Innovative Insights

SCIENTIFIC

# Improved Internal Diameter Control of Tubing Used for Gas Chromatography ›

Joe Macomber and Roland Fischer
Molex Incorporated

Synthetic fused silica capillary tubing is used in the manufacture of most Gas Chromatography columns. In this note we discuss recent advances in the dimensional control of the most popular tubing sizes.

A historical review of the dimensional specifications for flexible synthetic fused silica capillary reveals a clear effort to reduce both i.d. and o.d. tolerances. Previous work summarized the changes in published specifications and presented empirical data on i.d. and o.d. variability (1). Additional studies examined in-draw stability (2). Efforts to further improve the dimensional stability of tubing used to manufacture GC columns is warranted by the potential for improved chromatographic performance, as well as by ease of column use as the industry moves toward plug and play concepts. This application note provides a comparative review of these critical dimensional parameters, with a focus on the i.d.

## Experimental

Capillary tubing employed in this study included TSP250350 (0.25mm i.d.), TSP320450 (0.32mm i.d.) and TSP530660 (0.53mm i.d.). The tubing was manufactured using current draw conditions and operating parameters. Of noted difference to previous work, improvements have been implemented in the integration of new control limit concepts and trend monitoring software algorithms.

Measurements were collected using an Olympus PME3 inverted microscope, which was calibrated and configured as reported previously (1). Gauge R&R studies continue to be conducted so that operator contributions to variability are understood.

Over the period of the study samples were collected and measured from both ends of every spool of material produced. The study time frame ensured a minimum of 1,400 sample values (N > 1,400).

## Results

A summary of both past and present statistical data for the capillary i.d. can be found in Table 1. Mean values are on target for all products studied. The standard deviation on all products has decreased, indicating an improvement in process control. The Cpk values for i.d. obtained in the past study ranged from 1.02 for 0.53mm tubing to 1.23 for 0.25mm tubing. These have improved to 1.12 and 1.55, respectively. Recall that Cpk quantifies the process capability and is defined as the ratio of tolerance to 6$\sigma$. A Cpk value of 1.0 or better suggests that the process is in control. The reader is reminded that both ends of every spool produced is physically measured and must be within specification for release to the market. For comparative purposes, a histogram of TSP320450 is shown in Figure 1. Data on o.d. has shown similar improvements, although they have not been as dramatic. It should be noted that primary efforts have focused on reducing i.d. variability.

**Table I: Summary of GC tubing study results, past and present**

| Product | Current Specification | 2004 i.d. Statistical Data | | | 2011 i.d. Statistical Data | | |
|---|---|---|---|---|---|---|---|
| | ID | Mean | Std Dev | Cpk | Mean | Std Dev | Cpk |
| TSP250350 | 250 ± 6 | 250.4 | 1.62 | 1.23 | 250.5 | 1.29 | 1.55 |
| TSP320450 | 320 ± 6 | 320.5 | 1.76 | 1.14 | 320.9 | 1.44 | 1.39 |
| TSP530660 | 536 ± 6 | 536.4 | 1.97 | 1.02 | 536.3 | 1.78 | 1.12 |

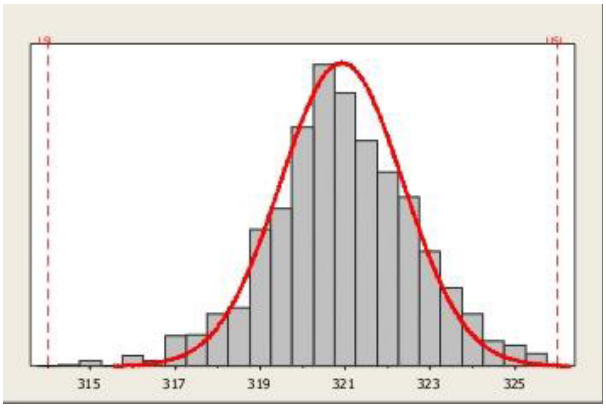


***Figure 1:*** *Histogram of TSP320450 i.d. data, N=2094*

## Conclusion

Continued efforts by Polymicro's production team have resulted in an overall improvement in both i.d. and o.d. variation, with i.d. standard deviations being reduced by as much as 20%. Polymicro continues to evaluate its manufacturing and metrology processes in an effort to ensure the tightest tolerances in the industry.

## References

(1) J. Macomber, P. Nico and G. Nelson, LCGC Applications Notebook, June 2004, p. 71

(2) J. Macomber and P. Nico, LCGC Applications Notebook, Feb. 2005, p. 55

*Originally Published in LCGC, The Application Notebook (February 2012)*

molex®
one company › a world of innovation