HORIZONTAL

Secrétariat Horizontal – Task Group 3

Task Group 3 "Organic parameters – AOX – PAH – PCB – LAS – NP – selected phtalates"

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TG 3 N 16

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Le comité membre français :

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Subject :	**HORIZONTAL – TG 3 "Organic parameters – AOX – PAH – PCB – LAS – NP – selected phtalates"**
Comments :	**Follow up of the comments on PCB given by TG 3**
For action :	For information

Association reconnue

d'utilité publique

Comité membre français

du CEN et de l'ISO

Siret 775 724 818 00015

Code NAF 751 E

Template for comments and secretariat observations	Date: 2006-03-28	Document: **Horizontal 3-12 PCB**
Horizontal 3-12 PCB		
Author of comments: ON/Austrian Standards Institute		

1	2	(3)	4	5	(6)	(7)
MB[1]	**Clause No./ Subclause No./ Annex** (e.g. 3.1)	**Paragraph/ Figure/Table/ Note** (e.g. Table 1)	**Type of com-ment**[2]	**Comment (justification for change) by the MB**	**Proposed change by the MB**	**Project Horizontal observations** on each comment submitted
FR			ge	The reports on PCB and PAH went beyond the requirements given for phase II (i.e. to produce a feasibility study). Nevertheless the feasibility studies were done on relevant matrices following an acceptable experiment plan. This method may go forward to phase III.		see next
		Expl. notes P2, last §	ge	Ruggedness is not available.	Provide a ruggedness report.	Testing is performed in this period. It should be realized that starting the project, it was assumed that several methods were already available and limited testing on ruggedness should be necessary. TG 3 requires for a ruggedness report to be available on HORIZONTAL homepage.
AT			ge	The draft standard is a very detailed description of all steps necessary for PCB analysis. Especially for the cleanup a lot of different methods are described. However this variety of methods makes it difficult to choose the right one for a specific sample. Therefore we suggest to provide a kind of guideline describing a combination of clean up steps which leads to clean extracts for the majority of samples		Some more guidance is given. Agreed by TG3
NL			ge	This standard contains a compilation of several modules. Because of a sufficient quality control, the Netherlands approves this new approach within HORIZONTAL.		Tanks for the support
NL				It is appreciated that The PAH and PCB standard are using the same elements. Most of the comments which have been given on the PAH standard are also		Relevant changes have been made together with the changes for PAH. See the

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				applicable on this standard and should therefore be added to the comments which are given below. Necessary changes should be the same (if possible) in both standards		document for PAH for the consequences. Agreed by TG3
NL			ge	In the principle it is mentioned that after pretreatment according to the methods referred to in 9.2, the test sample is extracted with a suitable solvent. In 9.2 only acetone and petroleum ether are mentioned	Adjust the Horizontal standard so that it is clear that other solvents for extraction and clean-up are allowed.	see note under table 1 Agreed by TG3.
UK	Foreword		ed	CEN 292…	Only include TC's involved in horizontal	CEN 292 has been involved in development.. It s agrred with CEN 292 to make the content of this standard and their standard in agreement and if possible to end up with one single standard Agreed by TG3

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Horizontal 3-12 PCB		
Author of comments: ON/Austrian Standards Institute		

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FR	3.5 Internal standard And 7.5.2 And 10.7		te	It is not an obligation to have an internal 13C for each native PCB. It is very hard to handle for operator and expensive as well. For example we could use a heavy internal standard (ex : PCB 209). Moreover, If the recovery is all right for the heavy, it should be correct for the others. A certified material in PCB could also control all the process.		In cases that an aliquot of the extract is used, it is allowed to use an unlabeled standard. Therefore 7.5.2, 10.3 and 10.7.5 have been changed. A certified material is not an assurance for each individual sample. Properties of the samples in the scope differ. In application of this Horizontal standard freedom is given in choices. On the other hand, an extra check on the performance is asked A minimum of three internal standards are required. They will recommended for each group of analyte.
UK	3.6		ge	The term 'injection standard' is used whilst in the dioxin draft 'recovery standard' is used for the same definition.	We need a common approach to terminology across all horizontal standards. Both terms are correct, just need to choose one.	Shall be discussed in Task-group TG 3 recommends to harmonise the terminology in the different standards.
UK	4		te	70% recovery limit	It is stated that this limit may be changed following the validation study. This is a common sense approach but it should be noted that this will be significantly reduced if multiple clean-up's have to be applied to a sample (even if a minimum	a lower recovery using multiple clean-up is accepted Agreed. Leave it as it is.

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					recovery of 80% is given for each clean-up). It is unlikely that this will be tested during validation. A note should be provided.	
NL	4	Line 16	ed	Change :at least 70 in: at least 70 %		Changed Agreed by TG3
UK	7		ge	Include all chemical formulae as required by CEN		Will be done Agreed by TG3.
FR	7.5.2.2 and 10.8 ECD detection		te	I agree with the choice of internal standard. We can use an heavy PCB above PCB 180 after verification if there is any trace of it in the sample ex : PCB 209.		PCB209 is advised Other are possible if not in the sample Already stated above.
NL	7.6		te	Standards are made in n-heptane, the final extract is PE. Has this influence on the chromatogram?		We do not expect changes, but will check this in the coming month The result of this checking will be made available on the website of Horizontal.
AT	5.1.2		te	The possible coelution of PCB-28 and PCB-31 on some kinds of GC columns should be mentioned here with the remark that the separation of this critical pair will be used as an column performance check. Both have the same molecular mass so this critical pair could not be separated by mass spectrometry		Text in 5.1.2 is changed Agreed by TG3
UK	8.1.1		te	Is the use of glassware advisable for sludge samples due to the risk of explosion	Aluminium sample containers may be more sensible	Aluminium is added Put the following note : Glass is not appropriate for sludges.

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AT	8.1.6		te	Both the 100ml flask and the thimble of 27*100mm are described as examples but unfortunately these pair does not fit together.		removed e.g. 100ml Agreed by TG3
UK	9.2		te	Complete drying is also recommended if samples are to be stored for a long period	Section 9.1 states that samples can be stored below -18 ºC for 1 year. Should these be dried?	a sentence on dry samples is added in 9.1 Agreed.
UK	9.2	Drying table	ge	Waste (shredder, plastic)	Waste is not part of horizontal, remove column	This standard is overlapping with a standard in development by CEN 292. This is also mentioned in the scope. This column makes the standard even more horizontal Agreed by TG3. It is possible to open the scope of the standard but wastes are not planned in the validation programme.
AT	10.2.		te	Spiking should be done in several portions directly onto the sample material. Therefore the minimum volume of the spiking solution is 100µl		is included in note 3 of 10.2.2 Agreed by TG3.

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FR	10.2.1		te	I am not confident in the extraction “agitation” for sludge.		Guidance is given for choice of the procedure. For organic rich materials, Soxhlet is preferred TG 3 accepts to mention the following sentence : “For sludge it has been shown that soxhlet or PLE is applicable.” , provided the ruggedness results are put on the homepage of Horizontal website.
NL	10.3		te	Why is only ‘nitrogen’ mentioned, while other gasses are also possible? Add note	Why is only ‘nitrogen’ mentioned, while other options are also possible? Add note	added: or other inert gas Agreed by TG3
DK	10.4		Te	Too many possible clean-up methods are included, since it will not be possible to include them in the validation study.	Remove the rarely used clean-up methods. Alternatively – wait for the validation study and remove clean-up methods which are not well documented in the validation study.	Because a lot of interfering compounds can be present, several clean-up procedures can be necessary. Only existing methods are mentioned. After the validation study all will be evaluated. This can also be subject of the meeting of the task groups. Because more possibilities are possible it is found to be necessary to include a check on performance for each sample. This is a kind of

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						internal validation. This Task Group advises the validation provider to be in close relation with the sub-workpackage leader to have precise designs of interlaboratory trials.
NL	10.4.1 to 10.4.11		te	Concentration to 0.5 ml with Kuderna Danish is not practical even when a keeper is used	Concentrate to approximately 7 ml. Then use a gentle nitrogen or air flow to concentrate to approximately 0.5 ml	concentrator is replaced by procedure described in 10.3 Agreed by TG3
NL	10.4.1		ge	The reference aluminium oxide (7.3.2.1) is not correct.	Change 7.3.2.1 in 7.3.1.1	Changed Agreed by TG3
NL	10.4.1		te	It is strange that the aluminium oxide will be eluted with approx. 10 ml of petroleum ether.	In ISO 10382 deactivated aluminium oxide with 10 % water will be eluted with 20 ml of petroleum ether. So change it.	Changed Agreed by TG3
NL	10.4.2		ge	The reference silica gel (7.3.4.2) is not correct	Change 7.3.4.2 in 7.3.2.2	Changed Agreed by TG3
NL	10.4.2		te	It is strange that the silica gel 10 % will be eluted with 100 ml of petroleum ether.	Normally is eluting of silica gel 5 % with 10 ml petroleum ether sufficient. So change it in: elute silica gel 5 % with 10 ml petroleum ether.	Changed Agreed by TG3
NL	10.4.4		te	It is difficult to evaporate up to 0.5 ml with KD. It should be made clear that KD is first used to evaporate up to 5-10ml. Next the evaporation can be done by blowing with a gentle gas flow.	Add the description of the last part of the evaporation which can be used to concentrate the extracts.	is changed in 10.3 Agreed by TG3
UK	10.4.11		ed	Give the concentrated extract….	Add the concentrated extract…	Is changed in agreement with the other clean-up procedures

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UK	10.7.2	Table 3	ed	Diagnostic ion 3 has an * with no note	Clarify or delete	Deleted Agreed by TG3
UK	10.7.4	2nd para	te	…a diluted extract has to be injected for proper quantification….	It should be added that for highly contaminated samples, they should be re-extracted with less sample taken.	is added Agreed by TG3
FR	10.8.5		ed	There is no good correlation between the relation and definitions of terms. Which one is the internal standard and which one is the standard injection?		It is made clear how interferences for the injection standards are recognized Agreed by TG3
UK	10.8.5	Eq 5	ed	Where… Re,143..207	Re,198…209. These are recommended earlier in the text and should be used throughout the explanation of equation 5.	Changed Agreed by TG3
UK	10.8.5	Last para	ed	..7o%	..70%..	Changed Agreed by TG3

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FR	11, 12		Te	Since this method is very modular, the validation of the chosen path applied to a type of sample should not only be proven by the suggested means. The results of the validation should be given to the "client" of the determination as a whole part of it, and thus should appear in the test report (clause 12).	Amend the test report content.	If performance criteria are not met, it is not allowed to report, or report with a remark. If performance criteria are met the quality is good. So not necessary to add to the report A part of this problem will be solved after the validation procedure. This Task Group advises the validation provider to be in close relation with the sub-workpackage leader to have precise designs of interlaboratory trials related to the handling of each kind of samples. The pertinence of all technical paths will be checked at this time.

TG 3 decides that the working range of the method will be checked after the interlaboratory trial.

TG 3 expresses the need to include one sample of each matrix with a content near the quantification limit.

It has been checked if comment on the PAH-standard is also applicable on the PCB standard. If so, changes made in the PAH-standard are also made in the PCB-standard.

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				The standard on waste (EN15308) in discussion in CEN 292 is almost the same as this standard. Both standards have been discussed in the CEN 292 meeting of April 27/28 in Berlin. The scope of both standards have been adjusted and cross reference has been made. The comment made for EN 15308 (document N845 of CEN 292) has been used to improve the Horizontal standard. It was agreed that a final goal of the standardization process should be one single Horizontal standard including waste. EN 15308 will be validated by CEN 292. Results will be applicable for this Horizontal standard.		

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