IDENTIFICATION OF CHLORINATED BIPHENYLS IN BIOLOGICAL RESIDUES

Object

This method describes a procedure for the identification of chlorinated biphenyls. The samples to be analyzed normally are the residues from biological extractions but the method is appropriate to other systems.

Principle

The components of the residue in an extract suspected of containing chlorinated biphenyls are first separated by gas chromatography. The separated materials are then identified by rapid scan mass spectrometry.

Instrumental Details

The gas chromatographic separations are achieved with a F&M 5751-B chromatograph using a flame ionization detector. The 1/8 inch stainless steel columns contain SE-54 packing on 80-100 mesh Chromosorb W. The injection and detector blocks are maintained at 280°C. The column temperature is programmed linearly from 170° to 280°C at 4° per minute. Programming the temperature sharpens the peaks with longer retention times and facilitates their identification via mass spectrometry. The helium flow rate through the column is 30 ml per minute. The column effluent is split 1:10 between the flame detector and the mass spectrometer.

A 0.010 inch i.d. stainless steel capillary connects the chromatograph outlet to a Biemann type helium separator. This line is heated to 300°C and the separator temperature is 280°C. The helium separator is connected to the ion source of an Atlas CH 4B mass spectrometer. The electron impact source is operated at 70 ev and 60 μ amp filament emission. The source temperature is 280°C. The ion source also contains a total ionization monitor whose analog output is proportional to the concentration of ionizable material in the source. This recorded signal produces a trace which resembles the original chromatogram.

Procedure

Residual solvent is evaporated to concentrate the residue to about 0.01%. A 20-30 μ liter aliquot of this concentrate is injected into the chromatograph and the temperature program started. The solvent quickly passes through the column and is pumped away through the glass frit in the Biemann separator. A valve between the separator and the mass spectrometer is then opened so that column effluent can pass into the ion source. A mass spectrum is scanned each time a peak is observed on the total ion monitor trace. After each component of the extract has been separated and scanned, the spectra are examined and the components identified.

Identification of Chlorinated Biphenyls

The mass spectra of chlorinated biphenyls are characterized by relatively abundant molecular ion peaks. Since naturally occurring chlorine consists of two isotopes, Cl^{35} and Cl^{37}, in the ratio of 3:1, the molecular ion peak actually is a multiplet of peaks. The distribution and relative intensities of the peaks within this multiplet allow the identification of the number of chlorine atoms per molecule. Using this information and

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the measured molecular weight, the empirical formula can be determined readily. These spectra can easily be distinguished from the spectra of pesticides including chlorine containing products.

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db

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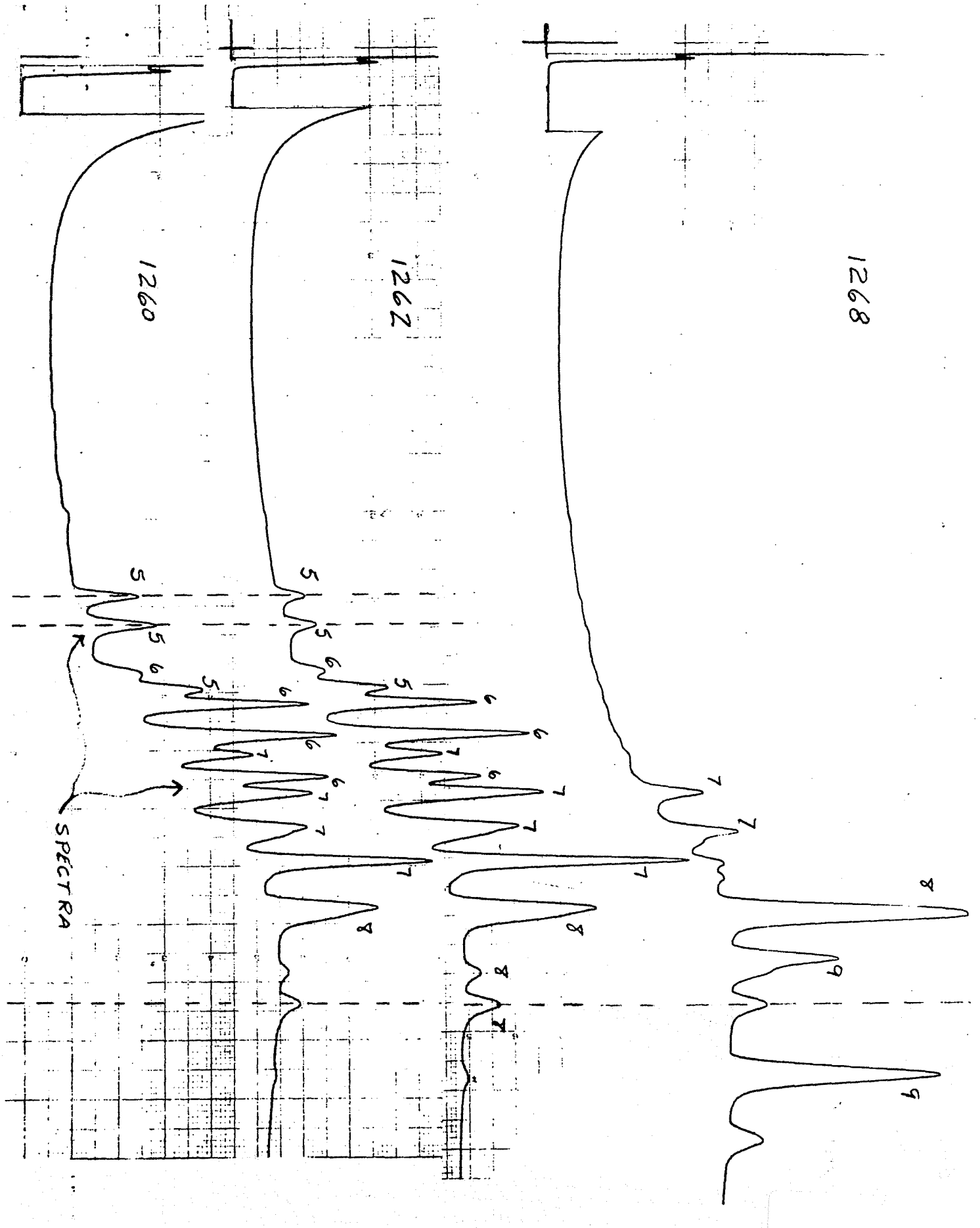
1260
1262
1268
SPECTRA
DSW 152712

GC FROM HS/GC

SE-54 PACKED COLUMN GC

NUMBERS ARE NUMBER OF CHLORINES PER MOLECULE

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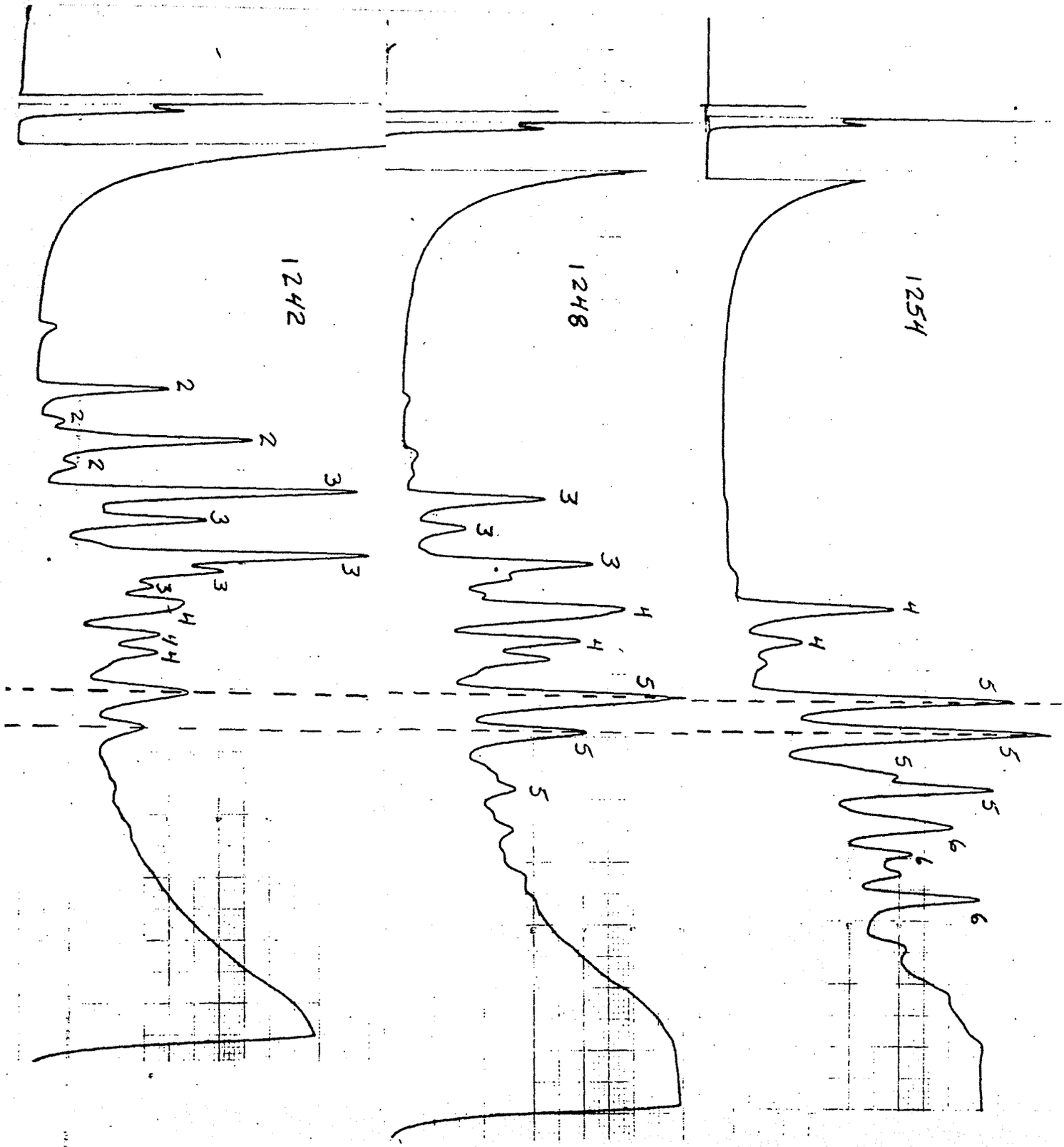

DSW 152713

GC FROM MS/GC

SE-54 PACKED COLUMN GC

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K&E 10 X 10 TO THE CENTIMETER 46 1512
18 X 25 CM. MADE IN U.S.A.
KEUFFEL & ESSER CO.

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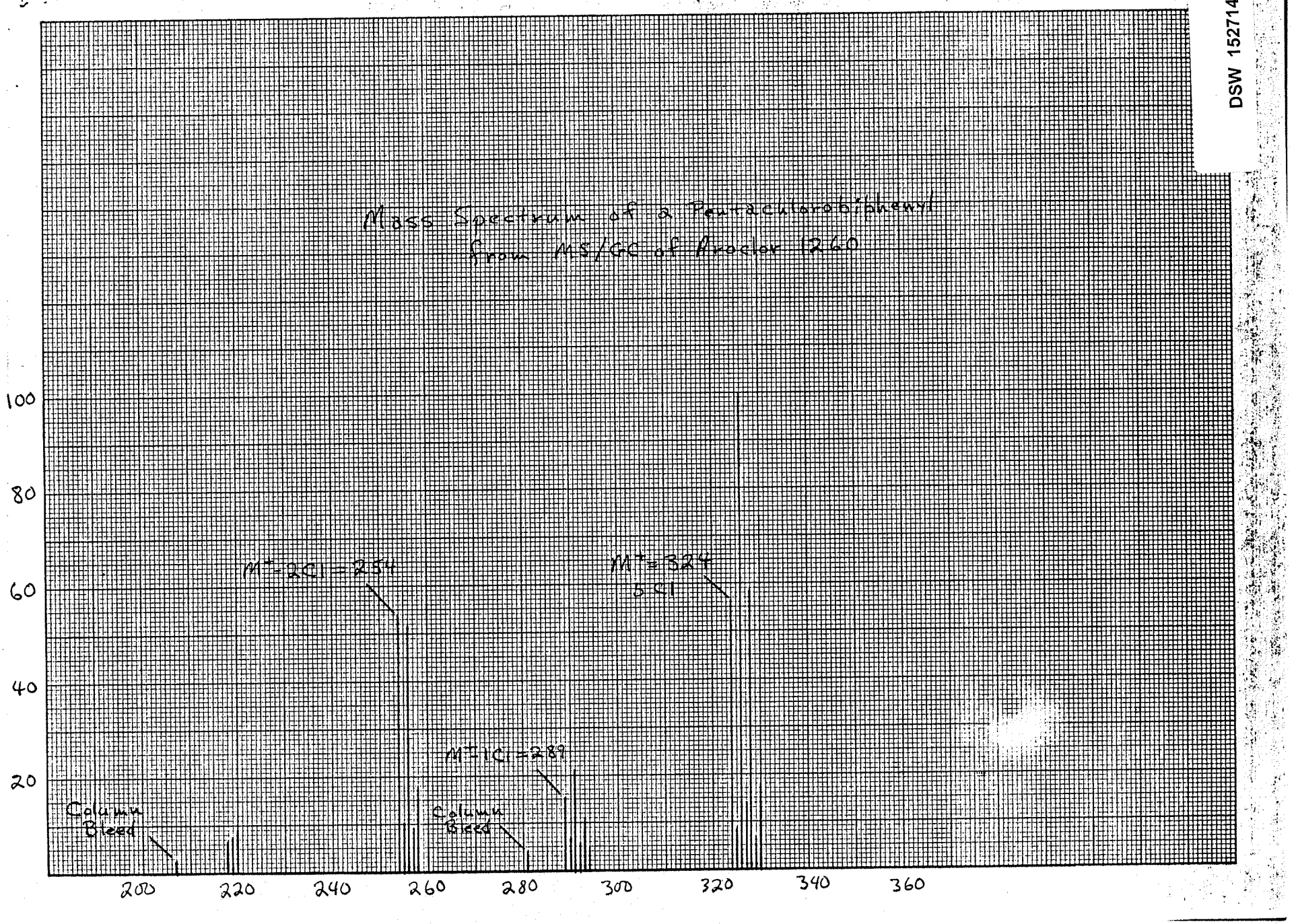

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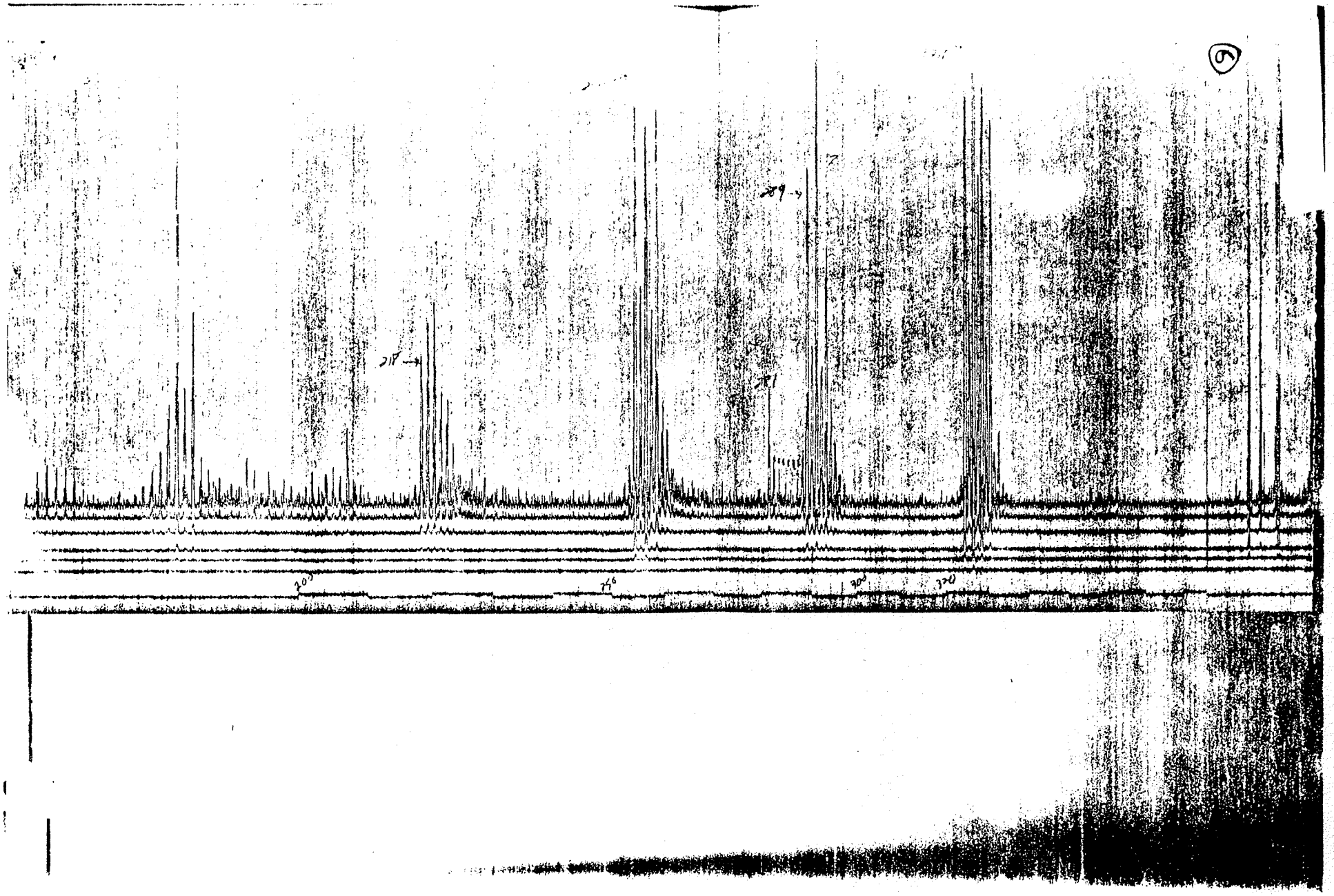

AROCLOR 1260
LOT AK 3

DSW 152716

K+E 10 X 10 TO THE CENTIMETER 46 1512
18 X 25 C.M. MADE IN U.S.A.
KEUFFEL & ESSER CO.

DSW 152717

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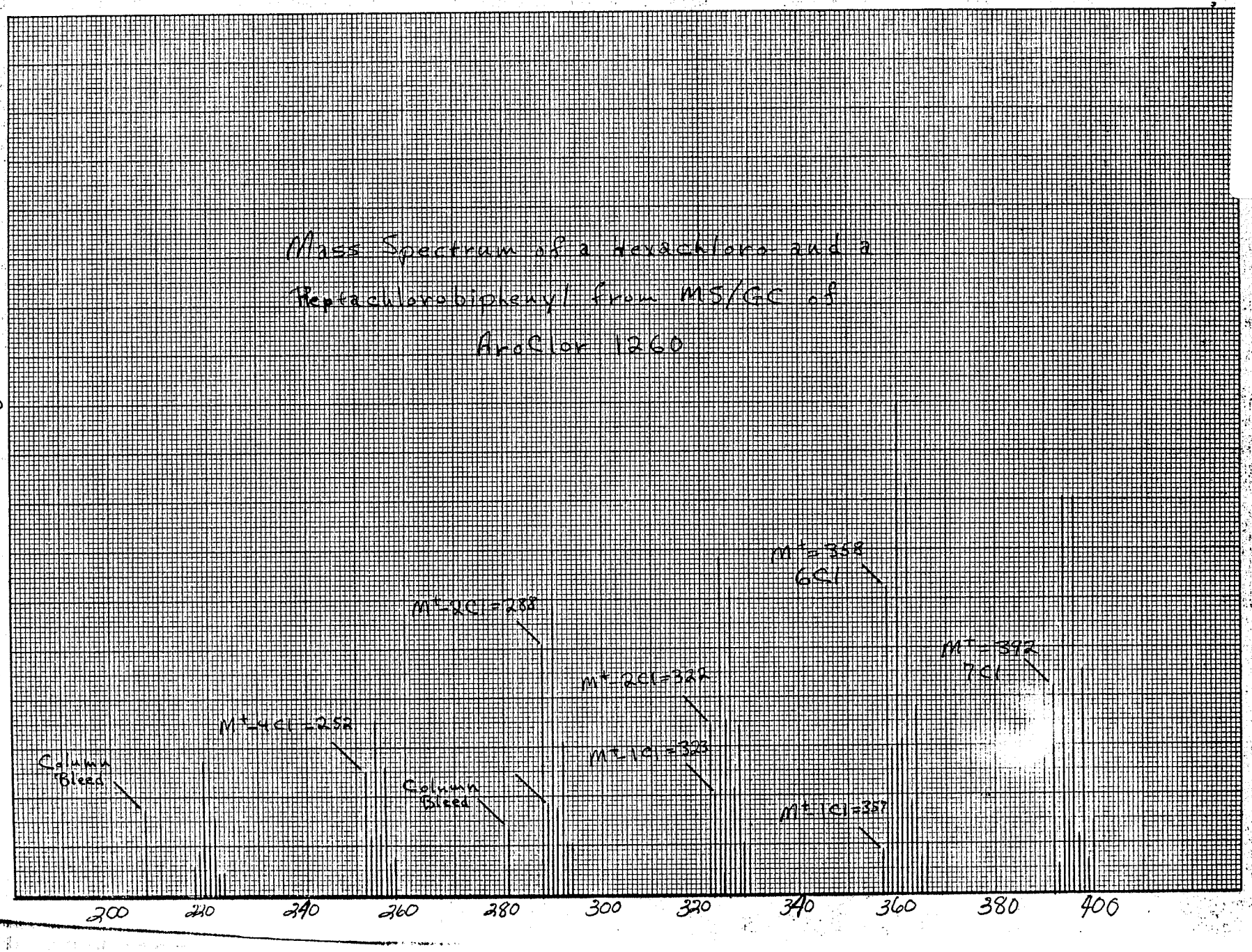

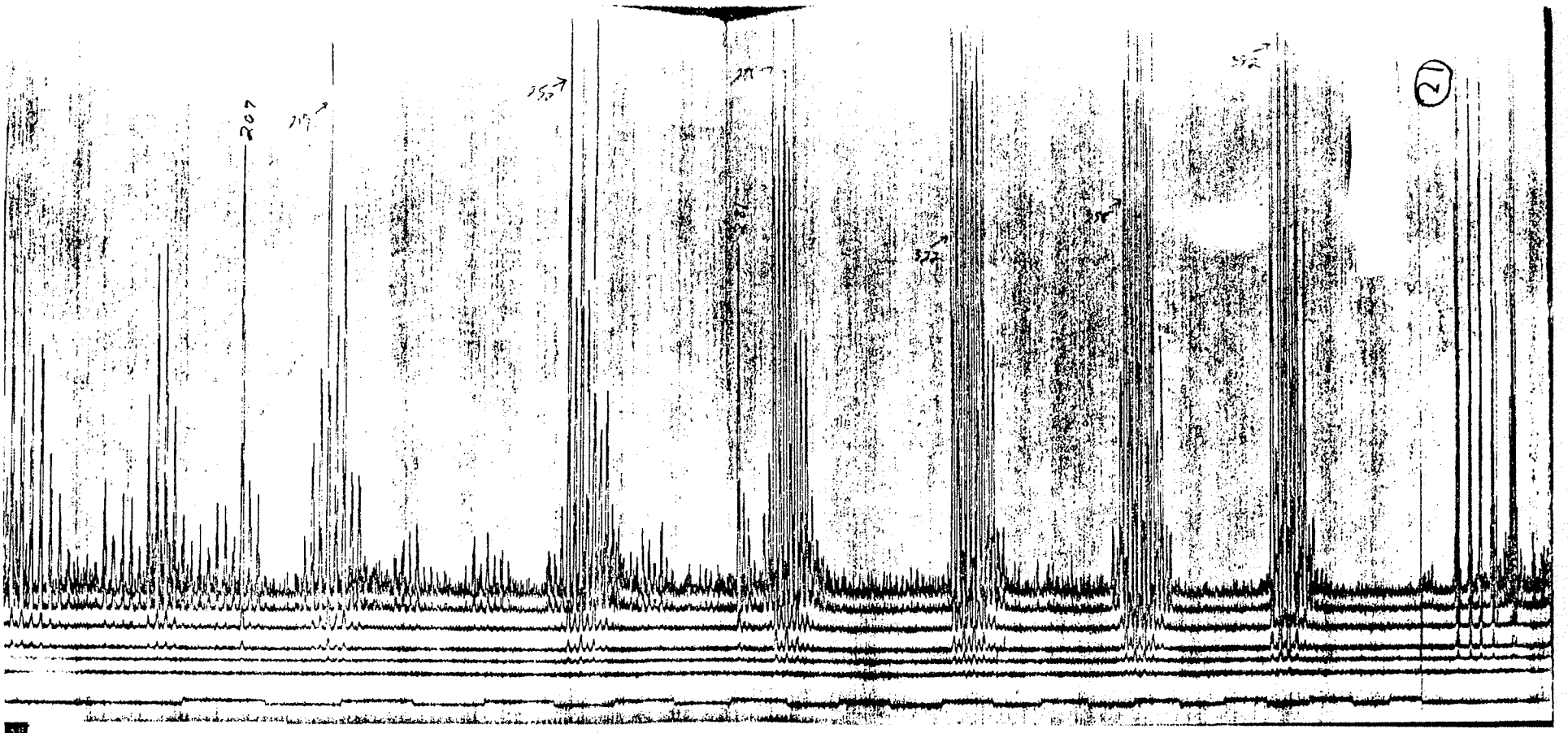

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AROCHLOR
1260
LOT #K3

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QUESTIONS ON JENSEN'S MASS SPECTRAL WORK

1. What were his GC conditions for GC/MS - supports, Temperatures, sample size, flow rate, split ratio to MS?
2. Did he program the column temperature - was column bleed a problem?
3. Did he use any unusual MS conditions?
4. Did he use stainless steel columns - if so, did he observe decomposition of some of the pesticides he studied?

MWD,JPM

PESTICIDES ANALYSIS BY MS/GC

The determination of several pesticides, including p,p' DDT and two of its metabolic products, was attempted. Under the chromatographic conditions used to identify chlorinated biphenyls, it was found that many of these pesticides decomposed to varying degrees. The chromatograms of Dieldrin, Aldrin, Lindane, and pp' DDE contained predominantly one peak, but pp' DDT, pp' DDD, and Heptaclor gave numerous broadened peaks. The decomposition is probably due to a combination of high temperatures and the catalytic effect of the stainless steel column and transfer line. The use of a glass column and transfer line might solve this decomposition problem.

COMPARISON OF ATLAS AND LKB INSTRUMENTS

	Atlas CH 4B	LKB 9000
Maximum Resolution (10% valley)	2800	1000
Fastest Scan Speed (magnetic)	14-448 in 2.2 sec.	12-400 in 1.7 sec.
Mass Range (2 mu)	2-2400	2-1400
Sensitivity for GC Inlet (μg)	0.001	0.01
Carrier Gas Separator and Max. Temp. (°C)	Biemann-Watson 300°	Becker-Ryhage 300°
Mass Marker	Yes	Available as Option
Field Ion Source	Yes	No

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