Trace Element Determinations in Foods and Biological Samples Using Inductively Coupled Plasma Atomic Emission Spectrometry and Flame Atomic Absorption Spectrometry

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Elemental food composition data are important to both consumers and health professionals, and recent food labeling legislation has highlighted this need. Rugged, accurate, and precise analytical methods are needed for elemental analyses, and atomic spectroscopic techniques are the best choice because of their widespread availability and ease of use. Flame atomic absorption spectrometry (AAS) and inductively coupled plasma atomic emission spectrometry (ICP-AES) methods were compared, focusing on the detection capability, precision, and accuracy obtainable with each technique. Ca, Cu, Cr, Fe, Mg, Mn, and Zn were determined by AAS, and Ca, Co, Cu, Cr, Fe, Mg, Mn, Ni, P, V, and Zn were determined simultaneously using ICP-AES. Detection limits for both techniques were typically in the part per billion range and in all cases were sufficient for the accurate quantitation of elements of nutritional interest. Precisions obtainable with both techniques were similar, and both provided accurate elemental food composition data based on the analysis of four certified reference materials and a variety of foods using either a wet ash or dry ash sample preparation procedure.

Keywords: *Atomic absorption; inductively coupled plasma emission; AAS; ICP; ICP-AES; food composition; elemental analyses*

INTRODUCTION

The Food Composition Laboratory (FCL) located in the Beltsville Human Nutrition Research Center, U.S. Department of Agriculture, conducts research to develop methods to measure constituents in foods which are of interest because of their impact on human health. The focus is on the measurement of analytes that are beneficial to human health (RDA, 1989) including essential trace elements such as calcium, cobalt, copper, chromium, iron, magnesium, manganese, nickel, phosphorus, potassium, sodium, vanadium, and zinc. In 1990, the Nutrition Labeling and Education Act mandated, for the first time, labels providing information about the nutritional content of nearly all processed foods (AOAC, 1993; NFPA, 1994). At the present time, the only trace element content required to be listed on the label is for the elements sodium, calcium, and iron. There is, however, a list of 34 voluntary label nutrients, of which 11 are trace elements including the following: phosphorus, magnesium, zinc, iodine, selenium, copper, manganese, fluorine, chromium, molybdenum, and chlorine. Most likely, labels will soon contain food composition data for many or all of these elements of nutritional interest. Rapid, accurate, and precise analytical methods are necessary for labeling and for food composition databases that are of interest to consumers as well as nutritionists and health professionals doing epidemiological studies.

The methods most suitable for the rapid and accurate determination of the elemental content of foods are atomic spectroscopic methods. Many people work in the field of food analysis, as evidenced by the extensive annual literature reviews in the *Journal of Analytical Atomic Spectrometry* in the section entitled Atomic Spectrometry Update—Clinical and Biological Materials, Food and Beverages (Taylor, 1994, 1995). Here, comprehensive reviews of literature reports on methods focus on the progress for individual elements, sampling and sample preparation, reference materials, and developments in analytical methodology and instrumentation. The techniques reported on include flame atomic absorption spectrometry (AAS), graphite furnace atomic absorption spectrometry (GFAAS), inductively coupled plasma atomic emission spectrometry (ICP-AES), inductively coupled plasma mass spectrometry (ICP-MS), and X-ray fluorescence. When reviewing food composition data, AAS remains the predominant technique used for the majority of the currently available data, but in the last five years, there has been a significant increase in the widespread use of ICP-AES for elemental food composition analyses, most likely owing to the multielement analysis capability ICP-AES provides. Flame AAS and ICP-AES have similar detection limits for most elements, but ICP-AES is better for refractory elements and nonmetals while AAS is better for volatile elements and group 1 metals. At the present time, ICP-MS is not widely used for food analyses but some researchers have developed methods for ultratrace analyses of water, wine, and beverage samples. Others have developed methods for measuring isotope ratios, and development of methods for speciation studies with online coupling with HPLC is underway. As the interest in determining low levels of a wide variety of trace elements increases, advancements in ICP-MS methods will surely be seen.

Elemental analyses performed at FCL are accomplished using atomic spectroscopic techniques such as flame AAS and GFAAS, flame atomic emission spectrometry for the determination of sodium and potassium, and ICP-AES. Although we have an ICP-MS system, the elements of widespread nutritional interest do not typically require the detection capability ICP-MS offers. The focus of this research was the development of rapid, accurate, and transportable methods

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Table 1. Perkin-Elmer Model 5100 PC AAS Operating Conditions[a]

element	wavelength (nm)	HCL current (mA)	slit	gas flow rates (mL/min) acetylene	air	range of std conc (μg/mL)
Ca[b]	422.7	10	0.7	3.5	10.0	0.5−4.0
Cu	324.8	15	0.7	2.6	10.0	0.1−2.0
Cr	357.9	25	0.7	3.5	10.0	0.1−2.0
Fe	248.3	30	0.2	2.5	10.0	0.2−4.0
Mg	285.2	6	0.7	2.5	10.0	0.1−2.0
Mn	279.5	20	0.2	2.8	10.0	0.1−2.0
Zn	213.9	15	0.7	2.4	10.0	0.1−2.0

[a] Sample uptake rate, 5 mL/min; data acquisition, triplicate 0.5 s read for all samples and standards. [b] Ca, standards and samples diluted using 4% (m/v) 8-hydroxyquinoline.

which may be used to determine elemental content of foods and biological samples. Although resources do not permit the routine analyses of large numbers of food samples, analytical methods developed by FCL staff are validated using a wide variety of foods as well as commercial standard reference materials. In addition to development of routine methods, FCL is involved in high-accuracy determinations in support of the development of reference materials and we are often involved in helping with the characterization of both in-house FCL quality control materials and commercial reference materials produced by the National Institute of Standards and Technology (NIST), the International Atomic Energy Agency (IAEA), the National Research Council of Canada (NRCC), and Agriculture Canada. For many years, most work was done using flame AAS (Miller-Ihli, 1988) or graphite furnace atomic absorption spectrometry (GFAAS) (Miller-Ihli, 1989); however, the acquisition of an ICP-AES instrument has allowed us to explore the benefits of multielement analyses. The purpose of this study was to compare AAS and ICP methods developed at FCL. In particular, we were interested in comparing the ease of operation, the detection capability, and the precision and accuracy achievable with each technique. During the course of this work, wet ashing and dry ashing sample preparation procedures were compared while evaluating the ICP-AES and AAS systems. AAS data for 7 elements and ICP-AES data for 11 elements in a wide variety of matrices are presented and compared.

EXPERIMENTAL PROCEDURES

AAS Instrumentation. Flame AAS determinations were made using a Perkin-Elmer Model 5100 PC system equipped with both a deuterium arc background correction lamp used for background correction in the ultraviolet region (180−300 nm) and a tungsten background correction lamp used for background correction in the visible region (300−900 nm). All analyses were performed using an air−acetylene flame. The detailed instrumental conditions are outlined in Table 1. Please note that six multielement standards were prepared to cover the specified calibration range [S1: 0.1 μg/mL Cr, Cu, Mg, Mn, and Zn; 0.2 μg/mL Fe; 0.25 μg/mL Na and K, (S2) 2.5 times concentration of S1; (S3) 5.0 times concentration of S1; (S4) 10 times concentration of S1; (S5) 15 times concentration of S1; (S6) 20 times concentration of S1]. Calibration was accomplished using a linear least-squares fit for all elements but Mg, for which the nonlinear calibration function available on the instrument was used because it provided the optimum fit. Please note that Ca was determined with the use of 8-hydroxyquinoline as a protecting agent to prevent the formation of refractory solute species. In all instances, three 0.5 s readings were made for each sample or standard aspirated.

ICP-AES Instrumentation. All ICP-AES determinations were made on a Leeman Labs PS3000, which is a combination, simultaneous and sequential high resolution, echelle-based ICP-AES system. All determinations were made in the simultaneous mode of operation, and the conditions used are summarized in Table 2. A total of five multielement calibration standards were used to cover the calibration range [(S1) blank; (S2) 0.1 μg/mL Cu, Mn, Zn; 1.0 μg/mL Fe; 5.0 μg/mL P, Mg; 10.0 μg/mL Ca; (S3) 5.0 times concentration of S2; (S4) 10.0 times concentration of S2; (S5) 50 times concentration of S2; (S6) 100 times concentration of S2]. Calibration was accomplished using the weighted linear calibration algorithm provided in the Leeman PS Series software (Version 3.001). In all instances, three 1.0 s readings were made for each sample or standard. The optimization of the viewing height was based on the use of a 10 ppm solution of Mn (257.6 nm) as the peaking element while doing both *X* and *Y* peaking during which time the imaging mirror that focuses the image of the plasma on the entrance slit is moved to optimize the emission signal. Background correction was routinely employed using off-line wavelengths selected on the basis of a wide range of sample scans. The "peak optics" routine on the instrument which adjusts the position of the aperture plate to locate the 296.73 nm Hg reference line, was run at the default interval of every 20 min.

Table 2. Leeman PS3000 ICP-AES Operating Conditions[a]

element	wavelength[b] (nm)	range of std conc (μg/mL)
Ca	317.9 II	10.0−1000
Co	228.6 II	0.1−10.0
Cu	324.8 I	0.1−10.0
Cr	267.7 II	0.1−10.0
Fe	259.9 II	1.0−100.0
Mg	279.1 II	5.0−500.0
Mn	257.6 II	0.1−10.0
Ni	231.6 II	0.1−10.0
P	214.9 I	5.0−500.0
V	310.2 II	0.1−10.0
Zn	213.9 II	0.1−10.0

[a] Generator power, 1100 W; peaking element, Mn (257.6 nm); auxiliary Ar flow, 0.5 L/min; coolant flow, 11 L/min; nebulizer pressure, 39 psi; pump speed, 1.4 mL/min; prealigned sample introduction system with Hildebrand grid nebulizer and modified Scott spray chamber; data acquisition, three integrations per reading; uptake time 20 s; scan integration time 1 s. [b] State of ionization, I and II indicate that spectral lines originate from the neutral atom and singly ionized state respectively.

Wet Ashing Sample Preparation. Typically, 0.5−2.0 g of homogenized sample was placed into acid-cleaned, silanized quartz or borosilicate test tubes and 1 mL of concentrated subboiling distilled nitric acid (Seastar, Seattle, WA) was added with 1−2 mL of 18 MΩ deionized distilled water. Test tubes were placed on a Multiblock heater (Lab-Line Instruments, Inc., Melrose Park, IL) and heated at 80 °C overnight. The precision of the weights used for the triplicate digests of each material was better than 5% RSD. The next day the digests were treated with 1 mL of 50% hydrogen peroxide (Fisher, Fair Lawn, NJ), added dropwise, and heated at 100 °C for several hours, repeating the peroxide treatment until sample digests were clear. The maximum amount of peroxide added never exceeded 5 mL. Digests were subsequently heated overnight at 80 °C, and then 1 mL of Ultrex HCl (J. T. Baker, Phillipsburg, NJ) was added and the digests were heated for 3−4 h. Digests were filtered using ashless 7 cm No. 41 filter paper (Whatman, Maidstone, England), and they were diluted to a final volume of 15 mL and stored in acid-cleaned polyethylene test tubes until analyzed. Sample preparation blanks were analyzed with each batch of samples and all data were blank corrected. Triplicate sample preparations of each sample were analyzed to determine the elemental content.

Dry Ashing Sample Preparation. Typically, 0.5−2.0 g of homogenized sample was weighed into acid-cleaned borosilicate or quartz test tubes and placed into a muffle furnace (Lindberg, Wattertown, WI). The temperature was slowly ramped to 480 °C, increasing the temperature in 50 °C intervals, and samples remained at 480 °C overnight. If the sample ash was white, the sample was diluted to a final

Table 3. Detection Limits

element	AAS wavelength (nm)	AAS detection limit (μg/mL)	ICP-AES wavelength (nm)	ICP-AES detection limit (μg/mL)
Ca	422.7	0.012	317.9	0.15
Co			228.6	0.006
Cu	324.8	0.009	324.8	0.006
Cr	357.9	0.011	267.7	0.003
Fe	248.3	0.029	259.9	0.021
Mg	285.2	0.0012	279.1	0.020
Mn	279.5	0.007	257.6	0.005
Ni			231.6	0.009
P			214.9	0.240
V			310.2	0.009
Zn	213.9	0.008	213.9	0.006

volume of 15 mL using 5% (v/v) subboiling distilled nitric acid. If the ash was not white, the sample was treated with 1 mL of subboiling distilled nitric acid, taken to dryness on the heating block and returned to the muffle at 480 °C overnight. If necessary, the 1 mL subboiling distilled nitric acid treatment was repeated. The sample ash was then diluted to a final volume of 15 mL using 5% subboiling distilled nitric acid. All samples were stored in acid-cleaned polyethylene test tubes until analyzed. One additional minor modification sometimes incorporated when samples with high silicon content such as leaves were analyzed was the addition of 0.5−0.75 mL of subboiling distilled hydrofluoric acid (Seastar, Seattle, WA) and 10 mL of 5% (v/v) subboiling distilled nitric acid prior to the final dilution. Once added, the sample was placed on the heating block and heated at 50−80 °C for 30 min prior to dilution to a final volume of 15 mL with 5% (v/v) subboiling distilled nitric acid.

RESULTS AND DISCUSSION

Many of the elements of nutritional interest do not require the use of an extremely sensitive method such as GFAAS. In general, flame AAS and ICP-AES provide sufficient detection capability for the quantitation of elements of interest in foods. One clear benefit of the use of ICP-AES, particularly in the simultaneous mode of operation, is the speed advantage provided by obtaining multielement data. For the PS3000 ICP-AES system, wavelengths for the simultaneous mode of operation were not necessarily selected to provide the best detection capability (e.g., lowest detection limits). Instead, wavelengths were selected taking into consideration the usual range of concentrations expected in sample digests resulting in the selection of less sensitive wavelengths. Please note that Na and K data are not included here. That is because those elements are done by flame emission spectrometry because it is the simplest and most suitable technique. What follows is a discussion of the detection capability, accuracy, and precision achievable with flame AAS and ICP-AES. Wet ashing and dry ashing sample preparation procedures are compared as are AAS and ICP-AES results for a wide variety of food samples.

Instrumental Detection Limits. The detection capability of an analytical technique is an important figure of merit. The detection limits achieved using both AAS and ICP-AES are compared in Table 3. The calculation of the detection limit was based on the recommendations of IUPAC (IUPAC, 1976; Epstein, 1987), which defines the detection limit as the concentration of analyte equal to a background corrected signal that is 3 times the standard deviation of the blank. This definition is in agreement with the Guidelines for Data Acquisition and Data Quality Evaluation in Environmental Chemistry (ACS, 1980). Both a blank solution and a low concentration standard solution were measured 20 times, and the standard deviation of the analytical signal was determined and converted to a solution concentration (μg/mL) based on the slope of the calibration curve. Instrumental detection limits were then calculated, and conservative values are listed in Table 3. Please note that these are not method detection limits, which take into consideration variability introduced during the sample preparation procedure and which may be affected by the homogeneity and mass of sample analyzed.

Detection limits measured on both instruments are in reasonable agreement with manufacturer's specifications and with expected values reported in the literature. When these detection limits are reviewed, note that less sensitive wavelengths were often selected for the simultaneous mode of operation for the ICP-AES system. That is why, for example, Ca and Mg detection limits are so much poorer for ICP-AES than for flame AAS. Also, note that flame AAS detection limits are not reported for Co, Ni, P, and V because we do not routinely determine these elements by flame AAS, not because they cannot be determined by AAS. The detection limits achievable by both techniques generally provide sufficient detection capability to determine elements of nutritional interest easily, at the concentrations present in foods.

Another point of interest is the range of optimum relative concentration precision (Miller-Ihli et al., 1984) on the calibration curves of the various elements. At the midrange of both flame AAS calibration curves and ICP-AES calibration curves, typical relative concentration precisions were in the 0.5−2.0% range with typical values being less than 1% over the whole range. This suggests that measurement precision is good over a wide range of concentrations for all elements and that extensive calculations need not be done to compute any necessary sample dilutions. Only dilutions to concentrations equal to, or below, that of the lowest standard might prove to be problematic. Although both techniques provide good precision over their entire calibration range, the dynamic range for ICP-AES is clearly larger, making the requirement for sample dilution less likely. Due to the high temperature of the plasma and the completeness of atomization, ICP-AES is less prone to chemical matrix interferences due to molecule formation than flame AA is, but there can be problems with

Table 4. Comparison of AAS and ICP-AES Data for Wet Ash and Dry Ash Sample Preparations of Spinach (NIST SRM 1570a)[a]

ashing method	analysis method	concentration determined (μg/g, dry wt)								
		Ca	Co	Cu	Fe	Mg	Mn	Ni	P	Zn
wet ash	AAS	14169 ± 590		12.2 ± 0.3	259 ± 4	8519 ± 36	71.8 ± 1.6			81.3 ± 1.8
	ICP-AES	14494 ± 324	0.31 ± 0.09	11.1 ± 0.3	248 ± 4	8603 ± 126	73.9 ± 1.6	1.58 ± 0.10	5267 ± 130	81.6 ± 2.1
dry ash	AAS	14080 ± 579		11.1 ± 0.3	256 ± 3	8662 ± 274	75.6 ± 1.7			81.7 ± 1.7
	ICP-AES	15026 ± 305	0.38 ± 0.03	10.3 ± 0.8	259 ± 7	8999 ± 173	76.6 ± 1.9	1.49 ± 0.10	5388 ± 107	82.5 ± 0.8
reference value[b]		15270 ± 410	0.39 ± 0.05	12.2 ± 0.6	[256 ± 11]	[8865 ± 185]	75.9 ± 1.9	2.14 ± 0.10	5180 ± 110	82 ± 3

[a] $n = 3$ subsamples; uncertainty represents ±1 standard deviation. [b] Brackets denote reference values obtained from collaborators; all other values are NIST SRM 1570a certified values.

spectral line overlaps (Montaser, 1987). Data for several samples will help evaluate these points.

Wet Ashing vs Dry Ashing. Table 4 contains data for Spinach SRM 1570a which was analyzed as an unknown material. The spinach was prepared using both a wet ash digestion procedure and a dry ash procedure. Triplicate subsamples were prepared using each of the two sample preparation procedures and were analyzed by both flame AAS and ICP-AES. In all instances, quantitation was accomplished using calibration against aqueous standards as specified earlier. This study facilitates the comparison of the two ashing methods and also allows the examination of the accuracy of flame AAS and ICP-AES methods.

There is no apparent, systematic difference between the wet ash and dry ash digests for any of the elements determined. There is also no significant difference in the mean concentration values determined using flame AAS or ICP-AES. When the data are compared to the reference values, it is clear that the Ca values are biased a little low (approximately 5% based on the mean reference value) regardless of the digestion procedure or the measurement technique. No ready explanation is available. Also, Ni ICP-AES data were significantly low as compared to the NIST certified range. Data for Co, Cu, Fe, Mg, Mn, P, and Zn were all in excellent agreement with the reference ranges. Cr was determined using only the dry ash procedure (1.98 ± 0.20 μg/g), but data are not reported because the Cr content of SRM 1570a is not certified.

Comparison of AAS and ICP-AES Data. Table 5 contains data for six food materials including cabbage, a freeze-dried mixed diet reference material representative of the daily intake of the U.S. population, a mixed meat and vegetables baby food, liver, milk powder, and infant formula. Three of these materials are very homogeneous freeze-dried certified reference materials, facilitating an evaluation of the accuracy achievable with both techniques. Flame AAS and ICP-AES data for the cabbage sample are in excellent agreement with the mean concentrations for the two methods, on average agreeing within ±6%. In the case of the Mixed Diet SRM 1548, AAS and ICP-AES data compare favorably with the exception of Mn, where the ICP-AES value is 10% lower than the AAS value. All of the other Mixed Diet SRM 1548 data are in excellent agreement with the certified concentration ranges. Flame AAS and ICP-AES data for the baby food are in good agreement with the exception of Mn again, where the ICP-AES value is 88% of the flame AAS value. In the case of Bovine Liver SRM 1577a, both flame AAS and ICP-AES data are in excellent agreement with the certified concentration range of all of the elements determined. Milk Powder SRM 1549 was analyzed with equivalent accuracy by both flame AAS and ICP-AES. Finally, the infant formula analyzed yielded similar results by flame AAS and ICP-AES with only Fe values being lower by ICP-AES.

The precision of the two techniques may be compared by considering the reported uncertainties for the three subsamples analyzed using each of the different techniques. Although the reported standard deviations clearly depend on the analyte concentration, relative standard deviations were typically in the 2−5% range and were almost always less than 10% for both flame AAS and ICP-AES. When analyte concentrations were very low (e.g., Fe and Mn in Milk Powder SRM 1549), precisions were slightly poorer. Neither flame AAS nor

Table 5. Comparison of AAS and ICP-AES Data for a Variety of Materials

material	analysis method	ashing method	concentration determined[a] (μg/g[b])									
			Ca	Co	Cr	Cu	Fe	Mg	Mn	Ni	P	Zn
cabbage	AAS	DA	19066 ± 438		1.33 ± 0.10	4.82 ± 0.04	140 ± 1	2005 ± 499	32.7 ± 1.5			38.6 ± 0.2
	ICP-AES		19035 ± 641	0.12 ± 0.02	1.12 ± 0.03	4.50 ± 0.33	142 ± 1	2202 ± 101	32.1 ± 0.7	0.77 ± 0.04	5363 ± 72	41.0 ± 1.1
diet SRM1548	AAS	WA	1762 ± 76			2.98 ± 0.11	34.4 ± 1.2	595 ± 20	5.05 ± 0.05			32.1 ± 0.2
	ICP-AES		1837 ± 72	0.082 ± 0.011	0.27 ± 0.07	2.73 ± 0.07	31.8 ± 2.5	565 ± 20	4.53 ± 0.22	0.43 ± 0.01	3273 ± 206	31.0 ± 0.9
certified value			1740 ± 70			2.6 ± 0.3	32.6 ± 3.6	556 ± 27	5.2 ± 0.1	(0.41)	3240 ± 40	30.8 ± 1.1
baby food	AAS	WA	223 ± 5			2.19 ± 0.05	7.88 ± 0.59	112 ± 4	10.2 ± 0.5			5.43 ± 0.28
	ICP-AES		204 ± 13	0.019 ± 0.004	0.049 ± 0.005	2.11 ± 0.24	7.50 ± 0.84	103 ± 7	8.81 ± 0.25		565 ± 26	5.20 ± 0.53
bovine liver	AAS	WA	125 ± 3			156 ± 5	194 ±10	600 ± 28	10.2 ± 0.5			128 ± 4
SRM1577a	ICP-AES		115 ± 5	0.24 ± 0.01		149 ± 18	196 ± 11	602 ± 24	8.8 ± 0.3		11364 ± 690	115 ± 13
certified value			120 ± 7	0.21 ± 0.05		158 ± 7	194 ± 20	600 ± 15	9.9 ± 0.8		11100 ± 400	123 ± 8
milk powder	AAS	WA	13189 ± 577			0.70 ± 0.02	1.80 ± 0.40	1198 ± 14	0.23 ± 0.04			47.4 ± 1.6
SRM 1549	ICP-AES		13502 ± 619			0.68 ± 0.03	1.96 ± 0.16	1134 ± 107	0.24 ± 0.02		10954 ± 500	46.5 ± 3.3
certified value			13000 ± 500	(0.0041)	0.0026 ± 0.0007	0.7 ± 0.1	1.78 ± 0.10	1200 ± 30	0.26 ± 0.06		10600 ± 200	46.1 ± 2.2
infant formula	AAS	WA	3798 ± 78			4.88 ± 0.03	60.5 ± 2.5	518 ± 20	0.27 ± 0.05			59.0 ± 0.6
	ICP-AES		3717 ± 42			4.66 ± 0.16	50.4 ± 3.5	507 ± 22	0.28 ± 0.02		2640 ± 150	51.2 ± 3.1

[a] $n = 3$ subsamples; uncertainty represents ±1 standard deviation. [b] Concentrations are on a dry weight basis except for baby food and infant formula, which are on an as-received basis (moisture: bovine liver 3%; diet 6%).

ICP-AES provided consistently superior precision as compared to the other technique.

Conclusions. The foods analyzed represent a wide range of matrices and reflect a wide range of analyte concentrations. These data clearly suggest that both flame AAS and ICP-AES can be used to obtain accurate quantitation of elements of nutritional interest. Our experience is that each technique has advantages. Flame AAS is simple to perform, and the flame can be stabilized in a short period of time. If only one or two elements are needed, flame AAS is usually faster. ICP-AES offers the benefit of not having to routinely dilute samples, but operational costs are a bit more expensive and ICP-AES instrumental readings take longer than AAS determinations. Simultaneous multielement determinations on the ICP-AES system apparently do not suffer greatly from the selection of compromise conditions, and ICP-AES allows the determination of nonmetals. Both techniques provide relative concentration precisions on the order of 1% over most of the dynamic calibration range. Clearly, ICP-AES covers a wider concentration range. In the case of Ca, the ICP-AES range is 250 times that of the AAS range and the use of 8-hydroxyquinoline is unnecessary in ICP-AES, which is clearly an advantage.

The purpose of this work was to determine which method was most appropriate for food composition analyses performed in our laboratory. The intent was never to conclude that one technique was superior. Rather, the capabilities of each were to be demonstrated and documented. From these data it is clear that both flame AAS and ICP-AES may be used for precise and accurate elemental analyses of foods. The multielement analysis capability provided by ICP-AES combined with its wide dynamic range will, however, make it the likely choice for any large-scale food composition studies done at FCL in the future. The estimated time savings provided by simultaneous multielement ICP-AES when 11 elements are determined is more than 4-fold, even though ICP-AES sample introduction and clean out is slower, since some of the elements such as Cr and Co would likely require graphite furnace atomization for quantitation by AAS, and that technique is significantly more time consuming than flame atomization.

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LITERATURE CITED

ACS. Guidelines for data acquisition and data quality evaluation in environmental chemistry, ACS Committee on Environmental Improvement. *Anal. Chem. 1980, 52*, 2242−2249.

AOAC. *Methods of Analysis for Nutrition Labeling*; AOAC; Arlington, VA, 1993; pp 3−26.

Epstein, M. S. Comparison of detection limits in atomic spectroscopic method of analysis. In *Detection in Analytical Chemistry: Importance, Theory, and Practice*; Currie, L., Ed.; ACS Symposium Series 361; American Chemical Society: Washington, DC, 1987; Chapter 6, pp 109−125.

IUPAC. IUPAC Commission on spectrochemical and other optical procedures for analysis. *Anal. Chem*. **1976**, *48*, 2294−2296.

Miller-Ihli, N. J. Calibration and curve fitting for extended range AAS. *Spectrochim. Acta* **1984**, *39B*, 1603−1614.

Miller-Ihli, N. J. Trace element determinations in biologicals using atomic absorption spectrometry. *J. Res. Natl. Bur. Stand*. **1988**, *93*, 350−354.

Miller-Ihli, N. J. Graphite furnace atomic absorption spectrometry for the analysis of biological materials. *Spectrochim. Acta* **1989**, *44B*, 1221−1227.

NFPA. *NFPA Guide to Nutrition Labeling*; National Food Processors Association: Washington, DC; 1994; pp 1−194.

RDA. *Recommended Dietary Allowances, 10th ed.;* National Research Council (U.S.) Subcommittee; National Academy Press: Washington, DC; 1989.

Taylor, A.; Branch, S.; Crews, H. M.; Halls, D. J.; White, M. Atomic spectrometry update—clinical and biological materials, food and beverages. *J. Anal. At. Spectrom*. **1994**, *9*, 87R−134R.

Taylor, A.; Branch, S.; Crews, H. M.; Halls, D. J.; White, M. Atomic spectrometry update—clinical and biological materials, food and beverages. *J. Anal. At. Spectrom*. **1995**, *10*, 61R−112R.

Thompson, M. Analytical performance of inductively coupled plasma-atomic emission spectrometry. In *Inductively Coupled Plasmas in Analytical Atomic Spectrometry;* Montaser, A., Golightly, D., Eds.; VCH: New York, 1987; Chapter 5, pp 163−199.

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