Measurement of Submilligram Masses Using Electrostatic Force

Gordon A. Shaw, and Julian Stirling[1]

Abstract— **The redefinition of the kilogram within the International System of Units (SI) provides a direct link between mass and Planck's constant. With this in place, it becomes possible to realize the kilogram using electrical metrology. We describe a method that scales this mass measurement approach to the submilligram level using an Electrostatic Force Balance (EFB). Through traceable determination of capacitance, voltage, and position within the balance, the mass values of submilligram artifacts are determined. An uncertainty analysis is carried out on these measurements. Results show a substantial reduction in uncertainty relative to those currently available through conventional approaches based on kilogram subdivision for true mass. Since the EFB measurements are carried out in vacuum, conversion to conventional mass requires an air buoyancy correction at the location of use. Despite additional uncertainty added by buoyancy correction, the use of the EFB method decreases uncertainty in submilligram mass measurement by an order of magnitude.**

Index Terms—metrology; force measurement; weight measurement; capacitance; voltage;

I. INTRODUCTION

IN the context of the SI redefinition planned for 2018, small mass and force metrology stands to reap substantial benefits; this is especially true for the mass regime below 1 milligram. Prior to the redefinition, mass realization has been based on the International Prototype Kilogram (IPK). The preparation and dissemination of mass less than this involved making copies of the IPK, and then creating submultiples of the primary standard [1]. The process of subdivision requires many measurements to progressively work down from a kilogram to a milligram, typically in decade increments. Each of these increments adds additional uncertainty to the measurement. The combined expanded uncertainty in mass, U, arises from several terms as

$$U = 2\sqrt{u_{air}^2 + u_{ref}^2 + u_{bal}^2 + u_{am}^2 + u_v^2 + u_t^2 + u_g^2}\ , \quad (1)$$

where u_{air}, u_{ref}, u_{bal}, u_{am}, u_v, u_t, and u_g are uncertainties in air density, the reference mass used for subdivision, balance repeatability, the added masses sometimes used to compensate for buoyancy differences in masses of different materials, volume of the standard and unknown masses, temperature due to volume expansion of the masses, and variations in local gravitational acceleration, respectively.

Below a milligram, statistical uncertainty in u_{bal} becomes the limiting factor. As of the writing of this article, the lowest repeatability specification available in a commercial balance is 0.15 µg, essentially equivalent to the balance resolution. For the smallest available commercial mass, 50 µg, this translates into a minimum expanded uncertainty of 0.3 %. In practice, commercial test masses at this level have expanded uncertainties of 0.7 µg. It is apparent that substantial improvement is still possible with existing commercial technology if a reference with small enough uncertainty can be used to calibrate the weight directly.

The calibration of these small masses has already proven to be essential to a variety of fields. The testing of automotive particulate emissions requires mass measurements at the level of the lowest commercially available artifacts [2,3]. The scanning probe and instrumented indentation communities use masses this size and smaller to calibrate instruments to test nanometer-scale mechanical properties [2-5]. In emerging applications, submilligram mass has been used to establish SI traceability for laser and RF power measurements [6,7].

Recent work has shown that electrostatic force can be used to weigh milligram mass artifacts [8]. This study established the basis for an EFB to use traceable electrical and dimensional metrology in generating a primary reference for mass 1 mg and higher. In addition to a substantial reduction in

[1] Gordon Shaw is with the U.S. National Institute of Standards and Technology, 100 Bureau Dr., Gaithersburg MD 20899 USA (email: gshaw@nist.gov).
Julian Stirling is with the University of Bath, Claverton Down, Bath BA2 7AY, UK (email: J.Stirling@bath.ac.uk)

uncertainty, the EFB method also saves a great deal of time. Rather than requiring a series of 6 different subdivision experiments to bridge the gap from 1 kg to 1 mg, the EFB realizes mass directly at the milligram level, potentially taking weeks off the time required for measurement. In the following work, the process necessary to extend the method below 1 mg is described with an emphasis on the problems particular to realization of mass in this regime.

II. BACKGROUND

The basis for SI traceability in electrostatic force has been described in detail elsewhere [8]. Briefly, the electrostatic force between two elements of a quasi-one dimensional capacitor is

$$F_e = \kappa(V + V_s)^2 \ , \qquad (2)$$

where V is the voltage applied to the capacitors and V_s is a surface potential from patch effect or adsorbed surface contaminants and

$$\kappa = \frac{dC/_{dz}}{2} \ , \qquad (3)$$

where dC/dz is the gradient in capacitance, C, with position, z, between the two capacitor elements.

The nonlinear relation between force and voltage in electrostatic systems can be used to the benefit of small force measurement. To illustrate, consider a simplified case where V_s is negligible. The EFB mass measurements are performed as a differential weighing, in which a null balance position is held by changing the applied voltage. In the case where the mass is off the balance, a bias voltage, V_0, holds the balance at its null position such that the electrostatic force on the inner capacitor cylinder is

$$F_0 = \kappa V_0^2 \ . \qquad (4)$$

When the mass is placed on the balance, the bias force decreases (as the balance is adjusted so that the neutral restoring force is opposite gravity) so the force on the inner cylinder is

$$F_m = \kappa V_0^2 - mg \ . \qquad (5)$$

The change in voltage on the capacitor necessary to maintain the null position is therefore

$$V_d = \sqrt{\frac{\kappa V_0^2 - mg}{\kappa}} - V_0 \ . \qquad (6)$$

The V_d necessary to balance a hypothetical 100 μg test mass is shown in Fig. 1 as a function of V_0 for $\kappa = 5 \times 10^{-10}$ F/m. As the bias voltage decreases, the change in voltage necessary for a given change in electrostatic force increases. The Johnson noise of the amplifier used to apply the desired voltages is constant regardless of the voltage level applied. In effect, this means that by operating the balance in a low bias voltage condition, the relative effect of the amplifier noise can be reduced by a factor of 25 for the test case.

The EFB provides a mechanism for choosing the operating voltage point: the tension spring. Originally included to reduce the balance stiffness by applying an adjustable buckling load to the balance mechanism [9], the tension spring can also be moved vertically to change the voltage necessary to maintain null (i.e. V_0.) Electronic actuators on the buckling spring allow remote adjustment, so the optimum operating voltage can be chosen in-situ.

In practice, there are limitations to this approach. V_0 must be high enough for stable balance operation. The decreasing sensitivity of the force to voltage changes at lower V_0 will also mean larger effects from seismic noise and thermal expansion on the measured voltage. The latter problems can be minimized by appropriate filtering and averaging schemes during data collection.

III. EXPERIMENTAL

The specifics of the EFB used in this work are available elsewhere [8]. Briefly, the balance consists of a concentric cylinder capacitor attached to a 4-bar linkage mechanism with a counterbalance opposite the capacitor. The mechanism permits motion of the inner cylinder with respect to the outer cylinder along a rectilinear path, and details of the balance alignment are given elsewhere [8]. When actuated by an auxiliary electrode on the countermass side of the balance, the capacitance gradient in κ is measured at discrete points using an Andeen-Hagerling capacitance bridge[2] and a Zygo laser interferometer [10]. The aforementioned buckling spring is attached at the floating link of the 4-bar mechanism and to mechanical ground. The system is mounted inside a vacuum chamber operating at approximately 10^{-4} Pa.

A digital control system reads out position from the interferometer and controls to a desired setpoint by changing the voltage on either the auxiliary electrode (when capacitance

[2] Certain commercial equipment, instruments, or materials are identified in this paper to foster understanding. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

is measured on the primary electrode as in a capacitance gradient determination) or the primary electrode (when a test mass is being weighed) as illustrated in Fig. 2. A Kepco amplifier is used to increase the voltage output from the control system to the appropriate level.

An automated mass exchange system places the test masses on the balance and removes them repeatedly to perform differential weighing experiments where the voltage necessary to maintain the balance null position with the mass on and off the balance is determined. Each differential weighing is accompanied by a voltage polarity reversal to remove the effects of V_s [8]. Between 50 and 600 automated differential weighings are conducted per mass measurement, and each mass measurement is bracketed by a capacitance gradient determination.

Voltage metrology is performed with Keysight 3458A multimeters calibrated traceably to a Josephson Junction Array. The capacitance bridge is traceable to a Quantum Hall device through an AC-DC transfer experiment, but is realized in practice from a Calculable Capacitor. Position metrology is traceable to the stabilized He-Ne laser used for interferometry. Local gravitational acceleration was measured using an absolute gravimeter in the same room as the EFB.

IV. RESULTS

Data from a 500 µg weighing are shown in Fig 3. In this experiment, over 1000 differential weighings occurred over the course of two weeks. The weight remains constant during this time, within the measurement uncertainty (see further discussion below for uncertainty analysis), indicating that the mass is stable over the course of many weighings in vacuum.

Similar data for a 50 µg weight are shown in Fig. 4. Again, the mass is stable over the course of more than 1000 weighings. The substantial reduction in the statistical uncertainty shown by the error bars in Fig. 4 relative to the 500 µg data is partly the result of the larger number of weighings per trial (approximately 500, as opposed to 75 for the larger mass) and partly due to the operation of the balance at lower voltage, as will be discussed further below. The low frequency drift in the voltage signal is primarily attributed to small temperature changes in the balance causing thermal expansion. It is suppressed by the differential measurement process, which subtracts the linear measurement drift.

Table 1 shows a summary of the measured submilligram mass results and their combined standard uncertainties. The weights used in this study were either commercial wire masses from Mettler-Toledo, or custom masses fashioned from high-purity Aluminum wire obtained from Goodfellow. The approximate mass of the homemade masses was coarsely adjusted by hand and repeated weighing on a commercial Sartorius ultramicrobalance. A fine adjustment required an etch in 1 mol/L aqueous hydrochloric acid solution.

Mass exchange was performed in-situ in the EFB vacuum chamber using an automated system. The masses were hung from a hook connected to a closed-loop positioning system before pumping the system to vacuum. After desired vacuum and temperature stability was reached, this system would drop the mass on a double-tine mass holder attached to the EFB, returning to a home position for the voltage measurement. Subsequently, the mass would be picked up and voltage measurement repeated with the actuator in its home position to obtain a differential measurement of electrostatic force.

V. DISCUSSION

To illustrate some of the advantages of operating at lower bias voltages, it is useful to consider the raw voltage data. Fig. 4 shows the measured change in V for the positive polarity measurements in one of the trials of the 50 µg weighing. The measured voltage noise on this signal is approximately 10 mV for each of these voltage measurements. This noise level is approximately constant over the range of voltages used in the EFB (100 to 1000 V), indicating that it is dominated by contributions from the balance electronics, likely Johnson noise from the fixed gain amplifier. This is evident if one considers that if the balance mechanism were dominating the noise, the voltage noise at higher V_0 would be smaller due to the square law dependence of electrostatic force on the control voltage.

Representative uncertainty analyses for the 500 µg and 50 µg masses are shown in Table 2. A detailed explanation of the uncertainty contributions appears elsewhere [8]. It is worth noting, however, that a transition occurs in the submicrogram regime. Whereas masses above 1 mg, the EFB measurement uncertainty is dominated by systematic uncertainty (notably the temperature dependence of the capacitance gradient,) the statistical uncertainty begins to dominate at lower masses as the measurements approach the balance resolution. Statistical uncertainty is calculated from the standard deviation of mass values of separate daily trials and bracketed by capacitance gradient measurements. The balance mechanism stiffness of the EFB during these measurements is approximately 10^{-2} N/m, and the position noise is approximately 1 nm, leading to an ultimate force resolution of 10 pN (the gravitational equivalent of approximately 0.1 ng.) The uncertainty of the 50 µg artifact weighings shown in Fig. 4 are approximately 150 pN. Clearly, the real-world conditions of the measurement preclude operation at the ultimate resolution of the balance; seismic noise, nonlinear thermomechanical drift, and more subtle effects such as small amounts of swinging motion of the mass while it is on the EFB cause additional measurement noise to propagate into the statistical uncertainty. To the extent that these effects cause random variation, it is possible to reduce measurement uncertainty by averaging.

A longer term study would be necessary to search for correlations in the daily mass values, however within a daily trial it is possible to examine whether white noise dominates statistical uncertainty by examining the Allan variance [11,12]. For the 100 µg and 500 µg, the slope of the linear decrease in Allan variance with averaging time indicates statistical uncertainty is dominated by white noise within the daily trials. The 50 µg measurements exhibit a slower decrease with averaging time than that expected from pure white noise. An autocorrelation analysis shows correlation close to zero over the time of a single measurement, indicating 1/f noise is not significant on this timescale. It is possible that a random walk or drift is present in the statistics of the noise for very low values of mass; the use of the standard deviation of multiple trials as a measure of statistical uncertainty provides a conservative estimate of uncertainty in the short term. Longer term stability will be an area of future research, and care must be taken to distinguish between changes in the measurement processes, and changes in the weights themselves since they can change from wear or accretion of particles.

The voltmeters and capacitance bridge used in the balance have built-in internal references. This enables relative measurement uncertainty to be maintained at the level of 10^{-6} for extended periods of time. Calibration data from comparison to the primary quantum standards at NIST shows annual relative calibration drifts of less than 10^{-6} at 500 V for one of the voltmeters used in this study. Because of this stability and the fact that the mass value is realized directly at the mass of interest, a large number of measurements can be done to reduce statistical uncertainty. So far, the amount of time spent per mass is limited by practical concerns.

Practical concerns also dictate whether further uncertainty reduction for mass artifacts in the milligram to submilligram range is justified. As physical objects, the weights used as mass references change over time. Changes in mass much greater than the uncertainty were measured over the course of a year's time in previous work [8].

It is also important to consider that while the EFB measurements are performed in vacuum, the mass artifacts will mainly be used in air. The precise effect of transitions between vacuum and air on the surfaces of the mass artifacts is still an area of active research [5]. Although extensive work has been done to assess the surface science of stainless steel and Pt-Ir mass artifacts, the aluminum and aluminum alloy surfaces used for submilligram weights have not been extensively examined by the mass metrology community. There is some evidence that the surface water layer does not cause a significant difference between mass measurements performed in air and in vacuum [8].

Another consequence of the vacuum to air transition is the effect of air buoyancy on the weight of the mass artifact. The results of primary mass calibrations at National Measurement Institutes (NMIs) are reported as true mass; the effect of buoyant force has been removed. Although measurements performed in vacuum remove the effects of buoyancy for all intents and purposes, the end user must still account for air buoyancy to maintain measurement accuracy if the mass is used in air.

The correction for air buoyancy can be carried out using

$$m_a = m_t \left(1 - \frac{\rho_a}{\rho_m}\right), \tag{7}$$

where m_a is the artifact's effective mass value in air, m_t is true mass, ρ_a is the density of the ambient air, and ρ_m is the density of the material the mass is composed of. The determination of air density has been examined in detail in previous work [13]. It may be instructive to consider two limiting cases.

In one instance, for a measurement performed at 0 % relative humidity, 15°C, and 1050 hPa (the highest barometric pressure ever recorded on Earth), the density of air is 1.27 kg/m^3. Similarly, for a measurement performed at 100 % relative humidity 27 ° C, and 870 hPa (the lowest barometric pressure ever recorded on Earth), the density of air is 0.995 kg/m^3. The midpoint of these extrema can be used to estimate the density of air, and their difference can be used to estimate standard uncertainty yielding 1.133 kg/m^3 for air density and 0.138 kg/m^3 for the uncertainty in air density. This covers all reasonable terrestrial scenarios. The uncertainty estimated by calculating the minimum and maximum buoyancy correction for a 100 µg commercial wire test mass (ρ = 2700(140) kg/m^3) results in an extra relative uncertainty of approximately 3×10^{-4} at k=2.

Under tightly-controlled conditions in a metrology lab, where temperature is controlled to 20 °C within 0.01 °C, relative humidity (RH) is controlled at 40 % to within 0.3 %, and barometric pressure is near standard pressure and recorded with an accuracy of 10 Pa, relative uncertainty from the buoyancy correction is approximately 1×10^{-4} at k=2.

These estimated buoyancy uncertainties indicate that the calibration methods described in this work are near their current practical limit. It should be noted, though, that an improvement in density determination for the mass artifacts could reduce uncertainty further for end users of submilligram masses.

The use of the EFB method provides a reduction in mass uncertainty; Fig. 5 summarizes the uncertainties achievable with this method and those available with current accredited commercial calibration methods at the UK National Measurement and Regulation Office based on kilogram subdivision [14]. The addition of uncertainties from buoyancy corrections when the masses are used in air adds a small amount of uncertainty, but does not appreciably change difference in uncertainties for the two methods. The direct realization of submilligram masses using electrical metrology to replace kilogram subdivision provides significantly decreased uncertainties in the submilligram regime. International metrology activity in the area of submilligram mass metrology is already underway to examine the standardization of submilligram mass across international borders [15, 16] to provide continuity with current methods in this technologically important mass regime [17].

VI. CONCLUSION

The determination of submilligram mass with electrostatics provides an alternative to mass realization from kilogram subdivision. Traceability can be maintained through SI electrical units based on fundamental physical constants, and is therefore compatible with the SI redefinition planned for 2018. The EFB design discussed allows reduction in uncertainty by tuning the operating range of the voltage used to apply electrostatic forces to the balance mechanism. A full uncertainty analysis has been presented showing the EFB method reduces uncertainty relative to current methods. Although end users requiring buoyancy correction to operate in air will have a slightly higher uncertainty, the effect is small enough that the overall effect is still a reduction in uncertainty from the EFB method. Further work with mass artifacts to improve density characterization has the potential to decrease this uncertainty still further.

ACKNOWLEDGMENT

The authors thank the NIST Mass and Force Group for valuable discussions on buoyancy correction.

REFERENCES

[1] Z. J. Jabbour, S. L. Yaniv, “The kilogram and measurements of mass and force,” J. Res. NIST, vol. 106, no. 1, pp. 25-46, Jan.-Feb. 2001.

[2] DIN 32567-3: Production equipment for microsystems-Determination of the influence of materials on the optical and tactile dimensional metrology-Derivation of correction values for tactile measuring devices.

[3] EPA 40 CFR 86.1312-2007 – Filter stabilization and microbalance workstation environmental conditions, microbalance specifications, and particulate matter filter handling and weighing procedures.

[4] M.-S. Kim, J.-H. Choi, J.-H. Kim, Y.-K. Park, “SI-traceable determination of spring constants of various atomic force microscope cantilevers with a small uncertainty of 1 %,” Meas. Sci. Technol. , vol. 18, no. 11, pp. 3351-3358, Sept. 2007.

[5] G. A. Shaw, “Current state of the art in small mass and force metrology within the International System of Units,” Meas. Sci. Technol., vol. 29, no. 7, pp. 072001, May 2018.

[6] J. Melcher, J. Stirling, F. G. Cervantes, J. R. Pratt, G. A. Shaw, “A self-calibrating optomechanical force sensor with femtonewton resolution,” Appl. Phys. Lett., vol. 105, no. 23, pp. 233109, Dec. 2014

[7] P. A. Williams, J. A. Hadler, R. Lee, F. C. Maring, J. H. Lehman, “Portable, high-accuracy, non-absorbing laser power measurement at kilowatt levels by means of radiation pressure,” Opt. Exp., vol. 25, no. 4, pp. 4382, Feb. 2017

[8] G. A. Shaw, J. Stirling, J. A. Kramar, A. Moses, P. Abbott, R. Steiner, A. Koffman, J. R. Pratt, Z. J. Kubarych, “Milligram mass metrology using an electrostatic force balance,” Metrologia, vol. 53, no. 5, pp. A86-A94, Sept. 2016.

[9] J. R. Pratt, D. B. Newell, J. A. Kramar, “A flexure balance with adjustable restoring torque for nanonewton force measurement,” in Proc. IMEKO Joint Intl. Congress, Celle, GE, Sept. 24-26 2002, VDI-Berichte, 1685, pp. 77-82.

[10] J. Stirling, G. A. Shaw, “Realising traceable electrostatic forces despite non-linear balance motion, Meas. Sci. Technol., vol. 28, no. 5, pp. 055003, March 2017.

[11] D. W. Allan, “Statistics of atomic frequency standards,” Proc. IEEE, vol. 54, no. 2, pp. 221-30, Feb. 1966.

[12] D. W. Allan, “Should the classical variance be used as a basic measure in standards metrology?” IEEE Trans. Inst. Meas., vol. IM-36, no. 2, pp. 646-54, June 1987.

[13] A. Picard, R. S. Davis, M. Gläser, K. Fujii, “Revised formula for the density of moist air,” Metrologia, vol. 45, no. 2, pp. 149-155, April 2008.

[14] Mettler Toledo, “Traceability with microgram weights, Mass standards down to 0.05 mg,” White Paper, Greifensee: Mettler Toledo AG, 2014.

[15] S. Davidson, “Report on EURAMET.M.M-S2: supplementary comparison of 100 gram, 10 gram, 500 microgram, 200 microgram and 100 microgram weights,” Metrologia, vol. 45, no. 1A, pp. 07005, Jan. 2011.

[16] S. Davidson, A. Valcu, N. Medina, J. Zuda, L. Snopko, I. Kolozinska, “Supplementary comparison of 500 microgram, 200 microgram, 100 microgram and 50 microgram weights-EURAMET.M.M-S9,” *Metrologia*, vol. 54, no. 1A, pp. 07023, Jan. 2017.

[17] G. A. Shaw, “Scaling of mass and force using electrical metrology,” *Proc. CPEM 2018*, Paris, FR, July 8-13, 2018.

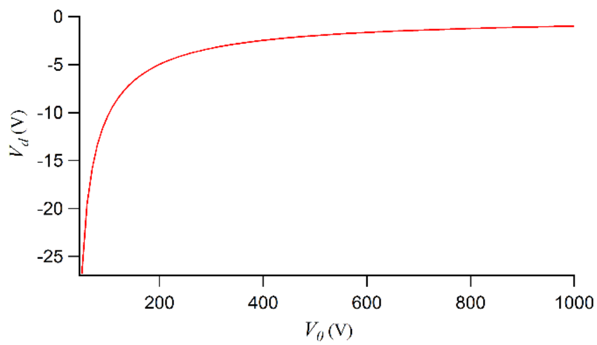

Figure 1. Simulation of the effect of bias voltage on the measured EFB voltage change for differential weighing of a 100 µg mass.

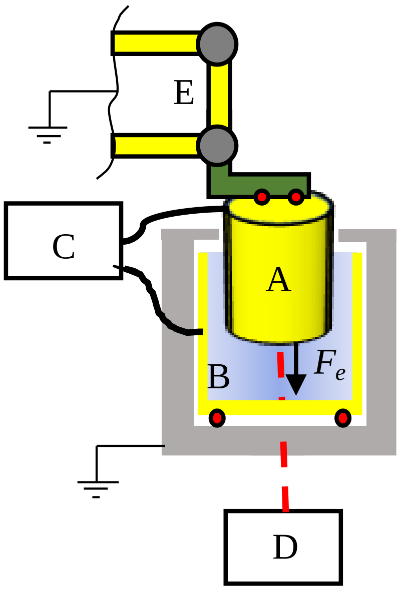

Fig. 2. Schematic diagram of the EFB. Inner capacitor cylinder (A) and outer capacitor cylinder (B) are connected to capacitance bridge (C) with coaxial cable (note, a relay network permits switching between the capacitance bridge and voltage amplifier, electrical connections denoted by solid curved lines). Laser interferometer (D) monitors the displacement of the inner cylinder guided by the balance mechanism (E), laser denoted by dashed line.

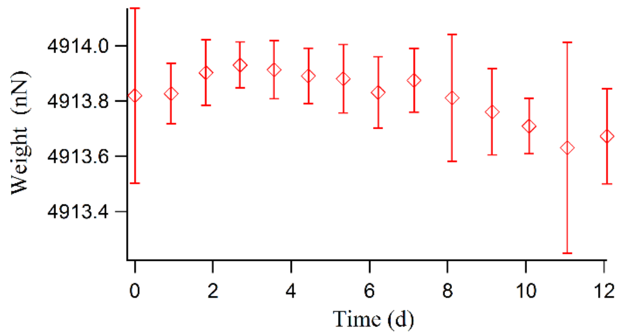

Fig. 3. Weight of a 500 µg mass artifact measured with the EFB. Error bars represent statistical uncertainty at k=2, determined from the standard deviation of the mean in the same fashion as [8], but doubled to accommodate the coverage factor of 2.

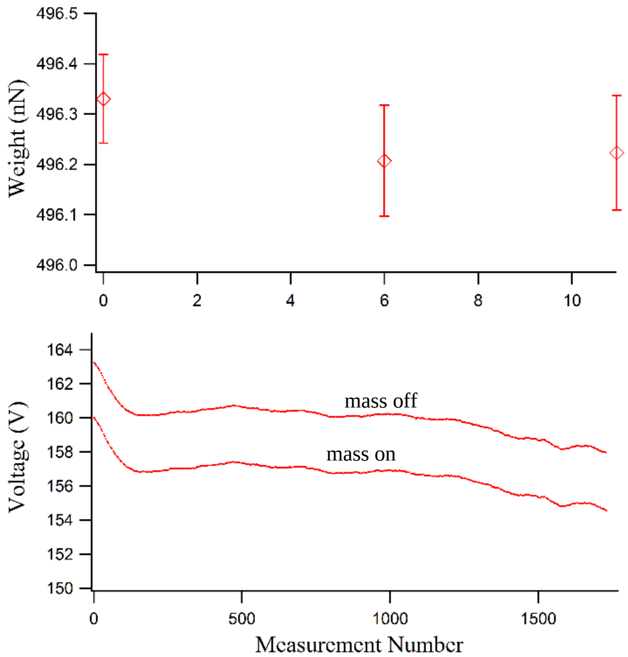

Fig. 4 Weight of a 50 µg mass measured with the EFB (top). Error bars represent statistical uncertainty at k=2, as described in the caption of Fig. 3. The voltage measurements used to determine the third weight in the top graph. Only the positive polarity data are shown.

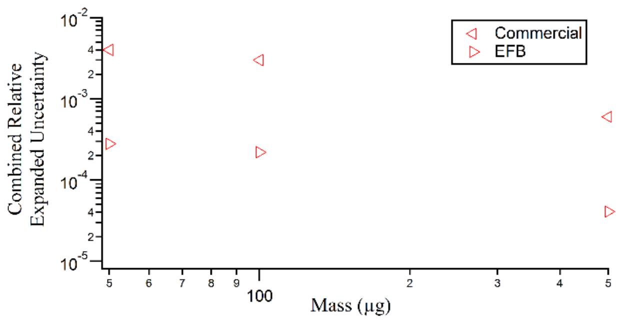

Fig. 5. Combined relative expanded uncertainty in submilligram mass calibration for submilligram masses. Data shown are the EFB calibrations shown in the current study and documented uncertainties from an accredited submilligram mass calibration facility.

TABLE I
EFB MASS MEASUREMENT RESULTS

Nominal Mass (µg)	Type of Artifact	Mass Value (uncertainty, k=2)
500	Aluminum wire, custom fabricated	501.83(2) µg
100	Commercial	101.52(2) µg
50	Aluminum wire, custom fabricated	50.63(1) µg

TABLE 2
UNCERTAINTY ANALYSIS OF EFB MASS MEASUREMENTS

Uncertainty Component	500 µg	100 µg	50 µg
Length Transfer	$1x10^{-7}$	$1x10^{-7}$	$1x10^{-7}$
Voltage Transfer	$4x10^{-6}$	$4x10^{-6}$	$4x10^{-6}$
Capacitance Transfer	$1.2x10^{-7}$	$1.2x10^{-7}$	$1.2x10^{-7}$
Stray Capacitance	$8.2x10^{-7}$	$8.2x10^{-7}$	$8.2x10^{-7}$
Capacitor Alignment	$1.3x10^{-7}$	$8.2x10^{-7}$	$8.2x10^{-7}$
Corner Loading	$6.0x10^{-7}$	$6.0x10^{-7}$	$6.0x10^{-7}$
Balance Hysteresis	$1.4x10^{-6}$	$1.4x10^{-6}$	$1.4x10^{-6}$
Balance Alignment	$7.3x10^{-7}$	$7.3x10^{-7}$	$7.3x10^{-7}$
Temperature Dependence of κ	$4.4x10^{-6}$	$4.4x10^{-6}$	$4.4x10^{-6}$
Statistical Weighing Uncertainty	$1.9x10^{-5}$	$1.1x10^{-4}$	$1.4x10^{-4}$
Combined Expanded Uncertainty	$4.1x10^{-5}$	$2.2x10^{-4}$	$2.8x10^{-4}$

Further information on this uncertainty analysis is available in [8]. Uncertainty components are shown as relative uncertainties at k=1, and combined expanded uncertainty is twice quadrature sum of uncertainty components.