EMPIR 16ENV17 AEROMET Project
TXRF analysis of aerosol samples

Bruker Nano Analytics, Berlin, Germany
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TXRF analysis of aerosol samples
EU EMPIR 16ENV07 AEROMET

Aerosol metrology for atmospheric science and air quality

www.aerometproject.com/

- Measurements of aerosol particles enforcing EU air quality regulations
  - to protect human health
  - research on climate change effects
- Metrics (mass concentration) as PM_{10}* and $PM_{2.5}$ are in use
  - level of uncertainty is too high
  - traceability is insufficient
- The project aims at implementing improvements in
  - A) the uncertainty of particle mass, size, number of measurements
  - B) in the characterization of regulated components in PM

*) PM_{10} / $PM_{2.5}$ = particulate matter < 10 µm / 2.5 µm

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Objectives of work packages

Objectives

1. WP 1
   - Development of reproducible reference methods for PM_{10} and $PM_{2.5}$
   - Design of aerosol chamber system for calibrating PM_{10} and $PM_{2.5}$ instruments
   - Preparation of representative aerosols
2. WP 2 (Bruker involved)
   - Traceable validated methods for the determination of major components of PM
   - Elemental and organic carbon, total carbon, anions and cations
   - Toxic metals (lead, arsenic, cadmium, mercury, nickel)
3. WP 3
   - Development of calibration procedures for Mobility Particle Size Spectrometers
   - Calibration facilities for measuring particle number concentration
4. WP 4 (Bruker involved)
   - Quantify PM with mobile x-ray spectroscopy combined with particle sampling
   - Prove by lab-based methods
5. WP 5 (Bruker involved)
   - Standardization, take up by accredited laboratories,

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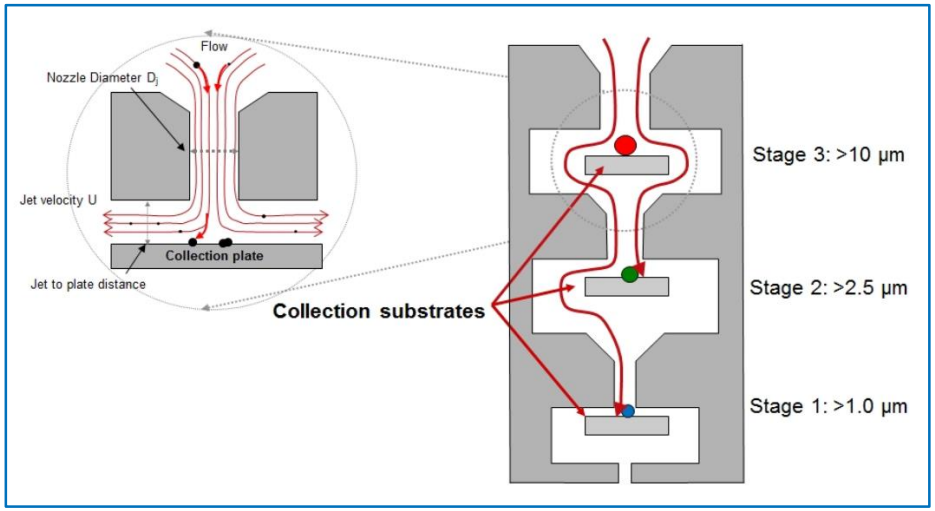
Activities WP 4

Aerosol sampling

- Aerosols are fractionated by size in an impactor
  - Typically aerosols are sampled on filter substrates
  - Long sampling time (~24 h) and digestion required for ICP analysis



Impactor working principle (www.dekati.com)

- Preliminary tests for TXRF
  - Direct analysis after sampling on acrylic discs is possible
  - Sampling times of down to 1 h deliver sufficient material for TXRF analysis

Special adapter ring for TXRF discs

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Activities WP 4

Two onsite campaigns

- Budapest, Hungary, May 2018,
  Cassino, Italy, September 2018
- Sampling with 13-stage Dekati impactor,
  sampling time 1 to 60 hours
- PM10 samplers for lab-based ICP-MS analysis,
  - sampling time >=14 h !
  - no size fractions !
- Table shows all analytical tasks and instruments used (see last slide for more details)

Purpose	Details	Instruments
Sampling	Size range: 30 nm – 10 µm	Cascade samplers (Dekati, May-type), PM10 samplers, ELPI
Quantitative element analysis	Mg to U down to 1 ppb	S2 PICOFOX TXRF spectrometer
Total particle mass concentration	$PM_{2.5}$, PM_{10} (black carbon)	TEOM, Aethalometer, DustTrak
Aerosol spectroscopy	Size range: UFP, FP, number conc.	OPSS, APS+MPSS, ELPI, CPC stand-alone, low-cost optical sensors

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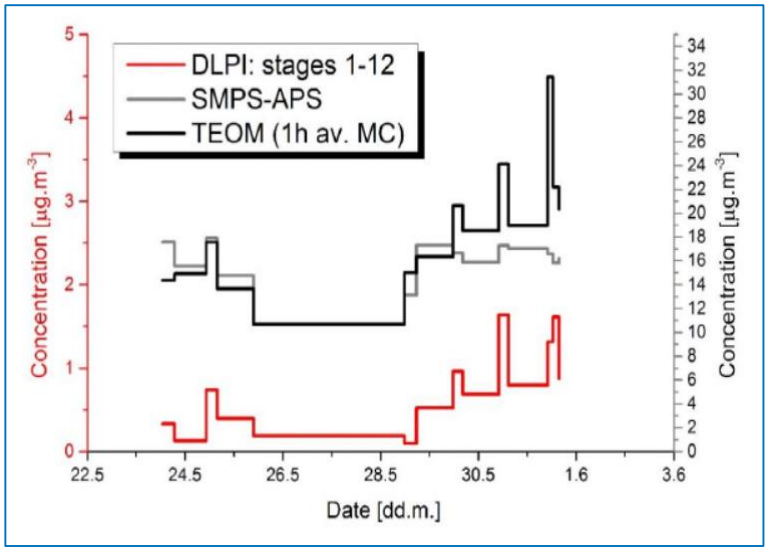
Results

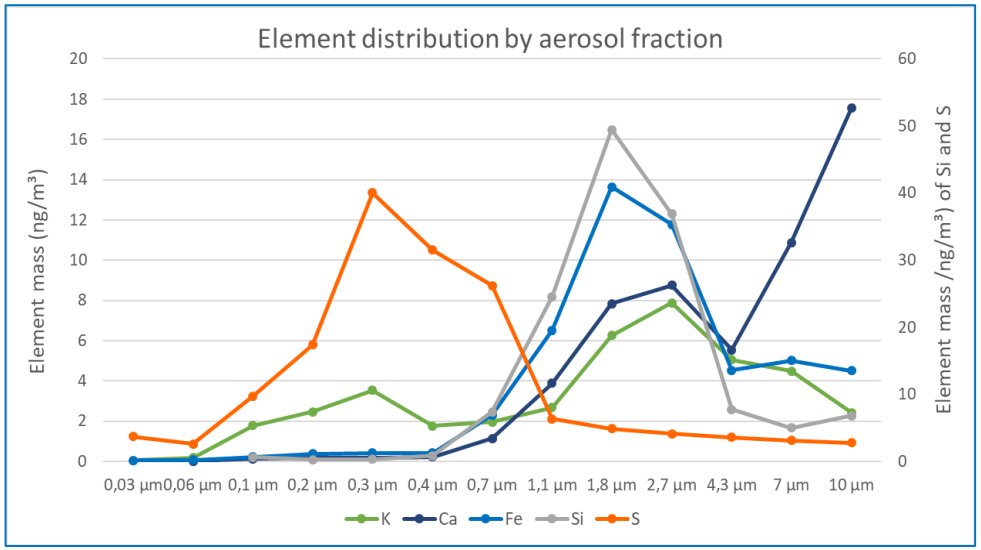
Trend of total mass

- Total particle mass was continuously measured with TEOM during 10 days (black curve below)
- Total inorganic mass detected with TXRF (red curve) shows exactly the same trend

Distribution of main elements

- Fractions from 1 to 4 µm typically contain geogene elements like Ca, Fe, Si
- Sulphur appears in particulate matter between 0,2 and 0,7 µm





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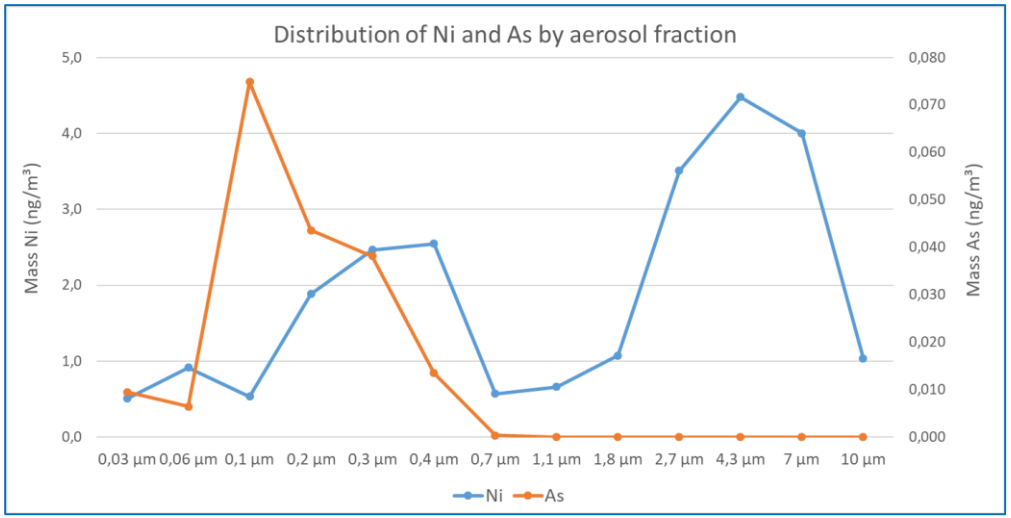
Results

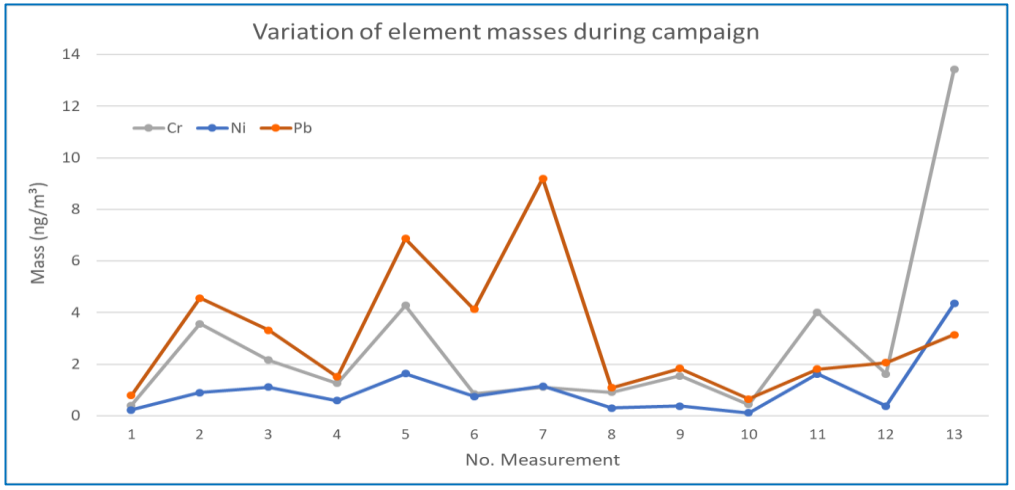
Toxic elements by particle fraction

- Ni (also Cr, Pb) detectable after short sampling times (mass up to 5 ng/m³ per fraction)
- As detectable after 14 h sampling time (< 70 pg/m³) present only in particles from 100 to 400 nm!

Trend analysis of toxic elements

- Cr, Ni and Pb mass variations could be monitored even at shortest sampling times (sum of all fractions shown)





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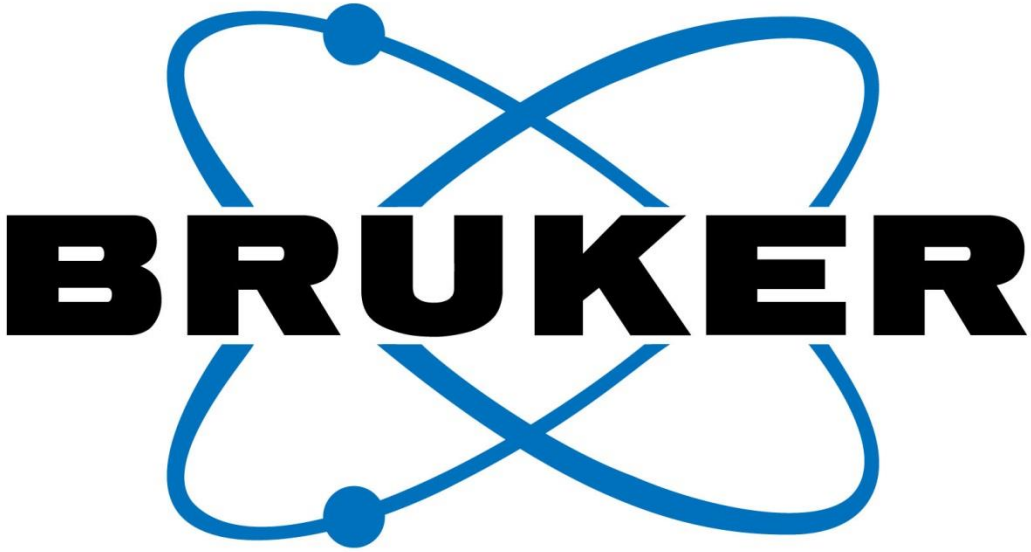
Outlook

Present work and outlook

- In-field results will be compared with lab-based results (synchrotron-TXRF and ICP-MS)
  lab system prove presence of more elements (e.g. Cd)
- Lab results and new aerosol standards will be applied to optimize results of direct TXRF measurements
- A draft standard operation procedure (SOP) for in-field TXRF measurements is available
- For standardization an ISO TR (technical report) will be proposed in 2019

Project funding

The EMPIR initiative is co-funded by the European Union's Horizon 2020 research and innovation programme and the EMPIR Participating States



Innovation with Integrity

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Background information

Instrumentation for aerosol metrology

- MPSS: Scanning Mobility Particle Size Spectrometer applied for particles below 1000 nm
- APS: Aerodynamic Particle Sizer
- TEOM: Tapered Element Oscillating Microbalance for real-time measuring of aerosol mass
- Aethalometer: concentration of optically absorbing (black) particulates