

**Proposal for APLAC APM011– Thermocouples
Calibration Proficiency Test Program on Type R thermocouple from 0° C to 1000° C**

1. Objective of the Programme

1.1 This inter-laboratory proficiency test programme will be organised by SAC under the auspices of APLAC to evaluate the competency of the accredited laboratories in calibrating noble metal thermocouple up to 1000°C.

2. Organization and Reference Laboratory

2.1 SAC will organise this programme with the National Metrology Centre (NMC) as the collaborator .

2.2 The NMC is responsible for providing the reference value for the programme and will carry out calibrations of the transfer artifacts before, half-way through and after the programme. SAC will provide two artifacts (2 units of type R thermocouples) and will carry out the coordination work among participating laboratories while NMC will be responsible for the analysis of the comparison results and issuing programme report. The protocol of the programme is in line with the comparison protocol for APMP Regional Comparison of Type R Thermocouples (APMP-T-S1-04) [1].

2.3 Participants are divided into two loops (see Appendix A). It will be a round robin type of comparison. The reference laboratory, NMC will check the inhomogeneity of the two transfer thermocouples and calibrate them according to NMC calibration method before, half-way through and after the programme. The two thermocouples will be sent to the participating laboratories according to the circulation schedule. Each laboratory will calibrate the thermocouple from 0 to 1000°C (incremental at 100°C), using their own calibration method. The calibration results will then be sent to the NMC in the required format (see section 4, instructions to the participating laboratories).

3. Selection of Participants

3.1 Laboratories from APLAC members will be invited through their accreditation bodies to participate. Each APLAC member may nominate up to a maximum of 2 laboratories from its economy and preference will be given to laboratories which have been accredited for the thermocouple calibration.

3.2 It is estimated that there will be a total of about 45 participant laboratories.

4. Description of Artifacts

4.1 The artifacts to be calibrated are two type R Isotech Standard thermocouples (Model no. 1600) with no cold junction (0°C to 1000°C). The thermocouple wires are kept in a ceramic sheath with diameter of 7mm and length of 600mm.

4.2 Two artifacts will be used in concurrent circulation loops to shorten the time for the proficiency test.

5. Stability of Artifacts

5.1 The artifacts will be measured by the reference laboratory before, half-way through and after the circulation to check for the stability of the artifacts.

6. Circulation of Artifacts

6.1 The artifacts are stored in rigid cases designed to restrict movement and prevent damage to the artifacts. SAC will send one thermocouple in its wooden box to each participating laboratory.

7. Calibration Procedures used by NMC

7.1 The Type R thermocouples will be calibrated by using fixed point method. Ice, Sn, Zn, Al, Ag and Cu fixed points will be used for the calibrations. The calibrations will be done from low to high temperatures following NMC calibration procedures. The measured thermocouple emf, E , at the fixed point temperatures are compared with the corresponding reference value, E_{ref} , given by the defined reference function. The calibration results in terms of $E - E_{ref}$ can be fitted to a polynomial as a function of temperature, T . The thermocouple emf at any other temperatures within the measured temperature range will be then generated by using the obtained polynomial.

7.2 The inhomogeneity of the thermocouples will be tested at 230 °C in a liquid bath with immersion up to 400mm before, half-way through and after the calibrations.

7.3 A pair of Cu wire will be connected to the cold junction ends of each thermocouple in order to measure the EMF of the thermocouple. The cold junction will then be immersed in an ice bath.

8. Instruction to the Participating Laboratories

8.1 Each participating laboratory will receive one unit of type R thermocouples.

8.2 Upon receiving the thermocouple, the participating laboratory shall inspect the thermocouple for any damage and report to SAC if damage is detected. SAC will give instructions on how to proceed.

8.3 A pair of Cu- wires should be connected to the CJ ends (open ends) of the thermocouple. The thermocouples shall be measured in as received conditions by comparison method.

8.4 The thermocouple emf will be determined from nominal temperatures 0 to 1000 °C with a step of 100 °C from low to high temperatures using the existing technique as practiced by the participating laboratory. The temperature values shall be referred to the ITS-90.

8.5 The immersion depth of the thermocouple in the calibration enclosure shall be **400 mm or less** from the tip.

8.6 If a calibrated DVM is used to measure the thermocouple EMF, during calibration, the CJ ends of the thermocouple shall be immersed a minimum of 190 mm in an ice bath or other suitable medium at 0 °C.

- 8.7 A digital thermometer with ACJC could be used as well. But in this case, the measured thermocouple temperature shall be converted to emf by using the defined reference function.
- 8.8 After completion of the calibration, the participating laboratory shall transfer the thermocouple to the next participating laboratory as instructed and send the comparison results to SAC. It is the responsibility of each participating laboratory to arrange transport of the thermocouple to the next participating laboratory after the calibration is completed. It is also the responsibility of each laboratory to obtain insurance for the transport and to cover the costs of transport and insurance to the next laboratory. The instrument shall be accompanied by an ATA carnet or customs declaration document and Received/Dispatched form as given in **Appendix E**.

Note: The participating laboratory **shall not dismantle** the thermocouple.

9. Reporting of Results

- 9.1 The participating laboratory shall send to SAC the following information **within 3 weeks** of receiving the thermocouple:
- 9.2 A general outline of the calibration procedure used by the laboratory and send this as an electronic file named '**procedure.doc**'.
- 9.3 Details of instruments used in the calibration as an attached file named '**Instrument.doc**' in the format as given in **Appendix B**.
- 9.4 The values of calibration results shall be given as an Excel spreadsheet named '**Calibrationdata.xls**' in the format as given in **Appendix C**.
- 9.5 The uncertainty shall be estimated according to the 'ISO Guide to the expression of Uncertainty in Measurement' in terms of microvolt. The assigned expanded uncertainty at the level of confidence of approximately 95% shall be given in '**Uncertainty.xls**' together with the values of the considered components of uncertainties used to calculate the expanded uncertainty.

10. Analysis of Results

NMC and SAC will analyse the results and produce a report.

11. Confidentiality

Each laboratory will be assigned with a unique identification code. This unique code will be used throughout the programme. The identity and results of the participating laboratories are strictly kept confidential.

11. Program Schedule

1 Jan 08 - 31 Jan 08	Invite participation and pre-condition & calibrate artifact in reference laboratory
1 Feb 08 - 31 Jul 09	Dispatch artifacts and collect results
1 Aug 09 – 30 Sep 09	Data Analysis and issuance of interim report
1 Sep 09 – 31 Oct 09	Prepare draft report and invite comments from participants
1 Nov 09 - 30 Nov 09	Submit draft report to APLAC PT Committee for approval
1 Dec 09 – 31 Dec 09	Issue final report

12. References

1. Protocol for APMP regional comparison of Type R thermocouple from 1 to 1100 °C APMP-T-S1-04

Appendix A: Circulation Proposal

Loop No. 1 (S/N 241718/1): Countries which are geographically above Singapore

Loop No. 2 (S/N 241718/2): Countries which are geographically below and west of Singapore

Appendix B: Measuring Equipments and Standards Used in the Calibration.

Laboratory Name: _____

Devices	Type	Manufacturer	Serial number	Description	Immersion (mm)
Standard used for reference temperature					
Bridge/DVM for measurement of reference standard					
Temperature enclosure used such as bath, tube furnace, etc					
Ice-Point used					
DVM/digital thermometer for Test TC					
Scanner (if used)					

Note: Please indicate the traceability of the reference standard used.

Appendix C: Calibration Results

Laboratory Name: _____

Temperature/°C	Eref /mV	E/mV	E - Eref /mV	Un (mV) 95%
0.0				
100.0				
200.0				
300.0				
400.0				
500.0				
600.0				
700.0				
800.0				
900.0				
1000.0				

Appendix D: Customs Declaration

TO WHOME IT MAY CONCERN

APLAC APM011 Inter-Laboratory Comparison Programme

The Asia Pacific Laboratory Accreditation Cooperation (APLAC) is a Specialist Regional body under APEC Sub-Committee on Standards and Conformity Assessment. This box contains a scientific equipment for an international inter-laboratory comparison coordinated by Singapore Accreditation Council, SPRING Singapore.

One very successful method used by the APLAC is the proficiency test programme performed by different laboratories on a given artefact. Successful completion of this programme adds confidence to the laboratories and supports the APLAC Mutual Recognition Arrangement (MRA).

As part of a major proficiency test program, the APLAC is conducting a programme on the calibration of type R thermocouple from 0 to 1000°C involving the participants given in the Laboratory Schedule (Table 1).

This program is coordinated by,
Singapore Accreditation Council (SAC)
SPRING Singapore
2 Bukit Merah Central
#15-01, Tower Block
Singapore 159835

The following artefact is circulated among the participants for calibration:

A type R Thermocouple, Serial number:

The purchase/manufacturing cost of the artifact was S\$ 5560. However it has no commercial value. It is meant solely for the test and will be re-exported immediately after the test is complete (see enclosed Schedule).

We request that the device is not handled or removed from the container/package. If a Customs inspection is required then please contact the relevant person listed in the attached schedule so that he/she can be present and help you unpack it.

For further enquiries regarding this programme, please contact
Ms Lee Ham Eng
Singapore Accreditation Council
SPRING Singapore
2 Bukit Merah Central
#15-01, Tower Block
Singapore 159835
Tel : +65-6279 1860 Fax : 65 6272 1937
Email : lee_ham_eng@spring.gov.sg

Appendix E: Received/Dispatched Form

A) ARTEFACT RECEIVED

To:... *(sender / coordinator)*....

APLAC APM011 Inter-Laboratory Comparison Programme

The ...*(artifact)*.... and its ATA Carnet was received at*(name of laboratory)*....
on ... *(date)*..

The condition when it was received was *in good physical and working order

*damaged – *(explain)*

(Name of participant)

B) ARTEFACT SHIPPED

To: *(recipient / coordinator)*

APLAC APM011 Inter-Laboratory Comparison Programme

The ...*(artifact)*.... and its ATA Carnet was hand delivered to.....*(name of person)*.....
at ...*(name of laboratory)*..... on*(date)*.....

(Name of Participant)