Closed-Vessel Microwave Acid Digestion of Foodstuffs and Trace Aluminum Determination by Graphite Furnace Atomic Absorption Spectrometry

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The estimation of the daily dietary intake of aluminum is part of an ongoing project aimed at the assessment of the daily intake of food constituents by the Belgian population. This requires a reliable analytical method that is applicable to different solid foodstuffs and can easily be implemented in an analytical laboratory. A method is proposed for the dissolution of food samples with microwave digestion (HNO_3–H_2O_2 2:0.5) and for the subsequent Al determination by graphite furnace atomic absorption spectrometry. The use of closed-vessel microwave acid digestion shortens the time for sample dissolution and improves the analysis quality. A direct calibration against aqueous solutions was used for all of the foodstuff digests. For the method validation, standard reference materials were analyzed. Aluminum was determined in 13 different types of bread and 30 types of fish. The limit of detection in solution is about 3 μg/L (3σ). Some samples with relatively higher aluminum content were identified.

INTRODUCTION

Aluminum (Al) is not an essential dietary element and, rather, has been considered a neurotoxin. It has been implicated in renal dialysis encephalopathy (Alfrey et al., 1976), microcytic anemia (O'Hare and Murnaghan, 1982), and ostemomalacia (Ott et al., 1982; Boyce et al., 1982). There is a growing public concern about the causal relationship between Al and Alzheimer's disease (Bjorksten, 1982). The acceptable daily intake (ADI) of Al established by WHO–FAO is 60 mg/60 kg of body weight (WHO–FAO, 1989). Therefore, monitoring Al in foods is necessary.

Graphite furnace atomic absorption spectrometry (GFAAS) is the method of choice for trace Al determination (Smeyers-Verbeke and Verbeelen, 1985, 1988). However, the analysis might be hampered by the contamination of samples from the environment when Al is measured at micrograms per liter levels (Skelly and Distefano, 1988; Smeyers-Verbeke et al., 1980; Greger, 1985). Studies show that good results can be obtained if relatively simple sample preparation and calibration methods are used (Taylor, 1988).

Solid food analysis usually requires a rather elaborate pretreatment to make AAS determination possible. For biological materials different sample preparation methods have been proposed, such as an extraction of the Al with ethylenediaminetetraacetic acid (EDTA) (LeGendre and Alfrey, 1976; Smeyers-Verbeke and Verbeelen, 1985), low-temperature ashing (Smeyers-Verbeke and Verbeelen, 1985), and solubilization of the sample in hot aqueous tetramethylammonium hydroxide (Stevens, 1984). The classical wet acid digestion techniques are known to have some disadvantages: they are tedious, and samples are easily contaminated. The use of a large quantity of acid(s), which contributes to an overall contamination load, and possible contamination from the environment may affect the quality of trace Al analysis (Skelly and Distefano, 1988).

The application of high-pressure closed-vessel acid digestion, assisted by microwave heating, has offered a promising alternative (Kingston and Jassie, 1988). The main merits of the microwave digestion technique are as follows: contamination is substantially minimized due to the use of a smaller quantity of acid and the complete separation of samples from the environment; a decomposition of samples is accomplished within a reduced time, due to the elevated pressure and temperature within the digestion vessels which accelerate sample digestion; within the closed system, losses of volatile elements are greatly decreased. This technique has been applied to the dissolution of biological materials prior to Al determination by GFAAS (Skelly and Distefano, 1988; Nicholson et al., 1989). Some researchers have also addressed its application in foodstuff preparation (Schelenz and Zeiller, 1993; Friel et al., 1990; Topper and Kotuby-Amacher, 1990; Xu et al., 1992). However, a low Al recovery has been observed. For the determination of Al in reference materials, frequently a great discrepancy was reported (Blotcky et al., 1992). In fact, there has been a difficulty in measuring trace Al in biological and food samples.

In this paper we present our work of food sample dissolution using microwave digestion and Al determination with GFAAS. The method was validated by establishing adequate requirements for performance criteria, such as accuracy, precision, and limit of detection. Finally, the analysis of different types of bread and fish was carried out as part of a project aimed at estimating the daily intake of food constituents by the Belgian population.

EXPERIMENTAL PROCEDURES

Apparatus. All measurements were performed with a Perkin-Elmer Zeeman 3030 atomic absorption spectrometer equipped with an HGA-600 graphite furnace, an AS-60 au-

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Table 1. Instrumental Conditions and Furnace Program

	Instrumental Conditions		
light source	hollow cathode lamp	spectral bandwidth (nm)	0.7
lamp current (mA)	25	mode	integrated absorbance
wavelength (nm)	309.3	injection volume (μL)	20

		Furnace Program		
step	temp (°C)	ramp time (s)	hold time (s)	Ar flow rate (mL/min)
1	110	5	45	300
2	600	30	10	300
3	1400	40	45	300
4	2500	0	5	0
5	2700	1	4	300

Table 2. Microwave Heating Program

step	1	2	3	4	5	6	7	8
power (W)	180	360	600	0	600	360	180	0
time (min)	3	3	3	3	3	3	3	2

tosampler, and a PR-100 printer (Perkin-Elmer). A pyrolytic graphite coated tube with pyrolytic graphite platform was used. The instrumental conditions and the furnace program are given in Table 1. Signal evaluation was by means of the integrated absorbance values (As) computed by the instrument.

The digestions were performed on a programmable Milestone 1200 microwave digestion system with a maximum power supply of 1200 W and equipped with the ACM-100 automatic capping module (Milestone, Germany). Teflon HPV 80 high-pressure vessels (80 mL) with safety shield that can withstand up to 120 bar of pressure and a temperature of 300 °C are used. The microwave heating program is given in Table 2.

Reagents. Standard Al solutions in 0.2% (v/v) nitric acid containing 0, 10, 20, 40, and 60 μg/L Al were prepared from a Titrisol concentrate containing 1000 mg/L Al (Merck, Darmstadt, Germany). Nitric acid (65% w/w) of highest purity was used for the preparation of the standards and for the digestion (Suprapur; Merck), while for labware cleaning, analytical reagent grade nitric acid (65% w/w) was used (pro analysi; Merck). Hydrogen peroxide (30% w/w) and hydrofluoric acid (48% w/w) were of analytical reagent grade (pro analysi; Merck). The water used for the preparation of all solutions was obtained from a Milli-Q water purification system (Millipore, Bedford, MA) and contained no detectable Al.

Materials and Their Cleaning. To avoid contamination from the containers, quartz volumetric flasks and polypropylene recipients are used. They are filled with 50% (v/v) nitric acid and kept for at least one night. Just before use, they are rinsed well with Milli-Q water. Eppendorf micropipets with removable tips are used for all solution preparation, but cleaning of the tips was not found to be necessary. Since with acidic solutions sometimes contamination of autosampler cups has been observed, the cups were soaked in 50% (v/v) nitric acid in a capped polypropylene container overnight and afterward rinsed with Milli-Q water. After this cleaning procedure, they are stored capped, in an acid-washed polypropylene container, to avoid contamination from the environment.

The digestion vessels are cleaned by submitting 8 mL of 50% (v/v) nitric acid (Suprapur) to the same microwave program as the samples. After this procedure, contamination from the vessels is controlled by analyzing digestion blanks. The cleaning procedure has to be repeated if the Al content of the digestion blank exceeds the detection limit, which in solution was generally about 3 μg/L (see further). In practice, however, this could generally be achieved with a single cleaning of the vessels, after which several sample or blank digestions could be performed with a simple water rinsing of the vessels in between. At the end of the day the vessels were filled with 50% (v/v) nitric acid and were placed capped in the hood. Just before use, they are rinsed well with water. When an increase in the Al concentration in the digestion blanks was observed, the complete cleaning procedure was repeated.

A plastic knife is used for reducing the samples. A plastic spoon and small disposable microbeakers are used to weigh the samples.

Samples. Thirteen different types of bread and bread products which are representative of the consumption in Belgium were purchased at two different locations. At each location three samples of the same type of bread and nine samples of the bread products were bought and pooled. Therefore, 26 test samples were obtained. All of the samples are freeze-dried to preserve them against microbiological degradation and were subsequently thoroughly homogenized with a pestle in a plastic mortar.

Thirty different types of fish, either fresh, canned, or smoked, were purchased at at least two locations or from different commercial brands. The fresh samples were prepared (baked or cooked) in the same way as they are consumed, and only the fish tissue was taken for the analysis. For the canned and smoked fish only the edible parts were used. As soon as possible after their arrival at the laboratory, the fish samples were freeze-dried and then subsequently thoroughly homogenized with a pestle in a plastic mortar.

Standard Reference Materials. Accuracy of the measurement was determined by the analysis of wheat flour with a certified Al value of 5.7 ± 1.3 μg/g (NIST 1567a), rice flour with a certified value of 4.4 ± 1.0 μg/g (NIST 1568a), total diet with an information value of 33 μg/g (NIST 1548), and fish tissue with an information value of 20.9 μg/g (IAEA RM MA-B-3/TM).

Microwave Sample Digestion. From each of the bread test samples two portions of ±0.1 g were digested. From each of the fish samples one or two portions of ±0.2 g were taken for the digestion. For the reference materials, six portions of either ±0.2 g (wheat flour) or ±0.1 g (other reference materials) were used in the analysis. After their transfer into the digestion vessels, to each portion were added 2 mL of concentrated nitric acid, 0.5 mL of hydrogen peroxide, and 1 mL of Milli-Q water. The microwave heating program is given in Table 2. It is known that the microwave irradiation efficiency differs with the position in the microwave oven. Preliminary experiments revealed that, without the use of a turntable, efficient sample decomposition could be obtained when three digestion vessels, inserted into safety shields, were positioned around the central point of the oven. Therefore, simultaneous digestion of three samples could be performed. After cooling, the pressure within the digestion vessel was vented into the hood. The rupture disk, used as an inner cap, was removed very carefully, and any condensate was recombined with the resulting digest into a quartz volumetric flask. The sample digests were diluted with Milli-Q water to 10–25 mL. The resulting solutions therefore contained 20% or 8% (v/v) nitric acid, respectively. The reference total diet and fish tissue digests were diluted to 100 mL. The resulting solutions therefore contained 2% (v/v) nitric acid. For reference rice flour and wheat flour, the digests were diluted to 25 mL with Milli-Q water, the resulting solutions containing 8% (v/v) nitric acid.

Corresponding digestion blanks were also prepared according to the above procedure.

RESULTS AND DISCUSSION

Control of the Contamination. The digestion vessels are one of the sources of Al contamination, since the high temperature and the high concentration of acids promote Al being leached from the surface of the vessels. Frequent monitoring of digestion blanks is therefore necessary for Al determinations at the microgram per liter levels. This is particularly important with new vessels or after use of the vessels for the digestion of samples containing high concentrations of Al. In our experiments uncontrolled digestion blanks were found to change from 0 to 35 μg/L ($n = 11$), which makes it impossible to accurately determine trace levels of Al. However, by means of the cleaning procedure proposed here, the digestion blanks ($n = 25$) could be reduced to below the detection limit, which in solution was calculated to be about 3 μg/L.

Effect of Nitric Acid on the GFAAS Determination of Al. Since, after the digestion, measurement solutions containing up to 20% (v/v) nitric acid are obtained, the effect of nitric acid on the Al determination was evaluated.

The optimal ashing temperature, which is about 1400 °C, is not affected by up to at least 20% (v/v) nitric acid. This temperature was also found to be useful for all digests with nitric acid concentration up to at least 20% (v/v).

It was also established that the Al absorbance was not affected by the nitric acid concentrations used in this study: the absorbances (As) for a 40 μg/L Al standard in 0.2%, 5%, 15%, and 20% (v/v) nitric acid are, respectively, 0.144, 0.140, 0.148, and 0.144. Also, the peak shape does not change obviously with increasing nitric acid concentrations. Finally, the high amounts of nitric acid did not, as we feared, decrease the lifetime of the graphite tube, since more than 260 injections could be performed without changing the performance characteristics.

Validation of the Proposed Method. *Detection of Matrix Interferences.* To check possible matrix interferences, the slopes of the aqueous calibration line and a standard addition line were compared. The method of additions has been performed on currant bread, sugar loaf, croissant, and whole-meal bread digests as well as on fresh cod, canned crab, and smoked trout digests. The slope ratios of the standard addition line over the standard calibration line were, respectively, 1.01, 1.03, 1.05, 1.08, 1.09, 0.92, and 0.97. For none of these digests was a statistically significant difference (*t*-test; $\alpha = 0.05$) in slope between the calibration line and the standard addition line observed. This points to the absence of matrix effects in the Al measurement. Hence, the determination of Al in all bread and fish samples was performed by a direct calibration against aqueous standard solutions.

Accuracy. The accuracy was evaluated by recovery experiments as well as by the analysis of reference materials.

Recovery experiments were first carried out on two types of bread (white bread and seven-cereal bread) and two types of fish (canned sardines in oil and baked sole). For each sample, four aliquots are spiked, digested and prepared as described previously. The addition of Al is performed by pipetting different amounts of a 1000 μg/L Al standard solution into the digestion vessels: 0.4 mL for white bread samples, 0.6 mL for seven-cereal bread and baked sole samples, and 0.5 mL for canned sardine samples. Al recoveries are 96 ± 3%, 97 ± 5%, 102 ±

Table 3. Analysis of Standard Reference Materials

reference material	exptl results[a] (μg/g of dry wt)	certified (c) or information (i) values (μg/g of dry wt)
rice flour (NIST)	4.65 ± 0.18	4.4 ± 1.0 (c)
total diet (NIST)	35.46 ± 2.85	33 (i)
fish tissue (IAEA)	16.03 ± 2.99	14.8–36.5 (i)
wheat flour (NIST)	4.43 ± 0.19	5.7 ± 1.4 (c)

[a] Mean ± standard deviation, $n = 6$.

Table 4. Repeatability and Reproducibility Expressed as the Relative Standard Deviation

sample	mean (μg/g of dry wt)	repeatability[a] (%)	reproducibility[b] (%)
milk bread	2.68	5	10
currant bread	12.05	9	5
mackerel (canned)	0.45	22	32
sardines in tomato sauce	15.34	5	9

[a] Six independent digests measured in one run. [b] Six independent digests digested and measured over 6 days.

Table 5. Al in 13 Different Types of Bread

type[a]	Al in fresh bread[b] (mg/100 g)	range[c]
white bread		
1	0.11	0.09
2	0.13	0.04
bread roll		
1	0.18	0.05
2	0.18	0.03
French stick		
1	0.20	0.01
2	0.22	0.07
French roll		
1	0.16	0.01
2	0.23	0.03
wheat whole-meal bread		
1	0.15	0.01
2	0.16	0.03
seven-cereal bread		
1	0.17	0.00
2	0.23	0.01
croissant		
1	0.24	0.00
2	0.32	0.04
currant bread		
1	0.57	0.06
2	0.92	0.06
sugar bread		
1	0.18	0.01
2	0.21	0.06
milk bread		
1	0.18	0.03
2	0.19	0.03
whole-meal bread		
1	0.23	0.02
2	0.64	0.08
brown bread		
1	0.17	0.03
2	0.19	0.04
rye bread		
1	0.25	0.05
2	0.28	0.01

[a] 1 and 2 indicate two locations of purchase. [b] Means from the measurements of two independent digests. [c] Range: the difference between the results for the two independent digests.

6%, 103 ± 10%, respectively, for canned sardines in oil, baked sole, white bread, and seven-cereal bread.

In Table 3, the results obtained for the standard reference materials are given, together with either certified or information values. The experimental results agree with the certified values or information

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Table 6. Distribution of Al in 30 Types of Fish

type[a]	Al in fresh fish[b] (mg/100 g)	range[c]	type[a]	Al in fresh fish[b] (mg/100 g)	range[c]
Fresh Fish					
herring (baked)			river eel (baked)		
1	0.05		1	0.06	0.03
2	0.01		2	0.06	0.01
turbot (baked)			salmon (cooked)		
1	0.05		1	0.01	0.01
2	0.04	0.04	2	0.02	0.00
cod (cooked)			sea eel (baked)		
1	0.01	0.01	1	0.02	
2	0.05	0.01	2	0.01	0.00
matie			sole		
1 (cooked)	0.01	0.01	1 (baked)	0.02	0.02
2 (baked)	0.00[d]		2 (baked)	0.04	
plaice (baked)			3 (raw)	0.02	
1	0.01	0.00	4 (baked)	0.01	0.00
2	0.01[d]		trout (baked)		
ray (baked)			1	0.01	
1	0.03		2	0.01	
2	0.03				
Smoked Fish					
salmon			herring		
1	0.05	0.00	1	0.02	0.01
2	0.06	0.07	2	0.01[d]	
			3	0.01[d]	
eel			4	0.01[d]	0.01
1	0.03		mackerel		
2	0.03		1	0.02	0.00
halibut			2	0.02	0.01
1	0.01[d]		salted plaice	0.03	
2	0.04		trout		
			1	0.02	
			2	0.02	
Canned Fish					
anchovies in oil			salmon nature		
1	0.12	0.04	1	0.16	0.01
2	0.06	0.01	2	0.21	0.05
Bismarck herring in mayonnaise			3	0.24	0.09
1	0.04		sardines in oil		
2	0.05		1	0.03	
Bismarck herring in vinegar			2	0.03	
1	0.02		sardines in tomato sauce		
2	0.01		1	0.39	0.05
crab nature			2	0.38	0.05
1	0.28	0.02	sardines nature		
2	1.76	0.18	1	0.16	0.06
mackerel in oil			2	0.23	0.09
1	0.09		tuna in oil		
2	0.03	0.01	1	0.03	
3	0.09	0.03	2	0.01[d]	0.00
mackerel			tuna nature		
1	0.08		1	0.02	0.00
2	0.01	0.00	2	0.01	
			3	0.01	

[a] 1–4 indicate samples of the same kind but from different commercial brands or purchased at different locations. [b] Result from single digest or from two independent digests if range is given. [c] Refers to the definitions of range given in Table 5. [d] Value below the limit of detection in solution.

values. This indicates the effectiveness of the microwave digestion and GFAAS measurements.

The use of HF in sample preparation for Al determination has been proposed because incomplete digestion might occur in the presence of silicious material (Schelenz and Zeiller, 1993; Zunk, 1990). We also checked this with the wheat flour reference material, for which, although the concentration found was within the limit certified, the result is on the low side. After the digestion with nitric acid and hydrogen peroxide, 0.5 mL of HF was added to the digests, and then the microwave heating program was repeated. However, no improvement was noticed. This was also observed in the analysis of NIES tea leaves (SRM No. 8) (Chizuko, 1988). Moreover, in our experiment HF was found to complicate the GFAAS measurement. Although we

observed that the analysis of standards containing up to 20% (v/v) HF could be performed, this was not the case for the sample measurements. After only about 10 injections, a large variation in the results occurred. In addition, a severe curvature of the calibration line was observed.

Precision. Precision was evaluated as the within-run (repeatability) and the between-run (reproducibility) relative standard deviation (RSD) obtained for two different bread and two different fish samples. The samples are milk bread and currant bread, as well as canned mackerel and canned sardines in tomato sauce, which are samples that for bread as well as for fish contain low and high concentrations of Al, respectively. The results are given in Table 4. The RSD values
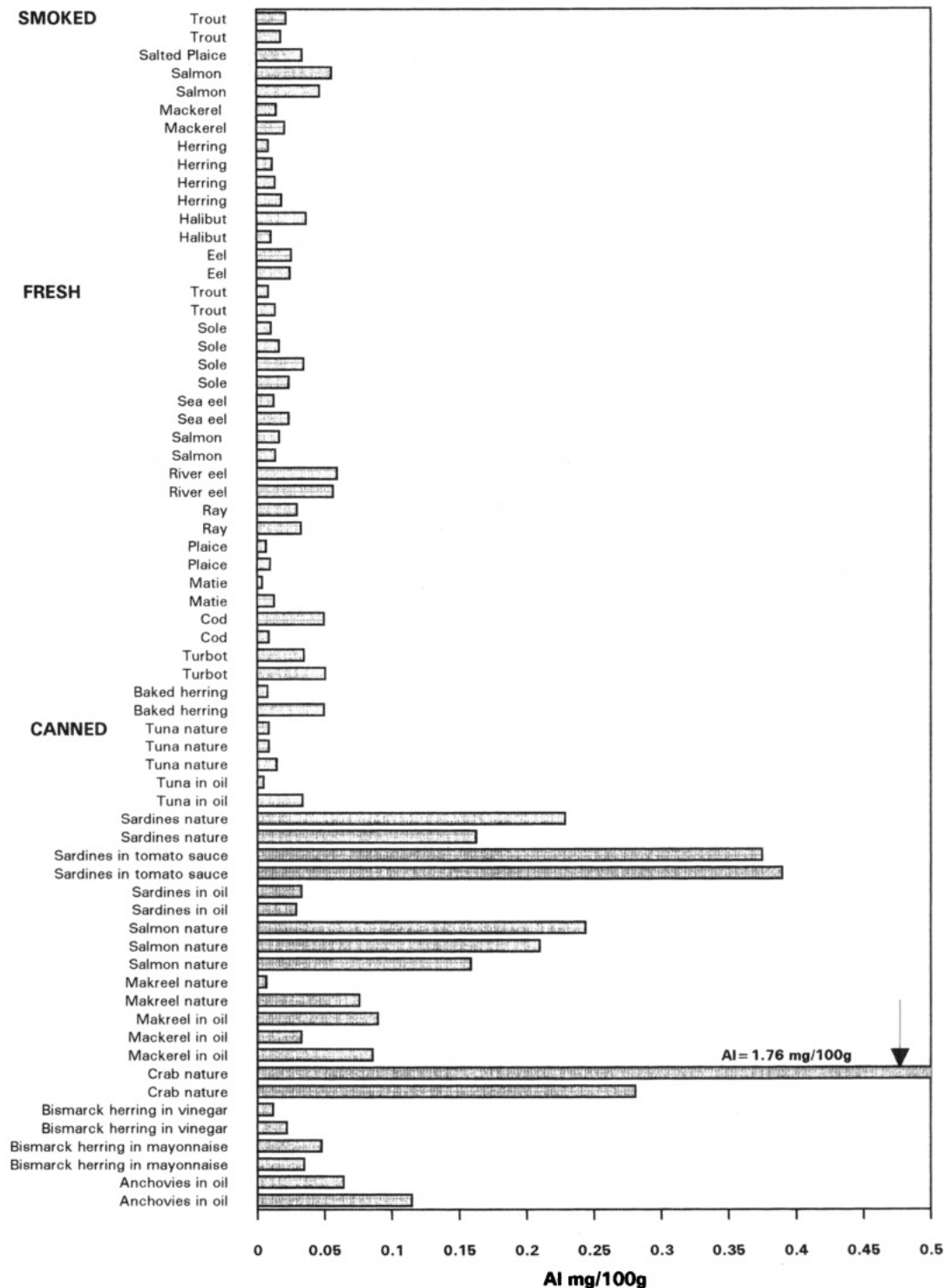
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Figure 1. Al distribution in fish samples.

indicate that, except for very low Al concentrations near the detection limit, the precision is better than 10%.

Limit of Detection. The limit of detection in solution is defined as 3 times the standard deviation of the blank absorbance divided by the slope of the calibration line (IUPAC, 1978). It was obtained from the absorbance of at least 10 independent digestion blanks and calculated to be about 3 μg/L. By taking into account the sample weight and the dilution used, we obtain the limit of detection in the dry product. For bread it is 0.30 or 0.75 μg/g for dilutions to, respectively, 10 and 25 mL; for fish it is 0.15 and 0.38 μg/g for dilutions to 10 and 25 mL, respectively.

Sample Analysis. *Bread.* In Table 5, the concen-

trations of Al in 26 bread samples are listed. The 26 samples are from 13 types of bread, purchased at two different locations. For each of these 26 samples, the Al concentration is obtained as the mean of two independent digests. The Al concentration in the fresh products is then obtained by taking into account the water content of each individual bread. The Al concentration in the bread ranges from 0.1 to 1 mg/100 g of fresh product. Currant bread contains the highest concentration of Al, which is very probably due to the high Al content of the currants present (Pennington, 1987). Except for whole-meal bread and currant bread, the interlocation variance is small, since similar results are obtained for bread samples purchased at two dif-

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ferent locations. The larger difference observed for the whole-meal and currant breads is possibly a result of the variation in Al content of the basic materials used in the production of the breads.

Fish. The concentrations of Al in 66 fish samples are given in Table 6. They are obtained from 30 types of fish purchased at at least two locations or from different commercial brands. The results given either are from a single determination or are the mean from two independent digests. As shown in Figure 1, most of the fish contains Al which is below 0.1 mg/100 g. The highest amounts are found for canned fish, especially for canned crab and sardines in tomato sauce. The two crab samples, which are from two different commercial brands, contain 0.28 and 1.76 mg of Al/100 g, while for sardines in tomato sauce values of 0.39 and 0.38 mg/ 100 g are observed.

According to Pennington (1987) most adults probably consume less than 15 mg of Al daily. Our results indicate that in general fish and bread seem to be minor food contributors to the Al intake and will not constitute a risk to human health, considering the ADI (60 mg/60 kg of body weight) established by WHO–FAO (1989). For the canned fish samples with a higher Al content, further tracing and control are suggested.

Conclusions. The proposed method has an important impact in simplifying the analysis of solid foods. The analysis time is greatly reduced by using closed-vessel microwave digestion. The proposed procedure effectively controls contamination. In addition, high concentrations of acid in sample solutions do not influence the determination. Therefore, without further treatments, a direct calibration against aqueous solutions is possible. These advantages, combined with a low limit of detection, indicate the usefulness of the method for the Al determination in a wide variety of foods. Recently, we have found that the sample preparation method is also suitable for the analysis of cadmium and lead in total diets.

The method has been successfully applied to determining Al in bread and fish. The results indicate that the contribution of these foods to the daily intake seems to be small.

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