

Application Note 82

Low Bleed, High Temperature GC Septa Improve Baseline Stability

Volatile materials from the septum accumulate at the head of the column during the cooldown portion of a temperature program. When the column is reheated to resolve the next sample, these accumulated volatiles are eluted, producing peaks, a general baseline rise, or both. Thermogreen LB-2 silicone rubber septa exhibit low bleed, resist leaks, and are easy to penetrate. They maintain these characteristics after many punctures, even at inlet temperatures up to 350°C.

Key Words:

- temperature-programmed GC analyses • septum bleed

As you perform a series of temperature-programmed analyses, you may observe ghost peaks or a baseline rise that cannot be traced to the sample or to column bleed. These disturbances are often caused by septum bleed. Volatile materials from the septum accumulate at the head of the column during the cooldown portion of the program. When the column is reheated to resolve the next sample, these accumulated volatiles are eluted, producing peaks, a general baseline rise, or both.

The septa you select for your gas chromatographic analyses should exhibit low bleed, resist leaks, and be easy to penetrate. Thermogreen™ LB-2 silicone rubber septa, an improved version of our low bleed Thermogreen LB-1 septa, exhibit all these traits. Moreover, they maintain these characteristics after many punctures, even at high temperatures.

Thermogreen LB-2 septa offer additional advantages over many other septa, including a wider temperature range (100°C to 350°C) and a higher degree of batch-to-batch uniformity. They are also conditioned as part of the manufacturing process, eliminating the need for solvent extraction, heating, or other treatment prior to use.*

To demonstrate the low bleed levels of Thermogreen LB-2 septa, we compared the bleed from three unconditioned septa at an inlet temperature of 300°C (Figure A). After conditioning these septa overnight (16 hours), we retested the bleed at four times the original sensitivity (Figure A). The septum that initially exhibited the highest bleed continued to bleed at a relatively high level, even after conditioning. The Thermogreen LB-2 septum, however, showed little bleed before or after conditioning. The Pyrosep™ septum produced the least bleed, but we recommend using these hard septa only at inlet temperatures of 300°C to 400°C.

Equal portions of the septa to be evaluated (approximately 40mg) are carefully weighed and placed, sequentially, in an unpacked 3' x 4mm ID glass column, 3 inches from the outlet. The column

Figure A. Isothermally Accumulated Septum Bleed

Column: 3% SP™-2100 on 100/120 SUPELCOPORT™, 6' x 2mm ID glass (conditioned until bleed free)
 Oven: 70°C (8 min, to accumulate bleed) to 200°C at 16°C/min, hold 8 min
 Carrier: nitrogen, 20cc/min
 Det.: FID
 Inj.: 300°C

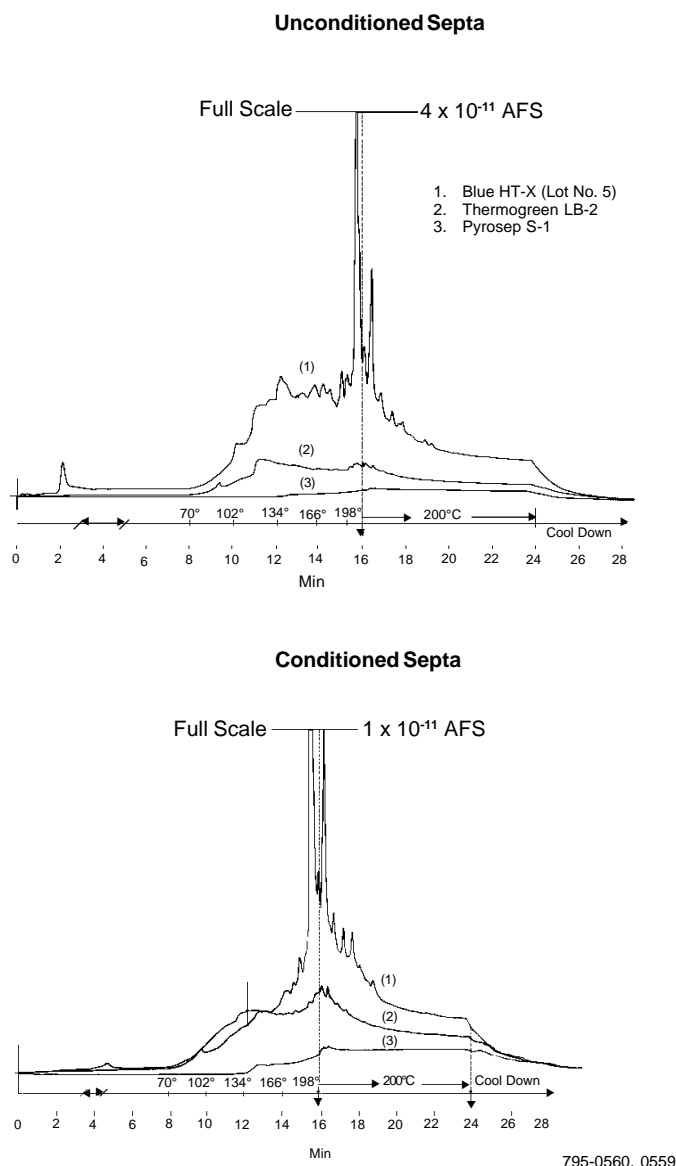
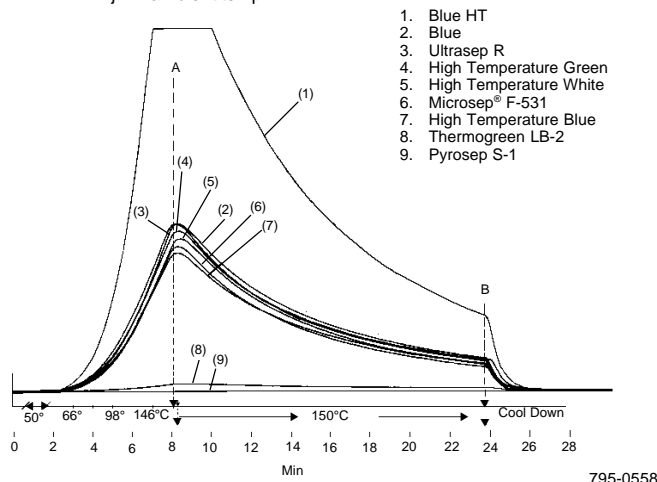


Figure B. Comparative Bleed Curves for High Temperature Septa

Column: empty 3' x 4mm ID glass containing 39 ± 0.2mg septum 3" from outlet
 Oven: 50°C (2 min, to accumulate bleed) to 150°C at 16°C/min, hold 16 min
 Carrier: nitrogen, 20cc/min
 Det.: FID (64 x 10⁻¹¹ AFS), 350°C
 Inj.: ambient temp.



Ordering Information: Thermogreen LB-2 Septa

Disc Diameter mm	Inch	Qty.	Cat. No.
5.0	3/16	50	20638
6.0	1/4	50	20651
9.5	3/8	50	20652
9.5	3/8	250	20666
9.5	3/8	1000	20677
10.0	13/32	50	20653-U
10.0	13/32	250	23156
10.0	13/32	1000	23157
11.0	7/16	50	20654
11.0	7/16	250	23163
11.0	7/16	1000	23164
11.5	11/24	50	23154
12.5	1/2	50	20660-U
12.5	1/2	250	20678
14.0	9/16	50	20662-U
16.0	5/8	50	20663
17.0	21/32	50	23159

Cylindrical, for Shimadzu Instruments

Plug Type	10	20608
Plug Type	50	20633

Drilled, for Solid Phase Microextraction

9.5	3/8	25	23161
9.5	3/8	50	23162-U
11.0	7/16	25	23167
11.0	7/16	50	23168

is then held at 50°C for 2 minutes, programmed to 150°C at 16°C/minute, and held for 16 minutes. (We use a 150°C temperature limit because we found that when the inlet temperature is 300°C, septum temperature is 150°C.) Under these conditions, the bleed from most septa rises rapidly as the column is programmed from 50°C to 150°C (Figure B), then gradually drops while the sample is held at 150°C and as it cools. The height of the curve at Point A can be used as a measure of rapidly purged septum volatiles. Point B indicates very high boiling components that slowly bleed from the septum. Samples of Thermogreen LB-2 septa exhibit minimum bleed at Point A and Point B.

In addition to exhibiting low bleed, Thermogreen LB-2 septa are easy to penetrate. However, to lengthen the leak-free life of the septum and to protect the needle, we strongly recommend using a needle guide with these or any other septa. A needle guide minimizes septum coring and tearing by ensuring that the needle consistently penetrates the septum at the same point. It also protects the needle by preventing it from buckling or hitting the edge of the column during injection (a damaged needle will cause excessive wear on the septum). Septum nuts with needle guides and other septum-related tools and apparatus are described in the Supelco catalog.

You can minimize septum bleed and leaks (and thereby reduce your cost per analysis) by choosing the proper septum. For long septum life and the lowest bleed among rubber septa at temperatures up to 350°C, we recommend Thermogreen LB-2 septa.

*We recommend a 9.5mm (3/8") septum to those who previously used the 9mm size.

†The amount of accumulated bleed depends on several factors: the type and brand of septum, the inlet temperature (bleed is greater at higher temperatures), the length of time the septum has been used (bleed gradually diminishes with septum use), and the duration of the cooldown period (long cooling periods increase bleed accumulation).

*Any new septum, including Thermogreen LB-2, should be installed at the end of the work day and conditioned overnight with carrier gas. This will minimize bleed the first time the new septum is used in a temperature-programmed analysis.

Trademarks

Pyrosep, SP, SUPELCOPORT, Thermogreen – Sigma-Aldrich Co.
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 for expert answers to your questions.

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