



PEROXYGENS

A sector group of Cefic

CEFIC PEROXYGENS H₂O₂ AM-7160

Hydrogen Peroxide for industrial use

Determination of apparent pH (pHa)

Potentiometric method

March 2003

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Introduction

Cefic, the European Chemical Industry Council, is the forum and the voice of the Chemical Industry in Europe. There are three distinct groups of Cefic members: National Federations across Europe, major international companies and business members.

Mission of Cefic is to maintain and develop a prosperous chemical industry in Europe by promoting the best possible economic, social and environmental conditions to bring benefit to society with a commitment to the continuous improvement of all its activities including its safety, health and environmental performance.

The Cefic Peroxygens Hydrogen Peroxide Sub group proposes a set of four standard analytical methods intended to be used as a reference as regards analysis of hydrogen peroxide for industrial use. Using this common reference may facilitate providers/purchasers relationship.

These standard analytical methods are:

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CEFIC PEROXYGENS H₂O₂ AM-7161

This document is the CEFIC PEROXYGENS H₂O₂ AM-7160. It comprises only one part, presented hereafter.

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Hydrogen peroxide for industrial use Determination of apparent pH (pHa) Potentiometric method

WARNING – Hydrogen peroxide shall be handled and stored subject to the appropriate precautions so as to avoid the risk of violent decomposition.

In particular only clean equipment made of materials compatible with the product (for example: borosilicate glass, unplasticised polyvinylchloride, HD polyethylene, PTFE) shall be used and containers should be vented to prevent build up of pressure. Contact with combustible substances should be avoided.

Personnel handling hydrogen peroxide shall be familiar with the detailed advice contained in the supplier's Safety Data Sheet, and shall be provided with the necessary means of protection against splashes in the eye or on the skin. Appropriate protection shall be worn during the operations of this method.

NOTE For transportation of hydrogen peroxide, national and international regulations have to be observed.

1 Scope

This standard specifies a potentiometric method for the determination of the apparent pH (pHa) of hydrogen peroxide for industrial use.

2 Principle

Potentiometric determination of pH of a test portion.

3 Reagents

During the analysis, use only reagents of recognized analytical grade.

3.1 Water

Carbon dioxide free, with conductivity not greater than 200 µS/m. Suitable water can be obtained by boiling deionized or distilled water for 15 min. This water must be protected from atmospheric carbon dioxide during cooling and storage by a soda lime tube or similar device.

3.2 Potassium hydrogen phthalate

Standard pH solution, approximately 0,05 mol/l, with a pH of 4,00 at 20°C. Dissolve 10,12 g of potassium hydrogen phthalate [CO₂KC₆H₄CO₂H] (which has previously been dried for 2 h at 100°C to 130°C and cooled in a dessiccator) in water and dilute to the mark in a 1000-ml one-mark volumetric flask. Prevent contamination by acids or alkalis. The addition of a thymol crystal prolongs the shelf life of the solution.

3.3 Potassium tetraoxalate

Standard pH solution, approximately 0,05 mol/l, with a pH of 1,68 at 20°C. Dissolve 12,61 g of potassium tetraoxalate [CO₂KCO₂H(CO₂H)₂ · 2H₂O] in water and dilute to the mark in a 1000-ml one-mark volumetric flask.

3.4 Nitrogen

Free from carbon dioxide.

4 Apparatus

Ordinary laboratory apparatus and:

4.1 pH meter

With a resolution of 0,01 pH unit, with temperature compensation and with slope control.

4.2 Combined glass electrode for measurement of pH

Alternatively, a glass electrode of a current type and a reference electrode, for example calomel electrode, may be used.

4.3 Beaker or measuring cell

Of glass, of about 150 ml capacity, fitted with a multi-necked cover (for the insertion of the electrode, the temperature probe and the nitrogen flow).

5 Procedure

5.1 Calibration of the pH meter

Follow the manufacturer's instructions for the calibration of the pH meter using the standard pH solutions described in paragraphs 3.2 and 3.3.

5.2 Measurement of the apparent pH of hydrogen peroxide

Allow the sample to obtain laboratory temperature.

Half fill the beaker or measuring cell (4.3) with the sample. Dip the electrode (4.2) into the solution and read the apparent pH, maintaining an inert atmosphere with a gentle stream of nitrogen (3.4).

6 Expression of the result

Record the pH value with two decimal places.

7 Repeatability

The difference between two single results, found on identical test material by one analyst using the same apparatus within a short time-interval, will exceed 0,05 pH units on average not more than once in twenty cases in the normal and correct operation of the method.

NOTE Due to a possible lack of stability of the hydrogen peroxide, no interlaboratory tests could be carried out. Thus no reproducibility figures are available.

8 Test report

The test report shall give the following indications:

- a) the identification of the sample;
- b) the reference to the method employed;
- c) the results, expressed with one decimal place;
- d) any particular points observed in the course of the test;
- e) any operations not specified in this standard or regarded as optional.