

Certificate of Analysis

CERTIFIED REFERENCE MATERIAL HRM – 2016A

Mercury in Water

Batch Number

STY-0150-001

Description

A unit of the certified reference material (CRM) HRM-2016A consists of five amber glass ampoules. Each ampoule contains approximately 10 mL of acidified purified water (approximately 3% v/v of nitric acid and 2% v/v of hydrochloric acid) spiked with mercury standard solution.

The CRM 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 Value

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 has been investigated and accounted for. The certified mass fraction value for mercury was determined by inductively coupled plasma mass spectrometer using isotope dilution mass spectrometry (ICP-IDMS) [4].

Certified Mass Fraction Value: $59.9 \pm 2.6 \mu\text{g/kg}$

The mass fraction value is expressed as the certified value $\pm$ 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, the concentration of calibration solution, weighing and bias from using different ion pair, in accordance with ISO/IEC Guide 98-3:2008 [5].

Homogeneity

Homogeneity testing on mercury in water was performed on eleven ampoules with two sub-samples taken from each ampoule using ICP-MS. The sample size taken for homogeneity testing was about 1.0 g. No significant differences in the between and within-unit 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-unit inhomogeneity.

Stability

Short-term stability testing on mercury in water at 45 °C (maximum allowable transportation temperature) showed that it was stable for up to 21 days.

The long-term stability of mercury in water at storage temperature (18 to 25 °C) was evaluated on three occasions over a period up to 3 months after preparation. The results showed that the analyte was stable over the study period. The u_{stab} was evaluated from the standard error of the slope.

Validity of Certified Mass Fraction Value

The certified mass fraction value is valid within its measurement uncertainty until **31 May 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 value 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 Method

The certified mass fraction of mercury in water was determined by exact-matching ICP-IDMS. Standard reference material from the National Institute of Standards and Technology (NIST, USA) (Product No. SRM 3133) was used as calibration standard for IDMS measurement. Enriched isotope ^{201}Hg from Oak Ridge National Laboratory (USA) was used as the internal standard. The calibration blends were prepared gravimetrically by mixing appropriate amount of calibration standard solution and internal standard solution. The sample blends were prepared by spiking appropriate amount of internal standard into the material. Quality control blends were also prepared and analysed concurrently.

Metrological Traceability

The certified mass fraction value is traceable to the International System of Units (SI) through the use of standard reference material from NIST.

Intended Use

For the validation of method or as quality control used to determine the mass fraction of mercury in water.

Instruction for Use

Prior to use, the material in the ampoule should be thoroughly mixed by gently inverting and tapping on the ampoule. Gentle pressure should be applied on the neck of the ampoule to ensure it breaks with a clean snap. **Always wear gloves and goggles when breaking off the top of the ampoule. Exercise precaution as the edges may be sharp.** The minimum sample size for each use should be about 1.0 g. Once the ampoule is opened, the contents should be used immediately.

Storage

The material should be stored at 18 to 25 °C in its original container. 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 provided 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 Record

Certificate Ref. No.	Date of issue	Reason for issuance
CML-HRM-2016A/01	31 May 2021	Issuance of first certificate
CML-HRM-2016A/02	14 April 2022	Extension of expiry date
CML-HRM-2016A/03	22 May 2023	Extension of expiry date
CML-HRM-2016A/04	29 May 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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