



EUROPEAN COMMITTEE FOR STANDARDIZATION
COMITÉ EUROPÉEN DE NORMALISATION
EUROPÄISCHES KOMITEE FÜR NORMUNG

Field Test Experiments and Validation of CEN/TS 1948-4 Dioxin-like PCBs from stationary sources

– CEN/TC 264/WG 1 “Dioxins and PCBs (Emission)” –

Annex 6c

Measurement report dilution method



Secretariat:

Kommission Reinhaltung der Luft im VDI und DIN – Normenausschuss KRdL

P.O. Box 10 11 39, KRdL@VDI.de

D-40002 Düsseldorf

Germany

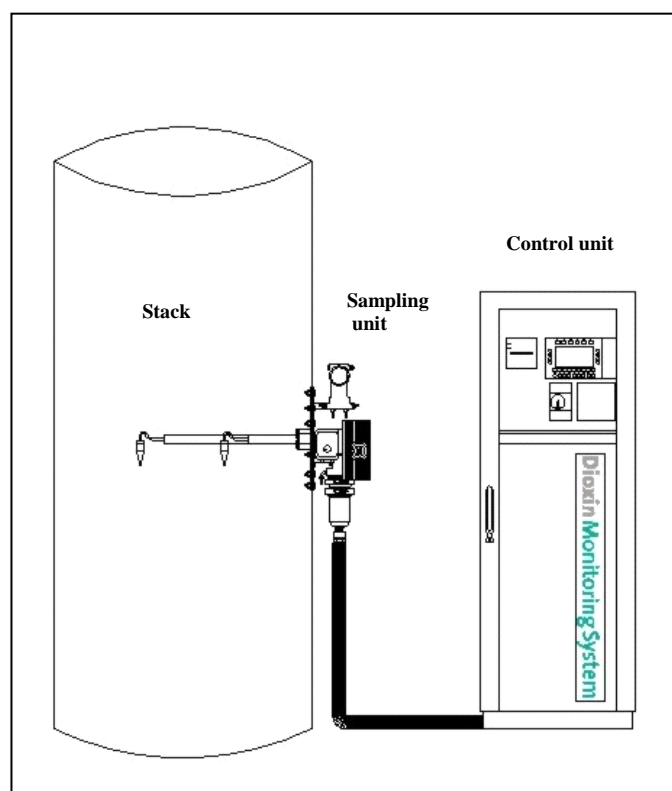
EN-1948-4 Validation Measurements in Vienna

19. -26. June 2007

Sampling device description:

For the sampling of PCBs at the Flötzersteig plant in Vienna there was employed a DioxinMonitoringSystem (Monitoring Systems, Kottingbrunn, Austria) using the dilution method described in EN-1948-1.

The three basic components of the sampling device are: the sampling unit, the control unit and the sampling cartridges.



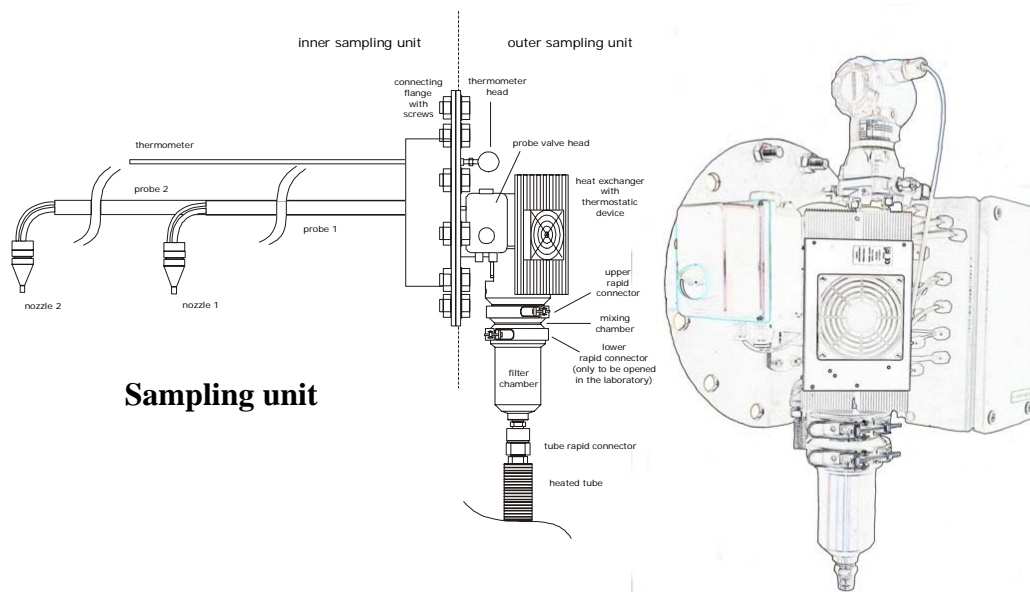
Schematics of the measurement



Measurements at the plant

The Sampling unit

The sampling unit is the part connected on the gas channel and extracts isokinetically a small part of the flue gas at one or two representative positions of the stack. Cool, dry and completely particle free dilution air is mixed with the extracted flue gas to produce a "mixed gas" with a dew point below 30°C.

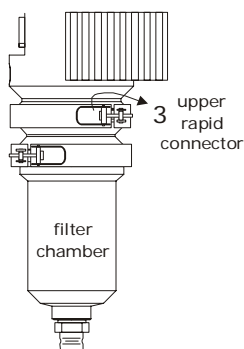


Sampling unit

The sampling unit was fitted with two heated titanium probes on the east side sampling point and with one heated titanium probes on the west side sampling point. In the case of two probes a switching device takes the flue gas sample alternately from one of the two single probes. After the switching device a titanium mixing chamber is arranged, where the flue gas sucked off is mixed with dust free and dehumidified dilution air. The temperature in the flue gas channel was measured constantly.

The Sampling cartridge (for PCDD/F and dioxinlike PCBs)

The sampling cartridge is made of titanium and is connected to the sampling unit. It consists of a mixing chamber and a filter holder with the prepared dioxin filter. It is also connected with hoses to the switch cabinets. In the filter unit the flue gas/dilution gas mixture is led over the filters. The filter set is made of a glass fibre filter and two poly-urethane plugs. At these filters the polychlorinated dioxins and furans, polychlorinated biphenyl, further organic components as well as the entire dust freight are separated. After the sampling the cartridge is transported to the laboratory.



Connection of cartridge

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The Control unit

The control unit controlled the start, the sampling process and the stop of the automatic measurement.



The Control unit

The measuring and control unit fulfills the following functions:

- independent control of isokinetic sampling at 2 positions in the stack
- periodic switching between the points of sampling in the chimney
- monitoring and control of the sampling temperatures
- documentation of the sampling
- monitoring of exceeding the control range

The sucked dilution air is filtered with a dust collector and brought by compressors into the membrane drying unit. Having passed the dryer the dilution air is led through the measuring and control systems. The measurement of the volumes of the diluent air is made by calibrated gasmeters, calibrated temperature measurement and calibrated pressure measurement. The dilution air is led to the sampling unit.



Laboratory determination working procedure:

The pre-extracted (soxleth 16 h) filter material used for sampling (Morawetz) was spiked, according EN-1948-4, before sampling with 1000 ng of PCB # 60, #127, #159 using 100 µl of Toluene(96%)/Tetradecane (4%) solution. Also the dioxin sampling standard according EN-1948-1 was added.

Sampling materials were stored in the dark before and after sampling. Before soxleth extraction (16 h) of the filter and the poly urethane plugs with toluene, extraction standards according EN-1948-4 for WHO-PCBs and marker PCBs were added.

After solvent exchange to hexane a sulphuric acid treatment followed by washing with water and drying with Na₂SO₄ was used. Multi step clean up on an automated sample clean up apparatus (Power Prep, FMS) was used to purify the extracts. Special pre-cleaned adsorbents for clean up are required in order to get sufficient low method blanks. "PCB free" multilayer silica gel (basic and acid) in series with "PCB free" basic aluminum oxide columns were used. The sample was loaded on the columns, washed by 100 ml of hexane. PCBs were eluted by 150 ml of hexane /dichloromethane 93/7 (V/V). Dioxins were eluted with hexane /dichloromethane 50/50 (V/V).

Counter flow evaporators reduced the volumes of the fractions to 0,5 ml. Recovery standards were added and a vacuum centrifuge was used to reduce the volume to 10 µl.

The standard clean up procedure used in our laboratory did not give sufficiently clean samples for non-ortho PCB determination at very low (unexpected low) concentration levels. So we added an additional carbon column clean up (6,35/10 MM Supelco, Dual Layer Carbon Reversible Tube system) to make the quantification easier. The sample was loaded on the columns, washed with 50 ml of hexane /dichloromethane 97/3. Elution of PCBs was done in counter flow with 100 ml of toluene.

Materials:

Standard solutions:

Following **calibration standards** were used for the calibration curve of **WHO-PCBs**.

| | |
|-------------------------|-------------------------|
| Wellington Laboratories | Expire Date: 10/23/2001 |
| Product code | Lot Number |
| P48-W-CVS1 | P48WCS11006 |
| P48-W-CVS2 | P48WCS21006 |
| P48-W-CVS3 | P48WCS31006 |
| P48-W-CVS4 | P48WCS41006 |
| P48-W-CVS5 | P48WCS51006 |
| P48-W-CVS6 | P48WCS61006 |

Following **calibration standards** were used for the calibration curve of **Marker-PCBs**.

| | |
|-------------------------|-------------------------|
| Wellington Laboratories | Expire Date: 10/27/2001 |
| Product code | Lot Number |
| P48-M-CVS0.1 | P48MCS0.11006 |
| P48-M-CVS1 | P48MCS11006 |
| P48-M-CVS2 | P48MCS21006 |
| P48-M-CVS3 | P48MCS31006 |
| P48-M-CVS4 | P48MCS41006 |
| P48-M-CVS5 | P48MCS51006 |

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Following labelled standards were used for the validation measurements

Sampling standard: 100 pg/μl ($^{13}\text{C}_{12}$) of #60, #127, #159
Wellington Laboratories Expire Date: 10/23/2001
Product code: P48-SS Lot Number: P48SS1006

Recovery standard: 100 pg/μl ($^{13}\text{C}_{12}$) of #70, #111, #170
Wellington Laboratories Expire Date: 10/23/2001
Product code: P48-RS Lot Number: P48RS1006

Extraction standard WHO PCBs: 1000 pg/μl ($^{13}\text{C}_{12}$) of #28, #52, #101, #138, #153, #180
Wellington Laboratories Expire Date: 10/23/2001
Product code: P48-W-ES Lot Number: P48WES1006

Extraction standard Marker PCBs: 1000 pg/μl ($^{13}\text{C}_{12}$) of #77, #81, #105, #114, #118, #123, #126, #156, #157, #167, #169, #189
Wellington Laboratories Expire Date: 10/23/2001
Product code: P48-M-ES Lot Number: P48MES1006

Solvents:

| | | | |
|------------------|---------------------|------|--------|
| Hexane: | Promochem Picograde | Lot. | 702402 |
| Heptane: | Promochem Picograde | Lot. | 705204 |
| Dichloromethane: | Promochem Picograde | Lot. | 701251 |
| Toluene: | Promochem Picograde | Lot. | 702906 |
| Acetone: | Promochem Picograde | Lot. | 703901 |

Adsorbents for clean up columns:

| | | | |
|-----------------------|--|------|-------|
| Multilayer silica | FMS, PCB free (PCBSABNSTD) | Lot. | 41 |
| Aluminum oxide, basic | FMS, PCB free (PCBSABAS011) | Lot. | 43 |
| Activated carbon: | Supelco, 6,35/10 MM Dual layer carbon reversible | Lot. | 20121 |

GC-MS:

Finnigan MAT 95 XP (Thermo Electron Corp.) coupled to 6890 series gas chromatograph (Agilent Corp.) with PTV injector.

Gas chromatography:

GC column: Agilent XLB, 60 m, 0.18 mm ID, 0.18 μm film
Oven temperature: 115°C (2 min.), 25°C/min to 220 °C, 15 min 220°C, 1,2°C/min. to 270°C, 7°C/min to 310°C, 1min. 310°C)
Injector: PTV in solvent vent mode, 60°C (0,1 min.), 700°C/min to 310 °C, 15 min 310°C
Carrier: Helium 0,8mL/min., transfer line: 260°C. 2μl injection volume

Mass spectrometer:

High resolution multiple ion detection (MID) operating in electron impact (EI) mode at 40 eV.
Resolving power > 10.000.

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Comments:

The unexpectedly low concentrations of PCBs found in the emission samples of the Flötzersteig incinerator in Vienna had the following consequences:

First, with our normal clean up procedure we were not able to get sufficient clean samples for very low level GC/HRMS determination. High background noise level made it impossible to quantify PCB #126 in any of the samples. So an additional activated carbon clean up procedure was applied.

This lead to unexpected recovery yields, because the retaining and fractionation mechanism is different to mono- and di-ortho PCBs. Also slightly higher method blanks can be expected. Second, already in the pre-validation measurements we did in December 2006 and April 2007 we observed that the PCB concentration levels of the blanks were not much lower than the concentration levels we measured in the emission sample. But the levels we measured during the validation were nearly ten times lower (see long term overnight measurement). So blank and sample concentrations could not be distinguished any more.

Long term overnight measurement

| Vienna overnight sampling on east side | | | | | |
|---|---------|--------------|----------|---------------|--|
| (90 m3 dry sampling volume and 11,0% O2) | | | | | |
| From 20.6. until 26.6.2007 | | | | | |
| Iupac PCB | pg/m3 * | Recovery % * | pg/m3 ** | Recovery % ** | |
| 28 | 182,2 | 45 | 220,1 | 56 | |
| 52 | 290,7 | 49 | 408,1 | 24 | |
| 77 | 2,7 | 56 | 4,5 | 78 | |
| 81 | 0,4 | 57 | 0,2 | 133 | |
| 60 | | 80 | | 72 | |
| 127 | | 65 | | 71 | |
| 101 | 157,3 | 55 | 299,3 | 37 | |
| 123 | 3,2 | 60 | 5,6 | 81 | |
| 118 | 47,5 | 57 | 79,5 | 79 | |
| 114 | 1,1 | 61 | 1,7 | 82 | |
| 105 | 12,3 | 62 | 20,8 | 84 | |
| 126 | 0,5 | 68 | 0,5 | 109 | |
| 138 | 71,7 | 65 | 118,1 | 54 | |
| 159 | | 88 | | 120 | |
| 153 | 154,5 | 136 | 251,7 | 112 | |
| 167 | 3,8 | 70 | 4,9 | 89 | |
| 156 | 9,0 | 62 | 13,4 | 63 | |
| 157 | 1,4 | 62 | 1,4 | 89 | |
| 169 | 0,4 | 73 | 0,5 | 124 | |
| 180 | 44,0 | 82 | 50,5 | 72 | |
| 189 | 1,2 | 62 | 1,7 | 101 | |
| * Normal clean up procedure: multilayer silica and basic aluminum oxide | | | | | |
| ** Additional carbon clean up: | | | | | |



The long term measurement results confirm the extremely low concentrations of non-ortho PCBs. Also the mono- and di-ortho PCB concentrations are very low.

Long term sampling permits to get a much higher amount of analytes in your sample.

In this case all chromatographic peaks of non-ortho PCBs are easy to distinguish from the background noise. The quantification is therefore much easier. Also the blank influence in the case of marker PCBs is reduced.

The measurement results of 6 h sampling are strongly influenced by the filed blank concentrations.

Bolzano, 24/10/2007

Dr. Werner Tirler

PCB

Results of the validation of EN-1948-4 in Vienna Recovery based on Alox clean up for some sample
 All results in ng/Nm3 dry gas on a 11% O2 reference basis using the O2 provided from Ole Schleicher (K-F
 O2 = 14,35% O2 = 14,39%

| | 1 | 2 | 3 | 4 | 6 | 5 |
|--------------|------------------|------------|-----------|--------|-----------|-----------|
| | Field Blanc 19-6 | 20-6 West | 20-6 East | 21-6 W | 21-6 East | 21-6 E BR |
| Sample N. | V1 | V2 | V3 | V4 | V6 | V5 |
| Vol dry(Nm3) | | 8.79 | 4.59 | 8.90 | 4.44 | 4.44 |
| | ng/Sample | Recovery % | Rec | Rec | Rec | Rec |
| iupac PCB | | 0.665 | Alox | 0.661 | x | Alox |
| 28 | 9.942 | 13 | 1.394 | 91 | 1.853 | 21 |
| 52 | 2.945 | 15 | 0.742 | 150 | 0.809 | 39 |
| 77 | 0.210 | 15 | 0.032 | 74 | 0.144 | 16 |
| 81 | 0.009 | 21 | 0.002 | 70 | 0.001 | 16 |
| 60 | 0.000 | 59 | 0.000 | 65 | 0.000 | 90 |
| 70 | 0.000 | 100 | 0.000 | 100 | 0.000 | 100 |
| 127 | 0.000 | 113 | 0.000 | 51 | 0.000 | 83 |
| 101 | 2.236 | 17 | 0.440 | 128 | 0.581 | 21 |
| 111 | 0.000 | 100 | 0.000 | 100 | 0.000 | 100 |
| 123 | 0.087 | 10 | 0.017 | 28 | 0.027 | 32 |
| 118 | 2.527 | 9 | 0.312 | 30 | 0.683 | 29 |
| 114 | 0.063 | 8 | 0.009 | 30 | 0.012 | 30 |
| 105 | 0.730 | 9 | 0.099 | 26 | 0.221 | 31 |
| 126 | 0.010 | 17 | 0.001 | 30 | 0.003 | 42 |
| 138 | 1.419 | 22 | 0.220 | 62 | 0.358 | 66 |
| 159 | 0.000 | 80 | 0.000 | 53 | 0.000 | 80 |
| 153 | 11.041 | 14 | 1.211 | 93 | 3.159 | 30 |
| 167 | 0.198 | 11 | 0.027 | 30 | 0.054 | 34 |
| 156 | 0.418 | 11 | 0.050 | 34 | 0.131 | 32 |
| 157 | 0.076 | 12 | 0.008 | 31 | 0.013 | 41 |
| 169 | 0.003 | 22 | 0.000 | 32 | 0.000 | 58 |
| 170 | 0.000 | 100 | 0.000 | 100 | 0.000 | 100 |
| 180 | 3.576 | 10 | 0.402 | 53 | 0.818 | 20 |
| 189 | 0.062 | 20 | 0.007 | 74 | 0.012 | 45 |

81, 123, 126 and 169 results not fulfilling SN criteria Break trough sample consisted of a additional comp

PCB

- results on a carbon clean up basis

Force)

Field Blanc absoluti in pg/sample

O2 = 11,30 %

O2 = 9,66 %

| | 9 | 7 | 8 | 11 | 16 | 10 |
|--------------|-----------|-----------|-----------|-----------|-----------|---------|
| | 22-6 FB | 22-6 West | 22-6 East | 23-6 West | 23-6 East | 23-6 BR |
| Sample N. | V9 | V7 | V8 | | | V10 |
| Vol dry(Nm3) | 0.97 | 4.56 | 2.91 | 6.79 | 3.91 | 3.91 |
| | Rec | Rec | Rec | Rec | Rec | Rec |
| iupac PCB | 0.97 Alox | Alox | Alox | 1.134 | Alox | |
| 28 | 6.315 | 1.493 | 2.385 | 1.297 | 2.119 | 0.897 |
| 52 | 9.336 | 2.248 | 3.598 | 1.159 | 2.995 | 1.242 |
| 77 | 0.175 | 0.048 | 0.064 | 0.018 | 0.023 | 0.019 |
| 81 | 0.005 | 0.002 | 0.003 | 0.002 | 0.002 | 0.001 |
| 60 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 70 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 127 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 101 | 5.136 | 1.615 | 1.569 | 1.184 | 2.218 | 0.892 |
| 111 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 123 | 0.052 | 0.027 | 0.030 | 0.014 | 0.015 | 0.012 |
| 118 | 1.175 | 0.410 | 0.548 | 0.332 | 0.422 | 0.245 |
| 114 | 0.044 | 0.013 | 0.021 | 0.008 | 0.009 | 0.007 |
| 105 | 0.294 | 0.133 | 0.162 | 0.100 | 0.124 | 0.076 |
| 126 | 0.002 | 0.002 | 0.005 | 0.002 | 0.004 | 0.000 |
| 138 | 1.905 | 0.874 | 0.986 | 0.813 | 1.216 | 0.475 |
| 159 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 153 | 6.098 | 1.854 | 2.322 | 1.815 | 3.668 | 1.587 |
| 167 | 0.040 | 0.034 | 0.027 | 0.034 | 0.039 | 0.015 |
| 156 | 0.144 | 0.079 | 0.127 | 0.077 | 0.091 | 0.064 |
| 157 | 0.019 | 0.011 | 0.015 | 0.011 | 0.017 | 0.010 |
| 169 | 0.000 | 0.000 | 0.003 | 0.001 | 0.001 | 0.000 |
| 170 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 | 0.000 |
| 180 | 0.948 | 0.485 | 0.290 | 0.508 | 0.642 | 0.205 |
| 189 | 0.018 | 0.011 | 0.014 | 0.007 | 0.016 | 0.005 |

let set of 1 filter and two PU plugs (the same as for the real sample)

PCB

| | O2 = 9,27 % | | | | O2 = 9,10 % | | | |
|--------------|------------------|---------------|-----------|-----------|-------------|--------------|--|--|
| | 17 | 14 | 12 | 13 | 18 | 15 | | |
| | 25-6 Field Blanc | 25-6 West | 25-6 East | 26-6 W | 26-6 E | 26-6 Break T | | |
| Sample N. | | | | | 0.964 | | | |
| Vol dry(Nm3) | | 6.92 | 3.78 | 7.34 | 3.92 | 3.92 | | |
| | ing /Nm3 | Rec | Rec | Rec | Rec | Rec | | |
| iupac PCB | Alox | Alox | 1.034 | 1.19 | | Alox | | |
| 28 | 7.277 | 69 1.235 50 | 2.478 35 | 1.521 59 | 4.102 49 | 2.123 66 | | |
| 52 | 5.679 | 55 0.893 54 | 1.954 14 | 1.441 19 | 5.782 23 | 2.318 66 | | |
| 77 | 0.072 | 35 0.026 59 | 0.025 59 | 0.023 103 | 0.004 91 | 0.025 86 | | |
| 81 | 0.003 | 51 0.001 55 | 0.003 69 | 0.001 117 | 0.000 106 | 0.001 112 | | |
| 60 | 0.000 | 69 0.000 75 | 0.000 80 | 0.000 64 | 0.000 72 | 0.000 92 | | |
| 70 | 0.000 | 100 0.000 100 | 0.000 100 | 0.000 100 | 0.000 100 | 0.000 100 | | |
| 127 | 0.000 | 59 0.000 51 | 0.000 85 | 0.000 96 | 0.000 87 | 0.000 64 | | |
| 101 | 4.422 | 29 0.714 39 | 1.416 24 | 1.159 30 | 4.975 28 | 2.274 54 | | |
| 111 | 0.000 | 100 0.000 100 | 0.000 100 | 0.000 100 | 0.000 100 | 0.000 100 | | |
| 123 | 0.051 | 91 0.013 62 | 0.020 63 | 0.016 80 | 0.036 74 | 0.021 83 | | |
| 118 | 1.110 | 95 0.249 58 | 0.380 62 | 0.286 79 | 0.638 77 | 0.464 87 | | |
| 114 | 0.032 | 101 0.006 60 | 0.009 60 | 0.009 72 | 0.022 68 | 0.012 88 | | |
| 105 | 0.327 | 114 0.072 59 | 0.105 65 | 0.087 83 | 0.216 77 | 0.132 82 | | |
| 126 | 0.002 | 113 0.001 57 | 0.004 86 | 0.001 133 | 0.001 147 | 0.000 82 | | |
| 138 | 2.993 | 91 0.591 51 | 0.960 32 | 0.707 37 | 1.748 45 | 1.367 61 | | |
| 159 | 0.000 | 55 0.000 64 | 0.000 88 | 0.000 86 | 0.000 72 | 0.000 82 | | |
| 153 | 10.546 | 45 1.182 121 | 1.938 74 | 1.448 99 | 7.894 62 | 3.349 63 | | |
| 167 | 0.129 | 61 0.020 59 | 0.032 73 | 0.025 87 | 0.039 89 | 0.039 85 | | |
| 156 | 0.321 | 107 0.049 57 | 0.082 63 | 0.061 83 | 0.113 86 | 0.090 64 | | |
| 157 | 0.037 | 98 0.006 61 | 0.013 74 | 0.008 97 | 0.015 81 | 0.010 68 | | |
| 169 | 0.004 | 117 0.001 54 | 0.007 94 | 0.000 154 | 0.000 159 | 0.000 80 | | |
| 170 | 0.000 | 100 0.000 100 | 0.000 100 | 0.000 100 | 0.000 100 | 0.000 100 | | |
| 180 | 2.006 | 38 0.337 69 | 0.509 49 | 0.393 61 | 0.820 53 | 0.744 36 | | |
| 189 | 0.044 | 57 0.005 49 | 0.016 89 | 0.006 118 | 0.014 92 | 0.011 37 | | |

Dioxins

Dioxin Results of the Vienna Validation

Field Blanc absoluti in pg

Values in pg /Nm3 dry flue gas on a 11% O2 reference basis using the O2 provided from Ole Schleicher (K-Force)

O2 = 14,35% O2 = 14,39% O2 = 11,30 % O2 = 9,66 %

| Sample N | V4 | V2 | V3 | V4 | V6 | V5 | V9 | V7 | V8 | V11 | V16 | V10 |
|--------------------|-------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|--------------|
| Volume dry in N m3 | | 8.790 | 4.590 | 8.900 | 4.440 | 4.440 | 3.735 | 4.560 | 2.910 | 6.790 | 3.910 | 3.910 |
| | Field Bland | 5.85 | 3.05 | 5.88 | 2.93 | Break tr. | Field Bland | 4.42 | 2.82 | 7.70 | 4.43 | Break tr. |
| | 19_6 FB E | 20_6 West | 20_6 East | 21_6 West | 21_6 E | 21_6 E BR | 22_6 FB | 22_6 West | 22_6 East | 23_6 W | 23_6 E | 23_6 E BR |
| | | | | | | | | | | | | |
| 2378 TCDD | 0.7 | 0.1 | 0.3 | 0.1 | 0.1 | 0.1 | 0.1 | 0.0 | 0.5 | 0.1 | 0.2 | 0.1 |
| 12378 PCDD | 1.0 | 0.3 | 2.1 | 0.3 | 2.7 | 0.2 | 0.3 | 0.2 | 1.1 | 0.2 | 0.5 | 0.1 |
| 123478 HxCDD | 1.9 | 0.8 | 4.7 | 0.6 | 3.4 | 0.6 | 0.6 | 0.2 | 1.9 | 0.6 | 0.6 | 0.2 |
| 123678 HxCDD | 2.2 | 0.9 | 8.8 | 0.7 | 6.2 | 0.8 | 2.7 | 0.8 | 3.7 | 1.4 | 1.6 | 0.2 |
| 123789 HxCDD | 1.4 | 0.9 | 6.9 | 0.6 | 4.2 | 0.7 | 1.8 | 0.5 | 2.2 | 0.9 | 1.4 | 0.1 |
| 1234678 HpCDD | 40.4 | 6.7 | 81.5 | 6.5 | 66.0 | 8.4 | 13.5 | 5.8 | 30.2 | 12.4 | 14.3 | 2.2 |
| OCDD | 40.9 | 9.3 | 73.0 | 8.7 | 125.5 | 10.8 | 31.2 | 8.7 | 52.6 | 18.0 | 28.6 | 4.4 |
| | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 |
| 2378 TCDF | 7.1 | 1.4 | 1.5 | 1.4 | 3.7 | 2.3 | 2.6 | 1.3 | 4.0 | 1.9 | 1.7 | 0.7 |
| 12378 PCDF | 1.3 | 0.6 | 2.9 | 0.7 | 3.0 | 0.7 | 1.6 | 0.5 | 3.8 | 1.1 | 1.1 | 0.3 |
| 23478 PCDF | 1.5 | 1.0 | 1.8 | 0.8 | 3.4 | 0.8 | 0.8 | 0.7 | 4.0 | 1.0 | 1.3 | 0.4 |
| 123478 HxCDF | 3.5 | 1.1 | 1.8 | 0.9 | 3.0 | 1.0 | 1.3 | 0.7 | 4.5 | 1.4 | 0.9 | 0.6 |
| 123678 HxCDF | 4.7 | 1.4 | 3.0 | 1.0 | 3.2 | 1.3 | 2.3 | 0.8 | 4.3 | 1.1 | 1.2 | 0.6 |
| 234678 HxCDF | 6.9 | 1.8 | 4.7 | 1.5 | 3.6 | 1.1 | 1.9 | 1.3 | 3.1 | 2.0 | 1.6 | 0.7 |
| 123789 HxCDF | 0.2 | 0.2 | 0.4 | 0.1 | 0.2 | 0.1 | 0.1 | 0.0 | 0.3 | 0.3 | 0.2 | 0.0 |
| 1234678 HpCDF | 21.5 | 6.8 | 11.0 | 5.8 | 15.2 | 5.2 | 8.4 | 4.3 | 13.3 | 8.5 | 5.5 | 1.7 |
| 1234789 HpCDF | 3.1 | 1.3 | 0.7 | 1.1 | 1.2 | 0.7 | 0.8 | 0.6 | 1.5 | 1.4 | 0.5 | 0.2 |
| OCDF | 8.3 | 7.0 | 2.3 | 6.3 | 4.8 | 2.8 | 6.0 | 3.9 | 5.4 | 9.0 | 2.6 | 1.1 |
| | | | | | | | | | | | | |
| TEQ | 5.4 | 1.8 | 6.6 | 1.5 | 7.0 | 1.6 | 2.3 | 1.2 | 6.2 | 2.0 | 2.2 | 0.7 |
| HCb in ng | 3.3 | 6.8 | 13.9 | 18.8 | 23.1 | 7.9 | 4.4 | 7.9 | 7.5 | 15.0 | 41.6 | 8.4 |

Dioxins

Field Blanc absoluti in pg

| Sample N | O2 = 9,27 % | | | | O2 = 9,10 % | | | |
|--------------------|-------------|--------------|--------------|--------------|--------------|--------------|--|--|
| | V17 | V14 | V12 | V13 | V18 | V15 | | |
| Volume dry in N m3 | | 6.920 | 3.780 | 7.340 | 3.920 | 3.920 | | |
| | Field Bland | 8.12 | | 8.73 | | Break tr. | | |
| | | 1.17 | | 1.19 | | | | |
| | 25_6 FB | 25_6 West | 25_6 East | 26_6 West | 26_6 East | 26_6 Br | | |
| 2378 TCDD | 0.0 | 0.5 | 0.6 | 0.1 | 0.0 | 0.1 | | |
| 12378 PCDD | 0.2 | 0.2 | 6.9 | 0.1 | 0.5 | 0.2 | | |
| 123478 HxCDD | 5.2 | 0.6 | 14.6 | 0.3 | 0.7 | 0.3 | | |
| 123678 HxCDD | 10.8 | 0.7 | 31.1 | 0.3 | 1.8 | 0.5 | | |
| 123789 HxCDD | 16.2 | 0.7 | 16.9 | 0.3 | 1.6 | 0.4 | | |
| 1234678 HpCDD | 297.9 | 5.7 | 462.4 | 2.2 | 34.1 | 3.2 | | |
| OCDD | 459.6 | 9.9 | 1026.1 | 3.9 | 100.1 | 5.9 | | |
| | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | 0.0 | | |
| 2378 TCDF | 1.4 | 1.0 | 5.3 | 0.6 | 1.0 | 1.1 | | |
| 12378 PCDF | 1.2 | 0.6 | 4.2 | 0.2 | 1.0 | 0.6 | | |
| 23478 PCDF | 1.3 | 0.8 | 7.2 | 0.5 | 0.7 | 0.5 | | |
| 123478 HxCDF | 3.3 | 0.6 | 9.0 | 0.3 | 0.9 | 0.7 | | |
| 123678 HxCDF | 5.9 | 0.9 | 11.4 | 0.4 | 1.3 | 0.7 | | |
| 234678 HxCDF | 23.2 | 0.9 | 22.8 | 0.4 | 1.8 | 0.7 | | |
| 123789 HxCDF | 2.4 | 0.1 | 0.7 | 0.0 | 0.1 | 0.1 | | |
| 1234678 HpCDF | 91.5 | 3.9 | 121.4 | 1.6 | 9.0 | 2.1 | | |
| 1234789 HpCDF | 24.2 | 0.9 | 2.4 | 0.2 | 1.0 | 0.2 | | |
| OCDF | 129.9 | 3.6 | 13.8 | 1.8 | 5.6 | 2.1 | | |
| | | | | | | | | |
| TEQ | 12.4 | 1.6 | 25.9 | 0.8 | 2.2 | 1.0 | | |
| | | | | | | | | |
| HCB in ng | 7.5 | 3.9 | 8.0 | 3.6 | 30.6 | 6.0 | | |

All results in ng/Sample

PCBs Extraction + Clean up Blank
(extraction of filter and PUF in soxhlet and clean up)

| lupac | Aug 07 | sep-07 | nov-07 |
|-------|--------|--------|--------|
| 28 | 2.32 | 1.88 | 5.07 |
| 52 | 2.53 | 2.06 | 5.89 |
| 77 | 0.06 | 0.05 | 0.07 |
| 81 | 0.00 | 0.00 | 0.00 |
| 60 | 0.00 | 0.00 | 0.00 |
| 70 | 0.00 | 0.00 | 0.00 |
| 127 | 0.00 | 0.00 | 0.00 |
| 101 | 2.31 | 1.88 | 2.72 |
| 111 | 0.00 | 0.00 | 0.00 |
| 123 | 0.03 | 0.02 | 0.03 |
| 118 | 0.60 | 0.49 | 0.77 |
| 114 | 0.01 | 0.01 | 0.03 |
| 105 | 0.21 | 0.17 | 0.29 |
| 126 | 0.00 | 0.00 | 0.00 |
| 138 | 1.90 | 1.55 | 2.25 |
| 159 | 0.00 | 0.00 | 0.00 |
| 153 | 1.77 | 1.44 | 2.93 |
| 167 | 0.07 | 0.06 | 0.04 |
| 156 | 0.20 | 0.16 | 0.28 |
| 157 | 0.02 | 0.02 | 0.03 |
| 169 | 0.00 | 0.00 | 0.00 |
| 170 | 0.00 | 0.00 | 0.00 |
| 180 | 0.93 | 0.75 | 0.61 |
| 189 | 0.03 | 0.02 | 0.01 |

| FB 19-06 | FB 22-06 | FB 25-06 |
|----------|----------|----------|
| 9.94 | 6.32 | 7.28 |
| 2.95 | 9.34 | 5.68 |
| 0.21 | 0.18 | 0.07 |
| 0.01 | 0.00 | 0.00 |
| 0.00 | 0.00 | 0.00 |
| 0.00 | 0.00 | 0.00 |
| 0.00 | 0.00 | 0.00 |
| 2.24 | 5.14 | 4.42 |
| 0.00 | 0.00 | 0.00 |
| 0.09 | 0.05 | 0.05 |
| 2.53 | 1.18 | 1.11 |
| 0.06 | 0.04 | 0.03 |
| 0.73 | 0.29 | 0.33 |
| 0.01 | 0.00 | 0.00 |
| 1.42 | 1.90 | 2.99 |
| 0.00 | 0.00 | 0.00 |
| 11.04 | 6.10 | 10.55 |
| 0.20 | 0.04 | 0.13 |
| 0.42 | 0.14 | 0.32 |
| 0.08 | 0.02 | 0.04 |
| 0.00 | 0.00 | 0.00 |
| 0.00 | 0.00 | 0.00 |
| 3.58 | 0.95 | 2.01 |
| 0.06 | 0.02 | 0.04 |