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ACTIVATION AND PURIFICATION OF SILICA GEL
FOR FLASH CHROMATOGRAPHY

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1. Procedure

A. Reactivation of Woelm 32-63 μm silica gel. A slurry of 500 g. (Note 1) of used silica gel in 1.5 l of 5% conc. hydrochloric acid in methanol (Note 2) is prepared in a 2-l. Erlenmeyer flask. The slurry is agitated for approximately 30 min. and filtered through a 2-l. coarse glass filter. The acidic methanol is discarded and the moist silica gel is scooped (Note 3) back into the 2-l. flask. The silica gel is thoroughly mixed with 1-l. of distilled water and filtered. A second wash with 1-l. of distilled water is performed. The silica gel is then heated without agitation on a steam bath in 1.5 l. of 5N nitric acid (Note 4) overnight. The next day, the supernatant liquid is decanted (Note 5) and the silica gel is washed, as previously described, until the filtrate is neutral (Note 6). The moist silica gel (Note 7) is scooped into two 100x190-mm crystallizing dishes and placed in a 100-120°C-drying oven until constant weight is attained (Note 8).

B. Purification of Alfa large-pore 58 μm silica gel (Note 9). The purification of Alfa's silica gel is the same as described above starting with the nitric acid wash.

2. Notes

1. The checkers find that two 500-g portions may be conveniently purified in tandem. A 40-mm column requires approximately 130 g of silica gel.
2. MCB reagent grade hydrochloric acid and Fischer reagent grade methanol were used.
3. A tablespoon is conveniently used for scooping silica gel.
4. 5N nitric acid is prepared by adding sufficient water to 298 ml of concentrated (Fischer, reagent grade) nitric acid to form 1 l. of solution. Overnight heating is not necessary for Woelm silica gel, but is recommended for Alfa silica gel.
5. Decanting the supernatant removes very fine particles that may cause undesirable high head pressures during Flash chromatography.
6. The pH of distilled water is commonly 5-6.
7. The silica gel becomes an unmanageable powder if allowed to dry in the funnel.
8. Drying requires approximately 72 hrs.

9. The color of Alfa's silica gel is variable, from white to "Pirkle Gray."

10. We have shown the Alfa silica gel to be superior to Woelm's in a series of test flashes. Results available upon request.

3. Methods of Preparation

These methods are fairly standard methods of purification.¹ The nitric acid wash is that used by Pirkle.²

4. Merits of Procedure

The prohibitive cost of Woelm silica gel (currently \$44/kg.) as well as its limited availability led us to regenerate it. During the development of this method, Pirkle suggested the use of Alfa's silica gel (currently \$8.50/kg.). While this silica gel can be used without purification, it is not recommended because polar solvents will extract a yellow residue from it.

This method is a simple procedure for obtaining high quality silica gel for routine flash chromatography. We have shown the Alfa silica gel to be superior to Woelm's in a series of test flashes. The results are available on request.

1. W. L. F. Armarego and D. O. Perrin, Purification of Laboratory Chemicals, Pergamon Press, N.Y., 1966.
2. Personal communication with Dr. W. H. Pirkle, University of Illinois.