



Evaluation of the performance of the determination of anions in the water soluble fraction of atmospheric aerosols

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ABSTRACT

The knowledge of the mass of particulate matter in air, its chemical composition and emission sources is of relevance for taking decisions concerning air quality management in urban areas. The interpretation of these data is a function of the quality of the measurement results expressed by their uncertainties.

This study aimed at developing models of the performance of the determination of anions in the water-soluble fraction of atmospheric aerosols, capable of determining, separately, the contribution of aerosols sampling, extraction of water-soluble fraction of atmospheric aerosols and quantification, by ion chromatography, of anions in the extract. The sampling procedure was assessed from the dispersion of results of duplicate parallel sampling after subtracting the analytical component of this dispersion. These models are used to evaluate the adequacy of the measurement procedure for the determination of urban aerosol composition and to support strategies for reducing measurement uncertainty or cost of analysis. The method performance was studied for the following ranges considering extract dilution up to five times: 0.23–8 $\mu\text{g m}^{-3}$ for chloride and nitrate, and 0.093–3.25 $\mu\text{g m}^{-3}$ for sulphate. Measurements are fit for the analysis of urban aerosols since the relative expanded measurement uncertainty is smaller than a maximum value of 40%. The percentage contribution of the uncertainty components varies with the analyte and its mass concentration, the major components being 24–93% for the extraction, 43–59% for sampling, 0.2–28% for the interpolation of the sample signal in the calibration curve and 4–8% for air volume measurement. The typical composition of analysed air is: $(1.12 \pm 0.26) \mu\text{g m}^{-3}$, $(1.02 \pm 0.30) \mu\text{g m}^{-3}$ and $(0.76 \pm 0.22) \mu\text{g m}^{-3}$ of chloride, nitrate and sulphate in the water soluble fraction of aerosol, respectively, for a confidence level of approximately 95% considering a coverage factor of 2.

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1. Introduction

Air composition, in terms of both gases and aerosols, is known to affect human-health. Hence, there is the need to perform measurements that provide relevant information about air quality. The European Directive 2008/50/EC [1] defines rules for ambient air monitoring programmes and sets limit values for particulate matter and some gaseous air components. This legislation also suggests assessing the origin of air pollution to infer which contaminants are expected to be present and should be tested.

Since the assessment of the compliance of ambient air with legislation or the evaluation of spatial or temporal differences in air composition depend on measurement quality, air composition data should be reported with respective uncertainty. Only when difference in estimated air composition with a limit value or between different air masses cannot be explained by measurement

uncertainty, should be considered physical-chemical meaningful. Directive 2008/50/EC [1] defines maximum measurement uncertainty for assessing compliance with defined limit or target values. In this directive, for measurements of some contaminants (namely, sulphur dioxide, nitrogen dioxide, oxides of nitrogen, carbon monoxide, benzene, particulate matter, lead, ozone and related NO and NO₂), maximum measurements uncertainty of 15–25% or 25–50% of limit or target values of the contaminant for fixed and indicative measurements, respectively, are defined. Quality requirements of indicative measurements are less strict than for fixed measurements used to access higher levels of pollutants.

The measurement uncertainty is known to result from the combined effect of various individual components [2]. The identification and separate quantification of the uncertainty components allows recognition of major contributions, so that procedures can be developed to reduce them, thus increasing the quality of results. This information can also be used for cost of analysis reduction by identifying minor uncertainty components that can be managed with fewer resources (e.g., use of cheaper and more uncertain volumetric equipment).

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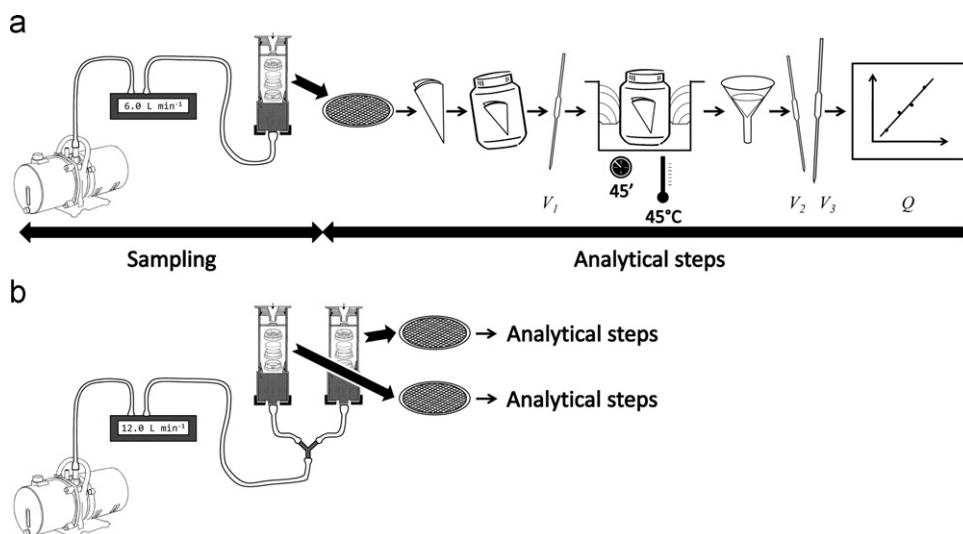


Fig. 1. Schematic representation of (a) the measurement procedure of the concentration of anion in the water-soluble fraction of atmospheric aerosols and (b) the measurement procedure used for estimating global measurement repeatability, s'_c (see glossary for notation).

In this work, a measurement procedure for the determination of anions in the water-soluble fraction of atmospheric aerosols is implemented and validated. This measurement procedure is based on active sampling of aerosols onto filters, followed by extraction with water, under defined conditions, and analysis of anions in the extracts by ion chromatography (Fig. 1a).

This work aims at developing a model of the measurement performance for assessing its adequacy for the analysis of urban aerosols and supporting needed performance improvements and/or cost of analysis reduction. The performance of all analytical steps, including sampling and filter extraction, are assessed separately. Air sampling is known to be a critical stage of these measurements [3].

The developed measurement procedure, including the measurement uncertainty, is applicable to the analysis of air in sites where air quality is, expectedly, representative of an area. Directive 2008/50/EC [1] describes how to proceed to guarantee the representativeness of air sampling. The uncertainty associated with the heterogeneity of the represented area is not considered in this work since measures can be implemented to reduce its impact on air quality evaluation and, whenever this effect is relevant, it varies with the specific problem faced.

The evaluation of the performance of the complex analytical steps (known to be difficult to model separately), namely sampling and filter extraction, was performed using the differential approach of the evaluation of the measurement uncertainty [4]. This approach involves the following stages: (1) quantification of the standard deviation of measurements precision, s ; (2) estimation of the combined uncertainty of modelled uncertainty components, u_{inc} ; (3) estimation of complex (not previously modelled) uncertainty components by difference between s and u_{inc} ; (4) development of a model of the global measurement uncertainty [4]. The magnitude of various complex uncertainty components can be quantified, separately, by estimating the precision at different stages of the measurement [4].

In this work, intralaboratory reproducibility (intermediate precision in the latest edition of the International Vocabulary of Metrology [2]) of measurements excluding sampling was used to develop models of the analytical steps. These models together with the measurement repeatability, including the sampling stage, were used to estimate the sampling uncertainty by difference.

The measurement repeatability was estimated from results of duplicate parallel sampling.

The developed models estimate measurements performance for a continuous mass concentration range of anions in the water soluble fraction of aerosols, assuming that the relative uncertainties of extraction and sampling are constant in the studied range.

2. Materials and methods

2.1. Sampling

The sampling system was placed on the roof terrace of the 4th floor of C8 building of the Faculty of Science of the University of Lisbon, Portugal.

Aerosols were sampled, using Gent type samplers, onto Whatman[®] 41 filters of 47 mm diameter collecting particulate matter with aerodynamic diameters smaller than 1 μm (PM_{10}). The measurement repeatability was studied from results of 10 parallel and simultaneous samplings performed by two parallel samplers, positioned at a distance of 15 cm, connected to the same air pump (Fig. 1b). The sampling was performed over 24-h periods of 10 consecutive days at a flow rate, θ , of 6 L min^{-1} (the pump was operating at 12 L min^{-1}). The geometry of air splitting device was tested by measuring air flow after each filter. Since air flow readings are the same in both branches, the air splitting differences were considered negligible.

2.2. Measurements of anions in the water soluble fraction of aerosols

Each sampled filter was placed in a 15 mL PTFE bottle to which 10 mL (V_1) of ultrapure water were added. The capped bottles were sonicated for (45 ± 2) min at a temperature of (45 ± 1) °C after which the extract was cooled to room temperature and filtered through a Whatman[®] 41 filter. The control of the extraction temperature aimed at keeping extraction efficiency independent of room temperature. The sample extract was diluted, when needed, by adding a volume V_3 of water to a volume V_2 of the extract, and analysed by ion chromatography (IC). Volumetric measurements were performed with Class A glassware.

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