MISSIONE 4
ISTRUZIONE
RICERCA

ITINERIS

D4.4.4: Report on realization of the technological systems, installation, and operability of the instruments at CNR-ISAC Lecce [B16]

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INTRODUCTION

This deliverable is prepared in the context of the ITINERIS project, within the Work Package 4 that deals with the integration of Research Infrastructures working in the atmospheric domain through synergistic approaches and cross boundaries developments. This deliverable reports the progress in the implementation of the WP4.4 activities for integration and harmonization with the Italian Network of Environment RIs. The main aim of the 4.4 activity is the instrumental strengthening of the observational capacity of the facilities in Lecce, in order to reinforce the Italian contribution to ACTRIS. In particular, it is planned to extend the observation capabilities for both online and offline chemical and physical characterization of atmospheric aerosols and gases. The instrumental strengthening includes three aspects: the laboratory for aerosol characterization of ISAC-CNR in Lecce; the exploratory platform MAGA; the Environmental-Climate Observatory (ECO).

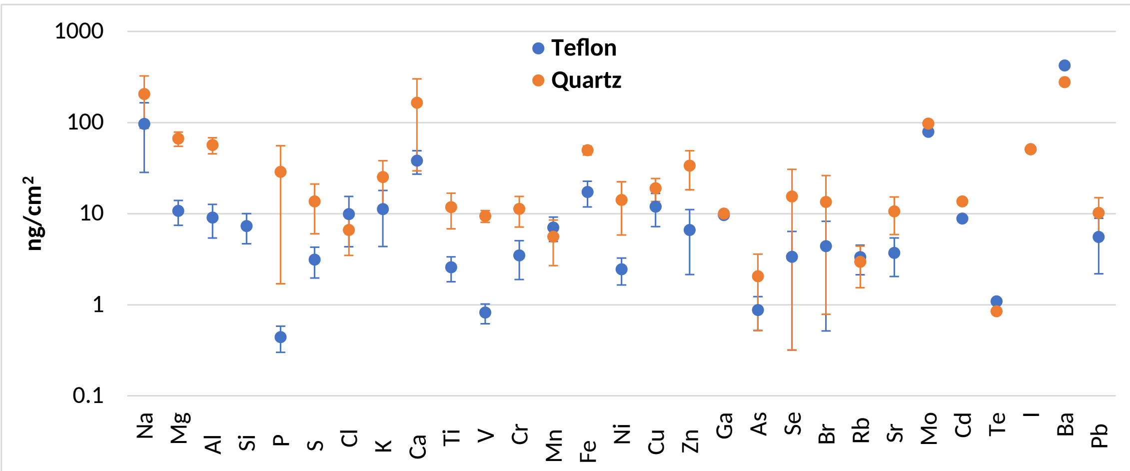
This document is structured in four different chapters: the introduction, and three chapters dealing with the three above-mentioned aspects. Also instruments not yet installed and operative at the time of writing are reported in this document.

STATUS AND OPERATIVITY OF THE INSTRUMENTS INSTALLED IN THE LABORATORY

The instrumental strengthening and operativity of the laboratory for aerosol characterization, was implemented starting from the spectroscopy room. The spectroscopy room of the laboratory (Figure

1) hosts the offline ED-XRF and the UV-VIS analyzer. The ED-XRF was used on samples collected during the ITINERIS campaigns for the activity on identification of aerosol sources for the activity 4.12. Efforts have been devoted to the evaluation of LODs and calibration of ED-XRF on quartz and Teflon samples using Micromatter standards. A collaborative work has been done between ISAC-CNR (Lecce), ARPA (Milan), IIA-CNR (Rome), UMH (Elche), and INFN (Florence) to intercompare analysis on PM samples of ED-XRF (Unga et al., 2025) and some examples are reported in Figure 2.

Figure 1: Spectroscopy room of the aerosol characterization laboratory at ISAC-CNR in Lecce.



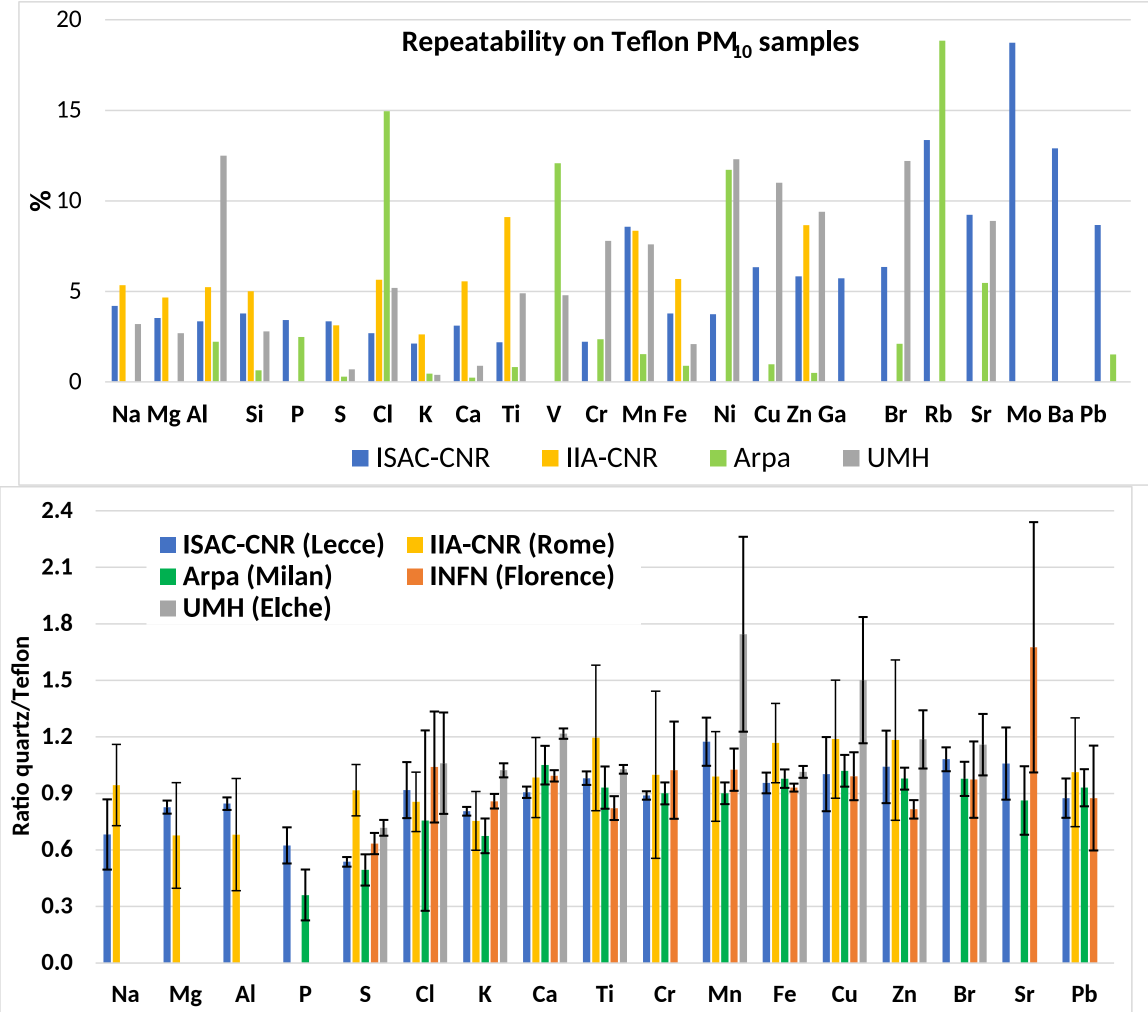


Figure 2: LODs on Teflon and quartz substrates. Repeatability of measurements on Teflon samples. Averages and standard deviations of the C_{quartz}/C_{Teflon} ratios measured by the different laboratories. Pearson coefficients were all above 0.8 with the exclusion of: P (0.72) for Arpa; Cr for INFN (0.72) and IIA-CNR (0.64) with all concentrations lower than 20 ng/cm^2; Cu for ISAC-CNR (0.72) and UMH (0.80); Sr for ISAC-CNR (0.64) and INFN (0.34) with all concentrations lower than 30 ng/cm^2; Rb (0.57, concentrations < 10 ng/cm^2) and Pb (0.49) for IIA-CNR.

The Thermo Scientific Dionex™ HPIC Modular System is a highly configurable ion chromatography (IC) system for the determination of ionic species and carbohydrates in aqueous extracts of particulate matter (PM2.5 and PM10) samples. In particular, the ICS-6000 system has been configured in 2 modules that allow the determination of inorganic and organic anions and simple sugars while the Integrion™ system is dedicated to the detection of alkali and alkaline earth metal cations (Figure 3). After installation, the instrument was made operational by setting up calibration curves and the analyses of aqueous extracts of particulate matter were started.

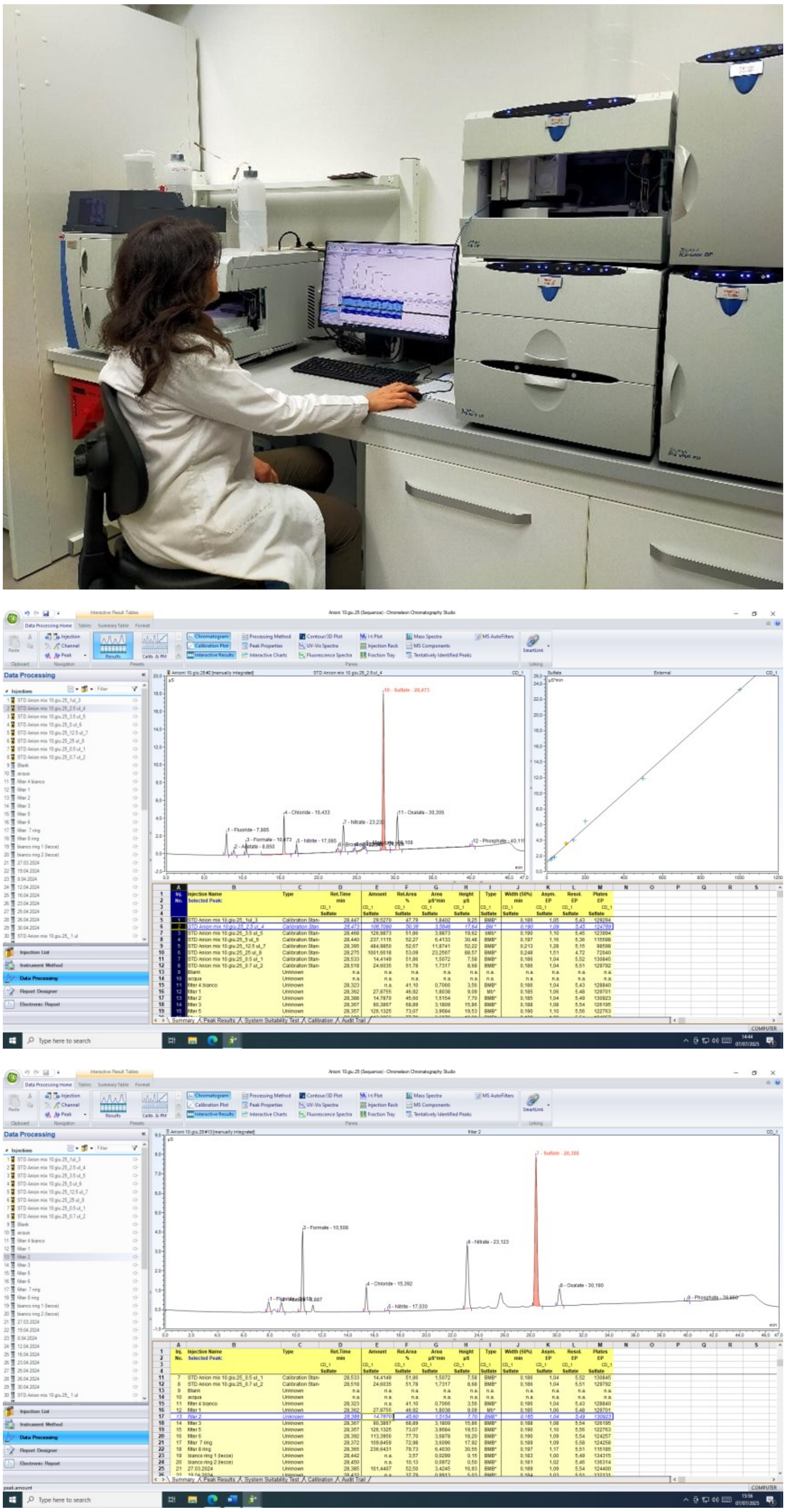


Figure 3: Ion chromatography (IC) system Dionex ICS-6000 HPIC System and Dionex™ Integrion™ HPIC™ System (Thermo Fisher Scientific) and example of calibration spectra.

Figure 4: Refrigerators, ultrasonic bath and analytical microbalance installed and operative at the ISAC-CNR laboratory in Lecce.

The laboratory for aerosol characterization was furtherly equipped (Figure 4) during ITINERIS with two laboratory refrigerators (DESMON) used to store samples (PM filters and aqueous solutions of PM extracts, aqueous solutions of analytical standards); an Ultrasonic Bath (ArgoLab-DU65) and a Bagnomaria used for the extraction of the water-soluble fraction of PM (ions, water-soluble organic and inorganic carbon); an Electronic Balance (Sartorius-Quintix 125D-1S) was installed, used for weighing chemical compounds, e.g. analytical standards, (Potì et al., 2025).

Further equipment (Figure 5) installed and operative in the implementation of action 4.4 of ITINERIS includes: a spectrophotometer (Agilent Cary Series UV-Vis-NIR) used for absorption and reflectance measurements on materials of environmental interest; a stereomicroscope (Nikon SMZ25 H550 L) for observation of particulate matter collected on opportune substrates; DNA sequencers (Avantor Thermocycler XT96 Gradient CYCL08 G) with centrifuge (Avantor MEGA STAR 600R) to be used for characterization of bioaerosol.

Figure 5: Spectrophotometer, stereomicroscope, and DNA sequencers.

A Thermo Fisher Scientific Nicolet ìS50 FTIR system was purchased and installed, tested, and is now fully operational (Figures 6 and 7). Specifically, in addition to the FTIR spectrometer, the various ATR and Raman accessories, the NIR module with Integrating Sphere, the TGA and gas analysis modules, the transmission measurement module, the Vis-IR microscope, and the system management software were installed. The functionality of the two sources (the patented Long Life-Time Polaris infrared source and the Tungsten-Halogen Vis-NIR source) and the three detectors integrated into the spectrometer (a DLaTGS detector with a KBr window at room temperature, a DLaTGS detector with a Polyethylene window, a MCT-A detector with a CdTe window to be cooled in liquid nitrogen, and the two dedicated detectors for Raman and ATR) were verified. The system uses OMNIC Paradigm software which operates automatic analysis to perform spectral searches, quality control or immediate peak identification using the numerous available libraries.

Figure 6: The Thermo Fisher Scientific Nicolet ìS50 FTIR system.

The installed accessories are:

- ATR Module – Total Internal Reflection
  - The ATR module was installed in the right shoulder of the spectrometer and is based on the ATR analytical technique, measuring atmospheric particulate matter samples to study particles up to 100 µm in size. It also provides excellent measurements in the Far IR range thanks to its dedicated detector.
- Raman Module
  - The iS50 Raman module is designed for installation in the sample compartment and adds the analytical power of FT-Raman spectroscopy to the Nicolet iS50 high-performance FTIR spectrometer. The iS50 Raman module is equipped with a 1064 nm laser, a movable stage for Raman spectroscopy, mapping, and multipoint data acquisition, an integrated camera for visualization and collection, and archival visual evidence showing where the data were acquired.
- NIR Module with Integrating Sphere
  - The NIR module was installed on the left side of the NIR spectroscopy system; the accessory uses the iS50's internal Vis-NIR source and interferometer. A series of additional accessories can be installed on top, allowing for the analysis of atmospheric particulate matter samples collected from different matrices (solid and liquid).
- TGA Module – Thermo-Gravimetric Analysis
  - The TGA module consists of a heated transfer line, a heated cell, and a temperature control system. FT-IR spectroscopy combined with thermal gravimetric analysis (TGA) is used to characterize materials by measuring the change in weight of a sample as a function of temperature or time. Accurate measurement of weight loss provides information, but does not identify what is changing. This weight change is usually accompanied by gas evolution caused by sample decomposition. FT-IR spectroscopy is used to identify these evolved gases and help determine sample characteristics.
- Gas Analysis Module
  - The Gas Analysis Module is designed for installation in the sample compartment and allows gas measurement via a long optical path using FT-IR spectroscopy. The optical path is achieved through a vertical configuration and a multipass cell with an optical length of up to 10 m.
- Transmission Module
  - This accessory allows measurements to be made on airborne particulate matter samples on filters, in suspension (for example, using cuvettes), or in gaseous form.
- High-Resolution Vis-IR Microscope – Nicolet RaptIR Plus
  - The RaptIR+ FTIR microscope allows reflection and transmission measurements using appropriate supports, with the aim of identifying airborne particulate matter particles with a resolution of 5 µm, both in terms of size and chemical composition. For observation, samples must be prepared in an ethanol suspension and deposited as a drop or smear on a metal or polyethylene slide. The microscope also features interchangeable detectors to work across multiple spectral ranges.

Among the various measurements performed, we report an analysis of fine atmospheric particulate matter (PM2.5) on a quartz filter, sampled from May 17 to 19, 2024, at the ACTRIS monitoring site of the University of Salento. The analysis revealed that the atmospheric particulate matter deposited on the filter is typical of desert dust, consistent with the back-trajectories.

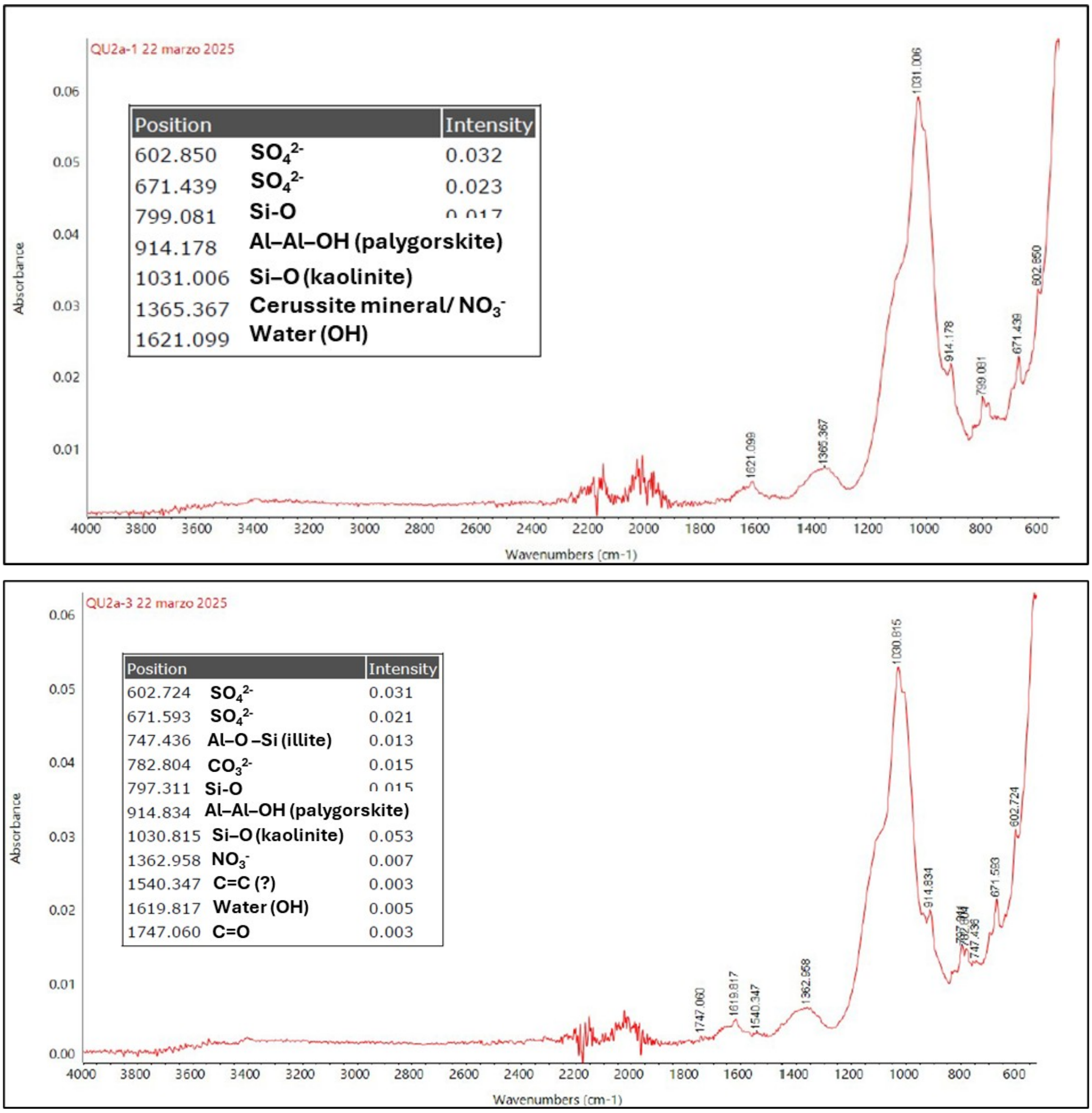


Figure 7: Spettro ed immagine tramite microscopio RaptIR+ FTIR di un campione di particolato atmosferico (PM10).

A Filter-based multi-mode microplate reader-FLUOstar Omega was purchased as part of the ITINERIS project. The activities carried out using this instrument is the development and application of protocols for estimating the oxidative potential (OP) of aqueous particulate matter extracts through acellular assays, comparable to methods reported in the literature, such as the dithiothreitol (DTT) assay and the ascorbic acid (AA) assay. Use of this instrument is advantageous for the multi-sample analysis in multimode microplate combined with the possibility to control the temperature as required for this protocol. Figure 8 includes some examples of applications conducted on samples collected at the Climatic-Environmental Observatory (ECO) of ISAC-CNR in Lecce. The first results obtained for oxidative potential of PM2.5 and PM10 were published (Potì et al., 2025).

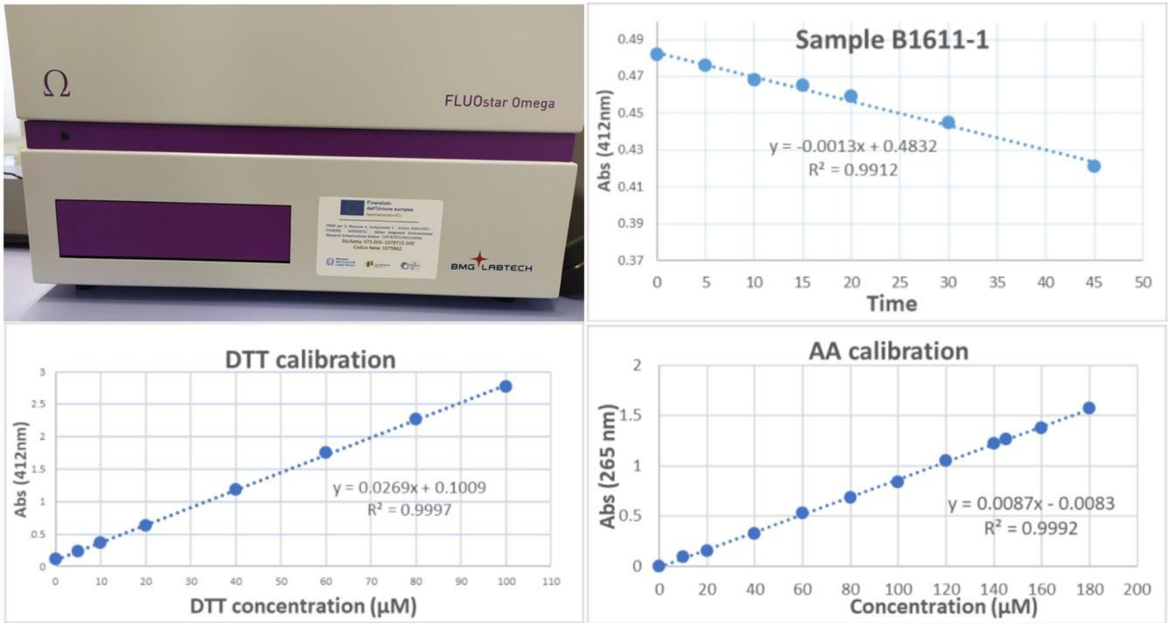


Figure 8:The plate reader and an analysis of absorbance of a real sample (B1611-1); examples of calibration for DTT at different concentrations (from 0 to 100 µM) and for AA at different concentrations (from 0 to 180µM).

STATUS AND OPERATIVITY OF THE INSTRUMENTS INSTALLED IN THE EXPLORATORY PLATFORM MAGA

The upgrades of the exploratory platform MAGA (the mobile laboratory showed in Figure 6 operative during a campaign performed in winter 2024) included the upgrade of the inlet system with a high-volume sampler (Figure 9) and the calibration system for gas analyzers. Specifically, the calibration lines (Figure 11) included zero, span and calibration cylinders for measurements of:

- Carbon Monoxide (CO) in air in trace levels with the Thermo Scientific™ Model 48i-TLE Enhanced Trace Level CO analyzer based on gas filter correlation and NDIR technology;
- Nitrogen Oxides (NO, NO2, NOx) in the air from sub-ppb levels up to 100ppm using chemiluminescence technology with the Model 42i (NO-NO2-NOx) analyzer;
- Sulfur Dioxide (SO2) in ambient air up to 100ppm with the Thermo Scientific™ Model 43i SO2 analyzer, using pulsed fluorescence technology.

The system was used to calibrate the analyzers during the measurement campaign in summer 2024 connected to ITINERIS and TOX-IN-AIR projects. The data collected will be used in the ITINERIS project to identify the main anthropogenic sources (combustions, transport and industry) and natural, local and long-range contributing to atmospheric pollutions.

Figure 9: Pictures of the MAGA platform during a measurement campaign in winter 2024 and of the newly installed high volume sampling system.

Furthermore, an “ad-hoc” dehumidification system for the aerosol inlet of the MAGA station (Figure 10) has been realized and installed. In this case a single aerosol high-volume (140-200 L/min) is used with dehumidification through nafion dryers after the splitter and a system able to quickly rearrange the size and the orientation of nafion when needed. The counter-flows is made with a pump recirculating the conditioned air inside the van after a further dehumidification with silica gel.

A dual channel sampler with capability to directly measure eBC on sampling filters was purchased, tested, and used during ITINERIS implementation (Figure 12). A demo version of this sampler was used during an intensive campaign in spring 2023 in which results of carbon measurements were intercompared with the other reference instruments of the observatory.

Figure 10: Pictures of the dehumidification system installed at the MAGA platform.

Figure 11: Picture of the calibration system installed and operative in the MAGA exploratory platform.

Figure 12: Dual Channel sampler with eBC measurements installed in Rome for a measurement campaign during autumn 2024.

A new SMPS system from the company TSI, ACTRIS compliant has been purchased for the MAGA facility. This will be used for measurement of size distributions and will be installed in August 2025.

A new aethalometer Magee Scientific AE36 was purchased and installed at the ACTRIS site in Lecce for determination of aerosol absorption at different wavelengths (Figure 13).

Two nephelometers AECOM NE300 (serials 24-0463 and 24-0464) have been purchased and installed. The instruments were initially tested and successively calibrated at the ACTRIS-ECAC Workshop in Leipzig (Germany) as shown in Figures 14 and 15.

Figure 13: Aethalometer AE36 operating at the ACTRIS site in Lecce.

Figure 14: the two NE300 at CNR-ISAC in Lecce (Italy) and the instruments at the calibration workshop of ACTRIS at TROPOS in Leipzig (Germany).

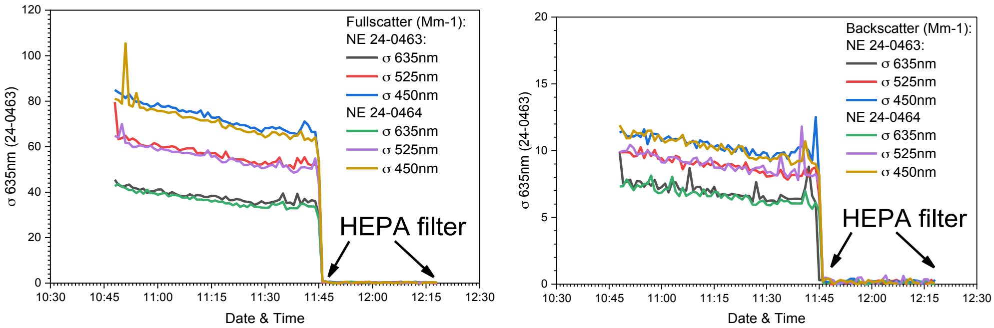


Figure 15: Fullscatter and Backscatter laboratory measurements of NE300-24-0463 and NE300-24-0464 during initial tests.

A sampling line for continuous aerosol measurements that allows the aerosol to be collected while reducing environmental humidity has been realised. The sampling line was installed with a PM10 inlet and includes up to 3 outputs with the measurement of external pressure, and the monitoring of pressure and temperature of the individual lines (Figure 16).

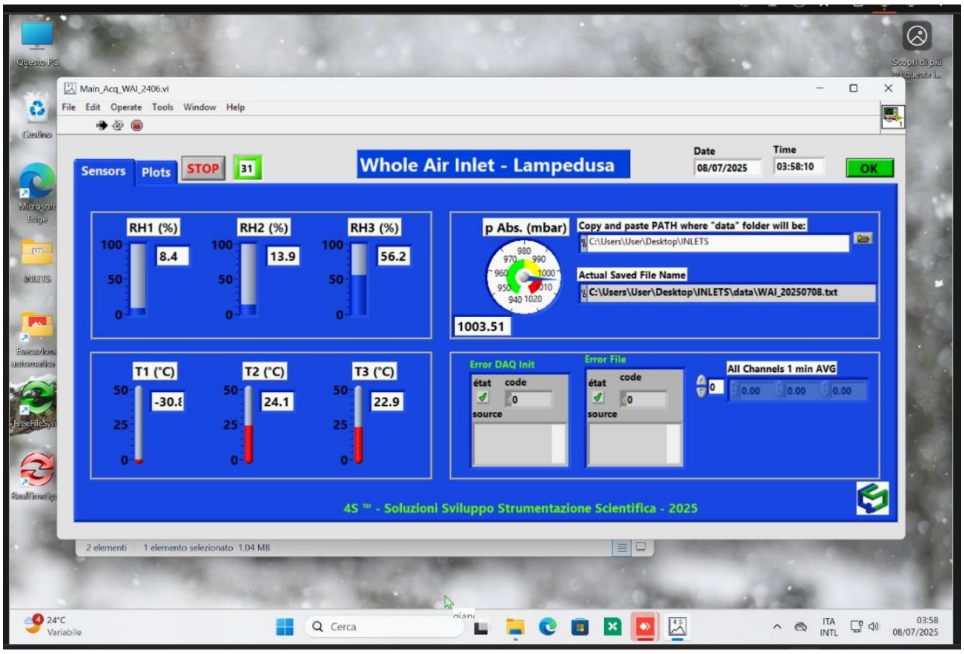


Figure 16: The control screen of the sampling line software with the display of the relative humidity and temperature values of the 3 lines.

Furthermore, a mobile platform for the study of radiative balance has been purchased and tested (Figure 17). The platform is mainly composed of: a Kipp&Zonen solar tracker model Solys2 with tripod for the measurement of global and diffuse irradiance of the shortwave and longwave components (see photo the right); a Kipp&Zonen pyrheliometer model CHP1 for the measurement of direct shortwave radiation; three Kipp&Zonen Pyranometers, a model CMP22 and two CMP21, for the measurement of shortwave radiation (global and diffuse), upward and downward; two Kipp&Zonen pyrgeometers model CGR4 for the measurement of up-down longwave radiation; Kipp&Zonen radiometer model SUV-E for the measurement of ultraviolet radiation in the erythemal action spectrum; two Licor brand quantum sensors model Li190R for the measurement of the downward and upward component of the Photosynthetically Active Radiation (PAR).

The radiometers can be brought into the field and installed on a Campbell Scientific tripod model CM110 and the data produced acquired by two CR1000X dataloggers.

Figure 17: the mobile platform for the study of radiative balance

STATUS AND OPERATIVITY OF THE INSTRUMENTS INSTALLED IN THE ECO OBSERVATORY

At ECO (shelter 1) the inlet line for gas measurements has been upgraded (Figure 18) with a new system that is electronically controlled, and automatically collects and stores the data from the line in realtime. This is the inlet at which the new instrument Picarro G4201 purchased during ITINERIS implementation is connected. The Picarro performs continuous measurements of CO, H_2O, CH_4, and CO_2 and it is fully operative and integrated with the data collection of the observatory (Figure 10).

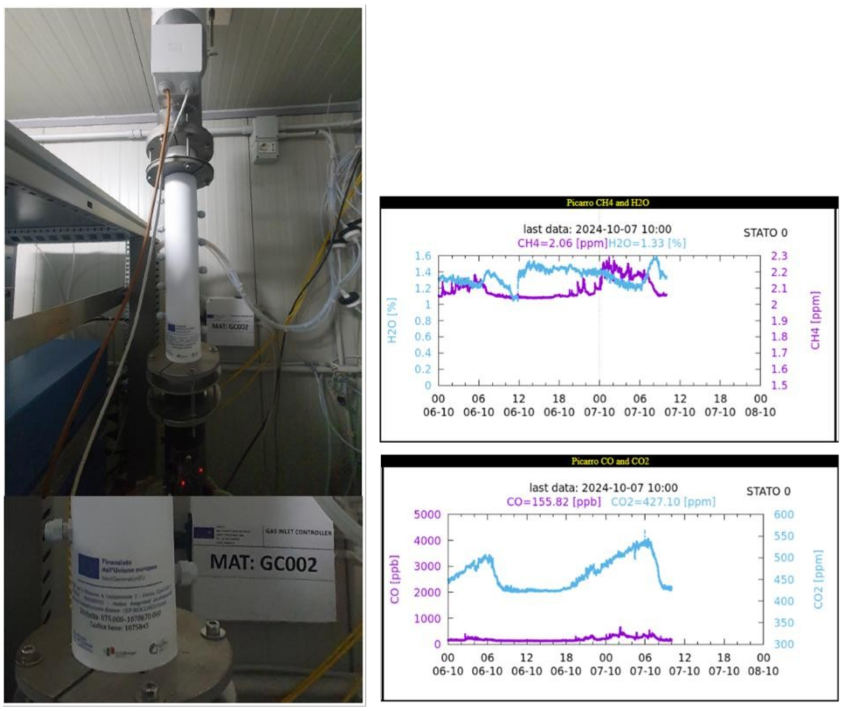


Figure 18: Inlet line for gas sampling and an example of measurements of Picarro G4201.

In addition, the air conditioning of shelter 1 has been upgraded and a new compressor with filter and dehumidification has been installed to use as zero air for some of the aerosol instruments.

Implementation of the activities of ITINERIS project required the installation of a second shelter (shelter 2) at ECO observatory dedicated to the ACTRIS component aerosol in situ. The new shelter was realized and installed dedicated to the main aerosol in situ component. This is shown in Figure 19. The shelter is realized with two independent rooms: one dedicated to the compressor and the pumps; and the other dedicated to instruments. Both rooms are conditioned and kept at a comparable temperature. The power supply includes lines with UPS (20 KVA) and standard lines connected to the main electrical supply of ISAC-CNR.

Two aerosol inlets have been installed and made operative. One inlet has a PM10 cut-off and operates at 2.3 m^3/h. It has an isokinetic splitter (stainless steel) to serve the MAAP, the AE33, the Aurora3000, and the APS. Each instrument has its own nafion dryer, system for measurement of temperature and relative humidity, and a differential pressure measurement system is used to automatically detect eventual non-operating instruments and automatically adjust the excess flow. The second inlet has a PM2.5 cut-off and operates at 1 m^3/h. It also has an isokinetic splitter (stainless steel) to serve the SMPS+CPC and the TOF-ACSM. The SMPS+CPC and the TOF-ACSM have their own nafion dryer on the inlet and measurement of temperature and relative humidity. In addition, a total pressure (for SMPS+CPC) and a differential pressure (for TOF-ACSM) measurement is used to automatically detect non-operating instruments and automatically adjust the excess flow. All the nafion dryers are connected to a distribution line for the counter-flows and served by a compressor able to provide filtered and dehumidified (dew point < -20°C) flow up to 160 L/min. This allows to use serve all nafion tubes with counter-flows between 2 and 3 times the inlet flow. The inlets system is connected to a PC that continuously monitors (and records) the data from the different sensors of the lines; the conditions (pressure, temperature, and relative humidity) at the two splitters; temperature, humidity and pressure inside the shelter. Figure 20 reports some photos including an overview of the solution adopted at the Observatory and the details of the PM2.5 and PM10 inlets. An example of data collected in the different lines is reported in Figure 21.

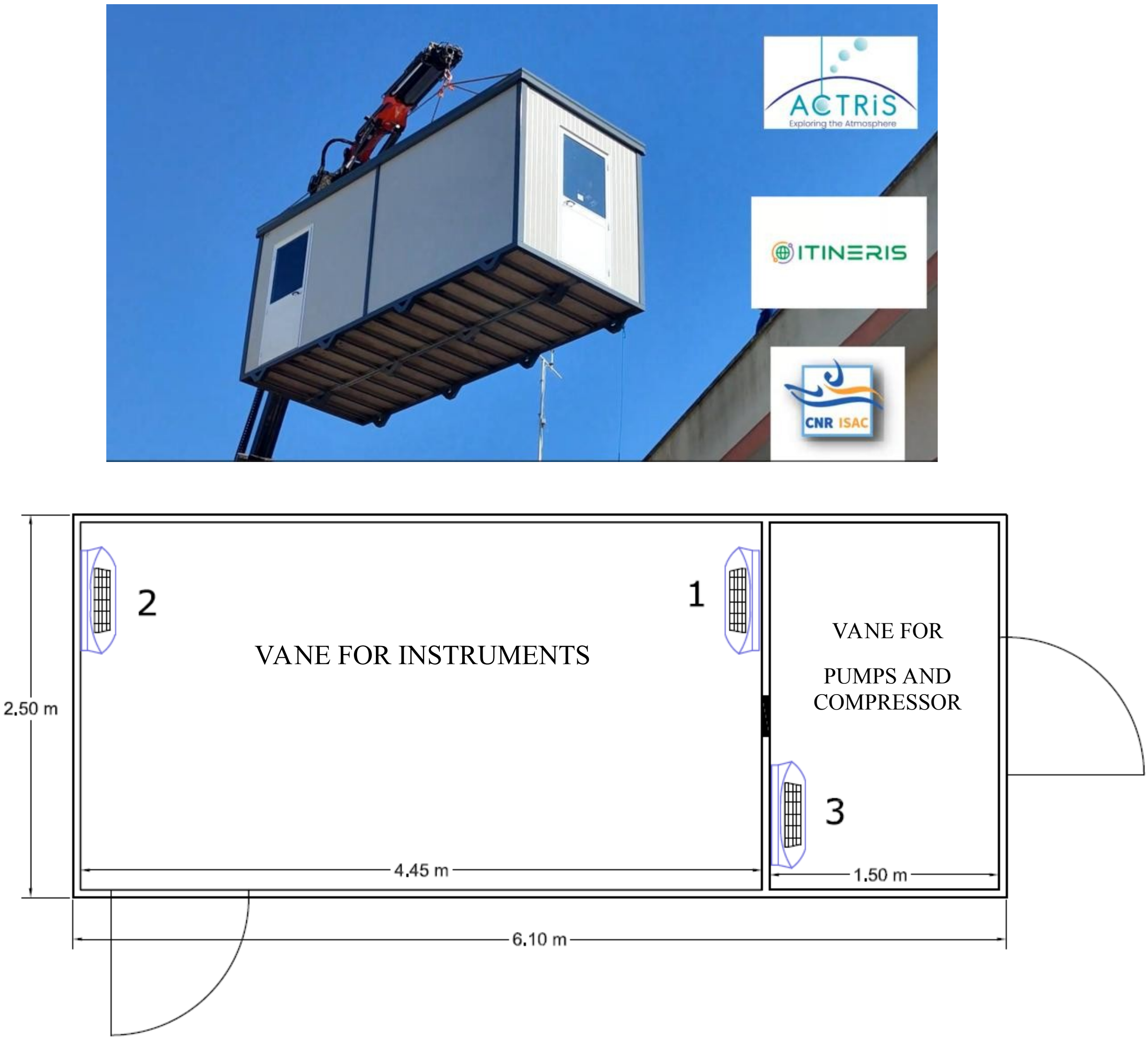


Figure 19: Pictures of the installation of the shelters and its internal organization with the position of the air conditioners.

Figure 20: Pictures of the interior of the new shelter and details of the PM10 and PM2.5 inlets.

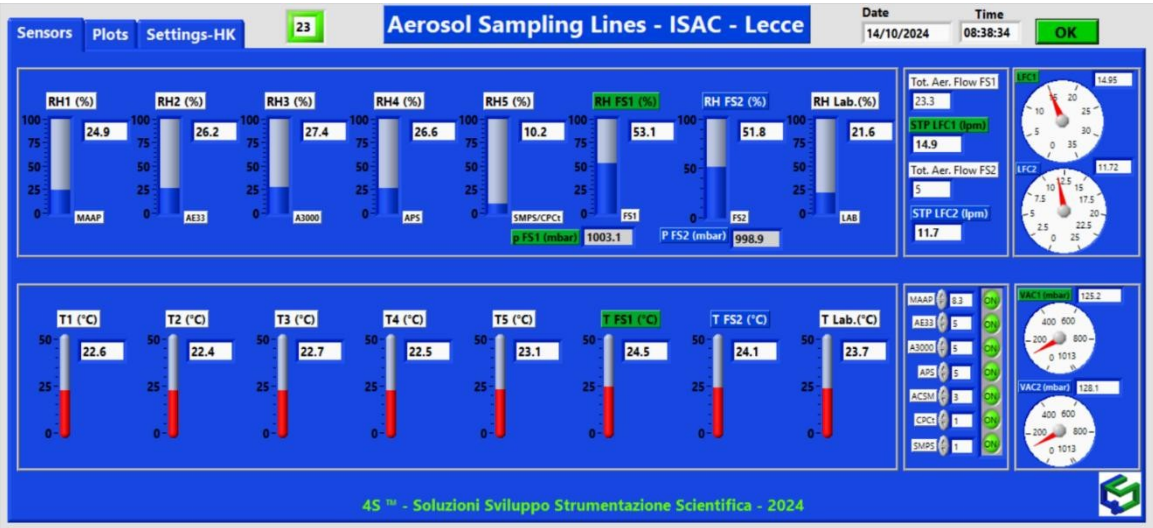


Figure 21: A screenshot of the realtime monitoring and data collection system of the two aerosol inlets.

The CPC (TSI 3750) purchased during ITINERIS implementation has been calibrated at CAIS-ECAC following the ACTRIS procedures and it was installed, as a total counter, at ECO observatory connected to the existing SMPS. It is now fully operative and data are continuously collected online.

The Sun/sky/lunar photometer CIMEL model CE318-T, calibrated following ACTRIS procedure, was installed on 14 November 2023, on the rooftop of CNR-ISAC Lecce building at 12 meters altitude from ground. Prior installation, the instrument has undergone ACTRIS calibration. Since the installation, it operates continuously, day and night, providing in clear sky conditions spectral column integrated aerosol properties and water vapor. The instrument provides direct measurements at 8 wavelengths (340, 380, 440, 500, 675, 870, 1020, 1640 nm) of AOD (aerosol optical depth), which is directly linked to the amount of aerosols in the atmosphere, Angstrom Exponent between 440 and 870 nm giving information about the dominant size of aerosols, and AOD fine and coarse. By an inversion algorithm, it also provides at 4 wavelengths (440, 675, 870, 1020 nm) column integrated, Single Scattering Albedo, Complex Refractive Index (real and imaginary part), Absorption Optical Depth, Extinction Optical Depth, Asymmetry Factor, Phase Functions (from 0 to 180 degrees) and Volume Size Distribution between 0.05 and 15 µm radius. The Sun/sky/lunar photometer CIMEL CE318-T is now installed in close proximity (1 km) to Lecce_University AERONET site for intercomparison purpose (Figure 22), where a long-term (~20 years) sun/sky/lunar photometer is installed. The CIMEL CE318-T is intended to be also used in mobile measurements campaigns in fixed locations, where no other sun/sky/lunar photometer exists in close proximity, and can be used for atmospheric aerosol characterization and for satellites validation. Since it's a solar powered instrument it can be installed in remote locations. The instrument is complementary to the CIMEL microLIDAR CE376 for aerosol characterization in each atmospheric layer and stratification analysis that has been installed during ITINERIS implementation (Figure 23).

Figure 22: View of the Sun/sky/lunar photometer CIMEL CE318-T located on rooftop of CNR-ISAC Lecce building

Figure 23: View of the LIDAR system installed on rooftop of CNR-ISAC Lecce building.

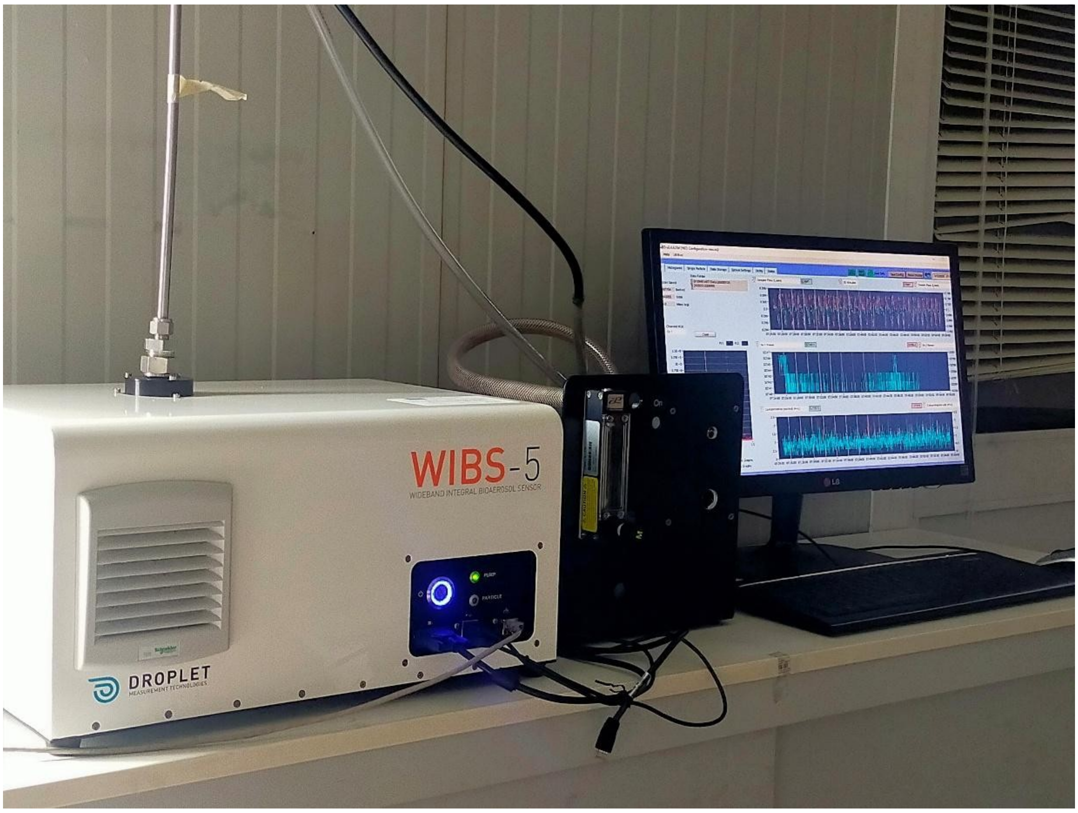


Figure 24: Wideband Integrated Bioaerosol Sensor WIBS-5/NEO (Droplet Measurement Technologies).

The Wideband Integrated Bioaerosol Sensor WIBS-5/NEO (Fig. 24) is an integrated, online, broadband detector that relies on fluorescence measurement to determine the precence of biological material in atmospheric particles. It provides highly sensitive and real-time data on size, shape and fluorescence properties of particles to enable classification of atmospheric bacteria, pollen, bacteria and fungi. The WIBS-5/NEO has been installed and it is currently operational at the ECO Observatory at CNR-ISAC.

The Xact® 625i system (Fig. 25) was purchased and installed at ECO Observatory. It is designed for high time resolution multi-metals monitoring of ambient air, enabling near-real-time analysis of metals in particulate matter with a 3-hour time resolution. The system uses reel-to-reel filter tape sampling and non-destructive energy dispersive X-ray fluorescence (ED-XRF) analysis. The air is sampled through a low volume particulate matter (PM) size-selective inlet and drawn through a filter tape. The resulting PM deposit is then advanced into the analysis area where the sample is analysed by ED-XRF for selected metals while the next sample is collected. The system is currently operative at the ECO facility.

The HALO wind lidar was purchased and tested in 2025 (Figure 26). The instrument measures vertical wind profiles by exploiting the Doppler effect of the aerosols carried by the wind itself. To obtain a good resolution of the horizontal and vertical component of the wind, the wind lidar must therefore measure at different zenith angles. To this end, the rotating ‘turret’, which allows measurements at different azimuthal ranges, and the rotating mirror, highlighted in green and red respectively in the figure, are highlighted in the figure. The management software (Figure 27) allows the definition of different types of measurement as well as displaying both the echo of the lidar signal, sensitive to the presence of aerosols, and the vertical turbulent motions.

Figure 25: Xact® 625i Ambient Metals Monitor (Sailbri Cooper).

Figure 26: LIDAR system.

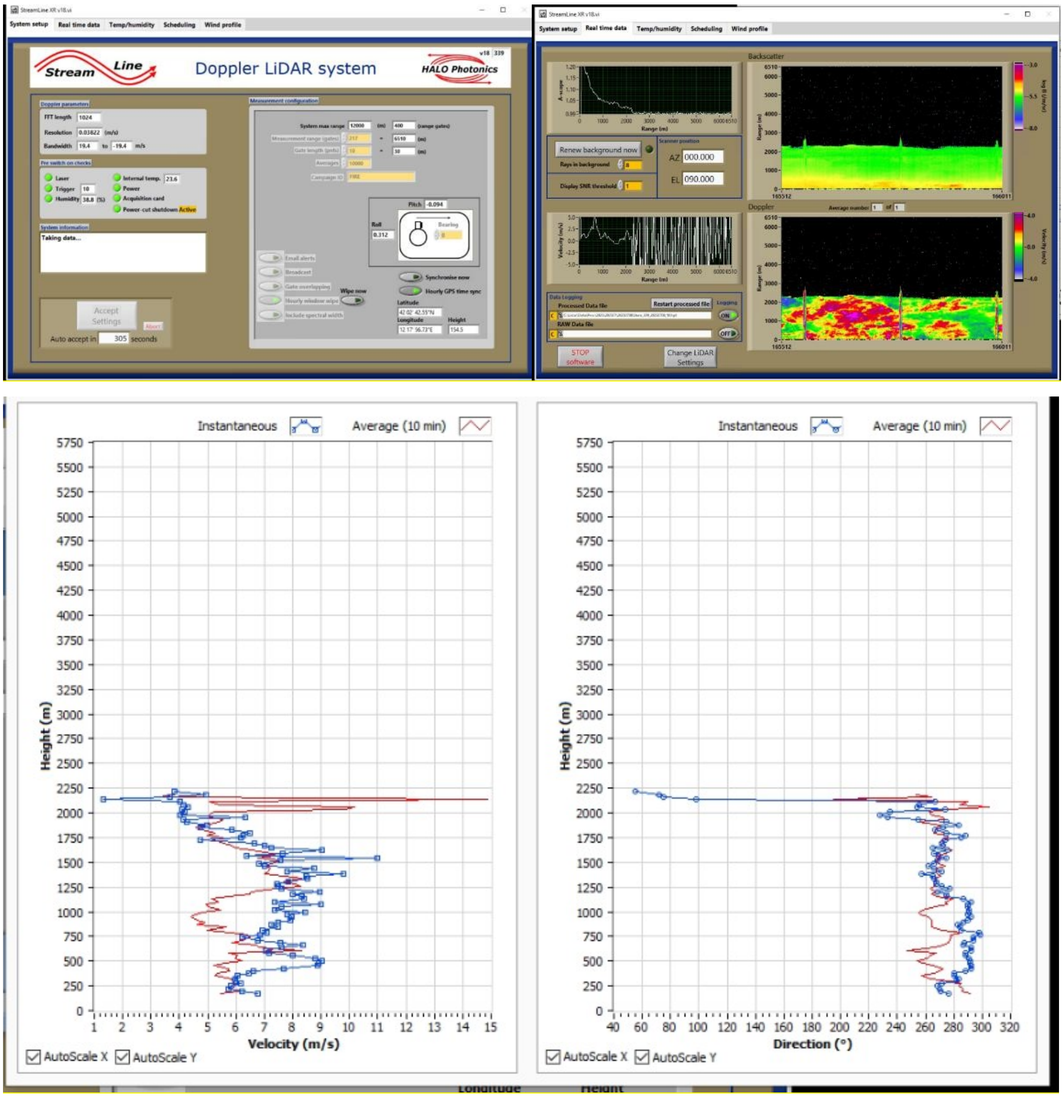


Figure 27: LIDAR software

The HATPRO radiometer will be delivered and installed in September 2025 due to the long time necessary for the purchase.

In addition, it has been purchased a Semi-Continuous VOC Analysis System for the ECO Observatory in Compliance with ACTRIS Guidelines for Reactive Trace Gases component. This instrument has been shipped and it is foreseen the installation and full operativity by the end of July 2025. As part of the environmental monitoring activities foreseen by the PNRR project IR000032 - ITINERIS – Italian Integrated Environmental Research Infrastructures System, a system for the semi-continuous sampling and analysis of volatile organic compounds (VOCs) was foreseen. The system is based on thermal desorption, gas chromatography, and mass spectrometry (TD-GC-MS) and is specifically designed to characterize atmospheric VOCs, with particular focus on ozone precursors, in accordance with the requirements and guidelines established by the European research infrastructure ACTRIS. To meet these standards, the following instrumentation was acquired:

- Gas Chromatograph: Agilent Technologies model 8890
- Mass Spectrometer Interface: Heated interface up to 350°C for connection to the MS

- Mass Spectrometer: Agilent Technologies 5977C with a monolithic hyperbolic quadrupole mass filter
- Vacuum System: 255 L/sec turbomolecular pump
- Thermal Desorber and Sampling System: UNITY–Air Server-xr, 3-channel Air Server with mass flow controllers (2–250 mL/min Air/N_2, 2–500 mL/min He) and 1 thermal desorption unit

To ensure full operability and compliance with international technical standards, the following additional components will be installed:

- GS-GASPRO chromatographic column (60 m length, 0.32 mm internal diameter), specifically designed for the separation of light VOCs, enabling the resolution necessary for the correct identification and quantification of ozone precursors as required by ACTRIS guidelines.
- Nafion membrane dryer, as recommended by the same guidelines, to remove humidity from samples before they enter the TD-GC-MS system. This step is critical to avoid analytical interferences, improve signal stability, and protect the integrity of the mass spectrometer.
- The acquisition and integration of these components are essential to ensure the quality, reliability, and comparability of the measurements performed within the ACTRIS network. The implementation of this system represents a key step in equipping the ECO Observatory with advanced, ACTRIS-compliant infrastructure for VOC monitoring.

CONCLUSIONS

The instruments that have been installed and made operative will be the base for collecting datasets necessary to the analysis of Aerosol Sources (action 4.12). Some of the datasets are already available and preparation of publications is ongoing. Additional datasets will be available when the instruments not yet installed will be made operative. Different datasets will be made available on the ITINERIS Hub by the end of the project.

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