

OMCL Network of the Council of Europe QUALITY MANAGEMENT DOCUMENT

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QUALIFICATION OF EQUIPMENT

ANNEX 9: CALIBRATION/QUALIFICATION OF PH-METERS

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ANNEX 9 OF THE OMCL NETWORK GUIDELINE “QUALIFICATION OF EQUIPMENT”

CALIBRATION / QUALIFICATION OF pH METERS

1. INTRODUCTION

This document is the 9th Annex to the core document “Qualification of Equipment”, which taken together should be used when planning, performing and documenting the qualification process of pH meters. The core document contains the introduction and general forms for Level I and II of qualification, which are common to all types of instruments.

Annex 9 contains instrument-related recommendations on parameters to be checked at Level III and IV of qualification and the corresponding typical acceptance limits, as well as practical examples on the methodology that can be used to carry out these checks.

2. AIM AND SCOPE OF THE GUIDELINE

This guideline describes the requirements for pH meters used for determination of pH in aqueous solutions. If measurements are made of test solutions of a non-aqueous or partially non-aqueous character, suspensions or emulsions, on a system calibrated as described above, the pH reading can only be considered to be an approximation of the true value (apparent pH). Appropriate electrodes should be used for such types of measurement.

The general principles of pH measurement together with apparatus and methods are provided in Ph. Eur. Chapter 2.2.3. ‘Potentiometric determination of pH’.

3. CONSIDERATIONS FOR LEVEL I AND II OF EQUIPMENT QUALIFICATION

At level I of the qualification of a pH meter (selection of instruments and suppliers) it is recommended to select a manufacturer of pH meters that can satisfy the needs of the laboratory and works under ISO 9001 certification. At level II of the qualification of a pH meter (installation and release for use) it is recommended to check all requirements set during the selection of the instrument, and calibration should be performed before putting into service by an accredited external service supplier, or internally by appropriately qualified personnel, using certified reference buffers according to an approved procedure.

4. MANAGEMENT OF ELECTRODES

Before measurement, the electrode(s) is (are) checked visually. The electrodes should be appropriately stored according to recommendations of the manufacturer (e.g. in a storage electrolyte). Refillable electrodes should be checked to ensure that the level of the inner electrolyte is satisfactory and no bubbles are present in the glass bulb. The diaphragm of the reference electrode should also be checked. Before first use, or if it has been stored out of electrolyte, it is usually necessary to condition the electrode according to the recommendation of the manufacturer. If the stabilisation of pH is too slow (long response time) or if there are difficulties in calibration,

the electrode will probably need to be cleaned or replaced. Cleaning procedure depends on the type of sample used as prescribed in the manual. Regular cleaning is recommended.

5. CALIBRATION AND MEASUREMENT CONDITIONS

The calibration consists of the determination of the slope and the offset of the measuring system. Unless otherwise prescribed in the method, all measurements are performed at the same temperature (± 2.5 °C) usually between 20 and 25 °C. The variation of pH with respect to temperature of a number of reference buffer solutions used for calibration is given in Ph. Eur. Chapter 2.2.3.. For the temperature correction, when necessary, follow the manufacturer's instructions. The apparatus is usually calibrated with two buffer solutions which should be chosen so that the expected pH value of the test sample lies in between the pH values of the buffer solutions. The range should be at least 2 pH units. The pH of a third buffer solution of intermediate pH read off on the scale must not differ by more than 0.05 pH units from the value corresponding to this solution. If the deviation is greater than ± 0.05 units, this may be because the range of the buffers chosen was too broad and re-calibration with a different system of buffers may be necessary.

Alternatively, a multi-point calibration may be performed to cover a wider pH-range. In that case it may be necessary to use more than one buffer solution for the verification depending on the expected pH values of the test samples.

The reference buffer solutions should preferably be certified reference materials. Otherwise, buffer solutions may be prepared as given in Ph. Eur. Chapter 2.2.3. These should be traceable to primary standards.

Immerse the electrodes in the solution to be examined and take the reading in the same conditions as for the buffer solutions.

Regular calibrations have to be performed as given in paragraph 6.

All solutions to be examined and the reference buffer solutions must be prepared using *carbon dioxide-free water R*.

STORAGE of buffer solutions

Store buffer solutions in suitable chemically resistant, tight containers, such as type I glass bottles or plastic containers suitable for aqueous solutions. Buffer solutions made in house- should not be kept for more than 3 months at room temperature. For commercially available buffer solutions, manufacturers' recommendations should be taken into account.

6. LEVEL III AND IV QUALIFICATION OF PH METERS

Most commercial pH meters offer a "self test" or "start-up test" where e.g. slope (and asymmetry potential) are tested and compared to the manufacturer's specifications.

Level III

Test to be performed	Minimum frequency	Typical tolerance limit	Who
Calibration or verification of thermometer	every two years	Manufacturer's specification or OMCL	external service supplier or OMCL

Level IV: Calibration/verification

Parameter to be checked	Typical frequency	Typical tolerance limit	Who
Slope	Each day of use or before each series of measurements	95.0 to 105.0 %	OMCL
Offset (pH-asymmetry)		+/-30 mV (offset of manufacturer +/- 0.5 pH units at defined temperature)	
Reference buffer solution		+/- 0.05 pH units	