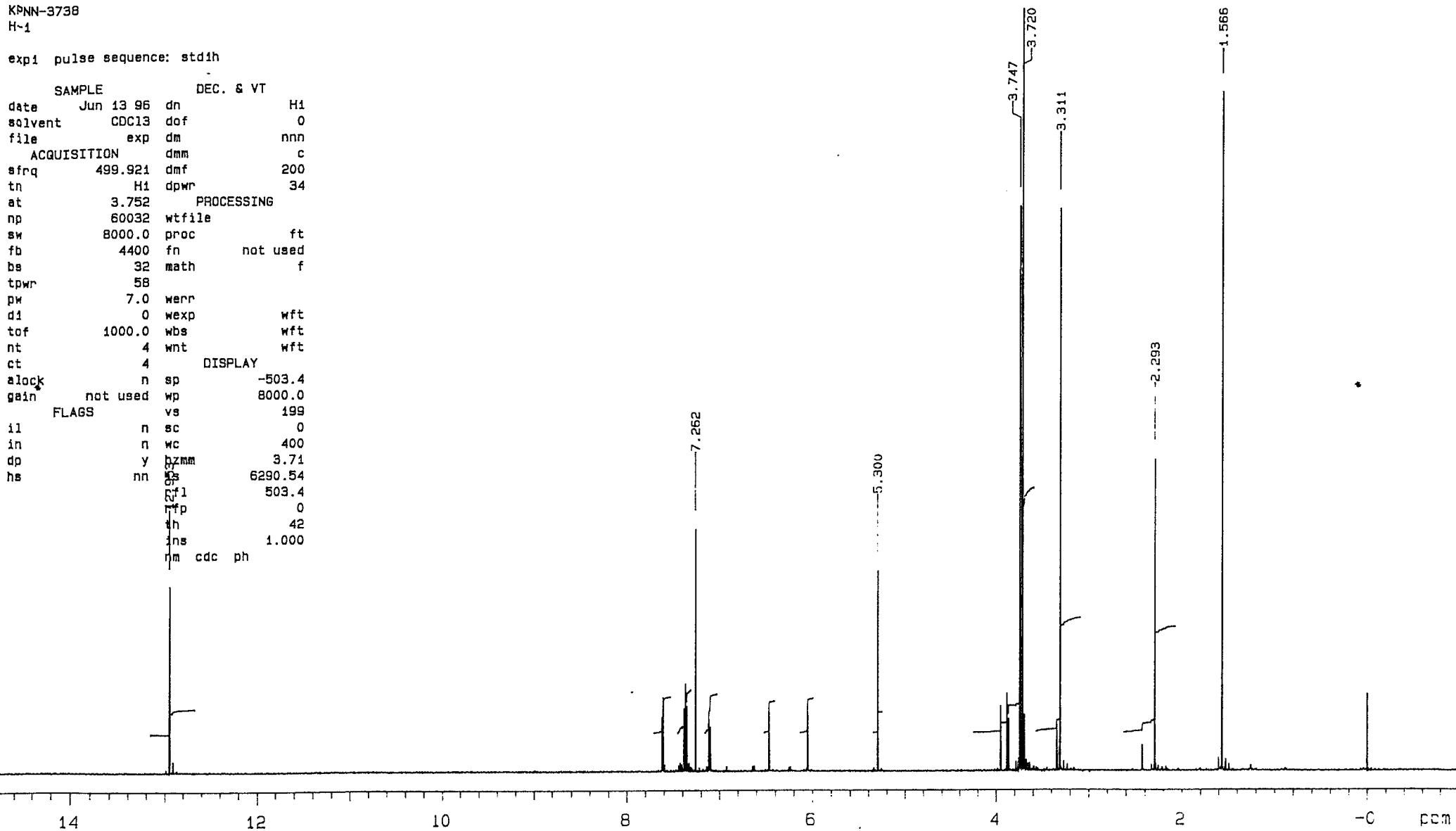
Number _____
File _____
Date Nov _____
XL- _____

Varian Jap.

KPNN-3738
H-1

exp1 pulse sequence: stdih

SAMPLE		DEC. & VT	
date	Jun 13 96	dn	H1
solvent	CDC13	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	499.921	dmf	200
tn	H1	dpr	34
at	3.752	PROCESSING	
np	60032	wtfile	
sw	8000.0	proc	ft
fb	4400	fn	not used
bs	32	math	f
tpwr	58		
pw	7.0	werr	
d1	0	wexp	wft
tof	1000.0	wbs	wft
nt	4	wnt	wft
ct	4	DISPLAY	
alock	n	sp	-503.4
gain	not used	wp	8000.0
FLAGS		vs	199
il	n	sc	0
in	n	wc	400
dp	y	bzmm	3.71
hs	nn	cs	6290.54
		cp1	503.4
		mp	0
		th	42
		ins	1.000
		rm	cdc ph



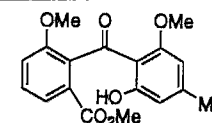
Nucleus	H1	Freq.	499.9214 MHz
Spec. Width	8000.0 Hz	Offset	1000.0 Hz
Acq. Time	3.752 sec	Delay	0 sec
Pulse Width	7.0 µsec	Transients	4

Nucleus	H1	Offset	0 Hz
Mode	nnn	Power	not used db
Modulation Mode	c	Freq.	200 Hz
Pulse Width	µsec	Power Mode	34

FN	not used	k	RE	sec	CD	sec
LN	not used	H	not used	sec	CCD	
Width	8000.0 Hz/ppm	Start	-503.4 Hz/ppm			
Reference						

Pulse Sequence	stdih
Tube O.D.	mm
Temp.	not used °C
Solvent	CDCl3

SAMPLE
KPNN-3738
H-1



22

NO 997260 J

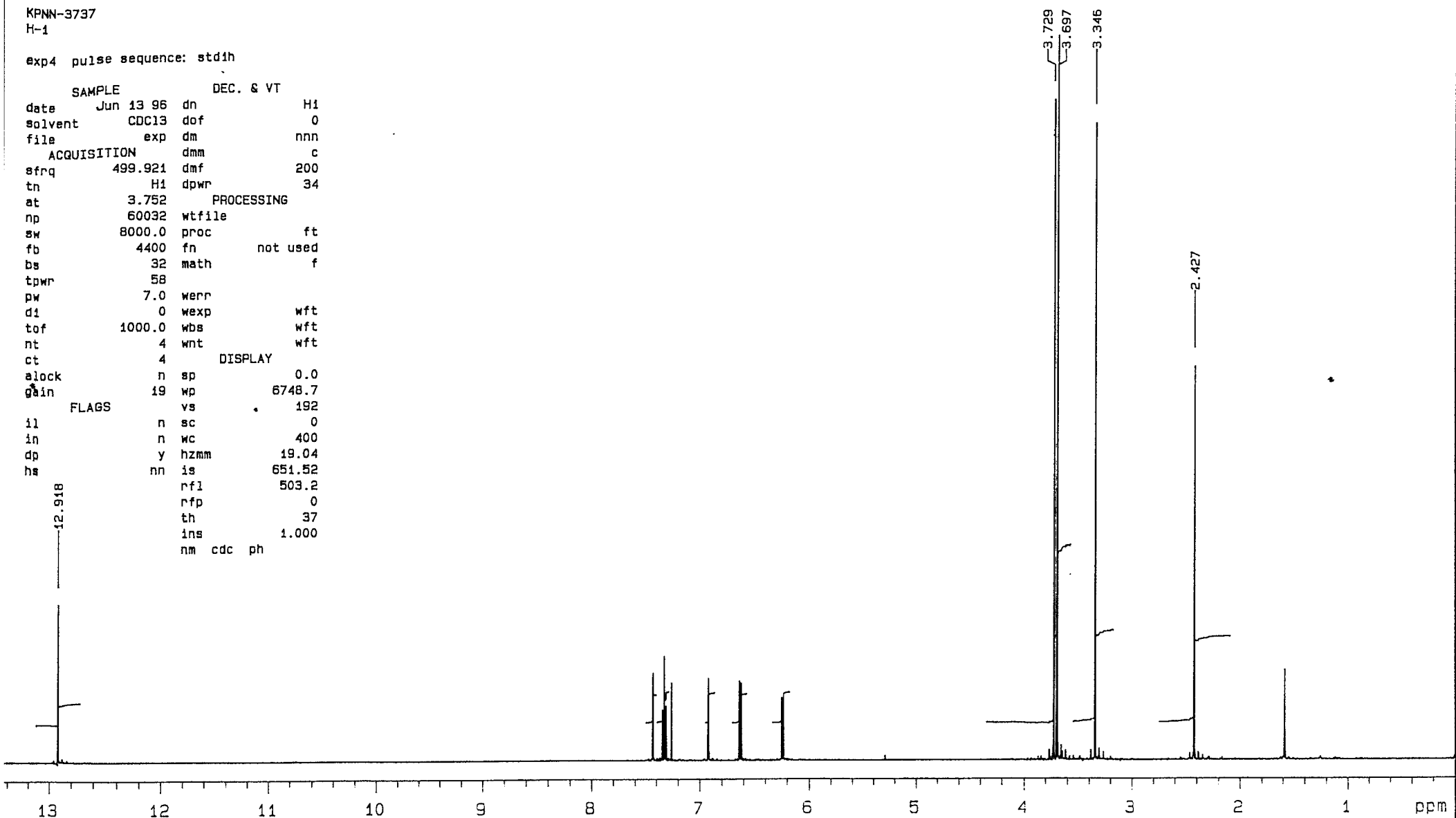
Number	
File	exp
Date	Jun 13 96
XL	500

Varian Japan Ltd. (V)

KPNN-3737
H-1

exp4 pulse sequence: std1h

SAMPLE DEC. & VT
date Jun 13 96 dn H1
solvent CDC13 dof 0
file exp dm nnn
ACQUISITION dmm c
sfrq 499.921 dmf 200
tn H1 dpwr 34
at 3.752 PROCESSING
np 60032 wtfile
sw 8000.0 proc ft
fb 4400 fn not used
bs 32 math f
tpwr 58
pw 7.0 werr
d1 0 wexp wft
tof 1000.0 wbs wft
nt 4 wnt wft
ct 4 DISPLAY
alock n sp 0.0
gain 19 wp 6748.7
FLAGS vs 192
il n sc 0
in n wc 400
dp y hzmm 19.04
hs nn is 651.52
rfl 503.2
rfp 0
th 37
ins 1.000
nm cdc ph



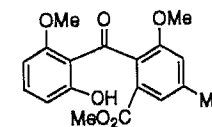
Nucleus H1 Freq. 499.9214 MHz
Spec. Width 8000.0 Hz Offset 1000.0 Hz
Acq. Time 3.752 sec Delay 0 sec
Pulse Width 7.0 μ sec Transients 4

Nucleus H1 Offset 0 Hz
Mode nmn Power not used db
Modulation Mode C Freq. 200 Hz
Pulse Width μ sec Power Mode 34

PL0T/PROCESSING
Pulse Sequence std1h
Tube O.D. mm
Temp. not used °C
Solvent CDCl3
Reference

EXPERIMENT
Pulse Sequence std1h
Tube O.D. mm
Temp. not used °C
Solvent CDCl3

SAMPLE
KPNN-3737
H-1



23

NO 987280 J

Varian Japan Ltd. (V)

Number
File exp
Date Jun 13 96
XL 500

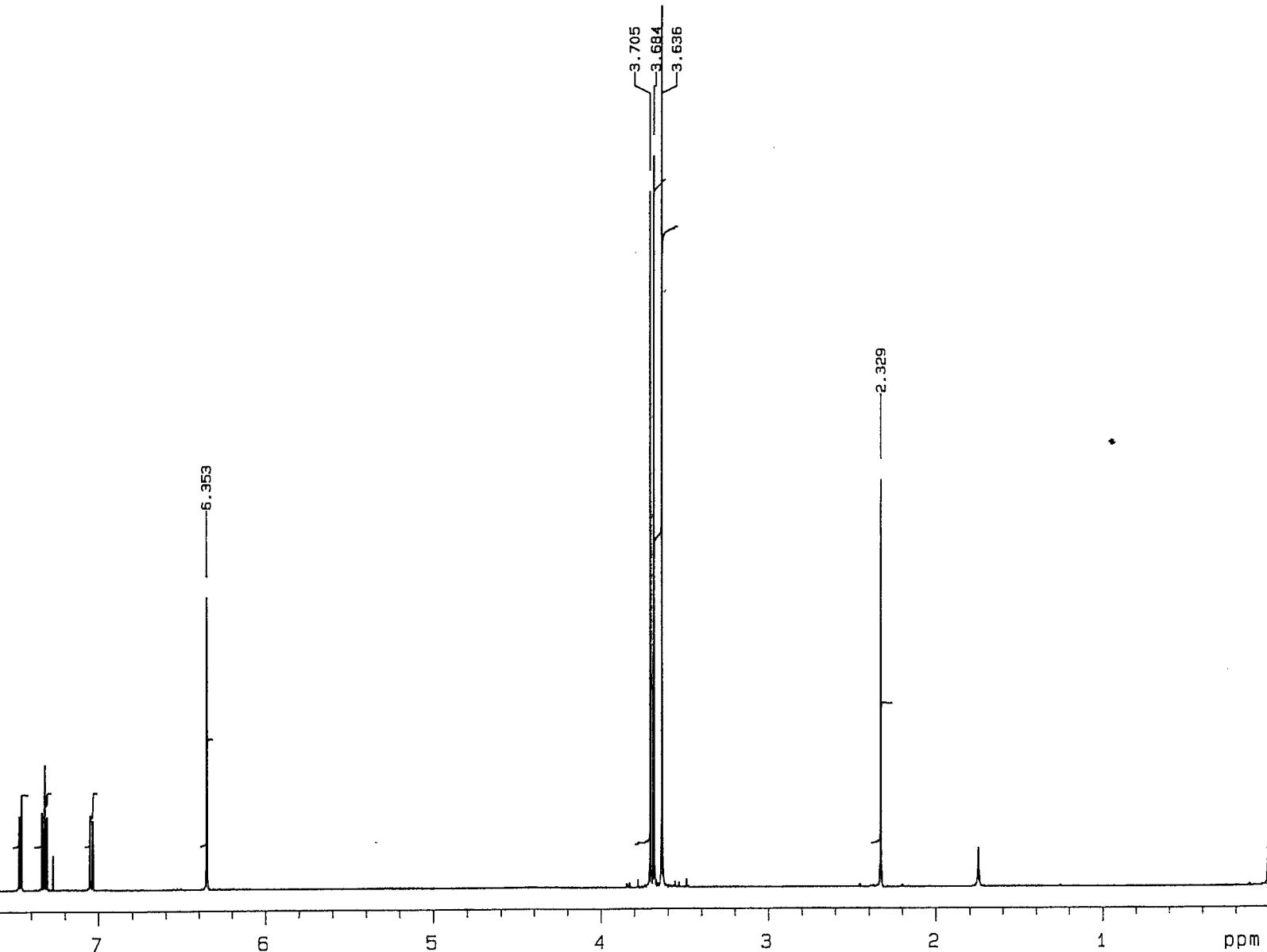
KPNN-3743
H-1

exp5 pulse sequence: std1h

SAMPLE DEC. & VT

date Jul 1 96 dn H1
solvent CDC13 dof 0
file exp dm nnn
ACQUISITION dmm c
sfrq 499.921 dmf 200
th H1 dpwr 36
at 3.752 PROCESSING
np 60032 wtfile
sw 8000.0 proc ft
fb 4400 fn not used
bs 32 math f
tpwr 60
pw 4.0 warr
di 0 wexp wft
tof 1000.0 wbs wft
nt 8 wnt wft
ct 8
*alock n sp -0.0
gain not used wp 4999.2
FLAGS vs 262
il n sc 0
in n wc 400
dp y hzmm 12.50
hs nn is 1500.00
rfl 497.8
rfp 0
th 42
ins 1.000
nm cdc ph

DISPLAY



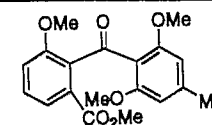
OBSERVE
Nucleus H1 Freq 499.9214 MHz
Spec Width 8000.0 Hz Offset 1000.0 Hz
Acq Time 3.752 sec Delay 0 sec
Pulse Width 4.0 µsec Transients 8

DECOUPLE
Nucleus H1 Offset 0 Hz
Mode nnn Power not used db
Modulation Mode c Freq 200 Hz
Pulse Width µsec Power Mode 36

PLBT/PROCESSING
RN not used K RE sec CD sec
LB not used H2 P used sec CCD
Width 4999.2 Hz/ppm Start -0.0 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence 52pu1
Tube O.D. mm
Temp. not used °C
Solvent CDCl3

SAMPLE
KPNN-3743
H-1



24

NO 997280 J

Number
File exp
Date Jul 1 96
XL 500

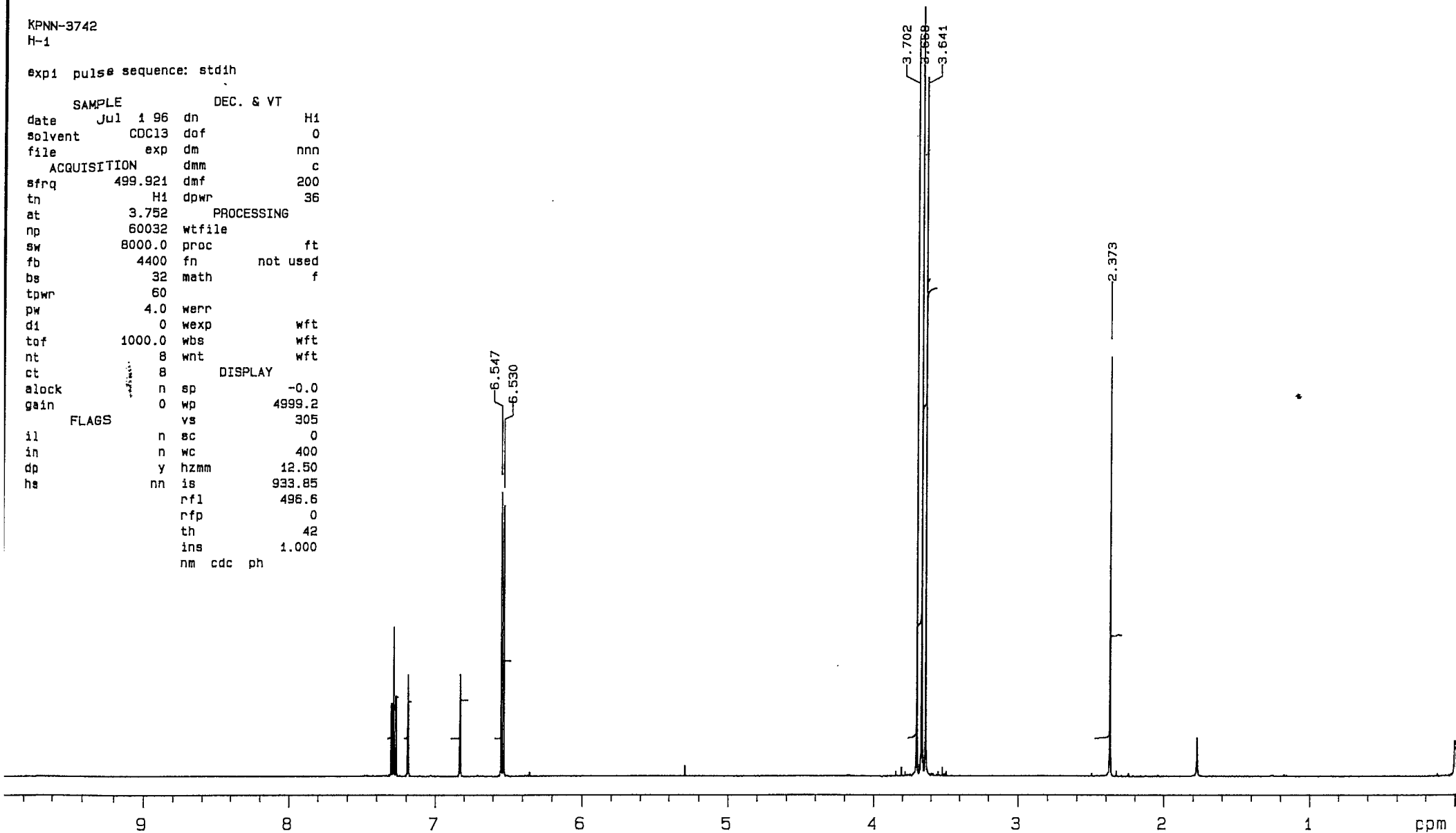
Varian Japan Ltd. (V)

KPNN-3742

H-1

exp1 pulse sequence: stdih

SAMPLE		DEC. & VT	
date	Jul 1 96	dn	H1
solvent	CDC13	dof	0
file	exp	dm	nnn
ACQUISITION			
sfrq	499.921	dmm	c
tn	H1	dmf	200
at	3.752	dpwr	36
PROCESSING			
np	60032	wtfile	
sw	8000.0	proc	ft
fb	4400	fn	not used
bs	32	math	f
tpwr	60		
pw	4.0	werr	
d1	0	wexp	wft
tof	1000.0	wbs	wft
nt	8	wnt	wft
ct	8	DISPLAY	
alock	n	sp	-0.0
gain	0	wp	4999.2
FLAGS			
il	n	sc	0
in	n	wc	400
dp	y	hzmm	12.50
hs	nn	is	933.85
		rfl	496.6
		rfp	0
		th	42
		ins	1.000
		nm	cdc ph



OBSERVE Nucleus <u>H1</u> Freq. <u>499.9214</u> MHz Spec. Width <u>8000.0</u> Hz Offset <u>1000.0</u> Hz Acq. Time <u>3.752</u> sec Delay <u>0</u> sec Pulse Width <u>4.0</u> μ sec Transients <u>8</u>		DECOUPLE Nucleus <u>H1</u> Offset <u>0</u> Hz Mode <u>nnn</u> Power <u>not used</u> db Modulation: Mode <u>C</u> Freq. <u>200</u> Hz Pulse Width <u> </u> μ sec Power Mode <u>36</u>		PLOT/PROCESSING FN <u>not used</u> K RE <u> </u> sec CD <u> </u> sec LB <u>not used</u> Hz PL <u>not used</u> sec CCD <u> </u> Width <u>4999.2</u> Hz/ppm Start <u>-0.0</u> Hz/ppm Reference <u> </u>		EXPERIMENT Pulse Sequence <u>s2pul</u> Tube O.D. <u> </u> mm Temp. <u>not used</u> °C Solvent <u>CDC13</u>		SAMPLE KPNN-3742 H-1 <chem>COc1cc(C)cc(C(=O)OC)c1C(=O)OC</chem>		Number <u> </u> File <u>exp</u> Date <u>Jul 1 96</u> XL <u>500</u>	
--	--	--	--	--	--	--	--	--	--	--	--

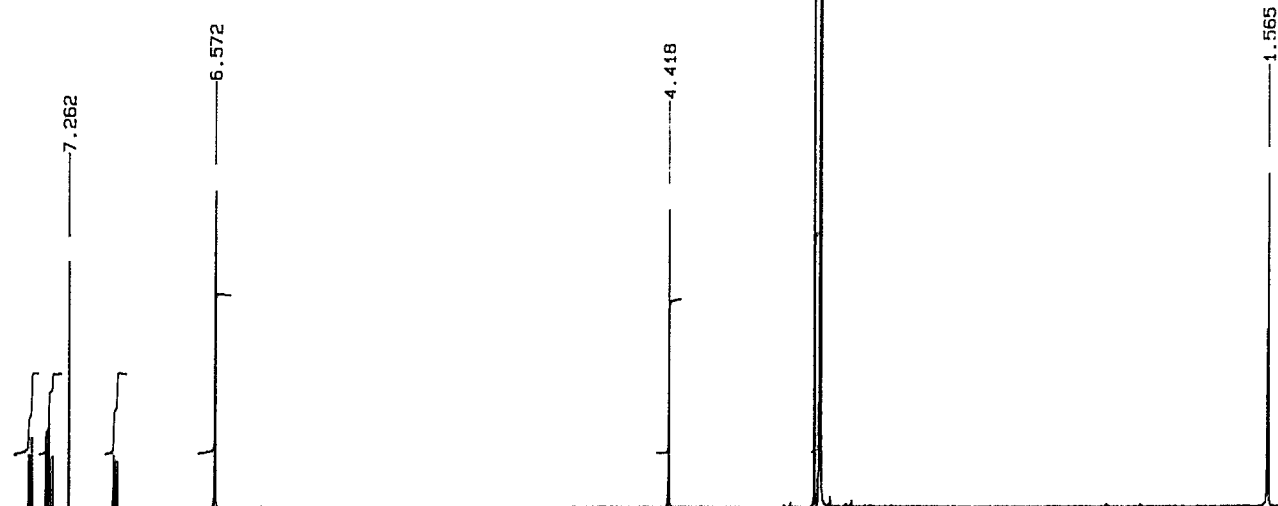
25
NO 997280 J

Varian Japan Ltd.

KPNM-3746
H-1

exp5 pulse sequence: std1h

SAMPLE DEC. & VT
date Jul 15 96 dn H1
solvent CDCl3 dof 0
file exp dm nnn
ACQUISITION dmm c
sfrq 499.921 dmf 200
tn H1 dpwr 36
at 3.752 PROCESSING
np 60032 wtfile
sw 8000.0 proc ft
fb 4400 fn not used
bs 32 math f
tpwr 60
pw 4.0 werr
di 0 wexp wft
tof 1000.0 wbs wft
nt 8 wnt wft
ct 8 DISPLAY
alock n sp -0.0
gain 14 wp 4999.2
FLABS vs 374
il n sc 0
in n wc 400
dp y hzmm 12.50
hs nn is 11825.19
rfl 503.0
rfp 0
th 42
ins 1.000
nm cdc ph



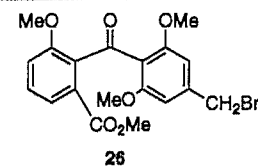
OBSERVE
Nucleus H1 Freq 499.9214 MHz
Spec. Width 8000.0 Hz Offset 1000.0 Hz
Acq. Time 3.752 sec Delay 0 sec
Pulse Width 4.0 µsec Transients 8

DECOUPLE
Nucleus H1 Offset 0 Hz
Mode nnn Power not used db
Modulation Mode c Freq 200 Hz
Pulse Width µsec Power Mode 36

PLOT/PROCESSING
Reference not used K RE sec CD sec
LB not used H2 A used sec CCD
Width 4999.2 Hz/ppm Start -0.0 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence std1
Tube O.D. mm
Temp not used °C
Solvent CDCl3

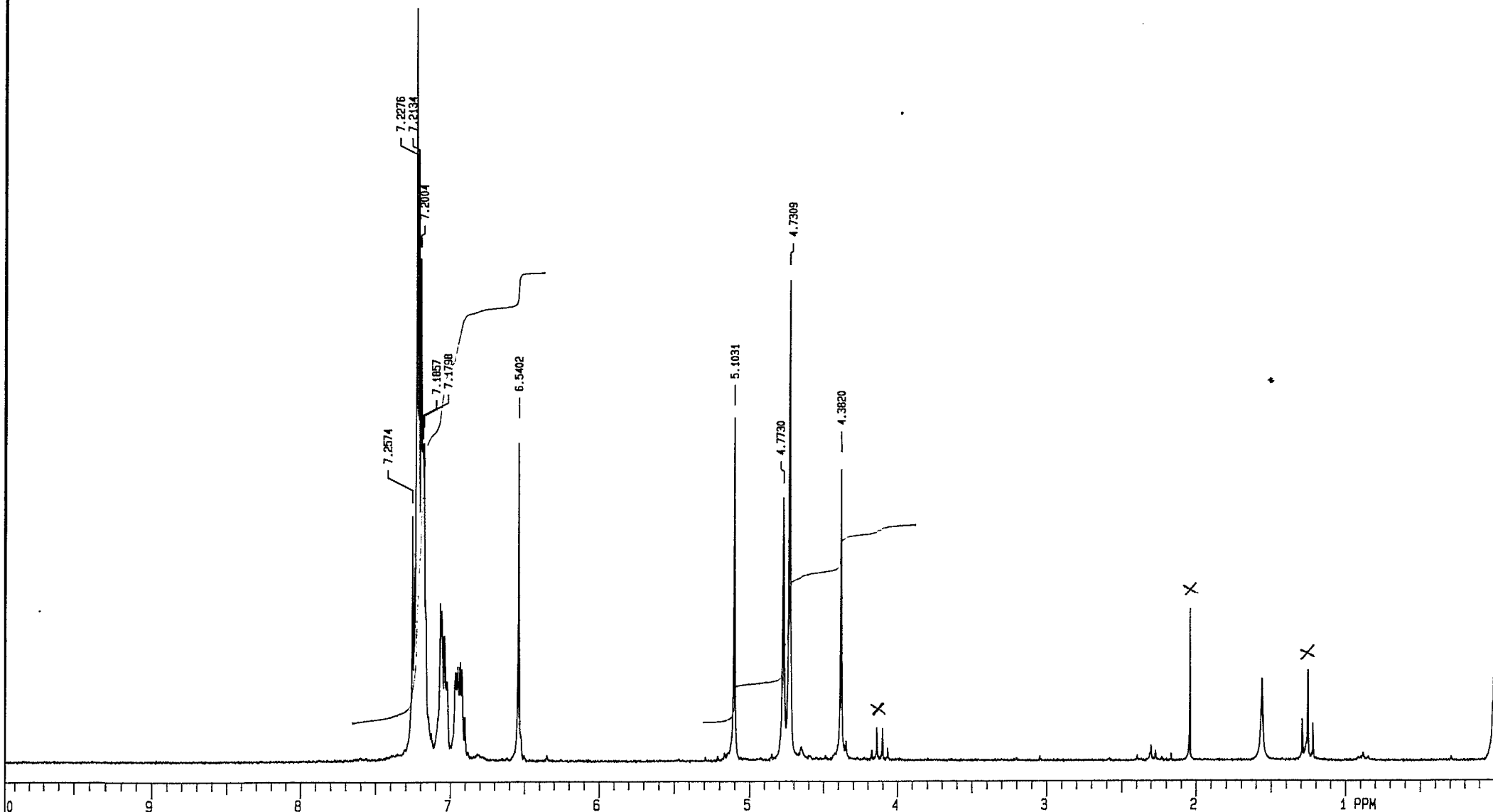
SAMPLE
KPNM-3746
H-1



Number
File exp
Date Jul 15 96
XL 500

NO 997260 J

Varian Japan Ltd. (V)



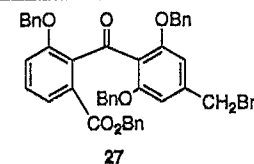
OBSERVE
 Nucleus 1.000 Freq. 200 MHz
 Spec. Width 3000.3 Hz Offset 0 Hz
 Acq. Time 2.666 sec Delay 0 sec
 Pulse Width 11.5 µsec Transients 16

DECOUPLE
 Nucleus 1.000 Offset -300.0 Hz
 Mode NNN Power 1750.0 db
 Modulation: CCC Freq. 200 Hz
 Pulse Width µsec Power Mode 1.0

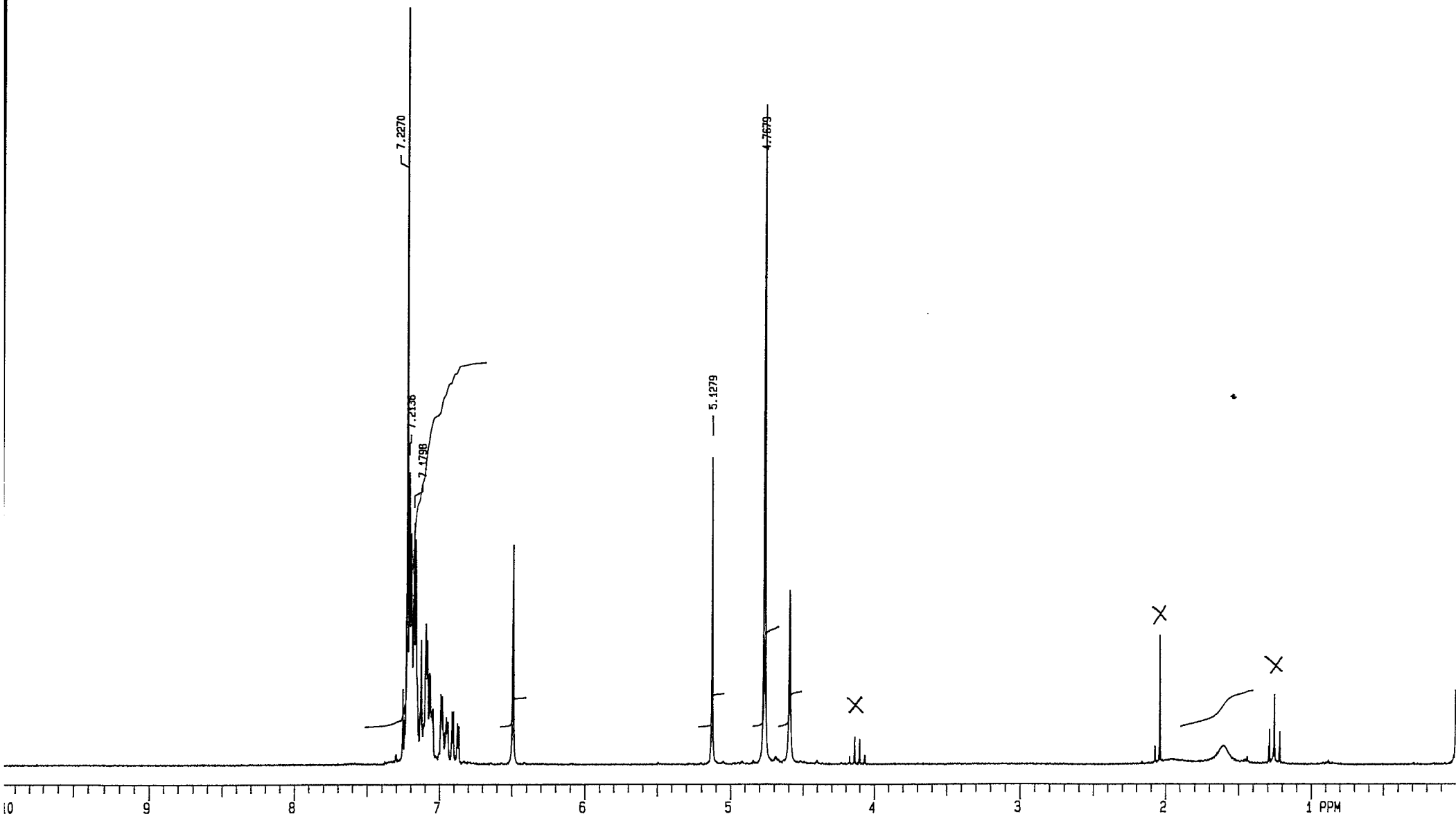
PLOT/PROCESSING
 FN 16 K RE sec CD sec
 LR Hz AF sec CCD
 Width 2000.0 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence S2PUL
 Tube O.D. mm
 Temp. °C
 Solvent CDCL3

SAMPLE
 LRS



Number 35
 File H
 Date 12-09-96
 15:47:18
 XL GEM 200



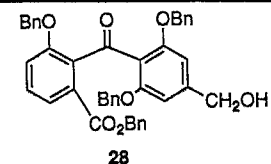
OBSERVE
 Nucleus 1.000 Freq. 200 MHz
 Spec. Width 3000.3 Hz
 Acq. Time 2.566 sec
 Pulse Width 11.5 µsec
 Offset 0 Hz
 Delay 0 sec
 Transients 16

DECOUPLE
 Nucleus 1.000 Offset -300.0 Hz
 Mode NNN Power 1750.0 db
 Modulation: Mode CCC Freq. 200 Hz
 Pulse Width µsec Power Mode 1.0

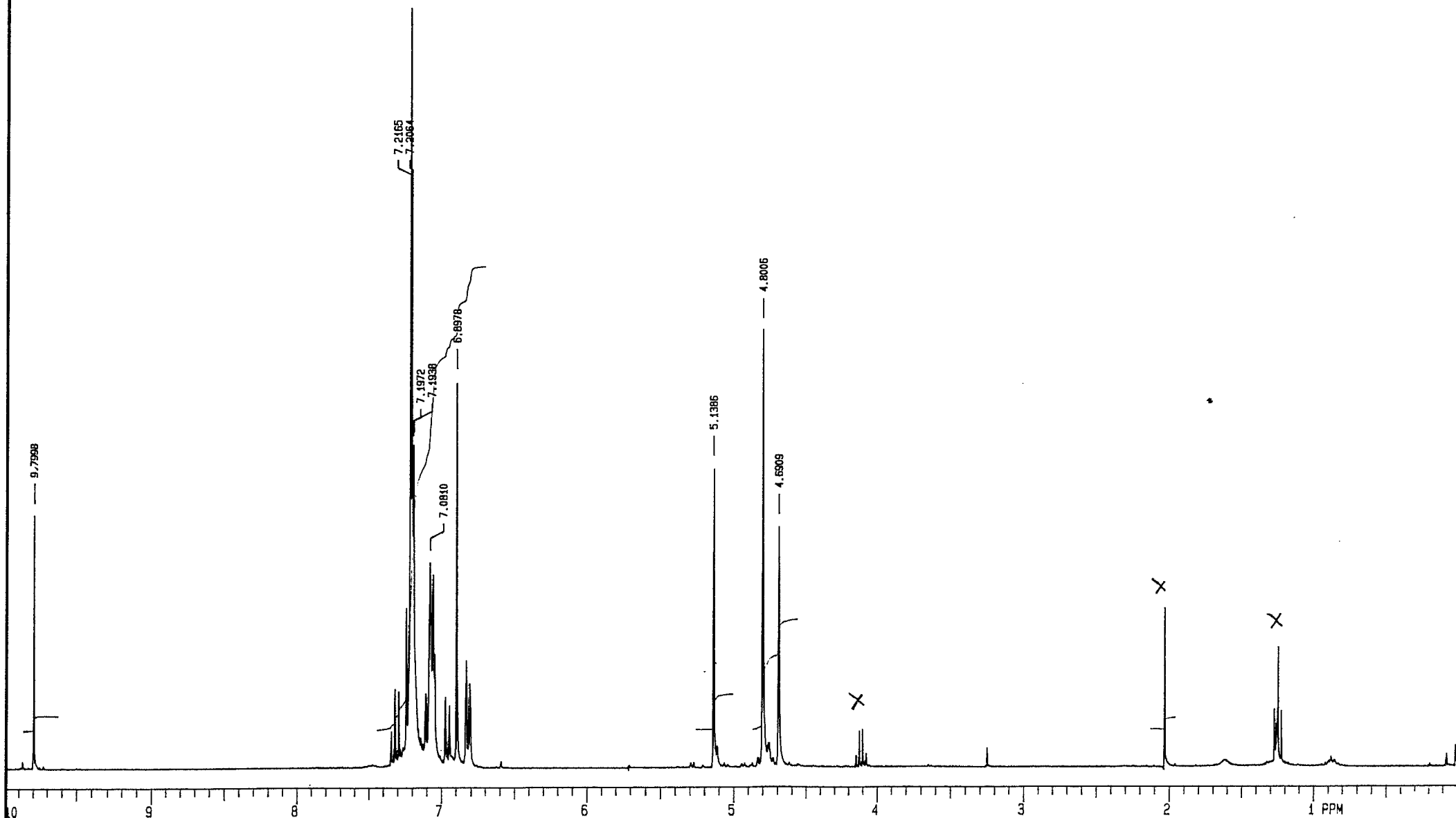
PLOT/PROCESSING
 F1 15 K RF sec CD sec
 LB Hz AF sec CCD
 Width 2000.0 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence S2PUL
 Tube O.D. mm
 Temp °C
 Solvent CDCl3

SAMPLE
 PURE ALC OHO



Number 79
 File H
 Date 08-09-96
 14:15:31
 XL GEM 200



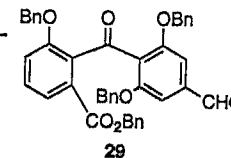
OBSERVE
 Nucleus 1.000 Freq 300 MHz
 Spec. Width 500.5 Hz Offset 0 Hz
 Acq. Time 3.335 sec Delay 1.670 sec
 Pulse Width 9.4 μsec Transients 16

DECOUPLE
 Nucleus 1.000 Offset -450.0 Hz
 Mode NNN Power 1700.0 db
 Modulation Mode C Freq 200 Hz
 Pulse Width μsec Power Mode 1.0

PLOT/PROCESSING
 FN 32 K RE sec CD sec
 LB Hz AF sec CCD
 Width 3000.9 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence S2PUL
 Tube Q.D. mm
 Temp °C
 Solvent CDCl3

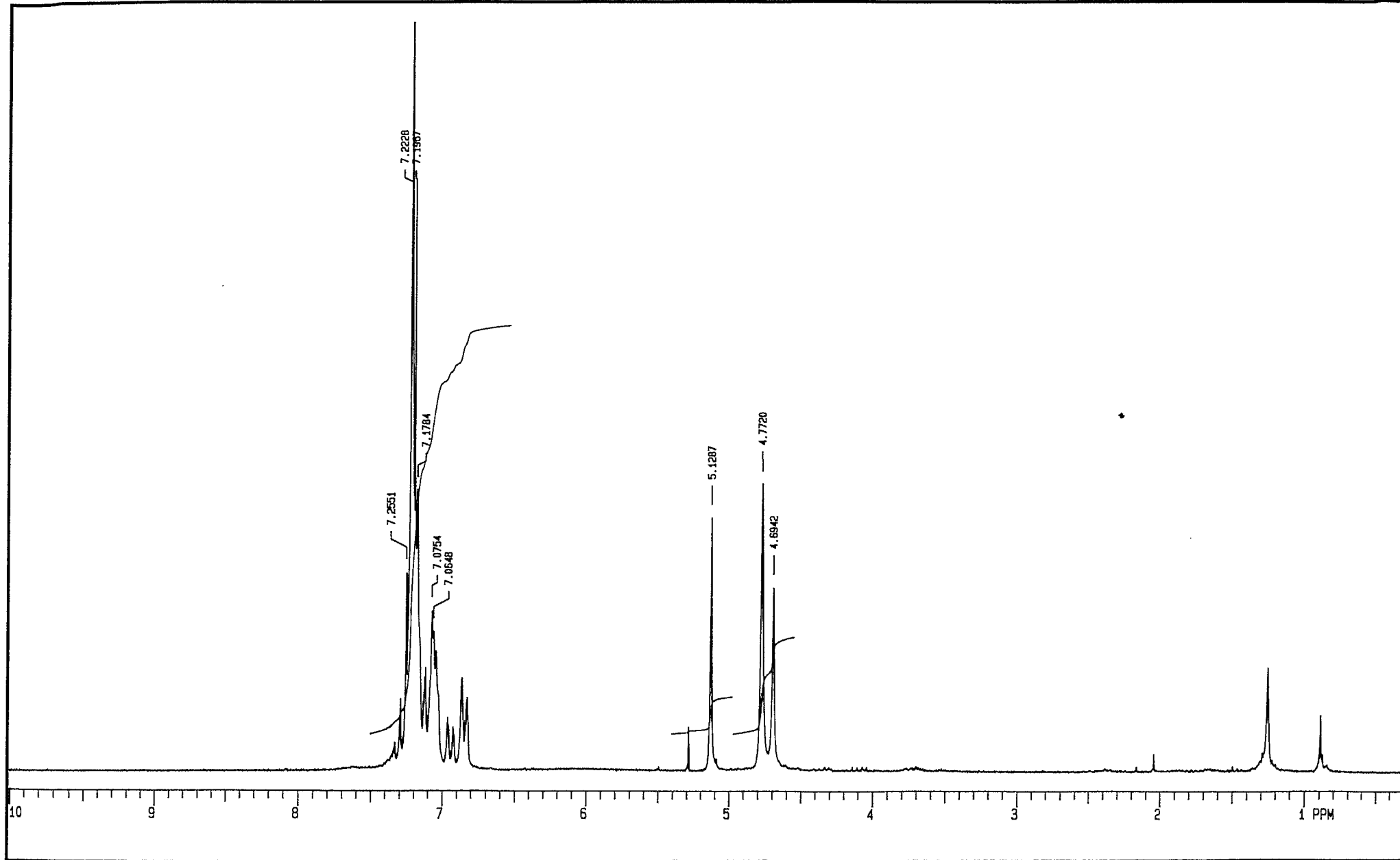
SAMPLE
 TPAP NMO



Number 71
 File H
 Date 12-19-95
 11:04:56
 XL 6EM 300

NO 997260 J

Varian Japan Ltd. (W)



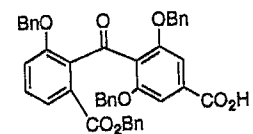
OBSERVE
 Nucleus 1.000 Freq 200 MHz
 Spec. Width 3000.3 Hz
 Acq. Time 2.666 sec
 Pulse Width 11.5 μ sec
 Offset 0 Hz
 Delay 0 sec
 Transients 16

DECOUPLE
 Nucleus 1.000 Offset -300.0 Hz
 Mode NNN Power 1750.0 db
 Modulation Mode CCC Freq 200 Hz
 Pulse Width μ sec Power Mode 1.0

PLOT/PROCESSING
 FN 16 K RE sec CD sec
 LB Hz AF sec CCD
 Width 2000.0 Hz/ppm Start 0 Hz/ppm
 Reference

EXPERIMENT
 Pulse Sequence SPUL
 Tube O.D. mm
 Temp. °C
 Solvent CDCl3

SAMPLE
 COOH PURE

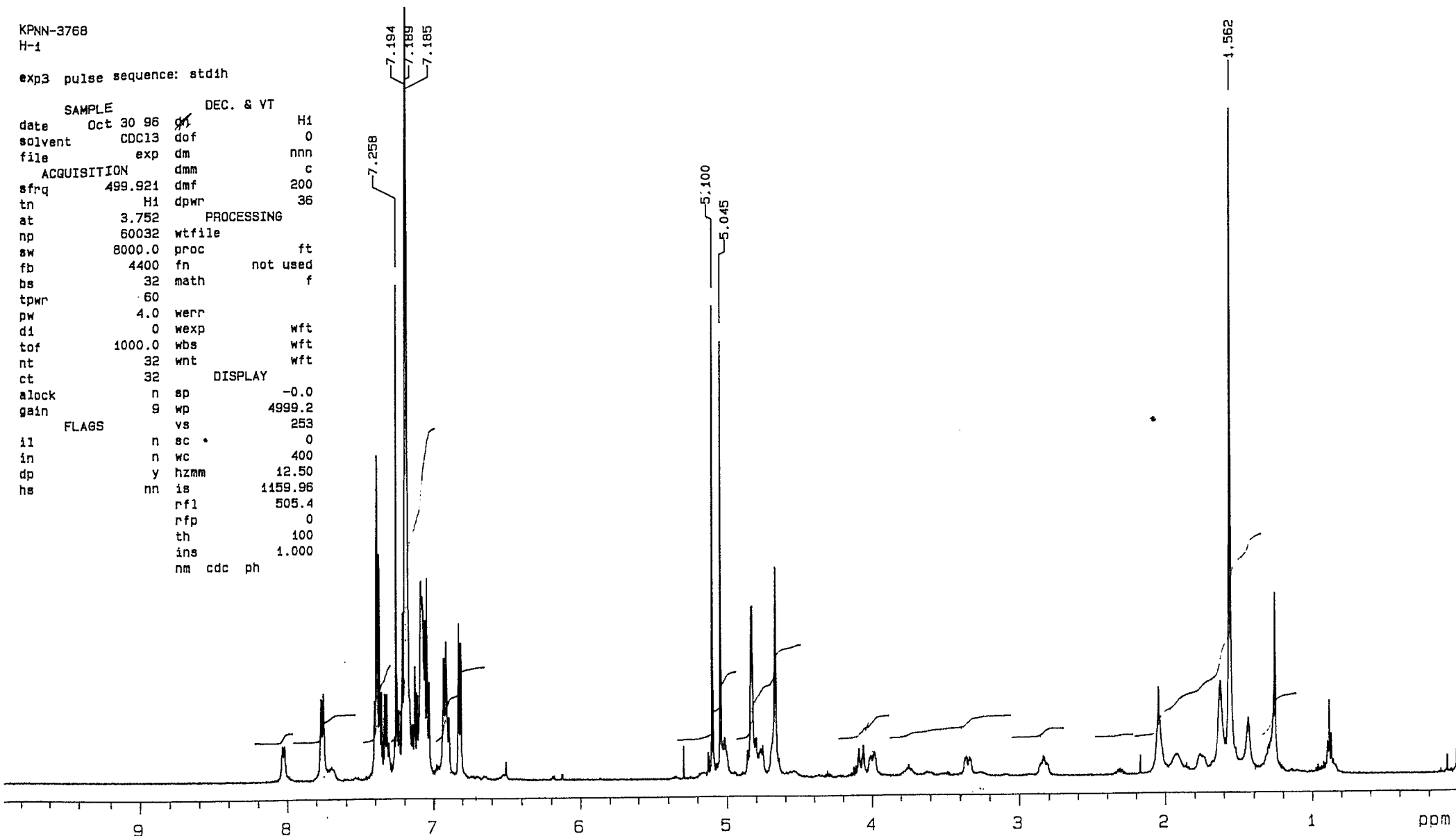


Number
 File H
 Date 10-24-96
 12:38:21
 XL 96M 200

KPNN-3768
H-1

exp3 pulse sequence: stdih

SAMPLE		DEC. & VT	
date	Oct 30 96	gt	H1
solvent	CDCl3	dof	0
file	exp	dm	nnn
ACQUISITION		dmm	c
sfrq	499.921	dmf	200
tn	H1	dpwr	36
at	3.752	PROCESSING	
np	60032	wtfile	
sw	8000.0	proc	ft
fb	4400	fn	not used
bs	32	math	f
tpwr	60		
pw	4.0	werr	
d1	0	wexp	wft
tof	1000.0	wbs	wft
nt	32	wnt	wft
ct	32	DISPLAY	
alock	n	sp	-0.0
gain	9	wp	4999.2
FLAGS		vs	253
il	n	sc	0
in	n	wc	400
dp	y	hzmm	12.50
hs	nn	is	1159.96
		rfl	505.4
		rpf	0
		th	100
		ins	1.000
		nm	cdc ph



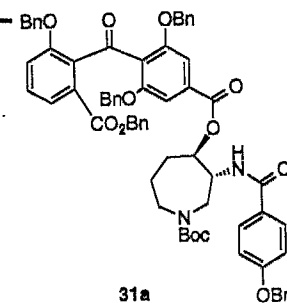
OBSERVE
Nucleus H1 Freq. 499.9214 MHz
Spec. Width 8000.0 Hz Offset 1000.0 Hz
Acq. Time 3.752 sec Delay 0 sec
Pulse Width 4.0 μ sec Transients 32

DECOUPLE
Nucleus H1 Offset 0 Hz
Mode nnn Power not used db
Modulation: Mode G Freq. 200 Hz
Pulse Width μ sec Power Mode 36

PLOT/PROCESSING
Pulse Sequence 2pul
Tube O.D. mm
Temp. not used °C
Solvent CDCl3

EXPERIMENT
Pulse Sequence 2pul
Tube O.D. mm
Temp. not used °C
Solvent CDCl3

SAMPLE
KPNN-3768
H-1



31a

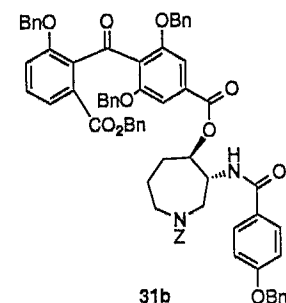
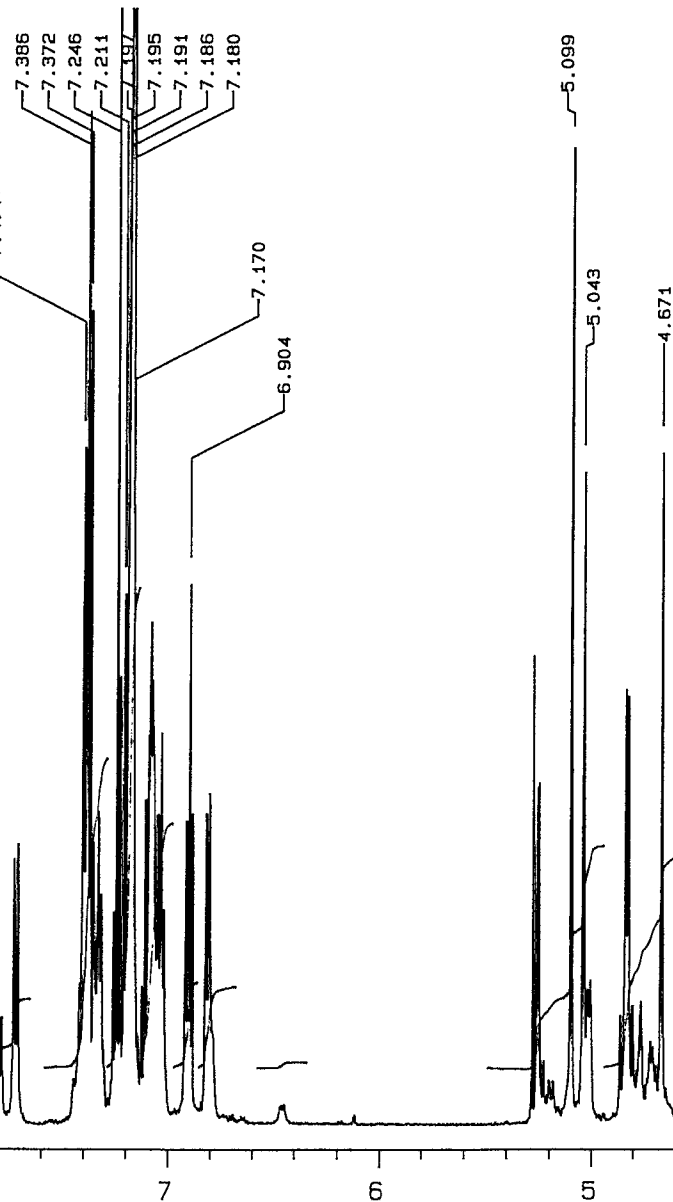
Number
File exp
Date Oct 30 96
XL 500

Japan Ltd. (V)

KPNN-3794
H-1

exp5 pulse sequence: std1h

SAMPLE DEC. & VT
date Feb 18 97 dn H1
solvent CDCl3 dof
file exp dm nm
ACQUISITION dmm
sfrq 499.921 dmf 200
tn H1 dpwr 36
at 3.752 PROCESSING
np 60032 wtfile
sw 8000.0 proc ft
fb 4400 fn not used
bs 32 math f
tpwr 60
pw 4.0 werr
d1 0 wexp wft
tof 1000.0 wbs wft
nt 16 wnt wft
ct 16
DISPLAY
alock n sp -0.0
gain not used wp 4999.2
FLAGS vs 464
il n sc 0
in n wc 400
dp y hzmm 12.50
hs nn is 1721.87
rf1 510.8
rfp 0
th 93
ins 1.000
nm cdc ph

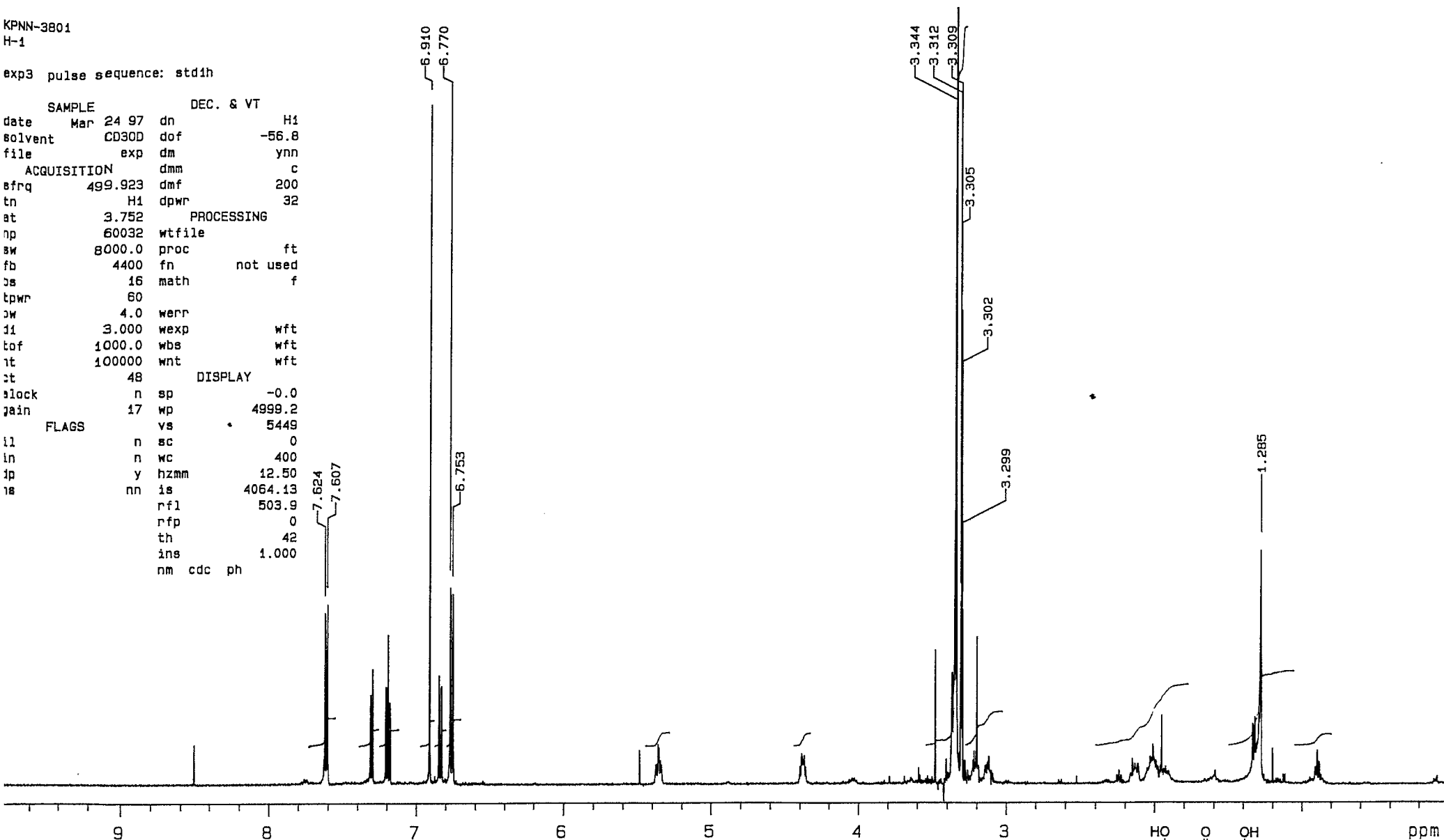


San Ltd. (M)

KPNN-3801
H-1

exp3 pulse sequence: std1h

SAMPLE		DEC. & VT	
date	Mar 24 97	dn	H1
solvent	CD3OD	dof	-56.8
file	exp	dm	ynn
ACQUISITION		dmm	c
sfrq	499.923	dmf	200
tn	H1	dpwr	32
at	3.752	PROCESSING	
np	60032	wtfile	
sw	8000.0	proc	ft
fb	4400	fn	not used
bs	16	math	f
tpwr	60		
pw	4.0	werr	
ti	3.000	wexp	wft
tof	1000.0	wbs	wft
it	100000	wnt	wft
st	48	DISPLAY	
clock	n	sp	-0.0
gain	17	wp	4999.2
FLAGS		vs	5449
il	n	sc	0
in	n	wc	400
ip	y	hzmm	12.50
is	nn	is	4064.13
		rfl	503.9
		rfp	0
		th	42
		ins	1.000
		nm	cdc ph



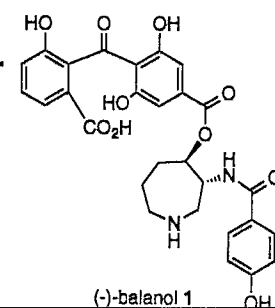
Nucleus H1 Freq. 499.9234 MHz
Spec. Width 8000.0 Hz Offset 1000.0 Hz
Acq. Time 3.752 sec Delay 3.000 sec
Pulse Width 4.0 μ sec Transients 48

DECOUPLE
Nucleus H1 Offset -56.8 Hz
Mode YNN Power not used db
Modulation Mode C Freq. 200 Hz
Pulse Width μ sec Power Mode 32

PLOT/PROCESSING
P~~not used~~ K RE sec CD sec
L~~not used~~ H~~not used~~ Q~~not used~~ sec CCD
Width 4999.2 Hz/ppm Start -0.0 Hz/ppm
Reference

EXPERIMENT
Pulse Sequence 20u1
Tube O.D. mm
Temp. not used °C
Solvent CD3OD

SAMPLE
KPNN-3801
H-1



(-)-balanol 1

Number
File exp
Date Mar 24 97
XL 500

Japan Ltd. (J)