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Teflon as a superior support for gas chromatography of organophosphorus pesticides and their metabolites

Although the elution pattern of a series of compounds is primarily dependent upon proper selection of liquid phase, in the GLC of polar compounds the proper selection of solid support is of major concern, especially when low-loaded columns are used. The surfaces of most supports are not completely inert and can interact with solutes and therefore can cause tailing, shifting retention, and occasionally even catalytic effects¹. Undesirable adsorption characteristics of the support may be partially avoided by using higher loading if conditions permit, by using polar phases, and by conditioning with on-column treatment with hexamethyldisilazane. Effective suppression of support activity has also been achieved by coating the support with silver² or teflon emulsion³.

These undesirable interactions are particularly noticeable when 1-2% of the liquid phase is used to coat the support, but it is fallacious to assume that they are absent on high-loaded columns, for even glass surfaces are not completely inert⁴. These effects are more noticeable when the liquid phase is nonpolar or less polar than the solute, which is then capable of reacting with the support through hydrogen bonding. Chemical reactions and isomerizations can also occur on the surface of support, much as they would with catalysts⁵.

In this communication is described the behavior of some organophosphorus pesticides on various supports and a technique of using a teflon support to avoid these difficulties. This appears to be the first study of the use of a teflon support for organophosphorus pesticide analysis by GLC.

Materials and methods

Various solid supports including Chromosorb W 80/100 mesh, Chromosorb W HMDS-treated, Gas-Chrom Q, Polypak-1 80/100 mesh, "Haloport" teflon 30/60 mesh (Hewlett-Packard, F & M Scientific Div.), glass microbeads 80/120 mesh (Applied Science Laboratories, Inc.), Porapak Q 80/100 mesh (Water Associates, Inc.), and Supelcoport 80/100 mesh (Supelco, Inc.) were used. These supports were usually coated with liquid phase by batch coating but sometimes by the infiltration technique⁶. Teflon was coated by the in-place coating method.

The GLC analyses were reproducibly performed as previously described⁷, using a Hewlett-Packard Model 402 high-efficiency gas chromatograph equipped with a hydrogen flame detector. The detector was modified for the thermionic determination of phosphorus by mounting a KCl pellet (Hewlett-Packard) on the burner jet.

Results and discussion

Methyl paraoxon could not be gas chromatographed on Chromosorb W with a coating of less than 5% SE-30. However, when Chromosorb W was coated by the infiltration technique⁶ with 20% SE-30, adsorption effects were not observed. On the other hand, Porapak Q and Polypak-1 were unsatisfactory for use with organophosphorus compounds. The glass microbeads, with liquid phase loadings of 0.1-3% by both batch-coating and infiltration techniques, also were unsatisfactory as a support;

even methyl parathion, which elutes on most columns with minimum conditioning, was not gas chromatographed on these columns. It is difficult to explain this anomalous behavior, though the use of glass beads is more an art than a science.

The use of teflon as a column support resulted in symmetrical sharp peaks even at low liquid-phase loadings and column temperatures. A conditioning period of as little as 2 h was sufficient as well. Though the actual percentage of the liquid phase coated could not be determined with any precision, this offers no real difficulty for practical purposes. The handling of teflon is difficult due to its physical properties and its use in organophosphorus pesticide analysis by GLC has not been previously reported. KIRKLAND⁸ studied its properties and devised a special way of coating, but it is cumbersome. The technique used here was as follows:

The glass column and the teflon support are cooled to 0°. The teflon is poured slowly in at one end of the column while a slight vacuum is applied to the other end, and the tube is gently vibrated with a pencil tip. Vigorous vibrations clog the column and produce dead spaces due to adhesion; once this occurs, it cannot be rectified by any amount of further vibration. The entire length of the column is first packed with teflon and then the desired amount of liquid phase in a volatile solvent is percolated through slowly under slight vacuum. The vacuum is continued for 2 h to remove the volatile solvent; otherwise, dead spaces will occur during conditioning. Thin plugs of silanized glass wool are lightly inserted in both ends of the column and, after conditioning for 2 h, the column is ready for use. Temperatures above 250° result in column deformation and noxious fumes are released; hence, use of the column is restricted to lower temperatures.

Gas-Chrom Q was found to be as effective as teflon at low liquid phase loadings (2–5% batch method); the glass columns were filled with gentle vibration sufficient to pack the column without discontinuities or shattering the coating on the particles. Supelcoport is comparable to Gas-Chrom Q support, but these supports differ from teflon in that longer conditioning periods are required to get constant response. It has also been reported in the gas chromatography of steroids that active sites in non-polar columns may be reduced if a small amount of polar phase is introduced into the nonpolar phase⁴. This amount is generally not sufficient to alter the properties of the major phase. However, for organophosphorus compounds even the addition of as little as 0.2% polar phase adversely affected retentions. Another method of coating involved coating the support with 0.2% Epon resin followed by coating with the desired nonpolar phase; no significant advantage was found with this method, however. Thus it can be concluded that for the GLC of organophosphorus compounds, teflon is superior as the solid support.

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