WAGENINGEN EVALUATING PROGRAMS

FOR ANALYTICAL LABORATORIES

Certificate of Analysis

International Sediment Exchange for Tests on Organic Contaminants

REFERENCE MATERIAL

SETOC sample 688

WAGENINGEN
UNIVERSITY & RESEARCH

Certificate of Analysis SETOC 688

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 °C and milled to pass a 0.5 mm sieve.

This SETOC sample 688 of Sediment from Netherlands is prepared for the WEPAL proficiency programs. The sample is used in 3 periods (or rounds). The results on which the values in this report are based were taken from the periods given in the following table.

Year	Round	Number
2023	2	2
2020	4	4
2017	4	1

Consensus Values SETOC 688

Method: Polycyclic aromatic hydrocarbons

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
benz(a)anthracene	µg/kg	5930	1174	19.8	72	5880	812	173	5650 - 6201
benzo(a)pyrene	µg/kg	2400	420	17.5	74	2390	279	61	2306 - 2500
benzo(ghi)perylene	µg/kg	1930	259	13.4	74	1940	174	38	1869 - 1989
benzo(k)fluoranthene	µg/kg	1740	279	16.0	72	1770	195	41	1675 - 1806
chrysene	µg/kg	6260	831	13.3	73	6230	571	122	6067 - 6455
dibenz(ah)anthracene	µg/kg	606	138.9	22.9	55	605	95.3	23.4	568 - 643
fluoranthene	µg/kg	11600	1920	16.6	71	11500	1330	290	11150 - 12060
indeno(1,2,3-cd)pyrene	µg/kg	1970	336	17.1	73	1950	225	49	1889 - 2045
phenanthrene	µg/kg	21500	4900	22.8	73	21700	3340	720	20320 - 22610
pyrene	µg/kg	14000	2430	17.4	55	13800	1650	410	13350 - 14670
EPA ∑PAH(16)	µg/kg	82700	17690	21.4	37	79900	11750	3640	76810 - 88600

Method: Polychlorobiphenyls

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
PCB 118	µg/kg	10.9	2.69	24.7	63	10.9	1.90	0.42	10.2 - 11.6
PCB 138	µg/kg	11.8	2.72	23.1	66	12.0	1.89	0.42	11.1 - 12.4
PCB 153	µg/kg	13.4	3.02	22.5	63	13.5	2.07	0.48	12.7 - 14.2
∑PCB(7)	µg/kg	66.2	15.08	22.8	27	66.0	10.51	3.63	60.2 - 72.1

Method: Other parameters

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
CN - Total	mg/kg	3.04	0.391	12.9	20	3.11	0.277	0.109	2.86 - 3.22
Organic carbon	g/kg	24.0	2.40	10.0	36	24.2	1.70	0.50	23.2 - 24.8

Method: Metals

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
As	mg/kg	9.11	0.960	10.5	36	9.10	0.715	0.200	8.79 - 9.44
Ba	mg/kg	113	17.5	15.5	25	114	12.4	4.4	106 - 120
Cd	mg/kg	1.28	0.097	7.5	36	1.29	0.065	0.020	1.25 - 1.32
Co	mg/kg	4.33	0.310	7.1	29	4.35	0.211	0.072	4.21 - 4.45
Cr	mg/kg	28.3	3.32	11.7	36	28.1	2.25	0.69	27.2 - 29.5
Cu	mg/kg	37.8	3.40	9.0	36	38.0	2.40	0.71	36.7 - 39.0
Hg	mg/kg	0.656	0.0531	8.1	34	0.653	0.0356	0.0114	0.638 - 0.675
Mo	mg/kg	0.593	0.0693	11.7	21	0.600	0.0481	0.0189	0.561 - 0.624
Ni	mg/kg	15.0	0.94	6.2	36	15.1	0.63	0.20	14.68 - 15.31
Pb	mg/kg	107	9.6	8.9	35	107	6.6	2.0	104.1 - 110.7
Zn	mg/kg	320	27.6	8.6	35	320	19.0	5.8	311 - 330

Indicative Values SETOC 688

Method: Polycyclic aromatic hydrocarbons

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
acenaphthene	µg/kg	3210	1543	48.1	55	3200	1083	260	2790 - 3620
acenaphthylene	µg/kg	1180	434	36.6	54	1220	291	74	1065 - 1302
anthracene	µg/kg	2600	837	32.2	73	2680	564	122	2403 - 2794
benzo(b)fluoranthene	µg/kg	3250	931	28.6	51	3290	623	163	2991 - 3514
fluorene	µg/kg	2760	1129	40.9	56	2620	789	189	2457 - 3061

Method: Polychlorobiphenyls

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
PCB 028	µg/kg	3.67	1.476	40.2	50	3.84	1.087	0.261	3.25 - 4.09
PCB 052	µg/kg	6.29	2.979	47.4	63	6.20	2.012	0.469	5.54 - 7.04
PCB 101	µg/kg	13.9	3.66	26.3	66	13.9	2.51	0.56	13.0 - 14.8
PCB 105	µg/kg	3.48	0.625	17.9	11	3.60	0.450	0.235	3.07 - 3.90
PCB 128	µg/kg	2.44	0.443	18.2	9	2.50	0.330	0.185	2.10 - 2.77
PCB 156	µg/kg	1.24	0.115	9.3	9	1.29	0.090	0.048	1.15 - 1.32
PCB 180	µg/kg	5.32	1.495	28.1	59	5.34	1.032	0.243	4.93 - 5.71

Method: Organochlorine pesticides

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
1,2,4 trichlorobenzene	µg/kg	6.64	2.852	42.9	11	7.00	2.000	1.075	4.75 - 8.54
1,3,5 trichlorobenzene	µg/kg	1.78	0.431	24.2	10	1.87	0.313	0.170	1.48 - 2.09
delta-HCH	µg/kg	0.925	0.1138	12.3	11	0.914	0.0860	0.0429	0.849 - 1.00
hexachlorobenzene	µg/kg	0.844	0.2061	24.4	10	0.891	0.1505	0.0815	0.698 - 0.989
p,p`-DDD	µg/kg	5.36	2.473	46.1	17	5.75	1.797	0.750	4.09 - 6.62
p,p`-DDE	µg/kg	2.91	0.297	10.2	15	2.95	0.208	0.096	2.75 - 3.08
pentachlorobenzene	µg/kg	0.554	0.1242	22.4	8	0.581	0.0945	0.0549	0.453 - 0.655

Method: Other parameters

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits
Particles < 2 µm	%	4.73	1.582	33.4	9	4.88	1.120	0.659	3.54 - 5.92
Particles < 63 µm	%	14.5	3.41	23.5	12	14.6	2.45	1.23	12.3 - 16.6

Method: Polycyclic aromatic hydrocarbons

Element	Unit	Median	MAD	N
naphthalene	µg/kg	1570	694	72

Method: Polychlorobiphenyls

					Results smaller than (<)	
Element	**Unit**	**Median**	**MAD**	**N**	**Median of <**	**N**
PCB 031	µg/kg	3.00	-	5		
PCB 077	µg/kg	0.800	0.1500	3	1.500	10
PCB 081	µg/kg	-	-	0	1.00	10
PCB 114	µg/kg	-	-	0	1.00	9
PCB 123	µg/kg	1.10	0.220	5	1.00	6
PCB 149	µg/kg	10.6	1.57	7		
PCB 157	µg/kg	0.500	0.2500	3	1.000	8
PCB 167	µg/kg	1.48	0.780	6	2.00	5
PCB 169	µg/kg	-	-	0	1.00	11
PCB 189	µg/kg	1.45	0.635	6	2.00	5

Method: Organochlorine pesticides

					Results smaller than (<)	
Element	**Unit**	**Median**	**MAD**	**N**	**Median of <**	**N**
1,2,3 trichlorobenzene	µg/kg	0.409	0.2615	4	1.000	9
Sum trichlorobenzenes	µg/kg	11.1	2.60	3		
1,2,3,4 tetrachlorobenzene	µg/kg	-	-	0	1.00	9
Sum tetrachlorobenzenes	µg/kg	0.990	0.2210	3		
aldrin	µg/kg	-	-	0	4.00	25
alpha-endosulfan	µg/kg	-	-	0	5.00	24
alpha-HCH	µg/kg	0.190	0.0710	3	4.500	26
beta-endosulfan	µg/kg	-	-	0	10.0	9
beta-HCH	µg/kg	1.96	0.837	6	5.00	23
dieldrin	µg/kg	2.00	0.411	3	5.00	24
endrin	µg/kg	-	-	0	5.00	23
gamma-HCH	µg/kg	-	-	0	2.50	26
heptachlor	µg/kg	-	-	0	4.50	24
heptachlor epoxide	µg/kg	6.00	1.000	3	4.00	19
hexachlorobutadiene	µg/kg	-	-	0	1.00	18
o,p`-DDD	µg/kg	0.974	0.1350	4	5.000	23
o,p`-DDE	µg/kg	3.57	3.436	4	2.00	23

Method: Organochlorine pesticides

Element	Unit	Median	MAD	N	Results smaller than (<) Median of <	N
o,p`-DDT	µg/kg	1.07	0.977	6	1.00	22
p,p`-DDT	µg/kg	3.71	1.644	14	10.00	16

(cont.)

Method: Other parameters

Element	Unit	Median	MAD	N	Results smaller than (<) Median of <	N
CN - Free	mg/kg	2.24	0.623	4	1.00	14
EOX	mg/kg	0.700	0.2100	7		
Inorganic carbon	g/kg	3.97	0.650	3		
Mineral oil, GC	mg/kg	771	274.4	54		
Particles > 63 µm	%	77.0	2.45	7		

Method: Brominated Flame Retardants

Element	Unit	Median	MAD	N
BDE 047	ng/kg	453	308.0	3
BDE 099	ng/kg	244	119.8	3

Method: Experimental

Element	Unit	Median	MAD	N
Tributyl Tin (TBT)	µg/kg	6.60	0.400	7