# Loss of Flavor Components in Glc Columns and a Test for its Occurrence

Important components of food flavor volatiles may be lost during glc. Such losses have been noted, for example, when Cheddar cheese volatiles are chromatographed. Some columns markedly changed the odor of the chromatographed volatiles while others caused only minor changes. Loss of critical components may account for failures to reconstruct food flavors from mixtures of identified compounds. A method is described for recovering the sample from the glc effluent for comparison of its odor with that of the unchromatographed sample. It is strongly recommended that such a comparison should be made with volatiles of the specific food to establish the suitability of the glc columns to be used.

In this note we wish to make three points: 1. During chromatography of food flavor volatiles, essential components of the flavor may be adsorbed or decomposed by the glc column; 2. It should be determined whether all of the components essential to the flavor emerge from the column during the glc analysis; 3. Such a determination may be made by trapping the total effluent and comparing the aroma of the condensate with that of the unchromatographed sample.

The importance of the first two points will be evident from the following observations, indicating adsorption of key components of Cheddar cheese volatiles by some glc columns.

When Cheddar volatiles were chromatographed on several different columns, the condensible material recovered from the total effluent had an aroma with little similarity to the characteristic cheese-like aroma of the sample of volatiles before chromatography. The columns exhibiting this phenomenon included columns packed with Carbowax 20M on Chromosorb W-HP, uncoated Porapak Q, and FFAP on Porapak T. The first two are specifically mentioned because they are frequently used in the analysis of flavor volatiles. The FFAP column has been recommended for the analysis of free fatty acids, and free fatty acids are recognized to be important contributors to Cheddar flavor.

The aromas of Cheddar volatiles recovered from two other columns were indistinguishable or just detectably different from the aromas of the unchromatographed volatiles. The columns were stainless steel, 183 cm × 2 mm i.d., packed with 10% OV 225 on 80–100 mesh Chromosorb W-HP, and 183 cm × 5 mm i.d., packed with 10% OV 101 + 0.5% Igepal CO 880 on 80–100 mesh Chromosorb W-HP.

In the case of Cheddar volatiles the choice of column is critical, since there is little hope of establishing the essential components of the flavor if some of those components are being removed or destroyed by the column. The selection of columns may be equally critical for the analyses of other food flavors.

Guadagni (1968) has pointed out the precautions that should be observed when using glc for flavor analysis, but since his paper was published there have been a number of papers in the literature in which failure to reproduce a food aroma with mixtures of identified components is ascribed to lack of quantitative data or failure to identify observed trace components. Despite the considerable information in the literature on the adsorption or decomposition of chromatographed components, the authors appear to have given no consideration to the possibility that key components were adsorbed by their columns. Guadagni's recommendations may have been disregarded because no specific example was cited to illustrate that essential flavor components can be lost during glc.

A simple way to confirm that all of the components essential to the flavor are emerging from the column is to recover the sample from the total glc effluent and confirm that its aroma is not significantly different from that of the sample prior to chromatography. The trapping system used to collect the glc effluent need not be elaborate; the principal requirement is that all of the flavor components eluted from the column, including trace components, are completely collected. We have found that a glass U-tube (100 $cm^3$; 16 mm i.d.) with the outlet closed by a "helium-type" balloon met this requirement. When the U-tube was cooled with liquid nitrogen, little or no odorous material passed through to the balloon. The very slight "rubbery" odor of the balloon caused no obvious interference with the aroma of Cheddar volatiles.

When making the odor comparison, care should be taken that the dilutions of the original and the recovered samples are the same. To arrive at a dilution equal to that of the recovered sample, we injected a sample of the unchromatographed volatiles, equal in volume to the sample subjected to glc into a trap identical to the effluent trap and similarly fitted with a balloon.

The amount of work involved in performing this test with enough replicates and panelists to permit a rigorous statistical treatment of the data will tend to be discouraging. However, a few trials with two or three experienced panelists may provide sufficiently convincing evidence to reject columns which markedly alter the sample aroma.

Further details will be provided on request.

LITERATURE CITED

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