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Background Ch	3
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Gas Chromatographic Analysis of Soil Atmospheres[1]

A. M. BLACKMER AND J. M. BREMNER[2]

ABSTRACT

A gas chromatographic procedure is described that permits rapid, specific, and precise determination of N_2, O_2, Ar, CO_2, CH_4, N_2O, and other gases in soil atmospheres. It involves use of an ultrasonic detector and two columns of Porapak Q at different temperatures, and it does not require temperature programming, column conditioning, stream splitting, column switching, or switching of recorder polarity. Unlike thermal conductivity and helium ionization detectors previously used for gas chromatographic analysis of soil atmospheres, the ultrasonic detector is not adversely affected by gases found in soil atmospheres and permits determination of these gases at concentrations ranging from <10 ng/ml to 100%.

Additional Index Words: **ultrasonic detector, soil gases, N_2, O_2, Ar, CO_2, CH_4, N_2O.**

ALTHOUGH many gas chromatographic procedures have been proposed for determination of gases in soil atmospheres (1, 2, 3, 4, 5, 6, 7, 8, 9, 10, 11, 12, 19, 21, 22, 23, 25), most permit determination of only a few of the gases of interest in research on soil atmospheres. Many of these procedures do not permit satisfactory determination of N_2 and (or) O_2, and only one (8) permits direct determination of N_2, O_2, and Ar (the three most abundant gases in well-aerated soils). Moreover, the methods that allow analysis of a single gas sample for several gases commonly found in soil atmospheres do not permit precise determination of these gases over a wide concentration range. Another disadvantage of these methods is that they involve use of thermal conductivity or helium ionization detectors, which are adversely affected by O_2 and other gases present in soil atmospheres and require frequent recalibration for precise analyses.

The purpose of this paper is to describe a gas chromatographic procedure that permits rapid, specific, and precise determination of the major constituents of soil atmospheres. This procedure involves use of an ultrasonic detector, which has important advantages over detectors previously used for gas chromatographic determination of the major constituents of soil atmospheres. These advantages include a linear dynamic range $> 10^6$, predictable response to all gases with molecular weights below 300, high stability, and no limitation on the choice of carrier gas (14, 18, 24). A particularly important advantage of this detector is that, despite its high sensitivity (10^{-9}g of most gases can be detected), it is not adversely affected by gases found in soil atmospheres and does not require frequent recalibration for accurate analyses.

MATERIALS AND METHODS

The gas chromatograph used is a Tracor Model 150G instrument (Tracor Inc., Austin, Texas) fitted with an ultrasonic detector and dual phase meters. A Westronics Model MT-22 recorder with dual pens and dual integrators is used for independent recording of both sides of the detector. Figure 1 shows a schematic diagram illustrating the sampling system (A), the two-column analysis system (B), and an optional sampling and column system (C).

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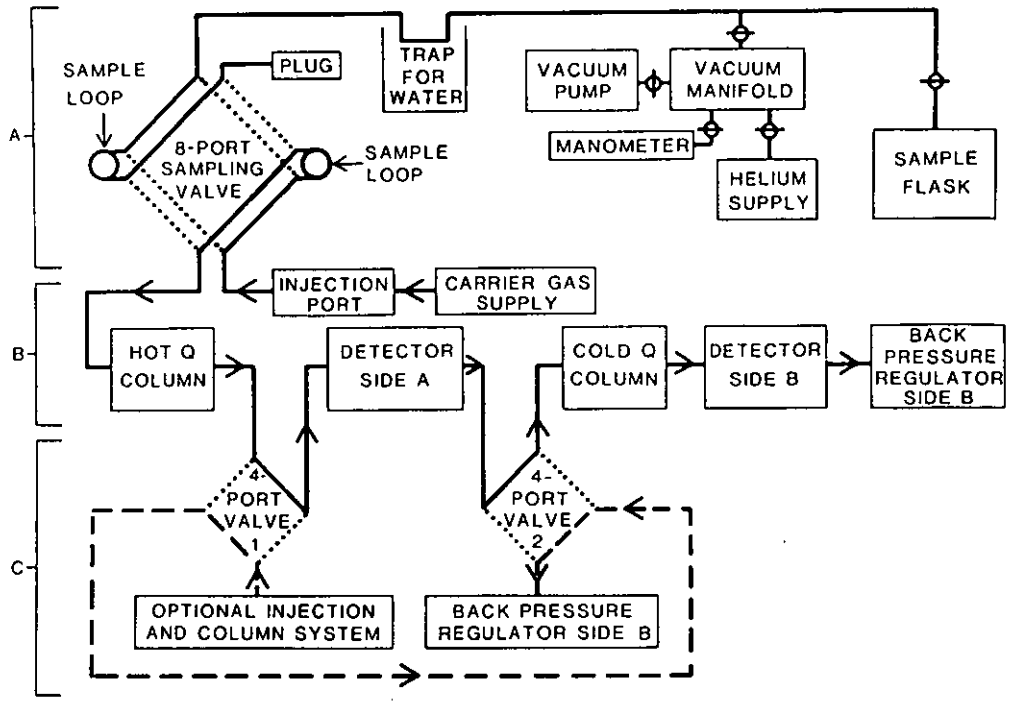

Fig. 1—Schematic diagram illustrating sampling system *(A)*, hot Q-cold Q column system *(B)*, and optional sampling and column system *(C)*. Dotted lines show alternative flow routes through valves; dashed lines show flow for optional system.

Atmospheres within any container sealed by a stopcock are sampled as follows: (i) the container (sample flask in Fig. 1A) is attached to the sampling system via a short piece of Tygon® vacuum tubing; (ii) the section from the closed stopcock on the sample flask to the plug on the sampling valve is evacuated, flushed with He, and reevacuated; (iii) the upper stopcock on the manifold is closed, and the stopcock on the sample flask is opened to allow gas from the flask to flow into the evacuated tube and sample loop; (iv) after 5 sec, the stopcock on the sample flask is closed and the 8-port sampling valve is rotated to transfer the contents of the appropriate sample loop to the carrier gas stream. Although the size of the sample loops can be readily changed, 1-ml loops have proven satisfactory for most analyses of soil atmospheres.

A trap for water (Fig. 1A) is included in the sampling system by placing a short (ca. 4 cm) U-shaped segment of the manifold tubing in methanol cooled by dry ice (the dry ice is maintained at least 3 cm below the tubing to eliminate any possibility of freeze-out of CO_2 from the gas sample).

Samples of soil atmospheres can also be introduced into the carrier gas stream by a gas-tight syringe via the injection port. Flowing gas streams can be sampled by connecting the gas stream to the 8-port sampling valve at the point labeled "PLUG" in Fig. 1A and by rotating the valve to inject the sample into the carrier gas stream.

In the two-column analysis system (Fig. 1B), gas samples placed in the carrier gas stream flow through the "hot Q" column {50-80 mesh Porapak Q® packed in stainless steel tubing (4.3 m, 3.2-mm outside diam (OD), 2.1-mm inside diam (ID)) maintained at 45°C}, detector side A, the "cold Q" column {50-80 mesh Porapak Q® packed in stainless steel tubing (7.6 m, 3.2-mm OD, 2.1-mm ID) submerged in a dry ice-methanol bath}, detector side B, and then out of the instrument through the back-pressure regulator for side B. The flow of He carrier gas (Matheson Ultra High Purity) is maintained at 50 ml/min by applying this gas at a pressure of 4.2 kg/cm² to the head of the hot Q column and by setting the back-pressure regulator for side B at 2.1 kg/cm².

The two dry-ice methanol baths employed in the procedure described are removed at the end of each working day. The flow of He carrier gas through the columns is maintained overnight.

Provision for an optional sampling and column system is included by addition of 4-port valves before and after detector side A (Fig. 1C). These valves allow the hot Q-cold Q column system to be placed in a standby status while detector side A is being used with the optional column. This optional column may be selected to perform analyses not possible with the hot Q-cold Q column system. The carrier gas supply for the optional column is not shown in Fig. 1.

Calibration curves and retention times are established for each gas by analyzing gas standards prepared by mixing pure gases or by generating gases within sealed flasks (e.g., N_2 can be generated by mixing solutions of nitrite and sulfamic acid).

RESULTS AND DISCUSSION

The sampling system illustrated in Fig. 1A has important advantages for studies of soil atmospheres. One is that determination of the concentration (wt/vol) of a gas within a sample flask is not affected by uptake or evolution of other gases within the flask because a constant fraction of the gaseous atmosphere is sampled regardless of the pressure in the flask. Another is that all-glass systems can be used for incubation of soils or transportation of samples of soil atmospheres. This eliminates the need for septa, which have a tendency to leak after being punctured (20), and for rubber closures, which can adsorb or release gases (16). Flasks fitted with glass stopcocks can be attached to the sampling system and sampled without contamination of the flask or sample with air and without the use of syringes, which frequently have tight-fitting plungers that are difficult to operate satisfactorily, loose-fitting plungers that leak, or needles that plug easily.

A chromatogram showing the response of detector side A after injection of a mixture of Ne, H_2, N_2, NO, CH_4, Kr, CO_2, and N_2O in He is illustrated in Fig. 2. If O_2 and Ar had been present in this mixture, they would have produced a single composite peak between the N_2 and NO peaks. The Ne, H_2, N_2, and O_2 + Ar peaks are too close for accurate

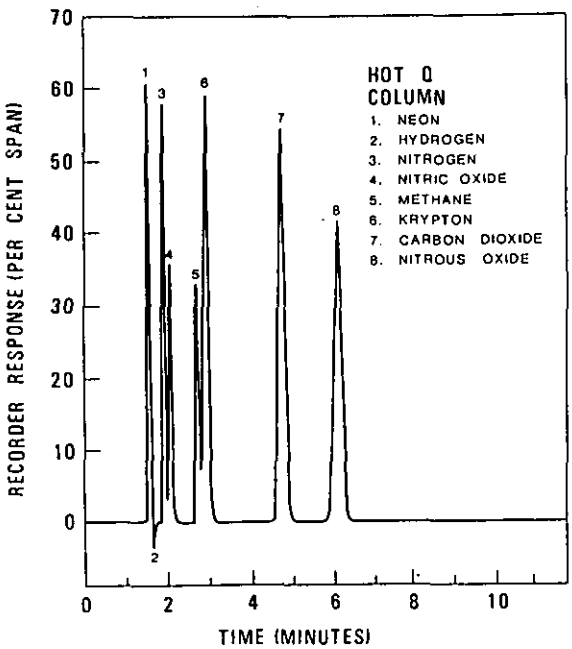

Fig. 2—Chromatogram showing response of detector side A after injection of 1 ml of He containing 0.1-0.2% (vol/vol) of Ne, H_2, N_2, NO, CH_4, Kr, CO_2, and N_2O (attenuator was set at ×128).

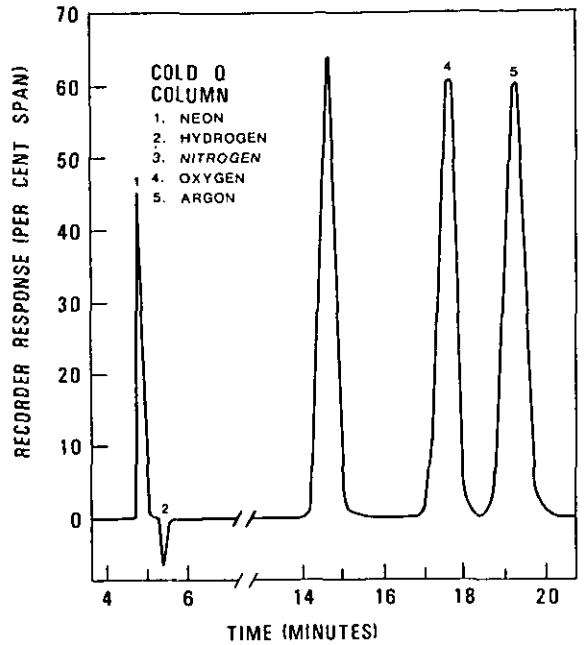

Fig. 3—Chromatogram showing response of detector side B after injection of 1 ml of He containing 0.1-0.2% (vol/vol) of Ne, H_2, N_2, O_2, Ar, CH_4, Kr, CO_2, and N_2O (attenuator was set at ×8)

determination of these gases using detector side A, except when these gases are present in low concentrations. However, complete resolution of these five gases is achieved in the cold Q column. This is illustrated in Fig. 3, which shows the response of detector side B after injection of a mixture of Ne, H_2, N_2, O_2, Ar, CH_4, Kr, CO_2, and N_2O in He. Because a dual pen recorder is used, the chromatograms illustrated in Fig. 2 and Fig. 3 appear on opposite sides of the same chart paper.

The retention times of 23 gases in the hot Q and cold Q columns are shown in Table 1. Gases having retention times in the cold Q column that exceed 2 hours appear only as slight baseline drift on detector side B unless very large amounts of these gases are injected. Gases retained by the cold Q column are released when the dry ice-methanol bath used to cool this column is removed at the end of each working day.

The necessary time interval between injections is dictated by the gases present in the mixture and the gases to be determined. Samples of most soil atmospheres can be injected every 6.5 min without overlapping of peaks because H_2, N_2, O_2, Ar, NO, CH_4, CO_2, and N_2O pass through detector side A within 6.5 min after injection, and N_2, O_2, and Ar pass through detector side B within 6.5 min after a 14-min delay in the cold Q column. Injection every 6.5 min results in continual and simultaneous use of both sides of the detector. It is not necessary, as with a thermal conductivity detector (5), to reverse the polarity of the recorder and delay subsequent injections until the cold Q column clears because the two sides of the ultrasonic detector function independently.

The procedure described permits detection of 3-9 ng of N_2, O_2, Ar, CO_2, N_2O, CH_4, or NO. Previous reports of the operating characteristics and performance of the ultrasonic detector (14, 24) indicate that a similar sensitivity can be expected for most gases with molecular weights <300. Our experience with thermal conductivity detectors and reports of their performance (1, 5, 8, 22) indicate that the ultrasonic detector has at least a 10-fold sensitivity advantage over thermal conductivity detectors for soil atmosphere research. Meaningful comparison of the sensitivities of these detectors is difficult because ultrasonic detectors can perform at maximum sensitivity without being adversely affected by gases found in soil atmospheres, whereas thermal conductivity detectors cannot be operated at maximum sensitivity in the presence of O_2 or other gases that react with their filaments when these filaments are operated at the high temperatures necessary for maximum sensitivity. Although He-ionization detectors are potentially more sensitive than the ultrasonic detector (15), reports of studies involving the use of He-ionization detectors for soil atmosphere research or similar applications indicate that, in practice, the sensitivity of He-ionization detectors is not significantly greater than that of the ultrasonic detector (6, 9, 13, 17). Our experience

Table 1—Retention times for 23 gases.

Gas	Hot Q column	Cold Q column
	retention time, min	
Neon	1.4	4.7
Hydrogen	1.6	5.3
Nitrogen	1.8	14.8
Oxygen	1.9	17.7
Argon	1.9	19.3
Carbon monoxide	1.9	22.4
Nitric oxide	2.0	18.1
Methane	2.8	L
Krypton	3.0	L
Carbon dioxide	4.8	R
Nitrous oxide	6.1	R
Ethylene	9.0	R
Acetylene	9.0	R
Other gases‡	>15	R

† L, retention time exceeds 2 hours; R, retained by column.
‡ Phosphine, hydrogen sulfide, carbonyl sulfide, water, sulfur dioxide, carbon disulfide, methyl mercaptan, dimethyl sulfide, dimethyl disulfide, ammonia.

Table 2—Precision of procedure described.

Gas determined	Concentration (%, vol/vol)	Attenuator setting	Peak heights (% of recorder span) Range	Mean	SD‡
N_2	77	X 4096	87.0-88.0	87.6	0.34
O_2	20	X 2048	70.0-71.0	70.3	0.37
CH_4	2	X 128	64.0-64.5	64.5	0.13
Ar	1	X 64	85.0-86.5	85.8	0.56
CO_2	0.03	X 32	51.0-53.5	52.4	0.85
N_2O	0.008	X 2	90.0-93.0	91.6	1.01

† Results of 15 analyses of a mixture of N_2, O_2, Ar, CH_4, CO_2, and N_2O (sample size, 1 ml).
‡ Standard deviation.

is that a new He-ionization detector is more sensitive than a new ultrasonic detector, but that this advantage is rapidly lost when these detectors are used routinely for soil atmosphere research.

The high precision of the procedure described is illustrated by the data in Table 2, which shows the results of replicate analyses of a mixture of N_2, O_2, Ar, CO_2, CH_4, and N_2O prepared by injecting small amounts of CH_4 and N_2O into 45 liters of laboratory air. All peaks were measured to the nearest 0.5% of the recorder span. The mean standard deviation of the replicate peaks for the six gases determined was 0.54% of the recorder span.

The procedure described permits analysis of 1-ml samples of gas for N_2, O_2, Ar, CO_2, CH_4, and N_2O at concentrations ranging from <10 ng/ml to 100%. The linearity of the relationship between detector response and weight of gas injected is illustrated in Fig. 4 with CO_2 used as an example. The wide linear range of the ultrasonic detector minimizes the need to adjust the sample size to the concentration of the gas to be determined because, as shown in Table 2, precise determinations of gases in the percentage range and ppm range are possible with a 1-ml sample.

The procedure described has been used extensively in our laboratory for almost 2 years, and no column degeneration or change in detector sensitivity has been observed during this time. Tests at intervals have shown that, unlike thermal conductivity or He-ionization detectors, the ultrasonic detector does not require frequent recalibration when used for analysis of soil atmospheres.

Besides being more durable than previous methods for analysis of soil atmospheres, the procedure described has the advantages that it allows use of different sampling techniques, can be readily modified to perform additional analyses through use of the optional column, and permits comprehensive analysis of a single sample of gas without temperature programming, stream splitting, column conditioning, column switching, or switching of recorder polarity. It has, therefore, a combination of features (simplicity, sensitivity, specificity, durability, versatility, wide linear range, etc.) recommending it as a basic analytical procedure for research on soil atmospheres.

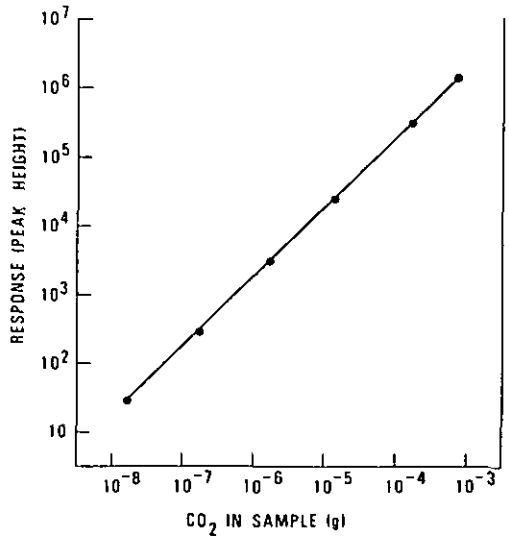

Fig. 4—Relationship between detector response and weight of CO_2 injected.

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Decomposition of Carbon-14 Labeled Plant Material Under Tropical Conditions[1]

D. S. JENKINSON AND A. AYANABA[2]

ABSTRACT

Ryegrass (*Lolium multiflorum*) and maize (*Zea mays*) tissue uniformly labeled with ^{14}C were mixed with soil and allowed to decompose under field conditions in the open or under shade. The incubations were done in the forest zone of Nigeria, using a range of contrasting Nigerian soils. Of the ryegrass C originally added to the soil, 20% remained after 1 year, falling to 14% after 2 years. After 1 year the soil retained slightly less maize C than ryegrass C, but the difference was small and the overall pattern of decomposition similar. There was little difference between the rate of decomposition under shade or in the open, even though soil temperatures were considerably greater in the open. A soil containing 6% clay (Apomu series) retained slightly less maize C after 1 year than a soil with 16% clay (Egbeda series), but in general the decomposition rates in the different soils were similar.

For ryegrass, the decomposition pattern was very similar under Nigerian conditions to that previously observed for the same plant material in England, except that the whole decomposition process was four times faster in Nigeria.

Additional Index Words: **ryegrass, maize, retention of C in soil, soil organic matter, soils of the humid tropics, Nigeria.**

IN INTRODUCING improved agricultural systems to the humid tropics it is important to consider their effects on the organic matter content of the soil and, in particular, to see if soil organic matter can be stabilized at levels that permit sustained operation of the new systems. The amount of organic matter in a soil that has been under a given system of cropping and management for a long time depends on how much organic matter enters the soil each year and how fast this organic matter decomposes in the soil. This paper is about the second of these processes, the rate of decomposition of plant material in soil under humid tropical conditions.

Plant material uniformly labelled with ^{14}C has been extensively used to follow decomposition under temperate conditions (3, 8, 9, 11, 12), but as yet only one comparable study has been made in the tropics (10). The aims of the present work were to follow the losses of C from different plant materials decomposing in a contrasting range of tropical soils, and thus to obtain accurate data on the rates of decomposition under these conditions. Labelled ryegrass (*Lolium multiflorum* Lam. cv. Westerwolth) was used in part of the work because material was available from a batch that had already been used in similar work in England. Although ryegrass is not a plant of the humid tropics, its use in these experiments allowed direct comparison between decomposition rates under cool temperate conditions and under hot humid conditions. Maize (*Zea mays* L.) was used in the other part of the study, as representative of a plant material often decomposed in tropical soils. This paper reports the results from the first 2 years of the ryegrass incubations and the first year of the maize incubations: the experiments are to be continued for several more years.

MATERIALS AND METHODS

Four soils were used in the initial studies, started in May 1974, and a further four in the main experiment, started in May 1975. Analytical data on all eight soils (sampling depth 0-15 cm) are given in Table 1. The analytical methods used were as described (5); all analyses are reported on an oven-dry basis (105°C for 24 hours). Seven of the soils came from Nigeria; the other, included for comparison, was a soil from England that had already been used in decomposition studies with labeled plant material (3). The plant material used in the 1974 experiment was ryegrass: a full account of its preparation and analysis is available (2). Roots and tops (both ground to pass a 0.5-mm sieve) were investigated separately; prior to use, the freeze-dried plant materials were stored at −15°C to minimize radiation damage. The freeze-dried tops contained 40.3% C, with a specific activity of 23.8 μCi/g C; the roots 33.9% C, with 21.0 μCi/g C. In the 1975 experiment maize was used, supplied by Mr. H. L. Hyland, USDA, Beltsville, Maryland. The material as received (leaves, ground to pass a 30-mesh sieve) contained 38.3% C and had a specific activity of 11.7 μCi/g C.

Incubation was done in polyethylene tubes, 12 cm long by 7 cm inside diam closed at both ends by stainless steel gauze (30 mesh). A glass-fiber filter paper was placed over the gauze at the bottom and the tube sunk into the soil to a depth of 6 cm. In the 1974 experiment, labeled plant material (92.1-mg ryegrass tops or 107.4-mg ryegrass roots) was mixed with a quantity of moist soil (previously put through an 8-mm sieve) that contained 150-g soil on an oven-dry basis, and placed in the incubation tubes. In the

Table 1—Analyses of soils.

Soil no.†	Soil series	Location	Previous history	pH	Clay	N	Organic C‡	CO_3-C
						%		
1	Egbeda	IITA	Secondary forest	6.7	17	0.096	1.08(1.04)	0
2	Apomu	IITA	Secondary forest	6.6	6	0.083	0.88(0.89)	0
3	Alagba	Ijebu-Ode	Bush regrowth	6.6	10	0.098	1.15(1.20)	0
4	Batcombe	Rothamsted	Arable	8.0	21	0.100	0.91	0.27
5	Egbeda	IITA	Secondary forest	6.2	16	0.107	1.17(1.14)	0
6	Apomu	IITA	Secondary forest	6.3	6	0.081	0.93	0
7	Alagba	Ikenne	Bush regrowth	7.0	12	0.135	1.56	0
8	Alagba	Benin	Bush regrowth	6.3	10	0.114	1.23(1.18)	0

† Soils 1–4 used in the experiment with labeled ryegrass; soils 5–8 with labeled maize.
‡ By wet combustion; figures in parentheses are by dry combustion.

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