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**Carbon-13 and Proton NMR Spectra for Characterizing
Thermosetting Polymer Systems 1: Epoxy Resins and
Curing Agents**

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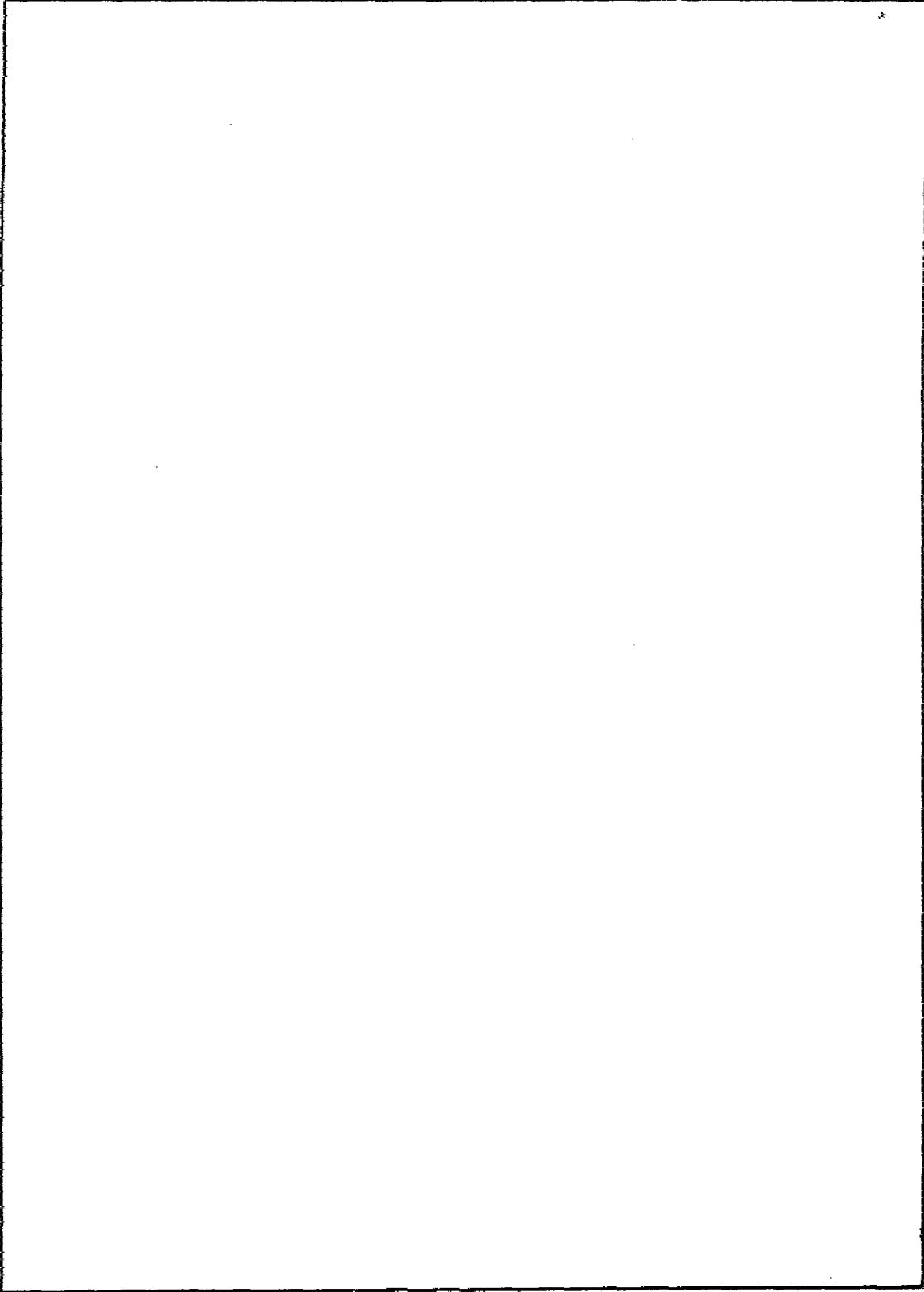
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CARBON-13 AND PROTON NMR SPECTRA FOR CHARACTERIZING THERMOSETTING POLYMER SYSTEMS

I. EPOXY RESINS AND CURING AGENTS

INTRODUCTION

High-resolution nuclear magnetic resonance (NMR) spectroscopy has become a valuable technique for the analysis of molecular structure of organic materials. The nuclei most studied are ^{13}C and hydrogen (proton). Recent advances in instrumentation have made both ^{13}C and proton NMR rapid and reliable. In addition, NMR, especially ^{13}C NMR, is much more specific than other spectroscopic techniques, such as infrared spectroscopy. Often, mixtures can be analyzed by inspection of the spectrum of the unseparated sample. Nuclear magnetic resonance has been widely employed in the study of thermoplastic polymers [1,2], but has been employed much less in the study of thermosetting polymers. New pulse NMR techniques are now making it possible to study solid polymers [3,4].

This report is a catalog that resulted from our need for reference proton and ^{13}C NMR spectra for use in characterizing epoxy resin systems [5]. Data on a few of these materials are scattered throughout the chemical literature. However, we feel that this collection of both proton and ^{13}C NMR spectra of epoxy resins and curing agents will be useful to others working in polymer characterization.

Because this catalog is designed for the practicing NMR spectroscopist, we have not presented any discussion of NMR theory or practice. However, a number of references [6-10] are included for the guidance of those in the polymer analysis field who are unfamiliar with NMR techniques.

SCOPE

This volume is restricted to epoxy resins and curing agents. A future volume will include plasticizers, more complex epoxy systems, and precursors (or prepolymers) of other thermosetting polymers such as polyimides.

We have not included the spectra of all the epoxy resins that we have investigated. In some cases, spectra for materials of the same viscosity range from different manufacturers were so similar that the inclusion of multiple spectra would serve no useful purpose. However, those cases are noted in the index of spectra that appears in Appendix A.

FORMAT

With the ^{13}C spectra the following information for each material is given: chemical name or chemical nature, trade name or other source, solvent used, structure, and table of chemical shifts. With the proton spectra only the name, source, and solvent appear. The structures shown on the ^{13}C spectra are from manufacturers' literature, from the handbook by Lee and Neville [11], or from the chemical name. We have generally used a standard chemical shorthand for the structures. Some examples follow:

<i>Chemical name</i>	<i>Chemical formula</i>	<i>Brief version</i>
Butoxy group	$\text{CH}_3\text{CH}_2\text{CH}_2\text{CH}_2\text{O}-$	
Glycidyl group	$\text{H}_2\text{C}=\text{O}-\text{CHCH}_2-$	
Phenyl ring		

Our spectral assignments for most of the ^{13}C spectra are based on chemical-shift substituent effects obtained from the literature [8,9] or calculated from the spectra of model compounds. We ran the spectra of model compounds if data for them were not available. In a few cases we ran proton-coupled ^{13}C spectra to aid in the assignments. The line assignments are indicated by a letter near the corresponding carbon atom in the structure. In some cases we have made no assignments either because we do not know the structure or because the spectrum is too complex. No assignments are given for any of the proton spectra.

Appendix A contains carbon-13 NMR line listings of three materials having spectra that are quite complex.

CAUTION

We remind the user of this catalog of the pitfalls of "fingerprint" spectra comparison. The first is the effect of solvents. In both ^{13}C and proton spectra, the concentration and the solvent can have widely varying effects on both line shape and position. Another pitfall is that the relative intensities of ^{13}C spectral lines are notoriously dependent on experimental conditions, such as viscosity and pulse repetition rate. Finally, because most of the materials are standard commercial products, manufacturing impurities, additives, and reaction products formed during storage may be present.

EXPERIMENTAL PROCEDURES

Most of the samples were prepared from material on hand. The remainder were either purchased from chemical supply houses or were samples received from manufacturers. The inclusion of a material in this catalog does not imply that the material is still

made by the manufacturer that we list as the source, that it is only available from that manufacturer, or even that it is still commercially available. Unless otherwise specified, materials were used as received.

Carbon-13 spectra were obtained at 25.15 MHz with a Varian HA-100 spectrometer system, modified for pulsed Fourier transform operation, proton decoupling, and external fluorine-19 field-frequency lock. The pulse interval and number of scans varied with the sample, but were usually 3 to 5 s and 500 scans, respectively. The spectra 250 ppm and collection size was either 4000 or 8000 data points. Data and processed with either a Nicolet 1080 or Nicolet 1180 NMR data syst solutions were prepared to obtain maximum concentration consistent with viscosity and solubility considerations. Solvents and approximate solvent concentration by volume are given on each spectrum. Solvent peaks in the spectra are indicated by the symbol x. Chemical shifts are in parts per million from tetramethylsilane (TMS). The solvents, whose chemical shifts relative to TMS were measured separately, served as internal references.

Proton spectra were obtained at 100 MHz on either a Varian HA-100 or Jeol PS-100 spectrometer system. The sweep width was 10 ppm, and spectra were recorded at 0.01 ppm/s. Sample concentrations were 10% or less by volume. The internal reference was TMS.

OTHER NMR SPECTRA CATALOGS

Listed here are the proton and ^{13}C NMR spectra catalogs that we know about. We would appreciate hearing of any which we have not listed.

Proton

1. Varian Associates High Resolution NMR Spectra Catalog, Combined Vols. 1 and 2, N.S. Bhacca, D.P. Hollis, L.F. Johnson, E.A. Pier, and J.N. Shoolery, Varian Associates, Palo Alto, Calif., 1962 and 1963
2. The Sadtler Guide to the NMR Spectra of Polymers, W.W. Simons and M. Zanger, Sadtler Research Laboratories, Inc., Philadelphia, Pa., 1973
3. Sadtler Standard Nuclear Magnetic Resonance Spectra, Sadtler Research Laboratories, Inc., Philadelphia, Pa., (continuing)*
4. The Aldrich Library of NMR Spectra, C.J. Pouchert and J.R. Campbell, Aldrich Chemical Company, Inc., Milwaukee, Wis., 1974.
5. Catalog of Nuclear Magnetic Resonance Spectral Data (American Petroleum Research Project 44 and Manufacturing Chemists Association Research Project), Chemical Thermo-dynamic Properties Center, Texas A&M University, College Station, Tex. (Loose leaf data sheets, extant.)

*Sadtler also markets a number of specialized smaller NMR spectra collections.

Carbon-13

1. Carbon-13 NMR Spectra, L.F. Johnson and W.C. Jankowski, John Wiley and Sons, Inc., New York, N.Y., 1972
2. Sadtler Standard Carbon-13 NMR Spectra, Sadtler Research Laboratories, Inc., Philadelphia, Pa., 1976

REFERENCES

1. F.A. Bovey, *High Resolution NMR of Macromolecules*, Academic Press, Inc., New York, 1972.
2. J. Schaefer, Chap. 4, in *Topics in Carbon-13 NMR Spectroscopy*, Vol. 1 (G.C. Levy, editor), Wiley Interscience, New York, 1974.
3. J. Schaefer, E.O. Stejskal, and R. Buchdahl, *Polym. Prepr. Div. Polym. Chem.*, 17, No. 2, 17 (1976).
4. A.N. Garroway, W.B. Moniz, and H.A. Resing, *ACS Coatings and Plastics Preprints* 36, No. 2, 133 (1976).
5. C.F. Poranski, Jr., and W.B. Moniz, *ACS Coatings and Plastics Preprints* 36, No. 2, 139 (1976).
6. J.A. Pople, W.G. Schneider, and H.J. Bernstein, *High Resolution Nuclear Magnetic Resonance*, McGraw Hill Book Co., Inc., New York, 1959.
7. J.W. Emsley, J. Feeney, and L.H. Sutcliffe, *High-Resolution Nuclear Magnetic Resonance Spectroscopy*, Pergamon Press, New York, 2 vols. 1965, 1966.
8. J.B. Stothers, *Carbon-13 NMR Spectroscopy*, Academic Press, New York, 1972.
9. G.C. Levy and G.L. Nelson, *Carbon-13 Nuclear Magnetic Resonance for Organic Chemists*, Wiley-Interscience, New York, 1972.
10. T.C. Farrar and E.D. Becker, *Pulse and Fourier Transform NMR*, Academic Press, Inc., New York, 1971.
11. H. Lee and K. Neville, *Handbook of Epoxy Resins*, McGraw Hill Book Co., Inc., New York, 1967.

Appendix A

CARBON-13 NMR LINE LISTINGS FOR THREE MATERIALS

In this we give the complete line listings for three materials for which the spectra are quite complex because of the presence of several structural isomers. These materials are 3,4-epoxycyclohexylmethyl-(3,4-epoxy)cyclohexane carboxylate (see Spectrum 23), 3,4-epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexane carboxylate (see Spectrum 24), and methyl-4-*endo*-methylene tetrahydrophthalic anhydride (see Spectrum 50). In the case of the anhydride, additional complexity may arise from the presence of dicarboxylic acids formed by hydrolysis. Line positions in parts per million from TMS follow:

1. 3,4-Epoxyhexylmethyl-(3,4-epoxy)cyclohexane carboxylate:

22.7	29.7	52.4
24.5	31.7	52.7
25.2	33.9	53.1
25.5	37.4	53.3
26.1	39.1	69.5
27.9	51.6	175.3
28.7	51.9	175.8
28.9	52.2	

2. 3,4-Epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexane carboxylate:

17.1	29.8	49.6
18.9	30.9	50.5
19.5	31.2	50.8
23.8	32.3	51.1
24.5	33.2	51.4
25.6	34.3	51.7
26.3	40.0	173.1
26.6	35.2	65.1
27.8	38.3	65.6
28.9	43.2	173.7
29.4	45.9	174.1

3. Methyl-4-*endo*-methylene tetrahydronaphthalic anhydride:

15.0	49.2	130.2
16.3	50.0	135.4
43.5	50.4	136.6
46.0	50.6	137.8
46.4	51.0	139.6
46.7	51.4	142.6
46.9	51.5	146.5
47.1	52.7	148.0
47.3	55.2	171.5
48.0	70.4	171.8
48.2	127.0	172.1

INDEX OF MATERIALS

The materials in this index are listed in alphabetical order by chemical names or chemical type. If the sample has a particular manufacturer's product designation, it is listed under that designation also. The "see" cross reference refers to a material that has a ^{13}C spectrum that is virtually identical to the spectrum of the material listed. The "similar to" cross reference refers to a material that has a spectrum that differs only slightly from the spectrum of the material listed.

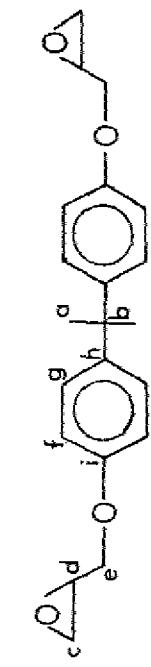
<i>Name</i>	<i>Spectrum No.</i>	<i>Name</i>	<i>Spectrum No.</i>
2-aminoethanol	31	<i>N,N</i> -dimethylbenzylamine	40
2-(2-aminoethylamino) ethanol . . .	33	<i>N,N</i> -dimethylethylenediamine	28
aminoethylethanolamine	33	<i>sym</i> -dimethylethylenediamine	28
bis(4-aminophenyl) sulfone	43	4,8-dioxatricyclo[5.1.0.0 ^{3,5}]octane	20
<i>p</i> -aminophenyl sulfone	43	4-DI-PIP	37
Araldite 502	9	DMP-30	41
Araldite 906	50	ECN 1235	19
Araldite 7071	3	Emery 9872	44
Araldite MY720	17	EP 2114. <i>See</i> ERL-2114.	
benzyldimethylamine	40	Epi-Rez SU-8	10
chlorendic anhydride	49	Epon 815	8
CIBA 906	50	Epon 828	2
CIBA 3794	12	Epon 1001. <i>Similar to</i> Araldite 7071.	
1,4-cyclohexane diepoxyde	20	Epon 1004	4
1,4-cyclohexane- <i>bis</i> (methylamine)	38	Epon 1007	5
D.E.N. 431	18	Epon 1031	13
D.E.R. 332. <i>See</i> D.E.R. 332LC.		Epoxide 201	24
D.E.R. 332LC	1	Epoxide 206	22
D.E.R. 542	11	3,4-epoxycyclohexylmethyl-(3, 4-epoxy)cyclohexyl carboxylate	23
D.E.R. 661. <i>Similar to</i> Araldite 7071.		4-(1,2-epoxyethyl)-1,2- epoxycyclohexane	22
diaminodiphenyl sulfone	43	3,4-epoxy-6-methylcyclohexylmethyl- 3,4-epoxy-6-methylcyclohexane carboxylate	24
1,3-diamino-2-propanol	32	Epoxy Novolac resin:	
<i>bis</i> (<i>N,N</i> -di(2,3-epoxypropyl)- 4-aminophenyl)methane	17	D.E.N. 431	18
3-diethylaminopropylamine	30	ECN 1235	19
diethylenetriamine	25		
diglycidyl ether of tetrabromo- <i>bis</i> -phenol A	11		
2,4,6- <i>tris</i> (dimethylaminomethyl) phenol	41		

epoxy resin, DGEBA type:	
Araldite 7071	3
D.E.R. 332. <i>See</i> D.E.R. 332LC.	
D.E.R. 332LC	1
D.E.R. 661. <i>Similar to</i>	
Araldite 7071.	
Epon 828	2
Epon 1001. <i>Similar to</i>	
Araldite 7071.	
Epon 1004	4
Epon 1007	5
epoxy resin, DGEBA type plus	
butyl glycidyl ether;	
Epon 815	8
di-n-butyl phthalate;	
Araldite 502	9
bis-(2,3-epoxycyclopentyl)	
ether; ERL 2258	6
n-octyl glycidyl ether;	
Genepoxy M195	7
epoxy resin, DGEBA type	12
epoxy resin, polyfunctional	10
bis(2,3-epoxycyclopentyl)ether,	
trans isomer	21
4-(2,3-epoxy)propoxy-N,N-bis(2,	
3-epoxypropyl)-aniline	15
N,N-bis(2,3-epoxypropyl)-2,4,	
6-tribromoaniline	14
ERL-0400	21
ERL-0510	15
ERL-2114	20
ERL-2258	6
ERLA-4221	23
ERX-67	14
ethanolamine	31
Genepoxy M195	7
H-221	34
1,4,5,6,7,7-hexachloro-5-	
norbornene-2,3-dicarboxylic	
anhydride	49
hexahydrophthalic anhydride	48
hexamethylenetetramine	35
HHPA	48
3,3'-imino-bis-propylamine	29
Kopox 159	16
maleic anhydride	45
1,4-bis(methylamino)cyclohexane	38
methyl-4- <i>endo</i> -methylene	
tetrahydrophthalic anhydride	50
4,4'-methylenedianiline	42
MXDA	39
MY720	17
NMA. <i>Similar to</i> CIBA 906.	
PAPA	44
phthalic anhydride	46
piperidine	36
polyazelaic polyanhydride	44
polyglycidyl ether of tetraphenylene	
ethane	13
polyglycoldiamine	34
resorcinol diglycidyl ether	16
tetraethylenepentamine	27
tetraglycidyl methylenedianiline	17
tetrahydrophthalic anhydride	47
tridimethylaminomethyl phenol	41
triethylenetetramine	26
triglycidyl-p-aminophenol	15
4,4'-trimethylene-dipiperidine	37
vinyl cyclohexene dioxide	22
<i>m</i> -xylenediamine	39
ZZLB 0822	34

Carbon-13 and Proton NMR Spectra

C1

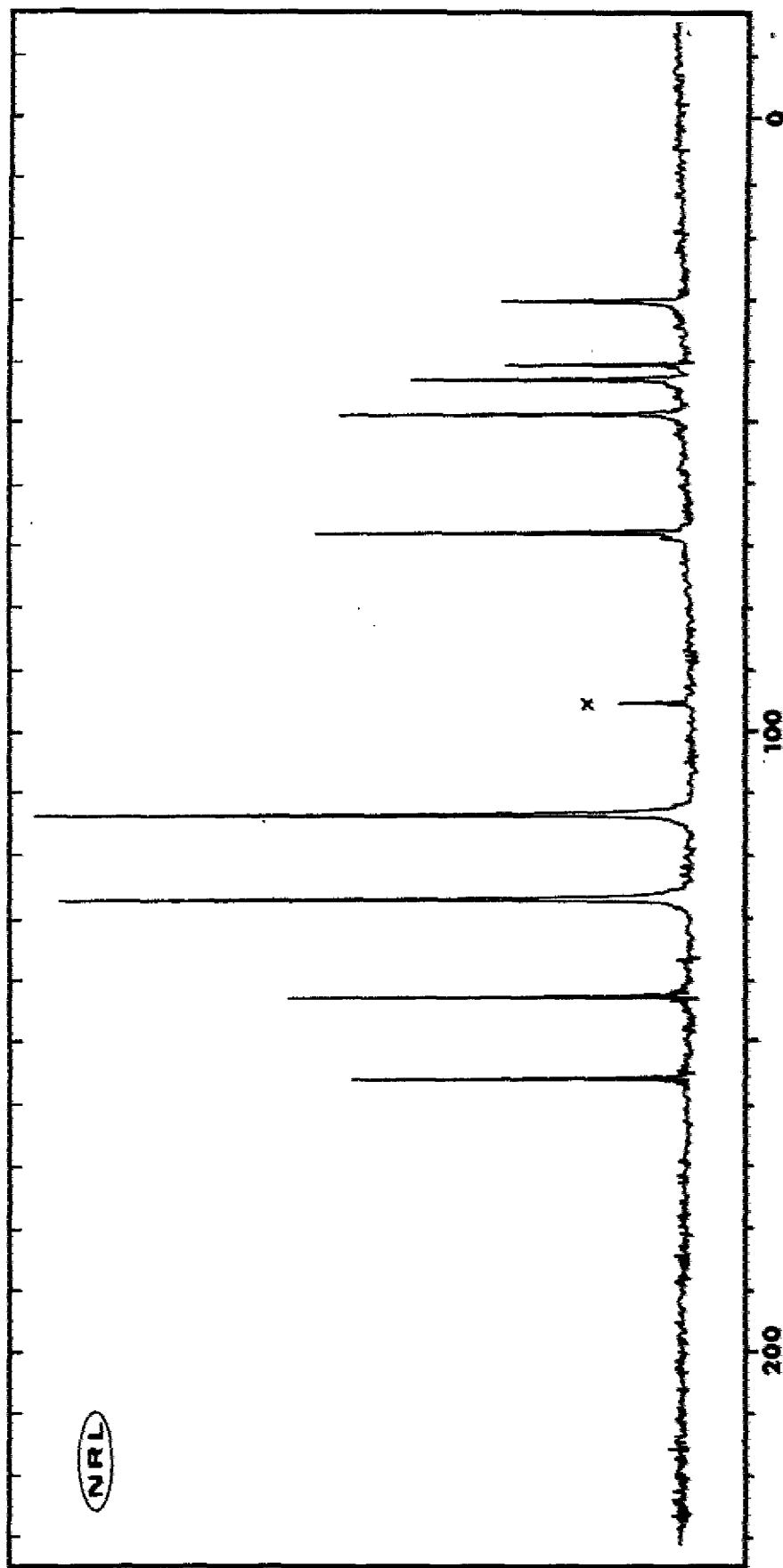
Epoxy Resin, DGEBA Type



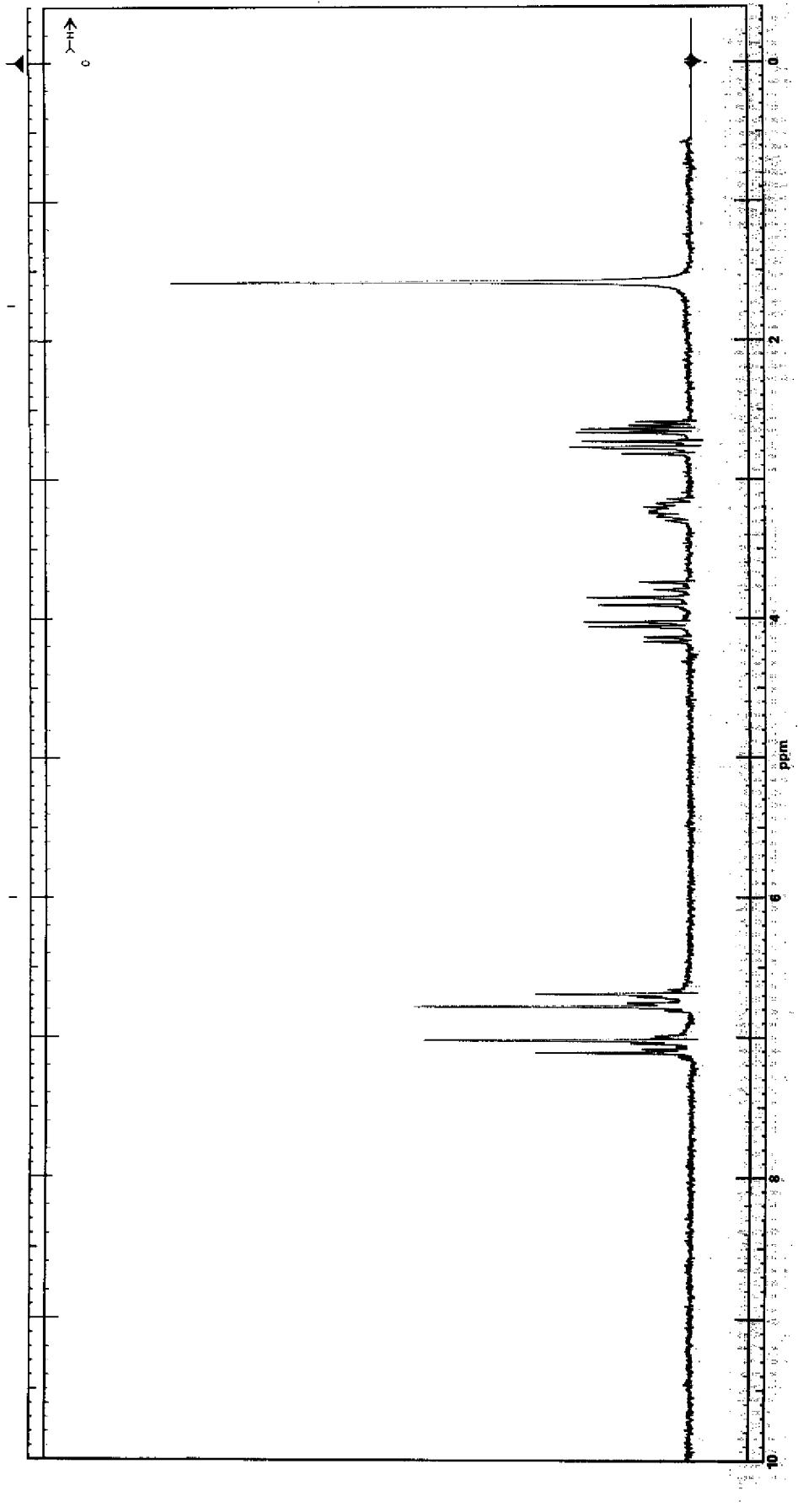
Assignments:	
a	30.1
b	40.7
c	43.1
d	49.1
e	68.1
f	113.2
g	126.7
h	142.4
i	155.4

Source: Dow D.E.R. 332LC

Solvent: 50% CCl_4 *



H1



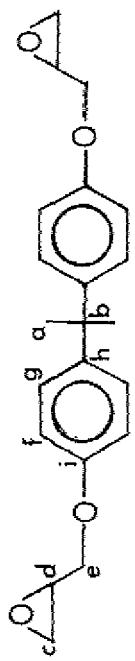
Spectrum 1 — Epoxy resin, DGEBA type (Dow D.E.R. 332LC); solvent: CDCl_3

C2

Epoxy Resin, DGEBA Type

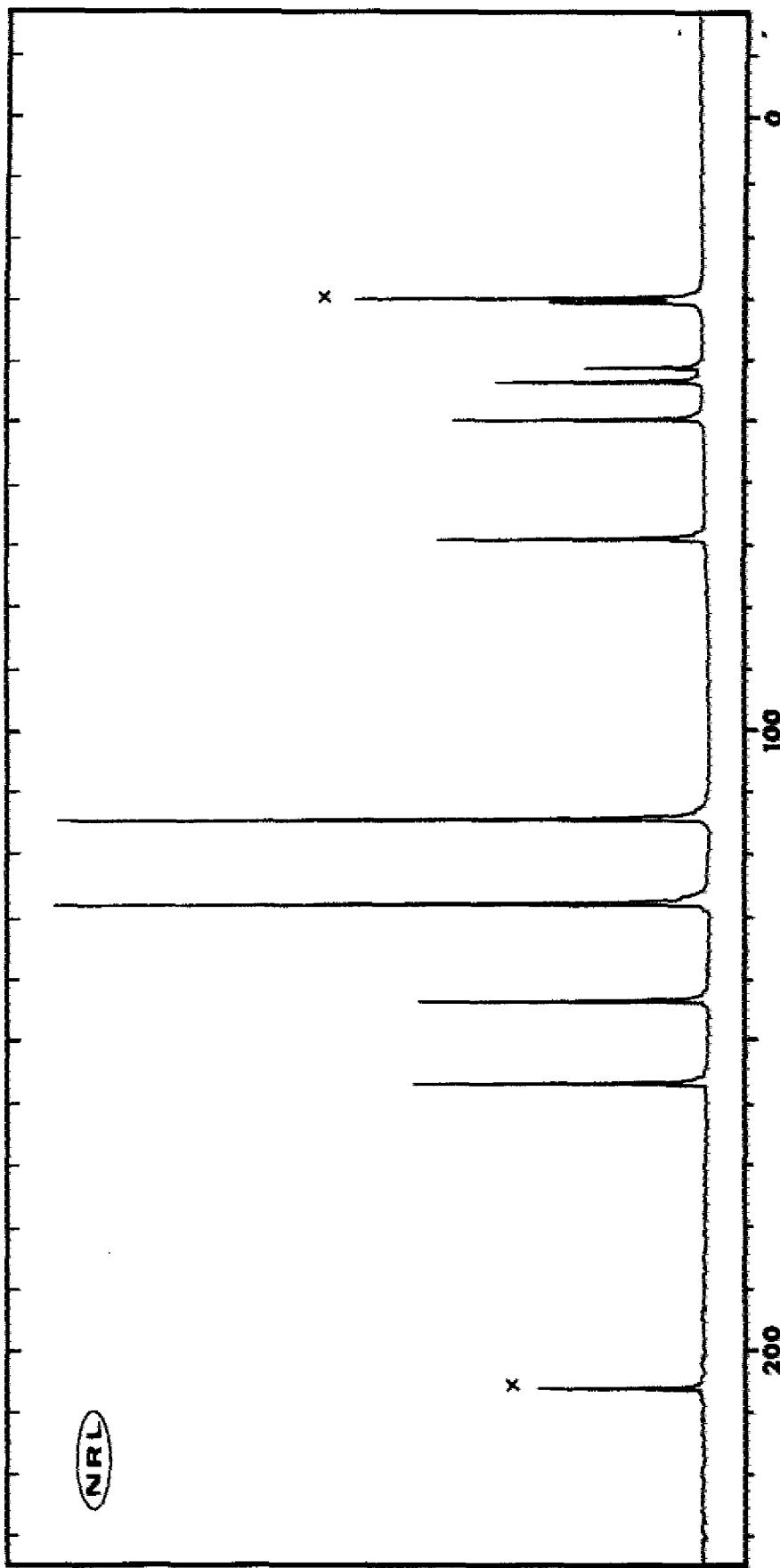
Assignments:

a	30.9	h	143.3
b	41.6	i	156.4
c	43.9		
d	50.0		
e	69.2		
f	114.0		
g	127.7		



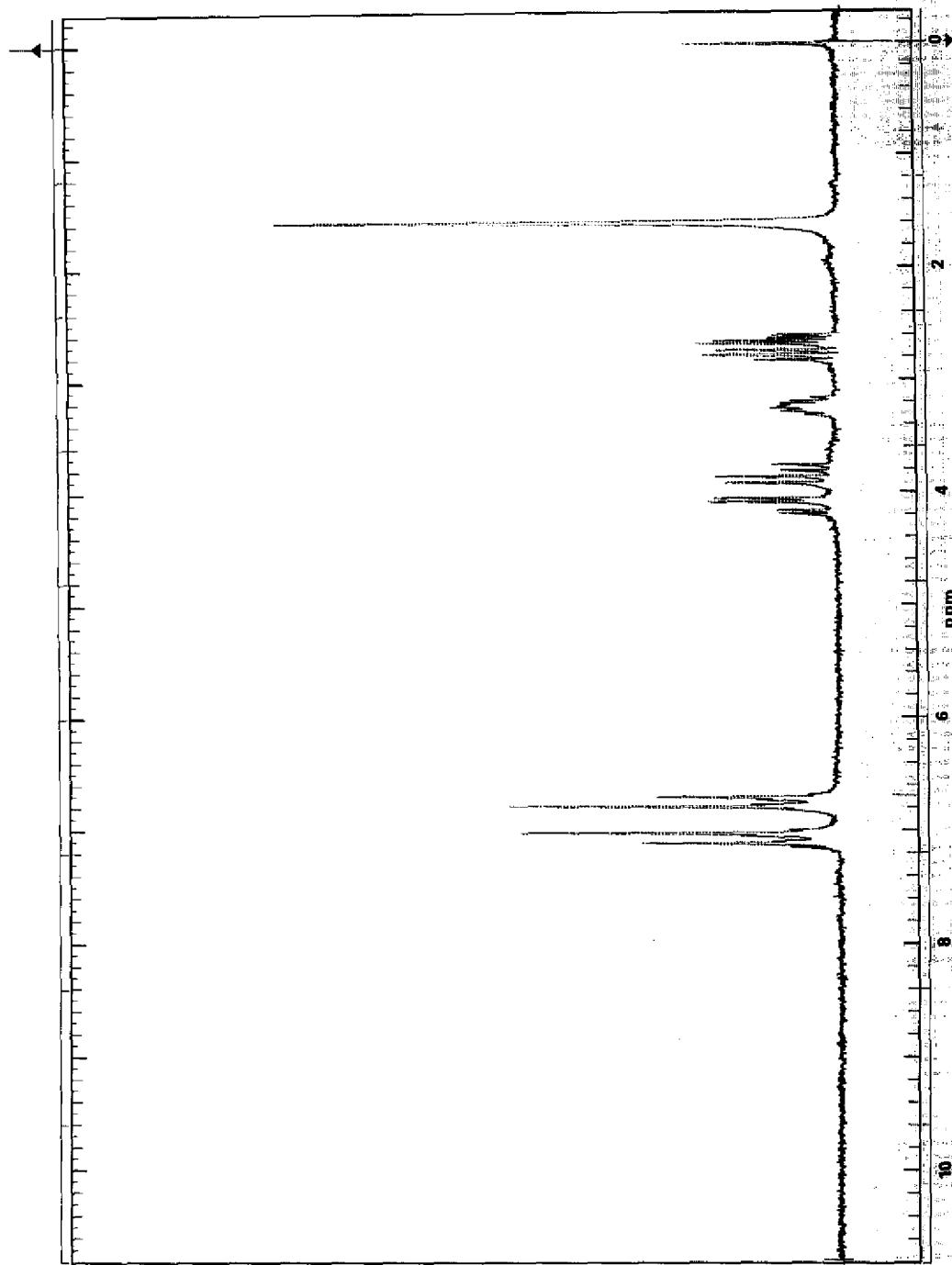
Source: Shell Epon 828

Solvent: 50% Acetone x



NRL

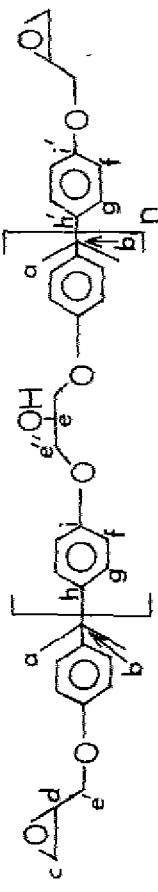
H2



Spectrum 2 — Epoxy resin, DGEBA type (Shell Epon 828); solvent: CDCl_3

C3

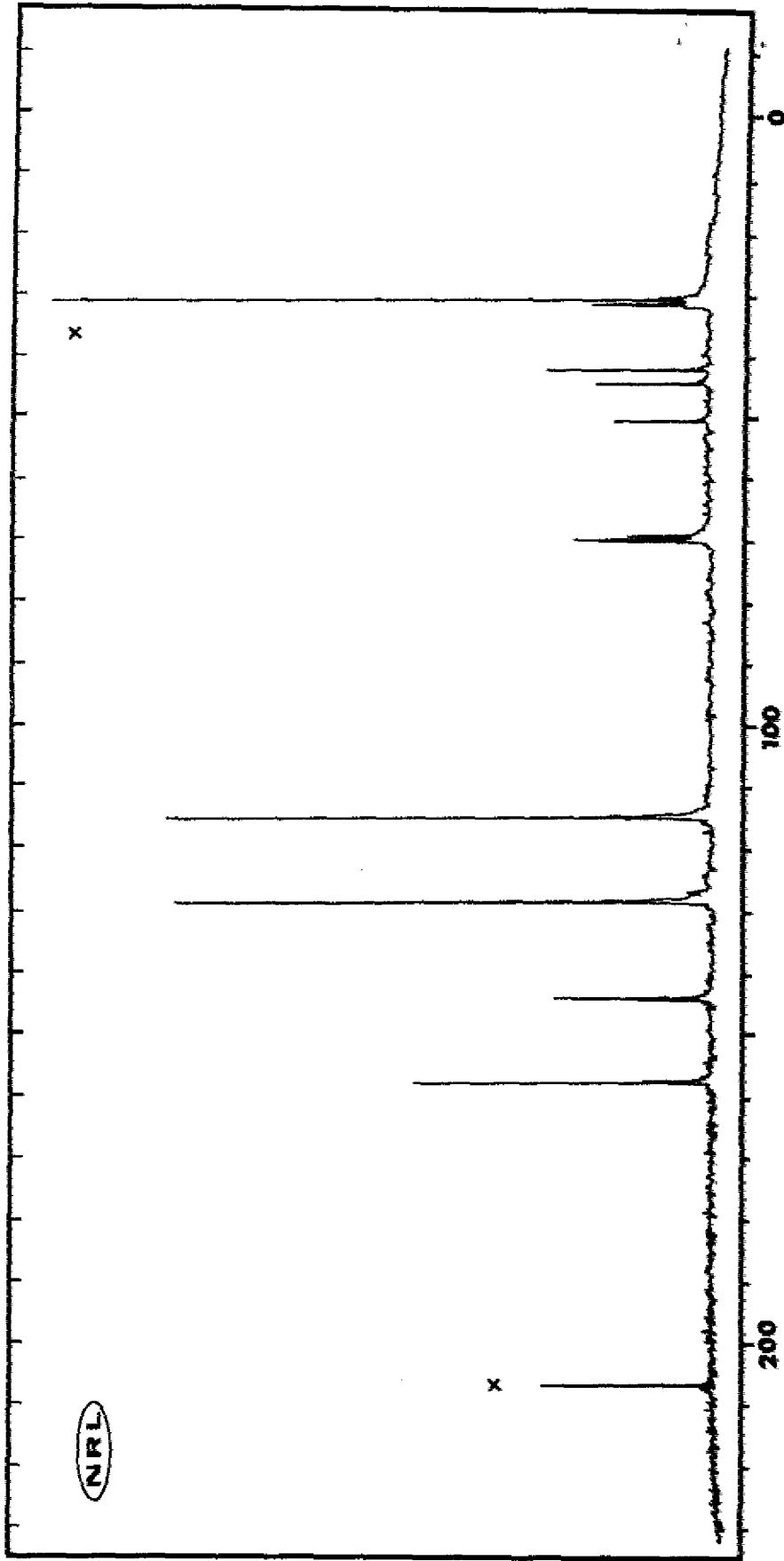
Epoxy Resin, DGEBA Type



Assignments:	
a	30.9
b	41.6
c	44.0
d	50.1
e	68.7
e'	69.3
f	114.1
g	127.7
h	143.2
h'	143.4
i	156.6
i'	156.8

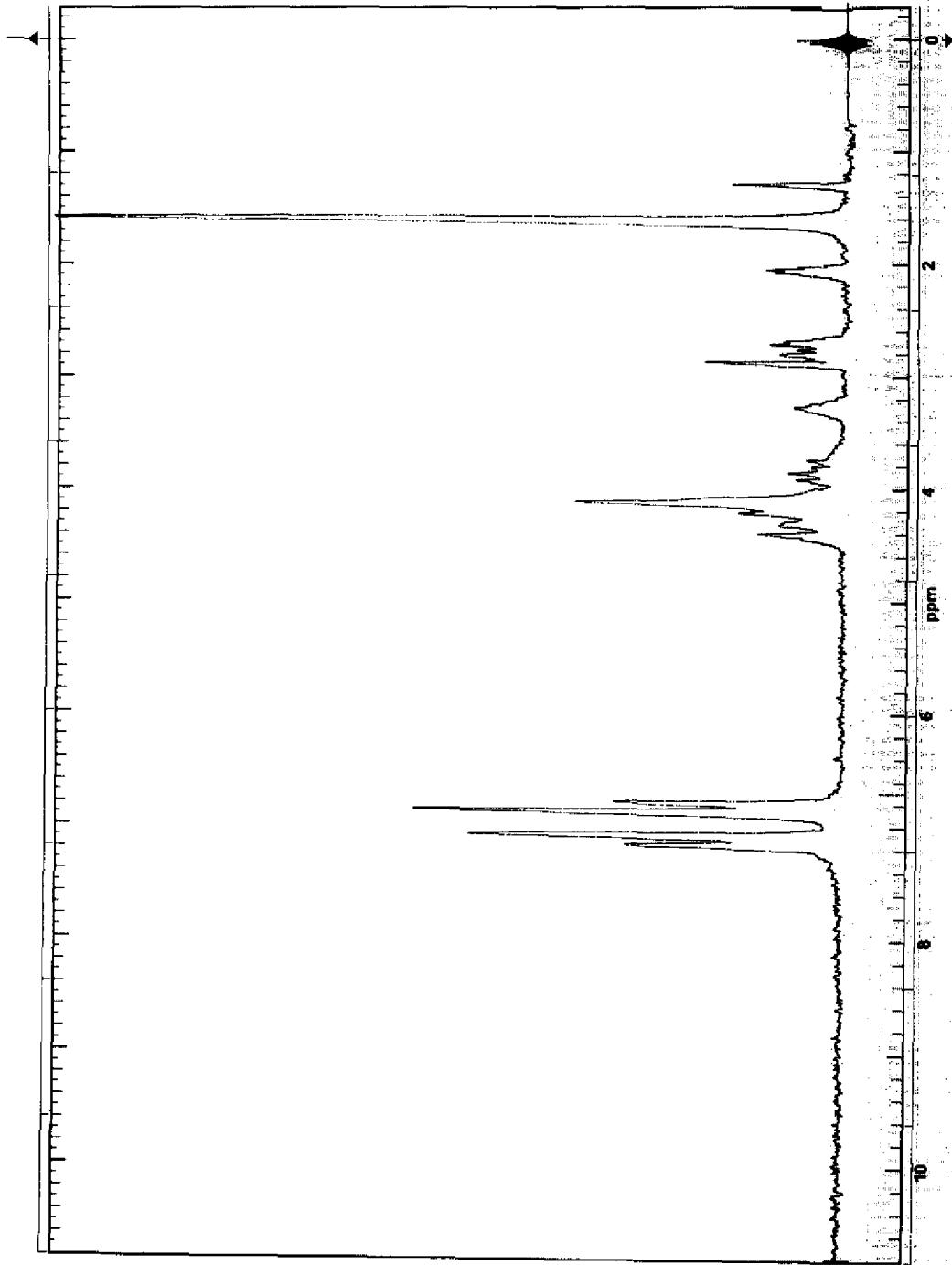
Source: Ciba Araldite 7071

Solvent: 50% Acetone X



NRL

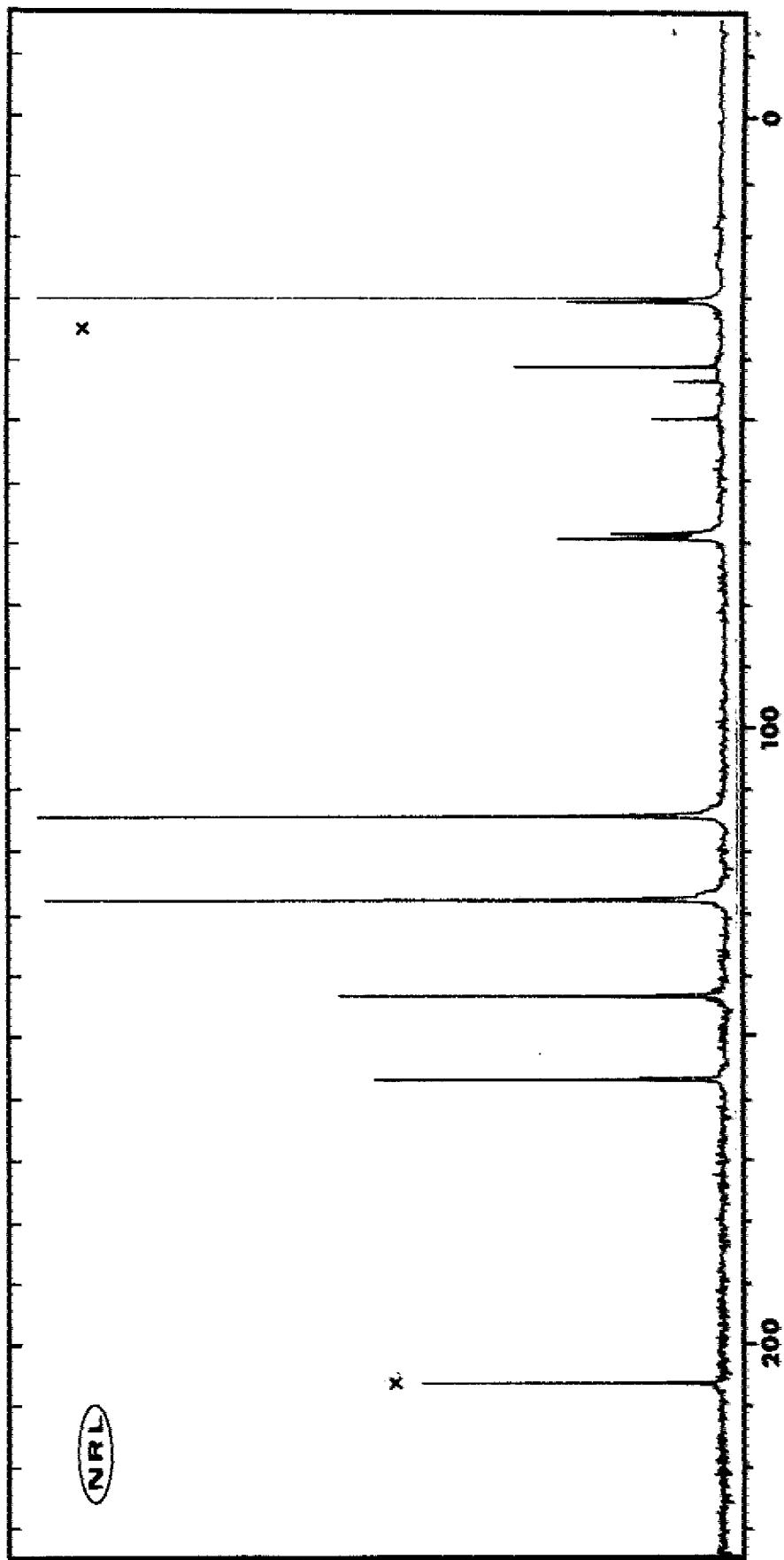
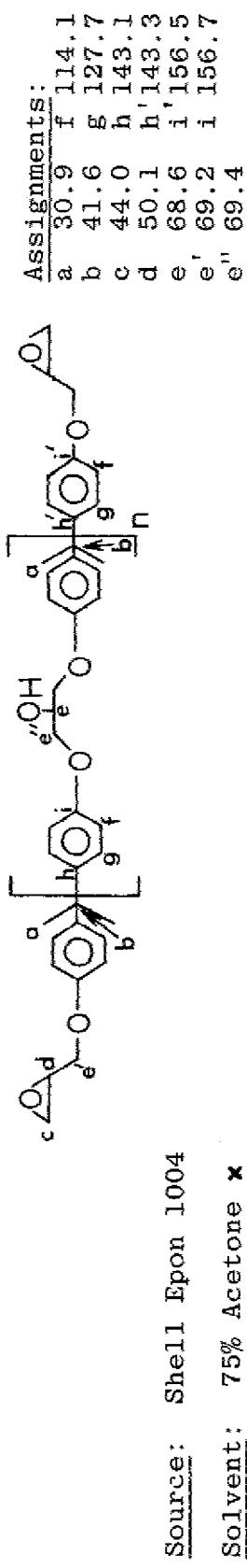
H3



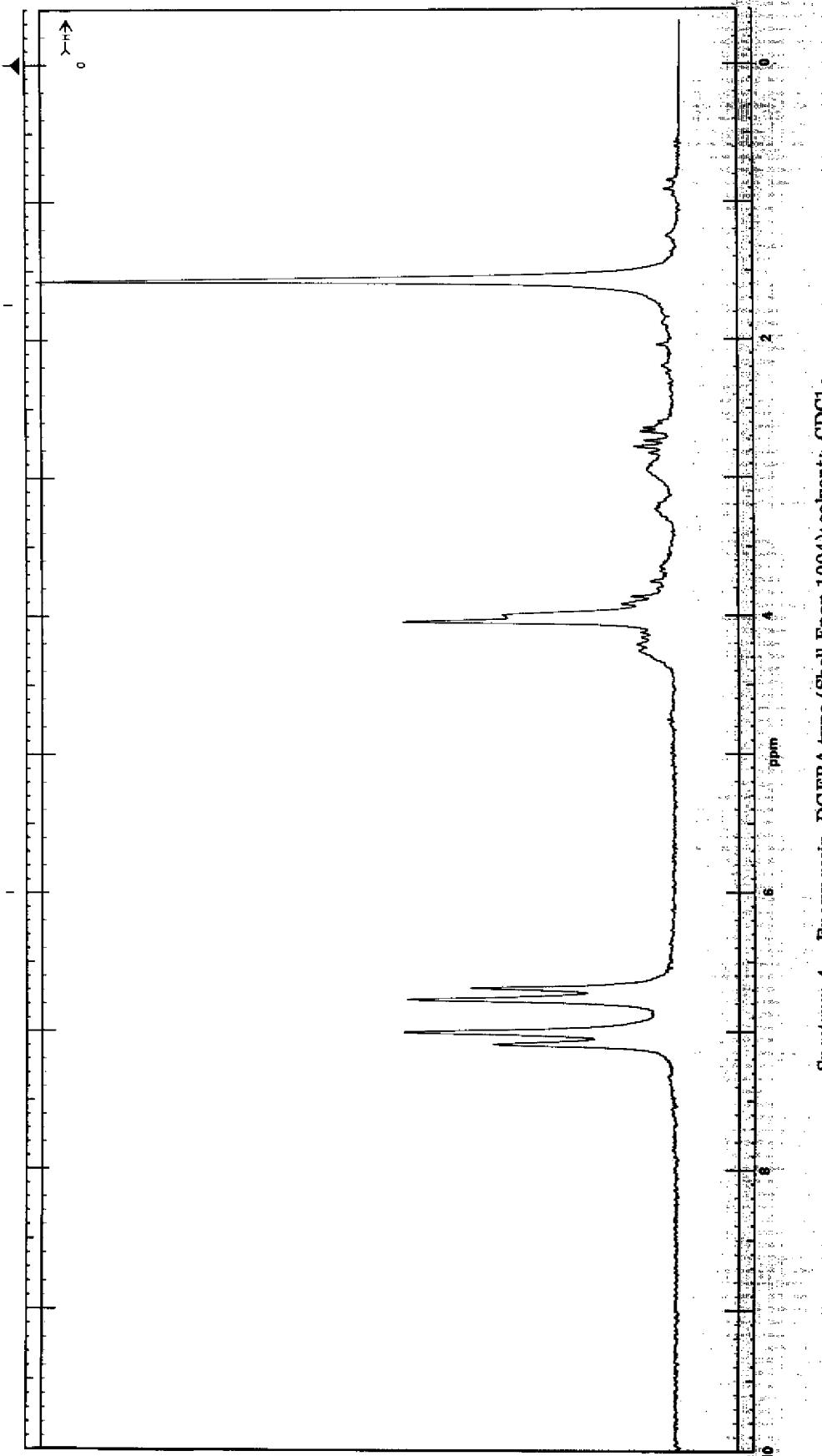
Spectrum 3 – Epoxy resin, DGEBIA type (Ciba Araldite 7071); solvent: acetone-d₆

C4

Epoxy Resin, DGEBA Type



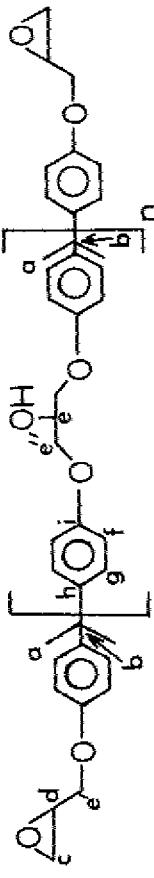
H4



Spectrum 4 — Epoxy resin, DGEBA type (Shell Epon 1004); solvent: CDCl_3

C5

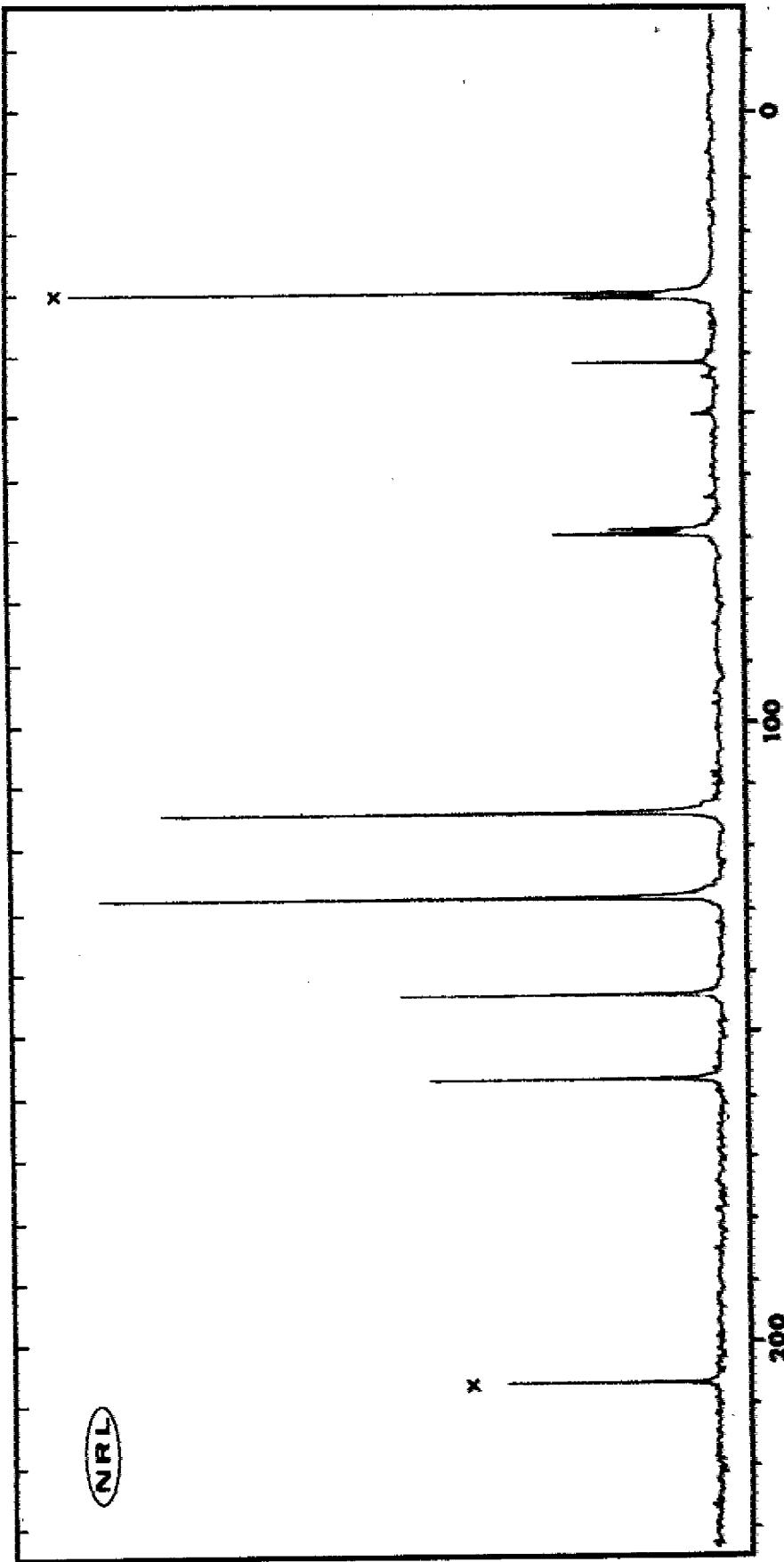
Epoxy Resin, DGEBA Type



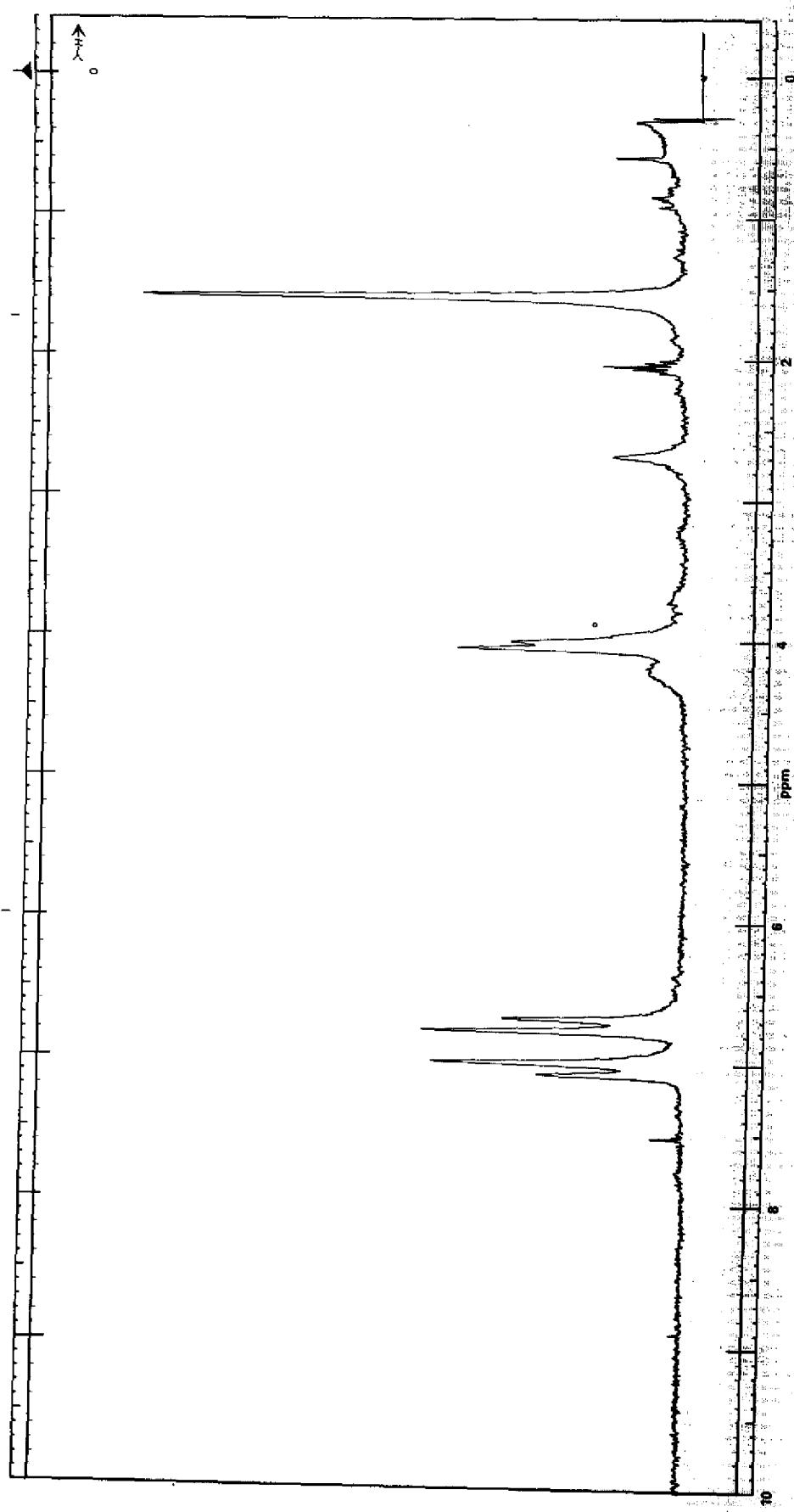
Assignments:	
a	31.0
b	41.6
c	50.0
d	68.7
e'	69.5
e''	114.1
f	128.2
g	143.2
h	156.7

Source: Shell Epon 1007

Solvent: 75% Acetone *



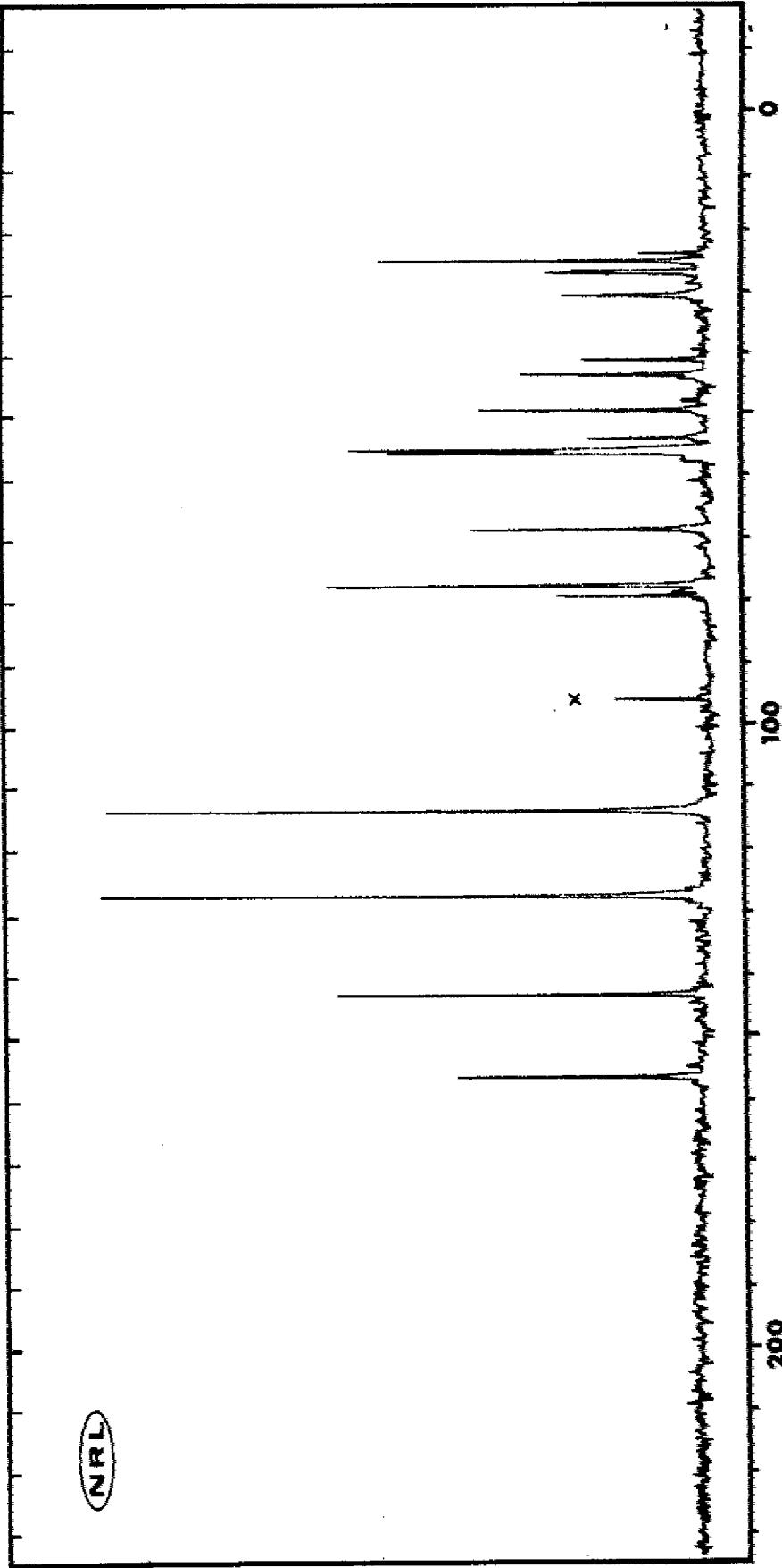
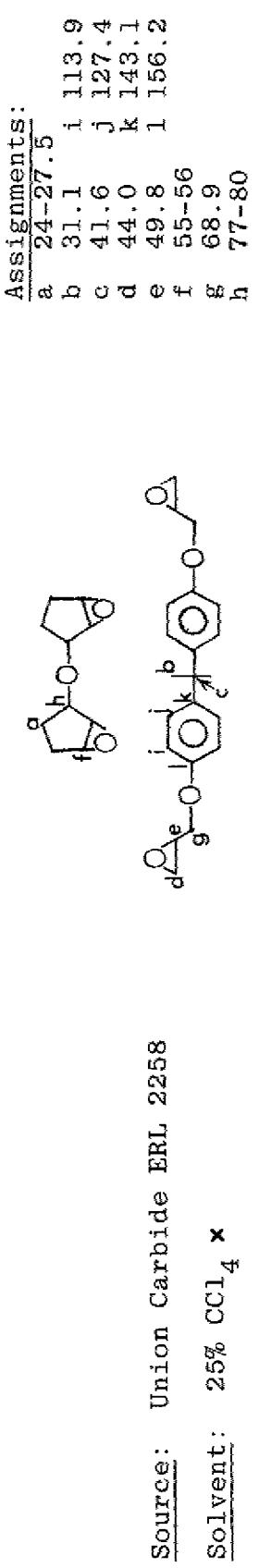
H5



Spectrum 5 — Epoxy resin, DGEBA type (Shell Epon 1007); solvent: CDCl_3 and acetone- d_6

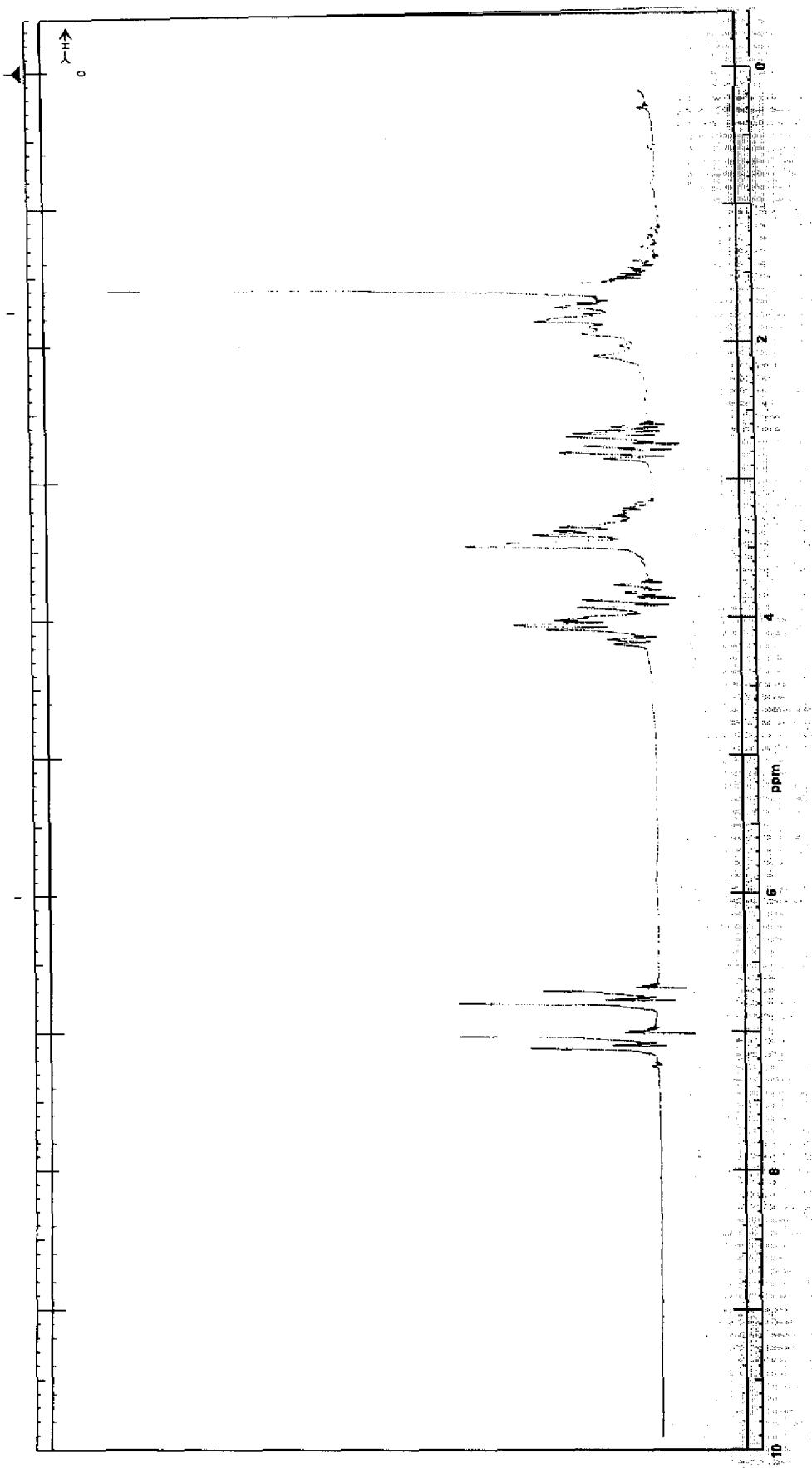
C6

DGEBA Type Epoxy Resin plus Bis(2,3-epoxycyclopentyl)ether



NRL

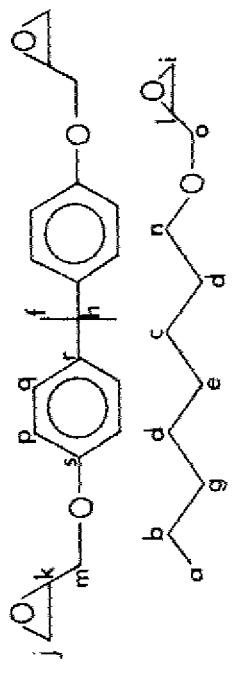
H6



Spectrum 6 — DGEBA type epoxy resin plus bis(2,3-epoxycyclopentyl)ether (Union Carbide ERL 2258); solvent: CDCl_3

C7

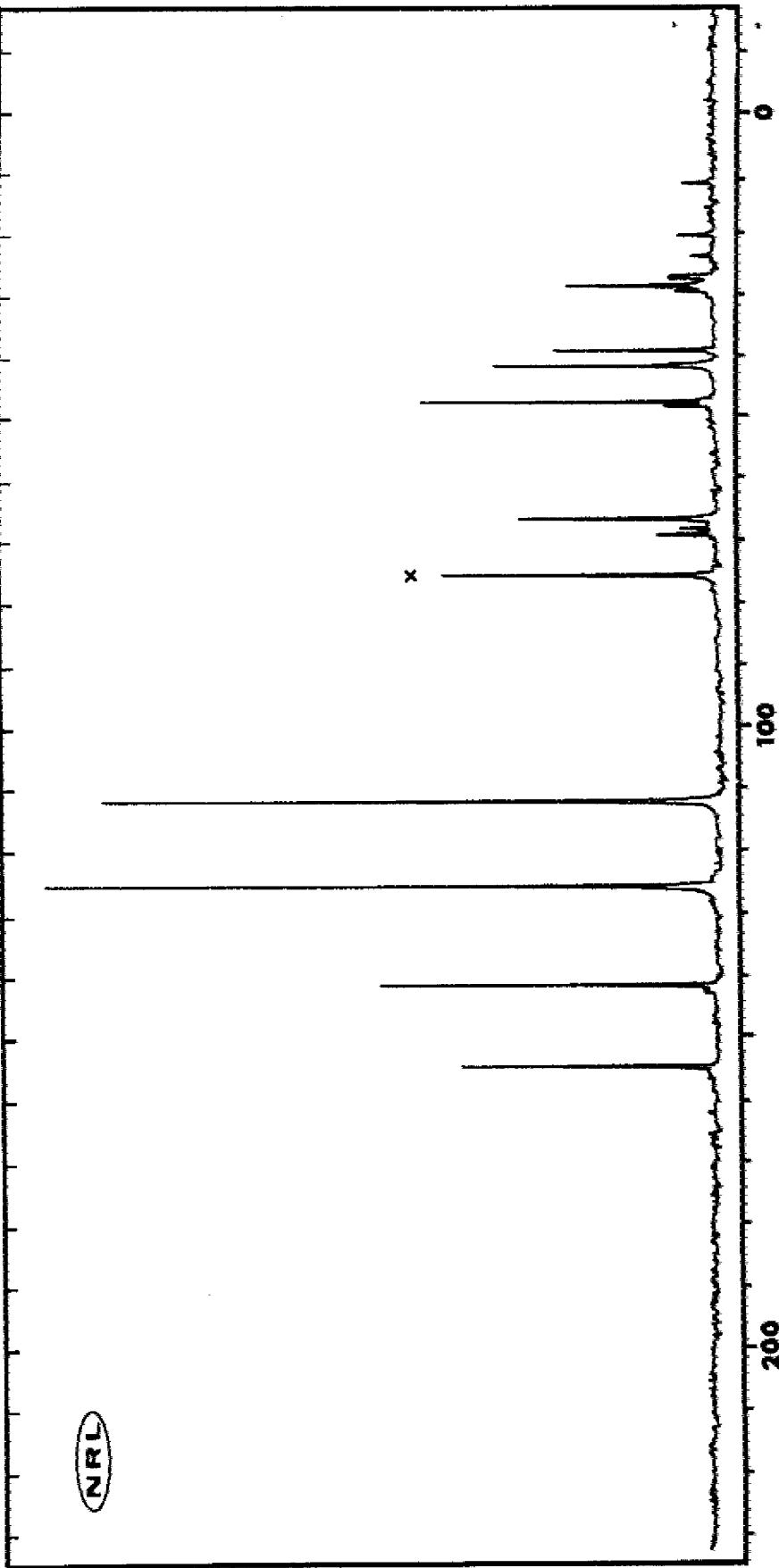
DGEBA Type Epoxy Resin plus n-Octyl Glycidyl Ether



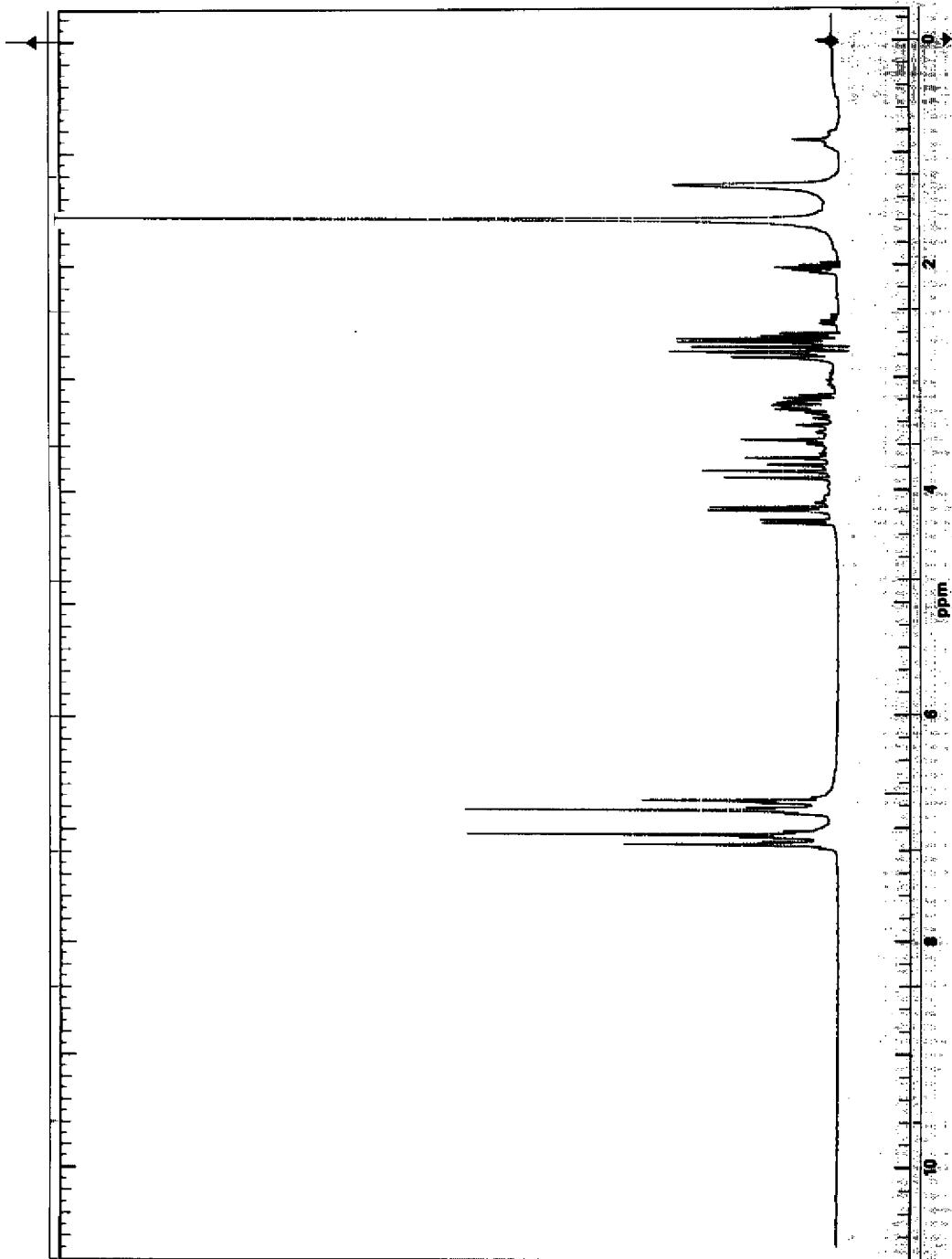
Assignments:	
a	13.5
b	22.0
c	25.4
d	28.7
e	29.0
f	30.3
g	31.2
h	40.9
i	43.1
j	43.5
k	49.3
l	50.1
m	68.2
n	69.8
o	70.8
p	113.3
q	126.8
r	142.5
s	155.6

Source: Genepoxy M195

Solvent: 25% CHCl₃ *



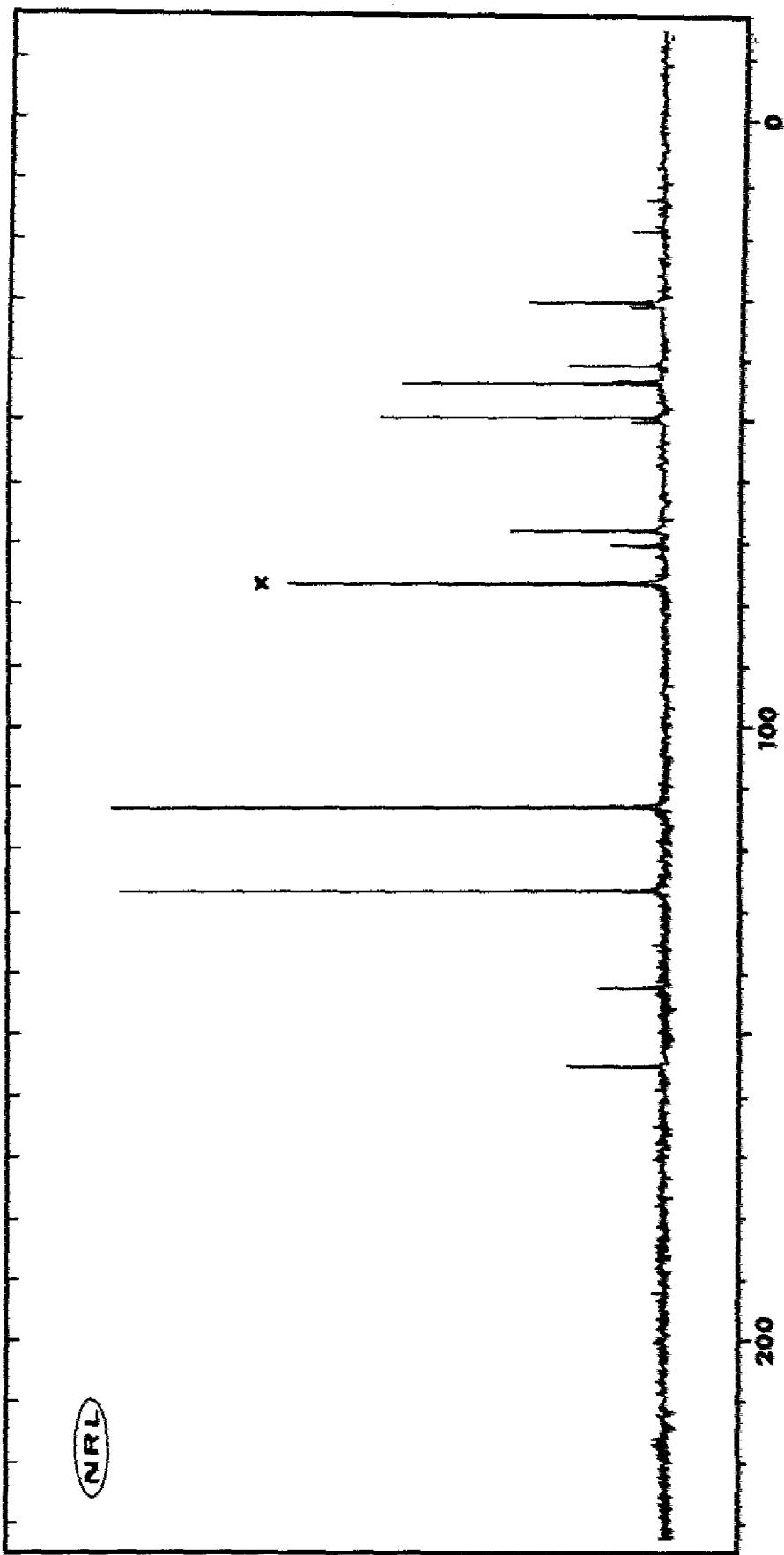
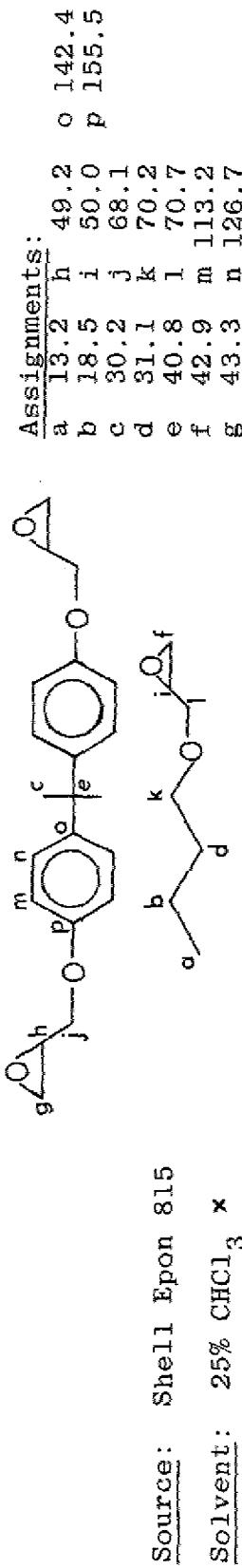
H7



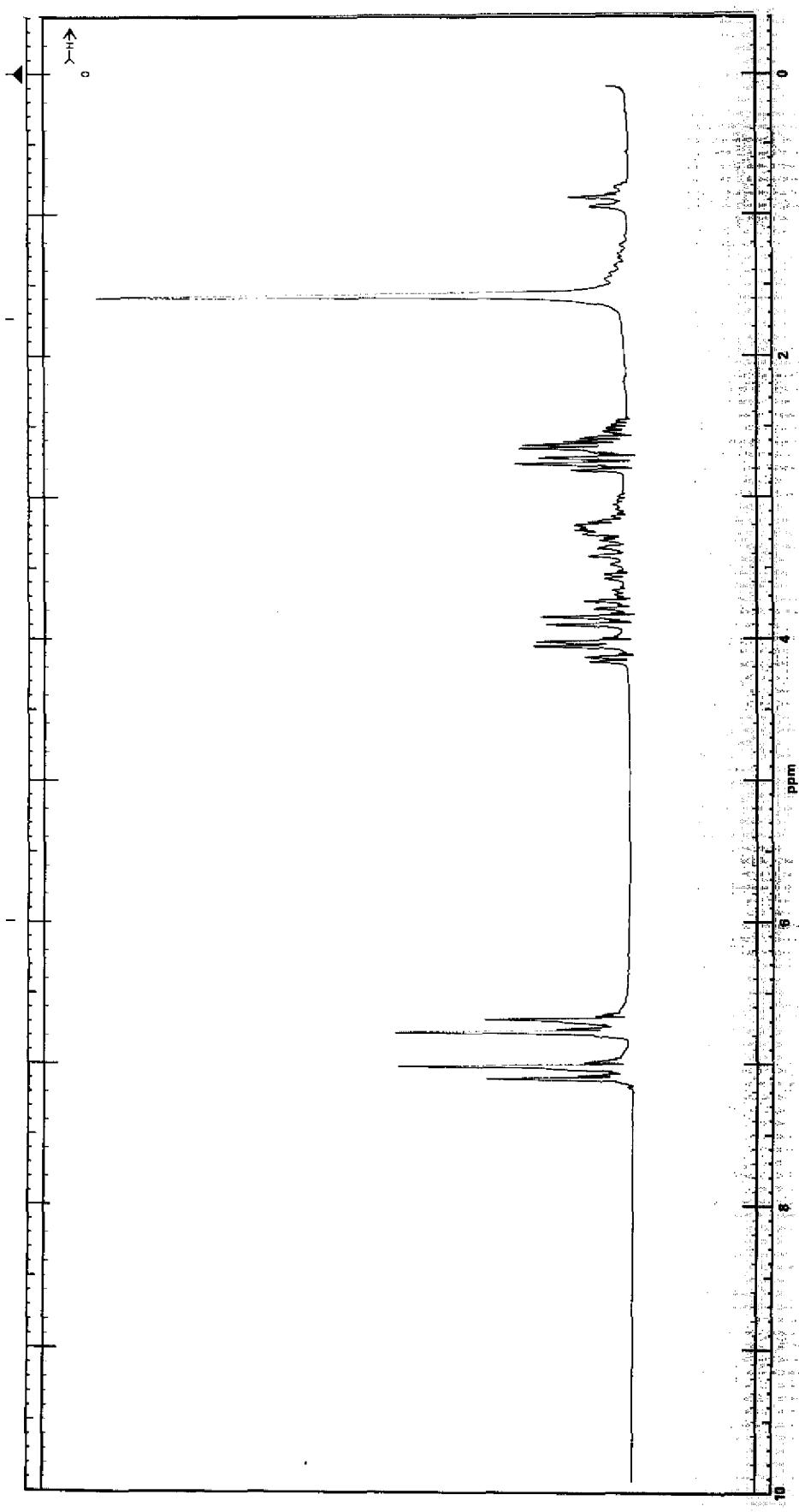
Spectrum 7 — DGEBA type epoxy resin plus *n*-octyl glycidyl ether (Genepoxy M195); solvent: acetone-d₆

C8

DGEBA Type Epoxy Resin plus Butyl Glycidyl Ether



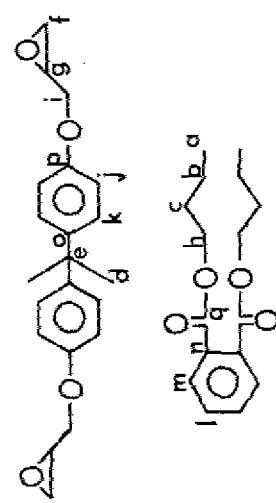
H8



Spectrum 8 – DGEBA type epoxy resin plus butyl glycidyl ether (Shell Epon 815); solvent: CDCl_3

C9

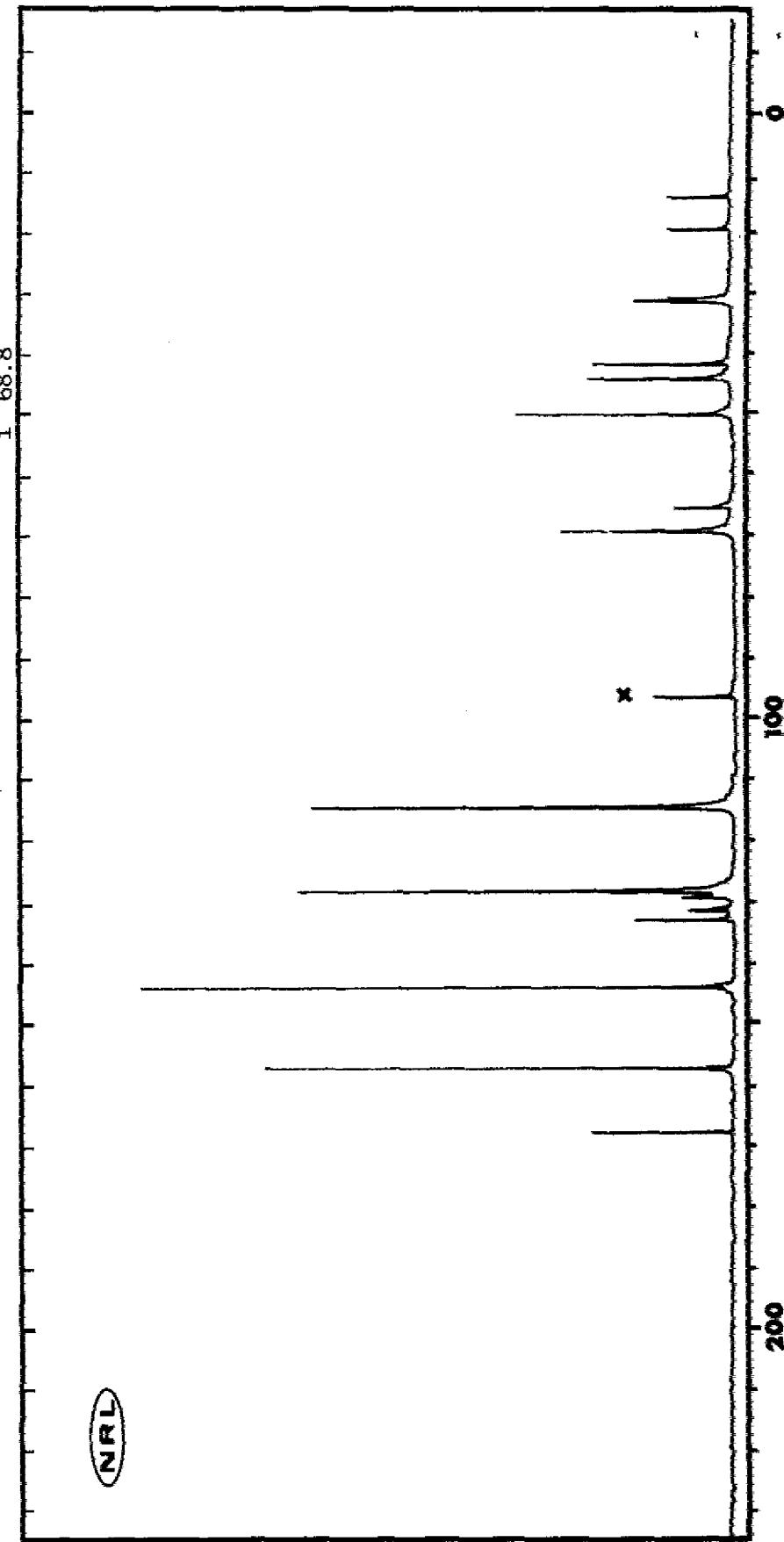
DGEBA Type Epoxy Resin plus Di-n-butyl Phthalate



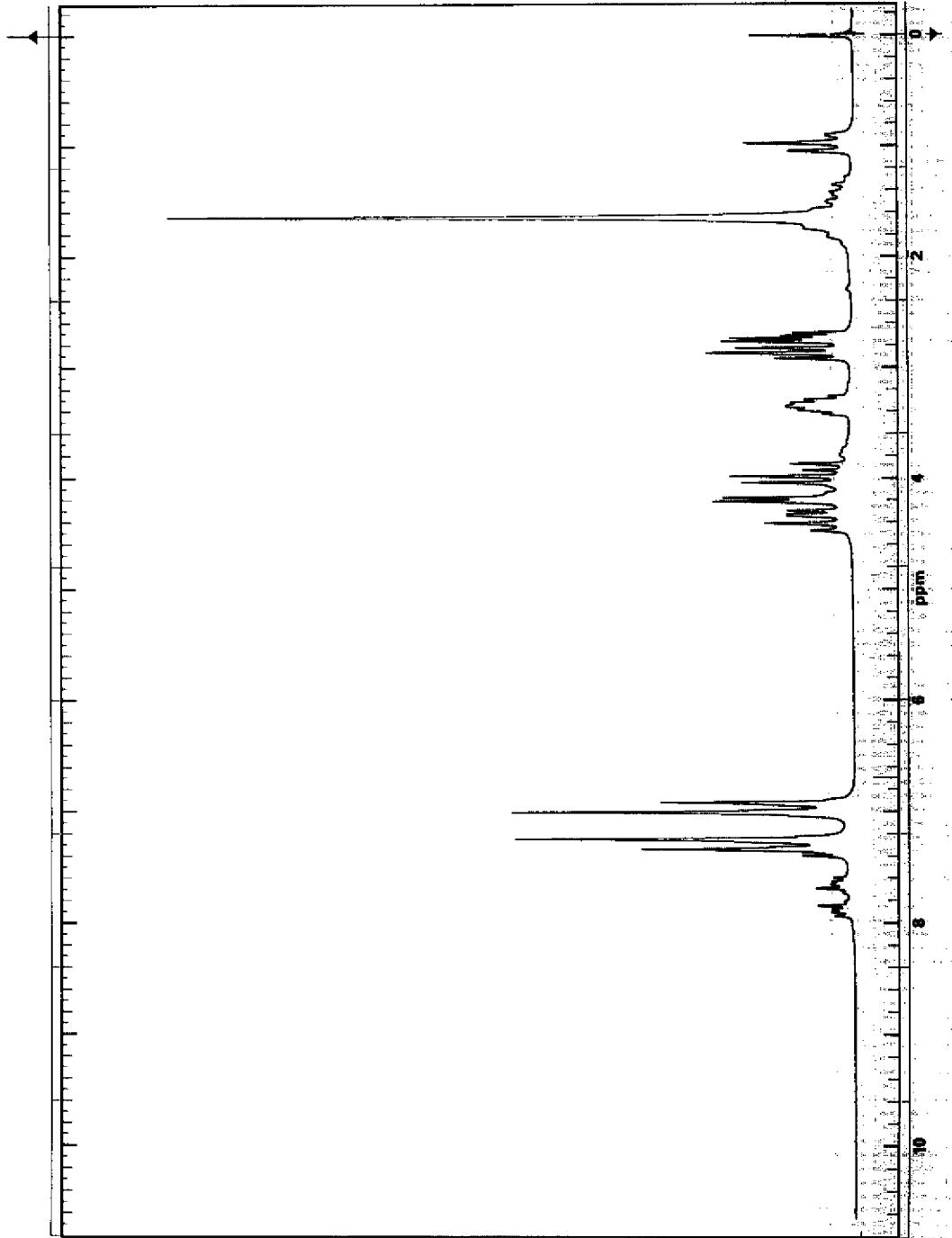
Assignments:	
a	13.8
b	19.2
c	30.6
d	31.0
e	41.5
f	44.0
g	49.8
h	65.0
i	68.8
j	113.9
k	127.4
l	128.5
m	130.7
n	132.2
o	143.1
p	156.2
q	166.8

Source: Ciba Araldite 502

Solvent: 20% CCl_4 *



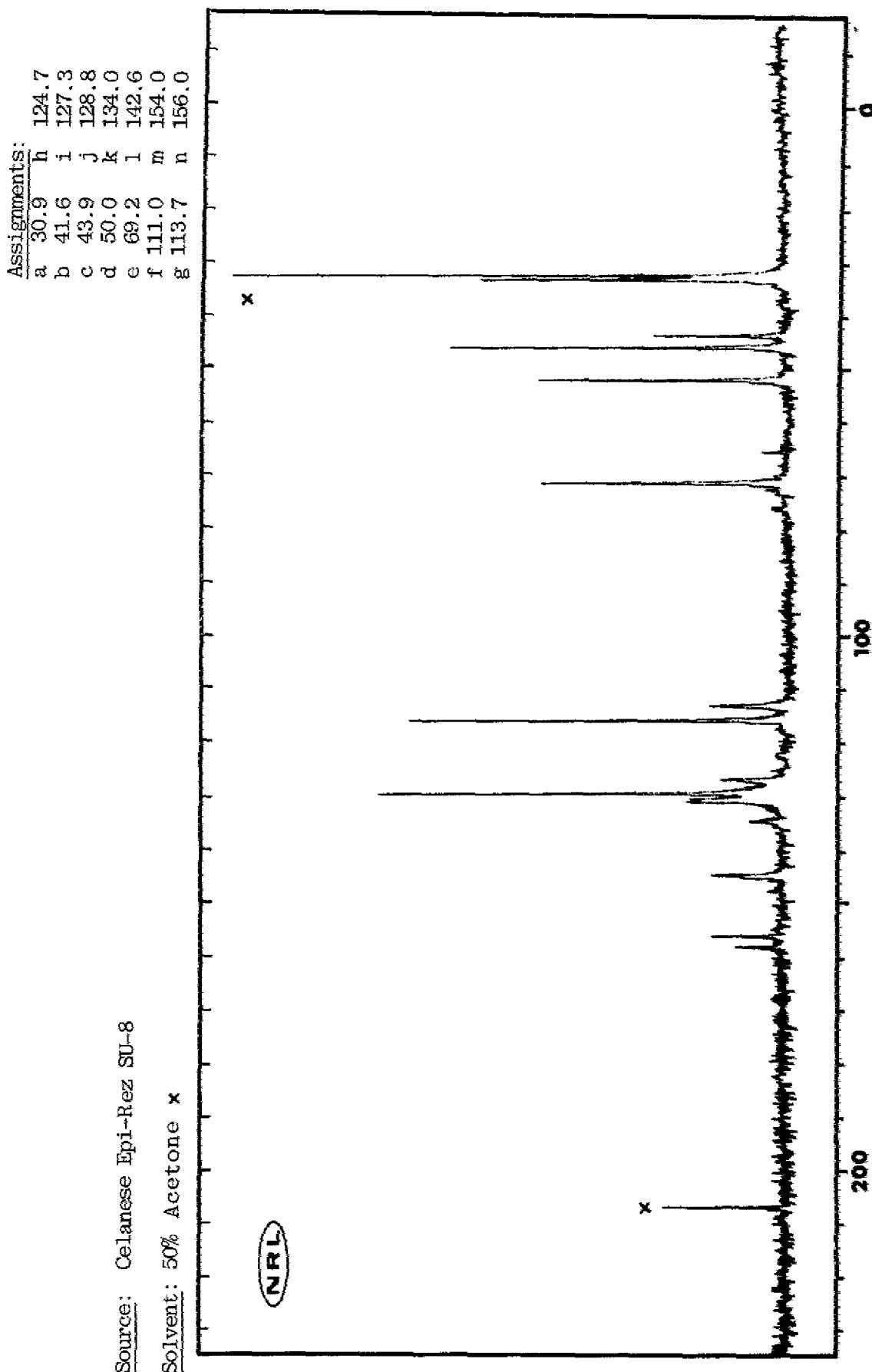
H9



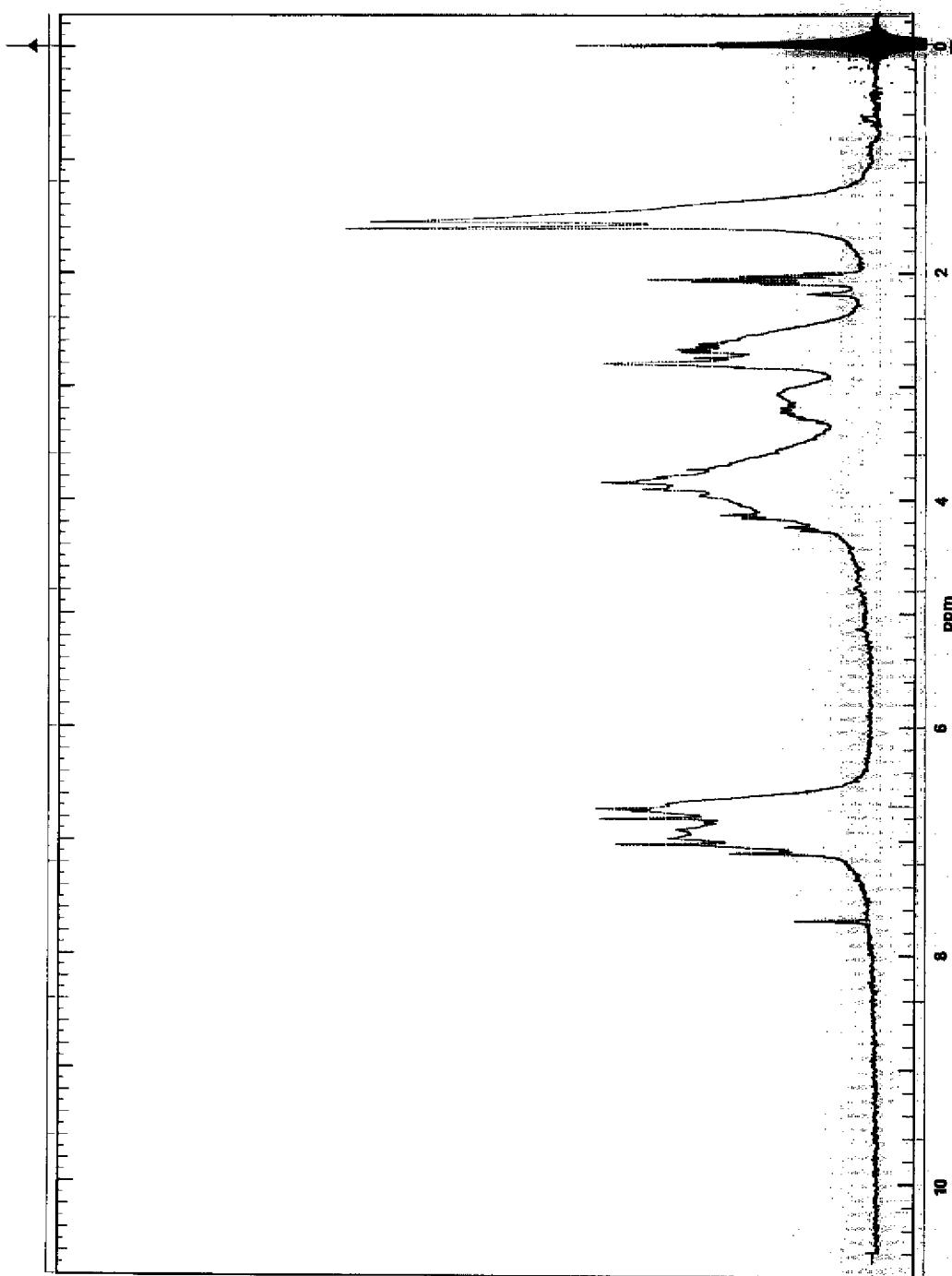
Spectrum 9 — DGEBBA type epoxy resin plus di-n-butyl phthalate (Ciba Araldite 502); solvent: CDCl₃

C10

Epoxy Resin, Polyfunctional



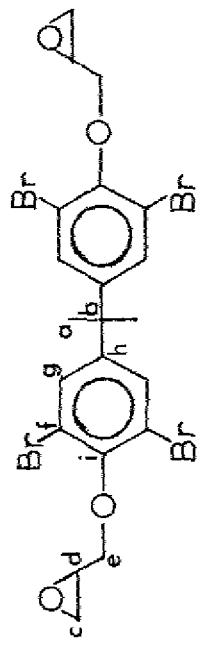
H10



Spectrum 10 — Epoxy resin, polyfunctional (Celanese Epi-Rez SU-8); solvent: acetone-d₆ and CDCl₃ (1:1)

G11

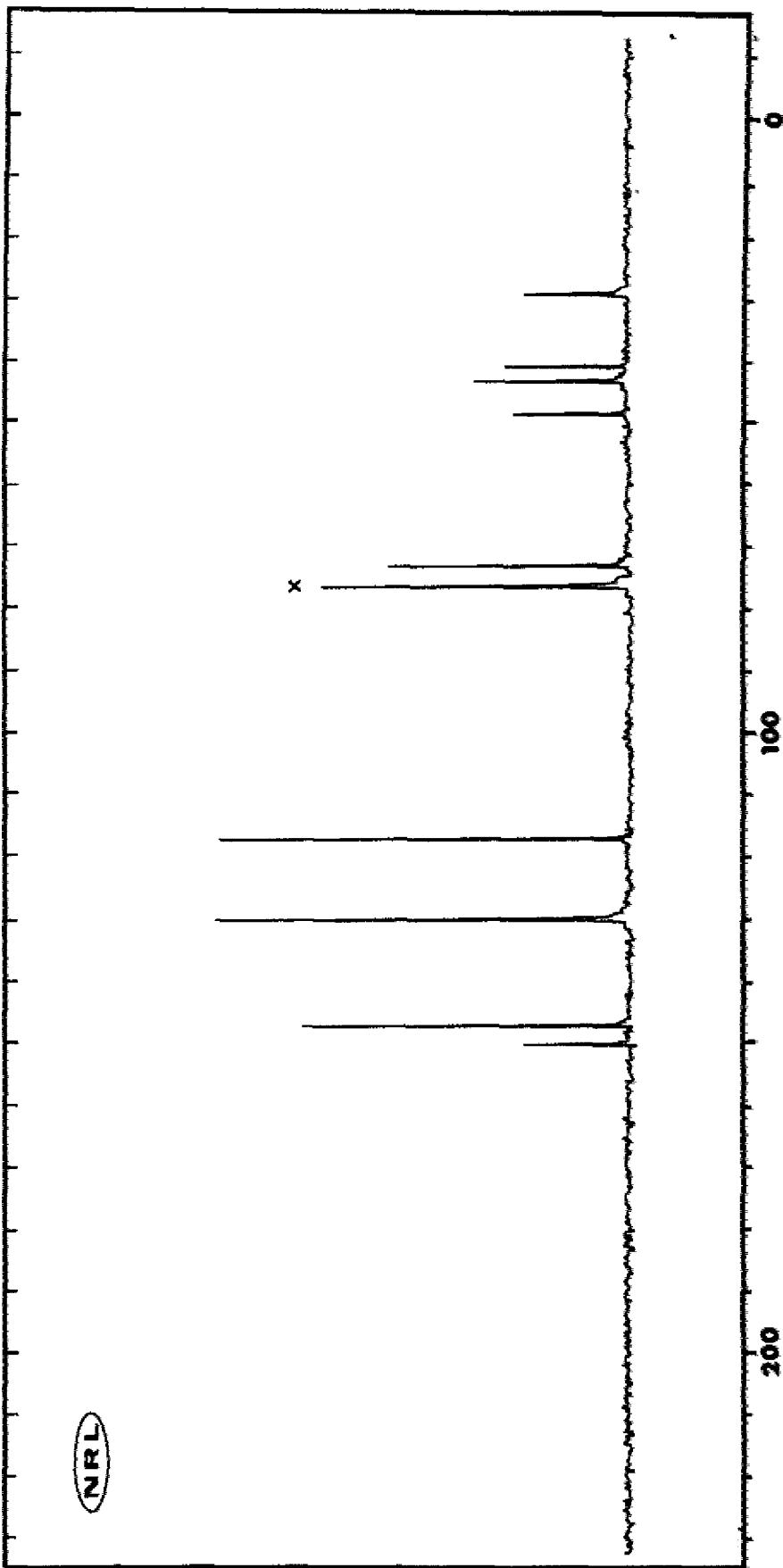
Diglycidyl Ether of Tetrabromobisphenol A



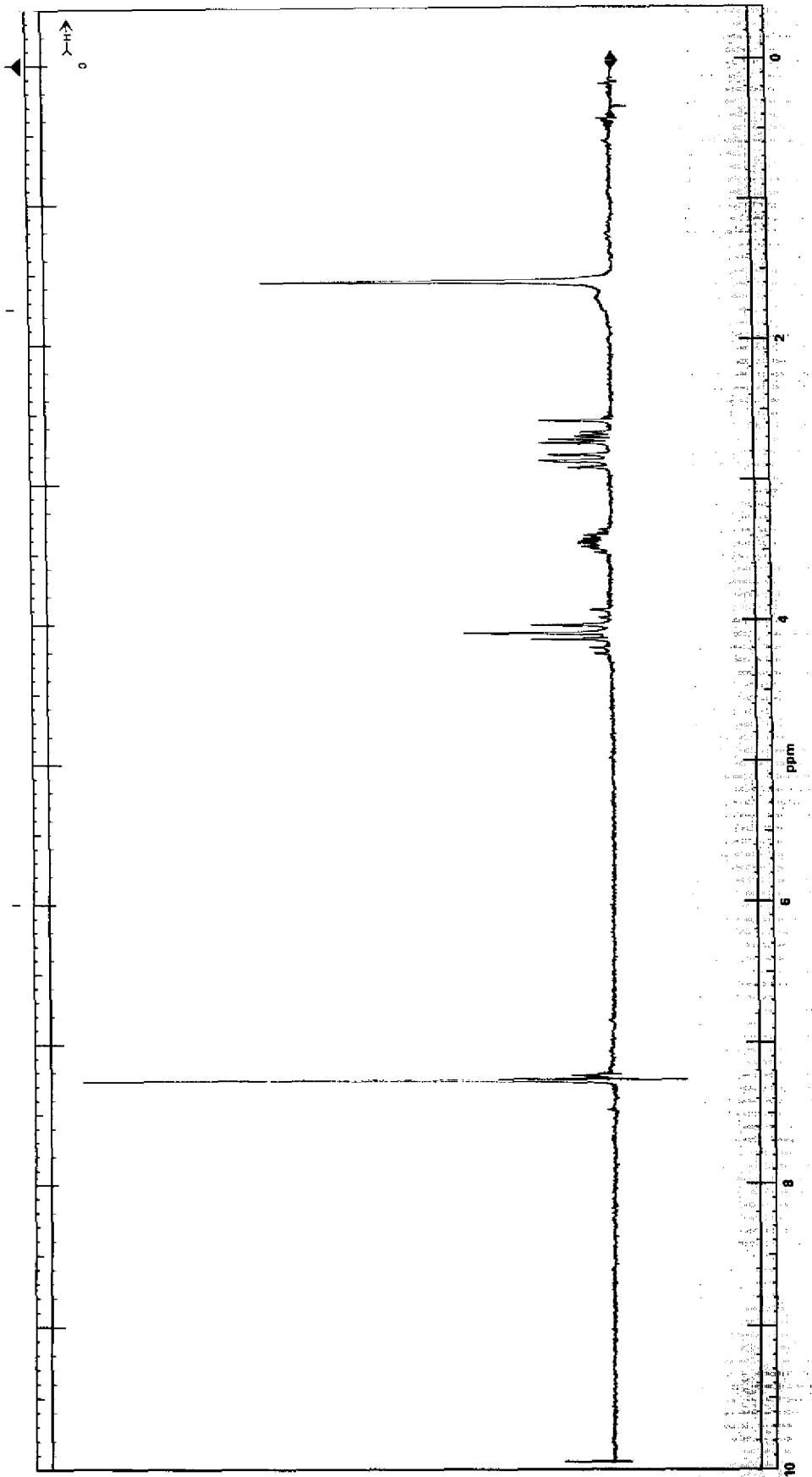
Assignments:		
a	30.0	h 147.3
b	41.8	i 150.4
c	44.2	
d	49.6	
e	73.8	
f	117.5	
g	130.4	

Source: Dow D.E.R. 542

Solvent: 50% CHCl₃ x



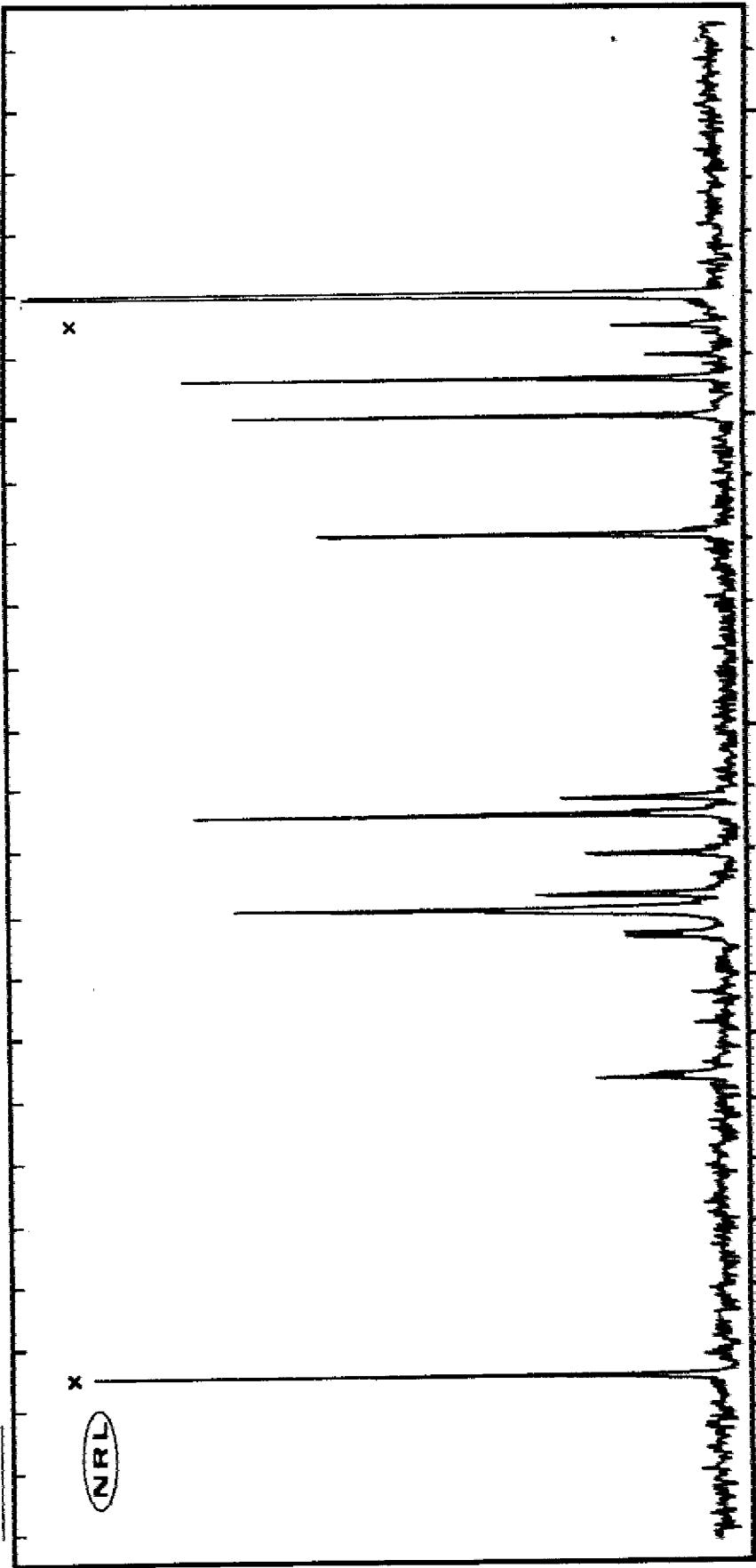
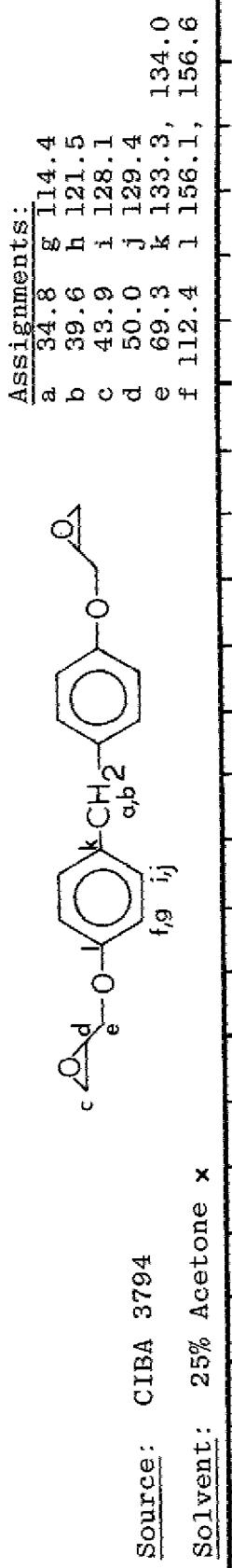
H11



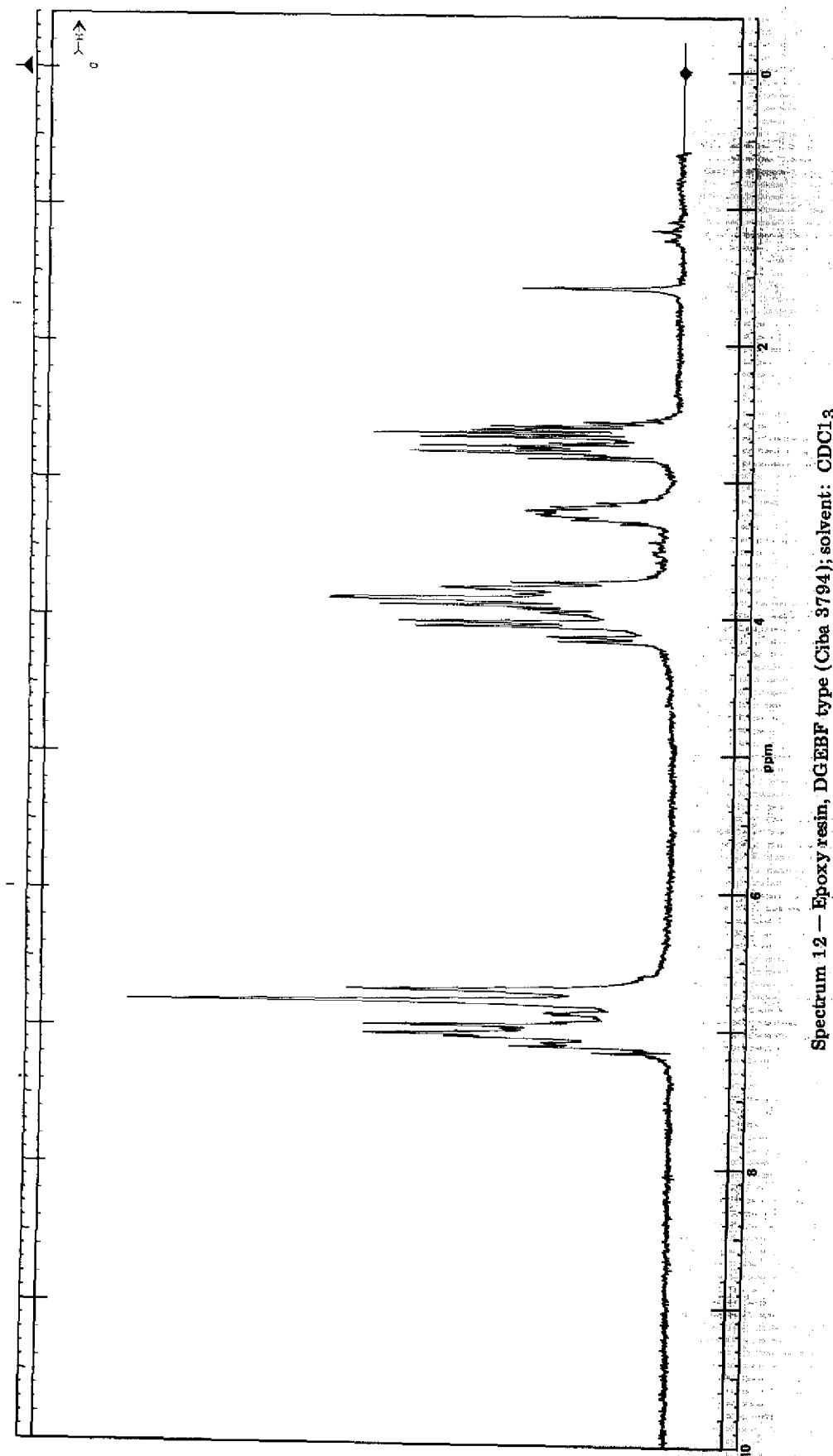
Spectrum 11 – Diglycidyl ether of tetrabromo-o-phenol A (D.E.R. 542); solvent: CDCl₃

C12

Epoxy Resin, DGEBF Type



H12

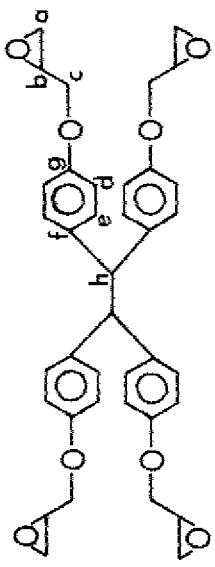


Spectrum 12 — Epoxy resin, DGEBA type (Ciba 3794); solvent: CDCl_3

C13

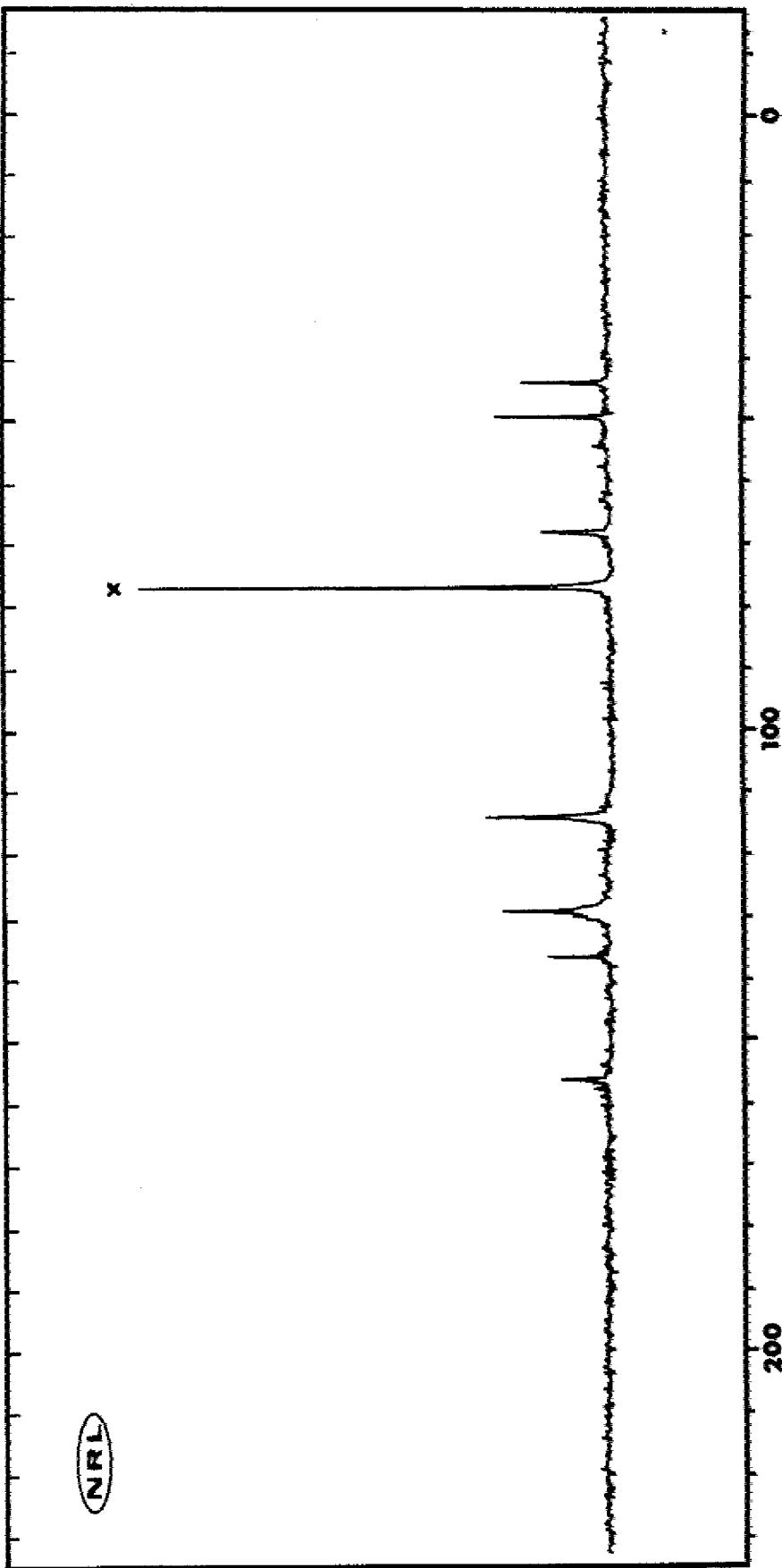
Polyglycidyl Ether of Tetraphenylene Ethane

Assignments:



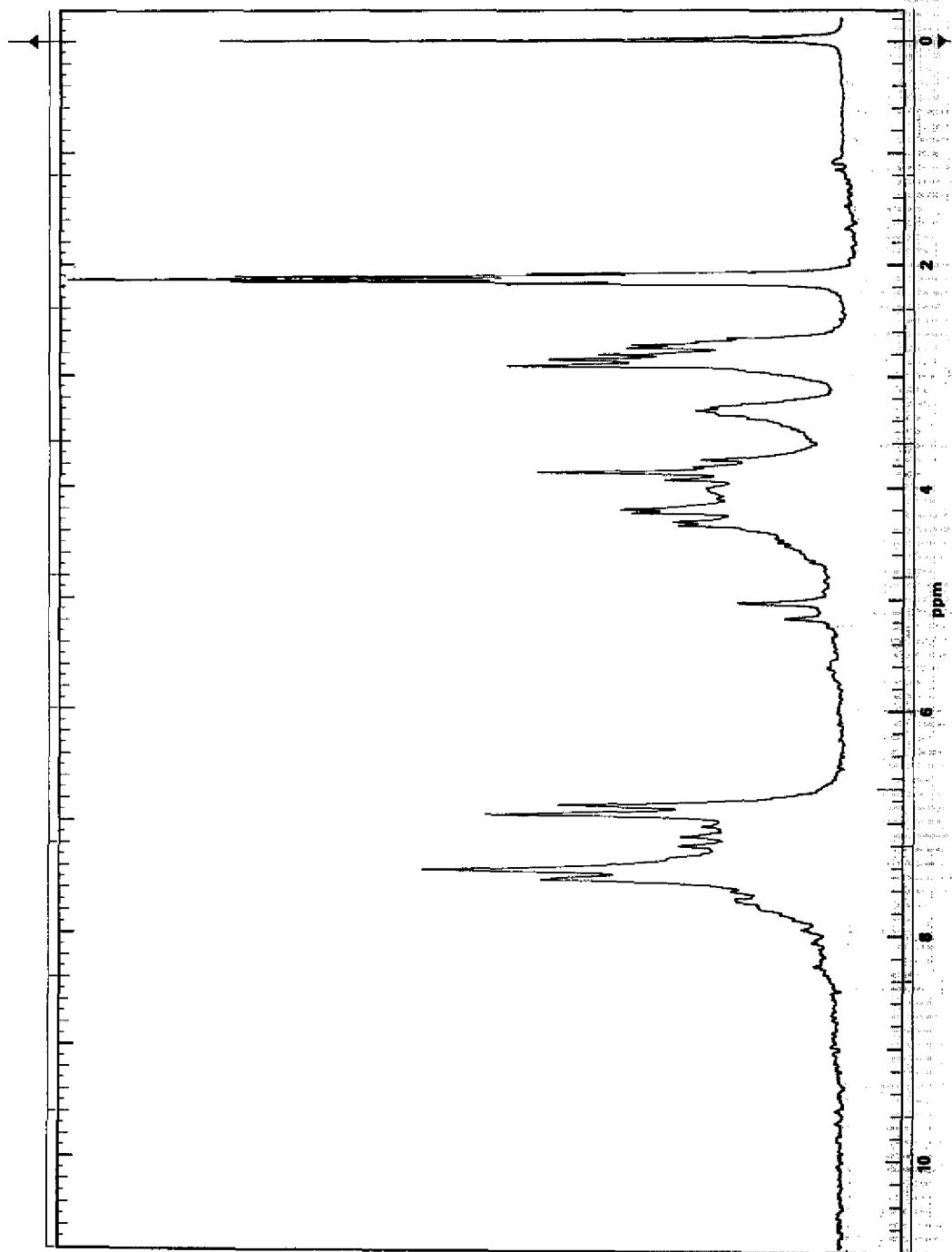
Source: Shell Epon 1031

Solvent: 75% CHCl_3 *



NRL

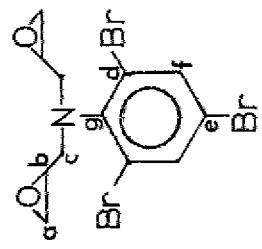
H13



Spectrum 13 — Polyglycidyl ether of tetraphenylene ethane (Shell Epon 1031) solvent: acetone-d₆

C14

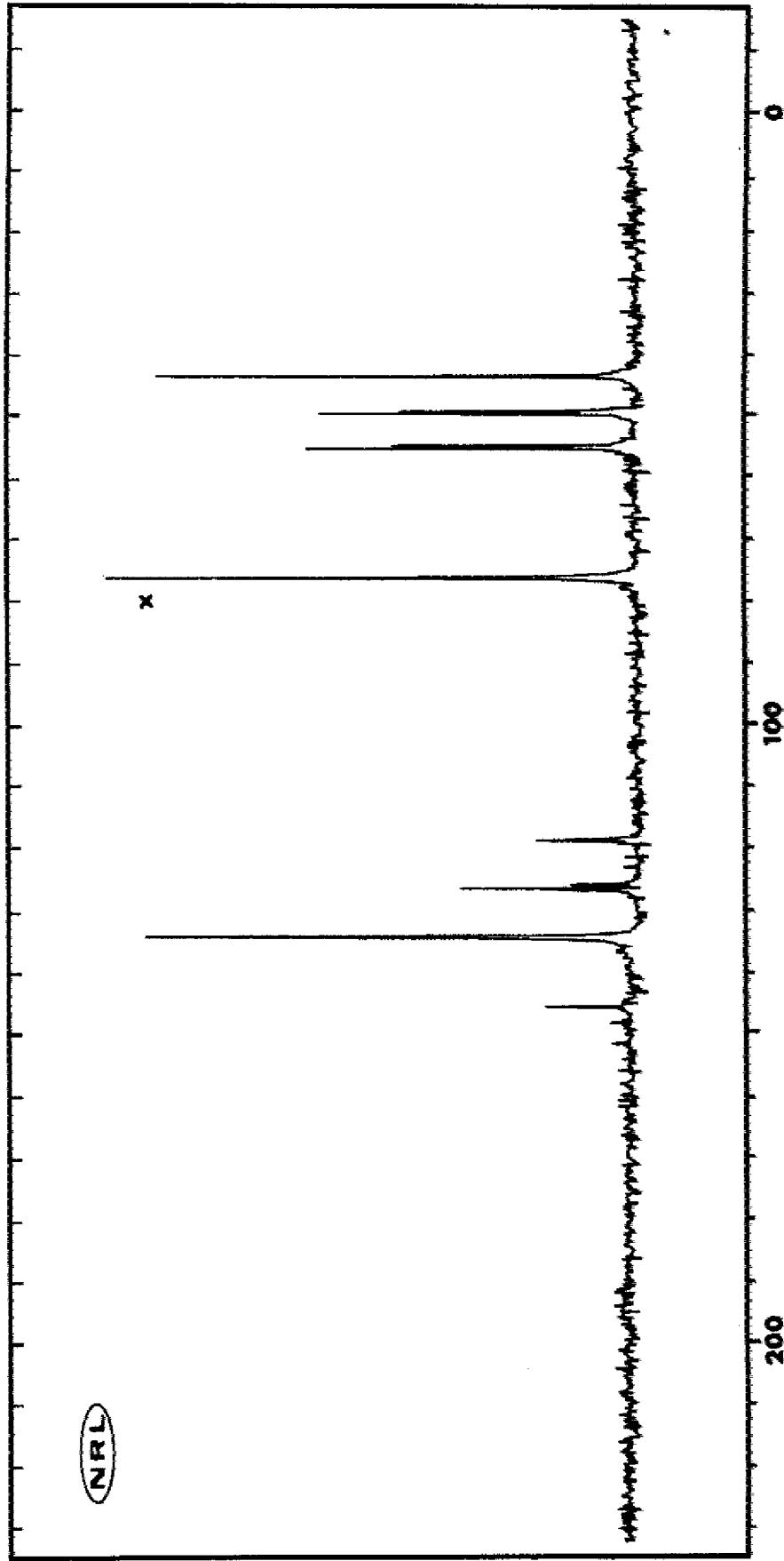
N,N-bis(2,3-epoxypropyl)-2,4,6-tribromoaniline



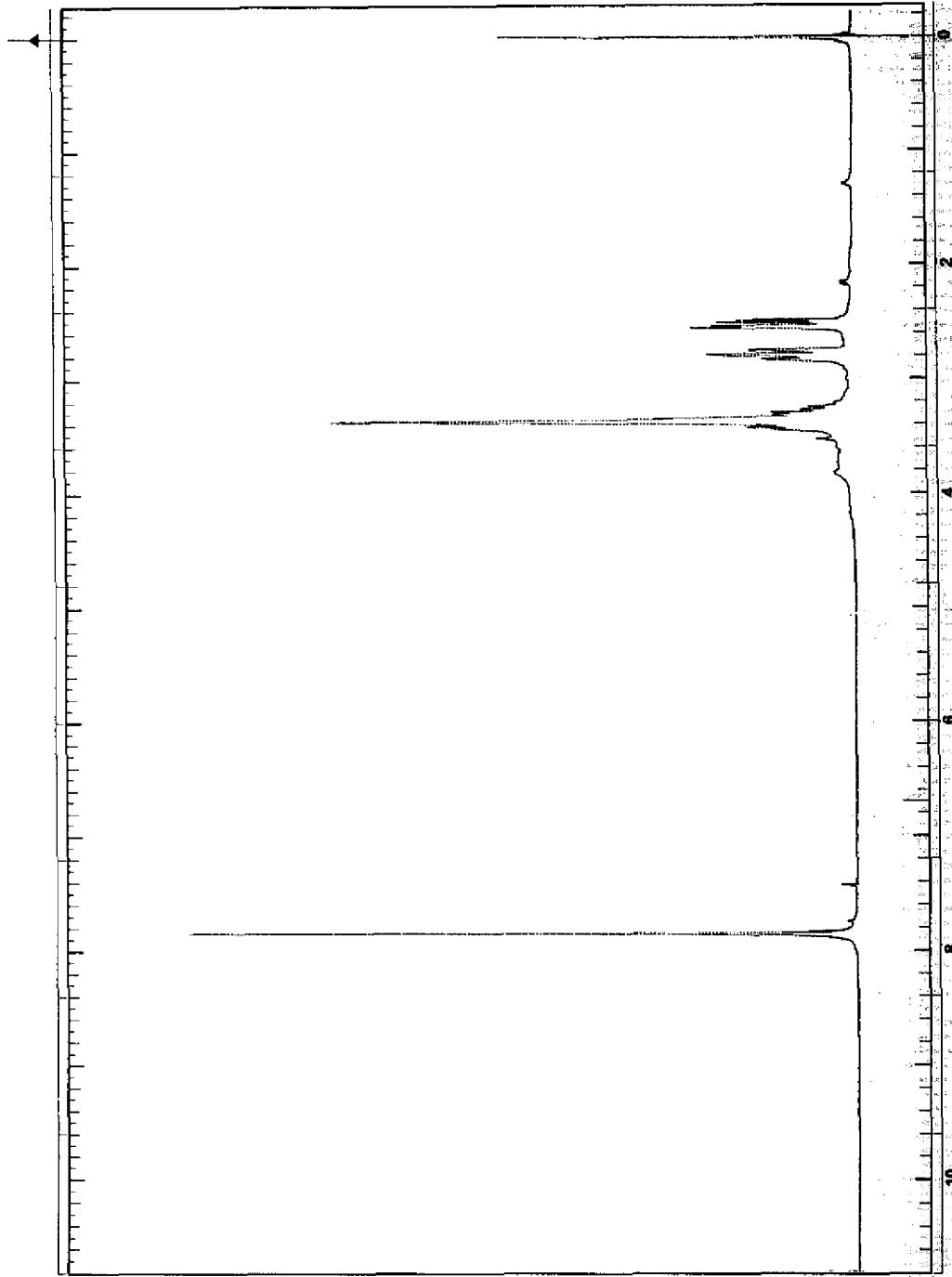
Assignments:	
a	44.7
e	126.9
b	50.4
f	134.6
c	50.8
g	145.8
d	56.0
	56.5
	119.3
	126.3

Source: Shell ERX-67

Solvent: 50% CHCl₃ *



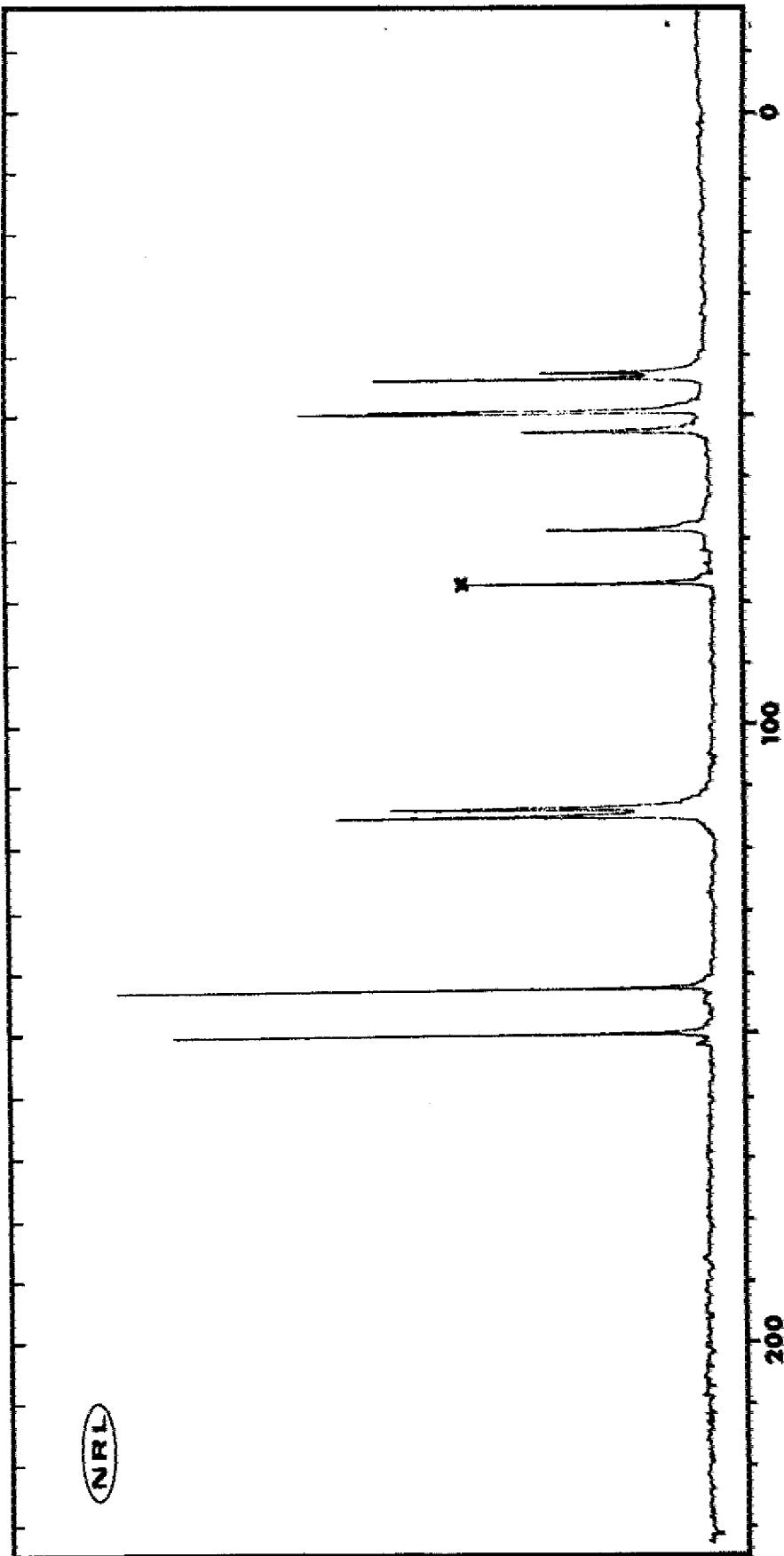
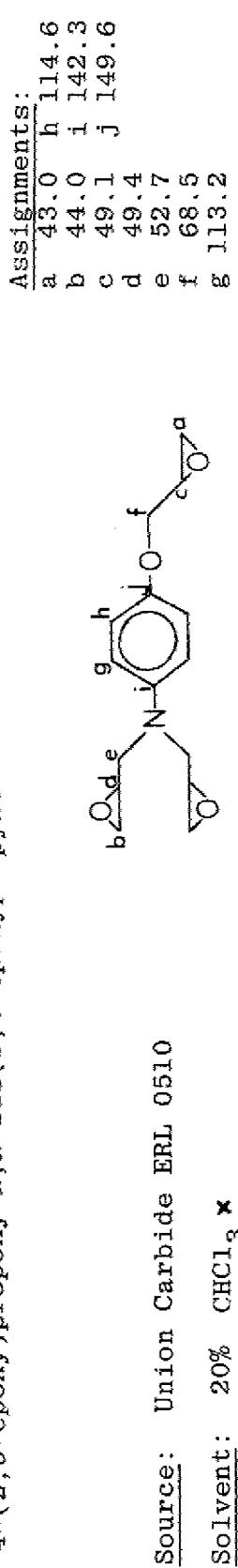
H14



Spectrum 14 — *N,N-bis(2,3-epoxypropyl)-2,4,6-tribromoaniline* (Shell ERX-67); solvent: CDCl_3

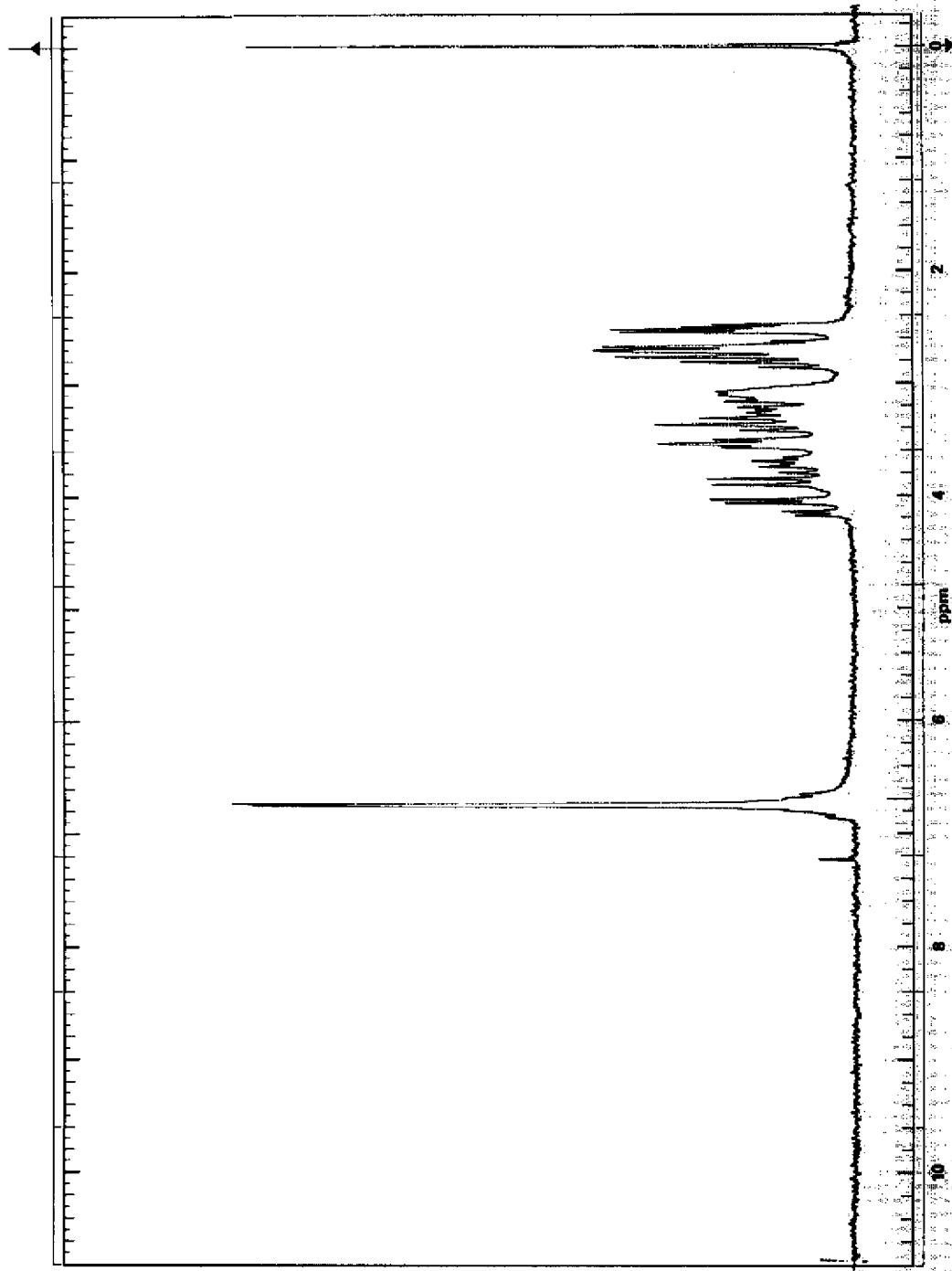
C15

4-(2,3-epoxy)propoxy-N,N-bis(2,3-epoxypropyl)-aniline



NRL

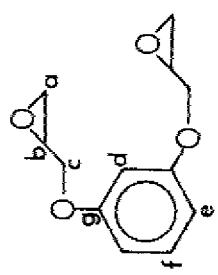
H15



Spectrum 15—4-(2,3-epoxy)propoxy-N,N-bis(2,3-epoxypropyl)-aniline (Union Carbide ERL 0510); solvent: CDCl₃

C16

Resorcinol Diglycidyl Ether

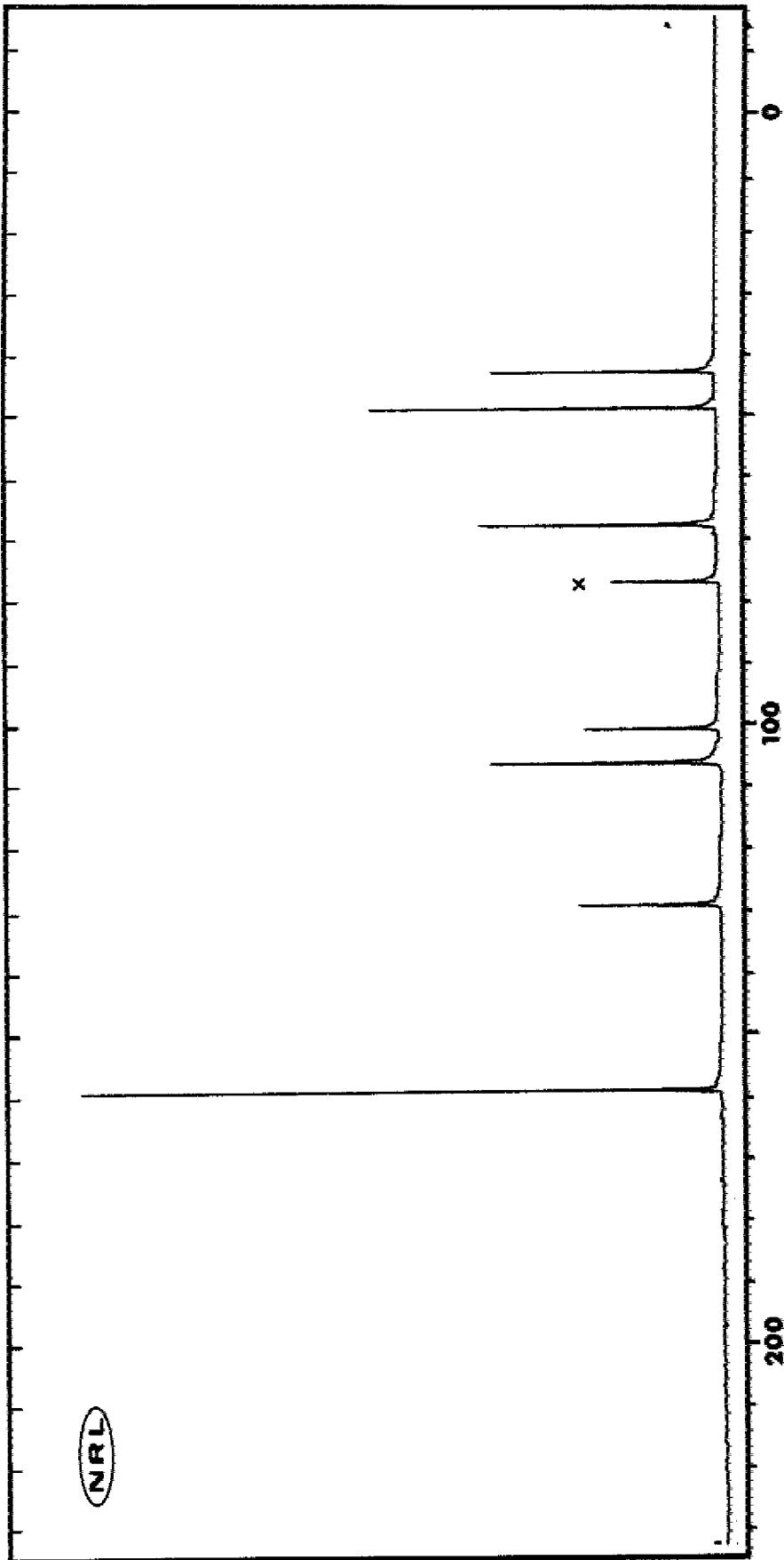


Assignments:

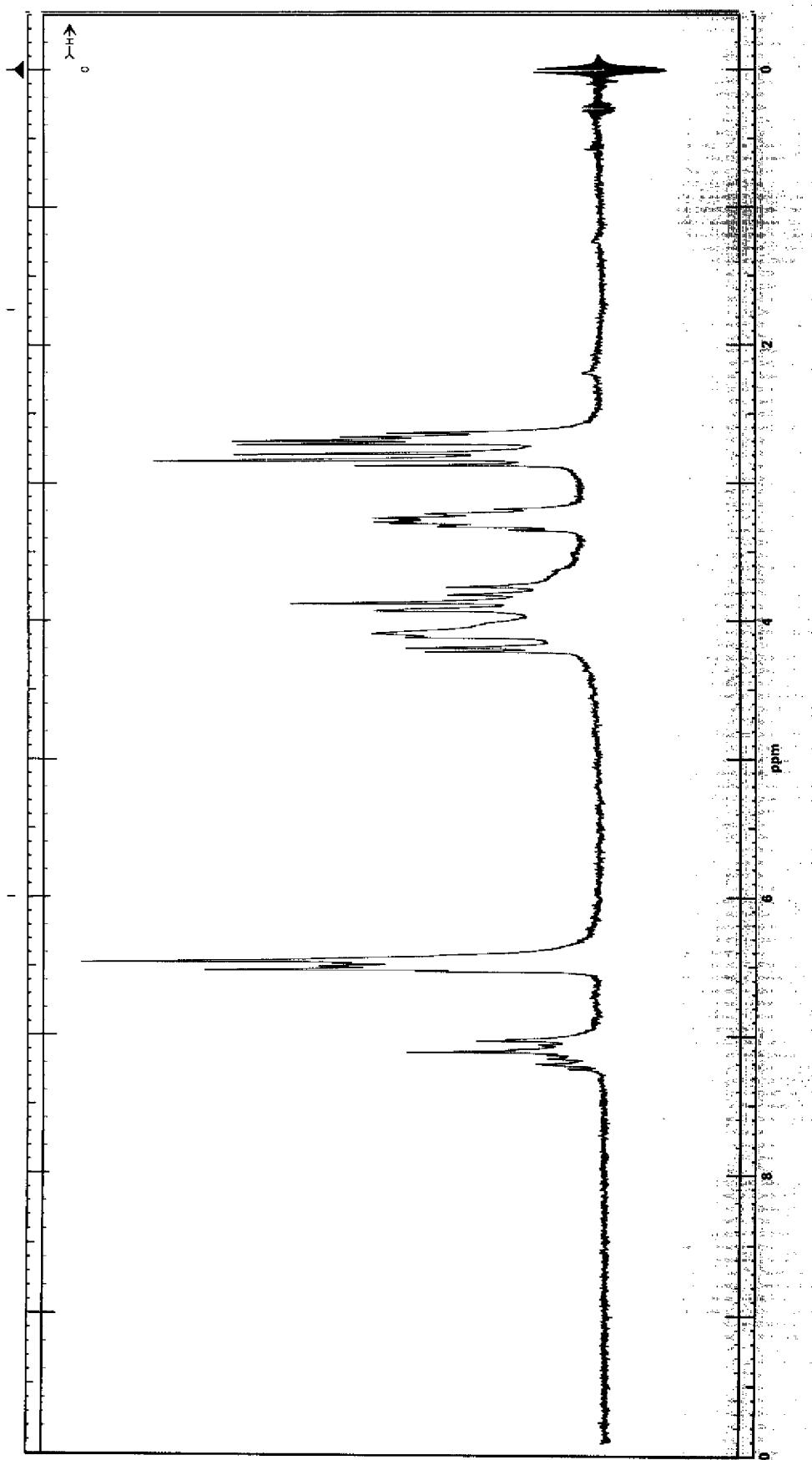
- | | |
|---|-------|
| a | 43.2 |
| b | 49.1 |
| c | 68.0 |
| d | 100.8 |
| e | 106.2 |
| f | 129.0 |
| g | 158.6 |

Source: Koppers Kopox 159

Solvent: 20% CHCl₃ *



H16



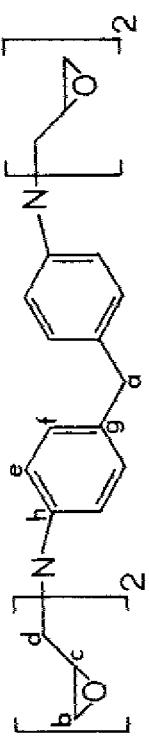
Spectrum 16—Resorecinol diglycidyl ether (Koppers Kopox 159); solvent: CDCl₃

C17

Bis(N,N-di(2,3-epoxypropyl)-4-aminophenyl)methane

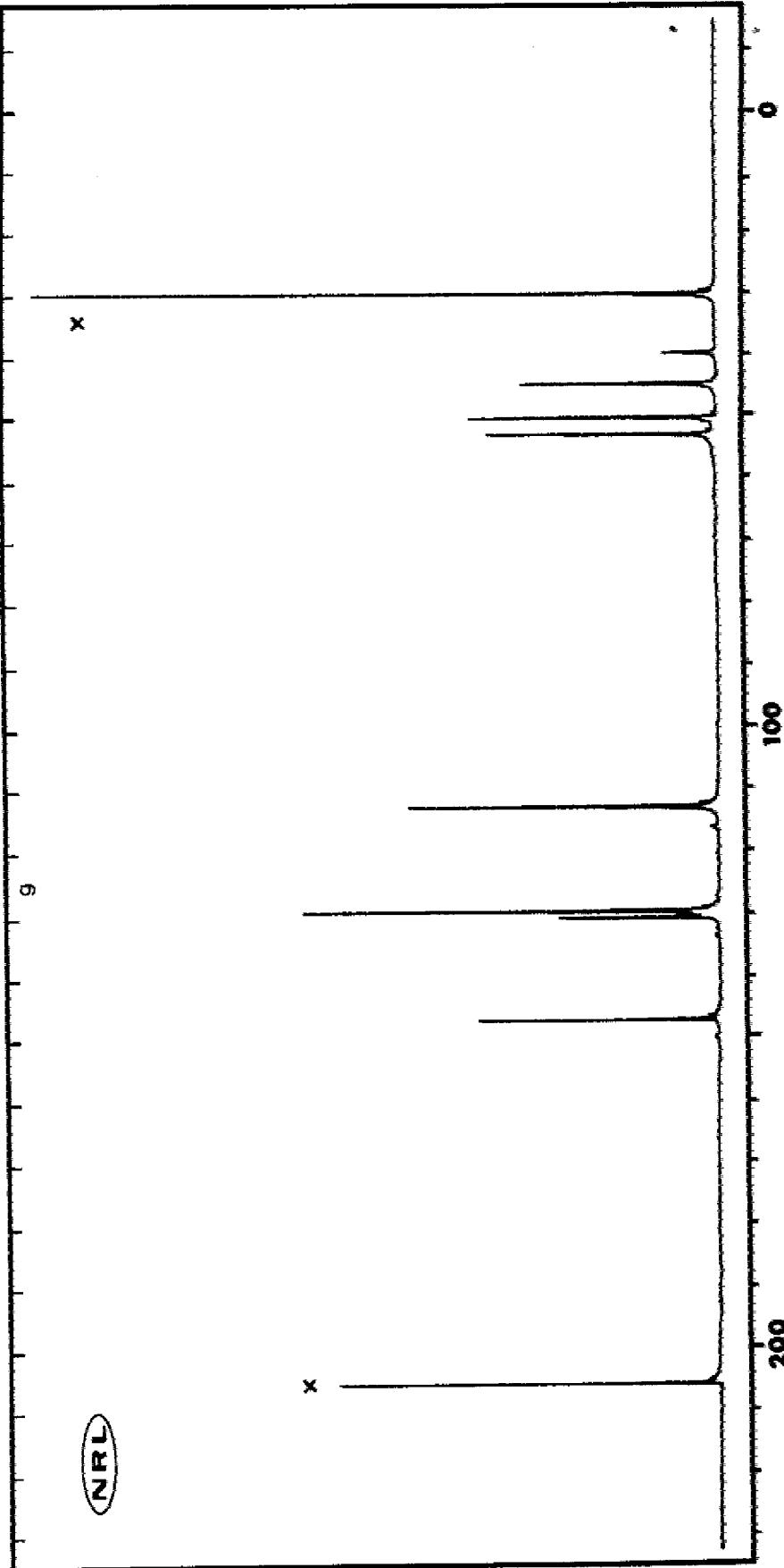
Assignments:

a	39.9t	h	146.9s
b	45.1t		
c	50.5d		
d	53.2t		
e	112.7d		
f	129.5d		
g	130.5s		

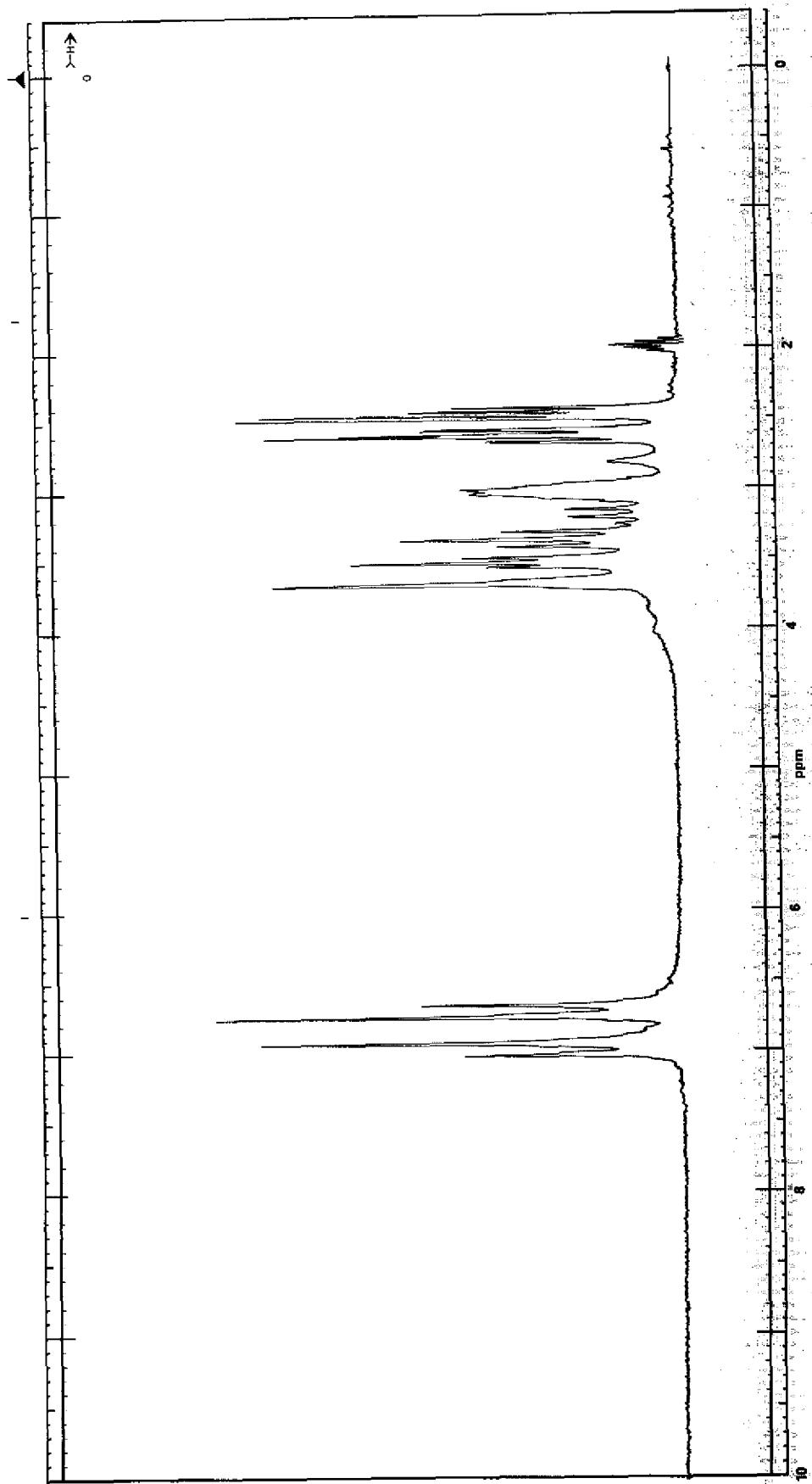


Source: Ciba-Geigy MY720

Solvent: 10% Acetone x



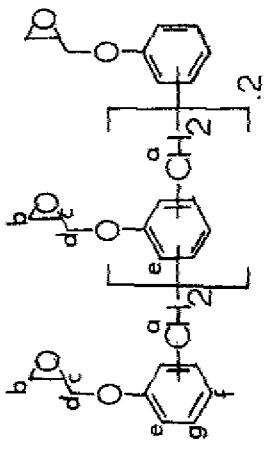
H17



Spectrum 17 — *bis(N,N-di(2,3-epoxypropyl)-4-aminophenyl)methane* (Ciba-Geigy M Y720); solvent: acetone-d₆

C18

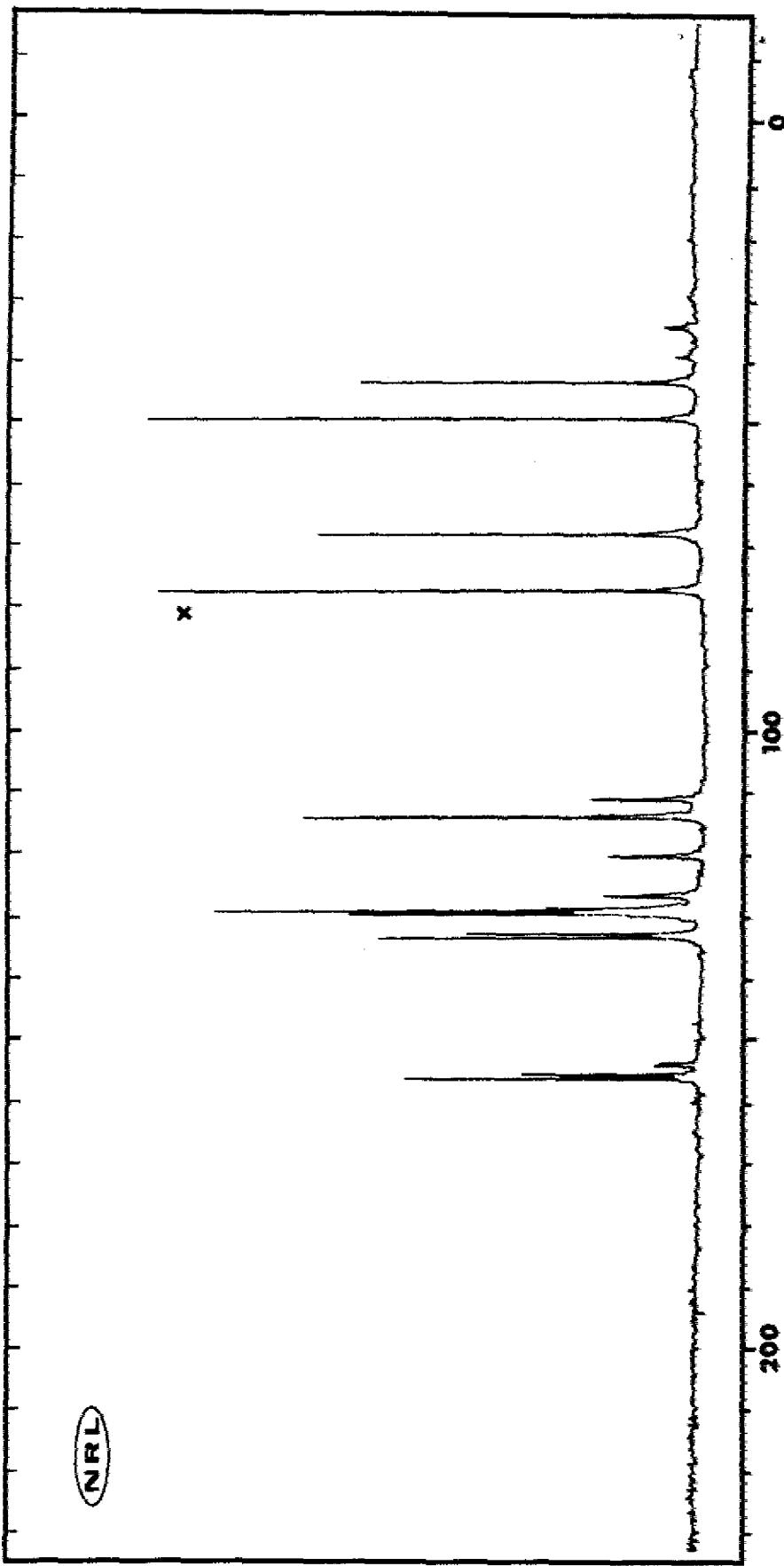
Epoxy Novolac Resin



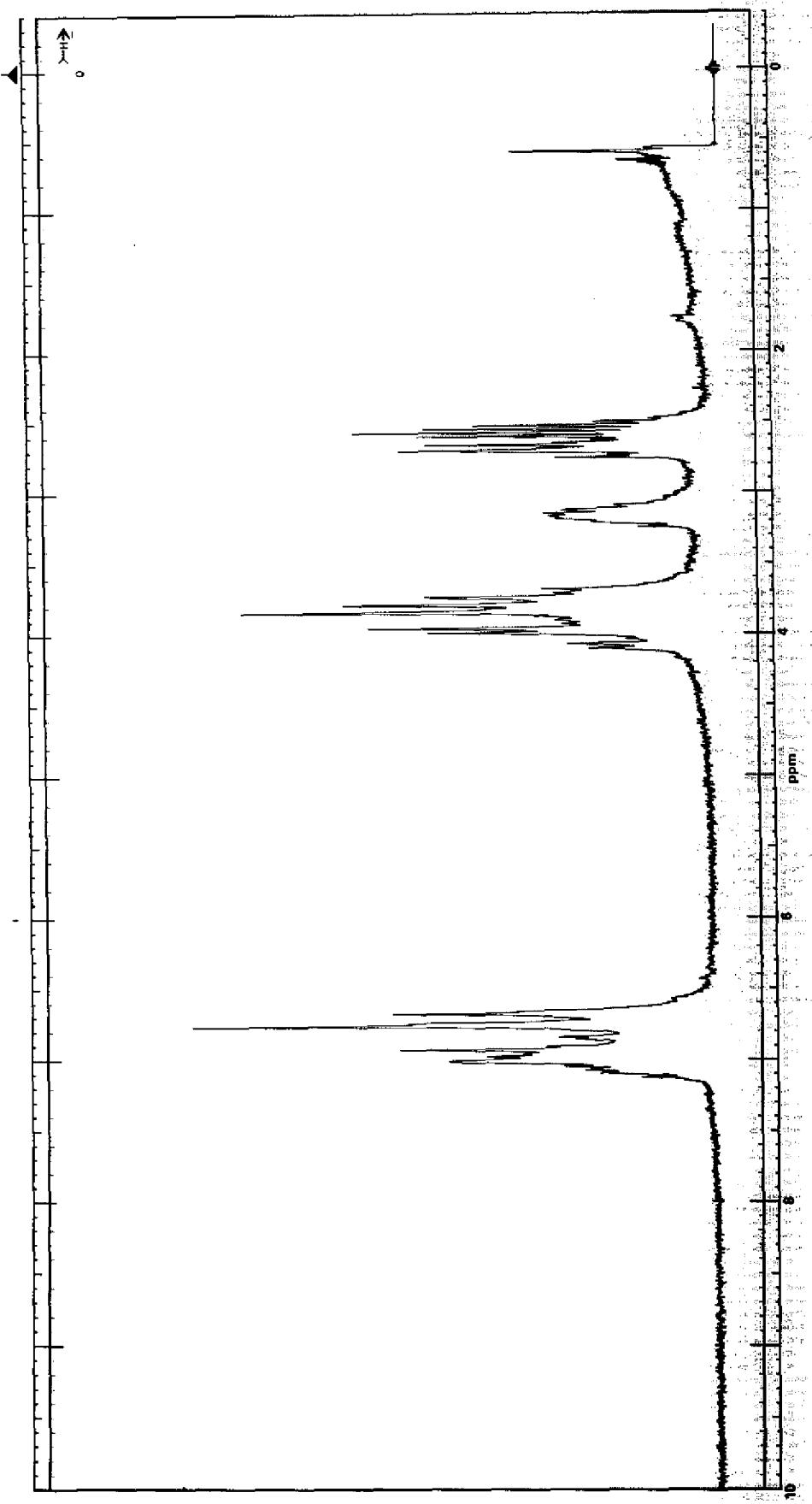
Assignments:		
a	29-40	g 126-130
b	43.3	h 132.6
c	49.2	i 133.2
d	68.0	j 153-156
e	110.8	
f	113.6	
	120.1	

Source: Dow D.E.N. 431

Solvent: 20% CHCl₃ *



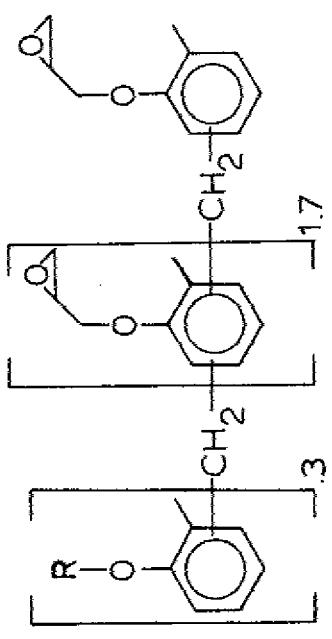
H18



Spectrum 18 — Epoxy Novolac resin (Dow D.E.N. 431); solvent: CDCl_3

C19

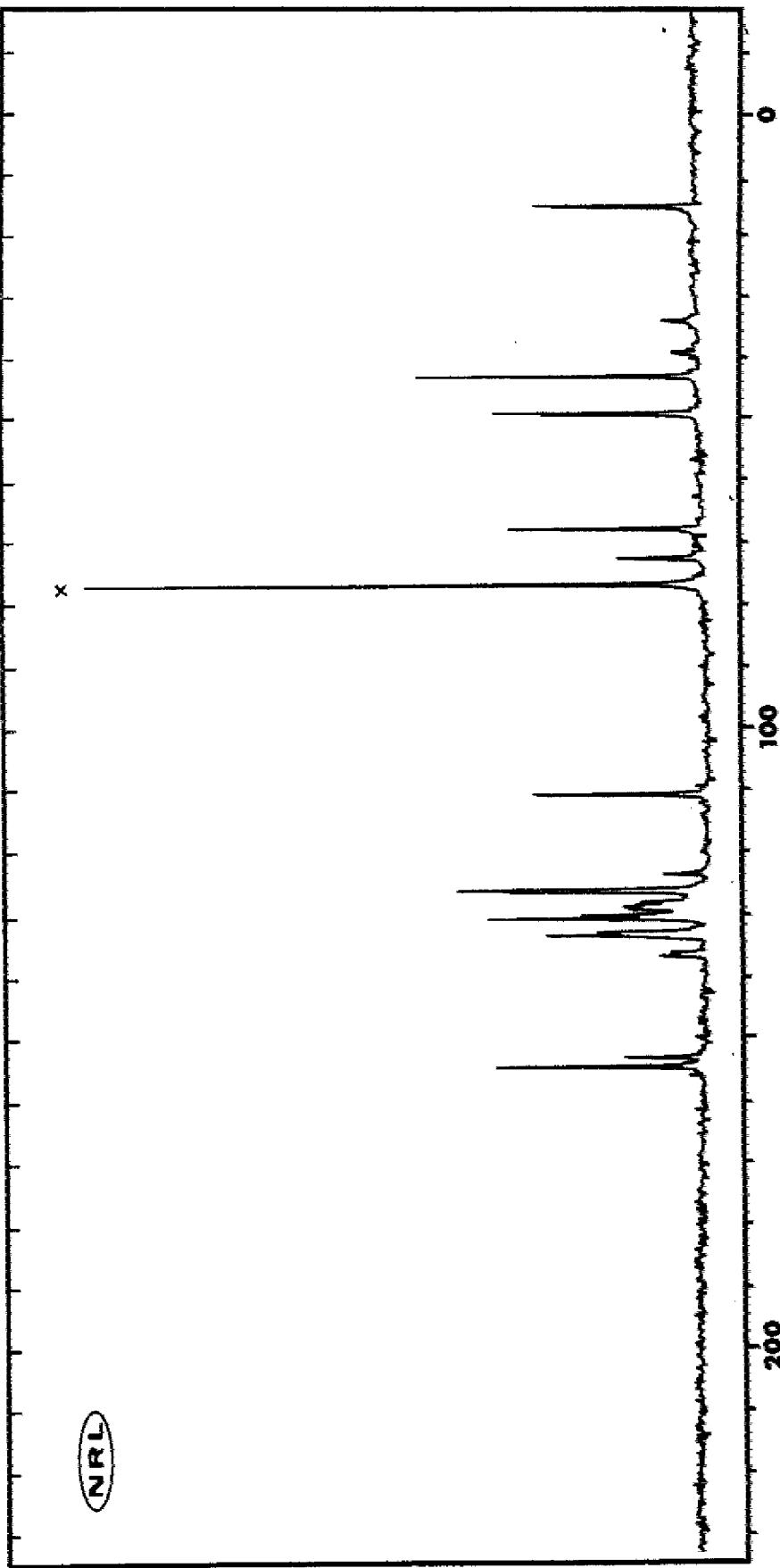
Epoxy Novolac Resin



Source: Ciba-Geigy ECN 1235

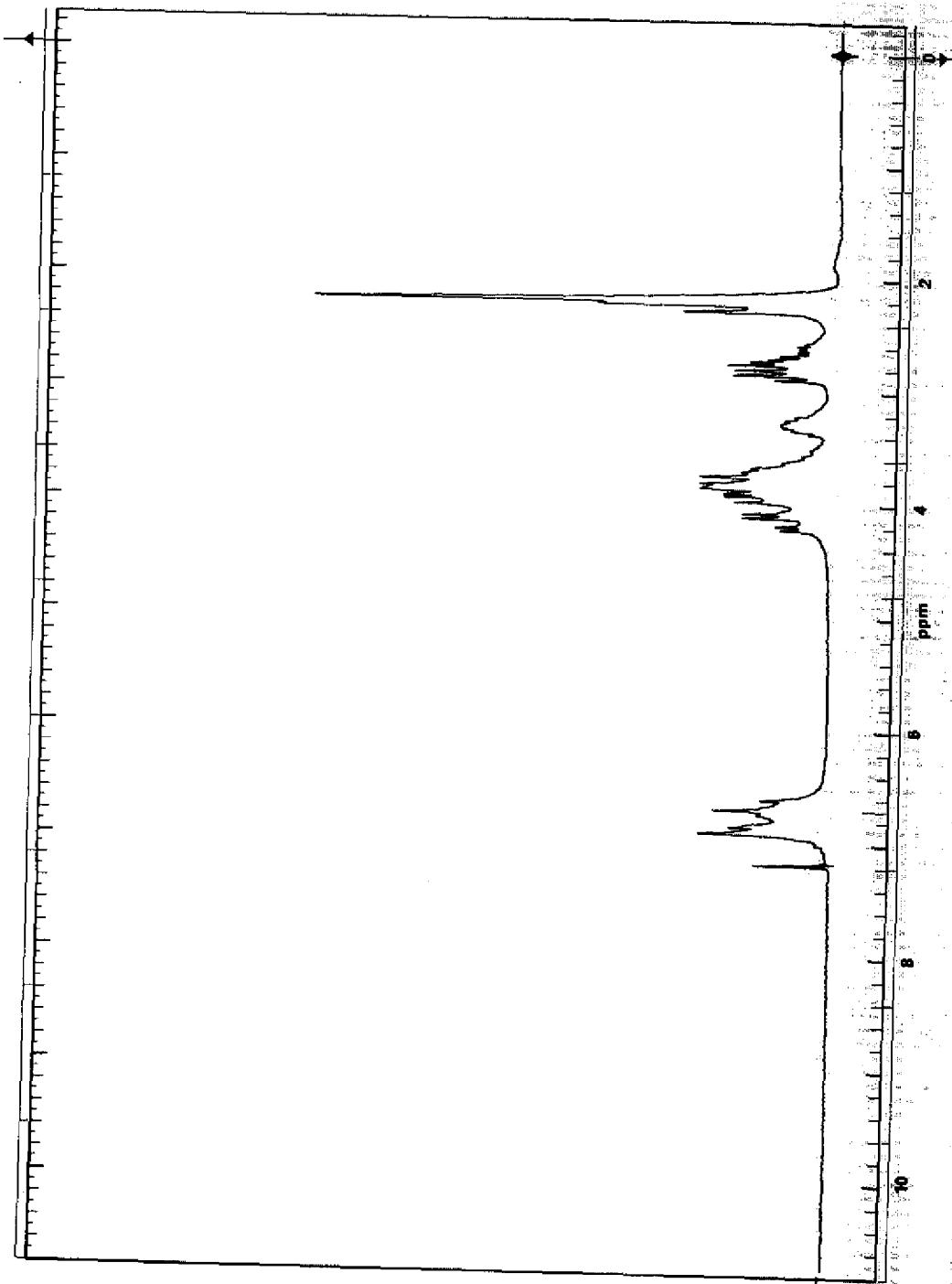
Solvent: 50% CHCl₃ x

Assignments:		
a	15.6	h 110.6
b	34.2	i 123.3
c	39.4	j 126.0
d	43.6	k 128.6
e	49.6	l 130.5
f	68.2	m 133.1
g	72.8	n 136.4
		o 152.6
		p 154.2



NRL

H19

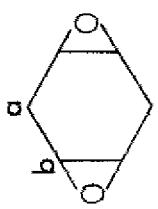


Spectrum 19 — Epoxy Novolac resin (Ciba-Geigy ECN 1235); solvent: CDCl_3

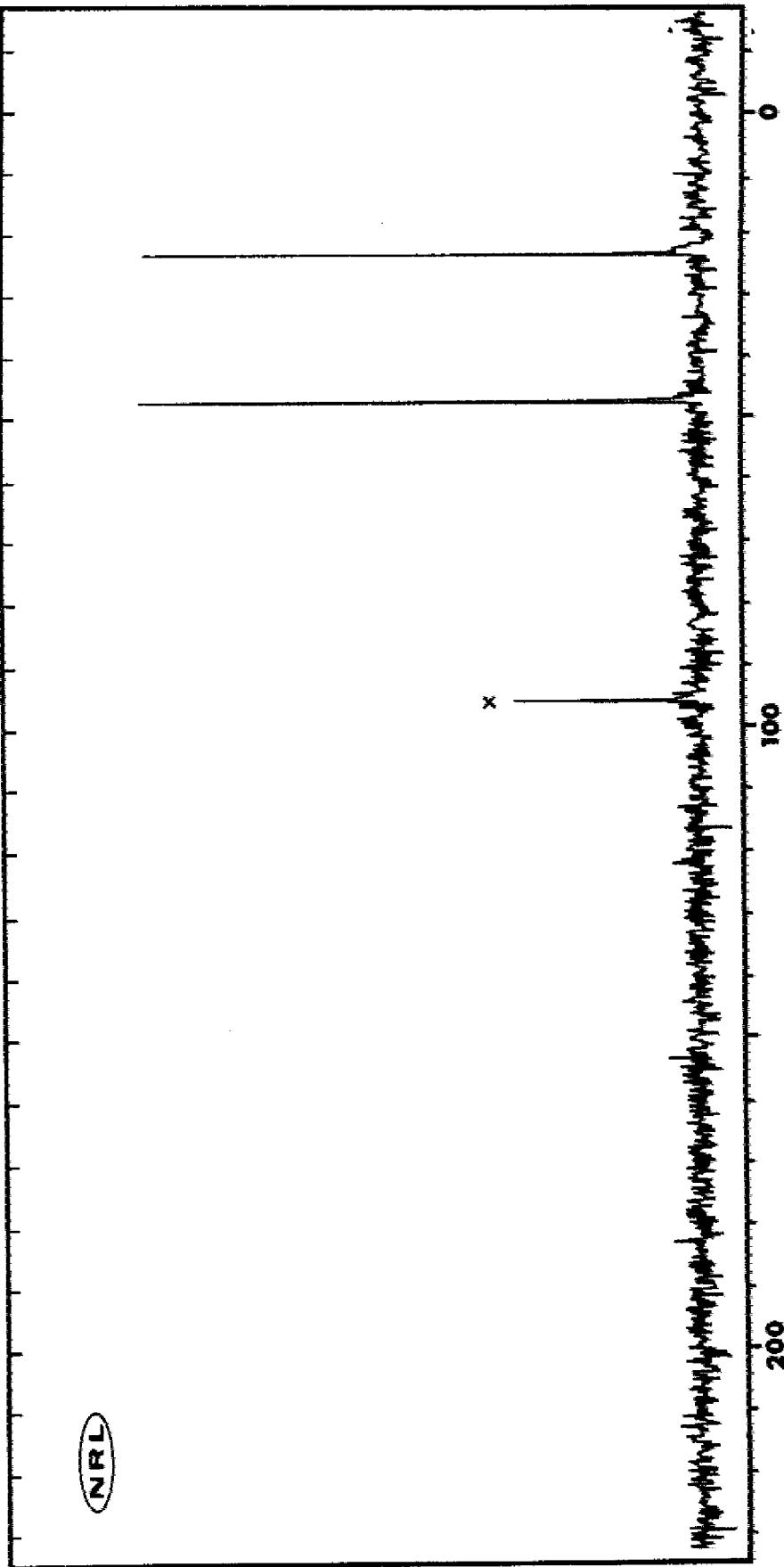
C20

4,8-Dioxatricyclo[5.1.0.0^{3,5}]octane

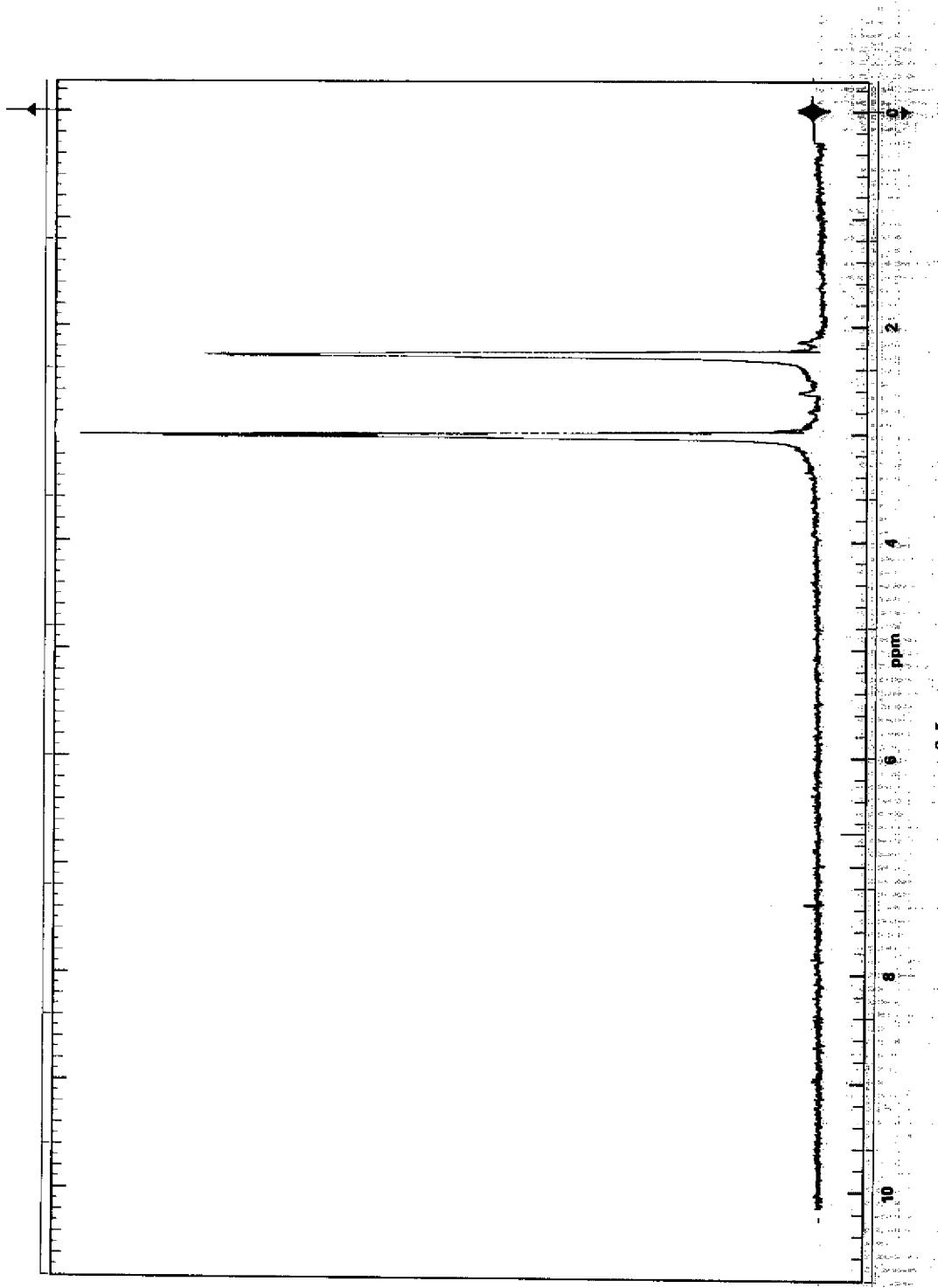
Source: Union Carbide ERL 2114
Solvent: 10% CCl₄ *



Assignments:
a 24.4t
b 48.4d



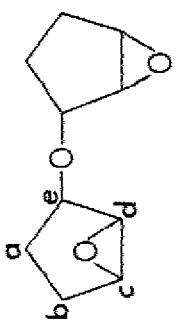
H20



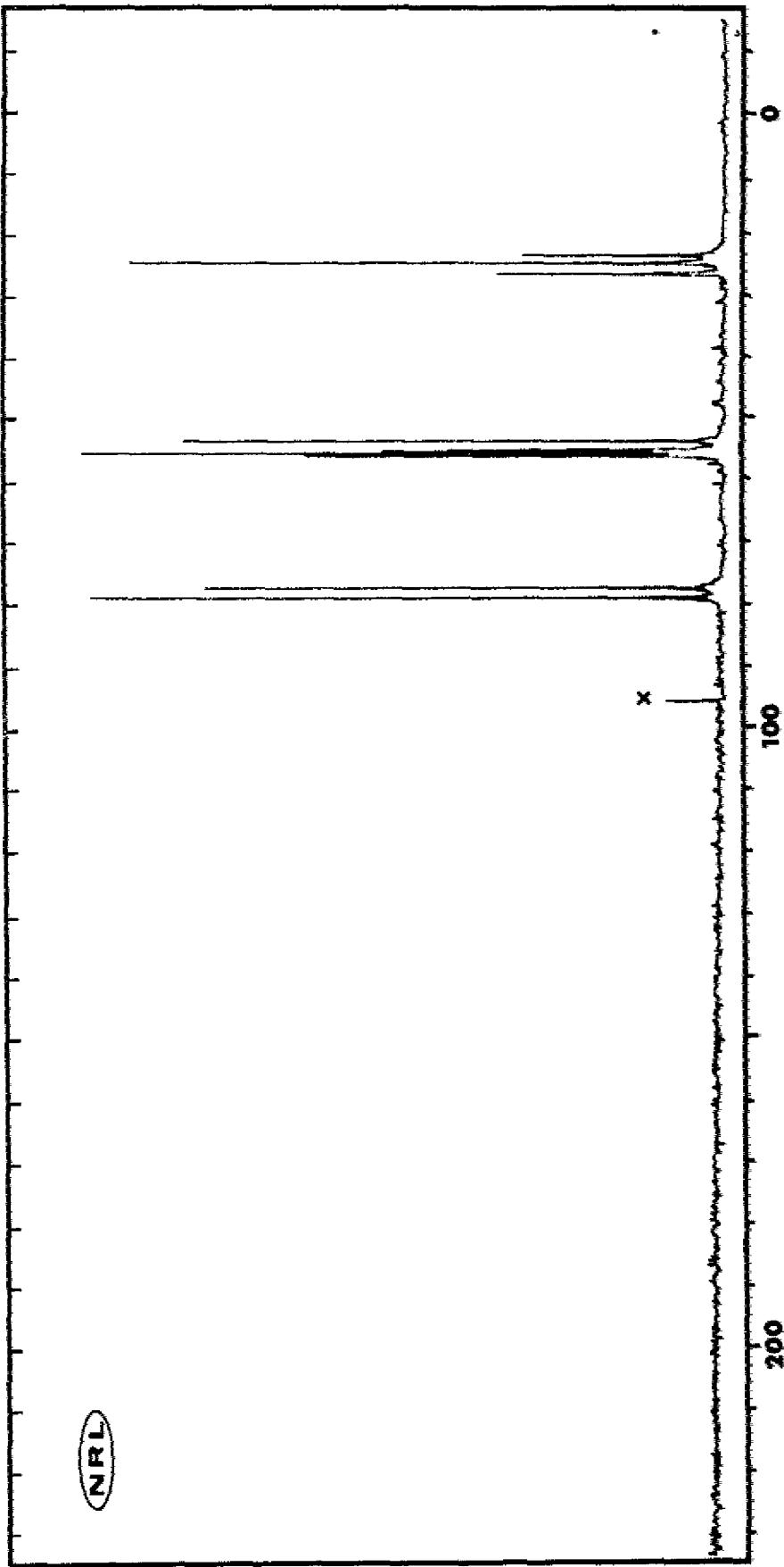
Spectrum 20 — 4,8-Dioxatricyclo [5.1.0.0^{3,5}]octane (Union Carbide ERI 2114); solvent: CDCl₃

C21

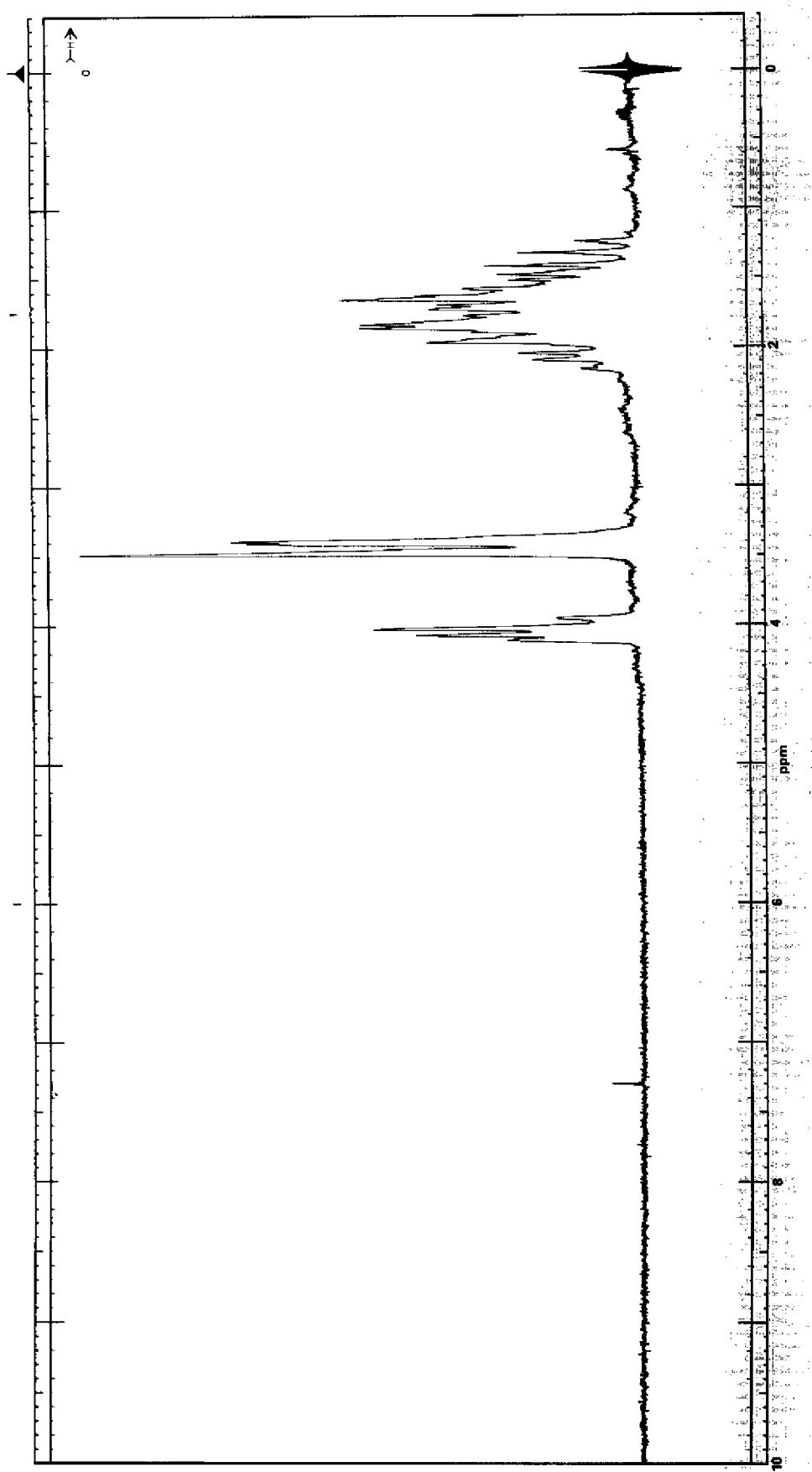
Bis(2,3-epoxycyclopentyl)ether



Source: Union Carbide ERL 0400
Solvent: 20% CCl₄ *



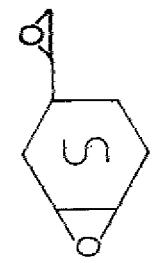
H21



Spectrum 21 – *bis(2,3-epoxycyclopentyl)ether* (Union Carbide ERL 0400); solvent: CDCl_3

C22

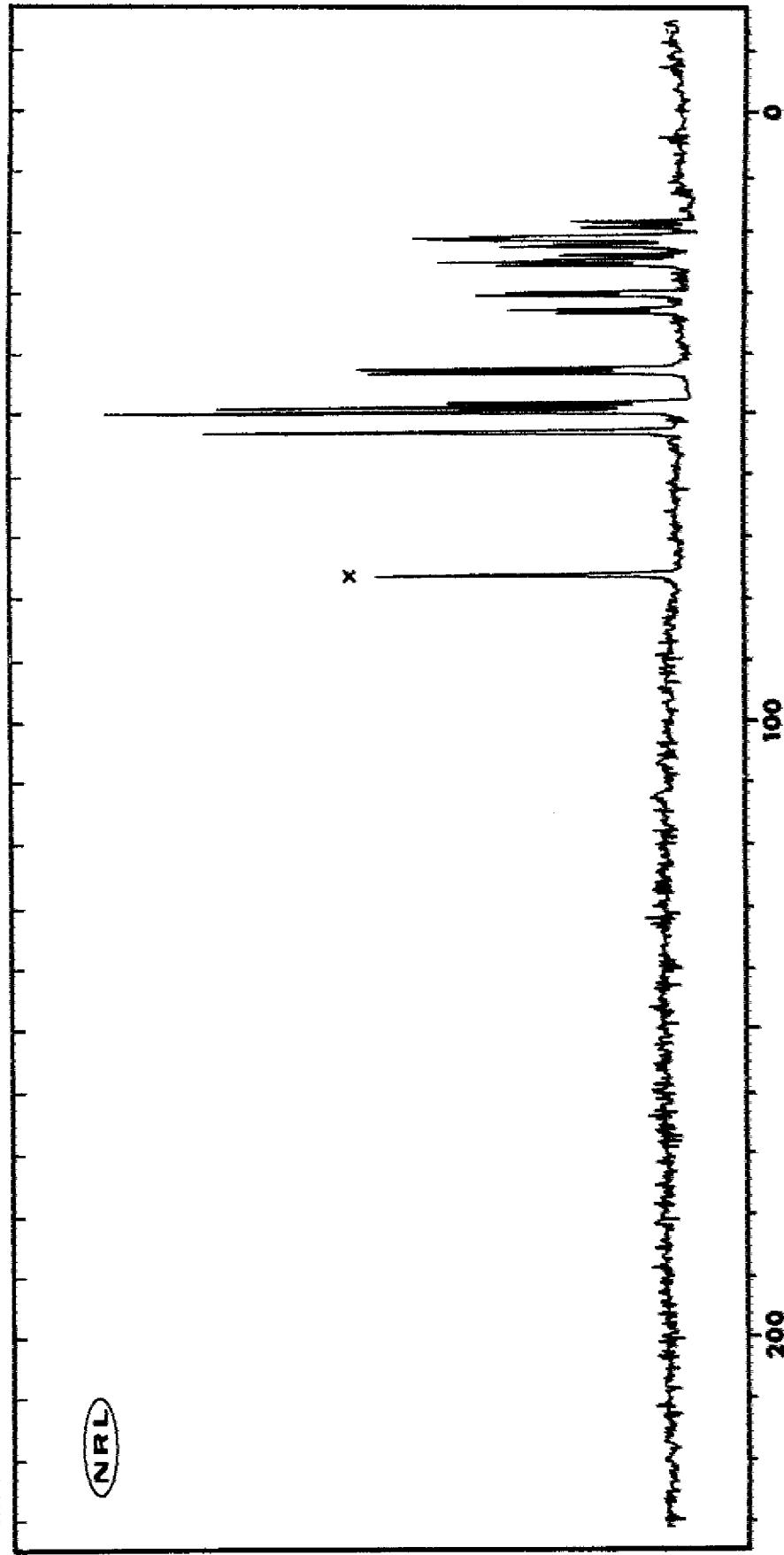
4-(1,2-Epoxyethyl)-1,2-epoxycyclohexane



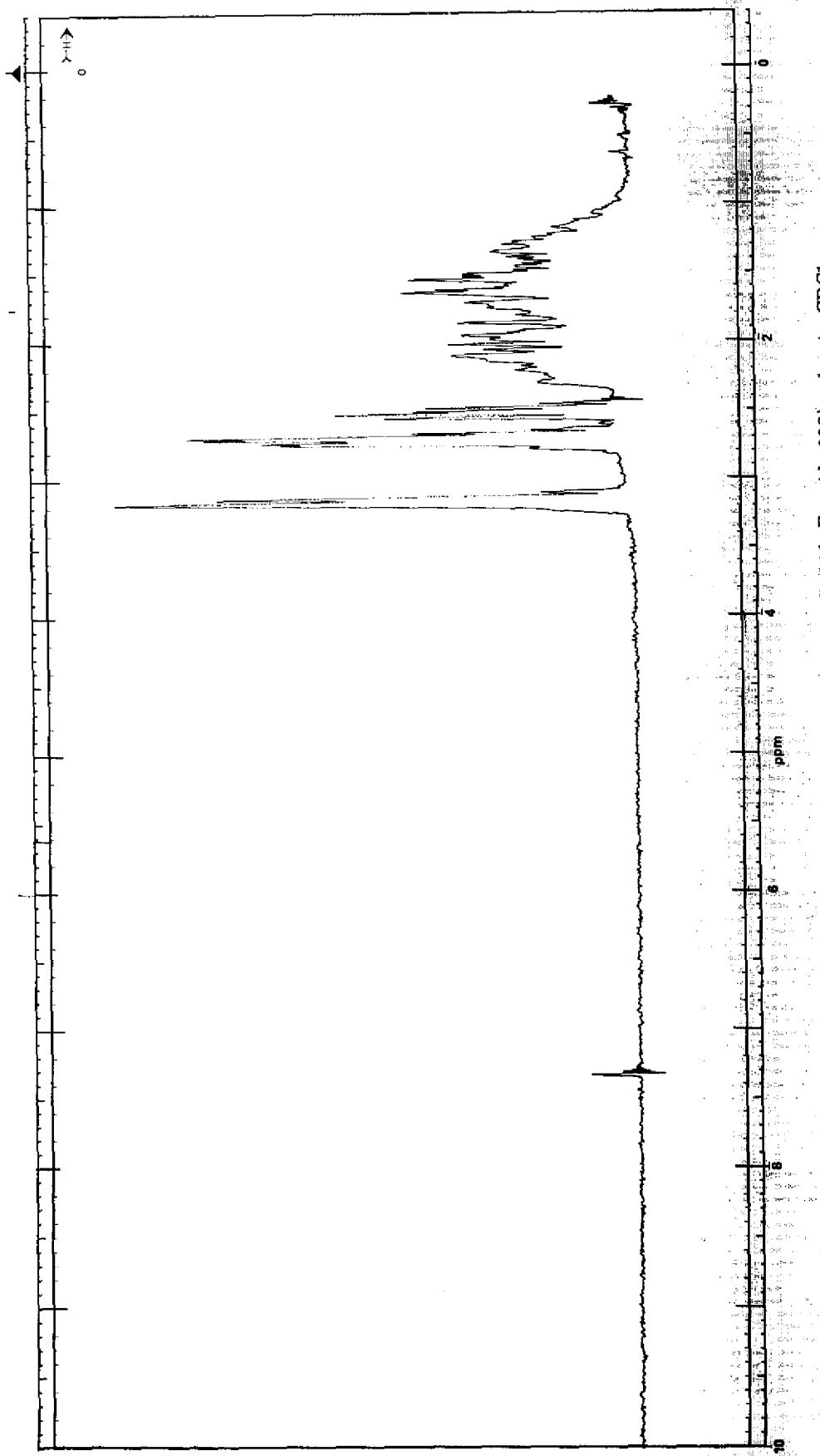
Assignments:	
a	19.4
b	20.3
c	21.8
d	22.2
e	22.4
f	23.3
g	23.5
h	25.0
i	25.8
j	26.0
k	26.7
l	31.0
m	31.5
n	34.0
o	34.6
p	43.6
q	44.3
r	49.3
s	49.9
t	50.7
u	54.0

Source: Union Carbide Epoxide 206

Solvent: 25% CHCl₃ *



H22



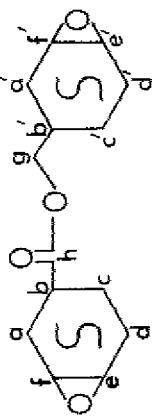
Spectrum 22 — 4-(1,2-Epoxyethyl)-1,2-epoxycyclohexane (Union Carbide Epoxide 206); solvent: CDCl_3

C23

3,4-Epoxy cyclohexylmethyl-(3,4-epoxy)cyclohexane Carboxylate

Assignments:

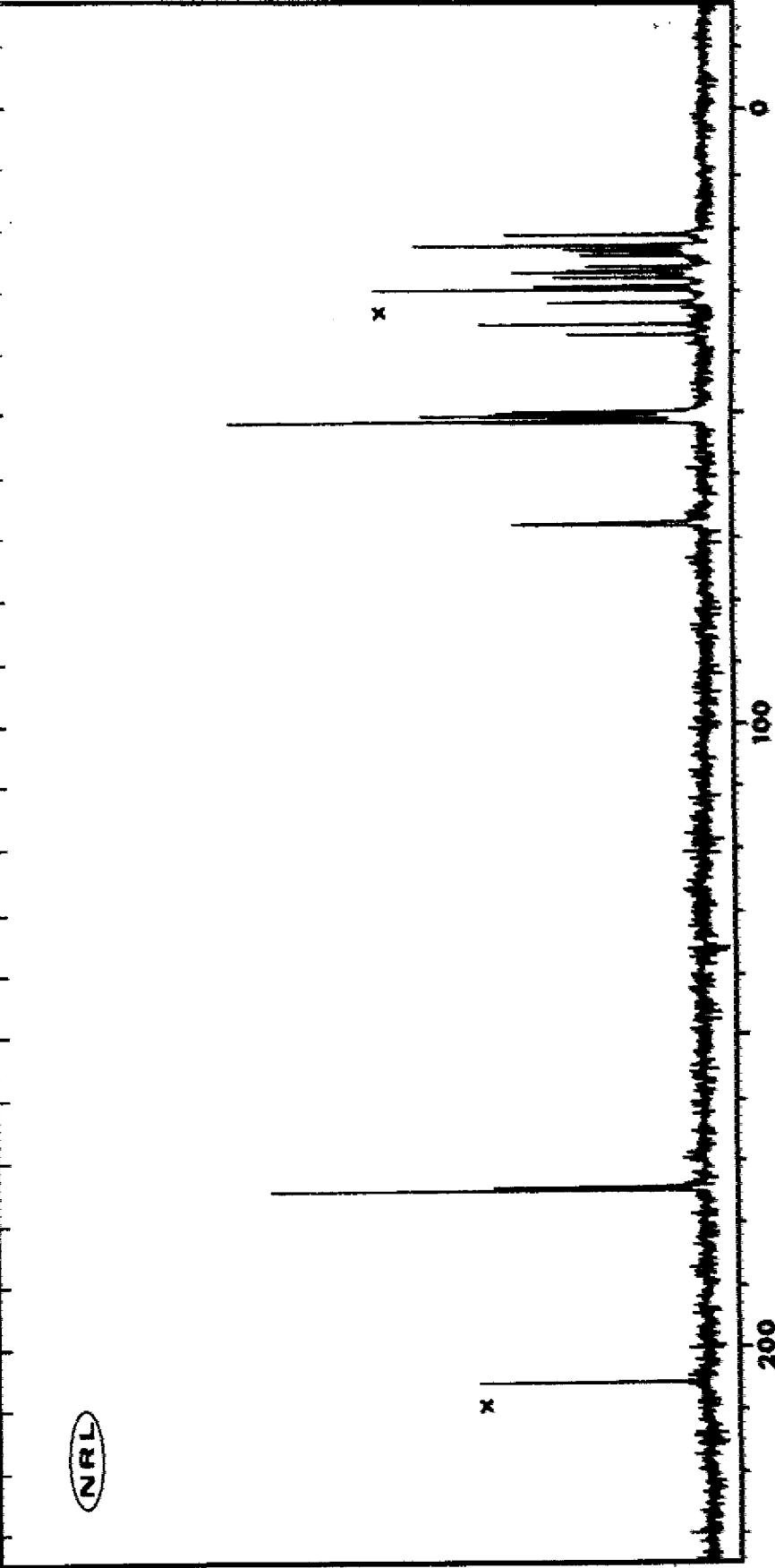
a, a'	20-40
b, b'	20-40
c, c'	20-40
d, d'	20-40
e, e'	50-54
f, f'	50-54
g	69.5
h	175.3, 175.8



Source: Union Carbide ERLA 4221

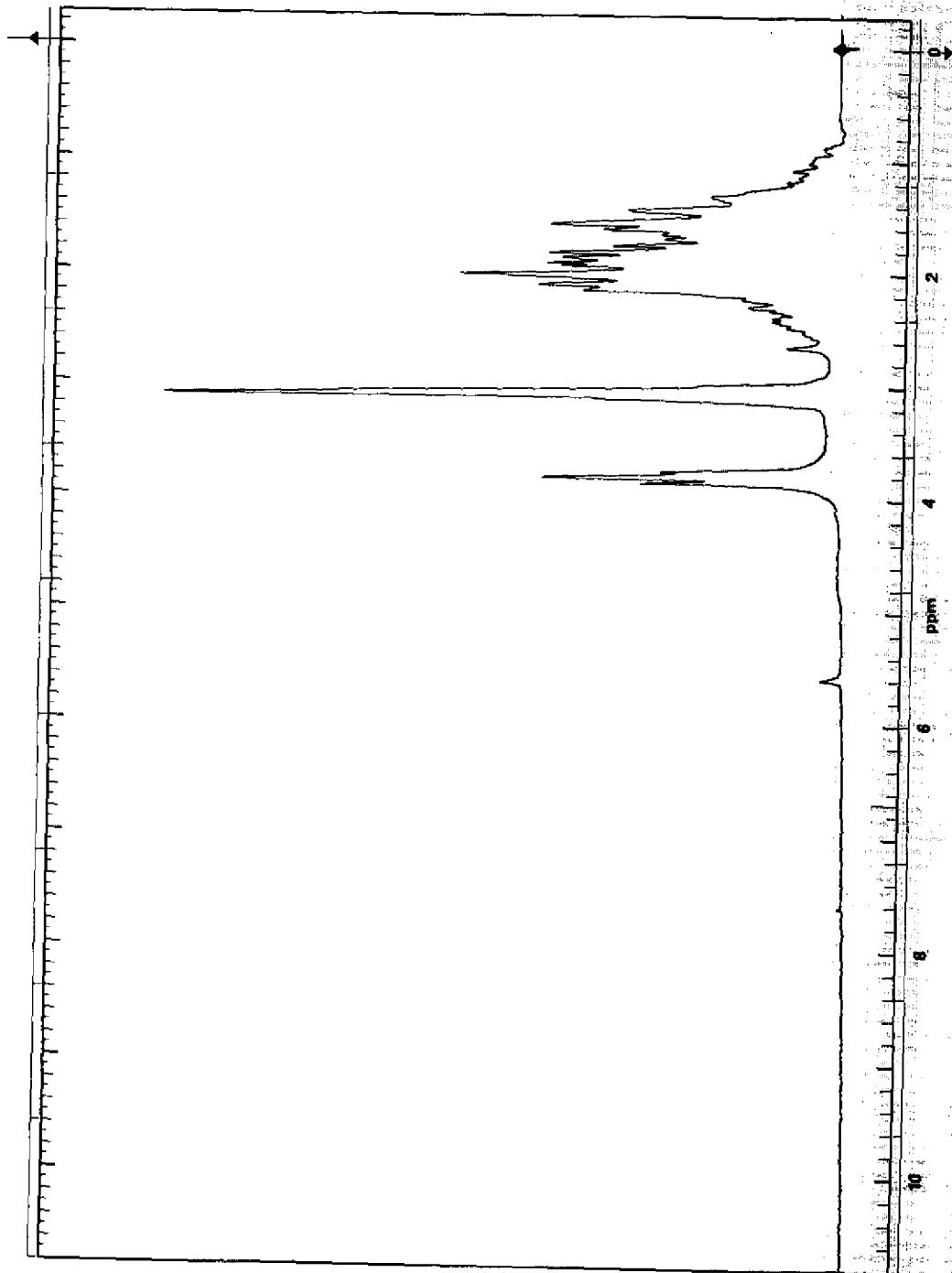
Solvent: 20% Acetone *

See Appendix for complete line listing



NRL

H23

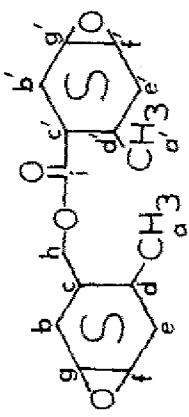


Spectrum 23 – 3,4-Epoxy cyclohexylmethyl-(3,4-epoxy)cyclohexane carboxylate (Union Carbide ERLA 4221); solvent: C₆D₆

C24

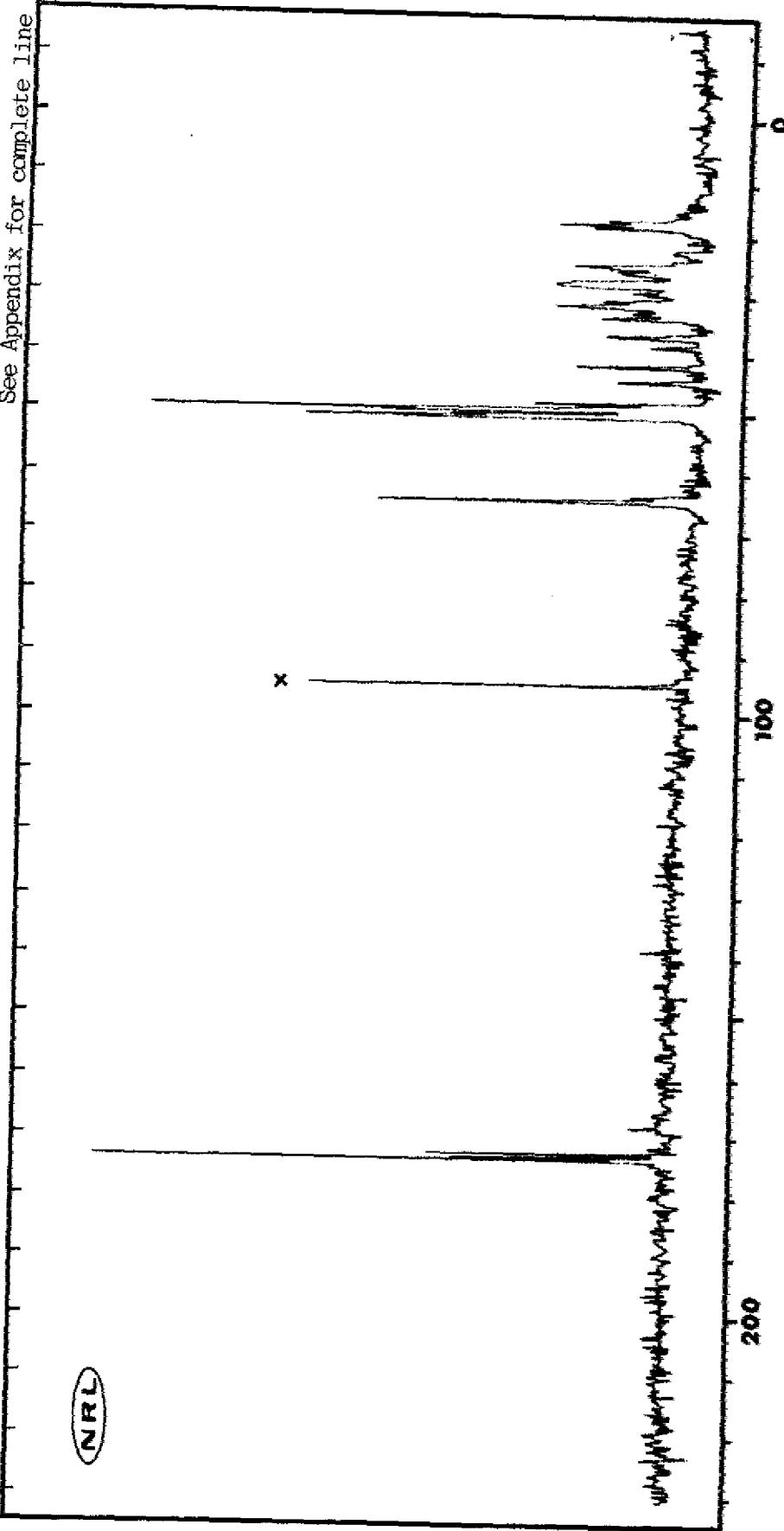
3,4-Epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexane Carboxylate

Source: Union Carbide Epoxide 201
Solvent: 25% CCl_4 *

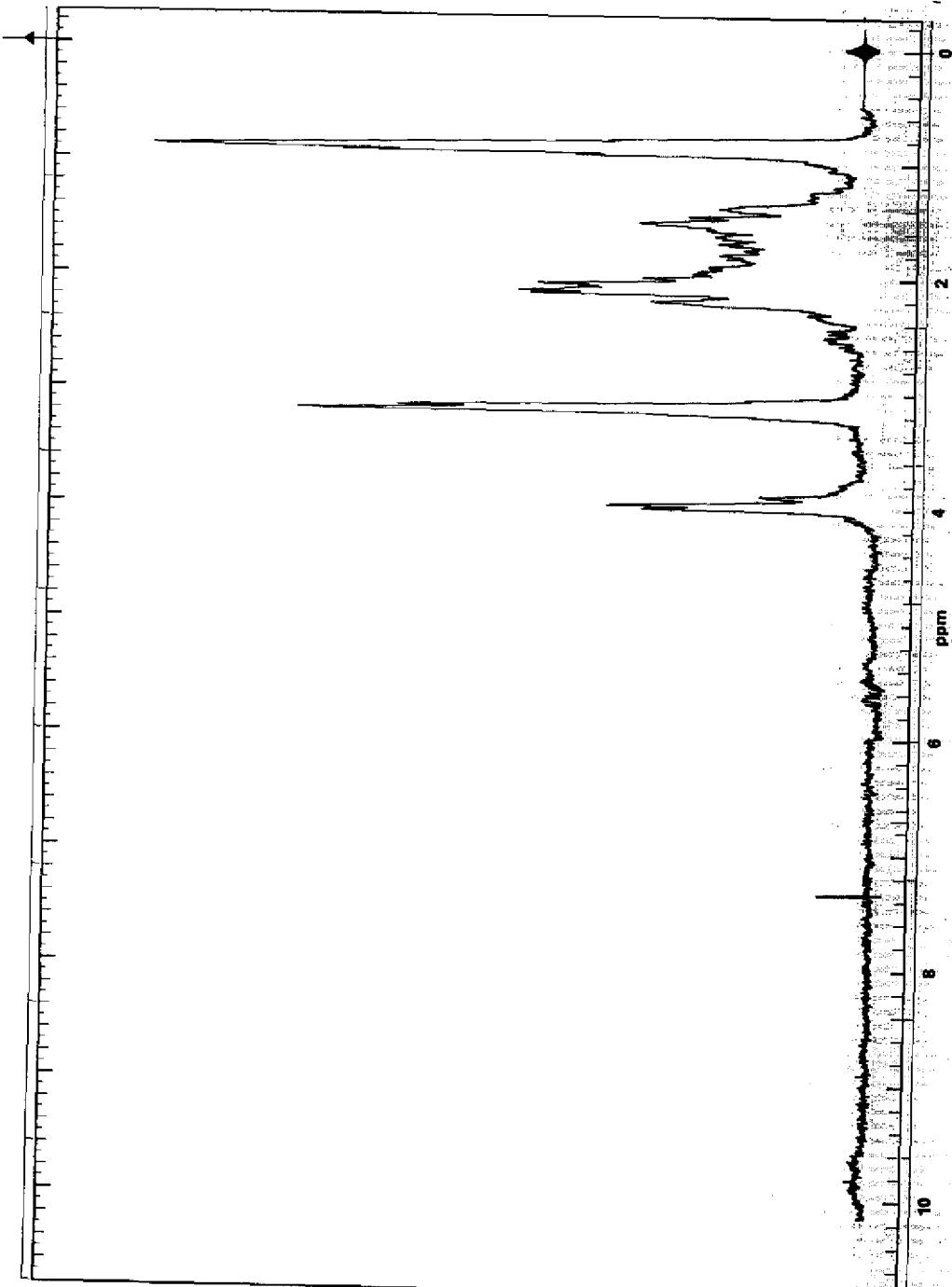


Assignments:	
a, a'	17-20
b, b'	23-46
c, c'	23-46
d, d'	23-46
e, e'	23-46
f, f'	49-52
g, g'	49-52
h	65
i	173-174

See Appendix for complete line listing



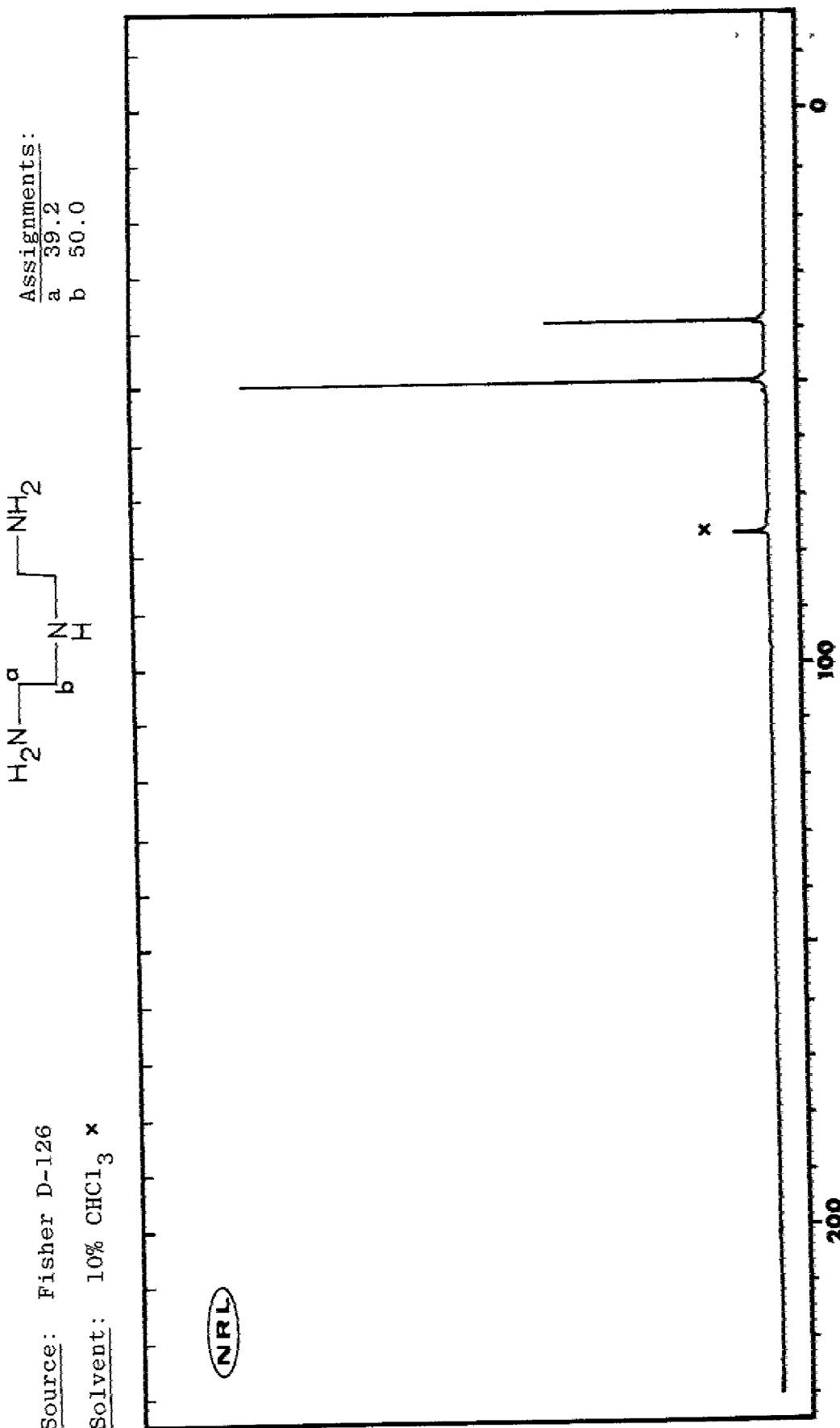
H24



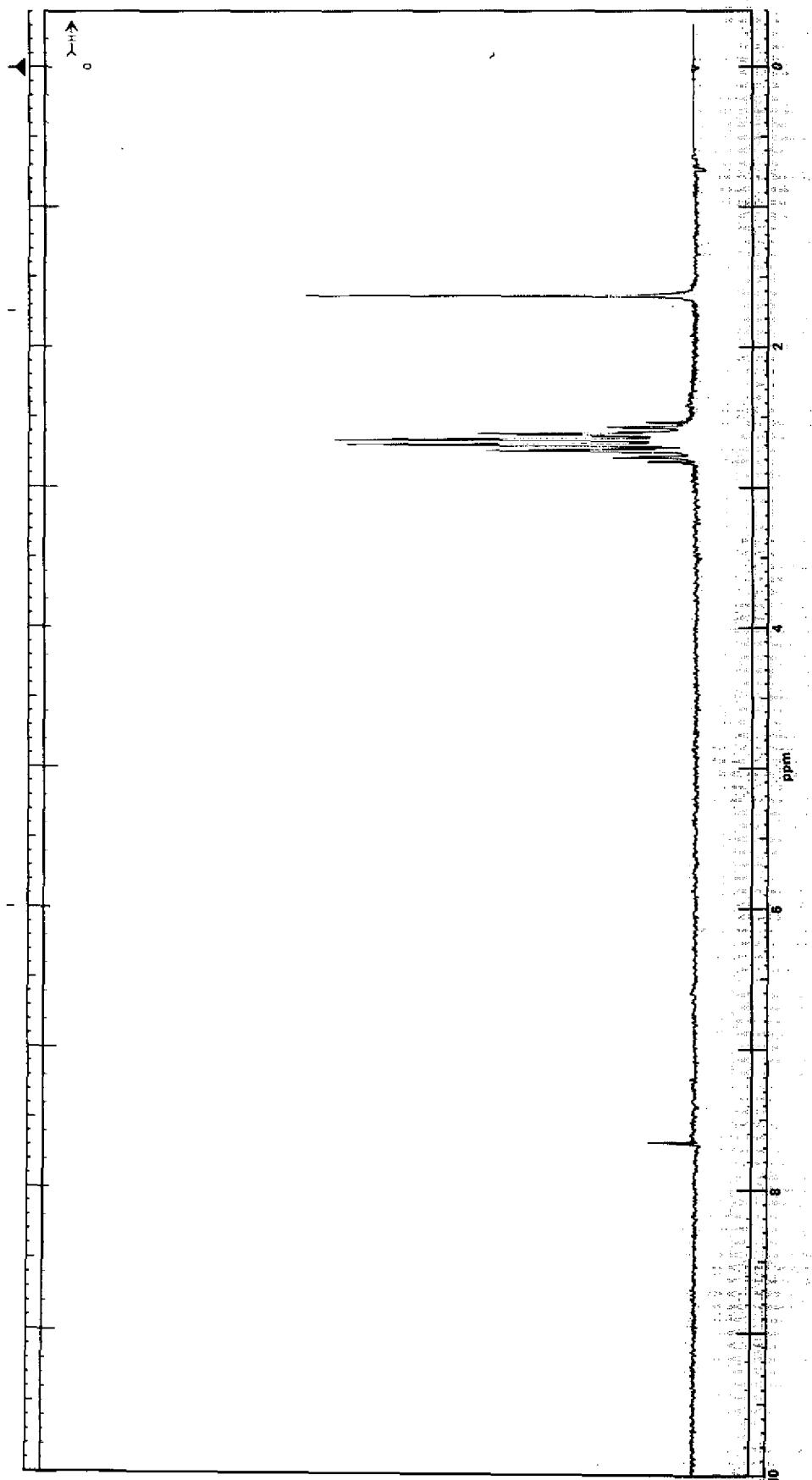
Spectrum 24 — 3,4-Epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexane carboxylate (Union Carbide Epoxide 201); solvent: CDCl_3

C25

Diethylenetriamine



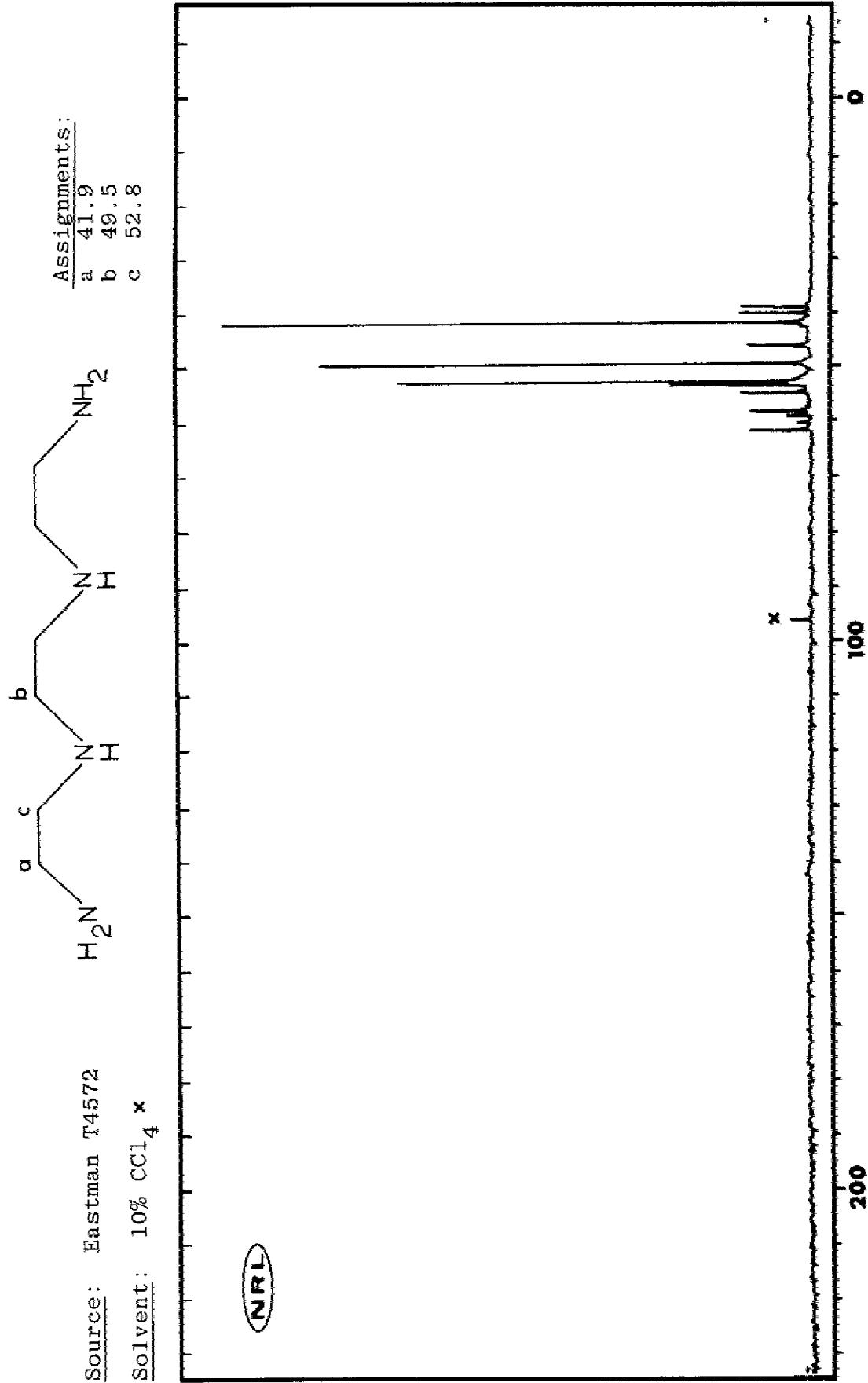
H25



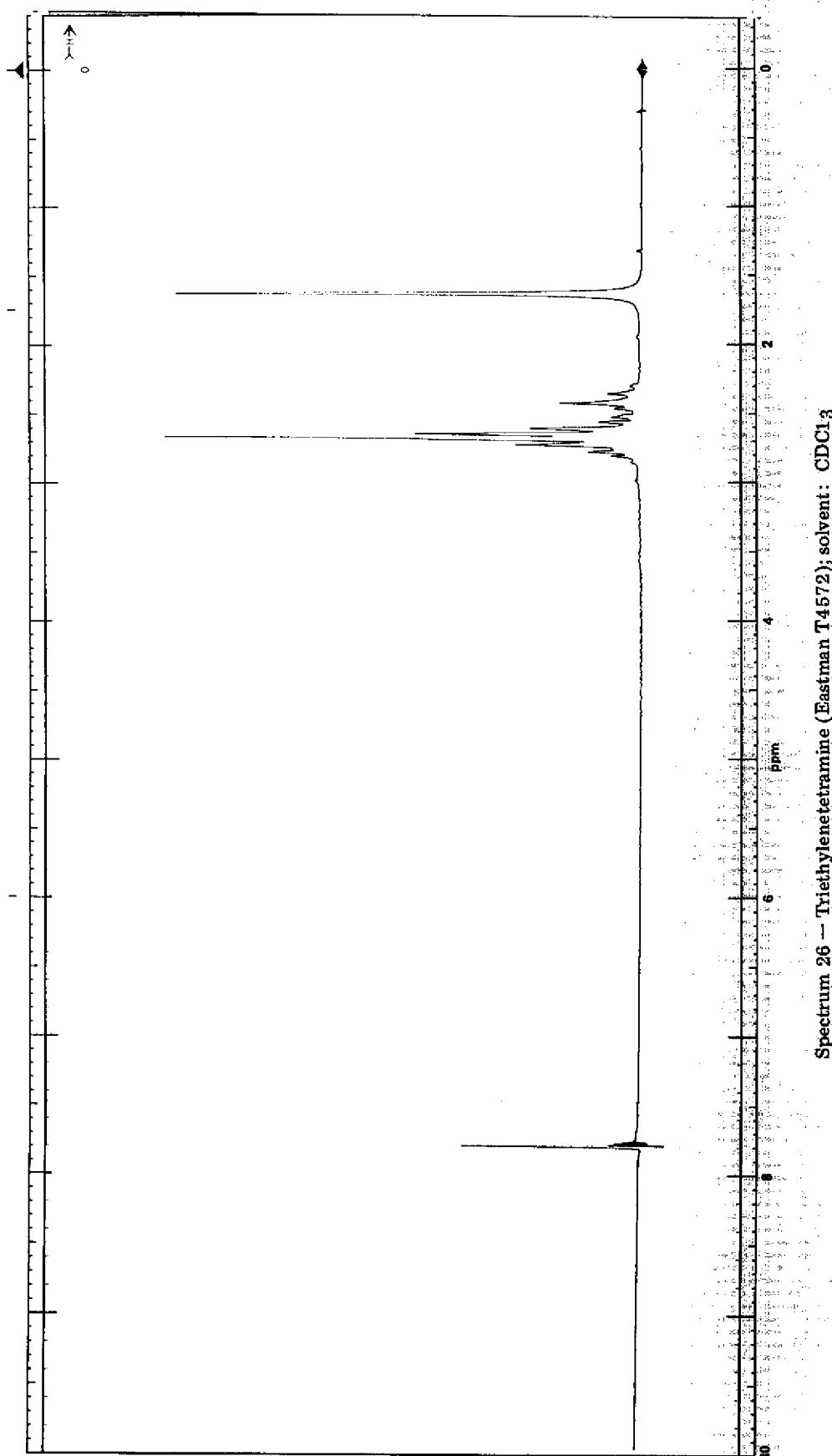
Spectrum 25 — Diethylenetriamine (Fisher D-126); solvent: CDCl_3

C26

Triethylenetetramine



H26

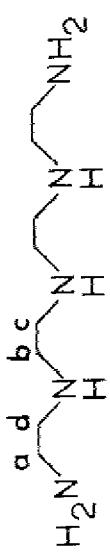


Spectrum 26—Triethylenetetramine (Eastman T4572); solvent: CDCl_3

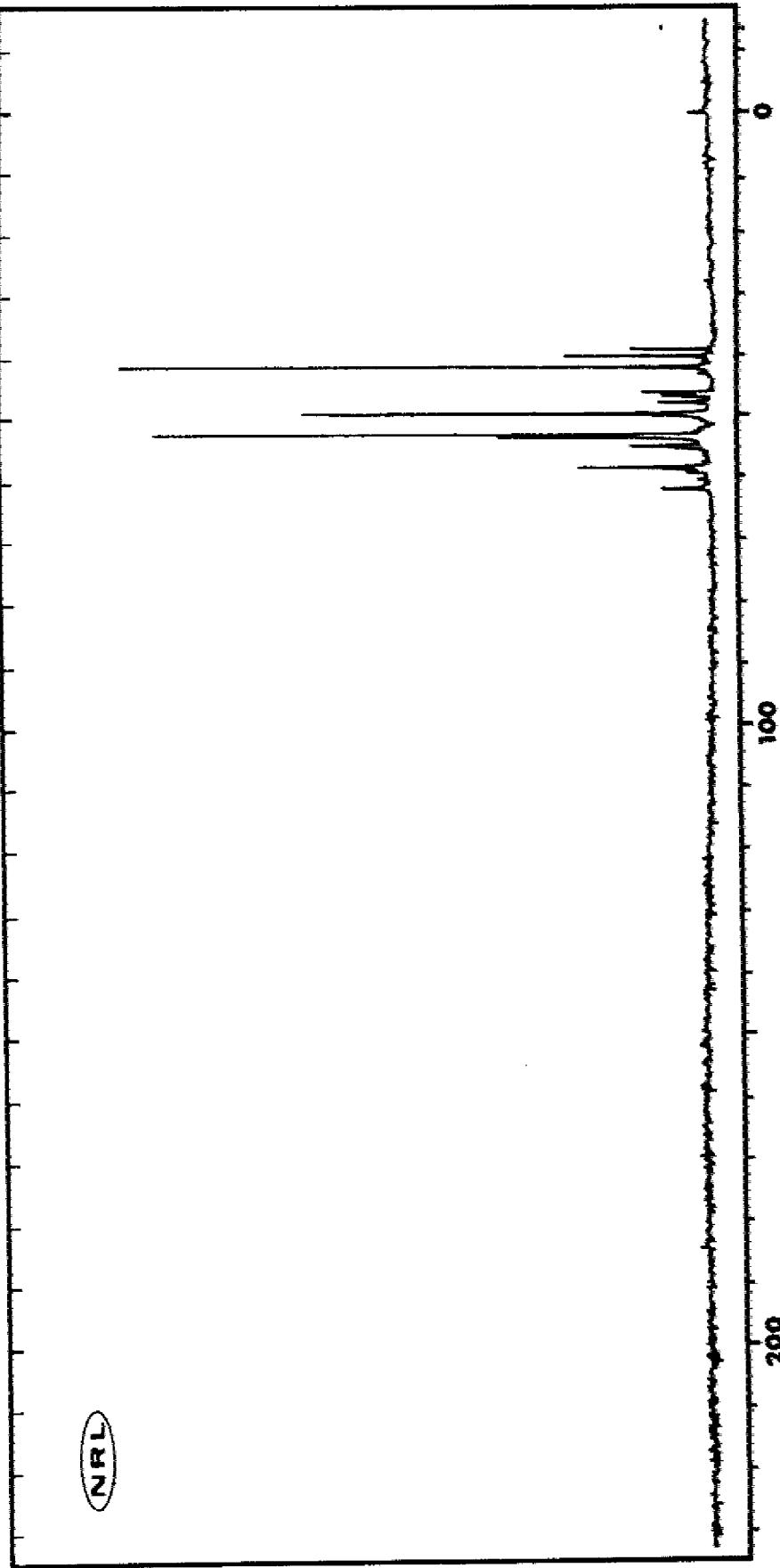
C27

Tetraethylenepentamine

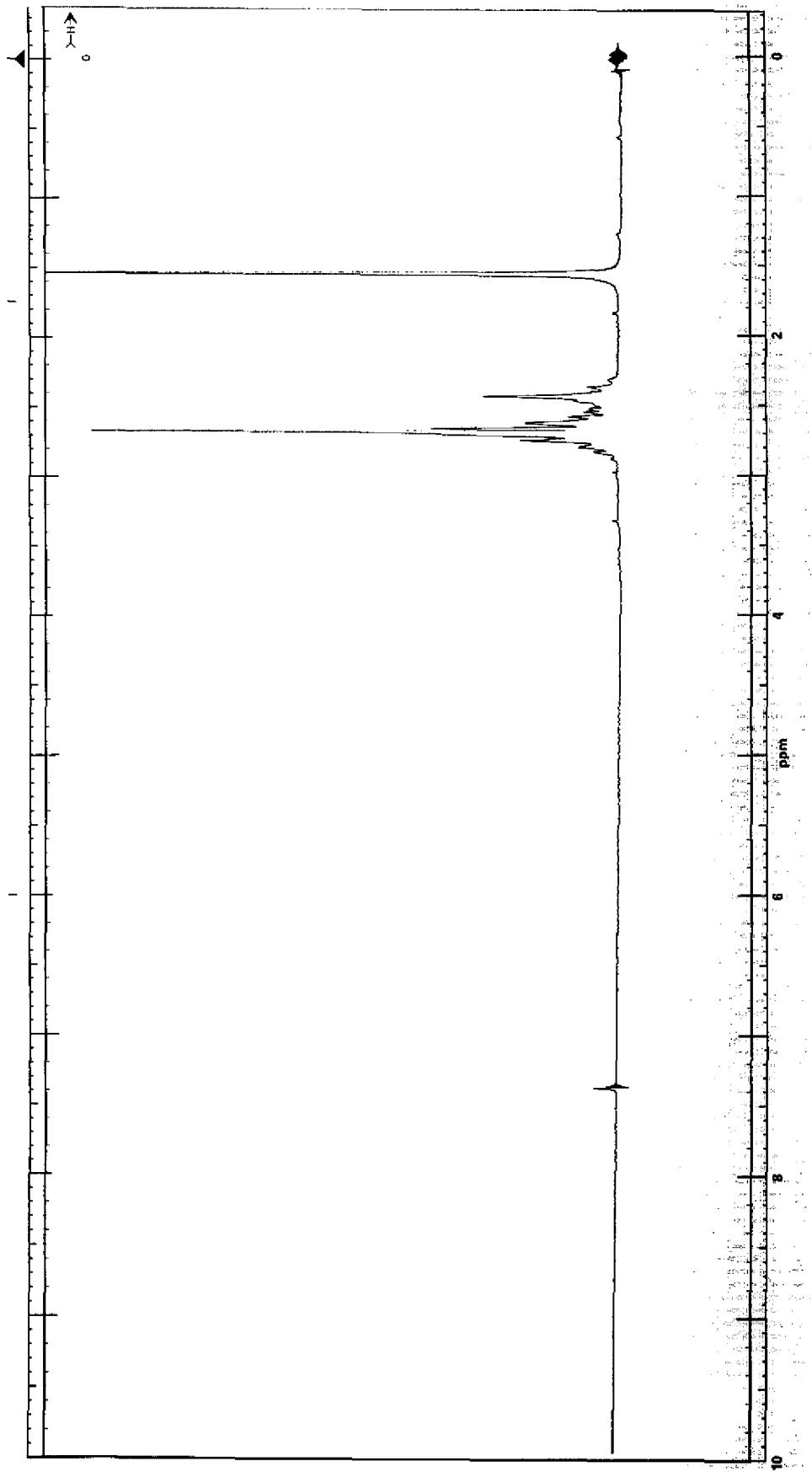
Source: Eastman T5902
Solvent: None (10 drops TMS added)



Assignments:
a 41.9
b, c 49.5
d 52.9



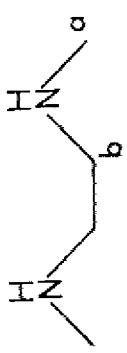
H27



Spectrum 27 — Tetraethylenepentamine (Eastman T5902); solvent: CDCl_3

C28

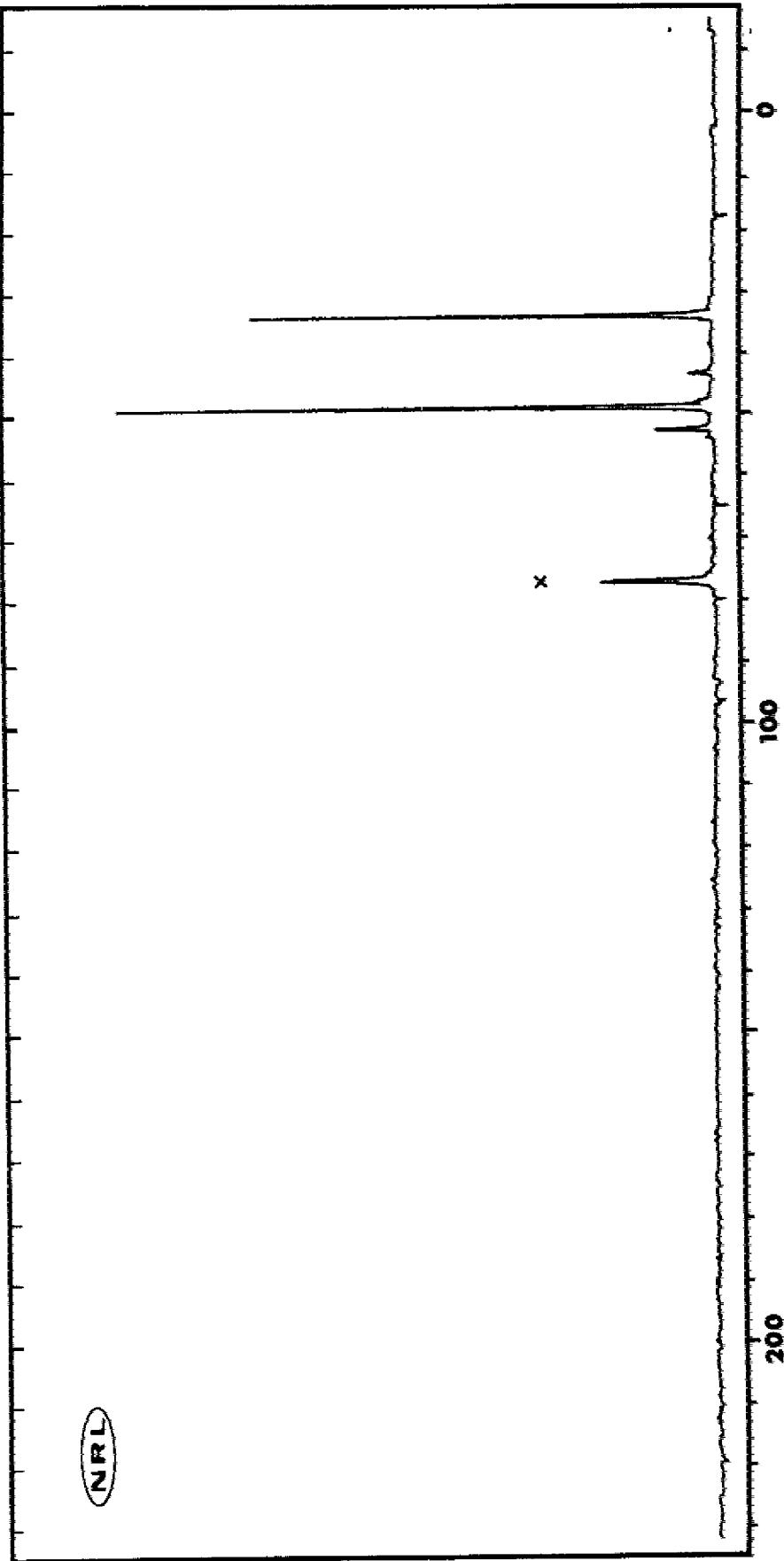
sym - Dimethylethylenediamine



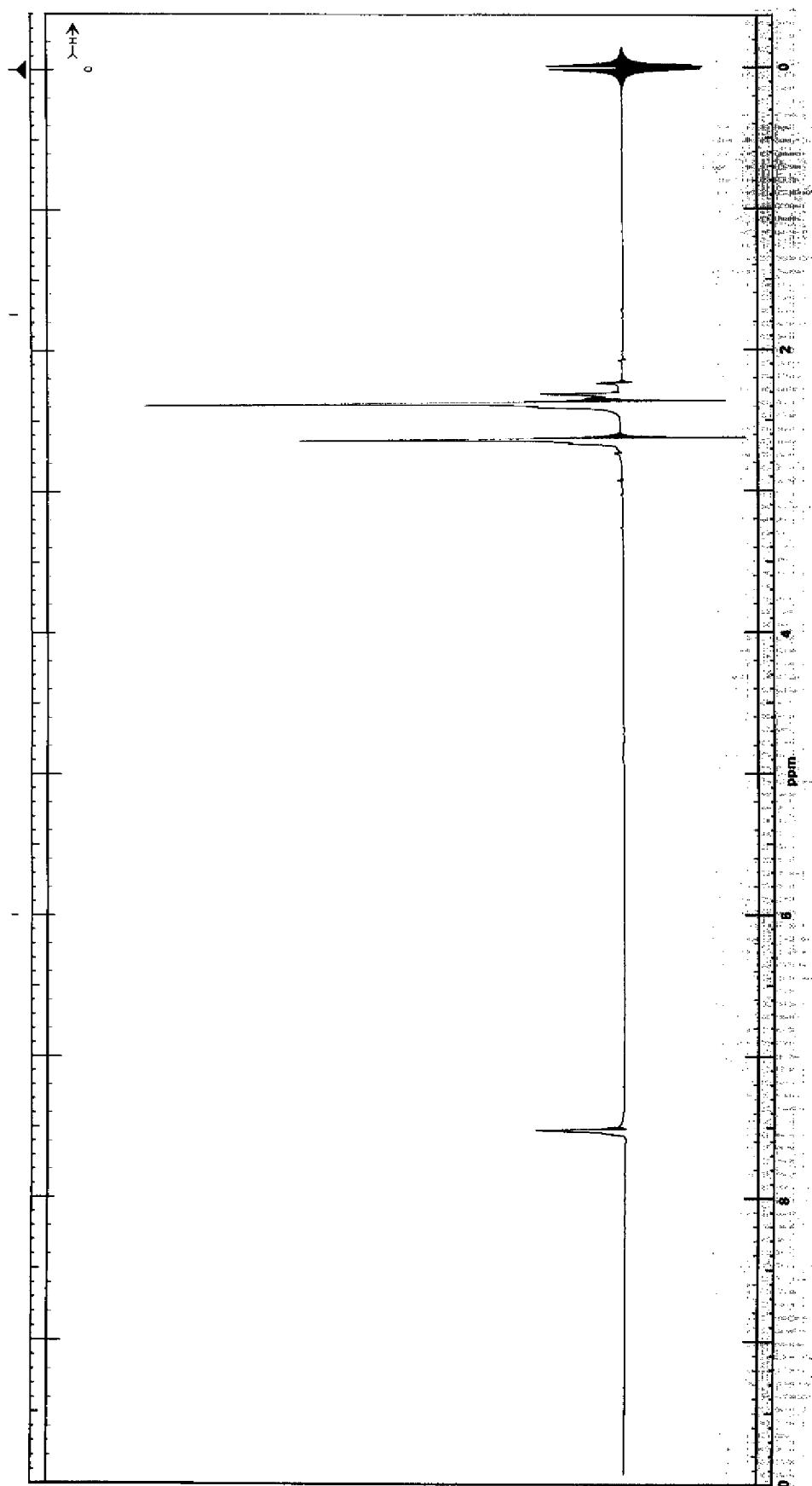
Source: Aldrich D15, 780-5

Solvent: 25% CHCl₃ ×

Assignments:
a 34.0
b 49.1



H28



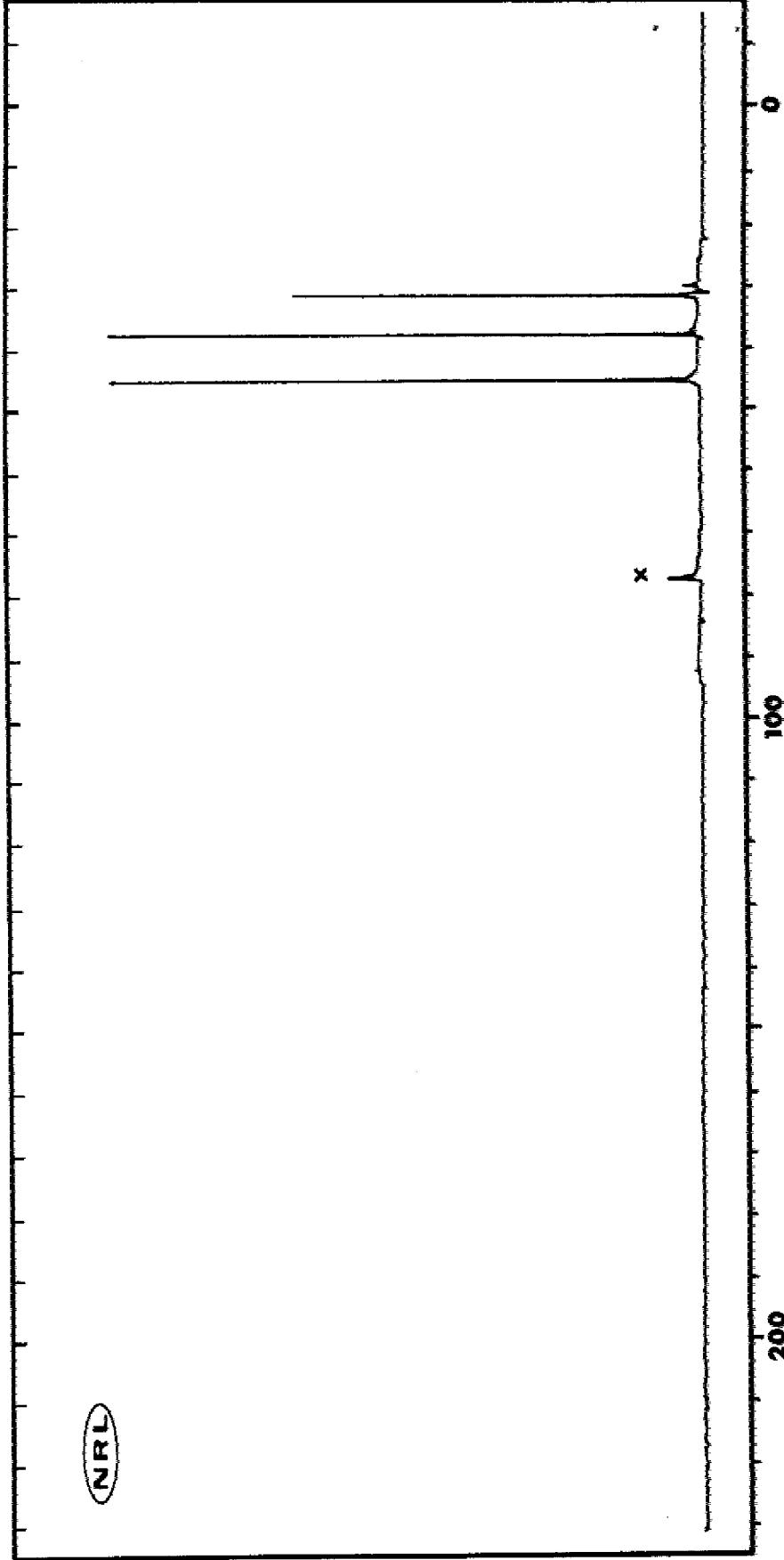
Spectrum 28 — sym-Dimethylethylenediamine (Aldrich D15,780-5); solvent: CDCl_3

C29

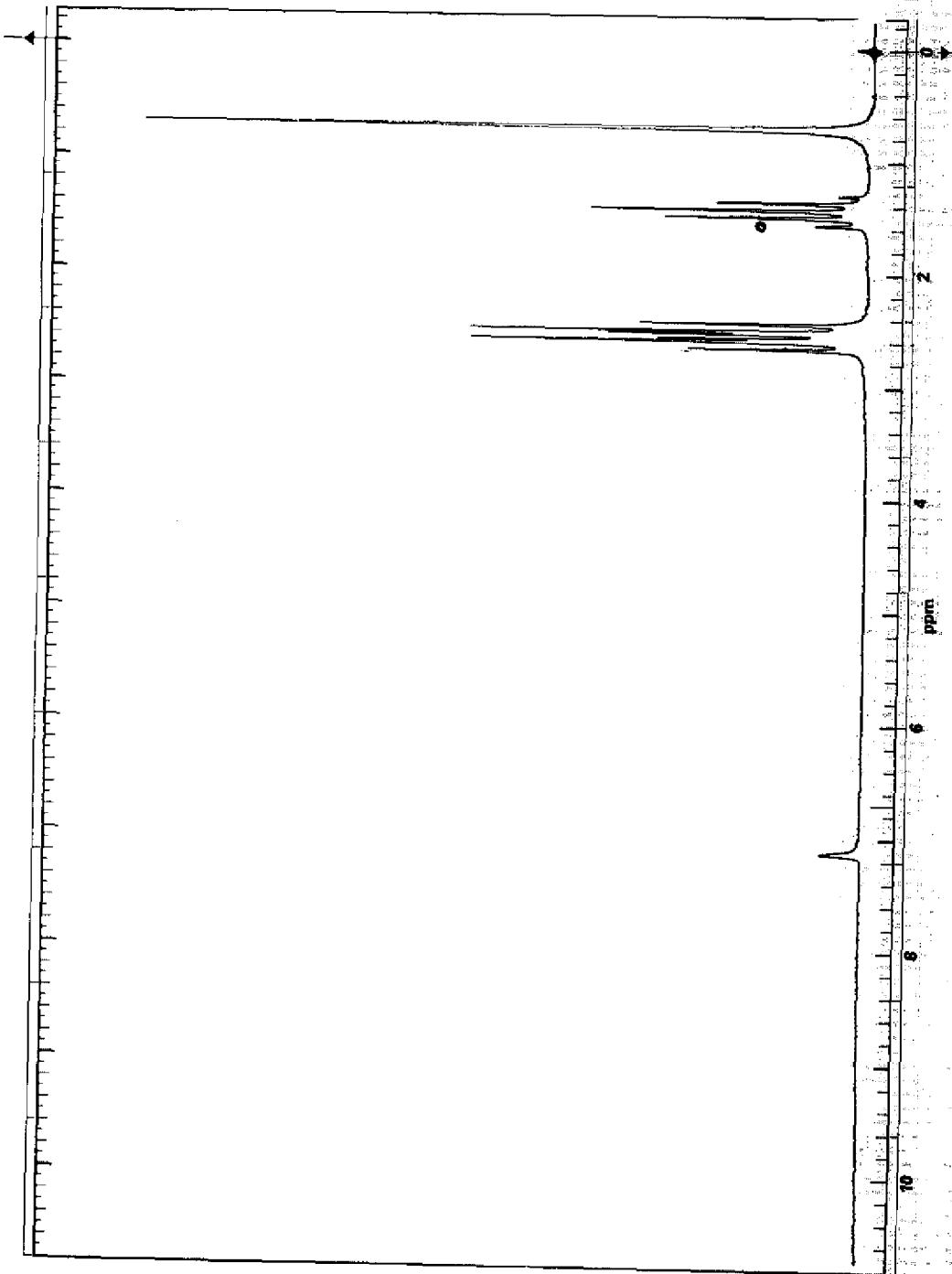
3,3'-Iminobispropylamine

Source: American Cyanamid Company

Solvent: 10% CHCl₃ ×



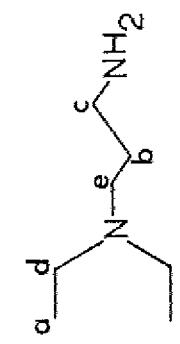
H29



Spectrum 29 — 3,3'-Imino-bis-propylamine (American Cyanamid Company); solvent: benzene-d₆

C30

3-Diethylaminopropylamine

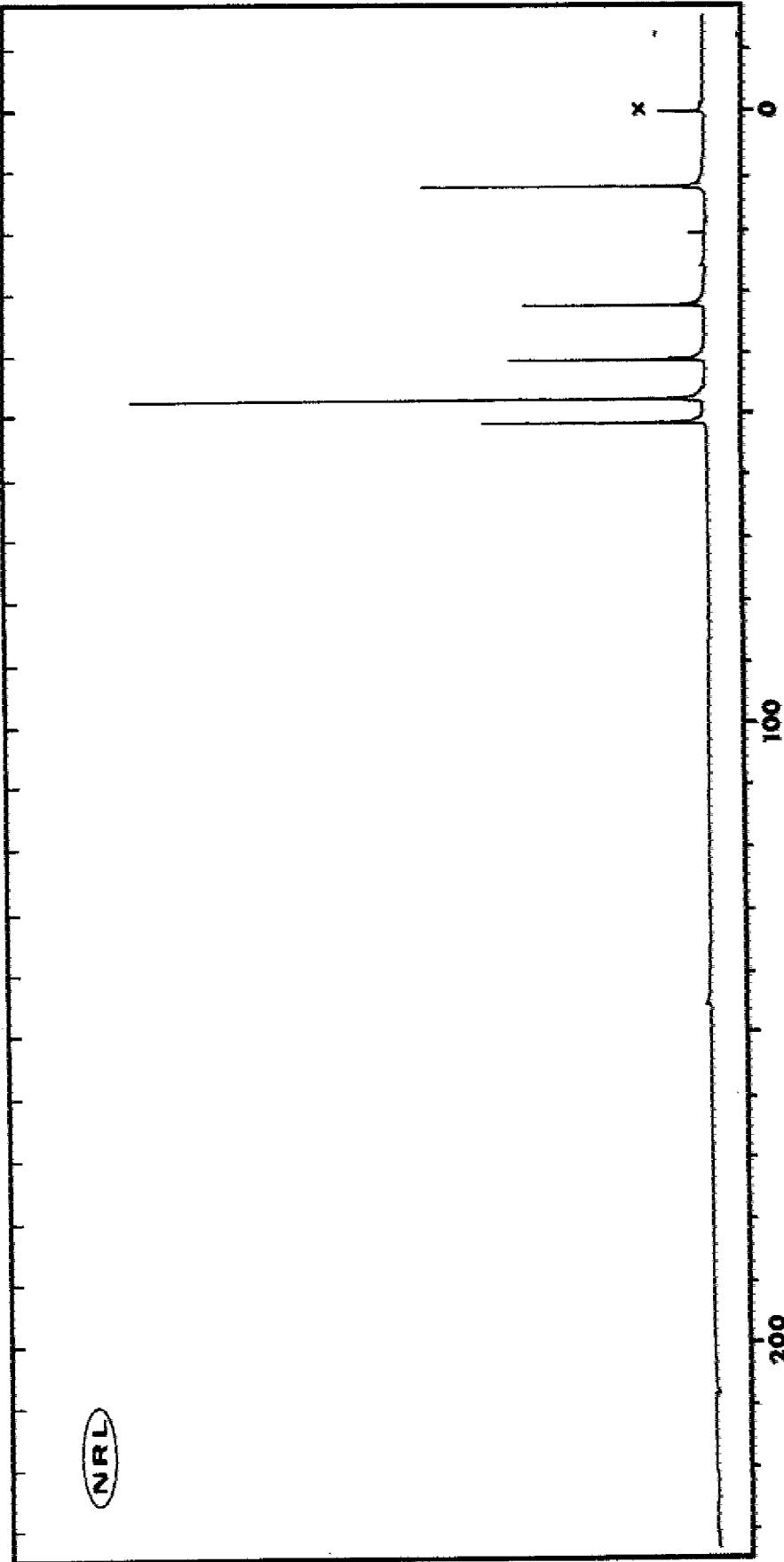


Assignments:

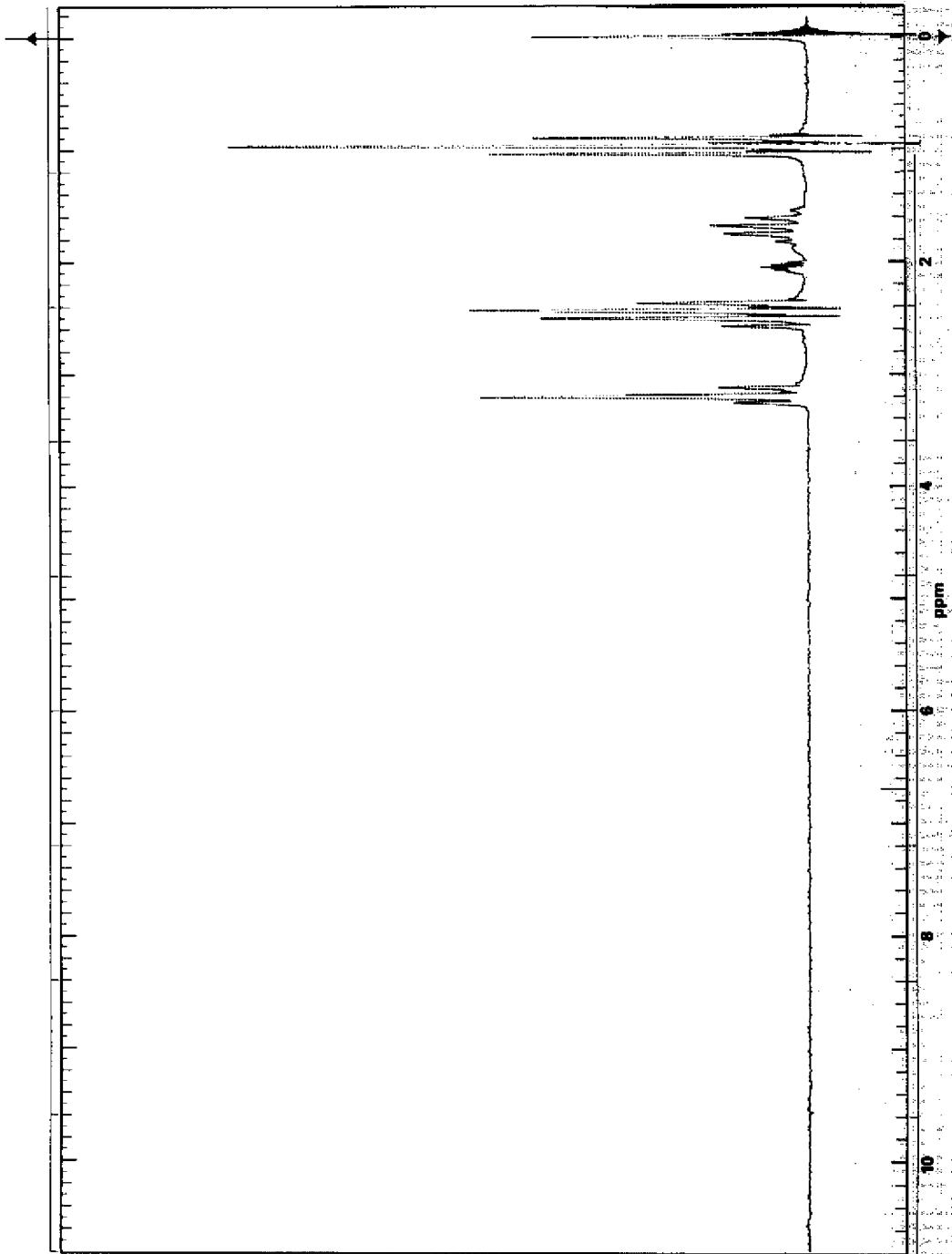
a	12.4
b	31.9
c	40.8
d	47.3
e	51.2

Source: Aldrich # D8, 920-4

Solvent: 10% TMS x



H30



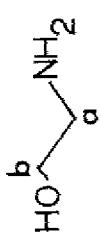
Spectrum 30 — 3-Diethylaminopropylamine (Aldrich D8,920-4); solvent: acetone-d₆

C31

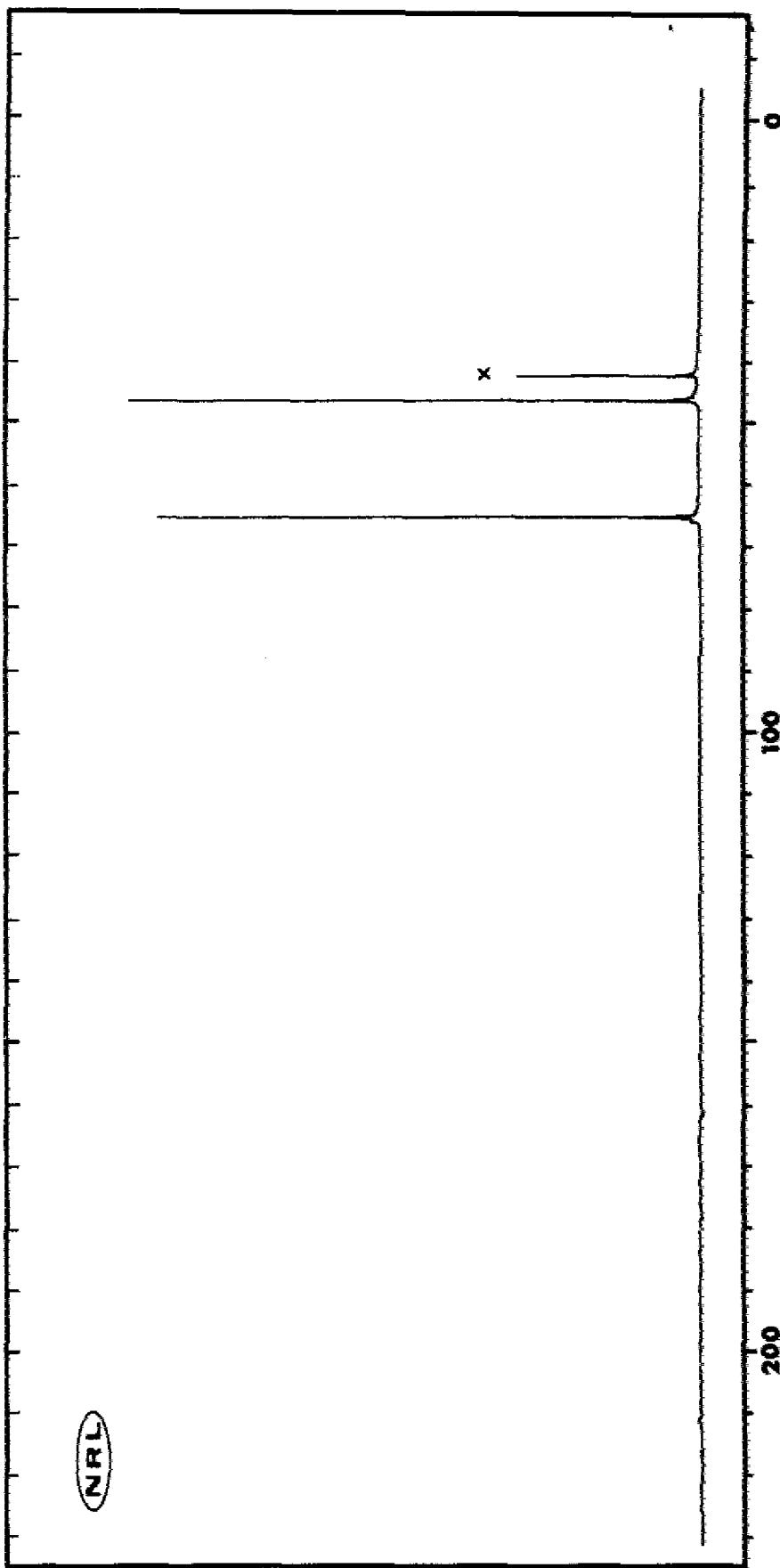
2-Aminoethanol

Source: Eastman 1597

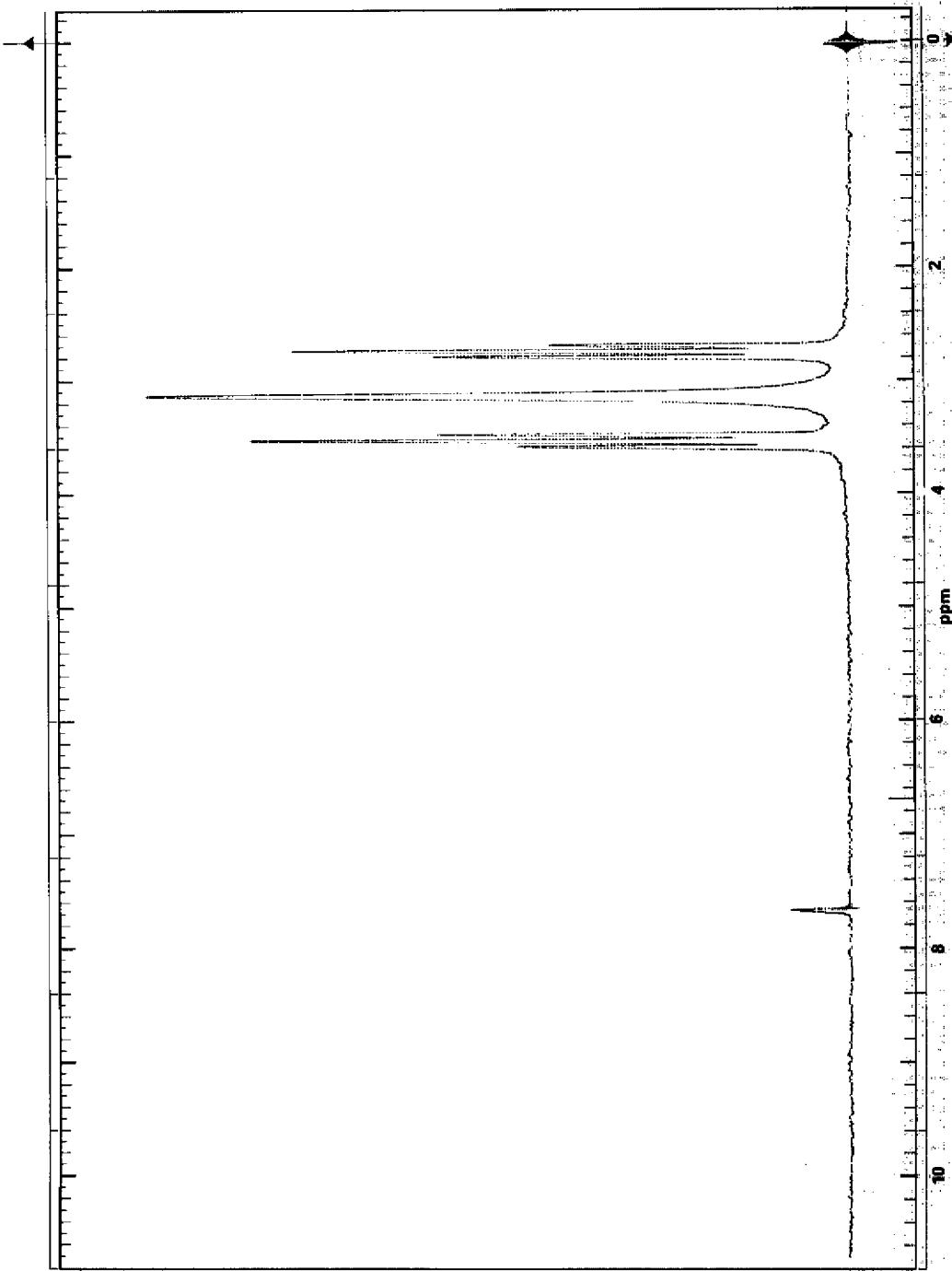
Solvent: 25% DMSO \times



Assignments:
a 45.9
b 64.7



H31

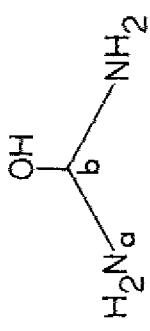


Spectrum 31 — 2-Aminoethanol (Eastman 1597); solvent: CDCl_3

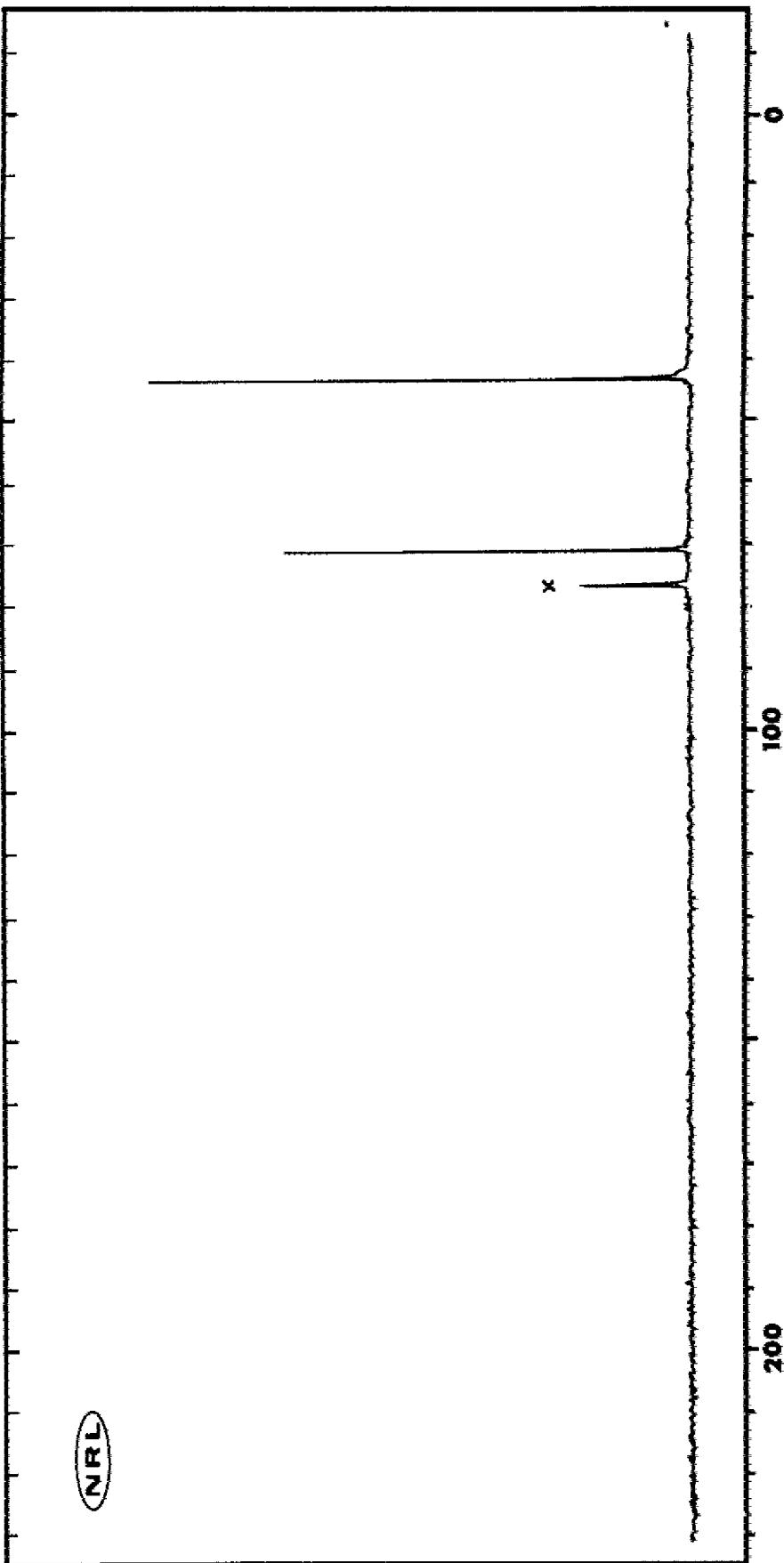
C32

1,3-Diamino-2-propanol

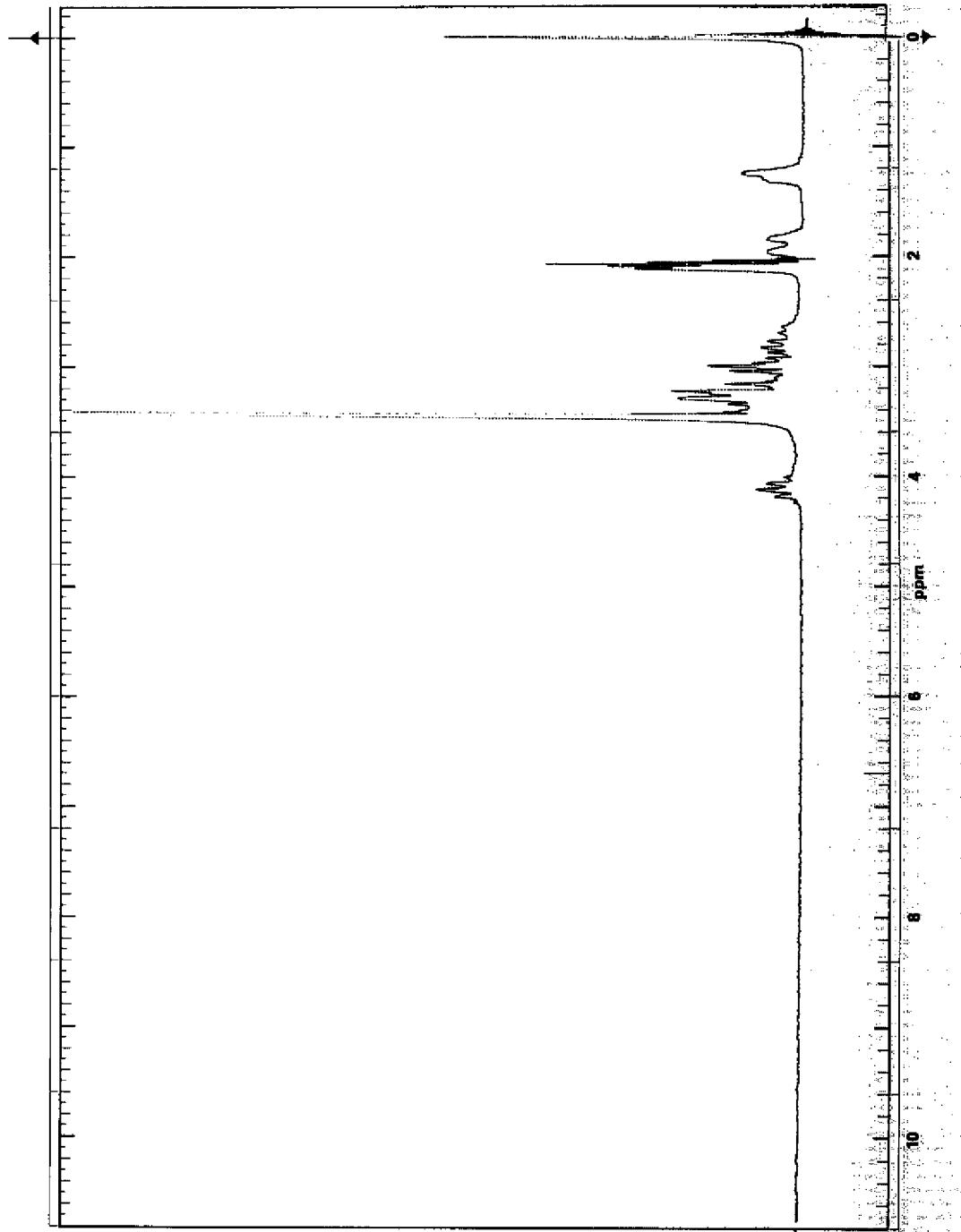
Source: Aldrich D1860
Solvent: 25% CHCl₃ ×



Assignments:
a 43.8
b 71.4



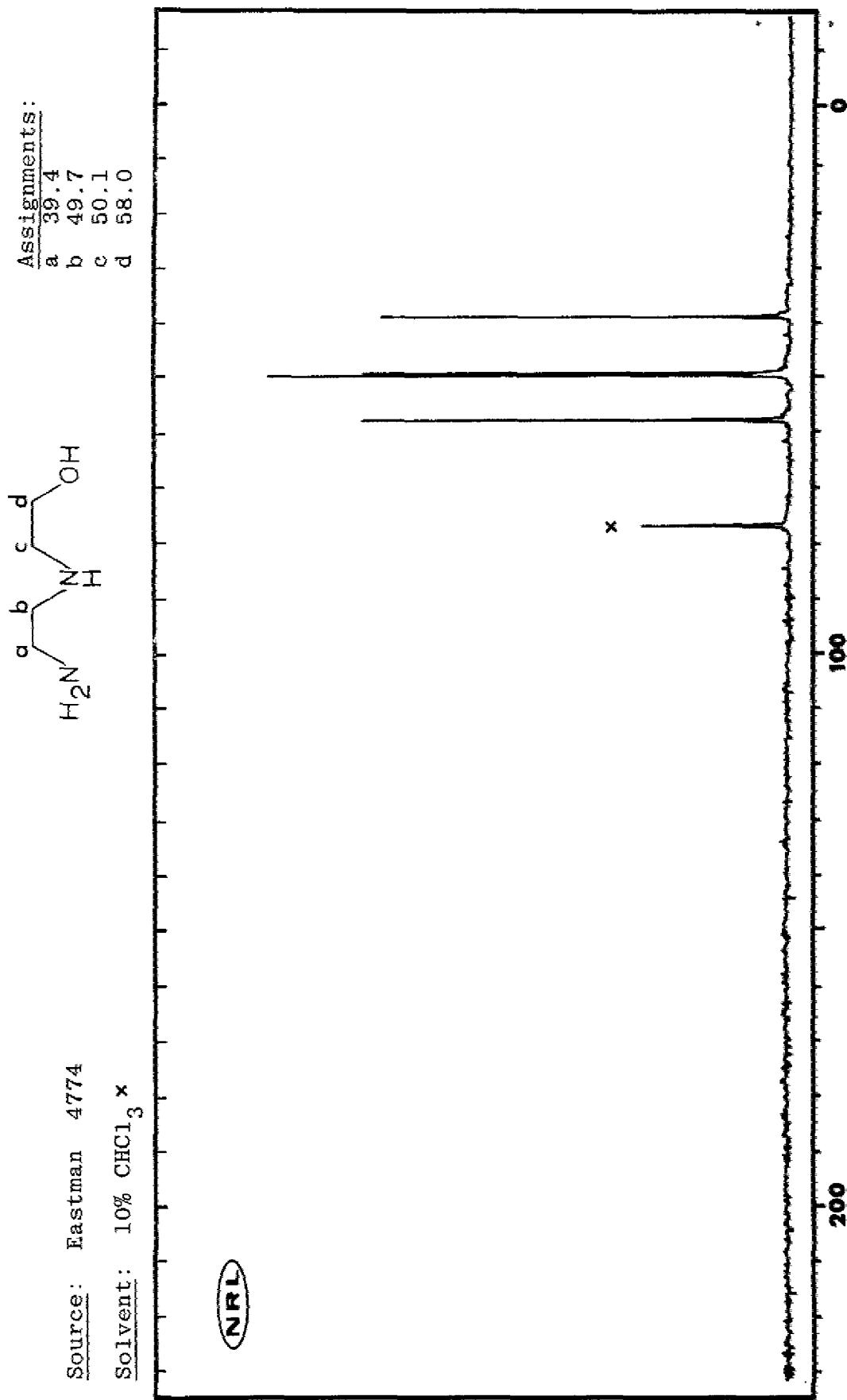
H32



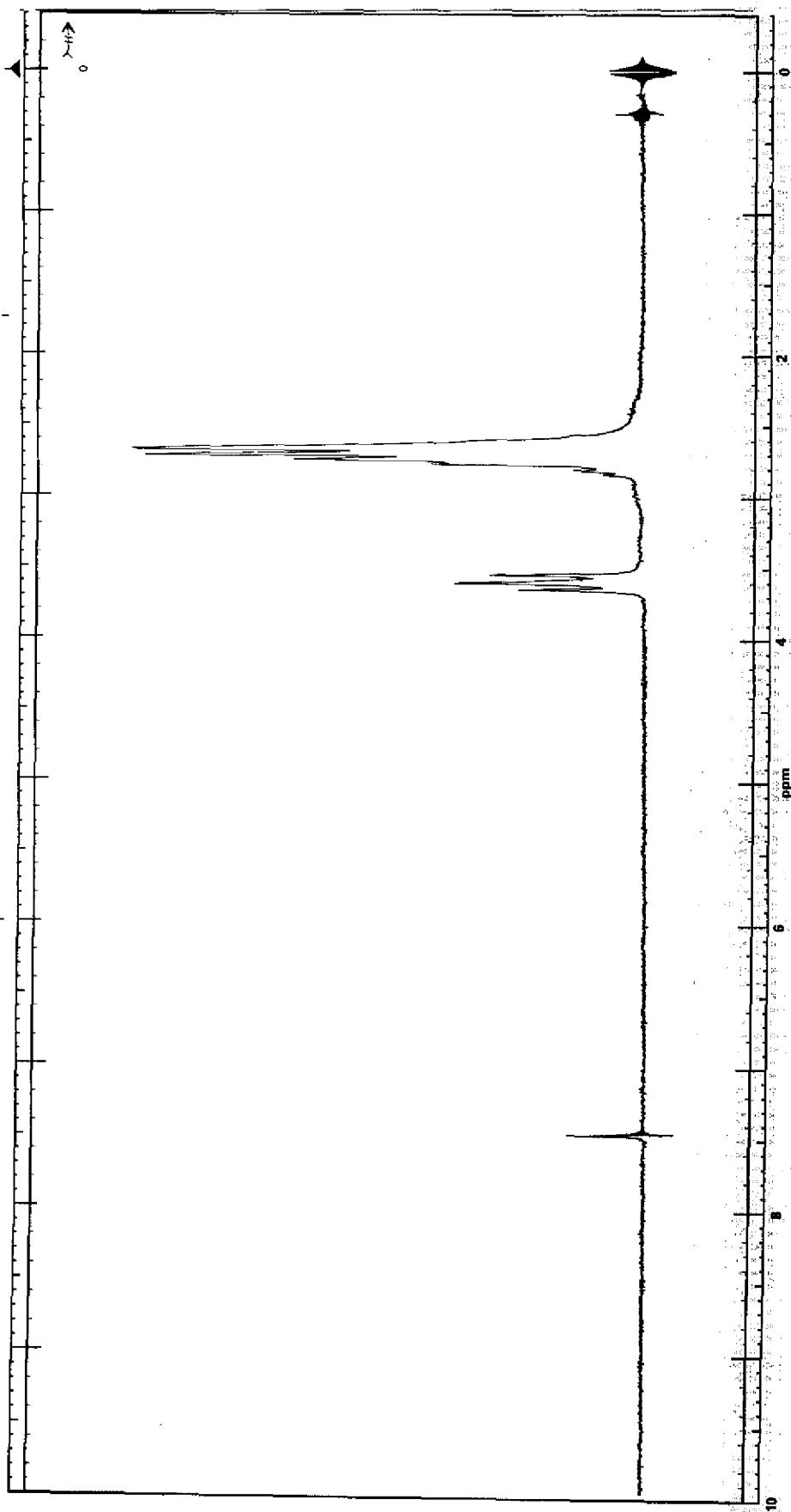
Spectrum 32 – 1,3-Diamino-2-propanol (Aldrich D1860); solvent: acetone-d₆

C33

2-(2-Aminoethylamino)ethanol



H33



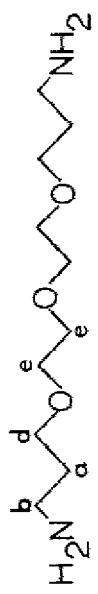
Spectrum 33 — 2-(2-Aminoethylamino)ethanol (Eastman 4774); solvent: CDCl₃

C34

Polyglycol Diamine

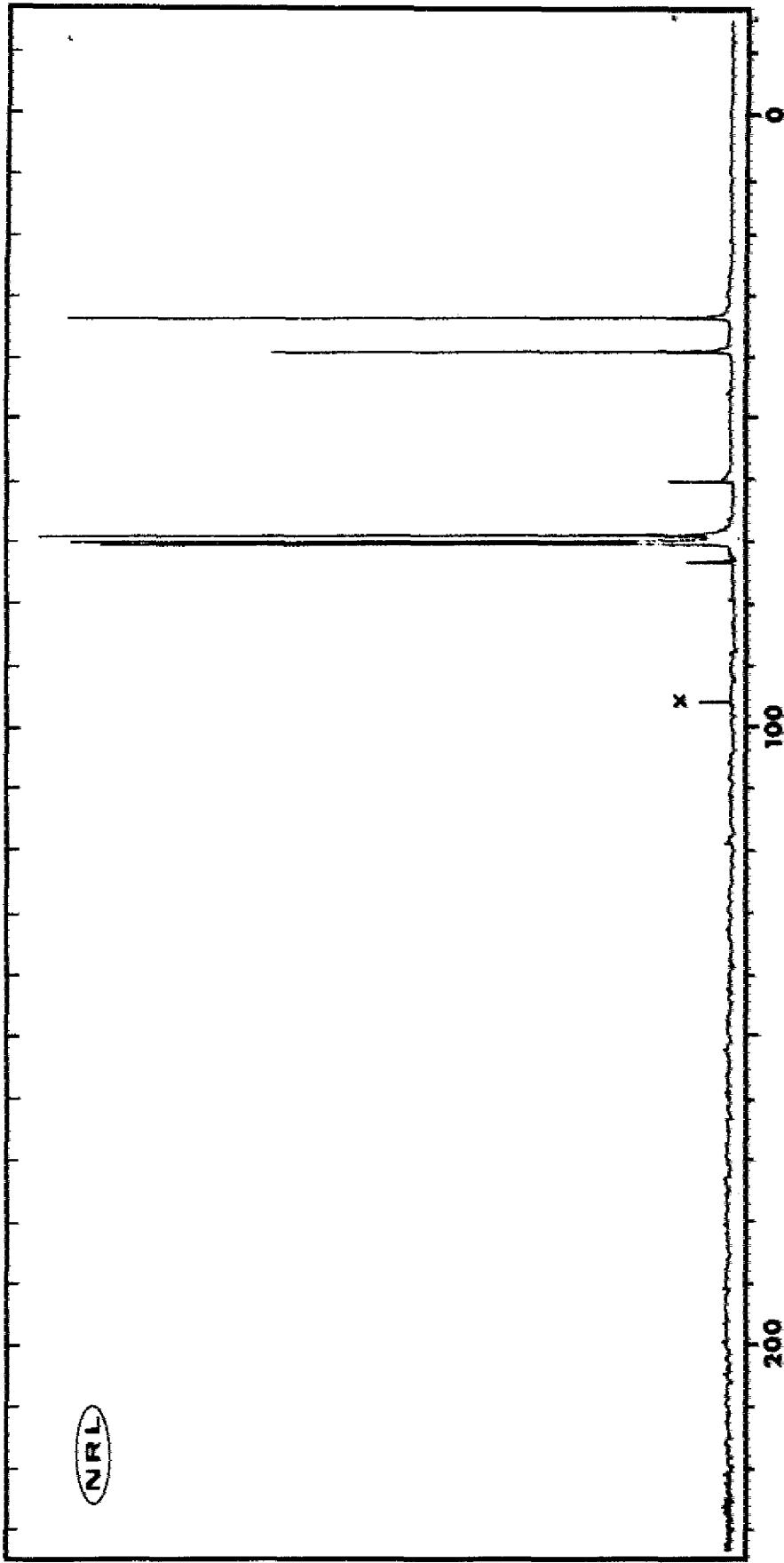
Assignments:

a	33.7
b	39.3
c	60.4
d	69.1
e	70.1
f	70.5
	73.4

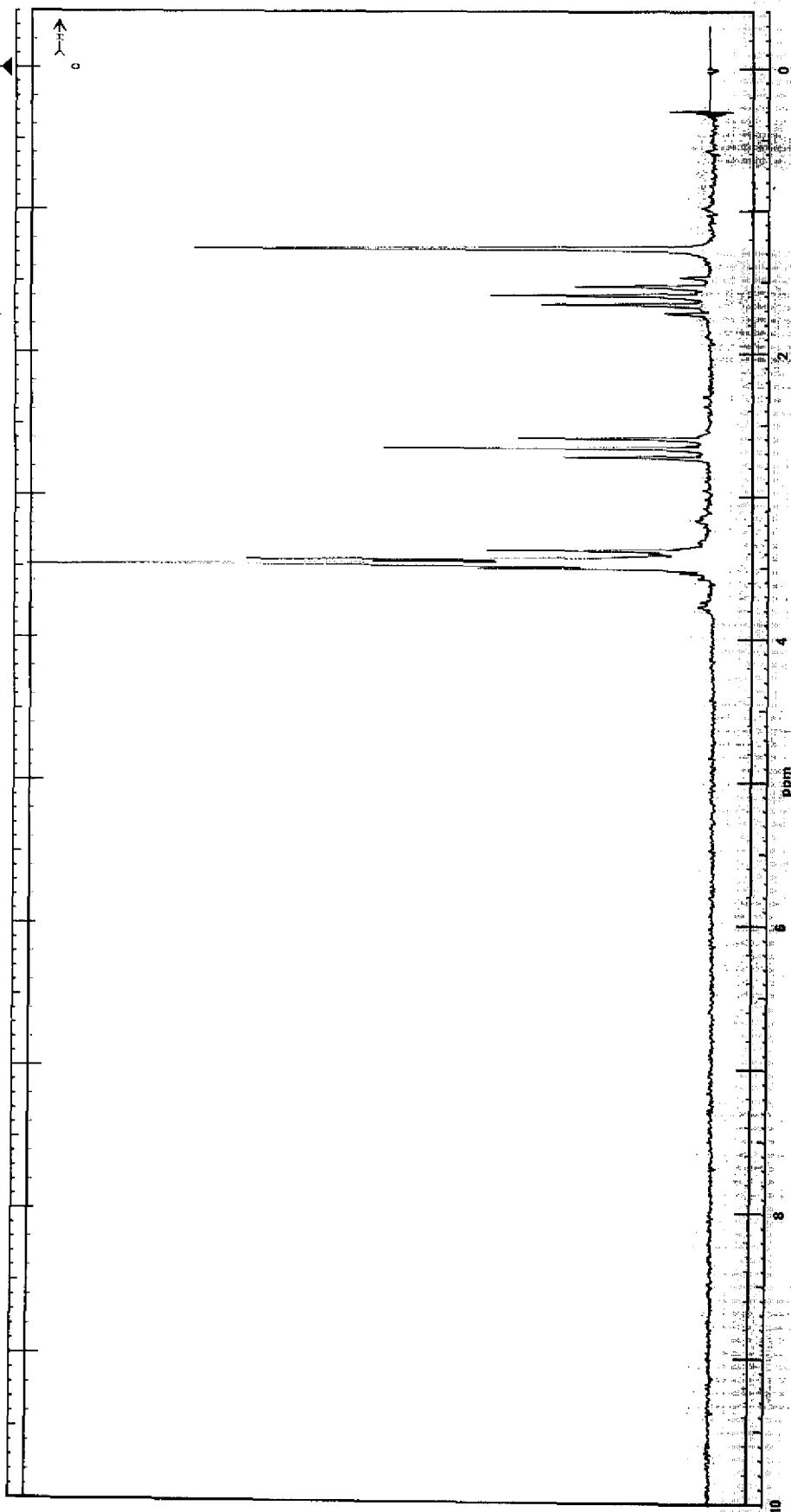


Source: Union Carbide H-221

Solvent: 20% CCl_4 x



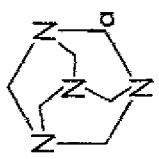
H34



Spectrum 34 — Polyglycoldiamine (Union Carbide H-221); solvent: CCl_4

C35

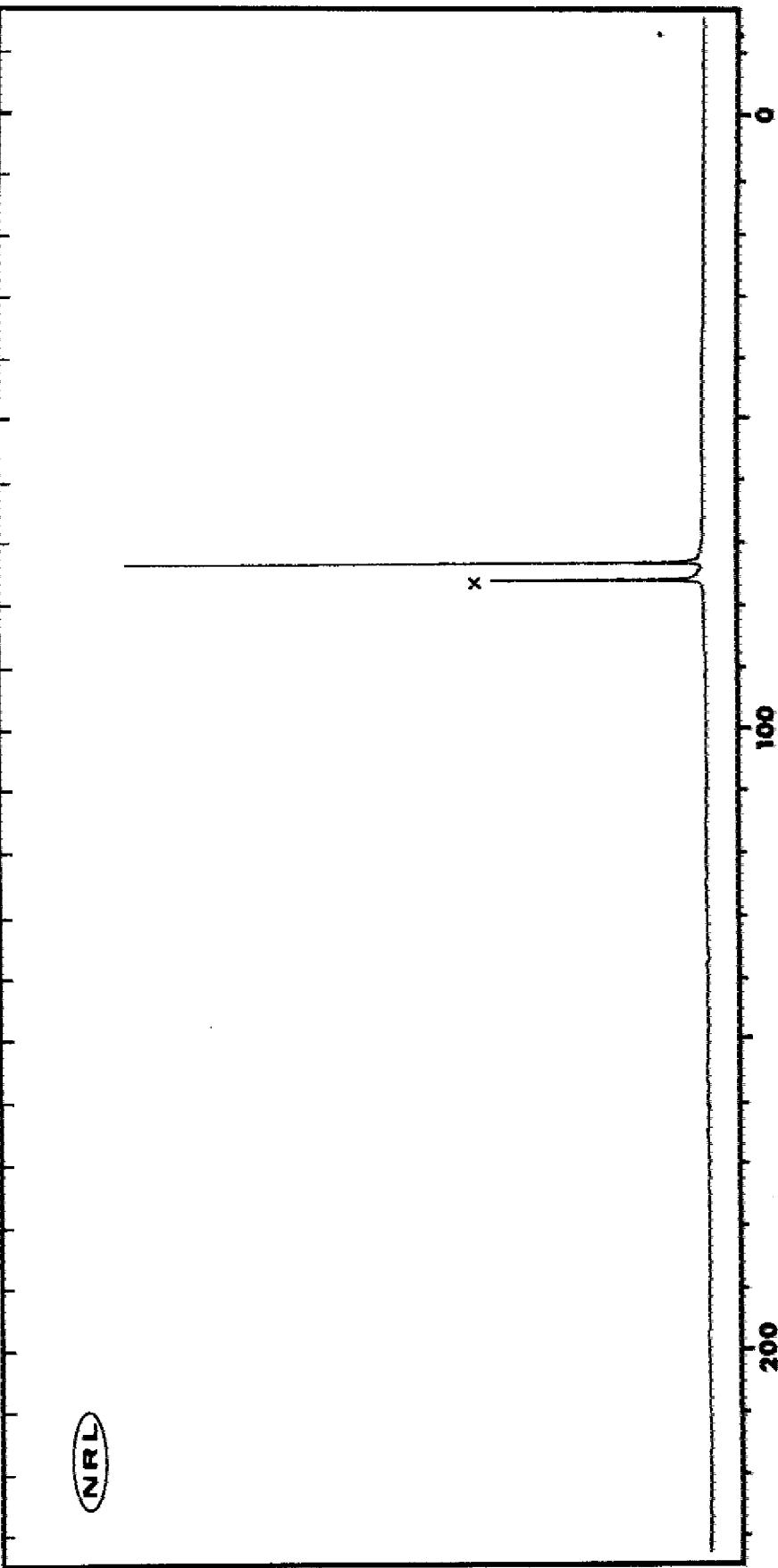
Hexamethylenetetramine



Source: Eastman 145

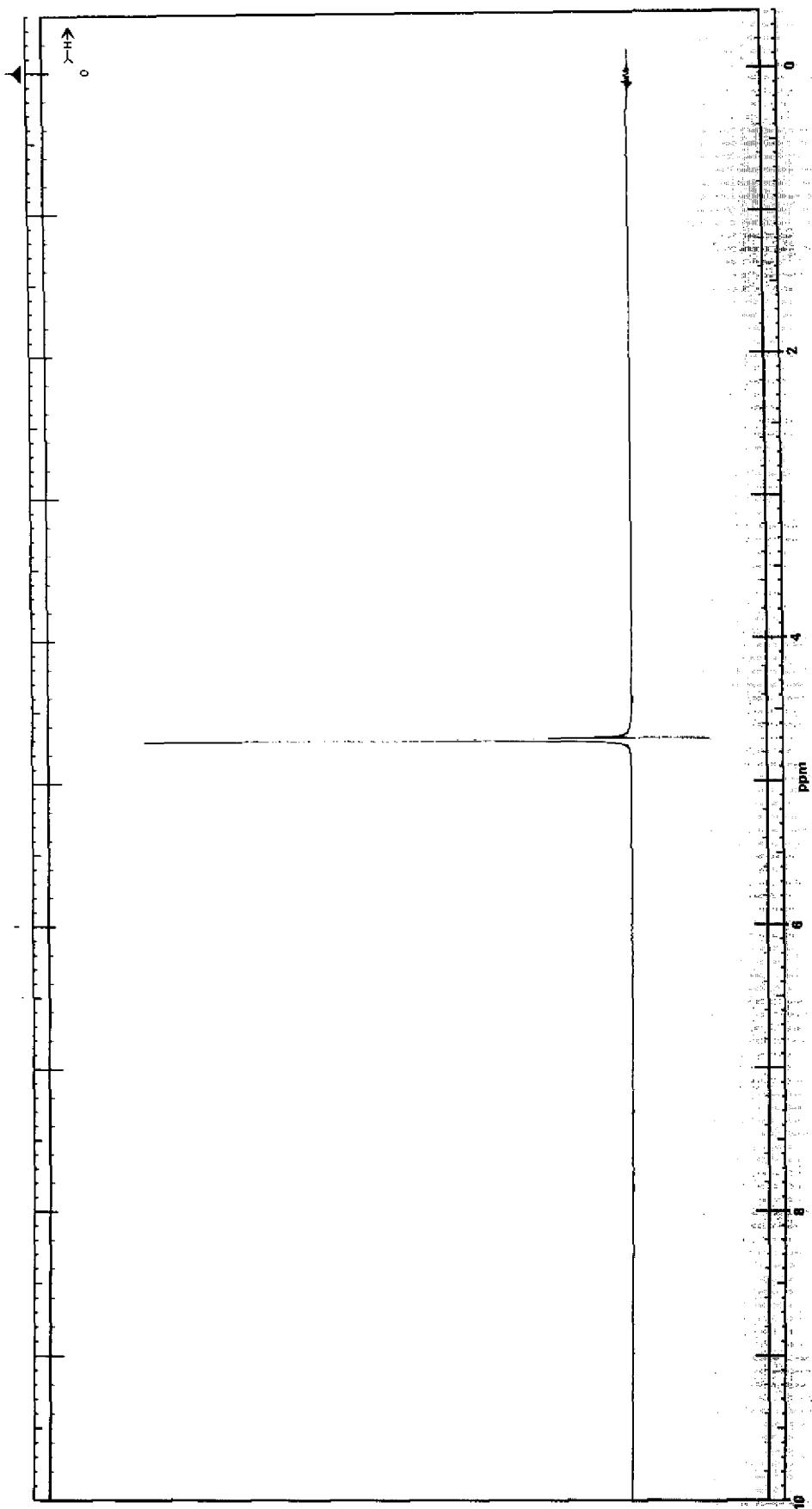
Solvent: 75% CHCl₃ x

Assignments:
a 74.4



(NRL)

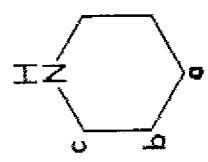
H35



Spectrum 35 — Hexamethylenetetramine (Eastman 145); solvent: CDCl_3

C36

Piperidine



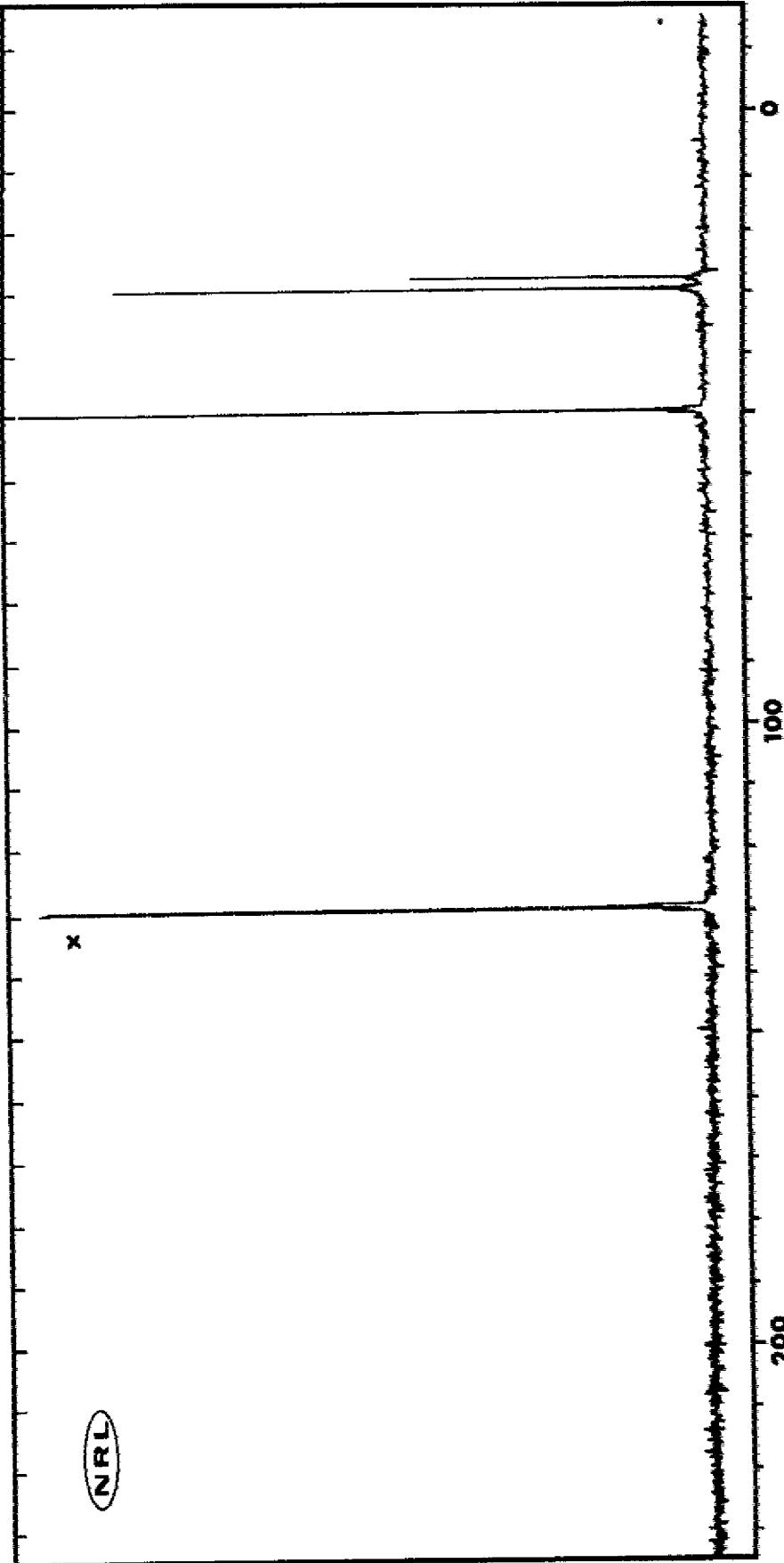
Assignments:

- a 26.6
- b 28.5
- c 48.6

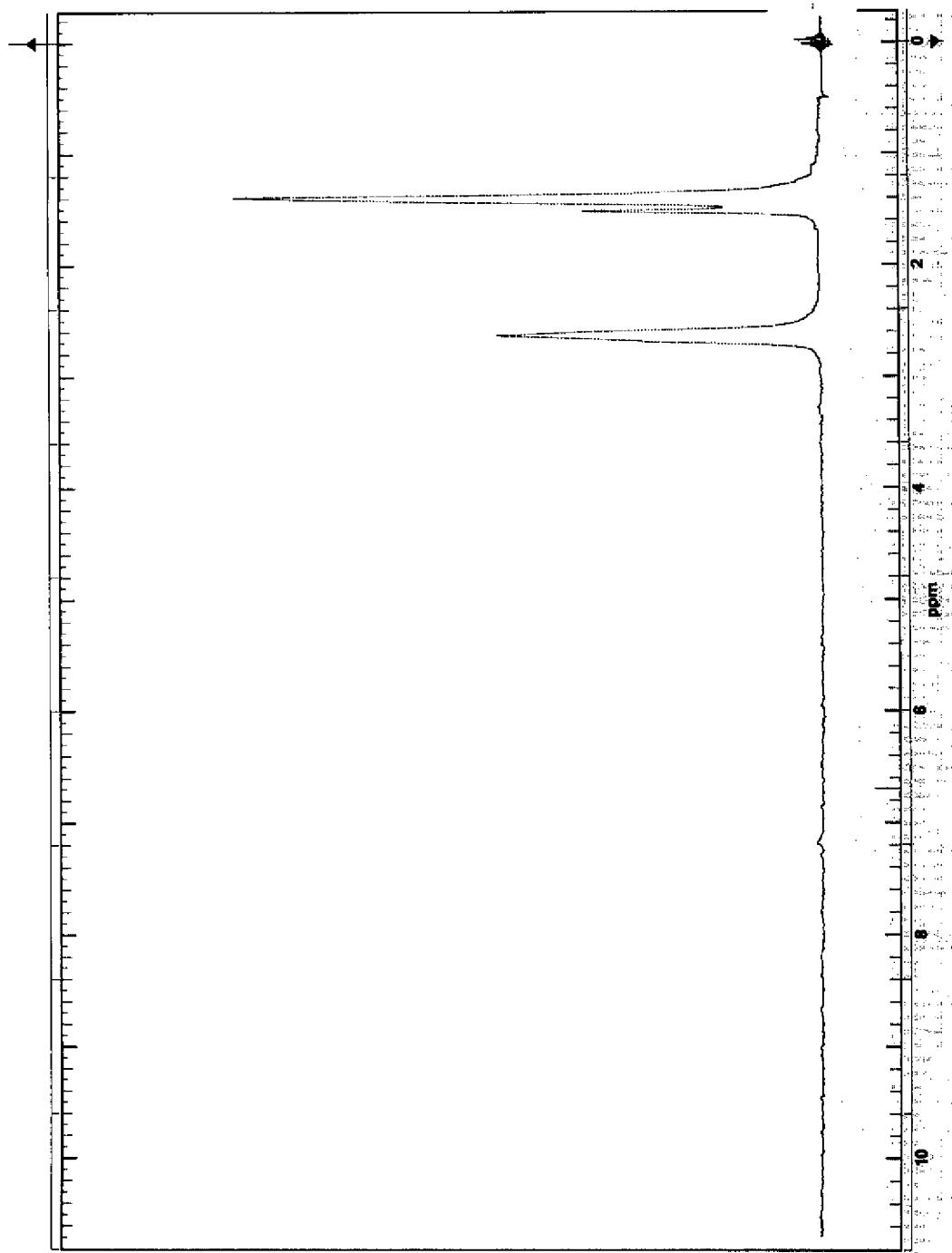
Source: Fisher P-125

Solvent: 75% benzene **x**

NRL



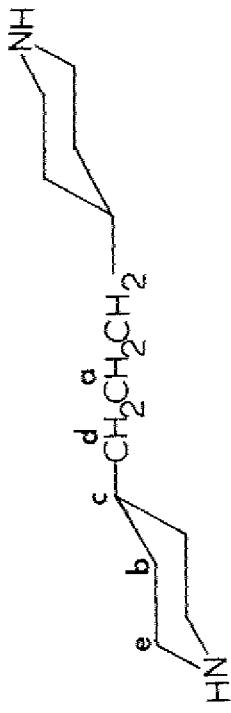
H36



Spectrum 36 — Piperidine (Fisher P-125); solvent: 75% benzene-d₆

C37

4,4'-Trimethylene-dipiperidine

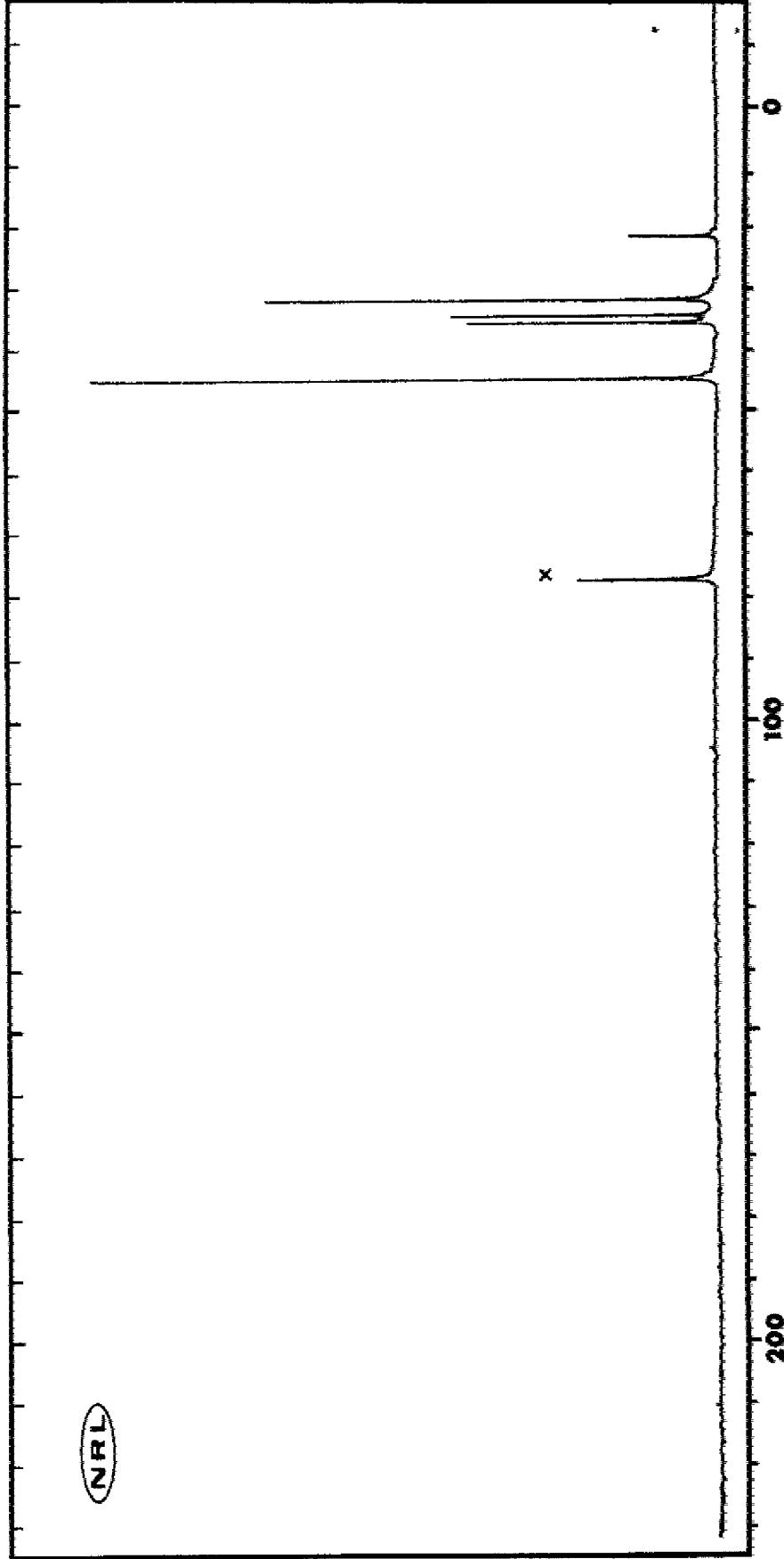


Assignments:

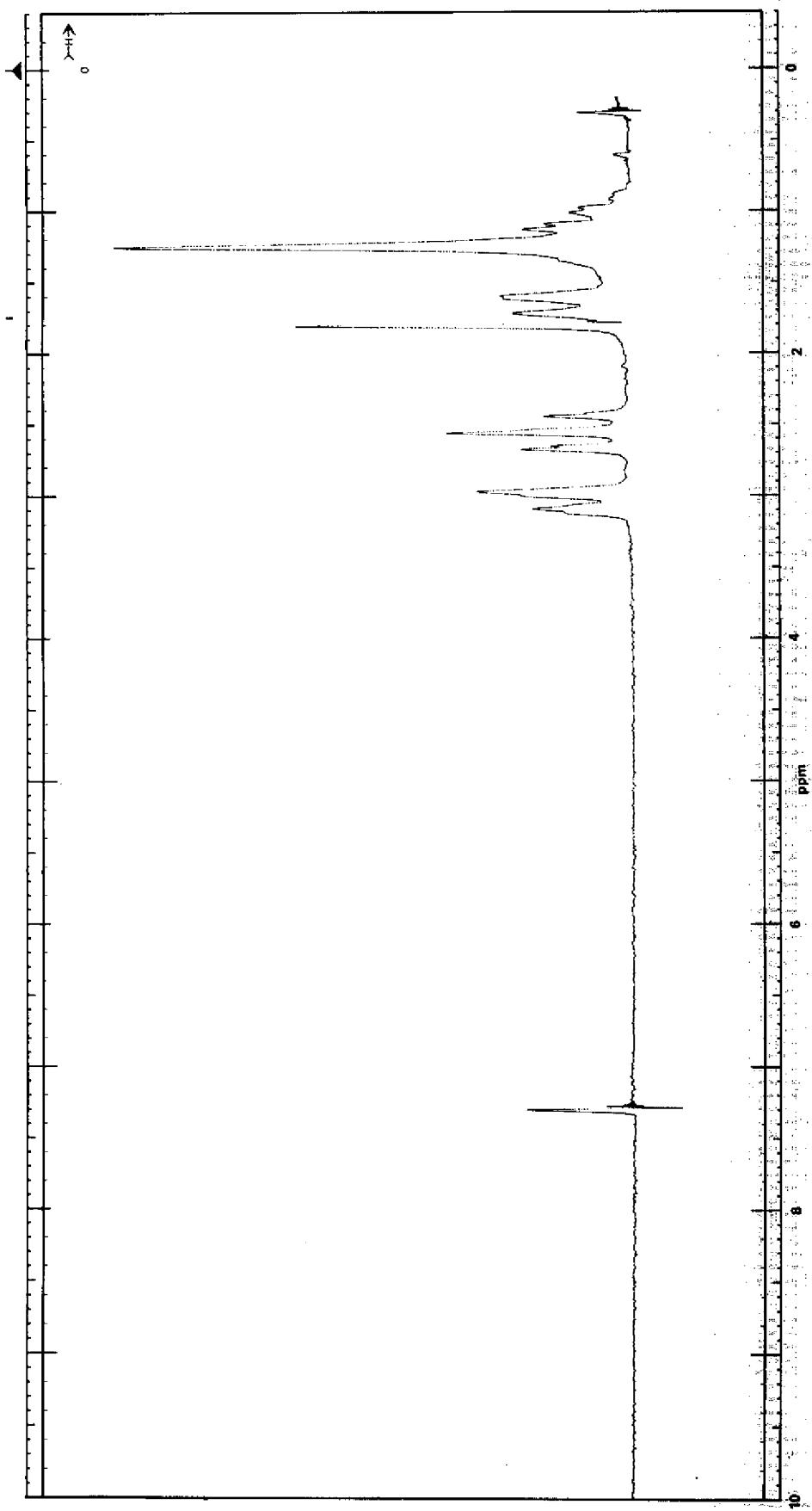
- | | |
|---|------|
| a | 21.2 |
| b | 31.5 |
| c | 34.2 |
| d | 35.4 |
| e | 44.6 |

Source: Reilly Tar & Chemical Corp. "4-DI-PIP"

Solvent: CHCl₃ *



H37



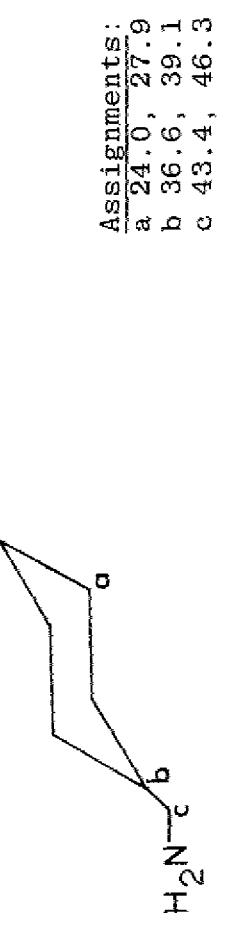
Spectrum 37 — 4,4'-Trimethylene-dipiperidine (Reilly Tar & Chemical Corp. "4-Di-PIP"); solvent: CDCl_3

C38

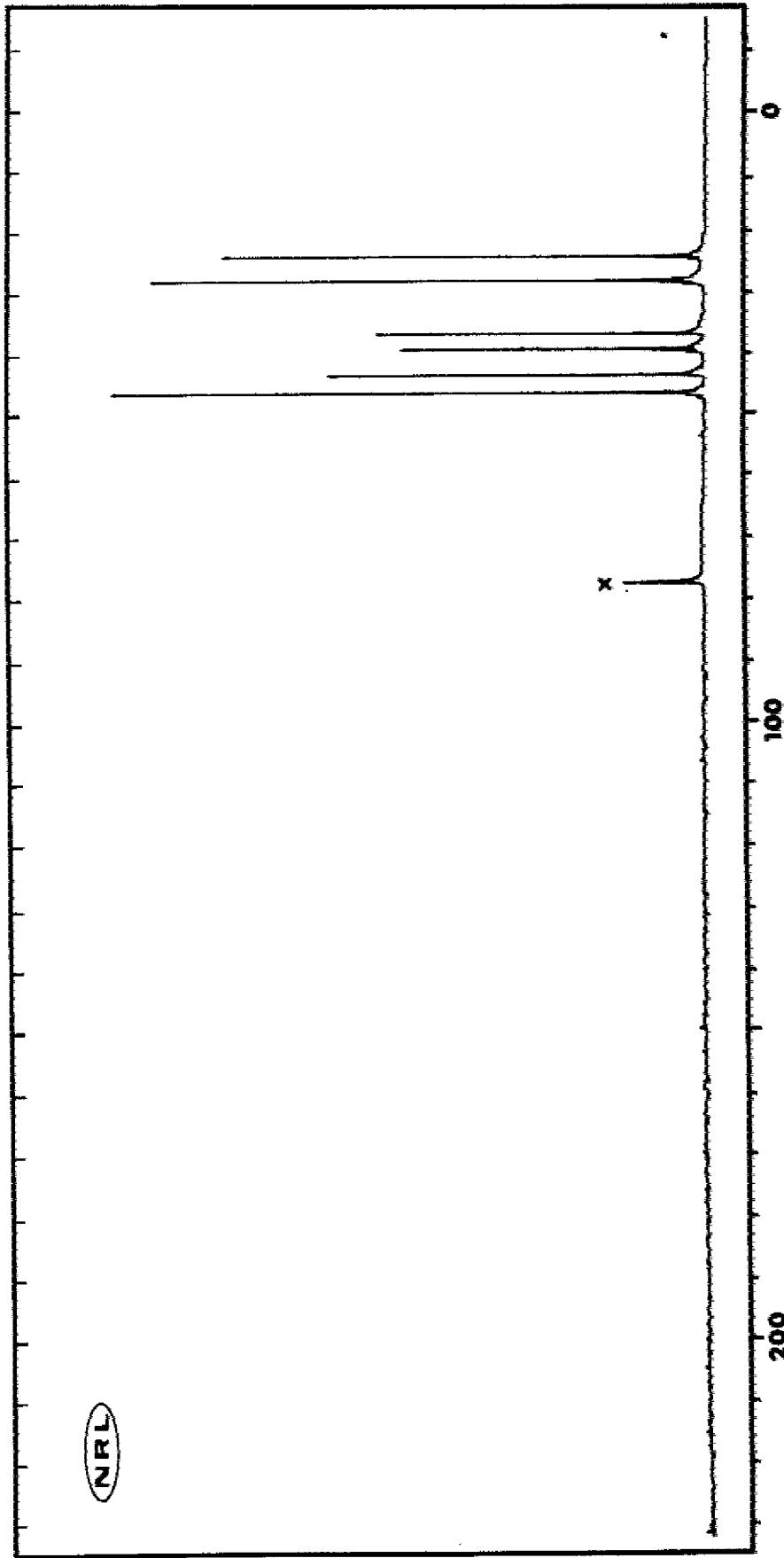
1,4 Cyclohexanebis(methylamine)



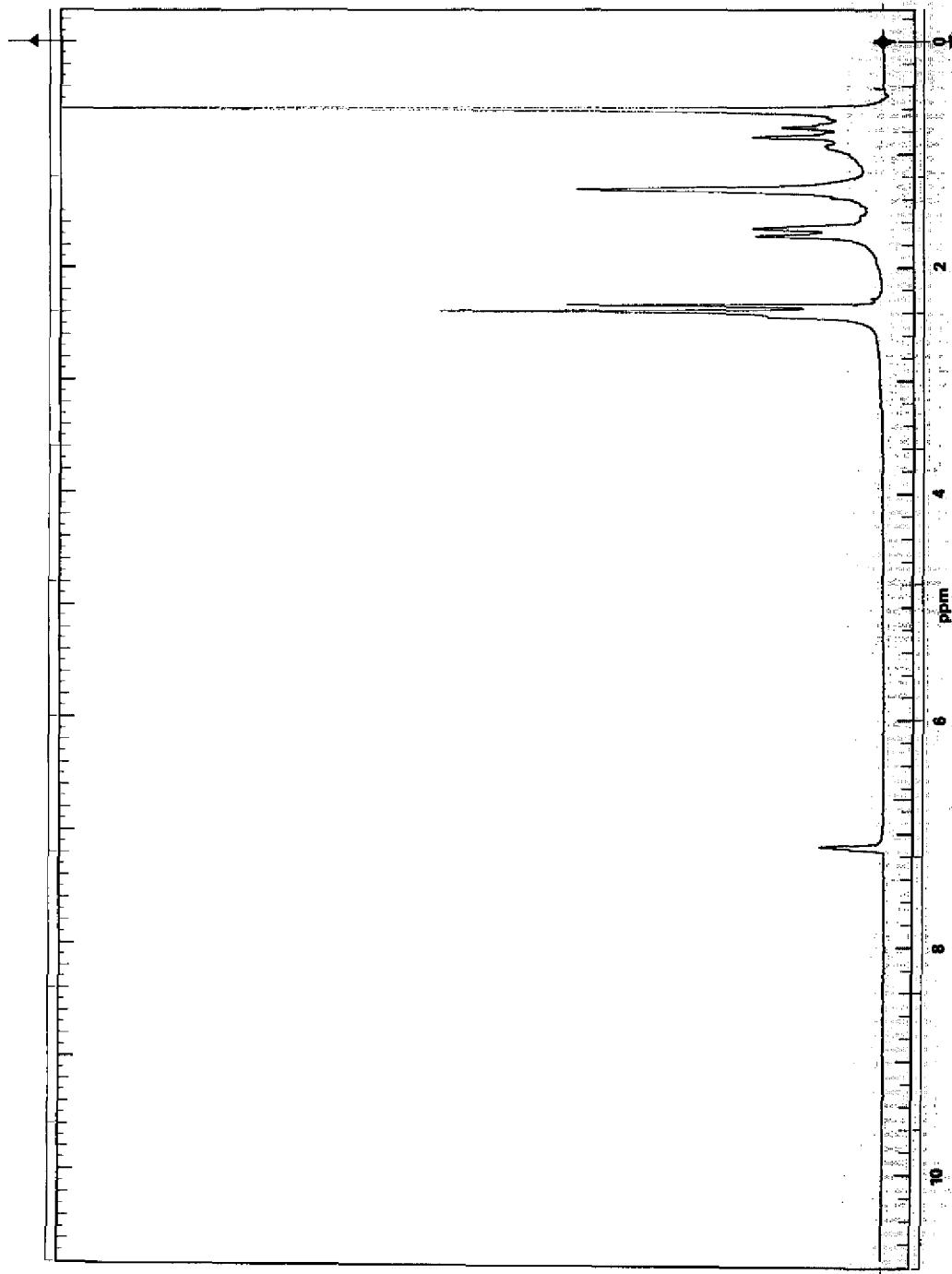
Source: Eastman P7562
Solvent: 10% CHCl_3 *



Assignments:
a 24.0, 27.9
b 36.6, 39.1
c 43.4, 46.3



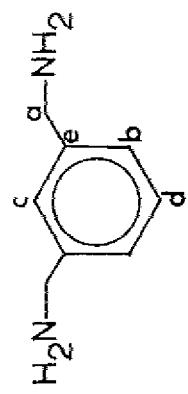
H38



Spectrum 38 — 1,4-Cyclohexane-*bis*(methylamine) (Eastman P7562); solvent: benzene-d₆

C39

m-Xylenediamine



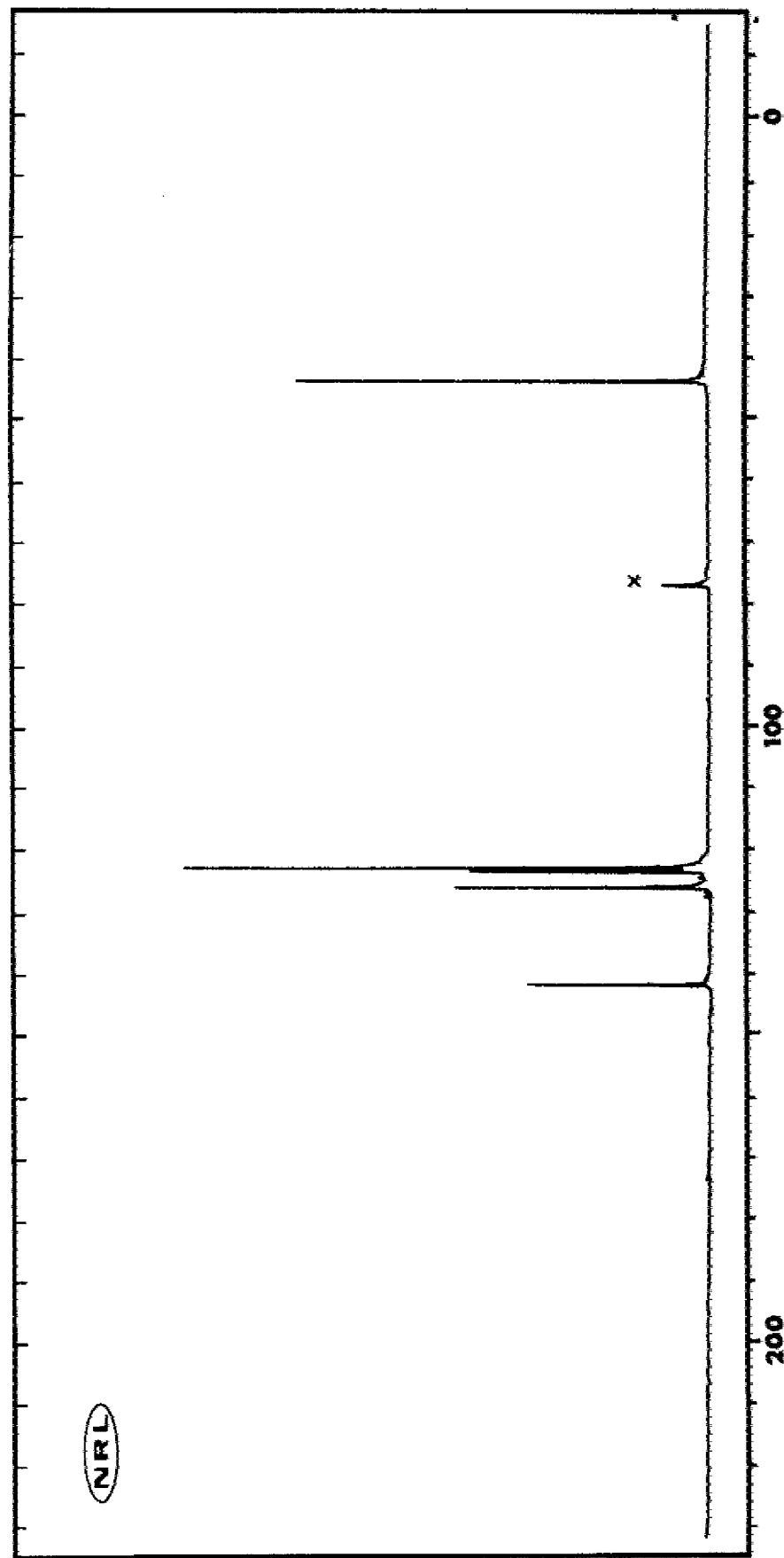
Assignments:

- a 44.0
- b 122.6
- c 123.2
- d 125.7
- e 141.4

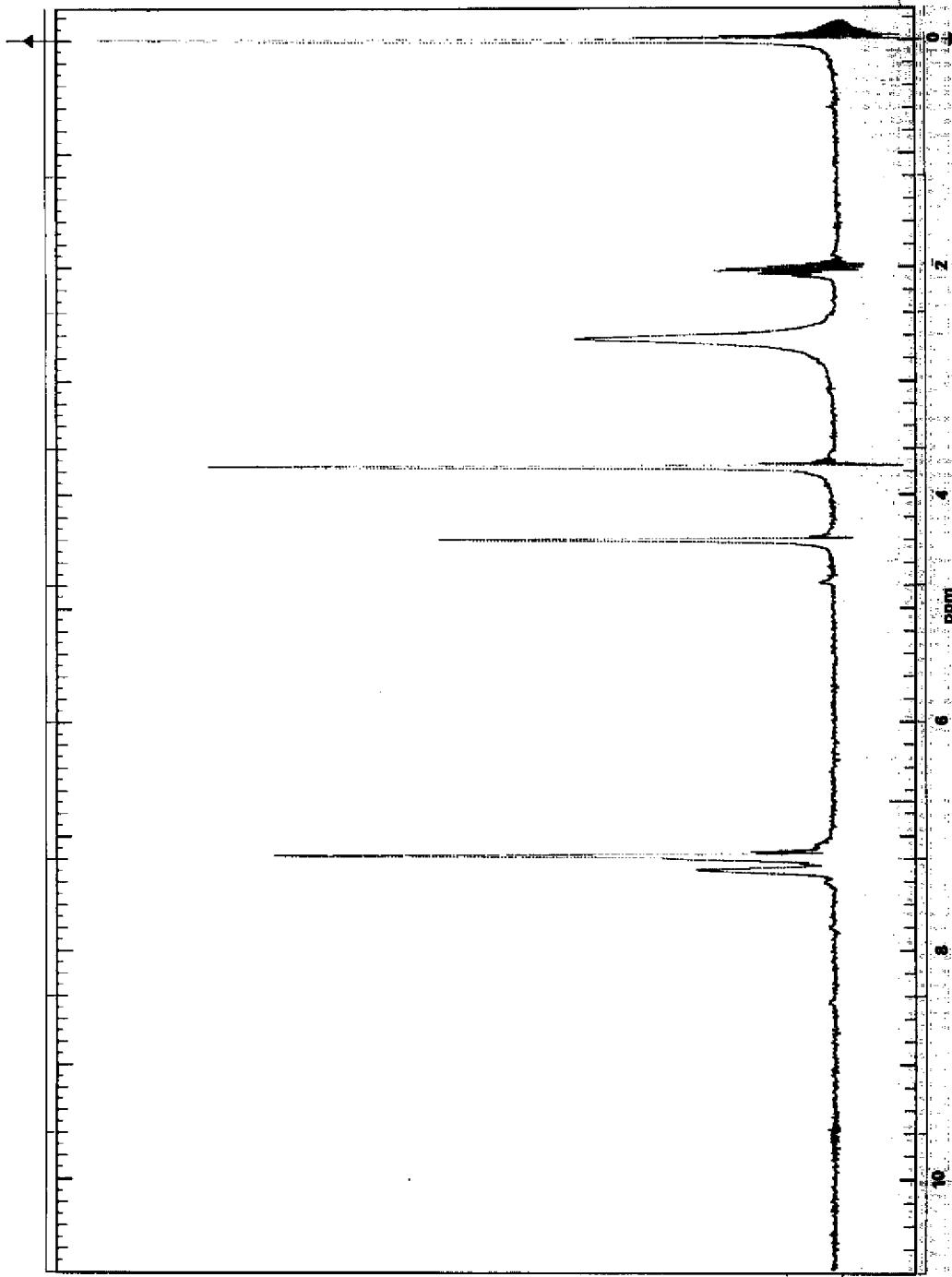
Source: unknown

Solvent: 10% CHCl₃ *

NRL



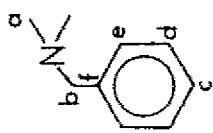
H39



Spectrum 39 — *m*-Xylenediamine (source unknown); solvent: acetone-d₆

C40

N,N-Dimethylbenzylamine

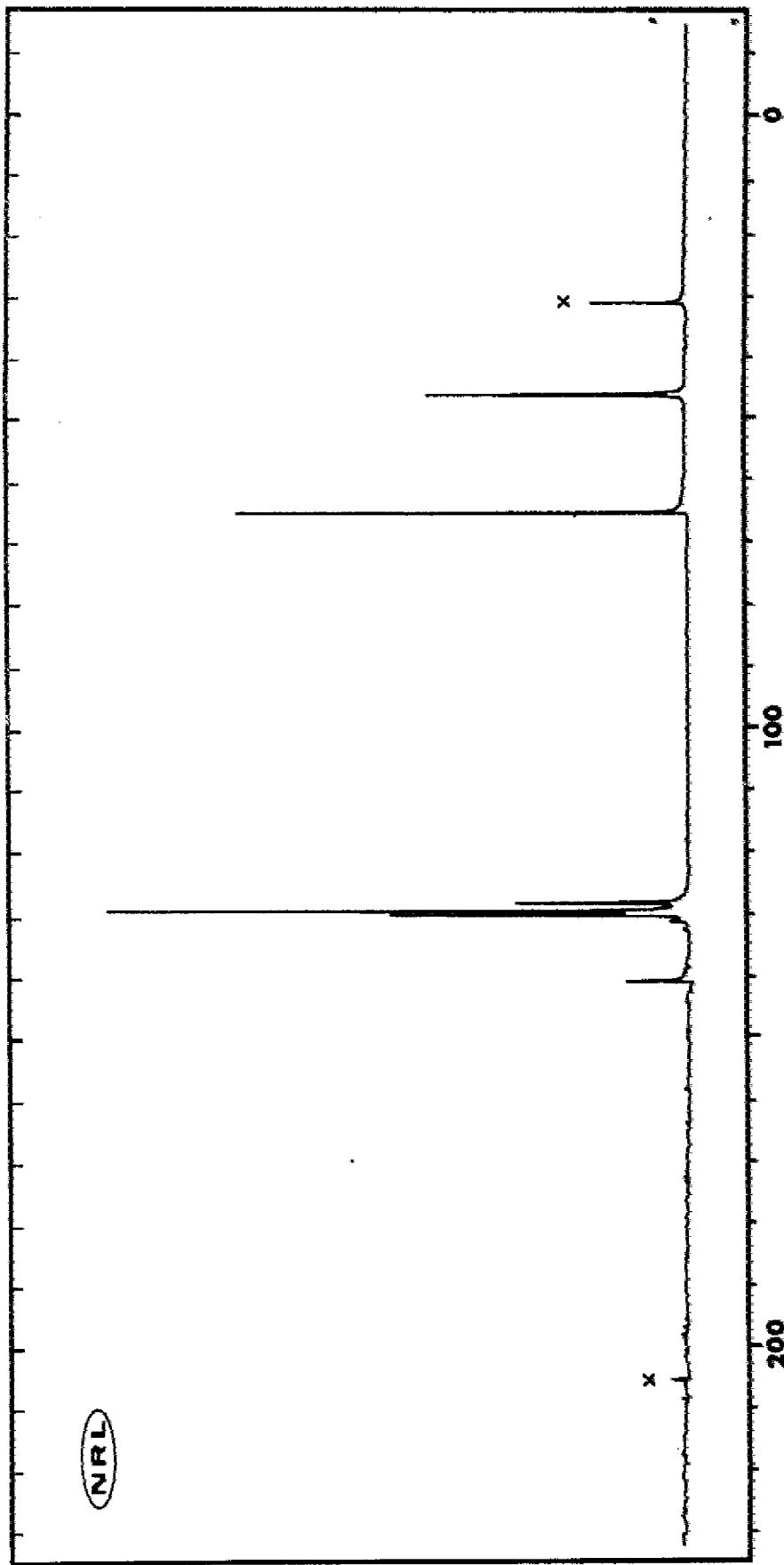


Assignments:

- a 45.3
- b 64.3
- c 126.9
- d 128.2
- e 128.8
- f 139.4

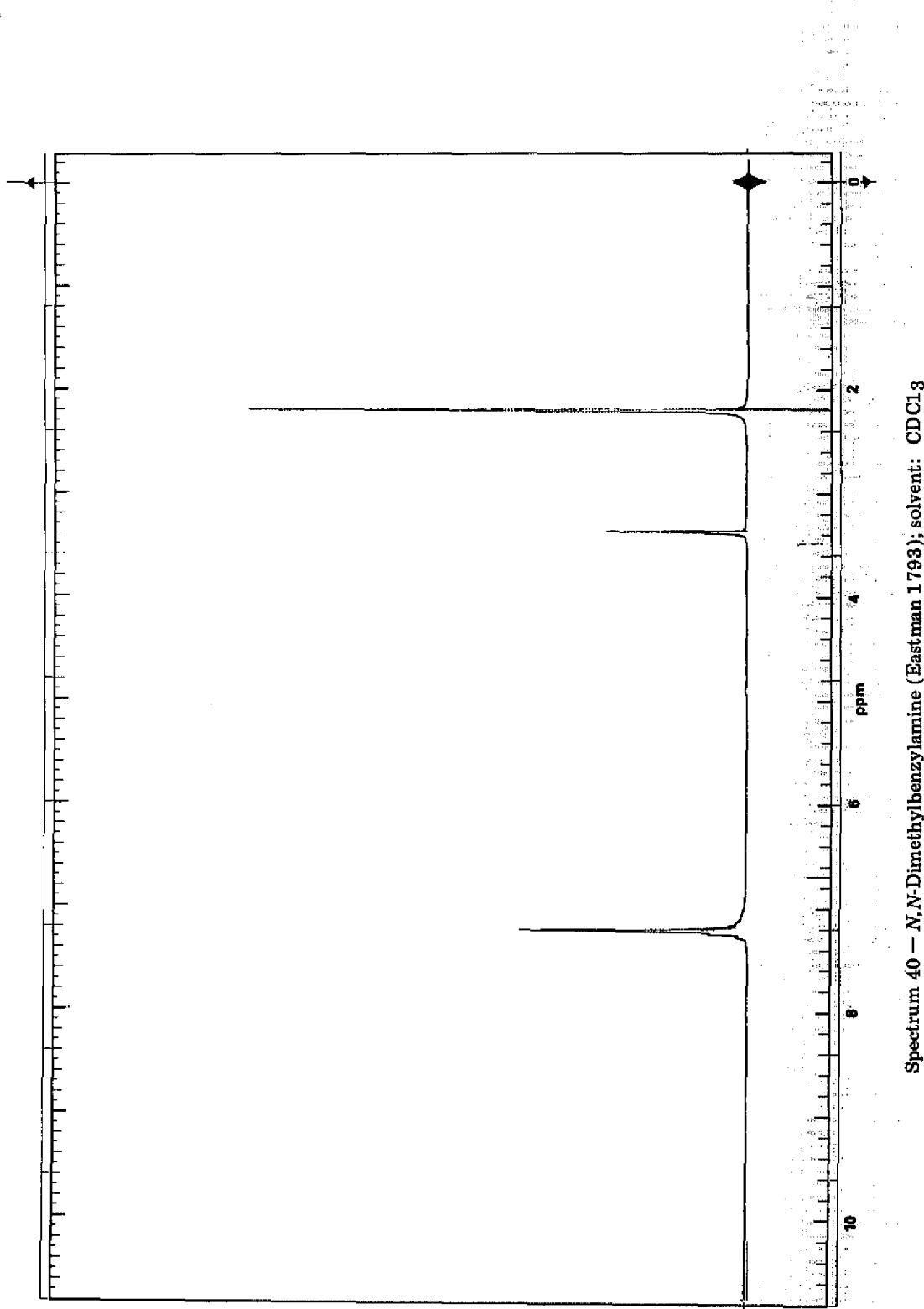
Source: Eastman 1793

Solvent: 25% Acetone x



NRL

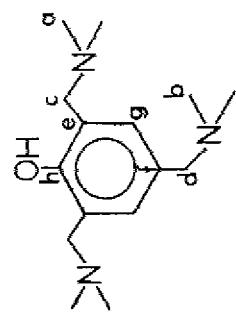
H40



Spectrum 40 — *N,N*-Dimethylbenzylamine (Eastman 1793); solvent: CDCl_3

C41

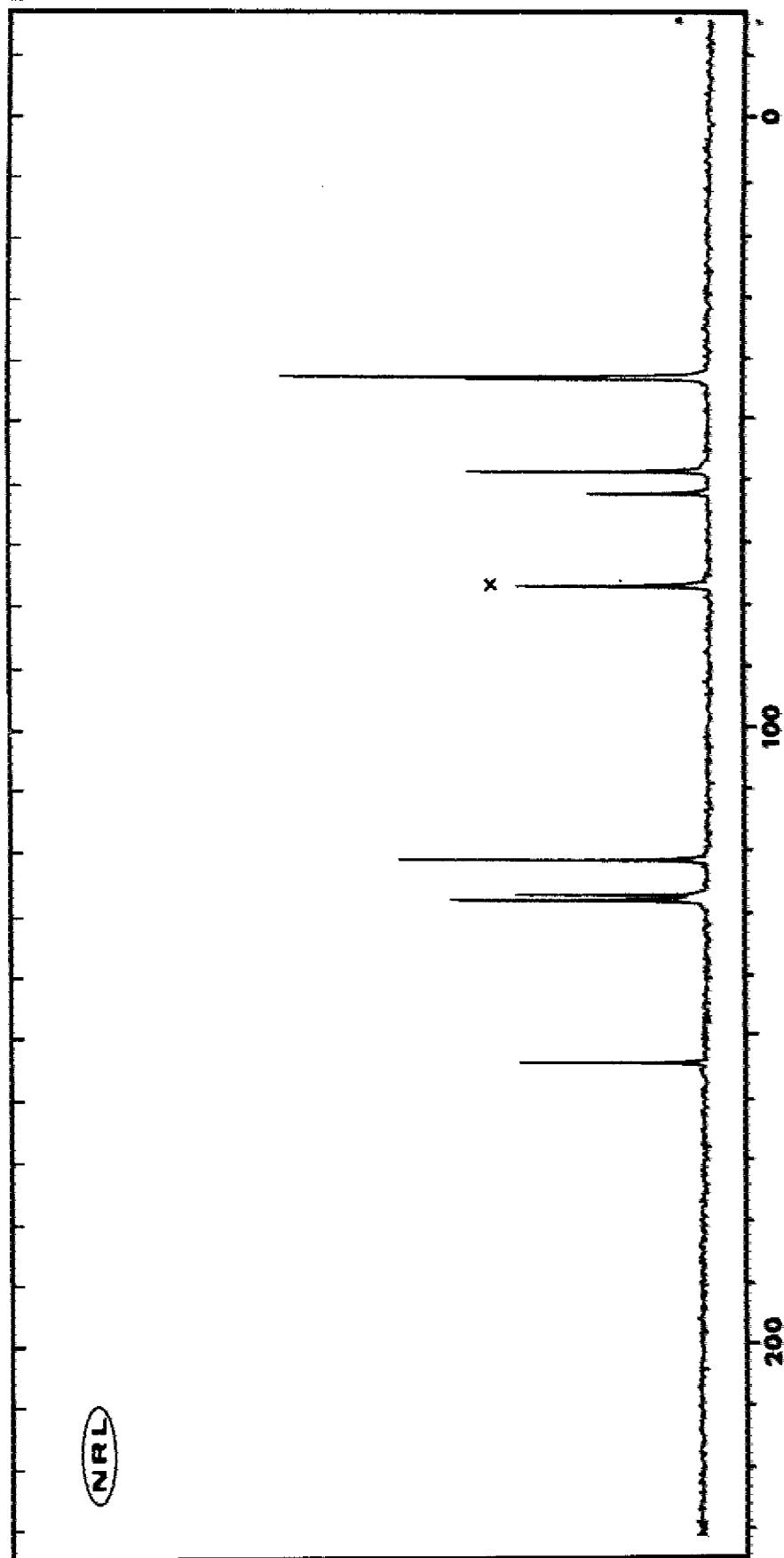
2,4,6-Tris(dimethylaminomethyl)phenol



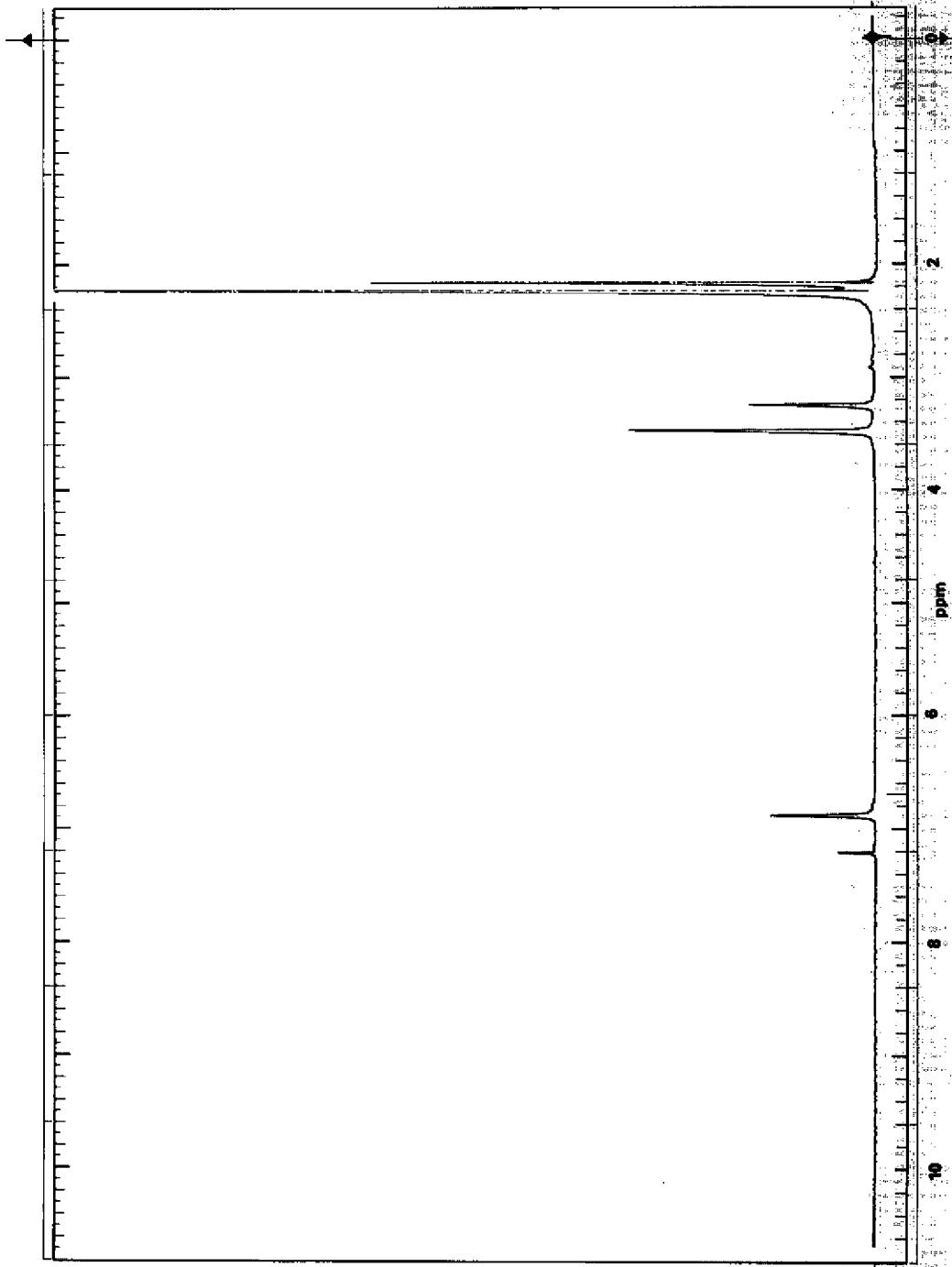
Assignments:	
a	43.1
b	43.5
c	58.6
d	62.2
e	121.0
f	126.6
g	127.5
h	153.6

Source: Rohm and Haas DMP-30

Solvent: 25% CHCl₃ x



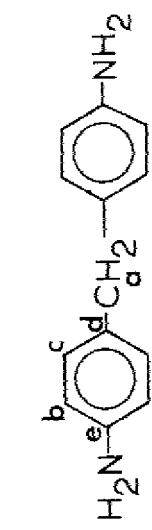
H41



Spectrum 41 — 2,4,6-tris(dimethylaminomethyl) phenol (Rohm and Haas DMP-30); solvent: CDCl_3

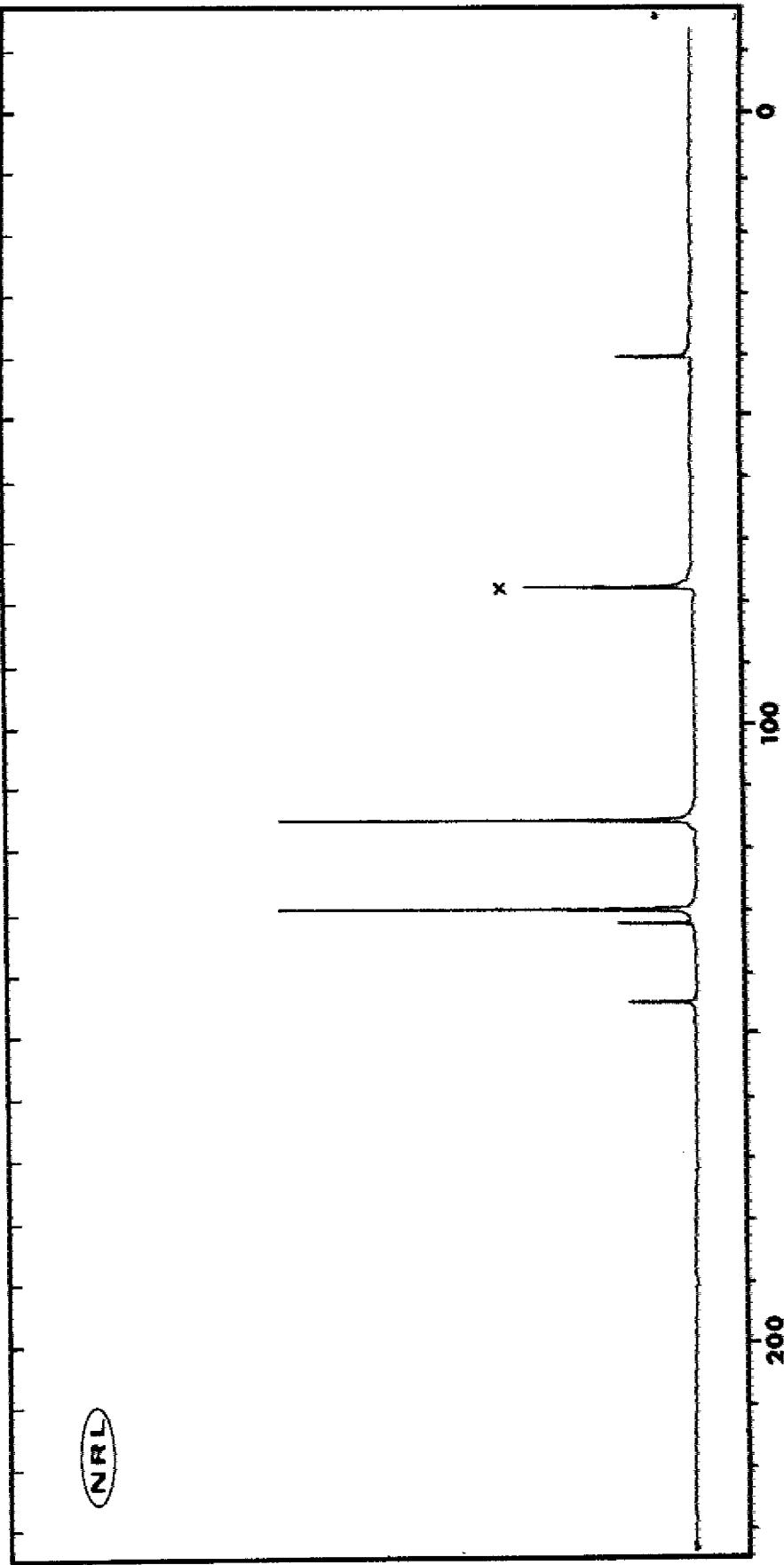
C42

4, 4'-Methylenedianiline

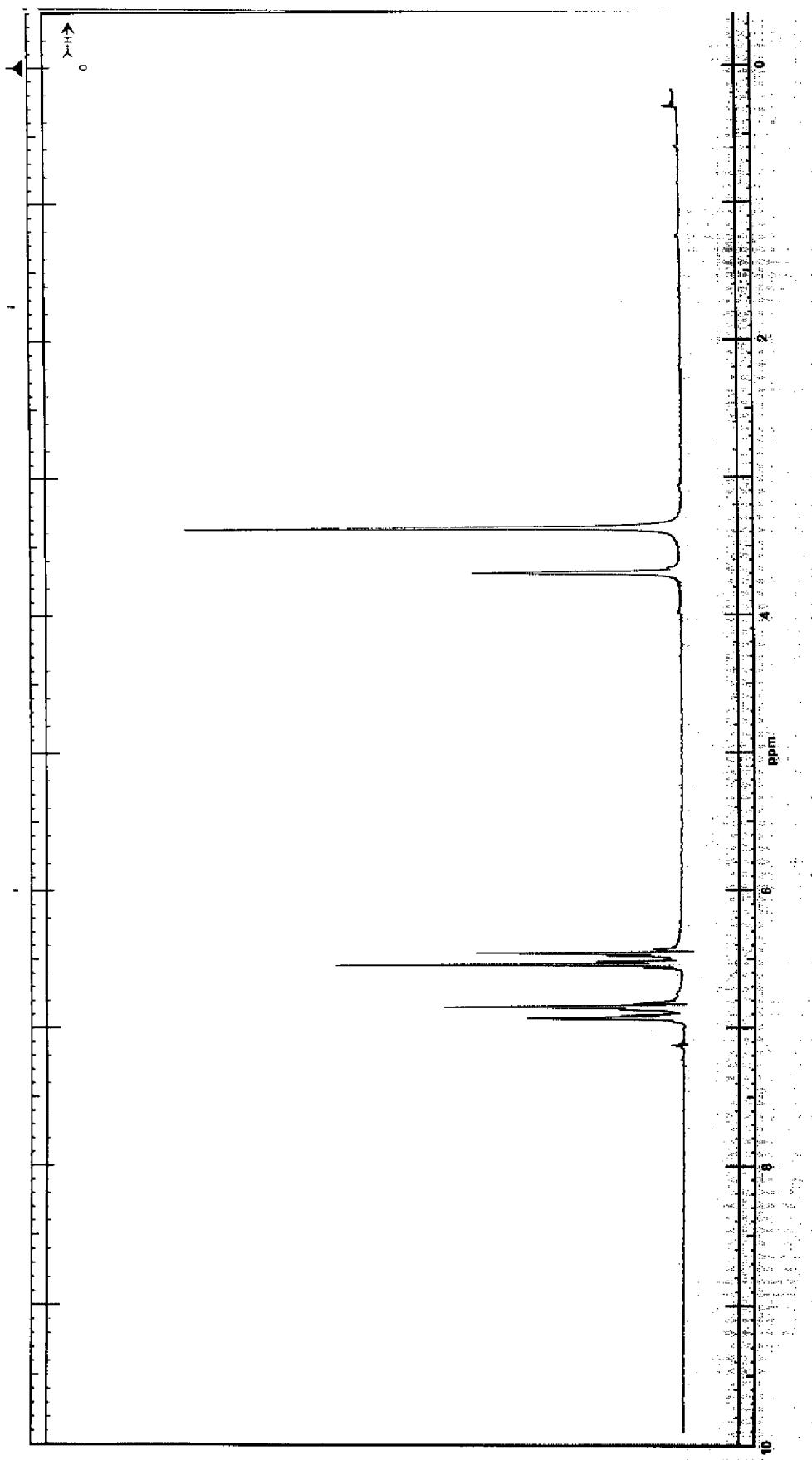


Assignments:	
a	39.7
b	114.6
c	128.9
d	131.1
e	143.8

Source: Eastman P756
Solvent: 75% CHCl₃ *



H42



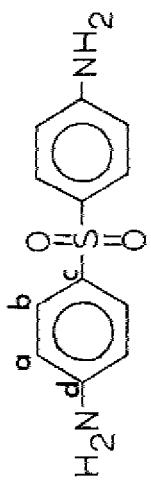
Spectrum 42 — 4,4'-Methylenedianiline (Eastman P756); solvent: CDCl_3

C43

Bis(4-aminophenyl)sulfone

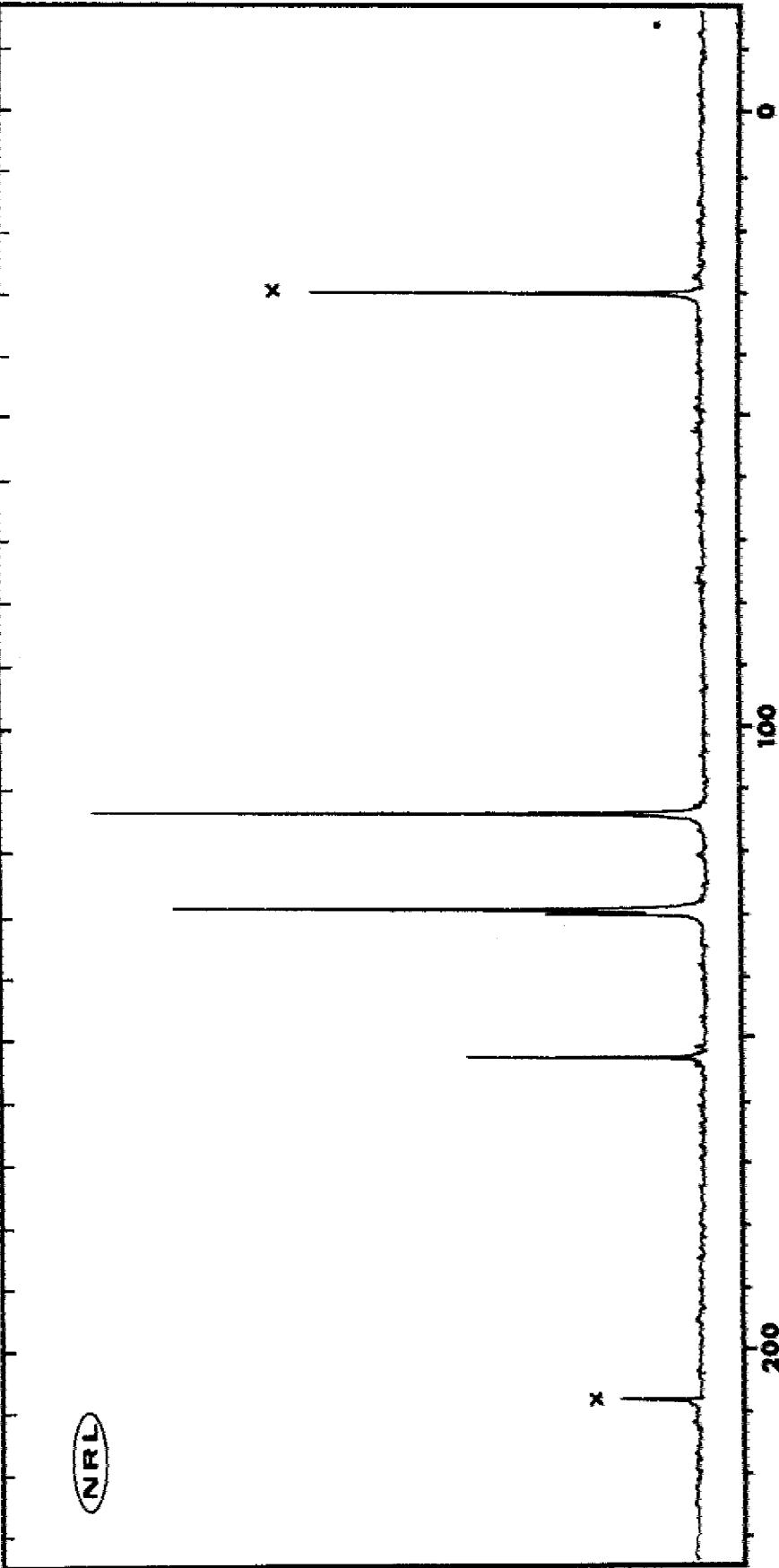
Assignments:

a 113.6d
b 128.9d
c 129.7s
d 152.3s

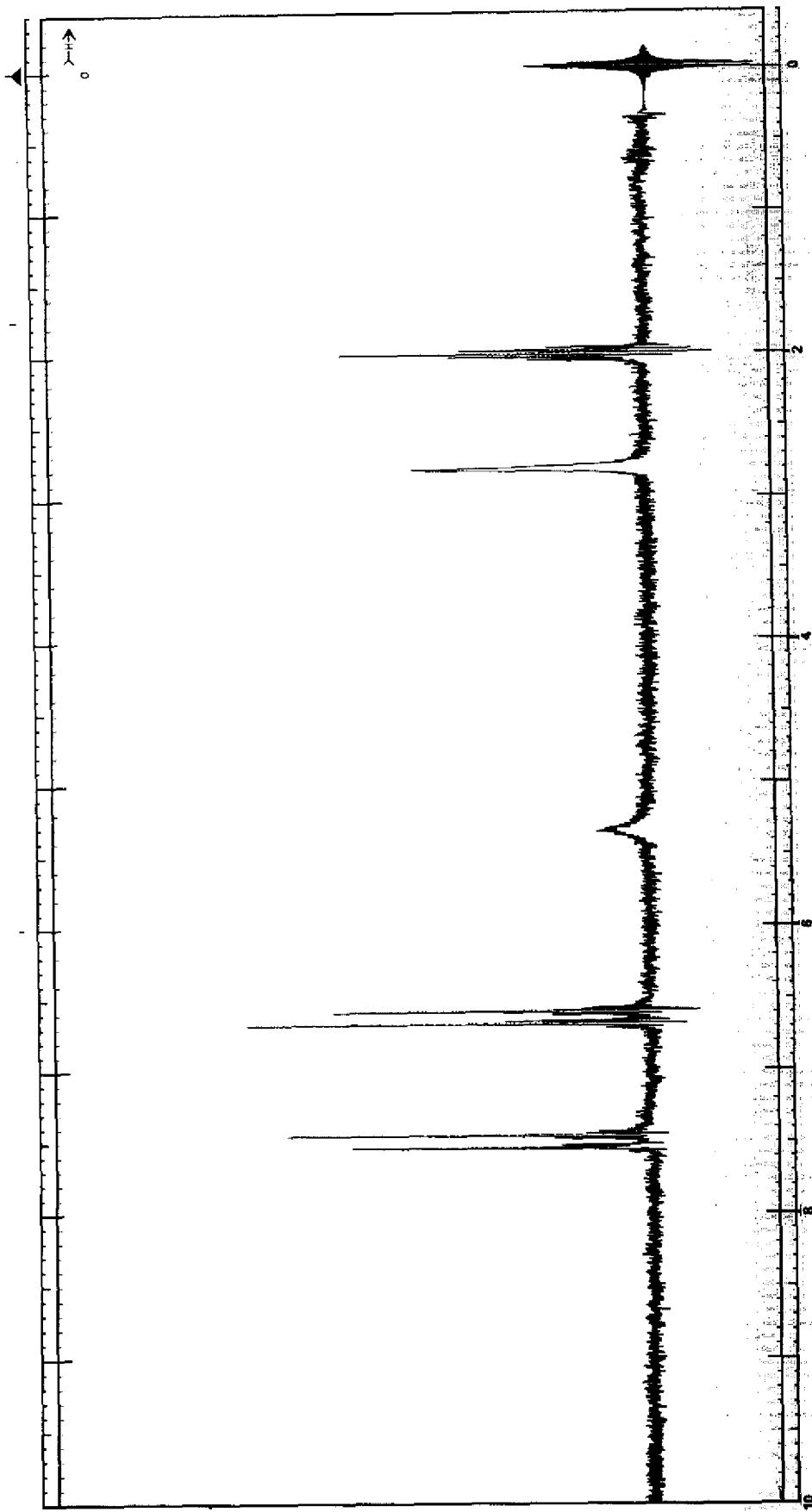


Source: Aldrich A7480-7

Solvent: 50% Acetone *



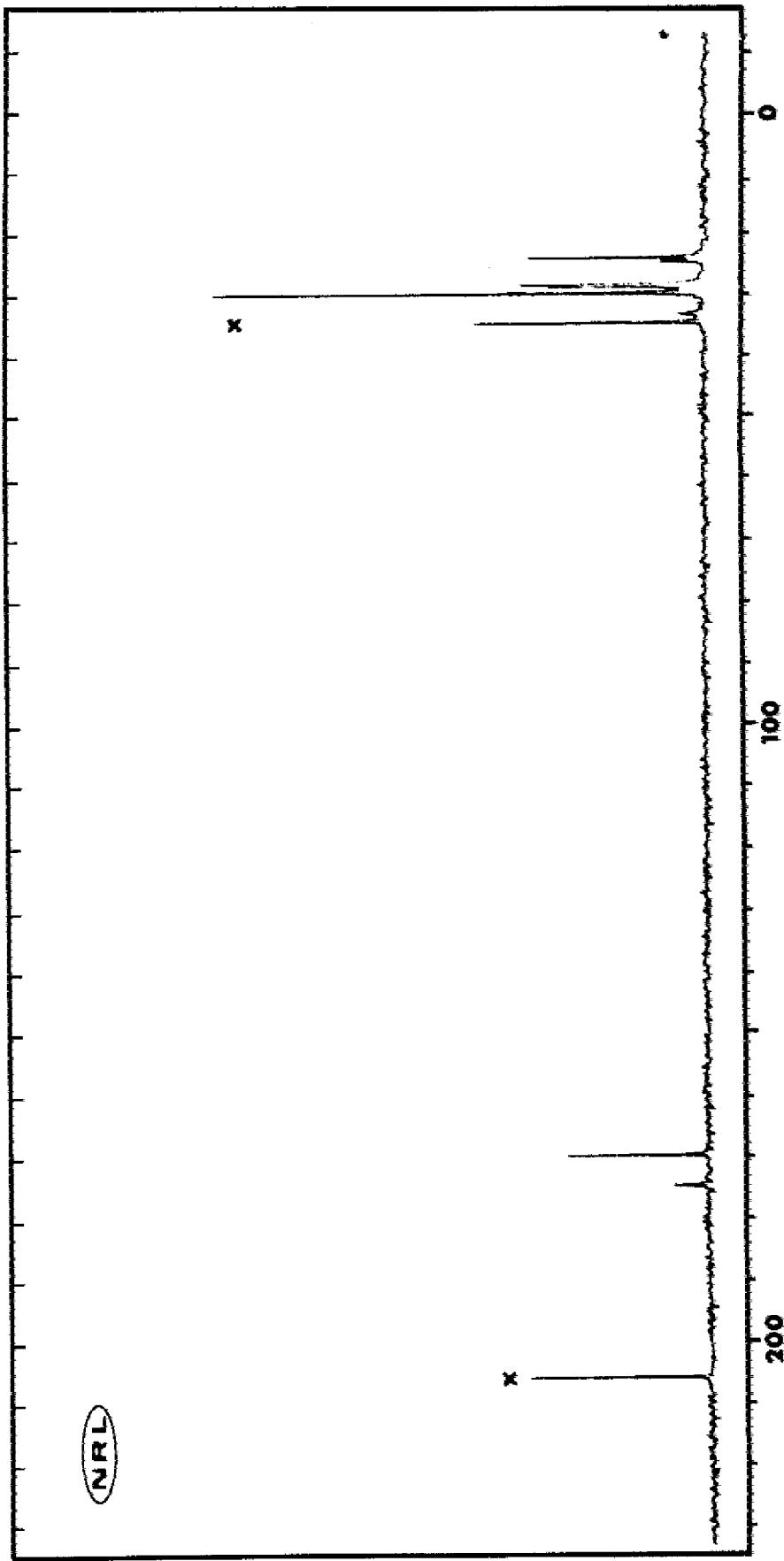
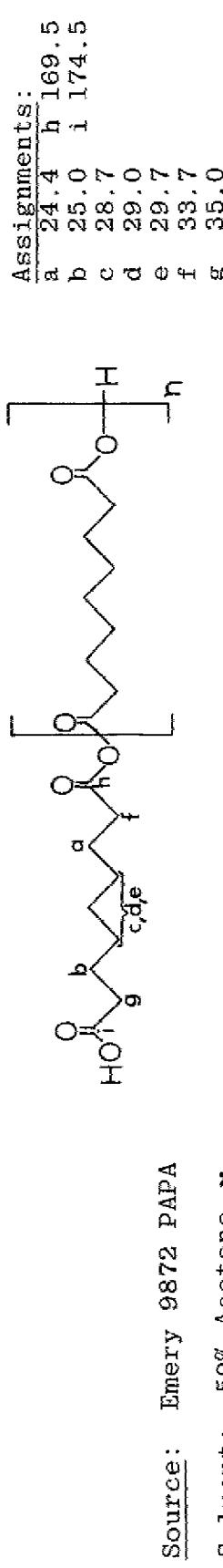
H43



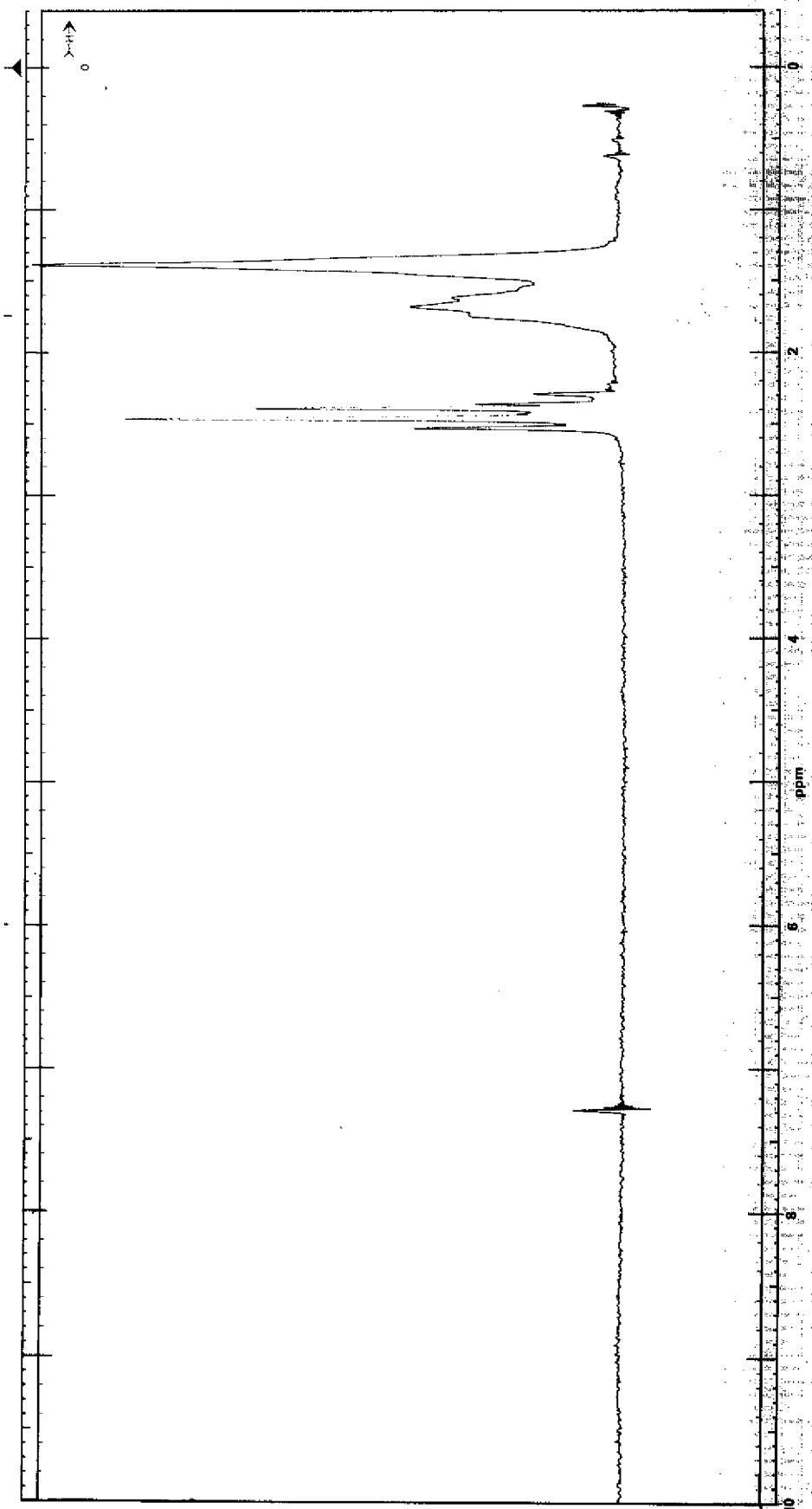
Spectrum 43 — *bis*(4-aminophenyl)sulfone (Aldrich A7480-7); solvent: acetone-d₆

C44

Polyazelaic Polyanhydride



H44

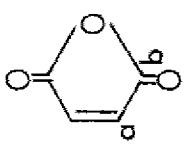


Spectrum 44—Polyazelaic polyanhydride (Emery 9872 PAPA); solvent: CDCl_3

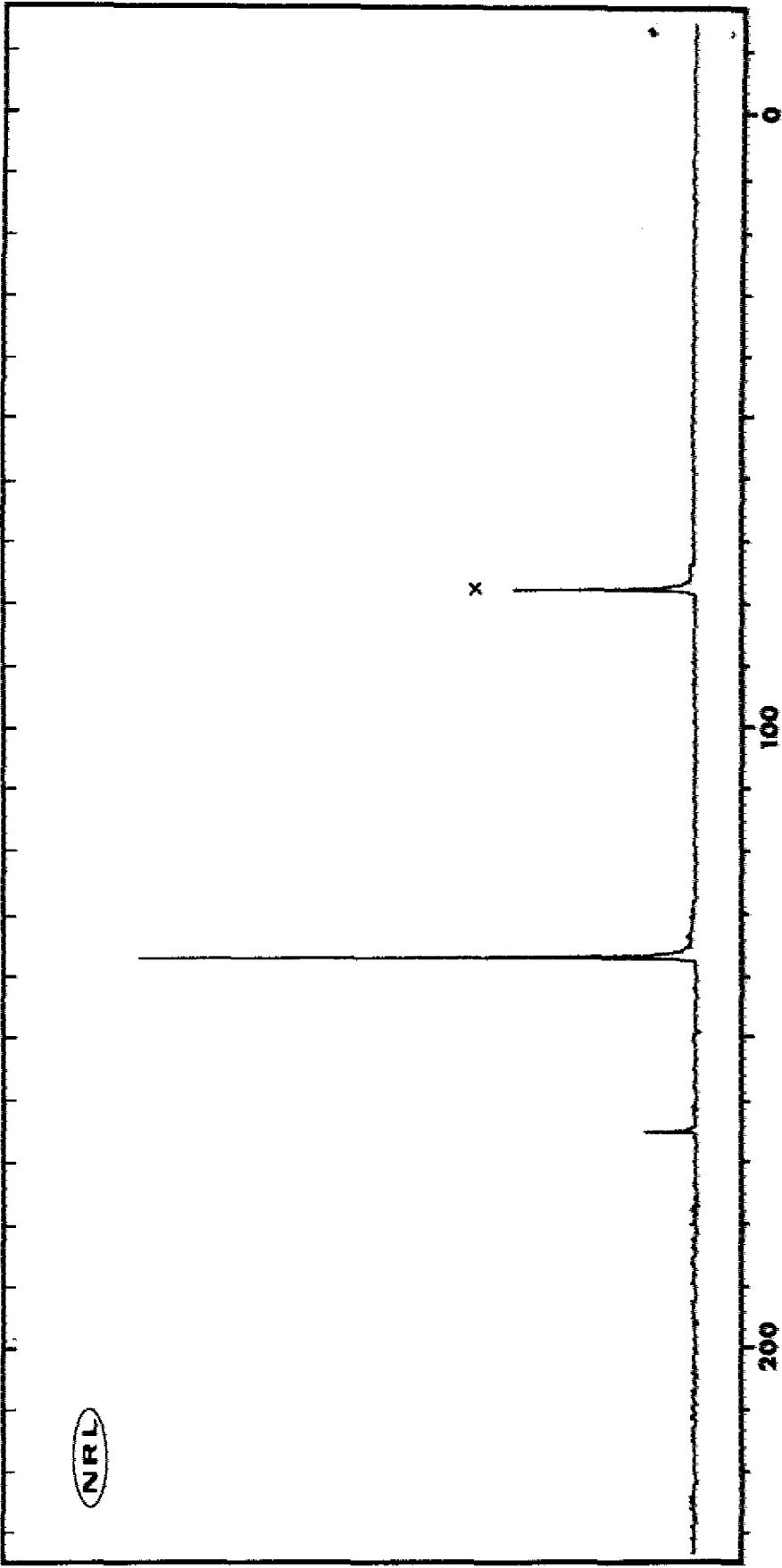
C45

Maleic Anhydride

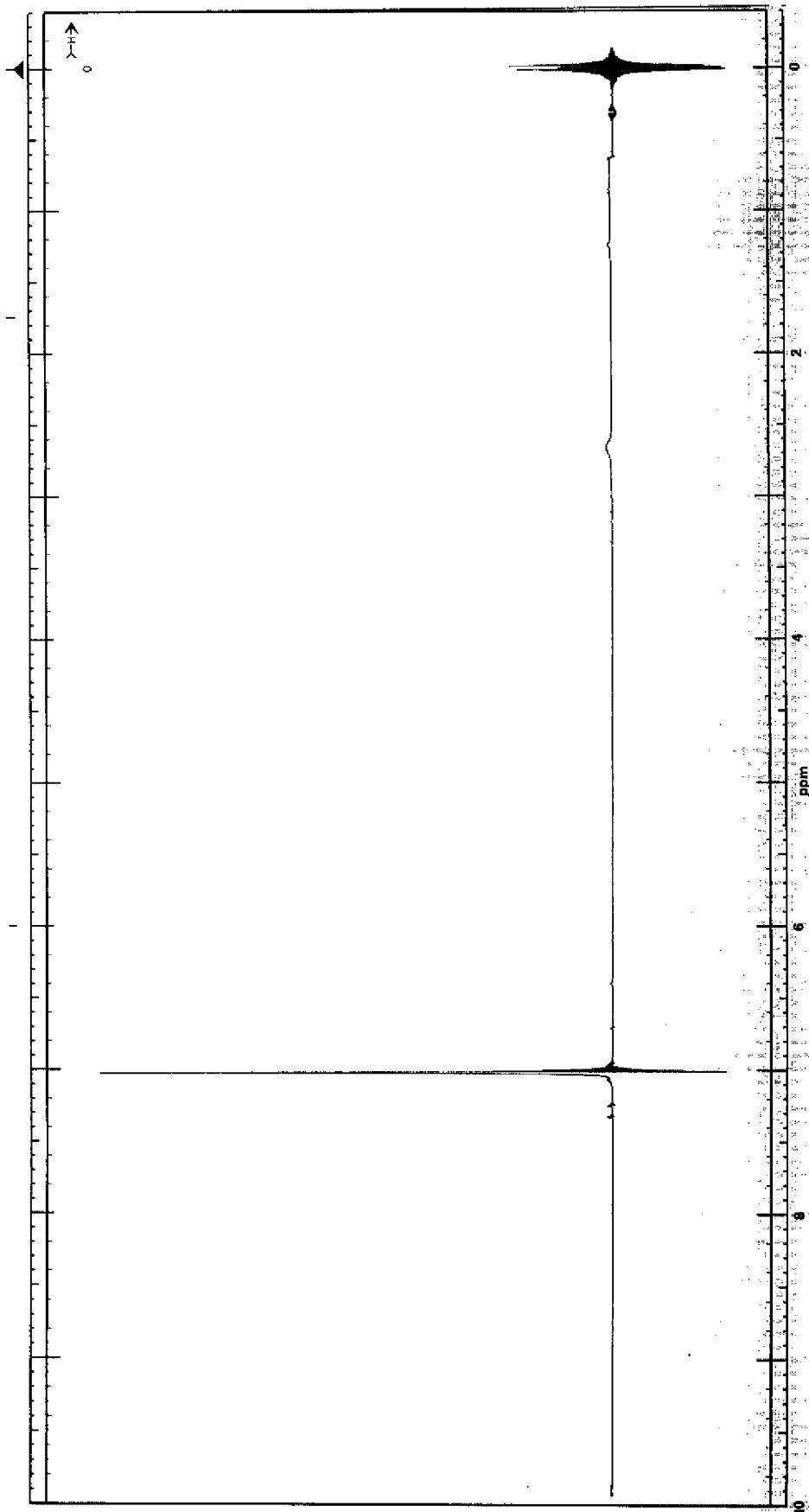
Source: Fisher A-168
Solvent: 50% CHCl₃ *



Assignments:
a 136.2
b 164.1



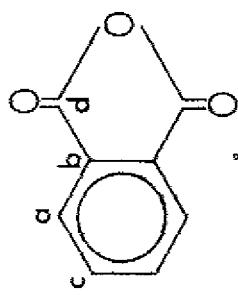
H45



Spectrum 45 — Maleic anhydride (Fisher A-168); solvent: CDCl₃

C46

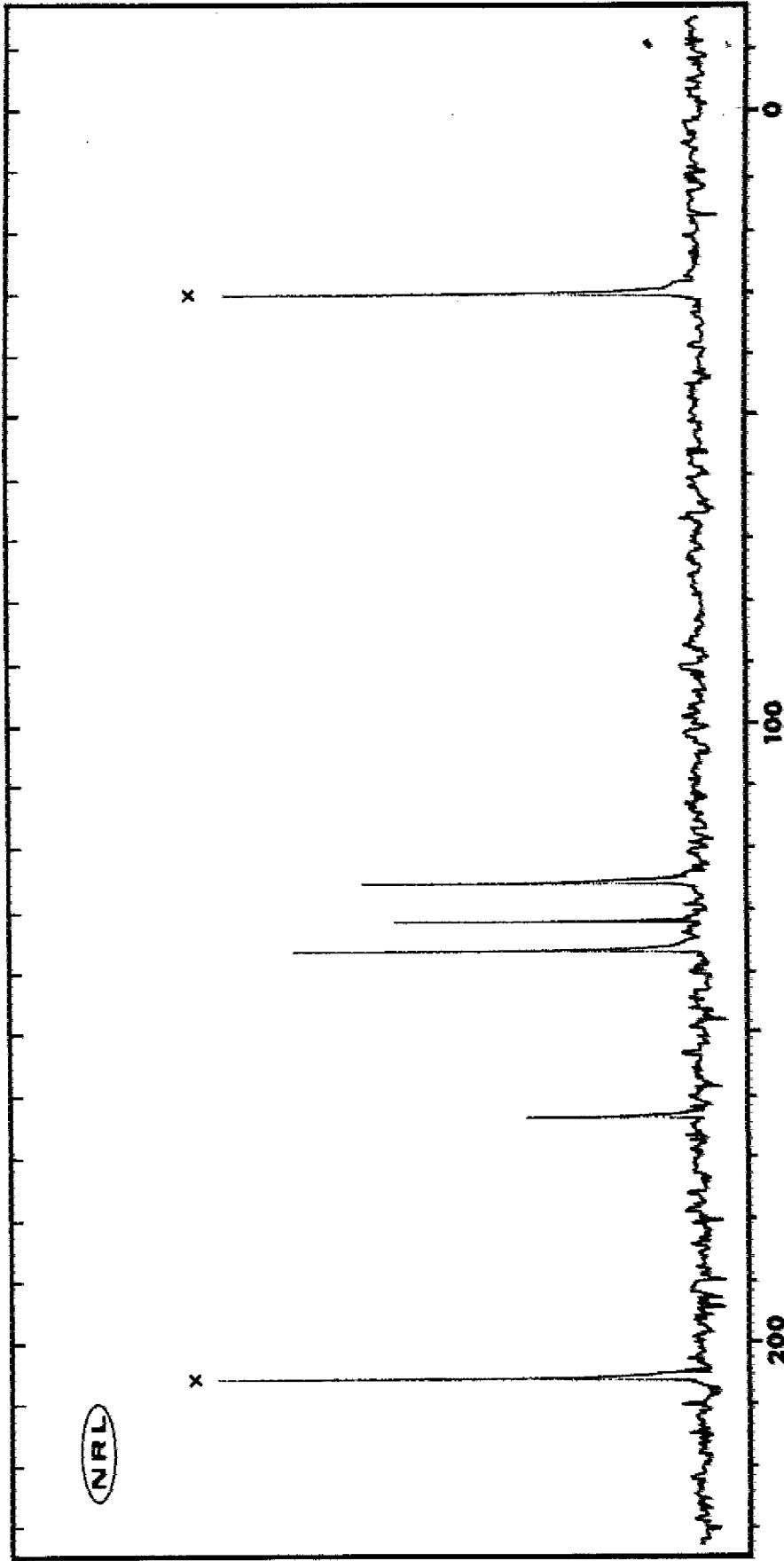
Phthalic Anhydride



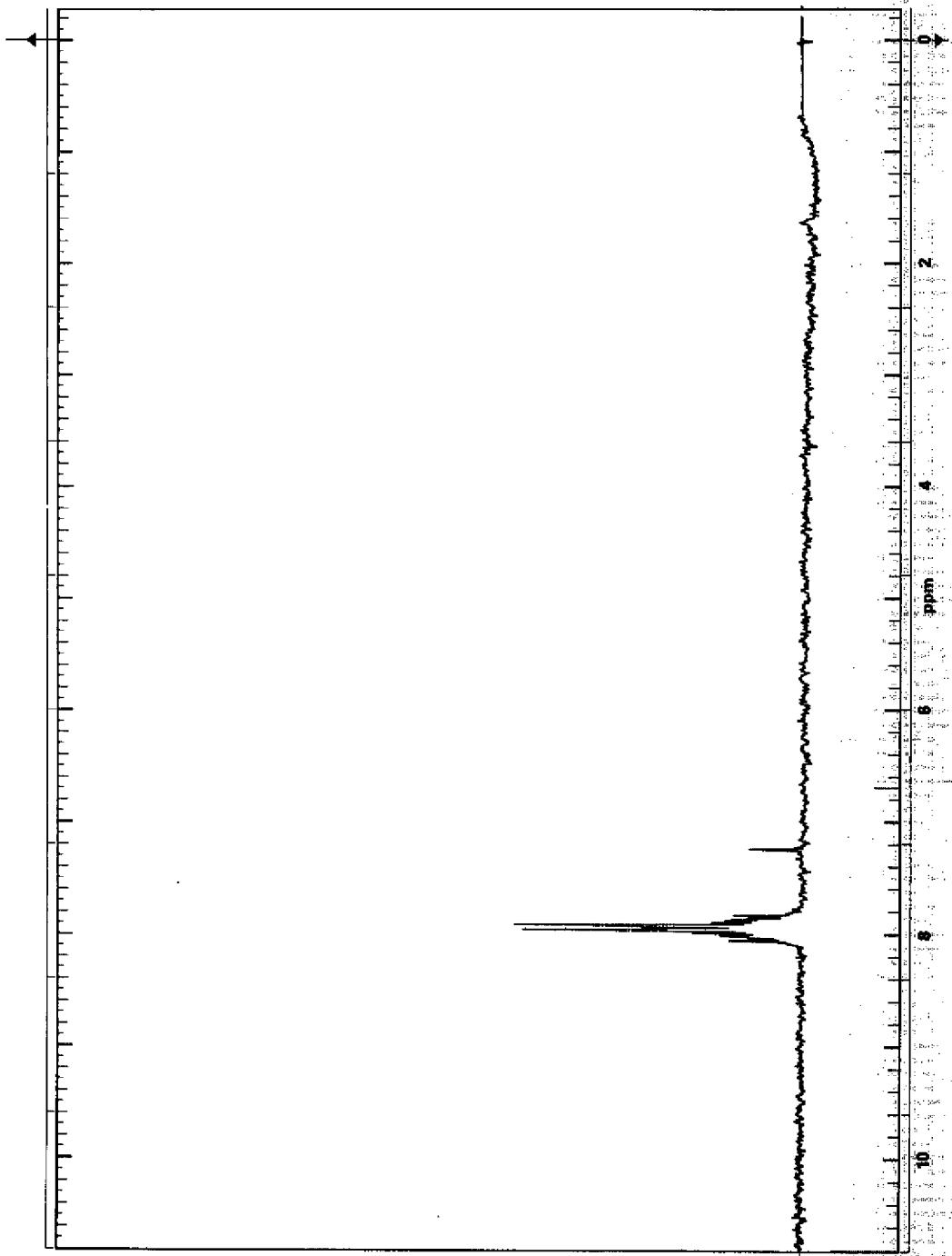
Source: Recrystallized from benzene

Solvent: 75% Acetone \times

Assignments:
a 125.5
b 131.6
c 136.4
d 163.2



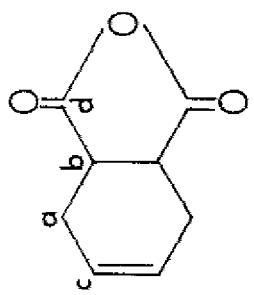
H46



Spectrum 46 — Phthalic anhydride (recrystallized from benzene); solvent: CDCl_3

C47

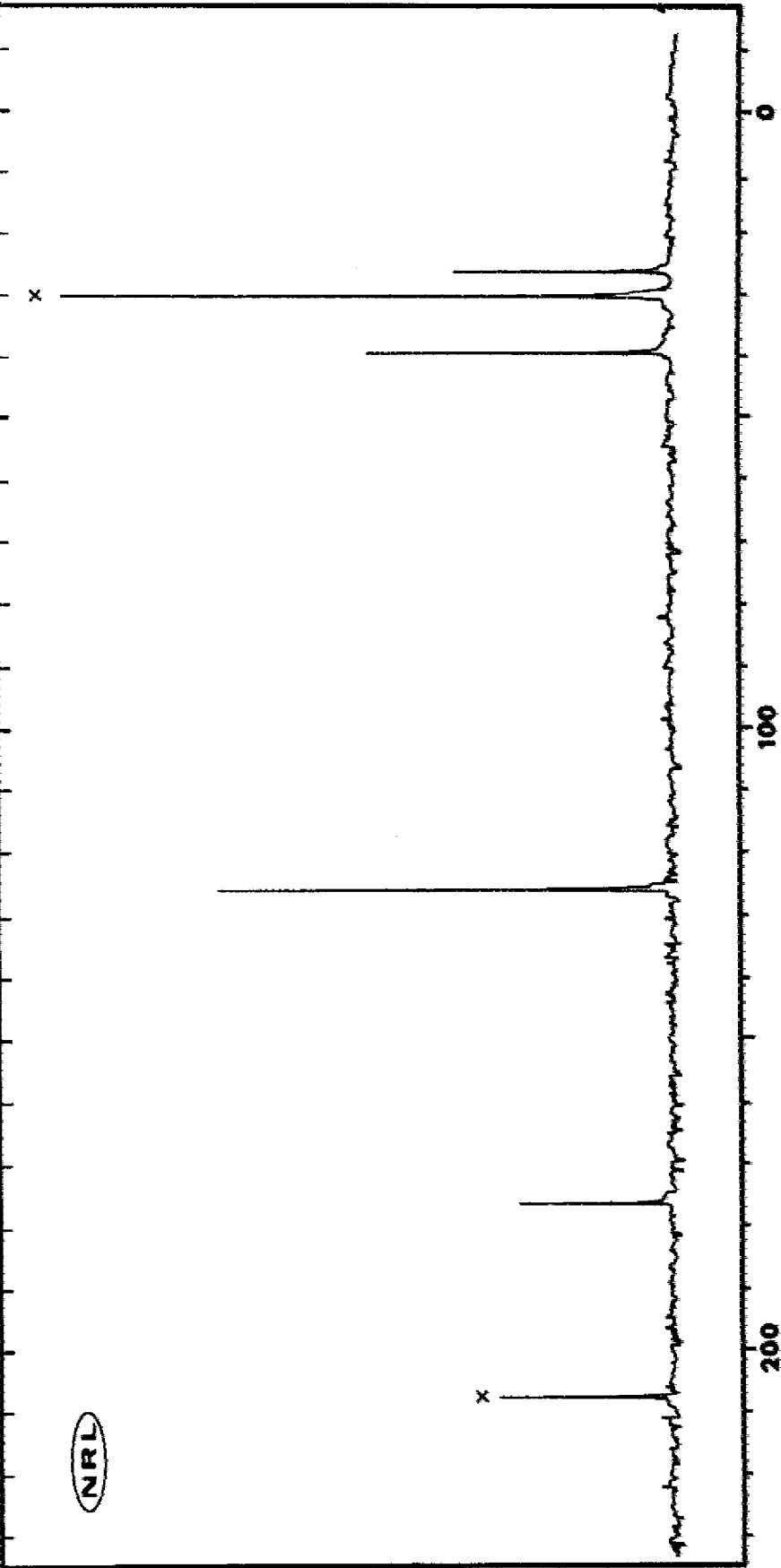
Tetrahydrophthalic Anhydride



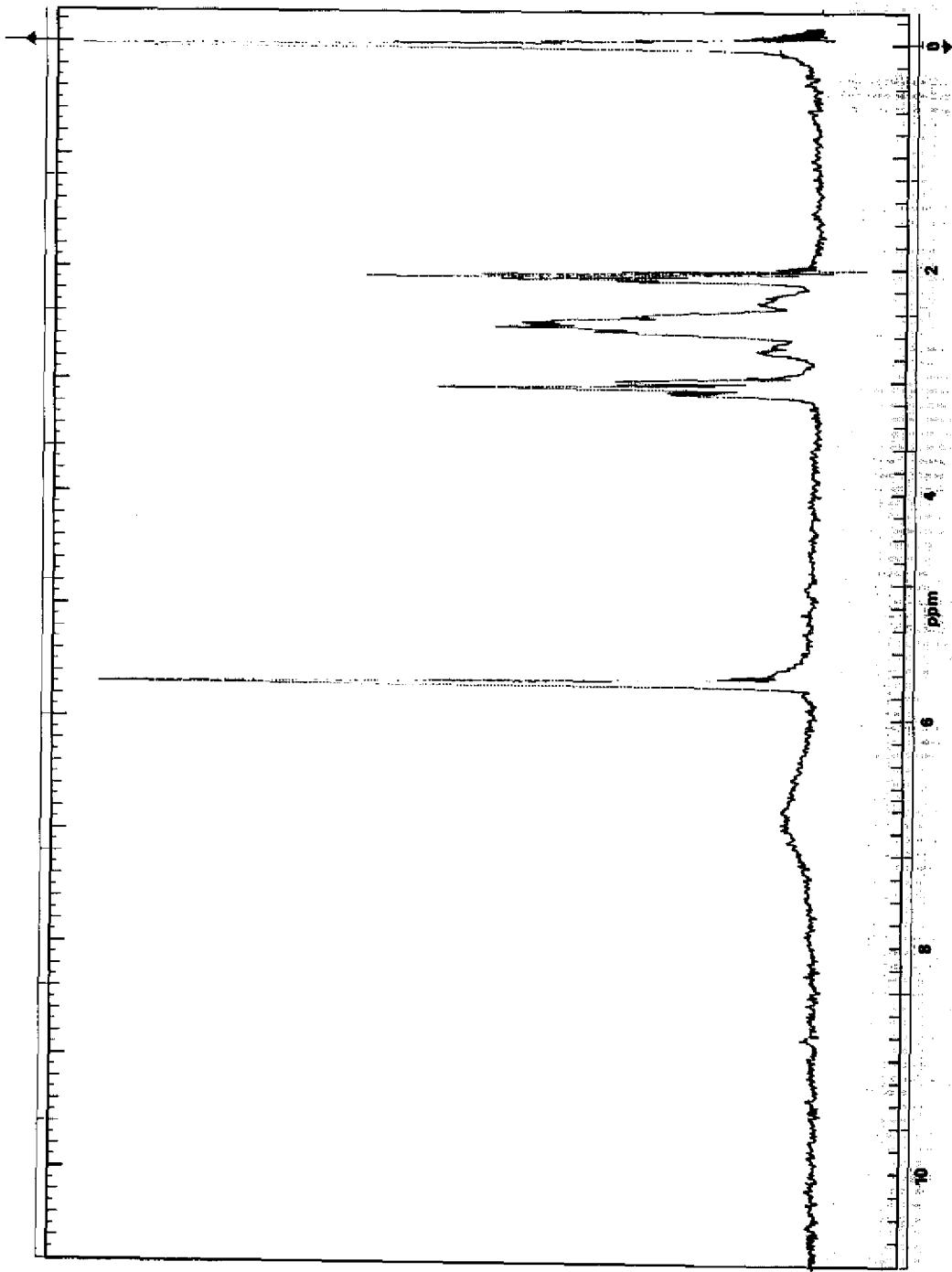
Assignments:
a 26.2
b 39.4
c 125.3
d 175.3

Source: Eastman #5724

Solvent: 50% Acetone x



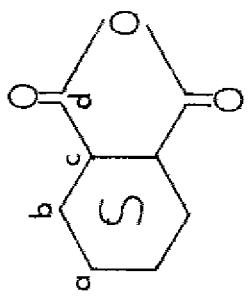
H47



Spectrum 47 — Tetrahydrophthalic anhydride (Eastman 5724); solvent: acetone-d₆

C48

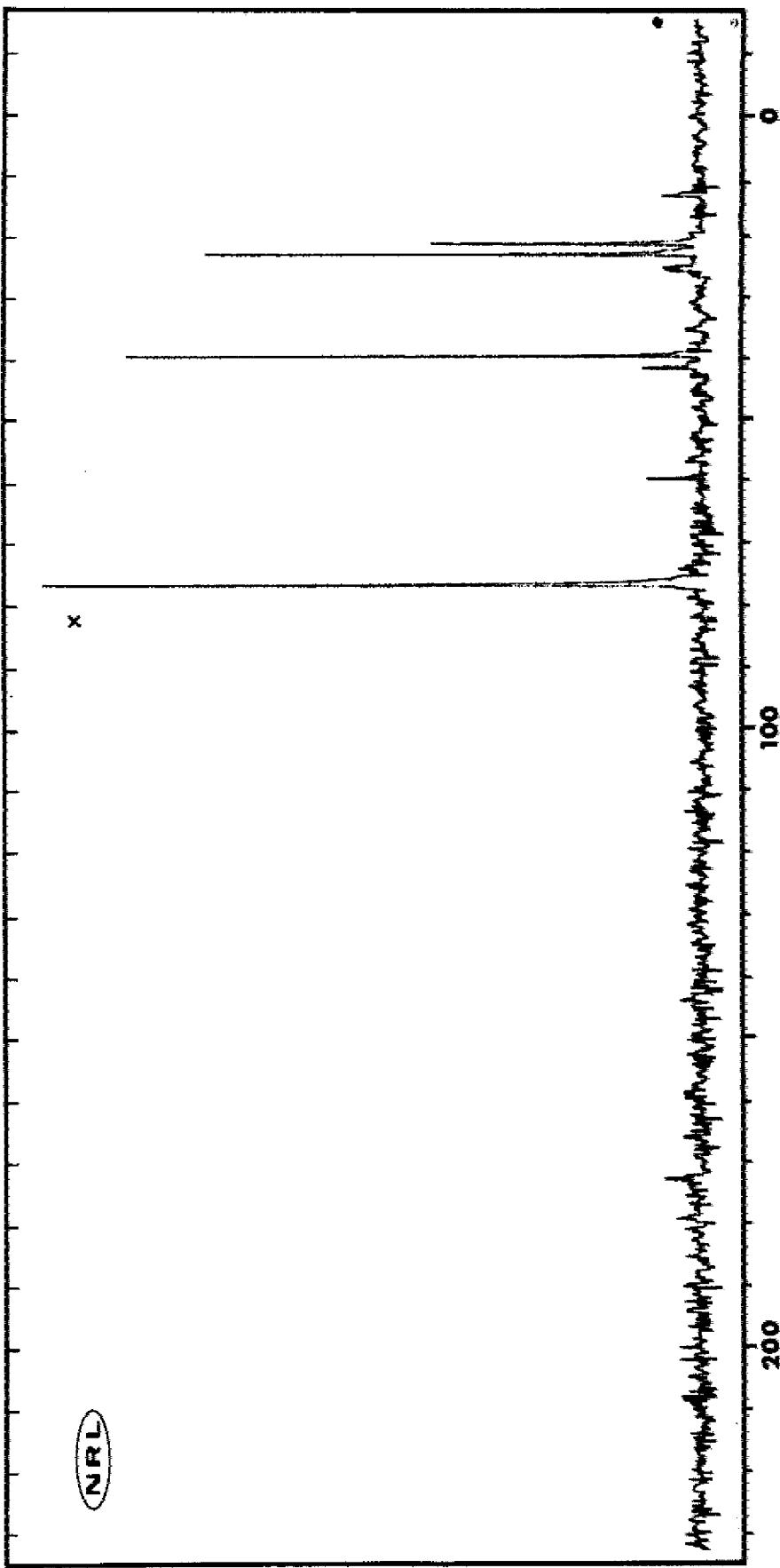
Hexahydrophtalic Anhydride



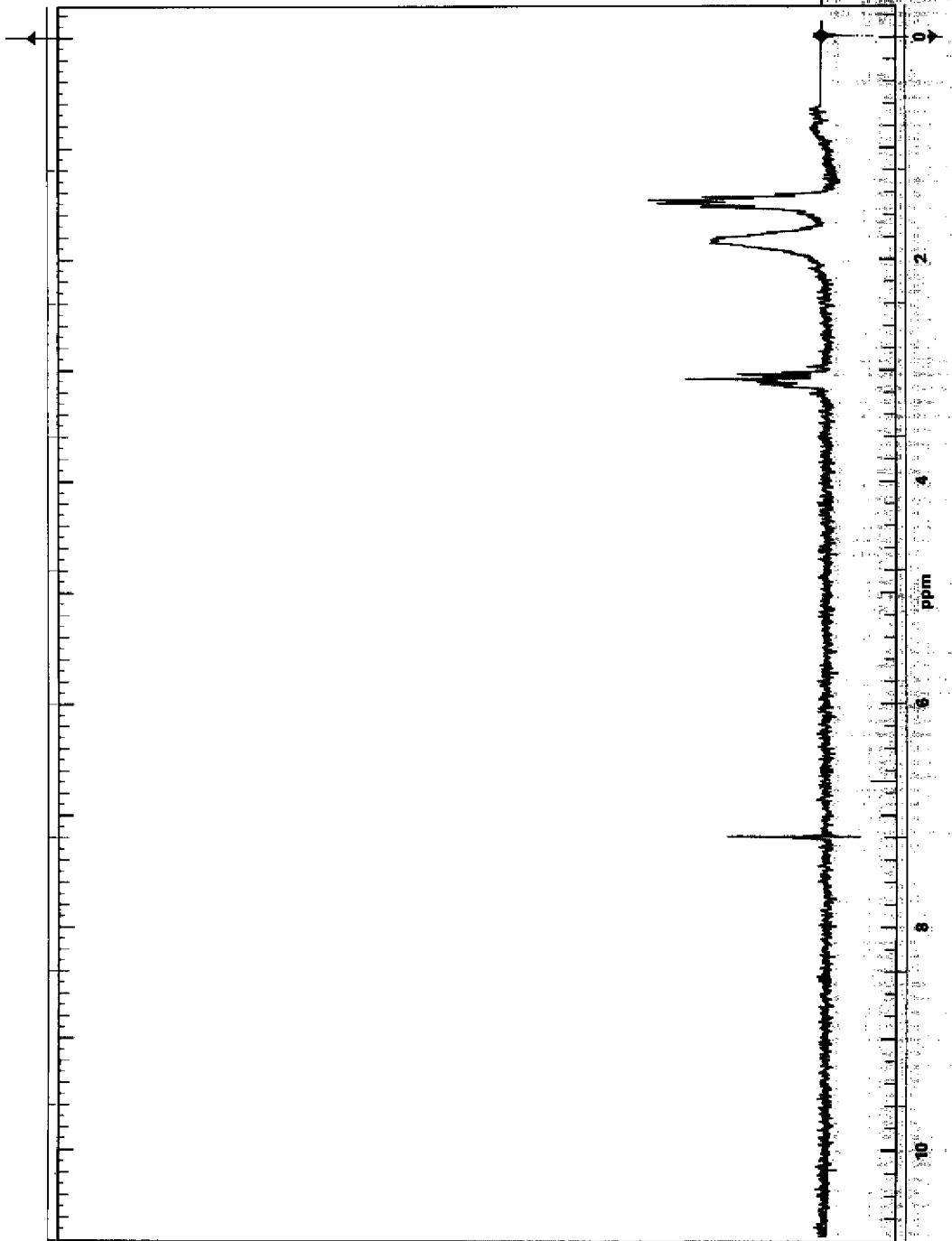
Source: Miller-Stephenson Chemical Co.

Solvent: 50% CHCl₃ *

Assignments:	
a	21.4
b	23.1
c	39.8
d	172.1



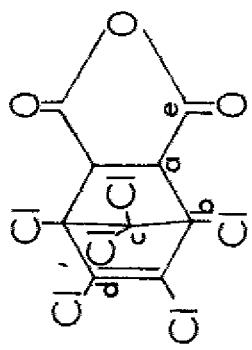
H48



Spectrum 48 — Hexahydropthalic anhydride (Miller-Stephenson Chemical Co.); solvent: CDCl₃

C49

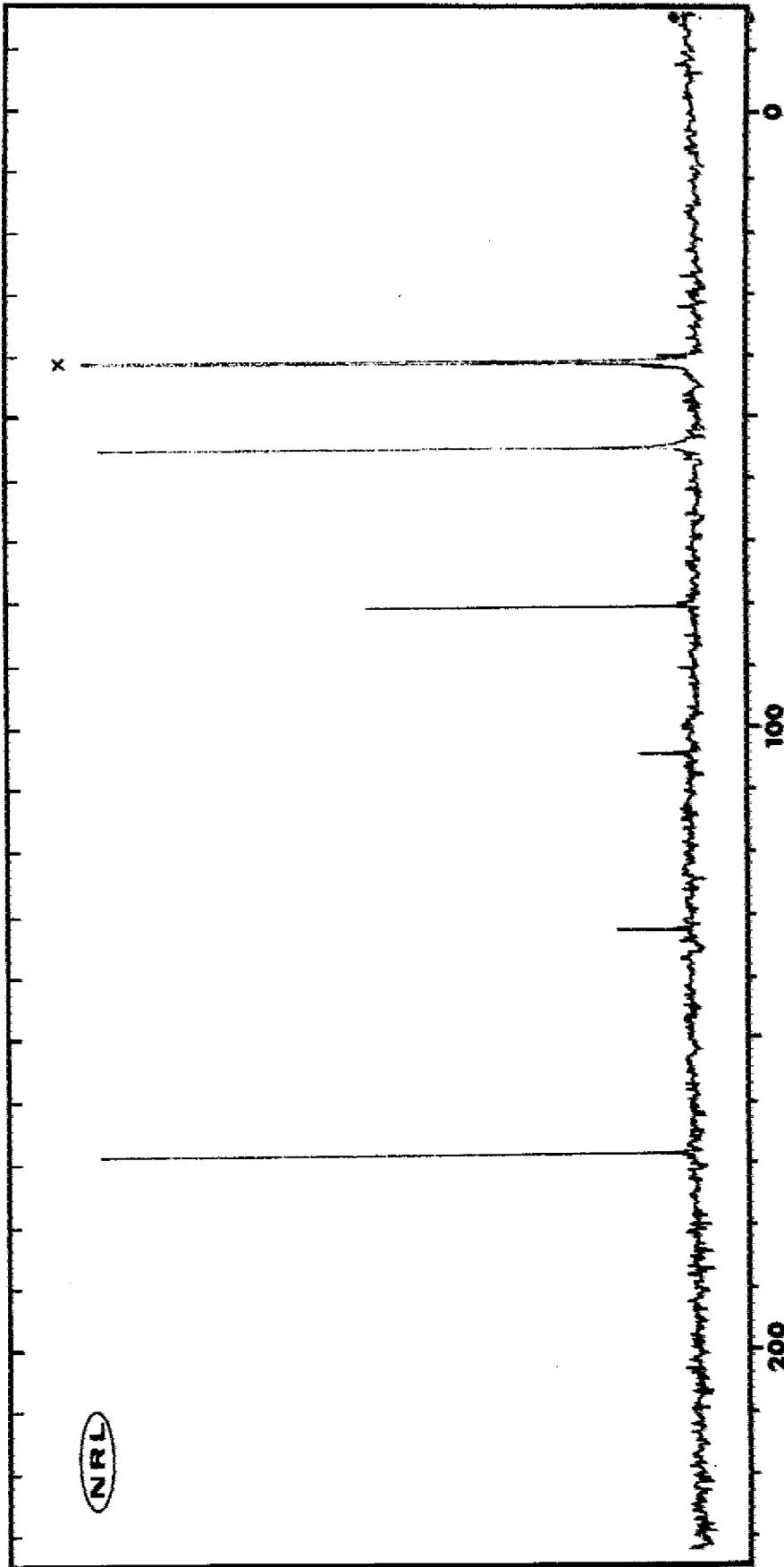
1,4,5,6,7,7-Hexachloro-5-Norbornene-2,3-Dicarboxylic Anhydride



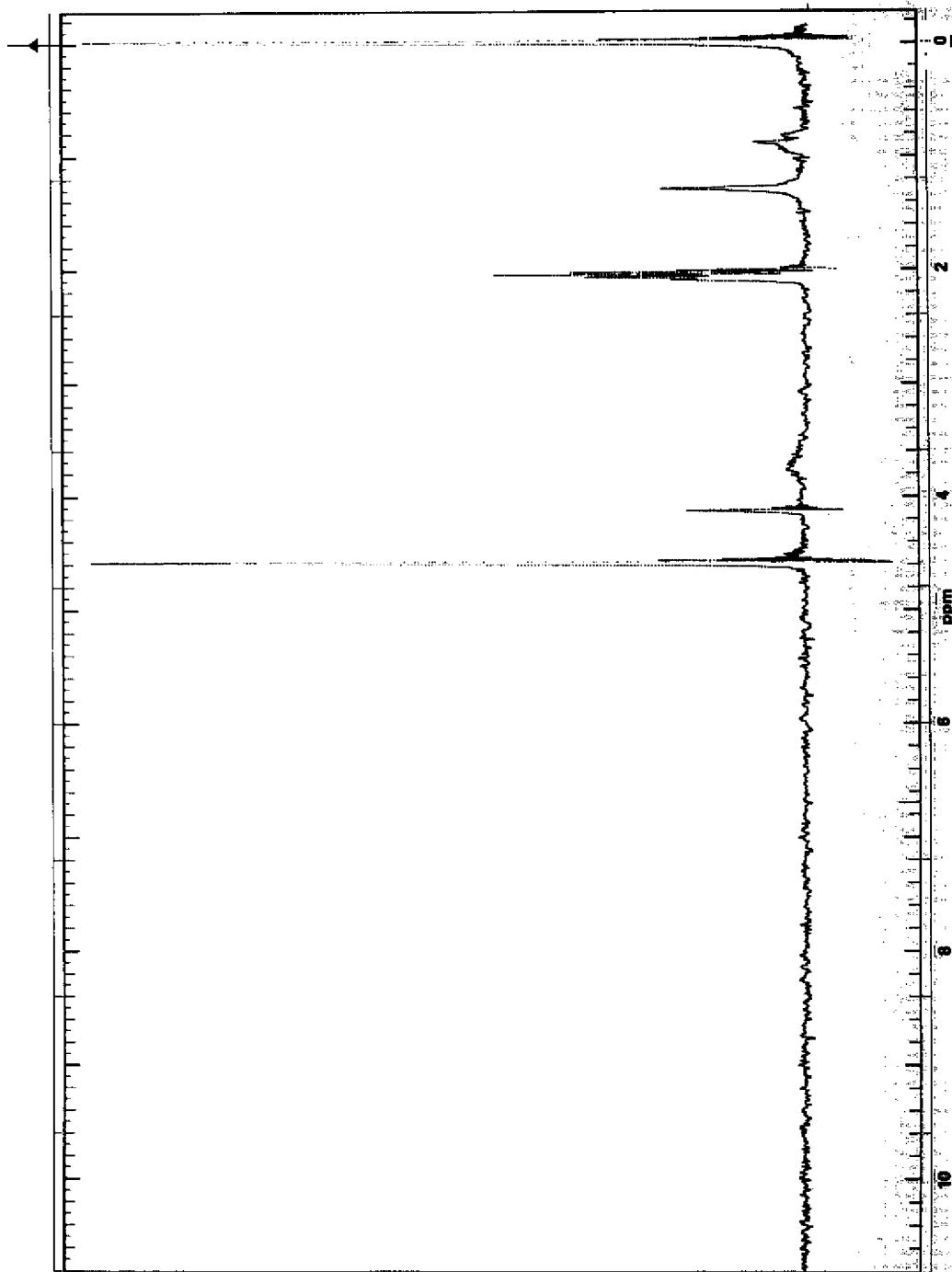
Assignments:
a 55.9
b 81.2
c 104.8
d 133.0
e 169.2

Source: Aldrich # 10,326-8

Solvent: DMSO (saturated solution) *



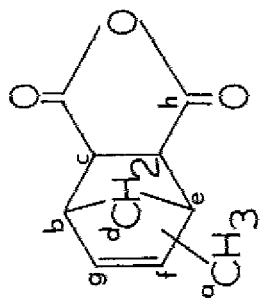
H49



Spectrum 49 — 1,4,5,6,7,7-Hexachloro-5-norbornene-2,3-dicarboxylic anhydride (Aldrich 10,326-8); solvent: acetone-d₆

C50

Methyl-4-endo-methylene Tetrahydrophthalic Anhydride



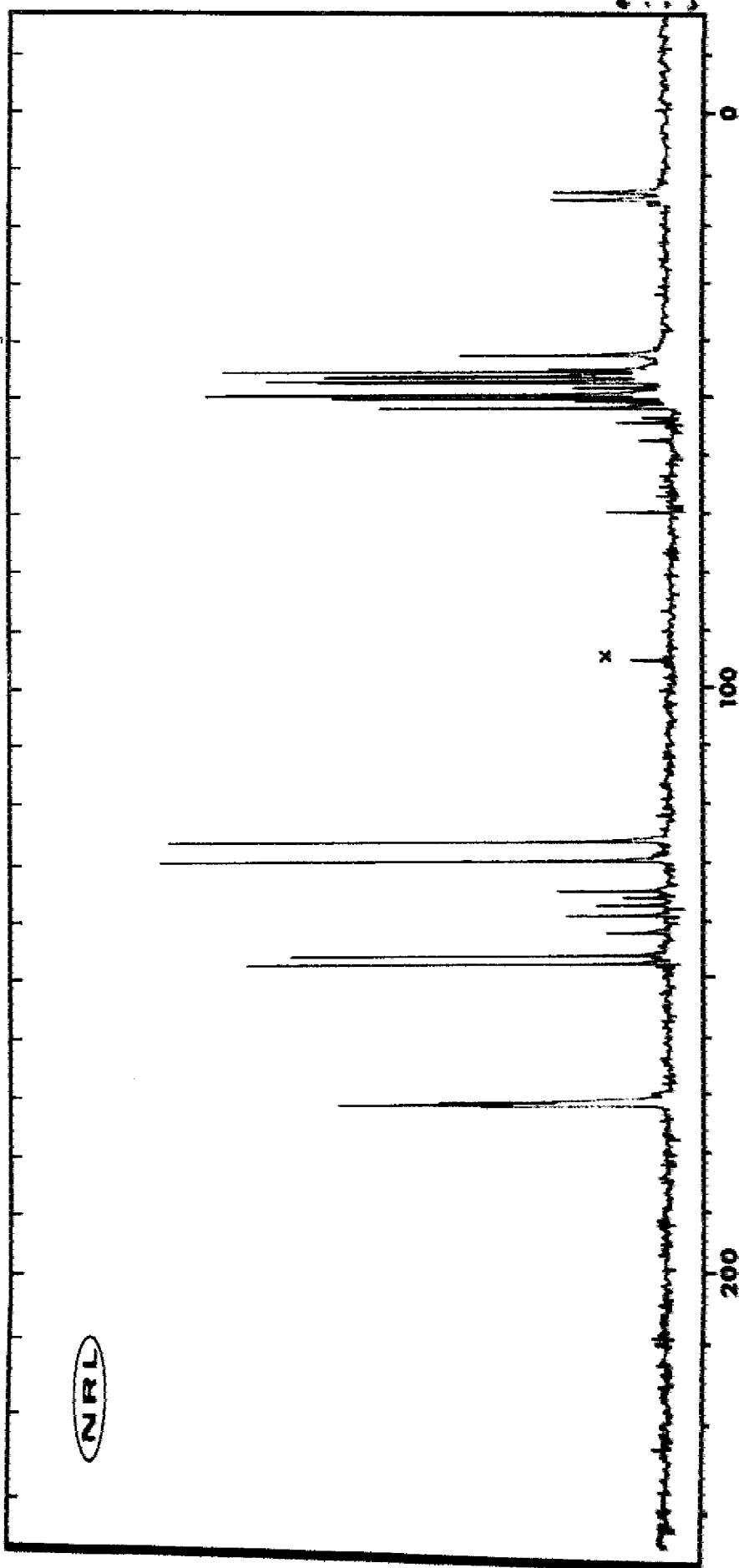
Assignments:

a 15-16.5	f 127-148
b 40-55	g 127-148
c 40-55	h 170-173
d 40-55	
e 40-55	

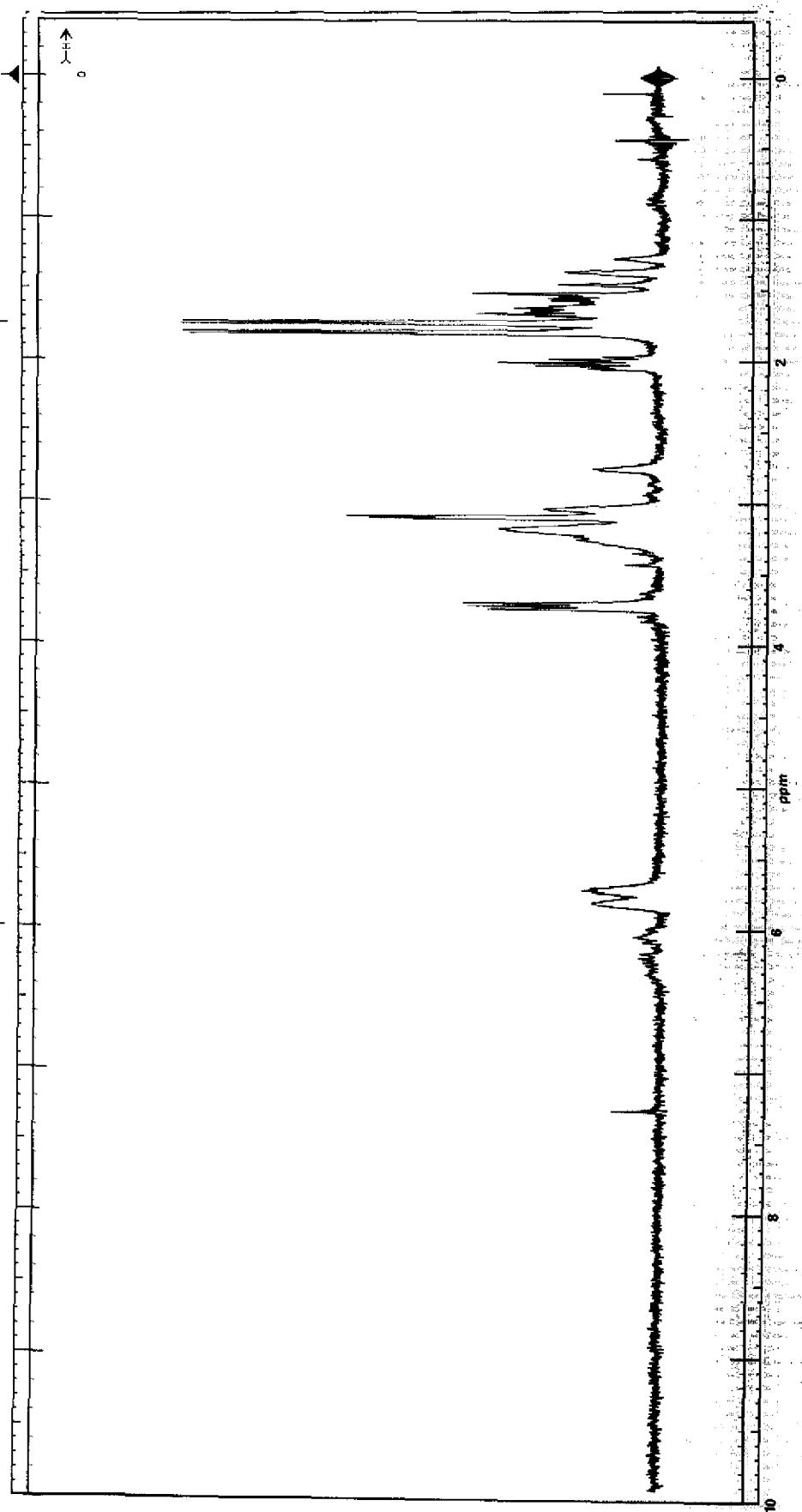
See Appendix for complete line listing.

Source: CIBA 906

Solvent: 10% CCl_4 *



H50



Spectrum 50 — Methyl-4-*endo*-methylene tetrahydronaphthalic anhydride (CIBA 906); solvent: acetone-d₆