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Unconventional Proteins as Aroma Precursors. Chemical Analysis of the Volatile Compounds in Unheated and Heated Rapeseed Protein Model Systems

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Model samples containing rapeseed protein were analyzed by gas chromatography and mass spectrometry. The headspace of samples, unheated and heated, with or without addition of fat and starch, was investigated. Over 100 compounds were identified, representing aliphatic hydrocarbons, alcohols, aldehydes, ketones, nitriles, and furan derivatives, and sulfur-containing compounds. Determinations were made of the absolute concentrations in the headspace gas of about 70 of the compounds judged to be of potential interest from the point of view of aroma. On heating of the samples, the concentrations of volatiles generally increased and new compounds were detected. Of importance for the aroma of the heated samples is the presence of aliphatic aldehydes, sulfur compounds, furans, and probably also nitrogen-containing compounds.

Rapeseed is an important oil crop in Northern countries. The protein part (25%) has such an amino acid composition that rapeseed cakes have been studied to find out whether it is possible to obtain a rapeseed protein concentrate with nutritional and functional properties adequate for human consumption (Appelqvist and Ohlson, 1972; Ohlson, 1973; Anjou et al., 1975). An important part of the study is to analyze the aroma properties of the rapeseed protein concentrate. A search of the literature failed to reveal any chemical or sensorical investigations of the properties of the aroma of rapeseed protein concentrate. The chemical composition of the headspace gas of unheated and heated samples containing different proteins has been determined earlier by Persson et al. (Persson and von Sydow, 1973, 1974a,b; Persson et al., 1973a,b) and by Qvist and von Sydow (1974).

In this part of the investigation we present results of gas chromatographic and mass spectrometric analyses of samples containing rapeseed protein. The analytical techniques employed were the same as those used by Qvist and von Sydow (1974).

EXPERIMENTAL SECTION

Materials and Processing. The rapeseed protein concentrate analyzed was produced by special solvent extraction of dehulled, detoxified, and prepressed rapeseed samples (glucosinolate content < 0.5 mg/g), obtained from AB Karlshamns Oljefabriker and Alfa-Laval AB. The extraction was carried out during 12 h in batches of 0.5 kg with redistilled isopentane in a Soxhlet extractor fitted with a distillation column and a reflux condensor. The residue was dried under vacuum in a rotating evaporator in order to remove residual solvents. In this way a rapeseed protein concentrate containing 65% (N × 6.25) protein, 0.77% fat, 2.3% water, and with an ash content of 6.9% was obtained.

For all experiments involving quantitative determinations one homogenous lot of the protein raw material was used. The fat used was minced pork back fat. The carbohydrate used was commercial potato flour (80% starch and 20% H_2O from Swedish Starch Producers Association). The iodized salt was commercial table salt (from K. N. Z. Henzelo, Holland). The water used was distilled and filtered through activated charcoal.

A standard protein raw material was made by mixing the protein with water so that a sample containing 21% protein (as in the meat used by Persson and von Sydow, 1973) was obtained. This mixture is called "protein–H_2O" in Table I. With this mixture as basic material the formulations presented in Table I were prepared. The cans used for experiments involving quantitative measurements were deep drawn from electrolytical tin plate (1.00/0.50 lb per base box (bb)), 73 × 28 mm in size, holding 80 g of

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Table I. Formulations and Treatments of the Model Samples Analyzed

Model no.	Treatment	Formulation, % Protein-H_2O	H_2O	NaCl	Starch	Fat
I	Unheated	79.3	20	0.7		
II	Heated (F_c = 28)	79.3	20	0.7		
III	Heated (F_c = 28)	61.3	20	0.7	5	13

material. The processing was carried out in a retort at 121 °C until an F_c value of 28 was reached, which occurred after 35–40 min. For further details about the retort processing cf. Qvist and von Sydow (1974).

Concentrates of Volatiles. For mass spectrometric identification (see below) it was necessary to concentrate the components in the headspace gas. A slurry was prepared by mixing 1.2 kg of protein model sample and 1.2 l. of carbon-filtered distilled water. The protein model sample was obtained from a number of cans (see Materials and Processing section) containing one of the formulations described in Table I. Volatiles from the protein model slurry were concentrated by low-temperature distillation according to Forss et al. (1967) at 1 kPa and 20 °C after initial degassing with the slurry stirred at 0 °C. A mechanical stirrer was used. The distillation column temperature was 6 °C and the volatiles were condensed in a cold trap cooled by liquid nitrogen. Several batches were distilled until about 1 l. of concentrate was obtained. This concentrate was redistilled, giving about 100 ml of distillate. This technique is suitable for qualitative but not for quantitative determinations.

Headspace Sampling Technique. The sampling technique described by von Sydow et al. (1970) was used with the following modifications: 75 g from one can was homogenized for 5 min at 0 °C with 75 ml of carbon filtered distilled water in a 750-ml flask with a stainless steel lid. This flask was also used as the headspace sampling flask. This avoided transfer of the material from a homogenizer flask to a headspace flask with a better reproducibility as a result. To obtain equilibrium the flask was rotated for 45 min in an inclined position in a water bath at 25 ± 0.1 °C. A 500-ml sample of headspace gas was conveyed to the cold trap, which contained 70 mesh glass beads in the lower U-shaped part. For analysis of the distillates (25-ml samples) a 200-ml flask and a 150-ml headspace sample were used.

When analyzing the sulfur compounds with a Melpar flame photometric detector a sampling system made entirely from glass and PTFE was used. After homogenization, the sample was transferred to a 750-ml headspace flask with a glass lid. The volatiles were transferred to the trap, as described. All connections in the valve oven were made of PTFE tubing and the eight-port switching valve was substituted with an eight-port FEP/PTFE valve (Valco VSV-8-CI). One of the stainless steel cold traps was replaced by a glass trap, otherwise having the same configuration as the original one. This cold trap was used in the way described by von Sydow et al. (1970). The sample size was 400 ml and the valve oven was maintained at 60 °C. The other cold trap was replaced by a 2-ml sample loop made of PTFE tubing (3.2 mm o.d. × 1.6 mm i.d.). This was maintained at room temperature and was used for analyzing hydrogen sulfide, methanethiol, and dimethyl sulfide which were present in such large amounts in some samples that the responses exceeded the working range of the FPD.

Gas Chromatography. The equipment consisted of a Perkin-Elmer Model 990 gas chromatograph with flame ionization detector and, when analyzing the sulfur compounds, a Perkin-Elmer Model 900 gas chromatograph with a Melpar sulfur-specific flame photometric detector (see below). The precolumn concentration equipment was connected to the chromatograph. When using the flame ionization detector the headspace gas was analyzed on two open tubular columns, a 0.76 mm i.d. × 170 m stainless steel tube coated with SF96/Igepal CO 880 (95/5) and a 0.76 mm i.d. × 170 m stainless steel tube coated with UCON 50 HB 2000. The temperature was programmed from 20 to 140 °C and from 35 to 135 °C, respectively, at 2 °C/min after an initial isothermal period of 6 min. The helium carrier gas flow rate was 12 ml/min.

For the absolute quantitative determinations of the components in the headspace gas the FID response factors were determined for the various compounds by experiment and from literature data (Dietz, 1967; Kaiser, 1962). The peak areas were measured by a Perkin-Elmer Processor (PEP-1). Using the absolute concentration of thiophene, determined in the headspace gas according to the method described below, and the responce factors, the absolute concentrations of the different compounds in the headspace gas were determined by using the peak areas (Persson and von Sydow, 1973).

When analyzing the sulfur compounds, the sulfur-specific Melpar flame photometric detector (FPD) was used in conjunction with a Perkin-Elmer 900 gas chromatograph, to which the glass–PTFE sampling system (described above) was connected. The column used was a 3.0 mm i.d. × 6.3 m long glass column packed with Chromosorb G (acid washed, DMCS treated, 80–100 mesh) coated with 5% Igepal CA 630 and percolated with 50 ml of 10% didecyl phthalate in acetone (Jansen et al., 1971). The column end and the detector were connected with a PTFE tube (0.8 mm i.d. × 1.6 mm o.d.). For the 2- and 400-ml samples the temperature was programmed from 20 to 120 °C at 10 and 4 °C/min, respectively, after an initial isothermal period of 10 min. The helium carrier gas flow was 45 ml/min. The procedure with permeation tubes calibrated gravimetrically was used for calibrating the FPD with various sulfur compounds (O'Keefe and Ortman, 1966; Scaringelli et al., 1970; Stevens et al., 1971).

Mass Spectrometry. The samples were analyzed in a combined gas chromatograph–mass spectrometer, Perkin-Elmer 990 (FID)–LKB 9000 (TIC detector) with parallel detection in the gas chromatograph and the mass spectrometer. The headspace precolumn equipment described above was connected to the gas chromatograph. The gas chromatographic separations were made on the SF96 open tubular column and on the UCON open tubular column (see above). Mass spectra were recorded at 70 eV. The separator temperature was 200 °C and the ion source temperature was 270 °C. The compounds in the samples were identified by comparison with our own reference spectra or spectra given in the literature.

RESULTS AND DISCUSSION

Three samples according to Table I were investigated. The headspace gas of a low-temperature distillate was analyzed in a combined gas chromatograph–mass spectrometer in order to obtain as much qualitative information as possible. In this way we identified or obtained structural information of altogether over 110 compounds, some of which are presented in Table II. Compounds not included in Table II consisted of 20 saturated hydrocarbons, 5 aliphatic esters, 4 nitrogen-containing compounds, 4 ketones, and 4 furan derivatives, most of them

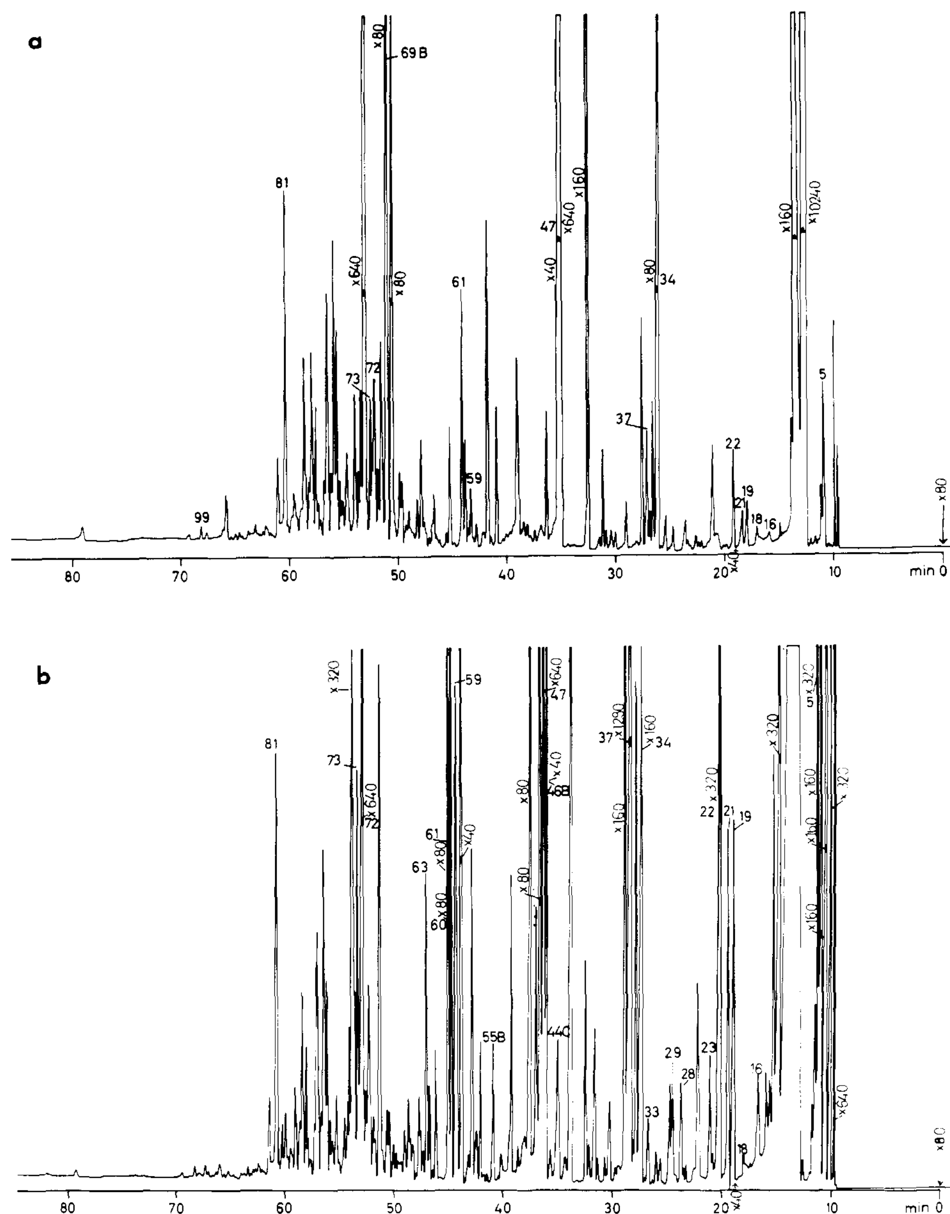

Figure 1. Gas chromatograms of 500-ml headspace samples of rapeseed protein concentrate in water: (a) unheated; (b) heated at 121 °C to $F_c = 28$; SF 96 column; peak numbers refer to Table II.

with only partially known structures. The saturated hydrocarbons are generally uninteresting because of their high odor thresholds and the other compounds were present in such low concentrations that they presumably played little or no role in these samples. The quantitative determinations were made by straightforward analyses of the headspace gas of the three samples.

The chromatographic separations and quantifications were carried out with an SF96 column and a UCON column with FID's for most of the compounds and an Igepal CA 630–didecyl phthalate column with an S-FPD for those containing sulfur. Typical examples of chromatograms obtained from analysis with FID with an SF96 column and a UCON column are given in Figures 1 and 2, respectively. Mass spectrometry was used again to ascertain the identity of the various eluted compounds. The three thiols (marked *f* in Table II) were not identified by mass spectrometry. Hydrogen sulfide was absorbed and masked by air, and the other two were broken down in the analyzing system. The compounds marked *f* were identified from retention data obtained in our own experiments with known chemicals.

The absolute concentrations were determined as described in the Experimental Section and by Persson and von Sydow (1973). The data are presented in Table II. Each value is the mean of three determinations. The relative standard deviation of different components normally varies between 5 and 10%, and in some cases as much as 25%. In a few cases, where the concentration is very low or the compound extremely volatile, the range of variation may be wider. Examples of compounds having a wide variation are 2-decanone and hydrogen sulfide. 2-Decanone is present in low concentrations and small differences between the different runs result in large percentual deviation (50%). This is partly due to the difficulties of measuring low concentrations. Hydrogen sulfide has a large standard deviation due to losses during sample preparation because of the high volatility. However, despite the relative high standard deviation for certain components, significant differences between the samples have been obtained at significance levels ≤0.05.

The influence of interfering substances on the peak areas of the individual compounds was minimized by analyzing the samples on the two different columns. Carbonyl sulfide

Table II. Absolute Concentrations (Parts per Billion, v/v) of Volatile Compounds in the Headspace Gas of Samples of Rapeseed Protein[a]

Compound	Peak no., see Fig 1 and 2	Rapeseed protein: Unheated	Rapeseed protein: Heated in H_2O[b]	Rapeseed protein: Heated with fat, starch, and H_2O[b]	Odor thresholds, ppb in air (v/v)	References
Methanol	7C	21	900	960	100 000	Leonardos et al. (1969)
Ethanol	117		14	14	10 000	Leonardos et al. (1969)
1-Butanol	118		41	4.9	303	Pliška and Reisenauer (1961)
1-Pentanol	130		1.6		255	Pliška and Reisenauer (1961)
1-Penten-3-ol	40B		1.2	0.6	400[d]	Buttery et al. (1971)
Ethanal	5	200	1280	1100	210	Leonardos et al. (1969)
Propanal	9	190	460	350	95[d]	Guadagni et al. (1963)
n-Butanal	19	21	92	66	9[d]	Guadagni et al. (1963)
n-Pentanal	34	95	290	230	12[d]	Guadagni et al. (1963)
n-Hexanal	47	600	1100	650	4.5[d]	Guadagni et al. (1963)
n-Heptanal	61	24	53	24	3[d]	Guadagni et al. (1963)
n-Octanal	73	8.4	24	7.8	0.7[d]	Guadagni et al. (1963)
n-Nonanal	81	15	31	9.8	1[d]	Guadagni et al. (1963)
n-Decanal	99	1.1	1.2	0.4	0.1[d] 0.2	Guadagni et al. (1963) Teranishi (1967)
2-Methylpropanal	16	3.0	60	53	1[d]	Guadagni et al. (1972)
2-Methylbutanal	29	1.3	14	17		
3-Methylbutanal	28	2.5	20	38	0.2[d]	Guadagni et al. (1972)
2-Methyl-2-butenal			Trace	Trace		
2-Methyl-2-pentenal			Trace			
Benzaldehyde	70	0.3	5.7	4.1	9 960	Pliška and Reisenauer (1961)
Furfural	123	5.3	7.9	4.0	3 000	Buttery et al. (1971)
2-Propanone	10	28	460	400	100 000	Leonardos et al. (1969)
2-Butanone	21	8.0	38	27	10 000	Leonardos et al. (1969)
2-Pentanone	33	1.6	6.1	4.1		
2-Hexanone	46	0.5	3.5	1.7		
2-Heptanone	59	6.0	24	9.2	217	Weurman (1963)
2-Octanone	120	0.7	5.5	3.2		
2-Nonanone	125	0.4	1.7	0.4		
2-Decanone	98	0.1	0.9	0.6		
3-Heptanone		Trace	Trace			
3-Octanone	119	2.5	2.8	0.4		
3-Methyl-2-pentanone	42B	0.7	1.9	0.8		
4-Methyl-2-pentanone	41	0.8	2.2	0.7		
2-Methyl-3-pentanone			Trace	Trace		
2,6-Dimethyl-4-heptanone[c]			Trace			
3-Octen-2-one	124		2.0	1.5		
6-Methyl-5-hepten-2-one	121	8.3	12	3.2	50[d]	Buttery et al. (1971)
3,5-Octadien-2-one	126	2.9	1.7	1.3		
3,5-Octadien-2-one	127	9.5	9.0	6.3		
Furan	11		330	190	4 500[d]	Mulders (1973)
2-Methylfuran	22	27	440	170	3 500[d]	Mulders (1973)
2-Ethylfuran	37	13	1370	320	8 000[e]	Evans et al. (1971)
2-Propylfuran	46B		30	2.6	6 000[e]	Evans et al. (1971)
2-Butylfuran	60	1.2	32	2.6	10 000[e]	Evans et al. (1971)
2-Pentylfuran	72	13	340	26	2 000[e] 6[d]	Evans et al. (1971) Buttery et al. (1971)
3-Methylfuran	23	0.5	17	5.7		
2,5-Dimethylfuran			Trace			
2-Methyl-5-ethylfuran	48		17	2.6		
2-Methyl-5-propylfuran	59		16	1.6		
2-Methyltetrahydrofuran	30		0.5	0.3		
2-Vinylfuran			Trace		1 000[e]	Evans et al. (1971)
A butenylfuran	55B	0.4	12	1.5		
A butenylfuran	63		23	3.3		
Carbonyl sulfide	(S1)	+	+	+		
Hydrogen sulfide[f]	(S2)	190	1550	490	4.7	Leonardos et al. (1969)
Methanethiol[f]	(S3)	0.8	240	130	2.1	Leonardos et al. (1969)
Ethanethiol[f]	(S4)		2.5	1.1	1.0	Leonardos et al. (1969)
Dimethyl sulfide	(S5)	2.7	830	750	1.0	Leonardos et al. (1969)
Carbon disulfide	(S6)		28	24	210	Leonardos et al. (1969)
Ethylene sulfide	(S9)		5.1	5.0		
Methyl ethyl sulfide	(S10)		1.9	0.8		
Methyl isopropyl sulfide	(S13)		5.0			
Dimethyl disulfide	(S21)	2.2	4.0	2.9	7.6	Wilby (1969)
Methyl pentyl sulfide			Trace			
Thiophene	(S16)		5.0	1.8		
2-Methylthiophene	(S22)		1.9	0.6		
3-Methylthiophene	(S23)		0.8			
2-Ethylthiophene	(S24)		5.7	0.7		
3,5-Dimethyl-1,2,4-trithiolane[c,f]	(S25)		11	8.2		

Table II (*Continued*)

Compound	Peak no., see Fig 1 and 2	Rapeseed protein: Un-heated	Heated in H_2O^b	Heated with fat, starch, and H_2O^b	Odor thresholds, ppb in air (v/v)	References
3-Butenyl isothiocyanate	69B	93		1.4		
4-Pentenyl isothiocyanate	129	8.4				
n-Hexylnitrile	128	1.6	1.8	0.5		
n-Heptylnitrile			Trace			
4-Methyl pentanenitrile	122	1.8	4.1	0.8		
A heptylnitrile	65		2.1	0.3		
4-Pentenenitrile	44C	0.3	32	24		
5-Hexenenitrile	56B	1.7	8.4	4.2		
A pentadienenitrile	44		3.2	2.6		

[a] In water heated with and without starch and fat (cf. Table I). [b] At 121 °C to $F_c = 28$. [c] Tentative. [d] Parts per billion in water. [e] Parts per billion in cottonseed oil. [f] Identified by retention data only; all other compounds identified by mass spectrometry.

Figure 2. Gas chromatograms of 500-ml headspace samples of rapeseed protein concentrate in water: (a) unheated; (b) heated at 121 °C to $F_c = 28$; UCON column; peak numbers refer to Table II.

was not determined quantitatively because of difficulties with the permeation tube technique.

Literature data for odor thresholds are given in Table II. The selection of compounds in Table II has been made from the odor point of view. Thus, the odor threshold data and the results from the investigation on canned beef (Persson and von Sydow, 1973, 1974a,b; Persson et al., 1973a,b) were used when selecting the compounds. This does not mean that all compounds are of importance for the odor, but only that they are of potential interest.

From a chemical point of view the following observations can be made. The concentrations of almost all volatiles increase on heating. Also, a number of new compounds are detected in the heated samples as a result of thermally induced reactions. This is particularly true for sulfur compounds which are an interesting group of compounds from the sensory point of view.

The concentrations of the straight-chain aldehydes in the samples are greatly affected by heat. The concentrations of the branched chain aldehydes of low molecular weight increase markedly on heating of the samples, probably as a result of Strecker degradation of the corresponding amino acids. The addition of starch and fat has only a limited effect on the concentrations of aldehydes. This may be the result of two opposite effects: when heated the fat generates some aldehydes (Watanabe and Sato, 1970) and the fat dissolves and retains some of the aldehydes generated through other mechanisms.

Heating increases the concentrations of the alcohols. With the exception of 1-butanol which decreases significantly, the addition of fat and starch has only a minor effect on their concentrations. A large number of ketones were detected and determined. Generally, the concentrations of the ketones increase on heating and the addition of fat reduces the increase, probably due to the solvent effect of the fat. Heat causes a large increase in the concentrations of the furans, but the addition of fat diminishes the concentration, probably by a solvent effect.

Unheated samples contain only a few demonstrable sulfur compounds. Heating results in drastic increase of concentrations and also results in the formation of sulfur compounds not detectable in the unheated samples. Exceptions are the two isothiocyanates which were found in the unheated sample, but not in the heated one. Evidently, these compounds are broken down by the heat treatment. The occurrence of the isothiocyanates in rapeseed has been reviewed by Göbel and Franzke (1975). The addition of fat has only a minor influence on the concentrations of the sulfur compounds.

Several nitriles are found in the samples and the concentrations are increased by heat. The occurrence of these compounds in rapeseed is also included in the review by Göbel and Franzke (1975). In rapeseed both isothiocyanates and nitriles are produced from glucosinolates by hydrolysis with myrosinases.

A comparison with the volatiles from other unconventional proteins (Qvist and von Sydow, 1974) and from heated beef (Persson and von Sydow, 1973) reveals a number of similarities and dissimilarities. Thus, aldehydes are important in all cases and the concentrations in the rapeseed protein samples are similar to those in the soy protein (Promine D) samples. This is true both for the unheated and the heat-treated samples. The important branched chain aldehydes are less abundant in rapeseed protein than in beef, Promine D, and fish protein (EFP 90) but equally abundant as in casein (Sodinol V).

More furans were detected in the rapeseed protein samples than in the samples of beef, Sodinol V, and EFP 90. The concentrations are similar to those found in Promine D except for 2-ethylfuran, 2-butylfuran, and 2-pentylfuran which are more abundant in Promine D.

Hydrogen sulfide, methanethiol, ethanethiol, and ethylene sulfide are present in higher concentrations in the heated beef samples than in the rapeseed protein sample. The concentrations of sulfur-containing compounds in rapeseed protein are similar to those found in Promine D with the exception of methanethiol and dimethyl sulfide, which are more abundant in the rapeseed protein sample. Another exception is thiophene which is present in much lower concentrations in the rapeseed protein sample.

An important difference between rapeseed protein samples and other protein samples, including beef, is the presence of the isothiocyanates and the nitriles in the rapeseed protein samples.

From the odor point of view the following preliminary conclusions can be drawn, based on odor threshold data and experience from samples of heat sterilized beef. The odor of the rapeseed protein samples depends on the presence of low molecular weight straight and branched chain aldehydes and sulfur compounds. Several furan derivatives and nitrogen-containing compounds are probably also important. These matters will be dealt with in greater detail in further contributions.

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LITERATURE CITED

Anjou, K., Fecske, A., Krook, G., Ohlson, R., Swedish Patent 377 652 (1975).
Appelqvist, L. Å., Ohlson, R., "Rapeseed", Elsevier, Amsterdam, 1972.
Buttery, R. G., Seifert, R. M., Guadagni, D. G., Ling, L. C., *J. Agric. Food Chem.* **19**, 524 (1971).
Dietz, W. A., *J. Gas Chromatogr.* **5**, 68 (1967).
Evans, C. D., Moser, H. A., List, G. R., *J. Am. Oil Chem. Soc.* **48**, 495 (1971).
Forss, D. A., Jacobsen, V. M., Ramshaw, E. H., *J. Agric. Food Chem.* **15**, 1104 (1967).
Guadagni, D. G., Buttery, R. G., Okano, S., *J. Sci. Food Agric.* **14**, 761 (1963).
Guadagni, D. G., Buttery, R. G., Turnbaugh, J. G., *J. Sci. Food Agric.* **23**, 1435 (1972).
Göbel, R., Franzke, C., *Lebensm.-Ind.* **22**, 30 (1975).
Jansen, H. E., Strating, J., Westra, W. M., *J. Inst. Brew. London* **77**, 154 (1971).
Kaiser, R., in "Chromatographie in der Gasphase III", Bibliographisches Institut, Mannheim, 1962, p 116.
Leonardos, G., Kendall, D., Barnard, N., *J. Air Pollut. Control Assoc.* **19**, 91 (1969).
Mulders, E. J., *Z. Lebensm. Unters.-Forsch.* **151**, 310 (1973).
Ohlson, R., *PAG Bull.* **3**(3), 21 (1973).
O'Keefe, A. E., Ortman, G. C., *Anal. Chem.* **38**, 760 (1966).
Persson, T., von Sydow, E., *J. Food Sci.* **38**, 377 (1973).
Persson, T., von Sydow, E., *J. Food Sci.* **39**, 406 (1974a).
Persson, T., von Sydow, E., *J. Food Sci.* **39**, 537 (1974b).
Persson, T., von Sydow, E., Åkesson, C., *J. Food Sci.* **38**, 386 (1973a).
Persson, T., von Sydow, E., Åkesson, C., *J. Food Sci.* **38**, 682 (1973b).
Pliška, V., Reisenauer, R., *Sb. Vys. Sk. Chem. Technol. Praze Potraviny* **5**(3), 21 (1961).
Qvist, I. H., von Sydow, E., *J. Agric. Food Chem.*, **22**, 1077 (1974).
Scaringelli, F. P., O'Keefe, A. E., Rosenberg, E., Bell, J. P., *Anal. Chem.*, **42**, 871 (1970).
Stevens, R. K., Mulik, J. D., O'Keefe, A. E., Krost, K. J., *Anal. Chem.* **43**, 827 (1971).
Teranishi, R., *Perfum. Essent. Oil Rec.* **58**, 1972 (1967).
von Sydow, E., Andersson, J., Anjou, K., Karlsson, G., Land, D., Griffiths, N., *Lebensm.-Wiss. Technol.* **3**, 11 (1970).
Watanabe, K., Sato, Y., *Agric. Biol. Chem.*, **34**, 1710 (1970).
Weurman, C., *Recent Adv. Food Sci.* **3**, 137 (1963).
Wilby, F. V., *J. Air Pollut. Control Assoc.* **19**, 96 (1969).

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