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A simple indirect automatic method to determine total iodine in milk products by flame atomic absorption spectrometry

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ABSTRACT

A simple, precise and accurate automatic method for the determination of total iodine in milk products by indirect atomic absorption spectrometry is proposed. Iodide in solutions resulting from alkaline ashing of samples is precipitated with silver ion in a precipitation–dissolution flow manifold, which allows performing on-line the retention of the silver iodide precipitate formed on a filter, its wash with diluted ammonia and its dissolution with a diluted thiosulfate solution. Dissolved silver is also determined on-line by flame atomic absorption, and the achieved amount of this metal is proportional to that of iodine in the sample. The proposed method is very selective, avoids interferences from anions present in the samples, which can be also precipitated with silver, because these silver compounds are dissolved with ammonia at the washing step. This method allows the determination of iodine in the range 0.011–0.35 μg mL^{-1} with a relative standard deviation between 1.3 and 6.8% at a rate of ca. 17 samples h^{-1}.

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1. Introduction

Iodine is an essential micronutrient for the biosynthesis of thyroid hormones in mammals, though excessive intake of iodine may lead to thyroid disorders [1]. Seafood, iodized table salt, milk and dairy products are common sources of iodine. The iodine concentration in cow milk is strongly influenced by the intake via the feed and by season, being often assumed that iodide is the only iodine species in milk [2]. Consequently, the exact and reliable determination of low levels of iodine, to which is usually found in milk products, is of great importance for nutritional purposes [3]. Taking into account these considerations, a great variety of analytical methodologies have been proposed to determine iodine in this kind of samples. These include amperometric detection [4], ion chromatography with photodiode array, series bulk acoustic wave or electrochemical detection [5–8], gas chromatography with electron capture [9] or mass spectrometry detection [10–12], ion selective electrodes [13], optical sensors [14], neutron activation analysis [15,16], catalytic spectrophotometry [17,18], isotope dilution mass spectrometry [19,20], inductively coupled plasma optical emission spectrometry [21,22] and inductively coupled plasma-mass spectrometry, above all carrying out the alkaline solubilization of the sample using tetramethylammonium hydroxide (TMAH) [23–27]. However, most of these approaches, although sensitive, either require expensive instrumentation or suffer from the need for an extensive sample pretreatment. For this, indirect methodologies by atomic absorption spectrometry (AAS) were proposed as an alternative to determine non-metallic elements and organic species [28–33], since AAS detectors, and above all flame AAS (FAAS), are available in many research laboratories because of their relatively simple and inexpensive equipment and running cost. AAS indirect methodologies to determine iodine in milk and milk products samples are based on complexation and precipitation reactions between iodine and a metal cation acting as reagent (mercury or silver). Thus, the signal of the tag element in the AAS detector is proportional to the iodine concentration in the sample. Nevertheless, all these methods were proposed in the batch mode, so are not used widely because of their tedious operation modes, including laborious and time-consuming separation procedures (manual liquid–liquid extraction or precipitation–filtration–dissolution processes) [28–30]. In addition, extraction procedures require the use of organic solvents such as IBMK [28,29], which are expensive, environmentally unfriendly and damage health. Iodide can also be precipitated with silver and subsequently dissolved by a cyanide solution. Bermejo-Barrera et al. [30] described an off-line method to determine iodide in infant formulas by electrothermal AAS (ETAAS) based on previous on-line procedures involving precipitation of silver iodide. Namely, a first one suggested by Martinez-Jimenez et al. [34] for the simultaneous determination of chloride and iodide in foodstuffs and wine, where chloride is determined directly by dissolution with ammonia, and iodide is determined by difference, and a second methodology not applied to real samples proposed by Esmadi et al. [35,36], which presented the sequential determination of chloride and iodide

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dissolving sequentially the precipitates with ammonia and cyanide solutions, respectively, and the individual determination of iodide, where silver iodide is dissolved by a cyanide solution. In this last work is demonstrated that in the presence of other anions such as chloride, bromide and phosphate the % recovery of iodide is greater than 100% as consequence of a positive interference of these anions. Milk and infant formula are complex samples that have high mineral content including other anions that can also precipitate with silver (mainly chloride, bromide and phosphate) [37]. Silver precipitates, of these anions and iodide, are soluble in cyanide solution because of the formation of silver–cyanide complexes [38]. Therefore, it is surprising that no interfering effects were observed when a cyanide solution was used to dissolve the silver precipitate obtained from an infant formula sample and the silver signal was detected in this cyanide solution by ETAAS [30].

Flow injection (FI) is a cheap, accurate, precise simple and rapid methodology that is now firmly established to facilitate sample pretreatment and analytical chemistry automation. However, in available literature, a few FI methods for the determination of iodine in milk products have been found [4,17,18,23,24] and none of these FI methods are based on an indirect atomic absorption spectrometric approach. The coupling of a FI system to AAS has proved to be an efficient way to automate sample preparation and analysis, as well as to develop indirect methods extending the analytical scope of this analytical technique [31].

In order to overcome the problems associated with previous methods, an on-line indirect methodology to total iodine determination in milk and infant formula samples by FAAS is developed. The present method utilizes thiosulfate as dissolving solution avoiding the use of toxic cyanide, and eliminates the interference of other anions that precipitate with silver, because it is based on insolubility of silver iodide in diluted ammonia.

2. Experimental

2.1. Apparatus

The atomic absorption spectrometer used was a Perkin-Elmer 5000 (PerkinElmer Life and Analytical Sciences, Shelton, CT, USA) equipped with a silver hollow-cathode lamp and coupled to a Perkin Elmer 50 servograph recorder with a range of 5 mV. The instrument was set at 328.1 nm and silver was determined using a fuel-lean air-acetylene flame. The aspiration flow-rate of the nebulizer was adjusted to the same as the channel of the dissolving solution of the precipitate. For the flow system a Gilson® Minipuls™-3 peristaltic pump (Gilson, France), four Rheodyne® injection or switching valves, Models 5041 and 5301 (Rheodyne, Rohnert Park, USA), PTFE tubing (0.8 mm i.d.) for the reaction coil, and a stainless-steel column fitted with a removable screen-type stainless-steel filter (pore size 0.5 μm and filtration area 3 cm^2) originally designed as a cleaning device for liquid chromatography were employed. The experimental designs were performed and the results evaluated using the Statgraphics Plus.5.1 statistical software package (Manugistic, Inc., Rockville, MD, USA).

2.2. Reagents

Ultrapure water of 18.2 MΩ cm resistivity, obtained from a Milli-Q water purification system (Millipore Corporation, Bedford, MA, USA) was used for the preparation of the reagents, standards and samples. Ethanolic solutions of potassium hydroxide (2 mol L^{-1}) and calcium nitrate tetrahydrate (0.4 mol L^{-1}) were used for the alkaline ashing procedure. Sodium sulfite (1 mol L^{-1}) and nitric acid (1 mol L^{-1}) solutions were used for the elimination of carbonates. A silver solution (0.75 mol L^{-1}) was prepared by dissolving 12.7425 g of silver nitrate in 100 mL of 1% (V/V) nitric acid. The iodide standard solution (100 μg mL^{-1}) was prepared from their potassium salt (dried at 110 °C) in ultrapure water. Working solutions were prepared daily by appropriate dilution. Ammonium thiosulfate (0.3 mol L^{-1}) and ammonia (2 mol L^{-1}) solutions were prepared by appropriate dilution in ultrapure water. All reagents used were of analytical grade (Merck Darmstadt, Germany). To avoid any contamination, all equipment was washed and kept for at least 48 h in 10% (V/V) nitric acid solution. Before being used, the equipment was rinsed several times with ultrapure water.

2.3. Samples and sample preparation

Infant formulas used as baby foods and powdered milks analyzed are commercially available in Spain. The method was validated using a certified reference material, CRM No. 151, skim milk powder (spiked) with a reported certified iodine content of 5.35 ± 0.14 μg g^{-1}, supplied by the Community Bureau of Reference (BCR, Brussels, Belgium).

Organic matter of samples is destroyed by an alkaline ashing technique by using potassium hydroxide and calcium nitrate as described elsewhere [22]. This decomposition procedure was selected because it is large safe and reproducible, iodine losses are kept at a minimum, and convert all iodine species to water-soluble iodide. Thus, 250 mg of sample was placed into a borosilicate test tube, potassium hydroxide solution (2 mL, 2 mol L^{-1} in ethanol) and calcium nitrate solution (2 mL, 0.4 mol L^{-1} in ethanol) were added to the sample and mixed by agitation. The mixture was dried in a drying oven at 70 °C (for 10 h) and then at 120 °C (5 h). The test tubes with the dried mixtures were then heated in muffle furnace (2 h at 240 °C, 1 h at 340 °C, 1 h at 440 °C and 4 h at 500 °C). When incineration was complete, sample ash was transferred quantitatively to a beaker and 10 mL of ultrapure water, sodium sulfite (1.3 mL, 1 mol L^{-1}) and nitric acid (1 mL, 1 mol L^{-1}) were added. As a result, carbon dioxide from carbonates was then eliminated by gentle boiling of the mixture and solid residue is dissolved at the same time. The resulting solution is then transferred to a volumetric flask and filled up to 10 mL by ultrapure water. The pretreatment with sodium sulfite and diluted nitric acid including in the procedure, allows the elimination of carbonates, which precipitate also with silver. Owing to the non-quantitative nature of ashing, the blank solution and calibration standards should be put through the ashing process with identical conditions as described previously for samples.

2.4. Continuous determination of total iodine by FAAS

The procedure consisted of three steps: (1) sample introduction and analyte precipitation, (2) washing of the precipitate, and (3) dissolution of the precipitate, and tag element (Ag) detection.

2.4.1. Sample introduction and analyte precipitation

The sample or standard solutions (2 mL) containing between 0.011 and 0.35 μg mL^{-1} iodine (as iodide) and the precipitating reagent (0.75 mol L^{-1} silver solution) are continuously passed through the flow manifold depicted in Fig. 1. In this step, both streams merged on-line and are mixed in the precipitation coil, where the precipitation occurs. The silver precipitate formed is retained on the filter device. During this step, the washing solution stream flows within its respective flow circuit, which send it to waste via SV1. At the same time, SV2 allows to send the filtrate to waste and select an ultrapure water channel to be aspirated by the nebulizer to flush it after each measurement. The duration of this step is 120 s.

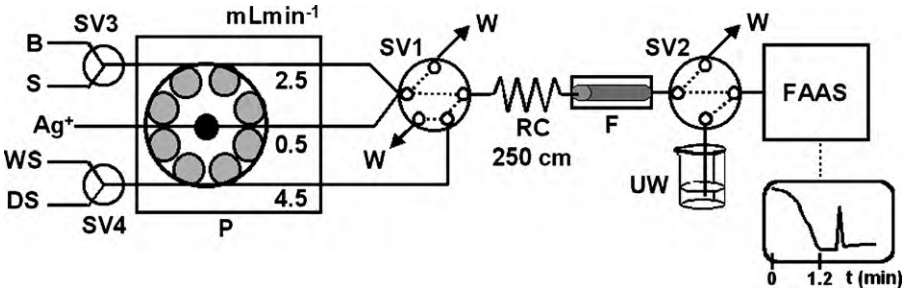

Fig. 1. Precipitation–dissolution flow manifold for the indirect determination of total iodine. Ag^+, precipitating reagent (0.75 mol L^{-1} silver solution); B, blank solution; DS, dissolving solution (0.3 mol L^{-1} thiosulfate); F, filter; FAAS, flame atomic absorption spectrometer; P, peristaltic pump; RC, reaction coil; S, sample; SV1–SV4, selecting valves; UW, ultrapure water; W, waste lines; WS, washing solution (2 mol L^{-1} ammonia).

2.4.2. Washing of the precipitate

After SV1 was actuated to introduce in the flow system the washing solution (diluted ammonia solution stream, 2 mol L^{-1}) for about 50 s (until zero response was obtained in the detector). The duration of this step was about 70 s.

2.4.3. Dissolution of the silver iodide precipitate and tag element (Ag) detection

The dissolving solution (0.3 mol L^{-1} thiosulfate) is introduced into the flow system through the same channel used for the washing solution selected by SV4. This solution flows through the filter to dissolve the silver iodide precipitate. Finally, dissolved silver iodide is transported on-line to the nebulizer for silver detection. The duration of this step is about 12 s.

3. Results and discussion

3.1. Sample pretreatment

The recovery of the alkaline ashing procedure was tested performing the proposed pretreatment with a sample with and without spiked with iodide (0.05 μg mL^{-1}). The recovery of the ashing step was found to be 68 ± 3% (five replicates). Recovery values are repeatable when keeping identical the experimental conditions. Accordingly, owing to the non-quantitative nature of ashing, calibration standards should be put through the ashing process with identical experimental conditions.

3.2. Optimization of the total iodine determination

Seven variables namely: sample flow-rate (A), silver solution flow-rate (B), flow-rate of the dissolving solution (C), reaction coil length (D), silver solution concentration (E), washing solution concentration (diluted ammonia solution) (F) and dissolving solution concentration (thiosulfate solution) (G) were selected to be examined in order to optimize the total iodine determination. For the evaluation of these variables at two levels a Plackett-Burman 2^7*3/32 design with one centerpoint involving 13 randomised runs was used in order to test statistical significance of the effects instead of the 27 experiments required for a full factorial design. The Plackett-Burman design makes possible to screen the influence of variables (A–G) with only 13 experiments. Thus, the optimization method allows estimation of the main effects of the variables and disregards interactions between them. Experimental matrix of the Plackett-Burman design summarizing the 13 experiments are shown in Table 1, which includes the low and the high levels for each variable. In order to keep constant the sample mass for all experiments, each experiment was run by continuously introducing 2 mL of a certified reference material (CRM No. 151) prepared as is described previously. The effect of changing a variable from low to high level value was examined on a selected response such as percentage recovery (mean of three replicates). The iodine recovery for each experiment is also presented in Table 1. The data obtained were evaluated by an ANOVA test, and the main effects visualized using the Pareto chart (Fig. 2A). In this graphic, the bar lengths are proportional to the absolute value of the estimated main effects. This figure also includes a vertical line corresponding to 95% confidence interval. An effect, which exceeds this reference line, may be considered significant as regards to response. Thus, the most important variables were sample flow-rate (A) and silver solution flow-rate (B). The sign of the effect showed that the response improved on passing from the highest to the lowest flow-rates. These variables are related to kinetics of the precipitation reaction, because if the total flow-rate is too high, the residence time of the precipitate in the reaction coil was short, which entailed incomplete precipitation. In this way, as can be seen in Fig. 2B (response surface graph), both variables were interrelated, when sample flow-rate was high and silver solution flow-rate was low, percentage recovery was quantitative (higher than 95%), and vice versa. As a time-based FI procedure was proposed in this methodol-

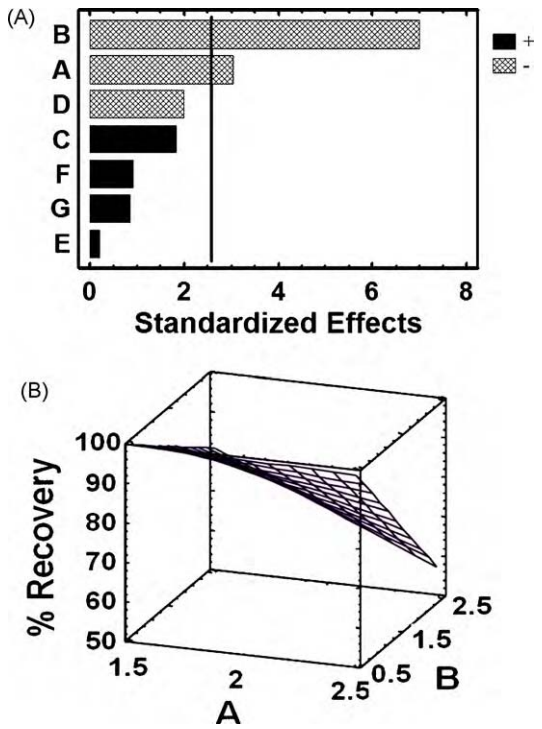

Fig. 2. (A) Pareto chart of the main effects obtained from Plackett-Burman 2^7*3/32 factorial design. (B) Response surface from the Plackett-Burman design considering the variables sample flow-rate (mL min^{-1}) (A) and silver solution flow-rate (mL min^{-1}) (B).

Table 1
Experimental variables, levels, Plackett-Burman matrix and response values.

Variable (units)	Key	Low level	High level
Sample flow-rate (mL min^{-1})	A	1.5	2.5
Silver solution flow-rate (mL min^{-1})	B	0.5	2.5
Flow-rate of the dissolving solution (mL min^{-1})	C	3.0	4.5
Reaction coil length (cm)	D	200	300
Silver solution concentration (mol L^{-1})	E	0.5	1.0
Washing solution concentration (mol L^{-1})	F	1.0	3.0
Dissolving solution concentration (mol L^{-1})	G	0.1	0.5

Run	A	B	C	D	E	F	G	% Recovery
1	2.5	0.5	4.5	300	0.5	3.0	0.1	98.4
2	2.5	2.5	3	300	0.5	1.0	0.1	44.6
3	1.5	0.5	3	300	1.0	3.0	0.1	96.1
4	2.5	2.5	3	300	1.0	1.0	0.5	60.8
5	1.5	2.5	4.5	300	0.5	3.0	0.5	76.7
6	1.5	2.5	3	200	0.5	3.0	0.5	80.6
7	1.5	2.5	4.5	200	1.0	1.0	0.1	86.9
8	2.5	0.5	4.5	200	0.5	1.0	0.5	96.2
9	2.5	2.5	4.5	200	1.0	3.0	0.1	64.4
10 (CP)[a]	2.0	1.5	3,75	250	0.75	2.0	0.3	90.9
11	2.5	0.5	3.0	200	1.0	3.0	0.5	95.3
12	1.5	0.5	3.0	200	0.5	1.0	0.1	99.9
13	1.5	0.5	4.5	300	1.0	1.0	0.5	100.1

[a] Central point.

ogy, the highest sampling throughput was obtained at the highest sample flow-rate. Therefore, as a compromise, a sample flow-rate of 2.5 mL min^{-1} and a silver solution flow-rate of 0.5 mL min^{-1} were chosen for further experiments. With regard to other variables: flow-rate of the dissolving solution (C), reaction coil length (D), silver solution concentration (E), washing solution concentration (diluted ammonia) (F) and dissolving solution concentration (thiosulfate solution) (G) were not significant. Taking into account these results, the latter four experimental factors were fixed at the central level: 250 cm (D), 0.75 mol L^{-1} (E), 2 mol L^{-1} (F) and 0.3 mol L^{-1} (G). The aspiration flow-rate of the nebulizer was adjusted to be the same as the flow-rate of the dissolving solution to keep constant the flow-rate of the FI system. Therefore, as the greatest aspiration flow-rate provides better sensitivity and the flow-rate of the dissolving solution had a positive effect, this variable was fixed in 4.5 mL min^{-1}. In addition, as was used the same flow-rate for the washing solution, this allows to short the washing step.

3.3. Study of interferences

Potential interferences with the determination of total iodine in milk products using the proposed FI method are those anions usually present in milk samples that yield a precipitate with silver ion in slightly acidic media (chloride, bromide, phosphate, sulfate, citrate and carbonate). These anions were assayed at concentrations up to 50 times that of iodide (0.3 μg mL^{-1}). The results obtained, shown in Table 2, indicate that the tolerable levels for these anions are satisfactory because an error of less than ±5% in the peak absorbance was obtained for all the cases. As a result, calculated recoveries ranged between 95.3 and 100.7%. The high selectivity of the proposed method can be explained by the fact that, even though these anions precipitate with silver ion, their precipitates are soluble in 2 mol L^{-1} ammonia.

Table 2
Interference studies (0.3 μg I mL^{-1}).

Species	Concentration (μg mL^{-1})	% Recovery
Chloride	15	99.8
Bromide	15	96.2
Phosphate	15	98.0
Sulfate	15	95.3
Citrate	15	100.7
Carbonate	15	97.1

3.4. Analytical performances

Standard calibration and standard addition graphs were obtained under the optimum chemical and flow conditions stated in Fig. 1. These graph equations with the linear regression between absorbance (A) and the iodine concentration (X in μg I per mL) were:

Standard calibration :

$$A = 0.3728X + 0.0002 \; (X = \; 0 - 0.35 \; \mu g \, mL^{-1}; \; r = 0.999; \; n = 9)$$

Standard addition :

$$A = 0.3675X + 0.0495 \; (X = \; 0 - 0.2 \; \mu g \, mL^{-1}; \; r = 0.999; \; n = 5)$$

The t-test was used to compare the slopes of both graph equations, leading to conclude that no significant difference was observed between them at a 95% confidence level, demonstrating

Table 3
Total iodine concentration ± standard deviation (n = 3) in infant formulas and powder milk samples and paired t-test results.

Sample	Total iodine (μg g^{-1}) Proposed method	Total iodine (μg g^{-1}) SP-C determination [17]
Infant formula 1	0.90 ± 0.10	1.03 ± 0.16
Infant formula 2	0.57 ± 0.06	0.53 ± 0.29
Infant formula 3	0.77 ± 0.06	0.73 ± 0.05
Infant formula 4	0.50 ± 0.10	0.57 ± 0.14
Infant formula 5	0.73 ± 0.06	0.69 ± 0.06
Infant formula 6	0.67 ± 0.06	0.71 ± 0.08
Infant formula 7	0.47 ± 0.06	0.44 ± 0.08
Powder milk 1	6.67 ± 0.12	6.72 ± 0.06
Powder milk 2	4.60 ± 0.10	4.72 ± 0.09
Powder milk 3	5.23 ± 0.12	5.02 ± 0.07
Powder milk 4	3.50 ± 0.10	3.75 ± 0.06

SP-C: Spectrophotometric catalytic determination. Experimental value of t = 0.71; critical value of t ($n - 1 = 10$; $P = 0.05$) = 2.23.

Table 4
Comparison of figures of merit of the present method with some previously reported for iodine determination in milk products.

Detection	DL ($\mu g\,L^{-1}$)	Recovery (%)	RSD (%)	Reference
A	0.6	No data	1.68–3.03	[4]
ICE	0.5	98.1–102.2	6.2	[5]
ICSBAW	5	97–105	3.9	[8]
GC-EC	0.11	No data	3.5–10.4	[9]
GC–MS	0.010–0.025	96.5–107.0	2.6–4.5	[12]
OS	94.4	100.6	1.38–2.83	[14]
NAA	$0.06\,\mu g\,g^{-1}$	No data	1–13	[16]
SP-C	0.99	94.5–105	5	[17]
IDA	5	No data	<14	[19]
ICP-OES	2 ($40\,\mu g\,g^{-1}$) 7 ($280\,\mu g\,g^{-1}$)	85–98	2–3	[21]
VG-ICP-OES	20	94–102	0.5–3.5	[22]
ICP-MS	0.001–0.002	97–98	3–4	[25]
ICP-MS	$7 \times 10^{-7}\,\mu g\,g^{-1}$	No data	7–15	[27]
ETAAS	3.7	98.1	<10	[28]
ETAAS	2.4	97.5–100.6	16.9	[29]
ETAAS	1.2 ($3.1\,\mu g\,g^{-1}$)	100	0.6–12.3	[30]
FAAS	2.75 ($0.11\,\mu g\,g^{-1}$)	98.1	1.3–6.8	Present method

A: amperometry; DL: detection limit; ETAAS: electrothermal atomic absorption spectrometry; FAAS: flame atomic absorption spectrometry; GC-EC: gas chromatography with electron capture detection; GC–MS: gas chromatography–mass spectrometry; ICE: ion chromatography-electrothermal detection; ICP-OES: inductively coupled plasma optical emission spectrometry; ICP-MS: inductively coupled plasma-mass spectrometry; ICSBAW: ion chromatography with series bulk acoustic wave detection; IDA: isotope dilution analysis; NAA: neutron activation analysis; OD: optical sensor; SP-C: spectrophotometric catalytic; VG-ICP-OES: vapour generation inductively coupled plasma optical emission spectrometry.

that iodine determination is free of matrix interferences. The standard addition curve as series of absorbance peaks are presented in Fig. 3.

Using the selected experimental conditions, the limit of detection (LOD) and quantification (LOQ), precision, accuracy and sample throughput of the procedure for the determination of total iodine in milk products were checked. LOD and LOQ were calculated according to 3Sb and 10Sb criteria, were Sb is the standard deviation of 15 independent measures of a blank. The obtained LOD and LOQ were 2.75 and 10.5 $\mu g\,L^{-1}$, respectively (0.11 and 0.42 $\mu g\,g^{-1}$ for 250 mg of sample). Repeatability (expressed as relative standard deviation, %RSD) checked on samples containing 0.75 and 2.55 $\mu g\,g^{-1}$ iodine ($n = 11$) was 6.8 and 1.3%, respectively. The accuracy of the procedure was evaluated using the reference material CRM No. 151 with a certified iodine content of $5.35 \pm 0.14\,\mu g\,g^{-1}$. The I content obtained (mean ± SD, $n = 3$) was $5.25 \pm 0.09\,\mu g\,g^{-1}$, which agrees with the certified value. Mean recovery obtained by comparison with certified value was 98.1%. The sample throughput achieved was ca. 17 samples h^{-1}.

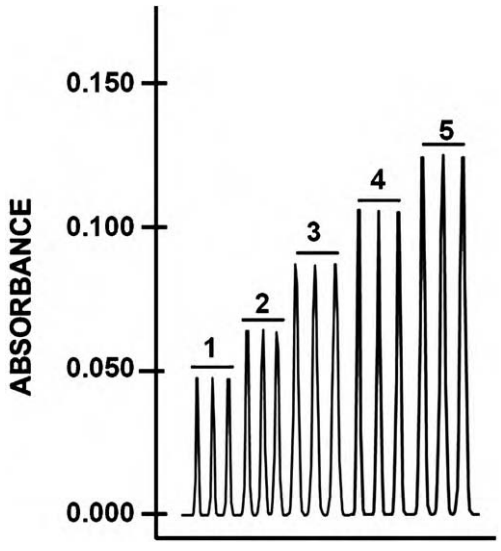

Fig. 3. Standard addition curve as series of absorbance peaks. 1: 250 mg of CRM No. 151; 2: 250 mg of CRM No. 151 + 0.05 $\mu g\,mL^{-1}$ I; 3: 250 mg of CRM No. 151 + 0.1 $\mu g\,mL^{-1}$ I; 4: 250 mg of CRM No. 151 + 0.15 $\mu g\,mL^{-1}$ I; 5: 250 mg of CRM No. 151 + 0.2 $\mu g\,mL^{-1}$ I.

3.5. Analysis of samples

The proposed method was applied to the determination of iodine in commercial infant formulas and powder milk samples. The results obtained are shown in Table 3. The total iodine ranged from 0.47 to 0.90 $\mu g\,g^{-1}$ and from 3.50 to 6.67 $\mu g\,g^{-1}$ for infant formulas and powder milk samples, respectively.

The samples were also analyzed by a classical spectrophotometric methodology based on the catalytic action of iodide on the color-fading reaction of the $FeSCN^{2+}$ complex [17]. The results obtained are also shown in Table 3. Applying the paired t-test at the level of 95% confidence no statistical difference was observed among the values obtained by both methodologies.

4. Conclusions

A miniaturized, fast, simple, practical and low-cost assay is developed for determination of total iodine as iodide in complex matrices, such as infant formulas and powder milk samples. The most outstanding feature of this approach is that avoids possible positive interferences in silver detection, which are not taken into account in previous works, because the proposed method is based on the insolubility of silver iodide in a diluted ammonia solution, and so the silver detected only comes from the silver iodide precipitate. In addition, this method avoids the use of extremely toxic cyanide, which was the dissolving solution chosen in previous works. Everything which contributes to improve the quality of the analytical methodology and to achieve a greener analytical chemistry. In a view glance to the data in Table 4, it is clear that the detection limit of inductively coupled plasma-mass spectrometric methodologies is better than those obtained with other detection techniques including that obtained by the proposed method. However, detection limit achieved is similar to that obtained by other indirect atomic absorption [28–30] and inductively coupled plasma optical emission spectrometric methodologies [21,22]. Nevertheless, the present method is superior in term of precision (expressed

as %RSD) and utilizes a simple instrumentation with an easy functioning and a low cost of acquisition and maintenance as FAAS. This method showed enough sensitivity for the determination of total iodine in infant formulas and powder milk samples, and it may be adapted to the determination of total iodine in other food types.

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