- Renold, W., Näf-Müller, R., Keller, U., Willhalm, B., Ohloff, G., Helv. Chim. Acta 57, 1301 (1974).
- Roberts, D. L., Rohde, A. A., Tob. Sci. 16, 107 (1972).
- Rodopulo, A. K., Bezzubov, A. A., Egorov, I. A., Skouin, K. P., Vinodel. Vinograd SSSR 34, 53 (1974a).
- Rodopulo, A. K., Egorov, I. A., Bezzubov, A. A., Skouin, K. P., Prikl. Biokhim. Mikrobiol. 10, 280 (1974b).
- Saijyo, R., Takeo, T., Plant Cell Physiol. 13, 991 (1972).
- Scherer, W., Doctoral Dissertation, Techn. Universität München, 1975.
- Schreier, P., Drawert, F., Z. Lebensm.-Unters.-Forsch. 154, 273 (1974a).
- Schreier, P., Drawert, F., Chem. Mikrobiol. Technol. Lebensm. 3, 154 (1974b).
- Schreier, P., Drawert, F., Junker, A., Z. Lebensm.-Unters.-Forsch. 154, 279 (1974a).
- Schreier, P., Drawert, F., Junker, A., Z. Lebensm.-Unters.-Forsch. 155, 98 (1974b).
- Schreier, P., Drawert, F., Junker, A., Z. Lebensm.-Unters.-Forsch. 157, 34 (1975).
- Schreier, P., Drawert, F., Junker, A., Z. Lebensm.-Unters.-Forsch., in press (1976).
- Speck, M., Doctoral Dissertation, Techn. Universität München, 1971.
- Stern, D. J., Lee, A., McFadden, W. H., Stevens, K. L., J. Agric. Food Chem. 15, 1100 (1967).
- Stevens, K. L., Bomben, J. L., Lee, A., McFadden, W. H., J. Agric. Food Chem. 14, 249 (1966).

- Stevens K. L., Bomben, J. L., McFadden, W. H., J. Agric. Food Chem. 15, 378 (1967).
- Stevens, K. L., Flath, R. A., Lee, A., Stern, D. J., J. Agric. Food Chem. 17, 1102 (1969).
- Stevens, K. L., Lee, A., McFadden, W. H., Teranishi, R., J. Food Sci. 30, 1006 (1965).
- Ter Heide, R., de Valois, P. J., Wobben, H. J., Timmer, R., J. Agric. Food Chem. 23, 57 (1975).
- Terrier, A., Boidron, J. N., Ribéreau-Gayon, P., C. R. Hebd. Seances Acad. Sci., Ser. D, 275, 495 (1972a).
- Terrier, A., Boidron, J. N., Ribéreau-Gayon, P., C. R. Hebd. Seances Acad. Sci., Ser. D, 275, 941 (1972b).
- Tressl, R., Drawert, F., Heimann, W., Z. Lebensm.-Unters.-Forsch. 142, 249 (1970a).
- Tressl, R., Drawert, F., Heimann, W., Emberger, R., Phytochemistry 9, 2327 (1970b).
- Usseglio-Tomasset, L., Ind. Agrar., 1 (1966).
- Van Wyk, C. J., Webb, A. D., Kepner, R. E., J. Food Sci. 32, 660 (1967).
- Webb, A. D., Kepner, R. E., Food Res. 22, 384 (1957).
- Webb, A. D., Kepner, R. E., Maggiora, L., Am. J. Enol. Vitic. 17, 247 (1966).
- Winter, M., Enggist, P., Helv. Chim. Acta 54, 1891 (1971).

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## Volatile Flavor Components of Leek

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The flavor complex from steam-distilled leek (Allium porrum L.) was studied by a combination of capillary gas chromatography and mass spectrometry. A total of 67 compounds were characterized, of which 57 have not been previously reported in leek. Odor properties were evaluated by running aromagrams on a thermal conductivity detector.

Much classical work has been done on aroma analysis since the advent of gas-liquid chromatography, especially in combination with mass spectrometry. Among vegetables, the *Allium* genus is one of the best investigated objects as to constitution, formation, and origin of volatile compounds.

The constitution of Allium species has been the subject of many publications. Allium cepa L. (onion) is undoubtedly the most important representative because of its very strong and pungent flavor. It has been studied intensively by Carson (1967), Bernhard (1968), Brodnitz et al. (1969, 1971), Boelens et al. (1971), and by Dembele and Dubois (1973). Other Allium species however, such as A. chinense G. Don (rakkyo), A. sativum L. (garlic), A. schoenoprasum L. (chive), A. porrum L. (leek), and A. victorialis L. (caucas), received little attention. A general and recent review of Allium constituents was published by Johnson et al. (1971).

The mechanism of flavor formation in *Allium* species has been investigated by Stoll and Seebeck (1948, 1949a,b, 1951), Schwimmer and Guadagni (1968), Schwimmer and Weston (1961), Calvallito and Bailey (1950), and some other workers. This paper deals with the isolation, separation, and characterization of the major volatile constituents of leek oil obtained by steam distillation. The components were analyzed using gas-liquid chromatography and mass spectrometry. The structure of these components was identified by direct comparison of their mass spectra and retention times with those of reference products. Many of those products, although known to occur in other *Allium* species, were not reported earlier in leek.

### EXPERIMENTAL SECTION

Sample Preparation. About 30 kg of freshly harvested leek (Nov 1974) was chopped and placed in a 60-l. glass vessel of the pilot plant installation shown in Figure 1. Steam under atmospheric pressure was blown through the leek by means of a 6-kW steam generator. The volatile components were stripped and the vapor condensed and cooled, yielding a total distillate of about 25 l. in 8 h. This distillate was submitted to continuous ether extraction (Figure 2) during 24 h and the extract rectified over a column in order to remove the solvent. The yield was about 1.5 ml of a dark brown leek oil with a strong smell which was very similar to that of boiled leek soup.

Gas Chromatography. The components of the ethereal essence were analyzed on a Varian 2400 gas chromatograph, equipped with a flame ionization detector and a subambient temperature programmer. The subambient system was operated by connecting a liquid nitrogen Dewar

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Figure 1. Steam distillation apparatus: (A) steam generator; (B) 60 l.; (C) condenser; (D) cooler; (E) recipient.

with its own pressure build-up (Cryoson), over a magnetic valve, to the GC oven. The valve was controlled by the linear temperature programmer.

Open tubular columns (600 ft, 0.03 in. i.d.) were prepared from widebore borosilicate glass tubings. The columns were drawn with a commercial glass drawing machine purchased from Hupe and Busch. Column coating was carried out according to the stationary method of Bouche and Verzele (1968) using silicone oil OV 1 at a concentration of 6 mg/ml of solvent. The solvent was dichloromethane. Special care was taken in order to avoid decomposition of products by means of contact with metal parts. Therefore, a 0.25 in. (0.02 in. i.d.) glass tubing was inserted in the injector block and drawn to a capillary end in the oven, in order to connect the column by means of P.T.F.E. shrink tubing. This shrink tubing was also used to connect 300-ft glass tubings in order to obtain a 600-ft column. Columns were conditioned by programming the temperature from 50 to 240°C at 1°C/min and leaving it overnight at 240°C. Operating conditions for GC are as follows: carrier gas nitrogen, 5 ml/min without make-up gas; hydrogen, 30 ml/min; air, 300 ml/min; injector and detector, 220°C; temperature programming, 0-230°C at 1°C/min.

Aromagrams were taken by analyzing the mixture with a microthermal conductivity detector under the following conditions: carrier gas hydrogen, 5 ml/min with make-up gas to 20 ml/min; injector, 220°C; detector, 240°C; isothermal, 15 min at ambient and later 1°C/min to 230°C; filament current, 225 mA.

Gas Chromatography-Mass Spectrometry. For GC-MS coupling a Varian 1200 gas chromatograph was modified and coupled to a MS 30 double beam mass spectrometer by means of a membrane separator sup-

Figure 2. Continuous ether extractor: (A) ether recipient; (B) condenser; (C) extraction column; (D) sintered glass disk.

ported by A.E.I., Manchester. An effluent splitter was installed in order to send about 75% of the total gas flow to the separator and 25% to the F.I.D. When necessary, solvent peaks were eliminated by means of a solvent dump valve placed between the column exit and the separator. Makeup gas was added at the column exit in order to obtain an optimum gas flow through the separator ( $\pm 20$  ml/min). Operating conditions for GC-MS are: carrier gas helium, 6 ml/min, and make-up gas, to 20 ml; injector, 220°C; separator oven and interconnecting lines, 200°C; ion source pressure,  $10^{-5}$  mmHg; ion source, 200°C; trap current, 300  $\mu$ A; filament voltage, 70 V; scan speed, 3 sec/decade.

**Mass Spectrometry.** Reference mass spectra were recorded on the same instrument. The pure products were introduced through an all glass heated inlet system (AGHIS). Operating conditions were as follows: filament voltage, 70 V; accelerating voltage, 4 kV; trap current, 300  $\mu$ A; ion source pressure, 5 × 10<sup>-7</sup> mm; AGHIS and ion source temperature, 200°C; scan speed, 10 sec/decade.

**Reference Compounds.** Some of the reference compounds are commercially available from Fluka, Merck, Riedel-de Haen, Union Chimique Belge, Schuchardt, or Aldrich. The supplier for each compound is indicated in Table I. Many reference spectra were obtained by GC-MS analysis of a commercially available onion oil with known composition (Naarden International, 1974). The composition of this oil has been earlier described in this journal by Boelens et al. (1971).

#### **RESULTS AND DISCUSSION**

As a result of the present investigation 67 components were identified in leek essential oil. Fifty-seven of them have not been reported earlier in leek. A typical gas

Table I. Identification of Volatile Components Found in Leek

no.   Component   plie*   Ip*   fraction   no.   Component   plie*   Ip*   freation     1   Methanal*   M   MS   T   pyraine   801   MS, T     3   Methanethiol*   S, F   MS, RT, A   43   Allyl methyl   895   MS, T     4   Propanal*   U   MS, RT, A   disulfide*   97   914   MS, RT     5   Ethanol   U, A   MS, RT, A   45   Methyl propyl   914   MS, RT     6   Dichloromethane*   U   MS, RT, A   46   Methyl propyl   922   MS, RT, A     1   Propanel   U, A   MS, RT, A   48   Dimethyl   932   MS, RT, A     12   1-Propanethiol*   MS, RT, A   43   Dimethyl   948   MS, RT, A     13   Methylbutanal   633   MS, RT, A   52   Pottanone   972   MS, RT, A     14   2-Methylbutanal   633   MS, RT, A   56	Peak		Sup-		Identi-	Peak		Sup-		Identi-
1   Methanal   MS <sup>d</sup> 42   2.6-Dimethyl- pyrazine   pyrazine     2   Ethanal   M   MS, RT, A   42   2.6-Dimethyl- pyrazine   891   MS, T     2   Ethanal   M   MS, RT, A   43   Allyl methyl   895   MS, RT     4   Propanal <sup>6</sup> U   MS, RT, A   44   Allyl methyl   895   MS, RT     6   Dicthyl ether <sup>4</sup> U   MS, RT, A   46   Methyl propyl   914   MS, RT     7   Dichloromethane <sup>6</sup> U   MS, RT, A   47   Bezaldehyde   A, U   932   MS, RT, A     1   2-Butanone <sup>6</sup> U   MS, RT, A   48   Dimethyl   948   MS, RT, A     12-Depanethiol <sup>6</sup> MS, RT, A   52   2-Ottanone   972   MS, RT, A     13   Ethyl acetate   U   603   MS, RT, A   52   2-Pyrrolearbox   1023   MS, RT, A     14   2-Methylbutanal   A   633   MS, RT, A   52   2-Pyrro	no.	Component	plier <sup>a</sup>	Ip <sup>b</sup>	fication	no	Component	plier <sup>a</sup>	Inb	fication
1 Methanali MS MS Particle Spring <t< td=""><td></td><td>Nether 10</td><td></td><td></td><td>Nod</td><td>40</td><td>0 C Dimethal</td><td></td><td></td><td>Mam</td></t<>		Nether 10			Nod	40	0 C Dimethal			Mam
2 Duration More than thiole More than thiole More than thiole More than thiole Spreadle <	1	Methanal <sup>®</sup>	M			42	2,0-Dimethyl-		991	W15, 1
a Methaletinol* 5, F MS, R1, A 43 Ality methyl 695 MS, R1   5 Bthanol U, A MS, R7, A disulfide* 914 MS, R7   6 Diethyl ether* U MS, R7, A 45 Methyl propyl 922 MS, R7   7 Diethyl ether* U MS, R7, A 46 Methyl propyl 922 MS, R7   7 Diethyl ether* U MS, R7, A 47 Benzaldehyde A, U 932 MS, R7, A   10 n-Propanol U, A MS, R7, A 47 Benzaldehyde A, U 932 MS, R7, A   11 2.Bithyl acetate U 603 MS, R7, A 52 2-Octanone 972 MS, R7, A   13 2.Methylbutanal A 633 MS, R7, A 53 2-Bithyl furyl 983 MS, R7, A   14 2.Methylbutanal A 675 MS, R7, A 53 Paryl acohol 1013 MS, R7, A   13 Allyl methyl 678 MS, R7, A 61 2-Pinylethonol 1060 MS, R7, A	2	Ethanal MathamathialC			MS, KI, A	40			005	
4 Propanal 0 MS, R1, A disulfide   6 Diethyl ether <sup>4</sup> U MS, RT, A disulfide   7 Dichformethane <sup>6</sup> U MS, RT, A disulfide   9 Allyl alcohol A MS, RT, A disulfide   9 Allyl alcohol A MS, RT, A disulfide   9 Allyl alcohol A MS, RT, A disulfide   12 Dropanch WS, RT, A 48 Dimethyl 948 MS, RT, A   13 Ethyl acetate U 603 MS, RT, A 52 2-Octanone 972 MS, RT, A   14 2-Methylbotanal A 633 MS, RT, A 58 Dimethyl 983 MS, RT, A   15 3-Methylbotanal A 675 MS, RT, A 58 Denzyl alcohol U 1013 MS, RT, A   16 2-Methylbotanol U 664 MS, RT, A 61 2-Noranone 1073 MS, RT, A   19 Allyl methyl 678 MS, RT, A 61 2-Dimethylshylbotanol 1066	3		5, F		MS, RT, A	43	disulfide		895	MS, RT
3 Entanol U, A Mis, RT, A 46 Methyl propyl 914 Mis, RT   7 Dichloromethane <sup>6</sup> U Mis, RT, A 46 Methyl propyl 922 Mis, RT   7 Dichloromethane <sup>6</sup> U Mis, RT, A 46 Methyl propyl 922 Mis, RT   10 n-Propaneli U, A Mis, RT, A 47 Benzaldehyde A, U 932 Mis, RT, A   11 2-Butanone <sup>6</sup> U Mis, RT, A 47 Benzaldehyde A, U 932 Mis, RT, A   12 1-Propanethiol <sup>6</sup> Mis, RT, A 47 Benzaldehyde A, U 932 Mis, RT, A   13 Ethyl acetate U 603 Mis, RT, A 53 2-Ethyl furyl 983 Mis, RT, A   14 2-Methylbutanal A 675 Mis, RT, A 56 2-Pyrrolearbox- 1023 Mis, RT, A   19 Allyl methyl 678 Mis, RT, A 60 n-Octanol A 1060 Mis, RT, A   2-Hydroxy-3- A 689 Mis, RT 65 2-Pyrolearbox- <td>4</td> <td>Fropanal</td> <td>U A</td> <td></td> <td>MS, RT, A</td> <td>45</td> <td></td> <td></td> <td>014</td> <td>Ma DW</td>	4	Fropanal	U A		MS, RT, A	45			014	Ma DW
b Detryl ether O MS, R1, A disulfide <sup>c</sup> Dichromethane <sup>e</sup> U MS, RT, A disulfide <sup>c</sup> 9 Allyl alcohol A MS, RT, A 46 Methyl propyl 922 MS, RT, A   9 Allyl alcohol A MS, RT, A 46 Methyl propyl 922 MS, RT, A   12 .Propanel U MS, RT, A 48 Dimethyl 948 MS, RT, A   12 .Propanethiol <sup>c</sup> MS, RT, A 52 2-Octanone 972 MS, RT, A   15 .3-Methylbutanal 633 MS, RT, A 52 2-Octanone 972 MS, RT, A   16 .2-Methylbutanal A 633 MS, RT, A ketone 1013 MS, RT, A   17 <i>n</i> -Butanol U 664 MS, RT, A idelylde 1023 MS, RT, A   18 <i>n</i> -Pentanal A 675 MS, RT, A idelylde 1013 MS, RT, A   18 <i>n</i> -Pentanal A 675 MS, RT, A 610 10-Octanol A 1060 MS, RT, A   2 2-Hydroxy-3. A 689 MS, RT, A 61 12-Diropyl </td <td>0</td> <td></td> <td>U, A</td> <td></td> <td>MS, RT, A</td> <td>45</td> <td>Metnyl propyl</td> <td></td> <td>914</td> <td>MS, RT</td>	0		U, A		MS, RT, A	45	Metnyl propyl		914	MS, RT
A lityl alcohol A MS, RT, A Ms, RT Ms, RT, A Ms, RT	07	Dietnyl etner	U U		MS, RT, A	40	disulfide		000	
9 Altyl alcohol A MS, RT, A disulfide*   9 Altyl alcohol U MS, RT, A 47 Benzaldehyde A, U 932 MS, RT, A   11 2-Butanone* U MS, RT, A 48 Dimethyl 948 MS, RT, A   12 12-Propanethiol* MS, RT, A 48 Dimethyl 948 MS, RT   13 Ethyl acetate U 603 MS, RT, A 52 -2-Otanone 972 MS, RT   14 2-Methylpotanol A 633 MS, RT, A 52 -2-Otanone 972 MS, RT, A   15 3-Methylputanal A 633 MS, RT, A 56 2-Pstrolearbox 1023 MS, RT, A   16 2-Methylputanal A 675 MS, RT, A 66 2-Pstrolearbox 1023 MS, RT, A   17 <i>n</i> -Butanol U 664 Ms projendehyde 1073 MS, RT, A   16 2-Methylputanol A 678 MS, RT, A 66 2-Dimethox 1038 MS, RT, A   2 Methylbutan			U A		MS, RI, A	40	Methyl propyl		922	MS, RT
10 $n$ -rropanol $0$ , A MS, RT, A 47 Benzalenyde A, U 932 MS, RT, A   12 12-Butanone' U MS, RT, A 48 Dimethyl $0$ 948 MS, RT   12 1-Propanethiol' MS, RT, A 48 Dimethyl 948 MS, RT   12 1-Propanethiol' MS, RT, A 52 -Octanone 972 MS, RT   14 2-Methylptranal A 627 MS, RT, A 53 2-Ehryl furyl 983 MS, RT   15 3-Methylptranal A 627 MS, RT, A 56 Benzyl alcohol U 1013 MS, RT, A   16 2-Methylputanal A 675 MS, RT, A 56 2-Pyrrolcarbox- 1023 MS, RT, A   18 Allyl methyl 678 MS, RT, A 61 2-Nonanone 1073 MS, RT, A   20 Hydroxy-3- A 689 MS, RT, A 65 2-Phenylethanol M 1096 MS, RT, A   21 Pyridine A 719 MS, RT 67 2	10	Allyl alconol	A		MS, RT, A		disulfide			
11 2-Butatone <sup>e</sup> 0 MS, R1, A 48 Dimetryl 948 MS, RT   12 1-Propanethiol <sup>e</sup> MS, RT, A 52 2-Octanone 972 MS, RT   13 Ethyl acetate U 603 MS, RT, A 52 2-Octanone 972 MS, RT   14 2-Methylbutanal A 633 MS, RT, A 52 2-Octanone 972 MS, RT   15 3-Methylbutanal A 633 MS, RT, A 52 2-Dyrolearbox- 1023 MS, RT, A   16 $n$ -Pentanal A 675 MS, RT, A 56 2-Pyrrolearbox- 1023 MS, RT, A   19 Allyl methyl 678 MS, RT, A 64 Dipropyl F 1096 MS, RT, A   10 2-Hydroxy-3- A 689 MS, RT, A 64 Dipropyl F 1096 MS, RT, A   2 2-Methylbuetanal A 719 MS, RT, A 64 2-Dimethox- 1114 MS, RT, A   2 2-Methylbuetanal A 719 MS, RT, A 12-	10	n-Propanol	U, A		MS, RI, A	47	Benzaldehyde	A, U	932	MS, RT, A
12 1.770 panetanio MS, R1 trisulfide   13 Ethylp acetate U 603 MS, RT, A 52 2-Octanone 972 MS, RT   14 2-Methylptopanol A 627 MS, RT, A 53 2-Octanone 972 MS, RT   15 3-Methylptanal A 633 MS, RT, A 53 2-Octanone 972 MS, RT   16 2-Methylptanal A 633 MS, RT, A 56 Benzyl alcohol U 1013 MS, RT, A   17 n-Butanol U 664 MS, RT, A 56 2-Pyrrolearbox- 1023 MS, RT, A   18 n-Pentanal A 675 MS, RT, A 64 Dipropyl F 1092 MS, RT, A   20 2-Hydroxy-3- A 689 MS, RT, A 64 Dipropyl F 1092 MS, RT, A   21 Pyridine A 719 MS, RT 66 1,2-Dimethoxy- R 1114 MS, RT, A   22 Methylbrontonl T22 MS, RT 67 Methylbr	11	2-Butanone	U		MS, RT, A	48	Dimethyl		948	MS, RT
13 EMP(1 acetate 0 603 MS, RT, A 52 2-Octanone 972 MS, RT   14 2-Methylbutanal A 633 MS, RT, A 52 2-Dethyl furyl 983 MS, RT   15 3-Methylbutanal A 633 MS, RT, A 56 Enzyl alcohol U 1013 MS, RT, A   16 n-Pentanal A 675 MS, RT, A 56 2-Pyrrolearbox- 1023 MS, RT, A   19 Ally methyl 678 MS, RT, A 61 2-Nonanone 1073 MS, RT, A   20 2-Hydroxy-3- A 689 MS, RT, A 64 Dipropyl F 1092 MS, RT, A   21 Dimethyl disulfide 723 MS, RT 66 2-Penylethanol M 1096 MS, RT, A   22 Methylpontanol' 722 MS, RT 66 1,2-Dimethoxy- R 1114 MS, RT, A   23 Methylpontanol' 722 MS, RT, A 72 2,5-Dihydro-3,4- 1193 MS, RT   24 2-Methylpentanol F 827<	12	1-Propanethiol			MS, RT		trisulfide			
14 2-Methylpotanol A 627 MS, RT, A 53 2-Ethyl furyl 983 MS, RT, A   15 3-Methylbutanal A 633 MS, RT, A 53 2-Ethyl furyl 983 MS, RT, A   16 2-Methylbutanal A 633 MS, RT, A 56 Benzyl alcohol U 1013 MS, RT, A   17 n-Butanol U 664 MS, RT, A 56 2-Pyrrolcarbox- 1023 MS, RT, A   19 Allyl methyl 678 MS, RT, A 66 2-Octanol A 1060 MS, RT, A   20 2-Hydroxy-3- A 689 MS, RT, A 66 2-Donanone 1073 MS, RT, A   21 Pyridine A 719 MS, RT, A 66 2-Dimethoxy- R 1114 MS, RT, A   22 Dimethyl disulfide 723 MS, RT 66 2-Dimethoxy- R 1114 MS, RT, A   23 Methylbunanol 722 MS, RT, A 71 2-Doemethoxy- R 1114 MS, RT, A   24 2-	13	Ethyl acetate	U	603	MS, RT, A	52	2-Octanone		972	MS, RT
	14	2-Methylpropanol	A	627	MS, RT, A	53	2-Ethyl furyl		983	MS, RT
16 2-Methylputanal 643 MS, RT, A 55 Benzyl alconol U 1013 MS, RT, A   17 n-Butanol U 6643 MS, RT, A 56 2-Pyrnolearbox- 1023 MS, T   18 n-Pentanal A 675 MS, RT, A 56 2-Pyrnolearbox- 1023 MS, T   19 Allyl methyl 678 MS, RT, A 60 n-Octanol A 1060 MS, RT, A   20 2-Hydroxy-3- A 689 MS, RT, A 61 2-Nonanone 1073 MS, RT, A   21 Pyridine A 719 MS, RT, A 64 Dipropyl F 1092 MS, RT, A   22 Dimethyl disulfide 723 MS, RT 66 2-Phenylethanol M 1096 MS, RT, A   23 Methylpotanol U 765 MS, RT, A 72 2-Dimethoxy- R 1114 MS, RT, A   24 2-Methylpotnanol U 765 MS, RT, A 72 2-Decanone 1174 MS, RT   21 Methylpotnanol <td>15</td> <td>3-Methylbutanal</td> <td>Α</td> <td>633</td> <td>MS, RT, A</td> <td></td> <td>ketone</td> <td></td> <td>1 0 1 0</td> <td></td>	15	3-Methylbutanal	Α	633	MS, RT, A		ketone		1 0 1 0	
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	10	2-Methylbutanal		643	MS, RT	22	Benzyl alcohol	U	1013	MS, RT, A
18 $n$ -Pentanal A 675 MS, RT, A aldehyde   19 Allyl methyl 678 MS, RT 60 $n$ -Octanol A 1060 MS, RT, A   20 2-Hydroxy.3- A 689 MS, RT, A 60 $n$ -Octanol A 1060 MS, RT, A   21 Pyridine A 719 MS, RT, A 61 $2$ -Nonanone 1073 MS, RT, A   22 Dimethyl disulfide 723 MS, RT 61 $2$ -Phenylethanol M 1096 MS, RT, A   23 3-Methylbutanol' 722 MS, RT 61 $12$ -Dimethoxy- R 1114 MS, RT, A   24 2-Methylpentanal 740 MS, RT, A 71 $2$ -Decanone 1174 MS, RT   25 Methylptyrazine 755 MS, RT, A 71 $2$ -Decanone 1174 MS, RT   26 $n$ -Hexanal A 779 MS, RT, A 72 $2$ -Decanone 1174 MS, RT   25 Methylpyrazine 798 MS, RT 73 Benzothiazole A	17	n-Butanol	Ų	664	MS, RT, A	56	2-Pyrrolcarbox-		1023	MS, T
	18	<i>n</i> -Pentanal	Α	675	MS, RT, A		aldehyde			
sulfide61 $2.Nonanone1073MS, RT202.Hydroxy-3-A689MS, RT, A64DipropylF1092MS, RT, A21PyridineA719MS, RT, A652.PhenylethanolM1096MS, RT, A22Dimethyl disulfide723MS, RT652.PhenylethanolM1096MS, RT, A233.Methylbutanol^{f}722MS, RT661.2.Dimethoxy-R1114MS, RT, A233.Methylbotanol^{f}722MS, RT67hetnyl nopyl1132MS, RT, A242.Methylpentanal740MS, RT67Methyl propyl1132MS, RT242.Methylpentanal740MS, RT, A77hetnyl nopyl1132MS, RT25Methylbiophene75569n-Nonanol1160MS, RT26n-PentanolU762MS, RT, A722.5-Dihydro-3,4-1193MS, RT29Methylpyrazine798MS, RT, A722.5-Dihydro-3,4-1196MS, RT, A302-PertaldehydeF, A804MS, RT, A78Decomposed129131trans-Hex-3-en-843MS, RT79Propenyl propyl1310MS, RT322.Methylphoino855MS, RT812.3-Dihydro-2n 1413MS, RT342.Furyl alcoholF846MS, RT, A$	19	Allyl methyl		678	MS, RT	60	<i>n</i> -Octanol	Α	1060	MS, RT, A
20 2-Hydroxy-3- butanone <sup>f</sup> A 689 MS, RT, A 64 Dipropyl F 1092 MS, RT, A   21 Pyridine A 719 MS, RT, A 64 Dipropyl F 1092 MS, RT, A   21 Pyridine A 719 MS, RT, A 65 2-Phenylethanol M 1096 MS, RT, A   23 Methylbutanol <sup>f</sup> 722 MS, RT 66 1,2-Dimethoxy- R 1114 MS, RT, A   24 2-Methylpentanal 740 MS, RT 67 Methyl propyl 1132 MS, RT   (2 isomers) 755 69 n-Nonanol 1160 MS, RT   25 Methylpyrazine 798 MS, RT, A 72 2,5-Dihydro-3,4- 1193 MS, RT   29 Methylpyrazine 798 MS, RT, A 73 Benzothiazole A 1196 MS, RT, A   20 2-Furaldehyde F, A 804 MS, RT, A 79 Propenyl propyl 1310 MS, RT   31 trans-Hex-3-en- 843 MS, RT 78		sulfide				61	2-Nonanone		1073	MS, RT
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	20	2-Hydroxy-3-	Α	689	MS, RT, A	64	Dipropyl	F	1092	MS, RT, A
$\begin{array}{cccccccccccccccccccccccccccccccccccc$		butanone'					disulfide <sup>c</sup>			
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	21	Pyridine	Α	719	MS, RT, A	65	2-Phenvlethanol	М	1096	MS. RT. A
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	<b>22</b>	Dimethyl disulfide		723	MS, RT	66	1.2-Dimethoxy-	R	1114	MS. RT. A
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	23	3-Methylbutanol <sup>7</sup>		722	MS, RT	-	benzene			,,
$\begin{array}{cccccccccccccccccccccccccccccccccccc$	<b>24</b>	2-Methylpentanal		740	MS, RT	67	Methyl propyl		1132	MS. RT
$ \begin{array}{cccccccccccccccccccccccccccccccccccc$	25	Methylthiophene		753-	MS, RT		trisulfide			<b>,</b>
26 $n$ -Pentanol U 762 MS, RT, A 71 2-Decanone 1174 MS, RT   28 $n$ -Hexanal A 779 MS, RT, A 72 2,5-Dihydro-3,4- 1193 MS, RT   29 Methylpyrazine 798 MS, T dimethylthio- phen-2-one 1174 MS, RT   30 2-Furaldehyde F, A 804 MS, RT, A 72 2,5-Dihydro-3,4- 1193 MS, RT   30 2-Furaldehyde F, A 804 MS, RT 73 Benzothiazole A 1196 MS, RT, A   9 Pothylpent-2- 811 MS, RT 73 Benzothiazole A 1196 MS, RT, A   71 2-Methylpentanol F 827 MS, RT, A 79 Propenyl propyl 1310 MS, RT   33 trans-Hex-3-en- 843 MS, RT 79 Propenyl propyl 1310 MS, RT   34 2-Furyl alcohol F 846 MS, RT, A 80 Propenyl propyl 1317 MS, RT   35 Dimethylthio- 855 M		(2 isomers)		755		69	n-Nonanol		1160	MS. RT
28 $n$ -HexanalA779MS, RT, A722,5-Dihydro-3,4-1193MS, RT29Methylpyrazine798MS, Tdimethylthio- $phen-2-one$ $methylthio phen-2-one$ 302-FuraldehydeF, A804MS, RT, A $phen-2-one$ $methylthio phen-2-one$ 312-Methylpent-2-811MS, RT73BenzothiazoleA $1196$ MS, RT, A322-MethylpentanolF827MS, RT, A79Propenyl propyl $1310$ MS, RT33trans-Hex-3-en-843MS, RT $trisulfide$ $trisulfide$ $ms, RT$ 1-ol4-855MS, RT $trisulfide$ $trisulfide$ 342-Furyl alcoholF846MS, RT, A80Propenyl propyl $1317$ MS, RT35Dimethylthio- phene (isomer)858MS, RT81 $2,3$ -Dihydro-2- $n$ - $1413$ MS, RT36Allyl propyl sulfide859MS, RT, A $81$ $2,3$ -Dihydro-2- $n$ - $1413$ MS, RT36Dimethylthio- phene (isomer)861MS, RT $82$ $2$ -Tridecanone $1477$ MS, RT392-Heptanone phene871MS, RT $83$ $2,3$ -Dihydro-2- $n$ - $1619$ MS, T413,4-Dimethylthio- phene887MS, RT $84$ $2$ -Pentadecanone $1681$ MS, RT413,4-Dimethylthio- phene887MS, RT $84$ $2$ -Pentadecanone $1681$ <t< td=""><td>26</td><td><i>n</i>-Pentanol</td><td>U</td><td>762</td><td>MS, RT, A</td><td>71</td><td>2-Decanone</td><td></td><td>1174</td><td>MS. RT</td></t<>	26	<i>n</i> -Pentanol	U	762	MS, RT, A	71	2-Decanone		1174	MS. RT
29Methylpyrazine798MS, Tdimethylthio-302-FuraldehydeF, A804MS, RT, A $phen-2-one$ 312-Methylpent-2-811MS, RT73BenzothiazoleA312-MethylpentanolF827MS, RT, A78Decomposed1291322-MethylpentanolF827MS, RT, A79Propenyl propyl1310MS, RT33trans-Hex-3-en-843MS, RT79Propenyl propyl1310MS, RT1-ol342-Furyl alcoholF846MS, RT, A80Propenyl propyl1317MS, RT1-ol36Allyl propyl858MS, RT812,3-Dihydro-2-n-1413MS, RT37n-HexanolU859MS, RT, AMS, RT38Dimethylthio- phene (isomer)812,3-Dihydro-2-n- furan-3-one1413MS, RT392-HeptanoneMS, RT413,4-Dimethylthio- phene413,4-Dimethylthio- phene413,4-Dimethylthio- phene	28	<i>n</i> -Hexanal	Α	779	MS, RT, A	72	2.5-Dihvdro-3.4-		1193	MS. RT
302-FuraldehydeF, A804MS, RT, Aphen-2-one312-Methylpent-2- enal811MS, RT73BenzothiazoleA1196MS, RT, A322-MethylpentanolF827MS, RT, A78Decomposed1291322-MethylpentanolF843MS, RT79Propenyl propyl1310MS, RT33trans-Hex-3-en- 1-ol843MS, RT, A79Propenyl propyl1310MS, RT342-Furyl alcoholF846MS, RT, A80Propenyl propyl trisulfide1317MS, RT36Allyl propyl sulfide858MS, RT812,3-Dihydro-2-n- hexyl-5-methyl-1413MS, RT37n-HexanolU859MS, RT, A selfide861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT, A selfide887MS, RT832,3-Dihydro-2-n- hexyl-5-methyl-1619MS, T413,4-Dimethylthio- phene871MS, RT, A selfide887MS, RT842-Pentadecanone furan-3-one1681MS, RT413,4-Dimethylthio- phene887MS, RT, A selfide85Diphenvlacetvlene>1700MS, T	29	Methylpyrazine		798	MS, T		dimethylthio-		1100	
312-Methylpent-2- enal811MS, RT73BenzothiazoleA1196MS, RT, A322-MethylpentanolF827MS, RT, A78Decomposed1291322-MethylpentanolF843MS, RT, A79Propenyl propyl1310MS, RT33 $trans-Hex-3-en-$ 843MS, RT79Propenyl propyl1310MS, RT342-Furyl alcoholF846MS, RT, A80Propenyl propyl1317MS, RT35Dimethylthio- phene (isomer)858MS, RT80Propenyl propyl1317MS, RT36Allyl propyl sulfide859MS, RT, A80Propenyl propyl (isomer)1413MS, RT37 $n$ -HexanolU859MS, RT812,3-Dihydro-2-n- furan-3-one1413MS, RT392-Heptanone871MS, RT822-Tridecanone furan-3-one1477MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone furan-3-one1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone stophene1681MS, RT	30	2-Furaldehyde	F, A	804	MS, RT, A		phen-2-one			
enal78Decomposed1291322-MethylpentanolF827MS, RT, A79Propenyl propyl1310MS, RT33 $trans-Hex-3-en-$ 843MS, RT $trisulfide$ (isomer)1310MS, RT342-Furyl alcoholF846MS, RT, A80Propenyl propyl1317MS, RT35Dimethylthio-855MS, RT80Propenyl propyl1317MS, RT36Allyl propyl858MS, RT812,3-Dihydro-2-n-1413MS, RT37n-HexanolU859MS, RT, Ahexyl-5-methyl- furan-3-one1413MS, RT38Dimethylthio- phene (isomer)861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT, A832,3-Dihydro-2-n- furan-3-one1619MS, T413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT45Diphene87MS, RT85Diphenylacetylene>1700MS, T	31	2-Methylpent-2-		811	MS, RT	73	Benzothiazole	Α	1196	MS RT A
322-MethylpentanolF827MS, RT, A79Propenyl propyl1310MS, RT33 $trans$ -Hex-3-en- 1-ol843MS, RT79Propenyl propyl1310MS, RT342-Furyl alcoholF846MS, RT, A80Propenyl propyl1317MS, RT35Dimethylthio- phene (isomer)855MS, RT80Propenyl propyl1317MS, RT36Allyl propyl sulfide858MS, RT812,3-Dihydro-2-n- furan-3-one1413MS, RT37n-HexanolU859MS, RT, A selfide861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT, A selfide881MS, RT, A selfide832,3-Dihydro-2-n- furan-3-one1619MS, T413,4-Dimethylthio- phene887MS, RT842-Pentadecanone selfide1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT		enal				78	Decomposed		1291	,,
33 $trans$ -Hex-3-en- 1-ol843MS, RT $trisulfide$ (isomer)1010 $hds, RT$ 342-Furyl alcoholF846MS, RT, A80Propenyl propyl1317MS, RT35Dimethylthio- phene (isomer)855MS, RT80Propenyl propyl1317MS, RT36Allyl propyl sulfide858MS, RT812,3-Dihydro-2-n- furan-3-one1413MS, RT37 $n$ -Hexanol phene (isomer)U859MS, RT, A 861812,3-Dihydro-2-n- furan-3-one1417MS, RT392-Heptanone phene871MS, RT822-Tridecanone furan-3-one1477MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone furan-3-one1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone furan-3-one1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone furan-3-one1681MS, RT	32	2-Methylpentanol	F	827	MS, RT, A	79	Propenyl propyl		1310	MS RT
$\begin{array}{c ccccccccccccccccccccccccccccccccccc$	33	trans-Hex-3-en-		843	MS, RT	10	trisulfide		1010	
342-Furyl alcoholF846MS, RT, A80Propenyl propyl1317MS, RT35Dimethylthio- phene (isomer)855MS, RT80Propenyl propyl1317MS, RT36Allyl propyl sulfide858MS, RT812,3-Dihydro-2-n- furan-3-one1413MS, RT37n-HexanolU859MS, RT, A61MS, RT1413MS, RT38Dimethylthio- phene (isomer)861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT832,3-Dihydro-2-n- furan-3-one1619MS, T40n-HeptanalA881MS, RT, Afuran-3-one1619MS, T413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT		1-ol					(isomer)			
35Dimethylthio- phene (isomer)855MS, RT1611MS, RT36Allyl propyl sulfide858MS, RT812,3-Dihydro-2-n- furan-3-one1413MS, RT37n-HexanolU859MS, RT, A phene (isomer)861MS, RT812,3-Dihydro-2-n- furan-3-one1413MS, RT38Dimethylthio- phene (isomer)861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT832,3-Dihydro-2-n- furan-3-one1619MS, T40n-HeptanalA881MS, RT, A furan-3-onefuran-3-one1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT85Dibhenvlacetylene>1700MS, T85Dibhenvlacetylene>1700MS, T	34	2-Furyl alcohol	F	846	MS, RT, A	80	Propenyl propyl		1317	MS RT
phene (isomer)858MS, RT(isomer)36Allyl propyl sulfide858MS, RT812,3-Dihydro-2-n- hexyl-5-methyl-1413MS, RT37n-HexanolU859MS, RT, Ahexyl-5-methyl- furan-3-one1413MS, RT37n-HexanolU859MS, RT, Afuran-3-one1417MS, RT38Dimethylthio- phene (isomer)861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT832,3-Dihydro-2-n- octyl-5-methyl-1619MS, T40n-HeptanalA881MS, RT, Afuran-3-one1619MS, T413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT85Diphenvlacetylene>1700MS, T85Diphenvlacetylene>1700MS, T	35	Dimethylthio-		855	MS, RT	00	trisulfide		1011	100, 101
36Allyl propyl sulfide858MS, RT(13-Dihydro-2-n- hexyl-5-methyl- furan-3-one1413MS, RT37n-HexanolU859MS, RT, Ahexyl-5-methyl- furan-3-one1413MS, RT37n-HexanolU859MS, RT, Afuran-3-one1413MS, RT38Dimethylthio- phene (isomer)861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT832,3-Dihydro-2-n- octyl-5-methyl-1619MS, T40n-HeptanalA881MS, RT, Afuran-3-one1619MS, T413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT85Diphenvlacetylene>1700MS, T85Diphenvlacetylene>1700MS, T		phene (isomer)					(isomer)			
sulfide1410MB, RT37 $n$ -HexanolU859MS, RT, Ahexyl-5-methyl- furan-3-one38Dimethylthio- phene (isomer)861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT832,3-Dihydro- $2 \cdot n$ - octyl-5-methyl-1619MS, T40 $n$ -HeptanalA881MS, RT, Afuran-3-one1619MS, T413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT85Diphene85Diphenylacetylene>1700MS, T	36	Allyl propyl		858	MS, RT	81	2 3-Dihydro-2-n-		1413	MS RT
37n-HexanolU859MS, RT, Afuran-3-one38Dimethylthio- phene (isomer)861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT832,3-Dihydro-2-n- octyl-5-methyl- furan-3-one1619MS, T40n-HeptanalA881MS, RT, A stringoctyl-5-methyl- furan-3-one1681MS, RT413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT85Diphenvlacetylene>1700MS, T100100100100		sulfide				01	hexyl-5-methyl-		1410	10, 101
38Dimethylthio- phene (isomer)861MS, RT822-Tridecanone1477MS, RT392-Heptanone871MS, RT832,3-Dihydro-2-n- octyl-5-methyl- furan-3-one1619MS, T40n-HeptanalA881MS, RT, A string0000413,4-Dimethylthio- phene887MS, RT842-Pentadecanone1681MS, RT85Diphenvlacetylene>1700MS, T000	37	<i>n</i> -Hexanol	U	859	MS, RT, A		furan-3-one			
phene (isomer)871MS, RT832,3-Dihydro-2-n-1619MS, T392-Heptanone871MS, RToctyl-5-methyl-40n-HeptanalA881MS, RT, Afuran-3-one413,4-Dimethylthio-887MS, RT842-Pentadecanone1681MS, RTphene85Diphenvlacetylene>1700MS, T	38	Dimethylthio-		861	MS, RT	82	2-Tridecanone		1477	MS RT
392-Heptanone871MS, RT1010MB, T40n-HeptanalA881MS, RT, Aoctyl-5-methyl-413,4-Dimethylthio-887MS, RT842-Pentadecanone1681MS, RTphene85Diphenvlacetylene>1700MS, T		phene (isomer)				83	2.3-Dihvdro-2-n-		1619	MS T
40 n-Heptanal A 881 MS, RT, A furan-3-one   41 3,4-Dimethylthio- 887 MS, RT 84 2-Pentadecanone 1681 MS, RT   phene 85 Diphenvlacetylene >1700 MS, T	39	2-Heptanone		871	MS, RT	00	octyl-5-methyl-		1010	, 1
41 3,4-Dimethylthio- phene 887 MS, RT 84 2-Pentadecanone 1681 MS, RT 85 Diphenylacetylene >1700 MS T	40	<i>n</i> -Heptanal	Α	881	MS, RT, A		furan-3-one			
phene 85 Diphenvlacetylene >1700 MS T	41	3,4-Dimethylthio-		887	MS, RT	84	2-Pentadecanone		1681	MS. RT
		phene				85	Diphenylacetylene		>1700	MS. T

<sup>a</sup> Supplier: F = Fluka, M = Merck, U = Union Chimique Belge; A = Aldrich; R = Riedel-de Haen; S = Schuchardt. <sup>b</sup> Retention index on OV 1 between C<sub>6</sub> and C<sub>17</sub> with linear temperature programming (Rasquinho, 1965). <sup>c</sup> Previously identified by other workers. <sup>d</sup> MS, mass spectrometry; RT, retention index; A, comparison with authentic compound. <sup>e</sup> Solvent peaks. <sup>f</sup> The steam distillation was carried out twice. This component was only present in one steam distillate.

chromatogram of steam-distilled leek oil, analyzed on OV 1, is shown in Figure 3. The volatile constituents were identified by comparing their mass spectra and retention times with those of reference materials. The compounds identified are presented in Table I and their peak numbers correspond to the numbers in Figure 3.

A weak lachrymatory factor is present in leek. It is noticed a few minutes after the leek tissue has been cut, and proved by the presence of *n*-propanal (peak 4) and 2-methylpent-2-enal (peak 31), two decomposition products from thiopropanal sulfoxide, the latter which is now proven to be the lachrymatory substance in *Alliaceae* (Brodnitz and Pascale, 1971).

Sulfur-containing components are the main flavoring constituents of leek essential oil, because of the high concentration in which they occur and their very low threshold values. Mainly saturated and unsaturated mono-, di-, and trisulfides are found in the analysis mixture besides some thiophene derivatives and thiols. Typical for steam-distillation procedures are the presence of alkanals and furan derivatives which are formed by Maillard reactions between amino acids and reducing sugars. A lot of n-alkanals occur in the mixture besides the peaks 30 and 34 which are present in large concentration.

No explanation is found for the presence of the furan-3-one derivatives, peaks 81 and 83, in such large concentrations. Their formation is not clear. They could be formed via the respective C<sub>11</sub>- and C<sub>13</sub>-hydroxy ketones as is suggested by Boelens et al. (1971). Probably furan-3-ones can also be formed as an effect of heating upon sugars since Tonsbeek et al. (1968) isolated 4-hydroxy-5-methyl-2,3-dihydrofuran-3-one and its 2-methyl derivative from heated beef extract. The former product was due to reaction of ribose 5-phosphate and taurin or pyrrolidone carboxylic acid.

Alkanals and alcohols are difficult to recognize by mass spectrometry because most of them show no molecular ion

Table II. Mass Spec	tral Data of Constituents of Leek Oil
Allyl methyl sulfide	41 (100), 88 (91), 73 (62), 39 (52), 43 (48), 45 (42), 56 (38), 47 (21)
Dimethyl disulfide	94 (100), 79 (48), 45 (41), 46 (25), 47 (16), 61 (13), 48 (9), 64 (8).
	96 (8)
2-Methylpentanal	43 (100), 58 (84), 41 (30), 29 (26), 27 (24), 71 (12), 100 (3)
n-Hexanal	43 (100), 44 (84), 41 (58), 56 (56), 45 (50), 72 (40), 57 (32), 55 (22),
	100 (16), 82 (6)
Methylpyrazine	94 (100), 67 (50), 39 (21), 40 (20), 53 (20), 95 (6), 52 (6), 79 (5)
2-Furaldehyde	96 (100), 95 (89), 39 (75), 29 (23), 38 (17), 94 (15), 67 (11)
2-Methylpent-2-	41 (100), 98 (53), 39 (45), 69 (38),
enal	27 (29), 55 (26), 43 (19), 29 (19), 83 (14)
2-Furyl alcohol	98(100), 97(54), 41(57), 81(50), 39(50), 53(39), 42(31), 69(24)
	70(22)
<i>n</i> -Heptanal	70 (100), 44 (97), 43 (91), 42 (89),
	55 (71), 41 (63), 57 (61), 81 (31),
0 4 D' (1 )(1 )	86 (20), 96 (9)
3,4-Dimethylthio-	111(100), 112(76), 39(63), 45
2 6 Dimethul	(20), 77 (10), 97 (0) 108 (100) 49 (65) 40 (28) 20 (21)
2,0-Dimethyl-	100(100), 42(00), 40(30), 39(31), 100(9) 91(6) 67(6)
Methyl propyl	80(100) 199(75) 43(70) 41(64)
disulfide	45(30) $47(16)$ $39(16)$ $64(13)$
Dimethyl trisulfide	126(100), 45(58), 79(57), 47(49),
	46 (24), 64 (21), 48 (19), 111 (16), 04 (10) 108 (12) (12) (12)
9.Fthyl furyl	94(10), 128(13), 01(13) 05(100) 194(98) 20(91) 97(15)
ketone	35(100), 124(20), 35(21), 27(10), 41(14), 99(14)
Pyrrolecarbox-	95(100) 94(66) 66(49) 39(46)
aldehvde	29(46), 41(40), 43(38), 45(26)
Dipropyl disulfide	43 (100), 150 (29), 41 (24), 108
	(19), 66 (8), 39 (7), 74 (5), 47 (5), 45 (5)
2-Phenylethanol	91 (100), 92 (57), 122 (23), 65 (12),
1 2-Dimethoxy.	138(100), 123(00), 95(37), 77
henzene	(32) $41$ $(20)$ $65$ $(18)$ $52$ $(17)$ $51$
benzene	13
Methyl propyl	43(100), 154(96), 47(70), 112
trisulfide	(63), 41 (60), 45 (44), 64 (38), 79 (30)
2.5-Dihvdro-3.4-	128 (100), 85 (56), 67 (52), 39 (50).
dimethylthio-	41 (46), 55 (36), 81 (33), 45 (31),
phen-2-one	99 (27), 73 (23), 59 (19), 83 (14),
Benzothiazole	100 (13) 135 (100), 108 (32), 69 (15),
Due menuel museumel	45 (12), 82 (7), 63 (6)
trigulfido	41 (100), 74 (91), 180 (58), 45 (56) 106 (40) 115 (22) 116
unsunnde	(30), 83 (30), 39 (28), 47 (12),
2,3-Dinydro-2-n-	30 (100), 111 (53), 41 (23), 43 (18), 68 (19), 20 (19) 55 (10), 100 (7).
furan-3-ono	00(13), 39(13), 55(12), 182(7), 00(7), 85(6), 119(4)
2 3-Dihydro-5-	98(100) 111(56) 41(10) 43(14)
methyl-2-n-	68 (11) 39 (10) 55 (8) 99 (6) 85
octvlfuran-3-one	(5), 112 (4), 210 (3)
Diphenylacetylene	178 (100), 76 (21), 176 (20), 179
	(17), 152 (15), 89 (15), 88 (13)

(see also mass spectral data). Alcohols, however, show tailing peaks in the chromatogram on the apolar phase OV 1 as is clearly illustrated by the peaks 5, 14, and 17.

However, the 2-methyl ketones are very easily recognized by their base peak m/e 58 and characteristic spectral pattern.

As another result of heating, the formation of large amounts of 3,4-dimethylthiophene, in addition to two other positional isomers has to be mentioned. These products can be formed by heating alkenyl disulfides with a loss of hydrogen sulfide as is unambiguously proven by Boelens

Table III. Odor Description of Figure 4

1.	Rotten	Ma	52.	Pungent	Μ
2	Rotten	S	53.	Sweet	Μ
3.	Aldehvde	ñ	54.	Fresh	W
4	Diethyl ether	M	55.	Onion	W
5	Brussels sprouts	S	56	Allium sativum	M
6	Onion	й	57	Violet	w
7	Alcoholic	w	58	Cabhage blossom	s
8	Buttory	Ň	59	Burnt potatoes	ŝ
a.	Fresh out look	S	60	Allium sativum	м
10	Pleasant	м	61	Burnt potatoes	S
11	Buttory	M	62	Fruity	ŝ
19	Muety	M	63	Aromatic	м
12.	Aldohydo	W .	64	Moldy	VS
11	Inplessent	9	65	Poss	ŝ
15	Allium ontinum	w	66	Pubbory	ŝ
10.	Potton	VQ	67	Placent	M
17	Drwiding	M	69	Look	S
10	Alecholic	111	60.	Leek	M
10.	Anconolic	¥¥ 117	70	Look amost	C IVI
19.	Alline	VV 317	70.	Leek, Sweet	g
20.	Allium satibum	VV 117	71.	Aromatic Look nungont	0
21.	Buttery	Ŵ	72.	Cueek, pungent	M
22.	Burnt rubber	5	73.	Green carrots	IVI C
23.	Fruity	IVI IVI	74.	Musty	3
24.	Allium sativum	W	75.	Moth balls	M
25.	Gaseous	W	76.	Moldy	S
26.	Nutty	W	77.	Unpleasant	M
27.	Aromatic	S	78.	Onion, irritating	M
28.	Pleasant, aldehyde	S	79.	Onion	S
29.	Burnt	M	80.	Tomatoes	W
30.	Rotten	W	81.	Rubbery	M
31.	Allium sativum	S	82.	Onion, irritating	S
32.	Flowers	М	83.	Onion	M
33.	Green, pleasant	S	84.	Caramel, hay	S
34.	Burnt potatoes	S	85.	Menthol	S
35.	Peas	Μ	86.	Boiled leek	W
36.	Sweet	W	87.	Onion, irritating	$\mathbf{s}$
37.	Caramel	М	88.	Carrots, pleasant	$\mathbf{S}$
38.	Burnt potatoes	vs	89.	Musty	W
39.	Musty, pungent	S	90.	Rubbery	Μ
40.	Burnt potatoes	vs	91.	Leek, irritating	W
41.	Leek	Μ	92.	Pleasant, meaty	Μ
42.	Allium sativum	Μ	93.	Almonds	W
43.	Allium sativum, fried	S	94.	Steam distillate leek	Μ
44.	Leek, irritating	VS	95.	Pleasant	Μ
45.	Onion	s	96.	Green carrots	M
46.	Onion, irritating	Ŵ	97	Steam distillate	S
47	Onion, irritating	s	5	leek	-
48	Cabbage	ŝ	98.	Leek	W
49	Leek irritating	м	99	Steam distillate	M
50	Spinach	S	50.	leek	-/-
51	Sweet, alcoholic	м	100	Meaty, pleasant	М
а – .	Odor intonsiter. 117		 м	modium, E - at	
-	Odor Intensity: W	- weak:	141 =	measure $\mathfrak{S} = \mathfrak{S}(\mathfrak{r}\mathfrak{O}\mathfrak{r})$	12.

<sup>a</sup> Odor intensity: W = weak; M = medium; S = strong; V = very.

and Brandsma (1972). Peak 78 disappeared during GC-MS coupling and is also not found when taking the aromagram, indicating the fact that there is still some thermal decomposition during the analysis.

Mass spectral data of the identified components are summarized in Table II. The peaks are given with their intensities (in parentheses) relative to that of the base peak (100%).

Four aromagrams were taken by sniffing the eluting products from the column at the collector exit of a microthermal conductivity cell. Three trained persons were involved in running the aromagrams. The mean results of these analyses are illustrated in Figure 4 and Table III where an odor description is given for each peak together with its odor strength. The numbers of Table III correspond to the peak numbers of Figure 4.

The aromagram indicates 11 leek-like odors in addition to a lot of onion odors. Sometimes it is very difficult to distinguish both. Components which are believed to contribute to the specific leek flavor are propanethiol, allyl





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methyl disulfide, methyl propyl disulfide, dipropyl disulfide, methyl propyl trisulfide, and some unidentified products.

From Ip 1500 it becomes difficult to distinguish odors at all, for a specific leeklike odor lasts around the collector exit.

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#### LITERATURE CITED

Bernhard, R. A., J. Food Sci. 33, 298 (1968).

- Boelens, H., Brandsma, L., Recl. Trav. Chim. Pays-Bas 91, 141 (1972)
- Boelens, M., de Valois, P. J., Wobben, H. J., van der Gen, A., J. Agric. Food Chem. 19, 984 (1971).
- Bouche, J., Verzele, M., J. Gas Chromatogr. 6, 501 (1968).
- Brodnitz, M. H., Pascale, J. V., J. Agric. Food Chem. 19, 269 (1971).
- Brodnitz, M. H., Pollock, C. L., Vallon, P. P., J. Agric. Food Chem. 17, 760 (1969).

- Calvallito, C. J., Bailey, J. H., J. Am. Chem. Soc. 66, 1944 (1950). Carson, J. F., in "Chemistry and Physiology of Flavors", Avi, Westport, Conn., 1967, p 390.
- Dembele, S., Dubois, P., Ann. Technol. Agric. 22, 121 (1973).
- Johnson, A. E., Nursten, H. E., Williams, A. A., Chem. Ind. (London), 556 (May 1971).
- Macleod, A. J., Flavour Ind. (Oct 1970).
- Rasquinho, L. M. A., J. Gas Chromatogr., 340 (1965).
- Schwimmer, S., Friedmann, M., Flavour Ind. (March 1972).
- Schwimmer, S., Guadagni, D. G., J. Food Sci. 33, 183 (1968). Schwimmer, S., Weston, W. J., J. Agric. Food Chem. 9, 301 (1961).
- Stoll, A., Seebeck, E., Helv. Chim. Acta 31, 189 (1948).
- Stoll, A., Seebeck, E., Helv. Chim. Acta 32, 197 (1949a).
- Stoll, A., Seebeck, E., Helv. Chim. Acta 32, 866 (1949b).
- Stoll, A., Seebeck, E., Adv. Enzymol. 11, 377 (1951).
- Tonsbeek, Ch. H. Th., Koenders, E. B., van den Zijden, A. S. M., Losekoot, J. A., J. Agric. Food Chem. 16, 1016 (1968).

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# Effects of Storage Temperature and Container Lining on Some Quality **Attributes of Papaya Nectar**

John E. Brekke,\* Catherine G. Cavaletto, T. O. M. Nakayama, and Robert H. Suehisa

Papaya nectar in plain tin- and enamel-lined cans was stored at 55, 75, and 100°F for 1 year. Samples were analyzed periodically to determine quality of the product. Corrosion of the tin lining was most rapid at 100°F; after 1 year, the tin content of the nectar was 400 ppm. Iron content increased more rapidly in enamel- than in tin-lined cans. Acid hydrolysis of sucrose was a first-order reaction. The samples stored at 100°F were dark and of poor flavor. Samples at 55°F were virtually unchanged after a year. Type of can lining was not as important as low temperature for quality retention in storage.

Papaya is marketed chiefly as fresh fruit, but interest in its processed products such as canned nectar has increased. The nectar is made from papaya puree, water. sugar, and citric acid. The stability of canned papaya nectar, as of any processed food product, is important to the producer. Storage temperature and type of can lining generally affect the stability of a product.

Enamel-lined cans have been recommended for papaya nectar because of the corrosiveness of papava products on plain, tin-lined cans (Lloyd, 1972). A positive correlation between nitrate content and rate of detinning has been established (Farrow et al., 1970; Board, 1973). Extensive detinning can cause container failure and may affect product flavor. Tolerance of tin in high acid foods has not been established in the U.S., but limitations have been imposed on the amount of SnCl<sub>2</sub> that can be added to some canned vegetables. No toxic effects were shown from ingestion of fruit juices that contained 730 ppm of Sn (Benoy et al., 1971).

The characteristic carotenoid color of products is often, but not always, retained better in tin- than in enamel-lined cans (Payumo et al., 1968; Lloyd, 1972). The rate of darkening of fruit products has been associated with hydrolysis of sucrose to reducing sugars (Stadtman, 1948; Hodge, 1953; Loeffler, 1941), which then enter the Maillard reaction or other reactions producing brown pigments. Inversion of sucrose in a fruit juice product can thus be viewed as a phenomenon associated with the early stages of quality deterioration.

We report herein our investigation of the effects of storage temperature and type of can lining on quality and composition of papaya nectar.

#### MATERIALS AND METHODS

Papaya nectar (13.4° Brix; pH 3.5) was prepared from papaya puree (25%), water, and sugar (10%). The puree had been prepared by the method of Brekke et al. (1972) and stored at 0°F for 1 year.

Half of the nectar (about 150 lb) was then canned in enamel-lined cans (G enamel; no side seam spray, 0.25 lb electrolytic inside, 1.00 lb electrolytic outside, MR 85 lb tin plate). The remaining 150 lb was canned in differential bright tin plate (1.00 lb inside and 0.5 lb outside). After vacuum sealing, the cans were spin-cooked in flowing steam for 3 min (Wang and Ross, 1965), then spin-cooled in water

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