

Certificate of Analysis

CERTIFIED REFERENCE MATERIAL HRM-1002A

Benzoic Acid

Batch Number

STY-0035-002

Description

A unit of the certified reference material consists of 5 g of benzoic acid in a screw-capped amber vial. The mass balance approach was adopted to determine the mass fractions (mg/g) of four classes of impurities; namely: structurally-related organic compounds, volatile organic compounds, total non-volatiles and water content, present in the reference material. The mass fraction value of benzoic acid was then obtained by subtracting the mass fraction values of the impurities from 1,000.

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 mass balance approach.

Certified Mass Fraction Value: 999.7 ± 3.2 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). It was evaluated by combining uncertainties in the measurement of mass fractions of the four classes of impurities and the homogeneity and stability of the reference material, in accordance with ISO/IEC Guide 98-3:2008 [4].

Homogeneity

Homogeneity testing on benzoic acid and structurally-related organic compounds was performed on two sub-samples taken from eleven bottles using HPLC-DAD. The sample size taken for homogeneity testing was approximately 5 mg to produce a solution with benzoic acid concentration of about 2,800 mg/kg. The solutions were diluted to about 950 mg/kg. 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.

Stability

The short-term and long-term stability of benzoic acid and structurally-related organic compounds were evaluated based on prior experience from previous batch of benzoic acid. For short-term stability, the material was demonstrated to be stable at 40 °C (maximum allowable transportation temperature) for up to 14 days. For long-term stability, the material was demonstrated to be stable for three years at room temperature (18 °C to 25 °C).

Validity of Certified Mass Fraction Value

The certified mass fraction value is valid within the specified measurement uncertainty until **19 Jul 2028**, provided that the reference material is subjected to the same handling and storage conditions as stated below.

The reference material 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 Methods

The sample was analysed by

- (i) high performance liquid chromatography with diode array detection (HPLC-DAD) to determine the structurally-related organic compounds. Gas chromatography with flame ionisation detection (GC-FID) was used to confirm the content of structurally-related organic compounds and for estimation of measurement uncertainty;
- (ii) headspace gas chromatography with mass spectrometry (GC-MS) and thermogravimetric analyser (TGA) to determine the total volatile organic compounds;
- (iii) TGA to determine the total non-volatiles; and
- (iv) Karl Fisher coulometer to determine the water content.

The Table below summarises the results obtained from the above determinations:

Component(s)	Technique	Mass Fraction (mg/g)	Standard Uncertainty (mg/g)
Structurally-related organic compounds	HPLC-DAD	0.036	0.232
Total volatile organic compounds	TGA	< 2.3 (LOD*)	0.66
Total non-volatiles	TGA	< 5 (LOD*)	1.44
Water	Karl Fisher coulometry ¹	0.27	0.24

*LOD: limit of detection

¹ Validated with water saturated 1-octanol (SRM 2890) from NIST, USA.

Metrological Traceability

The certified mass fraction value is traceable to the International System of Units (SI) through the mass balance method by the Health Sciences Authority (HSA).

Intended Use

The reference material is intended for use as a calibrant for the determination of benzoic acid or quality control (QC).

Instructions for Use

After use, the bottle must be tightly re-capped and protected from moisture and light. The minimum sample size for each use should be 5 mg.

Storage

The reference material should be properly sealed and stored at room temperature (18 °C to 25 °C) in a dry and cool area in its original bottle when not in use. Protect the material from moisture and light.

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 reference material 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 of 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-1002A/05	19 Jul 2017	Issuance of first certificate for 2 nd batch
CML-HRM-1002A/06	05 Sep 2017	Revision on certified value
CML-HRM-1002A/07	21 May 2019	Extension of expiry date
CML-HRM-1002A/08	14 May 2021	Extension of expiry date
CML-HRM-1002A/09	05 May 2022	Extension of expiry date
CML-HRM-1002A/10	14 Jul 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.



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