



EU-RL Proficiency test on Determination of PCDD/Fs and PCBs in Pork and Lard 2012

Announcement

13th December 2011

1. Introduction

This proficiency test (PT) on the determination of PCDD/Fs, dioxin-like PCBs and indicator PCBs in pork and lard is organized by the EU-RL for Dioxins and PCBs in Feed and Food to be performed between February and April 2012. The objective is to assess analytical performance of laboratories and the interlaboratory comparability of results from analyses of all relevant parameters (17 PCDD/F, 12 dioxin-like PCBs, 6 indicator PCBs) in one sample of pork sausage and one sample of lard.

In addition, this specific PT is focusing on

- the assessment of the analytical performance of alternative physico-chemical techniques to GC/HRMS methods (e.g. GC-MS/MS) for determination of PCDD/Fs and DL-PCBs. Laboratories applying these methods for PCDD/F and DL-PCB analysis are especially encouraged to participate in this PT.
- the application of the measurement uncertainty.



European Union Reference Laboratory for Dioxins and PCBs in Feed and Food



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National Reference Laboratories (NRLs) for Dioxins and PCBs from EU member states are requested to participate as part of their work programme for 2012. NRLs are invited to encourage the participation of **Official Laboratories (OFLs)** from their member states as part of their duties following Article 33 of Council Regulation 882/2004. Furthermore, participation of OFLs will allow the extension of the data basis for calculation of assigned values and evaluation of results.

To enlarge the data basis for this specific PT and the related focus, this PT is also open for **other official laboratories and commercial laboratories** applying GC/HRMS methods or alternative methods to GC/HRMS and/or bioanalytical screening methods.

The evaluated results will be discussed by representatives of EU Commission, National Reference Laboratories and the EU-RL at the next COM/EU-RL/NRL workshop in May 2012 in Vienna, Austria.

Participating OFLs and commercial laboratories will receive the results of the evaluation of the PT results in preliminary and final reports.

2. Participants

This PT is open for

- National Reference Laboratories (NRLs) of EU member states
- National Reference Laboratories of other European countries
- Official laboratories (OFLs) of EU member states and other European countries
- Other official and commercial laboratories (PCDD/Fs and DL-PCBs only).

The PT is mandatory for NRLs of EU member states (free of charge) and open for official laboratories and commercial laboratories (with fee for participation). NRLs are encouraged to inform OFLs and additionally for this specific PT also commercial laboratories in their member states. This specific PT is open for commercial laboratories using GC-MS/MS or GC/HRMS methods and/or bioanalytical screening methods for determination of PCDD/Fs and dioxin-like PCBs.

A coordination of the participation of the respective OFLs through NRLs of EU member states is required. The EU-RL will send the samples only to the NRLs, including the samples for the OFLs in the respective member state.



3. Analytes of interest

Participants are requested to determine at least on of the following parameters and report the results **in duplicate**:

- 17 2,3,7,8-substituted PCDD/Fs
- WHO-PCDD/F-TEQ (using WHO₂₀₀₅-TEF)
- 12 dioxin-like PCBs
- WHO-PCB-TEQ (using WHO₂₀₀₅-TEF)
- WHO-PCDD/F-PCB-TEQ (using WHO₂₀₀₅-TEF)
- Six indicator PCBs: PCB #28, 52, 101, 138, 153, 180
- Sum of six indicator PCBs
- PCDD/F-PCB-TEQ/BEQ, PCDD/F-TEQ/BEQ and/or PCB-TEQ/BEQ (using bioanalytical screening methods)
- Measurement uncertainty for WHO-PCDD/F-TEQ, WHO-PCB-TEQ and WHO-PCDD/F-PCB-TEQ
- Lipid content

4. Test samples

The pork sample is prepared of regular market food. There is no fortification of the sample with the analytes of interest. The lard sample is prepared from regular market food and is fortified with the analytes of interest using technical PCB mixtures and PCDD/F standards.

Pork Sausage	Sample no. 1201-PLA-xxx
Lard	Sample no. 1201-PLB-xxx

Each participant will receive about 100 g of pork and 20 g of lard.

The WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ, WHO-PCB-TEQ and sum of six indicator PCB concentrations in the samples are in the range of established maximum levels defined for these matrices (COMMISSION REGULATION (EU) No 1259/2011 of 2 December 2011 amending Regulation (EC) No 1881/2006 as regards maximum levels for dioxins, dioxin-like PCBs and non dioxin-like PCBs in foodstuffs; COMMISSION RECOMMENDATION of 23 August 2011 on the reduction of the presence of dioxins, furans and PCBs in feed and food, 2011/516/EU).



5. Methods

One or more of the following methods can be applied:

- GC/HRMS-methods for PCDD/Fs and dioxin-like PCBs
- GC-MS/MS (or other alternative methods for GC/HRMS) for PCDD/Fs and dioxin-like PCBs
- Bioanalytical screening methods for PCDD/Fs and dioxin-like PCBs
- Any kind of method for indicator PCBs
(NRLs and OFLs of EU member states only)

6. Statistical evaluation of results

Statistical evaluation of the PT results is performed by the EU-RL for Dioxins and PCBs in Feed and Food according to ISO 13528:2005, Statistical methods for use in proficiency testing by interlaboratory comparisons, International Organization for Standardization, and the International Harmonized Protocol for the Proficiency Testing of Analytical Chemistry Laboratories (IUPAC Technical Report, Pure Appl. Chem, Vol. 78, No. 1, pp-145-196, 2006).

The determination of the assigned value is performed according to "The international harmonized protocol for the proficiency testing of analytical chemistry laboratories" (IUPAC Technical Report, Pure Appl. Chem, Vol. 78, No. 1, pp-145-196, 2006) by estimating of the assigned value as the consensus of participants' results (using only GC/MS and GC/ECD results). The Huber robust mean is taken as assigned value after excluding extreme outliers (outside the range of ± 50 % of the median of all reported results) and examination of the distribution of the remaining results using histogram and kernel density estimation, if necessary.

The assigned value is calculated for WHO-PCDD/F-PCB-TEQ, WHO-PCDD/F-TEQ, WHO-PCB-TEQ, the sum of six indicator PCBs and individual PCDD/F and PCB congeners (including LOQs).

For scoring of results of physico-chemical methods the z-scores are calculated according to the following formula:

$$z = (x - x_a) / \sigma_p$$

x_a : assigned value

x : participants result

σ_p : fitness-for-purpose-based standard deviation for proficiency assessment

For WHO-PCDD/F-TEQ, WHO-PCB-TEQ and WHO-PCDD/F-PCB-TEQ the standard deviation for proficiency assessment σ_p is defined as 10 %, for the sum of six indicator



PCBs (PCB #28, 52, 101, 138, 153, 180) as 15 % and for evaluated individual PCDD/F and PCB congeners as 20 %.

For further evaluation of the performance of bioanalytical screening methods, **bioassay-scores** are applied: The reported TEQ/BEQ-values derived from bioanalytical screening methods are compared with the WHO-TEQ consensus values calculated on basis of the results of physical-chemical methods. Due to the focus of bioanalytical screening methods on the identification of compliance or potential non-compliance of a sample, direct comparison of bioassay-scores and z-scores is not possible. However, bioassay scores may serve as a tool to assess method performance within the scope of external quality control measures of the respective laboratory.

Bioassay-scores are calculated according to the following formula:

$$\text{bioassay-score} = (x - x_a) / \sigma_{\text{bioassay}}$$

x_a : assigned value (physical-chemical methods)

x : participants result (TEQ from bioanalytical screening method)

σ_{bioassay} : bioassay target deviation

For PCDD/F-TEQ/BEQ, PCB-TEQ/BEQ and PCDD/F-PCB-TEQ/BEQ the bioassay target deviation σ_{Bioassay} is defined as 20 %.

Further details on the statistical evaluation of results will be given in the **instructions** for this PT, which will be allocated after registration.

7. Confidentiality

The identity of participating laboratories will be kept confidential.

For NRLs of EU member states, the “Protocol for management of underperformance in comparative testing and/or lack of collaboration of National Reference Laboratories (NRLs) with Community reference laboratories (CRLs) activities” will be observed. The confidentiality of NRLs will be kept according to this protocol.

For OFLs of EU member states, a cooperation with the National Reference Laboratory (NRL) is required. The respective NRLs will inform the EU-RL for Dioxins and PCBs about the participating OFLs and will receive the respective laboratory codes, invoices for participation fee and certificates of participation of the OFLs.



8. Participation fee

The participation of NRLs of EU member states is free of charge.

For **OFLs** of EU member states (in cooperation with NRLs) the following participation fees have to be paid:

- 400 € for determination of PCDD/Fs and/or DL-PCBs, NDL-PCBs
- 250 € for determination of PCDD/Fs, DL-PCBs using bioanalytical screening methods only
- 250 € for determination of NDL-PCBs only

The participation fees for **other official laboratories and commercial laboratories** are:

- 500 € for determination of PCDD/Fs and/or DL-PCBs, NDL-PCBs using GC-HRMS, GC-MS/MS or other alternative GC/MS methods for GC/HRMS
- 350 € for determination of PCDD/Fs, DL-PCBs using bioanalytical screening methods only

Invoices for participation of OFLs and commercial laboratories will be sent before sending of the final report and the certificate of participation.

9. Registration

Additionally you will find a registration form for participation in this PT.

Please fill out the registration form. NRLs of EU member states are asked to give also additional information on participating OFLs from their member state.

Please return the filled out registration form until January 25th, 2012 to eurl-dioxin@cvuafr.bwl.de.

Registration for this PT and reporting of results/method information is only possible by e-mail using the above mentioned e-mail address.



10. Time schedule

EU-RL	Announcement	13 th December 2011
Participant	Return of registration form	Until 25th January 2012
EU-RL	Shipment of test material, instructions and spreadsheets	13 th February 2012
Participant	Confirmation of receipt of test material	Within 7 days
Participant	Reporting of results and method information (There will be no extension of the deadline.)	By 20th April 2012
EU-RL	Evaluation and preparation of a preliminary report	May 2012
EU-RL/NRLs	Discussion at COM/EU-RL/NRL workshop with NRLs	15 th - 16 th May 2012
EU-RL	Sending of final report to all participants	September 2012

Sincerely yours,

Alexander Kotz

EU-RL for Dioxins and PCBs in Feed and Food