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Analysis of the correlation of sorption coefficients to pressure

James Berry (NPL), Michael Borys (PTB), Martin Firlus (PTB), Richard Green (NRC), Andrea Malengo (INRIM), Michael Mecke (PTB), Paul-André Meury (LNE), Jaroslav Zůda (CMI)

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# ABSTRACT

This report summarises the results of an analysis of the correlation of sorption coefficients with pressure. Sorption measurements were made between humid air and pressures ranging from 0.1 Pa to 10<sup>-3</sup> Pa using platinum-iridium, silicon and stainless steel artefacts. The mass difference for all materials was found to stay the same over the 0.1 Pa to 10<sup>-3</sup> Pa pressure range and therefore consistent mass measurements between primary kilogram realisations can be achieved providing this pressure range is used.

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Approved on behalf of NPLML by Stuart Davidson, (Science Area Leader - Mass).

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#### **1** INTRODUCTION

The aim of this task was to analyse the correlation of sorption coefficients with pressure and to recommend appropriate pressure ranges for the mass transfer between vacuum and air in order to minimise the uncertainty in the dissemination of the mass scale from primary mass standards in vacuum to masses in air. A future fundamental constant definition of the kilogram will be realised in a vacuum and it is essential that any changes in sorption coefficients due to variations in pressure during the measurement of the artefacts used to provide traceability to the new definition are well understood. Sorption coefficients are applied when correcting the mass value of an artefact in air to allow for a change in humidity or when correcting the mass value of an artefact for the moisture lost/gained from its surface when it is transferred to/from vacuum.

The participating institutes are given in Table 1.

Participant	
Cesky Metrologicky Institut	CMI
Instituto Nazionale Di Ricerca Metrologica	INRIM
Laboratoire National de Métrologie et d'Essais	LNE
National Physical Laboratory	NPL
National Research Council Canada	NRC
Physikalisch-Technische Bundesanstalt	PTB

Table 1. List of participating labor	atories.
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Previous work on evaluating sorption effects on materials commonly used for primary mass standards, i.e. platinum-iridium (Pt-Ir), silicon and stainless steel, has been performed by Davidson [1], Picard and Fang [2], Schwartz [3] and Berry *et al* [4]. Recently published work by Berry and Davidson [5] has also examined the correlation of sorption coefficients to vacuum pressure for Pt-Ir, silicon and stainless steel artefacts. These published sorption values for the different materials and the pressure at which the measurements were performed are summarised in Table 2 with additional information on the time since the artefacts were cleaned. The work by Schwartz [3] and Berry and Davidson [5] measured sorption from atmospheric pressure to 10<sup>-3</sup> Pa and found evidence of hysteresis between atmospheric pressure and 0.1 Pa, however there was no change in the mass difference between the artefacts, and hence the sorption value, over the 0.1 Pa to 10<sup>-3</sup> Pa pressure range. As the work by Berry and Davidson showed that the sorption values were stable over a 0.1 Pa to 10<sup>-3</sup> Pa pressure range, this range was selected for evaluation in this study.

Author(s)	Material	Sorption value	Pressure	Time between cleaning
		/ µg cm <sup>-2</sup>	/ <b>Pa</b>	arteracts and measurement
Davidson [1]	Pt-Ir	-0.162	10-4	< 6 months
Davidson [1]	stainless steel	-0.154	10-4	< 6 months
Picard & Fang [2]	Pt-Ir	-0.080	0.1	< 1 month
Picard & Fang [2]	silicon	-0.030	0.1	< 1 month
Picard & Fang [2]	stainless steel	-0.040	0.1	< 1 month
Schwartz [3]	stainless steel	-0.024	0.1	< 8 months
Berry et al [4]	stainless steel	-0.13 to -0.25	0.05 to 10 <sup>-4</sup>	< 3 years
Berry & Davidson [5]	Pt-Ir	-0.070	0.1 to 10 <sup>-3</sup>	< 5 years
Berry & Davidson [5]	silicon	-0.050	0.1 to 10 <sup>-3</sup>	< 5 years
Berry & Davidson [5]	stainless steel	-0.150	0.1 to 10 <sup>-3</sup>	< 5 years

Table 2. Published sorption values of the materials used and pressure level of the measurements.

### 2 EXPERIMENTAL PROTOCOL

#### 2.1 SORPTION ARTEFACTS

Sorption artefacts are used to determine sorption coefficients and usually take the form of pair of artefacts with similar volumes but with different surface areas. The first artefact is usually a single integral artefact and the second artefact usually comprises a stack of discs separated by small spacers so that the effective surface area of the stack artefact is much greater than that of the integral artefact. Sorption artefacts manufactured from Pt-Ir, silicon and stainless steel were used in this study. Originally Tungsten artefacts made at NRC were included, but tests at NPL and NRC showed poor stability in vacuum and work is underway to improve the surface finish of these artefacts. The nominal volumes, volume uncertainties, surface areas and cubic coefficient of thermal expansion values for the sorption artefact materials tested in this study are given in Table 3. All of the participants in this study used Mettler-Toledo Mone mass comparators for the measurements.

Artefact Identification	Nominal mass	Material	Volume at 20 °C	Volume uncertainty (k = 1)	Surface area	Cubic coefficient of thermal expansion	
	/ g		/ cm <sup>3</sup>	$/ \mathrm{cm}^3$	/ cm <sup>2</sup>	Alpha / 10 <sup>-6</sup> °C <sup>-1</sup>	Beta / 10 <sup>-9</sup> °C <sup>-2</sup>
Pt_NPL_I	1 000	Pt-Ir	46.416 0	0.000 2	71.501 7	25.869	0.00565t
Pt_NPL_S	1 000	Pt-Ir	46.415 2	0.003 2	149.652 0	25.869	0.00565t
Si_NPL_I	500	silicon	213.795 7	0.001 4	207.241 3	7.8	0
Si_NPL_S	500	silicon	213.734 3	0.003 0	383.278 5	7.8	0
Steel_INRIM <sup>a</sup>	1 000	steel	-0.005 8	0.001 1	-145.0	48.0	0
Steel_PTB	1 000	steel	124.834 0	0.001 0	138.3	45.6	0
Steel_PTB	1 000	steel	124.834 0	0.004 0	573.8	45.6	0
Steel_NPL	1 000	steel	126.771 9	0.001 6	140.02	45.0	0
Steel_NPL	1 000	steel	126.769 9	0.002 4	285.38	45.0	0

**Table 3.** Sorption artefacts used in this study.

<sup>a</sup>Only the relative volume differences (integral – stack) with associated uncertainties were available for the INRIM stainless steel artefacts. The relative surface area difference between the INRIM artefacts is also given.

### 2.2 HANDLING AND CLEANING THE ARTEFACTS

#### 2.2.1 Handling the artefacts

The artefacts were handled using two layers of gloves. The inner layer comprised latex or nitrile gloves and the outer layer was Duraclean<sup>®</sup> Lycra<sup>®</sup> clean room gloves (as used in the National Measurement Institute Australia silicon sphere cleaning method). Small tweezers were used to manipulate the spacer artefacts that were used to separate the discs forming each stack artefact. The type of tweezers was not specified and so participants were free to use either plastic or metal ones. Any visible dust on the surface of the artefacts was removed using a soft brush.

#### 2.2.2 Cleaning the artefacts

A two-stage cleaning process was used to clean the artefacts before the start of the measurements and no further cleaning was performed once the measurements had started. The washing procedure was performed in a clean dust free environment and the artefacts were handled using the same gloves as described in 2.2.1.

The first cleaning stage consisted of washing in a solvent in an ultrasonic bath. The solvent used was reagent grade ethanol with a purity of at least 99.8%. Each artefact was placed in turn in the ethanol inside the ultrasonic bath and ultrasonic cleaning was performed for five minutes. The ultrasonic cleaning process was repeated with the other artefacts and finally the spacer weights that made up the stack artefacts were cleaned.

The second cleaning stage involved rinsing in distilled water. Distilled water was poured over the sorption artefacts in copious amounts for five minutes using a plastic squeeze bottle. Each artefact was inverted half way through to rinse the bottom of the artefact. The artefacts were then left to dry in air with large water droplets removed using clean lens tissue paper.

The PTB stainless steel sorption artefacts were not cleaned before the start of the measurements.

#### 2.3 MEASUREMENT PROCEDURE

The artefacts were left for a period of seven days after cleaning to allow them to stabilise before the first set of measurements. The first set of measurements (1) was performed at 10<sup>5</sup> Pa (atmospheric pressure) at a humidity of about 50% RH. The second set of measurements (2) were performed at a vacuum pressure of 0.1 Pa and the artefacts were left for 24 hours to stabilise before starting the measurements. The desired pressure within the vacuum chamber was attained by using a gas needle valve to leak filtered dry air from either a compressed air line or gas bottle into the vacuum chamber. The third (3) and fourth (4) sets of measurements were performed at vacuum pressures of 0.01 Pa and 0.001 Pa respectively which were achieved by reducing the flow of air through the needle valve. A stabilisation time of 12 hours was sufficient prior to the start of the third and fourth measurements. After completion of the fourth measurement the pressure within the chamber was raised by increasing the flow of air through the needle valve. The fifth (5) and sixth (6) measurements were then performed at 0.01 Pa and 0.1 Pa respectively. Again, a twelve hour stabilisation time was sufficient prior to the start of these measurements. The cycle of measurements was completed by switching off the vacuum pumps and taking the chamber back up to ambient pressure ( $10^5$  Pa). The humidity within the chamber was increased by flowing clean air through water within a glass bottle until the humidity within the chamber was stable to around 50% RH. The artefacts were then left to stabilise for at least 48 hours before the start of the measurements. A diagram depicting the measurement cycle is shown in Figure 1, between three and four repeat cycles were performed for each material.

It should be noted that the NPL measurements used an earlier version of the protocol in which the measurements in air were performed after a delay of 24 hours instead of 48 hours and the first measurement in vacuum was performed after a delay of 48 hours instead of 24 hours.

For comparison purposes this report also includes sorption data from artefacts measured solely on Mettler-Toledo M<sub>one</sub> comparators for task 2.3 [6] which includes the INRIM, LNE, NPL 2013 and PTB measurements on the NPL Pt/Ir artefacts, the PTB measurements on the PTB stainless steel artefacts and the NPL summer 2013 measurements on the silicon artefacts. The protocol for task 2.3 only required measurements at atmospheric pressure and at a pressure of 0.01 Pa and as such only data at these two points were available.

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**Figure 1.** Measurement cycle from  $10^5$  Pa to 0.001 Pa.

#### **3 RESULTS**

#### 3.1 MASS DIFFERENCES AND STANDARD UNCERTAINTIES

The average mass differences and standard uncertainties in both air and vacuum reported by the participants are given in Table 4. Two mass differences in air are shown for each measurement; the first value is the uncorrected mass difference in air and the second value is the mass difference in air corrected to a relative humidity of 50 % RH. The following formula was used to correct the mass differences to 50 % RH.

Where:

$$\alpha_h = A \times \delta_h \times S \tag{1}$$

Where:

 $\alpha_h$  = humidity correction A = humidity correction coefficient  $\delta_h$  = humidity difference to 50 % RH (50 - RH) S = Surface area difference

The humidity correction coefficients were calculated from the measurement results for each set of participant data according to the following formula:

$$A = \frac{M_{DIFF_{VAC}} - M_{DIFF_{AIR}}}{RH \times S}$$
(2)

Where:

 $M_DIFF_{VAC}$  = Average mass difference at 0.01 Pa  $M_DIFF_{AIR}$  = Average mass difference in air (uncorrected)

The applied humidity corrections are very small and do not significantly affect the calculated sorption coefficients. It is worth noting that equation (2) is not valid at an RH = 0.

The change in average mass difference between the artefacts relative to the initial mass difference at 0.1 Pa has been plotted against the average pressure in Figure 2, Figure 3 and Figure 4 for the Pt-Ir, silicon and stainless steel artefacts respectively. For illustrative purposes previously published values from Berry & Davidson [5] are also plotted on figures 2 to 4.

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#### 3.2 SORPTION VALUES AND UNCERTAINTIES

The calculated average sorption values and standard uncertainties are given in Table 5. Sorption values have been calculated for each pressure level and they have been adjusted so that they represent the correction going from air with a humidity of 50 % RH to vacuum. The signs of the sorption values are negative as they represent the correction required when going from air to vacuum. Calculation of the sorption values and calculation of the standard uncertainties have been done according to the method used by Berry *et al* [4]. The average pressure measured by the participants and the range of relative humidity values during the measurements is given in Table 6. The calculated sorption values have been plotted against the average pressure in Figure 5, Figure 6 and Figure 7 for the Pt-Ir, silicon and stainless steel artefacts respectively. For illustrative purposes sorption values published by Davidson [1], Picard and Fang [2], Schwartz [3] and Berry and Davidson [5] are also included in figures 5 to 7.

#### 4 CONCLUSIONS

The measured mass differences between all of the artefacts made from Pt-Ir, silicon and stainless steel did not change over the 0.1 Pa to 0.001 Pa pressure range. This confirmed the previous work by Schwartz [3] and Berry and Davidson [5] which also did not show a variation in mass difference over this pressure range. Therefore the main aim of this task has been met as a pressure range of 0.1 Pa to 0.001 Pa can be recommended as suitable for the measurement in vacuum of masses used in the dissemination of the mass scale. There was a difference however, between the absolute mass difference between the NPL Pt-Ir artefacts measured at NPL and the difference measured at NRC. This probably resulted from a change in the surface properties of the artefacts due to them being cleaned again at NRC and further work is required to examine the effect that surface cleanliness has on sorption values for different materials.

The sorption coefficients for the Pt-Ir artefacts measured by the participants showed good agreement within the uncertainty of the measurements and also good agreement with the values published previously by Picard and Fang [2], and Berry and Davidson [5]. However, the sorption coefficient reported previously by Davidson [1] was much larger. This was interesting as the Pt-Ir sorption artefacts used in this study and the work by Berry and Davidson [5] came from the same set of artefacts as those used in the measurements by Davidson [1]. This larger sorption coefficient was unlikely to be due to differences in surface cleanliness as there was good agreement between the sorption results in this study (which were cleaned before starting the measurements) and the work by Berry and Davidson [5] (which were cleaned 5 years before starting the measurements). INRIM also cleaned the Pt-Ir artefacts before performing their measurements and it was interesting that the

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sorption coefficient obtained was in good agreement with the PTB and NPL 2013 coefficients where the artefacts had not been cleaned since the measurements at INRIM.

The silicon sorption values measured by NPL in spring 2013 agreed well with the values reported by Picard and Fang [2], and Berry and Davidson [5]. The silicon sorption values measured by NPL in summer 2013 and those measured subsequently by NRC agreed well with each other but were significantly shifted by about 0.05  $\mu$ g cm<sup>-2</sup> from the NPL spring 2013 values. The absolute mass of the silicon artefacts also changed with the silicon stack gaining mass relative to the integral artefact suggesting that the artefacts had gained surface contamination between the two NPL measurements. Therefore this suggested that the surface cleanliness of the silicon artefacts could have an effect on the resulting sorption value despite previously obtaining good agreement between the NPL spring 2013 values, which were cleaned prior to the measurements, and the measurements by Berry and Davidson [5] which were not.

The stainless steel sorption values measured by PTB and INRIM agreed well with the values published by Picard and Fang [2] and Schwartz [3] but were much lower than the values published by Davidson and Berry *et al* [5]. The stainless steel sorption values measured by CMI were much higher than the values measured at PTB and INRIM but were in broad agreement with the values reported previously by Berry *et al* [4]. This could have been due to a difference in surface cleanliness of the artefacts or possibly due to differences in the surface roughness of the artefacts.

It was interesting to observe from both the previously published sorption values and the values published in this report, that there appeared to be as much variation in sorption between artefacts manufactured from the same material as there was between artefacts made from different materials. This suggested that sorption values were not influenced as much by the type of material the artefacts were made from but were more greatly influenced by factors such as surface cleanliness and surface roughness.

#### **5** ACKNOWLEDGEMENTS

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## 7 TABLES OF RESULTS

					$\Delta m$ (	Integral - Stack)	/ mg		
Participant	Artefact	Artefact	Air	Air	Vacuum	Vacuum	Vacuum	Vacuum	Vacuum
	material	Identification		(50% RH)	(0.1 Pa)	(0.01 Pa)	(0.001 Pa)	(0.01 Pa)	(0.1 Pa)
NPL	Pt/Ir	Pt_NPL	1.640 3	1.640 2	1.644 3	1.644 2	1.644 2	1.644 2	1.644 3
NRC	Pt/Ir	Pt_NPL	1.683 1	1.683 0	1.684 1	1.683 8	1.683 7	1.683 7	1.683 4
LNE	Pt/Ir	Pt_NPL	1.671 9	1.672 0		1.675 7			
INRIM	Pt/Ir	Pt_NPL	1.653 3	1.653 6		1.658 0			
PTB	Pt/Ir	Pt_NPL	1.655 3	1.654 1		1.658 7			
NPL	Pt/Ir	Pt_NPL	1.661 9	1.662 5		1.666 5			
NPL (spring 13)	silicon	Si_NPL	449.444 6	449.444 5	449.450 0	449.449 3	449.449 8	449.448 8	449.448 8
NPL (summer	Silicon	Si_NPL	449.366 6	449.367 1		449.381 9			
NRC	silicon	Si_NPL	449.383 8	449.382 9		449.397 3			
INRIM	stainless steel	SS_INRIM	-0.393 9	-0.393 4	-0.386 7	-0.387 0	-0.388 0	-0.387 0	-0.387 4
РТВ	stainless steel	SS_PTB	0.038 3	0.035 9		0.045 4			
CMI	stainless steel	SS_NPL	1.681 1	1.681 1	1.719 3	1.724 0	1.722 4	1.720 1	1.719 4
					$u_{\Delta m}$ (	Integral - Stack)	/ mg		
Participant			Air	Air	u∆m ( Vacuum	Integral - Stack) Vacuum	/ mg Vacuum	Vacuum	Vacuum
Participant			Air	Air (50% RH)	u <sub>∆m</sub> ( Vacuum (0.1 Pa)	Integral - Stack) Vacuum (0.01 Pa)	/ mg Vacuum (0.001 Pa)	Vacuum (0.01 Pa)	Vacuum (0.1 Pa)
Participant NPL	Pt/Ir	Pt_NPL	Air 0.004 0	Air (50% RH) 0.004 0	u <sub>Δm</sub> ( Vacuum (0.1 Pa) 0.001 0	Integral - Stack) Vacuum (0.01 Pa) 0.001 0	/ mg Vacuum (0.001 Pa) 0.001 0	Vacuum (0.01 Pa) 0.001 0	Vacuum (0.1 Pa) 0.001 0
Participant NPL NRC	Pt/Ir Pt/Ir	Pt_NPL Pt_NPL	Air 0.004 0 0.003 7	Air (50% RH) 0.004 0 0.003 7	u <sub>Δm</sub> ( Vacuum (0.1 Pa) 0.001 0 0.001 3	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4	Vacuum (0.01 Pa) 0.001 0 0.001 4	Vacuum (0.1 Pa) 0.001 0 0.001 3
Participant NPL NRC LNE	Pt/Ir Pt/Ir Pt/Ir	Pt_NPL Pt_NPL Pt_NPL	Air 0.004 0 0.003 7 0.003 6	Air (50% RH) 0.004 0 0.003 7 0.003 6	$\begin{array}{c} u_{\Delta m} (\\ Vacuum \\ (0.1 \ Pa) \\ 0.001 \ 0 \\ 0.001 \ 3 \end{array}$	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4	Vacuum (0.01 Pa) 0.001 0 0.001 4	Vacuum (0.1 Pa) 0.001 0 0.001 3
Participant NPL NRC LNE INRIM	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL	Air 0.004 0 0.003 7 0.003 6 0.003 8	Air (50% RH) 0.004 0 0.003 7 0.003 6 0.003 8	<u>u<sub>Am</sub> (</u> Vacuum (0.1 Pa) 0.001 0 0.001 3	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.000 4	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4	Vacuum (0.01 Pa) 0.001 0 0.001 4	Vacuum (0.1 Pa) 0.001 0 0.001 3
Participant NPL NRC LNE INRIM PTB	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL	Air 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9	Air (50% RH) 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9	<u>u<sub>Am</sub> (</u> Vacuum (0.1 Pa) 0.001 0 0.001 3	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.000 4 0.001 0	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4	Vacuum (0.01 Pa) 0.001 0 0.001 4	Vacuum (0.1 Pa) 0.001 0 0.001 3
Participant NPL NRC LNE INRIM PTB NPL	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL	Air 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0	Air (50% RH) 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0	<u>u<sub>Am</sub> (</u> Vacuum (0.1 Pa) 0.001 0 0.001 3	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.000 4 0.001 0 0.001 0	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4	Vacuum (0.01 Pa) 0.001 0 0.001 4	Vacuum (0.1 Pa) 0.001 0 0.001 3
Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13)	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL	Air 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1	Air (50% RH) 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1	<u>u∆m</u> ( Vacuum (0.1 Pa) 0.001 0 0.001 3 0.001 0	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.000 4 0.001 0 0.001 0 0.001 0	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4	Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0	Vacuum (0.1 Pa) 0.001 0 0.001 3 0.001 0
Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13) NPL (summer	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon silicon	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL Si_NPL	Air 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1 0.004 1	Air (50% RH) 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1 0.004 1	$\begin{array}{c} & u_{\Delta m} \left( \right. \\ Vacuum \\ (0.1 \ Pa) \\ 0.001 \ 0 \\ 0.001 \ 3 \end{array}$	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.000 4 0.001 0 0.001 0 0.001 0 0.001 0	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4	Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0	Vacuum (0.1 Pa) 0.001 0 0.001 3 0.001 0
Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13) NPL (summer NRC	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon silicon silicon	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL Si_NPL Si_NPL Si_NPL	Air 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1 0.004 1 0.004 2	Air (50% RH) 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1 0.004 1 0.004 2	<u>u<sub>Am</sub> ( Vacuum (0.1 Pa) 0.001 0 0.001 3</u> 0.001 0	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.000 4 0.001 0 0.001 0 0.001 0 0.001 0 0.001 0 0.001 0 0.000 4	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4	Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0	Vacuum (0.1 Pa) 0.001 0 0.001 3 0.001 0
Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13) NPL (summer NRC INRIM	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon silicon silicon silicon	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL Si_NPL SS_INRIM	Air 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1 0.004 1 0.004 2 0.001 4	Air (50% RH) 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1 0.004 1 0.004 2 0.001 4	$\begin{array}{c} & u_{\Delta m} \left( \right. \\ Vacuum \\ (0.1 Pa) \\ 0.001 0 \\ 0.001 3 \end{array}$	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.000 4 0.001 0 0.001 0 0.001 0 0.001 0 0.001 0 0.001 0 0.000 4 0.000 4	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4 0.001 0 0.001 0	Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.001 0	Vacuum (0.1 Pa) 0.001 0 0.001 3 0.001 0 0.001 0
Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13) NPL (summer NRC INRIM PTB	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon silicon silicon stainless steel stainless steel	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL Si_NPL Si_NPL SS_INRIM SS_PTB	Air 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1 0.004 1 0.004 1 0.004 2 0.001 4 0.005 0	Air (50% RH) 0.004 0 0.003 7 0.003 6 0.003 8 0.003 9 0.004 0 0.004 1 0.004 1 0.004 1 0.004 2 0.001 4 0.005 0	$\begin{array}{c} \underline{u_{\Delta m}} (\\ \hline Vacuum \\ (0.1 Pa) \\ 0.001 0 \\ 0.001 3 \\ \end{array}$	Integral - Stack) Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.000 4 0.001 0 0.001 0 0.001 0 0.001 0 0.001 0 0.000 4 0.000 4 0.000 4 0.001 0	/ mg Vacuum (0.001 Pa) 0.001 0 0.001 4 0.001 0 0.001 0	Vacuum (0.01 Pa) 0.001 0 0.001 4 0.001 0 0.001 0	Vacuum (0.1 Pa) 0.001 0 0.001 3 0.001 0 0.001 0

**Table 4.** Average mass differences reported for the artefacts with standard uncertainties (k = 1).

<sup>b</sup>Uncertainty values were not supplied with the CMI results

				Average sorption / $\mu g \ cm^{-2}$					
Participant	Artefact	Artefact	Air (50 % RH) -	Air (50 % RH) -	Air (50 % RH) -	Air (50 % RH) -	Air (50 % RH) -		
	material	Identification	Vacuum (0.1 Pa)	Vacuum (0.01 Pa)	Vacuum (0.001 Pa)	Vacuum (0.01 Pa)	Vacuum (0.1 Pa)		
NPL	Pt/Ir	Pt_NPL	-0.054	-0.053	-0.053	-0.053	-0.055		
NRC	Pt/Ir	Pt_NPL	-0.026	-0.023	-0.021	-0.021	-0.018		
LNE	Pt/Ir	Pt_NPL		-0.045					
INRIM	Pt/Ir	Pt_NPL		-0.061					
PTB	Pt/Ir	Pt_NPL		-0.059					
NPL	Pt/Ir	Pt_NPL		-0.050					
NPL (spring 13)	silicon	Si_NPL	-0.035	-0.031	-0.034	-0.028	-0.028		
NPL (summer 13)	silicon	Si_NPL		-0.083					
NRC	silicon	Si_NPL	-0.083	-0.083	-0.083	-0.084	-0.084		
INRIM	stainless steel	SS_INRIM	-0.043	-0.040	-0.041	-0.036	-0.037		
РТВ	stainless steel	SS_PTB		-0.020					
CMI	stainless steel		-0.263	-0.296	-0.244	-0.235	-0.231		
eim									
					$u_s$ / $\mu g$ cm <sup>-2</sup>				
Participant			Air (50% RH) -	Air (50 % RH) -	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) -	Air (50 % RH) -	Air (50 % RH) -		
Participant			Air (50% RH) - Vacuum (0.1 Pa)	Air (50 % RH) - Vacuum (0.01 Pa)	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa)	Air (50 % RH) - Vacuum (0.01 Pa)	Air (50 % RH) - Vacuum (0.1 Pa)		
Participant NPL	Pt/Ir	Pt_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053	Air (50 % RH) - Vacuum (0.01 Pa) 0.053	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053	Air (50 % RH) - Vacuum (0.01 Pa) 0.053	Air (50 % RH) - Vacuum (0.1 Pa) 0.053		
Participant NPL NRC	Pt/Ir Pt/Ir	Pt_NPL Pt_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051		
Participant NPL NRC LNE	Pt/Ir Pt/Ir Pt/Ir	Pt_NPL Pt_NPL Pt_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051		
Participant NPL NRC LNE INRIM	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047 0.049	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051		
Participant Participant NPL NRC LNE INRIM PTB	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047 0.049 0.038	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051		
Participant NPL NRC LNE INRIM PTB NPL	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047 0.049 0.038 0.053	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051		
Participant Participant NPL NRC LNE INRIM PTB NPL NPL NPL (spring 13)	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047 0.049 0.038 0.053 0.024	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.024	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024		
Participant Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13) NPL (summer 13)	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon silicon	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL Si_NPL Si_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047 0.049 0.038 0.053 0.024 0.024	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.024	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024		
Participant Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13) NPL (summer 13) NRC	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon silicon silicon	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL Si_NPL Si_NPL Si_NPL	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047 0.049 0.038 0.053 0.024 0.024 0.024	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.024 0.024	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024 0.024		
Participant Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13) NPL (summer 13) NRC INRIM	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon silicon silicon stainless steel	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL Si_NPL Si_NPL SS_INRIM	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024 0.024 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047 0.049 0.038 0.053 0.024 0.024 0.024 0.024 0.024	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051 0.024 0.024 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.024 0.024 0.024	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024 0.024 0.024		
Participant Participant NPL NRC LNE INRIM PTB NPL NPL (spring 13) NPL (summer 13) NRC INRIM PTB	Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir Pt/Ir silicon silicon silicon stainless steel stainless steel	Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Pt_NPL Si_NPL Si_NPL Si_NPL SS_INRIM SS_PTB	Air (50% RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024 0.024 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.047 0.049 0.038 0.053 0.024 0.024 0.024 0.024 0.024 0.011 0.012	u <sub>s</sub> / μg cm <sup>-2</sup> Air (50 % RH) - Vacuum (0.001 Pa) 0.053 0.051 0.024 0.024 0.024	Air (50 % RH) - Vacuum (0.01 Pa) 0.053 0.051 0.024 0.024 0.024	Air (50 % RH) - Vacuum (0.1 Pa) 0.053 0.051 0.024 0.024 0.011		

**Table 5.** Average sorption values for the artefacts with standard uncertainties (k = 1).

<sup>c</sup>Uncertainty values were not supplied with the CMI results

				Average pressure / Pa				
Participant	Artefact material	Artefact Identification	Humidity range	Air (50 % RH) - Vacuum (0.1 Pa)	Air (50 % RH) - Vacuum (0.01 Pa)	Air (50 % RH) - Vacuum (0.001 Pa)	Air (50 % RH) - Vacuum (0.01 Pa)	Air (50 % RH) - Vacuum (0.1 Pa)
			(% RH)					
NPL	Pt/Ir	Pt_NPL	43 – 55	0.109 5	0.011 5	0.002 9	0.011 9	0.112 0
NRC	Pt/Ir	Pt_NPL	44 - 48	0.141 4	0.009 1	0.001 1	0.010 7	0.172 7
LNE	Pt/Ir	Pt_NPL	51 - 52		0.020 0			
INRIM	Pt/Ir	Pt_NPL	48 - 58		0.011 0			
РТВ	Pt/Ir	Pt_NPL	32 - 42		0.009 0			
NPL	Pt/Ir	Pt_NPL	42 - 74		0.025 8			
NPL (spring 13)	silicon	Si_NPL	39 - 56	0.142 6	0.018 1	0.001 1	0.012 5	0.119 2
NPL (summer 13)	silicon	Si_NPL	45 - 58		0.037 8			
NRC	silicon	Si_NPL	41 - 52	0.130 4	0.010 4	0.001 6	0.010 1	0.097 2
INRIM	stainless steel	SS_INRIM	47 – 65	0.106 7	0.010 0	0.000 9	0.010 5	0.096 7
РТВ	stainless steel	SS_PTB	32 - 42		0.009 0			
CMI <sup>d</sup>	stainless steel	SS_NPL		0.100 0	0.040 0	0.001 0	0.040 0	0.100 0

Table 6. Average pressure measured by the participants and range of relative humidity values during the measurements.

<sup>d</sup>Humidity data was not supplied by CMI and all values have been assumed to be 50 % RH

### 8 FIGURES



Figure 2. Change in mass difference between the NPL Pt-Ir artefacts measured at NPL and NRC relative to the initial mass difference at 0.1 Pa. The artefacts were cleaned before both sets of measurements and error bars represent the standard uncertainty (k = 1). Open markers represent previously published values.



Figure 3. Change in mass difference between the NPL silicon artefacts measured at NPL and NRC relative to the initial mass difference at 0.1 Pa. The artefacts were cleaned before the NPL spring 2013 measurements and error bars represent the standard uncertainty (k = 1). Open markers represent previously published values.



Figure 4. Change in mass difference between the INRIM stainless steel artefacts measured at INRIM and the stainless steel artefacts measured at CMI relative to the initial mass difference at 0.1 Pa. The artefacts were cleaned before the measurements and error bars represent the standard uncertainty (k = 1). Open markers represent previously published values.



**Figure 5.** Sorption values for the NPL Pt-Ir artefacts. The artefacts were cleaned before the INRIM, NPL 2012 and NRC measurements but not before the LNE, PTB and NPL 2013 measurements. Open markers represent previously published values. The uncertainty on the participants values ranged from 0.038  $\mu$ g cm<sup>-2</sup> to 0.053  $\mu$ g cm<sup>-2</sup> (*k* = 1)



Figure 6. Sorption values for the NPL silicon artefacts measured at NPL and NRC. Open markers represent previously published values. The artefacts were cleaned before the NPL spring 2013 measurements and the error bars represent the standard uncertainty (k = 1).



Figure 7. Sorption values for the INRIM stainless steel artefacts measured at INRIM (closed blue diamonds), PTB stainless steel artefacts measured at PTB (closed red square) and the stainless steel artefacts measured at CMI (closed green triangles). Open markers represent previously published values. The artefacts were cleaned before the measurements and the error bars represent the standard uncertainty (k = 1).