

Measurement guidelines for the ACTRIS Aerosol In-Situ variable

“Mass concentration of
particulate organics”

Module 1

Centre for Aerosol In-Situ – European Centre for Aerosol
Calibration and Characterization (CAIS-ECAC)

OrGanic Tracers and Aerosol Constituents - Calibration
Centre (OGTAC-CC)

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1 About this document

The pan-European aerosol, clouds and trace gas research infrastructure (ACTRIS) aims at producing high-quality data sets for short lived atmospheric constituents. One of the major missions is the harmonization of measurements techniques between the individual National facilities (NFs) to ensure the generation of comparable, traceable and quality assured and quality-controlled data.

In the framework of ACTRIS, OGTAC-CC as a Topical centre unit of the Centre for Aerosol In-Situ Measurement – European Center for Aerosol Calibration and Characterization (CAIS-ECAC) provides service and support for the analysis of organic tracers and aerosol constituents, from particles collected on filters or impactor substrates. The structure of the central facility with its sub-unit TCs is shown in Figure 1.

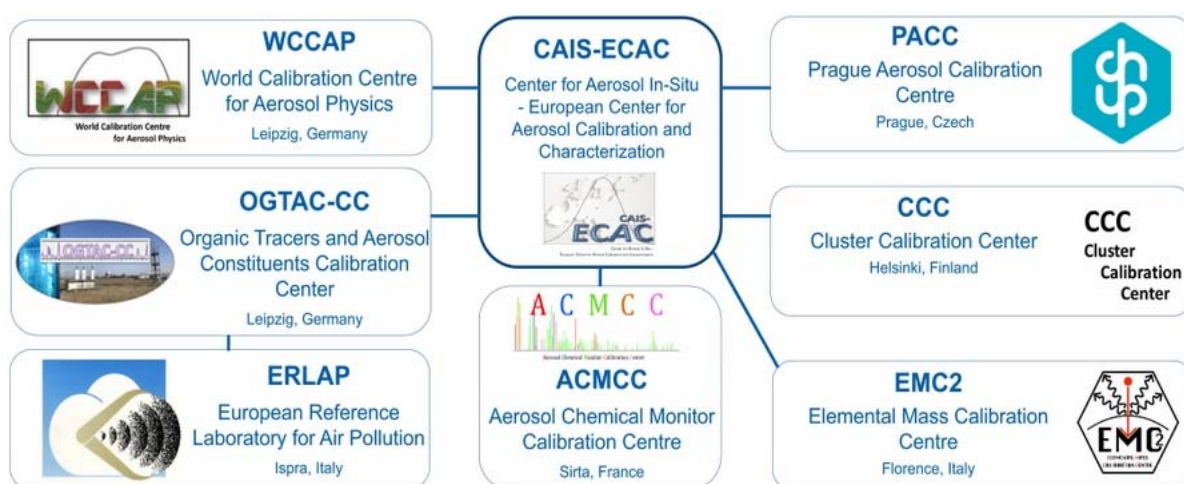


Figure 1: Structure of the CAIS-ECAC consortium, consisting of seven facilities (<https://www.actris-ecac.eu/>).

The document “Measurement Guidelines” considers guidelines and recommendations developed in close collaboration with the ACTRIS community based on former and current research. It provides an overview about general requirements (and recommendations) to be met if NFs aiming on the measurement of organic tracers and aerosol constituents as one of the additional variables within the aerosol in-situ framework. This document considers also guidelines and recommendations from other long-term observational networks, e.g., from WMO-GAW, EMEP EUSAAR and is based on the technical requirements documentation for particulate organics provided by OGTAC-CC.

The full measurement workflow comprises particle sampling at ACTRIS observatories, followed by sample transport, preparation, storage, until the final chemical analysis of the processed samples in the laboratory. At the current stage, OGTAC-CC supports a list of target analytes categorized in 7 modules, summarized in Table 1.

Table 1: List of modules summarizing the target compounds supported by OGTAC-CC.

Module	Target compounds	Module	Target compounds
1) Biomass burning	Levogluconan	6) SOA – nitroaromatics	2-Nitrophenol
	Mannosan		4-Nitrophenol
	Galactosan		2,4-Dinitrophenol
2) Primary biogenic	Erythritol		3-Nitrocatechol
	Arabitol		4-Nitrocatechol
	Glucose		2-Methyl-4-nitrophenol
	Fructose		4-Nitroguaiacol
	Mannose		4-Methyl-5-nitrocatechol
	Galactose		3-Methyl-5-nitrocatechol
	Mannitol	7) PAHs	Naphthalene
	Ergosterol		Acenaphthylene
3) Organic ions	Oxalate		Acenaphthene
	Formate		Fluorene
	Methane sulfonic acid		Phenanthrene
4) Biogenic SOA – acids	Terebic acid		Anthracene
	Vanillic acid		Fluoranthene
	Isovanillic acid		Pyrene
	MBTCA		Benzo[a]anthracene
	Norpinonic acid		Chrysene
	Pinic acid		Benzo[b]fluoranthene
	Pinonic acid		Benzo[k]fluoranthene
5) SOA – aldehydes	Vanillin		Benzo[a]pyrene
	Syringaldehyde		Indeno[1,2,3-cd]pyrene
	Coniferyl aldehyde		Benzo[g,h,i]perylene
	Acetosyringone (3,5-dimethoxy-4-hydroxyacetophenone)		Dibenzo[a,h]anthracene

The minimum requirement for the OGTAC-CC aerosol in-situ label is to provide on a regular base 2 target compounds from one or more modules. However, this does not limit the outcome of the analytical method. Since analytical methods for organic compounds can typically determine several of the listed analytes in parallel, the recommendation is to determine as standard the maximum outcome from one analytical procedure.

It should be mentioned that OGTAC-CC is open for new developments in both the field of sampling techniques and analytical systems to further improve the determination of particulate organics. New instrumentation will be thoroughly tested by the calibration centre together with the NF, by regular interlaboratory comparisons. Furthermore, it has to be noted, that the enrolment of further individual analytes not yet listed in the modules summarized in Table 1 will be evaluated by the calibration centre on an annual base.

OGTAC-CC will support and service all NFs within ACTRIS to ensure high data quality. To harmonize the full technical process between the NFs, it is necessary to define the measurement guidelines with respect to particle sampling, sample transfer steps and the laboratory protocol including sample preparation and the analytical procedure, summarized within the present document.

2 Data quality objectives – module 1

Data quality objectives (DQOs) define the type, quality and quantity required of primary data and derived parameters to yield information that can be used to support decisions. In particular, DQOs specify tolerable levels of uncertainty in the data, required completeness, comparability and representativeness based on the decisions to be made.¹

The DQOs in the present document are a compromise between the scientific need, the chemical (and physical) properties of interest of the target compounds and the existing instrumentation. The main general DQOs are summarized in the technical requirements documentation, whereas the specific DQOs for the target compounds from module 1 are listed in Table 2.

Table 2: Data Quality Objectives (DQOs) for the target compounds from module 1.

DQO	Description
Sampler	High- or Low-Volume sampler and cascade impactors according to DIN EN 12341:2023-10. ²
Field blank sampling	Minimum number shall correspond to 5% of the number of real measurement samples or minimum once a month.
Measurement time (data resolution)	It is recommended to measure on a continuous basis, minimum requirement is a weekly sample or one sample per week, exceptions: intensive measurement campaigns with a minimum requirement of 5 samples per campaign with two campaigns per year (ideally in two different seasons), need to be individually discussed with the calibration centre (CC).
Analytical instrument calibration	5 concentration levels, measured as triplicate
Maximum laboratory bias	20%
Limit of detection	Levolgucosan 0.05 mg L ⁻¹ Mannosan 0.05 mg L ⁻¹ Galactosan 0.10 mg L ⁻¹
Data coverage	≥ 80% of the planned yearly activity. Technical issues and maintenance to be done by the manufacturer are excluded.
Data provision	Beginning of the next year for the full year before: 1) submission to the CC - Deadline 31 st of March 2) submission to the DC - Deadline 31 st of May
Interlaboratory comparisons	Participation in biennial module-specific ILCs and stay within the maximum laboratory bias, the ILC data is discussed with the participants in a workshop after the data is assessed.

In any case, the NF must ensure that the operation of the technical part is organized in that way, that everything impacting the quality of the data is well monitored, documented and considered in the data control process.

3 Terms and definitions

For the purpose of this measurement guidelines documentation, the following terms and definitions apply:

Field blank sample

Filter/ substrate sample that is subjected to the entire sampling procedure (sample pretreatment, transfer, mounting in the sampling device) without switching on the sampling device and subsequently processed like a real measurement sample.

Laboratory blank sample

Unused filter/ substrate sample directly processed like a real measurement sample.

Method blank sample

Analysis without any filter/ substrate included in the sample to describe the analytical method performance.

Internal standard solution

A target compound solution with known concentration added to the sample before starting the analytical procedure.

Calibration

Operation performed on a measuring instrument or a measuring system that, under specified

conditions: 1. establishes a relation between the values with measurement uncertainties provided by measurement standards and corresponding indications with associated measurement uncertainties and 2. uses this information to establish a relation for obtaining a measurement result from an indication.³

Check standard

Is a calibration standard of a medium concentration to be measured as a control within an appropriate interval (e.g., every ten samples) to verify the stability of the instrument, the accuracy of the calibration and the overall system performance.

Limit of detection (LOD)

The Limit of Detection (LOD) is the lowest concentration of an analyte that can be reliably distinguished from the background noise or a blank sample with a specific level of confidence.

PM₁₀; PM_{2.5}; PM₁

Means particulate matter which passes through a size-selective inlet as defined in the reference method for the sampling and measurement of PM₁₀, EN 12341², with a 50% efficiency cut-off at 10 µm, 2.5 µm or 1 µm aerodynamic diameter, respectively.

4 Symbols and abbreviations

ACTRIS	Aerosol, Clouds and Trace Gases Research Infrastructure
CAIS-ECAC	Centre for Aerosol In-Situ measurement – European Center for Aerosol Calibration and Characterization
CC	Calibration Centre
DQO	Data Quality Objective
EMEP	European Monitoring and Evaluation Programme
EPA	Environmental Protection Agency
EUSAAR	European Supersites for Atmospheric Aerosol Research
GC-MS	Gas Chromatography – Mass Spectrometry
HPAEC-PAD	High Performance Anion Exchange Chromatography – Pulsed Amperometric Detection
LOD	Limit of Detection
NF	National Facility
OGTAC-CC	Organic Tracers and Aerosol Constituents – Calibration Centre
PAH	Polycyclic Aromatic Hydrocarbons
PM	Particulate Matter
SOA	Secondary Organic Aerosol
SOP	Standard Operation Procedure
WMO-GAW	World Meteorological Organization – Global Atmosphere Watch

5 Particle sampling protocol

This section is module independent and describes the general particle sampling protocol that need to be considered by ACTRIS observatories in order to provide particulate organics data within ACTRIS. After a brief description of general aerosol particle sampling recommendations, further details regarding the full aerosol particle sampling procedure, e.g. on particle samplers, filter/ substrate material, filter/ substrate transfer and storage and system checks are provided.

5.1 General considerations

All information about general aerosol sampling, aerosol inlet design under consideration of the individual NF circumstances and aerosol conditioning are described in the overall document “**ACTRIS Standard Procedures for In-Situ Aerosol Sampling, Measurements, and Analyses at ACTRIS Observatories**” (<https://www.actris-ecac.eu/actris-gaw-recommendation-documents.html> (03.06.2026)⁴) also referencing the WMO/GAW “Aerosol Measurement Procedures, Guidelines and Recommendations”.⁵

5.2 Documentation of the procedure

The technical requirements documentation together with the module specific measurement guideline document are the base for the development of the NF individual standard operation procedure (SOP). In the first part, the SOP contains the NF individual hardware and procedure information about the particle sampling protocol. An example of such a document is given in the Annex section 9.1.

5.3 Handling of aerosol particle sampling devices

The filter-based collection of aerosol particles can be done within the PM₁₀, PM_{2.5} or PM₁ size fraction, either by High-volume or low-volume sampling devices. Sampling on filters should be done in accordance with DIN EN 13241:2023-10.² Handling of the particle sampling device should be done following the manufacturer’s instructions. In general, two types of filter material, Teflon filters and quartz fibre filters are recommended following the WMO/GAW guidelines.⁵ Depending on the measurement, glass fibre filters are also applicable.⁶⁻⁸ If a station uses other types of filter it is needed to proof the applicability by, e.g. parallel measurements for a certain timeframe that has to be discussed individually with the CC. Typical filter sizes are 47 mm and 150 mm, respectively.

Cascade impactor sampling should in general follow the same requirements as the particle filter samplers. Impactor substrates are typically aluminum foil, Teflon filters and polycarbonate filters depending on the purpose of sampling.⁵

The general application of a denuder system prior the aerosol particle sample inlet is not requested for the determination of the particulate organic marker compounds. The NF only has to document in detail if a denuder system is applied, for example if sampling for the determination of particulate OC/EC⁹ following the EUSAAR sampling train is done in parallel.

Additional site-specific actions regarding the particle sampling protocol, e.g. greasing the impaction plate is to be documented.

5.4 Sample pretreatment, storage and transport

In general, it is highly recommended to minimize all filter handling steps, transport of samples and keep the storage duration as short as possible, at the measurement site and at the laboratory. If any filter

manipulation at the measurement site is needed use always new gloves (anti-static and powder free) and cleaned tweezers in a suitable clean workplace.

Sample pretreatment:

- Conditions for filter or impactor substrate pretreatment, like preheating temperature (°C) and time (h) should be documented
- Example of a routine sample pretreatment procedure is provided in section 9.1

Filter/ Impactor substrate weighing:

Weighing of filter/ impactor substrate samples is not required for the determination of particulate organics. However, the CC highly encourage the observatories to include the particulate mass determination within their measurement procedure.

Sample storage:

- Should be done protected from external contamination and in the dark, e.g. in light protected boxes or wrapped in aluminum foils, in zip-lock bags, clean glass petri dishes or similar containers
- Unsampld filters or impactor substrates can be stored at the measurement site as transported without a maximum storage time if an adequate field blank measurement is available, the CC recommends not to store the unsampled filter longer than 2 months²
- After sampling, samples should be removed from the sampling device and stored in a freezer, maximum sample storage time and conditions until further sample preparation in the laboratory is set either following literature values or individual tests performed at the CC
- Recommended maximum storage time of filter samples for module 1 is 84 days in a freezer based on OGAC-CC internal measurements (maximum up to 12 months¹⁰)
- Filter sample storage should be done as single filter, appropriate and carefully folding the filter is allowed but not recommended
- Impactor substrates are not permitted to be stored folded

Sample transport:

- Should be done protected from external contamination and in the dark, e.g. in light protected boxes or wrapped in aluminum foils, in zip-lock bags, clean glass petri dishes or similar containers
- Use sample transport boxes with the option of cooling to avoid excessive heating, e.g. by using cooling packs to prevent loss of volatile and semi-volatile materials during transport, temperature should not exceed 23°C (effects of condensation within the cooled transport box can be avoided by packing the samples in plastic bags or by sealing them)
- Recommendation to ship for daily sampling a one-week supply from the laboratory to the site, and vice versa, once every week
- Exceptions because of particular site conditions are possible but need to be discussed with the CC
- NFs are free to decide whether filters are transported in the filter holder to the measurement site and back to the lab or remove the filter on-site and transfer only the filter as described before

- Filter sample transfer should be done as single filter, appropriate and carefully folding the filter is allowed but not recommended
- Impactor substrates are not permitted to be transported folded

For ACTRIS observatories with special circumstances, e.g. very remote sites which cannot or only partly fulfill these requirements, all operational tasks must be organized in such a way that the sample quality does not suffer (e.g. cooling packages/dry ice for sample storage) and the full procedure is documented in detail to the calibration centre.

5.5 Control, cleaning and maintenance

Each individual sampling system should be controlled, cleaned and maintained in accordance with the manufacturer's specifications. The following parameters should be checked:

- Flow rate calibration once a year
- Pump maintenance yearly
- Sampling head/ inlet cleaning monthly or if an obvious error occurs
- Sensor check (temperature, pressure, humidity) every three months
- Sensor calibration yearly or if an obvious error occurs

More details on the performance evaluation of the sampler is given in DIN EN 12341.² Additional site-specific actions for instance shorter time intervals of cleaning procedures during the pollen season is to be documented. In general, as discussed before, for ACTRIS observatories with special circumstances, e.g. very remote sites all operational tasks must be organized in such a way that the sample quality does not suffer and the full procedure is documented in detail to the calibration centre.

5.6 Data resolution, coverage and provision

It is recommended to measure on a continuous basis. The minimum requirement is a weekly sample or one sample per week. In general, the time resolution should be according to the general scientific questions at the NF and of ACTRIS. Furthermore, sufficient aerosol mass for target compound detectability by the subsequent analytical method needs to be considered and therefore exceptions are allowed but need to be individually discussed with and approved by the CC.

A further exception could be a special intensive measurement campaign, here e.g. minimum 5 samples per campaign but then minimum two of these campaigns per year, ideally in two different seasons.

Minimum data coverage is set to 80% of the planned yearly activity. Technical issues and maintenance to be done by the manufacturer are excluded. Data provision (processing and submission) should be done minimum once a year. Data should be submitted beginning of the next year for the full year before:

- 1) submission to the CC - Deadline 31st of March
- 2) submission to the DC - Deadline 31st of May

Offline data submission procedure will be made available latest until 31st of May 2027 to enable the first NFs to upload the measurement data from module 1 for the year 2026.

6 Laboratory analysis protocol – module 1

General remarks

The recommended laboratory analysis protocol depends on the selected organic marker compounds separated in the modules presented in Table 1. The following subsections provide the detailed recommended and supported laboratory analysis protocol for the target compounds from the biomass burning module 1.

6.1 Biomass Burning module

Table 3 summarizes the recommended and supported analytical technique and the individual DQOs set by the CC for the compounds from the Biomass Burning module 1.

Table 3: Key facts of the laboratory analysis protocol and individual DQOs for the Biomass Burning module 1.

Target compound	CAS number	Recommended and supported analytical technique	Maximum laboratory bias	Analytical limit of detection [mg L ⁻¹]
Levoglucosan	498-07-7	HPAEC-PAD	20%	0.05
Mannosan	14168-65-1		20%	0.05
Galactosan	644-76-8		20%	0.1

The protocol specifies in detail the recommended sample preparation procedure and the application of the High-Performance-Anion-Exchange-Chromatography coupled to Pulsed-Aperometric-Detection (HPAEC-PAD) as recommended and supported analytical technique for the determination of levoglucosan, mannosan and galactosan in particulate matter collected on filters or impactor substrates. The entire analytical protocol considers regulations and recommendations from DIN EN 12341:2023², VDI 2444⁷ and the DIN CEN/TS 18044.⁶

6.1.1 Documentation

The technical requirements documentation together with the module specific measurement guideline document are the base for the development of the NF individual standard operation procedure (SOP). Besides the information about the particle sampling protocol (see section 5.2) the SOP contains in its second part the NF individual hardware and protocol information about the applied sample preparation and analytical method approved by the QA/QC procedures of the CC. An example of a laboratory analysis protocol and an overview about the requested data that need to be provided as metadata to the ACTRIS DC for each uploaded measurement result is summarized in the Annex (section 9.2 and 9.3) of the present document.

6.1.2 Sample preparation

The filter spot(s) or a defined area of substrate material is introduced into the extraction vessel and a defined volume of ultrapure water is added. Size and number of filter spots (filter area in total) should allow reliable measurements of the target compounds within the applied calibration range. Extraction

is done using a laboratory shaker or in an ultrasonic bath for a dedicated time. The extract is either filtered or centrifuged.

It is recommended to proceed with the laboratory analysis subsequently the sample preparation step. In case a temporary storage prior analysis is needed, it is required to store the extracts in a freezer. Storage time of extracts should be kept as short as possible but not exceed 112 days, based on OGTAC-CC internal measurements.

6.1.3 Analytical procedure

Analytical equipment for the HPAEC-PAD:

- Pump
- Autosampler (optional)
- Vacuum degasser or eluents under N₂ bubbling
- Precolumn (optional)
- Carbohydrate column
- Pulsed amperometric detector (with gold electrode)

Chemicals and accessories:

The eluent preparation should follow the instructions of the manufacturer of the instrument and the separation column, respectively. Further information regarding eluent preparation, technical considerations or trouble shooting can be found in the DIN CEN/TS 18044.⁶

- Sodium hydroxide as eluent (mobile phase)
- Ultrapure water ($R \geq 18.2 \text{ M}\Omega \text{ cm}$)
- Pure standard substances (see Table 3)

The analytical instrument is set up and operated according to the manufacturer's instructions.⁷

Calibration

The stock solution is prepared in a range of $400 \mu\text{g g}^{-1}$ to about 1 mg g^{-1} by a gravimetric procedure weighing the pure standard substance (levoglucosan, mannosan and galactosan) in a suitable vessel and add an adequate volume of ultrapure water. The stock solution can be kept in a refrigerator or freezer for at least one year, provided that it is protected from attack by microorganism (e.g. by cleaning the vessels with hot ultrapure water ($T > 90 \text{ }^{\circ}\text{C}$)). The respective calibration solutions are prepared timely to the measurement by diluting the stock solution with ultrapure water to produce concentrations that cover the entire working range. It is generally recommended to use commercial standard stock solutions or multi-component standards with certificate of analysis (e.g., from Merck, Sigma-Aldrich, NIST, etc). In any case, NFs must document the preparation of calibration solution comprehensively to ensure that calculation or dilution errors are traceable:

- Date of preparation
- Reagent type, purity & manufacturer and batch number
- All masses and/or volumes
- Operators name

First-time instrument calibration or after a longer standby-time it is recommended to apply 5 concentration levels for calibration (should be relatively evenly distributed and include a concentration close to the LOD of the method), measured as triplicate. The final calibration function is calculated from the obtained signal areas and the corresponding concentrations of the calibration solutions. The type of the calibration function, e.g. linear (expected for the present system) is examined based on the obtained regression values and updated for every series of analysis. Obtained sample concentrations that are more than 10% out of the prepared calibration range must be diluted and reanalyzed.

Sequence design

Before analysis, ion chromatographs should be equilibrated at least 30 min with the respective eluent concentration and stability of pump pressure and baseline conductivity should be checked. A method blank must be analyzed at the beginning and regularly within each sequence. The analytical sequence continues with the analysis of calibration solutions covering the entire range of concentrations expected (from lowest to highest concentration) followed by a simple water blank to check water background and potential carry over contamination from previous injections. The samples are subsequently analyzed and the chromatograms recorded. A single calibration standard (check standard) of a medium concentration should be measured as a control minimum every 20 samples. Results must be within 15% of the expected value. If results exceed this criterion, the cause must be investigated and the sample set must be reanalyzed. The entire set of calibration solutions should be repeated after a sample batch of maximum 50 samples, single measurements are sufficient. Field blanks are treated and handled as the real samples. Laboratory blanks are further control options, to check for unexpected impacts of the sample preparation procedure. An example sequence design is shown in Table 4.

Table 4: Example sequence design HPAEC-PAD.

Position	Vial	Description
1	Instrument blank	Equilibration and instrument (baseline) check
2	Instrument blank	
3	Water blank	
4	Water blank	
5	Cal std 1	Calibration
6	Cal std 2	
7	Cal std 3	
8	Cal std 4	
9	Cal std 5	
10	Water blank	Control
11	Sample 1	Samples
12	Sample 2	
13	Sample 3	
14	Sample 4	
15	Sample 5	
16	Sample 6	
17	Method blank 1	
18	Sample 7	
19	Sample 8	
20	Sample 9	
21	Sample 10	
22	Field blank	
23	Water blank	Control

24	Check standard	
25	Sample 11	Samples
26	Sample 12	
27	Sample 13	
28	Sample 14	
29	Sample 15	
30	Sample 16	
31	Method blank 2	
32	Laboratory blank	Control
33	etc...	

Structuring sequences in these patterns has several advantages, since system stability can be monitored easily and potential sources of problems can be isolated.

Method description

Adequate chromatographic separation of levoglucosan, mannosan and galactosan shall be ensured. A detailed description on the application of the HPAEC-PAD for the determination the compounds from the Biomass Burning module 1: levoglucosan, mannosan and galactosan in PM is given in the Annex 9.2 and subsections.

Data processing and quantification

After the measurements, a visual inspection of all chromatograms is essential to ensure quality. The baseline should be flat, smooth, and free of drift, spikes, or noise. A drifting baseline may indicate column degradation, eluent issues, suppressor malfunction, or contamination. Peaks should be symmetrical (Gaussian-like). Tailing or fronting may suggest column overload, chemical interactions, or matrix effects. Finally, the reproducibility of retention times and the consistency of peak integration should be verified.

The recorded raw data files are usually processed by the analytical software to produce the result files. The integrated peak areas of each compound, identified by the corresponding retention time, are converted into solution concentrations by using the pertinent calibration functions. Linear calibration is the default and sufficient for most applications when the response is proportional to concentration. Quadratic calibration is used only when non-linearity is observed, typically at high concentrations due to detector saturation, column overload, or suppressor limitations. In both cases, the function should normally not be forced through zero, but calculated with the offset. Since the absolute deviation from the regression tends to be greater at higher concentrations, where the response is larger, the higher-concentration points exert a disproportionate influence on the fit. As a result, the regression function is often pulled toward these points, which can lead to insufficient fit at the lower end of the calibration range. It is therefore recommended to use weighted calibration, to give greater importance (weight) to data points with lower response (lower concentration). The most common weighting schemes are: $1/\text{Response}$ ($1/x$) and $1/\text{Response}^2$ ($1/x^2$).

The final atmospheric concentration (e.g. ng m^{-3}) is derived by these values and the respective amount of extraction solvent, filter area and sampling volume.

Figures of merit (laboratory bias and analytical limit of detection)

Inter-laboratory bias is a measure of laboratory comparability and is used as DQO for the assessment of the NF data quality. The laboratory bias is calculated using the following equation (1)¹¹:

$$Bias = \frac{(C_{lab} - MedianC)}{MedianC} \quad (1)$$

With C_{lab} is the laboratory's reported concentration and $MedianC$ is the median concentration of all laboratories.

The LOD is typically described as the concentration of three times the blank value scatter or the blank value scatter multiplied by a statistically calculated factor (e.g. Student's t -test). However, since the recommended ion chromatographic method mostly do not show blank values for the target compounds and the signal to noise ratio cannot be determined because of technical reasons the LOD has to be derived through the following procedure: A real sample extract with a very low target compound concentration is repeatedly analyzed (e.g. ten times) and the corresponding standard deviation of the peak area calculated. The smallest reliably detectable peak area is then calculated according to equation (2):

$$\chi_{NG} = t_f \cdot s_F \cdot \sqrt{1 + \frac{1}{n}} \quad (2)$$

where

- χ_{NG} smallest peak area that can be reliably integrated
- t_f quantile of t-distribution for one-sided consideration (1,812 for $n = 10$)
- s_F standard deviation of the peak area for the target compound
- n number of repeated measurements

The LOD in final atmospheric concentration (e.g. ng m^{-3}) is derived by these values and the respective amount of extraction solvent, filter area and sampling volume. Data obtained below the LOD is not going to be reported. All quality control and sample results have to be verified by double checking through the NF PI in charge of the ACTRIS component before submission to the CC.

Interferences and quality management

Potential interferences and other issues impacting the quality and quantity of the discussed measurements are presented in DIN CEN/TS 18044.⁶

General QA/QC measures are summarized in section 7.

7 Data quality control and quality assurance

The general main QA/QC measure for the determination of particulate organics is to perform intercomparison studies. Such regular interlaboratory comparisons (ILCs) enable the CC to assess the applicability of the NF individual SOP according to the ACTRIS requirements. NFs have to participate in the ILCs biennially for each module data is uploaded to the DC. A detailed description of the ILC procedure is given in the general technical requirements documentation.

7.1 Particle sampling protocol

Blank values (field, laboratory and method) are to be determined on a regular basis. The minimum number of field blank samples shall correspond to 5% of the number of real measurement samples or minimum once a month.

Hardware QA/QC involves controlling and maintaining the particle sampling device in accordance with the manufacturer's specifications. The following parameters should be checked:

- Flow rate calibration once a year
- Pump maintenance yearly
- Sampling head/ inlet cleaning monthly or if an obvious error occurs
- Sensor check (temperature, pressure, humidity) every three months
- Sensor calibration yearly or if an obvious error occurs

Adequate training and instruction of site operators is of great importance for the data quality. In particular all new operators should receive their instructions directly from the PI responsible for the performance of the station. The training should take place at the actual measurement station.

In addition, OGTAC-CC is providing biannual training courses on general particle sampling procedures. Operators of NFs planning to measure particulate organics should consider the participation as a part of their QA/QC measures.

7.2 Laboratory analysis protocol

All steps must be documented precisely in an electronic or an analog laboratory book. For the latter one, only a ballpoint pen should be used for handwritten notes in order to ensure traceability (GLP). Changes in a laboratory notebook must be traceable, incorrect information must be clearly crossed out and marked (e.g. "typing error; changed on DD.MM.YYYY; person XY"). All laboratory journals must be archived. The applied chemical containers are to be labeled with the expiry date; the expiry date specified by the manufacturer (if available) is to be observed. The preparation date, exact designation and concentrations, as well as the responsible employee must be noted on all stock and standard solutions used for the system calibration.

Laboratory and method blank samples are to be analyzed for each set/sequence of samples as described before. In addition to that, a recommended but not required QC, is another control standard containing a pure compound that is not on the list of targets but still detectable by the applied analytical procedure

that can be used to examine on a regular basis the general stability of the analytical system by evaluating the retention time and the expected signal intensity. More optional quality assurance and quality control actions are listed in DIN CEN/TS 18044.⁶

Preventive maintenance of the entire analytical system has to be done on an annual basis according to the manufacturer's instructions.

Laboratory personnel should be properly trained by the PI of the NF in sample preparation and analytical work before they are allowed to carry out the routine analyses. In addition, OGTAC-CC is providing biannually training courses on sample preparation techniques and subsequent analytical procedures to measure particulate organics. Operators of NFs should consider the participation as a part of their QA/QC measures.

7.3 Data submission and data products

Data provision (processing and submission) should be done minimum once a year. Data should be submitted beginning of the next year for the full year before:

- 1) submission to the CC - Deadline 31st of March
- 2) submission to the DC - Deadline 31st of May

A standardized approach to the offline data workflow is still under discussion at the European level, and its implementation has therefore been delayed. The actual work on the tasks of the responsible TC units—which have not yet been fully defined (e.g., the development of machine-readable device databases) in collaboration with the data center requires more time and is therefore a dedicated work package activity within the recently started ACTRIS NEXT project. However, to make progress on the labeling of AIS NFs with regard to the mass concentration of particulate organics an interim offline data submission procedure will be made available latest until 31st of May 2027 to enable the first NFs to upload the measurement data from module 1 for the year 2026.

8 References

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4. Center for Aerosol In-Situ Measurement European Center for Aerosol Calibration and Characterization (CAIS-ECAC); *ACTRIS Standard Procedures for In-Situ Aerosol Sampling, Measurements, and Analyses at ACTRIS Observatories*; ACTRIS-ERIC Version 1.0; 2024.
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6. Engl. VDI/DIN-Kommission Reinhaltung der Luft (KRdL) - Normenausschuss; *Ambient air - Determination of the concentration of levoglucosan - Chromatographic method*; DIN CENT/TS 18044; 2024; p 46.
7. VDI/DIN-Kommission Reinhaltung der Luft (KRdL) - Normenausschuss; *Ambient air - Measurement of levoglucosan - Chromatographic method*; VDI 2444; 2020; p 34.
8. EMEP/CCC-Report 1/95; *EMEP manual for sampling and chemical analysis (Revision 2014)*; Norwegian Institute for Air Research: 1996.
9. VDI/DIN-Kommission Reinhaltung der Luft (KRdL) - Normenausschuss; *Ambient air - Measurement of elemental carbon (EC) and organic carbon (OC) collected on filters*; EN 16909:2017; 2017; p 63.
10. *European Committee for Standardization: Ambient air - Determination of the concentration of levoglucosan - Chromatographic method*; CEN/TS 18044:2024 CEN 2024; p 46.
11. Karin Acker, et al., *World Meteorological Organization, Global Atmosphere Watch 2004*,
12. VDI/DIN-Kommission Reinhaltung der Luft (KRdL) - Normenausschuss; *Particulate matter measurement - Gravimetric determination of the mass concentration of suspended particulate matter in ambient air - General principles*; 2024; p 13.

9 Annex

9.1 Example of a particle sampling protocol

The following section provides an example of a particle sampling protocol for sampling on a 150 mm quartz fibre filter using a High-volume Digitel sampler with PM10 inlet according to the OGTAC-CC measurement guidelines for module 1. It is the first part of the NF individual SOP to be set up.

Pretreatment at the laboratory:

- Quartz fibre filter order in batches (MK360, Ahlstrom-Munksjö), 1% of the filters from each batch is restored for blank experiments
- Preheating according to VDI 2463¹² at 105°C for 24 h, hot filters are transferred to a compartment drier to cool down to laboratory ambient temperature
- Equilibration for 72 h in a climate-controlled weighing room (if weighing is part of the sampling protocol), temperature and relative humidity are documented (typically within a range of 20.5-23.5°C and 25-60% relative humidity)
- Each step is documented with date and time

Weighing (optional part of the sampling protocol):

- Temperature and relative humidity (typically within a range of 20.5-23.5°C and 25-60% relative humidity) documented during weighing in an automated balance (METTLER AT261 DeltaRange)
- Filter samples transferred in light protected aluminum container, aluminum containers are precleaned using a laboratory dishwasher working with ultrapure water, if containers are new precleaning is done with neutracon, ethanol and ultrapure water (three times)
- Pretreated filters are stored within the aluminum containers in the weighing room until transport to the measurement site, maximum number of filter samples prepared in advance of the sampling is about 30 (one month)

Sample transport:

- Filter samples are placed within the respective filter holders for transport, each filter holder is wrapped in aluminum foil (marked to each sample)
- The wrapped filter holders are stacked in a small Zarges box to be transported via car to the measurement site (about one-hour drive)
- Transport 7 new filter samples once a week

Action at measurement site:

- Filter holders with filter samples unwrapped in a clean workplace and stored within a plastic container with dedicated holder options assisting the safe transfer to the Digitel high-volume filter sampler at the point of measurement (sampler is placed outside)
- Used aluminum foil to wrap the filter samples in the filter holders is stored and used for the transport back after sampling exactly for the identical sample

Action at the point of measurement:

- All 7 filter samples with filter holders inserted in the high-volume sampler at ambient conditions, samples have to be protected from rain
- Every week exchange of 7 sampled filters with 7 fresh pretreated filter samples, however, the sampler contains for safety reasons always 15 samples in total
- → Maximum time of filter samples within the sampler is 14 days
- → Maximum time of filter samples within the sampler after sampling is 7 days
- Sampling conditions: each filter 24 h at a flow rate of 500 L min⁻¹
- Details about sampling site, instrument identifiers and further operator information are part of the general metadata that need to be provided (see Annex 0)
- Sampled filters with filter holders are transferred back to the clean workplace at the measurement site again using the plastic container

Action at measurement site:

- Sampled filters and filter holders are wrapped in aluminum (same as the way there) that was stored folded at the clean workplace
- 7 wrapped filter holders are stacked in the small Zarges box for transport to the laboratory

Sample transport:

- The Zarges box including the sampled filters is handled and transported with care and attention of keeping the temperature within the box below 23°C (about one-hour drive in an air-conditioned car).

Action at the laboratory:

- Samples are typically stored as they arrive at the laboratory in a fridge at 5°C over night
- The next day, sampled filters are removed from the filter holders and transferred back in the previously used aluminum containers and stored in a freezer at -20°C until further action

Weighing (optional part of the sampling protocol):

- Aluminum containers out of the freezer and stored within the climate-controlled weighing room for about 2 h
- Sampled filters removed and placed in dedicated holders for 72 h of equilibration
- Weighing by using an automated balance (METTLER AT261 DeltaRange)
- Temperature and relative humidity documented, two times a (weighing-) day a reference filter is weighed 10 times to derive the standard deviation
- Note: accidentally collected small insects are removed before weighing, other obvious issues are documented via pictures in the protocol
- Filters are again stored within the aluminum containers in a freezer at -20°C until further processing, in the case the next sample preparation steps won't be shortly after the weighing the filters are not transferred to the aluminum containers but wrapped only in aluminum foil without folding and then stored in a freezer at -20°C (to save storage space)

9.2 Application example HPAEC-PAD for the compounds of the Biomass Burning module 1: levoglucosan, mannosan and galactosan

The following section provides an example of a laboratory analysis protocol using HPAEC-PAD for the compounds of the Biomass Burning module 1: levoglucosan, mannosan and galactosan according to the OGTAC-CC measurement guidelines. It is the second part of the NF individual SOP to be set up.

9.2.1 Apparatus

ThermoFisher Scientific ICS-6000

- DIONEX ICS-6000 DC with pulsed amperometric detection on an Au, Ag/AgCl electrode
- DIONEX ICS-6000 (SP) with single quaternary pump
- DIONEX ICS-6000 AS-AP autosampler
- Guard column Dionex CarboPac MG1
- Separation column Dionex CarboPac MA1
- PC with Chromeleon 7.2.10 ES

9.2.2 Operating parameters

- Column compartment temperature: 25 °C
- Sample consumption per analysis: 75 µL
- Sample loop: 25 µL
- Eluent flow rate: 0.4 mL min⁻¹
- Duration of chromatogram: 65 min
- Total analysis time: 65 min
- Separation column: Dionex CarboPac MA1, 7.5 µm, 4×250 mm
- Guard column: Dionex CarboPac MG1, 7.5 µm, 4×50 mm

9.2.3 Chemicals and accessories

- For sampling: quartz fibre filter, diameter 150 mm
- Punch support (e.g. 14 mm PVC support)
- Filter aliquot diameter 2x14 mm
- Gloves (e.g. Labsolute)
- Goggles
- Spatula
- Antistatic plastic forceps
- Precision wipes (e.g., Kimtech Science, item no. 7552)
- Bottle top dispenser, 2 ml (2 g) for dispensing ultrapure water
- Plastic bottles 30 ml, HDPE, with leak-proof caps
- 1.5 ml Eppendorf Safe-Lock tubes
- 1 ml disposable syringe

- 0.45 µm syringe filters (13 mm; IC-Acrodisc; PALL; item no.: 4685)
- 600 µl polypropylene sample vials (e.g., item no.: 301311; CS-Chromatographie)
- Caps with split septa for sample vials (item no.: 301396-Blue; CS-Chromatographie)
- Suitable sample racks
- Storage boxes for storing extracted samples (item no.: 2-7209; neoLab)
- Analytical balance (220 g max used)
- Eppendorf pipettes (e.g., 100 - 1000 µl, 1–10 ml) including suitable pipette tips
- Shaker (type KL-2 m-UHR 1–120; Edmund Bühler Laboratory Equipment)
- 2 l or 4 l chemical bottle
- Ultrapure water ($R \geq 18.2 \text{ M}\Omega \text{ cm}$; $\text{TOC} < 10 \text{ ppb}$)
- Sodium hydroxide solution, 50% to 52% in water, eluent for IC, carbonate-free
- Chemicals for standard solutions (analytical grade): myo-Inositol, meso-Erythritol, 1,6-Anhydro- β -glucopyranose, D-(+)-Arabitol, 1,6-Anhydro- β -D-Mannopyranose, D-Mannitol, D-(+)-Mannose, 1,6-Anhydro- β -D-Galactopyranose, D-(+)-Glucose, D-(+)-Galactose, D-(–)-Fructose, D (+)-Sucrose
- Helium (premium 5.2)

9.2.4 Procedure

9.2.4.1 Calibration standards

To prepare a stock solution, 30 mg of each standard are weighed into a 30 ml HDPE bottle. The exact weight of the substance is documented and dissolved in ultrapure water to achieve a target concentration of 1 mg mL^{-1} . The volume of added ultrapure water is also documented. The entire procedure is repeated for all substances.

A multicomponent stock solution is prepared with the association of the different compound standard solutions to correspond to the samples' relative concentrations, and stored frozen ($-20 \text{ }^{\circ}\text{C}$). Preparation is done for a concentration range of 0.1 to 10 mg L^{-1} according to the following scheme:

- For 10 mg L^{-1} : 300 µl of each stock solution + ultrapure water to a total volume of 30 ml
- For 5 mg L^{-1} : 150 µl of each stock solution + ultrapure water to a total volume of 30 ml
- For 2.5 mg L^{-1} : 75 µl of each stock solution + ultrapure water to a total volume of 30 ml
- For 1.25 mg L^{-1} : 37.5 µl of each stock solution + ultrapure water to a total volume of 30 ml
- For 1.0 mg L^{-1} : 30 µl of each stock solution + ultrapure water to a total volume of 30 ml
- For 0.6 mg L^{-1} : 1800 µl of 10 mg L^{-1} solution + ultrapure water to a total volume of 30 ml
- For 0.3 mg L^{-1} : 900 µl of 10 mg L^{-1} solution + ultrapure water to a total volume of 30 ml
- For 0.2 mg L^{-1} : 600 µl of 10 mg L^{-1} solution + ultrapure water to a total volume of 30 ml
- For 0.1 mg L^{-1} : 300 µl of 10 mg L^{-1} solution + ultrapure water to a total volume of 30 ml

0.5 ml of each calibration solution is transferred into separate vials for analysis using a 1 ml Eppendorf pipette.

9.2.4.2 Eluent preparation

The eluents for chromatographic separation are freshly prepared on the basis of necessity. Eluent bottles are rinsed with ultrapure water (minimum 3 times) before preparation. The eluent bottle used

for ultrapure water is filled completely and degassed by bubbling with helium for 15 minutes. To prepare the 1 M NaOH eluent, ultrapure water is degassed for 20 min by bubbling with helium and subsequent NaOH added to the bottle (in the fume hood). Finally, the completed eluent is degassed for 5 min with helium.

9.2.4.3 Sample preparation

The filter aliquots are transferred in 1.5 ml Eppendorf-tubes. 1 ml of ultrapure water is added to the filter from the Bottle top dispenser. The cap is closed and the sample transferred in a suitable sample rack on a shaker. After shaking for 2 hours at 420 rpm, the sample is filtered via a 0.45 µm syringe filter directly into a 600 µl sample vial. The prepared sample vials are either placed in the autosampler or stored frozen until measurement. The rest of the extract is kept frozen until reanalysis or pending other kind of analysis.

9.2.4.4 Preparation of instrumentation

All eluent bottles are purged for 5 minutes with a flow of 6 mL min⁻¹. Then the pump is started with 0.4 mL min⁻¹ flow and a starting gradient of sodium hydroxide 50% (A) and ultrapure water 50% (B). Then all compartments are turned on and the system run in these conditions for 2 h for equilibration.

The AS AP autosampler is equipped with a refrigerated compartment for vials set at 10 °C for better preservation of organic compounds, and to avoid microorganisms' proliferation. 25 µl of standard or sample is injected in the instrument.

9.2.4.5 Separation and detection conditions

A Dionex CarboPac MA1, 7.5 µm, 4×250 mm and a Dionex CarboPac MG1, 7.5 µm, 4×50 mm guard column are used at 25 °C for separation and column preservation, respectively. The analytical run is isocratic with 50% of sodium hydroxide (1000 mM) and 50 % water, at 0.4 mL min⁻¹. Pulsed amperometric detection is carried out with a gold electrode at 25 °C.

Total run time per sample is 65 min at 0.4 mL min⁻¹ with eluent proportions as follow:

- 0 min to 20 min: 50% water and 50% NaOH
- 20 min to 35 min: increasing to 65% NaOH and 35% water
- 35 min to 50 min: 65% NaOH and 35% water
- 50 min to 51 min: decreasing to 50% water and 50% NaOH
- 51 min to 65 min: 50% water and 50% NaOH

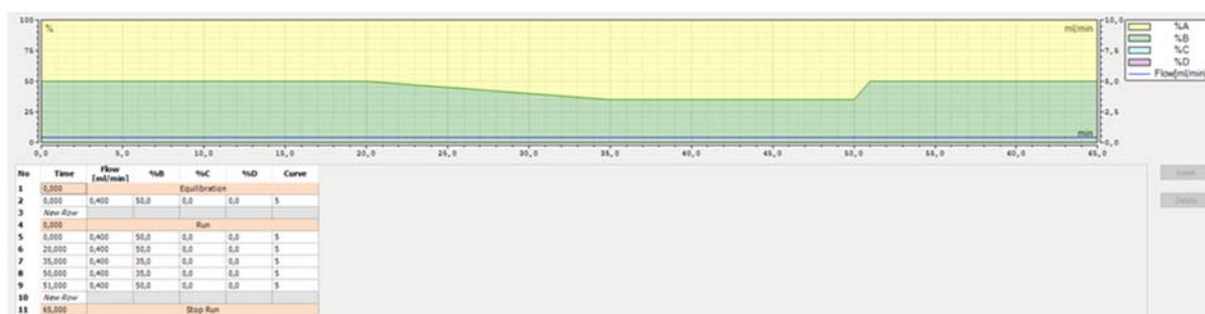


Figure 2: Gradient elution (screenshot).

9.2.4.6 Analysis

A typical sequence includes:

- Instrument blanks
- Water blanks
- Method blanks
- Field blanks
- Laboratory blanks
- Standards for initial calibration
- Check standards
- samples

9.2.4.7 Limits of detection

Table 5 shows the limit of detection values obtained for the HPAEC-PAD application example.

Table 5: Analytical limit of detection (LOD) for the investigated target compounds.

	Compound	Analytical LOD (mg L ⁻¹)
1	Inositol	0.002
2	Erythritol	0.002
3	Levogluconan	0.006
4	Arabitol	0.002
5	Mannosan	0.004
6	Mannitol	0.005
7	Mannose	0.005
8	Galactosan	0.010
9	Glucose	0.003
10	Galactose	0.003
11	Fructose	0.080
12	Sucrose	0.080

9.2.4.8 Chromatograms

Figure 3 shows a chromatogram of the 10 mg L⁻¹ standard solution. The corresponding retention times are summarized in Table 6.

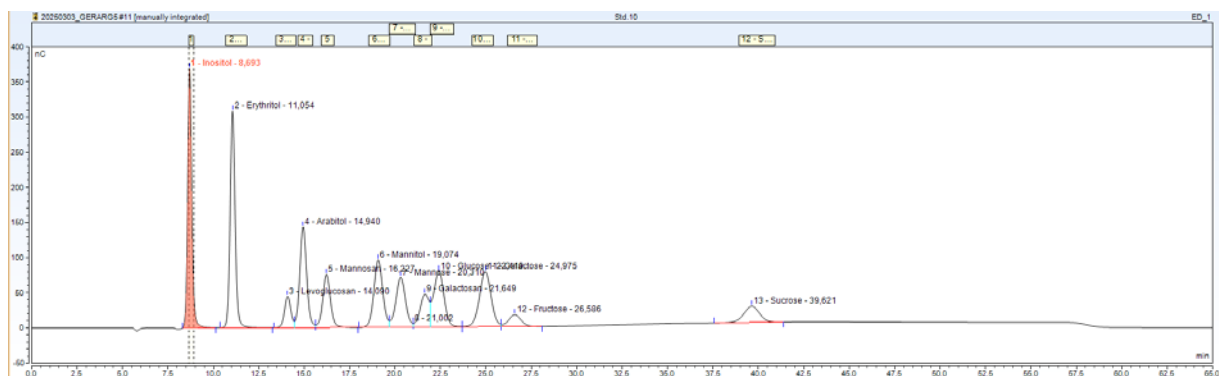


Figure 3: Chromatogram of the 10 mg L⁻¹ standard concentration (screenshot).

Table 6: Compounds retention time.

	Compound	Retention time (min)
1	Inositol	8.776
2	Erythritol	11.250
3	Levoglucosan	13.994
4	Arabitol	15.067
5	Mannosan	16.284
6	Mannitol	19.124
7	Mannose	20.396
8	Galactosan	21.547
9	Glucose	22.592
10	Galactose	24.825
11	Fructose	27.018
12	Sucrose	39.930

9.3 Draft offline data workflow OGTAC-CC for metadata provision

Content:

Institute/NF/Laboratory

Station latitude

Station longitude

Station altitude

Time zone

PI

File name

Protocol	Property	Obligation	Description	Comment
Medium pretreatment	medium type	M	substrate material, could be a drop-down list	Drop-down list?
	medium description	M	free text e.g. manufacturer or name or size...	EBAS: Make/manufacturer and filter size. Free text.
	medium identifier	M	internal handling name of the batch of medium	or substrate material
	treatment SOP	M	URL or version number of SOP	Individual NF SOP
	date	M	date of handling ISO8601 YYYY-MM-DD (HH:mm:ss)	
	medium prefiring	M	Temperature in Kelvin and duration of medium prefiring treatment, or 'Not prefired' if this is the case.	
	medium conditioning	M	Temperature (in K), relative humidity and duration of filter conditioning. Set to 'None' if filter is not conditioned.	

	medium storage condition	M	Temperature (in K)	could be also > < XYK
	medium transport	M	transport start/end date/time, duration, temperature, container, type of transfer	
Medium exposure	medium Identifier	M	internal handling name of the batch of medium	
	station code/name/ID	M	NF specific	Should only link to the NF general description
	exposure SOP	M	URL or version number of SOP	Individual NF SOP
	instrument type	M	Type of sampling device (high volume, low volume, impactor...)	
	exposure device identifier	M	PID of the exposure device	PID includes name, manufacturer, model, serial number and the connected inlet, therefore no further inlet information needed, size fraction?
	Size fraction	M	PM10, PM2.5 or PM1 or TSP	
	exposure start	M	start date of exposure ISO8601 YYYY-MM-DD HH:mm:ss	
	exposure stop	M	stop date of exposure ISO8601 YYYY-MM-DD HH:mm:ss	if identical to exposure start then "blank filter"
	exposure latitude	M	latitude of measurement location in degN	measurement location NOT station location
	exposure longitude	M	longitude of measurement location in degE	
	exposure altitude	M	altitude of measurement location in m a.s.l.	
	measurement height	M	height of the inlet above the ground in m a.s.l.	

	sampled air volume	M	unit???	
	exposure temperature	M	Temperature (in K)	as average?
	exposed substrate area	M	Size of the area of the substrate material that has been exposed, unit cm ²	not relevant for impactor sampling
	filter face velocity	M	Filter face velocity (cm s ⁻¹) = Flow rate (cm ³ min ⁻¹)/60 (s min ⁻¹)/ Exposed filter area (cm ²)	not relevant for impactor sampling
Medium storage and transport	loaded substrate identifier	M	internal handling name of the loaded substrate material (sample)	
	on site storage conditions	M	Temperature (in K)	could be also > < XYK
	loaded substrate transport	M	transport start/end date/time, duration, temperature, container, type of transfer	
	laboratory storage type	M	as loaded substrate material or as extract	
	laboratory storage conditions	M	Temperature (in K)	could be also > < XYK
Sample preparation	loaded substrate identifier	M	internal handling name of the loaded substrate material (sample)	
	date	M	date of handling ISO8601 YYYY-MM-DD HH:mm:ss	
	weighing-out prior sample preparation	M	Temperature (in K), relative humidity and duration of loaded substrate weighing-out. Set to 'None' if loaded substrate is not weighed-out.	
	sample preparation SOP	M	URL or version number of the SOP	Individual NF SOP
	laboratory code	M	if analysis is not performed at station, information on laboratory	EBAS Code?

	sample identifier	M	internal handling name of the sample (extract from loaded substrate material)	
	extracted filter area	M	Size of the area of the filter that has been extracted, unit cm ²	
	extraction type	M	device type used for extraction	shaker, ultrasonication
	extraction solvent	M	free text	to handle also mixtures
	extraction volume	M	(mL)	
	extraction conditions	M	time (min), temperature (K), speed (rpm)	
	filtration type	M	manufacturer name	
	sample storage	M	Temperature (in K), "No" means direct analysis after extraction	
Sample analysis	sample identifier	M	internal handling name of the sample (extract from loaded substrate material)	
	analytical measurement technique	M	drop down menu	example from EBAS
	analytical instrument type	M	PID	
	analysis SOP	M	URL or version number of the SOP	Individual NF SOP
	date	M	date of handling ISO8601 YYYY-MM-DD HH:mm:ss	
	sample analysis procedure identifier	M	internal sequence reference	file name
	internal standard	M	name + manufacturer; if more than one please separate by semicolon	free text
	external lab code	O	unique laboratory identifier if an external lab is doing the analysis	