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Development of a high precision balance for measuring quantity of dispensed fluid as a new calibration reference for the becquerel

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Abstract
The 2019 redefinition of the kilogram in the International System of Units (SI) connects the unit of mass with Planck's constant (h) through electrical metrology by means of precision balances. This not only changes the way the mass is defined, but also broadens the horizon for a direct realization of other physical units. Since mass measurement is essentially a force measurement scaled by the local gravitational acceleration, any physical action providing a force can be measured using a precision balance as a primary standard, connecting it to Planck's constant.

A new electrostatic force balance is under construction at the National Institute of Standards and Technology (NIST) to determine massic radioactivity of radionuclide systems by measuring the weight of dispensed radionuclide solution. The balance measures the differential mass (m) of a liquid droplet dispensed onto a superconducting transition edge sensor (TES). The TES measures the number and thermal energy of each radioactive decay process occurring per second, resulting in an activity measurement in becquerel, Bq. The combined results of these measurements estimates the massic radioactivity of the radionuclide soltution in $\mathrm{Bq{\cdot}kg^{-1}}$. Thus creating a direct relation between the Planck constant and massic radioactivity.[2]
The balance is designed to measure of approximately 5 mg (a volume of approximately 5 μL) with a relative uncertainty of less than 0.1 % with a coverage factor, $k = 2$. The liquid dispensing system is attached to the balance, whose mass is measured before and after dispensing the fluid. Doing this minimizes the impact of evaporation on the measured mass of the dispensed liquid [2].
The new system will be created as a compact tabletop version. A focus during the design is to create a highly automated system to speed up adjustment, balancing and capacitance gradient measurement processes. Another focus is to design the compliant guide mechanism in a modular fashion to vary its stiffness. Multiple means of stiffness reduction by preloading the guide mechanism are also being studied. A high degree of automation and the opportunity to adapt the flexural guiding mechanism of the system, to a specific use case, makes this electrostatic force balance attractive for scientific studies and industrial use.

1. Standard Reference Material

The NIST offers a number of Standards for radioactive activity measurement, and depending on the characteristics of the radioactive material, the standards can be in a gas, solid or liquid state. Because of the low uncertainty of the 4παβ liquid scintillation spectrometry method compared to other methods (see Table 1), liquids are commonly used as radioactive standards.

Table 1 Comparison of the NIST and NPL ^{210}Pb standards by five measurement methods [1]

Method	NPL/NIST ratio	Relative standard uncertainty, %
NPL and NIST certified values from primary standardizations	0.037484	1.5
4πγ(NaI) sandwich detector	0.037373	0.56
HPGe spectrometry	0.036542	0.71
4παβ(LS)	0.037249	0.17
^{210}Po assay (2πα Si spect.)	0.03736	0.75
Si(Li) low-energy spectrometry	0.0381	1.9

In addition, liquids are easy to handle and to portion during the production process of Standard Materials. Extensive characterization of the Standard Reference Material 4326a, also characterized by using the 4παβ liquid scintillation spectrometry method, makes it reasonable to use this standard for comparison with the method of this study to measure the massic alpha emission rate E_α in $\mathrm{kg^{-1}{\cdot}s^{-1}}$. This standard has a massic alpha emission rate of (39.01 ± 0.18) $\mathrm{g^{-1}s^{-1}}$ for $k = 2$ [1]. Reaching this uncertainty requires multiple purification and characterization steps. The spectrometry method uses a measurement principle based on photon counting [1], and takes round about 2000 hours to produce one Standard Reference Material 4326a. Furthermore, this production process requires multiple steps to refill the liquid resulting in extra effort to safely dispose of radioactive waste.

2. New measuring method

To reduce the production effort and radioactive waste, a new method is proposed. This method uses a transition edge sensor (TES) to estimate activity in the units becquerel by measuring the energy as a heat signature caused by radioactive decay. Since the dimensions of the transition edge sensor limits the size of the radioactive sample, the amount of dispensed liquid is limited to 5 mg. A volume of approximately 5 μL of radionuclide sample is dispensed onto a thin gold foil from a dispenser attached to the balance and placed on the TES. The change in mass of the dispenser's fluid reservoir is measured in-situ using the electrostatic force-based precision balance. The information from the TES and balance are used to determine the massic emission rate of the standard material in $\mathrm{Bq{\cdot}kg^{-1}}$. The primary goal for mass metrology is to measure a 5 mg mass of liquid with a relative uncertainty of 0.1 %.

To be able to achieve the metrology goal and to automate the preparation effort of the samples, a new electrostatic force balance will be designed. The new balance will be an absolute force balance, where the measured force is traceable to natural constants like the Planck constant via the redefinition of the kilogram.

3. Working principle of the electrostatic force balance

For the realization of the electrostatic force balance, multiple sub functionalities are required as illustrated in figure 1. The system requires a liquid dispensing system, which can store the radioactive liquid, L_1 and dispense a defined amount of liquid L_3 on a gold foil sample. The dropping system is attached to the guide mechanism of the balance. A number of samples are placed on another system, which feeds the unprepared samples S_1 and returns prepared samples S_2 on which the liquid is dropped. Due to the dispensed liquid, the mass (m) of the dispenser decreases, in turn reducing the force applied to the guide mechanism.

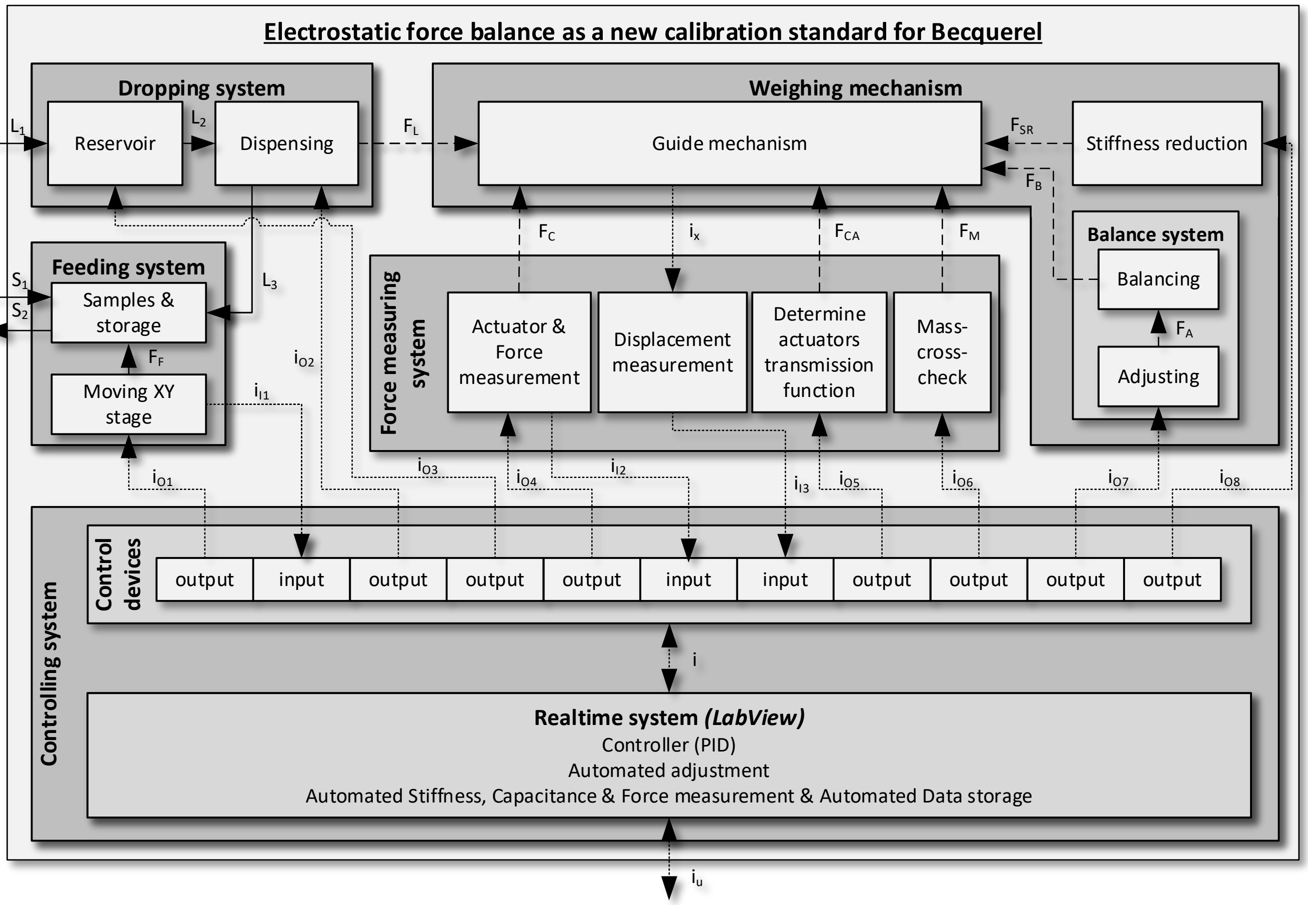

Figure 1. Functionalities of the electrostatic force balance to preparate the samples for the Transition Edge Sensor and measure the weight of the dispensed fluid. The arrows are illustrating: Liquids L, Samples S, Forces F and Information i.

$$F_L = m_L \times \vec{g}$$

The guide mechanism transfers and carries all the forces in our system and allows transverse movement in the direction of the gravitational vector. To keep the balance at its equilibrium position (also referred to as 'null position'), an electrostatic actuator applies a F_{el} using the displacement of the balance as the feedback measured using an interferometer. Due to the steady state displacement, Δx, of the guide mechanism, the stiffness, k, of the guide mechanism has an effect on the compensated force given by,

$$F_L = F_{el} + k\Delta \mathrm{x}$$

To reduce the impact of the balance stiffness, k must be reduced as much as possible by using different principles of stiffness reduction. In this case, and for a small displacement about a null position.

$$F_L = F_{el}$$

The applied electrostatic force F_{el} is given by the voltage, V applied to the electrodes of the actuator, and its capacitance gradient, dC/dx.

$$F_L = F_{el} = \frac{1}{2}\frac{dC}{dx}(V - V_s)^2$$

where V_s is a surface potential from patch effect that can be compensated by averaging the measurement of F_L at positive and negative polarity for V.[3]

Therefore, the mass of the dispensed liquid is given by,

$$m = \frac{1}{2}\frac{dC}{dx}\frac{\left(V_b^2 - V_a^2\right)}{g}$$

where V_b is the measured voltage averaged over positive and negative polarity measurements before the droplet is dispensed, and V_a is likewise after the droplet is dispensed. To measure the capacitance gradient dC/dx requires another subsystem that moves the capacitor to different locations along the x-axis while measuring the capacitance.

To communicate between all the subsystems, a control system with several inputs and outputs is necessary. And to keep the steady state displacement, Δx of the guide mechanism as small as possible, the controlling system uses a LabView realtime system, running on a NI PXI Linux system.

4. Subsystems of the experiment

A flow chart depicting of all these functionalities is illustrated in Figure 2 and a description of the design and realization is presented in the subsections below.

frequency disturbances from the environment. The modular design minimizes the effort in modification of the mechanism in case of damage. Because of the manufacturing method, the locations of the noches are well defined by the photomask, which increases the accuracy in translation motion and reduces the impact of the corner loading error [4]. In addition, the distance between the swivel joints in the upper and lower sheets (L_{Gx}) is equal to (L_{GY}) which reduces the impact of manufacturing tolerances occurring a corner loading error [4].

4.2. Counter balancing

Before dispensing the liquid, to hold the balance in an equilibrium position, a lever arm is connected to the guide mechanism. For this, a copper beryllium sheet with a low bending stiffness in z-axis can be used. The rotation joint of this lever will also be realized using copper beryllium sheets. To increase the operation speed of the balance, the adjustment of

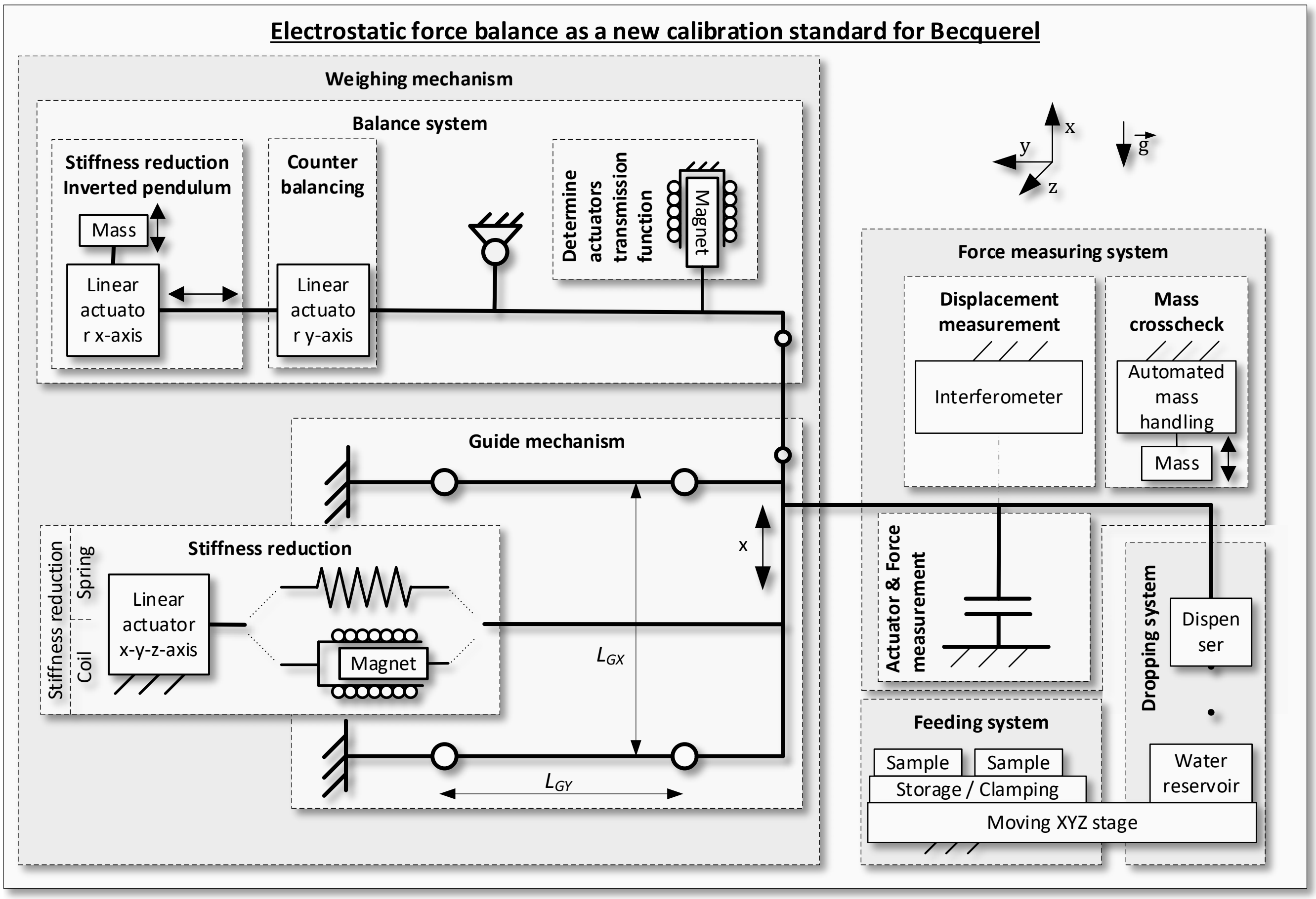

Figure 2. Technical principle of the electrostatic force

4.1. Guide mechanism

The compliant four bar linkage guide mechanism provides repeatable motion in the direction of gravity. In order to design the balance in a tabletop form factor, the distance between the swivel joints (L_{GY}) is limited to 100 mm. To reduce the hysteresis and difficult to characterize effects due to the clamping of intermediate links, both the links and joints of the mechanism are manufactured from a thin copper beryllium sheet. Flexible joints are produced by locally etching the thickness of the sheet (referred to as notches). Copper beryllium is chosen because of its small mechanical loss factor to reduce the hysteresis effects resulting from deformation of the notches. Realizing the four-bar mechanism using two of these sheets reduces system mass, increasing the natural frequency and reducing the impact of low

the counterweight in the y-axis can be automated by using an inchworm-type piezo actuator. Under consideration of thermal expansion of the lever arm and the four bar linkage, the lever arm can be designed to have the same thermal expansion to minimize relative movement between the guide mechanism and the lever arm in y-direction. To increase the thermal stability of the system, the two swings of the lever arm will be designed with the same length.

4.3. Stiffness reduction

Two methods of stiffness reduction are being considered.

(i) Another mass mounted on the lever arm can be adjusted automated in the x-axis to act as an inverted pendulum [4].

(ii) A spring or a voice coil can be mounted in the center of the guide mechanism. To vary the preload, the spring can be adjusted in x,y,z direction [5], or the coil voltage can be changed.

4.4. Force measuring system

The electrostatic actuator to hold the balance in the null position is realized using a capacitor, controlled by measuring the displacement using an interferometer. Three configurations of the actuator that are being considered are:

(i) Both electrodes are made of flat surfaces. This configuration reduces the manufacturing effort, but is susceptible to the tilt alignment of the faces of the electrodes.
(ii) One electrode is a sphere, and the other is a flat surface. This configuration reduces the effort of tilt alignment.
(iii) Two concentric cylinder configurations, which results in an approximately linear capacitance gradient for a long travel range (millimeters).

A goal is to design the capacitor in a modular fashion, to test the above mentioned configurations. Because of the fact, that every liquid drop is unique with a specific mass, a pick and place robot will be used to handle reference masses to calibrate the force measurement of the balance, and to determine its repeatability.

4.5. Dropping system

The dropping system is realized by using an automated lightweight inkjet-type dispenser. In order to minimize the weight of the balance and the maximum fore range, the dispenser is refilled by using the “dip and sip” method. Therefore the dispenser dips into a reservoir and aspirates the liquid by the capillary effect. To minimize shocks in forces, to the actuator, and to be able to dose the amount of dispensed liquid, the drops have the size of 20 to 80 µm. To reduce the impact of drift during the measurement and to speed up the preparation, the dispenser operates at high frequency [6]. To reduce the corner loading error, the dropping system is aligned to the center of the capacitor along the x- and z-axis [4].

4.6. Feeding system

To automate the whole process, a feeding system carries the small gold foil samples, with a size of round about 2x2 mm and a thickness of 15 µm, in a defined position to place the samples under the dropping system. The feeding system is moved by an automated XYZ stage.

4.7. Measure capacitance gradient

To measure the capacitance gradient, the capacitor is moved to different locations along the x-axis by using a voice coil actor, which is applied to the balance system. In each location, the capacitance is measured by a capacitance bridge.

5. Outlook

NIST runs similar electrostatic force balance projects. One example is a balance that measures the photon pressure of a laser beam which is pointed to a mirror, mounted to the balance. The force can be measured with an uncertainty of lower than 0.1 % for a mass of 35 mg [7]. This balance is also designed as a tabletop instrument, operating in air. Compared to this balance, the uncertainty in the new setup will be reduced by

(i) Reducing the impact of hysteresis, by using cooper beryllium instead of aluminum alloy 7075.
(ii) Reducing ground vibration operating in a higher natural frequency by reducing the weight of the mechanism.
(iii) Reducing the impact of airflowby not using a mirror

Therefore an uncertainty of 0.1 % for masses of 5 mg in the new setup is achievable in principle based on improvements to previous work.

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