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Direct Gas Chromatographic Analysis of Volatiles From Raw and Roasted Peanuts

ABSTRACT

Ground samples of raw and roasted peanuts were packed in glass liners and introduced into the heated injection port of a gas chromatograph where the volatiles were vaporized in situ. Chromatograms from ground peanuts are qualitatively similar to those from expressed oil samples. Fourteen compounds were tentatively identified.

Several investigators have used gas chromatographic (GC) profiles for assessing aroma and flavor of various foods and commodities, e.g., raw peanuts (1), roasted peanuts (2,3), cereal grains (4), grapefruit essence (5), apple essence (6) and popcorn (7). In general the isolation and separation of components is tedious and may lead to the formation of artifacts or loss of volatiles (1-3,5,7).

In the course of our investigation into the chemical composition of the volatile fraction of roasted and raw peanuts, the simple and very rapid technique of Dupuy and coworkers (8) was adapted for the GC analysis of peanuts and other foods. Our method is based on the in situ vaporization of the volatiles from peanuts and other food samples which have been inserted directly into the injection port of a gas chromatograph. The technique has been used with good success for assessing raw and roasted peanuts as well as for cabbage, grapefruit rind, meats and tobacco.

A plug of volatile-free glass wool is inserted in the bottom of a glass injection port liner from a gas chromatograph. A sample consisting of a few hundred milligrams or less of ground peanuts or oil is placed on top of the glass wool and backed with additional glass wool. The liner is then inserted into the heated injection port, and the port is sealed with the septum nut. Volatiles flash distil from the sample and are swept onto the GC column. After the chromatographic run has been completed, the liner is easily

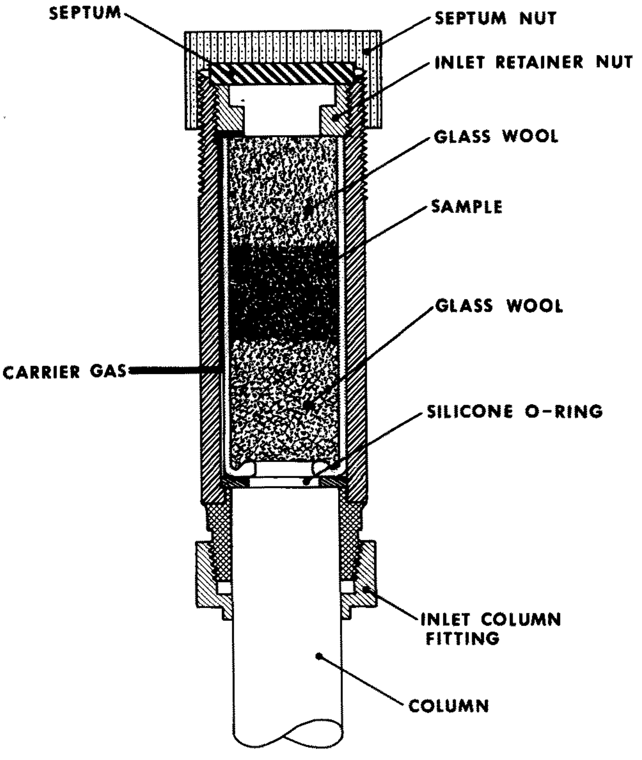

FIG. 1. Schematic diagram showing the injection port liner and peanut sample inserted in the gas chromatogram injection port.

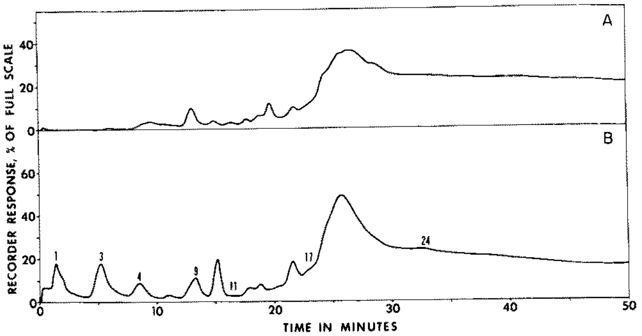

FIG. 2. Gas chromatograms of the volatiles present in raw peanuts and oil expressed from raw peanuts. (A) 200 mg expressed oil; (B) 500 mg whole peanuts. Tentative identifications of peaks: (1) methanol, (3) ethanol, (4) pentane and butanol, (9) pentanal, (11) hexanal and (17) octanal.

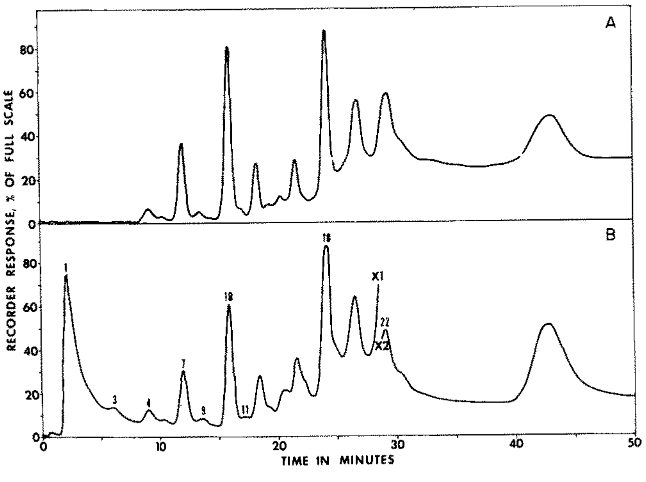

FIG. 3. Gas chromatograms of volatiles present in roasted peanuts and oil expressed from roasted peanuts. (A) 50 mg expressed oil; (B) 100 mg whole peanuts. Tentative identifications of peaks: (1) methanol, (3) ethanol, (4) pentane and butanol, (7) 2-methylpropanal, (9) pentanal, (10) 2-methylbutanal and 3-methylbutanal, (11) hexanal, (18) dimethylpyrazines, and (22) decanal and phenylacetaldehyde.

removed and can be cleaned and reused. A schematic drawing of an injection port with the inserted injection port liner containing a peanut sample between two glass wool plugs is shown in Figure 1.

A MicroTek 2000 MF gas chromatograph equipped with a flame ionization detector and a 2 ft x 1/4 in. OD stainless steel column packed with Porapak P was used in our work. The initial temperature of the injection port was 100 C, but the temperature was programed from 60 to 180 C at 5 C/min after an initial hold period of 2 min. The helium carrier gas flow rate was 60 ml/min.

Representative chromatograms of the volatiles from ground raw peanuts and from cold pressed raw peanut oil are shown in Figure 2. Chromatograms from ground roasted peanuts and the corresponding oil are shown in Figure 3.

The presence of at least 14 and 26 compounds was indicated in raw and roasted Florigiant peanuts, respectively. Tentative identifications for 14 of the compounds are listed under Figures 2 and 3. Identifications were based on cochromatography and retention times of known compounds on the Porapak column. Most of the compounds identified in roasted (2,3) and raw peanuts (1) were identified previously. Further work using a gas chromatograph coupled with a mass spectrometer for identification of the peaks has been started.

Although the first three peaks were missing from oil sample chromatograms, the chromatograms are otherwise qualitatively similar to the chromatograms obtained from ground peanuts. The method is rapid, requiring less than one hour for preparation of the sample and completion of the analysis, and formation of artifacts appears to be nil. It provides a practical screening technique to detect the presence of major volatiles associated with flavor, aroma, or off flavor in roasted peanuts and in oils.

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Determination of Sodium Carboxymethylcellulose in Detergent Products

ABSTRACT

Sodium carboxymethylcellulose in detergent products reacts with phenol in a sulfuric acid media to give a colored reaction product, the absorbance of which is measured at 490 mμ. No interference was experienced from the normal constituents of detergent products. The reproducibility was found to be ± 0.05% absolute.

Recently we needed a relatively quick and accurate method for the determination of sodium carboxymethylcellulose (CMC) in detergent products. Marier and Boulet (1) studied a colorimetric method developed by Barnett and Tawak (2) for determination of lactose in milk and cheese. This was, in turn, based on a paper by DuBois et al. (3). It appeared likely to us that the method of Marier and Boulet could be adapted to the determination of CMC in detergents.

Subsequently we showed that such an adaptation is quite feasible. We have had no interference from the common constituents of detergent products and reproducibility of the method was found to be ± 0.05% absolute.

The only reagents required are concentrated sulfuric acid and a phenol solution. The latter is prepared by mixing 8 g of phenol (Mallinckrodt 0008 or equivalent) and 2 g of deionized water in a small beaker, with subsequent heating on a stream bath to effect solution.

For the so-called "heavy duty" detergents presently being manufactured in this country, which contain CMC in percentages ranging from 0.5-2.5, the following procedure

TABLE I

Run No.	%CMC
1	2.15
2	2.11
3	2.11
4	2.05
5	2.15
6	2.25
7	2.17
8	2.27
9	2.15
10	2.15

TABLE II

CMC[a] originally present, %	CMC added, %	CMC found, %	Recovery, %
0.4	0.3	0.76	108.5
0.4	0.45	0.90	105.5
0.4	0.6	1.03	103.0
0.4	0.9	1.29	99.0
0.4	1.2	1.58	98.8
0.4	1.8	2.18	99.1

[a] CMC = carboxymethylcellulose.