Detection of the Addition of *Citrus reticulata* and Hybrids to *Citrus sinensis* by Flavonoids

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In some European countries there is a legal barrier against the marketing of some citrus juice mixtures under the denomination "orange juice", as allowed under certain conditions by the FDA for pasteurized and canned orange juice and for frozen concentrated orange juice. For this reason methods have to be developed to determine a juice addition of up to 10% *Citrus reticulata* and hybrids thereof and up to 5% *Citrus aurantium*. Using HPLC equipped with a photodiode array (PDA) detector we could establish that a combination of the flavanone glycoside (FG) and polymethoxylated flavone (PMF) pattern offers possibilities. For the flavanone glycosides the method is based on the presence of some specific compounds, on the didymin content, on a divergent ratio of hesperidin on narirutin, and/or on an unidentified component. In other cases, however, an *F* test applied on the relative PMF pattern offers a better solution calculating the % adulteration probability. We could conclude that, for all examined citrus samples added to *Citrus sinensis*, it is possible to detect the addition of 10% *C. reticulata* and hybrids thereof and of 5% *C. aurantium*.

Keywords: *Orange juice; flavonoids; Citrus reticulata addition; hybrid addition*

INTRODUCTION

Orange juice is the most popular and most consumed fruit juice in the world. Actually four commercially important groups of oranges are used in the manufacture of orange juice products: *Citrus sinensis* or sweet oranges, *Citrus reticulata* or mandarins and tangerines, *Citrus aurantium* or sour/bitter oranges, and tangors or hybrids of sweet orange and tangerine as Murcott, Temple, and Topaz. Many varieties of *C. sinensis* such as Hamlin, Navel, Parsons Brown, Pera, Shamouti, and Valencia are grown all over the world.

Most of the European countries do not accept the *Codex Alimentarius* definition of orange juice as such. The *Codex* gives a definition of orange juice and concentrated orange juice and states that both have to be "obtained by a mechanical process from the endocarp of sound, ripe oranges (*Citrus sinensis*), preserved exclusively by physical means. The juice may contain up to 10% (m/m) of mandarin juice (*Citrus reticulata*)" (*Codex Alimentarius*, 1992). The Food and Drug Administration (FDA) in the U.S. permits the addition of 10% (m/m) *C. reticulata* or hybrids thereof to pasteurized and canned orange juice. Moreover, frozen concentrated orange juice also may contain up to 5% (m/m) *C. aurantium* (Rouseff, 1988).

Mandarins and hybrids that have a better taste or color may be added to improve the juice quality of early-season juices. Although the fresh-fruit prices for hybrids are often two or three times that of sweet oranges, rather than wasting unaccepted whole fresh fruit due to size or shape, processors use these culled fruits for juice quality improvement.

In recent years some new citrus hybrids have been developed which yield trees and fruits with improved resistance to frost and blight damage. Subsequently the FDA ruled that the Ambersweet hybrid may be used without restriction in processed juice products (Wade, 1994).

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Nevertheless, most of the countries of the European Union do not allow the addition of non-*C. sinensis* juices, and this is based on Council Directive 93/77/EEC of September 21, 1993. In summary, there is no legal barrier in Europe against the use of mixtures of *C. sinensis* and other citrus juices, but there is a barrier against the marketing of mixtures thereof under the denomination "orange juice", which is exclusively reserved for juices from *C. sinensis* only (Korth, 1994).

In order to keep the discussion about the legal aspects of these additions from remaining just a theoretical one, methods have to be developed to detect these additions. They have to be based on relevant and specific parameters, which, moreover, must be difficult to manipulate. The aim of this paper is to determine the possibilities of flavanone glycosides (FG's) and polymethoxylated flavones (PMF's) as such parameters. Flavonoids are very promising for the determination of the authenticity of citrus juices for a number of reasons: they are ubiquitous, taxonomically very specific, generally not commercially available, and may not be inexpensively synthesized due to their structural complexity (Rouseff et al., 1987; Schnüll, 1990; Wade, 1992). For a review about the flavonoid analysis we refer to Hasegawa et al. (1996).

In two previous papers we have used FG's (Ooghe et al., 1994a) and PMF's (Ooghe et al., 1994b) to characterize orange juice (*C. sinensis*). For the flavanone glycosides we confirmed the findings of Rouseff (1988; Rouseff et al., 1987) that the addition to *C. sinensis* of low percentages of *Citrus paradisi*, *C. aurantium*, and/or *C. bergamia* juice may be detected by the presence of some specific flavonoids not present in sweet oranges, as naringin, neoeriocitrin, neohesperidin, and/or poncirin (Ooghe et al., 1994a). However, those compounds cannot be used to detect the addition of tangerine juice and hybrids thereof. The application of a statistical *F* test on the PMF pattern offers more possibilities, especially to detect additions to *C. sinensis* of tangerine and Murcott juice (Ooghe et al., 1994b).

S0021-8561(96)00626-7 CCC: $14.00 © 1997 American Chemical Society

Table 1. Code, Description, and Species of the Examined Samples

no.	sample code	description	species
1	0	orange juice concentrate, 1996 (Pera)	*C. sinensis*
2	MSBR 2/1	tangerine juice concentrate, Cravo, 1991; (Brazil)	hybrid
3	MSIS 3/1	temple juice concentrate, 1992 (Israel)	tangor hybrid
4	MSIS 7/0	easy-peeled juice concentrate Topaz, 1993 (Israel)	tangor hybrid
5	MSIS 8/1	easy-peeled juice concentrate, Temple, 1994 (Israel)	tangor hybrid
6	MSIS 11/1	easy-peeled juice concentrate, Nova, 1994 (Israel)	tangor hybrid
7	MSIS 17/1	mandarin juice concentrate, 1995 (Israel)	*C. reticulata*
8	MSFL 22/1	mandarin juice concentrate, Temple, 1996 (Florida)	tangor hybrid
9	MSFL 23/1	mandarin juice concentrate, Murcott and Temple, 1996 (Florida)	tangor hybrid
10	KMSAR 001	mandarin juice concentrate, 1995 (Argentina)	*C. reticulata*
11	KMSSP 002	mandarin juice concentrate, 1996 (Spain)	*C. reticulata*
12	KMSSP 003	mandarin juice concentrate, 1996 (Spain)	*C. reticulata*
13	KMSSP 004	mandarin juice concentrate, 1996 (Spain)	*C. reticulata*
14	KMSSP 005	mandarin juice concentrate, 1996 (Spain)	*C. reticulata*
15	KMSSP 006	mandarin juice concentrate, 1996 (Spain)	*C. reticulata*
16	KMSIT 007	mandarin juice concentrate, 1996 (Italy)	*C. reticulata*
17	KMSBR 008	murcott juice concentrate, 1995 (Brazil)	tangor hybrid
18	KMSBR 009	murcott juice concentrate, 1995 (Brazil)	tangor hybrid
19	KMSBR 010	cravo juice concentrate, 1995 (Brazil)	hybrid
20	KMSPA 011	kinno juice concentrate, 1995 (Pakistan)	hybrid

In this paper the investigation is extended with mandarins (*C. reticulata*) and some hybrids of *C. sinensis* as Cravo, Kinno, Temple, and Topaz.

MATERIALS AND METHODS

Description of the Samples. Nineteen juice samples were obtained from the Schutzgemeinschaft der Fruchtsaftindustrie (SGF), Zornheim (Germany): one fresh juice of 13.4 °Brix and eighteen frozen concentrates of *C. reticulata* and hybrids thereof. The code name and a short description of the samples are given in Table 1.

An authentic frozen Pera orange juice concentrate has been used as a reference sample (code 0). It also was used to prepare 19 adulterated samples by addition of 10% (m/m) of each of the 19 non-*C. sinensis* samples. For these adulterated samples the sample code name ends in V10.

Sample Preparation. The concentrates were diluted with demineralized water to 11.2 °Brix, as determined with an Abbe-Zeiss refractometer. Sample 4 (code MSIS 7/0), a fresh juice of 13.4 °Brix, was used as such.

The PMF extraction and the PMF sample preparation have been described in a previous paper; the same reagents and methods were used (Ooghe et al., 1994b). Only the sample preparation procedure for the FG determination has been modified with the use of dimethylformamide in order to enhance the FG solubility: 7.5 mL of juice and 7.5 mL of dimethylformamide (Acros 21058.5000) were mixed in a narrow 35 mL glass tube provided with a glass stopper. After shaking, the tube was placed in a boiling water bath for 10 min. After cooling, the procedure for centrifugation and ultrafiltration was followed as described previously (Ooghe et al., 1994a).

Chromatography. The Waters 600 MS gradient HPLC system equipped with a Waters PDA 991 detector and the chromatography conditions have also been described previously (Ooghe et al., 1994a,b).

The flavanone glycoside standard solution, however, consists here of eight components which elute as follows: 300 mg/L of hesperidin (29.1 min) and 200 mg/L of the glycosides eriocitrin (24.0 min), neoeriocitrin (25.0 min), narirutin (26.8 min), naringin (27.8 min), neohesperidin (30.1 min), didymin (35.4 min), and poncirin (36.5 min). This standard solution is diluted 1.33, 2.00, and 4.00 times in order to obtain four calibration solutions for each component.

RESULTS AND DISCUSSION

Flavanone Glycosides. On the basis of the (relative) retention times and the registered spectra, it was shown that eriocitrin, neoeriocitrin, naringin, neohesperidin, and poncirin are not detectable in the 39 analyzed samples. On the other hand, narirutin, hesperidin, and didymin are always present, although in strongly varying concentrations. Table 2 presents the flavanone glycoside concentrations and also the calculated ratios of hesperidin on narirutin.

Table 2. Flavanone Glycoside Concentration (mg/L) of Narirutin, Hesperidin, and Didymin and Hesperidin/Narirutin Ratio of the Examined Samples[a]

no.	sample code	narirutin	hesperidin	didymin	hes/nat
1	0	74.8	500.9	20.2	6.7
2	MSBR 2/1	56.3	993.8	34.1	17.7*
3	MSIS 3/1	169.3	285.3	61.3*	1.7*
4	MSIS 7/0	145.9	508.9	68.5*	3.5
5	MSIS 8/1	95.3	536.7	13.0	5.6
6	MSIS 11/1	87.9	684.9	39.8	7.8
7	MSIS 17/1	171.5	125.3	65.9*	0.7*
8	MSFL 22/1	94.0	405.1	8.6*	4.3
9	MSFL 23/1	177.4	419.6	43.2	2.4*
10	KMSAR 001	139.1	258.3	50.3*	1.9*
11	KMSSP 002	27.0	407.1	7.0*	15.1*
12	KMSSP 003	25.2	415.3	6.9*	16.5*
13	KMSSP 004	23.1	393.8	6.9*	17.1*
14	KMSSP 005	24.8	415.7	6.5*	16.7*
15	KMSSP 006	23.6	413.7	7.6*	17.5*
16	KMSIT 007	49.0	459.4	15.3	9.4
17	KMSBR 008	186.1	335.7	50.0	1.8*
18	KMSBR 009	245.7	168.5	63.2*	0.7*
19	KMSBR 010	78.8	840.0	32.4	10.7
20	KMSPA 011	155.6	493.3	123.3*	3.2

[a] Values marked with asterisks are not acceptable as *C. sinensis*.

Moreover, most chromatograms at 280 nm show two important unidentified peaks with relative retention times (t_{RR}) of 0.70 and 0.75 compared to hesperidin ($t_{RR} = 1.00$). The sum of the relative peak areas at 280 nm of both peaks even amounts to 18.2% for sample 7 (code MSIS 17/1), as presented in Figure 1. Further, it is striking that all samples, with relative peak areas above 10% for the sum of both peaks, have a didymin concentration of at least 50 mg/L and a hesperidin/narirutin ratio lower than 3.

For samples 7 (code MSIS 17/1) and 18 (code KMSBR 009) the narirutin content even is higher than the hesperidin content. Compared to orange juice with a narirutin content of 49.0 ± 27.6 mg/L (Ooghe et al., 1994a), all samples examined, with the exception of the mandarin juices, show rather high narirutin values. All Spanish mandarin juices (samples 11−15) are characterized by a low narirutin value (<30 mg/L), which gives rise to a hesperidin/narirutin ratio of 15 or more.

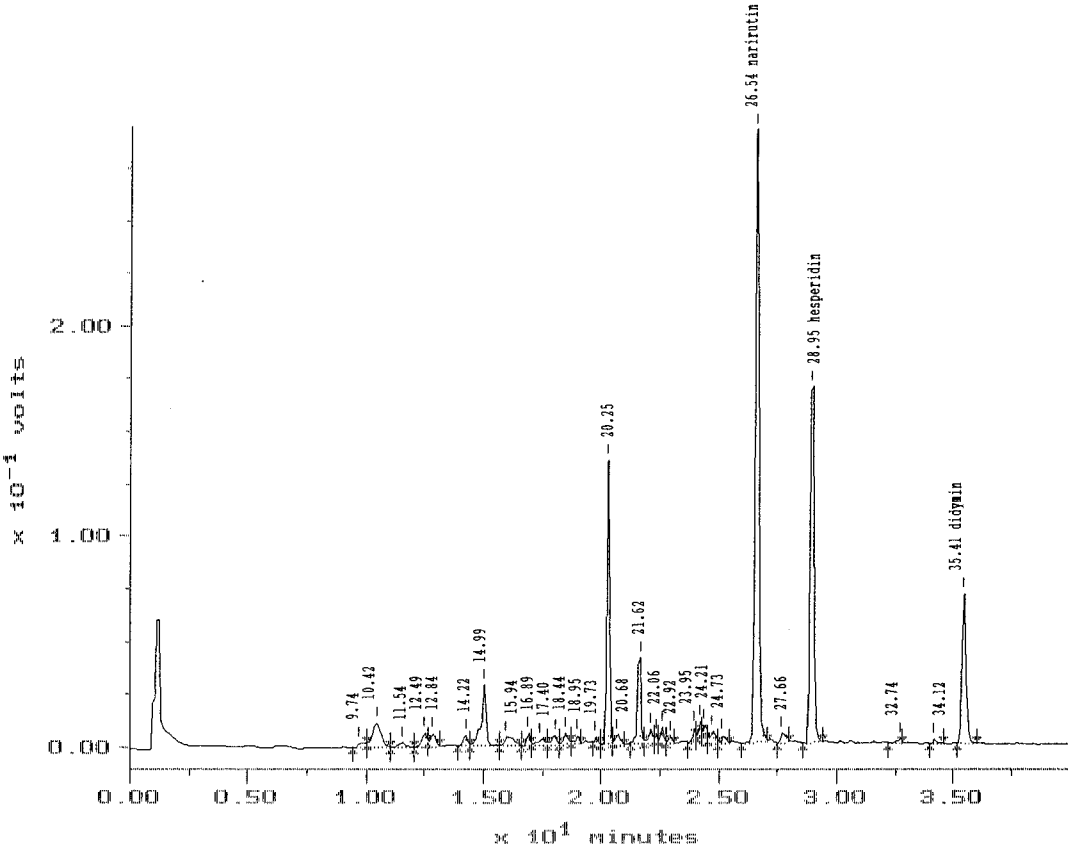

Figure 1. Flavanone glycoside HPLC chromatogram at 280 nm of a *C. reticulata* juice, containing two important unknown peaks at $t_{RR} = 0.70$ and 0.75.

The didymin values for *C. sinensis* range from 14 up to 30 mg/L with an average value of about 21 mg/L (Ooghe, unpublished data). Didymin values below 10 or above 50 mg/L may be considered as not specific for *C. sinensis* and this certainly when they are combined with a relative peak area of more than 5% for the peak with t_{RR} of 0.70 and with a low (<3) or high (>15) hesperidin/narirutin ratio. When these three rules are applied, only the FG's of reference sample 1 (code 0) and also of samples 5 (code MSIS 8/1), 6 (code MSIS 11/1), 16 (code KMSIT 007), and 19 (code KMSBR 010) are comparable to the FG's of *C. sinensis*.

So it is observed that it is not always possible to differentiate between *C. sinensis* and *C. reticulata* and hybrids thereof by the flavanone glycosides only. Moreover, the differentiation becomes more difficult when small amounts of such non-*C. sinensis* juices are present. Therefore a complementary method was developed in order to be able to detect even 10% additions to *C. sinensis*.

Polymethoxylated Flavones. As mentioned in a previous paper (Ooghe et al., 1994b), we could define a PMF standard for *C. sinensis* based on the relative PMF values at 340 nm of the seven following peaks: an unidentified peak with $t_{RR} = 0.40$ (X), sinensetin (SIN), quercetagetin (QUE), nobiletin (NOB), heptamethoxyflavone (HEP), scutellarein (SCU), and tangeretin (TAN), which is also used as a peak identification component ($t_{RR} = 1$). This PMF reference standard is based on 51 authentic orange juices, after elimination of 3 outliers using a statistical *F* test (99.5% probability), and is characterized by the 7 average relative peak areas and standard deviations at 340 nm and by 10 ratios, as presented in Table 3.

Table 3. Average Relative Peak Areas (Ratios) and Standard Deviations of Seven PMF's at 340 nm for Authentic Orange Juice ($N = 51$)

PMF	$\bar{x} \pm \sigma$
X	1.345 ± 0.179
SIN	26.779 ± 1.969
QUE	6.104 ± 1.056
NOB	34.576 ± 1.751
HEP	12.555 ± 2.232
SCU	11.962 ± 1.533
TAN	6.678 ± 1.215
PMF ratios	$\bar{r} \pm \sigma$
SIN/QUE	4.496 ± 0.707
SIN/HEP	2.201 ± 0.423
SIN/TAN	4.169 ± 0.942
NOB/SIN	1.299 ± 0.127
NOB/HEP	2.845 ± 0.550
NOB/SCU	2.935 ± 0.380
NOB/TAN	5.323 ± 0.867
HEP/SCU	1.082 ± 0.324
HEP/TAN	1.955 ± 0.539
TAN/QUE	1.152 ± 0.348

These seven PMF's seem to be present in measurable amounts in all examined samples, with the exception, however, of quercetagetin which is present in trace amounts in sample 20 (code KMSPA 011). Further, most of the examined chromatograms do not consist only of the 7 PMF's considered, but also some rather small unidentified peaks are present with t_{RR} values of 1.05, 1.19, and 1.22. The most important one is present in sample 19 (code KMSBR 010) at t_{RR} 1.19 and reaches 3.75% of the total PMF peak area at 340 nm. The PMF results obtained for the 39 examined samples are presented in Table 4.

In this way it is possible to compare these samples by means of an *F* test to the seven ($\bar{x} \pm \sigma$) values for authentic orange juice (see Table 3). In general, the

Table 4. Relative PMF Peak Areas at 340 nm of the Examined Samples (1−20) and of Their 10% (m/m) Addition (22−40) to *C. sinensis*

no.	sample code	X	SIN	QUE	NOB	HEP	SCU	TAN
1	0	1.42	26.56	6.48	34.24	12.61	12.75	5.94
2	MSBR 2/1	0.95	4.44	0.70	47.73	14.62	3.26	28.28
3	MSIS 3/1	2.53	12.09	0.76	45.33	5.03	14.19	20.07
4	MSIS 7/0	2.53	16.38	1.39	39.59	7.34	19.54	13.23
5	MSIS 8/1	7.65	30.37	1.53	48.59	2.27	5.84	3.75
6	MSIS 11/1	5.26	6.40	0.78	58.42	15.01	2.09	12.05
7	MSIS 17/1	2.72	7.71	0.20	60.18	5.75	1.82	21.61
8	MSFL 22/1	7.80	27.44	1.63	50.07	3.71	5.00	4.36
9	MSFL 23/1	5.80	19.86	0.91	55.70	4.25	3.71	9.76
10	KMSAR 001	3.11	9.37	0.83	59.48	5.90	3.37	17.94
11	KMSSP 002	1.91	9.40	7.03	29.50	32.53	9.63	10.01
12	KMSSP 003	1.84	9.65	7.17	29.57	32.29	9.17	10.30
13	KMSSP 004	1.85	10.84	7.39	28.88	31.24	9.71	10.08
14	KMSSP 005	1.96	10.68	7.52	29.11	30.64	10.19	9.90
15	KMSSP 006	1.85	10.46	7.25	29.17	31.03	9.58	10.65
16	KMSIT 007	2.63	7.46	1.35	57.28	9.81	2.72	18.76
17	KMSBR 008	1.94	15.79	2.66	50.35	8.17	6.54	14.53
18	KMSBR 009	1.97	8.39	0.72	59.68	5.44	3.03	20.78
19	KMSBR 010	1.23	5.44	0.55	49.47	10.28	3.45	29.58
20	KMSPA 011	7.31	6.26	0.01	65.23	3.79	0.69	16.71
22	MSBR 2/1 V10	1.29	22.36	5.33	37.00	12.62	11.32	10.08
23	MSIS 3/1 V10	1.85	19.66	3.74	39.64	9.01	13.77	12.39
24	MSIS 7/0 V10	1.73	22.92	4.83	36.40	10.39	15.59	8.13
25	MSIS 8/1 V10	2.16	27.17	5.86	36.27	11.10	12.13	5.31
26	MSIS 11/1 V10	1.73	24.80	5.97	36.27	13.15	11.76	6.34
27	MSIS 17/1 V10	1.79	19.90	4.25	43.69	9.93	9.33	11.10
28	MSFL 22/1 V10	2.05	26.50	5.94	36.15	11.34	12.54	5.48
29	MSFL 23/1 V10	2.07	25.87	5.70	37.62	11.59	11.15	5.99
30	KMSAR 001 V10	1.88	24.44	5.83	36.20	11.80	11.62	8.24
31	KMSSP 002 V10	1.74	23.28	6.74	31.92	17.03	11.79	7.49
32	KMSSP 003 V10	1.76	22.66	6.42	32.22	16.42	12.55	7.97
33	KMSSP 004 V10	1.86	23.90	6.57	32.30	15.74	12.12	7.51
34	KMSSP 005 V10	1.87	23.36	6.60	32.05	15.66	12.70	7.75
35	KMSSP 006 V10	1.90	23.82	6.69	32.06	15.49	12.48	7.56
36	KMSIT 007 V10	2.04	20.77	4.95	41.10	11.23	9.60	10.32
37	KMSBR 008 V10	1.72	25.47	6.15	34.46	11.84	12.37	7.98
38	KMSBR 009 V10	1.88	23.30	5.49	37.14	11.51	11.42	9.26
39	KMSBR 010 V10	1.60	24.02	5.74	34.97	12.29	11.27	10.11
40	KMSPA 011 V10	3.09	22.04	5.01	40.53	10.24	10.01	9.08

Table 5. *F* Value and Adulteration Probability of 19 Non-*C. sinensis* Juices and of 19 Adulterated Orange Juices Based on 7 Relative PMF Values

no.	sample code	*F* value	% probability	no.	sample code	*F* value	% probability
1	0	0.44	15.10	22	MSBR 2/1 V10	10.15	100.00
2	MSBR 2/1	368.77	100.00	23	MSIS 3/1 V10	43.76	100.00
3	MSIS 3/1	230.25	100.00	24	MSIS 7/0 V10	7.50	99.99
4	MSIS 7/0	75.43	100.00	25	MSIS 8/1 V10	9.30	100.00
5	MSIS 8/1	675.99	100.00	26	MSIS 11/1 V10	5.05	99.95
6	MSIS 11/1	771.73	100.00	27	MSIS 17/1 V10	62.08	100.00
7	MSIS 17/1	577.63	100.00	28	MSFL 22/1 V10	7.55	99.99
8	MSFL 22/1	776.34	100.00	29	MSFL 23/1 V10	12.55	100.00
9	MSFL 23/1	638.00	100.00	30	KMSAR 001 V10	14.27	100.00
10	KMSAR 001	518.50	100.00	31	KMSSP 002 V10	5.95	99.98
11	KMSSP 002	57.54	100.00	32	KMSSP 003 V10	7.25	99.99
12	KMSSP 003	56.74	100.00	33	KMSSP 004 V10	7.40	99.99
13	KMSSP 004	49.61	100.00	34	KMSSP 005 V10	8.29	100.00
14	KMSSP 005	53.95	100.00	35	KMSSP 006 V10	8.25	100.00
15	KMSSP 006	54.65	100.00	36	KMSIT 007 V10	58.09	100.00
16	KMSIT 007	463.31	100.00	37	KMSBR 008 V10	5.83	99.98
17	KMSBR 008	168.49	100.00	38	KMSBR 009 V10	22.17	100.00
18	KMSBR 009	434.81	100.00	39	KMSBR 010 V10	13.42	100.00
19	KMSBR 010	450.94	100.00	40	KMSPA 011 V10	109.76	100.00
20	KMSPA 011	very large	100.00				

non-*C. sinensis* juices (samples 2−20) differ from these PMF values by a smaller relative peak area for sinensetin, quercetagetin, heptamethoxyflavone, and scutellarein and by a larger peak area for nobiletin and tangeretin. Combining a smaller and a larger relative PMF value, the most relevant ratios to find out non-*C. sinensis* additions are SIN/TAN, NOB/SIN, NOB/HEP, NOB/SCU, HEP/TAN, and TAN/QUE.

We also could establish that, when only two or even one of the 10 PMF ratios mentioned are not within the limits defined by ($\bar{r} \pm \sigma$), there is a suspicion of adulteration. So it is recommended to perform the *F* test even when only one ratio is deficient. By means of this *F* test the % probability may be calculated to determine the acceptability of the 39 examined samples as a 100% authentic sweet orange juice. The larger the *F* value obtained, the higher the probability that the juice considered is not an authentic *C. sinensis* juice.

The results of the *F* test, as well those obtained for the 20 samples and those obtained for the 10% additions

to *C. sinensis*, are presented in Table 5. For the reference sample 1 (code 0) an *F* value of 0.439 is obtained corresponding to a very low adulteration probability of 15.1%, indicating that this sample is an authentic sweet orange juice.

From Table 5 it also may be clear that all *C. reticulata* juices and the hybrids thereof examined may be differentiated from *C. sinensis* as a result of the very high *F* values, indicating an adulteration probability of 100.0%. Moreover, we also obtained elevated *F* values for the 19 self-prepared adulterated juices, indicating an adulteration probability of at least 99.9%.

In summary, using an *F* test it is possible to detect all adulterations of an authentic sweet orange juice with 10% (m/m) or more of the 19 citrus juices examined in this study and belonging to *C. reticulata* or hybrids thereof.

CONCLUSION

In a previous paper (Ooghe et al., 1994a) we established that in the U.S. the FDA-allowed addition of *C. aurantium* to frozen concentrated orange juice may easily be detected at a 2% (m/m) level on the basis of the presence of the flavanone glycosides naringin and neohesperidin.

Here we established a statistical interpretation of the relative PMF pattern and the content and ratio of some flavanone glycosides to detect the addition to *C. sinensis* of 10% *C. reticulata* juices and hybrids thereof.

Both the determination of the flavanone glycosides and the pattern of the polymethoxylated flavones offer possibilities to detect the addition to *C. sinensis* of 10−15% from *C. aurantium* and *C. reticulata* and hybrids thereof, as allowed by the *Codex Alimentarius* and/or by the U.S. FDA legislation but not accepted by the European Union under the denomination “orange juice”.

ACKNOWLEDGMENT

We thank the Schutzgemeinschaft der Fruchtsaftindustrie e.V., Zornheim (Germany) for the samples and also Els Bastien for the analytical assistance.

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Received for review August 15, 1996. Accepted February 7, 1997.[⊗]

JF9606262

[⊗] Abstract published in *Advance ACS Abstracts*, April 15, 1997.