

BEHAVIOR OF VOLATILE AND EXTRACTABLE ORGANICS IN COMBINED  
BIOLOGICAL/PHYSICAL-CHEMICAL TREATMENT OF MUNICIPAL WASTEWATER

by

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## ABSTRACT

The purpose of this study was to examine qualitatively and semi-quantitatively the volatile (purgeable) organics and the semi-volatile liquid/liquid extractable organics present after the major steps of a reuse treatment system. Conventional GC and GC/MS methods were employed.

The combined biological/physical-chemical treatment reuse system at the EPA-DC Pilot Plant consisted of a screening device to remove coarse material, lime clarification (pH 10.5), dispersed growth nitrification, fixed film denitrification, carbon adsorption, dual media filtration, and chlorination for disinfection. This sequence of processes was chosen because they form a treatment system capable of producing a high quality water from municipal raw wastewater. Typically, effluent TOC values averaged 2 mg/l.

The final and intermediate effluents were examined for trihalomethanes and other highly volatile organics and for organics extractable by methylene chloride. Trihalomethanes and other purgeable organics typically totalled 40 µg/l.

The final and intermediate effluents were examined for trihalomethanes and other highly volatile organics and for organics extractable by methylene chloride. Trihalomethanes and other purgeable organics typically totalled 40 µg/l in the raw wastewater. A sharp decrease in concentration occurred following lime clarification and nitrification. Chloroform levels were reduced by 70 percent and the other trihalomethanes by 90 percent or better. Further significant reduction in purgeable organics did not occur in the treatment train. The trihalomethanes, after final chlorination to a free chlorine residual or approximately 1.0 mg/l, exhibited a sharp increase to typical values of 25 µg/l. Because of the low trihalomethane concentrations after chlorination, it was concluded that removal of the humus material by the system reduced the formation of trihalomethanes in the effluent.

The semi-volatile liquid/liquid extractable organics identified in the raw wastewater influent were generally reduced by the reuse system to levels not detectable by the analyses employed.

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# BEHAVIOR OF VOLATILE AND EXTRACTABLE ORGANICS IN COMBINED BIOLOGICAL/PHYSICAL-CHEMICAL TREATMENT OF MUNICIPAL WASTEWATER

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## INTRODUCTION

The increasing pollution of existing fresh water sources, as well as the need for wastewater reuse in many parts of the world, is focusing attention on more efficient methods of wastewater treatment. Effluents from the more common biological treatment processes contain considerable amounts of organic materials which are responsible for taste, odor, and color, some of which may possess the potential for severe toxic effects on the biota of freshwater bodies. In wastewater reuse for human consumption, considerable attention has been given to aesthetic, as well as physiological effects of the organic residuals thus far identified (1).

The organic materials in wastewaters affect, and are affected by, many of the physical-chemical and biological processes in use today. A better understanding of the composition and characteristics of the organic materials in raw wastewater, as well as effluents after various stages of treatment aid in better design and operation of wastewater treatment processes. The purpose of this study was to examine qualitatively and semi-quantitatively the behavior of the volatile (purgeable) organic materials and the semi-volatile methylene chloride extractable organic materials after the major steps of treatment in a combined biological/physical-chemical treatment reuse process.

In the physical-chemical treatment of raw wastewater, lime and a mineral salt such as  $\text{MgSO}_4$ ,  $\text{FeCl}_3$  or alum are added to the wastewater in the reactor to attain a pH of 10.5-11.6. Under these conditions, bicarbonate ions are converted to carbonate ions and precipitated by the excess calcium ions as calcium carbonate. Phosphorous is precipitated as calcium hydroxyapatite with soluble residuals below 0.1 mg/l (as P). The magnesium or ferric ions, converted to their gelatinous hydroxides, provide very efficient flocculation and sedimentation of the calcium carbonate, calcium hydroxyapatite and particulate organic matter in the clarifier following the reactor. Following lime clarification and sedimentation, ammonia removal is achieved by biological nitrification-denitrification or breakpoint chlorination. The wastewater is then treated with activated carbon (either granular or powdered) for the removal of dissolved organic materials. In some cases, effluents from carbon adsorption are "polished" by breakpoint chlorination for residual ammonia removal and disinfection.

In the combined biological/physical-chemical-treatment process examined in these studies, approximately 200 mg/l of CaO and 15 mg/l  $\text{FeCl}_3$  (as Fe) were added for lime clarification, at pH 10.5 (low pH-lime process). Nitrification (suspended growth) and denitrification (fixed film) were employed for ammonia removal. Methanol dosages corresponding

to methanol:NO<sub>3</sub>-N ratios of 2:1 to 4:1 were used in denitrification. Dissolved organic material was removed by passing the effluent from the denitrification stage through granular (8 x 30 mesh) activated carbon (Filtrisorb 300) contained in four columns in series at a 4.8 l/m<sup>2</sup>s loading rate (downflow packed bed operation). Following carbon adsorption, the wastewater was treated with approximately 5 mg/l of alum as Al and passed at approximately 2 l/m<sup>2</sup>s hydraulic loading rate, through a dual media filter, containing 0.6 meter of coal and 0.3 meter of sand. This final flocculation and filtration step was designed to remove particulate matter formed by, or escaping from, the carbon adsorption stage. The wastewater was then chlorinated to achieve a free available chlorine concentration of 1 mg/l for removal of any residual ammonia and for disinfection. The physical-chemical treatment process required a total detention time of 10.6 hours at a flow rate of 35 gpm ( $2.2 \times 10^{-3}$  m<sup>3</sup>/s).

## EXPERIMENTAL

Wastewater samples used in these studies were taken at the U.S. Environmental Protection Agency-District of Columbia Pilot Plant located at 5000 Overlook Avenue, S.W., Washington, D.C. The pilot plant was designed for research in the development and demonstration of more efficient methods of municipal wastewater treatment. The combined biological/physical-chemical treatment process examined in this study is shown schematically in figure 1. Chemical parameters typical of the District of Columbia raw wastewater and effluents after various stages of treatment are shown in Table 1.

Wastewater grab samples were taken after the following stages within the combined biological/physical-chemical treatment process:

Raw Wastewater (H-1)

Denitrification (I-7)

Carbon Adsorption (J-7)

Alum Coagulation and Filtration (K-7)

Chlorination and Filtration (Final Effluent) (L-7)

Samples for volatile organic analysis (VOA) were taken in 15 ml crimp-seal bottles containing a film of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> sufficient to reduce the total available chlorine in a 15 ml aliquot of water containing 30 mg/l of total available chlorine. The bottles were completely filled to avoid headspace losses and crimp-sealed with a teflon seal. Volatile organic materials were determined by GC/MS on a 5 ml sample employing the purge and trap procedures and apparatus described by Bellar and Lichtenberg (2). The trap used in these tests was filled one third with silica gel (Davidson 15, 35/60 mesh) and two thirds with Tenax-GC so that the purged volatile organic materials were exposed to the Tenax-GC first. The purged volatile organic materials adsorbed on the trap were thermally desorbed for 3 minutes in the modified injection port of the Finnigan 9500 gas chromatograph at 200°C and with fifteen cc/min helium flow, on to the chromatographic column held at ambient temperature.

Following the 3-minute thermal desorption in the injection port, the trap was removed from the injection port of the GC. The injection port

was immediately plugged with the prescribed quick-connect plug and the trap baked out at 200°C for 20 minutes under 15 cc/min helium flow. The trap was then plugged and allowed to cool to ambient temperature before reuse. The gas chromatograph was equipped with a 5' glass column of 1/4" OD (2 mm ID) and packed with Tenax-GC, 60/80 mesh (Supelco, Inc.). The injector port was maintained at 200°C for sample vaporization and thermal desorption of the Tenax-GC-silica gel trap. The separator oven and transfer lines were maintained at 250°. Temperature programming in the VOA involved maintaining the chromatographic column at ambient temperature during thermal desorption of the trap, until the water vapor eluted from the chromatographic column. The water vapor was observed as a pressure surge in the analyzer of the mass spectrometer--its elution required approximately two minutes. An additional two minutes were allotted at ambient temperature before temperature programming to 190°C at 8 deg/min. The final temperature was maintained for 10 minutes before ending data acquisition. Data acquisition on the Finnigan 3300 Mass Spectrometer continued following elution of the water vapor from the chromatographic column for a total data acquisition time of 32 minutes.

A Finnigan 3300 mass spectrometer was utilized in these studies to provide mass spectra of the components separated by the gas chromatograph. The Finnigan 3300 GC/MS system was interfaced to a Systems 150 GC/MS Data System (PDP-8) for rapid processing of mass spectral data.

The GC/MS system was operated in the control mode over a mass range of 30-200, 201-400 amu, using integration times of 8 ms/amu, for a 3.8 second scan time for the VOA. Sensitivity was set at 10<sup>-7</sup> amps/volt and electron energy was 70 ev. Real time attenuation was set so that 750 ng of chloroform produced a full scale response in order to have a working range of 1 to 150 ppb chloroform.

Grab samples taken for the analysis of methylene chloride extractable acidic, neutral, and basic organic materials were contained in 1 gallon wide-mouth glass jars under an aluminum foil seal. The jars were filled in order to avoid head space losses. The samples were not dechlorinated. The samples were packed in ice and shipped by air express to the Cincinnati laboratory for extraction. The time interval between sample selection and sample extraction was 24-48 hours. The samples, usually 4-liter aliquots, were extracted in 6-liter separatory funnels with methylene chloride for acidic, neutral, and basic materials. The methylene chloride extracts were dried, concentrated, and the solvent changed to acetone according to the procedure described in the EPA GC/MS Procedural Manual (3). The final acidic, neutral, and basic methylene chloride extractable organic materials (now in acetone), were concentrated, under a gentle flow of dry nitrogen, to 1.0 ml and held at 4°C for GC/MS analysis.

The Finnigan 9500 gas chromatograph used in the analysis of the acidic, neutral, and basic extracts was equipped with a 30-meter glass capillary column coated with SE-30 and possessing 86,000 effective theoretical plates.

The gas chromatograph was modified to allow the flow rate of the helium carrier gas to be controlled by adjusting the pressure regulator at the main cylinder rather than at the controls of the gas chromatograph. The Finnigan GC/MS system was further modified for these studies by installing a glass-lined transfer line to deliver the column effluent

directly to the analysis of the mass spectrometer, by-passing the separator.

The helium pressure was adjusted to approximately 7 psi and the Grob injector adjusted to yield a linear velocity of 30 cm/sec through the column at 100°C with pentane, and a split ratio of approximately 30:1. The injector and transfer lines were maintained at 270°C. The Grob injector was closed for at least 30 seconds prior to sample injection, and opened 30 seconds after injection of the sample.

Temperature programming selected for the analysis of the acidic, neutral, and basic extracts started at approximately 30°C (ambient temperature) and was programmed to 250°C at 4 degrees per minute. The final temperature was maintained for 15 minutes for a total data acquisition run time of 70 minutes. Temperature programming and data acquisition were begun 3 minutes after sample injection in order to avoid data acquisition during elution of the solvent.

Additional mass spectrometric conditions employed in the analysis of the acidic, neutral, and basic extracts included operation in the control-mode over the mass range of 20-200, and 201-500, with integration times of 2 and 4 ms/amu respectively. The electron energy was set at 70 ev. and the emission current was 0.5 amp. Amplifier sensitivity was set at  $10^{-7}$  amps/volt. The ion energy, extractor, and lens voltages were adjusted for optimum peak, shape and sensitivity.

#### QUALITY ASSURANCE

Quality assurance measures during the VOA involved achieving ion abundance data from perfluorotributylamine, which matched that taken when the mass spectrometer was properly tuned and verified from ion abundance data from decafluorotriphenylphosphine. Additional sensitivity and chromatographic performance checks were made by running fresh standard solutions.

Quality assurance measures throughout the analysis of the acidic, neutral, and basic extracts involved strict compliance with the GC/MS tuning and sensitivity recommendations prescribed by Eichelberger and Budde (4) with decafluorotriphenylphosphine under identical sample analysis conditions. Additional column performance and sensitivity checks were made by running a C<sub>18</sub>, C<sub>19</sub>, and C<sub>20</sub> hydrocarbon mixture. Sensitivity was achieved so that 10 ng of the hydrocarbons produced a 4:1 signal to noise ratio.

Compound identifications were made by computerized spectral matching techniques and by running and matching against known standards when available. Blanks were carried through each extraction process and compensations were made during data analysis. All glassware exposed to the sample prior to GC/MS analysis was previously cleaned by being washed with hot detergent solution, rinsed with tap water, rinsed with distilled water, air dried and heated in a muffle furnace at 400°C for 30 minutes.

The methylene chloride and acetone solvents used in these tests were "distilled in glass" quality obtained from Burdick and Jackson and were used as received. The anhydrous sodium sulfate for drying the extracts was analytical reagent quality, obtained from Fisher Scientific Co., and was used as received.

## DISCUSSION OF RESULTS

### A. Methylene Chloride Extractable Organics

The complexity of the total ion chromatograms of the extracts of methylene chloride extractable materials from wastewater after varying stages of physical-chemical treatment seemed to parallel the TOC and COD values (Table 2). The total ion chromatograms of the various extracts are presented to demonstrate effluent quality and are typical (Fig. 3-15). The methylene chloride extractable materials in the raw wastewaters (H-1) showed considerable variation in content as well as in complexity of the total ion chromatograms. No single organic compound was found in the 8/3/76 raw wastewater extracts above 10 ppb (Table 3). In the 11/4/76 raw wastewater extracts, only stearic and palmitic acids were found at concentrations above 10 ppb. These acids, as well as lauric and oleic were found in the neutral fraction and were not found at concentrations above 1 ppb in the acidic fraction. Caffeine was also found in the neutral fraction and not detected in the basic fraction (Table 4). The large number of saturated and unsaturated aliphatic hydrocarbons detected in the raw wastewater extracts indicate motor oil and other internal combustion engine by-products. No organic bases were detected in the raw wastewater extracts except caffeine and a trace of a compound tentatively identified as N,N-diphenylhydrazine (Table 4).

Following lime precipitation (clarification), biological nitrification and denitrification, (I-7), the methylene-chloride extractable organic materials in the wastewater were dramatically reduced from that of the raw wastewater (Tables 5 and 6) (Fig. 7-9). Only acetic acid was identified in the neutral fraction of I-7 at a concentration greater than one ppb (Table 6). This probably was sample contamination since acetic acid is sufficiently volatile to have been lost during Kuderna-Danish concentration and should not have been extracted into the neutral fraction.

Following carbon adsorption, GC/MS analysis of the K-7 extracts revealed further reduction of complexity in the total ion chromatograms (Fig. 10-12). The TOC and COD analyses revealed a parallel reduction of total organic matter. No compounds were identified in any of the K-7 extracts at concentrations greater than 1 ppb (Tables 7 and 8). Likewise, no peaks were observed in the total ion chromatograms of the K-7 extracts that could have represented concentrations greater than 1 ppb.

Following chlorination for residual ammonia removal and disinfection and alum addition and filtration for residual turbidity removal, the total ion chromatograms of the final effluent (L-7) extracts revealed no significant difference from those of the K-7 extracts (Tables 9 and 10) (Fig. 13-15). All of the compounds identified in the L-7 extracts were at the low ppb levels (1-5 ppb) or at the lower limits of detection by GC/MS under the conditions employed in these tests. These low concentrations encountered in the methylene chloride extracts of the final effluents from the wastewater treatment system made spectral identification difficult and was responsible for most of the tentative identifications made.

Considerable quantities of chlorinated and brominated cyclohexanes, cyclohexenes, and cyclohexanols were detected in the neutral and acidic extracts of the final effluent (L-7) (Fig. 13 and 14). The presence of



these compounds was attributed to the extraction of wastewater containing free chlorine residuals with methylene chloride preserved with cyclohexene (5).

## B. Volatile (Purgeable) Organics

Halogenated ethanes and methanes were compiled into a separate table here for special discussion due to the recent emphasis placed on these materials as possible carcinogens.

In the Washington, D.C. raw wastewater, chloroform concentrations averaged a rather steady 10  $\mu\text{g/l}$  in the summertime grab samples. Other halogenated methanes and ethanes identified in the raw wastewater were at concentrations below 10  $\mu\text{g/l}$  (Table 11). The concentration of chloroform in the wastewater was reduced 50 to 100 percent following biological nitrification and denitrification (I-7). The concentrations of other halogenated methanes and ethanes identified in the wastewater following biological nitrification and denitrification were reduced to levels not detectable by the conditions employed in these tasks. This reduction in the concentration of volatile halogenated methanes and ethanes was attributed to the purging action of the aeration step incorporated in the nitrification step previously described (Fig. 1). No further reductions in the volatile halogenated methanes and ethanes were observed following the carbon adsorption (J-7). Indeed, chloroform concentrations may have increased slightly following carbon adsorption as shown in the 7/1/76 to 7/8/76 tests.

After chlorination to a free chlorine residual of approximately 1 mg/l, the final effluent (L-7) contained concentrations of chloroform ranging from 5 to 10  $\mu\text{g/l}$ . Other halogenated methane and ethane concentrations increased from undetectable levels to levels ranging from 3 to 5  $\mu\text{g/l}$  (Fig. 16). More data is necessary to substantiate this apparent formation of halogenated methanes and ethanes by chlorination of treated wastewaters, because in these studies, no attempt was made to ascertain that the chlorine used in the chlorination step was not contaminated with chloroform and other volatile halogenated materials.

Other volatile (purgeable)/organic materials identified in the wastewaters are tabulated in Table 12. Organic compounds other than the halogenated methanes and ethanes were not consistently detected in the final effluent (L-7) above 1  $\mu\text{g/l}$ .

## CONCLUSIONS

The dramatic reduction in the total organic material in the wastewater following lime clarification and biological nitrification and denitrification, as monitored by COD and TOC paralleled the reduction in the complexity and number of peaks in the respective gas chromatograms (Fig. 2). The efficient removal of the specific organic matter by lime precipitation and biological nitrification and denitrification (ammonia removal step) suggested effective:

- a. Adsorption and absorption by the gelatinous hydroxides formed during the lime clarification step, with subsequent removal by sedimentation (occlusion).

- b. Purging of the more volatile organic materials from the water during the aeration step of biological nitrification.
- c. Biological degradation of the organic materials and/or adsorption into the bio-mass during the nitrification and denitrification processes.

The activated carbon process reduced the wastewater TOC from approximately 6 mg/l to 2 mg/l. The absence of peaks in the gas chromatograms of the acidic, neutral, and basic fractions of the extracts from treated wastewater samples taken after the activated carbon treatment step indicated that the residual organic materials (~ 2 mg/l of TOC) may be of amphoteric nature and thus not extractable by organic solvents used in analytical methods, or that any specific extractable organic may be at concentration levels below the level of detection employed in these tests. Further work is needed to characterize the 2 mg/l of residual TOC in the renovated water.

The results of these studies of the behavior of volatile and extractable organics in a combined biological/physical-chemical treatment of a municipal raw wastewater revealed final effluent concentrations of halogenated methanes and ethanes, other volatile organic materials, and the extractable materials to be similar to those found in finished drinking waters during the EPA National Reconnaissance Survey of 1975. The specific organic compounds identified in the final effluent have also been identified in finished drinking waters (6).

A portion of the VOA data generated in this study has been presented in a previous report (6). It is suggested that this reference be consulted for a detailed comparison of the reuse effluent with finished drinking waters.

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TABLE 1

Chemical Parameters Typical of the District of Columbia  
Raw Wastewater and After Various Stages in the  
Combined Biological/Physical-Chemical Treatment Process

Chemical Parameters	H-1	I-7	K-7	L-7
Alkalinity, P	-	-	-	-
Alkalinity, MO	126	-	-	96
pH	7.2	-	7.5	7.5
Conductivity (MHOS)	-	-	-	710,000
TOC	72	7.0	2.5	2.7
BOD	104	4.5	1.3	2.8
COD	237	18.5	6.6	6.5
Total P ( $\text{PO}_4$ )	15.0	0.3	0.17	0.16
TKN	19.0	0.76	0.25	0.22
$\text{NH}_3\text{-N}$	16.0	0.20	0.95	0.65
$\text{NO}_2^- + \text{NO}_3^- \text{-N}$	0.89	4.7	4.8	4.8
Suspended Solids	107	3.9	0.97	0.86
VSS	83.0	2.7	-	-
TS	-	-	-	368
$\text{Ca}^{++}$	32	-	-	56
T-Fe	1.3	-	-	0.6
$\text{Mg}^{++}$	6.5	-	-	5.4
$\text{Cl}^-$	-	-	-	68.7
$\text{SO}_4^{--}$	-	-	-	50.7
$\text{Na}^+$	-	-	-	34.1
$\text{K}^+$	-	-	-	8.2
$\text{F}^-$	0.7	-	-	0.7
MBAS	8.9	.28	-	.14
$\text{Al}^{+++}$	-	-	.25	-

TABLE 2

Organic Materials Identified in the 8/3/76  
Raw Wastewater Extract

Compound	Concentration (µg/l)
<u>H-1 Neutrals</u>	
Tetrachloroethylene	3
Methylethylbenzene	1
Trimethylbenzene	1
Trimethylbenzene	1
Saturated Aliphatic Hydrocarbon	1
Aromatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
<u>H-1 Acids</u>	
Caprylic Acid	1
Octanoic Acid	1
Nonanoic Acid	1
Decanoic Acid (Tentative)	1
Palmitic Acid	1
Oleic Acid	2
Stearic Acid	2
<u>H-1 Bases</u>	
Caffein	1

TABLE 3

Organic Materials Identified in the 11/4/76  
Raw Wastewater Extract

Compound	Concentration (µg/l)
<u>H-1 Neutrals</u>	
Tetrachloroethylene	2
1,3-Xylene	1
2-Butoxyethane	2
Dioctylether (Tentative)	1
1-Methylethylbenzene	1
?	1
Trimethylbenzene	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Trimethylbenzene	2
Methyl-isopropylcyclohexane	1
?	2
Saturated Aliphatic Hydrocarbon	1
?	3
?	3
?	1
Alpha Terpinol	1
Alpha Terpinol	2
?	3
?	3
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	2
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	2
Saturated Aliphatic Hydrocarbon	4
Saturated Aliphatic Hydrocarbon	2
1,3-dimethylnaphthalene	4
Saturated Aliphatic Hydrocarbon	2
Saturated Aliphatic Hydrocarbon	2
Trimethylnaphthalene	4
Saturated Aliphatic Hydrocarbon	1
Aliphatic Ketone	1
Saturated Aliphatic Hydrocarbon	2
Saturated Aliphatic Hydrocarbon	3
Lauric Acid	2
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	2
Saturated Aliphatic Hydrocarbon	1
Oleic Acid	3
Caffein	1
Saturated Hydrocarbon	1
Palmitic Acid	> 20
Saturated Hydrocarbon	-
Stearic Acid	> 20
?	2
Saturated Hydrocarbon	1
Saturated Hydrocarbon	1
Steroid	1
Steroid	1

TABLE 3 (Cont'd)

Organic Materials Identified in the 11/4/76  
Raw Wastewater Extract

Compound	Concentration (µg/l)
<u>H-1 Acids</u>	
3-methyl pentanal	1
Nitroethylpropionate (Tentative)	1
2-N-butoxyethanol	1
Hexanoic Acid (Tentative)	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	2
Saturated Aliphatic Hydrocarbon	3
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Unsaturated Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	3
2-Undecenal (Tentative)	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon C <sub>8</sub> -C <sub>12</sub>	3
Saturated Aliphatic Hydrocarbon C <sub>12</sub> -C <sub>14</sub>	2
Saturated Aliphatic Hydrocarbon	3
Saturated Aliphatic Hydrocarbon C <sub>8</sub> -C <sub>13</sub>	2
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon C <sub>12</sub> -C <sub>22</sub>	1
Unsaturated Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Palmitic Acid	1
Di(2-ethylhexyl) phthalate	1
?	1
<u>H-1 Bases</u>	
N,N-Diphenyl hydrazine	(trace)
?	1
?	1



TABLE 4

Organic Materials Identified in the 8/3/76  
I-7 Extract

Compound	Concentration (µg/l)
<u>I-7 Neutrals</u>	
5 methyl-1-hexene	1
1-octene	1
3-Nonone (Tentative)	1
3-hexanol	1
Tetraethyl lead	1
Tetramethylsilane	1
Butyl phthalate	1
<u>I-7 Acids</u>	
Saturated Aliphatic Hydrocarbon	1
Unsaturated Aliphatic Hydrocarbon	1
Benzothiazole	1
Butyl phthalate	1
<u>I-7 Bases</u>	
No peaks above 1 ppb	

TABLE 5

Organic Materials Identified in the 11/4/76  
I-7 Extract

Compound	Concentration (µg/l)
<u>I-7 Neutrals</u>	
Acetic Acid	3
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
1,2-ethane diacetate	1
Aliphatic Alcohol	1
Cyclohexenone	1
Saturated Aliphatic Hydrocarbon	1
?	1
4-heptanone	1
4-chlorocyclohexanol	1
?	1
?	1
Aliphatic Hydrocarbon	1
?	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
4-methyl-2,6-ditert-butylphenol	1
Saturated Aliphatic Hydrocarbon	1
4-ethyl-2,6-ditert-butylphenol	1
Diethylphthalate	1
Aromatic Hydrocarbon	1
?	2
1,2-dimethyl-4-benzylbenzene	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
Saturated Aliphatic Hydrocarbon	1
?	1
<u>I-7 Acids</u>	
No peaks above 1 ppb	
<u>I-7 Bases</u>	
No peaks above 1 ppb	

TABLE 6

Organic Materials Identified in the  
8/3/76 K-7 Extract

Compound	Concentration (µg/l)
<u>K-7 Acids</u>	
Acetic Acid	1
Unsaturated Hydrocarbon	1
Unsaturated Hydrocarbon	1
3-heptanol	1
3-heptanone	1
<u>K-7 Bases</u>	
No peaks above 1 ppb	

TABLE 7

Organic Materials Identified in the  
11/4/76 K-7 Extract

Compound	Concentration (µg/l)
<u>K-7 Neutrals</u>	
Unsaturated Aliphatic Hydrocarbon	1
Unsaturated Aliphatic Hydrocarbon	1
<u>K-7 Acids</u>	
Unsaturated Hydrocarbon	1
Unsaturated Hydrocarbon	1
Di-(2-ethylhexyl) phthalate	1
"α-phthalate"	1
<u>K-7 Bases</u>	
Unsaturated Hydrocarbon	1
Unsaturated Hydrocarbon	1
N,N-diphenylhydrazine (Tentative)	1

TABLE 8

Organic Materials Identified in the 8/3/76  
L-7 (Final Effluent) Extract

Compound	Concentration (µg/l)
<u>L-7 Neutrals</u>	
Unsaturated Hydrocarbon (C <sub>15</sub> -C <sub>19</sub> )	2
Isobutyl phthalate	3
<u>L-7 Acids</u>	
No peaks above 1 ppb	
<u>L-7 Bases</u>	
5-methyl-1-hexene	1
Trans-2-octenal (Tentative)	3

TABLE 9

Organic Materials Identified in the 11/4/76  
L-7 (Final Effluent) Extract

Compound	Concentration (µg/l)
<u>L-7 Neutrals</u>	
Unsaturated Aliphatic Hydrocarbon	2
Unsaturated Aliphatic Hydrocarbon	2
3-methyl-2-pentanone	1
Saturated Aliphatic Hydrocarbon	1
C <sub>20</sub> -C <sub>22</sub> Carboxylic Acid	2
Diisobutyl phthalate	1
2-butoxy-2-oxoethyl-butyl phthalate	4
Diisooctylphthalate	2
<u>L-7 Acids</u>	
Hydroxy-2-propanone	1
Acetic Acid	1
Unsaturated Hydrocarbon	1
Di-(2-ethylhexyl) phthalate	1
<u>L-7 Bases</u>	
No peaks from sample above 1 ppb	

TABLE 10

Halogenated Ethanes and Methanes Identified in  
Effluents of the Combined Biological/Physical-Chemical Process

Compound	Extract No.			
	H-1	I-7	J-7	L-7
<u>6/18/76</u>				
Chloroform	10	5	5	7
Bromodichloromethane	-	-	-	3
Dibromochloromethane	5	-	-	1
Bromoform	-	-	-	-
Carbon tetrachloride	2	-	-	-
Tetrachloroethylene	4	-	-	-
1,1,1-Trichloroethane	-	-	-	-
<u>7/1/76</u>				
Chloroform	10	-	5	10
Bromodichloromethane	-	-	-	5
Dibromochloromethane	1	-	2	5
Bromoform	-	-	-	-
Carbon tetrachloride	4	-	-	-
Tetrachloroethylene	3	-	-	-
1,1,1-Trichloroethane	2	-	-	-
<u>7/8/76</u>				
Chloroform	10	1	6	5
Bromodichloromethane	1	Trace	-	3
Dibromochloromethane	5	-	Trace	-
Bromoform	-	-	-	-
Carbon tetrachloride	4	-	-	-
Tetrachloroethylene	3	-	-	-
1,1,1-Trichloroethane	2	-	-	-

TABLE 11

Other Volatile (Purgeable) Organic Materials  
Identified in Effluents of the Combined  
Biological/Physical-Chemical Process

Compound	H-1	I-7	J-7	K-7
<u>6/18/76:</u>				
Acetaldehyde	-*	✓*	✓	-
Methanol	-	-	-	-
Acetone	✓	✓	✓	✓
Dichloromethane	10	-	1	1
Acrolein	-	✓	-	✓
Carbon disulfide	-	-	-	✓
Dimethyl disulfide	6	-	-	-
Toluene	2	-	-	-
Xylene	1	-	-	✓
Alkyl benzene	1	-	-	-
Benzaldehyde	-	✓	-	-
<u>7/1/76:</u>				
Acetaldehyde	✓	-	-	-
Methanol	-	-	-	-
Acetone	✓	✓	✓	✓
Dichloromethane	1	-	1	-
Acrolein	✓	-	-	-
Carbon disulfide	✓	-	-	-
Dimethyl disulfide	-	-	-	-
Toluene	2	-	✓	-
Xylene	-	✓	✓	✓
Alkyl benzene	✓	-	-	-
Benzaldehyde	-	✓	-	5
<u>7/8/76:</u>				
Acetaldehyde	-	-	-	-
Methanol	✓	-	-	-
Acetone	✓	✓	✓	✓
Dichloromethane	-	✓	2	✓
Acrolein	-	-	-	-
Carbon disulfide	-	-	✓	-
Dimethyl disulfide	-	-	-	-
Toluene	2	✓	✓	✓
Xylene	-	✓	✓	-
Alkyl benzene	-	-	-	-
Benzaldehyde	-	-	-	✓

\* Compounds represented by a dash (-) = not detected.

Compounds represented by a check (✓) = trace amounts (less than 1 µg/l).

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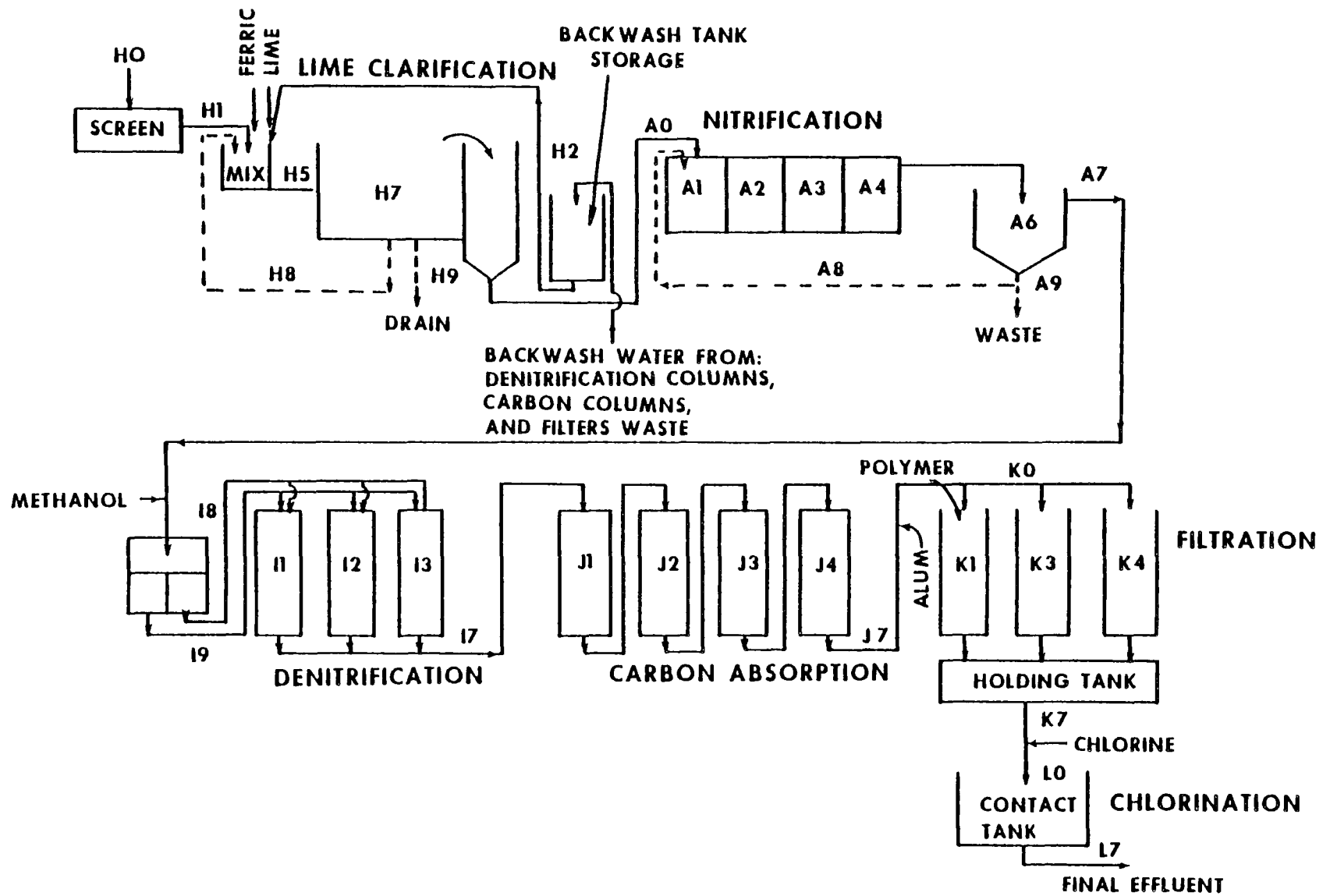


Fig. 1. Schematic Flow Diagram



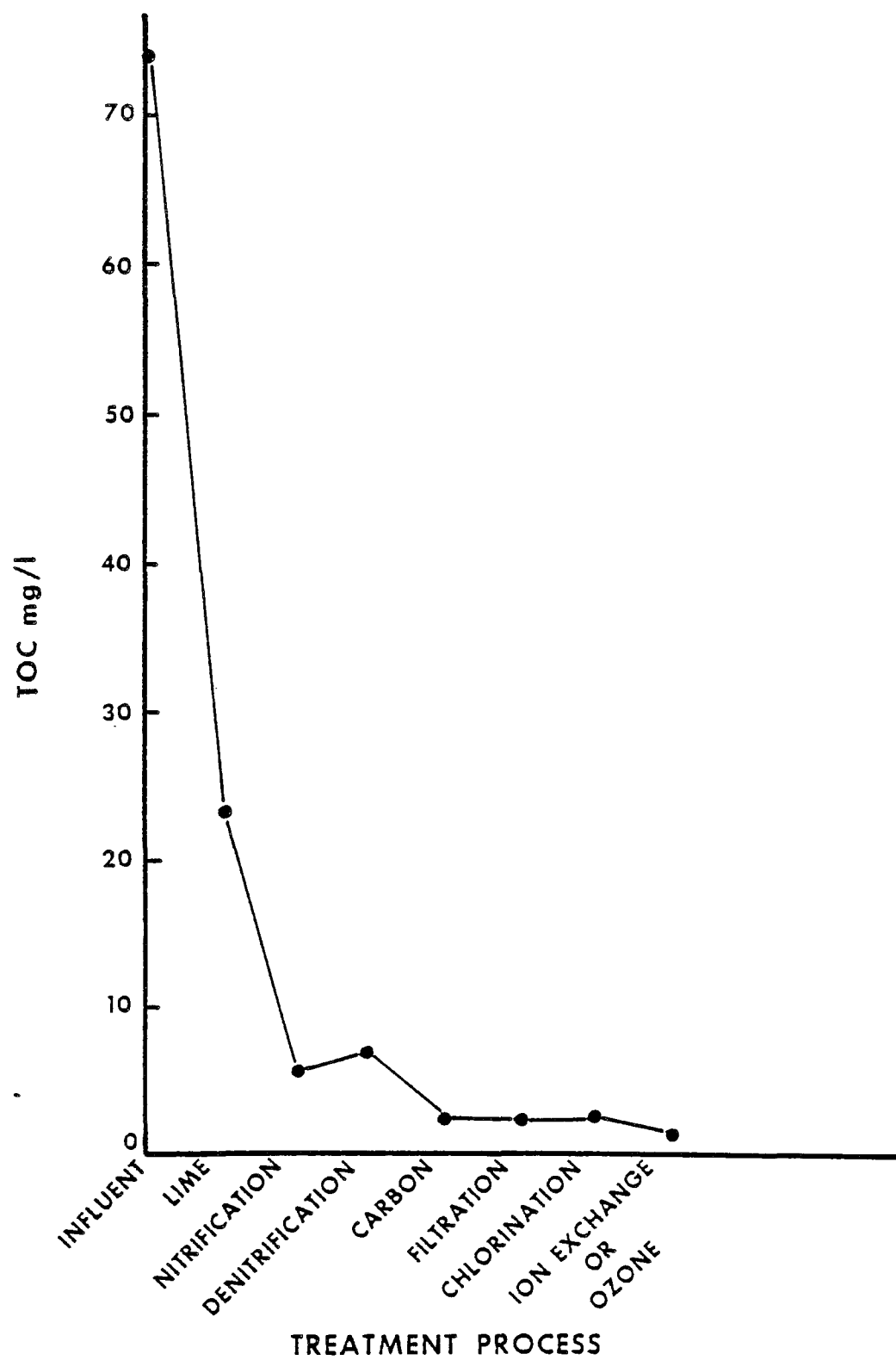


Fig. 2. Removal of TOC

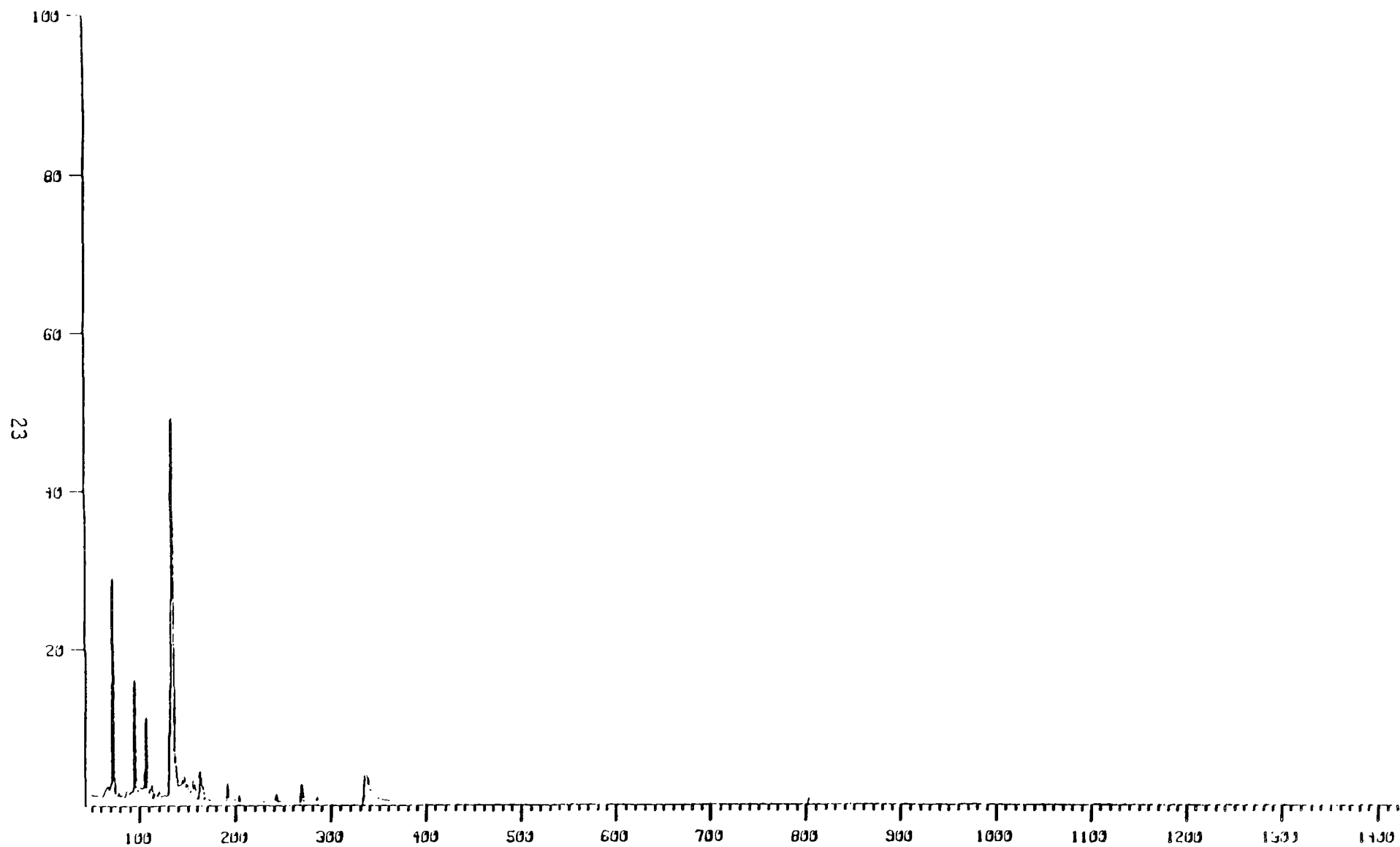


Fig. 3. Total Ion Chromatogram of Blank Extract

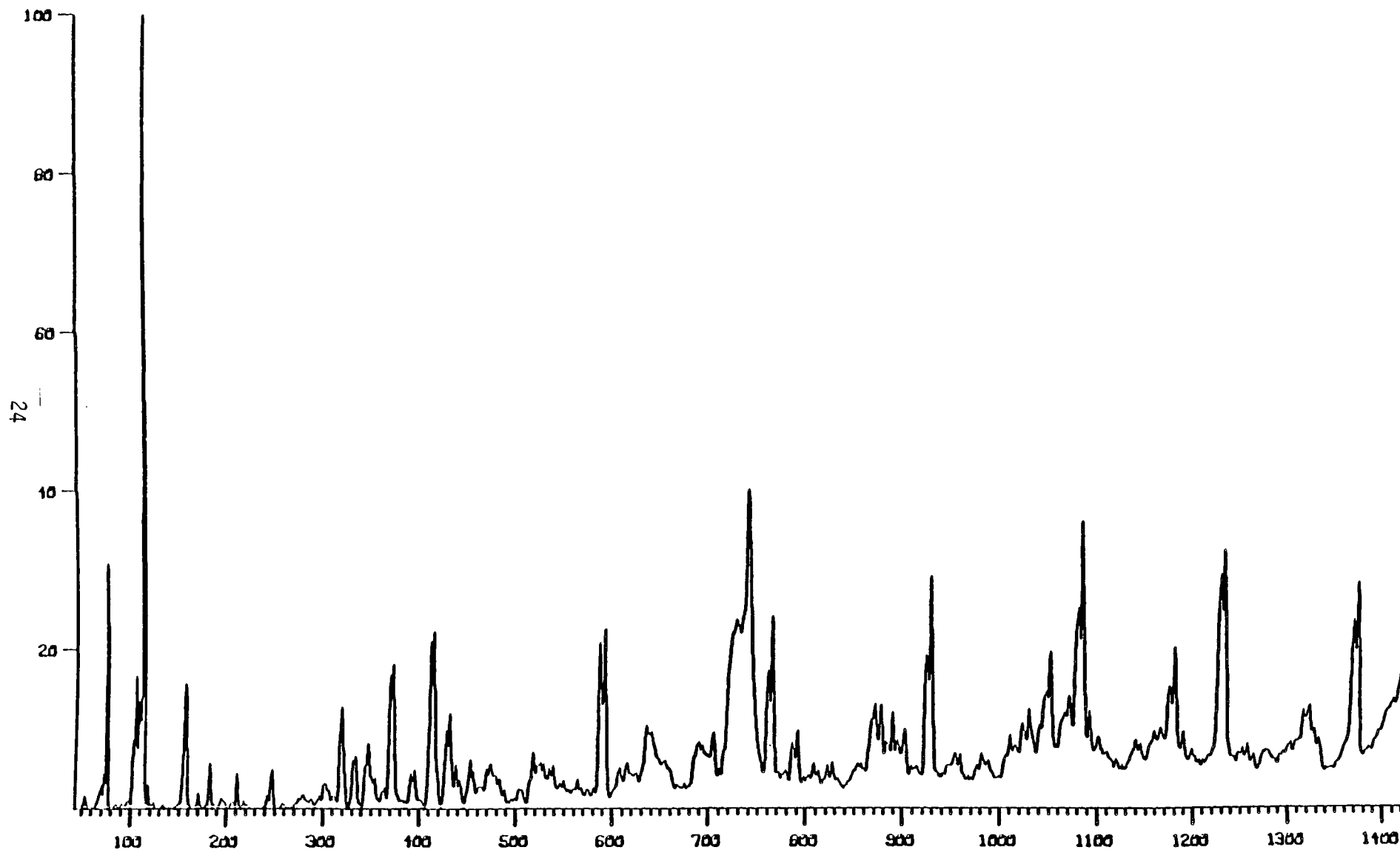


Fig. 4. Total Ion Chromatogram of 8/3/76 H-1 Neutral Extract

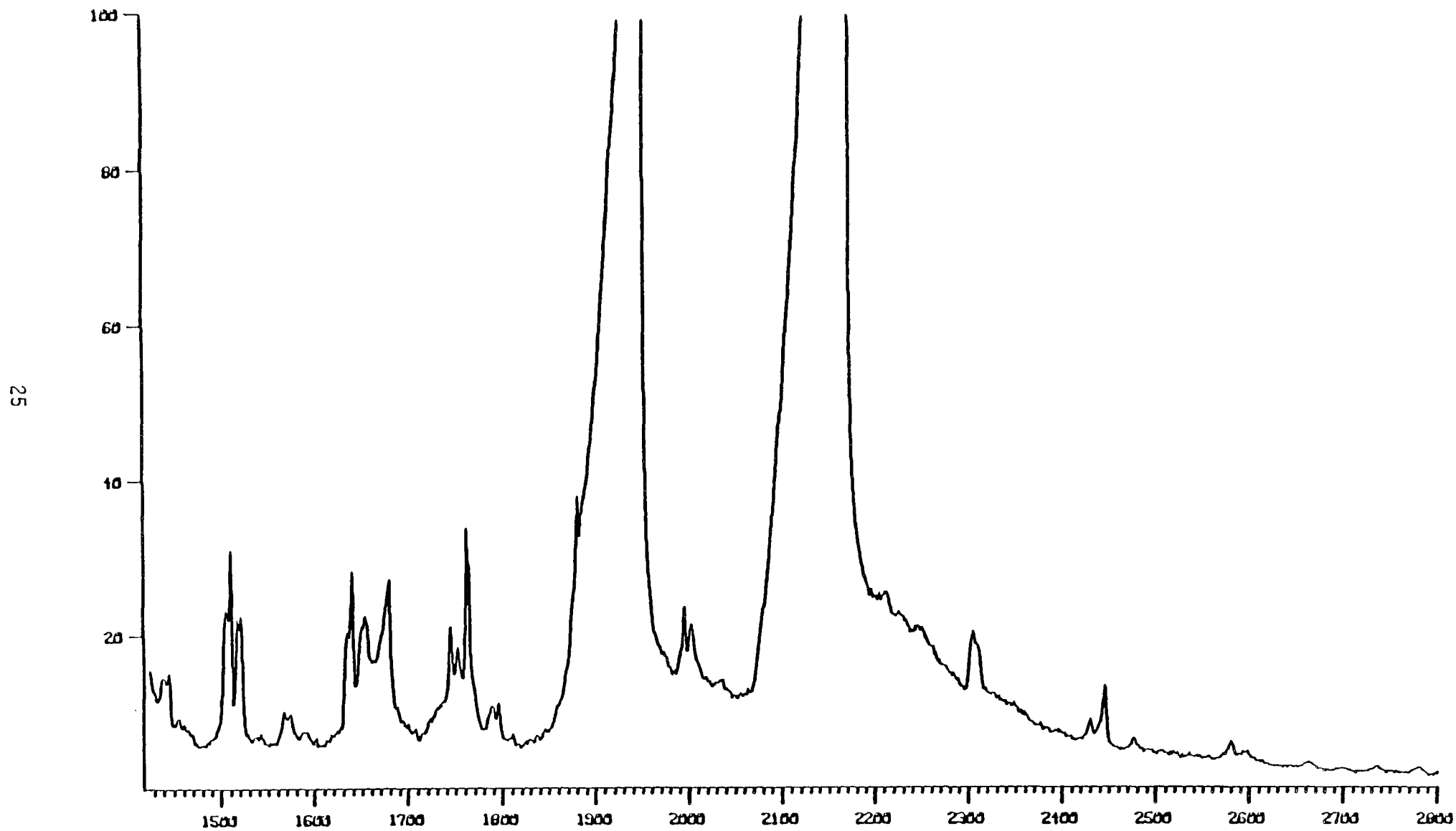


Fig. 4 (Cont'd). Total Ion Chromatogram of 8/3/76 H-1 Neutral Extract

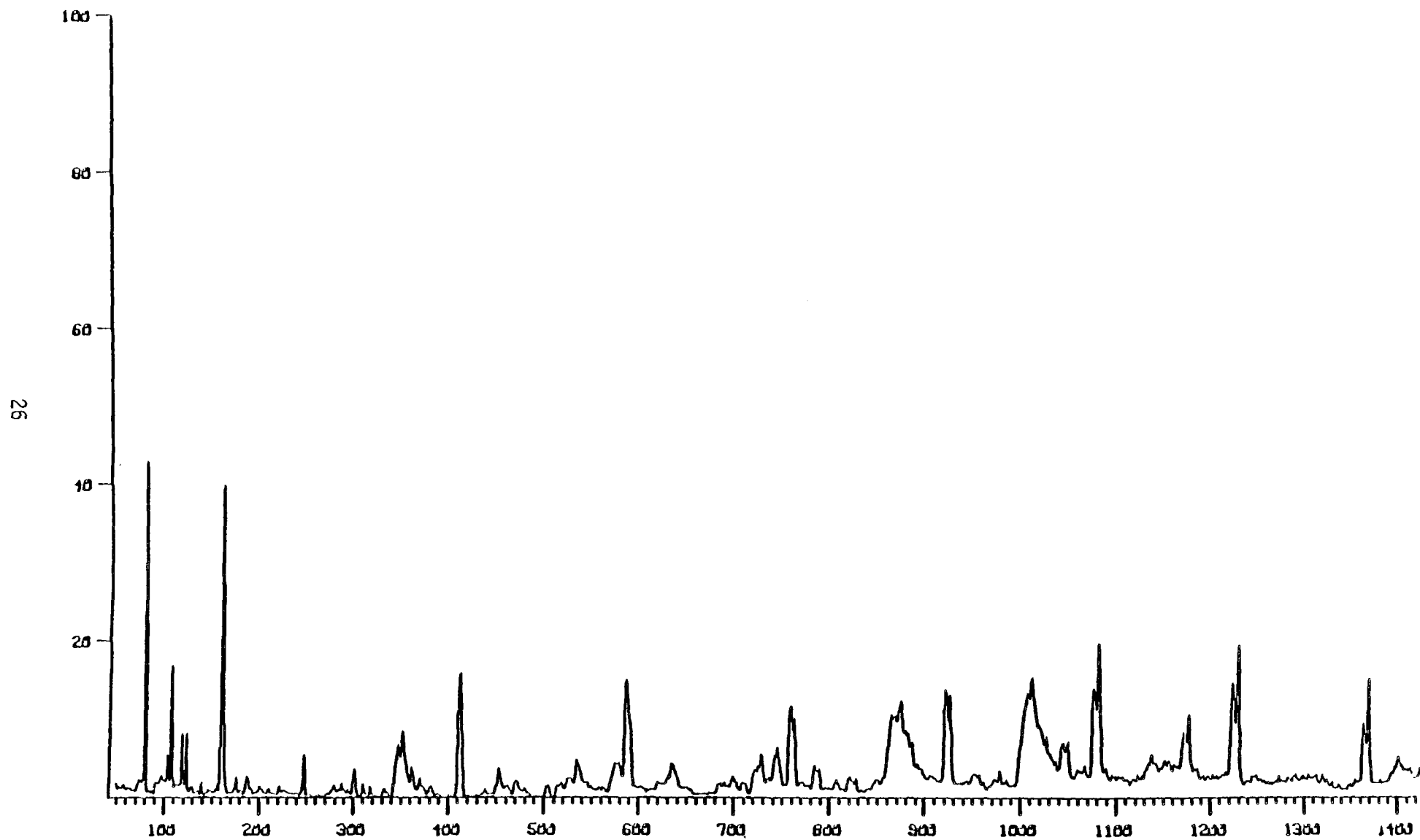


Fig. 5. Total Ion Chromatogram of 8/3/76 H-1 Neutral Extract

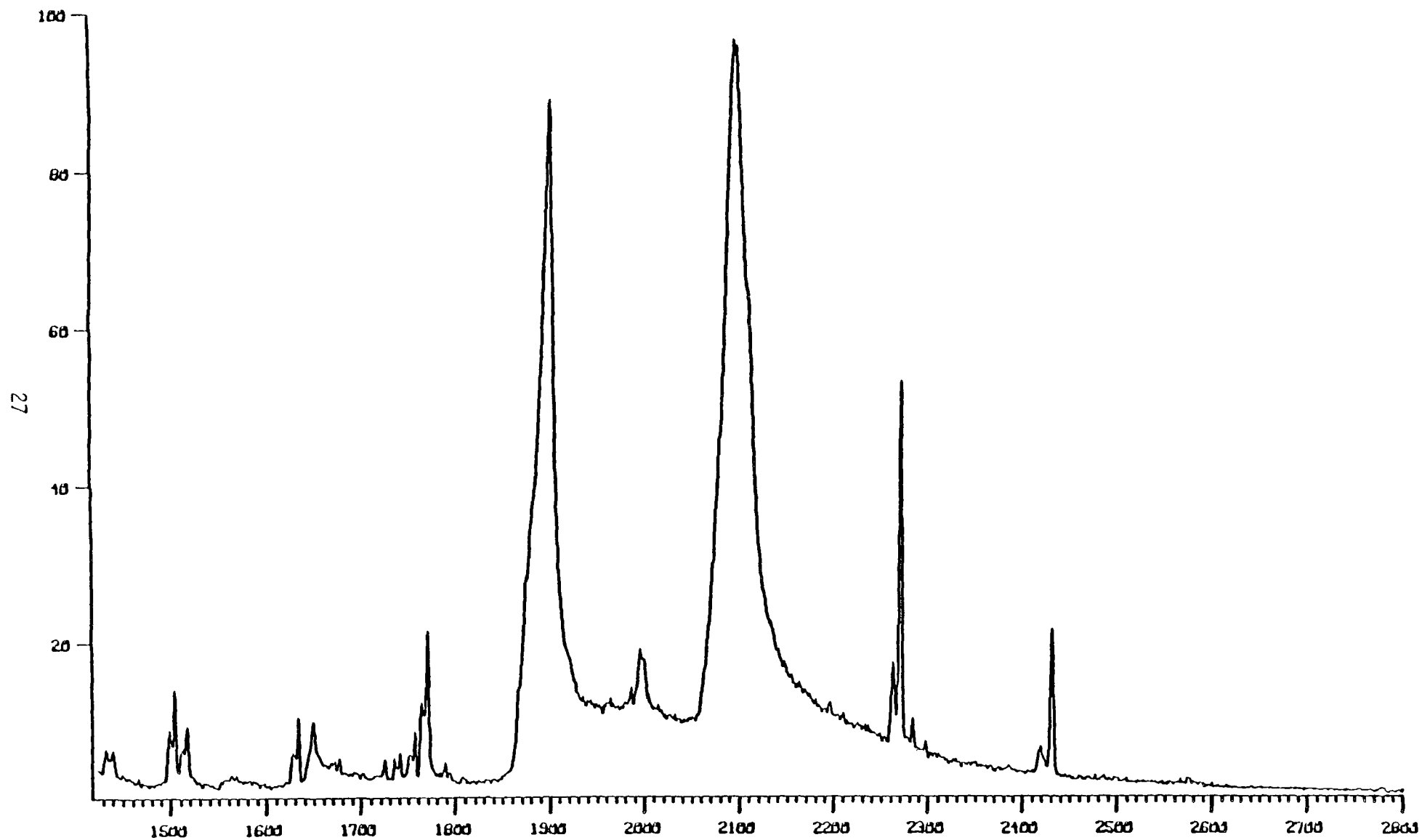


Fig. 5 (Cont'd). Total Ion Chromatogram of 8/3/76 H-1 Neutral Extract

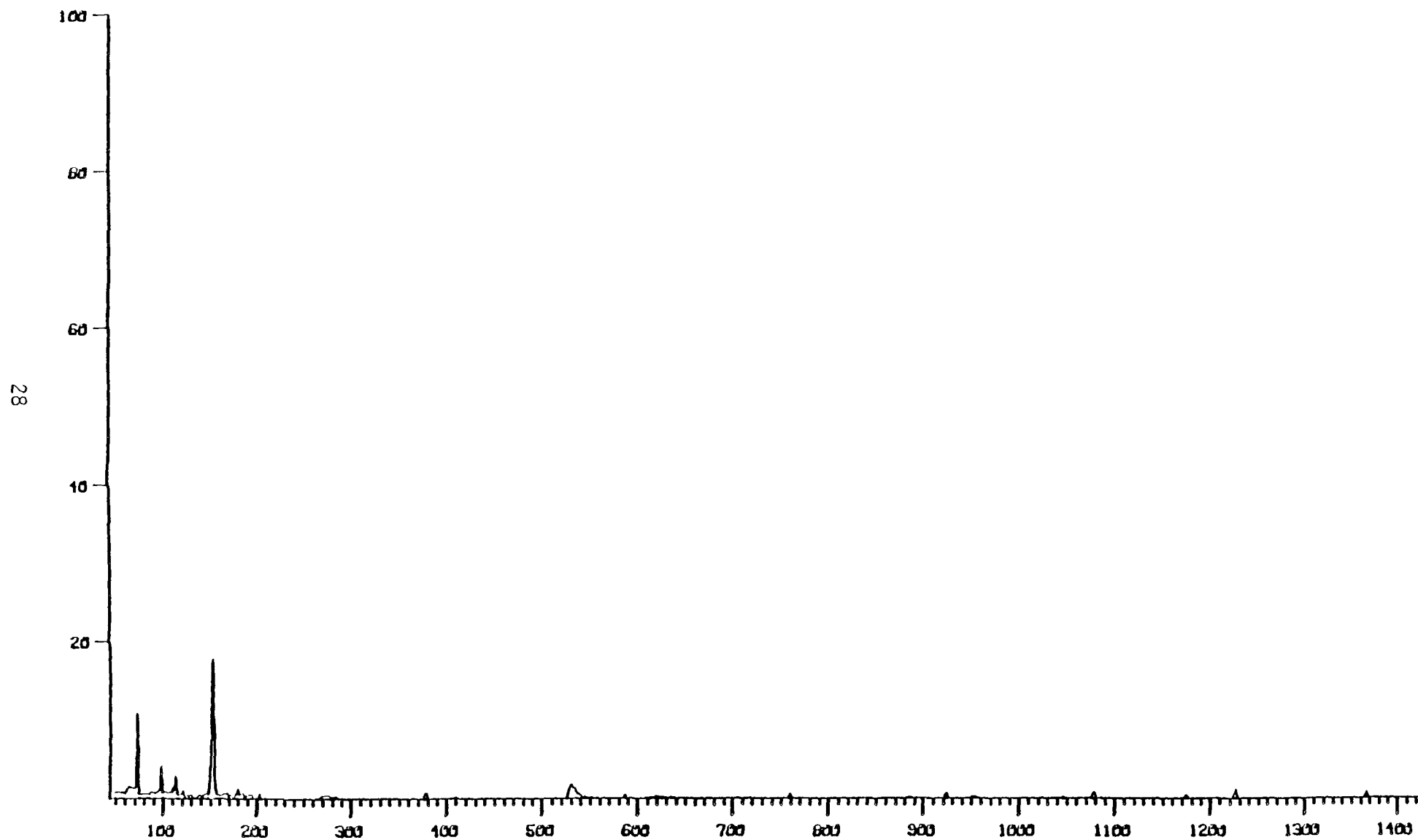


Fig. 6. Total Ion Chromatogram of 8/3/76 H-1 Base Extract

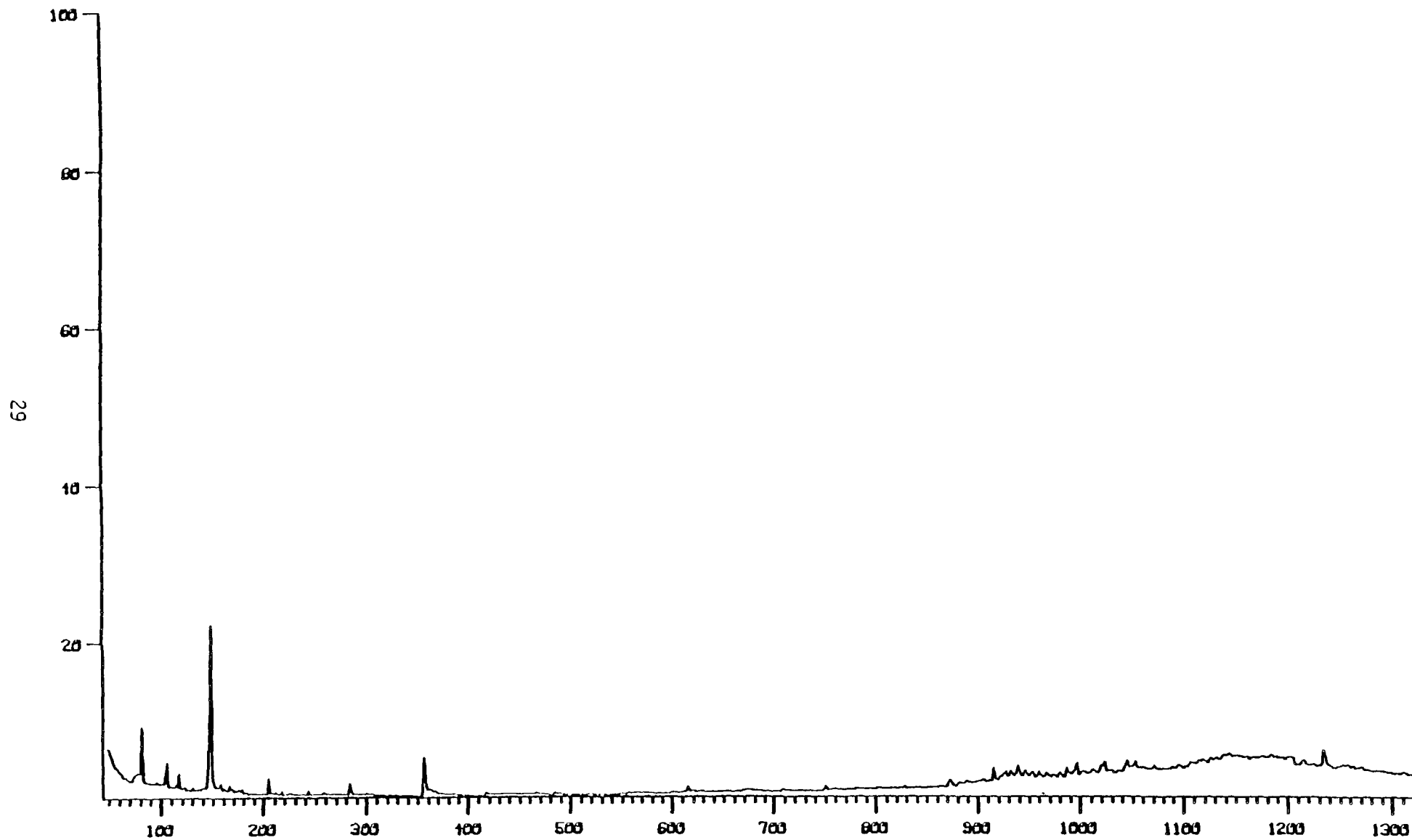


Fig. 7. Total Ion Chromatogram of 8/3/76 I-7 Neutral Extract



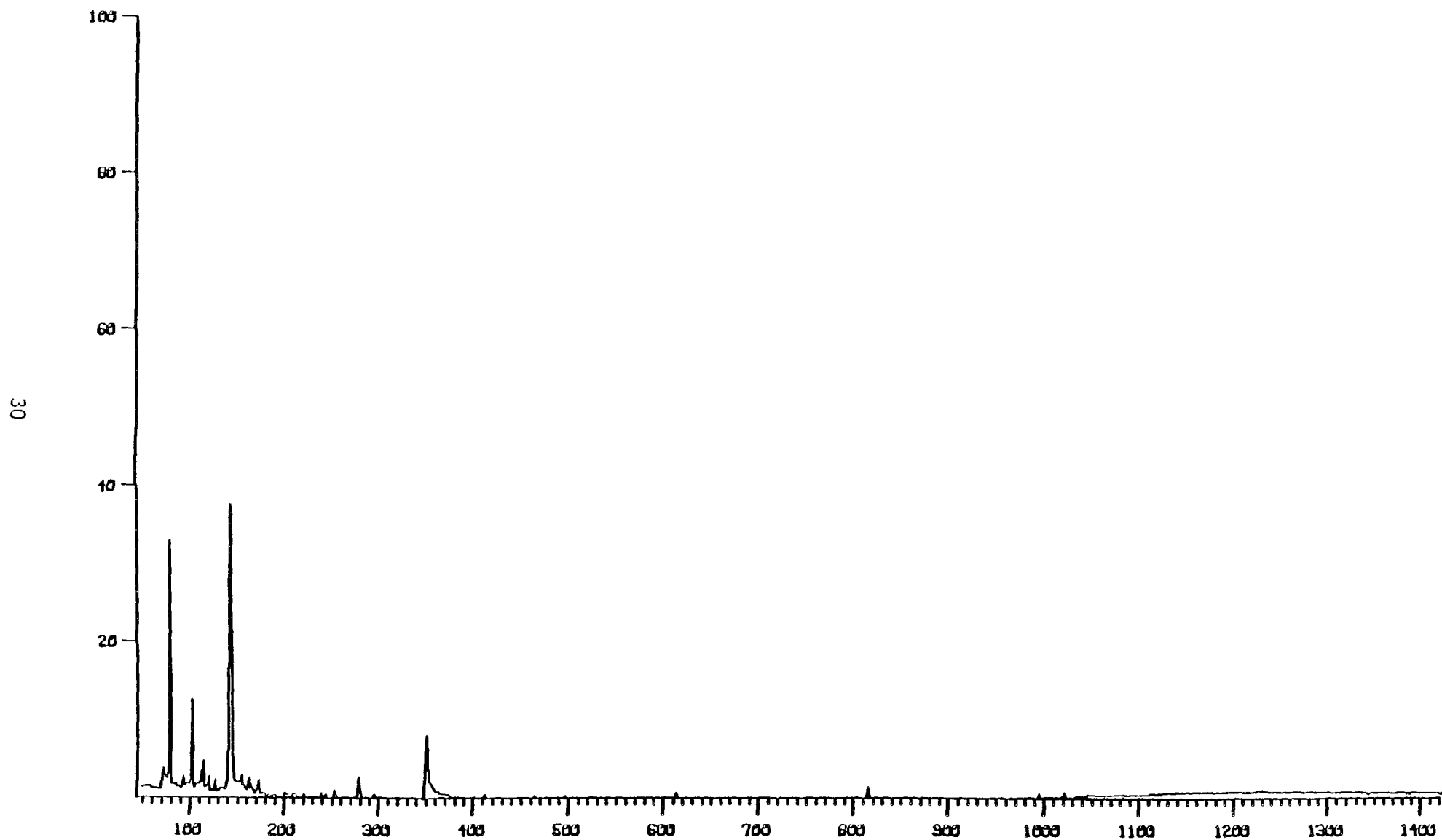


Fig. 8. Total Ion Chromatogram of 8/3/76 I-7 Acid Extract

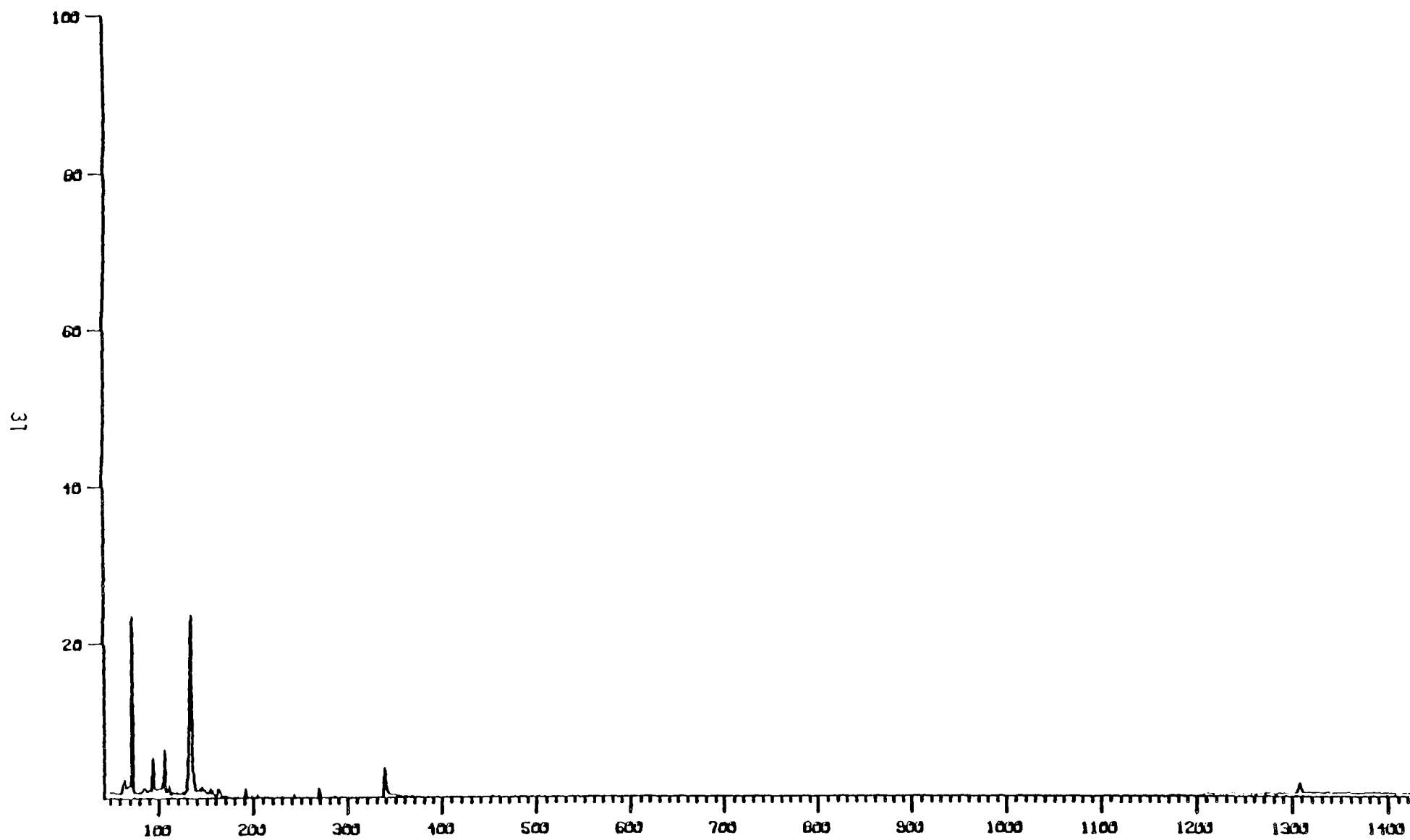


Fig. 9. Total Ion Chromatogram of 8/3/76 I-7 Base Extract

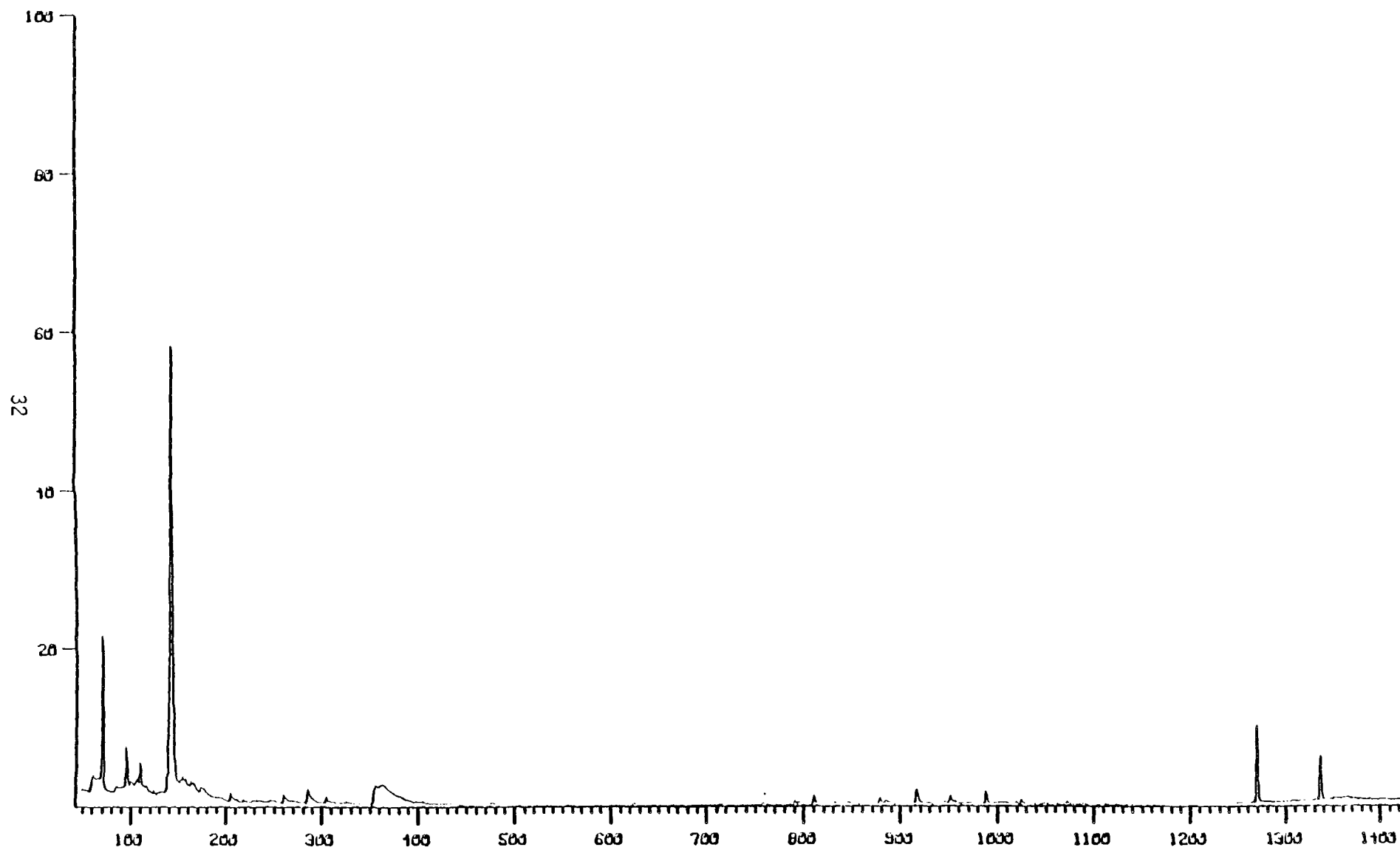


Fig. 10. Total Ion Chromatogram of 11/4/76 K-7 Neutral Extract.

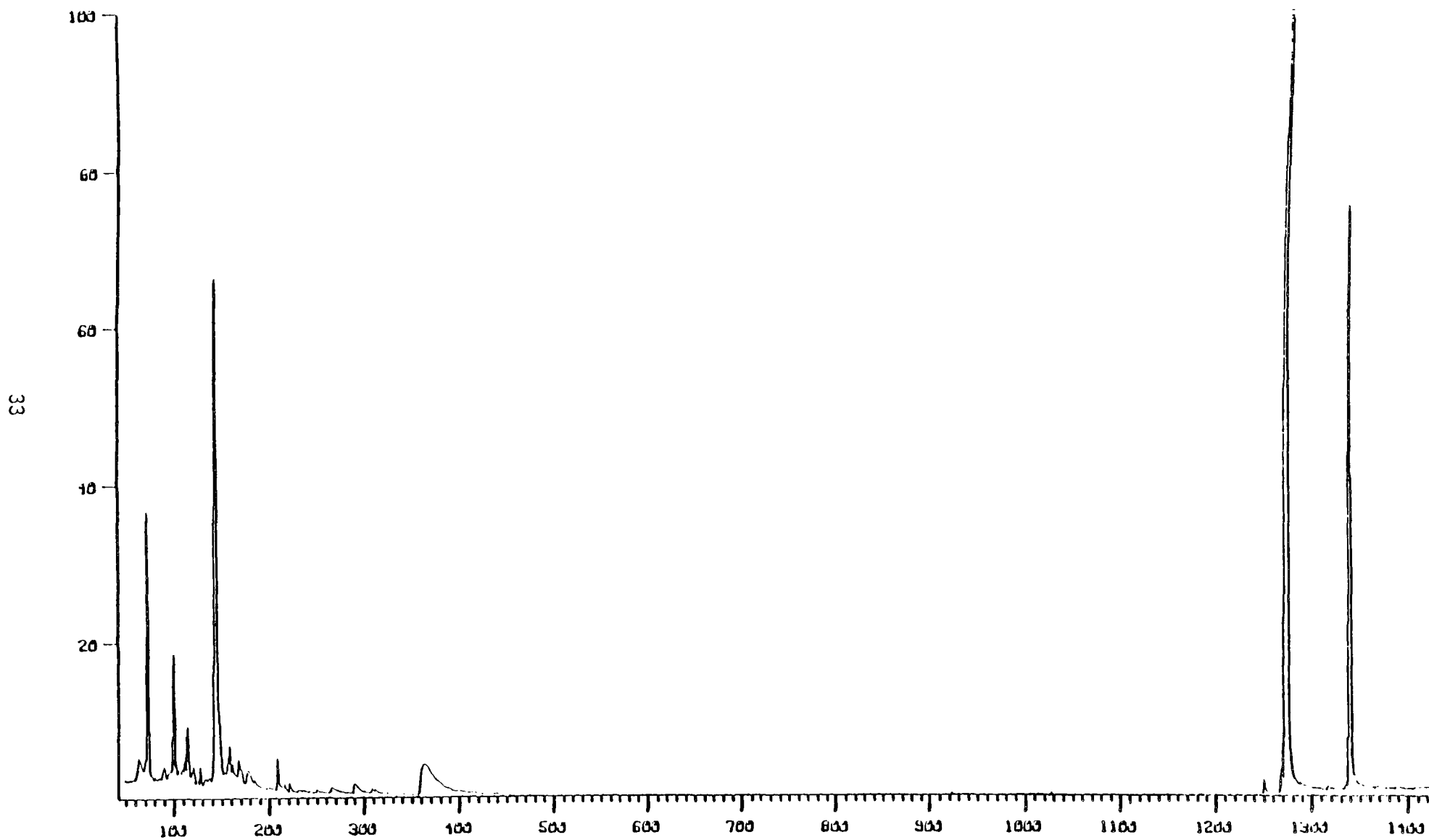


Fig. 11. Total Ion Chromatogram of 11/4/76 K-7 Acid Extract

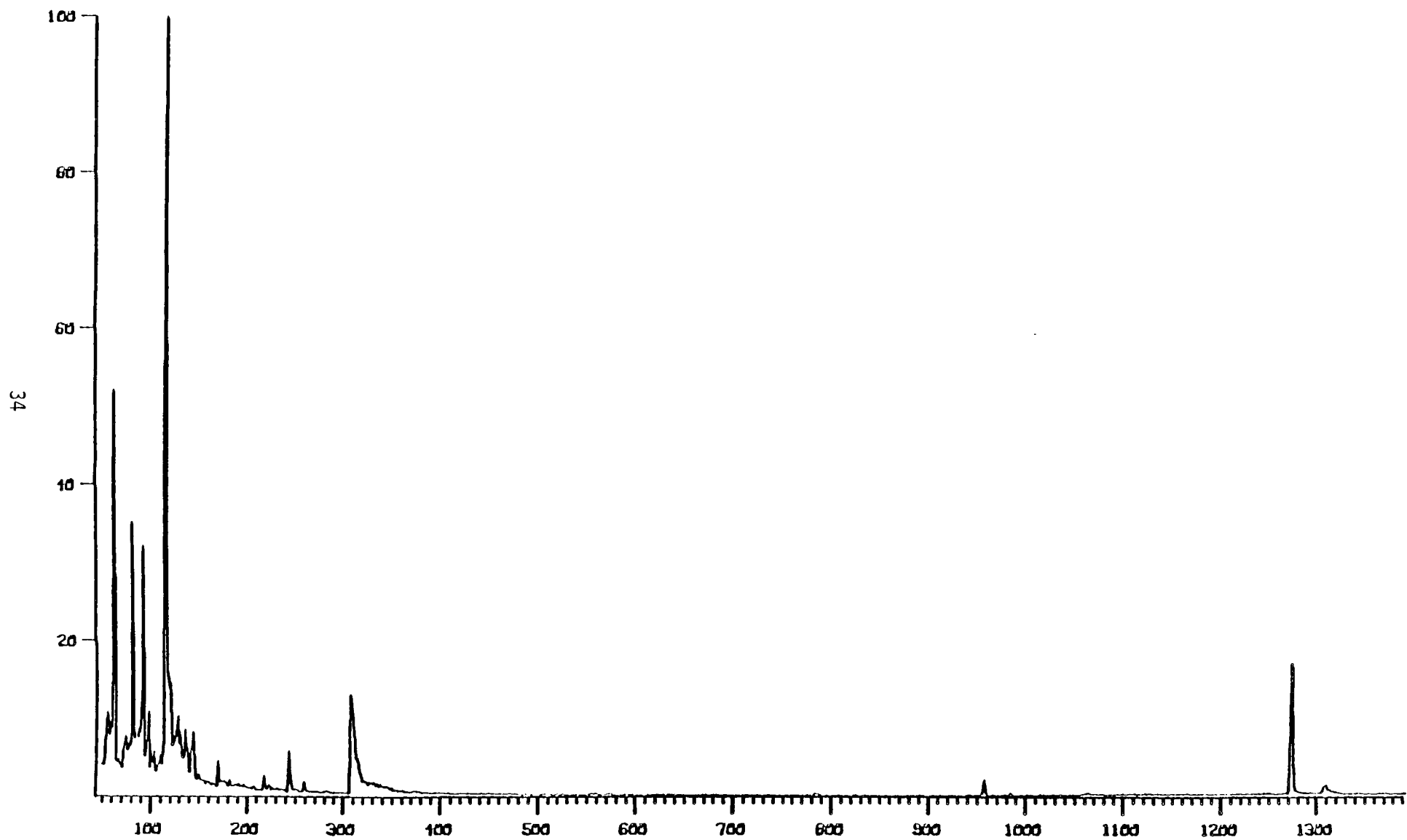


Fig. 12. Total Ion Chromatogram of 11/4/76 K-7 Base Extract

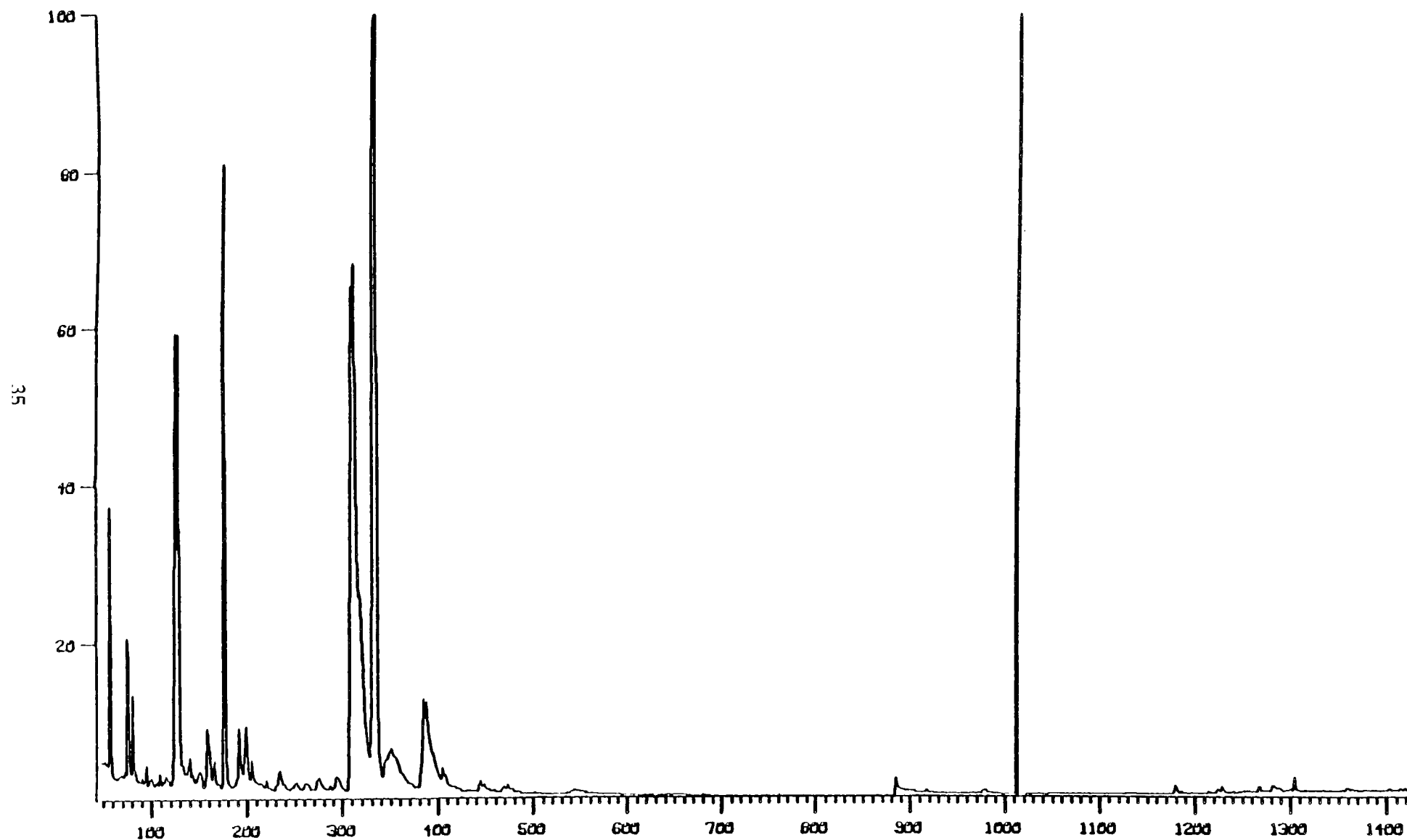


Fig. 13. Total Ion Chromatogram of 11/4/76 L-7 Neutral Extract

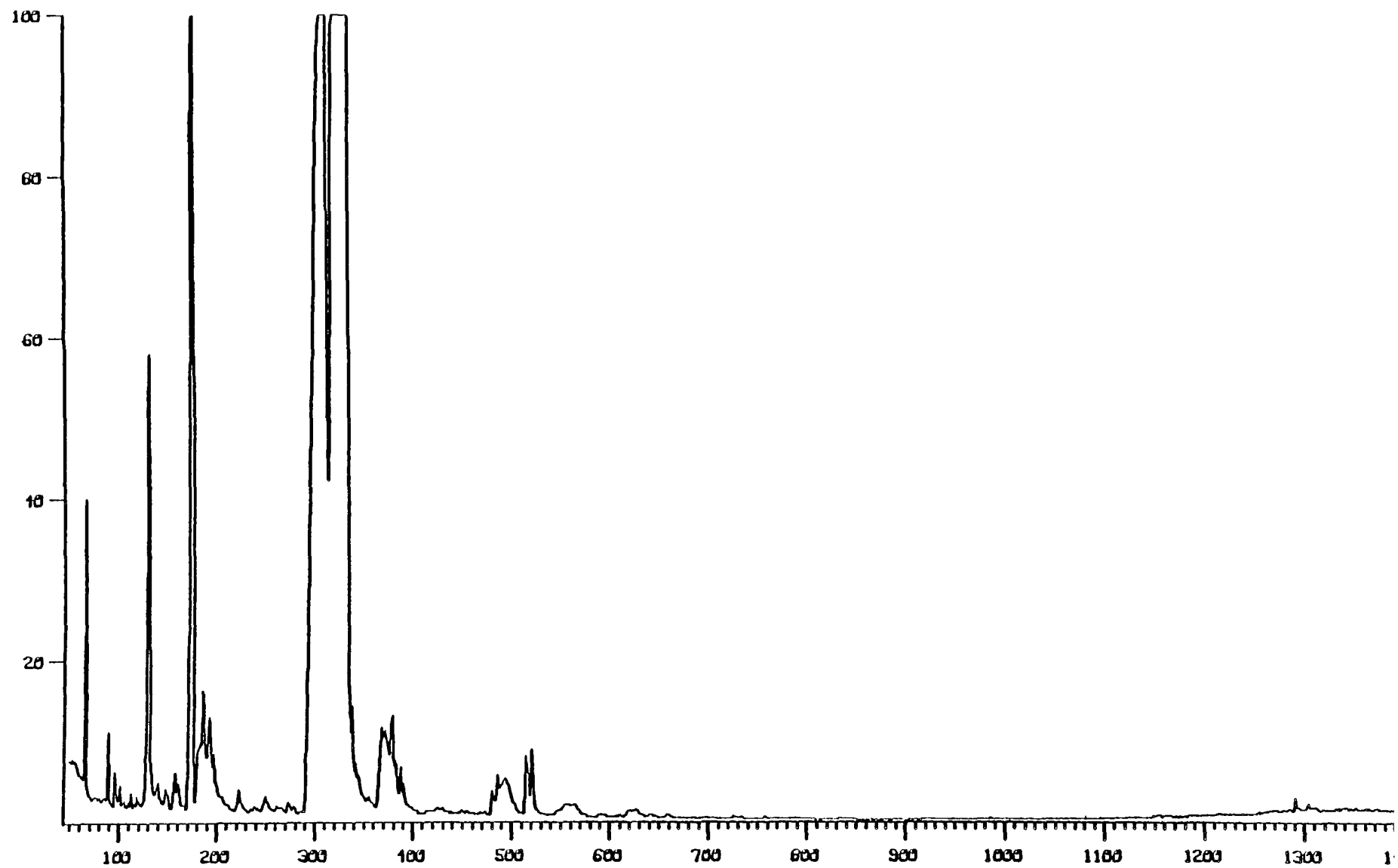


Fig. 14. Total Ion Chromatogram of 11/4/76 L-7 Acid Extract

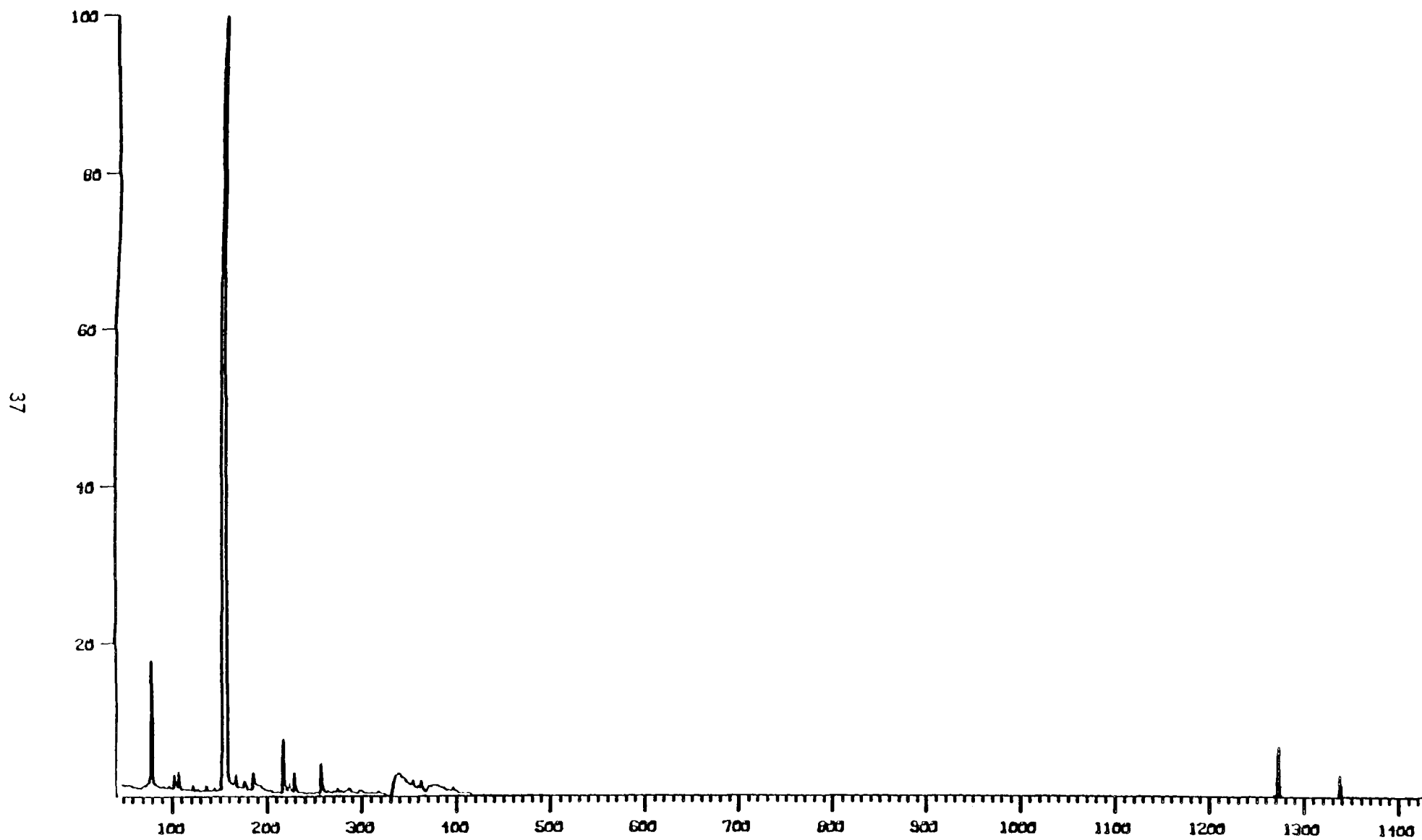


Fig. 15. Total Ion Chromatogram of 11/4/76 L-7 Base Extract



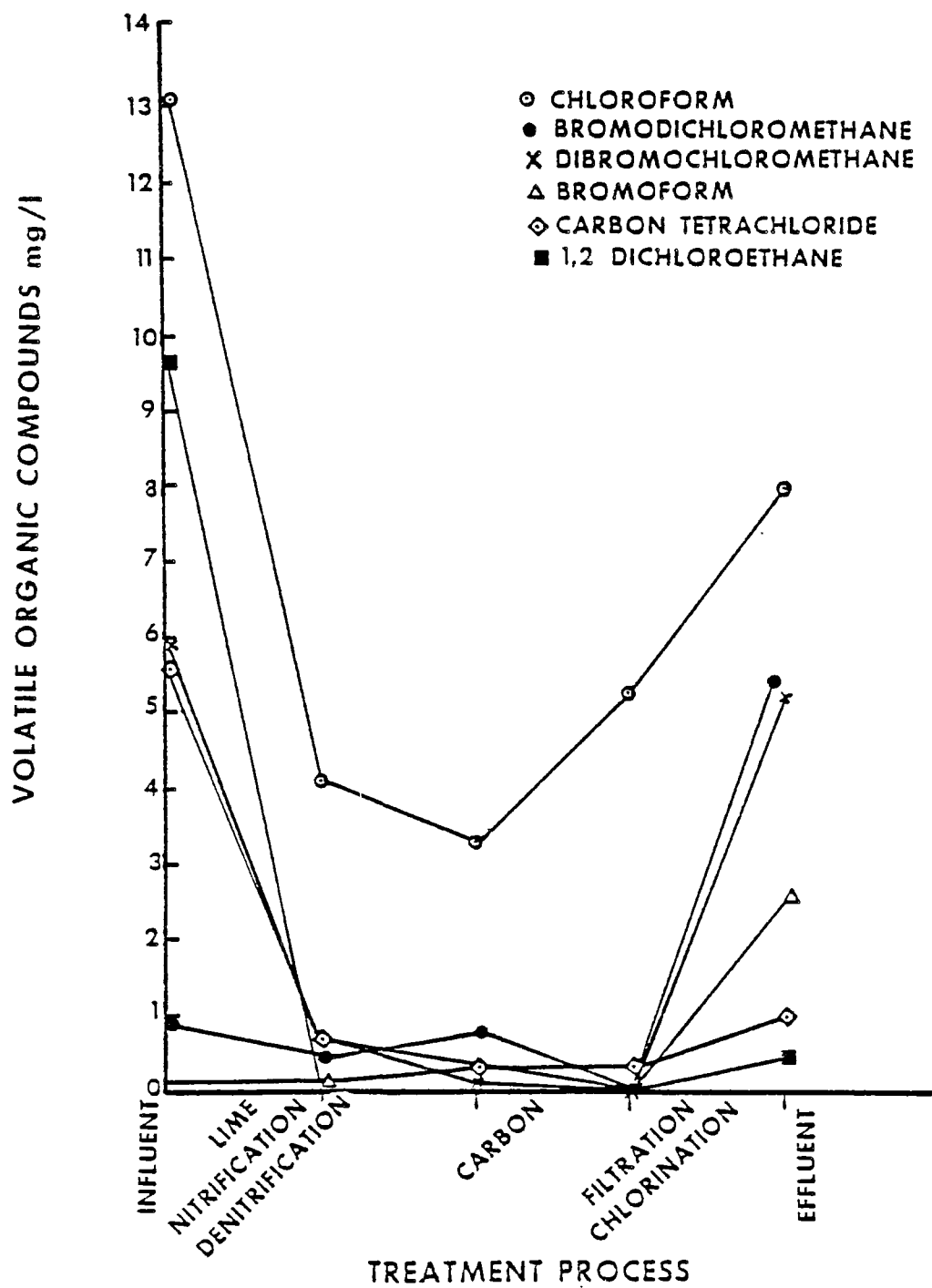


Fig. 16. Removal of Volatile Organics