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Application News

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Cosmetics / AA-7000/MVU-1A

Analysis of Mercury in Cosmetics by Cold Vapour Atomic Absorption Spectroscopy (CVAAS)

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❑ Introduction

There have been concerns regarding the presence of heavy metals such as mercury (Hg) in cosmetics. Hg could be absorbed through the skin or ingested and accumulate in the body over time, causing adverse long-term health consequences, such as skin irritation and nervous system damage [1]. Hence, it is important to regulate and screen for the presence of Hg in cosmetics. According to USFDA 21 CFR 700.13, Hg is not allowed in any cosmetics except in trace amount of less than 1 ppm [2]. The ASEAN Harmonized Cosmetic Regulatory Scheme (AHCRS) has also set 1 ppm as the maximum limit for Hg [3] and provides method for the determination of Hg in cosmetic using cold vapour atomic absorption spectrophotometer (CVAAS) [4]. This application news demonstrates the analysis of Hg in cosmetics using Shimadzu AA-7000 atomic absorption spectrophotometer (AAS) with MVU-1A Hg Vaporizer Unit.

❑ Experimental

The samples used in this analysis were Lipstick Certified Reference Material (CRM) (catalogue no. HRM-2012A) from Health Sciences Authority, Singapore and a commercially available cosmetic cream. Each sample, approximately 0.15 to 0.20 g was transferred to PTFE digestion vessels. This was followed by addition of 8 mL of 65 % nitric acid (HNO_3) from Merck, Germany and 2 mL of 30 % hydrogen peroxide (H_2O_2) from Kanto Chemical, Japan. After incubation for 15 minutes, the PTFE digestion vessels were placed in the Ethos EASY (Milestone, Italy) microwave digestion system and digestion was carried out using the heating program in Table 1. Two replicate preparations were carried out for the Lipstick CRM.

Table 1: Microwave Digestion Program

Step	Temperature	Power	Duration
1	200 ℃	1800 W	15 minutes (ramp)
2	200 ℃	1800 W	15 minutes (hold)

After microwave digestion, the content in each PTFE digestion vessel was transferred to a 25 mL volumetric flask and topped up with Type E-1 ultra-pure water (Milli-Q® Millipore system, Germany).

The 1000 ppm Hg standard and magnesium perchlorate [$Mg(ClO_4)$] were from Sigma Aldrich, USA. The 37% hydrochloric acid (HCl), was purchased from Merck, Germany whereas the 98% sulfuric acid (H_2SO_4) was from JT Baker, USA. Both tin (II) chloride dihydrate ($SnCl_2.2H_2O$) and potassium permanganate ($KMnO_4$) were purchased from BDH Chemicals, England. The MVU-1A reagents were prepared as in MVU-1A Instruction Manual [5].

The CVAAS measurement of Hg was carried out using Shimadzu AA-7000 AAS with MVU-1A Hg Vaporizer Unit (Figure 1) and Hg hollow cathode lamp from Hamamatsu, Japan, using the analytical conditions as shown in Table 2.

Figure 1: Shimadzu AA-7000 (left) and MVU-1A (right)

Table 2: Instrument and Analytical Conditions

Instrument	:	AA-7000 and MVU-1A
Wavelength	:	253.7 nm
Slit Width	:	0.7 nm
Background Correction	:	Deuterium lamp method
Lamp Current	:	4 mA
Repetition Times	:	3 times
MVU-1A measurement mode	:	Circulation mode

In MVU-1A, $SnCl_2$ is used to reduce Hg to atomic vapour form which is then transferred by in-built generated carrier air into a drying tube filled with $Mg(ClO_4)$ to remove moisture. The moisture-free Hg atomic vapour then flows into a glass flow cell for measurement. After measurement, the Hg is exhausted safely into a $KMnO_4$-H_2SO_4 Hg absorbing solution.

❑ Results and Discussion

Figure 2 shows the Hg calibration curve with a good correlation coefficient (R^2) of more than 0.9990.

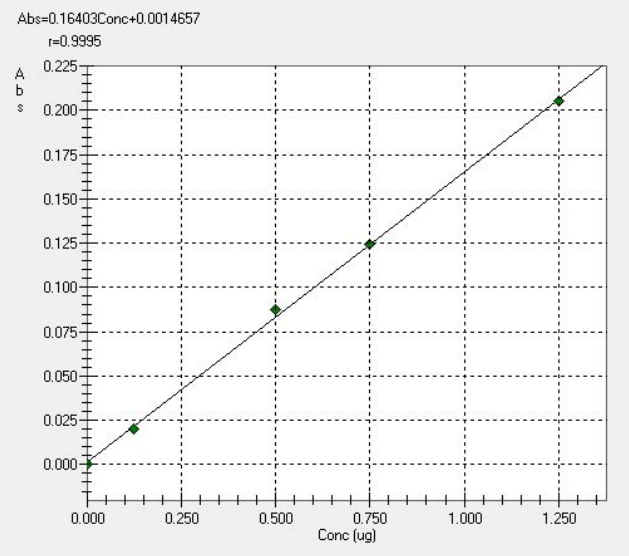

Figure 2: Hg Calibration Curve

The Hg results in Lipstick CRM are shown in Table 3. For the 2 replicate preparations, the results were accurate as the deviation was within 10 % of the certified value. The % Relative Standard Deviation (RSD) for both replicates was less than 1 %, which showed good precision of the analysis.

Table 3: Hg Results in Lipstick CRM

Lipstick CRM	Measured Content (mg/kg)	Certified Content (mg/kg)	% Accuracy
1st Preparation	2.16	1.99	108.5
2nd Preparation	2.17	1.99	109.0

The instrument limit of quantitation (LOQ) [6] and method detection limit (MDL) were calculated as followed.

[a]LOQ = 10 σ / slope of calibration curve

= 10 × 0.0005 / 0.16403 = 0.03 µg

[b]MDL = LOQ × conversion factor / weight factor

= 0.03 × 1.25 / 0.2 g = 0.19 µg/g or 0.19 ppm

where
a: σ is the standard deviation of 10 replicate blank measurements
b: conversion factor from 20 mL out of total digestion volume of 25 mL was used for measurement

As the MDL was 0.19 ppm, the CVAAS method could meet the 1 ppm maximum limit allowed by USFDA and ASEAN AHCRS regulations.

The Hg content in cosmetic cream was less than MDL of 0.19 ppm, which was less than the 1 ppm maximum limit allowed by USFDA and ASEAN AHCRS regulations. A spike recovery test was also carried out where the cosmetic cream was spiked with 0.8 µg of Hg prior to digestion. The spike recovery result of 93.7 % (Table 4) was within the spike recovery range of 88 – 111 % required by ASEAN Cosmetic Methods [4].

Table 4: Hg Results in Cosmetic Cream

Sample	Measured Conc (µg)	% Spike Recovery
Cosmetic cream	< LOQ	-
Cosmetic cream spiked with 0.8 µg Hg	0.75	93.7

❑ Conclusion

The quantitative CVAAS analysis of Hg in cosmetics could be carried out using Shimadzu AA-7000 with MVU-1A. The analysis of a CRM and spike recovery test results showed the method to be accurate. The MDL also proved the method to be precise and could meet USFDA and ASEAN AHCRS regulations.

❑ Reference

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