NPL

National Physical Laboratory

Good Practice Guide No. 140

Guide to Weighing in Vacuum

EXPERT

TECHNICAL LEVEL

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NPL Authors

Michael Perkin
Stuart Davidson

National Physical Laboratory
Hampton Road
Teddington
Middlesex
TW11 0LW

Good Practice Guide enquiries: **020 8943 6356**
www.npl.co.uk/contact

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Front cover image: *A silicon 'Avogadro' sphere being weighed on a vacuum balance. (Source: NPL).*

Guide Information

What is it about?

This guide presents information that will assist with establishing traceability to the primary relations of the redefined kilogram, i.e. watt balance and X-ray crystal density (Avogadro) experiments. The guide gives information on the establishment of weighing-in-vacuum facilities and on methodologies to achieve traceability from the realisation of the kilogram in vacuum to weights stored and used in air.

Who is it for / what is its purpose?

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

What is the prerequisite knowledge?

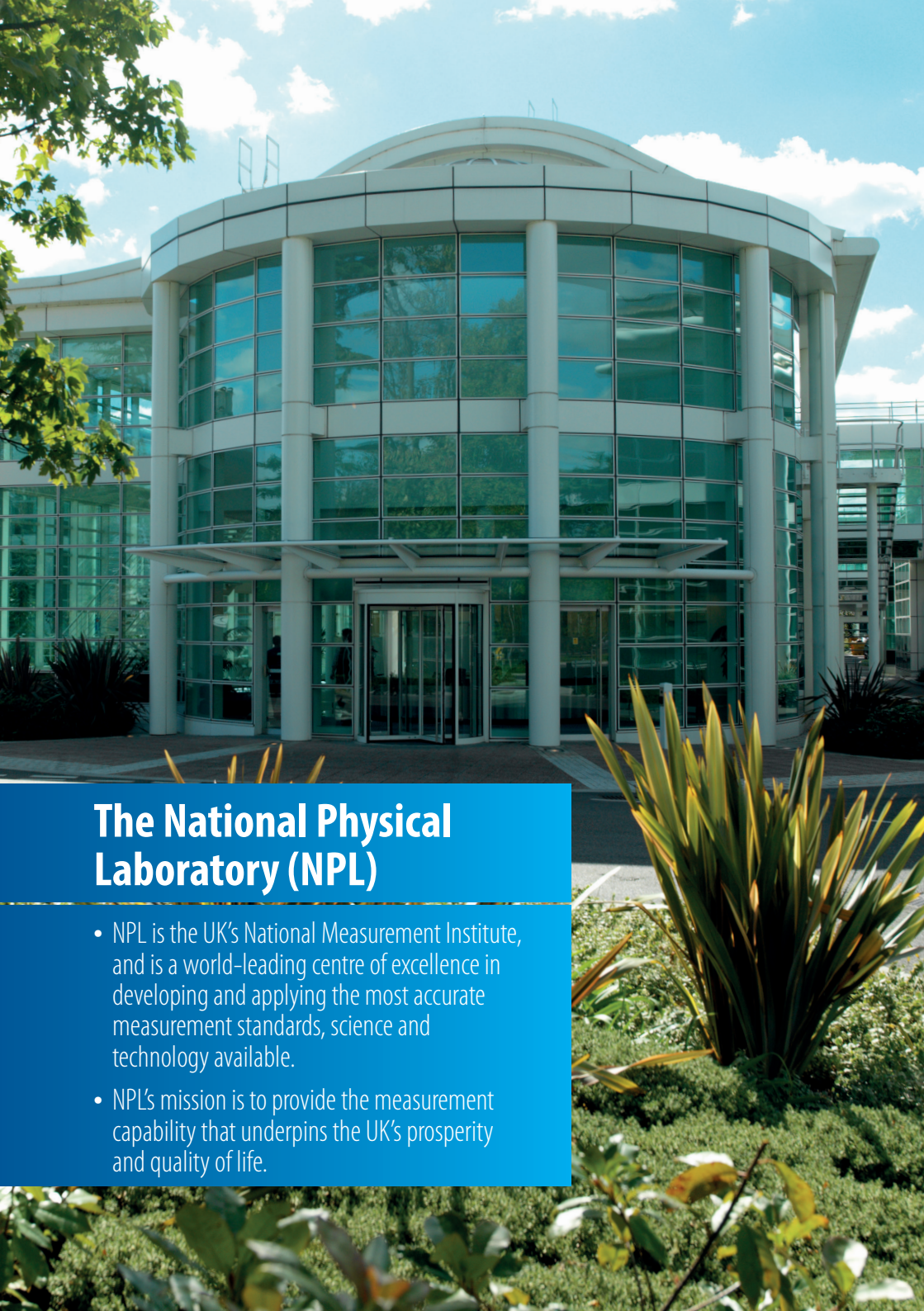
This guide is intended for users with a knowledge of the maintenance and dissemination of the SI unit of mass.

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
- The need for a new standard
- New approaches
- Redefinition
- In-vacuum weighing

Current definition

Since 1889 the SI unit of mass, the kilogram, has been defined as being the mass of a unique artefact named the International Prototype of the Kilogram (IPK), made from platinum-iridium, and stored at the BIPM (International Bureau of Weights and Measures) in France. It is the last of the seven SI base units to be defined in terms of an artefact rather than by relation to a fundamental physical constant. This has provided significant challenges, both in terms of the maintenance of the IPK and its official copies, and the traceable dissemination of the mass scale worldwide.

The need for a new standard

By definition the value of the mass of the IPK is exactly one kilogram and the uncertainty of this value is zero. As an artefact, however, the IPK cannot be absolutely stable although its stability is impossible to measure due to there being no higher reference value against which to check it. On four occasions between 1889 and 2014 the mass of the official copies and the working standards of the BIPM have been compared with the mass of the IPK [1]. The working standards are then used to assign values to the national prototypes. Before each periodic verification both the IPK and the copies were cleaned, latterly by a process known as nettoyage-lavage [2]. The results of the comparisons show some divergence with time as shown in Figure 1.

From this data, it can be assumed that in practice the mass of the IPK has also been changing over time. This cannot be proven since there is no other standard with which to compare it.

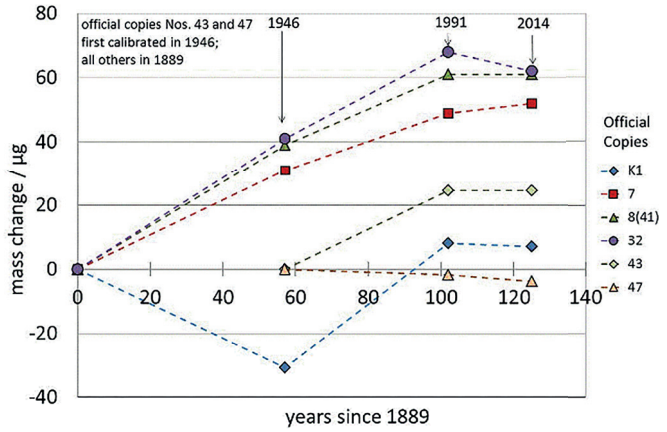

Figure 1: *Evolution in mass since the first calibration of the official copies of the IPK (Image courstesy of BIPM).*

New approaches

The believed instability of this artefact based definition has led to significant research throughout the world to redefine the SI unit of mass in terms of a fundamental constant. Research has concentrated upon two methods, the International Avogadro Coordination (IAC) and the watt balance. The Avogadro approach will relate the unit of mass to the Avogadro constant (N_A) and will realise the kilogram via a sphere of very pure single-crystal silicon 28. The watt balance projects will define the unit of mass in terms of the Planck constant (h) and realise the kilogram by relation to the electrical units of the volt and the ampere.

Redefinition

At the 24th meeting of the General Conference on Weights and Measures (CGPM) in 2011, it was agreed that the SI unit of mass should be redefined based upon fundamental constants, and the final steps required before the redefinition can occur in 2018 were set out [3].

In both the Avogadro and watt balance approaches, the realisation of the kilogram will need to be carried out in vacuum. This is in contrast to the existing kilogram artefact which is stored and used in air. The establishment of a link between mass in air and mass in vacuum is therefore crucial for both the maintenance and dissemination of a kilogram defined in terms of fundamental constants, and the maintenance of the mass scale between realisations.

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.

In-vacuum weighing

To provide traceability to working standards of mass there will therefore be an increasing demand for mass comparisons to be carried out in vacuum. This publication aims to provide guidance in the practical methods of weighing in vacuum, primarily related to the comparison of mass standards. It also summarises what needs to be considered when using mass standards in air that were calibrated in vacuum.

High accuracy mass comparators in use today in national standards laboratories are normally fully automated, with multi-position carousels allowing the comparison of four or six mass standards. Commercial mass comparators are available from several different manufacturers, with a number of them also available in chambers allowing vacuum operation.

Historically, the development of mass comparators that operate in vacuum was undertaken to remove the effect of air buoyancy. When determining the mass of a stainless steel kilogram by comparison with a platinum-iridium kilogram in air, the uncertainty in the buoyancy correction is typically the largest component in the uncertainty budget. This is because the volume of the stainless steel kilogram is approximately 79 cm^3 larger than the platinum-iridium kilogram. In air of density 1.2 kg m^{-3} the magnitude of the buoyancy correction is approximately 94 ppm, and a significant uncertainty that is introduced into the mass of the stainless steel artefact.

The buoyancy correction can be removed entirely by comparing the two weights in a vacuum, although this introduces additional effects, such as the desorption of water from the surface of the weight, that must be considered.

With the redefinition of the kilogram, weighing in vacuum has become necessary to provide traceability and to disseminate the standard of mass.

Chapter II

The new standard experiments

- Watt balance
- Avogadro experiment

Watt balance

The watt balance [3,4] was originally conceived by Kibble at the National Physical Laboratory (NPL). Its operating principal is shown diagrammatically in Figure 2. Mass is measured on the watt balance by comparing virtual electrical and mechanical power, VI=Mgu. During the first stage of the experiment, the voltage, V, and velocity, u, are measured, Figure 2(a). The current, I, and the weight, Mg, of the mass, M, are then measured in a separate stage, Figure 2(b). The two stages are linked using a coil of wire of length l placed perpendicular to the field B of a strong magnet. Using a two stage process removes the need to determine the values of B and l, provided they are stable between the two stages of the measurement. Both of the electrical quantities, voltage and resistance, can be measured in terms of the Planck constant via quantum mechanical effects: the Josephson effect and the Quantum Hall Effect. The Josephson effect occurs in superconducting systems at low temperatures (usually about 4 K) and can be used to produce a standard voltage. The QHE occurs in particular semiconductor structures at low temperatures and provides a standard resistance.

Avogadro experiment

This experiment relies upon the measurement of the mass of a single silicon atom. This is carried out by counting the number of atoms of silicon in a highly polished sphere of silicon. Direct counting of sufficient atoms is not possible; an indirect measurement can be carried out by measuring the volume occupied by an atom in a perfect crystal, measuring the volume of the crystal and dividing one by the other. From a measurement of the mass of the crystal, the mass of a silicon atom can be calculated. From this determination the Avogadro constant can be derived.

Polishing the crystal into a near perfect sphere simplifies the crystal volume measurement. The volume is determined from the average of a large number of diameter measurements of the sphere. Using a polished sphere eradicates any problems associated with having any edges or corners in other shapes. Any corrections associated with departures from a perfect sphere will require only small corrections to the volume measurement.

The volume occupied by an atom can be related to the SI by using X-rays to reveal the spacing of planes in the crystal lattice and then using an optical interferometer to measure the spacing in terms of a metre. Recent measurements have been carried out using a sphere of almost pure silicon 28, thus limiting the difficulty of making accurate measurements of the amounts of the different isotopes present in natural silicon [7].

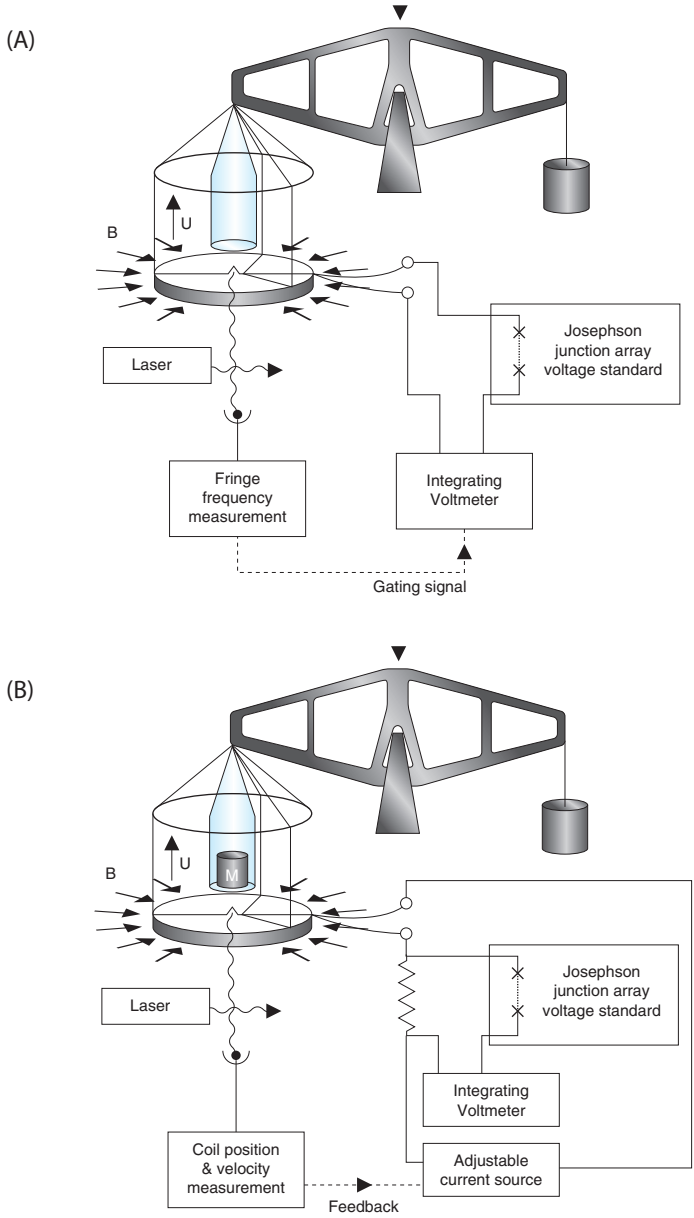

Figure 2: *Diagrams of the operation of a watt balance.*

The major contributors to the uncertainty of this technique are the measurement of the volume of the sphere, measurement of the surface layers and the spacing between atoms, and determination of the amounts of other silicon isotopes in the sphere.

Figure 3: *A sphere of natural silicon.*

Chapter III

Equipment for weighing in vacuum

- 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 to not introduce contamination to the weights. The vacuum chamber should be constructed from a UHV compatible material, for example stainless steel. In addition, any seals should be made using either UHV seals, typically utilising copper gaskets, or seals using 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 will be necessary, or 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 a 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 [7] 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 [8] when significant levels of contamination are present (i.e. at pressures above about 0.1 Pa), 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 materials used within the vacuum chamber should be vacuum compatible. Any lubricants used on the mass comparator should have a sufficiently low vapour pressure such 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 existing 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 [9]. This displayed little difference between the results of this comparison and the results of comparisons carried out in air.

Chapter IV

Weighing in vacuum

- Recommended pressure of operation
- Acclimatisation times
- Air–vacuum transfer
- Transfer apparatus and procedure

Recommended pressure of operation

An understanding of the sorption characteristics of the mass standards used is needed in order to achieve the link between mass in vacuum and mass in air, and is key to providing traceability to a primary realisation of the kilogram in vacuum. In order to do this, sets of sorption artefacts comprising mass standards with closely matched weights and volumes but with different surface areas can be used. A typical set of artefacts is shown in Figure 4.

Figure 4: *A typical set of kilogram artefacts..*

By transferring the set between air and vacuum the differential sorption of the individual artefacts can be used to calculate the adsorption characteristics of the material in terms of mass change per unit surface area. Figure 5 shows a typical sorption curve for a stainless steel mass standard transferred between air and vacuum. The mass standard loses a total of about 35 µg when transferred between air and vacuum. The mass loss is approximately the same for pressures between 0.1 Pa and 0.001 Pa, showing that this is the ideal pressure range for weighing in vacuum. The curve shows significant hysteresis, the mass standard only fully reabsorbs surface water when it is returned to ambient conditions (50 % relative humidity was used for the tests shown).

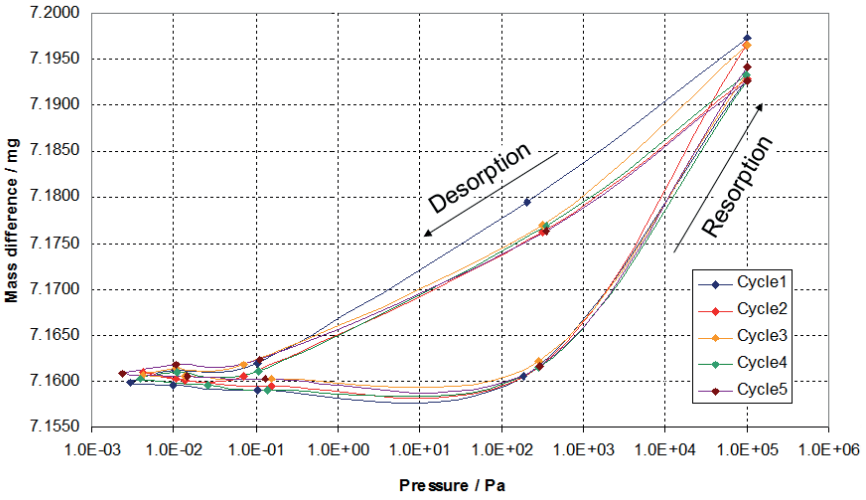

Figure 5: *A typical sorption curve for a stainless steel mass standard transferred between air and vacuum.*

Tests have been performed on the two materials typically used for primary mass standards (platinum-iridium and stainless steel) and sorption characteristics are broadly similar for weights with a good surface finish (R_a<100 nm) with the surface sorption being about 0.1 µg/cm^2 [10]. A comparison between national measurement institutes [11] has shown good agreement between sorption characteristics using the same set of artefacts but other research has shown significant variation between the sorption characteristics of different artefacts of the same material (platinum-iridium [12]. This suggest that in order to achieve the best uncertainty on air-vacuum transfer of a mass standard real time sorption measurements, using a set of artefacts of the same material and surface finish, should be used.

Acclimatisation times

The loss/gain in mass shown in Figure 5 does not occur instantaneously. Figure 6 shows the mass difference between a stainless steel weight after being transferred to vacuum and a stainless steel weight that had been in vacuum for several weeks. It can be seen from the graph that it took between 10 – 20 hours to become stable in a vacuum. This is consistent with the behaviour of platinum-iridium weights [10].

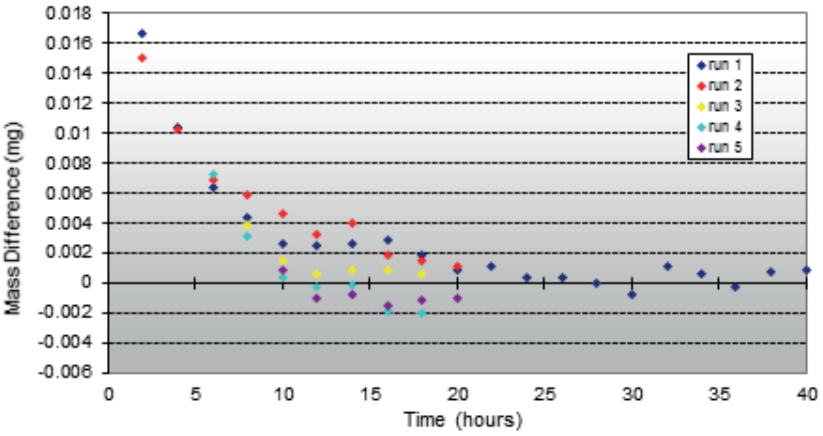

Figure 6: *The mass difference between a stainless steel weight after being transferred to vacuum and a stainless steel weight that had been in vacuum for several weeks.*

Therefore, the recommendation is that weights be left in vacuum for at least twenty-four hours prior to measurements being made.

Similarly it is recommended that weights returned to air after use in vacuum are left for a minimum of 12 hours to stabilise. After vacuum exposure the surfaces of the weights (and the vacuum chamber) will be dry and the use of a flow of humidified air is recommended to promote the re-adsorption of water onto the surfaces of the weights.

Air–vacuum transfer

At some point it will be necessary to transfer weights from air to vacuum and vice versa. Davidson [13] has reported that the repeated transfer of platinum-iridium and stainless steel artefacts from vacuum to ambient air introduced an exponential mass gain with time, compared with artefacts that had been stored solely in vacuum or in ambient air. However, Picard and Fang [12] performed similar measurements but did

not see evidence of comparative mass gain, which they attribute to the short period of time the weights were kept in air between measurements and that the artefacts stayed inside the clean balance case even when exposed to air.

Thus, the method of carrying out air-vacuum transfer can have a significant effect upon the stability of the mass standard.

Transfer apparatus and procedure

The simplest method of transfer is direct transfer to the comparator. Transfer is performed whilst the balance is at ambient pressure and the weight is loaded/unloaded through the normal access point of the comparator.

With the use of a load-lock assembly the comparator can remain under vacuum. 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.

In addition to the load lock, it is possible to incorporate a glove box assembly which will allow the transfer of artefacts that have been stored in an inert gas. Figure 7 shows the equipment used at NPL as described by Berry and Davidson [14].

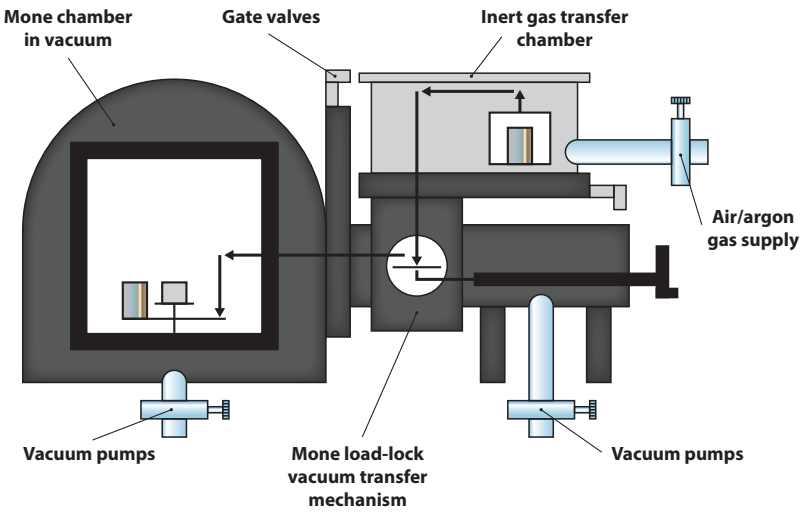

Figure 7: *The equipment used at NPL to test the effect on the mass stability of artefacts transferred and stored in air compared with artefacts that are transferred and stored in an inert gas.*

The procedure for loading a weight into the vacuum chamber is as follows:

- Place the storage vessel containing the artefact inside the glove-box and replace the lid.
- With the main chamber evacuated and the gate valve shut, fill the load-lock chamber and glove-box with the inert gas. This can be done either by evacuating the chamber and filling with the gas or by flowing the gas through the chamber to replace the air.
- Open the valve between the glove-box and the load-lock.
- Open the storage chamber and with the use of an appropriate tool, transfer the weight from the storage chamber to the load-lock transfer mechanism.
- Close the valve between the glove-box and the load-lock chamber and evacuate the load-lock chamber.
- When the load-lock chamber is evacuated, open the gate valve between the load-lock chamber and the main chamber.
- Use the load-lock mechanism to transfer the weight on to the mass comparator.
- Retract the load-lock mechanism and close the gate valve.

To unload a weight from the mass comparator the reverse process is employed.

Further information

- References
- Contact NPL

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National Physical Laboratory
Hampton Road
Teddington
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