FEASIBILITY TRAINING AND PERFORMANCE IN INTERLABORATORY COMPARISON STUDIES WITHIN UNEP'S ASSESSMENT AND CAPACITY BUILDING PROGRAM FOR THE STOCKHOLM CONVENTION ON POPS IN DEVELOPING COUNTRIES.

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Abstract

Within the UNEP programme 'Assessment of Existing Capacity and Capacity Building Needs to Analyse POPs in Developing Countries' several activities were undertaken during 2006 and 2007. The program is focussed on the analytical capacity for the POPs under the Stockholm Convention. After the selection of nine laboratories from UNEP's database in the beginning of 2006 from four different regions (Asia, South America, Africa and former Eastern Europe) on site inspection visits were completed in 2006. During the visit the capacity of the selected laboratories and the needs for training and instruction were assessed. Special training and instruction programs were designed driven by the needs of the selected laboratories. In addition, several consumables including injection syringes, GC liners and GC columns were made available by SGE EUROPE. The results of the program for the two selected laboratories from the Asian region with respect to dioxin and WHO TEF assigned PCBs, one laboratory in China (RCEES) with high resolution mass spectrometry capacity and one laboratory in Vietnam (VRTC) with only low resolution capacity were tested in an international intercalibration with good results (z-scores < 2).

Materials and Methods

Vietnam Russian Tropical Center (VRTC)

After the inspection a specially designed training course was given by means of lectures in the morning and practical instruction in the laboratory in the afternoon. Practical training was performed at the VRTC using samples from earlier rounds of international intercalibration studies. In addition to the PCDD/DF analysis in the soil/sediment samples in house methodology was tested for the analysis of the WHO TEF assigned PCBs. After the training on site, a so-called national sample was analysed both by the VRTC and the expert laboratory at MTM consisting of a soil and a sediment sample. Here after the VRTC was invited to take part in the 12th round of the international intercalibration (INTERCAL) under the guidance of the expert laboratory.

Research Center for Eco-Environmental Sciences (RCEES).
The inspection at the RCEES showed that the laboratory was one of the top 'dioxin' laboratories in China with very good facilities. At RCEES modern laboratory facilities and two high resolution GC/MS systems were operational. No need for immediate basic training was identified, and on site training was found to be unnecessary. The focus for the RCEES was re-directed to the participation in the international QA/QC study and a national or exchange sample of a more complex sample, human milk.

Results

Exchange / National samples
The national soil and sediment sample from the VRTC contained relatively high levels of the target compounds, which did cause some problems for the expert laboratory. However, after dilution of the extract the samples fitted within the calibration range. The results for the 2,3,7,8-substituted PCDD/DFs showed good agreement for all congeners. The TEQ values for the soil and the sediment are given in Table 1.

Table1. Results from the national sample soil (VR-3) and sediment sample (VR-5).

	VRTC (ng/g)	**VRTC** (ng/g)	**MTM** (ng/g)	**MTM** (ng/g)
Code:	**VR-3**	**VR-5**	**VR-3**	**VR-5**
2,3,7,8-TeCDD	28.71	3.06	32.74	4.59
1,2,3,7,8-PeCDD	1.63	0.050	1.80	0.087
1,2,3,4,7,8-HxCDD	0.13	0.034	0.151	0.036
1,2,3,6,7,8-HxCDD	0.49	0.110	0.662	0.143
1,2,3,7,8,9-HxCDD	0.026	0.058	0.432	0.116
1,2,3,4,6,7,8-HpCDD	3.24	1.87	2.86	2.48
OCDD	26.80	20.49	24.02	29.05
2,3,7,8-TeCDF	4.80	0.83	5.59	1.02
1,2,3,7,8-PeCDF	< 0.016	0.011	0.063	0.013
2,3,4,7,8-PeCDF	0.0940	0.012	0.110	0.020
1,2,3,4,7,8-HxCDF	< 0.024	0.025	0.021	0.035
1,2,3,6,7,8-HxCDF	< 0.015	0.017	0.007	0.029
1,2,3,7,8,9-HxCDF	< 0.010	0.0040	0.051	0.033
2,3,4,6,7,8-HxCDF	0.021	0.015	0.006	0.010
1,2,3,4,6,7,8-HpCDF	0.114	0.23	0.15	0.32
1,2,3,4,7,8,9-HpCDF	< 0.010	0.014	0.006	0.029
OCDF	0.08	0.48	0.12	0.80
TEQ (PCDD/DF)	30.99	3.25	35.33	4.86
PCB #77	NA	NA	0.11	2.46
PCB #126	NA	NA	0.014	0.053
PCB #169	NA	NA	0.14	0.012

The exchange sample for the RCEES consisted of a human milk sample, which was used in the 7th round of the food intercalibration. The first set of data did show differences between the RCEES results and the expert laboratory. These results were discussed in more detail at the workshop/training at the MTM laboratory. Especially the results for the mono-ortho PCBs were different from both the expert lab and the intercalibration results. Back at the RCEES the samples were rerun and the new results were in good agreement with both the expert lab and the results acquired during the food study.

International intercalibration study.

The final test of the capacity building project was the participation of both the RCEES and the VRTC in an international intercalibration study. Guidance was provided both at the two workshops and through a guidance document. Within the 12th round of the international intercalibration 3 fly ash samples were send to the RCEES, the VRTC and 70 expert laboratories world wide. The laboratories were asked to report the seventeen 2,3,7,8-substituted PCDD/DFs and the 12 WHO-TEF assigned PCBs.

The preliminary results of the intercalibration are given below in figures 1-3 for the total dioxin TEQ of the ash samples. The samples contained medium to high levels of the target compounds. Of the total of 78 laboratories 67 were able to report for Ash A and C and 68 for Ash B. After omitting obvious outliers, 62 respectively 63 results were used to calculate the mean, median and RSD. Although these results are preliminary and will be finalised after the Dioxin2007 meeting only minor changes are expected due to data transfer or writing mistakes. The preliminary results for Ash A showed an RSD for the PCDD/DF TEQ of 32% for 62 results after excluding 5 outliers. Corresponding results for Ash B and Ash C were 17%, which is considered to be very good for a fly ash sample. As can be seen from the preliminary results both laboratories performed very well with corresponding z-scores always < 2 and often < 1.

The results of the pilot labs within the UNEP program were very promising. Existing capacity was successfully tested by the participation in an international intercalibration study.

The importance of regional laboratories participating in international or regional intercalibration studies under expert guidance was found to be a key point in the expansion of the capacity for Stockholm Convention POP analysis in developing countries.

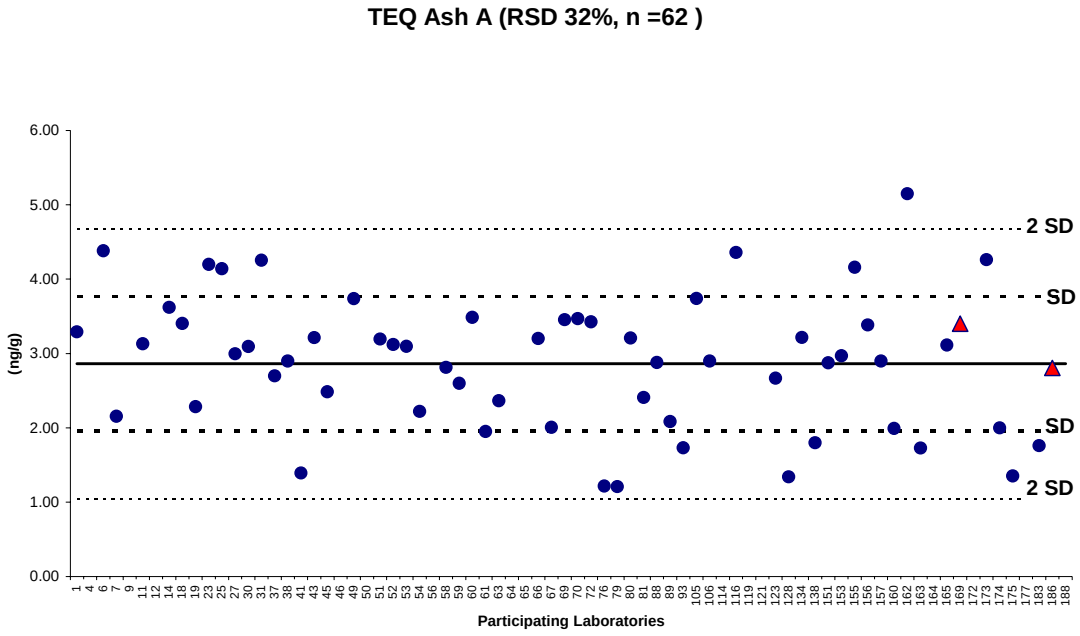

Figure 1. Results for Fly Ash A, UNEP pilot labs represented by the Δ symbol.

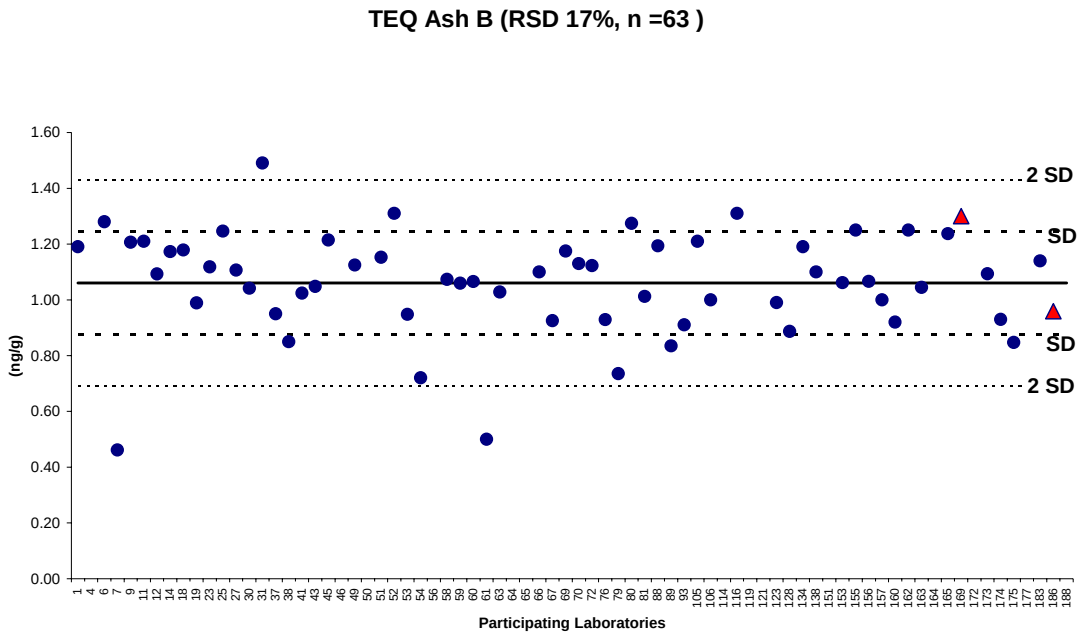

Figure 2. Results for Fly Ash B, UNEP pilot labs represented by the Δ symbol.

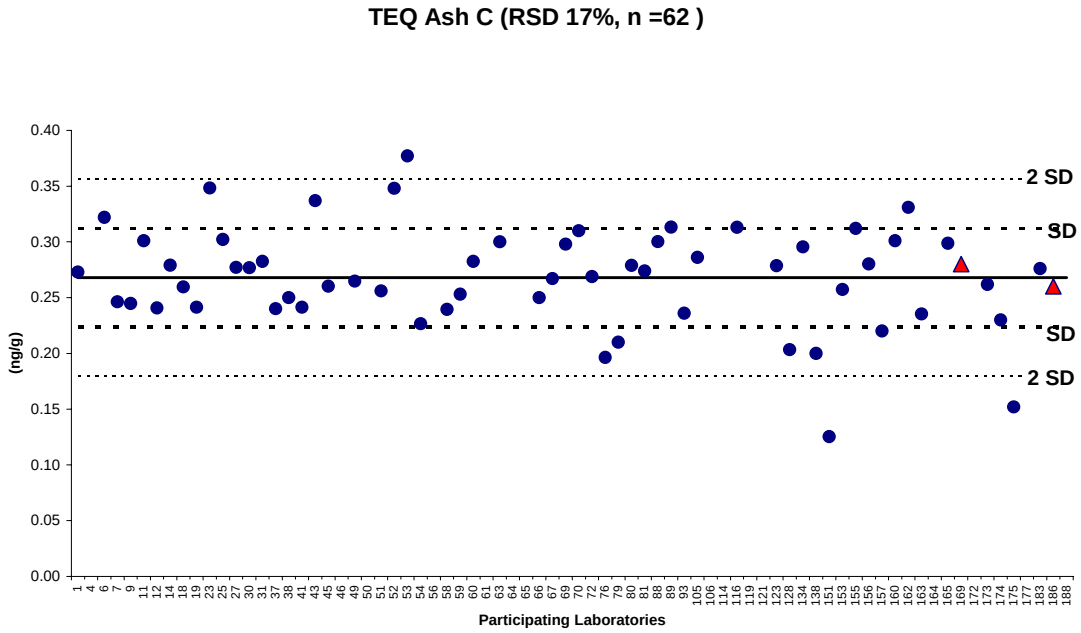

Figure 3. Results for Fly Ash A, UNEP pilot labs represented by the Δ symbol.

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