15th CCM meeting, 26-27 February 2015, BIPM

Report from WGDV

Kenichi Fujii, Chair
Henning Wolf, Vice-Chair

- **Unification of WGD and WGV in July 2014, approved by CIPM**
- **WGDV meeting held on 24 Feb 2014 at the BIPM**

  28 members: A*STAR, BEV, BIPM, Cannon, CEM, CENAM, GUM, INMETRO, INRIM, IPQ, KRISS, LNE-CNAM, METAS, NIM, NIS, NIST, NMIA, NMIJ, NMISA, NPL, NPLI, NRC, PTB, SMU, SP, UME, VNIIM, VSL

  1 guest: KEBS
- **Terms of Reference for WGDV**
- **Position of WGDV in the CCM Strategy 2014-2024**
- **Key and supplementary comparisons**
- **CMC and service category**

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.

CCM Strategy 2014-2024

➢ Section 7.1 Density

- In general, completed and planned KCs cover almost all of the CMCs on density. No frequent KCs are 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 *pρT* properties of fluids.
- As food industry and agriculture need a traceable standard of the refractive index of liquids for sugar content measurements, supplying refractive index standard liquids, which are similar to the density standard liquids, will be necessary.

➢ Section 7.8 Viscosity

- 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

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 (2015-)
Status	Technical Protocol in progress
Pilot	NMIJ (JP)

CCM.D-K4	Hydrometers (2011-2012)
Status	Report in progress, Draft A
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)

- CCM.D-K4 (hydrometer)
- Pilot: INRIM (IT)
- 11 participants
- 2 Groups: A & B
- Report in progress, Draft A

Group A	Group B	Scale div.	Diameter of body [mm]	Length of body [mm]	Diameter of stem [mm]	Length of stem [mm]	Weight [g]
0,600–0,610 g/cm^3	0,600 –0,610 g/cm^3	0,000 1 g/cm^3	28	260	5,5	140	90
0 – 10 % Vol.	0 – 10 % Vol.	0,1 % Vol	28	240	4	230	130
1,490 – 1,500 g/cm^3	1,490 – 1,500 g/cm^3	0,000 1 g/cm^3	32	270	4,5	140	290
1,980 – 2,000 g/cm^3	1,980 – 2,000 g/cm^3	0,000 2 g/cm^3	29	260	4	170	295

Participants

Laboratory	RMO
Istituto Nazionale di Ricerca Metrologica (**INRiM**) - ITALY	EURAMET
Physikalisch-Technische Bundesanstalt (**PTB**) - GERMANY	EURAMET
Magyar Kereskedelmi Engedélyezési Hivatal (**MKEH**) - HUNGARY	EURAMET
Central Office of Measures Glówny Urzad Miar (**GUM**) - POLAND	EURAMET
Laboratoire National d'Essais (**LNE**) - FRANCE	EURAMET
Centro Nacional de Metrologia (**CENAM**) - MEXICO	SIM
National Institute of Standards and Tecnology (**NIST**) - UNITED STATES of AMERICA	SIM
Laboratorio Tecnológico del Uruguay (**LATU**) - URUGUAY	SIM
Korea Research Institute of Standards and Science (**KRISS**) - REPUBLIC of KOREA	APMP
National Metrology Institute of Japan (**NMIJ**) - JAPAN	APMP
National Measurement Institute (**NMIA**) - AUSTRALIA	APMP

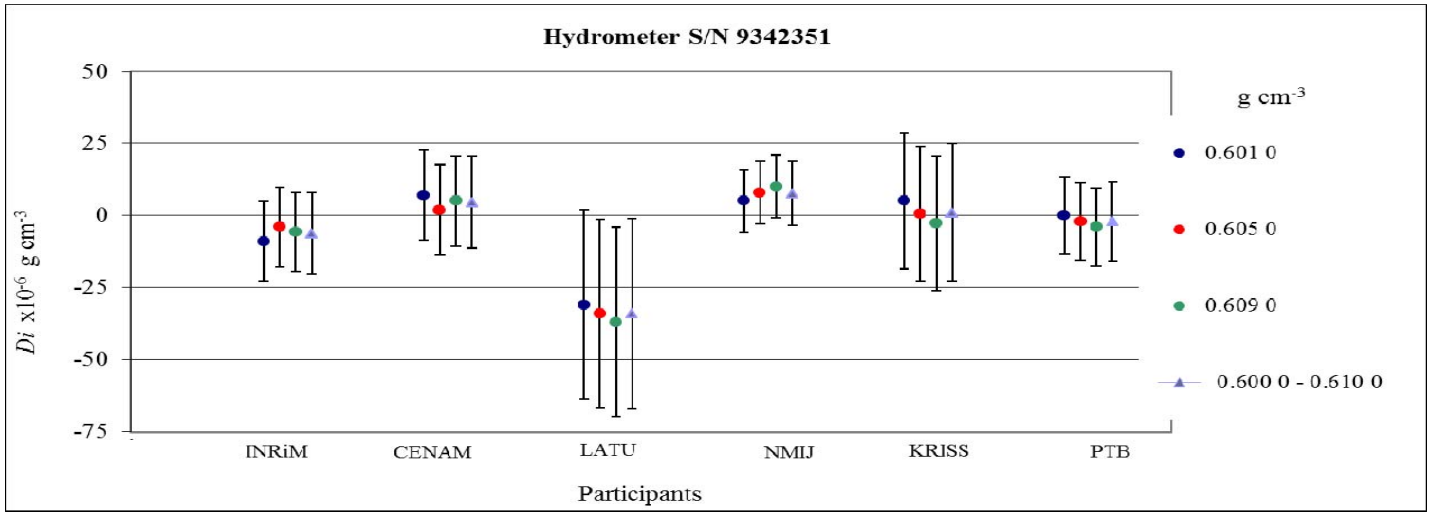
Circulation scheme for 2 groups

Group A			Group B	
Laboratory	**Data**		**Laboratory**	**Data**
INRIM	January 2011		INRIM	January 2011
CENAM	March – (15) April 2011		NMIA	March – (15) April 2011
LATU	June 2011		CENAM	June 2011
NMIJ	August 2011		NIST	August 2011
LNE	October 2011		KRISS	October 2011
to INRIM for ATA Carnet formality				
GUM	December 2011		MKEH	December 2011
PTB	February – March 2012		PTB	February – March 2012
INRIM	May - June 2012		INRIM	May - June 2012

Results of Group A

Hydrometer S/N 9342351 range 0.600 0 - 0.610 0 g cm^{-3}	x10^{-6} / g cm^{-3}	INRiM	CENAM	LATU	NMIJ	KRISS	PTB	$KCRV_A$ (weighted mean)	$U(KCRV_A)$
0.601 0		-104	-88	-126	-90	-90	-95	-95	7
0.605 0		-102	-96	-132	-90	-97	-100	-98	7
0.609 0		-96	-85	-127	-80	-93	-94	-90	7
Combined std uncertainty of corrections u_c		8	9	17	6	12	8	$\Pr\{\chi^2(\nu)>\chi^2_{obs}\}<0{,}05$?	
Expanded uncertaity of corrections $U_{95}=t_{95}u_c$		15	17	30	13	24	15	$\chi^2(5)=11.07>\chi^2_{obs}=(6.21; 6.03; 8.47)$	
Student t-factor t_{95}		1.98	1.97	1.97	2.07	1.97	1.96	The consistency test doesn't fail: procedure A	

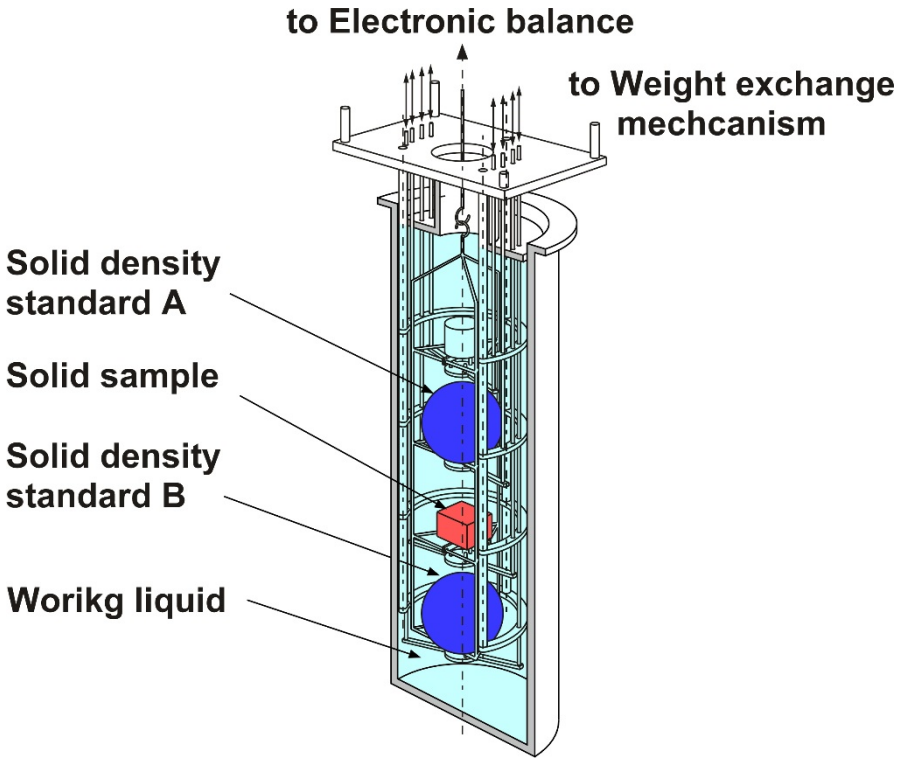
NMI	g cm^{-3}	0.601 0		0.605 0		0.609 0		0.600 0 - 0.610 0	
		Di x10^(-6)	$U(Di)$ x10^(-6)	Di x10^(-6)	$U(Di)$ x10^(-6)	Di x10^(-6)	$U(Di)$ x10^(-6)	Δj x10^(-6)	$U(\Delta j)$ x10^(-6)
INRiM		-9	14	-4	14	-6	14	-6	14
CENAM		7	16	2	16	5	16	5	16
LATU		-31	33	-34	33	-37	33	-34	33
NMIJ		5	11	8	11	10	11	8	11
KRISS		5	23	1	23	-3	23	1	24
PTB		0	13	-2	13	-4	13	-2	14



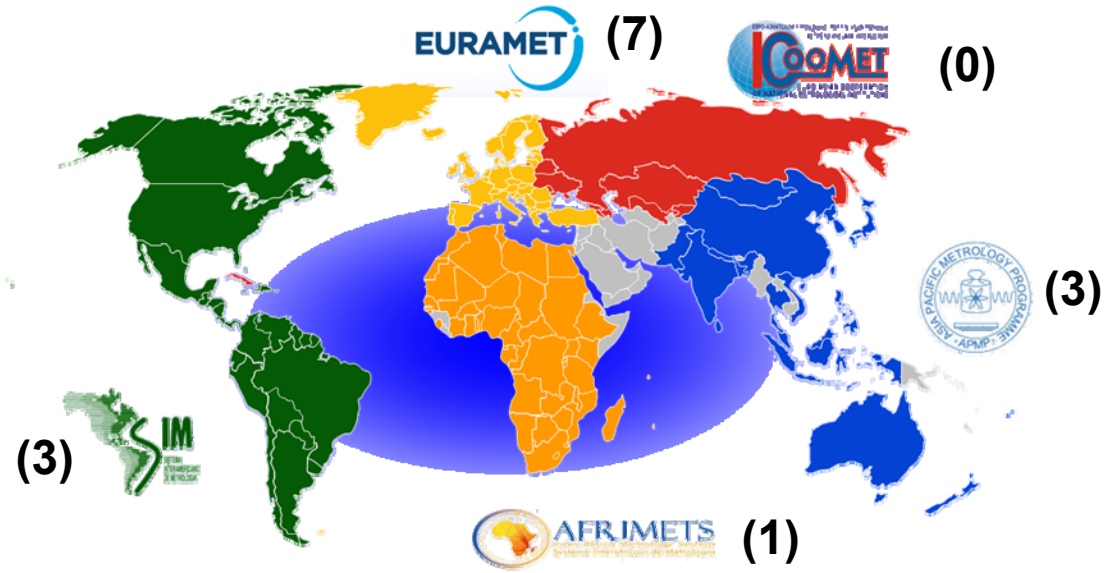
CCM.D-K3: Density of stainless steel weight

- CMC with ρ = 8000 kg/m^3: 15 NMIs
- Transfer standards: OIML type stainless steel weights
- Method of measurement: Hydrostatic weighing
- Masses: 1 kg, 200 g and 20 g
- Pilot: NMIJ (JP)

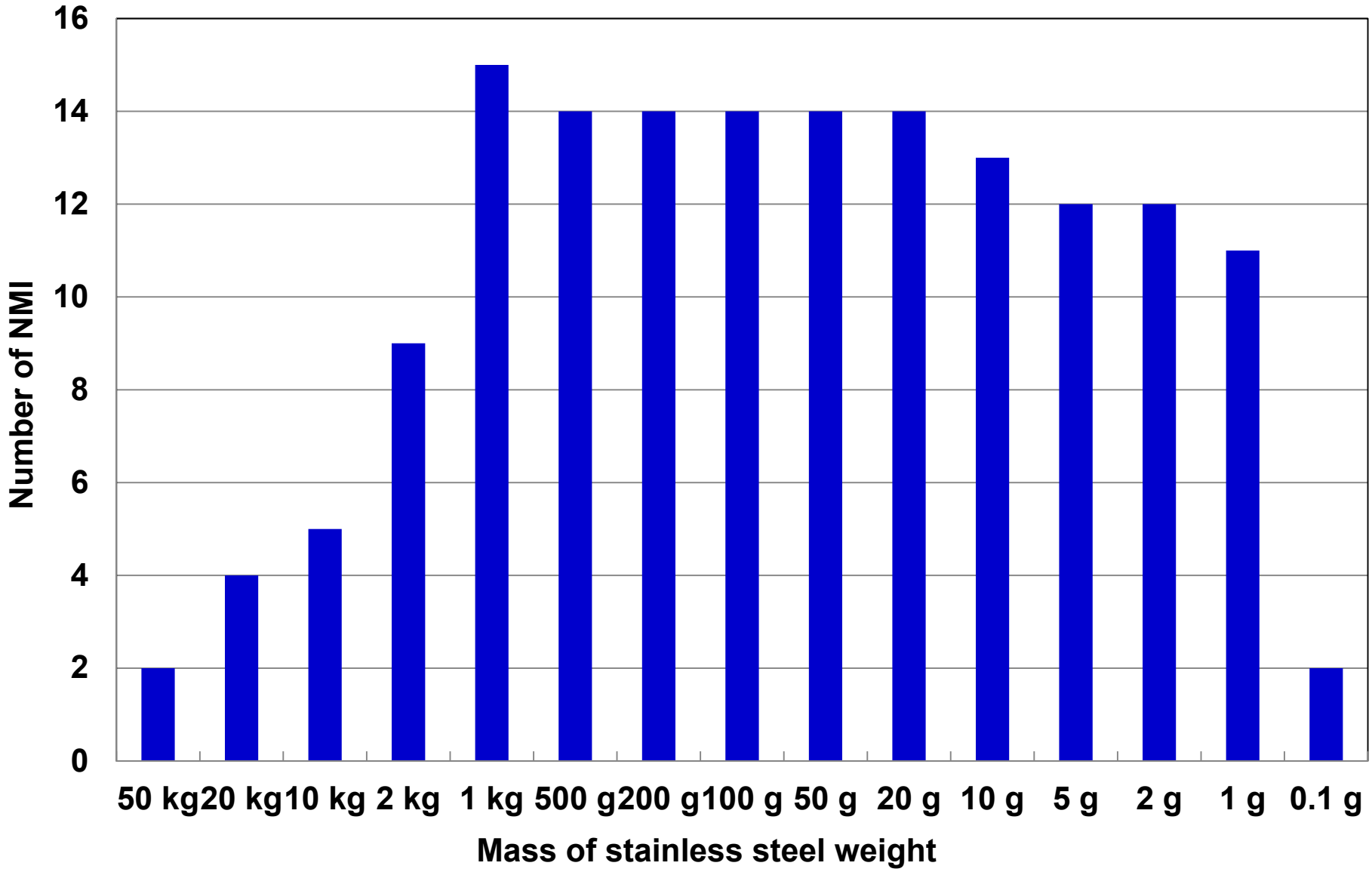
OIML type stainless steel weights



- **Answers from 14 NMIs and BIPM**
  - **APMP (3):** NIM (China), NIMT (Thailand), NMIJ (Japan)
  - **EURAMET (7):** CEM (Spain), INRIM (Italy), IPQ (Portugal), METAS (Switzerland), NPL (UK), PTB (Germany), UME (Turkey)
  - **AFRIMETS (1):** NMISA (South Africa)
  - **SIM (3):** CENAM (Mexico), INMETRO (Brazil), NRC (Canada)

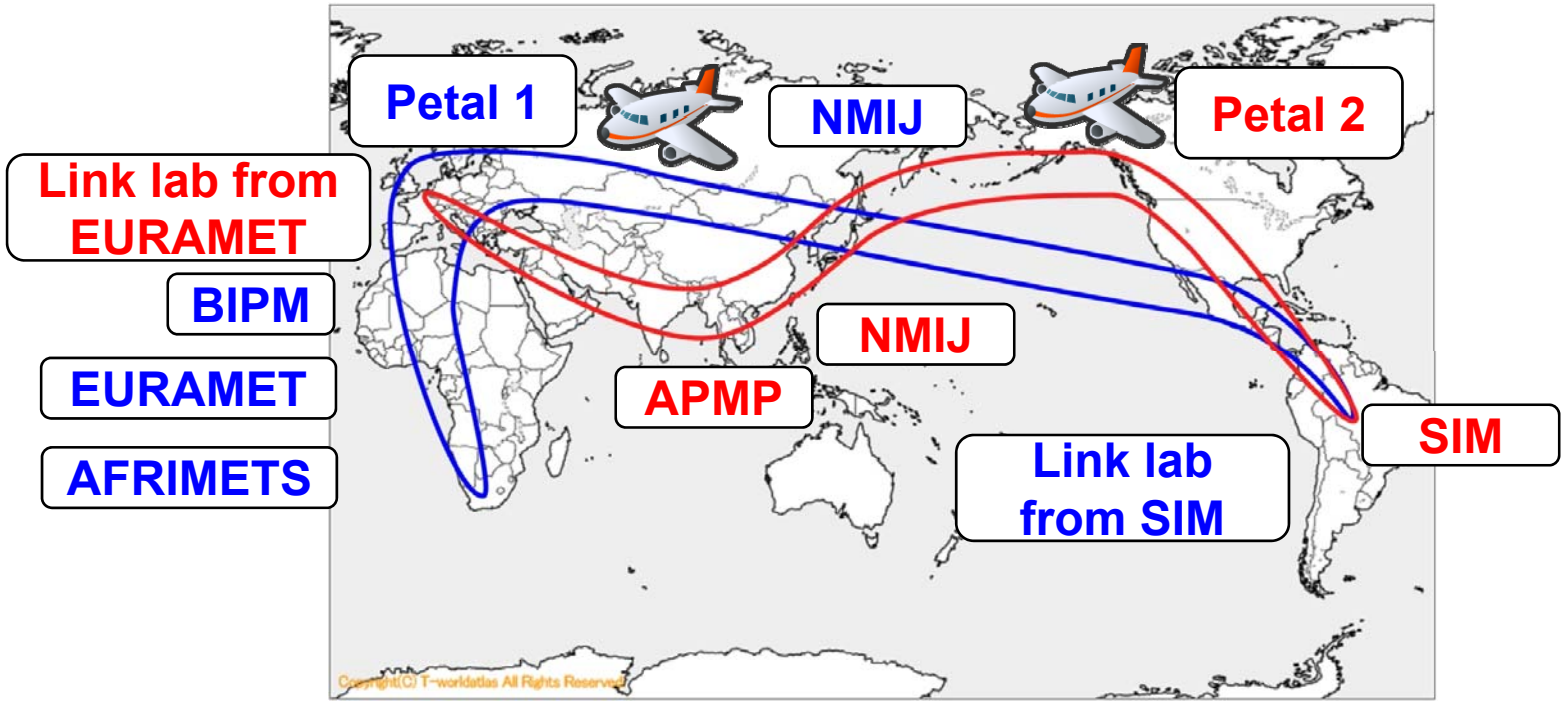


Q: Desired mass range of the stainless steel weights



Protocol will be prepared soon

- **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



CIPM Key Comparisons on Viscosity

CCM.V-K1	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)
CCM.V-K2	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)
CCM.V-K3	Three samples of Newtonian liquids: wide viscosity range (2012-2013)
Status	Report in progress, Draft A
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)

Link between CIPM 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
- CCM.D-K2 (Density of liquid) (2004) Approved for equivalence
  EURAMET.M.D-K2 (2008-2009) Report in progress, Draft B ← To be linked
  COOMET.M.D-S1 (2012-2015) Protocol complete
- CCM.D-K3 (Density of stainless steel weight) (2017) Planned
  SIM.M.D-K3 (2009-2010) Report in progress, Draft B
  SIM.M.D-S3 (2006) (volume of glass and stainless steel) Approved and published
  SIM.M.M-S11 (2012-2013) (Mass and volume of weight) In progress
- CCM.D-K4 (Hydrometer) (2011-2012) Report in progress, Draft A
  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 ← Linked
  COOMET.M.V-S1 (2013) Report in progress, Draft B
- CCM.V-K2 (wide temperature range)
- CCM.V-K2.1 (2008) (comparison to link Egypt and South Africa)
- CCM.V-K3 (2012-2013) (wide viscosity range) Report in progress, Draft A

New Key Comparisons on Density

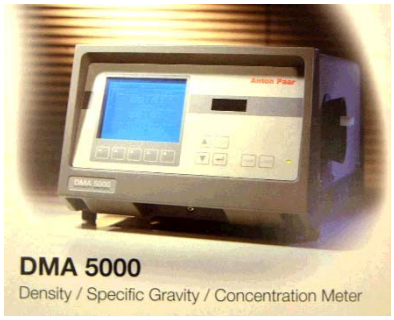
(1) Oscillation-type density meter

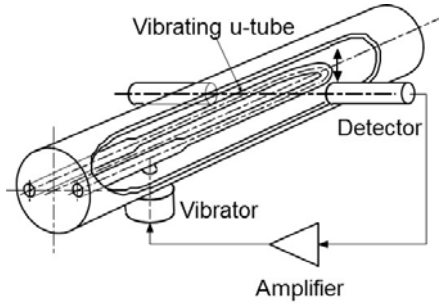
Concept of this KC

- 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.

Agreed as CCM.D-K5: BEV (AT) was proposed to be the pilot.

11 NMIs were interested in this KC.





New Key Comparisons on Density

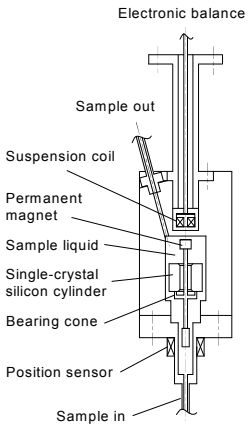
(2) Density measurement under high-pressure

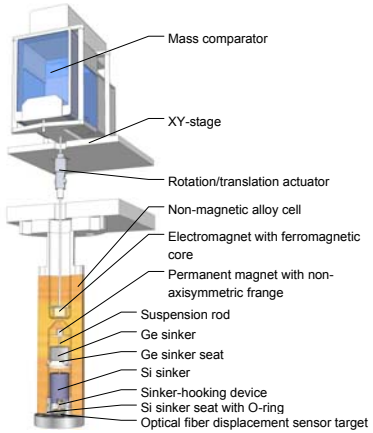
Concept of this KC

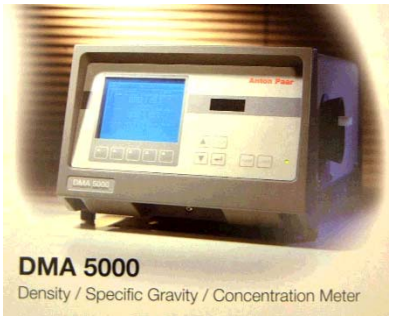
- Pilot institute distributes fluids to participants.
- Method of measurement
  - Magnetic-suspension density meter
  - Oscillation-type density meter
  - Pycnometer

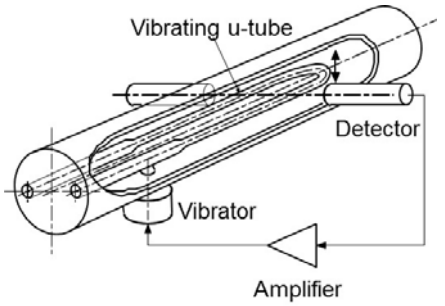
Agreed as CCM.D-K6

4 NMIs were interested in this KC.







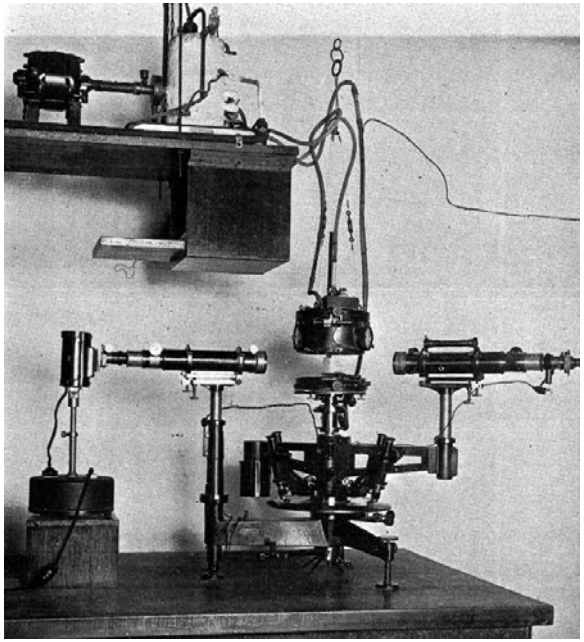


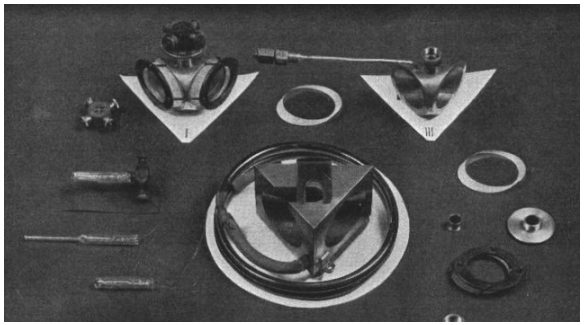
Demand for CMC on Refractive Index of Liquid

- Common method for evaluating the sugar concentration in food is to measure the refractive index of liquid sample
- Calibration of the refractometers using a liquid of known refractive index is therefore necessary.
- Practical solution is to use the **density standard liquids** also as the **refractive index standard liquids**.

Examples of density standard liquids used also as refractive index standard liquids

Traditional Method: Minimum Deviation Angle

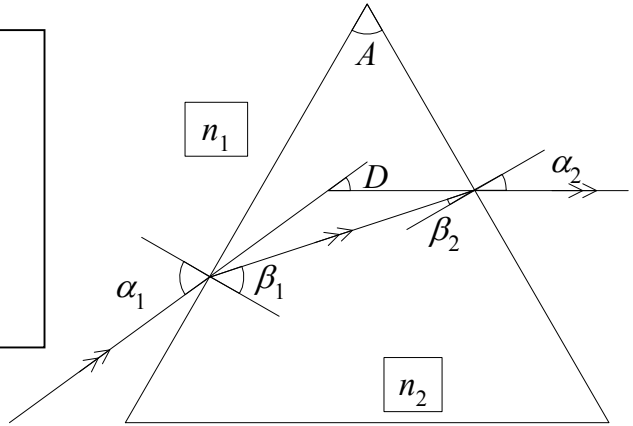




n_{water} by L. W. Tilton and J. K. Taylor.
J. Res. Natl. Bur. Stand., 20:419, 1938.

Snell's law

$$n = \frac{\sin\alpha}{\sin\beta}$$



$$n = \frac{\sin\left[(A + D_{\min})/2\right]}{\sin(A/2)}$$

Advantage

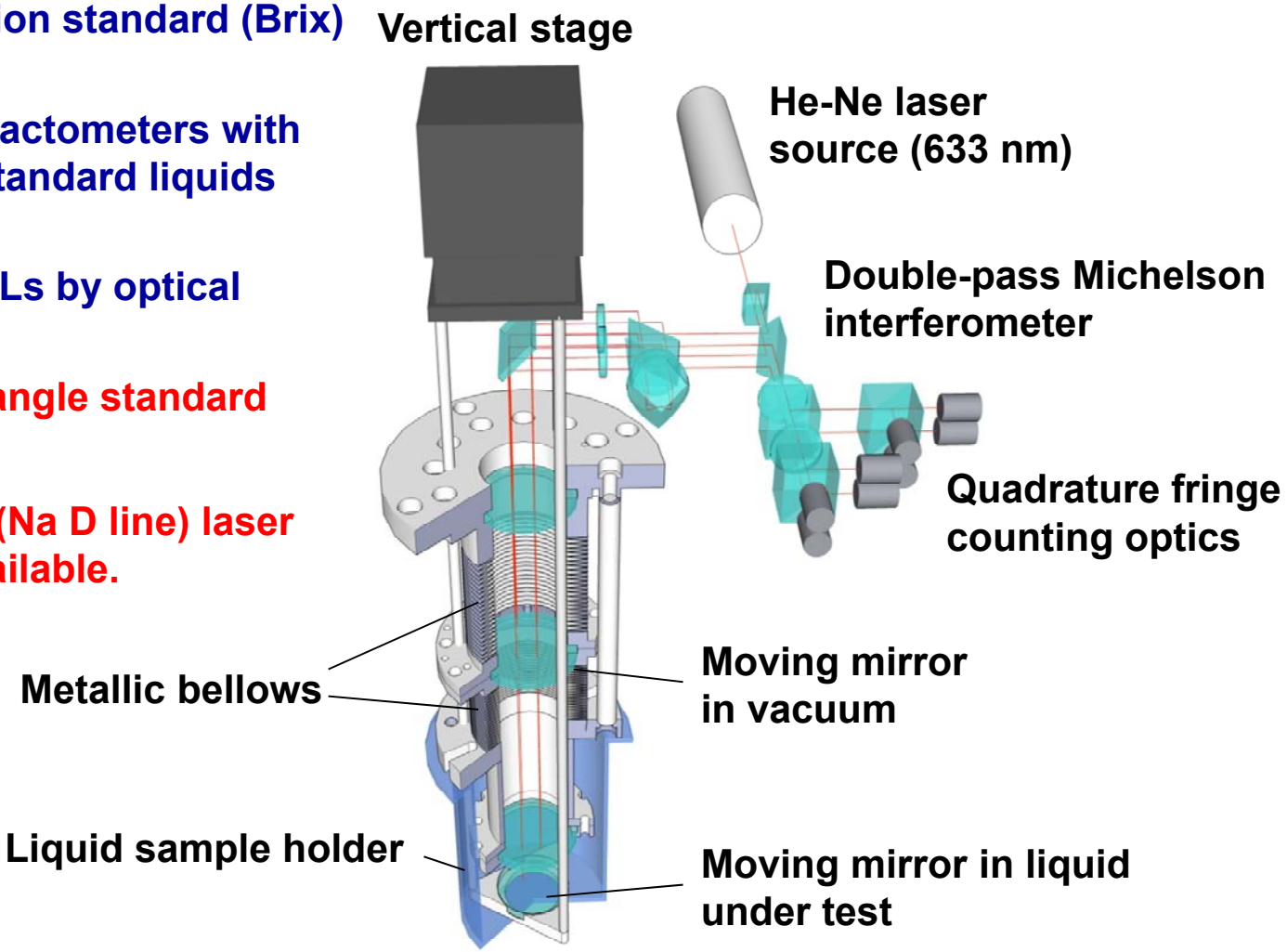
- Traceable to angle standards (goniometer, prism)
- Applicable for incoherent light sources (lamp, LED)

Disadvantage

- Limited resolution and accuracy (≈ 10^{-5})
- Complexity in setup for angle measurement

New Method: Optical Interferometry

- Sugar concentration standard (Brix) for food industry
- Calibration of refractometers with refractive index standard liquids (RISLs)
- Calibration of RISLs by optical interferometry
- No necessity for angle standard
- $u_r(n) \approx 3 \times 10^{-6}$
- Compact 589 nm (Na D line) laser source is now available.



Existing KC and CMC on Refractive Index

- **COOMET.PR-S3: Refractive index of solid glass**
  - **Participants: VNIIOFI (RU), PTB (DE), Ukrmetrteststandart (Ukraine), NMIJ (JP), INRIM (IT), KazInMetr (Kazakhstan)**
- **CMCs: index of refraction in CCL**
  - **GUM: Refractive index of materials measuring instruments Refractometers: refractive index, *n*, 1.32 to 1.7**
  - **NMIJ: Triangular prism, *n*, 1.51 to 1.52 Material: BK7 or equivalent glass**
  - **These CMCs in CCL are to be transferred to CCPR.**
  - **Service category for refractive index will be prepared soon at CCPR.**

Proposal

Considering that food industry often requires traceability in the refractive index standard liquids, practical solution would be as follow:

- Refractive index of liquid → CCM
- Other optical properties → CCPR

This proposal was accepted by the CCM WGS meeting yesterday.

New Key Comparison on Refractive Index

(3) Refractive index of liquid

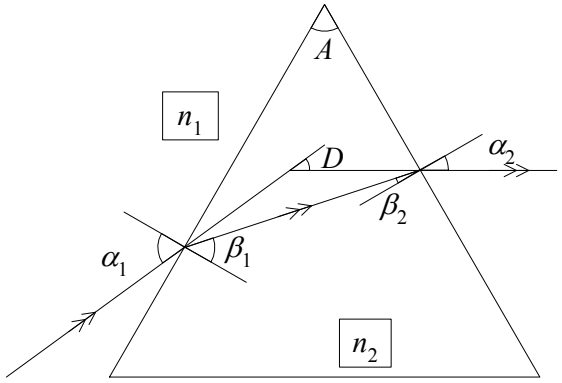
Concept of this KC

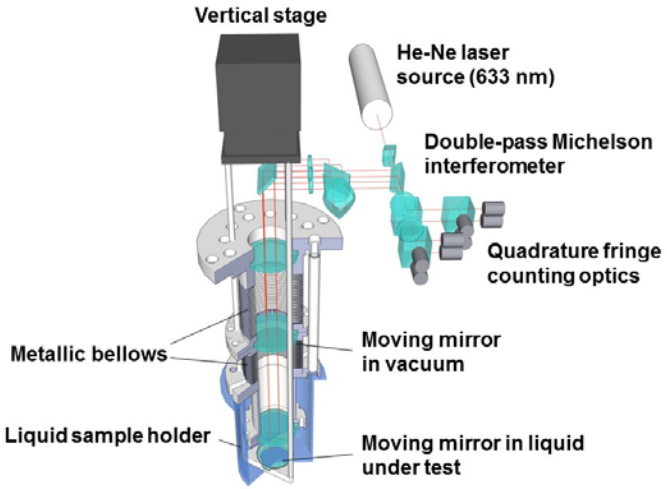
- Pilot institute distribute liquid samples to participants.
- Participants measure the refractive index of the liquid by their own refractometers.

Agreed as CCM.D-K7, Pilot: NMIJ

4 NMIs were interested in this KC.

Participants from other CCs are also welcome.





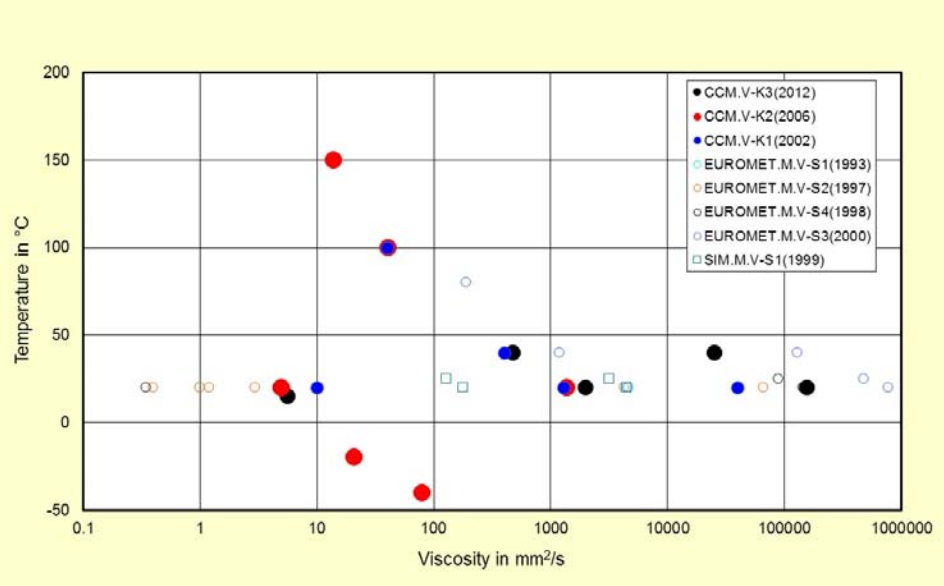
New Key Comparisons on Viscosity

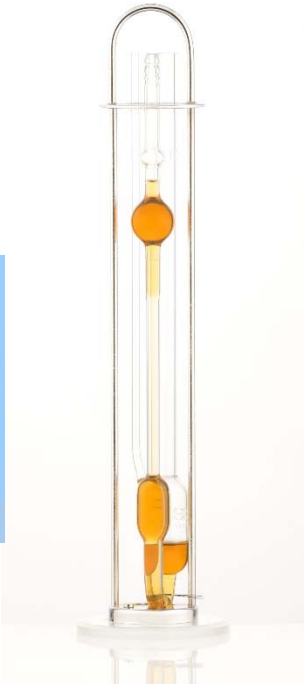
(4) Viscosity measurements in a wide temperature range

Concept of this KC

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

Agreed as CCM.V-K4, Pilot: CENAM (MX)





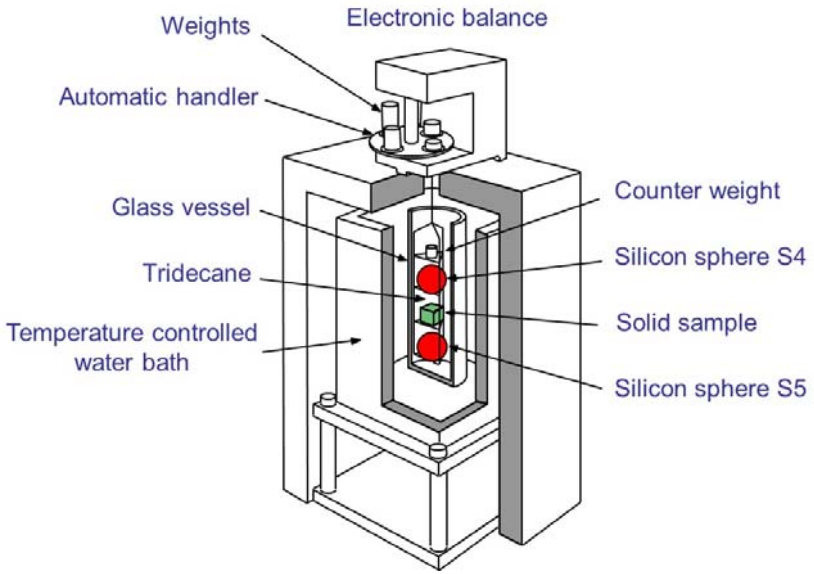
Repetition of Key Comparisons on Density

(5) Repetition of CCM.D-K1 (final report: 2006)

Concept of this KC

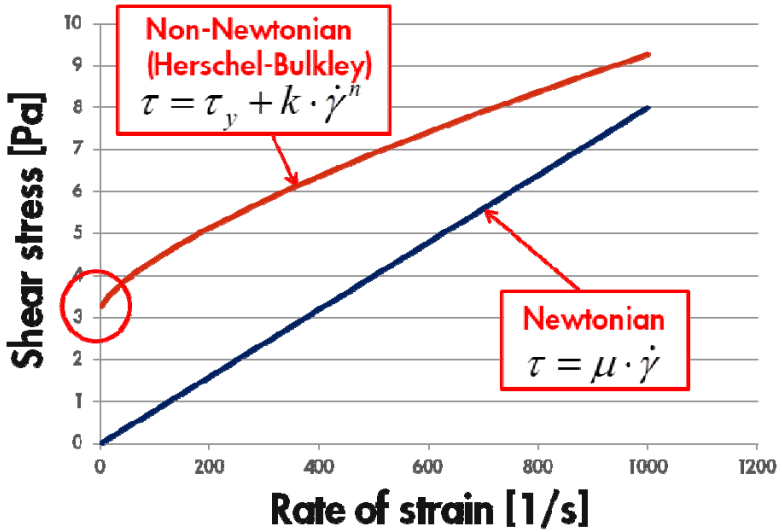
- Pilot institute circulate one or a few Si spheres to participants.
- Participants measure the density by hydrostatic weighing.

Agreed to repeat this KC for a period of 10-15 years

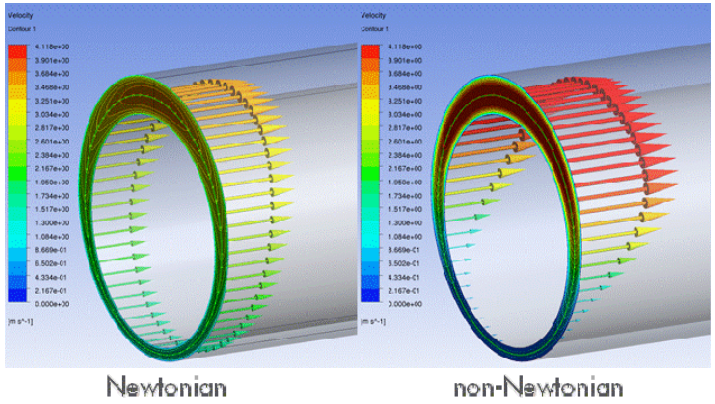


From Viscometry to Rheometry

(presented by Ronald Pagel, PTB)



blood viscosity (Fåhraeus-Lindquvist effect)
H. Stark, S. Schuster, J. Appl. Physiol., 2012, 113, 355-367



Non-Newtonian Fluids (presented by Ronald Pagel, PTB)

blood

mortar

toothpaste

mayonnaise

shaving foam

EMRP ENG 59 Non-Newtonian Liquids

"A complete, enhanced sensor package should be standard on every rig"

From conventional drilling fluid engineer and sensors

to future drilling fluid engineering and sensors

Objectives

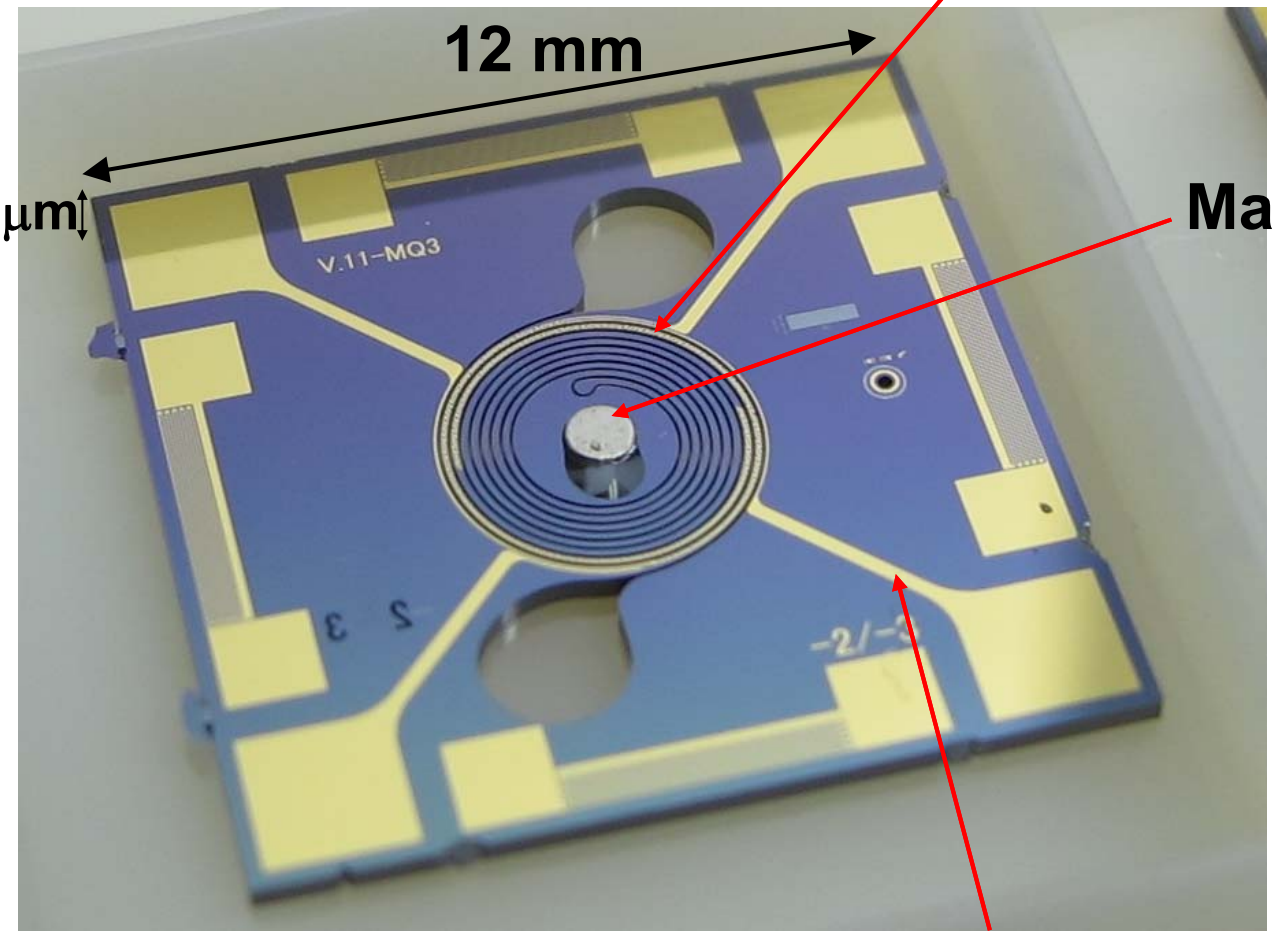
- Develop rheology measurement standard and reference materials
- Determine physical properties of non-Newtonian liquids
- Develop inline sensors including on-site calibration methods

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

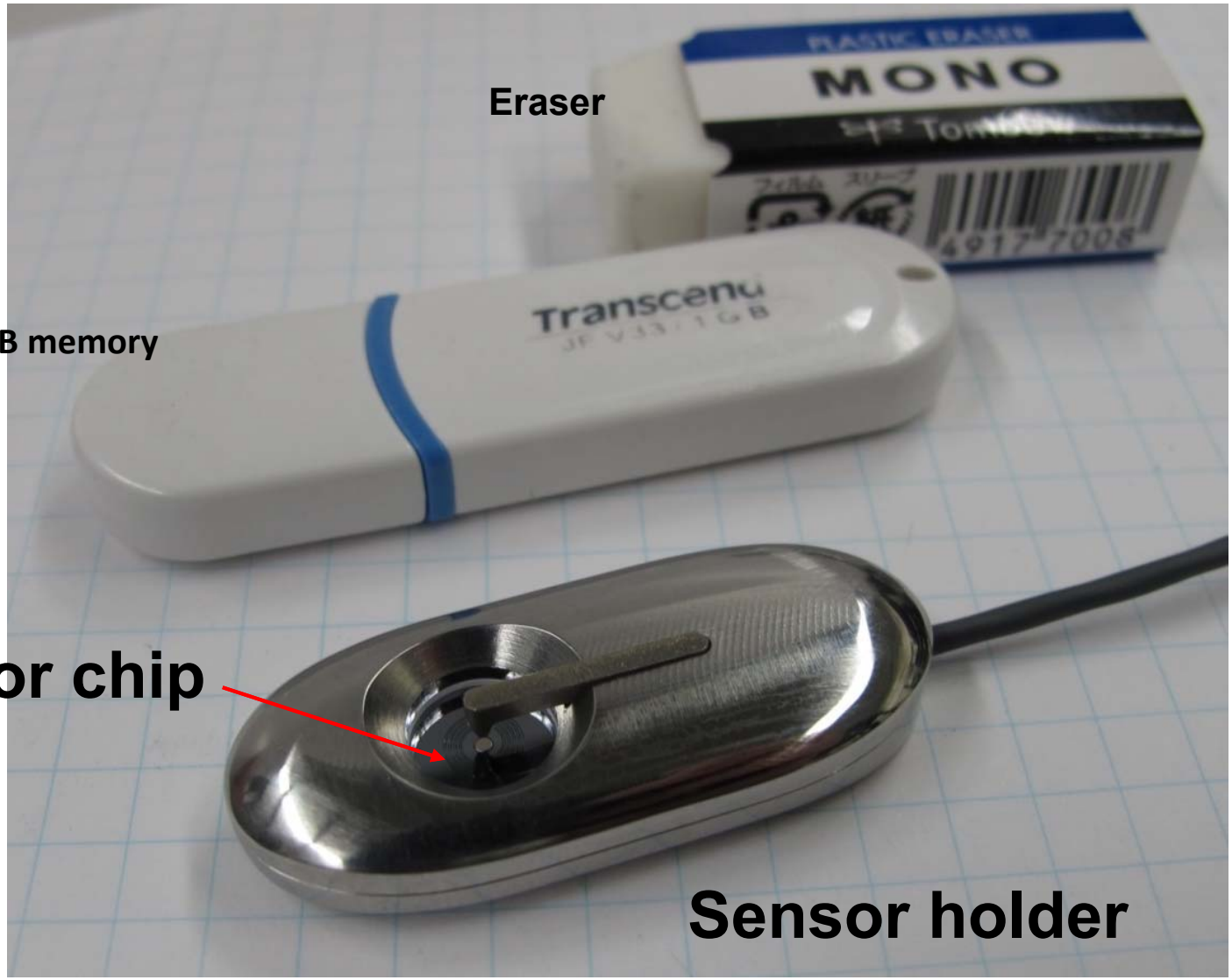
MEMS based viscosity sensor developed at NMIJ

MEMS Sensor chip

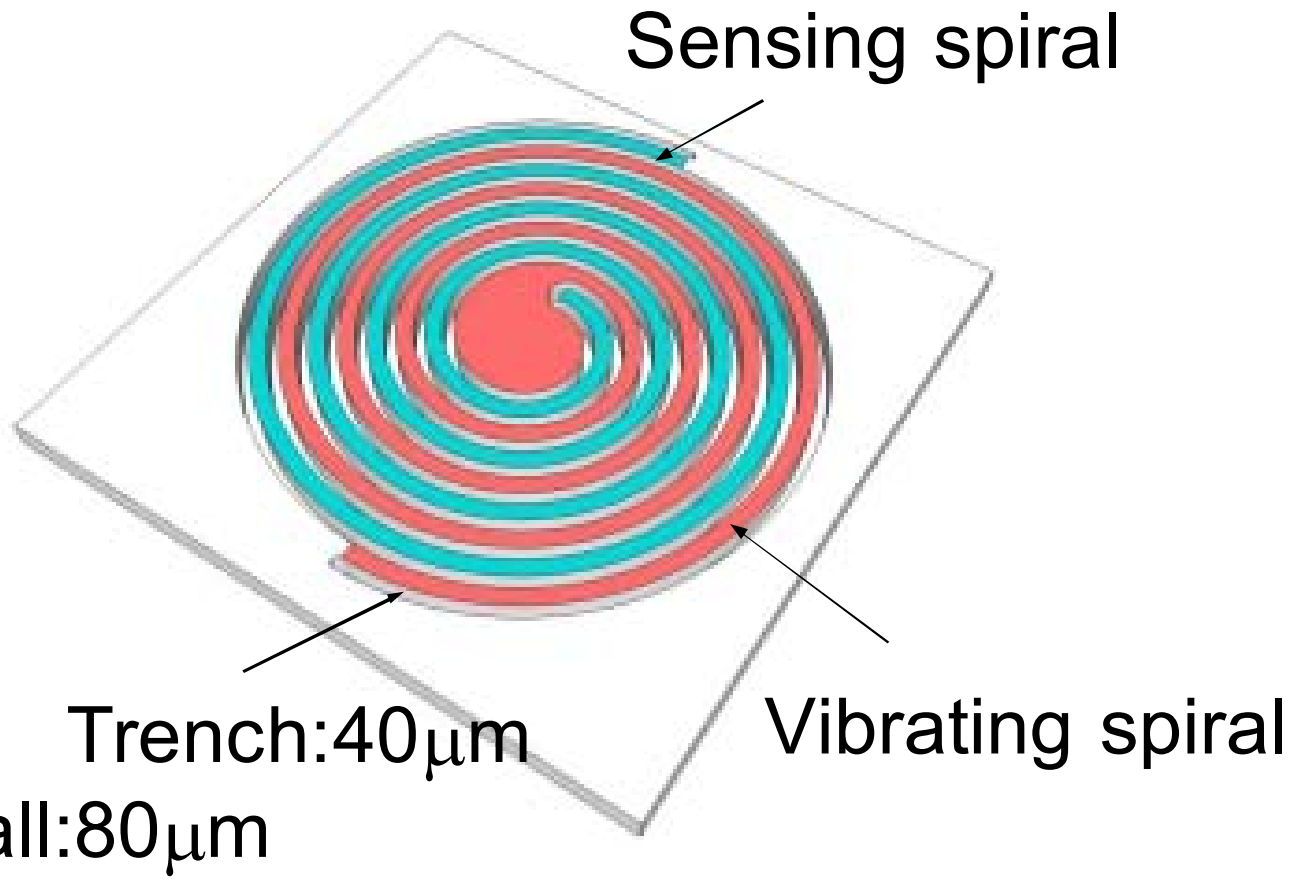


Application
- In-line
- Multipoint
- Built-in
- Array, etc.

Viscosity sensor holder

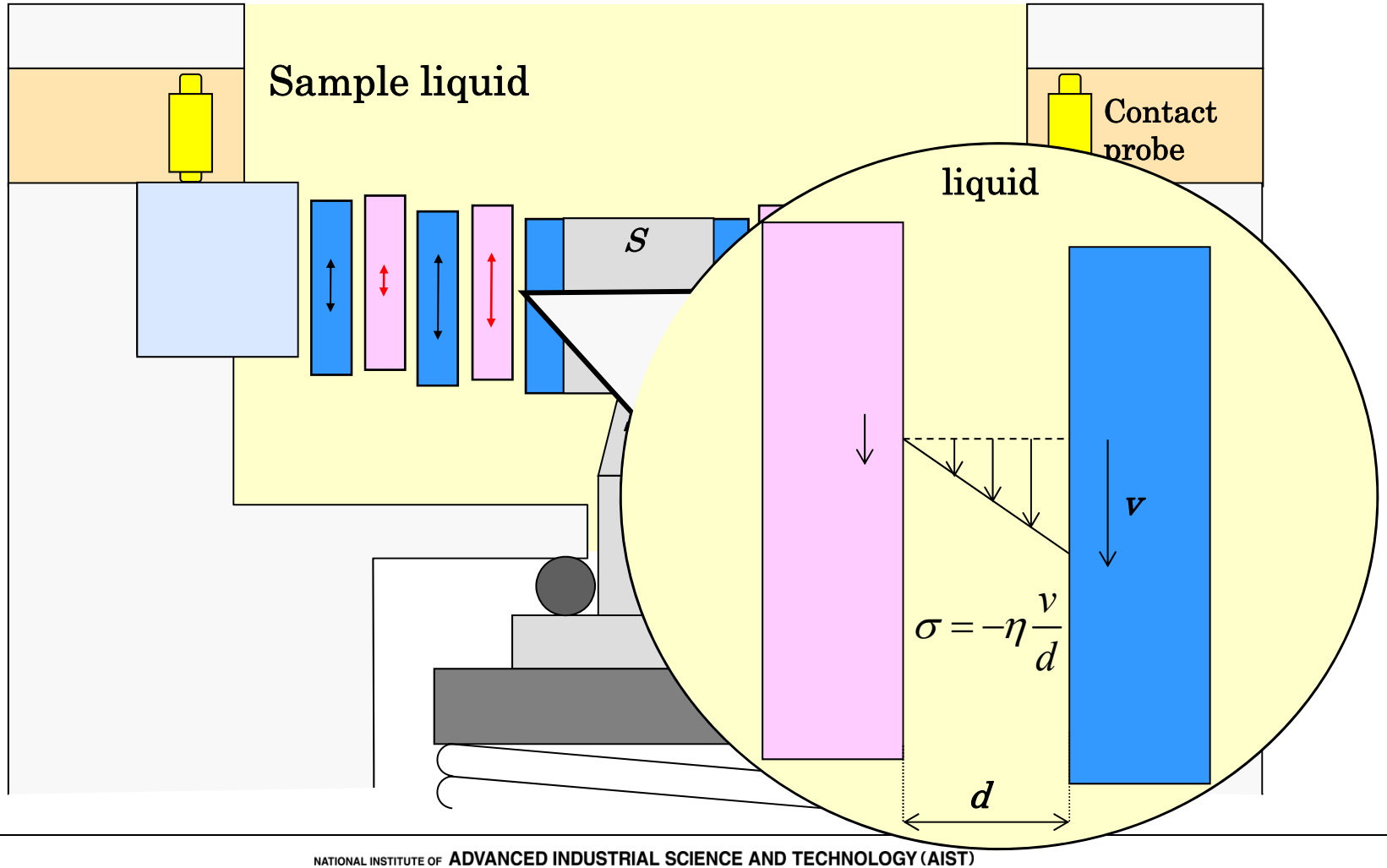


Schematic view of the sensor chip

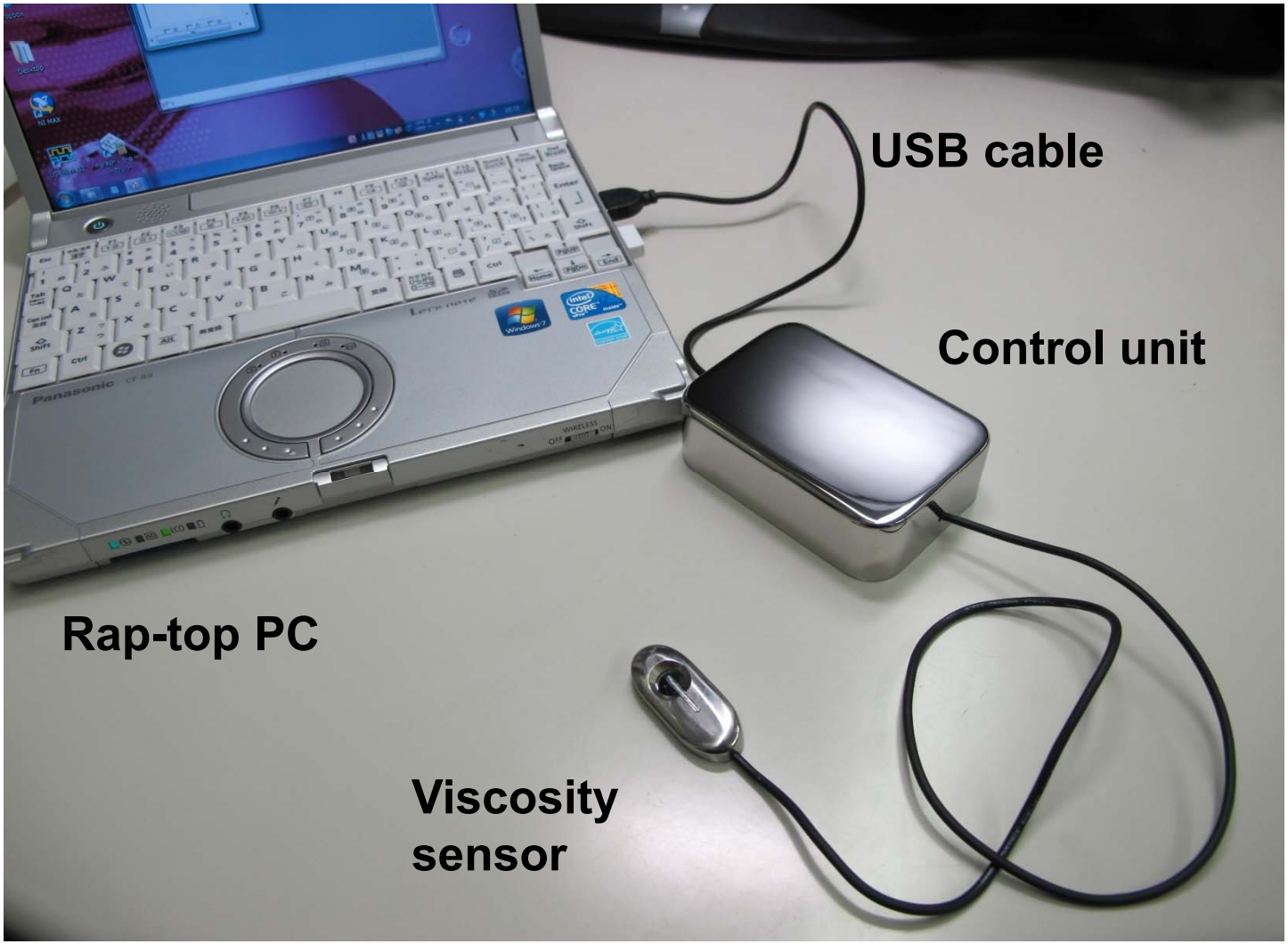


Principle of the viscosity sensor

Cross sectional view of the dual spiral



Measurement system of viscosity sensor



Viscosity calibration of Non-Newtonian liquids

Cylinder balance method

Advantage of translational method:
viscous force measurement
with traceability to mass standard

Calibration service started from 2010

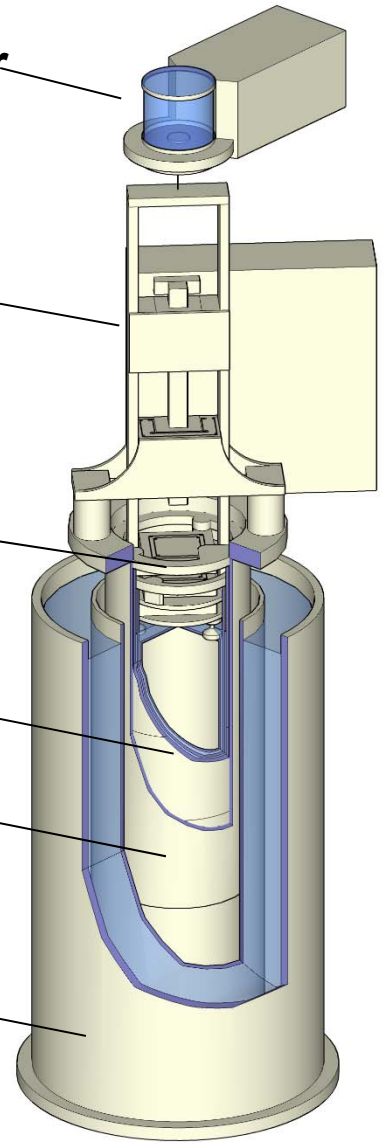
Shear stress : 5 X 10^{-3} Pa ～ 34 Pa

Shear rate : 0.1 s^{-1} ～17 s^{-1}

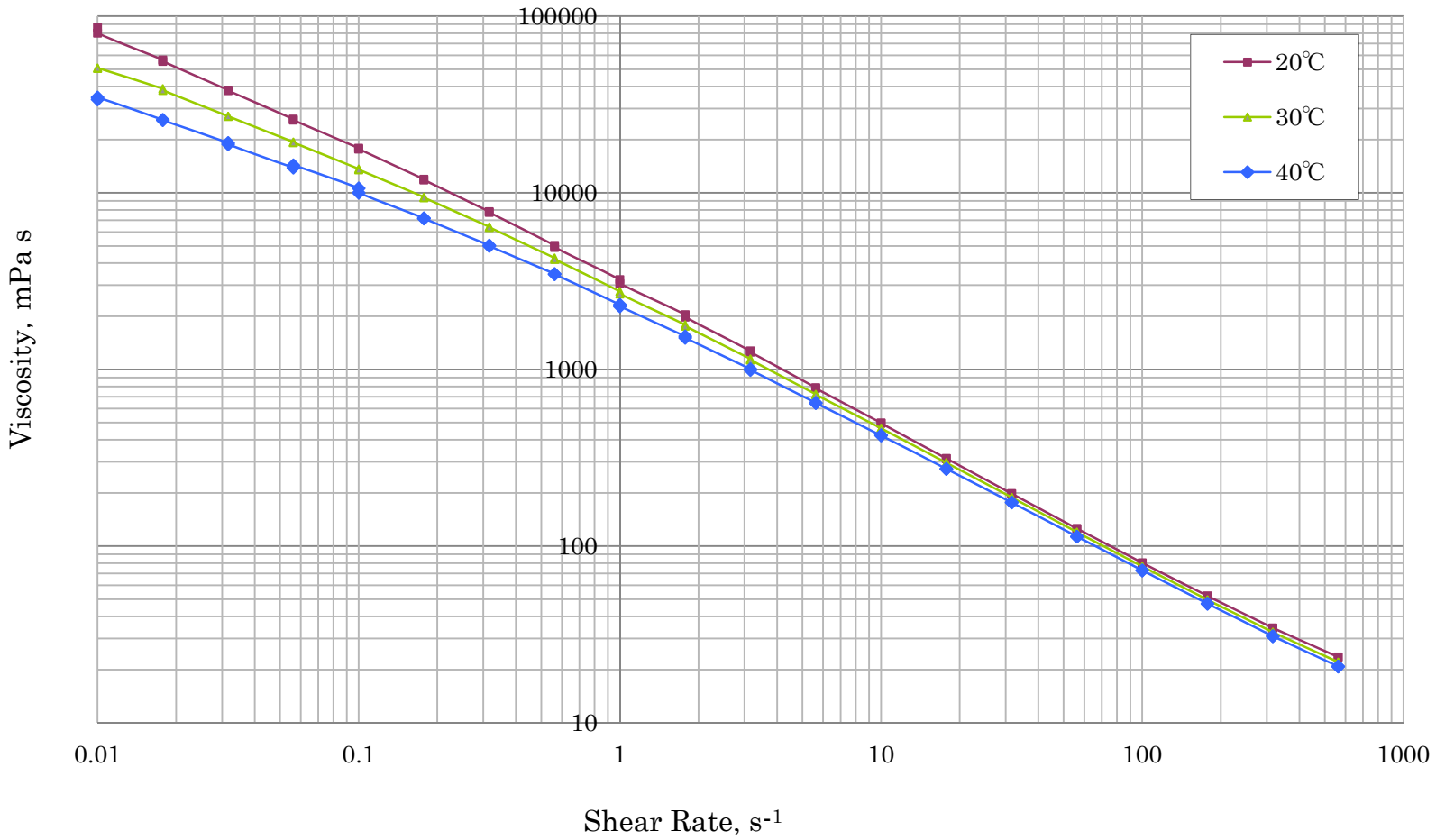
Temperature : 20 ºC

Uncertainty : 6 % (95 % level of confidence)

Searching for candidates for standard non-Newtonian liquids



Non-Newtonian properties of candidate substances



Conclusion

- After unification, the first WGDV meeting was held on Feb 24. There were 30 participants.
- Progress of CCM.D-K4 (hydrometer) and CCM.V-K3 (wide viscosity range) was discussed. They are close to Draft B.
- Scheme of CCM.D-K3 (stainless steel weight) were discussed.
- New KCs were planned and agreed:

  CCM.D-K5: Liquid density measurement by oscillation-type density meter

  CCM.D-K6: Density measurement under high-pressure

  CCM.D-K7: Refractive index of liquid

  CCM.V-K4: Measurement of viscosity standard liquids in a wide temperature range

- Reference materials and demand for non-Newtonian liquids were discussed.