



**WAGENINGEN EVALUATING PROGRAMS
FOR ANALYTICAL LABORATORIES**

Certificate of Analysis



International Sediment Exchange for Tests on Organic Contaminants

REFERENCE MATERIAL

SETOC sample 744



Certificate of Analysis SETOC 744

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 744 of Channel sludge 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
2002	4	3
2001	4	3
1998	4	4



Consensus Values SETOC 744



Method: Polychlorobiphenyls

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
PCB 118	µg/kg	28.1	6.26	22.2	93	28.0	4.14	0.81	26.9	-	29.4

Method: Other parameters

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Organic carbon	g/kg	64.6	5.99	9.3	26	65.2	4.05	1.47	62.1	-	67.0
Particles < 2 µm	%	17.9	0.99	5.5	23	17.9	0.70	0.26	17.5	-	18.3

Method: Metals

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
As	mg/kg	18.1	2.68	14.8	89	18.0	1.90	0.36	17.5	-	18.7
Cd	mg/kg	0.513	0.0721	14.1	74	0.510	0.0500	0.0105	0.496	-	0.530
Cr	mg/kg	56.2	7.96	14.1	96	56.0	5.40	1.01	54.6	-	57.8
Cu	mg/kg	43.9	3.45	7.9	95	44.0	2.40	0.44	43.2	-	44.6
Ni	mg/kg	25.3	2.61	10.3	95	25.0	1.80	0.33	24.7	-	25.8
Pb	mg/kg	32.4	3.78	11.7	96	32.5	2.55	0.48	31.6	-	33.1
Zn	mg/kg	328	29.2	8.9	97	331	20.0	3.7	323	-	334

Method: Dibenzo-P Dioxin

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
1,2,3,4,6,7,8 Cl7DD	ng/kg	17800	3060	17.2	29	17700	2140	710	16600	-	18920
1,2,3,6,7,8 Cl6DD	ng/kg	462	50.1	10.8	30	460	34.0	11.4	444	-	481
1,2,3,7,8,9 Cl6DD	ng/kg	64.0	8.19	12.8	30	64.8	5.75	1.87	60.9	-	67.0

Method: Dibenzofuran

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
1,2,3,4,6,7,8 Cl7DF	ng/kg	2330	313	13.4	29	2310	210	73	2212	-	2450
1,2,3,4,7,8 Cl6DF	ng/kg	43.2	7.79	18.0	29	45.0	6.00	1.81	40.2	-	46.1
1,2,3,4,7,8,9 Cl7DF	ng/kg	279	39.8	14.3	29	278	28.0	9.2	264	-	295
1,2,3,6,7,8 Cl6DF	ng/kg	28.6	7.15	25.0	29	29.0	5.00	1.66	25.9	-	31.3
2,3,7,8 Cl4DF	ng/kg	3.96	0.634	16.0	24	4.17	0.470	0.162	3.69	-	4.23
Cl8DF	ng/kg	8350	1669	20.0	29	8240	1155	387	7720	-	8990



Indicative Values SETOC 744

Method: Polycyclic aromatic hydrocarbons

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
benz(a)anthracene	µg/kg	74.9	22.50	30.1	161	77.0	16.00	2.22	71.4	-	78.4
benzo(a)pyrene	µg/kg	66.0	17.85	27.0	162	68.3	12.30	1.75	63.2	-	68.8
benzo(b)fluoranthene	µg/kg	125	33.2	26.5	148	130	23.5	3.4	120	-	130
benzo(ghi)perylene	µg/kg	66.6	24.02	36.1	146	70.0	16.85	2.48	62.7	-	70.5
benzo(k)fluoranthene	µg/kg	56.1	16.12	28.7	150	58.2	11.35	1.64	53.5	-	58.7
chrysene	µg/kg	108	31.0	28.8	163	110	21.0	3.0	103	-	112
fluoranthene	µg/kg	244	82.9	34.0	170	250	56.5	7.9	231	-	257
fluorene	µg/kg	21.8	10.00	45.9	111	23.0	7.00	1.19	19.9	-	23.7
indeno(1,2,3-cd)pyrene	µg/kg	74.6	23.85	32.0	155	78.0	16.60	2.39	70.8	-	78.3
phenanthrene	µg/kg	68.9	24.92	36.2	159	70.0	17.99	2.47	65.0	-	72.8
pyrene	µg/kg	157	50.3	32.0	159	159	35.0	5.0	149	-	165

Method: Polychlorobiphenyls

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
PCB 028	µg/kg	924	430.1	46.5	95	928	277.0	55.2	837	-	1012
PCB 052	µg/kg	319	85.9	26.9	106	321	59.0	10.4	303	-	336
PCB 101	µg/kg	31.3	8.46	27.0	106	31.6	5.69	1.03	29.7	-	32.9
PCB 105	µg/kg	19.1	5.14	26.8	26	18.9	3.15	1.26	17.1	-	21.2
PCB 138	µg/kg	10.9	3.20	29.4	99	11.0	2.20	0.40	10.3	-	11.5
PCB 149	µg/kg	7.31	2.578	35.3	13	8.00	1.800	0.894	5.76	-	8.85
PCB 153	µg/kg	11.0	2.88	26.1	101	11.0	2.00	0.36	10.4	-	11.6
PCB 156	µg/kg	0.973	0.1573	16.2	12	1.025	0.1250	0.0567	0.874	-	1.07
PCB 180	µg/kg	6.07	1.626	26.8	94	6.12	1.115	0.210	5.74	-	6.40

Method: Organochlorine pesticides

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
alpha-endosulfan	µg/kg	94.1	31.01	32.9	33	88.9	21.07	6.75	83.2	-	105
o,p'-DDE	µg/kg	8.02	2.892	36.1	17	9.10	1.900	0.877	6.54	-	9.50
p,p'-DDD	µg/kg	6.08	2.515	41.3	33	6.80	1.800	0.547	5.19	-	6.97
p,p'-DDE	µg/kg	33.7	11.97	35.6	54	33.4	8.00	2.04	30.4	-	36.9

Method: Other parameters

Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
AOX	mg/kg	94.8	8.88	9.4	11	94.0	6.00	3.35	88.9	-	101
EOX	mg/kg	10.6	3.95	37.1	66	10.7	2.62	0.61	9.67	-	11.6
Inorganic carbon	g/kg	3.88	1.239	31.9	13	3.80	0.900	0.430	3.14	-	4.62
Mineral oil, GC	mg/kg	142	49.8	35.2	72	151	35.0	7.3	130	-	153



Indicative Values SETOC 744

Method: Other parameters										(cont.)	
Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Particles < 63 µm	%	45.7	2.42	5.3	9	45.0	1.80	1.01	43.9	-	47.5
Method: Metals											
Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
Hg	mg/kg	0.138	0.0413	30.0	79	0.140	0.0300	0.0058	0.129	-	0.147
Method: Dibenzo-P Dioxin											
Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
1,2,3,4,7,8 Cl6DD	ng/kg	29.4	9.54	32.5	28	31.0	6.95	2.25	25.7	-	33.0
1,2,3,7,8 Cl5DD	ng/kg	5.12	1.477	28.9	26	5.24	0.990	0.362	4.52	-	5.72
2,3,7,8 Cl4DD	ng/kg	1.52	0.522	34.3	22	1.64	0.365	0.139	1.29	-	1.75
Cl8DD	ng/kg	287000	88700	30.9	26	302000	61300	21700	251060	-	322590
Method: Dibenzofuran											
Element	Unit	Mean	Std.Dev.	CV %	N	Median	MAD	Uncertainty	95 % confidence limits		
1,2,3,7,8 Cl5DF	ng/kg	2.83	0.990	35.0	23	3.00	0.710	0.258	2.40	-	3.26
2,3,4,6,7,8 Cl6DF	ng/kg	20.9	9.14	43.8	29	23.3	7.00	2.12	17.4	-	24.4



Informative Values SETOC 744



Method: Polycyclic aromatic hydrocarbons

Element	Unit	Median	MAD	N	Results smaller than (<)	
					Median of <	N
acenaphthene	µg/kg	10.00	5.000	68	45.00	88
acenaphthylene	µg/kg	6.50	3.820	44	50.00	97
anthracene	µg/kg	18.0	8.00	121	20.0	54
dibenz(ah)anthracene	µg/kg	20.0	6.90	109	30.0	50
naphthalene	µg/kg	16.1	6.52	73	50.0	93

Method: Polychlorobiphenyls

Element	Unit	Median	MAD	N	Results smaller than (<)	
					Median of <	N
PCB 128	µg/kg	2.20	0.850	13	3.00	8

Method: Organochlorine pesticides

Element	Unit	Median	MAD	N	Results smaller than (<)	
					Median of <	N
1,2,3 trichlorobenzene	µg/kg	-	-	0	2.00	9
aldrin	µg/kg	13.4	9.78	4	5.00	49
alpha-HCH	µg/kg	20.1	15.83	6	2.50	55
beta-endosulfan	µg/kg	50.0	30.75	14	1.00	12
beta-HCH	µg/kg	-	-	0	4.50	54
delta-HCH	µg/kg	-	-	0	1.50	38
dieldrin	µg/kg	3.00	1.445	4	5.00	49
endosulfan	µg/kg	-	-	0	10.0	5
endosulfan sulfate	µg/kg	11.8	3.75	5	2.00	13
endrin	µg/kg	3.91	1.000	4	5.00	47
gamma-HCH	µg/kg	1.17	0.515	4	3.00	54
heptachlor	µg/kg	11.32	4.225	8	5.00	43
heptachlor epoxide	µg/kg	-	-	0	5.00	45
hexachlorobenzene	µg/kg	2.00	1.070	13	4.00	45
isodrin	µg/kg	-	-	0	5.00	31
o,p`-DDD	µg/kg	5.90	4.330	13	5.00	34
o,p`-DDT	µg/kg	3.15	2.740	5	5.00	43
p,p`-DDT	µg/kg	3.95	2.940	16	5.00	41
pentachlorobenzene	µg/kg	-	-	0	5.00	30
trans-chlordane	µg/kg	-	-	0	1.50	20

Method: Other parameters

Results smaller than (<)



Informative Values SETOC 744

Element	Unit	Median	MAD	N	Median of <	N
CN - Free	mg/kg	0.380	0.1100	6	1.000	13
CN - Total	mg/kg	0.840	0.5600	21	1.000	16
Method: Other parameters					Results smaller than (<) (cont.)	
Element	Unit	Median	MAD	N	Median of <	N
Mineral oil, IR	mg/kg	130	50.0	51		
Particles > 63 µm	%	38.3	3.30	3		
Method: Dibenzofuran					Results smaller than (<)	
Element	Unit	Median	MAD	N	Median of <	N
1,2,3,7,8,9 Cl6DF	ng/kg	4.10	2.520	20	5.00	8
2,3,4,7,8 Cl5DF	ng/kg	4.00	1.425	26		