

PEROXYGENS A sector group of Cefic

CEFIC PEROXYGENS H₂O₂ AM-7161

Hydrogen Peroxide for industrial use

Stability test

Determination of percentage loss

of hydrogen peroxide after 16 h at 96°

March 2003

Notice

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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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This document is the CEFIC PEROXYGENS H₂O₂ AM-7161. It comprises only one part, presented hereafter.

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Hydrogen peroxide for industrial use Stability test Determination of percentage loss of hydrogen peroxide after 16 h at 96°C

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 method for the determination, under standard conditions, 16 h at 96°C, of the percentage loss of hydrogen peroxide in hydrogen peroxide for industrial use.

2 Normative references

This standard refers to the document CEFIC PEROXYGENS H_2O_2 AM-7157 – Hydrogen peroxide for industrial use – determination of hydrogen peroxide content – titrimetric method

3 Principles

Determination of the hydrogen peroxide content of a test portion before and after heating for 16 h at 96°C.

4 Reagents

4.1 General considerations

During analysis, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

4.2 Washing solution

Free from phosphates, for cleaning glassware.

Use a 5% solution of a suitable liquid detergent composed of an alkaline solution of a mixture of anionic and non-ionic surfactants.

5 Apparatus

Ordinary laboratory apparatus and:

5.1 Flasks

Capacity 100 ml, of borosilicate glass, with a neck at least 150 mm long and a reference mark 20 mm to 40 mm from the rim.

Use new scratch-free flasks and reserve them thereafter for this determination. Reject any flasks which are subsequently scratched in use.

Before use, fill the flasks with the washing solution (4.2), heated to 95°C to 100°C and maintain at this temperature for 1 h. Empty the flasks and then rinse thoroughly with water.

5.2 Pipettes

50 ml, selected, cleaned and reserved in the same way as the flasks (5.1).

5.3 Constant level water bath

Capable of being controlled at a temperature of $(96\pm1)^{\circ}$ C for 16 h. The bath is fitted with a lid through which the neck of the flasks can pass. The neck of each flask shall protrude at least 120 mm above the lid, the design of which shall prevent the neck from being heated by water vapor from the bath.

5.4 Aluminum foil

99,5% Al, grease-free.

6 Procedure

6.1 Test portion

Take 50 ml of the laboratory sample.

6.2 Preparation of tested solution

Introduce a test portion (6.1) into one of the flasks (5.1) ensuring that the solution does not touch the internal neck wall.

Cover the mouth of the flask with a piece of the aluminum foil (5.4), press gently with the thumb to push the centre of the foil inside the mouth of the flask and ensure also that it is pressed circumferentially against the outside of the neck.

Immerse the flask in the bath (5.3), controlled at a temperature of (96±1)°C. After 16 h remove the fask and rapidly cool to ambient temperature.

Wash the aluminum foil (5.4) used to cover the flask (5.1) and add the washings to the tested solution. Dilute the solution to the reference mark.

6.3 Determination of hydrogen peroxide content of tested solution (6.2)

Assume that the hydrogen peroxide content is half of the nominal content of the laboratory sample and determine the hydrogen peroxide content as described in CEFIC PEROXYGENS H₂O₂ AM-7157.

6.4 Preparation of untested solution

Empty the flask used for the test (6.2), rinse it with water and add a further test portion (6.1). Dilute to the reference mark and mix.

6.5 Determination of hydrogen peroxide content of untested solution (6.4)

Assume that the hydrogen peroxide content is half the nominal content of the laboratory sample and determine the hydrogen peroxide content as described in CEFIC PEROXYGENS H_2O_2 AM-7157.

7 Expression of results

The percentage loss of hydrogen peroxide is given by the formula:

$$\frac{W_0 - W_1}{W_0} \ge 100$$

Where

 W_0 is the hydrogen peroxide content, expressed as a percentage by mass, in the untested solution (6.4),

 W_1 is the hydrogen peroxide content, expressed as a percentage by mass, in the tested solution (6.2).

8 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 0,4% 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.

9 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 two significant figures, and the form in which these are expressed,
- d) any particular points observed in the course of the lest,
- e) any operations not specified in this standard or regarded as optional.