Statement of measurement

4005

River Sediment - PAHs

Reference Material LGC6188

Assessed Values

Constituent	Number of laboratories	Assessed value[1] mg/kg	Uncertainty[1,2] mg/kg	Weight[3] g	k[4]
Phenanthrene	9	0.74	0.29	3	2.02
Anthracene	9	0.231	0.081	3	2.09
Fluoranthene	9	1.52	0.32	3	2.02
Pyrene	9	1.24	0.50	3	2.02
Chrysene	8	0.63	0.16	3	2.02
Benzo[*a*]anthracene	9	0.60	0.19	3	2.02
Benzo[*b*]fluoranthene	9	0.68	0.18	3	2.12
Benzo[*k*]fluoranthene	8	0.323	0.084	3	2.16
Benzo[*a*]pyrene	9	0.51	0.16	3	2.07
Dibenzo[*a,h*]anthracene	8	0.086	0.023	3	2.09
Benzo[*g,h,i*]perylene	9	0.35	0.12	3	2.03

Notes:

1. Results are expressed on a dry weight basis.
2. The uncertainty quoted is the half-width of the expanded uncertainty interval calculated using an appropriate coverage factor, providing a level of confidence of approximately 95 %.
3. Weight of sample taken for homogeneity assessment.
4. Coverage factor

Date of issue: September 2000
Latest amendment: January 2023

Signed: _____
Gill Holcombe (Mrs)
for the Government Chemist

Queens Road, Teddington, Middlesex, TW11 0LY, UK
Tel: +44 (0)20 8943 7000 • Fax: +44 (0)20 8943 2767 • www.lgcgroup.com

The following figures are provided for information and should be regarded as indicative values.

Indicative Value

Constituent	Content[1] (mg/kg)
Acenaphthylene	0.05
Acenaphthene	0.03
Fluorene	0.05
Indeno[1,2,3-*cd*]pyrene	0.4
Naphthalene	0.2

Property	Indicative value[2] (g/100 g)
Loss on drying	2.0

Note:

1. The values are expressed on a dry weight basis.
2. Loss on drying measured on triplicate 1 g portions at (105 ± 1) ºC for 2 hours.

Preparation

A river sediment was taken from a monitoring station lagoon on the river Elbe close to the Czech-German border. The material was allowed to settle and coarse filtered on site to remove large particles then transported to a laboratory for preparation. The material was air-dried at 40 ºC, manually crushed then ground in a ceramic ball-mill to a particle size of less than 100 µm. The material was sieved, homogenised, divided into 30 g sub-samples and packaged in amber glass bottles with screw caps. The bottled material was then radiation sterilised using a ^{60}Co source at a dose of 25 kGy.

The material was tested for homogeneity by analysing randomly selected samples for each analyte using an appropriate technique. The material was judged to be homogeneous, for a 3 g portion, as the variation between the samples tested was not significantly greater than the method variation.

The nature of the matrix and analytes is such that deterioration over the life of the material is not anticipated. However, the stability of LGC6188 will be monitored as part of the LGC reference material stability testing programme. Customers will be notified of any changes in composition within the period of validity of their Statement of Measurement.

Characterisation

The assessed and indicative values for this material were obtained by means of an interlaboratory exercise organised by LGC in 2018. Analytical methods were not specified by LGC for the requested analytes, but laboratories were asked to carry out a loss on drying determination and report results on a dry weight basis. Details of the analytical methods used for the characterisation of this material are contained in the following pages.

The consensus values for the PAHs are based on the mean of individual laboratory means in the interlaboratory study, after elimination of outlying results

The indicative value for loss on drying is based on the median value.

Summary of Analytical Techniques Used by Laboratories in the 2018 Interlaboratory Characterisation Study

The techniques used for the characterisation of each analyte in this material are showe below. Each laboratory used the same analytical method to determine all the requested PAHs.

Laboratory	Techniques used
Lab01	Sample extracted using sodium sulphate, Florisil and an extractant solution. GC-FID.
Lab02	EPA Method 8270C:1996, SR EN 15527:2008
Lab03	Extracted using methanol, acetone, hexane, and water. GC-MS.
Lab04	Liquid solid extraction followed by GC-MS
Lab05	Hexane extraction, silica gel purification and GC-MS
Lab06	Extracted into dichloromethane and hexane, analysed by GC-MS
Lab07	MW-GC-MS
Lab08	Extraction by Acetone/Hexane. GC-MS
Lab09	ISO 13877:1999. Soxhlet extraction for 5 hours with toluene. Extract concentrated and purified on an aluminium oxide column. The final extract was in acetonitrile.
Lab10	DCM Extraction followed by GC-MS

A loss on drying determination was carried out by participants concurrently with the main analysis on a separate portion of material. Triplicate 1 g portions were taken to determine the weight loss on drying at (105 ± 1) ºC for 2 hours.

Metrological Traceability

The assessed and indicative values are traceable to the physical and chemical standards used by the interlaboratory study participants.

Accreditation

The assessed values on this document are within LGC's scope of accreditation to ISO 17034.

Intended Use

This material is intended for use in the development, validation or quality control of analytical methods for the determination of the polyaromatic hydrocarbons (PAHs) in sediments. The material may also be applicable to other matrices where more closely matched reference materials are not available.

Instructions for use

Before taking a sub-sample for analysis, the contents should be allowed to equilibrate to (20 ± 5) ºC and then thoroughly mixed by inversion. A loss on drying determination should be carried out by determining weight loss on a portion of sample by heating (105 ± 1 °C for 2 hours), to enable the results to be expressed on a dry sample weight basis.

The minimum sample size recommended for PAH analysis is the weight taken for the homogeneity assessment, which in this case was 3 g.

Storage

The material should be stored at (5 ± 4) °C in its original bottle. After the first opening, the material jar should be closed tightly and stored in the refrigerator at (5 ± 4) °C.

Shelf Life

Provided the sample is stored unopened under the recommended conditions, the assessed values will remain valid for 12 months from the date of shipment.

Certificate Revision

This certificate was revised in April 2018 to clarify the advice for users regarding opened units.

In July 2020:

- The assessed and indicative values were amended to reflect the values obtained in a new interlaboratory study.
- Assessed values for Acenaphthene, Fluorene, Indeno[1,2,3-*cd*]pyrene and Naphthalene were moved to indicative values, because these analyte values had a relatively large uncertainty .
- A value for loss on drying was added.
- The storage temperature was moved from (20 ± 5) ºC to (5 ± 4) ºC.
- Instructions for use were revised as there was no need to transfer the sample to a different container.
- The intended use statement was revised.

In January 2023, the UKAS symbol was updated, and a paragraph added to clarify the values within scope of LGC's ISO 17034 accreditation.

Participants in the Interlaboratory Exercise

The number of participants results used in the calculation of the assessed values is given in the table on page 1. The following organisations took part in the most recent interlaboratory exercise:

CEAEQ-MDDELCC	Canada
ENVIRA Ingenieros Asesores, S.L.	Spain
Eurofins Omegam	Netherlands
i2 Analytical Limited	UK
Lajedo S.R.L. Environmental Laboratory	Romania
MetropoliLab Oy	Finland
Nicholls Colton Group Limited	UK
NLS Leeds Laboratory (Environment Agency)	UK
The Environmental Laboratory (ELAB) Limited	UK
Wessling Romania Targu Mures (Environmental Laboratory)	Romania

Reference

1. ISO Guide 33:2015 - Reference Materials – Good practice in using reference materials.

Unit Number	Date of Shipment

Legal notice

(revised March 2009)

The values quoted in this document are the best estimate of the true values within the stated uncertainties and based on the techniques described herein. No warranty or representation, express or implied, is made that the use of the product or any information, material, apparatus, method or process which is the subject of or referred to in this document does not infringe any third party rights. Further, save to the extent: (a) prohibited by law; or (b) caused by a party's negligence; no party shall be liable for the use made of the product, any information, material, apparatus, method or process which is the subject of or referred to in this document. In no event shall the liability of any party exceed whichever is the lower of: (i) the value of the product; or (ii) £500,000; and any liability for loss of profit, loss of business or revenue, loss of anticipated savings, depletion of goodwill, any third-party claims or any indirect or consequential loss or damage in connection herewith is expressly excluded.

Production of this reference material was supported under contract with the Department of Trade and Industry as part of the National Measurement System Valid Analytical Measurement Programme.