

POLYIMIDE COATED CAPILLARY TUBING: INTERNAL SURFACE MODIFICATION

Polyimide coated fused silica capillary tubing is widely used in the separation sciences. In this application note, we share perspectives on modifying the internal surface of capillary tubing.

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INTRODUCTION

Fused silica capillary tubing is used in a broad range of analytical instrumentation and applications. Modification of the internal surface is established technology in fields such as GC and CE. Capillary with internal coatings can be purchased commercially in the form of GC columns or deactivated tubing. Some covalently bonded stationary phases used in CE are available as windowed capillary, but the selection is limited. Much academic work has been published, with Jorgenson routinely cited (1). Although there is copious published literature detailing surface modification, a short overview for the novice is provided. In addition, details for having an internally coated capillary produced on a custom basis are discussed.

SURFACE PREPARATION AND MODIFICATION

When drawn, the fused silica tubing internal surface is fully dehydrated and is comprised primarily of siloxane bridges (2). A uniform population of silanol functionalities is desired prior to i.d. surface modification. Although many hydration processes are documented in the literature, the following has been employed with good success on 50 μm i.d. tubing: 15–30 min wash with 1 M NaOH; 5 min rinse with DI water; 15 min wash with 0.1 M HCl to bring down the Na⁺ concentration; 15 min rinse with DI water; purge with inert gas to remove bulk water and then heat to 180°C with inert gas purge for 30 min, and bring back to room temperature under inert gas purge. All reagents and gases should be properly filtered. Reaction times vary with i.d. and tubing length; adjust times so at least three column volumes are eluted. This procedure will populate the surface

uniformly with silanol groups, remove excess cationic species, and eliminate the majority of physically adsorbed water. The surface will be ready for reaction with surface modification reagents such as chlorosilanes, silazanes, ethoxysilanes, methoxysilanes, etc. For the novice, ethoxysilanes are recommended, as the primary reaction byproduct is ethanol. By-products of the more aggressive reagents, i.e., chlorosilanes, should be considered and well understood prior to use.

CUSTOM PRODUCTION OVERVIEW

In many instances, the desired internal coating either is not commercially available, or is challenging to produce from an equipment standpoint, and must be made on a custom basis. With surface preparation defined earlier, the reaction steps and reagent requirements to achieve the desired final coating must be selected. Solutions are prepared in a dry box, transferred into small test tubes, and then loaded into the manifold shown in Figure 1. This 13-port, high pressure manifold is used for custom internal coating processes. Head pressures up to 2500 psi are used to force reagents through the capillary. An insulated water bath regulates reaction temperatures, with stability critical in some polymerization efforts. Effluent is collected and monitored as needed to verify the process. If higher temperatures are needed, a forced air oven is employed.

Production lengths vary with i.d. It is common to run 50–100 μm i.d. tubing in 100 m lengths with smaller i.d. requiring shorter lengths. Typical minimum batch size is 100 m. The system is designed for high volume production where job quantities can exceed 1 km per run when all 13 ports are employed.



Figure 1: One of Polymicro's 13-port, high pressure manifolds with associated temperature control bath.

CONCLUSION

Capillary tubing with internal coatings is vital in many capillary applications. Surface preparation and modification, as well as production considerations have been discussed. For questions on your specific application, please contact a Polymicro Technical Sales Specialist.

REFERENCES

- (1) J.W. Jorgenson and K.D. Lukas, "Capillary Zone Electrophoresis," *Science* 222(4621), 266–272 (Oct 1983).
- (2) J. Macomber and D. Stasiak, LCGC Application Notebook, p. 63 (June 2008).

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