


QA/QC of Speciation of Particulate Matter

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QA/QC of Speciation of Particulate Matter

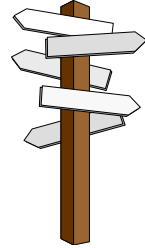
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Roadmap of presentation



- ▶ **Introduction - context**
- ▶ **Measurement traceability**
 - ▶ General
 - ▶ Realization for constituents of PM
- ▶ **Consequences for speciation**
 - ▶ What should be done ?
 - ▶ What can be done ?
- ▶ **Conclusions**

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Introduction

- ▶ **Relevance of speciation of PM**
 - ▶ Legal requirements (metals, PAH; ions, EC/OC)
 - ▶ Source apportionment
 - ▶ Natural sources
 - ▶ (Effectiveness of) source reduction measures
 - ▶ Exposure to constituents of PM
 - ▶ Modeling of transport, deposition, mass balances

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Introduction

- ▶ **Only accurate speciation data lead to meaningful interpretations:**
 - ▶ Measurement uncertainty shall be known and within specified limits
 - Legal requirements (metals, PAH)
- ▶ **Requires establishment of measurement traceability**
 - Method validation
- ▶ **Requires maintenance of measurement traceability**
 - Quality assurance / quality control

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What is “measurement traceability” ?

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Traceability - VIM

‘Relationship of a measurement value or the value of a standard whereby it can be related to stated references, usually (inter)national standards, through an unbroken chain of comparisons, all having stated uncertainties’

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Traceability – ISO 14111

'The ability to provide evidence of the uncertainty attributed to measurement results through documented calibrations, using measurement standards of known uncertainty and comparison measurements of known performance'

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Traceability

← SI Units

← (Inter)national (Primary) Standards

← Secondary Standards

← Work Standards/In-house Standards

← Measurement/Calibration

← uncertainty →

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Traceability

- ▶ **Establishment is prospective** → Validation
 - Uncertainty assessment of measurement results
 - "Source apportionment" of uncertainty budget
 - Critical points for quality control
- ▶ **Maintenance is required** → Quality Control
- ▶ **Essential element of EN-ISO 17025**
- ▶ **Can NOT be claimed on basis of participation in inter-laboratory comparisons !**

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Measurement traceability in practice

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Gases

→ Direct measurement using analysers

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Gases

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Constituents of PM

- ▶ **Sampling / filtration**
 - ▶ Sample volume
 - ▶ PM fractionation
 - ▶ Filtration efficiency
 - ▶ (Extraction efficiency)
 - ▶ Analytical accuracy

→ Requires separate "chains" for each step!

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Constituents of PM

→ Indirect analysis

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Constituents of PM

→ Direct analysis

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Constituents of PM

→ Extraction

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Gap analysis

- ▶ **Lack of standardized methods**
 - ▶ Extraction
 - ▶ Direct and indirect analysis
- ▶ **Lack of representative "matrix-matched" CRM**
 - ▶ Particle composition: physical and chemical
 - ▶ Filter materials used in practice
 - ▶ Optimum filter materials for methods may differ from those used in practice for "economic" reasons!

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QA/QC in practice

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General

- ▶ **Set performance requirements for each element of measurement**
 - ▶ Bases on validation results
 - ▶ Based on existing information
- ▶ **Use standardized methods**
 - ▶ E.g. from occupational hygiene sources
- ▶ **Use existing CRM for control purposes (only)**
- ▶ **Participate in inter-laboratory comparisons**

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Indirect analysis

- ▶ **Instrument calibration**
 - ▶ Certified liquid standards
 - ▶ Reagent matrix
 - ▶ Response drift
- ▶ **Analysis**
 - ▶ Reagent blank levels
 - ▶ Filter blank levels
 - ▶ Replicate analysis
 - ▶ Standard additions

} Procedure blank

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Direct analysis

- ▶ **Instrument calibration**
 - ▶ Certified solid standards
 - ▶ E.g. thin-film + powder standards for XRF
- ▶ **Analysis**
 - ▶ Filter blank levels
 - ▶ Replicate analyses !
 - ▶ Standard additions
 - ▶ CRM for control purposes
 - ▶ Indirect method for comparison/confirmation

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Extraction

- ▶ **Recovery**
 - ▶ Reagents of known purity
 - ▶ Controlled procedures
 - ▶ "Energy" supplied
 - ▶ Perform replicate extractions on single sample
 - ▶ With same reagent
 - ▶ With different reagent
 - ▶ CRM for control purposes

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What do we need ?

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Gap closure

- ▶ **Standardized methods**
 - ▶ Performance requirements, e.g., uncertainties
 - ▶ Established procedures
 - ▶ Not just for EC/OC and ions !
- ▶ **Representative CRM**
 - ▶ Particle size (distribution)
 - ▶ Filter material(s)
 - ▶ Mass fractions of species
- ▶ **Inter-laboratory comparisons**
 - ▶ Preferably as standing schemes

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Gap closure

- ▶ **Standardization**
 - ▶ CEN TC264: EC/OC and ions in PM_{2.5}
 - ▶ No uncertainty requirements !
- ▶ **Representative CRM**
 - ▶ IRMM: PM₁₀ powder materials
 - ▶ Regulated species (heavy metals; PAH)
 - ▶ Other elements ?
 - ▶ IRMM EU Report 23244
 - ▶ Final results pending
- ▶ **Inter-laboratory comparisons**
 - ▶ Initiatives by JRC-IES for EC/OC and PAH

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Conclusions

- ▶ **Number of missing links for QA/QC**
 - ▶ Unfinished traceability chains
 - ▶ Lack of standardized methods
 - ▶ No information about comparability of
 - ▶ Existing methods
 - ▶ Laboratories
- ▶ **Requirement for**
 1. Standardized methods
 2. Inter-laboratory comparisons
 3. Representative CRM

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Thank you !

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