Brazilian Journal of Analytical Chemistry
2025, Volume 12, Issue 48, pp 348-351

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ISO 21392: A turning point in heavy metals analysis of cosmetic products

This report was extracted from the Milestone White Paper ISO 21392:2021 method

The new ISO 21392:2021 method represents a turning point for laboratories that deal with cosmetic samples, because it univocally describes a method for quantification of trace levels of heavy metals in cosmetic products.

BACKGROUND

In effect since August 2021, the new ISO 21392:2021 is titled “Cosmetics — Analytical methods — Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique”. This document defines a universal method to determine the most common trace elements such as chromium, cobalt, arsenic, nickel, cadmium, antimony, and lead.[1]

What was the situation before? Before its implementation, the only existing document supporting this type of analysis was the technical report ISO / TR 17276: 2014 (“Analytical approach for screening and quantification methods for heavy metals in cosmetics”). This was an interesting overview containing general guidelines about the multiple analytical approaches available for heavy metal determinations, but it was lacking in accurate operational details and method performance data. Therefore, the feasibility and the application of this method was totally under control of the laboratories. Consequently, the uniformity of method was not assured, and results obtained by different labs could not be compared.

Due to the increased interest on this topic, in 2016 an international working group was created with the purpose of issuing a new standard method.

In 2020 a draft was published, reaching the status of FDIS (Final Draft International Standard) in 2021, according to the ISO procedures.

The new ISO 21392:2021 method

Why is the ISO 21392 method a turning point? Why is it considered so important?

The new ISO 21392:2021 method[2] does not actually replace the aforementioned ISO / TR 17276: 2014, but it integrates its contents relating to the ICP-MS analytical technique.

At the same time, this new standard can be defined as “self-supporting”. What does self-supporting mean? Any knowledge or any use of other standards is not required at all, because all analytical procedures are specified directly in this ISO method and no consultation of other standards is required for its correct application. This greatly simplifies its adoption in analytical laboratories.

The main strengths of the method described are:

1. The simultaneous and selective determination of all the cited metals is allowed.
2. The appointed detection technique is mass spectrometry because of its high sensitivity: it allows determination of metal traces down to 20 µg / kg (0.02 ppm) or even lower with a standard configuration.

What are the principles of the method?

The method specifically refers to the trace level determination of chromium, cobalt, nickel, arsenic, cadmium, antimony, and lead in cosmetic products.

Samples need to be digested prior analysis, under very high temperature and pressure conditions, typically by processing them in a microwave digestion system.

The method involves the acid mineralization of an aliquot of sample, with an oxidizing mixture composed of concentrated nitric acid + hydrochloric acid, under pressure and at about 200 °C, using a microwave digestion system, followed by dilution and analytical determination with inductively coupled plasma - mass spectrometry (ICP- MS). Instrument calibration is performed using a series of defined calibration standards. Instrument stability is ensured and matrix effects are corrected by using internal standards (rhodium and lutetium).

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 metals trapped in these inorganic materials is not significantly contributing to the exposure level of consumers to these heavy metals. The use of ICP-MS ensures reliable measurement of trace levels of heavy metals due to its proven high sensitivity and selectivity.

The overall concentration range for which the method was validated by the laboratories participating in the working group is between 0.1 and 50 mg/kg. This means that any cosmetic product, even those containing high levels of inorganic dyes, can be tested. The recovery percentages are in a range ± 40% with respect to the expected value, while the relative reproducibility is between 3.5% and 15%, depending on the metal and the matrix tested.

These values may seem high at first glance; however, they must be interpreted considering that the average concentrations of the metals refer to the results obtained by an interlaboratory study involving a series of comparison analyses on common test matrices.

It is worth mentioning that cosmetic products can have uneven distributions of the heavy metals they contain. This factor may lead to an intrinsic negative impact on the reported method reproducibility, irrespective of the operating conditions.

Microwave digestion

How to prepare cosmetic samples for elemental analysis? To be in compliance with the new ISO 21392 method, laboratories must equip themselves with a microwave digestion system. Not all microwave units are created equal. The method specifies that the microwave system and its pressurized vessels must be able to reach a temperature of 200 °C and a pressure of 40 bar.

Moreover, the method is valid for all type of cosmetics that contain different ingredients, so samples can be very different from one to another. An important parameter to be consider when choosing the right microwave digestion system for analytical laboratories, is the ability to deal with different sample reactivities.

There are two very different commercially- available designs of microwave digestion systems: rotor-based systems and single reaction chamber (SRC) technology. How one goes about selecting the optimum technology for a particular lab depends on the types of samples being run, productivity and throughput needed, and temperature and pressure required for the complete digestion of the different sample matrices being run. Having a good understanding of these parameters will allow you to identify the microwave technology best suited to your specific needs, while ensuring the quality of sample preparation necessary to achieve the accuracy of analysis you require.

Rotor-based microwave digestion

Microwave rotor-based closed-vessel digestion is the benchmark for numerous industries and applications, including food, feed, pharmaceutical, nutraceutical, polymers, consumer product testing, petrochemical, clinical, biological and cannabis. The first advantage that leveraged the use and the establishment of the microwave closed-vessel technology was its ability to reduce the sample preparation time from hours to minutes. Digestion was no longer limited by the atmospheric pressure boiling point of the mineral acid used, since closed vessels allow working at temperatures above it. However, several other benefits immediately emerged from the use of this technology:

- High digestion quality eliminates interferences;
- Full recovery of all elements, including volatiles;
- Lower blanks;
- No contamination from the environment;
- Superior working conditions and dramatic reduction of toxic digestion gases.

A real breakthrough was the introduction of the rotor technology specifically developed by Milestone for better reproducibility in 1990. The rotor technology, in combination with the vent-and-reseal technology[4] and the safety shield construction, led to the development of the first high-pressure rotor available on the market.

The vessel construction and the rotor technology need a minimum of parts (only vessel, cap and pressure shield), which offers easy handling and cleaning. All these improvements positively affected the evolution of closed-vessel microwave digestion, extending its applicability and capacity.

Along with the mentioned improvements, the microwave closed-vessel technology continued its incremental innovation in several areas and today offers the following advantages:

- High safety;
- A wide selection of rotors and vessels;
- Full reaction control of temperature, pressure, and leaking of vessels.

Microwave digestion has evolved along with the elemental analysis needs of labs and atomic spectroscopy instrumentation. The Milestone ETHOS UP (Figure 1) represents the latest evolution of rotor-based technology, which incorporates over 30 years of experience and knowledge in microwave sample preparation. It perfectly merges the four pillars of a microwave digestion system: microwave hardware, reaction vessels, reaction sensors, and user interface, providing:

- Enhanced ease of use and productivity
- Superior digestion quality with no method development
- Rotors with the highest throughput and performance
- Superior safety and reliability

Figure 1. Milestone's ETHOS UP microwave digestion system

SRC microwave digestion

Although conventional microwave systems have brought acid digestion to a high level of quality, the operator often has to go through tedious and time-consuming procedures to use them.

Milestone's Single Reaction Chamber (SRC) technology has revolutionized how industrial and research laboratories around the world prepare samples for analysis, with over a thousand units installed globally.

ultraWAVE (Figure 2), an SRC-based digestion system, transcends traditional closed- and open- vessel digestion, offering faster digestions, maximum throughput, and lower cost of ownership.

Figure 2. Milestone's ultraWAVE SRC-based digestion system.

ultraWAVE is able to enhance your lab workflow +50%, transforming the way analytical chemists prepare their samples for trace metal analysis. With double the pressure and higher temperature capabilities than conventional rotor-based microwave systems, the ultraWAVE allows the digestion of virtually any matrices.

With the ultraWAVE, there is no requirement to batch samples with a similar matrix. As a result, all sample types can be digested at the same time. With the unique nitrogen- pressurized capping system, samples of widely different analyte concentrations can be positioned next to each other in the chamber without concerns of cross-contamination.

The fast assembly of the vials and the automatic pressurization and venting make the digestion process more efficient, reducing the labour cost up to 50%.

Milestone offers a wide range of racks and vials, including racks that accommodate vials with different volume types to digest different sample types and amounts in a single microwave program, enhancing the lab workflow and productivity.

Unlike conventional microwave digestion systems, no vessel assembly or disassembly is required. The use of disposable glass vials eliminates the cleaning step, further enhancing the lab's workflow. The handling of the system is dramatically reduced, as the digestion vials are made of only two components, simplifying all the sample preparation process

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Running costs are significantly cut by decreased acid use, increasing the consumables lifetime, and by using inexpensive vials suitable for trace metals determinations.

CONCLUSIONS

Based on the above considerations, the new standard will be a significant asset for analytical laboratories which, in turn, will be able to communicate to the safety assessors of cosmetic products the contents of heavy metals obtained according to a globally recognized method[3].

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