TABLE II

Comparison between Air and Oxygen Bomb Ashing

Oil	Phosphorus (μg/g)		
	Air ashing	Oxygen bomb	Difference (%)
Soybean, crude	112	118	+5.4
Rapeseed, crude	231	229	-0.9
Corn, crude	880	857	-2.6
Blend of several oils	13	14	+7.7
↓	24	25	+4.2
	38	40	+5.3
Rapeseed, crude	245	246	+0.4
Soybean, crude	130	123	-5.4
Rapeseed, crude	242	237	-2.1
Corn, refined	6.6	5.9	
Corn, refined	3.5	4.3	
Soybean, refined	5.4	6.2	
↓	1.2	2.1	
	2.8	4.1	
	8.6	8.4	
Blend of several oils	8.0	7.2	

useful. If more than ca. 15 samples are to be analyzed, the analysis time approaches that for the air ashing. The reason is that the oxygen bomb ashes the samples sequentially, whereas the hot plate ashes the samples concurrently. When the analysis times for a group of samples are similar, the air ashing procedure is more economical because the analyst can be doing other work while the oil is ashing. The oxygen bomb procedure requires the full attention of the analyst.

Both the oxygen bomb and the air ashing methods described here are rather insensitive to variations of several factors. The presence of ZnO is essential for complete combustion of the oil. However, the actual amount of ZnO is not important as long as it exceeds 0.02 g. The volume of dilute nitric acid that is used to dissolve the ash is not critical up to 6 ml. Exceeding this volume decreases the absorbance. The latitude in the acid concentration shows that accurate pH control is unnecessary.

The oxygen bomb method has been used for over 1 year in this laboratory and has been found suitable for fast, accurate, phosphorus determinations in oils. When the number of samples at any one time is large, the air ashing procedure described in this report works equally well.

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⁂Rapid Instrumental Technique for the Analysis of Volatiles in Salad Dressing[1]

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ABSTRACT

A simple, rapid, efficient procedure for analyzing the volatile components in salad dressings is described. A unique inlet system, used in conjunction with gas chromatography and mass spectrometry, provides an objective, tangible profile of volatiles characteristic of the product. The individual components may be identified to qualify more effectively the odor and flavor quality of the specimen.

INTRODUCTION

The detection and identification of volatiles that contribute to food flavor quality have recently become the object of extensive research. Organoleptic methods of assessing food flavor quality, although reasonably effective, are complex and costly (1-3) and are ultimately limited by the taster's judgment. In 1971, Dupuy et al. (4) described a novel, direct, gas chromatography (GC) procedure for determining volatiles in vegetable oils. The method was further refined, and in 1973 (5) it was applied to the examination of oils and shortenings.

Other workers, using variations of this technique (6,7), found excellent correlation between taste panel flavor scores of oils and instrumental data. Although high-moisture foods are inherently unsuitable for combined GC-mass spectrometry (MS) determinations, Legendre et al. (8) have described a unique, versatile inlet system that accommodates both high- and low-moisture foods for GC-MS analysis. This work reports the use of this novel inlet system for the analysis of changes that occur in a salad dressing during storage.

EXPERIMENTAL PROCEDURES

The materials, instruments, GC and MS conditions used in this study have been detailed previously (8). For sample preparation, a 3-3/8 in. length of 3/8 in. od borosilicate glass tubing was packed at one end with ca. 200 mg of glass wool, and 300 mg of potassium carbonate was added. The potassium carbonate functions as a built-in clean-up column

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[2]One of the facilities of the Southern Region, Science and Education Administration, U.S. Department of Agriculture.

to remove interferences. The tubing was capped with ca. 100 mg of additional glass wool. A 200-mg sample of salad dressing was added and covered with a 50-mg plug of glass wool. A clearance of 1/4 in. was allowed at the bottom of the liner and 1/2 in. at the top.

RESULTS AND DISCUSSION

Instrumental analysis of flavorsome materials, e.g., salad dressing, inherently produces chromatograms showing the presence of numerous volatile materials. As shown in Figure 1, profiles of volatiles for a garlic French dressing reveal many compounds, 17 of which have been characterized. The fluctuations that occur in the number and intensity of these compounds during storage (chromatograms 1, 2 and 3) become an important indication of the changes that occur in product quality.

Chromatogram 1, for example, obtained from freshly prepared garlic french-style dressing, shows several peaks, many of moderate intensity, associated with the fresh product. After storage for 6 mo at an ambient temperature, however, changes occur (chromatogram 2) that reflect quality alterations associated with storage time. Propanal, methyl acetate and dimethyl sulfide concentrations are materially increased. Ethanol, dipentene and nonanal quantities remain essentially unchanged. Chromatogram 3, obtained from the same product after 1 yr of storage at an ambient temperature, reflects still greater changes. Most of the volatile peaks have increased considerably, indicating a progressive change toward intensification of the volatiles in the aged product.

These data suggest that a reliable "picture" of flavor-associated components can be produced by analysis of a salad-type dressing with the simple, unconventional, direct GC method. The unique external inlet device and associated condenser permit rapid removal of moisture and thus facilitate the identification of specific components by MS. The information provides a tangible assessment of flavor characteristics that is instrumentally precise, reproducible and objective.

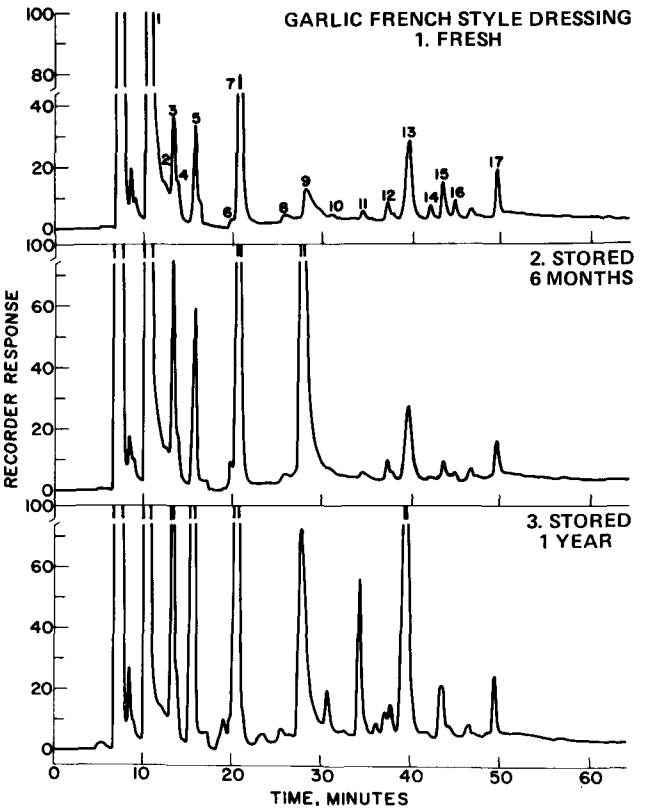

FIG. 1. Chromatograms of garlic French dressing showing effects of storage time. 1: Ethanol; 2: 2-propanol; 3: propanal; 4: acetone; 5: methyl acetate; 6: diacetyl; 7: ethyl acetate; 8: methyl allyl sulfide; 9: dimethyl disulfide; 10: hexanal; 11: 2-heptanone; 12: methyl allyl disulfide; 13: dipentene; 14: nonanal; 15: diallyl trisulfide; 16: pentylbenzene; 17: 3-vinyl-1,2-dithi-5-ene.

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