16th CCM meeting, 18-19 May 2017, BIPM

Report from WGDV

Kenichi Fujii, Chair
Henning Wolf, Vice-Chair

- Unification of WGD and WGV in July 2014
- WGDV meeting held on 15 May 2017 at the BIPM
  - 36 participants from BEV, BIPM, CEM, CENAM, GUM, INMETRO, INRIM, IPQ, KRISS, LNE-CNAM, METAS, NIM, NIS, NIST, NMIA, NMIJ, NMISA, NPL, NRC, PTB, SMU, UME and VSL
- Position of WGDV in the CCM Strategy 2017-2027
- Key and supplementary comparisons
- Country report and topical issues
- CMC and service category
- Terms of Reference for WGDV
CCM Strategy 2017-2027

- **Section 7.1 Density and viscosity**

  - In general, completed and planned KCs cover almost all of the CMCs on density. Frequent KCs are not necessary. A period of **10 to 15 years** is considered to be adequate.

  - As the gas density measurements will be of importance for **energy savings and energy transportations**, such a CMC may be covered by a new KC on the $\rho \rho T$ properties of fluids.

  - As **food industry and agriculture** need a traceable standard of the refractive index of liquids for sugar content measurements, supplying the refractive index standard liquids, which are similar to the density standard liquids, will be necessary.

  - The current situation is one key comparison every **6 years**, alternating between **broad viscosity range at moderate temperatures** and **moderate viscosities in a broad temperature range**.
CIPM Key Comparisons on density (1)

**CCM.D-K1**  Density measurements of a silicon sphere by hydrostatic weighing (2001-2003)
- Status: Approved for equivalence (Final report available)
- Pilot: NMIJ (JP)
- Pilot group: METAS (CH), NRC (CA)
- Participants: NMIJ (JP), PTB (DE), INRIM (IT), KRISS (KR), METAS (CH), NRC (CA), CEM (ES), CENAM (MX)

**CCM.D-K2**  Comparison of liquid density standards (2004-2005)
- Status: Approved for equivalence (Final report available)
- Pilot: PTB (DE)
- Pilot group: NMIJ (JP), NRC (CA)
- Participants: BEV (AT), NRC (CA), PTB (DE), OMH (HU), NMIJ (JP), KRISS (KR), CENAM (MX), VNIIM (RU)

**CCM.D-K3**  Density measurements of stainless steel weights (2017-)
- Status: Questionnaire distributed, Answers received, Technical Protocol in progress, Participants identified
- Pilot: NMIJ (JP)

**CCM.D-K4**  Hydrometers (2011-2012)
- Status: Approved for equivalence (Final report available)
- Pilot: INRIM (IT)
- Pilot group: CENAM (MX), PTB (DE)
- Participants: INRIM (IT), CENAM (MX), PTB (DE), LATU (UY), NMIJ (JP), LNE (FR), NMIA (AU), NIST (US), KRISS (KR)
## CIPM Key Comparisons on density (2)

<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Status</strong></td>
<td>Questionnaire distributed and answers received</td>
</tr>
<tr>
<td><strong>Pilot</strong></td>
<td>BEV (AT)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>CCM.D-K6</th>
<th>Refractive index of liquids</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Status</strong></td>
<td>Planned</td>
</tr>
<tr>
<td><strong>Pilot</strong></td>
<td>NMIJ (JP)</td>
</tr>
<tr>
<td><strong>Comment</strong></td>
<td>This KC is being organized as a joint KC with CCPR as because CMCs and KCs on other optical properties are in CCPR. A few NMIs in CCPR are interested participating in this KC.</td>
</tr>
</tbody>
</table>
KCs on hydrometer calibrations

- CCM.D-K4 (pilot: INRIM)
  - Draft-B approved after the last WGDV meeting held in 2015
  - Final report approved in 2015

- Linking EURAMET.M.D-K4 (pilot: INRIM) and SIM.M.D-K4 (pilot: CENAM) to CCM.D-K4 undertaken by INRIM and CENAM
  - Drafts A and B approved in 2016
  - Final report approved in 2017

- Covering degrees of equivalence for 28 NMIs!
Equivalence of participants in CCM.D-K4

![Graph showing the equivalence of participants in CCM.D-K4](image)

- **CCM.D-K4 ranges:**
  - 600 kg/m³ - 610 kg/m³
  - 985 kg/m³ - 1000 kg/m³
  - 1490 kg/m³ - 1500 kg/m³
  - 1980 kg/m³ - 2000 kg/m³

- Participants:
  - INRiM
  - CENAM
  - PTB
  - KRISS
  - MKEH
  - NMIJ
  - NIST
  - GUM
  - LNE
  - NMIA
  - LATU

The graph illustrates the equivalence of participants across the specified ranges.
Degrees of equivalence of the participants with respect to the KCRVs of CCM.D-K4

Blue diamonds: participants in CCM.D-K4.
Green triangles: participants in SIM.M.D-K4
Braun squares: participants in SIM.M.D-S2

\[ \Delta n (\text{kg/m}^3) \]

-0.5
-0.4
-0.3
-0.2
-0.1
0
0.1
0.2
0.3
0.4

600 - 620 kg/m\(^3\)

INRIM
CENAM
PTB
KRISS
MKEH
NMIJ
NIST
GUM
LNE
NMIA
LATU
INRIM
PTB
KRISS
MKEH
GUM
UME
SMU
VNIM
IPQ
MIKES
BEV
LNE
CENAM
BSJ
CENAMEP
CESMEC
IBMETRO
INDECOPI
INEN
INMETRO
INTI
LACOMET
LATU
NIST
NRC
SIC

\[ \Delta n (\text{kg/m}^3) \]

985 - 1010 kg/m\(^3\)

INRIM
CENAM
PTB
KRISS
MKEH
NMIJ
NIST
GUM
LNE
NMIA
LATU
INRIM
PTB
KRISS
MKEH
GUM
UME
SMU
VNIM
IPQ
MIKES
BEV
LNE
CENAM
BSJ
CENAMEP
CESMEC
IBMETRO
INDECOPI
INEN
INMETRO
INTI
LACOMET
LATU
NIST
NRC
SIC
CC and RMO comparisons on hydrometer succeeded in covering CMCs of 28 NMI

<table>
<thead>
<tr>
<th>NMI</th>
<th>CCM.D-K4</th>
<th>EURAMET.M.D-K4</th>
<th>SIM.M.D-K4</th>
<th>SIM.M.D-S2</th>
</tr>
</thead>
<tbody>
<tr>
<td>INRiM - Italy</td>
<td>X</td>
<td>X</td>
<td></td>
<td></td>
</tr>
<tr>
<td>MKEH (ex OMH) - Hungary</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>PTB - Germany</td>
<td></td>
<td>X</td>
<td></td>
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<tr>
<td>LNE France</td>
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<td>X</td>
<td></td>
</tr>
<tr>
<td>IPQ - Portugal</td>
<td></td>
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<td></td>
<td></td>
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<tr>
<td>VTT - MIKES - Finland</td>
<td>X</td>
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<td></td>
<td></td>
</tr>
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<td>BEV – Austria</td>
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<td>UME - Turkey</td>
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<td>SMU - Slovakia</td>
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<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>VNIIM - Russia</td>
<td></td>
<td></td>
<td></td>
<td>X</td>
</tr>
<tr>
<td>CENAM - Mexico</td>
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<td>X</td>
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<td>BSJ - Jamaica</td>
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<td>INDECOPI - Peru</td>
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<td>X</td>
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<tr>
<td>INEN - Ecuador</td>
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<td>X</td>
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<tr>
<td>INTI - Argentina</td>
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<td>X</td>
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<tr>
<td>LACOMET - Costa Rica</td>
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<td>X</td>
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<tr>
<td>LATU - Uruguay</td>
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<td>X</td>
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<tr>
<td>NIST - United States of America</td>
<td>X</td>
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<td></td>
</tr>
<tr>
<td>NRC - Canada</td>
<td></td>
<td></td>
<td>X</td>
<td></td>
</tr>
<tr>
<td>SIC - Colombia</td>
<td></td>
<td></td>
<td></td>
<td>X</td>
</tr>
<tr>
<td>KRISS – Korea (the Republic of)</td>
<td>X</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>NMIJ - Japan</td>
<td>X</td>
<td></td>
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</tr>
</tbody>
</table>
CCM.D-K3 (pilot: NMIJ): Density measurement of stainless steel weights

- Approved at the 14th CCM in 2011
- Measurement method: hydrostatic weighing
- Distribution of questionnaire in 2011
- Discussion on the answers at the CCM WGDV in 2015

\[
\rho_{\text{sample}} = \frac{m_{\text{sample}}}{V_{\text{sample}}}
\]

OIML type stainless steel weights

Hydrostatic weighting apparatus at NMIJ
Technical protocol

- Transfer standards
  - OIML type stainless steel weight
  - 1 kg, 200 g and 20 g
  - Two petals

- Transportation
  - Courier service (not hand-carry)

- Measurement method
  - Hydrostatic weighing with respect to a solid density standard
  - Hydrostatic weighing with respect to the density of water

- Cleaning
  - The participants use their own methods
Circulation of travelling standard

- **Petal 1:** NMIJ → EURAMET (including Link Lab) → BIPM → AFRIMETS → Link lab from SIM → NMIJ
- **Petal 2:** NMIJ → SIM (including Link Lab) → APMP → Link lab from EURAMET → NMIJ
- **Link labs:** NMIJ, 1 from EURAMET, 1 from SIM
Participants

- BIPM
- APMP: 4  NIM (China), NIMT (Thailand), NMIJ (Japan), A*STAR (Singapore)
- EURAMET: 5  BEV (Spain), INRIM (Italy), METAS (Switzerland), PTB (Germany), UME (Turkey)
- AFRIMETS: 2  NMISA (South Africa), NIS (Egypt)
- SIM: 3  CENAM (Mexico), INMETRO (Brazil), NRC (Canada)

- Coordinating group to help the pilot in drawing the technical protocol
  - 1 NMI from EURAMET (BEV), 1 NMI from SIM (CENAM)
Planned key comparison on density

➢ Oscillation-type density meter

Concept of this KC

• Pilot institute measures the density of a few litters of liquids.
• The liquids in small bottles are distributed to participants so that their densities can be measured only by oscillation-type density meters.

CCM.D-K5: piloted by BEV (AT)
Questionnaire distributed and answers received in 2016
Participants: 12 NMIs
Sample: 4 liquid samples will be distributed to the participants.
Planned key comparison on density

- Refractive index of liquid

  Concept of this KC
  - Density standard liquids are simultaneously used as refractive index standard liquids (CRMs).
  - High-demands from food industry and agriculture.
  - Pilot institute distribute liquid samples to participants.
  - Participants measure the refractive index of the liquid by their own refractometers.

CCM.D-K6: piloted by NMIJ (JP)
Joint KC with CCPR

Minimum deviation angle method

Interferometric method
Refractive Index Measurement
Presented by Olivier Pellegrino (IPQ)

Certified Reference Materials (CRM) to other NMIs
\[ U (k=2.00) = 0.000 \, 02 \] (aqueous solutions)

<table>
<thead>
<tr>
<th>PTB</th>
<th>GUM</th>
</tr>
</thead>
<tbody>
<tr>
<td>n-Heptan</td>
<td>Methyl silicone</td>
</tr>
<tr>
<td>1,387 77</td>
<td>1,400 00</td>
</tr>
<tr>
<td>Iso-Octan</td>
<td>Glycerol</td>
</tr>
<tr>
<td>1,391 49</td>
<td>1,450 00</td>
</tr>
<tr>
<td>Cyclohexan</td>
<td>Silicone oil CR 500</td>
</tr>
<tr>
<td>1,426 30</td>
<td>1,470 00</td>
</tr>
<tr>
<td>Tetrachlorethylen</td>
<td>Silicone oil NA 140</td>
</tr>
<tr>
<td>1,50 580</td>
<td>1,560 00</td>
</tr>
</tbody>
</table>

Mettler Toledo RE 50
Anton Paar Abbemat 550

SRM 1922 - Liquid Refractive Index - Mineral Oil
\[ 1,46945 \, \pm \, 6 \times 10^{-5} \]
<table>
<thead>
<tr>
<th>CCM.V-K1</th>
<th>Five samples of Newtonian liquids: wide viscosity range (2002)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Status</td>
<td>Approved for equivalence (Final report available)</td>
</tr>
<tr>
<td>Pilot</td>
<td>PTB (DE)</td>
</tr>
<tr>
<td>Pilot group</td>
<td>NMi VSL (NL), IPQ (PT), Cannon (US)</td>
</tr>
<tr>
<td>Participants</td>
<td>BNM-LNE (FR), Cannon (US), GUM (PL), CNR-IMG (IT), NMIJ (JP), NMI VSL (NL), NRCCRM (CN), PTB (DE), SMU (SK), UME (TR), VNIIM (RU), BEV (AT), CENAM (MX), INM (CN), PTB (DE), NIS (EG), NPLI (IN), SIRIM (MY)</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>CCM.V-K2</th>
<th>Six samples of Newtonian liquids: wide temperature range (2006)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Status</td>
<td>Approved for equivalence (Final report available)</td>
</tr>
<tr>
<td>Pilot</td>
<td>Cannon (US)</td>
</tr>
<tr>
<td>Pilot group</td>
<td>PTB (DE)</td>
</tr>
<tr>
<td>Participants</td>
<td>INRIM (IT), IPQ (PT), LNE (FR), NIS (EG), NMI VSL (NL), NMIJ (JP), NIM (CN), PTB (DE), VNIIM (RU), INMETRO (BR), SMU (SK), INM (RO), BEV (AT), Cannon (US)</td>
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<table>
<thead>
<tr>
<th>CCM.V-K3</th>
<th>Three samples of Newtonian liquids: wide viscosity range (2012-2013)</th>
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</thead>
<tbody>
<tr>
<td>Status</td>
<td>Report in progress, Draft B</td>
</tr>
<tr>
<td>Pilot</td>
<td>NMIJ (JP)</td>
</tr>
<tr>
<td>Pilot Group</td>
<td>PTB (DE)</td>
</tr>
<tr>
<td>Participants</td>
<td>Cannon (US), CENAM (MX), GUM (PL), INMETRO (BR), INRIM (IT), LNE (FR), NIM (CN), NMIJ (JP), PTB (DE), SMU (SK), UME (TK), NMI VSL (NL), BEV (AT), IPQ (PT), KEB (KE), NIS (EG), NMISA (ZA), NPLI (IN), SIRIM (MY)</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>CCM.V-K4</th>
<th>Two samples of Newtonian liquids: wide temperature range (2018-)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Status</td>
<td>Questionnaire distributed and answers received</td>
</tr>
<tr>
<td>Pilot</td>
<td>CENAM (MX)</td>
</tr>
</tbody>
</table>
# CCM.V-K3: comparison in a wide viscosity range

- **12 participants with independent scale**
  NIST/CANNON (USA), CENAM (Mexico), GUM(Poland), INMETRO(Brazil), INRIM (Italy), LNE (France), NIM (China), NMIJ/AIST (Japan), PTB (Germany), SMU (Slovakia), UME (Turkey), VSL(The Netherlands)

- **7 participants with scale calibrated by other NMIs**
  BEV (Austria), IPQ (Portugal), KEBS (Kenya), NIS (Egypt), NMISA (South Africa), NPLI(India), SIRIM (Malaysia)

- **Viscometer type used**
  VSL and NMIJ used Ostwald (U tube) type viscometers
  Other NMIs used Ubbelohde type

<table>
<thead>
<tr>
<th>Liquid sample</th>
<th>Temperature / °C</th>
<th>Nominal kinematic viscosity / mm²s⁻¹</th>
<th>Temperature coefficient of viscosity / K⁻¹</th>
<th>Density / gcm⁻³</th>
<th>Standard uncertainty / gcm⁻³</th>
<th>Surface tension / mNm⁻¹</th>
<th>Standard uncertainty / mNm⁻¹</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard liquid A</td>
<td>15</td>
<td>6</td>
<td>0.028</td>
<td>0.81243</td>
<td>0.00012</td>
<td>28.50</td>
<td>0.19</td>
</tr>
<tr>
<td></td>
<td>20</td>
<td>5</td>
<td>0.027</td>
<td>0.80900</td>
<td>0.00012</td>
<td>28.07</td>
<td>0.18</td>
</tr>
<tr>
<td>Standard liquid B</td>
<td>20</td>
<td>2000</td>
<td>0.082</td>
<td>0.88127</td>
<td>0.00013</td>
<td>32.83</td>
<td>0.18</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>500</td>
<td>0.063</td>
<td>0.86920</td>
<td>0.00018</td>
<td>31.04</td>
<td>0.22</td>
</tr>
<tr>
<td>Standard liquid C</td>
<td>20</td>
<td>160000</td>
<td>0.101</td>
<td>0.89632</td>
<td>0.00018</td>
<td>32.45</td>
<td>0.48</td>
</tr>
<tr>
<td></td>
<td>40</td>
<td>25000</td>
<td>0.083</td>
<td>0.88514</td>
<td>0.00019</td>
<td>31.40</td>
<td>0.36</td>
</tr>
</tbody>
</table>
Degrees of equivalence of each laboratory with respect to the reference value: Liquid A at 15 °C

Left-hand blue circle: laboratories maintaining an independent scale

Distance between two red lines: the expanded uncertainty of the reference value
Summary on results of CCM.V-K3

- Second Draft A was already circulated
- Draft B report will be circulated after this meeting

<table>
<thead>
<tr>
<th>Liquid samples</th>
<th>Temperature / °C</th>
<th>Reference value $x_{\text{ref}}$ / mm²s⁻¹</th>
<th>Expanded uncertainty $U_{g6}(x_{\text{ref}})$ / mm²s⁻¹</th>
<th>Relative expanded uncertainty $U_{r66}(x_{\text{ref}})$ / %</th>
<th>Procedure</th>
</tr>
</thead>
<tbody>
<tr>
<td>Standard liquid A</td>
<td>15</td>
<td>5.5833</td>
<td>0.0014</td>
<td>0.03</td>
<td>A</td>
</tr>
<tr>
<td>Standard liquid A</td>
<td>20</td>
<td>4.8737</td>
<td>0.0012</td>
<td>0.02</td>
<td>A</td>
</tr>
<tr>
<td>Standard liquid B</td>
<td>20</td>
<td>1972.4</td>
<td>0.95</td>
<td>0.05</td>
<td>A</td>
</tr>
<tr>
<td>Standard liquid B</td>
<td>40</td>
<td>472.62</td>
<td>0.29 0.31</td>
<td>0.06 0.07</td>
<td>B</td>
</tr>
<tr>
<td>Standard liquid C</td>
<td>20</td>
<td>154639</td>
<td>284 278</td>
<td>0.18 0.18</td>
<td>B</td>
</tr>
<tr>
<td>Standard liquid C</td>
<td>40</td>
<td>25050</td>
<td>31 31</td>
<td>0.12 0.12</td>
<td>B</td>
</tr>
</tbody>
</table>
Planned key comparison on viscosity

- Viscosity measurements in a wide temperature range (10 °C to 100 °C)

Concept of this KC

- Pilot institute distributes viscosity standard liquids to participants.
- Participants measure the viscosity of the liquids by their own capillary viscometers.

CCM.V-K4: piloted by CENAM (MX)

Questionnaire distributed and answers received in 2017

Sample: 2 liquids will be distributed.

10 NMIs and 1 non-signatory institute expressed their interest.
Link between CC and RMO KCs

Density

- CCM.D-K3 (Density of stainless steel weight) (2017) Planned
  SIM.M.D-K3 (2009-2010) Approved for equivalence
  SIM.M.D-S3 (2006) (volume of glass and stainless steel) Approved for equivalence
  SIM.M.M-S11 (2012-2013) (Mass and volume of weight) In progress
  SIM.M.D-S1 (2007) Approved and published
  SIM.M.D-S2 (2009-2010) Approved and published
  SIM.M.D-S4 (2009-2010) Protocol approved

Viscosity

  COOMET.M.V-K1 (2005-2006) Approved for equivalence
  COOMET.M.V-S1 (2013) Approved for equivalence
- CCM.V-K2 (wide temperature range) Approved for equivalence
- CCM.V-K2.1 (2008) (comparison to link Egypt and South Africa) Approved for equivalence
- CCM.V-K4 (2017-) (wide temperature range) Questionnaire distributed and answers received
EMRP ENG 59: Non-Newtonian Liquids
Presented by Patrick Ballereau of CNAM

Objectives
• Develop rheology measurement standard and reference materials
• Determine physical properties of non-Newtonian liquids
• Develop inline sensors including on-site calibration methods

Impact
• Economical: Increased recovery of European oil/gas fields
• Operational efficiency: sensor & model reliability, comparability
• Health & safety: less people in hostile environment
• Standardisation: ISO/NORSOK, API
Viscosity Measurements by Light Scattering
Presented by Jürgen Rauch (PTB)

Light Scattering by Surface Waves: Surface Light Scattering (SLS)

\[ \zeta(\mathbf{r}, t) = \sum \zeta_q(t) e^{i q \mathbf{r}} \]

Laser-induced Capillary Wave Technique

• Surface light scattering: spontaneous capillary waves

• Laser-induced grating

• Length scale: (10 – 100) μm

• Viscosity range: (10^{-1} – 10^{6}) mPa·s

• \[ I(t) \propto \Delta u(t)^2 = f(\eta, \sigma, \partial \sigma / \partial T, \rho, \lambda, a, \alpha, \mu) \]

CCM Service Category for Density

Previous categories (-2015)

2.1 Density of solid
   2.1.1 Density of solid: solid density artefact
   2.1.2 Volume of solid: solid artefact

2.2 Density of liquid
   2.2.1 Density of liquid: density measuring device,
   standard volume vessel

New categories (2016-)

2.1 Density of solid
   2.1.1 Density of solid
   2.1.2 Volume of solid

2.2 Density of liquid
   2.2.1 Density measuring device
   2.2.2 Density of liquid

2.3 Refractive index of liquid (new)
   2.3.1 Refractive index of liquid (new)

Fluid Flow
9.5 Volume of liquid
   9.5.1 Volume of liquid
   Example: CCM.FF-4.2.2011
   Calibration of micropipettes

Instrument Type or Method in CMC table

- hydrometer, oscillation-type density meter, pycnometer, etc.
- hydrostatic weighing, hydrometer, oscillation-type density meter, magnetic suspension density meter, pycnometer, etc.
Terms of reference for WGDV

- To improve techniques for realizing the SI units of density and viscosity;
- To review and make recommendations for fulfilling the traceability in density and viscosity;
- To identify and support future needs for key and supplementary comparisons in the field of density and viscosity;
- To perform CIPM key comparisons on density and viscosity;
- To establish and maintain CMC service categories lists, provide guidance to accept CMCs on density and viscosity and coordinate and conduct the CMC review process; and
- To coordinate research activities on metrology for density and viscosity.
Summary

- WGDV meeting was held on 15 May 2017 at the BIPM.
- A total of 36 participants.
- Final report of CCM.D-K4 (hydrometer) was presented, and relevant RMO KCs (EURAMET and SIM) were linked successfully: covering degrees of equivalence for 28 NMIs.
- Participants of CCM.D-K3 (density of ss weight) determined
- Result of CCM.V-K3 (wide temperature range) discussed
- Planned KCs:
  - CCM.D-K5: Liquid density measurement by oscillation-type density meter
  - CCM.D-K6: Refractive index of liquid
  - CCM.V-K4: Measurement of viscosity standard liquids in a wide temperature range
- Research for non-Newtonian liquids and optical (non-contact) method for viscosity measurements were discussed.