Spectroscopy
Method 70-1
Job No. 16303

IDENTIFICATION OF POLYCHLORINATED BIPHENYLS BY GAS CHROMATOGRAPHY/MASS SPECTROMETRY

SCOPE

This write-up describes a procedure for the identification of polychlorinated biphenyls (PCB) via gas chromatography/mass spectrometry (GC/MS). The method can be used to identify components in chromatographic fractions which have the number of chlorines and the molecular weight combinations typical of PCB's. Aromatic chlorine compounds having similar make-up can interfere with this identification.

PRINCIPLE

A solution to be examined for PCB's is injected into a gas chromatograph coupled to a rapid scan mass spectrometer. As peaks elute from the GC, they are directly introduced into the mass spectrometer. Mass spectra are obtained for GC peaks of interest. The mass spectra are used to determine the number of chlorines and molecular weight.

INSTRUMENTAL DETAILS

Gas chromatographic separations are carried out with an F&M 5751-B gas chromatograph using a flame ionization detector and 1/8 inch stainless steel columns containing SE-52 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. A helium flow rate through the column of 30 ml per minute and a 1:10 split between the flame detector and the mass spectrometer are maintained.

A 0.027 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. Remote actuated valves permit the venting of unwanted components such as solvent. The electron impact source is operated at 70 eV and 60 μ amp filament emission at a source temperature of 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

A 20-30 μ liter aliquot of a solution containing about 0.1% solute is injected into the chromatograph and the temperature program started. The solvent quickly passes through the column and is vented before reaching the Biemann separator. The vent valve is then closed

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and the column effluent passes directly into the ion source. A mass spectrum is scanned each time a peak is observed on the total ion monitor trace.

SPECTRAL INTERPRETATION

The mass spectra are examined for characteristic Cl^{35} and Cl^{37} multiplet peaks. An analysis of these multiplets gives the number of chlorines per molecule. The mass of the peak containing only Cl^{35} is measured. This gives the molecular weight of the PCB being examined. The relative intensity of the parent ion peak used for the molecular determination provides information about aromaticity of the compound. Mass data of this type make it easy to differentiate between pesticides and PCB's. This technique does not permit easy identification of PCB isomers having the same chlorine number.

NOTES

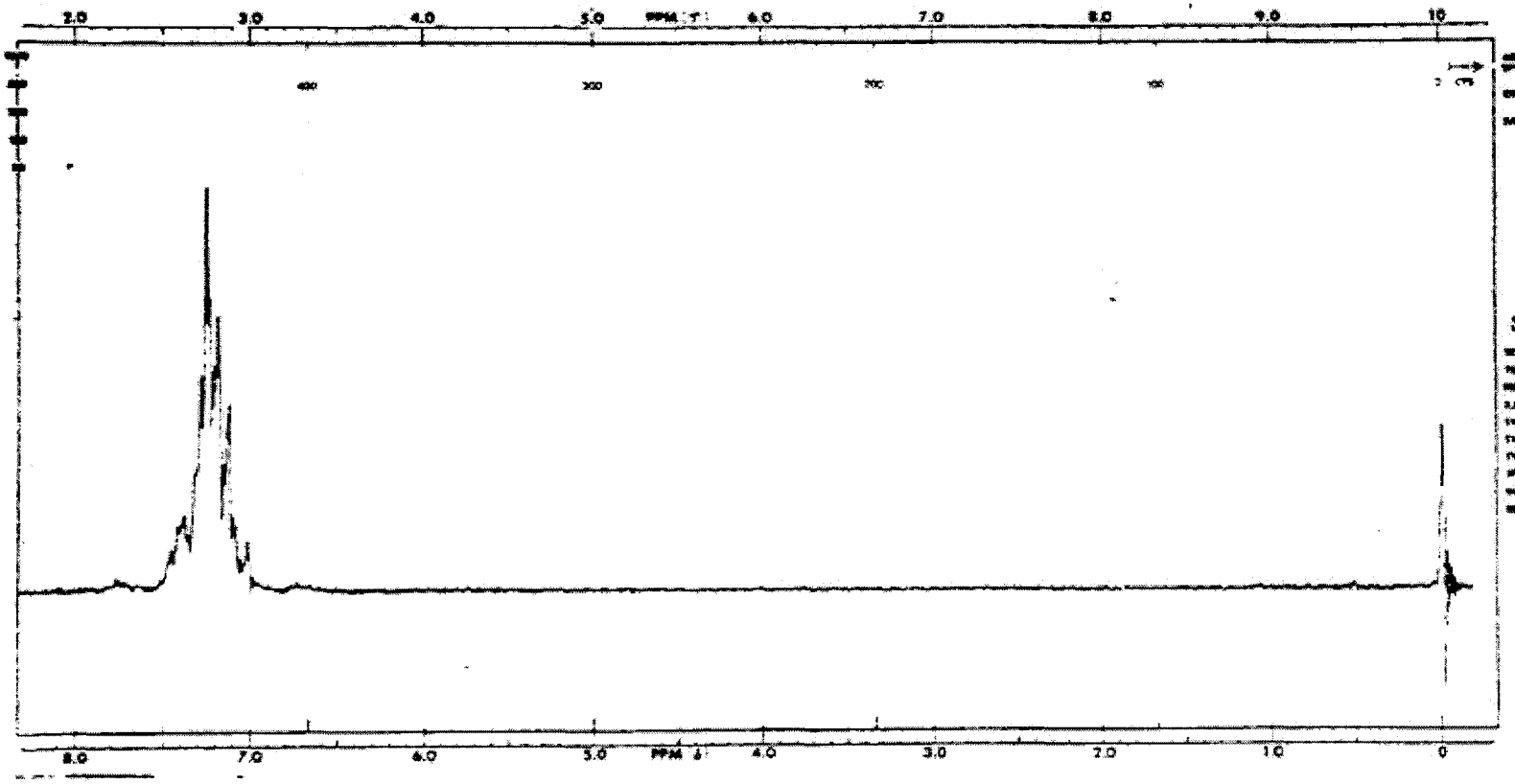
The flame ionization detector instead of an electron capture detector is used for GC/MS work. Work involving GC only is based on use of the electron capture detector. The latter provides increased sensitivity and specificity for trace determinations. This detector system is not easily compatible with the mass spectrometer because of the small sample sizes used. In order to provide 1000X increase in sample size for the mass work, it is preferable to use the less sensitive flame ionization detector given in this procedure. In addition, the flame ionization detector provides a more complete picture of interfering components being separated and introduced into the mass spectrometer.

db

Monsanto Company
Organic Chemicals Division
Research and Development Laboratories
Applied Sciences Section
St. Louis, Missouri

2/70-M. W. Dietrich, J. P. Mieure, R. E. Keller

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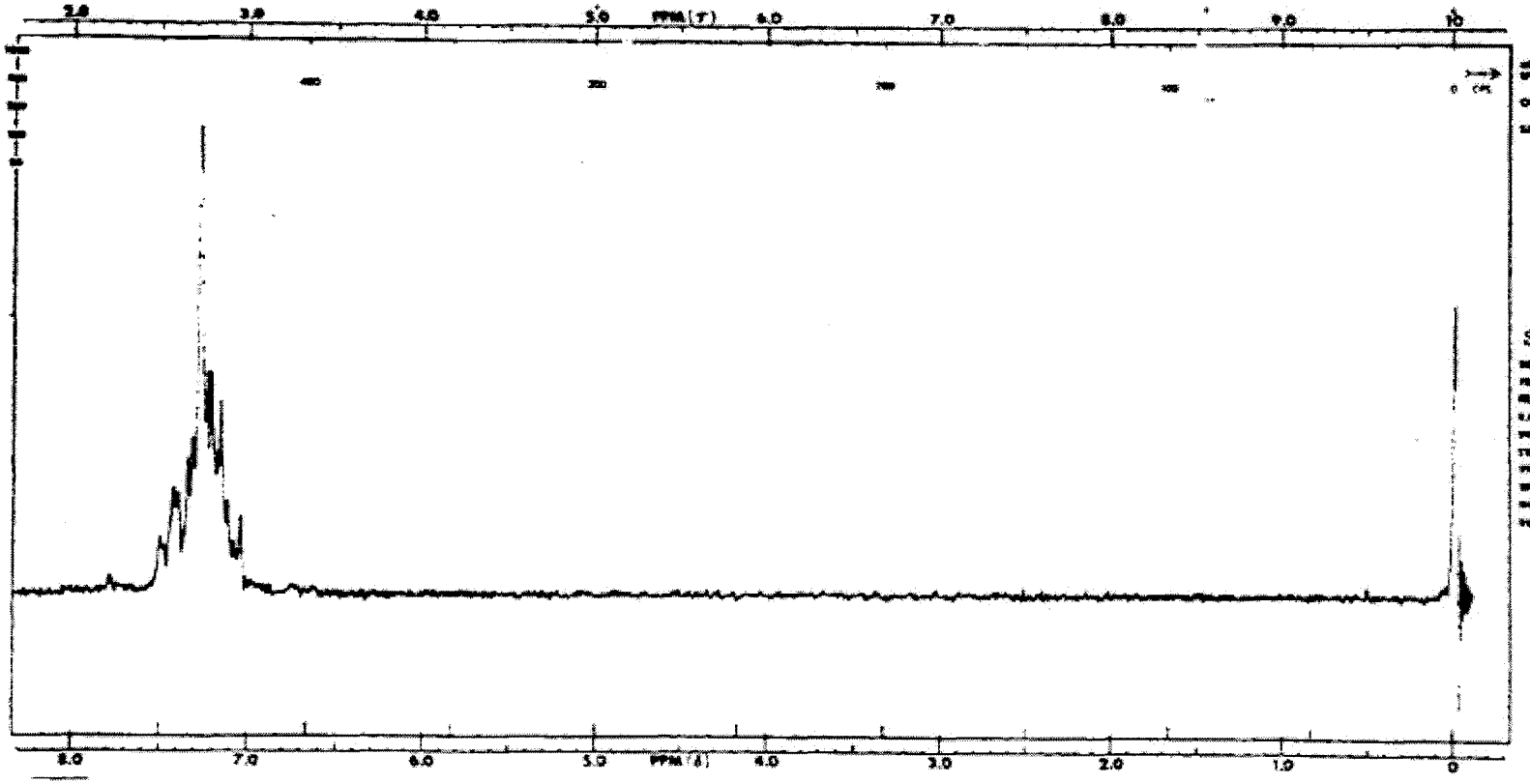
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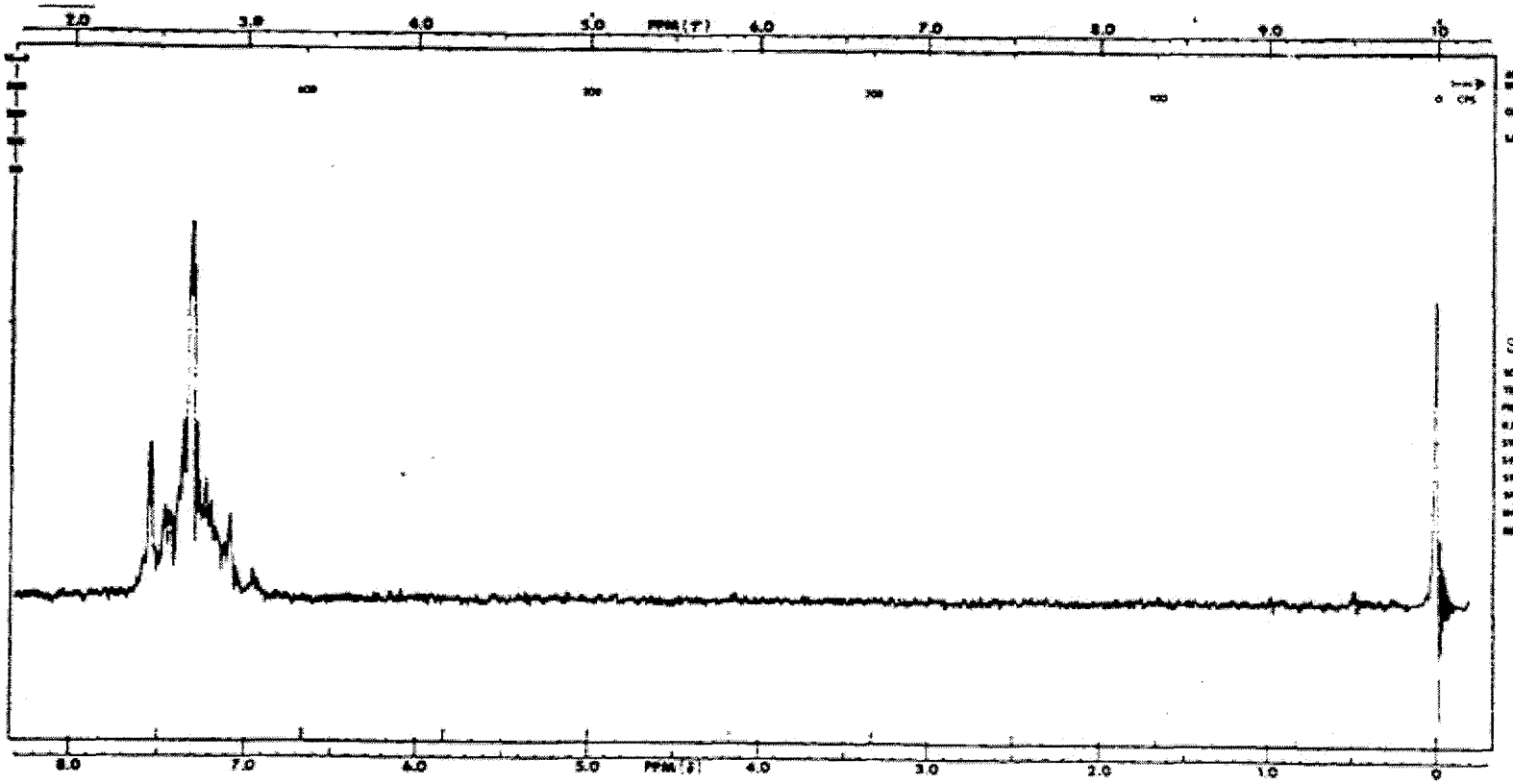
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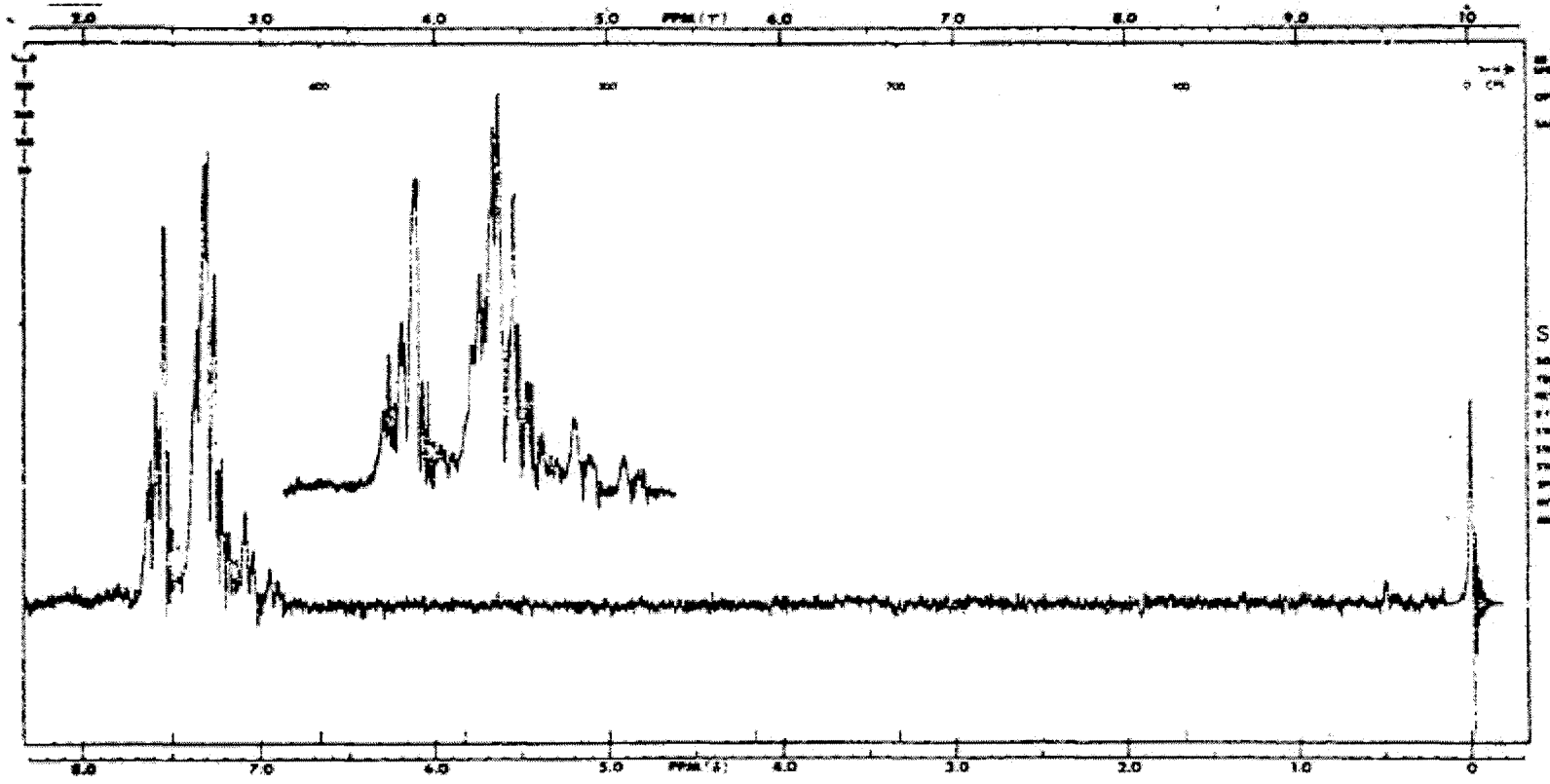
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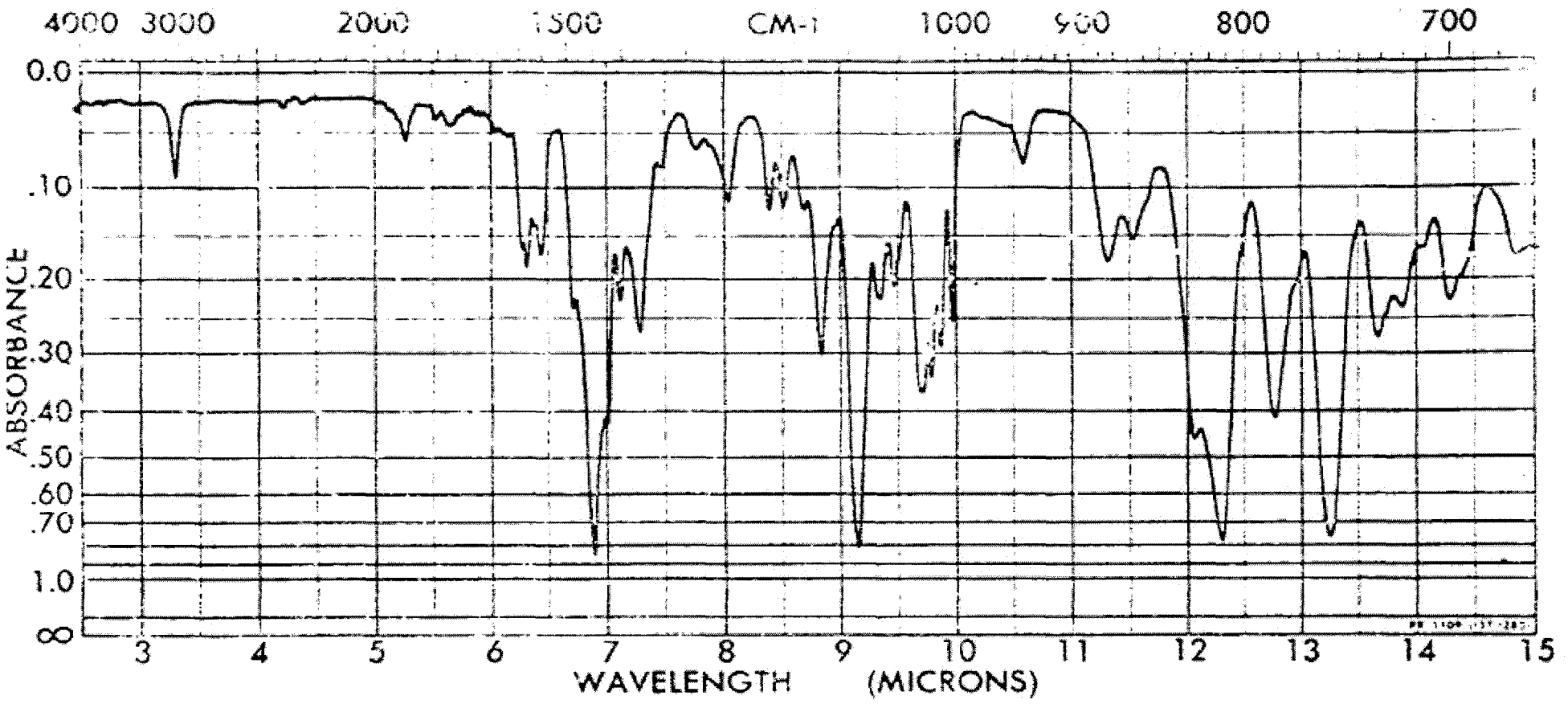
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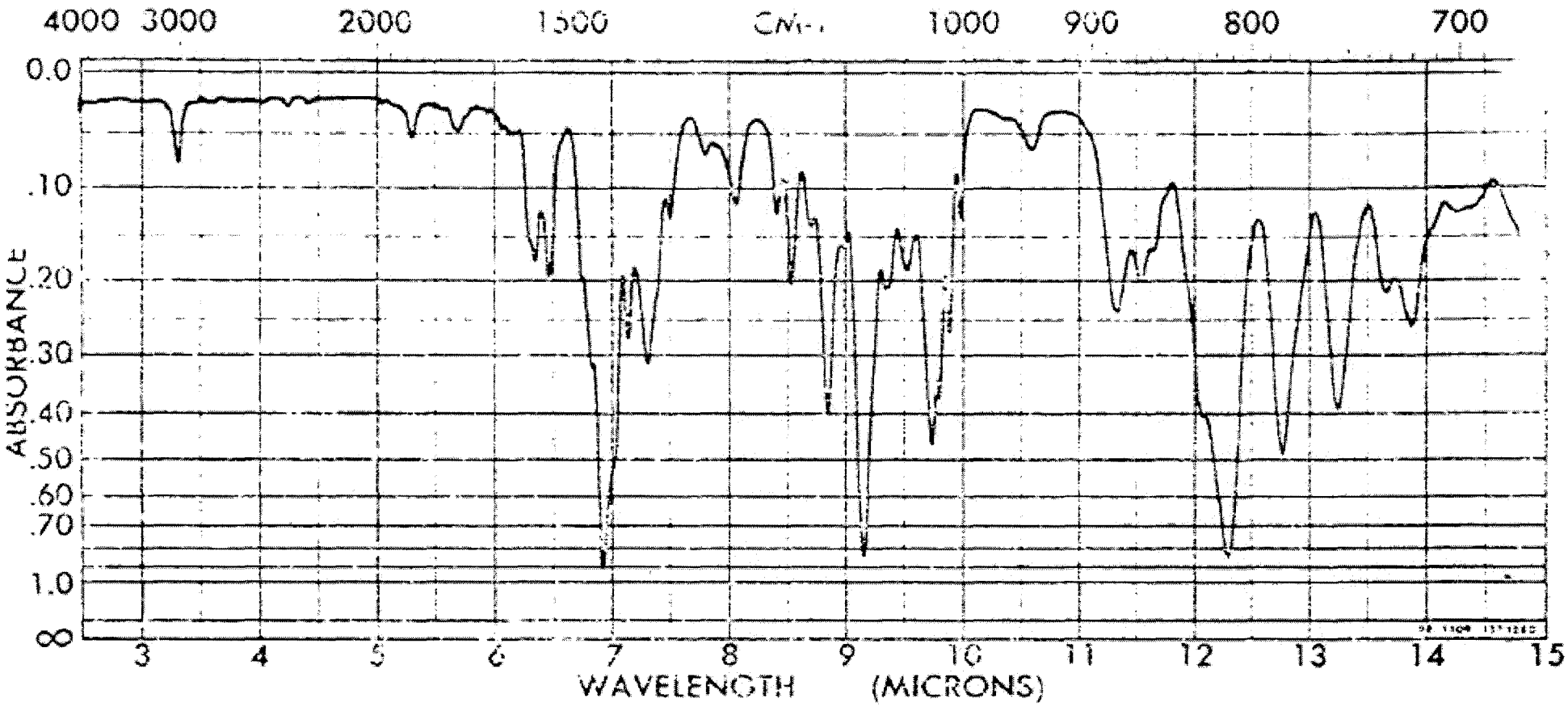


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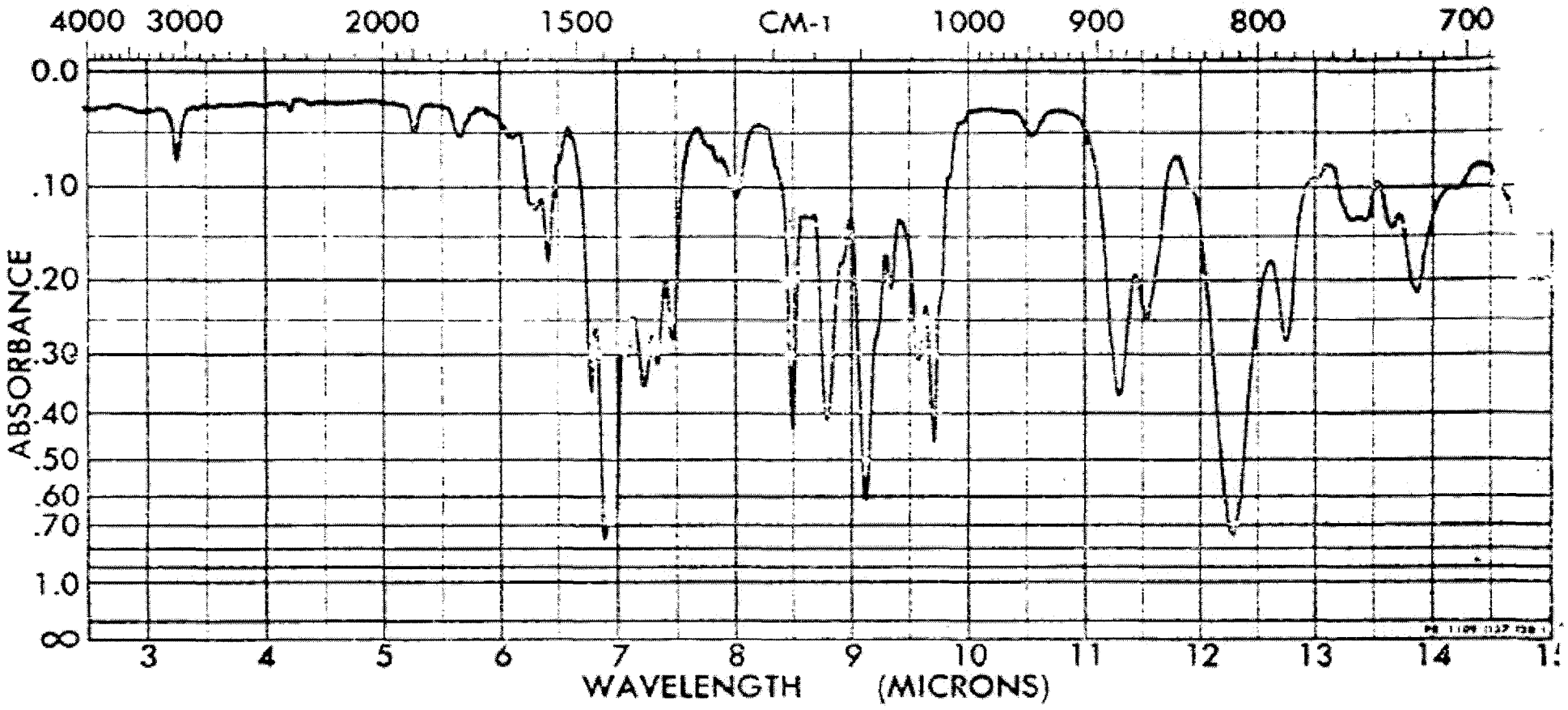


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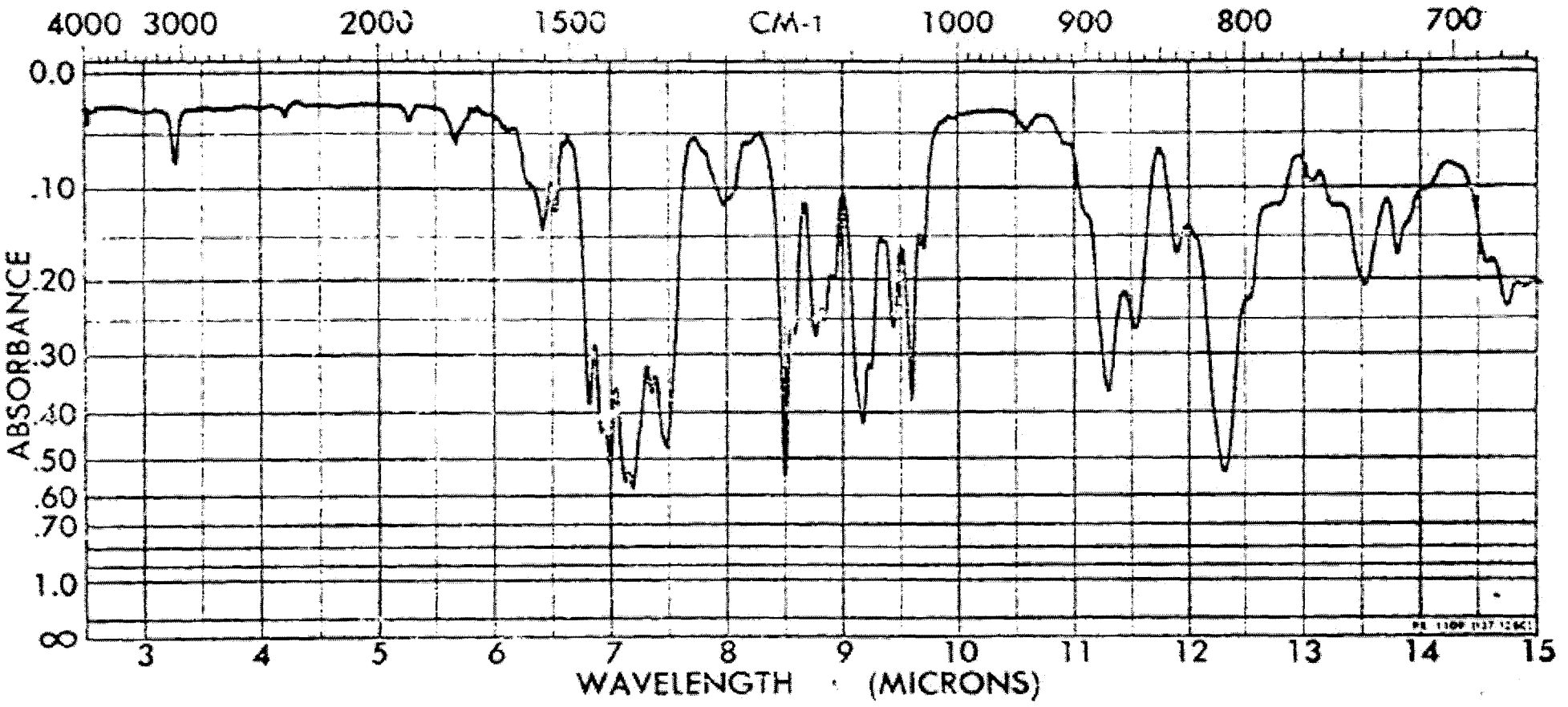


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CAPILLARY FILM

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CAPILLARY FILM

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