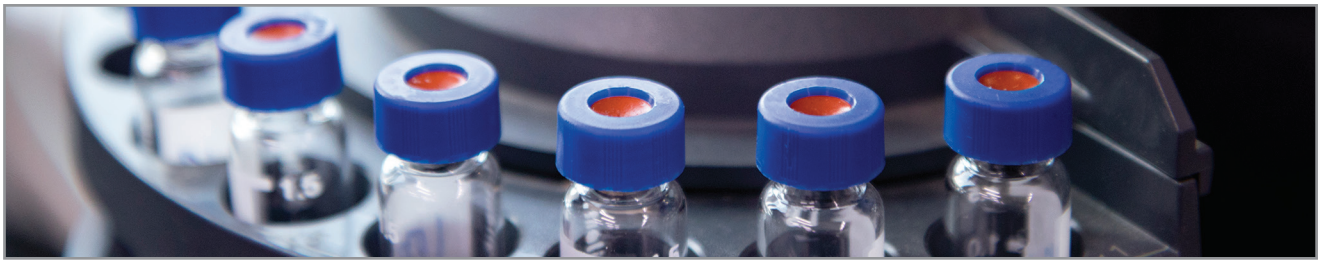


How to Diagnose GC Septum Bleed Contamination Sources:
Could it be Your Vial Cap?

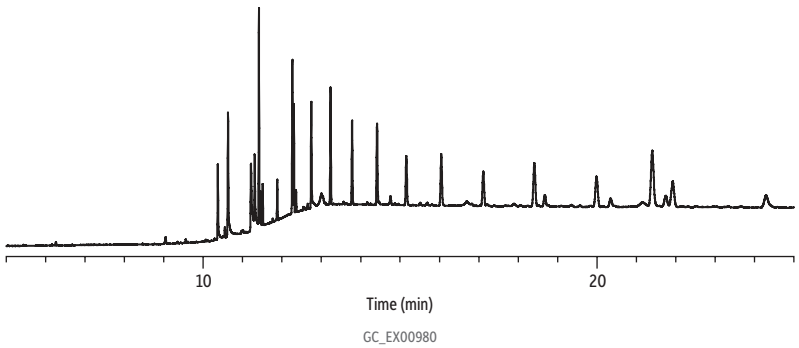
- Identifying vial cap septum bleed prevents lengthy inlet troubleshooting.
- Reduce interference with correct solvent-septum compatibility.

When septum bleed occurs, it is observed as sharp, repetitive peaks in high temperature portions of an analysis. Bleed peaks can come from either the inlet septum or the vial cap septum. Interfering peaks and inaccurate data can result, so it is important to correctly identify the source and to understand how to eliminate or minimize the bleed.

Diagnose GC Septum Bleed: What's the Source?

The bleed from either septum shows a similar pattern (Figure 1), but it is easy to determine the source with a simple test. Isolate the inlet by setting the instrument to perform a run without an injection. Perform an analysis; if the bleed disappears, then the vial cap septum was the source. Determining if the vial cap septum is the source of the bleed can save time by preventing unnecessary troubleshooting and maintenance. If the problem is in the inlet, review our technical article before replacing the septum. However, if the vial cap septum is causing the bleed, the issue can be eliminated or minimized with the following considerations.

Figure 1: The presence of sharp, repetitive peaks is a characteristic that helps identify GC septum bleed from either the vial cap or inlet.

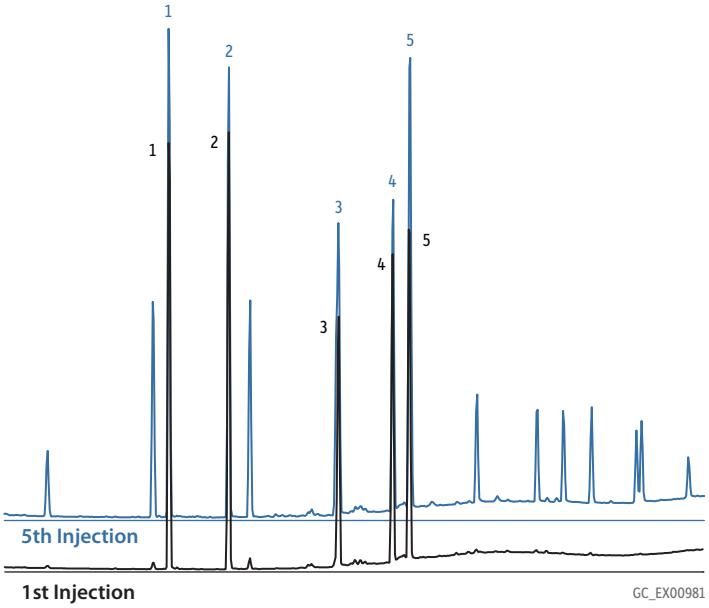


Column	Rtx-5MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 12623)
Sample	Methylene chloride blank
Injection	
Inj. Vol.:	1.0 µL split (split ratio 10:1)
Liner:	4.0 mm ID straight inlet liner w/wool (standard deactivation) (cat.# 20782)
Inj. Temp.:	240 °C
Oven	
Oven Temp.:	70 °C (hold 1 min) to 290 °C at 20 °C/min (hold 13 min)
Carrier Gas	He, constant flow
Flow Rate:	1.2 mL/min
Detector	FID @ 250 °C

Check Solvent-Septum Compatibility

Once you've diagnosed the GC septum bleed source as the vial cap, the first step in resolving the issue is to make sure that the vial cap septum material is compatible with your injection solvent. While septum bleed is not excessive most of the time, when a solvent and vial cap septum are incompatible, extreme contamination can occur. Figure 2 compares the first and fifth injections from a vial containing a derivatized amphetamine sample. In this case, the septum bleed peaks are almost as large as the analyte peaks. This level of bleed can interfere with analyses, especially those geared for trace levels. Reduce the risk of septum bleed by using a compatibility chart to determine which septum material is compatible with the sample solvent used.

Figure 2: Contamination from septum bleed can cause significant interference with target analytes.



Peaks
1. Amphetamine
2. Methamphetamine
3. MDA
4. MDMA
5. MDEA

Column	**Rtx-5MS, 30 m, 0.25 mm ID, 0.25 µm (cat.# 12623)**
Sample	**Amphetamine, methamphetamine, MDA, MDMA, and MDEA extracted from methanol and HFAA derivatized**
Conc.:	**100 µg/mL each**
Injection	
Inj. Vol.:	**1 µL splitless (hold 0.5 min)**
Liner:	**3.5 mm Custom splitless inlet liner w/ standard deactivated wool**
Inj. Temp.:	**220 °C**
Oven	
Oven Temp.:	**70 °C (hold 1 min) to 290 °C at 15 °C/min (hold 4 min)**
Carrier Gas	**He, constant flow**
Flow Rate:	**1.25 mL/min**
Detector	**MS**
Mode:	**Scan**
Transfer Line Temp.:	**280 °C**
Ionization Mode:	**EI**
Scan Range:	**43-450 amu**

Use Lined Septa

Most vial cap septa are lined with a protective layer of polytetrafluoroethylene (PTFE) to prevent solvent attack. As shown in Figure 3, PTFE effectively prevents septum breakdown due to solvent exposure. In comparison, unlined vial cap septa exhibit bleed after just 24 hours at room temperature. Bleed levels for unlined vial cap septa varied by material, but even a low level of bleed can interfere with integration and is of particular concern for trace analyses (Figure 4).

Figure 3: PTFE lining on vial cap septa prevents bleed due to solvent/septum interaction.

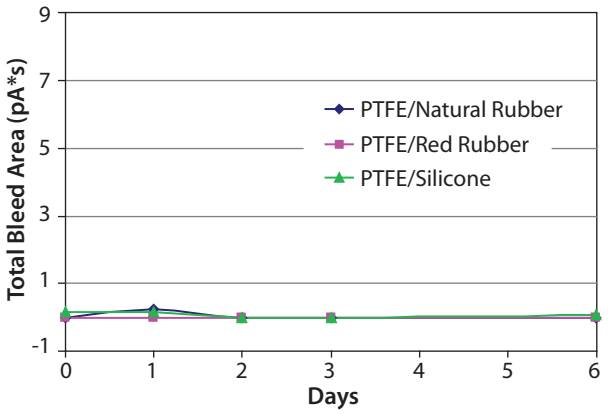
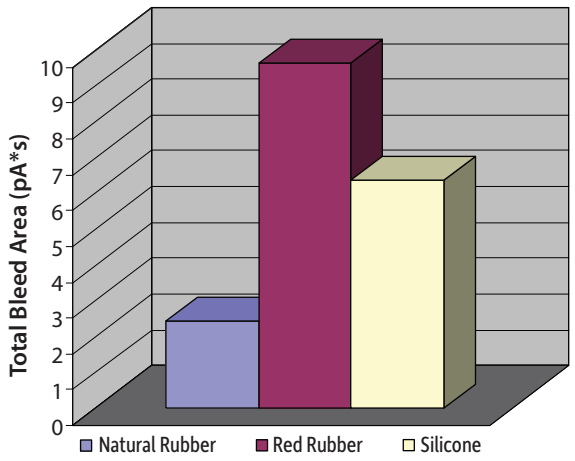


Figure 4: Unlined vial cap septa show bleed contamination within 24 hours.



Consider Resealability

Multiple injections can core the vial cap septum and lead to significant GC septum bleed. Resistance to coring varies by septum material (Figure 5). Coring can be minimized by preparing separate vials for replicate injections, when feasible, and by carefully considering the type of septum material when multiple injections are necessary. Septum resealability also affects evaporative loss, which can be a significant source of error for low volume samples. For example, a relatively nonvolatile analyte in a volatile solvent can concentrate significantly due to evaporative loss (Figure 6). Vials should be recapped when necessary for extended runs or long-term storage.

Figure 5: Bleed contamination increases over multiple injections.

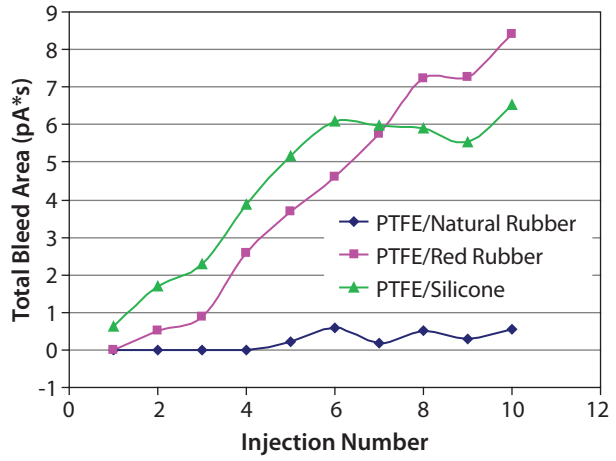
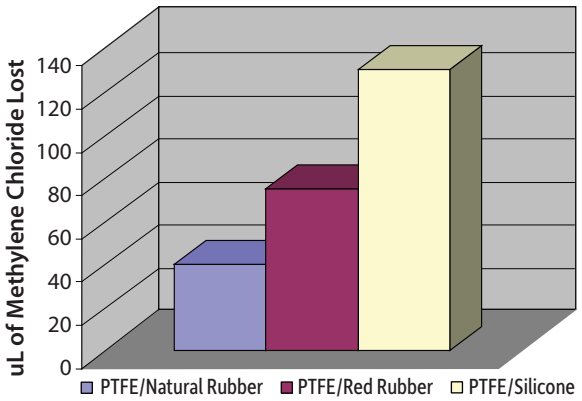


Figure 6: This bar graph shows the volume of solvent lost for three septum types. Vials containing 300 µL of methylene chloride were punctured and left at room temperature for 24 hours.



Conclusion

When septum bleed occurs, it is easy to assume the inlet septum is the source because the vial cap septum often is not considered. However, correctly identifying the GC septum bleed source can save time and effort by preventing unnecessary inlet maintenance. Effectively and efficiently reducing interfering peaks by controlling septum bleed can significantly improve analytical performance, particularly for trace analyses.

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