| INERIS | INTERLABORATORY COMPARISON 25-232849 |
|---|---|

# INTERLABORATORY COMPARISONS
## *Year 2025*

## Ambient air
## “Levoglucosan, mannosan, galactosan”

Ineris - 232849 - 2984842 - v1.0

13 June 2025

Organizer: LCSQA - Ineris


| Function | First and last Name | Contact details | |
|---|---|---|---|
| | | email | Phone |
| Head of “Methods and developments in environmental analysis” Unit | Hugues BIAUDET | hugues.biaudet@ineris.fr | +33 3 44 55 66 19 |
| Research Director on Air quality. Methods and developments in environmental analysis Unit | Alexandre ALBINET | alexandre.albinet@ineris.fr | +33 3 44 55 64 85 |
| Coordinator | Sylvain BAILLEUL | coordonnateur_air_ambiant@cil-ineris.fr | - |

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

☎ +33 (0)3.44.55.66.77

Internet: www.ineris.fr. www.lcsqa.org.


LCSQA
Laboratoire Central de Surveillance de la Qualité de l'Air

# TABLE OF CONTENTS

## 1. CONTEXT

Levoglucosan, and its isomers (galactosan and mannosan) are recognised as a biomass combustion marker and can therefore be used to evaluate the sources of particulate matter (PM) in ambient air from the chemical characterisation of PM samples collected on filters. Although it is not compulsory, the European Directive (EU) 2004/2881, on ambient air quality and cleaner air for Europe, recommends measuring levoglucosan as part of the chemical composition of PM. The reference method CEN/TS 18044, related for the determination of the concentration of levoglucosan by chromatography, meets this objective.

To assess the performance of laboratories in implementing this recent dedicated standard, the French Central Air Quality Monitoring Laboratory (LCSQA) and the French National Institute for industrial Environment and Risks (Ineris) are offering this analytical interlaboratory comparison (ILC).

In addition, article 9 of the french Order of 21 October 2010 on air quality monitoring procedures and public information requires laboratories carrying out chemical analyses on behalf of approved air quality monitoring bodies to participate in interlaboratory tests.

Whether it is for carrying out the preliminary assessment or for the actual monitoring of these compounds in France, the Approved Air Quality Monitoring Associations (AASQA) must work with analytical laboratories to extract and analyse the ambient air PM samples.

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

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 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 identify the problems related to the application of the technical specification XP CEN/TS 18044 for the measurement of levoglucosan.

## 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:

- Levoglucosan,
- Mannosan,
- Galactosan

***A test may be postponed or canceled if the number of participants is lower than 10.***

### 3.1. FILTER

Two ambient air PM10 samples (quartz fiber filters) and one field filter blank shall be analyzed to determine levoglucosan, mannosan and galactosan amounts.

### 3.2. REFERENCE MATERIAL

One reference material (urban dust or PM-like) shall be analyzed to determine levoglucosan, mannosan and galactosan amounts.

## 4. LABORATORIES CONCERNED

This ILC is intended for laboratories performing analyses of levoglucosan in ambient air according to the reference method CEN/TS 18044.

Due to technical limitations, the number of participants is limited to **20**. The priority is given to laboratories performing analyses for regulatory purposes.

## 5. GENERAL TECHNICAL REQUIREMENTS

### 5.1. REGISTRATION PROCEDURE

Period for registration is set to:

**16 June 2025 to 29 August 2025**

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 laboratory already has an account, it can access directly to the registration step below.

During the registration phase, the laboratory shall have the **enterprise identification** (SIRET, DUNS, …).

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

### 5.2. PARTICIPATION FEES

No fees are requested. This comparison is funded by the LCSQA.

### 5.3. INERIS COMMITMENTS

Ineris commits to abide by the technical requirements of EN ISO/CEI 17073 standard during the organization of this 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. Nevertheless, the list of participants will be included in the final report without possibility of linking the confidential code to a participant.

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 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.

### 5.4. 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 Form IM-1541;
- Submit the results in full integrity without falsification or 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 No. | Title |
|---|---|
| 1 | Description of the test |
| 2 | General organization of Proficiency Test |
| 3 | Statistical processing and restitution of test |

## Annex No.1: Description of the tests

| Test | 25-232849_Filtre (Filter) |
|---|---|
| Substances to be analysed | Levoglucosan, mannosan, galactosan |
| Date of receipt | Week 39 |
| Deadline for results submission | 24th October 2025 |
| Analytical methods | CEN/TS 18044 or equivalent |
| Test materials | $PM_{10}$ ambient air samples<br>(filter punch, ∅ ≈ 37 mm) |
| Number of samples | 2 ambient air filters + 1 blank filter |
| Number of measurements per sample | 3 |
| Stability | No |
| Refrigeration | Yes |
| Statistical analysis | Assigned value:<br>Robust average by application of the algorithm A from the ISO 13528 standard procedure<br>Standard deviation for assessment:<br>Robust standard deviation calculated by applying the algorithm A of standard ISO 13528 |
| | Performance:<br>z-score except for a population < 8 after exclusion of missing or aberrant results => indicative value |
| Monitoring of the homogeneity of test materials | Yes |
| Monitoring of the stability of test materials | Yes |

## Annex No.1: Description of the tests

| Test | 25-232849_Dust |
|---|---|
| Substances to be analysed | Levoglucosan, mannosan, galactosan |
| Date of receipt | Week 39 |
| Deadline for results submission | 24th October 2025 |
| Analytical methods | CEN/TS 18044 or equivalent |
| Test materials | Reference material (urban dust or $PM_{10}$-like) |
| Number of samples | 1 bottle of ≈ 120 mg |
| Number of measurements per sample | 3 |
| Stability | No |
| Refrigeration | Yes |
| Statistical analysis | Assigned value:<br>Robust average by application of the algorithm A from the ISO 13528 standard procedure<br>Standard deviation for assessment:<br>Robust standard deviation calculated by applying the algorithm A of standard ISO 13528 |
| | Performance:<br>z-score except for a population < 8 after exclusion of missing or aberrant results => indicative value |
| Monitoring of the homogeneity of test materials | No |
| Monitoring of the stability of test materials | No |

## Annex No. 2: 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 +31; 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 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 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.

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 document is 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.

Test materials are preferentially shipped at the beginning of the week 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 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;

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 analyses of substances representative of each family.

Some materials may be exempted from this control if previous data have shown that the preparation procedures provide sufficient homogeneity and stability or if a SRM is used.

## 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.

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 “<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 3 values**

| | 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 No. 3: 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:

- 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 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.

The standard deviation $\sigma_{pt}$ 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), $\sigma_{pt}$ may be set.

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.

### 2. Restitution of the results

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

- Sending a preliminary test report, one to three 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 with a repartition graph,
    - a distribution curve of the average with standard deviation of repeatability of all participants,
    - Mandel consistency statistics k (interlaboratory variability), if relevant,
    - general and individual advice.

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

It will be available, without restriction, on the following website:

- https://comparaisons-interlaboratoires.ineris.fr,
- https://www.lcsqa.org.

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