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***Research paper***

# Hydride generation atomic fluorescence spectrometry (HG-AFS) determination of total arsenic in lipstick

**M. E. Ghanjaoui[1,2]*, I. Adraoui[3], A. Gammoun[1], L. Cervera[1], M. El Rhazi[2], M. de la Guardia[1]**

[1]*Department of Analytical Chemistry, University of Valencia, 46100 Burjassot (Valencia) Spain*
[2]*Laboratory of Electrochemistry and Chemical Physics, Faculty of Sciences and Technologies (Mohammedia) Morocco*
[3]*Laboratory of Biotechnology, Materials and Environment, University Ibn Zohr, BP 8106, Cite Dakhla, Agadir, Morocco*
**Corresponding author E-mail:ghanjaoui2000@hotmail.com*

**Abstract**

This study aimed to determine the total arsenic concentration in lipsticks and evaluate the potential health risks due to daily ingestion of this toxic element from this product. The samples were analyzed using atomic fluorescence spectrometry technique after mineralization and hydride generation (HG-AFS). The calibration curve is linear over a concentration range from 0.125 to 2 µg $L^{-1}$ with a regression coefficient of the order of 0.9899. The limit of detection is reaches 3 ng $g^{-1}$. The standard deviation is of the order of 4%.The accuracy of this method was evaluated by calculating the recoveries of additions of arsenic to three lipstick samples. These results indicate recovery rates between 91% and 98%. Finely, the results found demonstrate that the method used in this work is suitable for arsenic detection in lipsticks samples.

***Keywords***: *Lipstick;Atomic Fluorescence Spectrometry; Mineralization; Hydride Generation;Risk Assessment.*

## 1. Introduction

Trace elements are present in the environment and can enter the raw materials in trace amounts. Generally cosmetics like skin-care creams, lotions, powders, perfumes, lipsticks and many other types of products are made from mixtures of chemical compounds some are derived from natural sources or synthetic. Dermal exposure would be the main route of exposure for cosmetics since most cosmetics are applied to the skin causes low-grade of toxicity. Dermal absorption of trace elements is relatively low; the absorption of individual elements is influenced by a number of factors including the physicochemical properties of mixtures [1].

Some of the ingredients in cosmetics are toxic to our health. Most have been narrowly tested and allergic responses are very common with the use of cosmetic. Heavy metals like lead, arsenic and cadmium are relatively added to the cosmetics for colour, odour and to increase surface adhesion or for biological response [2].

Lipstick is one of the decorative cosmetic products containing 65% castor oil, 15% beeswax, 10% carnuba wax, 5% lanolin, dyes, pigments and fragrances [3], which are applied to the color and texture of the lips.

Currently there are no international standards for trace element levels in cosmetics. The German Federal Government has conducted studies to identify the levels of trace elements in cosmetic products. According to its studies, the trace element levels in cosmetics exceeding the following values are considered technically unusable (Lead: 20 ppm, Arsenic: 5 ppm, Cadmium: 5 ppm, Antimony: 10 ppm) [4].

Manufacturers are theoretically aware of these issues and their products are normally subject to various controls, particularly from an allergenic point of view. A study carried out as part of the "Campaign for safe cosmetics" [5] by an American consumer protection association as part of the "Campaign for safe cosmetics" [5] showed that in 2007, lead was still found in 61% of the 33 brands of lipsticks tested and sold in the United States; lead levels ranged from 0.03 to 0.65 ppm, doses that remain relatively low, but when applied to mucous membranes, they are likely to be in contact with food and can be toxic. In one third of the cases, these lipsticks contained more lead than the amount allowed by the US Food and Drug Administration in candy (0.1 ppm) [5]. However, even in a country like the USA, there is a lack of standards for trace elements in this type of cosmetics. The study conducted by the American association therefore showed that the most expensive lipsticks were often those that contained the most lead [6].

In 2007, the U.S. Food and Drug Administration suggested that the comparison between the lead exposure pathway in the two cases (lipstick and candy) is not a fair comparison because candy is intended to be ingested over a regular period of time while lipstick is a product intended for topical use, and is ingested in much smaller amounts than candy [7].

Atomic fluorescence spectrometry (AFS) is an analytical technique that has been widely used for the determination of arsenic and other volatile elements [8] –[11]. The coupling of hydride generation (HG) and AFS allows determinations with low quantification limit, its instrumentation being cheaper than other conventional analytical techniques [12].

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In this work, we will focus more specifically on the development of gentle digestion method for the detection of trace elements in lipstick. The technique that will be used is atomic fluorescence spectrometry after mineralization and hydride generation (HG-AFS).

# 2. Materials and methods

## 2.1.Procedure for arsenic analysis by HG-AFS

The PS Analytical Millennium Excalibur atomic fluorescence spectrometer (Kent, UK) was used for the detection of arsenic. The system is equipped with an arsenic hollow cathode lamp (discharge lamps) (Official Distributor PHOTRON, Victoria, Australia).
A sand bath PL 5125 Scharlau Raypa (Barcelona, Spain) and a Eurotherm muffle furnace K1253 equipped with a temperature control program 902 Heraeus (Madrid, Spain) were used to perform the mineralization of our samples.

## 2.2. Reagents and solutions

All solutions were prepared using high-purity water. The stock standard solutions (1.000 mg $mL^{-1}$) of As (III) were obtained by dissolving appropriate amounts of 1.320 g of arsenic oxide $As_2O_3$ obtained from Riedel-de Haen (Hanover, Germany) in 25 ml of 20% (w/v) potassium hydroxide KOH, neutralization and dilution of the resulting solution was performed with 20% (v/v) $H_2SO_4$ sulfuric acid up to 1l.
For HG-AFS analysis, a 50% (w/v) potassium iodide reducing solution prepared from potassium iodide KI (Merck) and a 10% (w/v) solution of ascorbic acid obtained from charlau were used to reduce As(V) to As(III).
A 0.7% w/v solution of sodium tetrahydroborate $NaBH_4$ (Fluka) was prepared in 0.1 mol $L^{-1}$ of sodium hydroxide NaOH to generate the arsenic hydrides easily decomposable when analyzed by AFS atomic fluorescence spectrometry. This solution must be prepared and filtered daily before use.
The standard solutions have been prepared from 0.125 to 2 g $L^{-1}$ in 30 mL plastic vials with the addition of 600 L of potassium iodide KI reducing solution (50%) to each vial.

## 2.3.Preparation and the determination of arsenic by HG AFS

The high-temperature calcination of 0.5 ± 0.005 g of the lipstick samples was carried out with the addition of 2.5 mL of a stabilizing solution of 20% (w/v) $Mg(NO_3)_2$-$6H_2O$ and 2% (w/v) MgO which inhibits the formation of metal hydrides during mineralization and 5 mL of 50% (v/v) nitric acid. The mixture is first evaporated in a sand bath and then mineralized in a muffle furnace at 450°C, with a gradual increase in temperature [1]. The resulting white ash was treated with 1 mL of ultrapure water and 9 mL of 10% (v/v) hydrochloric acid. A volume of 3 mL of each of the resulting solutions was transferred to 50 mL plastic vials. 8.75 mL of 37% (v/v) concentrated hydrochloric acid HCl and 600 l of a 50% (w/v) reducing solution of KI and 10% ascorbic acid have been added. The solutions were adjusted with ultrapure water to 30 ml. Total arsenic was analysed by atomic fluorescence spectrometry according to the experimental conditions shown in Table 1.

**Table 1:** Operating Conditions of the HG-AFS Instrument for the Determination of Arsenic

| Parameter | Setting |
|---|---|
| Arsenic Wave length (nm) | 197,3 |
| $NaBH_4$ concentration (%) (w/v) | 0,7 |
| Flow rate of Argon (mL $min^{-1}$) | 330 |
| Flow rate of Air (L $min^{-1}$) | 2,5 |
| Sample flow rate ( $mLmin^{-1}$) | 9 |
| $NaBH_4$ flow rate ($mLmin^{-1}$) | 4,5 |

# 3. Results and discussions

## 3.1. Arsenic calibration curve

The arsenic calibration curve was performed using atomic fluorescence spectrometry after mineralization and hydride generation using the conditions shown in Table 2.
The calibration curve is linear over a concentration range from 0.125 to 2 µg $L^{-1}$ with a regression coefficient of the order of 0.9899. The limit of detection is calculated over five blank repeats in the lipstick samples analyzed, it reaches 3 ng $g^{-1}$. The standard deviation calculated on five different samples is of the order of 4% (Table 2).

**Table 2**: Regression Line Characteristics

| Parameter | Valeur |
|---|---|
| As concentration range (µg.$L^{-1}$) | 0.125 -2 |
| Regression equation | Y = 11 + 123*C |
| Regression coefficient ($r^2$) | 0,9899 |
| Standard deviation (RSD) n=5 | 4% |
| The limit of detection (ng $g^{-1}$) | 3 |

## 3.2. Analysis of total arsenic in lipstick by HG-AFS

The high-temperature calcinations procedure described in was combined with atomic fluorescence spectrometry and hydride generation to evaluate the arsenic content of lipstick samples. In **Table 3** we have presented the results obtained in four samples of lipstick purchased from the local valence market, the concentration range obtained for arsenic is between 30 and 250 ng $g^{-1}$. The concentration values obtained in all samples are well below the limit recommended by the German federal government (5ppm).

The accuracy of this method was evaluated by calculating the recoveries of tree additions (100, 200 and 300 ng $g^{-1}$) to the four lipstick samples. These results indicate that the recovery rates between 91% and 98%.

**Table 3:**Results of the Analysis of Arsenic Levels in Ng G-1 in Four Lipstick Samples Using HG-AFS.

| N° | Color | Added | As Found ng g-1 | Recovery (%) |
|---|---|---|---|---|
| 1 | Red | - | 250 ± 11 | - |
| 2 | Pink | - | 30 ± 2 | - |
| 3 | Dark red | - | 90 ± 3 | - |
| 4 | Shemmering beige | - | 61 ± 2 | - |
| 1 | Red | 100 ng g-1 | 342 ± 13 | 98 |
| 2 | Pink | 100 ng g-1 | 121 ± 5 | 93 |
| 3 | Dark red | 100 ng g-1 | 179 ± 6 | 94 |
| 4 | Shemmering beige | 100 ng g-1 | 154 ± 7 | 96 |
| 1 | Red | 200 ng g-1 | 438 ± 21 | 97 |
| 2 | Pink | 200 ng g-1 | 215 ± 9 | 93 |
| 3 | Dark red | 200 ng g-1 | 265 ± 14 | 91 |
| 4 | Shemmering beige | 200 ng g-1 | 255 ± 12 | 98 |
| 1 | Red | 300 ng g-1 | 520 ± 28 | 95 |
| 2 | Pink | 300 ng g-1 | 308 ± 16 | 93 |
| 3 | Dark red | 300 ng g-1 | 358 ± 15 | 92 |
| 4 | Shemmering beige | 300 ng g-1 | 343 ± 11 | 95 |

# 4. Conclusion

The proposed method has suitable for the determination of arsenic in lipstick samples. The arsenic contents found in the all the lipsticks samples analyzed are lower than the maximum limited permissible contamination. These results show that the samples analyzed do not present a real danger.

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