CERTIFICATE OF ANALYSIS

ERM®-CC013

Polycyclic aromatic hydrocarbons in soil

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Certified Values		
	Certified value [1]	Uncertainty [2]
Compound	Mass fraction in mg/kg	
Naphthalene	2.8	± 0.4
Acenaphthene	1.59	± 0.16
Fluorene	2.57	± 0.29
Phenanthrene	13.5	± 1.2
Anthracene	3.70	± 0.21
Fluoranthene	15.9	± 0.8
Pyrene	12.8	± 1.0
Benzo[*a*]anthracene	7.3	± 0.8
Chrysene	6.8	± 0.9
Benzo[*b*]fluoranthene	7.2	± 0.7
Benzo[*k*]fluoranthene	3.8	± 0.4
Benzo[*a*]pyrene	7.9	± 0.6
Dibenzo[*ah*]anthracene	1.7	± 0.3
Benzo[*ghi*]perylene	5.7	± 0.6
Indeno[1,2,3-*cd*]pyrene	5.3	± 0.7
Sum of PAH	99	± 5

[1] The certified values including the sum of PAH are the means of 13 laboratory means using HPLC/UV/DAD/F and 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 about *k*=2, corresponding to a level of confidence of 95 %, as defined in the Guide to the expression of uncertainty in measurement, ISO, 1993.

This certificate is valid until May 2005; this validity may be extended as further evidence of stability becomes available.

The minimum sample size for one determination is 2.5 g.

NOTE

European Reference Material ERM®-CC013 was originally certified as BAM-U013. It was produced and certified under the responsibility of Bundesanstalt für Materialforschung und –prüfung (BAM) according to the principles laid down in the technical guidelines of the European Reference Materials® co-operation agreement between BAM-LGC-IRMM. Information on these guidelines is available on the Internet (http://www.erm-crm.org).

Accepted as an ERM®, Berlin, 2004-04-14.

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DESCRIPTION OF THE SAMPLE

The intended purpose of reference material ERM®-CC013 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.
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 PAH content has been aged by weathering for decades.
The range of particle sizes of the soil material is (63 – 125) µm. The reference material comes in 100 ml brown glass bottles containing 75,0 g of soil. The screw caps contain PTFE-inlays and are sealed with shrinking foil.
The distribution of the majority of the certified PAH in this reference material is homogenous. In case of phenanthrene, chrysene, benzo[*k*]fluoranthene and benzo[*a*]pyrene, however, an analysis of variance revealed that their distribution in the matrix is to be regarded as (slightly) inhomogenous. Appropriate estimates for the inhomogeneity of these compounds were included in the calculation of the standard uncertainties, and confidence intervals (see chapters 4.2 and 7.3 of the certification report).
The 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 PAH has to be taken into consideration. Therefore – starting with the date of sale of the reference material – the validity of the certificate expires after 30 months. If periodical investigations on this material reveal any significant changes, all users known to BAM will be notified. If the stability of the reference material extends beyond the indicated period, a respective information will be published on the internet (http://www.bam.de/service/referenzmaterialien/).

PARTICIPANTS AND ANALYTICAL METHOD USED FOR CERTIFICATION

The following German laboratories participated in the certification exercise:

Laboratory	City
AUA, Agrar- und Umweltanalytik GmbH	Jena
BAM, BA für Materialforschung und –prüfung, (I.22)	Berlin
CBA GmbH	Böhlen
Dr.Blasy-Dr.Busse, NL der AGROLAB GmbH	Eching/Am.
GEO-data GmbH, DG für Geologie, Hydrogeologie, Umweltanalytik	Garbsen
G.E.O.S. Freiberg, Geotechnik-Erkundung-Oekologie-Sanierung	Tuttendorf
IUL, Industrie- und Umweltlaboratorium Vorpommern	Greifswald
LUA, Labor für Umweltanalytik GmbH	Schwerin
Porst&Partner, Labor für Umweltanalytik und Warenprüfung GmbH	Fürth
Ruhranalytik GmbH	Herne
SGS Controll-Co. m b H, Institut für Umweltschutzdienstleistungen	Wismar
Technische Akademie Wuppertal e.V., WBZ Wildau	Wildau
TÜV Süddeutschland, Bau und Betrieb GmbH	Duisburg

Ann.: Laboratories listed in alphabetical order.

The following procedures were employed by the participating laboratories (general overview):

Extraction method	Extraction solvent	Analytical method
ASE	Methanol	HPLC-UV/F
	Cyclohexane	GC-MS
US	Acetonitrile	HPLC-DAD/F
	Tetrahydrofurane	HPLC-UV/F
	Cyclohexane	HPLC-DAD
	Acetone/Hexane	GC-MS
SOX	Toluene	HPLC-DAD/F
	Cyclohexane	HPLC-UV/F
	n-Hexane	GC-MS
KE-US	Acetonitrile	HPLC-UV/F

ASE™	Accelerated solvent extraction
KE	Cold extraction
SOX	Soxhlet extraction
US	Sonication extraction
HPLC	High performance liquid chromatography
GC	Gas chromatography
MS	Mass spectrometry
DAD	Diode array detector
F	Fluorescence detector
UV	Ultraviolet detector

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

SAFETY INFORMATION

This reference material contains hazardous compounds (PAH) in mass fractions between 1.6 mg/kg and 16.0 mg/kg (Sum of 16 PAH: 99 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 subsample the bottle has to have reached room temperature. Thereafter, the bottle is to be closed tight 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 (paper copy; in German) describing the analytical procedures and the treatment of the analytical data used to certify material ERM®-CC013 is available on request from BAM.

REFERENCES

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

[2] E DIN ISO 13877: 06.95 – Determination of polycyclic aromatic hydrocarbons (PAH) – HPLC-Method

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

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