Volatile Flavor Components of Wood Apple (*Feronia limonia*) and a Processed Product

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Representative samples of the aroma volatiles of wood apple—a tropical fruit—and a canned, processed product (wood apple cream) were obtained by means of a modified Likens and Nickerson apparatus using trichlorofluoromethane as the solvent. Extracts were concentrated by a low-temperature-high-vacuum distillation procedure, and components of resultant essences were identified as far as possible by GC-MS using both EI and CI mass spectrometry. Odor evaluation at an odor port during GC revealed three components described as having characteristic wood apple aroma—methyl hexanoate, ethyl 3-hydroxyhexanoate, and butanoic acid. Most aroma components were esters (~45% of the total sample) and included  $\beta$ -hydroxy esters which have previously only been located in tropical fruits. Two similar series of chemically closely related compounds were recognized, together comprising ~70% of the samples. Overall wood apple cream provided very similar results to wood apple fruit and was therefore a good preserved substitute.

Wood apple (Feronia limonia) is a tropical fruit native to India and Sri Lanka which has not before been analyzed for its volatile flavor components. The brown edible pulp of the fruit is enclosed within a rough woody pericarp, and it possesses a most characteristic and unusual flavor. It is quite pleasant but with an odd, slightly fatty undertone such that it might not appeal to all palates. The fruit is widely consumed as such but the pulp is also used for making cordials, cream, and jelly, all of which retain the unusual flavor to some extent. Wood apple has recently become an economically important commodity in Sri Lanka because the cream is now canned and exported. The objectives of this project were thus to investigate in detail the nature of the volatile flavor components of wood apple fruit and then to locate, if possible, those compounds mainly contributing to its characteristic flavor. In addition, a canned product (the cream) was examined, and the intention was to compare the volatiles of this with those of the fruit; in particular, it was hoped to be able to pinpoint the significant flavor components in the fresh fruit which would be most desirable to retain in any processed product.

#### EXPERIMENTAL SECTION

Fresh wood apple fruit was transported by air from Sri Lanka such that it could be analyzed almost immediately on arrival in its ripe state. The canned wood apple cream was produced by the Sri Lanka Marketing Department and had been stored for  $\sim 2$  months before analysis.

Sample Preparation. Extracts of aroma components of wood apple fruit or cream were obtained by using either a conventional Soxhlet apparatus (but equipped in addition with a solid  $CO_2/acetone$  condenser) or a Likens and Nickerson (1964) apparatus as modified by MacLeod and Cave (1975). For the fruit, ~150 g of the inner pulp with embedded seeds was used; the pulp closest to the woody pericarp was rejected. Cream (200 mL) was diluted with 50 mL of water. The extracting solvent in all cases was trichlorofluoromethane (150 mL with the Soxhlet apparatus; 15 mL with the Likens and Nickerson apparatus), and extraction was carried out for 10 h with the Soxhlet but only 5 h with the Likens and Nickerson apparatus. At the end of these periods the residues did not possess any appreciable aroma. Extracts were concentrated to  $\sim 0.5$  mL by the technique of low-temperature-high-vacuum distillation previously described (MacLeod and Cave, 1975). The resultant essences had strong aroma, characteristic of either fruit or cream as appropriate.

Gas Chromatography. Essences were examined by gas chromatography using a Pye-Unicam 204 instrument equipped with heated FID. Three different columns were mainly employed. The first was an 18 ft  $\times$  4 mm i.d. glass column packed with 10% PEG 20M coated on 100-120 BSS mesh acid-washed Diatomite C. Nitrogen carrier gas was used (60 mL/min), and the best temperature program for fruit essences was 60 °C for 10 min followed by an increase at 16 °C/min to 195 °C for the remainder of the run. but for cream samples, 60 °C for 5 min followed by an increase at 12 °C/min to 160 °C was superior. Detector and injection temperatures were 250 °C, and typically 4  $\mu L$  of sample was injected. Retention times were determined by using this column and the appropriate operating conditions. The second column was a 100 ft  $\times$  0.02 in. i.d. stainless steel SCOT column with an OV-101 stationary phase. A flow rate of 5 mL/min was used, and the temperature program was 60 °C for 5 min followed by an increase of 4 °C/min to 150 °C for the remainder of the run. About 0.4  $\mu$ L of sample was injected. The third column was a 44 m  $\times$  0.5 mm i.d. glass SCOT column with an OV-101 stationary phase. A flow rate of 3 mL/min was used but the temperature program was the same as that employed with the metal column. Other operating conditions were also the same.

Gas Chromatography-Mass Spectrometry. Components in the essences were identified as far as possible by GC-MS using a Kratos AEI MS 25 instrument linked on-line to a DS 50 data processing system. All three GC columns were employed at various times under the conditions described above, but using helium as carrier gas. A single stage, all-glass jet separator was used at 250 °C. Both electron impact (EI) and chemical ionization (CI) mass spectrometry were performed, and at various times the MPM unit and the retrospective single ion monitoring facility of the data system were employed to good advantage. Significant operating parameters of the mass spectrometer during EI work were as follows: ionization potential, 70 eV; ionization current, 100  $\mu$ A; source temperature, 200 °C; accelerating voltage, 1.5 kV; resolution, 600; scan speed, 1 s/decade (repetitive throughout run). Identical conditions were employed during CI mass spec-

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trometry except for the following: reagent gas, methane (or isobutane or ammonia); ionization potential, 100–110 eV; emission current, 5 mA.

Odor Assessment. Aromas of the separated components of the essences were assessed at an odor port following gas chromatography using a Pye Unicam 104 instrument. The 18-ft glass PEG 20M column was used with an outlet splitter set at 10:1. The major fraction passed through a heated line to the outside of the oven for aroma assessment by a total of three subjects. An injection volume of 10  $\mu$ L was necessary.

## RESULTS AND DISCUSSION

Various methods of extraction using many different solvents were assessed, but it was found that a representative and fairly stable flavor extract of wood apple with an obvious and valid aroma of the fresh fruit could be obtained simply by using a Soxhlet apparatus but with trichlorofluoromethane as the extracting solvent. Optimum conditions for the procedure were determined, and it was found necessary to incorporate a solid  $CO_2/acetone$ condenser beyond the water condenser to eliminate loss of more volatile components and solvent.

However, much superior extracts (i.e., more concentrated yet still equally representative) were obtained more readily and more efficiently by using a Likens and Nickerson (1964) apparatus, but modified as described by MacLeod and Cave (1975). This apparatus was particularly advantageous with the wood apple cream. Again trichlorofluoromethane was the best solvent of those examined (including isopentane and dichloromethane). However, in addition it afforded other advantages for this type of work, including the fact that it is essentially odorless and that it does not extract ethanol (which is usually a major and potentially interfering component extracted from many fruits, but which does not itself contribute significantly to flavor). It is for these, and similar, reasons that trichlorofluoromethane has been the subject of a number of patents relating to the production of commerical aroma extracts [e.g., Stanley et al. (1963) and Scheide and Bauer (1967)], but surprisingly it has been rarely used in basic analyses of the type described here. Neverless, it can be recommended for this purpose. In a recent comparative survey using our modified Likens and Nickerson apparatus, it was found to be an efficient extracting solvent for a range of solutes with recoveries from model systems generally in excess of 80% (Au-Yeung and MacLeod, 1980). Since trichlorofluoromethane is heavier than water, it is necessary to reverse the Likens and Nickerson extractor (i.e., interchange sample and solvent flasks), but the modified apparatus functioned equally efficiently in this mode.

Extracts obtained as described above were concentrated to  $\sim 0.5$  mL by using the low-temperature-high-vacuum distillation procedure devised by MacLeod and Cave (1975), and the essences obtained were found to retain the genuine aroma qualities of the original extracts. They were examined initially by routine temperature-programmed gas chromatography using 18-ft packed glass columns containing either a polar stationary phase (10% PEG 20M) or a nonpolar one (5% OV-101). Superior resolution of the complex mixtures was achieved using 100-ft SCOT stainless steel columns, again employing either PEG 20M or OV-101 stationary phases. Also used was a 44-m SCOT glass capillary column containing OV-101. Most of this work was carried out on three columns, as described under Experimental Section.

Components in the essences were identified as far as possible by GC-MS, initially based on knowledge of basic mass spectral fragmentations but ultimately by comparisons with literature spectra. In all instances where positive identifies are quoted, agreement between sample spectra and literature spectra were near perfect, within instrumental error (i.e., variability). Chemical ionization mass spectrometry (usually employing methane as the reagent gas) was invaluable in defining the molecular weights of most components, hence rendering interpretation of conventional electron impact spectra somewhat easier.

Table I lists the volatile flavor components of wood apple fruit, together with GC retention data (also for reference compounds when available), quantitative data. and odor qualities of the various GC peaks. Table II details similar results for wood apple cream. Where no odor quality is given, this means either that no odor could be detected or that the GC peak was insufficiently resolved for a distinct odor to be recognized; toward the end of the wood apple cream runs a background aroma obscured and prevented reasonable descriptions. Retention times quoted were determined by using the 18-ft PEG 20M column under appropriate operating conditions for fruit or cream and are absolute (no internal standard was employed), so agreement between unknown and reference is not always good. However, all identifications are positive by GC-MS except where indicated by (?). At least 100 peaks were observed on GC of both fruit and cream by using the most efficient SCOT column, but the majority of these were minor components, together contributing less than 5% of the whole sample.

From Table I it can be seen that the wood apple fruit essence contained 46 main components, of which 26 (comprising ~96% of the sample) have been positively identified with a further 3 (0.3%) tentatively assigned. The cream essence (Table II) contained 50 main components, of which 33 (~97% of the sample) have been positively identified with a further 10 (2.4%) tentative or partially characterized. Clearly, the majority of components were common to both types of sample.

Aromas of the separated components of the essences were evaluated during GC at an odor port by up to three assessors at various times. None of the assessors was highly trained in sensory evaluation although all had experience. and all had sampled fresh wood apple; one (N.M.P.) was particularly familiar with the fruit. A number of GC peaks were determined to have definite wood apple quality. These corresponded to the following components identified in the fruit: methyl (ethyl) hexanoate, ethyl 3-hydroxyhexanoate, and butanoic acid. These same components were similarly assessed as having wood apple aroma in samples of the cream, but with hexan-1-ol in addition. It would appear that these three compounds are therefore significant contributors to characteristic wood apple flavor. and it is noteworthy that the acid, and to a lesser extent the hydroxy ester, were present in quite high concentrations in both fruit and cream. Pure samples of all three compounds were obtained, and at appropriate dilution they were found by the three assessors to possess aromas distinctly reminiscent of various odor qualities of wood apple. so the components could, perhaps, be considered as character impact compounds for the fruit. They do not in themselves, however, provide complete wood apple aroma. It is significant that all three were found in the cream as well as the fruit, and although the concentration of butanoic acid was rather less in the cream, concentrations of the other two components were comparable in both fruit and cream. It is noticeable that the odor descriptions of wood apple components generally fell into two broad categories, namely, fruity/estery/fragrant and rancid/

Table I.	Volatile	Flavor	Components of	Wood Apple Fru	iit
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peak no.	component	$t_{\rm R},$ min	t <sub>R</sub> (ref), min	% rel abund	mg/kg of fruit	odor quality
1	unknown	1.7		0.02	0.01	fragrant
2	unknown	3.0		0.02	0.02	fragrant
3	acetone	5.8	5.4	14.9	12.0	sweet, fruity
4	unknown	8.9		0.03	0.03	sweaty
5	carbon tetra-		8.9			
	chloride >	9.3		2.2	1.8	fruity
	ethyl acetate 🕽		9.0			
6	diacetyl (butanedione)	10.7		0.5	0.4	
7	unknown	11.3		0.1	0.08	fruity
8	unknown	11.5		0.01	0.005	-
9	methyl butanoate	12.3	12.4	8.3	6.7	fruity, ester
10	chloroform	13.0	13.2	1.2	1.0	medicinal
11	ethyl butanoate	13.4	13.6	26.0	20.8	sweet, fruity
12	(?)butyl acetate	13.8		0.1	0.09	, ,
13	toluene	14.0	13.6	1.4	1.1	
14	(?)pentan-2-ol	14.7		0.2	0.17	burnt, toffee
15	butan-1-ol	15.3	14.6	3.2	2.6	sweet
16	pent-3-en-2-one	16.4		0.4	0.29	sweet, fruity
17	ethyl but-2-enoate	16.7		1.75	1.4	spicy, aromatic
18	methyl hexanoate	17.4	16.0	0.04	0.03	-1 07
19	propyl butanoate	17.6		0.02	0.02	faint wood apple
20	ethyl hexanoate	17.7	16.4	0.7	0.5	••
21	(?)3-methylpentan-2-one	18.2		0.02	0.014	
$22^{}$	unknown	18.7		0.01	0.005	
23	3-hydroxybutan-2-one	19.2	18.8	4.4	3.5	fruity, toffee
24	hexan-1-ol	19.7	19.1	1.6	1.3	floral, fragrant
25	a trimethylbenzene	20.4		0.03	0.03	caramel
26	ethyl hex-2-enoate	21.1		0.4	0.3	fragrant, new cloth
27	unknown	21.7		0.13	0.1	carrots
28	unknown	23.0		0.11	0.09	burnt, sweet
29	unknown	23.3		0.02	0.014	faint toffee
30	methyl 3-hydroxybutanoate	23.9		1.1	0.85	green
31	ethyl 3-hydroxy butanoate	25.0	23.9	$\frac{1.1}{5.1}$	4.1	sweet, aromatic, nutmeg
32	unknown	26.8	20.0	0.12	0.1	Sweet, aromatic, nating
33	unknown	28.1		0.01	0.005	
34	butanoic acid	28.7	25.6	11.7	9.35	wood apple, spicy
35	unknown	30.5	20.0	0.3	0.22	aromatic
36	unknown	31.4		0.01	0.01	sweaty
37	unknown	32.2		3.7	2.9	spicy, caramel
38	methyl 3-hydroxyhexanoate	33.6		0.1	0.07	pleasant, estery
39	ethyl 3-hydroxyhexanoate	36.8	34.1	0.1	0.62	faint wood apple, sickly
40	unknown	40.0	04.1	0.13	0.1	unpleasant, sweaty
40	unknown	40.0		0.15	0.05	spicy, nuts
42	hexanoic acid	43.9	39.3	7.4	5.9	unpleasant, sweaty
43	δ-hexanolactone	43.9 44.2	00.0	0.8	0.6	arpicasario, 5 weary
43	2-phenylethanol	45.0		0.8	0.1	
45	$\delta$ -heptanolactone	43.0 48.2		0.2	0.5	sour
		10.4		0.0	0.0	5041

sweaty, and indeed a combination of these two broad qualities approximates the basis of wood apple flavor. There is little doubt that the high percentage of butanoic acid contributes greatly to the underlying fatty note of the flavor.

Considering the types of compounds identified in samples of wood apple volatiles, the majority were esters (ca. 46% for the fruit and 40% for the cream) with only two terpenes. The major component in both cases was ethyl butanoate ( $\sim 25\%$  of the total sample). The important butanoic acid was the second most abundant component but was at a higher relative percentage in the cream than in the fruit. The figure of 26% relative abundance for ethyl butanoate in fruit essences corresponded to an absolute concentration of  $\sim 21 \text{ mg/kg}$  of fruit. For the cream, 24.5% ethyl butanoate in the essence corresponded to  $\sim$ 4.4 mg/L in the cream itself, so on this basis as far as this aroma component is concerned the fruit had nearly 5 times the flavor strength of the cream. Other data in Tables I and II show the same relationship for other components, which explains the observed weaker aroma intensity of the cream. Presumably some dilution occurs during processing. However, overall the cream is an excellent product and is very representative of genuine wood apple flavor, since although it was weaker it did contain comparable concentrations of the character impact compounds and it did contain all the other significant constituents of the fruit. A few not unexpected additional compounds were obtained from the cream (e.g., furan-2aldehyde) presumably formed as artifacts during thermal stages of processing. These, and particularly the 14% content of furan-2-aldehyde, contributed to the very slight off-flavor noticeable for the cream.

A most interesting series of chemically related compounds was observed in the aroma volatiles of wood apple fruit and cream. With one minor exception, the methyl and ethyl esters of butanoic acid, of but-2-enoic acid, and of 3-hydroxybutanoic acid were identified, together with butan-1-ol and butanoic acid. All were present in reasonable amounts and some were major components, such that these compounds comprised ~57% of the total volatiles of the fruit and ~63% of the cream. An identical series was obtained for the C<sub>6</sub> parent compounds, but these were present to lesser extents (ca. 11% and 9% for the fruit and cream, respectively). These two series thus made up ~70% of the aroma volatiles of wood apple. Some indication was also obtained that similar series might exist for C<sub>2</sub> and C<sub>8</sub> compounds as well. The limitation to even

### Table II. Volatile Flavor Components of Wood Apple Cream

peak no	. component	$t_{R},$ min	t <sub>R</sub> (ref), min	% rel abund	mg/L of cream	odor quality		
1	acetone	4.3	4.6	11.7	2.1			
2	carbon tetra-		5.2					
	chloride >	4.5		0.8	0.14	sweet		
	ethyl acetate )		5.3					
3	unknown	5.3		0.05	0.01	sweet, floral		
4	diacetyl (butanedione)	6.3		0.5	0.1	faint caramel		
5	unknown	7.2		0.3	0.06	fruity, floral		
6	unknown	7.7		0.01	0.001			
7	unknown	7.9		0.01	0.002			
8	unknown	8. <b>2</b>		0.04	0.007			
9	methyl butanoate	8.5	8.6	2.6	0.46	estery, floral		
10	chloroform	9.2	9.3	0.06	0.01			
11	ethyl butanoate	9.5	9.2	24.5	4.4	estery, fresh		
12	(?)a trimethyl-1,3-dioxane	9.9		0.5	0.09	butterscotch		
13	(?)butyl acetate	10.2		0.2	0.04 🖇	butterscotch		
14	(?)pentan-2-ol	10.5		0.2	0.04			
15	butan-1-ol	11.1	11.3	1.0	0.18			
16	pent-3-en-2-one	11.4		4.6	0.83	toffee, burnt		
17	ethyl but-2-enoate	12.0		1.1	0.19	lachrymatory		
18	methyl hexanoate	12.3	12.9	0.6	0.11	faint wood apple		
19	propyl butanoate	13.0		0.02	0.003	green, fresh		
20	ethyl hexanoate	13.8	13.7	0.7	0.13	green, floral		
21	(?)3-methylpentan-2-one	14.3		0.01	0.002	rancid		
$\overline{22}$	hept-2-en-4-one	14.8		1.2	0.21	toffee, rancid		
23	3-hydroxybutan-2-one	15.2	15.4	2.3	0.41	fruity		
$\overline{24}$	hexan-1-ol	15.9	16.3	3.0	0.53	faint wood apple, fruity		
$25^{-1}$	ethyl hex-2-enoate	16.8	2010	0.06	0.01	estery, acidic, hay		
26	a $C_2$ lactone	18.3		0.2	0.04	cardboard		
$\frac{1}{27}$	unknown	19.1		0.03	0.004	wood		
28	ethyl octanoate	19.9		0.1	0.02	fruity		
29	a $C_2$ lactone	20.9		0.2	0.03	cucumber		
30	furan-2-aldehyde (furfural)	20.3 21.7		13.6	2.4	cooked, brown		
31	methyl 3-hydroxybutanoate	22.7		0.3	0.06	cooked, brown		
32	2-acetylfuran	23.5		0.0	0.13			
33	ethyl 3-hydroxybutanoate	20.0 23.7	24.5	2.8	0.10	sweet		
$33 \\ 34$	benzaldehyde	25.4	24.0	0.04	0.007	SWEEL		
35	unknown	26.7		0.04	0.007	pyrazine-like		
36	5-methylfuran-2-aldehyde	28.7		0.04 0.7	0.13	rancid butter		
37	terpin-4-ol	30.8		0.05	0.008	Tallelu Duttel		
38	butanoic acid	31.2	32.0	20.7	3.7	wood apple, rancid		
						pineapple		
39	methyl 3-hydroxyhexanoate	33.3		0.3	0.06			
40	ethyl 3-hydroxyhexanoate	36.4	37.2	4.2	0.74	faint wood apple		
41	terpin-2-ol	38.4		0.2	0.03	floral, fruity, violets		
42	a lactone	39.3		0.3	0.04	sweaty, sour		
43	a butyrate	42.4		0.3	0.04	faint wood apple		
44	methyl ester	43.5		0.1	0.02			
45	ethyl ester	46.3		0.4	0.07			
46	hexanoic acid	49.2		0.02	0.003			
47	δ-hexanolactone	49.5		0.15	0.025			
48	2-phenylethanol	50.5		0.01	0.001			
49	$\delta$ -heptanolactone	53.3		0.03	0.005			

Table III. Summaries of the Mass Spectra (Eight Most Intense Peaks) of Some of the Esters Identified in Wood Apple Aroma Volatiles

	m/e (% rel intensity) <sup>a</sup>							
methyl hexanoate	74 (100)	43 (84)	41 (40)	29 (40)	87 (31)	59 (30)	55 (27)	71 (23)
ethyl hexanoate	88 (100)	29 (97)	43 (92)	99 (54)	60 (52)	41 (50)	61 (40)	42 (32)
ethyl octanoate	88 (100)	101 (48)	43 (38)	41 (34)	29 (31)	55 (31)	73 (22)	57 (18)
ethyl but-2-enoate	69 (100)	29 (63)	41 (43)	39 (31)	99 (29)	86 (8)	87 (3)	114 (3)
ethyl hex-2-enoate	97 (100)	99 (80)	55 (75)	29 (61)	73 (51)	41 (45)	68 (24)	39 (18)
methyl 3-hydroxybutanoate	43 (100)	45 (45)	74 (38)	42 (32)	71 (24)	87 (15)	59 (12)	103 (12)
ethyl 3-hydroxybutanoate	43 (100)	45 (90)	29 (57)	42 (41)	60 (37)	71 (33)	87 (27)	88 (26)
methyl 3-hydroxyhexanoate	103 (100)	74 (82)	71 (78)	43 (64)	61 (32)	44 (27)	59 (24)	97 (12)
ethyl 3-hydroxyhexanoate	117 (100)	71 (98)	43 (78)	29 (52)	89 (43)	88 (42)	55 (33)	97 (25)

<sup>a</sup> Values are m/e. Numbers in parentheses are percent relative intensity.

carbon number parent compounds is reasonable and implies acetate precursor. It is also interesting perhaps to observe the presence of quite large amounts of hept-2en-4-one (and pent-3-en-2-one) and 3-hydroxybutan-2-one, which bear a structural relationship to some of the esters just mentioned. Thus, the heptenone is obtained by replacing the ester oxygen of ethyl but-2-enoate with a methylene group, and equally there is some similarity between the hydroxy ketone and the hydroxybutanoates. However, neither of these could be considered direct chemical or biochemical relationships, but the co-occurrence specifically of these ketones may have some significance.

All these components were positively identified by GC– MS, but since mass spectra of some of these esters have not been widely published, Table III provides summaries of the spectra of the less common compounds.

From a biosynthetic point of view the presence of two such similar, very closely related series of compounds is most intriguing, and presumably compounds within a series must be associated in some way, although this has not been suggested before. Heinz and Jennings (1966) have, however, pointed out that even carbon number carboxylic esters and their hydroxy derivatives could arise from condensation reactions involving acetyl-CoA and malonyl-CoA, but no involvement of related 2-enoates has been suggested. However, in the series reported here the double bond and the hydroxyl group are attached to the same carbon atom of the parent acid, so clearly the 2-enoates could be condensation products of the 3-hydroxy esters, or possibly the sequence could be reversed. It is also most interesting to find the parent acid and alcohol present in the same system.

The  $\beta$ -hydroxy esters have been reported as flavor components before, but not frequently, and they are very much less common aroma components than the parent esters. Nevertheless, certain members have been identified in pineapple (Creveling et al., 1968), passion fruit (Murray et al., 1972; Winter and Kloti, 1972), orange, grapefruit (Moshonas and Shaw, 1971), tangerine (Moshonas and Shaw, 1972), and grapes (Stern et al., 1967), although the full range found in wood apple has not been determined elsewhere. The 2-enoates are less common still, but ethyl but-2-enoate and ethyl hex-2-enoate have been found in passion fruit (Murray et al., 1972; Winter and Kloti, 1972) and grapes (Stern et al., 1967). What is immediately obvious and highly intriguing about this list of previous reports of these esters is that all apply to tropical or subtropical fruits. The results described here for wood apple augment this compilation, and it may even be that these particular esters might prove characteristic of a particular type of tropical fruit flavor.

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#### LITERATURE CITED

- Au-Yeung, C. Y.; MacLeod, A. J., unpublished experiments, 1980. Creveling, R. K.; Silverstein, R. M.; Jennings, W. G. J. Food Sci.
- 1968, *33*, 284–287.
- Heinz, D. E.; Jennings, W. G. J. Food Sci. 1966, 31, 69-80.
- Likens, S. T.; Nickerson, G. B. Proc. Am. Soc. Brew. Chem. 1964, 5-13.

MacLeod, A. J.; Cave, S. J. J. Sci. Food Agric. 1975, 26, 351-360.

- Moshonas, M. G.; Shaw, P. E. J. Agric. Food Chem. 1971, 19, 119–120.
- Moshonas, M. G.; Shaw, P. E. J. Agric. Food Chem. 1972, 20, 70-71.
- Murray, K. E.; Shipton, J.; Whitfield, F. B. Aust. J. Chem. 1972, 25, 1921–1933.
- Scheide, J.; Bauer, K. British Patent 1069810, 1967.
- Stanley, W. L.; Brekke, J. E.; Teranishi, R. U.S. Patent 3 113031, 1963.
- Stern, D. J.; Lee, A.; McFadden, W. H.; Stevens, K. L. J. Agric. Food Chem. 1967, 15, 1100–1103.
- Winter, M.; Kloti, R. Helv. Chim. Acta 1972, 55, 1916-1921.

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# Extractability of Nitrogenous Constituents from Iraqi Mung Bean As Affected by pH, Salt Type, and Other Factors

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Factors influencing nitrogen extractability (pH, temperature, time, and flour to solvent ratio) were studied extensively, and the extraction profile was determined with every variable when keeping other variables constant. In addition, the effects of seven salts in wide ranges of concentrations on nitrogen dispersibility were investigated. The maximum nitrogen extractabilities attained by these salts, arranged on a decreasing order, are as follows: 87.58% with 0.100 N sodium carbonate, 82.22% with 0.025 N sodium sulfite, 79.93% with 0.050 N sodium citrate, 78.78% with 0.500 N sodium chloride, 78.40% with 0.250 N sodium oxalate, 74.95% with 0.010 N sodium acetate, and finally 74.80% with 100 mg/L Na<sub>2</sub>EDTA compared with 79.00% with distilled water. Moreover, when the trends of the nitrogen extractability patterns obtained for the seven salts were compared, it was possible to divide them into three general types.

Nitrogen dispersibility for many legume seeds in aqueous solutions of hydrochloric acid and sodium hydroxide at different pH values was studied by numerous investigators (Evans and Kerr, 1963; Pusztai, 1965; Pant and Tulsiani, 1969, Hang et al., 1970; Fan and Sosulski, 1974; Coffmann and Garcia, 1977; Thompson, 1977). Their results indicated that the highest nitrogen dispersibility occurred at pH 1.0–2.0 in the acidic range and above pH 7.0 in the basic range with minimum solubility occurring in the pH range 4.0–5.0. Moreover, Hang et al. (1970) concluded that the amount of nitrogen extracted from mung beans at alkaline pH was greater than that produced at their neutral or acidic pH values and the minimum point of nitrogen dispersion occurred at pH 4.0. However, recovery from soybeans and horsebeans was increased by increasing the temperature to 45 °C and by efficient

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