

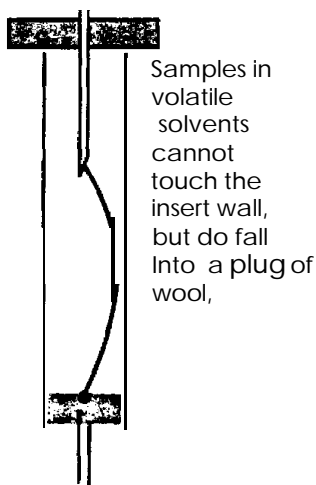


method (preheating the needle inside the injector before rapidly depressing the plunger). Nebulization in an empty liner provides gentle evaporation in the gas phase hardly involving any contacts with adsorptive and maybe dirty surfaces. Even high boiling, polar, and labile components are vaporized rather well.

### STOPPING SAMPLE LIQUID BY PACKING MATERIAL

Nebulization does not occur with fast injection auto-samplers. The sample liquid forms a thin band, like water running from the tap, and moves almost without resistance. It must, therefore, be stopped above the column entrance by other means, which is all but simple because of the Leidenfrost phenomenon.

Figure 3: Non-nebulized sample liquid must be stopped, e.g., by glass or quartz wool.



Heat consumption by evaporating liquid cools the source of the heat. If cooling is strong enough to reduce the surface temperature to the sample (solvent) boiling point, the liquid can contact the surface. This occurs with obstacles of a low thermal mass, such as glass or quartz wool. The liquid cools the nearest fibers it encounters and falls into the wool just as children jump into a haystack. Hanging in these fibers, the sample forms an island with a temperature corresponding to the solvent boiling point until the solvent is evaporated.

The smallest amount of wool which forms a short plug without major gaps (1-3 mg) serves the purpose. Additional amounts merely aggravate the problems-adsorption and degradation of labile compounds. There are two concepts for placing the packing-situated near the exit of the inserted needle, the packing will always receive the liquid and the solutes will always evaporate from its surface. This renders the process reproducible, but susceptible to the activity of the packing. Placed just above the column entrance, the packing rather serves as a safety net: nebulized samples will evaporate in the gas phase above the packing and pass the latter easily (adsorptive surfaces have less effect on passing vapors than on material evaporating from them). If the sample is only partially nebulized or not at all, the packing acts as a net underneath the acrobat in the circus. Packings of low thermal mass would be the most convincing solution to sample evaporation if they were inert.

Recently, Restek sent us some carbon material (Carbofrit) with the suggestion to test it as liner packing. Initially, I didn't even want to try it because carbon is usually highly retentive and catalytically active. As we nevertheless gave it a chance, we were highly surprised. It exhibited low retentive power and good inertness.

### LINERS WITH OBSTACLES

Injector liners containing solid obstacles, such as baffles or an inverted cup (Jennings cup), were conceived to enhance mixing the sample vapors with the carrier gas and stop "shooting" sample liquid. The inverted cup forces the gas flow to reverse directions twice, which seemed to guarantee that non-evaporated sample material would not pass. There was no solid proof, however, because it is difficult to derive from chromatograms what happened inside the injector. Recent visual experiments provided more direct evidence. Because of the Leidenfrost phenomenon, the sample liquid is able to curve around hot solid obstacles and change direction rather sharply. For instance, it performed perfect slalom around the baffles, hardly being slowed. When the obstacles stop the sample liquid it is for different reasons than what the originators thought. The main effects are due to the fact that liquids are hin-

dered to enter narrow channels (again, the Leidenfrost phenomenon). The inverted cup of the Hewlett-Packard liner usually stopped the sample liquid provided the sample volume did not exceed 1.5  $\mu$ l. The most effective liner was, however, the "laminar liner" from Restek.

### CONCLUSIONS

There are three principal concepts to achieve sample evaporation:

1. Sample evaporation in the gas phase of an empty liner provides the most gentle conditions, but presupposes partial evaporation inside the needle.
2. Well designed obstacles stop "shooting" sample liquid.
3. Packings with low thermal mass render vaporization most reliable, but evaporation occurs from a surface.

All three concepts may turn out best suited. You have to try.

1. *J High Resolut. Chromatogr* 15 (1992) 190  
 2. *J High Resolut. Chromatogr* 16 (1993) 429

