

# Summary of updates of realization & mass dissemination from NMIs

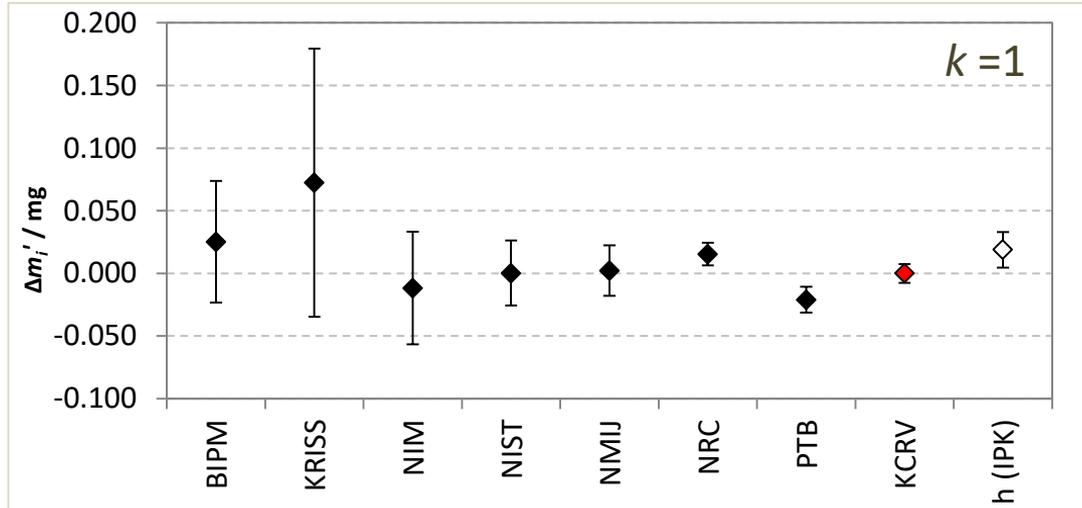
Richard Green

18<sup>th</sup> CCM meeting, 20-21 May 2021

**Bureau**  
↑ **International des**  
↓ **Poids et**  
↓ **Mesures**



# BIPM Kibble balance: measurement & improvement



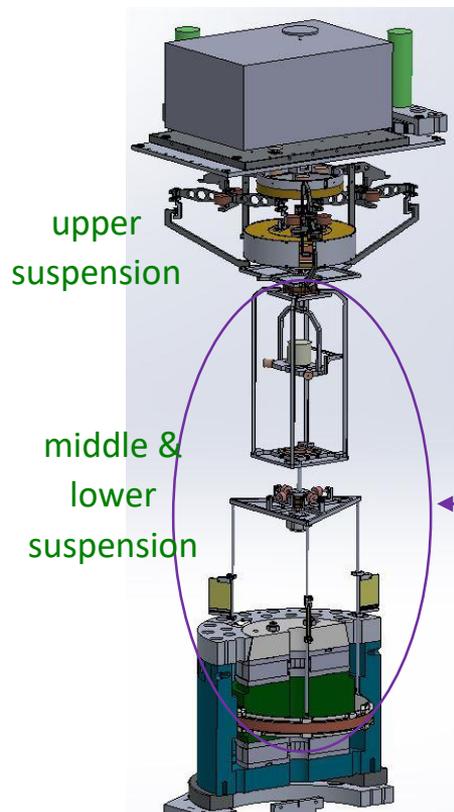
## Measurements in 2019

- participation in CCM.M-K8.2019
- *Metrologia* paper
- standard uncertainty 49  $\mu\text{g}$   
limited by parasitic coil motion & voltage measurement (electrical grounding)

## Various improvements in 2020

- Upgrade of laser frequency servo-control and of various electronics for position sensors
- automation & refinement of data processing programs
- investigation & improvement of electrical circuit → further investigation is required

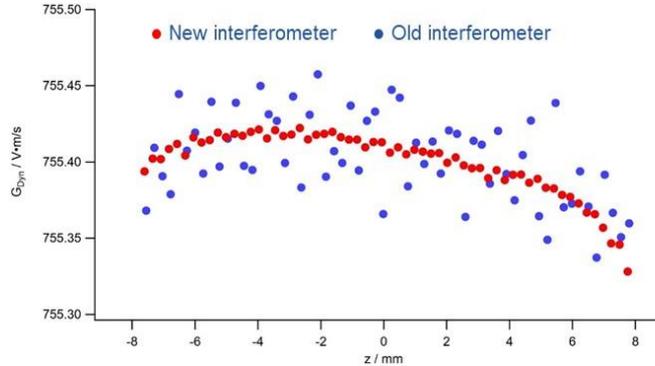
# BIPM Kibble balance: towards Mark II apparatus



- STEP I: new middle & lower suspension
  - much stiffer and more easily adjustable mechanics
  - new bifilar coil having a larger wire length
  - operation in vacuum confirmed
- STEP II: replacing upper suspension (motor + 3 levers for generating the coil vertical motion) → minimize unwanted coil motion (reduce type B uncertainty)
  - replacing commercial weighing cell
  - a first beam balance prototype being fabricated

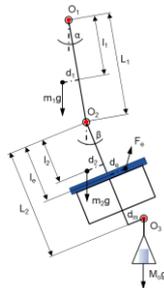
# Main improvements

- Significant noise reduction in the dynamic phase

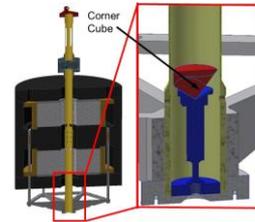


- Redesign of the interferometer physically attached to the magnet
- Mean of the fit residuals on one trajectory  $\sim 5$  ppm

- Alignment stability / Abbe error



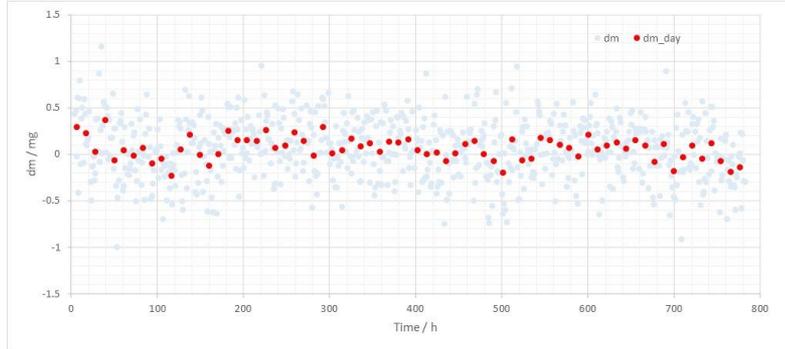
- $u_{\text{Alignment}} < 10$  ppb



- $u_{\text{Abbe}} < 10$  ppb

## Preliminary results / Next steps

- ~ 30 days measurement under vacuum

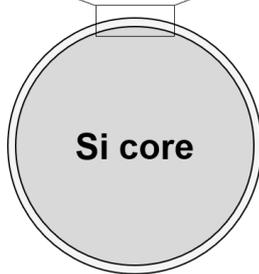
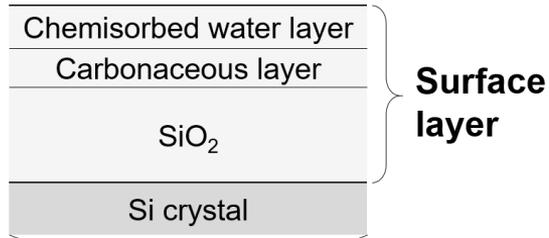


- $dm = 60 \text{ ug}$
- $std = 2 \text{ ug}$

- Complete realignment of the whole experiment
- Two campaigns with two different masses
  - Stainless steel
  - Gold plated copper

# Realization of the new kilogram at NMIJ by the XRCD method using AVO28-S5c

## Principle of the XRCD method



AVO28-S5c

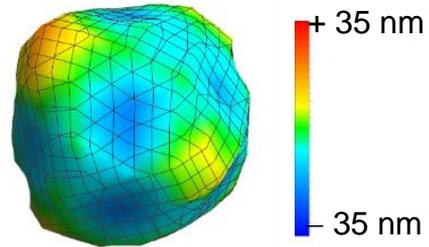
Surface model of the <sup>28</sup>Si-enriched sphere AVO28-S5c

$$m_{\text{sphere}} = \underbrace{\frac{2hR_{\infty}}{c\alpha^2} \frac{A_r(\text{Si})}{A_r(\text{e})} \frac{8V_{\text{core}}}{a^3}}_{\text{Si core}} - m_{\text{deficit}} + \underbrace{m_{\text{SL}}}_{\text{Surface layer}}$$

- Parameters measured for CCM.M-K8.2019
  - Volume of Si core,  $V_{\text{core}}$
  - Mass of surface layer,  $m_{\text{SL}}$
- Parameters taken from the previous works
  - Lattice constant,  $a$
  - Relative atomic mass of Si,  $A_r(\text{Si})$
  - Mass deficit due to impurities and vacancies,  $m_{\text{deficit}}$

# Volume measurement and surface characterization to realize the new kilogram

Volume measurement by optical interferometry

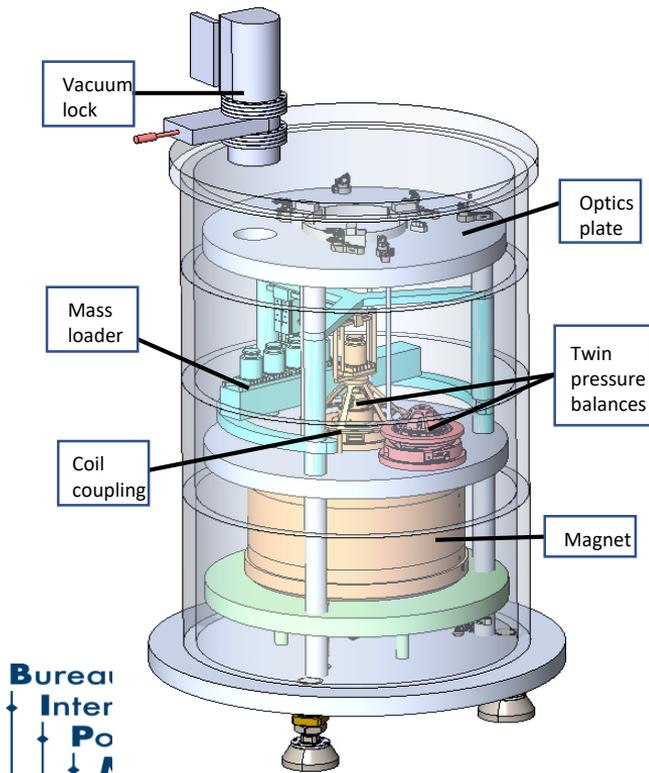


Surface characterization by X-ray photoelectron spectroscopy (XPS) and ellipsometry



- $u_r(m_{\text{sphere}}) = 2.1 \times 10^{-8}$ , 21  $\mu\text{g}$  for 1 kg
- N. Kuramoto *et al.*, *IEEE Trans. Instrum. Meas.*, DOI: 10.1109/TIM.2021.3061805

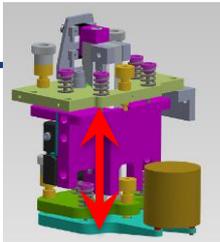
# MSL Kibble balance updates



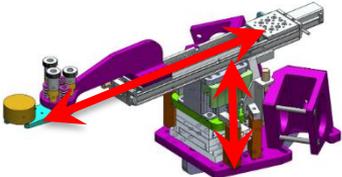
- Layout of modules has been decided, vacuum chamber being made.
- Measurement model under development.
- Still pursuing oscillatory moving mode.
- 3 experimental projects: pressure balances, interferometer, coil-magnet test.
- Pressure balances: rotating cylinder, syringe pump, repeatability of differential pressure transducer.
- Interferometer: voice coil, non-linearity error, AOM frequency shifting.
- Coil-magnet test: uniformity of field, parasitic forces, mass balance based KB.

# LNE Kibble balance progress report

## New mass lifters



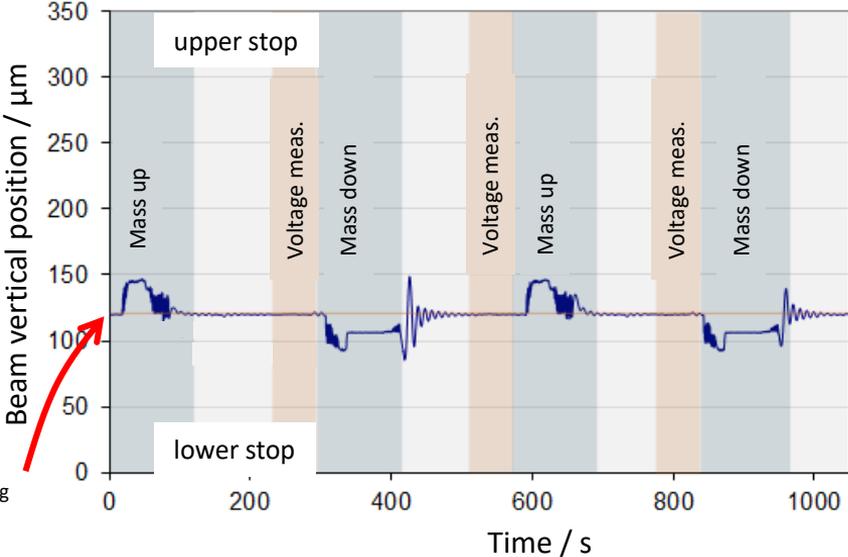
Lifter embedded in the translation stage:  
double weighing at different vertical positions



2-axis lifter: bring in and out the mass standard  
in the mass pan

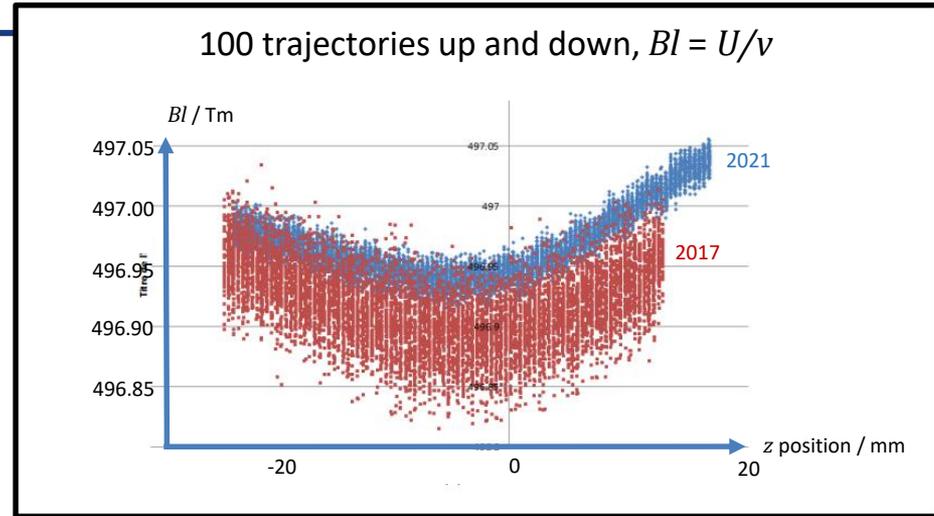
Altogether with servo control improvements:  
double weighing with a vertical beam excursion of only  
 $\pm 60 \mu\text{m}$

→ could reduce hysteresis in static phase



## Modification of the feet of the balance + modification around concrete slab

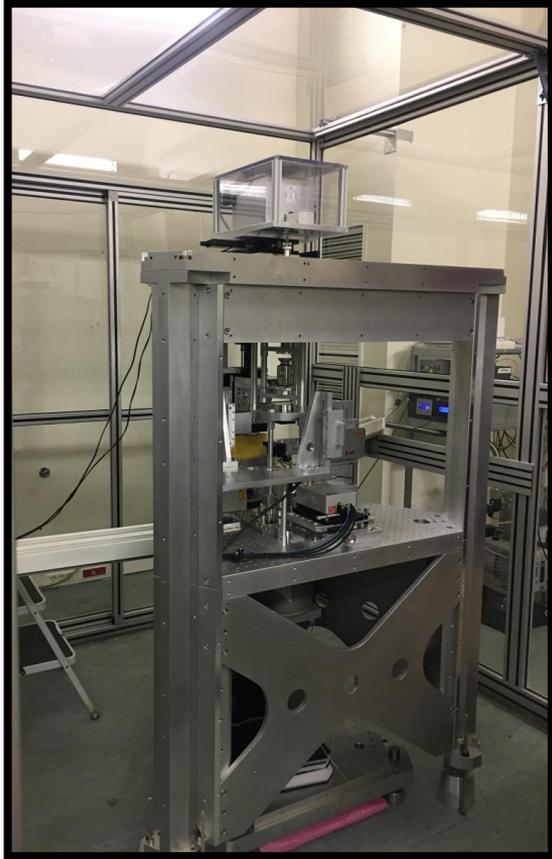
- Increase the **immunity of the experiment** to mechanical nearby activity
- Two-fold **improvement of the noise rejection** in dynamic phase



Works have been delayed some months due to the failure of the vacuum vessel lifter and of the current source

**Dynamic phases have been carried out in vacuum, as well as static phases: kilogram determinations will be the next step (mid-2021).**

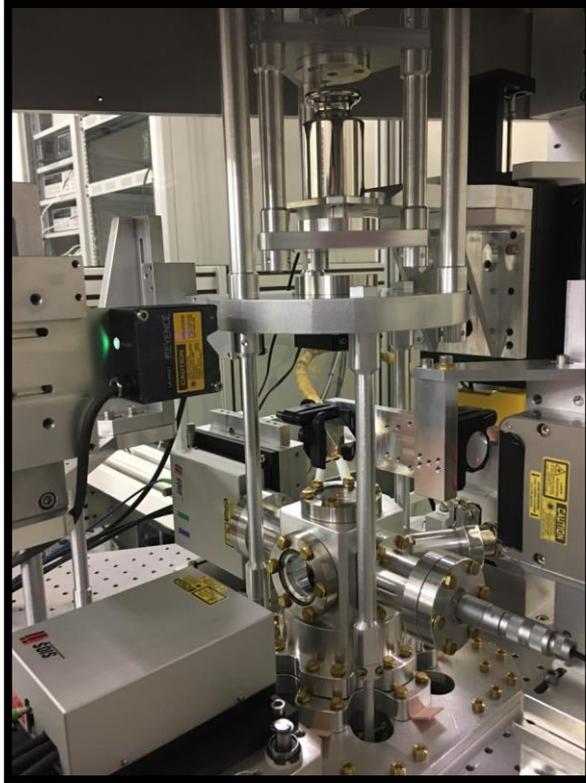
# UME-KB3



UME-KB3 was installed. Distinguishing aspects:

- Novel dynamical measurement procedure
- Local vacuum around the around the cubic mirror on the top of the moving magnet for determining the displacement of the magnetic circuit with respect to the coil
- Compact design including a moving magnetic circuit and a stationary coil
- Simultaneous testing of Faraday's Law of Induction and Ampere's Law of Force

# Current Status of UME-KB3



Local Vacuum & Interferometer Heads



Control Units & Instruments

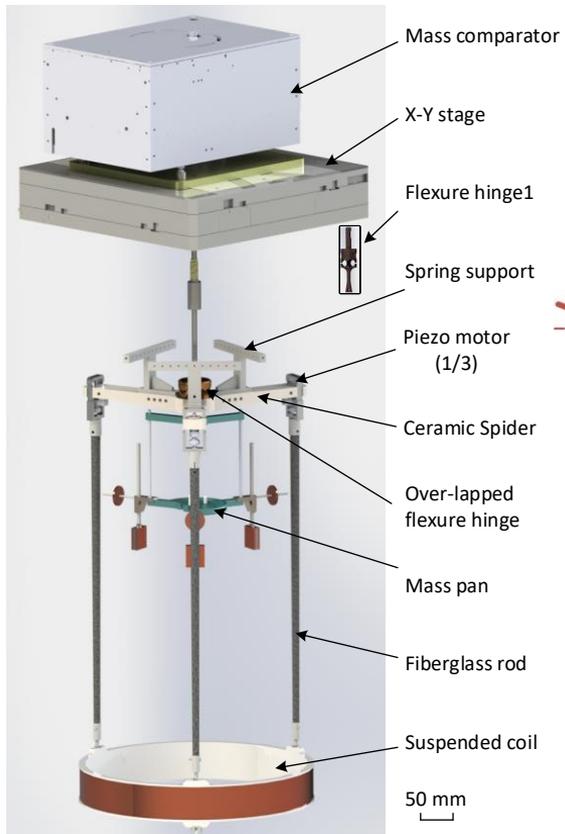
## Preliminary Measurement Results

Uncertainty parameter	Relative Uncertainty (ppb)
Alignment	45
Resistance	45
Mass	38
Others	19
Total	77



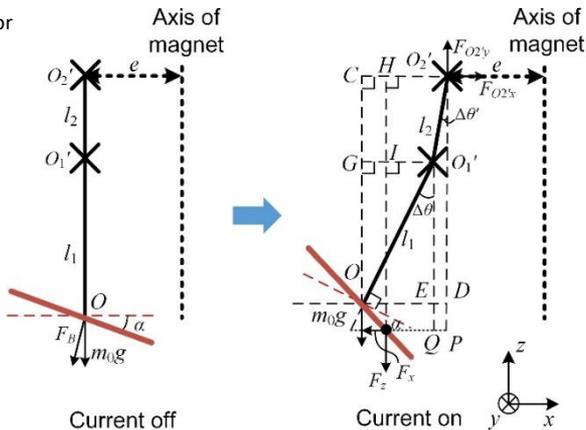
# NIM - Current status of the Joule balance

## 1. An automatic alignment technic for suspended coil in the Joule balance



Suspended structure in joule balance

### The principle of automatic alignment



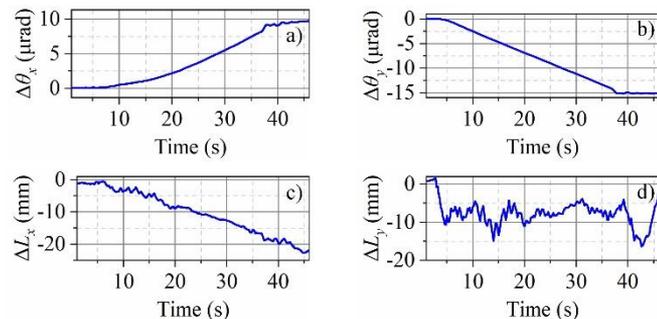
Linear Relationship

$$\begin{pmatrix} \alpha \\ e \end{pmatrix} \longleftrightarrow \begin{pmatrix} \Delta L \\ \Delta \theta \end{pmatrix}$$

Initial position /posture

Parasitic displacement

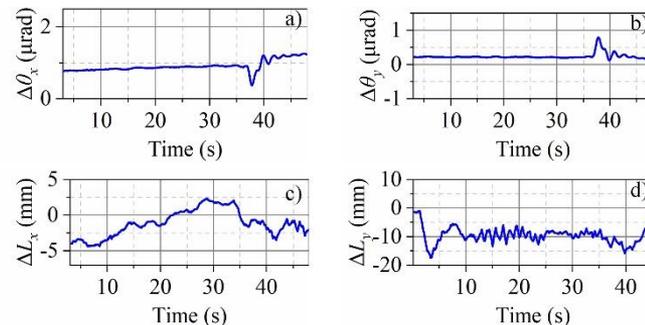
### Automatic alignment Test (Current off/on)



Non-alignment status

Automatic adjustment

Within several minutes

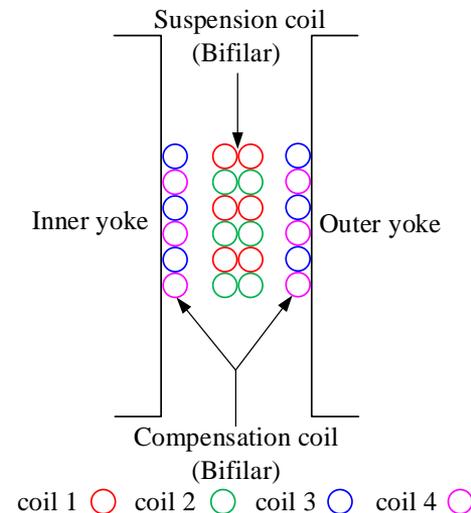
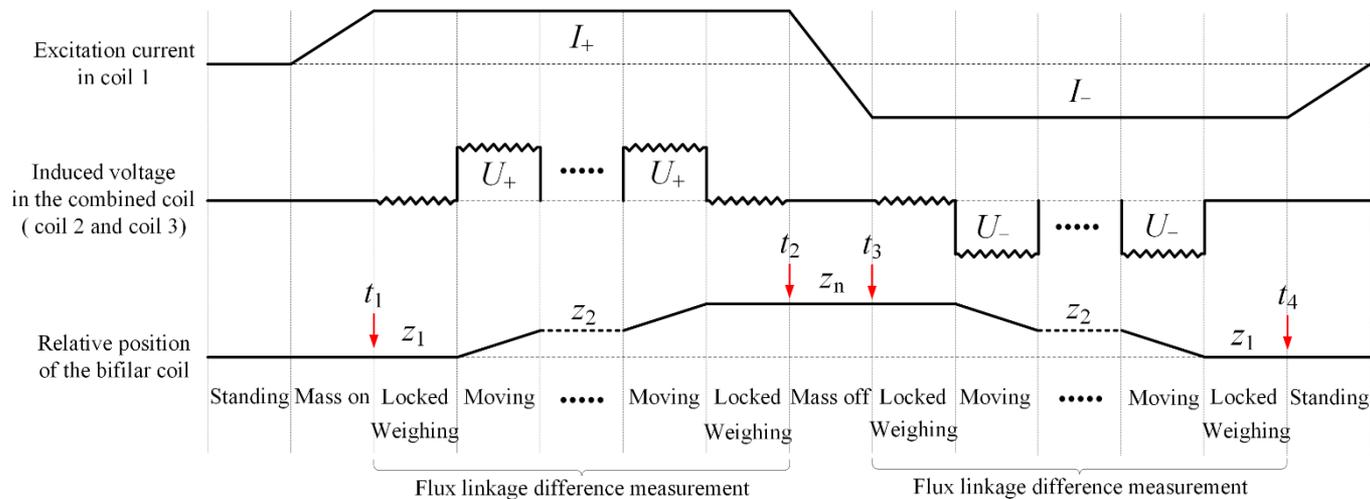


Alignment status

# NIM - Current status of the Joule balance

## 2. The one-mode one measurement phase (OMOP) scheme in the Joule balance

- 1) Consistency of the two independent coils of the bifilar suspension coil:  $\sigma < 3E-9$
- 2) Evaluation of the coil current effect,  $U_{rel} < 4.3E-9$
- 3) A bifilar compensation coil is new wound to suppress the effect of the external magnetic field and the magnetization effect of the suspension system simultaneously.
- 4) The whole system is still under construction and testing.



[1] Fang H, et al. Status of the BIPM watt balance. *IEEE Trans. Instrum. Meas.*, 2013, 62(6):1491-1498.  
 [2] Robinson I A. A simultaneous moving and weighing technique for a watt balance at room temperature. *Metrologia*, 2012, 49(1):108-112.  
 [3] Qian L S, et al. Preliminary Study on the OMOP Joule Balance. *IEEE Trans. Instrum. Meas.*, 2020, 69(9):6478-6489.  
 [4] Qian L S, et al. The interaction between the magnetized coil-suspension system and the compensation coil in the joule balance. *Metrologia*, 2020, 57(4):045010.

# Realisation with $^{28}\text{Si}$ spheres

## Reference $^{28}\text{Si}$ sphere AVO28-S8c (primary mass standard):

Source of uncertainty	Standard uncertainty / $\mu\text{g}$
Molar Mass	5.4
Lattice parameter	6.5
Point defects	3.8
Surface layer mass	7.7
Mean Diameter	9.4
Combined standard uncertainty ( $k = 1$ ), w.r.t. realisation of the definition of the kilogram	15.3

## Reference $^{28}\text{Si}$ sphere Si28kg01a (primary mass standard):

Source of uncertainty	Standard uncertainty / $\mu\text{g}$
Molar Mass	1.5
Lattice parameter	6.3
Point defects	3.4
Surface layer mass	7.0
Mean diameter	8.7
Combined standard uncertainty ( $k = 1$ ), w.r.t. realisation of the definition of the kilogram	13.4

- Realisation with two  $^{28}\text{Si}$  spheres (AVO28-S8c and Si28kg01a) made from two different crystals
- Previous results of material properties were used
- Volume and surface layers were measured anew
- Agreement of two realisations based on two different silicon crystals within  $1 \times 10^{-8}$

- New  $^{28}\text{Si}$  material (two single crystal boules) for future realisation and dissemination received
- Manufacture of two new  $^{28}\text{Si}$  spheres completed; another  $^{28}\text{Si}$  sphere in progress
- Extended investigations of material parameters (lattice parameter, impurities and their distribution) in preparation
- Density comparisons show agreement with mass/volume results of different spheres
- Investigation of the repeatability of the cleaning method applied for silicon spheres at PTB by gravimetric measurements and by XRF/XPS analysis of the surface layers; results show that the repeatability can be characterised by a standard deviation in the order of two micrograms
- “Planck-Balance” – improvement in the determination and correction of Abbe error; application of an AC-Josephson standard

# NIST will have two primary realizations.

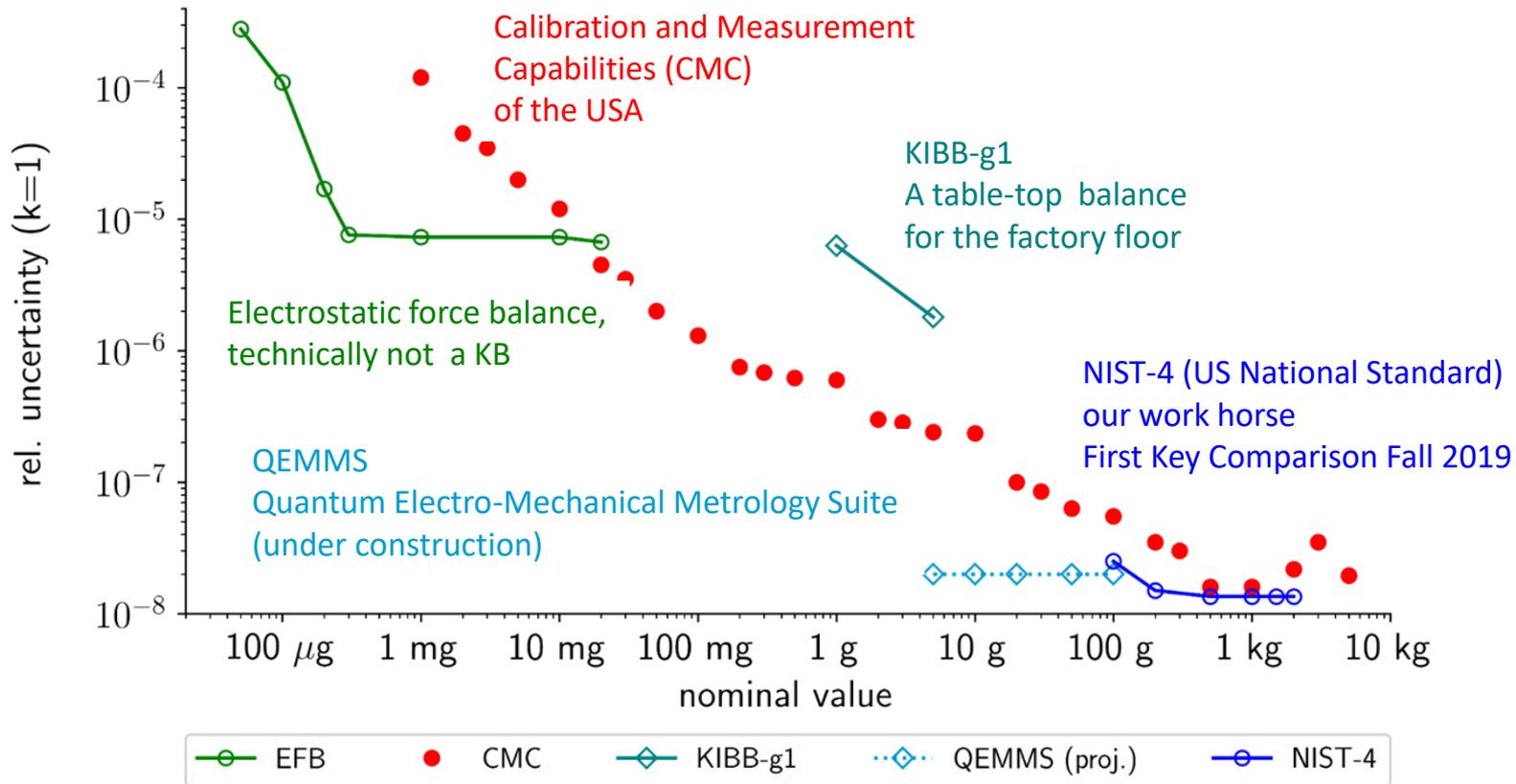
## NIST-4, the current workhorse (50g-2kg)

- Realization of two primary masses at 100g and 50g level.
- Getting ready for the next key comparison.

## QEMMS (10g-200g) Quantum Electro-Mechanical Metrology Suite (QEMMS) is composed of a Kibble balance, a Josephson voltage standard and a Quantum Hall resistance standard.

- Magnet/coil ready.
- Vacuum chamber to be delivered end of April 2021 .
- Cryogen Free PJVS ready.
- Cryogen Free Superconducting Magnet System for QHR in purchase.
- Flexure based mechanism for weighing and moving ready in few months.





# Pools of Artifacts for Mass Dissemination and SIM Kilogram Project

## VACUUM POOL

- **Artifacts:** (3) Pt-Ir kilograms, cylindrical,  
(2) Stainless-Steel kilograms, cylindrical or OIML
- Designed to act as “flywheel” for NIST-4 KB realizations
- **Traceability:** Consensus value of kilogram
- **Transfer of mass unit to AIR POOL** using measured vac to air correction
- **Stability:** ~ 0.003 mg over past 18 months; << Unc. (.021 mg)

## AIR POOL

- **Artifacts:** (3) Pt-Ir kilograms, cylindrical,  
(2) Stainless-Steel kilograms, cylindrical
- Designed to act as starting point of mass scale in air
- **Stability:** Pt-Ir: 0.001 to 0.003 mg over 18 months;  
Stainless-Steel: ~0.005 mg over 18 months

## SIM Kilogram Project

- Piloted by NRC and NIST
- SS Kilograms calibrated at NRC/NIST and distributed to 32 countries within SIM RMO in October 2018
- Storage conditions monitored in host labs (T, P, Rh)
- Scheduled to return to NRC/NIST in June 2021 for recalibration

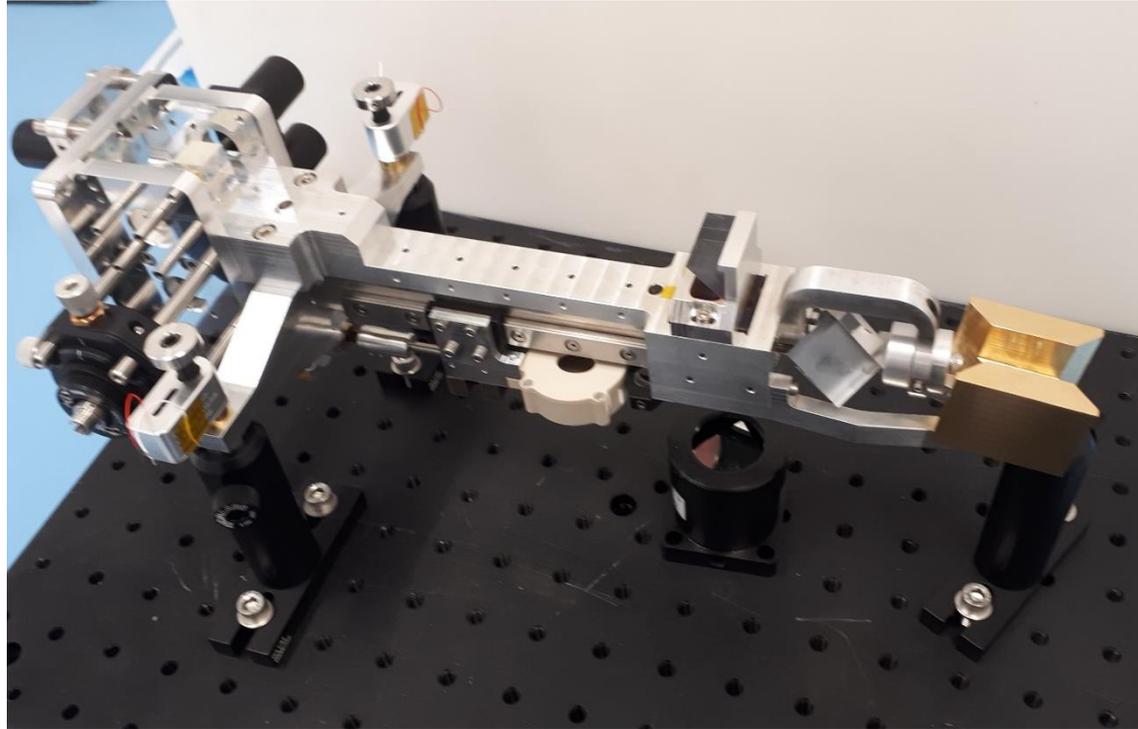
# NPL Kibble Balance developments

- We are starting the manufacture of the three Kibble balances required for the collaboration between NPL, NMISA (South Africa) and RISE (Sweden).
- The balances are based on a seismometer-like design and employ a bifilar wound main coil to allow single-mode two-phase operation.
- The balance is designed to be compact. Most of the electronic systems are built into hinged, shielded enclosures around its base and are isolated via the optical fibre ring.
- The balance will measure masses up to 250 g with an initial uncertainty of 1 part in  $10^7$ , but with the ultimate aim of 2 parts in  $10^8$  or better.

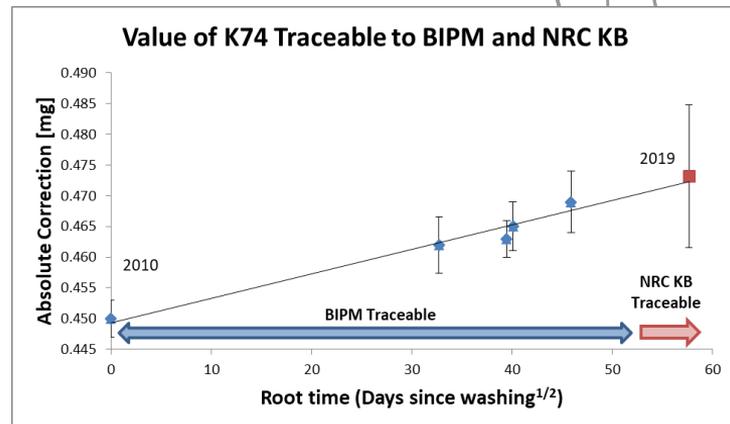
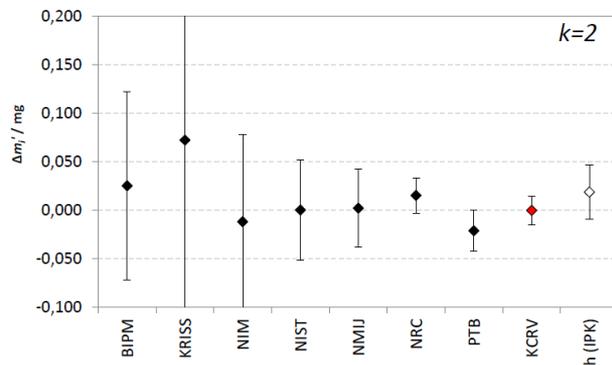
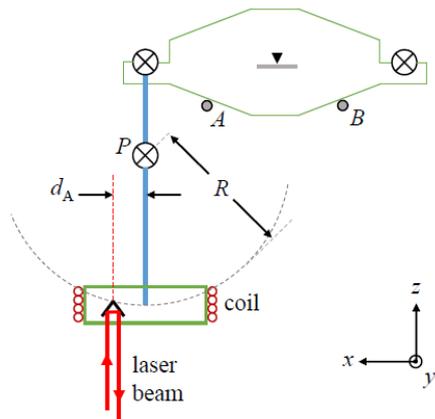


# NPL Kibble Balance developments

- The interferometer has been constructed and is intended to determine, and adjust, the verticality of the laser beam.
- We will be evaluating a small number of mechanisms for guiding the moving part of the balance.



# Update and Participation in CCM.M-K8.2019



C. A. Sanchez and B. M. Wood, "Abbe Offset Measurement in the NRC Kibble Balance," doi: 10.1109/CPEM49742.2020.919177

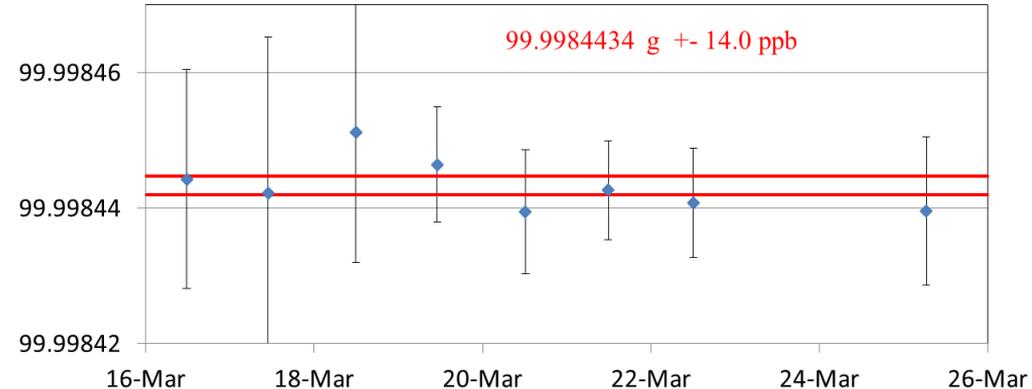
M Stock *et al* 2020 *Metrologia* 57 07030

- Re-evaluation of Abbe offsets yielding an uncertainty of 3 ppb were included in result of CCM.M-K8.2019
- Realization measurements demonstrated good stability wrt. to BIPM working standards and NRC mass scale (< 3.5 ppb)

# 100 g realization comparison with NIST: Interrupted by COVID

- Type A at 100 g was found to be 14 ppb in this study
- Type A at 500 g typically ~2.5 ppb
- Combined uncertainty at 100g expected to be between 20-25 ppb with type A and knife edge hysteresis the dominant components
- Slightly higher uncertainty than would be expected from subdividing from realizations at 500 g or 1 kg (~16 ppb)

NRC KB measurements of 100 g Silicon mass



# New kg realization via XRCD method at CMS/ITRI

Planck constant  $h$



Primary mass standard (vacuum)

Vacuum to air transfer



Secondary mass standard (air)

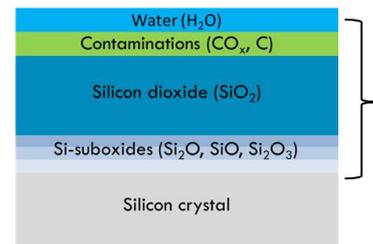
Dissemination

1 mg – 1000 kg

- Evaluate the mass of the Si-sphere *under vacuum*

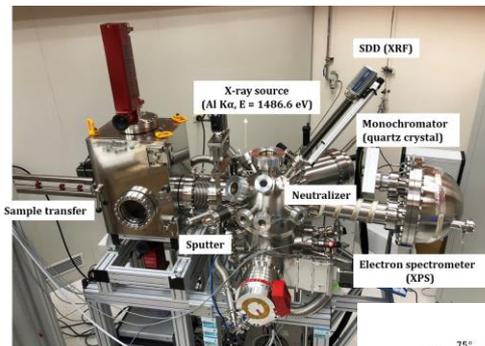
$$m_{sphere} = \frac{8V_{core} \sum_i x(iSi) A_r(iSi)}{a^3 A_r(e)} \frac{2hR_{\infty}}{c\alpha^2} - m_{defect} - m_{SL}$$

$m_{core}$

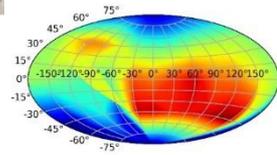


Surface layer model of Si-sphere

- XRF/XPS surface analysis system for the measurement of  $m_{SL}$



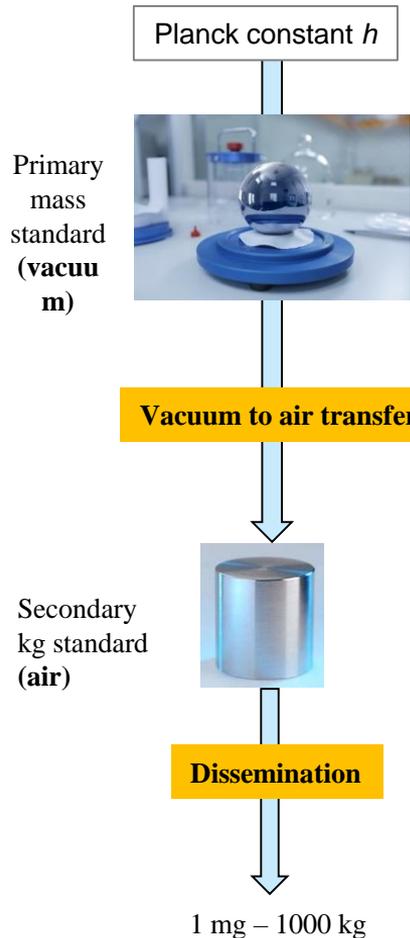
Total mass deposition of oxygen:  $36.68 \pm 3.37 \mu\text{g}$



- Evaluate  $m_{sphere}$  *under vacuum* based on  $m_{SL}$  and other pre-determined parameters of  $m_{core}$

$X_i$	$x_i$	$u(x_i)$	Unit	$c_i(y_j)$	$u_i(y_j)$
$M_e$	5.485799E-07	1.65E-16	kg · mol <sup>-1</sup>		
$M_u$	1.000000E-03	3.00E-13	kg · mol <sup>-1</sup>	1000	3.00E-10
$A_r(e)$	5.485799E-04	1.60E-14		1800	2.90E-11
$h$	6.626070E-34	0.00E+00	kg · m <sup>2</sup> · s <sup>-1</sup>		
$R_{\infty}$	1.097373E+07	2.10E-05	m <sup>-1</sup>	9.10E-08	1.90E-12
$c$	2.997925E+08	0	m · s <sup>-1</sup>		
$\alpha$	7.297353E-03	1.10E-12		270	3.00E-10
$M_{Si}$	2.797694E-02	1.40E-10	kg · mol <sup>-1</sup>	36	5.00E-09
$V_{core}$	4.310555E-04	5.06E-12	m <sup>3</sup>	2300	2.20E-08
$a$	5.430996E-10	1.46E-18	m	5.50E+09	8.10E-09
$m_{defect}$	1.740E-08	3.98E-09	kg	1	4.00E-09
$m_{SL}$	6.5840E-08	6.66E-09	kg	1	6.70E-09
$m_{sphere}$	1.0000783890	1.91E-8	kg		

# New kg realization via XRCD method at CMS/ITRI



- Vacuum-air transfer the mass from the Si-sphere to the stainless steel kg standard

$$m_{ss,air} = m_{ss,vac} + A_{ss} \cdot s \rightarrow s = \frac{\Delta m_{vac} - \Delta m_{air}}{\Delta A}$$

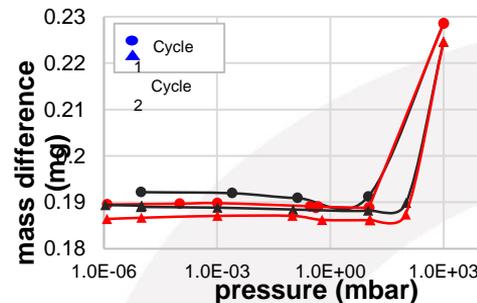
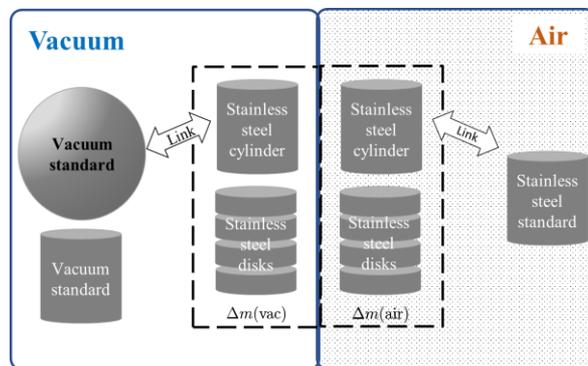
- ✓ Sorption coefficient  $s$  estimated by measuring the mass difference of sorption artefacts in air and vacuum with vacuum mass comparator.



Vacuum mass comparator



Sorption artefacts



	$\Delta m_{vac}$ (mg)	$\Delta m_{air}$ (mg)	$\Delta A$ (cm <sup>2</sup> )	$s$ ( $\mu\text{g}/\text{cm}^2$ )
10 disks	-0.1865	-0.1717	434.4310	0.0342



# SIM Kilogram Dissemination Project

- Collaboration Co-led by NIST and NRC

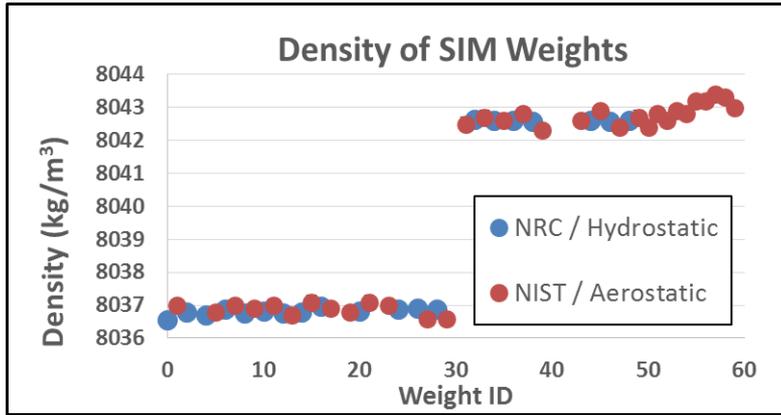
- **Objectives:**

1. To provide a practical artefact traceable to two Kibble balances in the region.
2. Develop a highly correlated set of masses for which characterization of a subset can be applied to the full set laying the foundation for studies correlating mass behavior with surface characteristics, handling and environment

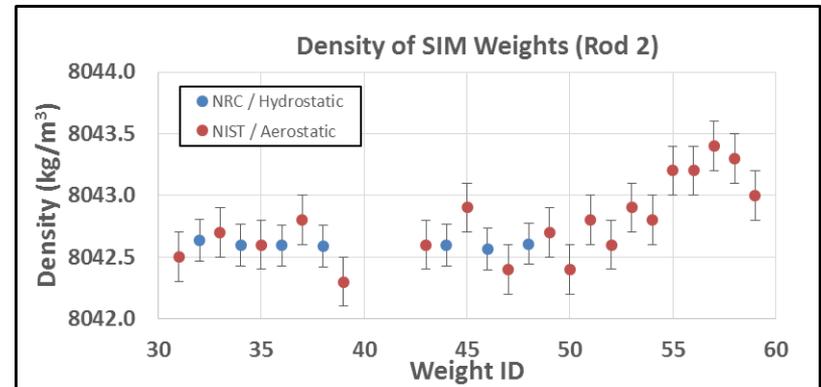
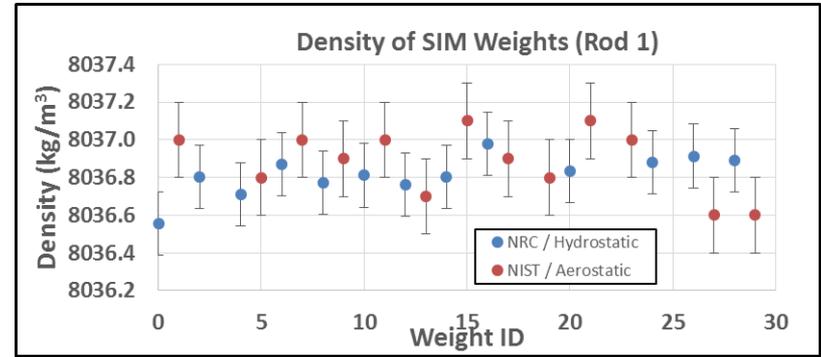




# NRC-NIST Bilateral Comparison: No masses were exchanged

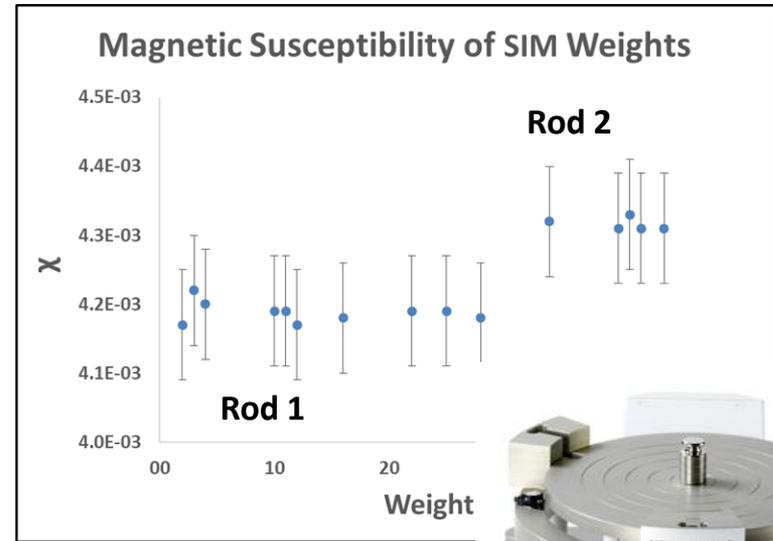


- $\rho_f \Delta V_{t-r} \rightarrow 2$  methods
- Aerostatic-  $\Delta$ Buoyancy with air density
- Hydrostatic-  $\Delta$ Buoyancy between fluids



# Present Status of Characterization: Magnetic properties

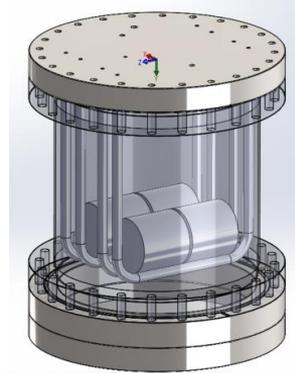
- **Magnetic Susceptibility measurements complete**
  - Full Susceptibility and permanent magnetization determined on subset of masses
  - All Masses checked to assure E1 class met
  - Average Susceptibility:  
0.00423 +/- 0.00007  
All masses satisfy E1 class criteria



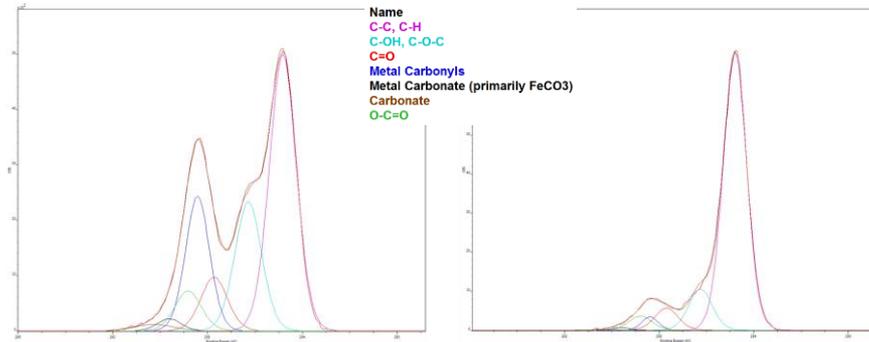
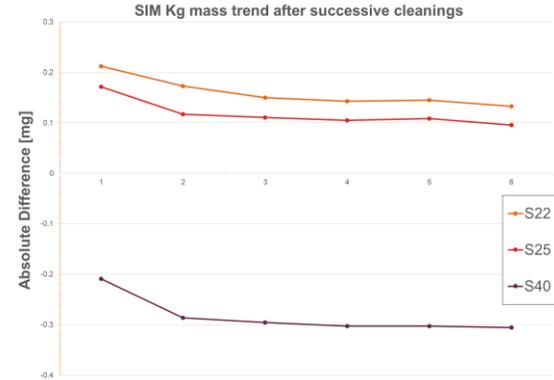
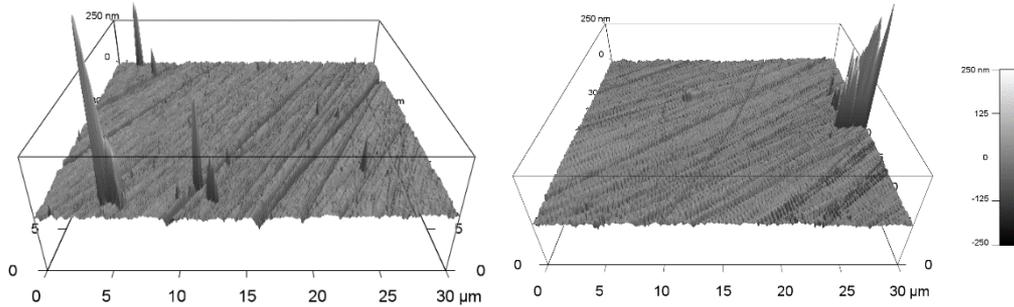
# Cleaning post density determination (NRC)

**Step 1)** Ethanol vapour degreaser

**Step 2)** Sonicate in cascade bath fed by ultrapure water (18.3 M $\Omega$ )

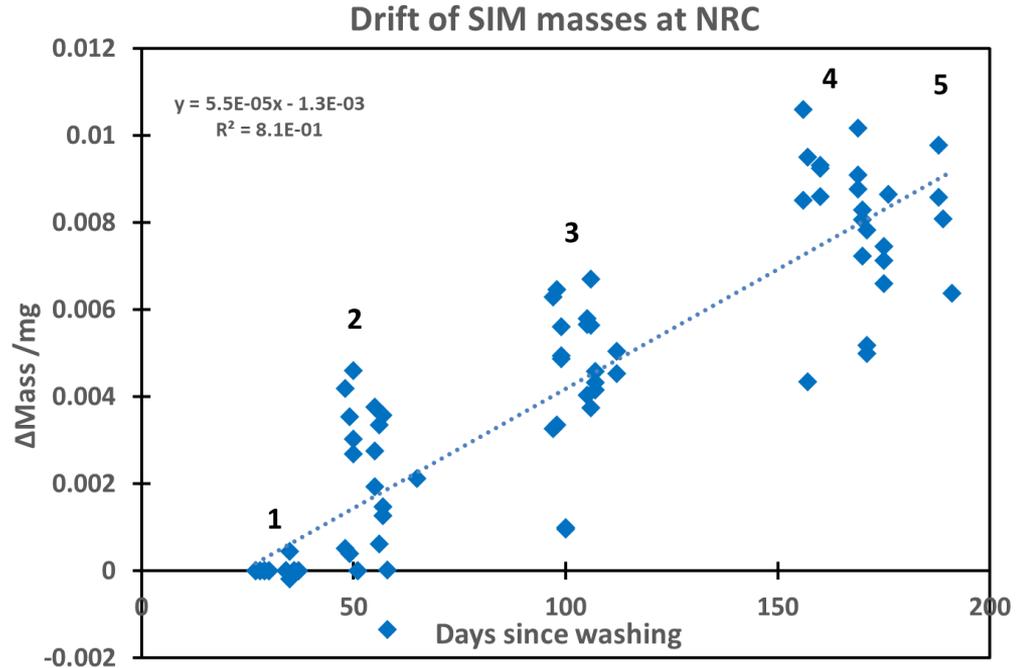


# Mass and Surface analysis: Impact of cleaning



# Mass Drift in Time

- Mean drift approximately 56 +/- 10 ng/day after washing



# Transport Enclosure: Traveling case contents



# Status

- Masses and kits distributed beginning October 2018
- Protocol and Data recording template complete translated and distributed to participants:
  - Frequency and number of stability of measurements recommended based on capability
  - Strategy to encourage participation through flexibility
  - High value even if no measurements are performed at assigned NMI
- First recalibration campaign delayed due to COVID-First round now planned for late June 2021
- First masses have been received at NRC
- Many NMIs have reported large disruption due to COVID impacting ability perform stability measurements but most have communicated intent to return mass for recalibration



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