Microwave Digestion with $HNO_3-H_2O_2-HF$ for the Determination of Total Aluminum in Seafood and Meat by Inductively Coupled Plasma Atomic Emission Spectrometry

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A sample preparation procedure employing microwave digestion procedure is described for the determination of total aluminum in seafood and meat samples by inductively coupled plasma atomic emission spectrometry (ICP-AES). Lyophilized samples were first digested in closed vessels with HNO_3 and HF. An additional digestion then proceeded in open vessels with H_2O_2. H_3BO_3 was employed to eliminate excess HF. Matrix effects of Ca, K, Mg, and Na, acid effects of HNO_3, and possible spectral interferences were investigated. The influences of amount of HF on analytical results were observed. The recoveries of spike (95.2−97.6%) and the analyses of NIST standard reference materials 1566a (oyster tissues) and 1577b (bovine liver) demonstrated the reliability of the method. Twelve representative seafood and meat samples were analyzed, and the analytical results were compared with those obtained with HNO_3-HClO_4 digestion on a hot plate and with $HNO_3-H_2O_2$ microwave digestion. It was shown that the digestion without the addition of HF was incomplete for the determination of total Al.

Keywords: *Aluminum; microwave digestion; inductively coupled plasma; atomic emission spectrometry; seafood; meat*

INTRODUCTION

Aluminum is one of the most abundant elements in the Earth's crust and is not an essential dietary element. Contrarily, more and more evidence has shown that this element is toxic to humans. For example, Al may accumulate in human body, where it can damage various tissues and cells in the central nervous system (Alfrey, 1986). Al is also believed to be a possible pathogenic factor in Alzheimer's disease (Jacobs et al., 1989; Romero, 1991). Consequently, the public concern about the toxicity and the acceptable daily intake (ADI) of Al is growing. The ADI established by World Health Organization (WHO) is 60 mg/60 kg of body weight (WHO, 1989). Therefore, accurate determination of Al in food, including seafood and meat, is necessary.

Recently, there have been several reports on the determination of Al in various food samples (Wang et al., 1991; Motkosky and Kratochvil, 1993; Schelenz and Zeiller, 1993; Krushevska and Barnes, 1994; Yang et al., 1994; Arruda et al., 1995; Negretti de Brätter et al., 1995; Tahán et al., 1995) and other biological materials (Jacobs et al., 1989; Novarro et al., 1992; Hu, 1994; Tahán et al., 1994; Roberts et al., 1996). The analytical techniques used include graphite furnace atomic absorption spectrometry (GFAAS; Motkosky and Kratochvil, 1993; Arruda et al., 1994; Tahán et al., 1994), energy dispersive X-ray microprobe (EDXRM; Jacobs et al., 1989), inductively coupled plasma atomic emission spectrometry (ICP-AES; Schelenz and Zeiller 1993; Krushevska and Barnes, 1994; Negretti de Brätter et al., 1995), inductively coupled plasma mass spectrometry (ICP-MS; Roberts et al., 1996), and electrothermal vaporization (ETV)-ICP-AES (Hu et al., 1994). Among these methods, the most widely used techniques for determination of Al in biological materials are GFAAS and ICP-AES. EDXRM is not a common technique, and ETV-ICP-AES is still under study. The main limit for the application of ICP-MS is the instrument cost. GFAAS is a very sensitive technique for the analysis of Al and most other metallic elements. Unfortunately, it cannot be used for multielemental determination. ICP-AES has been widely employed for multielemental analysis, including the determination of Al in various food samples, because of its powerful detection ability, good precision, and wide dynamic range of calibration. These factors make ICP-AES a preferred technique for this work.

For the analysis of total Al in food, the transformation of the solid sample into a solution is very important and a necessary step for the ICP-AES measurement. This transformation is generally accomplished by wet digestion with HNO_3 and/or mixed with H_2O_2 or other acids ($HClO_4$, HCl, or H_2SO_4). In the recent few years, microwave-based digestion has been extensively applied to sample preparation for many different materials. Wide acceptance of microwave digestion is based on the significant advantages of the technique: the contamination is significantly minimized due to the complete separation of the samples from the environment during digestion and the use of a smaller amount of acid; the losses of volatile elements are substantially decreased since the system is closed; and the time needed for a complete decomposition is notably reduced, due to the elevated pressure and temperature within the closed vessels which accelerate sample digestion (Neas and Collins, 1988). Applications of microwave digestion in the determination of Al in food have been conducted by some researchers (Schelenz and Zeiller, 1993; Yang et al., 1994; Arruda et al., 1995; Negretti de Brätter et al., 1995; Tahán et al., 1995). The digesting reagents used by the researchers are generally HNO_3, $HNO_3-H_2O_2$, and $HNO_3-H_2SO_4$. However, a low recovery of Al in seafood and meat has been observed with the above reagents. In fact, there has been difficulty in the determination of Al in seafood and meat samples.

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Table 1. Instrumentation and Operating Conditions

ICP spectrometer	
frequency, MHz	27.12
forward power, W	650
reflected frequency, W	<4
coolant gas, L/min	10.5
intermediate gas, L/min	0.8
carrier gas, L/min	0.8
observation height, mm	9.0
nebulizer uptake rate of solution, mL/min	3
analytical line, nm	396.152
integration time	1 s
microwave digestion program	
maximum power (100%), W	630 ± 70

step	time, min	power, %	vent manually
I	5	40	no
II	5	0	yes
III	5	65	no
IV	5	0	yes
V	10	25	yes

In this report, a procedure for the determination of total Al in seafood and meat based on microwave digestion has been developed that allowed the subsequent quantification by ICP-AES. Samples were digested with $HNO_3-H_2O_2-HF$ and the excess HF was eliminated by H_3BO_3. Good recoveries of Al have been found for the determination of Al in NIST (National Institute of Standards and Technology) standard reference materials and in the selected matrices (reagent blank, canned crab meat, and shrimp).

EXPERIMENTAL PROCEDURES

Precaution. *Digestions employ the use of both perchloric and hydrofluoric acids. Perchloric acid is very caustic and may deflagrate in contact with oxidizable substances. Hydrofluoric acid is very poisonous and can produce skin burns and cause severe irritation of eyes and eyelids. Eye and skin protection and the use of a fume hood are required during all operations in which these acids are employed.*

Apparatus. Nalgene polypropylene volumetric flasks (Nalge Co., Rochester, NY) were used for sample preparation with HF. Glass flasks were used for other sample preparation procedures. A Pipetman pipet (Gilson, France) was employed for the transfer of HF. Glass pipet were used to transfer all other reagents. The microwave system used for the sample digestion was MDS-81D microwave oven (CEM Co., Matthews, NC). All measurements were performed with an ARL 3410+ sequential ICP spectrometer with Minitorch (Fisons, Dearborn, MI). The sample solutions were introduced by a Meinhard concentric nebulizer (Type K). The main operating conditions are listed in Table 1.

Reagents. Deionized water (specific resistance: 18 MΩ), which was obtained from a Nanopure system (Barnstead, Dubuque, IA), was used throughout. TraceMetal grade HNO_3 (70%), HF (50%), and $HClO_4$ (70%) and certified ACS grade H_2O_2 (30%) (Fisher Scientific, Fair Lawn, NJ) were used for the preparation of solutions and samples. Reagent ACS grade HNO_3 was used for labware cleaning. The H_3BO_3 solution (4%, w/v) was prepared by dissolving 20 g of H_3BO_3 (99.99%, Aldrich Chemical Company, Milwaukee, WI) in 500 mL of water. The stock solution of aluminum (1000 μg/mL) was purchased from VHG Labs (Manchester, NH). The solutions for spiking and appropriate working standards were prepared from the above stock solution by serial dilution.

Contamination Control. The flasks for sample preparation were filled with 20% (v/v) HNO_3 and kept overnight. Just before use, they were rinsed with distilled water at least four times and then with Nanopure deionized water three times. All pipets were soaked in 2% Micro solution (Cole-Parmer Instrument Co., Niles, IL) overnight and then rinsed with deionized water and Nanopure deionize water. The microwave digestion vessels (12/group) were cleaned by the following procedures: (1) All surfaces including the safety pressure discs and the vessel caps were wiped with a lint-free paper towel moistened with ethanol. (2) All surfaces were scrubbed with dilute detergent and a small nylon bristle brush and then rinsed with distilled water. (3) To each vessel was added 15 mL of HNO_3 (70%), and the vessel caps were hand-tightened. The vessels were than heated in the microwave oven at 100% power for 5 min. (4) After cooling to room temperature, the vessels were vented manually and the acid was discarded. (5) The vessels were rinsed at least three times with deionized water and Nanopure deionized water, individually. The cleaned vessels were checked by analyzing digestion blanks. If the content of Al was found to be higher than the detection limit, the cleaning procedure was repeated.

Samples. Two NIST standard reference materials (SRM 1566a oyster tissues and SRM 1577b bovine liver) were prepared to test the accuracy of the methods. Twelve different types of seafood and meat samples were purchased at random from the local markets. Canned samples were drained of fluid before blending. SRM 1566a and SRM 1577b were dried following NIST instructions. All other samples were lyophilized at a reduced pressure of approximately 3 Pa and a condensing coil temperature of −50 °C with a freeze-dry system (Labcono, Kansas City, MO) and ground in a Retsch mill (Brinkmann Instruments, Westbury, NY) equipped with a 0.5 mm stainless steel sieve. After mixing, the samples were kept in Whirl-Pak bags (Nasco, Fort Atkinson, WI) at 4 °C until analysis.

Sample Digestion. The sample (0.5−1 g, as recommended by the manufacturer) was weighed into a 120 mL polytetrafluoroethylene (PTFE) vessel, and then 10 mL of HNO_3 (70%) and 0.5 mL of HF were added. For the spiking test, an appropriate mass of Al was also added. The safety valve was placed on the vessel, and then the cap was tightened using a capping station. Up to 12 vessels were placed in the turntable, and the venting tube was attached. The samples were digested in the microwave oven following the program listed in Table 1. After cooling to room temperature, the vessels were vented manually and opened with the capping station. The cap, relief valve, and the inside walls of each vessel were washed down with a small quantity of deionized water. 5 mL of hydrogen peroxide and 10 mL of 4% (w/v) H_3BO_3 solution were then added to each vessel, and the contents in the vessel were swirled well. The uncapped vessels were placed in the microwave oven, and the samples were digested at 100% power for approximately 30 min until each sample was evaporated to about 2 mL. The role of H_2O_2 is to further degrade any remaining organic materials, and that of H_3BO_3 is to eliminate the remaining HF. In addition, this step also decreases the amount of nitric acid to give a concentration of HNO_3 about 2% in each final solution to avoid any possible acid effect. The contents were allowed to cool and transferred into a 100 mL Nalgene volumetric flask. Corresponding blanks were prepared with the same procedure.

For comparison purposes, the samples were also analyzed with two other digestion procedures: (i) microwave digestion with $HNO_3-H_2O_2$; or (ii) digestion with HNO_3-HClO_4 on a hot plate. The $HNO_3-H_2O_2$ procedure was similar as above with the following exception: HF and H_3BO_3 were omitted. For the HNO_3-HClO_4 procedure, 0.5−1 g of sample was weighed into a 100 mL Kohlrausch flask. A 10 mL aliquot of HNO_3 (70%) was added, and the sample was cautiously heated on a hot plate until any vigorous reaction subsided. After cooling, 8 mL of $HClO_4$ (70%) was added and the sample solution was heated on the hot plate at a gentle boil until the solution was colorless or nearly so and the white fumes of $HClO_4$ were evolved. (**Caution:** *Do not allow contents to go dry*.) After the solution was cooled, 30 mL of water was added and the solution was boiled for an additional 15 min. The cooled solution was brought to final volume with water.

Determination. The samples were run without further dilution. Three calibration curves were made with three different media: 2% (v/v) HNO_3−0.4% (w/v) H_3BO_3, 2% HNO_3 only, and 2% HNO_3−5% (v/v) $HClO_4$, each corresponding to the three different digestion procedures. The concentration of Al

Table 2. Recoveries of Al in Reagent Blank, Canned Crab Meat, and Shrimp

matrix	Al concentration (μg/g)[a]	Al added (μg)	Al found (μg)[a]	average recovery ± SD (%)
reagent blank	0	1.25	1.22 ± 0.01	97.6 ± 0.8
canned crab meat	46.9 ± 0.4	25.0	23.8 ± 0.2	95.2 ± 0.8
shrimp	151 ± 1	75.0	72.1 ± 0.8	96.1 ± 1.1

[a] Results are expressed as the mean ± one standard deviation (SD) for triplicate determinations.

Table 3. Analytical Results of Al in Selected Samples with Three Different Sample Digestion Procedures (in μg/g)

sample	HNO_3-HClO_4	$HNO_3-H_2O_2$	$HNO_3-H_2O_2-HF$
SRM 1566a (oyster tissues)	109.1 ± 0.3	64.0 ± 2.7	194.1 ± 2.7[a,b]
SRM 1577b (bovine liver)	<1.4[c]	<2.1[c]	2.61 ± 0.34[d,b]
beef (biceps femoris muscle)	1.66 ± 0.26	<2.1[c]	3.71 ± 0.20[b]
chicken breast	4.77 ± 1.00	4.16 ± 0.39	5.12 ± 0.20
canned clams	864 ± 6	586 ± 8	1511 ± 14[b]
codfish	3.42 ± 0.22	5.86 ± 0.26	6.99 ± 0.66[b]
canned crab	35.5 ± 0.8	39.8 ± 0.8	46.9 ± 0.4[b]
flounder	2.20 ± 0.16	<2.1[c]	3.53 ± 0.78[b]
lobster	4.86 ± 1.00	4.17 ± 0.30	7.80 ± 0.64[b]
oyster	279 ± 9	182 ± 6	606 ± 9[b]
lean pork	<1.4[c]	<2.1[c]	<2.0[c,b]
sea scallops	12.2 ± 1.2	9.56 ± 0.26	23.0 ± 1.9[b]
shrimp	79.9 ± 5.5	64.1 ± 3.9	151 ± 1.0[b]
squid	1.68 ± 0.15	2.26 ± 0.46	3.95 ± 0.37[b]

[a] NIST certified value: 202.5 ± 12.5 μg/g. [b] Statistically significant ($p < 0.005$, Student's t test) when compared to parallel HNO_3-HClO_4 and $HNO_3-H_2O_2$ results. [c] Limit of determination in dried material (1 g of sample was digested and diluted to 100 mL). [d] NIST value for information: 3 μg/g.

was calculated from a linear regression equation on the basis of an average intensity of four separate determinations.

RESULTS AND DISCUSSION

Influences of HNO_3 on Al Emission Signal. Since the sample solutions contain different amounts of HNO_3 after digestion, the effect of HNO_3 on emission intensity of Al was examined. The results show that the emission signal of Al decreases with the increasing concentration of nitric acid when the concentration is less than 0.1 M. For higher nitric acid concentrations (>0.1 M), the variation of Al emission with the increase of nitric acid is slight: the maximum relative variation is less than 1.7% between 0.1 and 1.5 M. This indicates that the effects of HNO_3 are negligible in this work, because the concentration of nitric acid in all standards and sample solutions were kept between 0.1 and 0.8 M.

Matrix Effects of Ca, K, Mg, and Na on Al Emission. The effects of matrix elements (especially the easily ionized elements, such as K, Na, etc.) have been considered as one of the main error sources in ICP-AES. An enhancement as high as 200% or a depression as much as 50% may be caused if the concentration of a matrix element is high and/or the operating conditions, such as the forward power, the carrier gas flow rate, or the observation height, are not optimized (Sun et al., 1988). Since some seafood and canned meat products contain relatively high levels of Na, K, Ca, or Mg, the effects of the four elements on Al emission were evaluated. It is established that the emission of Al was not affected by up to 200 μg/mL of Ca or Mg and 400 μg/mL of K or Na. A 400 μg/mL of Ca or Mg caused a depression of 4% on emission signal of Al. Higher concentrations of Ca, Mg, K and Na brought about more serious suppression on the emission of Al. Generally, the concentrations of Ca and Mg are lower than 200 μg/mL and those of Na and K are below 400 μg/mL in the actual sample solutions (Nettleto, 1985; Franklin and Davis, 1981) with the dilution factor of 100−200 (0.5−1 g of the solid sample was digested and diluted to 100 mL), the matrix effects, therefore, would be negligible under the optimized ICP operating conditions in this work.

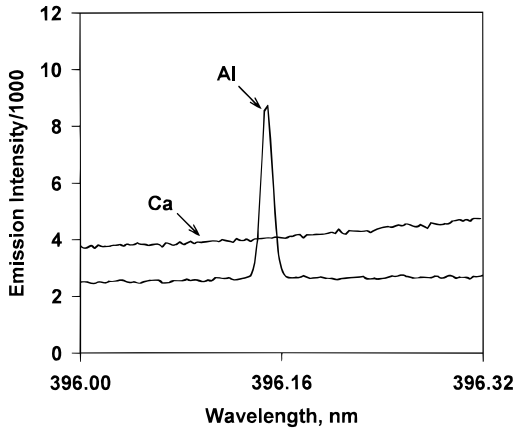

Figure 1. Spectral interference of Ca (100 μg/mL), scanned between 396.00 and 396.32 nm, superimposed on the scan of Al (0.5 μg/mL) which emits at 396.152 nm.

Spectral Interference. For seafood and meat samples, the most likely spectral interference on Al I 396.152 nm line is from the left wing of Ca II 396.847 nm line (Winge et al., 1985). To observe the magnitude of this interference, a pure Ca solution (100 μg/mL) was scanned between 396.00 and 396.32 nm and the scanned profiles were compared with the emission line profile of 0.5 μg/mL of Al (Figure 1). The results show that the spectral interference of Ca on Al I 396.152 nm is significant: 100 μg/mL of Ca can result in an apparent Al concentration of 0.11 μg/mL. Figure 1 also shows that the interference from Ca cannot be accurately corrected with the one-point off-peak method because the sloping profile of Ca. In this work, background signals and the spectral interference of Ca were corrected with an off-peak two-point correction method. The two points for the correction were set to left and right sides of the peak with a wavelength distance of 0.022 nm.

Influences of Amount of HF. The NIST SRM 1566a (oyster tissues) was used to test the influences of amount of HF. The sample weight for the tests was 0.5 g, and the other conditions and amounts of H_2O_2,

Table 4. Analytical Results of 14 Elements in Two NIST Standard Reference Materials with $HNO_3-H_2O_2-HF$ Digestion Procedure

	SRM 1566a (oyster tissues)		SRM 1577b (bovine liver)	
element	certified value	this work[a]	certified value	this work[a]
Ca	1960 ± 190	1830 ± 20	116 ± 4	116.6 ± 2.7
Cd	4.15 ± 0.38	4.16 ± 0.19	0.50 ± 0.03	N/A
Cr	1.43 ± 0.46	1.39 ± 0.10	N/A	N/A
Cu	66.3 ± 4.3	64.2 ± 0.3	160 ± 8	159.6 ± 1.9
Fe	539 ± 15	525.4 ± 14.6	184 ± 15	181.6 ± 2.3
K	0.790 ± 0.047	0.796 ± 0.002	0.994 ± 0.002	0.983 ± 0.003
Mg	1180 ± 170	1160 ± 10	601 ± 28	630.4 ± 14.2
Mn	12.3 ± 1.5	11.4 ± 0.5	10.5 ± 1.7	10.2 ± 0.2
Mo	N/A	N/A	3.5 ± 0.3	3.64 ± 0.18
Na	0.417 ± 0.013	0.423 ± 0.005	0.242 ± 0.006	0.239 ± 0.003
Ni	2.25 ± 0.44	2.55 ± 0.10	N/A	N/A
P	0.623 ± 0.018	0.616 ± 0.003	1.10 ± 0.03	1.18 ± 0.02
V	4.68 ± 0.15	4.94 ± 0.14	N/A	N/A
Zn	830 ± 57	832.4 ± 46.1	127 ± 16	128.2 ± 3.5

[a] Results are expressed as the mean ± one standard deviation (SD) for triplicate determinations. The units are wt % for K, Na, and P and μg/g for all other elements; N/A = not available.

HNO_3, and H_3BO_3 were kept unchanged. The results show that the determined concentration of Al was far less than the certified value (202.5 ± 12.5 μg/g) without the addition of HF. However, all of the determined concentrations of Al fell in the range of the certified value with addition of 0.05, 0.2, and 0.5 mL of HF.

Limit of Detection, Limit of Determination, and Precision. The limits of detection (2σ) and determination (10σ), which were calculated based on the standard deviations of 10 measurement signals of the blank solution [2% HNO_3 (v/v) − 0.4% (w/v) H_3BO_3], were 4.0 and 20 μg/L, respectively. The limit of determination is corresponding to 2.0 μg/g of Al in the dried material if 1 g of the sample is digested and diluted to 100 mL. The relative standard deviations (RSDs) were 0.5% and 1.8% for 10 determinations of two solutions, which contain 2% (v/v) HNO_3 and 0.4% (w/v) H_3BO_3, with the Al concentrations of 5.0 and 0.1 μg/mL, individually.

Recoveries of Spiking Al in Selected Matrices. In order to further verify the accuracy of the developed procedure, recoveries of spiking Al from three selected matrices (reagent blank, canned crab meat, and shrimp) were carried out. To improve the precision of the recovery data, all the matrices with or without spike were prepared in triplicate. Al was spiked from the pure Al solutions with a NIST calibrated pipetter (1000 μL, accuracy ±0.3%, precision ±0.2%). The analytical results are listed in Table 2. It is shown that good recoveries were obtained for different quantity of spiking Al and from different matrices.

Sample Analysis. Twelve representative seafoods, meats, and two NIST standard reference materials (SRM 1566a oyster tissues and SRM 1577b bovine liver) were analyzed, and the analytical results were compared with those obtained with the $HNO_3-H_2O_2$ microwave digestion without the addition of HF and the digestion with HNO_3-HClO_4. The analytical results are given in Table 3. The concentration values obtained with both HNO_3-HClO_4 and $HNO_3-H_2O_2$ methods were lower than those obtained with $HNO_3-H_2O_2-HF$ method for most of the samples, and only the results obtained with the latter method agreed with the NIST certified values. The reason for this observation may be related to the possibility that some of aluminum is combined with silicon, which cannot be dissolved by HNO_3 or $HClO_4$. The analytical results show that the crustaceous seafoods contain fairly high concentration of Al. By contrast, Al in meats and fish is relatively low. This probably reflects the fact that shellfish reside in, and filter, sediment which can be high in Al.

In addition, 14 other elements (Ca, Cd, Cr, Cu, Fe, K, Mg, Mn, Mo, Na, Ni, P, V, and Zn) in the two NIST standard reference materials were also analyzed with the developed procedure. The results are shown in Table 4. It can be seen that all the analytical results coincide with the NIST certified values, indicating that the advantages of multielement analysis of ICP-AES are not influenced.

Conclusion. The developed procedure provides an accurate and reliable way for the determination of total Al and 14 other elements in seafood and meat samples via ICP-AES. Digestion for the determination of total Al without the addition of HF gives erroneous results for some kinds of seafood and meat, especially for shellfish. The disadvantage of the procedure is the use of H_3BO_3, which could result in memory effect for future determination of boron.

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