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Strategies for Reducing Volumetric Variance in Precision Small-Bore Flexible Fused Silica Capillary Pieces ›

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Recent trends in separation sciences have driven an increased demand for tight-tolerance small-bore capillary tubing. In applications where volumetric precision is required, cut capillary pieces are proving invaluable. Strategies for producing tight-tolerance cut capillary pieces will be discussed.

Introduction

In recent years, separation science practitioners have continued to expand the use of tight-tolerance small-bore flexible fused silica capillary tubing. These capillaries allow researchers to achieve reproducible volumes within LC system components such as injection loops, connection lines and packed or monolithic columns. Microfabricated devices often employ tight tolerance capillary products for introduction of samples onto chips where reproducibility of system volumes is critical. As chromatographic separation devices continue to evolve and demand tighter volumetric control, precision small-bore capillary tubing will play an increasingly important role.

For over two decades, Polymicro has consistently demonstrated leadership in this area by periodically tightening i.d. tolerances on standard small-bore products. Advancements in manufacturing processes and metrology now allow for production of custom drawn small-bore capillary with i.d. tolerances down to ±1 µm. Strategies for the production of volumetrically precise cut pieces are a logical extension of such efforts and will be discussed herein.

Discussion

Cost-effective production of small-bore capillary pieces with optimal volumetric precision begins by drawing the capillary to tight tolerances followed by i.d. verification for conformance to specification; both of which have been discussed previously. (1) Reducing volumetric variance in subsequently produced cut capillary pieces is rooted in the frequency of i.d. verification. Polymicro has developed a production flow path which strikes an effective balance between measurement cost, confidence in adherence to specification and net production yield. The recommended i.d. verification frequency is every 10m for capillary with i.d.'s less than 50 µm. End samples from each 10m spool are measured and recorded; each spool is assigned a specific lot number for effective traceability.

Meeting volumetric specifications involves consideration of both length and i.d. tolerances; the later will be discussed first. Spool end sample measurements are averaged and this value is used to represent the i.d. of each 10m lot.

Table I: Effect of i.d. tolerance on volumetric error for 20 µm i.d. products. Part length is 100 ± 1 mm.

i.d. tolerance	% Maximum Volumetric Error
± 2 µm	22.20%
± 1 µm	11.40%
± 0.3 µm	4.10%
± 0.1 µm	2.01%

When all lots of precision small-bore tubing drawn with i.d.'s less than 50 µm were reviewed, the maximum variation within any 10m lot was found to be ± 0.3 µm. Greater than 95% of the lots examined showed a variation of ± 0.1 µm or less.

Length tolerances of ±1mm or less can be achieved on parts up to 1m. Table 1 shows the calculated maximum expected variation in volume for 100 ± 1mm long, 20 µm i.d. cut capillary pieces made from tubing produced to different i.d. tolerances as described above. It is clear that employing the strategy described herein yields the most precise volumetric control for individual lots of capillary.

Strategies for minimizing the influence of lot-to-lot i.d. variation on cut piece volumes have been considered. The desired volume and average i.d. are used to calculate the target length for parts produced from each lot. The parts are then cleaved to this target length. By utilizing the individual lot i.d. measurements and allowing the length to vary correspondingly, volumetric variance from lot-to-lot is minimized.

Conclusion

Variation in i.d. is the most significant factor influencing volumetric precision when producing cut capillary pieces. Therefore a strategy focused on limiting the impact of i.d. variation is being utilized. This concept has been effectively extended to flow rate control applications. Contact Polymicro for all of your precision capillary needs.

References

(1) J. Macomber, P. Nico and G. Nelson, *LCGC Application Notebook*, June 2003, p.63.

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