The Effect Of Using Low-Bleed Columns In The First Dimension For Comprehensive Two-Dimensional Gas Chromatographic Analyses

Jackson B. Webb[1], Katelynn Perrault Uptmor[1]
[1]Nontargeted Separations Laboratory, Chemistry Department, William & Mary, Williamsburg, VA

Background

In gas chromatography, the presence of column bleed impacts peak data quality. As siloxane-based stationary phase components fragment, they release bleed artifacts into the mass spectrometer (MS), producing interference and decreasing signal-to-noise ratios (S/N) of analytes.[1] When using comprehensive two-dimensional gas chromatography (GC×GC), a secondary column with an orthogonal stationary phase is used in providing improved separation between column bleed and analytes. However, it also introduces more stationary phase that can be degraded to produce more bleed artifacts.

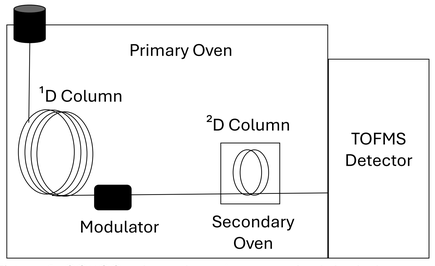

Figure 1. GC×GC schematic using a quad-jet dual-stage cryogenic modulator and TOFMS detector.

The goal of this research was to define how replacing the primary column in a GC×GC-MS system impacts the resulting S/N and analyte identification quality, in order to clarify how column bleed impacts analytes that appear to be chromatographically resolved from column artifacts in GC×GC output. This will demonstrate the advantage that low-bleed columns can provide for future analyses.

Results and Discussion

Two samples are represented in the contour plots of Figure 2, a sample of a 52-compound Indoor Air Standard and a Century Mix Standard that consisted of ~100 compounds.[2]

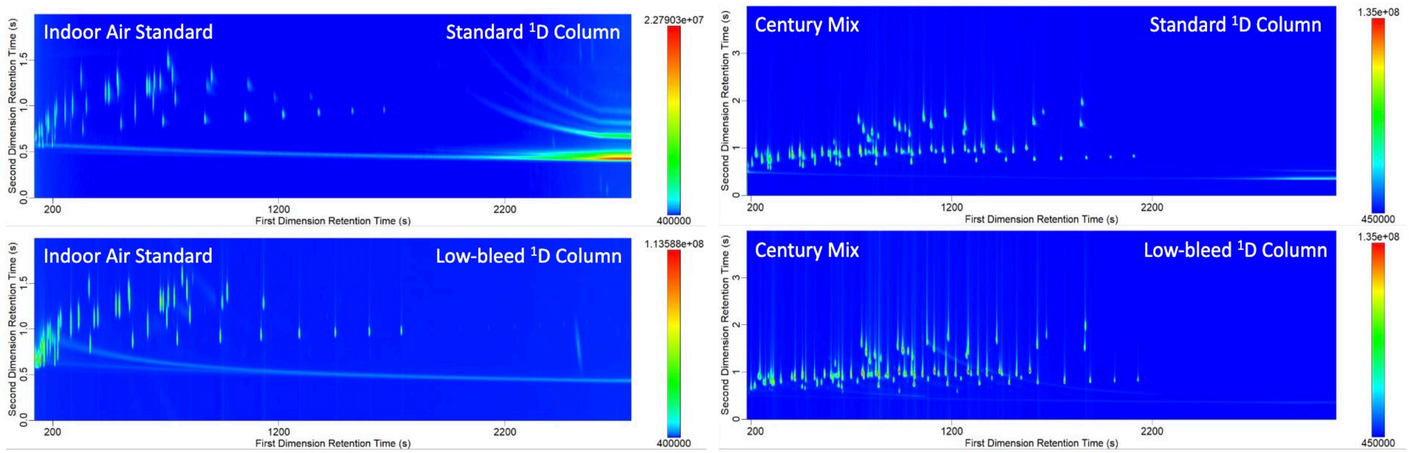

Figure 2. Total ion current contour plots of an Indoor Air and Century Mix samples run on the standard first dimension column and the low-bleed first dimension columns, respectively.

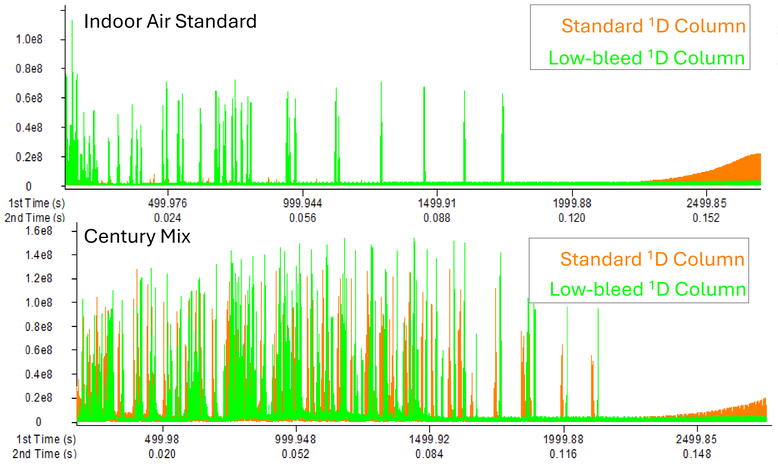

Figure 3. Total ion current chromatograms (1D representation of 2D data) for the Indoor Air and Century Mix samples run on the standard second dimension column and the low-bleed second dimension columns, respectively.

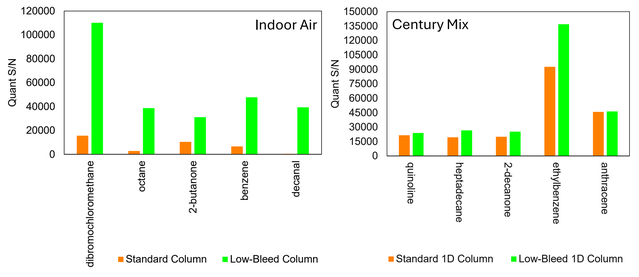

Figure 4. Bar graph of the Indoor Air and Century Mix samples showing the quantitative S/N.

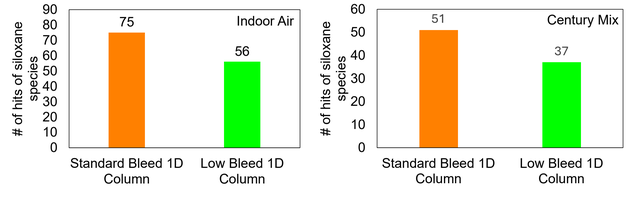

Figure 5. Bar graph of the Indoor Air and Century Mix samples showing siloxane hits identified.

Methods

Samples analyzed were the Century Mix[2], containing approximately 100 different analytes within different classes (i.e. Century Mix[2]) and a 52-component Indoor Air Standard. Both were assessed using a 5% phenyl/95% dimethylsiloxane standard column in the first dimension and a (50%-phenyl)-methylpolysiloxane standard column in the second dimension. Samples were then re-analyzed with a low-bleed column replacement in the first dimension.

Analysis of the samples was performed using the Pegasus BT4D GC×GC-TOFMS with a quad-jet dual-stage cryogenic modulator (LECO Corporation). ChromaTOF software (LECO Corporation) was used to analyze the chromatographic data.

GC×GC Parameters	Century Mix	Indoor Air
Carrier Gas	1.4 mL/min, Helium	1.0 mL/min, Helum
Inlet Conditions	200 °C, Split ratio 10	250 °C, Split ratio 20
Modulation Period	4.0 s (1.2 s hot pulse, 0.8 s cold pulse)	2.0 s (0.6 hot pulse, 0.4 cold pulse)
Oven Program	40 °C (2 min), increase 5 °C/min to 270 °C, hold 4 min	40 °C (2 min), increase 5 °C/min to 250 °C, hold 2 min
Secondary Oven Offset	+15 °C	+5 °C
Modulator Offset	+15 °C	+15 °C

TOFMS Parameters	Century Mix	Indoor Air
Ion Source Temperature	250 °C	270 °C
Transfer Line Temperature	250 °C	270 °C
Mass Range	35 – 450 *m/z*	35 – 500 *m/z*
Acquisition Rate	200 spectra/s	200 spectra/s

Acknowledgements

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Conclusions

This work demonstrated that samples run with the low-bleed first dimension column showed a reduction in the presence of column bleed components identified and maintained or improved the quantitative signal-to-noise ratios of components compared to the samples run on standard first dimension columns. This showed that that low concentration analytes were more reliably detected in the absence of column bleed, even when contour plots showed physically resolved components. In the future, this can be applied to improve the resolution of complex samples and increase analyte signal.

References

(1) Reese, A.; Vickers, A.; George, C. GC Column Bleed: A Mass PerSPECtive, Agilent Technologies, publication number B-0442, 2001.

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(3) Taylor, T. *GC Column Killers!* The LCGC Blog. Mar 6, 2023. Accessed on October 1st, 2025. https://www.chromatographyonline.com/view/gc-column-killers-.