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Ref. No.: CML-HRM-1026A/04
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Certificate of Analysis

CERTIFIED REFERENCE MATERIAL
HRM-1026A

4-Cumylphenol

Batch Number
STY-0131-001

Description
A unit of the certified reference material (CRM) consists of 1 g of 4-cumylphenol in a screw-capped amber bottle. The mass balance and quantitative nuclear magnetic resonance (qNMR) approaches were adopted to determine the mass fraction (mg/g) of 4-cumylphenol. For the mass balance approach, four classes of impurities; namely: structurally-related organic compounds, volatile organic compounds, total non-volatiles and water content, present in the reference material were determined. The mass fraction (mg/g) value of 4-cumylphenol was then obtained by subtracting the mass fraction values of the impurities from 1,000 mg/g. For the qNMR approach, the mass fraction (mg/g) of 4-cumylphenol was determined using a dimethyl sulfone CRM from the National Measurement Institute (NMI, Australia) as internal standard.

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 given below is an arithmetic mean based on the results obtained from mass balance and qNMR approaches.

Certified Mass Fraction Value: 996.6 ± 6.0 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 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 mass balance and qNMR approaches. The uncertainty from mass balance approach was evaluated by combining uncertainties in the measurement of mass fractions of the four classes of impurities, while the uncertainty from the qNMR approach was evaluated by combining uncertainties from method precision, purity of the internal standard, molecular weight of 4-cumylphenol, molecular weight of internal standard and weighing, in accordance with ISO/IEC Guide 98-3:2008 [4].

Homogeneity

Homogeneity testing on 4-cumylphenol and structurally-related organic compounds was performed on two sub-samples taken from ten 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 4-cumylphenol concentration of about 2,000 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 4-cumylphenol and structurally-related organic compounds was studied. The material was stored at 50 °C (maximum allowable transportation temperature) for up to 14 days. The results showed that 4-cumylphenol was stable over the study period.

The long-term stability of 4-cumylphenol at room temperature (18 °C to 25 °C) was evaluated on three occasions up to about 4 months after preparation. The results showed that 4-cumylphenol 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 **28 Sep 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

In mass balance method, the sample was analysed by

(i) HPLC-DAD to determine the structurally-related organic compounds;
(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 mass fraction value of 4-cumylphenol from mass balance method:

Component(s)	Technique	Mass Fraction (mg/g)	Standard Uncertainty (mg/g)
Structurally-related organic compounds	HPLC-DAD	0.48	0.008
Total volatile organic compounds	TGA	< 2.3 (LOD*)	0.66
Total non-volatiles	TGA	< 5 (LOD*)	1.44
Water	Karl Fischer coulometry[1]	0.29	0.18

*LOD: limit of detection
[1] Validated with water saturated 1-octanol (SRM 2890) from NIST, USA.

In qNMR method, the determination of the purity of the 4-cumylphenol CRM was carried out using a 500 MHz NMR (Bruker Avance Ascend 500) at the Chemical Metrology Laboratory, Health Sciences Authority (HSA). Dimethyl sulfone CRM (QNMR002) from NMI, Australia was used as the internal standard for the determination. The mass fraction was calculated from the mean of five results obtained from one determination each on the five sub-samples prepared from the CRM and the internal standard.

Metrological Traceability
The certified mass fraction value is traceable to the International System of Units (SI) through the mass balance method by HSA and through the use of dimethyl sulfone CRM from NMI, Australia in the qNMR method.

Intended Use
The CRM is intended for use as a calibrant 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. 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-1026A/01	28 Sep 2020	Issuance of first certificate
CML-HRM-1026A/02	18 Aug 2021	Extension of expiry date
CML-HRM-1026A/03	08 Sep 2022	Extension of expiry date
CML-HRM-1026A/04	12 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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