



## 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 18**

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June 2006

Le comité membre français :



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<i>Subject :</i>	<b>HORIZONTAL – TG 3 “Organic parameters – AOX – PAH – PCB – LAS – NP – selected phtalates”</b>
<i>Comments :</i>	<b>Comments on LAS</b>
<i>For action :</i>	For information

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Comité membre français

du CEN et de l'ISO

Siret 775 724 818 00015

Code NAF 751 E

Template for comments and secretariat observations

Horizontal 3.13.0 LAS

Date: 30/05/06	Document: <b>second draft</b>
(fill in)	(fill in)

1	2	(3)	4	5	(6)	(7)
MB <sup>1</sup>	Clause No./ Subclause No./ Annex (e.g. 3.1)	Paragraph/ Figure/Table/ Note (e.g. Table 1)	Type of comment <sup>2</sup>	Comment (justification for change) by the MB	Proposed change by the MB	Project Horizontal observations on each comment submitted
AFNOR				All this standard is restricted in term of type of extraction. Moreover, there is no evaluation report to demonstrate the ruggedness of the method in the website of horizontal for LAS.		The evaluation report about experimental work including ruggedness test has now been presented.
AFNOR	title		Ge	... - LAS by HPLC.... : as the LAS content is calculated as a sum of isomers, the title should refer to an index	... - LAS <b>index</b> by HPLC....	Not agree. LAS is the product, that consists of a mixture of homologues and isomers. No correction made.
AFNOR	1	§ 2	Ed	...may also be analysed by the method.	...may also be analysed by the method <b>if conditions are proven.</b>	Agree. Text will be changed accordingly.
AFNOR	1	§ 3 & 4	Te	Limit of detection : it would be useful to have a definition of the LOD and the way it will be determined by interlab trial	Give a definition of the LOD	The definition of LOD shall not be included in the standard. See the evaluation report now available. No correction made.
AFNOR	2		Ed	Horizontal standard 17-1 (draft) can't be addressed here.	Move to bibliography	This will be corrected at a later stage of the process. No correction made.
AFNOR	3.1	3 <sup>rd</sup> line	Te	...depending on the length of the alkyl group : the number of isomers doesn't depend on the length of the alkyl group, but on its degree of branching.	depending on the <b>branching degree</b> of the alkyl group	Not agree. The number of isomers within each homologue depends on the homologue (length). No correction made.

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AFNOR	4	4 <sup>th</sup> §	Te	Use of 2 ions for identification should not be optional but mandatory.(see below)	Delete “optional “ and rewrite in the “shall-mode”	This is discussed in the evaluation report now available. Only one ion will be described for MSD.  Text will be changed accordingly.
AFNOR	7	all	Ed		Add CAS number or reagents	Depends on the CEN rules for writing standards. Will be changed according to these rules.
AFNOR	7.7		Te	In order to avoid misunderstanding in the future, select only one component by CAS number	Delete one CAS	Both CAS numbers are used by suppliers of LAS.  No correction made.
AFNOR	7.11	note	Te	It would be useful to have information about the “freezability” of the calibration standards.	Make an addendum to note if info available.	This information is not available.  No correction made.
AFNOR	8.4		Te	The absence of information on the clean-up column is harmful for the validation trial, if not given prior. Too many possibilities would make interpretation hard.	Regarding the index type of the measurement, only one (max two) possibilities should be allowed.	The information about clean-up columns will be included, see evaluation report now available.
AFNOR	8.4 10.3		te	I think that a clean-up is necessary, even in the case of sludge, mainly if we use an HPLC-MS-MS if we don't want to clean all the time the detector. Moreover, there is a good chance the baseline of chromatogramm can be too noisy.		This is treated in the evaluation report now available.  More information about the use of the two detectors will be added in the method.

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AFNOR	8.6	Last §	Te	Why is isomeric separation more recommended for fluorescence detection, as in 10.5 LAS content is supposed to be the sum of homologues & isomers ?	If no further info, delete	Due to the lower selectivity of the FLD the isomeric pattern is more needed for FLD than for MSD.  More information about the use of the two detectors will be added in the method.
AFNOR	9.1		Ed	Regarding the status of HORIZONTAL 2.2 study, citation as a standard is delicate.	Refer as a report, addressed in bibliography	This will be corrected at a later stage of the process.  No correction made.
AFNOR	9.1	note	Ed	...may to preserved...	...may to <b>be</b> preserved...	Yes, text will be changed accordingly.
AFNOR	10.1.1	a)	Ed	Take 2-3 g	<b>Weight accurately</b> 2-3 g	Yes, text will be changed accordingly.
AFNOR	1.2.1	a)	Ed	Take 10-15 g	<b>Weight accurately</b> 10-15 g	Yes, text will be changed accordingly.
AFNOR	10.1		te	Why does the author impose the shaking extraction ?		The shaking is preferred as primary method since it is easy to adapt, less time-consuming, especially with many samples.  This is further treated in the evaluation report now available.  No correction made.

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AFNOR	10.1.1		te	10 ml of MeOH is really sufficient for extraction of sludge ? There is no evaluation report attached in the website of horizontal for LAS.		Yes, 10 ml is sufficient for sludge. This is treated in the evaluation report now available.  No correction made.
AFNOR	10.3	2 <sup>nd</sup> §	Te	Absence of necessity of the clean-up with MSD : not achieved with single ion detection ! For example, squalenes, highly represented in soils, give buckets of ions which may induce false positive detection.	Use at least 2 ions	This is treated in the evaluation report now available. Only one ion will be described for MSD. According to our experience the only need for a clean-up when using MSD, is the contamination of the detector.  No correction made.
AFNOR	10.3	3 <sup>rd</sup> §	Te	We would be glad to see some ruggedness to assess these affirmations.	Show ruggedness study (none found on HORIZONTAL web site)	This is treated in the evaluation report now available.  More information about the use of the clean-up and the two detectors will be added in the method.
AFNOR	10.4	2 <sup>nd</sup> line	Ed	What is optional? Blank or clean-up?	Be more accurate	Agree. New text in 10.4 1 <sup>st</sup> para: "Perform a blank determination following the procedure as described. Prepare the blank exactly as by the analysis of the sample, including the clean-up if the clean-up has been used for the samples."

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AFNOR	10.4		Te	Would be useful to have examples of "right" blank matrices.	Give examples	This is a matter for the laboratory to decide. No correction made.
AFNOR	10.4.2		Te	This § and the following ones seems to be based on the hypothesis that all homologues show identical responses on FLD. Has that been demonstrated?	Give evidences	Yes, it has been demonstrated, see the evaluation report now available. No correction made.
AFNOR	10.6.2		Te	The re calibration deals with calibration function. What is done to control that the LQ is maintained ?	Add a § on the control of achievement of the initially validated LQ	This is a part of the internal QA and is therefore not included in the standard. No correction made.
AFNOR	10.6.3	2 <sup>nd</sup> dot	Te	This requirement assumes that all technical mixtures found in environment, maybe after some degradation, have the same peak pattern as the standard. Has it been proven ? (asked by a desperate SCCP analyst ;-))	Give evidences	Yes, the same chromatographic peaks are found in standards and in real samples. However, the distribution may differ. No correction made.
AFNOR	10.6.3	Last §	Te	The phenomenon described here clearly indicates a coelution of an interfering compound. We don't be confident in the dilution of the sample to solve it.	Delete dilution process. May an adaptation of ISO/CD 18857-2 be suggested (in red, to be adapted) : <i>"These interference may lead to incompletely resolved signals and to additional signals in the chromatographic pattern of NP, NP1EO and NP2EO. They may, depending on their magnitude, affect accuracy and precision of the analytical results, since all 3 analytes are determined from the sum of a cluster of 8 to 10 chromatographic peaks. It is important that the interfering peaks are not included in the calculations."</i>	The paragraph is not connected to the treatment of interferences, but to the treatment of samples with high concentrations of LAS. No correction made.

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AFNOR	11		Te	... <i>provided</i> the recovery ... : should be at least verified before interlab trial	Show ruggedness study (none found on HORIZONTAL web site)	Agree, C8-LAS is a valid internal standard. This is treated in the evaluation report now available.  Text will be changed accordingly.
AFNOR	Annex A		Te	These conditions (diameter of the column, flow) are micro HPLC conditions. Is it possible to describe non-micro HPLC conditions, or is micro HPLC mandatory ?	Show examples.	The 2,0 mm columns was only used in order not to "stress" the MSD. Traditional 4,6 mm columns may also be used.

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