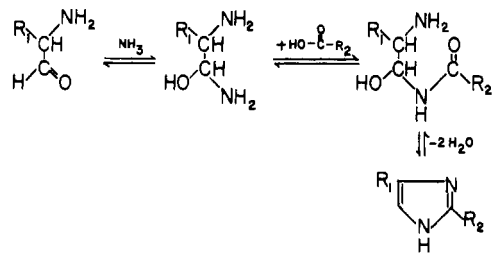
Scheme II



R_1: H, CH_3
R_2: H, CH_3, C_2H_5, CHO

pounds have been positively identified and 20 compounds tentatively identified. Most compounds have been found in foods or sugar–amine model systems with the exception of 2-methyloxazole, 2-ethylpyrrole, 2-ethyl-5-methyl-6,7-dihydro-5*H*-cyclopentapyrazine, 4,5-dimethyloxazole-2-carboxaldehyde, 5-methyl-5*H*-cyclopentapyrazine, 2-ethyl-5*H*-cyclopentapyrazine, 2-ethyl-3-methyl-5,8-dihydroquinoxaline, and 2-amino-5-methylpyridine.

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Flavor and Odor Thresholds in Water of Selected Orange Juice Components

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Odor and flavor thresholds in water were determined for four hydrocarbons, five alcohols, 13 aldehydes, six esters, and two ketones believed important to orange and other fruit flavors. In most cases no significant differences were found between odor and flavor threshold values. By correlating flavor threshold with level reported in orange juice, where available, the relative contribution of individual compounds to orange flavor was assessed. A comparison between these threshold values and previously reported threshold values in water showed generally good agreement with a few exceptions.

Although many common fruit owe their characteristic flavors to the aliphatic esters that are present, a major contribution to citrus flavors comes from the peel essential oil that contains mostly terpenoids. Over 90% of the essential oil of orange is the monoterpene hydrocarbon, *d*-limonene, but a major contribution to orange flavor is due to the minor oxygenated constituents, especially the aldehydes, esters, and alcohols (Kefford, 1959; Wolford and Attaway, 1967).

Well over 150 volatile compounds have been identified in orange juice or in flavor fractions derived from the juice (Shaw, 1977). Despite the extensive studies to identify volatile flavor components of orange juice, the primary flavor compound or mixture of compounds and their proportions needed for fresh orange flavor remain to be found. The quantities of most of the more abundant components present in orange juice have been estimated, but their significance to flavor has not been determined. Either odor or flavor thresholds have been determined for some volatile hydrocarbons, alcohols, aldehydes, and esters believed important to orange and to other fruit flavors (Berg et al., 1955; Buttery et al., 1971; Flath et al., 1967; Guadagni et al., 1963a; Lea and Swoboda, 1958). However, none of those studies reported a direct comparison between an odor threshold and a flavor threshold for a single compound. The values published for flavor and odor thresholds suffer from a lack of reproducibility, and no one study lists threshold values for most compounds believed important orange flavor. Since variations in threshold values are mainly due to differences in methodology (Pangborn, 1960), one study listing threshold values for all major volatile components of orange would be useful for determining relative flavor intensities, as well as providing flavor threshold values for components for which

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no threshold data are presently available.

In the current study, odor and flavor thresholds in water were determined for volatile hydrocarbons, alcohols, aldehydes, esters, and ketones believed important to orange flavor. A statistical correlation was made between odor threshold and flavor threshold values.

EXPERIMENTAL SECTION

For the purposes of this study, threshold concentration is defined as that concentration of a particular compound at which panelists can detect a difference from a specified standard 50% of the time (Patton and Josephson, 1957). Odor is defined as the response to smelling. Flavor is considered to be the combined taste, odor, and other oral sensations of a panelist when the sample is taken into the mouth.

Sample Preparation. Chemicals used were obtained from several sources; citral, decanal, dodecanal, ethanal, hexanal, nonanal, octanal, *trans*-2-hexenal, linalool, octanol, decanol, dodecanol, ethyl butyrate, and ethyl propionate were purchased from Aldrich Chemical Co. (Milwaukee, Wis.); butanal and *d*-limonene were purchased from Eastman Kodak Co. (Rochester, N.Y.); α-terpeniol, methyl butyrate, octyl acetate, and nonyl acetate were purchased from K & K Laboratories, (Plainview, N.Y.); and the remaining compounds used in this study were donated by the U.S. Citrus and Subtropical Laboratory (Winter Haven, Fla.). Aldehydes were stored at 1 °C, except when used, because of their instability at higher temperatures. Purity of these compounds were checked by gas–liquid chromatography prior to their use in the flavor tests. Purities were determined on a Varian Aerograph Model 1520 gas chromatograph equipped with a flame ionization detector. A 0.125 in. × 8 ft o.d. copper column packed with 10% Apiezon L on 60/80 mesh Chromosorb W/AW operating at 100 °C with a flow rate of 40 mL/min of helium was used. The injection port and detector temperatures were 206 °C. Samples of 1.0 μL each were injected, and the purity of the compound was calculated by dividing the area under the major peak by the total area of all peaks. All compounds were greater than 99.9% pure with the following exceptions: *d*-limonene, 96.5%; α-terpineol, 97%; *trans*-2-hexanal, 98.0%; and citral, a mixture of 67.0% geranial and 33.0% neral. These determined purities might have been lower if a more sensitive gas chromatograph system was used.

To prepare an aqueous solution of each compound, the chemical was first dissolved in a small amount of absolute ethanol, since some of the compounds tested were relatively insoluble in water (Guadagni et al., 1963a). The water used in this study was double-glass distilled and boiled for at least 1 h prior to use. Wide-mouthed amber glass bottles (56 cm^3) that were individually capped with a 5.1-cm square piece of Parafilm (American Can Co., Neenah, Wis.) were used as described previously (Schinneller et al., 1972).

Testing Procedures. A large group of 55–73 unscreened and untrained panelists was used as recommended by Kramer et al. (1961), Meijboon (1964), and Keith and Powers (1968) for determining thresholds. Odor and flavor threshold concentrations were determined at the rate of approximately two/month. Since the investigation lasted approximately 2 years there was some turnover (20–30%) in panel members. Thus, panelists determining odor thresholds were not necessarily the same as those determining flavor thresholds. In all cases, panels were replicated a sufficient number of times, so that a minimum of 100 responses were obtained for each concentration used in determining a particular threshold. There were 53 female and 26 male panelists ranging in age from 19 to 66 years with a mean of 27 years. Twenty-five percent of the panelists were smokers.

Threshold values for the aldehydes (Table I) were determined using the multiple paired comparison test employed by Guadagni et al. (1963a). Each pair of samples contained a reference and one dilution of the aldehyde under investigation. Five pairs were presented at each session. The concentrations were presented in ascending order as recommended by Gregson (1962) to reduce carryover effects. Panelists were asked to sample each pair in order and indicate if the samples were alike or different.

Threshold values for the remaining compounds (Table II) were determined using the single stimulus difference test employed by Siek et al. (1969) and Langler and Day (1964). The test involved presenting the panel members with several samples along with a standard of water for reference. Each sample was compared individually to the reference to determine if there was a difference between the two. Six samples were presented to each panelist during each session. The first bottle was a reference and contained only double-distilled water. The next five bottles consisted of the four different dilutions and a water sample identical with the reference. The four dilutions were placed in order of increasing concentration to prevent fatigue from a strong concentration masking a lesser dilution. The position of the water sample between different samples was arbitrarily changed from day to day. This method of sample presentation approximates the paired comparison test with regard to comparison of each sample with the reference. Probably a better term for this method should be multiple paired comparison test.

The testing area consisted of individual testing booths each equipped with its own light source using orange lighting of relatively low intensity. Air temperature was 24–27 °C and the humidity was 50–55%.

Statistical Analyses. The statistical analyses for determining the threshold values involved predicting the concentration that corresponded to 50% positive responses from the 100 judgements (Patton and Josephson, 1957). The prediction was made from the regression of Y (percent of detection) on X (log of concentration). The 95% confidence limit (Snedecor and Cochran, 1967) calculated for the threshold values was used as a measure of error. Significance of differences between odor and flavor thresholds were determined by the "Student's t" test (Snedecor and Cochran, 1967).

RESULTS AND DISCUSSION

Odor and flavor threshold levels were determined for aqueous solutions of hydrocarbons, alcohols, aldehydes, esters, and ketones believed to contribute to orange flavor. These threshold values are listed in Tables I and II with the 95% confidence limits. Also listed in these tables are previously reported threshold values for these compounds and amounts present in orange juice, where available.

The majority of panel members (70–80%) participated in both odor and flavor threshold tests, thus cross-comparisons between odor and flavor threshold results could be made. In all but four of the 30 compounds studied, there was no significant difference between odor and flavor thresholds. Octanal and citral had significantly higher odor thresholds when compared to the corresponding flavor threshold values. With nonanal and *trans*-2-hexenal, the flavor threshold was the higher value. Thus, for most of these compounds that are believed to be contributors to orange flavor, the flavor threshold values alone can be used to relate flavor threshold levels and in conjunction with the amounts present in orange juice to ascertain their

Table I. Odor and Flavor Thresholds in Parts per Billion of Selected Aldehydes in Water

Aldehyde	Odor threshold			Flavor threshold			Concn (ppb) in orange juice
	Probable threshold	Confidence limits (95%)	Reported values	Probable threshold	Confidence limits (95%)	Reported values	
Ethanal	17.0	13.4–21.7	4,[a] 15,[b] 21[c]	22.0	19.8–24.4	1500,[d] 1300[a]	3000[e]
Butanal	15.9	4.20–60.2	9,[a,f] 70[g]	5.26	4.42–6.27	70[h]	
Hexanal	9.18	1.43–58.9	4.5,[a] 5[b]	3.66	1.37–6.78	16,[j] 30,[h] 5[a]	40[e]
Octanal	1.41[k]	1.24–1.60	0.7[l,a]	0.52[k]	0.50–0.54	5[h]	60[e,l]
Nonanal	2.53[m]	2.12–3.02	1[f,a]	4.25[m]	3.19–5.64		9.2[n]
Decanal	1.97	0.79–4.90	0.1,[f] 0.3[a]	3.02	2.67–3.41	7[h]	70,[l] 2[n]
Dodecanal	0.53	0.30–0.95	0.2[f]	1.07	0.89–1.29	0.94[h]	7.9,[n] 8.5[b]
trans-2-Hexenal	24.2[m]	19.9–29.5	17[b,f]	49.3[m]	31.0–78.4		
Citral	85.3[k]	76.8–94.8	60[a]	41.4[k]	36.3–47.3		14[o]
Geranial			32,[g] 60[p]	40			17[l]
Citronellal	66			35			40[e]
Perillaldehyde	30.1	19.4–46.9		25.3	16.7–38.3		
β-Sinensal	3.8		0.2[q]	3.8			

[a] Stahl, 1973. [b] Flath et al., 1967. [c] Laffort, 1963. [d] Berg et al., 1955. [e] Kirchner and Miller, 1957. [f] Guadagni et al., 1963a. [g] Buttery et al., 1971. [h] Lea and Swoboda, 1958. [i] Weurman, 1963. [j] Siek et al., 1969. [k] Flavor threshold significantly lower than odor threshold at 95% confidence level. [l] Lifshitz et al., 1970. [m] Flavor threshold significantly higher than odor threshold at 95% confidence level. [n] Stanley et al., 1961. [o] Naves, 1947. [p] Kendall and Neilson, 1966. [q] Guadagni, 1965.

influence on orange flavor. Some research workers in sensory threshold methodology recommended the use of trained panelists while others recommended the use of untrained panelists. The flavor threshold value of *d*-limonene, as determined with untrained panelists (Table II), was identical with its threshold value (0.21 ppm) as determined by a trained panel (Ahmed et al., 1978), indicating good reproducibility between the two types of panelists.

Aldehydes play a major role in orange flavor, and the quality of the orange flavor fractions, aqueous essence, and peel essential oil are related to their aldehyde contents (Wolford et al., 1969; Kesterson et al., 1971). The 13 aldehydes in Table I were selected for study based upon the following information: (1) ethanal and butanal are the only two aldehydes detected in relatively large amounts in aqueous essence (Kirchner and Miller, 1957); (2) the most abundant aldehydes in the essential oil are octanal, nonanal, decanal (Moshonas and Lund, 1969), citronellal and geranial (Shaw and Coleman, 1974); (3) hexanal and *trans*-2-hexenal contribute an immature or "greenish" note to various fruits (Flath et al., 1967; Buttery et al., 1971); (4) citral, which is about a 2:1 mixture of geranial and neral, has long been associated with the flavor and aroma of citrus; (5) perillaldehyde is important to the flavor of *Citrus natsudaidai* (Ohta and Hirose, 1966); (6) β-sinensal has a very low odor threshold and a sweet, pungent aroma (Stanley, 1965).

The threshold values reported previously (Table I) for these aldehydes were in generally close agreement with those found in this study with a few exceptions. The reported flavor threshold for ethanal was 100 times higher (Berg et al., 1955), and those for butanal and octanal were 10 times higher (Lea and Swoboda, 1958) than the values found in the present study. The previously determined odor thresholds for β-sinensal (Guadagni, 1965) and decanal (Guadagni et al., 1963a) were 20 times lower than those found in this study. Disagreement in threshold values of some orange volatiles in the literature and those obtained in the present study could be due to differences in solvent polarity and method and order of sample presentation to the panelist. A nonpolar solvent was used by Meijboon (1964) vs. a polar solvent in the present study. Polarity of the solvent influences the vapor pressure of the solute (Morrison and Boyd, 1966), degree of affinity between solvent and solute (Klopping, 1971), shape of solute molecule (Amoore et al., 1964), and fatiguing effect of sensory receptor sites (Zotterman and Diament, 1959). These factors result in higher threshold values for a given compound present in a nonpolar solvent than the values for a polar solvent. Buttery et al. (1971) gently forced volatile compounds present in the headspace above solutions into the nasal cavity of the panelist while a sniff test was used in the present study. High concentration of the volatile compound was presented first to the panelist followed by decreasing concentrations until no panelist could detect the presence of this compound (Keith and Powers, 1968); while the reverse was true for the present study. If fatigue from the high concentration occurred, lower concentrations were not detected, thus resulting in a high threshold value.

For the aliphatic aldehydes, a definite relationship between threshold value and chain length, as stated by Meijboon (1964), could not be concluded from the data. However, as the chain length increased, the thresholds generally decreased (Table I). The aldehydes, octanal and dodecanal, containing carbon atoms in multiples of four, exhibited lower thresholds than would be expected from

Table II. Odor and Flavor Thresholds in Parts per Billion of Selected Hydrocarbons, Esters, Alcohols, and Ketones in Water

Compound	Odor threshold			Flavor threshold			Concn in orange juice, ppb
	Probable threshold	Confidence limits (95%)	Reported values	Probable threshold	Confidence limits (95%)	Reported values	
d-Limonene	60	20-180	10[a,h]	210	140-330		80,000[b]
Myrcene	36		13[c]	42			
α-Pinene	9.5	0.25-359.3	6[c]	1013.8	202.3-4971.6		
p-Cymene	11.4	0.006-16,360.7		13.3	4.0-40.0		
Ethyl butyrate	0.13	0.003-4.6	1[d,h]	0.13	0.0008-2.000	15,[e] 450[f]	
Ethyl propionate	9.9	3.3-29		4.9	0.9-25		
Methyl butyrate	43	15-120		59	23-150		
Methyl propionate	100			58			N[g]
Nonyl acetate	57	19-180		270	120-590		
Octyl acetate	47	14-150	12[c]	210	89-470		
Octanol	190	95-370		54	12-230		210[b]
Decanol	47	12-180		23	9.6-57		100[b]
Dodecanol	73	21-260		66	25-170		
Linalool	5.3	1.9-15	6[a,h]	3.8	1.4-10		930[b]
α-Terpineol	280	31-2000	350[a,h]	300	120-890		320[b]
d-Carvone	2.7	0.001-15454.2		86.0	47.9-151.0		
1-Penten-3-one	0.9	0.009-80.5	1.25[a]	1.2	0.42-3.0		

[a] Buttery et al., 1971. [b] Kirchner and Miller, 1957. [c] Guadagni et al., 1966. [d] Flath et al., 1967. [e] Siek et al., 1969. [f] Keith and Powers, 1968. [g] Not reported as a constituent of orange juice. [h] Stahl, 1973.

examining thresholds of neighboring aldehydes. Amoore et al. (1964) reported that strong odor seemed to be associated with chains of four and eight carbon atoms for certain aldehydes. Nonanal, the only *n*-alkanal tested with an odd number of carbon atoms, had a higher threshold than would have been predicted. Meijboon (1964) reported lower threshold values for aldehydes with an even number than with an odd number of carbon atoms and stated that such alternating effects usually referred to a physical property of crystalline state, perhaps analogous to a physical orientation that might be required for taste and odor perception.

Panelists reported distinctive aromas of flavors for several of the aldehydes tested. They found octanal, nonanal, decanal, and dodecanal to possess an orange-like flavor and aroma and a slightly bitter taste in 1 ppm aqueous solutions. A few panelists mentioned that perillaldehyde in its pure form had a floral, rose-like aroma, while citral possessed a typical lemon-like aroma.

Most of the aldehydes in Table I are present in orange juice at concentrations above threshold levels, with the exception of citral and geranial which possess flavor thresholds lower than the amounts present in orange juice. For four of the aldehydes studied, estimated quantities in orange juice have not been reported. Guadagni et al. (1963b) found that a mixture of unsaturated and saturated aldehydes in aqueous solution can have an additive effect on flavor and that the additivity of subthreshold concentrations of aldehydes appeared to be widespread. Thus, the presence of citral, geranial, and other aldehydes at subthreshold concentrations could have an effect on the flavor of orange juice particularly since octanal is one of the major aldehydes present.

Other oxygenated components (esters, alcohols, and ketones) as well as terpene hydrocarbons (which comprise over 95% of the peel oil) contribute to the flavor or orange juice (Kefford, 1959). The bases for selecting compounds listed in Table II from these classes for study were (1) the hydrocarbons *d*-limonene, myrcene, and α-pinene are the three major constituents of peel oil and *p*-cymene contributes to off-flavor in citrus peel oils; (2) ethyl and methyl butyrates and ethyl propionate are the volatile esters most important to flavor, and nonyl and octyl acetates are two of the more abundant higher molecular weight volatile esters; (3) octanol, decanol, linalool, and α-terpineol are the most abundant alcohols; (4) α-terpineol and *d*-carvone contribute to off-flavor in stored juice; and (5) 1-penten-3-one is a component of the aqueous essence phase that is believed to be important to the flavor of orange juice (Shaw, 1977).

Of the four hydrocarbons evaluated, two contribute greatly to orange flavor; *d*-limonene, which is by far the major component of orange peel oil, is present in juice at 400 times its flavor threshold level in water, and myrcene the next most abundant component of orange oil has a flavor threshold of only 42 ppb but is present at 2% of the concentration found for limonene in orange oil (Shaw and Coleman, 1974). *d*-Pinene has a much lower odor threshold than flavor threshold. Since its amount in orange juice has not been determined, the degree of its contribution to orange flavor is uncertain. Blair et al. (1952) suggested the presence of *p*-cymene in orange juice would be from storage deterioration, and Slater and Watkins (1964) have identified *p*-cymene as one of the major components in distilled lime oil that causes its "reverted" flavor.

Esters also make an important contribution to orange flavor (Kefford, 1959), and the level of total esters in aqueous essence has been used as an estimate of essence strength and quality (Attaway et al., 1967). Of the six esters used in this study (Table II), ethyl butyrate, ethyl propionate, and methyl butyrate probably are important contributors to the odor and taste of orange essence since they all have strong, fruity aromas. Methyl propionate is a volatile constituent of many fruits but has not been identified as a constituent of orange. It was included in this study to help show the relationship between odor and flavor thresholds for volatile methyl and ethyl esters. Thus, the ethyl esters of both propionic and butyric acids tended to have a lower flavor threshold than the methyl ester. Ethyl butyrate had the lowest flavor threshold level of any compound evaluated. Since ethyl butyrate is one of the major esters present in aqueous essence, its contribution to orange flavor is probably substantial. Nonyl acetate and octyl acetate had odor and flavor thresholds higher than those found for the more volatile esters. Since the quantities of these esters present in orange juice have not been established, no definite suggestions about their individual contributions to orange flavor can be made.

For four of the five alcohols included in this study, the concentration in orange juice meets or exceeds the odor

and flavor threshold as shown in Table II. The concentration of the fifth alcohol, dodecanol, has not been estimated in orange juice. The most potent alcohol studied, linalool, had a flavor threshold almost 250 times lower than its reported concentration in orange juice. Its contribution to orange flavor must be substantial. Octanol and decanol are present in orange juice at very near their threshold levels. The concentration of α-terpineol would be expected to vary according to storage and heat treatment of the juice (Blair et al., 1952; Rymal et al., 1968; Kirchner and Miller, 1957), but the amount reported by Kirchner and Miller is near the threshold levels found in this study and by previous workers (Buttery et al., 1971). Tatum et al. (1975) have shown that α-terpineol contributes to the off-flavor that develops in canned orange juice during storage. They found its flavor threshold in orange juice to be 2000 ppb, a level six times higher than that found in water.

Two ketones were evaluated that may be important to orange flavor, *d*-carvone possesses a caraway- or dill-like aroma and flavor that is objectionable when added to synthetic mixtures having orange-like aromas (Shaw, 1971). The level of carvone in orange juice has not been reported, but it is expected to increase during oxidative storage decomposition (Shaw, 1977). 1-Penten-3-one has the lowest odor threshold of any compound tested, and it is believed to contribute to the distinctive aroma of aqueous orange essence (Moshonas and Shaw, 1973). It has a profound influence on the aroma of synthetic oils even when added in trace quantities (Bonasera, 1973).

In this study, a definite contribution to orange flavor was indicated for certain volatile constituents of orange juice. Thus, ethanal, hexanal, octanal, decanal, dodecanal, *d*-limonene, and linalool are reported present in orange juice at concentrations more than eight times their flavor threshold values in water. Certain other components of orange juice, nonanal, citronellal, octanol, decanol, and α-terpineol are present at or slightly above their flavor threshold limits in orange juice and probably contribute significantly to orange juice flavor. A few compounds studied are present in amounts below their threshold values and the quantities present in orange juice for several others have not been determined. Some of the latter, such as β-sinensal, ethyl butyrate, and 1-penten-3-one, have such low flavor thresholds that they probably contribute to orange flavor either directly or through additive or synergistic effects with other orange flavor components.

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