# The Role of Sample Pre-Treatment in Analysis of Aluminium Traces in Food by GFAAS

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> Received 29 November, 1998 Accepted 22 February, 1999

#### **Abstract**

Investigations concerning the determination of nanogram amounts of aluminium by graphite furnace atomic absorption spectrometry (GFAAS) in food samples of plant origin (i.e. flour and fruit syrup) were carried out. Problems connected with the elimination of an organic matrix by different methods were shown. The test was also made to transfer the analyte to a solution using extraction.

The influence of different media used for the destruction of an organic matrix (HNO<sub>3</sub>, HClO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub>, and HF) was investigated. The obtained results were discussed.

On the basis of the obtained results the sample preparation methods for the determination of ultra traces of aluminium in flour and fruit syrups were proposed. The precision and accuracy of the proposed methods were evaluated using the certified material NBS SRM 1567a and taking into account the recovery of a standard.

Results of aluminium determination in different kinds of flour and fruit syrup of different origin and stored in containers made from different materials are also presented.

Keywords: Al determination, GFAAS, mineralization, food

#### Introduction

Medical and biological investigations carried out in recent years have proven the toxic activity of aluminium in relation to humans, animals and plants [1, 2]. It was found that this element - one of the most widespread - is an etiologic factor in the origin of several illnesses, resulting in increased interest in its metabolism and toxicity. The toxic activity of aluminium is connected with its accumulation in the body, which can lead to the impairment or even the destruction of tissue, organs and the central nervous system.

Aluminium and its compounds enter the human body through the digestive and respiratory systems. Some food products contain naturally cumulated (from soil, air, etc.) aluminium, others can be additionally contaminated during the production process and as a result of the use of food dyes, stabilizers and preservatives.

Apart from the direct toxic effect of aluminium itself, aluminium and its compounds act jointly with many metals and non-metals which may cause changes in the biological availability of some elements necessary for the proper functioning of living organisms. In the physiological processes

aluminium competes with such elements as zinc, calcium, iron, and chromium. There are also suggestions that aluminium cumulates in bones, brain and liver of people suffering from kidney disabilites and people on dialysis. It was also found that the long-term intake of drugs containing aluminium compounds (e.g. antacida) also has a negative effect on humans. The excess of aluminium was found in the brain of Alzheimer disease victims [3, 4, 5]. In the lysosoms of the brain, kidneys, and liver, aluminium replaces phosphates, in bones it replaces calcium, and in cell nuclei it replaces magnesium in heterochromatin [6, 7].

To determine the risks connected with aluminium contamination sensitive, fast, and specific methods for the determination of this element in different biological materials, physiological fluids and in environmental and food samples are needed.

Graphite furnace atomic absorption spectrometry (GFAAS) has all the above-mentioned characteristics. It is very useful for the determination of traces and ultra traces of aluminium in different materials. GFAAS is used in scientific investigations as well as for routine analysis and monitoring of the environment.

It has to be emphasized that factors like the character of

a matrix, properties of accompanying elements, substances introduced to the sample during its transfer to the solution, and instrument factors have a great impact on the analysis of ultra traces of aluminium. Many authors have dealt with investigations concerning the interference of the matrix components and searched for methods to eliminate them [8-11]. The works were mainly concerned with the efforts to use different modifiers (organic and inorganic) to eliminate the influence of a matrix in the process of the determination of different elements by GFAAS.

Interference from one of the main matrix components often has different characteristics depending on the concentration of the interferent in the sample (we dealt with this problem in our earlier work [12]). In the process of aluminium determination in different biological samples (containing calcium as one of the main components) it was necessary to use different modifiers and different temperatures of spectral analysis, depending on calcium concentration. The analysts were interested in this problem earlier; however, the investigation was conducted using exclusively standard solutions [13-15].

In the case of the analysis of biological and food samples the reagents introduced during the dissolving process are the source of great problems. Important is the presence of the perchloric acid, which is frequently used to mineralize organic matrix, and in most cases has a pronounced impact on the results of the determination of ultra traces of aluminium by flameless AAS method (the work of Slavin at al. [16] is devoted to this problem). The character of the interference from HClO<sub>4</sub> is similar to the effect of chlorides [15, 17, 18].

In our investigations related to the analysis of micro and nanogram concentrations of aluminium in a different matrix we encountered a problem connected with the fact that the end result depended on the way in which the sample solution had been prepared [19]. Generally, because of the contamination risk as well as economic factors there is a tendency to minimize pre-treatment time along with the number and amount of the reagents used for this purpose. However, one cannot overlook the credibility of the me-

thod and this is the main topic of our work. It is concerned with investigations into the determination of the nanogram concentrations of aluminium by GFAAS in food samples of plant origin in different brands of flour and fruit juices. The basic investigations were carried out for one kind of material: for the solid samples it was wheat flour and for liquid samples it was cherry syrup. For the above-mentioned samples the investigations with the purpose of optimizing the conditions for qualitative transfer of the analyte into the solution were carried out and the method for instrumental analysis was developed.

# **Experimental**

# Apparatus

- Perkin Elmer Model 2100, Atomic Absorption Spectro meter
- Perkin Elmer aluminium hollow cathode lamp
- Perkin Elmer deuterium (D2) lamp for background cor rection
- HGA-700 graphite furnace with AS-70 autosampler
- Perkin Elmer pyrolytically coated graphite tubes (with L'vov platform)
- Photolyser to mineralize samples with ultra violet radia tion (UV)

# Reagents and Materials

- Aluminium stock solution, 1000 mg/ml
- Matrix modifier Mg(NCO<sub>3</sub>)<sub>2</sub> aqueous solution of concentration 1 mg Mg/ml
- Inorganic acids: HNO<sub>3</sub>, HClO<sub>4</sub>, H<sub>2</sub>SO<sub>4</sub>, HF of "Suprapur" purity from Merck
- Water, triple distilled in a quartz distiller
- Wheat, maize, rye and potato flour
- Apple, raspberry, currant, wild strawberry, and cherry juices; of different origin and stored in various ways

Table 1. Instrumental conditions and furnace program used.

wavelength - 309.3 nm

slit - 0.7 nm

lamp current - 18 mA

the volume of the sample introduced to a graphite tube  $-5 \mu l$ 

the measurement of a peak area

universal matrix modifier  $Mg(NO_3)_2 - 5 \mu l$ ,  $(5 \cdot 10^{-2} \text{ mg})$ 

Step number	Furnace temperature [°C]	Ramp Time [s]	Hold Time [s]	Gas flow Argon [ml/min
1	90	5	10	300
2	110	5	10	300
3	400	10	10	300
4	1700	5	40	300
5	2500	0	3	0
6	2650	1	2	300
7	20	1	10	300

#### **Contamination Control**

All the vessels, quartz and polypropylene flasks were kept in 20% HNO<sub>3</sub> for 24 hours, washed many times with distilled water and left to dry under cover.

The samples were prepared taking into account the rigors of the analysis of ultra traces. For the purpose of contamination control each series of measurements included a blank. The signals from blanks depended on the samples but were never greater than 10% of the measured signal. All preparatory work was performed in a clean room.

## The Preparation of the Samples for Analysis

#### Drying of the Solid Samples

 $0.5~{\rm g}$  of each sample was dried in a vacuum dryer at  $250^{\circ}{\rm C}$  for 24 hours under 70 Pa (0.5 mm Hg) pressure.

#### The Transfer of the Analyte to the Solution

Two different methods for this stage were investigated. One, connected with the destruction of the organic matrix, was performed using different methods and media of mineralization, depending on sample consistency. The second consists of separating aluminium with the use of an acid and without the decomposition of a matrix. Furthermore, in the case of both methods of treating the solid samples (flour), some investigations were performed using initial treatment with hydrofluoric acid. The methods and mineralization media are shown in Table 2.

The sample solutions obtained as a result of the mineralization and the acid extracts were evaporated till dryness, dissolved using a solution 0.2 M HNO<sub>3</sub>, transferred into a 25 ml calibrated flasks, and diluted to volume. After that, the absorbance was measured in the conditions presented in Table 1.

#### Mineralization of the Samples

#### - Mineralization in a Teflon bomb

The investigated sample of flour (about 0.5~g) was placed in a Teflon container of the bomb, 5~ml of  $HNO_3$  or a mixture of  $HNO_3 + HClO_4$  (4+1) was added. A sample of cherry syrup (about 0.6~g) was treated with a mixture of  $HNO_3 + H_2O_2$  (5+1). The bomb containing the sample was placed in a dryer and mineralized for 1 hour at  $100^{\circ}C$  Then the sample was placed in a quartz evaporator, evaporated till dryness and the residue was dissolved and placed in a calibrated flask.

#### - Mineralization in an open system

The analyzed sample of flour (0.5 g) was placed in a quartz beaker, 5 ml of concentrated HNO<sub>3</sub> or 5 ml of a mixture of HNO<sub>3</sub> + HClO<sub>4</sub> (4+1) was added and was mineralized under a cover for 8 hours. After that, the sample was evaporated till dryness, the residue was dissolved and transferred to a calibrated flask. The same procedure was used for cherry-syrup samples; mineralization was performed in mixtures of HNO<sub>3</sub> + H<sub>2</sub>SO<sub>4</sub> (4+1) or HNO<sub>3</sub> + HClO<sub>4</sub> (4+1).

- Mineralization using a photolyser (only in the case of liquid samples)

A sample of cherry syrup (about 0.6 g) was diluted with water to volume of 5 ml, then 0.5 ml of  $HNO_3$  or  $H_2SO_4$  and 0.2 ml of  $H_2O_2$  were added and the sample was subjected to photolysis for 4 hours. After that, the sample was transferred to a calibrated flask and diluted to volume.

- Dry mineralization

About 0.5 g of flour or about 0.6 g of cherry syrup was placed in a platinum crucible, put into a cold muffle furnace and heated to 700°C; mineralization was carried out for two hours. The residue was dissolved and transferred to a calibrated flask.

- Direct mineralization in a graphite furnace

This mineralization mode was used only for liquid samples directly after their transfer into the graphite tube.

Table 2. Results of aluminium determination in flour and cherry syrup samples after wet mineralization.

1. Solid consistency (wheat flour)		2. Liquid consistency (cherry syrup)	
Mineralization system	Al found μg/g x ± SD	Mineralization system	Al found μg/g x ± SD
1.1. Teflon bomb		2.1. Teflon bomb	
a) HNO <sub>3</sub>	$1.4 \pm 0.3$	a) HNO <sub>3</sub> + H <sub>2</sub> O <sub>2</sub>	$0.26 \pm 0.02$
b) HNO <sub>3</sub> + HClO <sub>4</sub>	$1.7 \pm 0.3$		
c) HNO <sub>3</sub> + HClO <sub>4</sub> after	$3.5 \pm 0.3$	2.2. Open system	
the pre-treatment		a) HNO <sub>3</sub> + H <sub>2</sub> SO <sub>4</sub>	$0.26 \pm 0.025$
with HF		b) HNO <sub>3</sub> + HClO <sub>4</sub>	$0.26 \pm 0.02$
1.2. Open system			
a) HNO <sub>3</sub>	$1.1 \pm 0.3$	2.3. Photolysis	
b) HNO <sub>3</sub> + HClO <sub>4</sub>	$1.6 \pm 0.5$	a) $HNO_3 + H_2O_2$	$0.16 \pm 0.015$
c) HNO <sub>3</sub> + HClO <sub>4</sub> after the pre-treatment with HF	$3.3 \pm 0.4$	b) H <sub>2</sub> SO <sub>4</sub> + H <sub>2</sub> O <sub>2</sub>	$0.12 \pm 0.015$

# The Separation of Aluminium by Extraction

The experiments with the use of extraction were performed for solid samples (flour). The aim was to minimize the number of reagents used, and the number of operations performed, during initial chemical treatment to limit contamination and lower time consumption and costs of the analysis. - Extraction with nitric acid

0.5~g of flour was treated with 5 ml of  $HNO_3~(1+5)$  and subjected to extraction in a water bath (temp.  $100^{\circ}C$ ) for 2 hours. The obtained solution was filtered out, evaporated till dryness, dissolved, transferred into the calibrated flask, and its absorbance was measured

### The Methodology of Measurements

The optimal conditions for the instrumental analysis of the samples of flour and juice were selected with a view to obtaining maximal decomposition of a matrix (without losing an analyte in the process) and getting maximal analytical signal from aluminium and minimal signal from the background. The selected conditions are presented in Table 1. The dependence of the analytical signal for aluminium on the pyrolysis temperature and the temperature of the atomization for standard and the investigated matrices is shown in Figs. 1, 2.

#### **Results and Conclusions**

# The Influence of Acids and Hydrogen Peroxide on Aluminium Signal

The influence of the following reagents:  $HNO_3$ ,  $H_2SO_4$ ,  $HCIO_4$ , HF (introduced to the sample during its transfer to the solution) was investigated. Quantification was based on the measurement of a peak area absorbance. At the same time the position of the analytical signal on the axis of the atomization time was observed.

#### a) Perchloric acid

Perchloric acid causes considerable suppression of the signal, which increases with the increase of the acid concentration. At concentrations of  $HClO_4$  as low 0.2 M the absorbance is lowered by 50% (Fig. 3.). The position of the signal on the time axis practically does not change with the increase of the concentration, but at a concentration of 2 M of  $HClO_4$  a pronounced deformation of the analytical signal is observed (Fig. 5).

In the light of our experience, the most efficient way to eliminate the influence of HClO<sub>4</sub> is its removal by evaporation.

# b) Nitric acid and hydrogen peroxide

In the region of concentrations 0-4~M of  $HNO_3$  and 0-20% of  $H_2O_2$  no influence of the above-mentioned reagents on the process of aluminium determination was observed (Figs. 3, 4.). Moreover, the analytical signals from individual samples were practically on top of one another and they were not shifted on the atomization time axis (Figs. 6 and 7)

#### c) Sulphuric acid

The presence of sulphuric acid at concentrations of 0.2-1.0 M causes only a slight increase in the analytical signal

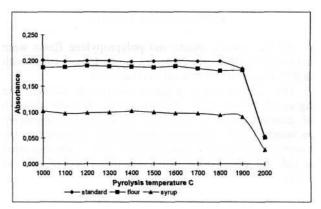


Fig. 1. The effect of pyrolysis temperature on the analitical signal of aluminium for various matrices.

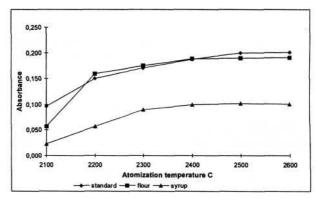


Fig. 2. The effect of atomization temperature on the analitical signal of aluminium for various matrices.

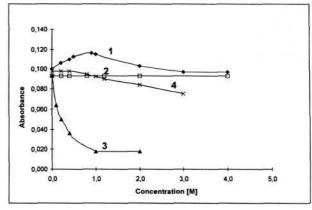


Fig. 3. The effect of acid concentration on the analytical signal from 50 ng Al/ml; 1 - H<sub>2</sub>SO<sub>4</sub>, 2 - HNO<sub>3</sub>, 3 - HClO<sub>4</sub>, 4 - HF.

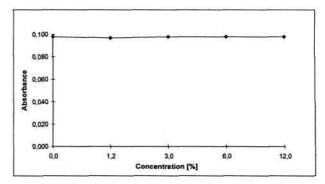


Fig. 4. The effect of  $\rm H_2O_2$  concentration on the analytical signal from 50 ng Al/ml.

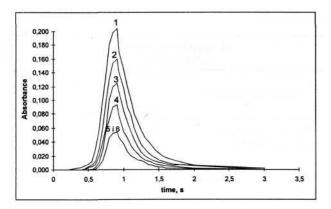


Fig. 5. The effect of the concentration of HC10<sub>4</sub> on the profile of the analytical signal from 50 ng Al/ml;

1 - 0 M HC1O<sub>4</sub>; 2 - 0 . 1 M HC1O<sub>4</sub>; 3 - 0 . 2 M HC1O<sub>4</sub>; 4 - 0 . 4 M HC1O<sub>4</sub>; 5 - 1 . 0 M HC1O<sub>4</sub>; 6 - 2 . 0 M HC1O<sub>4</sub>.

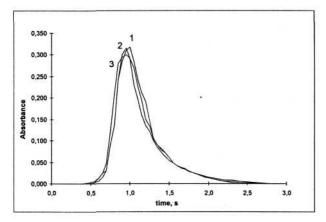


Fig. 6. The effect of the concentration of  $HNO_3$  on the profile of the analytical signal from 50 ng Al/ml;

1 - 0 M HNO<sub>3</sub>; 2 - 0.5 M HNO<sub>3</sub>; 3 - 4.0 M HNO<sub>3</sub>.

from aluminium (Fig. 3), and a small shift of the peak on the time axis in the direction of shorter atomisation times. Only at a concentration of 0.5 M of H<sub>2</sub>SO<sub>4</sub> is a shortening of atomization time clearly noticed. Moreover, at higher concentrations of H<sub>2</sub>SO<sub>4</sub> signals with double peaks appear (Fig. 8).

It is difficult to interpret the origin of the double peaks in the analytical signal from aluminium when sulphuric acid is used. Many spectroanalysts have investigated this problem in relation to the analysis process of different elements.

The appearance of double peaks in the signal from Pb in the presence of 10% of ascorbic acid was interpreted as a result of the creation of dimorphic lead oxide [8], but this was proved wrong.

Investigating the atomization mechanism of tin it was found that double peaks appear in the case of less volatile organic matrices, when during pyrolysis a lot of carbon appears. Some authors suggest [9] that active carbon reduces tin oxide in a complicated process and is responsible for the appearance of double peaks. Other researchers [10] claim that the carbon created during the thermal destruction of an organic matrix and its compounds activate the surface of a graphite tube, which causes an increase oxygen removal, and a lowering of initial atomization temperature. Imai and Hayashi [25] investigated the mechanism of lead

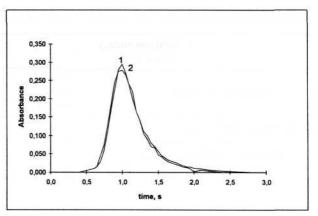


Fig. 7. The effect of the concentration of  $H_2O_2$  on the profile of the analytical signal from 50 ng Al/ml; 1-0 %  $H_2O_2$ ; 2-12 %  $H_2O_2$ .

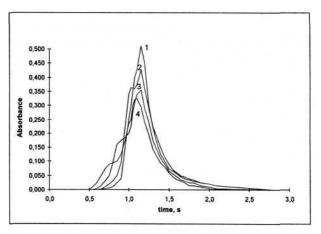


Fig. 8. The effect of the concentration of H<sub>2</sub>SO<sub>4</sub> on the profile of the analytical signal from 50 ng Al/ml;

1 - 0 M  $H_2SO_4$ ; 2-0.51 M  $H_2SO_4$ ; 3 - 2.0 M  $H_2SO_4$ ; 4 - 4.0 M  $H_2SO_4$ .

atomization in the presence of ascorbic acid at different concentrations using uncoated and coated tubes. In the case of the determination of lead in the presence of 1% ascorbic acid using uncoated tubes they observed double peaks of atomic absorption. They discovered that the double signal could be separated into two signals. The position of the first signal was in accordance with the position of the signal obtained in the absence of ascorbic acid when coated tubes were used (whereas the position of the second signal was in accordance with the position of signal in the absence of ascorbic acid when uncoated tubes were used). Their hypothesis - during the thermal destruction of the organic matrix pyrolytic graphite appears, covering some part of the surface of the tube walls. This is responsible for the appearance of the peak characteristic for the pyrolytical-lycoated tube. They showed the importance of the character and quality of the tube walls.

Salmon et al. [20] also suggest that the appearance of the signal with double peaks is connected with the properties of the internal walls of graphite tube. The authors made some experiments with the exclusive use of the tubes, which were pyrolytically coated and did not used ascorbic acid, instead using protective gas (argon containing 1% of O2). The authors claim that as a result of chemisorption permanent surface-oxides are being formed, constituting

1. Solid consistency (wheat flour)		2. Liquid consystency (cherry syrup)	
Mineralization system	Al found μg/g x ± SD	Mineralization system	Al found μg/g x ± SD
Muffle furnace temp. 700°C     a) direct sample mineralization     b) mineralization after the HF     pretreatment	$1.6 \pm 0.6$ $3.2 \pm 0.3$	2.1. Direct mineralization in a graphite tube	$0.15 \pm 0.01$

Table 3. Analysis results of the flour and cherry syrup samples after dry mineralization.

the most active sites on the surface of graphite. This differentiation in properties of the surface of graphite leads to the differentiation in the temperature at which gaseous lead is created and by the same token to the differentiation in the mechanism of the atomization process.

Some authors tried to explain the appearance of signals with double peaks on the basis of a kinetic approach to the atomization mechanism [21-25]. They have estimated the value of the activation energy and the initial temperatures for the solutions of different composition, which were investigated at different conditions using pyrolytically coated and uncoated tubes.

In summary, the approach based on the investigation of the kinetics of the process points out to the existence of two atomization mechanisms.

In light of literature data it is not easy to interpret the phenomenon of the appearance of double peaks in the analytical signal of aluminium. There is a need for deeper investigation into this phenomenon. Since aluminium is an element which shows a complex atomization mechanism, it is probable that the appearance of double peaks proves the existence of two types of atomization mechanism.

# d) Hydrofluoric acid

No influence of hydrofluoric acid in the concentration region of 0-0.4 M was found. Only above this concentration is a decrease in the analytical signal of aluminium observed (Fig. 3). This influence can be eliminated by the removal of the excess of the acid from the sample solution before the instrumental measurement, for example by evaporation.

# Methods of Analyte Transfer into the Solution and Credibility of the Results

Tables 2-4 show the results of the analysis of wheat flour and cherry syrup obtained for different methods of analyte transfer to the solution. They show that the initial treatment has a great effect on the results of aluminium determination and this is true for both types of the investigated materials.

#### Solid Samples

The foreground conclusion from the analysis of flour sample is that a two-times-bigger contents of aluminium was found when the mineralization, both wet and dry, and extraction were preceded by treatment with hydrofluoric acid. The problem of two different forms of aluminium appears here. When the sample was not pre-treated with hydrofluoric acid, only the aluminium existing in better

soluble compounds was determined. When hydrofluoric acid was used the obtained results showed higher aluminium content because this element was liberated from alumino-silicate or similar insoluble compounds.

The isolation of analyte by extraction, omitting the destruction of organic matrix (both with and without hydrofluoric acid) leads to considerable lowering of the results (Tab. 4). Moreover, it was found that nitric acid alone is not an effective medium for the quantitative transfer of aluminium into the solution; independently which mineralization method was used - a Teflon bomb or an open system (Tab. 2: 1.1a and 1.2a).

Similar results were obtained for mineralization with the use of a mixture of acids HNO<sub>3</sub> +HClO4 and for dry mineralization in a muffle furnace. The second method seems to be more competitive because of considerable shortening of mineralization time and the minimal usage of reagents, which is useful for limiting contamination (Tabs. 2: 1. lb,c and 1. 2.b, c; 3: 1. la, b).

On the basis of our results for the flour samples and other solid samples we suggest two methods for the transfer of aluminium into the solution:

Table 4. Analysis results of the flour after the extraction.

Solid cor (wheat	nsistency flour)
Extraction system	Al found $(\mu g/g)$ $x \pm SD$
a) HNO <sub>3</sub> (1 + 5)	$0.7 \pm 0.5$
b) HNO <sub>3</sub> (1 + 5) HF pre-treatment	1.8 ± 0.5

Table 5. Certified and experimental concentration of aluminium in wheat flour NBS SRM 1567a.

Method of sample pre-treatment	Found Al concentration µg/g x ± SD	Certified Al concentration µg/g x ± SD
1.1. Teflon bomb		
a) HNO <sub>3</sub> + HClO <sub>4</sub>	$5.0 \pm 0.7$	
<li>b) HNO<sub>3</sub> + HClO<sub>4</sub> after HF pre-treatment</li>	$5.2 \pm 0.8$	5.7 ± 1.3
1.2. Muffle furnace temp. 700°C  a) direct sample minerali-		
zation	$5.1 \pm 0.9$	Į.
b) mineralization after the	1	
HF pre-treatment	$5.3 \pm 0.8$	

Table 6. Aluminium content in different brands of flour samples.

No.	Sample	Al concentration μg/g x ± SD
1.	Wheat flour "Poznańska"	$1.7 \pm 0.3$
2.	Wheat flour "Krupczatka"	4.8 ± 1.0
3.	Maize flour	3.4 ± 0.7
4.	Rye flour	5.6 ± 1.0
5.	Potato flour	9.1 ± 1.3

- In the case of the determination of better soluble forms of aluminium wet mineralization in the mixture of HNO<sub>3</sub> + HclO<sub>4</sub> and dry mineralization in a muffle furnace.
- In the case of determination of total aluminium the above-mentioned methods preceded by the initial treatment of the sample with hydrofluoric acid.

Evaluation of the accuracy of the proposed methods was performed using certified reference material - wheat flour NBS SRM 1567a. The aluminium content was calculated as a mean value of eight parallel determinations. It was found out that both proposed methods are within the accuracy limits for certified aluminium content. Relevant data are summarized in Table 5.

#### Liquid Samples

The investigations carried out using samples of fruit syrup showed a great lowering of the results of aluminium determination when direct mineralization was performed in a graphite tube (about. 44%) (Tab. 3: 2.1) and in a photolyser (39% - 53%, depending on the medium) (Tab. 2: 2.3a,b). This is probably due to the incomplete destruction of the organic matrix; its residue in the solution influences the effectiveness of analyte atomization.

The rest of the methods are of equal value (Tab. 2: 2.1a and 2.2a, and b).

At the same time, for mineralization in the presence of H2SO4 less precise aluminium determination was achieved. This is probably connected with the complex mechanism of the atomization process of aluminium. This problem was discussed earlier.

The most highly recommended method for the analysis of fruit juice and other similar liquid samples was the method involving mineralization in a Teflon bomb using a mixture of HNO<sub>3</sub> + H2O2 (Tab. 2: 2.1). The advantages of this

method are: minimization of contamination, a short process time, and a lack of direct influence of the used reagents on the instrumental determination of aluminium. Because of the difficulties with finding certified material for the analyzed juices, the accuracy of the proposed method was confirmed by the recovery of the analyte, which was determined on the basis of six parallel analysis of the samples of cherry syrup spiked with 50 ng/ml of aluminium standard solution. Recovery was in the region of 94.0%-104.0%.

# The Determination of Aluminium in Samples of Flour and Fruit Juice of Different Origin

## The Analysis of Different Kinds of Flour

To determine the level of concentration of aluminium in five different kinds of flour a method of full mineralization in a closed system in a mixture of  $HNO_3 + HClO_4$  was chosen. In this case these samples the better-soluble forms of aluminium, especially apt to migration and (more importantly because of its toxic effects on living organisms, were determined. In five analyzed flours the aluminium content was in the region of 2.0-9.1 mg Al/g, depending on the kind of flour (Table 6).

In spite of the good precision of the method used, a rather high standard of deviations obtained for different kinds of flour can suggest non-homogeneous distribution of the analyte in the analysed materials. The difficulties in obtaining a higher level of homogeneity are an important problem in the determination of ultratraces of aluminium in samples of plant origin.

### The Analysis of Different Kinds of Fruit Juice

Using the method which we considered to be optimal, the content of aluminium was determined in samples of fruit juice obtained from numerous producers, different fruits and stored in various packages. The results of the analysis are shown in Table 7. In light of our investigations, as well as from literature [1], the natural level of aluminium concentration in the fruit juice is low. Nevertheless, we found out that the juice can be enriched in aluminium in the process of the production and warehousing. The biggest concentration of aluminium was found in the syrup obtained using an aluminium extractor. The concentration of aluminium in apple juice (stored in a container lined with aluminium foil) can be acknowledged as being relatively high; this drink is obtained by the multiple deletion of the

Table 7. Aluminium content in fruit syrup samples.

No.	Analyzed syrup	Container	Vendor	Al concentration $\mu g/g$ $x \pm SD$
1.	Cherry syrup	glass	Home-made	$0.26 \pm 0.02$
2.	Wild strawberry syrup	glass	"Victoria-cymes"	$0.49 \pm 0.015$
3.	Black currant syrup	glass	"Vitapol"	$0.79 \pm 0.02$
4.	Apple juice	box lined with aluminium foil	"Fortuna"	$0.36 \pm 0.025$
5.	Raspberry syrup	glass	home-made	$0.32 \pm 0.015$
6.	Black currant syrup	glass	prepared in an aluminium juice extractor	1.26 ± 0.015

concentrate. One can conclude that it would be reasonable to eliminate from the production process and storage of fruit juices the use of machines and packages made of aluminium.

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