HORIZONTAL

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

TG 3 N 17

Secrétariat Horizontal – Task Group 3

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

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Template for comments and secretariat observations	Date: 2006-03-28	Document: **Horizontal 3-11 PAH**
Horizontal 3-11 PAH		
Author of comments: National Standard Institutes		

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
AT			ge	It would be interesting to know standard or limit values from the different countries of Europe.	Add an informative annex	This is interesting, but no part of this standard. It is a regulatory purpose and TG 3 agrees with this answer.
AT			ge	Reduce the draft to essential requirements	Additional information could be given in an informative annex	Information is limited as much as possible to essential requirements. Due to the modular approach of this standard, users may subtract the modules they use. Agreed by TG 3
NL			ge	This standard contains a compilation of several modules. Because of a sufficient quality control, the Netherlands approves this new approach within HORIZONTAL.		Thanks for the support
NL			ge	It is appreciated that The PAH and PCB standard are using the same elements. Most of the comments given below are also applicable on the PCB standard. Necessary changes should be the same (if possible) in both standards		This was part in handling the comments TG 3 advises the sub-workpackage leader to take this into account.
UK	Introduction		te	..contamination level for PAH can lay in the range of about 0,001 mg/kg…	It is suggested that this is increased to 0,01 mg/kg as at present it does not fit with the current scope of the standard (0,01)..	Is changed Agreed by TG3
UK	1 Scope	4th para	ed	Since some of the target PAH are not so good soluble in the usual..	Since some of the target PAH are relatively insoluble in the usual..	Is changed Agreed by TG3
DK	1 Scope		Te	A list of the 16 PAH target analytes included in this standard should be mentioned in the scope or reference given to Table 1 (clause 7.5.1)	Include a table after the final paragraph listing the 16 PAH target analytes included in the method with names and CAS-nos. or refer specifically to	See table 1 Agreed by TG3.

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					Table 1 in clause 7.5.1.	Add information about interferences of benzo[j]fluorenthen in the interference paragraph.
NL	1		te	No definition of the "lower limit of application" is given, what is meant: "detection limit" and is it based on "repeatability" or "reproducibility" The definition of this term should be clear. Especially due to the last sentence [penultimate sentence] in which it is said that the method can only be used; "provided that all performance criteria in this method are met".		A definition of detection limit is included, which is based on the repeatability conditions with specific instrumentation of the laboratory, according to DIN 32645:Jan.2006 TG 3 suggests to change the drafting of the scope. Mr Win will redraft this clause in accordance with the proposals made by I. BARNABAS.
UK	2		ed	Are all of listed references normative?	Suggest moving some of the informative references to a bibliography	All informative references have been moved to Bibliography Agreed
FR	P2, last §		Te	The sentence “HPLC only suitable for high contaminated soils” is strange. In many cases, HPLC-Fluo is more sensitive than Mass detector.	Provide a ruggedness report.	We cannot find this sentence It was not put into the standard. It was done during the presentation.

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FR	3.3 Internal standard for GC-MS and 7.5.1Reference substances, internal standards 10.6.4		te	For GC-MS, It is not an obligation to have an internal 13C for each native PAH. For the operator it is very hard to handle so many internal standards and it is very expensive. Why not use the 6methyl chrysene as in the HPLC method for the internal calibration in GC and the recovery?	PAH and PCB : The report of ruggedness is insufficient. At some steps, the choice of the method is left to the user, without any criteria on the results. This means that the results are not equivalent. This method could be non-applicable to sludge. It seems not to be possible to go forward to phase III. The choice of solvents is much more important than in the extraction method, however there is no statement in the report on robustness to confirm this.	Minimum number is given 6-methylchrysene and other internal standard can also be used for GC. TG 3 agrees that at least 5 internal standard are needed. In both standards, elements of other standards have been used. Limited Ruggedness testing was foreseen in the project due to the availability of standards. The elements used are partly validated. More validation will be done in the coming months. TG3 requests the sub-workpackage leader to put the information on the website through H. VAN DER SLOOT; The user is the only one who can make choices. If wrong choices are made, performance criteria are not met. We should like to hear why the method is not applicable for sludges Phase III should be discussed in the task groups. TG 3 proposes to put on the homepage of Horizontal all the results of the ruggedness for all workpackages. TG 3 decides to go forward to phase III.

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						Extraction procedures have been used in other standards
UK	3.4		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 and suggests to retain : *recovery standards*.
UK	4		te	The 70% lower limit for recovery needs to be verified both from the ruggedness testing and the inter-lab trial	Change value (if necessary) to reflect actual method performance.	Is planned ; this will be checked after the interlaboratory trial.
DK	4 Principle	5	Te	Depending on sample and analytical performance, recovery of internal standards could not only be lower but also higher than 100%.	Describe an acceptable range for the recoveries of internal standards, e.g. 70-130%, and not just a lower limit.	Because very high recoveries are not to be expected, we used 105% as upper limit TG 3 suggests to retain 110 %.
NL	5.1.2	paragraph 2	te	The term "complete resolution" should be defined (is this 0,8; 1,0 or something else). An important question which should also be answered is if "complete resolution" is needed or not.		Resolution has been defined See terms and definitions Agreed by TG3
NL	5.1.2		te	An important interference can be benzo(j)fluoranthene which can not be separated (nor by chromatography nor by mass spectrometry)	If for benzo(b)fluoranthene interference is suspected it has to be stated in the report. HPLC results might give more reliable results	This is added to 5.1.2 Agreed by TG3
UK	5.1.3	2nd para	ed	Toluene extract are to be diluted sufficiently…	Samples extracted with toluene are to be….	Samples cannot be diluted TG 3 agrees on the following : "Extracts are to be diluted

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						…"
UK	6.1.1		ed	Correct numbering		There is no 6.11. 10.3.1 has been corrected Agreed by TG3
UK	7		ge	Include all chemical formulae as required by CEN		Shall be included as required by CEN Agreed by TG3
NL	7.1		te	The demand on the "allowable blank value" is normative but vague. In practice we use for the blanc value a value that should be below half the lowest reported value.	use for the blanc value a value that should be below half the lowest reported value	Text has been changed accordingly Agreed by TG3
NL	7.2.2		te	The boiling range can be changed to **30** °C to 60 °C. Within the Netherlands there is data available which supports this.	Change 34 by 30	This change has been made in the note Agreed by TG3
FR	7.2.2 Toluene or hexane-like for extraction		te	Toluene is very good to extract PAH, but it tends to degrade the solid phase of column.	We think that this is not appropriate for sludges.	Toluene is specially included for sludges TG 3 does not retain this comments. No change.
NL	7.2.3		te	Can toluene also be used as an extraction solvent. Specify the allowable solvents.		Note was on the wrong place, Agreed by TG3
UK	7.2.3	Note	ed	Where does the note belong? Is it under 7.2.2?	If the note belongs to 7.2.2 then suggest that the two temperature ranges are consistent.	It belongs under 7.2.2 Agreed by TG3
NL	7.2.4		te	Heating of the sodium sulfate is not always necessary. There are commercial products available which have a	Remove the sentences on heating	Is removed

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				negligible PAH / organic matter content. If the sodium sulfate should be heated will be visible during the blank determination which is obligatory before every new batch can be used.		Agreed by TG3
NL	7.3.1.2		te	"Deactivated aluminum oxide" in some cases other amounts of water will be needed e.g. 11%. This should be mentioned more explicitly.		We think the 11% is described 90 g + 10 g of water Agreed
NL	7.3.1.2		te	A maximum storage time is not mentioned for aluminum oxide, while this is mentioned for silica gel. Make this more consequent.		For both a period of two weeks is described Agreed by TG3
NL	7.3.4/10.4.1		te	This is a very uncommon cleanup method. According to the Netherlands this method can be removed or put into the appendix.		This will be discussed with the task group TG 3 decides to leave it as it is.
NL	7.3.4		ed	In 7.3.4 DMF is used. Further in the document i.e. in table 2 and in 10.4.5, DMF and DMSO are mentioned.	It must be clear which of the two solvents are used for the liquid-liquid partition cleanup	As mentioned choice will be made later TG 3 suggests to remove DMSO.
DK	7.5 Standards	Paragraph 1	Te	Available monofluorinated PAH standards (2-5 ring) could possibly also be used as suitable and cheaper alternatives to isotopically labeled (i.e. ^{13}C- and D-) PAH internal standards for both GC-MS and HPLC analysis.	Extend the description of suitable internal standards by including a description of available monofluorinated PAH.	To be discussed in task-group Leave the text as it is.
NL	7.5.1		te	The use of all 16 deuterated PAK makes this analysis unnecessarily expensive. The quality of the analysis can also be assured by the selection of 4 or 5 deuterated PAK with a wide range in boiling point / extractability. It should also be possible if only part of the PAK are to be		A minimum of 5 is included Agreed by TG3

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				determined that only part of the deuterated PAK have to be used (e.g. if PAK 10 VROM are to be analyzed).		
UK	7.7.3.3.	Note	te	Is the note applicable if only toluene or cyclohexane is allowed (from 7.7.3.2)	Delete note or amend text accordingly.	Use of cyclohexane is allowed, providing performance criteria are met The note has been deleted. Agreed by TG3
UK	7.7.4		ed	Transfer for example…	If this is any example, this should be a note	Changed in appropriate amount Agreed by TG3.
UK	8.1		te	All glassware… should be thoroughly cleaned	How is this achieved?	Normal laboratory procedures can be used. We do not want to describe this. If dirty glassware is used performance criteria are not met Agreed by TG3
UK	8.1.1.1		te	Is the use of glassware advisable for sludge samples due to the risk of explosion	Aluminium sample containers may be more sensible	Changed in glass, stainless steel or aluminium Glass is not appropriate for sludges. Put this in a note.
UK	8.2.1		ed	Separation between benzo(a) and benzo(e)pyrene should be at least (0.8)	Benzo(e)pyrene is not mentioned previously in section 7.5. If this is to be used for a resolution check then this should appear here.	Benzo(e)pyrene is added to table 1 Agreed by TG3
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

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						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.
UK	9.2	(e)	te	…and when volatile PAHs are analysed	Do volatile PAHs not have the potential to be lost during freeze drying?	Yes. Added: Losses of volatile PAHs is possible Agreed by TG3
UK	10.1		te	Blank values should be smaller than the detection limits for analytes concerned	Is this practical in all cases? Generally, blank values should be <10% the critical level of interest is acceptable. Blank correction should be allowed if necessary.	This has been changed. The calibration curve contains an intercept. This automatically includes blank correction TG 3 agrees on < 50 % of the lowest reporting level.
UK	10.2.1	2nd para	ed	…compost or 2-20g waste.	..or 2-20g of bio-waste.	(bio)waste Agreed by TG3. It was agreed for wastes.
FR	3.11	10.2.1	te	Will the agitation be sufficient for sludge?		Soxhlet extraction with toluene is foreseen for sludge. TG 3 accepts to mention the following sentence : "For sludges it has been shown

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						that soxhlet or PLE is applicable." , provided the ruggedness results are put on the homepage of Horizontal. website
NL	10.2.2		te	"12 h" shaking is to long: reduce to 15 min and add a note that in some cases longer extraction times are necessary.		We choose for 30 min two times. Not agreed. Put a note : "If the lab can prove sufficient extraction in a shorter time, it can be acceptable."
NL	10.2.2	paragraph preceding NOTE 2	te	Change twice "25 %" into "50 %". In the Netherlands data is available that samples with a water content of up to 50% can be effectively extracted with the stated procedure.		Not changed, because the ratio becomes smaller than 9:1 Agreed by TG3
NL	10.2.2		te	Extraction 1: No extraction time is mentioned for shaking with 50 ml acetone		Time of 30 min is added Agreed by TG3 . The first extraction with acetone is 30 min and the second one with hexane will be 12 hours. Note : If the laboratory can prove sufficient extraction in a shorter time, it can be acceptable.
UK	10.2..2	1st para	ed	Samplel	sample	Changed Agreed by TG3

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DK	10.2.3		Te	This clause is titled Soxhlet/Pressurised liquid extraction, however, no description of PLE is included.	Include a second paragraph that describes the use of PLE similar o that of Soxhlet.	PLE is allowed providing performance criteria are met. It can be described in Annex. Agreed by TG3
UK	10.3.1		te		There is no temperature limit for evaporation. However, this is prescribed during clean-up to 40 ºC	Temperature depends on equipment. 40 ºC is removed from10.4.4 Agreed by TG3
FR	10.4 Clean-up		te	We must take care of lipids or oils in the sample. It could disturb the GC analysis. Otherwise it is not so disturbing for HPLC. Nothing is proposed for the elimination of sulphur.		For lipids GPC is used (lipids are add to the table). For aliphatic DMF/DMSO is advised. Normally it is not necessary to make clean up of sulphur. If necessary performace criteria are given Agreed by TG3 : removal of the DMSO.
FR	10.5		te	Volume of standard injection can be estimated according to the medium point of calibration curve.		It shall be relevant and measurable see 7.8 and 7.9 Agreed by TG3
NL	10.4.1		te	"… at least 80% for all relevant PAH …" In Chapter 4 the minimum recovery is 70%. Define in which way these recoveries have to be obtained (with or without sample pretreatment, aging of the sample or only spiking of the extract etc…).		80% is for the clean-up. This can be checked with a standard (added to the text) Agreed by TG3

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NL	10.4.1	Table 2	te	Clean-up D is not mentioned in this table, therefore Table 2 is not complete and unclear.		Clean-up D is removed Agreed by TG3
NL	10.4.1	Table 2	te	Allow other necessary clean-up procedures	Other clean-up procedures are allowed, providing they remove the interfering compounds and have a recovery of at least 80% of the relevant PAH	Is added Agreed by TG3
UK	10.4.1.	General	te	..recoveries after the use of the clean-up are at least 80% for all relevant PAH	Is this limit necessary as the overall recovery limit for the analysis is 70% (section 4). This must already include clean-up.	This is only for clean-up Added: for a standard Same comment. Agreed by TG3
UK	10.4.5	title	ed	DMF	DMSO or DMF, which is correct?	Both, selection after validation TG 3 chooses DMF.
NL	10.6.2		te	Gas chromatographic conditions are exactly the same as for PCB. Is this correct?		Yes Agreed by TG3.
NL	10.6.4 10.6.5 10.6.6		te	Currently it is unclear if s (slope) of the initial calibration line or of the working standard should be used. The text of 10.6.6 is in general unclear (what is the need of the working standard). We advise to use a two point calibration and to skip the use of a single working standard for the GC-MS method. It should also be clear that the initial calibration function is only tested. If this test is passed the calculation of the samples should be done with the calibration function which is calculation using the two calibration standards which were measured within the series.		Has been made more clear, also in calculation Agreed by TG3
NL	10.6.4		te	In other standards not only the highest point is tested but also the 2nd and 3rd highest point are calculated to test the		Problem is not clear

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				calibration function (see for example NPR 6403 (a Dutch technical report on chromatography)). It is also unclear if a relative deviation should be used or not.		Leave it as it was.
NL	10.7.1		te	"Chromatographic separation. Use a column and chromatographic conditions which allow efficient separation of the PAH stated in the scope. For a choice of columns and the corresponding gradients see annex A." Define efficient separation, the current phrasing is unclear.		Annex is added Agreed by TG3
NL	10.7.4	paragraph 3	te	The "verification of a positive result" is not normative; rephrase.		Sentence has been changed Agreed by TG3
NL	10.7.4		te	The verification of a positive result may be obtained, if required, using different methods" This is not normative, rephrase.		Has been changed Agreed by TG3
NL	10.7.5.3	line 3	te	"actual sensitivity" is not normative described, rephrase.		Has been changed Agreed by TG3
NL	10.7.5.6		te	What is the definition of "response factor" f_i in equation 6?		This is the slope of the calibration curve. Has been changed Agreed by TG3
UK	10.6.2		te	Capillary column, non to medium polar…	5% phenyl methyl silicone is specified in 8.2.1.	We referred to 8.2.1 Agreed by TG3.
UK	10.6.3	Table 3	te	Is the list of PAHs in the order in which the deuterated standards are used for quantitation ?	Add a note to state that those PAHs below the internal standards are quantitated by using the d-labelled standard above.	Comment not clear TG 3 agrees to the addition of a note specifying which internal standard should be

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						used for the quantification of which native PAH.
FR	10.6.4		ed	Page 19 : a reference is made to paragraphs 9.6.4.1 and 9.6.4.2. However these paragraphs do not exist.		9 should be 10 Agreed by TG3
UK	10.6.7 (and 10.7.5.5)	2nd para	te	…a diluted extract has to be injected for proper identification or quantitation	It should be added that for highly contaminated samples, they should be re-extracted with less sample taken.	Re extraction is made possible Agreed by TG3
UK	10.6.9		te	See comments under section 4 on recovery limits	Bring in-line following validation exercise.	Has been done by allowing lower recoveries for multiple clean-up Agreed by TG3
NL	11		te	How an analytical method has to be validated should not be a part of the standard. At the moment it is unclear what the relation between the validation of the standard and other demands are. Most of the validation work for this standard is also demanded by the accreditation body. Another problem might be the availability of three reference materials. Especially for waste applications. Conclusion: in Chapter 11 there should only be references to available validation methods and programs and what the minimal recoveries are which should be obtained.		In this standard more freedom is given to the laboratory. It will be impossible to validate all routes, Therefore performance criteria are used and necessary validation steps are described, if a specific combination has not been validated before. The need for at least three materials is removed The removal of the 3 CRM is agreed. All clause 11 is moved in Annex.

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NOTE Columns 1, 2, 4, 5 are compulsory.

Template for comments and secretariat observations	Date: 2006-03-28	Document: **Horizontal 3-11 PAH**
Horizontal 3-11 PAH		
Author of comments: National Standard Institutes		

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	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. No necessary to add validation results to the report. It is not a common practice. A part of this problem will be solved after the validation trial This Task Group advises the validation provider to be in close relation with the 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. This comment is also suitable for PCBs.

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NL	12		te	Point d) goes too far for a report, this point should be removed.		Is removed Agreed by TG3.

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TG3 agrees that the following comments came to late to be implemented, and are included in the handled comments.

Dear colleagues,
here are some recommendation from Slovakia on the comments :

PCB (AT)
- nowadays, we work on specification of the most suitable clean up procedure

PCB (F)
- it is possible to determine a minimal number of labelled 13C internal standards (e.g. 2 internal standards and 1 injection standard), and using PCB 209 for GC/MS

PCB (NL)
- in our laboratory is used the same solvent for standards and extracted sample (hexane)
- nitrogen is most widely used inert gas

PAH (DK)
- recovery of internal standards can be in the range 70 - 130% (we agree) (4 Principle)
- if there was no noticed interference with analyt, it would be possible (monofluorinated PAH standards) (7.5 Standards, Paragraph 1)

PAH (F)
- we agree with using 4-5 deuterated PAH as internal standards (3.11)
- concentration and volume of internal standard: analyst should consider about it (3.11, 10.5)

PAH (AT)
- it is necessary to know comparison of legal provisions from the different countries of Europe (Directive EU)
- we do not agree with reduction of the draft to essential requirements

PAH (NL)
- condition of resolution of the critical pairs Rij = 0,8 (5.1.2)

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- sodium sulphate reactivate by heating once a week, 1 hour at 225°C (7.2.4)
- deactivate aluminium oxide once a week (7.3.1.2)
- we agree with using 4-5 deuterated PAH as internal standards
- extraction by shaking 12-16 hours (7.5.1)
- extraction by shaking for wet samples (content of water less than 50%)- if it is confirmed, we agree (10.2.2)
- shaking with acetone - by hand, short time, while the whole sample is soak and the clusters are disrupted (10.2.2)
- the initial conditions for PCB and PAH are the same
- explain the using of the working standard (10.6.6)
- it is possible to consider about testing of calibration curve (the 3 highest points) (10.6.4)
- we agree with attaching of general validation procedures to the draft (11)

We are sorry for the late reaction to the comments.
Have a nice weekend.

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