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Chemical Reagent - Ethanol

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(ISO 6353-2:1983, Reagents for Chemical Analysis - Part 2: Specifications: First Series, NEQ)

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Chemical Reagent - Ethanol

WARNING: some test procedures specified in this document may lead to hazardous situations, and it is the user's responsibility to take appropriate safety and health measures.

1 Scope

This document specifies the characters, technical requirements, test methods, inspection rules, packaging and marking of the chemical reagent ethanol (anhydrous ethanol).

This document is applicable to the inspection of the chemical reagent ethanol (anhydrous ethanol).

NOTE: the rational formula of the chemical reagent ethanol (anhydrous ethanol) is CH₃CH₂OH, the relative molecular mass is 46.07 (in accordance with the 2022 international relative atomic mass), and the CAS No. is 64-17-5.

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

GB/T 601 Chemical Reagent - Preparations of Standard Volumetric Solutions

GB/T 602 Chemical Reagent - Preparations of Standard Solutions for Impurity

GB/T 603 Chemical Reagent - Preparations of Reagent Solutions for Use in Test Methods

GB/T 605 Chemical Reagent - General Method for the Measurement of Color

GB/T 606 Chemical Reagent - General Method for the Determination of Water - Karl Fischer Method

GB/T 611-2021 Chemical Reagent - General Method for the Determination of Density

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 9721-2006 Chemical Reagent - General Rules for the Molecular Absorption Spectrophotometry

GB/T 9722-2006 Chemical Reagent - General Rules for the Gas Chromatography

Carrier gas and column flow: nitrogen, 1.0 mL/min.

Chromatographic column: 100% polydimethylsiloxane capillary column (or a capillary column that can achieve equivalent separation effects).

Column length: 30 m.

Column inner diameter: 0.32 mm.

Liquid film thickness: 0.5 µm.

Column temperature: maintain at 40 °C for 5 min, at a rate of 10 °C/min, raise to 150 °C and maintain or 2 min.

Vaporization chamber temperature: 150 °C.

Test chamber temperature: 150 °C.

Degree of separation of difficult-to-separate substance pairs: $R_{\text{ethanol and methanol}} \ge 1.5$.

Effective plate height of chromatographic column: $H_{\text{eff}} \le 2.7 \text{ mm}$.

Injection volume: 0.2 µL.

Split ratio: 50:1.

Air flow rate: 300 mL/min.

Hydrogen flow rate: 30 mL/min.

Relative retention value of a component relative to the main body: $r_{\text{methanol}} = 0.92$; $r_{\text{isopropanol}}$, $r_{\text{ethanol}} = 1.08$; $r_{\text{n-propanol}}$, $r_{\text{ethanol}} = 1.27$.

6.2.3 Quantitative method

In accordance with the stipulations of 9.2 in GB/T 9722-2006, conduct the determination.

Take the arithmetic mean of two parallel determination results as the determination result. The absolute difference between two parallel determination results shall be not greater than 0.1%.

6.3 Chroma

In accordance with the stipulations of GB/T 605, conduct the determination.

6.4 Density

In accordance with the stipulations of 4.2 or 4.4 in GB/T 611-2021, conduct the determination.

6.5 Water-mixing Test

Measure-take 15 mL of sample, add 45 mL of water, mix it well, and let it stand for 1 h. Conduct visual inspection. The solution shall be clear and odorless.

6.6 Evaporation Residue

Weigh-take 100 g (about 127 mL) [for liquid-phase chromatography purity, take 250 g (about 316 mL); for top-grade purity, take 200 g (about 253 mL)] of sample, in accordance with the stipulations of GB/T 9740, conduct the determination.

6.7 Acidity

In accordance with the stipulations of 5.1 in GB/T 9736-2008, conduct the determination. Measure-take 100 mL of carbon dioxide-free water, pour it into a 250 mL conical flask, add 4 drops of phenolphthalein indicator solution (10 g/L); use sodium hydroxide standard titration solution [c(NaOH) = 0.02 mol/L] to neutralize it, until the solution turns pink, and maintain for 30 s. Add 20 g (about 25.3 mL) of sample, use sodium hydroxide standard titration solution [c(NaOH) = 0.02 mol/L] to titrate it, until the solution turns pink, and maintain for 30 s. The result shall be calculated in accordance with the stipulations of 5.1.2 in GB/T 9736-2008.

6.8 Alkalinity

In accordance with the stipulations of 5.1 in GB/T 9736-2008, conduct the determination. Measure-take 100 mL of carbon dioxide-free water, pour it into a 250 mL conical flask, add 2 drops of methyl red indicator solution (1 g/L); use hydrochloric acid standard titration solution [c(HCl) = 0.02 mol/L] to neutralize it, until the solution changes from yellow to orange, and maintain for 30 s. Add 20 g (about 25.3 mL) of sample, use hydrochloric acid standard titration solution [c(HCl) = 0.02 mol/L] to titrate it, until the solution changes from yellow to orange, and maintain for 30 s. The result shall be calculated in accordance with the stipulations of 5.1.2 in GB/T 9736-2008.

6.9 Moisture

Weigh-take 4 g (about 5 mL) of sample and use 10 mL of methanol as the solvent. In accordance with the stipulations of GB/T 606, conduct the determination.

6.10 Methanol

6.10.1 Reagents, materials and instruments

- 6.10.1.1 Shall comply with the stipulations of Chapter 5 and Chapter 6 in GB/T 9722-2006.
- **6.10.1.2** Methanol: primary or secondary standard substance (with a metrology certificate).

6.10.1.3 Methanol external standard solution

Preparation: weigh-take an appropriate amount of sample, add an appropriate amount of methanol standard substance, so that the methanol concentration in the solution is close to the mass fraction of the item indicators specified in Table 1. Both weighings are accurate to 0.0001

Shall comply with the stipulations of Chapter 5 and Chapter 6 in GB/T 9722-2006.

6.11.2 Determination conditions

See 6.2.2.

6.11.3 Quantitative method

In accordance with the stipulations of 9.2 in GB/T 9722-2006, conduct the determination.

Take the arithmetic mean of two parallel determination results as the determination result. When the concentration is not greater than 0.0005%, the difference in the determination results shall be not greater than 20% of the arithmetic mean of two determinations; when the concentration is greater than 0.0005%, the difference in the determination results shall be not greater than 10% of the arithmetic mean of two determinations.

6.12 Carbonyl Compounds

Weigh-take 0.5 g (approximately 0.63 mL) of sample, in accordance with the stipulations of GB/T 9733, conduct the determination. The dark red color of the solution shall be not darker than the standard colorimetric solution.

The preparation of the standard colorimetric solution is to take carbonyl compound (CO) standard solution containing 0.015 mg (top-grade purity and analytical purity) or 0.025 mg (chemical purity) and process it in the same way at the same time as the sample.

6.13 Easily Carbonized Substance

In accordance with the stipulations of GB/T 9737-2008, conduct the determination. Measure-take 10 mL of sulfuric acid (top-grade purity, $95\% \pm 0.5\%$), cool it to 10 °C; under shaking, dropwise add 10 mL of sample (at this moment, the temperature of the solution shall be not higher than 20 °C) and let it stand for 5 min. The color of the solution shall be not darker than the R/40 (top-grade purity) or R/30 (analytical purity) or R/25 (chemical purity) standard color specified by 5.1 in GB/T 9737-2008.

6.14 Iron

Weigh-take 20 g (about 25.3 mL) of sample, place it in an evaporating dish, add 0.5 mL of sulfuric acid solution (10%); under an infrared lamp or on a water bath at about 75 °C, evaporate it to near dryness, and dilute to 15 mL. Use ammonia solution (10%) to adjust the pH of the solution to 2, then, in accordance with the stipulations of GB/T 9739, conduct the determination. The red color of the solution shall be not darker than the standard colorimetric solution.

The preparation of the standard colorimetric solution is to take the standard solution containing 0.002 mg of iron (Fe), add 0.5 mL of sulfuric acid solution (10%) and dilute to 15 mL; process it in the same way at the same time as the sample.

6.15 Zinc

6.15.1 Reagents, materials and instruments

In accordance with the stipulations of Chapter 5 and Chapter 6 in GB/T 9723-2007.

6.15.2 Instrument conditions

Light source: zinc hollow cathode lamp.

Wavelength: 213.5 nm.

Flame: acetylene-air.

6.15.3 Determination method

Weigh-take 5 g (about 5.3 mL) of sample, place it in an evaporating dish; under an infrared lamp or on a water bath at about 75 °C, evaporate it to dryness. Add 1 mL of hydrochloric acid solution (20%) to dissolve the residue and dilute to 10 mL. In accordance with the stipulations of 7.2.1 in GB/T 9723-2007, conduct the determination.

6.16 Reduced Potassium Permanganate Substance

6.16.1 Preparation of Solution A

In accordance with the stipulations of 4.3 in GB/T 9737-2008, prepare the solution.

6.16.2 Preparation of Solution B

In accordance with the stipulations of 4.5 in GB/T 9737-2008, prepare the solution.

6.16.3 Determination method

Weigh-take 19 g (about 24 mL) [for chemical purity, take 8 g (about 10 mL)] of sample, pour it into a dry colorimetric tube with a stopper, and adjust the temperature to 25 °C. Add 0.1 mL of potassium permanganate standard titration solution $[c(\frac{1}{5}KMnO_4) = 0.1 \text{ mol/L}]$, and shake it well. Tightly cap the colorimetric tube, at 25 °C, place it in the dark for 5 min. The pink color of the solution shall be not lighter than the standard colorimetric solution of the same volume.

The preparation of the standard colorimetric solution is to respectively measure-take 7.9 mL of Solution A and 6.0 mL of Solution B, pour it into a 100 mL volumetric flask, and use hydrochloric acid solution (1 + 40) to dilute to the scale.

6.17 Absorbance

6.17.1 Reagents and instruments

The water used for the test shall comply with Grade-2 water specifications in GB/T 6682.

UV-visible spectrophotometer shall comply with the stipulations of Chapter 6 in GB/T 9721-

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