LCSQA

Laboratoire Central de Surveillance de la Qualité de l'Air

# Interlaboratory comparison organized for the European laboratories involved in the analysis of levoglucosan and its isomers in ambient air

Comparaison interlaboratoires organisée pour les laboratoires européens impliqués dans l'analyse du lévoglucosan et de ses isomères dans l'air ambiant

June 2026

Groupement d'intérêt scientifique

RÉPUBLIQUE FRANÇAISE
Liberté
Égalité
Fraternité

IMT Nord Europe
École Mines-Télécom
IMT-Université de Lille

INERIS
maîtriser le risque pour un développement durable

LABORATOIRE NATIONAL DE MÉTROLOGIE ET D'ESSAIS
LNE

«L'expertise au service de la qualité de l'air»

Travaux réalisés par
Ineris
dans le cadre du

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

# Interlaboratory comparison organized for the European laboratories involved in the analysis of levoglucosan and its isomers in ambient air

Sylvain Bailleul - INERIS

**Vérification :** Alexandre Albinet - INERIS

**Approbation :** Ineris : Document approuvé le 15/06/2026 par DURIF MARC

**Liste des personnes ayant participé à l'étude**

Valérie Minguet – Ineris

# Table of contents

# Abstract

Levoglucosan and its isomers (galactosan and mannosan) are recognised as markers of biomass combustion and can therefore be used to assess the sources of particulate matter in ambient air through the chemical characterisation of filter samples. Although not mandatory, the European Directive (EU) 2004/2881 on ambient air quality recommends measuring levoglucosan as part of the chemical composition of PM. The reference method CEN/TS 18044 (2024), which specifies the determination of levoglucosan and its isomers by chromatography, meets this objective. To evaluate laboratory performance in implementing this recent reference method, the French Central Laboratory for Air Quality Monitoring (LCSQA) organised an interlaboratory comparison (ILC) in the second half of 2025. Registration was open to European laboratories.

This exercise included matrices with different concentration levels to reflect the usual working ranges of laboratories analysing filters from high-volume or low-volume samplers. Each participant therefore received two punches from ambient air $PM_{10}$ filters, one laboratory blank filter punch, and a certified reference material (CRM, NIST SRM 1649b, urban dust). Ten European laboratories, including five from France, took part in this ILC.

The main findings were as follows:

- The results obtained in this ILC did not allow any conclusions to be drawn regarding a potential method-related effect.
- The reproducibility standard deviations obtained for the ambient air filters were approximately 20 - 42% for levoglucosan, 35 - 58% for galactosan, 32 - 37% for mannosan. For the CRM, they were 93% for levoglucosan, 122% for galactosan, and 109% for mannosan. It should be noted that, due to an insufficient number of laboratories submitting quantified CRM results, performance scores could not be calculated for galactosan and mannosan.
- The uncertainties observed were higher than those described in the reference method CEN/TS 18044. Although filter sections F1 and F2 analysed in this ILC showed similar concentrations, the significant difference between their uncertainties was unexpected. For levoglucosan, uncertainties were 85% for filter F1 and 40% for filter F2, with respective concentrations of 76 and 68 µg per filter.
- Comparison with uncertainties obtained in previous ILCs revealed substantial variability. For levoglucosan, uncertainties ranged from 32% to 85%.

In conclusion, given the results obtained and the large dispersions observed, which prevent an effective assessment of laboratory performance, it would be advisable to organise a new ILC on the analysis of levoglucosan and its isomers in ambient air.

# Résumé

Le lévoglucosan et ses isomères (le galactosan et le mannosan) sont reconnus comme des marqueurs de la combustion de biomasse et peuvent donc être utilisés pour évaluer dans l'air ambiant les sources de particules collectées sur filtres. Bien que non obligatoire, la Directive européenne (UE) 2004/2881 relative à la qualité de l'air ambiant recommande la mesure du lévoglucosan dans le cadre de l'analyse de la composition chimique des PM. La méthode de référence CEN/TS 18044 (2024), qui spécifie la détermination du lévoglucosan et de ses isomères par chromatographie, répond à cet objectif. Afin d'évaluer la performance des laboratoires dans la mise en œuvre de cette récente norme, le Laboratoire Central de Surveillance de la Qualité de l'Air (LCSQA) a organisé une comparaison interlaboratoires analytique (CIL) au deuxième semestre 2025. Les inscriptions étaient ouvertes aux laboratoires européens.

Cet exercice comprenait des matrices de concentrations différentes afin de prendre en compte les gammes de travail habituelles des laboratoires réalisant l'analyse de filtres issus de prélèvements haut débit ou bas débit. Chaque participant a donc reçu deux poinçons de filtres issus de prélèvements $PM_{10}$ d'air ambiant, un poinçon de filtre blanc de laboratoire, un matériau de référence certifié (MRC, NIST SRM1649b, urban dust). Dix laboratoires européens, dont cinq français, ont participé à cette CIL.

Les éléments remarquables obtenus ont été les suivants :

- Les résultats observés dans le cadre de cette CIL ne permettent pas de tirer des conclusions quant à un éventuel effet lié à la méthode.
- Les écarts type de reproductibilité obtenus pour les filtres air ambiant étaient de l'ordre de de 20 - 42 % pour le lévoglucosan 35 - 58 % pour le galactosan, 32 - 37 % pour le mannosan. Pour ce qui concerne le MRC, ils étaient de 93 % pour le lévoglucosan, 122 % pour le galactosan et 109 % pour le mannosan. Il est à noter que compte tenu d'un nombre de laboratoires insuffisant dans la remise de résultats quantifiés pour le MRC, les scores de performance pour le galactosan et le mannosan n'ont pu être calculés.
- Les incertitudes observées étaient plus importantes que celles décrites dans la méthode de référence CEN/TS 18044. Bien que les sections de filtres F1 et F2 étudiés lors de cette CIL présentaient des concentrations similaires, la différence significative entre les incertitudes obtenues était surprenante. Elle était pour le lévoglucosan de 85 % pour le filtre F1 et de 40% pour le filtre F2 avec des concentrations respectives de 76 et 68 µg par filtre.
- La comparaison des incertitudes obtenues lors de CILs précédentes a révélé une grande variabilité dans les valeurs observées. Dans le cas du lévoglucosan, ces incertitudes varient de 32 % à 85 %.

Finalement, compte tenu des résultats observés et des dispersions importantes constatées, qui ne permettent pas d'évaluer efficacement les performances des laboratoires, il serait utile d'organiser une nouvelle CIL sur l'analyse du lévoglucosan et de ses isomères dans l'air ambiant.

# Definitions

| | |
|---|---|
| Bias | Difference between the laboratory mean and the assigned value of the test material (Spiking value, x* or $X_{CRM}$). It is expressed in relative or absolute value, |
| $CV_R$ | Reproducibility variation coefficient in %, |
| CVr | Repeatability standard deviation in %, |
| Standard deviation | Standard deviation of x measurements, |
| $IC_R$ | Reproducibility confidence interval, |
| $IC_r$ | Repeatability confidence interval, |
| Mean | Mean of x measurements, |
| Number of decimals | Number required in the instruction form, |
| $\sigma_{pt}$ | Standard deviation for assessment (stipulated, perceived or s*), |
| z score | Performance criterion provided to each participant making it possible to measure its deviation with respect to the assigned value, |
| z' score | Z score corrected with the uncertainty from assigned value or/and standard deviation of sample if nonhomogeneous. His value is lower than the z score, |
| s* | Robust standard deviation to evaluate the aptitude obtained by the A algorithm, |
| $S_L$ | Inter-laboratory standard deviation, |
| $S_R$ | Reproducibility standard deviation, |
| $S_r$ | Repeatability standard deviation, |
| $s_r$ | Repeatability standard deviation of the laboratory = standard deviation of the x measurements from the laboratory, |
| $s_s$ | Between sample standard deviation, |
| $u_{xpt}$ | Type uncertainty $u_{xpt}$, |
| Assigned value | Value assigned to a particular and recognized property, sometimes by convention, as having an uncertainty appropriate for a given use (NF ISO 13528), |
| x* | Robust mean obtained by the A algorithm, |
| $x_{pt}$ | Assigned value for assessment. |

# 1 Background

Levoglucosan and its isomers (galactosan and mannosan) are recognised as markers of biomass combustion and can therefore be used to assess the sources of particulate matter (PM) in ambient air through the chemical characterisation of PM samples collected on filters. Although not mandatory, European Directive (EU) 2004/2881 on ambient air quality recommends measuring levoglucosan as part of PM chemical composition. The reference method CEN/TS 18044 (2024), which specifies the determination of levoglucosan and its isomers by chromatography, fulfils this requirement.

To evaluate how effectively laboratories are applying this recent dedicated standard, the French Central Air Quality Monitoring Laboratory (LCSQA) and the French National Institute for Industrial Environment and Risks (Ineris) have organised in 2025 a European analytical interlaboratory comparison (ILC) focusing on the analysis of levoglucosan and its isomers in ambiant air. In addition, Article 9 of the French Order of 21 October 2010 on air quality monitoring procedures and public information requires laboratories performing chemical analyses on behalf of Approved Air Quality Monitoring Associations (AASQA) to participate in interlaboratory tests.

# 2 Participants

Ten participants were involved in this ILC as listed in Table 1.

**Table 1: List of the participants to the 2025 ILC on the analysis of levoglucosan and its isomers in ambient air**

| Participants | Countries |
|---|---|
| **ANECO** | Germany |
| **IGE** | France |
| **IMROH** | Croatia |
| **INERIS** | France |
| **IEE PAS** | Poland |
| **LANUK** | Germany |
| **LCPP** | France |
| **LUBW** | Germany |
| **ULCO** | France |
| **UPEC** | France |

The instruments used by the participants, the analytical procedures applied, and the results obtained are presented anonymously in this report. Each participant was assigned a confidential code when registering online for the ILC.

# 3 Organization of the interlaboratory comparison (ILC)

The ILC was organized and implemented by the authorized personnel cited in Table 2.

Table 2: Authorized personnel who implemented this ILC

| | First and last names | ILC role |
|---|---|---|
| **Ineris<br>Parc Technologique Alata,<br>60550 Verneuil en Halatte<br>☎ +33 3 44 55 66 77** | Sylvain Bailleul | ILC coordinator |
| | Nathalie Bocquet | Test material assistant |
| | Jonathan Barrier<br>Olivier Diago | Webmaster and designer of the statistical processing tool |

The ILC was organized as follows:

July 23rd, 2025. Week 7: Call for participation. Laboratories had to submit the registration form before August 29th, 2025.

September 8th, 2025: Ineris sent a confirmation of registration with a confidential code to each participant.

September 23th, 2025: Ineris shipped the different test materials to each participant. Opening of the website for on-line result submissions.

October 24th, 2025: Deadline for result submissions on the website.

# 4 Analyses to be performed

The compounds to be analysed, following the European standard procedure CEN/TS 18044, are listed below:

- Levoglucosan (1,6-Anhydro-β-D-glucopyranose), CAS n°498-07-7
- Mannosan (1,6-Anhydro-β-D-mannopyranose), CAS n°14168-65-1
- Galactosan (1,6-Anhydro-β-D-galactopyranose), CAS n°644-76-8

The analyses had to be performed under repeatability conditions. This required ensuring that the same operator carried out all analyses for a given parameter, within a short time frame, using the same equipment and the same method. These analyses also had to be treated as independent tests: every step, from sampling the vials to reporting the final result, had to be repeated in full.

# 5 Descriptions of the test materials

All the test materials sent to the participants are presented in Table 3.

Table 3: Test materials sent to the participants

| Matrices | References | Number and type | Number of measurements | Quantity |
|---|---|---|---|---|
| **Ambient air PM10 samples (quartz fibre filter punches)** | 25-232849-F0<br>25-232849-F1<br>25-232849-F2 | One lab blank filter<br>Two field samples | Three injections of each sample extract | Three filter punches of ∅ = 37 mm |
| **Certified Reference Material (CRM, solid powder)** | 25-232849-Dust | One bottle | Two extractions with one injection per extract | About 250 mg |

## 5.1 Ambient air sample filters

Natural ambient air aerosol samples were collected using a $PM_{10}$ high volume sampler (Graseby-Andersen working at 68 $m^3$ $h^{-1}$) in December 2024 at Verneuil-en-Halatte (France, sub-urban site). 20 equivalent punches of 37 mm diameter were performed in the quartz fibre filters (20.3 x 25.4 cm) to be sent to the participants.

## 5.2 Solid Certified Reference Material (CRM)

The standard reference material (SRM) 1649b (urban dust) was used as a solid CRM in this ILC. There are no certified values for levoglucosan, mannosan or galactosan. The values indicated below were extracted from the literature (Louchouarn et al., 2009). Note that, the concentration values reported in this study were determined using only an organic solvent for extraction followed by GC/MS analysis with derivatization of the extracts. However similar concentrations values were obtained during the first ILC organized by the LCQA (Verlhac et al., 2013).

Table 4: Concentration values of anhydrosugars in NIST SRM 1649b (urban dust)

| Compounds | Mass fraction (µg $g^{-1}$) | Uncertainty (µg $g^{-1}$, k=2) |
|---|---|---|
| **Levoglucosan** | 160.5 | 10.0 |
| **Mannosan** | 16.7 | 1.4 |
| **Galactosan** | 4.8 | 0.4 |

# 6 Homogeneity and stability of the ambient filter samples

Analyses for quality control were performed by Ineris using IC-PAD, applying the reference method CEN/TS 18044, on filter punches from a dedicated $PM_{10}$ field sample.

## 6.1 Homogeneity

The analysis of samples similar to those sent to the participants was performed using the methodology described in Annex 1. Standard deviations of 5% were obtained for levoglucosan, galactosan, and mannosan. These results fully complied with ISO 13528.

## 6.2 Stability

Filter stability was monitored throughout the participants' analysis phase using filter samples comparable to those distributed to participants. Punches from a single filter were analysed weekly over 28 days. Stability was assessed according to the methodology described in Annex 1.

Results obtained from the homogeneity and stability experiments showed that **the field sample filters could be considered stable and homogeneous for the entire analytical period.**

# 7 Results

## 7.1 Exploitation of the data

### 7.1.1 General information

The details of the statistical processing are provided in **Appendix 1**.

The assigned values were defined as follows:

Table 5: Assigned values

| | Assigned values ($x_{pt}$) | Standard deviations for assessment ($\sigma_{pt}$) |
|---|---|---|
| **25-232849-F1** | x*(robust average) | s* (robust standard deviation) |
| **25-232849-F2** | x*(robust average) | s* (robust standard deviation) |
| **25-232849-Dust** | x*(robust average) | s* (robust standard deviation) |

The statistical analysis performed on the data made it possible to determine:

- the assigned value for each parameter and each test, where this value was calculated, together with its associated uncertainty.
- the performance of each participant relative to the assigned values, using the z- or z′-score indicator.
- any suspicious or anomalous values.
- the repeatability and reproducibility confidence intervals for each compound and each test material.

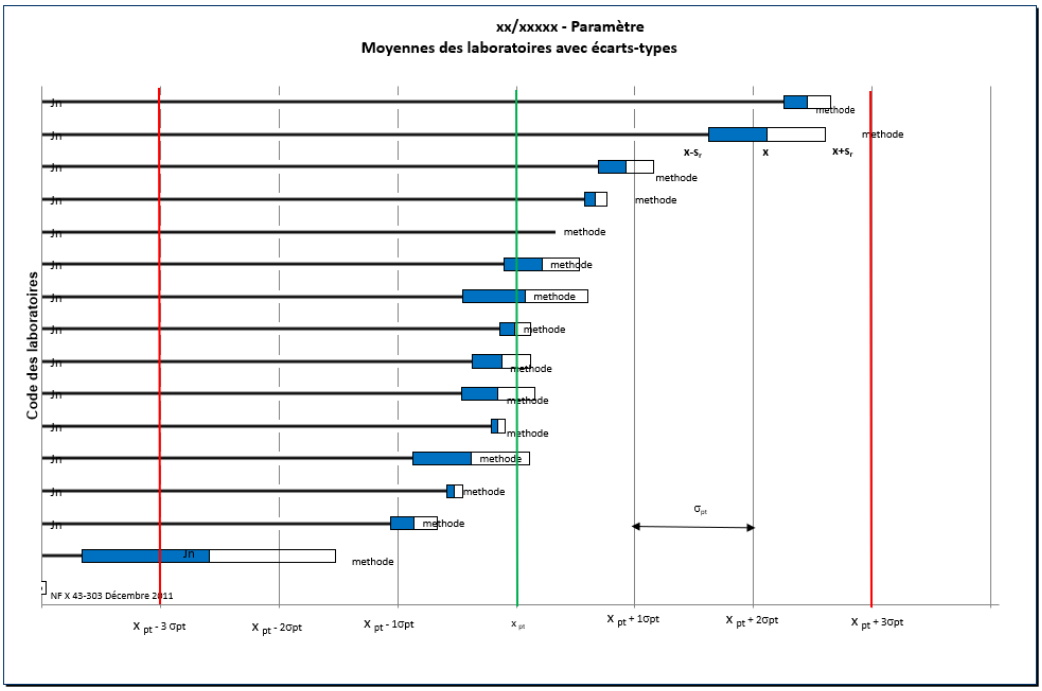
### 7.1.2 Presentation of the results

This section describes, for each test material, the results obtained after statistical processing, the means, repeatability standard deviations, biases and performance levels for each laboratory and a score (z or z') performance.

The legend below is used:

| Legend | |
|---|---|
| 1.4 | \|z\| (or \|z'\|) ≤ 2: satisfactory score |
| 2.3 | Laboratory with a 2 < \|z\| (or \|z'\|) < 3: questionable score requiring monitoring or preventive action |
| 3.56 | Laboratory with a \|z\| (or \|z'\|) ≥ 3 unsatisfactory score requiring corrective action (the analysis results are not acceptable) |
| ~~value~~ | Laboratories that did not perform the analysis or results excluded based on expert judgement |
| NA, # | Not analysed |
| NC | Not calculated |

Distribution curves are provided for each substance. They show the mean and standard deviation of each participant's results. Each curve is centred on the x-axis at the assigned value. The lower limit corresponds to the assigned value minus three times the standard deviation used for aptitude evaluation, and the upper limit corresponds to the assigned value plus three times that standard deviation. These limits may be adjusted to provide a clearer view of all laboratory results.




Each participant is shown on the y-axis using their confidential code. For each one, the assay method used is indicated, along with the extraction or analysis date of the samples. This type of visualisation allows each participant to compare the dispersion of their own results with the ones from all participants.

Other results for each test material are compiled in Appendix 2: Raw data, laboratory means and repeatability standard deviations, and values discarded based on expert judgement.

## 7.2 Results

Because of the significant variability observed between laboratories, some performance scores may fail to trigger an alert even when substantial measurement biases are present. Participants are therefore advised not to rely on this indicator alone, but to complement it by comparing their own results with the assigned value. The bias can be used for this purpose.

### 7.2.1 Test material 25-232849-F1, Filter 1

Table 6: Statistical parameters for assessment calculations

| Parameters | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|
| $x_{pt} = x^*$ (µg) | 76.233 | 3.717 | 6.524 |
| $s^*$ (µg) | 32.181 | 2.154 | 2.054 |
| Relative $s^*$ (%) | 42.2 | 57.9 | 31.5 |
| $u_{Xpt}$ (µg) | 13.409 | 0.952 | 0.908 |
| $u_{Xpt}$ added to $\sigma_{pt}$ | yes | yes | yes |
| $\sigma_{pt}$ (µg) | 34.862 | 2.355 | 2.246 |

Table 7: Other parameters considered

| Parameter | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|
| Population (n) | 9 | 8 | 8 |
| $S_L$ (µg) | 32.164 | 2.152 | 2.053 |
| $S_R$ (µg) | 32.213 | 2.157 | 2.058 |
| Relative $S_R$ (%) | 42.3 | 58.0 | 31.5 |
| $S_r$ (µg) | 1.779 | 0.146 | 0.146 |
| Relative $S_r$ (%) | 2.3 | 3.9 | 2.2 |
| Relative $IC_R$ (%) | 97.4 | 137.2 | 74.6 |
| Relative $IC_r$ (%) | 5.4 | 9.3 | 5.3 |

Table 8: Means, standard deviations, biases and scores for laboratories obtained for levoglucosan for Filter 1

| ID lab | Levoglucosan | | | | |
|---|---|---|---|---|---|
| | x (µg) | $s_r$ (µg) | CV | z' | Bias |
| **250700** | 38.017 | 0.384 | 1.01% | -1.10 | -50.13% |
| **250718** | 34.033 | 1.941 | 5.70% | -1.21 | -55.36% |
| **250740** | 89.403 | 1.230 | 1.38% | 0.38 | 17.28% |
| **250745** | 98.037 | 1.452 | 1.48% | 0.63 | 28.60% |
| **250751** | 76.713 | 0.076 | 0.10% | 0.01 | 0.63% |
| **250765** | 81.030 | 0.052 | 0.06% | 0.14 | 6.29% |
| **250766** | 81.293 | 2.593 | 3.19% | 0.15 | 6.64% |
| **250775** | 63.063 | 0.920 | 1.46% | -0.38 | -17.28% |
| **250778** | ~~2 674.723~~ | ~~15.435~~ | ~~0.58%~~ | 74.54 | 3408.63% |
| **250783** | 182.543 | 5.730 | 3.14% | 3.05 | 139.46% |

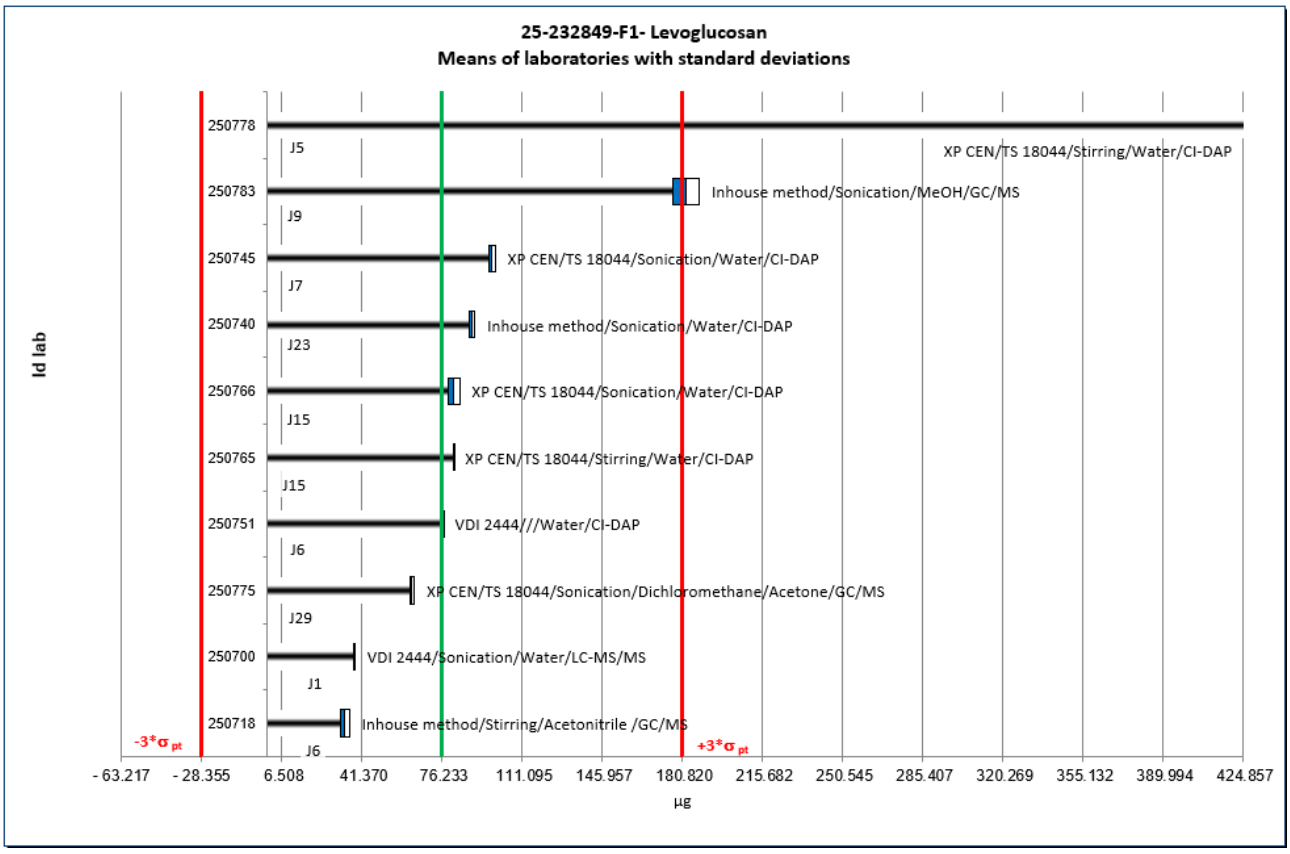



Figure 1 : Participants' dispersion obtained for levoglucosan for Filter 1 (25-232849-F1)

Performance tests do not provide reliable information on the actual measurement deviation because of the substantial interlaboratory dispersion. Participants are therefore advised not to rely solely on this indicator, but also to compare their own measurement with the assigned value.

Laboratory 250778 reported a result that was 35 times higher than the assigned value. Its results were excluded from the statistical analysis.

Table 9 : Means, standard deviations, biases and z' scores obtained for galactosan for Filter 1

| ID lab | Galactosan | | | | |
|---|---|---|---|---|---|
| | x (µg) | $s_r$ (µg) | CV | z' | Bias |
| **250700** | 1.433 | 0.029 | 2.01% | -0.97 | -61.44% |
| **250718** | 6.180 | 0.488 | 7.89% | 1.05 | 66.25% |
| **250740** | 3.140 | 0.089 | 2.83% | -0.25 | -15.53% |
| **250745** | 3.470 | 0.090 | 2.59% | -0.10 | -6.65% |
| **250751** | NA | NA | NA | NA | NA |
| **250765** | 2.080 | 0.072 | 3.47% | -0.70 | -44.04% |
| **250766** | 3.310 | 0.052 | 1.57% | -0.17 | -10.96% |
| **250775** | 3.177 | 0.042 | 1.31% | -0.23 | -14.54% |
| **250778** | ~~99.457~~ | ~~0.847~~ | ~~0.85%~~ | 40.66 | 2575.56% |
| **250783** | 15.060 | 0.286 | 1.90% | 4.82 | 305.14% |

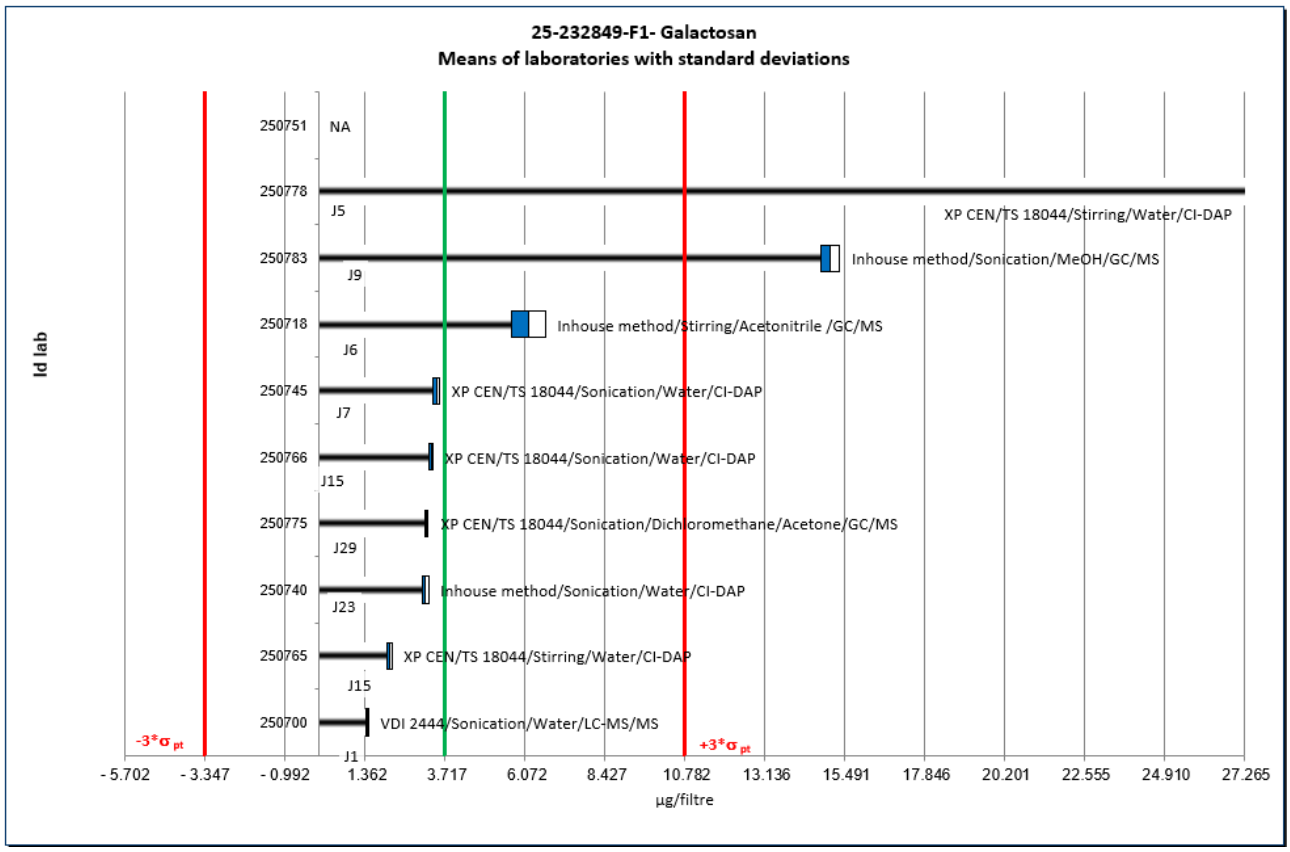



Figure 2: Participants' dispersion obtained for galactosan for Filter 1 (25-232849-F1)

Performance tests do not provide reliable information on the actual measurement deviation because of the substantial interlaboratory dispersion. Participants are therefore advised not to rely solely on this indicator, but also to compare their own measurement with the assigned value.

Laboratory 250778 reported a result that was 27 times higher than the assigned value. Its results were excluded from the statistical analysis.

Table 10: Means, standard deviations, biases and scores for laboratories obtained for mannosan for Filter 1

| ID lab | Mannosan | | | | |
|---|---|---|---|---|---|
| | x (µg) | $s_r$ (µg) | CV | z' | Bias |
| **250700** | 4.957 | 0.086 | 1.74% | -0.70 | -24.02% |
| **250718** | 5.830 | 0.173 | 2.98% | -0.31 | -10.63% |
| **250740** | 5.870 | 0.147 | 2.51% | -0.29 | -10.02% |
| **250745** | 7.500 | 0.106 | 1.41% | 0.43 | 14.97% |
| **250751** | NA | NA | NA | NA | NA |
| **250765** | 3.887 | 0.006 | 0.15% | -1.17 | -40.42% |
| **250766** | 8.010 | 0.087 | 1.09% | 0.66 | 22.79% |
| **250775** | 6.530 | 0.080 | 1.23% | 0.00 | 0.10% |
| **250778** | ~~230.397~~ | ~~0.879~~ | ~~0.38%~~ | 99.67 | 3431.77% |
| **250783** | 42.120 | 0.537 | 1.28% | 15.85 | 545.66% |

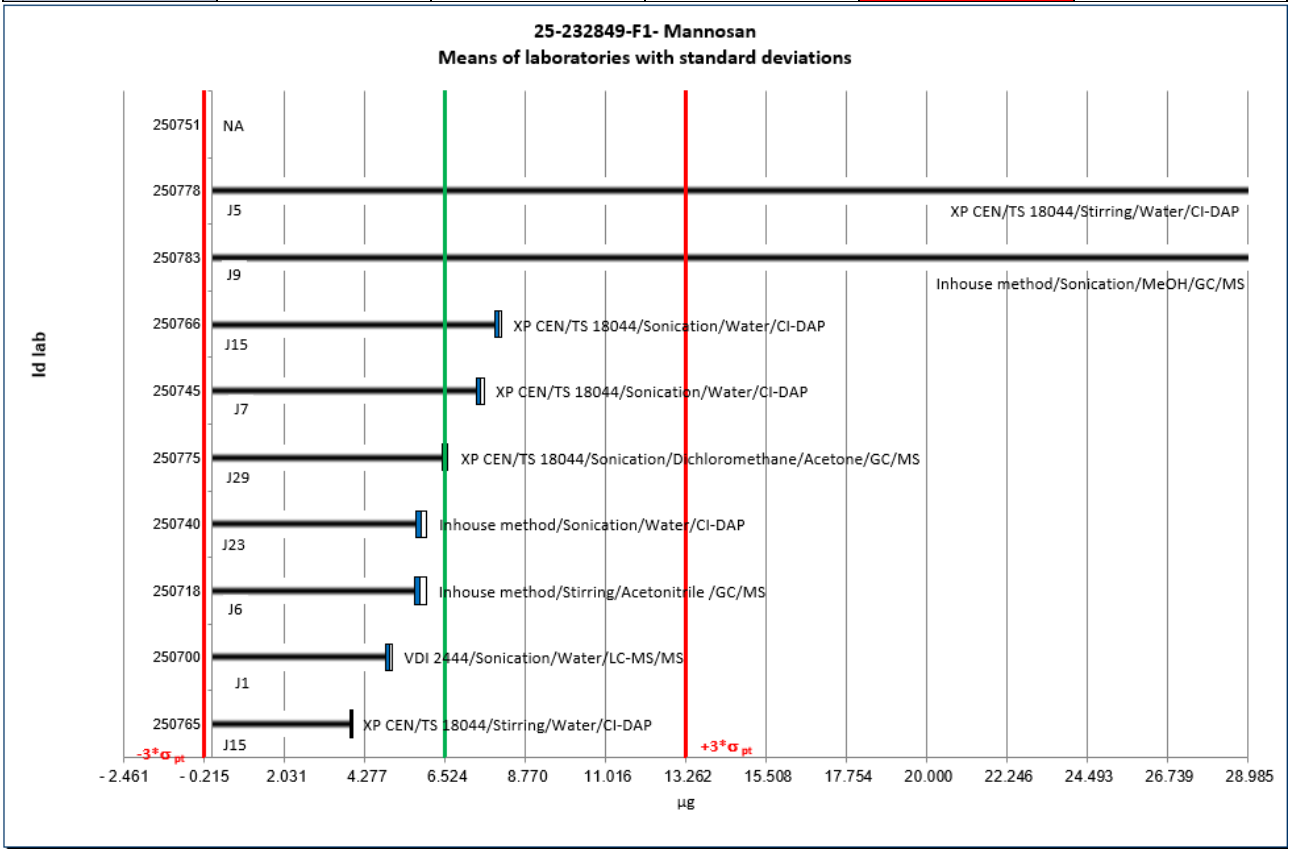



Figure 3: Participants' dispersion for mannosan for Filter 1 (25-232849-F1)

Laboratory 250778 reported a result that was 35 times higher than the assigned value. Its results were excluded from the statistical analysis.

### 7.2.2 Test material 25-232849-F2

Table 11: Statistical parameters for assessment calculations

| Parameters | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|
| $x_{pt} = x^*$ (µg) | 67.848 | 2.962 | 5.663 |
| $s^*$ (µg) | 13.485 | 1.032 | 2.105 |
| Relative $s^*$ (%) | 19.9 | 34.9 | 37.2 |
| $u_{xpt}$ (µg) | 5.619 | 0.456 | 0.930 |
| Added to $\sigma_{pt}$ | yes | yes | yes |
| $\sigma_{pt}$ (µg) | 14.608 | 1.129 | 2.301 |

Table 12: Other parameters considered

| Parameter | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|
| Population (n) | 9 | 8 | 8 |
| $S_L$ (µg) | 13.474 | 1.031 | 2.104 |
| $S_R$ (µg) | 13.505 | 1.034 | 2.107 |
| Relative $S_R$ (%) | 19.9 | 34.9 | 37.2 |
| $S_r$ (µg) | 0.919 | 0.080 | 0.121 |
| Relative $S_r$ (%) | 1.4 | 2.7 | 2.1 |
| Relative $IC_R$ (%) | 45.9 | 82.6 | 88.00 |
| Relative $IC_r$ (%) | 3.1 | 6.4 | 5.1 |

Table 13: Means, standard deviations, biases and scores for laboratories obtained for levoglucosan for Filter 2

| ID lab | Levoglucosan | | | | |
|---|---|---|---|---|---|
| | x (µg) | $s_r$ µg | CV | z' | Bias |
| **250700** | 53.437 | 0.166 | 0.31% | -0.99 | -21.24% |
| **250718** | 21.473 | 1.666 | 7.76% | -3.17 | -68.35% |
| **250740** | 85.413 | 0.918 | 1.07% | 1.20 | 25.89% |
| **250745** | 80.173 | 0.597 | 0.75% | 0.84 | 18.17% |
| **250751** | 68.403 | 0.132 | 0.19% | 0.04 | 0.82% |
| **250765** | 70.357 | 0.070 | 0.10% | 0.17 | 3.70% |
| **250766** | 62.930 | 0.423 | 0.67% | -0.34 | -7.25% |
| **250775** | 70.323 | 1.008 | 1.43% | 0.17 | 3.65% |
| **250778** | ~~2 278.197~~ | ~~42.335~~ | ~~1.86%~~ | 151.31 | 3257.80% |
| **250783** | 71.973 | 1.258 | 1.75% | 0.28 | 6.08% |

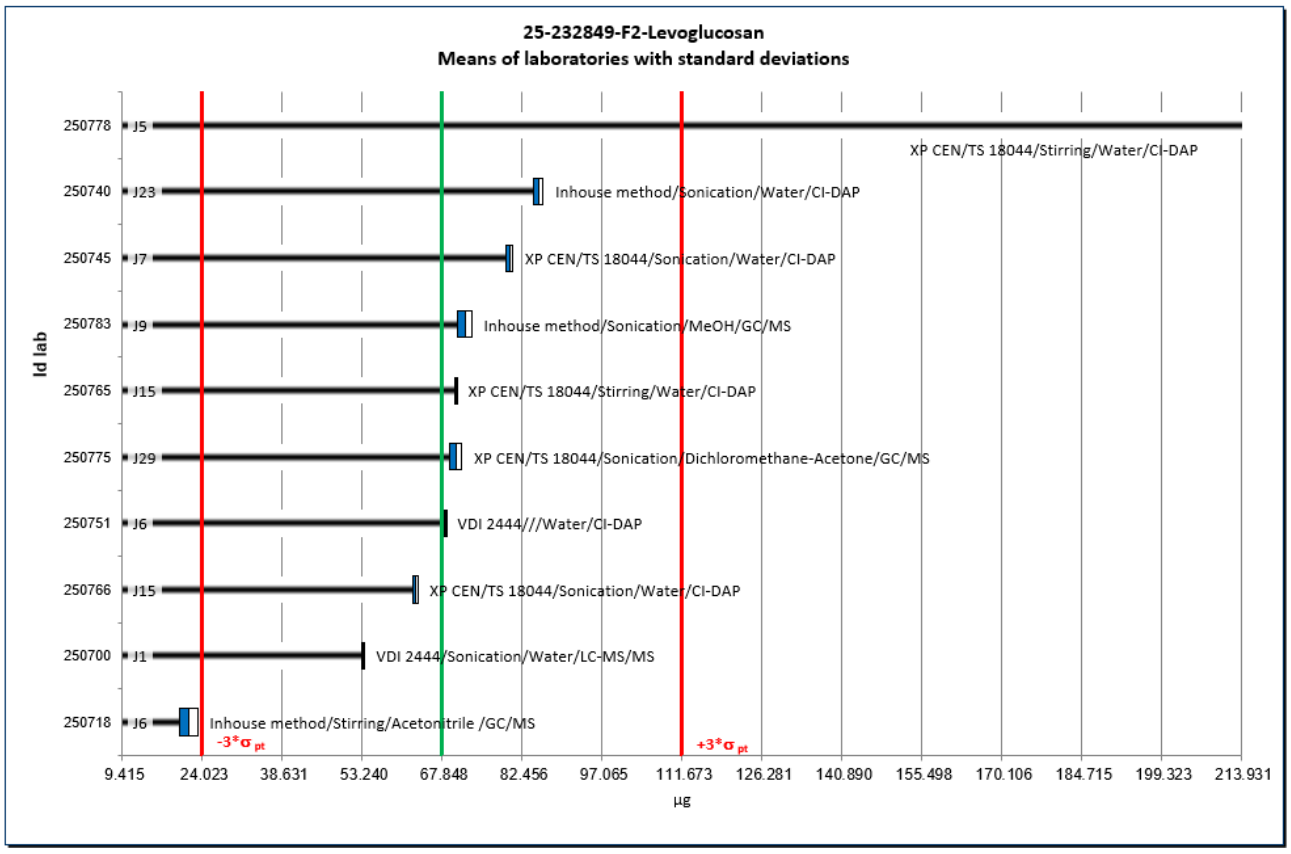



Figure 4: Participants' dispersion obtained for levoglucosan for Filter 2 (25-232849-F2)

Laboratory 250778 reported a result that was 34 times higher than the assigned value. Its results were excluded from the statistical analysis.

Table 14: Means, standard deviations, biases and scores for laboratories obtained for galactosan for Filter 2

| ID lab | Galactosan | | | | |
|---|---|---|---|---|---|
| | x (µg) | $s_r$ | CV | z' | Bias |
| **250700** | 1.797 | 0.046 | 2.57% | -1.03 | -39.35% |
| **250718** | 3.837 | 0.127 | 3.31% | 0.77 | 29.52% |
| **250740** | 3.097 | 0.090 | 2.89% | 0.12 | 4.54% |
| **250745** | 2.913 | 0.064 | 2.18% | -0.04 | -1.65% |
| **250751** | NA | NA | NA | NA | NA |
| **250765** | 1.927 | 0.045 | 2.34% | -0.92 | -34.96% |
| **250766** | 2.543 | 0.025 | 0.99% | -0.37 | -14.14% |
| **250775** | 3.073 | 0.055 | 1.79% | 0.10 | 3.75% |
| **250778** | ~~78.423~~ | ~~2.709~~ | ~~3.45%~~ | 66.86 | 2547.50% |
| **250783** | 4.993 | 0.081 | 1.62% | 1.80 | 68.57% |

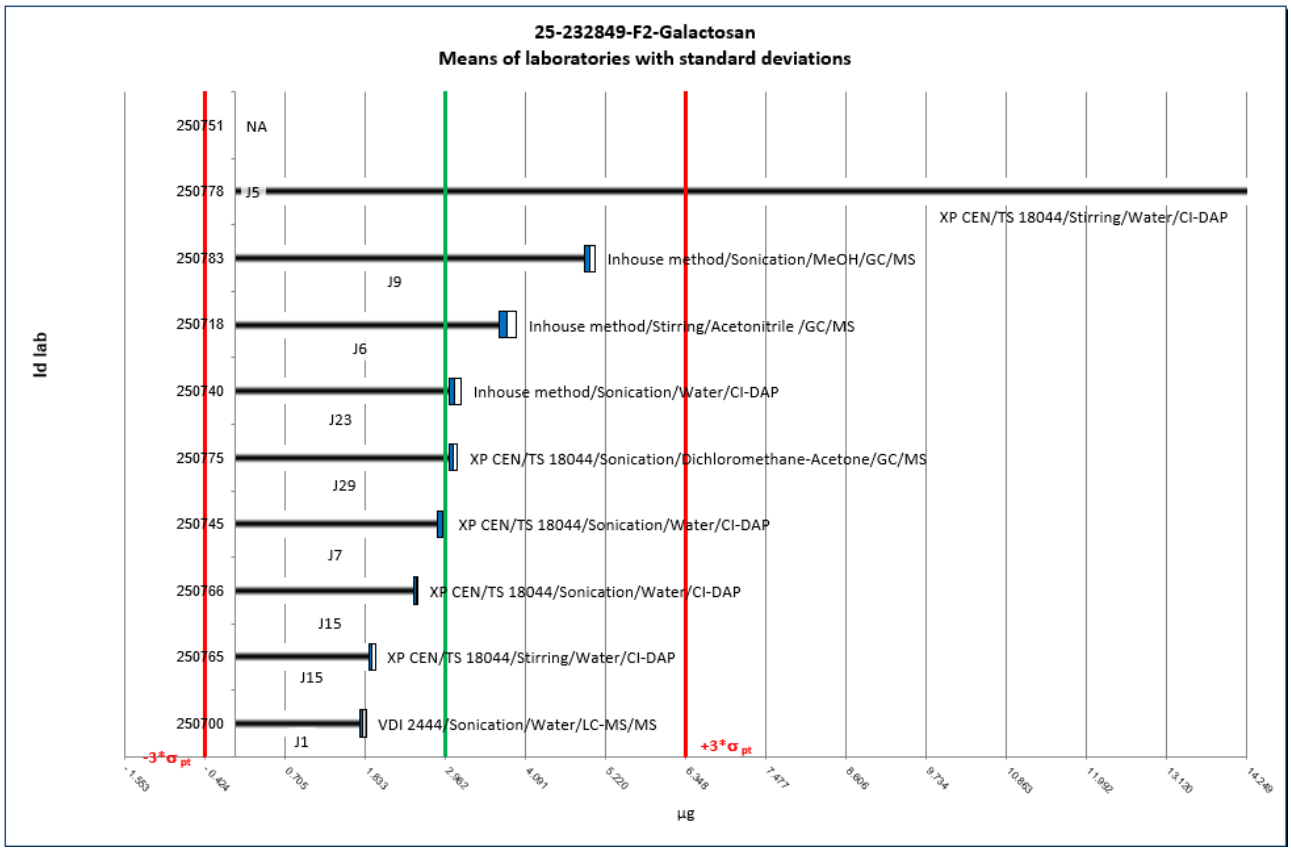



Figure 5: Participants' dispersion for galactosan for Filter 2 (25-232849-F2)

Laboratory 250778 reported a result that was 26 times higher than the assigned value. Its results were excluded from the statistical analysis.

Table 15: Means, standard deviations, biases and scores for laboratories obtained for mannosan for Filter 2

| ID lab | Mannosan | | | | |
|---|---|---|---|---|---|
| | x (µg) | $s_r$ | CV | z' | Bias |
| **250700** | 4.587 | 0.093 | 2.03% | -0.47 | -19.01% |
| **250718** | 3.660 | 0.370 | 10.12% | -0.87 | -35.37% |
| **250740** | 5.953 | 0.021 | 0.35% | 0.13 | 5.13% |
| **250745** | 6.553 | 0.015 | 0.23% | 0.39 | 15.72% |
| **250751** | NA | NA | NA | NA | NA |
| **250765** | 2.997 | 0.015 | 0.51% | -1.16 | -47.08% |
| **250766** | 6.357 | 0.055 | 0.87% | 0.30 | 12.25% |
| **250775** | 6.377 | 0.076 | 1.19% | 0.31 | 12.60% |
| **250778** | ~~196.977~~ | ~~2.644~~ | ~~1.34%~~ | 83.14 | 3378.37% |
| **250783** | 13.383 | 0.335 | 2.51% | 3.36 | 136.33% |

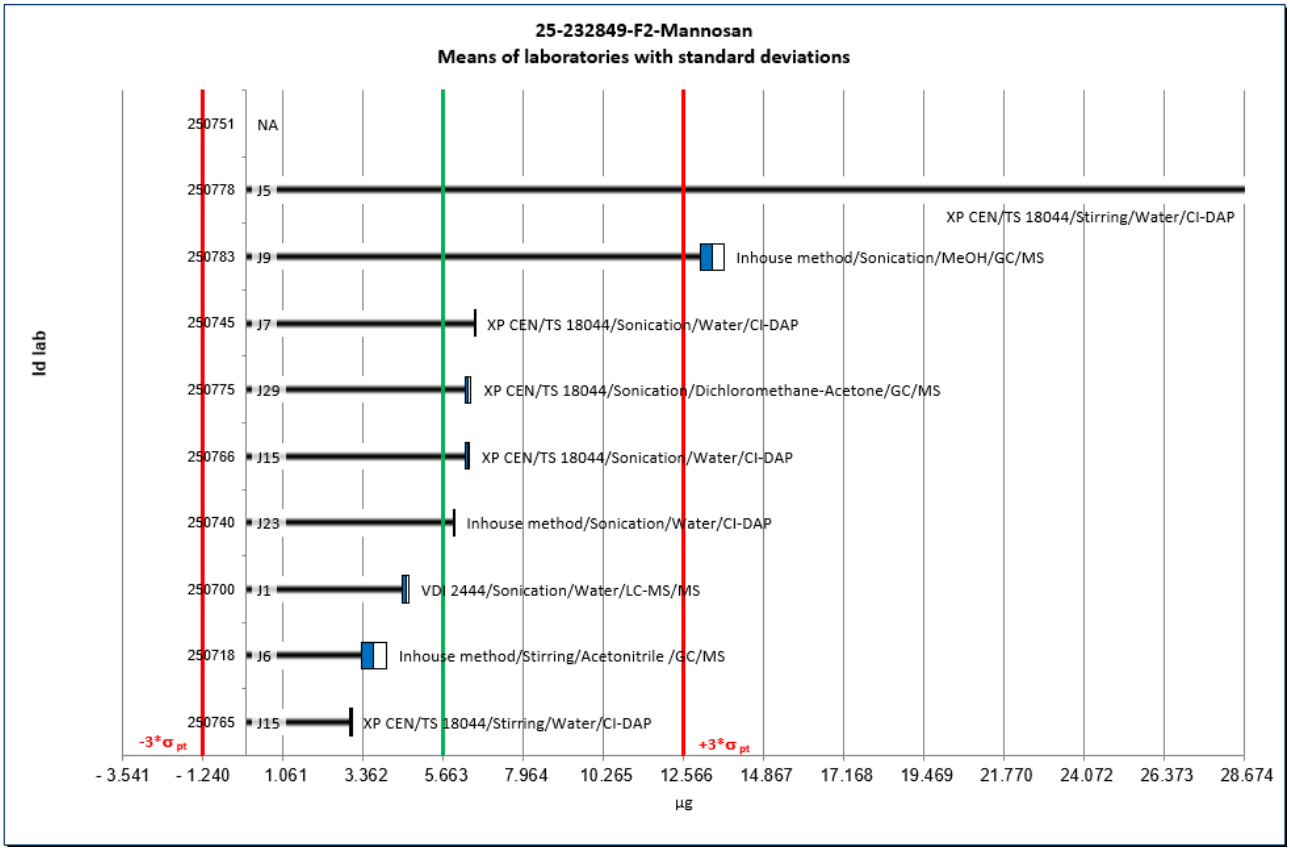



Figure 6: Participants' dispersion for mannosan for Filter 2 (25-232849-F2)

Laboratory 250778 reported a result that was 35 times higher than the assigned value. Its results were excluded from the statistical analysis.

### 7.2.3 Test material 25-232849-Dust

Table 16: Statistical parameters for assessment calculations

| Parameters | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|
| $x_{pt}$ = x* (mg $kg^{-1}$ dry mass) | 131.624 | *(3.148)* | *(16.163)* |
| s* (mg $kg^{-1}$ dry mass) | 122.201 | *(3.839)* | *(17.637)* |
| Relative s* (%) | 92.8 | *(121.9)* | *(109.1)* |
| $u_{xpt}$ (mg $kg^{-1}$ dry mass) | 54.006 | *(2.146)* | *(9.000)* |
| Added to $\sigma_{pt}$ | yes | *yes* | *yes* |
| $\sigma_{pt}$ (mg $kg^{-1}$ dry mass) | 133.603 | *(4.399)* | *(19.801)* |

*(value): Indicative value due to number of participants (n < 8)*

Table 17: Other parameters considered

| Parameters | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|
| Indicative value (mg $kg^{-1}$ dry mass) (Louchouarn et al., 2009) | 160.5 | 4.8 | 16.7 |
| Population (n) | 8 | 5 | 6 |
| $S_L$ (mg $kg^{-1}$ dry mass) | 122.172 | *(3.839)* | *(17.606)* |
| $S_R$ (mg $kg^{-1}$ dry mass) | 122.230 | *(3.840)* | *(17.668)* |
| Relative $S_R$ (%) | 92.9 | *(122.0)* | *(109.3)* |
| $S_r$ (mg $kg^{-1}$ dry mass) | 3.748 | *(0.101)* | *(1.488)* |
| Relative $S_r$ (%) | 2.8 | *(3.2)* | *(9.2)* |
| Relative $IC_R$ (%) | 219.6 | *(338.6)* | *(281.0)* |
| Relative $IC_r$ (%) | 6.7 | *(8.9)* | *(23.7)* |

*(value): Indicative value due to number of participants (n < 8)*

Table 18: Means, standard deviations, biases and scores for laboratories obtained for levoglucosan for the CRM

| ID lab | Levoglucosan | | | | |
|---|---|---|---|---|---|
| | x (mg/kg dry mass) | $s_r$ (mg/kg dry mass) | CV | z' | Bias |
| **250700** | 203.710 | 4.936 | 2.42% | 0.54 | 54.77% |
| **250718** | 18.245 | 0.304 | 1.67% | -0.85 | -86.14% |
| **250740** | 239.280 | 4.101 | 1.71% | 0.81 | 81.79% |
| **250745** | 272.855 | 12.396 | 4.54% | 1.06 | 107.30% |
| **250751** | 152.145 | 0.134 | 0.09% | 0.15 | 15.59% |
| **250765** | NA | NA | NA | NA | NA |
| **250766** | 151.885 | 3.557 | 2.34% | 0.15 | 15.39% |
| **250775** | 10.795 | 1.167 | 10.81% | -0.90 | -91.80% |
| **250778** | 4.075 | 0.247 | 6.07% | -0.95 | -96.90% |
| **250783** | NA | NA | NA | NA | NA |

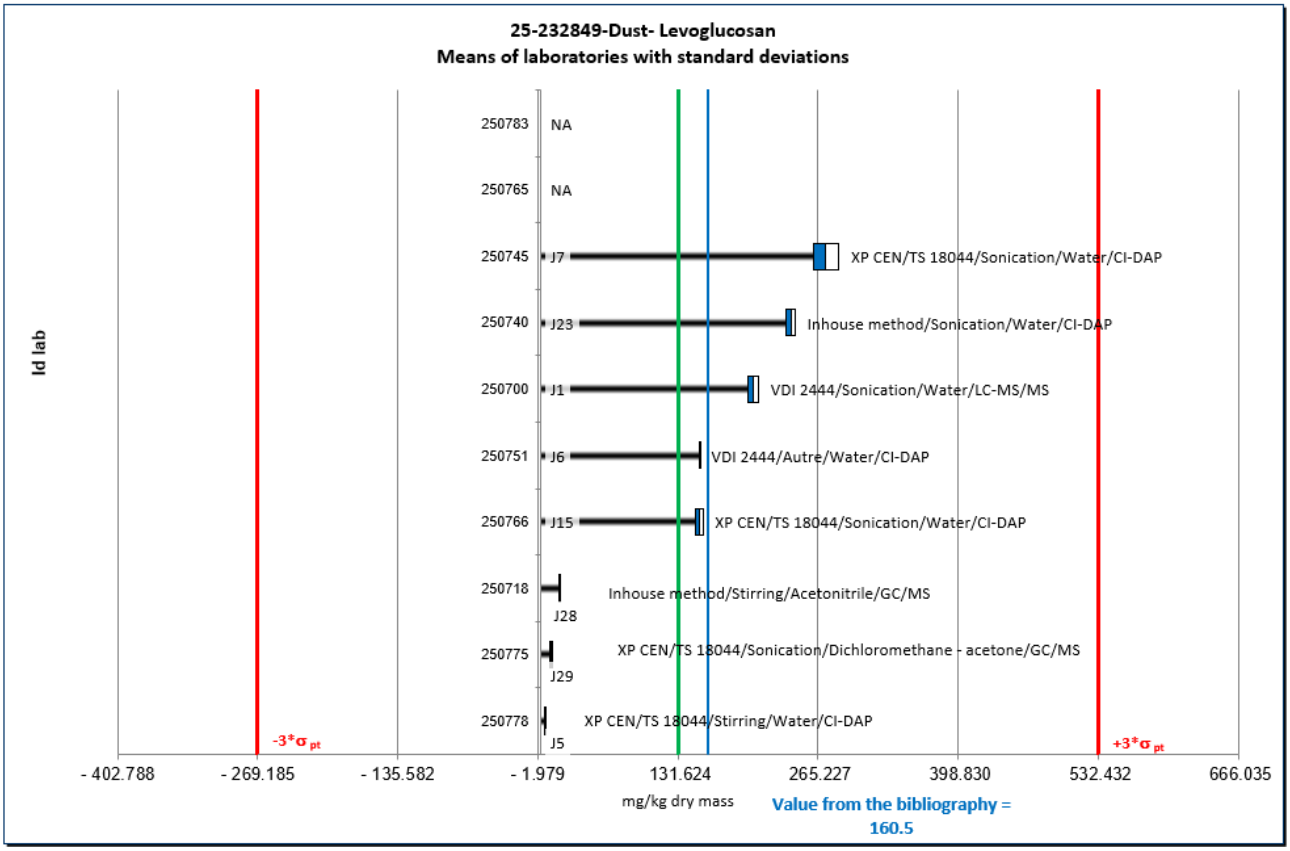



Figure 7: Participants' dispersion for levoglucosan for the CRM (25-232849-Dust)

The performance tests did not provide reliable information on the actual measurement deviation because of the substantial interlaboratory dispersion. Participants are therefore advised not to rely solely on this indicator, but also to compare their own measurement with the assigned value. With an overall dispersion of around 93%, the performance score did not give laboratories a sufficiently clear warning about the quality of their results.

Table 19: Means, standard deviations, biases and scores for laboratories obtained for galactosan for the CRM

| ID lab | Galactosan | | | | |
|---|---|---|---|---|---|
| | x (mg/kg dry mass) | $s_r$ (mg/kg dry mass) | CV | z' | Bias |
| **250700** | 2.390 | 0.085 | 3.55% | NC | -24.09% |
| **250718** | <6.800 | 0.000 | 0.00% | NC | (115.98%) |
| **250740** | <1.455 | 0.007 | 0.49% | NC | -(53.79%) |
| **250745** | 3.065 | 0.064 | 2.08% | NC | -2.65% |
| **250751** | NA | NA | NA | NA | NA |
| **250765** | NA | NA | NA | NA | NA |
| **250766** | 9.280 | 0.438 | 4.72% | NC | 194.74% |
| **250775** | 0.600 | 0.042 | 7.07% | NC | -80.94% |
| **250778** | 0.780 | 0.042 | 5.44% | NC | -75.23% |
| **250783** | NA | NA | NA | NA | NA |

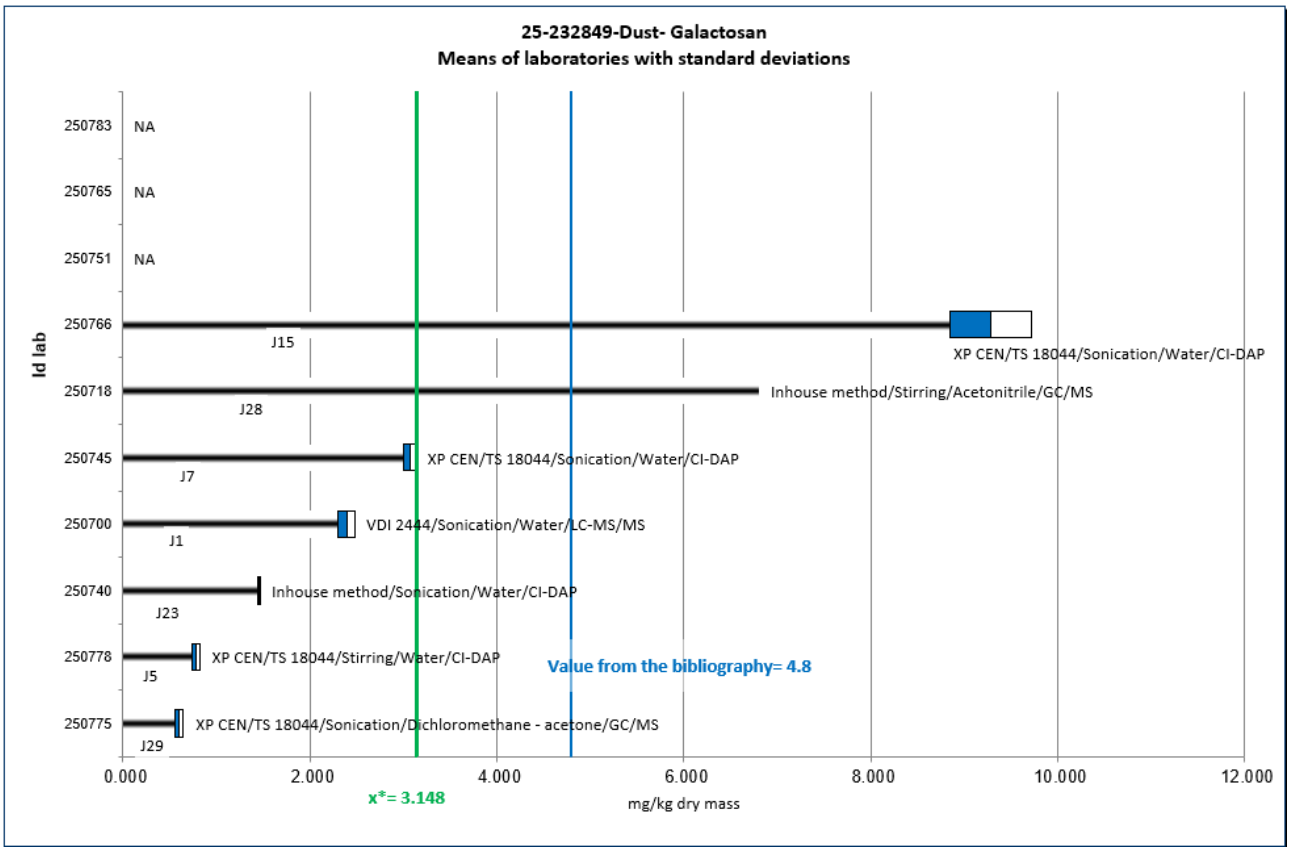



Figure 8: Participants' dispersion for galactosan for the CRM (25-232849-Dust)

As only a small number of laboratories reported quantified results, it was not possible to assess performance.

Table 20: Means, standard deviations, biases and scores for laboratories obtained for mannosan for the CRM

| ID lab | Mannosan | | | | |
|---|---|---|---|---|---|
| | x (mg/kg dry mass) | $s_r$ (mg/kg dry mass) | CV | z' | Bias |
| **250700** | 27.485 | 0.134 | 0.49% | NC | 70.05% |
| **250718** | 11.110 | 0.255 | 2.29% | NC | -31.26% |
| **250740** | 15.665 | 2.227 | 14.22% | NC | -3.08% |
| **250745** | NA | NA | NA | NA | NA |
| **250751** | NA | NA | NA | NA | NA |
| **250765** | NA | NA | NA | NA | NA |
| **250766** | 40.615 | 2.850 | 7.02% | NC | 151.29% |
| **250775** | 1.680 | 0.042 | 2.53% | NC | -89.61% |
| **250778** | 0.420 | 0.028 | 6.73% | NC | -97.40% |
| **250783** | NA | NA | NA | NA | NA |

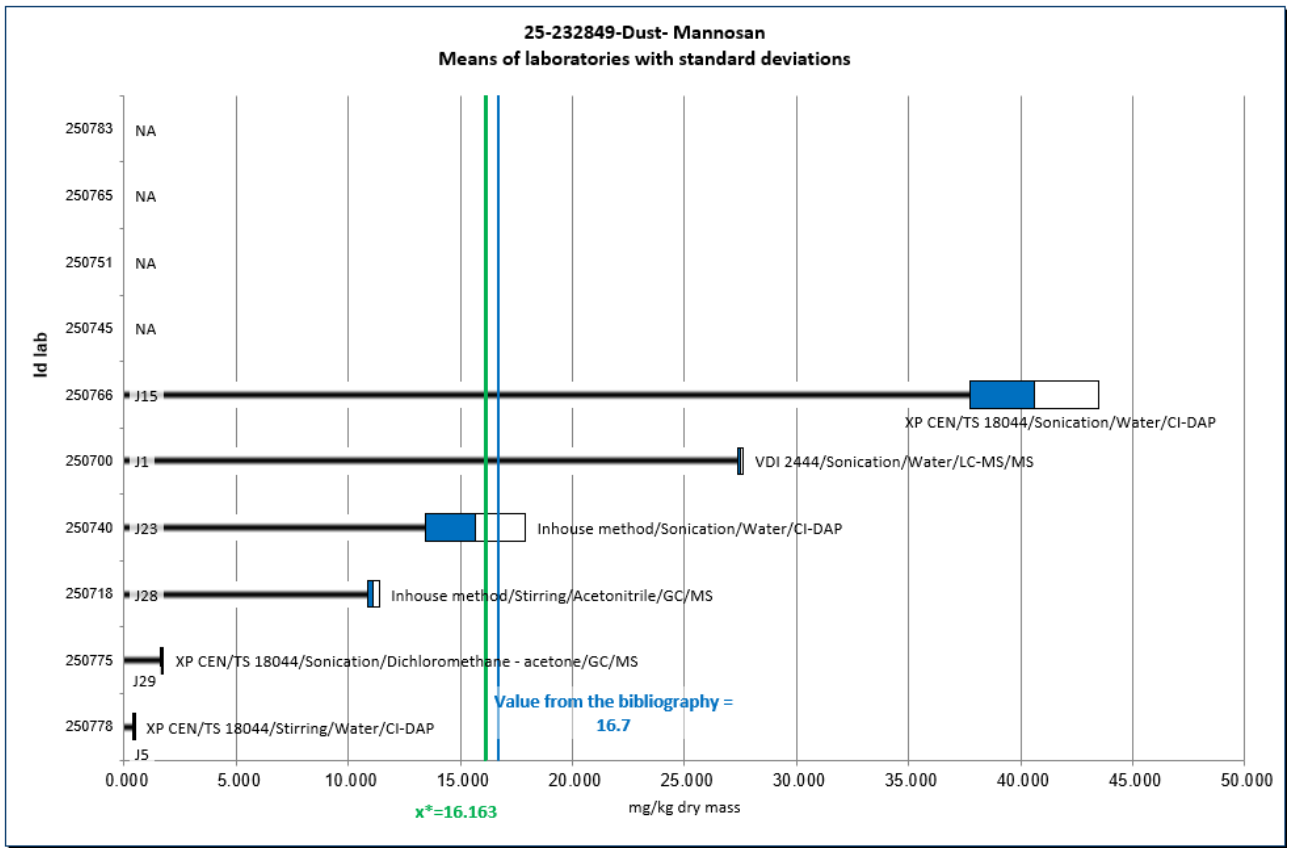



Figure 9: Participants' dispersion for mannosan for the CRM (25-232849-Dust)

As only a small number of laboratories reported quantified results, it was not possible to assess performance.

### 7.2.4 Test material 25-232849-F0 (Blank)

Table 21 summarises the limits of quantification reported by the participants for the test material 25-232849 – F0 (Blank).

Table 21: Observed values for the test material 25-232849-F0 (blank)

| ID lab | Analytical technique | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|---|
| | | x (µg) | x (µg) | x (µg) |
| **250700** | LC-MS/MS | 0.20 | 0.20 | 0.20 |
| **250718** | GC/MS | 0.15 | 0.15 | 0.15 |
| **250740** | CI-PAD | 0.05 | 0.07 | 0.09 |
| **250745** | CI-PAD | NA | NA | NA |
| **250751** | CI-PAD | 4.00 | NA | NA |
| **250765** | CI-PAD | 0.00 | 0.00 | 0.00 |
| **250766** | CI-PAD | 0.20 | 0.20 | 0.20 |
| **250775** | GC/MS | 0.22 | 0.01 | 0.05 |
| **250778** | CI-PAD | ***3.53*** | ***1.41*** | ***1.41*** |
| **250783** | GC/MS | 0.10 | 0.10 | 0.10 |

***In italic and bold***: value quantified; in red: value submitted equal to zero.

There was substantial variability in the reported limits of quantification, both overall and for individual compounds. For a given compound, the lowest and highest values differed by a factor of 4 to 80 (excluding values equal to 0, which have no physical meaning, as well as the quantified value).

### 7.2.5 Summary of the biases

Table 22 summarizes the measurement biases, estimated for each laboratory and test material, to get an overview of the laboratory performances by comparison to the assigned values.

Table 22: Measurement biases observed for all the test matrices by comparison to the assigned values

| ID lab | Levoglucosan | Galactosan | Mannosan | Levoglucosan | Galactosan | Mannosan | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|---|---|---|---|---|---|
| | F1 | F1 | F1 | F2 | F2 | F2 | CRM | CRM | CRM |
| **250700** | -50.13% | -61.44% | -24.02% | -21.24% | -39.35% | -19.01% | 54.77% | -24.09% | 70.05% |
| **250718** | -55.36% | 66.25% | -10.63% | -68.35% | 29.52% | -35.37% | -86.14% | *(115.98%)* | -31.26% |
| **250740** | 17.28% | -15.53% | -10.02% | 25.89% | 4.54% | 5.13% | 81.79% | *-(53.79%)* | -3.08% |
| **250745** | 28.60% | -6.65% | 14.97% | 18.17% | -1.65% | 15.72% | 107.30% | -2.65% | NA |
| **250751** | 0.63% | NA | NA | 0.82% | NA | NA | 15.59% | NA | NA |
| **250765** | 6.29% | -44.04% | -40.42% | 3.70% | -34.96% | -47.08% | NA | NA | NA |
| **250766** | 6.64% | -10.96% | 22.79% | -7.25% | -14.14% | 12.25% | 15.39% | 194.74% | 151.29% |
| **250775** | -17.28% | -14.54% | 0.10% | 3.65% | 3.75% | 12.60% | -91.80% | -80.94% | -89.61% |
| **250778** | 3408.63% | 2575.56% | 3431.77% | 3257.80% | 2547.50% | 3378.37% | -96.90% | -75.23% | -97.40% |
| **250783** | 139.46% | 305.14% | 545.66% | 6.08% | 68.57% | 136.33% | NA | NA | NA |

*In italic:* Biases calculated with the provided limit of quantification.

The table below summarizes the measurement biases, estimated for the test material 25-232849-Dust for each laboratory, to get an overview of the laboratory performances by comparison to the reference values from the bibliography (Louchouarn et al., 2009; Verlhac et al., 2013).

**Table 23: biases observed for 25-232849-Dust by comparison to the reference values for the CRM**

| ID lab | Levoglucosan | Galactosan | Mannosan |
|---|---|---|---|
| | CRM | CRM | CRM |
| **250700** | 26.92% | -50.21% | 64.58% |
| **250718** | -88.63% | *(41.67%)* | -33.47% |
| **250740** | 49.08% | *-(69.69%)* | -6.20% |
| **250745** | 70.00% | -36.15% | NA |
| **250751** | -5.21% | NA | NA |
| **250765** | NA | NA | NA |
| **250766** | -5.37% | 93.33% | 143.20% |
| **250775** | -93.27% | -87.50% | -89.94% |
| **250778** | -97.46% | -83.75% | -97.49% |
| **250783** | NA | NA | NA |

*(In italic):* Biases calculated with the provided limit of quantification.

### 7.2.6 Summary of the extraction and analysis methods used

Table 24 lists the different extraction and analytical methods implemented by the participants during this ILC.

Table 24: Extraction and analytic methods used by the particpants

| ID lab | Extraction technique | Solvent extraction | Analysis technique | Reference method |
|---|---|---|---|---|
| **250700** | Sonication | Water | LC-MS/MS | VDI 2444 |
| **250718** | Stirring | Acetonitrile | GC/MS | In house method |
| **250740** | Sonication | Water | IC-PAD | In house method |
| **250745** | Sonication | Water | IC-PAD | CEN/TS 18044 |
| **250751** | Not specified | Water | IC-PAD | VDI 2444 |
| **250765** | Stirring | Water | IC-PAD | CEN/TS 18044 |
| **250766** | Sonication | Water | IC-PAD | CEN/TS 18044 |
| **250775** | Sonication | Dichloromethane/Acetone | GC/MS | CEN/TS 18044 |
| **250778** | Stirring | Water | CI-PAD | CEN/TS 18044 |
| **250783** | Sonication | MeOH | GC/MS | In house method |

The most used analytical approach was ultrasonic water extraction combined with IC-PAD analysis. The results obtained in this ILC did not allow any conclusions to be drawn regarding a potential method-related effect.

### 7.2.7 Assessment of measurement uncertainties obtained

The expanded uncertainty of measurement U has been assessed using the reproducibility standard deviation $S_R$ applying the following equation:

$$U = 2 \times S_R$$

A comparison of the uncertainties and reproducibility standard deviation determined through this ILC with the ones indicated in the reference method CEN/TS 18044 (2024) and those obtained from previous ILCs for the analysis of ambient air filters (Chiara Pietrogrande et al., 2017; Jaffrezo et al., 2019; Verlhac et al., 2013; Yttri et al., 2015) is presented in Table 25.

Table 25: Comparison of uncertainties (%) and reproducibility (%) observed in previous ILCs for the analysis of ambient air filters

| Compounds | Parameters | Verlhac and al (2013) | Yttri and al. (2015) | Pietrogrande and al. (2017) | Jaffrezo and al. (2019) | CEN/TS 18044 | Present study | |
|---|---|---|---|---|---|---|---|---|
| | | Mean 2 filters | Mean 6 filters | Mean 26 filters | Mean 3 filters | Mean 3 filters | Filter 1 | Filter 2 |
| Levoglucosan | $S_R$ (%) | 21 | 22 | 41 | 16 | 23 | 42 | 20 |
| | U (%) | 42 | 44 | 82 | 32 | 45 | 85 | 40 |
| Mannosan | $S_R$ (%) | 21 | 40 | 38 | 21 | 38 | 32 | 37 |
| | U (%) | 41 | 80 | 76 | 42 | 76 | 63 | 74 |
| Galactosan | $S_R$ (%) | 43 | 53 | 38 | 33 | 33 | 58 | 35 |
| | U (%) | 86 | 106 | 76 | 66 | 66 | 116 | 70 |

Only the uncertainties obtained for filter F2 are comparable to those indicated in the reference method CEN/TS 18044. The uncertainties reported for filter F1 were significantly higher. Although filters F1 and F2 show similar concentrations, the large difference between their calculated uncertainties was unexpected.

When comparing these results with those from previous ILCs, a wide variability in the reported uncertainties is observed. For levoglucosan, for example, uncertainties range from 32% to 85%, indicating that further analytical improvements are still needed to enhance the quality of such measurements.

The uncertainties obtained for the CRM (NIST SRM 1649b) were significantly higher for all the anhydrosugars studied (Table 26). Additional sources of uncertainty, such as the weighing of the powder prior to analysis, may explain these differences. The results obtained in this ILC were also higher than those reported in a previous ILC (Verlhac et al., 2013). A marked deterioration in uncertainty was observed. For levoglucosan, the uncertainty increased from 52% in 2013 to 186% in 2025. For galactosan, it rose from 178% to 244%, and for mannosan, from 79% to 219%.

Table 26: Comparison of uncertainties (%) observed for the CRM with a previous ILC

| Compounds | NIST SRM 1649b (CRM) | | | |
|---|---|---|---|---|
| | 2025 | | 2013 | |
| Parameters | $x_{pt}$ (µg/g) | U (%) k=2 | $x_{pt}$ (µg/g) | U (%) k=2 |
| Levoglucosan | 131.624 | 186% | 176.975 | 52% |
| Mannosan | 16.163 | 219% | 18.665 | 79% |
| Galactosan | 3.148 | 244% | 11.595 | 178% |

# 8 Conclusions

The European ILC focusing on the analysis of levoglucosan and its isomers in ambiant air organized in 2025 by Ineris as part of its activities for the LCSQA showed the key following results for specific participants:

- Laboratory 250751 reported results only for levoglucosan.
- A substantial positive bias was observed for laboratory 250778 across all filters and all anhydrosugars analysed. The results suggest the presence of a systematic error of approximately a factor of 35 leading to significant overestimation. Note that, this laboratory analysed an 18 mm punch taken from the supplied filter.
- Laboratory 250783 obtained four performance scores requiring corrective actions.
- No CRM results were submitted laboratory 250783, nor by laboratory 250765, the latter due to poor analytical repeatability.

Overall, no methodological effect was identified for the analytical approaches applied in this ILC. The reproducibility standard deviations obtained for the filters ranged from 35–58% for galactosan, 20–42% for levoglucosan, and 32–37% for mannosan. For the CRM, the corresponding values were 122% for galactosan, 93% for levoglucosan, and 109% for mannosan. Due to the limited number of laboratories providing quantified CRM results, performance scores could not be established for galactosan and mannosan.

The uncertainties observed in this comparison were generally higher than those reported in CEN/TS 18044 or during previous ILCs. Despite similar concentrations measured on filter sections F1 and F2, the large discrepancy between their calculated uncertainties remains unexpected. Given the results obtained and the significant variability observed, it would be advisable to organise a new interlaboratory comparison.

# 9 References


- **CEN TS 18044: 2024,** Ambient air - Determination of the concentration of levoglucosan - Chromatographic method.
- **Chiara Pietrogrande,** M., Barbaro, E., Bove, M.C., Clauser, G., Colombi, C., Corbella, L., Cuccia, E., Dalla Torre, S., Decesari, S., Fermo, P., Gambaro, A., Gianelle, V., Ielpo, P., Larcher, R., Lazzeri, P., Massabò, D., Melchionna, G., Nardin, T., Paglione, M., Perrino, C., Prati, P., Visentin, M., Zanca, N., Zangrando, R., **2017.** Results of an interlaboratory comparison of analytical methods for quantification of anhydrosugars and biosugars in atmospheric aerosol. Chemosphere 184, 269–277. https://doi.org/10.1016/j.chemosphere.2017.05.131
- **Directive (EU) 2024/2881,** Directive (EU) 2024/2881 of the European Parliament and of the Council of 23 October 2024 on ambient air quality and cleaner air for Europe (recast), 2024
- **EN ISO/CEI 17043,** General requirements concerning to aptitude tests.
- **ISO 5725-1,** Application of statistics – Accuracy (correctness and reliability) of the measuring results and methods – Part 1: general principles and definitions,
- **ISO 5725-2,** Application of statistics – Accuracy (correctness and reliability) of the measuring results and methods – Part 2: basic method for determining the repeatability and reproducibility of a standardized measurement method,
- **ISO 5725-5,** Application of statistics – Accuracy (correctness and reliability) of the measuring results and methods – Part 5: alternative methods for determining the reliability of a standardized measurement method,
- **ISO 13528,** Statistical methods used in aptitude tests through inter-laboratory comparisons,
- **ISO 3534-2,** Vocabulary and symbols Part 2: Applied statistics,
- **Jaffrezo,** J.L., Putaud, J.-P., Swietlicki, E., **2019**. Deliverable D3.10: An Inter-laboratory comparison (ILC) studies for the targeted set of OA tracers. ACTRIS.
- **LAB-CIL REF 02,** Cofrac reference document relative to the organizers of inter-laboratory comparisons – Requirements for accreditation – Revision 06 – October 2024,
- **Louchouarn,** P., Kuo, L.-J., Wade, T.L., Schantz, M., **2009**. Determination of levoglucosan and its isomers in size fractions of aerosol standard reference materials. Atmospheric Environment 43, 5630–5636. https://doi.org/10.1016/j.atmosenv.2009.07.040
- **NF X 06-050,** Application of statistics – Study of the normality of a distribution.
- **Verlhac, S.**, Favez, O., Albinet, A., **2013**. Interlaboratory comparison organized for the European laboratories involved in the analysis of levoglucosan and its isomers. LCSQA.
- **Yttri, K.E.,** Schnelle-Kreis, J., Maenhaut, W., Abbaszade, G., Alves, C., Bjerke, A., Bonnier, N., Bossi, R., Claeys, M., Dye, C., Evtyugina, M., García-Gacio, D., Hillamo, R., Hoffer, A., Hyder, M., Iinuma, Y., Jaffrezo, J.-L., Kasper-Giebl, A., Kiss, G., López-Mahia, P.L., Pio, C., Piot, C., Ramirez-Santa-Cruz, C., Sciare, J., Teinilä, K., Vermeylen, R., Vicente, A., Zimmermann, R., **2015.** An intercomparison study of analytical methods used for quantification of levoglucosan in ambient aerosol filter samples. Atmos. Meas. Tech. 8, 125–147. https://doi.org/10.5194/amt-8-125-2015

# 10 Glossary

| Abbreviations | Labels |
|---|---|
| GC-MS | Gas Chromatography - Mass Spectrometry |
| HPLC-MS | High Performance Liquid Chromatography - Mass Spectrometry |
| IC-PAD | Ion Chromatography - Pulsed Amperometric Detection |
| ILC | An Inter-Laboratory Comparison is defined an implemented to allow laboratories to evaluate and demonstrate their performance level in specific testing, calibration or measuring sectors<br>NB: Three expressions may be used too: “inter-laboratory tests”, “inter-comparison tests” or “proficiency test” |
| LCSQA | Central Air Quality Laboratory |
| LoQ or LQ | Limit of Quantification |
| Test material | Matrix of interest containing the object subject to the inter-laboratory comparison, optionally added using a spiking solution |
| Matrix | Natural element, physical element and all of their components other than the species to be analyzed in which the substance subject to the inter-laboratory comparison is placed |
| OILC | Organizer of Inter-Laboratory Comparisons |

# 11 List of annexes

| Annexes | Titles | Pages |
|---|---|---|
| **Annex 1** | General organization, description of audits and algorithms | 10 |
| **Annex 2** | Input data, means and repeatability standard deviations of the laboratories and values discarded by expert opinion | 9 |

# Annex 1

*General organization, description of audits and algorithms*

**The organization is broken down into five separate phases**

① **23rd July to 08th September 2025**: Administrative phase including making contact, registration and sending the confidential code;

② **December 2024 to September 2025**: Preparation and test phase for test materials;

③ **23 rd September to 24 th October 2025**: Shipping, analysis phase at participants and results collection;

④ **January to April 2026**: Statistical analysis phase according to the procedures described later in the report, drafting of the final report.

**Number of laboratories participating in the test**

A minimum number of 10 participants is required to obtain a low level of uncertainty of assigned value.

If the number of participants is less than 10 for a campaign, the decision to postpone or maintain the campaign will be taken by the Advisory Group. If the campaign is maintained, the advisory group will have to define the most appropriate statistical treatment. The coordinator will inform the participants.

**Prior checks of the test materials**

Before the test materials are distributed, the organizer must demonstrate that the material subject to the test is stable and homogenous enough. In some cases, the homogeneity tests cannot be done prior to distribution for technical, historical or logistical reasons[1].

In our case, the homogeneity and stability monitoring have been performed prior the analysis by the laboratories. These two parameters were verified based on the evaluation criteria of appendix B of standard NF ISO 13528.

[1] Organizers of inter-laboratory comparisons, requirements for Accreditation – COFRAC Document LAB-CIL REF02.

## HOMOGENEITY

The following procedure applies when the homogeneity is evaluated via analysis.

The homogeneity of the material is verified when it is distributed, i.e., from the analytical results obtained on D+1.

ISO 13528 standard advises that the ratio between repeatability standard deviation from method and the proficiency testing standard deviation is < 0.5:

$$s_{rm}/\sigma_{pt} < 0.5$$

On the contrary, homogeneity criterion is enlarged with the formula described to paragraph B.2.3 from standard ISO 13528.

- Case n°1: $s_{rm}/\sigma_{pt} < 0.5$

The formulas are those quoted in standard ISO 13528, with:

$$S_x = \sqrt{\sum (x_t - X)^2/(g-1)}\ ,$$

with:

$S_x$: mean standard deviations of the determinations on D+1,
$x_t$ : the determination values,
X : mean of g $x_t$,
g : number of samples subject to homogeneity verification

$$Sw = \sqrt{\sum w_t^2 / 2g}\ ,$$

with:

$S_w$ : intra-sample standard deviation,

$w_t = x_{t1} - x_{t2}$,

$$Ss = \sqrt{S_X^2 - (\frac{S_w^2}{2})}$$

$S_s$ : inter-sample standard deviation,

For each parameter, the coordinator examines the homogeneity criterion. The test material will be considered to have a satisfactory homogeneity if and only if $S_s / \sigma_{pt} \leq 0.3$. If $S_s^2$ is negative, then the material is considered homogeneous.

with:

$\sigma_{pt}$ = population variability descriptor, robust standard deviation or pre-established value.

- Case n°2: $s_{rm}/\sigma_{pt} \geq 0.5$

The homogeneity criterion is enlarged to taken account the real repeatability standard deviations of the used method.
In addition to the previous formula, the next calculations are used

$$\sigma_{allow} = (0.3\ \sigma_{pt})^2$$

And

$$c = F1\ \sigma allow + F2\ s^2w$$

F1 and F2 are extracted from the table B.1 of standard ISO 13528.
If,

$$ss^2 \leq c$$

the material is homogenous.

In the case where an inter-laboratory test material cannot be considered homogenous ($s_s$ / $\sigma_{pt}$> 0.3 or $s_s^2$>c**),** the coordinator reserves the right, in accordance with chapter B.2 5C of standard ISO 13528, to include the inter-sample standard deviation in the standard deviation of the aptitude tests.

When the preparation process is the same year after year, the homogeneity study is not performed with an analysis step. The condition is to have an experience for a few years (5 years) on the parameter for the matrix used.

The process applied is described below:

- To summarize the relative robust standard deviations obtained during the five previous ILCs;
- To calculate the median (Meds*);
- To calculate the standard deviation ($s_{s^*}$) and to multiplicate by two,
- To check if:
  - $s^* \leq Med_{s^*} + 2s_{s^*}$

If this relation is right, the material provided is not different than the previous one.

If the relation is not checked, the homogeneity study is performed using analysis to verify if this variation is due to the sample itself. This study is performed after the analysis step by the laboratories.

## ■ STABILITY

The stability of the material is verified at a frequency defined in cooperation with the advisory group and based on each team's planning possibilities.

**Calculation of the means:**

- mean of the determinations on D+1, X,
- mean of the determinations on D+X, Y.

After statistical processing of the data sent by the participants, the coordinator adds the mean *m* and standard deviation *σ* values to each calculation sheet for the aptitude evaluation of the population.

For each parameter, the coordinator examines the stability criterion by using the evaluation criterion from standard ISO 13528.

The general mean of the determinations obtained during the verification of the homogeneity (on D+1, X) is compared to the general mean of the results obtain during verification of the stability (on D+X, Y) taking account analytical uncertainty. The samples are stable if:

$$|X - Y| \leq 0.3\sigma_{pt} + \sqrt{u_x^2 + u_y^2}$$ (Appendix B.5.2)

Otherwise, the coordinator examines the results with the affected technical experts and the statistician to decide on the outcome of the campaign for the affected parameter or family of parameters.

## STATISTICAL EXPLOITATION OF THE RESULTS

### Prior data audit before beginning statistical calculations

- ***Study of raw data***

All the raw data collected at the end of an ILC first undergoes an expert opinion step to eliminate certain values, if necessary, during the calculation of the assigned value. That is the case for:

- returned values below the limit of quantification, which are processed using the methodology described in table below.
- entered values equal to 0.
- values for which a dilution or retrieval error in the required unit is shown (for example, a factor 1000).

**Table: Methodology for processing responses containing one or more threshold values**

Retrieval of 4 values

| | Data received | Data considered |
|---|---|---|
| 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 |

Retrieval of 3 values

| | Data received | Data considered |
|---|---|---|
| 1st case | C, C, <LQ | C, C |
| 2nd case | C, <LQ, <LQ | none |
| 3rd case | <LQ, <LQ, <LQ | none |

Retrieval of 2 values

| | Data received | Data considered |
|---|---|---|
| 1st case | C, <LQ | none |
| 2nd case | <LQ, <LQ | none |

The abnormal nature of these values may be viewed using Henry's line. These exclusions are submitted for approval to the Advisory Group's experts and outlined in the inter-laboratory comparison report.

- ***Abnormal value and coherence tests***

Standard ISO 5725-5 (§ 6.1.4), allows determination of the repeatability and reproducibility of a measurement method, recommends applying the outlier value tests (Grubbs test and Cochran test) and coherence verifications (Mandel's *h* and *k* statistics), such that the participants and the organizer, in a measure to improve the implementation of the analysis methods, seek out, in particular based on comments provided by the laboratory on the results form or the observations, the origin of the abnormal nature of the values detected as being inconsistent (for example calculation error, or conversion error, etc.).

All the abnormal value and coherence tests are outlined in the inter-laboratory comparison report.

**Materials prepared by Ineris:**

The methodology implemented to determine the assigned value, its associated uncertainty and the standard deviation for tests prepared by the Ineris is described below:

- ***Principle of the robust analysis of the tests***

The basic method for determining the repeatability and reproducibility of a measurement method described in standard ISO 5725-2 requires using tests for abnormal values (Cochran and Grubbs tests) to identify the data that must be excluded from statistical calculations.

However, in practice, by applying the abnormal value tests, the data analysis may led to exercise judgment to decide what data must be excluded (for example, if the tests detect one abnormal value and several questionable values for a laboratory: partial or total elimination of its data?).

The data analyst's decision may therefore, in some cases, have a substantial impact on the calculated values of the repeatability and reproducibility standard deviations, as well as the calculation of the mean used as reference value and the performance evaluation (laboratory score). The interest of the robust data analysis as described in standard ISO 5725-5, relative to the basic analysis, lies in calculating the assigned value and other statistical parameters from all of the data, including that which could be deemed suspicious by an expert statement or abnormal value test: the applied data processing minimizes the weight of the suspicious values, i.e., "extreme" values, so that they do not significantly affect the value of that assigned value.

Thus, the calculations of the assigned value (reference value), confidence intervals and performance statistics are not affected by the data analyst's judgment. **Participants' results are processed completely impartially and transparently.**

- ***Determination of the assigned value***
  - ✓ ***Consensus value***

The assigned value for each parameter being subject to an inter-laboratory comparison, it is determined in accordance with standards ISO 13528 and ISO 5725-5.

The assigned value is also taken to be equal to the robust mean of the results provided by the participants in the inter-laboratory comparison (cf. appendix C of standard ISO 13528).

$$x_{pt} = x^*$$

Even if abnormal values are detected by the coherence and abnormal value tests, they are not excluded to calculate the robust mean.

The robust mean $x^*$ is calculated by applying the A algorithm. The iterations are repeated until the convergence is ensured, i.e., the 4th rounded decimal place of the robust mean and of the robust standard deviation no longer changes.

The mean $\overline{x_i}$ of each of the $p$ participants is calculated, then the $p$ means are ranked in decreasing order.

- The initial value of the robust mean $x^*$ is equal to the median of the $p$ means,

$$x^* = \text{median of } \overline{x_i} \qquad (i = 1.2,....p)$$

The initial value of the robust standard deviation $s^*$ is equal to:

$$s^* = 1.483 \times \text{median of } \left|\overline{x_i} - x^*\right| \qquad (i = 1,2,....p)$$

- The value of $x^*$ is updated as follows:

$$\varphi = 1.5 \times s^*$$

For each value $\overline{x_i}$, the following calculation is done: $x_i^* = \begin{cases} x^* - \varphi & \text{if} \quad \overline{x_i} < x^* - \varphi \\ x^* + \varphi & \text{if} \quad \overline{x_i} > x^* + \varphi \\ x_i^* & \text{otherwise} \end{cases}$

The new robust mean value is equal to:

$$x^* = \sum_{i=1}^{p} \frac{x_i^*}{p}$$

The new robust standard deviation value is equal to:

$$s^* = 1.134 \sqrt{\frac{\sum_{i=1}^{p} (x_i - x^*)^2}{p-1}}$$

- ✓ ***Formulation***

In case of population number < 10, it will be preferable to set the assigned value, when possible. This assigned value will be equal to the spiking value.

$$x_{pt} = x_{spiking}$$

In this case, all references related to the material, standards will be noted to ensure a metrological tracking and so, to calculate his uncertainty.

- ***Determination of the uncertainty associated with the assigned value (robust method)***
  - ✓ ***Robust method***

The uncertainty of the assigned value $u_{x^*}$ is estimated by:

$$u_{x^*} = 1.25 \times s^* / \sqrt{p}$$

When $u_{x^*}$ <0,3s*, chapter 9.2.1 of standard NF ISO 13528 recommends disregarding the uncertainty of the assigned value and not including it in the interpretation of the aptitude test results, i.e., in the statistical performance test. On the contrary, this uncertainty is included in the standard deviation for the aptitude evaluation:

$$\sigma'_{pt} = \sqrt{s^{*2} + u_{x*}^2}$$

  - ✓ ***Formulation***

The assigned value uncertainty is a combination of all uncertainties related to the material preparation, homogeneity, transport and stability.

$$u_{(xpt)} = \sqrt{u_{char}^2 + u_{hom}^2 + u_{trans}^2 + u_{stab}^2}$$

with,

$u(x_{pt})$ : assigned value uncertainty,

$u_{char}$ : characterisation uncertainty (material preparation),

$u_{hom}$ : homogeneity uncertainty (measured during the homogeneity study),

$u_{trans}$ : transport uncertainty incertitude–type liée au transport,

$u_{stab}$ : stability uncertainty.

The assigned value uncertainty is negligible if:

$$u_{xpt} < 0{,}3 \times \sigma_{pt}$$

Otherwise, uncertainty is included in the aptitude standard deviation calculation.

Note: If there is no transport and/or stability effect and /or homogeneity effect, their uncertainty will be equal to 0.

- ***Determination of the standard deviation for the aptitude evaluation $\sigma_{pt}$***

Among the 5 methods proposed by standard ISO 13528 to calculate the standard deviation $\sigma_{pt}$ for the aptitude evaluation (i.e., for the laboratory performance evaluation), that selected is the determination from the participants' results.

When the inter-laboratory comparison medium does not correspond to a Certified Reference Material, the standard deviation for the aptitude evaluation $\sigma_{pt}$ is determined from the participants' results. It is considered to be equal to the robust standard deviation $s*$ estimated by applying the A Algorithm, as previously described.

**Special case: Inhomogeneous material**

When the homogeneity tests for the test materials find that there is a lack of homogeneity, the coordinator takes the inter-sample standard deviation into account in the standard deviation of the aptitude tests, so as not to impute the bias related to the variability of the distributed test materials to the laboratories during performance tests. The standard deviation for the aptitude evaluation is recalculated as follows:

$$\sigma'_{pt} = \sqrt{s^{*2} + S_S^2}$$

Where:

$\sigma'_{pt}$: recalculated standard deviation for assessment.

s*: standard deviation for the aptitude evaluation, calculated by the robust analysis.

$S_s$ : the inter-sample standard deviation of the distributed test materials.

**Special case: Non negligible uncertainty and non-homogeneous material**

When an assigned value uncertainty is non negligible and a non-homogeneous material, there is a new calculation of aptitude standard deviation.

$$\sigma'_{pt} = \sqrt{{\sigma_{pt}}^2 + u_{xpt}^2 + Ss^2}$$

$\sigma'_{pt}$: new standard deviation for assessment,

$\sigma_{pt}$ : standard deviation for assessment (set or s*),

$u_{xpt}$: assigned value uncertainty,

Ss: between sample standard deviation.

**Statistical performance tests**

The laboratories' performance is evaluated using the z score or z' score.

- ***The z score or z' score***

The z score and z' score are calculated as follows:

$$z_i = \frac{\overline{x_i} - x_{pt}}{\sigma_{pt}} \qquad z'_i = \frac{\overline{x_i} - x_{pt}}{\sigma'_{pt}}$$

Where:

$\sigma_{pt}$ is the estimate of the standard deviation for the aptitude evaluation,

$\sigma'_{pt}$ is the estimate of the recalculated standard deviation for the aptitude evaluation as describe before,

$\overline{x_i}$ is the mean concentration measured by the laboratory i,

$x_{pt}$ corresponds to the considered assigned.

If the test material prepared by the Ineris is not homogenous and/or his uncertainty is not negligible, so z score becomes a z' score.

## Evaluation of each laboratory's performance:

A laboratory with a z or z' score greater or equal than 3.0 or less or equal than -3.0 leads to an "action signal". A z score strictly greater than 2.0 or strictly less than -2.0 leads to a warning signal (§9 4.2 standard ISO 13528).

## Bias

Although the bias (or difference) is not the chosen performance indicator, it can be provided to laboratories as an indication. It is calculated using the following equation:

$$\text{Bias} = \frac{x_i - x_{pt}}{x_{pt}} \times 100$$

$x_i$: measured value of the laboratory,
$x_{pt}$: assigned value.

# Annex 2

## Input data, means and repeatability standard deviations of the laboratories and values discarded by expert opinion

*For each studied matrix, all the participants' results are presented, including the discarded values of the statistical calculations (expert opinion). For the latter, the reason for which they are not considered is specified.*

## Table 1 – 25-232849 F1 – Galactosan - Raw data in µg

| Id lab | Meas. 1 | Meas.2 | Meas.3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction- Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 1.45 | 1.40 | 1.45 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 1.433 | 0.029 | 2.0% | OK | |
| 250718 | 6.35 | 6.56 | 5.63 | 10 | 30/09/2025 | 13/10/2025 | Inhouse method/Stirring/Acetonitrile /GC/MS | 6.180 | 0.488 | 7.9% | OK | |
| 250740 | 3.11 | 3.07 | 3.24 | 17 | 17/10/2025 | 18/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 3.140 | 0.089 | 2.8% | OK | |
| 250745 | 3.47 | 3.56 | 3.38 | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 3.470 | 0.090 | 2.6% | OK | |
| 250751 | # | # | # | | 30/09/2025 | 30/09/2025 | NA | NA | NA | NA | Excl. | |
| 250765 | 2.00 | 2.10 | 2.14 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 2.080 | 0.072 | 3.5% | OK | |
| 250766 | 3.34 | 3.25 | 3.34 | 25 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 3.310 | 0.052 | 1.6% | OK | |
| 250775 | 3.21 | 3.13 | 3.19 | 15 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane/Acetone/GC/MS | 3.177 | 0.042 | 1.3% | OK | |
| 250778 | ~~99.91~~ | ~~98.48~~ | ~~99.98~~ | 1 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 99.457 | 0.847 | 0.9% | Excl. | |
| 250783 | 15.21 | 15.24 | 14.73 | 40 | 03/10/2025 | 08/10/2025 | Inhouse method/Sonication/MeOH/GC/MS | 15.060 | 0.286 | 1.9% | OK | |

## Table 2 – 25-232849 F1 – Levoglucosan - Raw data in µg

| Id lab | Meas. 1 | Meas.2 | Meas.3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction- Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 37.79 | 37.80 | 38.46 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 38.017 | 0.384 | 1.0% | OK | |
| 250718 | 36.10 | 33.75 | 32.25 | 13 | 30/09/2025 | 13/10/2025 | Inhouse method/Stirring/Acetonitrile /GC/MS | 34.033 | 1.941 | 5.7% | OK | |
| 250740 | 88.36 | 90.76 | 89.09 | 21 | 17/10/2025 | 18/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 89.403 | 1.230 | 1.4% | OK | |
| 250745 | 99.71 | 97.11 | 97.29 | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 98.037 | 1.452 | 1.5% | OK | |
| 250751 | 76.78 | 76.73 | 76.63 | 24 | 30/09/2025 | 30/09/2025 | VDI 2444///Water/CI-DAP | 76.713 | 0.076 | 0.1% | OK | |
| 250765 | 80.97 | 81.06 | 81.06 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 81.030 | 0.052 | 0.1% | OK | |
| 250766 | 84.19 | 79.19 | 80.50 | 20 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 81.293 | 2.593 | 3.2% | OK | |
| 250775 | 64.00 | 62.16 | 63.03 | 15 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane/Acetone/GC/MS | 63.063 | 0.920 | 1.5% | OK | |
| 250778 | ~~2 688.31~~ | ~~2 657.94~~ | ~~2 677.92~~ | 1 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 2 674.723 | 15.435 | 0.6% | Excl. | |
| 250783 | 188.89 | 177.75 | 180.99 | 40 | 03/10/2025 | 08/10/2025 | Inhouse method/Sonication/MeOH/GC/MS | 182.543 | 5.730 | 3.1% | OK | |

## Table 3 – 25-232849 F1 – Mannosan - Raw data in µg

| Id lab | Meas. 1 | Meas.2 | Meas.3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction- Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 4.88 | 4.94 | 5.05 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 4.957 | 0.086 | 1.7% | OK | |
| 250718 | 5.79 | 5.68 | 6.02 | 18 | 30/09/2025 | 13/10/2025 | Inhouse method/Stirring/Acetonitrile /GC/MS | 5.830 | 0.173 | 3.0% | OK | |
| 250740 | 5.78 | 5.79 | 6.04 | 11 | 17/10/2025 | 18/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 5.870 | 0.147 | 2.5% | OK | |
| 250745 | 7.54 | 7.58 | 7.38 | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 7.500 | 0.106 | 1.4% | OK | |
| 250751 | # | # | # | | | | NA | NA | NA | NA | Excl. | |
| 250765 | 3.89 | 3.88 | 3.89 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 3.887 | 0.006 | 0.1% | OK | |
| 250766 | 8.11 | 7.95 | 7.97 | 20 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 8.010 | 0.087 | 1.1% | OK | |
| 250775 | 6.53 | 6.61 | 6.45 | 15 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane/Acetone/GC/MS | 6.530 | 0.080 | 1.2% | OK | |
| 250778 | ~~229.66~~ | ~~231.37~~ | ~~230.16~~ | 1 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 230.397 | 0.879 | 0.4% | Excl. | |
| 250783 | 41.79 | 41.83 | 42.74 | 40 | 03/10/2025 | 08/10/2025 | Inhouse method/Sonication/MeOH/GC/MS | 42.120 | 0.537 | 1.3% | OK | |

**Table 4 – 25-232849 F1 – Comment**

| Id lab | Comment |
|---|---|
| 250700 | VDI 2444 (2020-03) LC-MS/MS |
| 250718 | Solvant d'extraction : Acétonitrile<br>Filtration puis évaporation avant reprise dans un mélange ACN/BSTFA (agent dérivatisant)<br>Méthode d'analyse : Impact Électronique en full scan avec m/z allant de 50 à 500 puis extraction de l'ion d'intérêt m/z 204 pour la quantification des 3 composés. Utilisation d'un étalon interne : acide succinique-d4. Gamme d'étalonnage réalisées sur filtres blancs et extraites comme un échantillon. |
| 250740 | RESEARCH PROCEDURE (RP 34): Determination of biomass combustion markers and sugars using ion chromatography with pulsed amperometric detection |
| 250745 | Two methods were used for determination of anhydrosugars, one for levoglucosan and another for mannosan and galactosan. Samples were prepared on 10/01/2025. Date of analysis of levoglucosan was 10/01/2025, and date of mannosan and galactosan analysis was 10/03/2025. |
| 250751 | Technique d'analyse: IC-PAD, Méthode d'analyse : VDI 2444<br>This are our results of filter 25-232849_F1 |
| 250765 | The title of the tab is incorrect, The table heading shows the correct sample number. |
| 250766 | RAS |
| 250775 | DCM/Ace (1:1) |
| 250778 | Échantillons conservés à 5°C entre la réception et la préparation.<br>Extraction et l'analyse ont été réalisés par le même opérateur.<br>Le filtre n'a pas été extrait en totalité ; Un punch de 18mm de diamètre a été prélevé et extrait. |
| 250783 | méthode interne :<br>- agent de dérivation : MSTFA (N-méthyl-N-(triméthylsilyl)trifluoroacetamide) + 1% TCMS (triméthylchlorosilane),<br>- étalon interne de dérivation : méthyl-β-D-xylopyranoside<br>- étalon d'injection : phényldodécane (PDD)<br>Extraction aux ultrason après ajout de MeOH<br>Récupération de l'extrait méthanolique, concentration à l'ASE suivi d'une évaporation à sec à l'étuve, reprise avec pyridine anhydre + MSTFA + PDD.<br>Extrait à la pyridine étuvé à 80 °C pendant 2 heures<br>Analyse par GC/MS (mode SIM) |

## Table 5 – 25-232849 F2 – Galactosan - Raw data in µg

| Id lab | Meas. 1 | Meas. 2 | Meas. 3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 1.85 | 1.77 | 1.77 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 1.797 | 0.046 | 2.6% | OK | |
| 250718 | 3.91 | 3.91 | 3.69 | 11 | 30/09/2025 | 13/10/2025 | Inhouse method/Stirring/Acetonitrile /GC/MS | 3.837 | 0.127 | 3.3% | OK | |
| 250740 | 3.05 | 3.04 | 3.20 | 17 | 17/10/2025 | 18/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 3.097 | 0.090 | 2.9% | OK | |
| 250745 | 2.95 | 2.95 | 2.84 | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 2.913 | 0.064 | 2.2% | OK | |
| 250751 | # | # | # | | 30/09/2025 | 30/09/2025 | NA | NA | NA | NA | Excl. | |
| 250765 | 1.93 | 1.88 | 1.97 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 1.927 | 0.045 | 2.3% | OK | |
| 250766 | 2.57 | 2.54 | 2.52 | 25 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 2.543 | 0.025 | 1.0% | OK | |
| 250775 | 3.13 | 3.02 | 3.07 | 15 | 23/10/2025 | 20/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane-Acetone/GC/MS | 3.073 | 0.055 | 1.8% | OK | |
| 250778 | ~~81.04~~ | ~~78.60~~ | ~~75.63~~ | 4 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 78.423 | 2.709 | 3.5% | Excl. | According to outlier test |
| 250783 | 5.08 | 4.98 | 4.92 | 40 | 03/10/2025 | 08/10/2025 | Inhouse method/Sonication/MeOH/GC/MS | 4.993 | 0.081 | 1.6% | OK | |

## Table 6 – 25-232849 F2 – Levoglucosan - Raw data in µg

| Id lab | Meas. 1 | Meas. 2 | Meas. 3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 53.59 | 53.26 | 53.46 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 53.437 | 0.166 | 0.3% | OK | |
| 250718 | 22.87 | 21.92 | 19.63 | 20 | 30/09/2025 | 13/10/2025 | Inhouse method/Stirring/Acetonitrile /GC/MS | 21.473 | 1.666 | 7.8% | OK | |
| 250740 | 84.36 | 85.84 | 86.04 | 21 | 17/10/2025 | 18/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 85.413 | 0.918 | 1.1% | OK | |
| 250745 | 80.38 | 80.64 | 79.50 | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 80.173 | 0.597 | 0.7% | OK | |
| 250751 | 68.43 | 68.26 | 68.52 | 24 | 30/09/2025 | 30/09/2025 | VDI 2444///Water/CI-DAP | 68.403 | 0.132 | 0.2% | OK | |
| 250765 | 70.43 | 70.35 | 70.29 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 70.357 | 0.070 | 0.1% | OK | |
| 250766 | 63.40 | 62.58 | 62.81 | 20 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 62.930 | 0.423 | 0.7% | OK | |
| 250775 | 71.44 | 69.48 | 70.05 | 15 | 23/10/2025 | 20/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane-Acetone/GC/MS | 70.323 | 1.008 | 1.4% | OK | |
| 250778 | ~~2 315.20~~ | ~~2 287.36~~ | ~~2 232.03~~ | 2 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 2 278.197 | 42.335 | 1.9% | Excl. | According to outlier test |
| 250783 | 73.09 | 70.61 | 72.22 | 40 | 03/10/2025 | 08/10/2025 | Inhouse method/Sonication/MeOH/GC/MS | 71.973 | 1.258 | 1.7% | OK | |

## Table 7 – 25-232849 F2 – Mannosan - Raw data in µg

| Id lab | Meas. 1 | Meas. 2 | Meas. 3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 4.51 | 4.56 | 4.69 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 4.587 | 0.093 | 2.0% | OK | |
| 250718 | 3.94 | 3.80 | 3.24 | 36 | 30/09/2025 | 13/10/2025 | Inhouse method/Stirring/Acetonitrile /GC/MS | 3.660 | 0.370 | 10.1% | OK | |
| 250740 | 5.93 | 5.97 | 5.96 | 11 | 17/10/2025 | 18/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 5.953 | 0.021 | 0.3% | OK | |
| 250745 | 6.57 | 6.55 | 6.54 | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 6.553 | 0.015 | 0.2% | OK | |
| 250751 | # | # | # | | 30/09/2025 | 30/09/2025 | NA | NA | NA | NA | Excl. | |
| 250765 | 2.98 | 3.00 | 3.01 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 2.997 | 0.015 | 0.5% | OK | |
| 250766 | 6.42 | 6.33 | 6.32 | 20 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 6.357 | 0.055 | 0.9% | OK | |
| 250775 | 6.41 | 6.29 | 6.43 | 15 | 23/10/2025 | 20/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane-Acetone/GC/MS | 6.377 | 0.076 | 1.2% | OK | |
| 250778 | ~~199.53~~ | ~~197.15~~ | ~~194.25~~ | 2 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 196.977 | 2.644 | 1.3% | Excl. | According to outlier test |
| 250783 | 13.71 | 13.40 | 13.04 | 40 | 03/10/2025 | 08/10/2025 | Inhouse method/Sonication/MeOH/GC/MS | 13.383 | 0.335 | 2.5% | OK | |

**Table 8 – 25-232849 F2 – Comment**

| Id lab | Comment |
|---|---|
| 250700 | VDI 2444 (2020-03) LC-MS/MS |
| 250718 | Solvant d'extraction : Acétonitrile<br>Filtration puis évaporation avant reprise dans un mélange ACN/BSTFA (agent dérivatisant)<br>Méthode d'analyse : Impact Électronique en full scan avec m/z allant de 50 à 500 puis extraction de l'ion d'intérêt m/z 204 pour la quantification des 3 composés. Utilisation d'un étalon interne : acide succinique-d4. Gamme d'étalonnage réalisées sur filtres blancs et extraites comme un échantillon. |
| 250740 | RESEARCH PROCEDURE (RP 34): Determination of biomass combustion markers and sugars using ion chromatography with pulsed amperometric detection |
| 250745 | Two methods were used for determination of anhydrosugars, one for levoglucosan and another for mannosan and galactosan. Samples were prepared on 10/01/2025. Date of analysis of levoglucosan was 10/01/2025, and date of mannosan and galactosan analysis was 10/03/2025. |
| 250751 | Technique d'analyse: IC-PAD, Méthode d'analyse : VDI 2444 |
| 250765 | no commends |
| 250766 | RAS |
| 250775 | DCM/ACe (1:1) |
| 250778 | Échantillons conservés à 5°C entre la réception et la préparation.<br>Extraction et l'analyse ont été réalisés par le même opérateur.<br>Le filtre n'a pas été extrait en totalité ; Un punch de 18mm de diamètre a été prélevé et extrait. |
| 250783 | méthode interne :<br>- agent de dérivation : MSTFA (N-méthyl-N-(triméthylsilyl)trifluoroacetamide) + 1% TCMS (triméthylchlorosilane),<br>- étalon interne de dérivation : méthyl-β-D-xylopyranoside<br>- étalon d'injection : phényldodécane (PDD)<br>Extraction aux ultrason après ajout de MeOH<br>Récupération de l'extrait méthanolique, concentration à l'ASE suivi d'une évaporation à sec à l'étuve, reprise avec pyridine anhydre + MSTFA + PDD.<br>Extrait à la pyridine étuvé à 80 °C pendant 2 heures<br>Analyse par GC/MS (mode SIM) |

## Table 9 – 25-232849 Dust – Galactosan - Raw data in mg/kg dry mass

| Id lab | Meas. 1 | Meas. 2 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 2.33 | 2.45 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 2.390 | 0.085 | 3.6% | OK | |
| 250718 | <6.8 | <6.8 | 150 | 22/10/2025 | 20/10/2025 | Inhouse method/Stirring/Acetonitrile/GC/MS | 6.800 | 0.000 | 0.0% | Excl. | <LoQ |
| 250740 | <1.45 | <1.46 | 17 | 17/10/2025 | 19/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 1.455 | 0.007 | 0.5% | Excl. | <LoQ |
| 250745 | 3.02 | 3.11 | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 3.065 | 0.064 | 2.1% | OK | |
| 250751 | # | # | | 30/09/2025 | 30/09/2025 | NA | NA | NA | NA | Excl. | |
| 250765 | # | # | # | 13/10/2025 | 13/10/2025 | NA | NA | NA | NA | Excl. | |
| 250766 | 9.59 | 8.97 | 25 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 9.280 | 0.438 | 4.7% | OK | |
| 250775 | 0.63 | 0.57 | 15 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane - acetone/GC/MS | 0.600 | 0.042 | 7.1% | OK | |
| 250778 | 0.75 | 0.81 | 8 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 0.780 | 0.042 | 5.4% | OK | |
| 250783 | # | # | | 03/10/2025 | 08/10/2025 | NA | NA | NA | NA | Excl. | |

## Table 10 – 25-232849 Dust – Levoglucosan - Raw data in mg/kg dry mass

| Id lab | Meas. 1 | Meas. 2 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 207.20 | 200.22 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 203.710 | 4.936 | 2.4% | OK | |
| 250718 | 18.03 | 18.46 | 80 | 22/10/2025 | 20/10/2025 | Inhouse method/Stirring/Acetonitrile/GC/MS | 18.245 | 0.304 | 1.7% | OK | |
| 250740 | 242.18 | 236.38 | 21 | 17/10/2025 | 19/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 239.280 | 4.101 | 1.7% | OK | |
| 250745 | 281.62 | 264.09 | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 272.855 | 12.396 | 4.5% | OK | |
| 250751 | 152.24 | 152.05 | 24 | 30/09/2025 | 30/09/2025 | VDI 2444/Autre/Water/CI-DAP | 152.145 | 0.134 | 0.1% | OK | |
| 250765 | # | # | # | 13/10/2025 | 13/10/2025 | NA | NA | NA | NA | Excl. | |
| 250766 | 154.40 | 149.37 | 20 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 151.885 | 3.557 | 2.3% | OK | |
| 250775 | 9.97 | 11.62 | 15 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane - acetone/GC/MS | 10.795 | 1.167 | 10.8% | OK | |
| 250778 | 3.90 | 4.25 | 9 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 4.075 | 0.247 | 6.1% | OK | |
| 250783 | # | # | | 03/10/2025 | 08/10/2025 | NA | NA | NA | NA | Excl. | |

## Table 11 – 25-232849 Dust – Mannosan - Raw data in mg/kg dry mass

| Id lab | Meas. 1 | Meas. 2 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | 27.58 | 27.39 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444/Sonication/Water/LC-MS/MS | 27.485 | 0.134 | 0.5% | OK | |
| 250718 | 10.93 | 11.29 | 158 | 22/10/2025 | 20/10/2025 | Inhouse method/Stirring/Acetonitrile/GC/MS | 11.110 | 0.255 | 2.3% | OK | |
| 250740 | 14.09 | 17.24 | 11 | 17/10/2025 | 19/10/2025 | Inhouse method/Sonication/Water/CI-DAP | 15.665 | 2.227 | 14.2% | OK | |
| 250745 | # | # | 17 | 01/10/2025 | 01/10/2025 | NA | NA | NA | NA | Excl. | |
| 250751 | # | # | | 30/09/2025 | 30/09/2025 | NA | NA | NA | NA | Excl. | |
| 250765 | # | # | # | 13/10/2025 | 13/10/2025 | NA | NA | NA | NA | Excl. | |
| 250766 | 42.63 | 38.60 | 20 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044/Sonication/Water/CI-DAP | 40.615 | 2.850 | 7.0% | OK | |
| 250775 | 1.65 | 1.71 | 15 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044/Sonication/Dichloromethane - acetone/GC/MS | 1.680 | 0.042 | 2.5% | OK | |
| 250778 | 0.40 | 0.44 | 9 | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044/Stirring/Water/CI-DAP | 0.420 | 0.028 | 6.7% | OK | |
| 250783 | # | # | | 03/10/2025 | 08/10/2025 | NA | NA | NA | NA | Excl. | |

**Table 12 – 25-232849 Dust – Comment**

| Id lab | Comment |
|---|---|
| 250700 | VDI 2444 (2020-03) LC-MS/MS |
| 250718 | Solvant d'extraction : Acétonitrile<br>Filtration puis évaporation avant reprise dans un mélange ACN/BSTFA (agent dérivatisant)<br>Méthode d'analyse : Impact Électronique en full scan avec m/z allant de 50 à 500 puis extraction de l'ion d'intérêt m/z 204 pour la quantification des 3 composés. Utilisation d'un étalon interne : acide succinique-d4. Gamme d'étalonnage réalisées sur filtres blancs et extraites comme un échantillon.<br>Concernant la partie extraction de poussière nous avons ajouté une étape de centrifugation supplémentaire pour éliminer le culot de poussière après l'extraction par agitation. Pour la détermination des LQ nous avons utilisé notre premier point de gamme comme point bas mais en réalité nous n'observons aucun pic. La LQ réelle est probablement beaucoup plus basse mais pas encore déterminée car encore en cours de développement. |
| 250740 | RESEARCH PROCEDURE (RP 34): Determination of biomass combustion markers and sugars using ion chromatography with pulsed amperometric detection |
| 250745 | Could not achieve good separation of mannosan, due to coelution with some unknown analyte. |
| 250751 | Technique d'analyse: IC-PAD, Méthode d'analyse : VDI 2444 |
| 250765 | No repeatable and stable results, therefore no results for dust 25-232849 |
| 250766 | RAS |
| 250775 | DCM/Ace (1:1) |
| 250778 | Échantillons conservés à 5°C, extraits et analysés par le même opérateur.<br>Les pesées ont été effectuées à l'aide d'une balance de précision (±0,0001 g), contrôlée annuellement. |
| 250783 | méthode interne :<br>- agent de dérivation : MSTFA (N-méthyl-N-(triméthylsilyl)trifluoroacetamide) + 1% TCMS (triméthylchlorosilane),<br>- étalon interne de dérivation : méthyl-β-D-xylopyranoside<br>- étalon d'injection : phényldodécane (PDD)<br>Extraction aux ultrason après ajout de MeOH<br>Récupération de l'extrait méthanolique, concentration à l'ASE suivi d'une évaporation à sec à l'étuve, reprise avec pyridine anhydre + MSTFA + PDD.<br>Extrait à la pyridine étuvé à 80 °C pendant 2 heures<br>Analyse par GC/MS (mode SIM) |

## Table 13 – 25-232849 F0 – Galactosan - Raw data in µg

| Id lab | Meas. 1 | Meas. 2 | Meas. 3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | <0.2 | <0.2 | <0.2 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444 | 0.200 | 0.000 | 0.0% | Excl. | <LQ |
| 250718 | <0.15 | <0.15 | <0.15 | 35 | 13/10/2025 | 30/09/2025 | Inhouse method | 0.150 | 0.000 | 0.0% | Excl. | <LQ |
| 250740 | <0.07 | <0.07 | <0.07 | 17 | 17/10/2025 | 18/10/2025 | Inhouse method | 0.070 | 0.000 | 0.0% | Excl. | <LQ |
| 250745 | # | # | # | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044 | NA | NA | NA | Excl. | |
| 250751 | # | # | # | | 30/09/2025 | 30/09/2025 | VDI 2444 | NA | NA | NA | Excl. | |
| 250765 | 0.00 | 0.00 | 0.00 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044 | 0.000 | 0.000 | Pb. Moy.=0 | Excl. | |
| 250766 | <0.2 | <0.2 | <0.2 | 25 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044 | 0.200 | 0.000 | 0.0% | Excl. | <LQ |
| 250775 | 0.01 | 0.01 | 0.01 | 20 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044 | 0.010 | 0.000 | 0.0% | OK | |
| 250778 | <1.41 | <1.41 | <1.41 | | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044 | 1.410 | 0.000 | 0.0% | Excl. | <LQ |
| 250783 | <0.1 | <0.1 | <0.1 | | 03/10/2025 | 08/10/2025 | Inhouse method | 0.100 | 0.000 | 0.0% | Excl. | <LQ |

## Table 14 – 25-232849 F0 – Levoglucosan - Raw data in µg

| Id lab | Meas. 1 | Meas. 2 | Meas. 3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | <0.2 | <0.2 | <0.2 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444 | 0.200 | 0.000 | 0.0% | Excl. | <LQ |
| 250718 | <0.15 | <0.15 | <0.15 | 35 | 13/10/2025 | 30/09/2025 | Inhouse method | 0.150 | 0.000 | 0.0% | Excl. | <LQ |
| 250740 | <0.05 | <0.05 | <0.05 | 21 | 17/10/2025 | 18/10/2025 | Inhouse method | 0.050 | 0.000 | 0.0% | Excl. | <LQ |
| 250745 | # | # | # | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044 | NA | NA | NA | Excl. | |
| 250751 | <4 | <4 | <4 | 24 | 30/09/2025 | 30/09/2025 | VDI 2444 | 4.000 | 0.000 | 0.0% | Excl. | <LQ |
| 250765 | 0.00 | 0.00 | 0.00 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044 | 0.000 | 0.000 | Pb. Moy.=0 | OK | |
| 250766 | <0.2 | <0.2 | <0.2 | 20 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044 | 0.200 | 0.000 | 0.0% | Excl. | <LQ |
| 250775 | 0.23 | 0.21 | 0.22 | 20 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044 | 0.220 | 0.010 | 4.5% | OK | |
| 250778 | <3.53 | <3.53 | <3.53 | | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044 | 3.530 | 0.000 | 0.0% | Excl. | <LQ |
| 250783 | <0.1 | <0.1 | <0.1 | | 03/10/2025 | 08/10/2025 | Inhouse method | 0.100 | 0.000 | 0.0% | Excl. | <LQ |

## Table 15 – 25-232849 F0– Mannosan - Raw data in µg

| Id lab | Meas. 1 | Meas. 2 | Meas. 3 | Uncertainty % | Analysis start | Analysis end | Method - Extraction - Technique | x | $s_r$ | $CV_r$ | Expert opinion | Comment |
|---|---|---|---|---|---|---|---|---|---|---|---|---|
| 250700 | <0.2 | <0.2 | <0.2 | 20 | 25/09/2025 | 26/09/2025 | VDI 2444 | 0.200 | 0.000 | 0.0% | Excl. | <LQ |
| 250718 | <0.15 | <0.15 | <0.15 | 35 | 13/10/2025 | 30/09/2025 | Inhouse method | 0.150 | 0.000 | 0.0% | Excl. | <LQ |
| 250740 | <0.09 | <0.09 | <0.09 | 11 | 17/10/2025 | 18/10/2025 | Inhouse method | 0.090 | 0.000 | 0.0% | Excl. | <LQ |
| 250745 | # | # | # | 17 | 01/10/2025 | 01/10/2025 | XP CEN/TS 18044 | NA | NA | NA | Excl. | |
| 250751 | # | # | # | | 30/09/2025 | 30/09/2025 | VDI 2444 | NA | NA | NA | Excl. | |
| 250765 | 0.00 | 0.00 | 0.00 | 20 | 09/10/2025 | 09/10/2025 | XP CEN/TS 18044 | 0.000 | 0.000 | Pb. Moy.=0 | Excl. | |
| 250766 | <0.2 | <0.2 | <0.2 | 20 | 09/10/2025 | 13/10/2025 | XP CEN/TS 18044 | 0.200 | 0.000 | 0.0% | Excl. | <LQ |
| 250775 | 0.05 | 0.05 | 0.05 | 20 | 23/10/2025 | 19/10/2025 | XP CEN/TS 18044 | 0.050 | 0.000 | 0.0% | OK | |
| 250778 | 1.41 | <1.41 | <1.41 | | 29/09/2025 | 29/09/2025 | XP CEN/TS 18044 | 1.410 | 0.000 | 0.0% | Excl. | <LQ |
| 250783 | <0.1 | <0.1 | <0.1 | | 03/10/2025 | 08/10/2025 | Inhouse method | 0.100 | 0.000 | 0.0% | Excl. | <LQ |

**Table 16 – 25-232849 F0– Comment**

| Id lab | Comment |
|---|---|
| 250700 | VDI 2444 (2020-03) LC-MS/MS |
| 250718 | Solvant d'extraction : Acétonitrile<br>Filtration puis évaporation avant reprise dans un mélange ACN/BSTFA (agent dérivatisant)<br>Méthode d'analyse : Impact Électronique en full scan avec m/z allant de 50 à 500 puis extraction de l'ion d'intérêt m/z 204 pour la quantification des 3 composés. Utilisation d'un étalon interne : acide succinique-d4. Gamme d'étalonnage réalisées sur filtres blancs et extraites comme un échantillon. Pour la détermination des LQ nous avons utilisé notre premier point de gamme comme point bas mais en réalité nous n'observons aucun pic. La LQ réelle est probablement beaucoup plus basse mais pas encore déterminée car encore en cours de développement. |
| 250740 | RESEARCH PROCEDURE (RB 34): Determination of biomass combustion markers and sugars using ion chromatography with pulsed amperometric detection |
| 250745 | Two methods were used for determination of anhydrosugars, one for levoglucosan and another for mannosan and galactosan. Samples were prepared on 10/01/2025. Date of analysis of levoglucosan was 10/01/2025, and date of mannosan and galactosan analysis was 10/03/2025. |
| 250751 | Technique d'analyse: IC-PAD, Méthode d'analyse : VDI 2444 |
| 250765 | no commends |
| 250766 | RAS |
| 250775 | Extractions aux US avec un mélange DCM/Acetone (1:1) |
| 250778 | Échantillons conservés à 5°C entre la réception et la préparation.<br>Extraction et l'analyse ont été réalisés par le même opérateur.<br>Le filtre n'a pas été extrait en totalité ; Un punch de 18mm de diamètre a été prélevé et extrait. |
| 250783 | méthode interne :<br>- agent de dérivation : MSTFA (N-méthyl-N-(triméthylsilyl)trifluoroacetamide) + 1% TCMS (triméthylchlorosilane),<br>- étalon interne de dérivation : méthyl-β-D-xylopyranoside<br>- étalon d'injection : phényldodécane (PDD)<br>Extraction aux ultrason après ajout de MeOH<br>Récupération de l'extrait méthanolique, concentration à l'ASE suivi d'une évaporation à sec à l'étuve, reprise avec pyridine anhydre + MSTFA + PDD.<br>Extrait à la pyridine étuvé à 80 °C pendant 2 heures<br>Analyse par GC/MS (mode SIM) |

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

Le Laboratoire Central de Surveillance de la Qualité de l'Air est un groupement d'intérêt scientifique créé en 2005 et constitué des laboratoires de l'IMT Nord Europe, de l'Ineris et du LNE. Depuis 1991, ses équipes d'experts mènent des études et des recherches sur la qualité de l'air, en appui au ministère chargé de l'environnement, et en étroite collaboration avec les Associations Agréées de Surveillance de la Qualité de l'Air (AASQA). Le LCSQA est désigné par le ministère pour assurer la coordination technique nationale du dispositif de surveillance de la qualité de l'air en France. Il est également l'organisme de référence requis par les directives européennes et ses missions sont définies par l'arrêté du 16 avril 2021 relatif au dispositif national de surveillance de la qualité de l'air ambiant.

L'objectif principal du LCSQA est ainsi de contribuer à l'amélioration continue du dispositif de surveillance en apportant un appui scientifique et technique au ministère et aux AASQA.

Ses travaux portent, d'une part, sur l'ensemble de la chaîne de surveillance : la qualité des mesures en air ambiant (du prélèvement à l'analyse), les inventaires d'émissions, la modélisation des concentrations et leur cartographie, la centralisation ainsi que le traitement et la mise à disposition des données, et d'autre part, sur les méthodes d'évaluation des plans d'actions. Cette action s'inscrit à la fois dans le cadre des réglementations nationales et européennes, et dans une démarche prospective visant à anticiper les besoins de la surveillance de demain.

**Direction du LCSQA**
Ineris - Parc technologique Alata 2 -
BP2 - F60550 Verneuil-en-Halatte
Tél : 03 44 55 69 16
ww.lcsqa.org