The nutritional analysis of fruit juices

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Introduction

The United States Congress enacted the Nutrition Labeling and Education Act of 1990 (NLEA) on November 8, 1990 to help consumers make healthy dietary choices. NLEA mandates significant reform of food labels and requires full nutritional labeling for most packaged food products. The complete regulations were published (1) and a May 8, 1994 compliance deadline was established. The mandatory and voluntary NLEA nutrients are listed in Table 1. Part of the analytical challenge from NLEA results from the large numbers of samples to be tested and widely divergent levels of the elements of interest. New methodology developed for NLEA analysis should be generic and applicable to many elements and matrices simultaneously to allow effective sample throughput. This report discusses an Inductively Coupled Plasma-Optical Emission Spectroscopy (ICP-OES) method for the determination of NLEA metals and minerals in juice.

Experimental

Juice Sample Preparation

Single serving containers of ready-to-drink juices were used for this study. One container each of Florida orange juice, Florida grapefruit juice, cranberry juice cocktail, apple-cranberry juice cocktail, and orange-strawberry-banana juice beverage were sampled for this study. Four subsamples of each juice were prepared by microwave digestion using a MDS 2100 microwave system (CEM, Matthews, NC). Each juice was well shaken, opened and sampled by pouring directly from the original container into the microwave vessel. Teflon® PFA microwave vessels rated to 120 psig (CEM, Matthews, NC) were used for the digestion and closed using the Capping Station (CEM, Matthews, NC).Cranberry juice cocktail was selected for spiking to monitor recoveries. Samples were spiked at levels that were predicted for the diluted juice, and the spikes were digested concurrently with the juice samples to monitor any elemental losses in the digestion process. Clear, colored solutions resulted for all samples using the following digestion procedure modified from the CEM application note for Fruit Juice and Pulp (2).

Table 1: The Nutrition Labeling Education Act of 1990 Food Labeling Requirements

Mandatory Nutrients	Voluntary Nutrients		
Calories	Calories from Saturated Fat	Vitamin K	Copper
Calories from fat	Calories from Unsaturated Fat	Thiamin	Manganese
Total Fat	Calories from Carbohydrates	Riboflavin	Fluoride
Saturated Fat	Calories from Protein	Niacin	Chromium
Cholesterol	Unsaturated Fat	Vitamin B6	Molybdenum
Sodium	Polyunsaturated Fat	Folate	Chloride
Total Carbohydrates	Monounsaturated Fat	Vitamin B12	
Total Sugars	Sugar Alcohols	Biotin	
Dietary Fiber	Soluble Fiber	Pantothenic Acid	
Protein	Insoluble Fiber	Phosphorus	
Vitamin A	Protein as %	Magnesium	
Vitamin C	Potassium	Zinc	
Calcium	Vitamin D	Iodine	
Iron	Vitamin E	Selenium	

Digestion Procedure

Step 1: Weigh ≤ 10 g ***single strength*** juice into the Teflon PFA digestion vessels.
Step 2: Add 10 mL concentrated, ultrapure HNO_3 and 2 mL concentrated, ultrapure H_2SO_4 to the sample.
Step 3: Cap the vessel in the capping station.
Step 4: Digest following the Procedure Juice 1 in Table 2.
Step 5: Cool for approximately 5 minutes and vent vessels.
Step 6: Repeat Step 4 for juices containing pulp. Otherwise proceed to Step 5.
Step 7: Cool for 5 minutes, vent, and open the vessels using the capping station.
Step 8: Add 3 mL 30% H_2O_2 to the samples.
Step 9: After effervescence subsides, transfer the samples into clean, acid washed volumetric flasks and dilute to 100 ml with 18 megohm, distilled, deionized H_2O.
Step 10: Transfer the samples to clean high-density polyethylene bottles.

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Table 2: Microwave Digestion Program for Juice Analysis

Parameter	Stage 1	Stage 2	Stage 3	Stage 4	Stage 5
Power (%)	10	20	0	15	0
Power (watts)	51	141	0	96	0
Pressure (psig)	20	50	20	80	0
Run Time (min)	2	5	2	15	5
Time at Parameter (min)	2	5	2	15	5
Fan Speed (% of maximum)	100	100	100	100	100

All analyses were performed on a PerkinElmer Optima 3000™ ICP-OES equipped with the standard torch assembly, conventional cross flow nebulizer and AS-90 autosampler. The Optima 3000 operating conditions are listed in Table 3 and the wavelength and background correction points are listed in Table 4. No attempt was made to optimize plasma conditions for any particular analyte or to optimize the procedure for the best sample throughput.

Table 3: Instrument Conditions for the Optima 3000 Radial ICP-OES

Parameter	Setting
RF Power (watts)	1100
Nebulizer Flow (L/min)	0.950
Auxiliary Flow (L/min)	1.0
Plasma Flow (L/min)	15
Sample Flow (mL/min)	1.0
Source Equilibration Time (sec)	15
Viewing Height (mm)	15
Background Correction	Manual Selection of Points
Measurement Processing Mode	Area
Auto Integration (sec)	1 min – 50 max
Read Delay (sec)	45
Rinse Delay (sec)	45
Number of Replicates	3

Table 4: Wavelength Selections and Background Correction Points for Juice Analysis

Element	Wavelength (nm)	Lower Bgc	Upper Bgc
Ca	396.845	0.000	0.036
Cr	205.561	0.000	0.019
Cu	224.702	0.014	0.021
Fe	238.200	0.029	0.029
K	766.515	0.000	0.116
Mg	279.553	0.000	0.023
Mo	202.034	0.012	0.012
Na	589.589	0.055	0.055
P	177.436	0.011	0.011
Se	196.025	0.015	0.000
Zn	206.198	0.012	0.013

Results

No Cr, Mo or Se was detected in any juice product although the spike recoveries were acceptable for these elements. Spike recoveries are presented in Figure 1. Recoveries ranged from a low of 96.1% for Mg to a high of 106.1% for Se in the blank and 97.7% for Mg to 105.6 for Se in the juice. The orange juice was labeled 100% pure Florida juice and the results suggest that it was indeed from Florida (3). Reference values for Florida orange juice, based on dry ashing, have been included with the experimental data in Table 5. The grapefruit results compare favorably with USDA authentic data (4) and were consistent with product label claims. The poor precision obtained may be attributed to inhomogeneity caused by pulp in the sample. Reference values for grapefruit juice diluted from frozen concentrate, and label claims are presented with the exper-imental data in Table 6. The results obtained for the cranberry juice cocktail and apple-cranberry juice cocktail are consistent with blends of authentic juices and with the label claims and are presented in Table 7. The fruit beverage was labeled Orange Strawberry Banana juice beverage and grape juice, high fructose corn syrup, sucrose and sodium citrate were claimed in the ingredient list. Based on the Ca and Mg results, the beverage appeared to consist of approximately 50% juice. The poor precision for Fe may be due to sample contamination. A conclu-sion of loss due to the microwave digestion process is inconsistent with the results for other juices and the spike recoveries. Authentic orange, strawberry, grape and banana data from USDA (4) and experimen-tal data for the fruit beverage are presented in Table 8.

Conclusion

This work demonstrates that the analysis of metals and minerals stipulated by NLEA may be per-formed in juice using ICP-OES. It is

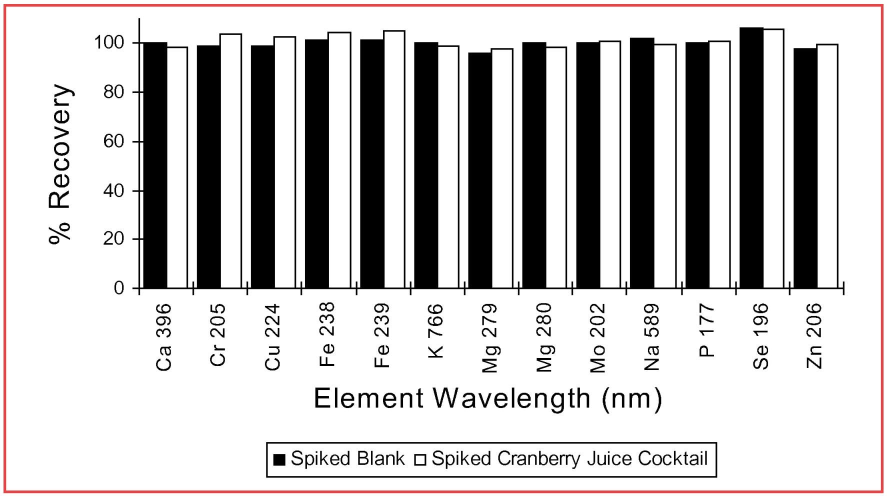

Figure 1: Juice Spike Recoveries

Table 5: ICP-OES Results for Orange Juice Compared With Authentic Florida Orange Juice Reference Values in µg/mL

Element	Wavelength	Experimental	Florida Orange Juice
Ca	396.845	85.3 – 0.4	67 - 123
Cu	224.702	0.43 – 0.01	0.239 - 0.460
Fe	238.200	0.84 – 0.01	0.641 - 5.58
K	766.515	1972 – 10	1520 - 2660
Mg	279.553	103.4 – 0.3	95 - 140
Na	589.589	8.64 – 0.07	3 - 9
P	177.436	206.4 – 2.2	124 - 240
Zn	206.198	0.4 – 0.1	0.242 - 0.480

Table 6: ICP-OES Results for Grapefruit Juice Compared With Package Label Claims and Authentic Grapefruit Juice Reference Values in µg/mL

Element	Wavelength	Experimental	Label	USDA Reference
Ca	396.845	103.2 ± 0.6		80
Cu	224.702	0.33 ± 0.02		0.33
Fe	238.200	0.79 ± 0.06		1.4
K	766.515	1327 ± 4		1360
Mg	279.553	88.3 ± 0.3		110
Na	589.589	23.36 ± 0.02	40.5 max	10
P	177.436	137.5 ± 1.3		140
Zn	206.198	0.9 ± 0.2		0.5

evident that ICP-OES will be an effective tool for the analyst attempting to meet the challenges imposed by NLEA. Microwave digestion has been shown to be a useful sample preparation procedure allowing acceptable precision, spike recoveries, and agreement with authentic values. Trace levels of elements were determined simultaneously with macro levels of nutrient metals and minerals. Sample preparation time, which was the primary limitation to sample throughput, was minimized due to the wide linear dynamic range and useful analytical range of the ICP-OES. All nutrients were determined simultaneously in all samples without multiple dilution/analysis steps. Although no attempts were made to optimize sample throughput, actual instrumental sample throughput was impressive relative to current validated methods.

Table 7: ICP-OES Results for Cranberry Juice Cocktail and Apple Cranberry Juice Cocktail Compared With Package Label Claims and USDA Authentic Juice Reference Values in µg/mL

Element	Wavelength	Experimental Cranberry Juice	Label Cranberry Juice	USDA Cranberry JuiceCocktail	Experimental Apple-Cranberry Juice	Label Apple-Cranberry Juice	USDA Apple Juice
Ca	396.845	31.8 ± 0.5	115 max	30	30.0 ± 0.2	79.1 max	200
Cu	224.702	0.04 ± 0.03		0.13	0.07 ± 0.01		.5
Fe	238.200	1.9 ± 0.1	2.1 max	1.6	1.3 ± 0.1	1.4 max	9.1
K	766.515	193.4 ± 1.5		240	261.8 ± 1.6	237	4480
Mg	279.553	10.6 ± 0.2		30	14.8 ± 0.2		170
Na	589.589	6.2 ± 0.2	57.5	40	20.0 ± 0.1	40 max	250
P	177.436	10.2 ± 0.2		10	21.4 ± 0.1		250

Table 8: ICP-OES Results for the Mixed Fruit Beverage Compared With USDA Authentic Juice and Fruit Reference Values in µg/mL

Element	Wavelength	Experimental	USDA Orange Juice	USDA Grape Juice	USDA Raw Strawberries	USDA Raw Banana
Ca	396.845	55.4 ± 0.7	100	90	140	60
Cu	224.702	0.14 ± 0.03	0.4	0.28	0.49	1.04
Fe	238.200	0.61 ± 0.40	1.7	2.4	3.8	3.1
K	766.515	584.3 ± 7.3	1900	1320	1660	3960
Mg	279.553	41.6 ± 0.4	110	100	100	290
Na	589.589	23.40 ± 0.02	10	30	10	10
P	177.436	137.5 ± 1.3	110	110	190	200
Zn	206.198	0.9 ± 0.2	0.4	0.5	1.3	1.6

References

(1) *Federal Register* **58**, 3 (1/6/93) 632-690, 2066-2964.

(2) CEM Application Note PM-3 (1988) Revision 6-88.

(3) McHard, J.A., Foulk, S.J., Jorgensen, J.L., Bauer, S., & Winefordner, JD. (1980) in *Citrus Nutrition and Quality, ACS Symposium Series*, Vol. 143, S. Nagy, & J.A. Attaway (Eds.) American Chemical Society, Washington D.C., pp. 382-383.

(4) United States Department of Agriculture Human Nutrition Information Service Handbook Number 8-9 (revised 1982), US Government Printing Office, Washington DC, pp 37, 38, 60, 90, 34, 139, 184, 269.

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