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Glycerine for industrial use — Determination of glycerol content — Titrimetric method

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FOREWORD

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It has been approved by the Member Bodies of the following countries :

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South Africa, Rep. of
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Glycerine for industrial use — Determination of glycerol content — Titrimetric method

1 SCOPE

This International Standard specifies a titrimetric method for the determination of the glycerol content of glycerine for industrial use.

2 FIELD OF APPLICATION

The method is applicable to the analysis of glycerines free from organic compounds other than glycerol having more than two hydroxyl groups on adjacent carbon atoms, such as sugars which can also give rise to formic acid and, for this reason, will interfere in the determination.

3 PRINCIPLE

Cold oxidation of the glycerol by sodium periodate in a strongly acid medium. Titration of the formic acid produced by the reaction with standard volumetric sodium hydroxide solution, using a pH meter.

4 REACTION

$$CH_2OH{-}CHOH{-}CH_2OH + 2NaIO_4 \longrightarrow HCOOH + 2HCHO + 2NaIO_3 + H_2O$$

5 REAGENTS

5.1 Distilled water, or water of equivalent purity, free from carbon dioxide.

5.2 1,2-ethanediol (ethylene glycol) diluted solution.

Dilute 1 volume of 1,2-ethanediol, free from glycerol and neutralized to phenolphthalein, with 1 volume of the water (5.1).

5.3 Sulphuric acid, approximately 0,1 N solution.

5.4 Sodium formate ($NaCHO_2$), approximately N solution.

5.5 Sodium metaperiodate ($NaIO_4$) neutral, titre at least 98 % (*m/m*); 60 g/l acidic solution.

5.5.1 *Verification of neutrality of the product*

Dissolve 5 g of the product in 150 ml of the water (5.1) in a 500 ml conical flask, add 5 ml of the 1,2-ethanediol (5.2) and allow to stand in the dark and protected from atmospheric carbon dioxide for 30 min. It shall then be possible to neutralize the solution to phenol red by means of less than 1 ml, and preferably less than 0,2 ml, of 0,1 N sodium hydroxide solution.

5.5.2 *Preparation of the acidic solution*

Dissolve, in the cold, 60 g, weighed to the nearest 0,1 g, of the sodium metaperiodate (5.5) in about 500 ml of the water (5.1) to which has been added 120 ml of the sulphuric acid solution (5.3). Dilute to the mark with the water (5.1) in a 1 000 ml one-mark volumetric flask and mix. Filter, if necessary, through a sintered glass filter.

5.5.3 *Verification of the acidity of the solution*

The volume of the sodium hydroxide solution (5.7) used during the blank test (7.2) should not be less than 4,5 ml, this corresponding to the acidity produced during the basic reaction (4).

5.5.4 *Storage of the solution*

Store the solution in a dark glass flask fitted with a ground glass stopper.

5.6 Sodium hydroxide, approximately 0,05 N solution.

5.7 Sodium hydroxide, 0,125 N standard volumetric solution, free from carbon dioxide.

NOTE – Until a primary standard acid is established internationally, it is recommended to interested parties that the standardization of this solution be carried out by means of the same batch of primary standard.

5.8 Phenolphthalein, 5 g/l ethanolic solution.

Dissolve 0,5 g of phenolphthalein in 95 % (*V/V*) ethanol and dilute to 100 ml with the same ethanol.

6 APPARATUS

As a general rule, cork stoppers should not be used and ground glass joints are recommended.

Ordinary laboratory apparatus and

6.1 Precision burette, 50 ml, complying with class A of ISO/R 385, with the tip drawn out so as to allow delivery of 30 drops per millilitre.

6.2 pH meter, fitted with a glass electrode. The pH meter should be calibrated by means of two buffer solutions :

— potassium hydrogen phthalate $[C_6H_4(COOK)(COOH)]$, 0,05 M solution (10,12 g/l, pH 4,00 at 20 °C).

— disodium tetraborate decahydrate $(Na_2B_4O_7.10H_2O)$, 0,01 M solution (3,81 g/l, pH 9,22 at 20 °C).

Use standard materials specially prepared for the measurement of pH.

7 PROCEDURE

The presence of carbon dioxide can introduce errors and it is advisable to cover the vessels containing the test solutions with a clock glass during the standing periods and also to avoid enriching the carbon dioxide content of the laboratory atmosphere by other operations carried out at the same time.

7.1 Test portion

Weigh, to the nearest 0,000 1 g, a test portion containing not more than 0,50 g of glycerol.

If the approximate glycerol content is not known, it should be determined by a preliminary test on 0,50 g of the sample.

In the case of glycerines of which the glycerol content is greater than 75 %, it is preferable to weigh, to the nearest 0,000 1 g, 5 ± 0,1 g of the test sample, to dilute it to the mark in a 500 ml one-mark volumetric flask with the water (5.1) and, after mixing, to take 50 ml of this solution.

7.2 Blank test

Carry out, at the same time as the determination and under the same conditions, a blank test without the test portion using the same quantities of reagents and diluting water as for the determination.

7.3 Determination

7.3.1 *Preparation of the test solution*

In the case of alkaline samples or samples giving a tarry precipitate on acidification, place the test portion (7.1) in a flask fitted with a reflux condenser, dilute to 50 ml if necessary, neutralize in the presence of 2 drops of the phenolphthalein solution (5.8) just to decolorization by means of the sulphuric acid solution (5.3) and add a 5 ml excess of the same acid. Bring to boiling and maintain there for 5 min. Allow to cool. Filter, if necessary, and wash the filter with the water (5.1). Transfer the solution quantitatively to a 600 ml tall-form beaker.

In all other cases, place the test portion (7.1) directly into the 600 ml tall-form beaker.

7.3.2 *Titration*

Dilute the test solution (7.3.1) with the water (5.1) to a volume of about 250 ml.

Adjust the pH to 7,9 ± 0,1 with the sodium hydroxide solution (5.6), stirring constantly and checking with the pH meter (6.2).

Add 50,0 ml of the sodium metaperiodate solution (5.5). Mix with moderate agitation, cover the beaker with a clock glass and allow to stand for 30 min in the dark at a temperature not exceeding 35 °C.

Then add 10 ml of the 1,2-ethanediol (5.2), mix and allow to stand for a further 20 min under the same conditions.

Add 5,00 ml of the sodium formate solution (5.4) and then titrate the acidity present with the standard volumetric sodium hydroxide solution (5.7) to a pH of 7,9 ± 0,2, checking with the pH meter (6.2).

8 EXPRESSION OF RESULTS

The glycerol content is given, as a percentage by mass, by the formula :

$$(V_1 - V_2) \times 0{,}125 \times 0{,}092\,1 \times \frac{100}{m} = 1{,}151\,\frac{(V_1 - V_2)}{m}$$

where

V_1 is the volume, in millilitres, of standard volumetric sodium hydroxide solution (5.7) used for the determination;

V_2 is the volume, in millilitres, of standard volumetric sodium hydroxide solution (5.7) used for the blank test;

m is the mass, in grams, of the test portion, taking account of any intermediate dilution.

9 TEST REPORT

The test report shall include the following particulars :

a) the reference of the method used;

b) the results and the method of expression used;

c) any unusual features noted during the determination;

d) any operation not included in this International Standard, or regarded as optional.