



PEROXYGENS

A sector group of Cefic

CEFIC PEROXYGENS H₂O₂ AM-7159

Hydrogen Peroxide for industrial use

Determination of apparent acidity

Titrimetric method

March 2003

Notice

This document is a standard proposed by the Cefic Peroxygens Hydrogen Peroxide Sub Group.

Copyright and Disclaimer

All documents and information contained in this document are the property of the Cefic Peroxygens Hydrogen Peroxide Sub group. This document may be copied without prior permission from Cefic but all copies are supposed to be used by the copier for working use only. The only reference document is the one posted on the Cefic Internet site (<http://www.cefic.org>).

No other use, including reproduction, retransmission or editing, may be made without the prior written permission of the PEROXYGENS Sector Group*.

This publication has been prepared by the member companies of the Cefic Peroxygens Hydrogen Peroxide Sub group and we have tried to make the information on or linked to this paper as accurate and useful as possible. However neither the Sector Group nor any individual member company can accept liability for accident or loss attributable to the use of the information given in this document.

* Please contact the Cefic PEROXYGENS Sector Group:

Cefic
Peroxygens Sector Group
Avenue E. van Nieuwenhuyse 4
B - 1160 Brussels
Belgium
tel. +32 2 676 72 11
fax. +32 2 676 73 00
Internet: mail@cefic.be www.cefic.org

Contents

Introduction	4
1 Scope.....	5
2 Principle.....	5
3 Reagents.....	5
4 Apparatus.....	6
5 Procedure	6
6 Expression of results.....	7
7 Repeatability.....	7
8 Test report.....	8

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:

CEFIC PEROXYGENS H₂O₂ AM-7157

CEFIC PEROXYGENS H₂O₂ AM-7159

CEFIC PEROXYGENS H₂O₂ AM-7160

CEFIC PEROXYGENS H₂O₂ AM-7161

This document is the CEFIC PEROXYGENS H₂O₂ AM-7159. It comprises only one part, presented hereafter.

Attention is drawn to the possibility that some of the elements of CEFIC PEROXYGENS H₂O₂ AM-7159 may be the subject of patent rights. Cefic shall not be held responsible for identifying any or all such patent rights.

Hydrogen peroxide for industrial use

Determination of apparent acidity

Titrimetric 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 International Standard specifies a titrimetric method for the determination of the apparent acidity of hydrogen peroxide for industrial use.

NOTE This method is applicable to all industrial grades of hydrogen peroxide but results may not be comparable between samples of different grades.

2 Principle

Dilution of a test portion such that the hydrogen peroxide content, expressed as a percentage by mass, does not exceed 4,5 %. Titration with sodium hydroxide solution in the presence of a mixed indicator or a pH electrode.

3 Reagents

During the analysis, use only reagents of recognized analytical grade and only carbon dioxide-free distilled water or water of equivalent purity.

3.1 Sodium hydroxide, standard volumetric solution A

The concentration of the sodium hydroxide standard volumetric solution A is to be:

$$c_A(\text{NaOH}) = 0,1 \text{ mol/l}$$

3.2 Sodium hydroxide, standard volumetric solution B

The concentration of the sodium hydroxide standard volumetric solution B is to be:

$$c_B(\text{NaOH}) = 0,01 \text{ mol/l}$$

This solution shall be freshly prepared.

Optionally,

3.3 Mixed indicator solution

Dissolve 200 mg of methyl red and 100 mg of methylene blue in 100 ml of ethanol, 95 % by volume.

4 Apparatus

4.1 Micro-burette

Capacity 5 ml, graduated at 0.02 ml intervals

Optionally,

4.2 pH meter

With a resolution of 0,01 pH units, with slope control, and calibrated at pH 4,0 and pH 7,0 according to the manufacturer's instructions.

4.3 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.

4.4 Stirrer

Magnetic with PTFE-coated follower.

5 Procedure

5.1 Test Portion

Take 10,0 ml of the laboratory sample.

5.2 Determination

This determination may be carried out to a visual end-point using a colour indicator (5.3), or to a fixed pH using a pH meter (5.4).

5.3 Determination to a visual end-point

5.3.1 Determination of apparent acidity

Transfer 200 ml of water to a 500-ml conical flask, add a test portion (5.1) and 5 drops of mixed indicator solution (3.3).

Titrate, with continuous shaking, with sodium hydroxide standard volumetric solution A (3.1) until the colour just changes from violet to light green.

If the titre is less than 0,1 ml, carry out the determination on a further test portion as described in 5.3.2.

5.3.2 Determination of low levels of apparent acidity

Transfer 200 ml of water into each of two 500-ml conical flasks and add to each 5 drops of the mixed indicator solution (3.3). Add hydrochloric acid solution, approximately 0,01 mol/l, dropwise to each flask until the colour just changes from light green to violet.

Titrate the solution in both flasks with sodium hydroxide solution B (3.2) to the same grey colour. Retain one of these solutions as reference.

Add a test portion (5.1) to the other conical flask and titrate with sodium hydroxide solution B (3.2) until the colour is the same as that of the reference solution.

5.4 Determination to a fixed pH using a pH meter

5.4.1 Determination of apparent acidity

Transfer 200 ml of water to a 500-ml beaker containing the PTFE-coated follower (4.4) and add a test portion (5.1).

Insert the pH electrode (4.3) into the solution and stir the solution gently. Titrate dropwise with sodium hydroxide solution A (3.1) to pH 5,3.

If the titre is less than 0,1 ml then repeat the determination on a further test portion (5.1) as described in 5.4.2.

5.4.2 Determination of low levels of apparent acidity

Transfer 200 ml of water to a 500-ml beaker containing the PTFE-coated follower (4.4) and add a test portion (5.1).

Insert the pH electrode (4.3) into the solution and stir the solution gently. Titrate dropwise with sodium hydroxide solution B (3.2) to pH 5,3.

6 Expression of results

The apparent acidity, *a*, expressed in millimol of sodium hydroxide/l, is given by the formula:

$$a = \frac{c \times V_1}{V} \times 1000$$

where:

c is the concentration, expressed in mol/l, of the sodium hydroxide solution used,

V is the volume, expressed in ml, of the test portion (5.1),

*V*₁ is the volume, expressed in ml, of the sodium hydroxide solution used for the determination.

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 the repeatability given in Table 1 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

Table 1 - Repeatability

Titrant	Apparent Acidity (millimol/l)	Repeatability (millimol/l)
NaOH, 0,1 mol/l	1,0 - 2,0	0,27
	> 2,0	0,59
NaOH, 0,01 mol/l	0,1 - 0,4	0,006
	0,4 - 1,0	0,028
	1,0 - 2,0	0,035

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 result in millimol of sodium hydroxide/litre, expressed with one decimal place;
- d) any particular point observed in the course of the test;
- e) any operations not specified in this standard or regarded as optional.