



evaporation are still the key techniques. However, a further improvement was important: the early vapor exit. When more than 50-100ul of solvent are introduced, discharge of the vapors through the whole column becomes slow and the flame detector turns into waste incinerator. A separate outlet is needed for the solvent vapors. The earlier this exit is positioned, the shorter the path is for the vapors and the faster is their release. On the other hand, the inlet must be long enough to retain the solutes, i.e. to achieve solvent/solute separation. The latter is achieved either by solvent trapping in the flooded zone (uncoated precolumn) or by the (less efficient) stationary phase trapping in the coated ("retaining") precolumn (see Figure 1). Partially concurrent evaporation provides solvent trapping and usually produces perfect peaks even for components

eluted immediately after the solvent. For fully concurrent evaporation, however, just stationary phase trapping is available (often reinforced by phase soaking), which restricts the analysis to solutes eluted several tens of degrees at least above the column temperature during solvent evaporation. With the early vapor exit, evaporation rates went up to typically 100-400 @/min. The new record for concurrent evaporation (from 1989) stands at 20,000 ul of a hexane solution introduced in 20 min.

**The future**

Presently the injection of volumes larger than 10ul is a subject at most meetings dealing with capillary GC. Two approaches are in the focus of the interest: Programmed Temperature Vaporizing (PTV) injection by the solvent split technique and large volume on-column injection. The

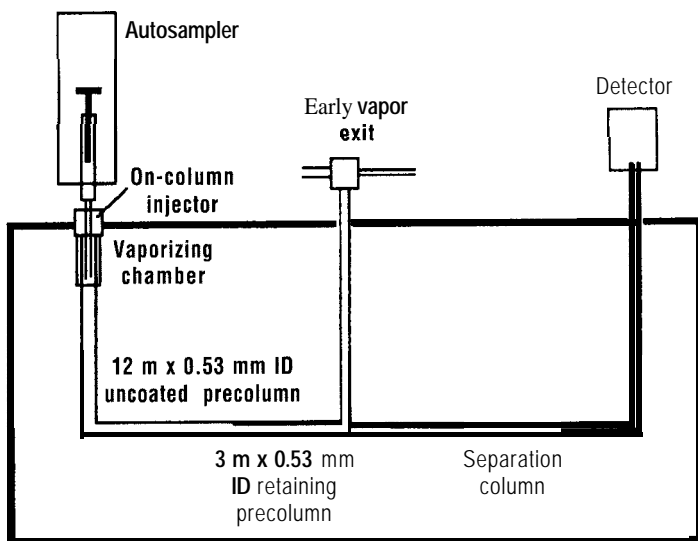
PTV technique is relatively robust regarding the injection of "dirty" samples", but the most volatile as well as the high boiling and labile components tend to be lost. The on-column technique avoids such losses and the results are highly quantitative, but the uncoated precolumn is sensitive to contamination by non-evaporating sample byproducts and to attack by aggressive components like water (humidity).

The future will show which technique wins, but the end of the development has not been reached yet. I believe that the on-column/retention gap technique provides the better basis and can still be improved. The first step has been made by the European leader in GC instrumentation, CE Instruments. Because the adjustment of appropriate conditions requires some understanding

of the background, a computer-guided instrument for sample volumes up to 250ul was designed. A standardized precolumn system ("Uncoret" - composed of an uncoated and a retaining precolumn in one piece, 15m x 0.53mm ID) is used and the software has evaporation rates for the most commonly solvents in its memory. It automatically adjusts the autosampler injection speed and closes the vapor exit at the appropriate moment for the analysis. In Europe, a good number of instruments are in use for trace analysis of fairly clean samples, and probably about half of them are in commercial laboratories doing water analysis.

The next step is the addition of a small bore, probably permanent, hot vaporizing chamber above the precolumn system (Figure 1) that serves as a filter for retaining "dirt" and for vaporizing non-wetting samples. This adds to the on-column system the robustness against "dirt" of the PTV but maintains the better and more reliable means for solvent/solute separation. In summary, GC is an excellent technique for trace analysis, but the small injection amounts ( typically 1-3ul) are as appropriate as wooden wheels on a sports car.

**Figure 1:** .....  
System for Large Volume Injection.



Dr. Konrad Grob can be reached by E-Mail at