Improvement of the Direct GC Analysis of Vegetable Oils' Volatile Profile by a Poly MPE-Tenax GC Column

JOSEPH L. WILLIAMS and **JEAN H. WILLE**, Kraftco Corporation R & D, Glenview, Illinois 60025

ABSTRACT

A Poly MPE-Tenax column has been successfully incorporated in Dupuy's method of direct GC analysis of volatiles in vegetable oils. This column provided a considerable reduction in column bleed, speed of analysis, and improved adsorptivity of volatiles, quantitation, and consistency of integrator counts.

INTRODUCTION

Dupuy et al. (1) in 1971 introduced a rapid, single, and extremely sensitive GC technique for obtaining and examining the GC flavor profile of vegetable oils. Subsequent work (2) and collaborative studies (private communication, Kraftco Corporation R&D and Southern Regional Research Lab, 1973-75) have shown that this method is a valuable tool in assessing the quality of vegetable oils, following the flavor deterioration during shelf life studies, and complementing flavor panel scores.

Routine in-house studies of vegetable oils' volatile profiles and collaborative testing of oils produced good agreement between laboratories (private communication, Kraftco Corporation R&D and Southern Regional Research Lab, 1973-75). However, in-house studies revealed that the use of Porapak columns detracted from the method as follows:

1. The use of temperature programmed Porapak P dual columns made the system difficult to balance.
2. The Porapak temperature limit of 230 C was a critical factor, since desorption by heat was limited to 200 C (3) and five peaks in the volatile profile eluted above 200 C.
3. The bleed-rate of Porapak began to produce a background at 145 C which steadily increased and caused the baseline to drift upward by 10-40%.

The consequences of the outlined problems above in the quantitative analysis of the volatile profile were the following:

1. Time was added to each analysis and necessitated that a person watch the instrument for an hour.
2. Peaks eluting at 200 C were skewed and hard to quantify. Components tended to build up on the column, and in time the flame ionization detector would become noisy.
3. The upward drifting of the baseline after 145 C mandated using two modes of automatic peak area integration. Up to 145 C, the perpendicular mode was used, and afterwards the manual tangent mode was used. These mixed modes of peak integration caused wide variations in counts even though the pen presentations of the volatile profiles of identical oils were similar.

EXPERIMENTAL PROCEDURES

Column Preparation

A 9 ft 10% Poly MPE-Tenax column was made by the method of Novotny et al. (4). This column was conditioned at 300 C over the weekend and applied to the direct GC analysis of volatiles in vegetable oils.

Concentration and Analysis of Volatiles

Gas chromatographic procedures were identical to those described by Dupuy et al. (2). An Autolab System IV was used to obtain peak areas.

RESULTS AND DISCUSSION

Comparative oil volatile profiles analyzed by Poly MPE-Tenax and Porapak P columns were given in Figures 1 and 2. The Poly MPE-Tenax presentation of the baseline is clearly superior to the Porapak P column. In addition, the consistency of counts on the same oil throughout the volatile profile improved, and the last five peaks in the

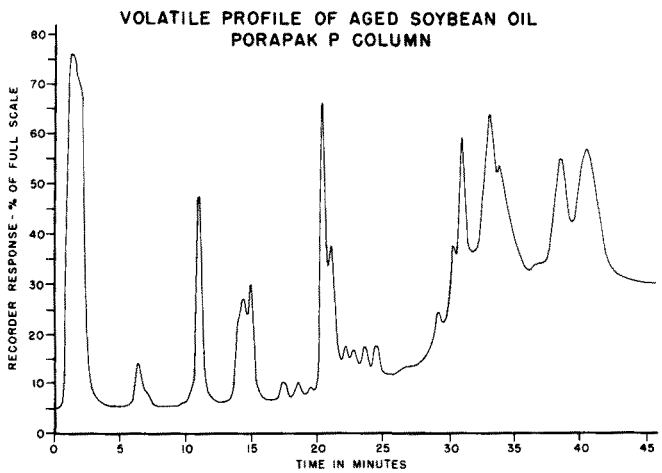

FIG. 1. Porapak P GC chromatogram illustrating the resolution of volatile components of soybean oil aged 5 weeks in the light of 22 C. Conditions are as described by Dupuy (2). An injection port temperature of 170 C was maintained while purging the volatiles from the glass liner.

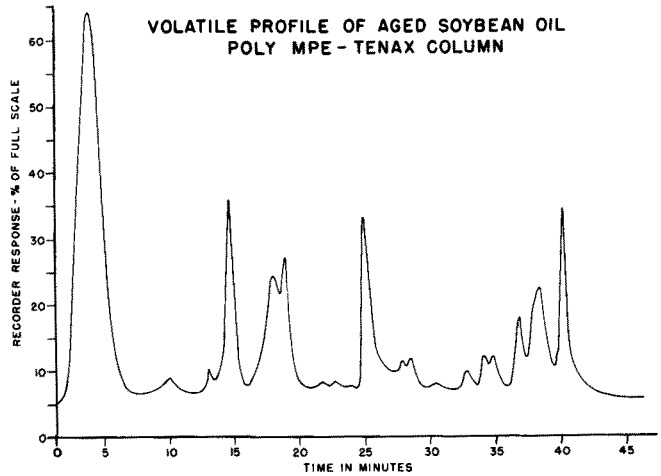

FIG. 2. Poly MPE-Tenax GC chromatogram illustrating the resolution of volatile components of soybean oil aged 5 weeks in the light at 22 C. Conditions are as described by Dupuy (2). An injection port temperature of 170 C was maintained while purging the volatiles from the glass liner.

volatile profile are more symmetrical since they eluted at 230 C.

If an oil leak occurs during the analysis, the column can be cleaned by injecting water, solvents, and reconditioning at 300 C.

ACKNOWLEDGMENT

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