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Note

Determination of ethylene oxide in air by gas chromatography

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Ethylene oxide is used as a sterilizing agent and as an insecticidal fumigant. It is usually applied in a mixture with compressed carrier gases such as carbon dioxide in order to reduce flammability.

For measurements of concentrations of ethylene oxide in air, reagents impregnated on papers¹ or aspirator tubes^{2,3} have been used. Analysis by titration⁴ or by colorimetric methods⁵⁻⁷ are lengthy and comparatively large samples being required, while analysis by spectrophotometry⁸ requires chemical reactions and lengthy sample preparation.

Gas chromatography was therefore investigated as a means for the rapid determination of ethylene oxide in air. The availability of small gas chromatographs with good stability and selectivity makes possible their use in the field when accurate results are required.

MATERIALS AND METHODS

A Perkin-Elmer Model 154-D gas chromatograph, equipped with a thermistor cell and a stainless-steel column (4 m × 6.3 mm I.D.) filled with Chromosorb W (40-60 mesh) as the solid phase and 30% didecyl phthalate as the liquid phase, was used. The column temperature was 50 °C and the flow-rate of the helium carrier gas was 50 ml/min. A 1-mV recorder with a 1-sec response and 10-in. chart paper was used to record the response of the detector to the fumigant. Ethylene oxide (99.7% purity) in compressed gas cylinders from Matheson of Canada Ltd. (Whitby, Canada) was used.

Ethylene oxide from the gas cylinder was liquefied by cooling with dry ice, and then introduced into weighed 5-ml glass ampoules. The ampoules were sealed with a flame and re-weighed to determine the content of ethylene oxide. They were then crushed inside a closed 525-l fumigation chamber and the gas was mixed with air by fan circulation for 10 min. Samples of the ethylene oxide-air mixture were taken from the chamber with a gas syringe of appropriate size and injected into the gas chromatograph.

A sample size of 5 ml was required for full-scale pen deflection on the recorder at a concentration of ethylene oxide in the air mixture of 1 mg/l. Concentrations

normally used range from 13 to 32 mg/l for insect control^{9,10} and from 450 to 900 mg/l for sterilization of equipment to eradicate microorganisms¹¹.

RESULTS AND DISCUSSION

When the sample was introduced into the gas chromatograph, the retention time was 140 sec for ethylene oxide and 35 sec for air.

Fig. 1 shows the separation of air and ethylene oxide using a 1-ml sample containing ethylene oxide at a concentration of 1 mg/l. Such a sample therefore contained 1 μ g of ethylene oxide. In this experiment, the standard deviation for ten consecutive determinations was 0.06 μ g.

The method was used with good results for the determination of ethylene oxide during fumigation experiments at different time intervals with a wide range of ethylene oxide concentrations (1–1000 mg/l).

The method was also found to be satisfactory for the analysis of carboxide (ethylene oxide-carbon dioxide, 1:9) which was used for the control of mites on apples¹⁰. The rapid and accurate analysis of a fumigant that can be achieved by this method allows the continuous monitoring of concentrations and immediate adjustments can then be made if gas is lost or absorbed at unexpected rates.

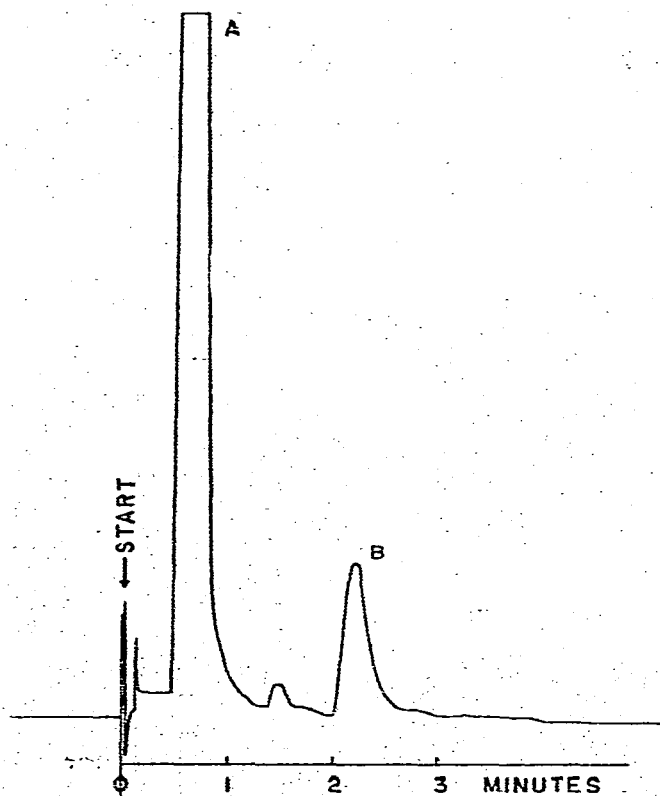


Fig. 1. Chromatogram of ethylene oxide in air (1 mg/l), using a thermistor detector at 1-mV sensitivity. Sample size: 1-ml. Peaks: A, air; B, ethylene oxide.

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