Type Approval of Ambient-air Measurement Methods to provide Conformance with the Requirements of EU Directives and EN standards

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RSC/AirMonTech Conference on “Current & Future Air Quality Monitoring” RSC 14th & 15th December 2010

What is the National Physical Laboratory?

The UK's national measurement institute
Founded in **1900**
•AOne of the world's leading **National Measurement Institutes**
•6600+ specialists in **Measurement Science**
•SState-of-the-art Facilities for research, calibration, and standards
•TCentre of the UK's **National Measurement System** to support UK's government, business, and society
•EExperienced in **Knowledge Transfer**

Legislative & Economic Drivers for improved air quality measurements

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Number of drivers for better measurements including:

- ***Regulations at national & international levels***
  - ***including international protocols (EC,UN/ECE, Kyoto);***
- ***Economic benefits from comparable measurements***
  - ***emissions trading, exports of goods etc;***
- ***Improved human health and reduced damage to ecosystems;***
- ***All stakeholders (regulators, industry, public etc) pressing for valid internationally-uniform results;***
- ***Pressures also for accreditation to provide greater regulatory and public confidence in the results.***

EU Directives Relating to Ambient Air Quality

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- Suite of ambient air-quality EC Directives published
- from 1996 onwards;
- covers 11 key air pollutants:
- 5 specific gaseous pollutants - SO_2, NO_x, CO, O_3 & C_6H_6;
- 6 particle-bound pollutants (PAHs, heavy metals,PM mass)
- requirements also for ozone precursor measurements of 30 hydrocarbons from C2 to C10
- anions/cations
- and organic carbon/elemental carbon.

New Directive 2008/50/EC

- ***New Directive on ambient air quality and cleaner air for Europe*:**

- major output from the Clean air for Europe (CAFE) Programme- replacing the 1996 "Framework" directive and 3 "Daughter" directives;

- defines responsibilities of "competent authorities" and "national reference laboratories (NRLs)";

- specifies short and long-term ambient-air limit values/target values and other data quality objectives;

- requires international comparisons to demonstrate the comparability of national calibration standards and the measurement uncertainties achieved;

- requires NRLs to be accredited to the EN ISO 17025 standard for ALL relevant QA/QC and other measurement activities they carry out.

- defines reference measurement methods (Annex VI) as those in EN standards;

- Type-approval requirements relate to automated gas methods only since these are specified in EN standards called up by Directive;

- PM automatic methods tested through the "Equivalence" Guidance.

CEN ambient air standards, Overview:

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- **The published EN standards all have the following component parts:**

  - requirement for type approval before use of method by a designated competent body:

  - requirements to demonstrate that the overall measurement uncertainty of the method conforms to the Directive's *DATA QUALITY OBJECTIVES (DQOs);*

  - followed by an initial suitability evaluation at *the selected sites* to ensure that the type approval conditions encompass the site conditions:

  - with subsequent checks at each *SITE* to demonstrate that the method operates within the specifications of the type approval tests;

  - Subsequently operation in the field with regular and on-going quality control/quality assurance requirements

  - requires calibration gases with known measurement uncertainties for checks

  - to ensure all results are produced continuously in the field with uncertainties which are STILL within the Directive's DQOs.

CEN ambient air standards: Type approval tests - Overview

- **Requirements for TYPE-APPROVAL of the specific EN standard reference methods before their use:**
  - **To meet EU Directive's uncertainty requirements;**
  - **Type-approval entails series of *laboratory AND field tests*, to demonstrate conformance with number of performance criteria given in the standards;**
  - **Further requirement for the overall uncertainty to conform with the Directive's measurement uncertainty requirements;**
  - **Member States are required to carry out type-approval tests (or accept other MS approvals!);**
  - **Tests procedures specified in detail in the EN Standards;**
  - **Requirements for known concentration test and calibration gases;**
  - **All tests must be completed successfully BEFORE Method may be used in the field.**

Gaseous pollutants covered by the Directive

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Highest priority species requiring accurate internationally-comparable calibration standards are:

Gaseous Pollutant	EU limit Target/Value	Calibration gas concentration generally required
SO_2	350 ug / m^3 1-hour mean	280 ppb
CO	10 mg / m^3 8-hour mean	10 ppm
NO	30 ug / m^3 NOx Annual limit	(720 ppb)
NO_2	200 µg / m^3 1- hour mean	720 ppb
0_3	120 ug / m^3	200 ppb
C_6H_6	5 µg / m^3 (annual mean)	Few ppb
BTEX 30 component hydrocarbons	ozone precursor measurements required No limit values	1 ppb – 10 ppb
Formaldehyde	Not specified	Few ppb

Calibration gas measurement uncertainties:
- should be small compared with uncertainty requirements in the Directive, and conform with EN standard requirements

CEN Ambient Air Standards

- Requirements for standards was mandated by EC DG Environment to CEN - including type approval and all other components:

- Applicable to continuous/automated measuring methodsusing the following principles:

1. Chemiluminescence for NO_x (NO & NO_2) – EN14211 Certification range 0µg/m^{-3} – 500 µg/m^3
2. Ultraviolet fluorescence for SO_2 - EN 14212 Certification range 0µg/m^{-3} - 1000µg/m^{-3}
3. Non-dispersive infrared for CO - EN 14626 Certification range 0mg/m^{-3} – 100mg/m^{-3}
4. Ultraviolet photometry for O_3 - EN 14625 Certification range 0µg/m^{-3} – 500 µg/m^{-3}
5. On-line gas chromatography for benzene - EN14662-3 Certification range 0.5µg/m^{-3}- 50µg/m^{-3}

- Standards are *mandatory* when Member States report data to the EC *BUT*:

- ***Other measurement principles are allowed if they have been demonstrated to be "equivalent" to the above methods.***

Overview of Type Approval Testing

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- **Type-approval is based on the evaluation of a range of performance characteristics of the continuous/automatic analysers, determined under a prescribed series of tests;**

- **The actual numerical values of the individual performance characteristics are determined by using at least two analysers tested both in the laboratory and in the field (for 3 months);**

- **The numerical values obtained shall each not exceed the values prescribed in the relevant EN Standards.**

Overview of Type Approval Testing

- In addition, all the uncertainty components derived from the individual performance characteristics, shall be summed together (statistically correctly) to obtain the overall uncertainty of the measured concentration:
- this also shall not exceed the prescribed numerical value for the maximum measurement uncertainty given in Directive 2008/50/EC;
- These laboratory and field type-approval tests should generally be performed by an organisation designated by the Competent Authority in the Member State;
- The analyser must be type-approved before use in the field for providing results required by the Directive;
- The Competent authority may accept the testing carried out in another Member State but it needs to convince itself these tests were carried out correctly;
- When significant design changes are implemented AFTER approval this shall be reported by the manufacturer to the Competent Authority or its appointed body.

Ambient air type approval tests

- 17 different performance characteristics are specified for testing in the laboratory (linearity, cross-interference etc)
- 5 more performance characteristics are specified for the 3 month duration field tests (e.g. long-term drift)
- All these characteristics have maximum numerical values (performance criteria) that shall NOT be exceeded
  - no one criterion is allowed to be exceeded
- IN ADDITION there is a further criterion on the OVERALL expanded measurement uncertainty of the Method (at hourly and annual levels)
  - to conform to the requirements of directive 2008/50/EC
- Evaluated according to EN ISO 14956

CEN Ambient air type-approval tests *in the field*

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- 3 month continuous test with 2 ‘identical’ analysers at a selected traffic-orientated monitoring station
  - With common sample system for both if practical;
  - Standard specifies further station and operational requirements.
- Performance characteristics tested in the field are:
  - Long-term drift at zero and span levels;
  - Reproducibility/repeatability over 3 months;
  - Period of unattended operation
    (duration when drift is within its performance criterion);
  - Period of availability
    (percentage of total time that valid results are obtained).
- Field tests used to demonstrate that laboratory determined uncertainties are realistic (as per EN ISO 14956).

CEN Testing Requirements

- **All the tests carried out must be performed in such a way that the *test procedures themselves* do not *FALSELY* cause the analyser performance characteristics to exceed the maximum allowed numerical values:**
- **EXAMPLE 1: Linearity (lack of fit to a straight line): is evaluated by injecting six KNOWN concentrations of the pollutant being measured over the full measurement range of the analyser. The analyser passes the test if the differences between the measured values of the injected concentration do not depart from a straight line by more than a prescribed numerical values.**
  However, *if the concentration of the injected test gases are poorly known these test gases could cause a false fail of the analyser.*
- **EXAMPLE 2: Long-term drift in the field: is evaluated by injecting a known stable-concentration gas mixture into the analyser every two weeks for the 3 month period.**

  If this mixture is not stable in concentration this will indicate falsely that the analyser is drifting out of specification.

CEN testing capabilities at NPL

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Accurate linearity testing at NPL using a binary weighted gas blending network for gas dilutions

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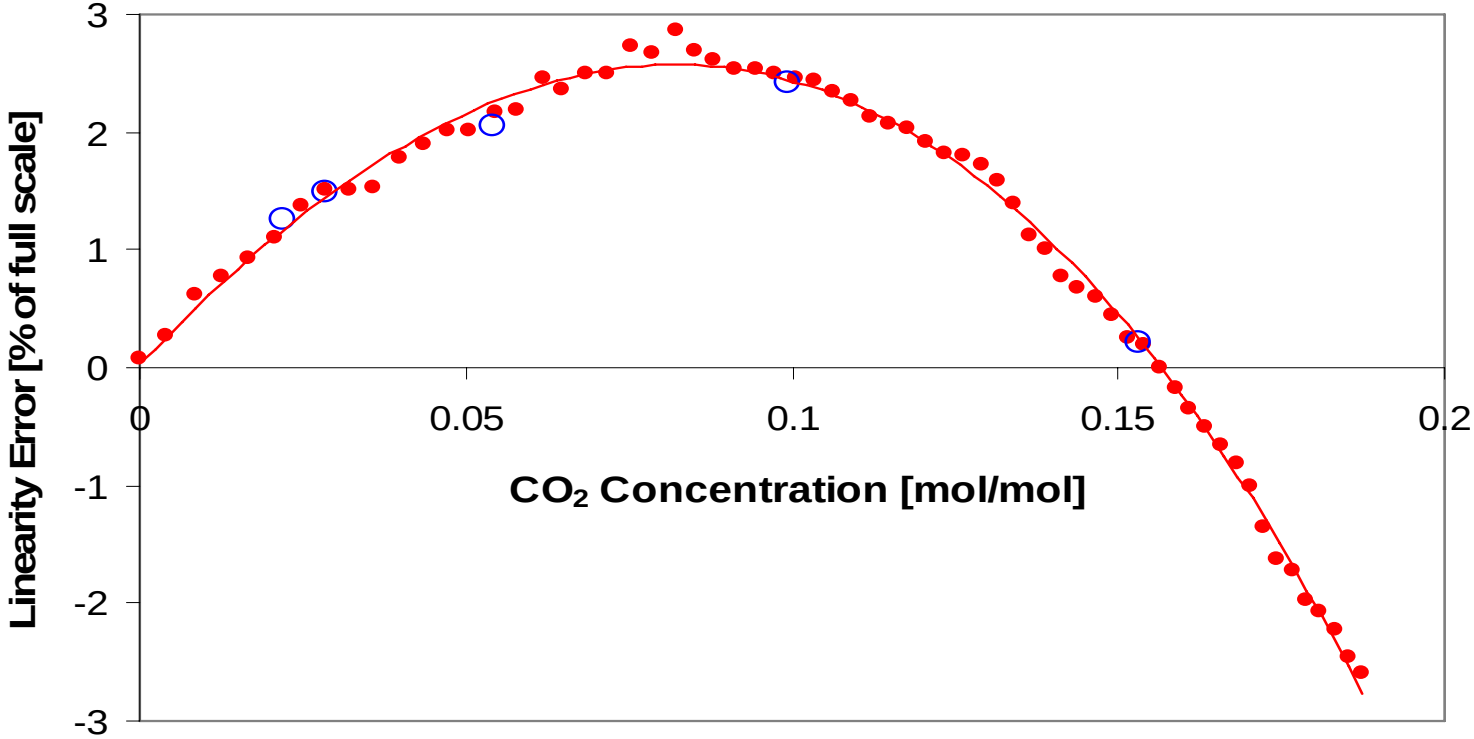

- Residual deviation from a linear response of a non-dispersive infrared analyser: ● Concentrations generated by the binary weighted network; O Primary NPL Gas Standard concentrations;
- *Deviations from the curve show the accuracy and repeatability of the NPL Network generated concentrations.*

Acceptance of CEN Ambient-air type-approval testing

- There is a requirement in Directive 2008/50/EC (Article 3) for laboratories that carry out the type approval tests specified in these EN standards at a Member State designated level should be accredited to the requirements of the EN ISO 17025 standard for the tests they make.
- There is a further requirement in this Directive that Competent Authorities designated in the Directive must accept test reports issued by other Member States where these are correctly and rigorously accredited to ISO 17025 (Annex IV Section E);
- This is proving difficult to implement at present - as the testing that is done so far has not generally been carried out in a manner that fully meets ALL the requirements of the EN standards, and/or is not accredited fully to ISO 17025;
- This and related issues (including the acceptance of “equivalence” testing reports from other Member States) is being discussed actively within the forum of AQUILA, the European Network of National Air Quality Reference Laboratories;
- AQUILA will provide assessments of the technical quality of these reports and guidance on accepting them.

Summary of the Presentation (1)

- **The new Directive 2008/50/EC has strengthened requirements for the measurement of regulated ambient-air pollutants by specifying the use of recently published CEN standard methods for gaseous pollutants – specifying them as *reference methods* for use when reporting data to the EU;**
- **These standards are aimed at ensuring that all the Directive's data quality objectives, including measurement uncertainties, are achieved in the field - for the reporting of the results to the EC and to ensure their comparability across Member States;**
- **These standards require a rigorous set of laboratory and field tests: – type approval before their use, commissioning and testing in the field, on-going QA/QC – to ensure the measurement uncertainty of all the results produced by these reference methods;**
- **Testing institutes need to be accredited to EN ISO 17025 for these tests, *and* there is a requirement that the tests carried out do not give rise to false rejections of the methods due to inadequate test capabilities;**
- ***Other measurement principles to the reference methods are acceptable if they have been demonstrated to be "equivalent" to the reference methods***
- **These tests should generally be performed by an organisation that is designated by the Competent Authority;**

Summary of Presentation (2)

- **It is the responsibility of the Competent Authority in a Member State to agree to accept any testing, *including that from another MS***
  – but it needs to be convinced that all the tests were performed rigorously to the required EN standards and that the test institute was accredited correctly to ISO 17025 for the tests;
- **Mutual acceptance is a sensible aim in future as specified in the Directive - but it is proving difficult to agree presently because of limitations in the programmes of the institutes involved and/or they do not yet meet the accreditation requirements fully;**
- **This issue, and related issues with mutual acceptance of " Equivalence" testing is being discussed within the EC AQUILA Forum:**
  - they plan to provide guidance to the Competent authorities, and technical reviews of (published) test reports;
- **International comparisons are carried out by National Measurement Institutes in many EU countries and elsewhere to demonstrate the comparability and accuracy of the national measurement standards;**
- **The Directive also requires comparisons to be carried out by the EC JRC (or similar) of the results obtained by the National Reference Laboratories**
  - to demonstrate that the reference methods are implemented fully and accurately.

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Thank you for your attention - Any questions?

Mutual Recognition Agreement for National Standards by other Countries

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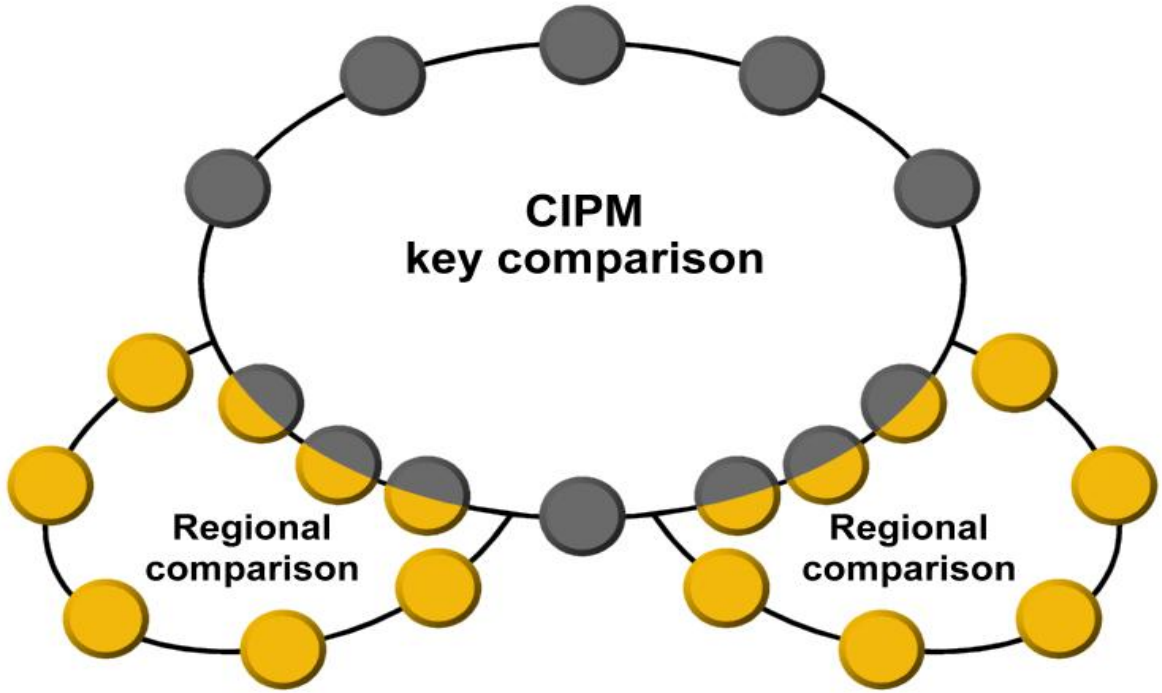
- The scientific basis of the MRA is:
  - the results of key and supplementary comparisons by participants on a regional or worldwide scale, and the linked calibration and measurement capabilities (CMCs) of the national metrology institutes



Mutual Recognition of National Standards by other Countries

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- **International Comparisons of national calibration standards for sulphur dioxide at 280ppb –stability tests also;**

Degrees of Equivalence

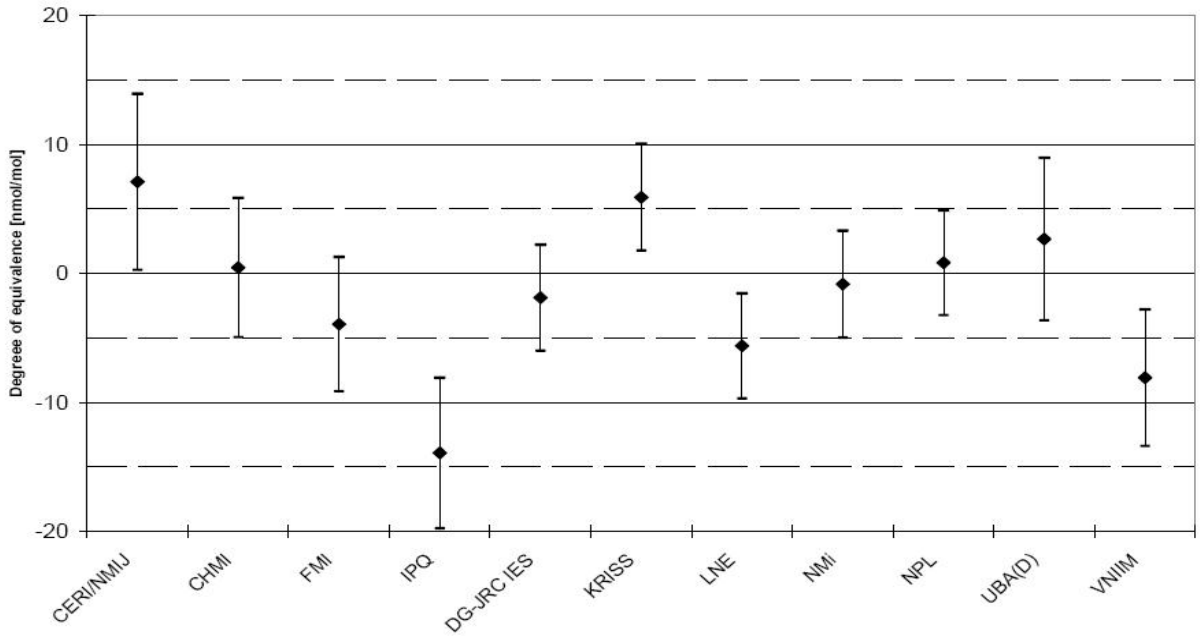

Figure 3 – Degrees of equivalence for CCQM K26b.

- **Results generally agree with the assigned value to ± 5 nmol/mol in 280 nmol/mol standards i.e. ~ ± 1.8%**

- Overall EC uncertainty requirements for the measurements ± 15%