

Online Collection of NMR Spectra Catalogs

Volume I

EM360/A/L 60 MHz NMR Spectra Set

Varian Associates

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Editor's Preface to the Online Collection of NMR Spectra Catalogs

In my opinion old spectra catalogs are not at all useless. It is true that the instruments and the techniques of yesterday may be quite obsolete today and that present performance specifications are usually much better than what they were years ago. This preempts the commercial usefulness of old spectra catalogs to the Companies who originally put them together. But there is always so much to learn when browsing through old experimental data! They are ideal for illustrating the basics of the various techniques from a historic perspectives, underlining on the way whatever progress has been achieved, and pointing out any problems which might still be with us. A researcher who does not understand historic data and long discarded techniques probably does not really comprehend modern techniques as well. In addition, Science spirals ahead through loops - there is always a going back and a recycling of old ideas in new contexts, so one never knows what data will become useful as a reference or even as a kind of quality check.

Through the years I have collected great many NMR spectra catalogs, all of which were originally used by major manufacturers as promotional handouts. Consequently, I should not need their consent for putting the catalogs online but, in doubt, I have decided to seek it anyway and met a universal understanding and approval.

The quality of the PDF reproductions of the ageing hardcopy catalogs is not always the best but it should be still quite sufficient to fulfill the educational and historic purposes (any help in this respect would be most welcome).

Editor's Preface to the Varian *EM360/A/L 60 MHz NMR Spectra Set*

This 1978 Varian spectra catalog was among the first ones of its kind and probably the first one collected explicitly for an educational purpose: teaching chemists the interpretation of NMR spectra of organic compounds. It therefore does not dwell much on illustrating the performance of the instrument itself (except for a few last pages). Readers are warmly welcome to test their present-day abilities by tackling the many eminently practical exercises presented here. Of course, some of you might become puzzled by the more "advanced" techniques shown towards the end, such as *tickling* and *INDOR*. Finding the logical relationships between these specialties and some of the modern 2D experiments is an additional challenge you might wish to face.

Naturally, you will need to mentally discard the "wiggles" in the peaks; an artifact we were so used to at that time that we barely noticed it. Apart from that, it is NMR as usual. The EM360 was a CW (continuous wave) machine based on a 60 MHz permanent magnet. It was enormously successful – the series was in production for over 15 years without any substantial modification. Hundreds of them were sold and untold numbers of NMR started their carriers doing nights at their modest consoles void of any computer.

Stanislav Sykora, Castano Primo, November 9, 2009

EM-360/A/I

60MHz NMR Spectra Set

Spectra 1-18 : These EM-360 spectra are used as interpretation exercises in our basic NMR courses. They demonstrate routine 60MHz NMR applications for structure elucidation and quantitative analysis. The use of basic spectrometer features (spectrum expansion, integration) is shown (e.g. spectrum 7). The use of the EM-360 Lock Decoupler for double resonance experiments is demonstrated with example 10, 11 and 12. (Solutions to exercises are given on a separate sheet).

Spectrum 19 : This spin tickling experiment demonstrates the extraordinary field stability of the 14Kgauss permanent magnet that is heart of all EM-360 systems. These critical experiments were performed without field-frequency lock, simply by using the EM-360 Lock Decoupler in the "decouple" mode and by very carefully adjusting the H₂ power for a low level.

Spectrum 20 : This example of a quantitative mixture analysis evidences the superior sensitivity of the EM-360L: To a sample containing unknown amounts of the glucocorticoid hormones Prednisolone and Cortisone, 4.6mg fumaric acid were added as a standard. The integral ratio, normalized to one hydrogen per molecule, is found to be 20:28:26 (Prednisolone:fumaric acid:Cortisone). The sample thus contains 10.2mg Prednisolone and 13.3mg Cortisone.

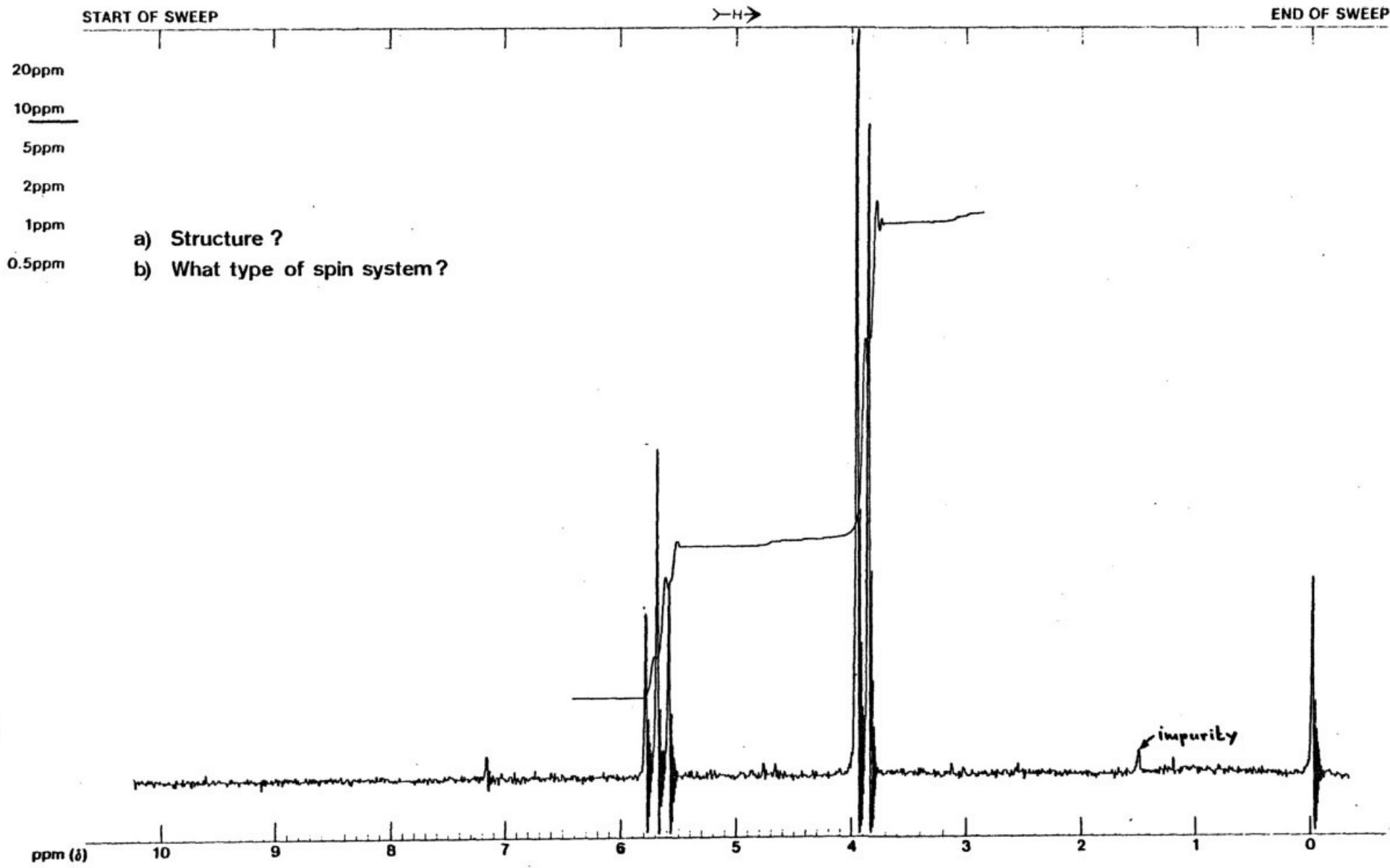
Spectrum 21 : The three fairly closely coupled aromatic ring protons of methoxy-p-anisidine provide a nice illustration for the EM-360L's decoupling capabilities. Signals only 0.4ppm apart are easily decoupled, and with the INDOF technique observation 0.2ppm from the monitoring peak is possible. The good signal/noise ratio in the INDOF trace is proof for the excellent overall system stability: every instability in field, frequency, or amplitude translates into baseline noise in the INDOF spectrum.

Spectrum 22 : This ¹⁹F NMR spectrum of p-fluoroacetophenone illustrates the EM-360L's other nuclei capabilities. Both resolution and sensitivity are on the same high level as the proton performance of this instrument. This 19F spectrum represents the X part of a AA'BB'X spin system.



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varian anaspect
palo alto, california



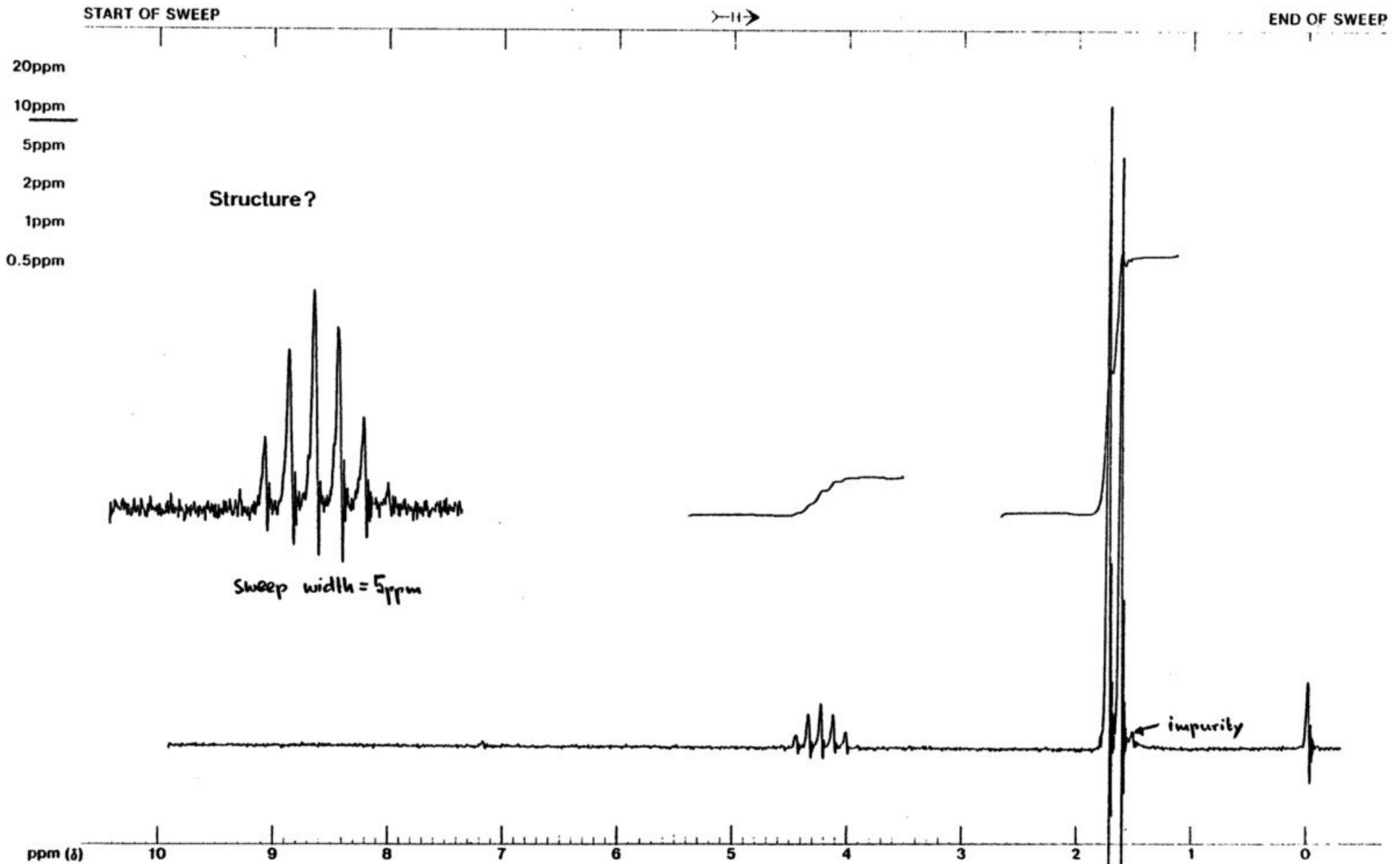
a) Structure ?
b) What type of spin system ?

SPECTRUM AMPL. 600 SWEEP TIME 5 min SAMPLE: $C_2H_3Cl_3$ REMARKS: OPERATOR C.F.
FILTER 0.05 sec SWEEP WIDTH 10 ppm or Hz DATE: _____
RF POWER 0.05 mG END OF SWEEP 0 ppm or Hz SOLVENT: $CDCl_3$ SPECTRUM NO. 1



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SPECTRUM AMPL. 100

SWEEP TIME 5 min

SAMPLE: C_3H_7Br

REMARKS:

OPERATOR T.W.

FILTER 0.02 sec

SWEEP WIDTH 10 ppm or Hz

DATE

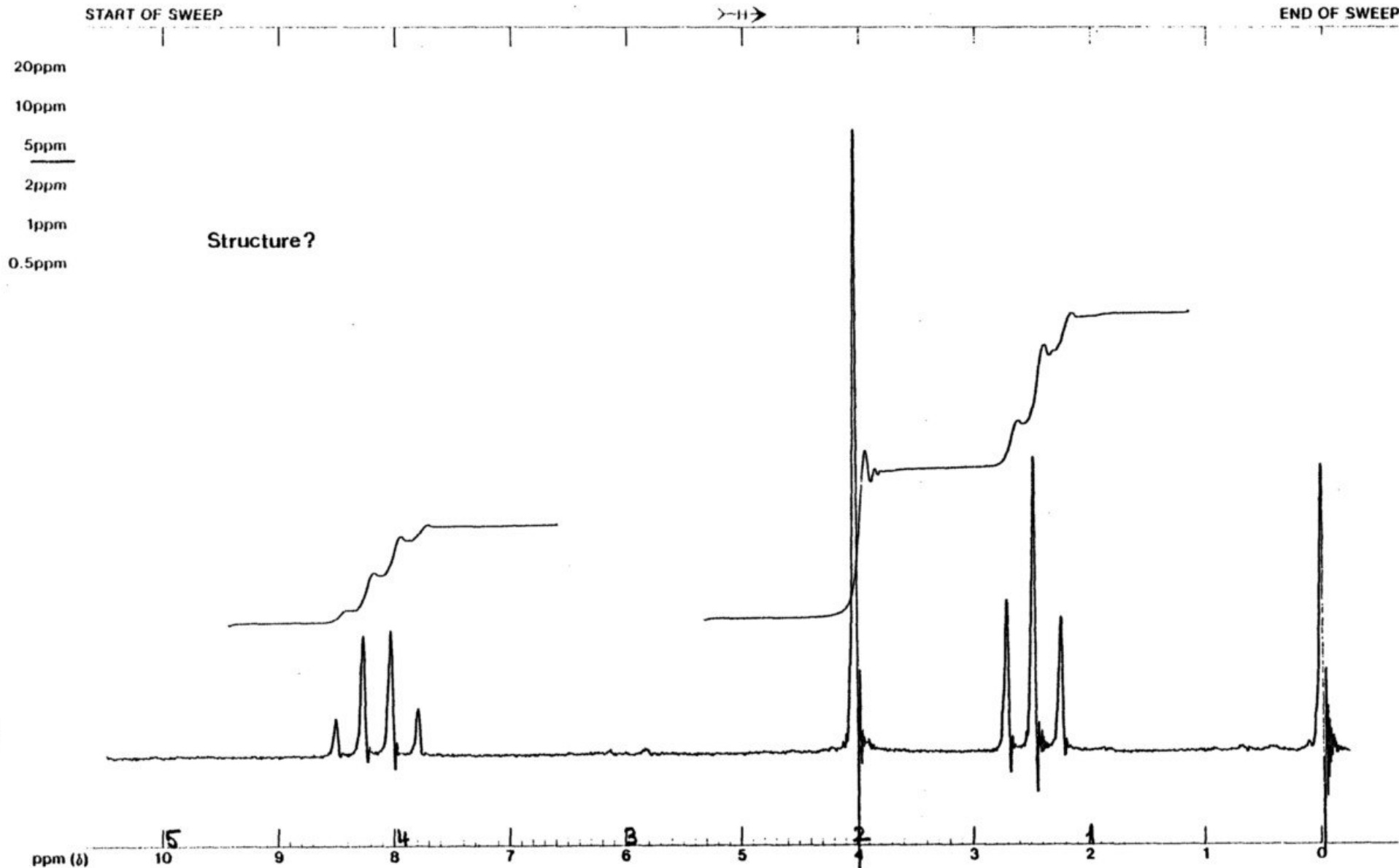
RF POWER 0.05 mG

END OF SWEEP 0 ppm or Hz

SOLVENT: $CDCl_3$

SPECTRUM NO. **2**

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SPECTRUM AMPL. 100

SWEEP TIME 5 min

SAMPLE: $C_4H_8O_2$

REMARKS:

OPERATOR T.W.

FILTER 0.02 sec

SWEEP WIDTH 5 ppm or Hz

DATE

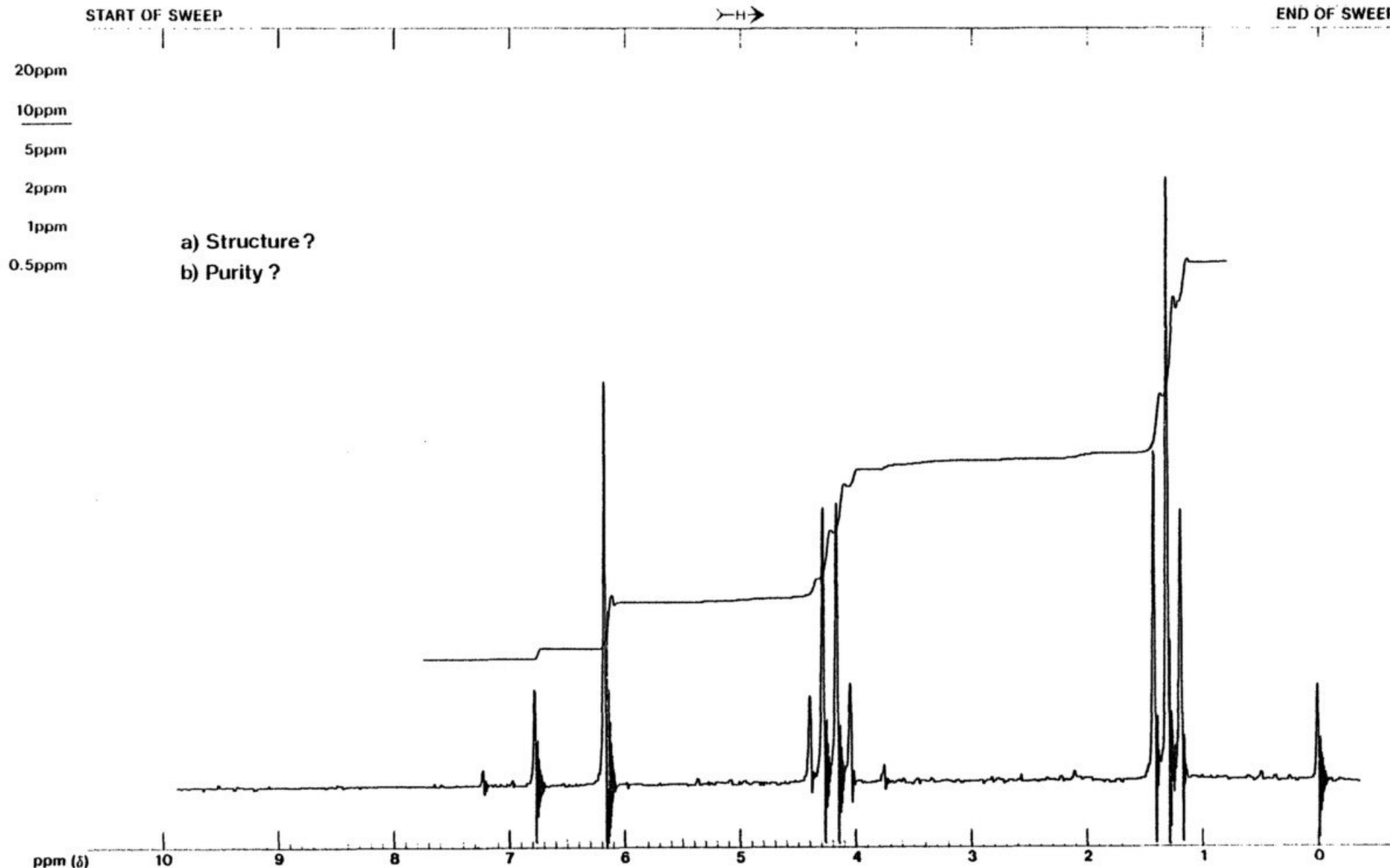
RF POWER 0.04 mG

END OF SWEEP 0 ppm or Hz

SOLVENT: $CDCl_3$

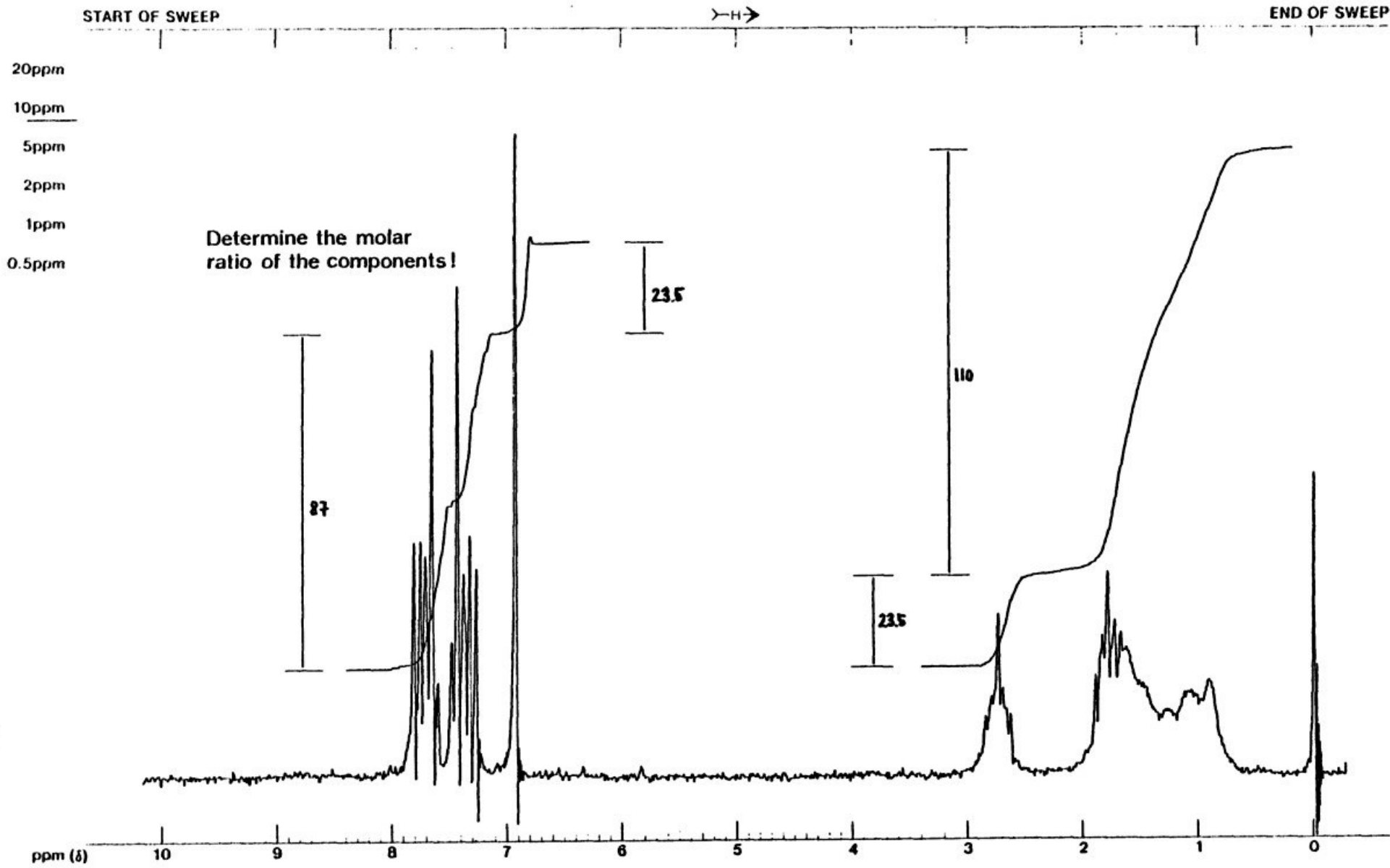
SPECTRUM NO. 3

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a) Structure ?
b) Purity ?

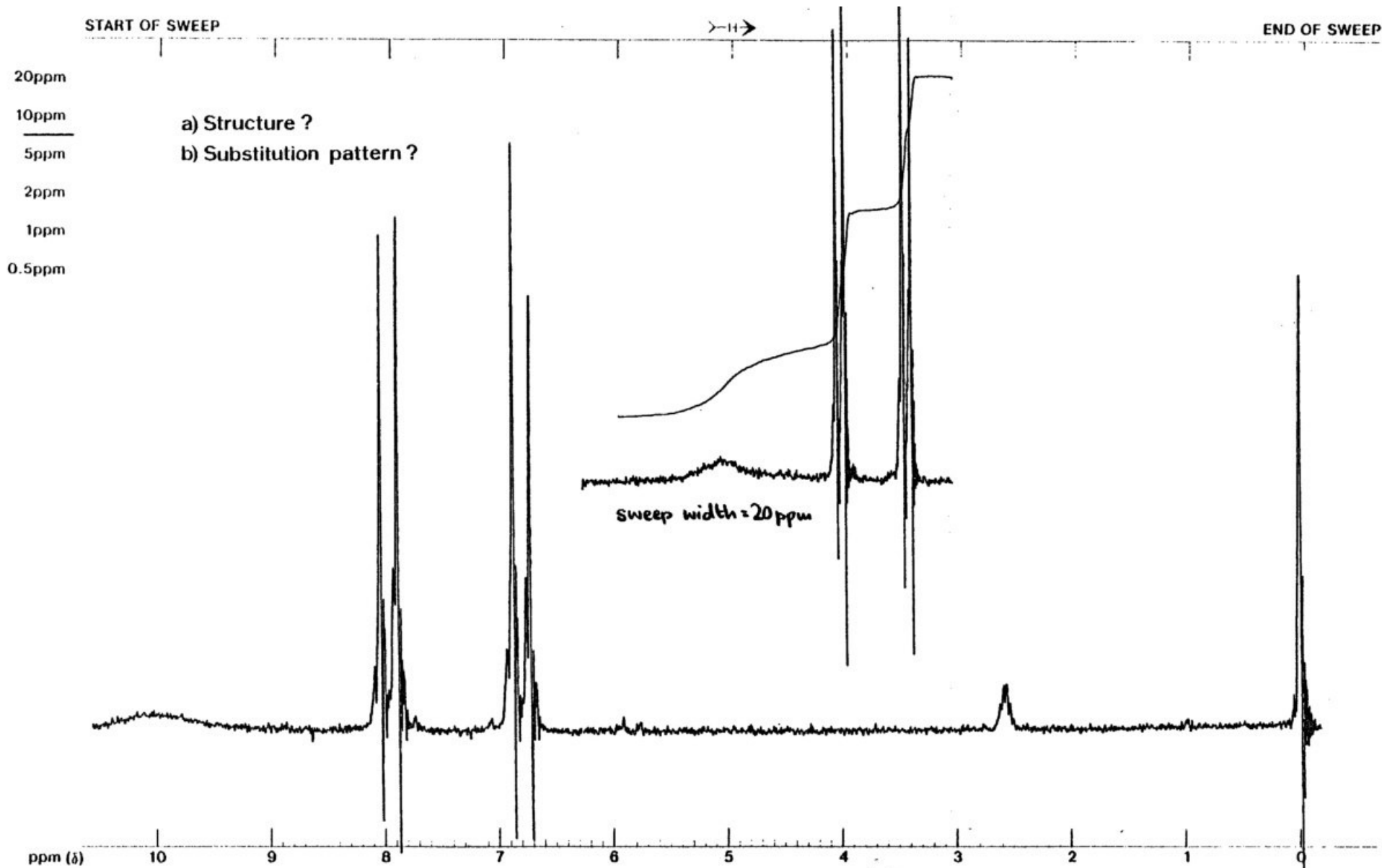
SPECTRUM AMPL. 250 SWEEP TIME 5 min SAMPLE: C₈H₁₂O₄ REMARKS: OPERATOR C.F.
FILTER 0.05 sec SWEEP WIDTH 10 ppm or Hz DATE
RF POWER 0.05 mG END OF SWEEP 0 ppm or Hz SOLVENT: CDCl₃ SPECTRUM NO. 4



Determine the molar ratio of the components!

SPECTRUM AMPL. <u>300</u>	SWEEP TIME <u>5</u> min	SAMPLE: Mixture of Naphthalene Tetralin Decalin	REMARKS:	OPERATOR <u>C.F.</u>
FILTER <u>0.05</u> sec	SWEEP WIDTH <u>10</u> ppm or Hz	SOLVENT: <u>CDCl₃</u>		DATE _____
RF POWER <u>0.05</u> mG	END OF SWEEP <u>0</u> ppm or Hz			SPECTRUM NO. <u>5</u>

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SPECTRUM AMPL. 400

SWEEP TIME 5 min

SAMPLE: $C_6H_5NO_3$

REMARKS:

OPERATOR T.W.

FILTER 0.02 sec

SWEEP WIDTH 10/20 ppm or Hz

DATE

RF POWER 0.1 mG

END OF SWEEP 0 ppm or Hz

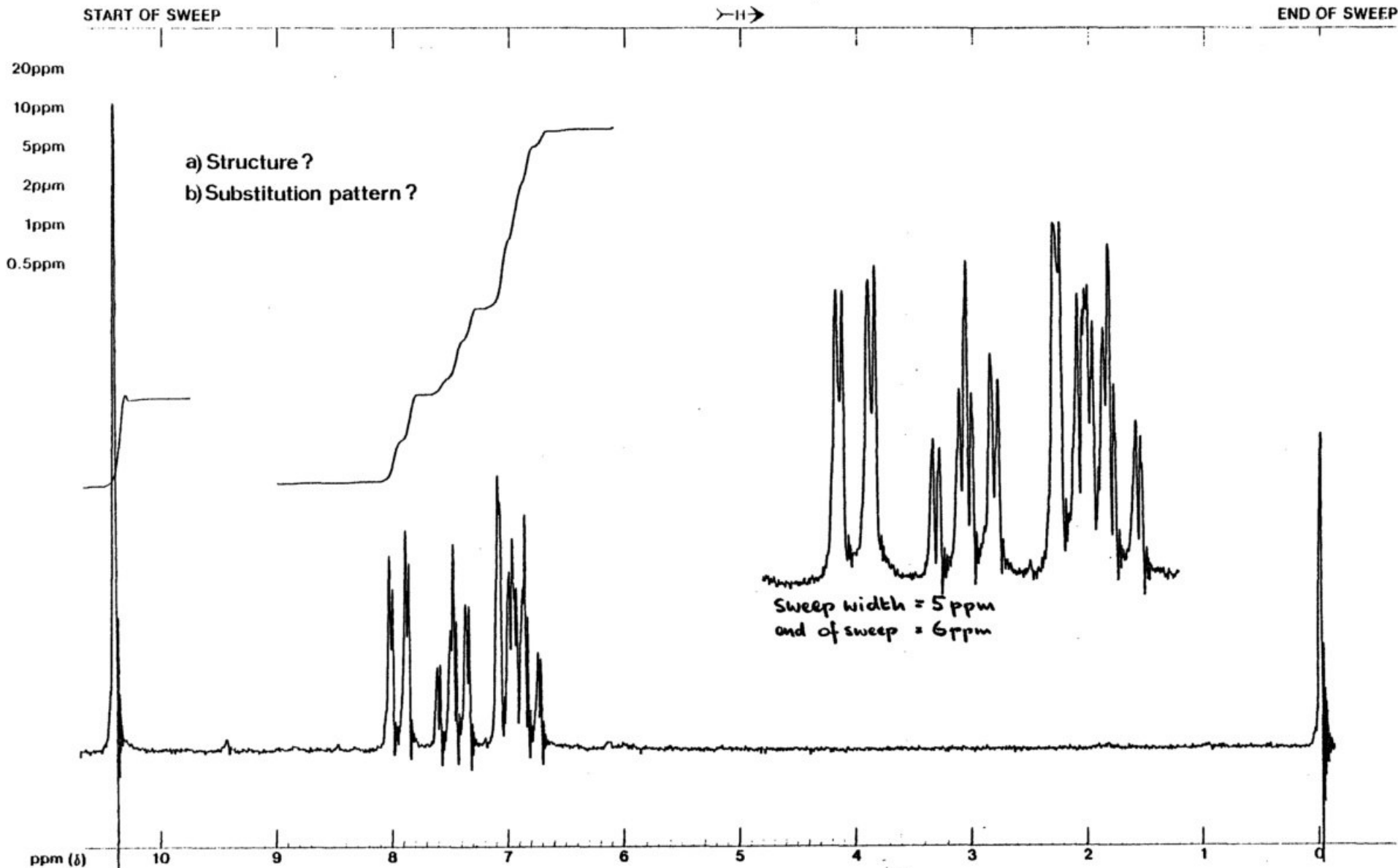
SOLVENT: $CDCl_3 / DMSO-d_6$

SPECTRUM NO.

6

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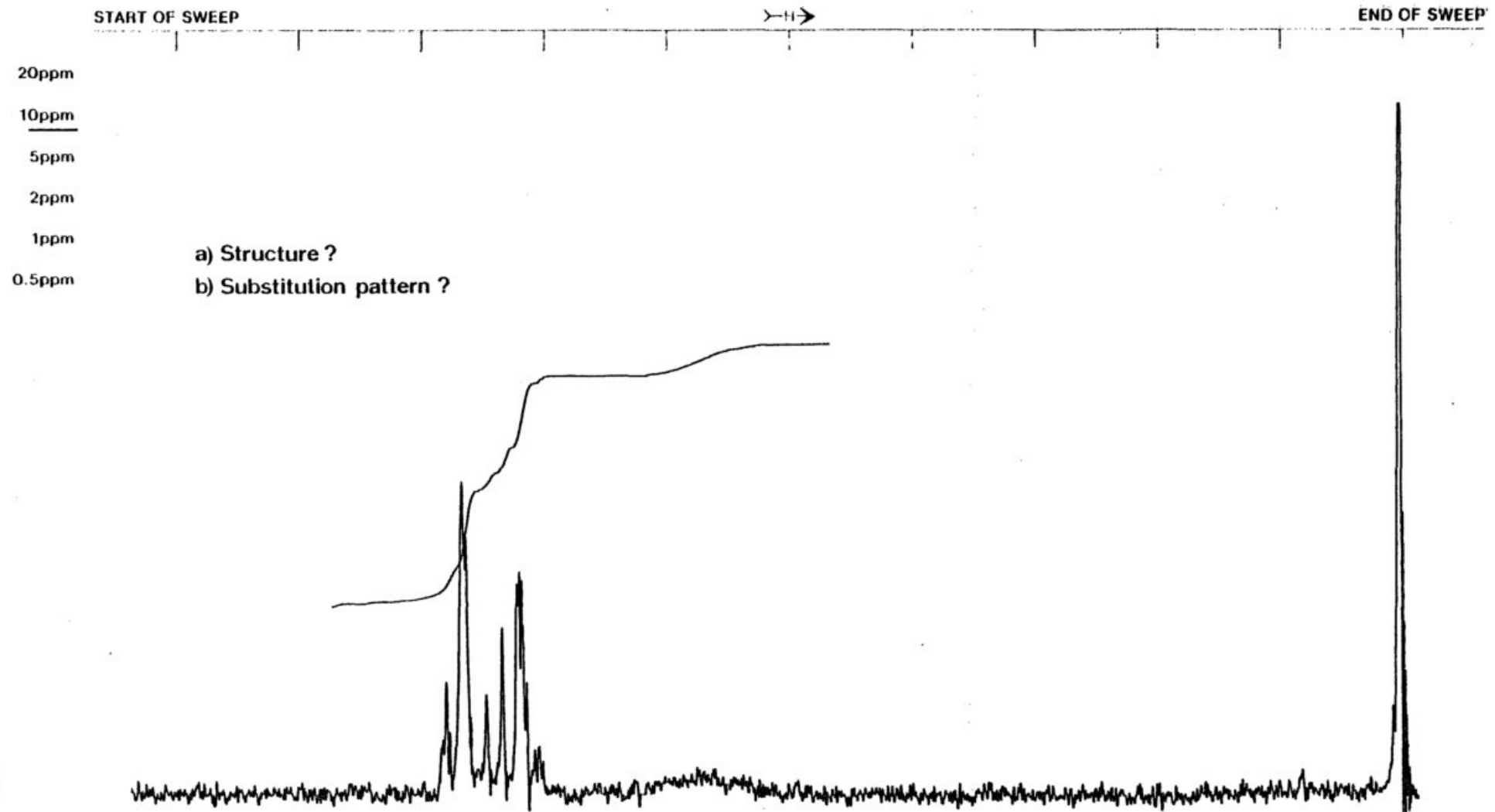


a) Structure ?
b) Substitution pattern ?

Sweep width = 5 ppm
and of sweep = 6 ppm

SPECTRUM AMPL. 200 SWEEP TIME 5 min SAMPLE: C₆H₅NO₃ REMARKS: OPERATOR T.W.
FILTER 0.02 sec SWEEP WIDTH 10 ppm or Hz DATE _____
RF POWER 0.1 mG END OF SWEEP 0 ppm or Hz SOLVENT: CDCl₃ SPECTRUM NO. 7

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ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

SPECTRUM AMPL. 1000 SWEEP TIME 5 min SAMPLE: C₆H₅NO₃ REMARKS: OPERATOR T.W.

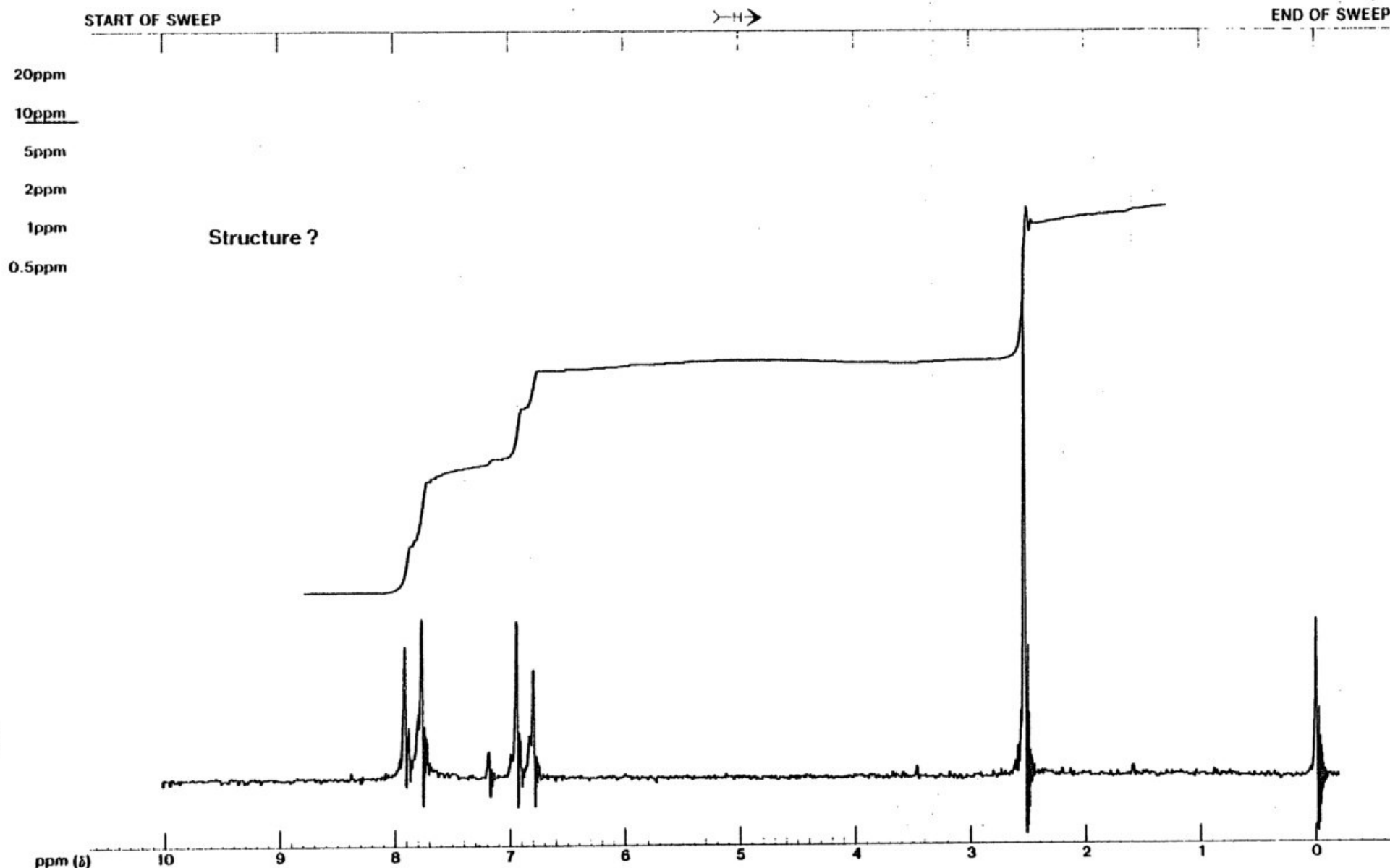
FILTER 0.1 sec SWEEP WIDTH 10 ppm or Hz DATE

RF POWER 0.1 mG END OF SWEEP 0 ppm or Hz SOLVENT: CDCl₃ SPECTRUM NO. 8



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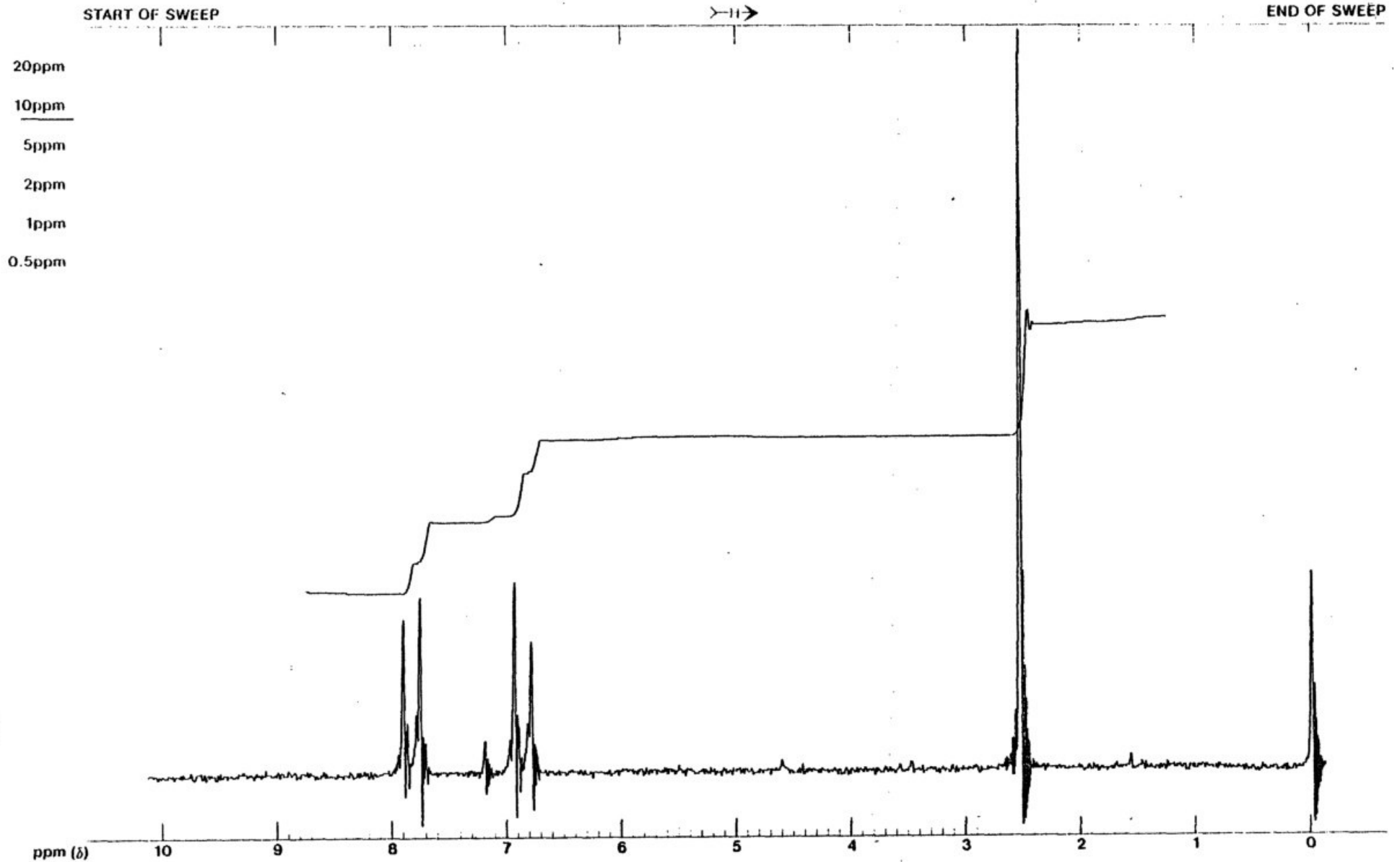


SPECTRUM AMPL. 250 SWEEP TIME 5 min SAMPLE: C₈H₈O₂ REMARKS: OPERATOR C.F.
 FILTER 0.05 sec SWEEP WIDTH 10 ppm or Hz DATE...
 RF POWER 0.05 mG END OF SWEEP 0 ppm or Hz SOLVENT: CDCl₃ SPECTRUM NO. 9a



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SPECTRUM AMPL. 250

SWEEP TIME 5 min

SAMPLE: C₂H₂O₂

REMARKS: with D₂O

OPERATOR C.F.

FILTER 0.05 sec

SWEEP WIDTH 10 ppm or Hz

DATE

RF POWER 0.05 mG

END OF SWEEP 0 ppm or Hz

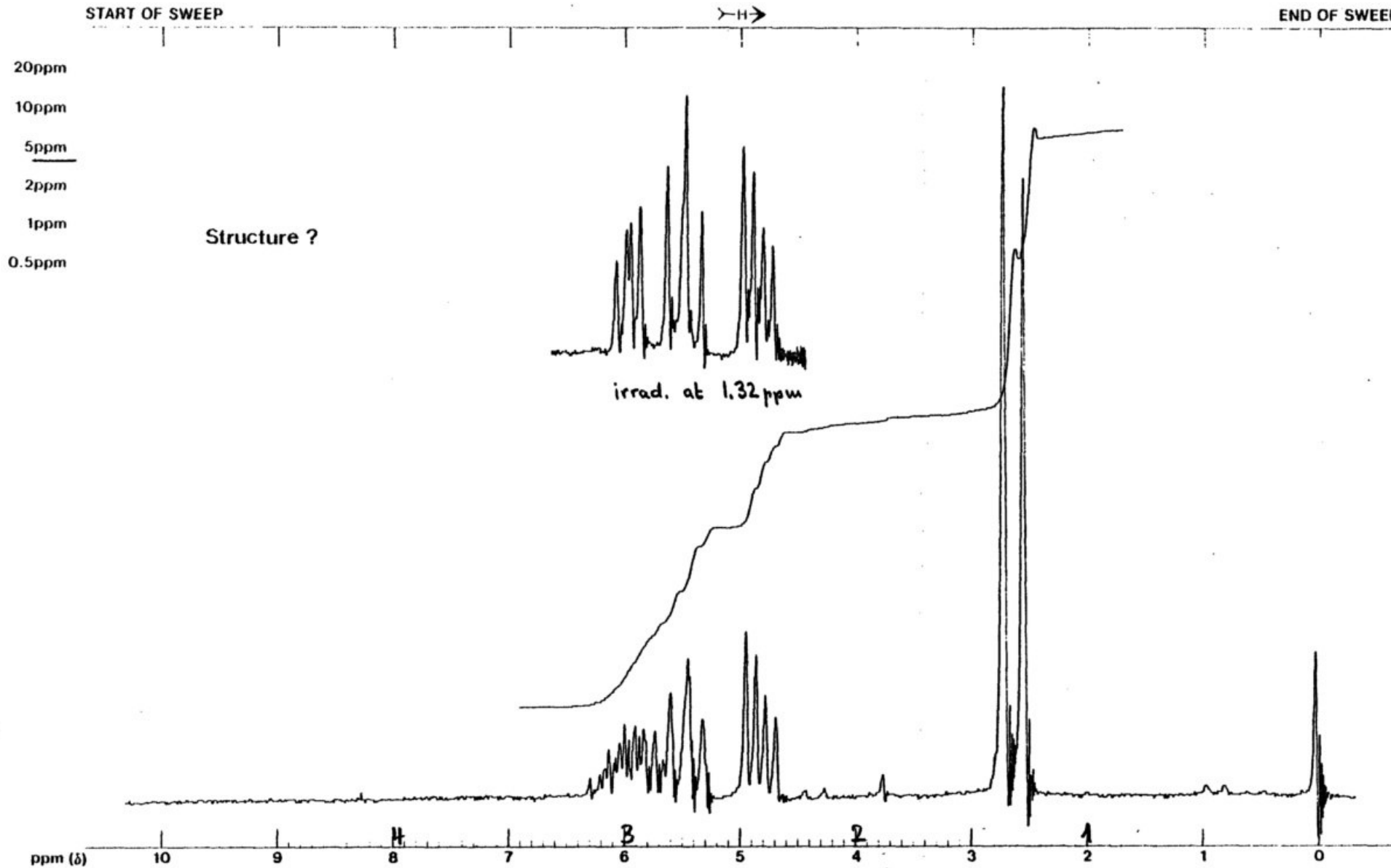
SOLVENT: CDCl₃

SPECTRUM NO. **9b**



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SPECTRUM AMPL. 120

SWEEP TIME 5 min

SAMPLE: C_3H_6O

REMARKS:

OPERATOR C.F.

FILTER 0.5 sec

SWEEP WIDTH 5 ppm or Hz

DATE

RF POWER 0.05 mG

END OF SWEEP 0 ppm or Hz

SOLVENT: $CDCl_3$

SPECTRUM NO. 10

START OF SWEEP



END OF SWEEP

20ppm

10ppm

5ppm

2ppm

1ppm

0.5ppm

irradiated at 1.93 ppm

Structure ?

sweep width = 5ppm

sweep width = 10ppm

sweep width = 5ppm
end of sweep = 4 ppm

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ppm (δ)

10

9

8

7

6

5

4

3

2

1

0

SPECTRUM AMPL. 250

SWEEP TIME 5 min

SAMPLE: C₆H₁₀O₂

REMARKS:

OPERATOR C.F.

FILTER 0.05 sec

SWEEP WIDTH 10 ppm or Hz

DATE

RF POWER 0.05 mG

END OF SWEEP 0 ppm or Hz

SOLVENT: CDCl₃

SPECTRUM NO.

11

START OF SWEEP



END OF SWEEP

20ppm
10ppm
5ppm
2ppm
1ppm
0.5ppm

Structure ?

irradiated at 1.26ppm

Sweep width = 5ppm
end of sweep = 0ppm

impurity

ppm (δ)

10

9

8

7

6

5

4

3

2

1

0

SPECTRUM AMPL. 300

SWEEP TIME 5 min

SAMPLE: C_4H_8O

REMARKS:

OPERATOR C.F.

FILTER 0.05 sec

SWEEP WIDTH 10 ppm or Hz

DATE:

RF POWER 0.05 mG

END OF SWEEP 0 ppm or Hz

SOLVENT: $CDCl_3$

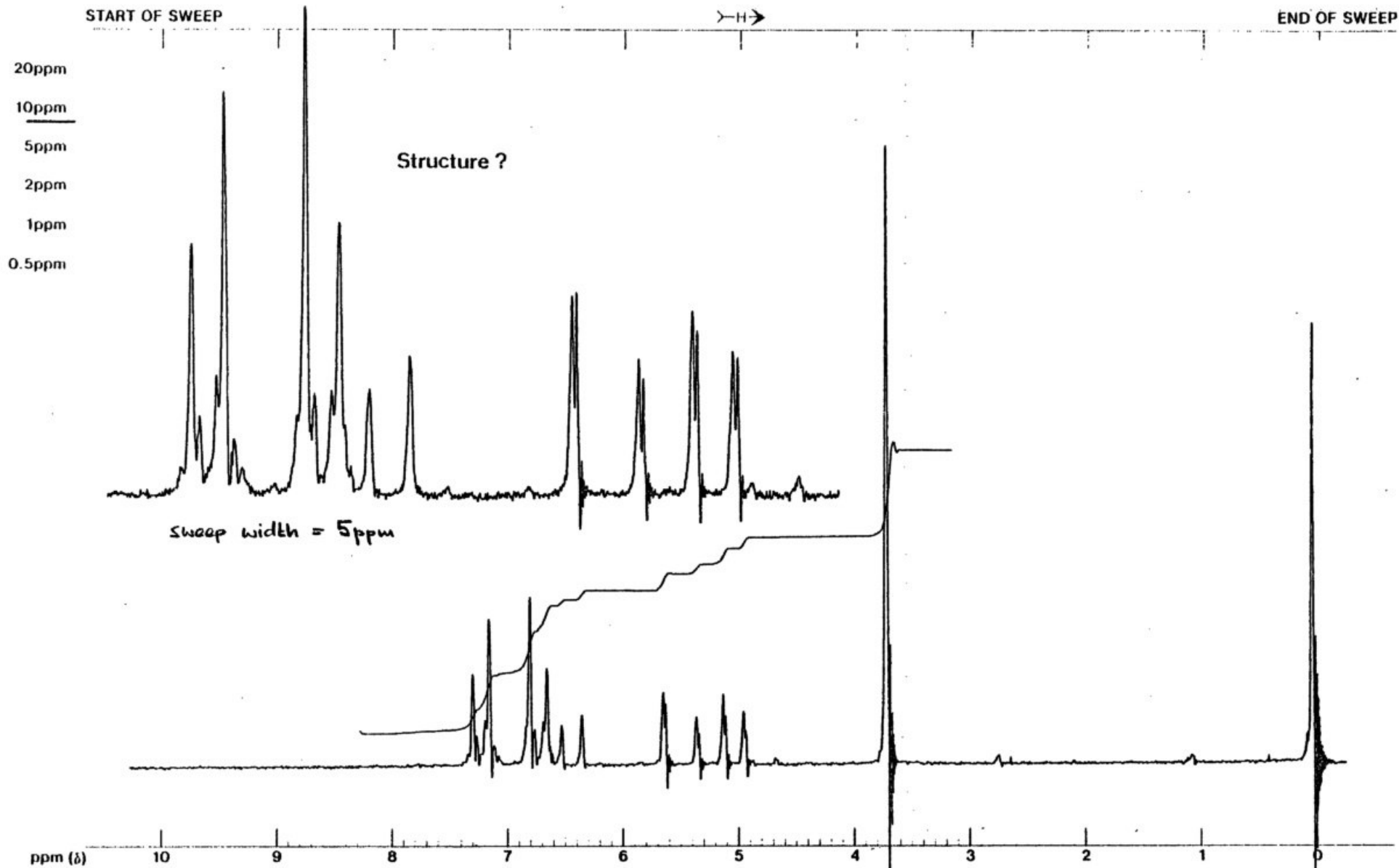
SPECTRUM NO. 12

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SPECTRUM AMPL. 100

SWEEP TIME 5 min

SAMPLE: C₉H₁₀O

REMARKS:

OPERATOR

T.W.

FILTER 0.02 sec

SWEEP WIDTH 10/5 ppm or Hz

DATE

RF POWER 0.05 mG

END OF SWEEP 0/2.5 ppm or Hz

SOLVENT: CDCl₃

SPECTRUM NO. 13



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START OF SWEEP



END OF SWEEP

20ppm
10ppm
5ppm
2ppm
1ppm
0.5ppm

Structure of this amino acid ?

ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

SPECTRUM AMPL. 400

SWEEP TIME 5 min

SAMPLE: $C_5H_9NO_2$

REMARKS:
Reference : DSS

OPERATOR T.W.

FILTER 0.02 sec

SWEEP WIDTH 10 ppm or Hz

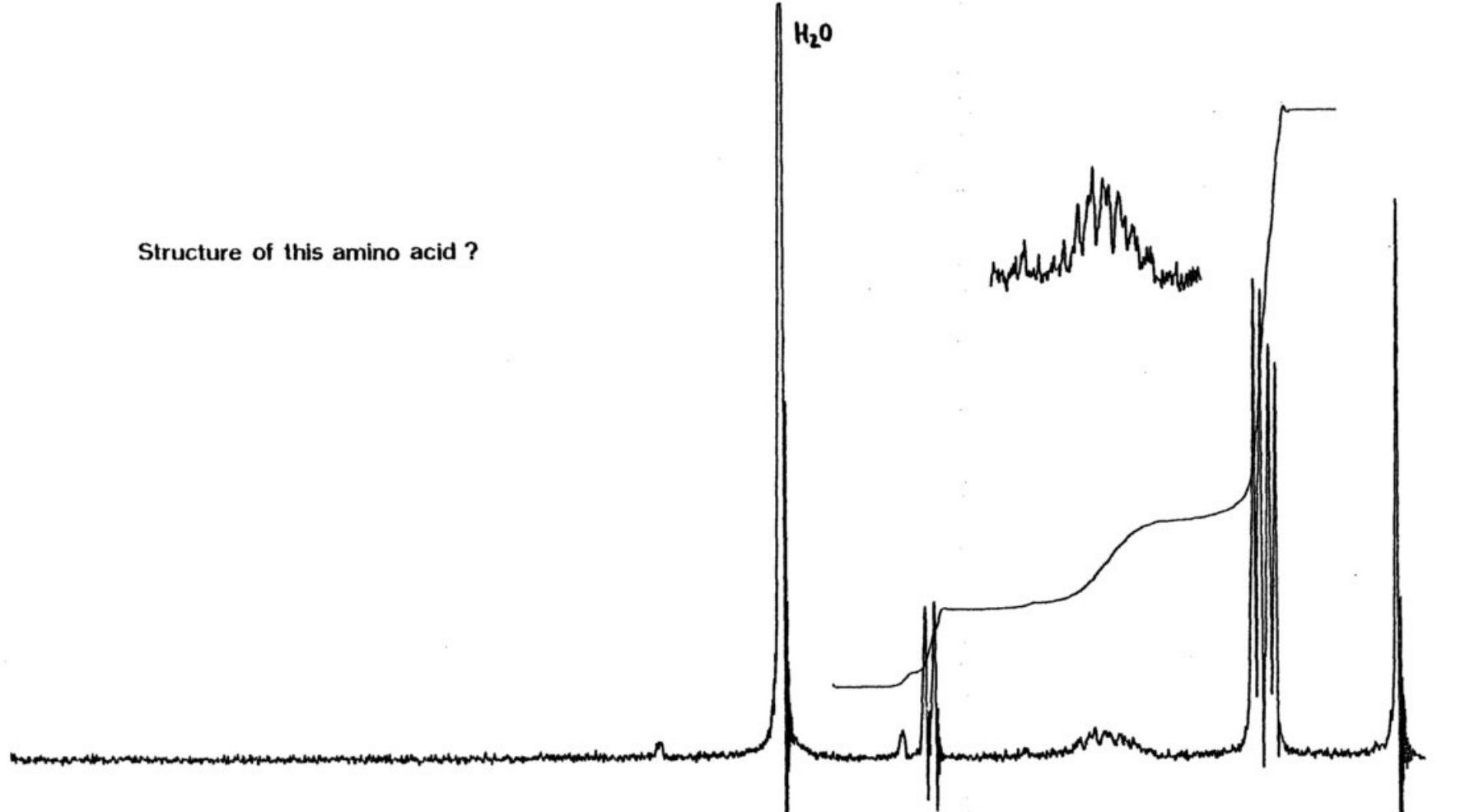
DATE

RF POWER 0.1 mG

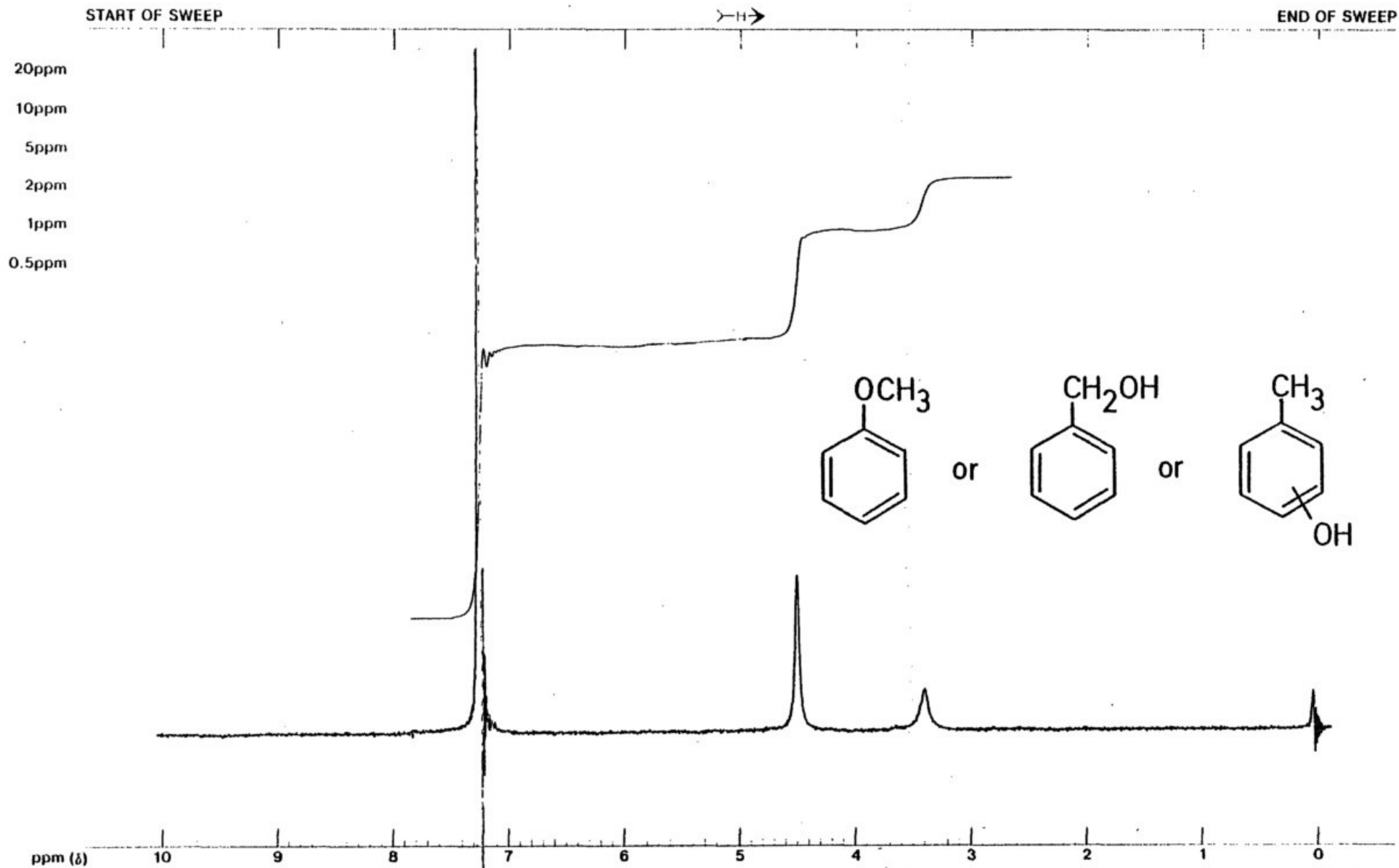
END OF SWEEP 0 ppm or Hz

SOLVENT: D_2O

SPECTRUM NO. 14



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SPECTRUM AMPL. 100

SWEEP TIME 5 min

SAMPLE: C₇H₈O

REMARKS:

OPERATOR K.H.

FILTER 0 sec

SWEEP WIDTH 10 ppm or Hz

DATE 5.8.74

RF POWER 0.04 mG

END OF SWEEP 0 ppm or Hz

SOLVENT: CDCl₃

SPECTRUM NO. 15



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START OF SWEEP



END OF SWEEP

20ppm
10ppm
5ppm
2ppm
1ppm
0.5ppm

Mixture Benzene/Cyclohexane
Determine molar ratios !

ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

SPECTRUM AMPL. 45

SWEEP TIME 5 min

SAMPLE: C₆H₆/C₆H₁₂ REMARKS:

OPERATOR K.H.

FILTER 0 sec

SWEEP WIDTH 10 ppm or Hz

DATE 5.8.74

RF POWER 0.03 mG

END OF SWEEP 0 ppm or Hz

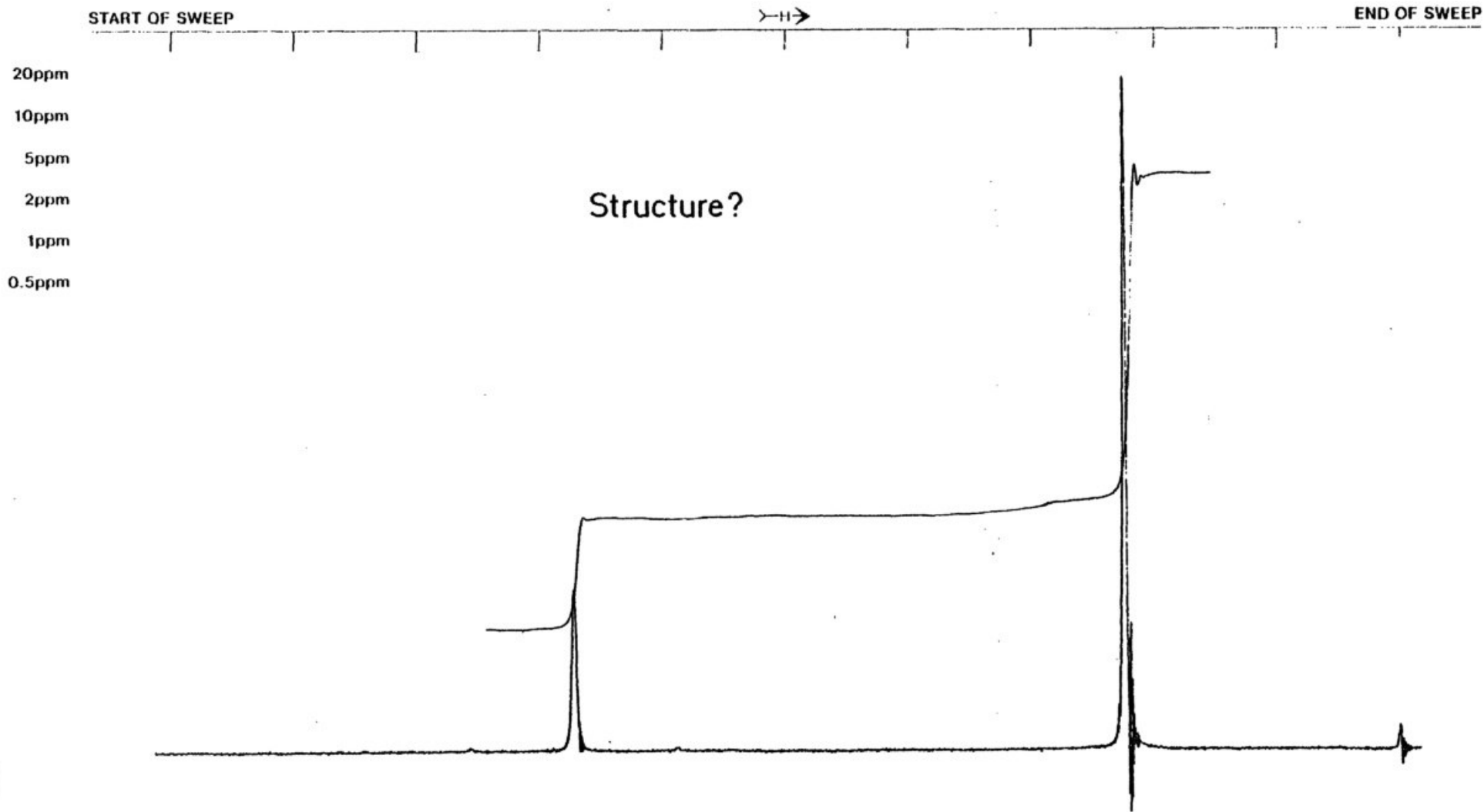
SOLVENT: CDCl₃

SPECTRUM NO. 16



varian anaspect

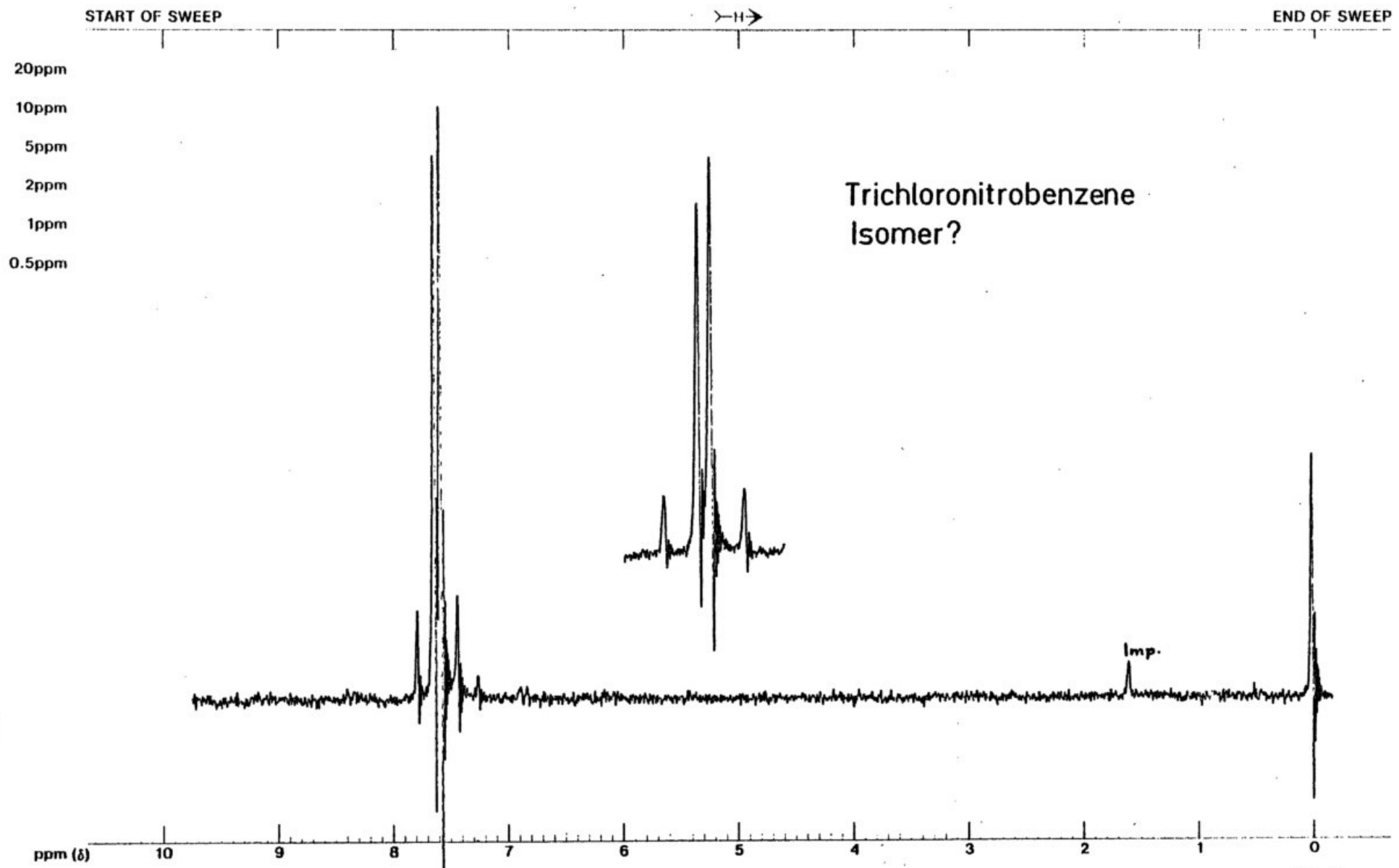
palo alto, california



ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

SPECTRUM AMPL. <u>90</u>	SWEEP TIME <u>5</u> min	SAMPLE: <u>C₉H₁₂</u>	REMARKS:	OPERATOR <u>K.H.</u>
FILTER <u>0</u> sec	SWEEP WIDTH <u>10</u> ppm or Hz			DATE <u>5.8.74</u>
RF POWER <u>0.04</u> mG	END OF SWEEP <u>0</u> ppm or Hz	SOLVENT: <u>CDCl₃</u>		SPECTRUM NO. <u>17</u>

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SPECTRUM AMPL. 900
FILTER 0.05 sec
RF POWER 0.05 mG

SWEEP TIME 5 min
SWEEP WIDTH 10 ppm or Hz
END OF SWEEP 0 ppm or Hz

SAMPLE: C₆H₂NO₂Cl₂ REMARKS:
SOLVENT: CDCl₃

OPERATOR K.H.
DATE 5.8.74
SPECTRUM NO. 18

START OF SWEEP

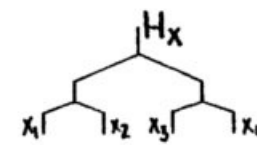
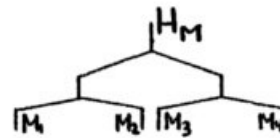
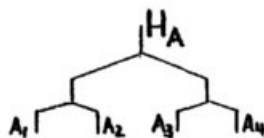
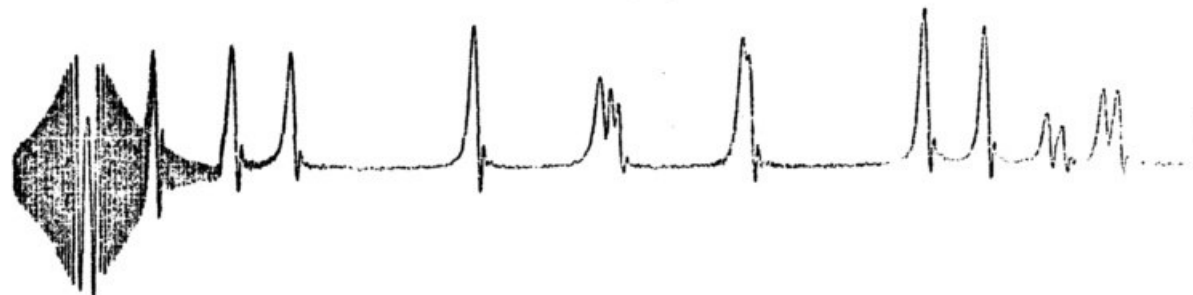
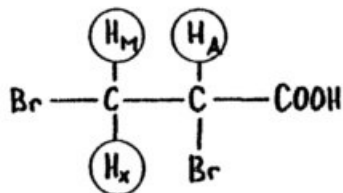


END OF SWEEP

20ppm
10ppm
5ppm
2ppm
1ppm
0.5ppm

SPIN TICKLING

(Determination of relative signs of coupling constants in an AMX system)



ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

SPECTRUM AMPL. 100

SWEEP TIME 5 min

SAMPLE: 20%
2,3-Dibromopropionic acid

REMARKS: H₂ power
.1 mG

OPERATOR F.L.

FILTER .05 sec

SWEEP WIDTH 120 ppm or Hz

DATE 6.6.74

RF POWER .05 mG

END OF SWEEP 120 ppm or Hz

SOLVENT: C₆D₆

SPECTRUM NO. 19



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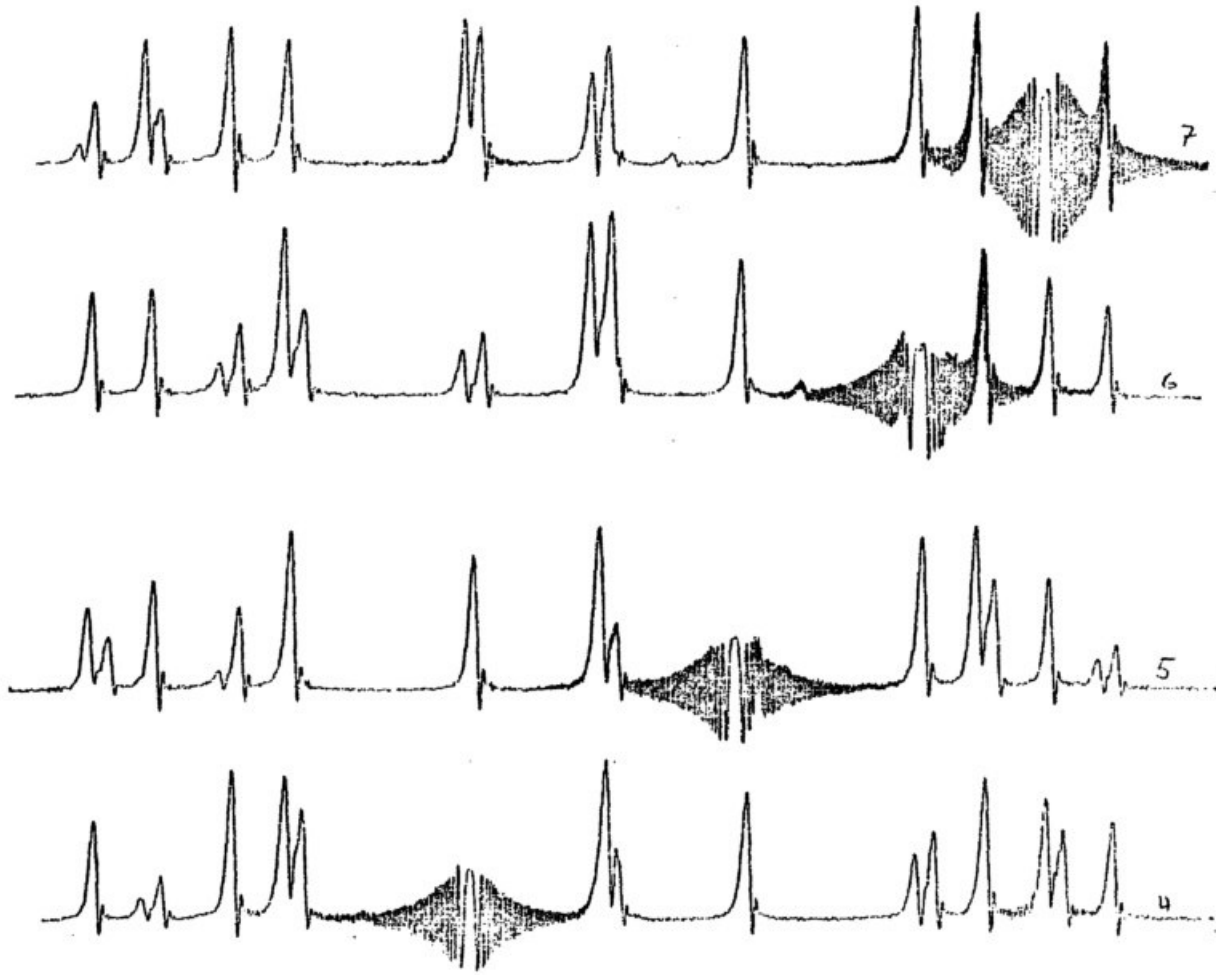
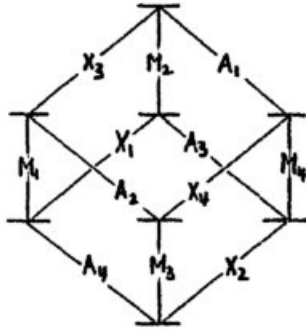
palo alto, california

START OF SWEEP



END OF SWEEP

20ppm
10ppm
5ppm
2ppm
1ppm
0.5ppm



ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

SPECTRUM AMPL. 100

SWEEP TIME 5 min

SAMPLE:

REMARKS:

OPERATOR F.L.

FILTER .05 sec

SWEEP WIDTH 120 ppm or Hz

DATE 6.6.74

RF POWER .05 mG

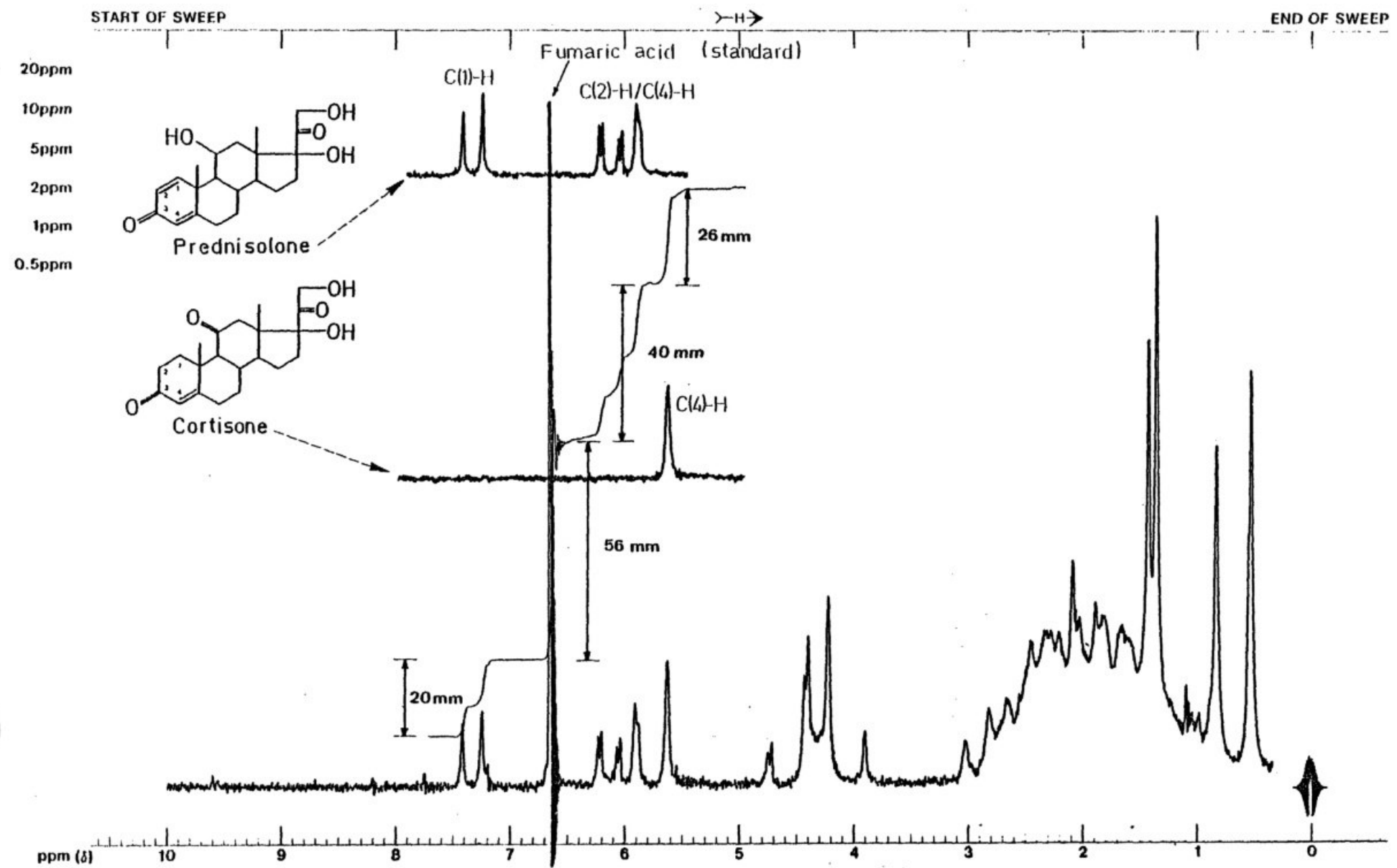
END OF SWEEP 120 ppm or Hz

SOLVENT:

SPECTRUM NO. 19a

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SPECTRUM AMPL. 2000
 FILTER 0.5 sec
 SWEEP TIME 10 min
 SWEEP WIDTH 10 ppm or Hz
 END OF SWEEP 0 ppm or Hz

SAMPLE: Prednisolone
 Cortisone
 SOLVENT: DMSO-d₆ + TMS

REMARKS: Standard 4.6mg Fumaric acid
 trace CF₃COOD added

OPERATOR R.L.
 DATE 28.12.77
 SPECTRUM NO. 20

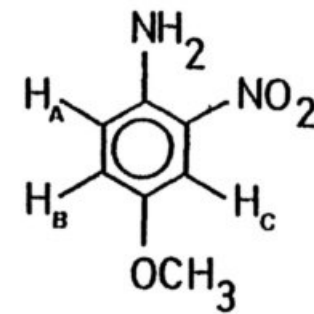
START OF SWEEP

>H<

END OF SWEEP

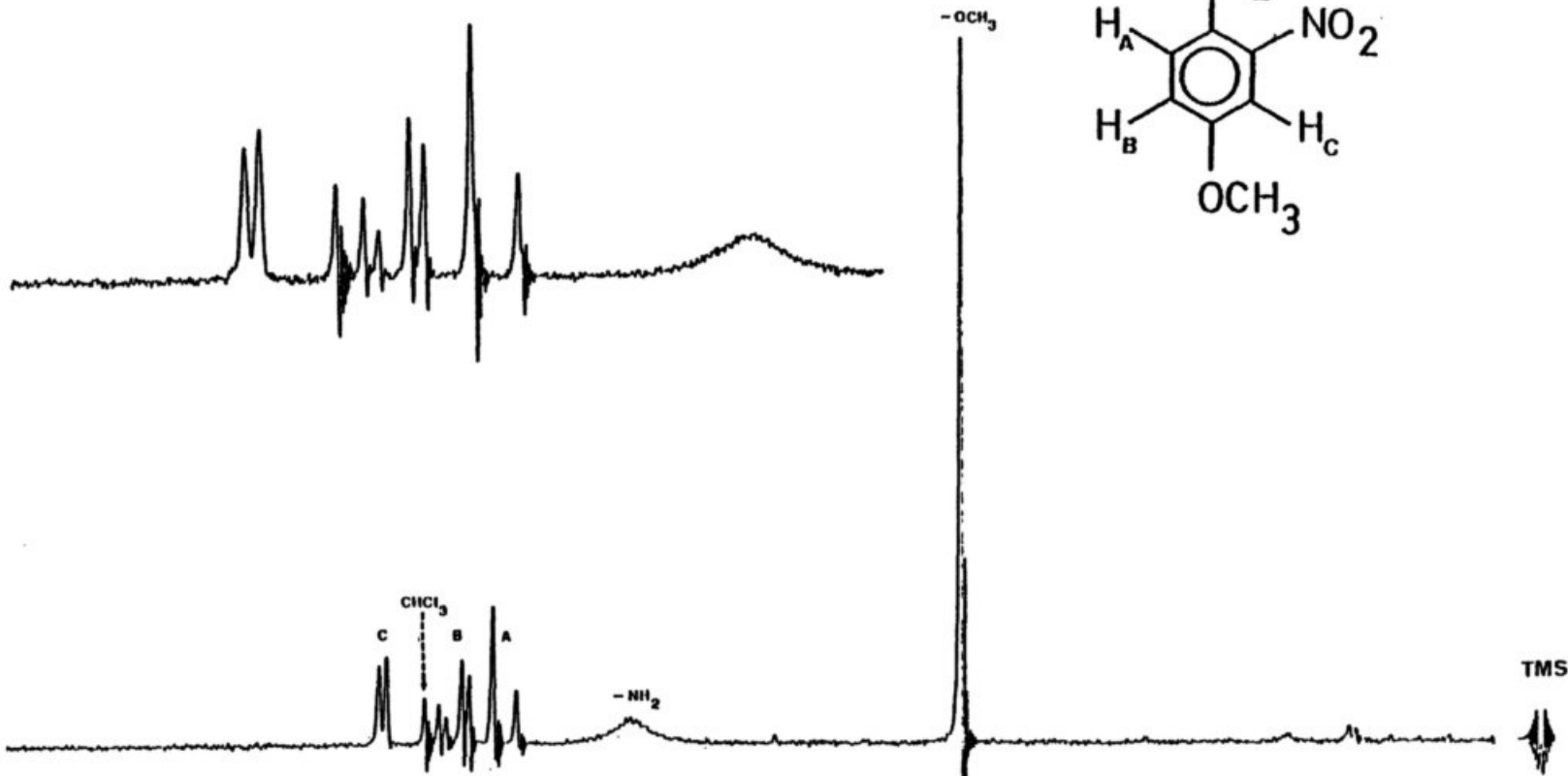
20ppm
10ppm
5ppm
2ppm
1ppm
0.5ppm

¹H EM-360L



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ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

SPECTRUM AMPL. 400
FILTER 0.05 sec
RF POWER 0.04 mG

SWEEP TIME 5 min
SWEEP WIDTH 10 ppm or Hz
END OF SWEEP 0 ppm or Hz

SAMPLE: Methoxy-p-Anisidin
SOLVENT: CDCl₃ + TMS

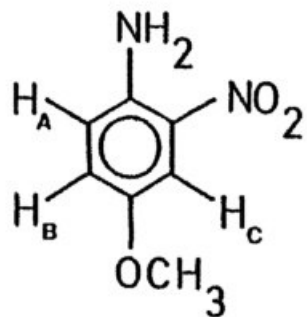
REMARKS:
OPERATOR R. L.
DATE 31. 1. 78
SPECTRUM NO. 21

START OF SWEEP

→H→

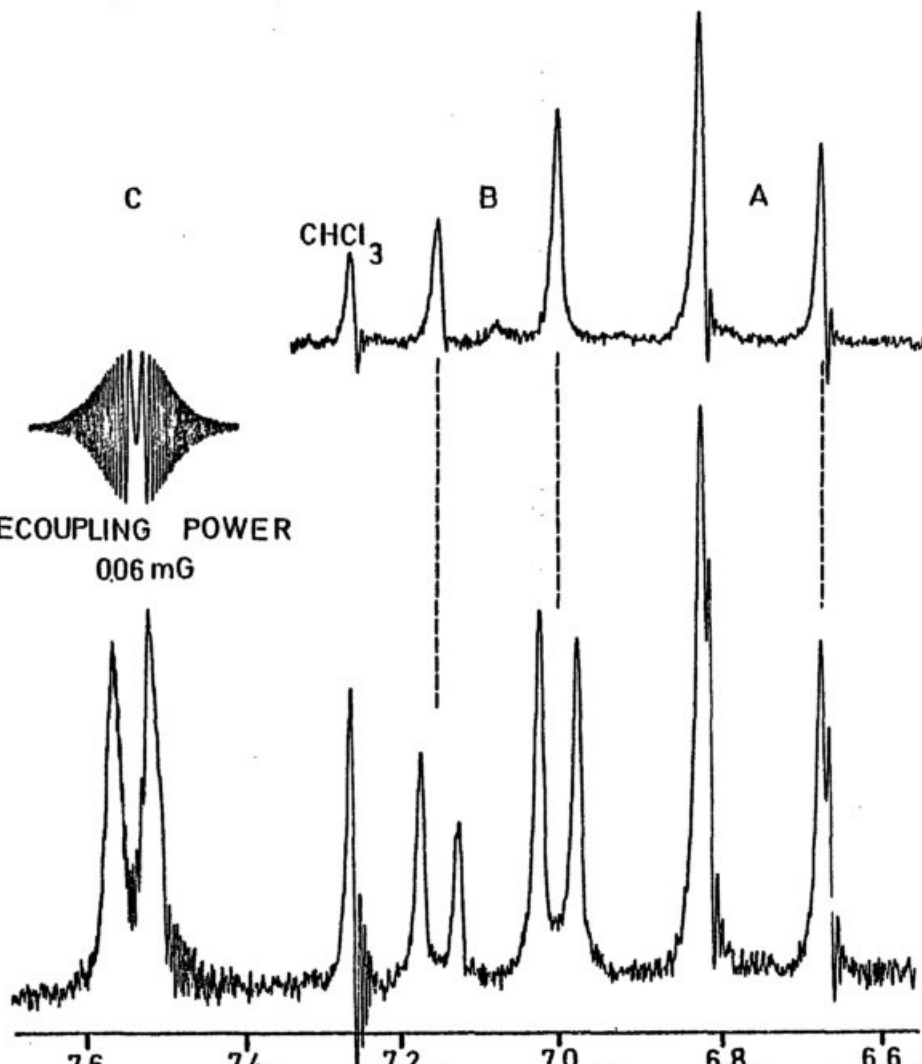
END OF SWEEP

20ppm
10ppm
5ppm
2ppm
1ppm
0.5ppm



EM-360L

DECOUPLING POWER
006 mG



ppm (δ) 10 9 8 7 6 5 4 3 2 1 0

SPECTRUM AMPL. 1000

SWEEP TIME 5 min

SAMPLE: REMARKS:

OPERATOR R. L.

FILTER 0.05 sec

SWEEP WIDTH 2 ppm or Hz

Methoxy-p-Anisidin

DATE 22.2.78

RF POWER 0.1 mG

END OF SWEEP 6.3 ppm or Hz

SOLVENT: CDCl₃ + TMS

SPECTRUM NO. **21a**

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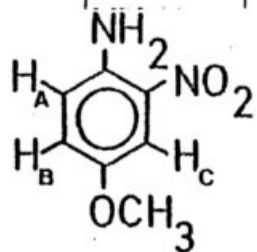
varian anaspect



START OF SWEEP

>--II-->

END OF SWEEP



INDOR
DECOUPLING POWER 0.08 mG

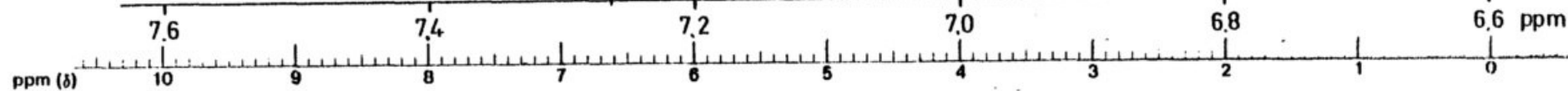
EM-360L

C

B

A

CHCl₃



SPECTRUM AMPL. 1900

SWEEP TIME 2 min

SAMPLE: Methoxy-p-Anisidin
REMARKS:

OPERATOR R. L.

FILTER 0.05 sec

SWEEP WIDTH 1 ppm or Hz

DATE 22.2.78

RF POWER 0.05 mG

END OF SWEEP 6.6 ppm or Hz

SOLVENT: CDCl₃ +TMS

SPECTRUM NO. **21b**

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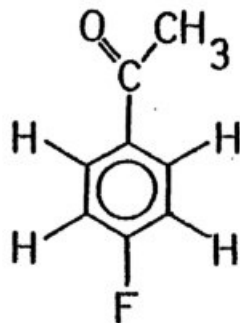
START OF SWEEP

→

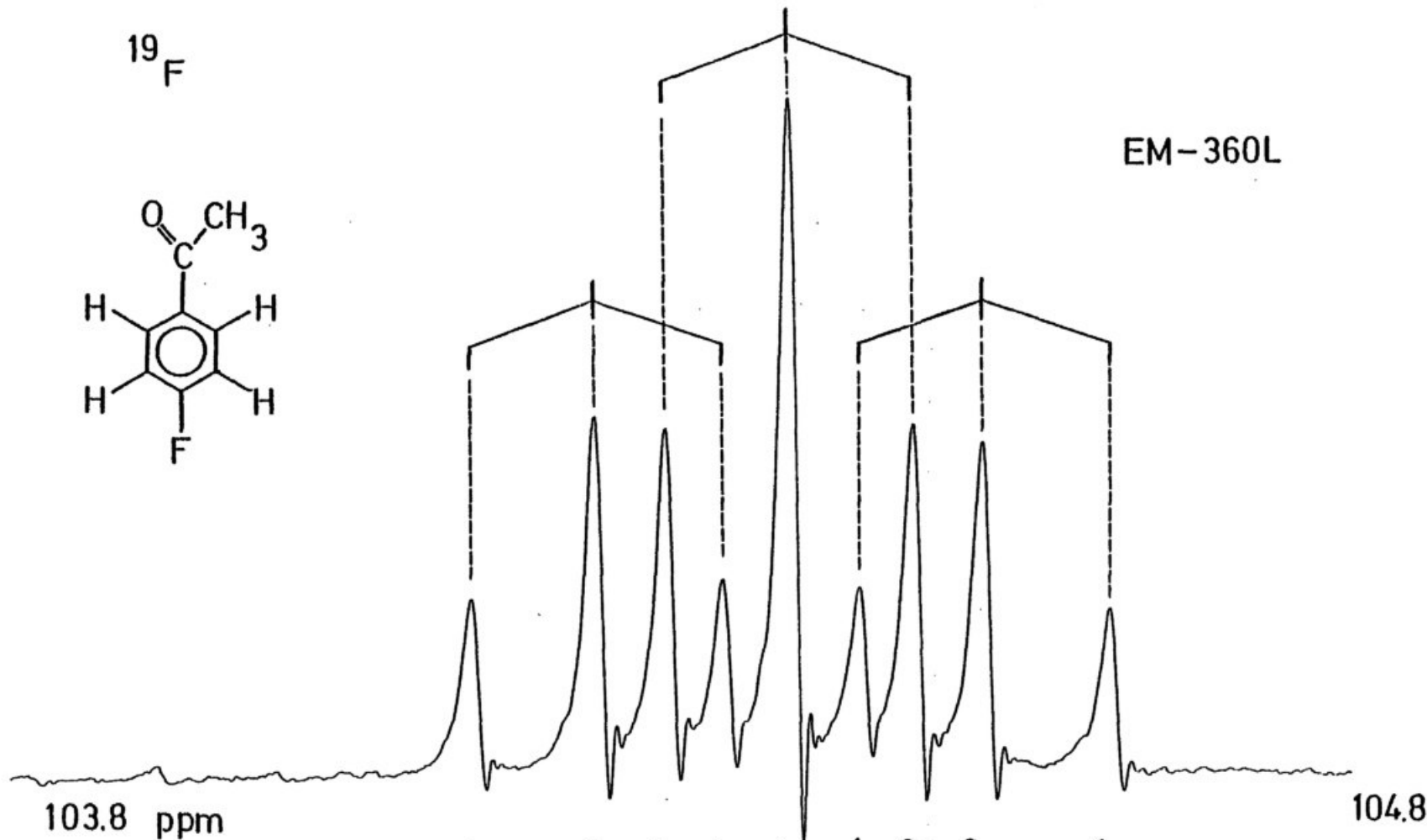
END OF SWEEP

10ppm
5ppm
2ppm

¹⁹F



EM-360L



103.8 ppm 1 : 2 : 2 : 1 : 4 : 1 : 2 : 2 : 1 104.8

LOCK POS. _____ ppm	SPECTRUM AMPL. <u>2000</u>	SWEEP TIME <u>2</u> min	NUCLEUS <u>¹⁹F</u>	SAMPLE: _____	OPERATOR <u>R. L.</u>
LOCK POWER _____ mG	FILTER <u>0.2</u> sec	SWEEP WIDTH <u>1</u> ppm	ZERO REF. <u>CFCl₃</u>	<u>p-Fluoroacetophenone</u>	DATE <u>20.12.7</u>
DECOUPLE POS. _____ ppm	RF POWER <u>0.1</u> mG	END OF SWEEP <u>+4.8</u> ppm	SAMPLE TEMP. <u>-1</u> °C	SOLVENT: <u>CDCl₃</u>	SPECTRUM NO. 22