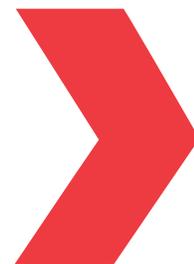


INTERNAL DIAMETER MEASUREMENT OF SMALL BORE CAPILLARY TUBING



Synthetic fused silica capillary tubing continues to play an ever-expanding role in the separation sciences. In this application note, we share some initial data related to ID control in small-bore capillary tubing.

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Scientists use fused silica capillary tubing routinely in a wide range of analytical applications including GC, CE, Capillary LC and CEC. Its unique properties make it a popular tool for interfacing in hyphenated techniques. The advent of monolithic based separation columns appears to be another ideal application for capillary. As the Separation Sciences press forward, a general trend toward smaller bore capillary tubing has emerged.

Polymicro is unmatched in meeting this market demand: 17 sizes with ID of less than 50 μm are available as standard products. In response to customer requests, Polymicro continues to explore tightening of the ID tolerances. It is clear that meeting this demand will require advancements in manufacturing processes and metrology. It is not enough to say one can make capillary tubing to $\pm 1\mu\text{m}$ tolerances. The key is to prove it through a mutually agreed upon methodology that both Polymicro and its customers can depend upon.

EXPERIMENTAL

Synthetic fused silica capillary tubing used in this study was TSP020375, drawn using standard draw conditions and normal operating parameters. Individual samples were measured using a Pinnacle Vision System. The system was outfitted with a 20X lens and then calibrated using a circular target which was independently certified to an accuracy of $< 0.5\mu\text{m}$. Gauge R&R studies indicate an expected error of 0.07 μm (6-sigma) for this method.

Traditional microscope methods have greater measurement error. Gauge R&R studies indicate an expected error of 0.7 μm (6-sigma). Although this is perfectly acceptable for large ID capillary, it is not optimum for smaller ID products with exacting specifications.

During the draw, individual lots of capillary were collected in predetermined lengths of 110m. Each lot was further segmented and end measurement samples taken every 10 m. The ID of each sample was then determined using the vision system. It should be noted that this system was used to verify the ID accuracy during tower set-up.

RESULTS

ID data collected from 4 lots of capillary are shown in Figure 1. The ID specification of TSP020375 is $20 \pm 2\mu\text{m}$, which was clearly met on all lots. The average value for the complete set of data is 20.1 μm , with a standard deviation of 0.54 μm . The range within each lot varied from 1.2 μm down to 0.4 μm . In fact, 96% of the data met a $20 \pm 1\mu\text{m}$ ID specification. 4-sigma performance was achieved. It is evident that through state of the art process control and metrology, providing tubing to ID tolerance of $\pm 1\mu\text{m}$ is achievable over long lengths.

CONCLUSION

As the chromatography market drives to smaller ID tubing, it is increasingly important to be able to offer material with tighter ID tolerances. It is equally important to have the metrology to verify that the material meets the specifications. The data presented in this note represents one of several studies currently underway at Polymicro.

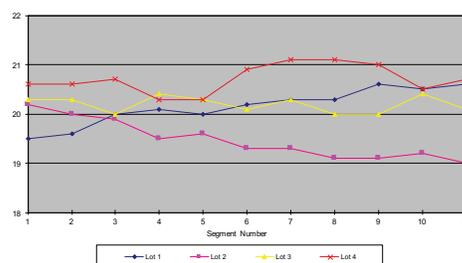


Figure 1: Lots 1, 2, 3 and 4 correspond to randomly selected lots taken at different points within a 1.5 km draw of capillary. Avg. = 20.1 μm , $\sigma = 0.54\mu\text{m}$.