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CSIR
NATIONAL METROLOGY LABORATORY(NML)

PROCEDURES FOR THE SADCMET PILOT COMPARISON OF LIQUID-IN-GLASS THERMOMETER CALIBRATIONS AGAINST ITS-90 OVER THE RANGE 0°C TO 50°C (SADC.T-P1)

21 November 2001

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INTRODUCTION

This SADCMET pilot comparison is being coordinated by the National Metrology Laboratory (NML), CSIR, South Africa, and extends from 0°C to 50°C. The comparison was proposed at the SADC Working Group on Thermometry meeting held 23 March 1999 in Mauritius. Invitations to participate were sent to all SADC countries. The countries that indicated their readiness to participate are listed in the attached Circulation Schedule.

Each participant will calibrate a mercury-in-glass thermometer and arrange for its transport to the next participating laboratory. The Circulation Schedule for SADC.T-P1, as at 3 January 2001, is included as an Appendix. The comparison results will be analysed by CSIR-NML.

The instructions which are given below should be followed by all participants in this comparison.

The travelling thermometer is to pass through the following sequence:
1) a visual inspection using a magnifying glass
2) rejoining, if necessary, of the mercury column
3) a rest period of 3 days after the initial inspection and possible re-joining of a broken mercury column
3) a measurement at the ice point
4) measurements at 10°C, 20°C, 30°C, 40°C and 50°C
5) a second measurement at the ice point

The travelling thermometer details are as follows:

Description: Enclosed-scale mercury-in-glass thermometer
Manufacturer: Amarell
Serial number: 73-00
Full range: -11,8°C to 52,2°C
Graduation: 0,1°C
Dimensions: 420 mm long, 8,7 mm diameter

A second thermometer of the same type is also circulated. If the above thermometer is damaged during transit, the measurements may be performed on the back-up thermometer instead.
The details of the back-up thermometer are as follows:

Description: Enclosed-scale mercury-in-glass thermometer
Manufacturer: Amarell
Serial number: 74-00
Full range: -11,8°C to 52,2°C
Graduation: 0,1°C
Dimensions: 420 mm long, 8,7 mm diameter

The thermometer artefacts are fragile, so they must be handled with care. For transport, each thermometer must be packed in one of the accompanying plastic bags and packed in the travelling case.

DETAILED INSTRUCTIONS

The SADCMET pilot comparison SADC.T-P1 is conducted with an enclosed scale mercury-in-glass thermometer provided by CSIR-NML. A second thermometer is available in case of damage to the thermometer.

Initial inspection

Upon receipt of the thermometer, the host laboratory must inspect the artefact for damage or a **separated mercury column**. If the mercury column has separated, but there is no other damage to the thermometer, it should be attempted to rejoin the column. Methods for doing this are discussed in an attached document.

Then the host must complete and forward (by fax or email) the attached **Artefact Received** form to CSIR-NML, to report the condition of the artefact.
If the thermometer is damaged or the host fails to rejoin a separated column, CSIR-NML will give instructions on how to proceed. Please describe the damage or the position of air bubbles or mercury drops on the Artefact Received form.

Recovery of thermometer

The thermometer should be allowed to **rest for 3 days** after the initial inspection and possible rejoining of a separated column, before starting measurements.

Measurements

Measurements should be recorded on the attached **Measurement Data** form.

The 0°C measurements should be made in an **ice point**. If an ice point is not available, these measurements may be made in a bath or other heat source, against a laboratory standard thermometer.

The other measurements should be made in the bath or other heat source that is usually used for liquid-in-glass thermometer calibrations, against a laboratory standard thermometer.
Readings may be taken with the bath temperature slowly increasing (a suitable rate is about 0,02°C / minute), or with the bath stabilised at the calibration temperature.

Immersion depth: **Total immersion**, i.e. immersion of the thermometer to the top of the mercury column, is preferred. If this is not possible, the thermometer may be **partially immersed**. In the case of partial immersion, the length and average temperature of the emergent liquid column (ELC) should be recorded on the Measurement Data form, and the reading corrected to total immersion according to the formula given in the "Emergent liquid column correction" section below.

[Fig. 8, p19, OIML Guide to practical temperature measurements]

The order of measurements should be: 0°C, 10°C, 20°C, 30°C, 40°C, 50°C, 0°C.
The second ice point measurement should be made immediately after the measurement at 50°C (about 30 minutes to 60 minutes after, when the thermometer has cooled to room temperature).

Emergent liquid column (ELC) correction

If the thermometer is calibrated at **partial immersion**, a correction for the temperature of the emergent liquid column (ELC) must be applied.

The average temperature of the emergent liquid column may be measured using Faden thermometer(s), thermocouple(s), platinum resistance thermometer(s) or liquid-in-glass thermometer(s). (Some methods are illustrated on the diagram below. Other methods may be used.) The method used should be recorded on the **Measurement Data** form.

[Fig. 1, p40, EA Th7 report]

ELC correction = k x n x ($t_{actual} – t_{ELC}$)

where k = differential coefficient of thermal expansion of mercury relative to glass = 0,000 160
n = length of emergent liquid column, expressed in number of degrees on thermometer scale
t_{actual} = actual temperature of the bath as measured by standard thermometer
t_{ELC} = average temperature of emergent liquid column

Reading corrected to total immersion = reading at partial immersion + ELC correction

Example: The actual temperature of the bath is 40,00°C. The reading of the thermometer is 39,00°C. The thermometer is immersed to the –5°C mark on the thermometer scale. The average temperature of the emergent liquid column is 25°C. Then,
n = 39°C – (-5°C) = 44°C
$t_{actual} – t_{ELC}$ = 40°C – 25°C = 15°C
ELC correction = 0,000160 x 44°C x 15°C = +0,11°C
Reading corrected to total immersion = 39,00°C + 0,11°C = 39,11°C

Reporting the results

The **Measurement Data** form should be completed and forwarded to CSIR-NML by fax or email before shipping the thermometers to the next laboratory. CSIR-NML will inform the host laboratory if the thermometers may be shipped, or if additional measurements are required before shipping.

A **calibration certificate** should be issued for the thermometer and posted to

Hans Liedberg
CSIR-NML
PO Box 395
Pretoria
0001
South Africa

within 3 weeks after completion of the measurements.

Uncertainty budget

The **Uncertainty Analysis** and **Instrumentation** forms should be forwarded to CSIR-NML within 3 weeks after completion of the measurements.

Below is an explanation of how to complete the attached **Uncertainty Analysis** form:

The uncertainty of **calibration of the standard thermometer** (as stated on its calibration certificate) should be recorded in the "Value" column.
The coverage factor (as stated on the calibration certificate) should be recorded in the "Divisor" column. If a level of confidence is stated instead of a coverage factor, use the following coverage factors:

68% level of confidence: coverage factor k=1
95% level of confidence: coverage factor k=2
99% level of confidence: coverage factor k=2,6
99,7% level of confidence: coverage factor k=3

The uncertainty value should be reduced to one standard uncertainty by dividing it by the coverage factor, and the uncertainty (now at a coverage factor of k=1) recorded in the "Uncertainty contribution (k=1)" column.
If the number of degrees of freedom of the uncertainty estimate is known (e.g. if stated on the calibration certificate), this should be recorded in the "Degrees of freedom" column, otherwise this cell should be left blank.

The **repeatability of the standard thermometer** is given by the standard deviation of the mean of repeated readings. The number of degrees of freedom = number of readings – 1.

E.g. Four successive readings are taken of the standard thermometer (with the bath at a stable temperature):

$x_1 = 39{,}98°C$, $x_2 = 40{,}00°C$, $x_3 = 39{,}97°C$ and $x_4 = 40{,}01°C$

Mean $\bar{x}$ = (39,98°C + 40,00°C + 39,97°C + 40,01°C) / 4 = 39,99°C

Standard deviation $= \sqrt{\dfrac{\sum_{i=1}^{n}(x_i - \bar{x})^2}{n-1}}$ = 0,018°C

Standard deviation of the mean = standard deviation / √(number of readings) = 0,018°C/√4 = 0,009°C

Number of degrees of freedom of the standard deviation of the mean = number of readings – 1 = 4 – 1 = 3

The mean of several readings follows a normal distribution, and is already at the one standard uncertainty level, so the divisor is 1.

NB: If the calibration is performed with the bath temperature slowly increasing, it will be difficult to estimate the repeatability of the standard thermometer during the calibration. In this case, the repeatability may be estimated before or after performing the calibration measurements, by taking repeated readings (preferably at least 10 readings) of the standard thermometer with the bath at a stable temperature.

The **resolution of the standard thermometer** contributes to the uncertainty because there is a range of temperatures over which the standard thermometer will give the same indication.
E.g. If the standard is a digital thermometer with a resolution of 0,01°C, then the value of this factor is 0,01°C / 2 = 0,005°C. A rectangular probability distribution is assumed, so the divisor (to reduce the value to the one standard uncertainty level) is √3. So, the value recorded in the "Uncertainty contribution (k=1)" column is 0,005°C / √3 = 0,003°C.
If the standard thermometer is a liquid-in-glass thermometer, this factor is the smallest fraction of a division that can be estimated accurately (using a telescope, this may be as small as 1/8th of a division or less).
As the resolution is known with a great deal of confidence, the number of degrees of freedom of this component is large. (For a digital thermometer, where the resolution is precisely known, the number of degrees of freedom is infinite.)

CSIR-NML will analyse the submitted uncertainty budget, and will decide whether to include the repeatability or resolution estimate in the final combined uncertainty. (Including both repeatability and resolution in the combined uncertainty may constitute “double-counting” of uncertainties.)

The **temperature gradient** between the standard thermometer and the test thermometer (i.e. the thermometer being calibrated) may be estimated from previous evaluations of the bath, or from the bath manufacturer’s specifications.
E.g. If the maximum temperature gradient between the standard and test thermometers is 0,004°C, this value should be recorded in the “Value” column. A rectangular probability distribution is assumed, so the value is divided by v3 to reduce it to the one standard uncertainty level. So, the value recorded in the “Uncertainty contribution (k=1)” column is 0,004°C / v3 = 0,002°C.
The number of degrees of freedom is given by the number of readings taken during evaluation of the temperature gradient, minus one. E.g. if the mean of 10 measurements is used to estimate the temperature gradient, then the number of degrees of freedom = number of readings – 1 = 10 – 1 = 9. If the bath manufacturer’s specification is used, a large number of degrees of freedom may be assumed, e.g. 50.

The **repeatability of the test thermometer** may be estimated using the variation in ice point readings before and after the 10°C to 50°C measurements, or using repeated readings at another temperature. As the number of repeated readings will probably not be enough to calculate an accurate standard deviation (the standard deviation of 2 readings is itself 76% uncertain!), the maximum difference between repeated readings may be used as an estimate of the width of a rectangular distribution.
E.g. If the ice point readings before and after the measurements at the other temperatures are 0,01°C and –0,02°C, respectively, then the width of the rectangular distribution is estimated as 0,03°C. The half-width is 0,03°C / 2 = 0,015°C. This value is recorded in the “Value” column. To reduce this value to the one standard uncertainty level, it is divided by v3 (the appropriate divisor for a rectangular distribution). So, the value recorded in the “Uncertainty contribution (k=1)” column is 0,015°C / v3 = 0,009°C.

The **resolution of the test thermometer** is the smallest fraction of a division that can be estimated accurately. E.g. If you can read the test thermometer to 1/4 of a division, then 0,1°C / 4 = 0,025°C is recorded in the “Value” column. To reduce this to the one standard uncertainty level, it is divided by v3, so the “Uncertainty contribution (k=1)” is 0,025°C / v3 = 0,01°C.

As for the standard thermometer, CSIR-NML will analyse the submitted uncertainty budget, and will decide whether to include the repeatability or resolution estimate in the final combined uncertainty.

The thermometer reading may be slightly affected by **changes in the external pressure** on the thin-walled bulb. The reference conditions are total immersion in water, with the atmospheric pressure of the air above the bath being 101 kPa (1 atmosphere).
If the thermometer is calibrated at partial immersion, the reading will be lower than at total immersion, owing to the lower hydrostatic pressure on the bulb. The size of this effect is about 0,001°C per 100mm that the immersion depth is reduced. If the atmospheric pressure is lower than 101 kPa, the reading will also be lower, by about 0,001°C per 1 kPa that the atmospheric pressure is lower than 101 kPa [Wise J.A., *NBS Monograph 150*, Washington, National Bureau of Standards, 1976].
The size of these pressure effects should be calculated and the values added together. The total should be recorded in the “Value” column, then reduced to the one standard uncertainty level by dividing by v3.
E.g. If the immersion at 50°C is 200mm less than total, and the atmospheric pressure is 90 kPa, the effect of reduced hydrostatic pressure is 0,002°C and that of reduced atmospheric pressure is 0,011°C. The sum is 0,013°C. The uncertainty contribution at the one standard uncertainty level is 0,013°C / v3 = 0,0075°C.

TRANSPORTATION

<u>Packaging</u>

Before shipping, each thermometer should be packed into one of the accompanying plastic bags, and carefully packed in the travelling case.

<u>Air freight</u>

The thermometers should be carried in the baggage compartment of the aeroplane (not in the passenger compartment), unless transport in the passenger compartment is permitted by local regulations for the air transport of small quantities of mercury.

The host laboratory is responsible for transport of the thermometers to the next laboratory on the Circulation Schedule. The thermometers may be transported unaccompanied or (preferably) accompanied. In either case, they should be packed as described above before transport. Local regulations regarding the air transport of mercury must be adhered to.

Circulation schedule

Please forward the **Artefact Shipped** form to CSIR-NML and to the next laboratory on the Circulation Schedule.

The circulation schedule allows 4 weeks for measurements and 3 weeks for transport between laboratories. Please try to complete measurements and send the completed Measurement Data form to CSIR-NML within the first 3 weeks, so that any additional measurements requested by CSIR-NML may be completed by the end of the 4th week.

APPENDIX A: CIRCULATION SCHEDULE FOR SADCMET REGIONAL COMPARISON SADC.T-P1

Laboratory	Contact person	Time allocated
CSIR-NML, South Africa	Mr Hans Liedberg / Mrs Brigitte Monard	
National Physical Laboratory, India	Dr Satya P. Varma	Sat 6 Jan 2001 to Mon 19 Feb 2001
CSIR-NML, South Africa	Mr Hans Liedberg / Mrs Brigitte Monard	Tue 20 Feb 2001 to Fri 2 March 2001
Mauritius Standards Bureau	Mr Christian Ng Ha Kwong	Fri 16 March 2001 to Fri 13 April 2001
CSIR-NML, South Africa	Mr Hans Liedberg / Mrs Brigitte Monard	Fri 4 May 2001 to Fri 11 May 2001
		Fri 1 June 2001 to Fri 29 June 2001
Kenya Bureau of Standards	Mr Joel M Kioko	Fri 20 July 2001 to Fri 17 August 2001
Seychelles Bureau of Standards – NPL	Ms Julia Rose / Mr Archange Sophola	Fri 7 Sept 2001 to Fri 5 Oct 2001
CSIR-NML, South Africa	Mr Hans Liedberg / Mrs Brigitte Monard	Fri 26 October 2001 to Fri 2 Nov 2001
SIRDC – NMI	Mr Victor Mundembe	Fri 23 Nov 2001 to Fri 21 Dec 2001
Botswana Bureau of Standards	Mr Keeper Morgan	Fri 11 Jan 2002 to Fri 8 Feb 2002
CSIR-NML, South Africa	Mr Hans Liedberg / Mrs Brigitte Monard	

Contact person	Organisation	Postal address	Country	Telephone	Fax	Email
Mr Hans Liedberg / Mrs Brigitte Monard	CSIR-NML	PO Box 395, Pretoria, 0001	South Africa	+27-12-841 2753	+27-12-841 2131/4458	hliedber@csir.co.za
Dr Satya P. Varma	National Physical Laboratory	New Delhi 110012	India	+91-11-578 6592	+91-11-585 2678/576 4189	spvarma@csnpl.ren.nic.in
Mr Christian Ng Ha Kwong	Mauritius Standards Bureau	Moka	Mauritius	+230-433 3648	+230-433 5051/5150	msb@intnet.mu
Mr Keeper Morgan	Botswana Bureau of Standards	P/Bag BO 0048, Gaborone	Botswana	+267-56 4044	+267-56 4042	k_morgan @bobstandards.bw
Mr Matchai Martinus Blasius	Ministry of Trade and Industry	PO Box 729, Windhoek	Namibia	+264-61-23 8694	+264-61-25 4566	
Mr Joel M Kioko	Kenya Bureau of Standards	PO Box 54974, Nairobi	Kenya	+254-2-502 543/211	+254-2-503 293	kebs@users.africaonline.co.ke
Mr Victor Mundembe	SIRDC – NMI	PO Box 6640, Harare	Zimbabwe	+263-4-86 0321/33	+263-4-86 0350/1	vmundembe@sirdc.ac.zw
Ms Julia Rose / Mr Archange Sophola	SBS – NPL	PO Box 953, Victoria, Mahe	Seychelles	+248-375 333	+248-375 151	sbsorg@seychelles.net

APPENDIX B: CUSTOMS DECLARATION

TO WHOM IT MAY CONCERN

The SADC Cooperation in Measurement Traceability (SADCMET) is an organisation representing the National Measurement / Standards Laboratories of Angola, Botswana, Democratic Republic of Congo (DRC), Lesotho, Malawi, Mauritius, Mozambique, Namibia, Seychelles, South Africa, Swaziland, Tanzania, Zambia and Zimbabwe. Its broad objective is to coordinate measurement activities and services in the SADC region, in order to provide regional calibration and testing services with readily available traceability to the SI units of measurement.

As part of an intercomparison program, SADCMET is conducting an intercomparison of realisations of the International Temperature Scale of 1990 (ITS-90) in the temperature range 0°C to 50°C involving the participants as given in the Circulation Schedule.

This intercomparison is coordinated by:
National Metrology Laboratory (NML)
Council for Scientific and Industrial Research (CSIR)
PO Box 395
Pretoria
0001
South Africa

The following artefacts are circulated among the participants for measurement:

Description: Mercury-in-glass thermometers
Manufacturer: Amarell
Serial numbers: 73-00 and 74-00

The thermometers are carried in accordance with local regulations regarding the air transport of mercury.

The purchase cost of the artefacts was ZAR 300. However, they have no commercial value (they are not for sale). They are meant solely for the comparison of national standards and will be re-exported immediately after the measurements are completed (see enclosed Circulation Schedule).

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

....

Comparison coordinator

Hans Liedberg, CSIR-NML
International phone number: +27-12-841 2753
International fax number: +27-12-841 2131 / 4458

APPENDIX C: ARTEFACT RECEIVED

TO:	CSIR-NML		
ATTN:	Hans Liedberg	DATE:	
FAX NO:	+27-12-841 2131/4458	TEL NO:	+27-12-841 2753
FROM:		NO OF PAGES:	1

SADCMET Regional Pilot Comparison SADC.T-P1

The Amarell mercury-in-glass thermometers serial numbers 73-00 and 74-00 were received at .... on (date) ....

The condition when they were received was

73-00: * in good physical and working order

* damaged (explain)

Visual inspection with magnifying glass: Air bubbles: .... Repaired? Yes No

Mercury drops: .... Repaired? Yes No

74-00: * in good physical and working order

* damaged (explain)

Visual inspection with magnifying glass: Air bubbles: .... Repaired? Yes No

Mercury drops: .... Repaired? Yes No

....

(name of participant)

APPENDIX C (cntd): ARTEFACT SHIPPED

TO:	CSIR-NML		
ATTN:	Hans Liedberg	DATE:	
FAX NO:	+27-12-841 2131/4458	TEL NO:	+27-12-841 2753
FROM:		NO OF PAGES:	1

SADCMET Regional Pilot Comparison SADC.T-P1

The Amarell mercury-in-glass thermometers serial numbers 73-00 and 74-00 were shipped to .... on (date) ....

The flight number / tracking number is ....

....

(name of participant)

APPENDIX D: MEASUREMENT DATA

TO:	CSIR-NML		
ATTN:	Hans Liedberg	DATE:	
FAX NO:	+27-12-841 2131/4458	TEL NO:	+27-12-841 2753
FROM:		NO OF PAGES:	1

SADCMET Regional Pilot Comparison SADC.T-P1

Laboratory name: ....

Metrologist: ....

Dates of measurement: .... to ....

Thermometer measured: 73-00 or 74-00 (circle whichever is applicable)

Before measurement:
Visual inspection with magnifying glass: Air bubbles: .... Repaired? Yes No

Mercury drops: .... Repaired? Yes No

Actual temperature (°C)	Thermometer reading (°C)	Immersed to ... °C mark	Average temperature of ELC (°C)	Reading corrected to total immersion (°C)

In the case of partial immersion:

ELC correction = k x n x (t_{actual} – t_{ELC})

where k = differential coefficient of thermal expansion of mercury relative to glass = 0,000 160
n = length of emergent liquid column, expressed in number of degrees on thermometer scale
t_{actual} = actual temperature of the bath, as measured by standard thermometer
t_{ELC} = average temperature of emergent liquid column

Reading corrected to total immersion = reading at partial immersion + ELC correction

Method of measuring average temperature of ELC: ....

....

....

APPENDIX E: UNCERTAINTY ANALYSIS

TO:	CSIR-NML		
ATTN:	Hans Liedberg	DATE:	
FAX NO:	+27-12-841 2131/4458	TEL NO:	+27-12-841 2753
FROM:		NO OF PAGES:	1

SADCMET Regional Pilot Comparison SADC.T-P1

Laboratory name: ....

Metrologist: ....

Source of uncertainty	Value (°C)	Probability distribution	Divisor	Uncertainty contribution (k=1)	Degrees of freedom
Calibration of standard thermometer		Normal			
Repeatability of standard thermometer		Normal	1		
Resolution of standard thermometer		Rectangular	v3		
Temperature gradient in bath		Rectangular	v3		
Repeatability of test thermometer					
Resolution of test thermometer		Rectangular	v3		
Effect of external pressure		Rectangular	v3		
Other (please specify)					

(See the Uncertainty budget section of the **Detailed Instructions** for an explanation of how to complete the uncertainty budget.)

APPENDIX F: DETAILS OF INSTRUMENTATION USED IN THE COMPARISON

TO:	CSIR-NML		
ATTN:	Hans Liedberg	DATE:	
FAX NO:	+27-12-841 2131/4458	TEL NO:	+27-12-841 2753
FROM:		NO OF PAGES:	1

SADCMET Regional Pilot Comparison SADC.T-P1

Laboratory name: ....

Metrologist: ....

Standard thermometer:

Manufacturer: ....

Model: ....

Serial number: ....

0°C: Measured in ice point or bath (circle whichever is applicable)

Type of heat source used for other temperatures (e.g. stirred water bath, dry block calibrator):

....

Manufacturer: ....

Model: ....

Measured on rising or stable temperature? Rising Stable

If rising, what was the rise rate (°C/minute)? ....

If stable, what was the peak-to-peak amplitude of the bath's control cycle (°C)? ....

Atmospheric pressure (Pa) ....

APPENDIX G: EXCERPT FROM *TECHNIQUES FOR APPROXIMATING ITS-90*

Preston-Thomas H., *Techniques for Approximating the International Temperature Scale of 1990*, Paris, Bureau International des Poids et Mesures, 1990, 205 p.

APPENDIX H: EXCERPT FROM *HANDBOOK OF TEMPERATURE MEASUREMENT*

Bentley R.E. (ed.), *Handbook of Temperature Measurement*, Vol. 2, Singapore, Springer-Verlag, 1998, 183 p.