

# **Report of key comparison CCQM – K18**

## **pH of carbonate buffer**

### **Final report**

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## Summary

The key comparison CCQM-K18 was started in order to evaluate the equivalence of metrology institutes as a follow-up of the previous study CCQM-P52. As a sample a carbonate buffer solution was used with a composition slightly different to the usual one.

There were 13 institutes participating in the comparison using primary method for pH measurement. The fair agreement between the results reflects increased difficulty in measurement, where the buffer composition may slightly change during the measurement and extrapolation to start time of measurement is usually necessary.

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## Introduction

The key comparison CCQM-K18 was carried out to demonstrate the capability of the interested National Metrology Institutes to measure the pH value of an unknown carbonate buffer by a primary method. The buffer of nominal pH ~ 10 was measured at 25 °C. The comparison was an activity of the Electrochemical Working Group (EAWG) of the CCQM and was coordinated by SMU (Slovakia).

All participants applied the primary method for pH. The result for the unknown buffer solution is the acidity function at zero chloride molality  $p_a^0$ .

### Metrology Area

Amount of Substance

### Branch

Electrochemistry

### Subject

Determination of the acidity functions at zero chloride molality of an unknown carbonate buffer, pH ~10 by Harned cell measurements at 25 °C.

### Time schedule

|                                     |                   |
|-------------------------------------|-------------------|
| Dispatch of the samples:            | August 17, 2006   |
| Deadline for receipt of the report: | October 5, 2006   |
| Draft A                             | October 10, 2006  |
| Draft B                             | December 12, 2006 |
| Final report                        | April 2007        |

## Participants

Table 1 *Table of participants*

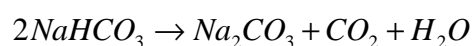
| Acronym | Participant   | Country        | Analyst   |
|---------|---|----------------|---|
| NMIJ    | National Metrology Institute of Japan   | Japan          | Susumu Nakamura; Igor Maksimov; Masaki Oohata                           |
| CENAM   | Centro Nacional de Metrología   | Mexico         | Marcela Monroy; Adrian Reyes  |
| CMI     | Český metrologický institut   | Czech Republic | Alena Vospelova   |
| GUM     | Główny Urząd Miar   | Poland         | Agata Mateuszuk; Monika Pawlina; Wladyslaw Kozlowski                    |
| INMETRO | Instituto Nacional de Metrologia, Normalização e Qualidade Industrial   | Brasil         | Paulo P. Borges; Isabel C. S. Fraga; Bianca S. R. Marques; Júlio C.Dias |
| INPL    | The National Physical Laboratory of Israel  | ISRAEL         | Elena Kardash   |
| LNE     | Laboratoire National d'Essais   | France         | Rachel Champion   |
| NCM     | National Center of Metrology  | Bulgaria       | Ludmila Dimitrova   |
| NIM     | National Institute of Metrology, Division of Metrology in Chemistry   | China          | Hongyu Xiu  |
| NIST    | National Institute of Standards and Technology  | U.S.A.         | Kenneth W. Pratt  |
| PTB     | Physikalisch-Technische Bundesanstalt   | Germany        | Janine Giera; Petra Spitzer   |
| SMU     | Slovenský metrologický ústav  | Slovakia       | Anna Mathiasova; Leos Vyskocil  |
| UMTS    | All-Ukrainian State Research and production Center of Standardization Metrology, Certification and Consumers' Rights Protection | Ukraine        | Sergey Nagibin; Vladimir Gavrilkin                                      |

## Sample description

### Sample preparation

The carbonate buffer solution was prepared from deionised water and NaHCO<sub>3</sub> (MERCK) as the starting material. The buffer composition differed from that of the standard buffer solution (pH~10.012).

About 200 g of sodium hydrogen carbonate was dried in a desiccator over silica gel at room temperature. A second portion (about 300 g) was ignited 5 h in a furnace at 300°C with intermittent mixing, to convert it to sodium carbonate:



After cooling in a desiccator about 109.210 g  $\text{NaHCO}_3$  was dissolved in 1500 mL hot deionised water. After dissolution, 165.551g  $\text{Na}_2\text{CO}_3$  was added and the solution was stirred until the dissolution was complete. The cooled down solution was quantitatively transferred into a 60 L HDPE bottle filled with about 45 L of deionised water.

Water was added slowly to reach the target balance reading, and then the solution was stirred 5 h with a vibration stirrer. Cleaned and dry 1 L HDPE bottles were filled with the prepared solution. The filled bottles were closed using a special key and after weighing they were sealed in a Mylar type foil to prevent access of  $\text{CO}_2$ .

The mass fraction of water in the solution was  $w = 0.995424$  g/g.

### Solution homogeneity

Interwoven between the 1 L bottles (after each 20 bottles), several 250 mL bottles were filled for homogeneity check (trend analysis). The solution in 250 mL bottles was measured immediately after filling using a glass electrode. The results are given in Table 2.

Table 2 *Results of homogeneity measurement*

| Sample  | pH     |
|---------|--------|
| Bottle1 | 10.095 |
| Bottle2 | 10.095 |
| Bottle3 | 10.095 |
| Bottle4 | 10.095 |

No observable difference between the bottles was seen, so it can be concluded that no systematic change occurred during the filling process and the solution can be considered homogeneous.

### Solution stability

Several bottles were set aside to measure solution stability. The value of acidity function was determined by primary measurement throughout the expected timescale of the comparison. From previous measurements it was known that the pH decreased if the bottles were not sealed in the protective foil. The present results confirmed that sealing the bottles in aluminised foil is effective method to minimise  $\text{CO}_2$  diffusion into the alkaline solution. The results are given in Table 3.

Table 3 Results of the measurement of buffer stability

| Bottle number | Date of measurement | Acidity function | Days elapsed |
|---------------|---------------------|------------------|--------------|
| 37            | 23-VIII-06          | 10.2003          | 3            |
| 38            | 25-VIII-06          | 10.1989          | 5            |
| 42            | 28-IX-06            | 10.2000          | 39           |
| 43            | 29-IX-06            | 10.1995          | 40           |
| 44            | 02-X-06             | 10.2008          | 43           |
| 45            | 04-X-06             | 10.1998          | 45           |
| 47            | 05-X-06             | 10.1978          | 46           |
| 48            | 03-XI-06            | 10.1999          | 75           |

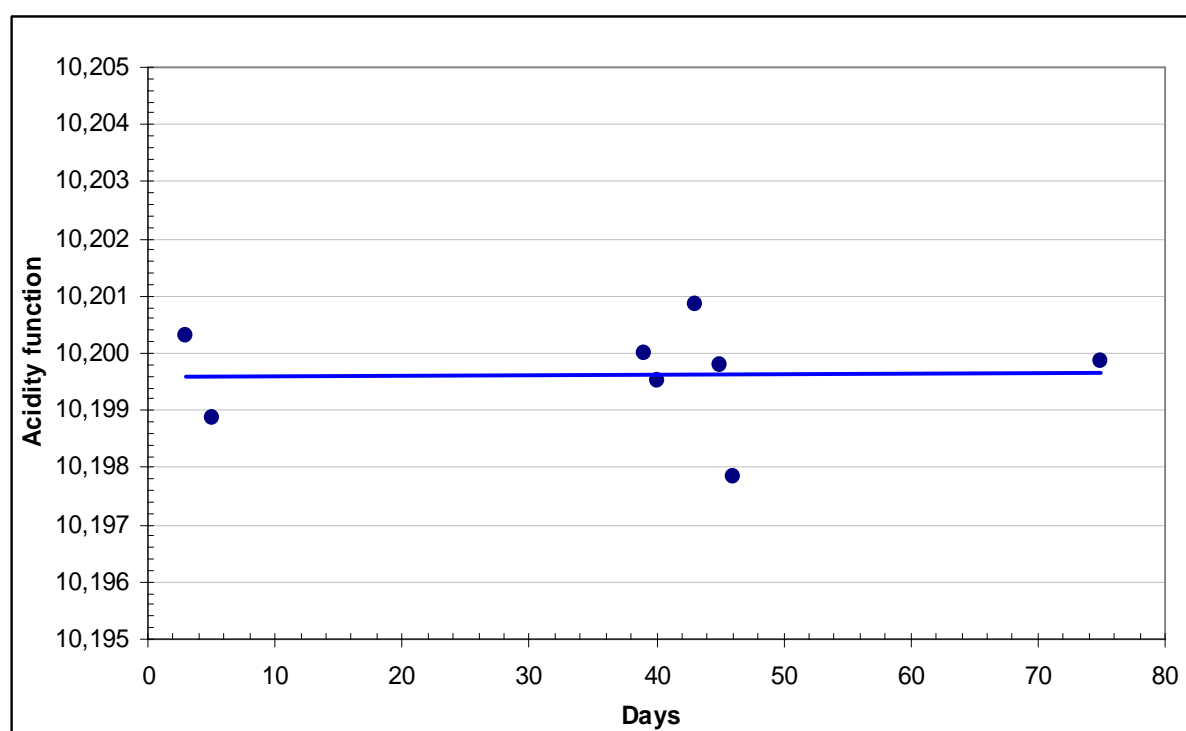


Figure 1 Results of stability measurement

Statistical test did not reveal a significant trend of the values, so the solutions can be regarded as stable for the period tested.

### Sample delivery

Each participant received three 1 L HDPE numbered bottles, sealed into foil bags to eliminate the influence of the carbon dioxide from ambient air. Shipment to participants was done by DHL. The bottles were shipped in a cardboard box by courier and the consignment number was reported by email to the contact person of the receiving laboratory for tracking purposes. The dates of receipt of the samples are given in Table 4.

### Check of the bottle integrity

Participants were requested to weigh the received bottles to verify that they were unchanged during the transport. No significant mass deviation above 0.5 g was observed compared to the bottle mass reported by SMU. No leaks were observed.

## Results and discussion

The dates of measurements and dates when the reports were sent are given in Table 4. After receipt of the results, INPL was requested to check the calculation for numerical errors. No numerical errors were found. Two laboratories requested the coordinating laboratory to postpone the deadline to 9 October due to public holidays and illness/evaluation problems. This was accepted.

Table 4 *Date of sample receipt, measurement period, and report date*

| Laboratory | Sample received | Measurement period | Report date |
|------------|-----------------|--------------------|-------------|
| NIM        | 22.08.06        | 26.08.06-26.09.06  | 9.10.2006   |
| LNE        | 18.08.06        | 24.08.06           | 26.9.2006   |
| NCM        | 18.08.06        | 20.09.06-28.09.06  | 4.10.2006   |
| NIST       | 24.08.06        | 11.09.06-14.09.06  | 27.9.2006   |
| GUM        | 18.08.06        | 21.09.06-29.09.06  | 5.10.2006   |
| UMTS       | 4.09.06         | 29.09.06-30.09.06  | 5.10.2006   |
| SMU        | 10.08.06        | 23.08.06           | 23.8.2006   |
| CMI        | 23.08.06        | 23.09.06-5.10.06   | 8.10.2006   |
| NMIJ       | 19.08.06        | 23.08.06-28.08.06  | 5.10.2006   |
| INMETRO    | 31.08.06        | 3.10.06            | 5.10.2006   |
| CENAM      | 24.08.06        | 18.09.06-29.09.06  | 5.10.2006   |
| PTB        | 21.08.06        | 29.08.06-11.09.06  | 19.9.2006   |
| INPL       | 22.08.06        | 09.06              | 5.10.2006   |

### Measurement of acidity function

Participants were requested to use only the primary method for acidity function measurement. The standard potential of the Ag/AgCl electrodes had to be measured using HCl of known molality very close to 0.01 mol/kg. The acidity function measurement is carried out by measuring the potential difference of the electrochemical cell without junction (1) (so called Harned cell) at several chloride molalities necessary in order to stabilize the potential of the silver-silver chloride electrode.





The voltage  $U^*$  corrected to standard pressure is calculated according to the equation:

$$U^* = U - \frac{\mathbf{RT} \ln 10}{2\mathbf{F}} \log\left(\frac{p - p_w}{p_0}\right) \quad (2)$$

The voltage  $U^*$  of cell (1) (corrected to hydrogen pressure of  $p_0 = 101.325$  kPa) depends on the hydrogen ion activity<sup>1</sup>  $a_{H^+}$  according to the equation:

$$U^* = \mathbf{E}^0 - \frac{\mathbf{RT} \cdot \ln(10)}{\mathbf{F}} \log(a_{H^+} \cdot b_{Cl^-} \cdot \gamma_{Cl^-}) \quad (3)$$

Where:

- $U$  voltage of cell, measured at laboratory pressure
- $U^*$  voltage of cell, corrected to standard pressure  $p_0 = 101.325$  kPa
- $\mathbf{E}^0$  standard potential of silver, silver chloride electrode
- $T$  absolute temperature in K
- $a_{H^+}$  activity hydrogen ions
- $b_{Cl^-}$  molality of chloride ions in mol/kg
- $\gamma_{Cl^-}$  activity coefficient of chloride ions
- $p$  barometric pressure
- $p_w$  pressure of saturated vapour of water

Equation (3) can be rearranged to give the acidity function so that there are only measurable quantities on the right hand side of equation (4), so acidity function is measured as a function of  $b_{Cl^-}$ .

$$-\log(a_{H^+} \cdot \gamma_{Cl^-}) = \frac{(U^* - \mathbf{E}_{Ag/AgCl}^0) \cdot \mathbf{F}}{\mathbf{RT} \ln(10)} + \log(b_{Cl^-}) \quad (4)$$

All terms on the right-hand side are known or experimentally accessible. The value of the left-hand side is plotted against the chloride ion molality in the graph. The intercept on the y-axis represents the acidity function value determined ( $\text{AF}^\circ$ ).

In the measurement of carbonate buffer some  $\text{CO}_2$  is eliminated from the buffer by the hydrogen stream, thus modifying pH of the buffer. Therefore the Harned cell voltage will not reach a stable value during the measurement and an extrapolation to the start time of measurement is usually necessary. The voltage drift is almost not detectable only at lowest flow-rates of hydrogen, at the expense of very long stabilisation times.

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<sup>1</sup> All the activities are relative to activity in standard state

All the measurements were performed only at one temperature, 25°C, due to logistical problems with preparation and distribution of more sample bottles. The results are given in Table 5 and Figure 2.

Table 5 *Results of key comparison CCQM-K18*

| Institute | Country | AF°     | u <sub>AF</sub> (k=1) |
|-----------|---------|---------|-----------------------|
| NIM       | CN      | 10.1815 | 0.0018                |
| LNE       | FR      | 10.1852 | 0.0036                |
| NCM       | BG      | 10.1945 | 0.0037                |
| NIST      | US      | 10.1969 | 0.0008                |
| GUM       | PL      | 10.1975 | 0.0017                |
| UMTS      | UA      | 10.1975 | 0.0030                |
| SMU       | SK      | 10.1995 | 0.0015                |
| CMI       | CZ      | 10.2003 | 0.0012                |
| NMIJ      | JP      | 10.2014 | 0.0013                |
| INMETRO   | BR      | 10.2051 | 0.0032                |
| CENAM     | MX      | 10.2102 | 0.0011                |
| PTB       | DE      | 10.2107 | 0.0015                |
| INPL      | IL      | 10.2648 | 0.0049                |

There are several possibilities for determination of the reference value, listed in Table 6, but the differences are insignificant. The result of INPL is omitted from the calculation (see below). The median was agreed as KCRV. Uncertainty of median was calculated according to the following equation:

$$u_{KCRV} = \frac{1,858}{\sqrt{n-1}} \text{median}\left(|AF_i^0 - \tilde{x}|\right) \quad (4)$$

Table 6 *Parameters describing the results' distribution (without INPL result):*

| Estimator       | Value          | u(k=1)        |
|-----------------|----------------|---------------|
| <b>Median</b>   | <b>10.1985</b> | <b>0.0019</b> |
| Arithmetic Mean | 10.1984        | 0.0025        |
| Weighted Mean   | 10.2002        | 0.0021        |
| MM-Median       | 10.1990        | 0.0020        |

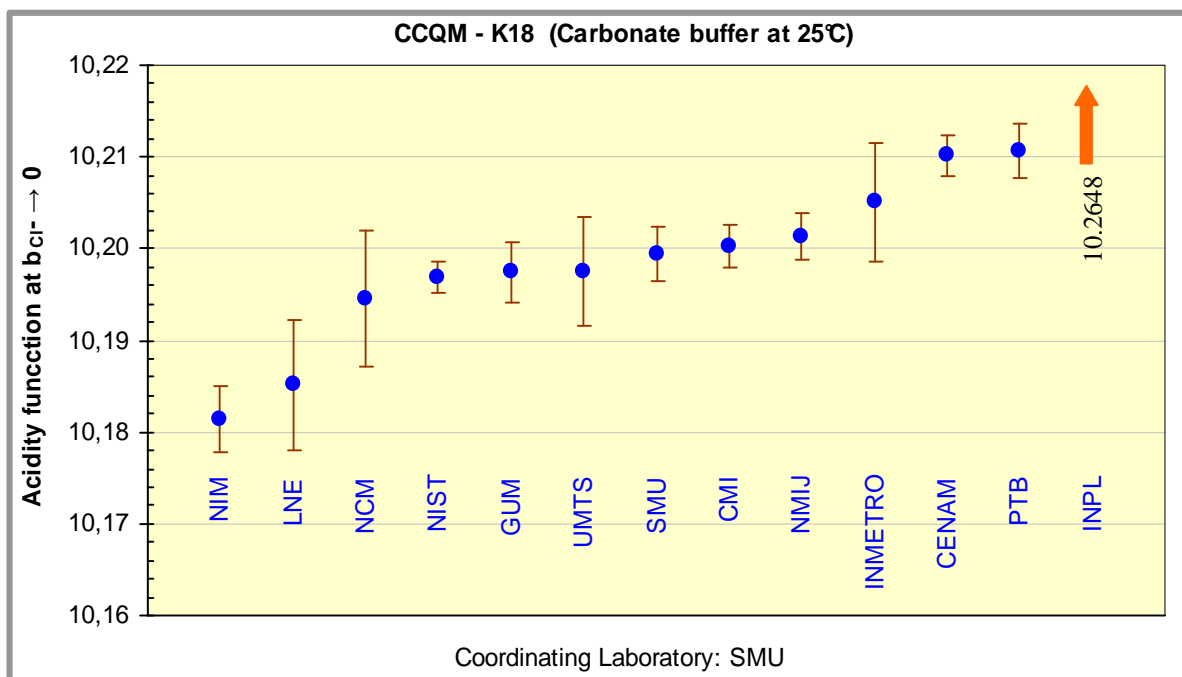


Figure 2 Results with expanded ( $k=2$ ) uncertainties given

### Measurements of standard potential $E^\circ$

The standard potential  $E^\circ$  is determined from a cell filled with hydrochloric acid of fixed molality, according to equation (5). The mean activity coefficient  $\gamma_{HCl}^\pm$  of HCl with molality  $0.01 \text{ mol kg}^{-1}$  at various temperatures is known [1].

$$E^\circ = U^* + \frac{2RT}{F} \cdot \ln(b_{HCl} \cdot \gamma_{HCl}^\pm) \quad (5)$$

Table 7 The standard potential of Ag/AgCl electrodes

| Institute | Mean $E^\circ$ | $U_{E^\circ} (k=2)$ |
|-----------|----------------|---------------------|
| INPL      | 0,21975        | 0,00055             |
| GUM       | 0,22212        | 0,00010             |
| NCM       | 0,22219        | 0,00019             |
| INMETRO   | 0,22232        | 0,00012             |
| CENAM     | 0,22234        | 0,00007             |
| LNE       | 0,22238        | 0,00016             |
| NIST      | 0,22242        | 0,00003             |
| PTB       | 0,22247        | 0,00009             |
| SMU       | 0,22250        | 0,00009             |
| NMIJ      | 0,22252        | 0,00009             |
| NIM       | 0,22253        | 0,00009             |
| CMI       | 0,22260        | 0,00010             |
| UMTS      | 0,22263        | 0,00021             |

In the measurement of INPL, an anomalous low value of the standard potential of the Ag/AgCl electrode is observed. The deviation compared to the usual values corresponds roughly to the INPL result deviation to the mean of the other participants' results. It might be therefore due to a problem in the assay of the hydrochloric acid used in its determination. Therefore the INPL result was not used in the calculation of reference value. The standard potential values given agree very well with values recalculated by the coordinating laboratory. On the other hand, a check of the calculated values of chloride molality using the supplied raw data showed that in some cases the values used were not correct, giving rise to bias in the order of up to 0.002 in terms of acidity function. Usually air buoyancy corrections in weighing of chloride salt and/or solution were not used or incorrect mass fraction of chloride was used.

The results of the latest comparisons in the field of pH are given in Table 8. The standard deviation of results (excluding outliers) is significantly higher than in previous comparisons [2, 3, 4]. The same holds for the spread of data in the third column. The measurement of the carbonate buffer is more complicated than that of other buffers; the spread is larger than that in the study CCQM-P52, which was done also on carbonate buffer.

Table 8 Dispersion characteristic<sup>2</sup> of results at different comparisons

| Type of comparison | Standard deviation | $x_{\max} - x_{\min}$ |
|--------------------|--------------------|-----------------------|
| K9                 | 0.0053             | 0,0189                |
| K17                | 0.0051             | 0,0143                |
| <b>K18</b>         | <b>0.0086</b>      | <b>0,0292</b>         |
| K19                | 0.0041             | 0,0142                |
| P52                | 0.0065             | 0,0209                |

### Measurement of HCl molality

The molality of HCl used was measured in most cases by coulometric, volumetric or weight titration (Table 9, Figure 4); the participants were in most cases able to prepare solution very close to target molality.

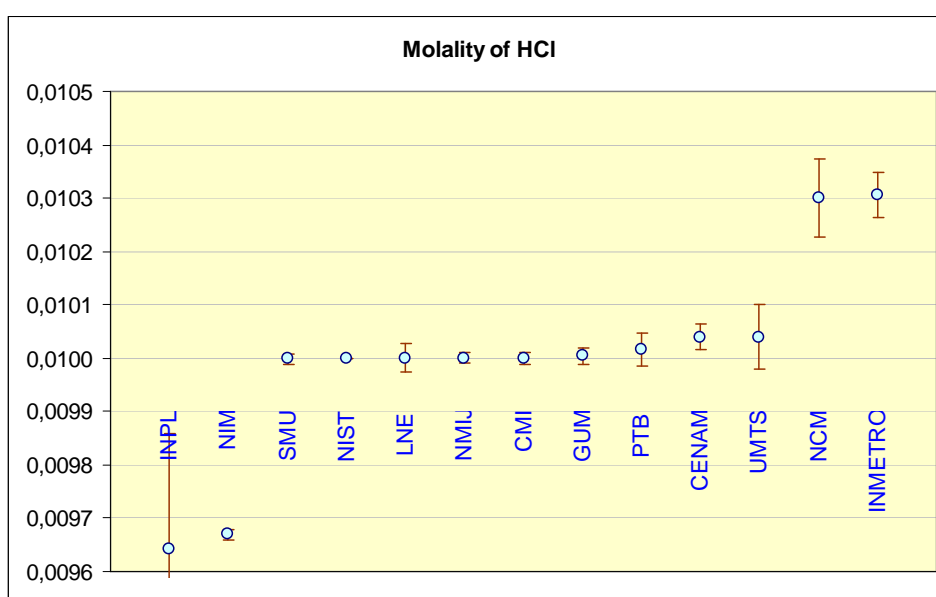


Figure 4 The molality of used HCl

<sup>2</sup> Without outliers

Table 9 *Molality of HCl used, its expanded uncertainty and method of determination*

| Institute | Molality of HCl | $U_{\text{HCl}}$<br>(k=2) | Method for HCl measurement used                         |
|-----------|-----------------|---------------------------|---|
| INPL      | 0,00964         | 0,00022                   | Potentiometric titration                                |
| NIM       | 0,009670        | 0,000010                  | Dilution from coulometrically determined HCl            |
| SMU       | 0,009998        | 0,000010                  | Conductimetry   |
| NIST      | 0,00999920      | 0,00000076                | Coulometric titration (H <sup>+</sup> )                 |
| LNE       | 0,010000        | 0,000027                  | Potentiometric titration AgCl                           |
| NMIJ      | 0,010000        | 0,000010                  | Coulometric titration (H <sup>+</sup> )                 |
| CMI       | 0,010000        | 0,000012                  | Dilution from coulometrically determined HCl            |
| GUM       | 0,010004        | 0,000016                  | Precipitation titrations (AgCl)                         |
| PTB       | 0,010016        | 0,000032                  | Coulometric titration (H <sup>+</sup> )                 |
| UMTS      | 0,010040        | 0,000024                  | Coulometric titration                                   |
| CENAM     | 0,010040        | 0,000060                  | Gravimetric potentiometric titration                    |
| NCM       | 0,010300        | 0,000074                  | Titrimetric method with NaOH                            |
| INMETRO   | 0,010307        | 0,000042                  | Potentiometric titration as acid, phthalate as standard |

### Uncertainties

Participants were requested to provide a full uncertainty budget according to GUM [5]. The structure of measurement uncertainties of participants' results is graphically depicted in Figure 5. Uncertainty of intercept (type A component), of  $E^\circ$ , and of type B without  $E^\circ$  contribution are given. The contribution from the  $E^\circ$  measurement is the main uncertainty source for some participants; usually the highest contribution to  $E^\circ$  uncertainty comes from the determination of HCl molality.

Different uncertainties of intercept and standard potential are understandable in view of differences in methods and equipment. Surprising differences can be found in the type B contributions, which should yield similar values. The differences are due to several reasons:

1. some sources are underestimated
2. some significant uncertainty sources are not taken into account
3. some sources are overestimated

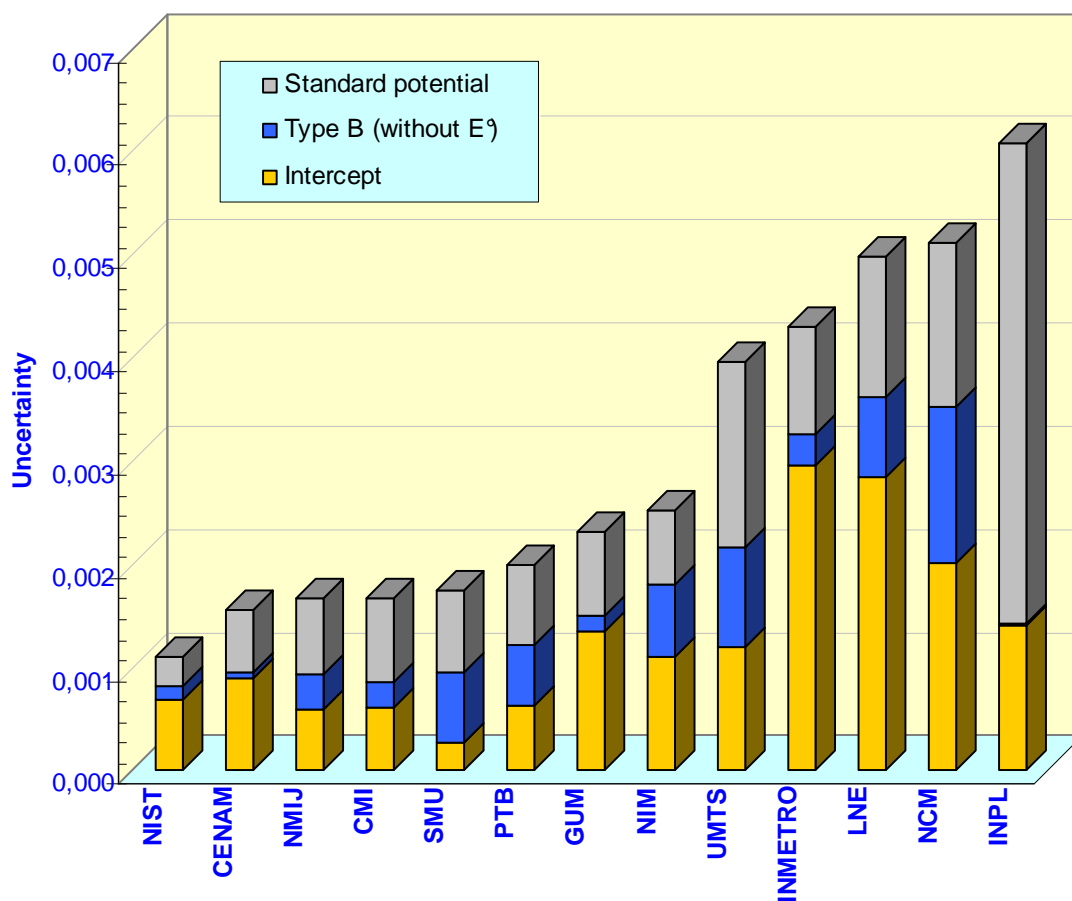


Figure 5 Structures of uncertainties

## Conclusions

There were 13 institutes participating in the comparison using primary method for pH measurement. The spread of data was slightly larger than in previous comparisons. The fair agreement between the results reflects increased difficulty in measurement, where the buffer composition may slightly change during the measurement and extrapolation to start time of measurement is usually necessary.

## Acknowledgments

The pilot laboratory gratefully acknowledges the contributions of all participants and of the members of the CCQM Working Group on Electrochemical Analysis and their valuable suggestions concerning the measurement protocol and the evaluation process.

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