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CARBON-13 AND PROTON NMR SPECTRA FOR CHARACTERIZING THERMOSETTI--ETC(U)
JUN 77 C F PORANSKI, W B MONIZ, D L BIRKLE

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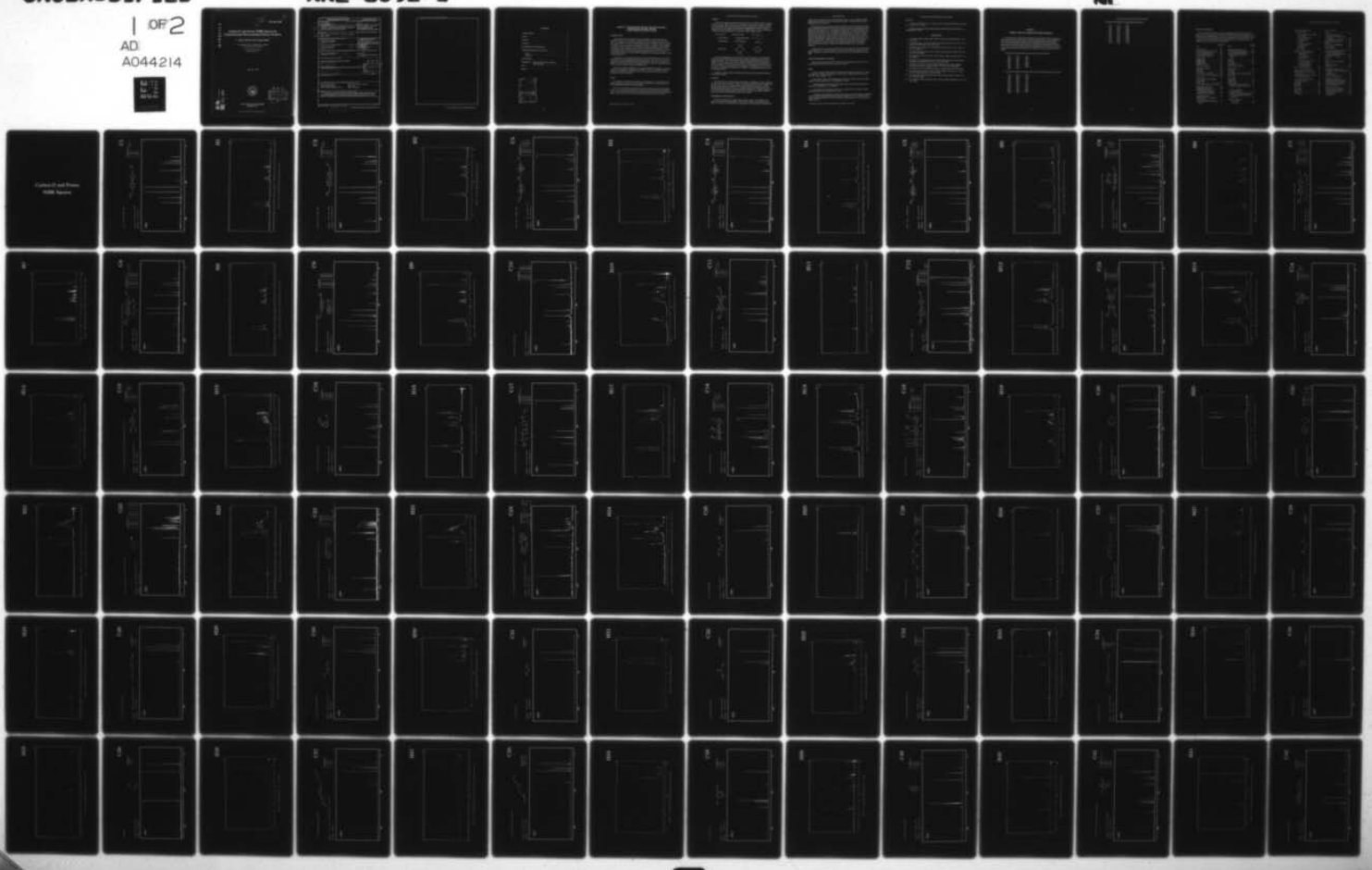

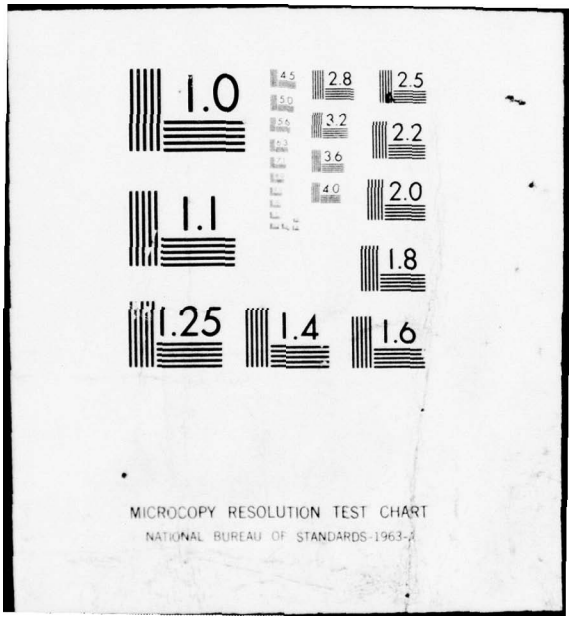
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NRL Report 8092

Carbon-13 and Proton NMR Spectra for Characterizing Thermosetting Polymer Systems

I: Epoxy Resins and Curing Agents

C. F. Poranski, Jr., W. B. Moniz, D. L. Birkle,
J. T. Kopfle, and S. A. Sojka

Organic Chemistry Branch
Chemistry Division

June 20, 1977

NAVAL RESEARCH LABORATORY
Washington, D.C.

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Epoxy curing agents

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This report presents carbon-13 and proton nuclear magnetic resonance spectra for 24 epoxy resins and 26 curing agents. In most cases the carbon-13 resonance lines have been structurally assigned. The spectra provide data useful for the qualitative analysis of many epoxy resin systems.

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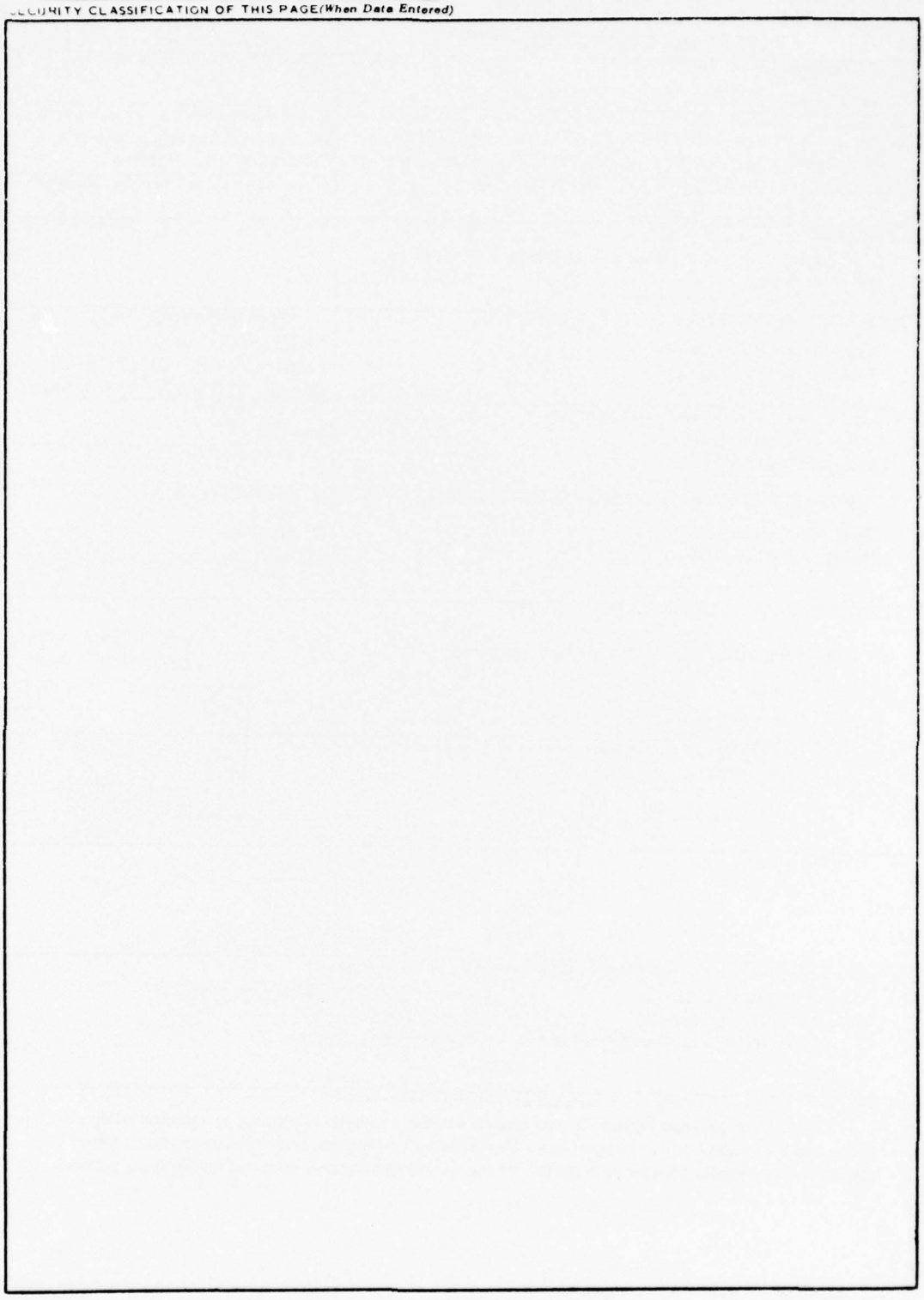
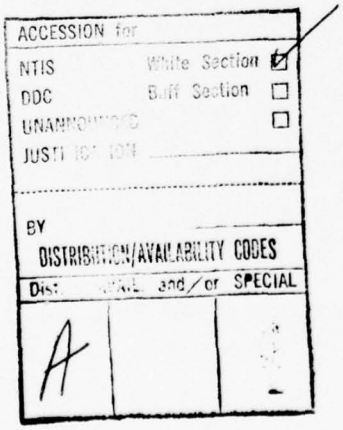


CONTENTS



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

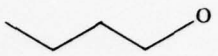
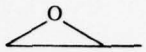
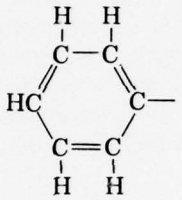
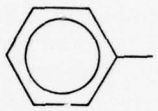
This volume is restricted to epoxy resins and curing agents. A future volume will include plasticizers, more complex epoxy systems, and precursors (or prepolymers) of other thermosetting polymers such as polyimides.

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.

Manuscript submitted December 2, 1976.

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:

Chemical name	*Chemical formula*	*Brief version*
Butoxy group	$CH_3CH_2CH_2CH_2O-$	
Glycidyl group	$H_2C\overset{O}{\diagdown\diagup}CHCH_2-$	
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 spectral window was 250 ppm and collection size was either 4000 or 8000 data points. Data were collected and processed with either a Nicolet 1080 or Nicolet 1180 NMR data system. Sample 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 Thermodynamic 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-Epoxycyclohexylmethyl-(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 tetrahydrophthalic 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

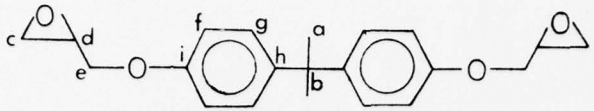
INDEX OF MATERIALS

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.

Carbon-13 and Proton NMR Spectra

C1

Epoxy Resin, DGEBA Type

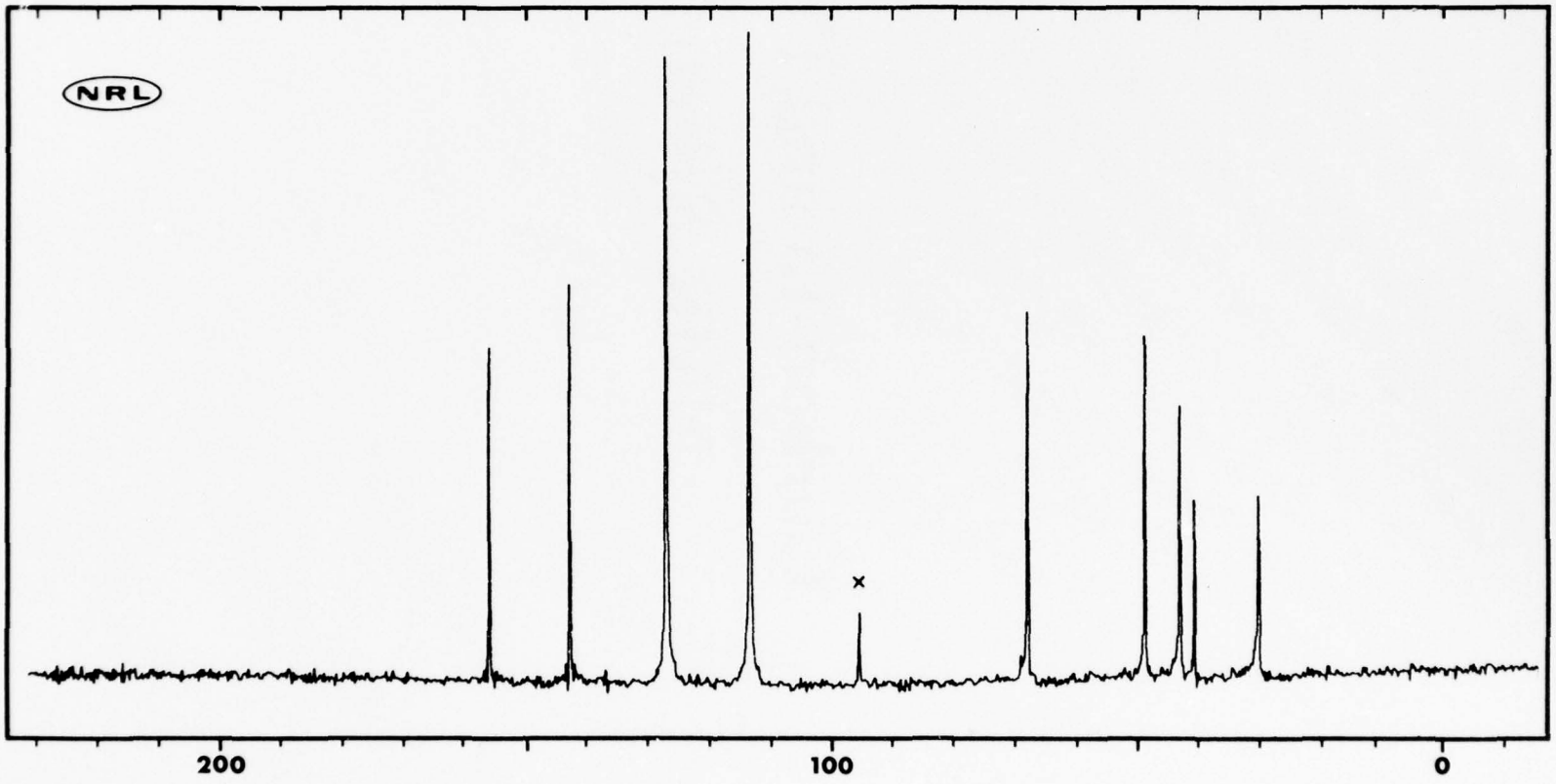


Assignments:

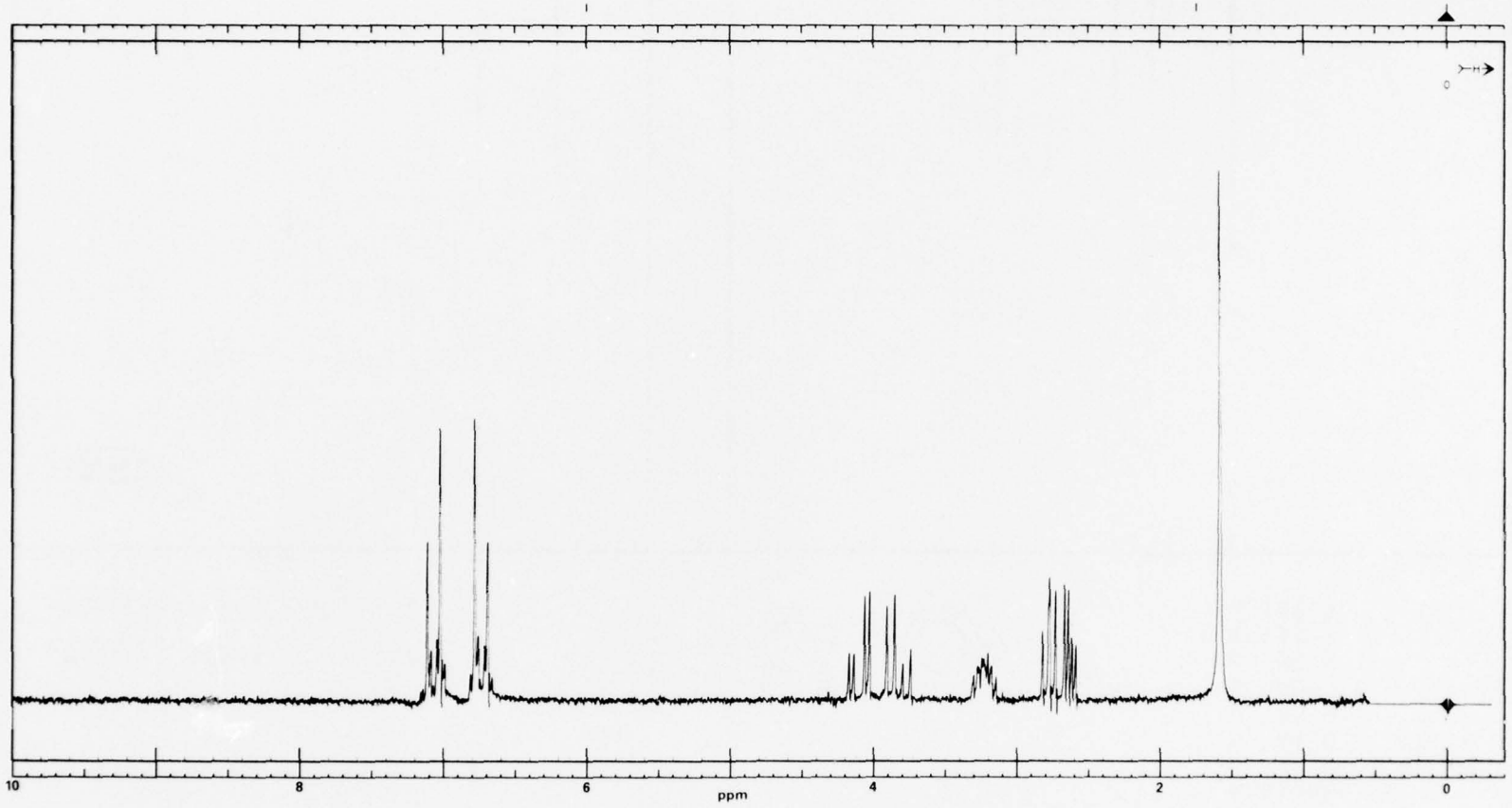
a	30.1	h	142.4
b	40.7	i	155.4
c	43.1		
d	49.1		
e	68.1		
f	113.2		
g	126.7		

Source: Dow D.E.R. 332LC

Solvent: 50% CCl_4 ×



H1



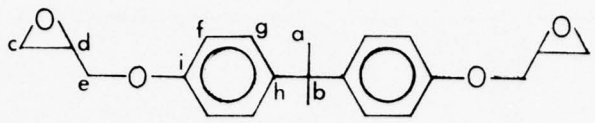
Spectrum 1 — Epoxy resin, DGEBA type (Dow D.E.R. 332LC); solvent: $CDC1_3$

C2

Epoxy Resin, DGEBA Type

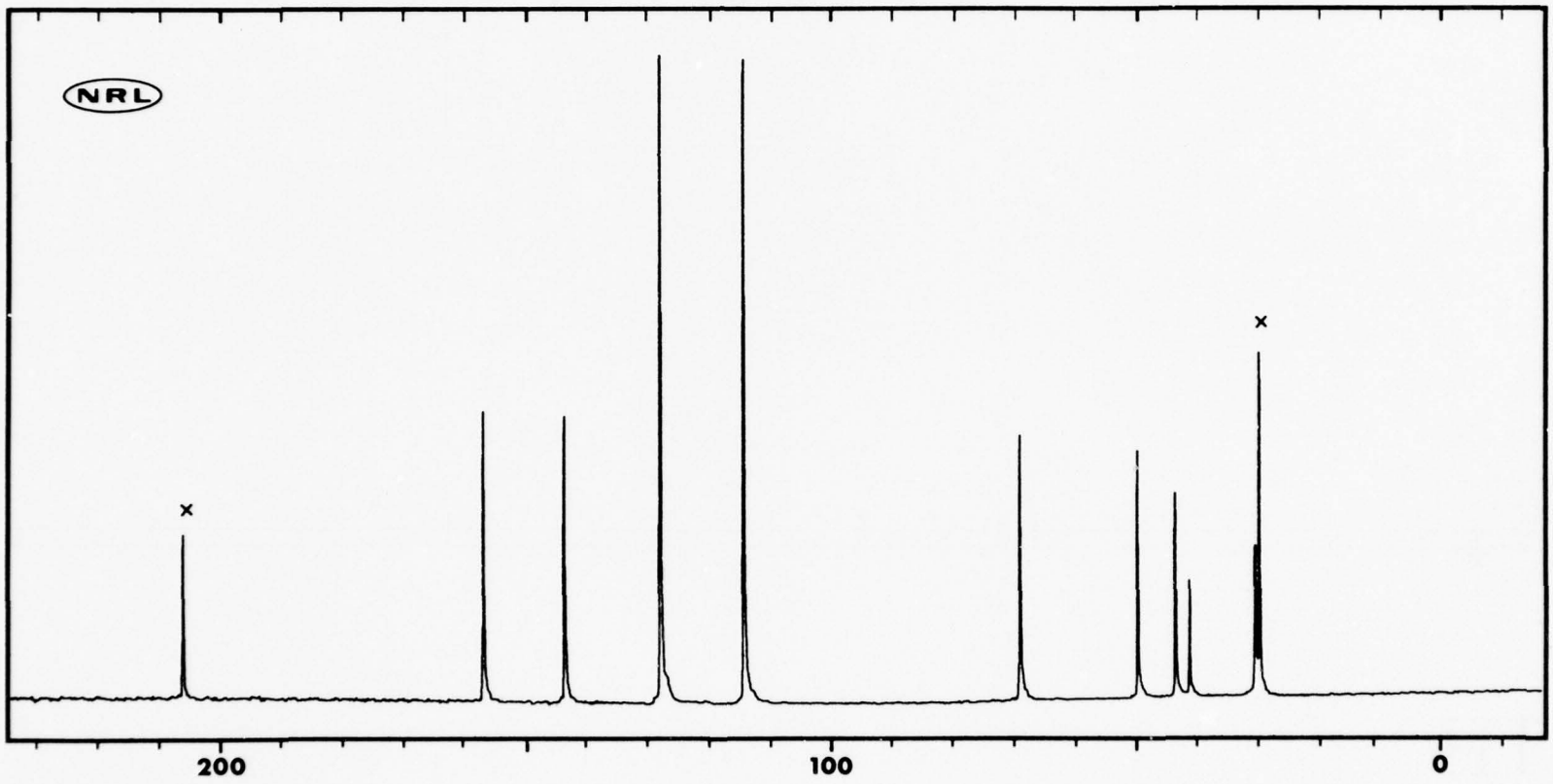
Source: Shell Epon 828

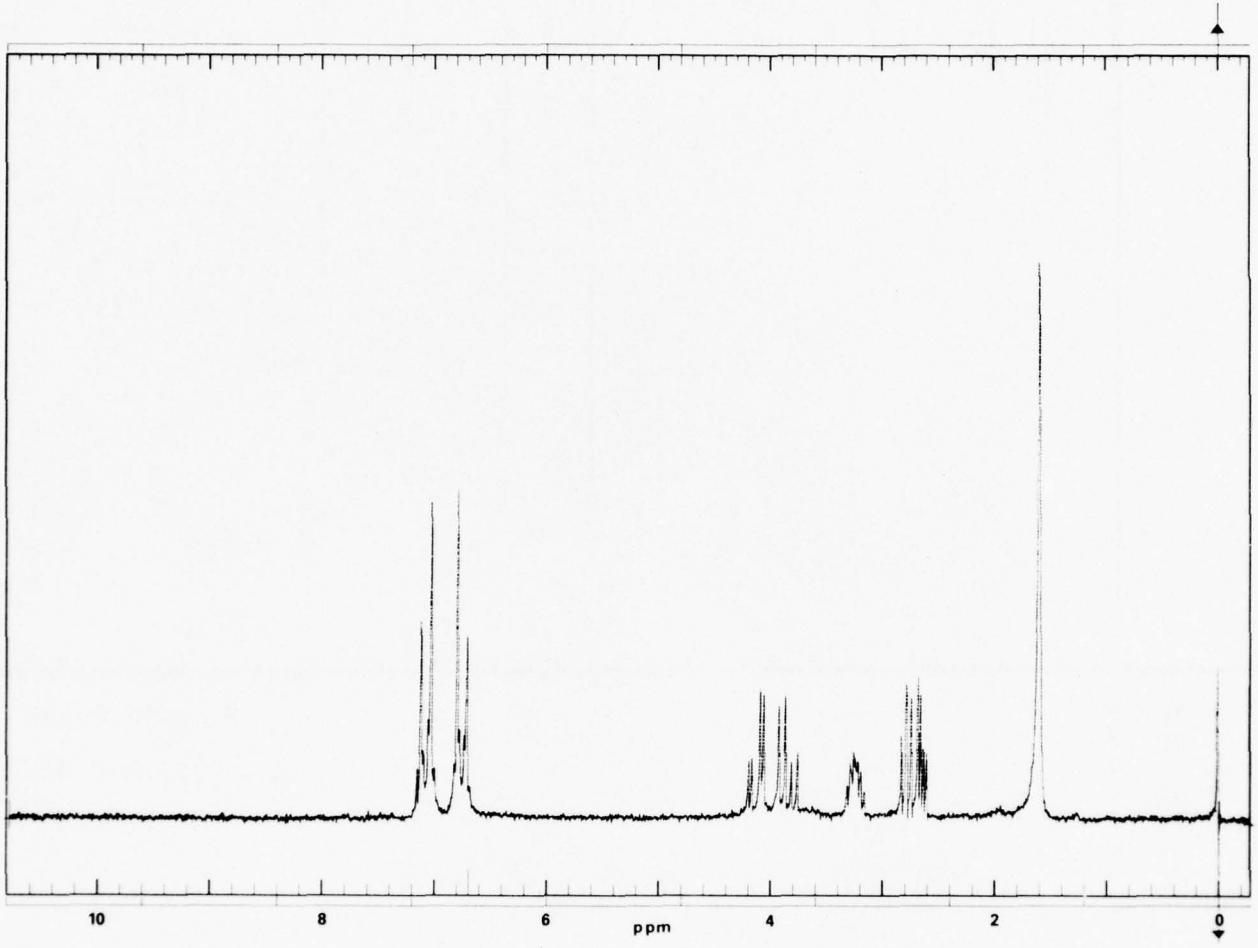
Solvent: 50% Acetone ×



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		

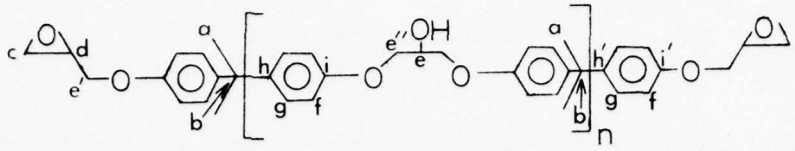




Spectrum 2 -- Epoxy resin, DGEBA type (Shell Epon 828); solvent: $CDCl_3$

C3

Epoxy Resin, DGEBA Type

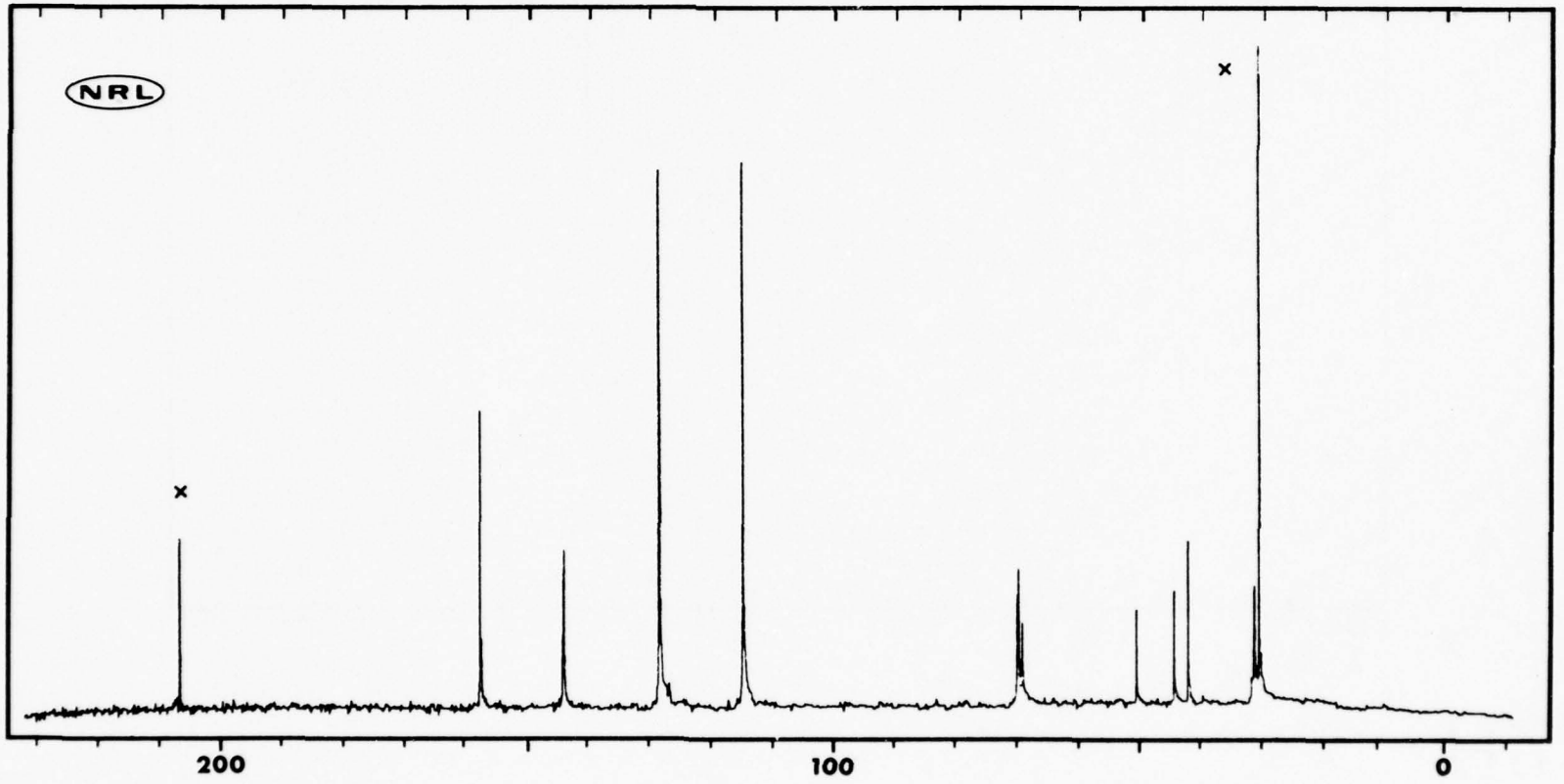


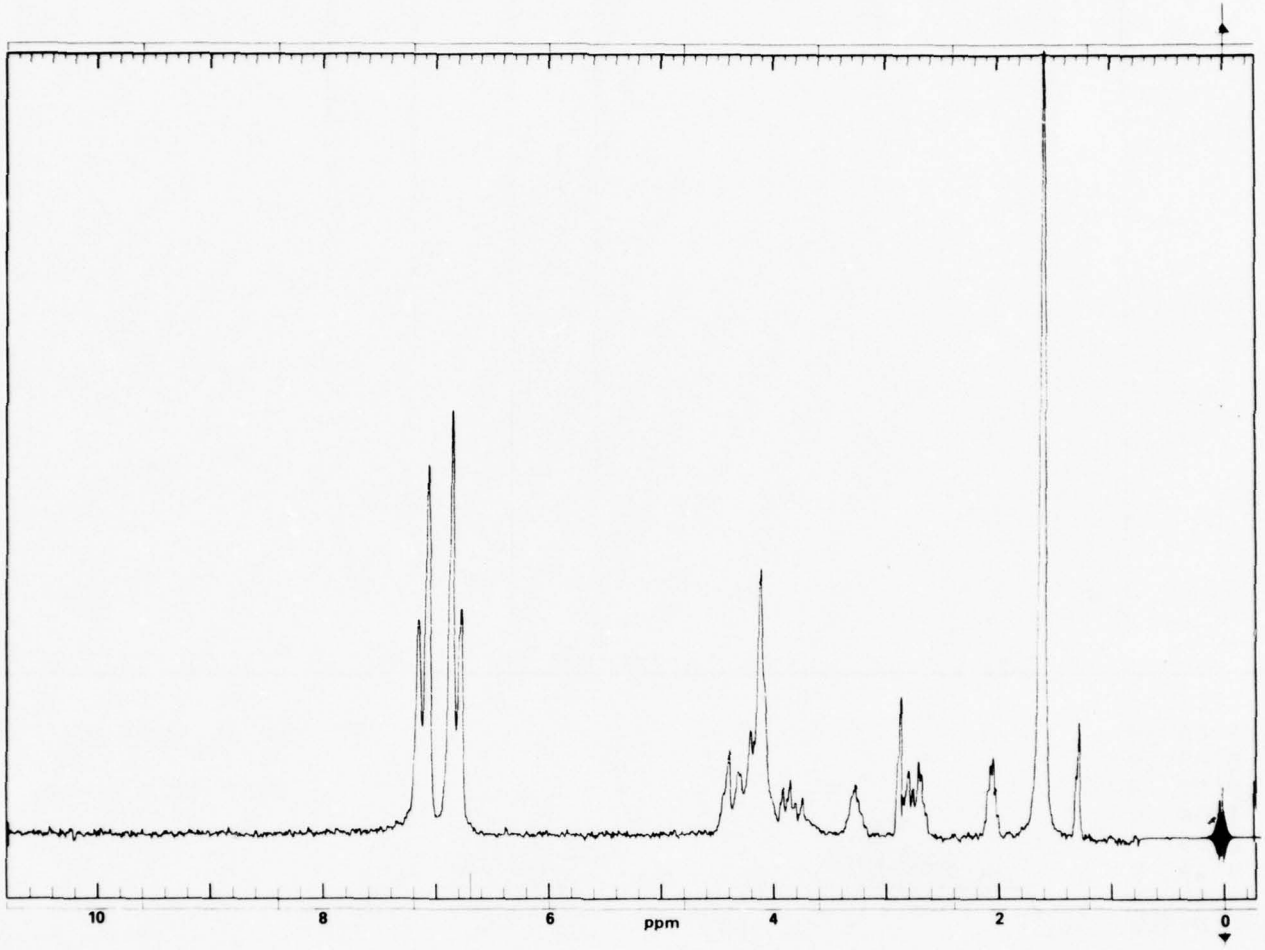
Source: Ciba Araldite 7071

Solvent: 50% Acetone ×

Assignments:

a	30.9	f	114.1
b	41.6	g	127.7
c	44.0	h	143.2
d	50.1	h'	143.4
e	68.7	i'	156.6
e'	69.3	i	156.8
e''	69.5		

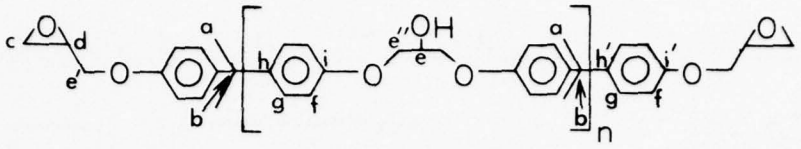




Spectrum 3 — Epoxy resin, DGEBA type (Ciba Araldite 7071); solvent: acetone-d_6

C4

Epoxy Resin, DGEBA Type

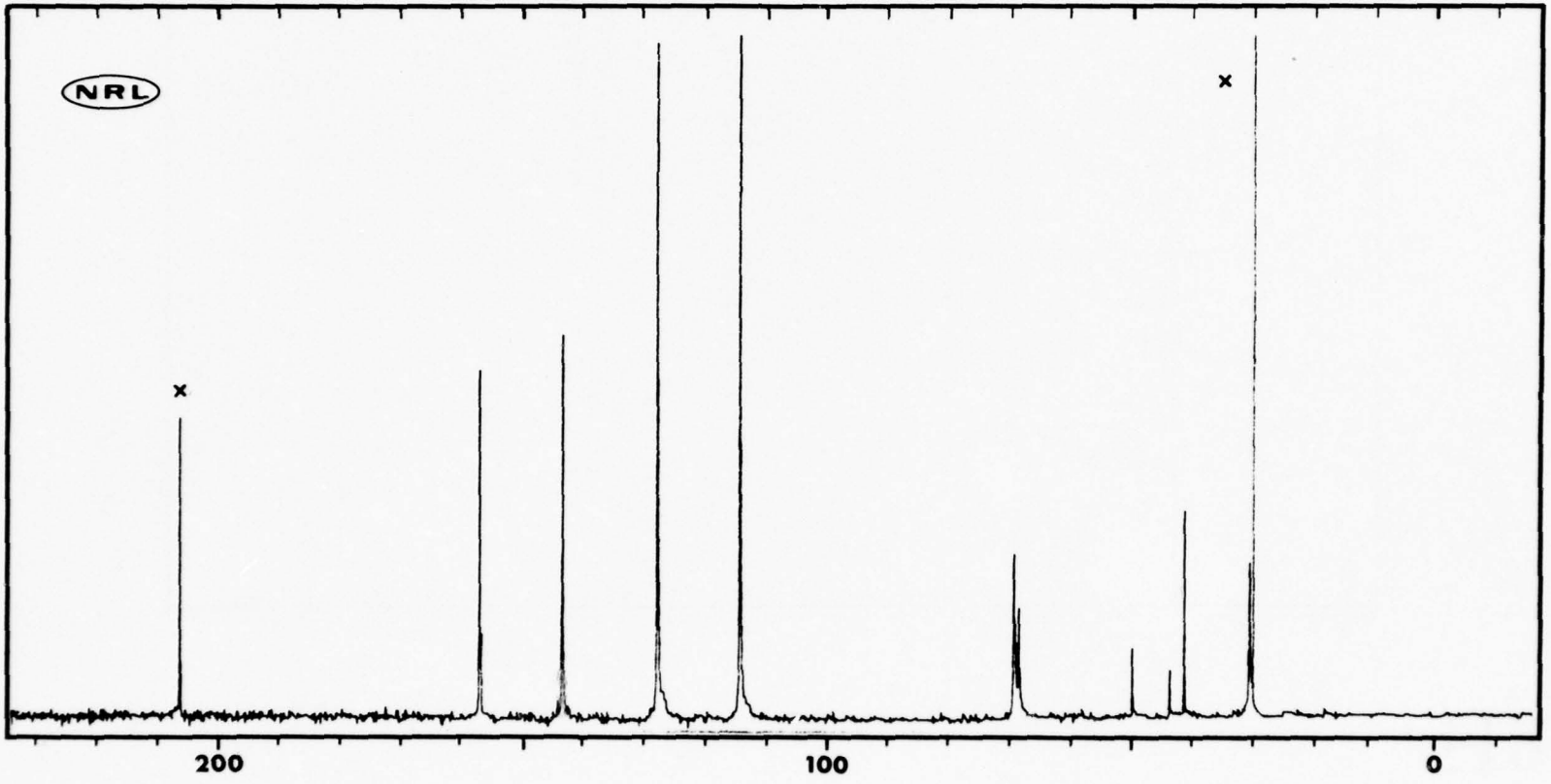


Source: Shell Epon 1004

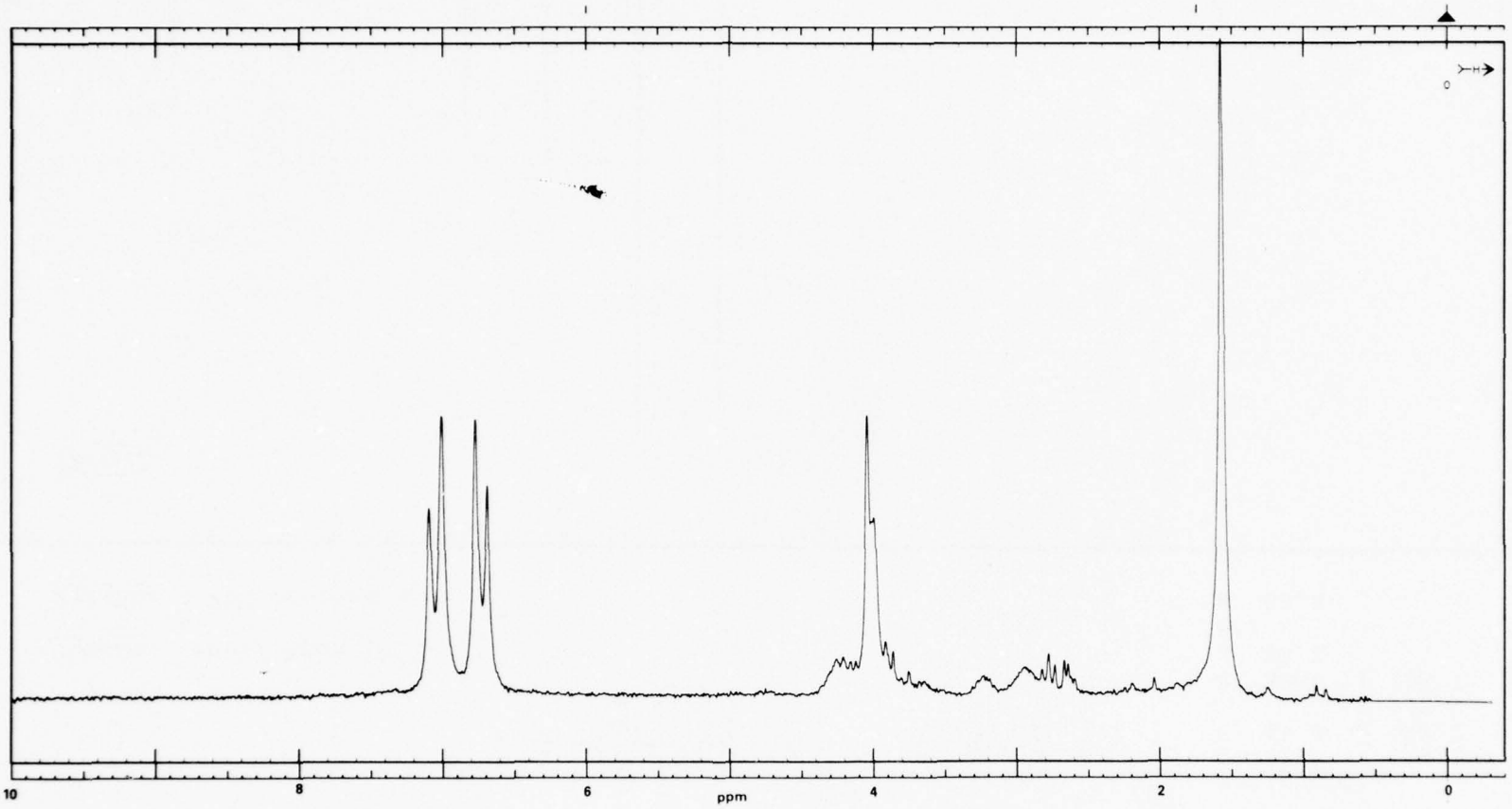
Solvent: 75% Acetone ×

Assignments:

a	30.9	f	114.1
b	41.6	g	127.7
c	44.0	h	143.1
d	50.1	h'	143.3
e	68.6	i'	156.5
e'	69.2	i	156.7
e''	69.4		



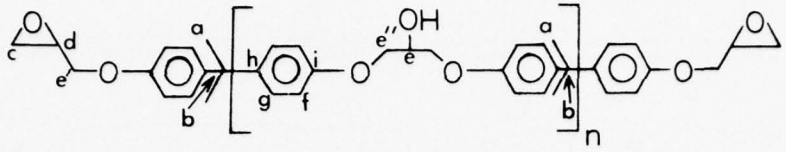
H4



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

C5

Epoxy Resin, DGEBA Type

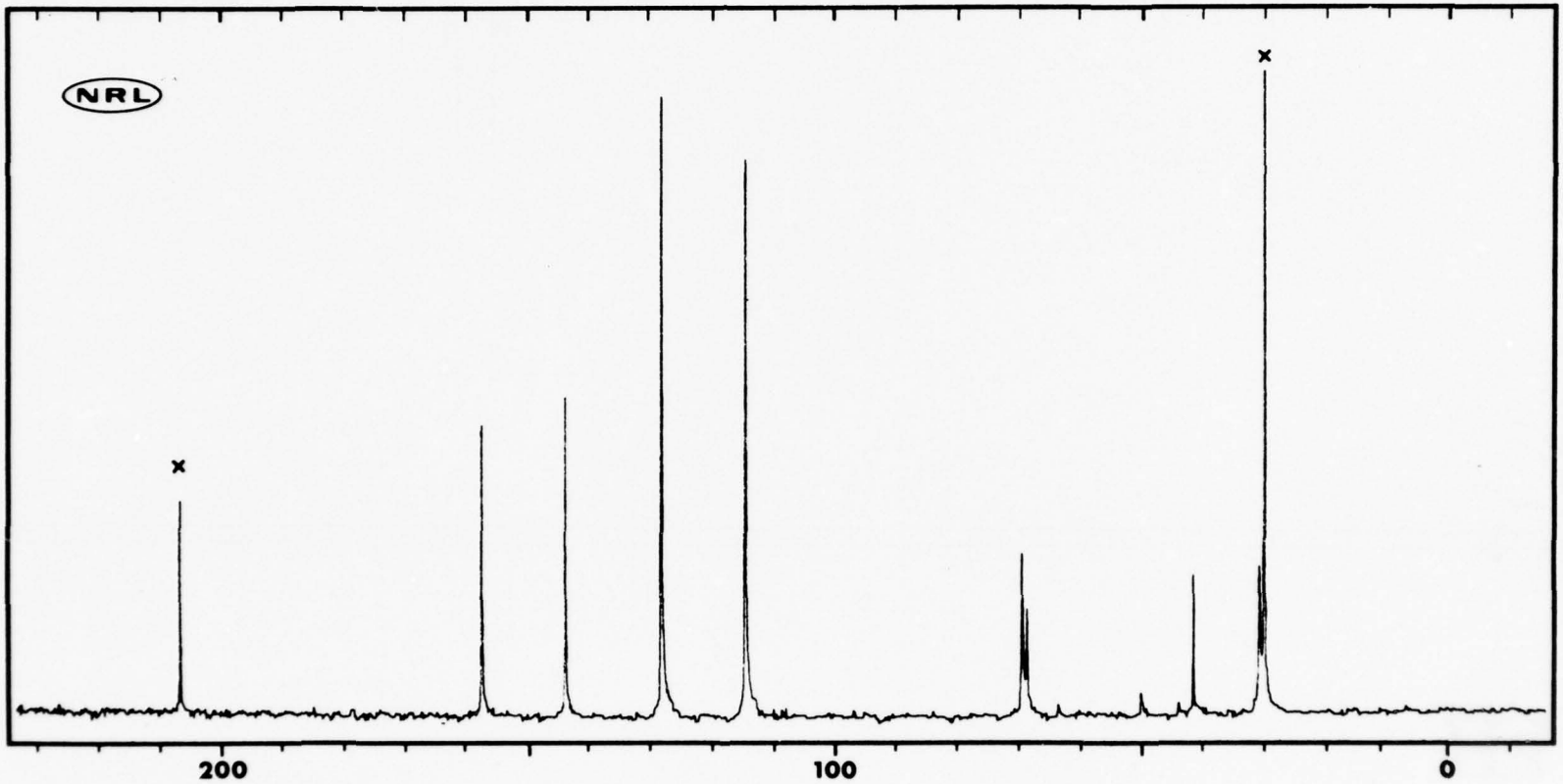


Source: Shell Epon 1007

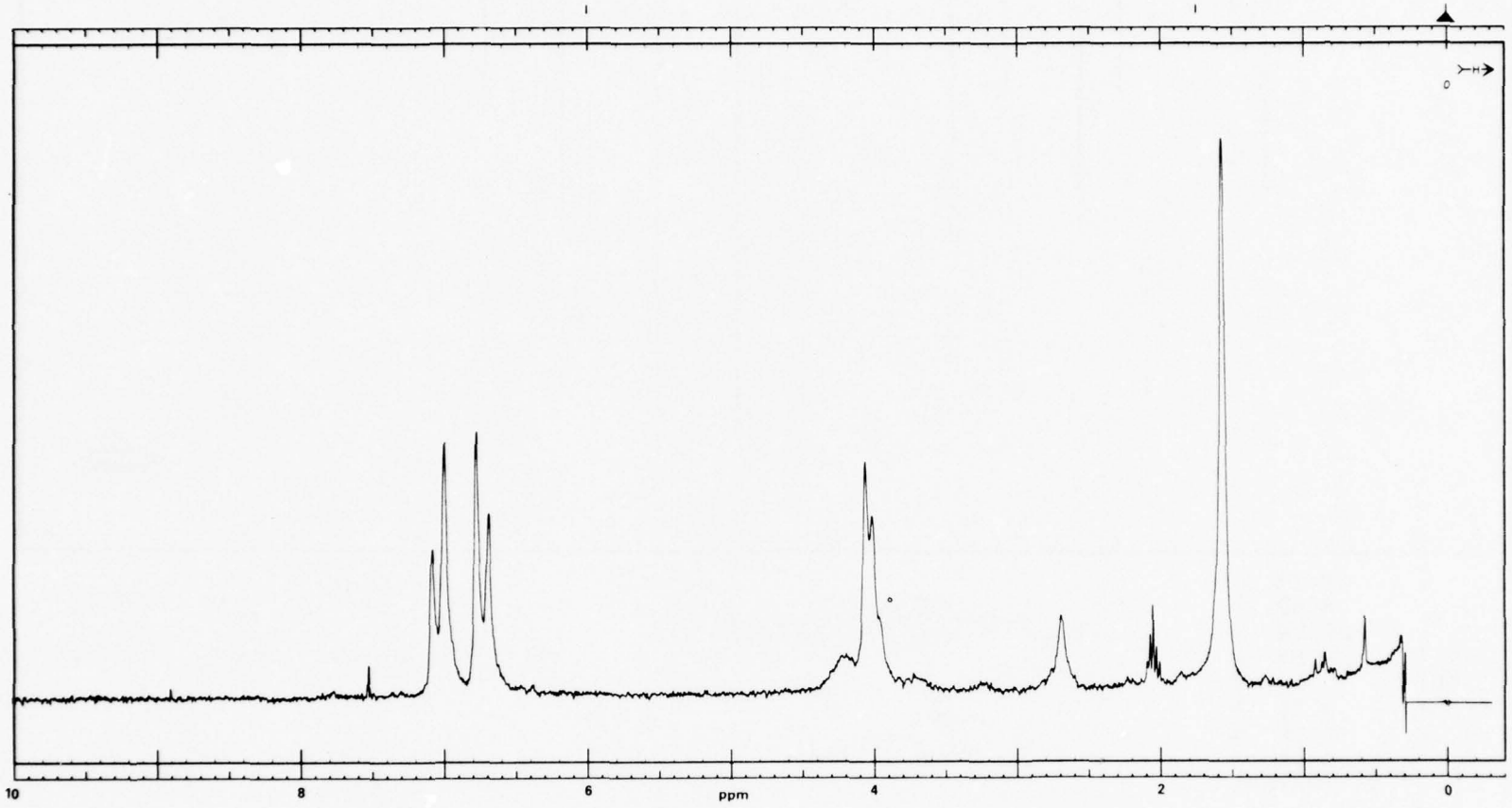
Solvent: 75% Acetone ×

Assignments:

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



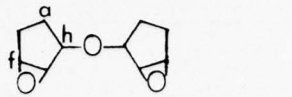
H5

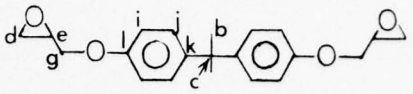


Spectrum 5 — Epoxy resin, DGEBA type (Shell Epon 1007); solvent: $CDCl_3$ and acetone-d_6

C6

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



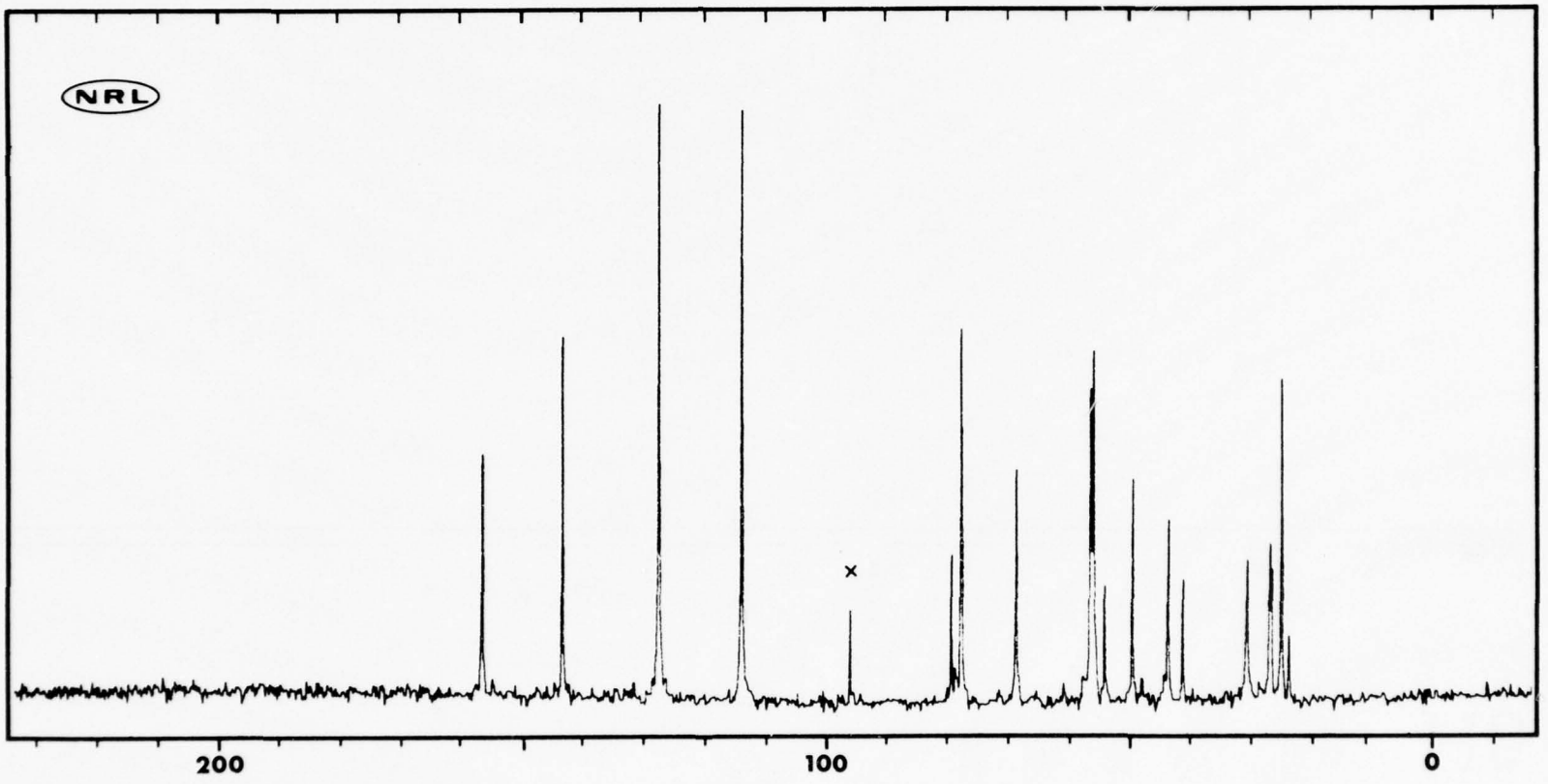


Source: Union Carbide ERL 2258

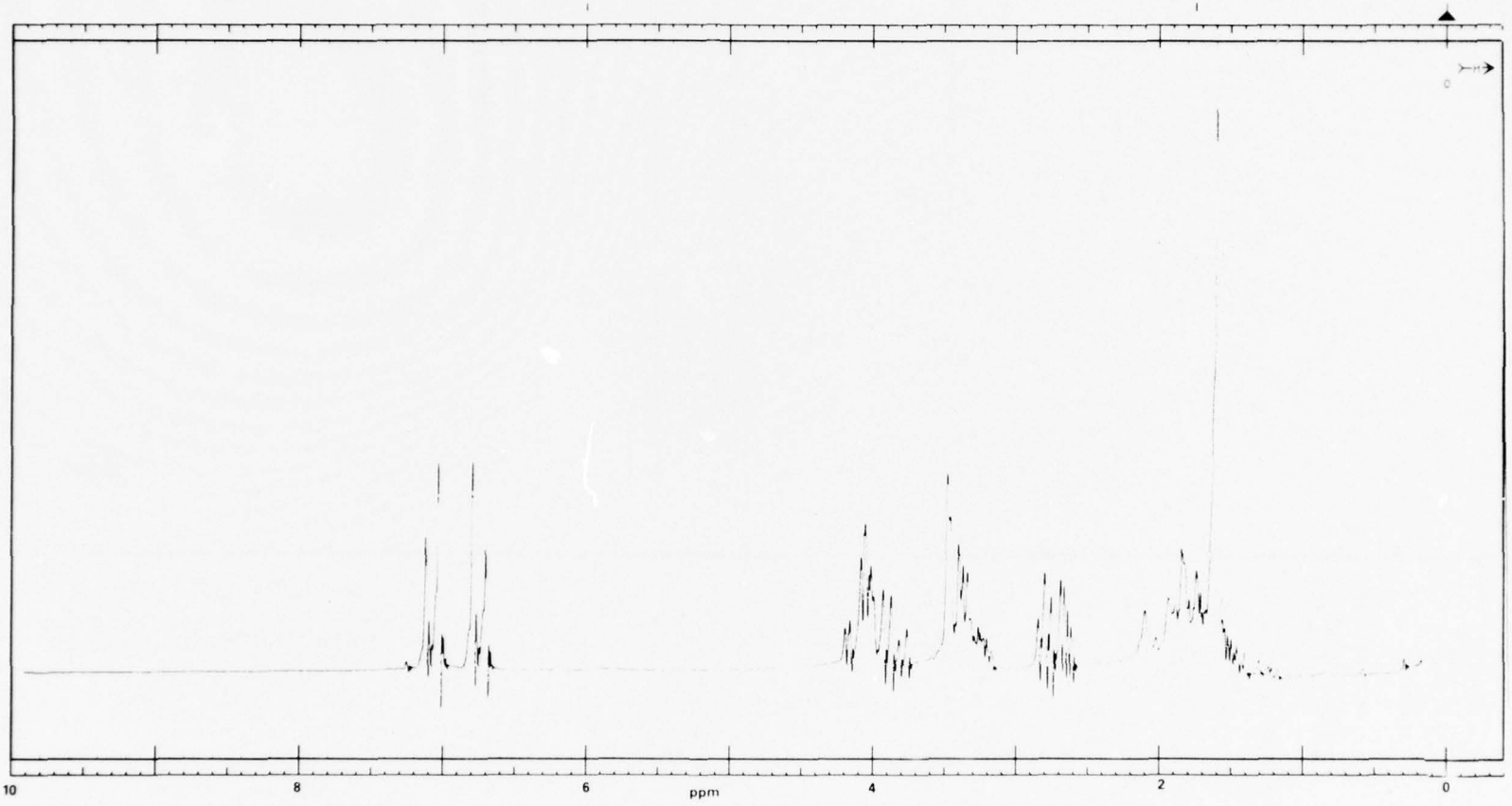
Solvent: 25% CCl_4 ×

Assignments:

a	24-27.5		
b	31.1	i	113.9
c	41.6	j	127.4
d	44.0	k	143.1
e	49.8	l	156.2
f	55-56		
g	68.9		
h	77-80		



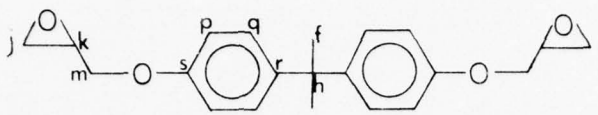
H6

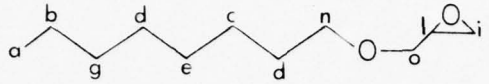


Spectrum 6 — DGEBA type epoxy resin plus *bis*(2,3-epoxycyclopentyl)ether (Union Carbide ERL 2258); solvent: $CDC1_3$

C7

DGEBA Type Epoxy Resin plus n-Octyl Glycidyl Ether



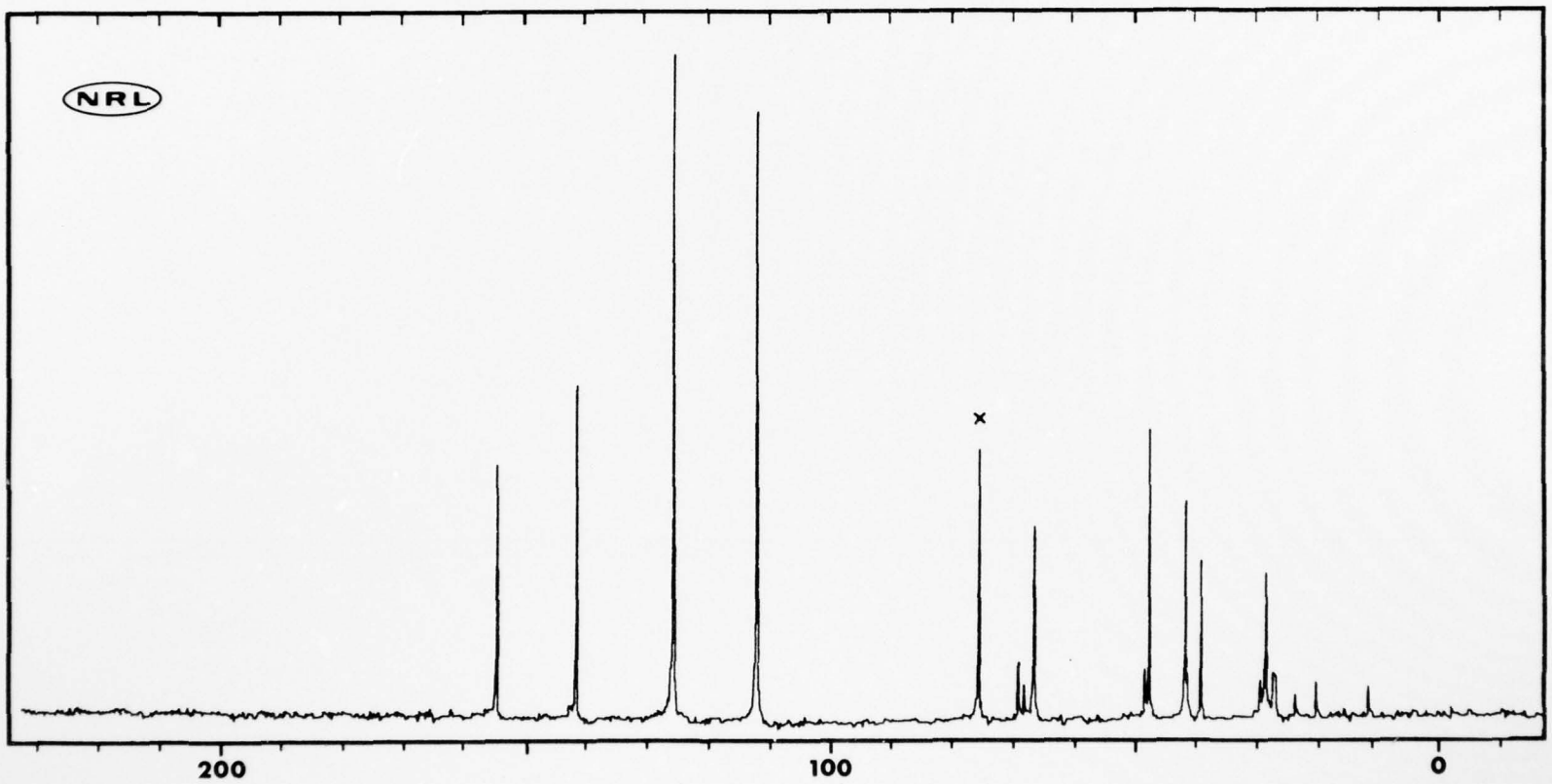


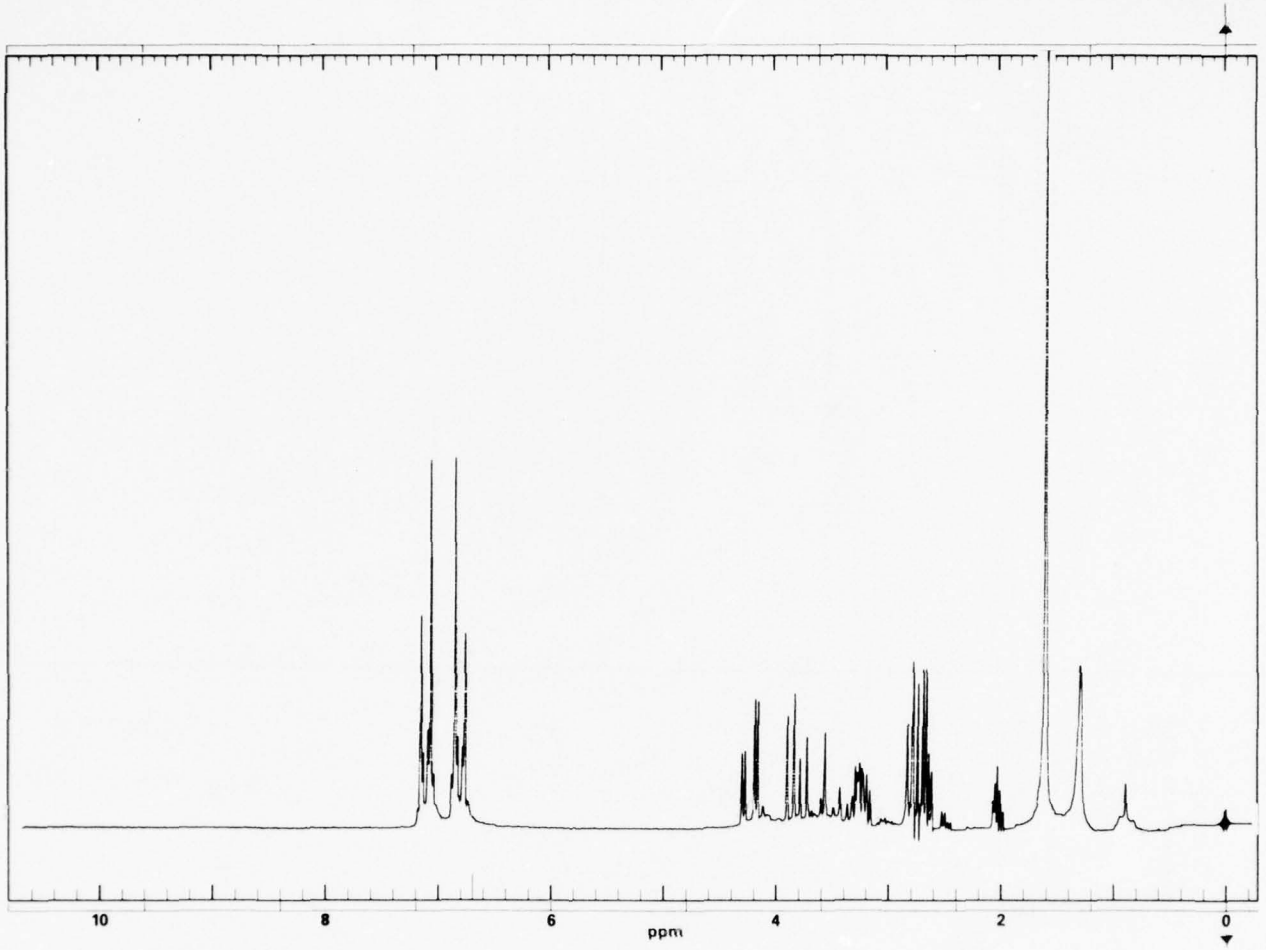
Source: Genepoxy M195

Solvent: 25% $CHCl_3$ ×

Assignments:

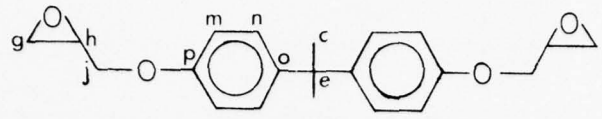
a	13.5	h	40.9	o	70.8
b	22.0	i	43.1	p	113.3
c	25.4	j	43.5	q	126.8
d	28.7	k	49.3	r	142.5
e	29.0	l	50.1	s	155.6
f	30.3	m	68.2		
g	31.2	n	69.8		

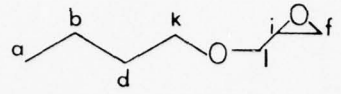




Spectrum 7 — DGEBA type epoxy resin plus *n*-octyl glycidyl ether (Genepoxy M195); solvent: acetone-d_6

DGEBA Type Epoxy Resin plus Butyl Glycidyl Ether



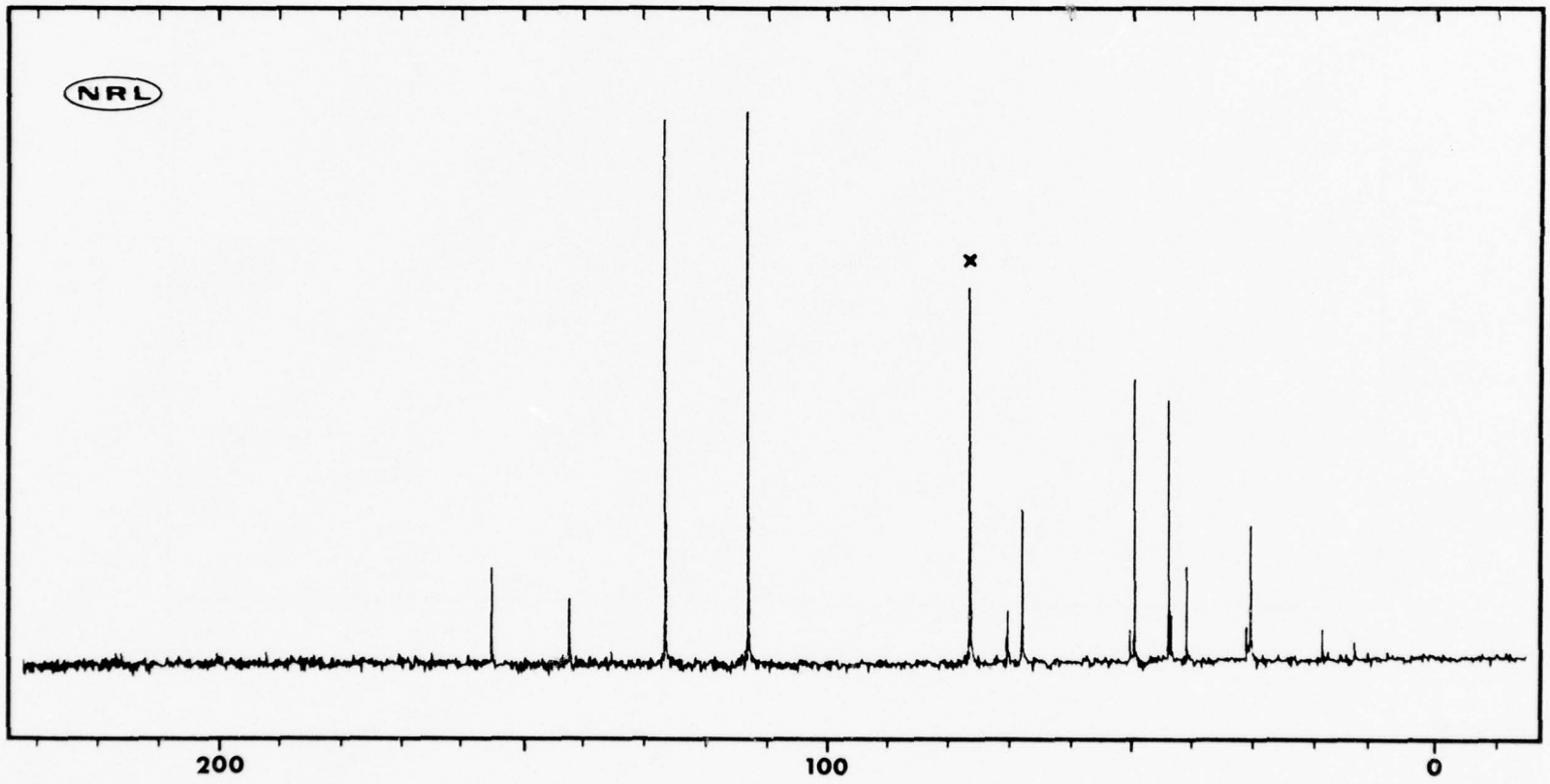


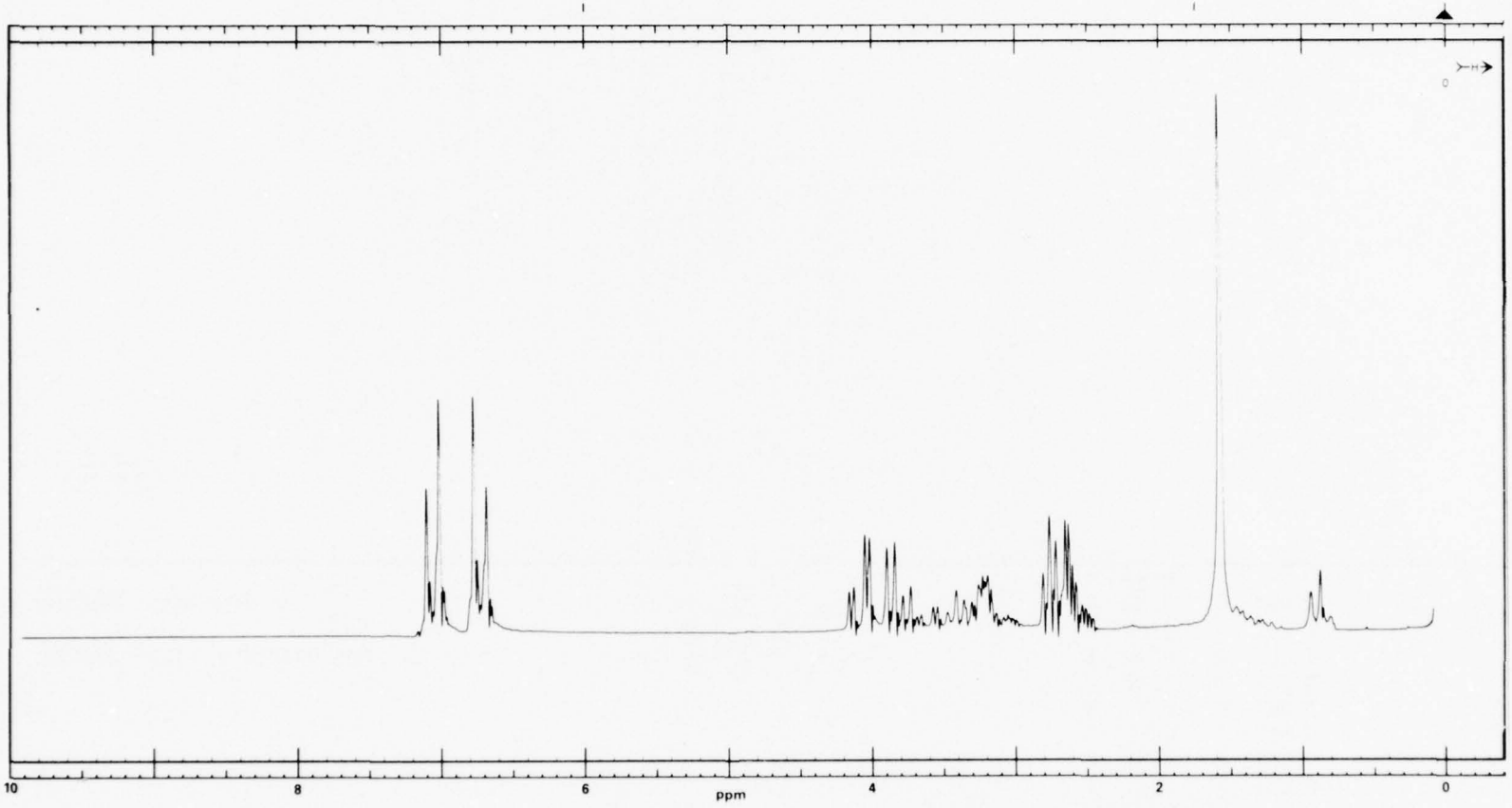
Source: Shell Epon 815

Solvent: 25% $CHCl_3$ ×

Assignments:

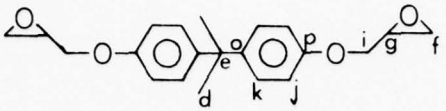
a	13.2	h	49.2	o	142.4
b	18.5	i	50.0	p	155.5
c	30.2	j	68.1		
d	31.1	k	70.2		
e	40.8	l	70.7		
f	42.9	m	113.2		
g	43.3	n	126.7		

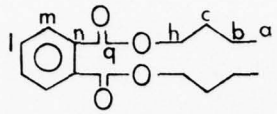




Spectrum 8 — DGEBA type epoxy resin plus butyl glycidyl ether (Shell Epon 815); solvent: $CDCl_3$

DGEBA Type Epoxy Resin plus Di-n-butyl Phthalate



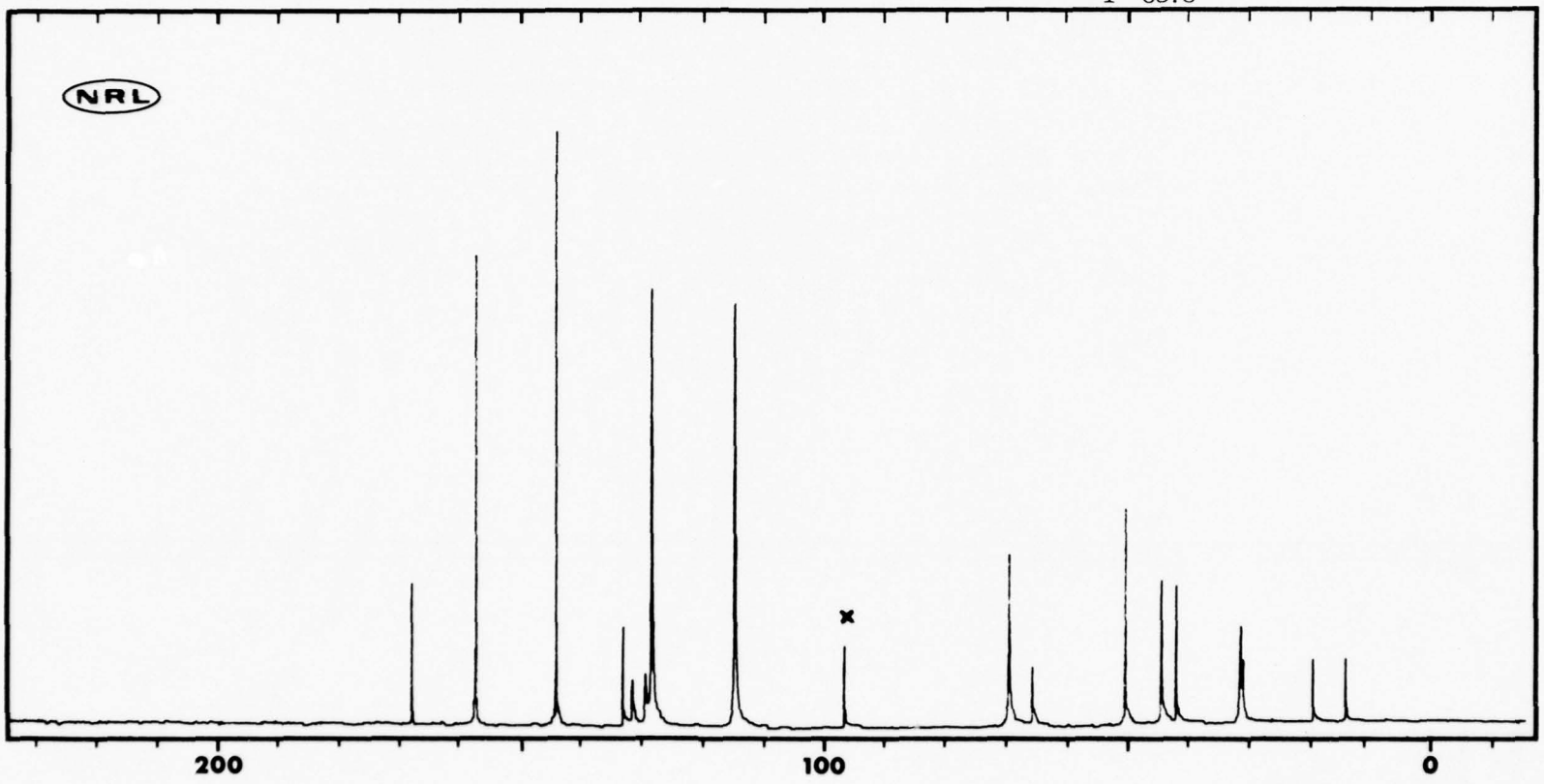


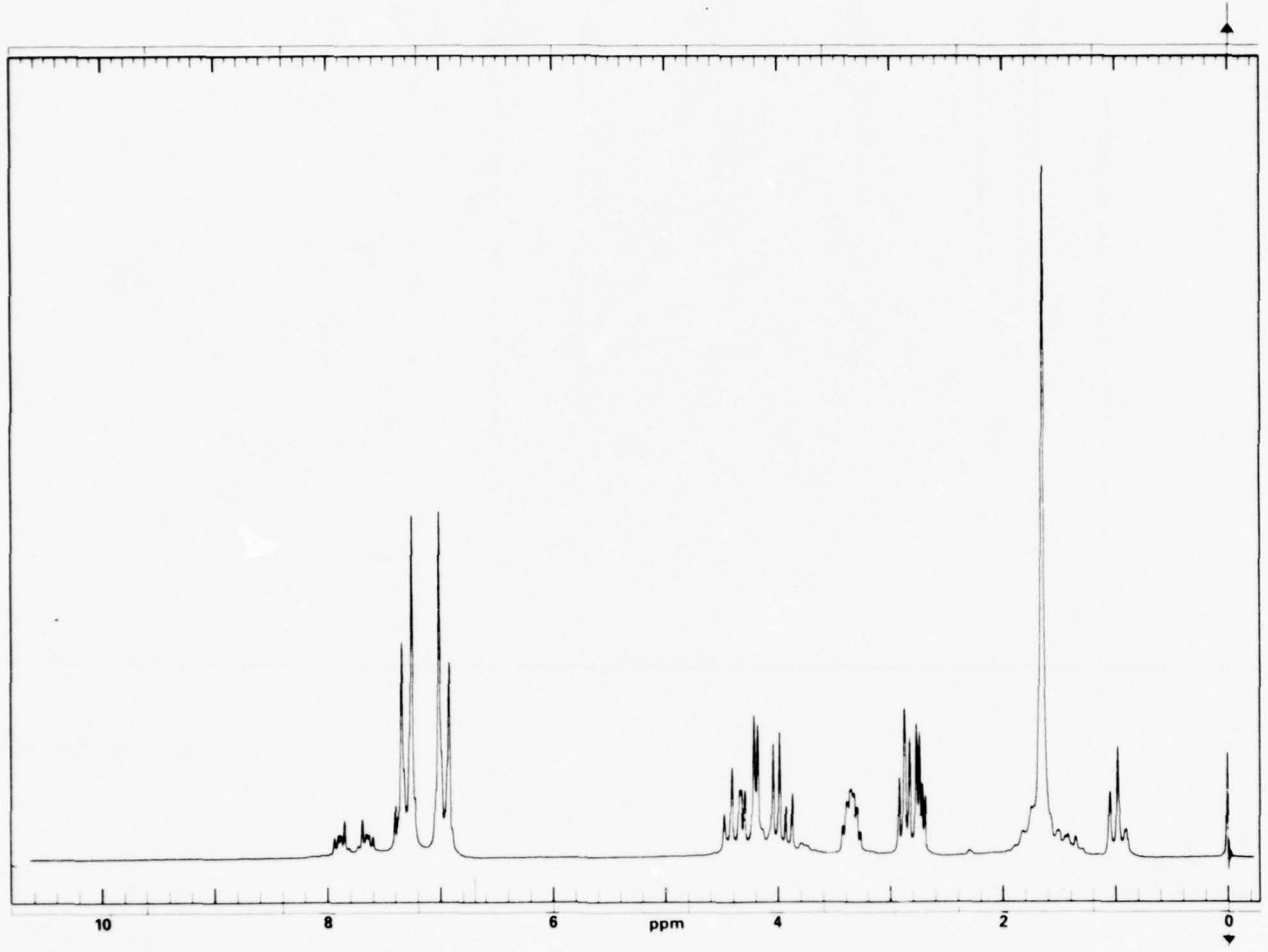
Source: Ciba Araldite 502

Solvent: 20% CCl_4 ×

Assignments:

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





Spectrum 9 — DGEBA type epoxy resin plus di-*n*-butyl phthalate (Ciba Araldite 502); solvent: $CDCl_3$

C10

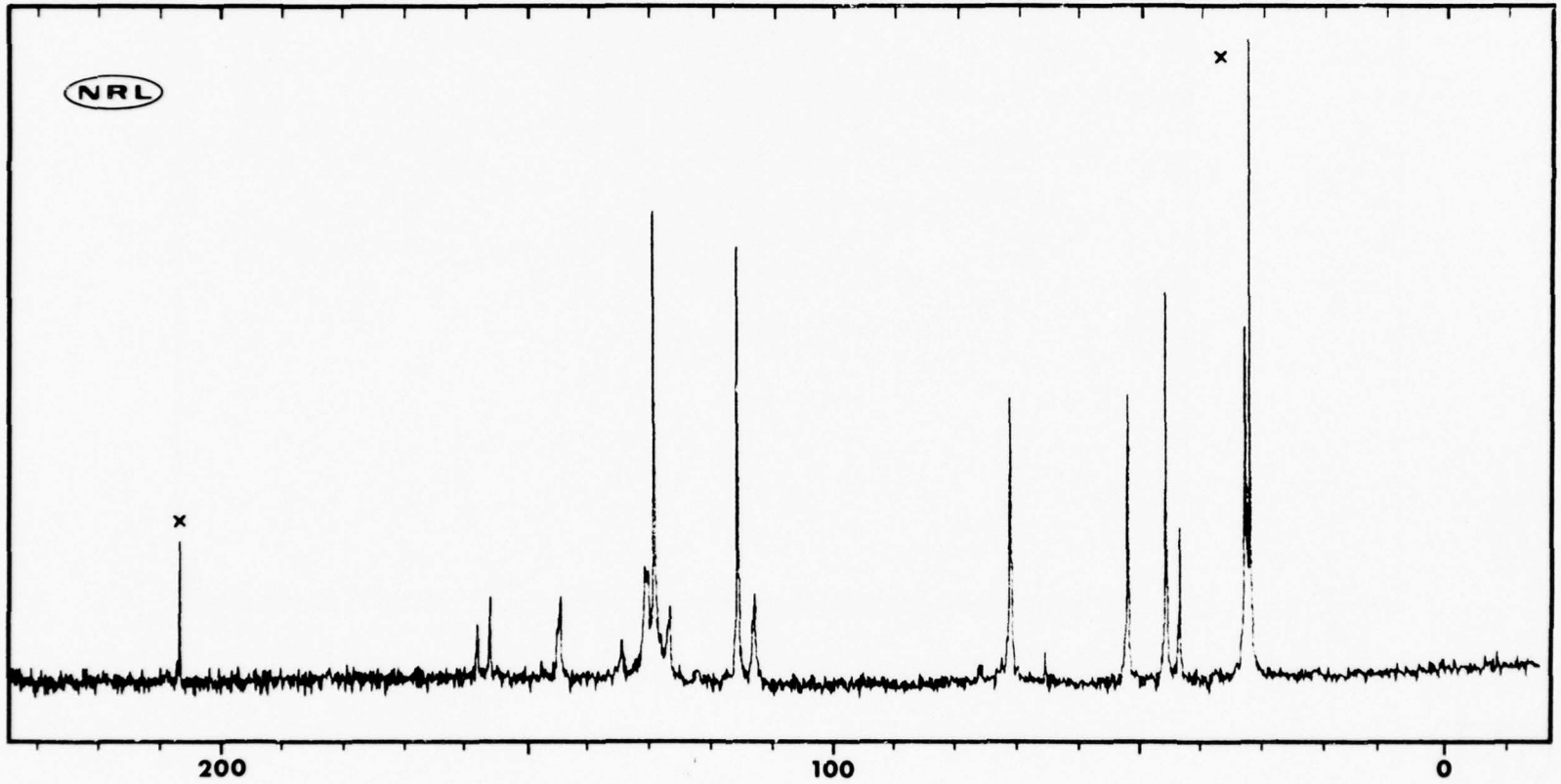
Epoxy Resin, Polyfunctional

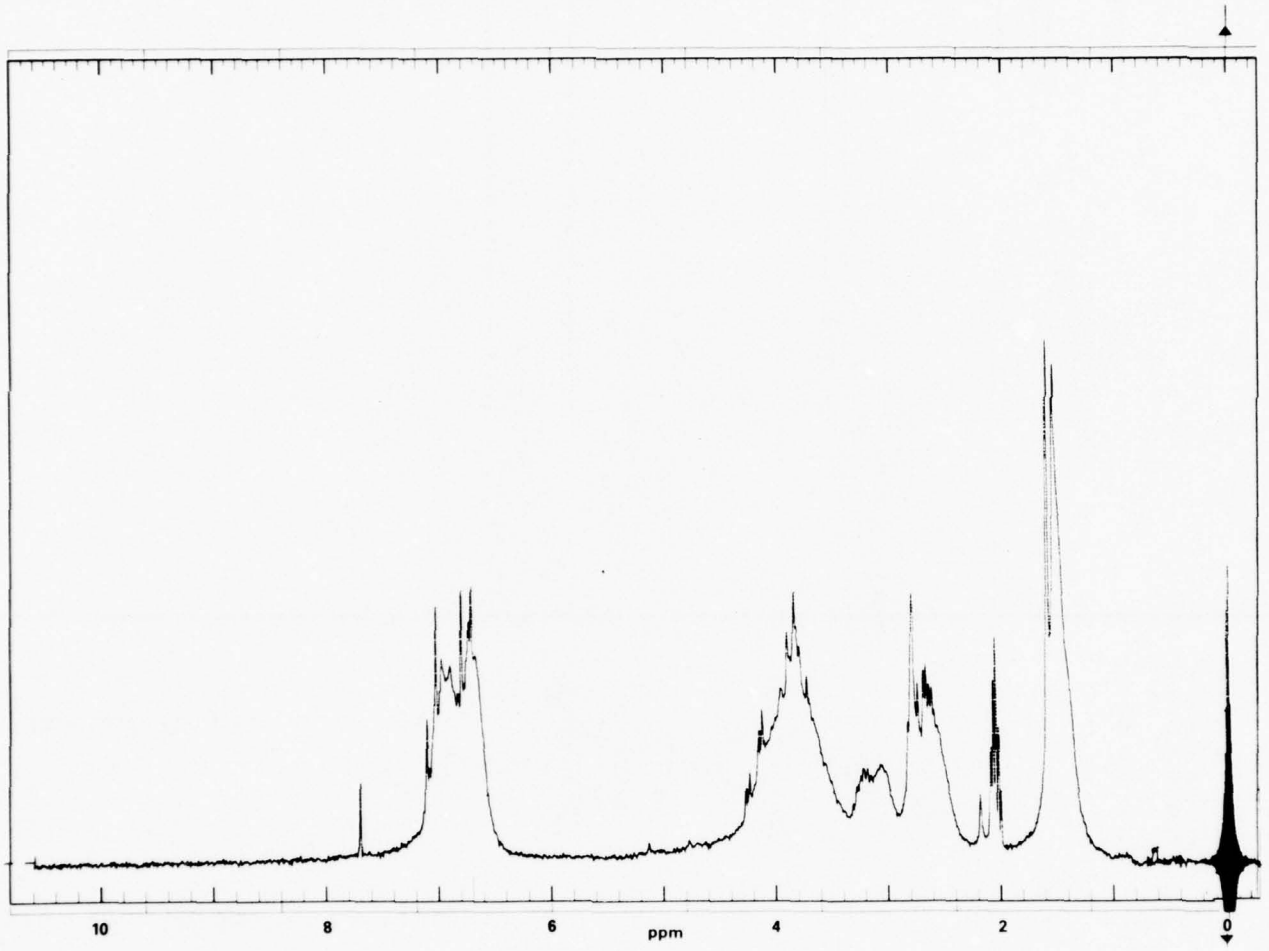
Source: Celanese Epi-Rez SU-8

Solvent: 50% Acetone ×

Assignments:

a	30.9	h	124.7
b	41.6	i	127.3
c	43.9	j	128.8
d	50.0	k	134.0
e	69.2	l	142.6
f	111.0	m	154.0
g	113.7	n	156.0

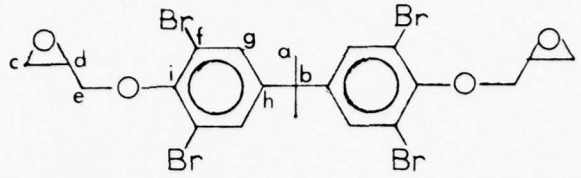




Spectrum 10 — Epoxy resin, polyfunctional *(Celanese Epi-Rez SU-8); solvent: acetone-d_6 and $CDCl_3$ (1:1)*

C11

Diglycidyl Ether of Tetrabromobisphenol A

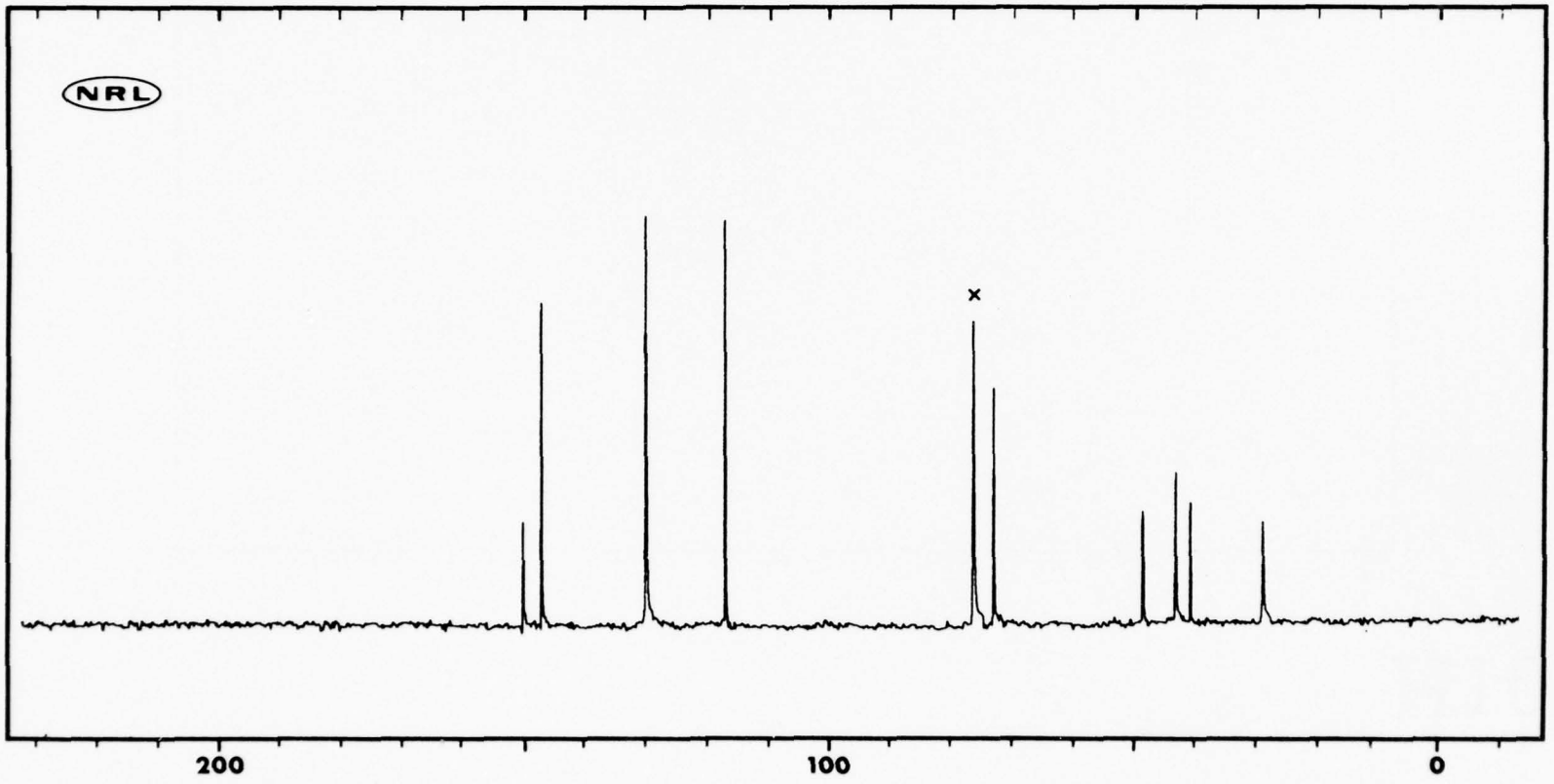


Source: Dow D.E.R. 542

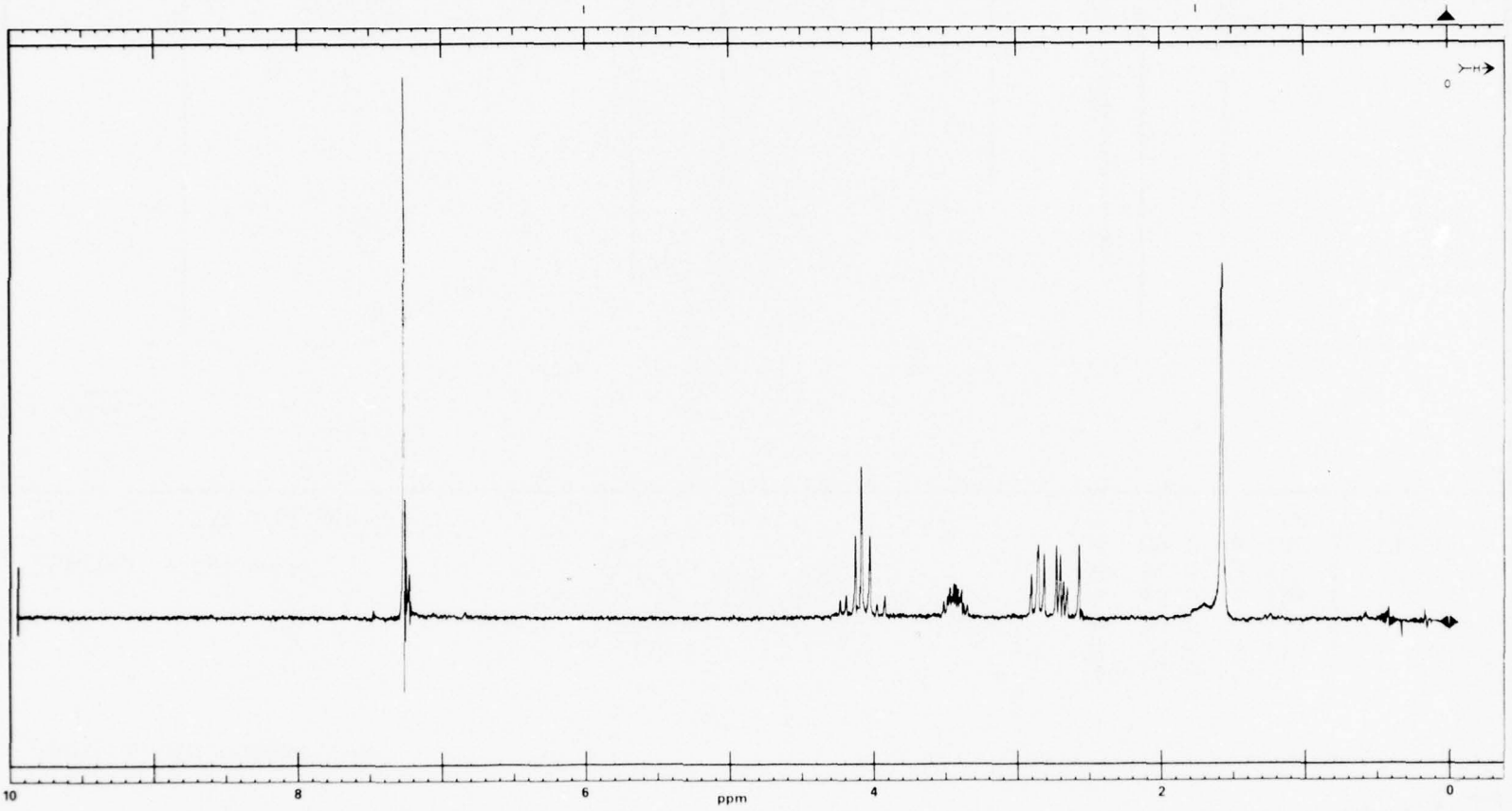
Solvent: 50% $CHCl_3$ ×

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		



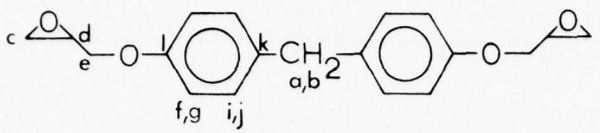
H11



Spectrum 11 — Diglycidyl ether of tetrabromo-*bis*-phenol A (D.E.R. 542); solvent: $CDCl_3$

C12

Epoxy Resin, DGEBF Type

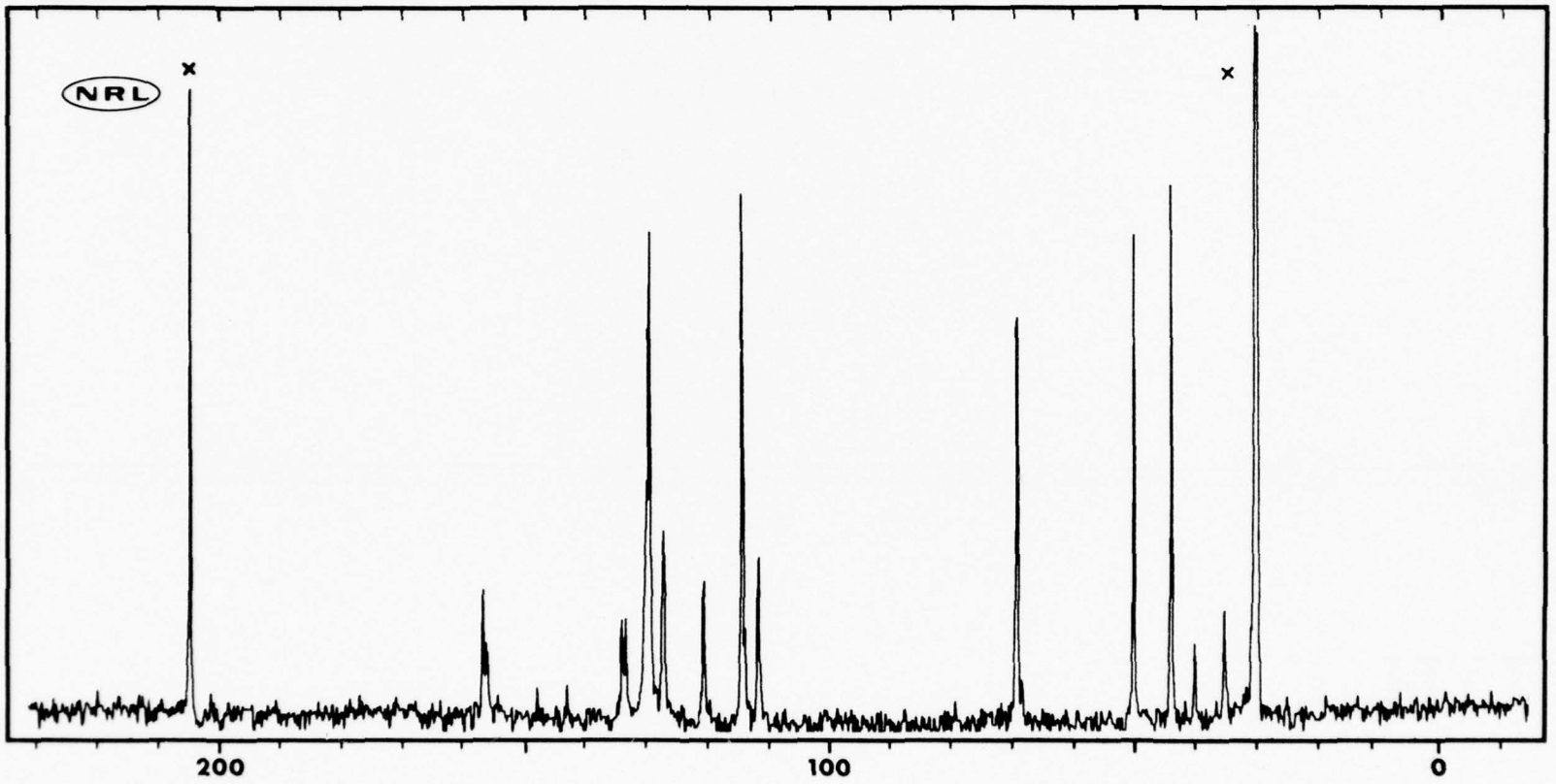


Source: CIBA 3794

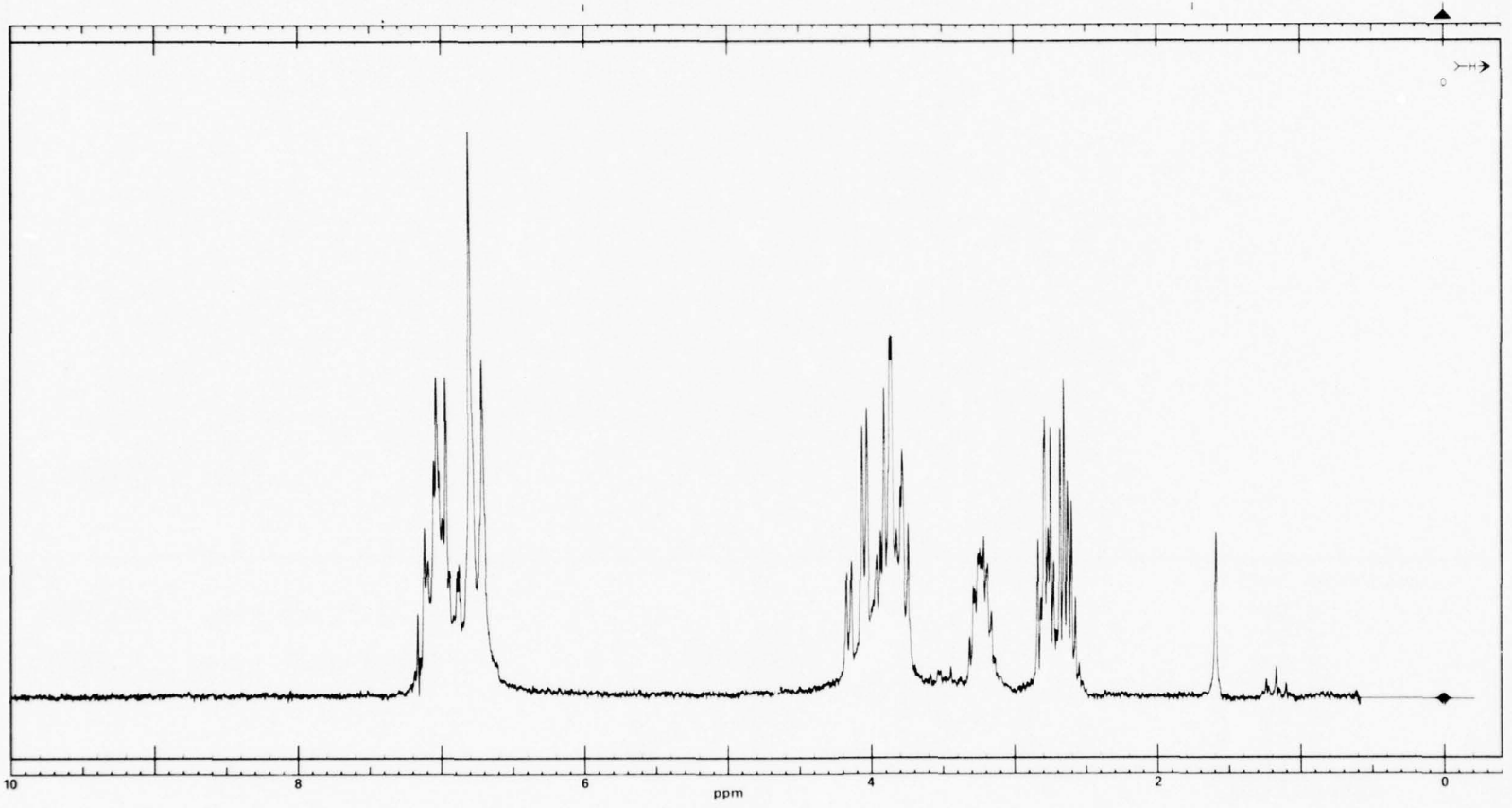
Solvent: 25% Acetone ×

Assignments:

a	34.8	g	114.4
b	39.6	h	121.5
c	43.9	i	128.1
d	50.0	j	129.4
e	69.3	k	133.3, 134.0
f	112.4	l	156.1, 156.6



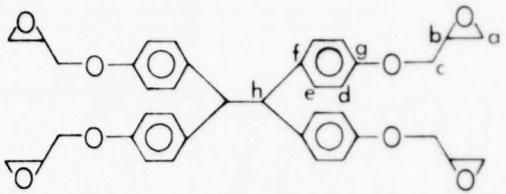
H12



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

C13

Polyglycidyl Ether of Tetraphenylene Ethane

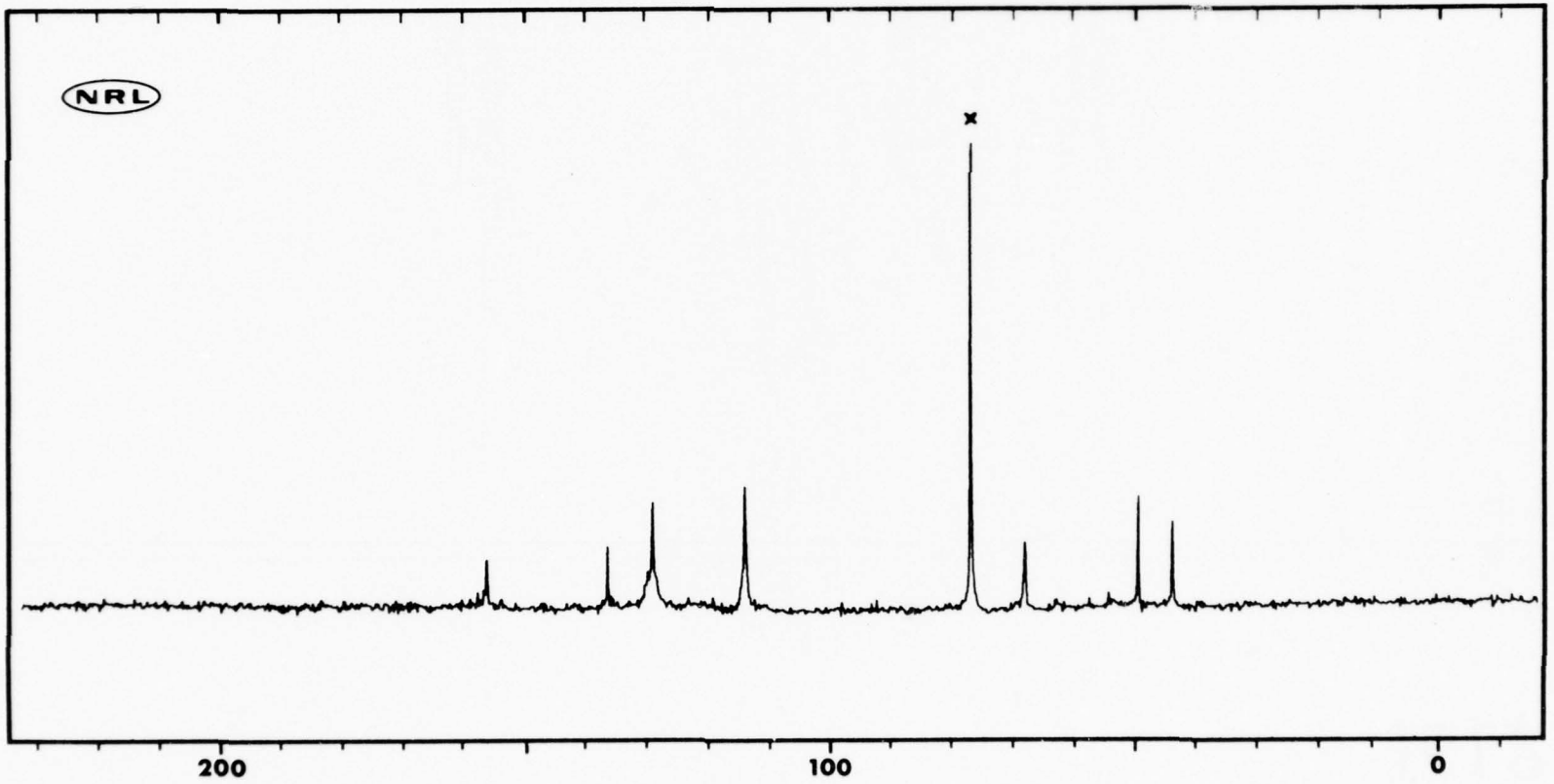


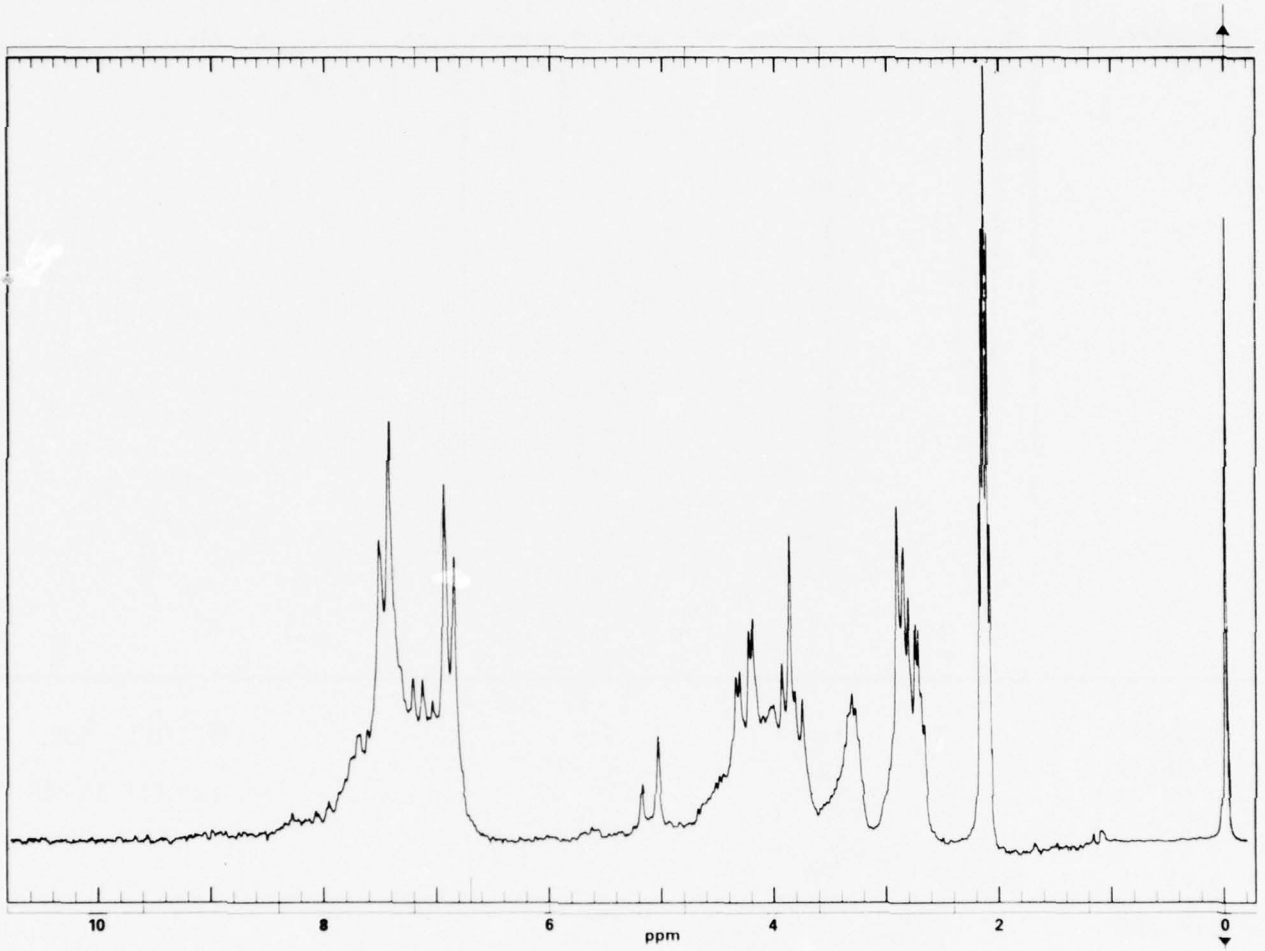
Assignments:

a	44.2
b	49.8
c	68.2
d	113.9
e	128.9
f	136.2
g	155.9
h	not observed

Source: Shell Epon 1031

Solvent: 75% $CHCl_3$ ×

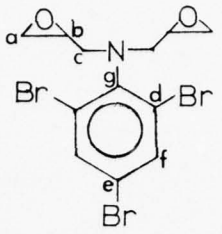




Spectrum 13 — Polyglycidyl ether of tetraphenylene ethane (Shell Epon 1031) solvent: acetone-d_6

C14

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

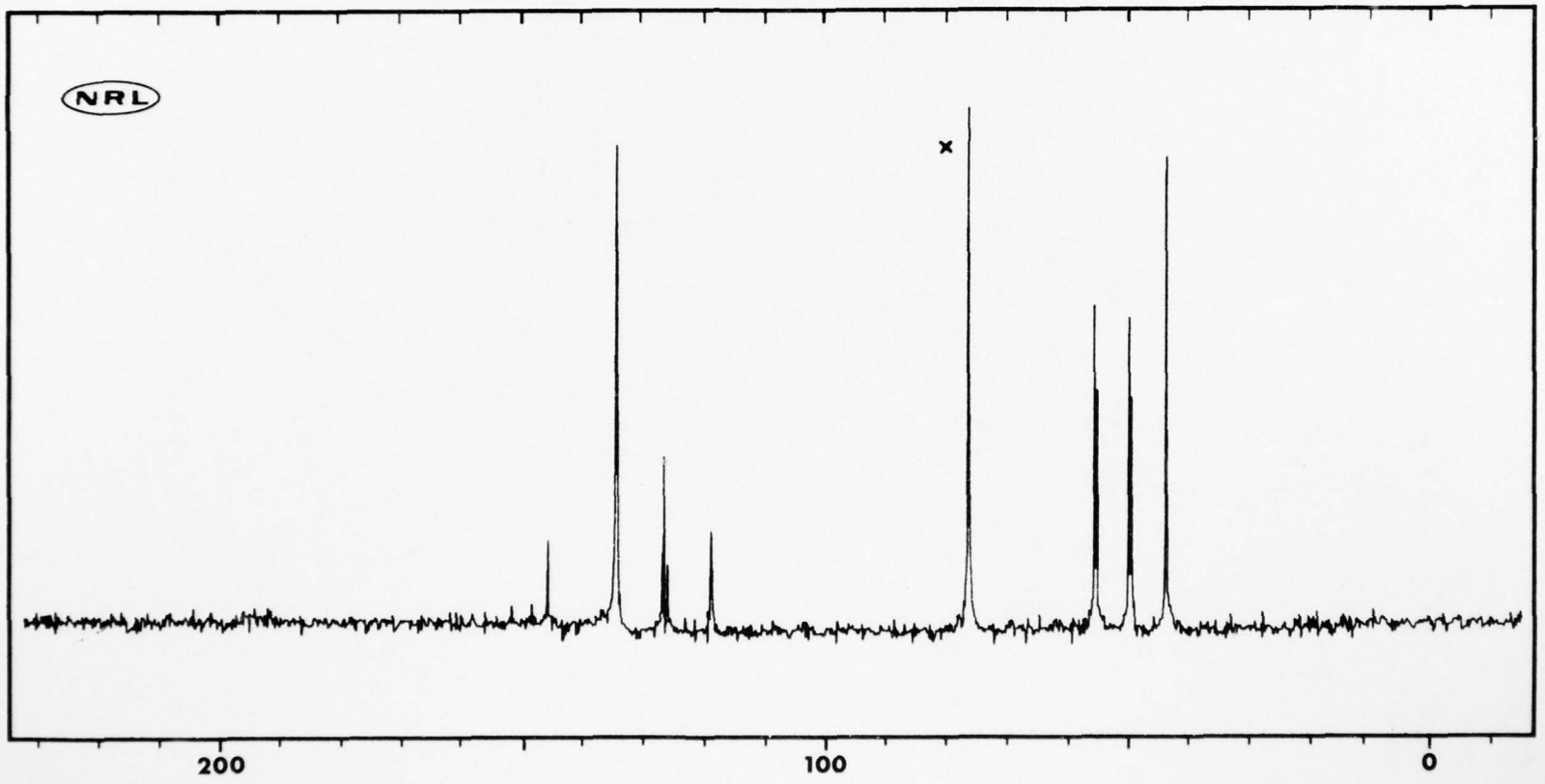


Assignments:

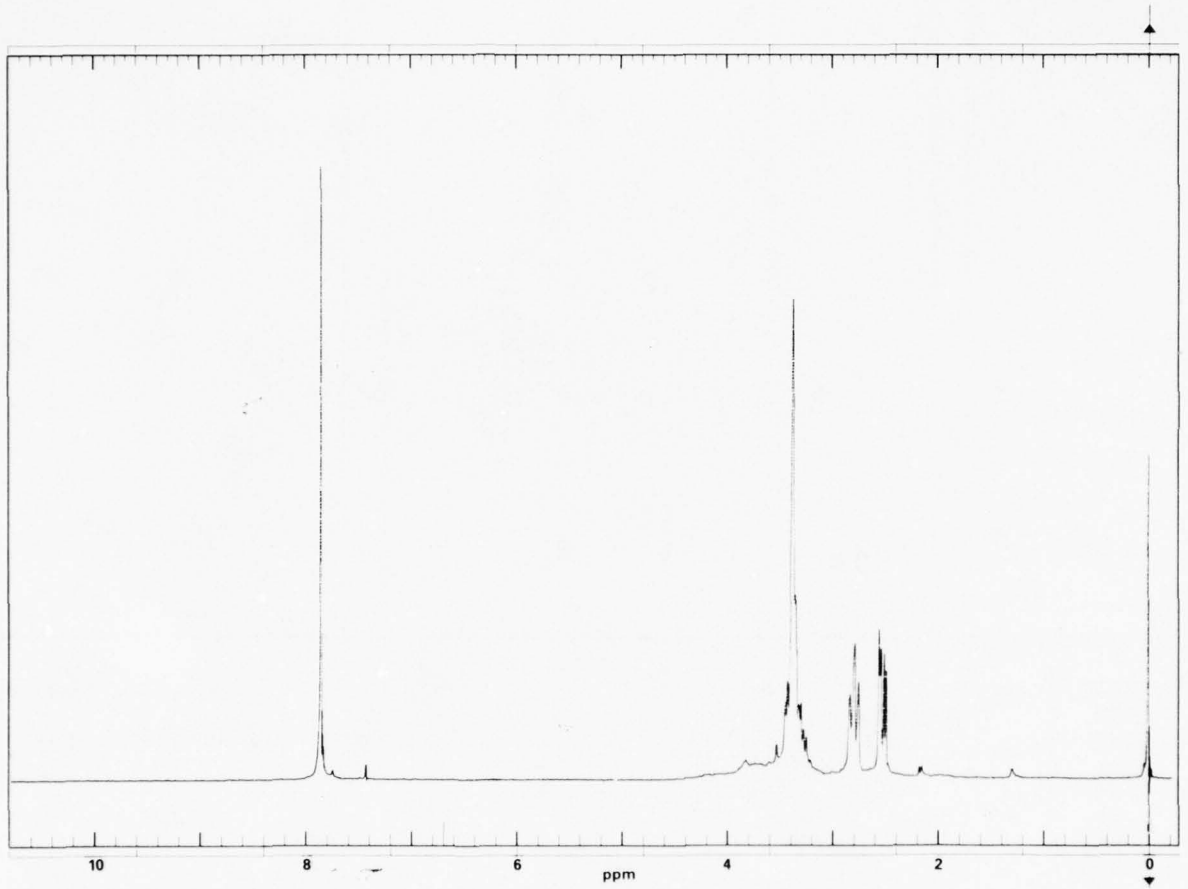
a	44.7	e	126.9
	50.4	f	134.6
b	50.8	g	145.8
	56.0		
c	56.5		
d	119.3		
	126.3		

Source: Shell ERX-67

Solvent: 50% $CHCl_3$ ✖



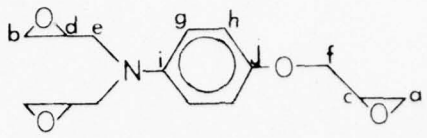
H14



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

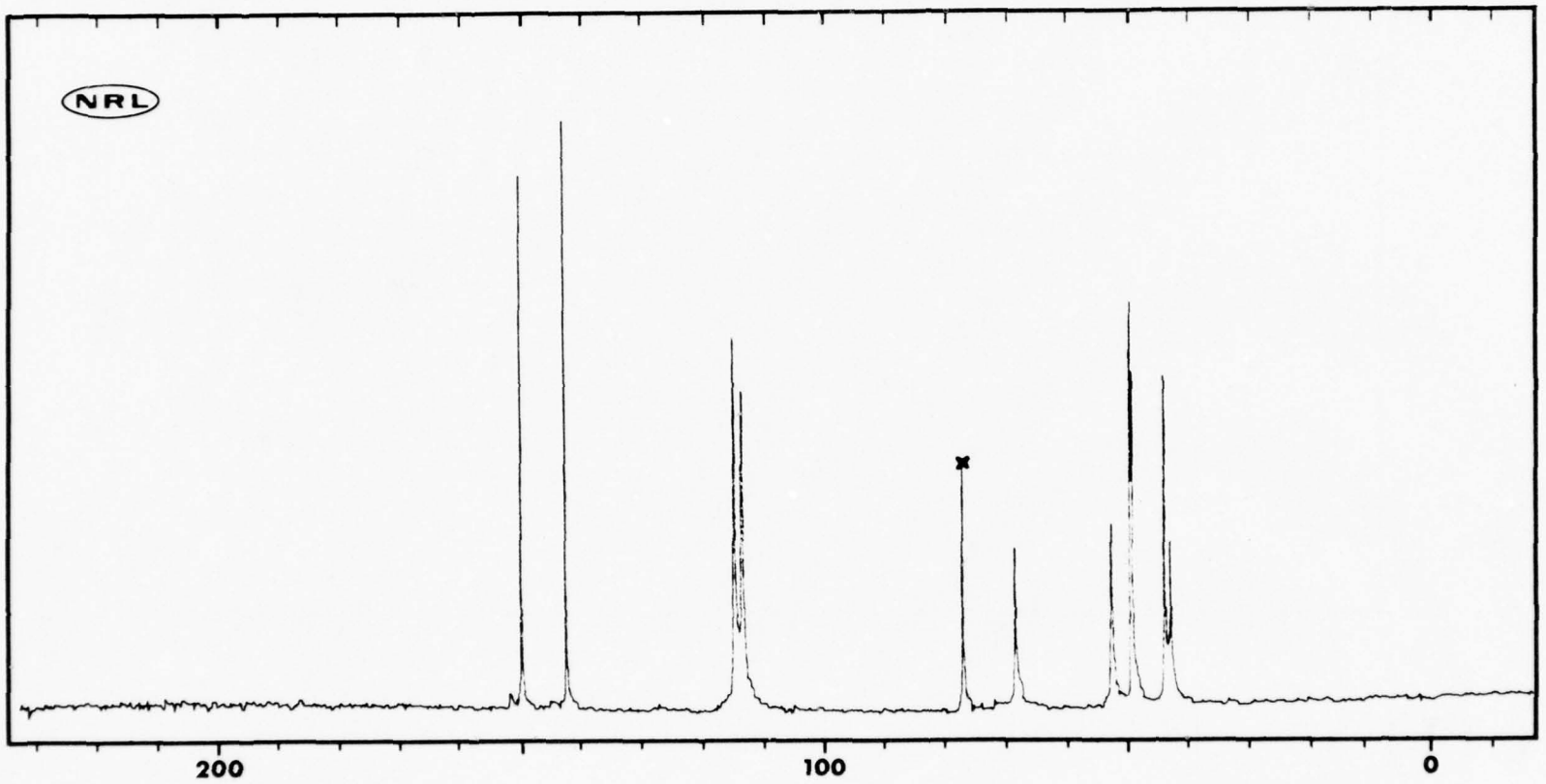


Source: Union Carbide ERL 0510

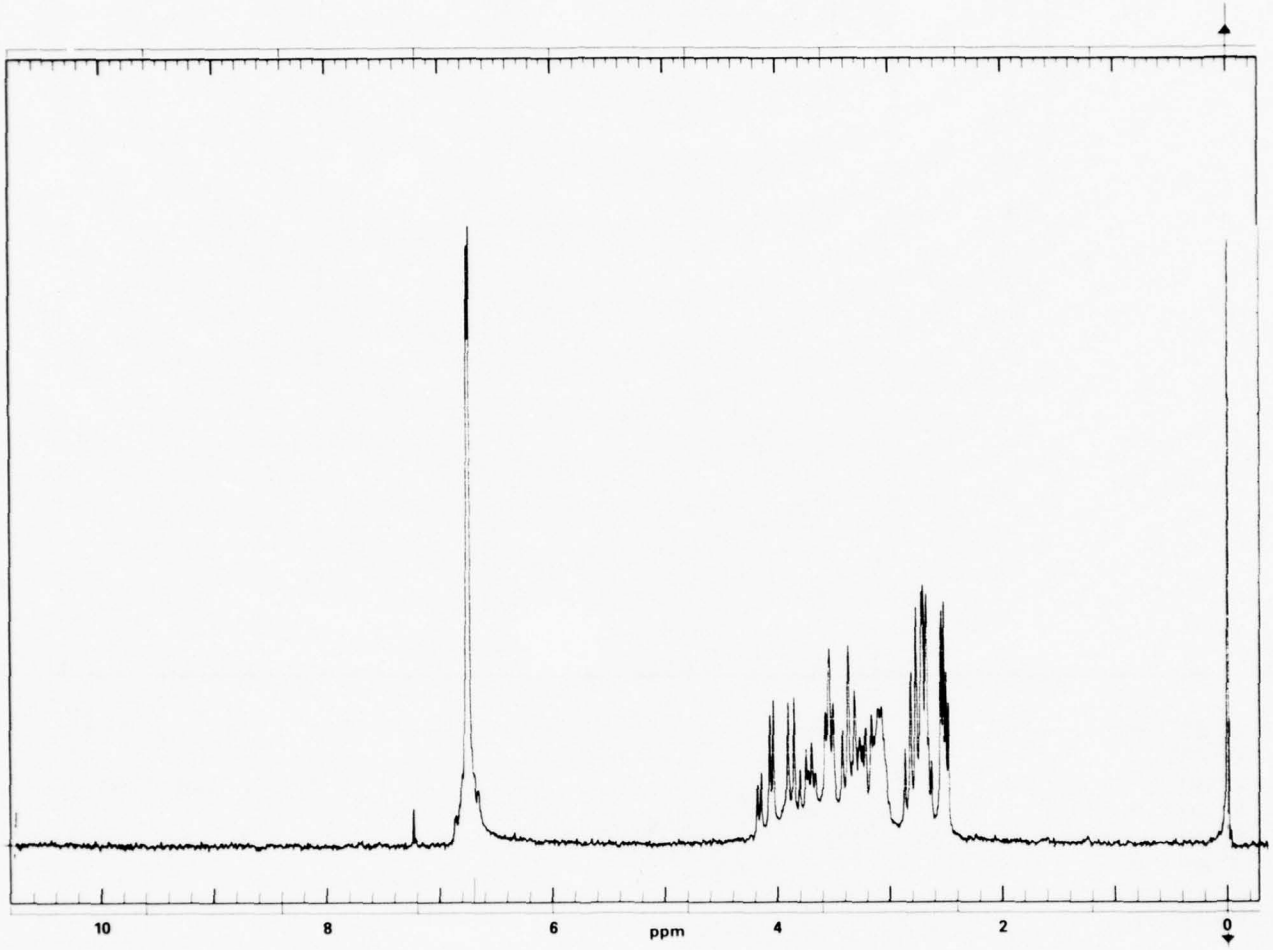
Solvent: 20% $CHCl_3$ ×

Assignments:

a	43.0	h	114.6
b	44.0	i	142.3
c	49.1	j	149.6
d	49.4		
e	52.7		
f	68.5		
g	113.2		



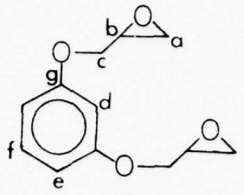
H15



Spectrum 15 — 4-(2,3-epoxy)propoxy-*N,N-bis*(2,3-epoxypropyl)-aniline (Union Carbide ERL 0510); solvent: $CDC1_3$

C16

Resorcinol Diglycidyl Ether

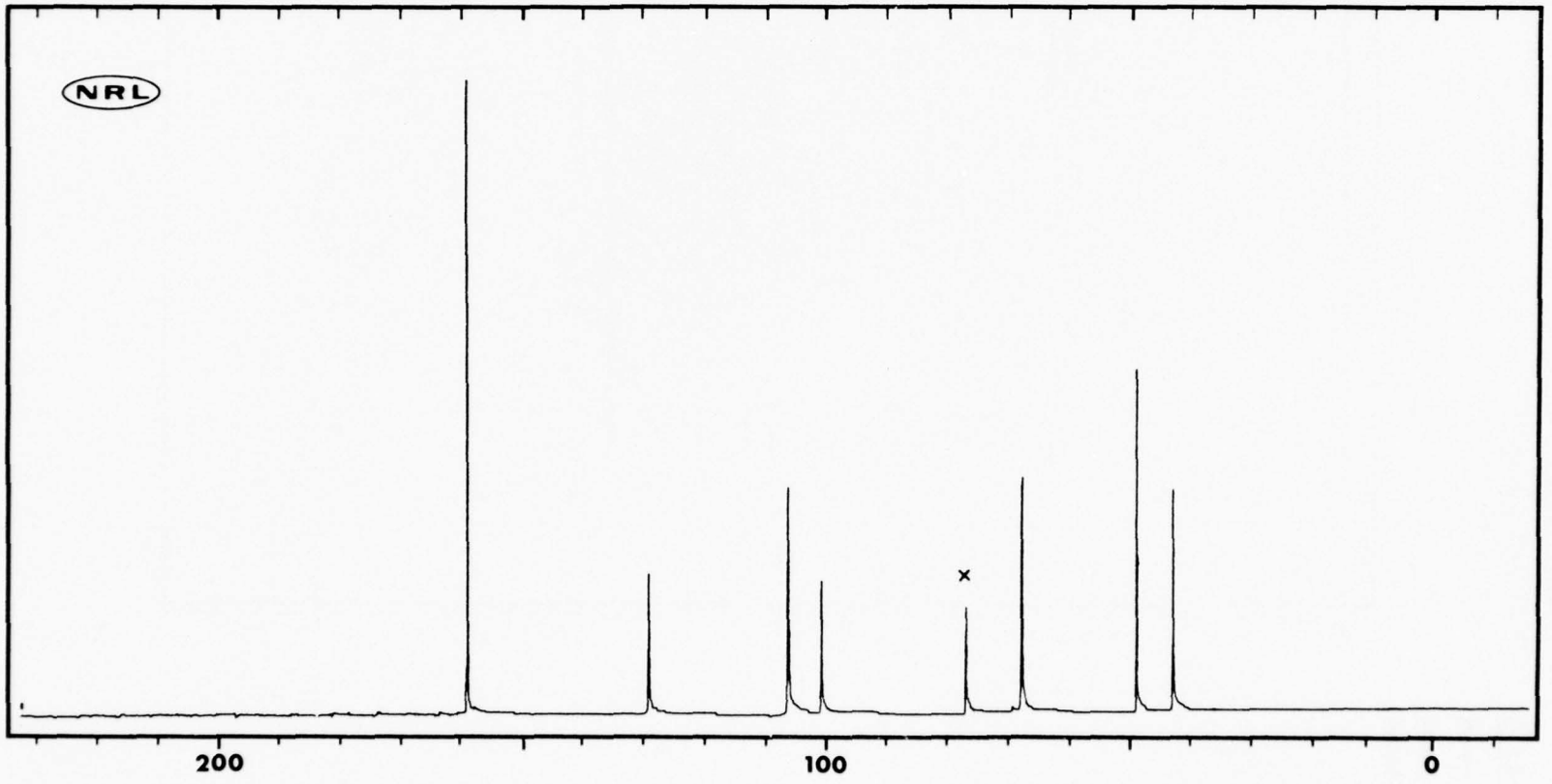


Assignments:

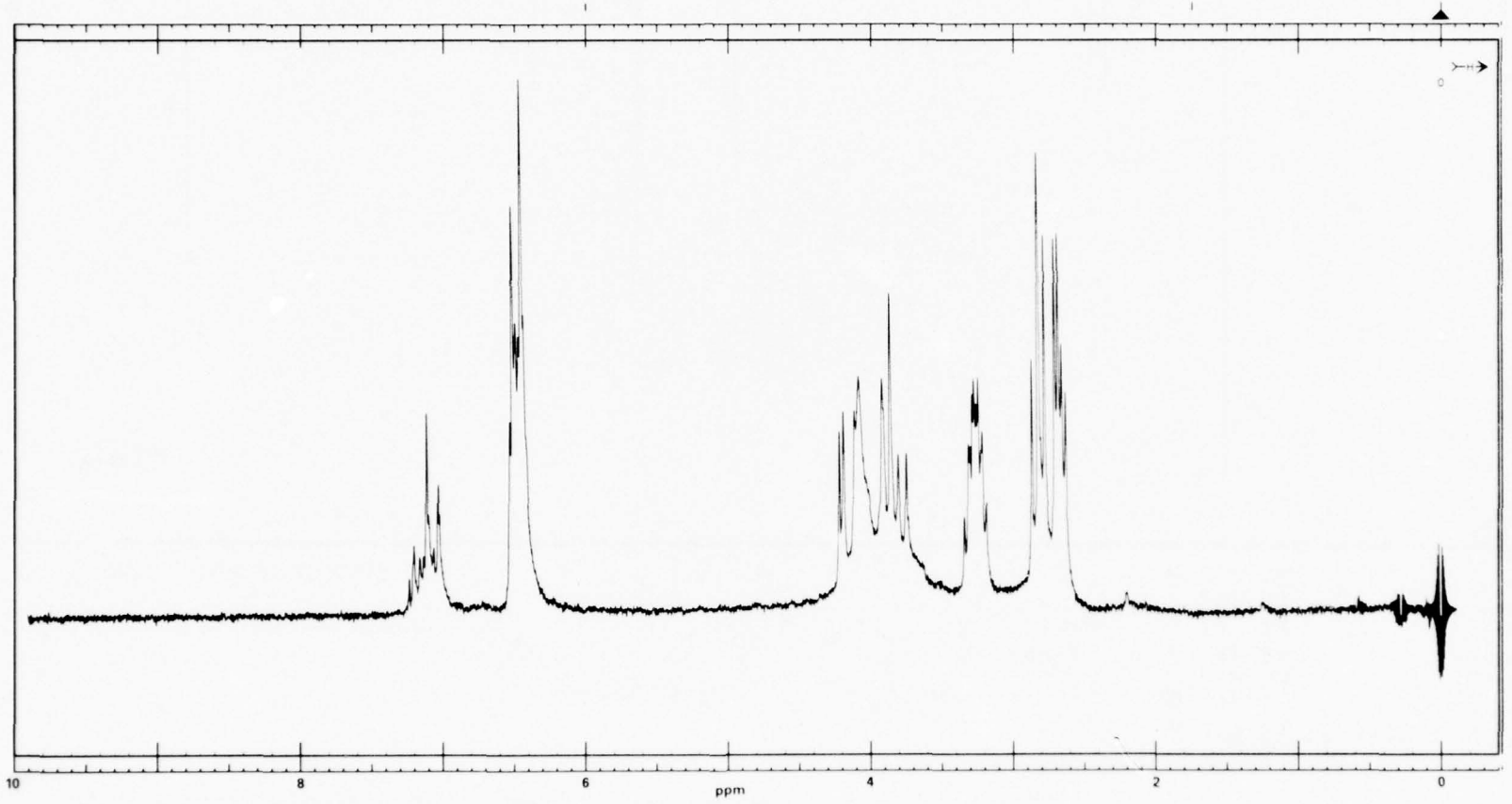
a	43.2
b	49.1
c	68.0
d	100.8
e	106.2
f	129.0
g	158.6

Source: Koppers Kopox 159

Solvent: 20% $CHCl_3$ ×



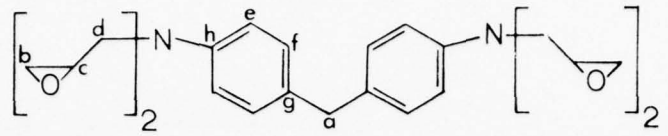
H16



Spectrum 16 — Resorcinol diglycidyl ether (Koppers Kopox 159); solvent: $CDCl_3$

C17

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

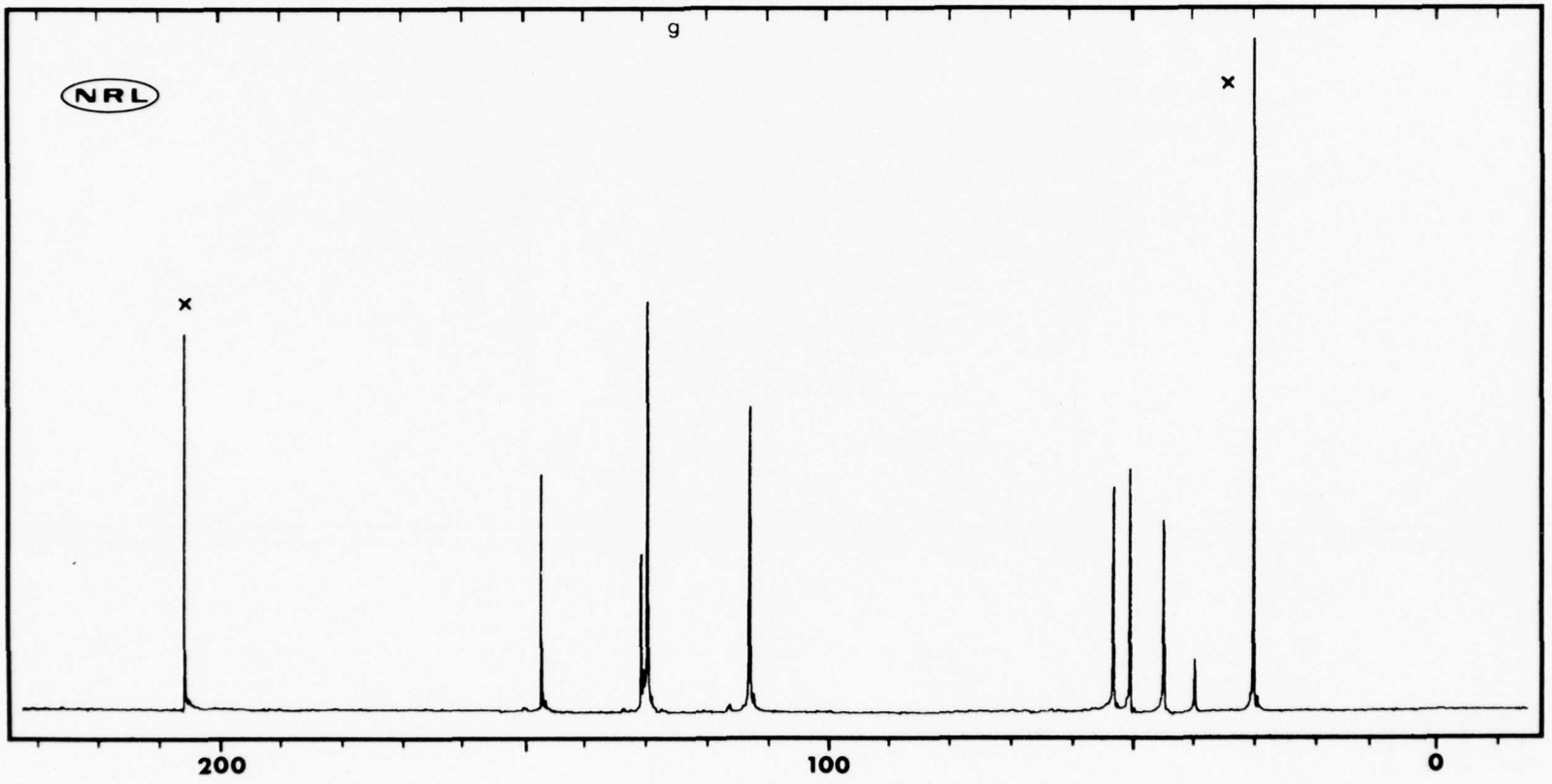


Source: Ciba-Geigy MY720

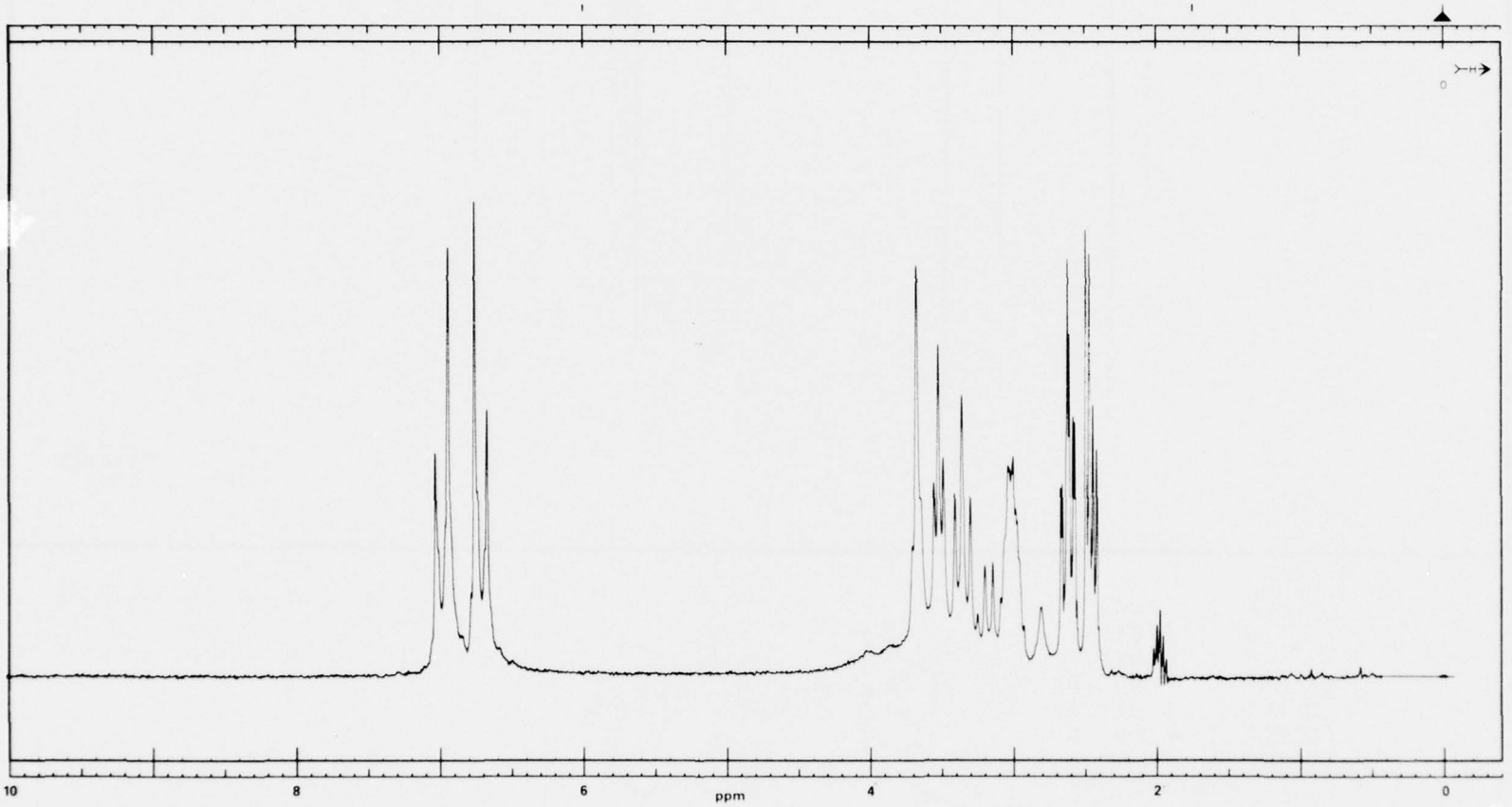
Solvent: 10% Acetone ×

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		



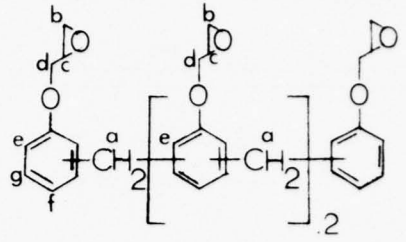
H17



Spectrum 17 — *bis*(*N*,*N*-di(2,3-epoxypropyl)-4-aminophenyl)*methane* (Ciba-Geigy MY720); solvent: acetone-d_6

C18

Epoxy Novolac Resin

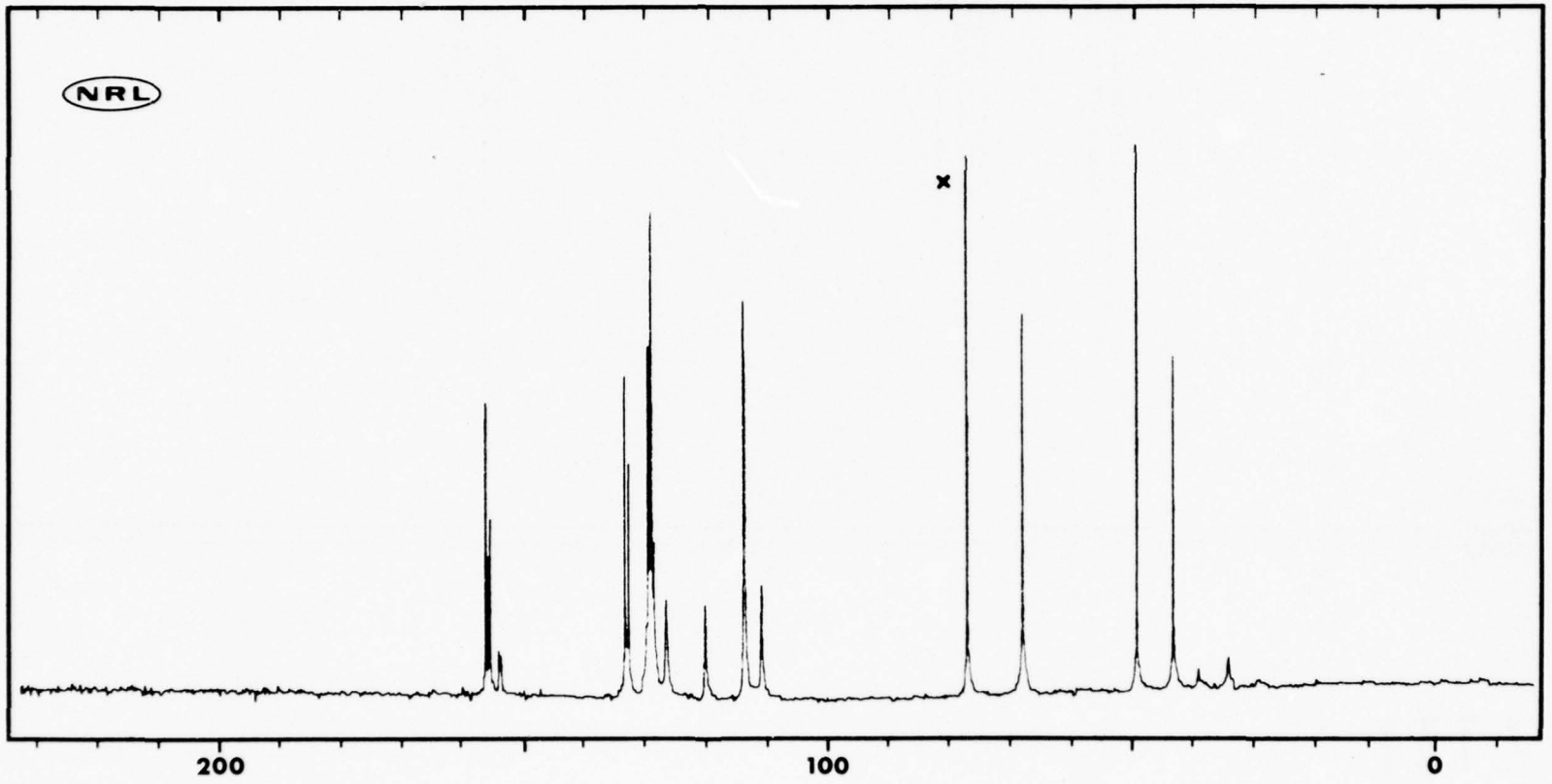


Source: Dow D.E.N. 431

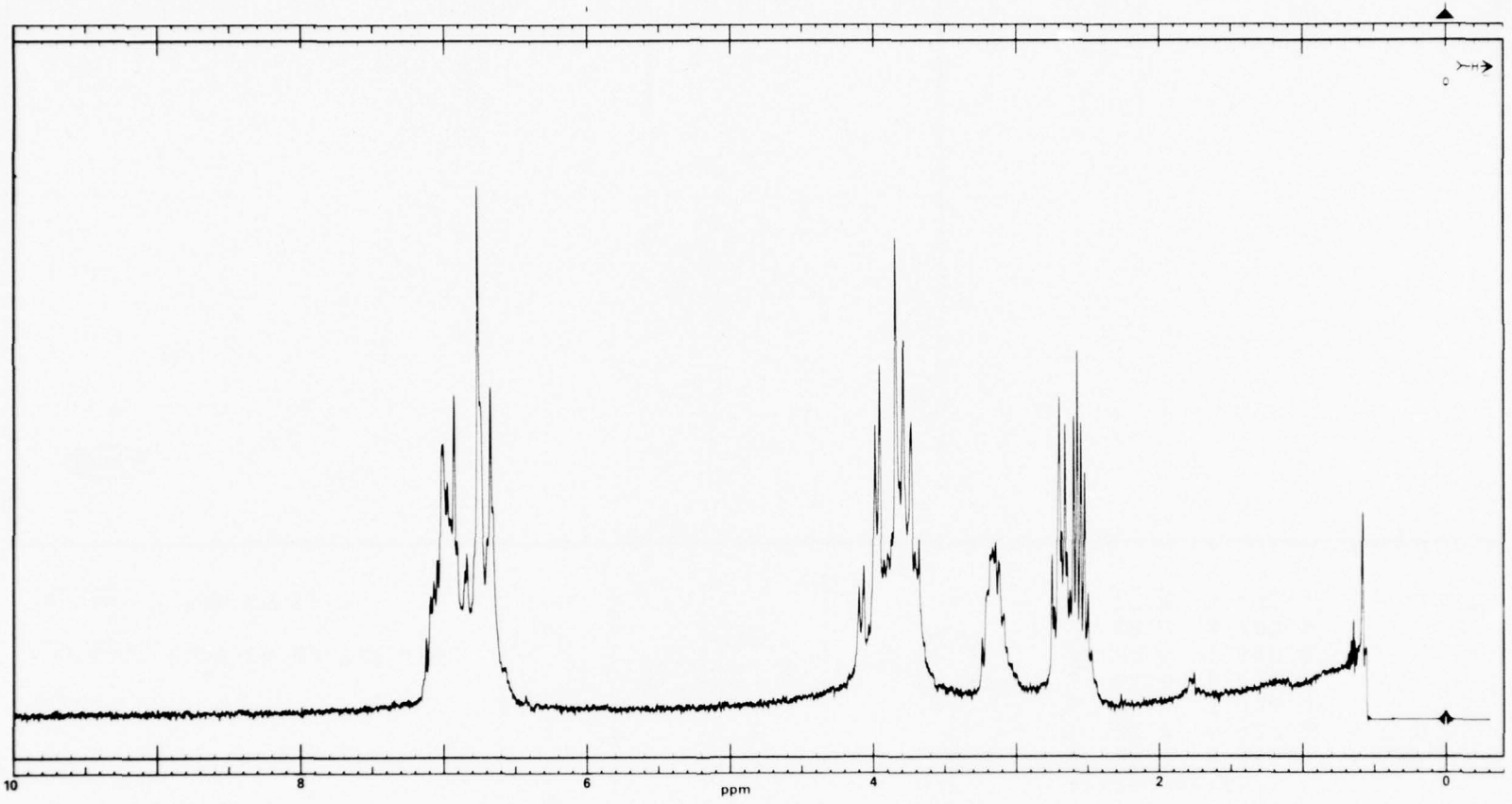
Solvent: 20% $CHCl_3$ ✖

Assignments:

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



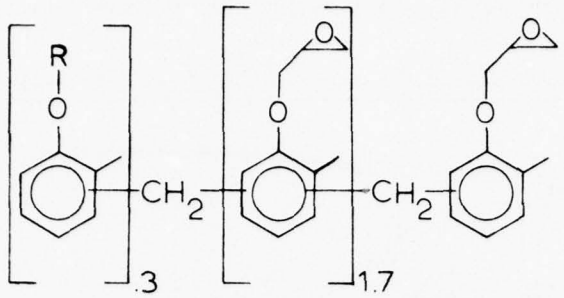
H18



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

C19

Epoxy Novolac Resin

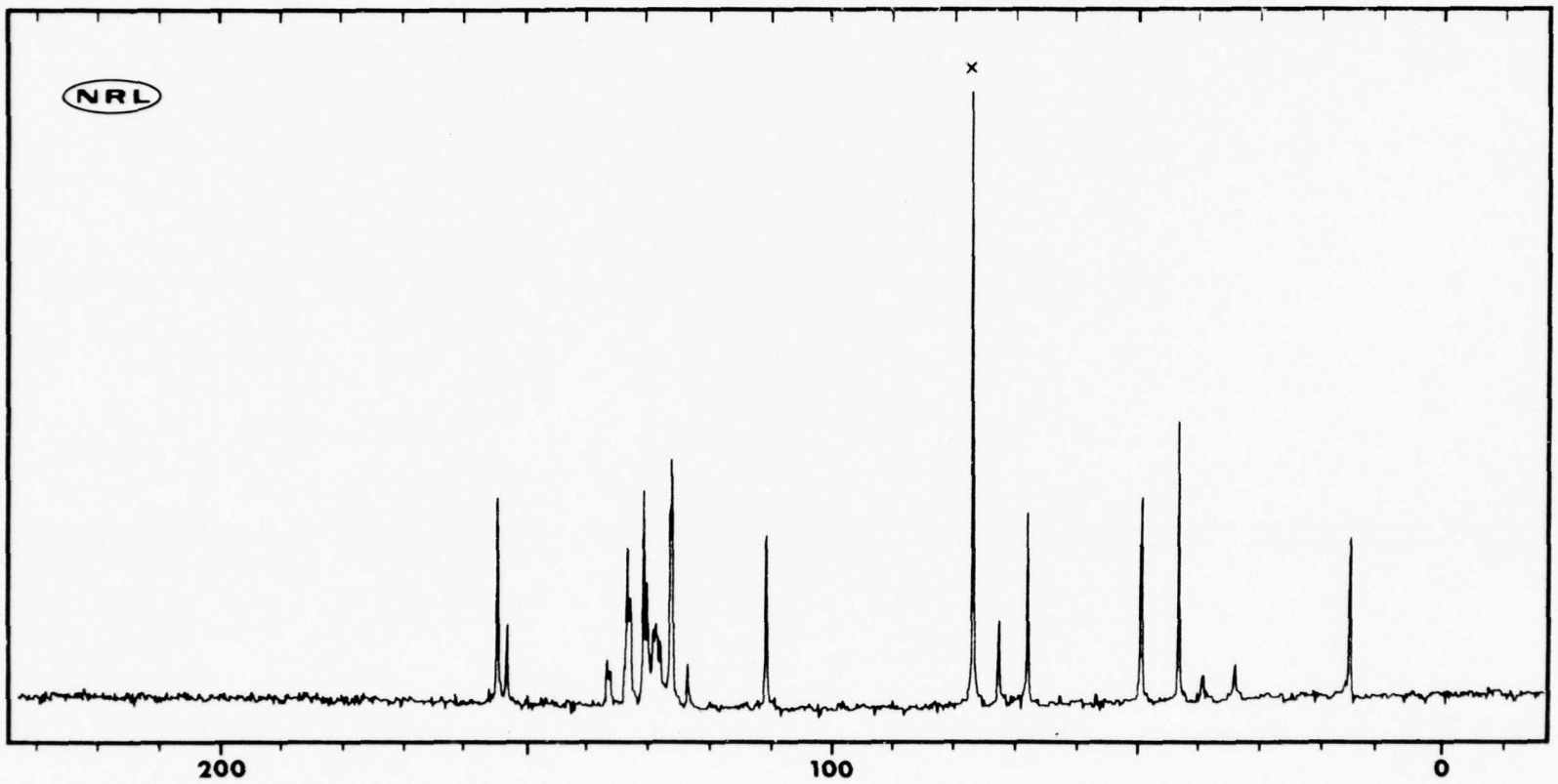


Source: Ciba-Geigy ECN 1235

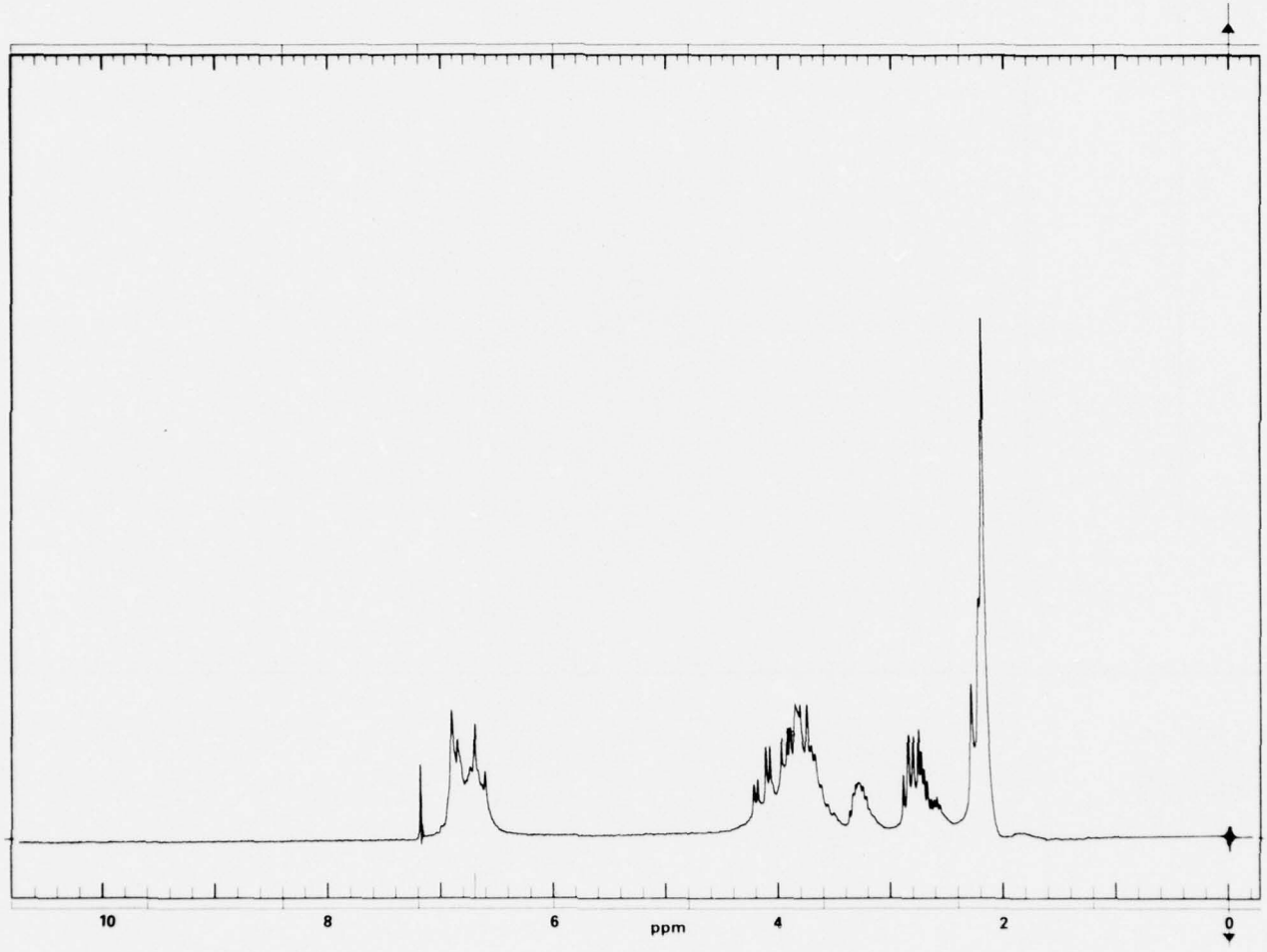
Solvent: 50% $CHCl_3$ ×

Assignments:

a	15.6	h	110.6	o	152.6
b	34.2	i	123.3	p	154.2
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		



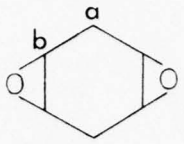
H19



Spectrum 19 — Epoxy Novolac resin (Ciba-Geigy ECN 1235); solvent: $CDC1_3$

C20

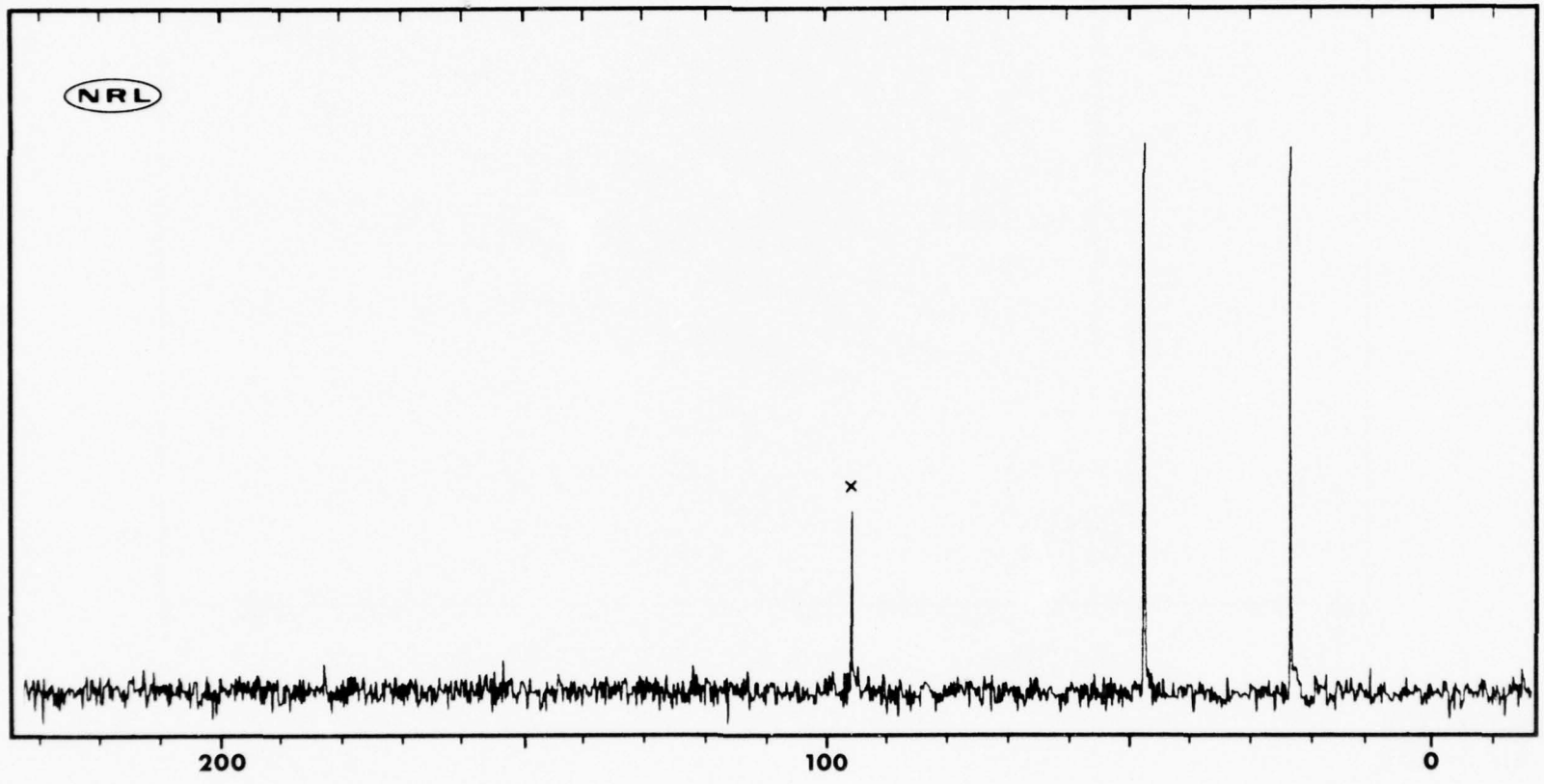
4,8-Dioxatricyclo[5.1.0.$0^{3,5}$]octane



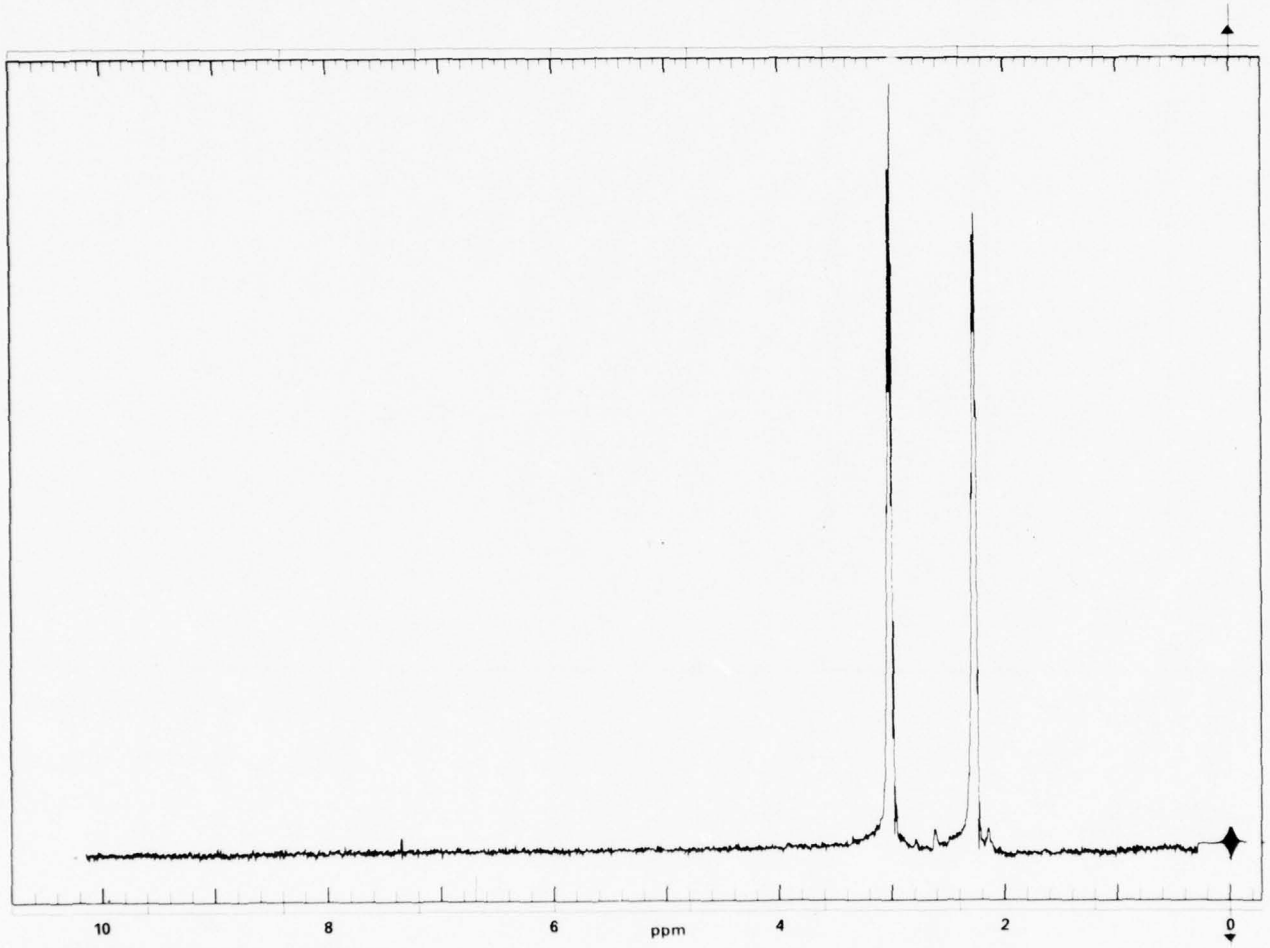
Source: Union Carbide ERL 2114

Solvent: 10% CCl_4 ×

Assignments:
a 24.4t
b 48.4d



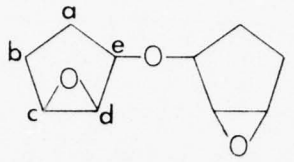
H20



Spectrum 20 —4,8-Dioxatricyclo [5.1.0.0^{3,5}] octane (Union Carbide ERL 2114); solvent: $CDCl_3$

C21

Bis(2,3-epoxycyclopentyl)ether

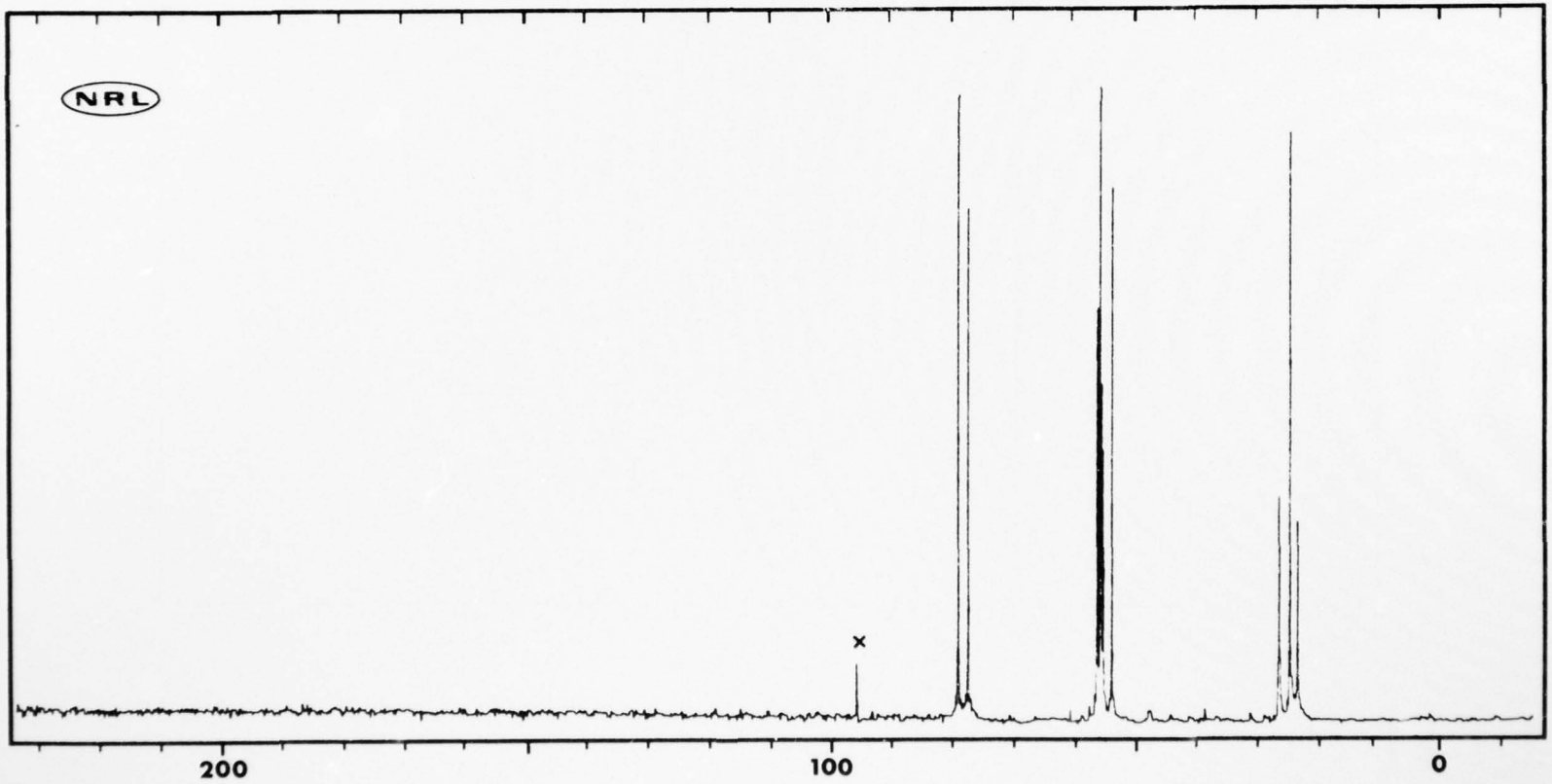


Source: Union Carbide ERL 0400

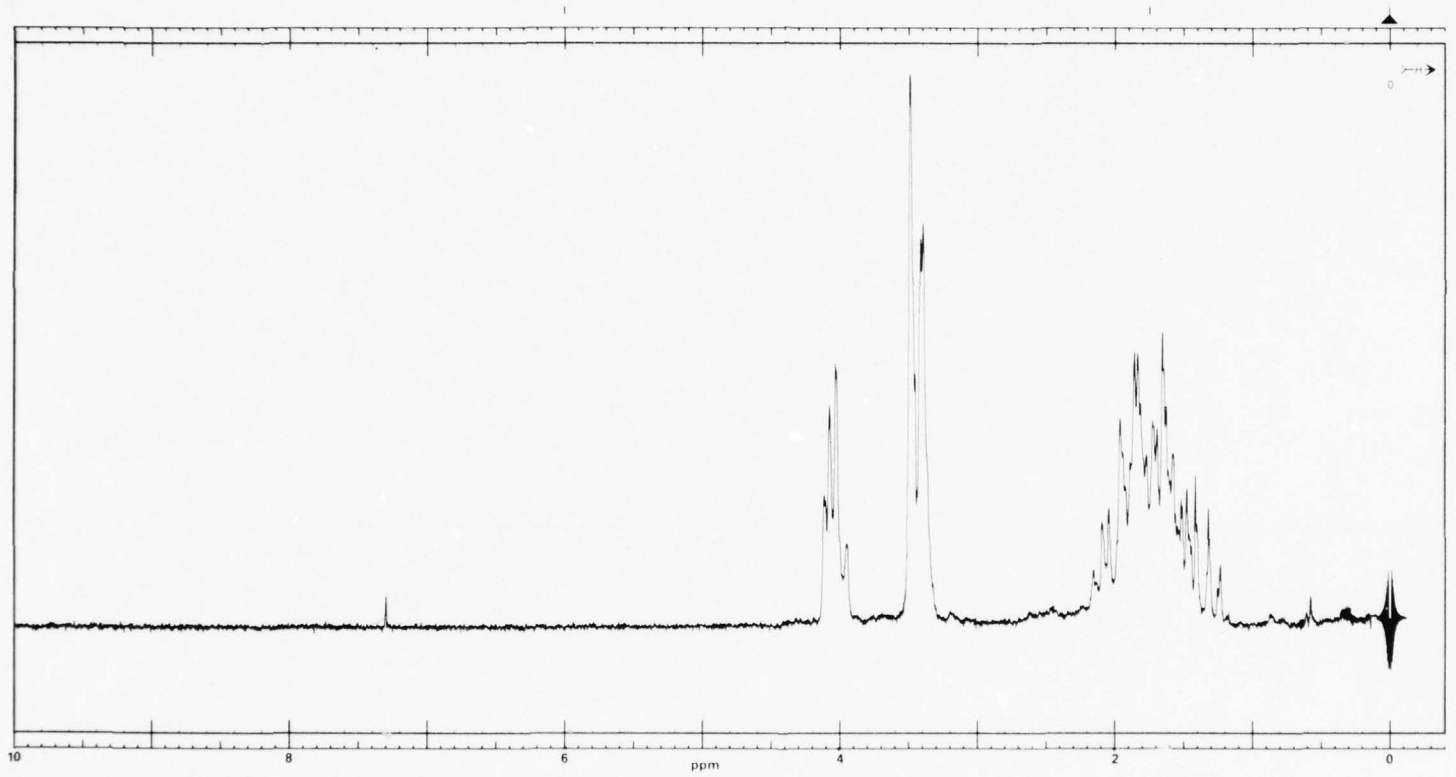
Solvent: 20% CCl_4 ×

Assignments:

a,b	24.1	c,d	56.0
a,b	25.3	c,d	56.3
a,b	27.2	c,d	56.7
c,d	54.3	e	77.9
c,d	55.8	e	79.5



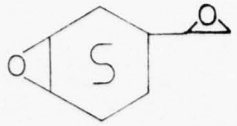
H21



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

C22

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

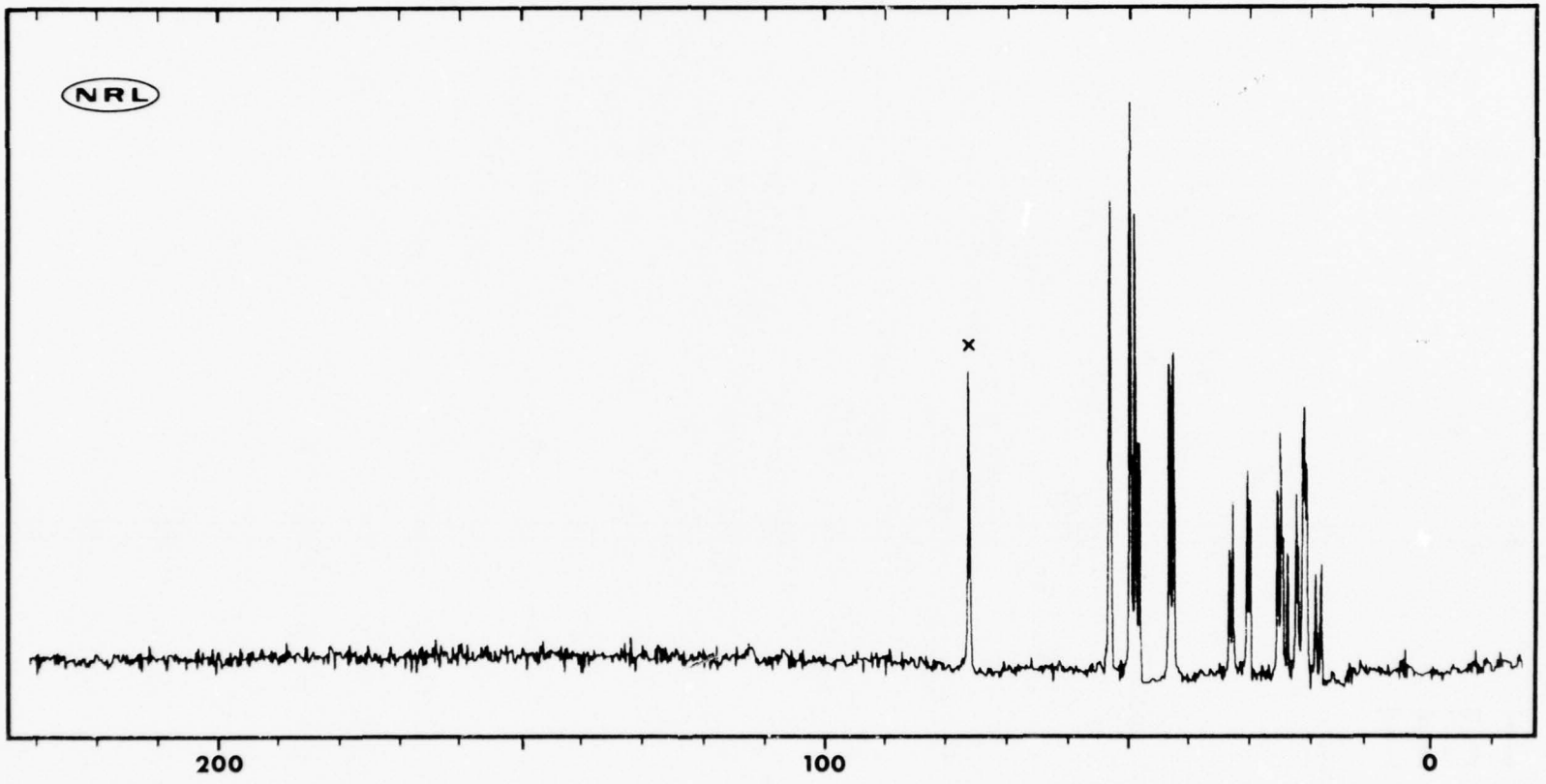


Source: Union Carbide Epoxide 206

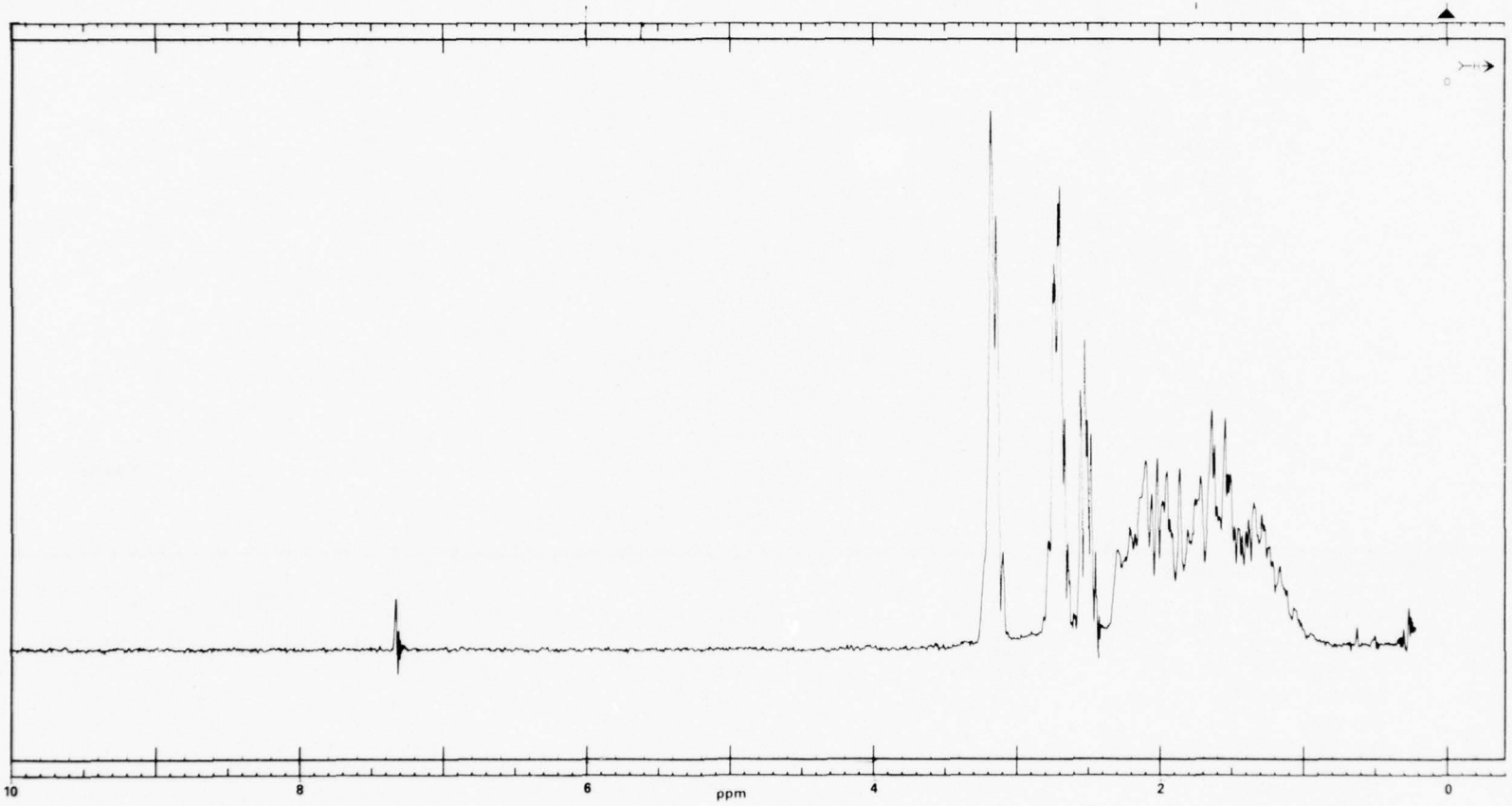
Solvent: 25% $CHCl_3$ ×

Assignments:

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



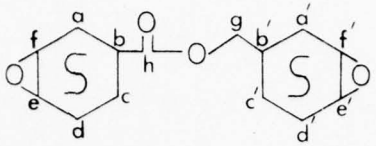
H22



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

C23

3,4-Epoxycyclohexylmethyl-(3,4-epoxy)cyclohexane Carboxylate

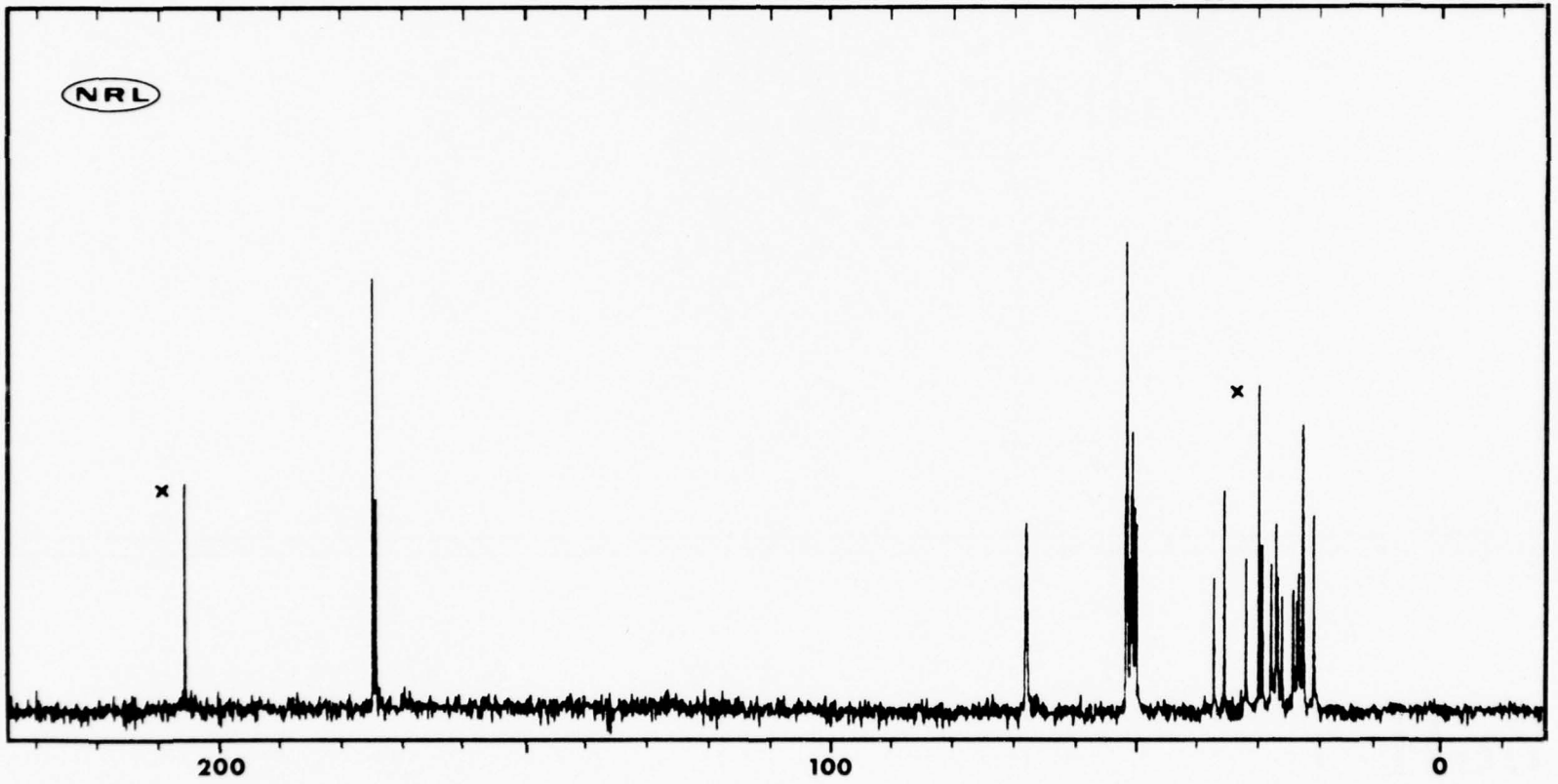


Source: Union Carbide ERLA 4221

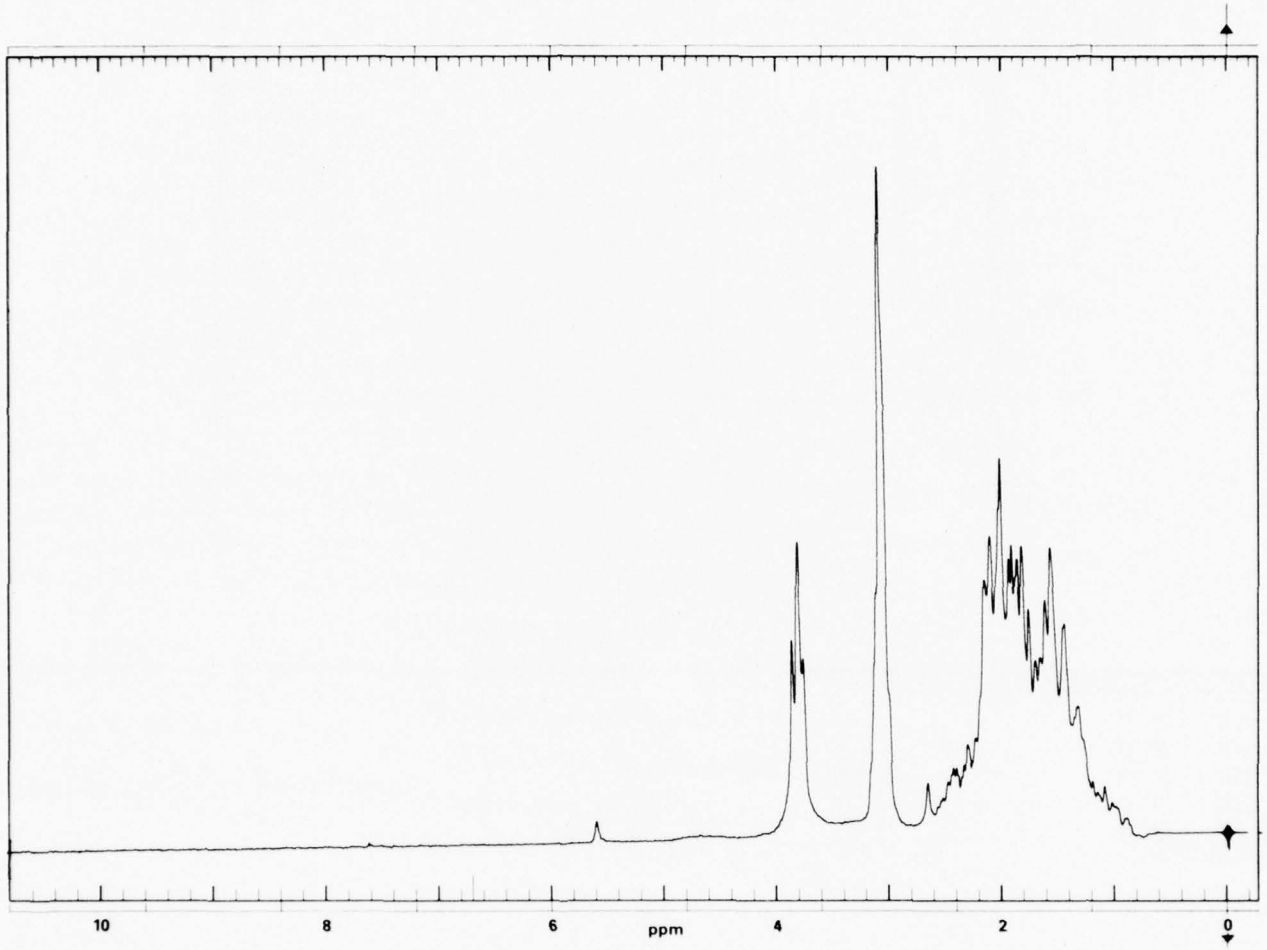
Solvent: 20% Acetone ✖

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

See Appendix for complete line listing



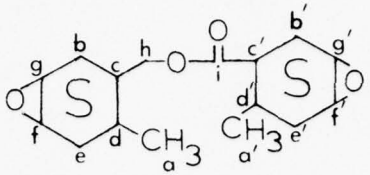
H23



Spectrum 23 — 3,4-Epoxycyclohexylmethyl-(3,4-epoxy)cyclohexane carboxylate (Union Carbide ERLA 4221); solvent: $CDCl_3$

C24

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



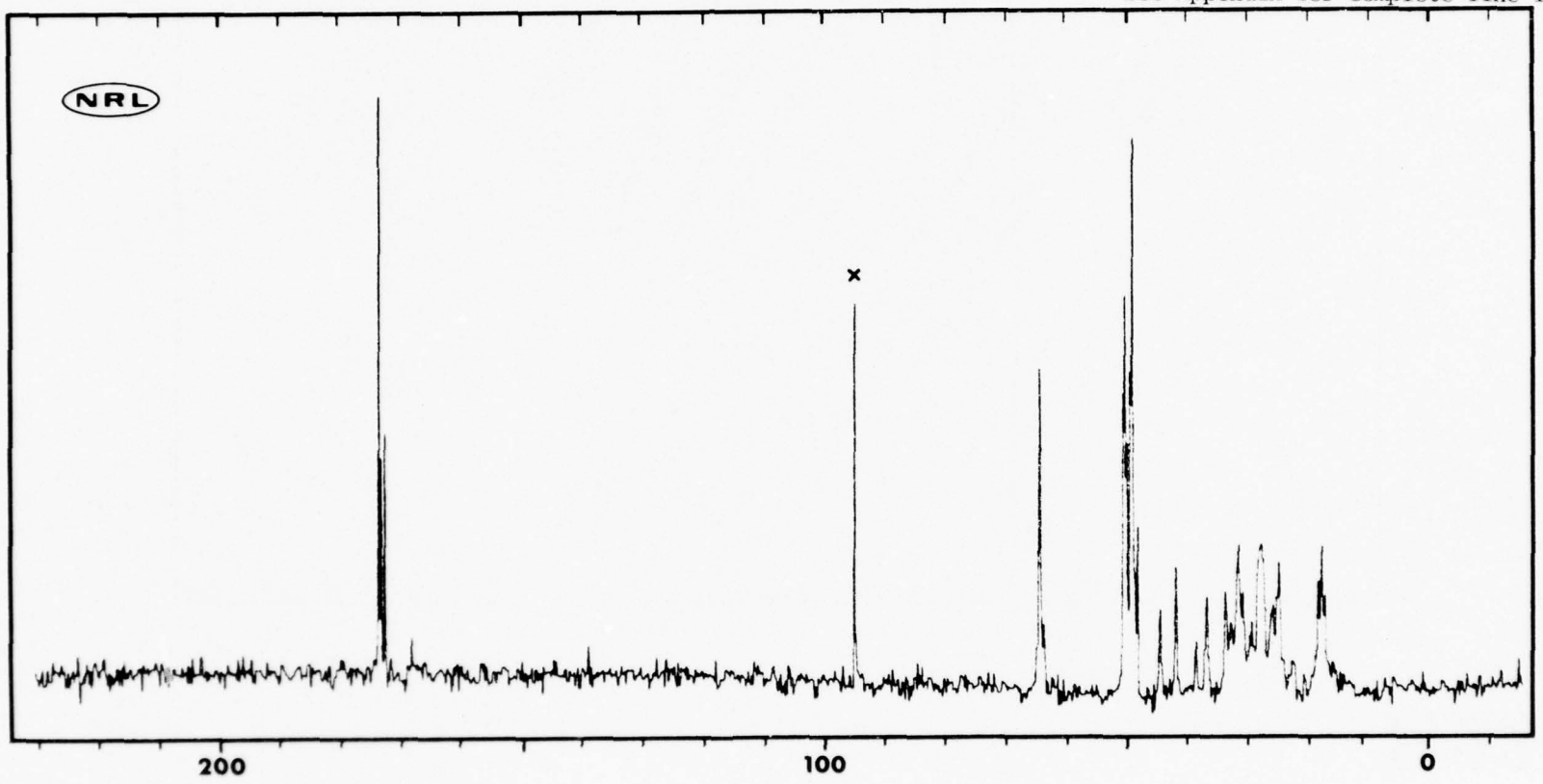
Source: Union Carbide Epoxide 201

Solvent: 25% CCl_4 ×

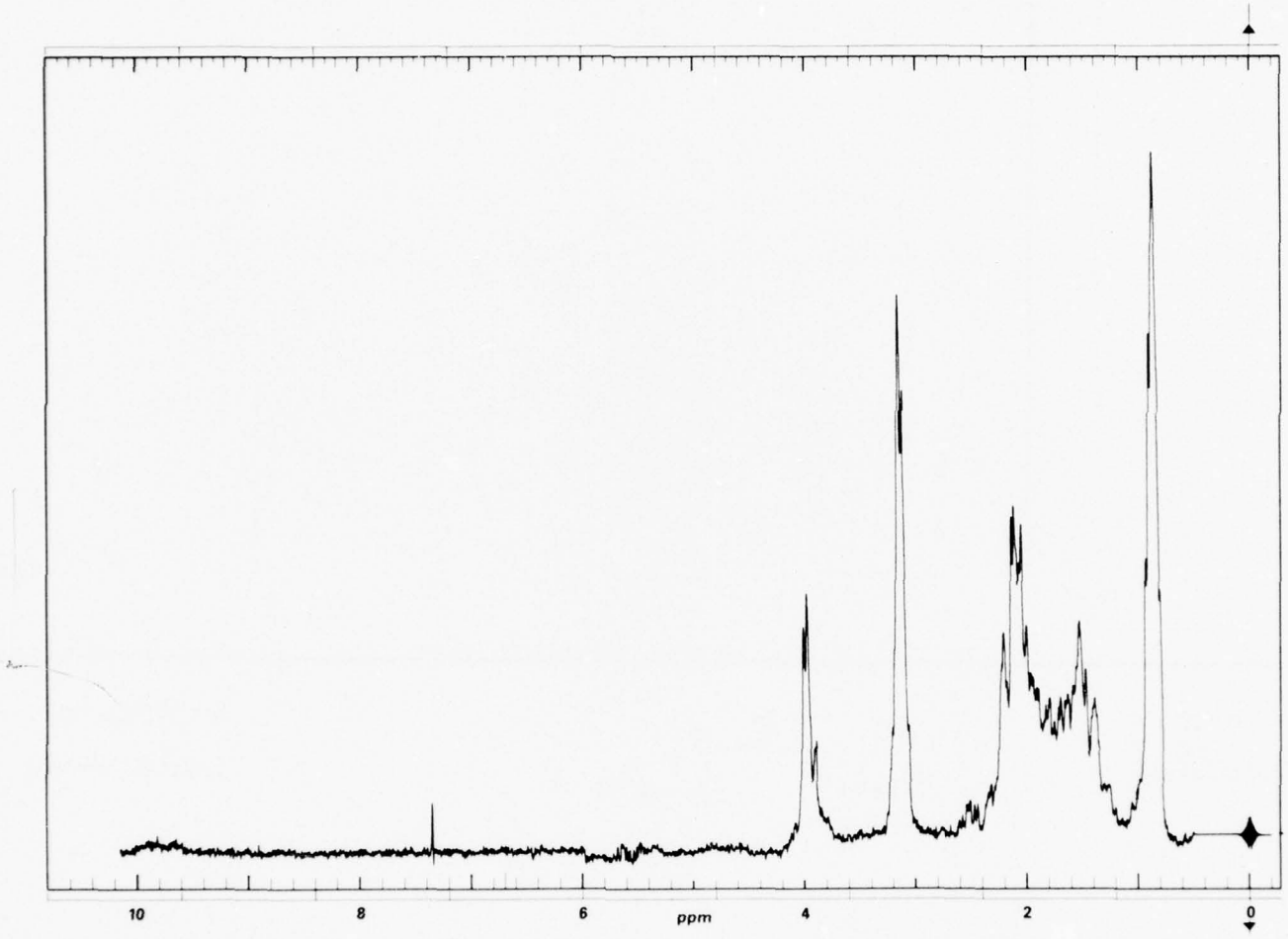
Assignments:

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

See Appendix for complete line listing



H24

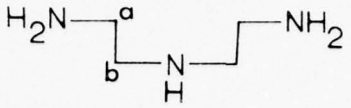


Spectrum 24 — 3,4-Epoxy-6-methylcyclohexylmethyl-3,4-epoxy-6-methylcyclohexane carboxylate (Union Carbide Epoxide 201); solvent: $CDCl_3$

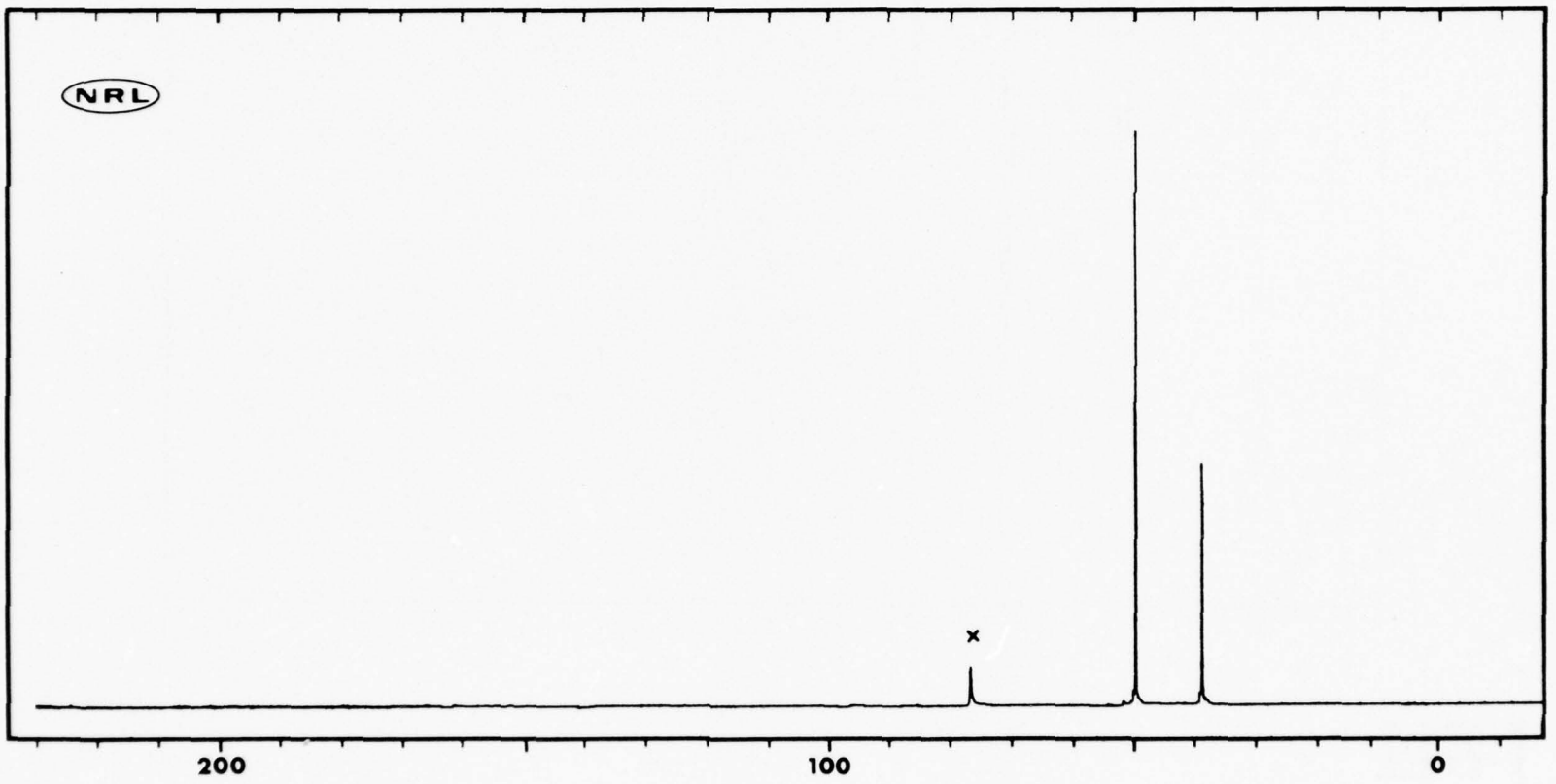
Diethylenetriamine

Source: Fisher D-126

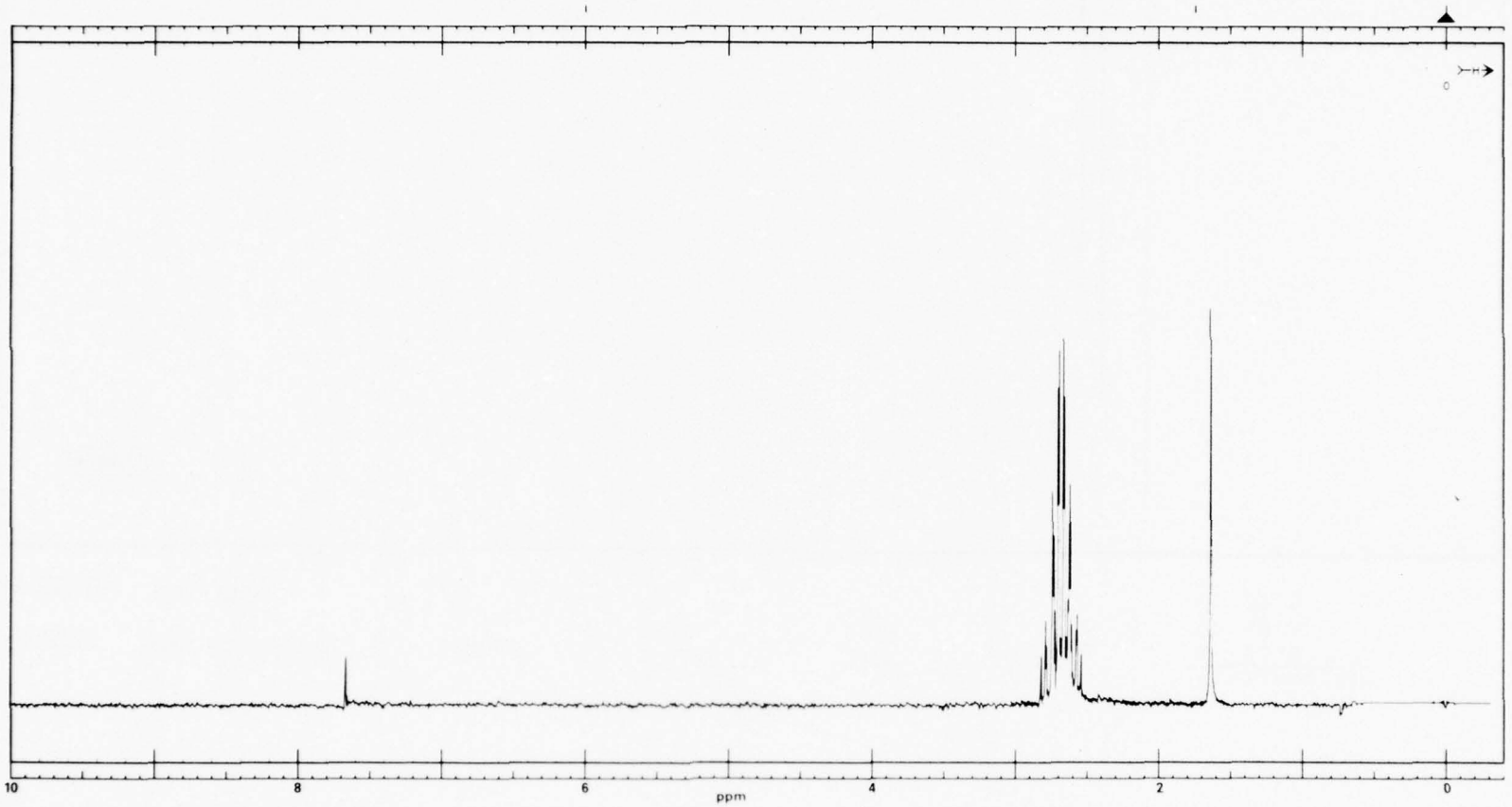
Solvent: 10% $CHCl_3$ ×



Assignments:
a 39.2
b 50.0



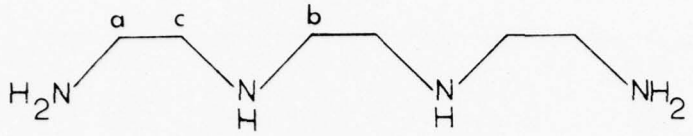
H25



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

C26

Triethylenetetramine

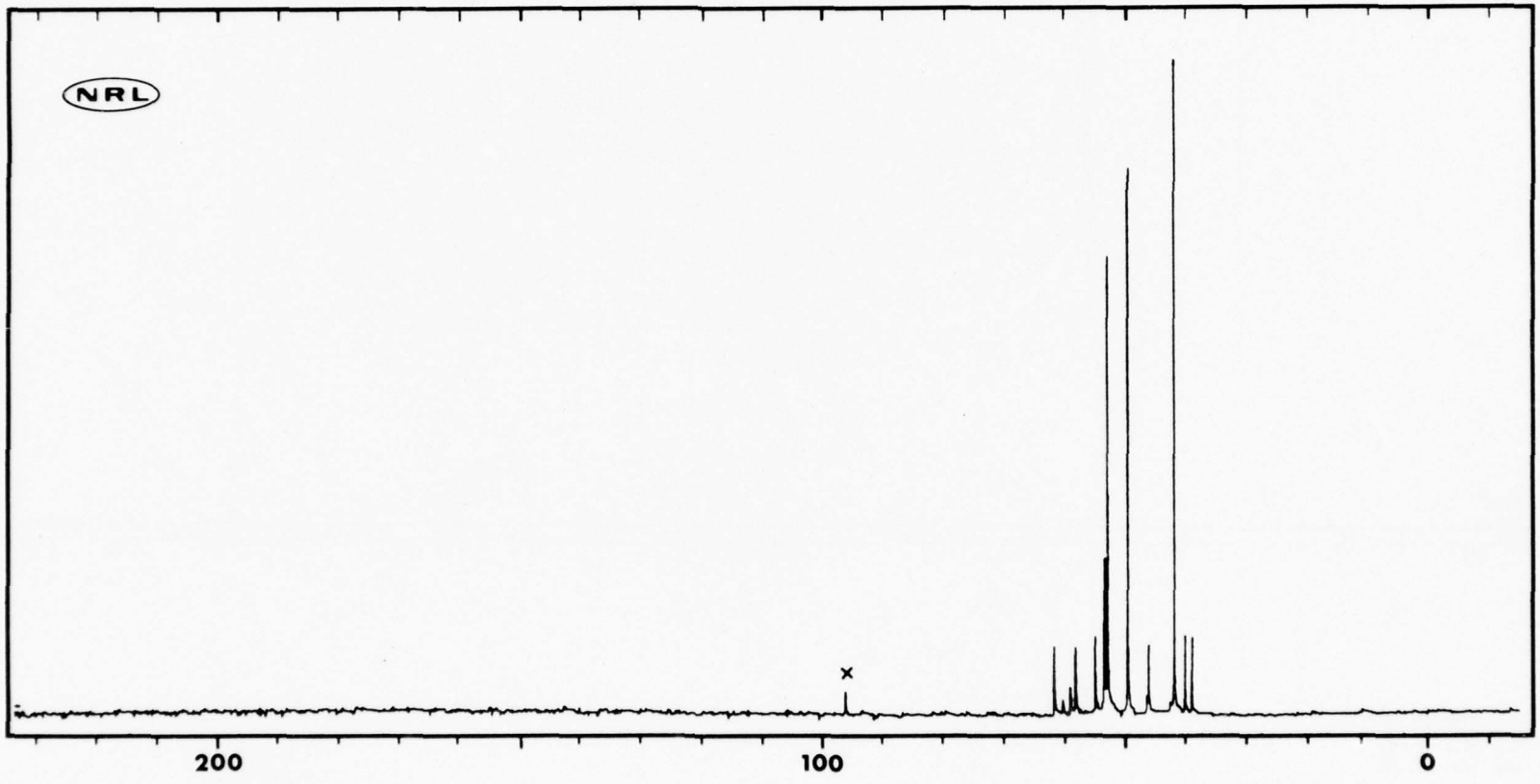


Source: Eastman T4572

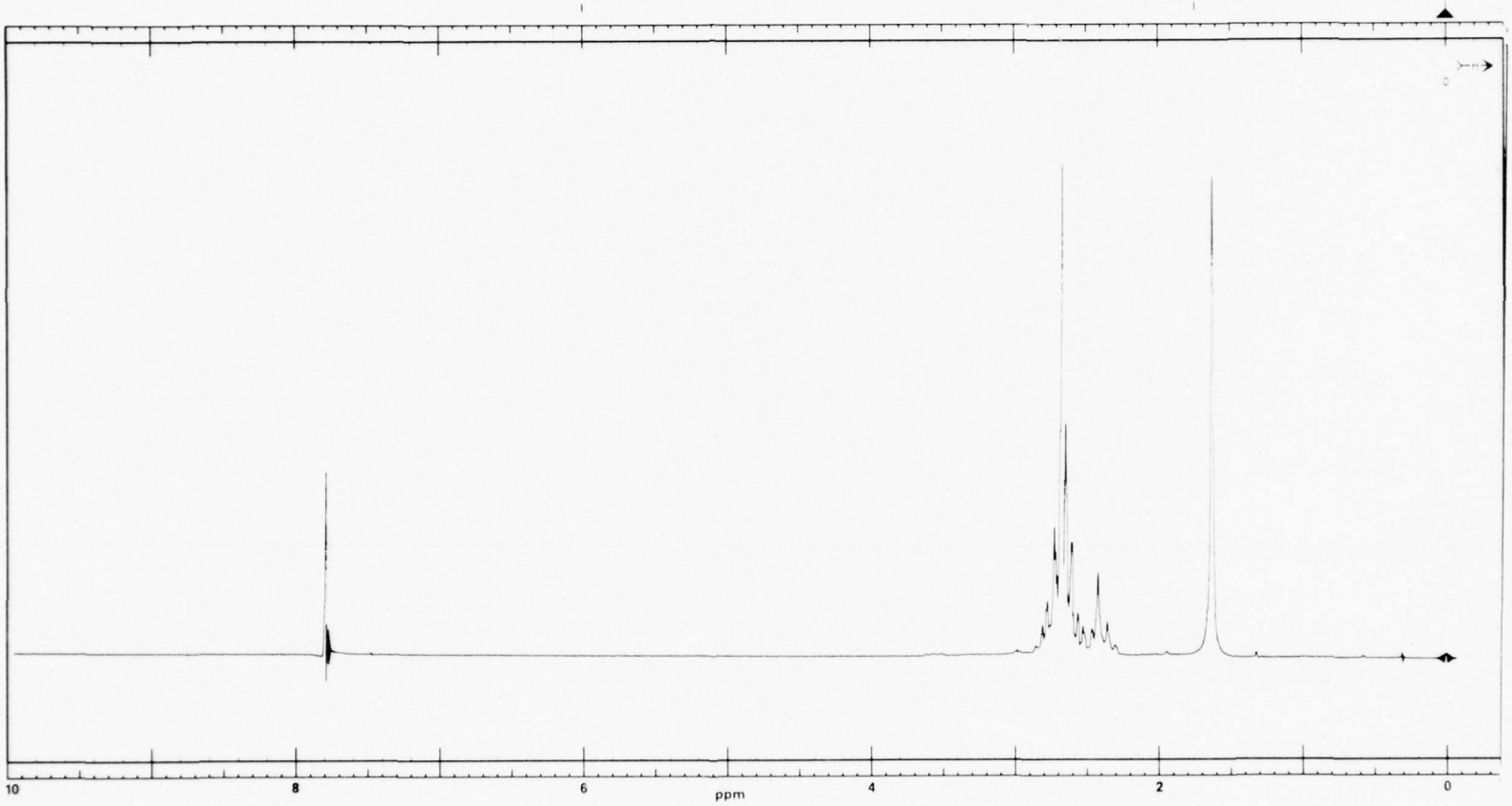
Solvent: 10% CCl_4 ×

Assignments:

a	41.9
b	49.5
c	52.8



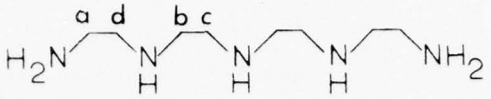
H26



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

C27

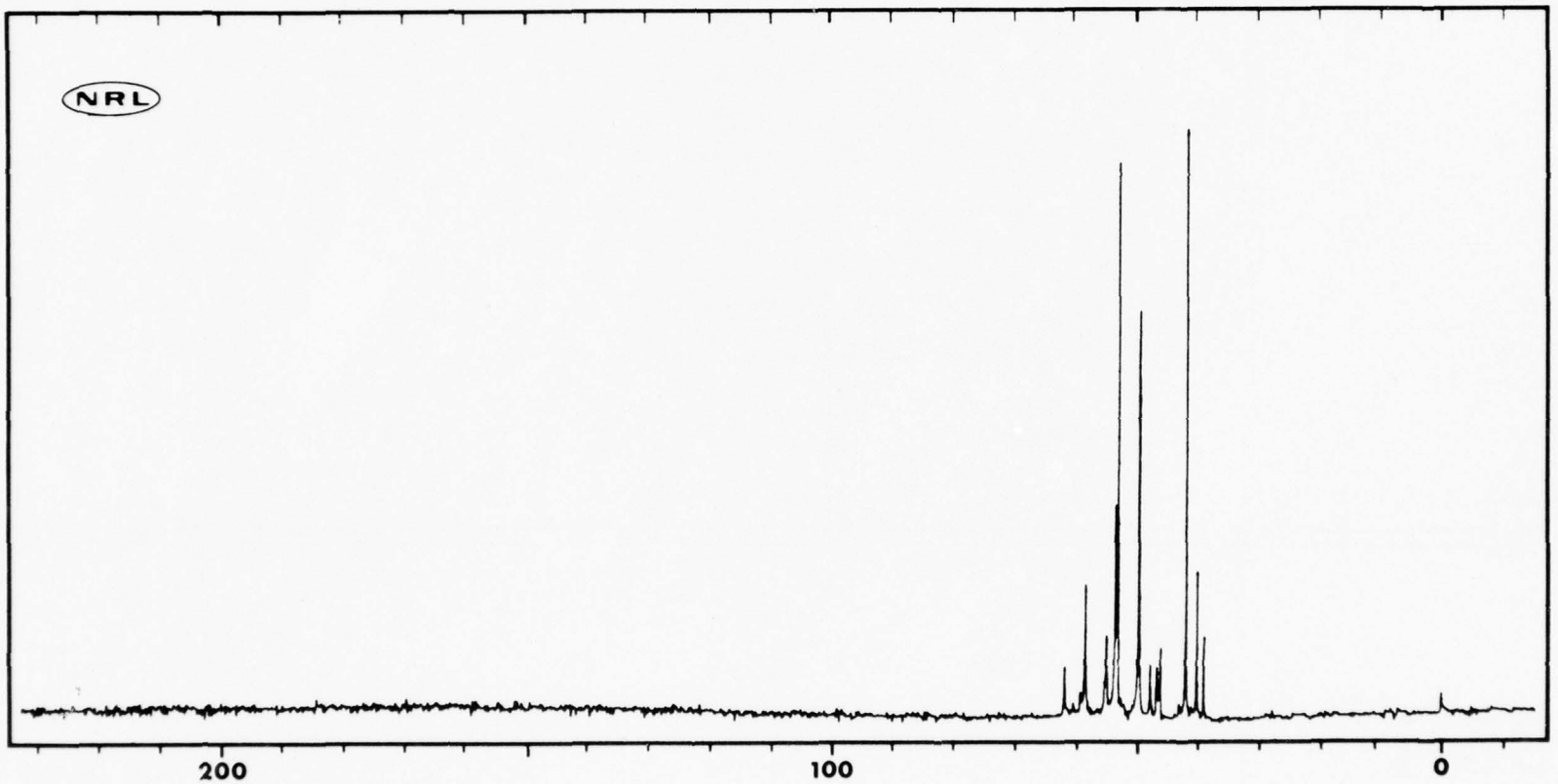
Tetraethylenepentamine



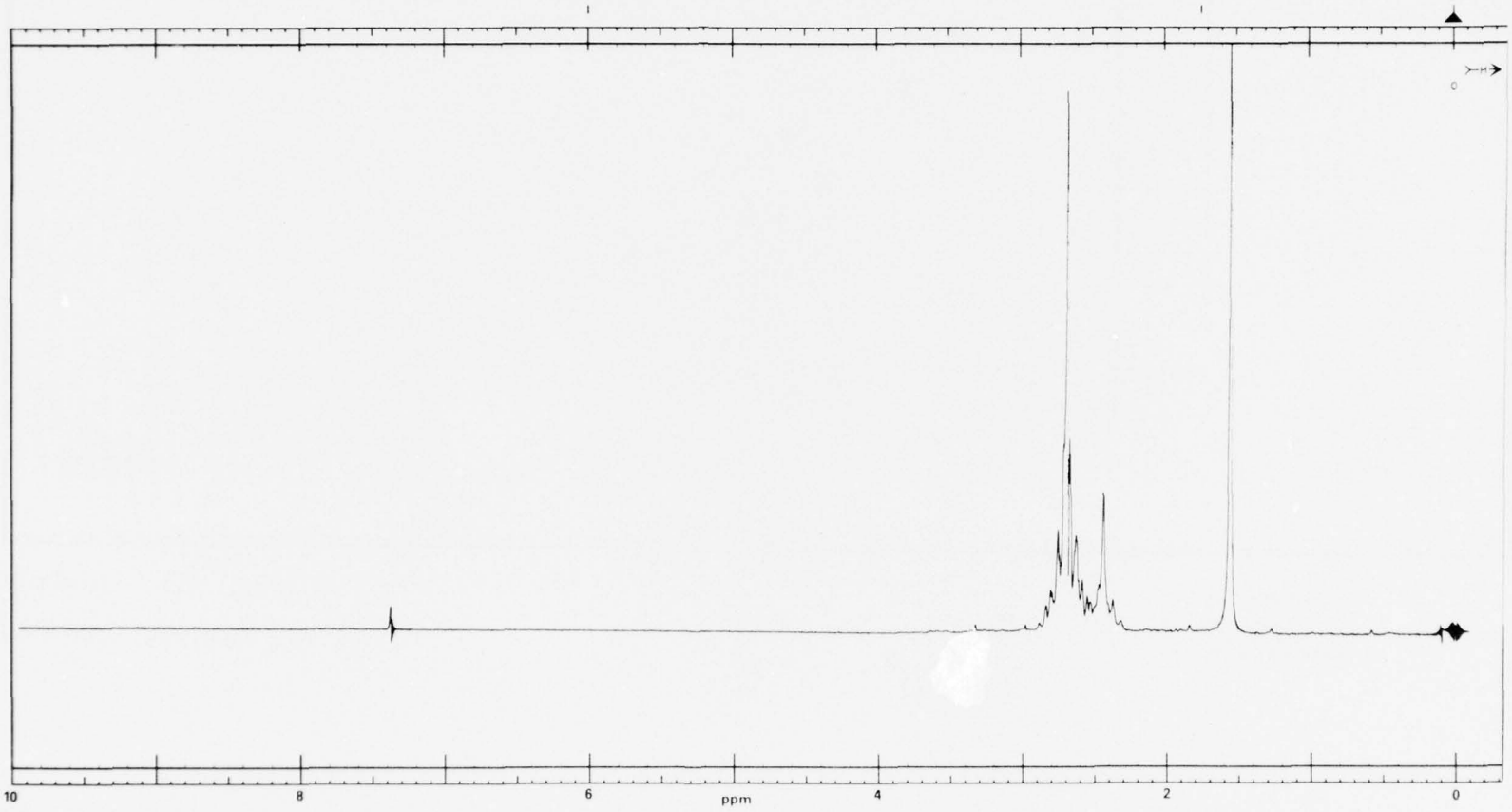
Source: Eastman T5902

Solvent: None (10 drops TMS added)

Assignments:
a 41.9
b,c 49.5
d 52.9

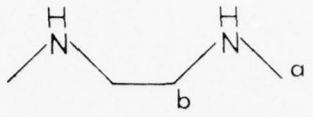


H27



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

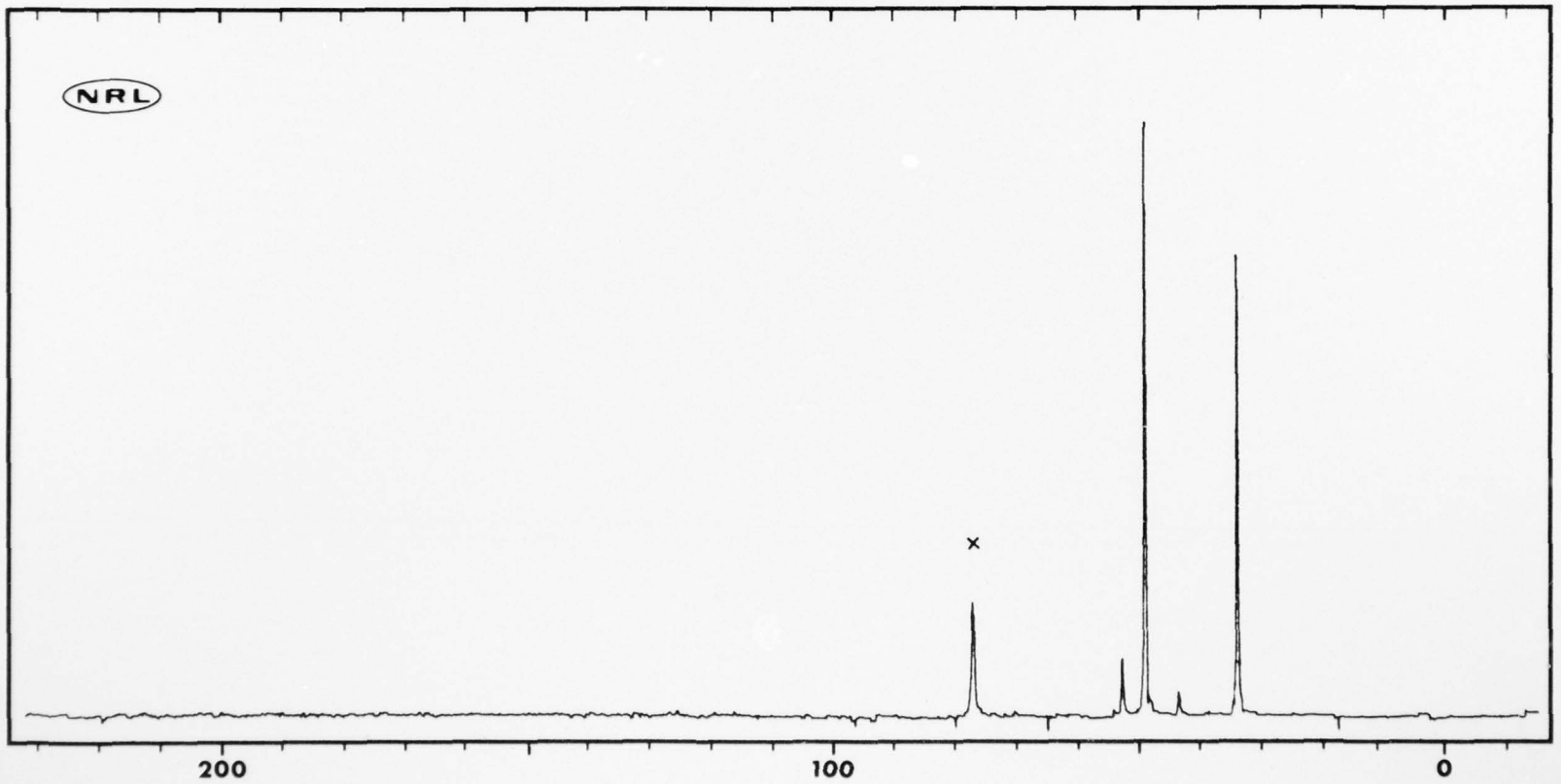
sym - Dimethylethylenediamine



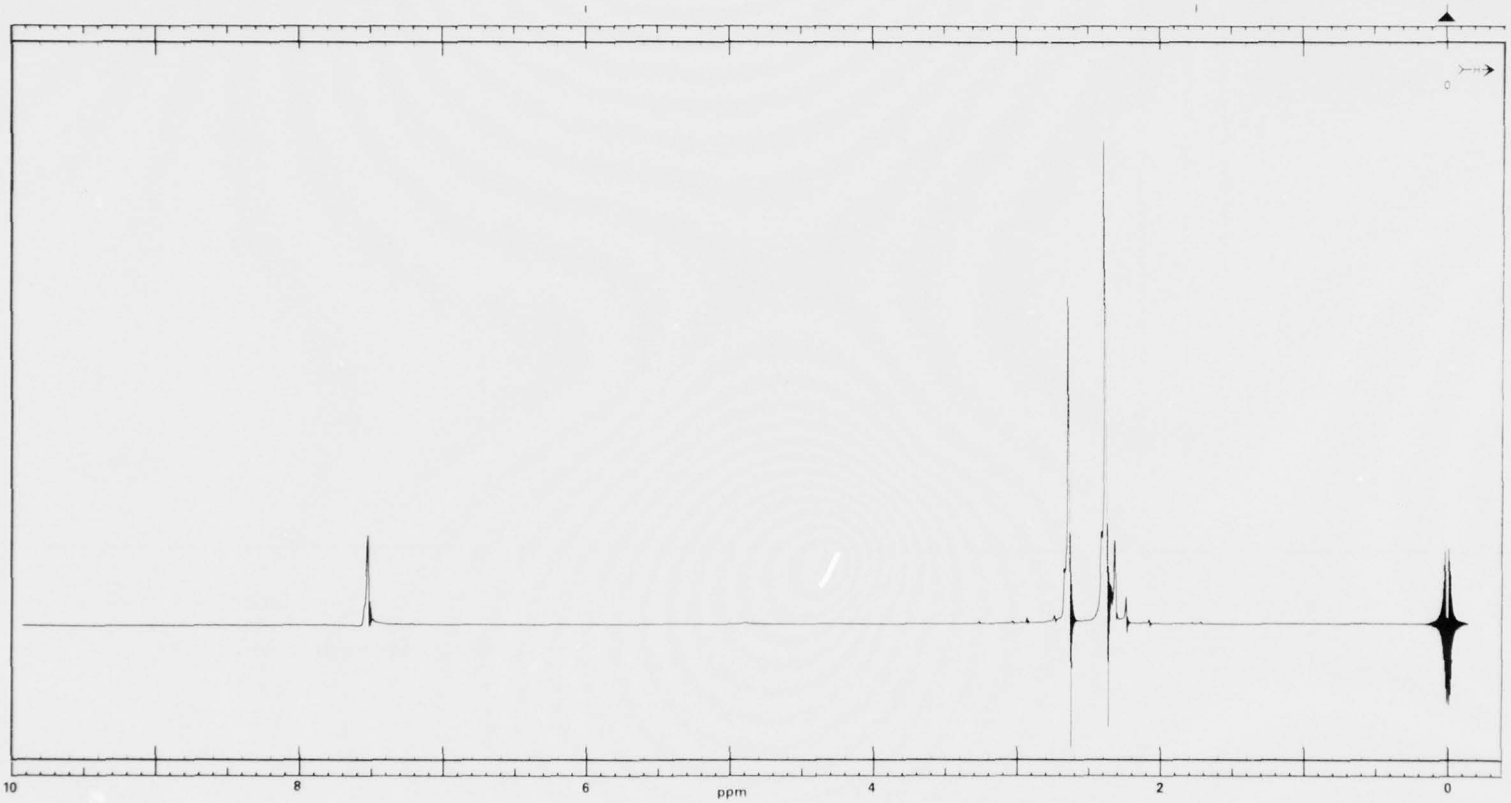
Source: Aldrich D15,780-5

Solvent: 25% $CHCl_3$ ×

Assignments:
a 34.0
b 49.1



H28

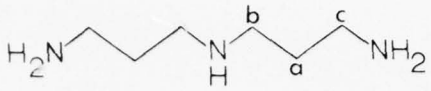


Spectrum 28 — *sym*-Dimethylethylenediamine (Aldrich D15,780-5); solvent: $CDCl_3$

3,3'-Iminobispropylamine

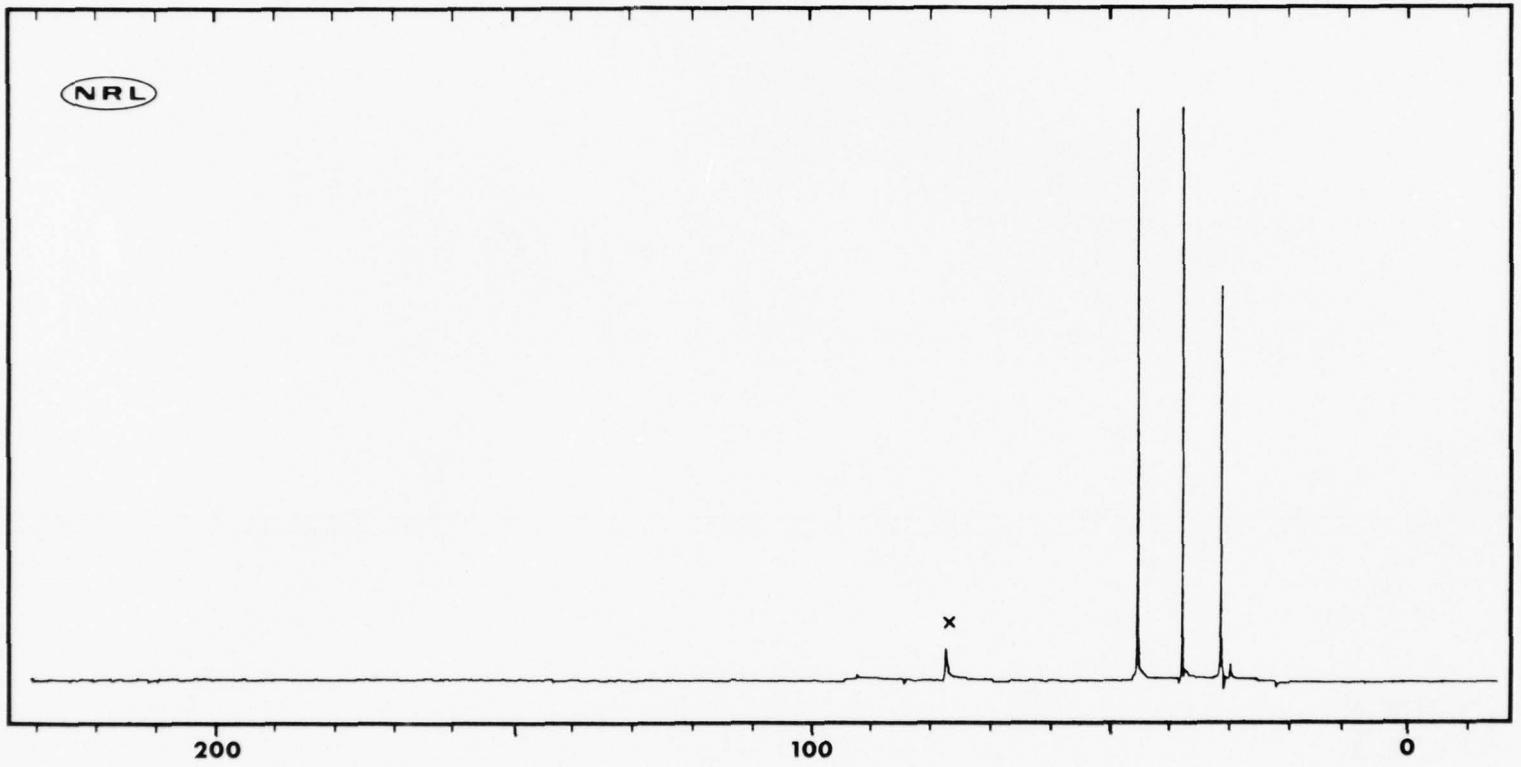
Source: American Cyanamid Company

Solvent: 10% $CHCl_3$ ×

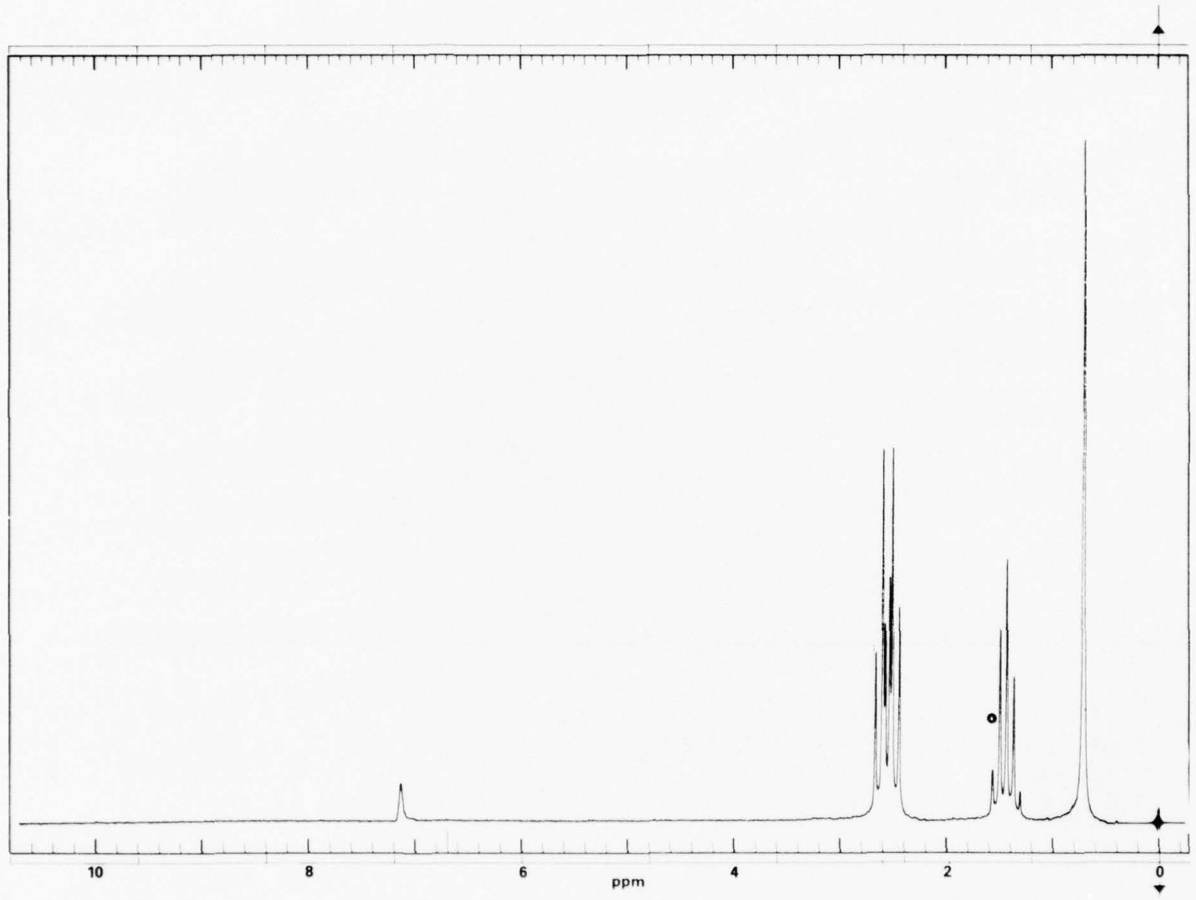


Assignments:

a	31.1
b	37.4
c	44.9



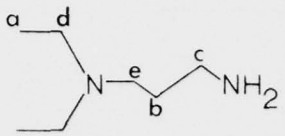
H29



Spectrum 29 — 3,3′-Imino-*bis*-propylamine (American Cyanamid Company); solvent: benzene-d_6

C30

3-Diethylaminopropylamine

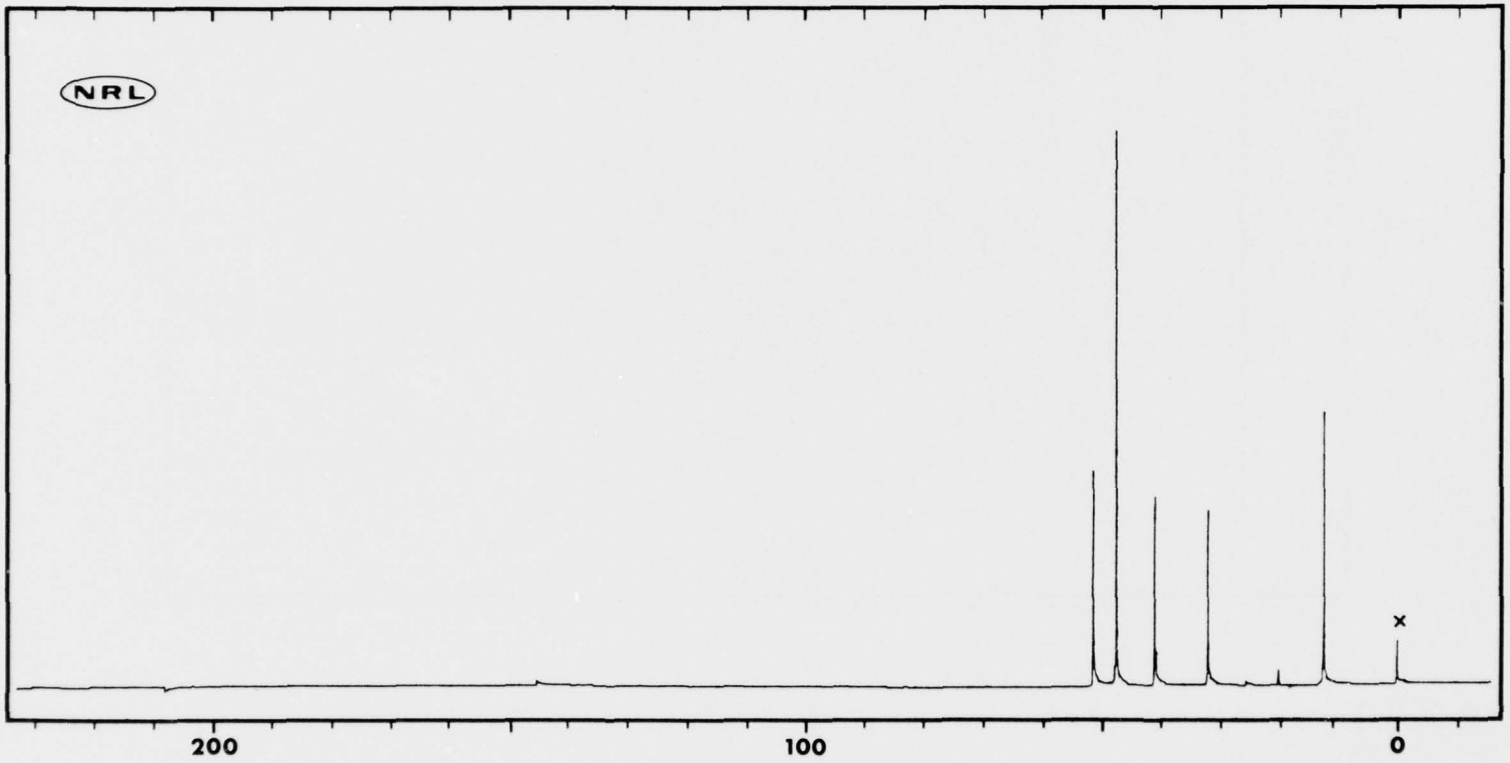


Assignments:

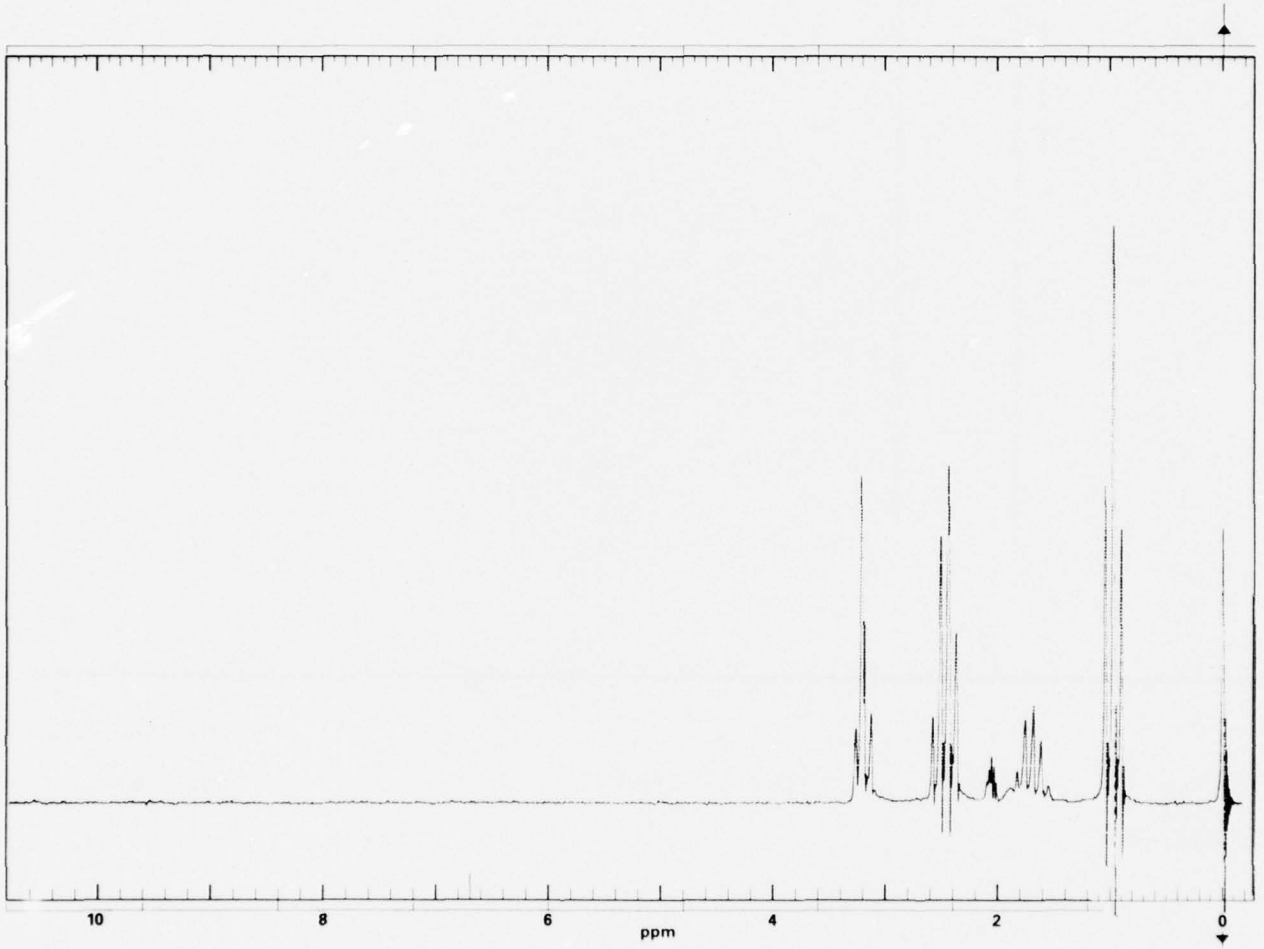
a	12.4
b	31.9
c	40.8
d	47.3
e	51.2

Source: Aldrich # D8,920-4

Solvent: 10% TMS ×



H30

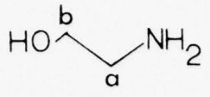


Spectrum 30 — 3-Diethylaminopropylamine (Aldrich D8,920-4); solvent: acetone-d_6

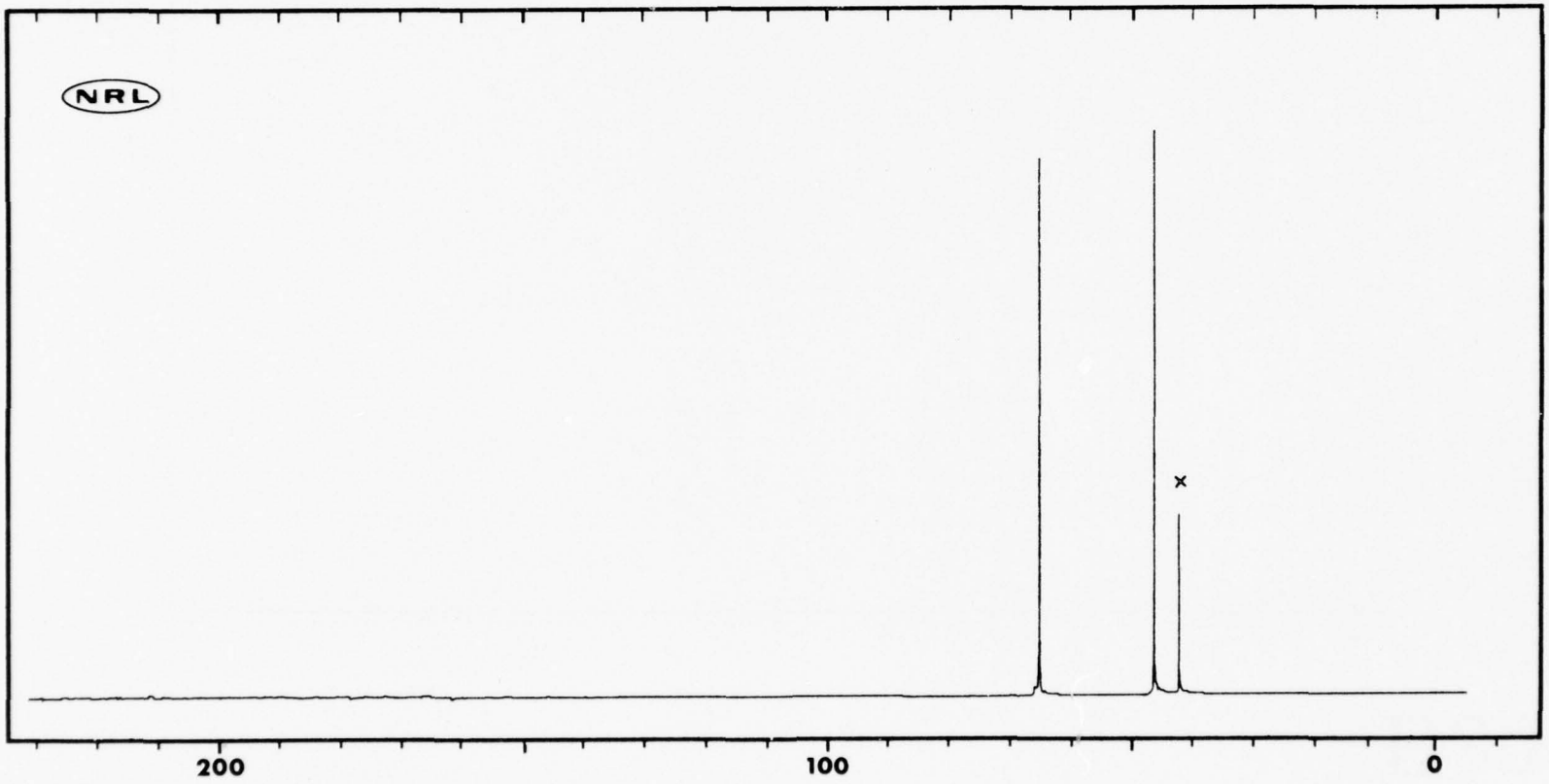
2-Aminoethanol

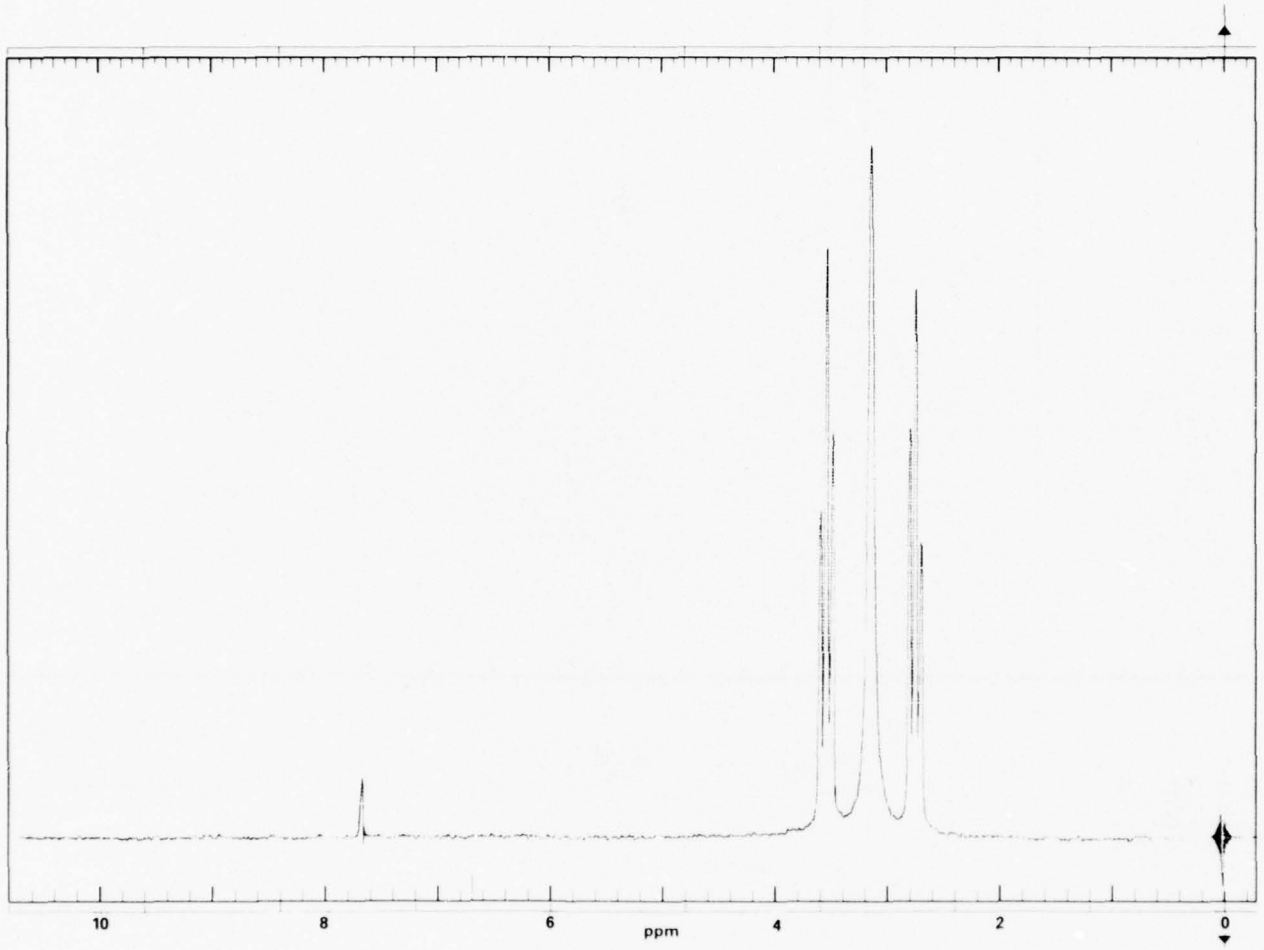
Source: Eastman 1597

Solvent: 25% DMSO ×



Assignments:
a 45.9
b 64.7

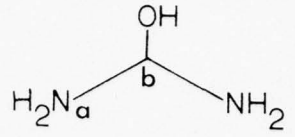




Spectrum 31 — 2-Aminoethanol (Eastman 1597); solvent: $CDCl_3$

C32

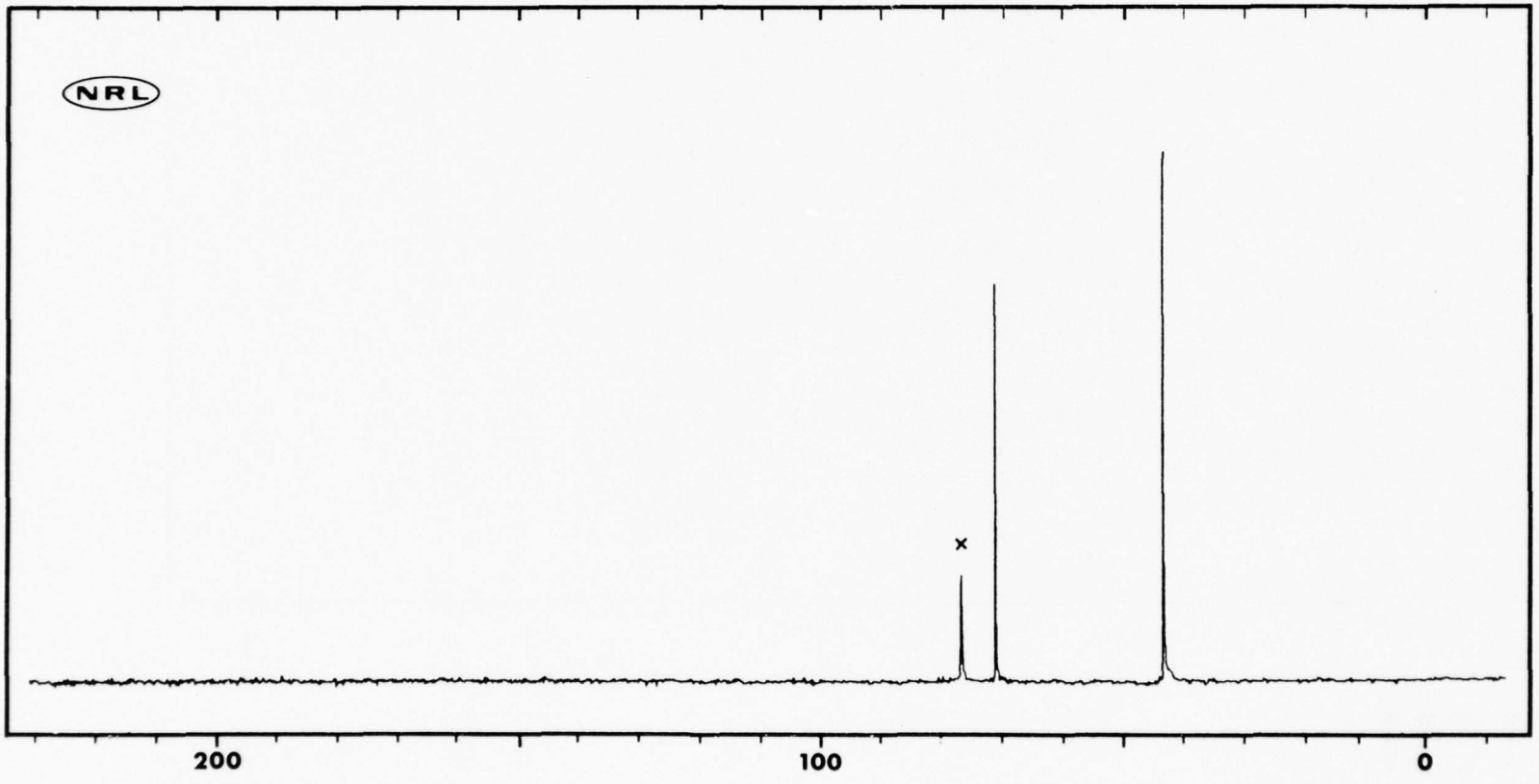
1,3-Diamino-2-propanol



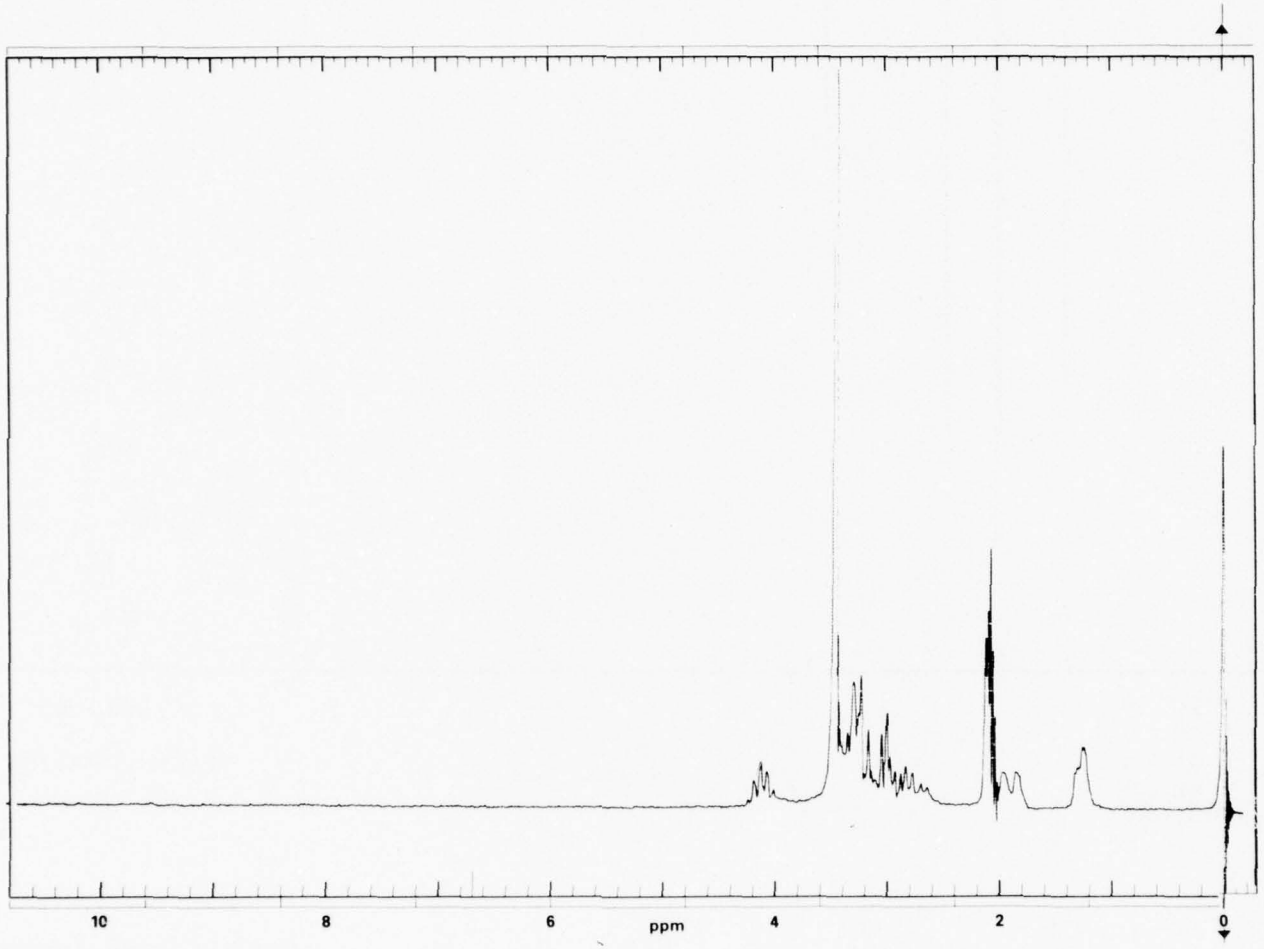
Assignments:
a 43.8
b 71.4

Source: Aldrich D1860

Solvent: 25% $CHCl_3$ ×

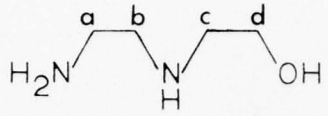


H32



Spectrum 32 — 1,3-Diamino-2-propanol (Aldrich D1860); solvent: acetone-d_6

2-(2-Aminoethylamino)ethanol

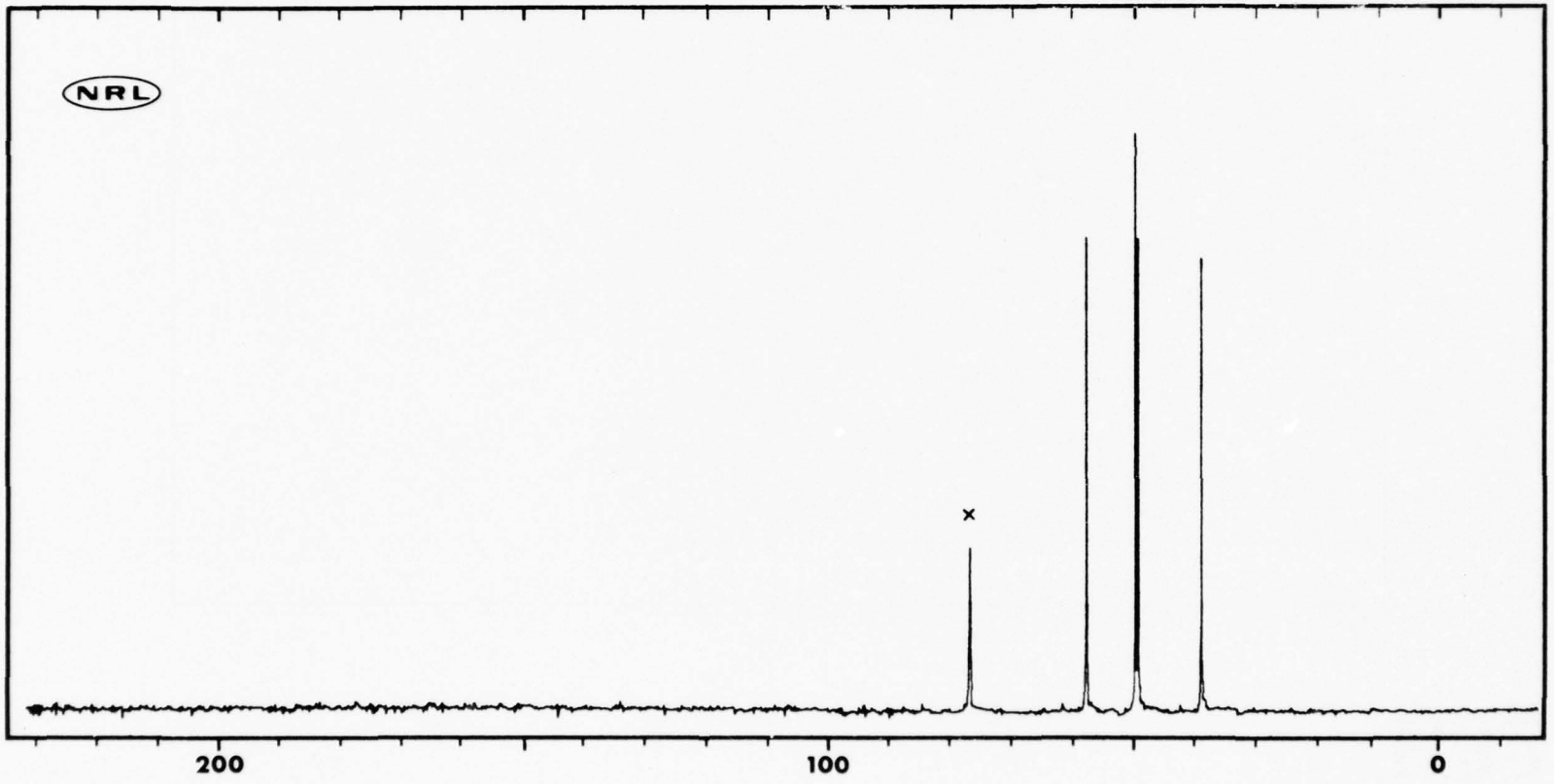


Assignments:

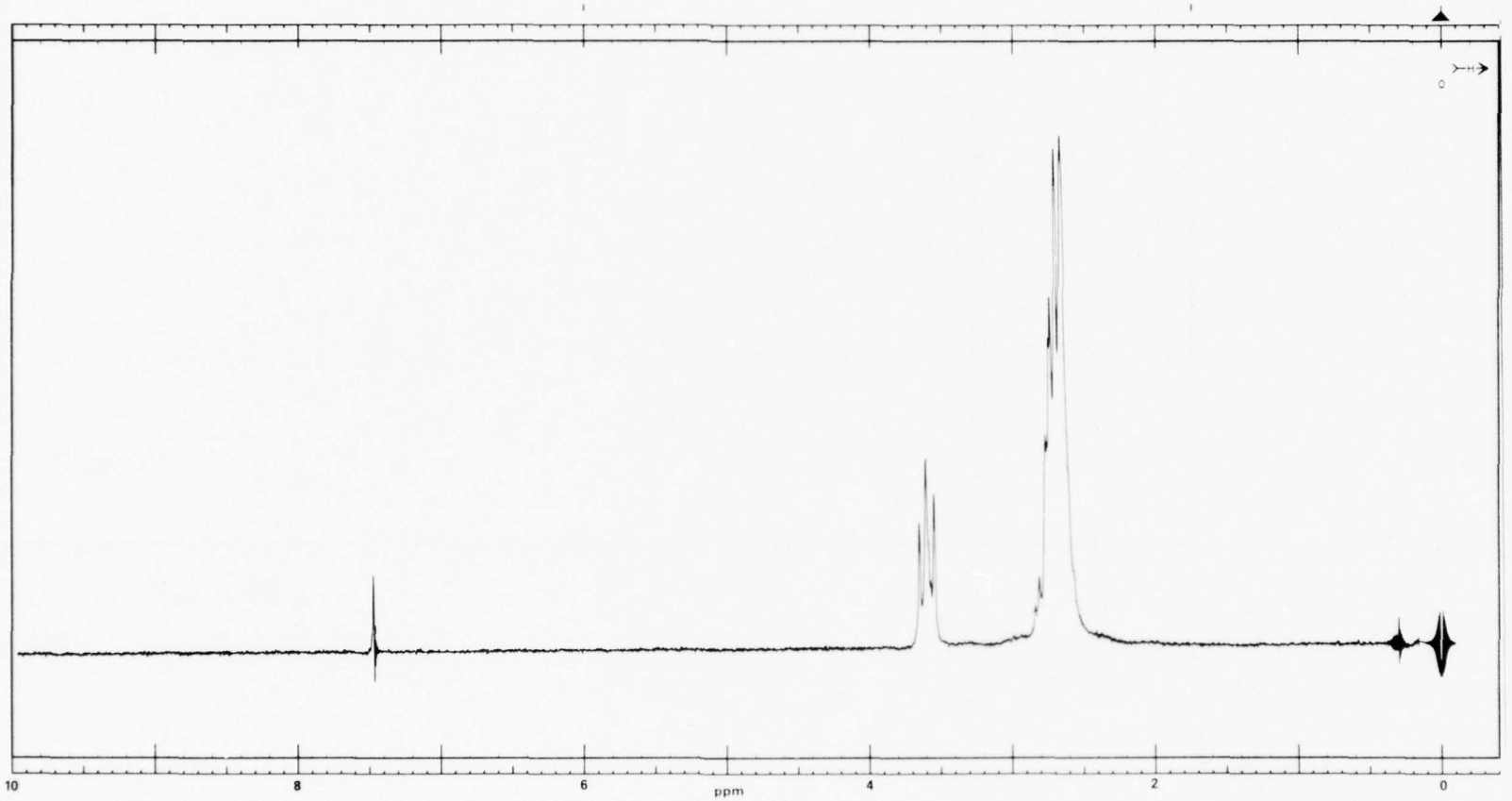
a	39.4
b	49.7
c	50.1
d	58.0

Source: Eastman 4774

Solvent: 10% $CHCl_3$ ×



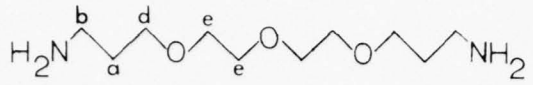
H33



Spectrum 33 — 2-(2-Aminoethylamino)ethanol (Eastman 4774); solvent: $CDC1_3$

C34

Polyglycol Diamine

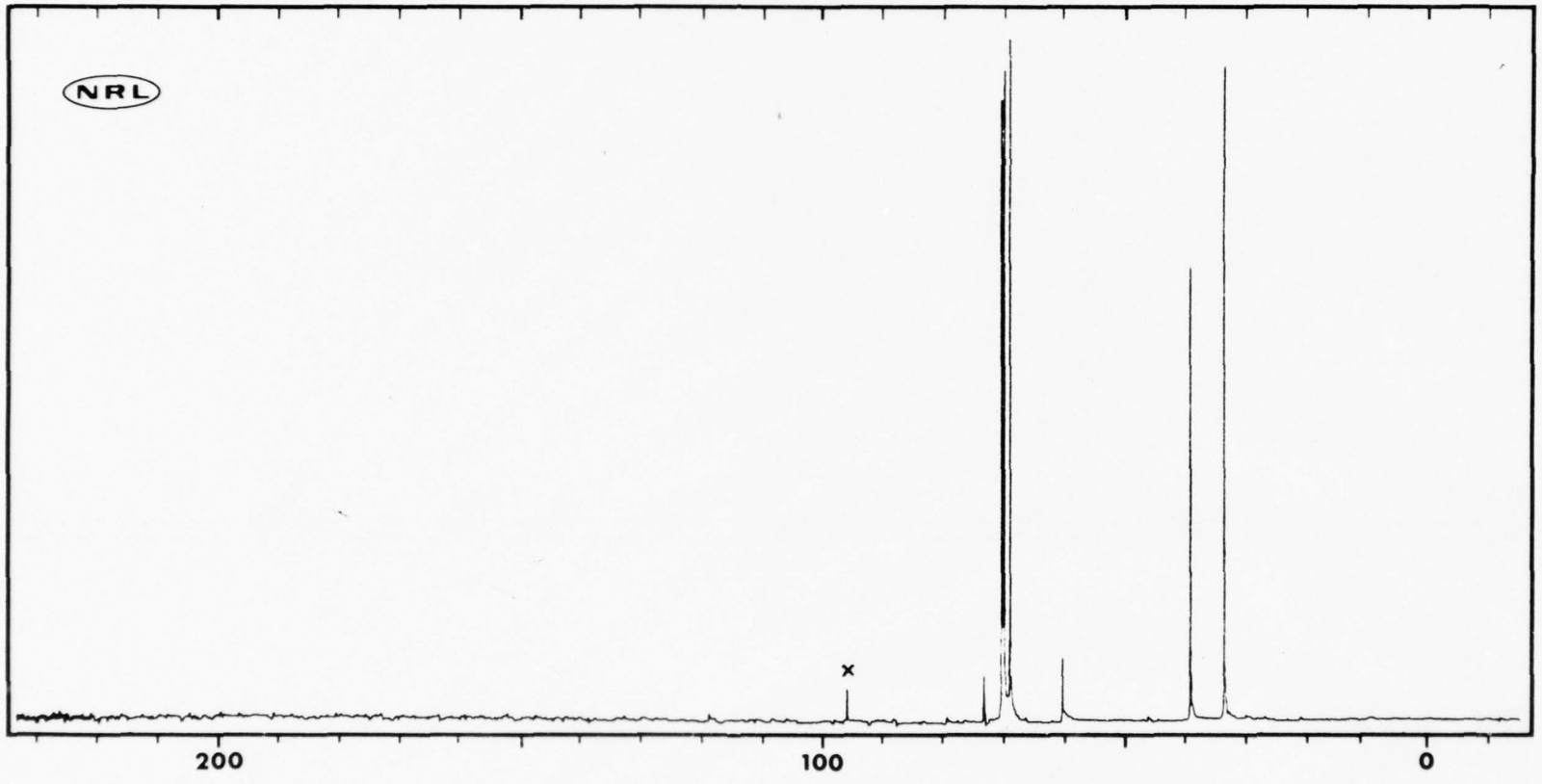


Assignments:

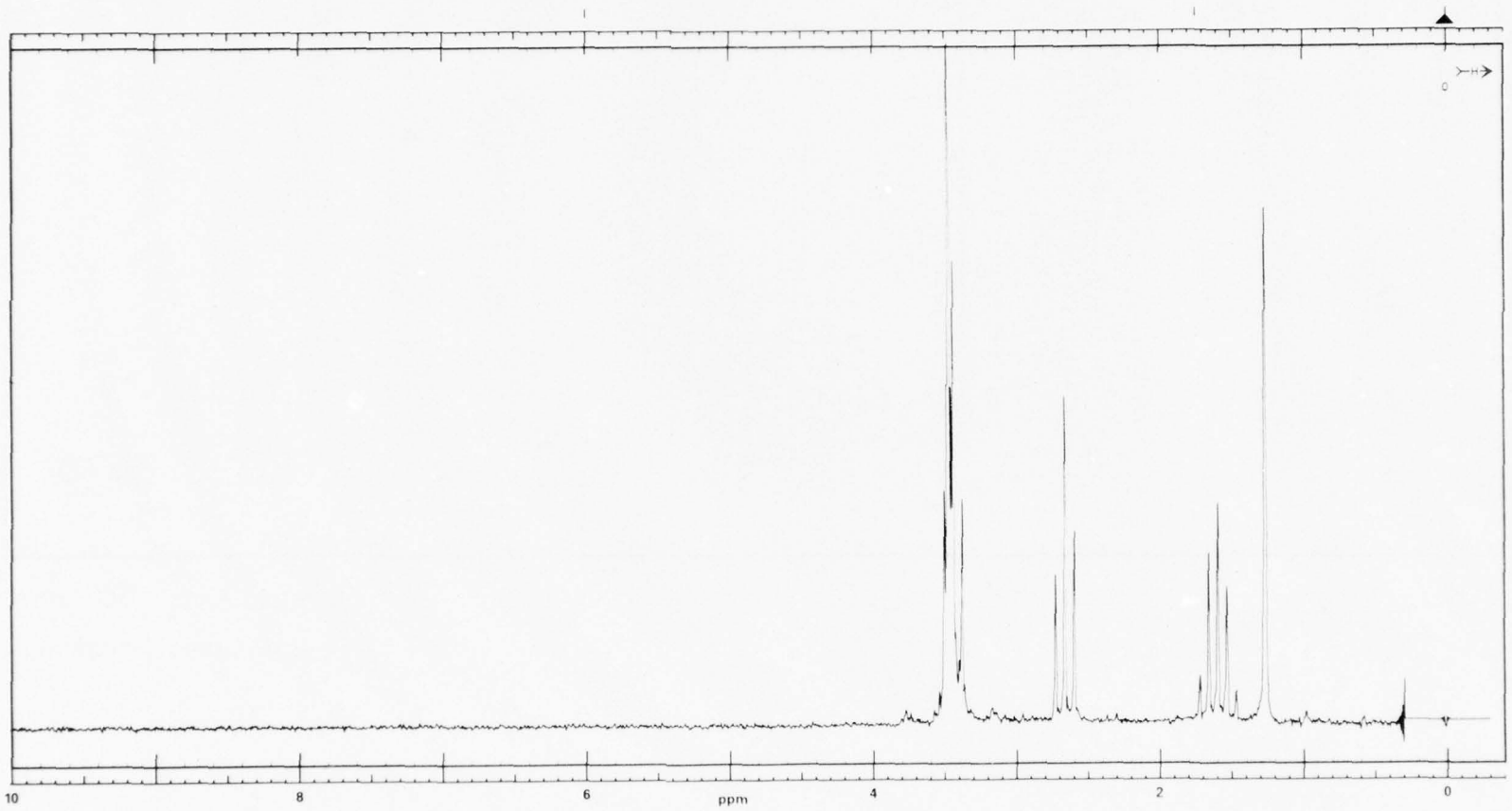
a	33.7
b	39.3
c	60.4
d	69.1
e	70.1
	70.5
f	73.4

Source: Union Carbide H-221

Solvent: 20% CCl_4 ×



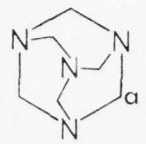
H34



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

C35

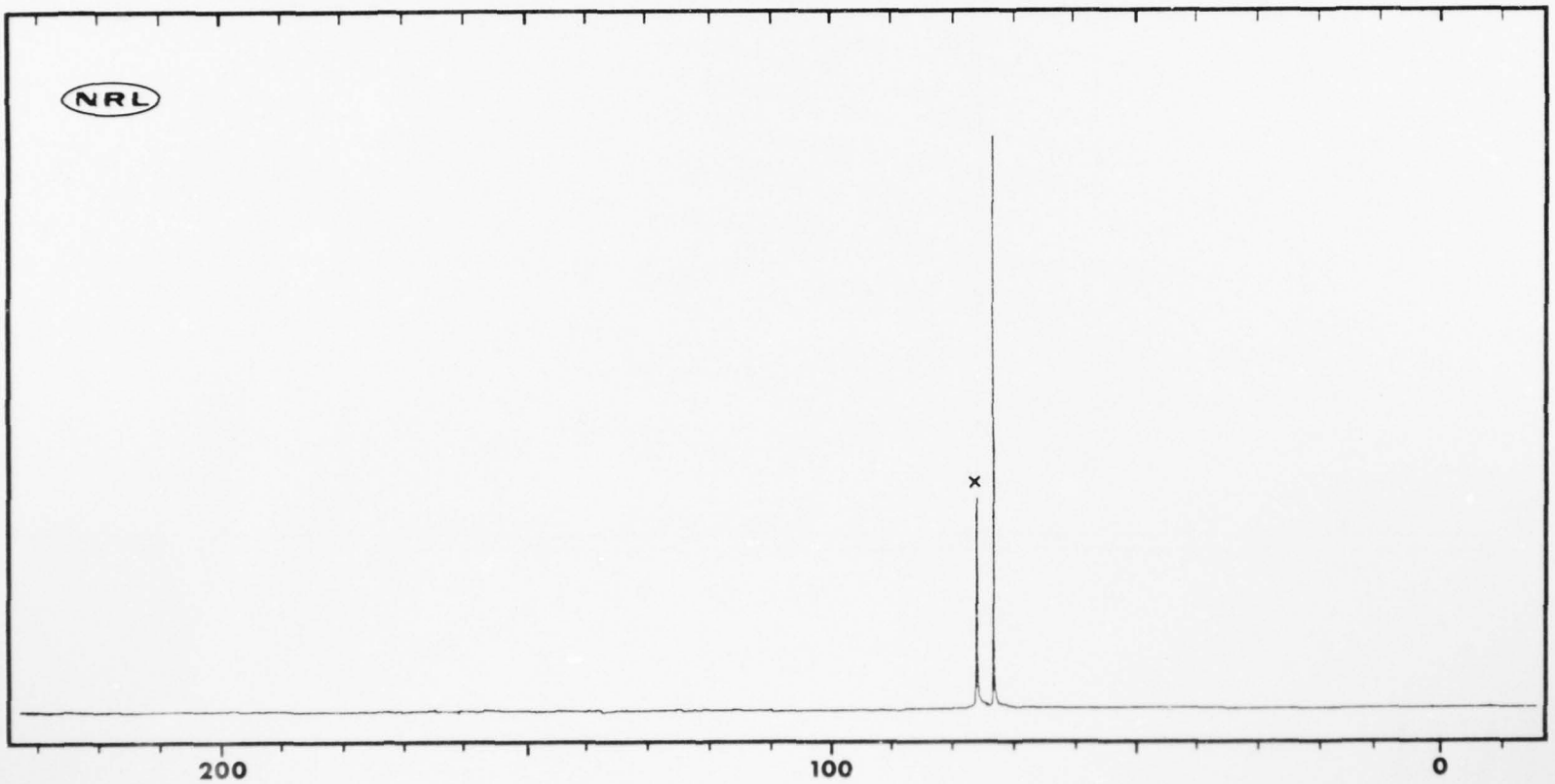
Hexamethylenetetramine



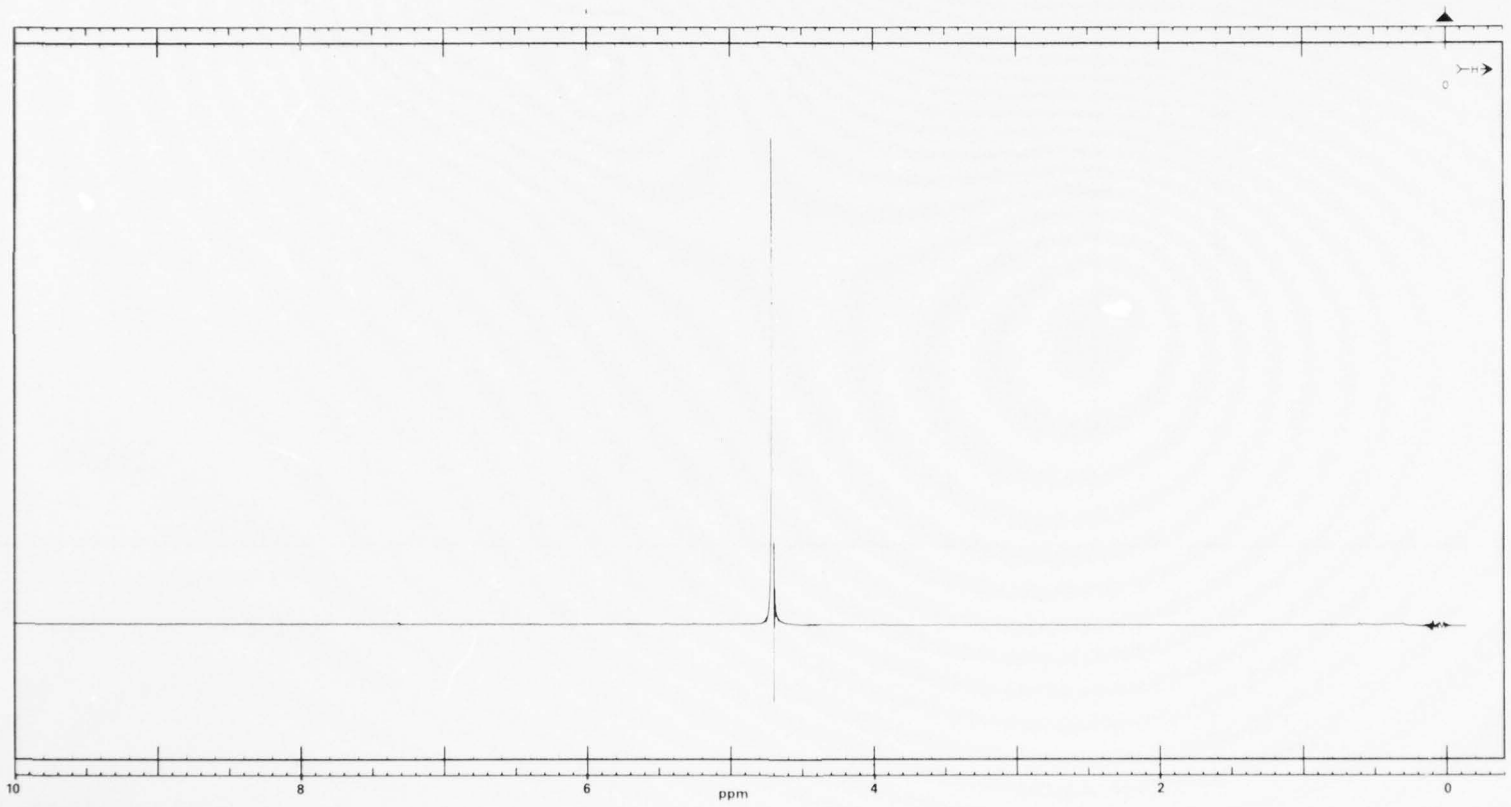
Source: Eastman 145

Solvent: 75% $CHCl_3$ ×

Assignments:
a 74.4



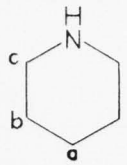
H35



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

C36

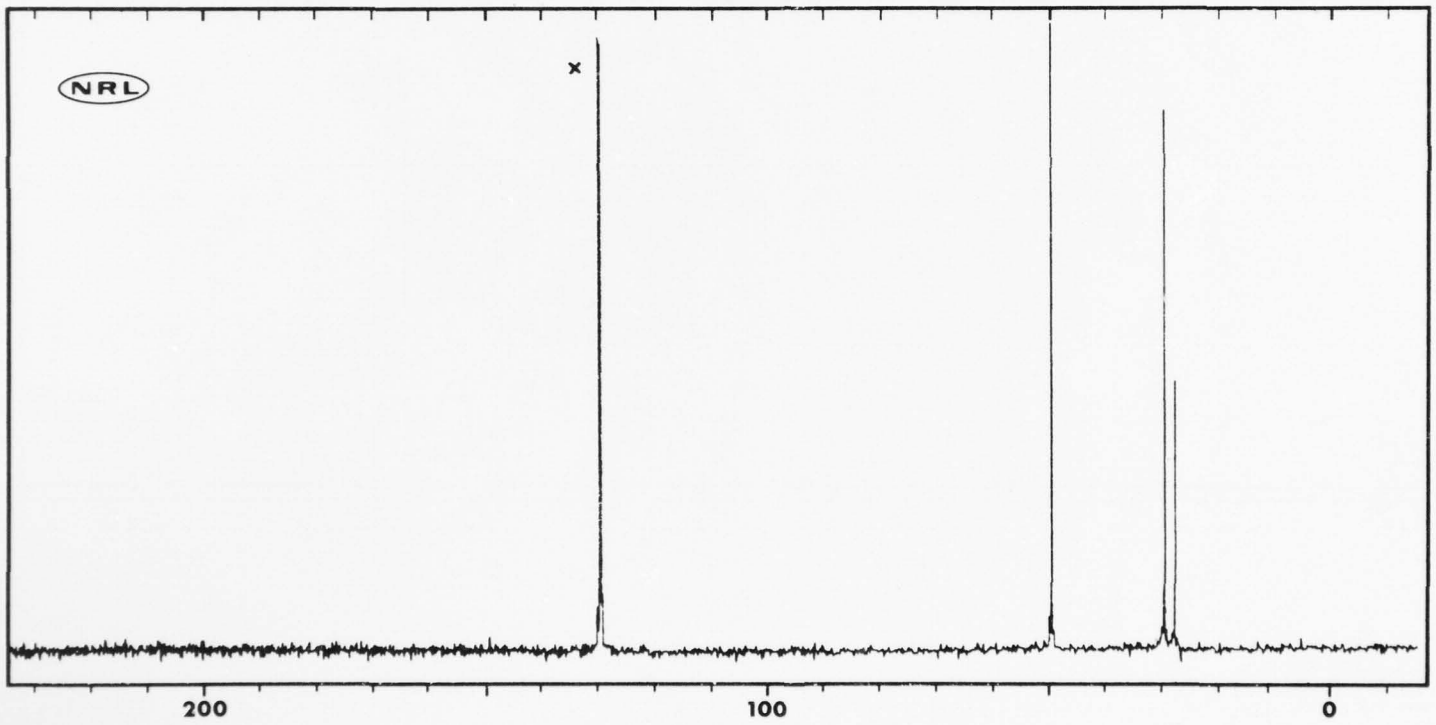
Piperidine



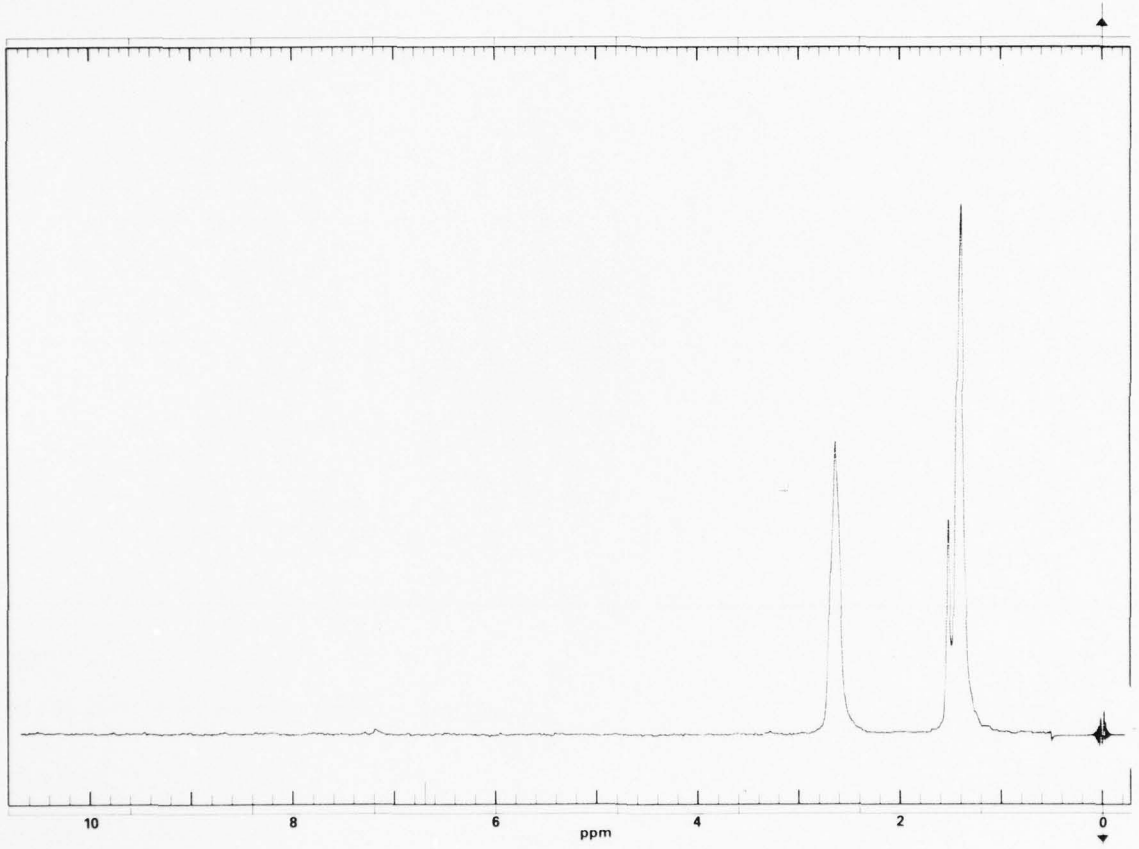
Assignments:
a 26.6
b 28.5
c 48.6

Source: Fisher P-125

Solvent: 75% benzene ×



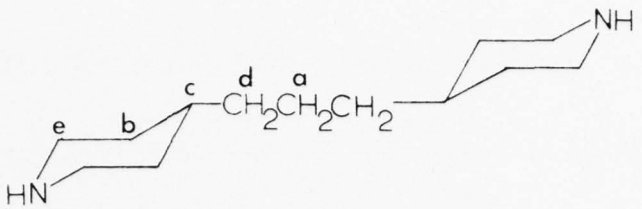
H36



Spectrum 36 — Piperidine (Fisher P-125); solvent: 75% benzene-d_6

C37

4,4'-Trimethylene-dipiperidine

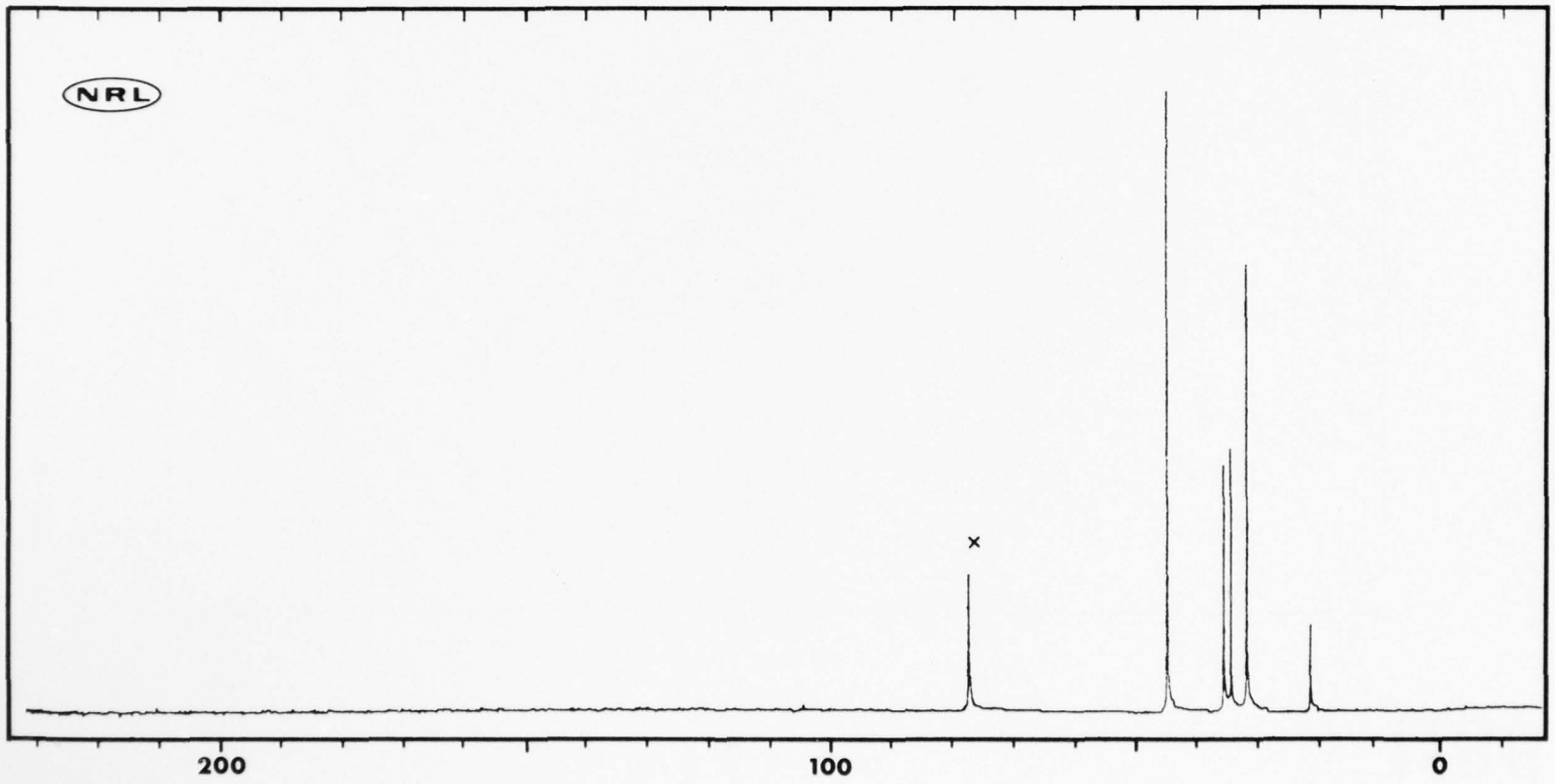


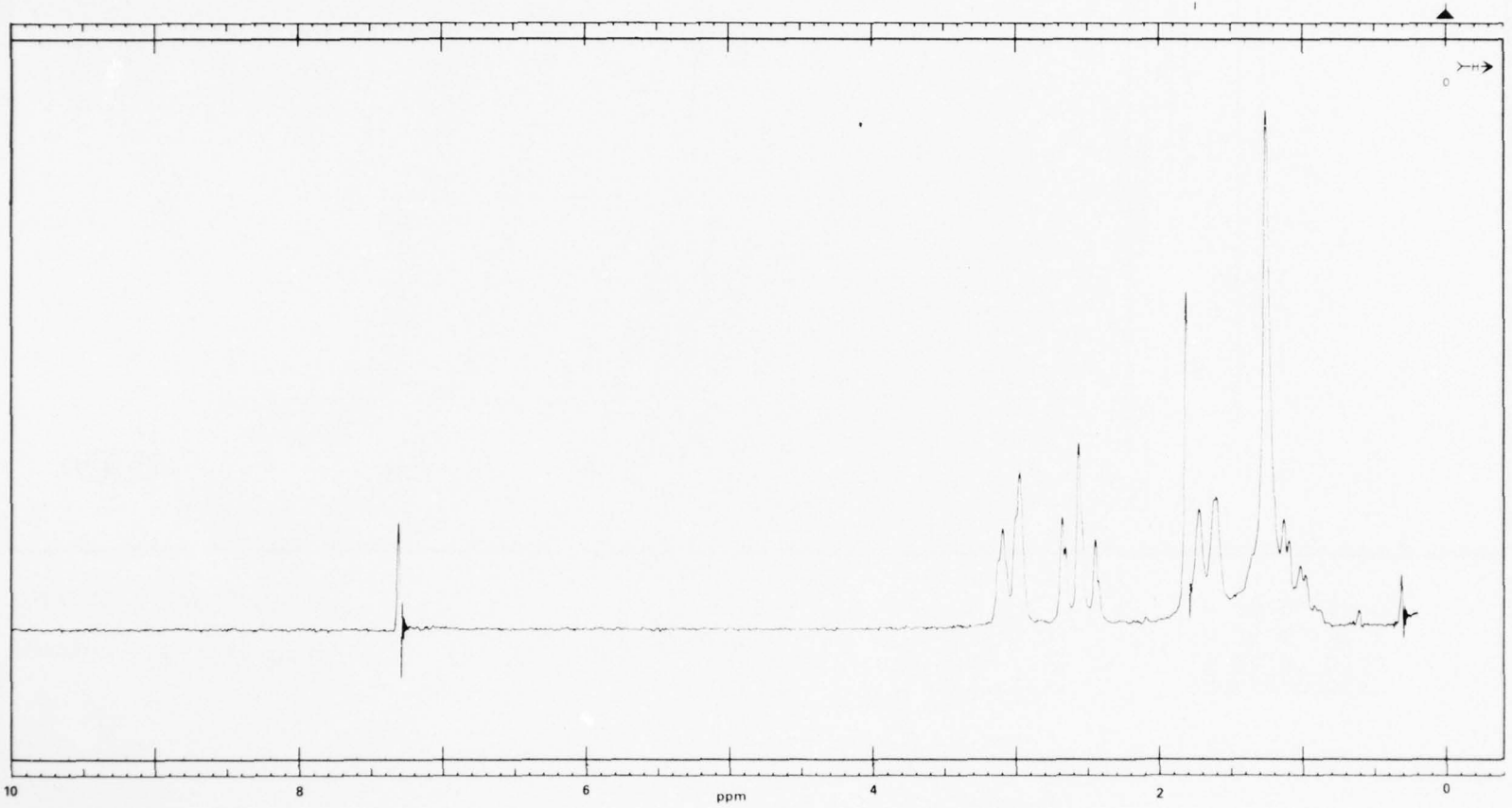
Source: Reilly Tar & Chemical Corp. "4-DI-PIP"

Solvent: $CHCl_3$ ×

Assignments:

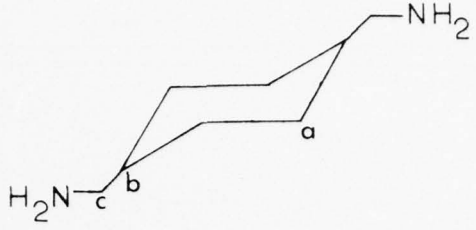
a	21.2
b	31.5
c	34.2
d	35.4
e	44.6





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

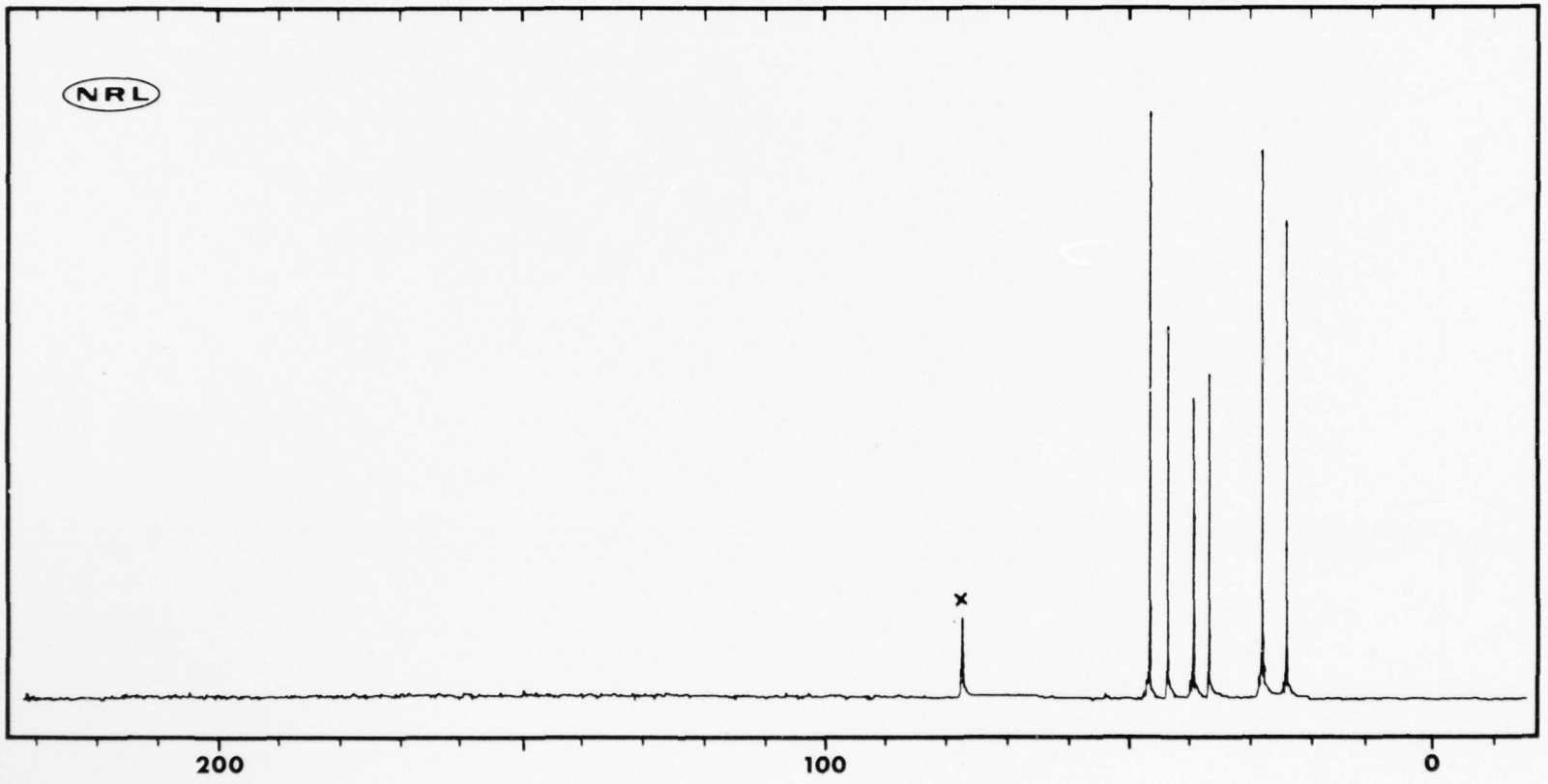
1,4 Cyclohexanebis(methylamine)

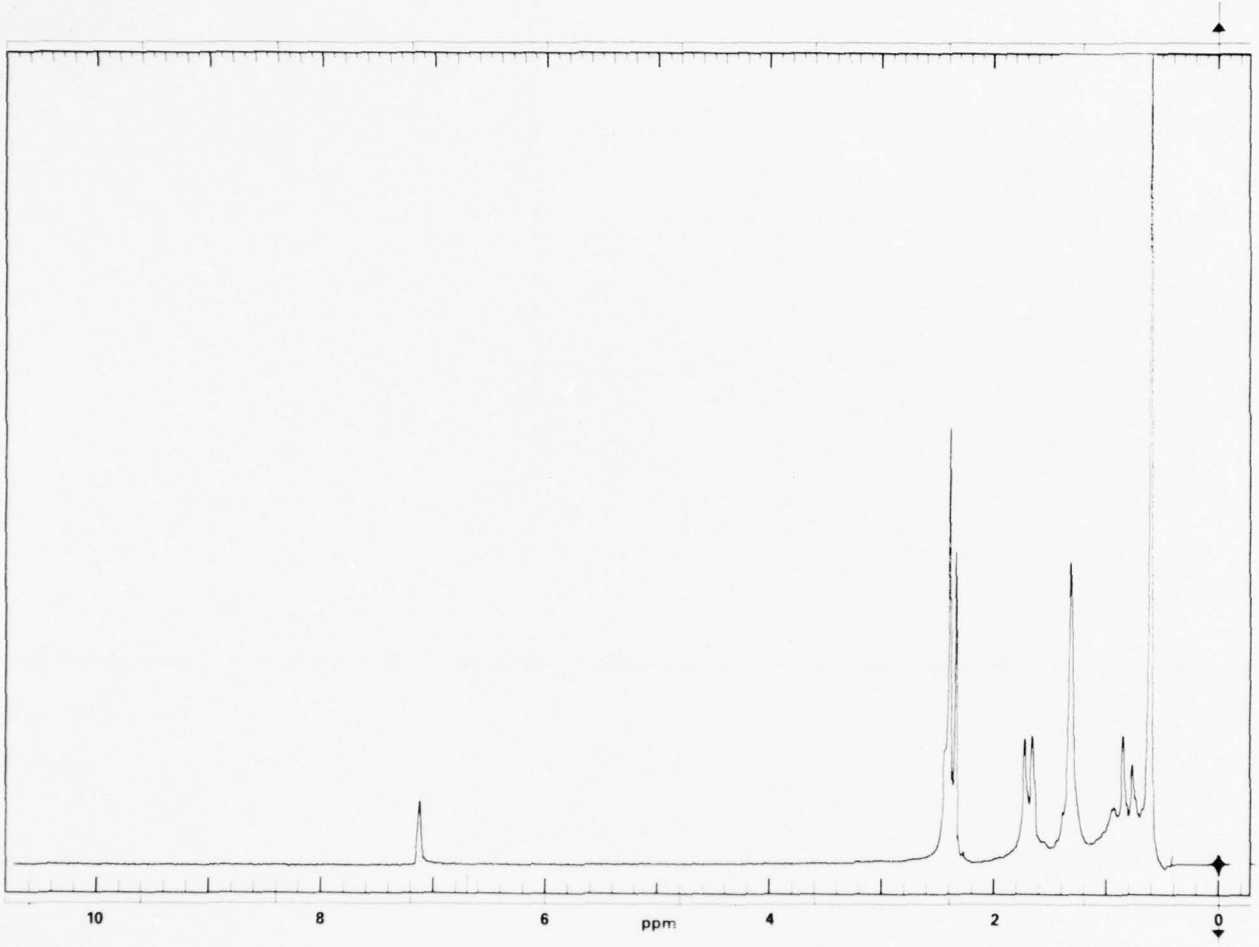


Source: Eastman P7562

Solvent: 10% $CHCl_3$ ×

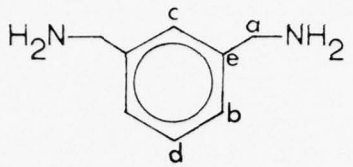
Assignments:
a 24.0, 27.9
b 36.6, 39.1
c 43.4, 46.3





Spectrum 38 — 1,4-Cyclohexane-*bis*(methylamine) (Eastman P7562); solvent: benzene-d_6

m-Xylenediamine

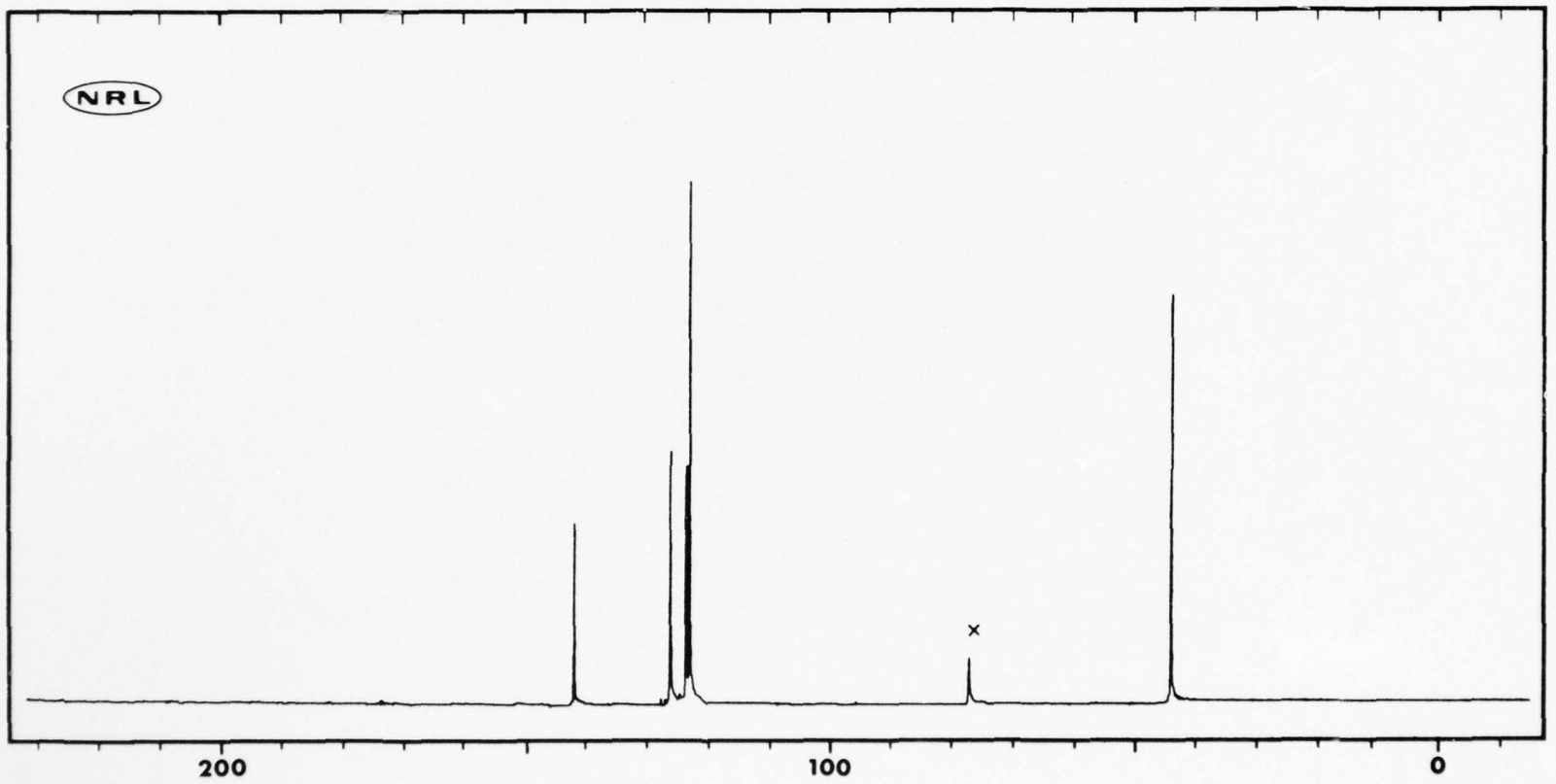


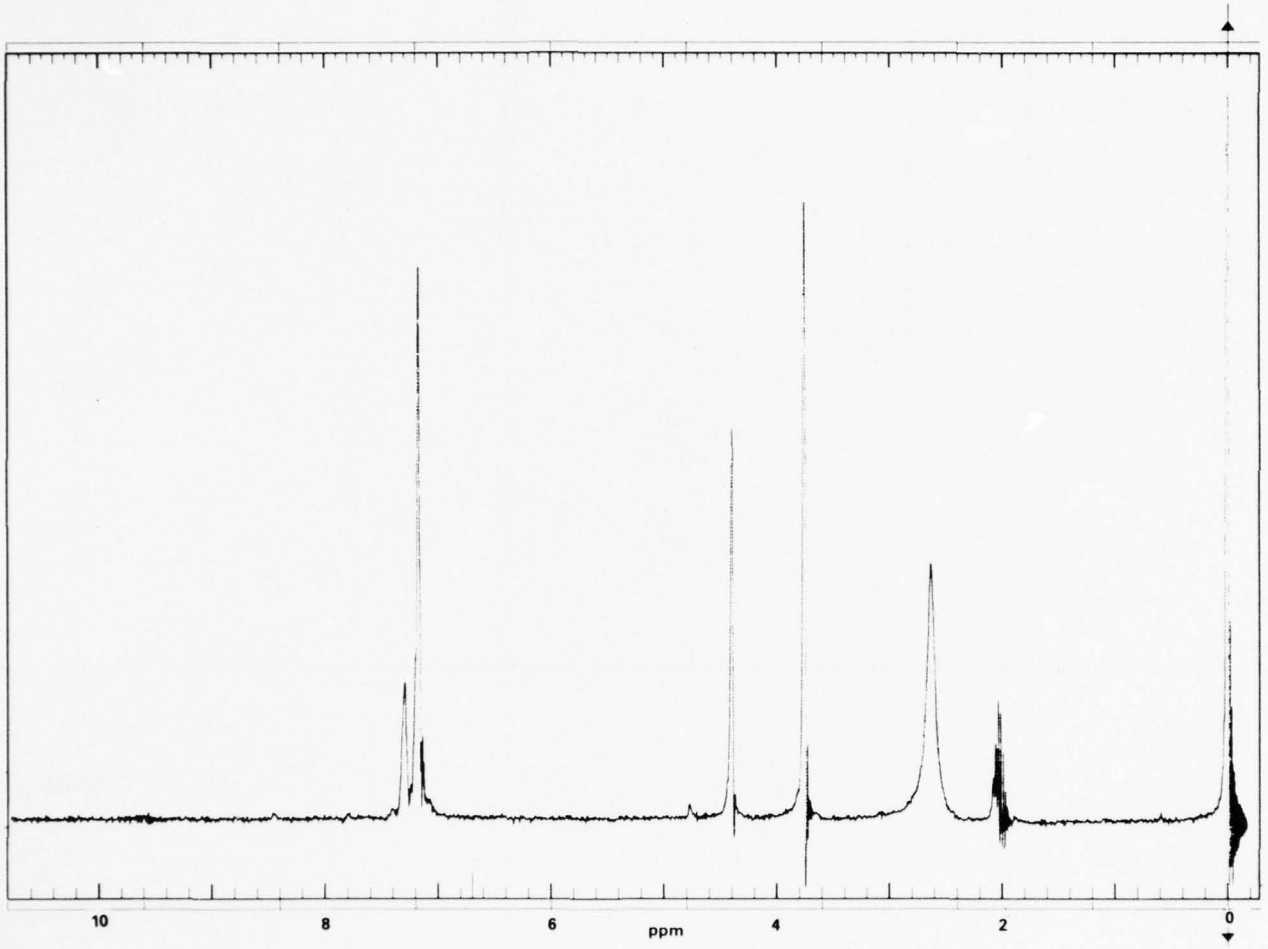
Assignments:

a	44.0
b	122.6
c	123.2
d	125.7
e	141.4

Source: unknown

Solvent: 10% $CHCl_3$ ×

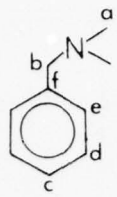




Spectrum 39 — *m*-Xylenediamine (source unknown); solvent: acetone-d_6

C40

N,N-Dimethyl benzylamine

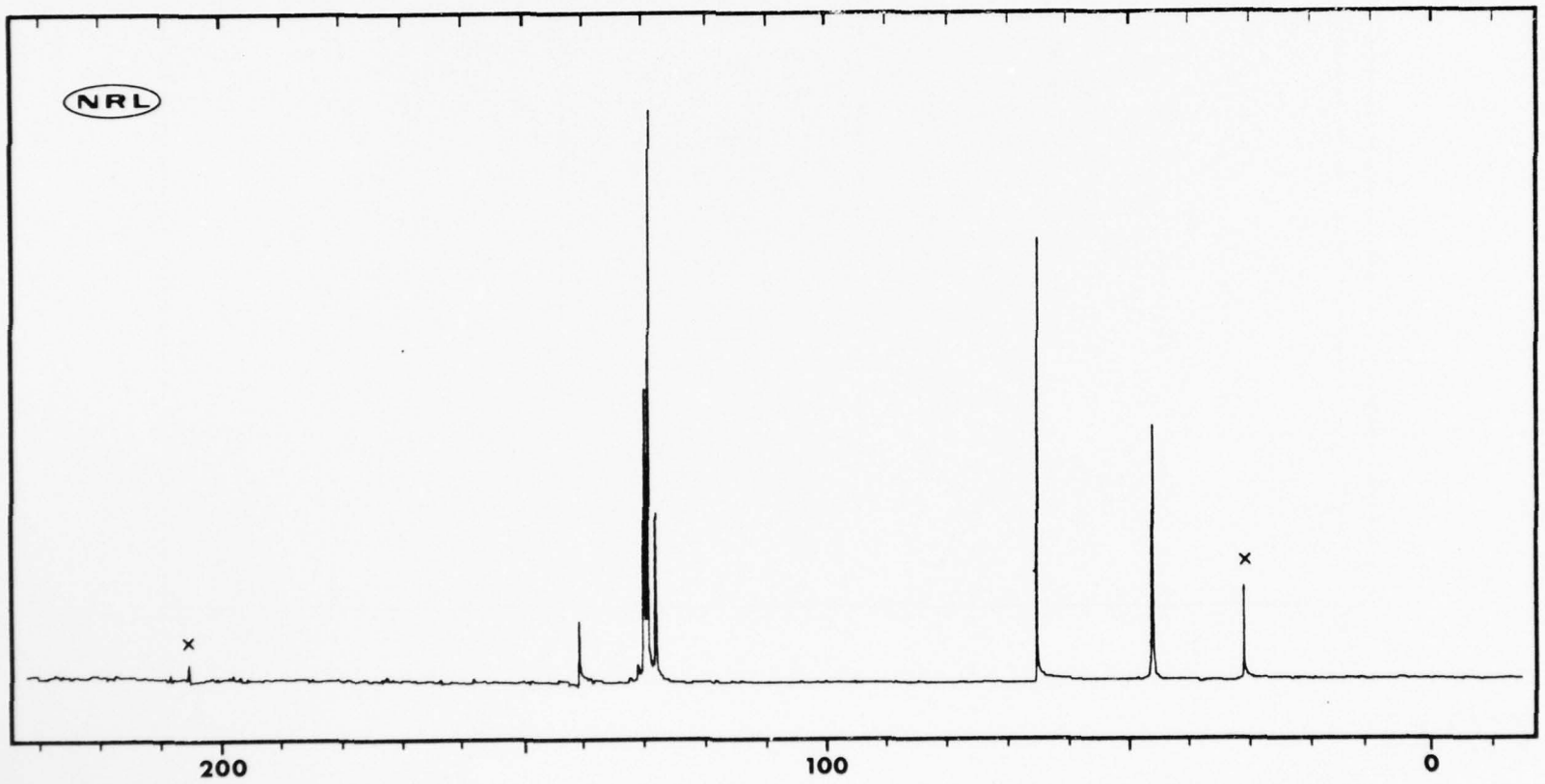


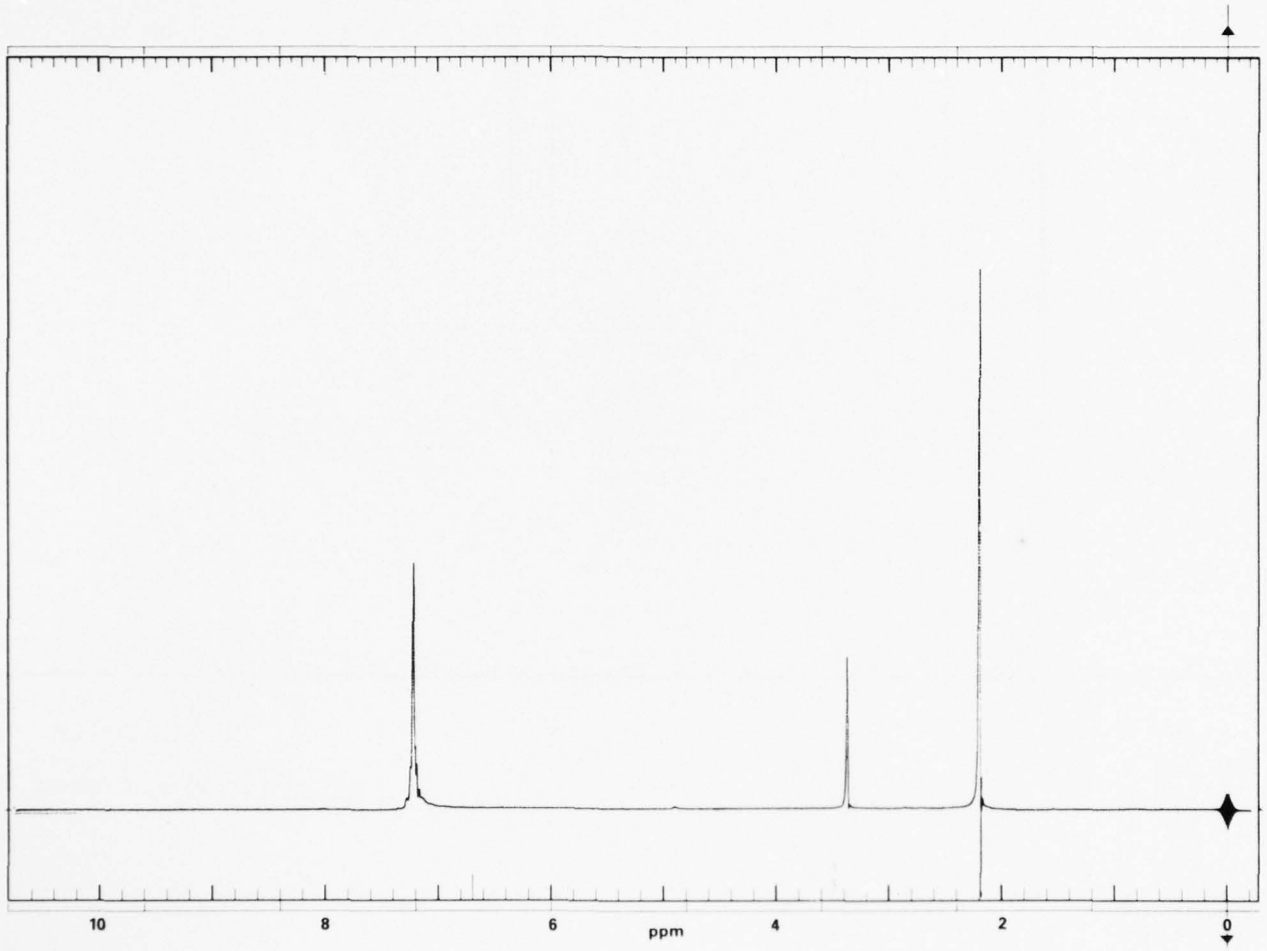
Assignments:

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

Source: Eastman 1793

Solvent: 25% Acetone ×

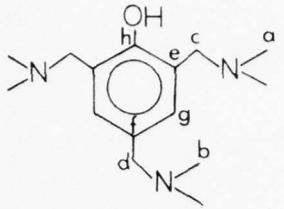




Spectrum 40 — *N,N*-Dimethylbenzylamine (Eastman 1793), solvent: $CDCl_3$

C41

2,4,6-Tris(dimethylaminomethyl)phenol

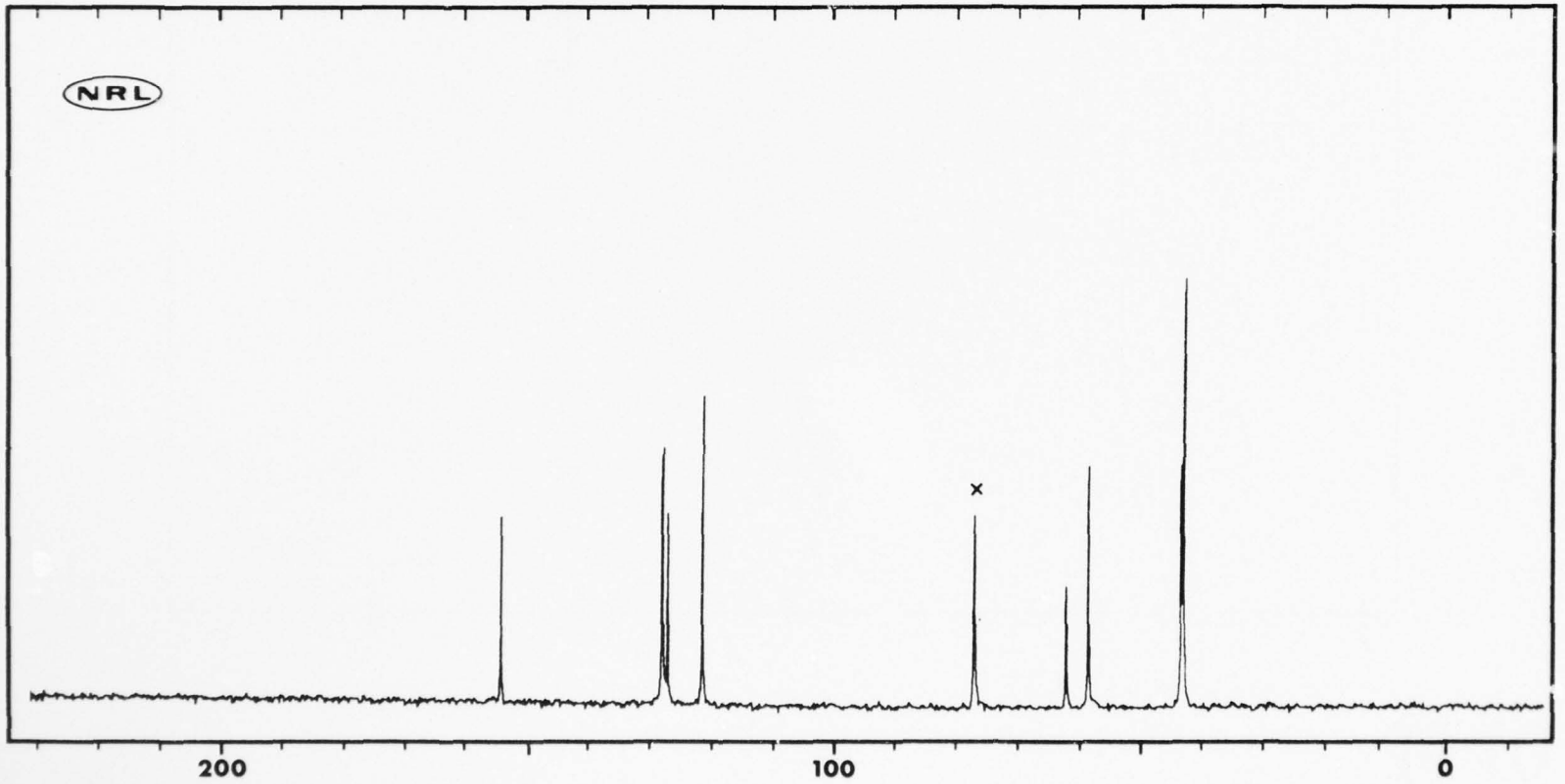


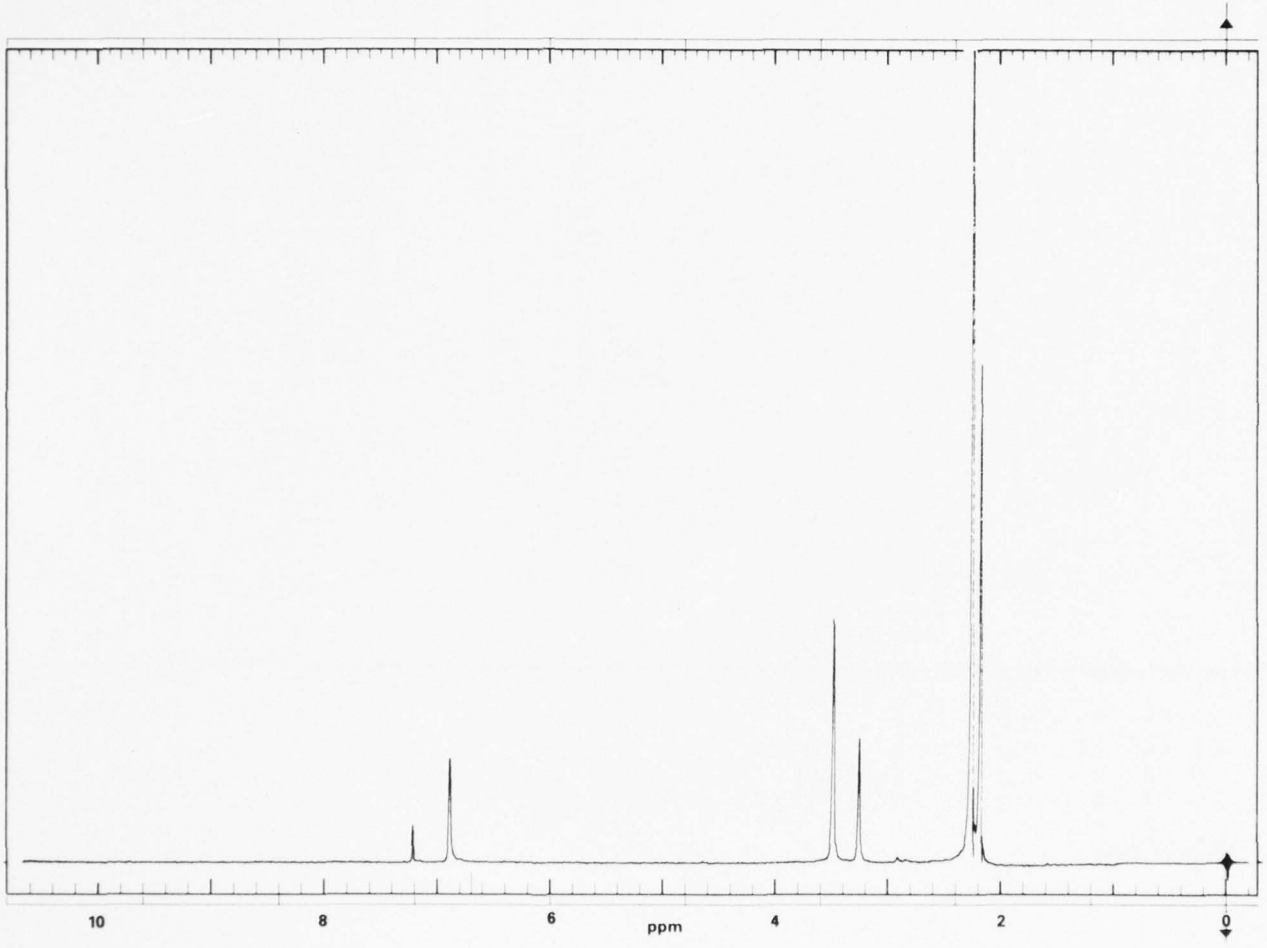
Assignments:

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

Source: Rohm and Haas DMP-30

Solvent: 25% $CHCl_3$ ×

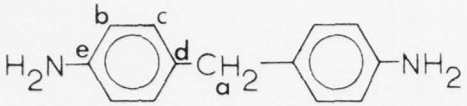




Spectrum 41 — 2,4,6-*tris*(dimethylaminomethyl) phenol (Rohm and Haas DMP-30); solvent: $CDCl_3$

C42

4,4'-Methylenedianiline

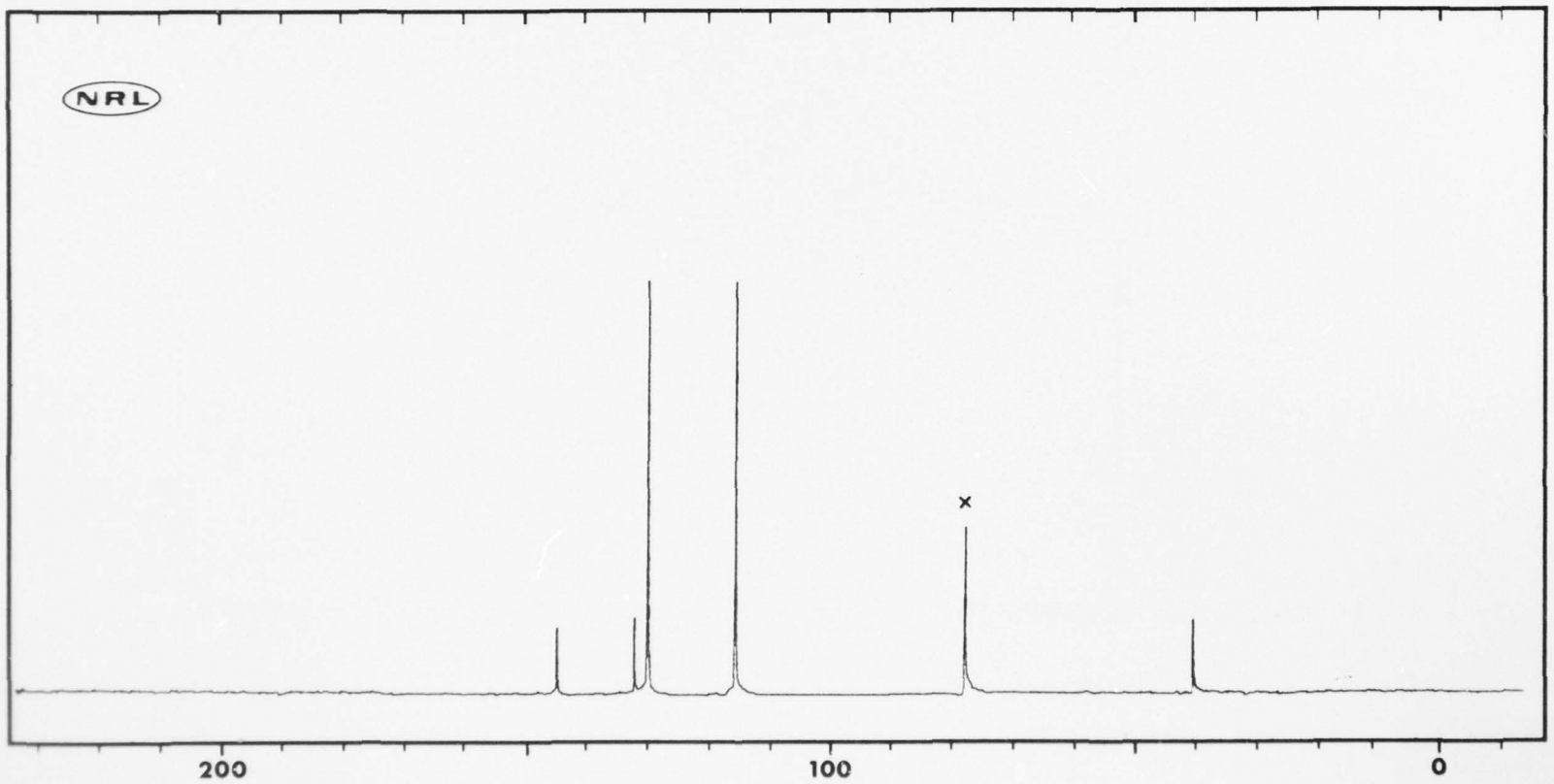


Assignments:

a	39.7
b	114.6
c	128.9
d	131.1
e	143.8

Source: Eastman P756

Solvent: 75% $CHCl_3$ ×



AD-A044 214 NAVAL RESEARCH LAB WASHINGTON D C F/G 11/9
CARBON-13 AND PROTON NMR SPECTRA FOR CHARACTERIZING THERMOSETTI--ETC(U)
JUN 77 C F PORANSKI, W B MONIZ, D L BIRKLE

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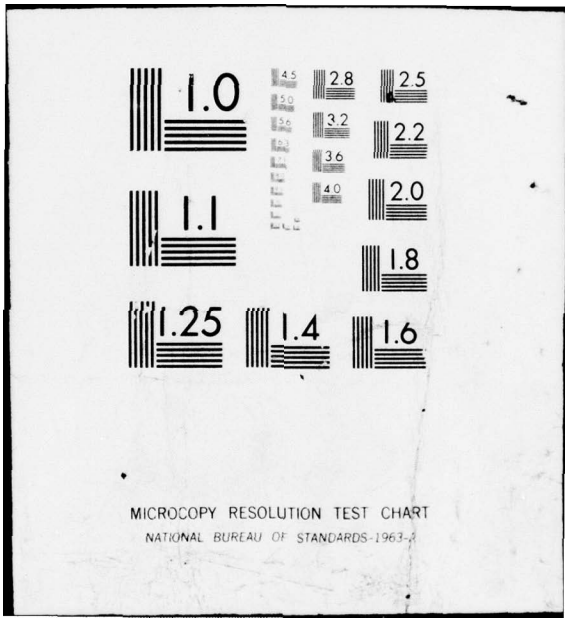
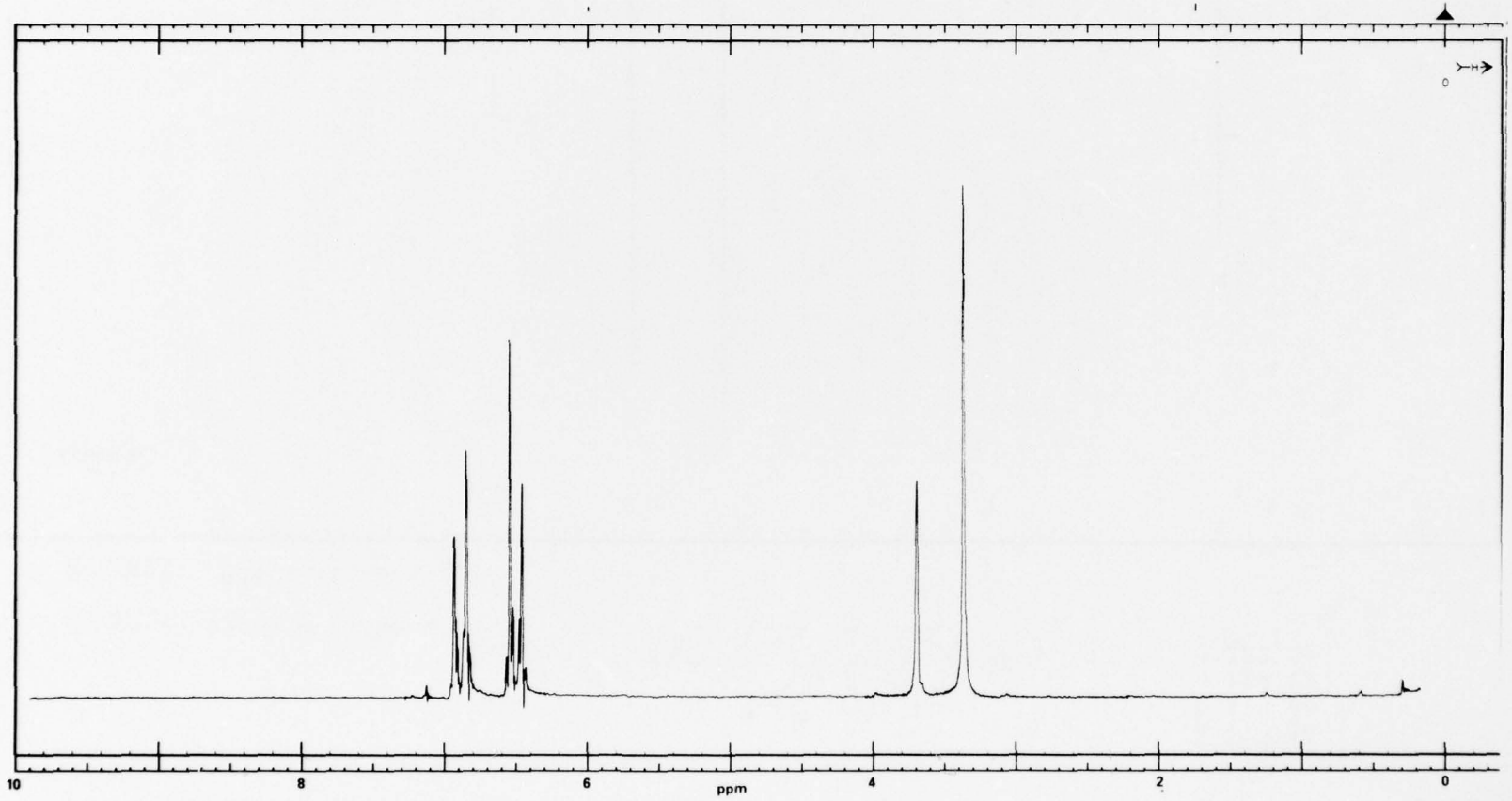
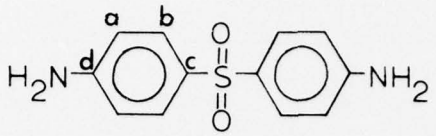

1.0
1.1
1.25
1.4
1.6
1.8
2.0
2.2
2.5
2.8
3.2
3.6
4.0
4.5
5.0
5.6
MICROCOPY RESOLUTION TEST CHART
NATIONAL BUREAU OF STANDARDS-1963-A

H42



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

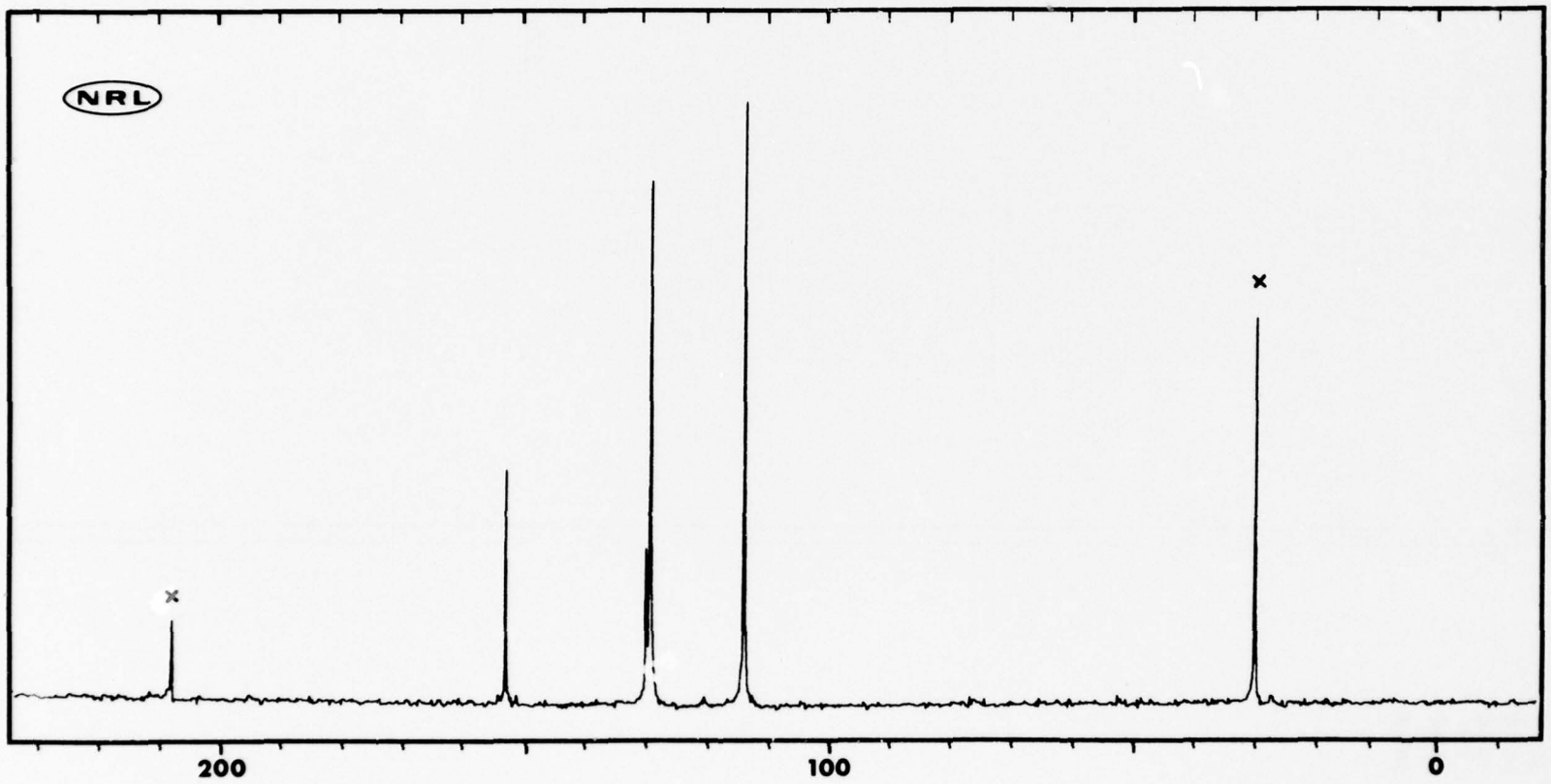
Bis(4-aminophenyl)sulfone



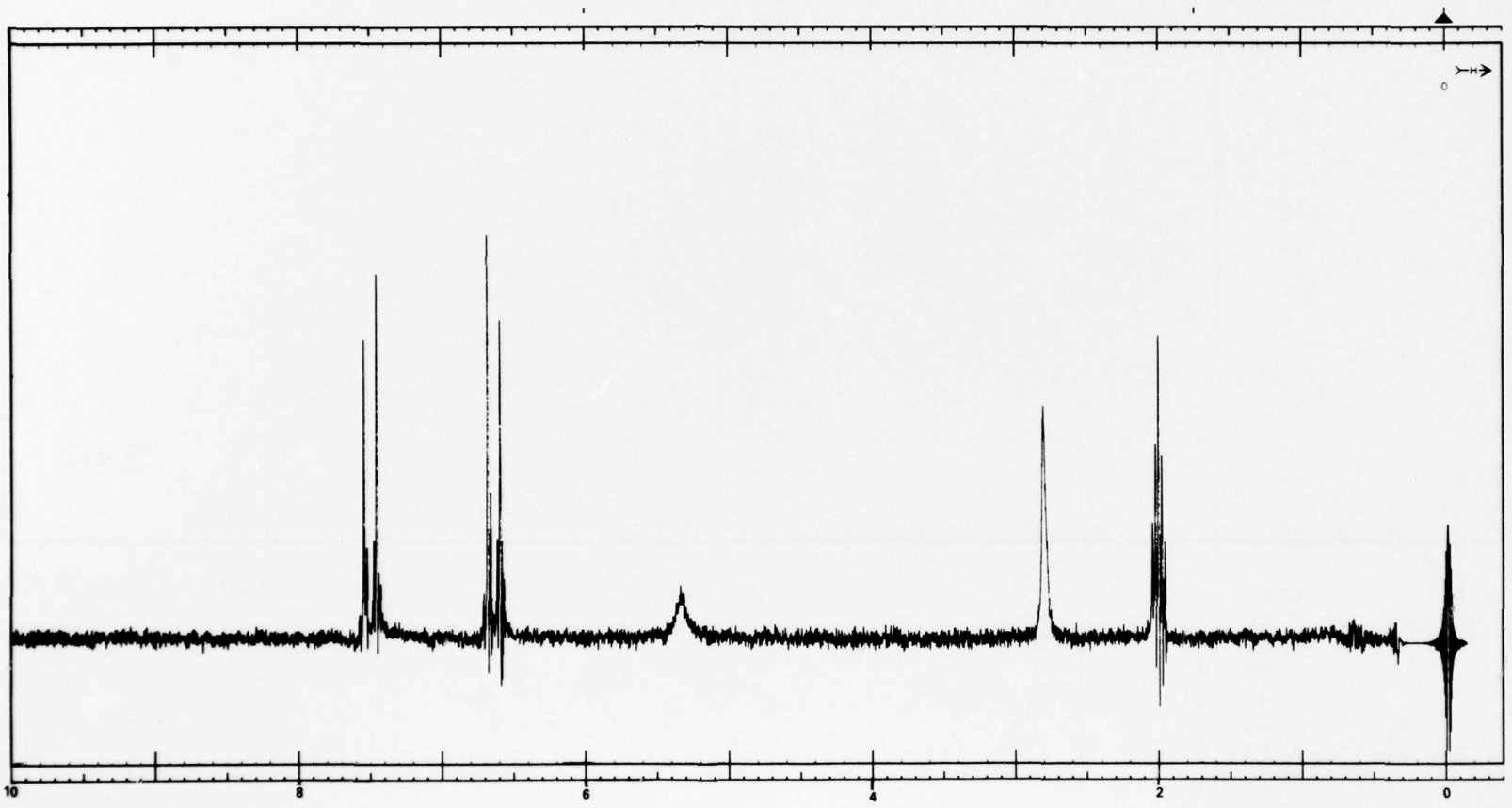
Assignments:
a 113.6d
b 128.9d
c 129.7s
d 152.3s

Source: Aldrich A7480-7

Solvent: 50% Acetone ×



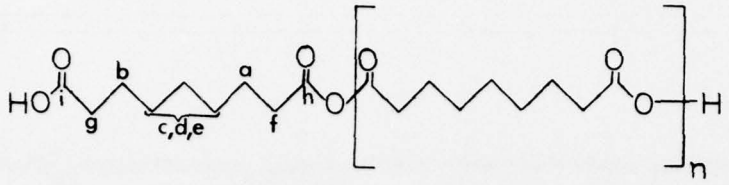
H43



Spectrum 43 — *bis*(4-aminophenyl)sulfone (Aldrich A7480-7); solvent: acetone-d_6

C44

Polyazelaic Polyanhydride

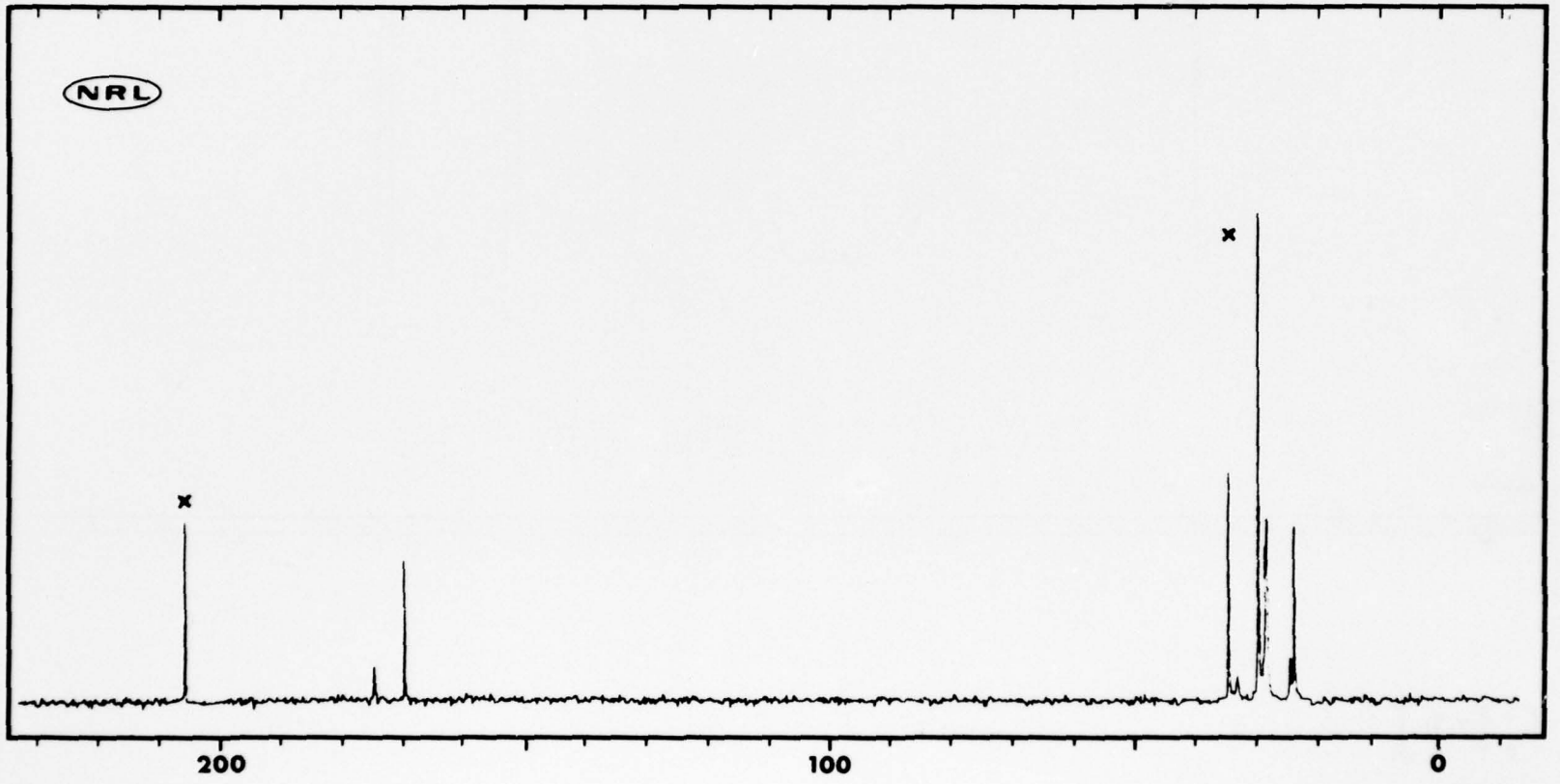


Assignments:

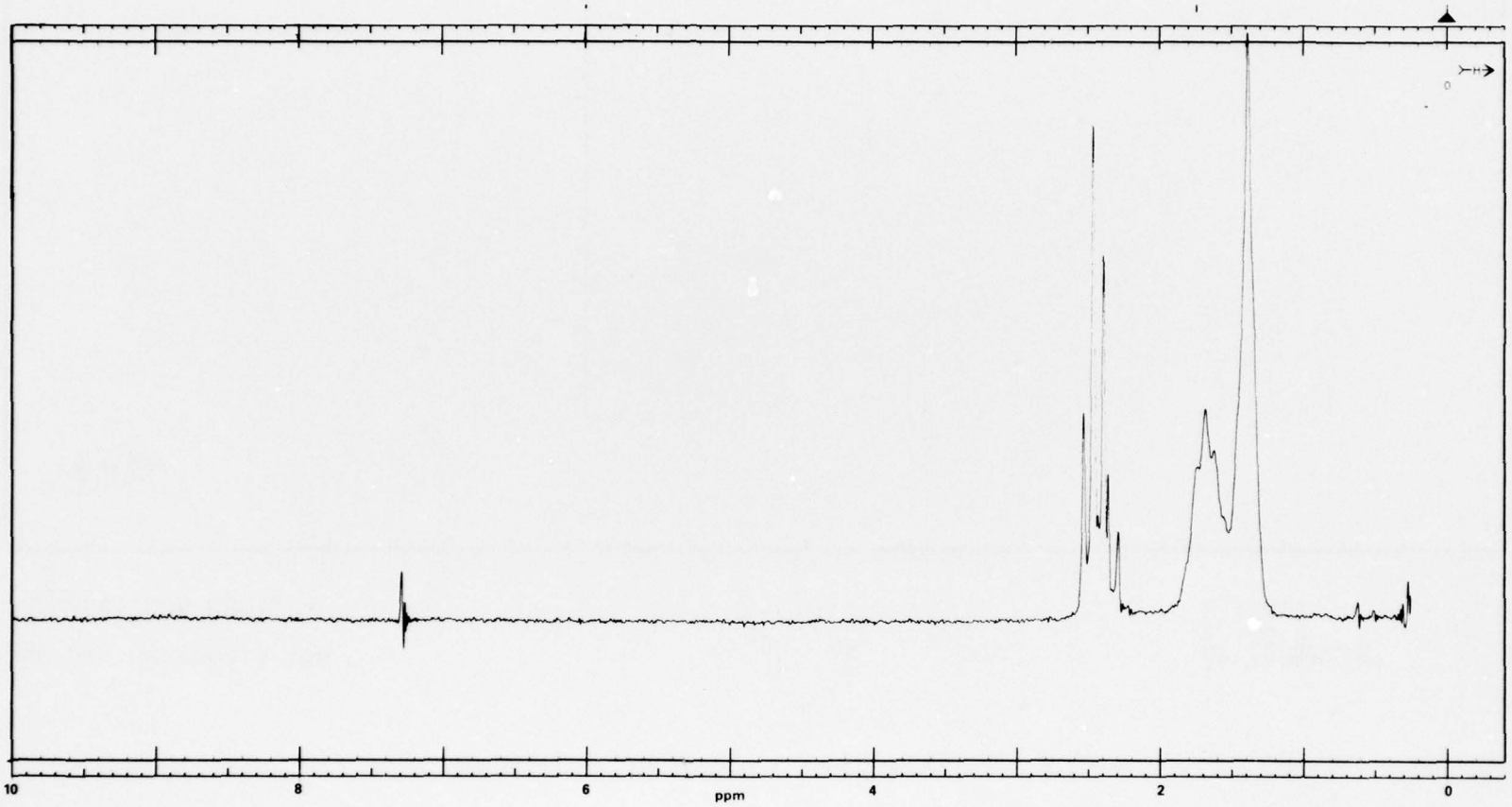
a	24.4	h	169.5
b	25.0	i	174.5
c	28.7		
d	29.0		
e	29.7		
f	33.7		
g	35.0		

Source: Emery 9872 PAPA

Solvent: 50% Acetone ×



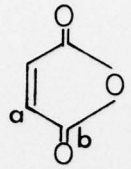
H44



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

C45

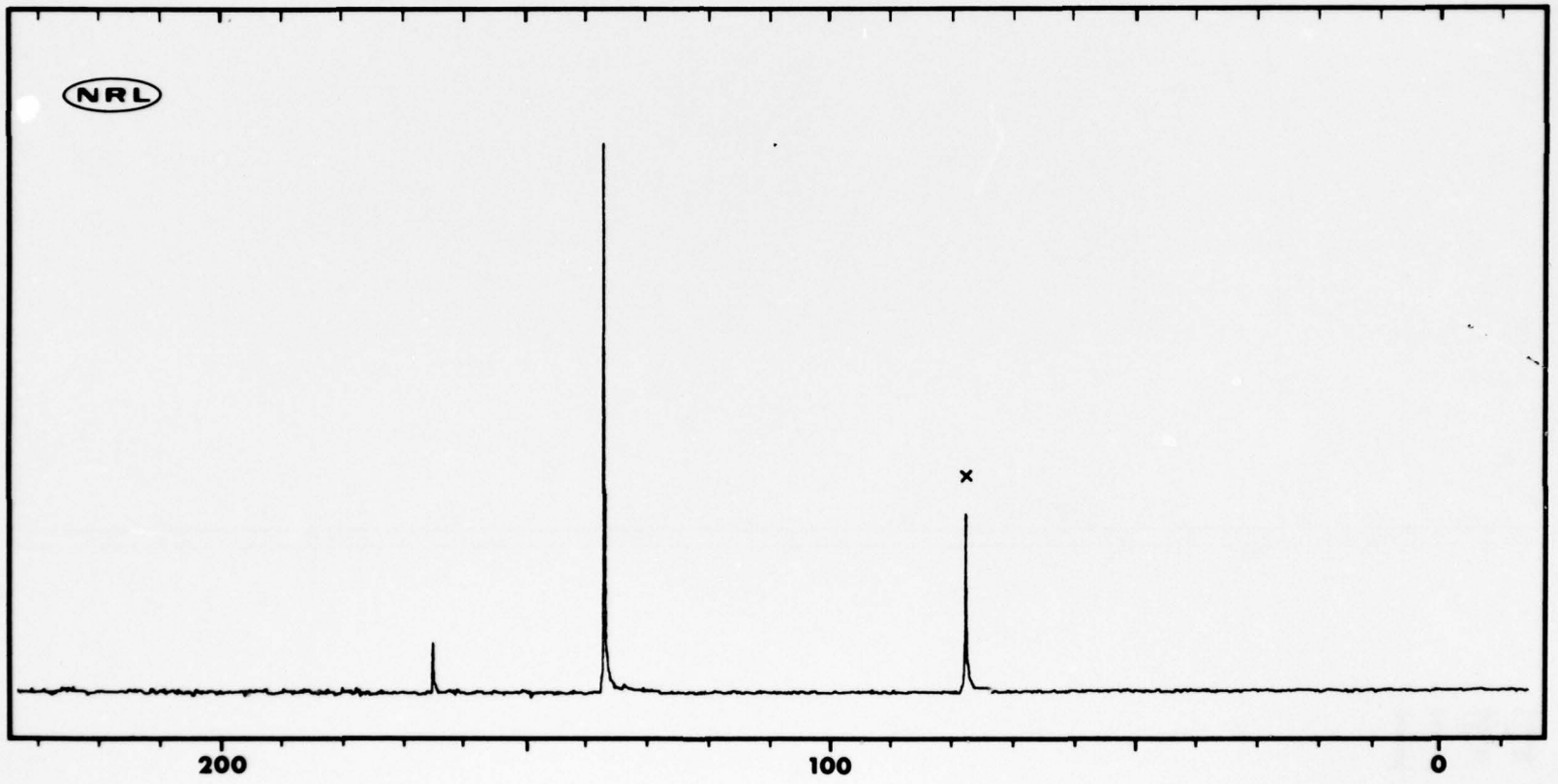
Maleic Anhydride

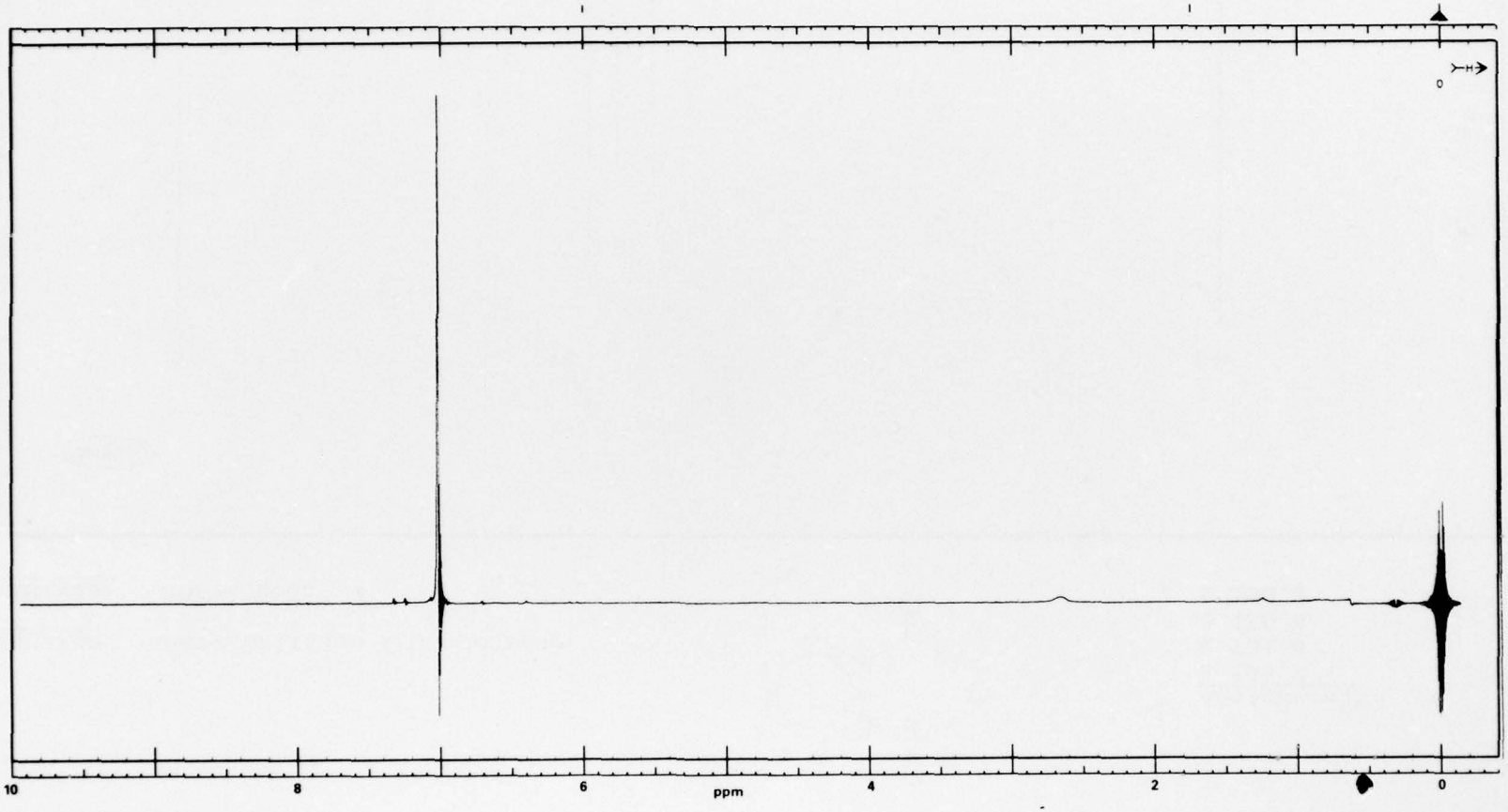


Source: Fisher A-168

Solvent: 50% $CHCl_3$ ×

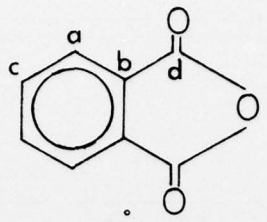
Assignments:
a 136.2
b 164.1





Spectrum 45 — Maleic anhydride (Fisher A-168); solvent: $CDC1_3$

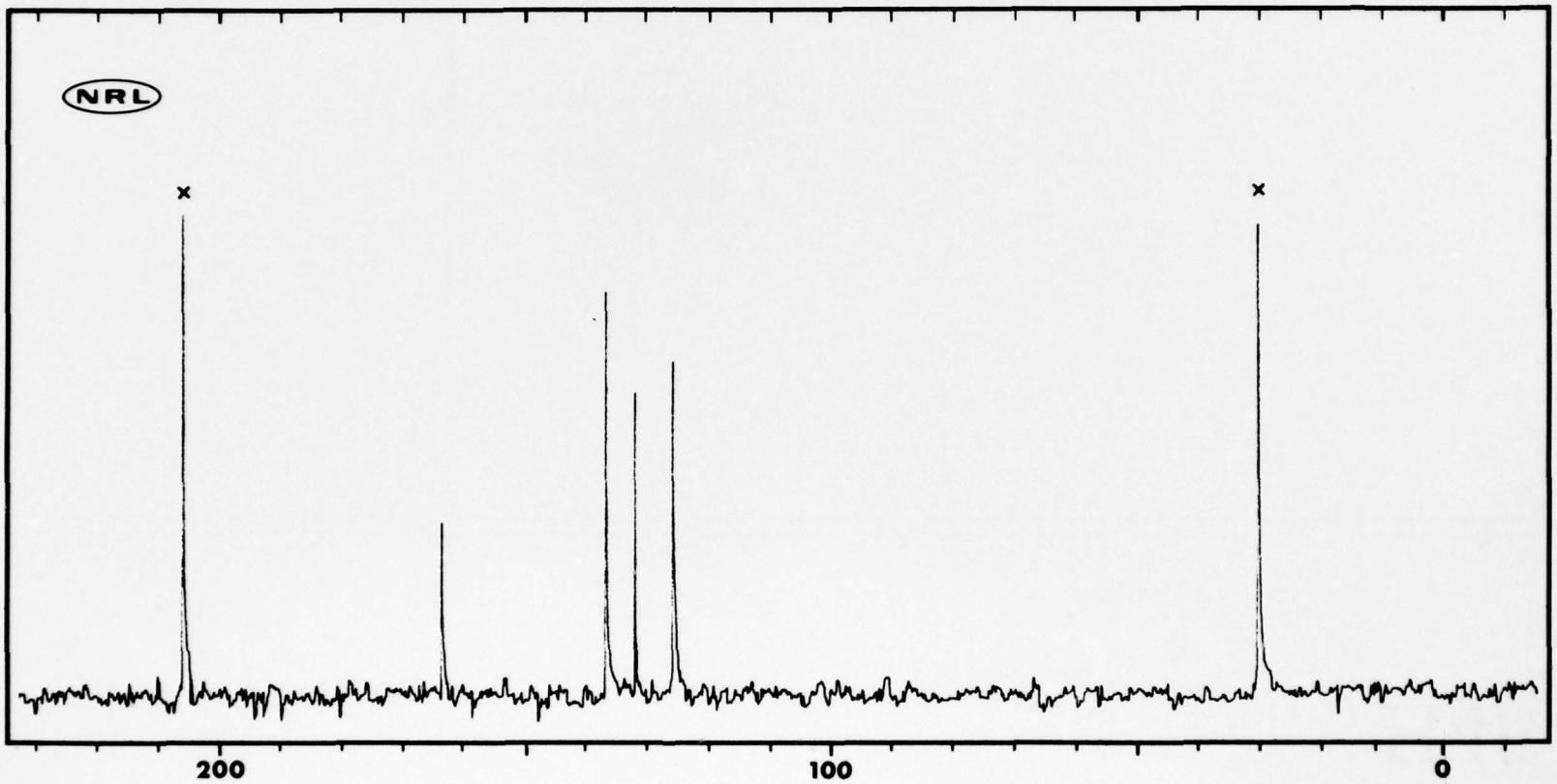
Phthalic Anhydride

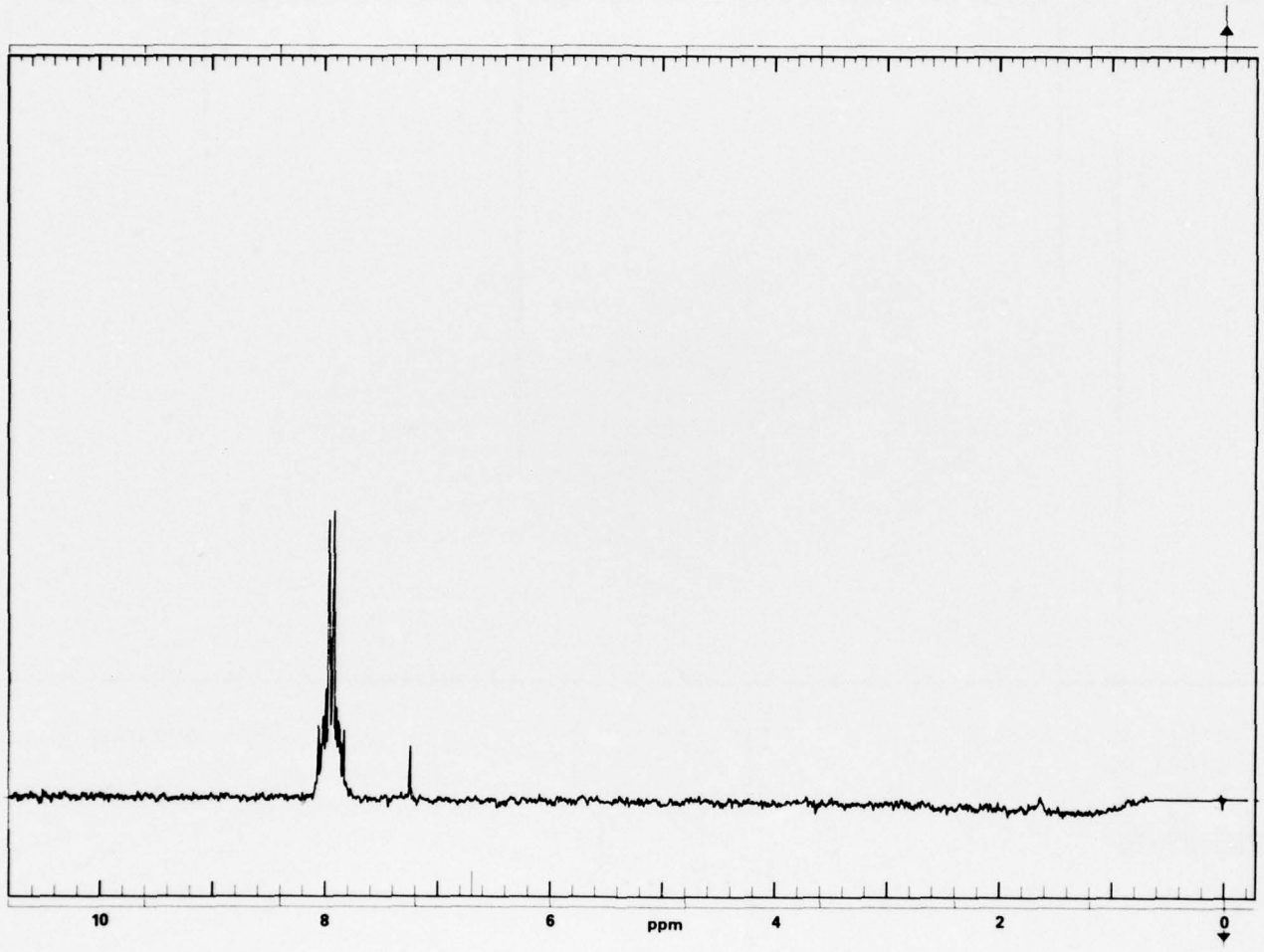


Assignments:
a 125.5
b 131.6
c 136.4
d 163.2

Source: Recrystallized from benzene

Solvent: 75% Acetone ×

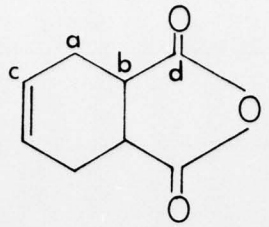




Spectrum 46 — Phthalic anhydride (recrystallized from benzene); solvent: $CDCl_3$

C47

Tetrahydrophthalic Anhydride

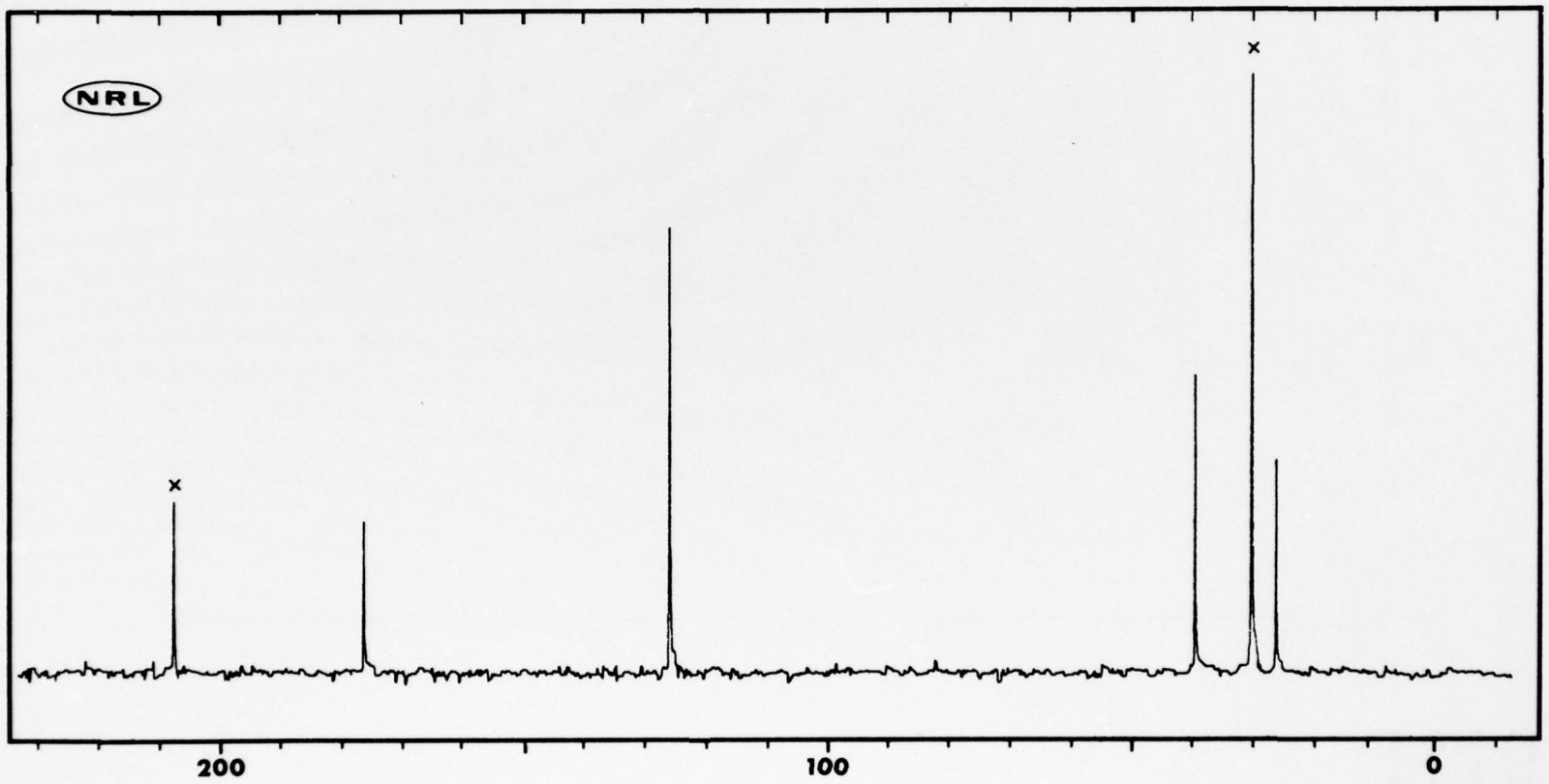


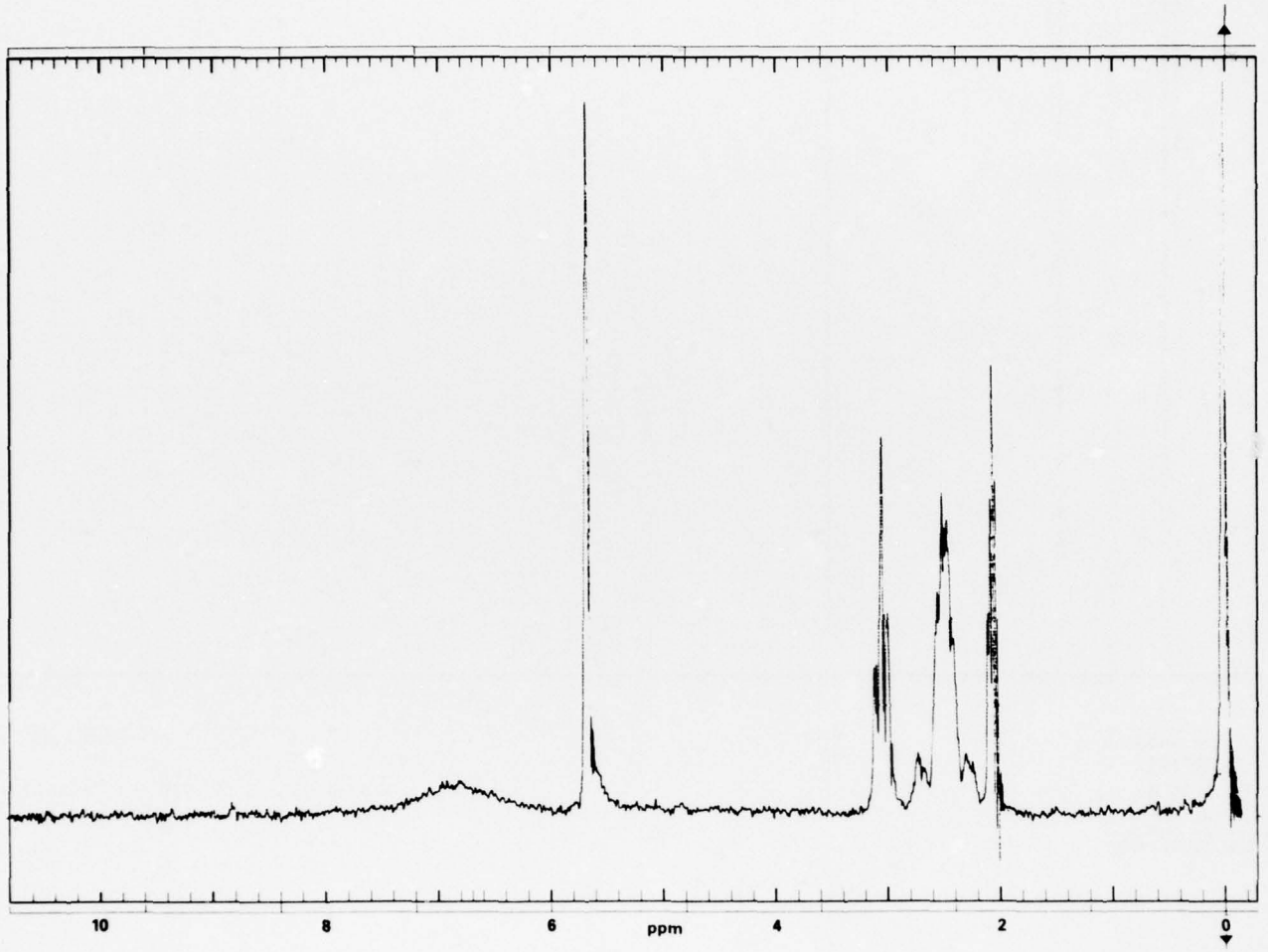
Source: Eastman #5724

Solvent: 50% Acetone ×

Assignments:

a	26.2
b	39.4
c	125.3
d	175.3

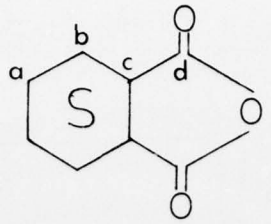




Spectrum 47 — Tetrahydrophthalic anhydride (Eastman 5724); solvent: acetone-d_6

C48

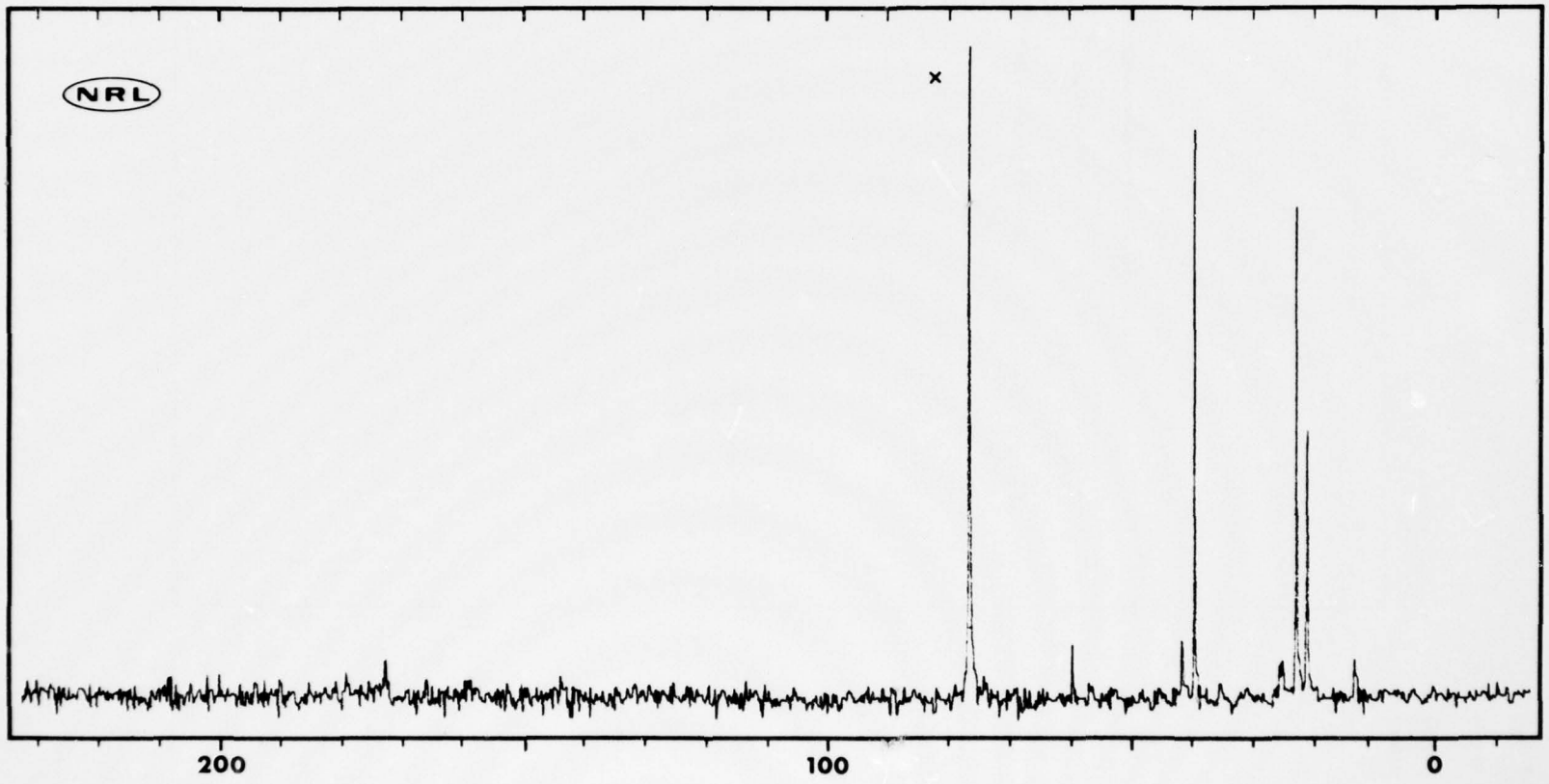
Hexahydrophthalic Anhydride

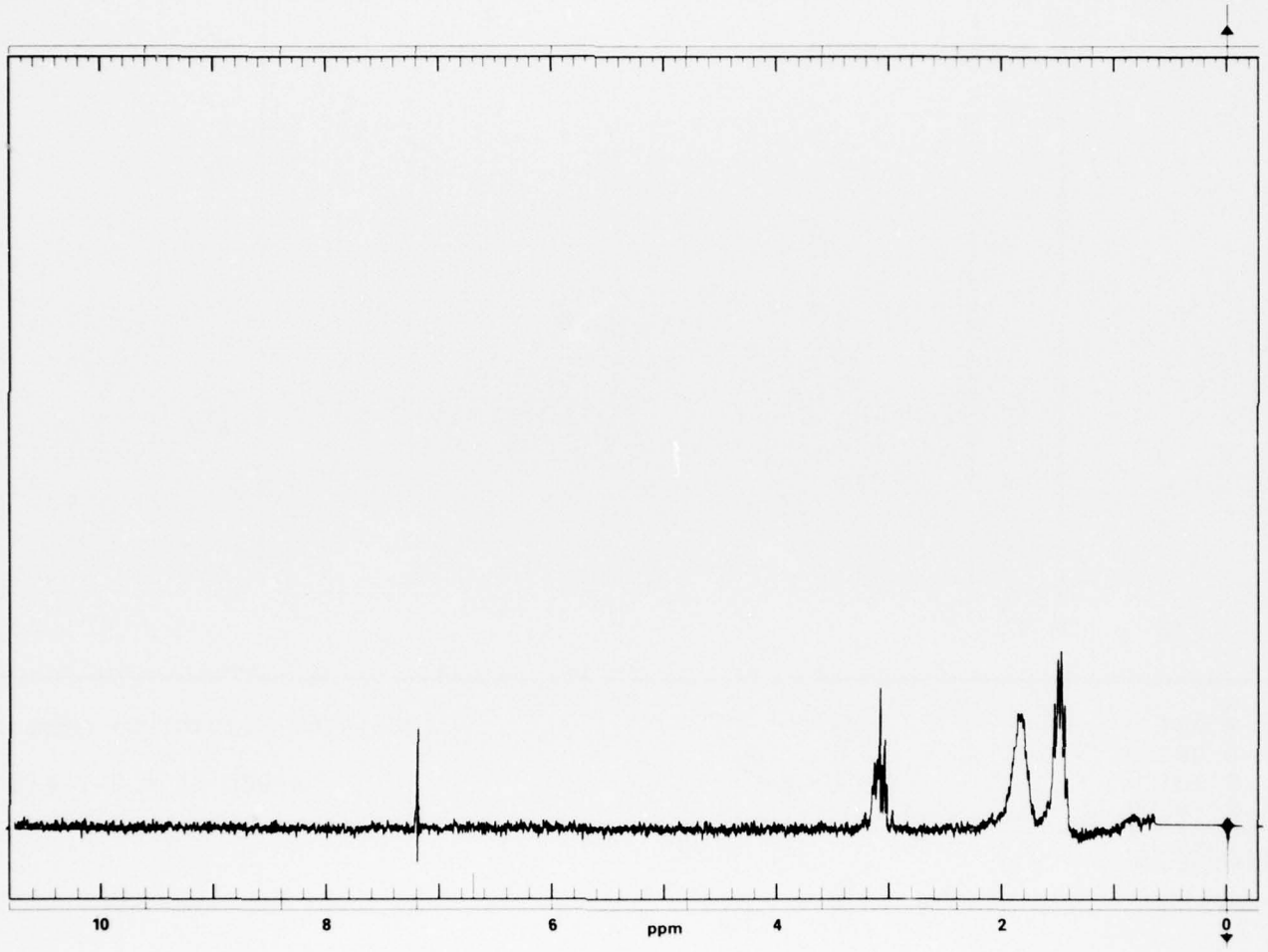


Assignments:
a 21.4
b 23.1
c 39.8
d 172.1

Source: Miller-Stephenson Chemical Co.

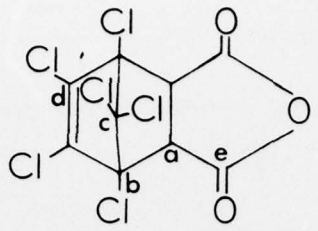
Solvent: 50% $CHCl_3$ ×





Spectrum 48 — Hexahydrophthalic anhydride (Miller-Stephenson Chemical Co.); solvent: $CDCl_3$

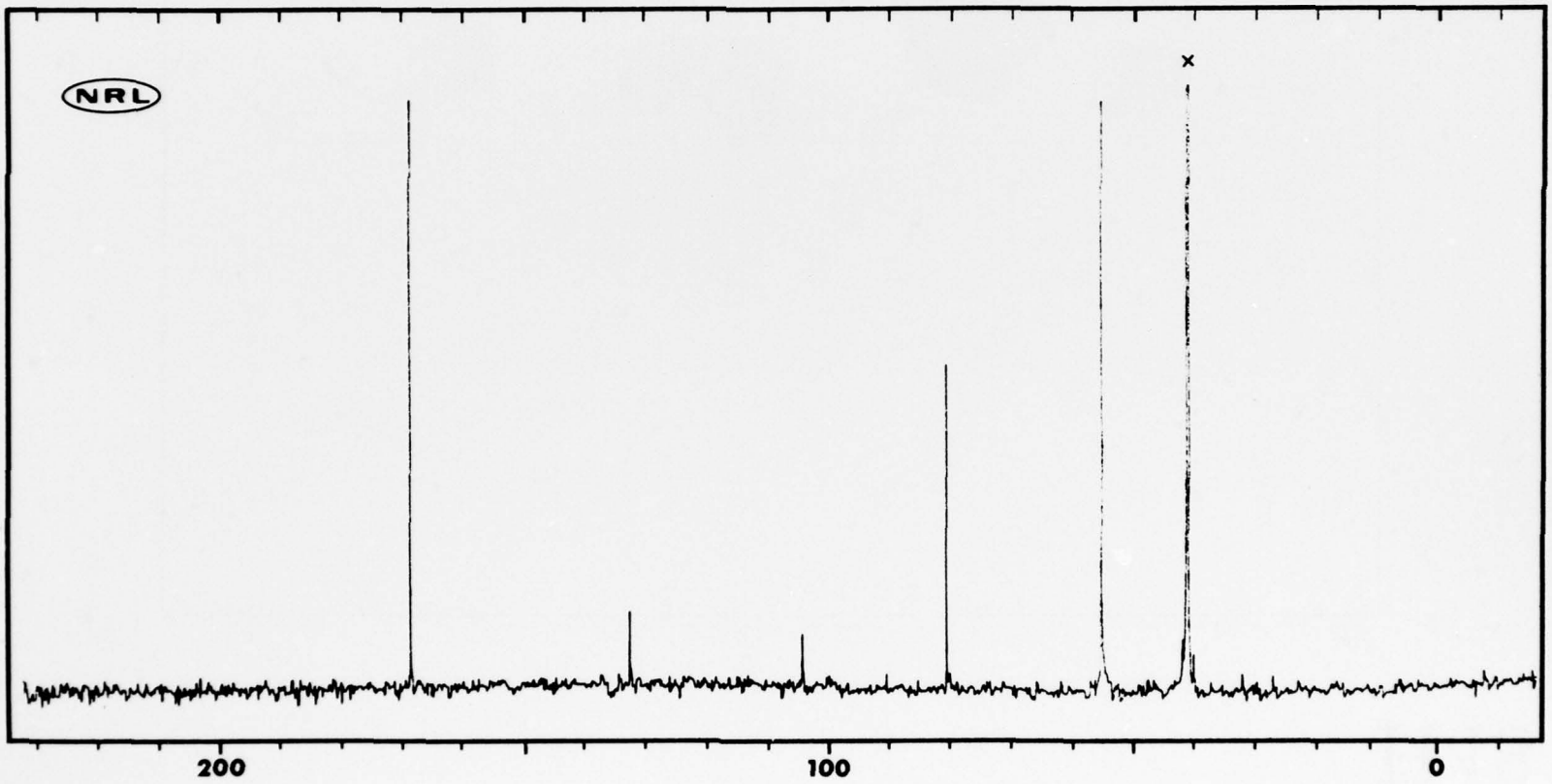
1,4,5,6,7,7-Hexachloro-5-Norbornene-2,3-Dicarboxylic Anhydride

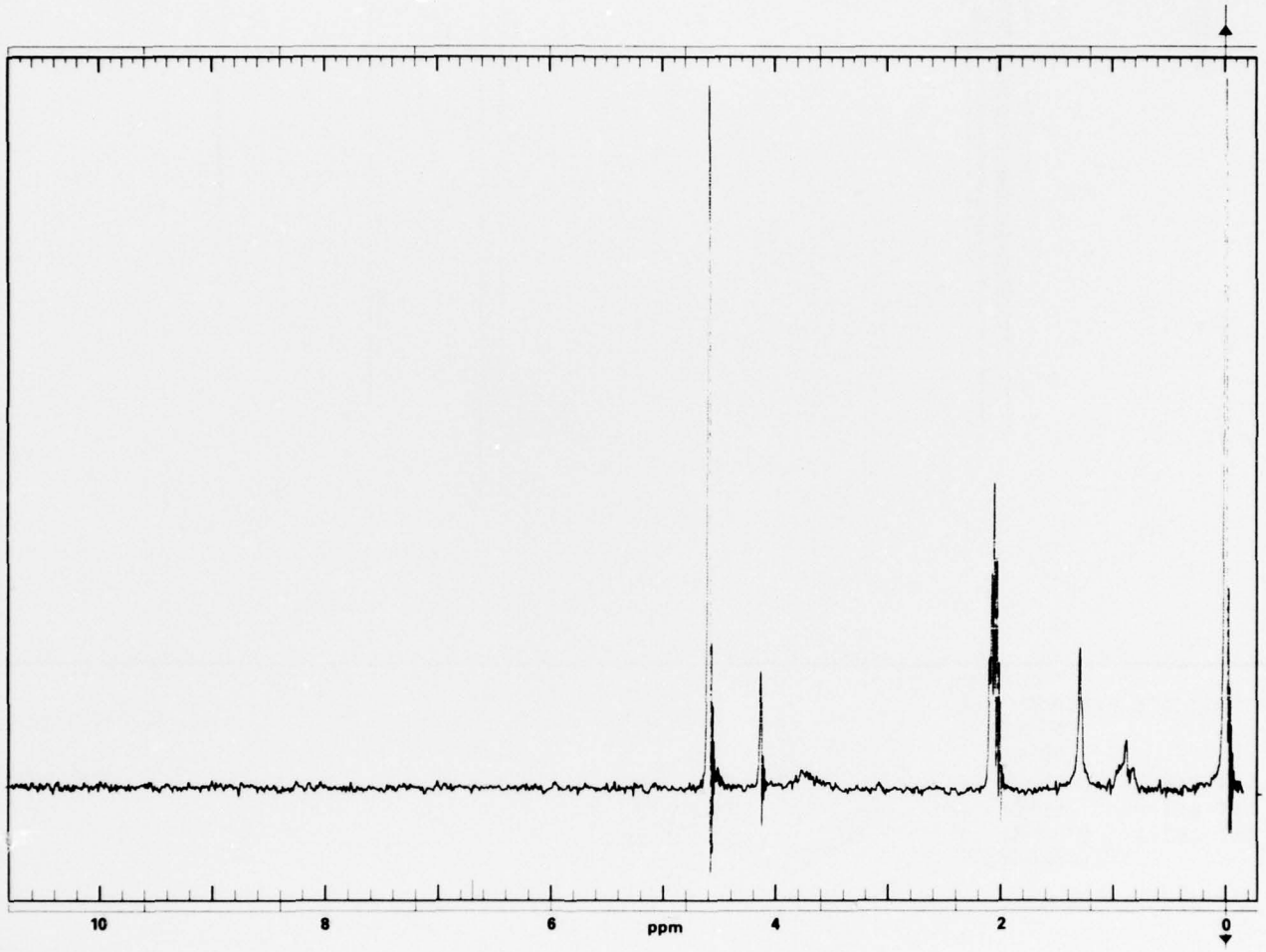


Assignments:
a 55.9
b 81.2
c 104.8
d 133.0
e 169.2

Source: Aldrich # 10,326-8

Solvent: DMSO (saturated solution) ×

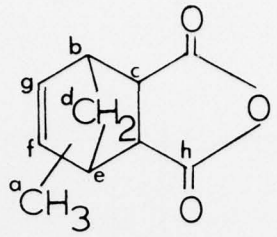




Spectrum 49 — 1,4,5,6,7,7-Hexachloro-5-norbornene-2,3-dicarboxylic anhydride (Aldrich 10,326-8); solvent: acetone-d_6

C50

Methyl-4-endo-methylene Tetrahydrophthalic Anhydride



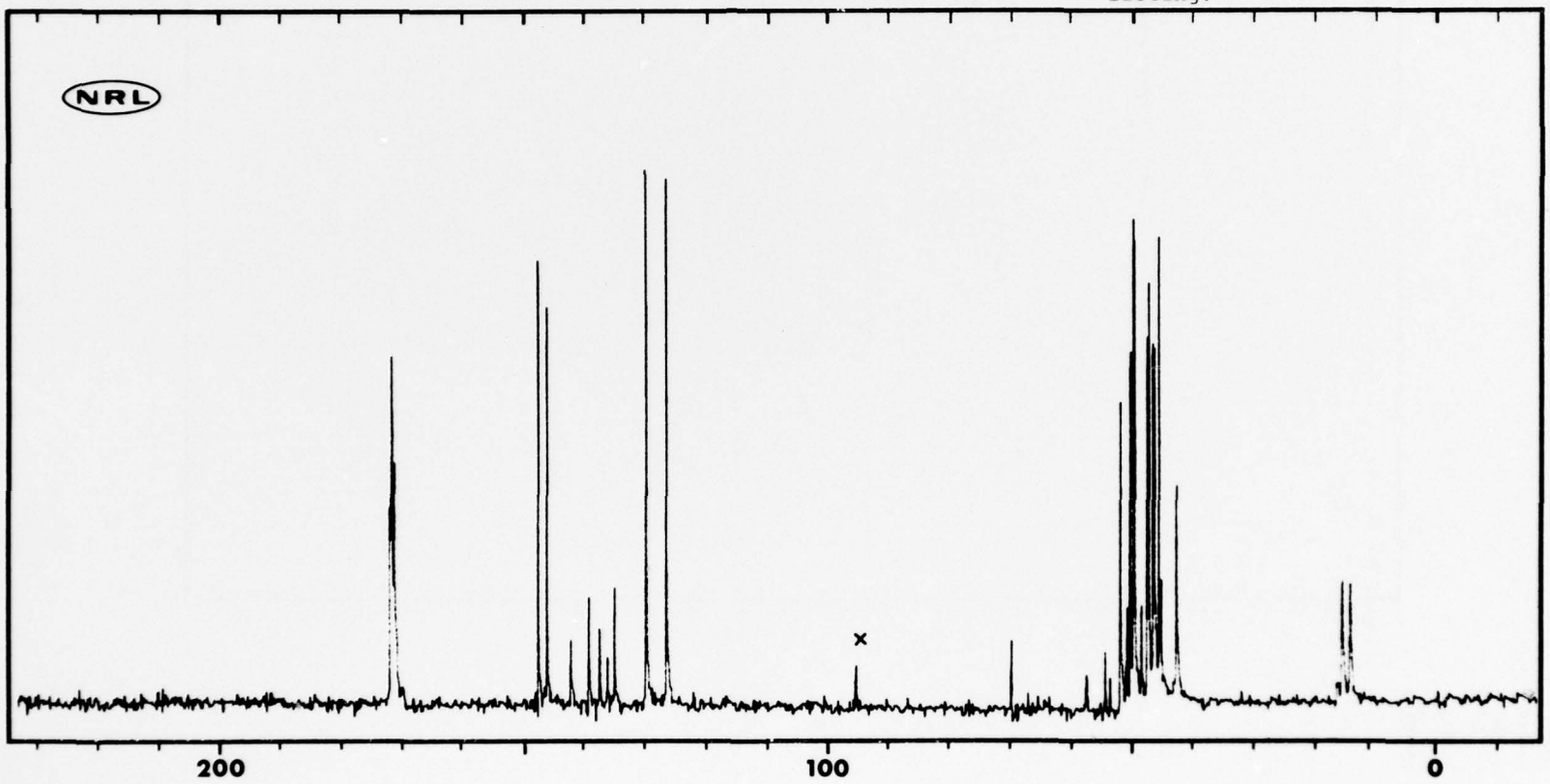
Source: CIBA 906

Solvent: 10% CCl_4 ×

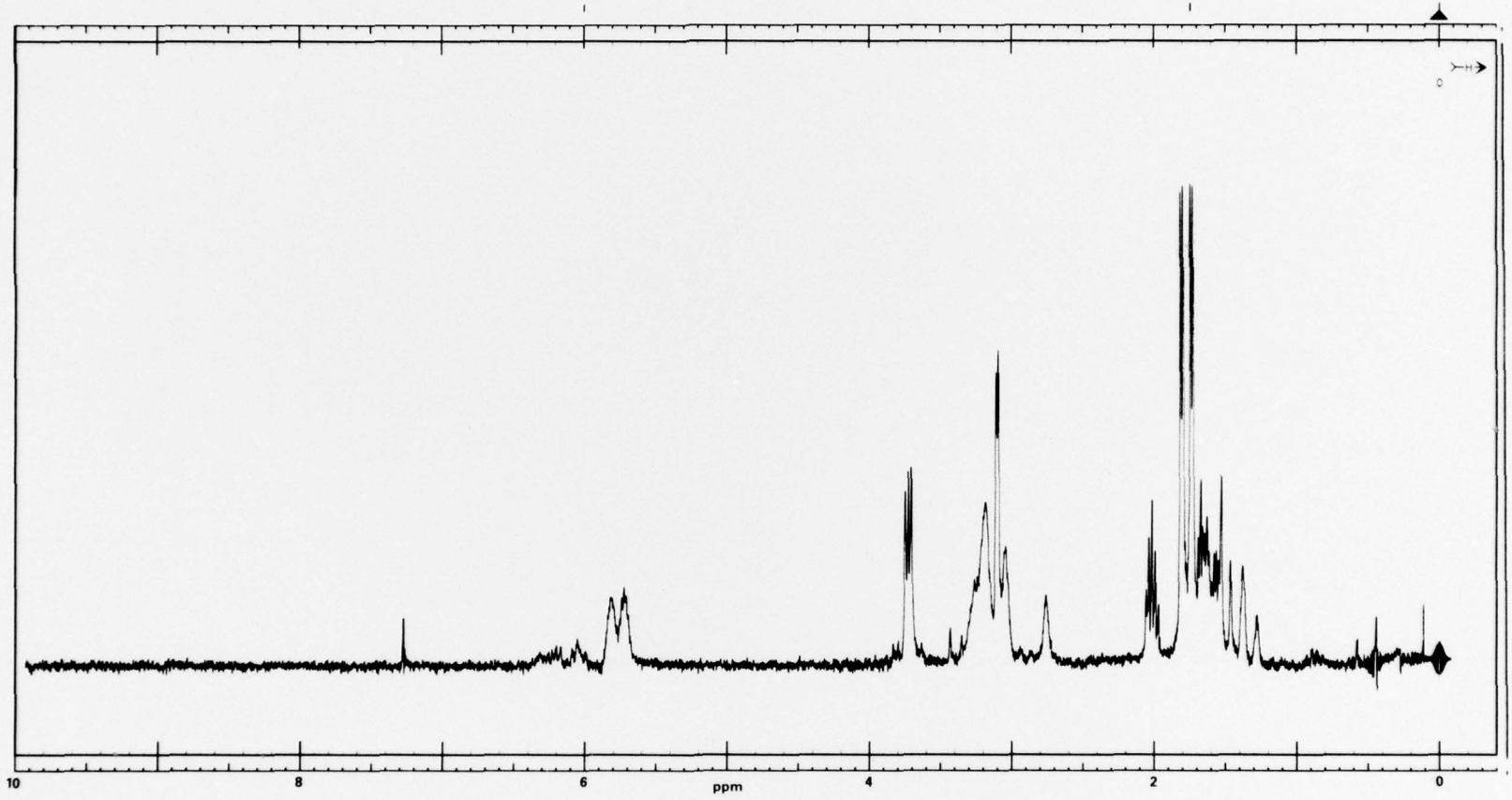
Assignments:

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.



H50



Spectrum 50 — Methyl-4-*endo*-methylene tetrahydrophthalic anhydride (CIBA 906); solvent: acetone-d_6