

CHROM. 4362

## Gas chromatography of gases emanating from the soil atmosphere

In different denitrification studies, many investigators<sup>1-3</sup> have studied the composition of the soil atmosphere. It is desirable to develop a method suitable for separation and analysis of oxygen, nitrogen, carbon dioxide, nitric oxide, nitrous oxide and nitrogen dioxide. The present studies were undertaken to separate the gases by gas chromatography at ambient temperature.

### Experimental

A method for the gas chromatographic analysis of a sample of soil atmosphere was developed using a three-column system and one detector. The columns used in three stages are Carbowax on glass beads<sup>4</sup>, Porapak Q<sup>5</sup> and molecular sieve<sup>4</sup> respectively. The details of the construction and operation of the three columns are as follows: the first is a 1 ft.  $\times$  1/8 in. O.D. stainless-steel column, externally placed and filled with 0.5% Carbowax 1500 on 60-80 mesh silanized glass beads; the second is a 18 ft.  $\times$  3/16 in. O.D. stainless-steel column packed with Porapak Q 80-100 mesh and activated before use at 230° for 1-2 h; the third is a 3 ft.  $\times$  1/4 in. O.D. stainless-steel column packed with 0.2-0.5 mm molecular sieve 5 A and activated at 220° for 24 h.

This three-column system is operated in series so that the sample enters the first external column, flows to the second Porapak Q column and passes to the third molecular sieve column.

The detector is a Gow Mac thermal conductivity cell with W<sub>2</sub>X filaments and is operated by a bridge current of 250 mA. The detector signal output is fed to a 1 mV recorder. Helium was used as carrier gas at a flow rate of 50 ml/min.

### Results and discussion

The first external column is placed in a bath of liquid air. At low temperature this column retains NO<sub>2</sub>, NO, CO<sub>2</sub> and N<sub>2</sub>O. The remaining gases (O<sub>2</sub> and N<sub>2</sub>) flow to the second and third columns. The Porapak Q column gives a composite peak of

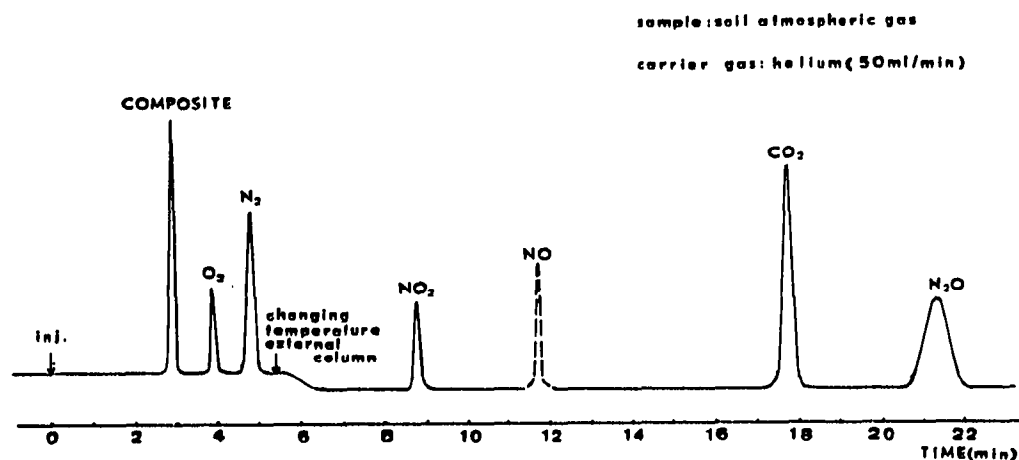


Fig. 1. Chromatogram of a mixture of O<sub>2</sub>, N<sub>2</sub>, NO<sub>2</sub>, NO, CO<sub>2</sub> and N<sub>2</sub>O gases. Sample, soil atmospheric gas; carrier gas, helium (50 ml/min).

O<sub>2</sub> and N<sub>2</sub>, and the molecular sieve column separates them. After O<sub>2</sub> and N<sub>2</sub> have been detected, the temperature of the first column is increased to a high temperature by boiling water so that NO<sub>2</sub>, NO, CO<sub>2</sub> and N<sub>2</sub>O flow to the Porapak Q column, where they are separated.

However, it is impossible to have oxygen and nitric oxide together in one sample because, in the presence of oxygen, nitric oxide is immediately oxidized to nitrogen dioxide<sup>6</sup>.

By changing the external column from low to ambient temperature the baseline is also changed; but after some time it will adjust itself.

Using helium at a flow rate of 50 ml/min, the column system gives, as shown in Fig. 1, a good separation for all the gases.

Acknowledgements are due to Prof. Dr. A. VAN DEN HENDE and Mr. L. BAERT for their encouragement and interest in this work and to I.R.S.I.A. for financial support of this investigation.

*Faculty of Agricultural Sciences, Coupure, Ghent (Belgium)*

O. VAN CLEEMPOT

- 1 R. G. BELL, *Soil Sci.*, 105 (1968) 78.
- 2 F. CADY AND W. BARTHOLOMEW, *Am. Proc. Soil Sci. Soc.*, 24 (1960) 477.
- 3 D. SMITH AND F. CLARK, *Am. Proc. Soil Sci. Soc.*, 24 (1960) 111.
- 4 J. M. TROWELL, *Anal. Chem.*, 37 (1965) 1152.
- 5 W. WILHITE AND O. HOLLIS, *J. Gas Chromatog.*, 6 (1968) 84.
- 6 P. PASCAL, *Nouveau Traité de Chimie Minérale X*, Masson, Paris, 1956.

Received September 8th, 1969

*J. Chromatog.*, 45 (1969) 315-316