

# **Horizontal Standardisation of Polychlorinated Biphenyls (PCBs)**

## **Report of the deskstudy on PCBs for the European project HORIZONTAL**

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## SUMMARY

This report describes the desk study to the horizontal standardisation of PCBs. The work is based on a critical evaluation of existing standards and draft. A discussion paper has been prepared for the relevant working groups of CEN 292, CEN308 and ISO/TC190. The paper has already been discussed in a joined group of CEN 292 and CEN 308.

Important parts in the standard to develop will be decision tables, because it is not possible to make one single working procedure for the matrices involved (waste, sludge and soil). Especially it will be necessary to have different procedures for extraction and clean-up. To assure reliable and comparable results it will be necessary to set minimum requirements for different steps in the method.

After acceptance of the approach described, a framework can be made that hopefully can be filled in with procedures already described in existing methods. Limited new procedures will need description. Critical steps in the procedure can be evaluated by pre-normative research before starting a comparison study.



## 1. INTRODUCTION

The European project HORIZONTAL is focussed on the standardisation of test procedures in environmental samples. During the preparation of the project, several desk studies have been started to elaborate the possibility of horizontal standardisation on specific subjects. One of the subject was the horizontal standardisation of Polychlorinated Biphenyls (PCBs) as described in this report.

PCB's are subject of standardisation in CEN 292 (waste), CEN 308 (sludge) and ISO TC190 (soil). Interaction between these groups has been stimulated already in the last years. Horizontal standardisation should lead to one standard suitable to analyse the matrices waste, sludge, soil, sediment, soil improvers and growing media.

In the desk study on PCBs, the different existing standards and drafts have been compared which has lead to a discussion paper (annex 11). This discussion paper is an intermediate to a framework, which must resulted in the final standard. This discussion paper has been discussed with the relevant working groups of CEN 292 and CEN 308 and will be discussed on the coming meeting of ISO TC 190 in Brno. The results of this last discussion may influence the further procedure, but based on personal reactions, it is to be expected that the general line as formulated in this report will continue.

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<sup>1</sup> This discussion paper has been prepared in co-operation with Tin Win, Peter Lepom and Petra Lehnik-Habrink

## 2. A FRAMEWORK FOR A HORIZONTAL STANDARD FOR PCBS

### 2.1 General description

PCBs are measured in several matrices. For environmental purposes it is necessary that the methods applied in these matrices are comparable and making use of the same principles and instrumentation. Several standards and/or draft standards (see annex 1) are available for measurement of PCBs in different environmental matrices. Reading these documents it becomes clear that depending on the properties of the matrix different or slightly different steps in the method can be necessary. Not all procedures are applicable for all different matrices. In a standard the choices to be made must be clear and hence, a decision structure has to be described. Later on, all the steps necessary to analyse the different matrices can be described in more detail using the technical content of the existing documents or using additional text. The analysis of PCBs can be described with the following steps:

- Pre-treatment of the sample and preparing of the test portion
- Extraction
- Clean-up
- Measurement by GC in combination with ECD or MS
- Requirements for identification and calculations

The different possibilities of this procedure is described in annex 2, which overview originated from Ute Brüll. If possible the steps should comparable with steps in other Horizontal standards (i.e. PAHs).

#### 2.1.1 Pretreatment

Pretreatment is necessary to deliver a homogeneous sample and a sample suitable for extraction. The procedure depends on the material to be treated. Important is the reduction of the water content to less than 25%. Pretreatment procedures are available for soil and waste and in development for waste. The horizontal standard for PCBs should refer to these procedures.

#### 2.1.2 Extraction

Waste, sludge and soil may differ in properties and also within one matrix properties may differ very much. For instance, waste can be soil-like, but may also contain plastic residuals. These differences make it impossible to describe one general extraction procedure. Choices in a standard must be clear. The final standard should contain a decision table based on the properties of the sample and the extraction procedure to be described in the standard. Two general lines will be followed, an agitation procedure or use of Soxhlet/ASE. Depending on the extraction procedure an existing pretreatment procedure should be followed.

#### 2.1.3 Clean-up

Clean up is necessary to remove present disturbing components. When they are not present, clean-up is not necessary. If present the specific interfering components have to be removed which asks for different procedures described in the standard. Again it will be necessary to include a decision table.



#### 2.1.4 Measurement by GC in combination with ECD or MS

GC in combination with MS is the most often used combination for measurement of PCBs. ECD, however, can be necessary in presence of oil components, which are difficult to remove or to have a larger sensitivity. Both methods should therefore be included. Internal standards are necessary for quantification in both cases.

#### 2.1.5 Requirements for identification and calculations

The principles described in ISO/DIS 22892 (GC-MS identification) will be used for identification. This standard makes use of identification points as already used in an European document.

### 3. EXISTING STANDARDS OR DRAFT STANDARDS

<b>CEN/TC19:</b>	EN12766-1- PCB in oils (GC/ECD)
<b>CEN/TC 292</b>	WI028, PCB in soil sludge and solid waste (version 2003-2) (GC/ECD/MS)
<b>ISO/TC190:</b>	ISO/FDIS 10382, PCB in soil (GC/ECD)
<b>DIN:</b>	DIN38414-20, PCB in sludge (GC/ECD)
<b>CEN/TC292 and 308:</b>	Consensus document ad-hoc group TC 292 and TC 308 (N0002)
<b>CEN/TC 292</b>	EN 12776-1 and EN 12776-2- PCB's in oil
<b>ISO/TC147:</b>	NWI dioxin-like PCB's (N0633) (GC/MS)
<b>ISO/TC 190:</b>	ISO/DIS 22892 GC-MS Identification
<b>ISO/TC 190:</b>	ISO/FDIS 14507, Pre-treatment of soil (organic contaminants)
<b>ISO/TC 190:</b>	ISO/CD 11464 , Pre-treatment of soil (physico-chemical analysis)
<b>CEN/TC292:</b>	WG5 N0296, Waste: preparation of test portions
<b>ISO/TC 147:</b>	ISO/FDIS 5667 part 3 and 15, Water Quality, Sampling
<b>National standards</b>	To be supplied by national standardisation bodies

## 4. EVALUATION OF DRAFTING A HORIZONTAL STANDARD

### 4.1 Discussion

As described in previous chapters it is not possible to make a standard with single procedures for each separate step that can be used for every sample to be analysed. A horizontal standard has to contain different possibilities. It has to be prevented that everything is allowed. Multiplying all the different steps will lead to a too large number of possibilities. A solution for this is the introduction of decision tables. Depending on the goal to achieve (extraction, clean-up etc) the table has to give a limited number of possibilities. This procedure has to be secured with description of effectiveness of the procedure. For instance:

- Extraction procedure: The recovery of added PCB should be at least ..%
- Clean up shall remove the disturbing components and recovery of PCB added before clean up should be at least ...%
- Identification: Use of identification points ask for a minimum effort. This will automatically lead to the right column and detector. If not enough identification points are gained, presence of the PCB cannot be reported.

A second option to limit the number of procedures is pre-normative work. Experimental work in a limited number of laboratories will make it possible to limit the number of procedures for a certain matrix and to evaluate critical steps. This has been discussed with Tin Win and should be possible in conjunction with the working package on PAHs.

A horizontal standard can be developed for the matrices:

- Solid wastes. Liquid wastes like oil and water are excluded
- Sludges
- Soils

Validation of the standard for all matrices on the short term will be difficult and expensive, because a standard containing decision tables allows different combinations of procedure. Validation has to be an ongoing process and needs co-ordination, which can be a task of the project HORIZONTAL. A steps wise validation, starting with the most important HORIZONTAL matrices, is proposed, followed with validation of specific matrices being part of the scope of the standard, in all cases using the most appropriate procedure for those matrices. It must be realised that the scope of the experts and committees involved in the standard of PCBs is wider than the scope of HORIZONTAL. As long as a full validation is not available some limited data must be available, even from one laboratory, for a procedure allowed in the standard. Validation may also lead to steps not yet described or excluding a specific step for a specific matrix. This will make adjusting of the decision tables necessary in new versions of the standard. It is expected that much of the validation work can be combined with that required for PAHs.

### 4.2 Steps to be taken

Horizontal standardisation will be successful if all parties involved are in favour of such a procedure. It is the experience that experts become enthusiastic seeing the similarities for different matrices. In a joint meeting of CEN 292 en CEN 292 on the subject of PCBs, their was a common opinion, that the way of working described in this report has to be continued. It is necessary that also ISO TC190 will contribute. This is not expected to be a problem, but the time schedule of this commission is not the same as this project. ISO TC 190 will met in September in Brno. It will be possible, but not easy to organise joined meetings of all committees. The budgets of experts involved are limited. It will be necessary to develop a way of working, fitting in the schedules of the standardisation bodies involved

and also fitting in available budgets of the experts. Email and internet can be used for this purpose but cannot replace all the personal contacts. Standardisation is a matter of consensus, which is mostly the result of personal interaction.

Assuming that ISO TC 190 will co-operate, the next step will be preparing a framework of the standard including all the decision tables needed. This has to be discussed in a joined group with experts from both CEN and ISO. If the framework is accepted, the technical details can be filled in using description in existing standards and drafts.

As described above it will be necessary to look to the possibilities of pre-normative research to limit the allowed possibilities in the standard. This can be in co-operation with studies within the Workpackage „Horizontal – Organic micropollutants“.

Pre-normative work will be followed by comparison studies, matrix by matrix.. It is expected that a complete validation of the Horizontal standard will be too expensive to do it in one comparison. It will be necessary to set priorities for validation that also will be taken over by European laboratories to assure partition in the comparison study.

### 4.3 Communication

The following communication scheme as suggested by ISO/TC190 (figure 1) is taken over. To be sure that also the Non-European members, which members are positive on the initiative and are willing to support, will be involved in the process of Horizontal standardisation.

The upper scheme is for the subject PCBs described in this report. The other schemes should be subject of other reports, but are also included here, because co-operation is to be expected. Specially for PAH the way of working will be comparable, as follows from the scheme.

The lower scheme is for LAS and NP. These compounds are not on the working program of TC 190. It is possible that they will be on the program in the near future. In order to stimulate the horizontal approach, TC190 would like to be informed, to be able to give information on the matrix soil, which may lead to incorporation of the matrix soil in the scope.

AOX is not an important item for soil and it is not necessary to inform TC190. Soil should therefore not be included in the scope of the horizontal standard to develop

For all measurements pretreatment is very important. A good communication within the work package pretreatment and the analytical measurements has to be stimulated.

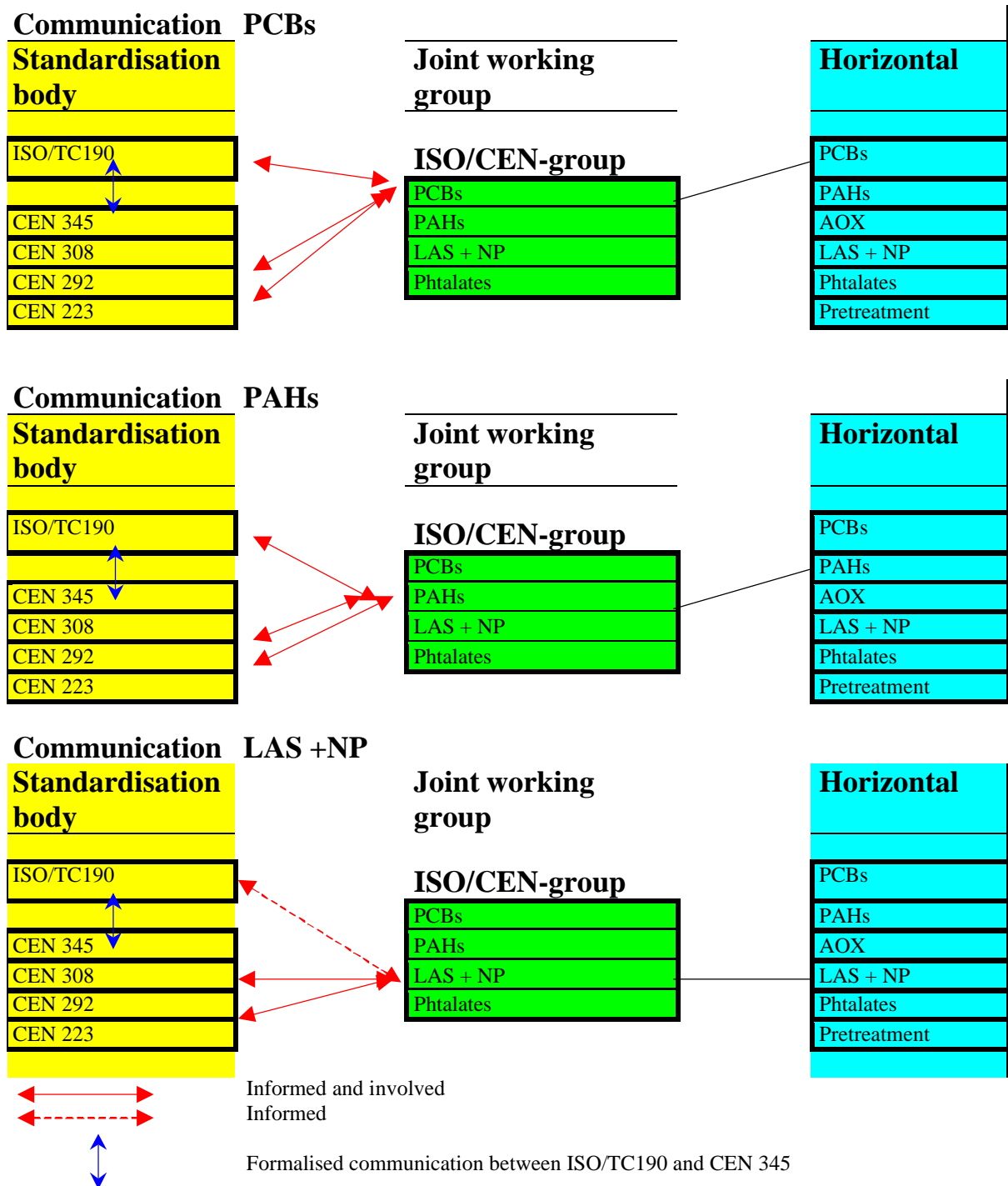


Figure 1. Communication Necessary during development of Horizontal Standards

## 5. CRITICAL POINT AND RECOMMENDATIONS

The Horizontal approach in standardisation is an accepted approach. Standardisation bodies, however, are still matrix orientated. At the moment it is necessary to fit the horizontal approach in the meeting schedule of the different standardisation bodies. If this is done support of the experts can be achieved. An important point is that experts do not have the time and budget to join meetings of all the different groups. It will be necessary to work on such a way that horizontal standards will be developed with the support of all experts in all different groups. If Horizontal standardisation is going faster than the standardisation bodies it may result in not accepted standards.

In the approach described in this report, decision tables are part of the standard. These tables make differences in applied methods possible, depending on the sample. Justification of the method will be assured by setting limits for recovery and prescription of the minimum requirements for identification. This approach has to be discussed within the project HORIZONTAL

## 6. DRAFT STANDARD (CEN TEMPLATE)

Will be the result of next step

## REFERENCES

See list of standards involved in chapter 3

EU, 2002. Commission Decission 12 August 2002. Implementing Council Directive 96/23/EC concerning the performance of analytical methods and the interpretation of results. Docnr: C(2002) 3044