A Primer on Weighing Uncertainties

in Radionuclidic Metrology

(<u>not</u> the uncertainty associated with mass calibrations)

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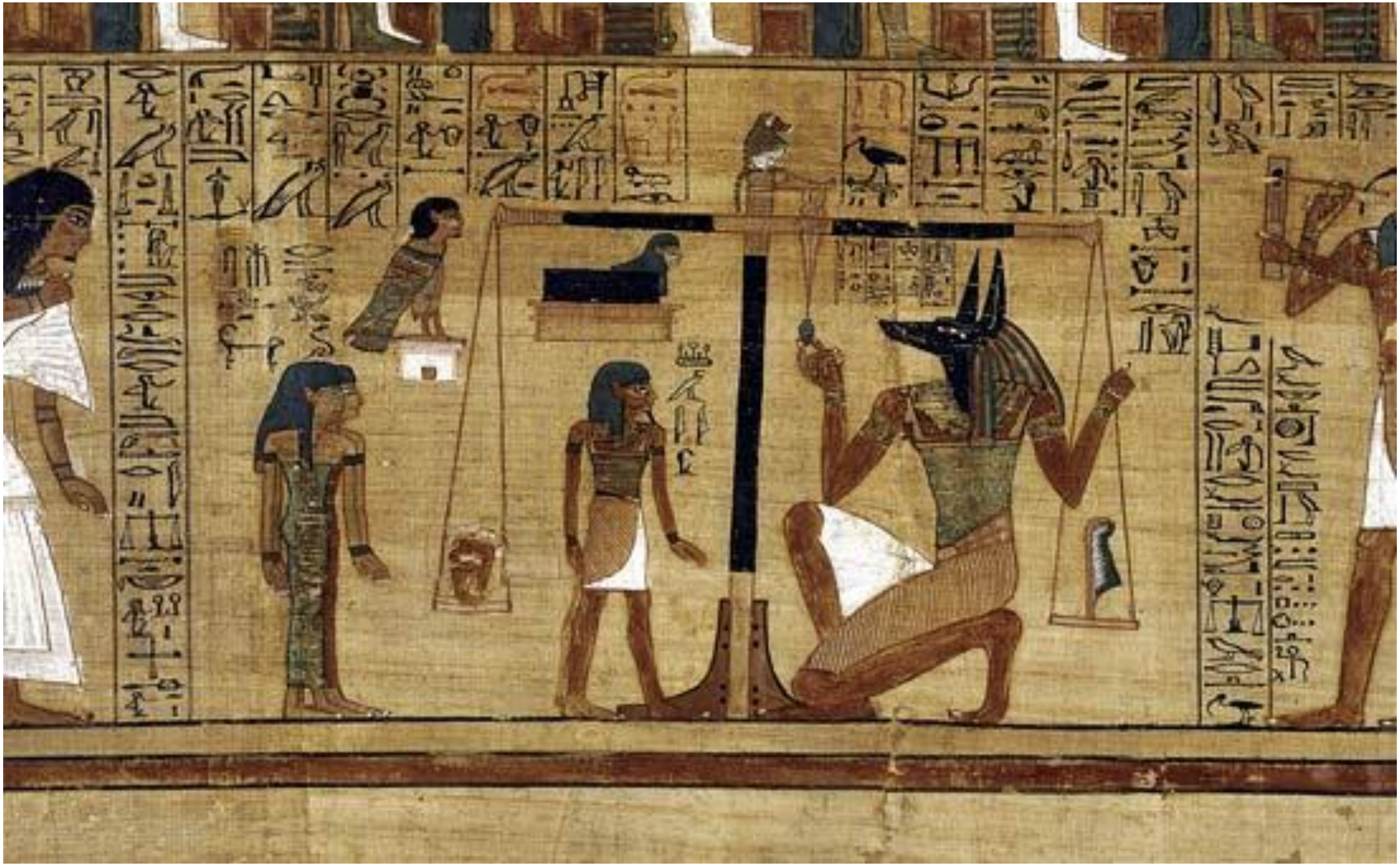
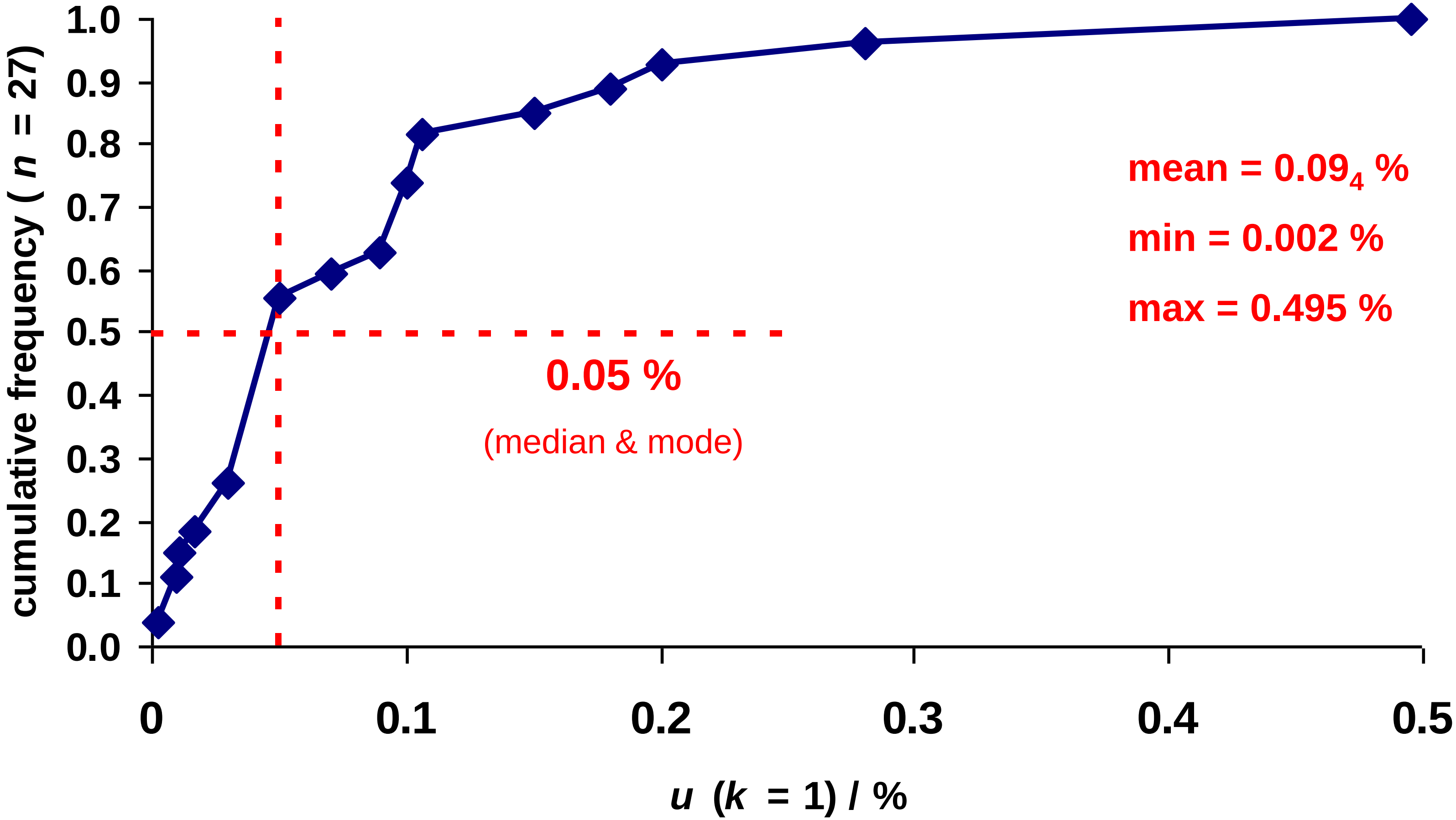
Anubis weighing the soul of the scribe Ani, from the Egyptian Book of the Dead, *c.* 1275 BCE.



WEIGHING UNCERTAINTIES REPORTED FOR ^{55}Fe COMPARISON



SOME PERSONAL OBSERVATIONS / OPINIONS

- weighing uncertainty not generally a major or dominant uncertainty component
- weighing uncertainty does not appear to be assessed very rigorously
- assignment of weighing uncertainty appears to be largely guessed at – or based on past practice & basis (but conditions have changed)
- weighing uncertainty (particularly for small masses – microweighing) is probably underestimated
- guidance on weighing uncertainty has not kept abreast of changes in uncertainty treatments (GUM)

1970's radionuclidic metrology -- ICRM

MICROWEIGHING for source preparation

Some excellent work

Janet Merritt (AECL)

Y. Le Gallic (LMRI)

NPL, NBS/NIST, et alia

emphasis on ≈ 20 mg

M-5 mechanical balance w/ built –in weights

Systematic evaluations of balance performance

Great guidance on practices

Even comparisons to test weighing

20 mg

best precision 2 μg

best 5 – 10 μg

typical ≥ 20 μg

can reach 100 μg

NOW ??

more applications (larger mass range)

+ bigger sources ! – LS

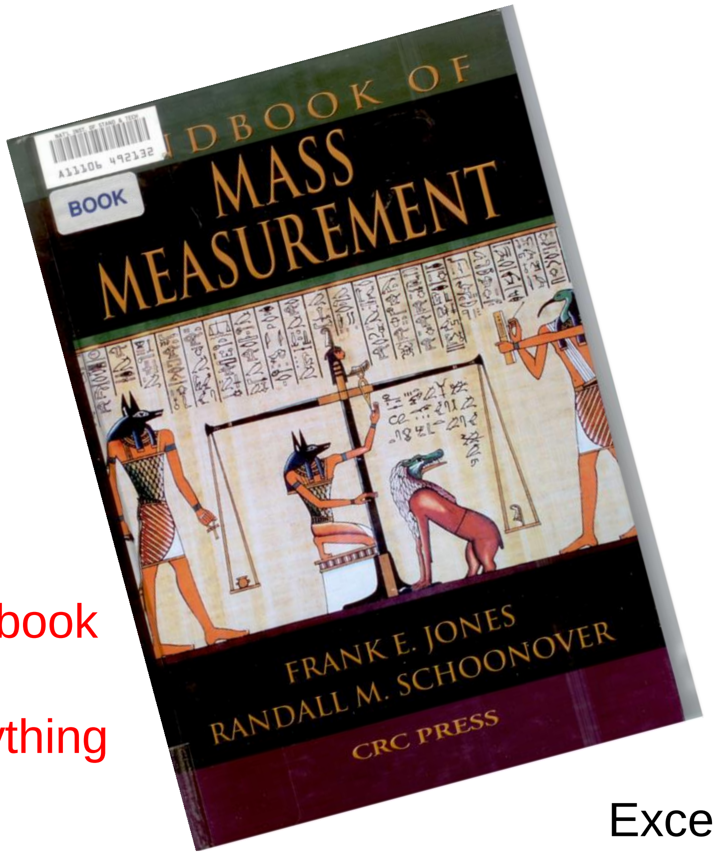
"electronic" EM force compensation balances

less "care" in labs ?

some preliminary stuff before starting

Many, many references available from the mass people in **<u>YOUR</u>** laboratories

particularly for mass calibrations



Principles,

Good practices,

Conditions

Weighing procedures

Corrections

Uncertainty treatments

this handbook has everything

Excellent documents from NPL, PTB, NIST, others

Mass is a physical quantity

Weighing is a measurement process

(used to obtain mass)

We measuring (certifying) massic activity, not mass ...

So we <u>want the uncertainty associated</u> with the

<u>weighing process</u> (includes mass standard)

We measure

Conventional Mass

(Mass in air versus reference density of 8.0 g/cm3)

<u>NOT</u>

True Mass ***(Mass in vacuum)***

Weight ***(gravitational force acting on mass)***

The ***conventional mass value*** *of a body is equal to the mass of a standard, of conventionally chosen density, at a conventionally chosen temperature, which balances this body at this reference temperature in air of conventionally chosen density.*

formal definition

The conventionally chosen conditions are:

reference density = 8.0 g·cm^{-3} ;
reference temperature = 20 °C;
normal air density = 1.2 mg·cm^{-3}

The unit of the quantity “conventional mass” is the kilogram.

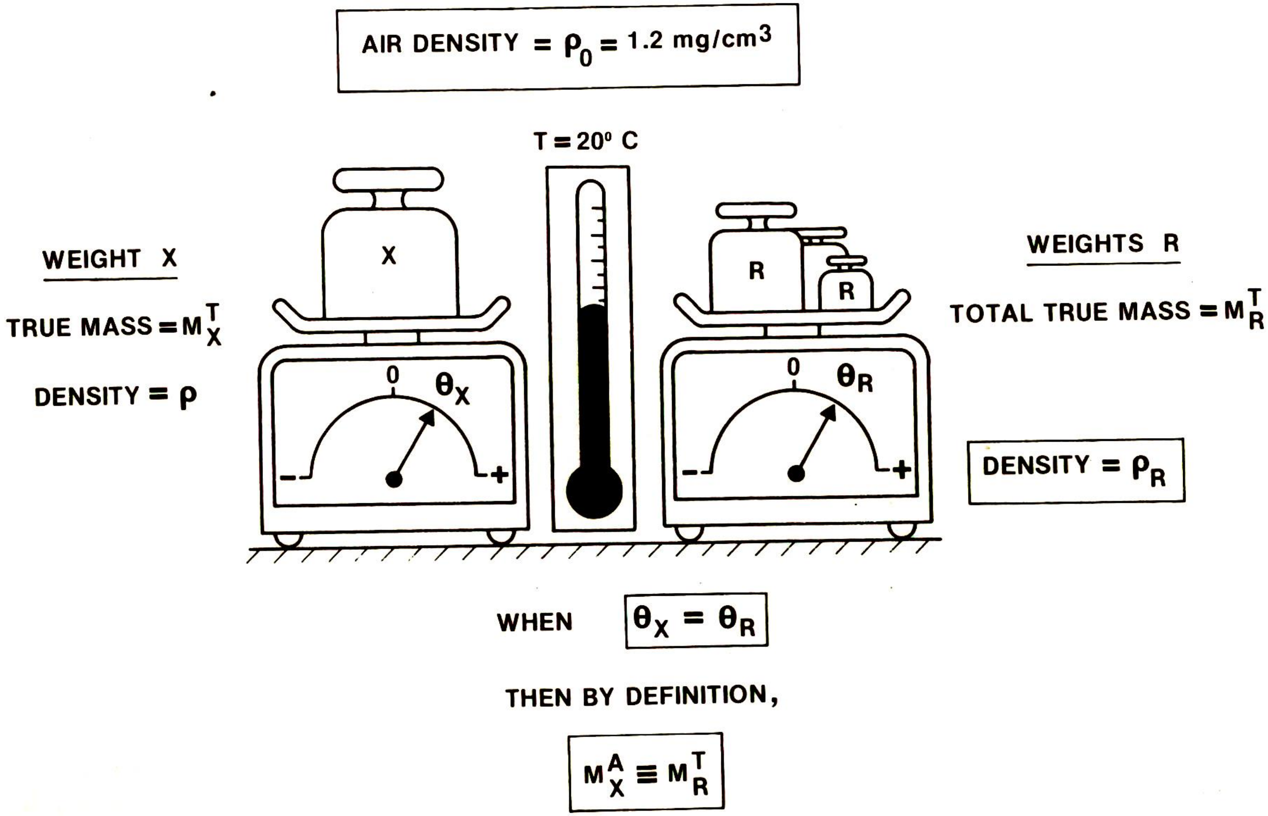
The conventional mass has the same unit as mass, because its values are defined through the multiplication of a mass by a dimensionless quantity.

Conventional mass was formerly called “apparent mass versus 8.0 g·cm^{-3}” in USA

Conventional value of the result of weighing in air
(Valeur conventionnelle du résultat des pesées dans l'air)
OIML D 28 Edition 2004 (E)
ORGANISATION INTERNATIONALE DE MÉTROLOGIE LÉGALE

Conceptually,

conventional mass of object X is equal to true mass of just enough reference material R to produce equal balance reading to that produced by X if measurements are done at 20 °C and with ρ_0 = 1.2 mg·cm-3



CONVENTIONAL MASS OF X = TRUE MASS OF R

Boxed information is specified in definition

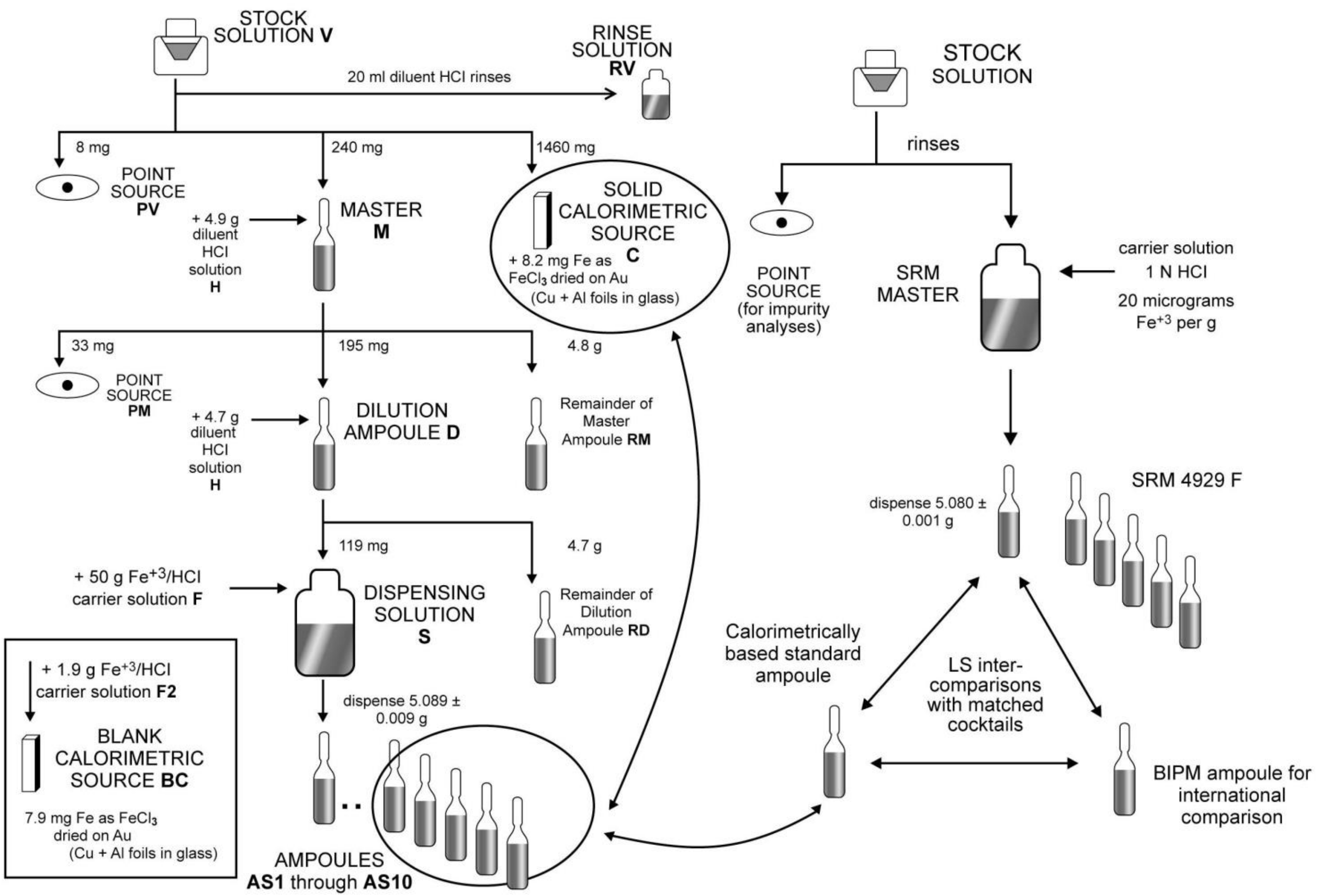
Let's first consider balances needed and used

need mass measurements over wide range of precision & capacity

precise microbalances for source preparations,

analytical balances for carrier solutions,

to large capacity balances for master solutions

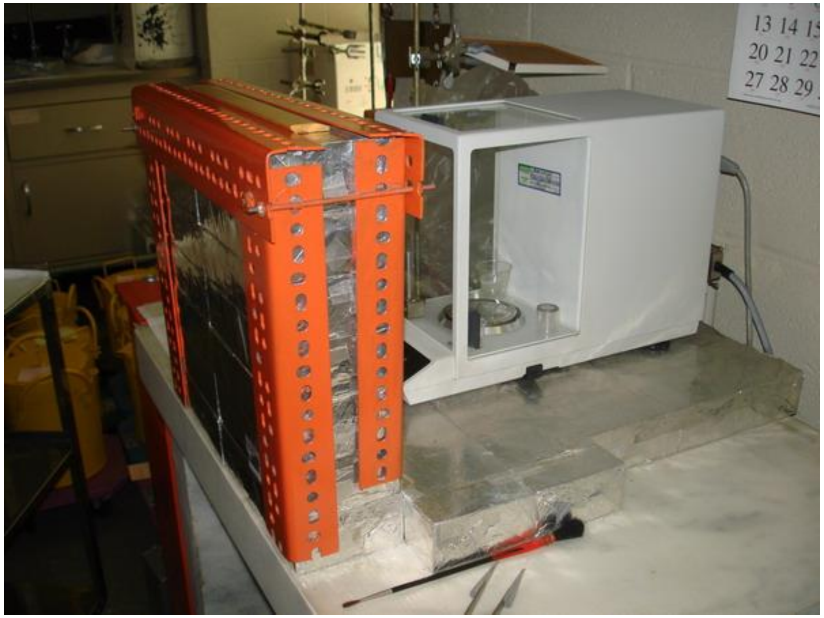


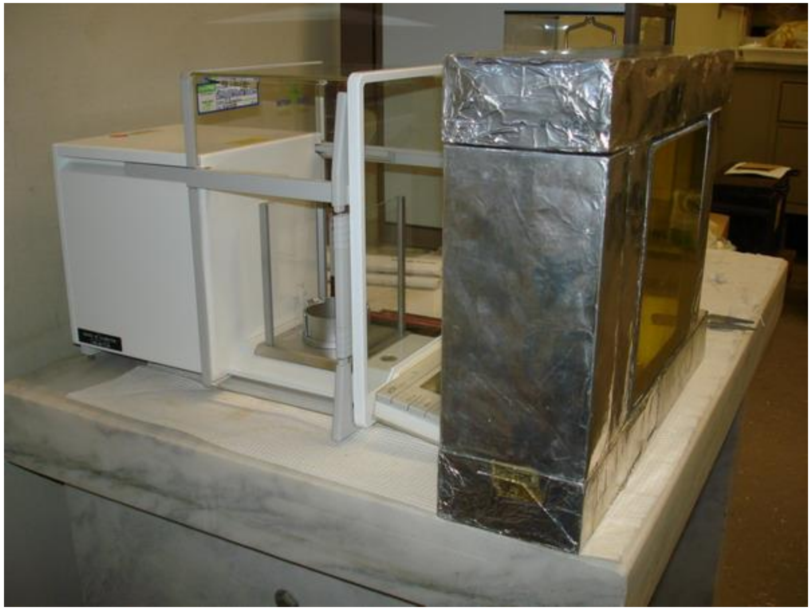
NIST Physics Lab -- Radioactivity Group Balances & Scales

Balance	Type	Room	Number	Load (g)	Readability (mg)
Microbalances					
Mettler M5	Dial Weights & Optical scale	B46 E106	3	20	0.001
Mettler AX26	Electronic	B152	1	22	0.001
Mettler AT20	Electronic	B46	1	22	0.002
Analytical					
Mettler B5	Dial Weights and Optical scale	E106 B152	2	200	0.1
Mettler XP205	Dial Weights and Optical scale	B152	1	220	0.01
Mettler H311	Dial Weights and Optical scale	B46	1	240	0.1
Denver Instr. M220D	Electronic	E103	1	220 31	0.1 0.01
Mettler AE240	electronic	B152 B156 C135	3	240 40	0.1 0.01
Laboratory					
Mettler B6	Dial Weights and Optical scale	C135	1	100	1
Mettler P120	Scale, optical	B46	1	120	10
Mettler H6T	Dial Weights and Optical scale	B48	1	160	1
Large capacity					
Mettler B4C1000	Dial Weights and Optical scale	C135	1	1 000	10
Voland Jupiter 3000	Dial Weights and Optical scale	B152	1	3000	0.01
Mettler PB3002	Electronic	C135	1	3 100	10
Mettler AX12004	weights + electronic (100 g)	B46	1	12 111	0.1
Adams CBC-15	Scale, electronic	B46	1	15 000	100
Mettler ID1 Plus	Scale, electronic	C138	1	150 000	1000

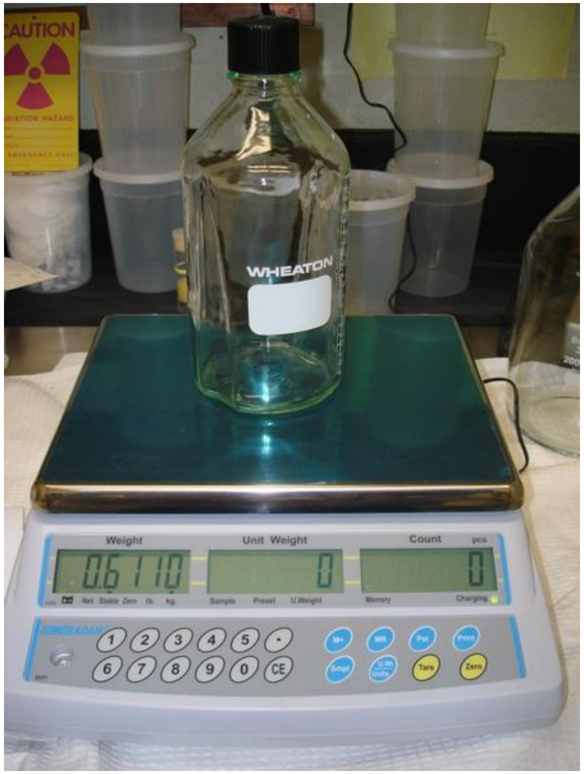
Balances with built-in dial weights & optical scale

"Electronic" (EMF compensation)

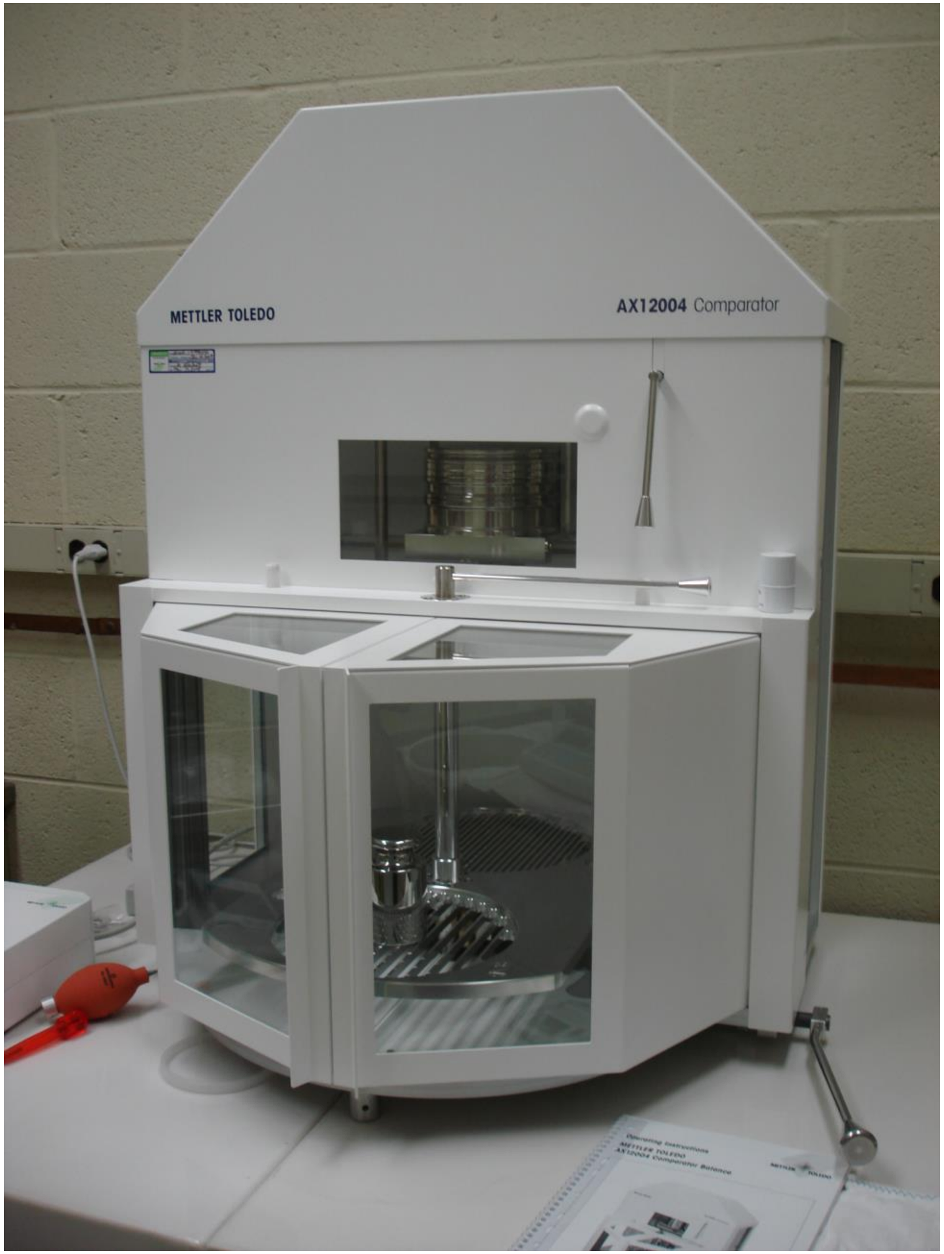





Mettler H311


CAUTION
WHEATON
Weight
Unit Weight
Count
0.6110
0
0

others



Mass
comparator

Now, let's talk about reference weights & density

Want balance to measure conventional mass vs. $\rho_R = 8.0\ g{\cdot}cm^{-3}$

For either single pan balances using built-in dial weights or "electronic" EMF compensation

Manufacturers adjust the dial weights or EM force so that conventional masses equal the value of the dial position or the electronic reading.

No matter what density the balance weights ρ_{BW} actually are, <u>user generally assumes</u> that <u>uncorrected</u> conventional mass is equal to the reading (either dial positions or digital value) and that $\rho_{BW} = \rho_R$

Equivalent force for $\rho_{BW} = \rho_R$ is easy for "electronic" balances

But $\rho_{BW} = \rho_R$ is not the case for many older balances

Extant NIST balances have ρ_{BW} = 7.76, 7.8, 8.0 -- even 8.4 $g{\cdot}cm^{-3}$ in very old

error about 10^{-4} %

Similarly the standard weight set used for calibrations and/or substitution weighing may also differ from $\rho_R = 8.0\ g{\cdot}cm^{-3}$

The OIML weight classes are defined as follows:

Class E1: Weights intended to ensure traceability between national mass standards (with values derived from the International Prototype of the kilogram) and weights of class E2 and lower. Class E1 weights or weight sets **shall be accompanied by a calibration certificate** (see 15.2.2.1).

Class E2: Weights intended for use in the verification or calibration of class F1 weights and for use with weighing instruments of special accuracy class I. Class E2 weights or weight sets **shall be accompanied by a calibration certificate** (see 15.2.2.2). They may be used as class E1 weights if they comply with the requirements for surface roughness, magnetic susceptibility and magnetization for class E1 weights, and if their calibration certificate gives the appropriate data as specified in 15.2.2.1.

Class F1: Weights intended for use in the verification or calibration of class F2 weights and for use with weighing instruments of special accuracy class I and high accuracy class II.

Class F2: Weights intended for use in the verification or calibration of class M1 and possibly class M2 weights. Also intended for use in important commercial transactions (e.g. precious metals and stones) on weighing instruments of high accuracy class II.

Class M1: Weights intended for use in the verification or calibration of class M2 weights, and for use with weighing instruments of medium accuracy class III.

Class M2: Weights intended for use in the verification or calibration of class M3 weights and for use in general commercial transactions and with weighing instruments of medium accuracy class III.

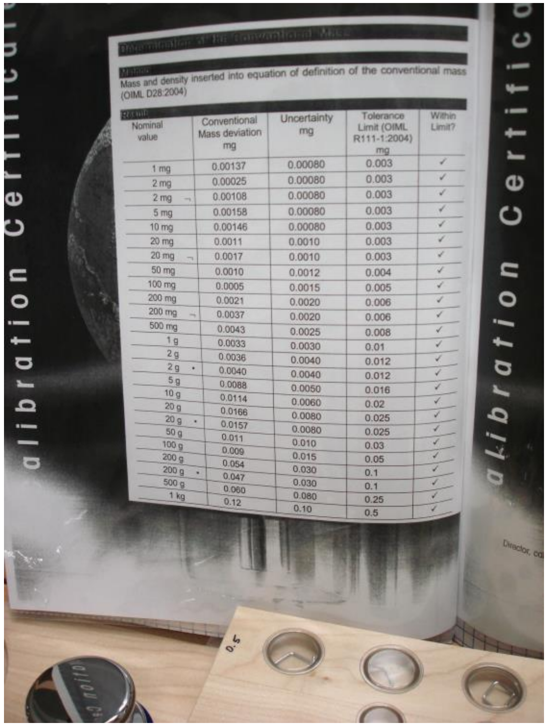
Class M3: Weights intended for use with weighing instruments of medium accuracy class III and ordinary accuracy class IIII.

Classes M1–2 and M2–3: Weights from 50 kg to 5 000 kg of lower accuracy intended for use with weighing instruments of medium accuracy class III.

Note: The error in a weight used for the verification of a weighing instrument shall not exceed 1/3 of the maximum permissible error for an instrument. These values are listed in section 3.7.1 of OIML R 76 *Nonautomatic Weighing Instruments* (1992).

1 mg to 5 kg standard weight set is a thing of beauty to behold

Get pretty certificate too



Mass and density inserted into equation of definition of the conventional mass (OIML D28:2004)

Nominal value	Conventional Mass deviation mg	Uncertainty mg	Tolerance Limit (OIML R111-1:2004) mg	Within Limit?
1 mg	0.00137	0.00080	0.003	✓
2 mg	0.00025	0.00080	0.003	✓
2 mg ¬	0.00108	0.00080	0.003	✓
5 mg	0.00158	0.00080	0.003	✓
10 mg	0.00146	0.00080	0.003	✓
20 mg	0.0011	0.0010	0.003	✓
20 mg ¬	0.0017	0.0010	0.003	✓
50 mg	0.0010	0.0012	0.004	✓
100 mg	0.0005	0.0015	0.005	✓
200 mg	0.0021	0.0020	0.006	✓
200 mg ¬	0.0037	0.0020	0.006	✓
500 mg	0.0043	0.0025	0.008	✓
1 g	0.0033	0.0030	0.01	✓
2 g	0.0036	0.0040	0.012	✓
2 g •	0.0040	0.0040	0.012	✓
5 g	0.0088	0.0050	0.016	✓
10 g	0.0114	0.0060	0.02	✓
20 g	0.0166	0.0080	0.025	✓
20 g •	0.0157	0.0080	0.025	✓
50 g	0.011	0.010	0.03	✓
100 g	0.009	0.015	0.05	✓
200 g	0.054	0.030	0.1	✓
200 g •	0.047	0.030	0.1	✓
500 g	0.060	0.080	0.25	✓
1 kg	0.12	0.10	0.5	✓

CONVENTIONAL MASS
(Apparent mass versus 8.00 g/cm3)

m/g		r/mg	u/mg	d/(kg/m3)	mass (g)	% unc
0.001		0.00137	0.0008		**0.00100137**	**0.080**
0.002		0.00025	0.0008		**0.00200025**	**0.040**
0.002	*	0.00108	0.0008		**0.00200108**	**0.040**
0.005		0.00158	0.0008		**0.00500158**	**0.016**
0.01		0.00146	0.0008		**0.01000146**	**0.0080**
0.02		0.0011	0.001		**0.02000110**	**0.0050**
0.02	*	0.0017	0.001		**0.02000170**	**0.0050**
0.05		0.001	0.0012		**0.05000100**	**0.0024**
0.1		0.0005	0.0015		**0.10000050**	**0.0015**
0.2		0.0021	0.002		**0.20000210**	**0.0010**
0.2	*	0.0037	0.002		**0.20000370**	**0.0010**
0.5		0.0043	0.0025		**0.50000430**	**0.00050**
1		0.0033	0.003	7998	**1.00000330**	**0.00030**
2		0.0036	0.004	8008	**2.00000360**	**0.00020**
2	*	0.004	0.004	8008	**2.00000400**	**0.00020**
5		0.0088	0.005	8007	**5.00000880**	**0.00010**
10		0.0114	0.006	8007.2	**10.00001140**	**0.000060**
20		0.0166	0.008	8012.7	**20.00001660**	**0.000040**
20	*	0.0157	0.008	8012.9	**20.00001570**	**0.000040**
50		0.011	0.01	8010.91	**50.00001100**	**0.000020**
100		0.009	0.015	8026.94	**100.00000900**	**0.000015**
200		0.054	0.03	8009.8	**200.00005400**	**0.000015**
200	*	0.047	0.03	8010.82	**200.00004700**	**0.000015**
500		0.06	0.08	8013.623	**500.00006000**	**0.000016**
1000		0.12	0.1	8012.199	**1000.00012000**	**0.000010**
2000		0.29	0.1	8010.25	**2000.00029000**	**0.000005**
2000	*	0.08	0.1	8011.4	**2000.00008000**	**0.000005**
5000		0	0.1	8010.9	**5000.00000000**	**0.000002**

NIST Radioactivity Group

Class E1 weight set

Calibrated June 2008

Let's next look at the air bouyancy correction

$$C = \left[\frac{1 - \dfrac{\rho_a}{\rho_R}}{1 - \dfrac{\rho_a}{\rho_S}} \right]$$

$$C \approx 1 + \rho_a \left[\frac{1}{\rho_S} - \frac{1}{\rho_R} \right]$$

Large 0.1 % effect for typical ambient ρ_a and ρ_x

Everyone knows about it and does it !

Formulas for air density and correction readily available (CIPM recommended)

Air density depends on temperature, pressure, humidity (not too sensitive)

Correction depends on air density, density of object being weighed, balance weight (or reference) density

OIML R111 calls for ρ_a range 1.2 mg·cm^{-3} ± 10 %

So typical condition requirements are:

T = 20 °C ± 5 °C

P = 101 kPa ± 6 kPa (760 mm Hg mbar

RH = 50 % ± 30 %

Typical T-controlled lab has ρ_a range of 1.2 mg·cm^{-3} ± 5 %

Typical routine lab <u>measurement capability</u> is

$$\left.\begin{array}{l}\text{T to 0.1 °C}\\ \text{P to 0.05 kPa (0.4 mm Hg)}\\ \text{RH to 5 \% (at dewpoint of 0.25 °C)}\end{array}\right\}\ \underline{\text{NOT}}\text{ stability}$$

air T, P, RH measurements

$\Downarrow$

Air density equation

Assumed reference density

corrected density of solution

$\rho_a \Downarrow$ $\rho R \Downarrow$ $\rho x \Downarrow$

$$\boxed{\text{BUOYANCY CORRECTION}}$$

Uncertainty components on correction:

ρ_a (T, P, RH meas. unc. including variability in T, P, RH)

ρ_R (may depended on ρ_{BW} or $\rho_{W\,R}$ differences)

ρ_x (density measurement, with unc. in mass volume, temp., etc.

PROPAGATE UNC. BY $$u^2(\rho_a) = \sum \left(\frac{\partial \rho_a}{\partial v_i}\right)^2 u^2(v_i)$$

& $$u^2(C) = \sum \left(\frac{\partial C}{\partial \rho_i}\right)^2 u^2(\rho_i)$$

$\boxed{\text{REAL DATA}}$

$T = (23.0 \pm 0.4)^\circ C$ (variability)

$P = 29.8 \pm 1.7$ in Hg (storm)

(101 ± 6) kPa

$RH = 45\% \pm 5\%$

$\rightarrow \rho_a = 1.1815$ mg/cm^3 $\pm 5.6\%$

$\rho_{BW} = 7.76$

$\rho_S = (1.015 \pm 0.002)\ g \cdot cm^{-3}$

$C = 1.001030$
± 0.000057 (or 0.006%)

effect of unc. on $\rho_S < 0.0002\%$

" ρ_R negligible

Obviously, buoyancy correction is important,

but once made - uncertainty due it is fairly small

Weighing uncertainty must consider other things

Like

the weighing PROCEDURE used

And

the procedure’s calibration

or basis of the weight standards

So, we now need to specifically address different weighing procedures

& how they affect uncertainty

Lots of weighing schemes for single-pan balance

Many available references to them

Direct reading
- Built-in dial weights & optical scale
- “electronic” EMF compensation

relies on balance calibration

Single substitution
- Without sensitivity weight
- With sensitivity weight

relies on weight standards

Double substitution

Higher order stuff

relies on weight standards – more fully accounts for drift

Adequacy of balance calibration or calibration of weight set usually not the biggest uncertainty concern

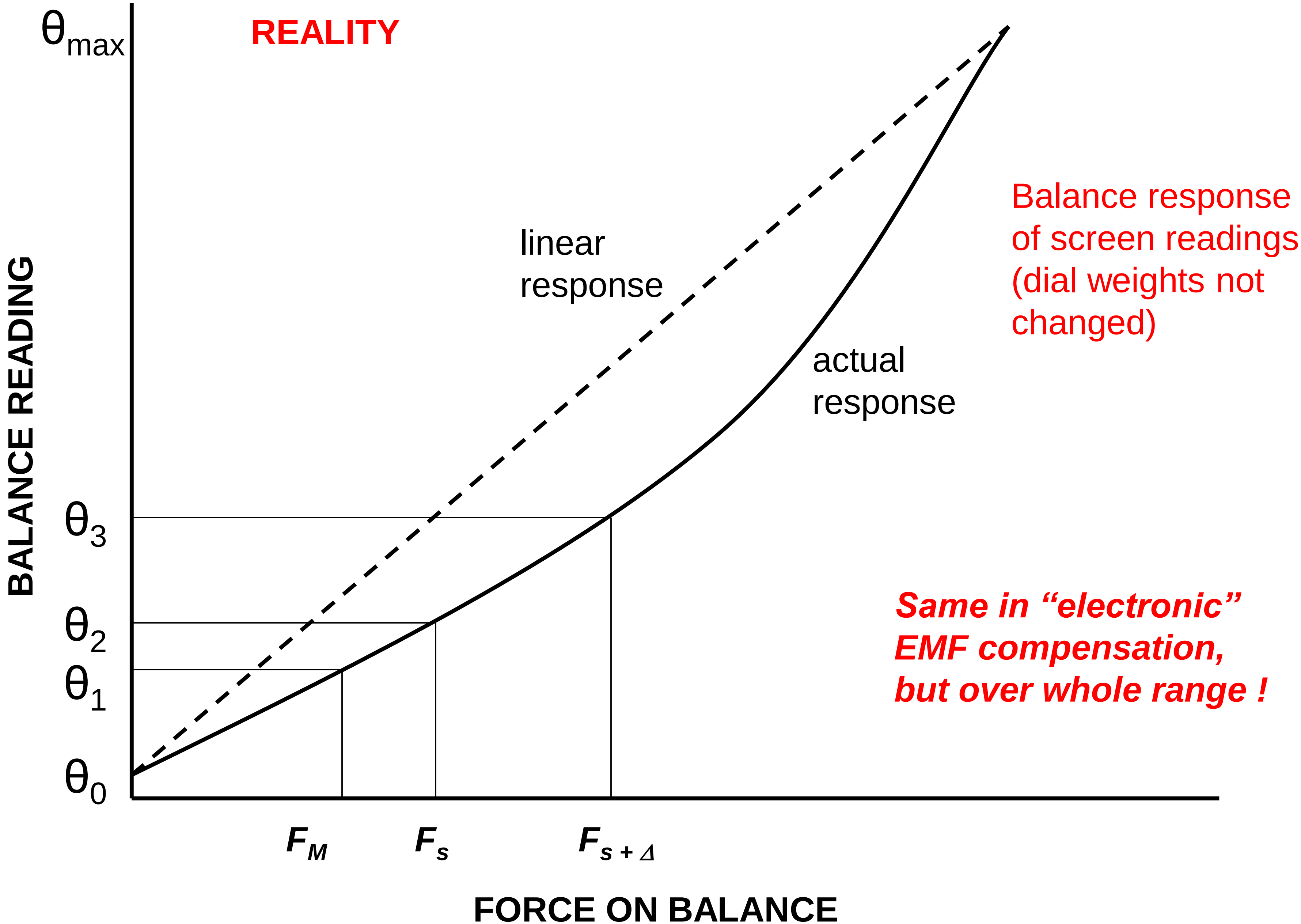
BUT, assumes that balances & standards handled appropriately

Built-in balance weights in high quality balances were ORIGINALLY as good as E2 weights. Probably still ok, if not F....ED with.

Bigger concern might be for small masses, linearity of optical scale

Important to realize that “calibrations” as part of routine balance maintenance (yearly) only do two point checks.

Linearity has same concerns in “electronic” balances – internal calibrations rely on reference voltage in precision resistor



θmax
REALITY
BALANCE READING
linear
response
actual
response
Balance response
of screen readings
(dial weights not
changed)
θ3
θ2
θ1
θ0
Same in "electronic"
EMF compensation,
but over whole range !
FM
Fs
Fs + Δ
FORCE ON BALANCE

All deviations from linearity can depend on temperature.

All weight sets + balances (“electronic”) have a temperature coefficient (TC) – indicates “sensitivity” change in mass reading per °C

Example Weigh 5 g on analytical balance with resolution of 0.1 mg. Tech specs for balance has TC $\leq$ 5(10-6) g/°C. With 2°C change (from calib temp. or lab variability), get change of 0.05 mg (or half resolution from TC alone)

Temperature also affects “drift” of ideal response curve – in both optical scale & EMF compensation

Consider conversion steps in typical EMF compensation balance

mass

↓ *misalignment, vibrations, electrostatics, air currents,...*

weight (force) on pan

↓ *forces on lever, alignment ...*

force on coil

↓ *aging, external magnetic fields, temp. coefficient, position of coil ...*

electrical current

↓ *change in precision resistor because of temp or aging, thermoelectric voltages ...*

voltage

↓ *change in reference voltage in ADC (aging & temp), offset voltage amplification changes ...*

digital readout

RETURN

Lots of weighing schemes for single-pan balance

Many available references to them

Direct reading	useful for routine work Preparation of many sources Can have dial weight calibrations
Substitution weighing	better for high accuracy source preparation takes time – certainly better for weighing metal standards – is it, for an evaporating liquid ?

Re-visit “electronic” EMF compensation balances

SOME BENEFITS

- tare control over whole range
- dual capacity *& precision
- filters noise
- computer compatible
- selectable sampling of reading (on some)

SOME PROBLEMS

- static effects (plastic pycnometers)
- weigh ferromagnetic materials
- EM radiation fields
- dust in magnetic poles

Biggest concerns

- Linearity errors
- Force to mass calibration
- Precision (probably dominant)

To identify uncertainty components in weighing need to look at what affects the determination

TWO BIG COMPONENTS

- the basis of the mass standardization
  - balance or weight set calibration
    (condition & handling)
  - linearity of screen or EMF reading
    (optical scale or electronic)

probably smaller
easier to handle

Some stop here !

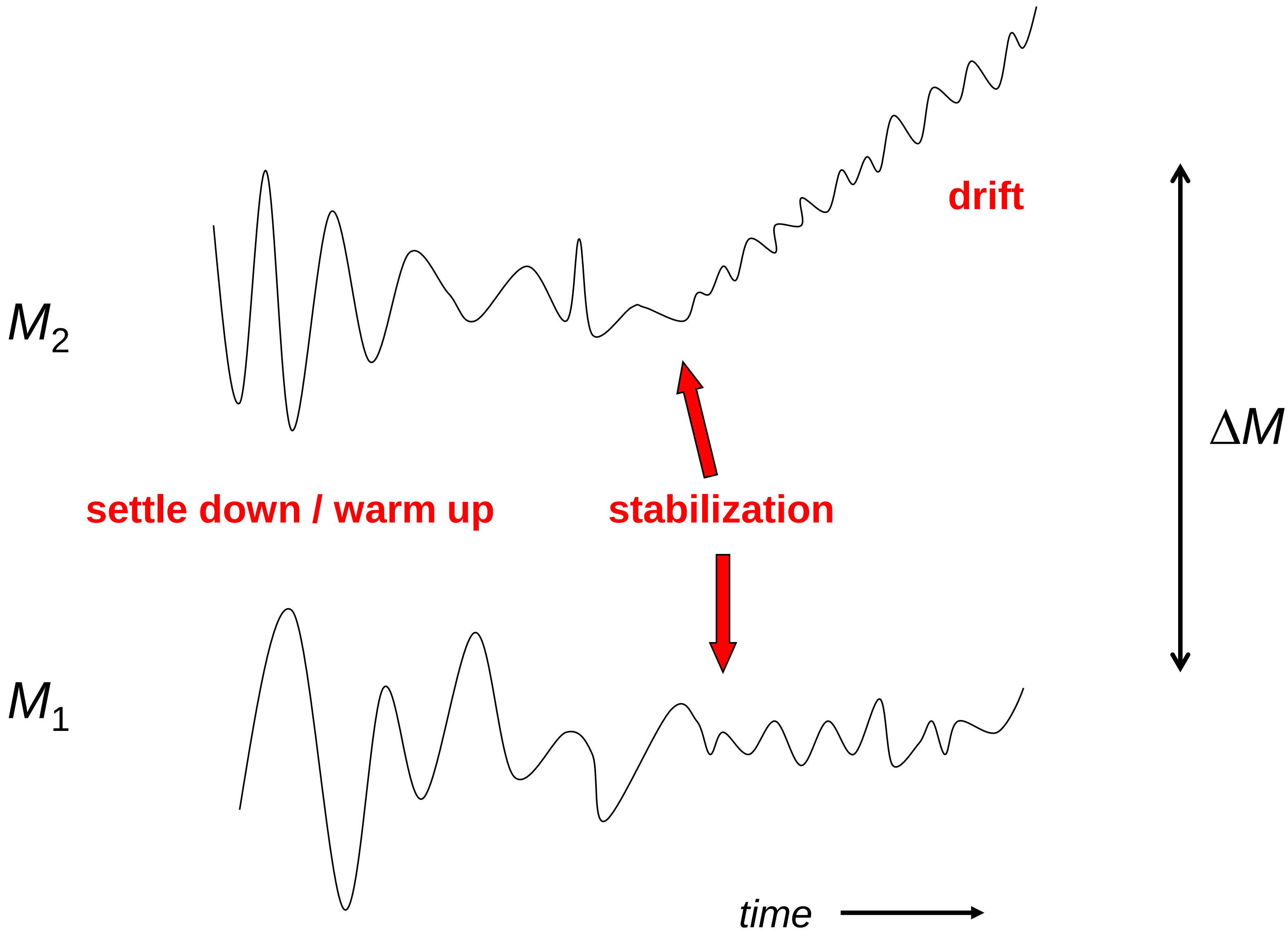
After making buoyancy correction and think the job is done

- environmental / operator effects
  - ambient lab controls (mainly temp.)
  - electrostatics
  - vibrations
  - air currents
  - evaporation losses (really tough)
  - practice / habits of operator
  - Many, many more ...

usually dominant

must evaluate for given conditions & operator

Some tests obvious (dispensed & contained mass differences; replications; etc.), but evaluation is hard

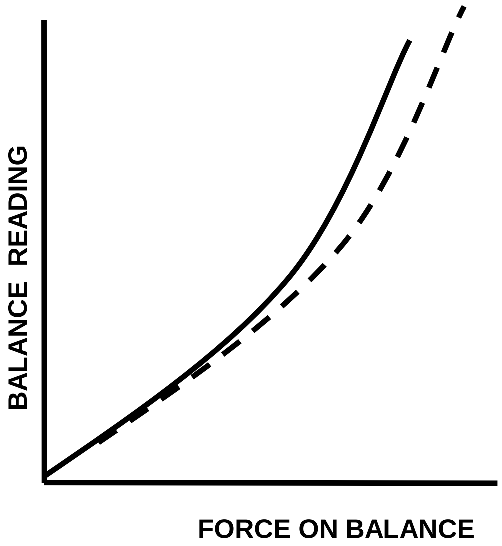


drift
M_2
ΔM
settle down / warm up
stabilization
M_1
time

Mass standards / balance calibration

Operator & practice

Weighing method



Balance / sample sources of inaccuracy

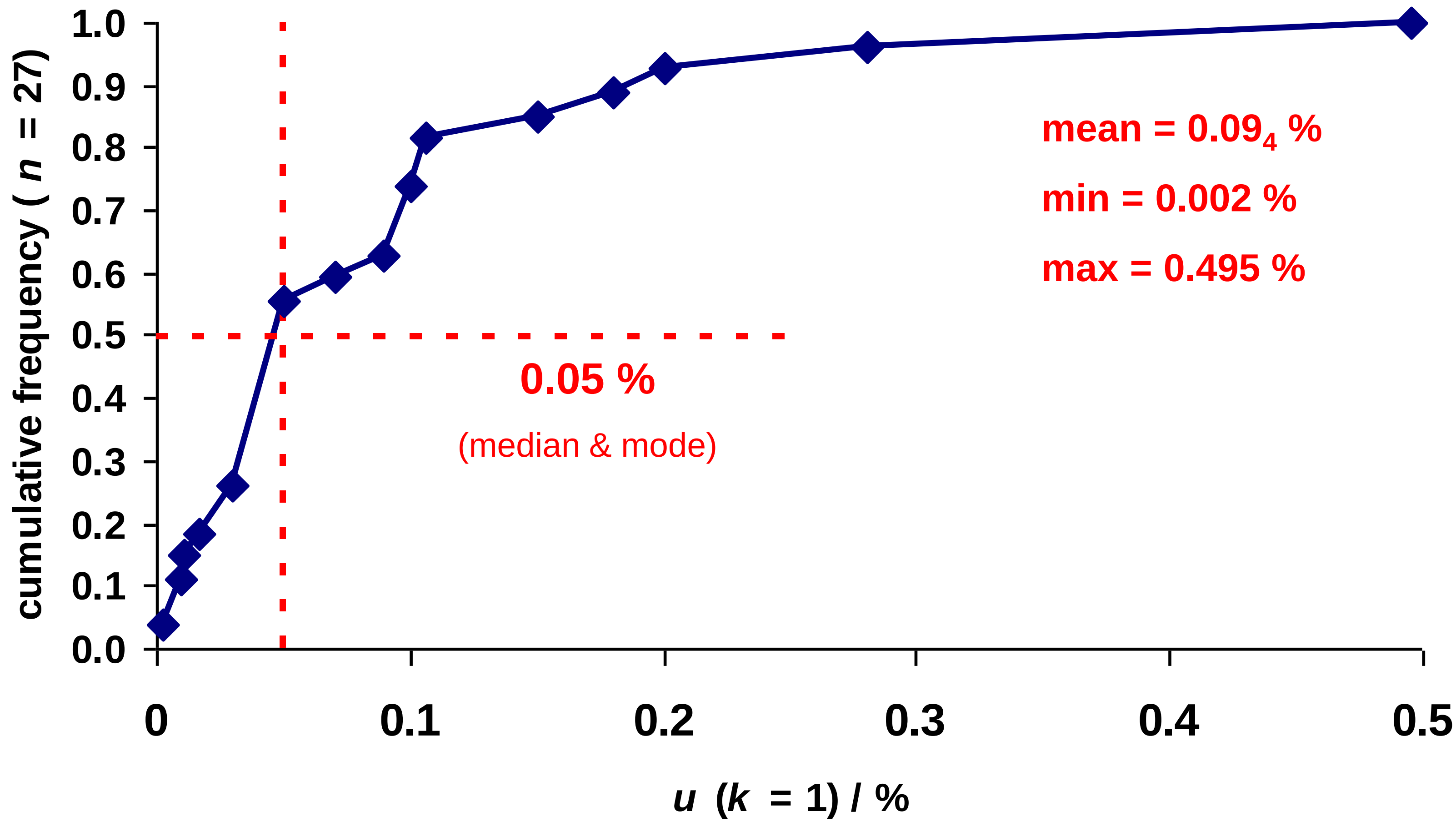
- sensitivity (slope) – slope ≠ 1
- linearity – sensitivity not constant
- hysteresis - different up & down
- drift – instability
- temperature effects
- buoyancy effects
- off-load (eccentricity) effects
- evaporation / condensation

Main environmental problems

- Floor & Bench / vibrational instability
- Temperature
- Humidity
- Light
- Air

Many evaluation tests available
– but lots of work to do

WEIGHING UNCERTAINTIES REPORTED FOR ^{55}Fe COMPARISON



What is a realistic weighing uncertainty ?

How many weighings enter into final value & how combine ?

Is 0.05 % realistic ?

- For a single 20 mg source, this is 10 μg.

  Not likely for direct reading measurement – many reasons

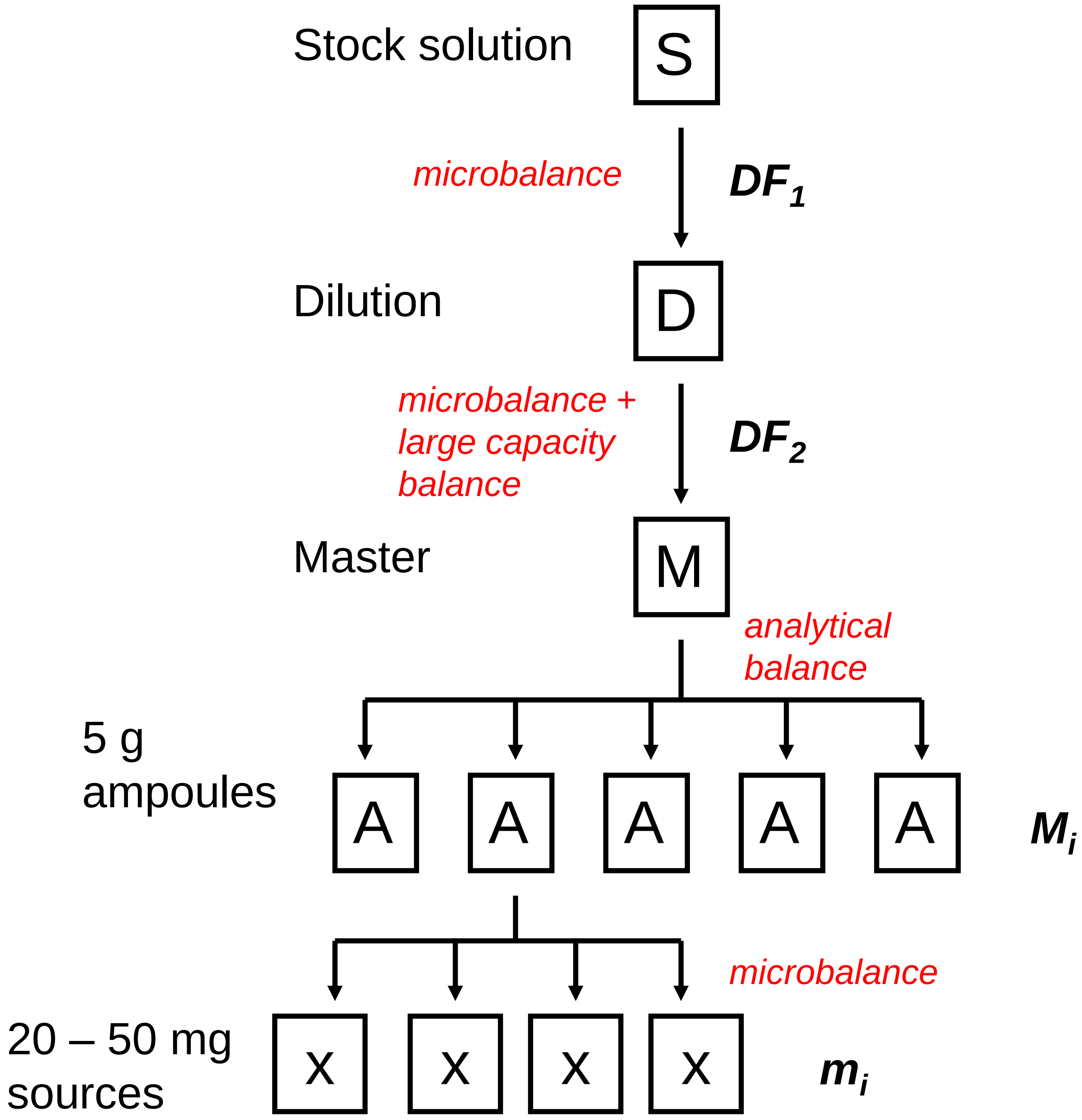
  For substitution weighing, the E1 mass standard tolerance limit is 3 μg alone ! Maybe for a very good operator with excellent ambient controls. But probably not, unless using multiple sources to get precision.

- For a single 5 g mass, this is 2.5 mg.

  Probably, but must have good analytical balance or better, good ambient controls, and reasonably good operator. May depend on what measuring.

- For a single 2 kg mass, this is 1 g.

  Definitely, assuming have reasonable calibrated large capacity balance or weight set, and adequate ambient conditions and operator. Again, may depend on what measuring.



What is weighing uncertainty ?

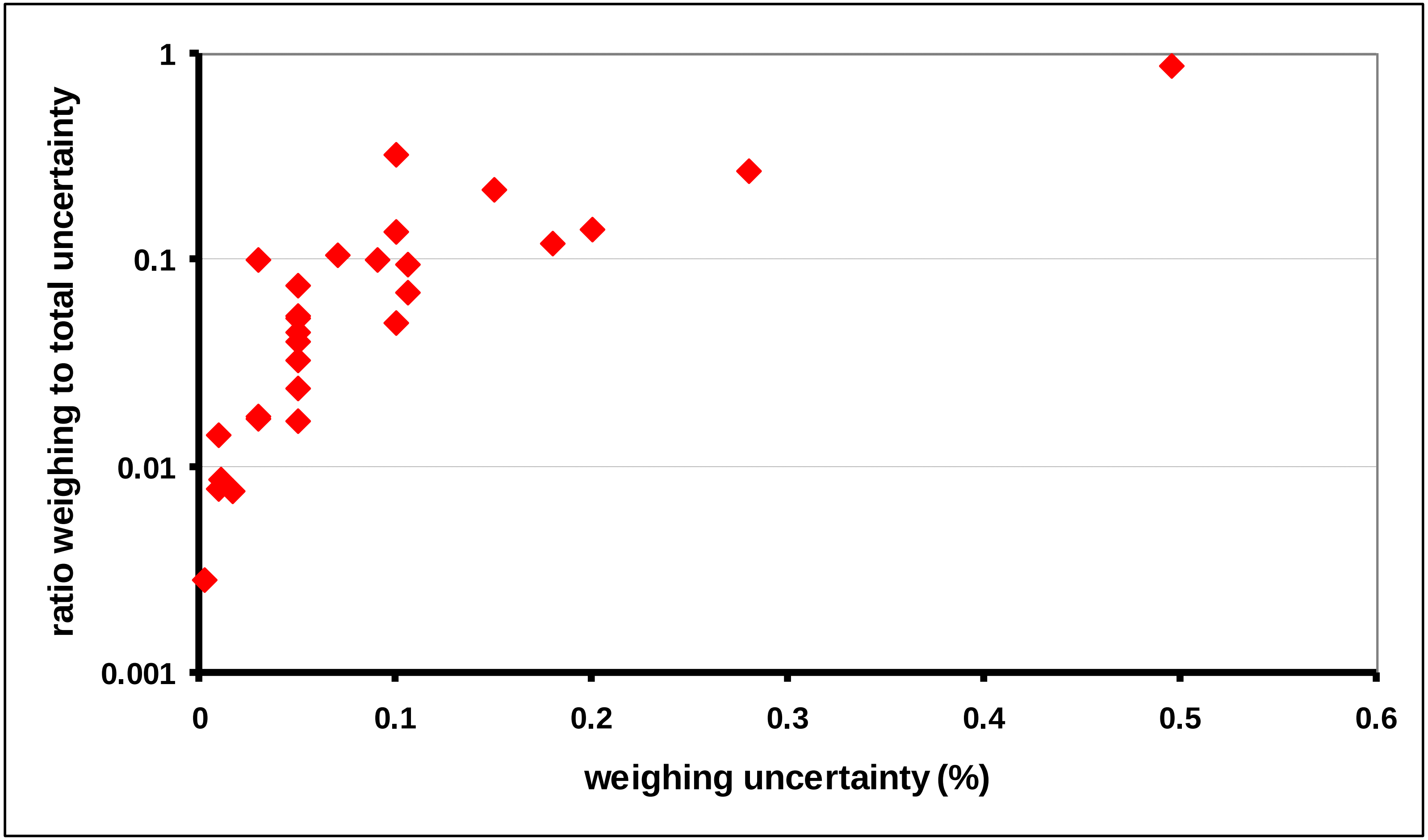
Report one value in uncertainty budget ?

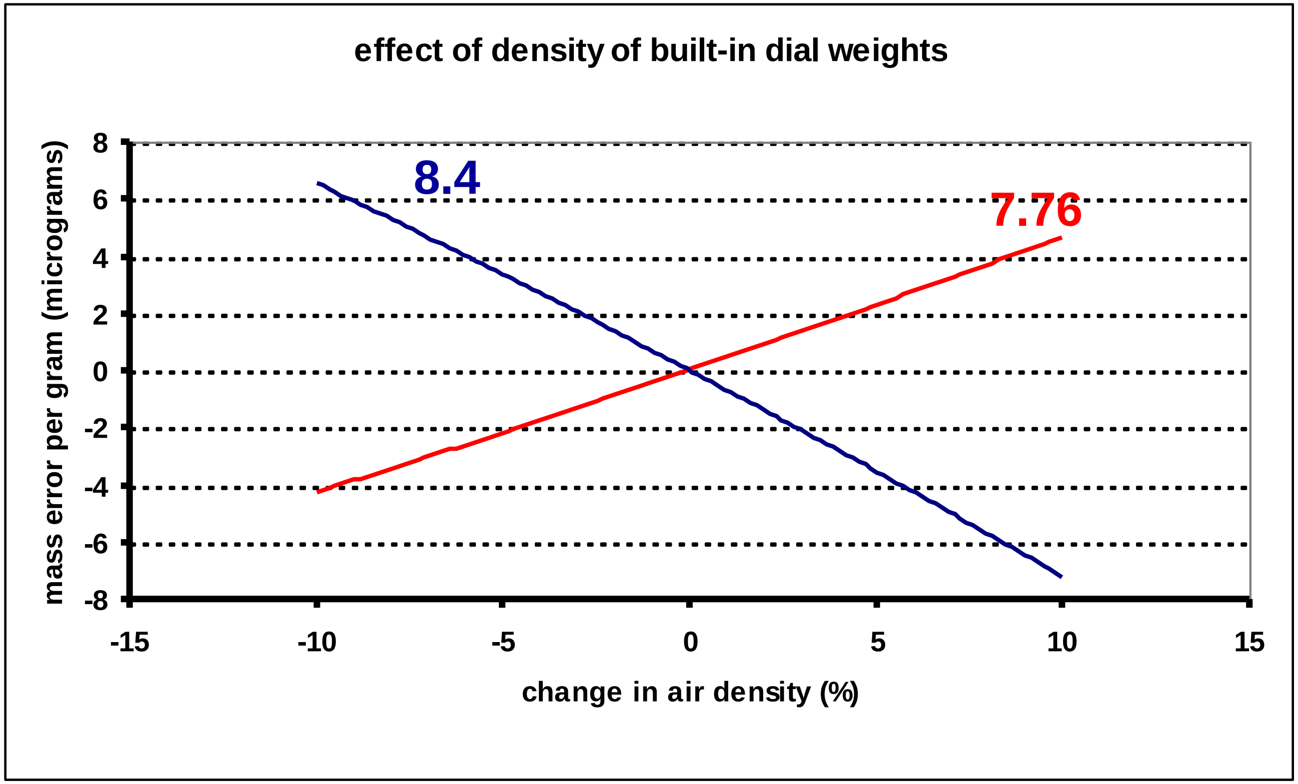
How combine ?

How handle evident correlations ?

Some support slides follow, if needed

uncertainties reported for 55Fe comparison





$\rho_0 = 0.00120\ \mathrm{g \cdot cm^{-3}}$

$\rho_R = 8.0\ \mathrm{g \cdot cm^{-3}}$

For

$\rho_{BW} = 7.76\ \mathrm{g \cdot cm^{-3}}$

and $8.4\ \mathrm{g \cdot cm^{-3}}$

$\rho_a = 0.0012\ \mathrm{g \cdot cm^{-3}} \pm 10\ \%$

$$\Delta M = W(\rho_0 - \rho_a)[1/\rho_R - 1/\rho_{BW}]$$

1 g