



D4.8.3: Report on the aerosol analysis made at INFN Firenze [B17]



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1. INTRODUCTION

This deliverable is prepared in the context of the ITINERIS project, within the Work Package 4, Atmosphere, that deals with the integration of Research infrastructures working in the atmospheric domain through synergistic approaches and cross boundaries developments.

The aim of this document is to describe the analysis of aerosol particle samples for external users under the transnational access scheme of ITINERIS–ACTRIS Access Pilot Call carried out at LABEC laboratory at INFN Firenze using Ion Beam Analysis (IBA) techniques and the data produced. This is in the context of 100% digital outcome of the ITINERIS project. The document is structured in five (5) different sections. After this introduction, an overview of the LABEC laboratory at INFN Firenze and its role in ACTRIS ERIC, the European Aerosol, Clouds, and Trace Gases Research Infrastructure Consortium, is reported in section 2. Section 3 reports aerosol analysis made at INFN Firenze under the ITINERIS–ACTRIS Access Pilot Call. Finally, section 4 and 5 report the procedures for recruiting personnel and procuring equipment, respectively, for the achievement of aerosol analysis made at INFN Firenze under the ITINERIS–ACTRIS Access Pilot Call. Annexes and references are reported at the end of the document.

2. INFN FIRENZE LABEC LABORATORY AND ITS ROLE IN ATMOSPHERIC RIS

The INFN Firenze LABEC laboratory (“Laboratorio di Tecniche Nucleari per l’Ambiente e i Beni Culturali”, Laboratory of nuclear techniques for the Environment and the Cultural Heritage) is located in the scientific campus of the University of Florence (Italy). LABEC is a high-qualified centre for the development of new technologies based on particle accelerators and ionizing radiations, and for their applications in environmental contexts and in heritage science [1]. The LABEC laboratory was born in 2003 with the installation of a “small” particle accelerator, namely a 3.0 MV Tandatron produced by High Voltage Engineering Europe, HVVEE, and stems from more than thirty years of experience of the research group members with accelerator-related analytical techniques. Accelerated particles are used to characterize the elemental composition of aerosol samples collected on filters by Ion Beam Analysis (IBA) techniques. IBA are a suite of analytical techniques that exploit the detection (and analysis) of radiation such as X-rays and gamma-rays, or charged particles, as emitted in the interactions obtained by the bombardment of the sample with an accelerated particle (usually proton) beam. Among IBA, the particle-induced X-ray emission (PIXE) technique has been widely used for the study of the aerosol composition [2], since up to 20 key elements (from Na to Pb) including important anthropogenic elements (S, V, Ni, Cu, Zn, As and Pb) and all the crustal elements (Al, Si, K, Ca, Ti, Mn and Fe) can be detected in only few minutes of measurement. Moreover, PIXE is sensitive to concentrations down to $\mu\text{g/g}$, it is highly quantitative (few %) and traceable, and it is a non-destructive technique, so further measurements with other complementary techniques can be carried out on the same samples. The analysis of the elemental composition of aerosol samples gives important information on several markers that may help in disentangling the contributions of different atmospheric aerosol sources.

Typically, aerosol sampling and analysis are carried out using 24-h averaged data, in order to have sufficient quantities of material to perform a chemical characterization as complete as possible, and thus to obtain indications on the predominant sources of atmospheric aerosol. However, most aerosol emissions as well as atmospheric dilution processes change within a few hours thus daily sampling cannot track these rapid changes. High-time resolution resolved measurements (i.e. less than few hours) require suitable techniques both for particle sampling and analysis. For time-resolved sampling one of the most suitable solutions is to use a system that automatically switches the particle collection substrate, for example every hour, to obtain a sequence of high-time resolution collected aerosol spots/deposits. However, this leads to a large number of “small” (as for mass and deposit area) collected samples to be analyzed. In this framework, PIXE technique is an optimal solution for offline, laboratory-based-measurements. The high-time resolution measurements of elements are currently carried out sampling the aerosol with special high time-resolution samplers (Size and Time Resolved Aerosol Sampler, STRAS [3]) designed by INFN, followed by offline PIXE analysis at LABEC of the aerosol deposits on a collecting polycarbonate membrane [4].

LABEC hosts the ECAC-CAIS unit Elemental Mass Calibration Centre (EMC2) of the CAIS-ECAC (Center for Aerosol In-Situ - European Center for Aerosol Calibration and Characterization) of ACTRIS ERIC [5]. The main goal of EMC2 in ACTRIS ERIC is: providing proficiency tests of individual laboratory analysis through the organization of inter-comparison and round-robin exercises; operation support for quality assurance and quality control for the measurement of mass concentration of particulate heavy metals and inorganic elements by both offline laboratory measurements (filter-based) and online, near-real time, in-situ measurements; hands-on training of operators and scientists from ACTRIS user community. EMC2 will work with the aim of harmonizing the analyses of the elemental composition of atmospheric aerosol samples, in order to make data from different atmospheric observatories fully comparable.

IBA techniques provide important information about the elemental composition of aerosol samples, but they do not fully capture the chemical composition and must be supplemented, for example, with measurements of the various chemical forms of the carbonaceous component present in the particulate matter, which often constitutes the majority fraction (up to 50% by mass) in urban environments. In this regard, a key distinction is between so-called "elemental" carbon and "organic" carbon. The production mechanisms and atmospheric emission sources of these two fractions differ: elemental carbon is essentially carbon in a graphitic form, refractory to combustion, and dark-black in color, whereas organic carbon refers to all organic components, typically lighter in color, consisting of compounds where carbon is bonded to hydrogen, oxygen, nitrogen, etc. Thermal-optical analysis [6] is a standard method for determining the carbon concentration in aerosols collected on quartz fibre filters and discriminating the total carbon into the two OC and EC components. This methodology is recommended in the CEN standard - EN 16909:2017: “Ambient air - Measurement of elemental carbon (EC) and organic carbon (OC) collected on filters”, and it is recommended by ACTRIS ERIC too.

The list of current available instruments (including status, repository where data are stored, link to data and metadata example) is included in the ANNEX 1.

3. AEROSOL ANALYSIS MADE AT INFN FIRENZE UNDER THE ITINERIS–ACTRIS ACCESS PILOT CALL

The activity of INFN Firenze will be focused to analyse aerosol particle samples for external users under the transnational access scheme of ITINERIS–ACTRIS Access Pilot Call carried out at LABEC laboratory using Ion Beam Analysis (IBA) techniques and the data produced. In the following details and statistics on the proposed experiments will be presented.

A total of nine proposals were submitted and approved for being performed at LABEC. All 200 hours of beamtime made available at LABEC were assigned for a total of more than 1400 aerosol particle samples to be measured by PIXE technique. The implemented hours of beamtime, as shown later in a chart, resulted in a lower number, since the increased measurement capabilities at LABEC allowed to analyse that number of samples in less time than initially foreseen.

Table 1 – Details and statistics of the proposed experiments for access at LABEC.

<i>Proposer name</i>	<i>Proposal nr.</i>	<i>Country</i>	<i>Scheduled hours</i>	<i>Implemented hours</i>	<i>Modality</i>
Manale Noun	0000000001	Lebanon		20	presence
Teresa Laurita	0000000034	Italy		10	virtual
Francesco Cardellicchio	0000000026	Italy		10	virtual
Roberto Fraile	0000000014	Spain		16	virtual
Manale Noun	0000000035	Lebanon		5	virtual
Hanan Sa'adeh	0000000131	Jordan	50		virtual
Manale Noun	0000000161	Lebanon		27	presence
Ketlyn Oliveira	0000000189	Portugal		15	virtual
Joana Lage	0000000152	Portugal	15		presence

The gender balance of the proposers was 22% male and 78% female.

In terms of hours of beamtime, 27% were devoted to proposals by EU users (Spain and Portugal), 12% by Italian users (CNR) and 61% by non-EU users (Lebanon and Jordan), testifying the capability of attracting users from developing or less performing countries in the Mediterranean region where the studies on air quality are urgent but the analytical capabilities are still lacking or yet to be built.

In terms of hours of beamtime, 37% were assigned to in-presence/hands-on measurements and 63% to virtual/hands-off measurements.

In terms of hours of beamtime, 36% were assigned to proposals submitted in 2024 and 64% to proposals submitted in 2025. It has to be noted that the ITINERIS–ACTRIS Access Pilot Call started in July 2024 and experiments had to be completed by July 2025, so only one year was actually available for the whole process from proposal submission, approval, scheduling and finally running the measurements.

At the end of the ITINERIS–ACTRIS Access Pilot Call the situation in terms of the 200 hours of beamtime initially allocated is represented in the following chart.

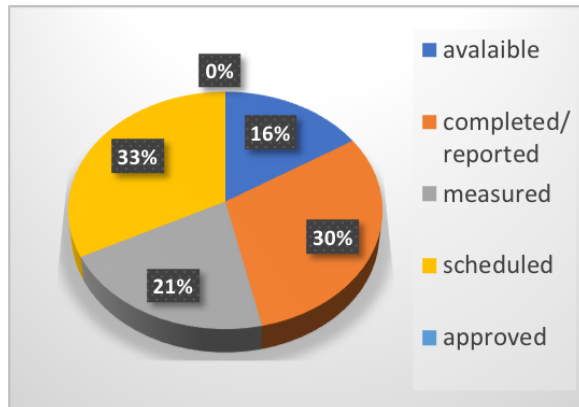


Figure 1 – Breakup of the 200 hours of beamtime of access at LABEC.

As regards the experiments that have been scheduled, but the access could not be completed within the ITINERIS–ACTRIS Access Pilot Call timeframe, the proposal nr. 0000000152 from Joana Lage, Portugal, scheduled in July 2025, could not be implemented and carried out due to external causes, samples shipping problems (the samples could not be delivered in time for the scheduled measurements), whereas the proposal proposal nr. 0000000131 from Hanan Sa’adeh, Jordan, scheduled in June 2025, could not be implemented and carried out due to external causes, accelerator malfunction (power diodes failure) and forced stop from the effect of a sudden blackout due to excessive heat in the area. However, these experiments will be rescheduled and the measurements will be anyway carried out at LABEC later on.

Here brief descriptions of the nine projects follow.

The proposal nr. 000000001 from Manale Noun of the Lebanese Atomic Energy Commission in Beirut (Lebanon) dealt with PIXE analysis of approximately 250 PM2.5 aerosol samples collected in Beirut between 2020 and 2024. Sampling occurred twice a week, on Wednesdays and Sundays, over 24 hours in urban areas, with an air volume of about 55 m³. Expected pollution sources include natural and anthropogenic origins such as dust storms, fires, vehicles, waste burning, and diesel generators. The elemental concentrations from PIXE together with the PM mass and Black Carbon data will be used to determine air pollution source profiles for Beirut and assess their regional impact in an eastern Mediterranean area where, despite increasing awareness, there is still a lack of studies on this topic as compared to western countries.

The proposal nr. 000000014 from Roberto Fraile of the University of León (Spain) dealt with the elemental analysis by PIXE of samples from 1-year PM10 measurement campaign (2023-2024) carried out in an area in the center of León (Spain) where the use of residential coal combustion devices is still very frequent. In order to obtain a complete characterization of the aerosol, many instruments were simultaneously measuring parameters such as the aerosols size distribution, the concentration of black carbon and total carbon, the aerosol light scattering and the chemical composition through the analysis of the collected filters. The main aim is to investigate the effects of coal combustion emissions from domestic devices on health and climate.

The proposals nr. 000000026 and 000000034 from Francesco Cardellicchio and Teresa Laurita from the CNR-IMAA institute in Potenza (Italy) had the objective of characterizing the composition of atmospheric particulates sampled on PTFE filters at the CIAO observatory (CNR IMAA Atmospheric Observatory) with PIXE technique and comparing the chemical data obtained through PIXE with those obtained from the in-situ aerosol instruments present in the CIAO observatory.

The proposal nr. 000000035 from Manale Noun of the Lebanese Atomic Energy Commission in Beirut (Lebanon) dealt with the study of how conflicts like the recent bombardments in Lebanon and the resulting fires impacted air quality in a region where air pollution is already a leading global health concern. This study examined elemental composition of PM2.5 aerosol samples collected in September 2024 from two Beirut sites, near and far from bombardments.

The proposal nr. 000000131 from Hanan Sa'adeh of the University of Jordan in Amman (Jordan) dealt with using PIXE technique to accurately quantify the elemental content in atmospheric particulate matter in Amman. This in turn will help the proposers identify the potential sources of air pollution in Amman. The obtained results will be a step forward in the understanding of Amman's atmosphere and eventually they will broaden the horizons towards monitoring and understanding the quality of the breathed air.

The proposal nr. 000000152 from Joana Lage of the Instituto Superior Técnico (IST), University of Lisbon in Lisbon (Portugal) dealt with the elemental characterization of PM2.5 filter samples collected in indoor bedrooms and their corresponding outdoor environments in 30 selected dwellings to determine infiltration levels, with the goal of assessing the population's integrated exposure to air quality and its respective health impact, with a particular focus on sleep. Given the potential health risks associated with PM2.5 exposure, these data are of great importance for subsequent source apportionment analysis, contributing to the development of the scientific knowledge, filling an identified gap of the scientific literature on the impact of indoor air quality on sleep.

The proposal nr. 000000161 from Manale Noun of the Lebanese Atomic Energy Commission in Beirut (Lebanon) dealt with the PIXE analysis of approximately 400 PM2.5 aerosol samples collected from Beirut City and selected Mediterranean countries between 2023 and 2025 to investigate the elemental composition and to identify pollution sources in Beirut and other Mediterranean regions, contributing to source apportionment studies. Analyzing samples from different countries in the Mediterranean region offers a broader perspective on air pollution dynamics, enabling comparisons between urban and rural areas and highlighting transboundary pollution effects.

The proposal nr. 000000189 from Ketlyn Oliveira of the Instituto Superior Técnico (IST), University of Lisbon in Lisbon (Portugal) dealt with PIXE measurements of PM2.5 sample collected from indoor and outdoor environments, from five primary school classrooms, one gymnasium and five private houses, during occupied periods in winter and summer seasons.

The plan is to correlate the detailed elemental composition of airborne particulate matter with toxicological assays, to understand the mechanisms that underlie numerous health problems, including respiratory and cardiovascular disorders, allergic reactions, cognitive deficits, and increased cancer risks in children exposed to airborne particulate matter and offer actionable insights for air quality management and health policy.

The ITINERIS scientific team working on aerosol analysis is composed by Massimo Chiari, Giulia Calzolari, Cosimo Fratticioli, Fabio Giardi, Franco Lucarelli and Silvia Nava.

4. PROCEDURES FOR THE PERSONNEL

No personnel have been hired specifically for this activity of aerosol analysis made at INFN Firenze within the ITINERIS–ACTRIS Access Pilot Call under the ITINERIS budget. The activity of aerosol analysis is part of the PNRR PhD project of a student (Cosimo Fratticioli) funded by ITINERIS.

5. EQUIPMENT PROCEDURES

No specific equipment has been purchased specifically for this activity of aerosol analysis made at INFN Firenze within the ITINERIS–ACTRIS Access Pilot Call under the ITINERIS budget.

ANNEX 1 –AVAILABLE INSTRUMENTS AT INFN FIRENZE LABEC LABORATORY

- Tandem accelerator (3.0 MV terminal voltage HVEE Tandetron) with 5 IBA beamlines and 5 measuring end stations. The accelerator is equipped with three independent ion sources, two of which (a single-sample Cs-sputtering and a Duoplasmatron) are used to produce all kind of beams (from protons to heavy ions), mainly for applications of Ion Beam Analysis.
- External proton milli-beam set-up high-current/high-throughput PIXE and PIGE analysis of atmospheric aerosol samples (PM_x samples, size-segregated samples, high time-resolution samples) collected on any type of substrate (Teflon, Nuclepore, Quartz fibre...) and by a large variety of commercial samplers.
- External scanning proton microprobe set-up with detectors for PIXE, PIGE and RBS/EBS analysis; the focusing system is an Oxford Microbeam quadrupole doublet and the scanning is achieved by magnetic and mechanical scanning.
- Pulsed beam facility for irradiation of devices in-vacuum or in-air with bunches of ions (ranging from proton to oxygen), counting from 1 ion to a few thousand ions per bunch, at a repetition rate of the bunches from single shot to a few kHz.
- Multi-purpose in-vacuum scattering chamber equipped for PIXE, PIGE and RBS/EBS analysis.
- External proton sub-milli-beam set-up for low-current PIXE, PIGE and RBS/EBS analysis of thick and bulk samples, such as cultural heritage objects.
- Analytical grade micro-balance with automated filter weighing (Sartorius) installed in a controlled environment laboratory for gravimetric analysis of atmospheric aerosols.
- Laboratory ECOC thermo-optical analyser (Sunset Lab Inc.) for filter-based measurements of the Elemental and Organic carbonaceous fractions in atmospheric aerosol samples;
- Laboratory ECOC thermo-optical analyser (Sunset Lab Inc.) with dual mode optics, NDIR CO₂ detector and autosampler for high-throughput filter-based measurements of the Elemental and Organic carbonaceous fractions in atmospheric aerosol samples.
- Field ECOC thermo-optical analyser (Sunset Lab Inc.) for near-real time measurements of the Elemental and Organic carbonaceous fractions in atmospheric aerosol samples with a time resolution down to 1 hour.
- Commercial “Epsilon 5” ED-XRF spectrometer (Panalytical) in polarizing geometry for elemental analysis of aerosol (the spectrometer is currently not operational and needs refurbishing, and software and hardware upgrades; however, since the instrument will be phased out by the manufacturer in the next years, it is planned to purchase a brand new spectrometer, whose cost would be roughly equivalent to refurbishing the existing one).
- XACT 625i Ambient Metal Monitor (SaliBri Cooper, formerly Cooper Environmental) for automatic in-situ sampling and XRF analysis of particulate elemental matter, such as metals and dust.

- Two (2) double channels sequential samplers for PM10, PM2.5 or PM1 (Dado Lab Gemini).
- Two (2) single channels sequential samplers for PM10, PM2.5 or PM1 (TCR Tecora Skypost).
- Three (3) Streaker high-time resolution aerosol samplers (PIXE International) for collection of the fine and coarse aerosol fractions with hourly resolution (instrument out of production).
- Two (2) STRAS (Size and Time Resolved Aerosol Sampler) high-time resolution aerosol samplers (designed by INFN) for collection of the fine and coarse aerosol fractions with hourly resolution.
- Multi-stage cascade impactor (Dekati SDI, Small Deposit Impactor) for the collection of aerosols in 12 dimensional classes from 40 to 8 μm (from nanoparticles to PM10).

Datasets produced by analysis of aerosol particle samples by PIXE and other IBA techniques, as well by other complementary techniques at LABEC (Thermo-Optical, XRF...) are stored in the INFN Open Access Repository (INFN OAR):

- <https://www.openaccessrepository.it/>

under the Creative Commons Attribution 4.0 license. The datasets are accessible and reusable.

Stored datasets include, at least:

- Experiment title
- Contributing authors (external and local)
- Description of the experiment
- Funding institution (grant)
- Scientific community of reference
- Raw data file (measured samples and reference standards)
- Scanned copy of the logbook (pdf)
- Spectra files converted for the dedicated IBA analysis codes
- Photos of the measured samples (if any)
- Plot of the temperature in the accelerator hall during the measurements

An example can be found, amongst others, at the repository link <https://www.openaccessrepository.it/record/77163> (DOI 10.15161/oar.it/77163)

REFERENCES

1. M. Chiari, S. Barone, A. Bombini, G. Calzolari et al., “LABEC, the INFN ion beam laboratory of nuclear techniques for environment and cultural heritage”, *Eur. Phys. J. Plus* 136 (2021) 472.
2. F. Lucarelli, G. Calzolari, M. Chiari, S. Nava, L. Carraresi, “Study of atmospheric aerosols by IBA techniques: The LABEC experience” *Nucl. Instr. and Meth. B* 417 (2018) 121
3. S. Nava, R. Vecchi, P. Prati, V. Bernardoni et al., “S. Nava, R. Vecchi, P. Prati, V. Bernardoni et al, "STRAS: a new high time resolution aerosol sampler for PIXE analysis” *Atmos. Meas. Tech.* 18 (2025) 2137
4. G. Calzolari, F. Lucarelli, M. Chiari, S. Nava, M. Giannoni, L. Carraresi, P. Prati, R. Vecchi, “Improvements in PIXE analysis of hourly particulate matter samples” *Nucl. Instr. and Meth. B* 363 (2015) 99
5. <https://www.actris-ecac.eu/>
6. M. Giannoni, G. Calzolari, M. Chiari, A. Cincinelli, F. Lucarelli, T. Martellini, S. Nava, “A comparison between thermal-optical transmittance elemental carbon measured by different protocols in PM2.5 samples” *Science of the Total Environment* 571 (2016) 195