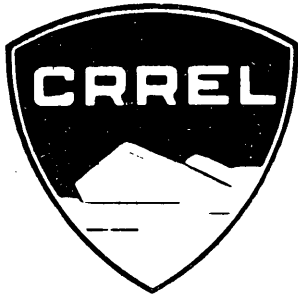


SR 209



Special Report 209

**IDENTIFICATION OF SOIL ORGANICS USING A
GAS CHROMATOGRAPHIC/
MASS SPECTROMETRIC METHOD**

William F. O'Reilly and Richard P. Murrmann

June 1974

**PREPARED FOR
DIRECTORATE OF MILITARY ENGINEERING AND TOPOGRAPHY
OFFICE, CHIEF OF ENGINEERS
DA PROJECT 4A162121A894**

BY

**CORPS OF ENGINEERS, U.S. ARMY
COLD REGIONS RESEARCH AND ENGINEERING LABORATORY
HANOVER, NEW HAMPSHIRE**

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PREFACE

This report was prepared by 1LT W.F. O'Reilly, Chemist, and Dr. R.P. Murrmann, Research Chemist, Earth Sciences Branch, Research Division, U.S. Army Cold Regions Research and Engineering Laboratory. The work was conducted under DA Project 4A162121A894, *Engineering in Cold Environments*, Task 02, *Expedient Defensive and Protective Structures in Cold Regions*, Work Unit 001, *Expedient Roads, Airfields and Heliports in Cold Regions*. The report was technically reviewed by Dr. Pieter Hoekstra and James Cragin of USA CRREL.

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Introduction

A requirement exists for stabilization of cold regions soils after both expedient and permanent construction to minimize erosion while revegetation takes place. One approach to this problem is the use of organic chemical additives. In the past, however, use of these additives for soil stabilization has met with limited success. Selection of the proper additives has been based primarily on empirical observations rather than on knowledge of the reactions of organic chemicals with soil constituents. Our understanding of organic reactions with soils in relation to soil physical properties can be increased through determining the role of naturally occurring organics in soil aggregate formation. Development of techniques for identification of soil organics is a first step in addressing this topic.

Pyrolysis has been used as a technique to determine the identity of organic constituents in soils^{6 9 14 15 23 28 29 30} as well as in extraterrestrial samples.^{1 2 5 13 22 24 26 27} In most investigations, a pyrolyzer has been interfaced directly to either a gas chromatograph^{9 14 15} or a mass spectrometer.⁶ The most powerful method utilizing pyrolysis for characterization of organics in soils is the use of a combined gas chromatograph/mass spectrometer system^{23 30} coupled with an on-line computer for data analysis.²³

Pyrolysis has been applied to the analysis of soil organic matter due to the large preponderance of high molecular weight compounds and the difficulties encountered in using other methods such as extraction,^{3 4 29} hydrolysis,²⁹ and reduction with sodium amalgam.^{3 4} Most investigations have been undertaken using cleavage products to attempt to identify the building blocks of humic material^{9 11 12 14 15 20 21 23 25 29 34} while less attention has been given to the direct analysis of free, low molecular weight organics.^{10 16 17 19 32 33}

Pyrolysis at high temperatures (700° - 900°C) degrades the humic material perhaps even more than the other methods mentioned. From the pyrolysis products formed it is sometimes possible to recreate the structure of organic material³⁰ but it is difficult to distinguish low molecular weight organic compounds originally present from pyrolysis products. With the use of a lower temperature (100° - 200°C) it should be possible to volatilize the low to medium molecular weight organic molecules for direct identification without cleaving higher molecular weight compounds. Increasing temperature increases the kinetic energy of adsorbed molecules, thus weakening the interaction with the soil. Consequently, when an inert carrier gas sweeps through the sample, the volatilized organics can be transferred from the soil to a collection trap. In this approach temperature control is critical. If the sample temperature is too low, the amounts of the organics volatilized and collected are insufficient to permit identification. If the sample is overheated, pyrolysis results, masking the presence of the free organic compounds in the soil.

This study was undertaken in order to determine if volatilization combined with gas chromatography/mass spectroscopy could be used as a tool for the analysis of free, low molecular weight organics in soil. The first step was the determination of the maximum temperature which could be used before significant amounts of pyrolysis products were observed. Three temperatures were chosen for comparison: 100°C, 150°C and 200°C. From our own experience and that of others,³² it was known that below 100°C very little sample could be collected due to interaction between organics and soil surfaces. Above 200°C, abnormally high quantities of low molecular weight hydrocarbons occur due to pyrolysis. After the appropriate temperature was selected, three soils were analyzed for their volatile organic components. During a third phase of the work, the same three soils were analyzed but an improved vapor collection procedure was utilized.

Experimental

Instrumentation. A Perkin-Elmer 270-B gas chromatograph/mass spectrometer (GC/MS) with a hydrogen flame ionization detector equipped to work concurrently with the mass spectrometer was used in this analysis. The mass spectrometer, a double-focusing instrument, was operated at 75 electron volts with the source and manifold temperature maintained at 150°C and the injection port at 200°C. Two different chromatographic columns were employed. The column used in the first two phases of the work was a 1/8 in. x 9 ft Durapak* (Carbowax 400 on Porasil C) column useful for analysis of low to medium molecular weight compounds. The second column, used in the third phase, also a Durapak type (1/8 in. x 2 ft, Carbowax 400 on Porasil F), was used for analysis of compounds in the middle molecular weight range and above. The data were processed on an on-line Digital Equipment Corporation PDP-12 computer.

Procedure. The first two phases of the work on optimum sample temperature determination and the first set of soil analyses were conducted using the following procedure. A 3-g soil sample was placed in a quartz tube (12.6 mm OD) and secured by plugs of silanized glass wool. Valves were placed on either side of the quartz tube. Temperature control was maintained using a tubular heater (Fig. 1) with a copper/constantan thermocouple placed between the heated wall and the wall of the quartz tube. All components exposed to the sample were preheated at 200°C to minimize contamination. The assembly containing the soil was flushed with helium for approximately five minutes before the sample was isolated by closing the valves. The tubular heater was set for the desired temperature (100°, 150° or 200°C) and the sample was heated for one hour after the set temperature was reached.

During the period the sample was heated, the temperature of the gas chromatograph oven was reduced to -75°C, forming a cold trap at the head of the analytical column. After one hour of heating, the quartz tube was purged with helium carrier gas for three minutes to transfer sample vapors onto the head of the Durapak column. The gas chromatograph was then programmed to increase temperature at the rate of 10°C/min from -75° to 150°C using a helium flow rate of 24 ml/min at -75°C.

The effluent from the chromatographic column was split 70/30 into the mass spectrometer (MS) and the hydrogen flame ionization detector (FID). As chromatographic peaks were noted on the FID chromatogram, mass spectra were obtained using the manual scanning mode, and the data were obtained and stored using the PDP-12 computer (Fig. 2). Compounds were identified by comparison with standard fragmentation patterns⁷ and by gas chromatographic retention times.

The procedure used for the third phase of the study varied from the original procedure in the following ways. The soil sample was secured as before in the quartz tube and heated to the same final temperature, but the helium carrier gas was allowed to pass continuously over the soil as it was heated. This permitted transfer of organics from the heated zone immediately upon volatilization, minimizing the possibility of decomposition. Although relatively larger quantities of water

*Waters Assoc., Inc., Framingham, Mass.

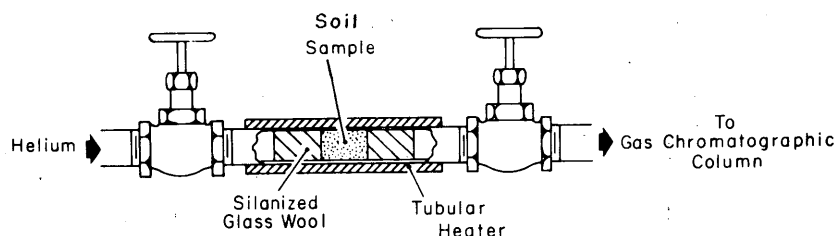


Figure 1. Sample holder and inlet to gas chromatograph.

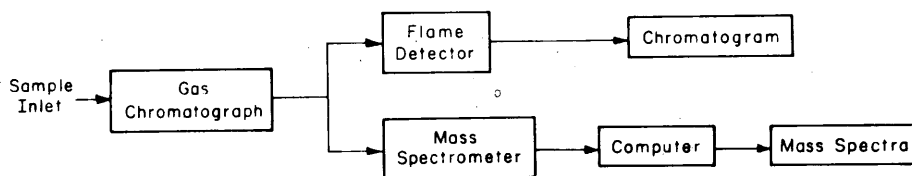


Figure 2. Block diagram of gas chromatograph/mass spectrometer/computer system.

were removed from the soil, water was not significantly retained by the collection trap used. The collection trap in this case was not the cold head of the chromatographic column but a gas adsorption tube.^{8, 18} The adsorption tube contained Chromasorb 102,* a porous polymeric material used in gas chromatographic columns. The adsorption tube was attached directly to the outlet of the quartz tube. The volatilized sample was collected for one hour at the desired temperature. The soil organics concentrated in the adsorption tube were then transferred to the cold chromatographic column by backflushing the tube heated to 120°C with helium carrier gas.

Results and discussion

Fairbanks silt was used in the first experiment to determine an optimum temperature for volatilization of soil organics. The chromatograms obtained are illustrated in Figure 3 in which peak numbers correspond to organic compounds identified in Table I. For the soil heated at 100°C, small amounts of ethylene, propylene and butene were found. Low molecular weight compounds such as these are known^{17, 26, 31, 32, 33} to exist in soils at low levels. For example, ethylene results from anaerobic microbiological activity.³² However, the presence of large amounts of these compounds is an indicator of pyrolysis. Several other organics, mainly hydrocarbons, were identified. The baseline drift at long retention time is due to the presence of water. At 200°C, both the amounts and number of identifiable compounds increased considerably in comparison with the other treatment temperatures but the low molecular weight unsaturated hydrocarbons such as ethylene, propylene and butenes have increased over what would be expected. The abnormal amount of methane present also indicates that pyrolysis of the complex organics present in the soil occurred. Evidently, at 200°C higher molecular weight compounds were broken down into toluene, benzene, lower paraffins, olefins, and methane. Usually, pyrolysis studies are conducted in the 700° to 900°C temperature range,^{13, 14, 15} however, as may be seen in Figure 3, even 200°C is sufficient to cause substantial pyrolysis. The chromatogram obtained at 150°C shows the presence of low molecular weight hydrocarbons but the amounts of these compounds seem to be in reasonable proportion to other types of organics; thus, the degree of pyrolysis must have been minimal. The analysis resulted in identification of 28 organic compounds. A temperature of 150°C was selected as most suitable for subsequent work.

*Johns-Manville, Framingham, Mass.

IDENTIFICATION OF SOIL ORGANICS

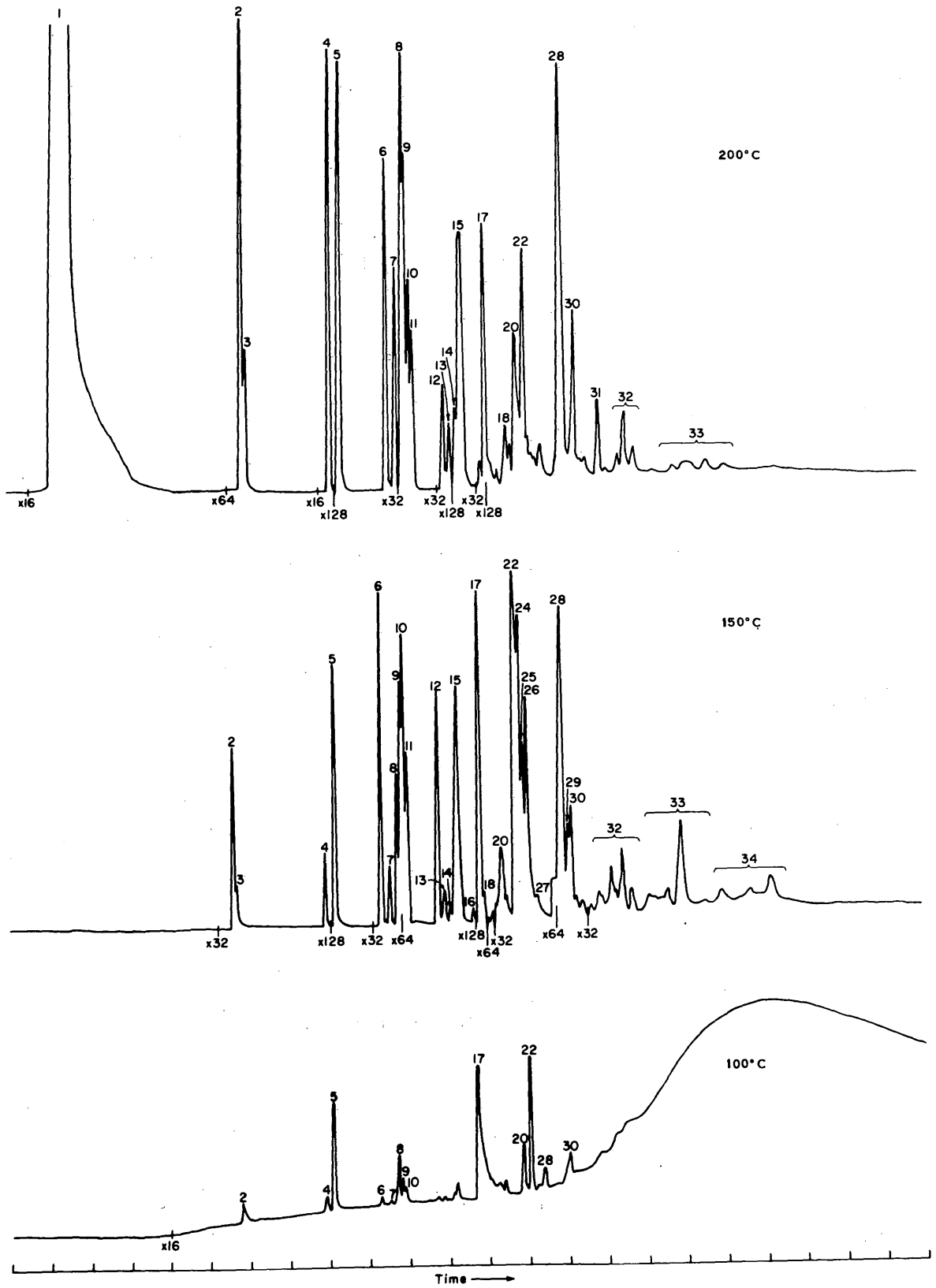


Figure 3. Gas chromatograms obtained for vapors evolved from Fairbanks silt maintained at temperatures of 100°C, 150°C, and 200°C.

Table I. Organic compounds evolved from Fairbanks silt maintained at three different temperatures.

Peak number*	Compound	Temperature† (°C)		
		100	150	200
1	Methane			●
2	Ethylene	●	●	
3	Ethane		●	●
4	Propane	●	●	●
5	Propene	●	●	●
6	Isobutane	●	●	●
7	N-Butane	●	●	●
8	Butene	●	●	●
9	Butene	●	●	●
10	Butene	●	●	●
11	Butadiene		●	●
12	Isopentane		●	●
13	N-Pentane		●	●
14	Pentene		●	●
15	Pentadiene		●	●
17	Acetaldehyde	●	●	●
18	C ₆ -C ₇ Hydrocarbon		●	●
20	C ₆ -C ₇ Hydrocarbon	●	●	●
22	Benzene	●	●	●
24**	Trichloroethylene		●	
25	C ₈ -C ₉ Hydrocarbon		●	
26	C ₈ -C ₉ Hydrocarbon		●	
27	C ₈ -C ₉ Hydrocarbon		●	
28	Acetone	●	●	●
29	C ₉ Hydrocarbon		●	
30	Toluene	●	●	●
31	C ₁₀ Hydrocarbon		●	●
32	Xylenes + C ₁₀ Hydrocarbons		●	●
33	C ₃ Aromatics		●	●
34	C ₄ Aromatics		●	

*Correspond to numbered chromatographic peaks in Figure 3.

†● indicates presence of corresponding compound.

**Present as contaminant.

The soils utilized in the second phase included Fairbanks silt, Manchester silt and Suffield clay, each heated at 150°C. The resulting chromatograms for each soil are shown in Figure 4. Compounds corresponding to the chromatographic peaks are listed in Table II. Approximately the same number of compounds were identified in the vapor from each of the three soils; there were no unusual compounds found in any single soil. There seem to be smaller quantities of organics eluted from the Manchester silt than from either the Suffield clay or the Fairbanks silt. The baseline drift seen in Figure 4 is due to the water released from the soil during heating. The presence of water undoubtedly limited the analysis of higher molecular weight compounds. The compounds identified varied in molecular weight range from ethane and ethylene to C₉ and C₁₀ hydrocarbons and C₃-C₄ alkylated aromatics. The most intense peaks corresponded to propylene, acetaldehyde and acetone.

The third phase was attempted in order to eliminate the baseline drift problem caused by water, to minimize the possibility of pyrolysis, and to increase the range of identifiable compounds.

IDENTIFICATION OF SOIL ORGANICS

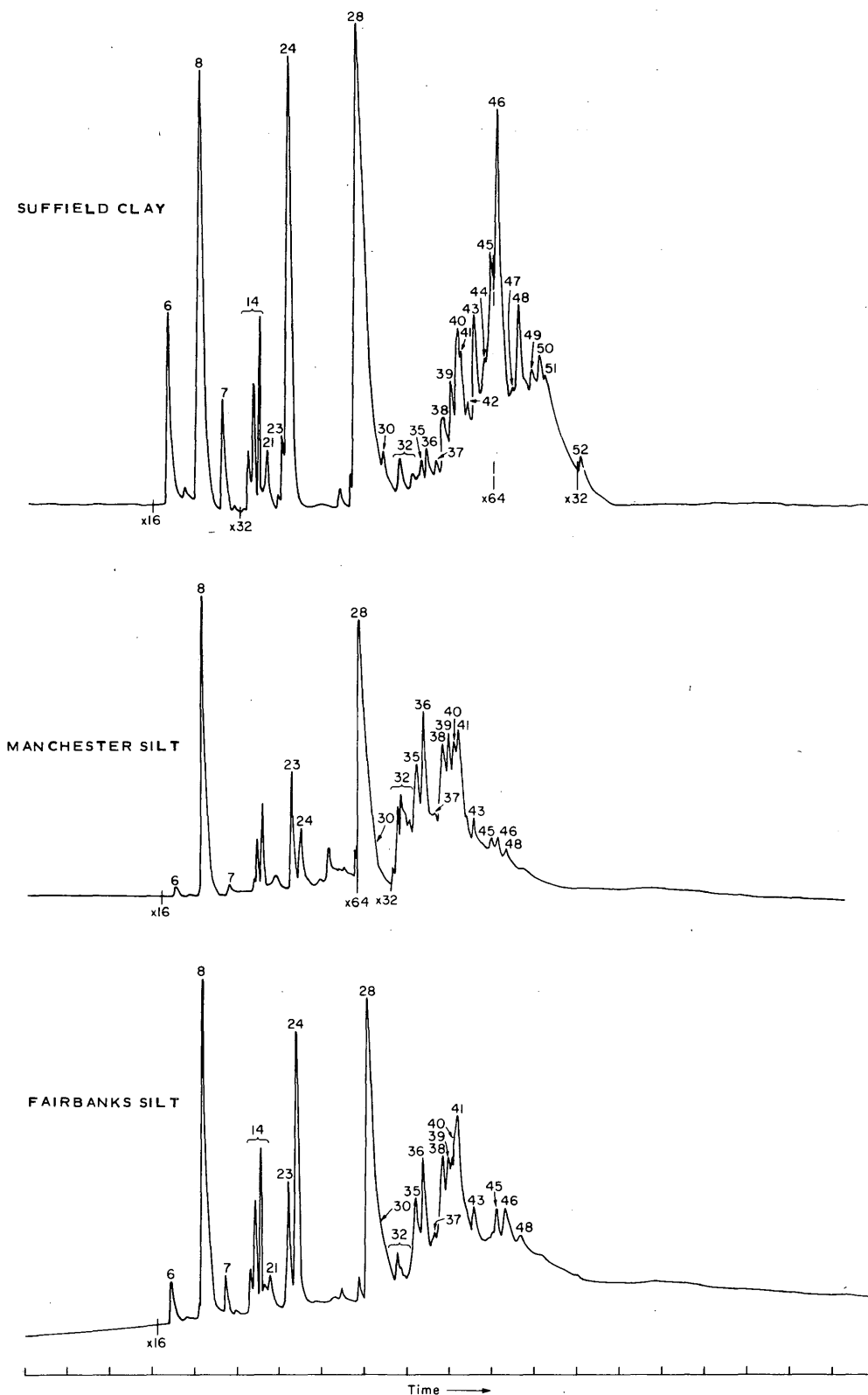


Figure 5. Gas chromatograms of vapors evolved from three soils maintained at a temperature of 150°C using the modified sample collection technique.

Table III. Compounds identified in vapors evolved from three soils heated at 150°C using modified sample collection technique.

Peak number*	Compound	Soil type†		
		Fairbanks silt	Manchester silt	Suffield clay
6	Isobutane	●	●	●
7	N-Butane	●	●	●
8	Butene	●	●	●
14	Pentene	●	●	●
21	Cyclohexene	●	●	●
23	Chloroform	●	●	●
24**	Trichloroethylene	●	●	●
28	Acetone	●	●	●
30	Toluene	●	●	●
32	Xylenes + C ₁₀ Hydrocarbon	●	●	●
35	Trimethylbenzene	●	●	●
36	C ₁₀ -C ₁₄ Hydrocarbon	●	●	●
37	C ₁₀ -C ₁₄ Hydrocarbon	●	●	●
38	Unknown	●	●	●
39	C ₁₀ -C ₁₄ Hydrocarbon	●	●	●
40	Naphthalene	●	●	●
41	Benzaldehyde	●	●	●
42	C ₁₀ -C ₁₄ Hydrocarbon	●	●	●
43	C ₁₀ -C ₁₄ Hydrocarbon	●	●	●
44	C ₁₀ -C ₁₄ Hydrocarbon	●	●	●
45	Methylnaphthalene	●	●	●
46	C ₁₀ -C ₁₄ Hydrocarbon	●	●	●
47	Acenaphthalene	●	●	●
48	Dimethylnaphthalene	●	●	●
49	Dibenzofuran	●	●	●
50	C ₁₀ -C ₁₄ Hydrocarbon	●	●	●
51	Ethylmethylnaphthalene	?	●	●
52	Unknown	●	●	●

*Corresponds to numbered chromatographic peaks in Figure 5.

†● indicates presence of corresponding compound.

**Present as contaminant.

In comparing the two techniques, it would appear that there is an advantage to using the continuous flow method as opposed to heating the soil in a closed system to generate samples of adsorbed organics. The continuous flow technique permitted collection, separation and identification of some 28 compounds generally in a higher molecular weight range than found by the closed system technique. Using both methods, about 50 compounds were identified. While it cannot be ascertained that these compounds were originally present in the soil, conditions were favorable for minimizing pyrolysis effects. Thus, this type of approach should be useful in identification of soil organics which are sufficiently volatile for direct analysis using gas chromatographic techniques.

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13. ABSTRACT
As part of a study on the role of natural organics in determining the physical properties of soils, the use of combined gas chromatographic/mass spectrometric methods for identification of the more volatile organic chemicals in soils were explored. Soil was first treated at 100°, 150°, and 200°C to select the optimum temperature where minimum pyrolysis of soil organic matter occurred. The vapors from three soils heated in a closed system at 150°C were then analyzed. Subsequent refinement in technique involved collection of vapor from the three soils maintained at 150°C using a constant flow sample holder/inlet system. About 50 organic compounds were identified. While application of this approach is restricted to the volatile compounds in the lower molecular weight range, conditions of analysis are thought to be suitable for identification of compounds originally present as opposed to pyrolysis products of soil organic matter.

14. Key Words

Gas chromatography	Soil chemistry
Mass spectroscopy	Soil properties
Soil analysis	Soil tests