Office for Product Safety & Standards

A Feasibility Study Investigating Action Limits for Certain Heavy Metal Impurities in Cosmetic Products.

Research

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A Feasibility Study Investigating Action Limits for Certain Heavy Metal Impurities in Cosmetic Products

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Executive Summary

Introduction

1. Cosmetic products are regulated in the UK by EU retained law Regulation (EC) No 1223/2009 which sets the rules on the supply of cosmetic products and lists prohibited substances not allowed to be present in the cosmetic product.

2. There is an exemption for non-intended presence of a small quantity of a prohibited substance, from impurities of natural or synthetic ingredients, the manufacturing process, storage, migration from packaging, technically unavoidable in good manufacturing practice, which shall be permitted provided it is not harmful to health of the user.

3. Metals identified as heavy metals including antimony, arsenic, cadmium, lead and mercury, often described as heavy metals, are prohibited substances within the scope of the Cosmetic Products Regulation, although may be present in cosmetic products as impurities.

4. There is no UK guidance on the maximum content for impurities of antimony, arsenic, cadmium, lead and mercury in a cosmetic product to fulfil the exemption of technically unavoidable in good manufacturing practice.

5. OPSS commissioned LGC to investigate the feasibility in developing UK guidance for technically unavoidable levels of antimony, arsenic, cadmium, lead and mercury impurities in cosmetic products in a UK market survey.

Scope and methodology

6. A literature review to identify guidance or legal requirements in other countries on technically unavoidable heavy metal levels in cosmetic products and analytical methods with corresponding data.

7. Identification and verification of a suitable method for determining metal impurities in cosmetic products was performed. Purchase of cosmetic products was based on exposure risk, across a price range from high street and online retailers.

Literature review

8. China has set regulatory maximum content limits in cosmetic products for arsenic (2 mg/kg), cadmium (5 mg/kg), lead (10 mg/kg) and mercury (1 mg/kg). Thailand has a single regulatory maximum limit for cadmium (3 mg/kg) in cosmetic products.

9. The USA has a maximum content limit for mercury (1 mg/kg) in cosmetic products but also regulatory limits for colour additives used in cosmetic products for including arsenic, lead and mercury. In addition, a guidance limit for lead (10 mg/kg) in cosmetic lip products has been advised.

10. Japan, Canada, Germany, and member countries in the South East Asian Association (ASEAN) have set different non-regulatory guidance for maximum content limits. Germany (BVL) has the lowest guidance limits with general limits for technically unavoidable heavy metal levels for antimony (<0.5 mg/kg), arsenic (<0.5 mg/kg), cadmium (<0.1 mg/kg), lead (<2 mg/kg) and mercury (<0.1 mg/kg).

11. The literature review identified a variety of acids and procedures used for determining heavy metals in cosmetic products using either a microwave system, oven or hot plate. The only publication describing a standardised method for determining metals in cosmetic products is ISO 21392:2021 – Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique.

Experimental

12. Ninety-one cosmetic products were purchased from high street and online retailers representing multiple brands and price ranges for 10 product types: (1) lipstick, (2) lip gloss, (3) lip liner, (4) toothpaste, (5) mouthwash, (6) sunscreen (7) foundation, (8) mascara, (9) eye liner and (10) eye shadow.

13. ISO 21392:2021 was identified as the suitable method and adapted to include hydrofluoric acid to extract metals from silicates and other similar ingredients that could retain metals preventing complete extraction from the cosmetic product. It was verified using predefined qualification criteria and quality control materials. All samples were analysed in duplicate.

Results

14. Antimony was not detected (<0.1 mg/kg) in 58 out 91 products. Applying the BVL (German) guidance limit (<0.5 mg/kg), 5 products (2 lip liners, 2 eyeshadows and 1 foundation cream) exceeded the limit where the highest result was in an eye shadow ~1.05 mg/kg.

15. Arsenic was not detected (<0.1 mg/kg) in 34 out 91 products. Compared to the BVL guidance limit (<0.5 mg/kg), 4 products (1 lip liner and 3 eye shadows) exceeded the limit where the highest result was in an eye shadow ~1.62 mg/kg.

16. Cadmium was not detected (<0.005 mg/kg) in 30 out 91 samples. Using the BVL guidance limit (<0.1 mg/kg), no product exceeded this, with the highest result found in an eye shadow at ~0.05 mg/kg.

17. Lead was not detected (<0.2 mg/kg) in 16 out 91. Applying the BVL guidance limit (<2 mg/kg), 10 products (3 lip liners, 6 eyeshadows and 1 eye liner) exceeded the limit where the highest result was in an eye shadow ~8.8 mg/kg.

18. Mercury was not detected (<0.07 mg/kg) in 50 out 91 samples. Using the BVL guidance limit (<0.1 mg/kg), 4 products (3 eye shadows and 1 lip gloss) were above this, with the highest result was found in an eye shadow ~0.52 mg/kg.

Discussion

19. Price and place of purchase did not appear to have an influence on the levels of metals found in products.

20. Using the BVL guidance limits for technically unavoidable impurities in cosmetic products, the management risk acceptance criteria of 90% passing the guidance limits could be met from the UK products tested within this study.

Conclusion

21. This report reviewed international legislation and guidance, assessed the feasibility of method implementation and performed a small-scale survey to determine the current UK market status. This has established the feasibility of setting guidance values for technically avoidable heavy metals in UK cosmetic products to meet regulatory requirements.

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1. Introduction

1.1. Overview

Cosmetic products are regulated in the UK by EU retained law Regulation (EC) No 1223/2009 which sets the rules on the supply of cosmetic products where the overriding consideration is ensuring safe cosmetic products for the consumer. To meet this objective the cosmetics regulation contains annexes setting out legal requirements for certain ingredients/chemicals in cosmetic products, in Annex II to Regulation (EC) No 1223/2009 listing approximately 1300 prohibited substances in cosmetic products. However, there are no notes in the Articles or Annex II to describe how compliance is demonstrated by the manufacturer or enforced by authorities. Therefore, no listed substances in Annex II can be present in a cosmetic product at any concentration unless otherwise stated by Regulation (EC) No 1223/2009. Manufacturers are therefore required to ensure when sourcing ingredients and during manufacture that no prohibited substances are present in the cosmetic products.

Certain metals (commonly referred to as ‘heavy metals’) are prohibited substances in cosmetic products due to their toxicity but as these heavy metals can be naturally found in the environment, it is possible that trace amounts will be present in the minerals used in providing colour to cosmetic products. Unless these heavy metals are removed from the cosmetic ingredient prior to inclusion or during the manufacturing process then trace amounts of heavy metals will also be present in the cosmetic product.

The UK Cosmetic Products Regulation (CPR) specifies the non-intended presence of a small quantity of a prohibited substance (stemming from impurities of natural or synthetic ingredients, the manufacturing process, storage, migration from packaging, etc.) which is technically unavoidable in good manufacturing practice, shall be permitted provided the cosmetic product is safe. The UK currently does not provide guidance on the levels of prohibited heavy metals considered ‘technically unavoidable in good manufacturing practice’, but it is recognised that an ‘action limit’ for each prohibited metal could provide clarity for manufacturers and enforcement agencies. This approach in specifying guidance for technically unavoidable levels for prohibited metals in cosmetic products has already been adopted by some countries in North America, the European Union (EU) and Asia.

This project is part of the BEIS Office for Product Safety and Standards (OPSS) Strategic Research Programme (SRP), launched in March 2018. The SRP provides high quality strategic research to strengthen the evidence base for Safety and Standards policy development, delivery, and enforcement, giving business the confidence to innovate and protecting consumers from unsafe products. The wide range of evidence-based research supported by the SRP helps to address critical questions relating to current product safety, and/or issues that might arise due to future market developments. In considering potential UK guidance for setting technically unavoidable levels or action limits for prohibited heavy metals in cosmetic products, OPSS aims to incorporate best practice based on the latest knowledge and technology to provide clear guidance to national authorities, test houses and industry regarding acceptable levels of unavoidable heavy metal impurities, thus strengthening the safety of UK cosmetic products.

To fulfil this aim, the following objectives have been set by OPSS for the project:

- Carry out a short literature review of relevant work previously completed to ensure a considered approach.
- Engage with key stakeholders to ensure consideration of practical aspects from end-user perspectives.
- Identify a variety of suitable cosmetic products that provide a representative sample of the UK population and market.
- Utilise an acceptable analytical approach for a minimum of five heavy metals including antimony (Sb), arsenic (As), cadmium (Cd), lead (Pb), and mercury (Hg).
- Carry out data analysis to determine action limits for the heavy metals examined.

1.2. Project Implementation

Undertaking a monitoring exercise of toxic elements in cosmetic products requires robust validated methods to be able to determine quantitatively all the target metals in different cosmetic matrices. It is also important to establish that the validated method is fit for purpose and can be applied by both public analyst and industry laboratories, which will be necessary for demonstrating comparability of data and for compliance purposes.

Section 2 of this report covers a review of the literature surrounding the regulations, guidance, analytical methods and existing data on heavy metals in cosmetic products from international sources. This will inform the sample selection and analytical methodology as it has been recognised that the variety of matrices can suffer from difficulties such as the presence of refractory ingredients such as silicon dioxide (SiO_2) and titanium dioxide (TiO_2) which do not dissolve under standard protocols without the use of hydrofluoric acid (HF). However, it should be noted that HF has toxicological and corrosive properties, that require careful handling in the laboratory, but this should be within the abilities of a competent analytical laboratory.

An important consideration will be the limit of detection (LOD) and quantification (LOQ) of the method where laboratories will have different instruments. Consideration may be needed regarding the lowest level of metal impurities that can be detected by a high performing laboratory compared to most competent laboratories. In addition to the measurement of metal impurities, it is important that correct sampling procedures are followed, as dispersion of pigments in waxes can occur and therefore any impurities may be unevenly distributed throughout the sample, e.g. lipsticks or high viscosity matrices such as toothpaste. A validated method encompassing these points is essential to ensure the project is a success.

To achieve the objectives within the project constraints, the product groups were rated based on the presence of coloured pigments likely to be ingested, where the risk of heavy metals was considered to present the greatest risk to the consumer. Therefore, product groups with a risk rating of 1 and 2 were prioritised, with risk ratings 3 and 4 considered depending on budget/timescales.

Table 1: Target Product Groups for the Study and Consumer Risk Rating

Product Group	SiO_2/TiO_2 present	Potential pathway according to intended use	Risk Rating
Lipsticks/lip gloss and lip liners	Yes	Ingestion	1
Toothpastes/teeth whitening products/mouthwash	Yes	Ingestion	1
Make-up powders	Possible	Eyes	2
Eye makeup products	Yes	Eyes	2
Sun protection	Possible	Ingestion	2
Body and baby powders	Yes	Inhalation	3
Body and facial creams	Possible	None	3
Deodorants	Possible	None	4

Samples were collected from high street stores and online retailers, covering 3 price ranges. This will enable a viewpoint on the current status of the UK cosmetics market with respect to heavy metals. The results will provide valuable data to consider future guidance for the UK to ensure consumer safety.

1.3. Stakeholder Engagement

Virtual meetings were held with stakeholders consisting of officials from OPSS and representatives from the public analyst laboratories, the industry trade body and trading standards.

The first meeting was held on the 13th December 2021 to discuss the scope of the project and for LGC to receive guidance.

The second meeting was held on the 30th May 2022 to discuss the findings of the project and to receive feedback from stakeholders on the results of analysis.

2. Literature Review

2.1. Approach

To identify existing literature concerning technically unavoidable levels or action limits for prohibited heavy metals in cosmetic products, we searched public data services as well as utilising sources available through LGC's involvement in product safety issues over many years. Peer-reviewed literature was identified with search terms in public databases using keywords. Titles and abstracts of the publications returned by the literature search were assessed to eliminate duplicates and then screened to identify a subset of "key" sources that meet criteria for relevance and usefulness for the report or issue papers. Key sources were "tagged" to assist in identifying the most relevant sources for topics covered in the report. The search of peer-reviewed literature focused on references relevant to the analysis of metals in cosmetic products since 1976 when the exemption in Europe to allow unavoidable prohibited trace metal impurities in cosmetic was first included in legislation.

The following databases were searched for relevant peer-reviewed literature:

- **Google Schola**r: Google Scholar provides a simple way to broadly search for scholarly literature. From one place, you can search across many disciplines and sources: articles, theses, books, abstracts and opinions, from academic publishers, professional societies, online repositories, universities and other web sites.
- **ScienceDirect**: ScienceDirect is Elsevier's platform of peer-reviewed scholarly literature. It includes thousands of books, journal articles, and other reference materials.
- **PubChem**: US National Library of Medicine National Institutes of Health. PubChem provides a collection of freely accessible chemical information. Search chemicals by name, molecular formula, structure, and other identifiers.
- **EU SCIENCE HUB**: The European Commission's science and knowledge service.

The search focused on four aspects to the project considered important in helping to understand and meet the aims of the project namely:

Regulations

To determine best practice, it is important to compare regulations made in different countries where the concept of technically unavoidable levels or action limits for prohibited heavy metals in cosmetic products has been established, to examine the scope of the regulation and application.

Guidance

There is guidance linked to regulation but more likely there will be non-regulatory guidance produced by governments and institutions that will inform current practice.

Methods

It is important to understand the analytical techniques used for determining the metal content and the availability of standardised methods. The sample preparation and extraction protocols are also important consideration. Furthermore, the limits of quantification (LOQ) should be assessed for the different extraction techniques and instruments.

Data from publications

There will be papers published on analyses of cosmetic products around the world providing information on ranges of heavy metals detected in cosmetic products to provide indicative levels.

2.2. Regulations

United Kingdom and Europe

Cosmetic products are regulated in the UK by EU retained law Regulation (EC) No.1223/2009 (European Union, 2009) setting out the rules on the supply of cosmetic products where the overriding consideration is ensuring safe cosmetic products are placed on the UK market and EU member countries. Regulation (EC) No.1223/2009 is a revision of Directive 76/768/EEC (European Union, 1976) which was the first regulation to set requirements in 1976 for cosmetic products for members of the European Economic Community (EEC).

Regulation (EC) No.1223/2009 describes legal requirements in Articles with Annexes identifying specific substances and ingredients that are banned or permitted in cosmetic products. Prohibited substances are listed in Annex II 'List of Substances Prohibited in Cosmetic Products' where there are currently approximately 1300 banned substances. The listing of prohibited substances was based on knowledge at the time the legislation was originally drafted of toxic substances known or suspected of being present in cosmetic products and considered harmful.

Annex II does not define the scope of the prohibition and therefore the only interpretation that can be made is that the prohibited substance cannot be present in the cosmetic product at any concentration. It is, however, recognised that some prohibited substances may inadvertently be present in ingredients as impurities. To address this situation, there is allowance for impurities to be present subject to certain conditions described in Article 17 'Traces of prohibited substances' which states '*The non-intended presence of a small quantity of a prohibited substance, stemming from impurities of natural or synthetic ingredients, the manufacturing process, storage, migration from packaging, which is technically unavoidable in good manufacturing practice, shall be permitted provided that such presence is in conformity with Article 3.* Where Article 3 'Safety' states *'A cosmetic product made available on the market shall be safe for human health when used under normal or reasonably foreseeable conditions of use…'.* Article 17 also needs to be read in conjunction with Article 8 'Good Manufacturing Practice' where *'Compliance with good manufacturing practice shall be presumed where the manufacture is in accordance with the relevant harmonised standards, the references of which have been published in the Official Journal of the European Union.'*

In summary, provided there is only a small quantity of the prohibited substance which has not been deliberately created or unintentionally added to the cosmetic product that has been formulated under good manufacturing processes and where the small quantity of the prohibited substance is assessed as not harmful to health, the prohibited substance is exceptionally permitted.

The list of prohibited substances in Annex II for metals and their compounds includes entry 40 for antimony (Sb), entry 43 for arsenic (As), entry 68 for cadmium (Cd), entry 221 for mercury (Hg) and entry 289 for lead (Pb). Additionally, entry 54 for beryllium (Be), entry 297 for selenium (Se), entry 312 for tellurium (Te), entry 317 for thallium (Th), entry 391 for zirconium (Zr), entry 1093 for nickel (Ni) and entry 1646 for cobalt (Co) are listed.

The group of metals including antimony, arsenic, cadmium, mercury and lead are commonly referred to as 'heavy metals' although the term is not defined and has no chemical meaning but is generally used as a shorthand to avoid repetitively listing each metal. As the term 'heavy metal' can include other metals, care is needed when the term 'heavy metal' is used in papers or reports to identify which elements are included.

Canada, Japan, China & USA

There are similar restrictions elsewhere in the world to Europe where heavy metals are prohibited in cosmetic products and regulations applicable to Canada (Canada Government, 2019), Japan (Japan Government, 2000), China (China Food and Drugs Administration, 2015) and the USA (USA Government, 2022) were reviewed.

Regulations in Canada and Japan do not identify individual prohibited substances but describe requirements in broad terms, as shown in the following translated Japanese text: '*Ingredients of cosmetics including any impurities contained therein shall not contain anything that may cause infection or that otherwise makes the use of the cosmetics a potential health hazard'*. In absence of specific limits values in the legal text, Japan and Canada have chosen to provide non-regulatory guidance. As Japan and Canada have non-regulatory guidance, it is left to manufacturer and enforcement agency to consider whether action is needed for each type of cosmetic

The regulatory system in China is based on 'Regulations Concerning the Hygiene Supervision Over Cosmetics' approved by the State Council on 26 September 1989 issued by Decree No. 3 of the Ministry of Public Health on 13 November 1989. The legislation is underpinned by the Technical Safety Standard for Cosmetics 2015 defining the technical requirements for the safety of cosmetics, including general requirements, restriction of component requirements, and test evaluation methods.

The status of cosmetics regulations in China was not obvious when reviewing but websites (C&K Testing Technic Co, 2022) and papers describing services to meet China's cosmetic regulations indicate the following content limits apply as shown in Table 2.

Table 2: Technical Safety Standard for Cosmetics in China.

Metal	Content Limits for Metals in Cosmetic Products (mg/kg)
As	≤2
Cd	≤5
Pb	≤10
Hg	≤1

The US Code of Federal Regulations (USA Government, 2022) § 700.13 only specifies trace limits for mercury in cosmetic products with two conditions '*… a cosmetic containing no more than a trace amount of mercury and such trace amount is unavoidable under conditions of good manufacturing practice and is less than 1 part per million (0.0001 percent), calculated as the metal' and '… a cosmetic intended for use only in the area of the eye, it contains no more than 65 parts per million (0.0065 percent) of mercury, calculated as the metal, as a preservative, and there is no effective and safe nonmercurial substitute preservative available for use in such cosmetic'*.

Therefore, the US restrictions can therefore be expressed as <1 mg/kg Hg in a cosmetic product except for its use as a preservative used in the eye area where there is a higher regulatory limit of <65 mg/kg calculated as the metal.

In addition to the limits for trace amounts of Hg in cosmetic products, the USA also sets restrictions in legislation for colour additives (USA Government, 2022) when used as a cosmetic ingredient where there is a certification process. There is a list of permitted colours that may be safely used in colouring cosmetics, including cosmetics intended for use around the eye, in amounts consistent with good manufacturing practice. Generally, limit values for the heavy metals Pb, Cd, As and Hg are specified for the referenced colorant as an ingredient and not in the final cosmetic product.

2.3. Guidance

As there are few regulatory limits specified for avoidable trace impurities of heavy metals in cosmetic products, there is reliance on published guidance. The following section describes guidance set by authorities at international, regional and national level.

Global

The organisation International Cooperation on Cosmetics Regulation (ICCR) is a voluntary international group of cosmetics regulatory authorities (International Cooperation on Cosmetics Regulation, 2007) including Brazil, Canada, Chinese Taipei, the European Union, Japan, Republic of Korea and the United States. The regulatory authorities meet on an annual basis to discuss common issues on cosmetics safety and regulation, as well as entering constructive dialogue with relevant cosmetics industry trade associations.

The ICCR produced papers on traces of impurities and contaminants in cosmetic products starting with a draft report '*Principles for the Handling of Traces of Impurities and/or Contaminants in Cosmetic Products'* (International Cooperation on Cosmetics

Regulation, 2011). This provided a framework to address trace impurities acknowledging different countries will apply different prohibitions or restrictions but recognising the technical challenge where '*Due to the ever-increasing sensitivity of analytical methods, lower levels of traces of unwanted substances may be detected in cosmetic products, even if they are produced according to state-of the art sourcing and manufacturing practices'*

Accompanying the draft report on principles for handling trace impurities, are two reports addressing lead and mercury impurities in cosmetic products.

The first report '*Considerations on Acceptable Lead Levels in Cosmetic Products (Excluding Products Used in the Oral Cavity)*' (International Cooperation on Cosmetics Regulation, 2013) looks at exposure and tolerance to Pb in different ICCR jurisdictions before recommending that *'trace levels of lead in finished cosmetic products (excluding products used in the oral cavity), should be kept below a target level of less than or equal to 10 ppm total lead, using a lead control system…'*. The report further notes that '*For products that are found to contain trace amounts of lead in quantities greater than 10 ppm, it is recommended that steps be taken by individual companies and/or regulatory authorities, over a reasonable and specified period of time, to lower the total lead content t*o 10 ppm or less…'. Furthermore, *'… once the recommended target for trace levels of lead is met, it should be maintained or improved over time*.''

The second report '*Recommendation for Acceptable Trace Mercury Levels in Cosmetic Products'* (International Cooperation on Cosmetic Regulation, 2016) also considered current regulations in different jurisdictions and exposure to Hg where *'…available analytical data for mercury in cosmetics indicates that the majority of products had mercury levels that were below the limit of detection (LOD), with LOD values in the low ppb range (3 – 28 ppb).'* The report observed that in testing over thousand samples, only two had a mercury content greater than 1 ppm (>1 mg/kg).

The ICCR *'…concluded that mercury levels in cosmetic products should be kept below a target level of less than or equal to 1 ppm mercury, determined as total mercury, in finished cosmetic products using either approach of mercury control system (raw materials or finished products).'*

European Union

The EU Commission have not provided guidance on how to determine traces of prohibited and technically unavoidable substances in cosmetic products to comply with Regulation (EC) No.1223/2009.. Nevertheless, some countries in the EU, notably Germany and the Netherlands, have considered developing national guidance. The Federal Office of Consumer Protection and Food Safety (Bundesamt fur Verbraucherschutz und Lebensmittelsicherheit, BVL) provided details (Bund, 2017) on the development of guidance values to establish technically avoidable trace metals in cosmetic products between 1985 and 1990 to inform manufacturers and authorities. In 2005, the 1985 -1990 values were revised since it was expected that the levels of toxic metals would have declined in cosmetic products and therefore, lower technically unavoidable levels could be achieved through good manufacturing practice. In 2011, a monitoring scheme was established to have a data base to derive current representative orientation values with an adequate sample size within all relevant product categories. A total of 1735 samples from the product groups baby powder, lipstick, lip rouge, lip powder, lip liner, mascara, eyelid line, eye liner, eye shadow, tinted cream, camouflage, rouge as well as theatre, or carnival make-up, children's toothpaste and toothpaste were analysed for lead, cadmium, mercury, arsenic and antimony. The BVL recommended the limit values for heavy metals considered technically avoidable in general cosmetic products but with exceptions for certain heavy metals present in toothpaste and theatre make-up products used around the eye, based on the 90th percentile which are provided in Table 3.

Table 3: German (BVL) Guidance Limit Values for Technically Avoidable Heavy Metals in Cosmetic Products.

Meta	General (mg/kg)	Theatre Make-up (mg/kg)	Toothpaste (mg/kg)
Sb	<0.5		
As	<0.5	<2.5	
Cd	<0.1		
Pb	<2.0	<5.0	<0.5
Hg	<0.1		

The BVL do not give details on analytical methods used in the monitoring exercise. The latest dataset from BVL in 2019 are presented in Section 2.5 in Tables 9a and 9b.

It is noted that there is no similar guidance provided by other countries in Europe and that the German guidance values are often cited when considering guidance values for trace metals.

South East Asia

The Association of South East Asian Nations (ASEAN) established in 1967 represents ten countries, namely Cambodia, Indonesia, Malaysia, Philippines, Singapore, Thailand, Brunei Darussalam, Viet Nam, Lao PDR and Myanmar. The association has produced guidelines (Association of South East Asian Nations, 2019) setting limits for heavy metal impurities in cosmetics which includes As, Cd, Pb and Hg. It is recommended that where analysis using the ASEAN Cosmetic Method is employed as part of the in-market inspection process, the following limits are applied as shown in Table 4.

Table 4: ASEAN Guidance Limits for Heavy Metal Impurities in Cosmetic Products

Metal	ASEAN Guidance Limit Values (mg/kg or mg/L or ppm*)
As	<5
Cd**	<5
Pb	<20
Hg	<1

** The ASEAN guidance limits are expressed in three different forms. Equivalence is presumed for mg/kg, mg/L and ppm but for accurate chemical measurements traceable to the international (SI) unit for mass, i.e. the kilogram, results should be expressed as mg/kg.*

***The Thailand regulatory limit for cadmium is 3 mg/kg and the ASEAN guidance value for cadmium does not apply*

Canada

Guidance on metal impurities in cosmetic products in Canada is available on the Health Canada website (Canada Government, 2012) '*Guidance on Heavy Metal Impurities in Cosmetics*'. The website highlights that although heavy metals are prohibited, they may be present as impurities in cosmetics where the purpose of the guidance '…*is to determine and communicate appropriate limits of these impurities in cosmetic products. The focus is on the heavy metals with known significant toxicological properties: lead, arsenic, cadmium, mercury and antimony.'*

The Canadian authorities indicate they have adopted a similar approach to Germany in establishing heavy metal impurity limits by considering *'impurities in cosmetics should be reduced to the extent that is technically feasible.'* This included a review and analysis of the results from heavy metal testing on cosmetics sold in Canada by the Health Canada Product Safety Laboratory. The guidance states heavy metal impurity concentrations in cosmetic products are seen to be technically avoidable when they exceed the following limits as shown in Table 5.

Table 5: Health Canada Guidance Limits for Metal Impurities in Cosmetics

Metal	Health Canada Guidance Limit Values (ppm)
Sb	>5
As	>3
Cd	>3

It is useful to note there is flexibility in the guidance when heavy metals are found in products above the limits and will be evaluated on a case-by-case basis, with an assessment to determine the level of risk posed by the product, which would then determine the appropriate enforcement action.

Although there is reference to testing by the Health Canada Product Safety Laboratory, no methods to determine metal impurities in cosmetic products have been referenced.

USA

Guidance is provided by the USA Federal Government on metal impurities in cosmetic products on the US Food and Drugs Administration (FDA) website (USA Government, 2022) stating that the regulatory requirement is '*mercury is not allowed in any other cosmetic products except in a trace amount of less than 1 ppm [1 mg/kg] and only if its presence is unavoidable under good manufacturing practice (GMP)'.*

Other legislative requirements concerning trace metals in cosmetic products are in relation to colour additives which must have FDA approval for use and have content limit values for heavy metals. These limits may vary according to the additive but typically are:

- *As: Not more than 3 ppm*
- *Pb: Not more than 20 ppm*
- *Hg: Not more than 1 ppm*

A guidance value for Pb in cosmetic lip products has been set for industry by the FDA described on their webpage (US Food and Drug Administration, 2016) with '*a maximum level of 10 ppm [10 mg/kg] for lead as an impurity in cosmetic lip products and externally applied cosmetics should be readily achievable by manufacturers that source their ingredients appropriately and use good manufacturing practices. Modern analytical capability permits determination of lead at ppm levels, thus enabling manufacturers to avoid the purchase of ingredients with unacceptably high levels of lead and to determine whether lead is introduced into their products during the manufacturing process.'*

2.4. Methods

In reviewing regulations and guidance, there is only one reference to a reference method to determine metal content and this is found in the ASEAN Guidelines (Association South East Asian Nations, 2007). Where guidance values for total heavy metal content are made without specifying the test method, it is likely that there will be differences between test laboratories in reporting results. To avoid such circumstances, there is development of interlaboratory validated methods or use of standard procedures (e.g., ISO, ASTM, AOAC) to demonstrate accuracy, repeatability and robustness but it is likely laboratories have developed their own methods based on various studies and publications. Furthermore, a lack of appropriate matrix matched reference materials adds to this challenge.

For the determination of metal content in cosmetic products, this review has considered methods described by authorities in setting guidance limits, international organisations involved in metrology and industry partners. Other procedures with corresponding results in published papers are reviewed in Section 3.4.

International Cooperation on Cosmetics Regulation (ICCR)

The ICCR report ‘Recommendation for Acceptable Trace Mercury Levels in Cosmetic Products’ describes a number of options based on digestion of the sample using acid mixtures with Inductively Coupled Plasma (ICP) techniques used for the detection of mercury as well as other metals at the same time. Alternatively, for greater sensitivity, common methods for mercury detection are cold vapor atomic absorption spectrometry (CVAAS) and cold vapor atomic fluorescence spectrometry (CVAFS). In these systems, mercury (Hg^{2+}) is converted into elemental mercury vapor (Hg^0) which is introduced into an absorption cell or a fluorescence cell. These systems allow detection of mercury at very low levels from 0.1 to 1 part per trillion (ppt), depending on the instrument. The ICCR advise mercury must be analysed with caution, considering volatility and stability behaviour. Contamination of the samples by other sources of mercury as well as losses during the preparation of samples must be avoided. An alternative technique to acid digestion is direct determination by thermal decomposition using specific atomic absorption instruments which are equipped with a gold amalgamator that allows the detection of mercury after desorption of the element. These systems are less sensitive than CVAFS systems (with detection limits at the ppb level) but have the advantage of the ability to directly analyse the samples without sample preparation.

For other heavy metals, the ICCR have made no recommendations concerning suitable methods except to note Pb *‘is determined as total lead in the product based on scientifically sound method(s) leading to validated results.*’

South East Asian Nations (ASEAN)

The ASEAN guidance specifies the ASEAN test method is used for determining heavy metal content. The method described three extraction techniques: (1) microwave digestion for As, Cd, Pb and Hg using nitric acid/hydrogen peroxide (HNO_3/H_2O_2) mixture; (2) dry ashing using magnesium nitrate ($MgNO_3$) in a muffle oven at 500ºC and dissolution into hydrochloric (HCl) acid for As, Cd and Pb; and (3) wet digestion for determining Hg using HNO_3 acid in a digestion tube with a screw-cap. The determination of arsenic, cadmium and lead utilised graphite furnace atomic absorption spectrometry (GF-AAS), whereas for mercury, the flow injection analysis system-atomic absorption spectrometry (FIAS-AAS) with cold vapour technique was employed. The reported accuracy for arsenic, cadmium, lead, and mercury from spiked cosmetic creams were 84-86%, 66-71%, 85-99% and 95-108% respectively with the following ‘determination limits’: As <2.5 µg/g, Cd <1 µg/g, Pb <10 µg/g and Hg <0.5 µg/g Hg.

EU Joint Research Centre (JRC)

The BVL paper (Bund, 2017) recommending limit values for heavy metals does not describe the methods used in the surveys to determine the limit values but does reference the Joint Research Centre (JRC) report EUR 24886 EN (European Union, 2011), concerning the results of a European survey on determining lead in lipsticks which in turn references JRC report EUR 25838 EN (European Union, 2013). This suggested the method used in the survey for lead in lipsticks was based on microwave-assisted digestion with HNO_3 and hydrofluoric acid (HF) acid with analysis of lead by inductively coupled plasma-mass spectrometry (ICP-MS).

US Food and Drugs Administration (FDA)

The FDA has based its guidance for lead after surveying the market using a validated method (Hepp, et al., 2009), detailing the determination of lead content in 20 lipsticks from the USA market. This survey was later expanded to 400 lipsticks in 2010. Both studies measured total Pb content by using microwave assisted digestion with HNO_3/HF, neutralising with boric (H_3BO_3) acid and analysis by ICP-MS. The first survey found lead levels ranging from 0.09 to 3.06 mg/kg with a detection limit estimated at 0.04 mg/kg observing Pb in certain lipstick samples appeared to be incorporated in the refractory mineral pigments, which required HF acid for complete digestion.

In a second series of surveys (Hepp, et al., 2014), the total content for metals As, Co, Pb, and Hg as well as calcium (Ca) and chromium (Cr), were determined in 150 different types of cosmetic products. Although the FDA webpage reported that '*a more common extraction method with chemicals that are not as strong as hydrofluoric acid and are easier to handle*' was used it appears that a further modification of the digestion procedure was necessary. Consequently, a new extraction procedure was introduced whereby samples were digested in closed vessels using 3:1 HNO_3/HF acid mixture with HCl acid and placed in an oven at 130ºC for 12 hours. The digestion solution was then cooled and diluted with water. Nitric acid was added followed by repeated evaporation (except for mercury analyses) on a hot plate with additions of HNO_3 acid until dissolution of the sample. The metals were determined using ICP-MS except for mercury where gold amalgamation CVAFS was employed.

The main difference between the surveys was the change in the digestion procedure in using a conventional oven rather than using microwave digester rather than omitting hydrofluoric acid.

The limit of detection (LOD) was determined on ten replicate analysis of the reagent blank for each element with the LOD calculated as three times the standard deviation of the results. The limit of quantification (LOQ) was determined on ten replicate analysis of the reagent blank spiked with each element at a low concentration where LOQ is calculated as ten times the standard deviation of the results. The reported values are shown in Table 6.

Table 6: LOD and LOQ Values for Determining Metals in Cosmetic Products Using Microwave Acid Digestion and ICP-MS.

Metal	LOD (mg/kg)	LOQ (mg/kg)
As	0.048	0.16
Cd	0.018	0.058
Pb	0.0084	0.028
Hg	0.0010	0.0032

International Standardisation Organisation (ISO)

In the absence of official methods, laboratories will use published methods in standards produced by ISO or the European Standards Organisation CEN. There are only two standards found in the literature concerning determining metals in cosmetic products produced by ISO.

- ISO/TR 17276:2014 is only a guidance document titled '*Cosmetics — Analytical approach for screening and quantification methods for heavy metals in cosmetics*.' (International Standards Organisation, 2014)

- ISO 21392:2021 *Cosmetics — Analytical methods — Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique*. (International Standards Organisation, 2021)

ISO also have two standards in development for the determination of mercury in cosmetics but are not yet available, and therefore not reviewed.

- ISO/DIS 23821. *Cosmetics — Analytical methods — Determination of traces of mercury in cosmetics by atomic absorption spectrometry (AAS) cold vapour technology after pressure digestion*.

- ISO/DIS 2367. *Cosmetics — Analytical methods – Direct determination of traces of mercury in cosmetics by thermal decomposition — Atomic absorption spectrometry (mercury analyser)*.

Technical report ISO/TR 17276:2014 described the typical analytical approaches for screening and quantification of heavy metals in the individual ingredients as well as in the final cosmetic product. This ranged from techniques using traditional colorimetric reactions, which can be performed using common laboratory instruments, to more sophisticated techniques such ICP-MS which allows detection of elements at the μg/kg level but requires more skilled analysts and is more costly to purchase and operate. Technical report ISO/TR 17276:2014 does not go further than describing different techniques including the preparation of samples but does give a view of advantages of each of the following techniques:

- Colorimetric reaction
- X-ray fluorescence spectrometry (XRF)
- Atomic absorption spectrometry (AAS)
- Inductively coupled plasma (ICP)

It was noted in the Technical Report that ICP coupled to an optical emission spectrometer (ICP-OES) or a mass spectrometer (ICP-MS) have advantages such as multi-element capability for fast sample analysis but requires metals to be extracted into solution. Where samples cannot be extracted, direct determination of the metal content by GF-AAS is considered an alternative option but this involves single rather than multi-elements per analysis.

Standard ISO 21392:2021 is the only recognised method by either ISO or CEN for quantifying trace levels of chromium, cobalt, nickel, arsenic, cadmium, antimony and lead in cosmetic products using ICP-MS. The metals are extracted into solution by microwave-assisted acid digestion with mineral acids (HNO_3/HCl acid mixture) in sealed vessels heated to 200°C, followed by quantification by ICP-MS. The method highlights some limitations, as it is possible some inorganic ingredients, such as silica or titanium dioxide, are not completely digested under these conditions and therefore the heavy metal might not be fully extracted. However, the level of heavy metals trapped within these fractions was not considered to significantly contribute to the exposure level of consumers.

The method described in standard ISO 21392:2021 has been subject to an inter-collaborative trial using 10 laboratories and evaluation using standard ISO 5725-2, Accuracy (trueness and precision) of measurement methods and results — Part 2: Basic method for the determination of repeatability and reproducibility of a standard measurement method. It was noted for the determination of lead in 7 different cosmetic samples the relative repeatability ranged between 7.5 – 15.5% and relative reproducibility data ranged between 15 – 80%.

Manufacturers Application Notes

Application notes for determining heavy metals in cosmetic products produced by instrument manufacturers were reviewed.

Perkin-Elmer provided an application note (Perkin Elmer, 2011-12) evaluating the heavy metal content in lipstick, nail polish and skin cream by dissolving the sample into HNO_3 acid and HF acid mixture using microwave-assisted digestion and quantifying the metals by ICP-MS. It was remarked that cosmetics contain a variety of components that can be challenging to digest, including fats and silica-based compounds, which may require the addition of hydrofluoric acid. In the absence of certified reference materials for cosmetic products, a food-based material was used (NIST SRM 1548a Typical Diet). Recoveries of the certified values were within ± 20%, considered an acceptable performance. Estimated detection levels for the metals in a solid cosmetic product are shown in Table 7.

Table 7: Estimated Detection Levels for Determining Metals in Cosmetic Products Using Microwave Acid Digestion and ICP-MS.

Metal	Detection Levels (mg/kg)
As	0.014
Cd	0.0069
Pb	0.0096
Hg	0.037

Agilent provide an application note (Agilent Technologies Inc, 2019) describing a method for accurate measurement of trace quantities of Hg in cosmetics using ICP-MS/MS. The presence of other elements can cause interferences in ICP-MS and for mercury, tungsten can cause an interference which could lead to a positively biased result for Hg. The method described 100-fold dilution of a cosmetic lotion (containing high levels of tungsten) which was shaken to homogenise, followed by direct injection into the ICP-MS/MS. The ICP-MS/MS was operated in with oxygen reaction cell gas mode to resolve the interference from tungsten, enabling accurate of trace levels of mercury.

2.5. Data from publications

There are several papers published on the determination of metals in cosmetic products including surveys on samples taken from the local market where methods and data have been highlighted and are presented below in chronological order.

In 1980, the Dutch government agency Keuringsdienst van Waren (KvW), produced a report (Keuringsdienst Van Waren Voor Het Gebied Enschede, May 1980) proposing limits for heavy metal impurities considered technically unavoidable in cosmetic products to meet the EU regulations using a survey to establish guidance values. The method for determining metals content in the cosmetic product involved dissolving the sample in concentrated HNO_3 acid at 140ºC or dry-ashing in a muffle oven at 500ºC followed by dissolution into acid. The metals were determined using flame atomic absorption spectrometry (FAAS) except for mercury where CVAAS was used. The authors identified difficulties in digesting some matrices containing inorganic materials such as silicates or sulfates in eye make-up, talc and face powder. KvW suggested the following maximum values for contamination of cosmetic products by heavy metals, as shown in Table 8.

Table 8: KvW Maximum Values for Heavy Metal Contamination of Cosmetic Products.

Metal	KvW Guidance Limit Value (mg/kg)
Sb	<5
As	<5
Cd	<5
Pb	<20
Hg	<1

As previously highlighted JRC report EUR 24886 EN provided results of a European survey on Pb content in lipsticks. The survey analysed 113 samples, bought on the European market between 2010-2011. The method used for the analysis was in-house validated by the US FDA. It was based on the microwave-assisted acid digestion of lipsticks followed by ICP-MS determination. In total, 81 lipsticks and 32 lip glosses representing 34 different brands were purchased in 12 EU Member States. Lip glosses showed generally lower levels of Pb than in lipsticks. Only one lip gloss out of 32 (3%) exceeded a value of 1 mg/kg, whereas 30 lipsticks out of 81 (37%) were found to be above 1 mg/kg. The sample with the highest content (3.75 mg/kg) was a red lipstick.

The report concluded '*a lead content in the range of 1 to 2 mg/kg represents the norm rather than the exception for manufacturers present on the EU market'*.

Similarly, the JRC report EUR 25838 EN described a collaborative trial within the International Measurement Evaluation Programme (IMEP) to assess the performance of methods and laboratories in determining metal content in cosmetic products. The interlaboratory comparison involved 17 laboratories analysing a single sample composed of a blend of commercially available lipsticks with an assigned value for lead content. It was reported that good laboratory performance appeared to be correlated to the following parameters:

- Appropriate amount of the test sample taken for analysis (test portion). Very low-test portions were, generally, leading to poorer performance
- Appropriate acid mixture (HNO_3/HF) for sample digestion
- Use of microwave digestion

Other studies on the determination metals in cosmetic products have been reported from around the world.

In India, a report (Centre for Science and Environment New Dehli, India, 2014) provided results for analysing heavy metal content including Cd, Pb and Hg in 73 cosmetic products comprising of lipsticks, skin lightening creams, lip balm and anti-ageing cream obtained from the local markets in New Delhi, India. Two different extraction methods were used. For the determination of Pb and Cd content in lipstick, lip-balm and anti-ageing cream, the samples were wet digested using 4:1 nitric/perchloric (HNO_3/$HClO_4$) acid mixture and heated on a hotplate at 90ºC for 2-3 hours. The process was repeated with an additional amount of the acid mixture to complete digestion followed by cooling and dilution with water after filtration. The filtered extract was then analysed using FAAS. For skin lightening creams, only Hg was determined, where samples were pre-digested in a 1:1 sulfuric/nitric (H_2SO_4/HNO_3) acid mixture at 80ºC for 1½ hours. The sample extract was allowed to cool then 5% potassium permanganate ($KMnO_4$) solution and 3% HCl were added and heated to 95ºC for 2 hours and allowed to cool. A 12% hydroxylamine (H_3NO) solution was added to neutralise unreacted $KMnO_4$. The extract was made up to a known volume with 3% HCl. The sample extract was determined for mercury content using CVAAS. The results were grouped into three categories (1) lipsticks, (2) lip-balm and anti-ageing cream and (3) skin lightening creams. No Pb or Cd content was detected in any lipsticks, lip-balms, or anti-ageing creams. In 14 of the 32 skin lightening creams, Hg was detected in the range 0.1 mg/kg to 1.97 mg/kg.

In Saudi Arabia, a paper (Salama, 2015) reported on the analysis of heavy metals including Cd and Pb in 35 cosmetic face creams taken from the market. The method applied a wet digestion procedure using a 4:1 HNO_3/HCl acid mixture and heating on a hotplate to complete digestion followed by cooling and dilution with water. The solutions were analysed using ICP-OES. The samples contained Pb in the range 0.1 to 7.0 mg/kg and Cd was 0.1 to 1.3 mg/kg but only 3 results were above the detection limit for cadmium. The paper considered the impact of pricing of products considering lower priced cosmetics are more likely to contain heavy metals, especially lead. The authors concluded '*All cosmetics contain lead below 4 ppm, which is consistent with the guidelines of SFDA, USFDA, and Canada. However, only one high and few low-quality products exceeded the German limit (2 ppm)'.*

In the Middle East (Massadeh, et al., 2017), Cd and Pb content was determined in selected cosmetics taken from Jordanian, Sudanese and Syrian markets. The selected cosmetics included eyeliner, eye pencil, mascara, lipsticks, face cream, body cream, sun block, Vaseline and kohl. The digestion procedure used a 3:2 HNO_3/HCl mixture and heating at 70ºC on a hotplate to complete digestion followed by cooling and dilution with water. The solutions were analysed using FAAS. The heavy metal content in samples collected from Jordan were in the range for Pb of 0.30-15.4 mg/kg and 0.03 to 0.10 mg/kg for Cd. For samples collected in Sudan, the Pb content was 0.02-3.8 mg/kg, and the Cd content was 0.01 to 0.15 mg/kg. Finally, for the Syria samples, the Pb was found at 4.85-27.70 mg/kg and the Cd levels were 0.04-0.056 mg/kg. In the conclusion, it was remarked *'It is not possible to completely eliminate the presence of heavy metals from cosmetic products after manufacturing. However, the quality of the products can be improved by careful selection of raw materials, taking in consideration heavy metal levels. (ii) Heavy metal concentration in lipstick differs with different manufacturers' colors and shade. Statistically significant associations between Pb level and the cosmetic type were found.*

In Pakistan, a comparative study (Hussain, et al., 2017) of heavy metal content was made including lead and cadmium, on cosmetics taken from local markets. The study analysed 15 different materials including shampoo, talcum powder, lipsticks, surma (kohl eye makeup) and cream. Two different extraction methods were used, where dry-ashing was applied for most of the samples and wet digestion for the remaining creams and lotions not suitable for dry ashing. The procedure for dry ashing involved heating the sample at 105ºC in an oven until dry and then ashing at 550ºC in a muffle oven for a few hours. The residue then dissolved into 1 molar (M) HNO_3 acid, evaporated on a hot plate and the residue reconstituted in water. The wet digestion technique used a 4:1 $HNO_3/HClO_4$ mixture and heating on a hotplate to near dryness for 2-3 hours followed by cooling and dilution with water. The solutions were measured using ICP-MS. The study showed particularly high Pb content for the surma/kohl where the average content reported for 3 samples was 692.90 ± 0.02 mg/kg.

In Bangladesh, a paper (Alam, et al., 2019) on an assessment of heavy metals in commonly used cosmetics reported on six different brands of beauty creams. The method for sample preparation involved wet digestion using 3:1 $HNO_3/HClO_4$ mixture and heating for 2-3 hours at 90ºC on a hotplate. The measurement of cadmium and lead was performed by FAAS and CV-AAS was employed for mercury. The concentrations found in beauty creams were in the range 14.38-50.39 mg/kg for Pb, in the range 2.77-6.27 mg/kg for Cd and in the range 0.17-0.48 mg/kg for Hg.

Germany regularly monitors the cosmetics market to assess manufacturers observance with the 2011 BVL guidance values. Most recently, results on metals content for 290 lipsticks with and without glitter were reported (BVL-Report, 2019) comparing cosmetics to the BVL guidance values for avoidable trace levels including As, Cd, Pb, and Hg. In the report it was noted that a quarter of the cosmetics sampled orginated from Germany, a quarter from other EU countries, a quarter from third countries and a quarter of unknown origin. The authors reported that 98% of lipsticks without glitter and 92% of lipsticks with glitter had detectable values for As, Cd, Pb, and Hg content below the BVL guidance values. Where samples were shown to exceed limit vales, these concerned Sb and Hg in the lipsticks containing glitter. There was no reference to the method used but it is presumed the same methodology was used as reported in the original paper (Bund, 2017). The values for heavy metals detected in lipsticks including lipsticks containing glitter are shown in Table 9a.

The monitoring report (BVL-Report, 2019) had concluded that the results were comparable to the original survey on lipsticks (Bund, 2017).

Table 9: BVL Survey of Heavy Metals in Lipsticks With (9a) and Without (9b) Glitter.

9a

Metal	No. of samples analysed	No. of samples with quantifiable values	Average (mg/kg)	Median (mg/kg)	Max (mg/kg)	Threshold limit guidance values (mg/kg)	No. of samples exceeding guidance values
Sb	139	11	0.096	0.050	0.410	0.5	0
As	135	37	0.124	0.063	0.507	0.5	**1**
Cd	168	6	0.022	0.025	0.030	0.1	0
Pb	169	59	0.401	0.250	6.130	2.0	**3**
Hg	152	13	0.028	0.013	0.428	0.1	**2**

9b

Metal	No. of samples analysed	No. of samples with quantifiable values	Average (mg/kg)	Median (mg/kg)	Max (mg/kg)	Threshold limit guidance values (mg/kg)	No. of samples exceeding guidance values
Sb	103	14	0.587	0.125	24.2	0.5	**8**
As	100	22	0.070	0.019	0.997	0.5	**1**
Cd	103	3	0.013	0.005	0.014	0.1	0
Pb	103	51	0.330	0.190	7.610	2.0	**1**
Hg	108	23	0.033	0.033	0.431	0.1	**6**

A proficiency testing study (Institute for Interlaboratory Studies, November 2019) reported on determining trace metals in mouthwash and toothpaste. The samples were spiked with known amounts of the target metals: Cd, Pb and Hg. There were ten participants but only 8 laboratories submitted results, with seven laboratories employing ICP-MS and one used ICP-OES for lead and cadmium and AFS for mercury. The evaluation of the results concluded that determination of the metals in mouthwash and toothpaste were non-problematic. The uncertainties in the test results were favourably calculated as 8-9% for Cd, 9-10% for Pb and 10-14% for Hg when compared to the target values based on Horwitz values of 12% for Cd at 5 mg/kg, 10% for Pb at 20 mg/kg and 14-16% for Hg at 1-2 mg/kg.

2.6. Discussion

In reviewing threshold limits for unavoidable impurities of heavy metals, there appears to be only a few examples of countries setting minimum limit values into legislation as shown in Table 10.

Table 10: Countries Setting Minimum Content Limit Values for Heavy Metals in Legislation

Country	Cosmetic Type	Sb (mg/kg)	As (mg/kg)	Cd (mg/kg)	Pb (mg/kg)	Hg (mg/kg)
USA	General					≤1
USA	Eye					≤65*
China	General		≤2	≤5	≤10	≤1
Thailand	General			≤3		

** When Hg is used as a preservative in cosmetics used in the eye area.*

The basis of the threshold limit values chosen by countries is unclear particularly in respect to selection of the metals and whether the values have been established through studies on the country's cosmetics market, technological capability to measure at certain concentrations or derived from evaluation of toxicological data based on consumer exposure. Some of the limit values have been in place for many years and have not been reviewed to suggest that decisions to have a threshold limit for certain heavy metals were made to address a particular problem at the time requiring regulatory action.

More often countries have chosen to set a conditional requirement in legislation that identifies metals which are prohibited in cosmetic products unless unavoidable and cannot be removed during manufacture unless the metals present are considered technically unavoidable in the cosmetic product and not a danger to the consumer's health.

In setting a conditional requirement in legislation that prohibits specific metals but recognising these metals may be present as impurities does create uncertainty both for the manufacturer and the enforcement agency. It must be determined at what concentration the presence of metals can be considered technically unavoidable reflecting good manufacturing practice, as well as ensuring the concentration of the impurity is not a health risk. To address this situation, some countries have taken the approach to issue guidance which, in many cases, is stated as non-binding, although usually available on official government websites or in published papers to inform manufacturers, suppliers and enforcement agencies. Establishing the minimum content values for certain heavy metals was largely based on analysis of cosmetic products supplied in the country's market to ascertain what can be regarded as good manufacturing practice. However, whether these guidance limit values are considered toxicologically acceptable has not been elaborated in many cases.

An overview of guidance provided by different countries or associations on the content limits for certain heavy metals is given in Table 11.

Table 11: Guidance Values for Heavy Metal Content in Cosmetic Products.

Authority	Cosmetic Type	Sb (mg/kg)	As (mg/kg)	Cd (mg/kg)	Pb (mg/kg)	Hg (mg/kg)
ICCR	General				≤10	≤1
BVL (Germany)	General	<0.5	<0.5	<0.1	<2.0	<0.1
BVL (Germany)	Toothpaste				<0.5	
BVL (Germany)	Theatre Make-up		<2.5		<5.0	
ASEAN	General		<5	<5	<20	<1
Health Canada	General	≤5	≤3	≤3	≤10	≤1
FDA (USA)	General including lip products				≤10	

The range of guidance values for heavy metal shown in Table 11 vary across authorities and within cosmetic types. It is apparent that guidance values indicated by BVL are consistently lower for all the specified heavy metals in cosmetic products. BVL has based the threshold values on the results of metals content within the 90th percentile of products analysed in surveys of the German market and subsequently compared in monitoring exercises, where greater than 90% of cosmetic products tested are below the guidance threshold limits. This may suggest the BVL guidance limits are the optimum levels that most manufacturers supplying the European market can be considered to reasonably achieve.

That said, it is noticeable that other countries in North America and Asia have set higher guidance limits that are several orders of magnitude higher than the BVL guidance limits. Given these other countries have also based their guidance values on surveys of cosmetic products placed on their markets, their values will be considered equally valid being reflective of the market. However, it is recognised in international forums such as the ICCR, that it is desirable to try and set ever lower threshold guidance values where-ever possible and the BVL guidance values for cosmetic products manufactured or supplied to the German market suggests this is possible.

Comparison of guidance values in Table 11 for the same heavy metal content in different types of cosmetic product raises the question how far consideration of good manufacturing practice should apply to different materials and manufacturing processes used for different cosmetic products. One approach is to set a single maximum content value for each metal based on all the cosmetic products analysed in a survey. Although reflecting the whole market and likely to see a higher overall guidance value being set for the maximum metal content. This provides simplicity in approach and is seen in the guidance values provided by Health Canada. Alternatively, and this has been applied by BVL is to have different guidance limits for different products recognising the challenges some manufacturers will have in processing materials and removing contaminants as well as considering the different exposure risks between ingestion and dermal absorption.

BVL in drawing up guidance has made distinctions for different cosmetic product types where there are three guidance values for Pb namely general, theatre/carnival make-up and toothpaste. The Pb limit value is <2.0 mg/kg for general cosmetics but there is a lower threshold limit of <0.5 mg/kg in toothpaste and a higher value of <5.0 mg/kg in theatre or carnival make-up. Whilst it may be considered that toothpaste present a greater risk, the setting of limit values reflects the market survey. Consequently, it is likely that toothpaste manufacturers will have greater quality control and processes, given the use of the product, such that Pb levels in toothpaste will be lower than that found in general cosmetics. Conversely theatre or carnival make-up is highly coloured and therefore likely to use more pigments leading to higher levels of Pb in these types of cosmetics from the impurities. Consequently, care is needed when considering results to decide whether different types of cosmetics need separate guidance values for the same metal.

Another issue in conducting surveys is ensuring results are accurate and reproducible to establish the correct guidance limit values. In reviewing data generated by different organisations, it is apparent that although similar approaches using acid digestion were taken, these differ in their extraction ability and that cosmetics contain a wide variety of materials that are difficult to dissolve into solution. If metals are not fully solubilised and a standard procedure is not applied, it could lead to variable results where one laboratory use a more efficient extraction method than another, such that an incorrect decision could be made regarding whether a product may pass or fail the threshold guidance limit value. It can also provide difficulty when comparing results from different studies.

In reviewing analytical methods used to determine limit or guidance values, there is broadly a similar approach in using acid digestion to extract the metal from the cosmetic product and detection using spectrometry. The advantages of different detection systems are described in *ISO/TR 17276:2014 – Analytical approach for screening and quantification methods for heavy metals in cosmetics'*, with ICP-OES and ICP-MS offering multi-element capability, good linear dynamic range and sensitivity. This is not to dismiss other techniques such as GF-AAS and AFS, but the modern analytical laboratory is more likely to have ICP-based capabilities. A more contentious issue is how to extract metals from the cosmetic samples.

In the literature review, a variety of acids and procedures were used, including digestion with HNO_3, HNO_3/HF or a HNO_3/HCl/HF mixture followed by heating using either a microwave system, oven or hot plate. Additionally, in some studies, dry-ashing was applied at high temperatures to remove the organic matrix followed by redissolving the residues into dilute acids. This carries the risk of loss of the analytes and insoluble residues. The only published standard describing a method for determining metals in cosmetic products is *ISO 21392:2021 – Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique*.

In the standard ISO 21392:2021, samples are digested using HNO_3 and HCl in a sealed vessel and heated to 200ºC with microwave energy. Even under these extreme conditions, it was reported that cloudy solutions or precipitate may remain in the solutions. Some cosmetic inorganic ingredients, such as silicon dioxide (SiO_2) or titanium dioxide (TiO_2), do not completely dissolve without the use of HF. This approach was identified in papers and application notes for the determination of lead in samples such as lipsticks. This suggests where inorganic ingredients, such as SiO_2 or TiO_2 are indicated, HF should be used to ensure accurate concentrations are obtained for the heavy metals. It is recognised that use of HF can present corrosive and health risks, requiring expert use but should be feasible for most competent laboratories. It is noted that the use of perchloric acid ($HClO_4$) was described in some papers as an extraction acid, but this should be avoided due to its reactive and potential explosive nature.

In reviewing information in published papers, it has been helpful to see what extraction conditions and instrumental techniques were used. However, it is difficult to compare whether the different laboratories and methods were efficient/accurate in determining the total metal content without the use of reference materials or measures of performance through proficiency testing schemes. Therefore, it is important to consider the comments from the JRC, who conducted an interlaboratory trial analysing lead in lipsticks, that good laboratory performance correlated with an appropriate sample mass and utilising HNO_3/HF with microwave-assisted digestion.

It was noted there was minimal reporting on the determination of Sb content in cosmetic products. This may suggest technical reasons such as there are few Sb impurities present in cosmetic ingredients or the methods used to determine Sb may be insufficiently sensitive or problematical. Furthermore, there is significantly less toxicological data available for Sb compared with elements such as lead or mercury.

2.7. Summary

To undertake a monitoring exercise of toxic elements in cosmetic products requires robust validated method or methods to be able to determine quantitatively all the target metals in different cosmetic matrices. It is also important in establishing that the robust validated method(s) is fit for purpose and can be applied by both public analyst and industry laboratories to enable comparison to the guidance limits developed from the monitoring exercise in order to demonstrate compliance.

Standard *EN ISO 21392:2021 Cosmetics — Analytical methods — Measurement of traces of heavy metals in cosmetic finished products using ICP/MS technique,* provides a starting point but as it was only recently published on the 10th of September 2021 and requires further evaluation. Significantly, the scope of *BS EN ISO 21392:2021* explains the method uses HNO_3/HCl mixture to extract trace levels of heavy metal into solution for measurement. It is possible that some cosmetic inorganic ingredients, such as SiO_2 or TiO_2, are not completely digested under these conditions and that heavy metal in such ingredients are not fully extracted.

Many of the cosmetic samples that have the greatest risk will be those intended to be placed on the face around the mouth and eyes where ingestion or migration through mucous membranes may occur. Complete dissolution of the cosmetic product is required to determine the accurate and reproducible measurement of metals present in the sample matrix. This requirement has been identified in publications where analysis of cosmetic products have taken place, for the determination of lead in lipstick, as earlier reported by the JRC and the FDA. In both cases, it was recognised that the use of hydrofluoric acid is necessary to completely dissolve the sample to fully extract the metals.

Where there is absence of mineral ingredients, such as silica, indicated in the cosmetic product, complete digestions should be achieved with the HNO_3/HCl as described by ISO 21392:2021 which would be beneficial for laboratories by using a standard procedure with corresponding validation data. For those cosmetic products containing such ingredients, it is probable that the use of HF acid will be necessary to ensure complete dissolution. This means two methods specifying the use of HNO_3/HCl mixture with and without HF may be required by a laboratory. However, it may be more effective to implement the use of HNO_3/HCl mixture with HF to ensure complete dissolution and increase efficiency of the process.

An important consideration is the selection of an appropriate method with suitable LODs and LOQs such that any competent laboratory can determine the metals content accurately and reproducibly in cosmetic products at the relevant concentration levels. This is necessary to demonstrate conformance with the legislative or guidance limit thresholds, whilst also considering the precision or uncertainty of the method.

In addition to the measurement of metal impurities, it is important that correct sampling procedures are followed as dispersion/separation of pigments in waxes can occur and therefore any impurities may be unevenly distributed throughout the sample. As shown in the German monitoring scheme, the scale of testing is potentially immense and therefore consideration should be made to limiting this monitoring to cosmetic samples presenting the greatest risk, such as toothpaste and lipsticks.

A validated method encompassing these points is important to ensure the success of the monitoring exercise.

3. Experimental

3.1. Sample Selection and Information

Guidance for sample selection was provided by OPSS and designed to give a snapshot of the current UK environment. It covers low, mid and high price products, a mix of high street and online retailers (~70/30 spilt) and a range of colours/shades. A full list of the samples selected is provided in Appendix 1, covering the following product groups:

- Lipstick
- Lip gloss
- Lip liner
- Toothpaste and whitening toothpaste
- Mouthwash
- Sun protection creams
- Makeup powders
- Foundation liquid/creams
- Mascara
- Eye liner
- Eye shadow

3.2. Method Development

Method development focussed on homogenising sample types that could not be mixed easily where lipsticks, lip liner and eye liner proved to be more challenging.

Standard ISO 21392:2021 warns the user that samples having high fat and oil content need to be treated carefully to avoid potentially extreme reactions or explosions during microwave heating as well as to ensure samples are fully immersed in solution to avoid overheating and damage to the digestion vessel. Attempts to suspend the sample in water as suggested proved not successful and it was decided that as the method planned to only use 0.1 g of sample, the volume of acids being used is sufficient to address safety concerns.

Lip and eye liners sampled were in various formats and each were homogenised differently. Pencil liners were split open with stainless steel scalpels and contents chopped. Gel liners were scraped out and liquid liners decanted into secondary containers. For the lip liners, an additional stage using dry ice (solid CO_2) was required to assist with the homogenisation.

Initial microwave digestion was performed using the acid combination and digestion parameters for each product group as described in standard ISO 21392:2021. The short method was used for the majority of sample groups tested with the exception of lipsticks, lip gloss and lip liner. Where a clear and particulate-free solution was not obtained, the volumes of acids were adjusted, the long microwave programme was used and HF was added to the mix to improve the digestion. However, despite this, many samples did not provide a clear, particulate-free digestion, therefore these samples were centrifuged prior to analysis.

As highlighted in the literature review, there is a lack of suitable matrix matched certified reference materials (CRMs) covering cosmetic products. Therefore, for this work alternative products were explored. A lipstick and lip gloss material from a previous proficiency testing scheme (LGC Standards, UK) were sourced, utilising the consensus values as reference values. Additionally, the CRM NIST SRM 2709a San Joaquin Soil was selected as it contains high levels of refractory elements such SiO_2 and TiO_2.

3.3. Method Verification

Method verification was performed using the developed method which resulted in the clearest digestion for each sample group. For each sample group, 8 reagent blanks and 1 sample prepared in duplicate were prepared alongside triplicate preparations of the sample spiked at a known concentration. A quality control (QC) material (lipstick and lip gloss from the proficiency testing scheme) or NIST SRM 2709a were also prepared in the same manner. For the mouthwash samples, neither the QC materials nor CRM were considered appropriate therefore the triplicate sample spikes were deemed adequate. Additionally, an internal quality control standard (IQC) was prepared from an independent source of calibration standards.

The verification exercise was considered successful if these criteria passed

- Calibration linearity was >0.99
- Internal standard recovery was within 75-125%
- IQC standard recovery was with 80-120%
- The recovery of the triplicate spiked samples was within 70-150%
- The QC material or CRM was within 75-125% (excluding mouthwash)
- The duplicate sample analysis was within 20% RSD

For sample matrices, the method verification was successful.

3.4. Sample Preparation

Following the method development and verification stages, the samples were prepared accordingly. The materials were prioritised by the risk rating. Due to time and budgetary constraints, it was not possible to complete the sample analysis for the makeup powders, but the matrix was successfully verified.

3.4.1. Lipstick

Remove from device into a suitable plastic container. Heat at 85ºC for approximately 1 hour to homogenise prior to weighing.

3.4.2. Lip Gloss

Transfer contents of Lip Gloss to a suitable plastic container and stir to mix.

3.4.3. Lip Liner

With a pencil style lip liner, place in a clamp and cut the pencil open using a clean scalpel. Remove the inner contents using a plastic spatula and place in a clean plastic bag, seal the bag and place in dry ice for approximately 1hr 30mins. Remove the bag from the dry ice and use a pestle to crush the sample, then flex the bag to break the pencil into fine powder. If the lip liner is in a plastic applicator twist the applicator to fully unwind and cut out the inner and place in a clean plastic bag and continue as with the pencil style.

3.4.4. Toothpaste, Mouthwash, Sun Protection, Liquid Foundation

Mix and transfer sufficient quantity of toothpaste to a suitable plastic container.

3.4.5. Mascara

Where possible, remove brush and disassemble tube by removing the top part using clean sharp tweezers. If required, a pipe cutter may be used to open the tube instead. Transfer contents to a secondary container with a plastic spatula.

3.4.6. Face Powders, Eye Powders

Face powders (blusher, foundations) should be knocked out of the container into a plastic bag and then crushed using a pestle and mortar prior to transferring to a secondary container.

3.4.7. Eye Liners

With a liquid eyeliner where possible, remove applicator and disassemble bottle by removing the top part using clean sharp tweezers. Transfer contents to a secondary container with a plastic spatula. With a pencil style eye liner, place in a clamp and cut the pencil open using a clean scalpel. Remove the inner contents using a plastic spatula and place in a secondary container. For the eye liner twist the applicator to fully unwind cutting off the inner plastic applicator placing it in a secondary container. As samples are provided in different forms and packaging there is a need for the analyst to apply judgement in preparing samples.

3.4.8. Sample Digestion

The samples were digested in the Multiwave Go (Anton Paar, UK) using Teflon vessels, except for the mouthwash where the Discover SP-D (CEM Corporation, UK) with quartz vessels was also used. Acids were of trace element grade quality (SpA, Romil, UK). Following the method development stage, it was found that different acid mixes were required depending on the product type and two microwave methods were necessary. Within in each sample batch, either the QC materials or NIST SRM 2709a were included following the same procedure, except for the mouthwash where triplicate spiked samples were performed. Additionally, procedural blanks were prepared. The digestion parameters for each sample type are provided in Tables 12 and 13.

Table 12: Instrument Parameters for the Microwave

Short Method Digestion Parameters	
Time (mins)	**Temperature Programme**
0 – 30	Ramp to 200°C
30 – 60	Hold at 200°C
60+	Cool to vessel release
Long Method Digestion Parameters	
Time (mins)	**Temperature Programme**
0 – 25	Ramp to 160°C
25 – 40	Hold at 160°C
40 – 50	Ramp to 180°C
50 – 60	Hold at 180°C
60 – 95	Ramp to 200°C
95 – 125	Hold at 200°C
125+	Cool to vessel release

Table 13: Acid Quantities Used for Sample Digestion and Microwave Programme Selection

Sample	Sample Mass (g)	HNO_3 (mL)	HCl (mL)	H_2O_2 (mL)	HF (mL)	Temperature Programme
Lipstick, Lip Gloss, Lip Liner, Mascara, Eye powders, Eye Liners, Face Powders, Liquid Foundation	0.1 ± 0.01	8	1	-	0.5	Long
Mouthwash	0.1 ± 0.01	5	1	1	-	Short
Toothpaste	0.1 ± 0.01	8	1	-	0.5	Short
Sun protection	0.1 ± 0.01	8	1	-	0.5	Short

After the digestion programme, the samples were made up to the final volume with ultrapure water (18.2 MΩ.cm resistivity). For analysis, the samples required subsequent dilution using 8% HNO_3/1% HCl. The dilution levels are shown in Table 14.

Table 14: Final Sample Volume and Dilution Factors

Sample	Final volume in water (mL)	Dilution for analysis	Total overall dilution factor
Lipstick, Lip Gloss, Lip Liner, Mascara, Eye powders, Eye Liners, Face Powders, Liquid Foundation	50	2-fold	1000
Mouthwash	20	5-fold	1000
Toothpaste	50	2-fold	1000
Sun protection	50	2-fold	1000

3.5. Sample Analysis

3.5.1. Instrumentation

The analysis was performed using a collision/reaction cell ICP-MS (7700x or 7900, Agilent Technologies, UK). The ICP-MS was operated in standard mode (no gas) and helium (He) mode which was used to reduce and overcome interferences on the selected isotopes. The He mode removes polyatomic interferences with the same nominal mass as the analyte ions by collision induced kinetic energy discrimination. The typical instrumental parameters used for the analysis are detailed in Table 15.

Table 15: Instrumental Parameters for the ICP-MS

Agilent 7700x or 7900 ICP-MS	
General Parameters	
Sample Depth:	8 mm
Nebuliser Pump Speed:	0.1 rps
Spray Chamber Temperature:	2°C
RF Power:	1550 W
Carrier Gas Flow:	1.00-1.11 L/min
Helium Gas Flow (in He Mode)	4-5 mL/min
Acquisition Parameters	
Number of points per mass	1
Number of replicates	3
Number of sweeps	100
Measured Isotopes	He mode: ^{75}As No gas mode: ^{111}Cd, ^{121}Sb, ^{202}Hg, ^{208}Pb Internal Standards: ^{115}In, ^{128}Te, ^{159}Tb, ^{175}Lu

For Hg and Pb, the results are reported as the sum of several isotopes to improve counting statistics and minimise effects from natural isotopic variation. Additionally, ^{95}Mo was monitored to correct for any potential interference on ^{111}Cd. For some sample types, it was observed that In as the internal standard for As did not fully correct for matrix effects and Te performed better in these instances.

3.5.2. Calibration standards

Quantification of target elements in the samples was performed *via* external calibration using calibration standards from an ISO 17034 accredited supplier (Romil, UK). The working standards were prepared by dilution in 8% HNO_3/1% HCl. The calibration standards also contained methanol to mitigate ionisation effects.

As noted in the method verification, IQC standards were prepared from secondary calibration standards as an independent source and check on the calibration accuracy. These were sourced from alternative batches from the ISO 17034 accredited supplier (Romil, UK).

3.5.3. Quality Control

For each ICP-MS analysis, the following quality checks and criteria were applied:

- Calibration linearity was >0.99
- Internal standard recovery was within 75-125%
- IQC standard was with 80-120%
- The recovery of the triplicate spiked samples was within 70-150% (for mouthwash)
- The QC material or CRM was within 75-125% (excluding mouthwash)

3.5.4. Semi-Quantitative Analysis

During the method verification stage, semi-quantitative analysis was undertaken to establish if any other components of the sample may cause interferences for the analytes of interest. A number of elements were found to be present, e.g. zinc, copper, barium, chromium, some of which correlated to the ingredients list. This additional information is provided as <u>indicative only</u> and semi-quantitative in nature.

4. Results

In total, 91 samples were analysed across 10 product groups. As noted, above, due to time and budgetary restraints, it was not possible to analyse the makeup powder samples. However the verification was successful therefore the method is ready for any future follow up work.

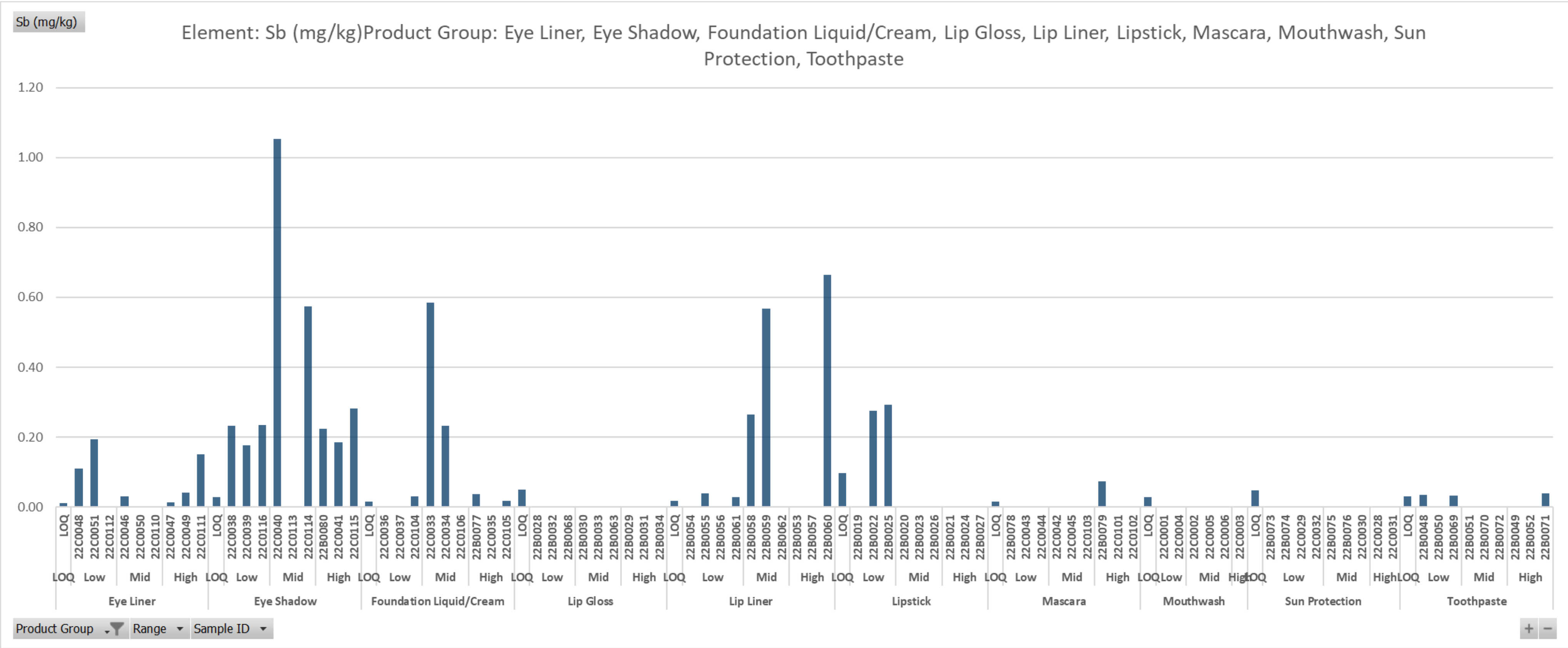
The results are presented graphically by element, using the average of the two replicates for each sample. The numerical results are provided in Appendix 2. All concentrations are given as mg/kg. Where no bar is present, the sample was below the LOQ. The samples are arranged in ascending price order for each product type.

There were 12 instances where one replicate of the sample was <LOQ and the second provided a result above the LOQ. In these cases, the value above the LOQ was presented as considering the worst-case scenario. However, it should be noted that the result was only marginally above the LOQ.

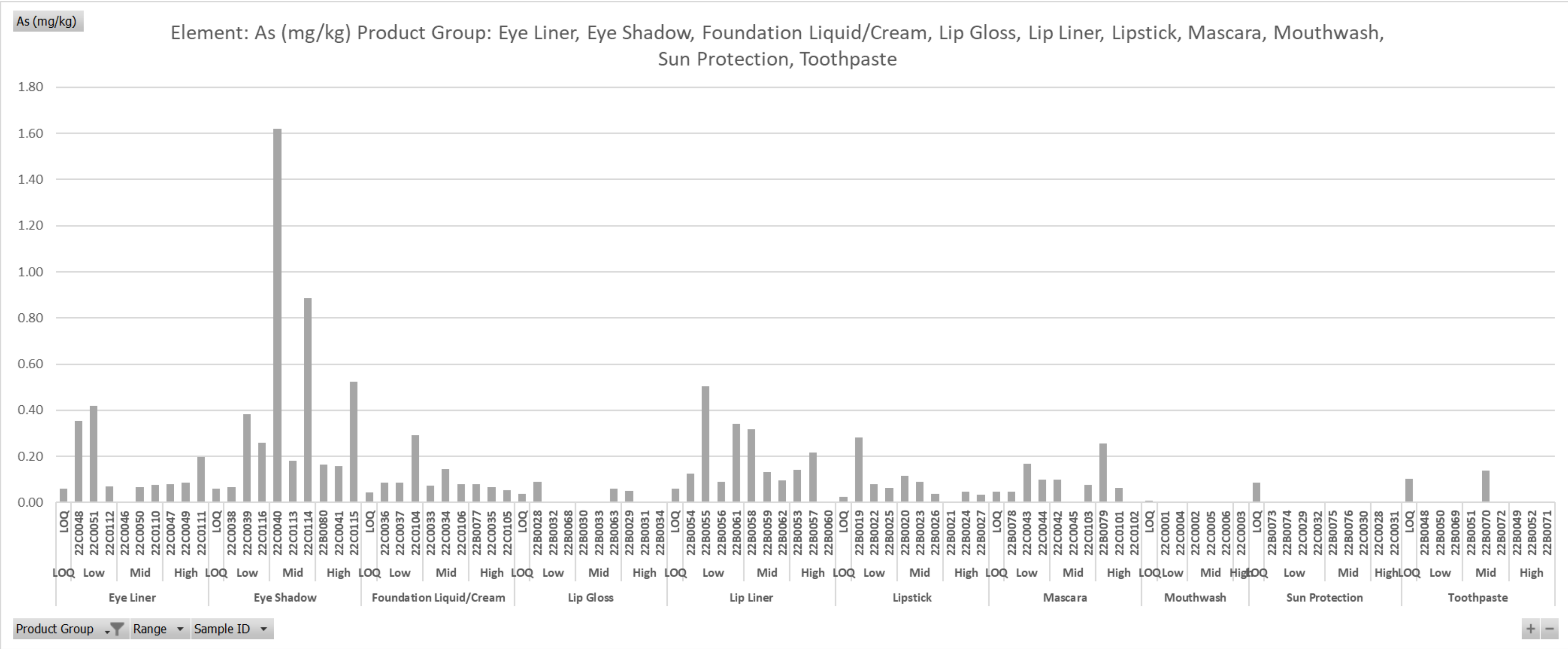
Additionally, indicative semi-quantitative data is also provided where significant levels were observed and shown in Appendix 3.

Information on price, place of purchase internet or high street, and the colour of the product have been included in Appendix 1 as well as providing price and the colour of the product against numerical results in Appendix 2.

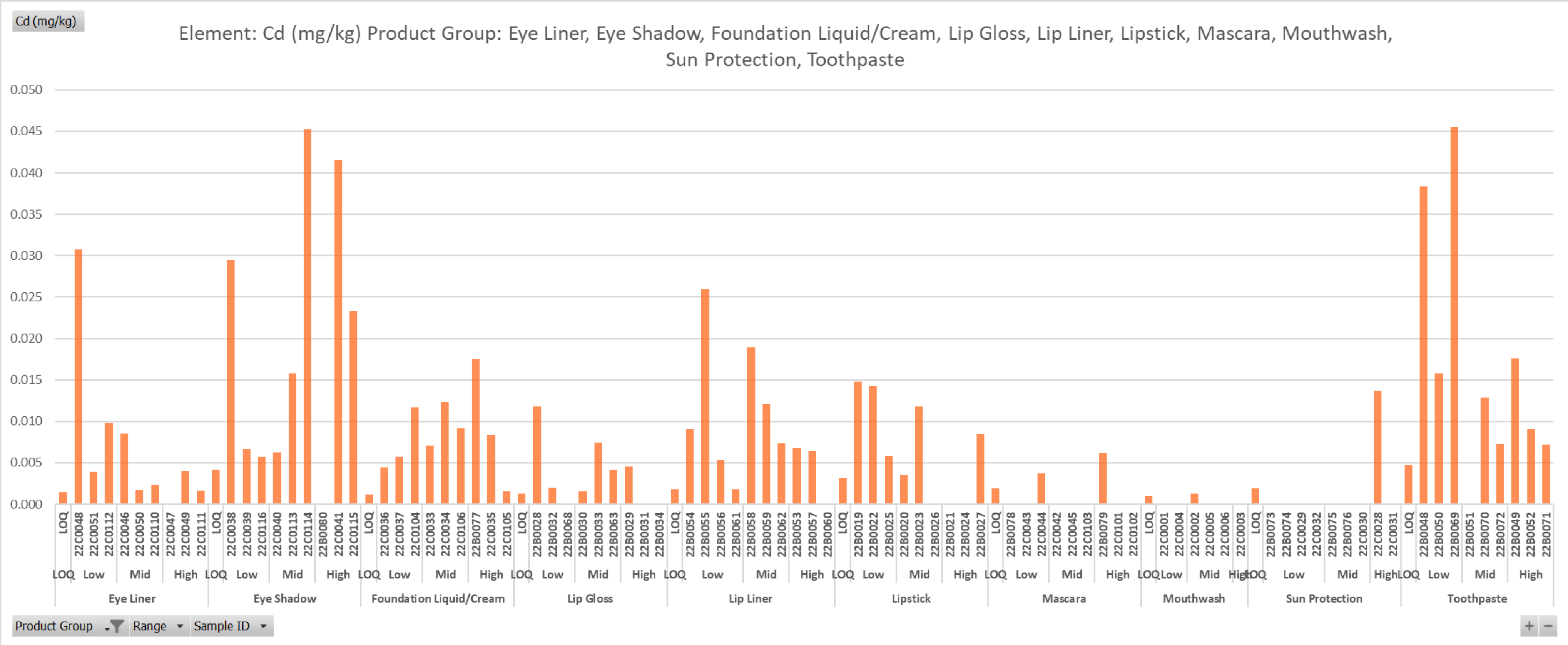
4.1. Antimony (Sb)



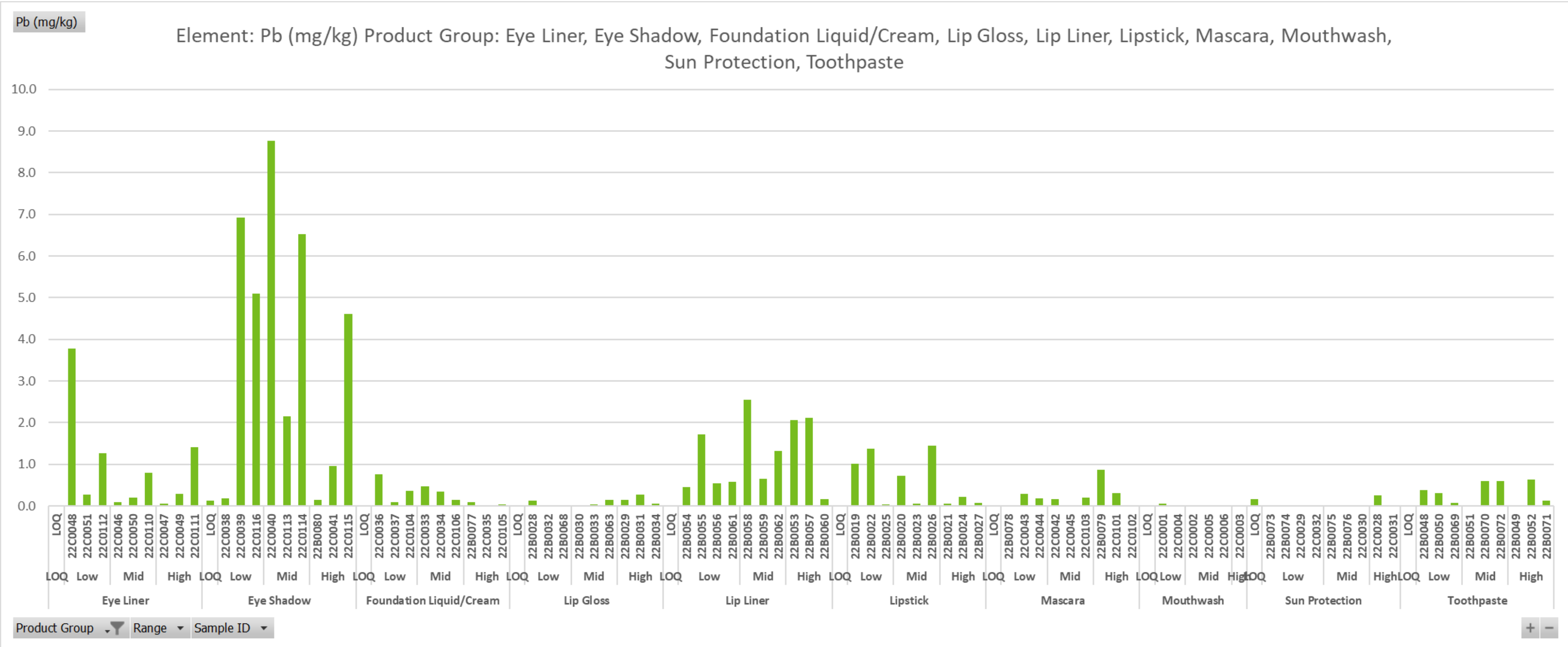
4.2. Arsenic (As)



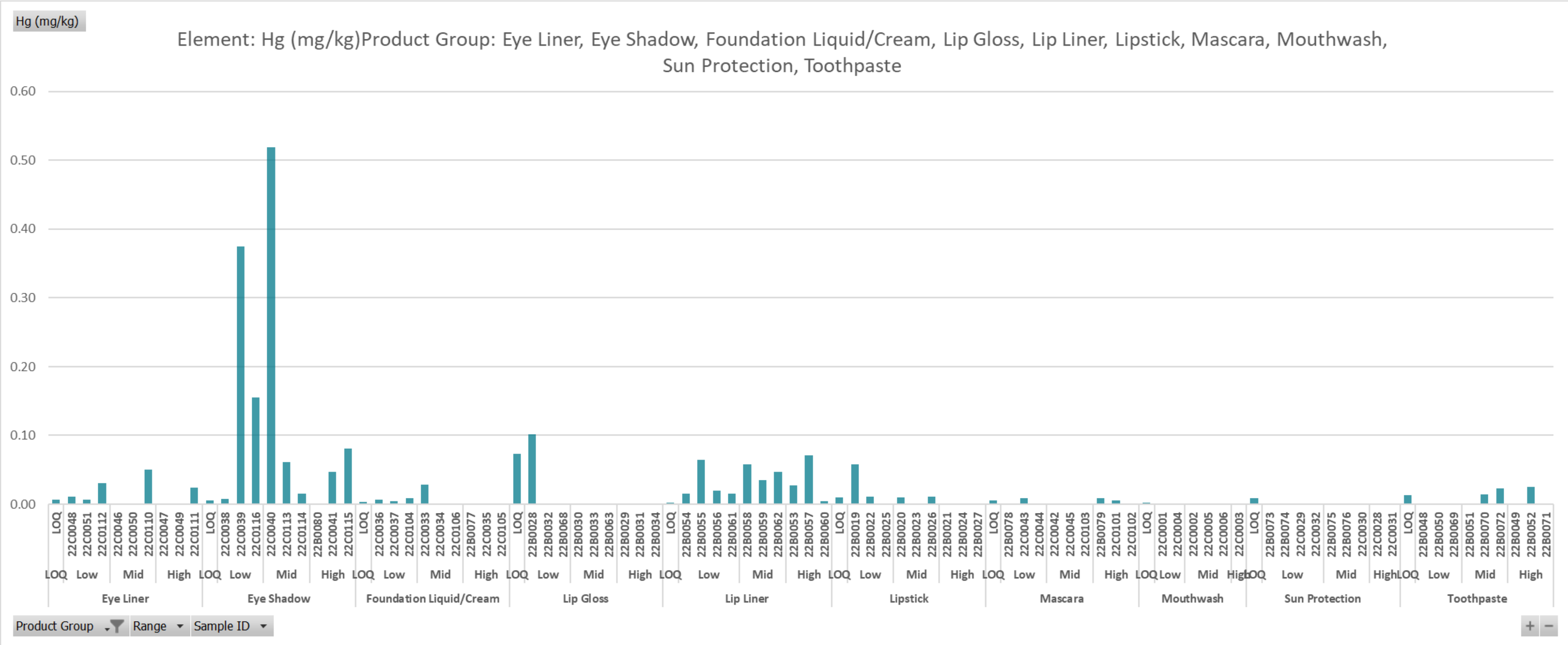
4.3. Cadmium (Cd)



4.4. Lead (Pb)


Pb (mg/kg)
Element: Pb (mg/kg) Product Group: Eye Liner, Eye Shadow, Foundation Liquid/Cream, Lip Gloss, Lip Liner, Lipstick, Mascara, Mouthwash, Sun Protection, Toothpaste
Eye Liner
Eye Shadow
Foundation Liquid/Cream
Lip Gloss
Lip Liner
Lipstick
Mascara
Mouthwash
Sun Protection
Toothpaste
Product Group
Range
Sample ID

4.5. Mercury (Hg)



4.6. Data Summary

Tables 16 a-e. provides a summary of the results for Sb, As, Cd, Pb and Hg compared with Table 16 f. BVL guidance limits for technically avoidable heavy metal content in cosmetic products. The number of samples analysed for each product type is indicated in brackets.

Additional, indicative/semi-quantitative values for other metals are shown in Appendix 3.

Table 16 a: Summary of Heavy Metal Content for Sb in Comparison to BVL (German) Guidance Values for Technically Avoidable Heavy Metals Content

Product Type	LOQ (mg/kg)	No. of samples <LOQ	Nos. of samples >BVL limit	Max Content (mg/kg)
Lip stick (9)	<0.096	7	0	0.292
Lip gloss (9)	<0.050	9	0	0.050
Toothpaste (9)	<0.031	6	0	0.040
Mouthwash (6)	<0.029	6	0	0.029
Lip liner (10)	<0.018	5	**2**	0.665
Sun Protection (9)	<0.047	9	0	0.047
Eye shadow (9)	<0.028	1	**2**	1.053
Mascara (9)	<0.015	8	0	0.074
Eye liner (9)	<0.011	3	0	0.194
Foundation (9)	<0.015	4	**1**	0.585

Table 16b: Summary of Heavy Metal Content for As in Comparison to BVL (German) Guidance Values for Technically Avoidable Heavy Metals Content

Product Type	LOQ (mg/kg)	Nos. of samples <LOQ	Nos. of samples >BVL limit	Max Content (mg/kg)
Lip stick (9)	<0.023	1	0	0.283
Lip gloss (9)	<0.039	6	0	0.090
Toothpaste (9)	<0.101	8	0	0.140
Mouthwash (6)	<0.007	6	0	0.007
Lip liner (10)	<0.060	1	**1**	0.505
Sun Protection (9)	<0.087	9	0	0.087
Eye shadow (9)	<0.060	0	**3**	1.620
Mascara (9)	<0.048	2	0	0.255
Eye liner (9)	<0.060	1	0	0.418
Foundation (9)	<0.045	0	0	0.144

Table 16c: Summary of Heavy Metal Content for Cd in Comparison to BVL (German) Guidance Values for Technically Avoidable Heavy Metals Content

Product Type	LOQ (mg/kg)	Nos. of samples <LOQ	Nos. of samples >BVL limit	Max Content (mg/kg)
Lip stick (9)	<0.003	3	0	0.015
Lip gloss (9)	<0.001	3	0	0.012
Toothpaste (9)	<0.005	1	0	0.046
Mouthwash (6)	<0.001	5	0	0.001
Lip liner (10)	<0.002	1	0	0.026
Sun Protection (9)	<0.002	8	0	0.014
Eye shadow (9)	<0.004	1	0	0.045
Mascara (9)	<0.002	7	0	0.006
Eye liner (9)	<0.001	1	0	0.031
Foundation (9)	<0.001	0	0	0.018

Table 16d: Summary of Heavy Metal Content for Pb in Comparison to BVL (German) Guidance Values for Technically Avoidable Heavy Metals Content

Product Type	LOQ (mg/kg)	Nos. of samples <LOQ	Nos. of samples >BVL limit	Max Content (mg/kg)
Lip stick (9)	<0.012	0	0	1.447
Lip gloss (9)	<0.009	1	0	0.267
Toothpaste (9)	<0.026	2	**3**	0.639
Mouthwash (6)	<0.007	4	0	0.050
Lip liner (10)	<0.009	0	**3**	2.545
Sun Protection (9)	<0.168	8	0	0.248
Eye shadow (9)	<0.129	0	**6**	8.769
Mascara (9)	<0.011	1	0	0.859
Eye liner (9)	<0.014	0	**1**	3.780
Foundation (9)	<0.008	0	0	0.764

Table 16e: Summary of Heavy Metal Content for Pb in Comparison to BVL (German) Guidance Values for Technically Avoidable Heavy Metals Content

Product Type	LOQ (mg/kg)	Nos. of samples <LOQ	Nos. of samples >BVL limit	Max Content (mg/kg)
Lip stick (9)	<0.010	5	0	0.058
Lip gloss (9)	<0.073	8	**1**	0.101
Toothpaste (9)	<0.014	6	0	0.025
Mouthwash (6)	<0.002	6	0	0.002
Lip liner (10)	<0.003	0	0	0.071
Sun Protection (9)	<0.008	9	0	0.008
Eye shadow (9)	<0.005	1	**3**	0.519
Mascara (9)	<0.005	6	0	0.009
Eye liner (9)	<0.006	4	0	0.050
Foundation (9)	<0.015	4	**1**	0.585

Table 16 f. BVL Guidance Limits for Technically Avoidable Heavy Metal Content in Cosmetic Products.

BVL Guidance Limits	Sb (mg/kg)	As (mg/kg)	Cd (mg/kg)	Pb* (mg/kg)	Hg (mg/kg)
General	<0.5	<0.5	<0.1	<2.0	<0.1
Toothpaste				<0.5	
Theatre make-up				<5.0	

**The guidance limit values for Pb in theatre make-up products has not been applied.*

5. Discussion

This project provides an analytical comparison for trace heavy metals content, including Sb, As, Cd, lead and Hg in a variety of cosmetics products available on the UK market, identifying the challenges and difficulties in analysing many different complex matrices to ensure accurate measurement.

The digestion methodologies reviewed in the literature revealed many approaches and acid mixtures, with varying advantages and disadvantages, but without consensus on the best approach. The newly released ISO 21392:2021 standard goes someway to address this issue, however, in this work it was found that cloudy solutions and/or particulates still remained after a long and severe microwave assisted digestion method using HF acid. An additional consideration is the lack of suitable certified reference materials for cosmetic products which provide significant support to developing appropriate sample preparation methods and give testing laboratories suitable materials for validation. In this work, this issue was mitigated by use of alternative materials, namely the proficiency testing materials and a soil CRM. The lipstick and lip gloss were prepared for the cosmetics proficiency testing scheme under ISO 17043 accreditation by LGC Standards. The reference values were the consensus values obtained from the scheme. Whilst having a matrix QC material is hugely beneficial, there can be drawbacks to relying on consensus data such as biases, large uncertainty ranges and lack of SI traceability. Certified reference materials can address these issues through application of high accuracy calibration methods such isotope dilution analysis, which can provide lower uncertainties and traceability. However the cost of producing such materials is high, especially considering the large variety of matrices within the cosmetics arena. The use of NIST SRM 2709a, a soil CRM, enabled the methodology to be challenged as soils contain significant amounts of refractory compounds such as SiO_2 and TiO_2 which are also common ingredients in cosmetic products. Recoveries for the QC materials and CRM were within 75-125% throughout this work for all elements, demonstrating good performance and, with the use of spiked samples, provided confidence in the data for the samples under investigation.

The results of analysis showed levels of heavy metals content in most cosmetic products near or below the limit of quantification for each heavy metal such that this would be considered not detected. There are, however, notable exceptions where significant levels of heavy metals including As, Sb and Pb were found to be present particularly in eye shadow and lip liner products. Where As, Sb, Hg and Pb were detected, the Pb content was significant with 13 out of 91 samples having a lead content greater than 2 mg/kg, 4 out of 91 samples having a lead content greater than 5 mg/kg although no samples had a lead content greater than 10 mg/kg.

The graphs in Sections 4.1 to 4.5 are arranged in ascending price order. When examining the results for lead in the eye shadow and lip liner products, there does not appear to be a correlation between the higher lead levels and price as the values are split across the price ranges. The colour of the products are shown in Appendix 1, with the concentration levels and colour provided in Appendix 2. Again, considering the eye shadow and lip liner, it appeared that products containing orange-red, brown and red colours had significant levels of Pb and Sb compared with other samples. Attempting to identify the corresponding colour to the pigment in the ingredients list was difficult, as in most cases the manufacturer had given multiple pigments. For eye shadows and lip liners, the commonly listed pigment with a reddish colour appeared to be CI 77491 (iron oxide).

Other metals determined on a semi-quantitative basis including nickel (Ni), cobalt (Co) and zirconium (Zr) are shown in Appendix 3 and may suggest a possible future project to consider whether guidance values should be considered for these prohibited metals.

The significance of the UK results is difficult to evaluate with the small number of samples analysed in this project so it is useful to compare with data from studies implementing similar methodology. The BLV (Germany) studies provide a useful comparison and the data was used to set the guidance values for technically avoidable heavy metals content in cosmetic products (from 1735 samples). Using these guidance values and the 90% compliance criteria, it can be seen from UK market data in Table 16 that this could be applied to the UK with minimal impact. However, this work was intended as exploratory and has a limited selection of products, therefore caution must be applied and further testing is required.

Additionally, when considering the BLV guidance values, it should be noted that the authorities made an additional risk management decision for lead content in different product categories. For toothpaste, this was set at 0.1 mg/kg reflecting the higher risk through potential ingestion and frequency of use. Conversely a higher value was set for carnival products at 5 mg/kg, suggesting the use of highly coloured pigments and minerals containing heavy metal impurities. The scope of products considered as carnival cosmetic products was not described by BLV but it is noted that the UK survey identified eye shadows as the cosmetic product that exceeded the 2 mg/kg guidance limit value. If the 5 mg/kg level was applied to eye shadows, the 90% acceptance criteria of 2 mg/kg for lead content for all other cosmetic products is more likely to have been met. Consequently, it could be considered that eye shadows may have a different guidance limit value for technically avoidable heavy metals content but would require clear definitions to establish the products which would fall within the scope of eye shadows.

Further work with a larger and more extensive market coverage should be considered. Additionally, the scope could be extended to consider other elements, e.g. chromium (Cr) as highlighted by the indicative data.

6. Conclusion

In conclusion, this work has achieved the aim and objectives of the project by reviewing existing literature for analytical methodology, summarising international legislation and guidance, assessed the applicability of method implementation and performed a small market survey to establish the feasibility in setting guidance values for technically avoidable heavy metals in UK cosmetic products to meet regulatory requirements.

Appendix 1 – Sample List

Product Type	Price Range	Price (£)	Retail	Sample No.	Colour
Eyeliner	Low	2.00	High Street	22C0048	White
		3.00	High Street	22C0051	Purple
		3.99	Online	22C0112	Blue
	Mid	4.99	High Street	22C0046	Black
		4.99	High Street	22C0050	Black
		6.00	Online	22C0110	Red
	High	6.99	High Street	22C0047	Brown
		8.99	High Street	22C0049	Green
		18.00	Online	22C0111	Yellow-Green
Eye Shadow	Low	5.49	Online	22C0038	Yellow
		5.99	High Street	22C0039	Red-Purple
		5.99	High Street	22C0116	Orange-Yellow
	Mid	6.99	High Street	22C0040	Brown
		8.99	High Street	22C0113	Orange-Yellow
		13.00	Online	22C0114	Blue
	High	16.00	High Street	22B0080	Blue-Green
		18.00	Online	22C0041	Purple
		21.00	High Street	22C0115	Orange-Red

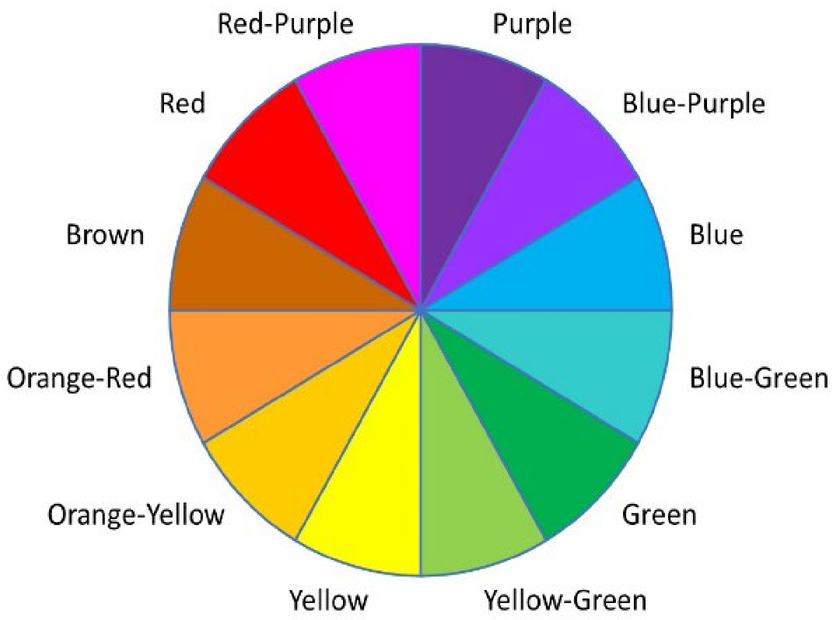
Product Type	Price Range	Price (£)	Retail	Sample No.	Colour
Foundation Liquid/Cream	Low	6.40	Online	22C0036	Orange-Yellow
		7.99	High Street	22C0037	Brown
		8.99	High Street	22C0104	Orange-Red
	Mid	9.99	High Street	22C0033	Orange-Red
		9.99	High Street	22C0034	Red-Purple
		10.00	Online	22C0106	Orange-Yellow
	High	11.49	High Street	22B0077	Orange-Red
		18.60	Online	22C0035	Brown
		29.00	High Street	22C0105	Orange-Yellow
Lip Gloss	Low	4.99	High Street	22B0028	Purple
		5.00	Online	22B0032	Red-Purple
		6.99	High Street	22B0068	Red-Purple
	Mid	9.99	High Street	22B0030	White
		10.00	High Street	22B0033	Brown
		11.00	Online	22B0063	Blue
	High	17.50	High Street	22B0029	Red
		18.00	Online	22B0031	Orange-Red
		23.00	High Street	22B0034	Red
Lip Liner	Low	1.99	High Street	22B0054	Brown
		3.99	High Street	22B0055	Red
		3.99	High Street	22B0056	Red-Purple
		4.45	Online	22B0061	Purple
	Mid	4.99	High Street	22B0058	Red-Purple
		6.95	High Street	22B0059	Orange-Yellow
		13.00	Online	22B0062	Orange-Red
	High	15.50	Online	22B0053	Red
		19.00	High Street	22B0057	Orange-Red
		19.00	High Street	22B0060	Red

Product Type	Price Range	Price (£)	Retail	Sample No.	Colour
Lipstick	Low	1.99	Online	22B0019	Red-Purple
		3.00	High Street	22B0022	Orange-Red
		3.00	High Street	22B0025	Brown
	Mid	7.99	High Street	22B0020	Red-Purple
		8.00	Online	22B0023	Brown
		8.99	High Street	22B0026	Blue-Purple
	High	17.50	High Street	22B0021	Red
		18.00	High Street	22B0024	Red-Purple
		20.00	Online	22B0027	Red
Mascara	Low	3.50	High Street	22C0078	Black
		4.99	Online	22C0043	Black
		6.00	High Street	22C0044	Black
	Mid	10.00	High Street	22C0042	Black
		11.00	High Street	22C0045	Brown
		12.00	Online	22C0103	Black
	High	26.00	Online	22C0079	Black
		26.50	High Street	22C0101	Black
		28.50	High Street	22C0102	Brown
Mouthwash	Low	0.99	High Street	22C0001	Blue-Green
		2.00	High Street	22C0004	Red-Purple
	Mid	5.00	High Street	22C0002	Blue
		5.00	High Street	22C0005	No Colour
		5.00	High Street	22C0006	No Colour
	High	8.00	High Street	22C0003	No Colour

Product Type	Price Range	Price (£)	Retail	Sample No.	Colour
Sun Protection	Low	4.00	High Street	22C0073	White
		4.00	Online	22C0074	White
		6.00	High Street	22C0029	White
		6.00	High Street	22C0032	Yellow
	Mid	6.99	Online	22C0075	White
		8.00	High Street	22C0076	Yellow
		8.00	High Street	22C0030	Yellow
	High	19.00	Online	22C0028	White
		20.00	High Street	22C0031	Yellow
Toothpaste	Low	1.50	High Street	22B0048	Red-White-Blue
		2.00	High Street	22B0050	Blue
		2.00	Online	22B0069	Red-Purple
	Mid	3.99	High Street	22B0051	Orange-Yellow
		6.99	High Street	22B0070	White
		7.00	Online	22B0072	Red-Purple
	High	12.00	Online	22B0049	Red-Purple
		16.00	High Street	22B0052	White
		20.00	High Street	22B0051	White

The colour of the cosmetic product was matched closest to the colour shown in the colour wheel in figure 1.

Figure 1



Colour wheel

Appendix 2 – Numerical Sample Results

The data in the following table is the average of the two replicates prepared for each sample.

Product Type	Price	Sample No.	Sb (mg/kg)	As (mg/kg)	Cd (mg/kg)	Pb (mg/kg)	Hg (mg/kg)	Colour
Eye Liner	Low	22C0048	0.110	0.353	0.031	3.78	0.011	White
		22C0051	0.194	0.418	0.004	0.275	0.007	Purple
		22C0112	<LOQ	0.069	0.010	1.27	0.031	Blue
	Mid	22C0046	0.031	<LOQ	0.009	0.092	<LOQ	Black
		22C0050	<LOQ	0.067	0.002	0.192	<LOQ	Black
		22C0110	<LOQ	0.075	0.002	0.801	0.050	Red
	High	22C0047	0.014	0.080	<LOQ	0.052	<LOQ	Brown
		22C0049	0.040	0.085	0.004	0.294	<LOQ	Green
		22C0111	0.150	0.197	0.002	1.41	0.024	Yellow-Green
		LOQ	**0.011**	**0.060**	**0.001**	**0.014**	**0.006**	
Eye Shadow	Low	22C0048	0.110	0.353	0.031	3.78	0.011	White
		22C0051	0.194	0.418	0.004	0.275	0.007	Purple
		22C0112	<LOQ	0.069	0.010	1.27	0.031	Blue
	Mid	22C0046	0.031	<LOQ	0.009	0.092	<LOQ	Black
		22C0050	<LOQ	0.067	0.002	0.192	<LOQ	Black
		22C0110	<LOQ	0.075	0.002	0.801	0.050	Red
	High	22C0047	0.014	0.080	<LOQ	0.052	<LOQ	Brown
		22C0049	0.040	0.085	0.004	0.294	<LOQ	Green
		22C0111	0.150	0.197	0.002	1.41	0.024	Yellow-Green
		LOQ	**0.028**	**0.060**	**0.004**	**0.129**	**0.005**	

Product Type	Price	Sample No.	Sb (mg/kg)	As (mg/kg)	Cd (mg/kg)	Pb (mg/kg)	Hg (mg/kg)	Colour
Foundation Liquid/ Cream	Low	22C0036	<LOQ	0.085	0.004	0.764	0.007	Orange-Yellow
		22C0037	<LOQ	0.086	0.006	0.086	0.004	Brown
		22C0104	0.031	0.292	0.012	0.369	0.009	Orange-Red
	Mid	22C0033	0.585	0.074	0.007	0.463	0.028	Orange-Red
		22C0034	0.233	0.144	0.012	0.351	<LOQ	Red-Purple
		22C0106	<LOQ	0.080	0.009	0.146	<LOQ	Orange-Yellow
	High	22B0077	0.037	0.079	0.018	0.099	<LOQ	Orange-Red
		22C0035	<LOQ	0.068	0.008	0.008	<LOQ	Brown
		22C0105	0.017	0.053	0.002	0.034	<LOQ	Orange-Yellow
		LOQ	**0.011**	**0.060**	**0.001**	**0.014**	**0.006**	
Lip Gloss	Low	22B0028	<LOQ	0.090	0.012	0.130	0.101	Purple
		22B0032	<LOQ	<LOQ	0.002	0.013	<LOQ	Red-Purple
		22B0068	<LOQ	<LOQ	<LOQ	0.014	<LOQ	Red-Purple
	Mid	22B0030	<LOQ	<LOQ	0.002	<LOQ	<LOQ	White
		22B0033	<LOQ	<LOQ	0.007	0.036	<LOQ	Brown
		22B0063	<LOQ	0.062	0.004	0.141	<LOQ	Blue
	High	22B0029	<LOQ	0.051	0.005	0.137	<LOQ	Red
		22B0031	<LOQ	<LOQ	<LOQ	0.267	<LOQ	Orange-Red
		22B0034	<LOQ	<LOQ	<LOQ	0.061	<LOQ	Red
		LOQ	**0.028**	**0.060**	**0.004**	**0.129**	**0.005**	
Lip Liner	Low	22B0054	<LOQ	0.126	0.009	0.460	0.015	Brown
		22B0055	0.038	0.505	0.026	1.72	0.064	Red
		22B0056	<LOQ	0.090	0.005	0.543	0.019	Red-Purple
	Mid	22B0061	0.027	0.341	0.002	0.581	0.015	Purple
		22B0058	0.264	0.318	0.019	2.55	0.058	Red-Purple
		22B0059	0.567	0.134	0.012	0.645	0.035	Orange-Yellow
	High	22B0062	<LOQ	0.095	0.007	1.32	0.047	Orange-Red
		22B0053	< LOQ	0.142	0.007	2.06	0.027	Red
		22B0057	<LOQ	0.216	0.006	2.12	0.071	Orange-Red
		LOQ	**0.018**	**0.060**	**0.002**	**0.009**	**0.003**	

Product Type	Price	Sample No.	Sb (mg/kg)	As (mg/kg)	Cd (mg/kg)	Pb (mg/kg)	Hg (mg/kg)	Colour
Lipstick	Low	22B0019	<LOQ	0.283	0.015	1.02	0.058	Red-Purple
		22B0022	0.275	0.078	0.014	1.37	0.011	Orange-Red
		22B0025	0.292	0.064	0.006	0.030	<LOQ	Brown
	Mid	22B0020	<LOQ	0.115	0.003	0.721	0.010	Red-Purple
		22B0023	<LOQ	0.090	0.012	0.051	<LOQ	Brown
		22B0026	<LOQ	0.036	<LOQ	1.45	0.011	Blue-Purple
	High	22B0021	<LOQ	<LOQ	<LOQ	0.052	<LOQ	Red
		22B0024	<LOQ	0.046	<LOQ	0.213	<LOQ	Red-Purple
		22B0027	<LOQ	0.034	0.008	0.080	<LOQ	Red
		LOQ	**0.096**	**0.023**	**0.003**	**0.012**	**0.010**	
Mascara	Low	22B0078	<LOQ	0.049	<LOQ	0.021	<LOQ	Black
		22C0043	<LOQ	0.168	<LOQ	0.290	0.008	Black
		22C0044	<LOQ	0.099	0.004	0.175	<LOQ	Black
	Mid	22C0042	<LOQ	0.101	<LOQ	0.166	<LOQ	Black
		22C0045	<LOQ	<LOQ	<LOQ	0.018	<LOQ	Brown
		22C0103	<LOQ	0.077	<LOQ	0.190	<LOQ	Black
	High	22B0079	0.074	0.255	0.006	0.859	0.009	Black
		22C0101	<LOQ	0.063	<LOQ	0.309	0.005	Black
		22C0102	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	Brown
		LOQ	**0.015**	**0.048**	**0.002**	**0.011**	**0.005**	
Mouthwash	Low	22C0001	<LOQ	<LOQ	<LOQ	0.050	<LOQ	Blue-Green
		22C0004	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	Red-Purple
	Mid	22C0002	<LOQ	<LOQ	0.001	0.016	<LOQ	Blue
		22C0005	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	No Colour
		22C0006	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	No Colour
	High	22C0003	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	No Colour
		22C0001	<LOQ	<LOQ	<LOQ	0.050	<LOQ	Blue-Green
		LOQ	**0.029**	**0.007**	**0.001**	**0.007**	**0.002**	

Product Type	Price	Sample No.	Sb (mg/kg)	As (mg/kg)	Cd (mg/kg)	Pb (mg/kg)	Hg (mg/kg)	Colour
Sun Protection	Low	22B0073	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	White
		22B0074	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	White
		22C0029	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	White
		22C0032	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	Yellow
	Mid	22B0075	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	White
		22B0076	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	Yellow
		22C0030	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	Yellow
	High	22C0028	<LOQ	<LOQ	0.014	0.248	<LOQ	White
		22C0031	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	Yellow
		LOQ	**0.047**	**0.087**	**0.002**	**0.168**	**0.008**	
Toothpaste	Low	22B0048	0.034	<LOQ	0.038	0.382	<LOQ	Red-White-Blue
		22B0050	<LOQ	<LOQ	0.016	0.315	<LOQ	Blue
		22B0069	0.032	<LOQ	0.046	0.068	<LOQ	Red-Purple
	Mid	22B0051	<LOQ	<LOQ	<LOQ	<LOQ	<LOQ	Orange-Yellow
		22B0070	<LOQ	0.140	0.013	0.588	0.014	White
		22B0072	<LOQ	<LOQ	0.007	0.600	0.022	Red-Purple
	High	22B0049	<LOQ	<LOQ	0.018	<LOQ	<LOQ	Red-Purple
		22B0052	<LOQ	<LOQ	0.009	0.639	0.025	White
		22B0071	0.040	<LOQ	0.007	0.128	<LOQ	White
		LOQ	**0.031**	**0.101**	**0.005**	**0.026**	**0.014**	

Appendix 3 – Screening Results for Other Indicative Elements

When determining the heavy metals in a cosmetic product, it is possible to perform semi-quantitative analysis of other elements present in the same sample. Indicative results are shown for each type of cosmetic product in the following tables:

Product Type	Ba (mg/kg)	Co (mg/kg)	Cr (mg/kg)	Cu (mg/kg)	Li (mg/kg)	Mo (mg/kg)	Ni (mg/kg)	Sn (mg/kg)	Tl (mg/kg)	V (mg/kg)	Zr (mg/kg)
Lip stick	>2[a]	>2	>2		>2						>2
Lip gloss	>2				>2			>2			>2
Toothpaste	≤17		>2					≤5,000			≤15
Mouthwash	-		>0.1								
Lip liner	≤2,000	>0.5	>1.5	>1.5	>1.5	>1.5	>1.5	>1.5		>1 .5	>1.5
Sun Protection											>10[b]
Eye shadow			>5[c]	>1	>10	>0.5	>5	[d]	>0.5	>10	>5
Mascara	>3	>1	>1		>3	>0.3	>1	>0.3		>1	>1
Eye liner	>10	>3	>3	>0.5	>3	>0.5	>3	>10	>0.2	>3	>3
Foundation	>3	>0.5	>0.5		>3		>0.5	>3		>0.5	>10
Face powder	>2[a]		>3		>3		>3	>3			>70

[a] Higher priced products generally have higher barium (Ba) content up to 20,000 mg/kg.
[b] Found in a single product.
[c] One product found to have chromium (Cr) content up to 90,000 mg/kg
[d] One product found to have tin (Sn) content up to 1,500 mg/kg.

Glossary

AFS	Atomic fluorescence spectrometry
AAS	Atomic absorption spectrometry
As	Arsenic
ASEAN	Association of South East Asian Nations
BVL	Bundesamt fur Verbraucherschutz und Lebensmittelsicherheit
Ba	Barium
Ca	Calcium
Cd	Cadmium
CEN	European Standards Organisation
Co	Cobalt
Cr	Chromium
CRM	Certified reference material
CVAAS	Cold vapor atomic absorption spectrometry
CVAFS	Cold vapor atomic fluorescence spectrometry
Cu	Copper
FAAS	Flame atomic absorption spectrometry
FDA	United States Food and Drug Administration
GF-AAS	Graphite furnace atomic absorption spectrometry
HBO_4	Boric acid
HCl	Hydrochloric acid
$HClO_4$	Perchloric acid
HF	Hydrofluoric acid
Hg	Mercury
HNO_3	Nitric acid
H_2O_2	Hydrogen peroxide
H_2SO_4	Sulfuric acid
ICCR	International Cooperation on Cosmetics Regulations
ICP	Inductively coupled plasma
ISO	International Standards Organisation
$KMnO_4$	Potassium permanganate
JRC	Joint Research Council
Li	Lithium
LOD	Limit of detection
LOQ	Limit of quantification
Mo	Molybdenum
MS	Mass spectrometry
MS/MS	Tandem mass spectrometry
Ni	Nickel
OES	Optical emission spectrometry
Pb	Lead
QC	Quality control
Sb	Antimony
SiO_2	Silicon dioxide
Sn	Tin
Ti	Titanium

TiO_2	Titanium dioxide
V	Vanadium
XRF	X-ray fluorescence spectrometry
Zr	Zirconium
ppm	Parts per million
mg/kg	Milligrams per kilogram equivalent to ppm
µg/g	Micrograms per gram equivalent to ppm
ppb	Parts per billion
µg/kg	Micrograms per kilogram equivalent to ppb
≤	Less or equal to
≥	Greater or equal to
<	Less than
>	Greater than

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