

Preparation of Volatile Carbon-14-Labeled Compounds for Counting

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VOLATILE C^{14} -labeled compounds are difficult to handle in Geiger-Müller and proportional counters. Reproducible results cannot be obtained because of losses from planchets during storage and counting. The technique to be described greatly reduces losses from planchets during storage and eliminates contamination of counting chambers and windows. It reduced the volatilization of a C^{14} -labeled herbicide, 2-chloro-4-isopropylamino-6-diethylamino-*s*-triazine (ipazine), and a C^{14} -labeled nematocide, *O,O*-diethyl-*O*-2-pyrazinylphosphorothioate (Zinophos), from counting planchets. The method conceivably can be employed with other radioactive, volatile, organic compounds.

One-inch diameter, sand-blasted, glass planchets were plated initially with 0.25 μg . of C^{14} -ring-labeled ipazine in 10 λ of absolute ethyl alcohol and with 1.15 μg . of C^{14} -ethyl-labeled Zinophos in 10 λ of hexane. Counting was performed with an aluminized-Mylar (1

mg. per sq. cm.), end-window, gas-flow, Geiger-Müller tube.

Volatilization of C^{14} -ipazine and C^{14} -Zinophos from uncoated planchets occurred rapidly and showed similar trends for both chemicals (Figure 1, lower curve). Since uncoated planchets could not be counted directly without contaminating the counting chamber and end window, acrylic plastic was applied to duplicate planchets at the indicated times prior to counting. The activities measured were subsequently corrected for absorption by the plastic film with the relationship developed in Figure 2. All planchets were kept in a hood at room temperature (30°C .) for 24 hours to restrict contamination evolving from the uncoated planchets. After 24 hours, coated planchets were stored in a constant-temperature room (22°C .). Planchets were coated with a clear acrylic plastic applied from a pressurized can. (Seymour's No. 121, Seymour of Sycamore, Inc., Sycamore, Ill., was used, but Clear Finish No. 54-115, Pittsburgh Plate Glass Co., Pittsburgh, Pa., was equally effective.)

The density thickness of the film was obtained by weighing tared planchets 20 minutes after application of the plastic. The weight of film applied to the planchets ranged from 0.4 to 0.8 mg. per sq. cm. Planchets coated at zero time lost only a minor amount of activity in 72 hours (Figure 1, upper curve). Loss was delayed considerably, but was not completely eliminated. Twenty-eight days after the plastic was applied, the C^{14} -ipazine and C^{14} -Zinophos planchets had retained 88.9 and 82.9%, respectively, of their initial activities. At the end of 90 days, the residual activity measured 86.4% for ipazine and 79.6% for Zinophos.

Effects of the thickness of the acrylic-plastic coat on the relative activities of C^{14} -labeled ipazine, Zinophos, and sodium carbonate are shown in Figure 2. Data for the figure were obtained by successively applying light coats of acrylate, weighing, and counting paired planchets for each of the chemicals. This sequence of operations was repeated four times. The radioactivity after each coat was divided by the uncoated activity (or the activity after one coat for the volatile chemicals) to obtain the relative activity. The ipazine and Zinophos planchets were coated immediately after evaporation of the solvent, and the activity after

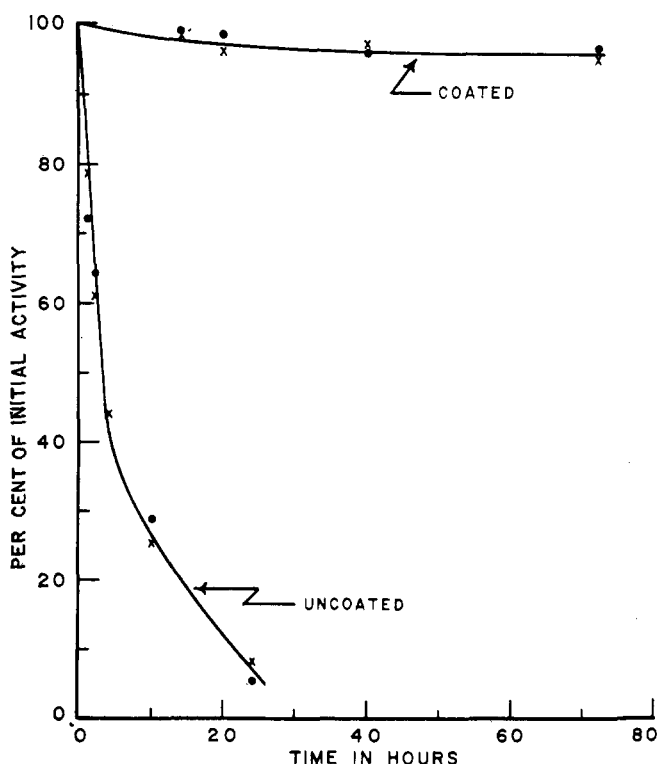


Figure 1. Effect of coating with an acrylic plastic on loss of C^{14} -2-chloro-4-isopropylamino-6-diethylamino-*s*-triazine (—●—) and C^{14} -*O,O*-diethyl-*O*-2-pyrazinylphosphorothioate (—×—) from glass planchets

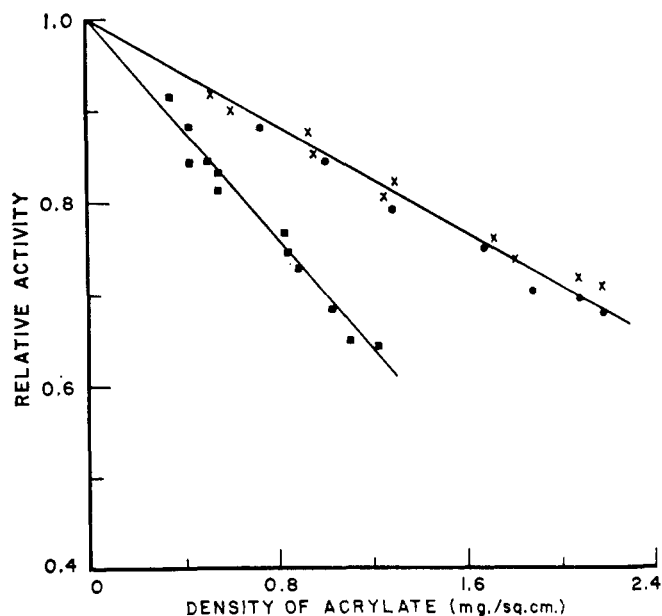


Figure 2. Effect of the thickness of acrylic plastic on the relative activity of C^{14} -labeled 2-chloro-4-isopropylamino-6-diethylamino-*s*-triazine (—●—); *O,O*-diethyl-*O*-2-pyrazinylphosphorothioate (—×—); and sodium carbonate (—■—)

this initial coat was assumed to equal full activity. Consequently, density thicknesses shown for the volatile organic chemicals have had the weight of the initial coat subtracted (0.27 to 0.48 mg. per sq. cm.). This was the only way an initial activity determination could be made due to the volatile nature of the chemicals. The manipulation appeared to be valid since the relationship between activity and thickness was linear to about 2.2 mg. per sq. cm. for both ipazine and Zinophos. A similar linear relationship was obtained for the low-volatile C^{14} -ring-labeled 2-chloro-4,6-bis(ethylamino)-s-triazine (simazine), when full activity could be represented by an uncoated

planchet. The absorption curve for C^{14} -Zinophos (Figure 2) is essentially identical with the one for ipazine. The absorption curve for sodium C^{14} -carbonate has a much steeper slope. The difference in slope may be attributed to solubilization of the labeled organic chemicals in the constituents of the plastic aerosol preparations, which resulted in a uniform distribution of the materials throughout the plastic coat. Sodium carbonate would probably be insoluble in the plastic solvents, and thus the entire coat thickness would have to be penetrated by the β -radiation from $Na_2C^{14}O_3$.

The same technique has been used successfully with a windowless propor-

tional chamber and aluminum planchets (1.25 inches diameter).

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A Rapid Method for Volume Calibration

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THE ACCEPTED METHODS for calibrating volumes in analytical vacuum systems either involve filling the areas with known amounts of mercury (1) or introducing a weighed quantity of gas (2). While these methods are quite accurate, the manipulations involved require considerable time and are impractical if the volumes are frequently altered. In this laboratory a procedure originally developed for the calibration of gas chromatography sample loops has been extended to include volume determinations of the incorporated vacuum-line and the attached sample containers. The method described in this paper considerably decreases the time involved, yet it maintains the required accuracy.

APPARATUS

The apparatus consists of a Loenco gas chromatograph, Model 1A (Loe Engineering Co., Altadena, Calif.), used in conjunction with a complete gas sampling vacuum system. A Brown-Honeywell recorder equipped with a Model K1-3 disk integrator was used. The chromatography column, packed with 20- to 60 mesh silica gel, was 6 feet in length and constructed of $1/4$ -inch o.d. copper tubing. Figure 1 shows the vacuum manifold and sample introduction system. Any commercial gas sampling valve, however, can easily be incorporated into this arrangement.

PROCEDURE

The gas chromatography unit is operated in the conventional manner with a helium flow of 200 ml. per minute and

column temperature of 60° C. A calibration of peak area *vs.* sample volume is obtained by injecting known samples (atmospheric pressure) of pure, dry, carbon dioxide into the chromatograph with a highly accurate gas syringe. Corrections for temperature and barometric pressure are then applied.

The system volume or the sample container to be determined is attached and evacuated to less than 10 microns. The volume is filled with carbon dioxide gas, and the pressure is accurately measured. After isolating the container and evacuating the system, the carbon dioxide is transferred to the sample loop which is immersed in liquid nitrogen. When the transfer is com-

plete, the loop is isolated and allowed to reach ambient temperature. The gas is then introduced into the gas chromatograph, and the peak area is recorded. Since a direct relationship exists between the recorded peak area and the moles of gas introduced from the containers, the volume is easily determined.

If the quantity of carbon dioxide injected for calibration and the quantity from the containers are of the same order of magnitude, the volume can easily be reproduced within ± 0.2 ml. Although carbon dioxide was used for this procedure, light hydrocarbons ob-

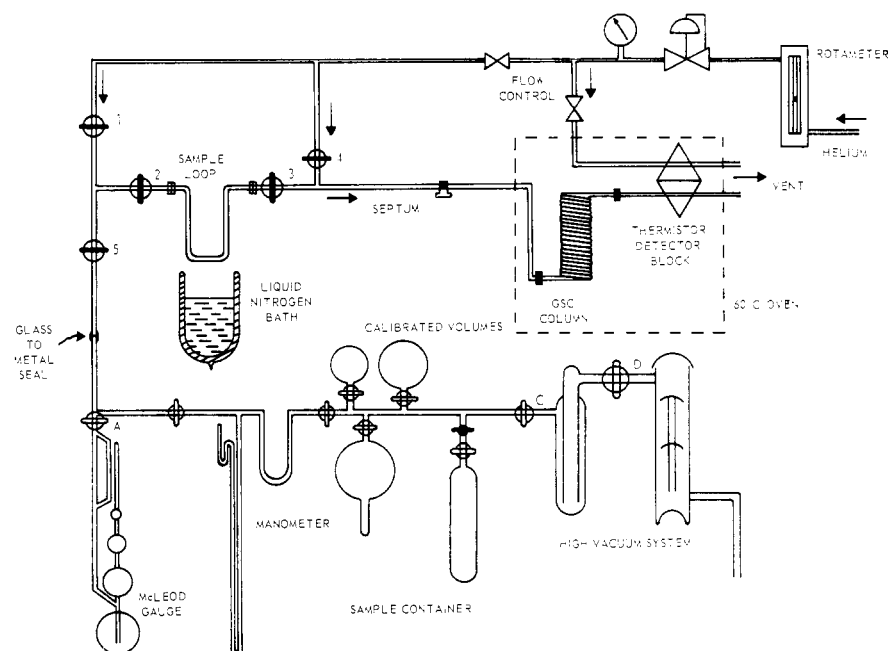


Figure 1. Vacuum manifold and sample introduction system

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