ISO/TC 158

Analysis of gases

MACPoll

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Scope TC 158

- ISO/TC 158 is the technical committee for standardization in the field of gas analysis, including
  - Terminology
  - Preparation of calibration gas mixtures
  - Sampling / handling / proper use of calibration gases
  - Analytical methods including evaluation of characteristics of analysers.
  - Quality assurance in gas analysis

Areas of gas analysis

- Gas analysis plays an important role in:
  - Environmental analysis, both global and localized pollution measurement
  - Natural gas, LNG, other energy gases and alternative fuels
  - Gas analysis in the hospital
  - Development and calibration of sensors
  - Electronics industry / lightning
  - Safety / work place atmospheres
  - Legal metrology

Participants

- At present participants in the TC come from
  - Specialty gas suppliers
  - Metrology Institutes
  - Consultants in gas analysis
- No participation from
  - Users of calibration gases
  - Gas analyzer manufacturers
- Liaisons with TC 146 ,193, Remco and EIGA
- Active participation from UK, DE, FR, NL, SE

Formal Membership

- 15 P-members:
  - Belgium, Czech Republic, France, Germany, Italy, Kenya, Korea, Netherlands, Poland, Portugal, Russia, Spain, Sweden, Ukraine, United Kingdom
- 26 O-members:
  - Australia, Austria, Bosnia-Herzegovina, Brazil, China, Croatia, Cuba, Denmark, Egypt, Finland, Hong Kong, Hungary, India, Indonesia, Ireland, Japan, Pakistan, Romania, Saudi Arabia, Serbia, Slovakia, South Africa, Switzerland, Thailand, Tunisia, Turkey
- 23 standards published

Highlighting 158 Standards

- ISO 6142; preparation of calibration gas mixtures – gravimetric method
  - Most accurate method to prepare cylinders with mixtures of known composition
  - Reference method for all metrology institutes and specialty gas producers
  - Currently a revision is started
    - Introduction of 2 classes depending on type of verification
    - Purity analysis moved to separate standard

Highlighting 158 Standards

- ISO 6143; Comparison methods for determining and checking the composition of calibration gas mixtures
  - High quality standard for certification of gas mixtures
  - State of the art regression analysis; clear acceptance criteria
  - Evaluation of uncertainty budgets

Highlighting 158 Standards

- ISO 6145; dynamic methods for preparation of calibration gases
  - For mixtures not stable in cylinders
  - Methods covered:
    - Dilutors:
      - volumetric pumps; capillary devices; critical orifices
    - Permeation / diffusion / saturation
    - Continues syringe injection
    - Thermal mass flow controllers

Highlighting TC 158 Standard

- ISO 14912; Conversion of gas mixture composition data
  - Conversion of common units in gas analysis
    - mol/mol - vol/vol – vol% - mol% - mg/m^3
    - uncertainty propagation included
- ISO 16664; Handling and use of calibration gases and gas mixtures – Guidelines
  - Good practices in using expensive calibration gas mixtures

Work Programme

- Revision of ISO 6145-1, Gas analysis – Preparation of calibration gas mixtures using dynamic volumetric methods – Part 1: Methods of calibration
- Revision of ISO 6145-6, Gas analysis – Preparation of calibration gas mixtures using dynamic volumetric methods – Part 6: Critical orifices
- Revision of ISO 6141, Gas analysis – Requirements on certificates for calibration gases and gas mixtures
- Revision of ISO/TS 14167 , Gas analysis – General guidelines on quality assurance aspects in the use of calibration gas mixtures

Work Programme

- New Standard in preparation: ISO 12936; Measurement protocols and data evaluation techniques for general analytical applications
  - single point exact-match calibration (SPEM),
  - single-point through origin calibration (SPO),
  - two-point calibration using a single point and a blank (TPB),
  - two-point calibration: bracketing using two reference standards (TPC),
  - multi-point calibration (MPC) using at least three calibration points.

Workprogramma

- Revision of ISO 7504 (Vocabulary)
  - Definition of zero gas:

    "*gas or gas mixture with sufficiently low content of the component(s) of interest, used to produce the zero response of a given instrument for a given range of content*"

    Note 1 to entry: A component of interest could cause interference in the instrument.

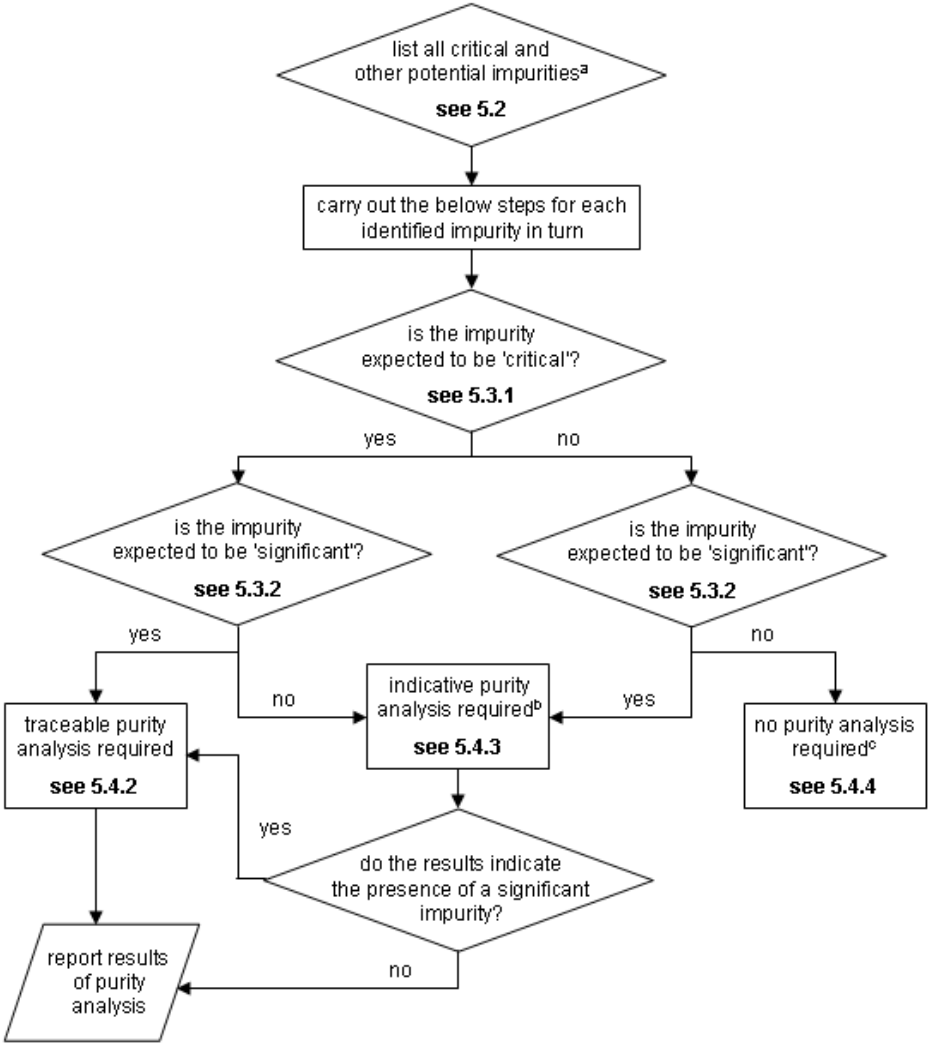
Possible new work

- Specification of performance characteristics of gas analyzers. (future ISO 13280)
- Sampling Guidelines

Purity analysis

- New standard in preparation (ISO 19229)
- Applicable for both ISO 6142, 6144 and 6145 standards
- Can we expand to accommodate zero gas protocols?

ISO 19229, principle



Is an impurity critical and or significant?

Traceable analysis required?

How to estimate the uncertainties

FprEN 14212:2012; SO2 by UVF

- Clause 8.4.2.3 Test gases: For the determination of the various performance characteristics, test gases traceable to (inter)nationally accepted standards shall be used unless otherwise stated in this European Standard. Methods for the generation of test gases are given in Table 3.
- Table 3 lists methods: ISO 6142, 6143, 6145-6, 6145-7, 6145-10 or ISO 6144
- The uncertainties in zero and span gases used for the laboratory and field tests shall be proven to **be insignificant**. Possible contamination of zero and span gas shall not significantly influence the results of the laboratory tests. Therefore the span gases and zero gas shall meet the following specifications:
  - Maximum permitted expanded uncertainties (95 % confidence) in the concentration of gases used for laboratory tests: 3 %;
  - Tolerance of the concentration levels for gases other than water vapour: ± 10 %; for water vapour the tolerance is ± 2 %.

Spec's on zero gas

- Example: FprEN 14212:2012
  - Table 4b – specification for purity of zero gas for interference testing

Pollutant	Concentration
H_2S	≤ 0,1 µmol/mol
NH_3	≤ 2 nmol/mol
NO	≤ 1 nmol/mol
NO_2	≤ 1 nmol/mol
m-Xylene	≤ 1 nmol/mol
Water vapour	≤ 150 µmol/mol
SO_2	≤ 1 nmol/mol

  - ➢ Critical impurities??? Significant???

Test mixtures

- Test for sensitivity with following gas mixtures:

Performance characteristic	Performance criterion for SO_2
H_2O 19 mmol/mol	≤ 10 nmol/mol
H_2S 200 nmol/mol	≤ 5 nmol/mol
NH_3 200 nmol/mol	≤ 5 nmol/mol
NO 500 nmol/mol	≤ 5 nmol/mol
NO_2 200 nmol/mol	≤ 5 nmol/mol
M-xylene 1µmol/mol	≤ 10 nmol/mol

The concentration of the mixtures of the test gases with the interferent shall have an expanded uncertainty of ≤ 5 % and shall be traceable to (inter)nationally accepted standards

Calibration requirements

- Zero and span gas can be generated (after dilution, if required) by an external calibrator unit or internally in the analyser. The concentration *ct of the span gas shall be around 70 % to 80 % of the maximum of the certification* range or the user-defined range (see 9.5.1).

- The stability of gases used for span and zero checks shall be verified at least every six months with use of reference gases traceable to (inter)nationally accepted standards. The zero gas shall give no instrument readings higher than the detection limit, and the gas used for span checks shall not differ by more than 5 % of the last measured value.

- The purity of the gases may be as specified in Table 4. However, it is possible to relax the impurity specifications in the gases for water vapour and ammonia. In this case the uncertainty due to the presence of excess impurities shall be included in the uncertainty budget where significant.

Relations to ISO TC 158

- All listed methods for test gas preparation originate from TC 158
- Purity analysis is important for all methods of preparation at these low levels
- Traceability is required

Questions

- Can this work be done by the environmental labs themselves?
  - Maybe with sufficient guidance…
    - Methods, equipment
  - Can they test generators? Frequency?
- Can ISO help?
  - New standards, methods?

Way forward

- Let’s discuss during the afternoon sessions

- But in any case…..

Join ISO TC 158

- TC 158 Standards are widely is use as methods in producing and analyzing gas mixtures
- For easy acceptance in accreditation
- Your input can help to improve the standards
- No participation = no input = no influence on content
- Standards should continue to describe the state of the art in gas analysis

More information

- www.iso.org >> technical committees >> list of technical committees >> ISO/TC 158
- ISO/TC 158 secretariat: NEN – Netherlands Standardization Institute >> energy@nen.nl
- Your national standardization body

Thanks for you attention