COMPARISON OF NIST SI FORCE SCALE TO NPL SI MASS SCALE

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INTRODUCTION

Recent advances in device manufacture and object manipulation at the micro- and nanometre level have intensified the need for quantitative understanding of forces acting at this scale. Impacted industries include automotives, pharmaceuticals and communications. To meet this need, several major national metrology institutes (NMIs) have developed specialist balances to measure low forces traceable to the International System of Units (SI). These include efforts at the National Institute of Standards and Technology (NIST) [1], National Physical Laboratory (NPL) [2], Physikalisch-Technische Bundesanstalt (PTB) [3] and Korea Research Institute of Standards and Science (KRISS) [4]. Forces measured by the first three are ultimately traceable through electrical and length units, as opposed to the International Prototype Kilogram (IPK).

Well-characterised MEMS artefacts can be used to transfer force calibrations to users in industry [5]. The traditional choice for transferring a well-defined force, the weight of a known mass, is not suitable in the low force regime. Small masses are cheap but difficult to manipulate and easily contaminated in the typical industrial environment, invalidating associated calibration data. At the nanonewton level relative uncertainties typically approach and exceed unity.

Nevertheless, small masses are still very relevant for pharmaceutical dosing applications, where the infrastructure for the clean handling of small objects is already in place and uncertainty in mass measurement is not the limiting factor. Further, in the specialist facilities at the larger NMIs, small deadweights remain a suitable artifact for the validation of electrostatic force balances. Repeated weighings highlight systematic discrepancies, which, if explained in terms of base principles and verified independently of the weighings, lower the uncertainties on the traceability route through electrical units. These comparisons require an investment of time and equipment only practicable as a one-off.

This international comparison, funded by the UK National Measurement System Engineering Metrology Programme, is validated by the CIPM Mutual Recognition Agreement that allows quantitative comparability of work at different NMIs.

OVERVIEW

Small masses in the 1.0 mg to 0.1 mg range were developed and calibrated at NPL with traceability to the IPK. These masses were transported to NIST at Gaithersburg and used as deadweights on the NIST electrostatic force balance, to facilitate a mass-force scale comparison.

As a result of the experimentation a Type B uncertainty leading to a 10 nN under-read in weighing was highlighted. Subsequent work has fully identified and confirmed this systematic uncertainty and work is underway to eliminate it.

NPL MASS SCALE

Calibrated mass measurements at NPL are traceable to the IPK *via* kilogram 18. In order to minimise traceability uncertainties, small masses are calibrated by subdivision from the kilogram using an overconstrained matrix of equations. This standardised procedure permits estimates of the departure of the masses from nominal values and facilitates calculation of type A uncertainties. Comparisons are carried out on a suite of balances from several manufacturers. In this approach, only the repeatabilities of the balances are important; absolute values are not. Mass balances remain an acceptable tool for traceable milli- to micronewton force measurement, but care must be taken to understand the uncertainties behind the apparent ease of operation.

NIST FORCE SCALE

Traceability in the micro- to nanonewton range is realised at NIST using the Electrostatic Force Balance (EFB) in which studied forces are traceable via calibrated displacement, voltage and capacitance transducers. The balance consists of a nested cylindrical capacitor on a counter-balanced flexure system and interferometrically measured displacement is used to null the balance position to an appropriate set-point. The development of the EFB was particularly motivated by the need for traceable force measurement in MEMS devices and AFM, and has been successfully employed in this way [6].

In practice, the EFB performance is usually not limited by the uncertainties in the realisation of the electrical force, but rather by the ability to accurately compare this force to other mechanical forces using the balance mechanism. For example, for weighings at the level of tens of micronewtons, Type A uncertainty sources due to seismic and servo control noise have dominated the uncertainty budget. Similarly, for calibration of stiffness artefacts or force transducers, the uncertainty budget is dominated by contact mechanics, not by the EFB realization of force.

MASS ARTEFACTS, HANDLING

The NPL-developed small mass artefacts were formed manually from small-gauge aluminium wire and shaped to suit. Aluminium was chosen for increased mass size through low density and its non-magnetic properties. A number of small masses were prepared to allow optimised calibration with the NPL comparator balance and to allow for a range of experimentation. The masses ranged from 1.0 mg to 0.05 mg.

The repeated loading and unloading of the test mass on the EFB represents the greatest challenge to success. Considerable dexterity and patience is required for manual placement where the mass artefacts, as tiny springs, are easily ejected and lost. The EFB is equipped with a three-axis sample-stage micro-positioning system, the sample stage adapted to suit each experiment. The repeated exchange cycles, required to statistically reduce measurement uncertainties on the EFB, demand an extremely reliable exchange mechanism. A 'safely' dropped mass requires a full vacuum cycle to re-seat, which is costly in terms of time.

Two successful lifting mechanisms were developed for this work. Small wire masses were folded into a free-standing shape with a lifting point over the centre of gravity (Figure 1a, b). This latter specification prevents rotation on exchange that can cause the mass to 'walk off' the lift hook. Longer masses were straightened and exchanged between fixed and moving hook pairs (Figures 1c, 3). It is important to note that reshaping to suit a given set-up represents a significant risk to the test mass due to work hardening and subsequent failure.

For small objects at this scale surface interaction forces tend to dominate over gravitational weight and introduce considerable difficulties to otherwise straightforward mechanical operations. In air, humidity-dependent capillary forces adhere otherwise clean surfaces and suggest the need to reduce the interacting surface area. In vacuum these forces are removed, but electrostatic charge may perturb the measured force unless controlled through adequate grounding.

Under correct EFB displacement servo operation, the force output difference measures the applied weight. Figure 3 shows typical mass exchange force curves. Slight asymmetries in the hand-shaped masses result in two-stage exchanges. In the mass touch-down exchange in air, capillary hook-mass adhesion causes the still-lowering hook to pull the landed mass downwards such that the EFB registers a significant additional force. When the adhesion fails, the sudden force imbalance causes the platen to ring back to zero displacement. The size of the resultant error is not negligible due to otherwise insignificant effects such as flexure hysteresis. On air liftoff, and in vacuum, this adhesion is not observed.

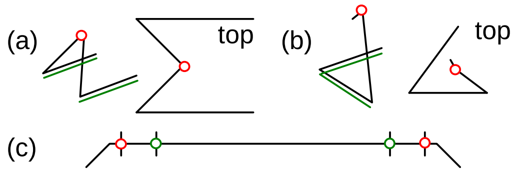

FIGURE 1. Masses reshaped for exchange (lifting points labeled red, mass touch-down points green). (a, b) are single hook systems, (a) the most stable; (c) is a two-hook system. Note end turndowns on (c) to prevent mass walk-off.

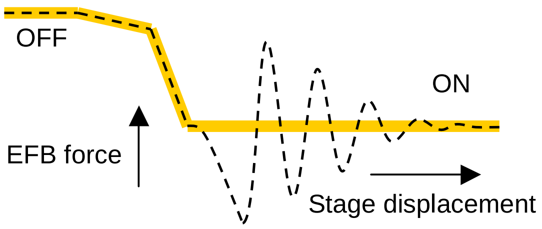

FIGURE 2. Mass exchange force curves. Black dashed is air touchdown curve, with adhesion and ringing on hook release; yellow solid is air liftoff and both transitions in vacuum.

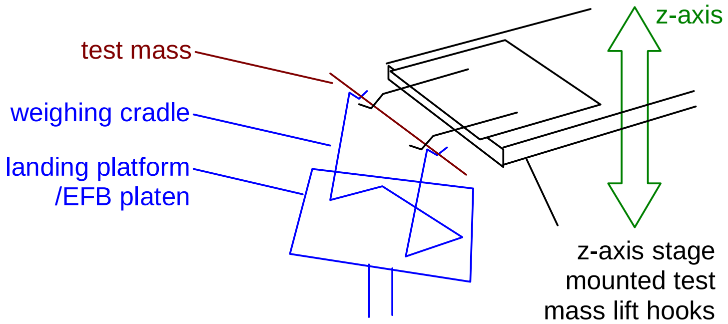

FIGURE 3. Mass exchange system. A Au/Pd coated thin glass square of side approximately 7 mm forms the landing platform. This accepts the mass directly, for single-hook experiments, or supports a weighing cradle as shown for two-hook experiments. The mass lift hook plate is floated on vacuum grease to attenuate stage mechanical noise; the lifter system is also Au/Pd coated to conduct away electrostatic charge in the exchanger.

For larger small masses (0.5 mg to 1.0 mg), denser metals were later found to be acceptable due to resultant smaller wire diameters and in turn smaller hook-mass interaction areas. This was verified qualitatively using 2.0 mg and 1.5 mg PtW alloy test masses. A smaller interaction area reduces capillary forces acting at the interface.

The vacuum-compatible precision stages used for the mass exchange were found to be a significant heat source when in closed-loop mode. Three energised axes produced a peak temperature as much as 20 ºC above ambient in sections of the metrology loop; for a single axis energised, the peak was 10 ºC above ambient. To overcome this rise, open-loop operation was used, with intelligent force-curve monitoring to detect lift-off and reset the encoder scales on each cycle. This was an acceptable approach because the exact position of the stage motion endpoints was not critical for the application, and errors scaled well with distance traversed.

RESULTS

Mass calibration

The full small mass artifact set was calibrated at NPL prior to experimentation at NIST. Values for the artifact subset used to obtain the reported results, specifically 1.0 mg, 0.8 mg and 0.1 mg, are given in Table 1. As per standard comparison procedures the masses were recalibrated on their return to NPL; this data is also reported. The calculated changes in mass across the calibration transfer for the 0.1 mg, 0.8 mg masses are within the $k = 2$ uncertainty for each calibration, suggesting the same for the 1.0 mg mass, which was lost before recalibration could take place. Thus the reshaping required between use at NPL and NIST appears to have had an insignificant effect on the total mass. Air calibrated values were buoyancy corrected using the known mass volumes and an estimate for air density at the NPL site.

Force calibration

After initial trial weighings with locally produced PtW alloy test masses, formal weighings of the mass subset were carried out in vacuum and air on the EFB using repeated mass exchange cycles. The vertical stage motion required to effect the exchange was minimised to increase cycling speed and was typically around 100 µm to 500 µm. Statistical analysis of the results of typically around fifty load cycles gave a Type A uncertainty in the final weight estimate of about 1 nN. A previously determined local gravitational acceleration value was used to convert weight to mass to enable comparison. The results are presented in Figure 4. The ‘force-scale’ error bars show the measurement set repeatability but neglect further unknown, possibly significant, systematic errors. The discrepancy of the 0.8 mg vacuum value against the other force-scale data is likely due to non-optimum conditions such as a thermal or mechanical non-equilibrium state.

Systematic error: sample stage magnetic crosstalk

In Figure 4, a systematic discrepancy in the measured weights of the calibrated masses inconsistent with the measurement uncertainties was highlighted.

TABLE 1. NPL calibrated masses, with values from before and after NIST experiments. k=2 uncertainties are 0.3 µg on each calibration and 0.4 µg on the combined average.

	NPL Calibration (µg)		
Nominal mass (µg)	Initial (June 07)	Repeat (May 08)	Change
1000	1002.3	*lost*	*n/a*
800	801.6	801.4	-0.2
100	105.8	105.9	0.1

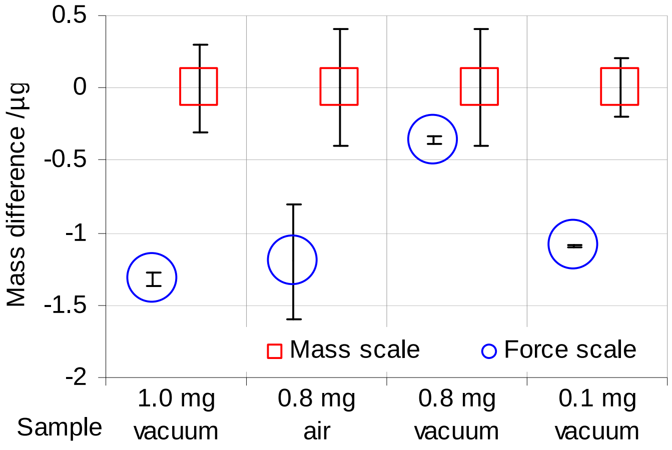

FIGURE 4. Key results of comparison between NPL mass and NIST force scales. Values are offset by the NPL mass calibration for each sample to give a 'mass difference'. An under-read in mass calculated from the NIST weighing was observed in each case. The values compare via a local gravitational acceleration, previously determined for the NIST laboratory, and a buoyancy factor determined from standard environmental factor values based on NPL laboratory specifications. The gravitational acceleration and buoyancy adjustments have uncertainties at the 5×10^{-6} and 1×10^{-6} level respectively.

Further experimentation identified the observed under-read as a product of an interaction between the EFB and the sample stage. Empty weighings, where the empty lifting stage was moved repeatedly between 'on' and 'off' park positions, recreated a fictitious negative weight with a dependence on the stroke (Table 2). The magnitude of the fictitious negative weight for the strokes used for the actual weighings is consistent with the weighing force discrepancies. It is suspected that the weak magnetism in the vertical stage used for mass exchange exerts a small force on the EFB's flexures, which are made from a ferromagnetic material.

A discrepancy of this magnitude had appeared in previous EFB-mass comparisons with larger masses [7]. In that case, the difference was 13 nN relative to a 20 mg test mass and was within the $k = 2$ uncertainty bounds. Note that this error is not a systematic in the force measurement itself, but is only due to the mass exchange stage motion. Therefore, it would not have influenced cantilever stiffness measurements or piezoresistive force transducer measurements previously made.

TABLE 2. "Empty weighing" forces.

On-off position stage stroke (10^3 actuator steps)	Apparent force (nN)
20	-5.0
40	-7.3
50	-11.9
100	-16.3

CONCLUSIONS

In this work the newly emerged independent force traceability route has been verified against the mature traceable mass scale at a level not previously attempted. The use of sub-milligram test masses on the NIST EFB has successfully led to the identification of a systematic error of nanonewton size that may now be removed or corrected for. This work exemplifies the benefits of international cooperation between NMIs to bring added value to their individual stakeholders.

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