Capillary for Gas Chromatography: Examination of Internal Diameter ›

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Gas Chromatography column manufacturers rely on synthetic fused silica capillary tubing as the substrate for many of their column products. This application note examines the dimensional control of key parameters within a single production run of capillary tubing.

Introduction

Gas Chromatography has seen a long list of advances through its history. Improvements in the capillary substrate itself have been numerous; they include increased product durability, longer spool lengths and higher purity raw materials. Notable improvements in product specifications have also been realized, with dimensional tolerances growing increasingly tighter over the material's 25-year history. The end result of these many advancements is production of more reliable, reproducible GC columns.

A previous study investigated the dimensional control of GC capillary products from a statistical perspective. End of spool measurements from several months' worth of production were analyzed and the results summarized.[1] In a continuing effort to better define existing capabilities, a single production run has been closely examined and the results are discussed below.

Experimental

Synthetic fused silica capillary tubing product TSP320450 (0.32mm ID) was used in this study. Product specifications are: ID 320 ± 6µm, OD 435 ± 10µm. Tubing was drawn from a standard preform and produced using normal draw operating parameters. The production run, totaling ~9000m, was cut approximately every 200m and end samples collected for measurement **(Data Set A)**. One 200m section was further segmented and end measurement samples taken every 10m **(Data Set B)**. Finally, a 10m section, from within the 200m segment, was cut every 0.5m and samples collected for measurement **(Data Set C)**.

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 < 0.5µm. A gauge R&R study of a single piece measured a minimum of 30 times indicated an expected error of 1.2µm (6-sigma) for this method.

Collected samples were cleaved using Polymicro's semi-auto cleaver. The polyimide was removed from the tip of each cleaved sample so the Vision system lighting allowed for the most accurate measurement of ID. ID was based on the best-fit circle using a minimum of 50 annular data points.

Results

Figure 1 summarizes the findings of this study. The first data point, labeled "Spec.", indicates the current ID specification for TSP320450, with the error bar representing the tolerance. Chart labels A, B & C represent the data sets described earlier, with the error bars indicating the standard deviation. Data Set A had an average ID value of 322.6µm with a standard deviation of 1.37µm. Data Sets B & C had average ID values of 321.7µm & 321.1µm, respectively and corresponding standard deviations of 0.85µm & 0.31µm.

This data suggests that there are only sub-micron variations in the capillary ID over typical GC column lengths. Further, the data shows that during a typical draw the actual range of capillary ID produced spans only part of the tolerance range. The ID range over the complete draw was less than 7µm.

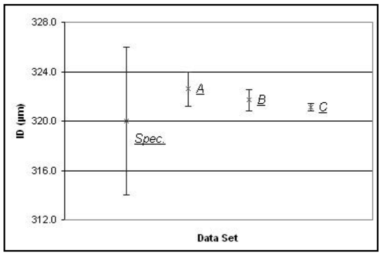

Figure 1: *Summary of average ID by Data Set. Spec. = Product Specification and Tolerance,* ***A*** *= Data Set A (322.6µm, σ 1.37µm),* ***B*** *= Data Set B (321.7µm, σ 0.85µm),* ***C*** *= Data Set C (321.1µm, σ 0.31µm). Error bars on Data Sets indicate Standard Deviation.*

Conclusion

The data above shows excellent stability of capillary ID over the course of a typical production run of 0.32mm ID GC capillary. Additional data related to ovality and concentricity will be presented in a future note as part of Polymicro's ongoing commitment to advancements in capillary manufacturing.

References

(1) J. Macomber, P. Nico and G. Nelson, LCGC Applications Notebook, Feb. 2004, p. 66.

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