Certificate of Analysis

PAHs - Clay Soil

Certified Reference Material

Product ID CRM170-50G

Lot LRAB3196

Expiration DateDecember 31, 2019Storage ConditionsStore at 4°C

Analyte	Units	Certified ^{1,4} Value	k 5	Standard ² Deviation	Confidence Interval	Prediction Interval
Naphthalene	ug/Kg	665 ± 107	2.00	158	558 to 772	309 to 1,020
Acenaphthene	ug/Kg	523 ± 113	2.00	167	424 to 622	151 to 895
Acenaphthylene	ug/Kg	662 ± 111	2.00	164	549 to 775	293 to 1,030
Anthracene	ug/Kg	149 ± 27.2	2.00	42.6	121 to 176	54.4 to 243
Benzo(a)anthracene	ug/Kg	145 ± 23.8	2.00	35.1	123 to 168	66.7 to 224
Benzo(a)pyrene	ug/Kg	214 ± 43.8	2.00	64.6	171 to 257	68.6 to 359
Benzo(b)fluoranthene	ug/Kg	225 ± 41.3	2.00	60.9	183 to 268	87.8 to 363
Benzo(g,h,i)perylene	ug/Kg	267 ± 49.3	2.00	72.7	219 to 316	104 to 431
Benzo(k)fluoranthene	ug/Kg	89.2 ± 16.1	2.00	23.7	72.7 to 106	35.7 to 143
Chrysene	ug/Kg	86.8 ± 13.1	2.00	19.4	73.6 to 130.0	43.2 to 130
Dibenzo(a,h)anthracene	ug/Kg	340 ± 61.1	2.00	90.1	279 to 401	137 to 542
Fluoranthene	ug/Kg	478 ± 88.9	2.00	139	388 to 567	169 to 786
Fluorene	ug/Kg	350 ± 80.8	2.00	119	270 to 430	81.9 to 618
Indeno(1,2,3-cd) pyrene	ug/Kg	152 ± 39.2	2.00	57.7	116 to 187	22.8 to 281
Phenanthrene	ug/Kg	588 ± 139	2.00	218	454 to 722	107 to 1,070
Pyrene	ug/Kg	337 ± 50.9	2.00	79.6	276 to 398	157 to 516

Sample Information

DESCRIPTION

The organic sample is a soil containing extractable PAHs for analysis by 8100, 8270, 8310 or equivalent methods.

This product consist of a 5 vials each containing 10g of soil for analysis of PAHs. Each vial is identical and has been tested show homogeneity. Only one vial is need for test the remaining vials are to be used for multiple methods or routine testing.

The soil has been sterilized to minimize degradation of the sample.

The sample has been sized to 100 mesh.

The sample has been intentionally prepared with an apparent headspace.

STORAGE

The sample should be stored at 4°C. It has been determined to be stable for the duration of the expiration date.

The shelf life of the product was determined by historic stability of similar CRM's. The expiration date may be extended based on stock and popularity upon successful stability testing by a 17025 accredited laboratory.

Stability and shelf life after opening must be determined by the user, taking into account sampling frequency/volume and all local conditions.

SAMPLE PREPARATION

Extract the complete contents of a single vial. Transfer entire contents of one vial to extraction vessel. Rinse vial and cap with extraction solvent.

Assume a 10g sample size for all calculations.







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Note: Sample extracts and calibration solutions should be in the same solvent.

Report all results on a wet weight basis, do not correct for moisture.

NOTE for method 8100 and using a packed column gas chromatographic method or cannot adequately resolve the following may coelute in four pairs of compounds: anthracene and phenanthrene; chrysene and benzo(a)anthracene; benzo(b)fluoranthene and benzo(k)fluoranthene; and dibenzo(a,h)anthracene and indeno(1,2,3-cd)pyrene.

SCOPE AND APPLICATION

The Polyaromatic Hydrocarbons (PAHs) in Soil Certified Reference Material (CRM) consists of a 5 amber glass sample vials, with a Teflon lined closure, each containing approximately 10 grams of soil, fortified with PAHs. Being a natural matrix waste sample the analyst is challenged by the same preparation problems, analytical interferences, etc. as is typical for similar matrices received by the laboratory for analysis. Rigorous analyses identified, quantified, and certified various aliphatic and aromatic banding which are listed on the enclosed Certificate of Analysis. The sample has been analyzed by a minimum of 18 independent laboratories in a round-robin to meet the requirements specified by the ISO Guides 34 and 35, and ISO 17025.

EVALUATION OF RESULTS

The Reference Value, 95% confidence interval(C.I.) for the Reference Value and 95% Prediction Interval (P.I.) around the Reference Value were obtained by the methods identified in the 'Scope and Application' section of this Certificate of Analysis. Samples were selected in a random fashion from the beginning to the end of the bottling sequence and sent for analysis by an independent laboratory round-robin. The data produced in the round-robin was used to calculate reference values by the USEPA EMSL-CINN's computer program "BIWEIGHT".

The generated BIWEIGHT mean, BIWEIGHT standard deviation and BIWEIGHT standard deviation of the mean are used to calculate the 95% Confidence Interval (CI) for the mean and the 95% Prediction Interval (PI). For normally distributed data, the BIWEIGHT 95% CI compares well to the classical calculation method used to generate a 95% CI. For non-Gaussian data sets, the BIWEIGHT method is more robust in data treatment.

BIWEIGHT data are also used to calculate a 95% PI. The 95% PI compares well to a 95% tolerance limit calculated using classical methods. For normally distributed data, the BIWEIGHT 95% PI typically represents approximately a ±2 BIWEIGHT standard deviation window around the BIWEIGHT mean. Again, the BIWEIGHT method is more robust than classical methods when handling non-Gaussian data sets.

Laboratories performing the same analytical procedures on a sample whose values have been determined by the BIWEIGHT method can assume that the true mean, as determined by the method, is within the 95% CI window. Laboratories analyzing the sample should have results within the 95% PI window 19 out of 20 analyses. Laboratories should use the PI as guidance for laboratory performance.

Additional information on the program may be obtained by referring to the reference or by downloading the program from the EMSL-CINN web site. Additionally contact MilliporeSigma for additional guidance - 1(307)742-5452 - www.sigmaaldrich.com

HEALTH AND SAFETY INFORMATION

All RTC Certified Reference Materials are intended only for professional use by properly trained laboratory personnel. This CRM has been reviewed for both health and safety and shipping risks. It is classified as non hazardous and is not classified as hazardous goods for shipping by road, sea or air transport.

A full international MSDS as a downloadable pdf file is available at www.SigmaAldrich.com

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2 The standard deviation is the robust statistical standard deviation from the round robin interlaboratory study.

3 Acceptance limits are based on Interlaboratory Study Results. These ranges are recommendations only.

4 Ucrm - Uncertainty values in this document are expressed as Expanded Uncertainty (Ucrm) corresponding to the 95% confidence interval. Ucrm is derived from the combined standard uncertainty multiplied by the coverage factor k, which is obtained from a *t*-distribution and degrees of freedom. The components of combined standard uncertainty include the uncertainties due to characterization, homogeneity, long term stability, and short term stability (transport). The components due to stability are generally considered to be negligible unless otherwise indicated by stability studies. The mathematical representation of the Ucrm calculation is as follows:

$$u_{CRM} = \sqrt{u_{char}^2 + u_{hom ogeneity}^2 + u_{stability}^2}$$

5 k: Coverage factor derived from a t-distribution table, based on the degrees of freedom of the data set. Confidence interval = 95%

Traceability: The standard was manufactured under an ISO/IEC 17025:2005 certified quality system. The balance used to weigh raw materials is accurate to +/- 0.0001g and calibrated regularly using mass standards traceable to NIST. All dilutions were preformed gravimetrically. Additionally, individual analytes are traceable to NIST SRMs where available and specified above.

Homogeneity: Homogeneity was assessed in accordance with ISO Guide 35. Completed units were sampled using a random stratified sampling protocol. The results of chemical analysis were then compared using a one-way analysis of variance approach as described by TNI EL-V3-2009 Appendix A.2. See Instructions for minimum sub-sample size.

THIS PRODUCT WAS DESIGNED, PRODUCED AND VERIFIED FOR ACCURACY AND STABILITY IN ACCORDANCE WITH ISO/IEC 17025:2005 (ANAB Cert AT-1467) and ISO GUIDE 34:2009 (ANAB Cert AR-1470).

Robert O'Brin

Robert O'Brien - QC Supervisor

Certification Date Version October 25, 2016 285-10252016

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Mark Pooler - QA Supervisor

LPTP11-S4





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¹ Certified value - based on the robust mean of round robin, interlaboratory study and analytically verified by RTC with associated uncertainties from the preparation and analytical procedures.