AEROSOL METROLOGY FOR ATMOSPHERIC SCIENCE AND AIR QUALITY

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Περίληψη

Οι μετρήσεις των αιωρουμένων σωματιδίων είναι ζωτικής σημασίας για την επιβολή των ευρωπαϊκών κανονισμών ποιότητας αέρα για την προστασία της ανθρώπινης υγείας και την έρευνα για την επίδραση της κλιματικής αλλαγής. Χρησιμοποιούνται συνήθως μετρήσεις των PM_{10} και $PM_{2.5}$ (σωματίδια με αεροδυναμική διάμετρο μικρότερη των 10 μm και 2.5 μm αντίστοιχα) αλλά η αβεβαιότητα των μετρήσεων είναι υψηλή και η ιχνηλασιμότητα ανεπαρκής. Στο πλαίσιο αυτό το πρόγραμμα "AEROMET" (EMPIR) εγκρίθηκε πρόσφατα από την ΕΕ με τη συμμετοχή 21 φορέων από 15 χώρες μεταξύ των οποίων και το εργαστήριό μας. Η διάρκεια του προγράμματος είναι 36 μήνες (Ιούνιος 2017- Μάιος 2020).

Σε αυτή την εργασία θα παρουσιασθεί το περίγραμμα του προγράμματος, ο σκοπός και οι στόχοι του καθώς και ορισμένα προκαταρκτικά αποτελέσματα.

Το AEROMET στοχεύει α) στην ανάπτυξη μεθόδων ιχνηλασιμότητας και βαθμονόμησης διαφόρων οργάνων μέτρησης σωματιδίων που μπορούν να καλύψουν το περιβαλλοντικά απαιτούμενο εύρος μεγεθών από μερικά nm έως 10 μm καθώς και τις σχετικές συγκεντρώσεις μάζας και αριθμό αιωρουμένων σωματιδίων β) και στον χαρακτηρισμό των συστατικών των αιωρουμένων σωματιδίων όπως απαιτείται από τα δίκτυα παρακολούθησης της ποιότητας του αέρα στις χώρες της ΕΕ.

Ειδικότερα οι στόχοι του προγράμματος είναι:

- 1. Ο σχεδιασμός και η κατασκευή ενός συστήματος θαλάμου αιωρουμένων σωματιδίων για τη βαθμονόμηση των οργάνων μέτρησης των PM_{10} και $PM_{2.5}$ σωματιδίων (WP1)
- 2. Η εφαρμογή ιχνηλάσιμων επικυρωμένων μεθόδων για τον προσδιορισμό των διαφόρων ειδών άνθρακα καθώς και τοξικών στοιχείων όπως As, Cd, Hg and Ni, προκειμένου να ικανοποιηθεί η τρέχουσα νομοθεσία (WP2).
- 3. Η ανάπτυξη διαδικασιών βαθμονόμησης για φορητά φασματόμετρα μέτρησης μεγέθους σωματιδίων MPSS -Mobility Particle Size Spectrometers- (WP3)

- 4. Η χρήση φορητών φασματομέτρων ακτίνων X, για τον ποσοτικό προσδιορισμό της σύστασης των σωματιδίων στο πεδίο και σε πραγματικό χρόνο ανάλυσης. Τα αποτελέσματα θα συγκριθούν με εργαστηριακές αναλύσεις (πχ. ICP-MS) των δειγμάτων (WP4)
- 5. Η τεχνολογία και η υποδομή των μετρήσεων να υιοθετηθεί από διαπιστευμένα εργαστήρια, οργανισμούς τυποποίησης προτύπων και τελικούς χρήστες από άλλα ευρωπαίκά δίκτυα παρακολούθησης (WP5).

Το εργαστήριό μας συμμετέχει σαν εξωτερικά χρηματοδοτούμενος εταίρος στα πακέτα εργασίας WP2 και WP4. Συντονιστής του AEROMET είναι το PTB (Physikalische-Technische Bundesanstalt, Γερμανία) και συμμετέχουν πολλά Εθνικά Ινστιτούτα Μετρολογίας (NMIs), όπως BAM (Γερμανία), NPL (Ηνωμένο Βασίλειο), METAS (Ελβετία), INRIM (Ιταλία), TROPOS (Γερμανία), LNE (Γαλλία), DMF (Δανία) κλπ.

Σκοπός του προγράμματος είναι να έχει μια σημαντική και μετρήσιμη επίδραση στις μελέτες ποιότητας του αέρα μέσω ενός συνδυασμού αλληλεπίδρασης των κυριότερων ενδιαφερομένων φορέων και των τελικών χρηστών.

Λέζεις- κλειδιά: Ερευνητικό πρόγραμμα AEROMET(EMPIR), αιωρούμενα σωματίδια της ατμόσφαιρας PM_{10} , $PM_{2.5}$, φορητά φασματόμετρα, ανάλυση πεδίου σε πραγματικό χρόνο, ποιότητα αέρα

Abstract

Measurements of aerosol particles are vital for enforcing EU air quality regulations to protect human health, and for research on climate change effects. Although metrics such as PM_{10} and $PM_{2.5}$ (i.e. air of particles with aerodynamic diameter smaller than 10 μ m or 2.5 μ m) are currently in use, the level of uncertainty is too high and the traceability is insufficient. In this context an EMPIR project "AEROMET" was recently approved by the EC with the participation of 21 European partners from 15 different countries, involving our Laboratory from NTUA. The duration of the project is 36 months (June 2017-May 2020).

This presentation will give an overview of the project outlining, its aim, objectives and some preliminary results. AEROMET aims, a) to develop and demonstrate methods for traceability and calibration of different aerosol instruments capable for investigations of the environmentally relevant size range from several nm up to $10~\mu m$ and the regulatory relevant mass concentrations and number concentrations of PM (Particulate Matter) and b) the characterization of regulated components in airborne particles as needed by air quality monitoring networks within the EU.

The specific objectives of the project are:

- 1. The design and building of a demonstration aerosol chamber system for the calibration of the PM_{10} and $PM_{2.5}$ instruments (WP1)
- 2. The establishment of traceable validated methods for the determination of the different carbon species and toxic elements, like As, Cd, Hg and Ni, in order to meet the current regulation (WP2).

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- 3. The development of calibration procedures for Mobility Particle Size Spectrometers (WP3)
- 4. The use of mobile X-ray spectrometers for the quantification of particle compositions in the field in real time analysis. Results will be compared with lab-based (e.g. ICP-MS) reanalysis of the samples (WP4).
- 5. The take up of the technology and measurement infrastructure of the project by the accredited laboratories, by standards developing organizations and end users from other European Monitoring Networks (WP5).

Our Lab participates as an external funded partner in this project in the WP2 and WP4 packages. The coordinator of AEROMET is PTB (Physikalische-Technische Bundesanstalt, Germany) and several other NMIs (National Metrological Institutes), as BAM (Germany), NPL (U.K.), METAS (Switzerland), INRIM (Italy), TROPOS (Germany), LNE (France), DMF (Denmark) etc. participate. The whole intention of the project is to have a significant and measurable impact on air quality studies through a combination of interaction with the main stakeholders and end-users.

Keywords: EMPIR AEROMET Project, particulate matter PM_{10} , $PM_{2.5}$, mobile spectrometers, in field real time analysis, air quality

1. Introduction

Atmospheric pollution by airborne particles contributes significantly to climate change and has been linked to adverse health effects, such as respiratory, cardiovascular diseases and lung cancer [Fuzzi et al., 2015, Kim et al. 2015, WHO-REVIHAAP, 2013]. It has been estimated that only in Europe, more than 500,000 deaths per year can be attributed to PM exposure, whereas in pollution hot spots of PM alone are responsible for a loss in statistical life expectancy of up to 12 to 36 months [Fuzzi et al., 2015]. For the member states of the EU, air quality monitoring - as laid down in the Air Quality Directive 2008/50/EC - is mandatory and comprises quantification of airborne particles and their components. The most important metric to monitor particulate air pollution is the mass concentration, more specifically the total mass per unit volume of air of particles with aerodynamic diameter smaller than 10 µm or 2.5 µm, commonly referred to as PM₁₀ and PM_{2.5}, the ambient limit values of which have been established in Europe [Directive 2008/50/EC], the USA [National Ambient Air Quality Standards, E.P.A.] and elsewhere. All local, national or EU action plans and measures to reduce particulate air pollution rely on air monitoring networks [Umweltbundesamt Oesterreich, REP-0121, 2007, JRC-AOUILA Position Paper, 2013], the quality of data they provide and the methods in use [Aerosol Measurement Procedures, 2016, WMO-report 227, Guidance to the demonstration of equivalence of ambient air monitoring methods, 2010]. It is recognised that the regulated metrics for particulate air pollution suffer from severe methodological deficiencies.

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PM mass concentration was established as the default metric of PM on the basis that mass measurements can be easily made in a traceable manner. The gravimetric filter based reference methods for PM₁₀ and PM_{2.5} (EN 12341:2014; EN 14907:2005), however, fall short in areas such as sensitivity and ongoing Quality Assurance and Quality Control due to the following reasons: Many particles are hygroscopic, so that their mass and size depends strongly on the humidity. There can be significant sampling issues such as impaction (removing larger particles) and diffusive losses to walls (removing smaller particles) resulting in uncertainties of the total mass determination of up to several 10 %. Collecting particles on filters for subsequent chemical analysis can give rise to additional sampling issues relating to chemical interactions and differing effects of different filter materials. Moreover, automatic PM monitoring systems, which were developed in order to avoid these disadvantages and enable time resolutions below 24 h, must demonstrate their equivalency to the reference manual gravimetric method. This is problematic due to both the inconsistency in the automatic instruments based on different working principles and the ambiguity within the aerosols used for comparison. PM₁₀ and PM_{2.5} methods are being revised within CEN TC 264 WG 15, where it is recognized that further research is needed to bring the reliability of measurements in line with that for other regulated air quality metrics. The proposed solution in AEROMET project is the implementation of traceable reference methods for the harmonized lab based calibration of automatic PM measuring instruments (e.g. Tapered Element Oscillating Microbalance Filter Dynamics Measurement Systems (TEOM), Beta attenuation monitors).

It is evident that mass concentration is too crude a metric regarding spatial and temporal resolution and sensitivity to characterize ambient aerosol particles for the purposes of understanding their sources, health effects and possible effects on climate. Additional metrics with high time resolution such as particle number concentration and size distribution need to be integrated into air quality networks in order to make variations in time and space measurable, and to better identify sources, primary particles and peak concentrations. This is especially necessary for the fraction of ultrafine particles (UFP), as their mass is negligible in comparison to PM_{2.5} and PM₁₀. Current metrics for UFP are not as harmonized as those for PM₁₀, PM_{2.5} or black carbon and the reliability of the existing data is not ensured. General calibration concepts for the measurement of particle number concentration and particle size (with CPCs and MPSS) are already described in fundamental standards (ISO 15900:2009 and ISO 27891:2012). A CEN working group is adapting them to the needs of ambient air quality monitoring networks (FprCEN/TS 16976). These calibrated "network"-CPCs should have an upper limit of 10⁵ per cm³ in particle number concentration in ambient air; lower and upper particle size limits should be 7 nm and a few micrometres, respectively. Consequently, monitoring networks need reliable calibration services. The project AEROMET is aiming at providing the necessary independent calibration facilities.

Anions and cations constitute important parameters for understanding secondary inorganic aerosol formation and also for measuring primary ionic PM such as sea salt [Ochsenkühn et

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al., 2008, Ochsenkühn and Ochsenkühn, 2010, Tsopelas et al., 2008, Ochsenkühn et al., 2010]. Manual reference methods for anions and cations, and EC/OC are close to being completed in CEN TC 264 WGs 34 and 35, respectively, but a deeper understanding of potential measurement biases such as on filter artefact and the loss of volatile ionic PM is required before the SI traceability can be put in place. The accurate measurement of natural sources is particularly important since legislation allows them to be subtracted from a Member State's PM concentrations.

As conventional methods for quantitative elemental analysis of environmental aerosols – in field filter sampling and lab based analysis – show substantial reliability and sensitivity restrictions there is a strong need to develop new flexible approaches with increased accuracy, time resolution and speed of analysis. In field XRF in combination with appropriate sampling could - within the framework of SI traceability and calibration at NMIs and DIs - bring regulatory measurements of PM composition to the required high quality level. A definition of the spatial representativeness of measuring points in air quality networks is still missing in the legislation and the knowledge gap has to be filled by individual temporary measurement campaigns for which in field XRF in combination with appropriate sampling is a suitable candidate method. XRF based aerosol analysis offers additional analytical tools for a more reliable analysis of particulate components which corresponds to the needs from atmospheric sciences [Academic-industrial roadmap of the International Initiative on X-ray fundamental parameters, 2012].

The overall aim of the current project AEROMET is to develop and demonstrate methods for traceability and calibration of different aerosol instruments covering the environmentally relevant size range from several nm up to at least 10 μ m and the regulatory relevant mass concentrations (0.1 to 1000 μ g/m³) and number concentrations (0.1 to 10⁷ particles per cm³).

2. Methodology-Structure of AEROMET

The structure of the AEROMET project is demonstrated in Table 1, reporting the individual Work Packages (WP) with the corresponding Work Package Leaders (WPLs) from several European NMIs (National Metrological Institutes).

Table 1: Structure of the AEROMET project

Work	WP1	WP2	WP3	WP4	WP5	WP6	WP7
packag							
e (WP)							
Title	New	Methods	Calibration	Quantifying	Development	Creating	Manage
	reference	for the	of MPSS	airborne	of reliable		ment
	methods	analysis of	and CPC	particle	XRF	impact	and

	for PM ₁₀ and	major components		composition in the field	techniques		coordina tion
	$PM_{2.5}$						
Leader	METAS	NPL	TROPOS	BAM	INRIM	NPL	PTB

ETAS: Eidgenoessisches Institut fuer Metrologie (Swiss); NPL:Management Limited (UK); TROPOS: Leibniz Institute for Tropospheric Research (Germany); BAM:Bundesanstalt fuer Materialforschung und Pruefung (Germany); INRIM:Istituto Nazionale di Ricerca Metrologica (Italy); PTB:Physikalisch-Technische Bundes anstalt (Germany)

More specifically, the tasks undertaken in different work packages (WP) are:

In **WP1** the design and construction of a demonstration aerosol chamber system for the labbased calibration of PM-measuring instruments is under investigation by using synthetic ambient aerosols.

WP2 investigates the establishment of traceable validated methods for the analysis of major components of particulate matter, such as elemental (EC)-, organic (OC)- and total carbon (TC), anions and cations, toxic trace elements (As, Cd, Hg, Ni) in order to meet the data quality of current regulation (EC Directives on ambient air quality).

WP3 deals with the improvement of the quality of number-based aerosol measurements in order to meet regulatory needs and to make environmental aerosol data more reliable and comparable.

WP4 applies mobile and on-site X-Ray spectroscopy techniques combined with mobile onsite particle sampling techniques for real-time mass concentration measurements of ECregulated elements (As, Cd, Hg, Ni) in ambient aerosols. Re-analysis of the samples with conventional lab-based techniques (ICP-MS) will be carried out.

WP5 develops traceable and reliable X-Ray analytical methods to measure the elemental mass deposition per unit area, elemental composition and the chemical binding state (speciation) of airborne particles deposited on flat homemade nanostructured substrates, which will be used in cascade impactors for size discriminating of coarse and ultrafine fractions.

WP6 comprises knowledge transfer via stakeholder meetings, conferences, publications in open access journals, workshops, training and exploitation to the benefit of the end users (calibration services, reference materials and interaction with instrument manufacturers).

WP7 aims to the management and coordination of AEROMET project through meetings and project reporting.

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3. Results and Discussion

In Fig. 1 (a) the tri-modal size distribution of ambient aerosols and a representative aerosol composition Fig.1 (b) are demonstrated. Aerosols or Particulate Matter (PM) have not a distinct chemical composition, but they consist from variable combustion particles salts, metal oxides, organic substances and other materials. Several factors affect the measured PM weight, as humidity on filter material and on hygroscopic PM, loss of filter material, temperature during sampling, storage and transport on semi-volatile material, weather conditions in general, chemical reactions on the filter, static electricity, balance drift etc. Therefore a suitable standard calibration aerosol for calibrating commercial PM-measuring instruments does not exist.

Preliminary results already obtained, together with future actions for the different WPs are as follows:

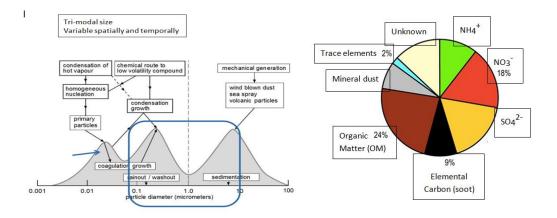


Figure 1: Tri-modal size distribution of ambient aerosols (a) and a representative aerosol composition (b) [K.Vasilatou, METAS, AEROMET WPL1, Project meeting, Turin 2/2018]

In **WP1** a synthetic ambient aerosol was produced using a test dust having a representative aerosol composition (Fig. 1 b) and different atomizer systems tested with dry and wet processes. Furthermore, the design of a mixing aerosol chamber based on computational simulations is in progress, where a stable and reproducible generation of the synthetic aerosol will happen. Finally, the development of a protocol for a lab-based calibration of automatic PM instruments will be prepared using this validated aerosol chamber.

The leader of **WP2** (NPL) has already participated in ACTRIS/AQUILA inter-laboratory comparison exercises for TC and EC measurements using conventional methods. In Figure 2

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the z-scores for EC/TC ratio calculated using σ^* from the data obtained in this Proficiency Testing Scheme (PTS) are demonstrated. The carbonaceous chemical components of some ambient air samples obtained during the AEROMET project will furthermore be analyzed by X-Ray Absorption Fine Structure Spectroscopy (XAFS) in order to develop complementary analysis methods to the conventional ones [Bolbou et al., 2010]. For the analysis of the required cations (As, Cd, Hg, Ni) except from the participation in relevant PT schemes using ICP-MS, a new TXRF (Total X-ray Fluorescence) instrument based on internal standardization will be tested for its capability to provide independent chemical and physical traceability chains and to extend the range of metals analyzed beyond those required for regulation.

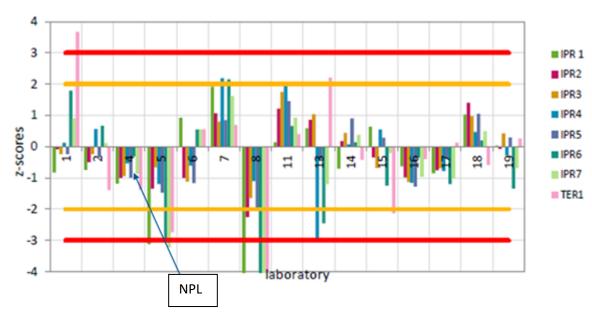


Fig. 2: z-scores for EC/TC ratio calculated using σ^* from data obtained in a round of a proficiency testing scheme [P.Quincey, NPL, AEROMET WPL2, Project meeting, Turin 2/2018]

From **WP3**, the establishment of specific calibration procedures for Mobility Particle Size Spectrometers (MPSS) is already carried out [Wiedensohler et al., 2018]. Furthermore, the provision of calibration facilities for Condensation Particle Counters (CPCs) to the CEN standard FprCEN/TS 16976 are in progress, as well as the investigation of particle losses as function of flow rate and particle diameter (sub-10nm). Continuous feedback of findings will be delivered to standardization committees (CEN&ISO) and observational networks (ACTRIS&GAW).

Within **WP4** the selection of two representative sampling sites (Site1, urban: Cassino, Italy and Site 2, suburban background: Budapest, Hungary) is already decided (Fig.3), as well as the duration and dates of the field campaigns (May and September 2018) and the instruments

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and equipment to be used. At these two sites, real time in-situ measurements on element mass concentration will be carried out by using mobile and semi-automated devices (multi- stage cascade impactors). The filters obtained from the different particle size fractions (from 10 µm down to several nm) will be measured for the regulated elements in aerosol samples as As,Cd, Hg, Ni by non destructive in-situ mobile XRF. Later on a backup analysis of the same filters by conventional techniques (ICP-MS, lab-based calibrated XRF) will be carried out for comparison of the results obtained by the mobile XRF.



Figure 3: Site map of the selected sampling sites [S.Seeger BAM, AEROMET WPL4, Project meeting, Turin 2/2018]

In **WP5** the design and production by different lithographic approaches of artificial microand nanostructured reference samples that are suited for the qualification of calibration standards for TXRF and GIXRF (Grazing Incidence X-Ray Fluorescence) quantitative and size dependent analysis of aerosols are already in progress. In this framework the fabrication

of some micro and nanostructured substrates and some micro and nanoparticles samples, as flat and patterned Si/Ge substrates with disordered and ordered nanoparticles from Fe₃O₄ seeds, core-shell nanoparticles (silica core and polystyrene shell with different sizes), gold-coated silicon pillars etc. is ongoing or completed. By using these substrates, the XRF methods mentioned above will be tested for quantitative and size sensitive analysis of PM. A SOP (Standard Operation Procedure) on the traceable calibration of bench top GIXRF and TXRF instruments and sampling devices for the measurement of elemental mass concentration in ambient PM from PM10 to the nanoscale will be prepared.

4. Conclusions

The AEROMET project will have impact on metrological, scientific and industrial communities, as well as on relevant standards. Especially, the new lab-based calibration procedure of PM-measuring instruments will have direct impact on the revision of standards and national air quality monitoring networks. The novel particle chamber test facilities for the generation of synthetic ambient aerosols can be applied in the performance assessment of air filters contributing to the related standards. The novel XRF methodologies in WPs 4 and 5 enhance the quality of aerosol component analysis in air quality monitoring networks by providing flexible in-situ multi element analysis.

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