

Certificate of Analysis

CERTIFIED REFERENCE MATERIAL HRM – 1024A

Benz[a]anthracene and Benzo[a]pyrene in Olive Oil

Batch Number

STY-0117-001

Description

The certified reference material (CRM) consists of 40 mL of olive oil fortified with benz[a]anthracene and benzo[a]pyrene. The material was bottled in an aluminium bottle and screw-capped under nitrogen.

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 Values

A certified value is a value for which a laboratory has the highest confidence in its accuracy, in that all known or suspected sources of biases have been investigated and accounted for. The certified mass fraction values for benz[a]anthracene and benzo[a]pyrene listed in the Table below were determined by gas chromatography-isotope dilution mass spectrometry (GC-IDMS).

	Certified Mass Fraction	Unit
Benz[a]anthracene	8.05 ± 0.48	µg/kg
Benzo[a]pyrene	9.85 ± 0.75	µg/kg

The mass fraction value 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, weighing, the concentration of calibration solution (which included the purity of the standard), bias using different GC columns, bias using different solid phase extraction (SPE) cartridges for clean-up, bias using different calibration methods (i.e. exact matching IDMS vs linear regression IDMS), bias using different sources of calibration standard and method recovery, in accordance with ISO/IEC Guide 98-3:2008 [4].

Homogeneity

Homogeneity testing on the analytes in the olive oil was performed on nine bottles with two sub-samples taken from each bottle. GC-IDMS was employed for the determination of all the analytes. The sample size taken for homogeneity testing was about 1.8 g. 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 the analytes in the material was studied. The material was stored at 50 °C (maximum allowable transportation temperature) for up to 13 days. The results showed that the analytes were stable over the study period.

The long-term stability of the analytes in the material at storage temperature (18 °C to 25 °C) was evaluated on three occasions over a period of up to 10 months after preparation. The results showed that the analytes were stable over the study period. The u_{stab} was evaluated from the standard error of the slope.

Validity of Certified Mass Fraction Values

The certified mass fraction values are valid within their respective measurement uncertainties until **19 Aug 2026**, provided that the CRM 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 values 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 certified mass fractions of benz[a]anthracene and benzo[a]pyrene in the material were determined by exact-matching GC-IDMS. CRM of benzo[a]pyrene from Health Sciences Authority (HSA) (HRM-1017A) and a reference material of benz[a]anthracene obtained from Sigma Aldrich, which was purity assessed in-house using quantitative proton nuclear magnetic resonance (qNMR) approach [with the use of a CRM of dimethyl sulfone from the National Measurement Institute (NMI, Australia) (QNMR002) as the internal standard] were used as calibration standards. Isotope-labelled $^{13}\text{C}_6$ -benz[a]anthracene and $^{13}\text{C}_4$ -benzo[a]pyrene from Cambridge Isotope Laboratories Inc. were used as the internal standards.

The calibration blends were prepared gravimetrically by mixing appropriate amount of calibration standard solutions and internal standard solutions. The sample blends were gravimetrically prepared by mixing of 1.8 g of olive oil and internal standard solutions. Quality control blends were also prepared and analysed concurrently. The sample blends and quality control blends were left to equilibrate at about 4 °C for overnight and/or at ambient temperature (18 – 25 °C) for at least 1 h before subjected to extraction and clean-up procedures. Each sample and quality control blends were bracketed with a series of calibration blends and analysed three times.

Metrological Traceability

The certified mass fraction values for benz[a]anthracene and benzo[a]pyrene are traceable to the International System of Units (SI) through the use of CRMs from NMI, Australia and HSA, respectively.

Intended Use

For the validation of methods or as quality controls used in the determination of the mass fraction of benz[a]anthracene and benzo[a]pyrene in edible oil and materials of similar matrix.

Instructions for Use

Prior to use, the material should be **thoroughly** mixed by shaking the bottle; The minimum sample size for the analysis should be about 1.8 g. The bottle should be recapped tightly immediately after use. If results differ from certified value in subsequent sampling, customers are advised to purchase a new CRM.

Storage

The CRM should be stored at room temperature (18 °C – 25 °C) in its original bottle. Exposure to direct intense light, ultraviolet radiation and other temperatures should be avoided.

Safety Precautions for Users

Treat the material as hazardous substance. Use appropriate work practices when handling the material, in order to avoid skin or eye contact or ingestion.

Further Information

Please direct all enquiries regarding this CRM to the contact provided in this COA.

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-1024A/01	19 Aug 2019	Issuance of first certificate
CML-HRM-1024A/02	23 Jul 2020	Extension of expiry date
CML-HRM-1024A/03	12 Jul 2021	Extension of expiry date
CML-HRM-1024A/04	24 Jun 2022	Extension of expiry date
CML-HRM-1024A/05	13 Aug 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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