INTERLABORATORY COMPARISON
Year 2024

Ambient air

Polycyclic Aromatic Hydrocarbons (PAHs)

MIV-227298-2789795-v1.0

Organizer: Ineris
Environmental Characterisation and Impacts on Human and Biodiversity Department

Function	First Name/Last Name	E-mail	Phone
Head of "Methods and developments in environmental analysis" Unit	Hugues BIAUDET	hugues.biaudet@ineris.fr	+33 3 44 55 66 19
Scientist Air quality Methods and developments in environmental analysis Unit	Alexandre ALBINET	alexandre.albinet@ineris.fr	+33 3 44 55 64 85
Coordinator	Sylvain BAILLEUL	sylvain.bailleul@ineris.fr	-

Ineris

Parc technologique Alata – PO Box 2- F-60550 Verneuil-en-Halatte

☎ *+33 (0)3.44.55.66.77 -* 🌐 *: www.ineris.fr*

TABLE OF CONTENTS

1. BACKGROUND

The Directive 2004/107/EC of the European Parliament enforces the monitoring of polycyclic aromatic hydrocarbons (PAHs) in ambient air.

Note: several annexes have been amended by the Directive (UE) 2015/1480.

Article 9 from the French law of October 21st, 2010 regarding air quality monitoring and public information imposes "the participation to interlaboratory comparisons (ILCs) on laboratories performing the chemical analyses for the French air quality monitoring networks".

In this aim, Ineris organizes an ILC to satisfy this requirement.

This document summarizes all the requested details for the registration to this ILC.

The following information included is:

- Participation conditions,
- ILC description.

2. OBJECTIVES

An ILC participation gives the opportunity to the laboratories:

- to compare its results with those of other laboratories carrying out same type of analysis;
- to assess skills of the operators for a given technique;
- to evaluate their analytical (quantification) procedure for a given matrix;
- to meet the requirements of quality references;
- to improve its measurement quality;
- to demonstrate its measurement proficiency in a given environment in order to meet the regulatory requirements to obtain, for an example, an approval or an accreditation.

The achievement of these objectives is evaluated with a performance score (z-score) allowing participants to estimate the accuracy of their results from an assigned value known as reference value, calculated by proven statistical algorithms.

Furthermore, this exercise will help identifying any problems related to the analysis of PAHs in ambient air.

3. AIM OF THE STUDY

The purpose of the study is to implement and carry out an ILC for the analysis of the following species on 3 PM_{10} ambient air filter samples:

- ✓ Benzo[a]anthracene (B[a]A),
- ✓ Benzo[g,h,i]perylene (B[g,h,i]P),
- ✓ Benzo[a]pyrene (B[a]P),
- ✓ Benzo[b]fluoranthene (B[b]F)*,
- ✓ Benzo[j]fluoranthene (B[j]F)*,
- ✓ Benzo[k]fluoranthene (B[k]F)*,
- ✓ Dibenzo[a,h]anthracene (D[a,h]A),
- ✓ Indeno[1,2,3-cd]pyrene (In[1,2,3-cd]P).

* *Note*: the sum of the benzofluoranthenes must be also reported.

This ILC exercise may be postponed or cancelled if the number of participants is lower than 12.

4. PARTIES INVOLVED

This ILC is intended for laboratories performing analyses of PAHs in ambient air.
Due to technical limitations, the number of participants is limited to **25**. The priority is given to laboratories performing analyses for regulatory purposes.

5. GENERAL TECHNICAL REQUIREMENTS

5.1. REGISTRATION PROCEDURES

Period for registration is set to:

26 January 2024 to **28 June 2024**

on the following website:

https://comparaisons-interlaboratoires.ineris.fr

For the **first connection,** i.e without account already existing, the laboratory shall create its account for accessing to website utilities. For that, the laboratory shall have the information below:

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

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

Online help is available on the website.

If the account already existing, the laboratory will access directly to the registration step below.

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

- Enterprise identification (SIRET, DUNS, …),
- A payment **order** from their internal services.

Please note: The BIPEA quote and the form available on the website are not accepted as valid orders. Without a valid order, report will not be provided to the participant.

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

5.2. PRICE

Title	Amount in € before tax	20% VAT	Total amount in €, tax included
24-227298-PAH	1799.00	359.80	2158.80

Invoice is established **when sending samples**.

Payment by credit card is not accepted.

5.3. 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.

Ineris commits to take into consideration any claim and to act if required. All claims should be submitted to the attention of the ILC coordinator.

5.4. PARTICIPANTS' COMMITMENTS

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 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 the case of instrumental troubleshooting which shall be reported prior to the result submission deadline;
- Supply all related metadata upon request.

5.5. 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.

6. ANNEXES

Annex number	Title	Pages
1	ILC specifications	1
2	PAHs listed in the Directive 2004/107/EC	1
3	Overall organization of an ILC	3
4	Statistical analysis and results	2

Annex 1: ILC specifications

Test	24-227298_Filtre (Filter)
Substances to be analysed	Benzo[a]anthracene, Benzo[a]pyrene, Benzo[g,h,i]perylene, Benzo[b]fluoranthene, Benzo[j]fluoranthene, Benzo[k]fluoranthene (or sum of benzofluoranthenes), Dibenzo[a,h]anthracene, Indeno[1,2,3,c-d]pyrene
Date of receipt	Week 37
Deadline for results submission	11th October 2024
Analytical methods	EN 15549:2008, ISO 12884 :2000, CEN/TS 16645 or equivalent
Test materials	PM_{10} ambient air samples (filter punch, ∅ ≈ 37 mm)
Number of samples	3 ambient air filters + 1 blank filter
Number of measurements per sample	3
Stability	No
Refrigeration	Yes
Statistical analysis	Assigned value: Robust average by application of the algorithm A from the ISO 13528 standard procedure Standard deviation for assessment: 17% for B[a]P, 25% for the others PAH.
	Performance: z-score except for a population < 8 after exclusion of missing or aberrant results => indicative value
Monitoring of the homogeneity of test materials	No[1]
Monitoring of the stability of test materials	No[1]

[1]: see §7 Annex 3

Annex 2: PAHs listed in the Directive 2004/107 / EC of 15 December 2004

Polycyclic Aromatic Hydrocarbons	
SUBSTANCES	Benzo[a]anthracene Benzo[a]pyrene* Benzo[b]fluoranthene Benzo[j]fluoranthene Benzo[k]fluoranthene Dibenzo[a,h]anthracene Indeno[1,2,3-cd]pyrene

* Regulated compound with target value on annual average of 1 ng m^{-3} in the PM_{10} fraction.

Annex 3: Overall ILC organization

The typical ILC schedule is specified below.

- ILC feasibility study (in order to define and assess outline of the optimal test conditions);
- Sampling and sample packaging (sample fortification, if required);
- 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 +31).
  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 of the final report including a satisfaction survey form.

The general organization of the ILC is as follows:

1. ILC feasibility study

Each test material undergoes a feasibility study over several weeks. The homogeneity and the stability have been already evaluated previously on similar test materials (matrices, concentration levels). Test materials will be prepared using similar procedures so that the feasibility study won't be performed again this year.

2. ILC announcement

Ineris informs the laboratories of the organization of an upcoming test by sending the annual program (form IM-1540).

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.

Sending of the test materials is performed by Ineris.

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 to Ineris;

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.

6. Receipt and analysis of the test materials

Upon opening of the package, participants shall:

- Perform a temperature control check and write the results on the receipt form IM-0223;
- 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;
- Inform Ineris of the receipt and conditions of package by returning the receipt form IM-0223 by email.

Participants shall start the analysis of the test materials as soon as possible. The envelope containing temperature recorder shall be returned to Ineris.

7. Checking of the test materials

Given the strong experience and knowledge on such test materials (ambient air filter samples), homogeneity and stability evaluations will be not performed again during this new ILC. The homogeneity of samples collected using the Graseby – Andersen sampler has been tested several times during previous ILCs (Verlhac, 2014, Verlhac and Albinet, 2015).

8. Data Submission

The usual time period for the participants to achieve the analyses and submit their results is of 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 result 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 (LQ)*;
- 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:

	Data received	Data considered
1st case	C, C, C	C, C, C
2nd case	C, C, <LQ	C, C
3rd case	C, <LQ, <LQ	None
4th case	<LQ, <LQ, <LQ	None

Annex 4: Statistical processing and return of the test

1. Statistical Analysis

The statistical analysis of the results is performed according to the EN ISO/CEI 17043 standard procedure and the following requirements:

- Standard methods 1, 2 and 5 of the ISO 5725 series: “Accuracy (trueness and precision) of measurement methods and results”;
- ISO 13528 standard procedure: “Statistical methods for use in proficiency testing by interlaboratory comparisons »;
- X06-050 standard procedure “Statistical application – Study of distribution normality.” The determination of assigned values is done as follows:

The standard deviation for the evaluation of the chosen proficiency σ_{pt} is:

- Equal to the third of the measurement uncertainty specified in the European Directive 2004/107/EC i.e. 50%/3 = 16.7% rounded up to 17% for B[a]P;
- Equal to 75% / 3 = 25% for the other PAHs.

The performance evaluation will be carried out using the z-score. Each participant will be able to position itself in relation to the assigned value.

2. ILC restitution of the results

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

- Sending a preliminary 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 report will allow participants to have a first return of the test results.
- 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 the dataset;
  - Raw results;
  - The averages and standard deviations after statistical analysis;
  - The statistical distribution graphs;
  - A bar chart illustrating the lab performances on the same graph (z-score).

The distribution of the final test report will be **restricted**. It will be sent in electronic format only to the participants.

A satisfaction survey will be sent along with the final test report.