

Use of silica gel G slurries in thin-layer chromatography

Silica gel G coatings for use in thin-layer chromatography have been prepared from individual batches of a silica gel suspension in an aqueous medium. Conventionally, a mixture of two parts of water or aqueous solution and one part of silica gel G is prepared by thorough shaking; then the plates are coated within a minute or two before the mixture sets to an unworkable gel¹.

The preparation of a large amount of suspension which could be used to coat a number of plates over a period of time would be considerably more convenient. It has been determined that a mixture of 2.5 parts of either water or of some aqueous solutions to one part of silica gel G will form a slurry* which can be used for at least three months to prepare satisfactory coatings. DAUVILLIER² prepared silica gel suspensions whose setting time was increased by an unstated amount. However, this procedure required modification of the commercially available silica gel G.

Experimental

(a) *Apparatus and reagents.* Silica gel G and silica gel H were Merck & Co. products. All other chemicals used were reagent grade. Brinkmann thin-layer chromatographic equipment was used for the preparation of the coatings.

(b) *Procedure.* 200 g of the desired silica gel was stirred into 500 ml of either distilled water, 0.1 *M* sodium hydroxide, or 0.1 *M* potassium bisulfate. The mixture was shaken mechanically for 0.5 h and then allowed to stand until needed. Just prior to the coating operation the slurry was shaken vigorously to resuspend the silica gel.

Fifty ml of the slurry was used to coat five 20 × 20 cm glass plates. The coated plates were allowed to stand until the slurry set. When water was used, a normal setting time was observed; both the acidic and basic solutions retarded setting for about 10 min. The coated plates were then oven-dried or air-dried as desired.

Discussion

Slurries of silica gel G in water, 0.1 *M* sodium hydroxide and 0.1 *M* potassium bisulfate have each been used to prepare coated plates at intervals over a period of three months. Their use over a longer period of time seems feasible. R_F values obtained on these coatings for a number of organic bases fell within the range of values found³ using coatings prepared from the conventional mixture of two parts of water to one part of silica gel G. The coatings prepared from each of the slurries show as good or better adhesion to the plates and resistance to abrasion as do the conventional coatings. Formation of aggregates of silica gel has not been noted with any of the slurries prepared using the above solutions.

A mixture of one part of silica gel G and 2.5 parts of water sets within 10 min to a loose watery gel; when this gel is stirred it breaks up, giving two phases. Coatings prepared from a mixture which had gone through a "set" stage gave more variable R_F values than the slurries prepared as directed. Mixtures prepared from one part of silica gel G to 2 parts of each of the three solutions given above will set in less than 10 min despite vigorous shaking throughout this time. Shaking is only useful in preventing setting of these mixtures when the proper ratio of solution to silica gel is achieved.

* The word "slurry" is reserved for those mixtures prepared as described in the experimental section.

Shaking the slurry during its make-up probably prevents any structure formation during the hydration of the calcium sulfate. During the drying step this structure formation then takes place in such a manner as to produce a more water-resistant coating. Plates which have been coated using a slurry, and then thoroughly air-dried, lose only the surface layer of the coating when rinsed in a stream of cold water. Most of the silica gel adheres to the glass and must be removed by rubbing. Coatings prepared from the conventional 2:1 mixture are almost completely washed off the plate when treated in the same manner. The adherent layer on plates coated from slurries makes these coatings more resistant to flaking when they are developed in solvent systems containing a high percentage of water.

Not all solutions can be used to prepare satisfactory slurries by the procedure outlined. Slurries prepared with solutions 0.1 *M* in phosphate ion produced, within three days, crystalline appearing aggregates which presumably were calcium phosphate. A slurry prepared using McILVAINE's citrate-phosphate buffer, pH 3.2, was too thin to coat. A mixture prepared using the same buffer in a 2:1 ratio of it to silica gel G coated satisfactorily and did not set to a gel even if not shaken.

Silica gel H slurries are also usable over a period of time. Aside from the higher cost of silica gel H, only one disadvantage of this material has been noted. A silica gel H slurry in 0.1 *M* NaOH after one week's standing contained aggregates of silica gel which could not be dispersed by shaking. The coatings prepared using silica gel H slurries have the same mechanical properties as those prepared from silica gel G slurries. Silica gel H would have an advantage when coatings are prepared using materials, e.g. phosphate ion, which react with the calcium sulfate in silica gel G.

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Separation of trifluralin and some related compounds by two-dimensional thin-layer chromatography

Trifluralin (I) (α,α,α -trifluoro-2,6-dinitro-N,N-dipropyl-*p*-toluidine) is a selective pre-emergent herbicide for use on agronomic crops¹⁻³. It is active against a great variety of broadleaf weeds and annual grasses.

Prior to starting metabolic studies of trifluralin in plant and soil systems, it was necessary to investigate the chromatographic behavior of this compound,

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