INERIS	INTERLABORATORY COMPARISONS ANNUAL PROGRAM 2018

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INTERLABORATORY COMPARISONS
2018

« Waters »

"Alkylphenols – Bisphenols Water Framework Directive Compatible"

DRC-17-158303-07275B/ December, 17th 2017

ERRATUM : Test on artificial Waste Water is out our scope of accreditation

Organizer: INERIS- DRC

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Accreditation No. 1-2291, ILC, Scope available on www.cofrac.fr

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1. CONTEXT

The analysis of alkylphenols and compound is historically recognized as extremely problematic. A number of actions at the level of AQUAREF (development of methods, study of the purity of the commercial standards, technical days, summury about alkylphenols compounds of water framework directive and at the statutory level (DCE 2013/39/CE; Decree monitoring water 2015, accreditation notice 2015) were undertaken. The quality of the analysis on these parameters is essential and is very important for monitoring waters in water framework directive. The French OCILs working on these compounds continues to meet difficulties during the exploitation of these data restored by laboratories.
This document contains all the information necessary for fully informed registration in the Proficiency Test (PT).

It contains:

- the participation procedures
- a description of the inter-laboratory programme for 2017-2018

2. OBJECTIVES

The Interlaboratory comparisons “Alkylphenols – Bisphenols Water Framework Directive compatible 2017-2018” organized by INERIS in waters will allow to establish the performance of the participating laboratories.

This participation in a proficiency testing programme has several objectives:

- To verify the appropriation of the works and the technical recommendations realized since 2013 in France on alkylphenols;
- To know the practices operated within laboratories French persons receiving benefits by the study of a questionnaire which will resume all the metadatas associated with the analytical methods operated on this family of substances (nature of the standard, the correction purity of the standards, etc.);
- To keep improving the quality of analyses especially for alkylphenols in waters.

3. PERSONS CONCERNED

This **free** interlaboratory comparison is opened to any French laboratory or European laboratory realizing measures in natural waters and/or contributing to monitoring of environment according to the Water framework directive.

The number expected is 30 participants. If this number is exceeded, the organizers reserve the right to review the participation of each laboratory. If less than 10 participants, the decision of postponement or continuance of this trial will be studied by the organizer.

4. GENERAL TECHNICAL REQUIREMENTS

4.1. REGISTRATION PROCEDURE

The participant will confirm its participation in the INERIS before

January, 26th, 2018

by returning the application form IM-1542 duly completed, together with an order and the payment of the participation fee for the program.

During the registration phase, the laboratory will specify unambiguously:

- the mailing address for submitting administrative documents and
- the address for the delivery of the test materials.

After the close of registration, INERIS assigns a confidential code, an identifier and a password to each participant. At the latest fifteen days after the date of the close of registration, a confirmation is sent to the participants by mail using Form IM-1063 "Registration Confirmation". The confidential code should be quoted in all correspondence with the coordinator.

4.2. INERIS COMMITMENTS

INERIS undertakes to comply with standard EN ISO/IEC 17043 and LAB CIL REF 02 of COFRAC in the organization of its interlaboratory comparisons.

INERIS undertakes to ensure the confidentiality of information when returning results online and anonymity when sending the report by assigning to each participant a confidential code, an identifier and a password.

INERIS undertakes to preserve the confidentiality of the identity of each participant by limiting access to the confidential code to a limited number of people collaborating in the coordination of the tests.

INERIS undertakes to promptly notify participants of any changes in the design or the operation of the proficiency testing program.

INERIS undertakes to examine all claims and to take action if necessary. Claims must be made in writing to the attention of the coordinator.

4.3. COMMITMENTS OF PARTICIPANTS

At the time of their registration, participants undertake to:

- complete and return the acknowledgment of receipt IM-0223,
- respect for each parameter the method specified in Annex 1 and in the Instruction Form IM-1541,
- restitute the results with full integrity, without falsification or collusion,
  - In case of NO-COMPLIANCE, INERIS reserves the right not to take into account the data of the participant concerned and initiate appropriate actions.
- restitute the results as scheduled, except in case of equipment failure reported before the results restitution deadline
- provide the associated metadata, as requested.

4.4. COMMUNICATION

All exchanges between INERIS and participants are mainly electronic. INERIS cannot be held liable for No-receipt of an email. The confidential code should be quoted in all correspondence with the coordinator.

The test related documents can be downloaded from the site dedicated to INERIS ILC **http://www.ineris.fr/cil/.**

5. ANNEXES

Annex No.	Title
1	Description of the programs “Alkylphenols – bisphenols Water framework directive compatible 2018”
2	General organization of Proficiency Test
3	Statistical processing and restitution of test

Annex No.1: Alkylphenols – Bisphenols Water framework directive compatible 2018

***The following tests will not realized under cover of our accreditation**

Accreditation No. 1-2291, ILC, Scope available on www.cofrac.fr

Family	ALKYLPHENOLS – BISPHENOLS	
Substances	**Substances alkylphenols - bisphenols**	**CAS Number**
	NP : 4-nonylphenol	84852-15-3
	OP : 4-tert-octylphenol	140-66-9
	NP1OE : 4-nonylphenol monoethoxylate	27986-36-3
	NP2OE : 4-nonylphenol diethoxylate	27176-93-8
	OP1OE : 4-tert-octylphenol monoethoxylate	2315-67-5
	OP2OE : 4-tert-octylphenol diethoxylate	2315-61-9
	Bisphenol A*	80-05-7
	Bisphenol S*	80-09-1
Date of receipt	First half of february	
Analytical standards	In choice of the participant	

Quality Assurance (QA)	Put materials at room temperature before analysis Blank (matrix supplied by the organizer) Standard solution (matrix supplied by the organizer) Specific investigation to know the practices in every laboratory (metadatas questionnaire)	
Tested matrices	Natural water Artificial Waste water* Extract solution	
Level of concentration	A single level by matrix	
Samples	**Quality control**	A glass bottle about 1000 ml for the matrix blank A glass bottle about a few ml with the standard solution
	Tested Matrices	<u>1/ Natural water and waste water:</u> 2 amber glass identical bottles about 1000 ml for the material natural water 2 amber glass identical bottles about 1000 ml for the material waste water 1 amber glass bottle with a few ml of extract solution
Stabilization	Yes : Materials « natural water » and « waste water »: acidification to pH 2 with HCL 37% Material Quality assurance “standard solution” and Material “Extract solution”: prepared in acetone	
Refrigeration	Yes	

Number of measures per bottle	**1 measure by bottle**: materials “natural water” and “waste water” and material quality assurance “blank” **4 measures by bottle**: material quality assurance “standard solution” and material “extract solution”	
Statistical processing implemented	**Assigned value**	1/ Materials Natural water and waste water: Robust Mean of all participants' results and robust standard deviation determined from all the participants of the results by applying the algorithm A of the NF ISO 13528. 2/ Material extract solution: Value calculated (formulation) and robust standard deviation determined from all the participants of the results by applying the algorithm A of the NF ISO 13528.
	Performance	1/ Materials Natural water and waste water: Score z 2/ Material extract solution: Score z Score zeta (if obtaining uncertainty on the result by the participant)
Treatment of QA test materials	**Blank**	Identification of results with concentrations greater than LOQ.
	Standard solution	Value calculated (formulation) and robust standard deviation determined from all the participants of the results by applying the algorithm A of the NF ISO 13528 Performance: Score z Score zeta (if obtaining uncertainty on the result by the participant)
Monitoring of the homogeneity and the stability	**Organization**	*INERIS*

Annex No. 2: General organization of an interlaboratory comparison

For each test, the sequence of events will be as follows:

- feasibility study to determine the best conditions for future testing if necessary
- sampling, possible doping, packaging;
- shipping (j=0) of the test materials to the various participants by INERIS. receipt by the participants (j= +1);
- analysis of the test materials by participants (j = +1 to + XX) according to the parameters; and tracking at INERIS of homogeneity and stability of test materials sent;
- entry of results by participants (j=+ XX max) on the website http://www.ineris.fr/cil/;
- data processing and statistical analysis by INERIS;
- dissemination of the final report together with the satisfaction survey.

The general organization of the interlaboratory comparison is as follows:

1. Feasibility study of the test

Each test material is subject to a feasibility study over several weeks. However, if the homogeneity and stability have been the subject of a previous study on similar test materials (matrix, concentration level) and prepared following the same procedures, the feasibility study will not be renewed.

2. Test announcement

INERIS informs the laboratories of the organization of a test by transmitting the Annual Program IM-1540 and the Registration Form IM-1542.

3. Registration of participants

INERIS receives the Registration Forms, gives each participant a confidential code, a identifier and a password and confirms the registration of each participant by mail by communicating its confidential code, its identifier and its password using Form IM-1063.

The IM1541 Instructions Form is submitted to participants before or/and when sending test materials, to inform them of the instructions (analytes, conservation means used, type of bottling used, etc.) and deadlines. It may also be posted on the website **http://www.ineris.fr/cil/**

4. Preparation of test materials

The test materials are prepared and packaged by INERIS, in compliance with the requirements of analytical standards. These requirements relate in particular to the nature of the implemented matrix, the level of concentration and mainly the preparation of test materials to ensure their quality in terms of stability and homogeneity.

The test materials are shipped in non-returnable containers by INERIS.

5. Shipping of test materials

The shipping of test materials is made by express delivery. The quality of the service is subject to tracking by INERIS.

The following documents will be attached to the test materials:

- Acknowledgement of Receipt IM-0223: **upon receipt of the packages**, the participant must send the completed document to INERIS
- Instruction Form IM-1541

The test materials are preferably shipped at the very beginning of each week to allow participants to initiate the analytic process before the end of the week.

Result entry forms are available on the site **http://www.ineris.fr/cil/**

6. Receipt and analysis of test materials by the participant

Immediately upon opening the package, the participant:

- will conduct a temperature control in the case of a shipment in refrigerated chamber. It will report the result of its measure in the Acknowledgment of Receipt IM-0223;
- will monitor the condition of the package as well as its composition and will record its results in the Acknowledgment of Receipt IM-0223;
- will immediately implement appropriate conservation means;
- will promptly inform INERIS of the receipt of the packages and of their condition by returning the Acknowledgment of Receipt duly completed by fax or email.

The participant will initiate the analytical process, using the methods specified.

7. Monitoring of test materials by the organizer

Controls on test materials sent will be performed during the analysis phase by the participants. INERIS will ensure that the test materials are stable and homogeneous by performing repeatability tests on several samples during the analysis phase, or a minimum of 10 analyzes of substances representative of each family.

8. Return of test data

The participant has a limited time to conduct analyzes and return its results. This period is usually 3 weeks.

The results will be transmitted by the participant via the **http://www.ineris.fr/cil/** site. For its online entry, the participant must have its confidential code, its identifier and its password. All these codes can be found in the Registration Confirmation MI-1063.

For some tests, a complementary form may be submitted to the participants. In this case, the entry of the results will be validated only after having completed it.

Assisted data entry is available online to help the participant use this entry software package.

A participant may, for reasons of its own, not perform the analysis of one or more substances. Incomplete analyzes papers are accepted.

In all cases, the results not taken into account in the statistical processing are:

- restituted values as "<LoQ",(*)
- values entered as zero "0";
- values for which a dilution or restitution error in the imposed unit is revealed (e.g. a factor of 1000)

(*) The rule applied is as follows:

Restitution of 4 values

	Data received	Data taken into account
1st case	C, C, C, <LQ	C, C, C
2nd case	C, C, <LQ, <LQ	C, C
3rd case	C, <LQ, C, <LQ	C, C
4th case	C, <LQ, <LQ, <LQ	none

Restitution of 2 values

	Data received	Data taken into account
1st case	C, <LQ	none
2nd case	<LQ, <LQ	none

Annex No. 3: Statistical processing and reporting of results

Statistical processing

The statistical processing of results shall be conducted by INERIS conforming to the provisions of:

- EN ISO/CEI 17043: « Conformity assessment - General requirements for proficiency testing». It gives the guidelines for implementing proficiency tests,
- ISO 5725 series: « Application of statistics. Accuracy (trueness and precision) of measurement methods and results »,
- ISO 13528: « Statistical Methods for Use in Proficiency Testing by Interlaboratory Comparisons ».

The determination of the assigned values depends on the type of test materials used:

- When test materials prepared from real matrices representative of the environment are implemented during the test, the assigned value will be based on the consensus of the results of the entire population participating in the test. It will be calculated using robust statistical methods.

  The advantage of the robust analysis is that the calculations of the assigned value (reference value), the confidence intervals and the performance statistics are not affected by the judgment of the data analyst. **The results are studied and treated with objectivity.**

  The standard deviation σ_{PT} chosen for the assessment of suitability is equal to the robust standard deviation s^*. It is determined from the results of the participants by applying Algorithm A of standard ISO 13528.

- When certified type test materials (certified standard solution, certified extract) are implemented during the test, the assigned value will be based on the reference value of the certified material used. In this framework, the participant must restitute its standard uncertainty in the result, respecting the enlargement factor mentioned in the Instruction Form IM-1541 to assess the performance of laboratories using the zeta score.

  The standard deviation σ_{PT} chosen for the assessment of suitability is equal to the robust standard deviation s^*. It is determined from the results of the participants by applying Algorithm A of standard ISO 13528.

The performance evaluation will depend on the type of test materials used:

- When test materials prepared from real matrices representative of the environment are implemented during the test, the evaluation of the performance will be performed using the z score. Thus, each participant will be able to position itself relative to the reference value.
- When test materials of certified material type (certified standard solution, certified extract) are used during a test, each participant may then determine its position with respect to this reference value through an assessment of its analytical performance expressed in z-score and zeta-score terms. If there is a significant difference between the z-score and zeta score, participant will examine its test procedure step by step, in order to consider the uncertainty, it has assessed in its procedure.

Furthermore, the research of suspect values and outliers will be realized with statistical tests of Cochran, Grubbs and Mandel. The aim is to help the organizer and the participant to identify the problem (repetability, trueness). The organizer can return to the participants that the results of some tests because the objectives of some of them are the same.

Reporting

The reporting of the test will be conducted in two steps:

- Sending an interim test report, one month after the closing date of entry of the results online. This report will gather the raw results of all participants, the mean, the standard deviation of repeatability, the variation of repeatability coefficient and the performance of each participant for each parameter and each test material. At this stage, **no detailed analysis of the data is performed**. This interim report will allow participants to have a first return of the test results.

The interim test report will be sent only to the participants.

- On completion of full statistical processing, and within 3 months after the intermediate report sending, the final report and satisfaction survey will be sent to participants. It will describe the conducting of the test and the results of statistical processing of data submitted by participants. This report will contain:
  - raw data,
  - values excluded from data set,
  - mean, standard deviation of repeatability for each laboratory,
  - descriptors before and after statistical treatment,
  - z score and zeta score (if certified material type) with a repartition graph,
  - a distribution curve of the average with standard deviation of repeatability of all participants,
  - the results of Cochran and Grubbs tests or Mandel, if relevant,

- Mandel consistency statistics h (trueness) and k (interlaboratory variability), if relevant,
- general and individual advices.

The final test report is public. It will be sent to:

- the participants,
- the French Biodiversity Agency (AFB),
- the French ministry of environment.

It will be available, without restriction, on the website http://www.ineris.fr/cil/

A satisfaction survey will be sent at the time of sending the final test report.

A meeting with the participants may be organized to present the test results and allow an exchange of information.