# *CCM WG Low pressures: Report to CCM 2013*

**Content of presentation**

1. **Membership/Meetings**
2. **Reports on comparisons**
3. **General problems for discussion**
4. **New developments, plans**

**CCM WG LP in May 2011**

# *CCM WG Low pressures – Membership/Meetings*

**Membership (20 NMI)**

AStar (Singapore), CENAM (Mexiko), CEM (Spain), CMI (Czech Republic), INMS-NRC (Canada), INRIM (Italy), KRISS (Korea), LNE (France), METAS (Switzerland), MSL-NZ (New Zealand), NIM (China), NIST (USA), NMIA (Australia), NMIJ (Japan), NMISA (South Africa), NPL-I (India), PTB (Germany), SMU (Slowakia), UME (Turkey), VNIIM (Russia)

Personal member: Dr. Janez Setina (MIRS, Slovenia)

Regularly invited: INMETRO (Brasil), IPQ (Portugal)

In total 31 individuals.

**Meetings:** Typically every 3 years. Next meeting probably Sept 2013.

# *CCM WG Low pressures - KCs*

**CCM.P-K12** (Leak/flow rates at $8 \times 10^{-14}$ mol/s and $4 \times 10^{-11}$ mol/s):

Participants: 11 NMIs (APMP, COOMET, EURAMET, SIM)

Pilot Lab: PTB

Measurements: 2007-2009

Draft A: approved in July 2010

Final report published: December 2012

Agreement at last CCM WG CMC meeting 2011: new service category 9.4.2 „Molar flow rate“ to adopt new CMC entries from participants.

# *CCM WG Low pressures - KCs*

**CCM.P-K12.1** (Leak/flow rates at $3 \times 10^{-11}$ mol/s):

Participants: IMT/CMI bilateral

Pilot Lab: IMT

Planned (motivation: CMI showed inconsistent data in K12)

# *CCM WG Low pressures - KCs*

**CCM.P-K14** ($10^{-4}$ Pa to 1 Pa):

Participants: 7 NMIs (APMP, EURAMET, SIM)

Pilot Lab: METAS

Measurements: 2010-2011 (within 12 months!)

Draft A: January 2013 still confidential, consistent results.

# *CCM WG Low pressures - KCs*

**CCM.P-K3.1** ($3 \cdot 10^{-6}$ Pa to $9 \cdot 10^{-3}$ Pa):

Pilot Lab: NIST

Participants: bilateral NIST/PTB due to non-equivalence of PTB

Measurements: 2011-2012

Draft A: expected May 2013. Preliminary result: Equivalence of PTB proved after repair of standard.

# *CCM WG Low pressures - KCs*

**CCM.P-K4.2012** (1 Pa to 10 kPa):

Participants: 7 NMIs (AFRIMETS, APMP, COOMET, EURAMET, SIM)

Pilot Lab: NIST

Protocol complete

Measurements: running since January 2012

# *CCM WG Low pressures - KCs*

**CCM.P-K3.201X** ($3 \cdot 10^{-9}$ Pa to $3 \cdot 10^{-4}$ Pa):

Decided 2011.

Pilot Lab: NMIJ

Protocol under development, expected spring 2013.

# *CCM WG Low pressures – Comparisons (pressure)*

CCM

RMO

Other

CCM.P-K3 (2002/2010, 2013…)

NIST, PTB, NPL-UK, IMGC, NPL-I, KRISS

CCM.P-K3.1 (2009…) NIST, PTB

$3 \cdot 10^{-6}$ ... $9 \cdot 10^{-3}$

CCM.P-K14 (2010…) follows K9

METAS, PTB, INRIM, NIST, CENAM, NMIJ, KRISS

$1 \cdot 10^{-4}$ ... 1

CCM.P-K4 (2002, 2012…)

NIST, PTB, NPL-UK, IMGC, NPL-I, CSIRO, KRISS

1 ... 1000 (10,000)

EUROMET.M.P-K1.b (2000-2004)
PTB, BNM-LNE, CEM, IMT,IMGC, NPL-UK, UME
M.P.K1.b.1 (2008…) SP, PTB
$3 \cdot 10^{-4}$ ... 0.9

EUROMET.M.P-K1.a (1999-2004)

IMGC, BNM-LNE, PTB, CEM, OMH, MIKES, SP, NMI, NPL-UK, UME

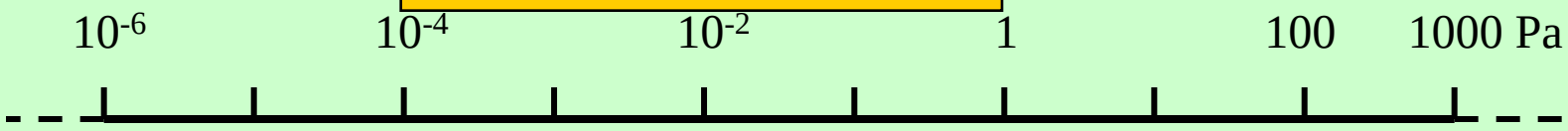
(0.1) 1 ... 1000

SIM-EUROMET.M.P-BK3

(2002-2004)

PTB, CENAM

$3 \cdot 10^{-4}$ ... 0.9



# *CCM WG Low pressures – Comparisons (pressure)*

RMO

EURAMET.M.P-S7 (**2007**…)

METAS, CMI, EIM, MIKES

$1 \cdot 10^{-4}$ ... 1

EUROMET.M.P-S2 (2007)

PTB, CMI 30 ... 3000

EURAMET.M.P-K4.2010 (2010…)

CMI, INRIM, LNE, MIKES, PTB

1…15 000

APMP.M.P-K3 (2010)

KRISS, NMIJ

$3 \cdot 10^{-6}$ ... $9 \cdot 10^{-3}$

APMP.M.P-K4 (**2002**…)

NMIJ, CMS-ITRI, KRISS, NIM, NIMT, NPL-I, SPRING

1 ... 1000

Other

APMP.M.P-S2 (2008)

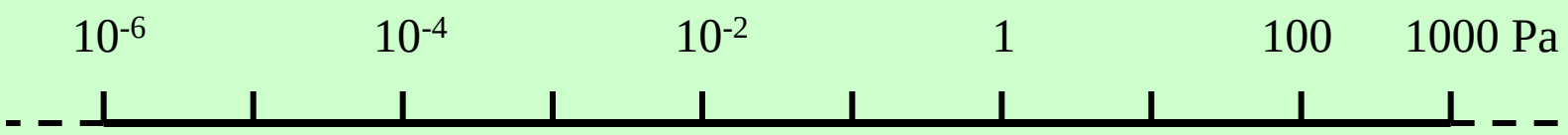
NPL-I, NIST

0.05

SIM.M.P-S1 (**2007**…)

NPL-I, NIST

100 … 70 000



# *CCM WG Low pressures – Comparisons (leak rate)*

CCM

CCM.P-K12 (2012)

PTB, ASTAR, CMI, IMT, INRIM, LNE, NIM, NIST, NPL-I, NMIJ, VNIIM

$10^{-14}$ and $4 \cdot 10^{-11}$

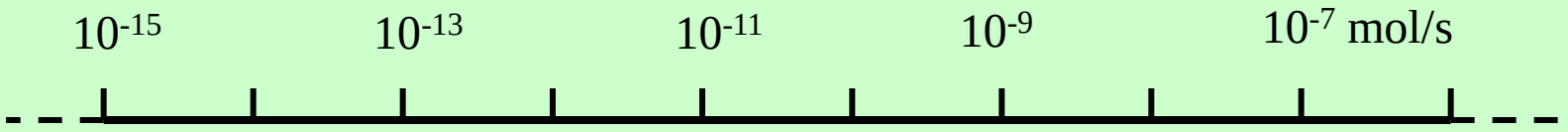
CCM.P-K12.1 (2013…)

IMT, CMI

$3 \cdot 10^{-11}$

RMO

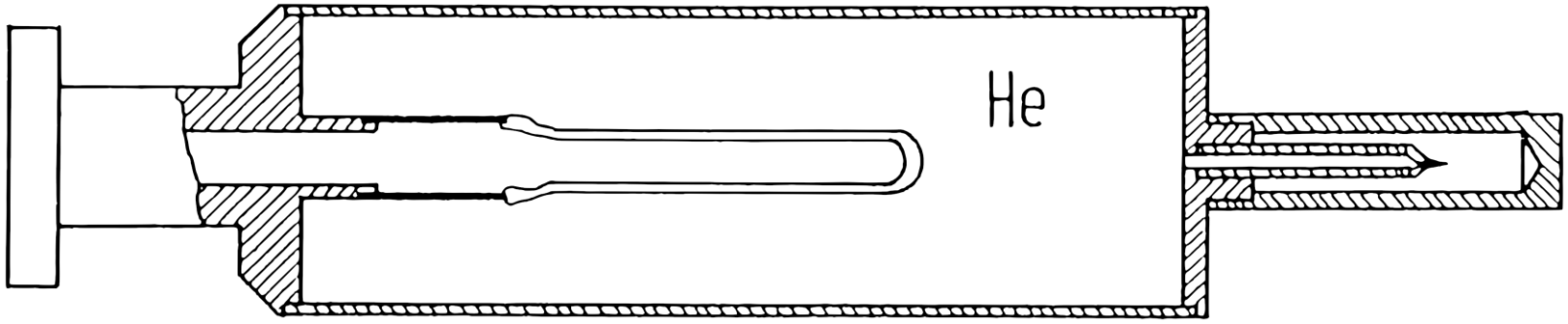
Other



**Problems that came up with K12 – Introduction**

Quantity to be measured and compared:

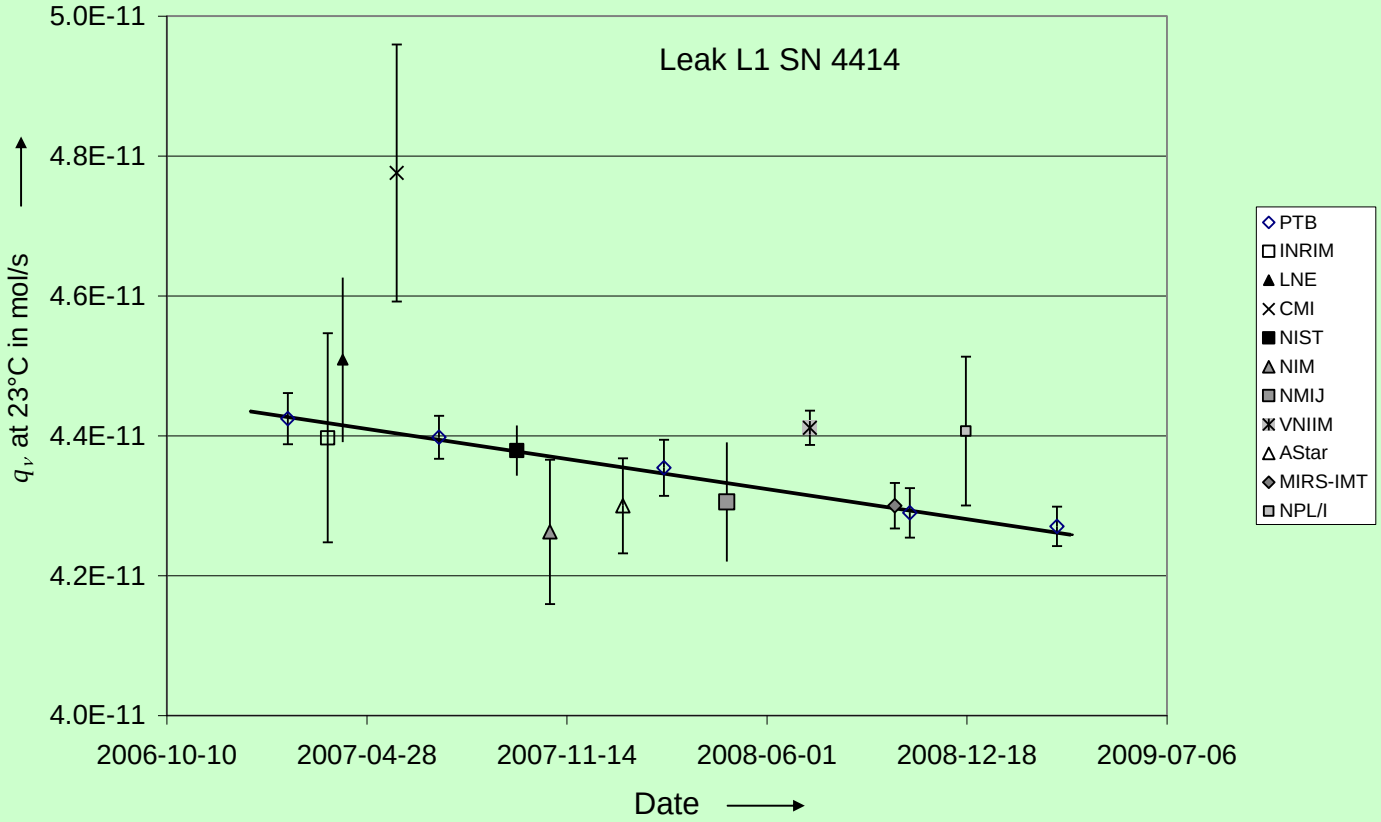
$$q_\nu = \frac{\Delta \nu}{\Delta t} \qquad \text{at 23 C.}$$



# *CCM WG Low pressures: Problems with P-K12*

The linear drift of the transfer standard was not a principal problem, but intensified the problem with inconsistent data due to

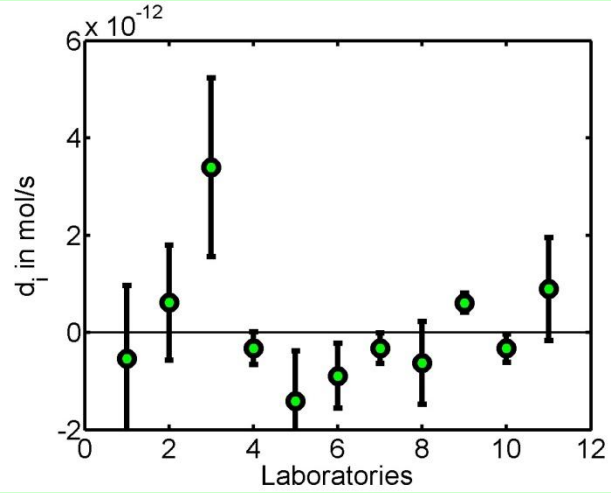
- The influence of inconsistent data on the slope
- Lack of existing and published evaluation methods for a drifting standard, particularly with inconsistent data.

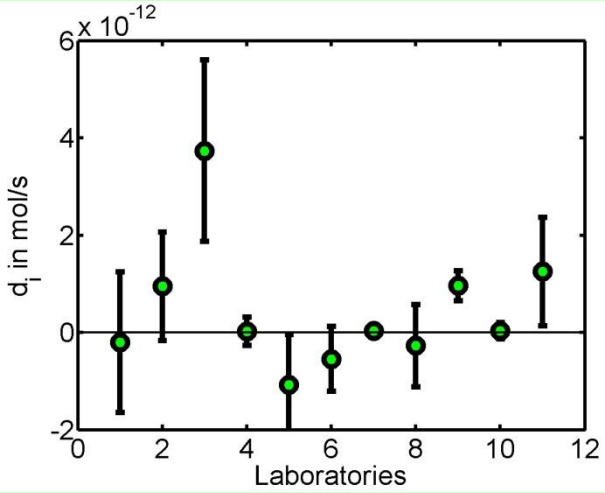


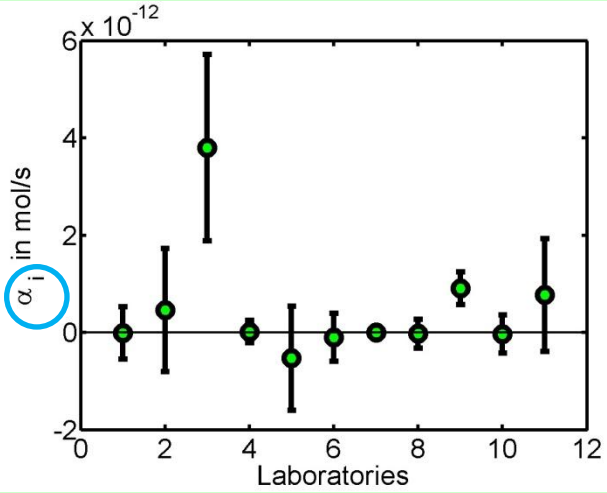
# *CCM WG Low pressures: Problems with P-K12*

## Degrees of equivalence $d_i = x_i - x_{ref}$ , $u(d_i)$

**Zhang** **LCS method** **BMA method**




$X_i = c_i + \beta_{Pilot} t + E_i$ , $X_i = \mu + \beta t + E_i$ $X_i = \mu + \beta t + \alpha_i + E_i$

bias

# *CCM WG Low pressures: Problems with P-K12*

- Statistical approach accounting for linear trends [1]
- Largest consistent subset (LCS) method* [2]
- Bayesian model averaging employing a fixed effects model* [3]

***Extended for linear drift**


[1] Zhang N F, Liu H, Sedransk N and Strawerman W E 2004 Statistical analysis of key comparisons with linear trends, *Metrologia* **41** 231-237.

[2] Cox M G 2007 The evaluation of key comparison data: determining the largest consistent subset, *Metrologia* **44** 187-200.

[3] Elster C and Toman B 2010 Analysis of key comparisons: estimating laboratories' biases by a fixed effects model using Bayesian model averaging, *Metrologia* **47** 113-119.

# Conclusions from CCM.P-K12

**The MRA Technical Supplement T2**:

“The degree of equivalence of each national measurement standard is expressed quantitatively by two terms: its deviation from the key comparison reference value and the uncertainty of this deviation (at a 95 % level of confidence)”

**does not allow any statistical evaluation of the degree of equivalence.**

**Do we really want to have this door closed ???**

**Note:** At present no bias is assumed to evaluate RV, then this RV is used to select, which labs have a biased value (and determine a consistent subset).

## Conclusions from CCM.P-K12

**As a consequence, finally we applied the random effects model** that explicitly recognizes the possibility that the between-laboratory variability may exceed the typical measurement uncertainty associated with the individual measured values.

- Rukhin (2009) Metrologia 46, 323 – 331
- Toman and Possolo (2009) Accreditation and Quality Assurance 14, 553 – 563

**Disadvantage:** Higher uncertainty of reference value.

# Conclusions from CCM.P-K12

**CIPM MRA-D05 says in Section 4.7** "Once the final version of Draft A, which includes the proposed key comparison reference value and degrees of equivalence, is approved by the participants, the report is considered as Draft B."

What means "approved"? Possibility of "Veto"?

Richard Davis: Yes, all need to agree.

Make this clearer in **CIPM MRA-D05!**

Note: In the first KC guideline (1999) Draft A was a mere presentation of the results, only Draft B had to contain a RV.

# Conclusions from CCM.P-K12

**Further it is written in MRA-D05 in Section 4.7:**

"In the event of disagreement concerning the results or the interpretation of the results of a key comparison, which cannot be resolved by the participants, by the key comparison working group or by the Consultative Committee, the matter is referred to the CIPM for a decision."

**This is no clear procedure! Who would decide then after the WG cannot agree? The CCM chair? The CCM delegates? How will they decide? With majority?**

Make this clearer in **CIPM MRA-D05!**

# Conclusions from CCM.P-K12

## Again MRA-D05 in Section 4.7:

"Once the final version of Draft A, which includes the proposed key comparison reference value and degrees of equivalence, is approved by the participants, the report is considered as Draft B. It must then be submitted for approval by the corresponding Consultative Committee. At this stage, the results are not considered confidential and **can be used to support CMCs** and can be used for presentations and publications**, except for the key comparison reference value and the degrees of equivalence** which must be considered confidential until they are approved by the Consultative Committee and published in the KCDB."

## CMCs can be supported before agreement on reference value?

**Confusing!** Make this clearer in **CIPM MRA-D05!**

**Environmental and safety regulations**

Customer request calibration of sniffer test leaks (test leaks with flow into atmosphere)

NMIs established calibration standards for this:

CMI, INRIM, LNE, PTB

Others are planning.

Next meeting: Discussion of KC for molar flow rate against atmosphere.

# *CCM WG Low pressures – Other activities*

Research for establishing pressure scale (> 1 Pa, < 400 kPa) by refractive index measurement of helium by NIST

Dynamic vacuum pressure measurement (PTB, EMRP IND 12): Achieved in 1/2013: Within 18 ms from 100 kPa to 100 Pa.

Cooperation between NMIs and „rarefied gas dynamics“ community to improve predictability of gas flows without calibration (EMRP IND12).

Establishing traceability for partial pressure measurement and outgassing rate measurement (implications for mass comparisons in vacuum?) . Collaboration with ISO TC 112.