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Note

A true full-length thermostating water jacket for Swagelok-terminated high-pressure liquid chromatography columns

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The theoretical significance of the adequate thermostatic control of high-pressure liquid chromatography (HPLC) columns is well known, although in practice it is frequently overlooked. Sometimes, the resolution between two compounds can change by as much as 12% for a 1° change in column temperature[1]. The widespread use of chemically bonded phases also calls for elevated column temperatures, and thermostatic control with an oven or water jacket is currently employed in commercially available HPLC systems. Apart from a very few carefully designed (although usually complicated) water-jacket systems[2], most systems give inadequate thermostatic control of the full length of the column, and their design is also rather complex. A relatively simple jacket system has recently been described[3] for jacketting long, narrow-bore, coiled ion-exchange columns.

In this paper we describe a simple, efficient, easy-to-use and cheap means of jacketting home-made or commercially available HPLC columns equipped with Swagelok or similar column terminators. Our solution is based on the principle that the nuts of the terminator can be used to achieve leak-free jacketting with the help of elastic septa.

Commercial HPLC columns are nowadays sold closed either at both ends (the usual type, *e.g.*, μBondapak-NH, Waters Assoc., Milford, Mass., U.S.A.; Partisil, Whatman, Clifton, N.J., U.S.A.) or only at one end (*e.g.*, MicroPak-NH_2, Varian Aerograph, Walnut Creek, Calif., U.S.A.). Our system can be readily employed with either type, and it is not limited as far as the column dimensions are concerned. In this paper we consider the dimensions of the jacket of a 0.3 m × 1/4 in. O.D. μBondapak-C_{18} column (Waters Assoc.) as an example.

The water jacket, shown in Fig. 1, can be made of either glass or metal. Sealing between the jacket and the column is achieved with a silicone rubber septum compressed into the conical end of the jacket. Compression is effected by tightening the nuts on the 1/16-in. sides of the 1/4–1/16-in. terminators. In order to ensure uniform compression and safe, tight sealing, a thin metal washer is placed between the nut and the septum. The surface of the Swagelok terminator at the 1/4-in. side is generally sufficiently large to act as a counter surface to achieve proper compression. Should this not be the case with other terminator sizes, however, an additional washer placed between the Swagelok body and the septum solves the problem.

The design of the water jacket permits easy, *in situ* assembly. Assuming that

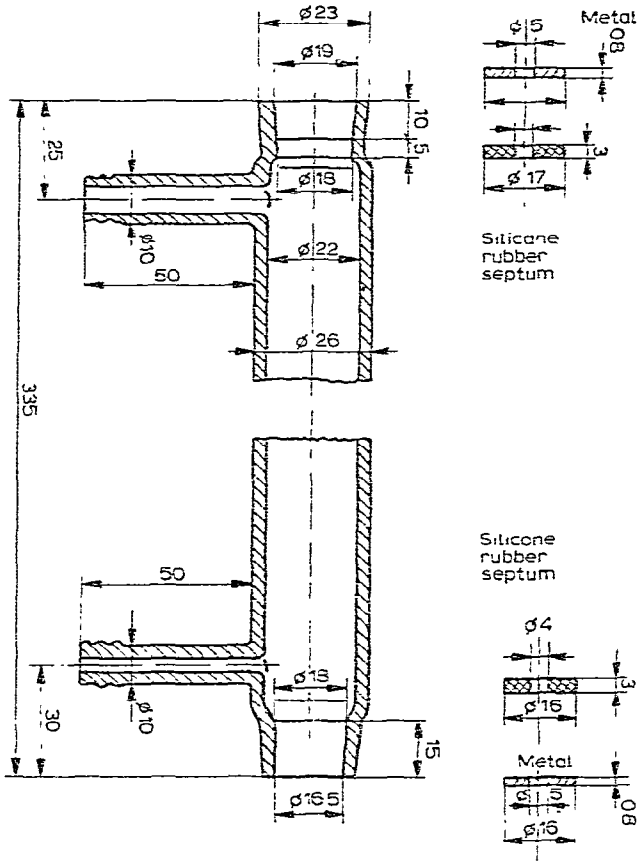

Fig. 1. Dimensions (mm) of the glass jacket of the 0.3 m × 1/4 in. O.D. μBondapak-C_{18} column.

the column is coupled directly to a U6K universal injector (Waters Assoc.) and a UV detector (Varian Aerograph), the jacket is assembled as follows (Fig. 2).

Cut a silicone rubber ring, run the ring on to the 1/16-in. side of the column outlet terminator and run a metal washer over the ring. Push the column into the jacket, connect the 1/16-in. inlet tube of the UV detector to the outlet terminator by tightening the nut on it so as to achieve a leak-free column–detector coupling. The silicone rubber easily swells out and does not prevent tightening of the nut. The nut is best tightened with a special, sleeved hexagonal tube-wrench.

Allow the inlet side of the column to project about 1/2-in. out from the jacket. Run another silicone rubber septum and metal washer on to the 1/16-in. side of the Swagelok inlet terminator, connect the eluent inlet line and tighten the nut to give a leak-free joint while the inlet terminator is projecting from the jacket. Start the pump and check for occasional leaks, and re-tighten the nuts if necessary (upon completion of the assembly of the jacket, slight leaks can no longer be detected at the nuts). Tightening the nuts makes the flexible rings swell sufficiently to ensure a leak-free seal. Push the assembled column system tightly into the jacket until the septa are sitting firmly in the conical parts of the jacket. Start the circulation of the thermostating water-

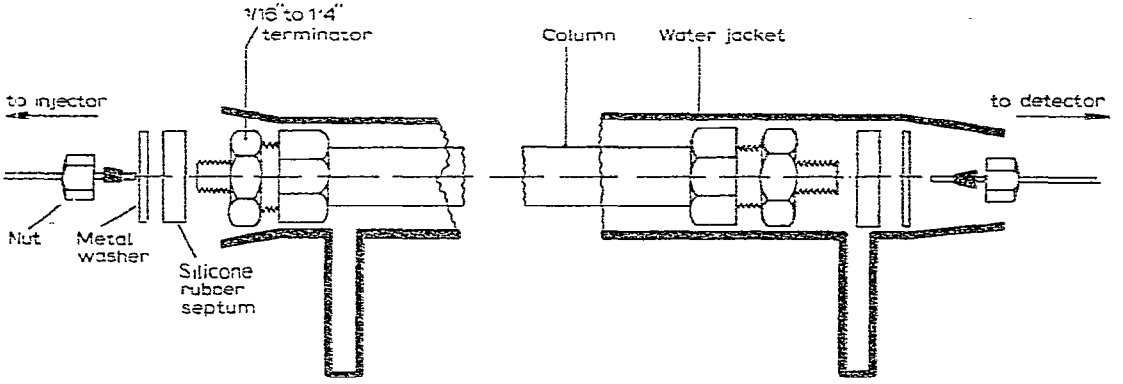

Fig. 2. Schematic diagram of the assembled jacket.

bath. If leaks are observed, push the column deeper into the jacket so that the septa sit more firmly. To change the column or to dismantle the jacket, reverse the operations given above.

If columns that are terminated only at one end, *e.g.*, MicroPak-NH_2, are to be jacketted and the columns are to be coupled to the same U6K injector, the procedure is the same with a suitable 1/16–1/8-in. fitting coupled to the column. If the column is to be connected to other injectors (*e.g.*, a Varian low-pressure septum injector, Cat. No. 02-001469, or a high-pressure stop-flow injector, Cat. No. 02-001652), the

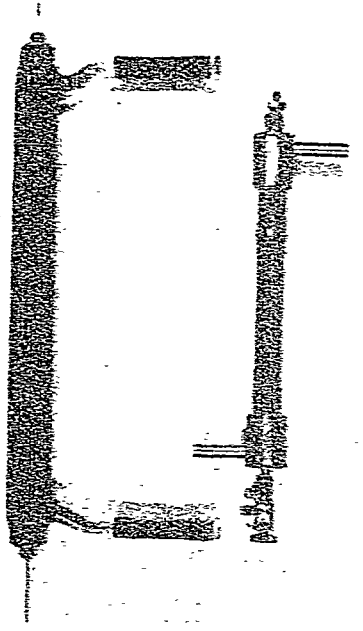

Fig. 3. Photograph of a commercially jacketted column (Varian Aerograph) (right) and of a μBondapak-C_{18} column (Waters Assoc.) jacketted by our method (left).

outlet terminator is treated as before. At the inlet side, however, a metal washer is run on to the injector followed by the silicone rubber septum and another metal washer. Finally, the nut on the column is connected to the injector, making the septum swell out properly. The column is then pushed firmly into the jacket to achieve a tight seal.

Fig. 3 shows a commercial jacketted column (Varian Aerograph) compared with a μBondapak-C_{18} column (Waters Assoc.) jacketted by our method.

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