


Requirements and Methods for Calibration of Equipment in Clinical Laboratories



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- Calibration of Electronic Balance
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Purpose of Equipment calibration

- a. To determine the accuracy and precision of the parameters of an equipment in relation to its use.
- b. To determine the uncertainty of the parameters within a stated confidence level (usually 95%).
- c. To maintain the traceability of the results provided by the equipment





Parameters of an instrument to be calibrated

Any parameter of an instrument that will affect the quality of the products/test results has to be calibrated.

(e.g., a refrigerated centrifuge :
speed, time, temperature)





General Requirements of Equipment Calibration

- a. All equipment must be uniquely identified and labelled.
- b. Acceptance criteria should be set for each type of equipment
- c. All equipment should be calibrated prior to being placed into service, recalibrated on schedule and after major repair.
- d. Equipment calibration method must meet the accuracy and precision requirements of the equipment.





General Requirements of Equipment Calibration

- d. External equip. calibration shall be preformed by organizations nationally accredited for the equipment calibration (e.g., HOKLAS)
- e. In-house equip. calibration must be preformed by trained and competent staff using reference standards traceable to national standards





General Requirements of Equipment Calibration

- f. Equipment calibration records should include the acceptance ranges and results sufficient to support the accuracy and precision of the equipment within the acceptance ranges
- g. All equipment shall carry calibration status labels with at least the following information:
 - Equipment Identification
 - Date of calibration due
 - Signature of authorized staff



Calibration Report of Temperature Measuring Device (TMD) (for equipment calibration and preventive maintenance)

TMD being calibrated : _____ Date of calibration : _____
 TMD Equipment (EI) No. : _____ Env. Temp. (°C) : _____
 Scale division (°C) : _____ Work Instruction No. : _____
 Operating Temp range : _____ Accuracy required : _____
 (°C)
 Reference (Calibrated) : _____ Scale division (°C) : _____
 TMD used
 Reference (Calibrated) : _____ Uncertainty of Ref : _____
 TMD EI. No. : _____ TMD (provided by QD) ± _____
 Calibration programme: [] Full [] one point check

Ice-point calibration:

Indicated steady temperature (°C){IST} : _____
 Difference between ice-point temperature with : _____
 indicated steady temperature(°C){0 - IST } _____
 Expanded uncertainty of calibration at 95% confidence level {Uc1} : ± _____
 (See Appendix 1 for calculation)

Scale Calibration:

Reference (Calibrated) TMD		TMD to be calibrated	Reference (Calibrated) TMD		TMD to be calibrated
Reading	Correction stated if applicable	Reading	Reading	Correction stated if applicable	Reading
The maximum difference between Reference (calibrated) TMD with TMD to be calibrated (°C)				±	

Expanded uncertainty of calibration at 95% confidence level {Uc2} : ± _____
 (See Appendix 1 for calculation)
 Overall error at 95% confidence level : ± _____
 (Combine ice-point and scale calibration)
 (Max Expanded uncertainty among Uc1 & Uc2 + Max difference among ice pt & scale calibration)

Date of latest full check: _____ Acceptable for use with restriction: _____

Conclusion:

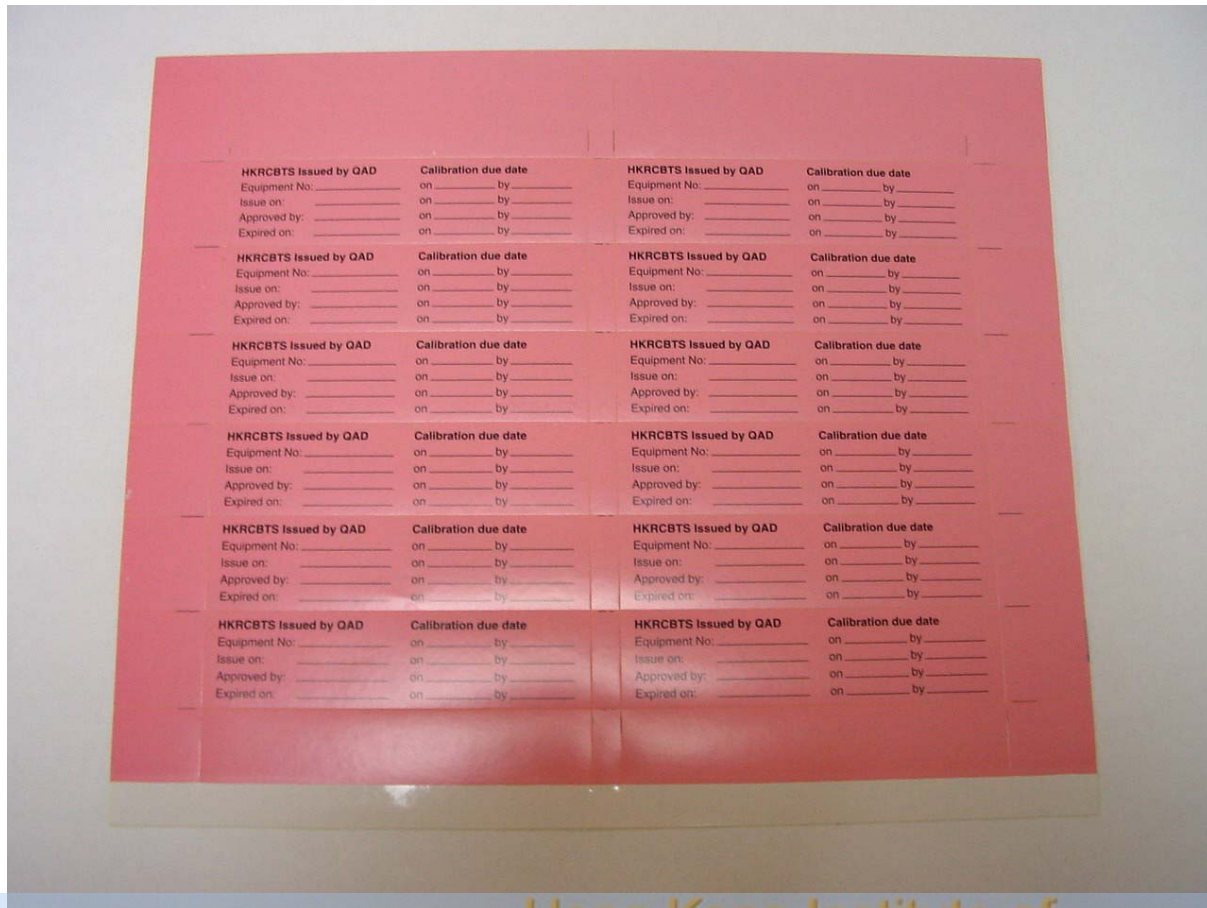
- [] The accuracy of TMD being calibrated is acceptable
- [] Adjustment and re-calibration are required
- [] The accuracy of TMD being calibrated is acceptable with restrictions: _____
- [] The accuracy of TMD being calibrated is not acceptable for its intended use

Name	Signature	Date
Performed by:		
Reviewed by: (Workplace Supervisor)		
Reviewed approved by QD (for issuing of calibration label):		

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Equipment Calibration Label





Equipment Calibration Label

<i>HKRCBTS</i> Issued by QD Equipment No: _____ Issued on: _____ Approved by: _____ Expired on: _____	Calibration due date on _____ by _____ on _____ by _____ on _____ by _____ on _____ by _____ on _____ by _____ on _____ by _____
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Calibration Method

- 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 repeat measurements





Requirements of reference equipment used 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 of the equipment being used, its condition and previous calibration results.
- b) It should ensure detection of malfunction and continued optimal function of the equipment
- c) It should be determined by each institute for each equipment in use based on the manufacturer's instruction or national guidelines (e.g., HOKLAS Supp. Criteria No. 2)

d) It varies from one month to ten years



Calibration of Balance

Requirements:

- a) Every three years: full calibration comprising accuracy, precision, off-centre loading and hysteresis check
- b) Every six months: precision
- c) Daily or day of use: scale value check
- d) Each weighing : zero check





Calibration of Balance

Off-centre loading test

- This test is to determine the effect of eccentric loading of the balance
- Use a nominal mass of approximately $1/3$ - $1/2$ of the full range of the balance
- Weigh the mass successively at the centre, 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)



Calibration of Balance

Off-centre loading test

III) Test for Off-centre Loading

Mass used = 100 g (approximate 1/3 - 1/2 range of balance)							
	1 st Zero	1 st Reading	2 nd Reading	2 nd Zero	Average	Average	
Location	Z_{i1}	m_{i1}	m_{i2}	Z_{i2}	m_i	Z_i	$m_i - Z_i$
Front	0.0000	100.0220	100.0220	0.0000	100.0220	0.0000	100.0220
Rear	0.0000	100.0220	100.0220	0.0000	100.0220	0.0000	100.0220
Left	0.0000	100.0220	100.0220	0.0000	100.0220	0.0000	100.0220
Right	0.0000	100.0220	100.0220	0.0000	100.0220	0.0000	100.0220
Centre	0.0000	100.0220	100.0220	0.0000	100.0220	0.0000	100.0220
Range (difference between maximum and minimum measured mass) = 0.0000							





Calibration of Balance

Precision Check

- 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 throughout the successive weighting)
- Calculate the σ for these three series of readings
- Take the largest σ as the σ of the balance
- The σ should not be greater than the 0.0005 g for a 200 gm four-digit balance.





Calibration of Balance

Accuracy Check (5 ~ 10 point check)

- Weigh a single reference mass of value 'M' ($\sim 1/10^{\text{th}}$ of the full range of the balance) by a symmetrical weighing.
- Calculate the correction of each check point ($C_i = M - (m_i - z_i)$)
- Calculate the Limit of Performance of balance $F = 3\sigma + \text{max. of } C_i$



Standard Masses for Cal. Of Balance

- The uncertainty of the standard mass should not be detected by the balance





Certificate of Calibration (Continuation Page) 校正証書(續頁)

Certificate No. MM030331

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1. The seven standard weights consisted of :-
 - (a) four integral cylindrical stainless steel weights of nominal values from 200 g to 1 g, each having a handling knob on its top, and
 - (b) three fractional weights of nominal values from 100 mg to 10 mg, made of stainless steel sheet.
2. All the weights were housed in a wooden box.
3. Scratch was found on the side of the 100 g weight.
4. The mass of each weight was determined by comparison with a standard weight of the laboratory.
5. The weights were allowed to stabilise in the laboratory environment at 20 ± 1 °C and 50 ± 8 % RH for over 24 hours and placed in or near the balance chamber for 16 hours prior to start of the measurements.
6. Results :

Nominal Value (g)	Measured Value, y (g)	Expanded Uncertainty, U (g)	Coverage Factor k
200	199.999 93	0.000 08	1.99
100	99.999 96	0.000 05	1.98
20	19.999 990	0.000 023	1.99
1	1.000 001	0.000 007	2.01
0.1	0.100 002	0.000 005	1.99
0.05	0.050 003	0.000 004	1.99
0.01	0.009 978	0.000 002	2.00



(II) Check of Accuracy (Departure from Nominal Value)

Reference	1 st Zero	1 st Reading	2 nd Reading	2 nd Zero	Average	Average	C _i
Mass (M _i)	Z _{i1}	m _{i1}	m _{i2}	Z _{i2}	m _i	Z _i	M _i - (m _i -Z _i)
19.999990	0.0000	20.0004	20.0004	0.0000	20.0004	0.0000	0.0004
±0.000023	20.0132	40.0128	40.0128	20.0132	40.0128	20.0132	0.0004
	40.0334	60.0337	60.0337	40.0334	60.0337	40.0334	0.0003
	60.0280	80.0280	80.0280	60.0280	80.0280	60.0280	0.0000
	80.0405	100.0403	100.0403	80.0405	100.0403	80.0405	0.0002
	100.0280	120.0279	120.0279	100.0280	120.0279	100.0280	0.0001
	120.0411	140.0408	140.0408	120.0411	140.0408	120.0411	0.0003
	140.0616	160.0616	160.0616	140.0616	160.0616	140.0616	0.0000
	160.0564	180.0564	180.0564	160.0564	180.0564	160.0564	0.0000
	179.0638	199.0636	199.0636	179.0638	199.0636	179.0638	0.0002

Magnitude of maximum correction (C_m) : 0.0004

The uncertainty (expanded) of the balance at 95% confidence level (μ, refer to Appendix 2 of WIQCL802): 4.8 x 10⁻⁵

Limit of performance of Balance (F = 3μ + C_m): 0.00054





Calibration of Balance

Hysteresis test

- This test checks the variability of a measured mass when its value is reached from the two possible directions of the scale (i.e. direction of increasing load and direction of decreasing load)
- Use a nominal mass, M , $\sim 1/2$ of the full range of the balance
- Zero the balance, (z_1).





Calibration of Balance

Hysteresis test

- Place the mass on the pan and record the scale reading m_1 .
- Add extra weights to the pan until the balance reads full-range.
- Remove extra weights (Do NOT remove the mass) and record scale reading m_2 .
- Remove the mass and record zero (z_2).
- Repeat the test three times and express the hysteresis as the average of the differences between ($m_1 - m_2$) and ($z_1 - z_2$).



Calibration of Balance

IV) Test for Hysteresis

Mass used = 100 g (approximate 1/2 range of balance)					
Initial Zero	1st Reading	2nd Reading	Final Zero		
z_1	m_1	m_2	z_2	$m_1 - m_2$	$z_1 - z_2$
0.0000	100.0220	100.0220	0.0000	0.0000	0.0000
0.0000	100.0220	100.0220	0.0000	0.0000	0.0000
0.0000	100.0220	100.0220	0.0000	0.0000	0.0000
Average				0.0000	0.0000
Average of the difference between $(m_1 - m_2)$ and $(z_1 - z_2)$				0.0000	

Test for Hysteresis

- [✓] The Hysteresis of the balance is acceptable up to the resolution of the balance.
 [] The Hysteresis of the balance is acceptable up to the _____ readable digit.





Calibration of Balance

Daily Scale Check

- Check the balance with a mass close to the maximum capacity of the balance or to the weight to be usually weighed.
- It should be within $\pm 3\sigma$.
- Check the mass every six months and re-state the weight of the mass if it is out of the range.





Acceptance criteria

- Off-centre loading: not more than σ .
- Hysteresis : not more than σ .
- $F (3 \sigma + \max. \text{ of } C_i)$: not higher than the resolution of balance (e.g., 0.0005 gm for a 200 gm 4-digit balance)
- Daily Scale check: within 3σ .





Calibration of Autopipette

Requirements:

- Check the delivered volumes (accuracy and precision) by weighting method every 3 months
- For adjustable autopipette, check at least five delivered volumes including max., min., and optimal calibration volume
- For multi-channel autopipette, check each channel as one single autopipette





Optimal Calibration Volume

- The accuracy and precision of an autopipette are not the same throughout the whole volume range.
- It usually creates more relative error in the lower volumes than in the higher volumes.
- To achieve the smallest relative inaccuracy throughout the entire volume range, the autopipette should be adjusted at the optimal calibration volume (V_{cal}) which is equal to:
$$(2 \times V_{max} \times V_{min}) / (V_{max} - V_{min})$$





Optimal Calibration Volume

Volume Range	Calculated V_{cal}	V_{cal} for calibration
40 – 200 ul	66.7 ul	70.0 ul
200 – 1000 ul	333 ul	300 ul



Balance for Autopipette Calibration (British Standards)

Volume Range	Readability	Repeatability
1 ul – 10ul	0.001 mg	± 0.002 mg
10 ul – 50ul	0.01 mg	± 0.02 mg
Above 50 ul	0.1 mg	0.2 mg





Calibration of Autopipette

Procedure:

- Carefully weigh the dispensed volume of distilled water ten times
- Calculate the average and standard deviation (sd) of these ten data
- Divide the average weight by the density of water to get the volume value (V)
- Accuracy = $(1 - |V-v|/v) \times 100\%$
- Precision (CV%) = $(sd/av. wt) \times 100\%$



Calibration of Autopipette





Calibration of Autopipette

Acceptance Criteria

- Both accuracy and precision are equal to or less than that stated in the package insert of the autopipette



Adjustment of Autopipette

Optimal Cal. Volume	Average dispensed Volume	Acceptable range (± 2 sd)
300 ul	297 ul	295 -305 ul
300 ul	306 ul	295 -305 ul



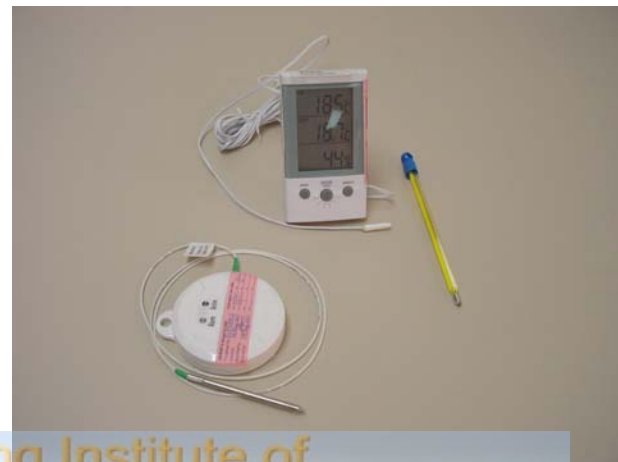
Calibration of Dispensing unit of PK7200



Calibration of Thermometer

Requirements:

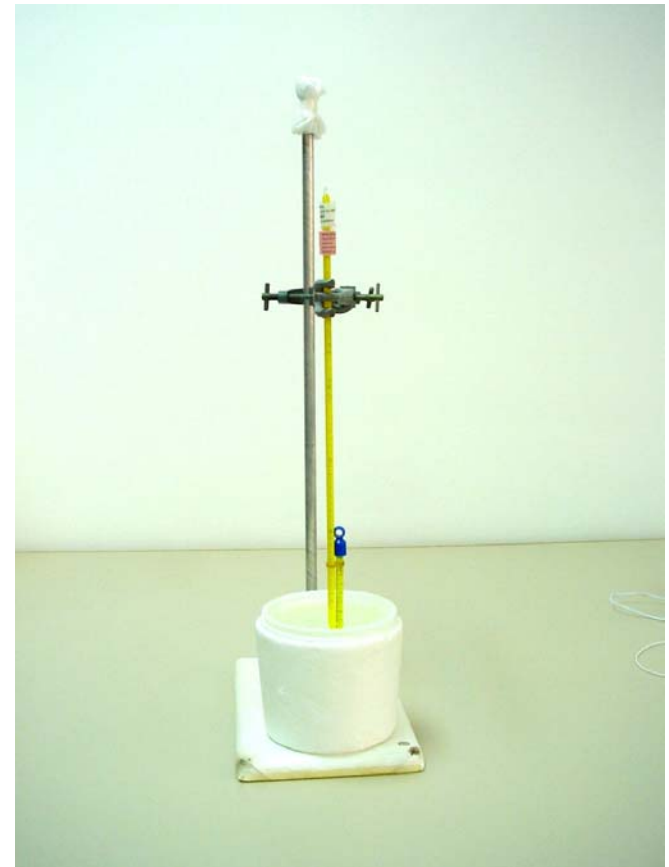
- Every three years: at least three points check (including ice-point) to cover the working range of the thermometer
- Every six months : one point check at the ice point or other working temperature



Calibration of Thermometer

Procedure

- Place the sensors of the both reference thermometer and the thermometer to be calibrated close to each other in a bath
- Read and record their temp. readings





Preparation of Ice-bath

- Fill a thermos flask with flaked ice.
- Add distilled water to the flaked ice unit it just loses its opacity.
- Mix ice with water thoroughly.
- Allow the mixture for thermal equilibrium (~ 10 minutes).





Calculation of Uncertainty

If both reference and test thermometers are digital type thermometers, the overall uncertainty of the temperature measured by the test thermometer is:

Expanded Uncertainty of the reference thermometer + the max. difference between the readings of the reference thermometer and the test thermometer



Calibration Report

校正報告

Report No. TE000322

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9. The calibration results are as follows :

UUT Reading (°C)	Observed Correction to UUT Reading (°C)	Uncertainty of Correction, U (°C)	Coverage Factor, k
Note 1	Note 1, 2	Note 3	Note 3
-80.0	+0.3	± 0.1	2.0
-40.0	+0.1	± 0.1	2.0
20.0	+0.1	± 0.1	2.0
60.0	+0.1	± 0.1	2.0
79.9	+0.2	± 0.1	2.0

Note 1 : The values of the “UUT Reading” and the “Observed Correction to UUT Reading” are stated to the nearest least significant digit (LSD) of the UUT.

Note 2 : The “Observed Correction to UUT Reading” is defined as the temperature measured by the laboratory standard minus the “UUT Reading”.

Note 3 : The reported uncertainties are the expanded uncertainties U for a level of confidence of 95%, together with their coverage factors k. The combined standard uncertainties u_c can be calculated as $u_c = U/k$ and their degrees of freedom ν_{eff} are given by the t-distributions with their respective k values.





Acceptance Criteria

- Thermometer

- The accuracy of the thermometer should be suitable for its intended application

e.g. $\geq 0^{\circ}\text{C}$ = not more than $\pm 0.5^{\circ}\text{C}$

$< 0^{\circ}\text{C}$ = not more than $\pm 2^{\circ}\text{C}$





Calibration of Temp. Controlled Enclosure

Requirements:

- Every two years: multiple points check (e.g., nine points) to ensure that the temperature throughout the enclosure is within the working temperature.
- Every six months : center-point check





Calibration of Temp. Controlled Enclosure

Procedure (multiple points check):

- a. Place the calibrated temperature loggers at the centre and the eight corners of the temperature enclosure without touching any wall of the temp. controlled enclosure (TCM).
- b. Record the temperature change of the temperature loggers and check if there is any temperature out of the working temperature range of TCM



Calibration of Platelet Incubator Temperature



Calibration of Timer

- a) Requirement :
check the accuracy
every 3 months
- b) Standards:
Time signals from
HK Radio or HK
Observatory (Tel.
No. 1878200)





Calibration of Timer

Procedure

- Compare the time measured by the timer with the standard time for at least 10 minutes.
- Repeat the time comparison as appropriate
- Calculate the % of deviation and report the max. % of deviation as the inaccuracy of the timer
(Acceptance criterion: less than 1% deviation from the standard time)





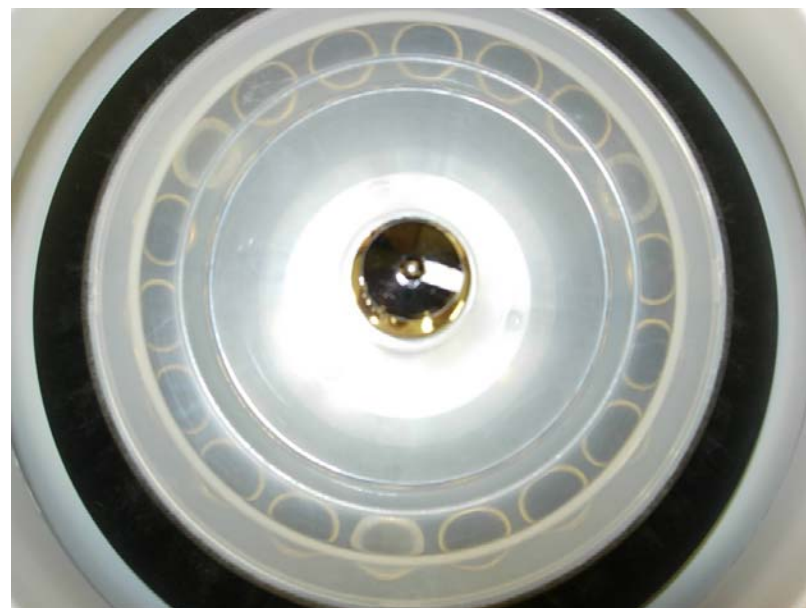
Calibration of Centrifuge

Requirements:

- Speed :
Quarterly by Tachometer
- Timer :
Quarterly by calibrated timer
- Temperature :
Semi-annually by calibrated thermometer



Calibration of Centrifuge Speed





Calibration of Centrifuge

Acceptance Criteria:

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



Calibration Report

The unit-under-test (UUT) was allowed to stabilize in the laboratory for over 24 hours before the commencement of the test.

Test equipment :

Equipment ID
CL154

Description
Multifunction Waveform Synthesizer

Certificate No.
C040106

Test procedure : MISC011N.

Results :

Measurement Accuracy (Optical Mode)

Applied Value (rpm)	UUT Reading (rpm)	Mfr's Spec.	Uncertainty of Applied Value (rpm)
600.00	599.9	±(0.01 % + 1 dgt)	± 0.01
1 000.00	999.9		
2 000.00	2 000.0		
3 000.00	2 999.7		
5 000.0	4 999.5		
10 000.0	10 001	±(0.02 % + 1 dgt)	± 0.2
20 000.0	19 993	±(0.05 % + 1 dgt)	
30 000.0	29 985		
50 000.0	49 958	±(0.1 % + 1 dgt)	
90 000.0	89 955		

Remark : The uncertainties are for a confidence probability of not less than 95 %.

Note :

The values given in this Calibration Report only relate to the values measured at the time of the test and any uncertainties quoted will not include allowance for the equipment long term drift, variations with environment changes, vibration and shock during transportation, overloading, mis-handling, or the capability of any other laboratory to repeat the measurement. Sun Creation Engineering Limited shall not be liable for any loss or damage resulting from the use of the equipment.





Thank You

