

## *Certificate of Analysis*

### **CERTIFIED REFERENCE MATERIAL HRM-1022A**

#### **n-Propyl Paraben**

#### **Batch Number**

STY-0112-001

#### **Description**

A unit of the certified reference material consists of 250 mg of n-propyl paraben in a screw-capped amber bottle. 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 n-propyl paraben 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:  $999.2 \pm 3.2$  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 n-propyl paraben and structurally-related organic compounds was performed on two sub-samples taken from eleven bottles using high performance liquid chromatography with diode array detection (HPLC-DAD). The sample size taken for homogeneity testing was approximately 5 mg to produce a solution with n-propyl paraben concentration of 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 stability of n-propyl paraben and structurally-related organic compounds was studied. The material was stored at 50 °C (maximum allowable transportation temperature) for up to 15 days. The results showed that n-propyl paraben was stable over the study period.

The long-term stability of n-propyl paraben at room temperature (18 °C to 25 °C) was evaluated on three occasions over a period of 3 months after preparation. The results showed that n-propyl paraben was stable over the study period.

## Validity of Certified Mass Fraction Value

The certified mass fraction value is valid within the specified measurement uncertainty until **28 Aug 2028**, 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 sample was analysed by

- (i) 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 evaluation 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 Fischer coulometer to determine the water content.

The Table below summarises the results used for the determination of the certified mass fraction value:

Component(s)	Technique	Mass Fraction (mg/g)	Standard Uncertainty (mg/g)
Structurally-related organic compounds	HPLC-DAD	0.36	0.12
Total volatile organic compounds	TGA	< 2.3 (LOD*)	0.66
Total non-volatiles	TGA	< 5 (LOD*)	1.44
Water	Karl Fischer coulometry <sup>1</sup>	0.395	0.087

\*LOD: limit of detection

<sup>1</sup> 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 n-propyl paraben or quality control (QC).

### **Instructions for Use**

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

### **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 Records**

Certificate Ref. No.	Date of issue	Reason for issuance
CML-HRM-1022A/01	28 Aug 2018	Issuance of first certificate
CML-HRM-1022A/02	4 Jul 2019	Extension of expiry date
CML-HRM-1022A/03	23 Jul 2020	Extension of expiry date
CML-HRM-1022A/04	29 Jun 2021	Extension of expiry date
CML-HRM-1022A/05	11 Aug 2023	Extension of expiry date
CML-HRM-1022A/06	22 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