P E R O X Y G E N S
A sector group of Cefic

CEFIC PEROXYGENS H_2O_2 AM-7157

Hydrogen Peroxide for industrial use

Determination of hydrogen peroxide content

Titrimetric method

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Notice

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

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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:

CEFIC PEROXYGENS H_2O_2 AM-7157
CEFIC PEROXYGENS H_2O_2 AM-7159
CEFIC PEROXYGENS H_2O_2 AM-7160
CEFIC PEROXYGENS H_2O_2 AM-7161

This document is the CEFIC PEROXYGENS H_2O_2 AM-7157. It comprises only one part, presented hereafter.

Attention is drawn to the possibility that some of the elements of CEFIC PEROXYGENS H_2O_2 AM-7157 may be the subject of patent rights. Cefic shall not be held responsible for identifying any or all such patent rights.

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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 titrimetric method for the determination of the hydrogen peroxide content of hydrogen peroxide for industrial use.

2 Principle

Titration of a test portion in acid solution with a standard volumetric solution of potassium permanganate.

3 Reagents

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

3.1 Sulfuric acid

Concentration of the sulfuric acid solution is to be 490 g/l.
This solution is to be free from reducing substances.
Check for the absence of reducing substances by adding two drops of the potassium permanganate solution (3.2.1) to 1 l of the sulfuric acid solution. The pink colouration shall persist for at least 30 min.

3.2 Standard volumetric solution of potassium permanganate

The concentration of this standard volumetric solution is to be:

$$c(\frac{1}{5}\,KMnO_4)=0{,}25\ mol/l$$

3.2.1 Preparation of the solution

- Weigh about 39,7 g of potassium permanganate;
- dissolve in 1 l of water;
- boil 60 min;
- filter through a sintered glass filter (porosity: 40 μm to 100 μm);
- dilute to 5 l;
- allow to stand for two weeks;
- filter through a sintered glass filter (same porosity as above);
- standardize with sodium oxalate.

3.2.2 Standardization

3.2.2.1 Reagent: sodium oxalate

- Dry the sodium oxalate between 105°C and 110°C for 2 h;
- allow to cool in a dessiccator.

3.2.2.2 Procedure

- Weigh, to the nearest 0,0001 g, 0,67 g of this dry material;
- introduce it into a 500-ml conical flask and add 250 ml of water and 15 ml of sulfuric acid solution (3.1);
- heat the mixture to 40°C by means of a hot water bath;
- add quickly from a burette 38 ml of the potassium permanganate solution (3.2.1). Discolouration should occur after shaking for several seconds – if necessary, raise the temperature of the test solution slightly (55°C to 60°C);
- continue adding the potassium permanganate solution, drop by drop, while shaking the flask, until a pink colouration is obtained which persists for 30 s.

3.2.2.3 Calculation

The concentration (c) of the potassium permanganate solution (3.2.1), expressed in mol/l, is given by the formula:

$$c = \frac{2}{M_1} \times m_1 \times \frac{1000}{V_1}$$

where

m_1 is the mass, expressed in g, of the weighed sodium oxalate (3.2.2.1);

M_1 is the molecular weight, expressed in g/mol, of sodium oxalate (M_1 = 134,0);

V_1 is the volume, expressed in ml, of the potassium permanganate solution used for the titration.

4 Apparatus

Ordinary laboratory apparatus and borosilicate glass containers, with a capacity of about 1,5 ml (reserved only for the weighing of hydrogen peroxide).

5 Procedure

5.1 Test portion

Depending on the hydrogen peroxide content of the sample, weigh, to the nearest 0,0001 g, into one of the clean glass containers (4), the mass of the sample indicated below:

- about 0,5 g for solution less than 33 % by mass;
- about 0,35 g for solution from 33 % to 55 % by mass;
- about 0,25 g for solution from 55 % to 75 % by mass;
- about 0,15 g for solution greater than 75 % by mass.

5.2 Determination

- Introduce 60 ml of water and 15 ml of the sulfuric acid solution (3.1) into a 500-ml conical flask;
- add the potassium permanganate solution (3.2.1), drop by drop, shaking continuously, until a slight pink colouration is obtained which persists for 30 s (1 drop is usually sufficient);
- add the test portion (5.1) in the glass container to the flask and titrate with the potassium permanganate solution (3.2.1) until a pink colouration is obtained which persists for 30 s.

6 Expression of results

The hydrogen peroxide content, w, expressed as a percentage by mass, is given by the formula

$$w = \frac{V_2}{1000} \times c \times \frac{M_2}{2} \times \frac{100}{m_2}$$

where

V_2 is the volume, expressed in ml, of the potassium permanganate solution (3.2.1) used for the determination;

c is the concentration of the potassium permanganate solution, expressed in mol/l (see 3.2.2.3);

m_2 is the mass, expressed in g, of the test portion (5.1);

M_2 is the molecular mass, expressed in g/mol, of hydrogen peroxide (M_2 = 34,01).

Calculate the result with four significant figures.

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 0,15 percent by mass 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 test 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 three significant figures, and the form in which these are expressed;

d) any particular points observed in the course of the test;

e) any operations not specified in this standard or regarded as optional.