OUTLINE

- About MML
- Calibration
  - Standard Weight
  - Balance
- Verification
  - Balance

MASS METROLOGY LABORATORY (MML)

Institute of Advanced Technology (ITMA UPM)

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MML

- Ditubuhkan pada tahun 2011 dengan bajet peruntukan diperolehi daripada TNCPI
- Di awal penubuhan, MML diwujudkan untuk memberikan perkhidmatan tentukur ‘balance’ dan ‘standard weight’ di Fakulti Kejuruteraan.
- Kemudian diluaskan operasi perkhidmatan kepada seluruh PTJ dalam UPM dan luar UPM
- Mendapat akreditasi ISO/IEC 17025 bermula 15 Julai 2017
- Secara rasminya MML dipindah tanggungjawab dan operasi ke ITMA pada 1 Julai 2020 bagi mengoptimumkan perkhidmatan tentukur

Calibration vs Verification

Why it is needed?

Measuring Devices / Equipment

Measurement : Accuracy and Precision

ACCURACY

- How close a measurement or attempt is to the actual or target value

ACCURATE!

ACCURATE!

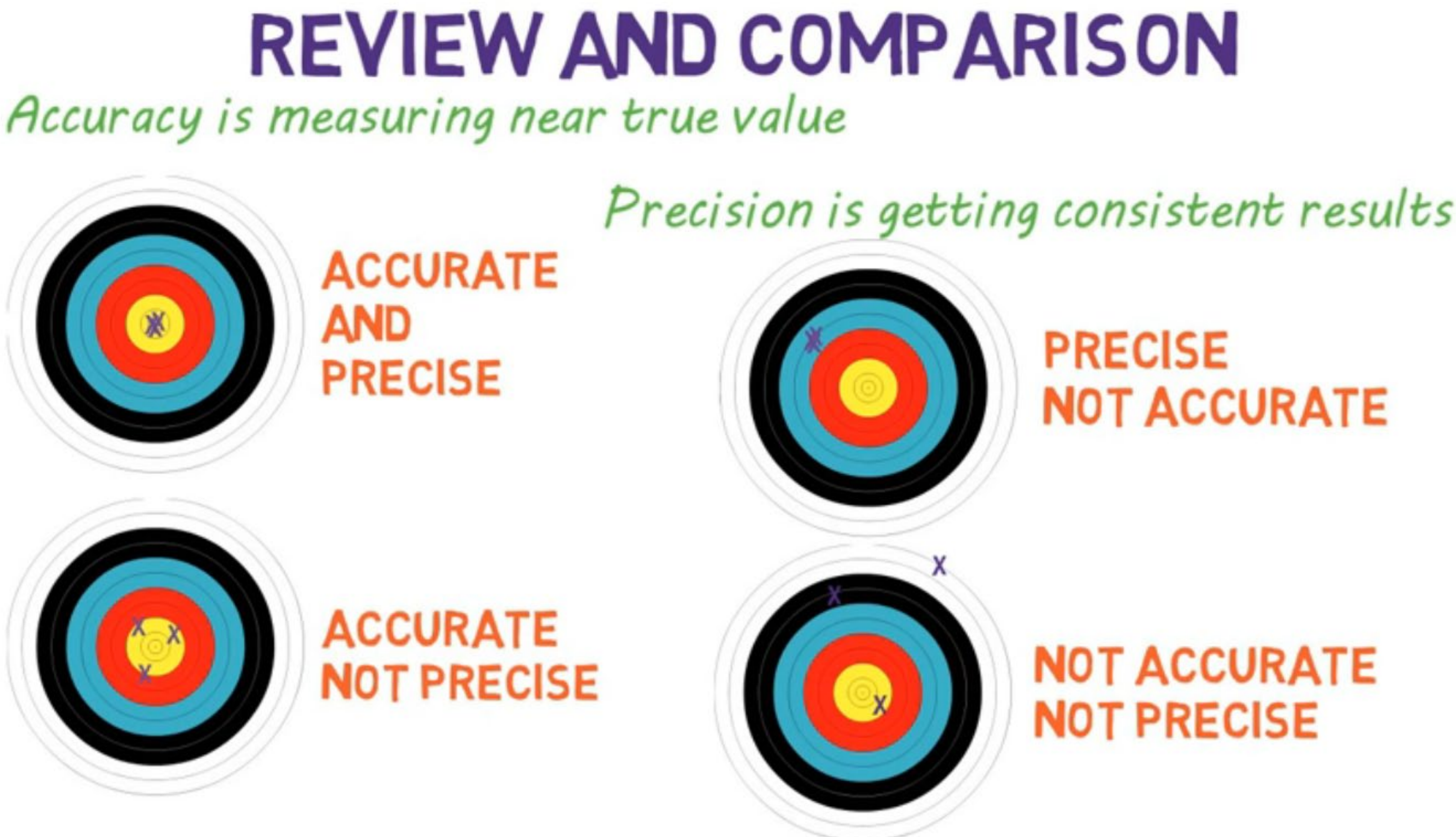
PRECISION

- How consistent our results are reagardless of proximitey to actual or target

PRECISE!

PRECISE!

Calibration vs Verification



It is important that measuring devices are accurate and precise

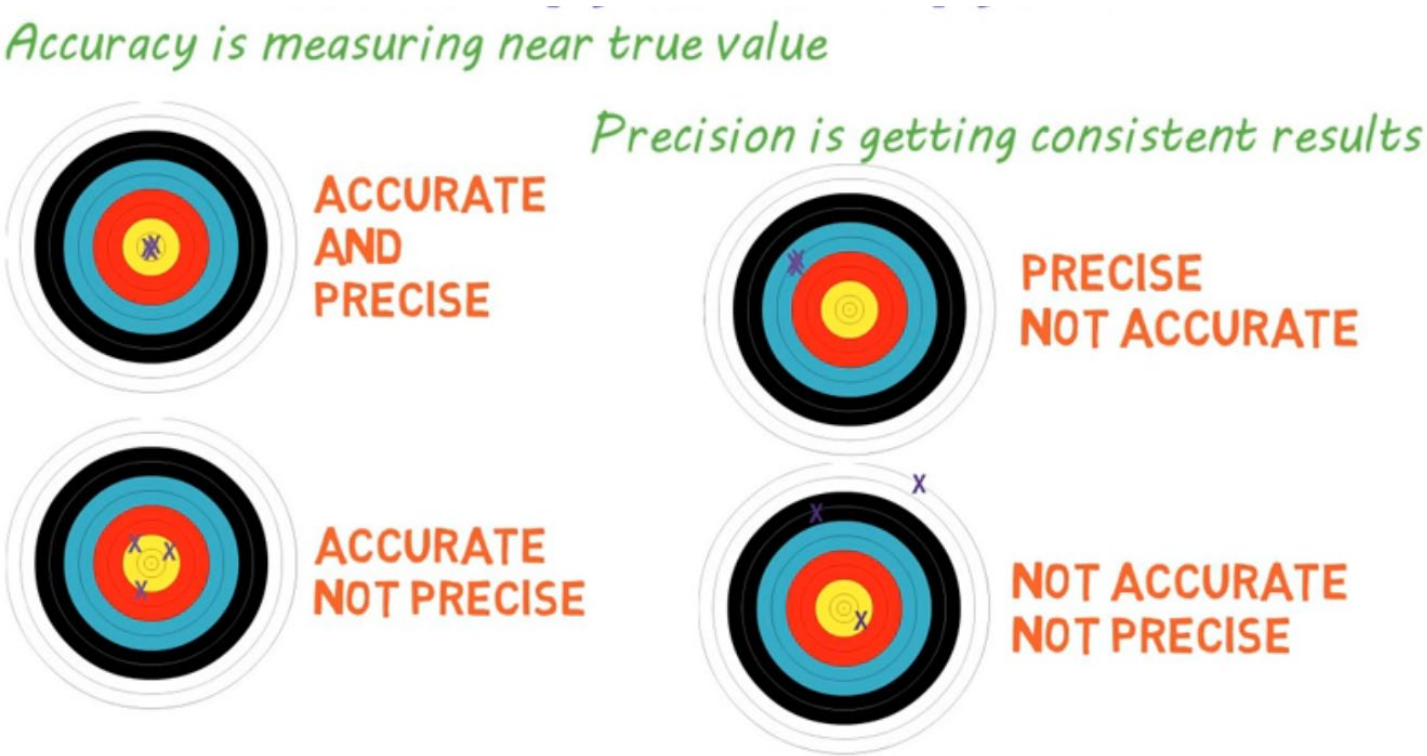
If not, results can be misleading or even **FATAL**

Calibration vs Verification

A **calibration** indicates the error **of** the instrument and compensates **for** any lack **of** trueness by applying a correction.

A **verification** indicates that the measurement error is smaller than a so called maximum permissible error.

Calibration
vs
Verification
vs
Service/Repair



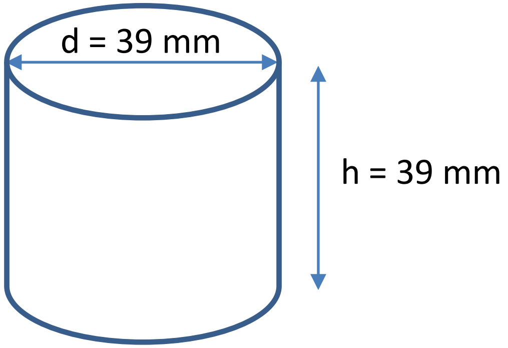
Calibration of Standard Weight

OIML R111

Standard Weight

SI Base Unit for Mass - Kilogram

- The kilogram is the unit mass and is defined as the mass of the international prototype of the kilogram.
- It is a cylinder of height 39 mm and diameter 39 mm made of platinum – iridium alloy comprising 90% platinum and 10% iridium with density of approximately 21,500 kgm-3.
- The international prototype is kept with its six official copies in a vault at BIPM (The International Bureau of Weights and Measures) – France
- Forty copies of the kilogram were commissioned and distributed to the major national standard laboratories to be their primary standard



International prototype of the kilogram

Standard Weight

Klasifikasi

- OIML — Organisation Internationale de Metrologie Legale (International Organisation of Legal Metrology)

  Ref No.: OIML R111

  Classes: E1, E2, F1, F2, M1, M1-2, M2, M2-3, M3

  Range: 1 mg – 5000 kg

- ANSI/ASTM — American Standards

  Ref No.: E617

  Classes: 1, 2, 3, 4, 5, 6, 7

  Range: 1 mg – 5000 kg

Standard Weight

Maximum Permissible Errors

Value of the **measurement error**, with respect to a known **reference quantity value**, permitted by specification or regulations for a given **measurement**, **measuring instrument**, or **measuring system**.

Maximum permissible errors for weights – OIML R 111-1:2004 (page 12)

Table 1 Maximum permissible errors for weights (± δm in mg)

Nominal value*	Class E_1	Class E_2	Class F_1	Class F_2	Class M_1	Class M_{1-2}	Class M_2	Class M_{2-3}	Class M_3
5 000 kg			25 000	80 000	250 000	500 000	800 000	1 600 000	2 500 000
2 000 kg			10 000	30 000	100 000	200 000	300 000	600 000	1 000 000
1 000 kg		1 600	5 000	16 000	50 000	100 000	160 000	300 000	500 000
500 kg		800	2 500	8 000	25 000	50 000	80 000	160 000	250 000
200 kg		300	1 000	3 000	10 000	20 000	30 000	60 000	100 000
100 kg		160	500	1 600	5 000	10 000	16 000	30 000	50 000
50 kg	25	80	250	800	2 500	5 000	8 000	16 000	25 000
20 kg	10	30	100	300	1 000		3 000		10 000
10 kg	5.0	16	50	160	500		1 600		5 000
5 kg	2.5	8.0	25	80	250		800		2 500
2 kg	1.0	3.0	10	30	100		300		1 000
1 kg	0.5	1.6	5.0	16	50		160		500
500 g	0.25	0.8	2.5	8.0	25		80		250
200 g	0.10	0.3	1.0	3.0	10		30		100
100 g	0.05	0.16	0.5	1.6	5.0		16		50
50 g	0.03	0.10	0.3	1.0	3.0		10		30
20 g	0.025	0.08	0.25	0.8	2.5		8.0		25
10 g	0.020	0.06	0.20	0.6	2.0		6.0		20

Standard Weight

Technical Requirements

- Marking
- Density
- Construction
- Shape
- Material
- Magnetism
- Surface condition

Class M: normally made from cast iron

Standard Weight with Adjusting Cavity
(Adjusting material: lead dust/powder)

Standard Weight

10 Tips of Handling Standard Weight

How to handling your standard weight

https://www.youtube.com/watch?v=eUx--gh1dRI

Standard Weight

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10 Tips of Handling Standard Weight

1. Store weight in the original packing
2. Store weights near the balance
3. Handle weight with care
4. Clean according to OIML/ASTM recommendation
   - OIML Class E1 – F2 weights, use clean soft brush and small bulb type puffer blower
   - OIML Class M weights, should remove any loose material
5. Use specialized handles for bigger weight
6. Inspect for damage prior to use
7. Do not touch weights with bare hands
8. Do not forget periodic calibration of test weights
9. Do not use a weight with dust or water on its surface
10. Do not clean with abrasive or caustic chemicals

Standard Weight

Storage

- Keep in storage cupboard where they can be protected from dust and atmospheric pollution by glass covers
- Weights should be contained in specially-built boxes
- Boxes should have individual compartment for each weights

Calibration of Standard Weight

- Value of any weight – may change with time and use
- Weights may become lighter through wear and tear, and become heavier through chemical depositions or atmospheric pollution
- THUS, it is necessary to calibrate weight prior to use.

- The interval of calibration varies with use and precision.
- In general E1 and E2 should be calibrated at interval not exceeding 2 years and all other weights being calibrated on an annual basis (recommended)

Calibration of Standard Weight

- Calibration condition for standard weight:

(a) Room temperature: $(20 \pm 1)\ ^{O}C$ (Class E2, F1, F2 & M1)
$(23 \pm 1)\ ^{O}C$ (Class M2 and below)
(b) Room relative humidity: (60 ± 5) %RH (Class E2, F1, F2 & M1)
(60 ± 10) % RH (Class M2 and below)
(c) Warm-up time: Minimum 30 minutes

- Reference weight used:

For the calibration of weights of the particular accuracy class, reference standard weights of the next higher class should be used

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- Environment condition

Table C.1 Ambient conditions during calibration (Typical values recommended for obtaining successful results)

Weight Class	Temperature change during calibration
E1	± 0.3 °C per hour with a maximum of ± 0.5 °C per 12 hours
E2	± 0.7 °C per hour with a maximum of ± 1.0 °C per 12 hours
F1	± 1.5 °C per hour with a maximum of ± 2.0 °C per 12 hours
F2	± 2.0 °C per hour with a maximum of ± 3.5 °C per 12 hours
M1	± 3.0 °C per hour with a maximum of ± 5.0 °C per 12 hours

Weight class	Range of relative humidity (hr) of the air
E1	40% to 60% with a maximum of ± 5% per hour
E2	40% to 60% with a maximum of ± 10% per hour
F	40% to 60% with a maximum of ± 15% per hour

Calibration of Standard Weight

- Comparison Method - ABBA vs ABA

A = Reference standard
B = Unknown weight

ABBA	ABA
$M_x = \left(\frac{b_1 + b_2}{2}\right) - \left(\frac{a_1 + a_2}{2}\right) + M_R$	$M_x = b - \left(\frac{a_1 + a_2}{2}\right) + M_R$

M_x – test weight
M_R – nominal value

Calibration of Standard Weight

- Work Example

Nominal value: 50 g

Run	Std Wt a1	Test Wt b1	Test Wt b2	Std Wt a2	Measured Difference
1	50.00023	49.98703	49.98704	50.00024	?
2	50.00024	49.98704	49.98705	50.00026	?
3	50.00026	49.98706	49.98706	50.00028	?
Average Measured Difference					?

$$M_x = \left(\frac{b_1 + b_2}{2}\right) - \left(\frac{a_1 + a_2}{2}\right) + M_R$$

Calibration of Standard Weight

- Measurement Uncertainty (MU)

Source of Uncertainty	Type	Formula
Uncertainty from reference, U_{ref}	B	$U_{ref} = \frac{U_{cert}}{k}$
Uncertainty from drift, U_{drift}	B	$U_{drift} = \frac{MPE}{3\sqrt{3}}$
Uncertainty from equipment resolution, U_{res}	B	$U_{res} = \frac{d}{2\sqrt{3}} * (\sqrt{2})$
Uncertainty from air buoyancy, U_{ab}	B	$U_{ab} = \frac{MPE}{4\sqrt{3}}$
Uncertainty from calibration, U_{rep}	A	$U_{rep} = \frac{\sigma}{\sqrt{n}} (t)$, where t=2.3

Calibration Cert for Standard Weight

NATIONAL METROLOGY INSTITUTE OF MALAYSIA

Issued to : **UNIVERSITI PUTRA MALAYSIA**
INSTITUTE OF ADVANCED TECHNOLOGY (ITMA),
UPM SERDANG, 43400 SERDANG,
SELANGOR, MALAYSIA.

Calibration Date : 09 June 2021

Job No. : NMIM2021-0581

Certificate No. : NMIM-1494-M-21

Name of Instrument : Standard Weight

ID : S00512532

Class : F_1/ Stainless Steel Weight (1 g - 10 kg) - 18 pcs.

Manufacturer : Mettler Toledo

Calibration Procedure : CP-084-0401

Environment Conditions : Temperature : 20.2 °C to 20.4 °C
Humidity : 59 %rh to 61 %rh

Uncertainty : The uncertainties stated in this certificate have been evaluated in accordance with JCGM 100:2008 Evaluation of Measurement Data – Guide to the Expression of Uncertainty in Measurement. The expanded uncertainties are based on estimated confidence probability of approximately 95% and have a coverage factor of *k*=2 unless stated otherwise.

Procedure : Direct comparison against Standard Masses.

Measurement Standard Used :

Description:	Maker/Model No.:	Serial No.:	Traceability:
Standard Mass	Mettler Toledo	449 MCL 0014	NMIM
Standard Mass	Mettler Toledo	449 MCL 0015	NMIM
Standard Mass	Mettler Toledo	449 MCL 0016	NMIM

Results :

Nominal Value	Conventional Mass Value	Uncertainty (±)
10 kg	10 kg + 15 mg	15 mg
5 kg	5 kg + 14 mg	5 mg
2 kg	2 kg + 1 mg	2 mg
2 kg (.)	2 kg 0 mg	2 mg
1 kg	1 kg + 2.2 mg	0.8 mg
1 kg (.)	1 kg + 2.2 mg	0.8 mg
500 g	500 g + 0.1 mg	0.5 mg
200 g	200 g + 0.2 mg	0.2 mg
200 g (.)	200 g + 0.2 mg	0.2 mg
100 g	100 g + 0.06 mg	0.08 mg
50 g	50 g + 0.18 mg	0.06 mg
20 g	20 g + 0.02 mg	0.05 mg
20 g (.)	20 g + 0.02 mg	0.05 mg
10 g	10 g + 0.01 mg	0.04 mg
5 g	5 g + 0.01 mg	0.04 mg
2 g	2 g 0.00 mg	0.03 mg
2 g (.)	2 g 0.00 mg	0.03 mg
1 g	1 g 0.00 mg	0.03 mg

Note:

Conventional mass is the mass of a reference weight of a density of 8 000 kg/m^3 which it balances in air of a reference of density 1.2 kg/m^3 .

END OF RESULTS

Calibration of Analytical Balance

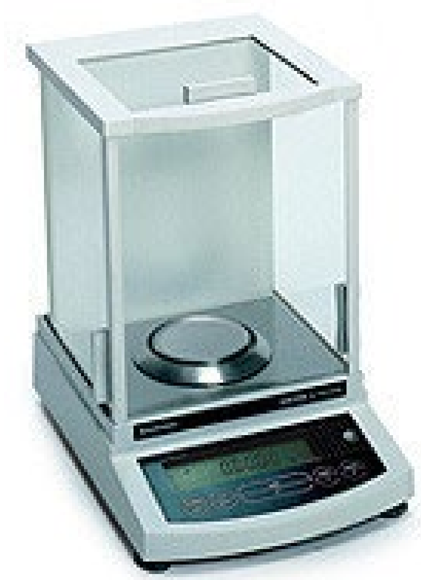
http://www.jsm.gov.my/accredited-organisation-directories

OIML R76

Many Types of Balances

Analytical Balance

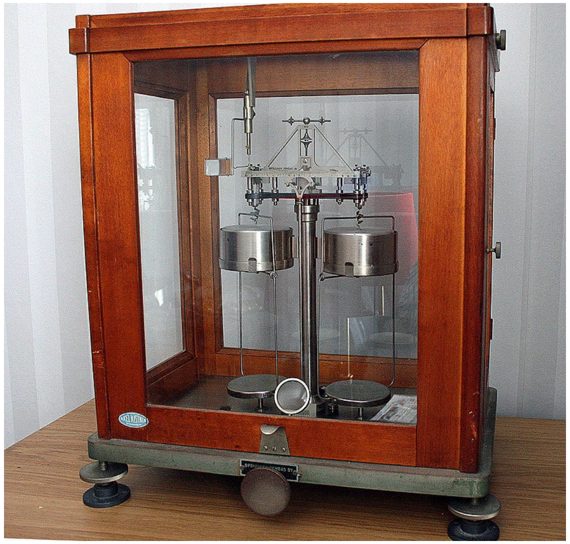
OIML R76
NAWI: Non-Automatic Weighing Instrument



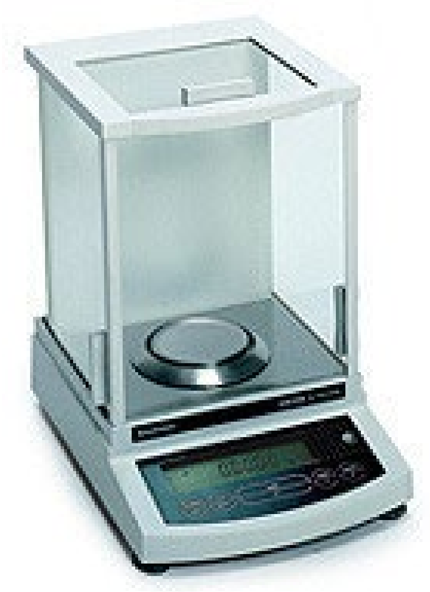
Definition:
An instrument that requires the intervention of an operator during the weighing process, for example to deposit on or remove from the receptor the load to be measured and also obtain the result.

Know Your Balance

Common Type of Balances



Mechanical balance



Electronic balance

Know Your Balance

Types of Electronic Balance

Balance	Readability / Resolution	Quantity of decimal points
Precision balance (Top pan balance)	1 g - 1 mg	1 g - 0.001 g
Analytical balance	0.1 mg	0.0001 g
Semi-micro balance	0.01 mg	0.00001 g
Micro balance	0.1 µg	0.000001 g
Ultra micro balance	0.01 µg	0.0000001 g

Most common use balance in UPM:
Analytical balance and Precision balance

Balance

How to use analytical balance

How to use analytical balance

https://www.youtube.com/watch?v=mmgiehwrK54

Balance

Leveling the balance

[Leveling the balance](https://www.youtube.com/watch?v=G_tUQk7nYx0)

https://www.youtube.com/watch?v=G_tUQk7nYx0

Balance

Leveling the balance

Adjustable Feet

Decrease height ↺ counter clockwise

Increase height ↻ clockwise

Location & Operation of Balance

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Location of balance:

- Weighing bench
- Temperature
- Light
- Work room
- Atmospheric humidity
- Air

Operation of balance:

- Levelling
- Draft shield
- Weighing vessel
- Reading
- Switching on/off
- Weighing pan
- Care and maintenance

Refer to balance manual

Operating Range Starting Point

Balance Readability (d)			Operating Range Starting Point	
			Optimal †	Typical ††
0.1 µg	0.0001 mg	0.0000001 g	0.082 mg	1 mg
1 µg	0.001 mg	0.000001 g	0.82 mg	2 mg
10 µg	0.01 mg	0.00001 g	8.2 mg	20 mg
100 µg	0.1 mg	0.0001 g	82 mg	100 mg
1000 µg	1 mg	0.001 g	820 mg	900 mg
	10 mg	0.01 g	8.2 g	8.2 g
	100 mg	0.1 g	82 g	82 g
	1000 mg	1 g	820 g	820 g

† *optimal operating range if the SD from 10 measurements is less than 0.41d*
†† *actual results may vary depending on the installation location/environmental conditions*

USP (United States Pharmacopeia): Chapter 41

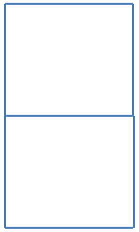
Calibration of Analytical Balance

LAB14: In-house Calibration and Use of Weighing Machines

By: UKAS (United Kingdom Accreditation Service)

Visual Inspection

- Is the instrument being used in an appropriate manner? Ex: Pan is missing
- Is the instrument broken?
- Ex: LCD cannot display reading (must have 7 segment / digit)
- Is the instrument clean?
- Is the instrument operational?
- Is the instrument level?
- Are there any apparent obstructions to the operation of the instrument?



Calibration of Analytical Balance

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Selection of weight

Table 1 A possible selection table of weights for calibration of weighing machines

	Resolution							
Capacity	**100 g**	**10 g**	**1 g**	**100 mg**	**10 mg**	**1 mg**	**0.1 mg**	**<0.1 mg**
Up to 50 g		M3	M3	M3	M2	F2	E2	E1
Up to100g	M3	M3	M3	M3	M1	F1	E1	E1
Up to 500 g	M3	M3	M3	M2	F2	E2		
Up to 1 kg	M3	M3	M3	M1	F1	E1		
Up to 5 kg	M3	M3	M2	F2	E2			
Up to 10 kg	M3	M3	M1	F1	E1			
Up to 50 kg	M3	M2	F2	E2				
Up to 100 kg	M3	M1	F1					
Up to 500 kg	M2	F2	E2					

Note: This table should be interpreted in conjunction with paragraphs 4.2.2 and 4.2.4.

Calibration of Analytical Balance

Commence the Calibration

1. Repeatability Test
2. Off-Center Error
3. Weighing Performance Test
4. Hysteresis

Calibration is carried out on-site

Calibration of Analytical Balance

Repeatability Test

Repeatability is a measure of how well a balance will weigh. The repeatability is normally expressed in terms of the standard deviation obtained from a series of repeated readings.

10 repeat readings are taken, with the weight unloaded and reloaded between each.

Do 2 set of reading using ½ cap and full cap of balance

Calibration of Analytical Balance

Off-Center Error

When the center of the mass of the object being weighed is off-center on the pan, shift or corner load, error may occur

The test is designed to enable user to decide how accurately objects must be positioned on the pan for this effect to be negligible

The test is performed by placing a weight of approximately 1/3 to 1/4 of the capacity of the balance at positions:

center – left – front – right - center

Calibration of Analytical Balance

Weighing Performance Test

Measurements should be at no less than ten (roughly evenly spaced) points from zero to full capacity using calibrated reference weights. The scale should be zeroed each time before placing weights on the pan

A linearity measurements is basically consists of taring the balance, placing the standard weight on the pan and record the reading.

Calibration of Analytical Balance

Hysteresis

Hysteresis occurs when, for a given weight, the balance displays a different reading depending on whether the load is increasing or decreasing.

Steps:

i.	Zero the balance	Z_1
ii.	Place a mass M_1 equal to half of the range	M_1
iii.	Add mass (M_2) to bring the balance reading to full cap	M_1+M_2
iv.	Remove the mass (M_1), read the balance with M_2 still on the pan	M_2
v.	Remove M_2 and read the zero	Z_2

Measurement of Uncertainty (MU)

Adobe Acrobat Document

Source of Uncertainty	Type	Formula
Uncertainty from reference, U_{ref}	B	$U_{ref} = \frac{U_{cert}}{k}$, where U_{cert} is the worst measurement uncertainty of the standard weights (r) used in the calibration from the calibration certificate. The value of $k = 2$
Uncertainty from drift, U_{drift}	B	$U_{drift} = \frac{MPE}{3\sqrt{3}}$, where MPE is the highest maximum permissible error of the standard weights used in the calibration.
Uncertainty from equipment resolution, U_{res}	B	$U_{res} = \frac{d}{2\sqrt{3}}$, where d is resolution of the balance
Uncertainty from air buoyancy, U_{ab}	B	$U_{ab} = \frac{MPE}{4\sqrt{3}}$
Uncertainty from calibration, U_{rep} (for all three tests, repeatability, weighing performance, and off-center loading)	A	$U_{rep} = \frac{\sigma}{\sqrt{n}}(t)$, where σ is standard deviation, t = 2.3. Choose the highest U_{rep} among the three tests

Balance Calibration Cert

Report No. :	2020/FK/MML/RI020
Job No. :	2020/FK/MML/JI005.12
Date of Received :	20/07/2020
Date of Calibration :	06/08/2020
Date of Report Issued :	02/09/2020
Environmental Condition	
Ambient Temperature :	(23.5 to 24.3) ° C
Relative Humidity :	(56 to 59) % Rh
Calibrated Instrument	
Brand :	AND
Model :	GR-200
Capacity :	210 g
ID No. :	S00391075 / SAA34531
Resolution :	0.0001 g
Calibration Procedure :	In accordance with EURAMET Calibration Guide No. 18 Version 4.0 (11/2015) and LAB 14 In-house Calibration and Use of Weighing Machines Edition 5, (July 2015).

The uncertainties quoted in this report have been calculated at the 95% level of confident with a coverage factor k = 2.

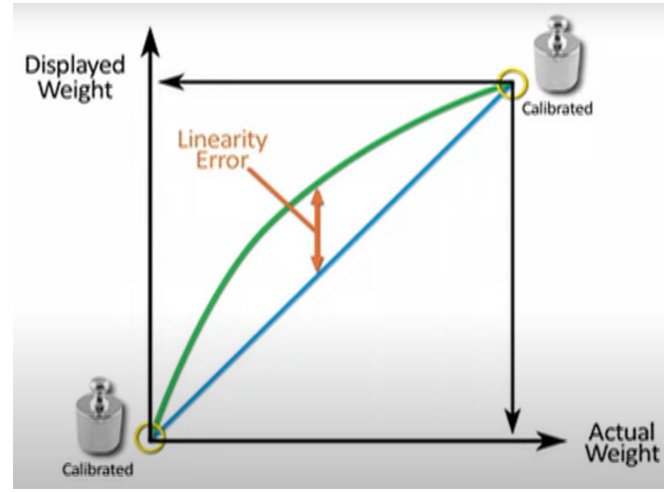
Equipment Used

Description	Certificate No.	Calibration Due Date	Tracebility
Standard Weight	NMIM-0684-M-20	10-March-2021	NMIM
Thermohygrometer	PSYP-20032220	11-June-2021	NMIM

Balance Calibration Cert

<u>Result of Calibration</u>

Unit in : g

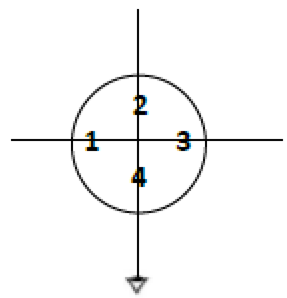


1. Weighing Performance Test

Nominal Load	Error
20	0.0006
40	0.0012
60	0.0017
80	0.0021
100	0.0030
120	0.0037
140	0.0044
160	0.0051
180	0.0056
200	0.0064

2. Repeatability Test

	Half Capacity	Full Capacity
Load	100	200
Standard Deviation	0.0002	0.0002



3. Off-Centre Test

Load	:	50			
Position	:	1	2	3	4
Error	:	0.0000	± 0.0002	± 0.0002	0.0000

4. Hysteresis

M1 - M2	:	0.0000
Z1 - Z2	:	-0.0001

Measurement Uncertainty

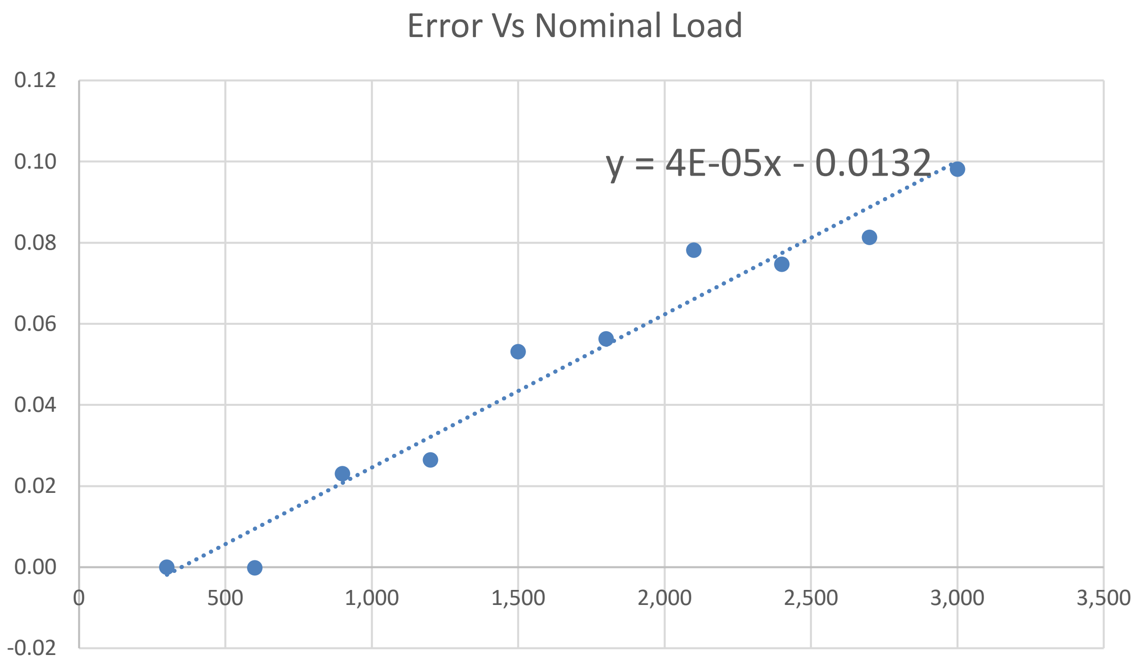
The estimated uncertainty of the calibration result with 95% confidence level with a coverage factor of K=2 is

U = ± 0.0013 g

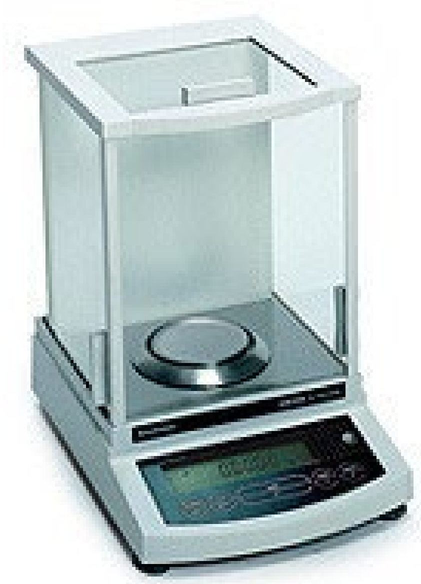
Calibration of Analytical Balance

Next step after getting the calibration certificate…

Plot graph Error vs Nominal Load



Verification of Analytical Balance



Verification is the confirmation, based on evidence (test results) that a certain number of specified requirements have been fulfilled. For example, the verification of an analytical balance will prove that the performance of the balance is still in agreement with the calibration certificate.

1. Verify the accuracy / trueness

Trueness (% relative error), comparing to the weights of certified standard weights. The average weight $x_{average}$ of ten consecutive measurements of a certified standard weight is measured using the analytical balance. The absolute error is calculated and then the **% relative error** according to:

$$\text{Absolute error} = x_{average} - x_t$$

$$\text{\% relative error} = [\,(x_{average} - x_t)/\, x_t]\ \times 100$$

% relative error are a measure of the **accuracy** (trueness) of the analytical balance.

The definition of trueness according to VIM 2010 (International Vocabulary of Terms in Legal Metrology) is: ***closeness of agreement between the average of an infinite number of replicate measured quantity values and a reference quantity value.***

2. Verify the precision

Repeatability (% coefficient of variance, %RSD), comparing to the weighs of certified standard weights.

The **standard deviation** from the mean value of ten consecutive measurements of a certified weight is determined. Then the % relative standard deviation (%RSD) is calculated.

$$\text{Standard deviation} = s = [\Sigma(x_i - x_{average})^2 / (n - 1)]^{1/2}$$

Where $x_{average}$ the average value of the ten consecutive measurements and x each individual measurement and n the number of measurements

$$\%\ \text{relative standard deviation (\%RSD)} = s / x_{average} * 100$$

Repeatability (%RSD) is a measure of the precision of the analytical balance.

The definition of precision according to VIM 2010 (International Vocabulary of Terms in Legal Metrology) is: *closeness of agreement between indications or measured quantity values obtained by replicate measurements on the same or similar objects under specified conditions.*

Acceptance / Rejection Criteria for Analytical Balances	
Trueness, % Relative Error	0.01
Repeatability, % RSD	0.0001

Reference:

http://chem-net.blogspot.com/2012/10/performance-verification-of-analytical.html

Microsoft Excel Worksheet

Verification of Balance Worksheet

Repeatability Test

https://www.youtube.com/results?search_query=repeatability+test+of+balance

Acceptance / Rejection Criteria for Analytical Balances	
Trueness, % Relative Error	0.01
Repeatability, % RSD	0.0001

Reference:

http://chem-net.blogspot.com/2012/10/performance-verification-of-analytical.html

Microsoft Excel Worksheet

Verification of Balance Worksheet

Repeatability Test

https://www.youtube.com/results?search_query=repeatability+test+of+balance