

CCM meeting on 21-22 February 2013, BIPM

## WGD Report for CCM 2013

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Chair, CCM WG on Density

- **WGD meeting held at the BIPM on May 9th, 2011**
  - 16 members: BEV, BIPM, CEM, CENAM, INRIM, KRISS, NIM, NMIA, NMIJ, NPL, NRC, PTB
  - 9 guests: CESMEC, GUM, Inmetro, IPQ, LATU, NIMT, NMISA
- **Terms of Reference**
- **Present state of key and supplementary comparisons**
- **Density of water**
- **Major progress**
- **Technological trends and challenges**

# Terms of reference of WGD

- To improve techniques for realizing the SI unit of density;
- To exchange information on the density standard;
- To perform CIPM key comparisons for supporting CMCs on density;
- To coordinate RMO key and supplementary comparisons for promoting the CIPM MRA in the field of density;
- To provide guidance to accept CMCs on density;
- To coordinate activities for density measurements at NMIs; and
- To assess metrological needs and seeds on density.

# CIPM Key Comparisons

**CCM.D-K1** Density measurements of a silicon sphere by hydrostatic weighing (2001-2003)  
 Status Approved for equivalence  
 Pilot NMIJ (JP)  
 Pilot group NMIJ (JP), 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 Final report  
 Pilot PTB (DE)  
 Pilot group PTB (DE), 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 (2011-)  
 Status Planned (questionnaire distributed and answers received)  
 Pilot NMIJ (JP)

**CCM.D-K4** Hydrometers (2011 - 2012)  
 Status Report in progress, Draft A  
 Pilot INRIM (IT)  
 Pilot group INRIM (IT), CENAM (MX), PTB (DE)  
 Participants INRIM (IT), CENAM (MX), PTB (DE), LATU (UY), NMIJ (JP), LNE (FR), NMIA (AU), NIST (US), KRISS (KR)

# RMO Key and Supplementary Comparisons (1)

<b>EUROMET.M.D-K1</b>	Volume and density comparisons of three ceramic spheres (1996-1999)
Status	Approved for provisional equivalence
Pilot	METAS (CH)
Participants	METAS (CH), SP (SE), PTB (DE), BEV (AT), INRIM (IT), NPL (UK), SM (BE), CEM (ES), BNM-LNE (FR), FORCE (DK), OMH (HU), UME (TR)
Other designation	EUROMET Project No 339
Comment	No report from SM (BE)
<b>EURAMET.M.D-K1.1</b>	Density measurements of silicon spheres (2008-2010)
Status	Report in progress, Draft A
Pilot	PTB (DE)
Participants	PTB (DE), NPL (UK), INRIM (IT), LNE (FR), MIKES (FI), NMIJ (JP), IPQ (PT), METAS (CH), GUM (PL), CEM (ES), METAS (CH), EIM (GR), BEV (AT), VNIIM (RU), UME (TR), DZM (HR), NIS (EG), INM (RO)
Other designation	EURAMET Project No 1031
<b>EUROMET.M.D-K2</b>	Density of liquids (2001-2002)
Status	Final report accepted
Pilot	PTB (DE)
Participants	PTB (DE), MIKES (FI), BNM-LNE (FR), OMH (HU), INRIM (IT), JV (NO), GUM (PL), CSIR-NML (ZA)
Other designation	EUROMET Project No 627
Comment	<a href="#">This KC is an old version, and replaced with a new KC in 2008</a>
<b>EURAMET.M.D-K2</b>	Density of liquids (2008-2009)
Status	Final Report
Pilot	BEV (AT)
Participants	BEV (AT), PTB (DE), GUM (PL), IPQ (PT), CEM (ES), TUBITAK-UME (TR), INM (RO), INRIM (IT), MIREN (FI), DZM (HR), JV (NO), MKEH (former OMH) (HU), LNE (FR), NPL (UK), SMU (SK)
Other designation	EURAMET Project No 1019
<b>EURAMET.M.D-S1</b>	Density of liquids (2012-2013)
Parameter(s)	Temperature: 15 °C to 40 °C, Atmospheric pressure
Status	In progress
Comment	see EURAMET.M.D-K2

# RMO Key and Supplementary Comparisons (2)

<b>EUROMET.M.D-K4</b>	Comparison of the calibrations of high resolution hydrometers for liquid density determination (2003-2005)
Status	Approved for equivalence
Pilot	INRIM (IT), PTB (DE), OMH (HU)
Participants	BEV (AT), PTB (DE), MIKES (FI), BNM-LNE (FR), OMH (HU), INRIM (IT), GUM (PL), IPQ (PT), SMU (SK), UME (TR), VNIIM (RU)
Other designation	EUROMET Project No 702
<b>EUROMET.M.D-K4.PREV</b>	Hydrometers comparison for liquid density determination (1993-1994)
Status	Approved for provisional equivalence
Pilot	INRIM (IT) (former IMG-CNR)
Participants	INRIM (IT), OMH (HU), PTB (DE), SMU (SK), IPQ (PT)
Other designation	EUROMET Project No 236
<b>EURAMET.M.D-K4.S1</b>	Supplementary comparison to EUROMET.M.D-K4 (temperature extension for hydrometers) 2011-
Status	Approved by EURAMET, Protocol in preparation
Pilot	INRIM (IT)
Participants	INRIM (IT), LNE (FR), GUM (PL), NMI (RO), IPQ (PT)
<b>SIM.M.D-K3</b>	Volume of solid weights (2009-2010)
Status	In progress
Pilot	INTI (AR)
Participants	INTI (AR), XENAM (MX), LACOMET (CR), INMETRO (BR), CESMEC Ltda. (CL), LATU (UY), NRC (CA)
Other designation	SIM.M.M.K52
<b>SIM.M.D-K4</b>	Comparison of the calibration of density hydrometers (2007-2008)
Status	Approved for equivalence
Pilot	CENAM (MX)
Participants:	CENAM (MX), NRC (CA), NIST (USA), BSJ (JM), LACOMET (CR), CENAMEP (PA), INMETRO (BR), IBMETRO (BOLIVIA), SIC (CO), INEN (EC), INDECOPI (PE), CESMEC Ltda. (CL), LATU (UY)
Other designation	SIM.7.33
<b>SIM.M.D-S1</b>	Comparison of the calibration of hydrometers for liquid density determination (bilateral CENAM - INRIM) 2007
Status	Approved and published
Participants	CENAM (MX), INRIM (IT)

# RMO Key and Supplementary Comparisons (3)

<b>SIM.M.D-S2</b>	Comparison of the calibration of hydrometers for liquid density determination (bilateral INMETRO - INRIM) 2009-2010
Status	Approved and published
Participants	INMETRO (BR) and INRIM (IT)
<b>SIM.M.D-S3</b>	Comparison of volume of solids by hydrostatic weighing (bilateral INMETRO - INRIM) 2006
Status	Approved and published
Participants	INMETRO (BR) and CENAM (MX)
<b>SIM.M.D-S4</b>	Comparison of calibration of hydrometers for liquid density determination 2012
Status	Protocol complete
<b>APMP.M.D-K4</b>	Comparison of calibrations of density hydrometers (2007-2010)
Status	Report in progress, Draft A
Pilot	KRISS (KR)
Pilot group	KRISS (KR), NMIJ (JP)
Participants	NMIA (AU), CSIR (ZA), SIRIM (MY), IRL (NZ), NML (PH), NMIJ (JP), NIM (CH), NIMT (TH), NPL (IN), KRISS (KR)

# Other Bilateral and International Comparisons

- 1) Anna M. Peuto, Attilio Sacconi, Maria Mosca, Kenichi Fujii, Mitsuru Tanaka, and Yoshiyuki Nezu, "Comparison of Silicon Density Standards at NRLM and IMGC," IEEE Trans. Instrum. Meas., Vol. 42, No. 2, pp. 242-246, 1993.
- 2) Horst Bettin, Michael Gläser, Frank Spieweck, Hans Toth, Attilio Sacconi, Anna M. Peuto, Kenichi Fujii, Mitsuru Tanaka, and Yoshiaki Nezu, "International Intercomparison of Silicon Density Standards," IEEE Trans. Instrum. Meas., Vol. 46, No. 2, pp. 556-559, 1997.
- 3) Kyung-Ho Chang, Yong-Jae Lee, and Jin-Wan Chung, "Final Results of Bilateral Density Comparison between NMIJ and KRISS for 1 kg Weight," KRISS/MO-2001-058, June 2001.
- 4) H. Bettin, H. Toth, A. Waseda and K. Fujii, "Comparison of density difference measurements at PTB and NMIJ," IEEE Trans. Instrum. Meas., Vol. 54, No. 2, pp. 877-881, 2005.
- 5) SIM.7.34-B: Bilateral Comparison on volume of solids by hydrostatic weighing between CENAM-Mexico and INMETRO-Brazil.  
Status: Report published
- 6) Loreface S., Heinonen M., Madec T. "Bilateral comparisons of hydrometer calibrations between the IMGC-LNE and the IMGC-MIKES," Metrologia, 2000, Vol. 37, No. 2, pp. 141-147.
- 7) International comparison of absolute diameter measurements by optical interferometry at NMIJ, PTB, and NMIA (2006-2009) in IAC project  
Status: Measurements completed
- 8) Bilateral comparison of absolute volume calibrations by optical interferometers at NMIJ and KRISS (2006-2007)  
Status: Measurements completed
- 9) Bilateral comparison of absolute volume calibrations by optical interferometers at NMIJ and NIM (2008)  
Status: Planned
- 10) Kari Riski, "Mass and volume comparisons at MIKES—Additional results to the EA intercomparison of weights 1 mg – 100 g (Ma1) and to the EUROMET intercomparison of ceramic spheres (EUROMET 339)," Julkaisu J4/2000

# Links between Key, Supplementary, Bilateral, and International Comparisons

CIPM key comparison	RMO key comparison	Other bilateral and international comparisons
CCM.D-K1 (completed)	EUROMET.M.D-K1 (completed) EURAMET.M.D-K1.1 (Draft A in progress)	1), 2), 4), 7), 8), 9), 10)
CCM.D-K2 (completed)	EUROMET.M.D-K2 (completed) EURAMET.M.D-K2 (completed)	EURAMET.M.D-S1
CCM.D-K3 (questionnaire)	SIM.M.D-K3 (in progress)	3), 11)
CCM.D-K4 (Draft A in progress)	EUROMET.M.D-K4 (completed) EURAMET.M.D-K4 (completed) SIM.M.D-K4 (completed) APMP.M.D-K4 (Draft A in progress)	SIM.M.D-S1 SIM.M.D-S2 SIM.M.D-S3 SIM.M.D-S4 5), 6)

EUROMET.M.D-K1	→ CCM.D-K1:	density of solids (silicon crystals)
EURAMET.M.D-K1.1	→ CCM.D-K1:	density of solids (silicon crystals)
EUROMET.M.D-K2	→ CCM.D-K2:	density of liquids (reference liquids for calibrating densitometers)
EURAMET.M.D-K2	→ CCM.D-K2:	density of liquids (reference liquids for calibrating densitometers)
SIM.M.D-K3	→ CCM.D-K3:	density of solids (stainless steel weights)
EUROMET.M.D-K4	→ CCM.D-K4:	hydrometers
EURAMET.M.D-K4	→ CCM.D-K4:	hydrometers
APMP.M.D-K4	→ CCM.D-K4:	hydrometers
SIM.M.D-K4	→ CCM.D-K4:	hydrometers



# CIPM Key Comparisons being discussed in WGD

<b>CCM.D-K5</b>	Comparison of volume measurements by optical interferometry
Status	discussed in WGD meeting 2011
Participants	NMIJ (JP), PTB (DE), NMIA (AU), KRISS (KR), NIM (CH)
<b>CCM.D-K6</b>	Comparison of density measurements by vibrating-tube densitometers
Status	discussed in WGD meeting 2011
Opinions in WGD	It may not be a capability of NMIs, may be conducted by ILAC and etc.
<b>CCM.D-K7</b>	Comparison of density measurement under high pressures and high temperatures ( $p\rho T$ properties)
Status	discussed in WGD meeting 2011
<b>CCM.D-K8</b>	Comparison of refractive index of liquids
Status	discussed in WGD meeting 2011

# Progress of absolute measurement of the density of water at PTB reported by H. Wolf

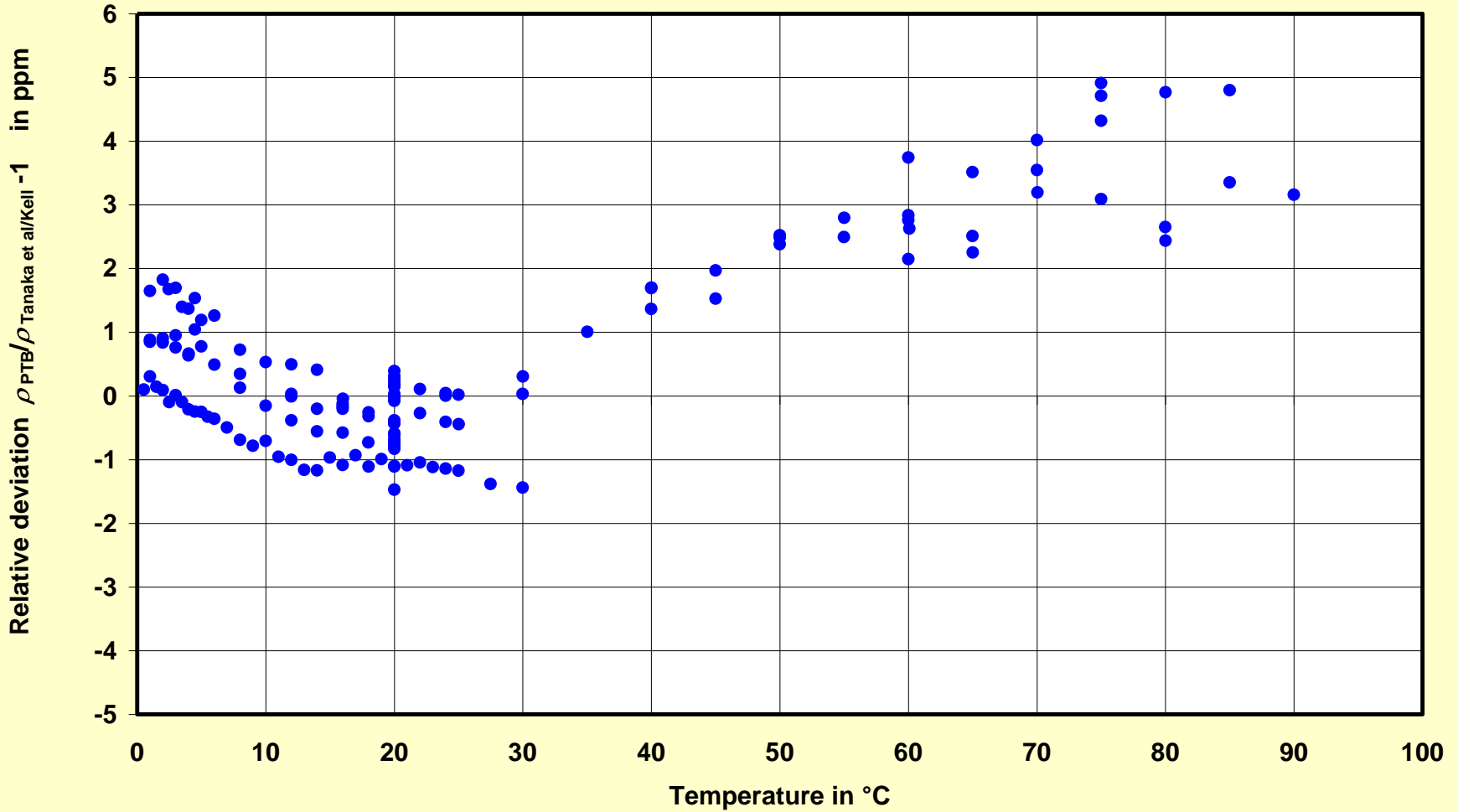


**Suspension with sinker**

**Optical height detection**

**Magnet coil**

# Results of measurements



# Summary of the Progress

- The water density given in the CIPM formulation is confirmed by the PTB measurements at a 1 ppm level.
- The NMIJ measurements of the water density at temperatures higher than 40 °C are confirmed by the PTB measurements at a 1 ppm level, too.

In a range  $t > 40$  °C:

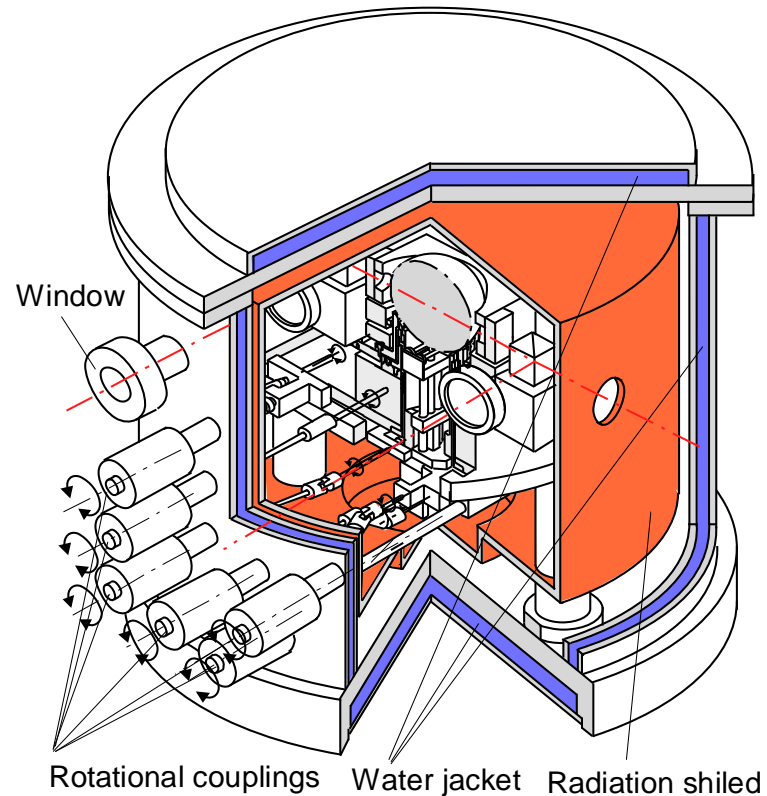
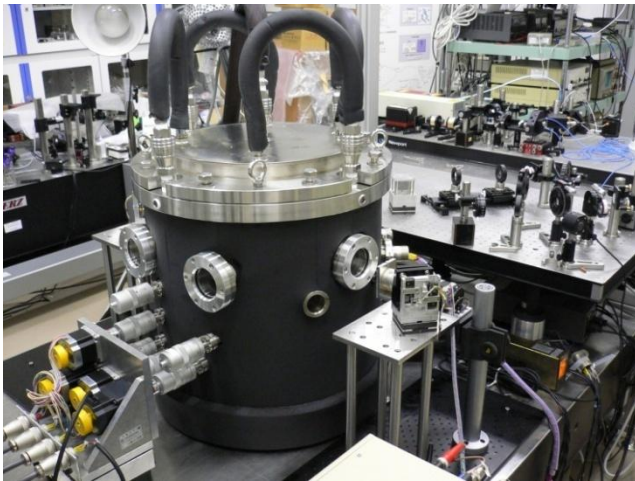
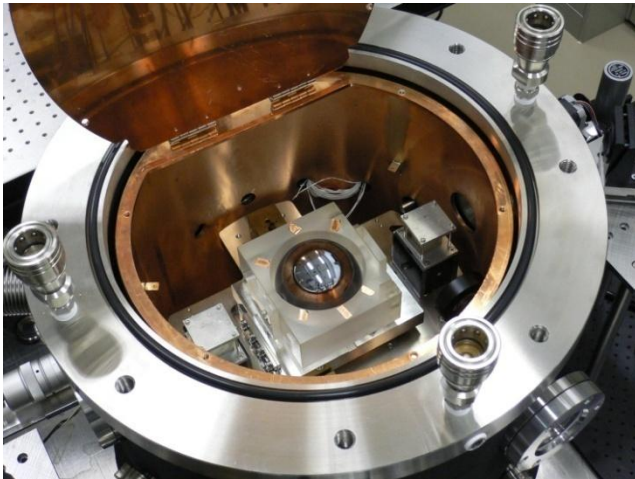
- Deviations to the Kell formulation are up to 4 ppm.
- Deviations to the IAPWS 95 formulation are up to 2 ppm.
- Measurements were completed at PTB.

# Proposed as CCM.D-K5

## Diameter and volume comparisons by optical interferometry

- At least 5 NMIs have operating sphere interferometers.
- First attempt for diameter comparisons within the International Avogadro Coordination (IAC) project completed successfully
- Equivalence in the absolute volume standards at NMIs will be evaluated.

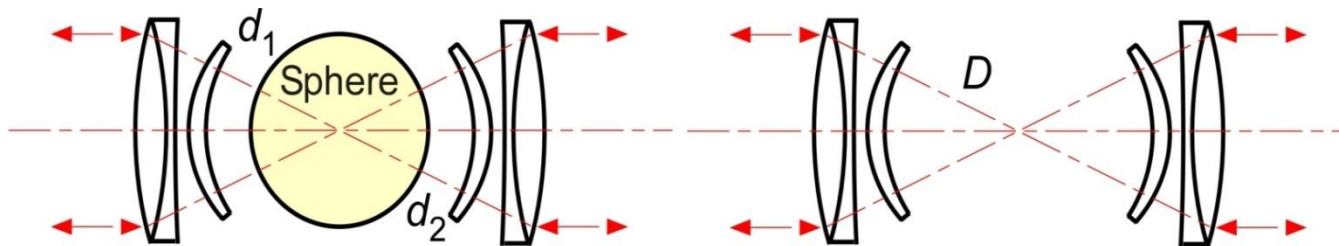
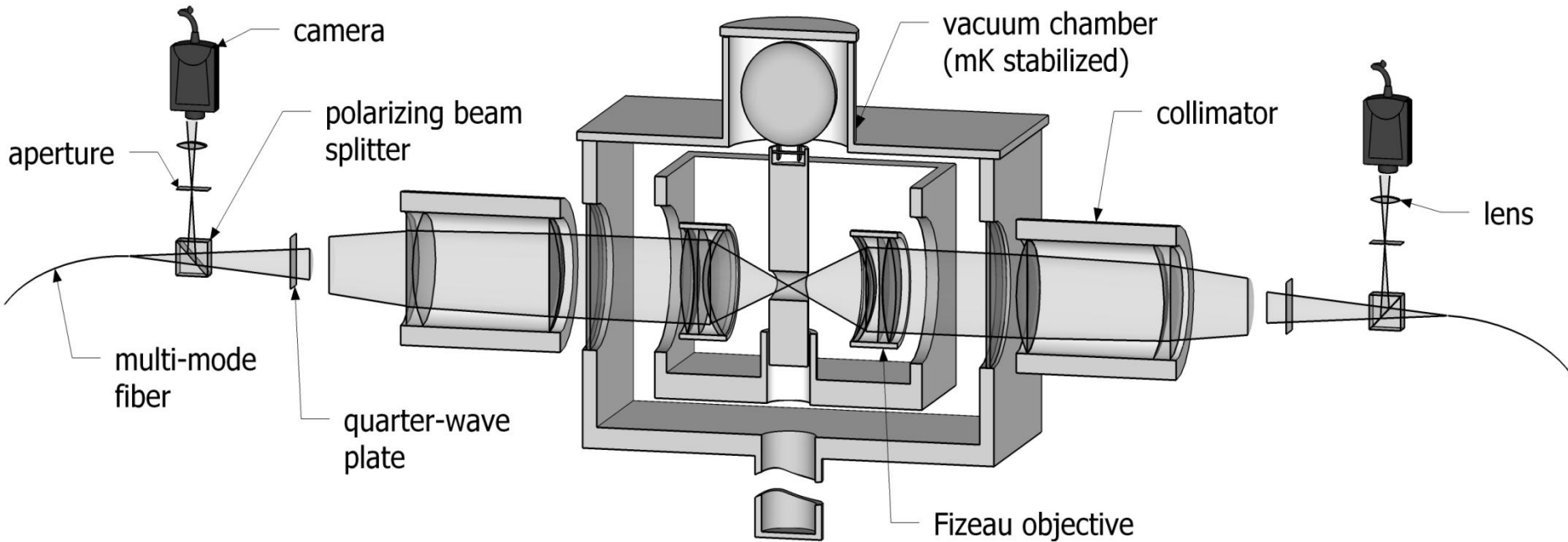
# NMIJ Interferometer with active radiation shield



**Thermal uniformity and stability < 1 mK**

N. Kuramoto, K. Fujii, and K. Yamazawa, "Volume measurements of  $^{28}\text{Si}$  spheres using an interferometer with a flat etalon to determine the Avogadro constant," *Metrologia*, 48, S83-S95, 2011

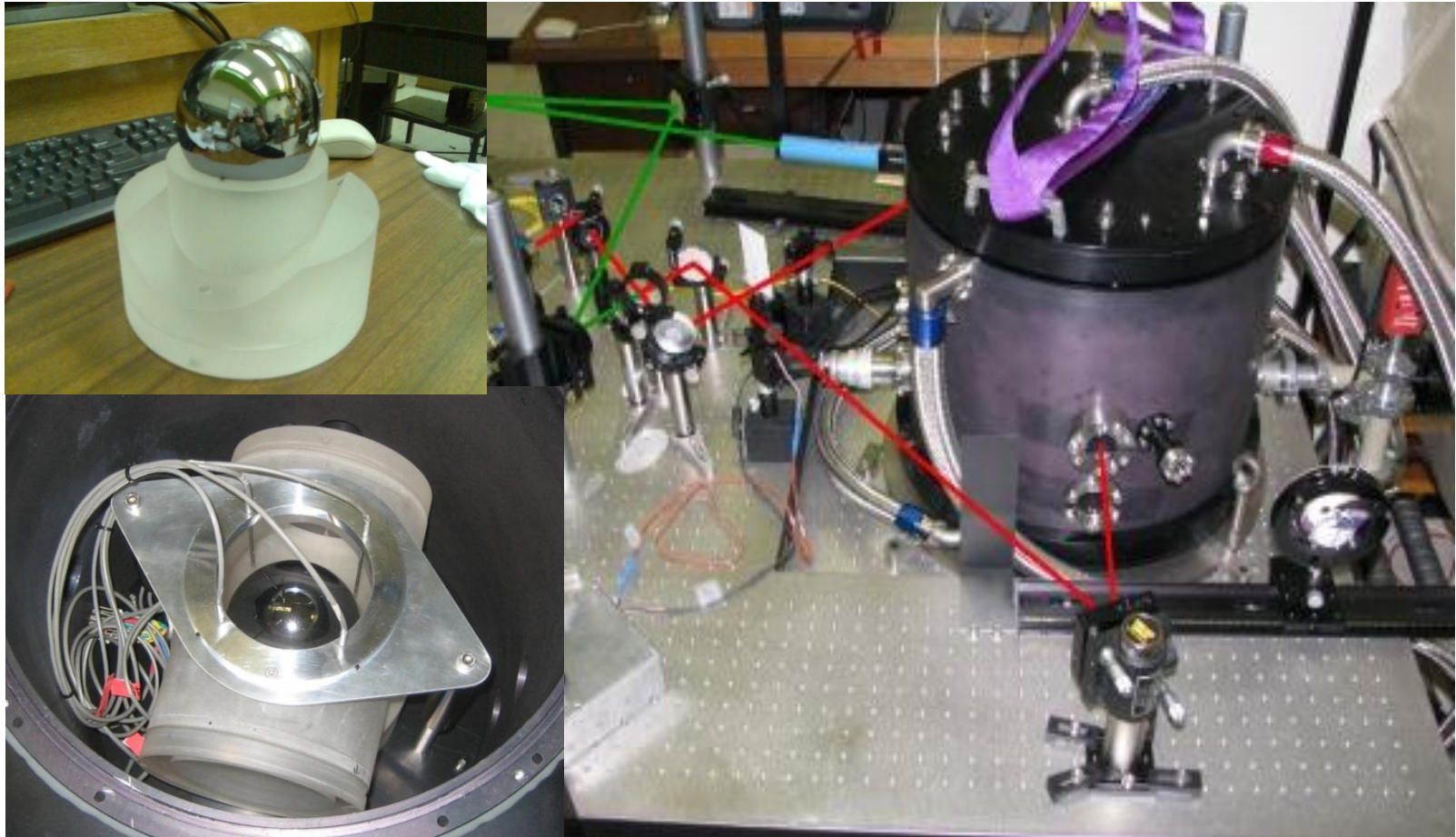
# PTB interferometer with spherical etalon



$$D = L - (d_1 + d_2)$$

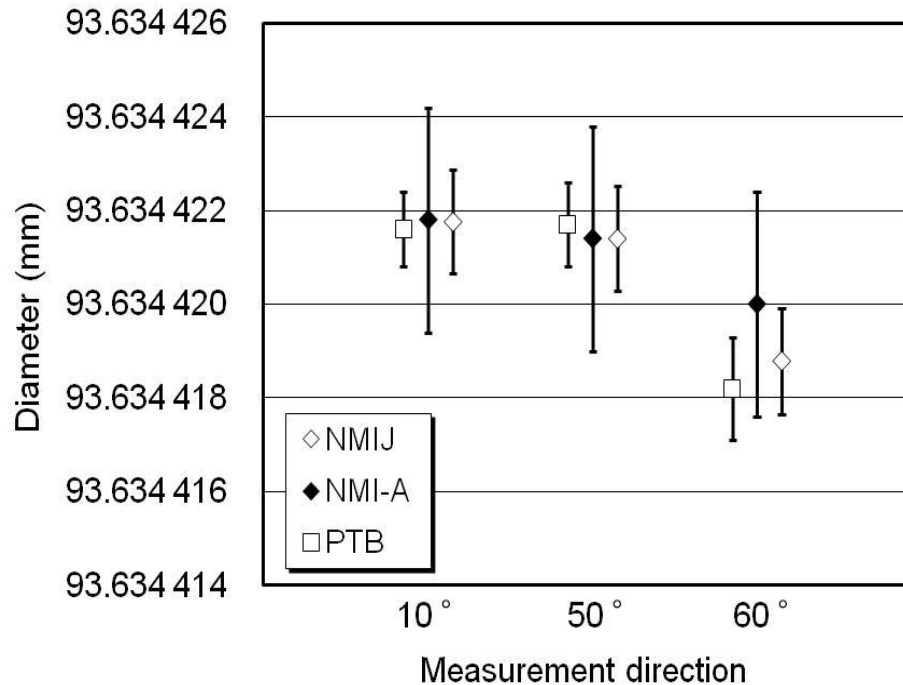


# NMIA interferometer with flat etalon





# Result of comparison



$u_c$ (observed diameter)

NMIJ : 1.1 nm

PTB : 1.0 nm

NMIA : 2.4 nm

Maximum difference

10° : 0.2 nm

50° : 0.3 nm

60° : 1.8 nm

N. Kuramoto, K. Fujii, A. Nicolaus, G. Bartl, M. Gray, P. Manson, and W. Giardini, "Diameter comparison of a silicon sphere for the international Avogadro coordination project," *IEEE Trans. Instrum. Meas.*, 60-7, 2011, pp. 2615-2620.

# Major progress since the last CCM meeting

- **CCM.D-K2: final report was submitted.**
- **CCM.D-K3: questionnaire was distributed and answers were received from 13 NMIs.**
- **CCM.D-K4: Draft A in progress**
- **In cooperation with CCM WGAC, the uncertainty of the volume measurement of silicon spheres has been reduced down to  $2 \times 10^{-8}$ , contributing to fundamental reduction of uncertainty in the density standard.**

# Technological trends and challenges

- Density measurements under high-pressures and high-temperatures (***ppT properties***) for green innovation, energy saving, and biofuel technologies (NMIJ)
  - New refrigerants for heat pump systems (low GWP, low flammability, no toxicity, no ozone-depletion, and high COP), such as HFOs
  - Natural gases and biofuels
- **Refractive index of liquids** for food industry and agriculture (mass concentration of sucrose: Brix)
  - This quantity is not density, however, both quantities are closely related with each other by the Lorentz-Lorenz equation. GUM (PL) is the only institute of possessing this CMC placed in CCL. IPQ (PT) and INRIM (IT) expressed their interest to the refractive index standards.
- Some NMIs hope to include **surface tension** as a new scope of WGD.
- Contribution to **energy, environment, food industries** will be of importance in the field of density standard.