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Denatured fuel ethanol

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Denatured fuel ethanol

1 Scope

This standard specifies the terms and definitions, requirements, test methods, inspection rules and marking, packaging, transportation, and storage of denatured fuel ethanol.

This standard applies to the fuel ethanol which is produced by using such raw materials as starch, sugar, cellulose and so on through fermentation, distillation, and dehydration, AND denatured by adding the denaturant.

2 Normative references

The following documents are essential to the application of this document. For the dated documents, only the versions with the dates indicated are applicable to this document; for the undated documents, only the latest version (including all the amendments) are applicable to this Standard.

GB 190 Packaging symbol of dangerous goods

GB/T 191 Packaging – Pictorial marking for handling of goods

GB/T 601 Chemical reagent – Preparation of standard volumetric solutions

GB/T 602 Chemical reagent – Preparations of standard solutions for impurity determination

GB/T 603 Chemical reagent – Preparations of reagent solution for use in test methods

GB/T 611-2006 Chemical reagent – General methods for the determination of density

GB/T 683 Chemical reagent - Methanol

GB/T 4472 Determination of density and relative density for chemical products

GB/T 6682-2008 Water for analytical laboratory use – Specification and test methods

GB/T 8019 Standard test method for gum content in fuels – By jet evaporation

GB/T 9722 Chemical reagent – General rules for the gas chromatography

GB/T 9724 Chemical reagent – General rule for the determination of pH

GB/T 9725-2007 Chemical reagent – General rule for potentiometric titration

GB 18351 Ethanol gasoline for motor vehicles (E10)

GB/T 22030 Blendstocks of ethanol gasoline for motor vehicles

GB 50016 Code of design on building fire protection and prevention

GB 50074 Code for design of oil depot

SH/T 0253 Determination method for total sulfur content in light petroleum products (colorimetric method)

SH/T 0689 Standard test method for determination of total sulfur in light hydrocarbons motor fuels and oils by ultraviolet fluorescence

3 Terms and definitions

The following terms and definitions apply to this document.

3.1

Fuel ethanol

It refers to the ethanol to which no denaturant is added AND which can be used as fuel.

3.2

Denaturant

It refers to the ethanol gasoline blending component oil or ethanol gasoline for motor vehicles which is added into the fuel ethanol to make it inedible.

3.3

Denatured fuel ethanol

It refers to the fuel ethanol to which the denaturant is added for the purposes of blending the ethanol gasoline for motor vehicles. It is inedible. It can be

a) During sample injection

b) During sinking

Figure 1 -- Sampler

6.2.1.2 Products in tanker or tank car is sampled from the middle part for one time. As for the products contained in tanker or tank car, it shall take sample from three locations: upper part, middle part, and lower part of the liquid in the tank, in the volume proportion of 2:3:2 from the vertical tank AND in the volume proportion of 1:3:1 from the horizontal tank, AND contain it in the glass bottle AND mix it uniformly.

6.2.2 Sampling amount

TAKE 2 L of sample from each batch; MIX it uniformly; respectively CONTAIN it into two 1 L narrow-mouthed reagent glass bottle; MAKE it labelled and indicated of the following information: product name, batch number (tank, tanker number), manufacturer name, sampling date, location, and sampler. SEND one bottle to the laboratory for detection, and SEAL and PRESERVE the other bottle for one month for reference.

6.3 Inspection classification

6.3.1 Exit-factory inspection

6.3.1.1 Before exit-factory, the quality inspection department of the manufacturer shall carry out the inspection in accordance with the provisions of this standard, AND it can only be exit-factory for sale after passing the inspection AND obtaining the quality certificate.

6.3.1.2 Exit-factory inspection items: appearance, ethanol, methanol, moisture, acidity and pHe.

6.3.2 Type inspection

6.3.2.1 Type inspection items are all physical and chemical requirements in Table 1.

6.3.2.2 Type inspection shall be carried out once every six months AND it shall also be carried out in any of the following cases:

- a) When replacing equipment or main raw materials;
- b) In case of production restoration after long-term shutdown;
- c) When there is major difference between the exit-factory inspection results and the last type inspection results;

Appendix A

(Normative)

Determination of ethanol and methanol in denatured fuel ethanol (gas chromatography)

A.1 Scope of application

A.1.1 This method is applicable to the determination of ethanol and methanol in denatured fuel ethanol by gas chromatography. The scope of determination of this method is the ethanol having a mass fraction from 93% to 97% AND the methanol having a mass fraction from 0.01% to 0.6%. The mass fraction of ethanol and methanol beyond this scope may also be determined by this method, BUT its precision may be not suitable. Meanwhile, this method also provides the formula of converting the contents of these two alcohols from mass fraction to volume fraction.

A.1.2 This method cannot determine the moisture content of denatured fuel ethanol. Moisture shall be measured in accordance with Appendix B, AND the alcohol content as determined by gas chromatography is corrected.

A.2 Method summary

The sample enters into a chromatograph of a quartz capillary column equipped with a bonded methyl silicone, carried by the carrier gas helium, the evaporated sample is passed through the column, each component of the sample is separated in the chromatographic distribution process, the component flowing out of the column is detected by the flame ionization detector, AND the signal obtained by the detector is processed by the electronic data receiving system. Under the completely same conditions, its preservation time is compared with that of the analytical standard sample to quantify the ethanol and methanol, AND the peak area normalization method is used for qualitative, to determine the mass fraction of each component, AND it is then converted into the volume fraction.

A.3 Instruments and equipment

A.3.1 Gas chromatograph

A.3.1.1 It shall be capable of operating under the chromatographic conditions listed in Table A.1.

dried; USE methanol to make its volume reach to the mark; MIX it uniformly to prepare for use.

B.5 Operation steps

B.5.1 Commissioning instrument

FOLLOW the operation manual of the automatic Karl Fischer titration device to install and commission the instruments.

B.5.2 Calibrating Karl Fischer reagent

B.5.2.1 Karl Fischer reagent requires daily calibration.

B.5.2.2 It may use either the water standard solution OR the water-methanol standard solution to make calibration.

B.5.2.3 PIPETTE 25 mL to 50 mL of methanol or any other suitable solvent into a water-free and clean titration vessel; PRE-TITRATE the solvent in accordance with the instruction manual of the titration device, until to the titration end point.

B.5.2.4 USE the mass method to weigh the water standard solution or USE the volume method to pipette the water-methanol standard solution.

- a) USE the mass method to weigh the water standard solution: USE the micro-syringe to draw about 20 μ L of water standard solution; USE the filter paper to wipe off the water which is attached to the needle tip; PLACE it into the analytical balance which matches the instrument to weigh it; then quickly TAKE off the plug from the sample injection inlet; INJECT it into the titration vessel (attention: the last drop of water shall be drawn into the syringe, AND do not leave it on the needle tip); PLACE the micro-syringe back again into the analytical balance to weigh it; USE the weighing method to weigh the mass of the water standard solution which is actually added, accurate to 0.001 g; INJECT it into the instrument; SET and INITIATE the parameters and procedures which are used during calibration by water, to perform automatic titration until to the end point. Karl Fischer reagent consumption and water equivalent results will be automatically displayed or printed out.
- b) USE the volume method to draw 2.00 mL of water-methanol standard solution; quickly OPEN the plug of the sample injection inlet; INJECT it into the titration vessel; SET and INITIATE the parameters and procedures which are used during calibration by water-methanol standard solution, to perform automatic titration until to the end point. Karl Fischer reagent consumption and water equivalent results will be automatically displayed or printed out.

The reagents used in this test method, unless otherwise specified, refer to the analytical pure; AND the water used shall comply with the level 2 and above requirements as specified in GB/T 6682-2008 unless otherwise specified.

C.1.4.1 Nitric acid solution (1 + 3): MEASURE 1 volume of concentrated nitric acid; MIX it uniformly with 3 volumes of water.

C.1.4.2 5% nitric acid solution: MEASURE 7.7 mL of 65% concentrated nitric acid; USE water to make its volume reach to 100 mL.

C.1.4.3 Acetone.

C.1.4.4 Sodium chloride reference solution [$c(\text{NaCl}) = 0.01 \text{ mol/L}$]: WEIGH 0.5844 g of the reference reagent sodium chloride (OR the analytical pure sodium chloride which had been burnt at $500^\circ\text{C} \sim 600^\circ\text{C}$ to constant volume), accurate to 0.0001 g; first USE a small amount of water to dissolve it; then TRANSFER it all into a 1000 mL volumetric flask; USE water to make its volume reach to the mark; SHAKE it uniformly; PREPARE for use.

C.1.4.5 Silver nitrate standard titration solution [$c(\text{AgNO}_3) = 0.02 \text{ mol/L}$].

C.1.4.5.1 Preparation: WEIGH 3.40 g of silver nitrate, accurate to 0.01g; first USE a small amount of water to dissolve it; then TRANSFER it all into a 1000 mL volumetric flask; USE water to make its volume reach to the mark; SHAKE it uniformly; PRESERVE it in the dark (OR preserved in brown reagent bottle); PREPARE for use.

C.1.4.5.2 Calibration

C.1.4.5.2.1 PIPETTE 10.0 mL of 0.01mol/L sodium chloride reference solution in a 50 mL beaker; ADD 0.2 mL of (1 + 3) nitric acid solution and 25 mL of acetone. IMMERSE the glass electrode and silver electrode in the solution; PLACE the magnetic stir bar in; START the stirrer to start stirring. First USE the burette to add the silver nitrate standard titration solution (V) which is about 90% of the required amount; DETERMINE the potential E of this solution. MEASURE the potential every time adding 1 mL afterwards. When approaching to the end point or reaching to the end point, MEASURE the potential every time adding 0.1 mL afterwards. CONTINUE titration until the potential change is on apparent. RECORD the volume and potential every time adding the silver nitrate standard titration solution.

Note: After measuring a sample, it is required to use water to rinse the glass electrode and silver electrode, AND to use the cotton ball impregnated with 5% nitric acid solution to wipe the silver electrode.

C.2.4.5 Mercury nitrate standard titration solution II (1 mL is equivalent to 0.25 mg of Cl⁻).

PIPETTE the mercury nitrate standard titration solution (1 mL equivalent to 0.5 mg of Cl⁻); USE water to accurately dilute and prepare it ($T_2 = \frac{1}{2} T_1$).

C.2.5 Operating procedures

C.2.5.1 Sample treatment

C.2.5.1.1 PIPETTE 100.0 mL of sample into a clean porcelain evaporator dish; PLACE it over the boiling water bath to evaporate it dry.

C.2.5.1.2 As for the evaporation dish after being evaporated dry, first USE about 100 mL of water to soak it for about 20 min; carefully TRANSFER the soaking solution into a 250 mL volumetric flask; USE water to rinse it for several times, to ensure that the chloride ion in the evaporation dish is completely dissolved AND transferred into this volumetric flask; USE water to make its volume reach to the mark; SHAKE it uniformly to prepare for use, which is the sample solution.

C.2.5.2 Determination

C.2.5.2.1 PIPETTE 100.0 mL of sample solution into a 250 mL conical flask; ADD 5 drops of the mixed indicator; ADD the nitric acid solution (1 + 65) dropwise to adjust the acidity of the sample solution, until the solution changes from blue to green; and ADD another 1 mL.

C.2.5.2.2 Under constant shaking, USE the mercury nitrate standard titration solution to titrate it; when approaching to the end point (the solution color is changed from yellow green to dark green), SLOW down the titration speed; AND when the solution color changes into purple, it indicates the end point; RECORD the volume of the mercury nitrate standard titration solution consumed (V_4).

C.2.5.2.3 When the chloride content of the sample solution is greater than 10 mg/L, directly USE the mercury nitrate standard titration solution I (1 mL of titer equivalent to 0.5 mg/L) and a 10 mL burette for titration; AND when the chloride content in the sample solution is less than or equal to 10 mg/L, it shall use the mercury nitrate standard titration solution II (1 mL of titer equivalent to 0.25 mg/L) and a 5 mL micro-burette.

C.2.5.3 Blank test

Appendix D

(Normative)

Method for determination of acidity of denatured fuel ethanol

D.1 Scope of application

This method is suitable for the determination of the acidity of denatured fuel ethanol.

D.2 Method summary

In accordance with the principle of acid and alkali neutralization reaction, phenolphthalein is used as the indicator reagent AND the sodium hydroxide standard titration solution is used to titrate the sample to light red, AND if it is maintained for 30 s without fading, it indicates the end point.

D.3 Instrument

D.3.1 Stoppered conical flask: 250 mL.

D.3.2 Pipettes: 0.5 mL and 50 mL.

D.3.3 Alkaline burette: 5 mL.

D.4 Reagents and solutions

The reagents used in this test method are of analytical pure when they are not specified in other specifications. The water used shall comply with the level 3 water requirements in GB/T 6682-2008 unless otherwise specified.

D.4.1 Phenolphthalein indicator solution (10 g/L): Prepared in accordance with GB/T 603.

D.4.2 Carbon dioxide-free water: Prepared in accordance with GB/T 603.

D.4.3 Sodium hydroxide standard stock solution [$c(\text{NaOH}) = 0.1 \text{ mol/L}$]: Prepared and calibrated in accordance with GB/T 601.

D.4.4 Sodium hydroxide standard titration solution [$c(\text{NaOH}) = 0.05 \text{ mol/L}$]: In case of use, USE the carbon dioxide-free water to accurately dilute the 0.1 mol/L sodium hydroxide standard stock solution for 2 times.

D.5 Operating procedure

Appendix E

(Normative)

Method for determination of copper content in denatured fuel ethanol (atomic absorption graphite furnace method)

E.1 Scope of application

E.1.1 The method is applied to use the atomic absorption spectrometry graphite furnace method for direct sample injection to determine the content of copper in the denatured fuel ethanol.

E.1.2 The copper content range as determined by the atomic absorption graphite furnace method is 0.005 mg/L ~ 0.1 mg/L.

E.2 Method summary

An atomic absorption spectrometer with a graphite furnace is used to directly inject the sample onto the platform of the graphite tube to evaporate it dry, ash it, and atomize it, AND the absorbance value of the copper atom at the wavelength of 324.7 nm during atomization, which is compared with the standard working curve to determine the copper content.

E.3 Instruments and equipment

E.3.1 Atomic absorption spectrometer: it is used at wavelengths of 324.7 nm AND has a background correction function.

E.3.2 Copper hollow cathode lamp: single element lamp is suitable, BUT multi-element lamp can also be used.

E.3.3 Graphite furnaces: it is able to reach to a sufficient temperature to atomize the element under determination, AND it also has the fast temperature rise and cooling function.

E.3.4 Graphite tube: matched with the graphite furnace. In order to avoid absorption peak tailing, it is recommended to use pyrolytic coated graphite tubes.

E.3.5 Micro-pipette: the micro-pipette having a replaceable suction tip, the specification range is 1 μ L ~ 100 μ L, AND it is selected as required. It is recommended to use the automatic sample injector.

E.5.3 DRAW a standard working curve.

E.5.3.1 Respectively PIPETTE 0 mL, 0.50 mL, 1.00 mL, 1.50 mL, and 2.00 mL of copper standard use solution in a 50 mL volumetric flask; USE the anhydrous ethanol to make its volume reach to the mark; AND the copper content is respectively 0.00 mg/L, 0.010 mg/L, 0.020 mg/L, 0.030 mg/L and 0.040 mg/L.

E.5.3.2 In accordance with the usual operating procedures, SET the appropriate injection volume and operating parameters; INJECT the standard use solution into the graphite furnace device; AND the instrument will automatically record the absorbance value, draw the standard working curve (or establish a linear regression equation), and display and print the test results.

E.5.4 Determination: Under the same conditions of drawing the standard working curve, INJECT the same volume of sample into the graphite furnace device; AND the instrument will automatically record the absorbance value, draw the standard working curve (or establish a linear regression equation), and display and print the test results. For sample with higher copper content, it may be determined after being appropriately diluted by the anhydrous ethanol.

Note 1: Note that the drying temperature shall not be set too high, so as to avoid the sample from boiling splash, thus reducing the accuracy.

Note 2: When preparing standard sample OR diluting sample, it shall select the anhydrous ethanol having lower copper content as solvent.

E.6 Calculation

E.6.1 CHECK the standard working curve (or calculation by linear regression equation) to obtain the copper content (mg/L) of each sample.

E.6.2 For samples with higher copper content, it is calculated in accordance with the formula (E.1):

$$\rho(\text{Cu}) = \rho(\text{A}) \times n \quad \cdots \cdots \cdots (\text{ E. 1 })$$

Where:

$\rho(\text{Cu})$ - The copper content in the sample, in milligrams per liter (mg/L);

$\rho(\text{A})$ - The copper content of the diluted sample as obtained by directly reading or checking the standard operating curve (or calculation by a linear regression equation), in milligrams per liter (mg/L);

n - The dilution factor of the sample.

Appendix F

(Normative)

Method for determination of pHe of denatured fuel ethanol

F.1 Scope of application

F.1.1 This method is suitable for the determination of the acid strength of denatured fuel ethanol (or fuel ethanol) at a high concentration of ethanol content [70% (by volume fraction) or more].

F.1.2 The pHe of the alcohol solution cannot be directly compared to the pH of the water solution.

F.1.3 Determination of pHe depends on the degree of mixing of the denatured fuel ethanol, the stirring speed, and the time of action of the electrode in the solution.

F.2 Method summary

F.2.1 SOAK the composite electrode in the test solution to form an original battery, its electromotive force is related to pHe; AND the pHe of the denatured fuel ethanol can be determined by the electromotive force of the acidity meter.

F.2.2 Activation of the composite electrode is a necessary step to ensure the accuracy of the measurement. Therefore, the electrode shall be immersed in a standard buffer solution of pH = 7 after each sample is measured, to prepare for measuring the next sample.

F.2.3 During the entire analysis, since the solvent will affect the reading of the electrode, it is required to record the pHe reading at 30 s.

F.3 Instruments and equipment

F.3.1 pH meter: A acidometer (OR pH meter) having a special ion electrode system suitable for the determination of the pHe of denatured fuel ethanol, having a relative high resistance, AND being able to perform temperature compensation, AND the accuracy is 0.01 pH units.

F.3.2 Composite electrode: because the electrode has a greater impact on the determination of the results, it is required to select the electrode of high precision and good reproducibility (the same model).

soaked in the 1 mol/L sodium hydroxide solution and 1 mol/L sulfuric acid solution (or 1 mol/L hydrochloric acid solution) for several times in turn, 30 s for each soaking operation. After soaking, TAKE out the electrode, USE water to rinse it in other vessels; USE the filter paper to absorb the water attached to the outside of the electrode. Then IMMERSE the electrode in the standard buffer solution at pH = 7.

F.5.2 Calibration

F.5.2.1 OPEN the pH meter; CONNECT the electrodes and the temperature compensator in accordance with the manufacturer's instruction manual; MAKE pre-heating and temperature compensation for a certain period of time, to make the standard buffer solution and the electrode be heated in an equalized manner. When performing temperature compensation manually, it shall adjust the temperature compensator of the instrument to make it consistent with the temperature of the sample under determination.

F.5.2.2 TAKE 50 mL of standard buffer solution of pH = 6.86 into a 100 mL beaker; INSERT the electrode; CONNECT the automatic temperature compensator; PLACE in a magnetic stir bar; START stirring operation; ADJUST the acidometer to make its pH value at the standard pH value under this temperature (CHECK the manufacturer's temperature calibration table or GB/T 9724); TAKE out the electrode; USE water to rinse it in other vessel; USE the filter paper to absorb the water attached to the outside of the electrode.

F.5.2.3 TAKE 50 mL of standard buffer solution of pH = 4.00 into a 100 mL beaker; INSERT the activated composite electrode; CONNECT the temperature compensator; START stirring; ADJUST the acidometer to make its pH value at the standard pH Value under this temperature (same as F.5.2.2). TAKE out the electrode; USE water to rinse it in other vessel; USE the filter paper to absorb the water attached to the outside of the electrode.

F.5.2.4 After calibration, the slope of the acidometer shall be in the range of 95% ~ 100%, otherwise the electrode will need to be re-cleaned and activated or replaced. TAKE out the electrode; USE water to rinse it in other vessel; USE the filter paper to absorb the water attached to the outside of the electrode. PLACE the electrode back into the standard buffer solution with a pH of 7.

F.5.3 Determination

F.5.3.1 PIPETTE 50 mL of the sample into a 100 mL beaker; PLACE in the magnetic stir bar; PLACE the beaker on the magnetic stirrer; START stirring; ADJUST the stirring speed to make it produce a small vortex of 6 mm ~ 8 mm deep. INSERT the temperature compensator to ensure that the sample temperature is measured at $22\text{ }^{\circ}\text{C} \pm 2\text{ }^{\circ}\text{C}$.

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