Report on the Work Programme of the Mass Department 2009-2012 and Outlook 2013-2019

CCM February 2013

Work Packages 2009-2012

Ongoing activities

M-A1: Mass calibrations for NMIs and the BIPM (incl. volume and magnetic susceptibility determination)

M-A2: Improvement of mass metrology at 1 kg

- weighing of Si spheres (IAC)
- mass transfer between air-vacuum

M-A3: Provision of 1 kg PtIr prototypes to Member States

M-A4: Coordination activities (CCM, CCT, RMOs, OIML,...)

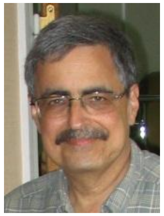
New projects

M-P1A: Maintenance of a reference facility for 1 kg comparisons under vacuum or inert atmospheres

M-P1B: Creation of ensemble of 1 kg mass standards stored in inert atmospheres (ERMS) to facilitate dissemination

Staff of Mass Department

Director of Department



Richard Davis

→ 1 Nov. 2010

Alain Picard

Present kilogram definition

Mass calibration

Ms Pauline BARAT

Principal Technician (also Avogadro project, ERMS, submultiple masses)

Volume calibration

Mrs Cécile GOYON

Assistant (80 %)

(also pressure, submultiple masses, QMS)

New kilogram definition

Watt balance

Dr Hao FANG

Principal Physicist (also magnetic susceptibility calibration)

Mr Adrien KISS

Assistant

Ensemble of reference mass standards, ERMS

Dr Estefania de MIRANDES

Since Nov. 2010

Principal physicist (also watt balance)

Mr Faraz IDREES

Since Feb. 2009

(50 %) Technician (also volume and pressure)

Mass calibrations for NMIs since 2009 - PtIr

Calibrations (Pt-Ir)		Mass	Volume
2009			
[21]	Mexico	☑	
[49]	Austria	☑	
[52] & [55]	Germany	☑	
[60] & [64]	China	☑	
[66]	Brazil	☑	
[67]	Czech Republic	☑	
[94]	Japan	☑	☑
2010			
[20] & [92]	USA	☑	
[58]	Egypt	☑	
[74]	Canada	☑	
[83]	Singapore	☑	
[95]	Kenya (from 2008 to 2010)	☑	☑
2011			
[4]	USA	☑	
[23]	Finland	☑	
[36]	Norway	☑	
[48]	Denmark	☑	
[96]	Mexico	☑	☑
2012			
[57]	India	☑	
[72]	Rep. of Korea	☑	
[79] & [85]	USA	☑	

24 NP calibrated

Mass calibrations for NMIs since 2009 - st. st.

Calibrations (Stainless steel)

2009

		Mass	Volume	Mag
1 kg	INMETRO, Brazil	☑		
2 x 1 kg	NML-SIRIM, Malaysia	☑	☑(1)	☑(1)
2 x 1 kg	VSL, Netherlands	☑		☑
1 kg	NMC A*STAR, Singapore	☑		
1 kg	CEM, Spain	☑		

2010

		Mass	Volume	Mag
1 kg	BIM, Bulgaria	☑		
2 x 1 kg	NIS, Egypt	☑		
3 x 1 kg	KazInMetr, Kazakhstan	☑		
2 x 1 kg	VSL, Netherlands	☑		
3 x 1 kg	MSL, New Zealand	☑		
1 kg	NMC A*STAR, Singapore	☑		
3 x 1 kg	NIMT, Thailand	☑		

2011

		Mass	Volume	Mag
2 x 1 kg	VSL, Netherlands	☑		
2 x 1 kg	JV, Norway	☑		
1 kg	LATU, Uruguay	☑	☑	☑
3 x 1 kg	NMC A*STAR, Singapore	☑	☑	☑

2012

		Mass	Volume	Mag
2 x 1 kg	INTI, Argentina	☑		
1 kg	SMD, Belgium	☑		☑
1 kg	CESMEC, Chili	☑		☑
4 x 1 kg	HMI, Croatia	☑	☑(1)	☑
2 x 1 kg	EIM, Greece	☑		
3 x 1 kg	KIM-LIPI, Indonesia	☑		☑(1)
2 x 1 kg	VSL, Netherlands	☑		
1 kg	CEM, Spain	☑		

46 MS calibrated

Volume magnetic susceptibility

1 susceptibility standard

2 susceptometers (4 calibrated standards)

1 test mass for LISA pathfinder

2 comparisons (EURAMET & SIM)

Calibrations for the BIPM since 2009

Mass

Mass department
Chemistry department
IR department
Watt balance
> 300 standards

Pressure

13 calibration campaigns (101 certificates issued)
and 15 manometer verifications

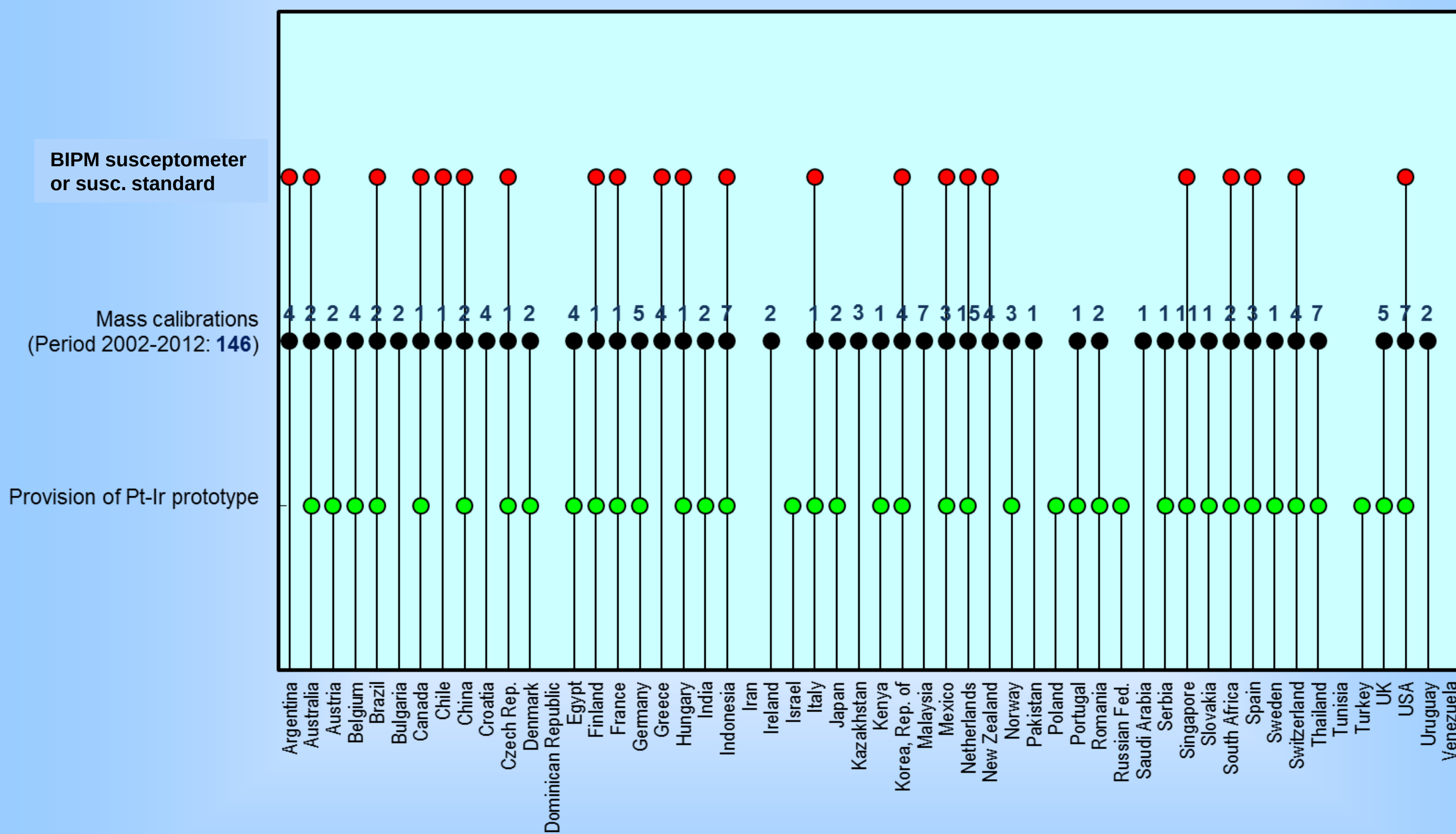
Volume

1 kg	Pt-Ir stack of discs (ERMS)
1 kg	st-st stack of discs (ERMS)
4 x 1 kg	Pt-Ir prototypes (ERMS)
4 x 1 kg	st-st mass standards (ERMS)
1 g & 95 mg	Sensitivity weights of M-one
50 g, 2 x 20 g, 10g	RD1
10 g	AP2
5 g, 2 x 2 g, 2 x 1 g	ZW3
2 x 50 g, 2 x 25 g	BIPM Watt Balance

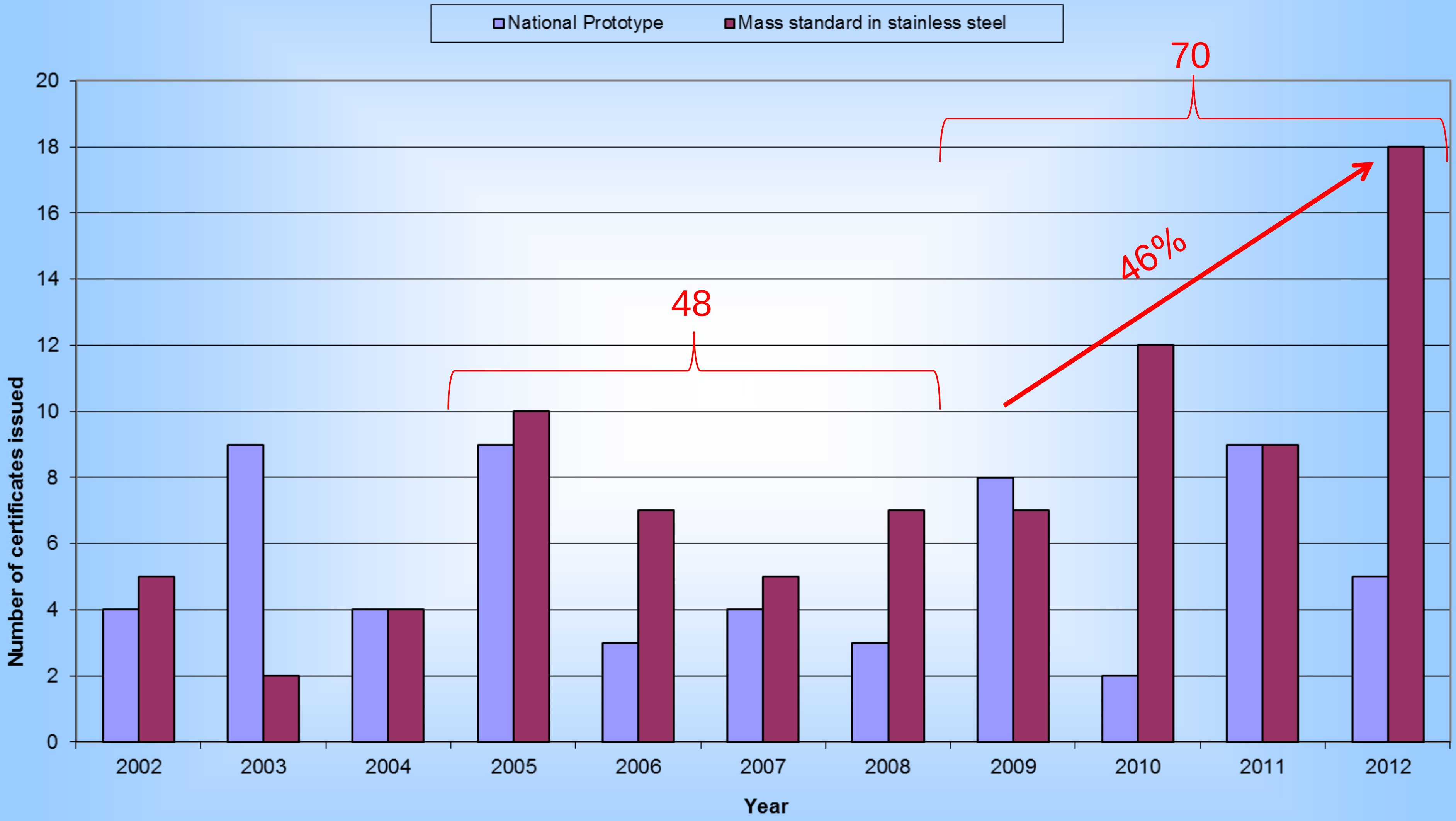
Fabrication of PtIr standards

For Mexico	1 kg prototype
For the ERMS	1 kg stack of discs
	4 x 1 kg prototype
For BIPM working standard	2 x 1 kg prototype, to replace two degraded prototypes

Services provided by the Mass Department to Member States



Number of calibration certificates



Improvement of mass metrology at 1 kg

Renovation of Room 104 (Metrotec balance for mass calibration services)

Four comparisons using all BIPM working standards
to verify all the calibration chain

Improvement of mass metrology at 1 kg

New Mettler Toledo M-one mass comparator
- air and vacuum measurements
- will also be used for mass calibrations in future

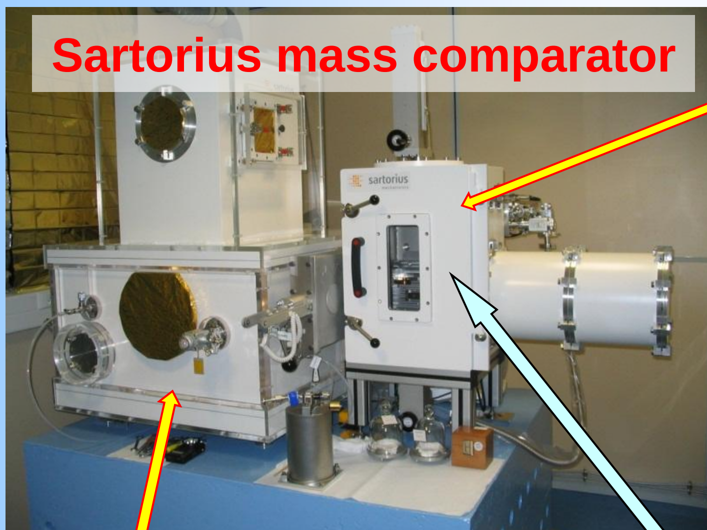
- vacuum capability
- std dev. 0.1 µg
- 6 positions
- load lock

Commissioning
is completed in air and
under vacuum

Next step:
integration in QMS

1 kg comparison facility for vacuum and air

research:
- International Avogadro Coordination
- CCM WGM TG1 (air-vacuum transfer)



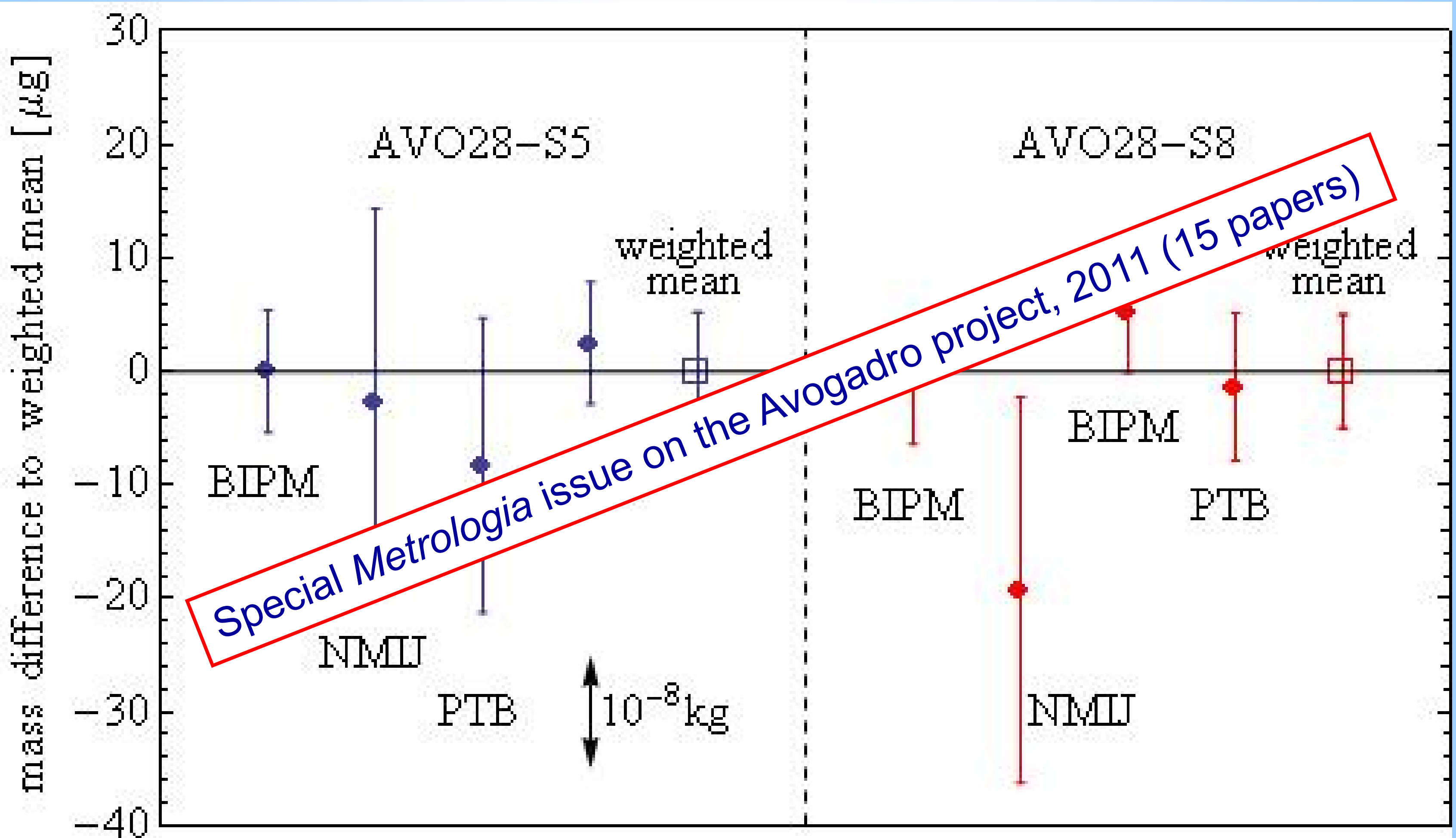
Vacuum Transfer System (VTS)

Mass comparator

Standard inert gas glove box equipped with vacuum oven

Automatic loadable vacuum container

Mass comparison under vacuum on the ^{28}Si spheres S5 & S8



$u_r(S5) = u_r(S8) = 4.0 \times 10^{-9}$

Trilateral cooperation between BIPM, NPL and METAS

- to provide to the BIPM support (due to lack of resources and budget) for the preparation of the *mise en pratique* of the new definition of the kg

- The cooperation was based on the initial 2009-2012 PoW

- NPL provided a scientific co-operation through one physicist, working at the NPL at the level of 60% per year

- The contribution from METAS was carried out as required, by one physicist at the level of 50% per year

Objectives of the cooperation

- Study methodology of air-vacuum mass comparisons using surface artefacts
- Surface contamination analysis on samples of different materials (XPS, CAM-Contact Angle Measurement)
- Develop mass transport containers suitable for different atmospheres (vacuum or inert atmosphere)
- Effectiveness of cleaning methods (BIPM, UVOx, solvents and hydrogen plasma)

A lot of experience has been accumulated which will be useful for the future *mise en pratique*

In future we look for cooperation in the field of surface analysis:

- optimize the storage conditions in the ERMS
- optimize cleaning technique

History of BIPM mass standards - I

A general method to reproduce the mass values assigned to BIPM working standards from 1889 to 2010

Starting point: available mass **differences** among 18 BIPM 1kg prototypes measured from 1889 to 2010.

Objective: re-analyze these data and deduce the **absolute mass** values over time of the involved prototypes.

• The results of this new determination of the absolute masses of the prototypes are compared to the absolute masses **historically assigned** by the BIPM to those prototypes.

• Notice that during the period **1992-2010** no direct mass measurement against **IPK** is available.

Rationale: differences between WGM TG2 study and historical BIPM values.

History of BIPM mass standards - II

Model to determine absolute masses from mass differences

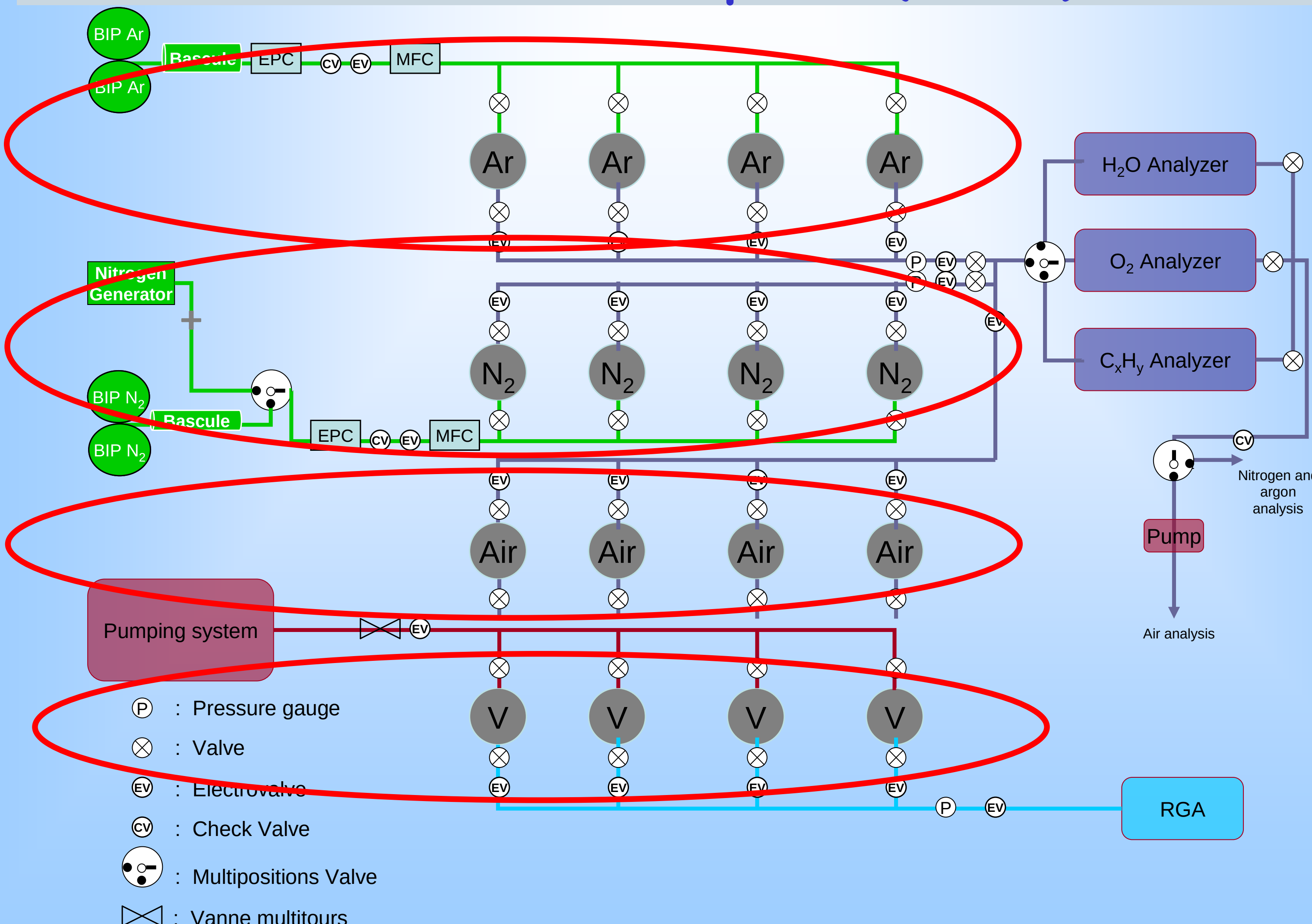
136 *series* of mass comparisons distributed from 1889 to 2010.

- Each *series* comprises mass differences among 2-11 prototypes.
- Cleaning times of each prototype are known.
- In order to compute absolute mass values starting from mass differences, **one additional hypothesis** equation needs to be added to the system.
- Our model tests all possible reasonable hypotheses. Each hypothesis leads to a set of absolute mass values. The model chooses the “best" absolute mass values by a weighted mean of the sets.

The absolute mass values assigned historically by the BIPM to the prototypes could be confirmed (within several micrograms on average)

Work was presented to CCM WGM TG2, to be published in *Metrologia* later 2013

Creation of ensemble of 1 kg mass standards stored in inert atmosphere (ERMS)



Status of standards belonging to ERMS

The ERMS will be constituted by:

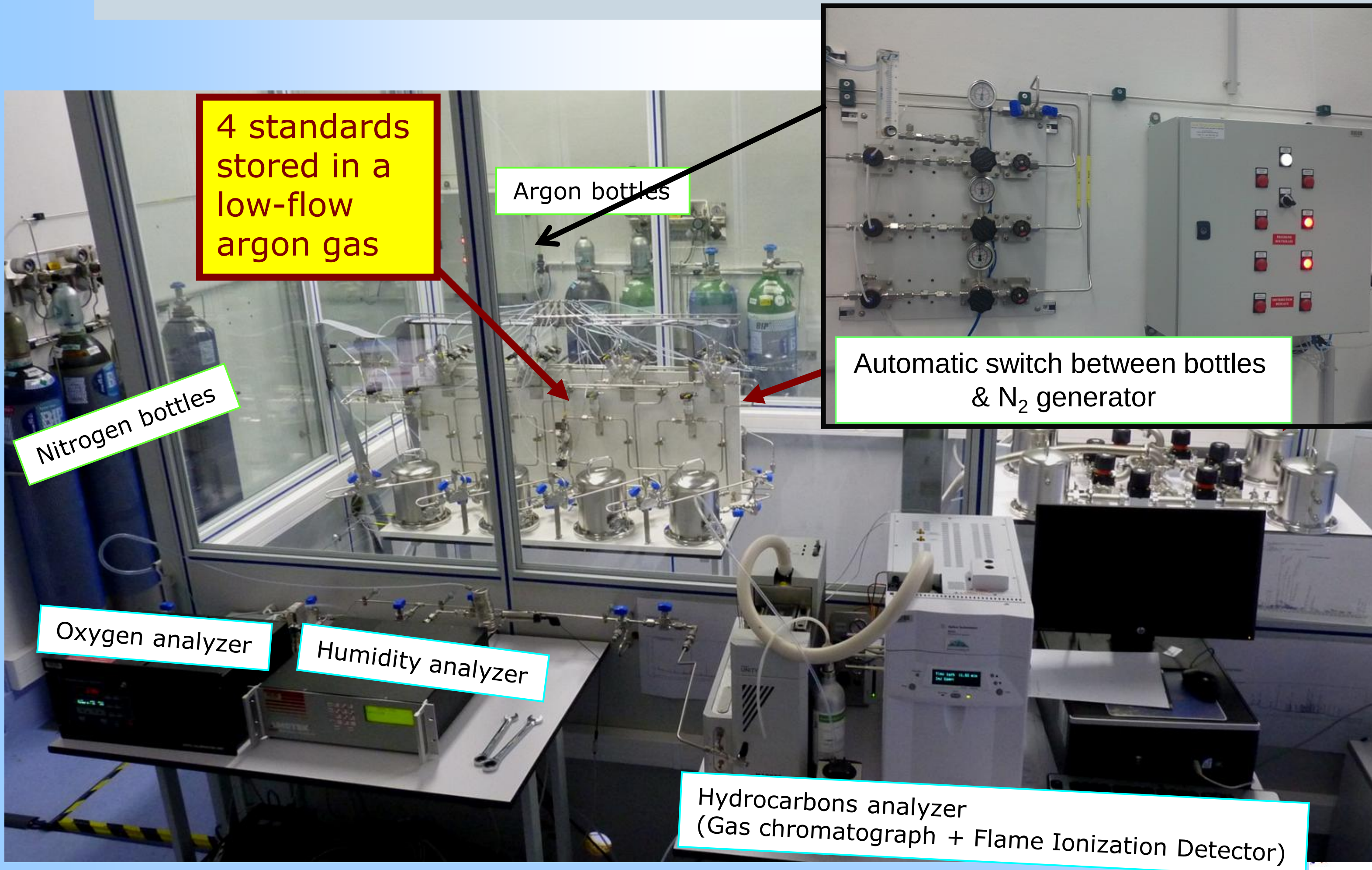
	Manufacture	Mass	Volume	surface characterization
- 4 x 1 kg prototypes	☑	☑	☑	☐
- 4 x 1 kg st-st standards	☑	☑	☑	☐
- 4 x 1 kg silicon spheres	☑ (3 of 4)	☐ (postponed)	☑ (2 spheres done at NMIJ & 1 sphere done at PTB)	☑ (one done at the PTB)

Surfaces artefacts (sorption artefacts)

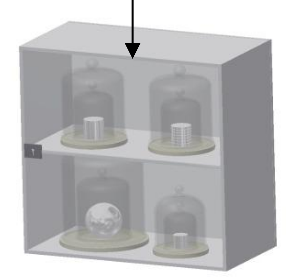
	Manufacture	Mass	Volume	surface characterization
- 1 x 1 kg Pt-Ir stack of discs	☑	☐(postponed)	☑	☐
- 2 x 1 kg st-st stack of discs	☑	☐(postponed)	☑	☐
- 1 x 1 kg silicon stack of discs		(not yet ordered)		

Standards and samples for surface analysis will be placed in the storage network later this year

Laboratory for the BIPM ensemble of mass standards

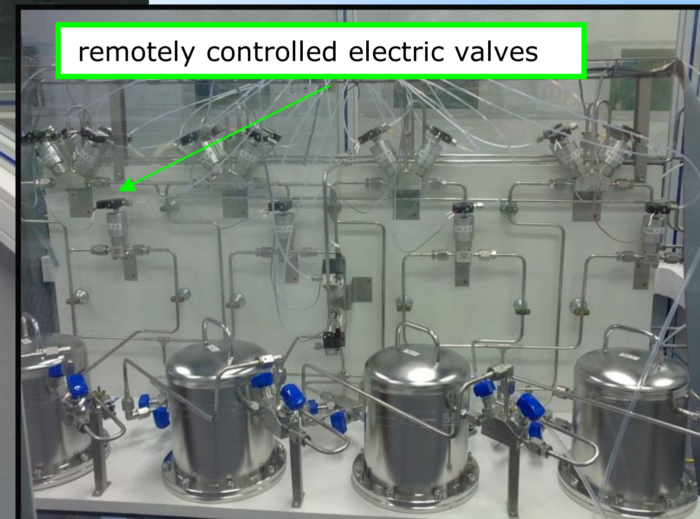


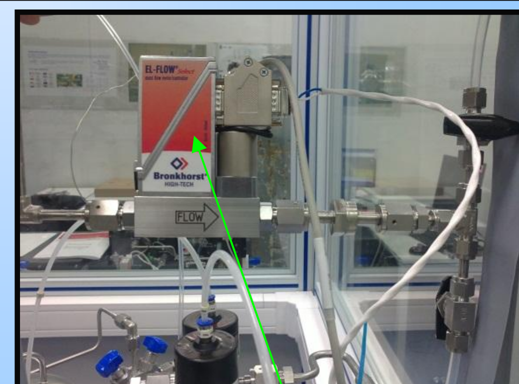
In preparation: 4 standards in air, to be kept inside the cabin



Temperature, humidity and pressure are continuously monitored within the cabin.
Outside the cabin, temperature is actively controlled.

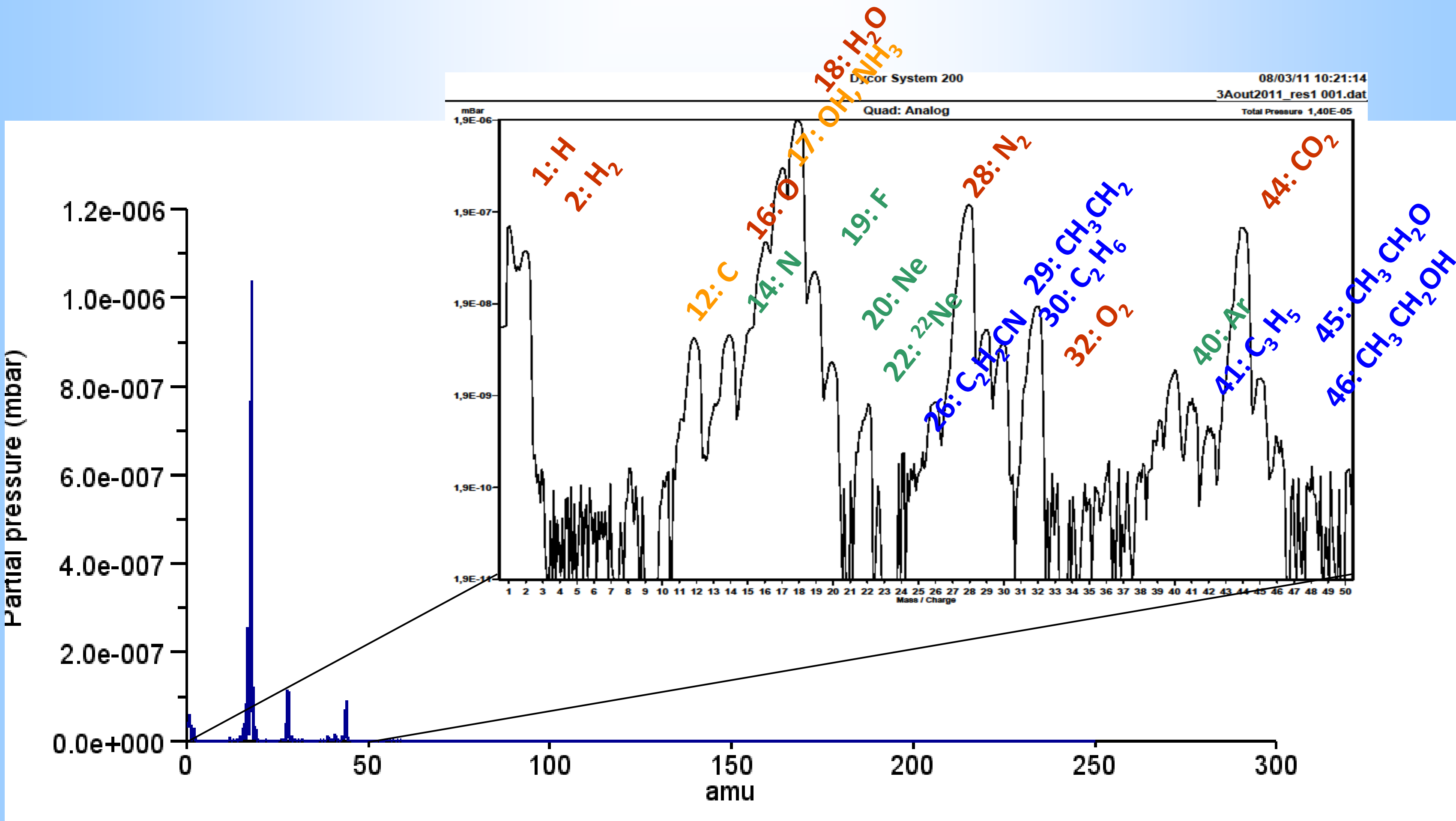
Gas storage network





- A continuous gas flow (argon (left) and nitrogen (right) is maintained through all the containers.
- The gas network is fully automated: 28 remotely controlled electric valves and 2 gas flow-meters allow to choose to analyze the gas passing through one container at a time while ensuring an adequate gas flow through all the others.
- Gas containers are equipped with manual isolation valves which allow each container to be removed and put back into the network without altering the storage conditions of the standard inside.
- Gas storage containers have a bypass tube which allows the tubes of the network to be purged without flowing the purge gas through the container.

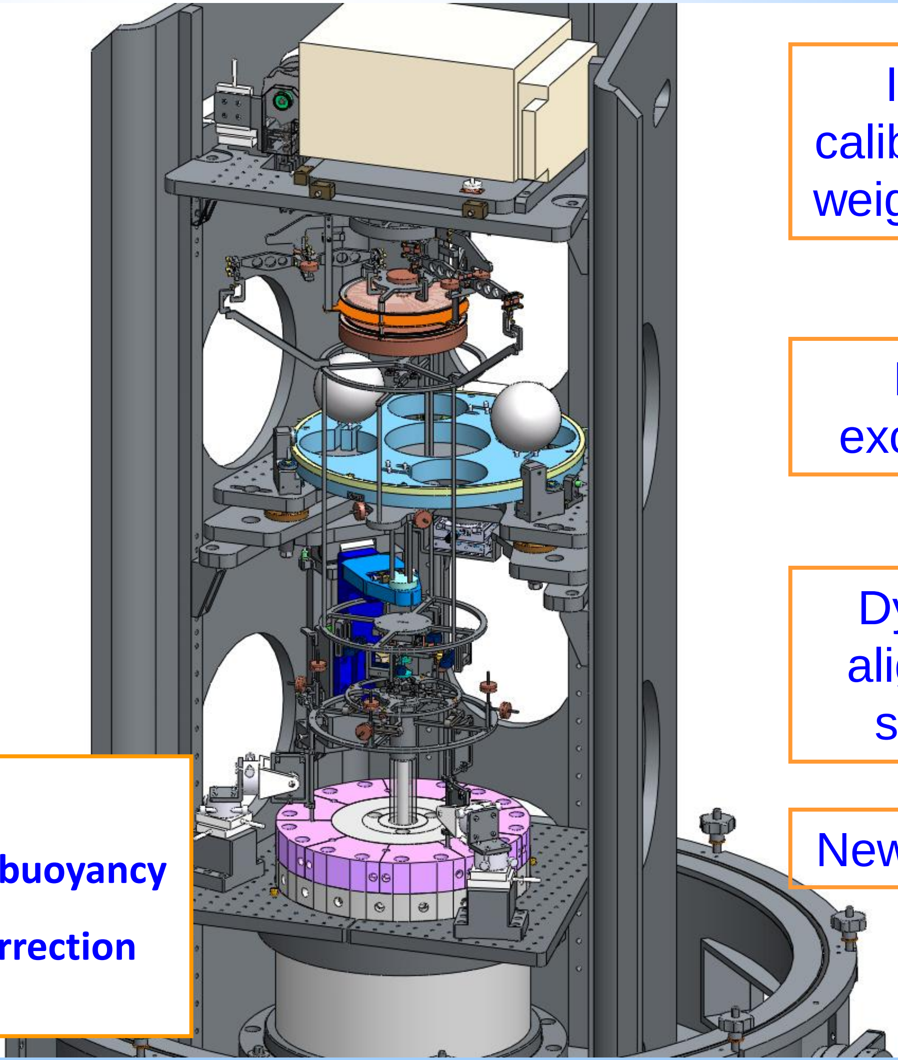
A typical scan of our RGA



Watt balance: Current status

- Current apparatus installed inside vacuum chamber in the new laboratory
- First measurements being made to evaluate improvement due to noise reduction

Watt balance: future developments



In-situ calibration of weighing cell

Mass exchanger

Dynamic alignment system

New magnet

vacuum: < 0,01 Pa

no air convection; no air buoyancy

no air refractive index correction

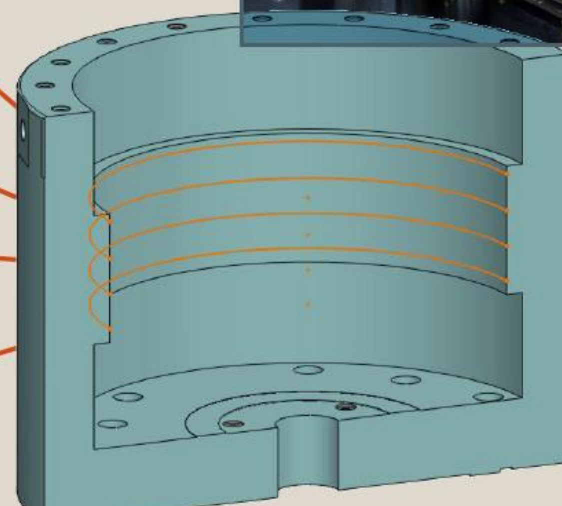
Watt balance: magnet fabrication

Diameter size results

CMM measurements of the size of the Ø263 mm diameter ID show less than 2.2 µm variation.

This is the largest source of error and contributes 1.1 µm to cylindricity.

Diameter large ID Top	263.003
Diameter large ID Upper Mid	263.003
Diameter large ID Lower Mid	263.004
Diameter large ID Bottom	263.005



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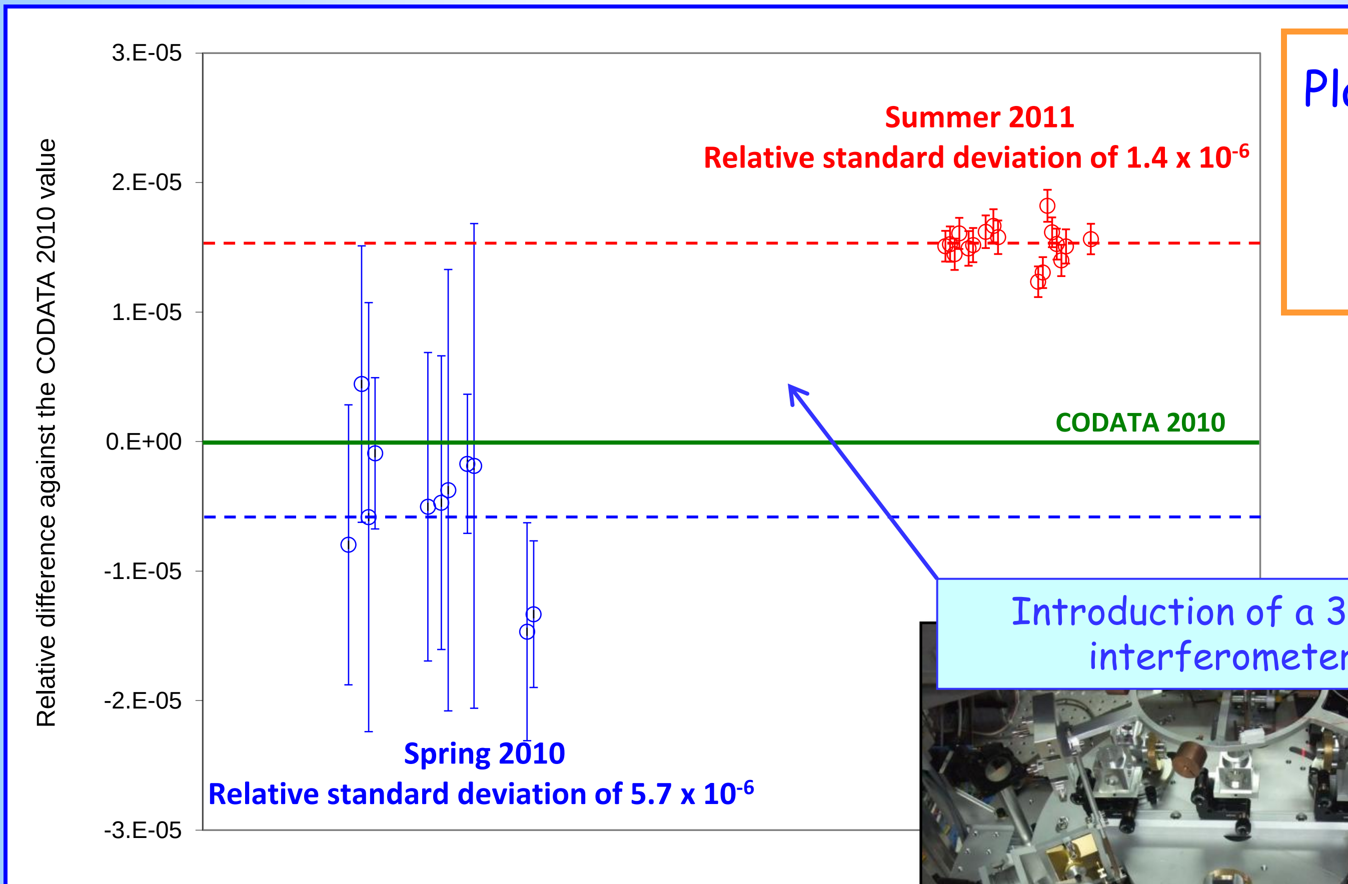
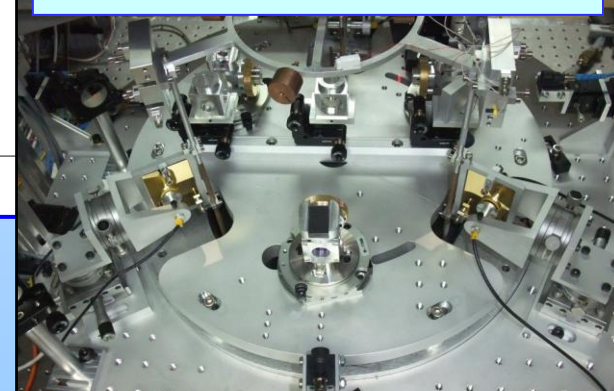
Watt balance: Planck constant measurements



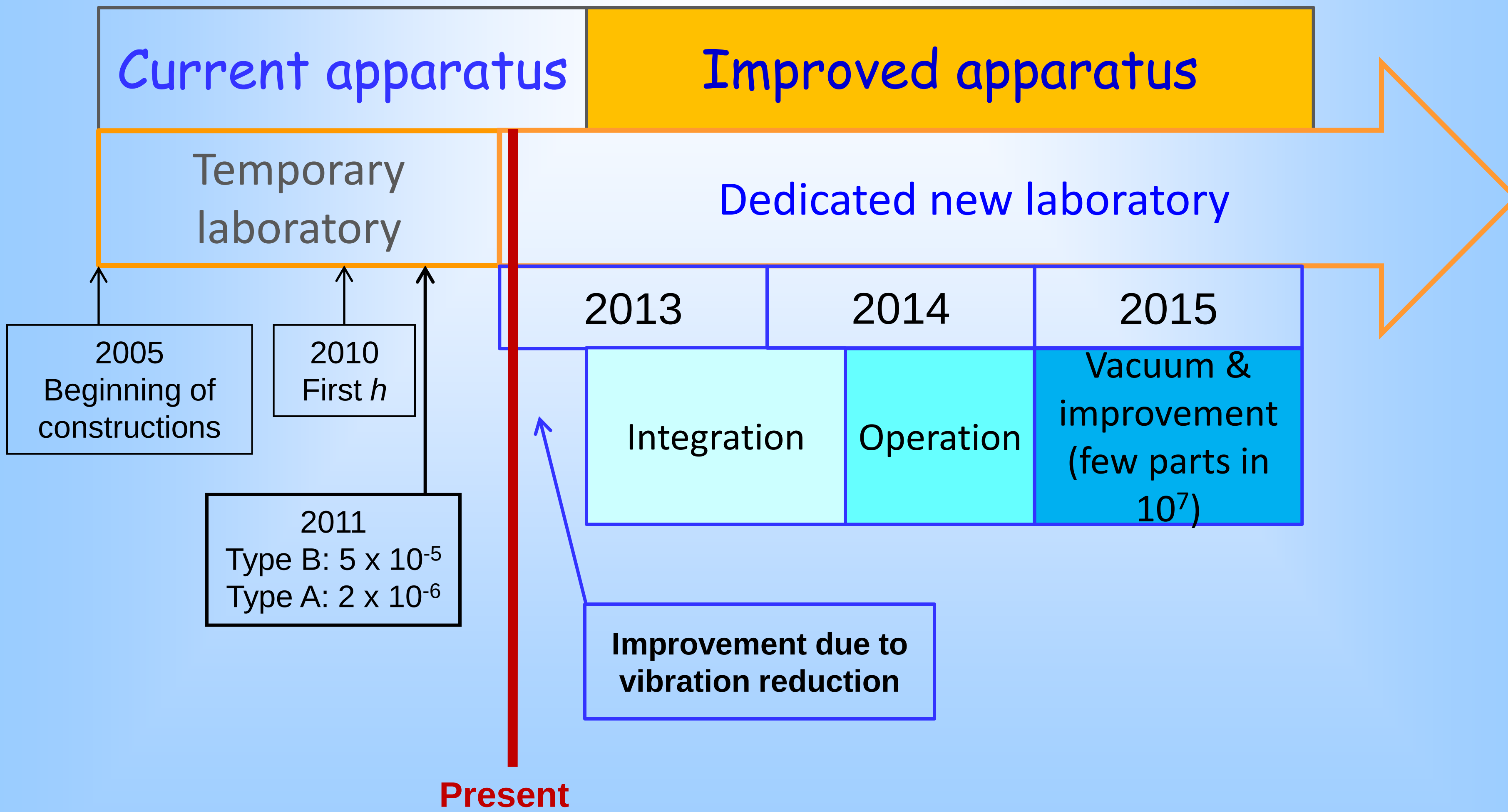
Planck constant *h*

- Type B: 10^{-5}
- Type A: 10^{-6}

Introduction of a 3-axis interferometer



Watt balance: Work plan 2013-2015



Coordination activities

CCM

CCM meetings (May 2010, May 2011)

- 10 WGs and 2 TGs
- **Draft of the MeP kg in the frame of the WG SI**
- Follow up of the requests from Chairs of WGs

WS on MeP kg (Novembre 2012)

- Draft report has been distributed

CCT

CCT meetings (May 2010 and May 2012)

- 10 WGs and 2 TGs:
- Draft of the MeP K in the frame of WG1 TG MeP-K
- Follow up of the requests from Chairs of WGs.

EMRP SI Broader scope

- SIB-03 Realization of the awaited definition of the kilogram - resolving the discrepancies
- SIB-05 Dissemination of the new kg

CCM.M-K4

- piloted by BIPM
- Four petals (2 x 1 kg in stainless steel per petal)
- 16 participants
- Participant measurements completed
- 16 reports received, Draft A planned for March/April 2013

Outlook PoW (2013-2015), approved

Continued activities

• **Provision of prototypes to Member States**	
• **Mass & volume calibrations**	PtIr and st. st. standards for Member States
• **Ensemble of mass standards**	- optimization of storage conditions - investigation of cleaning techniques, if necessary - monitoring of masses
• **Watt balance**	continue development towards $u_r < 5 \times 10^{-8}$
• **Internal calibration services**	pressure, volume, mass
• **Avogadro project**	re-determine mass of spheres AVO28-S5 & -S8
• **EMRP**	-SIB-03 (KNOW) Realization of the awaited definition of the kilogram - resolving the discrepancies -SIB-05 (NewKilo) Dissemination of the new kg
• **Coordination activities**	CCM, CCT, RMOs, ...

Outlook PoW (2013-2015), approved

New activities

- **Extraordinary calibrations against the IPK (~15 months, 1-2 persons)**
  - no "normal" calibrations for M.S. during this period
- **BIPM key comparison of primary realizations of the kg**
  - within the framework of CIPM MRA
  - just before redefinition
  - to be repeated, if necessary, after 5-10 years
  - ongoing
- **Significant increase of requests for mass calibrations ??**

Stopped activities

- **Magnetic susceptibility calibration**
- **Provision of susceptometers**

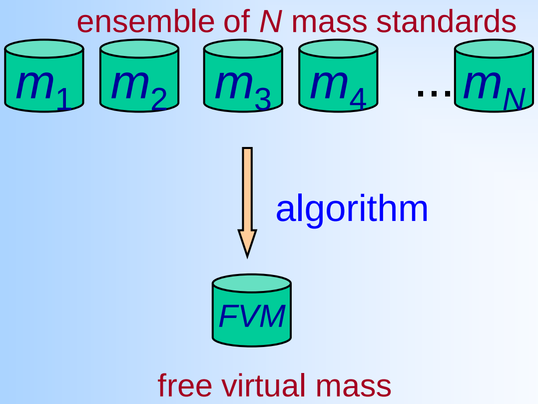
Long-term outlook PoW (>2015)

- **provision of prototypes**
- **mass calibrations (using ERMS after redefinition) in air**
- **new calibration service for masses under vacuum**
- **finish development of watt balance and achieve u_r < 5 x 10^{-8}**
  - realization of kg
  - calibration of masses of ERMS
- **continue the ongoing KC of primary realizations**
- **repeat KC of all existing primary realizations, if necessary**
- **ERMS**
  - continue to monitor storage conditions
  - monitor surface contamination, in cooperation with NMIs, to be developed
  - monitor mass changes
  - link surface contamination (if any) to mass changes
- **Coordination (CCM, CCT, RMOs,...)**

Thank you !

Algorithm for the Ensemble of Mass Standards

Purpose of the algorithm: it uses as input data the mass differences (m_i-m_j) between the elements of the ensemble and calculates a free virtual mass (*FVM*) optimized to have a stability and a robustness superior to those of any individual element of the ensemble.



$$m_{\mathrm{FVM}}(t) = \sum_{i=1}^{N} \omega_i(t)\left[m_i(t) - \delta_{i,\mathrm{pred}}(t)\right]$$

with $$\sum_{i=1}^{N} \omega_i(t) = 1$$

• The two key decisions to take are the choice of the best ***weighting scheme*** and ***prediction method*** to optimize both the stability and robustness of the free virtual mass.

the prediction of the quantity

$$\delta_i(t) = \left[m_i(t) - m_{\mathrm{FVM}}(t)\right]$$

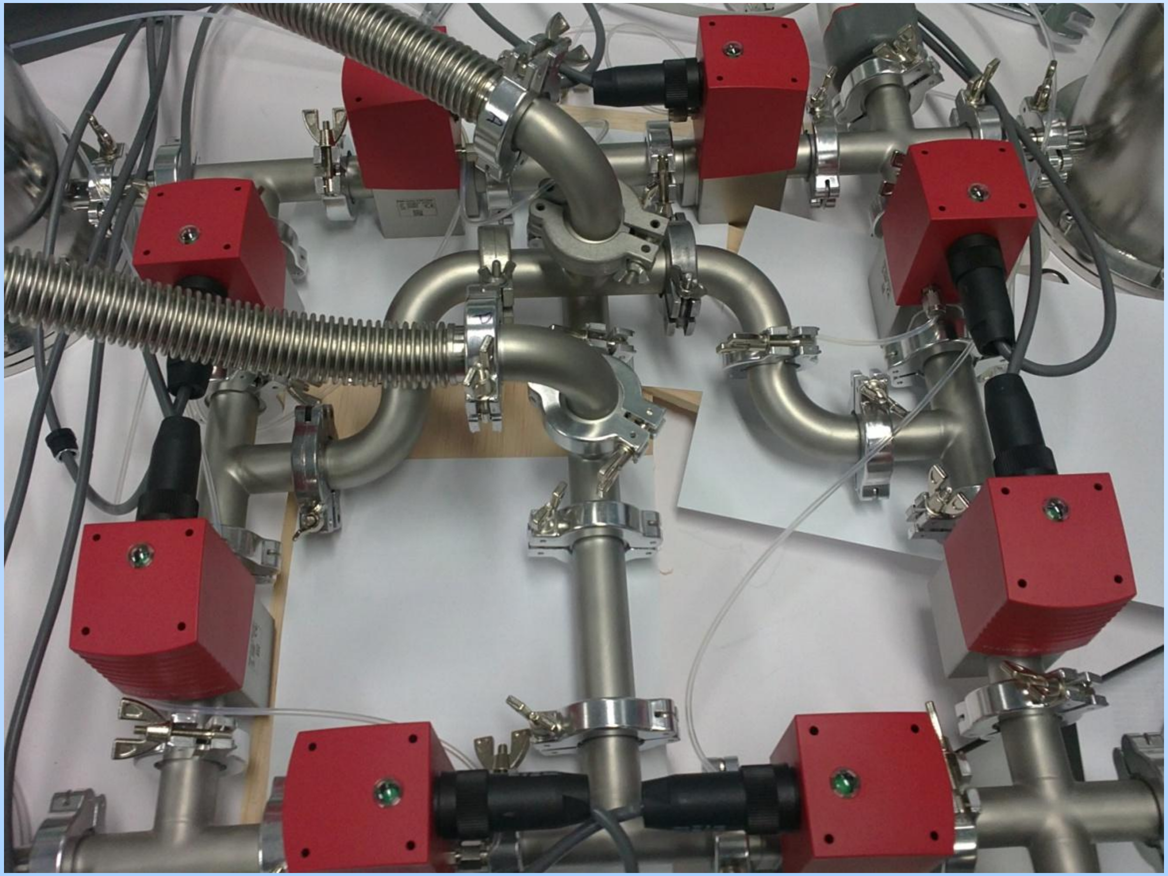
of the *i*'th element at the time *t*.

Optimal weighting scheme $$\omega_i(t) = \frac{1/\sigma_i^2(t)}{\sum_{i=1}^{N} 1/\sigma_i^2(t)}$$

• We suggest: $\omega_{\max} = \frac{C}{N}$ → numerical constant which needs to be chosen (1.5) → total number of standards

• Several iterations are needed to let the statistical weights ω_i converge to the optimal value.

Towards a second version of the vacuum network



Manual isolation valves have been changed successfully to computer controlled isolation valves

Towards a second version of the vacuum network

Containers compatible with our VTS are fabricated at BIPM workshop

Calibrations for the BIPM since 2009

Mass calibrations for internal use

Mass Department

Mass determination of the BIPM working standards in Pt-Ir determined in **April 2009, December 2009, in June 2011 and July 2012** with respect to prototype **No. 25** and its check standard, **No. 73**, both reserved for special use.

Routine mass comparisons of BIPM working standards in PtIr and st-st carried out **8 and 14 times respectively**.

1 kg	[841] Pt-Ir mass standard (2009, 2011 & 2012)
900 g	900ZW St-St standard (2009)
100 to 500 g	ZWE1 & ZWE2 St-St sets (2009, 2010, 2011 & 2012)
100 to 500 g	ZW1 & ZW2 St-St sets (2009, 2011 & 2012)
100 to 500 g	DHI set of pressure balance (2009 & 2011)
100 to 500 g	Pt1 Pt-Ir set (2009 & 2012)
2 x 50 g	St-St standards for BIPM Watt balance (2009)
2 x 25 g	St-St standards for BIPM Watt balance (2010)
1 to 5 g	ZW3 St-St set (2009 & 2011)
1 g & 2 x 2 g	St-St standards for BIPM Watt balance (2012)
10 mg to 5 g	RD1 (2009, 2010, 2011 & 2012)
2 x 1 g, 100 mg, 96 mg, 95 mg	Sensitivity weights (2009 & 2011)

Chemistry Department

2 g & 200 g (2010 & 2012)

IR Department

36 g (2012)