Chemical Metrology Division
Applied Sciences Group
Health Sciences Authority
1 Science Park Road, #01-05/06,
The Capricorn, Singapore Science Park II,
Singapore 117528
Tel: 65 6775 1605 Fax: 65 6775 1398
Website: www.hsa.gov.sg
Email: HSA_CML@hsa.gov.sg

Ref. No.: CML-HRM-2012A/05
Date of Issue: 24 Oct 2023

Certificate of Analysis

CERTIFIED REFERENCE MATERIAL
HRM – 2012A

Trace Elements in Lipstick

Batch Number
STY-0094-001

Description
The certified reference material (CRM) was produced by spiking lipstick material with solutions of $Cu(NO_3)_2.3H_2O$, $Pb(NO_3)_2$, $Na_2HAsO_4.7H_2O$, $HgCl_2$ and a stabilising agent. The material (10 g) was bottled in a clean brown glass bottle and sealed with a screw cap.

The reference material was produced with reference to the requirements set out in ISO/IEC 17025:2017 [1], ISO 17034:2016 [2] and ISO Guide 35:2017 [3].

Certified Mass Fraction Values
A certified value is a value for which a laboratory has the highest confidence in its accuracy, in that all known or suspected sources of biases have been investigated and accounted for. The certified mass fraction values for the four elements in the CRM are listed below. The certified mass fraction values for Cu, Hg and Pb were determined by inductively coupled plasma mass spectrometer using isotope dilution mass spectrometry (ICP-IDMS) [4]. The certified mass fraction value for As was determined by inductively coupled plasma high resolution mass spectrometer (ICP-HR-MS) using standard addition method.

Analyte	Mass fraction	Unit
Arsenic	7.94 ± 0.53	mg/kg
Copper	207.4 ± 6.5	mg/kg
Mercury	1.99 ± 0.16	mg/kg
Lead	22.7 ± 1.7	mg/kg

The mass fraction value is expressed as the certified value ± the expanded uncertainty.

The uncertainty listed with the certified value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95% confidence). The certified value has an associated measurement uncertainty attributed to uncertainty contribution from characterisation of the material

(u_{char}), uncertainty in the homogeneity of the material (u_{bb}) and uncertainty in the stability of the material (u_{stab}). The u_{char} was evaluated by combining uncertainties from method precision, method recovery, the concentration of calibration solution, weighing and different ion pairs used (for Hg and Pb), in accordance with ISO/IEC Guide 98-3:2008 [5].

Homogeneity

Homogeneity testing on the analytes in the material was performed on eleven bottles with two sub-samples taken from each bottle. ICP-MS was employed for the determination of the analytes. The sample size taken for homogeneity testing was about 0.35 g. No significant differences in the between and within-bottle variances were found using one-way ANOVA at 95 % confidence level [3]. Thus, the material was regarded to be sufficiently homogeneous. The u_{bb} was evaluated from the uncertainty due to between-bottle inhomogeneity.

Stability

Short-term stability testing on the analytes in the material at 50 °C (maximum allowable transportation temperature) showed that they were stable up to 14 days.

The long-term stability of the analytes at storage temperature (18 °C to 25 °C) was evaluated on three occasions over a period of up to 4 months after preparation. The results showed that the analytes were stable over the study period. The u_{stab} was estimated from the standard error of the slope.

Validity of Certified Mass Fraction Values

The certified mass fraction values are valid within its measurement uncertainty until **19 Nov 2026**, provided that the CRM is subjected to the same handling and storage conditions as stated in this Certificate of Analysis (COA).

The CRM will be continuously monitored during the validity period to determine if any substantive change to the certified values has occurred. If necessary, its user will be advised or an updated COA may be issued when the property value of the CRM is found to have changed.

Analytical Methods

The certified mass fractions of Cu, Hg and Pb in the material were determined by exact-matching ICP-IDMS. Standard reference materials from the National Institute of Standards and Technology (NIST, USA) (Product No. SRM3114 for Cu, SRM3133 for Hg and SRM3128 for Pb) were used as calibration standards for IDMS measurements. Enriched isotopes ^{65}Cu, ^{201}Hg and ^{206}Pb from Oak Ridge National Laboratory were used as the internal standards. The calibration blends were prepared gravimetrically by mixing appropriate amount of calibration standard solutions and internal standard solutions. The sample blends were prepared by spiking appropriate amount of internal standard into the material. Quality control blends were also prepared and analysed concurrently. Both sample and quality control blends were subjected to microwave acid digestion using 5 mL HNO_3, 0.2 mL HF and 2 mL H_2O_2 to give a clear digest.

The certified mass fraction of As was determined by ICP-HR-MS using standard addition method. Ga standard reference material from NIST (Product No. SRM3119a) was added to the sample digest as internal standard. Different amounts of As standard reference material from NIST (Product No. SRM3103a) were then spiked into the sample digest to produce sample blends. Quality control blends were also prepared and analysed concurrently.

Metrological Traceability

The certified mass fraction values are traceable to the International System of Units (SI) through the use of standard reference materials from NIST.

Intended Use

The CRM is intended for use in the validation of methods or as quality control (QC).

Instruction for Use

After use, the bottle should be re-capped, sealed with Parafilm and stored at storage temperature. The minimum sample size for each use should be about 0.35 g.

Storage

The material should be stored at 18 °C to 25 °C in its original bottle. Exposure to direct intense light and ultraviolet radiation should be avoided.

Safety Precautions for Users

Treat the material as hazardous substance. Use appropriate work practices when handling to avoid skin or eye contact or ingestion.

Further Information

Please direct all enquiries regarding this CRM to the contact in this COA.

References

1. ISO/IEC 17025:2017 General requirements for the competence of testing and calibration laboratories.
2. ISO 17034:2016 General requirements for the competence of reference material producers.
3. ISO Guide 35:2017 Reference materials – Guidance for characterisation and assessment for homogeneity and stability.
4. Sargent, M.; Harrington, C.; Harte, R.; *Guidelines for Achieving High Accuracy in Isotope Dilution Mass Spectrometry*, RSC Publishing, 2002.
5. ISO/IEC Guide 98-3:2008 Uncertainty of measurement – Part 3: Guide to the expression of uncertainty in measurement (GUM:1995).

Certificate Revision Records

Certificate of Analysis CML-HRM-2012A/02 replaces Certificate CML-HRM2012A/01 issued on 19 November 2018.
Certificate of Analysis CML-HRM-2012A/03 replaces Certificate CML-HRM2012A/02 issued on 05 November 2019.
Certificate of Analysis CML-HRM-2012A/04 replaces Certificate CML-HRM2012A/03 issued on 13 November 2020.
Certificate of Analysis CML-HRM-2012A/05 replaces Certificate CML-HRM2012A/04 issued on 18 October 2021.

Note

HSA does not assume any liability with respect to any loss caused by improper use and/or storage of the reference material by the customer.

Dr Teo Tang Lin
Division Director
Chemical Metrology Laboratory
Chemical Metrology Division