Report of the meeting of the CCM Working Group on Low Pressures on Feb 25, 2014, at the BIPM in Sevres

Karl Jousten, PTB Berlin, Chair WGLP

Final version after corrections of participants: 2014-03-17

Appendix 1 list the participants. Appendix 2 contains a summary of the workshop on SRG stability held the day before.

The participants agreed to the following final agenda:

1. Welcome, taking of the minutes and membership of CCM WG LP
2. Publication of CCM.P-K12 (leak) and its impact
3. Use of units for leak rates in Japan and discussion
4. CCM.P-K12.1: Progress report
5. Results of CCM.P-K3.1
6. Results and discussion of CCM.P-K14 (SRG)
7. Status on CCM.P-K14.1
8. Progress and first results of CCM.P-K4.2012 (1 Pa to 10 kPa), discussion
9. Pilot study CCM.P-K3: Progress report
10. Next key comparisons organised by the WG LP, Report of WG LP
11. APMP.M.P-K4 and K14: Progress reports
12. Results of EURAMET.M.P-K4.2012 (1 Pa to 15 kPa, FPG as transfer standard)
13. Status of supplementary comparisons (EURAMET.M.P-S7, -S10 and SIM.M.P-S1)
14. New developments in NMIs
15. Future developments and challenges for vacuum metrology and ongoing projects (EMRP IND12)
16. Information on new ISO standards on vacuum metrology
17. New CCM and JCRB documents and forms, new guidelines
18. Various (next meeting)
19. Closure

The following report will refer to this numbering of the agenda.

1. The chair of CCM WG LP, Karl Jousten, welcomed the attendees and thanked for their coming. Nieves Medina, CEM, agreed to take the minutes.
   The following 3 members of the WG LP were neither active in the past years nor
present at the last two meetings and shall be asked, if they still want to be a member of the WG LP: A*Star (Singapore), INMS-NRC (Canada), and NMIA (Australia). From SMU (Slovakia) it is known that Peter Farar has retired. For this reason also SMU shall be asked, if they can actively participate in the future. Some NMIs named new representatives: Ashok Kumar (NPL/I), Jackson Silva Oliveira (INMETRO) and Rifat Kangi (UME). For another two it is still unclear, if the representatives of this meeting will be this also the ones in the future: Kenta Arai (NMIJ) and Irina Sadkovskaya (VNIIM).

2. Publication of CCM.P-K12 (leak rates) and its impact
The final report of the comparison was published in the Technical Supplement of Metrologia in December 2012. PTB and NMIJ have approved corresponding CMCs. Other participants should follow. As a consequence, laboratories are now able to be accredited for leaks calibration (the document ILAC-P10 allows this only, if traceability to CMCs is given).

Unfortunately, NMIJ used the unit of Pa m³/s for their CMCs instead of mol/s, which was approved by the Flow Technical Committees from the corresponding RMOs. This is discussed further in point 3.

After the publication in Metrologia, Takeo Tsuchiya, Secretary of ISO/TC 135 (Non-destructive testing)/SC 6 (Leak detection methods) approached the pilot laboratory for help in drafting an international standard for helium leak calibration. This shows that comparisons do have impact on standardization.

In the final discussion of the K12 report it became clear that the rules for managing KCs are not clear enough in case of disputes between participants (no consensus). After Jousten’s report on the last CCM meeting on this, which included a proposal, the new CCM chair took measures and issued the new document “CCM_Guidelines_on_FinalReports.pdf”, which clarifies the rules of KCs management for the CCM in better detail. This document is available on the BIPM/CCM website.

As a further consequence of the K12 it became clear that a statistical evaluation of the reference value and the degree of equivalence, which at present interpretation is in contradiction to the MRA, should be made possible. Jousten made a pertinent proposal to the CCM, but it became clear that the MRA wording cannot be changed and that such an initiative should be made by the NMIs represented in the CIPM. Jousten has
taken such an initiative at PTB, but other participants and members of the WG are encouraged to do so as well.

3. Use of units for leak rates in Japan and discussion
Kenta Arai from NMIJ makes a presentation: In principle it is better to express leaks in mol/s because the corresponding temperature dependency is avoided, but Japanese standards and Japanese manufacturers of leak detectors use the unit Pa m³/s. That is the reason why the CMCs have been applied in Pa m³/s to avoid any misunderstanding.

Karl Jousten explains that it is not intended that leak detectors should indicate mol/s instead of Pa m³/s or mbar L/s. The purpose of using mol/s is that the user of an NMI certificate of a standard leak is aware of the two temperature dependencies of it: the temperature of the permeation or capillary element and the temperature at which the helium pressure is measured. The use of mol/s forces the user to think about this.

It is agreed that at least a reference temperature at which pressure is measured is included in the Japanese CMCs declaration, if not changed to mol/s. In this context, Jousten also encourages NMIJ to use only one CMCs entry for the full range instead of three CMCs for three sub ranges.

4. CCM.P-K12.1 (Progress report): The participating institutes are MIRS and CMI. Measurements have been completed a year ago. Final results were received in September. Evaluation and Draft A are in work.

5. Results of CCM.P-K3.1: The participating institutes were NIST and PTB and the measurement range was high and ultra high vacuum. Two SRGs have been used as transfer standards for 9·10⁻⁴ Pa and two Stabil-Ion gauges from 3·10⁻⁶ Pa up to 3·10⁻⁴ Pa with a normalisation to the value at 9·10⁻⁴ Pa. The gas medium was nitrogen. Ionization gauges measurements performed by NIST differ from 1.2 % up to 2 % and PTB results are in the range from 0.4 % up to 1.5 % taking the NIST measurements average as a reference. The maximum difference between PTB and NIST SRGs measurements is 0.2%. All the results are in excellent agreement and the standards equivalent.

The comparison results and the Draft B report are approved by the Working Group.
6. CCM.P-K14 (SRG)

Christian Wüthrich (METAS) or a deputy from METAS could not attend the meeting. Measurements were completed in 2011. Draft A is available since January 2013. Jousten reports that he answered some questions by Wüthrich in the preparation of Draft B. METAS should complete the work as soon as possible.

7. Status on CCM.P-K14.1

This is a bilateral comparison between METAS (pilot) and NIM. First Draft A was completed and commented by NIM in 2013. Draft B is awaited.

8. CCM.P-K4.2012: Jay Hendricks presents the results in anonymous form. The measurement range is from 1 Pa up to 10 kPa. The transfer standards are two resonant silicon gauges and two CDG (133 Pa). The participating institutes are CMI, PTB, NMISA (who could not deliver any data, since a staff member passed away, and did not participate in the end), NMIJ, VNIIM, CENAM and NIST (pilot). Measurements were completed within 18 months. NIST has performed three measurement sets for this comparison with its 130 kPa mercury barometer as the reference. The applied corrections are offset and thermal transpiration. Youden plots have been performed for each measurement pair. RSGs measurements present higher dispersion for one gauge than the other one; however CDGs measurements agree completely. In general terms results are in agreement, 90% for $k = 2$ (weighted mean is not used). Draft A is expected by the end of March. Gauge stability is good enough to ensure the declared CMCs by the participants.

9. Pilot study CCM.P-K3: The measurement range compared to previous K3 shall be extended to comprise $3 \times 10^{-9}$ Pa to $9 \times 10^4$ Pa.

The study intends to study the transfer standards stability for ultra high vacuum and the influence of baking (200 °C is the maximum temperature), in particular in the (new) 3 extended lower decades. The participating institutes are NMIJ and PTB. The measurements were recently completed. The transfer standards are an extractor gauge, an AxTran (AT) gauge and two SRGs. Nitrogen is used as the gas medium to generate the pressure. The first SRG suffered a high drift (more than 3%), but the other did not (its rotor material was Invar). The transportation spring was loose for the SRG that suffered the drift and there were leaks in both SRGs after baking at PTB due to too long screws in tapped holes.
Also the extractor gauge suffered a considerable drift (around 5%) taking the NMIJ standards (XHS and DES) into consideration. This did not happen with the AT gauge, where the results obtained by both NMIJ standards and PTB are in agreement.

The different tests performed before this study at NMIJ were more encouraging. The bent beam gauge (3B) was also investigated. There was no significant different result (stability) for argon and nitrogen either. The baking tests showed no significant influence of baking and posterior air exposure. The study of the temperature influence shows it is important to wait until stabilization.

The study for long term drift shows variations from 8% up to 13% in 13 years for AT and extractor gauges.

On the last meeting CMI indicated that they may be ready by 2013 with a suitable standard to participate in this new K3. Prazak, however, commented that the standard is still under development. Since at the moment NMIJ is too busy with APMP comparisons to make the necessary changes with the transfer standards for K3, it is agreed that the measurements should start only in 2016. This delay gives the CMI more time to make a decision, if they can take part or not.

10. Next key comparisons organised by the WG LP and report of WGLP to CCM chair: Jousten shows the document listing the KCs to be performed regularly by the WG LP. Due to the fact that in Europe there are several, mainly environmental, regulations which require calibrated standard refrigerant leaks against atmosphere, in Germany, the first companies indicated that they want to be accredited for this kind of calibration. For this reason, a key comparison for leaks against air is proposed, starting 2017. The participating institutes could be LNE, CMI, PTB, INRIM from EURAMET and NMIJ and CENAM from other regions. The transfer standard could be a refrigerant gas leak with a few grams/year loss (8·10^{-6} Pa m^{3}/s at 23°C or 3·10^{-9} mol/s) and the comparison will be for one single value. Jousten asks, if LNE could pilot such a comparison. Pierre Otal is in favour of this, but needs to ask the LNE management for approval. He will inform the chair about the decision.

The document listing the KCs to be performed regularly is updated with this comparison. It is confirmed that only one KC should be started in a triennium so that adding another KC to the four existing ones will extend the repetition period to 15 years. Jousten explains that this is in line with the request of the new CCM chair who
requested proposals to extend the repetition period of KC in the frame of the new CCM strategy.

Jousten shows the updated draft of the annual report (due Feb 28) of the WG LP to the CCM chair. It is agreed that each participant should have the chance to read the update at home. The draft will be sent out by Jousten on Feb 28 to receive comments in the following week. He will inform the CCM chair that the report will be delayed by about 10 days.

11. APMP.M.P-K4 and K14: APMP.M.P-K4 (1 Pa…1000 Pa, protocol complete since 2002), report by Seung-Soo Hong. KRISS has taken over the pilot role from NPL/I. The participants have not been decided. They may be KRISS, NMJ, NIM, NPLI, etc. The transfer standards will be two CDGs (133 Pa and 333 Pa) joint in a set. This set is installed in a thermostatic chamber with a thermal stabilization of 0.01 K which is currently under study to be improved (this is important because the gauges’ heating is disconnected). Hendricks comments that 0.01 K stability may be a challenge, depending on the laboratory conditions. On the other hand, it is questionable that this stability is needed, when no resonance silicon gauge is used and the long-term instability of CDG contributes to the total uncertainty. It is expected to organise the comparison and start the measurements during this year.

K14, report by Kenta Arai: The range is from $10^{-4}$ Pa up to 1 Pa with SRGs as the transfer standards. The measurement conditions are the same as in the CCM.P-K4 comparison. Nine institutes participate. The measurements are in progress and finish in May 2014.

12. EURAMET.M.P-K4.2012, report by Dominik Prazak: The measurement range is from 1 Pa up to 15 kPa. The participating institutes are LNE, INRIM, PTB and MIKES. CMI is the pilot laboratory. The transfer standard is the FPG. The reference standards for the other laboratories were other FPGs, except PTB, which used a mercury barometer, a Furness pressure balance and a static expansion system. The results are in agreement in all cases. This comparison has been linked with CCM.P-K2 and CCM.P-K4. On the other hand, a supplementary comparison has been performed (EURAMET.M.P-S12) between CMI and INRIM for negative gauge pressures, where the obtained results are also in agreement.

This Draft B report is approved by the Working Group members.
13. Status of supplementary comparisons (EURAMET.M.P-S7, -S10 and SIM.M.P-S1): SIM.M.P-S1 measurements completed, EURAMET.M.P-S7 piloted by METAS (no report available), EURAMET.M.P-S7 published.

14. New developments in NMIs: For time reasons no presentations. These were partly given on the workshop on the day before.

15. Future developments and challenges for vacuum metrology and ongoing projects (EMRP IND12): This item was discussed under item 10 (report to the CCM chair). Jousten adds that the final workshop of the EMRP IND 12 presenting its results will be held on June 25-27, 2014 in Berlin. Further information is on the project website http://www.ptb.de/emrp/ind12-newsevents.html and soon on a related website.

16. Information on new ISO standards on vacuum metrology: The standards ISO 14291 (Definitions and specifications for quadrupole mass spectrometers) and for the new version of ISO 3529-3 (Vocabulary vacuum gauges) (FDIS) the voting has recently closed. For time reasons, it is not possible to discuss them, but Jousten asks the attendees to check them, because there are implications for the WG LP work. E.g., the term “cold cathode gauges” has been given up, the term “sensitivity” newly defined, the terms “spinning rotor gauge” and “ion energy analysing gauge” were introduced. Standard drafts are being developed for CDG and Pirani calibration. Technical specifications for the calibration of mass spectrometers and the determination of outgassing rate are also being developed.

17. New CCM and JCRB documents and forms, new guidelines: Jousten mentions that there are new guidelines by the CCM (see also item 2) for KCs. It is no more required to calculate pair-wise degrees of equivalence between all participants, if more than 2. Jousten recommends checking the pertinent documents on the BIPM website.

18. Various: Important issues concerning the WG LP and the WG HP will be discussed in the joint meeting to follow. The next meeting of the WG LP is planned for 2017 after the next CCM conference on pressure and vacuum metrology in 2017, organised by METAS.
Appendix 1

List of represented NMIs and their representatives in alphabetic order

CENAM, Mexico, represented by Jorge Torres; CEM, Spain, Nieves Medina; CMI, Czech Republic, Dominik Prazak; INMETRO, Brazil, Jackson da Silva Oliveira; INRIM, Italy, Mercede Bergoglio; KRISS, Korea, S. Hong, Sam-Yong Woo; LNE, France, Pierre Otal, Frédéric Boineau; NIM, China, Yuanchao Yang; NIST, USA, Douglas Olson, Jay Hendricks, James Fedchak; NMJJ, Japan, Kenta Arai, Hiroaki Kajikawa; NMISA, South Africa Brian Yalisi; PTB, Germany, Karl Jousten, Wladimir Sabuga; TÜBITAK (UME), Turkey, Rifat Kangi; VNIIM, Russia, Irina Sadkovskaya

Personal members present: Janez Setina, MIRS(IMT), Slovenia.

Excused: Christian Wüthrich, METAS

Members, but not present: A*STAR, INMS-NRC, NMIA, NPL/I, MSL-NZ.

Appendix 2

Workshop “Experiences on the stability of the accommodation coefficient of the spinning rotor gauge” on Feb 24, 2014

Summary

Several NMIs represented their experiences and investigations on the stability the accommodation coefficient ($\sigma_{\text{eff}}$) of the spinning rotor gauge. As a conclusion of the workshop it was decided that a recommended practice for use of SRG as transfer or reference standards (how to treat SRGs for best stability) should be developed by the WG LP. Jim Fedchak volunteered to draft this and take the lead to complete it.

Highlights

- The NIST publication by Chang and Abbott (JVST A 25 (2007), 1567-1576) is a thorough investigation on the stability of SRGs, but there are some weaknesses (e.g. no information on SRG controller given)
- The pre-calibration procedure at NIST seems to affect the stability of $\sigma_{\text{eff}}$ (see Chang and Abbott)
- Re-suspension is an important factor for a change of $\sigma_{\text{eff}}$ (about 0.1%) due to new alignment of rotational axis
- The stability of $\sigma_{\text{eff}}$ of customer gauges appears to be somewhat smaller at PTB (no pre-calibration procedure) compared to NIST (0.5% vs. 0.9%)
• SRG check standard at NIST 0.09%/a for 20 years
• Residual drag may abruptly change by 4E-8 s\(^{-1}\) for about 10% of SRGs (Fedchak)
• ±2° in angle of suspension head does not affect \(\sigma_{\text{eff}}\) (Fedchak)
• Spread of true diameter of rotor of 4.50 mm not more than 0.06 mm
• Commercial controller can handle 4 mm to 4.8 mm rotor diameter
• Bake-outs always increase \(\sigma_{\text{eff}}\) (NMIJ and PTB). Up to about 2.5%
• Rotor crashes always let \(\sigma_{\text{eff}}\) unchanged or decrease it
• Long-term stability during a comparison as always worse than in a pre-investigation in one lab
• The more non-isotropical rough the rotor the stronger the instability
• What appears to help for stability: No exposure to air, no handling of rotor outside, no rotor crash, no removal of suspension head (rarely possible), no rolling in thimble
• Research options: New rotor materials, coated rotors