

Certificate of Analysis

CERTIFIED REFERENCE MATERIAL HRM – 1019A

Aspartame

Batch Number

STY-0090-001

Description

A unit of the certified reference material (CRM) consists of 200 mg of aspartame in a screw-capped amber vial. Quantitative nuclear magnetic resonance (qNMR) approach was adopted to determine the mass fraction (mg/g) of the reference material using an acesulfame potassium CRM (HRM-1012A) from the Health Sciences Authority (HSA), Singapore as internal standard.

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 Value

A certified value is a value for which a laboratory has the highest confidence in its accuracy. The certified mass fraction value given below is based on the results obtained by the qNMR approach:

Certified Mass Fraction Value: 970.7 ± 6.3 mg/g

The final result 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 the 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, purity of the internal standard, molecular weight of aspartame, molecular weight of internal standard and weighing, in accordance with ISO/IEC Guide 98-3:2008 [4].

Homogeneity

Homogeneity testing on aspartame was performed on two sub-samples taken from nine bottles (a total of 18 samples) using qNMR. The sample size taken for homogeneity testing was approximately 20 mg. 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

The short term stability of aspartame was studied. The material was stored at 50 °C (maximum allowable transportation temperature) for up to 14 days. This was carried out as “isochronous measurements”, i.e. all samples of the stability study were analysed under repeatability conditions. The results showed that aspartame was stable over the study period.

The long term stability of aspartame was evaluated at 25°C on three occasions for up to 3 months. The results showed that aspartame 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 the specified measurement uncertainty until **27 Jul 2026**, provided that the reference material is subjected to the same handling and storage conditions as stated in this *Certificate of Analysis* (COA). The validity of the certified mass fraction value has been confirmed using a 500 MHz NMR (Bruker Avance Ascend 500) at the Chemical Metrology Laboratory, HSA.

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 if the property value of the CRM is found to have changed or an updated COA may be issued.

Analytical Methods

The determination of the purity of the aspartame CRM was previously carried out using a 500 MHz Bruker Ultra Shield NMR at the Department of Chemistry, National University of Singapore. Acesulfame potassium CRM (HRM-1012A) from HSA was used as the internal standard for the determination. The certified mass fraction was calculated from the mean of 18 results obtained from one determination each on the 18 sub-samples prepared from the CRM and the internal standard.

Metrological Traceability

The certified mass fraction is traceable to the International System of Units (SI) through the use of acesulfame potassium CRM from HSA.

Intended Use

The CRM is intended for use as a calibrant for the determination of aspartame.

Instructions for Use

Prior to use, the material should be equilibrated to room temperature and rotated gently before sampling. The minimum sample size for each use should be 20 mg. After use, the bottle must be tightly re-capped and protected from moisture and light to ensure the validity of the certified value. If results differ from certified value in subsequent sampling, customers are advised to purchase a new CRM.

Storage

The CRM should be properly sealed and stored at a temperature of 18°C to 25 °C in its original bottle. Exposure to moisture and light 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, ingestion or inhalation of dust.

Further Information

Please direct all enquiries regarding this CRM to the contact above.

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] 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-1019A/01	27 Jul 2017	Issuance of first certificate
CML-HRM-1019A/02	26 Jun 2018	Extension of expiry date
CML-HRM-1019A/03	04 Jun 2019	Extension of expiry date
CML-HRM-1019A/04	07 Jul 2020	Extension of expiry date
CML-HRM-1019A/05	26 Jul 2021	Extension of expiry date
CML-HRM-1019A/06	03 Jun 2022	Extension of expiry date
CML-HRM-1019A/07	21 Jun 2024	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.



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