

Determination of Copper, Iron, Lead, Cadmium, Cobalt and Nickel by Atomic Absorption Spectrometry in Baking Powder and Baking Soda Samples after Preconcentration and Separation

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ABSTRACT

The contents of copper, iron, lead, cadmium, cobalt and nickel in baking powder and baking soda samples have been determined by atomic absorption spectrometry after separation and preconcentration. Two different separation/preconcentration methods including APDC-Chromosorb 102 solid phase extraction method and $\text{Ce}(\text{OH})_3$ coprecipitation method have been used for this purpose. The effects of main components of the baking soda and powder were examined. The methods were successfully applied for the determinations of Cu, Fe, Pb, Cd, Co and Ni in the baking soda and baking powder samples purchased from the local markets in Kayseri City-Turkey (recoveries greater than 95%). The results found the two preconcentration methods generally agree with each other. The levels of the analytes in the samples were below the allowed limit values given by Turkish Authorities.

Key words: Baking powder, Baking soda, Separation, Preconcentration, Atomic Absorption Spectrometry

INTRODUCTION

Important positive and negative roles of trace heavy metal ions in human health are known⁽¹⁻²⁾. Lots of studies have been performed for the determination of trace metal ions in various media including some body tissues and fluids, natural waters etc⁽³⁻⁷⁾. Also the investigation of trace heavy metal contents in food samples including honey, vinegar, lemon juice, sour cream, yogurt, buttermilk, chocolate, cocoa, honey, molasses and other food samples are an important part of analytical chemistry⁽⁸⁻¹⁴⁾.

Baking powder and baking soda are used in recipes that contain acidic ingredients (e.g., fruits and maple syrup). These foods are frequently consumed by human. The determination of heavy trace metals in baking powder and baking soda is important for human health⁽¹⁵⁻¹⁷⁾. The level of trace heavy metal ions the food samples including baking soda and powder samples are generally mg/g. Also foods have complex matrix for the determination of trace metal ions by atomic absorption spectrometry. In atomic absorption spectrometric determinations of heavy metal ions, even by using GFAAS, low metal concentration is another problem. These two problems (low metal concentrations and matrix interferences) are generally prevented by using various separation/enrichment methods such as solvent extraction, cloud point extraction, membrane filtration, evaporation, electroanalytical techniques, ion exchange, flotation, coprecipitation and solid phase extraction⁽¹⁸⁻²³⁾.

Solid phase extraction is an effective method of preconcentration for trace heavy metal ions. In solid phase extraction, many sorbents such as activated carbon, Amberlite XAD resins, naphthalene, Diaion HP-20, fullerenes, activated carbon and other sorbents have been used for separation and preconcentration of trace amounts of heavy metal ions from various matrices. Solid phase extraction (SPE) has several advantages over solvent extraction method, such as simple and fast extractor system, easily adaptable to the preconcentration and to the determination of trace metal ions by flow injection analysis technique. It has a relatively high concentration factor and the ability of treating large volume samples free from contamination. In solid phase extraction (SPE), analyte ions are adsorbed on an adsorbent, and then desorbed with a suitable eluent. Metal determinations were performed in this solution⁽²⁴⁻²⁷⁾.

Coprecipitation is frequently used in the enrichment of trace elements, generally using a milligram quantity of a carrier element to ensure complete trace recoveries and to facilitate the separation of the precipitate from the mother liquor. Also, coprecipitation is widely applied in the analysis of natural waters. Various coprecipitation procedures including use of organic and inorganic coprecipitants have been developed and well documented. Among inorganic coprecipitants, metal hydroxides is most popular due to good trace recovery and sufficient separation factors for alkali and alkaline earth metals. Widely used metal hydroxide coprecipitants include a number of metal hydroxides such as magnesium, samarium, iron, lanthanum, indium, zirconium, hafnium, yttrium and gallium hydroxides, etc⁽²⁸⁻³⁰⁾.

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Table 1. Conditions for Perkin Elmer 3110 flame atomic absorption spectrophotometer

Element	Wavelength (nm)	Slit (nm)	Lamp Current (mA)	Flow Rate of flame gases	
				Air (l/min)	Acetylene (l/min)
Pb	283.3	0.7	15	9.5	2.3
Ni	232.0	0.2	30	9.5	2.2
Fe	248.3	0.2	30	9.5	2.3
Cu	324.8	0.7	15	9.5	2.3
Co	240.7	0.2	30	9.5	2.2
Cd	228.8	0.7	8	9.5	2.2

Table 2. Instrument settings and conditions for Hitachi Z8000 GFAAS

Parameter		Element		
		Pb	Co	Cd
Wavelength	(nm)	283.3	240.7	222.8
Slit width	(nm)	1.3	0.2	1.3
Lamp current	(mA)	7.5	15	7.5
<u>Step</u>	<u>Condition</u>			
Dry	time (sec)	15	15	15
	temp (°C)	120	120	100
Ash	time (sec)	30	30	30
	temp (°C)	400	600	300
Atomize	time (sec)	10	10	10
	temp (°C)	2400	2700	1500

In our literature review, limited study about trace metal contents of baking powder samples have been found⁽³¹⁻³²⁾. The upper-limit levels of trace metal ions in baking soda samples were given by Turkish Authorities in Turkish Standard (TS9063)⁽³³⁾. Only limit values of lead and copper were given in the investigated analyte ions in this standard. The upper-limit values for lead and copper were 10 µg/g and 30 µg/g, respectively.

In the present work, the concentrations of Cu, Fe, Pb, Cd, Co and Ni ions in the baking powder and baking soda samples purchased from local markets in Kayseri-Turkey were determined by flame and/or graphite furnace atomic absorption spectrometry after separation/enrichment procedures with two methods including solid phase extraction and coprecipitation.

EXPERIMENTALS

I. Apparatus

The detection system used was a Hitachi Model Z8000 Zeeman and a Perkin-Elmer Model 3110 atomic absorption spectrometer. The operating parameters for working elements were set as recommended by the manufacturer. They were given in Table 1 for flame atomic absorption spectrometry (FAAS) and in Table 2 for graphite furnace atomic absorption spectrometry (GFAAS). Atomic absorption measurements for our working model were carried out in air/acetylene flame. While the determination of copper, nickel and iron in the baking soda and baking powder sam-

ples were performed by using flame atomic absorption spectrometer, the determination of lead, cobalt and cadmium were performed by using graphite furnace atomic absorption spectrometer.

II. Reagents

Analytical reagent-grade chemicals were employed for the preparation of all solutions. Freshly prepared double distilled water was used in all experiments. Metal ion solutions (1000 mg/l) were prepared by dissolving appropriate amounts of the sulphates or nitrates in double-distilled water and were diluted daily for obtaining reference and working solutions.

Chromosorb 102 resin (Phase Separations, Inc., Norwalk, CT, USA, 750 614) is a porous styrene-divinylbenzene copolymer having a surface area in the range of 300-400 m²/g and 80-100 mesh of particle size. The stopcock of the glass column (100 × 10 mm) was covered with a fritted glass disc. The column was filled with 500 mg of Chromosorb-102 resin. An ammonium pyrolydinedithiocarbamate (APDC) solution (0.05%) was prepared by dissolving the requisite amounts of APDC in water.

For coprecipitation procedure, an Erlenmeyer flask with ground stopper was used. The membrane filter used was made of cellulose nitrate purchased from Advanced MFS, Inc (0.45 µm pore size, 47 mm diameter). A cerium (IV) solution (1 mg/ml) for carrier was prepared freshly by dissolving Ce(SO₄)₄ · 4H₂O (Merck) 0.2886 g in small amounts of nitric acid and diluting to 100 ml with water.

III. Model Studies for Baking Powder and Baking Soda Samples

(I) Chromosorb-102/APDC Solid Phase Extraction Method.

Twenty five ml of the model solution containing 1-10 µg each of metal ions was buffered to desired pH. Three ml of 0.05% APDC solution was added to this solution. It was loaded to the top of the preconditioned column and passed through at a flow rate of 2 ml/min. The sample solution was permitted to flow through the column under gravity. After passing of this solution, the column was rinsed twice with 10 ml of water. Then, the retained metal ions were eluted with 10 ml of acetone. The effluent was evaporated to near dryness on a hot plate and then cooled. It was diluted to 5-10

ml with 1M HNO₃. The analytes in the final solution were determined by FAAS and/or GFAAS.

(II) Ce(OH)₄ Coprecipitation Method.

Sodium hydroxide solution, 1 M, was added to 50 ml of sample solution containing 600 µg cerium(IV), 1-10 µg of each metal ions to adjust the pH to different values. The cerium (IV) hydroxide formed and loaded with the analytes was collected on a cellulose nitrate membrane filter of 0.45 µm size and 47 mm diameter and then dissolved with 0.5 ml of concentrated nitric acid. The solution was diluted to 5 or 10 ml with a high-purity water. The analytes in final solution were determined with flame or graphite furnace AAS according to their concentration level.

The blank solutions for both preconcentration methods did not contain any matrix components of soda samples on the optimization studies. In the investigation of the effects of the matrix components on the recoveries of the analyte ions, each matrix component was added to the model solutions. In the works for accuracy studies and detection limits, the blank solutions contained matrix of the samples.

IV. Procedure for Baking Powder and Baking Soda Samples

A sample of 1.00 g was dissolved in a mixture of 500 µl concentrated HNO₃ and 20 ml of distilled water. The solution was neutralized by using 1.0 M NH₃ and then separation/preconcentration procedures given above were separately applied. The metal contents of the final solution were determined by flame AAS or graphite furnace AAS according to their concentration levels.

RESULTS AND DISCUSSION

Before the application of the Chromosorb-102/APDC solid phase extraction method and the Ce(OH)₄ coprecipitation method for the determination of certain trace metal ions in the baking powder and baking soda samples, some analytical parameters including pH of the aqueous solution, reagent amounts, matrix effects etc. were optimized. The percentages of metals were calculated from the amounts of metal in the starting sample and the amounts of metal in the final solution.

I. Chromosorb-102/APDC Solid Phase Extraction Method

The optimal conditions determined were as follows. The optimum pH for quantitative recovery of the investigated ions was found as 6. The effects of volume of acetate buffer solution were also examined in the range of 2-10 ml for 25 ml of sample solution. The recoveries of analyte ions were quantitative in the buffer volume range of 2-10 ml. Therefore the pH of the sample solution were adjusted by using 2.0 ml of ammonium acetate buffer for all the subsequent experiments.

The optimum amounts of APDC for quantitative recov-

eries of the investigated metal ions was found as 1.5 mg. The amounts of Chromosorb-102 resin in the column was 500 mg that can be used 200 times. For the elution of metal ions with eluents like acetone, 1 M HCl in acetone, 1M HNO₃ in acetone was investigated by using 10 ml of eluent. The quantitative recoveries of Cu, Fe, Pb, Cd, Co and Ni were obtained with acetone. There is no influence of flow rate from 2 ml/min to 8 ml/min on the retention of analytes. These values are comparable to those described in Literature⁽³⁴⁾.

The recoveries of metal ions was not affected by sample volume up to 600 ml. After 600 ml of sample volume, the recoveries of Cu, Fe, Pb, Cd, Co and Ni (750 ml) were not quantitative. In order to determine the detection limits of working elements, model solutions were used. The detection limits found in model solutions containing matrix of the baking soda for analytes based on three times the standard deviations of the blank ($N=20$, $X_L=X_b+3\sigma$, X_L : Limit of Detection, X_b : Blank Value) were in the range of 0.06 µg/g for Cd and 0.26 µg/g for Fe.

II. Ce(OH)₄ Coprecipitation Method

The optimal conditions for quantitative recoveries of Cu, Fe, Pb, Cd, Co and Ni with Ce(OH)₄ coprecipitation method were previously examined in detail⁽³⁵⁾. The optimum conditions determined for separation/preconcentration procedure were as follows. Metal ions can quantitatively (>95%) be recovered at the pH range of 10-11 so that pH 10.5 as optimum pH was used in further experiments. The recoveries of the metal ions were quantitative with 600-800 µg cerium(IV) as coprecipitant. Quantitative recoveries (>95%) were obtained for the sample volume in the range of 50-750 ml for all metal ions. The highest preconcentration factor was found to be 375.

The relative standard deviations were found in the range of 6-8%. The detection limits of the separation-preconcentration procedure in the blank solutions containing matrix of the soda sample varied from 0.008 µg/g for Cd to 1.2 µg/g for Fe ($N=21$) (based on 3 times the standard deviation of the blank) for 50 ml of samples. The detection limit can be decreased by one order of magnitude by increasing the sample volume.

III. Matrix Effects

Analytical preconcentration/separation procedures for trace elements in the high salt content samples can be strongly affected by the matrix constituents of the sample. This is known as matrix effect. Before the application of the two preconcentration method for the determination of analyte ions in baking soda samples, the influences of some alkaline and alkaline earth ions on the recoveries of the analyte ions were also investigated. The results are given in Table 3 for Chromosorb-102/APDC Solid Phase Extraction Method and for Ce(OH)₄ Coprecipitation Method. The tolerance limit is defined as the ion concentration causing a rela-

tive error smaller than $\pm 5\%$ related to the preconcentration and determination of analytes. Metal ions were quantitatively recovered at large amounts for alkaline and earth alkaline ions and some anions.

The recoveries of sodium, potassium, magnesium and calcium ions with both preconcentration methods were determined using flame photometer and/or FAAS and found to be less than 1%. The matrix ion contents in the eluent solutions were found to be significantly lower and suitable for atomic absorption spectrometric determinations. The small amounts retained of the matrix ions were removed by washing with 10-15 ml of the accompanying buffer solution. The concentrations of investigated matrix ions in the baking soda and powder samples were within tolerable limits.

IV. Accuracy Studies

The accuracy of results was verified by analyzing the concentration after addition of known amounts of analytes into (D) baking powder sample. For this purpose, 1.0 g of baking powder samples was in mixture of 500 μ l concentrate HNO_3 and 20 ml of distilled water, the solution was

Table 3. Effects of the matrix ions on the recoveries of the examined metal ions for both techniques (N=3)

Ion	Added As	Tolerance Limits, mg/l
Na^+	NaCl	50000
K^+	KCl	2500
Mg^{2+}	MgCl_2	5000
Ca^{2+}	CaCl_2	2500
Cl^-	NaCl	75000
SO_4^{2-}	$(\text{NH}_4)_2\text{SO}_4$	2500
HCO_3^-	NaHCO_3	7500
PO_4^{3-}	Na_3PO_4	3000

neutralized with 1.0 M NH_3 and 2.5-20 μ g of each metal was added. As can be seen in Table 4, good agreement was obtained between the added and analyte recovered content using the experimental procedure with some exceptions. Some results were also obtained with the amounts of metal ions below 2.5 μ g.

Although the recoveries of iron for method A (Chromosorb-102/APDC Solid Phase Extraction Method) and copper for method B ($\text{Ce}(\text{OH})_4$ Coprecipitation Method) were quantitative when the studies were performed in each matrix ions separately, the recoveries of iron for method A and copper for method B were not quantitative in the real samples due to total effect of the matrix. Because of this point, the determinations of iron and copper in the baking soda and powder samples could not be performed by Method A and Method B, respectively.

V. Metal Contents of the Baking Powder and Baking Soda Samples

Copper, iron, lead, cadmium, cobalt and nickel contents of the baking powder and baking soda samples were determined with FAAS and/or GFAAS after separation and preconcentration. The results given in Table 5 and Table 6 have been calculated based on the assumption of 100% recovery of analyte ions. The relative standard deviations (RSD's) of the determinations for both separation/preconcentration techniques were generally found to be below 10%.

CONCLUSION

Chromosorb-102/APDC solid phase extraction method and $\text{Ce}(\text{OH})_4$ coprecipitation method were satisfactorily applied for the determination of some metal ions in baking

Table 4. Recovery studies of trace metal ions in (D) baking powder sample

Analytes	Added, μ g	Method A		Method B	
		Found, μ g	Recovery, %	Found, μ g	Recovery, %
Cu	0	4.2	-	N.D.	-
	5.0	9.0	96	1.9	38
	20.0	25.1	104	11.0	55
Fe	0	N.D.	-	6.4	-
	5.0	-	-	11.4	100
	20.0	-	-	25.8	97
Ni	0	3.2	-	3.3	-
	5.0	8.2	100	8.3	100
	20.0	23.2	100	22.3	95
Co	0	N.D.	-	N.D.	-
	5.0	5.0	100	5.0	100
	10.0	10.0	100	10.0	100
Pb	0	0.2	-	0.2	-
	10.0	10.2	100	10.2	100
	20.0	19.2	95	20.2	100
Cd	0	N.D.	-	N.D.	-
	2.5	2.5	100	2.5	100
	10.0	9.6	96	9.5	95

N.D.: Not Detected

Method A: Chromosorb-102/APDC Solid Phase Extraction Method

Method B: $\text{Ce}(\text{OH})_4$ Coprecipitation Method.

Table 5. The concentration of some traces metal ions in baking soda samples after preconcentration

Sample→	Concentration of Metals, µg/g*					
	Y		Z		V	
	Method A	Method B	Method A	Method B	Method A	Method B
Analytes						
Co	BDL	BDL	BDL	BDL	BDL	BDL
Pb	0.22 ± 0.02	0.21 ± 0.01	0.10 ± 0.02	0.15 ± 0.03	0.09 ± 0.01	0.13 ± 0.05
Ni	3.17 ± 0.29	2.22 ± 0.43	1.66 ± 0.01	1.83 ± 0.29	0.67 ± 0.01	0.67 ± 0.01
Fe	N.D.	1.44 ± 0.05	N.D.	2.88 ± 0.05	N.D.	1.93 ± 0.05
Cu	1.32 ± 0.50	N.D.	BDL	N.D.	BDL	N.D.
Cd	0.023 ± 0.003	0.020 ± 0.005	0.024 ± 0.003	0.020 ± 0.001	0.023 ± 0.003	0.010 ± 0.001

BDL: Below the detection limit, N.D.: Not Determined,

* P=0.95, $x \pm ts/\sqrt{N}$, N=5

Table 6. The concentration of some metal ions in baking powder samples after preconcentration

Sample→	Concentration of Metals, µg/g*					
	D		X		E	
	Method A	Method B	Method A	Method B	Method A	Method B
Analytes						
Co	BDL	BDL	BDL	BDL	BDL	BDL
Pb	0.18 ± 0.04	0.18 ± 0.01	0.20 ± 0.02	0.19 ± 0.01	0.18 ± 0.04	0.18 ± 0.01
Ni	3.16 ± 0.29	3.30 ± 0.37	2.42 ± 0.26	3.00 ± 0.29	2.02 ± 0.08	1.83 ± 0.29
Fe	N.D.	6.36 ± 0.67	N.D.	5.88 ± 0.69	N.D.	3.83 ± 1.22
Cu	4.15 ± 0.86	N.D.	3.40 ± 0.89	N.D.	3.76 ± 0.85	N.D.
Cd	0.021 ± 0.003	0.020 ± 0.003	0.053 ± 0.004	0.050 ± 0.005	0.024 ± 0.006	0.020 ± 0.001

BDL: Below the detection limit, N.D.: Not Determined,

* P=0.95, $x \pm ts/\sqrt{N}$, N=5

powder and baking soda samples by atomic absorption spectrometry. The results found the two methods generally agree each other with some exceptions. The levels of the investigated ions were below the limit values given by Turkish Authorities⁽³³⁾.

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利用預濃縮配合原子吸收光譜儀測定發酵粉及小蘇打中銅、鐵、鉛、鎘、鈷和鎳之成分含量

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摘 要

發酵粉與小蘇打中銅、鐵、鉛、鎘、鈷和鎳經分離及預濃縮後，續利用原子吸收光譜法進行測定。本研究採用兩種不同的分離／預濃縮方法，分別為APDC-Chromosorb 102固相萃取法及Ce(OH)₄共沉澱法。研究中特別針對小蘇打和發酵粉中主要成分對待測元素在分析時可能造成的影響進行探討。所建立的分析方法成功地應用於測定購自土耳其區域市場（Kayseri City-Turkey）小蘇打和發酵粉中的銅、鐵、鉛、鎘、鈷和鎳之成分含量（回收率高於95%）。實驗的結果顯示，這兩種預濃縮方法對同一樣品的分析結果大致頗為相符。這些金屬在小蘇打和發酵粉中的含量均低於土耳其官方的最低限量值。

關鍵詞：發酵粉，小蘇打，分離，預濃縮，原子吸收光譜