



**WAGENINGEN EVALUATING PROGRAMS
FOR ANALYTICAL LABORATORIES**

Certificate of Analysis



International Sediment Exchange for Tests on Organic Contaminants

REFERENCE MATERIAL

SETOC sample 717



Certificate of Analysis SETOC 717

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 717 of Marine 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 |
|------|-------|--------|
| 1998 | 1 | 2 |
| 1996 | 3 | 3 |
| 1994 | 3 | 2 |



Consensus Values SETOC 717

Method: Polycyclic aromatic hydrocarbons

| Element | Unit | Mean | Std.Dev. | CV % | N | Median | MAD | Uncertainty | 95 % confidence limits | | |
|----------------------|-------|------|----------|------|-----|--------|-------|-------------|------------------------|---|------|
| benzo(k)fluoranthene | µg/kg | 90.8 | 20.81 | 22.9 | 151 | 90.0 | 14.00 | 2.12 | 87.5 | - | 94.2 |
| fluoranthene | µg/kg | 283 | 65.2 | 23.1 | 167 | 290 | 45.0 | 6.3 | 273 | - | 293 |

Method: Other parameters

| Element | Unit | Mean | Std.Dev. | CV % | N | Median | MAD | Uncertainty | 95 % confidence limits | | |
|------------------|-------|------|----------|------|----|--------|------|-------------|------------------------|---|------|
| AOX | mg/kg | 41.0 | 6.67 | 16.3 | 18 | 40.1 | 4.42 | 1.97 | 37.7 | - | 44.3 |
| Particles < 2 µm | % | 10.0 | 1.47 | 14.7 | 25 | 10.0 | 1.00 | 0.37 | 9.41 | - | 10.6 |

Method: Metals

| Element | Unit | Mean | Std.Dev. | CV % | N | Median | MAD | Uncertainty | 95 % confidence limits | | |
|---------|-------|-------|----------|------|----|--------|--------|-------------|------------------------|---|-------|
| As | mg/kg | 15.7 | 1.96 | 12.5 | 84 | 15.7 | 1.37 | 0.27 | 15.3 | - | 16.1 |
| Cd | mg/kg | 1.76 | 0.175 | 9.9 | 94 | 1.79 | 0.115 | 0.023 | 1.73 | - | 1.80 |
| Cr | mg/kg | 47.2 | 7.36 | 15.6 | 94 | 47.1 | 4.90 | 0.95 | 45.7 | - | 48.7 |
| Cu | mg/kg | 19.8 | 1.64 | 8.3 | 93 | 19.9 | 1.10 | 0.21 | 19.44 | - | 20.11 |
| Hg | mg/kg | 0.440 | 0.0713 | 16.2 | 80 | 0.450 | 0.0500 | 0.0100 | 0.424 | - | 0.456 |
| Ni | mg/kg | 12.9 | 1.59 | 12.4 | 94 | 13.0 | 1.05 | 0.21 | 12.55 | - | 13.21 |
| Pb | mg/kg | 36.4 | 3.30 | 9.0 | 95 | 36.8 | 2.20 | 0.42 | 35.8 | - | 37.1 |
| Zn | mg/kg | 135 | 10.7 | 8.0 | 95 | 134 | 7.0 | 1.4 | 132.5 | - | 136.9 |



Indicative Values SETOC 717

Method: Polycyclic aromatic hydrocarbons

| Element | Unit | Mean | Std.Dev. | CV % | N | Median | MAD | Uncertainty | 95 % confidence limits | | |
|------------------------|-------|------|----------|------|-----|--------|-------|-------------|------------------------|---|------|
| acenaphthene | µg/kg | 20.0 | 9.21 | 45.9 | 76 | 21.5 | 6.64 | 1.32 | 17.9 | - | 22.1 |
| anthracene | µg/kg | 44.6 | 16.63 | 37.3 | 137 | 45.5 | 11.50 | 1.78 | 41.7 | - | 47.4 |
| benz(a)anthracene | µg/kg | 128 | 32.9 | 25.8 | 160 | 130 | 22.0 | 3.3 | 123 | - | 133 |
| benzo(a)pyrene | µg/kg | 126 | 35.5 | 28.2 | 161 | 128 | 24.0 | 3.5 | 120 | - | 131 |
| benzo(b)fluoranthene | µg/kg | 198 | 54.6 | 27.5 | 156 | 200 | 37.5 | 5.5 | 190 | - | 207 |
| benzo(ghi)perylene | µg/kg | 114 | 31.2 | 27.3 | 153 | 119 | 21.0 | 3.1 | 109 | - | 119 |
| chrysene | µg/kg | 125 | 41.9 | 33.6 | 159 | 129 | 29.0 | 4.2 | 118 | - | 131 |
| dibenz(ah)anthracene | µg/kg | 28.0 | 13.94 | 49.8 | 119 | 30.0 | 10.00 | 1.60 | 25.4 | - | 30.5 |
| fluorene | µg/kg | 28.4 | 12.87 | 45.3 | 107 | 30.0 | 9.00 | 1.55 | 26.0 | - | 30.9 |
| indeno(1,2,3-cd)pyrene | µg/kg | 131 | 43.8 | 33.5 | 149 | 130 | 30.0 | 4.5 | 124 | - | 138 |
| phenanthrene | µg/kg | 131 | 35.4 | 27.1 | 159 | 131 | 24.0 | 3.5 | 125 | - | 137 |
| pyrene | µg/kg | 229 | 64.5 | 28.1 | 157 | 221 | 44.6 | 6.4 | 219 | - | 240 |

Method: Polychlorobiphenyls

| Element | Unit | Mean | Std.Dev. | CV % | N | Median | MAD | Uncertainty | 95 % confidence limits | | |
|---------|-------|------|----------|------|----|--------|-------|-------------|------------------------|---|------|
| PCB 052 | µg/kg | 2.34 | 0.990 | 42.3 | 65 | 2.34 | 0.660 | 0.154 | 2.10 | - | 2.59 |
| PCB 101 | µg/kg | 3.59 | 1.347 | 37.6 | 79 | 3.59 | 0.910 | 0.189 | 3.29 | - | 3.89 |
| PCB 118 | µg/kg | 2.69 | 1.094 | 40.7 | 61 | 2.80 | 0.800 | 0.175 | 2.41 | - | 2.97 |
| PCB 138 | µg/kg | 4.61 | 1.529 | 33.2 | 84 | 4.80 | 1.050 | 0.208 | 4.28 | - | 4.94 |
| PCB 149 | µg/kg | 3.04 | 1.416 | 46.5 | 9 | 3.20 | 0.920 | 0.590 | 1.98 | - | 4.11 |
| PCB 153 | µg/kg | 5.01 | 1.602 | 32.0 | 85 | 5.00 | 1.100 | 0.217 | 4.66 | - | 5.35 |
| PCB 180 | µg/kg | 2.98 | 1.017 | 34.1 | 81 | 3.00 | 0.700 | 0.141 | 2.76 | - | 3.21 |

Method: Organochlorine pesticides

| Element | Unit | Mean | Std.Dev. | CV % | N | Median | MAD | Uncertainty | 95 % confidence limits | | |
|-------------------|-------|------|----------|------|----|--------|-------|-------------|------------------------|---|------|
| hexachlorobenzene | µg/kg | 1.14 | 0.444 | 39.0 | 22 | 1.18 | 0.315 | 0.118 | 0.941 | - | 1.33 |
| p,p'-DDD | µg/kg | 2.02 | 0.702 | 34.7 | 34 | 2.00 | 0.500 | 0.150 | 1.78 | - | 2.27 |
| p,p'-DDE | µg/kg | 1.44 | 0.597 | 41.5 | 33 | 1.41 | 0.410 | 0.130 | 1.23 | - | 1.65 |

Method: Other parameters

| Element | Unit | Mean | Std.Dev. | CV % | N | Median | MAD | Uncertainty | 95 % confidence limits | | |
|-------------------|-------|-------|----------|------|----|--------|--------|-------------|------------------------|---|-------|
| EOX | mg/kg | 0.441 | 0.1850 | 41.9 | 72 | 0.465 | 0.1350 | 0.0273 | 0.398 | - | 0.485 |
| Organic carbon | g/kg | 14.4 | 5.62 | 39.0 | 15 | 14.0 | 4.00 | 1.81 | 11.3 | - | 17.5 |
| Mineral oil, GC | mg/kg | 122 | 22.3 | 18.2 | 14 | 125 | 15.5 | 7.5 | 110 | - | 135 |
| Mineral oil, IR | mg/kg | 105 | 31.9 | 30.4 | 15 | 108 | 21.0 | 10.3 | 87.3 | - | 122 |
| Particles < 63 µm | % | 38.2 | 5.37 | 14.1 | 12 | 38.5 | 3.80 | 1.94 | 34.8 | - | 41.6 |



Indicative Values SETOC 717



Method: Polycyclic aromatic hydrocarbons

| Element | Unit | Median | MAD | N | Results smaller than (<) Median of < | N |
|----------------|-------|--------|-------|-----|---|----|
| acenaphthylene | µg/kg | 16.0 | 11.80 | 39 | 50.0 | 94 |
| naphthalene | µg/kg | 60.0 | 23.00 | 109 | 80.0 | 49 |

Method: Polychlorobiphenyls

| Element | Unit | Median | MAD | N | Results smaller than (<) Median of < | N |
|---------|-------|--------|--------|----|---|----|
| PCB 028 | µg/kg | 1.80 | 0.690 | 54 | 2.25 | 50 |
| PCB 031 | µg/kg | - | - | 0 | 1.00 | 7 |
| PCB 105 | µg/kg | 0.650 | 0.2100 | 7 | 2.000 | 5 |
| PCB 128 | µg/kg | 0.730 | 0.0300 | 5 | 1.000 | 10 |
| PCB 156 | µg/kg | 0.475 | 0.1150 | 4 | 1.000 | 9 |

Method: Organochlorine pesticides

| Element | Unit | Median | MAD | N | Results smaller than (<) Median of < | N |
|----------------------------|-------|--------|--------|----|---|----|
| 1,2,3 trichlorobenzene | µg/kg | - | - | 0 | 3.50 | 8 |
| 1,2,3,4 tetrachlorobenzene | µg/kg | 0.350 | 0.0800 | 3 | 3.500 | 12 |
| aldrin | µg/kg | - | - | 0 | 1.00 | 64 |
| alpha-endosulfan | µg/kg | - | - | 0 | 1.00 | 60 |
| alpha-HCH | µg/kg | 1.30 | 1.190 | 3 | 1.00 | 70 |
| beta-endosulfan | µg/kg | - | - | 0 | 1.00 | 25 |
| beta-HCH | µg/kg | 3.20 | 1.300 | 7 | 1.00 | 65 |
| cis-chlordane | µg/kg | - | - | 0 | 1.00 | 21 |
| delta-HCH | µg/kg | - | - | 0 | 1.00 | 38 |
| dieldrin | µg/kg | 2.00 | 0.530 | 7 | 1.35 | 62 |
| endosulfan | µg/kg | - | - | 0 | 5.00 | 5 |
| endosulfan sulfate | µg/kg | - | - | 0 | 1.00 | 21 |
| endrin | µg/kg | - | - | 0 | 1.00 | 66 |
| gamma-HCH | µg/kg | 1.000 | 0.4850 | 6 | 1.000 | 66 |
| heptachlor | µg/kg | - | - | 0 | 1.00 | 67 |
| heptachlor epoxide | µg/kg | 1.60 | 1.320 | 3 | 1.00 | 64 |
| hexachlorobutadiene | µg/kg | - | - | 0 | 1.00 | 25 |
| o,p`-DDD | µg/kg | 1.75 | 0.955 | 10 | 1.50 | 50 |
| o,p`-DDE | µg/kg | 1.45 | 0.710 | 6 | 1.00 | 53 |
| p,p`-DDT | µg/kg | 3.00 | 1.800 | 9 | 1.05 | 60 |
| pentachlorobenzene | µg/kg | 0.455 | 0.0800 | 4 | 2.500 | 32 |



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| | | | | | | |
|-----------------|-------|-------|--------|---|-------|----|
| telodrin | µg/kg | 0.680 | 0.2200 | 3 | 1.000 | 40 |
| trans-chlordane | µg/kg | - | - | 0 | 1.00 | 21 |



Indicative Values SETOC 717



Method: Other parameters

| Element | Unit | Median | MAD | N | Results smaller than (<) Median of < | N |
|-------------------|-------|--------|--------|----|---|----|
| CN - Free | mg/kg | - | - | 0 | 1.00 | 16 |
| CN - Total | mg/kg | 0.225 | 0.1250 | 14 | 1.000 | 22 |
| Inorganic carbon | g/kg | 12.0 | 2.00 | 7 | | |
| Particles > 63 µm | % | 52.2 | 1.00 | 3 | | |