An Apparatus and Method for the Reanalysis of TLC Spots

During the tlc analysis of labile organic materials there exists the possibility of a chemical reaction between the sample and the stationary phase (adsorbent).[1,2] This possibility must be routinely considered when purifying materials by preparative tlc. If an adsorbent-substrate interaction is suspected, it becomes necessary to compare the chromatographed material and the original material on a less active or an entirely different stationary phase.

The transfer of a spot from a developed chromatogram to a second adsorbent is readily accomplished using the apparatus and procedure described herein. A 5-in. × 5-mm i.d. piece of soft glass tubing is heated mid-way between the ends and drawn out to form a thin capillary tip. A small plug of glass wool is placed in the body of the pipet, and the stationary phase containing the material to be reanalyzed is placed on top of the glass wool *via* a small spatula or powder funnel. Several drops of the appropriate solvent are used to elute the sample from the stationary phase. When the solvent reaches the end of the capillary tip, a new spot is made on the second adsorbent. If a volatile solvent is used to elute the sample from the stationary phase, the apparatus should be held with a clamp to allow controlled spotting.

When nonfluorescent compounds are to be reanalyzed, the original spots must not be visualized by the common methods, i.e., sulfuric acid charring, phosphorescent or fluorescent sprays or iodine adsorption. The initial analysis should involve the simultaneous development of two chromatograms. The nonfluorescent material on one of the chromatograms is visualized by the appropriate method and the R_f values of the spots calculated. The spot on the undeveloped chromatogram can now be located, removed, and reanalyzed.

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[1] Smith, L. L., and Foell, T., *J. Chromatog.*, **9,** 339 (1962).
[2] Schreiber, K., and Adam, G., *Monatsh. Chem.*, **92,** 1093 (1961).