

CHROM. 5184

Adjustable fittings for very small glass columns

In amino acid analysis the use of 9-mm diameter columns is very common. Many fittings for 9-mm columns are accordingly commercially available. However, the use of smaller columns in amino acid analysis and other chromatographic analytical systems is attractive, since the use of smaller columns increases the sensitivity. The use of these small columns is especially inviting when automatic sampling¹⁻⁵ is possible, because otherwise the sample introduction may cause problems. However, in this case, it is necessary to have an adjustable plug above the resin bed. In our laboratory a simple and practical combination of adjustable plug and fitting has been designed for columns with diameters of 6 and 4 mm. Most parts of these fittings are commercially available.

Description of the fittings

Two kinds of plugs have been designed. The design of the adjustable plug is shown in Fig. 1. A 316 stainless steel Crawford reducer with Zytel ferrules is used⁶. Part of the reducer is a hexagonal screwcollar [5/16 in. = 8 mm], which is 4.9 mm wide. 2 mm of this collar is partially turned off and a round 316 stainless steel plate is welded to the reducer in this position (4, Fig. 1). A circular groove is turned in this plate, in which the viton O-ring (5) is placed, such that 0.5 mm of the O-ring projects beyond the steel. A 316 stainless steel injection tube with outside diameter 1.6 mm (6) is fitted in the reducer. The reducer had been slightly bored out, to allow this tube to pass through. When the nut (1b) is adjusted, the tube becomes tightly applied to the reducer. A heated plug (7) was allowed to cool and contract on to one end of the tube. In this plug a groove for the O-ring (8) is turned. The outside of the plug has been cut so as to leave six small supports and a brim in relief (9). This has been de-

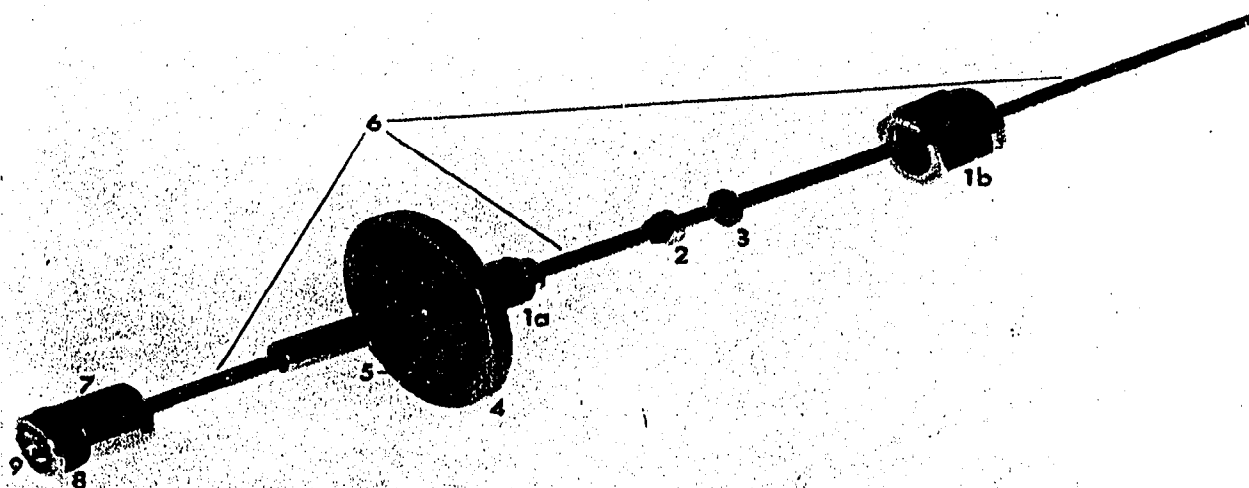


Fig. 1. Design of the adjustable plug. 1a, b = Reducer 100-R-2-316; 2 = front ferrule 103-1 (Zytel); 3 = back ferrule 104-1 (Zytel); 4 = 316 stainless steel plate, thickness 2 mm; 5 = viton O-ring, parker size 014 (012); 6 = injection tube; 7 = plug; 8 = viton O-ring, parker size 006 (003 for 4 mm columns); 9 = projections in the plug.

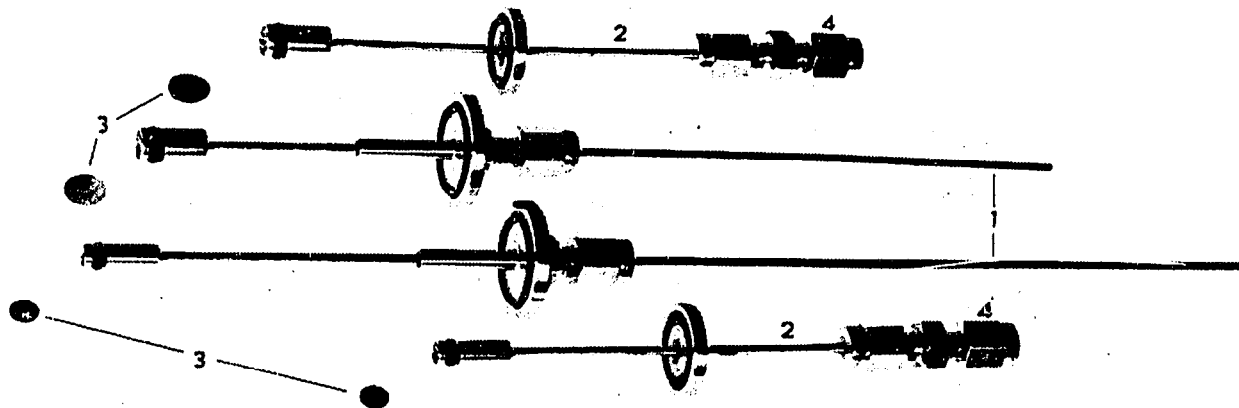


Fig. 2. Different fittings. 1 = adjustable fittings; 2 = fixed fittings; 3 = stainless steel filters; 4 = Union 100-6-316 for connection with pump or coil and ninhydrin system.

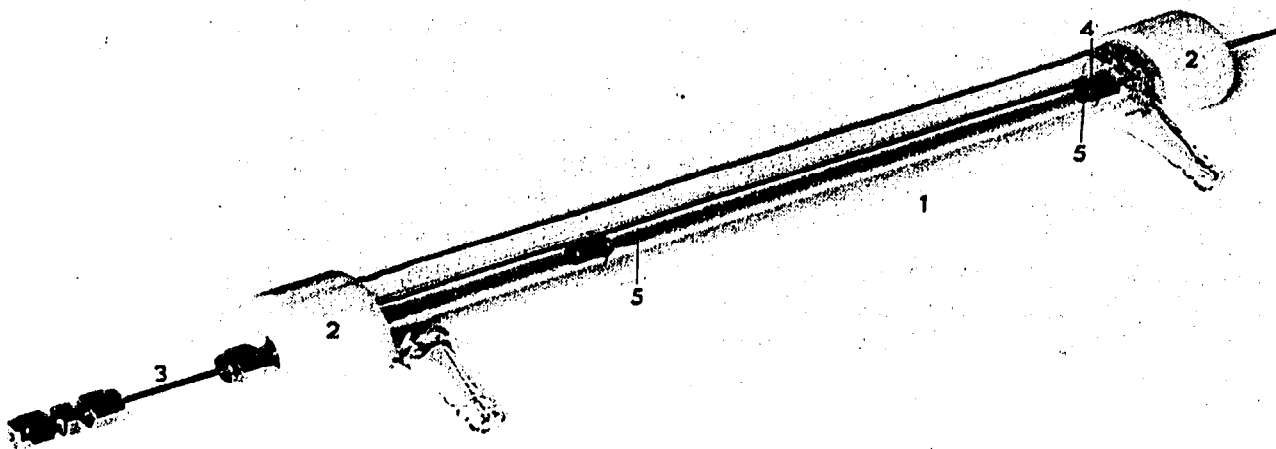


Fig. 3. Column and fittings. 1 = column; 2 = Delrin screw caps; 3 = adjustable fitting; 4 = non-adjustable fitting; 5 = filters.

signed so that a stainless steel filter (3, Fig. 2) is supported under the brim and centred.

The stainless steel filters are made from Beckman stainless steel filters No. 938514. The non-adjustable plug for the base of the column differs only in the fact, that the plate (4, Fig. 1) has been welded directly on to the injection tube. Fig. 2 gives an illustration of the different connectors. In Fig. 3, the attachment of the fittings to the column is shown. A fitting screw thread was pressed on the end of the column (precision bore), and two Delrin screw caps tighten the fittings to the glass column by means of the stainless steel plate (4, Fig. 1).

Discussion

The design for the column fittings as given here is the product of experiments with different kinds of prototypes. The critical points are:

(1) The possibility of adjusting the plug on top of the resin bed. The solution to this problem has been found in the use of Zytel ferrules in a stainless steel reducer.

(2) The necessity of avoiding cracking the column when adjusting the fitting on top of the column. This has been solved by the use of the O-ring (5, Fig. 1).

(3) The adjustment of the plug above the resin bed can be performed simply on the column. When the Delrin screw caps (Fig. 3) are fastened the O-ring (5, Fig. 1) will resist against turning with respect to the glass of the column. The nut (1b, Fig. 1) can be loosened, the plug adjusted, and the nut fastened again.

(4) The contraction of the plug (7, Fig. 1) on to the injection tube (6, Fig. 1). The sodium citrate buffers in use in amino acid analysis are very corrosive. Therefore welding the plug on the tube, *e.g.*, with silver, was not an attractive method. Contracting the plug onto the tube proved to be the solution. In practice there appeared to be no leakage.

(5) The filters (2, Fig. 2) must not be deformed on the column fitting. Therefore the protruding supports (9, Fig. 1) were made.

(6) The filters should fit very tightly in the column. Therefore columns of precision bore (0.6 and 0.4 mm) were used. The filters were very carefully cut from Beckman stainless steel filters No. 938 514.

All the problems (1-6) were solved by the design as given. In practice the fittings prove to be very satisfactory.

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