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Report on the evaluation of medium-term (6-month) storage in nitrogen compared with storage in air

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Zaccaria Silvestri (CNAM)**

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ABSTRACT

A study evaluating the benefits of storing stainless steel mass standards for 6-months in nitrogen compared with storage in air has been undertaken at three National Measurement Institutes. X-ray photoelectron spectroscopy results showed little difference between storage in air for 6-months and storage in nitrogen. Gravimetric weighing results showed excellent mass stability in the artefacts stored in air compared with significant mass gains in the artefacts stored in nitrogen.

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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 compare the effect of storing mass standards for a 6-month period in nitrogen with mass standards stored conventionally in air. The current definition of the unit of mass is realised by a cylinder of platinum-iridium alloy 39 mm in diameter and 39 mm high which is stored at the BIPM under a triple bell jar in air [1]. As a future fundamental constant definition of the kilogram will be realised in a vacuum [2], [3] improved stability of the mass artefacts used to provide traceability to the new definition may be possible if the artefacts are stored in a pure gas such as nitrogen between measurements instead of storage in air.

The institutes which participated in this work are given in table 1.

Table 1. List of participating institutes.

Participant	
Conservatoire national des arts et metiers	CNAM
National Physical Laboratory	NPL
Physikalisch-Technische Bundesanstalt	PTB

Previous work by Berry and Davidson [4] compared the stability of mass standards stored in argon gas and mass standards stored in air, in each case transferring the standards to vacuum for measurement. Both stainless steel and silicon artefacts were used for this work. They found that the repeated transfer of artefacts between argon and vacuum showed improved mass stability compared with the transfer of artefacts between air and vacuum. Fuchs *et al* [5] measured the long-term stability of PtIr and Au artefacts using X-ray Photoelectron Spectroscopy (XPS) after hydrogen and oxygen low-pressure plasma cleaning. After cleaning the artefacts were stored in air, argon or vacuum for a total of three years with XPS measurements performed every six to nine months. Similar levels of contamination were found on the surface of the artefacts stored in all three storage media with the exception of the oxygen plasma cleaned and vacuum stored artefacts which exhibited a larger gain in carbon contamination after cleaning.

2 EXPERIMENTAL PROCEDURE

2.1 MASS COMPARATORS AND SURFACE ANALYSIS EQUIPMENT

Details of mass comparators and surface analysis equipment used by each participating institute are given in Table 2.

Table 2. Mass comparators and surface analysis equipment used by the participants.

Participant	Mass Comparator	Surface Analysis Equipment
CNAM	Mettler-Toledo AT106	Thermal Desorption Spectrometry (TDS) [6]
NPL	Mettler-Toledo Mone 6 place mass comparator	Kratos Axis Ultra XPS apparatus
PTB	Sartorius AG CCL1007 mass comparator	None

The following parameters were used for the NPL XPS measurements.

Survey spectra in the range 1400 to -10 eV binding energy were taken at an emission angle of 0° to the surface normal from an area of each sample using an Al monochromated x-ray source operated at 15kV, 5mA emission. Analysis conditions used were 160eV pass energy, 1eV steps, 0.2 sec dwell per step and 1 scan.

The survey scans have been corrected using the latest NPL transmission function calibration and Average Matrix Relative Sensitivity Factors (AMRSFs) were applied. Three measurements were performed at three different locations on the surface of each stainless steel sample.

2.2 CNAM MEASUREMENTS

2.2.1 Artefact information

Four 100 g gravimetric artefacts in stainless steel (E0 class), manufactured by Häfner were used for this study. The artefacts were 48 mm in diameter and 8 mm thick (see Figure 1).



Figure 1. Pictures of the 100 g stainless steel artefacts.

In order to perform the gravimetric measurements, a 100 g reference mass in stainless steel conforming to the requirements specified by the International Organisation of Legal Metrology (OIML) [7] for an E2 class weight was chosen to eliminate the need for air buoyancy corrections. Table 3 presents information on the artefacts used for this work.

Table 3 - CNAM stainless steel artefacts used in this study.

Identification	Type	Storage Media	Surface area / cm ²
E2ref	OIML E2 stainless steel	Nitrogen	150.9
MB1	E0 100g stainless steel cylinder	Dry air	21.1
MP1	E0 100g stainless steel cylinder	Dry air	21.1
MB2	E0 100g stainless steel cylinder	Nitrogen	21.1
MP2	E0 100g stainless steel cylinder	Nitrogen	21.1

2.2.2 Storage vessel information

Figure 2 shows an example of a storage vessel used at CNAM for the storage of mass standards in air or nitrogen. In order to control the pressure and to limit gas consumption, a mass flow controller was used and set to give a flow of about 15 ml min⁻¹.

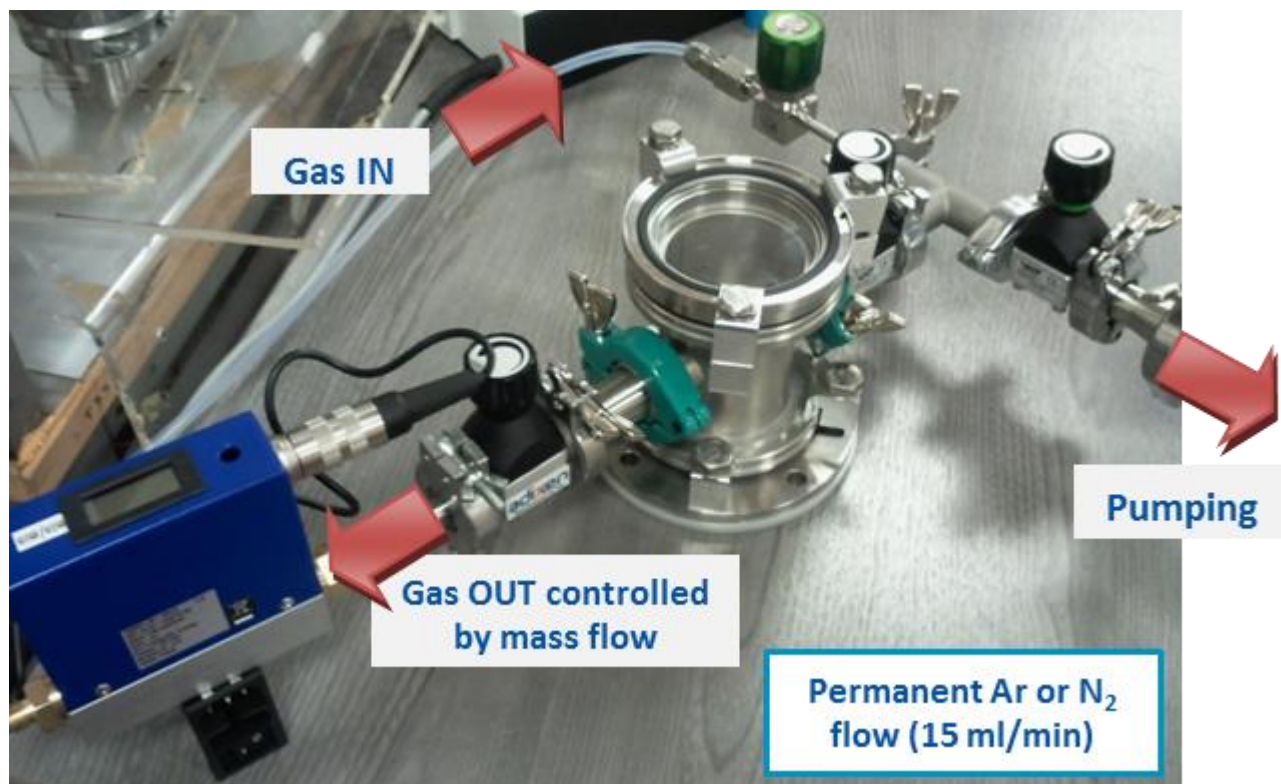


Figure 2 - Inert gas/vacuum compatible storage vessel used in this study at CNAM.

Two identical vacuum compatible storage vessels were used to store the artefacts. The vessels do not have clamps to secure the artefacts, instead the weights are placed on acid free paper and the vessels stored in a clean room.

2.2.3 Cleaning procedure

For this study, two cleaning methods have been tested and compared:

- Air-plasma cleaning;
- Nettoyage-lavage BIPM method [8].

It was not possible to use Hydrogen-plasma at CNAM due to safety restrictions. Air-plasma cleaning was selected as an effective replacement technique. Before comparing the two cleaning methods, a pre-clean (ultrasonic bath with high purity ethanol for 30 minutes) was performed and then an analysis undertaken of the contamination on the surface using the TDS device [6]. The reference E2 mass was not cleaned.

2.2.4 CNAM measurement procedure

The experimental measurement protocol is presented in Table 4. All artefacts were pre-cleaned before the gravimetric measurements except the reference mass used for the comparison.

Table 4. Experimental protocol for the 6-month storage study.

<i>Date</i>	<i>Mass</i>	<i>Operation</i>	<i>Operation 2</i>	<i>Measurements</i>
07/10/2014	MP1	Pre-cleaning 1 : Ultrasonic bath with ethanol duration 30 min	Air-plasma 1 hour	TDS
08/10/2014	MP2		Nettoyage-lavage BIPM	
09/10/2014	MB1			
10/10/2014	MB2			
15/10/2014	MP1	Pre-cleaning 2 : Ultrasonic bath with ethanol duration 30 min	Air-plasma 1 hour	Gravimetric A1
	MP2		Nettoyage-lavage BIPM	
	MB1			
	MB2			
16/10/2014	MP1	Storage in air lab. 1 hour		Gravimetric A2
	MP2			
	MB1			
	MB2			
16/10/2014	MP1	Storage in vessel with over pressured dry air		
	MP2	Storage in vessel with over pressured N ₂		
	MB1	Storage in vessel with over pressured dry air		
	MB2	Storage in vessel with over pressured N ₂		
18/05/2015	MP1	Out of storage vessels in Air lab.		Gravimetric B1
	MP2			
	MB1			
	MB2			
22/05/2015	MP1	Storage in air		Gravimetric B2
	MP2			
	MB1			
	MB2			
27/05/2015	MP1	Storage in air		Gravimetric B3
	MP2			
	MB1			
	MB2			

Just after the gravimetric measurements (A2), the two artefacts cleaned with the air-plasma method were placed in the two storage vessels: the MP1 artefact in the air vessel and the MP2 artefact in the nitrogen vessel. The same procedure was applied to the two artefacts cleaned with the nettoyage-lavage BIPM cleaning method: MB1 artefact was placed in the air vessel (same vessel as MP1) and MB2 placed in the nitrogen vessel (same vessel as MP2). The pressure in the storage vessels was set to about 1000 Pa above atmospheric pressure.

The four artefacts were stored for 7 months (due to technical problems in the clean room, measurements after 6 months could not be performed). At the end of the 7 month storage period the artefacts were

removed from the storage vessels and weighed against the reference E2 mass. Measurements were carried out immediately after removing the artefacts from storage (gravimetric B1), four days after (gravimetric B2) and 9 days after (gravimetric B3).

2.3 NPL PROCEDURE

2.3.1 Artefact information

Six stainless steel artefacts were used for these measurements; two of them were stainless steel kilograms of a shape prescribed by the OIML [7] and the remaining four were circular stainless steel surface samples (diameter 10 mm thickness 1 mm) which were used for the XPS measurements. Additionally two stainless steel reference mass standards traceable to the international prototype kilogram (IPK) were used to monitor any mass changes in the two test kilogram artefacts. Information on the artefacts is given in table 2.

Table 5. NPL gravimetric artefacts and surface samples used in this study.

Identification	Type	Storage Media	Volume at 20 °C / cm ³	Surface area / cm ²
61D	Cylindrical kilogram (reference standard)	Air	124.279 7	134.7
61DD	Cylindrical kilogram (reference standard)	Air	124.279 2	134.7
59	OIML shape kilogram (test artefact)	Air	127.291 3	150.9
59D	OIML shape kilogram (test artefact)	Nitrogen	127.292 4	150.9
Sample 2	Stainless steel surface sample	Air	0.127 6	2.2
Sample 3	Stainless steel surface sample	Air	0.127 6	2.2
Sample 4	Stainless steel surface sample	Nitrogen	0.127 6	2.2
Sample 5	Stainless steel surface sample	Nitrogen	0.127 6	2.2

2.3.2 Storage vessel information

Two nominally identical storage vessels were used to store the artefacts (59 and 59D) and the steel surface samples (2, 3, 4 and 5) as shown in Figure 3. The vessels are constructed from vacuum compatible components allowing them to be evacuated or filled with an inert gas and the clamps used to secure the gravimetric artefacts are manufactured from Polyether ether ketone (PEEK) material. Small glass jars are used to store the surface samples within the storage vessels and these jars are also used to transport them to the XPS apparatus.



Figure 3. NPL inert gas/vacuum compatible storage vessel used in this study.

2.3.3 Cleaning procedure

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 reference standards were not cleaned). The washing procedure was performed in a clean dust free environment.

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 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.

A similar procedure was used to clean the surface samples except they were rinsed in a beaker of distilled water after the solvent cleaning instead of using the squeeze bottle.

2.3.4 NPL Measurement procedure

Initially the masses of artefacts 59 and 59D were determined by gravimetric weighing against the reference standards (61D and 61DD) and XPS measurements were performed on the four surface samples. All the artefacts and surface samples were then cleaned using the procedure described in 2.3.3. XPS measurements were then repeated on the surface samples and weighing of the gravimetric artefacts against the reference standards was performed after allowing a period of 48 hours for the artefacts to stabilise post cleaning.

Gravimetric artefact number 59 and surface samples 2 and 3 were then loaded into one storage vessel and artefact number 59D and surface samples 4 and 5 were loaded into the other storage vessel. The storage vessels were then sealed and evacuated to remove the air within them. The storage vessel containing gravimetric artefact 59 and samples 2 and 3 was then filled with dry compressed air and the other storage vessel (59D and samples 4 and 5) was filled with dry nitrogen to a pressure approximately 10 000 Pa above atmospheric pressure. Both sets of artefacts were then left within their storage vessels for a 6 month period. The pressure within the vessels was found to drop by between 100 Pa day⁻¹ and 400 Pa day⁻¹ and so it was necessary to add extra air or nitrogen gas to the storage vessels every month to take both vessels back up to 10 000 Pa above atmospheric pressure. At the end of the 6 month storage period the gravimetric artefacts were removed from the storage vessel and weighed again against the reference kilograms after allowing a 24 hour period for the artefacts to stabilise. XPS measurements were also made on all four surface samples.

2.4 PTB PROCEDURE

2.4.1 Artefact Information

Six stainless steel cylindrical kilogram artefacts were used in this study in addition to one stainless steel kilogram reference standard traceable to the IPK that was used to monitor any mass changes in the test artefacts. Information on the artefacts is given in Table 6

Table 6. PTB gravimetric artefacts used in this study

Identification	Type	Storage Media	Volume @ 20 °C / cm ³	Surface area / cm ²
1 kg H _A E	OIML shape kilogram (reference standard)	Air	124.417 2	150.9
1 kg P1	Cylindrical kilogram (test artefact)	Air	124.834	138.7
1 kg P2	Cylindrical kilogram (test artefact)	Air	124.841	138.7
1 kg P3	Cylindrical kilogram (test artefact)	Nitrogen	124.839	138.7
1 kg P4	Cylindrical kilogram (test artefact)	Nitrogen	124.840	138.7
1 kg P5	Cylindrical kilogram (test artefact)	Vacuum	124.841	138.7
1 kg P6	Cylindrical kilogram (test artefact)	Vacuum	124.840	138.7

2.4.2 Storage vessel information

Four storage vessels manufactured by Sartorius AG were used to store the two test artefacts P3 and P4 in nitrogen gas and the two test artefacts P5 and P6 in vacuum. The two artefacts P1 and P2 were stored in air under glass bell jars.

2.4.3 Cleaning procedure

The artefacts were cleaned twice with ethanol (purity of at least 98 %) in an ultrasonic bath for fifteen minutes. After the ethanol cleaning, the artefacts were cleaned in distilled water for about 5 minutes. The remaining water drops were removed by means of clean lens tissue. The reference mass was not cleaned.

2.4.4 PTB measurement procedure

The six stainless steel cylinders P1 to P6 were compared with the reference standard before and after the cleaning in order to determine the mass loss due to the cleaning procedure (Tables 12 to 14). The mass determination was performed after leaving the artefacts to stabilise for seven days after cleaning.

After completion of the mass determination, the stainless steel cylinders P1 and P2 were stored in air under bell jars. The storage containers for the storage of the cylinders P3 to P6 were evacuated. The cylinders P5 and P6 were transferred into their respective storage containers and stored in vacuum for six months. During this period, the pressure in the storage containers was stabilised to be in a pressure range between 0.1 Pa and 0.5 Pa. The storage containers for the storage of cylinders P3 and P4 were

filled with nitrogen and the cylinders P3 and P4 were then transferred into their respective storage containers. During the storage period of six months, the nitrogen pressure was measured and kept at a pressure of about 1.1×10^5 Pa.

At the end of the six month storage period the six stainless steel cylinders were transferred into the mass comparator and compared again in air with the reference standard after allowing 48 hours for them to stabilise.

3 RESULTS AND DISCUSSION

3.1 CNAM RESULTS AND DISCUSSION

The changes in mass of the CNAM artefacts after applying two cleaning methods and then storing them in dry air or nitrogen gas for 7-months are given in Table 7 and Table 8 respectively along with the calculated standard uncertainties in the measurements. After stabilisation of the artefacts, air-plasma cleaning removed between 50 μg and 60 μg of mass from the surface of artefacts MP1 and MP2. In contrast the BIPM cleaning method added between 60 μg and 86 μg of mass to the surface of artefacts MB1 and MB2. After storage for 7-months all of the artefacts decreased in mass relative to the reference mass. The mass decrease was more pronounced for the nettoyage-lavage cleaned artefacts (-38 μg and -30 μg) compared with the air-plasma cleaned artefacts (-1 μg and -16 μg). The most likely explanation for the relatively large mass losses in the air and nitrogen stored artefacts previously cleaned by the BIPM cleaning method was due to a loss of volatile compounds from the surface of the artefacts formed during cleaning. This was supported by the mass gains observed in these artefacts after application of the BIPM cleaning method suggesting that the cleaning method had added contamination to the surface of the artefacts. If the contamination added to the surface during the BIPM method cleaning was volatile then a gradual desorption from the surfaces of the artefacts during the 7-month storage period would be expected. Storage for 7 months in nitrogen seemed to have no advantage in comparison to dry air storage.

Table 7. Results of the gravimetric weighing of the air stored artefacts MP1 (plasma cleaned) and MB1 (BIPM cleaned).

Process	Air stored / Plasma cleaned artefact (MP1)	Standard uncertainty	Air stored / BIPM cleaned artefact (MB1)	Standard uncertainty
Mass change due to cleaning / μg	- 51	5	+ 60	5
Mass change due to storage for 7-months / μg	-1	5	- 38	5

Table 8. Results of the gravimetric weighing of the nitrogen stored artefacts MP2 (plasma cleaned) and MB2 (BIPM cleaned).

Process	Nitrogen stored / Plasma cleaned artefact (MP2)	Standard uncertainty	Nitrogen stored / BIPM cleaned artefact (MB2)	Standard uncertainty
Mass change due to cleaning / μg	- 60	5	+ 86	5
Mass change due to storage for 7-months / μg	- 16	5	- 30	5

3.2 NPL RESULTS AND DISCUSSION

3.2.1 Gravimetric measurements

The changes in mass of the gravimetric artefacts 59 and 59D measured relative to the reference standards as a result of cleaning them and then storing them in air or nitrogen for 6-months are given in Table 9 along with the calculated standard uncertainties in the measurements. The results show that the air stored artefact has lost about 7 μg of mass after storing it for 6-months which could have been due to additional volatile materials remaining after cleaning that subsequently evaporated from the surfaces of the artefact. The nitrogen stored artefact gained about 22 μg in mass after storage for 6-months. It seems unlikely that the nitrogen in the storage vessel was directly responsible for the mass gain observed in the artefact particularly as the XPS results (see 3.2.2) did not show any significant difference between the two storage environments. The most plausible explanation is that contamination was transferred to the artefact during the transfer process to/from the storage vessel or material was transferred to the artefact from the clamps used to secure the artefact within the vessel.

Table 9. Results of the gravimetric weighing of artefacts 59 and 59D

Process	Air stored artefact (59)	Standard uncertainty	Nitrogen stored artefact (59D)	Standard uncertainty
Mass change due to cleaning / μg	- 100	2	-130	2
Mass change due to storage for 6-months / μg	-7	2	+22	2

3.2.2 XPS measurements

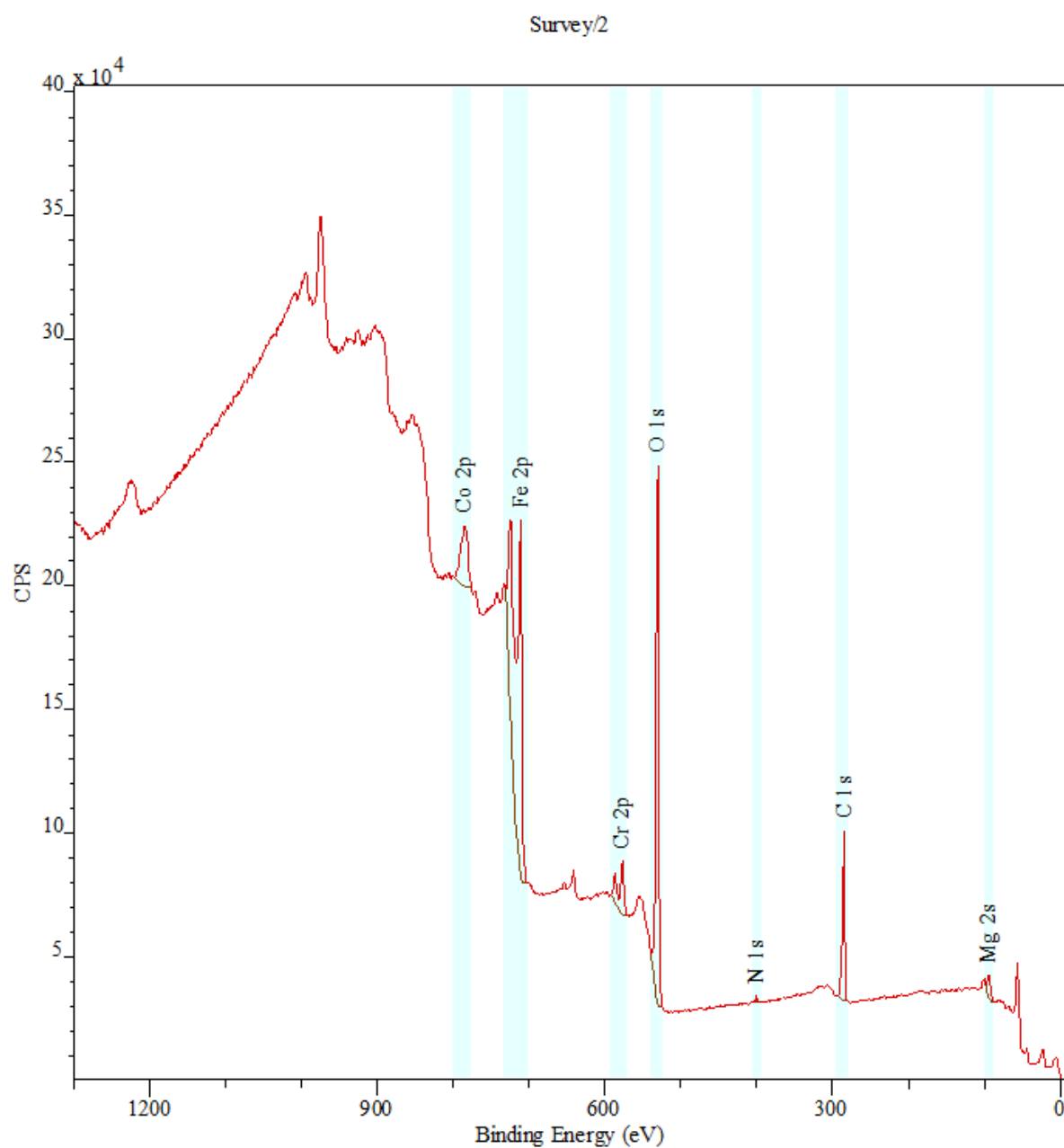
The elements on the surface of the four samples identified from the XPS survey spectra and their average atomic percentages measured at an angle of 0° to the surface normal after the samples were cleaned and then stored for 6-months in air or nitrogen are listed in Table 10 and Table 11 respectively. Typical XPS survey scans performed on sample 2 directly after cleaning and after storage in air for 6-months are shown in Figure 4 and Figure 5 respectively. XPS survey scans on the nitrogen stored sample 5 directly after cleaning and after storage in nitrogen for 6-months are shown in Figure 6 and Figure 7 respectively. All samples showed a small increase in carbon contamination of between 1.4 % and 3.9 % after storing them for 6-months but there was no significant difference between the air-stored samples and the nitrogen stored samples. This suggested that the growth of carbonaceous contamination observed on the samples was independent of the gas used to store them. The contamination could have formed as a result of outgassing of material from the walls of the vessels used to store the samples or it may have formed during the periods when the samples were exposed to laboratory air during the transfers to/from the XPS apparatus to the storage vessels.

Table 10. Average atomic percentages measured by XPS after cleaning

Sample	Area Quantification						
	O1 S	C1 S	N1 S	Co 2P	Fe 2P	Mg 2S	Cr 2P
Air stored sample 2	41.3	27.6	0.4	2.3	15.8	10.3	2.3
Air stored sample 3	39.7	31.2	0.6	2.2	13.7	10.8	1.9
N2 stored sample 4	39.2	28.2	0.5	2.4	17.1	10.2	2.5
N2 stored sample 5	41.3	28.3	0.6	2.2	14.8	10.3	2.6

Table 11. Average atomic percentages measured by XPS after storage for 6 months

Sample	Area Quantification					
	O1 S	C1 S	N1 S	Co 2P	Fe 2P	Cr 2P
Air stored sample 2	39.9	31.6	0.5	2.2	14.2	9.5
Air stored sample 3	40.0	32.6	0.6	2.1	13.3	9.8
N2 stored sample 4	38.3	31.7	0.6	2.2	15.2	10.2
N2 stored sample 5	39.2	32.1	0.7	2.2	13.9	9.7

**Figure 4.** XPS survey scan of the air stored stainless steel sample after cleaning.

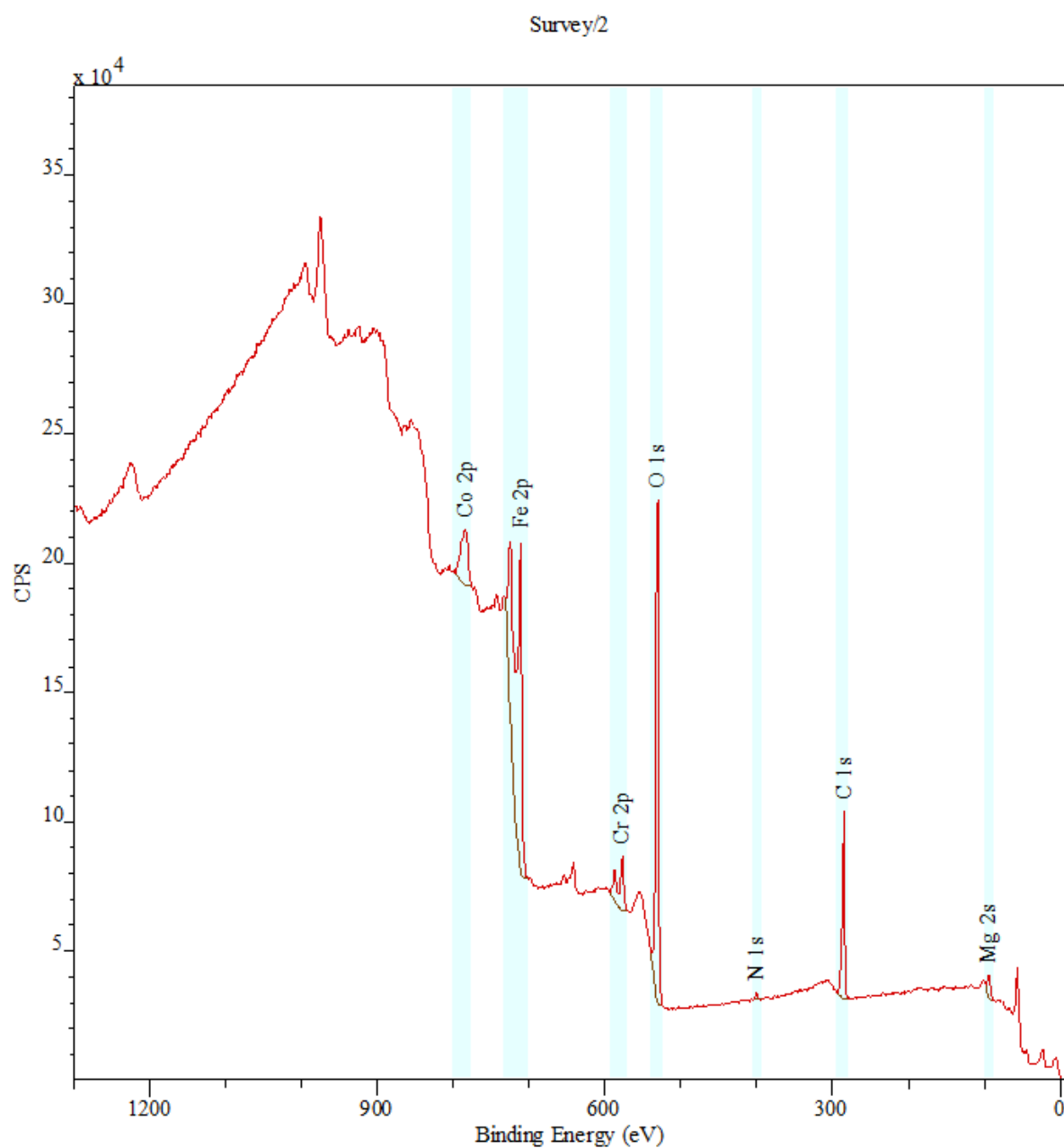


Figure 5. XPS survey scan of the stainless steel sample after storage in air for 6-months.

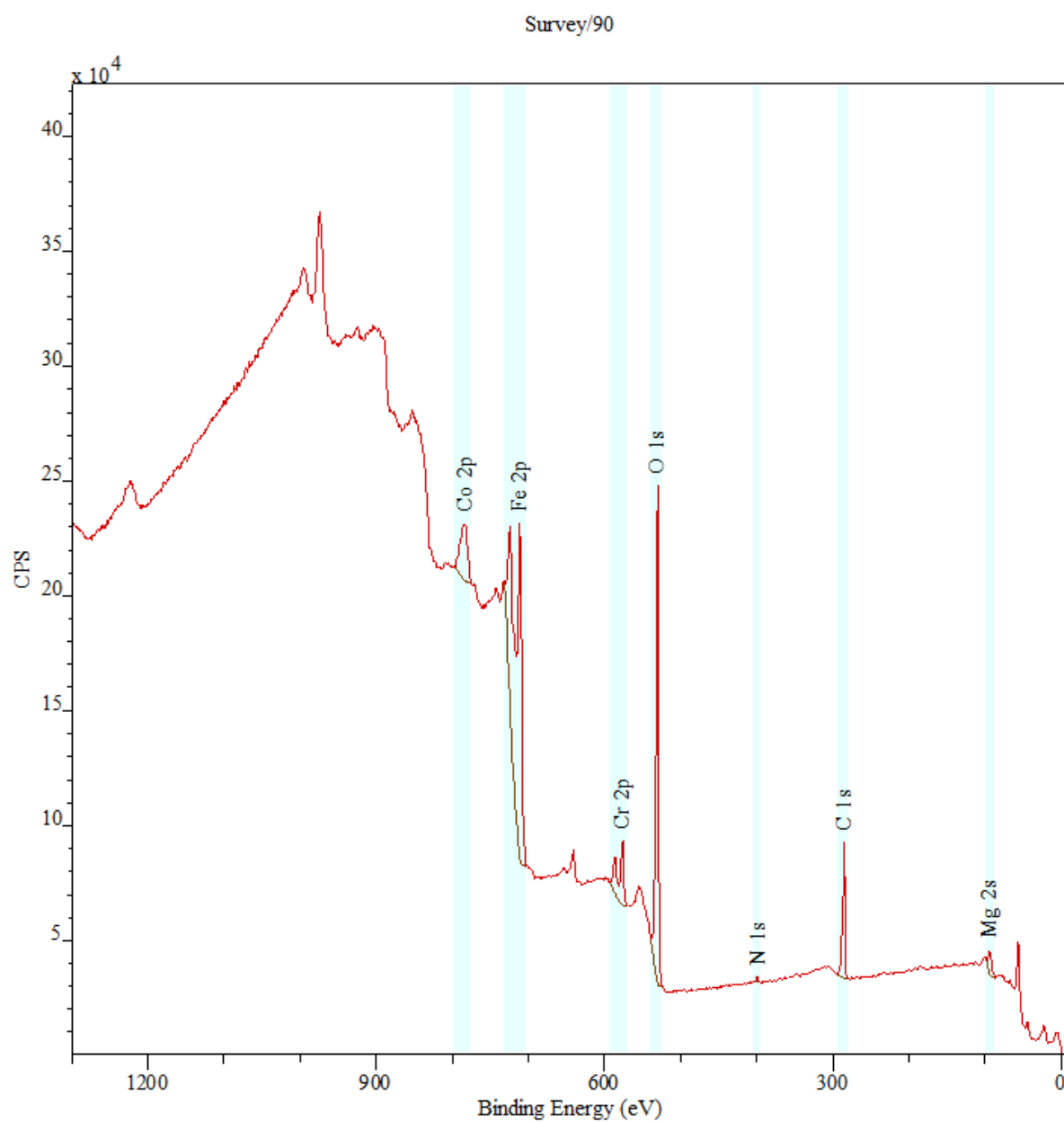


Figure 6. XPS survey scan of the nitrogen stored stainless steel sample after cleaning

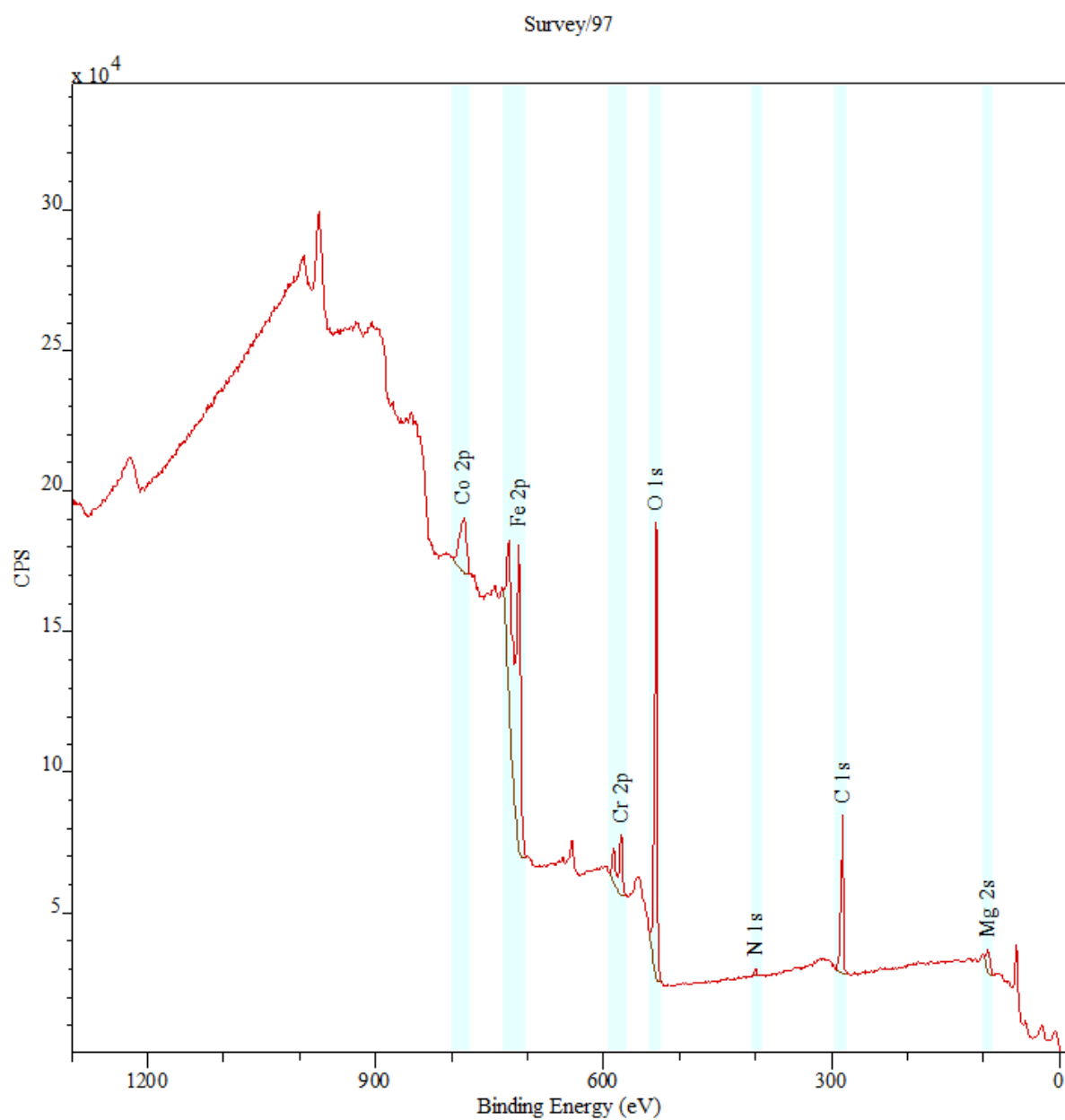


Figure 7. XPS survey scan of the stainless steel sample after storage in nitrogen for 6-months.

3.3 PTB RESULTS AND DISCUSSION

The change in mass of the PTB air stored artefacts P1 and P2, nitrogen stored artefacts P3 and P4 and vacuum stored artefacts P5 and P6 as a result of cleaning and then storing them for 6-months are given in Table 12, Table 13 and Table 14 respectively along with the calculated standard uncertainties in the measurements. All the artefacts lost mass ranging from 12.9 μg to 34.5 μg as anticipated after cleaning. The air stored artefacts showed excellent stability over the 6-month period with no significant changes in mass of either of the two artefacts. Both the nitrogen stored artefacts and the vacuum stored artefacts showed similar mass gains ranging from 18.7 μg to 28.6 μg after storage for 6-months. Although it was possible that the nitrogen and vacuum environments were responsible for the observed mass gains, the use of the same specification of storage vessel for both media compared with the use of bell jars for the air stored artefacts suggests that the likely reason for the mass gain was due to material being transferred to the artefacts as a result of storing them within the storage vessels. This material could have been transferred to the artefacts from the clamps used to secure them within the vessels or the material could have been transferred during the process of evacuating the vessels or filling them with gas. In the latter case the contamination could have come from the pipework connected to the vacuum pump, the pipework connected to the gas supply or may simply have been transferred from the walls of the storage vessels.

Table 12. Results of the gravimetric weighing of the air stored artefacts P1 and P2

Process	Air stored artefact (P1)	Standard uncertainty	Air stored artefact (P2)	Standard uncertainty
Mass change due to cleaning / μg	- 34.5	2	-23.5	2
Average mass change due to cleaning / μg	- 29			
Mass change due to storage for 6-months / μg	- 1.7	2	+1.2	2
Average mass change due to storage / μg	- 0.2			

Table 13. Results of the gravimetric weighing of the nitrogen stored artefacts P3 and P4

Process	Nitrogen stored artefact (P3)	Standard uncertainty	Nitrogen stored artefact (P4)	Standard uncertainty
Mass change due to cleaning / μg	- 34.3	2	-27.5	2
Average mass change due to cleaning / μg	- 30.9			
Mass change due to storage for 6-months / μg	23.0	2	18.7	2
Average mass change due to storage / μg	20.8			

Table 14. Results of the gravimetric weighing of the vacuum stored artefacts P5 and P6

Process	Vacuum stored artefact (P5)	Standard uncertainty	Vacuum stored artefact (P6)	Standard uncertainty
Mass change due to cleaning / μg	- 12.9	2	-13.4	2
Average mass change due to cleaning / μg	- 13.1			
Mass change due to storage for 6-months / μg	28.6	2	22.7	2
Average mass change due to storage / μg	25.7			

4 CONCLUSIONS

XPS results showed no significant difference in carbonaceous growth on the surface of stainless steel samples stored in nitrogen for 6 months compared with those stored in air for six months. The XPS results suggest that there is no advantage in storing artefacts in nitrogen compared with conventional air storage over timescales of 6-months or shorter.

The CNAM gravimetric results on stainless steel artefacts did not show any significant difference between storing the artefacts in dry air and storing them in nitrogen gas. CNAM did find significant differences in the mass change of stainless steel artefacts after applying an air-plasma cleaning method compared with the nettoyage-lavage BIPM cleaning method. The CNAM results showed the air-plasma technique to be an effective cleaning procedure for stainless steel artefacts in contrast to the nettoyage-lavage BIPM method which resulted in contamination being added to the artefacts. The artefacts cleaned using the BIPM method subsequently lost between 30 μg and 38 μg of the 60 μg to 86 μg of contamination that had formed as a result of the applied cleaning process. . This mass loss was attributed to the contamination formed during the nettoyage-lavage BIPM method cleaning being volatile in nature and thus it desorbed from the surface of the artefacts during the 7-month storage period.

Both the NPL and PTB gravimetric results on stainless steel artefacts stored in air for 6-months did not show any significant gain in mass. This contrasted with the nitrogen stored artefact at NPL and the nitrogen and vacuum stored artefacts at PTB all of which showed significant mass gains of around 20 μg over this period. It is unlikely that this contamination was due to the nitrogen gas environment as the stainless steel samples used in the NPL XPS measurements did not show any significant evidence of carbonaceous contamination and the percentage of hydrocarbons found on the surface was similar to the air stored samples. A possible explanation for the mass gain could be due to the contact made by the clamps used to secure the artefacts within the storage vessels. Contamination on the surface of the clamps or material from the clamps themselves could be transferred onto the surface of the artefacts. Alternatively the contamination could have been introduced during the procedure used to evacuate the vessels or fill them with gas. Therefore further investigations are required in order to reduce or eliminate this potential source of contamination. Alternatively if the artefacts are not required to be transported between measurements then storing them under bell jars in nitrogen or another inert gas may offer the same excellent stability observed in the air stored artefacts at PTB.

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