NPL

National Physical Laboratory

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Guide to the Storage of Primary Mass Standards

EXPERT

TECHNICAL LEVEL

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Front cover image: *Equipment for the long-term storage of mass standards in inert gas. (Source: NPL).*

Guide Information

What is it about?

This guide presents information that will assist with maintenance of the SI unit of mass at National Measurement Institutes. The information presented is primarily aimed at establishing the technical infrastructure and procedures that will allow the robust maintenance of a national mass scale following the redefinition of the kilogram. This guide gives information on the evaluation of different storage methods with a view to optimising the medium- and long-term stability of primary mass standards.

Who is it for?

This guide has been produced to provide information primarily for National Measurement Institutes involved with the realization and/or dissemination of the SI unit of mass.

What is its purpose?

The purpose of this guide is to provide the information necessary to establish the technical infrastructure and procedures to allow for the maintenance of a national mass scale following the redefinition of the kilogram.

What is the prerequisite knowledge?

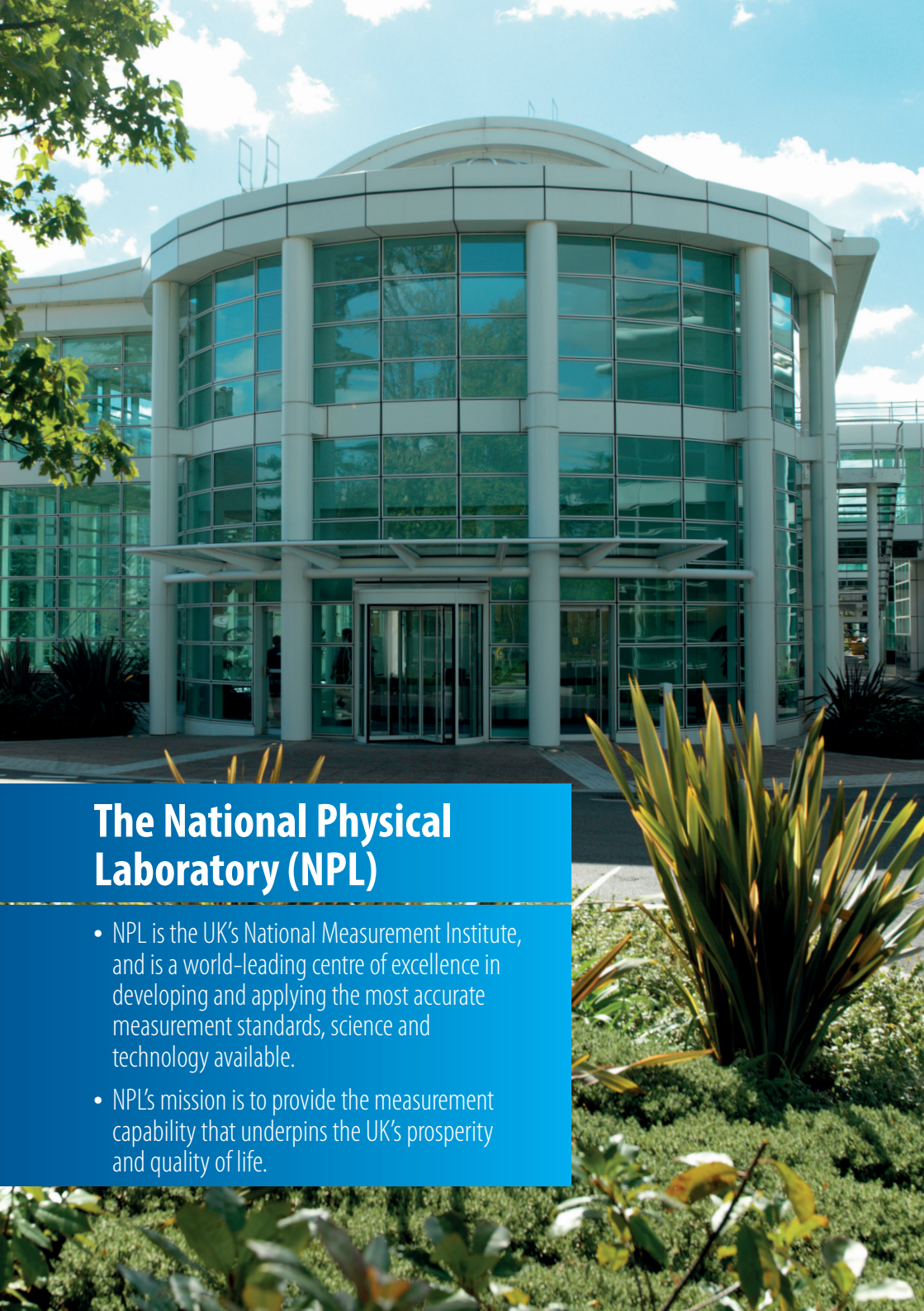
The guide is intended for users with a knowledge of the maintenance and dissemination of the SI unit of mass.

Key to icons:

Need to know

Good to know

Checklist



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- NPL is the UK's National Measurement Institute, and is a world-leading centre of excellence in developing and applying the most accurate measurement standards, science and technology available.
- NPL's mission is to provide the measurement capability that underpins the UK's prosperity and quality of life.

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Partners

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- Mettler-Toledo GmbH, *Switzerland*
- Häfner Gewichte GmbH, *Germany*
- Technische Universitat Ilmenau, *Germany*

Chapter I

Introduction

- Current definition of the kilogram
- Weight stability

Current definition of the kilogram

Currently, the SI unit of mass is defined as being the mass of the international prototype kilogram, IPK or just $\mathcal{K}$. $\mathcal{K}$ is a physical artefact made from an alloy of platinum and iridium (Pt-Ir), which is stored at the Bureau International des Poids et Mesures (BIPM) under bell jars containing filtered ambient air.

Traceability to $\mathcal{K}$ is maintained by comparison with BIPM working standards against which the national prototype copies are weighed on mass comparators in ambient air. Periodically (about every 40 years) all the national prototype copies are compared directly with the IPK.

Redefinition

Work is underway to replace the current kilogram with something more stable. There are currently two feasible ways to realise a kilogram with relation to a fundamental constant of nature. The first is the watt balance project and the second is the Avogadro experiment. Both of these methods operate under vacuum.

Traceability for the redefined kilogram will therefore require both the weighing of standard weights in a vacuum and the transfer of standards between vacuum and air.

The watt balance

The watt balance defines the mass of a kilogram in terms of a current and a voltage. Since current and voltage units are defined in terms of a fundamental physical constant (the Planck constant), this will provide an alternative definition of the kilogram in terms of an absolute constant.

The Avogadro experiment

This technique measures the mass of a single silicon atom by counting the number of atoms in a macroscopic sample (a highly polished sphere). Direct counting of sufficient atoms is out of the question, but the task can be done indirectly by measuring the volume occupied by an atom in a perfect crystal, measuring the volume of the crystal and dividing one by the other. If the mass of the crystal is also measured the mass of a silicon atom can be calculated and the Avogadro constant derived.

Weight stability

Weight stability in air

One of the main reasons for the redefinition of the kilogram is the perceived instability of $\mathcal{K}$ and the national prototype copies. Extensive studies have been carried out on the stability of both Pt-Ir and stainless steel (SS) mass standards stored in ambient air, this work being summarized by Davidson [1].

Both Pt-Ir and SS weights usually gain weight with time, this being primarily due to the accretion of hydrocarbon contamination from the atmosphere. Typical mass changes for Pt-Ir mass standards are in the range 1 µg year^{-1} to 3.5 µg year^{-1}.

Weight stability in vacuum

On being transferred from air to vacuum, mass standards typically lose weight. Davidson [2] has reported on the stability of a Pt-Ir mass standard over a period of thirty days after exposure to vacuum. An exponential mass loss of 2 µg over the first thirty days was observed, after which the mass was stable.

Contrary to this, there are also reports [3,4] of mass standards gaining weight in vacuum. This can be attributed to contamination by sorption of oil originating, for example, from vacuum pumps. The use of inverted magnetron gauges and ionisation gauges at higher pressures can also lead to increased contamination from oil vapour within the vacuum chamber. This suggests that great care should be taken in the choice of equipment used in the vacuum chamber itself.

Transfer of weights from air to vacuum

The mass stability of weights repeatedly transferred from vacuum to air has been compared to the stability of weights maintained in vacuum and air. Davidson [2] reports that both Pt-Ir and SS artefacts exhibited an exponential mass gain with time, and both gained more mass than those stored permanently in air or vacuum. This is attributed to the exposure to vacuum removing the loosely bound over-layers of water molecules from the surface, thus allowing a higher probability of accreting hydrocarbon contamination before the layer of water molecules is restored.

However, Picard and Fang's similar experiments on Pt-Ir, SS and silicon artefacts [5] did not show any evidence of mass gain from contamination when they were cycled between air and vacuum. This they attributed to the short period of time the artefacts were kept in air and the fact that the artefacts were not removed from the clean balance case during the experiments.

Storage in vacuum

Therefore, transferring weights between vacuum and air has the potential to increase the amount of contamination, and thus increases the instability of the mass of the artefacts. Ideally artefacts would be stored and transferred between the different watt balance and Avogadro experiments in a vacuum. However, this will introduce excessive costs and complexities into the requirement to transfer weights.

Storage in inert gas

The alternative to storing artefacts in air, with the associated instabilities, or in vacuum, with its associated costs and complexity, is to store the artefacts in an inert gas, for example argon or nitrogen.

This would provide the following benefits:

- The artefacts will be exposed to fewer potential sources of contamination.
- The transfer chamber and transfer mechanism will be less complicated and costly.
- The weight of the storage vessel is reduced.
- To prevent the leaking in of air contaminated with hydrocarbons, the vessel can be maintained at a positive pressure with relation to ambient.

An additional benefit is the possibility of 'humidifying' the gas within the storage vessel. This will maintain a surface layer of water on the artefact inhibiting contamination of the surface.

Chapter II

Weight storage

- Requirements for weight storage
- Requirements for storage vessels

Requirements for weight storage

The redefinition of the kilogram and its associated requirement for comparison between the various watt balance and Avogadro experiments, together with the subsequent maintenance and dissemination of mass traceability, provides a requirement for apparatus to:

- Transfer mass standards between vacuum mass comparators and watt balance apparatus.
- Transport weights between National Measurement Institutes (NMIs) to allow international comparisons providing validation of vacuum mass comparator performance, watt balance performance, and traceability to the existing physical artefact, $\mathcal{K}$.
- Store weights in optimal conditions in both the short and medium term (up to 10 years). This will provide on-going traceability for mass between primary realisations of the kilogram.

Requirements for storage vessels

The majority of artefacts transferred or stored will be 1 kilogram standards of cylindical, or possibly spherical construction. The storage vessel will need to accommodate such artefacts within a sealed environment of either inert gas or vacuum. In addition, the storage vessel must be readily transportable and allow for the transfer of the weight between the vessel and the apparatus without exposure to ambient air. This will require the use of a glove box and/or load-lock mechanism attached to the apparatus.

Different NMIs will have different apparatus and different means of transferring artefacts from the apparatus to the storage vessel. Therefore the storage vessels used for international comparisons must be compatible with the different apparatus.

Chapter III

Equipment

- Current apparatus
- In-house apparatus
- Commercial apparatus
- Load-lock

Current apparatus

There are two primary types of apparatus currently used for the storage and transfer of mass artefacts in vacuum or inert gas. These are custom, in-house constructed storage vessels and commercially available storage vessels and transfer apparatus.

Figure 1: *Inert-gas storage vessels as used at NPL.*

In-house apparatus

Traditionally, in-house constructed storage vessels are based around conventional vacuum components, and incorporate custom plastic inserts made from PTFE or PEEK for supporting the artefacts. To transfer the artefact a glove box mounted on the apparatus is used.

Figure 2: *NPL glove box mounted on load-lock assembly.*

However, the use of a glove box precludes the transfer of artefacts directly from vacuum to vacuum. This is because the glove box transfer is carried out in an inert gas.

The majority of these storage vessels utilise vacuum clamps to seal the vessel that must be unscrewed/tightened when transferring the weights. Frictional wear of the threads of these clamps can potentially introduce particles into the glove box. A recommendation is that the vacuum clamps be replaced by a single clamp that requires only one bolt, thus reducing the amount of potential contamination as well as the time taken to remove the vessel lid. The use of nylon for the construction of the clamps will reduce the possibility of particulate contamination but does not allow as effective a long-term seal, since nylon is known to creep over time.

Commercial apparatus

The primary advantage of the commercial storage vessels and transfer apparatus is that it permits the transfer and storage of artefacts in either vacuum or an inert gas. Both Sartorius and Mettler Toledo provide storage/transfer vessels, although their use can be limited to compatible mass comparators.

In addition, the height of these vessels is typically greater than that of other storage vessels, and the lack of a viewing window precludes inspection, which may be required in customs and airport security. Both of these are disadvantages when transferring the weights between NMIs.

Load-lock

With the use of a load-lock assembly the comparator can remain under vacuum whilst a weight is transferred. The main chamber is connected via a gate valve to a load-lock which permits the loading/unloading of the weight without breaking the vacuum in the main chamber.

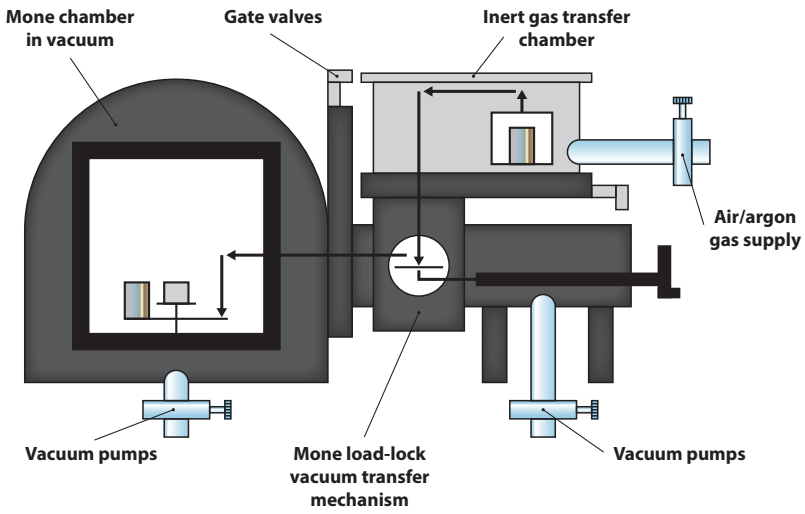

Figure 3: *NPL glove box and load-lock assembly attached to Mone comparator in vacuum chamber.*

Chapter IV

Vacuum equipment

- Vacuum chamber
- Pumps
- Gauges
- Materials
- Commercial weighing in vacuum apparatus

Vacuum chamber

The primary requirements for the vacuum chamber are for it to be leak-free whilst still allowing operation of the mass comparator, and not allowing contamination of the weights. The vacuum chamber should be constructed from a vacuum-compatible material, for example stainless steel. In addition, any seals should be made using either UHV seals, typically utilising copper gaskets, or of materials such as viton or nitrile. Whilst a UHV seal of copper should allow the generation of lower pressures within the vacuum chamber, the fact that they are single use means that for purposes where the seal is likely to be broken regularly, for example on an access door, the use of viton or nitrile seals is more practical. It is useful to incorporate several vacuum ports on to the chamber to allow the connection of equipment such as pressure gauges (both vacuum and atmospheric) and gas inlets. Electrical lead-throughs designed for use in vacuum are readily available. These will allow the comparator to be read as well as the connection of ancillary measurement equipment, such as platinum resistance thermometers.

The control motors for operating the comparator carousel should ideally be placed outside the vacuum chamber to avoid thermal effects. This will require the use of mechanical connections that are vacuum sealed. Again, these are commercially available.

Access to the weights should also be considered. If weights are to be loaded under vacuum, a load-lock assembly should be considered; if being loaded from storage in an inert gas, a glove box may be appropriate.

Pumps

In order to achieve the required pressures within the chamber a turbo-molecular pump with backing pump is required. These can be a major source of contamination, particularly if an oil-operated rotary pump is used as the backing pump. Schwartz [6] describes mass gains of 0.012 µg cm^{-2} day^{-1} which he attributes to sorption of oil particles from a turbo-molecular pump. Preferably oil-free pumps should be used, or if these are not available an oil mist trap should be incorporated into the backing line. The filter in this should be regularly cleaned or replaced. Additionally, an automatic isolation valve should be incorporated to prevent back streaming of oil vapour when the system is let up to atmosphere.

Gauges

Care should also be taken in selection of the vacuum gauges used for measuring the pressure within the chamber. Contamination of weights has been observed as a result of using inverted magnetron gauges [7] and it is therefore recommended that this type off gauge not be used. Preferably, a solid state Pirani gauge, or for higher accuracy a spinning-rotor gauge, should be used.

Materials

Any material used within the vacuum chamber should be vacuum compatible. Any lubricants used on the mass comparator should have a sufficiently low vapour pressure that no contamination is introduced, for example DC976 or Apiezon L. Additionally, any cables should have insulation that will not outgas significantly when exposed to vacuum.

Commercial weighing in vacuum apparatus

Comparators for weighing in vacuum are available commercially from Mettler Toledo and Sartorius. In addition, some laboratories have incorporated exisiting mass comparators into custom-built vacuum chambers. For example, NPL has for many years been using a Mettler Toledo HK1000 mass comparator inside a vacuum chamber.

A comparison of weighing on different vacuum operated mass comparators has been carried out as part of JRP WP2 [8]. This displayed little difference between the results of this comparison and the results of comparisons carried out in air.

Chapter V

Storage and transfer experiments

- Short-term storage and transfer
- Long-term storage
- Medium-term storage

Short-term storage and transfer

Berry and Davidson [9] have undertaken two experiments comparing the effect of transferring artefacts between air and vacuum and between an inert gas, argon, and vacuum.

In the first test, artefacts manufactured from Pt-Ir, SS and silicon were used to compare the effect of cycling them between vacuum and humidified laboratory air, as well as vacuum and humidified argon gas. The results of the humidified argon/vacuum tests showed excellent mass stability for all three materials, despite a shift in the SS data. This was attributed as being due to either adsorption/desorption of contamination during one of the vacuum measurements, or a shift in the centre of gravity of the SS stack. It was not believed to have been an effect due to the pressure cycling.

The results of the air/vacuum tests showed mass losses for the Pt-Ir and SS artefacts and a slight mass gain for the silicon artefacts. Overall the stability of artefacts cycled between humidified argon and vacuum was at least as good as, if not better than, that of the same artefacts cycled between humidified air and vacuum.

The second test involved repeatedly transferring SS and silicon artefacts between vacuum and either storage in argon or storage in laboratory air. The results for the air-stored SS and silicon artefacts showed a mass gain during the experiment that was not seen in the first static test. This was attributed to hydrocarbon contamination from the laboratory air forming on their surfaces during the period when they were outside the vacuum chamber either during transfer to the storage vessel or during storage. The argon-stored SS and silicon artefacts showed slight mass losses during the experiment that were again not seen in the first static test. This was assumed to be due to the removal of existing contaminative material from the surface of the artefacts as they were transferred between the vacuum chamber and the inert gas storage vessels. Overall, test two demonstrated that repeatedly transferring artefacts between storage in laboratory air and vacuum increased the mass gain of the artefacts, which was not seen in the artefacts that were stored and transferred in argon.

Long-term storage

The long-term stability of Pt-Ir and gold artefacts was measured by Fuchs et al [10] 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.

Medium-term storage

As part of EMRP SIB-05 Work Package 4 [11], a study evaluating the benefits of medium-term storage (six-months) of SS artefacts in nitrogen compared with storage in air was undertaken at three National Measurement Institutes (NMIs): NPL, Conservatoire National des Arts et Métiers (CNAM - France) and Physikalisch-Technische Bundesanstalt (PTB - Germany). PTB also tested the storage of artefacts in vacuum.

Prior to measurements being undertaken, the artefacts were cleaned and their masses determined gravimetrically before being placed into storage. No further cleaning was carried out during the experiment. After storage, the mass of the artefacts was re-determined. In addition, NPL carried out XPS measurements on circular SS surface samples which were subjected to the same conditions as the artefacts.

XPS results showed no significant difference in carbonaceous growth on the surface of SS samples stored in nitrogen for six-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 shorter than six-months.

The CNAM gravimetric results on SS artefacts did not show any significant difference between storing the artefacts in dry air and storing them in nitrogen gas.

Both the NPL and PTB gravimetric results on SS artefacts stored in air for six-months showed no significant gain in mass. This contrasted with the nitrogen stored artefact at NPL and the nitrogen and vacuum stored artefacts at PTB 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 SS samples used in the NPL XPS measurements did not show any evidence of surface contamination. 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. Therefore further investigations are required in order to reduce 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.

Further information

- References
- Contact NPL

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