

CHOOSING THE RIGHT INLET LINER FOR YOUR APPLICATION

Part Two

– Increasing Sensitivity of Low Boiling Point Compounds

For Part One – Increasing Sensitivity of High Boiling Point Compounds, please refer to the last edition of “Solutions”. (Issue 1/2000)

Most people believe that to stop volatile discrimination, it is simply necessary to make sure the liner has a larger volume than the vaporized sample to stop the sample “overflowing” and entering the purge flow lines. This is generally a good rule but in some cases the opposite is true (as discussed later) and the “overflowing” concept doesn’t really apply to all the properties of gases.

When a liquid sample expands in a hot inlet liner it causes a quick increase in pressure in the area where the expansion first occurs. The pressure is then relieved by the sample flowing

into other areas of the injection port. In splitless mode, if a liquid solvent lands near the bottom of the liner it will expand out the top and even into the carrier gas inlet lines in order to balance out the pressure. If any volatile analytes vaporize at the same time as the solvent, they will also be swept into the inlet or purge lines, causing low boiling point discrimination. So, if 1 μ L of dichloromethane expands to 400 μ L, it doesn’t fill the liner up to the 400 μ L mark and stop. The vapor will fill as much of the volume inside the injection port as possible before it is swept out. **Figure 1** shows this effect.

The expansion of the gaseous sample through the top of the liner becomes more obvious when the carrier gas velocity is low in this area. In splitless mode it can be quite a problem when using low injection port flows and pressures. Also, if the sample vaporizes in the mid-point of the liner then it will have even less distance to travel to exit via the top of the liner, causing even more volatile losses.

There are two things that should be considered when choosing a new liner in order to limit low boiling point discrimination:

1. The position of the vaporizing sample.

Low boiling point discrimination often occurs in liners that contain glass wool, or some other object, which is placed in the middle of the liner. When the sample reaches this point, most of it is retained (depending on the liner’s design) and begins to vaporize. This makes it easy for the volatile components to escape via the top of the liner because they don’t have far to travel. Therefore, when attempting to avoid low boiling point discrimination the liner must not have anything placed in the middle to stop the sample from reaching the bottom. (An example of this is Part No. 092019 for Agilent Technologies GCs – see **Figure 3**)

2. The velocity of the carrier gas through the liner.

To stop the gaseous sample floating upwards the carrier gas velocity can be increased as it flows down through the liner. Increasing the carrier gas velocity can be achieved without increasing the pressure or flow. If the internal diameter of the liner or part of the liner is decreased, the velocity of the carrier gas will increase making it difficult for the volatiles to escape. So choose a liner with a taper at the top or a small internal diameter rather a large ID, non-tapered liner. (Suitable examples include the double-tapered liner Part No. 092018, and the 2mm ID goosenecked liner Part No. 092013, for Agilent Technologies GCs)

Figure 1. Expansion of sample in a hot inlet liner.

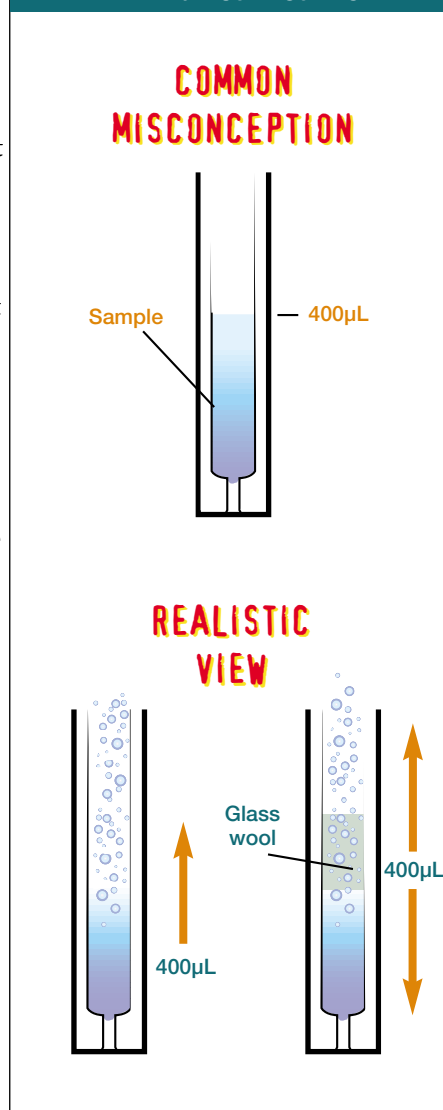


Figure 2. The difference in boiling point discrimination between two liners that give opposite results.

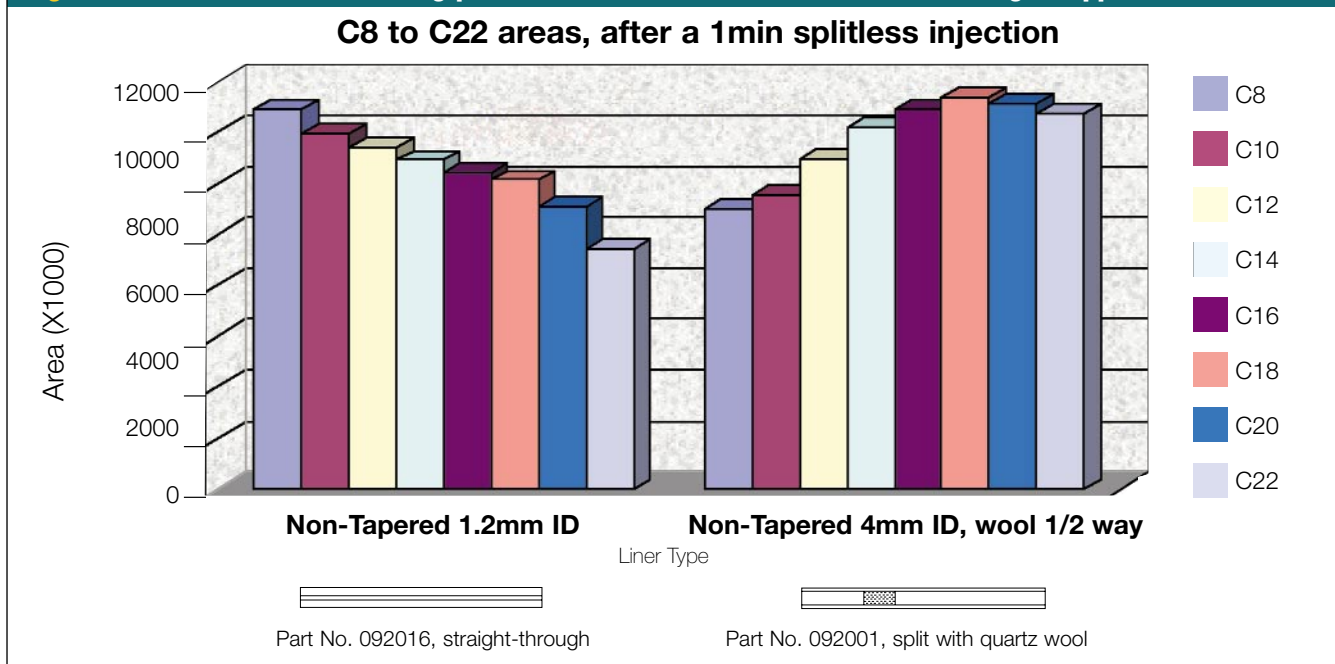


Figure 2 shows a 1.2mm ID liner compared to a larger 4mm ID liner with wool. The sample is C8 to C22 in pentane. The 1.2mm ID liner gives much higher sensitivity for low boiling point compounds even though it has less volume. This is because the sample vaporizes at the bottom of the injection port and the carrier gas velocity through the liner is higher than the 4mm ID liner.

The 4mm ID liner is the opposite, the sample vaporizes high in the liner and the velocity of the carrier gas through the liner is low. This increases the chance that low boiling point compounds will reach the top of the liner with the initial expansion of the solvent. So the boiling point discrimination exhibited by each liner is completely different.

The main problem with using narrow ID liners to limit low boiling point discrimination is they can be overloaded very easily. Even a 1µL injection can overload a 1.2mm ID liner if the wrong solvent is used. A more robust way of maximizing the sensitivity for low boiling point compounds is to use top tapered liners. The velocity of the carrier gas through a taper is much higher than in the body of the liner because the internal diameter of a taper is much smaller. This means the taper effectively acts as a lid that keeps volatiles in the liner.

The dual tapered liner is the best at increasing the sensitivity for low boiling point compounds (Figure 3), An upside down single tapered liner can be used obtaining suitable results for the low boiling point compounds but the heavier components in the sample will suffer. Small ID liners are better, but care must be taken when injecting volumes greater than 1µL.

Figure 2 also shows differences in the sensitivity of the high boiling point compounds (>C18). (This was explained in Part One of Choosing the Right Inlet Liner for Your Application, featured in "Solutions" issue 1/2000.)

Figure 3. Various liners to reduce low boiling point discrimination.

