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SOME MASS MEASUREMENT PROBLEMS

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Faced with the problem of determining the thickness of a target, one perceives that an uncomplicated approach is to measure its mass and area and take the quotient. This paper examines the mass measurement aspect of such an approach.

The first question encountered is the selection of a balance. There is a wide choice commercially available: the classical 2-pan balance, the single-pan substitution balance with a direct reading optical scale and the more recent electronic balances, some of which have 2 pans and others are substitution. In addition to examining the specifications for capacity, size of weighing chamber, readability, convenience and precision, it also is necessary to consider whether an adequate balance room can be provided, because most types of balance will not perform satisfactorily in an unsuitable environment and the degree of adverse affect varies.

Generally desirable features for precise weighing are as follows(1): a sufficiently large room so that the presence of the operator will not significantly alter the room temperature or air currents, a heavy vibration free balance table that does not contact the walls of the room, as these readily transmit building vibrations, absence of direct sunlight particularly on or near the balance, location and selection of lighting fixtures so that they do not heat the balance or the objects to be weighed, and airconditioning that minimizes draughts, controls humidity, maintains the temperature constant to ± 0.5°C and allows a small positive thermal gradient, i.e. warmer at the top. Preferably the balance room should be located in the basement or on the ground floor and well away from elevators.

For the classical 2-pan balance all of these balance room qualities are essential and it is worth noting that a negative thermal gradient within the balance case has been observed to give large instabilities, e.g. changes of 0.6 mg with a 200-g balance when the top was 0.4°C cooler than the base(2). Unfortunately most air-conditioning systems blow cool air across the top of the room and create the undesirable thermal gradient; this can be corrected by directing the air flow with baffles and low speed fans. Some scientists prefer to turn off the air-conditioning system for the weighing period if it will give conditions of slowly rising room temperature with the temperature change of the balance itself lagging slightly behind(2).

The substitution-type balance (2 knife-edge single pan) is less sensitive to thermal gradients and draughts and thus has become the choice of many workers. Nevertheless a well designed balance room is advantageous for this type of balance also. A sturdy balance table is a necessity, as the substitution-type balance with an optical scale is very sensitive to tilt and so must be maintained level. Another important requirement for these balances is temperature control to reduce error caused by zero drift. Figure 1 shows this effect for a particular model of substitution balance that gives readability of 1 µg and has a capacity of 20 g. The zero drift is not negligible and seems related to the temperature fluctuations which are indicated by the triangles; closer temperature control clearly would be worthwhile.

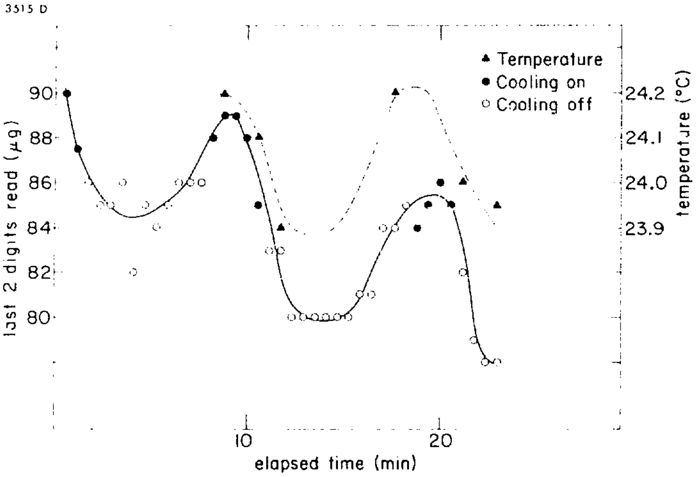

Figure 1: The effect of temperature fluctuations upon the zero reading of a Mettler M-5 balance (reproduced from Nuclear Instruments and Methods 112 (1973) 329).

The electronic balances are reported to be less sensitive to environmental conditions. However, the sail effect caused by air currents acting on the pan is as serious for these balances as any other, so that the usual precautions for the control of thermal disturbances should be taken. For those balances that do not have knife edges, however, the vibration-free requirement is not severe.

Another consideration that may affect the choice of balance are the maintenance requirements. All balances should be serviced and cleaned by an expert at least once a year. A knife edge can be seriously damaged by a small particle of dust or dirt lodged between it and the plane on which it rests during a weighing. Thus cleanliness is important. At the first sign of performance deterioration, the knife edges and planes should be cleaned to prevent further damage. Apart from mechanical damage caused by dirt, any accumulation of dust, dirt or films on the pans or the weights will cause error. This is a good reason for putting a "NO SMOKING" sign on the door to the room. It also is a reason why the substitution type balance has come to enjoy such widespread use, i.e., the built-in set of weights is enclosed and protected from room dust, as is the weight manipulation mechanism; thus the weights do not have to be cleaned and recalibrated nearly as frequently as a set of weights that is hand manipulated for every weighing, such as for the classical 2-pan balance or most electronic balances.

Other reasons for the popularity of substitution balances, which also pertain for the electromagnetic balances are the speed of response, direct reading and relative independence of the sensitivity with load. Speed is valuable not only as a time-saver, but also because it reduces error caused by zero drift or mass changes taking place in the sample being weighed, such as oxidation or evaporation. An interesting study of the basic design features and the relative advantages of the substitution-type and classical 2-pan balance was published by Peiser(3) who strongly recommends the substitution balance for routine weight calibration work.

The U.S. National Bureau of Standards (NBS) has established various classes of weights(4) for which the materials and construction details of the weights are described, as well as their allowed limits of deviation from their nominal values. Most balance manufacturers and suppliers of sets of weights have chosen to produce weights that conform to the NBS requirements for these various classes, probably in order to market their products readily in the U.S.; but fortunately the result is a well defined set of tolerances for the weights used in most commercially available balances. As an example the NBS tolerance requirements for class M are given in Table 1. Sets of weights meeting such specifications are commercially available. Calibration service according to an NBS procedure(5) can also be purchased. In many countries the national standardizing laboratories have cooperated in providing weight calibrations to satisfy particular scientific needs.

The material used for the fundamental standards of mass, the international prototype kilogram, is a platinum iridium alloy. The recommended material for high quality weights in the 1 g - 1 kg range is a chrome nickel steel; smaller weight pieces are made of tantalum and in the mg range aluminum is used. Brass weights electroplated with gold or rhodium are no longer favoured because they were found less stable to changing atmospheric conditions. It is important that

Table I

NBS Tolerances for Class M Weights

Denomination	Individual tolerance (mg)	Group tolerance (mg)
100 g	0.5	not specified
50	0.25	
30	0.15	
20	0.10	
10	0.05	
5	0.034	0.065
3	0.034	
2	0.034	
1	0.034	
500 mg	0.0054	0.0105
300	0.0054	
200	0.0054	
100	0.0054	
50	0.0054	0.0105
30	0.0054	
20	0.0054	
10	0.0054	
5	0.0054	0.0105
3	0.0054	
2	0.0054	
1	0.0054	
0.5	0.0054	

The deviation of a weight from its nominal value shall be small enough: (1) that it falls within the individual tolerance listed and (2) that no combination of weights within its group shall differ from the sum of the nominal values of the combination by more than the amount listed under the group tolerances.

weights be handled with forceps whose tips are of a softer material than that of the weights themselves; ivory tipped forceps are often used. The forceps should be sufficiently long that the operator's hand does not heat the weights nor enter the weighing chamber during manipulations. Also important is that the weights be kept clean. As well as taking normal precautions about cleanliness, a daily wipe with lens paper is a good idea. More serious cleaning is advisable from time to time and various methods have been advocated. Among them is one in which the weight pieces are rubbed with a cleaned wet chamois cloth then dried with a cleaned dry chamois(6); the chamois cloths are cleaned by soaking in soluble detergent solution then rinsing many times with pure water to completely remove the soluble detergent. Table II shows an example of some high quality weights that were cleaned in this manner; before cleaning the weights looked bright and shiny yet this cleaning procedure removed considerable dirt from 2 of them, then gave an acceptably constant weight value after repeated cleaning. Some laboratories advise cleaning with a jet of steam, particularly for 1-piece weights(7). Wiping with a soft camel-hair brush is frequently done, and here again, the brushes should be cleaned in detergent, rinsed with pure water, then alcohol and allowed to dry(7).

Table II

Results of Cleaning Weights

	1g	$2g_1$	$2g_2$
Before	1.000066	1.999985	1.999973
After 1st cleaning	1.000042 1.000040	1.999961 1.999962	1.999975 1.999972
After 2nd cleaning	1.000041 1.000041	1.999963 1.999963	1.999976 1.999973
1 day later	1.000039 1.000040	1.999963	1.999975

All materials used - weights, brushes, forceps and objects to be weighed - should be placed in the balance room for at least an hour before weighings are performed so that they will be in thermal equilibrium and therefore not cause any disturbing convection currents or buoyancy changes. It has been claimed that improved balance performance is obtained if the operator sits in front of the balance about 15 minutes before commencing a series of weighings.

As a further precaution against thermal disturbances a reflecting blanket of aluminized mylar has even been worn by the operator when performing weighings using a balance with readability of 0.1 μg.

The performance of any balance should be tested routinely to ascertain whether any problems have developed. Some excellent test procedures have been described in the literature(8,9) that give good outlines for simple tests and are designed so that the sequence of operations is representative of actual weighings. Such procedures test the direct-reading scale to see if the deflections over the lower and upper halves are equal. A difference of more than 3 standard deviations is a clear indication of trouble; it may be caused by electrostatic effects, dirt or damaged knife edges. Other tests should be run to monitor the precision, the course of the zero with time, and the sensitivity. Supposing that after running such tests, one finds that the zero drift is significant and follows some pattern, such as temperature fluctuations shown in Fig. 1; this is a source of possible systematic error. Accordingly the appropriate weighing procedure to follow is one that takes it into account, e.g. the ASTM recommendation(8) in which the zero is observed just before and right after a pair of weight observations; the average of the 2 zero readings is subtracted.

The set of weights should be tested and if necessary recalibrated. Many weight calibration procedures have been published, both for sets of weights(6,10) as well as for the built-in weights of substitution balances(8,11). Also the direct reading scale should be calibrated. An example of this is the procedure of Bowman et al(12) who observed the response of the indicating scale to the addition of the same small weight at different points along the scale. Small non-linearities are common in the optical scales of substitution balances and calibration will provide suitable corrections that can be applied; however electromagnetic scales are fundamentally linear and so any observed non-linearity indicates trouble that needs the attention of service personnel.

Having tested and calibrated an instrument and taken some observations on the weight of a target, the final problem is to compute the target's mass. First the corrections to the weights used for a given observation are applied. Also any correction for the on-scale reading is applied. Then, except for specialized work where the weighing is done in vacuum, the correction for air buoyancy(1,12-14) must be computed. Some examples of the magnitude of air buoyancy corrections for observations versus steel weights are given in Table III. The relationship of the true mass of an object, m, to the observed reading m_a, when it is weighed in air versus weights of known mass is given by the equation

$$m = m_a \left[1 + \rho_a \left(\frac{1}{\rho_m} - \frac{1}{\rho_w} \right) \right] \qquad (1)$$

where ρ_a is the air density, ρ_m is the density of the object weighed and ρ_w is

Table III

Examples of Air Buoyancy Corrections

Object weighed	% Correction
Platinum	- 0.009
Germanium	- 0.002
Aluminum	+ 0.035
Beryllium	+ 0.05
Water	+ 0.10

the density of the weights. The air density can be computed from observations of the temperature, pressure and relative humidity and it varies by about 3% in any one location for the normal fluctuations in atmospheric conditions(1,12); typically at sea level it is 1.18 mg cm^{-3} and at elevations of 1500 m about 1.0 mg cm^{-3}. For a rigorous treatment of the air buoyancy correction, the densities of the object weighed and the various weights used should be determined; this is a great deal of work; furthermore it frequently happens that weights of several materials will have been used in performing a weighing, e.g. stainless steel for the ≥ 1 g weights, aluminum for the < 50 mg weights and tantalum for the intermediate weights, and this adds to the complexity. So for routine work this becomes impractical.

Thus the apparent mass basis was established to simplify the problem and yet remain sufficiently accurate for most work(1,13). The basis is this: that weights made of various materials are adjusted so that they appear equal to their *nominal values when* compared in air with weights of the "ideal" density. The normal conditions specified for air density are 1.2 mg cm^{-3} at 20°C; in the U.S. the specified "ideal" density is 8.4 g cm^{-3} at 0°C (corrected for thermal expansion it is 8.3909 g cm^{-3} at 20°C) and most commercial equipment is adjusted on this basis; however, in some countries 8.0 g cm^{-3} has been selected as the *ideal density*(15). Thus it is important that the user ascertain from the manufacturer's data on what basis the weights are adjusted so that the appropriate density value (8.0, 8.39 or some other) is used in computing the buoyancy correction according to equation (1). Adoption of the apparent mass basis adds an inaccuracy of $< 10^{-4}$% unless the air density is considerably lower, e.g. *at elevations of 2000 m the* error introduced is $\approx 3 \times 10^{-4}$%; even this is acceptable for most purposes and very likely is a negligible part of the error in determining small masses.

Workers in many fields have reported precision such as is shown in Table IV. With sensitive instruments, precision of ± a few μg frequently is obtained.

For our purposes one concludes that the accuracy of mass determinations of targets should be similar to this precision if care is taken in maintaining the performance capability of the balance and in its calibration.

Table IV

Precision Reported by Workers in Various Fields

	Precision	Load	Type of Balance
Bowman *et al.*(12)	± 60 μg	1 kg	Substitution
BIPM(16)	± 1.5 μg	1 kg	Classical 2-pan
Almer *et al.*(11)	few in 10^7	100 g	Semi-micro substitution
Lashof & Macurdy(9)	± 44 μg	40 g	Semi-micro substitution
Marinenko & Foley(17)	± 3 μg	2 g	Micro substitution
Henins(18)	± 1 μg	5-18 g	Micro substitution
Green(19)	± 2 μg	1 g	Classical 2-pan
Bowman *et al.*(12)	± 0.5 μg	2 g	Electronic
Green(19)	± 0.1 μg	various	Electronic

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