

Technical Protocol

Key comparison APMP.EM.BIPM-K11.3: 10 V and 1.018 V DC VOLTAGE

Ver. 8.4 (Feb. 19, 2010)

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1. INTRODUCTION

At the APMP TCEM meeting, held in Jeju on 5 September 2005, KRISS proposed to organize an APMP key comparison (KC) of 10 V and 1.018 V DC voltage. At the same meeting NMIJ, Japan kindly agreed to provide Zener standards for traveling standards. The proposal was approved by the meeting. As a preparative step for the KC, a pilot comparison between KRISS and NMIJ was carried out in August to September, 2007 to test conditions of stabilization after transport and to check the uncertainty contributions due to the transport. This KC APMP.EM.BIPM-K11.3 covers comparison of both 1.018 V and 10 V which corresponds to KCs identified by BIPM.EM-K11.a and BIPM.EM-K11.b.

2. TRAVELING STANDARDS

2.1 General requirements

The traveling standard should have good stability of its output voltages during transportation. To reduce the consequences of any unexpected behavior of the traveling standards, several Zener standards are usually used [1]. Since different environmental conditions are used among participating labs, appropriate correction of measurement results against temperature, humidity and pressure is necessary. This makes it necessary for us to prepare a set of traveling standards with data on their environmental coefficients. Humidity effect of the Zener standards is known to have very slow time response [2]. In view of time schedule of comparison, the humidity effect will be treated as a drift effect when reference value is calculated by interpolation between two reference measurements as in the earlier EUROMET KC [3].

Characteristics of the standards

In Table 1 an overview is given of the temperature and pressure coefficients of the output voltages U_{measured} of the traveling standards as determined by NMIJ. The temperature effect is expressed in terms of the environmental temperature (α_T) and in terms of the oven thermistor resistance (α_R). The coefficient α_R will be used to make corrections for temperature effects (see measurement procedure).

Table 1: Temperature, humidity and pressure coefficients of 10 V and 1.018 V outputs.

(The uncertainties are stated in terms of combined standard uncertainty, 1 sigma)

Standard	Output	Reference thermistor resistance at R_0 (k Ω)	Temperature coefficient α_R (nV Ω^{-1})	Humidity coefficient α_H (nV %RH $^{-1}$)	Pressure coefficient α_p (nV hPa $^{-1}$)
TZS-1	10 V	39.65	4.3 ± 2.9	$<15 \pm 3.0$	17.8 ± 3.6
TZS-2	10 V	38.65	1.9 ± 2.9	$<15 \pm 3.0$	16.5 ± 3.5
TZS-3	10 V	39.41	1.3 ± 2.9	$<15 \pm 3.0$	21.3 ± 3.8
TZS-1	1.018 V	39.65	0.3 ± 2.9	$<1 \pm 2.9$	2.0 ± 2.9
TZS-2	1.018 V	38.65	0.2 ± 2.9	$<1 \pm 2.9$	1.4 ± 2.9
TZS-3	1.018 V	39.41	0.2 ± 2.9	$<1 \pm 2.9$	2.1 ± 2.9

The resistance of the oven temperature thermistor will be used as an indicator for the temperature of the Zener standards.

2.2 Description of standards

The traveling standards, three Fluke 732B electronic DC reference standards, have identification as follows:

TZS-1	s/n 6950001-3
TZS-2	s/n 6950002
TZS-3	s/n 6950004

The Fluke 732 B electronic DC reference standard has two output voltages, nominally 1.018 V and 10 V, respectively. Within the comparison, both the 10 V and the 1.018 V output will be measured. Each Fluke 732B electronic DC reference standard is fixed in an upgrade-box (18.0 cm x 21.0 cm x 47.0 cm) (Fig. 1). Two additional batteries are installed inside the upgrade-box. These batteries are used to increase the working time of the internal battery of the Fluke 732B. A BNC type female connector is provided for the measurement of internal thermistor resistance (see **‘Measuring the internal thermistor resistance’ in Clause 4.2**). The total weight of the upgrade box (with Fluke 732B and batteries) is around 14 kg. Each upgrade box is packed in a transportation case (27 x 27.5 x 55) cm. The two additional batteries are connected in parallel to MONITOR/EXT BAT IN connectors on rear panel of the 732B. These batteries are attached to the Fluke 732B inside of the Upgrade box. Note that the internal battery is already fixed in the original position inside of the Fluke 732B. It is possible to recharge all three batteries at the same time by the automatic charging circuit of the Fluke 732B.



Fig. 1: An upgrade-box with Fluke 732B and additional batteries

2.3 Quantities to be measured

DC voltage outputs 1.018 V and 10 V for the three standards.

2.4 Method of computation of the KCRV

Time drift of the traveling standards will be characterized using results of the Pilot Laboratory and BIPM. The difference between participant's result and the interpolated time drift will be calculated. Robust evaluation [4] using median of the difference can be used for computation of the KCRV for this comparison.

3. ORGANIZATION

3.1 Coordinator and members of the support group

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The KRISS will coordinate the comparison and act as reference laboratory.

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3.2 Participants

A participating laboratory that joins this Key Comparison (KC) is required to accept the following duties

- Prompt communication with pilot lab regarding the transport information, status of the standards and measurement report via both email and FAX.
- The transport standard should be handled carefully and be stored in a stabilized environment where relative humidity should be below 55 % R.H.
- Participating lab should fully recharge the transit battery and built-in operation battery (see 'Powering the standard' in Clause 3.5) before starting measurement.
- The sending lab is responsible for choosing an express delivery agent that provides a tracking number, with a facility for a real time web-check for the transportation status on the way to the next destination.
- The sending lab should arrange and pay the charge (incl. insurance) for the door-to-door transportation of the standard to the next scheduled lab.

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3.3 Time schedule

The comparison will be organized as Table 2.

Table 2: Time schedule

Year	Date of Measurement	Laboratory	Country or Economy
2009	8 October – 31 October	KRISS, Pilot laboratory	Korea (South)
	8 November – 30 November	NMIA	Australia
	8 December – 31 December	NMC A*STAR	Singapore
2010	8 January – 31 January	KRISS, Pilot laboratory	Korea (South)

	8 February – 28 February	NML-SIRIM	Malaysia
	8 March – 31 March	CMS	Chinese Taipei
	8 April – 30 April	KRISS, Pilot laboratory	Korea (South)
	8 May – 31 May	NMIJ (battery replacement, if necessary) NMIJ	Japan
	8 June – 30 June	NIMT	Thailand
	8 July – 31 July	BIPM	BIPM
	8 August – 31 August	SCL	Hong Kong China
	8 September – 30 September	NPLI	India
	8 October – 31 October	BIPM	BIPM
	8 November – 30 November	NMISA	South Africa
2011	8 December – 31 December	NIS	Egypt
	8 January – 31 January	BIPM	BIPM
	8 February – 29 February	VNIIM	Russia
	8 March – 31 March	KRISS, Pilot laboratory	Korea (South)

If unforeseen circumstances prevent a laboratory from carrying out the measurements within the time allocated, it should send the standards as originally scheduled without delay to the next laboratory in the schedule. Afterwards, the laboratory may be allowed to carry out the measurements before the end of the KC.

3.4 Transportation

The standards will normally be accompanied by an ATA carnet. Each participant is expected to ship using express door-to-door delivery service or to hand-carry the standard to deliver it to the next scheduled laboratory.

Because the standards should always be in the “IN CAL” state, both during transit and measurement, quick and safe transport is essential. Prompt communication with pilot laboratory should be ensured by the participating laboratory regarding the transport information and status of the standards via both email and FAX.

Every arrival and departure of the standards must be communicated to the pilot laboratory and the next scheduled laboratory using the forms that are attached in the Appendix C of this protocol.

Two or three weeks will be allowed for each participant to keep the standards in his (her) laboratory. This period includes recharging of the operation batteries, stabilization to the laboratory environment, and the measurements. **The standards must be sent to the next laboratory according to the schedule (Table 2), even if the laboratory could not finish all measurements.**

If the receiver could pick up the standards from the customs earlier than the schedule, the laboratory will be able to have more measurement days. **One week is allocated as the maximum period for the door-to-door transportation of the standards to the next participant.** Both the receiver laboratory and the sender laboratory should report promptly to the pilot laboratory about the transportation. If any delay is expected, the sender and the receiver should promptly contact the pilot laboratory that will give specific instructions.

Please be sure to fully recharge the standards before sending them.

If any participants want to hand-carry the standards by themselves, they may arrange the transportation taking responsibility of the traveling cost. In this case, the transportation information of the standards should be reported to the pilot laboratory.

3.5 Unpacking, handling, packing

The traveling standards should be handled carefully. Extreme temperature, humidity or pressure changes as well as violent mechanical shocks must be avoided.

Package

The package contains the following items:

- Fluke 732B electronic DC reference standard s/n 6950001 AIST ref. 00AB6277
- Fluke 732B electronic DC reference standard s/n 6950002 AIST ref. 00AB6278
- Fluke 732B electronic DC reference standard s/n 6950004 AIST ref. 00AB6280
- SUNJEM 9600A upgrade boxes (incl. batteries) (3x)
- Transit cases (3x) AIST ref. 00AF7410 AIST ref. 00AF7411 AIST ref. 00AF7412
- Reusable wooden box which can contain the three transit cases
- Fluke 732B instruction manual
- AC line power cord (3x)
- TR-72U datalogger for temperature and humidity s/n 00E609
- ATA carnet (732B:JY950,000.-, Carry box: JY100,000.-, TR-72U JY30,000.-, 9600A JY800,000,- cord :JY1,200.- , each)

When the package arrives at your laboratory, fill the "Receiving-the-standard form" in Appendix C and send it to pilot by both email and FAX.

When you are preparing the package for sending, fill the "Shipping-the-standard checklist form" in the Appendix C and put it in the envelope for the next lab in line.

Powering of the standard

As soon as the standards arrive at the laboratory, each Fluke 732B must be supplied from the AC power line so that the attached batteries are fully charged with the self-contained automatic charger. **Be sure to check each AC line voltage selector at the rear of the Fluke 732B before connecting the AC power cable. Be careful not to supply higher than rated voltage to the Fluke 732B!** The full recharge will take about half of the transit time. If any problems are encountered in

charging the transit batteries, this must be immediately reported to the pilot laboratory, which will give specific instructions.

After measurements on each working day, the standards must continuously receive uninterrupted voltage from the AC line power overnight or on weekend to fully recharge the standards for next day measurements. At least half of total battery operation time is required to recharge the Fluke 732B. The front panel **AC PWR** indicator lights when the standard is connected to the AC line power.

During measurements, the Fluke 732B should be disconnected from the AC line power. If the internal battery voltage drops low, the front panel **LOW BAT** indicator will start blinking. Then the standard must be plugged into the AC line power immediately to allow the battery to be recharged. The **IN CAL** indicator must be lit “on” during the whole comparison. In any case that the indicator is found to be “off”, the laboratory should report immediately to the pilot laboratory, which will give specific instructions.

In order to simplify the charging process, all the additional batteries in the ‘Upgrade box’ are permanently connected in parallel to the internal battery of the Fluke 732B, so that no other charging devices are required. By connecting the power cable to the ‘Upgrade Box’ the self-contained automatic charger of the Fluke 732B will do work of charging.

Front panel indicators

- **AC PWR**

The AC PWR indicator lights whenever the standard is connected to AC line power (e.g. 220 V, 60 Hz). **Be sure to adjust each AC line voltage selector at the rear of the Fluke 732B before connecting the AC power cable. Be careful not to supply higher than rated voltage to the Fluke 732B!**

- **IN CAL**

The IN CAL indicator goes out after excessive drops in battery operating voltage or gross changes in oven temperature.

If the IN CAL indicator doesn’t light, you must immediately contact the pilot laboratory, which will give specific instructions how to proceed.

- **CHARGE**

The CHARGE indicator lights on when the standard is connected to the AC line power and the internal battery is in the charging mode. When the battery is near full charge, the CHARGE indicator goes off.

- **LOW BAT**

The LOW BAT indicator blinks when approximately 5 hours of battery operation time remains. The standard can keep its internal oven at normal temperature for at least 7 days with the help of permanently attached three batteries.

When LOW BAT blinks, plug the Fluke 732B into the AC line power immediately to avoid extinguishing the IN CAL indicator. The battery is recharged in about half of the used time with the self-contained automatic battery charger.

3.6 Failure of the traveling standard

In case of any damage or malfunctioning of the standards, the participating laboratory must report immediately to the pilot laboratory. If the standards happen to be cooled because of a delay in

customs clearance at receiving laboratory's country, additional uncertainty for the thermal hysteresis will be imposed to the uncertainty of the standards.

3.7 Financial aspects, insurance

The sending laboratory is responsible for choosing an express delivery agent, who is capable of providing a tracking number, which will enable a real time web-check of the transportation status on the way to the next destination (door-to-door).

The sending laboratory should pay the charge for the transportation (incl. insurance: 430,000 ¥ per each Fluke 732B) of the standard to the next laboratory.

In case the prepared ATA carnet is not accepted in the participant's economy, the customs duty, if applicable, on his/her border should be paid by the participating laboratory.

4. MEASUREMENT INSTRUCTIONS

4.1 Tests before measurements

Precautions

- Do not short the outputs.
- Make sure not to disconnect the standard from the AC line power for too long.
- Avoid extreme temperature, humidity or pressure changes as well as violent impacts.

Stabilization of the standards

After arrival in the participant's laboratory, the standards should be allowed to stabilize in a temperature and, possibly, humidity controlled room for at least four days before the measurements can begin.

The traveling standard should be handled carefully and be stored in a stabilized environment where relative humidity should be below 55 %.

Powering of the standard during the measurements

When not carrying out measurements, the standards must be connected continuously to the AC line power. Measurement can be carried out after full charge, i.e., after charge indicator turns off.

Measurements should be carried out with the standard disconnected from the AC line power. To allow the standard to stabilize, measurements should not begin any sooner than 4 hours after disconnecting the standard from the AC line power. Connect the AC line after finishing the measurements to recharge the standards. (See 'LOW BAT' in **Clause 3.5**)

In addition to the battery-operated measurements, measurements can be made (and submitted to the pilot laboratory) with the standards connected to the AC line power. Notice that connection to the AC line power during measurement will probably have consequences for the connection of guard and/or ground.

4.2 Measurement Performance

Guarding

Assuming that you carry out the voltage measurements with the Fluke 732B's disconnected from the AC line power, instead of the internal GUARD binding post of the Fluke 732B, the CHASSIS (green terminal marked as "GROUND") of the upgrade box should be connected to the guard of your measuring system. At one point in your system the guard should be connected to ground.

Measuring the internal thermistor resistance

The internal thermistor resistance must be reported for each measurement result of output voltage. The thermistor resistances of the standards have nominal values between 38 k Ω and 40 k Ω (see Table 1). To avoid heating of the thermistor, the test current should **not exceed 10 μ A**. This implies that most DMMs can not be used in their 100 k Ω range or auto-range setting.

Environmental conditions

The ambient temperature, humidity and pressure must be measured. Corrections must be made for temperature and pressure effects (see next section). Recommended measurement conditions are 23 °C and below 55 %RH.

During transport and stay at the participant's laboratory, the environmental temperature and humidity will be recorded by the data-logger in transit case to check any extreme change in environment. However, please use your own measurement instruments to report more precisely the temperature, relative humidity, and atmospheric pressure during your measurement.

4.3 Method of measurement

Making corrections for temperature and pressure effects

The measured voltages U_{measured} should be corrected for temperature and pressure effects. The temperature effect is taken into account through the thermistor resistance R . The following formula should be used to calculate the corrected voltages $U_{\text{corrected}}$:

$$U_{\text{corrected}} = U_{\text{measured}} - \alpha_R \cdot (R - R_0) - \alpha_p \cdot (p - p_0),$$

where α_R and α_p are the temperature and pressure coefficients as given in Table 1, p is the ambient air pressure, and $p_0 = 1013.25$ hPa the reference air pressure. The reference thermistor resistances R_0 depend on the specific standard and are given in Table 1.

Obviously, the uncertainties of both the thermistor resistance measurement and the air pressure measurement contribute to the total uncertainty of measurement.

5. UNCERTAINTY OF MEASUREMENT

5.1 Main uncertainty components, including sources and typical values

The uncertainty calculations must comply with the requirements of the 'Guide to the Expression of Uncertainty in Measurement' (issued by the International Organization for Standardization, first edition 1993, ISBN 92-67-10188-9). Foreseen sources of uncertainty:

- Type A
- DVM or null-detector gain-error uncertainty
- Uncertainty due to irreversibility of scanner or switch
- Leakage-error uncertainty
- Uncertainty due to uncompensated offset voltages
- Microwave-frequency uncertainty
- Uncertainty due to EMI
- Calibration uncertainty of measurement equipment (e.g., for measuring the thermistor resistance, pressure, etc.)

This is not a complete list and should be extended with uncertainty contributions that are specific for the participant's measurement system.

5.2 Scheme to report the uncertainty budget

See Appendix B and Chapter 6

6. MEASUREMENT REPORT

Software

The participant's report must be sent to the pilot laboratory within two months from the completion of his measurements. Reports should be submitted electronically, using the following software:

- Word 2003 or later version for the report including the participant's results
- Excel 2003 or later version for the raw data and detailed uncertainty budget

Contents of report

The report must contain:

- **The results of the measurement**
For each reported value the following information must be provided using the form attached in Appendix:
 - identification of standard
 - method of measurement
 - date and time of measurement
 - waiting time before starting measurement after disconnect AC line from the Fluke 732B
 - measured voltage
 - thermistor resistance
 - ambient temperature, humidity, and pressure
 - values of correction for temperature and pressure effects
 - measured voltage corrected for temperature and pressure effects
 - the Type A standard uncertainty
 - the Type B standard uncertainty
 - combined standard uncertainty

- the expanded uncertainty of measurement (confidence level of appr. 95 %)
 - effective degrees of freedom
- **Uncertainty budget and calculation**
The uncertainty analysis should include a list of all sources of Type B uncertainty, together with the associated standard uncertainties as well as their evaluation method. For clarity, it is recommended to present the uncertainty budget in the form of a table (see, e.g., chapter 4 of the EA-4/02 document ‘Expression of the Uncertainty of Measurement in Calibration’). For each reported value, the expanded uncertainty of measurement and the coverage factor k must be given for confidence level of approximate 95 %.
 - **Description of the method of measurement**
This includes information on:
 - the method applied for correction of offset voltages (manual or automatic switching, reversal of null-detector or not, etc.)
 - the method applied for guarding and shielding, and connection to earth
 - method applied for biasing the Josephson array (bias on or off during measurement)
 - method for Josephson step number adjustment and maximum value of null voltage
 - ‘bandwidth’ of the voltage measurement (null-detector analog or digital filtering, number of samples, averaging, etc.)
 - **A statement of traceability**
This is only required if the national standard is not considered to be a primary standard.

7. REPORT OF THE COMPARISON

The draft version of the final report will be issued within four months after completion of the comparison. The draft report will be sent to the participants and will be discussed. The whole procedure will be based on the CCEM Guidelines document WGLF/2007-12.

REFERENCES

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- [4] J.W. Mueller, “Possible Advantages of a Robust Evaluation of Comparisons,” J. Res. Natl. Inst. Stand. Technol. **105**, 551, 2000.
- [5] EA-4/02 “Expression of the Uncertainty of Measurement in Calibration”.

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APPENDIX B: Forms for Summary Report

K11.3.a (1.018 V)

Identification of standard	TZS1	TZS2	TZS3
Method of measurement			
Date and time of measurement (from to)			
Measured voltage (V)			
Thermistor resistance (ohm)/ Ambient temperature (°C)			
Humidity (% R.H.)/ Pressure (hPa)			
Corrected voltage at R_0 and p_0 (V)			
Number of measurements			
Type A standard uncertainty (nV)			
Type B standard uncertainty (nV)			
Combined standard uncertainty (nV)			
Expanded uncertainty (nV)			
Coverage factor k			
Effective degrees of freedom			

K11.3.b (10 V)

Identification of standard	TZS1	TZS2	TZS3
Method of measurement			
Date and time of measurement (from to)			
Measured voltage (V)			
Thermistor resistance (ohm)/ Ambient temperature (°C)			
Humidity (% R.H.)/ Pressure (hPa)			
Corrected voltage at R_0 and p_0 (V)			
Number of measurements			
Type A standard uncertainty (nV)			
Type B standard uncertainty (nV)			
Combined standard uncertainty (nV)			
Expanded uncertainty (nV)			
Coverage factor k			
Effective degrees of freedom			

APPENDIX C: Forms for Transportation Report

(See next pages)

APMP.EM.BIPM-K11.3 COMPARISON

Shipping-the-standard form No 1

(Send this form to the pilot as soon as you have shipped the standard)

Date **Pages**.....(including this one)

TO

Dr. Kyu-Tae KIM KRISS, Daejeon, KOREA FAX: + 82 42 8685018, E-mail: ktkim@kriss.re.kr

FROM

--

Comments on the behavior of the standard:

The standard has been shipped to the address:

Shipped on: *Date*..... *Time*

Means of transport: *Airplane* *Other*

Carrier:

Comments on shipment (include tracking number):

APMP.EM.BIPM-K11.3 COMPARISON

Shipping-the-standard form No 2

(Send this form to both the pilot and the lab next in line, as soon as you have shipped the standard)

Date **Pages**.....(including this one)

TO

FROM

Comments on the behavior of the standard:

The standard has been shipped to the address:

Shipped on: Date..... *Time*

Means of transport: Airplane *Other*

Carrier:

Comments on shipment (include tracking number):

APMP.EM.BIPM-K11.3 COMPARISON

fax

Receiving-the-standard form

(Send this form to the pilot laboratory as soon as you receive the standard)

Date Pages.....(including this one)

TO

Kyu-Tae Kim KRISS, Daejon, KOREA FAX: +82 42 868 5018
--

FROM

--

Arrival at the lab: *Date* *Time*.....

<i>IN CAL lamp</i>	<i>ON</i> <input type="checkbox"/>	<i>Off</i> <input type="checkbox"/>
<i>LOW BAT lamp</i>	<i>Blinks</i> <input type="checkbox"/>	<i>Off</i> <input type="checkbox"/>

<i>Was the TR-72U datalogger for temperature and humidity working well?</i>	<i>Yes</i> <input type="checkbox"/>	<i>No</i> <input type="checkbox"/>
---	-------------------------------------	------------------------------------

<i>Was the package damaged?</i>	<i>Yes</i> <input type="checkbox"/>	<i>No</i> <input type="checkbox"/>
<i>Comments:</i>		

<i>Was the standards damaged?</i>	<i>Yes</i> <input type="checkbox"/>	<i>No</i> <input type="checkbox"/>
<i>Comments:</i>		

<i>Was all the material available, following the receiving checklist?</i>	<i>Yes</i> <input type="checkbox"/>	<i>No</i> <input type="checkbox"/>
---	-------------------------------------	------------------------------------

Comments:

APMP.EM.BIPM-K11.3 COMPARISON

Shipping-the-standard checklist form.

(While you are making the package ready, check that all material is included)

Are these items in the package?

YES NO

Three Fluke 732B's with upgrade box

Digital thermometer YES NO

Digital hygrometer YES NO

Fluke732B instruction manual YES NO

ATA Carnet YES NO

Sealed envelopes for laboratories next in line in your circulation loop YES NO

Recharge of the batteries:

Did you fully recharge the operation batteries? YES NO

Please, when the package is ready, seal it in the most convenient way for you in order to prevent unauthorised access to the instrument. Refer to the pilot laboratory co-ordinator if you need further information.

Checked by _____

Date _____