INERIS

INTERLABORATORY COMPARISON ANNUAL PROGRAM

INTERLABORATORY COMPARISONS

Year 2026

« Water »

“ Per- and polyfluoroalkyl substances (PFAS)” and “Total Oxidizable Precursors (TOP)”

Ineris - 235991 - 3049559 - v1.0

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

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1. CONTEXT

In the regulatory context of monitoring per- and polyfluoroalkyl substances (PFAS) in surface and groundwater (Directive (EU) 2026/805), and in industrial and urban wastewater (Orders of 20 June 2023 and 3 September 2025, and Directive (EU) 2024/3019), AQUAREF is organising an **interlaboratory comparison (ILC)** on the analysis of **43 PFAS using targeted methods**.

The wastewater-related orders also require the use of indicator methods to allow an overall assessment of PFAS contamination. Currently, two approaches have been identified:

- the **AOF** method (**Adsorbable Organic Fluorine**), according to the NF EN 18127 standard (2026);
- the **TOP** method (**Total Oxidizable Precursors**).

In this context, the 2026 PFAS ILC will also offer **volunteer laboratories the option of evaluating the TOP method**, providing initial feedback on its implementation. The Aquaref report[1] on the development of the TOP method is available to laboratories, even though the goal of this ILC is not to assess the laboratories on this same protocol.

This document contains all the information necessary for registration in the Proficiency Test. It contains:

- the participation procedures
- a description of the inter-laboratory programme

2. OBJECTIVES

The interlaboratory comparison 'Per- and polyfluoroalkyl substances (PFAS) and total oxidizable precursors (TOP)', organised by Ineris, aims to evaluate the performance of participating laboratories.

Participants will be able to see how accurately their results align with the reference values and evaluate their repeatability (see Appendix 4).

The exercise is also intended to:

- **Assess the evolution** of analytical practices and laboratory performance regarding PFAS analysis using targeted methods, compared to the ILC conducted in 2023 and the recommendations[2] issued in 2025;
- **Provide an initial overview** of practices and analytical capabilities related to the TOP method to build future Aquaref recommendations on a harmonised protocol.
- **Improve the monitoring** of these substances in aquatic environments in the future.

This interlaboratory comparison “Per- and polyfluoroalkyl substances (PFAS)” using targeted methods will be carried out under an accreditation in accordance with the ISO/IEC 17043 standard (accreditation No. 1-2291 - scope available at www.cofrac.fr) with the exception of the waste water test materials and of the substances identified by the symbol * and supplemented by the words "outside scope of accreditation" whenever possible (Tableau *1*, annex 1).

[1] Claudine Châtellier, Yohann Baillon, Ahmad El-Masri, Nina Huynh – Méthode indiciaire d’analyse pour les PFAS : l’analyse des précurseurs oxydables (TOP assay) – Rapport AQUAREF 2026

[2] Bénédicte Lepot, Nina Huynh – Comparaison interlaboratoires sur l’analyse de 30 PFAS par méthodes ciblées sur diverses eaux – Rapport AQUAREF 2024

Tableau 1 : Substances outside scope of accreditation

Substances* (outside scope of accreditation)	**Abreviation**	**CAS number**	**Type of PFAS**
Perfluorononane sulfonic acid*	PFNS*	68259-12-1	**PFSA** (Perfluoroalkyl sulfonic acids)
Perfluoroundecane sulfonic acid*	PFUnDS*	749786-16-1	
Perfluorotridecane sulfonic acid*	PFTriDS*	791563-89-8	
Trifluoroacetic acid*	TFA*	76-05-1	**PFCA** (Perfluoroalkyl carboxylic acids)
Perfluorooctadecanoic acid*	PFODA*	16517-11-6	
2H,2H-perfluorodecanoic acid* (2-perfluorooctyl ethanoic acid)*	8:2 FTCA*	27854-31-5	**FTCA** (Fluorotelomer carboxylic acids)
10,2 Fluorotelomer sulfonic acid*	10:2-FTSA*	120226-60-0	**FTSA** (Fluorotelomer sulfonic acids)
Perfluorobutanesulfonamide*	FBSA*	30334-69-1	**FASA and derivatives** (Per-polyfluoroalkane sulfonamides or acetic sulfonamide acids)
Perfluorohexanesulfonamide*	PFHxSA*	41997-13-1	
6:2 Fluorotelomer sulfonamide betaine*	6:2 FTAB*	34455-29-3	
2-perfluorohexyl ethanol (6:2)*	6:2 FTOH*	647-42-7	**FTOH** (Fluorotelomer Alcohol)
3-2-perfluorooctyl ethanol (8:2)*	8:2 FTOH*	678-39-7	

3. LABORATORIES CONCERNED

This interlaboratory comparison is opened to any French or European laboratory realizing measurements in natural waters and/or contributing to environmental monitoring.

The number expected is 20 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

Period for registration is set to:

July 10th to September 8th, 2026

on the following website:

https://comparaisons-interlaboratoires.ineris.fr

For the **first connection,** i.e without existing account, the laboratory shall create its account to access the website utilities. For that, the laboratory shall have the following information:

- enterprise identification (SIRET, DUNS,…),
- VAT Intra-community number (Europe only).

After validation of the profile by Ineris, the laboratory will be able to register to the proposed ILC.

Online help is available on the website.
If the laboratory already has an account, it can access directly to the registration step below.

During the registration phase, the laboratory shall have the information below:

- enterprise identification (SIRET, DUNS,…),
- an order from their internal services

Please note: Registration will be confirmed only after Ineris has received the corresponding order.

A confirmation will be sent to the participant within 15 days after the end of registration with his lab identification.

4.2. PRICE

AQUAREF is responsible for both organising and using the results. A financial contribution is requested to cover the costs of preparing and sending the ILC PFAS test materials using the targeted method.

Test	Amount in € excluding tax	20% VAT	Amount in € including tax
Per- and polyfluoroalkyl substances (PFAS) using targeted method	1 100	220	1 320
Total Oxidizable Precursors (TOP) including an analysis of PFAS before oxidation (PreTOP) and after oxidation (PostTOP)	/	/	/

Registration for the CIL TOP means that participants undertake to analyse at least the 20 perfluoroalkyl carboxylic acids (PFCA) and perfluoroalkyl sulfonic acids (PFSA) listed in Annex 2 of the Drinking Water Directive.

Invoices and payment will be established **when sending samples.**

Payment by credit card is not accepted.

4.3. INERIS COMMITMENTS

Ineris commits to abide by the EN ISO/CEI 17043 standard during the organization of the ILC.

Ineris commits to ensure information confidentiality in the online result submissions and anonymity in the result report by assigning a confidential code to each participant.

Ineris commits to protect participant identity confidentiality by only giving access to the confidential code to a limited number of people within the coordination group.

Ineris commits to notify all participants of any amendment to the ILC schedule and rules as soon as possible.

Confidential information, defined in the general terms and conditions of sale, and where applicable, by the confidentiality agreement, may be communicated to:

- the auditors commissioned by the certifying and accrediting bodies or in the context of an outsourced internal audit, which is themselves subject to a confidentiality agreement. The customer's order constitutes agreement to carry out these communications;
- an authority if the institute is required to do so by law.

Ineris commits to take into consideration any claim in accordance with the provisions of the Quality Manual §5.3: 'Stakeholders and listening to customers / Claims) available on the Ineris website at the following address: www.Ineris.fr.

4.4. COMMITMENTS OF PARTICIPANTS

Once registered, all participants commit to:

- Fill and return the "Return form" IM-0223 as soon as reception;
- Comply for each parameter of the standard procedures outlined in Annex 1 and in Annex 2 and Form IM-1541;
- Submit the results in full integrity without falsification nor collusion;
    - In case of NON-COMPLIANCE, Ineris reserves the right to disqualify the participant and to take appropriate action.
- Submit all the results on time, except in case of instrumental troubleshooting which shall be reported prior to the results submission deadline;
- Supply all related metadata upon request.

4.5. COMMUNICATION

Correspondence between Ineris and the participants should be mainly done electronically. Ineris rejects any responsibility in case of undelivered emails. The confidential code must be specified for further correspondence with the coordinator.

All documents related to the ILC can be downloaded at https://comparaisons-interlaboratoires.ineris.fr.

5. ANNEXES

Annex No.	Title
1	**ILC "Per- and polyfluoroalkyl substances (PFAS)" using targeted method**
2	**ILC "Total Oxidizable Precursors" TOP**
3	General organization of Proficiency Test
4	Statistical processing and restitution of test

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Annex No.1: ILC "Per- and polyfluoroalkyl substances (PFAS)" using targeted method

*** the following parameters/tests will not be carried out under our accreditation**

Program	ILC "Per- and polyfluoroalkyl substances (PFAS)" using targeted method	
Substances	**Substances**	**CAS number**
	PFSA (Perfluoroalkyl sulfonics acids)	
	Perfluorobutane sulfonic acid (PFBS)	375-73-5
	Perfluoropentane sulfonic acid (PFPeS)	2706-91-4
	Perfluorohexane sulfonic acid (PFHxS)	355-46-4
	Perfluoroheptane sulfonic acid (PFHpS)	375-92-8
	Perfluorooctane sulfonic acid (PFOS)	1763-23-1
	Perfluorodecane sulfonic acid (PFDS)	335-77-3
	Perfluorododecane sulfonic acid (PFDoDS)	79780-39-5
	PFCA (Perfluoroalkyl carboxylics acids)	
	Perfluorobutanoic acid (PFBA)	375-22-4
	Perfluoropentanoic acid (PFPeA)	2706-90-3
	Perfluorohexanoic acid (PFHxA)	307-24-4
	Perfluoroheptanoic acid (PFHpA)	375-85-9
	Perfluorooctanoic acid (PFOA)	335-67-1
	Perfluorononanoic acid (PFNA)	375-95-1
	Perfluorodecanoic acid (PFDA)	335-76-2
	Perfluoroundecanoic acid (PFUnDA)	2058-94-8
	Perfluorododecanoic acid (PFDoDA)	307-55-1
	Perfluorotridecanoic acid (PFTriDA)	72629-94-8
	Perfluorotetradecanoic acid (PFTeDA)	376-06-7
	Perfluorohexadecanoic acid (PFHxDA)	67905-19-5
	FTSA (Fluorotelomer sulfonics acids)	
	4,2 Fluorotelomer sulfonic acid (4:2-FTSA)	757124-72-4
	8,2 Fluorotelomer sulfonic acid (8:2-FTSA)	39108-34-4
	6,2 Fluorotelomer sulfonic acid (6:2-FTSA)	27619-97-2
	FASA et dérivés (Per-polyfluoroalkane sulfonamides ou Sulfonamides acetic acids)	
	Perfluorooctanate sulfonamide (FOSA)	754-91-6
	N-ethyl perfluoroctanesulfonamide (N-EtFOSA)	4151-50-2
	N-methylperfluorooctane sulfonamidoacetic acid (N-MeFOSAA)	2355-31-9
	N-ethylperfluorooctane sulfonamidoacetic acid (N-EtFOSAA)	2991-50-6
	N-methyl perfluorooctane sulfonamidoethanol (N-Me FOSE)	24448-09-7

	Acides per-polyfluoroalkyl ether carboxyliques	
	Dimer acid of hexafluoropropylene oxide (HFPO-DA)	13252-13-6
	4,8-Dioxa-3H-perfluorononanoic acid (DONA)	919005-14-4
	Others	
	2,2,3,3,4,4-hexafluoro-4-(trifluoromethoxy)butanoic acid (PFMBA)	863090-89-5
	Perfluoro-3,6-dioxaheptanoic acid (NFDHA)	151772-58-6

Substances outside scope of accreditation	**Substances***	**CAS**
	PFSA (Perfluoroalkyl sulfonics acids)	
	Perfluorononane sulfonic acid (PFNS) *	68259-12-1
	Perfluoroundécane sulfonic acid (PFUnDS) *	749786-16-1
	Perfluorotridécane sulfonic acid (PFTriDS) *	791563-89-8
	PFCA (Perfluoroalkyl carboxylics acids)	
	Trifluoroacetic acid (TFA)*	76-05-1
	Perfluorooctadécanoic acid (PFODA) *	16517-11-6
	FTCA (Fluorotelomer carboxylics acids)	
	2H,2H-perfluorodecanoic acid (2-perfluorooctyl ethanoïc acid) (8:2 FTCA)*	27854-31-5
	FTSA (Fluorotelomer sulfonics acids)	
	10,2 Fluorotelomer sulfonic acid (10:2-FTSA) *	120226-60-0
	FASA and derivatives (Per-polyfluoroalcanes sulfonamides or sulfonamides acetic acids)	
	Perfluorobutanesulfonamide (FBSA)*	30334-69-1
	Perfluorohexanesulfonamide (FHxSA)*	41997-13-1
	6:2 Fluorotelomer sulfonamide betaine (6:2 FTAB)*	34455-29-3
	FTOH (Fluorotelomer Alcohol)	
	2-perfluorohexyl ethanol (6:2) (6:2 FTOH)*	647-42-7
	3-2-perfluorooctyl ethanol (8:2) (8:2 FTOH)*	678-39-7

Date of receipt	**Week 39**	
Analytical standards	**Participant's choice**	
Test material	**Quality assurance**	Put materials at room temperature before analysis Blank (matrix provided by the organizer) Standard solution (matrix provided by the organizer) Specific investigation to identify the practices implemented in each laboratory (metadata survey)
	Tested matrices	1 natural water 1 wastewater from the treatment plant*

Concentration level	**A single level by matrix**	
Characteristics of Bottle	**Quality Assurance**	1 glass bottle of approximately 1000 ml for the blank matrix 1 plastic bottle (PE) of a few millilitres containing the standard solution
	Tested matrices	2 identical PE bottles of approx. 1000 ml for the natural water test material 2 identical PE bottles of approximately 1000 ml for the wastewater* from the treatment plant test material
Stabilisation	**No**	
Refrigerated transport	**Yes**	
Number of measurements per bottle	1 measurement per bottle: materials “natural water” and “wastewater” * and material quality assurance “blank” 2 measurements per bottle: material quality assurance “standard solution”	
Specific metadata	Specific survey to know the practices implemented in each laboratory	
Statistical processing implemented for test material – tested matrices	**Assigned value**	Materials “Natural water” and “Wastewater” *: 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.
	Performance	Materials “Natural water” and “Wastewater” *: z- score or z’-score (with uncertainty or/and heterogeneity material)
Statistical processing implemented for test material – quality assurance	**Blank**	Identification of results with concentrations greater than LOQ..
	Standard	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

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Annex No.2: ILC "Total Oxidizable Precursors (TOP)"

All tests will not be carried out under our accreditation

Program	**ILC "Total Oxidizable Precursors (TOP)"** This ILC includes the quantification of PFAS before (PreTOP) and after (PostTOP) oxidation. Participants taking part in the PFAS ILC using a targeted method can use the results from Annex 1 for the PreTOP results in Annex 2 if they apply the same analytical method for analysing both targeted PFAS and PFAS before oxidation (It will therefore not be necessary to repeat the analyses). Indeed, the test materials (Annex 2) come from the same batch as those in Annex 1. If this is not the case, the PreTOP quantification will need to be performed on the test materials listed in Annex 2. In any case, the PostTOP quantification will also need to be performed on the test materials listed in Annex 2.

Substances outside scope of accreditation

Substances *	CAS number
PFSA (Perfluoroalkyl sulfonics acids)	
Perfluorobutane sulfonic acid (PFBS) *	375-73-5
Perfluoropentane sulfonic acid (PFPeS) *	2706-91-4
Perfluorohexane sulfonic acid (PFHxS) *	355-46-4
Perfluoroheptane sulfonic acid (PFHpS) *	375-92-8
Perfluorooctane sulfonic acid (PFOS) *	1763-23-1
Perfluorononane sulfonic acid (PFNS) *	68259-12-1
Perfluorodecane sulfonic acid (PFDS) *	335-77-3
Perfluoroundécane sulfonic acid (PFUnDS) *	749786-16-1
Perfluorododecane sulfonic acid (PFDoDS) *	79780-39-5
Perfluorotridécane sulfonic acid (PFTriDS) *	791563-89-8
PFCA (Perfluoroalkyl carboxylics acids)	
Perfluorobutanoic acid (PFBA) *	375-22-4
Perfluoropentanoic acid (PFPeA) *	2706-90-3
Perfluorohexanoic acid (PFHxA) *	307-24-4
Perfluoroheptanoic acid (PFHpA) *	375-85-9
Perfluorooctanoic acid (PFOA) *	335-67-1
Perfluorononanoic acid (PFNA) *	375-95-1
Perfluorodecanoic acid (PFDA) *	335-76-2
Perfluoroundecanoic acid (PFUnDA) *	2058-94-8
Perfluorododecanoic acid (PFDoDA) *	307-55-1
Perfluorotridecanoic acid (PFTriDA) *	72629-94-8
Perfluorotetradecanoic acid (PFTeDA) *	376-06-7
Perfluorohexadecanoic acid (PFHxDA) *	67905-19-5

	Trifluoroacetic acid (TFA) * — 76-05-1
	Perfluorooctadecanoic acid (PFODA) * — 16517-11-6
	FTSA (Fluorotelomer sulfonics acids)
	4,2 Fluorotelomer sulfonic acid (4:2-FTSA) * — 757124-72-4
	8,2 Fluorotelomer sulfonic acid (8:2-FTSA) * — 39108-34-4
	6,2 Fluorotelomer sulfonic acid (6:2-FTSA) * — 27619-97-2
	10,2 Fluorotelomer sulfonic acid (10:2-FTSA) * — 120226-60-0
	FASA and derivatives (Per-polyfluoroalkane sulfonamides or Sulfonamides acetic acids)
	Perfluorobutanesulfonamide (FBSA)* — 30334-69-1
	Perfluorohexanesulfonamide (FHxSA)* — 41997-13-1
	Perfluorooctanate sulfonamide (FOSA) * — 754-91-6
	N-ethyl perfluoroctanesulfonamide (N-EtFOSA) * — 4151-50-2
	N-methylperfluorooctane sulfonamidoacetic acid (N-MeFOSAA) * — 2355-31-9
	N-ethylperfluorooctane sulfonamidoacetic acid (N-EtFOSAA) * — 2991-50-6
	N-methyl perfluorooctane sulfonamidoethanol (N-Me FOSE) * — 24448-09-7
	6:2 Fluorotelomer sulfonamide betaine (6:2 FTAB) * — 34455-29-3
	Per-polyfluoroalkyl ether carboxylics acids
	Dimer acid of hexafluoropropylene oxide (HFPO-DA) * — 13252-13-6
	4,8-Dioxa-3H-perfluorononanoic acid (DONA) * — 919005-14-4
	FTCA (Fluorotelomer carboxylics acids)
	2H,2H-perfluorodécanoic acid (2-perfluorooctyl ethanoïc acid) (8:2 FTCA) * — 27854-31-5
	FTOH (Fluorotelomer Alcohol)
	2-perfluorohexyl ethanol (6:2) (6:2 FTOH) * — 647-42-7
	3-2-perfluorooctyl ethanol (8:2) (8:2 FTOH) * — 678-39-7
	Others
	2,2,3,3,4,4-hexafluoro-4-(trifluoromethoxy)butanoïc acid (PFMBA) * — 863090-89-5
	Perfluoro-3,6-dioxaheptanoic acid (NFDHA) * — 151772-58-6
	The PostTOP analysis should at least focus on the 20 PFCA and PFSA from the drinking water directive. These substances are highlighted in blue in the above table.
Date of receipt	**Week 39**
Analytical standards	**Participant's choice**

Test material	**Quality assurance**	Put materials at room temperature before analysis Blank (matrix provided by the organizer) Specific investigation to identify the practices implemented in each laboratory (metadatas survey)
	Tested matrices	1 natural water * 1 wastewater from the treatment plant*
Concentration level	**A single level by matrix**	
Characteristics of Bottle	**Quality Assurance**	1 glass bottle of approximately 1000 ml for the blank matrix
	Tested matrices *	Natural water *: 2 identical PE bottles of approx. 250 ml, 1 for PreTOP and 1 for PostTOP *Note: Depending on the number of participants, the number of vials may be doubled.* Wastewater from the treatment plant*: 2 identical PE bottles of approx. 250 ml, 1 for PreTOP and 1 for PostTOP *Note: Depending on the number of participants, the number of vials may be doubled.*
Stabilisation	**No**	
Refrigerated transport	**Yes**	
Number of measurements per bottle	1 measurement per bottle: materials “natural water *” and “wastewater *” and material quality assurance “blank”	
Specific metadata	Specific survey to know the practices implemented in each laboratory	
Statistical processing implemented for test material – tested matrices	**Assigned value**	Materials “Natural water *” and “Wastewater *”: 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.
	Performance	Materials “Natural water *” and “Wastewater *”: z- score or z’-score (with uncertainty or/and heterogeneity material)
Statistical processing implemented for test material – quality assurance	**Blank**	Identification of results with concentrations greater than LOQ..
Monitoring of the homogeneity and the stability	**Organization**	As the test materials (see Annex 2) are from the same batch as those in Annex 1, Ineris will only monitor the homogeneity and stability of the materials in Annex 1.

Annex No. 3: General organization of an interlaboratory comparison

The typical ILC schedule is specified below.

- ILC feasibility study (in order to define and assess outline of the optimal test conditions);
- Sampling, sample fortification if required; sample packaging;
- Sending (d = 0) of the test materials to the participants.
- Receipt of the test materials by the participants (d = +1);
- Analysis of the test materials by the participants (d = +1 to +15); and checking by Ineris of the test material homogeneity and stability all along the ILC, if necessary;
- Result submissions by the participants at https://comparaisons-interlaboratoires.ineris.fr;
- Data processing and statistical analysis performed by Ineris;
- Sending a preliminary report;
- Sending of the final report including a satisfaction survey form.

The general organization of the interlaboratory comparison is as follows:

1. Feasibility study of the test

Each test material undergoes a feasibility study over several weeks. However, if homogeneity and stability were 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 and publishing information on the website.

3. Participant registration

Ineris receives the registration request and confirms the registration of the participant by email and provides its laboratory identification.
The instruction form IM1541 is forwarded to participants prior to, or with, the test materials in order to provide instructions (compounds to quantify, storage, handling, etc.…) and deadlines. The form is also available at https://comparaisons-interlaboratoires.ineris.fr

4. Test material preparation

Test materials are prepared and packaged by Ineris in compliance with official guidelines. Such guidelines deal especially with the nature of the matrices, the concentration levels and the test material preparation in order to assure their quality in terms of stability and homogeneity.

The test materials are sent by Ineris. They are sent in disposable packaging.

5. Sending of the test materials

The test materials are sent by express delivery post. The delivery quality is monitored by Ineris.

The following documents are included in the package:

- Receipt acknowledgements (form IM-0223). Upon receipt of the package, participants are required to send it back to Ineris fully completed;
- Instruction form IM-1541.

Test materials are preferentially shipped at the beginning of the week in order to allow the participants to start the analytical process before the end of the week.

All the forms are available at https://comparaisons-interlaboratoires.ineris.fr.

Shipment of test materials to foreign and overseas:

- Visible labeling of the package and detailed proforma invoice to emphasize the need to keep the samples refrigerated (5 ±3°C);
- Shipment of the package in refrigerated conditions to compensate for the effects of any delays, independent of our organisation, on our materials;
- Communication to participants by e-mail of the date on which the package was taken over by the carrier and the tracking number for tracking the package (carrier, customs, delivery, etc.).

6. Receipt and analysis of test materials

Upon opening of the package, participants shall:

- Perform a temperature control check and write the results on the receipt form IM-0223;
- Returning the envelope containing temperature recorder to Ineris (if present);
- Inspect the package as well as its contents and write any relevant information on the receipt form IM-0223;
- Put the test materials in appropriate storage conditions immediately;

Participants shall start the analysis of the test materials as soon as possible.

7. Checking of the test materials

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, i.e a minimum of 10 analyzes of substances representative of each family.

8. Data Submission

The usual time period for the participants to achieve the analyses and submit their results is 4 weeks.

The results will be sent by the participant using the https://comparaisons-interlaboratoires.ineris.fr site with its personal account.

For specific tests, additional forms may be provided to the participants. Results can only be validated once the requested forms are fully filled.

The technical support is available online in order to help the participants to use the website for results submissions.
Incomplete results may be accepted if a participant, for specific reasons, is not able to analyze one or several compounds.
Results not considered for the statistical analysis are the following:

- values below the limit of quantification “<LOQ”,*
- values entered as zero "0";
- values for which a systematic error (errors of dilution or unit of measurement) is identified (for instance by a factor 1000).

* The methodology will be as follows:

Restitution of 2 values

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

Annex No. 4: Statistical processing and reporting of results

1. Statistical processing

Statistical processing of the results shall be carried out by Ineris in accordance with the requirements of:

- EN ISO/CEI 17043 giving the guidelines for implementing proficiency tests,
- ISO 5725 series: « 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:

Materials "natural water" and "wastewater":

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.

Nevertheless, in the case of an insufficient population size (<10), the assigned value can be set as, for example, equal to the spiking value.

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. However, if regulatory or normative requirements exist regarding uncertainty or when the population size is too reduced (<10), σ_{pt} may be set.

Material "Standard solution":

The assigned value will be based on the reference value of the certified material used or of the value calculated from the masses (formulation). 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. However, if regulatory or normative requirements exist regarding uncertainty or when the population size is too reduced (<10), σ_{pt} may be set.

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

- When test materials prepared from real matrices representative of the environment (materials "natural water" and "wastewater") are implemented during the test, the evaluation of the performance will be performed using the z (or z') score. Thus, each participant will be able to position itself relative to the assigned value.

- When test materials of type “standard solution” 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 has assessed in its procedure.

Furthermore, the research of suspect values and outliers will be achieved 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 organiser reserves the right to return only the results of certain tests to participants, as some of them have the same objective.

2. Restitution of the results

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

- Sending a preliminary test report, two months 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 preliminary report will allow participants to have a first return of the test results.

The preliminary report will be sent to participants only.

- On completion of full statistical processing, and within 3 months after the preliminary 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:
  - values discarded from data set,
  - raw data,
  - means, standards deviation after statistic treatment,
  - z score and zeta score (case of standard solution) 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 **restricted**. It will be sent to:

- the French National Agency for Water and Aquatic Environments
- the French ministry of environment.
- the participants and / or available at https://comparaisons-interlaboratoires.ineris.fr via their personal space.

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

A meeting with the participants may be organised to present the test results and encourage information sharing.

A public summary report will be prepared by AQUAREF to present the performance of the laboratories, identify the analytical difficulties encountered, and make recommendations.