Available online www.jsaer.com

Journal of Scientific and Engineering Research, 2018, 5(2):240-243

Research Article

ISSN: 2394-2630
CODEN(USA): JSERBR

Validation of Hygrometers Non-Standard Calibration Method

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Abstract As part of the world's attention precisely measuring, accurate, reliable and comparable measurement results are of vital importance. Therefore, the standards which regulate laboratory requirements (ISO/IEC 17025, ISO 15189) demand a metrological approach to the measurement processes, requiring method validation. This paper study the validation of hygrometers non-standard calibration method using saturated salt solution cells. The study was done using the potassium acetate cell (22.5%), potassium iodide cell (68.9%) and potassium sulphate (97.3%) at 25°C to cover low, medium and high relative humidity. These cells were prepared [1] and measured at National Institute for Standard (NIS-Egypt) to calibrate the hygrometers with probe. The study was carried out using one shape and volume half liter spherical per each cell which was prepared. The artifact which used in calibration system was digital hygrometer in base of capacitive polymer sensor. The artifact was calibrated using three measurement methods. **First**, using saturated salt solution cells which is under test. **Second**, humidity chamber with standard reference hygrometer which is valid by validation report and reviewed by accreditation provider. **Third**, using two-pressure humidity generator with standard reference hygrometer which is valid by validation report and reviewed by accreditation provider.
After analysis the results using E_n value [2], the comparison results were shown that the calibration method using saturated salt solution cells is valid to use.

Keywords validation method; saturated salt solution, E_n value, hygrometers

1. Introduction

Validation method is often based on the combined use of validation procedures [3]. The validation used can be "direct" or comparative. The selection of the validation procedures should also be justified on a cost-benefit basis as long as the fitness for purpose is maintained. Focusing the effort on the most critical factors affecting the test method will lead to a different solution for the validation of "exact" physical and chemical test methods as compared to that for product or subjective testing. The frequency of use of the test method should also be considered when determining the extent of validation.

2. Choices of Validation Method

There are many choices to valid the internal calibration method, the most of them are:

- Participate in proficiency test program
- Intercomparison with other labs
- Internal experimental work. First, fixe artifact, standard equipment, environmental condition and method, just change the stuff. Second, change the standard equipment. Third, change the method.

In this paper, the validation of method was used last method which is change the method

3. Validation procedure

Both calibration laboratories and accreditation bodies are looking for procedures and guidelines for planning and controlling the test method validation process.

However, the discussion above has clearly indicated that one single procedure cannot be developed. Consequently, a palette of different choices of validation techniques has to be developed. How detailed the validation will be, depends on the circumstances (needs, costs, possibilities, risks, etc.).

The validation of the test methods is, of course, of interest also to the accreditation bodies. The principle to be applied should be that the laboratory describes the way it is validating the calibration methods and the accreditation body should make the judgment if the procedure used is acceptable in that case. The techniques used for method validation can be one of, or a combination of, the following :

a) Calibration and/or evaluation of bias and precision using reference standards or reference materials ;

b) Systematic assessment of the factors influencing the result ;

c) Calibration method robustness through variation of controlled parameters such as incubator temperature, volume dispensed, et;

d) Comparison of results achieved with other validated methods ;

e) Interlaboratory comparisons ;

f) Evaluation of measurement uncertainty of the results based on scientific understanding of the theoretical principles of the method and practical experience .

The laboratory shall record the following as evidence of validation :

a) The validation procedure used ;

b) Specification of the requirements ;

c) Determination of the performance characteristics of the methods ;

d) Results obtained ;

e) Verification that the requirements can be fulfilled by using the method; and

f) A statement on the validity of the method, detailing its fitness for the intended use.

In this paper, the validation was done by comparison of results achieved with other validated methods. The validation was used three methods.

4. Apparatus and Method

4.1. First method using saturated salt solution

4.1.1. Apparatus

The apparatus were used in the validation method are:

1- Three saturated salt solutions cells, potassium acetate cell (22.5%), potassium iodide cell (68.9%) and potassium sulphate (97.3%).
2- Water bath as stable medium at 25^{o}C.
3- Digital hygrometer under calibration

4.1.2. Procedure

1- Place the hygrometer under calibration inside the potassium acetate cell (22.5%)
2- Adjust the water bath at 25^{o}C.
3- Wait until the humidity/temperature is stable.
4- Take 10 readings and record the time of taking the reading in the datasheet.
5- Change the saturated salt solution cell by potassium iodide cell (68.9%).
6- Repeat clauses no. 2 to 4
7- Change the saturated salt solution cell by potassium sulphate (97.3%)
8- Repeat clauses no. 2 to 4

4.2. Second method using humidity chamber and reference hygrometer

4.2.1 Apparatus

1- Humidity chamber
2- Standard reference hygrometer

3- Digital hygrometer under calibration

4.2.2 Procedure

1- Place the hygrometer under calibration inside the climatic chamber together with the reference hygrometer probe.
2- Set the humidity and temperature of chamber to the 22.5 % and 25°C
3- Wait until the humidity/temperature inside the climatic chamber is stable. It possible to determine the stability by observing the indication of the reference hygrometer probe then measurements can be taken.
4- Take 10 readings and record the time of taking the reading in the datasheet.
5- Readjust the humidity and temperature of the climatic chamber for 68.9 % and 25°C.
6- Repeat clause no. from 3 to 4
7- Readjust the humidity and temperature of the climatic chamber for 97.3 % and 25°C.
8- Repeat clause no. from 3 to 4

4.3. Third method using humidity generator

4.3.1. Apparatus

1- Humidity generator
2- Standard reference hygrometer
3- Digital hygrometer under calibration

4.3.2. Procedure

1- Place the hygrometer under calibration inside the humidity generator together with the reference hygrometer probe.
2- Set the humidity and temperature of humidity generator to the 22.5 % and 25°C
3- Wait until the humidity/temperature inside the humidity generator is stable. It possible to determine the stability by observing the indication of the reference hygrometer probe then measurements can be taken.
4- Take 10 readings and record the time of taking the reading in the datasheet.
5- Readjust the humidity and temperature of the humidity generator for 68.9 % and 25°C.
6- Repeat clause no. from 3 to 4
7- Readjust the humidity and temperature of the humidity generator for 97.3 % and 25°C.
8- Repeat clause no. from 3 to 4

5. Results and Analysis

$$En = \frac{x - X}{\sqrt{U_{lab}^2 - U_{ref}^2}}$$

where

X is the assigned value determined in a reference laboratory;
U_{ref} is the expanded uncertainty of X;
U_{lab} is the expanded uncertainty of a participant's result x [4] [5].

Table 1: Results of calibration using saturated salt solution cells

Value of saturated salt solution RHs %	**Reading of digital hygrometer under calibration RHm %**	**Correction ΔRH= RHs-RHm %**	**Measurement Uncertainty St ± %**	**Measurement Uncertainty m ± %**	**E_n Value**
22.5	22.1	0.4	1.2	1.7	0.24
68.9	68.2	0.7	1.2	1.8	0.52
97.3	96.1	1.2	1.2	2.1	0.69

Table 2: Results of calibration using humidity chamber

Value of Standard reference hygrometer RHs %	Reading of digital hygrometer under calibration RHm %	Correction ΔRH= RHs-RHm %	Measurement Uncertainty St ± %	Measurement Uncertainty m ± %	E_n Value
22.5	22.3	0.2	0.9	1.3	0.25
68.9	68.5	0.4	0.9	1.3	0.43
97.3	96.5	0.8	0.9	1.5	0.67

Table 3: Results of calibration using humidity generator

Value of Standard reference hygrometer RHs %	Reading of digital hygrometer under calibration RHm %	Correction ΔRH= RHs-RHm %	Measurement Uncertainty St ± %	Measurement Uncertainty m ± %	E_n Value
22.5	22.2	0.3	0.5	0.7	0.61
68.9	68.4	0.5	0.5	0.8	0.80
97.3	96.5	1.0	0.5	1.0	0.92

When E_n value is equal or less than one, so the in-house method is pass. If E_n value is bigger than one, so the in-house method is fail and must to analysis the wrong and take corrective action

6. Conclusion

Measurement uncertainty is a property of measurement result, not of the method, i.e. of the testing/measurement process. Assuming that the testing/measurement process is the chief source of uncertainty, it is possible to estimate measurement uncertainty on the grounds of its performance characteristics. The first information thereof is obtained through validation experiments. Afterwards, it is essential to monitor their performance characteristics in order to prove the validity of assessment. Should it be noticed that the performance characteristics have changed, it is necessary to update the assessment with new data.

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