Why Uncoated Capillary Precolumns Enable Injection of Large Volumes

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This issue of Koni's Korner deals with uncoated capillary precolumns or desolvation precolumns. The development of the retention gap technique for introduction of large volumes of sample was an exciting experience of which I would like to give a summary here. Uncoated precolumns are used for two totally different purposes: for on-column injection of large volumes and as a garbage bin (guard column, disposable inlet) for the analysis of samples with non-evaporating by-products ("dirty" samples).

The "Retention Gap"

During the first two years of using on-column injection, we were puzzled by occasional splitting of peaks that eluted at several ten degrees above the oven temperature during injection. In 1981, using glass capillaries, we saw how the injected sample liquid moved rapidly along the capillary wall and deeper into the column. In a 0.32 mm ID column, 2ul easily "flooded" 50 cm of the column inlet. Even worse, sample liquids not wetting the stationary phase (e.g. solutions in methanol on apolar silicones) just left a droplet here and there (as water on a window pane) and entered the column up to several meters for every microliter injected. It was obvious this would not produce the sharp initial bands required. Flooded zones of 20-40 cm seemed to be the maximum for avoiding noticeable peak broadening. A paper by W.L. Saxton (HRC 1984) confirmed this conclusion. This enables injections up to 1-2ul of a wetting sample.

During these experiments, we were puzzled by certain columns that did not produce broad or split peaks even when we injected 5ul It took some time and several cups of coffee to discover these were the columns which we had prepared with 0.5-lm of uncoated inlet. We realized that straightening the end sections of the columns,

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