

grammed temperature (PTGC) runs, the 50-cc. flow rate applied only to the initial column temperature.

With 10% SE-30 as stationary phase, the  $t_R$  of the various fumigant gases were shorter, the sensitivity was higher, and the peaks were sharper and more symmetrical than those obtained with a loading of 30% SE-30. However, smaller aliquots must be used with the 10% loading.

To separate the solvent peaks from the fumigant peaks, *m*-xylene was used as trapping solvent when the boiling points of the fumigants were below 80° C., and *n*-pentane was used for fumigants boiling above 80° C. The  $t_R$  of *m*-xylene (8.75 minutes at 60° C.) was sufficiently long to permit the separation from relatively large amounts of air of fast-eluting gases such as methyl bromide, methyl chloride, and ethylene oxide that normally would be encompassed in the air or *n*-pentane peak. The use of trapping solvent can be omitted for methyl bromide ( $t_R$  = 1.00 minute at 60° C.) if the methyl bromide concentration is greater than 1500 µg. per liter of air, in which case 1 cc. of air can be injected directly into the injection port.

Levadie (18, 19) had previously used *m*-xylene as a trapping solvent for organic vapors. As indicated above, *m*-xylene was not suitable for HCN and PH<sub>3</sub>, which are relatively polar, and 1-butanol was found to be better suited for these gases.

The determination by gas chromatography of parts per billion amounts in air of fumigants, air pollutants, organic solvents, etc., is expedited with trapping solvents, such as *n*-pentane, *m*-xylene, or 1-butanol, to aggregate the trace amounts and to reduce the air peak and the interfering effect of water vapor. Trapping solvents are particularly useful in sampling room or outside atmospheres, where sample sizes of 15 to 20 liters of air or more may be taken, and are relatively simpler and more convenient to use in this regard than by collecting the trace constituents on cold activated charcoal or in cold traps (29). However, where sample size must be limited by practical considerations, as in the determination of fumigant residues of foods or soil, cold traps may be preferred, since they permit analysis of the total volatiles trapped, rather than of an aliquot as with trapping solutions. Nevertheless, by increasing the sensitivity of detection, such as can be achieved with a hydrogen flame ionization (1) or an electron-capture detector (15, 21, 22), the advantages of trapping solvents can be extended to problems in which sample size is limited. An additional advantage of use of a hydrogen flame detector is the elimination of the air peak that is normally "seen" with a thermal conductivity detector.

Applications for the methods as indi-

cated herein are abundant. Examples such as assessment of physiological effectiveness, toxic hazards, comparative efficiency of different methods of application, reaction kinetics, chemisorption, photodecomposition, fumigant residues, synergism or potentiation, air pollution, efficiency of respirator canisters, gas permeability of materials, chromatographic properties of cereal crops, etc., illustrate the many potential areas of use. In this regard, gas chromatography will play an increasingly important role in the identification and microdetermination of fumigant gases.

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#### CORRECTION

#### Nutrient-Conserving Agents. Loss of 2-Chloro-6-(trichloromethyl)-pyridine from Soil

In this article by C. T. Redemann, R. W. Meikle, and J. G. Widofsky [*J. Agr. Food Chem.* **12**, 207 (1964)] the following corrections should be made.

Under the subheading, "6-Chloropicolinic-C<sup>14</sup> Acid," the preparation of this acid is described. When we attempted to repeat this synthesis, we discovered that it was necessary to use 2-bromo-6-chloropyridine instead of 2,6-dichloropyridine and that the starting material for our first synthesis was the 2-bromo- derivative. at that time erroneously identified as 2,6-dichloropyridine. We have now demonstrated to our satisfaction that the dichloropyridine does not react with *n*-butyllithium under the conditions described.

2-Bromo-6-chloropyridine was prepared by means of a Sandmeyer reaction employing 2-amino-6-bromopyridine, cuprous chloride, and 6N hydrochloric acid (m.p. 87-88°).

ANALYSIS. Calculated for C<sub>5</sub>H<sub>3</sub>BrClN: C, 31.31; H, 1.57; N, 7.28. Found: C, 31.62; H, 1.45; N, 7.02.

The source of *n*-butyllithium used in this reaction was critical. The commercially available reagent dissolved in *n*-hexane or heptane will not react with 2-bromo-6-chloropyridine. However, a butyllithium preparation in ether, stored in a refrigerator and only a few days old, reacts very readily. Presumably, a small amount of lithium ethoxide is a necessary adjunct for a successful halogen-metal interconversion in this case. The obvious experiment in which lithium ethoxide is added to a butyllithium preparation in hexane was not carried out because the preparation in ether solved our problem.

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