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## Note

### Polyamide-polyimide block copolymers as ferrules in gas chromatography

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Until recently, ferrules of PTFE, filled PTFE (including that with glass fibre) or graphite were used in our laboratory for sealing glass columns to gas chromatography systems. While pure PTFE presented difficulties at temperatures above 200°, owing to its irreversible flowing, ferrules of filled PTFE or graphite permitted temperatures of up to 300° to be used for a short time. However, undesirable distortions occurred with an adverse effect on the life of the seals and their repeated use.

Ferrules of commercially available polyimide also have shown irreversible distortions when subjected to mechanical pressure and elevated temperatures, as encountered during GLC procedures, leading to difficulties in removing the columns from the connections after use.

We have now been able to overcome some of these difficulties by using ferrules made of thermoplastic polyamide-polyimide block copolymers developed at Ciba-Geigy Central Research, Basle<sup>1</sup>. These materials have glass-transition temperatures in the region of 250-300° and have excellent thermal oxidative stability. They have outstanding mechanical properties (*e.g.*, flexural strength, according to VSM 77103, of moulded 60 × 10 × 4 mm bars of up to 35 kg/mm<sup>2</sup>) and good dimensional stability under compressive loads at elevated temperatures.

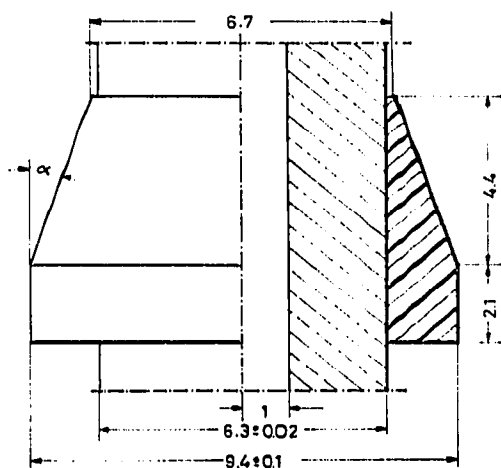


Fig. 1. Ferrule of Ciba-Geigy polyamide-polyimide block copolymer. Calculated angle,  $\alpha = 19-19.5^\circ$ ; empirically derived angle,  $\alpha = 18^\circ$ .

The calculation of the optimum cone angle (the angle which gives a maximum sealing surface with minimum surface pressure) of ferrules for Swagelok fittings gave a value of 19–19.5°. In practical tests with 1/4-in. glass columns, a slightly smaller angle of 18° gave the best performance, combining good sealing properties with ease of disassembly after high-temperature operation (Fig. 1).

Ferrules made of thermoplastically workable Ciba-Geigy polyamide–polyimide block copolymers showed excellent sealing properties up to 300°. A very minute deformation of the material under load (tightening the ferrules), observed at temperatures in the glass-transition region, even enhanced the sealing properties.

For the best results, a torque wrench (*ca.* 3 J) should be used for tightening the ferrules. The controlled initial pressure thus produced ensures secure sealing and prevents glass breakage. After a short heating period, it is advisable to re-tighten the system so as to ensure reliable sealing over a long period, even under conditions of temperature programming.

#### REFERENCE

- 1 D. Lohmann, P. Furrer, R. Darms and G. Greber, *Deutsche Offenlegungs Schriften*, 2 342 464, 22.8.73/7.3.74.