



# Determination of Pb in lipsticks by flow injection chemical vapor generation isotope dilution inductively coupled plasma mass spectrometry



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## ABSTRACT

A flow injection vapor generation method is described for the analysis of lipstick for Pb by isotope dilution inductively coupled plasma mass spectrometry (ICP-MS). A sample homogenate was prepared as emulsion containing 1% m/v lipstick, 0.5% m/v  $K_3Fe(CN)_6$ , 0.5% v/v  $HNO_3$  and 0.5% m/v Triton X-100 and was injected into a VG-ICP-MS system followed by sodium borohydride for the vapor generation of Pb. Since the efficiency of hydride generation of Pb in emulsion and that of aqueous solution was quite different, isotope dilution and standard addition methods were used for the determination of Pb in selected lipstick samples. The influences of vapor generation conditions on the Pb ion signal were reported. This method has been validated by the determination of Pb in GBW09305 Cosmetic (cream) reference material. The method was also applied for the determination of Pb in lipsticks of different colors obtained locally. The analytical results obtained by isotope dilution and standard addition methods were in good agreement and also with those of digested samples analyzed by pneumatic nebulization ICP-MS. Under the optimum operating conditions, the detection limit obtained was  $0.001 \text{ ng mL}^{-1}$  in prepared emulsified solutions, corresponding to  $0.11 \text{ ng g}^{-1}$  of Pb in the original lipstick samples.

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## 1. Introduction

Most analyses by inductively coupled plasma mass spectrometry (ICP-MS) are carried out on solutions using a conventional pneumatic nebulizer. However the types of analytical tasks that are solved by ICP-MS can be extended using a number of other sample introduction techniques. Vapor generation (VG) is one of the sample introduction techniques that are currently employed in ICP-MS. Several reports showed that the sensitivity and detection limits of vapor-forming elements are greatly improved when they are introduced into the plasma as vapors [1–5]. Additionally, it was also shown that appropriate vapor generation conditions can be used to remove or separate the analyte from problematic matrix species which would otherwise cause spectral and non-spectral interferences [5,6]. Vapor introduction in flow injection mode is a simple and rapid technique that has been coupled to ICP-MS previously [7,8].

Cosmetics have become a part of modern lifestyle for cleansing, beautifying and promoting appearance. However, stringent quality control is needed for cosmetics as they may be contaminated with toxic

elements during production that cause detrimental effects on human health. Moreover, some of the metals are the subject of food and drug legislation [9,10]. For instance, according to the regulations of the Taiwanese government, the maximum allowed concentration of Pb in cosmetics is  $10 \mu\text{g g}^{-1}$  [11]. Thus, the determination of trace elements in cosmetics, in order to measure the levels of toxic elements, is important. Toxic elements in cosmetics represent an important source of pollution, even at low concentrations, as cosmetics are used daily with longer contact times. ICP-MS has been applied for the determination of trace elements in various cosmetic samples [12–18]. Most of the techniques require sample pretreatment steps [19–22]. These pretreatment methods are often tedious and time consuming with the consequent risk of sample contamination and analyte loss. A sensitive, rapid and simpler procedure is desirable for routine monitoring of lead in lipstick samples.

In our previous study, VG-ICP-MS has been applied for the determination of As, Cd, and Hg in emulsified vegetable oils [23], Hg and Pb in fuels [24], and for the determination of As, Cd and Hg in cereals [25]. The aim of the present work is to develop a rapid and accurate flow injection isotope dilution ICP-MS method with vapor generation sample introduction device for the determination of Pb in lipsticks. Isotope dilution (ID) techniques have been used in several previous ICP-MS applications to ensure accurate determinations [23,26]. In this study, a

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continuous-flow vapor generation system was coupled with ICP-MS for the determination of Pb using FI analysis [27]. The sample was introduced by FI to minimize the deposition of solids on the sampling orifice. The lipstick samples were diluted in 0.5% m/v of Triton X-100 to form the emulsified solutions and treated with NaBH<sub>4</sub> for the determination of Pb by FI-VG-ICP-MS. The influences of vapor generation conditions on the Pb ion signal are reported. This method has been applied for the determination of Pb in various lipsticks obtained locally.

## 2. Experimental

### 2.1. Apparatus and conditions

An ELAN 6100 DRC II ICP-MS (PE-SCIEX, Concord, ON, Canada) was used for this study. Sample solutions were introduced by a pneumatic nebulizer with a Scott-type spray chamber. In this study, the ICP operating conditions and the operating conditions for the vapor generation were optimized by the flow injection (FI) method. An emulsified lipstick was selected as the model to optimize the operating conditions of the ICP and vapor generation system. The ICP-MS operating conditions used in this work are summarized in Table 1.

In this study, a continuous-flow vapor generation system was coupled with ICP-MS for the determination of Pb using FI analysis [27]. It was assembled from a six-port injection valve (Rheodyne type 50) with a 100 µL sample loop. Vapor generated from the VG system was delivered to the ICP-MS system for Pb determination via Tygon tubing. A schematic diagram of the flow injection vapor generator system is shown in Fig. S1 (Supplementary data).

### 2.2. Reagents

All reagents were of analytical-reagent grade, and deionized water was used throughout. NaBH<sub>4</sub>, NaOH, Suprapure HNO<sub>3</sub>, HCl and HF, citric acid and K<sub>3</sub>Fe(CN)<sub>6</sub> were obtained from Merck (Darmstadt, Germany). NaBH<sub>4</sub> solution containing 0.2% m/v NaOH was freshly prepared just prior to analysis. Tartaric acid and H<sub>2</sub>O<sub>2</sub> were obtained from Riedel-deHaën (Seelze, Germany). Triton X-100 and antifoam Y-30 were obtained from Sigma Chemicals (St. Louis, MO, USA). Oxalic acid was from Fluka (Switzerland). Lead element standard solution was from AccuStandard (New Haven, CT, USA). Enriched isotopes of <sup>206</sup>Pb (99.9%) was purchased from Merck.

### 2.3. Samples and sample preparation

The applicability of the method to real samples was demonstrated by analyzing Pb in GBW09305 As and Pb in Cosmetic (cream) reference material (National Research Centre for Certified Reference Materials, China) and seven lipstick samples obtained locally. Since the sensitivity of Pb in various matrices was different, the lipstick samples were analyzed by isotope dilution and standard addition methods. The emulsion was prepared using the following procedure. A 0.1-g portion of the lipstick was transferred into a 10 mL flask. 0.5 mL 10% m/v Triton X-100, 0.05 mL HNO<sub>3</sub> and 5 mL H<sub>2</sub>O were added. The solution was heated at 85 °C for 30 min to melt the lipstick. After cooling, suitable amount of enriched isotope or Pb element standard solution (0.1–2 ng mL<sup>-1</sup>), K<sub>3</sub>Fe(CN)<sub>6</sub> and a drop of antifoam were added, the emulsion was then diluted to the mark with pure water. A blank solution was also prepared, as outlined above, to correct any Pb in the reagents used for sample preparation. These solutions were analyzed for Pb using ICP-MS by injecting 100 µL of the emulsified solution into the VG system. The concentration of Pb in the sample was calculated by the equation described in a previous paper [26] and/or from the standard addition calibration curve. Owing to the mass bias effect, the sensitivity of the instrument at different m/z might be different. Hence, the isotopic composition of Pb in both natural element and enriched isotope was obtained by determining the intensities of all isotopes by ICP-MS with solution nebulization.

Since there is no reference value for the real-world samples, in order to check the accuracy of the VG-ICP-MS method, we need to compare the Pb concentrations in the samples using the present procedure and complete dissolution method. A MARS microwave digester (CEM, Matthews, NC, USA) was used to digest the lipstick samples. The digestion of the samples has been carried out in a closed XP1500 PTFE vessel using HNO<sub>3</sub>. To 0.5 g each of samples taken in a vessel, 4 mL HNO<sub>3</sub> and 2 mL HF were added and closed. The microwave system was programmed to maintain the temperature at 200 °C for 30 min with a ramp time of 30 min. The digest was diluted with pure water and analyzed by pneumatic nebulization ICP-MS with 1 ng mL<sup>-1</sup> of rhodium as the internal standard.

## 3. Results and discussion

### 3.1. Selection of vapor generation conditions

Lead shows higher vapor generation efficiency if it is oxidized to Pb(IV) before the VG reaction. Different combinations of oxidant and

**Table 1**  
ICP-MS equipment and operating conditions.

ICP-MS instrument	Perkin-Elmer Sciex ELAN 6100 DRC II
Plasma conditions	
Rf power/w	1200
Plasma gas flow/L min <sup>-1</sup>	15.0
Intermediate gas flow/L min <sup>-1</sup>	1.10
Carrier gas flow/L min <sup>-1</sup>	1.10
Mass spectrometer settings	
Resolution	0.7 amu at 10% peak maximum
Isotopes monitored	<sup>206</sup> Pb, <sup>207</sup> Pb, <sup>208</sup> Pb
Dwell time/ms	50
Sweeps per reading	16
Reading per replicate	160
Peak signal	Integrated
Autolens	On
Vapor generation system	
Sample volume/µL	100
Carrier solution	0.5% m/v K <sub>3</sub> Fe(CN) <sub>6</sub> in 0.5% v/v HNO <sub>3</sub>
NaBH <sub>4</sub> solution	1.25% m/v NaBH <sub>4</sub> in 0.2% m/v NaOH
Solution flow rate/mL min <sup>-1</sup>	0.85
Volume of mixing coil	80 µL (1.0 mm i.d. × 10 cm length)
Emulsion sample	1% m/v lipstick, 0.5% m/v Triton X-100 and 0.5% m/v K <sub>3</sub> Fe(CN) <sub>6</sub> in 0.5% v/v HNO <sub>3</sub>

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