

CHROM. 8718

Note

Preparation of octadecyl Porasil for reversed-phase liquid chromatography

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(Received September 2nd, 1975)

In an earlier communication, we described the use of reversed-phase liquid chromatography in the separation of various indole alkaloids¹. Owing to the expense of commercially available bonded phase packings, we sought a reproducible method for the preparation of the octadecyl phase (C₁₈-Porasil) used in this work. Several methods for the preparation of bonded phases have been published previously, including a helpful recent general paper by Majors and Hopper², and the subject has recently been reviewed³, but comparison with currently available packing materials is not available for most of these. This note reports a simple method for the preparation of a bonded octadecyl phase packing which approximates closely in its chromatographic properties a widely available commercial packing.

EXPERIMENTAL

Chemicals

Octadecyltrichlorosilane and trimethylchlorosilane were obtained from Aldrich (Milwaukee, Wisc., U.S.A.) and were used as supplied. Porasil B[®] and Bondapak C₁₂/Porasil B[®] were purchased from Waters Assoc. (Milford, Mass., U.S.A.). Toluene was reagent grade and was dried over calcium hydride and distilled before use.

Octadecyl Porasil B

Porasil B (300 g) was heated for 12 h at 100° in a mixture of 500 ml concentrated nitric acid and 500 ml concentrated sulfuric acid. After cooling and decantation of excess acid, the residue was washed with distilled water by decantation until neutral, and then dried at 110° for 12 h. The dry Porasil B was suspended in 800 ml dry toluene and 90 ml octadecyltrichlorosilane, and the mixture heated under reflux for 14 h. The excess toluene and chlorosilane were decanted off, the residue washed with dry toluene to remove any remaining chlorosilane, and the bonded chlorosilane was then hydrolyzed by stirring the Porasil with 50% aqueous acetonitrile for 2 h at room temperature. The aqueous mixture was then decanted and the residue washed thoroughly with water and dried. Final protection of free hydroxyl groups was achieved by reaction of the dry powder with 10% trimethylchlorosilane in dry toluene (500 ml) for 2 h under reflux, followed by final washes with toluene and methanol and drying at 110° for 3 h.

RESULTS

The octadecyl Porasil B obtained by the above procedure was compared with the commercially available Bondapak C₁₈/Porasil B by elemental analysis and by chromatographic properties. The results of elemental analysis are shown in Table I, and clearly the extent of coverage of the bonded phase is very similar for the commercial material and for the homemade material.

TABLE I
ELEMENTAL ANALYSIS

Packing	Carbon (%)	Hydrogen (%)
Bondapak C ₁₈ /Porasil B	11.07	1.95
Octadecyl Porasil B	11.21	1.98

The homemade material was also compared with the commercial material as a chromatographic packing. Columns (2 ft. × 1/8 in.) of each material were packed by the tap-fill method, and evaluated using a test mixture of benzene, naphthalene and biphenyl. The chromatogram obtained with the homemade material using a solvent of methanol-water (75:25) was essentially identical to that obtained with the commercial material using an 80:20 mixture of the two solvents. The homemade material thus can serve as an acceptable substitute for the commercial packing. In addition, the bonding technique described here should be applicable in the preparation of bonded phases of microparticulate materials such as the Partisils[®], Spherisorb[®], and μ Bondapak C₁₈[®], where the octadecyl bonded phase packings are not yet commercially available.

ACKNOWLEDGEMENT

Support of this work by grant number CA-12831-04 of the National Cancer Institute is gratefully acknowledged.

REFERENCES

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