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Ref. No.: CML-HRM-1028A/05
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Certificate of Analysis

CERTIFIED REFERENCE MATERIAL
HRM-1028A

Non-Volatile Inorganics in n-Butyl Paraben (for Thermogravimetric Analysis)

Batch Number
STY-0113-001

Description
A unit of the certified reference material consists of 1 g of n-butyl paraben fortified with a non-volatile compound. The material was bottled in a screw-capped amber glass bottle.

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. The certified mass fraction value for total non-volatiles (TNV) given below was determined using thermogravimetric analysis (TGA) method.

Certified Mass Fraction Value: 4.0 ± 1.6 mg/g

The final result 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, Type B uncertainty of the TGA microbalance and method recovery, in accordance with ISO/IEC Guide 98-3:2008 [4].

Homogeneity

Homogeneity testing on TNV content in n-butyl paraben was performed on two sub-samples taken from seven bottles using TGA method. The sample size taken for homogeneity testing was approximately 30 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 TNV content in n-butyl paraben was studied. The material was stored at 50 °C (maximum allowable transportation temperature) for up to 20 days. The results showed that TNV content was stable over the study period.

The long-term stability of TNV content in n-butyl paraben at room temperature (18 °C to 25 °C) was evaluated on five occasions over a period of up to 12 months after preparation. The results showed that TNV content 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 **16 Oct 2026**, provided that the reference material 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 Methods

The CRM was prepared by fortifying n-butyl paraben with alumina. The certified mass fraction of TNV content in n-butyl paraben was determined by using TGA method. The assigned value for the TNV content was calculated by averaging the measurement results from six subsamples taken from three bottles (i.e. two subsamples from each bottle). Each subsample (about 30 mg) of the material was placed in a TGA crucible and gravimetrically weighed using a 6-decimal place balance before and after the ashing by TGA. The results were determined from the percentage weight difference of the TGA crucible and sample before and after the ashing by TGA. The TGA was validated using another n-butyl paraben CRM and an in-house quality control material.

A thermogravimetric analyser (Mettler Toledo TGA/DSC 3+) was employed for the determination of TNV content in n-butyl paraben. The operating parameters of TGA were as follows:
Under nitrogen flow, heat from 30 °C to 100 °C at 5 K/min, heat to 900 °C at 50 K/min then hold for 10 min. Switch to oxygen flow at 900 °C and hold for 10 min.

Metrological Traceability

The certified mass fraction value is traceable to the International System of Units (SI) through the use of reference weights calibrated by the National Metrology Centre, A*STAR.

Intended Use

For the validation of TGA methods or as quality controls used in the determination of the mass fraction of TNV content in an organic compound using TGA. Similar operating parameters of TGA (e.g. reagent gas, temperature program) may need to be used to obtain the certified mass fraction value.

Instructions for Use

After use, the bottle must be tightly re-capped. The minimum sample size for each use should be 30 mg. 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 room temperature (18 °C to 25 °C) in a dry and cool area in its original bottle when not in use.

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 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-1028A/01	16 Oct 2020	Issuance of first certificate
CML-HRM-1028A/02	28 Oct 2021	Extension of expiry date
CML-HRM-1028A/03	14 Sep 2022	Extension of expiry date
CML-HRM-1028A/04	10 May 2023	Addition of TGA experimental conditions and the intended use
CML-HRM-1028A/05	5 Sep 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.

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