Validation and QA/QC of air monitoring methods
The Diffusive Sampling case

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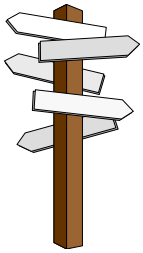
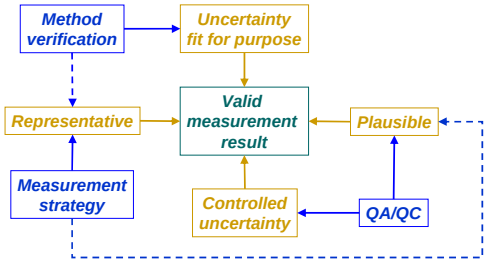
Overview



- Introduction
- Method *verification*
  - Current concepts
  - Discussion items
- Quality assurance/quality control
  - Current concepts
  - Discussion items
- Conclusions

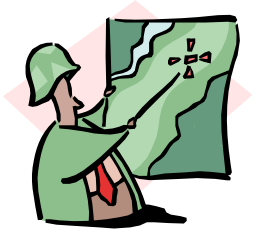
Introduction



Terminology & definitions

- *Validation => verification*
  - Provision of objective evidence that a given item fulfils specified requirements
    - *EXAMPLE 2 Confirmation that performance properties or legal requirements of a measuring system are achieved*
    - *EXAMPLE 3 Confirmation that a target measurement uncertainty can be met*

Source: VIM 2008



Measurement strategy

Measurement strategy

- *Dictated by purpose of measurement*
- *Diffusive sampling & OLCM*
  - Measurement of gases and vapours
  - Long(er)-term averages
  - Spatial variations in concentrations
  - Personal exposure measurements
  - Measurements at remote locations

➔ Screening, mapping
➔ Validation of models
➔ Compliance monitoring

Verification

Verification

- *"Device" verification:*
  - Formulation of criteria for fitness-for-purpose
  - Formulation of test programmes
  - Testing of representative "samples" of devices
  - Evaluation of test results against criteria

Verification

- *Subtle difference with other methods for gases and vapours*
  - No direct traceability of measurement results unless device performs "ideally"
  - ➔Zero effects on performance of (variations in) parameters
    - Concentration of measurand
    - Presence of interferents, humidity, wind speed
    - Radiation, precipitation, deposition
  - ➔Quantified effects on performance of (variations in) air temperature and pressure

Diffusive sampler

- *Sampling rate is*

$$\upsilon = \frac{A}{L} D$$

- *Relative uncertainty is*

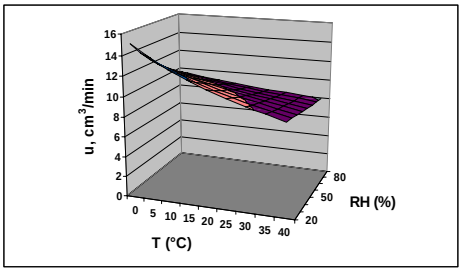
$$w^2(\upsilon) = w^2(A) + w^2(L) + w^2(D)$$

➔*Even "ideal" sampler requires verification*

Verification protocols

- *E.g., EN 838, EN 13528-2*
  - Extensive tests ("full-factorial designs")
    - *Highly time consuming*
  - Require sophisticated facilities
  - Require test conditions that have insignificant effects on uncertainty of υ
    - *Extensive QA/QC*
  - Verification only "valid" for range of experimental conditions
  - Confirmation by field tests

Lab test examples



Lab test examples

$$\upsilon = \tfrac{1}{2}(134 + 0{,}86RH - 0{,}00130\,t + 1{,}28T + 3{,}18w)10^{-5} \cdot \left(1 + \sqrt{1 - \frac{0{,}64.10^{-5} m}{t\,((134 + 0{,}86RH - 0{,}00130\,t + 1{,}28T + 3{,}18w)10^{-5})^2}}\right)$$

υ =	sampling rate in ng.ppb^{-1}.min^{-1}
T =	temperature in °C
RH =	relative humidity in %
w =	wind speed in m.s^{-1}
m =	analytical mass of measurand in ng
t =	exposure time in min

Buzica et al., JEM 7 (2005) 169-174

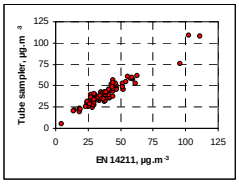
Verification

- *Guide to Demonstration of Equivalence*
  - **Minimum laboratory protocol requires 2 tests under "extreme" conditions**
    - **➔Sufficient if difference in υ small enough for device to be fit for purpose**
    - **➔Else: more tests**
  - **Field tests by parallel measurements with reference method**
    - **Serve to confirm uncertainty claim**

Verification

- *Field tests*
  - **Minimum number of representative sites**
  - **Replicate samplers in parallel with reference method**
  - **Minimum number of sampling periods per site**
  - **Different meteorological conditions**
  - **➔Requires appropriate QA/QC of operation of reference method**

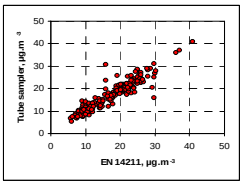
Field test examples



Annual mean values

Tube = 1,02 (0,03) Ref + 0,6 (1,2)
U_r = 9,8 µg.m^{-3}

Data from AEA Technology



Four-week values

Tube = 0,95 (0,05) Ref + 0,5 (1,0)
U_r = 3,2 µg.m^{-3}

Data from Lig'Air

Verification

- *Current situation:*
  - **Current protocols may be unnecessarily elaborate**
  - **"Anyone's business"**
  - **Only voluntary "link" to quality requirements, e.g. ISO 9001 certification or EN-ISO 17025 for device testing**

Verification

- ***➔Conceptually similar to automated measuring system (AMS) certification cf. EN 15267***
- ***Subtle differences with EN 15267 concept***
  - **No formal requirement**
  - **No competent authority ***
  - **No assessment of manufacturer's quality system**
- ***Would provide "high" profile for devices***

*** Unless equivalence is claimed**

EN 15267 certification

- Controlled production process
- Formulation of test programmes
- Testing of representative "samples" of devices by qualified laboratories
- Evaluation of test results against performance criteria
- Post-validation product surveillance
- Re-testing in case of significant design changes
  - → Re-testing in case of changes in batches of sorbents

Verification

- Revise current protocols to include more "relaxed" test regimes where appropriate ?
- Should / can we link device verification – at least for ambient and indoor air monitoring - to EN 15267 certification?
- Should a similar system be introduced for workplace air monitoring ?
- If yes, how far should we go in e.g. prescribing device re-testing ?

Quality assurance/ quality control

Quality assurance/quality control

- Production and testing of devices
  - Voluntary in current situation
  - Covered by product certification requirements
- Practical application
- Plausibility checks of results

QA/QC - application

- Elements described in generic standards and manufacturer's manuals
  - Use of appropriate shelters
  - → Devices exposed in replicate
  - → Parallel measurements with independent methods
  - Field / transport blanks
  - → Measurement of air temperature, pressure
  - → Measurement of humidity, interferents, wind speed
  - Analytical QA/QC

QA/QC - application

- Do we need to expose replicates if devices are properly produced and validated ?
  - If so, how many ?
  - What is the basis for acceptance criteria of replicate results ?
- Comparison with independent method requires
  - Requires acceptance criteria based on knowledge of uncertainties of (aggregated) values for both methods
- Who measures temperature, pressure, etc. ?

Replicate measurements

- *How many replicates ?*
  - Generally 2
  - What should be the maximum permissible difference ?
    - Based on between-sampler replicability
    - Guide to Demonstration of Equivalence: 5%
- *What if Δ > 5% ?*
  - Reject both values ?
  - Use one ? If so, which one ?

QA/QC - plausibility

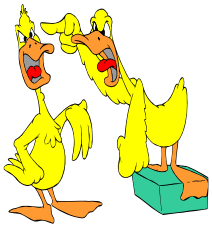
- *Comparison of results with expected values/ ranges*
  - Measurement strategy
  - Existing information - modelling
  - Correlations
  - Criteria ?

➔ *Need for development of protocols for plausibility checks*

Conclusions

Conclusions

- *Revision of ENs required to simplify verification test protocols where appropriate*
- *Verification of devices may be linked to certification schemes*
  - Provides confidence to users
  - Principles of EN 15267 for ambient and indoor air
- *Some aspects of QA/QC should be re-considered and elaborated*
- *Harmonized protocols for plausibility checks should be developed*



Discussion ?