

Results from the 4th round of the International Intercalibration Study on PCDDs, PCDFs, mono-ortho and planar PCBs: Part 2 Sediment/Soil/Sludge

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Introduction

To serve as a QA/QC tool for dioxin analysis the Umeå University organises so-called intercalibration studies by sending ‘dioxin’ samples to colleagues in the field. This year this study was organised for the fourth time. Since the 3rd round¹ soil/sediment/sludge samples are included in this study, which on request by the participants is now organised on a yearly basis. The first two rounds involved mostly incineration related samples. For the 4th round of this study a total of 59 laboratories are participating, creating together with the parallel organised incineration study one of the largest studies in this field with a total of 75 participants. In this paper the preliminary results from the soil/sediment/sludge study are presented.

Material and Methods

The following samples were shipped to the participants: sample A harbour sediment, sample B an industrial sludge and sample C an extract from soil from an chlor-alkali production facility. Sample A and B were air dried before homogenisation using a planetary micro mill. The homogenised samples were packed in 10.5 and 14 ml glass vials. The extract was obtained by extracting the industrial soil with toluene using accelerated solvent extraction. This extract was diluted to 100 ml and 1 ml of this dilution was added to a glass ampoule. The glass ampoule was sealed in a flame and control weighted several times before shipment. The laboratories were asked to treat the samples as routine samples and use their standard procedures and spiking protocols. The participants were to report the 2,3,7,8-substituted PCDD/DFs, the total TEQ, the three planar PCBs (PCB #77, #126, #169) and as an option *mono ortho* PCBs which recently have been assigned an TEF value by the WHO².

Results and Discussion

A total of 49 out of the 59 participants were able to report within the set deadline. The total TEQs and 2,3,7,8-substituted congeners were reported by all 49 reporting participants. The planar PCBs were reported by 33 of the 49 participants and 23 reported also the optional *mono ortho* PCBs. In general the reporting rate of over 80 % is a good result, another 5 participants reported after the deadline.

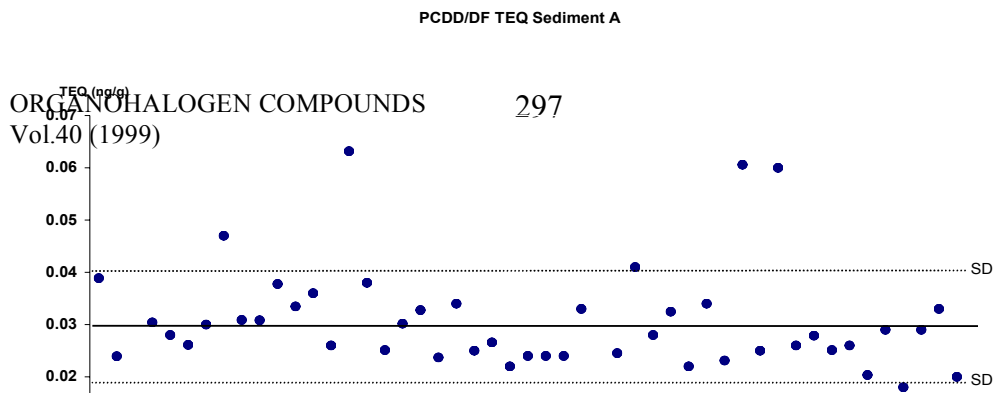


Figure 1. Harbour Sediment Sample A, PCDD/DF results in ng TEQ/g. The average of all results is represented by the straight line, one time the standard deviation (SD) by the dotted line.

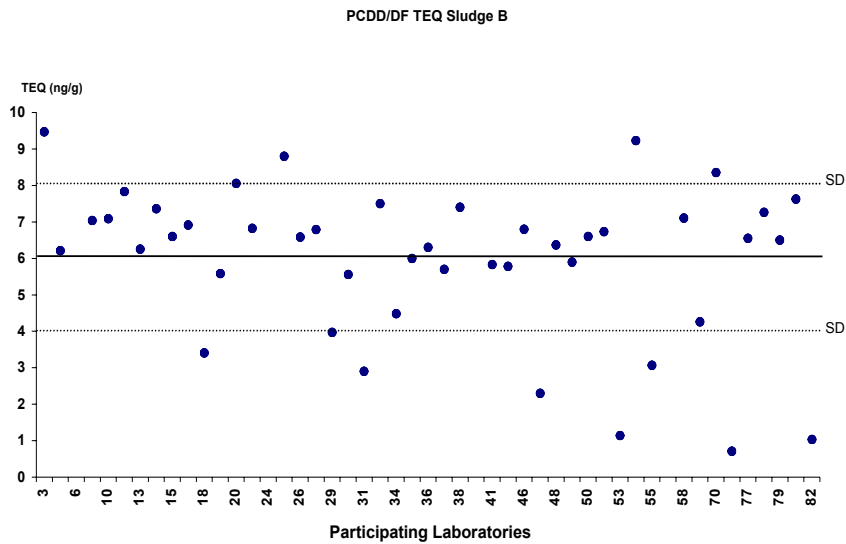


Figure 2 Industrial Sludge Sample B, PCDD/DF results in ng TEQ/g. The average of all results is represented by the straight line, one time the standard deviation (SD) by the dotted line.

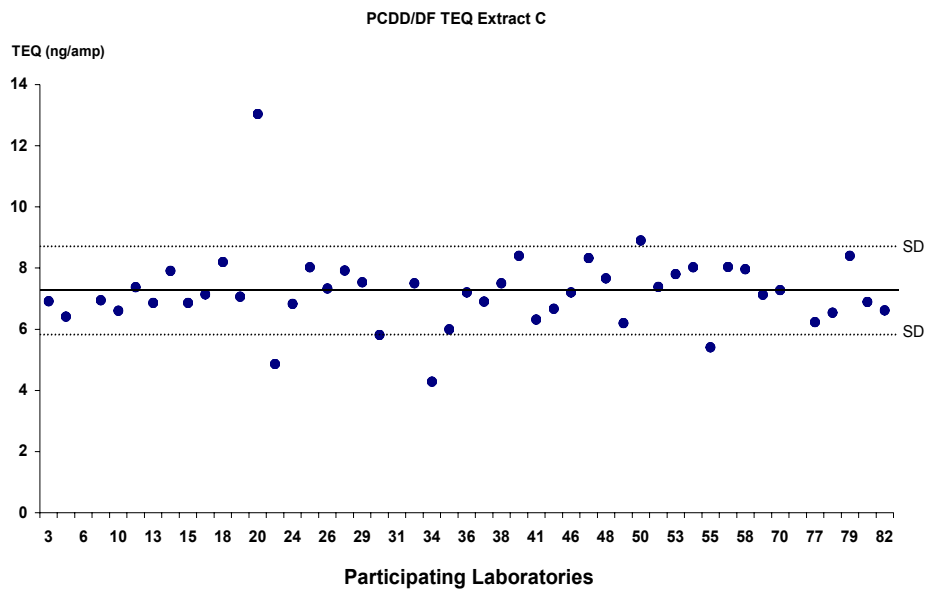


Figure 3 Industrial Soil Extract C, PCDD/DF results in ng TEQ/ampoule. The straight line represents the average of all results, one standard deviation (SD) by the dotted line.

The results for the harbour sediment are shown in Figure 1. This sample contained only low concentrations of PCDD/DFs (0.03 ng/g) but despite this there is good agreement between most of the laboratories with a RSD of only 34% between the 49 reporting participants. This RSD is about the same (35%) for the highly contaminated industrial sludge with an average concentration of 6.0 ng TEQ/g. The results of the analysis of the extract of contaminated industrial soil were even better with a RSD of less than 20 % when one extreme outlier was omitted from the data.

In conclusion can be stated that surprisingly good agreement existed between the majority of the laboratories performing dioxin analysis in sediment/soil/sludge samples. Studying the results in more detail by evaluating the congener specific data behind the TEQ values will give us further insight in the analytical problems and so further improve the analytical quality of dioxin analysis.

Acknowledgement

All the participants listed in the participants list are acknowledged for taking part in the study and their constructive comments on the study. A grant from MISTRA (Foundation for Strategic Environmental Research) within the national research program COLDREM (Soil Remediation in a Cold Climate) is gratefully acknowledged for making this study possible.

Participants list

AES Ltd England, Triangle Labs USA, Latvian Environment Data Centre Latvia, SGS Depauw & Stokoe N.V. Belgium, ENICHEM - CENTRO RICERCHE NOVARA Italy, Consorzio Interuniversitario Nazionale la Chimica per l'Ambiente Italy, Environment Canada, Maxxam Analytics Inc. Canada, Ontario Ministry of the Environment Canada, CARSO France, Technical Research Centre of Finland, GfA mbH Münster Germany, ENICHEM - Research Center Italy, Institute of Environ. Sci. & Research Limited New Zealand, Environmental Management Corp. South Korea, Korea Ocean Research and Development Institute South Korea, UMEG GmbH Germany, National Institute of Environment Analysis Taiwan, Wellington Laboratories Canada, Lancaster University England, Environment Agency England, Umweltbundesamt - Analytik II Austria, Local Hygiene Station Frydek-Mistek Czech Republic, Teijin Eco-Science Japan, Towa Kagaku Japan, Tokyo Technical Service Japan, Ebara Japan, Unitica Kankyou Gijyutsu Center Japan, Environmental Control Center Japan, Kobelco Kaken Japan, Japan Food Research Laboratories Japan, Japan Quality Assurance Organization Japan, Shimadzu Techno Research Japan, Nippon Sogo Kagaku Japan, Nishi-nippon Kankyo Gijyutsu Center Japan, Yagai Kagaku Japan, Shin-Nippon Meteorological & Oceanographical Consultant Japan, Fukuoka Institute of Health and Environmental Sciences Japan, Takuma Japan, Riken Bunsui Center Japan, Yokohama National University Japan, Homantin Government Offices Hong Kong National Public Health Institute Finland, Unilever Research Colworth Laboratory England, Norwegian Institute for Air Research Norway, Institute for Environmental Chemistry Hungary, University of Amsterdam The Netherlands, Ökometric GmbH Germany, CID-CSIC Spain, British Steel England, Axys

Analytical Services Ltd. Canada, Quanterra Environmental Services USA, Stockholm University Sweden, National Environmental Research Institute Denmark, Hüls Infactor GmbH Germany, Alta Analytical Laboratory Inc. USA, TNO-MEP The Netherlands, Umeå University Sweden^a, AKZO Nobel Sweden^a.

^a Laboratory participating outside the study.

References

¹ Results from the Third Round of the International Intercalibration Study on PCDDs, PCDFs and planar PCBs: Part 2 Soil/Sewage Sludge. Bert van Bavel, Håkan Wingfors, Christoffer Rappe and Nobuo Takeda. *Organohalogen Compounds* 35 (1998) 75-78.

² Toxic Equivalency Factors (TEFs) for PCBs, PCDDs and PCDFs for humans and wildlife. Martin Van den Berg et al. *Environmental Health Perspectives* (1998) 106:775-792.