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Recent Developments in the Study of the Surface-Stability of Platinum and Platinum-Iridium Mass Standards

Quantifying mercury and carbon contamination on platinum-iridium alloy surfaces using XPS

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We review developments in the study of the stability of platinum-iridium standard weights, in particular the kilogram prototypes manufactured from alloy supplied by Johnson Matthey in the 1880s that still stand at the heart of the International System of Units (abbreviated SI from the French: *Système international d'unités*). The SI has long since moved on from length standards based on physical artefacts fabricated from this alloy, but the SI unit of mass is still defined in this way, as the mass of a real physical object. The stability of these reference masses has been a concern since the 1930s, with mass loss or gain at the surface being the principal concern. In recent years X-ray photoelectron spectroscopy (XPS) has been particularly valuable in elucidating the types of contamination present and the mechanism by which contamination takes place. While direct studies on the International Prototype Kilogram are understandably difficult, at Newcastle University we have examined the surfaces of six Pt mass standards also manufactured in the mid-19th century, using XPS to identify contamination chemically. XPS shows a significant quantity of mercury on the surfaces of all six. The most likely source of Hg vapour is the accidental breakage of thermometers and barometers, and the mechanism of contamination may be similar to the poisoning of platinum group metal (pgm) catalysts by Hg, an effect known for almost a century.

Introduction and History

The history and technology of mass and length standards fabricated from pgms have been described previously in *Platinum Metals Review* (1, 2). One of the earliest contracts in the history of Johnson Matthey Plc was the supply of high-purity Pt-Ir alloy that formed the basis of artefact standards for the metre and kilogram. Johnson Matthey supplied material (3) for the fabrication of 30 standard metres and 40 standard kilograms following an agreement with the French government in 1882 and it is these artefacts that defined the SI units of length and mass through most of the 20th century. Many more have been supplied since (see for example the prototype kilograms illustrated in **Figure 1**).

In the second half of the 20th century there was a concerted move to replace artefact standards with definitions in terms of fundamental quantities, so that artefact length standards are now of purely historical interest. Of all the SI units only the kilogram remains as an artefact standard (4) and even though this may not be the case for much longer, the stability of transfer standards will be crucial for many years to come. The unit of length, the metre, is relatively inexpensive to realise by interferometry, whereas alternative methods (5–7) for defining a unit of mass are extremely expensive to realise and will likely only ever occur at one site (or a very small number of sites) internationally. It therefore seems certain that even when the kilogram is redefined in terms of fundamental constants by these
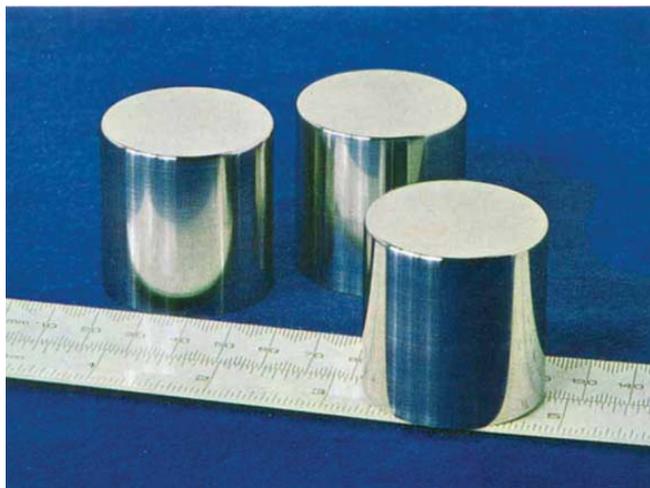
Fig. 1. A group of three platinum-iridium kilogram weights fabricated by Johnson Matthey Noble Metals for the Bureau International des Poids et Mesures. Each is 39.2 mm in diameter and 39.5 mm high

means there will be a continuing need to disseminate the standard of mass *via* physical standard weights and even the existing Pt-Ir kilogram standards may have a key role as secondary or transfer standards for many years to come.

Research on Surface Stability

It had been suspected since around 1939 and known since 1992 that the mass of prototype kilogram standards increases slightly over time (8–11) and surface effects have long been suspected as being responsible (12). The most obvious source of potential mass variation at the surface is water adsorption and this has been the focus of a number of studies (13). Kochsiek (14) measured water adsorption onto thin Pt-Ir foils having higher surface area per unit mass than kilogram prototypes. For the relative humidity range 30% to 70% this resulted in only the equivalent of a 10 μg mass change of a Pt-Ir kilogram with respect to a stainless-steel reference weight. Adsorption was also reversible, indicating that the water is physisorbed and therefore eliminating water as a possible cause of any long-term mass gain.

It is known that solvent cleaning can remove some or all of the added mass. Bigg (15) examined weighing data on the UK national prototype, number 18 and concluded that within a year of an ethanol/ether/ammonia wash it regained 'significant' mass. Bigg concluded that cleaning should be repeated prior to the most

accurate comparisons. Calcatelli *et al.* (16) studied the adsorption of contaminants gravimetrically and reported a gain in mass over the first twelve months after cleaning of 9.8 μg for a Pt-Ir kilogram prototype. On the basis of published measurements by the International Bureau of Weights and Measures (BIPM) and National Physical Laboratory (NPL) for the UK prototype kilogram 18, Martin Seah and myself at NPL developed a 'diffusion limited' model of carbonaceous growth (17). This gave a quantitative prediction of mass increase of carbonaceous contamination, Δm_C, of the form (Equation (i)):

$$\Delta m_C(T) = \rho\sigma \sqrt{2D} \, (T + c)^{1/2} \qquad (i)$$

where D is an effective diffusion constant for small molecules through the existing contamination of density ρ on a prototype of surface area σ, T is the time elapsed since the last cleaning procedure and c is a constant describing quantity of contamination that builds up in the first minutes or hours after cleaning, before the process becomes diffusion-limited. This is consistent with historical weighing data over more than 60 years. Some of the measurements giving rise to Equation (i) were for material removed during cleaning, so Equation (i) models only the reversible part of the contamination of the surface (i.e. which can be cleaned using solvent washing or other cleaning). There remained the possibility of an additional irreversible contribution from other species (which, as we shall see, can come from Hg vapour).

Meanwhile, in the period 1960–1990 a suite of surface characterisation methods and instruments had been developed by the surface science community and progressively applied to practical problems such as the control of contamination in semiconductor manufacture, for example. Notable amongst these techniques are X-ray photoelectron spectroscopy (XPS), secondary ion mass spectrometry (SIMS) and atomic force microscopy (AFM). The XPS technique is an excellent tool to address many types of contamination problem and is a key tool in the characterisation of practical heterogeneous catalysts (18). Briefly, monochromatic X-rays are directed on a specimen surface and the energies of emitted photoelectrons are analysed. The spectrum of emitted photoelectron intensity as a function of kinetic energy gives information on the composition of the top few nanometres of the surface, a depth limit set by the short distance through which electrons at those energies can pass through matter without energy loss. XPS must be performed

in ultra-high vacuum to prevent contamination of the surfaces under analysis and this makes XPS a relatively expensive analytical technique, but it can be extremely powerful in unambiguous identification of all chemical elements except hydrogen and helium, together with some information about their oxidation state. The key benefit of XPS is that when applied to the mass stability problem it gives an unambiguous identification of the chemical nature of the contamination, which weighing alone cannot do.

Surface analytical techniques such as XPS were first applied to the analysis of mass standard surfaces by Ikeda *et al.* (19). Later Martin Seah and myself at NPL, UK, found XPS particularly valuable in showing for the first time the unexpected presence of Hg on samples of Pt-Ir alloy foil stored in the same laboratory environments as kilogram weights. Using XPS we were able to demonstrate that: (a) atmospheric Hg contamination (20) is a problem in causing part of the monotonic increase (21) in Pt-Ir prototype masses; and (b) the remaining mass increase is largely due to the growth of a carbonaceous layer, consistent with Equation (i). Adsorption of Hg onto the surfaces of pgms, with consequent inhibition of their catalytic activity, has been known for almost a century (22, 23) but was a surprise to those working in the field of mass metrology.

Recent Work at Newcastle University

It is twenty years since Hg was first identified on samples of Pr-Ir alloy exposed to laboratory air in a weighing laboratory by XPS analysis of small pieces of metal foil (20). More recently XPS instrumentation has increased in capability in several respects. In particular the sample size that can be accepted into the analytical chambers of modern instruments is much larger than previously possible. This has been largely driven by the needs of the semiconductor community to analyse whole silicon wafers of four inches or more in diameter. Therefore when we came to commission a new state-of-the-art XPS instrument at Newcastle University in 2012 it became clear that we could revisit the problem of Hg contamination on these standard weights directly. We could now analyse the surface of an intact standard weight inside our new XPS instruments.

Of course the Pt-Ir prototype kilograms that form part of the SI are far too valuable to study by these methods. Neither can new ones be fabricated, since it would be difficult to replicate with confidence the polishing

techniques or the exposure of these surfaces over the 130 years since manufacture. Instead, we have analysed imperial weights made of Pt in the Victorian era, but which are now museum pieces:

• Reference weight RS1: a Pt troy pound held by the Royal Society in London since the 1840s

• Reference weight RS2: a cylindrical Pt avoirdupois pound held by the Royal Society since at least 1853 (the container made for it bears this date, though the weight itself has 1844 stamped on it; there is no written record that the Royal Society owned it earlier than 1853, although an earlier date cannot be ruled out)

• Reference weights SM1, SM2, SM3 and SM4: small Pt weights of 1, 2, 3 and 6 grains which have been kept in a circular threaded ivory box, probably since 1830, now held by the Science Museum in London.

No records appear to exist on the origin of the metal for these weights. Consideration of the fabrication dates alone mean that it is possible that the metal for SM1 to SM4 was refined using Thomas Cock's powder metallurgical route, possibly at the then Johnson company that later became Johnson Matthey (24).

XPS spectra were acquired using a Thermo Theta Probe spectrometer (Thermo Scientific, East Grinstead, UK). Survey spectra were at a pass energy of 200 eV and narrow scans at 80 eV. Argon cluster ion sputtering was performed using the Thermo Scientific™ MAGCIS™ gun at 6000 eV cluster ion energy and using settings that the manufacturer has observed to lead to clusters of approximately 1000 atoms with a wide size distribution (25).

XPS spectra from both RS1 and RS2 show large C 1*s* peak intensities originating in a thick layer of carbonaceous contamination. RS1 and RS2 show a number of surface species such as zinc, sodium, tin and others and approximately one atomic layer of Hg. A survey spectrum from RS1 is shown in **Figure 2**.

The steeply rising inelastic background (26, 27) under the Hg and Pt peaks indicates that they are below a thick carbon contamination layer. The spectrum in **Figure 3** is from SM4, which shows by far the greatest level of Hg contamination in our study. Remarkably, the Hg 4*f* peaks in **Figure 3** are much stronger than the Pt 4*f* peaks, showing (given that the intrinsic XPS sensitivity to both elements is similar) that within the XPS accessible depth there is more Hg than Pt. Indeed, if the same Hg accumulates on a Pt-Ir prototype kilogram per unit surface area then that prototype would increase in mass by just
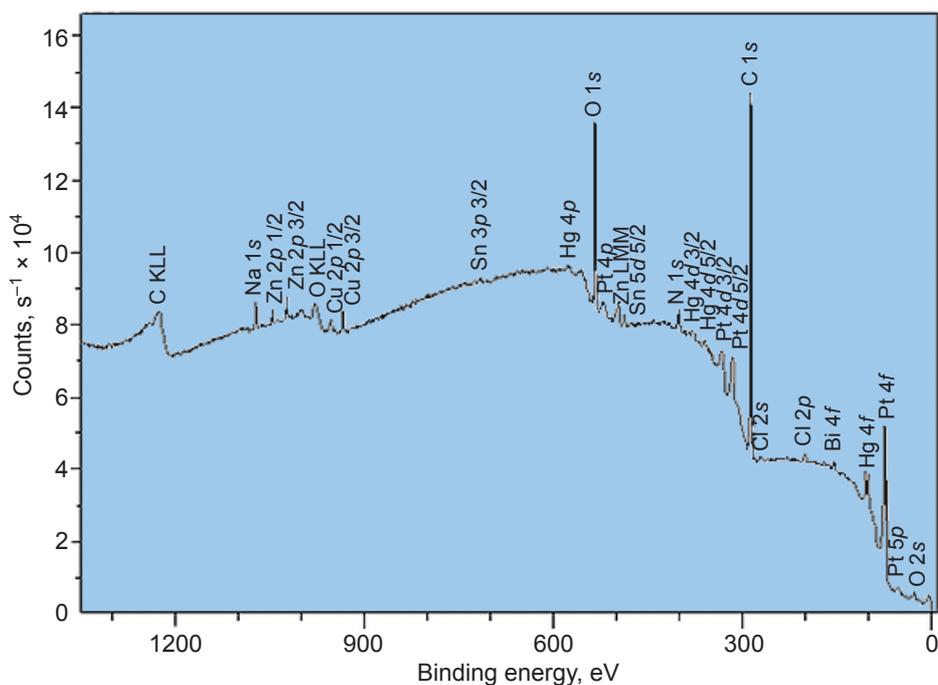
Fig. 2. XPS survey spectrum from reference weight RS1. Mercury and carbonaceous contamination are clearly present. Auger and photoemission features are both present (though the binding energy axis properly applies only to the photoemission peaks)
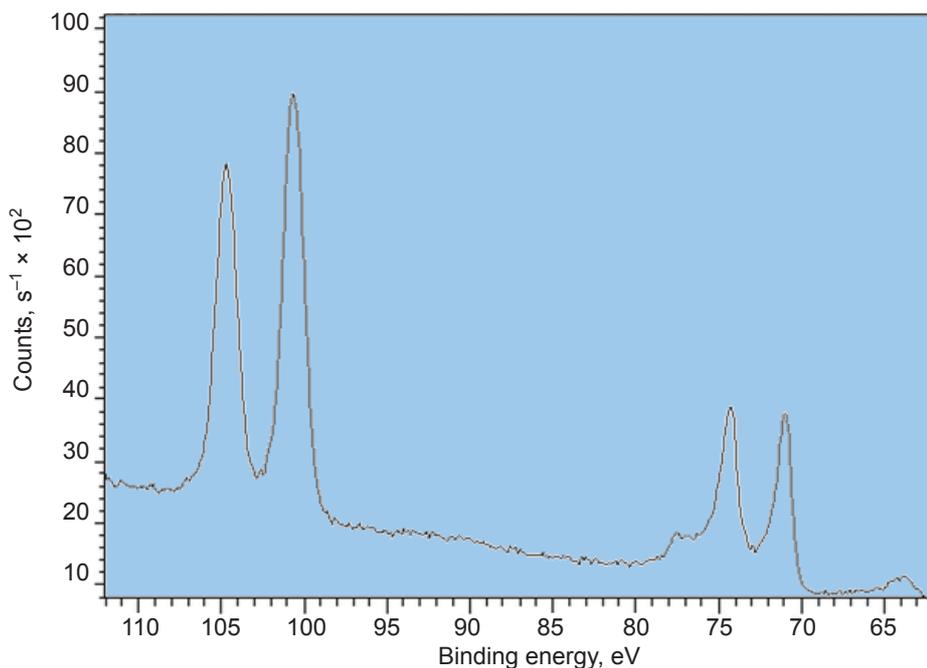

Fig. 3. Narrow scan spectrum from the surface of SM4. Hg dominates the XPS spectrum here, with the Pt 4*f* peaks between 70 eV and 76 eV in binding energy having a much lower intensity. Therefore, remarkably, most of the metal within the XPS sampling depth is Hg contamination, not Pt. The region of the Pt peaks between 70 eV and 78 eV is somewhat complex, and probably comprises Pt metal, Pt surface oxide(s) and some intensity from an Al 2p peak originating in alumina inclusions, possibly from polishing during manufacture

under one quarter of a milligram or 0.25 parts per million (ppm).

Argon cluster sputtering of RS1 was continued and after a total of 1320 seconds the Hg and carbonaceous contamination had been very effectively removed, leaving a clean Pt surface and a small amount of inorganic tin contamination. Argon clusters are a relatively new tool in XPS, with the ability to remove carbonaceous material while having a low sputter rate in any underlying inorganic material. Compared to Hg, Pt has some unusual potential electron scattering properties at kinetic energies of around 200 eV (28), an effect which is not relevant however at the specific kinetic energies used in this work.

Recently the presence of Hg at the surface of a 19th century Pt kilogram prototype in Switzerland has been confirmed (29), so that all seven 19th century Pt weights so far directly analysed by XPS show significant Hg contamination.

Carbonaceous Contamination

Figure 4 shows measurements from the present work added to the diffusion-limited growth model of Equation (i). The thickness of carbonaceous contamination, d_C, can be calculated from Equation (ii):

$$d_C = \lambda \ln\left(\frac{I_{Pt,\,GCIB\,cleaned}}{I_{Pt,\,as\,rec'd}}\right)\cos\theta \qquad (ii)$$

We used an attenuation length $\lambda = 3.0$ nm in this equation for consistency with earlier published work on this problem, though more accurate estimates of the attenuation length are possible in principle (30). The intensity ratio of Pt 4f peaks before ($I_{Pt,\,as\,rec'd}$) and after ($I_{Pt,\,GCIB\,cleaned}$) argon gas cluster ion beam (GCIB) removal (31) of the carbonaceous contamination therefore gives the thickness of carbonaceous contamination. The emission angle for photoelectrons analysed is θ. The mass of carbonaceous contamination is then obtained by multiplying the thickness by the surface area of a prototype and the estimated density of the contamination (Equation (iii)).

$$m_C = \rho A d_C \qquad (iii)$$

The estimate of density is the main contribution to the uncertainties in this entire calculation, which leads to relatively large error bar in **Figure 3**. We estimate a density of 1300 kg m^{-3}. This still seems a reasonable value for a dense cross-linked layer,
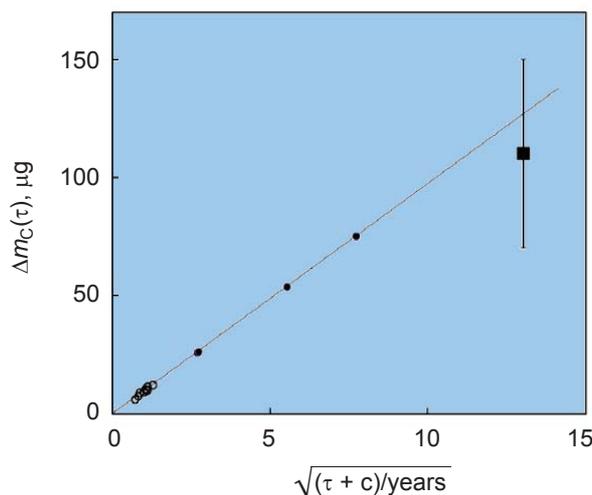

Fig. 4. Growth in carbonaceous contamination, Δm_C, on a Pt-Ir prototype kilogram as a function of the time since last cleaning, τ. Here c = 0.387 years, representing a small initial contamination layer acquired rapidly before this diffusion-limited model becomes valid. The filled square is calculated from XPS measurements on standard weight RS1, scaled to the larger surface area of a kilogram prototype. The straight line is a fit to out diffusion-limited accretion model (though the fitted parameters are determined only using the weighing data, not the XPS measurement)

but in this work we place a large uncertainty on this value, of around 40% following recent work by Davidson where a much lower density was found (32).

Ultraviolet (UV)/ozone treatment (often used in cleaning carbonaceous contamination from silicon wafers in the electronics industry) is also effective in removing organic contamination from Pt and Pt-Ir surfaces (33), at the expense of a monolayer of oxidation of platinum. **Figure 5** shows weighing results for RS2 as a methanol, water and UV/ozone (UVOPS) treatment (33) is applied five times in succession. The weight lost at the nth cleaning step approaches zero and XPS confirms the surface to be as clean as air-exposed sputtered samples after five cleaning steps.

The surface analytical community will welcome the widespread availability of GCIB sources because they are expected to have two key properties:
(a) Removing organic material with the minimum of damage to underlying organic material (34) and
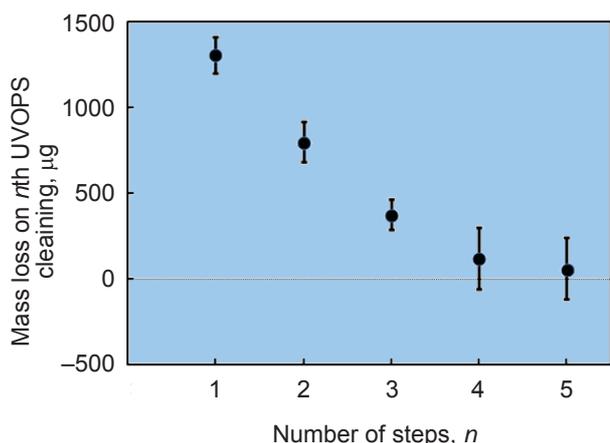(b) Leaving inorganic surfaces (such as metals) undamaged.

Fig. 5. Weighing results for five successive cleanings of reference weight RS2 by the UV/ozone method. Points represent the mass lost during cleaning

The second property is particularly interesting from the point of view of cleaning mass standards in vacuum, where UV/ozone treatment cannot be applied. However our measurements using GCIB and XPS suggest that argon clusters are not sufficiently selective in this application. Exposure sufficient to remove the carbonaceous contamination also removes an unacceptable mass of substrate metal (35). GCIB does not therefore offer a viable cleaning method for Pt-Ir prototype kilograms, at least under presently available source conditions, though it has been very useful to us in analysis, helping to quantify the carbonaceous contamination on RS2.

Quantifying Mercury Contamination

Hg is present in significant quantities on all six of the 19th century standard weights we examined (36). The most intense peaks in the XPS spectra of Pt and Hg lie very close to each other in kinetic energy. Therefore the photoelectrons from these peaks have almost the same kinetic energy for Hg and Pt (differing by only around 2%). This means that the inelastic mean free path (IMFP) of photoelectrons from Hg and Pt will be almost the same when passing through any particular material. This allows us to apply a relatively simple but precise equation for estimating the thickness of the Hg contamination (Equation (iv)), a method originally proposed by Hill *et al.* (37) and later applied by Iwai *et al.* (38) and Cumpson (39):

$$d_{Hg} = \lambda \ln\left[1 + \frac{(I_{Hg}/s_{Hg})}{(I_{Pt}/s_{Pt})}\right] \cos\theta \qquad (iv)$$

where d_{Hg} is the effective thickness of the Hg layer, I_{Hg} is the intensity of the Hg photoelectron peak, I_{Pt} the intensity of the Pt photoelectron peak and s_{Hg} and s_{Pt} are sensitivity factors due to Schofield (40). **Table I** shows that all six standard weights contained significant Hg at their surfaces.

However most of the Hg contamination may already be 'buried' in defects and grain boundaries and be invisible to XPS. Normally one would perform a sputter depth-profile, either by XPS or SIMS, gradually removing layers of metal using an ion beam, to measure

Table I XPS-Based Measurements of Mercury at the Surface of Six Platinum Weights Made in the 19th Century (34)[a]			
	Mercury determined by XPS		
Platinum reference weight	**Peak intensity ratio** I_{Hg} / I_{Pt}	**Equivalent mercury thickness** d_{Hg}, nm	**Equivalent mass of mercury if on a Pt-Ir kilogram prototype** Δm_{Hg}, μg
RS1	0.17	0.23	23
RS2	0.15	0.21	20
SM1	0.77	0.90	87
SM2	0.41	0.53	52
SM3	0.93	1.04	100
SM4	3.75	2.57	249

[a]We estimate uncertainty in the equivalent mass on a kilogram prototype to be around ±20% at one standard deviation

the concentration of Hg as a function of depth into the surface. This, however, would be destructive. It is very difficult to envisage any possible experiments that are both non-destructive and sufficiently sensitive to detect all the Hg present. Neutron activation analysis (NAA) would have the sensitivity and selectivity to identify Hg within the top few micrometres of the surface, but due to the long-lived activated Ir species created (41) this may be straightforward only for Hg within pure Pt weights, not Pt-Ir alloys. NAA facilities are also quite rare. The best approach may be a skilled application of transmission electron microscopy (TEM), wavelength-dispersive X-ray analysis in a scanning electron microscope (SEM) or electron-probe microanalysis (EPMA) system and dynamic SIMS depth profiling applied to sample Pt-Ir surfaces.

Hg vapour from accidental spillages from barometers and thermometers is sufficient to explain the Hg accumulation. Hg has been used in a wide range of scientific instruments over the last 200 years. Davis (42) describes the current storage location of the international prototype kilogram. Figure 3 of that paper is a photograph of the safe where the international prototype and its six official copies have been stored since the late 1980s. Beside the bell jars enclosing the international prototype is a small white thermometer containing alcohol and Hg (43). The Hg is fully enclosed and therefore no immediate danger to the prototypes around it. Nevertheless the presence, today, of a glass tube containing liquid Hg even within the storage location of all the prototypes at the centre of the SI shows how pervasive the use of Hg within scientific instruments has been. Other sources of Hg exist. It has been suggested that compact fluorescent light fittings may release between 1 mg and 4 mg of Hg when broken (44). Hg vapour has been measured in the breath of some people who have Hg amalgam dental fillings (45). Measurements of Hg released range from around 0.7 ng (46) to 1.4 ng (47) per breath. Therefore, even when Hg thermometers and barometers are discarded, there remain a few sources of Hg that are almost impossible to eliminate completely.

In industrial catalysis, where Hg vapour can poison pgm catalyst surfaces, Hg can be removed from feedstock chemicals by surface adsorption on metal sulfide pellets. Four pure metals have been used previously as Hg 'getters' in vacuum systems (48): sodium, gold, cadmium and indium. Oxidation of the metal surface can prevent Hg uptake, so for operation in air, of these four candidates only gold is suitable. We have therefore proposed (36) that it would be sensible to store kilogram prototypes under a thin, clean, gold foil or mesh to reduce Hg reaching the prototype kilogram. This gold 'mercury getter' could be inserted inside the existing glass bell jars in which kilogram prototypes are generally stored. It may be that this simple and inexpensive measure alone would halt the divergence of the prototype kilograms (42) suspected since 1939 and confirmed since 1992.

Conclusions

Kilogram weights made from Pt-Ir alloy have formed the basis of the SI unit of mass for over a century. The choice of material from which to make these weights was an excellent one, given what was known at the time. Barely a few decades later, increasing understanding about the catalytic properties of pgms (as well as the poisoning of such catalytic behaviour by traces of Hg, for example) presaged some modest problems of surface mass-stability that would be increasingly clear in weighing data in the second half of the twentieth century. From around 1990 modern techniques of surface chemical analysis shed light on the mechanisms of this mass instability. Recent measurements on six 19th century Pt artefact mass standards confirm the presence of Hg. It now seems almost certain that the prototype kilograms that form the basis of the SI have accumulated significant quantities of mercury contamination and that this has caused some (or perhaps most) of the divergence between kilogram prototypes observed in recent years.

Carbonaceous contamination built up over more than a century has been modelled and removed by well-documented solvent and UV/ozone cleaning protocols. Though there is a strong trend to remove all artefact standards from the SI (in the way that, for example, the unit of length has been redefined in terms of fundamental quantities instead of the length of a physical bar of metal) the kilogram is still defined as the mass of the international prototype of Pt-Ir alloy. Comparisons of mass using state-of-the-art balances are still more precise than the accuracy of other methods of realising a mass standard from fundamental constants or atomic properties. Therefore, once the stability issues arising from Hg and organic contamination are controlled, Pt-Ir prototype kilograms still make extremely good mass standards. It is just possible that they will continue to define the unit of mass in the SI for rather longer than expected.

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