

eliminate matrix interferences, the analysis is fairly rapid and reliable, at least 4-5 times more rapid than the accepted atomic absorption method of the AOAC (1973).

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Volatile Constituents of Pressure Cooked Pork Liver

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The volatile constituents of pressure cooked pork liver were isolated by simultaneous steam distillation and continuous solvent extraction. Analysis by gas chromatography and coupled gc-mass

spectrometry led to the identification of 179 compounds. The mass spectral identifications were confirmed by matching retention indices. Pyrazines were the largest group of compounds found.

As a food, liver is an important source of protein, fat, and vitamins, particularly vitamins A, D, E, and vitamin B complex. A search of the literature, however, has revealed that no previous work has been reported on the volatile constituents of cooked liver. Pork liver was chosen for this study because its odor is typical of liver, and it is considerably stronger than calf or beef liver.

EXPERIMENTAL SECTION

Locally procured pork liver (42.5 lb) was sliced into small pieces and passed through a Fitz Mill Model D Comminuter (knives forward, no screen). The sliced liver was transferred to a 20-gal, steam jacketed, doubly stirred, stainless steel, pressure reaction vessel (Groen Div., Dover Corporation, Elk Grove, Ill.) and then slurried with 27 l. of distilled water. The vessel was sealed, heated to 325°F, and held at this temperature for 15 min. The maximum head pressure attained was 98 psi. At the end of the cooking period the vessel was cooled by passing water through the jacket, and the contents, which had a typical cooked liver aroma, were filtered through cheese cloth and a wire basket funnel into 5-gal polyethylene containers and stored at -20° until used.

The filtrate was atmospherically steam distilled, and the distillate continuously extracted with distilled diethyl ether in a scaled-up (22 l.) model of Williams's apparatus (Williams, 1969). The distillation-extraction was carried out over a 48-hr period for each of the two batches.

The extracts were combined, dried over anhydrous sodium sulfate, and initially concentrated to about 80 ml by careful distillation in a 1-l. Kuderna-Danish concentrator

(Kontes Glass Co., Vineland, N. J.) equipped with a 508 mm × 25.4 mm i.d. reflux column packed with 6 mm × 6 mm Raschig rings. The extract was further concentrated to about 15 ml in a 100-ml round-bottomed flask with a 35 mm × 10 mm o.d. test tube sealed to the bottom and equipped with a 300 mm × 13 mm i.d. Vigreux reflux column. The open container was then allowed to stand at room temperature until the sample had concentrated to a final volume of about 10 ml. The concentrated extract possessed a typical cooked liver aroma.

The concentrate was analyzed on a Hitachi Model RMU-6E mass spectrometer coupled with a Hewlett-Packard Model 5750 gas chromatograph using a Watson-Biemann helium separator (Watson and Biemann, 1965). The chromatographic columns used were 1000 ft × 0.03 in. i.d. stainless steel open tubular columns coated with SF-96 and Carbowax 20M. Further analysis was preceded by area trapping in Varian 1-ml collection bottles cooled with Dry Ice-isopropyl alcohol. The instrument used was a Varian Series 712 preparative gas chromatograph with a flame ionization detector employing a 12 ft × 3/8 in. stainless steel column packed with 20% SE-52 on 45-60 mesh acid-washed, DMCS-treated Chromosorb W. The oven temperature was programmed from 80 to 225° at 4°/min after a 5-min post-injection hold. The injector and detector temperatures were 230° and the helium flow rate was approximately 300 ml/min. Six traps were collected at arbitrary intervals and analyzed in the gc-mass spectrometry system. Traps 1-4 were analyzed on 1000 ft × 0.03 in. stainless steel open tubular SF-96 and Carbowax 20M columns. Trap 5 was analyzed on a 500 ft × 0.03 in. stainless steel open tubular SF-96 column and an 8 ft × 1/8 in. stainless steel column packed with 10% Carbowax 20M on 80-100 mesh acid-washed, DMCS-treated Chromosorb W,

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Table I. Compounds Identified from Cooked Pork Liver

Compound	Retention index ^a				Mass spectral data ^b
	CBW		SF-96		
	Known	Unknown	Known	Unknown	
					Hydrocarbons
2-Pentene		<1.00			55, 70, 42, 41, 39, 29
Heptane			3.05	3.05	43, 41, 29, 27, 57, 100
Pentadecene				10.77	41, 55, 57, 69, 83, 56...210
Pentadecane		9.10			57, 71, 43, 41, 85, 55...212
Hexadecene		10.43			41, 43, 55, 57, 83, 69...224
Hexadecane					57, 43, 71, 41, 29, 85...226
Heptadecane				12.38	57, 43, 71, 41, 85, 55...240
Benzene	2.92	2.80			78, 51, 52, 50, 77
Ethylbenzene			4.74	4.76	91, 106, 51, 39, 65, 27
Hexylbenzene					43, 77, 91, 162, 27, 29
Toluene			3.75	3.62	91, 92, 39, 65, 61
<i>m</i> -Xylene	5.17	5.17			91, 106, 105, 39, 51, 77
Limonene			6.49	6.33	68, 93, 39, 67, 41, 27...136
					Alcohols
Methanol			<1.00	<1.00	31, 32, 29, 28, 18
Ethanol			<1.00	<1.00	31, 45, 29, 27, 46
2-Propanol	2.30	2.26			45, 43, 27, 29, 41...60
1-Butanol	5.00	5.00			56, 31, 41, 43, 27, 42...74
2-Methyl-1-propanol	4.37	4.38			43, 31, 41, 42, 27, 33...74
2-Butanol	3.55	3.66			45, 27, 59, 31, 29, 43...74
3-Methyl-1-butanol	5.55	5.50			41, 29, 55, 31, 57...88
2-Methyl-2-butanol	3.45	3.40			59, 73, 43, 55, 31...88
2-Methyl-3-buten-2-ol	3.82	3.85			71, 43, 59, 41, 39...86
1-Octen-3-ol	7.97	8.00			57, 43, 41, 72, 55, 27...128
					Aldehydes
Acetaldehyde			<1.00	<1.00	29, 44, 43, 26, 42, 27
Propanal	<1.00	<1.00			29, 28, 27, 58, 26, 57
Butanal			1.79	1.76	44, 43, 72, 41, 27, 29
Pentanal			2.79	2.81	44, 29, 27, 41, 58, 28...86
Hexanal			4.00	4.00	41, 29, 43, 27, 56, 57...100
Heptanal			5.11	5.00	29, 27, 41, 44, 43...114
Octanal	6.59	6.67			43, 29, 41, 44, 57, 55...128
Nonanal			7.20	7.17	41, 43, 57, 56, 44, 29...142
Dodecanal			10.14	10.26	43, 41, 57, 82, 29, 68...184
Tridecanal	11.78	11.70			57, 43, 41, 82, 55...198
Tetradecanal			12.26	12.13	43, 41, 57, 55, 29, 82...212
Pentadecanal					43, 41, 57, 55, 29, 82...226
Hexadecanal					43, 41, 57, 55, 82, 29...240
Heptadecanal					43, 41, 57, 55, 29, 82...254
Octadecanal					43, 41, 57, 55, 82, 29...268
Isobutyraldehyde			1.28	1.31	43, 41, 72, 27, 29
Isovaleraldehyde			2.37	2.35	44, 41, 43, 46, 58, 29...86
2-Methylbutanal			2.49	2.44	29, 41, 27, 57, 58, 39...86
2-Butenal	4.05	4.00			41, 70, 39, 69, 29, 44
2-Methyl-2-butenal	4.67	4.58			55, 29, 84, 27, 39
2-Heptenal			5.63	5.52	41, 83, 55, 57, 56, 39...112
2-Octenal	8.00	7.95			41, 29, 27, 55, 39, 70...126
2-Phenyl-2-butenal			8.94	9.10	146, 117, 115, 91, 116, 39
5-Methyl-2-phenyl-2-hexenal			11.02	11.12	188, 91, 117, 115, 43, 39
Benzaldehyde			5.79	5.80	77, 105, 106, 51, 50
Phenylacetaldehyde			6.65	6.81	91, 92, 39, 65, 120
					Ketones
Acetone	1.00	1.00			43, 58, 27, 26, 42, 29
2-Butanone			1.74	1.65	43, 29, 27, 72, 26, 57
2-Pentanone			2.60	2.70	42, 27, 86, 41, 39, 58
2-Heptanone			4.89	4.89	43, 58, 27, 71, 29, 41...114
2-Octanone	6.56	6.61			43, 58, 41, 71, 59, 27...128
2-Decanone			8.00	8.00	58, 43, 59, 71, 41...156
2-Tridecanone	11.70	11.58			58, 43, 59, 71, 41, 57...198
2-Heptadecanone					58, 43, 71, 85, 69, 83...254
3-Pentanone			2.75	2.81	57, 29, 27, 86, 26, 42
3-Octanone	6.24	6.26			43, 57, 29, 71, 72, 99...128
4-Methyl-2-pentanone			3.47	3.28	43, 58, 57, 41, 29, 85...100
3-Buten-2-one	2.94	2.80			55, 43, 27, 70, 26, 42
3-Methyl-3-buten-2-one			2.55	2.50	43, 41, 84, 69, 39, 42
3-Penten-2-one	4.90	4.85			69, 41, 84, 43, 39, 40
2,3-Butanedione			1.69	1.82	43, 86, 42, 29, 41
2,3-Pentanedione			2.84	2.90	43, 29, 57, 27, 15, 100
Cyclopentanone	5.55	5.42			55, 28, 41, 84, 42, 56
2-Cyclopenten-1-one	7.30	7.11			39, 82, 27, 54, 53, 26
1-Phenyl-2-propanone			7.49	7.58	43, 91, 92, 134, 65, 39
1-Hydroxy-2-propanone				3.37	43, 31, 74, 29, 42, 45

Table I (continued)

Compound	Retention index ^a				Mass spectral data ^b
	CBW		SF-96		
	Known	Unknown	Known	Unknown	
3-Hydroxy-2-butanone			3.08	3.50	45, 43, 27, 29, 18, 28...88
3-Hydroxy-2-pentanone			4.25	4.38	59, 31, 43, 45, 41, 58...102
2-Hydroxy-3-pentanone			4.27	4.45	45, 59, 31, 43, 29, 57...102
			Esters		
Methyl formate			<1.00	<1.00	31, 29, 32, 60, 15
Ethyl formate	1.00	1.00			31, 29, 27, 45, 74
Ethyl acetate			2.00	2.00	43, 29, 27, 28, 45...88
Pentyl acetate	5.40	5.35			43, 70, 55, 15, 41, 27...130
Acetol acetate			4.89	5.10	43, 29, 42, 116, 86, 27
Ethyl propionate			3.00	3.00	29, 57, 27, 28, 26, 45...102
Ethyl isovalerate	4.28	4.30			29, 57, 27, 88, 41, 85...130
Ethyl dodecanoate	12.00	12.00			88, 29, 101, 41, 43, 27...228
Ethyl tetradecanoate			14.00	14.00	88, 43, 41, 29, 101, 55...256
Methyl hexadecanoate					74, 87, 75, 69, 143, 83...270
			Acids		
Acetic acid					43, 45, 60, 28, 15, 42
Octadecanoic acid					43, 57, 41, 55, 71, 73...284
			Lactones		
δ -Nonalactone			10.32		99, 42, 27, 41, 29, 71...156
δ -Decalactone	15.28				99, 27, 42, 41, 71, 43...170
			Furans		
2-Methylfuran	2.00	1.95			82, 53, 81, 39, 27, 29
2-Ethylfuran			3.00	3.00	81, 39, 96, 41, 51, 65
2-Butylfuran			5.02	5.16	81, 82, 124, 53, 39, 41
2-Pentylfuran			6.00	5.85	81, 82, 138, 53, 41, 39
2-Furaldehyde			4.62	4.91	39, 96, 95, 29, 38
5-Methyl-2-furaldehyde			5.61	6.00	110, 109, 53, 27, 29, 51
Furfuryl alcohol			5.33	5.90	39, 98, 41, 29, 81, 53
2-Acetylfuran			5.20	5.53	95, 110, 43, 39, 96, 68
Propionylfuran			6.39	6.40	95, 124, 39, 96, 67, 55
Furfuryl methyl ketone			5.67	5.80	43, 81, 53, 82, 124
2-Methyltetrahydrofuran-3-one			4.32	4.18	43, 72, 100, 29, 45, 27
Furfuryl formate			5.32	5.46	81, 53, 126, 52, 80, 27
Furfuryl acetate			5.88	6.11	81, 43, 98, 52, 53, 140
Furfuryl propionate			7.06	7.03	81, 27, 29, 52, 53, 98...154
Furfuryl butyrate	10.18	9.81			81, 27, 98, 43, 53, 52...168
Furfuryl pentanoate			8.85	8.26	81, 27, 29, 98, 53, 41...182
Furfuryl hexanoate	12.00	12.02			81, 27, 98, 53, 43, 29...196
Ethyl furoate	9.79	9.81			95, 29, 39, 112...140
Ethyl furfuryl ether			5.09	5.08	81, 82, 53, 27, 29, 126
Furfuryl ether			9.38	9.42	81, 82, 53, 27, 97, 39...178
2,2'-Methylenedifuran			6.87	7.16	148, 91, 120, 147, 39, 65
5-Methyl-2,2'-methylenedifuran			7.84	7.95	162, 91, 43, 147, 161, 119
2(or 3)-Phenylfuran			8.40 ^c	8.64	144, 115, 145, 63, 116, 89
			Thiazoles		
Thiazole	6.18	6.25			58, 85, 57, 45, 26, 32
4-Methylthiazole	6.50				99, 71, 72, 45, 39, 69
2-Acetylthiazole			6.30	6.62	43, 127, 99, 58, 57, 112
			Thiophenes		
2-Methylthiophene	4.62	4.58			97, 98, 45, 39, 99, 27
2-Thiophenecarboxaldehyde			6.19	6.54	111, 112, 39, 29, 45, 83
3-Thiophenecarboxaldehyde			6.27	6.62	111, 112, 39, 45, 83, 57
3-Methyl-2-thiophene-carboxaldehyde			7.48	7.21	125, 126, 97, 45, 53, 127
5-Methyl-2-thiophene-carboxaldehyde			7.81	7.75	125, 126, 53, 97, 45, 127
2,5-Dimethyl-3-thiophene-carboxaldehyde			8.23	8.27	140, 139, 111, 59, 29, 45
2-Acetylthiophene			6.90	7.44	111, 126, 39, 43, 45, 83
3-Acetylthiophene					111, 43, 126, 39
5-Methyl-2-acetylthiophene			8.36	8.48	125, 140, 53, 45, 43, 97
Thiopheneacrolein	14.92	15.52			138, 45, 110, 29, 109, 39
			Sulfur Compounds		
Methanethiol			<1.00	<1.00	47, 48, 45, 46, 15, 44
Methyl sulfide	<1.00	<1.00			62, 47, 45, 46, 61
Methyl disulfide			3.30	3.47	94, 45, 79, 46, 47, 43
Methyl sulfone	12.12	12.10			79, 94, 45, 29, 48, 63
Methyl sulfoxide	9.29	9.41			63, 78, 45, 29, 46, 61
Methyl thioacetate			3.00	3.00	43, 90, 45, 47, 48, 42
Methyl thiopropionate	4.93	4.92			29, 57, 104, 27, 45, 47
Ethyl thiopropionate	5.31	5.30			57, 29, 118, 27, 61, 58
Benzyl methyl sulfide	10.29	10.26			91, 138, 45, 65, 39, 92
Furfuryl methyl sulfide	8.58	8.38			81, 128, 53, 27, 45, 51
Furfuryl methyl disulfide			8.39	8.46	81, 53, 27, 45, 51, 160

Table I (continued)

Compound	Retention index ^a				Mass spectral data ^b
	CBW		SF-96		
	Known	Unknown	Known	Unknown	
					Pyrroles
Pyrrole-2-carboxaldehyde			8.44	8.27	95, 94, 39, 66, 38, 28
5-Methylpyrrole-2-carboxaldehyde			8.32	8.53	109, 108, 80, 53, 27, 29
1-Acetylpyrrole	9.06	8.95			67, 109, 39, 43, 41, 40
2-Acetylpyrrole			8.39	8.18	94, 109, 39, 66, 38, 43
2-Propionylpyrrole	13.56	13.70			94, 123, 66, 39, 95, 28
					Pyrazines
Pyrazine			3.30	3.50	26, 80, 53, 28, 52, 51
Methylpyrazine			4.38	4.38	94, 67, 26, 39, 40, 55
Ethylpyrazine			5.21	5.30	107, 108, 26, 27, 39, 79
2,3-Dimethylpyrazine			5.45	5.30	67, 108, 42, 41, 39, 52
2,5-Diethylpyrazine			5.17	5.21	42, 108, 39, 38, 27, 81
2,6-Dimethylpyrazine			5.12	5.28	108, 42, 39, 38, 67, 41
2-Ethyl-5-methylpyrazine			5.96	6.00	121, 122, 39, 27, 56
2-Ethyl-6-methylpyrazine			5.91	5.95	121, 122, 39, 42, 40, 27
2,5-Diethylpyrazine			6.93	7.00	136, 121, 135, 39, 27
Trimethylpyrazine			6.16	6.09	42, 122, 39, 27, 81, 54
2-Ethyl-3,5-dimethylpyrazine			6.83	6.93	135, 136, 39, 42, 56, 54
2-Ethyl-3,6-dimethylpyrazine			6.90	6.81	135, 136, 42, 56, 39, 108
5-Ethyl-2,3-dimethylpyrazine					135, 136, 42, 39, 56, 108
2,3-Diethyl-5-methylpyrazine			7.63	7.59	150, 135, 56, 39, 149, 41
2,5-Diethyl-3-methylpyrazine	8.70	8.52			149, 150, 39, 135
3,5-Diethyl-2-methylpyrazine	8.60	8.47			149, 150, 39, 122, 53, 135
Triethylpyrazine	9.06	9.08			164, 149, 163, 39, 56, 136
Tetramethylpyrazine	8.41	8.30			54, 136, 42, 27, 52, 39
2-Methyl-3-vinylpyrazine	6.28	6.42			119, 120, 67, 26, 39, 52
2-Methyl-5-vinylpyrazine			6.26	6.40	120, 52, 54, 39, 51, 27
Acetylpyrazine			6.30	6.66	43, 122, 80, 79, 52, 53
2-Methyl-5(or 6)-acetylpyrazine			7.27 ^d	7.50	136, 43, 94, 93, 39, 67
2-Ethyl-3-acetylpyrazine			7.81	7.93	43, 107, 150, 52, 27, 79
2-Ethyl-5(or 6)-acetylpyrazine			7.70 ^e	7.90	150, 43, 107, 108, 53, 27
6,7-Dihydro-5H-cyclopentapyrazine			7.18	7.43	120, 119, 39, 65, 52, 27
2-Methyl-6,7-dihydro-5H-cyclopentapyrazine	10.73	10.60			134, 133, 39, 66, 40, 107
5-Methyl-6,7-dihydro-5H-cyclopentapyrazine			7.47	7.56	119, 134, 133, 39, 27, 52
2(or 3)-5-Dimethyl-6,7-dihydro-5H-cyclopentapyrazine	10.26	10.26			148, 66, 147, 39, 27, 107
Quinoxaline	12.71	12.55			130, 76, 103, 50, 75, 51
2-Methylquinoxaline			9.24	9.39	144, 117, 76, 50, 77
6-Methylquinoxaline		13.08			144, 90, 89, 117, 143, 145
5,6,7,8-Tetrahydroquinoxaline			8.05	8.22	134, 133, 52, 106, 39, 27
2-Methyl-5,6,7,8-tetrahydroquinoxaline			9.00	9.05	148, 147, 39, 52, 120, 79
(2-Furyl)pyrazine	12.80	13.35			146, 93, 63, 64, 92, 39
2-(2-Furyl)-5(or 6)-methylpyrazine			9.87	9.64	160, 92, 63, 39, 93, 64
					Miscellaneous
Ethyl vinyl ether			<1.00	<1.00	44, 43, 72, 29, 27
Diethyl ether			1.10	0.97	31, 59, 29, 45, 27, 74
Acetaldehyde, diethyl acetal			3.20	3.18	45, 73, 29, 27, 43...118
Phenol	13.46	13.40			94, 39, 66, 65, 40
Trimethyloxazole	5.50	5.68			43, 42, 111, 55, 68, 27
Vanillonitrile					134, 149, 106, 29, 51, 78

^a Compounds with an I_E value lower than that of ethyl formate (1.00) are given as <1.00. ^b These data were taken from the mass spectra of known compounds. However, in every case the data for the unknown compound matched that for the known. The mass spectrometer used had a source temperature of 150°, an ionizing voltage of 70 eV, and an ionizing current of approximately 3.5 A. Numbers in italics are the molecular weights of the compounds. ^c The 2 and 3 isomers have very similar retention indices and mass spectra. The authentic compounds were synthesized by E. Klaiber and A. Ø. Pittet of IFF. ^d I_E for 2-methyl-5-acetylpyrazine. ^e I_E for 2-ethyl-5-acetylpyrazine.

and trap 6 was analyzed on a 3 ft \times $\frac{1}{8}$ in. stainless steel column packed with 2% OV-17 on 80-100 mesh acid-washed, DMCS-treated Chromosorb W. All capillary analyses were carried out at a helium flow rate of 10 ml/min. The oven temperature was programmed from 70 to 190° at 1°/min for the total extract and trap 1, and at 2°/min for traps 2-5.

Identifications were based on the comparison of known and unknown mass spectra and confirmed wherever possible by determining the retention indices, or I_E values, relative to a series of ethyl esters of normal alkanic acids (van den Dool and Kratz, 1963). Additional confirmation for certain compounds was provided by the gc analysis of the total extract and each trap for the presence of sulfur and/or nitrogen containing compounds. The instrument used was a Tracor MT 220 gas chromatograph modified in our laboratory as described by Walradt (1973).

For identification purposes, some compounds for which mass spectral data were not available were synthesized: furfuryl methyl ketone (Hass *et al.*, 1950); ethyl furfuryl ether (Kirner, 1928); propionylfuran (Heid and Levine, 1948); furfuryl methyl disulfide (Milligan and Swan, 1963); 2-thiophene acrolein (Hori, 1958); 2,4,5-trimethyl-oxazole (Reppe and Magin, 1963); ethyl thiopropionate, by a modification of the procedure of McFadden *et al.* (1965); and furfuryl propionate, by a modification of a method described by Blatt (1941). The bicyclic pyrazines were synthesized by Pittet *et al.* (1974).

RESULTS AND DISCUSSION

Table I lists the compounds that were identified. In some cases, the small amount of sample available precluded the determination of I_E values. In other cases the "known" compound was not readily available. In the case of some very polar compounds, for example furfuryl alcohol, the known and unknown I_E values may differ considerably due to the fact that the I_E value of the unknown is determined on a complex mixture "spiked" with ethyl esters where its retention time may be affected by the other components. The I_E value for the known compounds, however, is determined on a mixture of the pure compound with a series of ethyl esters. This effect is particularly evident with free acids. Where there is a discrepancy in the I_E value, the compound was only reported if its mass spectral fragmentation pattern was very characteristic. In those cases where the position of the double bond was not specified, this information could not be unambiguously determined from the data.

Table II shows some quantitative data for the major classes of compounds found in cooked pork liver. The figures were determined by computer area normalization and incorporate data for both the positively identified com-

Table II. Relative Per Cent of Major Classes of Compounds in Volatiles from Cooked Pork Liver

Class	Quantity, %
Ketones	0.53
Esters	0.56
Hydrocarbons	1.33
Miscellaneous	1.38
Sulfur compounds	2.16
Alcohols	4.21
Aldehydes	14.07
Furans	28.80
Pyrazines	40.96
Total 94.00	

pounds reported in this paper as well as some tentatively identified compounds. It should be pointed out that pyrazines were by far the largest class of compounds found, accounting for almost 41% of the isolated components amenable to gc analysis.

SUMMARY

Pork liver, cooked superatmospherically at 325°F, was analyzed by gc and coupled gc-mass spectrometry. A total of 179 components have been identified. Although none of these chemicals can be considered to have a typical liver character, many of them are believed to contribute to the overall flavor quality.

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