WAGENINGEN EVALUATING PROGRAMS

FOR ANALYTICAL LABORATORIES

Certificate of Analysis

International Sediment Exchange for Tests on Organic Contaminants

REFERENCE MATERIAL

SETOC sample 713

WAGENINGEN
UNIVERSITY & RESEARCH

Certificate of Analysis SETOC 713

General Information

In this report an overview is given of analytical data for this sample collected in our proficiency testing program. The consensus values are calculated using a robust statistical model. With this NDA model mean and standard deviation are calculated using all reported data when at least 8 results are left after removal of reported 'lower than' (<) and 0 (= zero) values. No outliers are removed.

This report is divided into three sections: Consensus Values, Indicative Values and Values for Information. The division is made on the reliability of the data. Consensus Values are based on at least 16 results while the coefficient of variation is smaller than 25 %. Indicative Values are based on at least 8 and less than 16 results or a coefficient of variation between 25 % and 50 %. Other values, based on more than 2 and less than 8 results or a coefficient of variation higher than 50 %, are given for information only.

In the sections with Consensus Values and Indicative Values the following parameters are given: mean, standard deviation, coefficient of variation, number of results, median and MAD (Median of Absolute Deviation) and the uncertainty in the consensus values. The confidence limits (at 95 % probability) are calculated for these determinands.
In the section with Information Values the following parameters are given: median, MAD and number of results. For determinands which have at least 5 results reported as smaller than (<) the median of these ‘smaller than results’ is calculated. In some cases this median of ‘<’ values is much smaller than median and mean of the indicative values. This may be caused by a too optimistic (too low) value for the detection limit reported by a (small) majority of participating laboratories who report ‘<’-values.

All values, expressed on a weight basis (kg or %), are reported in oven dry (105 °C) material. Moisture is reported in the material as received.

Sample information

WEPAL reference materials are from natural sources only. There is no spiking, mixing or other alterations of the samples. For sample preparation the SETOC samples are dried at 40 ^{o}C and milled to pass a 0.5 mm sieve.

This SETOC sample 713 of Marine Sediment from Netherlands is prepared for the WEPAL proficiency programs. The sample is used in 6 periods (or rounds). Only results from the last 5 periods are used. In this way the consensus values will reflect the latest 'state of the art' in the analytical techniques used in the laboratories. The results on which the values in this report are based were taken from the periods given in the following table.

Year	Round	Number
2001	2	4
1999	3	4
1998	1	1
1996	3	1
1995	4	3

Consensus Values SETOC 713

Method: Other parameters

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
AOX	mg/kg	33.2	7.20	21.7	26	34.1	5.15	1.77	30.2	-	36.1
Particles < 2 µm	%	7.08	1.046	14.8	46	7.02	0.700	0.193	6.77	-	7.39
Particles < 63 µm	%	17.0	1.86	10.9	17	16.7	1.30	0.56	16.0	-	17.9

Method: Metals

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
As	mg/kg	5.24	0.710	13.6	122	5.30	0.490	0.080	5.11	-	5.37
Cr	mg/kg	17.9	4.23	23.7	156	18.0	3.00	0.42	17.2	-	18.5
Cu	mg/kg	3.48	0.752	21.6	111	3.60	0.500	0.089	3.34	-	3.63
Ni	mg/kg	9.10	1.356	14.9	152	9.10	0.900	0.137	8.88	-	9.31
Pb	mg/kg	9.47	1.911	20.2	130	9.51	1.350	0.210	9.13	-	9.80
Zn	mg/kg	27.8	2.95	10.6	159	28.0	2.00	0.29	27.3	-	28.2

Indicative Values SETOC 713

Method: Polycyclic aromatic hydrocarbons

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
benz(a)anthracene	µg/kg	10.6	4.97	47.0	125	11.0	3.58	0.56	9.71 - 11.5
benzo(a)pyrene	µg/kg	11.2	2.90	25.9	158	12.0	2.00	0.29	10.7 - 11.6
benzo(b)fluoranthene	µg/kg	23.6	8.02	34.0	184	24.5	5.55	0.74	22.4 - 24.8
benzo(ghi)perylene	µg/kg	18.5	7.42	40.2	173	20.0	5.20	0.71	17.4 - 19.6
benzo(k)fluoranthene	µg/kg	11.6	3.23	27.9	138	12.0	2.21	0.34	11.0 - 12.1
chrysene	µg/kg	11.5	5.68	49.5	123	12.0	4.00	0.64	10.5 - 12.5
fluoranthene	µg/kg	19.5	7.00	35.9	191	20.0	5.00	0.63	18.5 - 20.5
indeno(1,2,3-cd)pyrene	µg/kg	20.6	7.60	36.9	169	20.7	5.30	0.73	19.4 - 21.7
pyrene	µg/kg	15.1	5.72	38.0	168	16.0	4.00	0.55	14.2 - 15.9

Method: Other parameters

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
Inorganic carbon	g/kg	5.11	1.419	27.8	15	5.30	0.900	0.458	4.33 - 5.89

Informative Values SETOC 713

Method: Polycyclic aromatic hydrocarbons

Element	Unit	Median	MAD	N	Results smaller than (<) Median of <	N
acenaphthene	µg/kg	7.84	6.650	48	20.00	204
acenaphthylene	µg/kg	1.485	1.2250	30	50.000	195
anthracene	µg/kg	2.92	1.870	70	10.00	207
dibenz(ah)anthracene	µg/kg	4.76	2.260	63	10.00	200
fluorene	µg/kg	3.81	2.335	64	10.00	194
naphthalene	µg/kg	10.7	5.26	88	50.0	175
phenanthrene	µg/kg	20.0	6.55	170	30.0	109

Method: Polychlorobiphenyls

Element	Unit	Median	MAD	N	Results smaller than (<) Median of <	N
PCB 028	µg/kg	0.680	0.3800	15	1.000	165
PCB 052	µg/kg	1.34	1.045	14	1.00	170
PCB 101	µg/kg	1.100	0.7500	22	1.000	165
PCB 105	µg/kg	0.255	0.1000	4	1.000	22
PCB 118	µg/kg	0.890	0.6700	15	1.000	138
PCB 128	µg/kg	-	-	0	1.00	23
PCB 138	µg/kg	1.000	0.6600	28	1.000	157
PCB 149	µg/kg	0.695	0.4100	6	1.500	16
PCB 153	µg/kg	1.050	0.5500	26	1.000	160
PCB 156	µg/kg	0.0300	0.0200	3	1.0000	21
PCB 180	µg/kg	1.000	0.7250	24	1.000	162

Method: Organochlorine pesticides

Element	Unit	Median	MAD	N	Results smaller than (<) Median of <	N
1,2,3 trichlorobenzene	µg/kg	-	-	0	2.00	15
1,2,3,4 tetrachlorobenzene	µg/kg	-	-	0	4.00	21
aldrin	µg/kg	-	-	0	1.00	104
alpha-endosulfan	µg/kg	3.65	3.390	4	1.00	98
alpha-HCH	µg/kg	-	-	0	1.00	114
beta-endosulfan	µg/kg	-	-	0	1.00	47
beta-HCH	µg/kg	2.30	0.250	4	1.00	109
cis-chlordane	µg/kg	-	-	0	1.00	32
delta-HCH	µg/kg	0.500	0.3500	3	1.000	64
dieldrin	µg/kg	5.00	3.970	3	1.00	106

endrin	µg/kg	-	-	0	1.00	107
gamma-HCH	µg/kg	5.14	4.420	3	1.00	109
heptachlor	µg/kg	0.400	0.1600	5	1.000	102

Method: Organochlorine pesticides — **Results smaller than (<)** (cont.)

Element	Unit	Median	MAD	N	Median of <	N
heptachlor epoxide	µg/kg	-	-	0	1.00	102
hexachlorobenzene	µg/kg	12.8	10.70	5	1.00	105
hexachlorobutadiene	µg/kg	-	-	0	1.00	45
o,p`-DDD	µg/kg	-	-	0	1.00	92
p,p`-DDD	µg/kg	-	-	0	1.00	107
p,p`-DDE	µg/kg	2.30	1.520	4	1.00	109
p,p`-DDT	µg/kg	3.50	2.680	6	1.00	107
pentachlorobenzene	µg/kg	-	-	0	2.00	57

Method: Other parameters — **Results smaller than (<)**

Element	Unit	Median	MAD	N	Median of <	N
CN - Free	mg/kg	1.50	1.300	3	1.00	32
CN - Total	mg/kg	0.200	0.1800	9	1.000	53
EOX	mg/kg	0.160	0.0700	63	0.100	61
Organic carbon	g/kg	3.33	1.370	32		
Mineral oil, GC	mg/kg	20.7	9.99	20	25.0	44
Mineral oil, IR	mg/kg	18.0	7.00	29	25.0	28
Particles > 63 µm	%	81.0	2.00	7		

Method: Metals — **Results smaller than (<)**

Element	Unit	Median	MAD	N	Median of <	N
Cd	mg/kg	0.0600	0.0400	27	0.3000	130
Hg	mg/kg	0.0300	0.0100	48	0.1000	99