

Application News

Analysis of Heavy Metals in Cosmetics by ICP-MS - ISO 21392 -

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User Benefits

- ◆ Safety assessment of cosmetics can be conducted using ICP-MS measurements as described in ISO 21392.
- ◆ Reduced running costs due to the use of a mini torch that uses less argon gas.

Introduction

Cosmetics are directly applied to the skin. Therefore, any Impurities in them must be carefully monitored. The International Cooperation on Cosmetics Regulation (ICCR) recommended limits for Pb (10 ppm) in 2013 and for Hg (1 ppm) in 2016. Regarding analyzing elements in cosmetics, ISO 21392 (Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique) was issued on August 17, 2021. This test method demonstrates the technique for measuring heavy metals in cosmetic products using ICP-MS. In this Application news, eight elements, including those listed in ISO 21392 (Cr, Co, Ni, As, Cd, Sb, Pb) and mercury (Hg), were analyzed with the Shimadzu ICPMS-2040/2050 ICP mass spectrometer (Fig. 1).

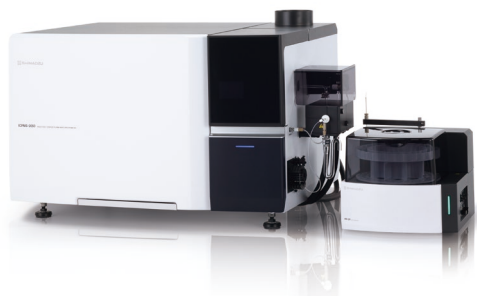


Fig. 1 ICPMS-2040/2050 and AS-20 Autosampler

Sample

Analysis was conducted on lipstick, foundation and eyeshadow.

Sample Preparation

Following the method described in ISO 21392, approximately 200 mg of the sample was mixed with nitric acid, hydrochloric acid, and pure water and then subjected to digestion using a microwave digestion system. A smaller digestion vessel (quartz insert) was used inside a standard digestion vessel.

After the solution was made up to 50 mL, the residue was filtered* and further diluted four times. In other words, the dilution factor from the solid sample to the measurement solution is 1000. Fig. 2 shows the detailed procedure of the sample pretreatment process.

* ISO 21392 states, "It is possible that some cosmetic inorganic ingredients, such as silica or titanium dioxide, are not completely digested under the conditions of this document and that heavy metal confined in such ingredients are not fully extracted. However, the level of heavy metal trapped in these inorganic materials is not considered to significantly contribute to the exposure level of consumers to these heavy metals."

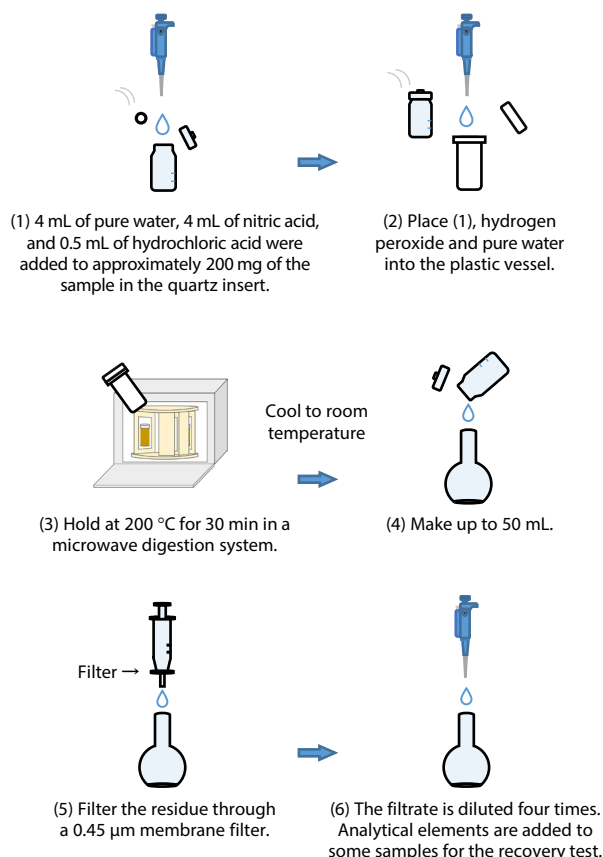


Fig. 2 Sample Preparation Flow

Standard Solution

● Calibration Standards

Calibration Standards were prepared to the concentration shown in Table 1. The same volumes of nitric acid and hydrochloric acid as the sample solutions were added to them.

● Internal Standard Solution

Commercially available single-element standard solutions were diluted and mixed to prepare an internal standard solution containing Rh and Ir at 100 µg/L. The same volumes of nitric acid and hydrochloric acid as the sample solutions were added to the internal standard solutions.

An online internal standard kit was used to automatically add the internal standard solution to a sample at the internal standard ratio of approximately 9:1 to reduce the labor involved in sample preparations.

Table 1 Concentration of Calibration Standards (µg/L)

	BLK	STD1	STD2	STD3	STD4	STD5	STD6	STD7
Cr	0	0.5	1.0	2.0	5.0	10	20	30
Co								
Ni								
As								
Cd								
Sb								
Pb								
Hg								

Equipment Configuration and Analysis Conditions

Table 2 shows the equipment configuration, and Table 3 shows the analysis conditions.

A mini torch, which consumes 2/3 less argon gas than an ordinary torch, was used to reduce running costs.

Only He gas was used as the cell gas to eliminate spectral interference. The gases that can be introduced into the cells of ICPMS-2040 and ICPMS-2050 are shown in Table 4. The same analysis as in this paper is possible with any of the instruments.

Table 2 Equipment Configuration

Instrument:	ICPMS-2040/2050
Nebulizer:	Nebulizer DC04
Chamber:	Cyclone chamber
Torch:	Mini-torch
Sampling Cone:	Nickel
Skimmer Cone:	Nickel
Autosampler:	AS-20
Internal Standard Elements:	Online Internal Standard Kit (sample: internal standard = about 9: 1)

Table 3 Analysis Conditions

RF Power:	1.20 kW
Sampling Depth:	5 mm
Pump Speed:	20 rpm
Plasma Gas Flowrate:	9.0 L/min
Auxiliary Gas Flowrate:	1.10 L/min
Carrier Gas Flowrate:	0.45 L/min
Dilutions Gas Flowrate:	0.40 L/min
Cell Gas:	He

Table 4 Available Cell Gases

Instrument	He	H ₂	3 rd gas (option)
ICPMS-2040	○	×	×
ICPMS-2050	○	○	○

Table 5 Concentration in Cosmetics (mg/kg)

Elements	ICCR recommended limit	LOQ (10σ)	Preparation Blank	Lipstick	Foundation	Eyeshadow
⁵² Cr		0.04	0.05	0.43	3.5	15
⁵⁹ Co		0.01	<	1.1	0.42	6.5
⁶⁰ Ni		0.1	<	0.6	2.0	18
⁷⁵ As		0.04	<	0.05	0.10	0.15
¹¹¹ Cd		0.006	<	<	0.03	<
¹²¹ Sb		0.03	<	<	<	<
²⁰⁸ Pb	10	0.003	0.008	0.030	3.2	2.4
²⁰² Hg	1	0.03	0.05	<	<	<

LOQ (mg/kg): Standard deviation upon repeated analysis of the blank solution × 10 × Calibration curve slope {50 (mL) × 4/0.2 (g)}/1000

<: below the limit of quantitation

Concentration in Cosmetics (mg/kg): {Concentration of Solution - Concentration of Preparation Blank (µg/L)} × {50 (mL) × 4/0.2 (g)}/1000

Measurement Results

Calibration curves were prepared using the standard solutions in Table 1, and the heavy metal content in the lipstick, eyeshadow, and foundation was analyzed. The results are shown in Table 5.

All samples contained less than the ICCR recommended limits for Pb and Hg. In addition, the ICPMS-2040/2050 provided lower limits of quantification than the ICCR recommended limits.

Table 6 shows the Result of Spike Recovery when 5 µg/L of the analyte element was added to the sample solution after sample pretreatment. Good results were obtained, falling within the 100 ± 10 % range.

Table 6 Result of Spike Recovery

Elements	Internal Standard Elements	Lipstick	Foundation	Eyeshadow
		Recovery (%)	Recovery (%)	Recovery (%)
⁵² Cr	¹⁰³ Rh	98	94	103
⁵⁹ Co	¹⁰³ Rh	96	91	97
⁶⁰ Ni	¹⁰³ Rh	95	96	95
⁷⁵ As	¹⁰³ Rh	94	93	95
¹¹¹ Cd	¹⁰³ Rh	94	95	95
¹²¹ Sb	¹⁰³ Rh	94	97	95
²⁰⁸ Pb	¹⁹³ Ir	96	94	99
²⁰² Hg	¹⁹³ Ir	97	94	93

Conclusion

Simultaneous analysis of heavy metals in cosmetics was performed by ICP-MS with reference to ISO21392.

It was found that the ICPMS-2040/2050 could be used for analysis with a sensitivity that was well within the required value. The recovery test results were good, and the validity of the analysis was confirmed.

By using a mini torch, the argon gas consumption and running costs were reduced.

<References>

- December 2013 ICCR-7 Traces Working Group : Considerations on Acceptable Lead Levels in Cosmetic Products
- September 2016 ICCR Joint Traces Working Group : Recommendation for Acceptable Trace Mercury Levels in Cosmetic Products
- ISO 21392:2021 Cosmetics - Analytical methods - Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique