Developments in Food Science

37A

FOOD FLAVORS: GENERATION, ANALYSIS AND PROCESS INFLUENCE

Edited by
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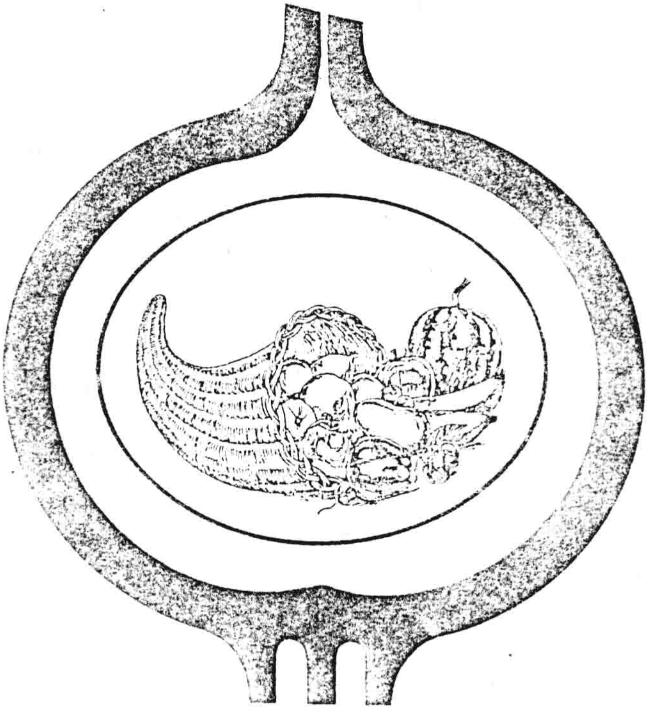

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drying chamber were thermostatically controlled at 180 °C. The dryer was operated in the temperature about 70 °C for exit air.

2.5. Shelf-life prediction

Shelf-life determination was carried out by accelerated method based on the measurement of critical moisture content having incubated in the chamber of 60%, 70%, 80% and 85% RH (Labuza and Schmild, 1985).

2.6 Storage

Products in the form of powder were packed in aluminum-foil pouch, plastic pouch (LDPE) and bottle, and then were stored at ambient conditions. Into some products, anticaking (SiO_2, 2 %), or natural antioxidant (vitamin C, 1%) or synthetic one (BHA-BHT, 1%), or the mixture of anticaking plus antioxidant, were added.

2.7. Analytical methods

The liquid and powder flavor formulations were analyzed for their chemical and physical properties. To avoid degradation, samples were protected against light, air and stored at -10 °C.

2.7.1.Chemical properties Chemical properties observed consisted of: moisture content, ash content (AOAC, 1984), as well as volatile components by VRS and GC/GC-MS analysis.In addition, acid value and peroxide number was observed on stored powder products.

GC and GC-MS. The profile of the volatile components in the extracts was analyzed by using a gas chromatograph (Shimazu GC-9AM) equipped with FID. A polar fused silica capillary column, 50 m x 0.22 mm (carbowax 20M) was operated with a temperature program (40°C for 5 min, then 3°C/min up to 220°C). The carrier gas was Helium (1 ml/sec). Detector and injection port heaters were 250°C and 240°C, respectively. GC-MS analyses were conducted in a Hewlett-Packard 5890A. Mass spectra were obtained by electron ionization (EI). For EI-GC-MS, the ion source temperature was 140 °C; electron energy was 70eV. The GC column was DB-5, 15m x 0.25mm, and was operated under the following temperature condition: 60 °C for 5 min, then 4°/min up to 230°C. The injection was in split-less mode with temperature 240°C. The carrier gas was He.

2.7.2.Physical properties. The observation consisted of: extract yield (AOAC, 1970), powder yield (AOAC, 1970) and solubility test for powder product (AOAC, 1970).

2.7.3.Sensory evaluation. Organoleptic test consisted of: test of threshold perception, multi comparative test as well as hedonic test (Heath, 1981). In addition, rank test (with respect to aromatic strength and solubility) was also applied on powder product. On *multi comparative test*, flavor extract was applied on citrus sauce, and using fresh leaves sauce for comparative standard. *Hedonic test* using scale 1 (very dislike) until 7 (like very much) was carried out for taste and aroma of the flavored powder product applied into "tom yam" soup. "Tom yam" soup added with fresh citrus leaves was used for comparative standard. On *rank test*, the sample was dissolved in water in which the smaller the value means the better the quality.

3. RESULTS AND DISCUSSION

3.1. Pre-treatment on raw material

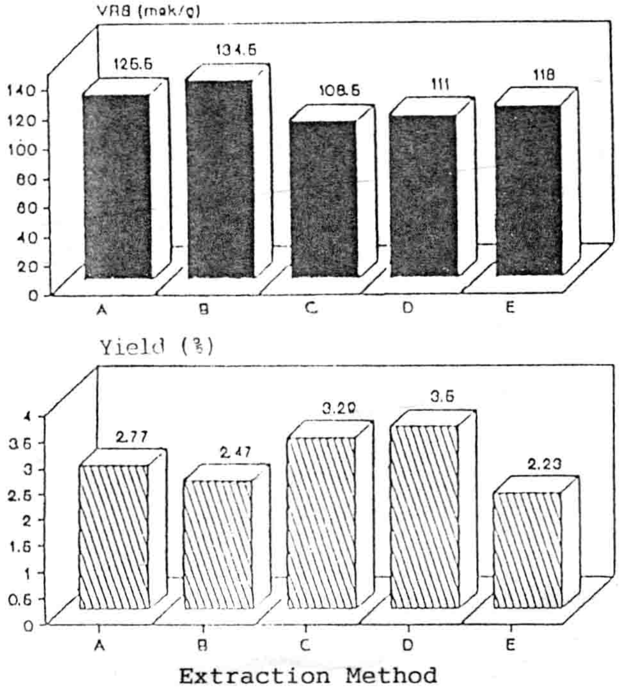
Results of the VRS test suggested that, prior fermentation/aging on shredded leaves for 2, 4 and 6 hours at room temperature of about 26°C did not provide a better result to the product. The declining of VRS value during fermentation might be caused by oxidation of volatile components which have already existed in the culture. The other possibility, as Furia (1975) stated, leave tissue softening occurred during fermentation that facilitated the loosening of volatile components.

3.2. Liquid flavor preparation

Five methods of extraction have been used in preparing the liquid flavor. In the case of solvent selection for maceration and percolation methods, hexane provided a better aroma than alcohol. These results suggest that *C. hystrix* specific aroma are mainly contributed by low-polarity components.

The yields of liquid flavor obtained by various extraction methods are shown in Figure 1. The yield of direct solvent extractions were higher than those of distillation system. On the contrary, the VRS value of the distillation extracts were higher than that of solvent extractions.

Sensorically, except percolation extract, there was no aromatic difference among the five extracts. The percolation extraction gave different aroma from fresh *C. hystrix* leaves, and was less preferred by most of the panelists. Continuous contact with solvent at high temperature might cause the degradation of the "key" flavor compounds.



A=steam distillation; B=water distillation;
C=percolation; D=maceration; E="Likens-Nickerson"

Figure 1. Effect of extraction methods on VRS and yield of *C. hystrix* extracts.

GC profile of water-distilled extract as shown in Figure 2 indicates that *C. hystrix* leaves contained citronellal as the major compounds. Other oxygenated compounds found in great amounts were linalool and citronellol. The occurrence of citronellal as the major component of *C. hystrix* was reported in previous studies using leaves of *C. hystrix* grown in Thailand and Malaysia (Sato et. al., 1990 and Muhammad Nor, 1992). GC-MS profiles and comparison with standards also revealed the presence of *cis* and *trans*-oxide linalool peaks. These are suspected as GC-artifacts of linalool. The occurrence of terpene alcohol and isopulegol peaks in a relatively high concentration are suspected as GC-artifacts of citronellal, the major flavor compound of *C. hystrix* leaves (Anonymous,1980).

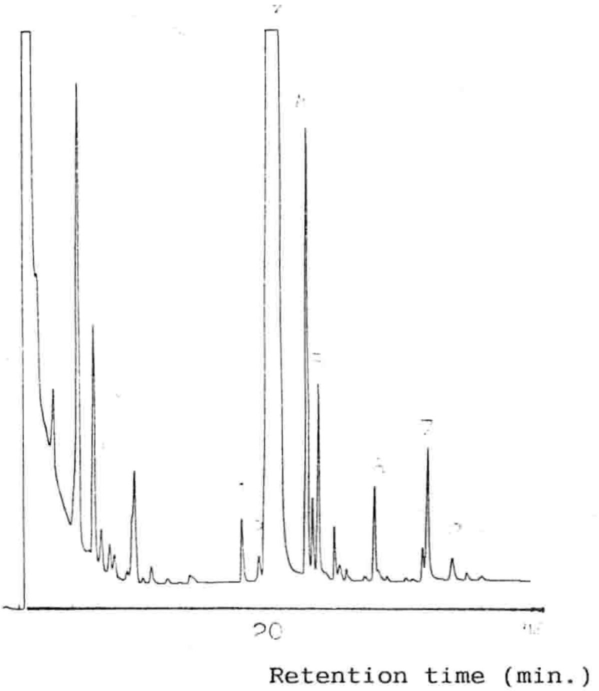


Figure 2. Gas chromatogram of the volatile constituents of *C. hystrix* water-distilled extract. (1) *Cis* oxide-linalool, (2) *Trans* oxide-linalool, (3) Citronellal, (4) Linalool, (5) Isopulegol, (6) Citronellil acetate, (7) Citronellol, (8) Geraniol

The percentage of major compounds of the five liquid flavor extracts are shown in Table 1. Compounds like linalool, citronellol and geraniol in steam and water distilled extracts were lower than in maceration and "Likens-Nickerson's" extracts. In 1985, Ketaren reported that heat will cause oxidation of essential oil. In the presence of organic acids geraniol is oxidized to geranial, citronellol to citronellal, and linalool to linalil ester. Such phenomenon matches with the results of Pickett *et al.* (1975), that alcohol terpene compounds are reduced during the distillation process. The various oxygenated compounds especially citronellol, geraniol and linalool yielded to a different aroma softness.

Table 1.
The percentage of major volatile compounds of the five liquid flavor extracts.

Compounds	Extraction methods[1]				
	A	B	C	D	E
Citronellal	80.67	79.67	59.55	50.32	20.87
Linalool	1.36	0.91	4.81	4.22	0.12
Citronellil acetate	1.22	2.00	0.65	1.83	0.09
Citronellol	6.92	6.51	7.28	14.92	2.28
Geraniol	0.50	0.47	0.09	0.85	0.03

Note:[1] A=water dist., B=steam dist., C=maceration, D=likens-nickerson, E=percolation

In general, "Likens-nickerson" extraction method provides the best aroma flavor extract. It is known that components which are well dispersed in water such as alcohol terpene can not completely extracted in water distillation system. In this case, "Lickens-Nickerson" distillation-extraction system will give better extraction efficiency because of the solvent-water extractor system (Likens and Nickerson, 1964)). In this system, water soluble compounds, such as linalool, geraniol and citronellol (Furia, 1975) were extracted by the solvent. As the result, their percentage in the extract were higher. To some extent, the presence of these compounds can influence the flavor character of the extract.

In the contrary, percolation gave the smallest percentage of these alcohol terpenes. It is predicted that the uncontinuous extraction system limit the contact between solvent and material. Besides continuous heating of the extract in extractor-still may cause components damage. This result confirmed the above sensory evaluation which showed that percolation gave the poorest aroma acceptance.

In the threshold evaluation, extracts which were diluted by one hundred thousand times could still be recognized by 75% of the panelists. The threshold values of each flavor extract are shown in Table 2.

Table 2.
The threshold values of the five liquid flavor extracts.

Extraction methods	Threshold value (% v/v)
Steam-distillation	$5,629 \times 10^{-5}$
Water-distillation	$5,919 \times 10^{-5}$
Likens-nickerson	$6,163 \times 10^{-5}$
Maceration	$5,574 \times 10^{-5}$
Percolation	$9,093 \times 10^{-5}$

3.3. Flavor powder production

Most liquid food flavorings are volatile and chemically unstable in presence of air, light, moisture and high temperature. Microencapsulation has become an attractive approach to transform liquid food flavorings into stable and free flowing powder which are easy to handle and incorporate into a dry food system (Bhandari et al., 1992).

Based on the result of organoleptic/sensoric test, yield, gas chromatography profile, VRS test and technical consideration, water distillation extraction is chosen in this study to be used as liquid flavor extraction method, in which the extract will be further proceed as powdered flavor by spray-drying method. Although powder production can be proceed through various methods of microencapsulation (Dziezak, 1988), it has been reported that spray drying is one of the best known and most economical among others. This technique provides a high retention of flavors during drying, a perfect material solvent, less flavor changing, short heat contact and easy operation, so can produces a powder with quite satisfactory stability (Hall, 1979; Reineccius, 1988).

3.3.1. Coating agents

Two coating agents, gum arabic and dextrin, were used in different levels of concentration. As seen in Table 3, dextrin produced a white and more soluble product than gum arabic. However, the free-flowing of the product from gum arabic was superior to dextrin. The threshold level of the two products was similar.

Table 3.
Physico-chemical properties of *C. hystrix* flavor powders with various coating agents and concentrations.

Coating agents	Yield (%)	Moisture content (%)	Solubility (%)	Threshold values (% v/v)	Sensory evaluation[1] A	B	C[2)]
Gum arabic							
1%	1.7	1.2	71.0	$2,12\text{x}10^{-3}$	4	1	3
2%	2.3	1.2	76.1	$1,99\text{x}10^{-3}$	4	1	3
3%	3.8	1.4	73.9	$2,00\text{x}10^{-3}$	4	1	3
Dextrin							
2%	1.8	2.2	96.3	$1,93\text{x}10^{-3}$	1	2	1
3%	3.0	2.0	96.0	$2,05\text{x}10^{-3}$	1	2	1
4%	5.1	2.4	98.3	$2,13\text{x}10^{-3}$	1	2	1

Note :1) A = powder color, B = free-flowing, C = solubility
2) Smaller value means better quality

Based on the above results, we experimented to combine the two coating agents. Bhandari et al. (1992) stated that a very low retention capacity of dextrin rose steeply even with an addition of minimal quantity of gum. This was due to both the better film forming and the improved emulsifying/stabilizing properties of the gum arabic (Sankarikutty et al., 1988). Therefore, the optimal proportions should be determined. The concentration of 4 % was chosen because of its high yield and better solubility without affecting its threshold level.

Table 4 shows the physico-chemical properties of the encapsulated flavor. A dextrin:gum arabic combination of 3:1 gave the best result with relatively low moisture content, high solubility and high yield. The VRS analysis of this product shows the presence of reducing components in high amount, while the product's aroma itself sensorically is relatively low. It is indicating a successful encapsulation. Subsequently, this product was analyzed for its storage stability.

Table 4
Physico-chemical properties of *C. hystrix* flavor powders with several coating agents combination ratio.

Coating agents ratio	Yield (%)	Moisture content (%)	Ash content (%)	Solu-bility (% v/v)	VRS	Sensory[1] evaluation			
						A	B	C	D
Dex.: G.A.									
4 : 1	4.2	2.1	0.3	97.7	105.6	1	1	1	3
3 : 1	4.8	1.9	0.3	96.9	111.3	1	2	2	3
2 : 1	4.1	1.8	0.4	92.2	117.4	2	3	3	2
1 : 1	3.2	1.8	0.4	89.2	132.4	3	4	4	2
1 : 2	3.1	1.5	0.3	85.7	76.2	3	5	5	1

Note :1) A = powder color, B = free-flowing, C = solubility , D = aroma
2) Smaller value means better quality

3.3.2. Yield and powder properties

The best product of this experiment was produced by encapsulation 0.1% water distilled oil/extract with 4 % coating agent (gum arabic:dextrin = 1:3) in alcohol. The powder flavor produced has 5.6% yield, relatively low moisture content 1.5 %, ash content 0.52%, high VRS of 138.5% mek/g and better water solubility of 99.9%.

Organolepticaly, based on hedonic test, the preferences of the panelists to both odor and taste of the product as well as after the application in "tom yam" soup, did not provide any difference with the fresh leaves. The product even obtained high score averagely. The score showed that the product was preferred. Flavor powder of *C. hystrix* seems possible to substitute the fresh leaves.

3.4. Effect of anticaking and antioxidant on the storage stability of the flavor powder.

Powdered products in general are easy to clump together (Winarno, 1989). In addition, flavor components of citrus family are easy to become rancid/stale because of oxidation (Fang and Ts'eng, 1986; Kimura et al., 1982). Therefore, the shelf-life of the flavor powder was predicted based on the occurrence of caking and rancidity.

When stored in a package at room temperature, caking tend to occur faster than rancidity. Based on this observation, shelf-life prediction was then predicted based on the rate of caking occurrence.

An accelerated study based on the rate of water absorption at different RH was conducted and the critical moisture content of each product can be seen in Table 5. The shelf-life of the products was then predicted using those data and additional information such as permeability and correction factor of the packaging material. When packaged in LDPE plastic without anticaking and antioxidant, the products have a shelf-life of 73 days at Rh 70 and 38 days at RH 85. In HDPE plastic, the shelf-life was longer, i.e. 291 days at RH 70 and 152 days at RH 85. The product shelf-life in other air tight packaging material such as aluminum foil was 1238 days at RH 70 and 650 days at RH 85.

Table 5
The critical moisture content of products at several relative humidity levels.

Humidity	Critical moisture content (%)					
	Control	+SiO_2	+Vit. C	+BHA-BHT	+SiO_2& vit. C	+SiO_2 & BHA-BHT
RH 60%	13.95	13.45	13.82	13.45	13.51	13.45
RH 70%	13.72	12.89	13.29	13.39	12.65	13.39
RH 80%	8.61	8.24	8.13	8.22	8.47	8.82
RH 85%	7.21	7.75	7.43	7.20	6.95	7.47

Beside giving more "free-flowing" characteristic to the initial product, the addition of anticaking also prolong the shelf-life especially in high humidity condition. According to the prediction, the shelf-life of product with SiO_2 packaged in aluminum foil would be 1302 days at RH 70 and 782 days at RH 85.

During storage at room temperature for 6 weeks, the moisture content of the product increased gradually, especially the one packaged in LDPE plastic. In general, aluminum foil and lass-bottle give lower moisture content. The addition of nticaking lower the product' moisture content in all cases.

Acid value of the products tend to increase during storge, however, peroxide value was not detected in all cases, ven after 6 weeks storage. Antioxidant addition, especially HA-BHT reduce the acid value of the product in all packaging aterials. Eventhough BHA-BHT suppressed the acid value, they ave negatives effects on the product aroma, aroma strength and roduct solubility. Considering that the shelf-life of the roduct are more dependent on the caking occurrence before the ccurence of rancidity, the BHA-BHT addition to the product oes not seem necessary.

Furthermore, organoleptic tests on the product as is as ell as after solubilization in water, no caking and rancidity ere detected. Rank test of the aroma strength and solubility howed that the product with anticaking (SiO_2) added as well as nticaking (SiO_2) and antioxidant (vitamin C) added have the ighest scores.

Storage tend to reduce the VRS of the product. However, rganoleptic analysis shows that even after 6 weeks storage, no eviation was detected and the flavor was still acceptable.

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G. Charalambous (Ed.), Food Flavors: Generation, Analysis and Process Influence

SIMPLE ANALYTICAL TECHNIQUE FOR THE DETERMINATION OF RASPBERRY FLAVOR IN A COMPLEX GELATIN MATRIX, DATA AND METHODOLOGY .

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ABSTRACT:

A quantitative gas chromatographic method was developed for the analysis of raspberry flavor from a complex gelatin matrix. The concentrations of a wide range of raspberry flavor volatiles were determined in this study. In particular the concentration of raspberry ketone, raspberry ketone methyl ether and α- and β- ionone as well as several other key components were measured quantitatively. All the thirteen components were also determined quantitatively over a period of time to check the stability of the raspberry flavor in th gel matrix in glass and polypropylene containers.

The analysis was achieved using a home made apparatus "Reflux Concentration Technique". Comparison of the" Reflux Concentration" vs "Liquid Liquid Distillation Extraction[1]" ,the Static and the Dynamic headspace will be discussed. Recoveries of the various components in the raspberry flavor were made at the level of 2 and 20 ppm in the gel matrix. Recoveries between(92-119%) with RSTD (±5% for all the components) were obtained using the reflux concentration technique.

INTRODUCTION:

Raspberry flavor is a complex mixture of many compounds of different compositions and volatilities. Over 230 components have been identified so far(Maarse, 1989). Few of these components are responsible for the raspberry flavor character. An important constituent of the aroma is (p-hydroxyphenyl)butan -2-one the so called raspberry ketone[2,3,4] was first discovered by Sehniz and Seidel[5] (1957) and described as possessing the odor of raspberry by Winter[6] (1961). It was also reported by the Honkanen et al[7], (1980); and Larsem & Poll[8] (1990) as the major compound contributing to unique aroma of raspberry fruit.

Schimidlin Meszaros[9] mentioned that α- and β -ionones are also important components for overall raspberry aroma. They reported very low threshold of 0.0004 ppm for α- ionone[10] and 0.000007ppm for β- ionone[11] as compared with less than 0.1 ppm for the raspberry ketone. It was reported by Hemo Hiresoln[12] et al that the ionones are the compounds responsible for the distinctive flavor of the raspberry. Ionones are volatile compounds with strong scent and taste, whose aroma is described by Ayres et al(1964) as "violet like". Wilson(1939) was the first to investigate the β- ionone of raspberry as the m- nitrobenzhydrazine derivative. Nursten[13] reported that the overal aroma consists of a character impact compound and a few contributory flavor compounds. The impact compound is a raspberry ketone and the contributttory compounds are in particular the ionones.

Although modern analytical methods permitted the analysis of (p-hydroxyphenyl)butan-2-one in raspberry fruit from different geographic origins (Bruan and Hieke, 1977)[14], no attempt has been made, to date, to analyze some of the other important key components such as α- and β- ionones and the other volatile components, and to correlate these analytical data with their organoleptic properties.

In this paper quantitative determination of (p-hydroxyphenyl) butan-2-one) , raspberry ketone methyl ether, α - and β- ionones, and the other important volatiles was achieved from a gel matrix. The organoleptic evaluation of the raspberry flavor with time, in this gel matrix was also investigated, in glass and polypropylene containers. A major objective of this study is to develop a simple quantitative analytical technique for the determination of all the key components of raspberry flavor in gel matrix.

MATERIALS AND METHOD

The flavors were monitored over time in a gel matrix prepared from: gelatin, sugar, acid, water, color and the raspberry flavor. The gel matrix was stored at 40^0F for up to 150 days in both glass and polypropylene cups. Samples from both glass and plastic contaniers were withdrawn at intervals of 0, 14, 21, 30, 60, 90, 120, 13and 150 days for gas chromatographic analysis.

The flavor components were exhaustively extracted from the gel-matrix using a home made "Reflux-Concentration Technique" illustrated in Fig 1. The reflux temperature was controlled at 60^0C. and the condenser temperature was held at -4°C. First, 250 g of the gel is introduced in a 500 ml round -bottom flask, 100 ml of methylene chloride (CH_2Cl_2), containing hexadecane(IS) was then added. The gel and the CH_2Cl_2 mixture was then refluxed for one half hour at the controlled temperatures for both heating and cooling. The CH_2Cl_2 layer was then separated and centrifuged at 6000 RPM for 10 mins. 50 ml aliquot of CH_2Cl_2 was dried over anhydrous sodium sulfate and then concentrated to 2 ml under nitrogen gas, and used in the chromographic analysis.

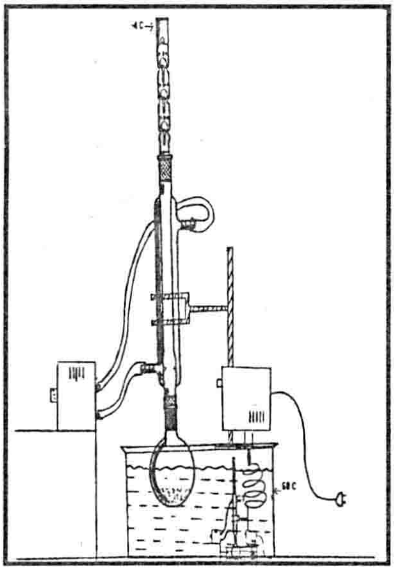

Figure 1. "Reflux-Concentration technique"

Response factor was determined for each of the measured components by the use of a calibration mixture. Pure components were obtained from Aldrich, Sigma and Kodak and included in the calibration mixture at levels equivelant to their levels in raspberry extracts.

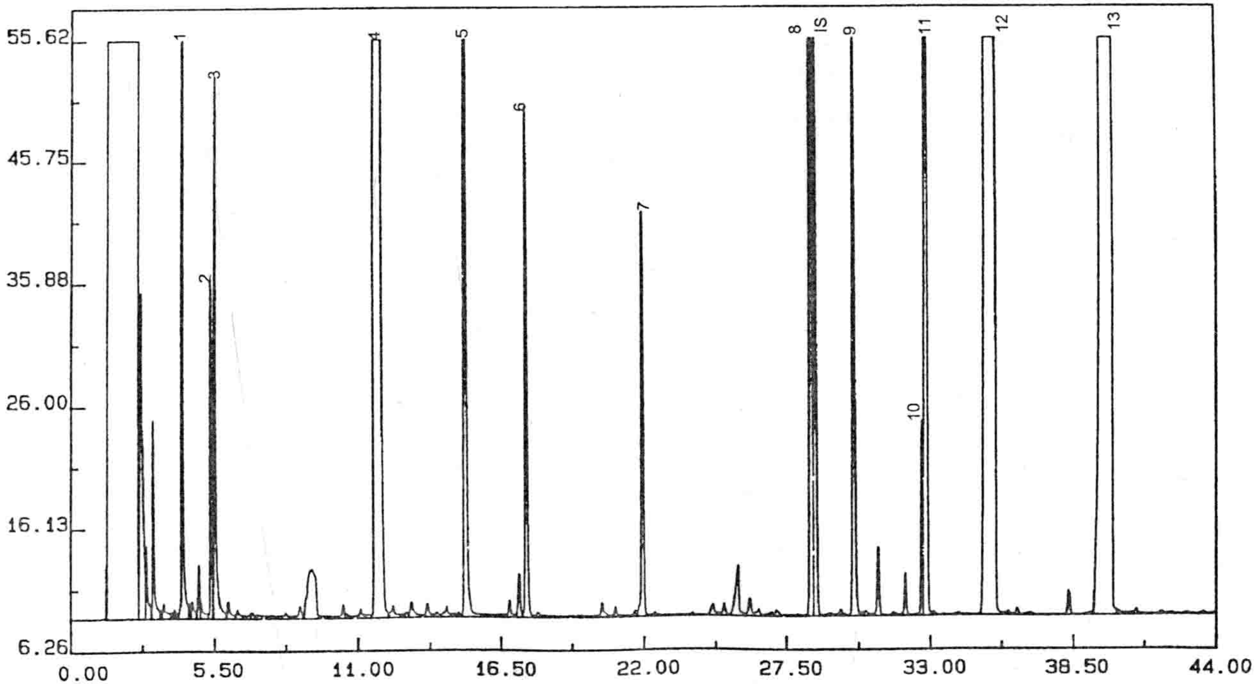

Figure 2. Gas Chromatogram of typical raspberry peaks in gelatin matirx 1) ethyl butyrate 2) ethyl 2 methyl butyrate 3) 3 hexenol 4) benzyl alcohol 5) maltol 6) benzyl acetate 7) benzyl propionate 8) vanillin 9) α ionone 10) β ionone 11) rapberry ketone methyl ether 12) raspberry ketone 13) triethyl citrate IS= internal standard Chrompack Cp sil 8 capillary 25mm 0.32mm

Recovery studies:

Recovery studies were made comparing "Reflux Concentration Technique" with direct injection. A quantity of pure raspberry flavor was diluted in CH_2Cl_2 and then directly injected into the GC; the same amount of flavor was spiked into the Gel matrix and extracted by the "Reflux Concentration Technique" described above, before GC injection. The percent recovery of each component, which is defined as the ratio of the concentration after reflux and concentration as compared wituh direct injection, is given in Tables I & II. Good recoveries were achieved for almost all the components except maltol, which was 64-78% (but it was reproducible). Percent recoveries ranged from(92-119%) with relative standard deviation of (±5%). A comparison was also made between this technique and the liquid/liquid distillation technique. Recovery data from the liquid/liquid distillation technique are given in Table III. Percent recoveries for the raspberry ketone, benzyl alcohol, maltol, raspberry ketone methyl ether, and trietheyl citrate we very poor.

High Performance Gas Chromatography

Quantitative gas chromatographic measurements were made using a Hewlett Packard 5890 gas chromatograph equipped with an auto-sampler , a flame ionization detector, and interfaced with a Hewlett Packard LAS computer system. The column used was a Chrompack #771 Cp sill 8 CB capillary with a length of 25m, an inside diameter of 0.32mm, and a film thickness of 1 mircormeter. The program began with an initial temperature of 80^0C, held for 1 minute followed by a ramp to 200^0C at a rate of 3^0C per minute. The final temperature was held for 5 minutes.

RESULTS AND DISCUSSION

The analytical procedure used in this study proved to be accurate and highly reproducible even though the flavor components are held tightly by the gelatin matrix and are also present in a very low concentration. Using the reflux concentration technique at 60^oC , the heat was adequate to melt the gel matrix and release the flavor components (Fig 1). At the same time the temperature was not very high to cause flavor degradation. The flavor was then extracted with CH_2Cl_2 for half an hour at this temperature, followed by concentration and injection into the GC. 13 components were monitored quantitatively in our study. They are shown in a typical gas chromatogram in Fig 2 and are listed in Table IV.