Publishable Summary for 16ENV07 AEROMET
Aerosol metrology for atmospheric science and air quality

Overview

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 the mass concentration of airborne particulate matter (PM) are currently in use, the level of uncertainty is too high, and the traceability is insufficient. Therefore, the project established reproducible reference methods for PM_{10} (inhalable particles with diameters of 10 micrometres and smaller) and $PM_{2.5}$ (fine inhalable particles, with diameters of 2.5 micrometres and smaller) by developing a new facility for artificial aerosol generation. In accordance with EU regulation and air quality monitoring networks requirements, the project achieved target uncertainties of below 15 % for aerosol measurements and characterised regulated components in airborne particles for PM instrument calibrations. In addition, the project produced quantification procedures for elemental compositions of airborne particles, based on x-ray analytical techniques that use chemical and physical traceability chains, along with complementary analytical techniques such as ICP-MS, for validation purposes. The partners also engaged with key members of the supply chain; from accredited laboratories to standards developing organisations (such as ISO and CEN), as well as end users (such as the AQUILA network); in order to facilitate the take up of technology and measurement infrastructure developed during the project.

Need

Regulatory bodies, air quality networks and atmospheric instrument manufacturers all require the improvement of air quality monitoring; however, there is currently a lack of traceable calibration standards and harmonised calibration procedures for measuring airborne PM. In addition, methods measuring PM_{10} and $PM_{2.5}$ (particle mass concentration) within the EU Air Quality Directive 2008/50/EC need improving, in order to ensure the comparability of local data measured by instruments relying on different working principles (e.g. gravimetric vs. optical measurements). Therefore, reference methods for measuring PM_{10} and $PM_{2.5}$ and calibration methods for the instruments used for such measurements are needed.

The chemistry of aerosols (elemental composition analysis) is also part of the existing regulation and it is necessary to understand their origins, behaviour and environmental fate and impacts (i.e. effects on health and climate). However, current methods for the quantification of regulated aerosol components (e.g. Elemental Carbon and Organic Carbon (EC/OC), metals, anions, and cations) are notoriously inflexible in terms of time, spatial resolution and lack accuracy. Therefore, validated methods for the determination of major components of PM are needed as well as reliable procedures for standard instrumentation such as Mobility Particle Size Spectrometers (MPSS) and Condensation Particle Counters (CPC). Modern x-ray analytical techniques have the potential to enable the quantitative chemical analysis of airborne particles directly at their emission sources, therefore new SI-traceable x-ray techniques is a development desideratum.

Objectives

The overall aim of the project was to develop and demonstrate methods for traceability and calibration of different aerosol instruments capable of covering the environmentally relevant size range from several nm up to 10 µm and the regulatory relevant mass concentrations (0.1 µg/m^3 to 1000 µg/m^3) and number concentrations (10^2 to 10^6 particles per cm^3). The traceability and calibration considered the above metrics, as

well as mass concentration and chemistry of particle components. The project also focussed on providing the necessary EU wide calibration infrastructure for aerosol instruments. The specific objectives of the project are:

1. To develop reproducible reference methods for PM_{10} and $PM_{2.5}$, including the design and building of a demonstration aerosol chamber system for calibrating PM_{10} and $PM_{2.5}$ instruments using representative generated aerosols and to achieve target uncertainties below 15 %.
2. To establish traceable validated methods for the determination of major components of particulate matter, such as elemental and organic carbon (EC/OC), total carbon, anions and cations and major metals (e.g. arsenic, cadmium, mercury, nickel) in order to meet the data quality objectives of current regulation.
3. To develop calibration procedures for Mobility Particle Size Spectrometers (MPSS) for ambient measurements in the size range up to 1000 nm - in support of standardisation requirements from ISO TC 24 WG 12 and CEN TC 264 WG 32. To provide calibration facilities for measuring particle number concentration using Condensation Particle Counters (CPCs) in ambient air – as required by the standard FprCEN/TS 16976.
4. To apply mobile x-ray spectroscopy techniques combined within particle sampling techniques for quantifying particle compositions in the field for real time analysis. Results will be corroborated by a backup lab-based reanalysis of samples. To qualify a synchrotron based GIXRF setup as a traceable reference for a quantitative chemical aerosol analysis with mobile and bench top GIXRF instrumentation. Full traceability will be achieved by the use of fabricated micro and nanostructures and subsequently calibration along a complete traceability chain will be described in standard operation procedures.
5. To facilitate the take up of the technology and measurement infrastructure of the project by the measurement supply chain (accredited laboratories), by standards developing organisations (such as ISO TC 24 and CEN TC 264 and those linked to the EU Air Quality Directive 2008/50/EC) and end users (e.g. Network of National Air Quality Reference Laboratories (AQUILA) and the European Monitoring and Evaluation Programme (EMEP).

Progress beyond the state of the art

In WP1, a new facility has been developed which allows for a stable and reproducible generation of ambient-like aerosols in the laboratory. The setup consists of multiple aerosol generators, a custom-made flow tube homogeniser, isokinetic sampling probes and a system to control aerosol temperature and humidity. Model aerosols containing elemental carbon, secondary organic matter from the photo-oxidation of α-pinene, inorganic salts such as ammonium sulphate and ammonium nitrate, mineral dust particles and water were generated at different environmental conditions and different number and mass concentrations. The aerosol physical and chemical properties were characterised with an array of experimental methods, including scanning mobility particle sizing, ion chromatography, total reflection X-ray fluorescence spectroscopy, and thermo-optical analysis. The facility is very versatile and can find applications in the calibration and performance characterisation of aerosol instruments monitoring ambient air. In this project, we performed, as proof of concept, an intercomparison of three different commercial PM (particulate matter) monitors (TEOM 1405, DustTrak DRX 8533 and Fidas Frog) with the gravimetric reference method under three simulated environmental scenarios. The laboratory-based method for simulating ambient aerosols developed in the course of this work could provide in the future a useful alternative to time-consuming and expensive field campaigns, which are often required for instrument certification and calibration.

For EC/OC, anions, cations and metals, the major focus has been on building practical links between the NMIs and stakeholders, such as members of AQUILA, through comparisons using reference methods, and contributions to standardization bodies. These links did not exist before and represent an important step forward. One notable example is a comparison, organised by the project, for cations (lithium, sodium, ammonium, magnesium, potassium and calcium), a set of regulated pollutants for which no regular comparisons were available. Early stage investigations into fundamental but non-reference analysis methods for metals and the chemical nature of organic carbon in EC/OC have taken place. New data has also been generated in relation to carbonaceous emissions from wood burning and candles, including the relationship between EC and black carbon. A harmonised calibration infrastructure on EU and international scales will be created for MPSSs and CPCs for ambient particles below 1000 nm in diameter. These calibrations are much more advanced than those provides by the manufacturers. This will offer users with different applications (such

as atmospheric observational networks, regulators, sciences and instrument manufacturers) SI-traceable calibration services for CPCs and MPSSs. To able to conduct such calibrations for low particle number concentration (<1000 cm ^{3}), different types FCAEs have been successfully tested for different flow rates to solve this problem.

Compared to standard field sampling methods (filter), size selective sampling offers combined with XRF, particle size resolved quantification of elements. The combined field application of spatially and temporally flexible sampling and in-situ XRF based quantitative analysis of regulated and non-regulated species in ambient aerosols was successfully tested during two field campaigns for particles down to the nm size. The following main conclusions can be drawn: An overall good quantitative agreement was found between the laboratories in ICP-MS analyses on filter samples although unexpected and unaccountable high background contaminations made a proper background correction partly problematic. The attempt to use cascade impactors and mobile TXRF for a measurement of element mass concentrations in ambient aerosols was successful and now enables short sampling intervals below 6 h and the detection of element mass concentrations as low as 1 ng/m^{3}. The ability of the cascade impactor/TXRF method to deliver results at higher time resolution than the traditional PM10-filter/ICP-MS method could be demonstrated.

The combination of cascade impactor sampling and TXRF analysis is comparable to the traditional method of PM10 filter sampling and ICP-MS for the determination of element mass concentrations in ambient aerosols. TXRF spectrometers need a careful recalibration with an independent reference. Deviations between can be kept within 50% at maximum for most of the elements investigated. TXRF results with the Dekati impactor (mobile TXRF) and the May-type impactor (lab-based TXRF) agreed quite well within 40% for most of the elements analysed. More than five artificial micro and nanostructures were fabricated by optical laser-writing lithography, nanospheres and block copolymers lithography to control the dimensional features and elemental composition with reproducible results as evidenced from scanning electron microscopy characterisation. More than five polymeric and core-shell nanoparticles samples were synthesized, characterised and spread of solid substrates. GIXRF and GEXRF experiments were performed on artificial samples. Aluminum nanodisks were identified as a promising reference sample candidate for laboratory TXRF. Experimental measurements were supported by theoretical modelling of the X-ray standing wave field of the reference structures with varying mass deposition and structures size.

Non-destructive elemental composition analysis was performed on deposited aerosol samples by comparing TXRF and GIXRF data. The comparison allowed to estimate the influence of the X-ray standing wave field on TXRF at different amounts of PM, the XSW is drastically changed for high particulate load leading to difficult quantification. The sample loading is crucial for an accurate quantification. Finally, the chemical binding state was analysed through X-ray adsorption spectroscopy. This activity will be going beyond the current state of the art in the follow-up to this project with artificial substrates reproducing cascade impactors patterns for GIXRF and TXRF analysis.

Results

Objective 1: New reproducible reference methods for PM10 and PM2.5

The requirements of a representative ambient aerosol for testing PM mass concentration instruments have been determined, and four different model aerosols have been defined in terms of physical and chemical properties. These are intended to simulate ambient roadside aerosols in winter and summertime, at normal and elevated levels of pollution. Moreover, generators based on wet or dry dispersion have been tested successfully with respect to stability and reproducibility and, therefore, suitable generators for the production of primary aerosols (ammonium sulphate/nitrate and mineral dust) have been identified. The prototype Micro Smog Chamber (MSC), developed by the University of Applied Sciences and Arts North Western Switzerland, has been partially automated by METAS to enhance the reproducibility in aerosol generation. With the MSC it has been possible to generate soot particles coated with secondary organic matter in a stable manner, mimicking thus a process known as atmospheric ageing. Moreover, METAS have designed a new cylindrical flow tube homogeniser for mixing the primary aerosols (i.e. fresh and aged soot, inorganic salt and dust particles). Validation of the flow tube homogeniser with salt and dust particles has shown that an aerosol homogeneity of about 2% in number concentration can be achieved at the different aerosol sampling ports. Bimodal size distributions that simulate the accumulation and coarse mode of ambient aerosols can be generated in a stable and reproducible manner. A workshop on the calibration of PM monitors was held at METAS in January 2020, with participation from project partners BAM, DFM, DTI, NPL and LNE. Automated PM monitors, such as the DustTrak DRX 8533, the Fidas Frog and the TEOM 1405, were successfully

calibrated with respect to the reference gravimetric method under three different simulated environmental scenarios (i.e. different temperature, relative humidity and aerosol chemical composition). The expanded relative uncertainty in the reference PM_{10} mass concentration lied in the 6 -12% range. Overall, this objective was fully achieved.

Objective 2: Methods for the analysis of major components of particulate matter and metals

Project partners have taken part in two inter-comparisons of EC/OC measurements, both were jointly organised by AQUILA and ACTRIS for EU air quality reference laboratories. Promising results were obtained, for example calibration results were within 1.5% of the assigned value, when three reference laboratories had values of > 10%. NPL is actively contributing towards extending the scope of the CEN EC/OC standard. A systematic evaluation of methods for Total Carbon calibration has been made by the project partners. New data has also been generated relating to carbonaceous emissions from wood burning and candles, including a comparison of EC and black carbon. In a comparison of anion analysis capabilities, the consortium participated in regular rounds of the Air and Stack Emissions Proficiency Testing Scheme AR024 run by LGC, and the results have been used to evaluate and reduce uncertainties. An Aeromet-organised comparison for cations (lithium, sodium, ammonium, magnesium, potassium and calcium) included 5 non-Aeromet AQUILA participants. Of the 132 reported results, only 6 deviated from the assigned value by more than 20%, the largest deviation being 38.5%. Project partners participated directly and indirectly in an AQUILA comparison for regulatory metals. The usability of these results was limited due to low concentrations and high blank values, however, AEROMET partners have done further comparative work independently. Among the regulated materials, X-ray fundamental parameters have been determined for As (arsenic), so that the quantification of this element is more reliable and accurate, when applying X-ray fluorescence analysis free of calibration samples. XRR measurements were realized in that respect and a method to compare these results with theoretical calculations is being currently developed. This objective has been fully achieved.

Objective 3: Establishment of specific calibration procedures for MPSS for ambient measurements and the provision of calibration facilities for CPCs to the CEN standard FprCEN/TS 16976

A report describing the state-of-the-art knowledge of the analysis of particles number size distribution based on MPSS and traceability scheme for MPSS has been finalized. Work on standardization committees (CEN and ISO) and observational networks (ACTRIS and GAW) is on track. A review of ISO 15900:2009 “Determination of particle size distribution of differential electrical mobility analyser” is finished and the discussion on provision of calibration facilities for support is successfully evolving. The paper Wiedensohler et al., 2018 served as basis for the technical report and SOP. All partners continued and will continue their participation in the CEN and ISO committee meetings – usually at least two per year and committee - related to MPSS. A WCCAP workshop was held during a pilot intercomparison of reference CPC of all partners, including NMIs outside of Europe. Intensive experimental studies have been performed with different flow rates to optimize the primary SI-traceable reference instrument (FCAE) for lower particle number concentration (<1000 particles cm^{-3}). These suggestions are brought to atmospheric communities such as ACTRIS and WMO-GAW that they become internationally accepted. Four European laboratories (TROPOS. NPL, BAM, and LACP) have been selected for the provision of calibration facilities for MPSS and CPC to establish permanent calibration set-up for the CPC CEN standard FprCEN/TS 16976 and the MPSS CEN standard FprCEN/TS 17434. This objective has been fully achieved.

Objective 4: Quantifying airborne particle compositions in the field and development of traceable and reliable x-ray analytical techniques for chemical analysis of airborne particle

A comparison report is available and describes the recalibration of mobile TXRF spectrometers for the analysis of cascade impactor samples by means of an XRF lateral scanning (mapping) of the entire sample performed using PTB instrumentation at the synchrotron radiation facility BESSY II. The recalibration considers deposition patterns which are specific for the cascade impactor type used. The application of GIXRF, with the use of radiometrically calibrated instrumentation at well-characterised beamlines and knowledge on atomic and instrumental fundamental parameters, on the block copolymers (BCPs) layers infiltrated with alumina Al_2O_3 allowed performing quantification of the mass deposition of target elements of the infiltration (i.e. aluminium and carbon) without the employment of any reference material or calibration sample. Theoretical modelling of GIXRF was continued using two solvers, FDTD solver Gsvit (http://gsvit.net) for near-field optical phenomena and ray-tracing solver Xray-trace (http://nanometrologie.cz/xraytrace) for larger geometries via geometrical optics. Both solvers were optimized for computation on graphics cards to significantly speed up numerical calculations. The solvers are capable of handling roughness, either via its simulation or as measured using some surface measurement technique. To validate this approach, a set of reference structures manufactured

by PTB was measured by Atomic Force Microscopy and numerical model was set up. The results were compared with GIXRF data measured by PTB. A paper has been prepared together with PTB.

GIXRF experiments were performed as well on aerosol samples collected with one of the cascade impactors used during the Budapest and Cassino field campaigns (Task 4.1). The reference-free quantification scheme was used to quantify in absolute and physically traceable manner different elements like Na, Mg and Al collected on the sampling substrates in the TXRF regime as well as in the XRF regime. Furthermore, the chemical binding state of the sampled aerosols was investigated on carbon and nitrogen by means of X-ray absorption spectroscopy. Samples collected near wood-burning, coal-burning and diesel exhaust sources, as well as ammonium sulfate, sodium nitrate and calcium nitrate particles deposited on Si wafers were investigated for comparison. Different reference samples for each element were measured as well in order with the aim to establish a chemical traceability chain. This objective has been fully achieved.

Impact

The project partners have given 40 presentations and exhibited 17 posters, at European and International conferences. The consortium prepared 17 publications, 13 of these have been published and the rest have been drafted and/are submitted to peer-reviewed journals. In 2017 and 2019, the project organised 5 workshops on key project outputs (such as i) the Operational use of Mobility Particle Size Spectrometers, ii) the Comparison of FCAEs and the determination of diffusion losses at small particle sizes). The project results were also mentioned in 8 trade journals in various countries (such as Greece, Denmark, Hungary, France, Spain and the United States). During its lifetime the project gained 12 collaborators and stakeholders, moreover, the consortium regularly updated the project website to disseminate results and maximise impact.

Impact on relevant standards

Partners have been contributing to many European and international standard committees. Metrological advice relating to PM, EC/OC, anions and cations, metals, and particle number concentration and sizing for regulatory purposes has already been disseminated to CEN Working groups and other relevant bodies. For example, advice on total carbon (TC) calibration was passed on at the AQUILA (Association of European Air Quality Reference laboratories) meeting in February-March 2018. Advice on more general aspects of aerosol particle number concentration and sizing measurements is being provided through ISO TC 24. After a brief introduction of the AEROMET project during the ISO meeting in Oct. 2018, members of two working groups on aerosol metrology (WGs 9 “Single Particle Light Interaction” and 12 “Electrical mobility and number concentration analysis for aerosol particles”) of ISO TC 24/SC 4 “Particle Characterization” have expressed their deeper interest in the AEROMET project methods and results. The outcomes of the work on the generation of reference aerosols has been seen to directly correspond to future work items of WG9 while WG12 is interested in the calibration procedures for MPSS including the calibration facilities of CPCs and quantitative in field analysis. An update on the AEROMET project with emphasis on this work was presented to TC24/SC4, WGs 9 and 12 during the ISO meeting in Graz, Austria, in April 2019. Within the research in WP3 a knowledge gap concerning the quantification of sub 30 nm particle losses in inlets of Faraday Cup Aerosol Electrometers could be closed. The result will be presented to the ISO working group and considered for the next revision of the respective ISO standard (ISO27891) for the calibration of Condensation Particle Counters (CPC). The x-ray analytical results of the project had been presented to ISO TC201 SC10.

Impact on industrial and other user communities

Broad stakeholder interests were addressed in the regular project meetings (June 2017, February 2018, November 2018, September 2019) by means of dedicated presentations and related Q&A sessions. A stakeholder committee was formed during the first stakeholder meeting. The developed and successfully demonstrated methodology for aerosol sampling and TXRF analysis has initiated development activities at least by one manufacturer for XRF instrumentation towards automated particle collection and analysis devices. The MPSS of the participating institutes, such as the national metrology institutes from Europe, Russia, Korea, China, India, and Japan, University Lund and Aarhus have been upgraded and the representatives have been trained to evaluate the data. Feedback of the calibration workshop has been given to the manufacturers, as TSI Inc. and GRIMM Aerosol Technik Ainring GmbH & Co. KG. The harmonised decisions about the MPSS calibration among AEROMET, WMO-GAW, ACTRIS, ISO and CEN working groups are beneficial for the manufacturers as well for the community so that there is more consistent and comparable data available in the future.

Impact on the metrology and scientific communities

Aerosol metrology is included in the CIPM Consultative Committee framework through CCQM's Gas Analysis Working Group (GAWG). The early focus within GAWG has been on particle number concentration and particle charge concentration, as covered in the project, and discussed at GAWG meetings in BIPM (April 2018), in Mexico (October 2018), and Paris (April 2019). 39 presentations have been made to predominantly scientific audiences since the start of the project, including at the International Aerosol Conference in St Louis in September 2018 and the European Aerosol Conference in Gothenburg in September 2019. In order to enhance the dissemination of the AEROMET project in Europe the consortium has decided to arrange for four dedicated project sessions at the European Aerosol Conference (EAC) 2019, the 11th International Conference on "Instrumental Methods of Analysis" IMA 2019 in Ioannina, Greece, as well as at the RSC 2019 meeting in London and the E-MRS 2020 spring meeting symposium ALTECH. The dedicated sessions at EAC 2019 and IMA 2019 received about 150 attendees each.

Two dedicated workshops presenting the AEROMET project with emphasis on work on the quantification of airborne particles in the field were held at the University of Cassino (02-28-2018) and the Hungarian Academy of Sciences (05-25-2018). The workshops were attended respectively by local air quality experts of the Italian environmental agency "ARPA Lazio" and the Hungarian Meteorological Service, operator of the Hungarian Air Quality Network. Other representatives such as the head of the Department of Civil and Mechanical Engineering of the University of Cassino and the president of the Hungarian Aerosol Society as well as scientists from the departments of University of Cassino and research centres of Hungarian Academy of Sciences also attended and expressed their interest.

Longer-term economic, social and environmental impacts

The project results will provide relevant methodological improvements and innovations in ambient aerosol monitoring in order to overcome existing deficiencies in the analysis of PM, (i.e. the quantification of aerosols and other air pollutants, sizing and quantitative chemical analysis of airborne particles). More accurate, standardised and traceable routine measurement techniques, which are flexible in time and space, will also bring a better understanding to the formation, evolution and removal of PM and gaseous pollutants in the environment. This will have an important impact on the design of targeted actions to minimise adverse effect on human health and climate change. Various stakeholder communities are directly involved in the mitigation of environmental problems related to air pollution and they will directly benefit. Additionally, a wider environmental impact has also been generated for relevant CEN standardisation and ISO projects. The main economic impact of the project results is its contribution to the prevention, or at least the reduction, of future costs for the compensation of environmental damages. Further economic impacts will be achieved through the availability of relevant new information for upcoming applications with regard to ambient air quality e.g. automotive brake dust tests, friction and wear tests, filter performance tests, particle emission from consumer products, additive manufacturing and other industrial processes. Social impact in this project has been generated by analysing adverse effects on human health and climate change using accurate and traceable measurement techniques that can bring quantitative understanding to the formation, evolution and removal of particulate matter from the atmosphere. Typical uncertainties are expected to reduce from 25 % to 15 % for $PM_{2.5}$ and from 40 % to 25 % for metals. This project has supported the improvement of EU environmental measures by creating awareness to revise existing EU directives.

List of publications

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4. Real-time pollen monitoring using digital holography, Eric Sauvageat, Yanick Zeder, Kevin Auderset, Bertrand Calpini, Bernard Clot, Benoît Crouzy, Thomas Konzelmann, Gian Lieberherr, Fiona Tummon,

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7. Facility for production of ambient-like model aerosols (PALMA) in the laboratory: application in the intercomparison of automated PM monitors with the reference gravimetric method; Horender, S., Auderset, K., Quincey, P., Seeger, S., Skov, S. N., Dirscherl, K., Smith, T. O. M., Williams, K., Aegerter, C. C., Kalbermatter, D. M., Gaie-Levrel, F., and Vasilatou, K; Atmos. Meas. Tech.;14, 1225–1238; https://doi.org/10.5194/amt-14-1225-2021
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9. Interaction of nanoparticle properties and X-ray analytical techniques; Rainer Unterumsberger, Philipp Hönicke, Yves Kayser, Beatrix Pollakowski-Herrmann, Saeed Gholhaki, Quanmin Guo, Richard E. Palmer & Burkhard Beckhoff; J. Anal. At. Spectrom; (2020) 35, 1022-1033 https://doi.org/10.1039/D0JA00049C; https://arxiv.org/abs/2004.02955
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This list is also available here: https://www.euramet.org/repository/research-publications-repository-link/

Project start date and duration:	June 2017, 40 months	
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Internal Funded Partners: 1 PTB, Germany 2 BAM, Germany 3 CMI, Czech Republic 4 DFM, Denmark 5 INRIM, Italy 6 LNE, France 7 NILU Norway 8 NPL, United Kingdom	External Funded Partners: 9 CIEMAT, Spain 10 DTI, Denmark 11 FORCE, Denmark 12 IRSN, France 13 JSI, Slovenia 14 LUND, Sweden 15 MTA EK, Hungary 16 NTUA, Greece 17 TROPOS, Germany 18 UNICAS, Italy 19 UPO, Italy	Unfunded Partners: 20 Bruker, Germany 21 METAS, Switzerland
RMG1: INRIM, Italy (Employing organisation); PTB, Germany (Guestworking organisation)		