Simple Gas Chromatographic System for Analysis of Microbial Respiratory Gases

The problem:
To develop a simple, sensitive gas chromatographic system for the determination of the microbial respiratory gases, hydrogen, nitrogen, oxygen, methane, and carbon dioxide, and to fabricate the system to be small, light in weight, and with low power consumption so that it can be used in remote life-detection equipment for space experiments.

The solution:
A room-temperature chromatograph consisting of a pair of capillary columns packed with Porapak Q that has been treated with phosphoric acid, a microbead thermistor detector, and a micro gas-sampling valve. The columns are wrapped snugly around a detector block and packed within a 15-cm cube of high-density Styrofoam.

How it’s done:
Each of the columns are about 7 meters long and fabricated from stainless steel tubing, nominally \( \frac{1}{8} \) OD with 0.010" wall (1.6-mm OD, 0.25-mm wall). They are packed with 100/120 mesh Porapak Q which has been treated with enough methanolic phosphoric acid solution to produce an 0.01% coating when dried in a rotary evaporator. The phosphoric acid treatment prevents carbon dioxide from being absorbed in trace amounts, and thus the symmetry of carbon dioxide peaks on the chromatograms is improved.

The chromatographic system is operated at 100 psig (abt 800 kN/m² absolute) with high-purity helium at a flow of 15 ml/min. The column temperature is approximately 20°C, and only minor insulation is required to provide adequate temperature control; detector current is optimized at 11 mA.

The device has been used to measure gas composition changes in the head-space gas within a 6-ml metabolic chamber. Since multiple 100-µl gas samples used in the experiments represented a significant proportion of the chamber volume, krypton was added initially to the chamber as an internal standard; because krypton is not involved in metabolic activity, it is possible to make a correction for losses or pressure changes due to periodic withdrawal of samples.
In typical chromatograms obtained with this system, resolution of nitrogen and oxygen is 98%; carbon monoxide and argon are not resolved from oxygen, but these gases generally are not found in the products of microbial respiration. Hydrogen gas can give a negative deflection when helium is used as the carrier, but the small amounts of hydrogen occurring in microbial respiration products do not give rise to this anomalous behavior.

Minimum detectable limits of the unit are of the order of $10^{-3}$ moles H₂, $10^{-4}$ moles CH₄, CO₂, and Kr, and $10^{-6}$ moles O₂ and N₂; these values are for a sample loop volume of 100 µl and a signal-to-noise ratio of 2 at a 25-µV noise level.

The dual column configuration was found to be insensitive to ambient pressure and temperature changes, and produced chromatograms with lower noise level and greater baseline stability than a single column. Thus, the advantage to be gained by using single-column systems in spacecrafts to reduce weight, conserve carrier gas and operate at lower power levels were outweighed by the superior performance of the dual-column ambient temperature system.

Reference:

Note:
No additional documentation is available. Specific questions, however, may be directed to:
Technology Utilization Officer
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Reference: B72-10207

Patent status:
No patent action is contemplated by NASA.
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