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Tech Tip

GC Ferrules – Graphite and Polymeric Ferrules

This month we continue last month's discussion of ferrules used in gas chromatography. We discuss ferrules of graphite, Vespel® (polyimide), Vespel®-graphite mixtures, and Teflon® (polytetrafluoroethlyene, PTFE). Attributes of these ferrule materials are summarized in Table 1.
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Featured Applications

BenchTOF-dx and TargetView – high-de?nition, versatile technologies for the identi?cation of TO-15/ TO-17 'air toxics'

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TCA and Precursors in Red Wine Using In-Matrix Derivatization Followed by SPME on the SLB-5ms

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Matthew Klee

All About GC Ferrules – Part 2, Graphite and Polymeric Ferrules

This month we continue last month's discussion of ferrules used in gas chromatography.

Ferrules of graphite, Vespel® (polyimide), Vespel®-graphite mixtures, and Teflon® (polytetrafluoroethlyene, PTFE) will be discussed. Even though graphite is not a polymer, its characteristics are more similar to the synthetic polymers than metal ferrules discussed last month, so it is included. Attributes of these ferrule materials are summarized in Table 1.

Graphite and polymeric ferrules have several advantages for use in gas chromatography over metal (especially hard metal) ferrules:

- They can seal against imperfect surfaces with little force
- They can be used with virtually any type of tubing or column including glass and fused silica
- They can also be hand drilled with a pin vise to get the right size for any given tube or column

However, because they are organic and porous, graphite and polymeric ferrules do have some general weaknesses that constrain their uses as well:

- Polymers have a limited temperature range (compared to metal ferrules)
- They are more permeable to air infiltration (a function of polymer density)
- They sometimes come out of their commercial packaging, or the lab drawer, contaminated. They are then a source of ghost peaks and baseline disturbances. Contamination comes from poor manufacturing processes as well as poor choices in packaging (the contamination comes from the packaging).
- They can interact with sample components or solvent causing tailing or losses, especially at trace levels

Graphite has been a favorable ferrule material for capillary column use from the beginning of gas chromatography. It is very forgiving because it is so soft and can deform and seal in almost any space. Graphite is easy to identify because it can be deformed by pinching with your fingers, or scratched easily with your fingernail. I have successfully used the wrong size ferrule (because I ran out of the right size) and was able to either easily expand the hole to accommodate a larger column or to compress a larger ferrule just by putting it on the column and tightening the fitting a little more than usual. The graphite reformed in both cases to create

Figure 1

Figure 1: Examples of graphite and polymeric ferrules.

a seal. However, this malleability can lead to one of the biggest problems with graphite ferrules as well. This ability of graphite to reform can cause it to extrude through openings in fixtures into the neighboring spaces. Then, graphite pieces end up contaminating areas like the bottom of the inlet or the detector jet. These graphite pieces can interact with sample causing losses, tailing, and can become a constant source of contamination (graphite acts as a "chemical sponge").

A second major problem with 100% graphite ferrules is that they are very permeable to air. So, when using air sensitive columns (e.g., carbowax) or detectors (e.g., mass spectrometers or ECDs) one should choose something else.

Some inlet and detector designs use graphite ferrules wherein the graphite is contained within a secondary metal tube. This provides the benefits of graphite ferrule material, while greatly addressing the issues of deformation, extrusion, and infiltration of air.

Polyimide is a polymer with high temperature stability and low outgassing. Because of its relatively high temperature stability, polyimide is the coating of choice for the outside of fused silica columns. Polyimide ferrules are easy to identify because they are brown (see Figure 1). Polyimide is fairly rigid and can be molded into shapes that match fixtures, fittings, and devices typically designed for metal ferrules. Even though it has high temperature stability, polyimide is very sticky when heated, so it is not a good choice in high-heat zones. It can seize on the tube and in worst-case scenarios, it sticks inside the fitting. For this reason, 100% polyimide ferrules find most use outside the GC oven. I prefer them over metal ferrules for room temperature or low temperature connections since they are much more forgiving than metal ferrules when sealing against flawed tubing, they can be removed without having to be cut off the tubing, and they have low air permeability.

The stickiness of polyimide can be greatly reduced by adding a small amount of graphite (15% is common). The mixture retains the benefits of:

- Being able to be molded into desired shapes
- High temperature stability

Table 1

Ferrule Type	Temperature Limit	Typical Uses	Advantages	Limitations
Graphite (100%)	450 °C	General purpose for capillary column connections to inlets and detectors	Easy to use, can be tightened to hold column in place without siezing, column can be readjusted	Allows air diffusion. Not for MS or oxygen-sensitive detectors
		Recommended for high temperature and cool on-column applications	Can be removed easily	Overtightening can extrude graphite into inlet or detector. Pieces flake off and stay behind, causing peak tailing and sample losses
			High temperature limit	Soft, easily deformed or destroyed
			Most forgiving of receiver imperfections	Interacts with solvent, causing tailing
			Can be re-used	
Polyimide/Graphite (85%/15%)	350 °C	General purpose for capillary and packed glass columns	Easy reliable connections	Design specific to fitting
		Recommended for MS or oxygen-sensitive detectors	Reusable and remakeable	Shrinks with tempearture cycling. Must re-tighten frequently
			Maintains shape, does not flake off particles	Siezes on column, cannot be re-adusted, must be cut off
		Reliable, leak-free connection	Fairly forgiving of receiver imperfections	
Polyimide (100%)	280 °C	Isothermal operation	Easy reliable connections	
		Reliable seals even with flawed receiver surfaces, forms to sealing surface	Reusable and remakeable	Can shrink at elevated temperatures, must re-tighten frequently
		Excellent sealing material when making metal or glass connections	Can be removed easily if used at room temperature	Can glue connections together if exceeeding recommended temperature limit and destroy fitting
		Excellent for external	Low air permeability	
PTFE	< 260 °C	External connections not involving carrier gas	Easy connections	Allows air diffusion. Not for MS or oxygen-sensitive detectors
		Valve actuator gas lines	Can be re-used	Limited use inside oven
		Glass packed columns used at low temperatures	Very forgiving of receiver imperfections	
			Can seal with very little pressure.	

Table 1: Summary of attributes for graphite and polymeric GC ferrules.

- Ferrule does not extrude when tightening
- Can seal against imperfect surfaces
- Has low air permeability
- Soft enough for use with fused silica

Because of these benefits, graphite/polyimide ferrules are the dominant ferrule type when making fused silica column connections in the GC oven. graphite/polyimide ferrules do tend to shrink with temperature cycling, so they need to be retightened several times (after cool down) within the first 10 temperature programmed runs or so.

PTFE ferrules are very inert and have minimal interaction with samples and solvents. They are very easy to seal, often requiring only hand tightening. When disconnecting fittings, PTFE fittings slide right off the tubing and can be re-used multiple times. However, they have the most restricted upper temperature limit and are somewhat permeable to air, so use inside the GC oven is quite limited. PTFE ferrules are used primarily for low temperature applications requiring the most inert connections, and for tubing connections outside the oven when one wants reliable, easily adjustable and removable connections. PTFE fittings are often used for compressed air supply lines for valve actuators.

Dr Matthew S. Klee is internationally recognized for contributions to the theory and practice of gas chromatography. His experience in chemical, pharmaceutical and instrument companies spans over 30 years. During this time, Dr Klee's work has focused on elucidation and practical demonstration of the many processes involved with GC analysis, with the ultimate goal of improving the ease of use of GC systems, ruggedness of methods and overall quality of results.

Featured Applications

BenchTOF-dx and TargetView – high-definition, versatile technologies for the identi?cation of TO-15/TO-17 ‘air toxics’

Company: ALMSCO

This Application Note shows that ALMSCO’s BenchTOF-dx MS instrument and TargetView software are excellent tools for testing a complex semi-rural air sample for the presence of 62 ‘air toxics’ using US EPA methods TO-15 and TO-17. We demonstrate that, by combining the mass sensitivity of the BenchTOF-dx and the library-matching capability of TargetView, confident identification of target compounds can readily be achieved at ultra-trace levels. In addition, the versatility of these technologies is illustrated by the rapid identification of unknowns.

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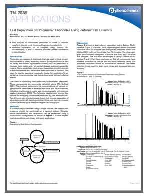

Fast Separation of Chlorinated Pesticides Using Zebron GC Columns

Company: Phenomenex

One class of commonly used pesticides is chlorinated pesticides. This application note illustrates how the fast analysis of chlorinated pesticides in under 10 minutes results in shorter cycle times and improved productivity. Baseline separation of all analytes using Zebron ZB-MultiResidue -1 and -2 columns provides high levels of confidence.

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Analyze More Semivolatiles Samples Per Shift Using Split Injection

Company: Restek

Semivolatiles are typically analyzed using splitless injection, but this technique results in slow analysis times and injection-to-injection variability. In contrast, using split injection under the conditions established here allows faster sample throughput and improved repeatability. Here we evaluate the applicability of split injection using higher oven start temperatures and faster cycle times in terms of sample throughput, sensitivity, and linearity for EPA Method 8270D.

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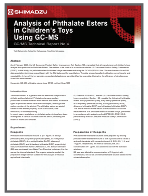
Versatile Automated Pyrolysis GC Combining a Filament Type Pyrolyzer with a Thermal Desorption Unit

Company: Gerstel

This paper describes an automated pyrolysis system for gas chromatography (GC) based on a fi lament type pyrolyzer combined with a commercially available thermal desorption instrument, onto which the pyrolysis module is installed. Automated sample introduction for both pyrolysis and thermal desorption is performed using a commercially available autosampler.



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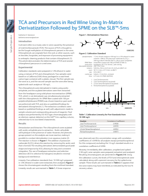
Fast Analysis of Acrylonitrile in ABS Polymer Using a Backflush GC System

Company: Shimadzu

Residual organic solvent in food packaging materials is a problem that receives serious attention because of the implications to food safety and public assurance. Acrylonitrile, which is controlled as a carcinogen by the U.S. Food and Drug Administration (FDA) and by regulations in the European Union (EU), is one specific compound of concern. This Application News introduces a fast analysis of acrylonitrile based on the use of the backflush GC system.



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TCA and Precursors in Red Wine Using In-Matrix Derivatization Followed by SPME on the SLB-5ms

Company: Sigma-Aldrich

Cork taint refers to a musty odor in wine caused by the presence of 2,4,6-trichloroanisole (TCA). The source of TCA is thought to be the fungal methylation of chlorophenols present in t he wine. Chlorophenols can originate from the cork or other sources, such as biocides, fungicides, and exposure of processing equipment to antiseptic cleaning products that contain chlorophenols. This article demonstrates the determination of TCA and several chlorophenol precursors in a red wine.

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