

Application News

AA-7000/GFA-7000/Cosmetic

Analysis of Heavy Metals in Cosmetics by Graphite Furnace Atomic Absorption Spectrometry (GFAAS)

□ Introduction

Heavy metals such as arsenic (As) and lead (Pb) are naturally occurring elements in the environment, including raw materials used in the manufacturing of cosmetics. This has raised concerns among consumers as heavy metals are detrimental to human health [1]. To address such issues, regulatory bodies from different countries have established guidelines to limit heavy metals impurities in cosmetics. According to the ASEAN Harmonized Cosmetic Regulatory Scheme (AHCRS), the maximum limit of As and Pb in cosmetics should be less than 5 ppm and 20 ppm respectively [2]. To determine the amount of heavy metals in ASEAN Cosmetic Methods [4], graphite furnace atomic absorption spectrometer (GFAAS) is used. Here, we introduce the analysis of As and Pb in cosmetics using Shimadzu AA-7000 atomic absorption spectrophotometer (AAS) with GFA-7000 graphite furnace atomizer.

□ 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. The samples were prepared with microwave digestion according to the procedure in Application News AD-0219 [3]. Two replicate preparations were carried out for the Lipstick CRM. Prior to analysis, the sample solutions were further diluted 20 times with Type E-1 ultra-pure water (Milli-Q® Millipore system, Germany).

The 1000 ppm As and Pb standards were purchased from Sigma Aldrich, USA. The hollow cathode lamps from Hamamatsu, Japan were used. The ammonium phosphate matrix modifier was from Riedel-de Haën, Germany whereas magnesium nitrate matrix modifier was purchased from Merck, Germany.

Analysis was performed using Shimadzu AA-7000 AAS with GFA-7000 graphite furnace atomizer and the ASC-7000 autosampler, using the analytical conditions in Tables 1 to 3.

Table 1: Instrument and Analytical Conditions

| Element | As | Pb |
|-----------------------|--------------------------------|------------------------------|
| Analytical Wavelength | 194.7 nm | 283.3 nm |
| Slit Width | 0.7 nm | |
| Lamp Current | 12 mA | 10 mA |
| Background Correction | Deuterium lamp method | |
| Graphite tube | Pyrolytic coated graphite tube | |
| Injection Volume | 20 µl | |
| Matrix Modifier | 0.2 % magnesium nitrate, 5 µl | 1 % ammonium phosphate, 5 µl |

Table 2: Temperature Program for As

| | Temp. | Time (sec) | Heat Mode | Sensitivity | Gas Type | Flow Rate |
|---|-------|------------|-----------|-------------------------------------|----------|-----------|
| 1 | 60 | 3 | RAMP | <input type="checkbox"/> | #1 | 0.10 |
| 2 | 120 | 20 | RAMP | <input type="checkbox"/> | #1 | 0.10 |
| 3 | 1000 | 20 | RAMP | <input type="checkbox"/> | #1 | 1.00 |
| 4 | 1000 | 20 | STEP | <input type="checkbox"/> | #1 | 1.00 |
| 5 | 1000 | 3 | STEP | <input checked="" type="checkbox"/> | #1 | 0.00 |
| 6 | 2200 | 3 | STEP | <input checked="" type="checkbox"/> | #1 | 0.00 |
| 7 | 2500 | 2 | STEP | <input type="checkbox"/> | #1 | 1.00 |

Atomization Stage No 6

Table 3: Temperature Program for Pb

| | Temp. | Time (sec) | Heat Mode | Sensitivity | Gas Type | Flow Rate |
|---|-------|------------|-----------|-------------------------------------|----------|-----------|
| 1 | 60 | 3 | RAMP | <input type="checkbox"/> | #1 | 0.10 |
| 2 | 120 | 20 | RAMP | <input type="checkbox"/> | #1 | 0.10 |
| 3 | 250 | 10 | RAMP | <input type="checkbox"/> | #1 | 0.10 |
| 4 | 700 | 10 | RAMP | <input type="checkbox"/> | #1 | 1.00 |
| 5 | 700 | 10 | STEP | <input type="checkbox"/> | #1 | 1.00 |
| 6 | 700 | 3 | STEP | <input checked="" type="checkbox"/> | #1 | 0.00 |
| 7 | 2000 | 3 | STEP | <input checked="" type="checkbox"/> | #1 | 0.00 |
| 8 | 2500 | 2 | STEP | <input type="checkbox"/> | #1 | 1.00 |

Atomization Stage No 7

Results and Discussion

Figures 1 and 2 show the calibration curves of As and Pb with good correlation coefficient (R) of more than 0.9990.

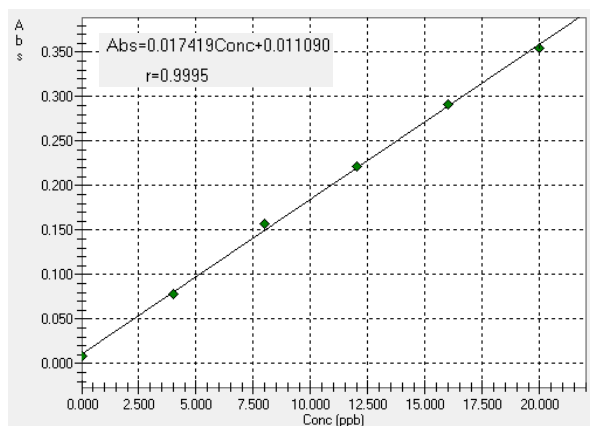


Fig. 1: As Calibration Curve

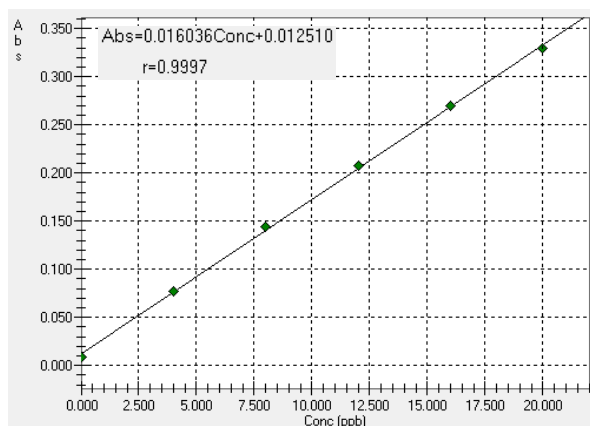


Fig. 2: Pb Calibration Curve

The As and Pb results in Lipstick CRM are shown in Table 4. For the 2 replicate preparations, the results were accurate as the recovery % was within 10 % of the certified values for both elements.

Table 4: As and Pb Results in Lipstick CRM

| Element | As | Pb |
|--|----------------------|-----------------------|
| Certified Content | 7.94 mg/kg | 22.7 mg/kg |
| Lipstick CRM 1 st Preparation | 8.62 mg/kg (108.6 %) | 24.78 mg/kg (109.2 %) |
| Lipstick CRM 2 nd Preparation | 8.14 mg/kg (102.5 %) | 23.17 mg/kg (102.1 %) |

Values in parentheses indicate the % recovery.

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The instrument limit of quantitation (LOQ) and method detection limit (MDL) were calculated as in Application News AD-0219 [3]. Table 5 shows the LOQ and MDL of both elements.

Table 5: LOQ and MDL of As and Pb

| Element | As | Pb |
|---------|-----------|-----------|
| LOQ | 1.03 ppb | 0.31 ppb |
| MDL | 2.58 µg/g | 0.78 µg/g |

From the MDL results, the GFAAS method could meet the 5 ppm and 20 ppm maximum limit allowed by ASEAN AHCRS regulations for As and Pb respectively.

Table 6 shows the measurement result of the cosmetic cream. A pre-digestion spike recovery test was carried out where the cosmetic cream was spiked with 5 ppb for both As and Pb. Both spike recovery tests show recoveries of 100 ± 5%. The spike recovery results for both As and Pb were within the spike recovery range required by ASEAN Cosmetic Methods [4].

Table 6: As and Pb Results in Cosmetic cream

| Element | As | Pb |
|--|----------|----------|
| Cosmetic cream | < MDL | < MDL |
| Cosmetic cream spiked with 5 ppb As and 5 ppb Pb | 4.88 ppb | 5.16 ppb |
| % Spike Recovery | 97.6 % | 103.2 % |

Conclusion

The GFAAS analysis of As and Pb in cosmetics could be carried out using Shimadzu AA-7000 with GFA-7000 as demonstrated in this application news. The analysis of CRM and spike recovery test results show the method to be accurate. The MDL also proved the method could meet ASEAN AHCRS regulations.

References

- Järup, L. (2003). *Hazards of heavy metal contamination*. British Medical Bulletin, 68(1), 167–182.
- ASEAN Guidelines On Limits Of Contaminants For Cosmetics Version 3.0 (2019). Page 5.
- Lee, Z. H., Chua, A. M., and Lim, J. (2020). *Analysis of Mercury in Cosmetics by Cold Vapour Atomic Absorption Spectroscopy (CVAAS)*. Application News, No. AD-0219.
- ASEAN Cosmetic Method 005 (2013). *Determination of Heavy Metals (Arsenic, Cadmium, Lead and Mercury) in Cosmetic Products*.

Related Products

Some products may be updated to newer models.



> AA-7000

Atomic Absorption Spectrophotometer

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