

Laboratory Equipment Calibration

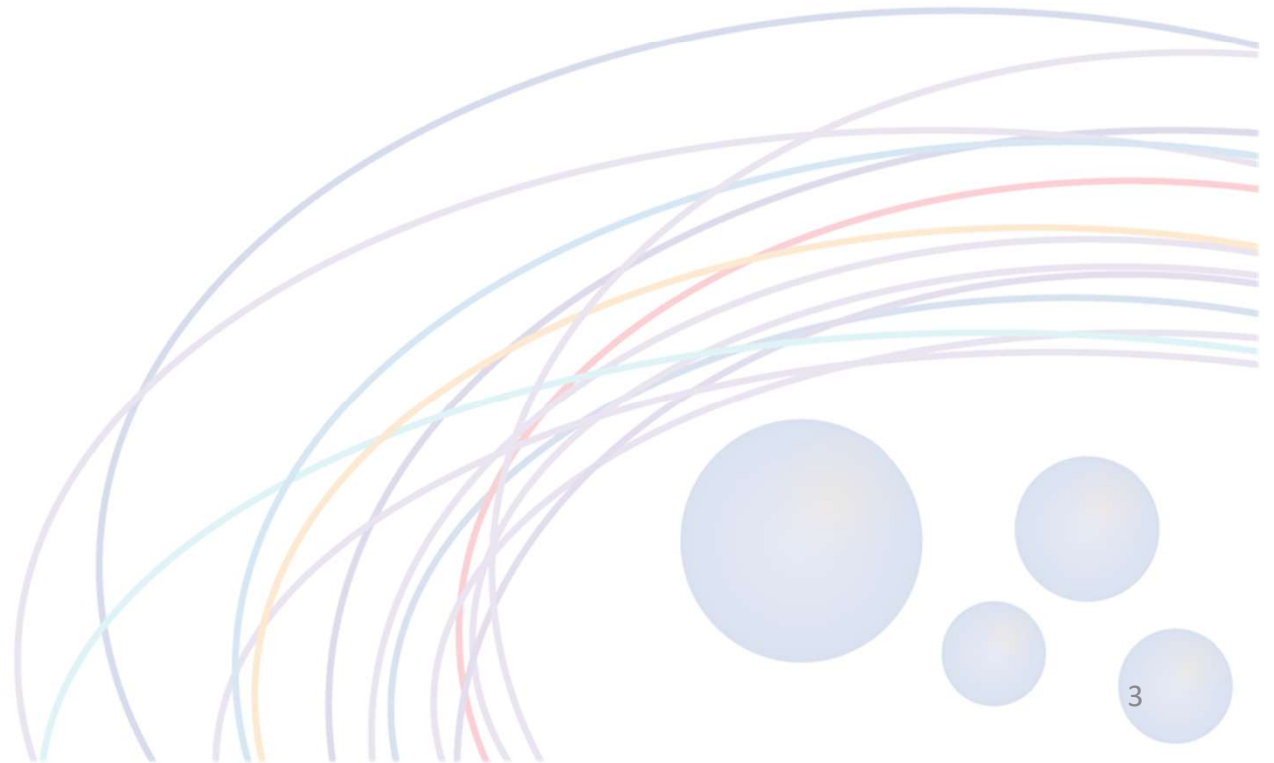
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Contents

- Introduction
- Measurement Uncertainty (MU)
- Equipment Calibration
 - Balance
 - Autopipette (≥ 10 ul)
 - Temperature Measuring Device
 - Temperature Controlled Enclosure
 - Timing Device
 - Centrifuge

Introduction

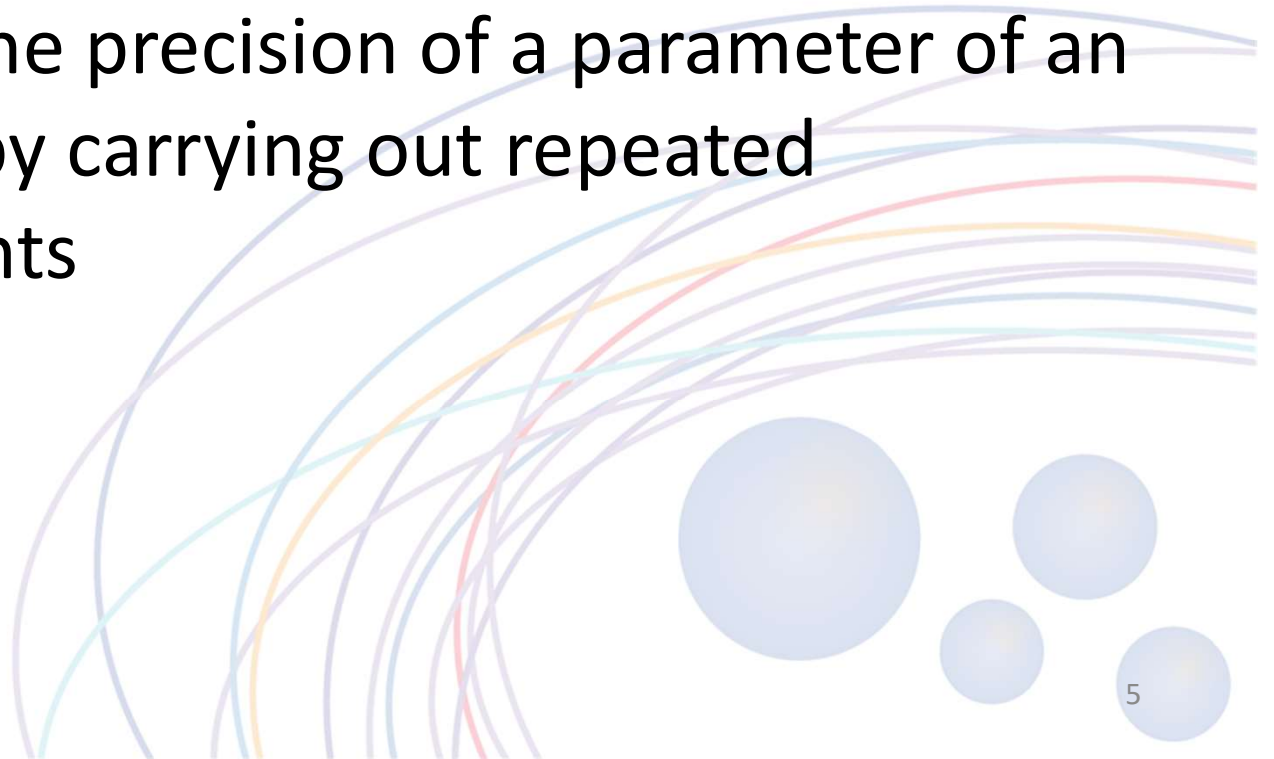


Purpose of Equipment calibration

- To determine the accuracy of the principle parameters of equipment in relation to its use.
- To determine the uncertainty of the principle parameters of equipment within a stated confidence level (usually 95%).
- Any parameter of equipment that will affect the quality of the products has to be calibrated.

Calibration Methods

- a) Determine the accuracy of a parameter of an instrument by comparing with a reference value
- b) Determine the precision of a parameter of an instrument by carrying out repeated measurements



Requirements of Reference Equipment for Calibration

- a) The reference equipment must have a certified accuracy traceable to international/national standards and at least three times (preferably five times) better than that required for equipment being calibrated.
- b) The reference equipment should only be used for calibration purpose and stored securely.

Intervals for Equipment Calibration

- a) It depends on the frequency the equipment being used, its condition and previous calibration and maintenance history, the accuracy and precision required by the test methods for which it is used and the likely influence of the laboratory environment (corrosion, dust, vibration etc.) on the equipment
- b) It should ensure detection of malfunction and continued optimal function of the equipment.
- c) It should be determined by each institute for each piece of equipment in use, using the manufacturer's instruction if available, national guidelines (e.g., HOKLAS SC-02).

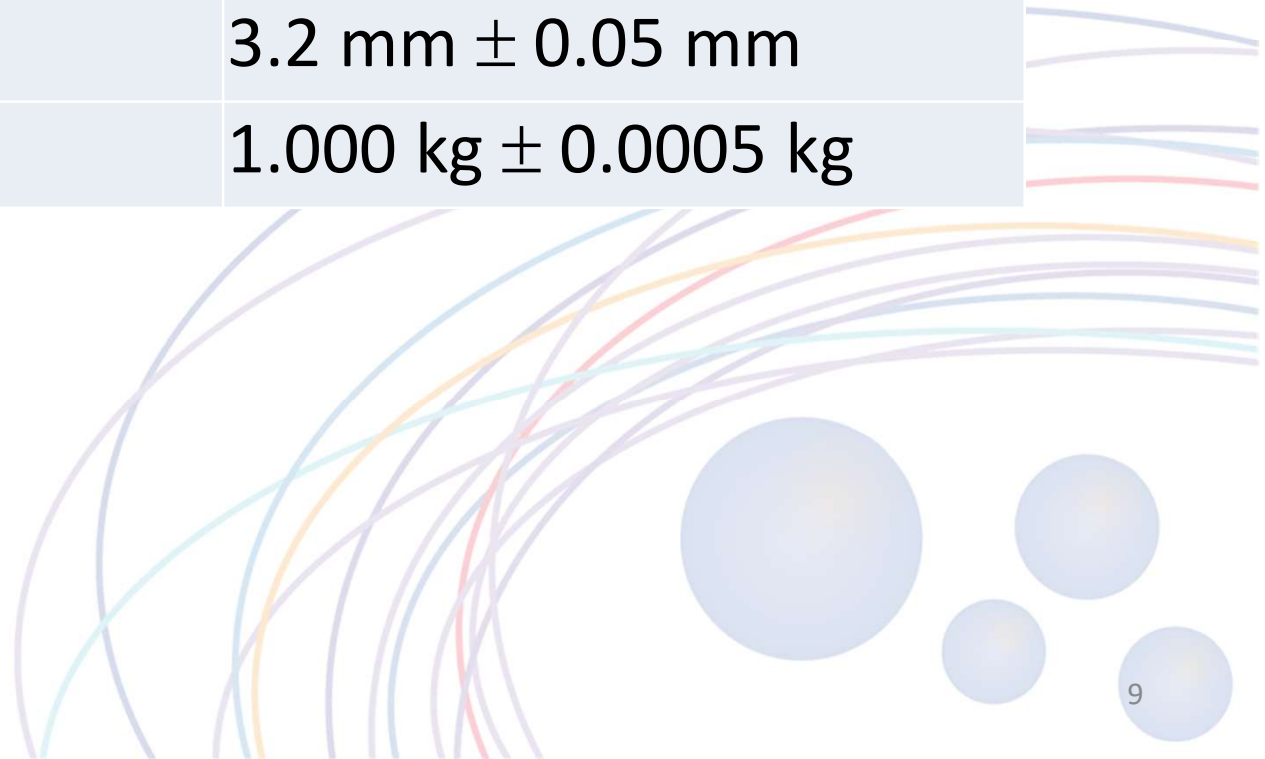
Requirements of Equipment calibration

- HOKLAS SC-13 section 6 Explanatory Notes
 - Laboratories should as far as possible calibrate an instrument using the same measurement method and under the same environmental conditions in which the instrument will normally be used by the client.
 - It is usually not necessary to quote uncertainties to more than two significant figures. Where an uncertainty value is required for subsequent calculations, it may be necessary to retain additional figures to reduce round-off error.
 - The reported estimate for the measurand should be rounded to a number of significant figures consistent with its uncertainty. For example uncertainty is 0.016, the reported estimate for the measurand should be rounded to 7.088.

Working with Numbers

Parameters with no stated tolerance

<u>Stated Parameter</u>	<u>Implied Accuracy</u>
6 mm	6 mm \pm 0.5 mm
3.2 mm	3.2 mm \pm 0.05 mm
1.000 kg	1.000 kg \pm 0.0005 kg

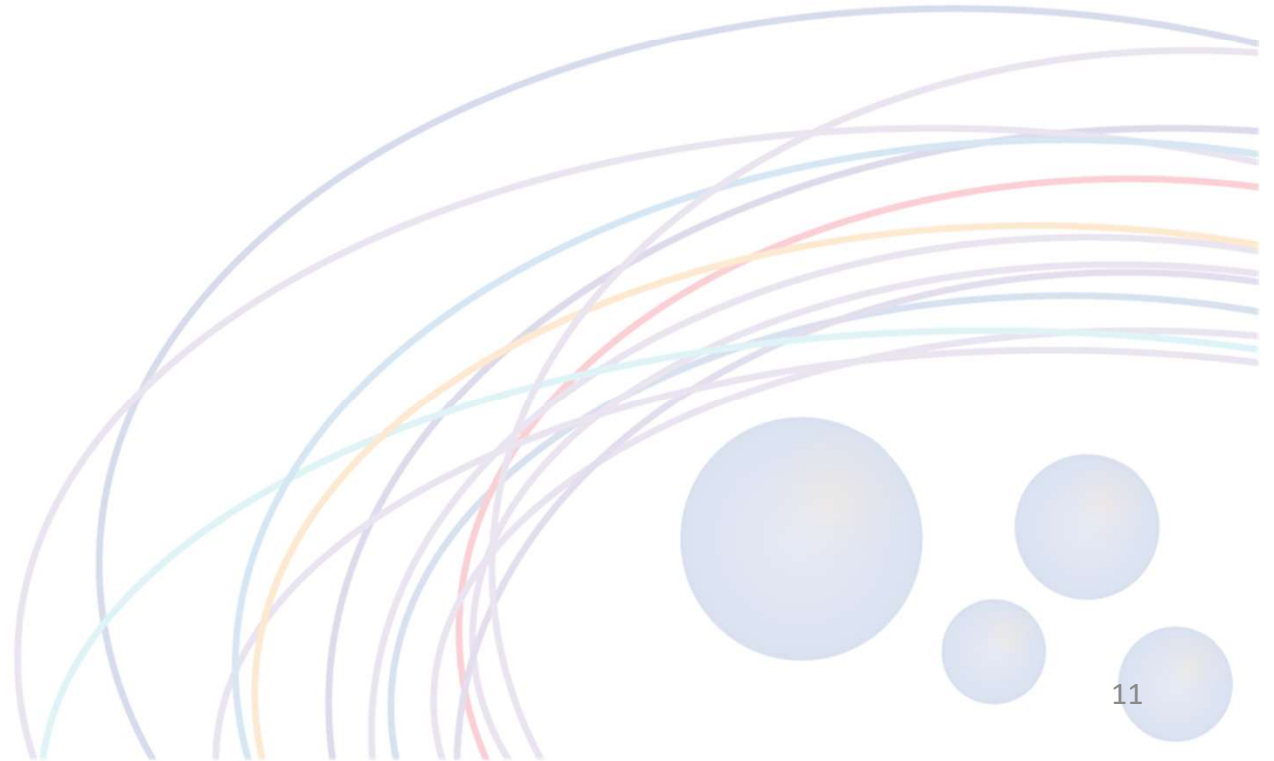


Working with Numbers

Resolution of Equipment with an analogue scale

- When the scale interval is >2.5 mm wide, the resolution is $1/10$ th of a scale interval
- When the scale interval is >1.25 mm wide and <2.5 mm wide, the resolution is $1/5$ th of a scale interval
- When the scale interval is <1.25 mm wide, the resolution is $1/2$ th of a scale interval

Measurement Uncertainty



What is Measurement Uncertainty(MU)?

- MU can be defined as the probable range of a measurement at a specified confidence level (usually at 95%).
- MU is different from an error.

Error	MU
A single value	A range of measurement
Can be applied as a correction to the result	Cannot be used to correct a measurement result

Types of Uncertainty

a) Type A Uncertainty:

Those uncertainties that can be derived by statistical methods.

b) Type B Uncertainty:

Those uncertainties that are derived by means of other than statistical methods. For examples: manufacturer's specifications, data provided in calibration reports and uncertainties assigned to reference data taken from handbooks.

c) Combined Uncertainty:

It is the total of Type A and Type B Uncertainty.

d) Expanded Uncertainty:

It provides an interval (confidence interval) within which the measurement is believed to lie at a specified confidence level.

⇒ MU

Determination of Type A Uncertainty

It is derived from repeated measurements (at least 7, preferably 10).

a) The experimental standard deviation (δ) :

$$\delta = \sqrt{\frac{\sum(x_i - \bar{x})^2}{(n-1)}} \quad \text{where } n = \text{no. of measurement, } \bar{x} = \text{mean of } n \text{ measurements and } x_i \text{ is the } i\text{th measurement}$$

b) The Experimental Standard Deviation of the Mean (ESDM) = $\frac{\delta}{\sqrt{n}} \Rightarrow$
Type A Uncertainty (u_A)

c) The number of Degrees of Freedom (ν) = $n - 1$

d) The expanded uncertainty at 95% confidence level due to Type A uncertainty (U_A) = $k \times \text{ESDM}$,

(obtain k (coverage factor) from the Student's t -table (t -distribution) for a specified degrees of freedom (ν) at a 95% confidence level)

Student t-table (95% confidence level)

v	k	v	k	v	k	v	k
1	12.7	9	2.26	17	2.11	50	2.01
2	4.30	10	2.23	18	2.10	60	2.00
3	3.18	11	2.20	19	2.09	70	1.99
4	2.78	12	2.18	20	2.09	80	1.99
5	2.57	13	2.16	25	2.06	90	1.99
6	2.45	14	2.14	30	2.04	100	1.98
7	2.36	15	2.13	35	2.03	120	1.98
8	2.31	16	2.12	40	2.02	Infinite	1.96

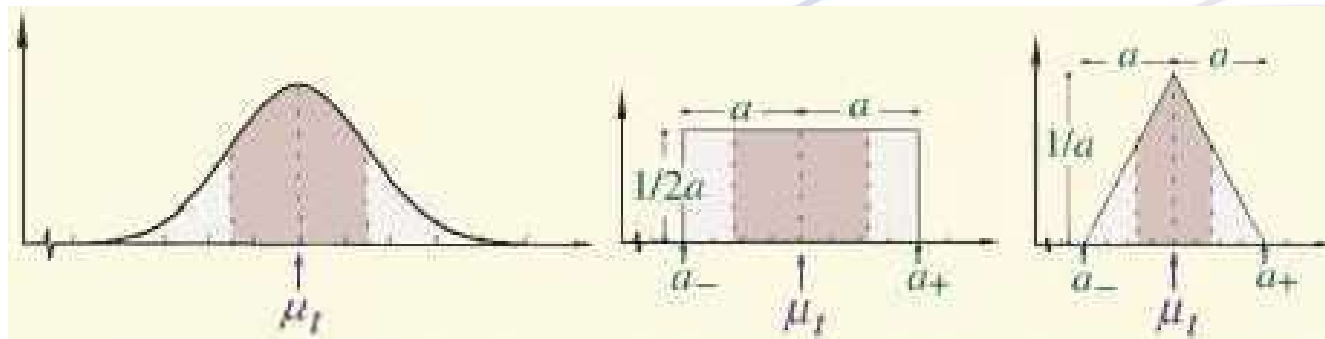
v: Degree of freedom

k: Coverage factor

Determination of Type B Uncertainty

- Obtain a range of limits for a value Y (range limits = $\pm a$)
- The uncertainty due to Type B Uncertainty (u_B) = $n \times a$,

where



Normal
 $n = \frac{1}{2} = 0.5$

Rectangular
 $n = \frac{1}{\sqrt{3}} = 0.577$

Triangular
 $n = \frac{1}{\sqrt{6}} = 0.408$

Determination of Type B Uncertainty

c) Determine the relative unreliability for the range limits (R%)

d) The number of Degrees of Freedom (ν) = $\frac{1}{2} \left(\frac{100}{R} \right)^2$

e) Obtain the k (coverage factor) from the Student's t-table (t-distribution) for a specified degrees of freedom (ν) at a 95% confidence level

f) The expanded uncertainty at 95% confidence level for Type B
Uncertainty (U_B) = $k \times u_B$

Determination of Type B Uncertainty

Example:

The scale division of a working thermometer is 1°C .

The readability is $\pm 0.5^{\circ}\text{C}$ (scale interval $< 1.25\text{mm}$)

The uncertainty of readability (u_B) = $n \times a = \frac{1}{\sqrt{3}} \times 0.5^{\circ}\text{C} = 0.29^{\circ}\text{C}$

The relative unreliability for the readability (R%) = 10%

The number of Degree of Freedom (ν) = $\frac{1}{2} \left(\frac{100}{R} \right)^2 = 50$

The expanded uncertainty of readability at 95% confidence level
= $k \times u_B = 2.01 \times 0.29^{\circ}\text{C} = 0.58^{\circ}\text{C} \Rightarrow 0.6^{\circ}\text{C}$

Calculation of Combined Uncertainty

The combined uncertainty is obtained by combining the individual uncertainties u_i , whether arising from a type A Uncertainty or Type B Uncertainty by the following two simple rules:

a) Rule 1

$$u_y^2 = u_p^2 + u_q^2 + u_r^2 + \dots$$

$$u_y = \sqrt{u_p^2 + u_q^2 + u_r^2 + \dots}$$

b) Rule 2

$$\left(\frac{u_y}{y}\right)^2 = \left(\frac{u_p}{p}\right)^2 + \left(\frac{u_q}{q}\right)^2 + \left(\frac{u_r}{r}\right)^2 + \dots$$

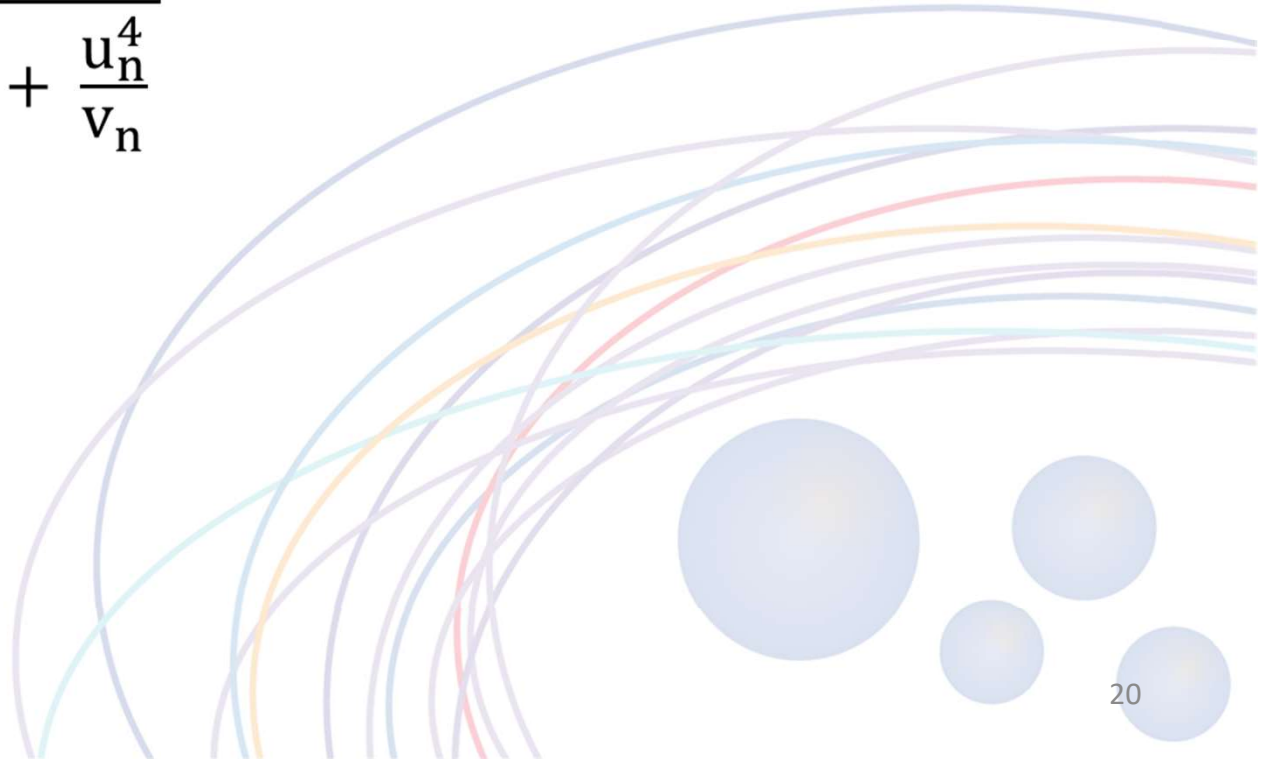
$$u_y = y \sqrt{\left(\frac{u_p}{p}\right)^2 + \left(\frac{u_q}{q}\right)^2 + \left(\frac{u_r}{r}\right)^2 + \dots}$$

where $\frac{u_p}{p}$ etc. are the relative uncertainties of that parameters

Calculation of Effective Degree of freedom

The effective degrees of freedom is obtained from the Welch-Satterthwaite formula:

$$v_{\text{eff}} = \frac{u_c^4}{\frac{u_1^4}{v_1} + \frac{u_2^4}{v_2} + \dots + \frac{u_n^4}{v_n}}$$



Calculation of Expanded Uncertainty (U)

- a) It is obtained by multiplying the combined uncertainty by k (coverage factor) from the Student's t -table for a specified effective degrees of freedom.

$$U = k \times u_c \quad \text{where } k \text{ is called "Coverage factor"}$$

- b) Typically, the coverage factor is in the range of 2 to 3. When the measurement results and the combined uncertainty is approximately normal distribution, $k = 2$ defines an interval having a level of confidence of approximately 95 percent and $k = 3$ an interval having a level of confidence greater than 99 percent.
- c) $U \Rightarrow MU$

ILAC Policy for Uncertainty in Equipment Calibration

ILAC P14:01/2013

- The uncertainty covered by the calibration and measurement capability shall be expressed as the expanded uncertainty having a specific coverage probability of approximately 95 %.
- The reported expanded uncertainty of measurement is stated as the standard uncertainty of measurement multiplied by the coverage factor k such that the coverage probability corresponds to approximately 95 %.

Steps for Determination of MU

- 1) Specify measurand (Particular quantity subject to measurement e.g., time temperature, pressure)
- 2) Identify uncertainty sources (e.g., Reagent purity, instrument effects and measurement conditions)
- 3) Quantify uncertainty components by appropriate methods (Type A / Type B uncertainty)

Steps for Determination of MU

- 4) Calculate combined uncertainty (Rule 1 / Rule 2)
- 5) Calculate expanded uncertainty ($U = k \times u_c$)
- 6) Report MU at a specified confidence level (95%)



Calibration of Balance

- Laboratory analytical balance
- Top-pan electronic balance
- Frequency of calibration
 - Check of precision using standard masses for every **six months**
 - Full calibration comprising check of accuracy and precision, tests for off-centre loading and Hysteresis for every **three years**
 - Check of scale value using nominal masses (Daily or day of use)
 - Zero check before every weighing

Pre-calibration Preparation

- Calibrate the balance in the room where it is located under known ambient conditions.
- Place the reference masses near the balance for **at least one hour** to allow thermal equilibrium and record the temperature.
- Switch on the balance and allow **at least one hour** for warm up.
- Optimize the level of the balance if level indicator is available.
- Use weighing tweezers to handle reference masses. For heavy masses, wear disposable gloves or powder-free surgical gloves during handling of the reference masses.
- Set the unloaded balance to zero and apply the reference and/or nominal masses gently to the centre of the load pan of the balance. Do not zero the balance during the procedure unless specified.

Calibration of Balance



Check of Precision (Repeatability Check)

- Check the consistency of the reading by estimating the standard deviation of a series of repeated readings
- Take a minimum of 10 successive readings at near zero weight, half the full range and full range of the balance. (Note: the balance **should not be zeroed** during the series of successive readings)
- Calculate the standard deviation, σ , for these three series of readings by :

$$\sigma = \left[\frac{1}{(n-1)} \sum_{i=1}^n (R_i - M)^2 \right]^{1/2} \text{ or calculated by Excel i.e. " =STDEV(N7:N16) "$$

where

R_i = scale reading (m_i) - zero reading (z_i)

M = the average of series of readings (R)

n = the number of readings in the series

- Take the largest standard deviation among these three check points as the **maximum standard deviation (msd)** of the balance
- The new msd should be less than 2 x the previous σ if applicable.
- The maximum difference between successive readings in a series should not increase significantly over the previous value.
- The msd should be less than the resolution of the balance.

Check of Accuracy

- Weigh a single reference mass of value 'M' not greater than $1/10$ th of the nominal range of the balance for the first check point by a symmetrical weighing method

- **Symmetrical Weighing**

- A symmetrical weighing is performed in the following sequence, the weight being removed from the pan momentarily and placed back on the pan for the second reading:

zero	(z_1)	prior to putting weight on balance pan
scale reading	(m_1)	first reading
scale reading	(m_2)	second reading after removal and replacement of weight
zero	(z_2)	after removing weight

The measured mass of the object being weighed, m

$$= (m_1 + m_2) / 2 - (z_1 + z_2) / 2$$

Check of Accuracy

- Remove the reference mass and place tare masses on the pan until the balance displays the previous scale value reading m_1 (or close to this value). This effectively becomes the new zero reading (**DO NOT zero the balance**)
- Re-weigh the reference mass with m_1 as the new zero reading by the symmetrical weighing method for the second check point
- Repeat the above steps for the other check points until the full range is reached.
- Calculate the correction for the i th check point as follows:
 - $C_i = M - (m_i - z_i)$ & find the C_m
 - Calculate the Limit of Performance for an electronic balance (F)
 - $F = 2.26 s_w + |\text{Corr}_w| + U(C_{\text{Cal}})$

Limit of Performance

- $F = 2.26 s_w + |Corr_w| + U(C_{Cal})$

s_w	is the larger standard deviation of the repeatability of either: a) that reported on the last full calibration report S_r ; or b) that determined by the user repeatability check $S_{user\ check}$
$ Corr_w $	is the absolute value of $ C_{Cal} + \Delta C_{user\ check} $
$ C_{Cal} $	is the largest correction reported on the last full calibration report.
$\Delta C_{user\ check} $	is the change in user check correction from that measured at the time of the last full calibration , to that measured now.
$U(C_{Cal})$	is the expanded uncertainty associated with $ C_{Cal} $ as shown on the calibration report.

Uncertainty Calculation

- **Type A uncertainty:**

- Standard deviation, σ (msd) , of precision check/ repeatability of reading
- The Experimental Standard Deviation of the Mean (ESDM) = σ / \sqrt{n}
- where n = no. of readings and $\nu_{\sigma} = n-1$.

Calibration results of reference weights

<u>Nominal Value (g)</u>	<u>Measured Value, y (g)</u>	<u>Expanded Measurement Uncertainty, U (g)</u>	<u>Coverage Factor k</u>
200	199.999 95	0.000 08	1.99
100	99.999 96	0.000 05	1.98
20	19.999 987	0.000 020	1.99
1	1.000 003	0.000 006	2.02
0.1	0.099 996	0.000 004	2.00
0.05	0.050 006	0.000 003	2.00
0.01	0.009 978	0.000 002	2.00

Uncertainty Calculation

- **Type B uncertainty:**

- Reference weights_100 g (M)_Actual weight: 99.99996 g
- Uncertainty of calibration + 0.00005 g at 95% confidence level for the reference mass being used
- Coverage factor is 1.98
- $\mu_s = 0.00005 / 1.98 = 0.000025$ and $\nu_s = 100$
- Combined uncertainty in balance = $\mu_c = \sqrt{(\mu_s^2 + (\text{ESDM})^2)}$
- Effective degrees of freedom = $\nu_c = \mu_c^4 / ((\text{ESDM})^4 / \nu_s + (\mu_s)^4 / \nu_s)$
- By ν_c find the factor, k, from the table.
- $k * \mu_c$ gives the expanded uncertainty of the balance at 95% confidence level.

Student t-table (95% confidence level)

v	k	v	k	v	k	v	k
1	12.7	9	2.26	17	2.11	50	2.01
2	4.30	10	2.23	18	2.10	60	2.00
3	3.18	11	2.20	19	2.09	70	1.99
4	2.78	12	2.18	20	2.09	80	1.99
5	2.57	13	2.16	25	2.06	90	1.99
6	2.45	14	2.14	30	2.04	100	1.98
7	2.36	15	2.13	35	2.03	120	1.98
8	2.31	16	2.12	40	2.02	Infinite	1.96

Test for Off-centre Loading

- Use a nominal mass, M , of approximately $1/3$ of the full range of the balance.
- Weigh the mass successively on the front, rear, left and right position on the “lip” of the pan by the symmetrical weighing method. The “lip” of the pan is the area where the flat surface of the pan starts to curve up.
- Calculate the range (the difference between the maximum and minimum measured mass) of these five sets of readings.

Test for Hysteresis

- Use a nominal mass, M , approximately $1/2$ of the full range of the balance
- Zero the balance, (z_1).
- Place the mass on the pan and record the scale reading m_1 .
- Add extra mass to the pan until the balance reads full-range.
- Remove extra mass (**Do NOT remove the mass**) and record scale reading m_2 .
- Remove the mass and record zero (z_2).
- Repeat the above steps for three times and express the hysteresis as the average of the differences between ($m_1 - m_2$) and ($z_1 - z_2$).

Routine Check for Scale Value

- One-point check using a known mass on each day of use
- Prepare a mass close to the maximum capacity of the balance or a mass equivalent to the weight to be usually weighed.
- Check the balance with this mass **daily or day of use** to monitor any drift over time.
- Record the daily or day-of-use scale value reading. Consecutive readings of the daily or day-of-use scale value should **not differ by more than $\pm 3\sigma$** (σ =maximum standard deviation; msd)
- The “ σ ” is the largest/ maximum standard deviation from the last precision check of the balance. It shall be revised on a half-yearly interval.
- Each weighing - Zero check after tare.

Operation of balance

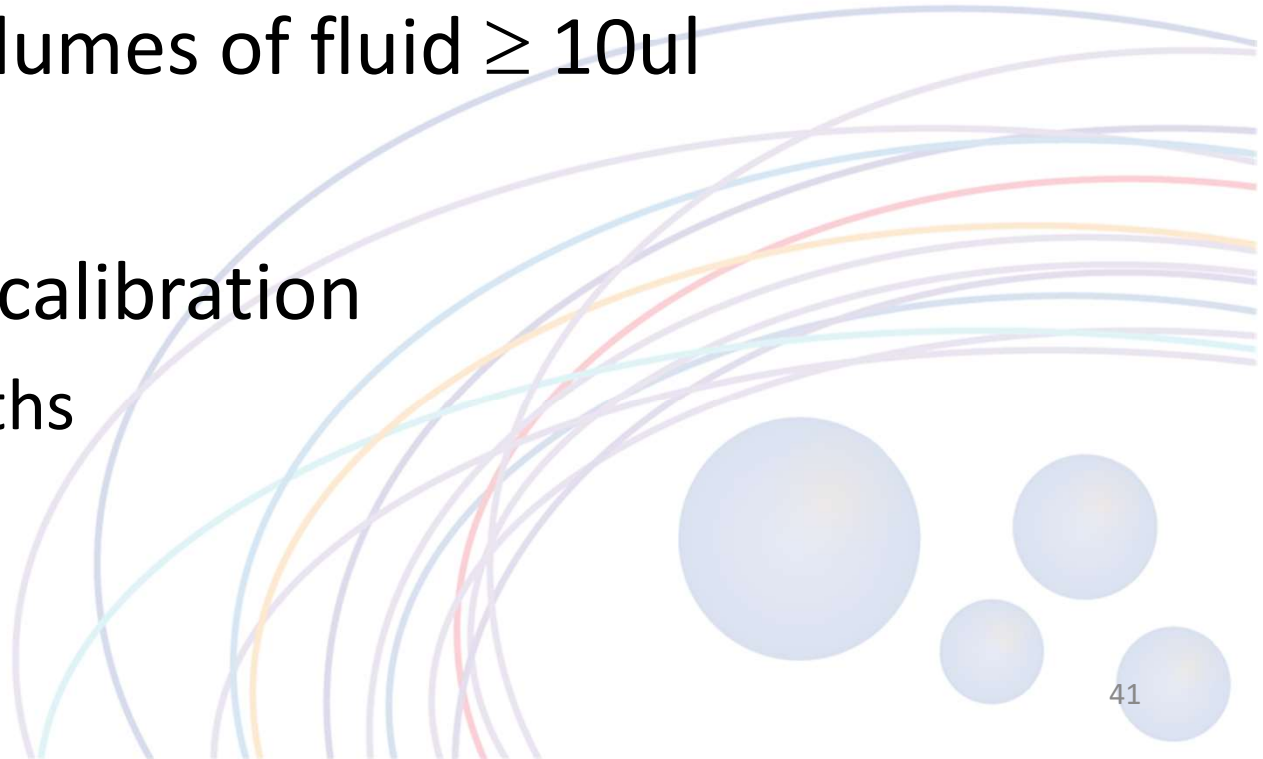
- Cleaning shall be performed without moving the balance.
- Balances should be kept away from windows to avoid temperature changes.
- Balances should be maintained as dust free as possible.
- Air movement (drift) should be minimized.
- Balance should be located on a stable bench and far away from door.
- Heavy objects and centrifuge should not be placed near the balance to avoid vibration.
- Electrical supply shall be kept at all time.
- Balance shall be at zero prior to each weighing.
- Full calibration shall be carried out after relocation of the balance.

Limit of Performance

- *When the balance is operated under an appropriate environment similar to that for its calibration, the balance should be able to give the correct mass of an object within the limit of performance with a probability of not less than 99 times out of a hundred.*

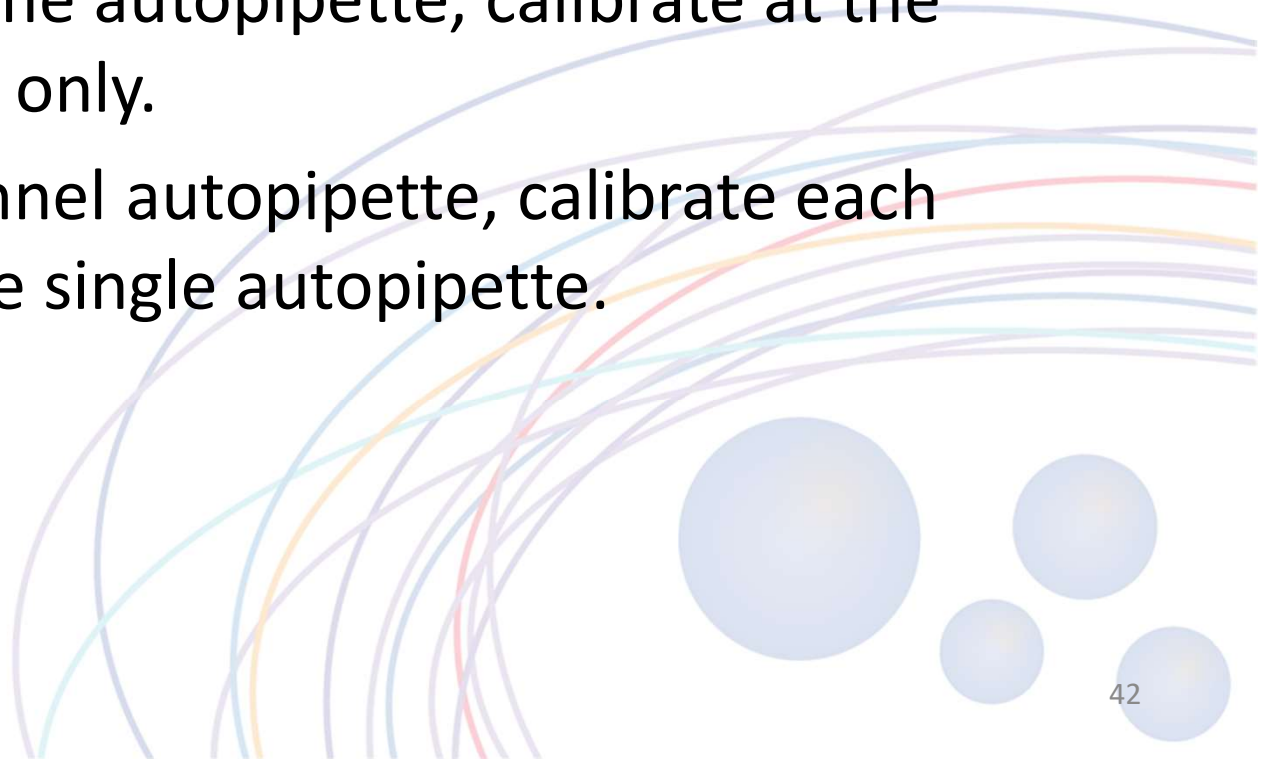
Calibration of Autopipette

- Fixed volume and variable volume autopipette (single or multi-channels) for measuring volumes of fluid $\geq 10\mu\text{l}$
- Frequency of calibration
 - Every 6 months



Calibration of Autopipette

- For variable autopipette, calibrate at least three different volumes including maximum, minimum settings and optimum calibration volume
- For fixed volume autopipette, calibrate at the nominal value only.
- For multi-channel autopipette, calibrate each channel as one single autopipette.



Calibration of Autopipette

- Conduct the calibration in an area that is free from vibration and dust
- The relative humidity shall be between 45% and 80%
- Temperature of $(20 \pm 3)^{\circ}\text{C}$ with a maximum variation of $\pm 0.5^{\circ}\text{C}$
- Place a cup of distilled water near the balance for at least two hours to allow thermal equilibrium
- Set the autopipette to the lowest setting as stated on the autopipette
- Press down the push button and hold the pipette in vertical position, immerse the tip just below the liquid and allow the push button slowly and smoothly to move up to the top stop position
- Wrap the tip and place the pipette tip against the inside wall of the receiving vessel, depress the push-button smoothly and slowly to the bottom stop position

Calibration of Autopipette

- Carefully weight the volume of distilled water dispensed
- Repeat 9 more times and obtain an average
- Measure the temperature of the water and environment, calculate the **corrected volume** ($\text{Density of water} = \text{Absolute density of water} - \text{Density of dry air}$)
- Repeat this procedure using other settings of the autopipette including the maximum dispensable volume and approximately 50% of the nominal volume
- Gently aspirate and dispense distilled water without bubble, keep upright position of autopipette during dispensing

Calibration of Autopipette



Absolute Density of water, g/cm³

°C	0	1	2	3	4	5	6	7	8	9
15	0.999099	084	069	054	038	023	007	*991	*975	*959
16	0.998943	926	910	893	877	860	843	826	809	792
17	774	757	739	722	704	686	668	650	632	613
18	595	576	558	539	520	501	482	463	444	424
19	405	385	365	345	325	305	285	265	244	224
20	203	183	162	141	120	099	078	056	035	013
21	0.997992	970	948	926	904	882	860	837	815	792
22	770	747	724	701	678	655	632	608	585	561
23	538	514	490	466	442	418	394	369	345	320
24	296	271	246	221	196	171	146	120	095	069
25	044	018	*992	*967	*941	*914	*888	*862	*836	*809
26	0.996783	756	729	703	676	649	621	594	567	540
27	512	485	457	429	401	373	345	317	289	261
28	232	204	175	147	118	089	060	031	002	*973
29	0.995944	914	885	855	826	796	766	736	706	676
30	646	616	586	555	525	494	464	433	402	371

Density of Dry Air, g/mL

	Pressure in Centimeters					
	72.0	73.0	74.0	75.0	76.0	77.0
°C						
15	0.001161	0.001177	0.001193	0.001210	0.001226	0.001242
16	157	173	189	205	221	238
17	153	169	185	201	217	233
18	149	165	181	197	213	229
19	145	161	177	193	209	255
20	0.001141	0.001157	0.001173	0.001189	0.001205	0.001221
21	137	153	169	185	201	216
22	134	149	165	181	197	212
23	130	145	161	177	193	208
24	126	142	157	173	189	204
25	0.001122	0.001138	0.001153	0.001169	0.001185	0.001200
26	118	134	149	165	181	196
27	115	130	146	161	177	192
28	111	126	142	157	173	188
29	107	123	138	153	160	184
30	0.001104	0.001119	0.001134	0.001150	0.001165	0.001180

Calculation (Type A uncertainty)

- The standard deviation, σ is used for determining the expanded uncertainty of the measurements:-

- $ESDM(u_a) = \sigma/\sqrt{n}$ ($n = \text{no. of readings taken}$)
 $= \sigma/\sqrt{10} = 0.316\sigma$

- Assuming a coverage factor (t) of 2 at 95% confidence level. The expanded uncertainty = $\mu t = 0.63\sigma$

- Degrees of Freedom (v_a) = $n-1 = 10 - 1 = 9$

Calculation (Type B uncertainty)

- Uncertainty of balance = u_b
- Degrees of Freedom (ν_b) = 60 (Assume the coverage factor = 2)
- Combined Uncertainty:

$$u_c = \sqrt{u_a^2 + u_b^2}$$

- Calculation of effective degrees of freedom

$$\nu_{\text{eff}} = \frac{u_c^4}{\frac{u_a^4}{\nu_a} + \frac{u_b^4}{\nu_b}}$$

- Expanded Uncertainty (U)

$$U = t_\nu \times u_c$$

Calibration of Temperature Measuring Device (TMD)



- **Frequency of calibration**

- One point check at the ice point or the operating temperature at every six months
- Full calibration comprising ice-point and scale checks at every three years

Sources of Uncertainty

- Uncertainty of reference TMD
- Uncertainty due to readability of reference TMD
- Uncertainty due to readability of working TMD



Uncertainty Calculation (TMD)

Ice-point calibration

- Only consider **readability of working thermometer**
- Assume the working thermometer has a scale division of $1\text{ }^{\circ}\text{C}$ and rectangular distribution, so the readability is $\pm 0.5\text{ }^{\circ}\text{C}$. ($\mu_w = W / \sqrt{3}$)
 - i.e. $\mu_w = 0.5 / \sqrt{3} = 0.29$
- From the student t-table, assume coverage factor (k) is 2 and $\nu_c = 60$ and hence the **expanded uncertainty** in ice-point calibration at 95% confidence level is $k \times \mu_w$
 - i.e. $\pm 2 * 0.29 = \pm 0.58\text{ }^{\circ}\text{C}$
- Scale division: $0.5\text{ }^{\circ}\text{C}$
 - expanded uncertainty = $\pm 0.29\text{ }^{\circ}\text{C}$
- Scale division: $0.1\text{ }^{\circ}\text{C}$
 - expanded uncertainty = $\pm 0.06\text{ }^{\circ}\text{C}$



Student t-table (95% confidence level)

v	k	v	k	v	k	v	k
1	12.7	9	2.26	17	2.11	50	2.01
2	4.30	10	2.23	18	2.10	60	2.00
3	3.18	11	2.20	19	2.09	70	1.99
4	2.78	12	2.18	20	2.09	80	1.99
5	2.57	13	2.16	25	2.06	90	1.99
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7	2.36	15	2.13	35	2.03	120	1.98
8	2.31	16	2.12	40	2.02	Infinite	1.96

Uncertainty Calculation (TMD)

Full calibration (Scale calibration)

- Assume the working thermometer has a scale division of 1°C , so the readability is $\pm 0.5^{\circ}\text{C}$. ($\mu_w = W / \sqrt{3}$)
 - i.e. $\mu_w = 0.5 / \sqrt{3} = 0.29$ and $v_w = 50$
- Assume the reference thermometer has a scale division of 0.1°C , so the readability is $\pm 0.05^{\circ}\text{C}$. ($\mu_r = X / \sqrt{3}$)
 - i.e. $\mu_r = 0.05 / \sqrt{3} = 0.029$ and $v_r = 50$
- Assume uncertainty of calibration of the reference thermometer is $\pm 0.1^{\circ}\text{C}$ at 95% confidence level and a coverage factor of 2
 - $\mu_s = 0.1/2 = 0.05$ and $v_s = 60$

Uncertainty Calculation

Combined uncertainty in scale calibration = $\mu c = \sqrt{(\mu w^2 + \mu r^2 + \mu s^2)}$

- i.e. $\mu c = \sqrt{(0.29^2 + 0.029^2 + 0.05^2)} = \underline{+0.3} \text{ } ^\circ\text{C}$
- Effective degrees of freedom = $\nu c = \mu c^4 / (\mu w^4 / 50 + \mu r^4 / 50 + \mu s^4 / 60) = 57$
- From the student t-table, the coverage factor (k) is 2 and hence the **expanded uncertainty** in the scale calibration at 95% confidence level. is k x μc
- i.e. $2 * 0.3 = \underline{+0.6} \text{ } ^\circ\text{C}$
- The main uncertainty component is the readability of the working thermometer if the uncertainty of reference thermometer is very low and the scale division of reference thermometer (readability) is 0.01°C . For example: uncertainty: 0.02°C ; $\mu w = \mu c$
- **Expanded uncertainty** = $2 * 0.29 = \underline{+0.58} \text{ } ^\circ\text{C}$

Temperature Controlled Enclosure (TCE)

- Blood and Reagent Storage Equipment (refrigerator & freezer)
- Incubators for blood tests
- Water bath



Types of calibration

- Centre-point (every six months)
- Full calibration at multiple points (temperature gradient check) at annually



Number of Points for Full Calibration

The number of points of calibration for full calibration are determined by the volume of the TCE

TCE with volume (Litre)	Points of Calibration
501 – 2000	9
251 – 500	5
101 – 250	3
≤100	1

Clearance of calibrated TMD

- Place the calibrated TMD (temperature loggers or thermocouples) at the centre and the eight corners of the temperature enclosure with a clearance specified in the following table:

Volume of chamber (litre)	Clearance
<1000	L/10 or 50 mm, whichever is larger
1000 to 2000	L/10 or 100 mm, whichever is larger

Where L is the corresponding dimension of the chamber

Reference: Guidance on Calibration and Performance Verification of Temperature Chambers (Informative); HKAS Information Notes No. 3; Issue No. 4; Issue Date: 25 July 2022

Centre-point Calibration

- Place a reference TMD at the **centre** of the TCE. Wait for temperature to be stable and then take the reading
- Record the reading and check whether the temperature is within the TCE operating temperature range
- The acceptable temperature range of reference TMD shall be calculated by considering the uncertainty of reference TMD.

Full calibration (spatial/gradient check)

- Allow the temperature of the temperature controlled enclosure to stabilize for at least one hour
- Place the reference TMD at various points (e.g. at the centre and the eight corners of the temperature enclosure) without touching any wall of TCE
- Record the readings and check whether the temperature is within the TCE operating temperature range
- The acceptable temperature range of reference TMD shall be calculated by considering the uncertainty of reference TMD.

Calibration results of Ref. TMD

UUT Reading [°C] (See Note 1)	Correction to UUT Reading			
	Value, y [°C] (See Notes 1 & 2)	Measurement Uncertainty		
		Expanded Measurement Uncertainty, U [°C] (See Note 3)	Coverage Factor, k (See Note 3)	
Sensor labelled “PROBE C (CH1)” connected to Channel 1	-80.16	+0.23	0.02	2.0
	-20.07	+0.19	0.01	2.0
	0.00	+0.16	0.01	2.0
	21.99	+0.13	0.02	2.0
	99.94	+0.10	0.01	2.0
Sensor labelled “PROBE D (CH3)” connected to Channel 3	-80.02	+0.09	0.02	2.0
	-19.96	+0.07	0.02	2.0
	0.01	+0.06	0.01	2.0
	22.00	+0.03	0.01	2.0
	99.95	+0.04	0.01	2.0

Calibration of Timing Device

- Stopwatch
- Hand-held timer
- Centrifuge timer
- Frequency of calibration
 - Calibrate every 6 months



Accuracy Check

- Compare the time measured by the timing device with the time HK Observatory (Telephone No.: 1878200, choose 1 for Cantonese, then 6 and 1; or
- Web clock: https://www.hko.gov.hk/en/gts/time/clock_e.html) or
- A calibrated timing device for a period of time long enough for determining the inaccuracy of the timing device. (e.g., 10 minutes as recommended by HOKLAS Supplementary Criteria No. 2)
- Repeat the time comparison as appropriate
- Record all readings for the timing device
- Calculate the % of deviation for each time in second (s) and report the maximum % of deviation as the inaccuracy of the timing device

Calibration of Centrifuge

- Bench-top centrifuge
- Micro-centrifuge
- Frequency for calibration of speed, timer & temperature
 - Speed : Semi-annually
 - Timer : Semi-annually
 - Temperature : Semi-annually



Calibration of Centrifuge

- **Speed**
 - Select the routinely operated centrifuge speed setting(s) and check the speed with tachometer twice
- **Timer**
 - Select the routinely operated timer setting(s) and check the time with calibrated timer twice
- **Temperature**
 - Put the thermometer probe near the centrifuge built-in temperature detector and set the temperature for calibration
 - Close centrifuge cover and allow it to equilibrate by running if necessary
 - Compare the temperature measured with the thermometer versus the centrifuge display.

Acceptance Criteria

- Speed: $\pm 10\%$ of the set or displayed speed
- Timer: $\pm 10\%$ of the set time
- Temperature: $\pm 2^{\circ}\text{C}$ of set temperature

References

- JCGM100:2008 (GUM)
- JCGM200:2012
- HOKLAS SC-02
- HOKLAS SC-13
- ILAC Policy for Uncertainty in Calibration(ILAC P14:01/2013)
- Guidance on Calibration and Performance Verification of Temperature Chambers (Informative); HKAS Information Notes No. 3

Thank you!

