


Validation and Performance of Measurement Methods

NMi

QA/QC of monitoring of Indoor Air Pollutants

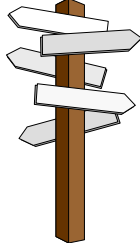
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Roadmap of presentation



- ▶ Introduction
 - ▶ Problem definition
- ▶ QA/QC building blocks
- ▶ State of the art in Indoor Air Monitoring
- ▶ Activities at NMi
- ▶ Summary

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VITO Indoor Air Workshop 2007

- ▶ **Problem:**
 - ▶ “The lack of *comparable* and *representative indoor pollution exposure data* at member state levels as well as at European level is *preventing* quantitative risk assessment and is impairing the establishment of comprehensive and effective health and environment policies regarding air pollution in general, the impact of indoor pollution being very poorly known.”

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“Buzz words”

- ▶ **Representativity**
 - ▶ What, when, where, how long, how often ?
- ▶ **Exposure**
 - ▶ Who, how ?
- ▶ **Comparability**
 - ▶ Do different labs get similar answers when measuring “the same”?
- ▶ **Uncertainty**
 - ▶ Measurement results can only be compared when their uncertainties are known

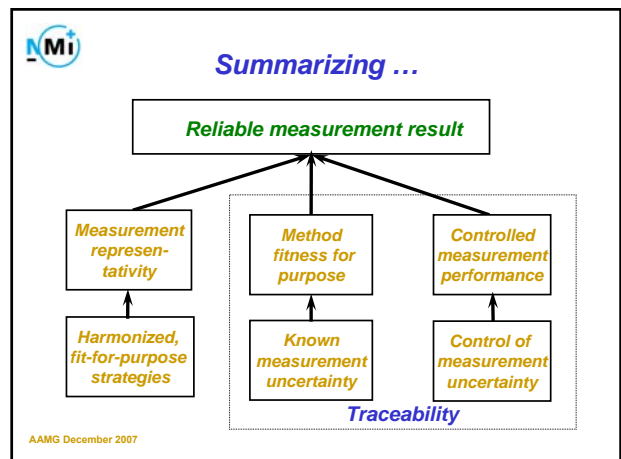
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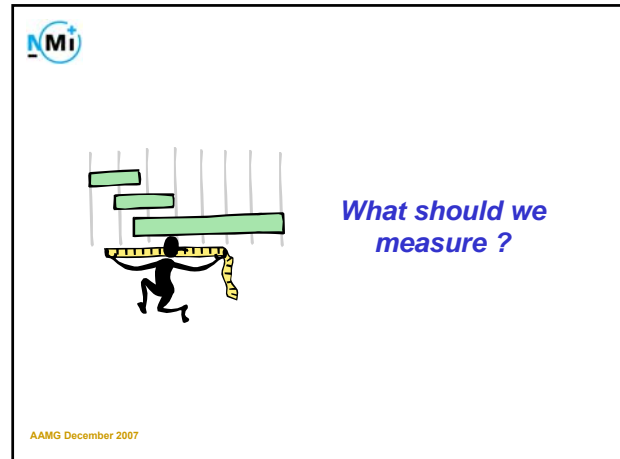
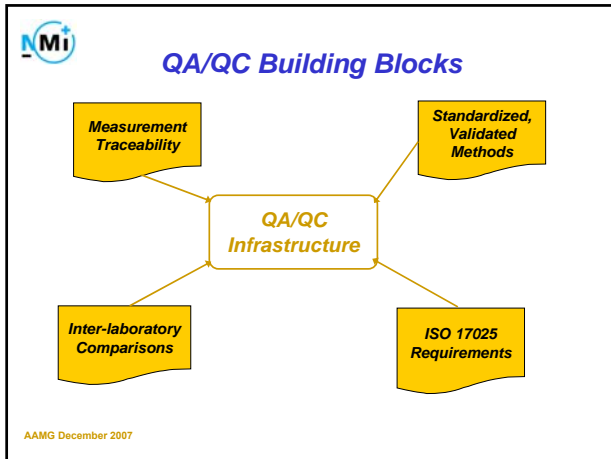
Summarizing

- ▶ **Valid risk assessments and effective health and environmental policies require reliable measurement results, i.e.**
 - ▶ Measurements results that are representative for their purpose(s)
 - ▶ Measurement results *with known* uncertainties
 - ▶ Results that are comparable between bodies measuring for similar purposes

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Priority pollutants (INDEX)

Group 1	Group 2	Group 3
Benzene	Acetaldehyde	α -Pinene
Formaldehyde	Xylenes	Limonene
Nitrogen dioxide	Styrene	Ammonia
Carbon monoxide	Toluene	
Naphthalene		
(ETS)		

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- Priority pollutants (others)**
- ▶ Particles
 - ▶ Polycyclic aromatic hydrocarbons
 - ▶ Organophosphates
 - ▶ Biological compounds
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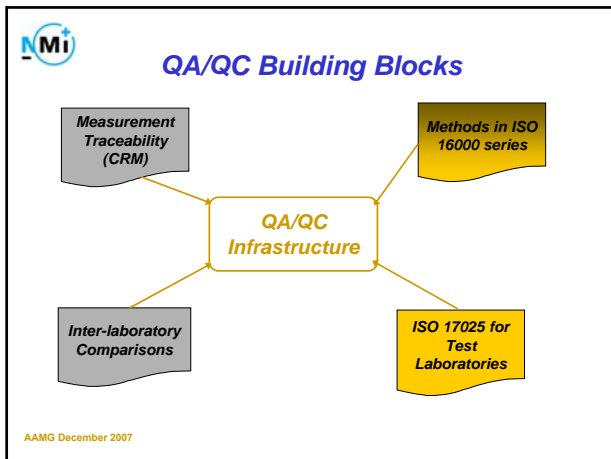
Expolis VOC

n-Hexane	Styrene	Hexanal
n-Nonane	Naphthalene	Benzaldehyde
n-Decane	Propylbenzene	Octanal
n-Undecane	Trimethylbenzenes	Trichloroethene
Cyclohexane	2-Butoxyethanol	Tetrachloroethene
Benzene	2-Methyl-1-propanol	1,1,2-Trichloroethane
Toluene	2-Ethylhexanol	d-Limonene
Ethylbenzene	Phenol	1-Methyl-2-pyrrolidinone
m/p-Xylene	1-Octanol	Δ 3-Carene
o-Xylene	1-Butanol	α -Pinene

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- TVOC**
- Concept of TVOC ('Total Volatile Organic Compounds')**
- ▶ Sampling on Tenax TA
 - ▶ Analysis by TD-GC using an apolar GC column
 - ▶ Consider part of chromatogram between *n*-hexane and *n*-hexadecane
 - ▶ Quantify as many compounds as possible based on individual response factors
 - ▶ reference to list of 63 VOC (EUR Report 17675)
 - ▶ at least 10 most abundant compounds
 - ▶ Quantify other compounds using response factor of toluene
 - ▶ Sum of masses = TVOC
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Standardization

- ▶ **ISO 16000 series**
 - ▶ Measurement strategies (e.g. parts 1, 2, 5, 7, 12, 15)
 - ▶ Measurement methodologies (e.g. parts 3, 4, 6, 13/14)
 - ▶ Materials emission testing
- ▶ **ISO 16017 parts 1 and 2**

→ Do we know measurement uncertainties ?

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ISO 16000-4

11 Quality control

The user shall adhere to the *requirements of ISO/IEC 17025*.

Field blank and replicate samples shall be taken as part of each investigation or batch of samplers. These should equal at least of the total number of samplers used and be a minimum of one for small studies. It is also recommended that

a) for internal quality control, *diffusive sampling rates be checked routinely*; at least once during large surveys. This should be done by exposing samplers in laboratory standard atmospheres or by laboratory or field comparisons with an independent (e.g. pumped) method, and

b) for external QA, *diffusive sampling rates be checked routinely*; at least once during large surveys. This should be done by *laboratory or field inter-comparisons* (one sampler, various laboratories), which should be conducted by experienced institutes.

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Inter-laboratory comparisons

- ▶ **'One-off'**
 - ▶ JRC: TVOC in previous century
 - ▶ JRC: diffusive sampling of ambient NO₂
- ▶ **Proficiency testing**
 - ▶ HSL: ambient NO₂, RICE, UK MMMF, EnAct
 - ▶ BGIA: VOC on Tenax
 - ▶ BGIA, VITO: sampling of VOC
 - ▶ Range of VOC seldom goes down to low vapour pressures
 - ▶ Formaldehyde, carbon monoxide ?

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Vapour pressures

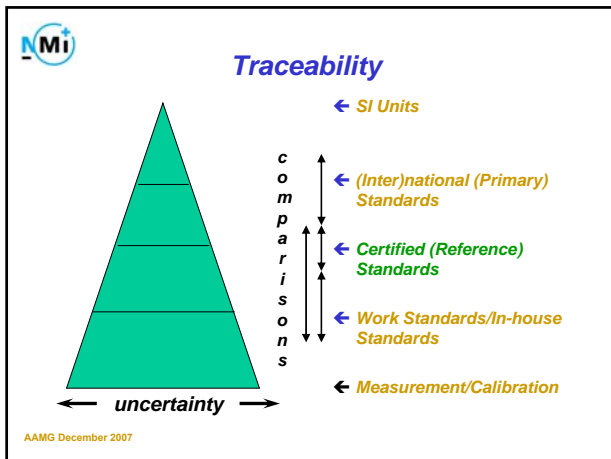
	BP (°C)	Pv (Pa)	Cmax (ppmv)	Cmax (mg/L)
Hexane	69	17332	171053	570
Octane	126	1400	13817	61
Decane	174	127	1253	6.9
Butoxyethyl acetate	192	31	306	2.0
Dodecane	216	13	128	0.84
Naphthalene	218	13	128	0.68
Tetradecane	254	1.5	15	0.12
Hexadecane	287	0.12	1.2	0.010
Octadecane	311	0.0093	0.09	0.0009
Diethylphthalate	295	0.046	0.454	0.0042
Dibutylphthalate	340	0.0023	0.02	0.00026
Di(ethylhexyl)phthalate	385	0.00086	0.0085	0.00014

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Traceability of indoor air measurement results

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Traceability

- ▶ Establishment is prospective → **Validation**
- ▶ Maintenance is required → **Quality Control**
- ▶ Essential element of ISO 17025
- ▶ Can **NOT** be claimed on basis of participation in Inter-laboratory Comparisons !

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What is available ?

- ▶ **Carbon monoxide**
 - ▶ cylinder reference standards
- ▶ **Formaldehyde**
 - ▶ BCR CRM 546 and 553
- ▶ **Nitrogen dioxide**
 - ▶ 'pilot' CRM produced by JRC-ERLAP for Palmes tube diffusive sampler in *Cermatair* project
- ▶ **VOC**
 - ▶ BTX on Tenax
 - ▶ NMI tailor-made CRS of VOC on thermally desorbable sorbents

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Example of benefit of CRS

- ▶ **Measurement of HCBd in indoor air**
 - ▶ CRS at 300 ng
 - ▶ Used for testing liquid spiking procedure for preparation of calibration standards
- ▶ **Effect**
 - ▶ Recovery before use: 85%
 - ▶ Recovery after use: 101%

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Previous Work at NMI (2004)

- ▶ **Certified Reference Standards for VOC**
 - ▶ Two batches
 - ▶ Approximately 50 ng per compound on Tenax
 - ▶ Stable for ≥ 6 months (except aldehydes)

Hexane	1,1,1-Trichloroethane
Benzene	2-Heptanone
Toluene	Toluene
m-Xylene	p-Cymene
o-Xylene	Limonene
α-Pinene	Δ3-Carene
1-Butanol	1,4-Dichlorobenzene
2-Ethylhexanol	2-Butoxyethoxyethanol
Hexanal	Undecane
Octanal	

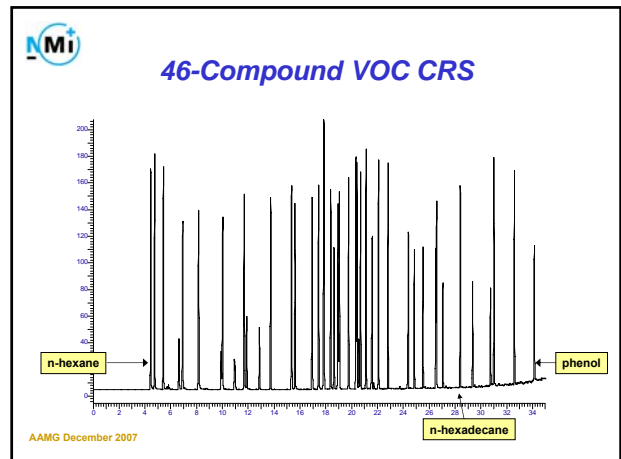
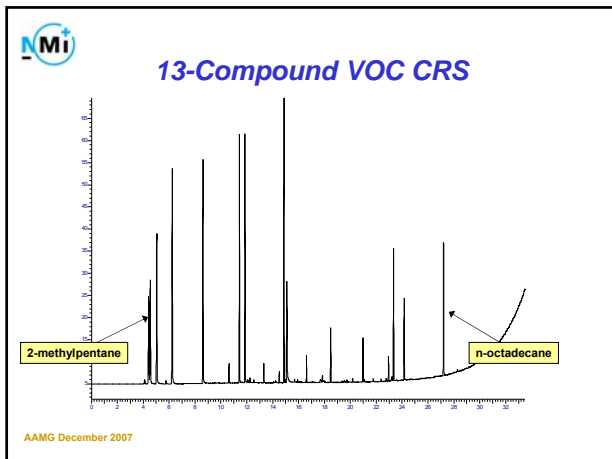
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Current Work at NMI

- ▶ **Production of Certified Reference Standards for IAQ Monitoring of VOC**
 - ▶ Range of C₆ to C₁₈
 - ▶ Approximately 50-200 ng per compound on Tenax
 - ▶ Containing frequently occurring pollutants
 - ▶ By sampling from standard atmospheres
 - ▶ Presenting analytical 'problems' :
 - ▶ separation
 - ▶ desorption efficiency

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- ### Conclusions
- ▶ **Need for representative and comparable indoor air exposure data calls for**
 - ▶ Standardized measurement strategies and methods
 - ▶ Measurement traceability
 - ▶ (System of) inter-laboratory comparisons
 - ▶ **Number of initiatives, but at the “easy” end of the compound range**
 - ▶ **Developments needed at the “rough” end of the range**
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