

High Temperature Headspace Vial Septa: The Difference between the Success and Failure of High Temperature Headspace-Gas Chromatography Analyses

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Introduction

Static headspace (HS) coupled with gas chromatography (GC) has been widely used for collecting quantitative and qualitative information on the volatile components that are present in complex sample matrices.

- Volatile components released into vapor phase by heating the sample.
- Agilent 7697 HS samplers extend temperature range of the sample being heated up to 300°C.
- High temperature helps release a wider range of volatile components from sample matrix, thus provide more qualitative and quantitative fingerprint information.

Current widely used PTFE/Silicone based headspace vial septa are not recommended to use for HS applications above 200°C.

- A series of siloxanes coming from the septa become observable and eventually become the predominant volatile species as baking temperature increased.
- These interference peaks significantly impact the reliability and efficiency of headspace analysis at high temperature.

Agilent new High Performance septa have been demonstrated to provide dramatically cleaner vial blank background with minimum septa contaminations observable.

- HS background at high temperature and pressure conditions were monitored and compared with market available PTFE/silicone septa.
- Septum chemical compatibility with common solvents was tested.

Experimental

GC/MS Instrument conditions

- Agilent 7890A GC/7697A HS sampler/5975C MSD
- S/SL inlet under split mode @ 250°C with 10:1 of split ratio
- Inlet liner: Direct liner 2 mm ID, deactivated (Agilent 5181-8818)
- He, constant flow @ 2.5 mL/min
- DB-5MS UI, 30m x 0.25 mm, 0.25 µm (Agilent p/n 122-5532UI)
- Oven profile: 40 °C (1.5 min), 15 °C/min to 325 °C (2.5min)
- MSD: Transfer line @ 250°C, Source @ 230°C, Quad @ 150°C

HS conditions

- Oven/Loop & valve/Transfer line temperature varied from 85 °C to 300 °C for different tests
- Vial equilibrate time: 30 min
- Vial pressurization gas: He
- Fill pressure: varied from 15 psi to 75 psi
- Final loop pressure: 10 psi
- Sample loop volume: 1 mL
- Vial size: 20 mL

Tested caps/septa

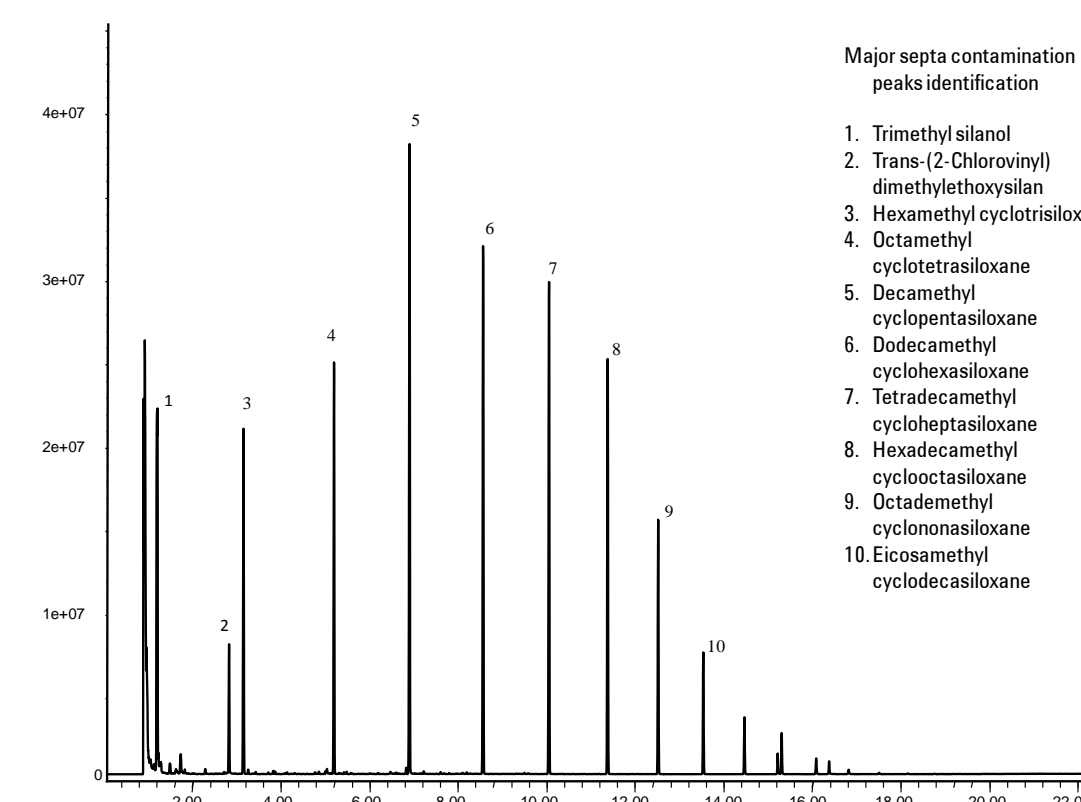
- High Performance septa w/ steel crimp caps (Agilent p/n 5190-3987)
- High Performance septa w/ steel screw caps (Agilent p/n 5190-3986)
- Market available HS caps w/ PTFE/Silicone septa



Results and Discussion

Agilent High Performance septa provided significantly cleaner HS background under high temperature sample equilibrium condition

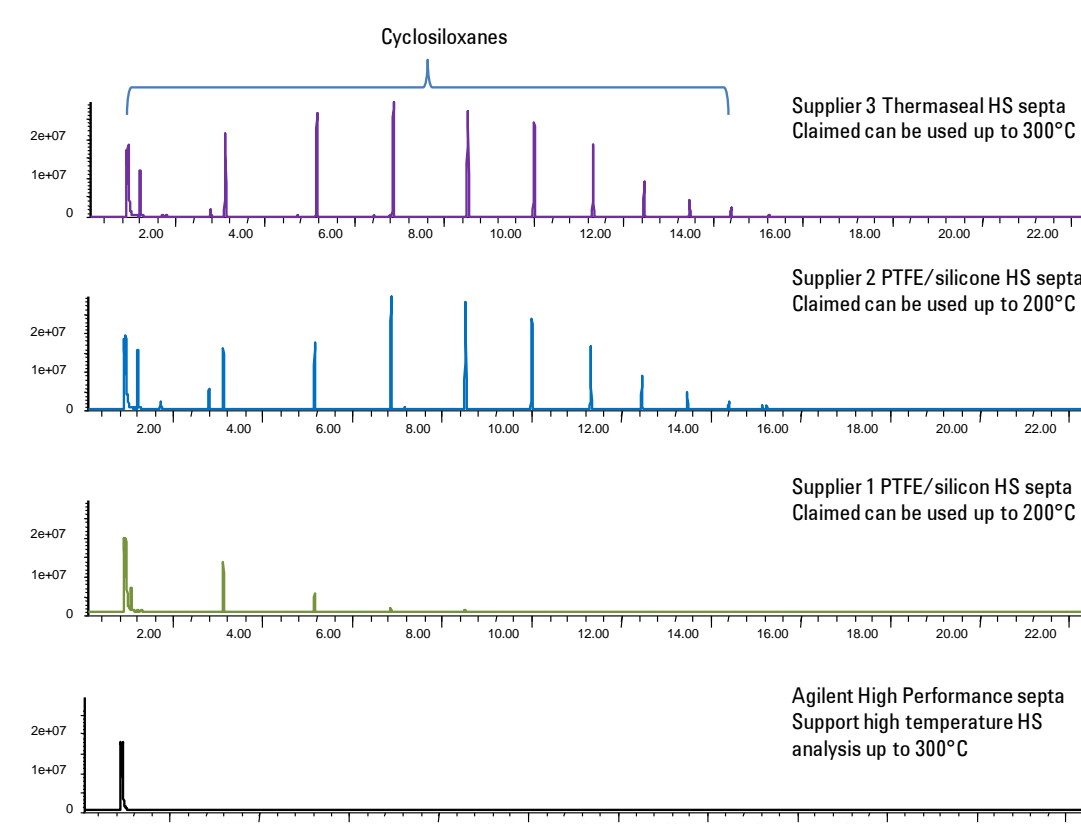
PTFE/Silicone HS vial septa cause significant contamination at high temperature



- Major septa contamination peaks identification
1. Trimethyl silanol
 2. Trans-(2-Chlorovinyl) dimethyllethoxysilan
 3. Hexamethyl cyclotrisiloxane
 4. Octamethyl cyclotetrasiloxane
 5. Decamethyl cyclopentasiloxane
 6. Dodecamethyl cyclohexasiloxane
 7. Tetradecamethyl cycloheptasiloxane
 8. Hexadecamethyl cyclooctasiloxane
 9. Octadecamethyl cyclononasiloxane
 10. Eicosamethyl cyclodecasiloxane

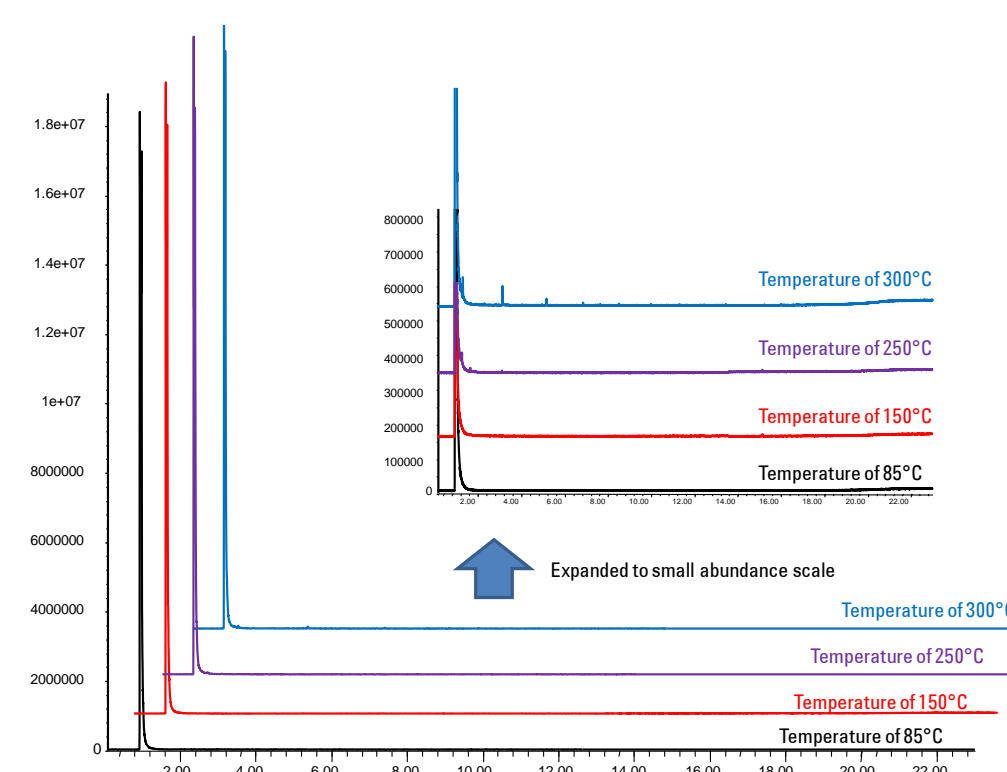
GC/MS chromatogram of vial blank with PTFE/silicone headspace septa heated at 300°C shows that PTFE/silicone septa cause significant septa contamination when sample being heated at high temperature.

Agilent High Performance septa provide dramatically cleaner HS background with minimal contamination at high temperature



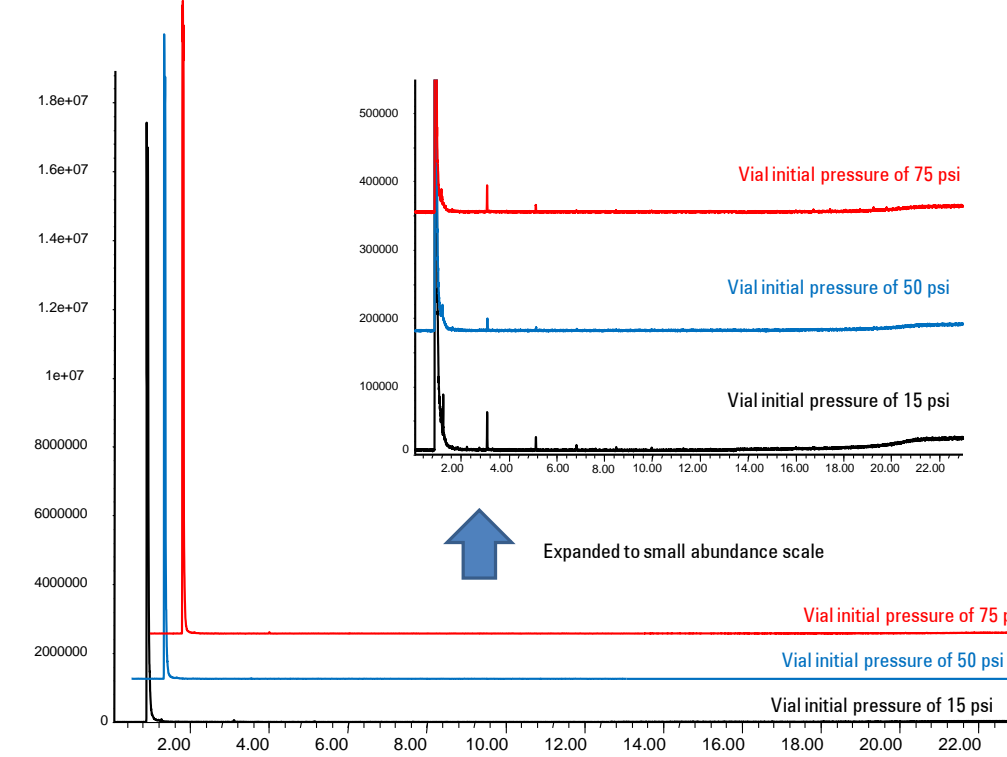
Vial blank samples were equilibrated at 300°C for 30 min. By using High Performance septa, the HS blank chromatogram clearly shows significantly cleaner HS background with minimal contamination.

Agilent High Performance septa provide consistent and cleaner HS background at various temperature



Vial blank samples were equilibrated at each temperature for 30 min and initially pressurized to 15 psi but then vented to 10 psi prior to injection. The chromatograms clearly show that the High Performance septa provide consistent and cleaner chromatographic backgrounds with minimal contamination across the entire temperature range examined.

Agilent High Performance septa provide consistent and cleaner HS background at various vial initial pressure



The pressure listed represents the initial pressure each vial was charged to prior to venting. All vials were vented to 10 psi before injection. Vials were equilibrated at 300°C for 30 min. By using High Performance septa, the vial blank chromatogram clearly shows chromatograms with minimal septa contamination from low to high vial initial pressure.

Results and Discussion

Agilent High Performance septa provided favorable chemical compatibility with common HS used solvents

Solvent compatibility test conditions

Solvent	Solvent blank with PTFE/silicone septum	Solvent blank with High Performance septum
Water	2e+07 1e+07	2e+07 1e+07
10% acetic acid in water	2e+07 1e+07	2e+07 1e+07
TEA	4e+07 2e+07	4e+07 2e+07
Toluene	4e+07 2e+07	4e+07 2e+07
DMF	4e+07 2e+07	4e+07 2e+07
DMAC	4e+07 2e+07	4e+07 2e+07
DMI	4e+07 2e+07	4e+07 2e+07

TEA: Triethylamine
DMF: N,N-dimethylformamide
DMSO: Dimethyl sulfoxide
DMAC: Dimethylacetamide
DMI: 1,3-dimethyl-2-imidazolidinone

GC/MS chromatograms of solvent blank with PTFE/silicone septa and High Performance septa were shown to be equivalent with minimal differences, indicating High Performance septa are compatible with common headspace solvents.

Conclusions

The qualification tests for the new High Performance septa on the 7697HS-GC/MS system demonstrated:

- High Performance septa can provide significantly cleaner HS chromatographic background at high temperatures (up to 300°C) compared to their PTFE/silicone counterparts. However, for high temperature applications, it is not recommended for longer than one hour sample equilibrium time.
- High Performance septa have displayed satisfactory chemical compatibility with common headspace solvents.
- Steel crimp/screw caps reinforce the seal to the vial and prevent leak during high temperature equilibrium.
- The High Performance septa fully support the feature functions of the Agilent 7697 HS sampler for high temperature applications.

To learn more about Agilent High Performance septa, visit us online at http://www.chem.agilent.com/en-US/Products/columns-supplies/generalchromatography/hp_septa