Elution and Analysis of Volatiles in Vegetable Oils by Gas Chromatography

ABSTRACT

A simple and very sensitive technique was devised to analyze volatiles in vegetable oils by direct gas chromatography. A large sample of oil is diffused on glass wool in an injection port liner. After the liner is inserted in the injection port, the volatiles are rapidly swept into the column of the gas chromatograph for analysis.

Hydroperoxides, which decompose to produce volatile flavor components, develop upon storage of vegetable oils. Enrichment techniques have been developed to attain a sufficient concentration of these volatiles for analysis by gas chromatography (GC) and mass spectroscopy. Pyrolysis techniques have been used to decompose the hydroperoxides, and the hydrocarbons produced have been analyzed by GC. The GC flavor profiles produced, however, are extremely complex and of limited value for routine evaluation of oil flavor (1-4).

In the process of developing a simple procedure for the quantitative determination of residual solvent in vegetable oils, a rapid and very sensitive GC technique was developed which can determine volatiles in oils at levels as low as 100 ppb without prior enrichment. In most cases a chromatogram with several peaks is obtained, and these peaks are generally resolved from one another. The number of peaks increases slightly; but the size of some of the peaks increases tremendously, after the seal of a bottle of oil is broken and the oil is left standing in the laboratory. These changes, however, vary with different types of vegetable oils.

A large plug of volatile-free glass wool is inserted at the bottom of the glass liner of the injection port of a GC. The sample of oil is placed on top of the glass wool and covered with a small additional amount of glass wool. The liner is then inserted in the heated injection port which is sealed with the septum nut. The volatiles are eluted rapidly from the oil by the carrier gas and are swept into the column of the GC without contaminating the column with oil. Elution takes place with no apparent decomposition of oil and trace amounts of volatiles are easily detected. The liner with the sample of oil on the glass wool is easily removed from the injection port after the chromatographic run has been completed. Representative chromatograms of a 500 mg sample of a commercial salad oil taken immediately after breaking the seal and of another sample taken after standing in the laboratory for a month are shown in Figure 1. A schematic diagram of the injection port and the liner which contains the sample of oil on the glass wool is shown in Figure 2.

Since this direct GC technique is very sensitive and requires little work, it should be of interest in following degradation of vegetable oils upon aging. This procedure may be useful as an indication of flavor quality and as a guide to predict the shelf life of vegetable oils.

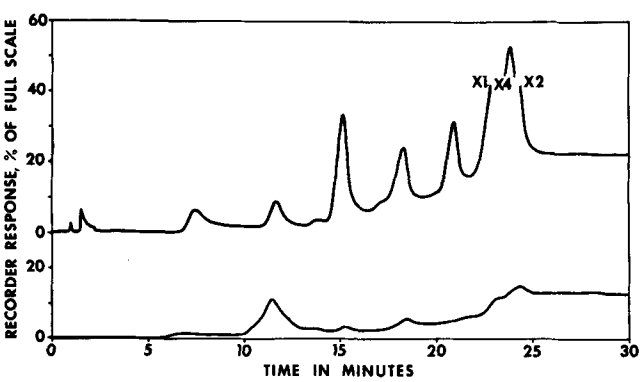

FIG. 1. The lower curve is a chromatogram of volatiles eluted from a sample of commercial salad oil taken immediately after first opening; the upper curve is a chromatogram of volatiles eluted from this oil after standing in the laboratory for a month.

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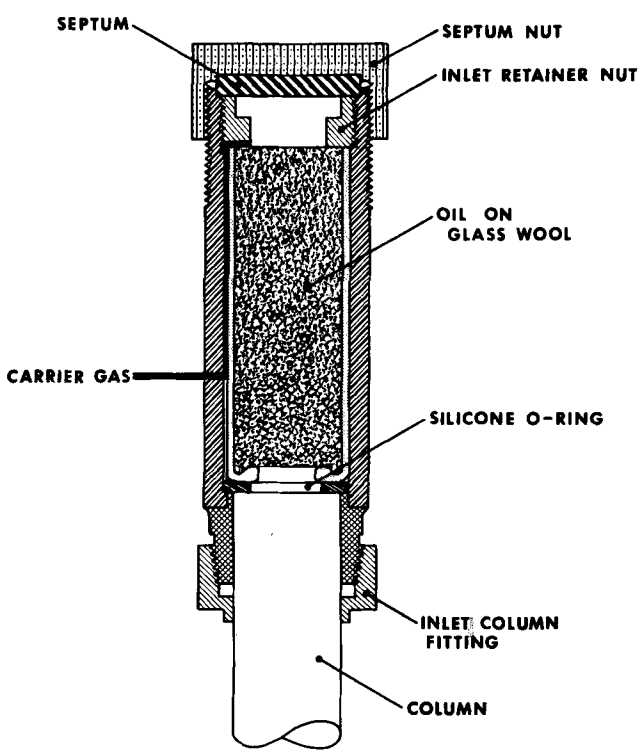

FIG. 2. Schematic diagram of GC injection port and liner.