Direct Measurement of 14CO₂ by Liquid Scintillation Counting

The problem:
To develop a technique for the direct measurement of carbon-14 in carbon dioxide. Previous methods suffered from such drawbacks as nonquantitative absorption or reaction of the CO₂, quenching from the addition of absorbents or reactants, and isotope effects during successive reactions.

The solution:
A liquid scintillation counting technique for measurement of 14C activity. The carbonaceous sample is first converted into CO₂, and the 14CO₂ is then measured by a liquid scintillation counter. This method has high counting efficiency and eliminates many of the basic problems encountered with previous measuring techniques.

How it's done:
Three different counting systems have been used with equal success: a room temperature single-multiplier phototube (RCA-8575) system; a −20°C single-multiplier phototube (DuMont-6292) system; and a 0°C coincidence liquid scintillation counter.

Two scintillator solutions were used to match emission from the solution with response of the multiplier phototubes. With the RCA-8575 multiplier phototube, a solution of 3 g of PPO in a liter of toluene was used; with the DuMont-6292 multiplier phototube, a solution of 7 g of PPO and 0.5 g of M₂-POPOP in a liter of toluene was used.

The CO₂ gas samples were prepared by normal vacuum line manipulations. The CO₂ was condensed with liquid nitrogen onto a previously degassed and frozen volume of the scintillator solution. The sample tubes were then sealed by flame, minimizing the volume of space above the scintillator solution.

The solubility of CO₂ in toluene base scintillator solution is sufficient to allow the 14C measurement at room temperature. By the use of a special type of sample container, the space above the solution was minimized to the extent that greater than 99% of the CO₂ was dissolved in the solution.

To determine the concentration quenching of CO₂, samples were prepared with varying concentrations of CO₂. The relative scintillation yields were determined by the Compton edge technique. There was no detectable quenching up to 5 cc (STP) per ml of liquid scintillator solution.

To determine the counting efficiency, several samples of known volume of 14C containing CO₂ were dissolved in 2 ml of scintillator solution and counted in a room temperature single-multiplier phototube system. The counting rates were determined by the integral counting technique. Aliquots of the same gas were counted in a gas counter. The extrapolated integral counting rates of samples in the liquid scintillators agreed with ±2% of the absolute disintegration rate obtained with the gas counter.

This method can be useful for either assay or absolute measurement of 14C radioactivities in samples converted into CO₂. Counting efficiencies of greater than 90% are obtained with low backgrounds. With an integral counting technique, a counting efficiency of (100 ± 2) % was obtained. The lower level of detection of 14C by this method is 10⁻⁷ Ci per cc (STP) of CO₂ for a 10 cc sample (about 2 disintegrations per minute).

Notes:
1. The use of scintillation counters is not new, but this particular measurement is a novel application.
2. The technique can be used to achieve a percent substitution reaction, and may be of interest to university and hospital laboratories as an analytical technique.

3. Inquiries concerning this report may be directed to:
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Patent status:
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