

Certificate of Analysis

CERTIFIED REFERENCE MATERIAL HRM – 2013A

Inorganic Elements in Herbal Material

Batch Number

STY-0122-001

Description

The certified reference material (CRM) was produced from a batch of herbal material which was found to contain appropriate levels of arsenic, cadmium and lead. The powdery material was homogenised using a drum mixer for a period of about 10 days. The material was packed into pre-cleaned 30-mL amber glass bottles, each containing about 10 g of herbal material. The bottled samples were then disinfected by γ -irradiation process at about 5-10 kGy¹.

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 arsenic, cadmium and lead in the CRM are listed below. The certified mass fraction values for arsenic was determined by inductively coupled plasma high resolution mass spectrometer (ICP-HR-MS) using standard addition method. The certified mass fraction values for cadmium and lead were determined by inductively coupled plasma mass spectrometer using isotope dilution mass spectrometry (ICP-IDMS) [4].

Analyte	Certified mass fraction	Unit
Arsenic	0.471 $\pm$ 0.065	mg/kg
Cadmium	0.487 $\pm$ 0.026	mg/kg
Lead	2.263 $\pm$ 0.094	mg/kg

The mass fraction value is expressed as the certified value $\pm$ the expanded uncertainty.

¹ The irradiation work was performed by a subcontractor.

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, the concentration of calibration solution, weighing, bias from using different ion pairs (for cadmium and lead only) and the relative atomic mass (for lead only), 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 0.3 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 for up to 14 days.

The long-term stability of the analytes at storage temperature (18 to 25 °C) was evaluated on three occasions over a period of up to 15 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 **26 Feb 2028**, 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 if the property value of the CRM is found to have changed or an updated COA has been issued.

Analytical Methods

The certified mass fraction of arsenic was determined by ICP-HR-MS using standard addition method. Gallium standard reference material from the National Institute of Standards and Technology (NIST, USA) (Product No. SRM3119a) was added to the sample digest as internal standard. Different amounts of arsenic 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.

The certified mass fractions of cadmium and lead in the material were determined by exact-matching ICP-IDMS. Standard reference materials from NIST (Product No. SRM3108 for cadmium and SRM3128 for lead) were used as calibration standards for IDMS measurements. Enriched isotopes ^{111}Cd 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.

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

For the validation of methods or as quality controls used to determine the mass fraction of arsenic, cadmium and lead in herbal material.

Instruction for Use

The bottle should be re-capped as soon as possible after opening, sealed with Parafilm and stored at storage temperature. To reduce moisture absorption, weighing needs to be performed as quickly as possible and the bottle should not be left open after sampling. The minimum sample size for each use should be about 0.3 g. The certified mass fraction values may not be valid if smaller amounts are taken.

Storage

The material should be stored at 18 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 Ref. No.	Date of issue	Reason for issuance
CML-HRM2013A/01	26 Aug 2020	Issuance of first certificate
CML-HRM2013A/02	03 Feb 2023	Extension of expiry date
CML-HRM2013A/03	20 Feb 2025	Extension of expiry date

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.



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