Coping With the "Christmas Tree" Effect with Fused Silica Columns in Low Thermal Mass Cages

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1 Introduction

Peak broadening, distortion, and splitting caused by solvent flooding at the inlet to capillary columns have been reported in the literature [1,2]. However, perturbations of peak shape generated at the detector limb are less well documented. Recently, we found that fused silica columns are particularly susceptible to thermal gradients in GC ovens. This is because of their thin wall cross-sections and correspondingly low thermal masses. Rapidly responding to changes in oven temperature, the columns may undergo thermal cycling as the oven heaters are pulsed to regulate isothermal or temperature-programmed conditions. In the last few centimeters of the detector limb, the pulsation, undamped by later stationary phase absorption, produces a jagged edged peak, sometimes referred to as the "Christmas tree" effect.

The effect is especially observable in instruments equipped with fast electrometers and recorders with low time constants. The Carlo-Erba 4160 high resolution gas chromatograph is particularly prone to this problem. Although improved air flow has reportedly eliminated the effect in the newer Magnum series instruments, owners of the older 4160 model must cope with various solutions to the problem, including wrapping the column cage with aluminum foil, installing a special sleeve-washer on the detector limb, or using a high thermal mass column cage. In all of these "fixes" it is recommended that the detector limbs be kept short to help minimize the severity of the effect.

2 Experimental and Results

We found the recommended procedures awkward and defeating the advantages of handling fused silica columns (ease of installation). Since the critical problem is the thermal mass of the last few centimeters of column, an alternative procedure for increasing thermal mass in this region was sought. In place of the sleeve-washer assembly provided by Carlo-Erba, we used a sleeve of braided wire, approximately 0.156 cm o.d. and about 30 cm in length. The sleeve is readily flared at one end to ease slipping it over the column. When installing, the sleeve is readily pushed down the column and out of the way. Once installed, the metal sleeve is pushed up to the detector nut and held in place by a length of aluminum foil or wire.

This remedy for the Christmas tree effect does not restrict the length of the detector limb and does not interfere with the installation of the column. Its efficacy is demonstrated in the chromatograms presented in **Figure 1**. Using the braided wire sleeve, the Christmas tree effect is eliminated. Although many sources of braided wire may be conceived, we obtained ours from a length of common no. 40 gauge shielded cable. Oxidation of the tinning of the shielding gives the sleeve a dull grey appearance after operating the oven at high temperatures, but this does not affect performance.

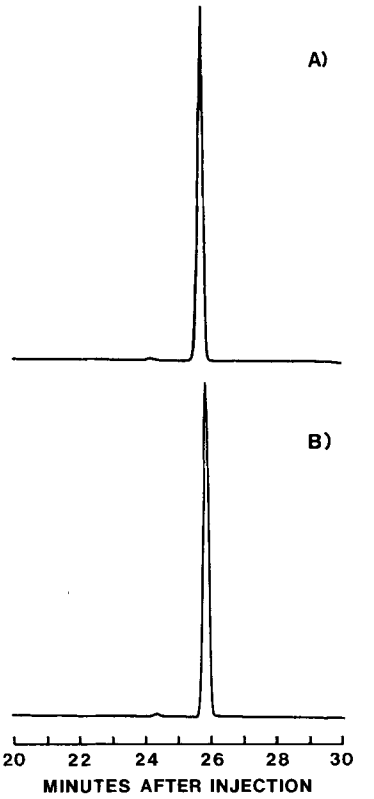

Figure 1

Elimination of the Christmas tree effect by a braided wire sleeve: A) no metal sleeve; B) sleeve installed. Column is DB-1, 30 m × 0.25 mm i.d., 0.25 μm film thickness from J & W Scientific. Sample is 0.2 μL *n*-pentadecane in hexane, approximately 500 ng/μL. The injection was split 1:50 with the oven isothermal at 120°C. Only the *n*-pentadecane peak is shown in order to clearly demonstrate the improvement.

References

[1] *K. Grob, Jr.*, J. Chromatogr. **213** (1981) 3-14.

[2] *C. A. Saravalle, F. Munari,* and *S. Trestianu*, J. Chromatogr. **279** (1983) 241-250.

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