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**Photography — Photographic-grade  
chemicals — Test methods —**

**Part 13:  
Determination of pH**

*Photographie — Produits chimiques de qualité photographique —  
Méthodes d'essai —*

*Partie 13: Détermination du pH*

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Case postale 56 • CH-1211 Geneva 20  
Tel. + 41 22 749 01 11  
Fax + 41 22 749 09 47  
E-mail [copyright@iso.ch](mailto:copyright@iso.ch)  
Web [www.iso.ch](http://www.iso.ch)

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**Contents**

	Page
1 Scope .....	1
2 Normative reference .....	1
3 Term and definition .....	1
4 Hazards .....	1
5 Reagents .....	1
6 Apparatus .....	2
7 Sampling .....	2
8 Procedure .....	2
8.1 Calibration and standardization .....	2
8.2 pH measurement .....	3
9 Test report .....	3
 <b>Annex</b>	
A Electrode care.....	4

## Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

International Standards are drafted in accordance with the rules given in the ISO/IEC Directives, Part 3.

Draft International Standards adopted by the technical committees are circulated to the member bodies for voting. Publication as an International Standard requires approval by at least 75 % of the member bodies casting a vote.

Attention is drawn to the possibility that some of the elements of this part of ISO 10349 may be the subject of patent rights. ISO shall not be held responsible for identifying any or all such patent rights.

International Standard ISO 10349-13 was prepared by Technical Committee ISO/TC 42, *Photography*.

ISO 10349 consists of the following parts, under the general title *Photography — Photographic-grade chemicals — Test methods*:

- *Part 1: General*
- *Part 2: Determination of matter insoluble in water*
- *Part 3: Determination of matter insoluble in ammonium hydroxide solution*
- *Part 4: Determination of residue after ignition*
- *Part 5: Determination of heavy metals and iron content*
- *Part 6: Determination of halide content*
- *Part 7: Determination of alkalinity or acidity*
- *Part 8: Determination of volatile matter*
- *Part 9: Reaction to ammoniacal silver nitrate*
- *Part 10: Determination of sulfide content*
- *Part 11: Determination of specific gravity*
- *Part 12: Determination of density*
- *Part 13: Determination of pH*

Annex A of this part of ISO 10349 is for information only.

# Photography — Photographic-grade chemicals — Test methods —

## Part 13:

### Determination of pH

#### 1 Scope

This part of ISO 10349 specifies a general test method for the determination of pH of solutions of photographic-grade chemicals to within  $\pm 0,1$  pH units.

This method is not intended to monitor the pH of formulated solutions used directly in photographic processing, e.g. developers.

#### 2 Normative reference

The following normative document contains provisions which, through reference in this text, constitute provisions of this part of ISO 10349. For dated references, subsequent amendments to, or revisions of, this publication do not apply. However, parties to agreements based on this part of ISO 10349 are encouraged to investigate the possibility of applying the most recent edition of the normative document indicated below. For undated references, the latest edition of the normative document referred to applies. Members of ISO and IEC maintain registers of currently valid International Standards.

ISO 10349-1:—<sup>1)</sup>, *Photography — Photographic-grade chemicals — Test methods — Part 1: General*

#### 3 Term and definition

For the purposes of this part of ISO 10349, the following term and definition apply.

##### 3.1

##### **calibration buffers**

materials, purchased from various chemical supply sources, that are used to calibrate the pH meter in preparation for measuring samples

#### 4 Hazards

General hazard warnings for the handling of chemicals are specified in ISO 10349-1.

#### 5 Reagents

##### 5.1 Calibration buffers

##### 5.1.1 pH 4,0 buffer solution

Potassium hydrogen phthalate based, with an accuracy of  $\pm 0,01$  pH units. Available from several vendors.

1) To be published. (Revision of ISO 10349-1:1992)

### 5.1.2 pH 7,0 buffer solution

Potassium/sodium phosphate based, with an accuracy of  $\pm 0,01$  pH units. Available from several vendors.

### 5.1.3 pH 10,0 buffer solution

Potassium carbonate/borate hydroxide based, with an accuracy of  $\pm 0,01$  pH units. Available from several vendors.

## 6 Apparatus

All glassware subject to heating shall be of heat-resistant borosilicate glass<sup>2)</sup>.

Pipettes and other volumetric glassware shall meet the requirements specified in ISO 10349-1.

**6.1 pH meter**, with resolution/accuracy of at least  $\pm 0,01$  pH units.

**6.2 Electrodes**, pH glass electrode with a range of 0 to 14; an accuracy of  $\pm 0,02$  pH units minimum; a sodium error of  $\pm 0,05$  pH units minimum; and a suitable reference electrode with appropriate filling solutions.

The choice of the reference electrode can be critical to the application and shall be chosen accordingly.

For routine measurements of solutions with a pH value greater than 10, a sodium-corrected electrode is strongly recommended.

NOTE A combination electrode may be used in place of a glass/reference pair. The same considerations apply when choosing the electrode.

**6.3 Constant temperature water-bath**, with pump and heating system designed to maintain a circulating current and constant temperature ( $\pm 1$  °C).

The bath shall be deep enough to keep the part of the sample container that is filled with the sample below the surface of the water. The bath temperature shall be set at 25 °C.

## 7 Sampling

Sampling shall be in accordance with ISO 10349-1.

## 8 Procedure

### 8.1 Calibration and standardization

**8.1.1** Consult the manufacturer's instruction manual for proper set-up and initial conditions of the pH meter (6.1).

**8.1.2** Rinse the electrodes (6.2) with water and blot excess water from the tip of the electrode with a soft tissue, taking care not to rub the tissue against the electrode membrane surface. It should be sufficient to hold the tissue near the surface of the electrode and allow water to be drawn into it.

Immerse the electrodes into the pH 7,0 buffer solution (5.1.2). Wait 2 min for the electrodes to equilibrate and the pH-meter reading to stabilize. If needed, adjust the "calibration control" of the pH meter to obtain the assigned buffer value.

2) Pyrex® is an example of suitable glassware available commercially. This information is given for the convenience of the users of this part of ISO 10349 and does not constitute an endorsement by ISO of this product.

**8.1.3** Rinse the electrodes with water and blot dry.

For low pH samples, immerse the electrodes into the pH 4,0 buffer solution (5.1.1). For high pH samples, immerse the electrodes into the pH 10,0 buffer solution (5.1.3).

Wait 2 min for the electrodes to equilibrate and the pH-meter reading to stabilize. If needed, adjust the "slope control" of the pH meter to obtain the assigned buffer value.

Note the slope value for this electrode pair. If this value drops below 95 %, repeat the calibration. If the slope remains below 95 %, corrective action should be performed before any samples are tested (see annex A).

**8.1.4** Rinse the electrodes with water; blot dry and proceed with sample measurements or properly store the electrodes for future use.

**8.1.5** Calibration of the pH meter shall be done just prior to sample measurement.

**8.1.6** Calibration buffers (5.1) shall be replaced daily.

## **8.2 pH measurement**

**8.2.1** After temperature equilibration and calibration of the pH meter for the desired pH range, the pH of the sample may be determined.

**8.2.2** Weigh the test portion specified in the appropriate International Standard, transfer it to a 100 ml beaker, and dissolve the sample in 50 ml of freshly boiled and cooled water.

**8.2.3** Rinse the electrodes with water and blot dry. Immerse the electrodes into the sample to be measured and wait 2 min for the electrodes to equilibrate and the pH meter reading to stabilize.

**8.2.4** Record the pH of the solution to the nearest 0,1 pH units.

## **9 Test report**

The test report shall specify the method used and the test result obtained.

It shall also mention all operating details not specified in this part of ISO 10349, or regarded as optional, together with details of any incidents which may have influenced the test result.

The test report shall include all information necessary for the complete identification of the sample.

## Annex A (informative)

### Electrode care

#### A.1 Storage

There is much conflicting information on suitable storage solutions for glass electrodes. Follow the manufacturer's recommendation for electrode care when possible.

When the electrode pair is out of service, it is recommended that the pH 7 phosphate buffer be used for glass-electrode storage, and the 3,5 mol/l potassium chloride buffer for reference electrode storage.

For temporary storage of the electrode pair between measurements, use a storage buffer consisting of 0,1 mol/l potassium chloride in a pH 7 phosphate buffer.

#### A.2 pH electrodes — Preconditioning/rejuvenation

Follow the manufacturer's recommendations for preconditioning the glass pH electrodes. A minimum soaking time of 2 h in the pH 7 phosphate buffer is recommended before use for pH measurement; overnight soaking is better.

Take the following rejuvenating steps if an electrode fails to meet the slope criterion, cannot achieve the assigned buffer values, or gives an unsatisfactory value when the control buffer is measured.

- a) Detach the electrode leads from the pH meter (6.1).
- b) Place the glass electrode tip in 1,0 mol/l hydrochloric acid for 5 min, followed by 5 min in 1,0 mol/l sodium hydroxide. Return the electrode (6.2) to 1,0 mol/l hydrochloric acid for an additional 5 min. Rinse the electrode with water.
- c) Soak the electrode in the pH 7,0 buffer solution (5.1.2) for 2 h.
- d) Reconnect the electrode and try another calibration. If there is no improvement, discard the electrode.

NOTE More severe reconditioning procedures are not recommended, due to both the toxicity of required reagents and the cost in analyst time versus the cost of electrode replacement.

- e) If there is an improvement but the electrode is still not reading the required values, repeat the sodium hydroxide and hydrochloric acid soaks and try another calibration. If there is no improvement, discard the electrode.

#### A.3 Reference electrode — Care/rejuvenation

##### A.3.1 Calomel reference electrode

Before putting a new calomel reference electrode into service, withdraw the saturated potassium chloride filling solution and refill with 3,5 mol/l potassium chloride solution. The lower salt concentration produces less crystallization inside the electrodes and in the reference junction.

At the beginning of each shift or day, withdraw the potassium chloride filling solution and refill the electrode with fresh 3,5 mol/l potassium chloride solution.

##### A.3.2 Silver/silver chloride electrodes

Before putting a new silver/silver chloride reference electrode into service, withdraw the saturated potassium chloride filling solution and refill with 3,5 mol/l potassium chloride solution **saturated with silver chloride** (the potassium



chloride filling solution is to be saturated with silver chloride, otherwise the internal silver chloride element will begin to dissolve).

At the beginning of each shift or day, withdraw the potassium chloride filling solution and refill the electrode with fresh 3,5 mol/l potassium chloride solution **saturated with silver chloride**.

When poor performance of the pH-measurement system does not improve after substitution of a new pH electrode, reference-electrode junction clogging may be the problem, especially where inaccurate or unsteady readings are obtained. If the filling solution becomes contaminated, refill with fresh solution and recheck the system.

Check frit-type junctions for flow by pressing just the tip of the reference electrode gently against a paper towel several times. A small wet spot will be visible if the junction is flowing.

### A.3.3 Clearing clogged reference electrodes

Clear a clogged calomel reference electrode by soaking the electrode junction for 0,5 h in a warm (not above 50 °C) solution of 3,5 mol/l potassium chloride diluted with water (1 + 9). Drain the electrolyte, replace with fresh 3,5 mol/l potassium chloride solution and retest the electrode.

For clogged silver/silver chloride reference electrodes, a 10-min soak in 10 % ammonium hydroxide solution can remove precipitated silver chloride from the junction. The electrode contains filling solution during this procedure. Avoid higher concentrations of ammonium hydroxide solution or longer periods of soaking, because the reference element can be damaged in some types of silver/silver chloride reference electrodes.

As with glass electrodes, more severe rejuvenation procedures are not recommended because they are costly and in many cases do more to damage the electrode than to improve its performance.

### A.3.4 Reference electrode accuracy check

**A.3.4.1** Assess errors of liquid-junction potential in reference electrodes by measuring the pH of the pH 7,0 buffer solution (5.1.2) at two ionic strengths differing by a factor of 10.

**A.3.4.2** Standardize the meter with the pH 7,0 buffer solution (5.1.2) and the pH 4,0 buffer solution (5.1.1) as in the "low-range pH meter calibration".

**A.3.4.3** Dilute 110 ml of the pH 7,0 buffer solution (5.1.2) to 1 litre with water.

**A.3.4.4** Measure the pH of the diluted buffer. The meter reading should be 7,065 pH  $\pm$  0,010 pH units for a properly functioning electrode reference junction.

## A.4 Combination electrodes

Follow the manufacturer's recommendations for care and rejuvenation of combination electrodes.

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**ICS 37.040.30**

Price based on 5 pages

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