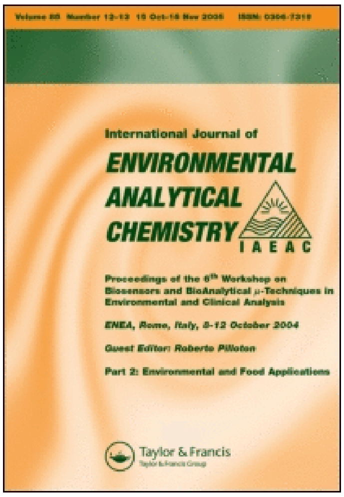

International Journal of Environmental Analytical Chemistry
Publication details, including instructions for authors and subscription information:
http://www.informaworld.com/smpp/title~content=t713640455

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To cite this Article Aziz-alrahman, A. M.(1984) 'An Atomic Absorption Spectrophotometric Method for the Determination of Trace Amount of Zinc in Canned Juices After Ion Exchange Separation', International Journal of Environmental Analytical Chemistry, 19: 1, 55 — 61

To link to this Article: DOI: 10.1080/03067318408077017

URL: http://dx.doi.org/10.1080/03067318408077017

Intern. J. Environ. Anal. Chem., 1984, Vol. 19, pp. 55–61
0309-7319/84/1901-0055 $18.50/0

An Atomic Absorption Spectrophotometric Method for the Determination of Trace Amounts of Zinc in Canned Juices After Ion Exchange Separation

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An atomic absorption spectrophotometric method is described for the determination of microgram quantities of zinc in canned juices. After sample digestion in concentrated nitric acid, the solution is evaporated till near dryness, and then a solution of 2 M HCl is added to form tetrachlorozincate (II) ion. This acid solution, containing the zinc complex is passed through an ion-exchange column (anion exchange resin, chloride form, which is preconditioned by passing 1 M HCl solution). Zinc is eluted from the column with 0.01 M HCl solution. After evaporation to dryness, the residue is dissolved in 1% (v/v) HNO_3, and then atomized into an air-acetylene flame. The limit of detection of the method is $0.15\,\mu g\,ml^{-1}$ Zn. The analytical aspects of the proposed method, including the standard addition technique are discussed.

KEY WORDS: Zinc determination, atomic absorption spectrophotometry, solvent extraction, canned juices analysis.

INTRODUCTION

The United State Environmental Protection Agency has defined that zinc and ten other metals as hazardous elements.[1] Zinc is an essential and beneficial element in body growth, but concentrations above 5 $\mu g\, ml^{-1}$ of zinc can give a bitter astringent taste for drinking waters.[2] It is known that zinc is one of the less hazardous elements, but its toxicity may be enhanced by the presence of arsenic, lead, cadmium and antimony.[1] It is well known too that acid foods and beverages can dissolve enough zinc from the galvanized containers to cause poisoning. Therefore, there is a continuing need to determine this metal in canned juices at trace levels.

Undoubtedly analytical atomic spectroscopic techniques are the most convenient and sensitive methods for determining microgram quantities of this element. Flame atomic absorption and plasma spectroscopic methods were used for determining zinc in different juices.[3-5] The atomic absorption methods are based on the direct atomization of the samples, after centrifuging and dilution process;[3] and the solvent extraction of the metal from the acid solutions of orange and lemon juice samples, as zinc diethyldithiocarbamate complex into methylisobutyl ketone (MIBK).[4] The plasma method depends on carbonizing the orange juice samples under heat lamps, ashing in a muffle furnace and then dissolving the ash in 0.1 M HNO_3.[5]

Preliminary studies by the author,[6] revealed that zinc can be extracted as iodide complex from acid solutions containing 12% (m/v) potassium iodide, into MIBK, but the acid recovery for zinc was about 50% which was not encouraging.

A close examination to the chemistry of the metal revealed that Zn(II) ion form tetrachloro complex in HCl media. The formed tetrachlorozincate(II) ion can exchange with anion exchange resin. As the concentration of HCl in the eluent is decreased the complex will break down to the simple hydrated cation. The affinity of the metal for the resin will be lost, and it will be eluted.[7-9]

The proposed method is based on this technique, which was fully established after obtaining the optimum conditions for analysis. The acid recovery for zinc was 100%. The method, results and the analytical aspects for determining trace amounts of zinc in canned juices are presented and discussed in this paper.

EXPERIMENTAL

Apparatus

A Varian Model AA-475 double-beam atomic absorption spectrophotometer, equipped with deuterium background corrector was used with an air-acetylene flame. The resonance line at 213.9 nm from a zinc hollow-cathode lamp (Varian) was employed with a slit width of 0.5 nm on the spectrophotometer. The lamp current was 5 mA and the flow rates of air and acetylene were 7.0 l min^{-1} at 60 psi and 1.5 l min^{-1} at 12 psi respectively. The instrumental response being optimized according to the manufacturers instructions.

Anion-exchange columns

These columns contained Amberlite IRA-400 (100–200 mesh, Cl^- form) resin. Excess fines were removed by floatation before use. The resin-bed was 180 mm deep and 23 mm in diameter. The columns were preconditioned with 200 ml 1 M HCl.

Reagents

Analytical reagent grade reagents were used.

Hydrochloric acid (sp. gr. 1.18).

Nitric acid (sp.gr. 1.42).

Standard zinc nitrate solution (1000 μg ml^{-1} of Zinc). Dissolve 1.00 g of zinc metal in 30 ml (1 + 1) concentrated HNO_3 and dilute to 1 l, using double-distilled de-ionized water.

Materials

Six international and two national commercial brands of canned juices were selected for determining their zinc contents. Seventeen samples of these eight brands were purchased at food markets and subjected to the proposed method.

Analysis of the samples

Pipette 5 ml of the sample into a 100-ml beaker, add 30 ml of concentrated HNO_3 and heat till near dryness and then add 100 ml 1 M HCl. Pass the solution through the anion-exchange column, and then wash the column by passing 200 ml of 1 M HCl. Elute the zinc, using 200 ml 0.01 M HCl and collect the eluate in a 500-ml. beaker. Evaporate the solution to dryness and then leave to cool. Dissolve the residue in 30 ml 1% (V/V) HNO_3 and transfer to a 50-ml volumetric flask. Wash the beaker with 10 ml 1% (V/V) HNO_3 and transfer to the volumetric flask. Make up the volume to the mark, using the 1% HNO_3 solution. Atomize this solution into an air acetylene flame. Read off the zinc contents of the sample from the calibration graph of the metal.

Preparation of calibration graphs

Prepare a $10\,\mu g\,ml^{-1}$ Zn solution by appropriate dilutions of the standard Zn solution, using 1% (V/V) HNO_3. Into six 100-ml volumetric flasks, pipette 0, 3, 6, 9, 12 and 15 ml of the $10\,\mu g\,ml^{-1}$ Zn solution. Make up the volumes to the mark, using the 1% HNO_3. Atomize these zinc standard solutions ($0.0-1.5\,\mu g\,ml^{-1}$ Zn) into an air-acetylene flame, employing the given instrumental settings.

RESULTS

The resonance line at 213.9 nm from the zinc hollow-cathode lamp was employed to construct calibration graphs for $0.0-1.5\,\mu g\,ml^{-1}$ of zinc for the determination of zinc between 0.50 and $12\,\mu g\,ml^{-1}$ Zn by atomizing the standard solutions and measuring absorbances versus concentration, followed by atomizing and measuring the absorbances of the samples solutions. The concentration of zinc in the samples were deduced from the calibration graphs. The background corrector was switched on during all runs, in order to correct for molecular absorption and/or scattering of light that might occur in the region of 213.9 nm resonance line.

The calibration graphs were straight lines through the origin, slightly curved at the upper end towards the concentration axis. The concentration of Zn which gives 1% absorption was about $0.01\,\mu g\,ml^{-1}$ Zn. Seventeen samples of canned juices were analyzed according to the described method, in order to obtain their zinc contents. The results are shown in Table I. The relative standard deviation of the method is 5.7%. The limit of detection is $0.15\,\mu g\,ml^{-1}$ Zn, calculated as twice the standard deviation for 12 determinations of an apple juice sample. The probable error, P,[10–13] which equals $0.67\,\sigma$, where σ the standard deviation, is $0.05\,\mu g\,ml^{-1}$ Zn.

TABLE I
Results for the determination of zinc in canned juices

Sample no	Type of canned juice	Zinc found ($\mu g\,ml^{-1}$)
1	Tomato	6.0
2	Tomato	5.0 & 5.2[a]
3	Orange	3.0
4	Orange	1.9
5	Apple	1.3[b]
6	Apple	2.2
7	Apple	1.5
8	Apple	1.3
9	Mango	3.8
10	Mango	2.0
11	Pineapple	2.9
12	Pineapple	4.8
13	Pineapple	1.7
14	Grape (red)	1.7
15	Grape (white)	2.0
16	Lemon	2.9
17	Grapefruit	1.4

[a] Using standard addition technique.
[b] Average of 12 determinations. Other quoted results are the average of 2 determinations.

DISCUSSION

Before analyzing the samples, a test for recovery of zinc in acid solutions was carried out, by analyzing a set of 10 sample solutions containing 0.90 $\mu g\,ml^{-1}$ Zn, according to this described method. The average of zinc amounts recovered was 0.90 $\mu g\,ml^{-1}$ Zn, which indicated that the method ensures 100% recovery of the metal, and hence the method was fully established for analyzing the canned juice samples.

Sample No. 5, an apple juice (Table I), was analyzed twelve times, in order to obtain the precision and detection limit of the method. The reported limit of detection of 0.15 $\mu g\,ml^{-1}$ of zinc could probably be improved further if one sample of canned juice could be found that contained less than 1.3 $\mu g\,ml^{-1}$ of zinc.

To verify the results reported in Table I, standard addition technique was carried out in analyzing sample No. 2, a tomato juice.

A zinc content of $5.2\,\mu g\,ml^{-1}$ Zn was found. This value is in a very good agreement with the value of $5.0\,\mu g\,ml^{-1}$ Zn, reported in Table I.

The zinc concentration of U.S. drinking waters, might reach upto $7.0\,\mu g\,ml^{-1}$ Zn. This high level is due to the zinc uptake, which is attributed to the deterioration of galvanized iron and the dezincification of brass.[2] A test for this zinc uptake of juices due to the storage in cans, was carried out. A pineapple juice sample of the same brand of sample No. 12 (Table I), was analyzed after the expiry date. The zinc content of the sample was $7.4\,\mu g\,ml^{-1}$ Zn, which was the average of two determinations. This result verifies the zinc uptake of juices stored in cans.

To compare the reliability and accuracy of this proposed method, with the other methods which were used for determining zinc in different juices,[3-5] a close examination to the chemistry of the methods must be taken into consideration. In the first atomic absorption method,[3] which based on centrifuging and dilution process, the precipitate may retain some of the zinc and other elements being determined. In the second one, the solvent extraction method,[4] the pH of the solution must be rigidly controlled, to prevent the hydrolysis of Fe(III) ion, where zinc and other metals of interest coprecipitate with the hydrated Fe(III).[14,15] In plasma spectroscopic method, all samples were prepared for analysis by dry ashing,[5] which is a time consuming procedure, plus the fact that dry ashing is not suitable for many elements having low boiling points, such as zinc, because the ashing process might lead to incomplete recovery, due to the partial volatilization of these elements.[16,17]

There is every reason to believe that the results reported in Table I, are accurate. This proposed method is fast, reliable, accurate and gives reproducible results for zinc in canned juices. Up to 50 samples can by analyzed during the working hours of any production laboratory, when using a microprocessored atomic absorption spectrophotometer, equipped with a deuterium background corrector and an auto-sampler. The results can be obtained directly from the read-out device, after calibrating the instrument, using three different concentrations of zinc.

Acknowledgement

The author thanks Mr. M. A. Mohammed for the technical assistance.

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