Activation and Standardization of Purified Silica Gel for Column Chromatographic Cleanup of Pesticide Residue Extracts

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The introduction of high purity grades of silica gel for column chromatographic cleanup procedures made available effective adsorbents for pesticide residue analysis. These adsorbents have been used for separating pesticides from interfering biological materials (2, 3, 4). However, the need still exists for a general method of reactivating and standardizing silica gel so that its activity can be consistently reproduced.

The objective of the present investigation is to determine the conditions necessary for reactivating the purified silica gel to a satisfactory degree for cleanup of pesticide residue extracts.

Materials and Methods

Reagents and Equipment. All reagents and equipment were the same as previously described (2, 3, 4) with the addition of a muffle furnace.

1Contribution No. 975, Department of Entomology, Kansas Agricultural Experiment Station, Manhattan, Kansas, U.S.A. Research supported in part by Regional Research Projects NC-37 and NC-85, and Kansas projects 687 and 481. The assistance of Mrs. Marie Finocchio is greatly appreciated.
for reactivating the silica gel. The furnace, of a heavy box construction, was electrically heated with a powerstat control.

**Procedure.** Different lots of high purity silica gel grade 950 (60-200 mesh) and 923 (100-200 mesh) were used as received without heat treatment as controls. A 100-gm portion of each lot was weighed out for heat treatment and placed in a shallow porcelain evaporating dish. The furnace was pre-set at the desired temperature. The silica gel in the porcelain dish was positioned in the middle of the furnace with the thermocouple junction directly above it. Heat treatments were conducted at 130°C, 300°C, and 650°C for 2- and 24-hr periods. Upon completion of heating, the sample was put in a desiccator to cool.

In all cases activation was measured by the microcolumn cleanup method (2) as a function of rate of elution of aldrin, dieldrin and retention of a combined pool of evaporated extractives from plants, fish, mice and soil. Hexane and 40% benzene in hexane were used as eluting solvents for aldrin and dieldrin, respectively.

**Results and Discussion**

Different lots of adsorbent do not always yield the same results when used for the cleanup of tissue extracts by liquid-solid chromatography. This variation in lots of adsorbent can be attributed to their origin or minor differences in processing or activation methods. Reactivation and standardization studies were conducted to prepare an effective silica gel for sample cleanup.