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Replacing GB 338-2004

Methanol for industrial use

工业用甲醇

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Foreword

Provisions in Chapter 6.1 of this Standard are mandatory, and the others are recommendatory.

This Standard is drafted according to the rules given in GB/T 1.1-2009.

This Standard replaces GB 338-2004 *Industrial Methanol*. Compared with GB 338-2004, the main technical changes in this Standard are as follows:

- Modified the preparation method of this Standard colorimetric solution used for the potassium permanganate time test (see Chapter 4.7 of this edition; and Chapter 4.4 of Edition 2004);
- Added the correction formula for the readings of the full immersion thermometer and the mercury barometer used in the boiling range determination method (see Annex C of this edition);
- Modified the test method for the determination of ethanol content (see Chapter 4.14, Annex D of this edition; and Chapter 4.11 of Edition 2004);
- Modified the factory inspection items (see Chapter 5.1 of this edition; and Chapter 5.1 of Edition 2004).

Using re-drafting method, this Standard modifies-adopts the American Society for Testing Materials standard ASTM D1152:2006 *Methanol*. Compared with ASTM D1152:2006, there are adjustments on the structure. Annex A lists out the Contrast List between chapter and article number in this Standard and ASTM D1152:2006. The technical differences and their reasons between this Standard and the ASTM D1152:2006 are as follows:

- Indicators are divided into superior products, first-class products and qualified products (see Chapter 3.2). Which is determined based on the classification guideline of the relevant industrial products in China;
- Added the water miscibility item and the ethanol content item (see Chapter 3.2). This is to strictly control the product quality;
- The color scale, boiling range, acid or alkali and other indicators of the superior products are better than those specified in ASTM D1152:2006 (see Chapter 3.2);
- In addition to the use of the method specified in the ASTM D1152:2006 for the sulfuric acid washing test, other test methods all adopt the national standards for products testing method of China (see Chapter 4).

Methanol for Industrial Use

1 Scope

This Standard specifies the requirements, test methods, inspection rules, marking, packaging, transportation, storage and safety of the methanol for industrial use.

This Standard applies to the methanol for industrial use compounded by coal, natural gas, light oil and heavy oil as the materials.

Molecular formula: CH_4O

Relative molecular mass: 32.042 (according to the international relative atomic mass in 2007)

2 Normative references

The articles contained in the following documents have become part of this Standard when they are quoted herein. For the dated documents so quoted, all the modifications (including all corrections) or revisions made thereafter shall be applicable to this Standard.

GB 190 Packing symbol of dangerous goods

GB/T 601 Chemical reagent - Preparations of standard volumetric solutions

GB/T 603 Chemical reagent - Preparations of reagent solutions for use in test methods (GB/T 603-2002, ISO 6353-1:1982, NEQ)

GB/T 3143 Color determination method of liquid chemicals (Hazen unit; Platinum-cobalt scale)

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

GB/T 6283 Chemical products - Determination of water Karl ·fischer method (general method) (GB/T 6283-2008, ISO 760:1978, NEQ)

GB/T 6324.1 Test method of organic chemical products - Part 1: Water miscibility test of liquid organic chemical products

GB/T 6324.2 Test method of organic chemical products - Part 2: Determination of dry residue after evaporation on a water bath for volatile organic liquids (GB/T 6324.2-2004, ISO 759:1981, MOD)

Note: See Annex B when there is a need to calculate the mass fraction of methanol.
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a Including $64.6^{\circ}\text{C} \pm 0.1^{\circ}\text{C}$.
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4 Test methods

4.1 Warning

Some processes specified by the test method may lead to dangerous situations, so the operator shall take appropriate safety and protective measures.

4.2 General provisions

Unless otherwise specified, only confirmed analytically pure reagent and Grade-3 water met the GB/T 6682 are used in the analysis.

If there is no other requirements noted, the standard titration solution, preparations and products used in the analysis shall be prepared in accordance with provisions of GB/T 601 and GB/T 603.

4.3 Determination of the characters

Add the specimens in the colorimetric tube with a plug; carry out a sensory detection under fluorescent lamp or daylight.

4.4 Determination of the color scale

It shall be conducted according to the method specified in GB/T 3143.

4.5 Determination of density

It shall be conducted according to the pycnometer method specified in GB/T 4472. Other test methods that can meet the analysis requirements can also be used. In the range of $15^{\circ}\text{C} - 35^{\circ}\text{C}$, the temperature correction coefficient of the specimen density is $0.000\ 93\ \text{g} / (\text{cm}^3 \cdot ^{\circ}\text{C})$.

Take the arithmetic mean value of two parallel determination results as the report results. The absolute difference value of two parallel determination results shall not be greater than $0.0005\ \text{g}/\text{cm}^3$.

4.6 Determination of boiling range

It shall be conducted according to the method specified in GB/T 7534. Distillation flask is of 100mL; indication range of the thermometer is $50^{\circ}\text{C} - 70^{\circ}\text{C}$, with the division value of 0.1°C . For the correction of the full immersion thermometers and mercury manometer, see Annex C.

4.7 Potassium permanganate test

It shall be conducted according to the provisions of visual colorimetric method in GB/T 6324.3-2011. Among which, the specimen volume is of 50mL; the addition of the potassium permanganate solution is 1mL; temperature is controlled at $15^{\circ}\text{C} \pm 0.5^{\circ}\text{C}$; the colorimetric method is lateral observation.

4.8 Water miscibility test

It shall be conducted according to the method specified in GB/T 6324.1. The proportions of the selected specimen and the water miscibility respectively are: 1 + 3 (superior products), 1 + 9 (first-class products).

4.9 Determination of the moisture

It shall be conducted according to provisions of GB/T 6283.

Take the arithmetic mean value of two parallel determination results as the determination results. The absolute difference value of two parallel determination results shall not be greater than 0.01%.

4.10 Determination of acid or alkali content

4.10.1 Method summary

The specimen is diluted by water that does not contain carbon dioxide, with the bromothymol blue as indicator; for the acidic specimen, the sodium hydroxide standard titration solution is used to titrate the free acid, and for the alkaline specimen, the sulfuric acid standard titration solution is used to titrate the free alkali.

4.10.2 Reagents

4.10.2.1 Sodium hydroxide standard titration solution: $c(\text{NaOH})=0.01 \text{ mol/L}$.

4.10.2.2 Sulfuric acid standard titration solution: $c(1/2\text{H}_2\text{SO}_4) = 0.01 \text{ mol/L}$.

4.10.2.3 Bromothymol blue indicating liquid: 1 g/L.

4.10.2.4 Water without carbon dioxide.

4.10.3 Apparatuses

Burette: 10mL, with a scale division of 0.05mL.

4.10.4 Analytical procedures

4.10.4.1 The specimen is diluted by isometric water that does not contain carbon dioxide; add 4 - 5 drops of bromothymol blue indicating liquid to identify; in case the specimen is yellow, determine the acid contents; in case the specimen is blue, and determine the alkali contents.

5.4 Sampling shall be conducted according to the provision of GB/T 6678 and GB/T 6680 that the liquid is flowing under the normal temperature; the total amount of specimen shall not be less than 2L. After mixing the specimen thoroughly, respectively pack them in two dry and clean glass bottles with ground stopper; one bottle is used for partition test, another for reserved inspection.

5.5 The determination of test results shall be conducted according to rounding-off value comparison method in GB/T 8170. For any indicator that does not meet the requirements of this Standard, it shall be re-sampled and re-inspected from two times amount of the packaging unit; for canned products, it shall be multi-point sampled and inspected. Even if there is only one indicator of the re-inspection result not meeting the requirements of this Standard, then the entire batch of products are deemed as unqualified.

6 Marking, packaging, transportation and storage

6.1 Packaging containers of methanol for industrial use shall be coated with firm marking, with the contents including manufacturer name, product name, this Standard number, and the “Flammable Liquid” and “Poisonous Substance” marking specified by GB 190.

6.2 The methanol for industrial use shall be packaged with clean and dry containers, and packaging containers shall be strictly sealed.

6.3 The transport of the methanol for industrial use shall comply with the relevant provision of the transport of dangerous chemicals.

6.4 The methanol for industrial use shall be stored dry, ventilated and cool places which are away from sun exposure, heat and fire.

7 Safety

7.1 Warning against danger: Methanol is a kind of colorless, flammable liquid with a flash point of 8°C, auto-ignition temperature of 436°C. The volume fraction of explosion range in air for the methanol vapor is 6% - 36.5%. Methanol vapor has stimulation on nervous system, if inhaled; it may cause blindness and poisoning. If ingested wrongly, it will cause drunken feeling, headache, nausea, emesis, blurred vision; it will cause in severe cases blindness and even death.

7.2 Safety measures: Wash with water immediately if methanol spills out. When methanol catches fire, put out fire with sand, foam extinguisher, asbestos cloth, etc.. Avoid methanol contact with skin; if it spills on the skin and eyes, wash with plenty of water and rapid for medical treatment. After ingested wrongly, conduct gastric lavage with 1% - 2% sodium bicarbonate solution. At the same time, in order to prevent the methanol metabolism, take 50% ethanol solution of food grade, with average 0.5mL per kilogram of

Annex D

(Normative)

Determination of ethanol content in methanol for industrial use – Gas chromatography

D.1 Method summary

Under the selected operating conditions, the specimen goes through capillary-column chromatography after being vaporized; all constituents can be separated, detected by a flame ionization detector. Quantify by the internal standard method; then calculate the mass fraction of ethanol.

D.2 Reagents

D.2.1 Methanol.

D.2.2 Isopropyl alcohol: Internal standard substance; chromatography pure.

D.2.3 Hydrogen: Volume fraction is not less than 99.9%, and conduct drying and purification by silica gel and molecular sieve.

D.2.4 Nitrogen: Volume fraction is not less than 99.95%, and conduct drying and purification by silica gel and molecular sieve.

D.2.5 Air: Conduct drying and purification by silica gel and molecular sieve.

D.3 Apparatuses

D.3.1 Gas chromatograph: Equipped with a flame ionization detector, the whole sensitivity and stability complies with the relevant provisions of GB / T 9722;

D.3.2 Recorder: Chromatographic data processor or chromatography workstation;

D.3.3 Specimen injector: 1 μ L and 25 μ L micro injectors.

D.3.4 Chromatographic columns and typical chromatography operating conditions

Recommended capillary-column chromatography and typical chromatography operating conditions are shown in Table D.1. A typical capillary-column chromatography is shown in Figure D.1. Other chromatographic columns and chromatography operating conditions that can achieve the same degree of separation can be also used.

D.4.1.1 Add 0.50mL of ethanol into a clean and dried 100mL volumetric flask with the known weight, accurate to 0.0001g; dilute to the mark line with methanol; weigh; and mix.

D.4.1.2 Respectively add 0.00mL, 0.20mL, 0.40mL, 0.80mL, 1.60mL of the above-mentioned mother-liquor into 5 clean and dried 50mL volumetric flasks; then respectively add 25 μ L of isopropyl alcohol with the known mass; dilute to the mark line with methanol; mix well. After the operating conditions of apparatuses are being stabilized, respectively conduct the standard injection analysis. Injection amount is 1 μ L.

D.4.2 Determination of the relative correction factor

D.4.2.1 Start gas chromatograph; adjust the apparatuses based on chromatographic operating conditions listed in Table D.1.

D.4.2.2 After the apparatuses is stabilized, pipette 1 μ L of standard solution (D. 4.1.2) with a micro-syringe; inject specimen, and analyze.

D.4.2.3 Calculation of the relative correction factor f'

The relative correction factor f' of ethanol is calculated according to Equation (D.1):

$$f' = \frac{A_s \times m_i}{A_i \times m_s} \quad \text{..... (D.1)}$$

Where:

A_i ; — The value of peak area of ethanol constituent;

m_s — The mass value of isopropyl alcohol, the unit is in g;

A_s — The value of peak area of isopropyl alcohol;

m_i — The mass value of ethanol, the unit is in g.

D.4.3 Specimen determination

D.4.3.1 Add the appropriate amount of specimens into a 50mL volumetric flask with the known mass. Pipette 25 μ L of the internal standard substance isopropyl alcohol with injection syringe, and weigh it; then add the isopropyl alcohol into the volumetric flask. Fix the volume, mix well and weigh.

D.4.3.2 After the apparatuses is stabilized, pipettes 1 μ L of specimen (D.4.3.1) with a micro-syringe; inject specimen, and analyze.

D.5. Result calculation

The value of mass fraction w of ethanol is expressed in %, and calculated according to Equation (D.2):

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