

*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  $p\rho T$  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)

<b>CCM.D-K1</b>	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)
<b>CCM.D-K2</b>	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)
<b>CCM.D-K3</b>	Density measurements of stainless steel weights (2017-)
Status	Questionnaire distributed, Answers received, Technical Protocol in progress, Participants identified
Pilot	NMIJ (JP)
<b>CCM.D-K4</b>	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)

**CCM.D-K5** Density measurements by oscillation-type density meters (2001-2003)

Status **Questionnaire distributed and answers received**

Pilot BEV (AT)

**CCM.D-K6** Refractive index of liquids

Status **Planned**

Pilot NMIJ (JP)

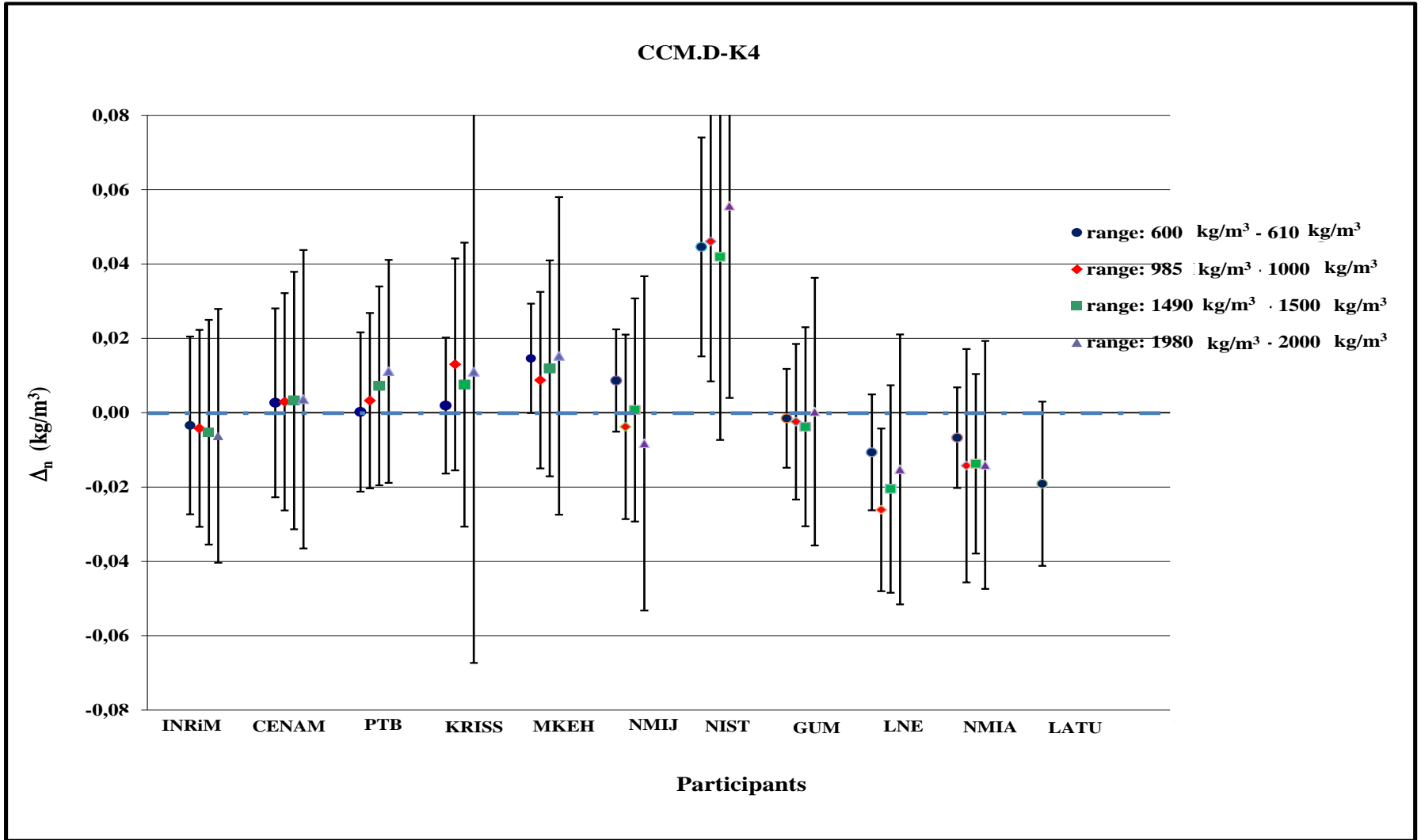
Comment **This KC is being organized as a joint KC with CCPR as because CMCs and KCs on other optical properties are in CCPR. A few NMIs in CCPR are interested participating in this KC.**

# 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 !**

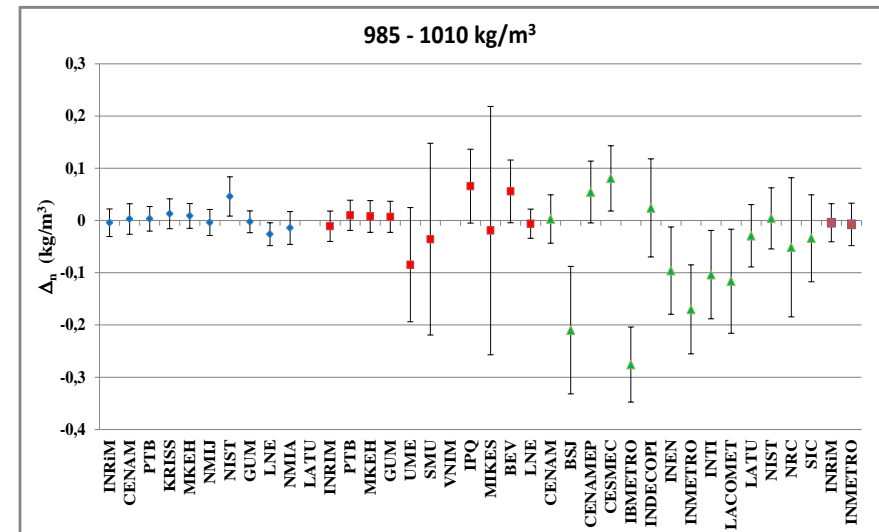
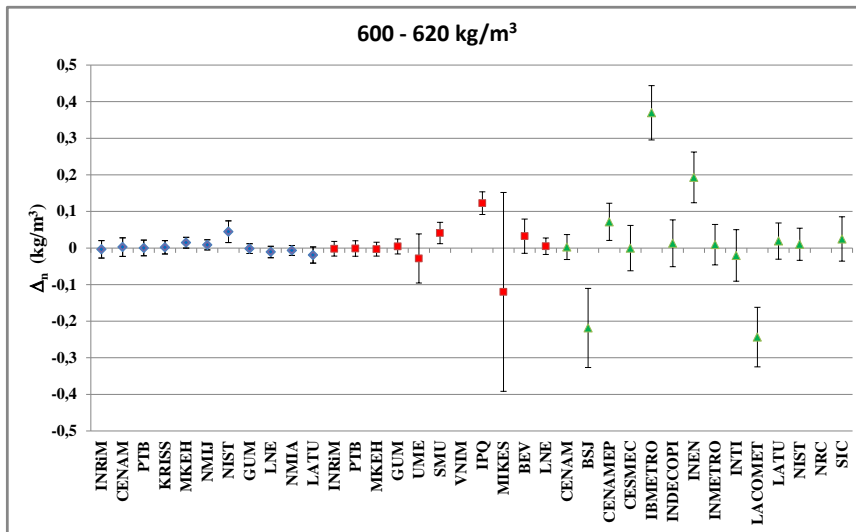


# Equivalence of participants in CCM.D-K4



# Degrees of equivalence of the participants with respect to the KCRVs of CCM.D-K4

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



# CC and RMO comparisons on hydrometer succeeded in covering CMCs of 28

## NMIs

NMI	CCM.D-K4	EURAMET.M.D-K4	SIM.M.D-K4	SIM.M.D-S2
INRiM - Italy	X	X		X
MKEH (ex OMH) - Hungary	X	X		
PTB - Germany	X	X		
LNE France	X	X		
IPQ - Portugal	X	X		
VTT - MIKES - Finland		X		
BEV – Austria		X		
UME - Turkey		X		
GUM - Poland	X	X		
SMU - Slovakia		X		
VNIIM - Russia		X		
CENAM - Mexico	X		X	
BSJ - Jamaica			X	
CENAMEP - Panama			X	
CESMEC - Chile			X	
IBMETRO - Bolivia			X	
INDECOPI - Peru			X	
INEN - Ecuador			X	
INMETRO - Brazil			X	X
INTI - Argentina			X	
LACOMET - Costa Rica			X	
LATU - Uruguay	X		X	
NIST - United States of America	X		X	
NRC - Canada			X	
SIC - Colombia			X	
KRISS – Korea (the Republic of)	X			
NMIJ - Japan	X			

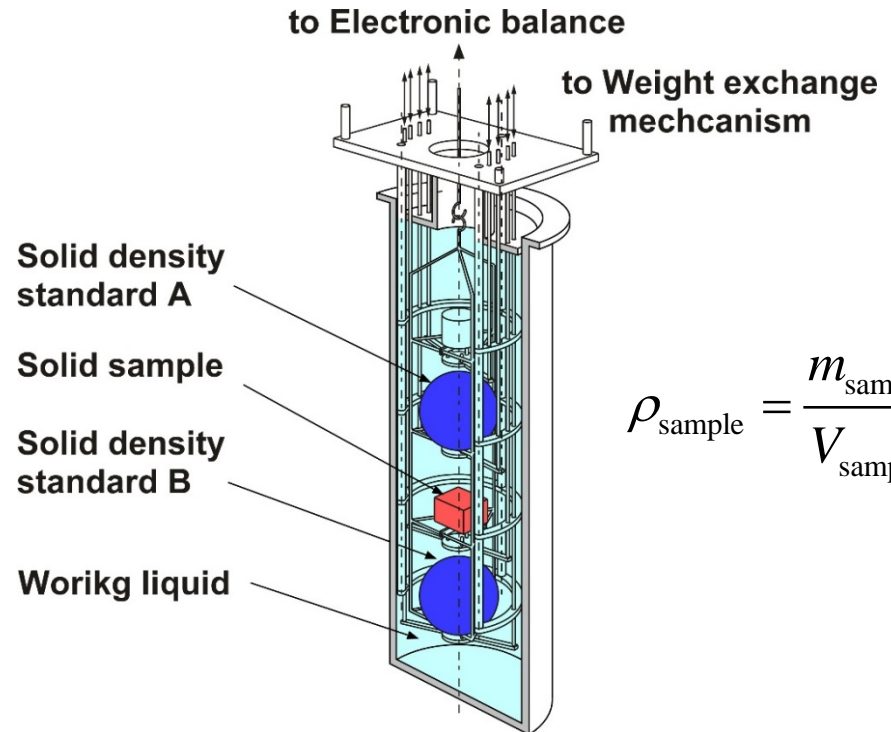


# CCM.D-K3 (pilot: NMIJ): Density measurement of stainless steel weights

- 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



OIML type stainless steel weights



$$\rho_{\text{sample}} = \frac{m_{\text{sample}}}{V_{\text{sample}}}$$

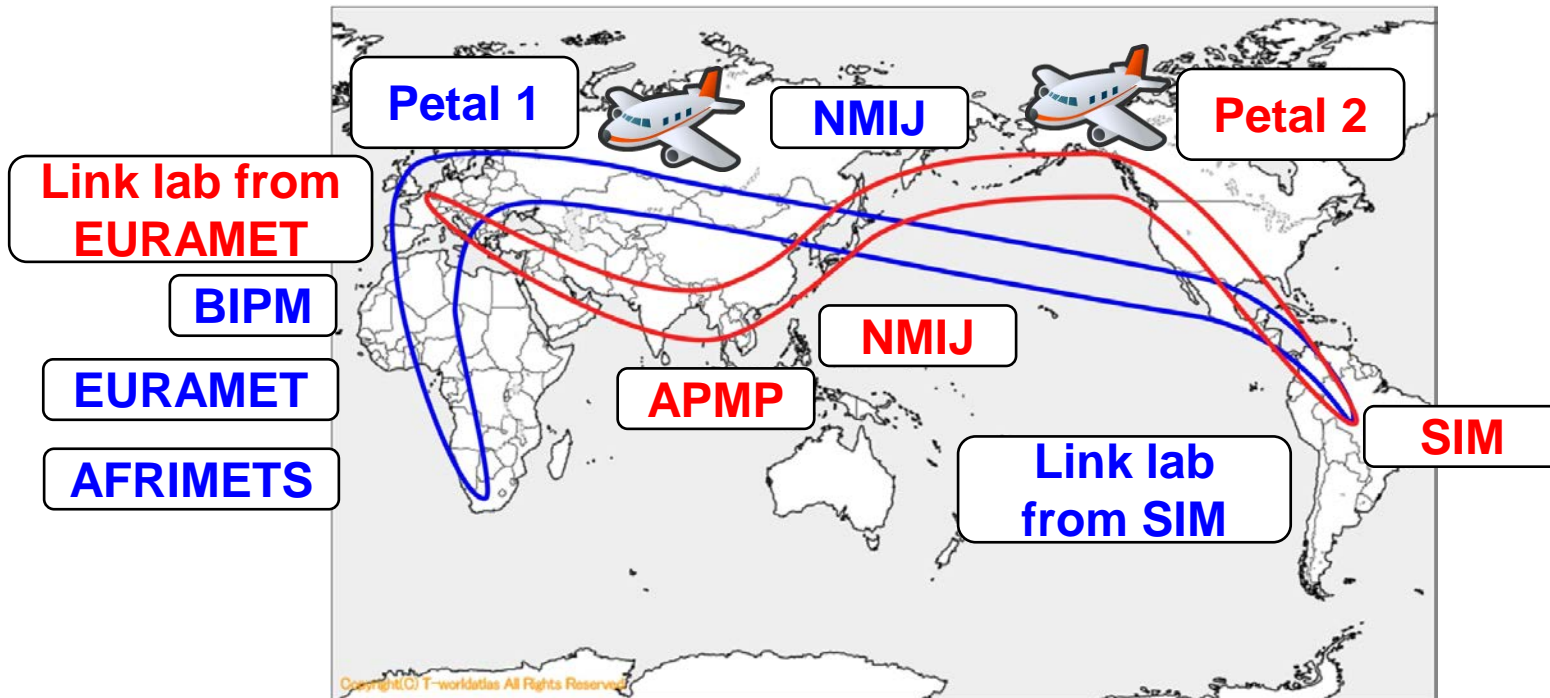
Hydrostatic weighing apparatus at NMIJ

# 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

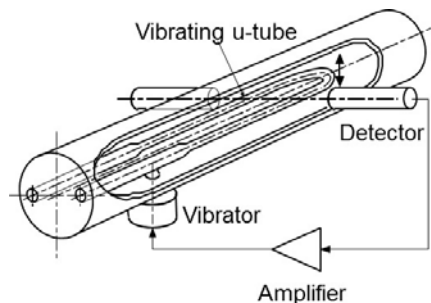
- Pilot institute measures the density of a few liters of liquids.
- The liquids in small bottles are distributed to participants so that their densities can be measured only by oscillation-type density meters.

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

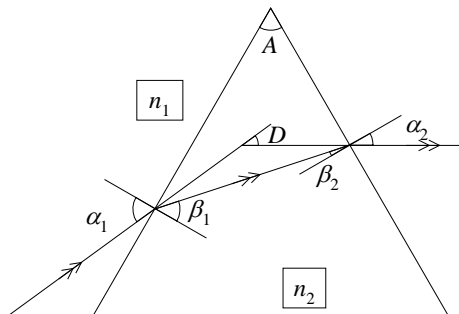
## ➤ Refractive index of liquid

### Concept of this KC

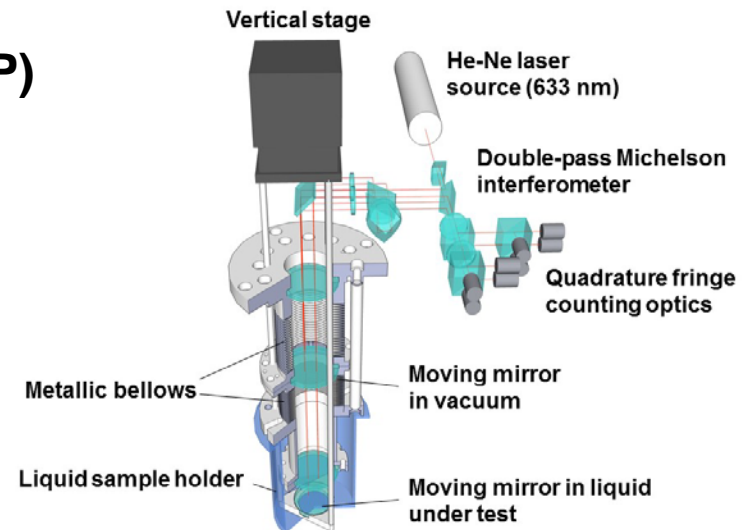
- Density standard liquids are simultaneously used as refractive index standard liquids (CRMs).
- High-demands from food industry and agriculture.
- Pilot institute distribute liquid samples to participants.
- Participants measure the refractive index of the liquid by their own refractometers.

**CCM.D-K6: piloted by NMIJ (JP)**

**Joint KC with CCPR**



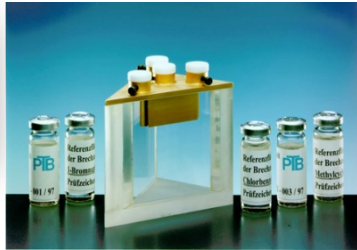
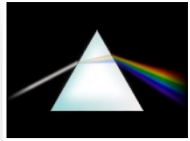
**Minimum deviation angle method**



**Interferometric method**

# Refractive Index Measurement

## Presented by Olivier Pellegrino (IPQ)



Mettler Toledo RE 50



Anton Paar Abbemat 550

**Certified Reference Materials (CRM) to other NMIs**  
 $U (k=2.00) = 0.000\ 02$  (aqueous solutions)



**PTB**

n-Heptan	1,387 77
Iso-Octan	1,391 49
Cyclohexan	1,426 30
Tetrachlorethylen	1,50 580



CENTRAL OFFICE OF MEASURES

**GUM**

Methyl silicone	1,400 00
Glycerol	1,450 00
Silicone oil CR 500	1,470 00
Silicone oil NA 140	1,560 00

**NIST** National Institute of Standards and Technology  
 U.S. Department of Commerce

SRM 1922 - Liquid Refractive Index - Mineral Oil

$1,469\ 45 \pm 6 \times 10^{-5}$



# CIPM Key Comparisons on Viscosity

<b>CCM.V-K1</b>	Five samples of Newtonian liquids: wide viscosity range (2002)
Status	Approved for equivalence (Final report available)
Pilot	PTB (DE)
Pilot group	NMi VSL (NL), IPQ (PT), Cannon (US)
Participants	BNM-LNE (FR), Cannon (US), GUM (PL), CNR-IMGC (IT), NMIJ (JP), NMi VSL (NL), NRCCRM (CN), PTB (DE), SMU (SK), UME (TR), VNIIM (RU), BEV (AT), CENAM (MX), INM (RO), IPQ (PT), NIS (EG), NPLI (IN), SIRIM (MY)
<b>CCM.V-K2</b>	Six samples of Newtonian liquids: wide temperature range (2006)
Status	Approved for equivalence (Final report available)
Pilot	Cannon (US)
Pilot group	PTB (DE)
Participants	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)
<b>CCM.V-K3</b>	Three samples of Newtonian liquids: wide viscosity range (2012-2013)
Status	Report in progress, Draft B
Pilot	NMIJ (JP)
Pilot Group	PTB (DE)
Participants	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), KEBS (KE), NIS (EG), NMISA (ZA), NPLI (IN), SIRIM (MY)
<b>CCM.V-K4</b>	Two samples of Newtonian liquids: wide temperature range (2018-)
Status	Questionnaire distributed and answers received
Pilot	CENAM (MX)



# 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)

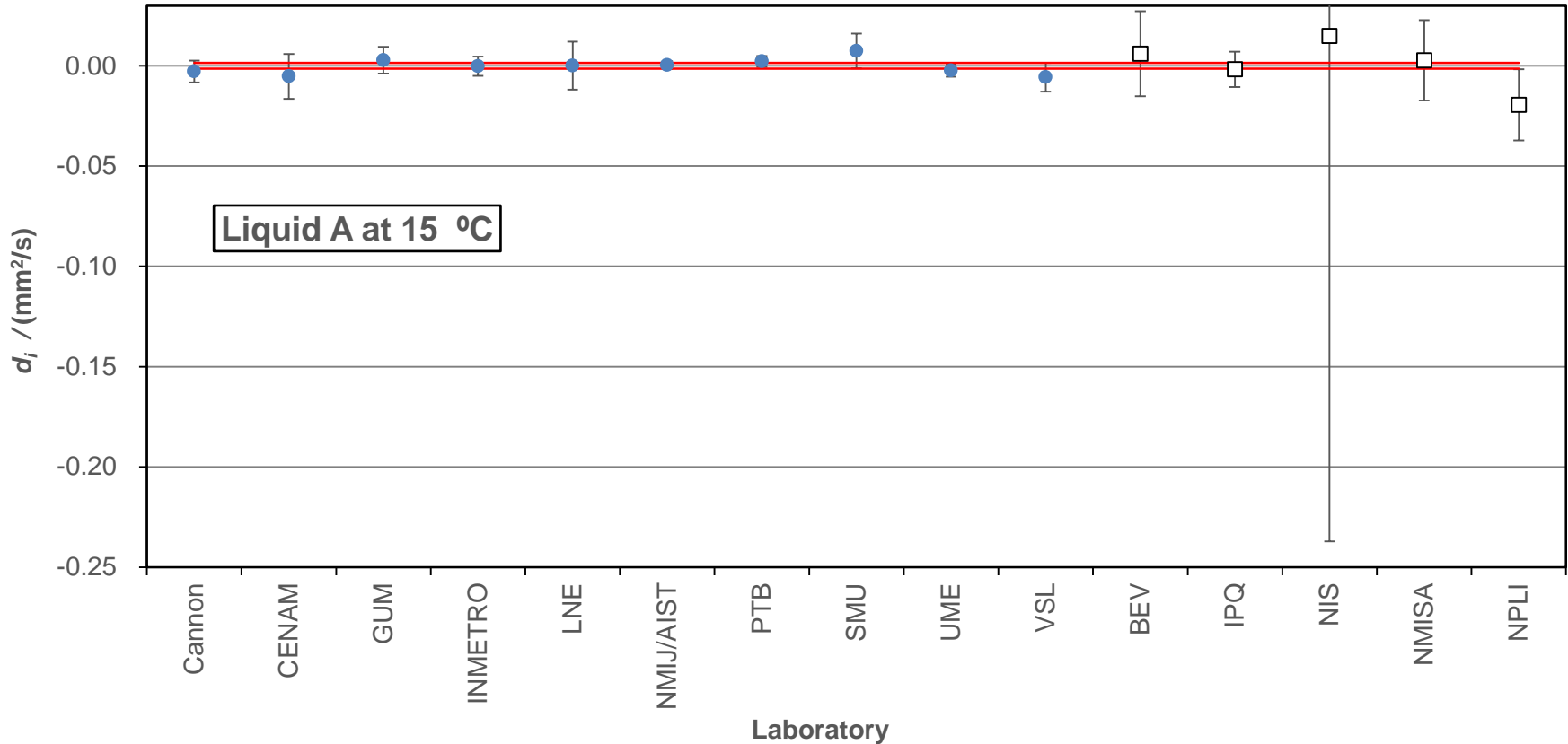
## ➤ Viscometer type used

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

Liquid sample	Temperature / °C	Nominal kinematic viscosity / mm <sup>2</sup> s <sup>-1</sup>	Temperature coefficient of viscosity / K <sup>-1</sup>	Density / gcm <sup>-3</sup>	Standard uncertainty / gcm <sup>-3</sup>	Surface tension / mNm <sup>-1</sup>	Standard uncertainty / mNm <sup>-1</sup>
Standard liquid A	15	6	0.028	0.81243	0.00012	28.50	0.19
	20	5	0.027	0.80900	0.00012	28.07	0.18
Standard liquid B	20	2000	0.082	0.88127	0.00013	32.83	0.18
	40	500	0.063	0.86920	0.00018	31.04	0.22
Standard liquid C	20	160000	0.101	0.89632	0.00018	32.45	0.48
	40	25000	0.083	0.88514	0.00019	31.40	0.36



# 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

Liquid samples	Temperature / ° C	Reference value $x_{ref}$ / mm <sup>2</sup> s <sup>-1</sup>	Expanded uncertainty $U_{95}(x_{ref})$ / mm <sup>2</sup> s <sup>-1</sup>	Relative expanded uncertainty $U_{r95}(x_{ref})$ / %	Procedure
Standard liquid A	15	5.5833	0.0014	0.03	A
Standard liquid A	20	4.8737	0.0012	0.02	A
Standard liquid B	20	1972.4	0.95	0.05	A
Standard liquid B	40	472.62	0.29 0.31	0.06 0.07	B
Standard liquid C	20	154639	284 278	0.18 0.18	B
Standard liquid C	40	25050	31 31	0.12 0.12	B

# Planned key comparison on viscosity

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

## Concept of this KC

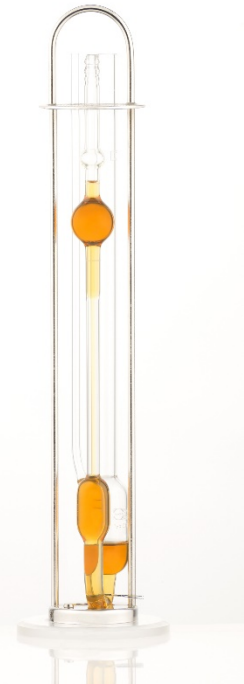
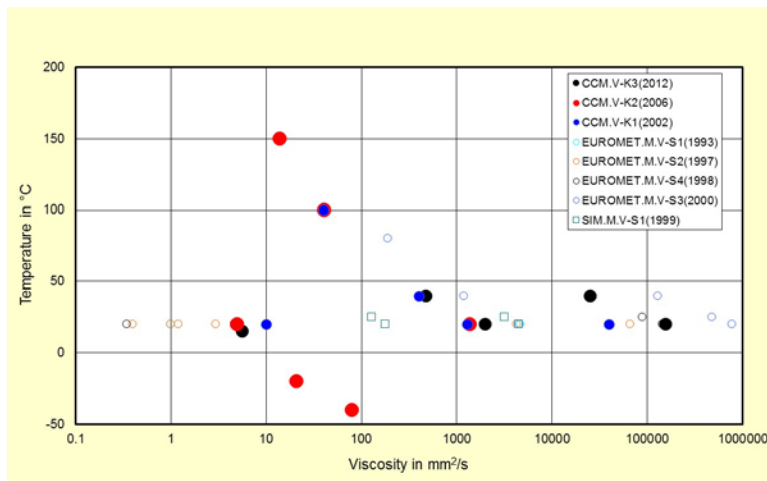
- Pilot institute distributes viscosity standard liquids to participants.
- Participants measure the viscosity of the liquids by their own capillary viscometers.

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

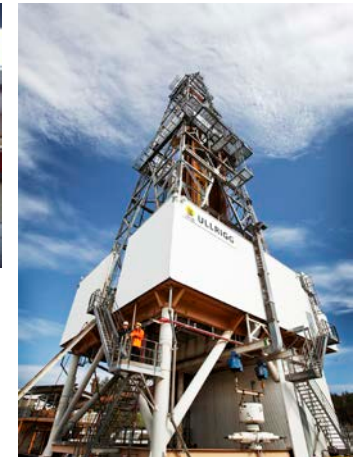
- CCM.D-K1 (Density of silicon sphere) (2001-2003) Approved for equivalence  
EURAMET.M.D-K1.1 (2008-2010) Report in progress, Draft A2
- CCM.D-K2 (Density of liquid) (2004) Approved for equivalence  
EURAMET.M.D-K2 (2008-2009) Approved for equivalence
- CCM.D-K3 (Density of stainless steel weight) (2017) 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
- CCM.D-K4 (Hydrometer) (2011-2012) Approved for equivalence  
APMP.M.D-K4 (2007-2008) Report in progress, Draft A  
**EUROMET.M.D-K4 (2003-2005) Approved for equivalence**  
**SIM.M.D-K4 (2007-2008) Approved for equivalence**  
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

- CCM.V-K1 (wide viscosity range) (2002) Approved for equivalence  
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-K3 (2012-2013) (wide viscosity range) Report in progress, Draft B
- CCM.V-K4 (2017-) (wide temperature range) Questionnaire distributed and answers received

# EMRP ENG 59: Non-Newtonian Liquids

Presented by Patrick Ballereau of CNAM



**From conventional drilling fluid engineer and sensors**

**to future drilling fluid engineering and sensors**

## Objectives

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

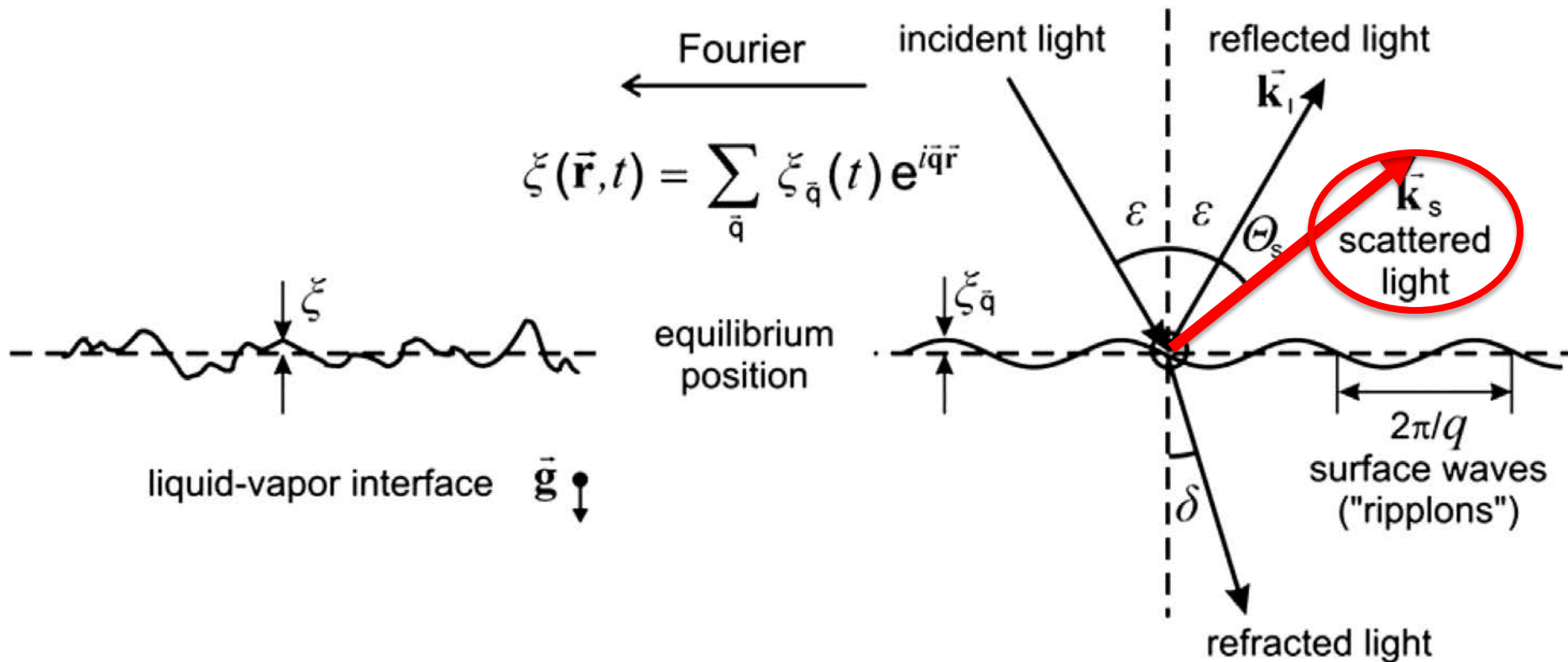
## Impact

- **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)

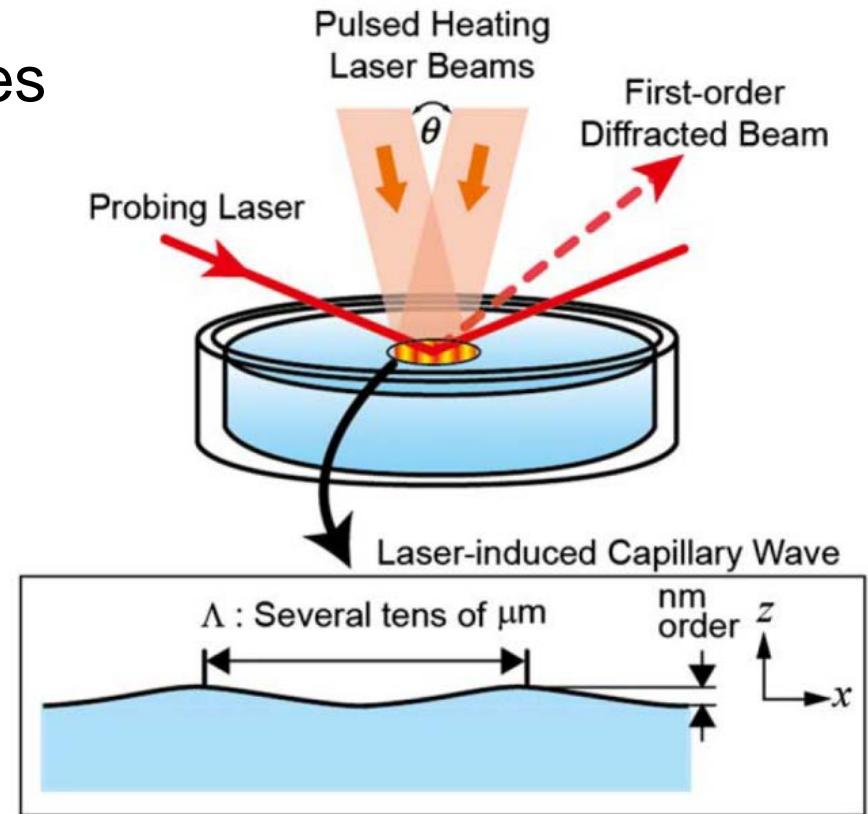


A. P. Fröba and A. Leipertz, Accurate Determination of Liquid Viscosity and Surface Tension Using Surface Light Scattering (SLS): Toluene under Saturation Conditions between 260 K and 380 K, *Int. J. Thermophys.*, 2003, 24, 898



# Laser-induced Capillary Wave Technique

- Surface light scattering: spontaneous capillary waves
- Laser-induced grating
- Length scale: (10 – 100)  $\mu\text{m}$
- Viscosity range: (10<sup>-1</sup> – 10<sup>6</sup>) mPa·s



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

Y. Nagasaka, Experimental Thermodynamics Vol. IX – Advances in Transport Properties of Fluids, 2014; Ed. Assael, Goodwin, Vesovic and Wakeham



# 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**

**Fluid Flow**

**9.5 Volume of liquid**

**9.5.1 Volume of liquid**

Example: CCM.FF-4.2.2011

Calibration of micropipettes

## 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)**

**Instrument Type or Method in CMC table**

hydrometer, oscillation-type density meter, pycnometer, etc.

**Instrument Type or Method in CMC table**

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.**