Report
on
Comparison Measurement

Phosphate buffer

2010 / 2011

1 Objectives

The comparison measurement was carried out in preparation of application for accreditation / CMC entries for the pH calibration laboratory of SNM INDECOPI.

2 Participants

Pilot laboratory
DKD-K-06901
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3 Time schedule

Start of comparison measurement:	October 2010
Submission of results by participant:	March 2011
Evaluation of results:	April 2011

4 Calibration object

Measuring object:	pH buffer solution / Phosphate
Nominal value:	pH 7
Prepared by:	ZMK -ANALYTIK- GmbH
Lot No.:	F1589786936 // A0135473017
Bottle No. (Participant)	13 and 14
Date of bottling:	15th October 2010
Date of calibration (by ZMK):	18th October 2010
Measuring temperature:	25℃

Homogeneity of the batch was determined before the comparison measurement was started.
SNM INDECOPI received 2 bottles of 250 ml (bottle no. 13 and 14).

5 Applied calibration methods

5.1 DKD-K-06901 / ZMK -ANALYTIK- GmbH

For the determination of the reference value the ***Differential potentiometry*** was used by the pilot laboratory.
The measuring cell is constructed according to Baucke [1].
The electrode system consists of two platinum electrodes that are surrounded by hydrogen.
The measuring cell is placed in a thermostatic bath with known stability and homogeneity.
A certified reference buffer solution was used as standard (Lot. No. 81680/73050, Batch No. 32, $U(k=2)=0.003$).

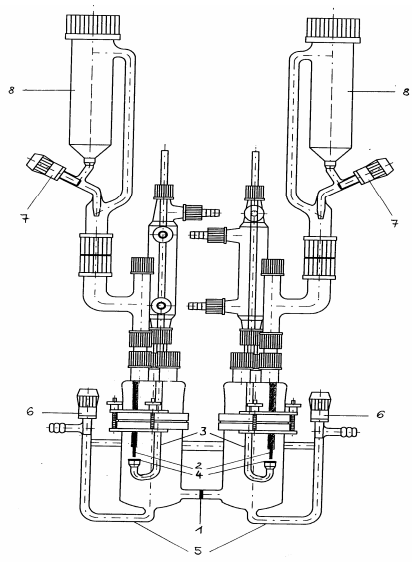

Fig. 1 Baucke cell design

[1] F.G.K. Baucke, J. Electroanal. Chem. 368 (1994), 67-75

5.2 SNM INDECOPI

The participant took part in the comparison measurement with two calibration methods.

Differential potentiometry

The measuring cell is constructed according to Baucke [1].
The electrode system consists of two platinum electrodes that are surrounded by hydrogen.
The measuring cell is placed in a thermostatic bath with known stability and homogeneity.
The potential difference was measured with a precision multimeter (Agilent 34420A).

Fig. 2 Baucke cell of SNM INDECOPI

A certified reference material with an uncertainty of $U(k=2)=0.003$ was used as standard:

Phosphate Lot. No. 73050/81680 certificate No.: 10-0241/DKD-K-06901/09-02

Multipoint calibration by glass electrode system

The calibration was carried out using a glass electrode measuring system.

Glass electrode:	Type:	Inlab Science
	Manufacturer:	Mettler Toledo
pH Meter:	Type:	PP50
	Manufacturer:	Sartorius
	Resolution:	0.1 mV

The following five certified reference materials were used as standards:

Tetraoxalate	Lot. No. 41908152	certificate No.: 10-0239/DKD-K-06901/09-02
Phthalate	Lot. No. A915674743	certificate No.: 10-0240/DKD-K-06901/09-02
Phosphate	Lot. No. 73050/81680	certificate No.: 10-0241/DKD-K-06901/09-02
Borate	Lot. No. 71840	certificate No.: 10-0242/DKD-K-06901/09-02
Carbonate	Lot. No. DMR-324-Ia / DMR-324-IIa	certificate No.: CNM-MR-630-0459/2009

The EMF of each reference solution and the sample was measured.
Using the calibration results a regression line and its parameters were determined.
These parameters were used to calculate the pH value of the unknown sample.
The calibration was carried out in a water bath with known stability and homogeneity.

6 Measuring results

The calibration results were reported in evaluation excel-sheets stating all raw data, calculation results and measuring uncertainties. The results of the comparison measurement are summarized in table 1.

In order to compare the results the value of E_n was calculated (see equation 1).

$$E_n = \frac{\left| x_{lab} - x_{ref} \right|}{\sqrt{U(x_{lab})^2 + U(x_{ref})^2}} \qquad (1)$$

This value represents the deviation between the measuring results x_{lab} and x_{ref} of the participating laboratory and the pilot laboratory under consideration of the measuring uncertainty. For acceptable measurements the value of E_n must be less than 1.

Table 1: comparison measurement results

Measuring temperature	ZMK (DKD-K-06901)			SNM INDECOPI			E_n
	Calibration method	Reference pH value x_{ref}	exp. uncertainty U_{ref}	Calibration method	pH value x_{lab}	exp. uncertainty U_{lab}	
25 ℃	Differential potentiometry	6.866	0.003	Differential potentiometry	6.865	0.003	0.24
25℃	Differential potentiometry	6,866	0.003	Multipoint calibration	6,87	0.02	0.20

7 Summary

The determined E_n value for the comparison measurement on pH is less than 0.3.

The comparison measurement was passed successfully by the participant SNM INDECOPI.

Bitterfeld-Wolfen, 29th April 2011

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