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CERTIFICATE OF ANALYSIS

ERM®-CC013a

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Polycyclic aromatic hydrocarbons in soil		
	Certified value [1]	Uncertainty [2]
Compound	Mass fraction in mg/kg	
Naphthalene	2.4	± 0.5
Fluorene	1.14	± 0.11
Phenanthrene	12.0	± 0.6
Anthracene	1.41	± 0.22
Fluoranthene	12.9	± 0.7
Pyrene	9.6	± 0.3
Benzo[*a*]anthracene	5.6	± 0.5
Chrysene	5.3	± 0.8
Benzo[*b*]fluoranthene	7.1	± 1.0
Benzo[*k*]fluoranthene	3.4	± 0.4
Benzo[*a*]pyrene	4.9	± 0.7
Benzo[*ghi*]perylene	4.6	± 0.5
Indeno[1,2,3-*cd*]pyrene	5.2	± 1.0

[1] The certified values are the means of six laboratory means using HPLC/DAD/F or GC/MS. The values are traceable to the SI (Système International d'Unités) via calibration using sufficiently pure substances.

[2] Estimated expanded uncertainty *U* with a coverage factor of *k*=2, corresponding to a level of confidence of about 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1995.

This certificate is valid for a period of 12 months beginning with the dispatch of the reference material from BAM. The minimum sample size for one determination is 3 g.

NOTE

The European Reference Material ERM®-CC013a replaces ERM®-CC013 and was produced and certified under the responsibility of Bundesanstalt für Materialforschung und –prüfung (BAM) according to the principles laid down in ISO Guide 35:2006 and the technical guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on the latter is available via the Internet (http://www.erm-crm.org).

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BAM Berlin
Department I
Analytical Chemistry;
Reference Materials
12200 Berlin, Germany

Prof. Dr. U. Panne
(Head of Department)

BAM Berlin
Division I.2
Organic Chemical Analysis;
Reference Materials
12200 Berlin, Germany

Prof. Dr. I. Nehls
(Head of Division)

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INDICATIVE VALUES

The mass fractions of acenaphthene (0.75 mg/kg), acenaphthylene (0.77 mg/kg), and dibenz[*ah*]anthracene (1.1 mg/kg) are given as not certified indicative values without an uncertainty statement.

DESCRIPTION OF THE SAMPLE

This reference material was produced from a loamy sandy soil sampled on a former gasworks site in the Berlin-Brandenburg area, Germany, after air drying and homogenisation. The anthropogenic contents of polycyclic aromatic hydrocarbons (PAHs) had been aged by weathering for decades.

The upper limit of particle size of the material is 63 µm. The reference material is provided in 100 ml brown glass bottles containing 81 g of soil. The screw caps contain PTFE-inserts and are sealed with shrinking foil.

The distribution of the certified PAHs in this reference material is sufficiently homogenous. Appropriate estimates for possible inhomogeneities hidden by the employed analytical method were included in the calculation of the standard uncertainties and confidence intervals (see chapter 3 of the certification report).

The initial stability study after storage of selected units of the reference material at different temperatures revealed a shelf life of several years when kept at –20°C. On storing the reference material at higher temperatures up to a maximum of 23°C, a deterioration of the content of some of the individual PAHs has to be taken into consideration. Therefore – starting with the date of sale of the reference material – the validity of the certificate expires after 12 months. Periodical investigations on the stability of this material will be carried out in order to keep this information up to date.

INTENDED USE

The intended purpose of reference material ERM®-CC013a is validation of analytical procedures for the determination of polycyclic aromatic hydrocarbons (PAH) in soil according to ISO 13877 [2] by HPLC and alternative procedures using GC/MS, and quality assurance in analytical laboratories.

PARTICIPANTS AND ANALYTICAL METHOD USED FOR CERTIFICATION

The certification exercise was performed by six independent operator/equipment combinations in the Division "Organic Chemical Analysis; Reference Materials" of BAM employing the following analytical procedures:

No.	Extraction method	Extraction solvent	Analytical method
1	PFE	Toluene	GC/MS
2	Soxhlet	Toluene	GC/MS
3	FBE	Toluene	GC/MS
4	US	Cyclohexane/acetone (1:1)	GC/MS
5	US	Acetonitrile	HPLC/DAD/F
6	PFE	Methanol	HPLC/DAD/F

PFE	Pressurized fluid extraction
US	Sonication extraction
FBE	Fluidized bed extraction
HPLC	High performance liquid chromatography
GC	Gas chromatography
MS	Mass spectrometry
DAD	Diode array detector
F	Fluorescence detector

Certified calibration substances, e. g. [1] were employed for the analytical determination of the contents of the PAHs. Details on the procedures for extraction and analysis can be found in the certification report.

SAFETY INFORMATION

This reference material contains hazardous compounds (PAHs) in mass fractions between 0.7 mg/kg and 13 mg/kg (Sum of 16 EPA-PAHs: 78 mg/kg).

Proper use of the reference material is essential for avoiding potential harm to the user.

It is strongly recommended to handle and dispose the reference material in accordance with the guidelines for hazardous materials legally in force at the site of end use and disposal.

INSTRUCTIONS FOR USE

Before withdrawing a sub-sample the bottle has to have reached room temperature. Thereafter, the bottle is to be closed tightly and stored at –20°C. The stability of the reference material is not affected by short periods of handling at ambient temperature during transport and use.

STORAGE

The reference material is to be stored at –20°C in its original bottle.

TECHNICAL REPORT

A detailed technical report describing the analytical procedures and the treatment of the analytical data used to certify material ERM®-CC013a is available on request from BAM.

LEGAL NOTICE

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REFERENCES

[1] Standard Reference Material 1647d, Priority Pollutant Polycyclic Aromatic Hydrocarbons (in Acetonitrile), National Institute of Standard & Technology

[2] E DIN ISO 13877:2000 – Determination of polycyclic aromatic hydrocarbons (PAH) – HPLC-Method

Supply of Reference Materials by Bundesanstalt für Materialforschung und –prüfung:

Richard-Willstätter-Straße 11, D-12489 Berlin, Germany

Phone: +49 30 8104 2061 e-mail: sales.crm@bam.de

Fax: +49 30 8104 1117 internet: www.bam.de

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