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Heavy Metal Analysis of Cosmetics & Personal Care Products: A Critical and Unavoidable Global Challenge

By Matthew Linsey* and Ian Milnes**

Global cosmetic and personal care products are a multi-billion dollar revenue producing industry that appears to be growing regardless of economic conditions. Due to the growing demand for these products and the significant amount of sales being generated, the industry has become a target for increased regulatory scrutiny around the world.

One aspect of concern to many is the safety of these products being sold to consumers. The European Union and other geographical areas have instituted a variety of regulations intended to protect the public. Further, legislators in both the United States and Canada have recently proposed laws to address product safety concerns.

The Safe Cosmetics Act of 2010 (H.R. 5786) was introduced in the U.S. House of Representatives in July of 2010. This proposal's objective is to protect consumers of cosmetic and personal care products from being exposed to harmful, and potentially, carcinogenic substances. The Act would require cosmetic and personal care product companies to assess their products for the presence of unhealthy materials, whether inherent or contaminant in nature and to make sure any detectable levels found are in compliance with any current health hazard exposure levels. The Act, in many ways, would be analogous in nature to what the Consumer Product Safety Initiative Act (CPSIA) was designed for, and continues to be, for the children's toy industry.

One of the main focal points for the cosmetics and personal care industry is the presence of heavy metals in products introduced into the stream of commerce. Unlike product forms like nanoparticles, and the potential toxicity unknowns associated with them, heavy metals are far more easy to define and regulate as clear health concerns because of their known toxicity and carcinogenic effects. There is considerable data on the toxicity of heavy metals and their ability to bio-accumulate, even with daily exposure to trace level amounts.

It has been shown in many recent studies that several cosmetic products contain trace amounts of heavy metals. These include, but are not limited to: arsenic, cadmium, lead, and mercury. For example, a study conducted by Environmental Defense in Canada con-

cluded that all 49 facial cosmetic products they tested contained trace amounts of at least one heavy metal. The results of the study have led many to clamor for Canadian officials to enact guidelines on impurities in cosmetics. These guidelines were first set forth in early 2009.

Types of Analysis

There are two basic types of methods used to assay for heavy metals. Classical methods use colorimetric bench chemistry techniques where the concentrations of the heavy metals are measured as a group of like elements. Newer methods utilize sophisticated instrumentation to measure individual elements, where the total sum of heavy metals is derived by adding the individual elemental results.

Classical colorimetric techniques can be performed in the most basic of laboratories using normal laboratory glassware, reagents, and equipment. They do not require any expensive instrumentation. These methods are generally simple to perform. The disadvantages for these methods are they have high detection levels and extremely limited specificity. Such approaches are becoming generally viewed as being antiquated and unsatisfactory for the purposes of current and emerging product safety compliance.

Instrumental Methods

There are four main instrumental methods routinely used to measure levels of heavy metals. All of these methods rely on sample preparations that dissolve samples using concentrated acids, such as nitric and hydrofluoric acids, and hydrogen peroxide.

The first and oldest instrumental method is Flame Atomic Absorption Spectroscopy (FAAS). This method relies on the electrochemical properties of metals that allow them to absorb energy from light of specific wavelengths. The relationship between the amount of light absorbed and the concentration of analytes present in known standards can be used to determine sample concentrations by measuring the amount of light that they absorb.

* Assistant Technical Director, Chemical Solutions Ltd., 273 Mulberry Dr. Suite 9, Mechanicsburg, PA 17050, (717)-697-7536, www.chemicalsolutionsltd.com

** President Chemical Solutions Ltd.

A second instrumental technique employed is Graphite Furnace Atomic Absorbance Spectroscopy (GFAAS). This method is very similar to FAAS but uses a different sampling system. By using an electrically heated graphite tube instead of a flame, GFAAS is able to atomize the entire sample and retain it in the path of the light for an extended period of time. This difference leads to a significant improvement in detection limits. One major disadvantage of both FAAS and GFAAS, however, is their inability to measure multiple elements simultaneously.

One of the most common instrumental methods used to assay for heavy metals is Inductively Coupled Plasma-Atomic Emission Spectroscopy (ICP-AES or ICP-OES). This technique uses argon inductively coupled plasma maintained by the interaction of a radio frequency field and ionized argon gas to excite atoms to unstable energy configurations. When the atoms return to more stable configurations, energy is released in the form of specific emitted light wavelengths. The wavelengths of the energy released are specific to the elements in the sample, and the intensity of the emission is a function of the concentration of atoms that are affected.

The final instrumental technique utilizes the newest and most expensive equipment. It is known as Inductively Coupled Plasma-Mass Spectroscopy (ICP-MS). ICP-MS is very similar to ICP-AES in that they both use the same sample introduction system and are both capable of measuring multiple elements simultaneously. However in ICP-MS, the atomic ions produced by the argon plasma are directed into a mass spectrometer, which separates the ions according to their mass-to-charge ratio. Ions of the selected mass-to-charge ratio are directed to the detector, which measure the number of ions present thereby producing identification and quantification of the elements of interest.

All of the above-mentioned instrumental methods have advantages and disadvantages to them. Some examples of these include: Interferences, detection limits, precision, ease of use, operating costs, sample throughput, and cost per sample. While detailed discussion of all of the advantages and disadvantages in depth is beyond the scope of this article Table 1 does provide a comprehensive overview. The most important aspects to highlight are detection limit, followed by interferences, dynamic range, and precision.

As shown in Table 2, detection limits vary from element to element and method to method, but for the four main heavy metals (arsenic, cadmium, lead, and mercury) the best overall technique is ICP-MS. ICP-MS is also the technique with the least amount of interferences and its dynamic range tends to be superior even to that of ICP-AES.

Most companies do not have the means or technical expertise to do this type of testing within the confinement of their own in-house businesses (a new ICP-MS instrument can cost in the area of

\$200,000). An appropriate alternative is to use an independent, third-party laboratory. Such independent testing laboratories are an integral part of data collection for many companies requiring such testing. One such laboratory specializing in the area of trace level elemental analysis is Chemical Solutions Ltd (CSL). CSL maintains three Perkin-Elmer ICP-MS instruments, including their newest model, the NexION (Figure 1).



Figure 1

Three Recommendations for How To Proceed Responsibly

For cosmetic and personal care product companies beginning the process of assessing the safety of their products. It is imperative to recognize the regulatory standards one is, or will be, required to comply with. This is a challenging job in view of the fact that said regulations vary globally and constantly seem to be in a state of flux. This is not a simple task and depends in part on where the particular product is intended to be sold. Individuals and companies responsible must familiarize themselves and stay abreast of the limits set forth in these laws. Individuals responsible for such product safety analyses will then be able to identify and determine which analytical tests to perform on one's products and how to better interpret the product's analytical testing results.

Another consideration is the establishment of baseline values of the testing parameters chosen for each product. This is typically done by testing many different lots of the same material on a regular basis. Testing frequency may then be decreased if the product has consistently been shown to be within acceptable limits by means of good statistical protocols.

Finally, it is always a good idea to periodically check and verify ingredient suppliers' Certificates of Analysis (CoFA). This will ensure the quality of products from suppliers. This last suggestion is based on legal responsibilities of companies in the product chain which can generally range from manufacturer through distributor.

	FAAS	GFAAS	ICP-AES	ICP-MS
Detection limit	Very good for some elements	Excellent for some elements	Very good for some elements	Excellent for most elements
Analytical capability	Single element	Single element	Multi-element	Multi-element
Linear dynamic range	10 ³	10 ²	10 ⁵	10 ⁵
Sample through put	10 sec/element	2 min/element	5-30 elements/min/sample	All elements 2-6 min/sample
Precision	0.1-1%	1-5%	0.3-2%	1-3%
Interferences spectral	Few	Very few	Common	Few
Interferences chemical	Many	Many	Very few	Some
Interferences physical	Some	Very few	Some	Some
Dissolved solids	Up to 5 %	Up to 10%	Up to 20%	0.1-0.4%
Applicability	>60%	>50%	>70%	>80%
Method development	Easy	Easy	Easy	More difficult
Ease of use	Easy	Easy	Easy	Easy
Initial cost	Low	Medium	High	Very high
Operating cost	Low	Medium	Low	High
Cost per sample	Low	Medium	Low	Medium

Table 1. Comparison of Various Instrumental Techniques¹

Element	FAAS	GFAAS	ICP-AES	ICP-MS
Lead	15-20	0.5	1	<0.05
Arsenic	150	1	20	<0.05
Mercury	300	0.6	1	<0.05
Cadmium	0.8	0.002	0.1	<0.05

Table 2. Detection Limit² Comparisons ($\mu\text{g/L}$ or ppb)

1 Information in Tables 1 and 2 extrapolated from Tyler G. ICP-MS, or ICP-AES and AAS? – a comparison. Varian Australia Pty Ltd. April 1994 (<https://www.varianinc.com/media/sci/apps/ispms01.pdf> accessed on December 30, 2008); and from: Anon. Guide to Inorganic Analysis. 2004. Perkin Elmer, Inc. (http://las.perkinelmer.com/content/Manuals/GDE_InorganicAnalysis.pdf accessed on December 30, 2008).

2 For FAAS, ICP-AES, and ICP-MS the detection limit is defined on the basis of 3 standard deviations of the blank. For GFAAS sensitivity (0.0044 absorbance) is measured with 20 μl of sample. ■

SLI Chemicals GmbH
Trading in Fine Chemicals

60487 Frankfurt am Main, Insterburger Strasse 7
Tel.: +49 (0)69 74 74 28 0 - Fax: +49 (0)69 74 74 28 20
e-mail: info@slichemicals.com



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