

Certificate of Analysis

Certified Reference Material



PAHs, PCBs, and Pesticides in Fresh Water Sediment

Number **CNS391-50G**

Lot 022142


Solvent (Matrix) Fresh Water Sediment

Hazard Irritant

Storage & Handling Store at 4°C.

Expiration Date See Sample Label

Certification Date: July 12, 2013

Certified By:  Christopher Rucinski - QA Director

Analyte	Units	Certified ^{1,4} Value	k ⁵	Standard ² Deviation	Confidence Interval	Prediction Interval
Naphthalene	µg/Kg	464 ± 39.9	2.00	118	413 - 515	310 - 618
Acenaphthene	µg/Kg	29.9 ± 6.43	2.00	19.0	21.0 - 38.8	3.16 - 56.7
Acenaphthylene	µg/Kg	53.4 ± 10.8	2.00	31.9	38.4 - 68.4	8.35 - 98.4
Anthracene	µg/Kg	15.0 ± 3.35	2.00	9.91	10.3 - 19.7	1.02 - 29.0
Benzo(a)anthracene	µg/Kg	338 ± 26.6	2.00	78.6	304 - 372	237 - 439
Benzo(a)pyrene	µg/Kg	38.2 ± 4.77	2.00	14.1	9.80 - 66.6	0.00 - 80.8
Benzo(b)fluoranthene	µg/Kg	210 ± 8.09	2.00	23.9	162 - 258	114 - 353
Benzo(g,h,i)perylene	µg/Kg	139 ± 10.1	2.00	29.7	126 - 152	101 - 177
Benzo(k)fluoranthene	µg/Kg	300 ± 11.6	2.00	34.4	285 - 215	256 - 344
Chrysene	µg/Kg	376 ± 13.1	2.00	38.8	359 - 393	326 - 426
Dibenzo(a,h)anthracene	µg/Kg	294 ± 11.8	2.00	34.9	279 - 309	248 - 340
Fluoranthene	µg/Kg	557 ± 29.5	2.00	87.1	519 - 595	443 - 671
Fluorene	µg/Kg	409 ± 42.3	2.00	125	355 - 461	248 - 568
Hexachlorobenzene	µg/Kg	34.5 ± 2.82	2.00	8.34	32.6 - 40.5	24.8 - 48.3
Indeno(1,2,3-cd) pyrene	µg/Kg	235 ± 12.0	2.00	35.4	220 - 250	189 - 281
Phenanthrene	µg/Kg	660 ± 34.5	2.00	102	616 - 704	529 - 791
Pyrene	µg/Kg	331 ± 31.6	2.00	93.4	291 - 371	211 - 451
Aldrin	µg/Kg	16.2 ± 1.34	2.00	3.95	14.3 - 18.0	10.6 - 21.8
alpha-Endosulfan	µg/Kg	14.2 ± 1.32	2.00	3.91	12.2 - 16.2	8.27 - 20.1
4,4'-DDD	µg/Kg	13.9 ± 0.985	2.00	2.91	12.6 - 15.3	9.82 - 18.0
4,4'-DDE	µg/Kg	18.8 ± 1.23	2.00	3.64	17.1 - 20.5	13.6 - 23.9
4,4'-DDT	µg/Kg	10.2 ± 1.27	2.00	3.74	8.40 - 11.9	4.88 - 15.4
Dieldrin	µg/Kg	25.7 ± 2.00	2.00	5.90	22.9 - 28.5	17.4 - 34.0
Endrin	µg/Kg	10.4 ± 2.14	2.00	6.31	7.30 - 13.5	1.03 - 19.8

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alpha-HCH	µg/Kg	37.1 ± 3.31	2.00	9.77	32.3 - 41.9	22.8 - 51.4
beta-HCH	µg/Kg	21.1 ± 2.05	2.00	6.05	18.2 - 24.1	12.3 - 30.0
gamma-HCH	µg/Kg	9.50 ± 0.721	2.00	2.13	8.50 - 10.5	6.39 - 12.6
Heptachlor	µg/Kg	6.54 ± 1.67	2.00	4.93	3.70 - 9.40	0.00 - 15.1
Heptachlor epoxide	µg/Kg	33.1 ± 2.40	2.00	7.08	29.5 - 36.7	22.4 - 43.8
2,4'-DDD	µg/Kg	15.5 ± 1.11	2.00	3.28	13.9 - 17.1	10.7 - 20.2
2,4'-DDE	µg/Kg	39.5 ± 2.39	2.00	7.07	35.8 - 43.3	28.3 - 50.8
2,4'-DDT	µg/Kg	43.0 ± 3.79	2.00	11.2	37.7 - 48.4	27.0 - 59.1
2,2',5,5'-Tetrachlorobiphenyl (PCB 52)	µg/Kg	64.6 ± 4.23	2.00	12.5	58.7 - 70.5	47.0 - 82.2
2,2',4,5,5'-Pentachlorobiphenyl (PCB 101)	µg/Kg	45.7 ± 3.13	2.00	9.24	41.4 - 50.0	32.7 - 58.7
2,3',4,4',5-Pentachlorobiphenyl (PCB 118)	µg/Kg	24.0 ± 1.31	2.00	3.87	22.2 - 25.8	18.6 - 29.5
2,2',3,4,4',5'-Hexachlorobiphenyl (PCB 138)	µg/Kg	34.6 ± 2.68	2.00	7.91	26.7 - 42.5	18.8 - 50.4
2,2',4,4',5,5'-Hexachlorobiphenyl (PCB 153)	µg/Kg	50.1 ± 2.59	2.00	7.65	42.4 - 57.8	34.8 - 65.4
2,2',3,4,4',5,5'-Heptachlorobiphenyl (PCB 180)	µg/Kg	54.7 ± 3.01	2.00	8.90	50.5 - 58.9	42.2 - 67.2
2,4,4'-Trichlorobiphenyl (PCB 28)	µg/Kg	44.9 ± 3.31	2.00	9.78	40.3 - 49.5	31.1 - 58.7

Additional Information

Description, Storage, and Handling

The sample size provided is 50g of soil.

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

The sample has been sized to 100 mesh.

Required storage condition is 4°C.

The sample has been intentionally prepared with an apparent headspace. After sampling replace cap immediately and return to the refrigerator.

Sample Preparation

The entire sample lot has been tested and certified for inter-sample homogeneity; due to potential settling and stratification in storage, shipping and handling the sample must be thoroughly mixed as stated in the method.

Recommended minimum sampling size is 1 gram.

Values are based on a dry weight basis.

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

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Scope and Application

The PAHs, PCBs, Pesticides on Soil Certified Reference Material (CRM) consists of a single amber glass sample jar, with a Teflon lined closure containing approximately 50 grams of soil, fortified with chlorinated pesticide compounds, PAHs and PCB congeners. 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 the compounds which are listed on the enclosed Certificate of Analysis. The sample has been analyzed by 30 independent laboratories in a round-robin to meet the requirements specified by the ISO Guides 34 and 35, and ISO 17025. The sample was certified by Dustch standard methods (NEN 5771, 5718, and 5719) and EPA extraction methods 3540/3541 and 3550A, followed by Method 8270. The sample is suitable for use by these and other similar methods.

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 to 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 RTC for additional guidance - 1(307)742-5452 - support@rt-corp.com - www.rt-corp.com

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- 1 Certified values are the robust statistical mean when prepared according to instructions from an Interlaboratory Study and internal rigorous testing.
- 2 The standard deviation is the robust statistical standard deviation from the round robin interlaboratory study.
- 4 Expanded Uncertainty (U_{crm}) - All uncertainty values in this document expressed as \pm value are expanded uncertainties.
- 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 17025 certified quality system. The balance used to weigh raw materials is accurate to ± 0.0001 g and calibrated regularly using mass standards traceable to NIST. All dilutions were performed gravimetrically. Additionally, individual analytes are traceable to NIST SRMs where available and specified above.

HOMOGENEITY ASSESSMENT: Between-bottle homogeneity was assessed in accordance with ISO Guide 35. Completed units were sampled over the course of the bottling operation. Samples were taken in the following manner: the units produced in the bottling operation were divided into three chronological groups, those from the Early third, the Middle third, and the Late third (Groups). A pre-determined number of sample units were then randomly selected from each group. A subset of each group was then randomly selected for chemical analysis. The results of the chemical analysis were then compared by Single Factor Analysis of Variance (ANOVA).

UNCERTAINTY STATEMENT: Uncertainty values in this document are expressed as Expanded Uncertainty (U_{crm}) corresponding to the 95% confidence interval. U_{crm} 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.

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

MSDS reports for components comprising greater than 1.0% of the solution or 0.1% for components known to be carcinogens are available upon request.

Manufactured and certified by Sigma-Aldrich RTC, Inc.

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