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Test methods for glycerin

甘油试验方法

(ISO 1615:1976, ISO 1616:1976, ISO 2096:1972, ISO 2099:1972, ISO 2211:1973, ISO 2879:1975, MOD)

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Test methods for glycerin

1 Scope

This standard specifies the sampling method of barreled glycerol, as well as the method for the determination of its transparency, odor, color, density at 20 °C, content, chloride limits, sulfated ash, acidity or alkalinity, saponification equivalent, arsenic limits, heavy metal limits, reduction substances.

2 Normative references

The provisions in following documents become the provisions of this Standard through reference in this Standard. For the dated references, the subsequent amendments (excluding corrections) or revisions do not apply to this Standard; however, parties who reach an agreement based on this Standard are encouraged to study if the latest versions of these documents are applicable. For undated references, the latest edition of the referenced document applies.

QB/T 2739-2005 Preparations of standard volumetric solutions of general test methods for washing products

3 Terms and definitions

The following terms and definitions apply to this standard.

3.1

Color and luster unit for Hazen

The color of solution, each liter of which contains 1 mg of platinum (calculated as chloroplatinic acid) and 2 mg of cobalt chloride hexahydrate.

3.2

Density at 20 °C

The mass of material per unit volume at 20 °C, expressed in grams per milliliter.

4 Sampling method of barreled glycerin

4.1 General

This method is suitable for refined glycerol, which has no solid sedimentation or suspended matter in the barrel. It is also applicable to barreled refined glycerin, that has been frozen and can return to its original state after being warmed.

Samples for laboratory measurement are prepared and stored according to this method.

4.2 Principle

Insert the sampling tube from the plug hole to the bottom of the barrel. Take samples from the entire depth of the barrel. Take an equal amount of samples from each sample barrel. Combine all samples from the same batch. Mix evenly. Divide the samples into the required number of laboratory samples.

4.3 Instruments

4.3.1 Sampling tube

As shown in Figure 1. It consists of two cylinders made of stainless steel or other chemical-resistant materials. The inner cylinder and the outer cylinder are closely matched. There are two rows of staggered and intermittent longitudinal grooves on each of the two cylinders. The width of the groove accounts for one quarter of the circumference of the cylinder; the length of the grooves is equally distributed over the entire length of the cylinder. The grooves on the inner cylinder and the outer cylinder can be exactly coincident or sealed, by turning the inner cylinder handle with a pointer. The position indicated by the pointer on the scale, which is equipped on the outer cylinder, means the relative position of the grooves on the inner cylinder and the outer cylinder. In the "filling" position, the inner and outer grooves form two rows of staggered openings, allowing samples from all depths in the barrel to enter the sampling tube at the same time.

There are holes drilled in the bottom of the inner and outer cylinders. When the pointer is in the "empty" position, the bottom holes overlap to form an opening, whilst the longitudinal groove remains sealed.

The length of the sampling tube shall be proportional to the depth of the material to be sampled; its effective volume shall be approximately 0.1% of the barrel volume.

4.3.2 Wipe plug

Match the plug hole of the barrel to be sampled.

4.3.3 Cylindrical collector

It is made of the same material as the sampling tube, but preferably glass; it is equipped with a sealing cover, has a volume of about 1.5 L, can be applied to each ton of product to be sampled.

4.4.1.1 Containers used for mixing and storing samples shall be sealed. The containers shall be kept closed during filling AND in the operation space for taking each sample.

4.4.1.2 When sampling, the container shall be shielded as much as possible, especially to prevent rain and other accidental contamination.

4.4.1.3 All instruments and containers shall be clean and dry when used.

4.4.1.4 The laboratory sample, which is obtained by dividing the mixed sample, shall completely fill the sample bottle (4.3.4).

4.4.2 Sample preparation

Insert the sampling tube (4.3.1), whose groove hole is closed, into the bottom of the barrel, through the wiper plug (4.3.2). Rotate the handle, to move the pointer to the "filling" position. Open the longitudinal groove. Close the groove hole, after the sampling tube is full. Pull out the sampling tube. Use the wiper plug to clean the outer wall of the tube.

Insert the sampling tube, which is filled with glycerin, into the collector (4.3.3). Rotate the handle, to move the pointer to the "empty" position. Empty the sampling tube. In this way, take equal amounts of samples from each sample barrel in sequence, so that the total amount is greater than the required amount. In the interval between the two emptying operations, it must keep the collector closed.

Seal the sample container. Lie down and roll it. Quickly mix all the sample. Immediately take about 500 g (or other required amounts) of the sample and put it into a sample bottle (4.3.4), thus preparing the required number of sets of the same laboratory sample. Tighten the stopper or sealing cap. Seal with sealing wax (or glue). Attach the sample label, which indicates sample name, batch number, specifications, sampling date, signature of the sampler.

If the glycerin in the sample barrel has been frozen due to freezing, the barrel shall be gently heated and rolled over, to allow the glycerin to thaw and mix evenly, before sampling as described above.

5 Determination of transparency

5.1 Instruments

Commonly used laboratory instruments, as well as the following:

5.1.1 Nessler colorimetric tube, 50 mL.

5.1.2 Milky white electric lamp.

5.2 Procedure

Mix the glycerol sample evenly. Use vacuum or ultrasonic degassing. Take 50 mL and place it in a Nessler colorimetric tube. Observe it at room temperature, under a milky white electric lamp. Then place it in front of a white curtain to observe with reflected light. If there is no turbidity, it is judged as transparent.

6 Determination of odor

Place a small amount of glycerin sample on the back of your hand and smear it. Smell it. If there is only the special smell of glycerin and no other odor, it is judged to have no odor.

7 Determination of color

7.1 Principles

Visually compare the color of the specimen with the color of the platinum-cobalt standard colorimetric solution. Express the results in color and luster unit for Hazen (platinum-cobalt colorimetric).

7.2 Reagents

Unless otherwise stated, only reagents confirmed to be of analytical grade and distilled or deionized water or water of equivalent purity are used in the analysis.

Note: Applicable to all tests in this standard.

7.2.1 Cobalt chloride hexahydrate ($CoCl_2 \cdot 6H_2O$) (GB/T 1270).

7.2.2 Potassium chloroplatinate (K_2PtCl_6).

7.2.3 Hydrochloric acid (GB/T 622), density (ρ_{20}) is about 1.19 g/mL.

7.3 Instruments

Commonly used laboratory instruments, as well as the following:

7.3.1 Spectrophotometer, wavelength range 420 nm ~ 800 nm.

7.3.2 Nessler colorimetric tube, 50 mL or 100 mL, which has a line mark 100 mm above the bottom.

7.3.3 Colorimetric tube rack. Usually the colorimetric tube support plate is white. It is best to install fluorescent lighting, to improve the effect of observing colors.

7.4 Procedure

prepared according to 4.4 in QB/T 2739-2005.

9.3.2.4 Sodium formate ($NaCHO_2$) (HG 3-966), which is 68 g/L solution.

9.3.2.5 Sodium periodate ($NaIO_4$) (HG 3-1157), which is 60 g/L acidic solution.

a) Preparation of acidic solution:

Weigh 60 g of sodium periodate (accurate to 0.1 g). Dissolve it in about 500 mL of water (9.3.2.1), to which 120 mL of sulfuric acid solution (9.3.2.3) has been added. Cool it whilst adding. Transfer it into a 1000 mL volumetric flask. Use water to dilute it to the mark. Mix well. If necessary, use a glass filter to filter it.

b) Acidity check of solution:

The volume of sodium hydroxide standard titration solution (9.3.2.7), which is used in the blank test (9.3.4.4), shall be no less than 4.5 mL. It is equivalent to the acidity, which is produced in the basic reaction (9.3.1).

c) Storage of solution:

The solution is stored in a brown glass bottle, which has a ground glass stopper.

9.3.2.6 Sodium hydroxide (GB/T 629), about 0.05 mol/L solution, which is prepared according to 4.1 in QB/T 2739-2005.

9.3.2.7 Sodium hydroxide (GB/T 629), c(NaOH) = 0.125 mol/L standard titration solution, without carbon dioxide, which is prepared and calibrated according to 4.1 in QB/T 2739-2005.

9.3.2.8 Phenolphthalein (GB/T 10729) indicator solution, 10 g/L ethanol solution, which is prepared according to 5.1 in QB/T 2739-2005.

9.3.3 Instruments

Commonly used laboratory instruments, as well as the following:

9.3.3.1 Burette, 50 mL.

9.3.3.2 pH meter, sensitivity 0.02 pH, which is equipped with glass measuring electrode and calomel reference electrode. The buffer solution is:

a) Potassium hydrogen phthalate (GB/T 6857), c[$C_6H_4(COOK)(COOH)$] = 0.05 mol/L (10.12 g/L) solution, which has a pH of 4.00 at 20 °C;

b) Disodium tetraborate decahydrate (GB/T 6856), c($Na_2B_4O_7 \cdot 10H_2O$) = 0.01 mol/L (3.81 g/L) solution, which has pH of 9.22 at 20 °C.

9.3.4 Procedure

The presence of carbon dioxide can cause errors. When placing it, it is best to cover the container containing the test solution with a watch glass. Avoid simultaneous operations, that will increase the carbon dioxide content in the laboratory air.

9.3.4.1 Test portion

Weigh the test portion, which has a glycerin content of no more than 0.50 g (accurate to 0.0001 g). If the approximate content of glycerin is not known, it shall weigh 0.50 g of the specimen, for prediction.

If the glycerol content is greater than 75%, it is best to weigh (5.0 ± 0.1) g of the specimen (accurate to 0.0001 g). Place it in a 500 mL volumetric flask. Use water (9.3.2.1), to dilute it to the mark. Mix well. Transfer 50.0 mL for determination.

9.3.4.2 Preparation of test solution

For alkaline specimens or when tar precipitation occurs after acidification of the specimen, the test portion (9.3.4.1) can be put into a flask, which is equipped with a reflux condenser. Dilute it to 50 mL if necessary. Add 2 drops of phenolphthalein indicator solution (9.3.2.8). Use sulfuric acid solution (9.3.2.3) to neutralize it, until it just fades. Add another 5 mL. Boil it for 5 minutes. Cool it. Filter if necessary. Use water (9.3.2.1) to rinse the filter. Transfer the solution quantitatively into a 600 mL beaker.

In the absence of the above situation, place the test portion (9.3.4.1) directly into the 600 mL beaker.

9.3.4.3 Titration

Use water to dilute the test solution (9.3.4.2), to a volume of approximately 250 mL. Under constant stirring, use a pH meter (9.3.3.2) to indicate. Add sodium hydroxide solution (9.3.2.6). Adjust the pH to 7.9 ± 0.1.

Add 50.0 mL of sodium periodate solution (9.3.2.5) and mix gently. Cover the watch glass. Place it in a dark place, where the temperature does not exceed 35 °C, for 30 minutes.

Then add 10 mL of dilute ethylene glycol solution (9.3.2.2). Mix it. Leave for another 20 minutes under the same conditions.

Add 5.0 mL of sodium formate solution (9.3.2.4). Use a pH meter to indicate. Use sodium hydroxide standard titration solution (9.3.2.7), to titrate the pH to 7.9 ± 0.1.

9.3.4.4 Blank test

While measuring, under the same conditions, use the same amount of reagents and

10.2.1 Nitric acid (GB/T 626), (136→1000) dilute solution (by volume).

10.2.2 Silver nitrate (GB/T 670), 17 g/L solution.

10.2.3 Sodium chloride (GB/T 1253), 5 mg/L standard solution containing chloride ions (Cl^{-1}).

Accurately weigh 0.0842 g of sodium chloride, that has been burned to constant weight at 500 °C ~ 600 °C. Dissolve it in water. Transfer it into a 100 mL volumetric flask. Dilute it to the mark. Use a pipette, to accurately pipette 10.0 mL of this solution, into a 1000 mL volumetric flask. Dilute to the mark. Mix well.

10.3 Instruments

Commonly used laboratory instruments, as well as the following:

10.3.1 Volumetric flask, 50 mL.

10.3.2 Nessler colorimetric tube, 50 mL.

10.4 Procedure

10.4.1 Weigh 25.0 g of the specimen into a volumetric flask (10.3.1). Use distilled water to dilute it to the mark. Mix evenly.

10.4.2 Pipette 10.0 mL and 1.0 mL of glycerol solution (10.4.1) into two Nessler colorimetric tubes (10.3.2), respectively. Use water to dilute it to 15 mL. Add 1 mL of nitric acid solution (10.2.1). Immediately add 1 mL of silver nitrate solution (10.2.2). Shake well. Keep it in a place away from bright light for 2 minutes.

10.4.3 Pipette 10.0 mL of sodium chloride standard solution (10.2.3) into a Nessler colorimetric tube. Use water to dilute it to 15 mL. Add the same volume of nitric acid and silver nitrate solution as in 10.4.2. Shake well. Place it aside.

10.5 Results

Place the specimen tube and standard tube on the test tube rack. Face the black background, to observe and compare laterally. When the turbidity of the specimen solution is not stronger than the turbidity of the chloride standard solution, for 10.0 mL of the specimen solution pipetted, the chloride (Cl^{-1}) content is less than 0.001%; for 1.0 mL of the specimen solution pipetted, the chloride (Cl^{-1}) content is less than 0.01%.

11 Determination of sulfated ash

This method is applicable to various glycerol products, which have a sulfated ash content of no more than 0.5% (mass fraction).

$$H = \frac{m_1 - m_0}{m} \times 100\% \qquad \cdots\cdots(7)$$

Where:

m_1 - The mass of the porcelain evaporating dish containing sulfated ash, in grams (g);

m_0 - The mass of the empty evaporating dish, in grams (g);

m - The mass of the test portion, in grams (g).

The arithmetic mean of two parallel measurement results shall be expressed to three decimal places, as the measurement result.

11.5.2 Precision: The absolute difference between two independent test results, which are obtained under repeated conditions, shall not be greater than 0.005%, provided that the absolute difference greater than 0.005% shall not exceed 5%.

12 Determination of acidity or alkalinity

12.1 Principles

Use phenolphthalein as an indicator. Use hydrochloric acid or sodium hydroxide standard titration solution to titrate the test solution.

12.2 Reagents

12.2.1 Distilled water, which is boiled for at least 10 minutes, to remove carbon dioxide.

12.2.2 Sodium hydroxide (GB/T 629), c(NaOH) = 0.01 mol/L standard titration solution, which is prepared and calibrated according to 4.1 in QB/T 2739-2005.

12.2.3 Hydrochloric acid (GB/T 622), c(HCl) = 0.01 mol/L standard titration solution, which is prepared and calibrated according to 4.3 in QB/T 2739-2005.

12.2.4 95% ethanol (GB/T 679).

12.2.5 Phenolphthalein (GB/T 10729) indicator solution: Dissolve 1.0 g of phenolphthalein in 95% ethanol, to prepare a 100 mL solution. Add dropwise 0.01 mol/L sodium hydroxide solution, until pink color just appears.

12.3 Instruments

Commonly used laboratory instruments and micro-burettes without and with stoppers, which has a division of 0.01 mL.

14.2.9 Arsenic trioxide (GB/T 1256).

14.2.10 Arsenic standard solution: Dry arsenic trioxide (14.2.9) in a sulfuric acid desiccator to constant weight. Accurately weigh 0.1320 g in a 100 mL beaker. Add 5 mL of sodium hydroxide solution (14.2.5) to dissolve it. Add sulfuric acid (14.2.6) for neutralization. Then add 10 mL of sulfuric acid. Use water to transfer it to a 1000 mL volumetric flask. Dilute to the mark. Mix well. This solution is arsenic standard stock solution, which contains arsenic 0.1 mg/mL.

Accurately transfer 1.0 mL of the arsenic standard stock solution into a 100 mL volumetric flask. Add 1 mL of sulfuric acid (14.2.6). Use water to dilute it to the mark. Mix well. This solution is arsenic standard use solution, which contains arsenic 1 μg/mL. It is prepared before use.

14.3 Instruments

Commonly used laboratory instruments, as well the following:

14.3.1 Graduated cylinder, 25 mL.

14.3.2 Pipettes, 1 mL, 2 mL, 5 mL, 10 mL.

14.3.3 Volumetric flask, 100 mL, 1000 mL.

14.3.4 The arsenic measuring device, as shown in Figure 3, includes:

a) Conical flask, 100 mL;

b) Rubber stopper, which has a hole in the middle to match the arsenic measuring tube;

c) Glass arsenic measuring tube, which has a total length of 18 cm, thick at the top and thin at the bottom. The inner diameter from the mouth of the tube down to 14 cm is about 6.5 mm, then becomes tapering; the inner diameter at the end is about 1 mm ~ 3 mm. There is a small hole, which has a diameter of about 2 mm, at 1 cm from the end. The narrow part is inserted tightly into the rubber stopper, so that the lower hole protrudes from the rubber stopper by at least 3 mm. The top is a round flat tube mouth, which has a smooth surface and a hook on each side of the diameter to fix the glass cap. The thicker part, at 3 cm below the top nozzle, is equipped with 5 cm ~ 6 cm long lead acetate absorbent cotton;

d) Glass cap: the lower end surface is ground flat; there is a meniscus groove on the upper surface; there is a round hole with a diameter of 6.5 mm in the center.

When in use, insert the rubber stopper at the lower end of the glass arsenic measuring tube into the mouth of the conical flask. Cover the upper end of the glass arsenic measuring tube with a glass cap, so that the round holes match each other. Sandwich

specimen is not darker than the color of the arsenic spots in the arsenic standard solution, it is considered that the arsenic content in glycerol is not higher than 2 mg/kg.

14.4.3 Results reporting

If two parallel tests obtain the same result, that is the final result.

The test report shall indicate that the color of the arsenic spots on the specimen is darker, lighter, or equivalent to the color of the arsenic spots on the arsenic standard solution.

15 Limit test of heavy metals

15.1 Principle

When glycerol is in acetic acid solution, heavy metals and hydrogen sulfide saturated solution generate colored metal sulfide precipitates. The color of the precipitate suspended in the solution is compared with the color generated by a certain amount of lead standard solution under the same conditions.

15.2 Reagents

15.2.1 Acetic acid (GB/T 676), (58→1000) dilute solution (by volume).

15.2.2 Nitric acid (GB/T 626), (10→1000) dilute solution (by volume).

15.2.3 Lead nitrate.

15.2.4 Lead standard solution: Accurately weigh 0.1598 g of lead nitrate. Place it in a 1000 mL volumetric flask. Add 100 mL of distilled water, which contains 1 mL of nitric acid solution (15.2.2). Use water to dissolve and dilute to the mark. This stock solution contains lead 0.1 mg/mL.

Pipette 10.0 mL of lead nitrate stock solution. Place it in a 100 mL volumetric flask. Add distilled water to dilute to the mark. Mix well. This solution contains lead 10 μg/mL.

This solution shall be diluted before use. The glass containers used to prepare and store the solution must not contain lead.

15.2.5 Saturated hydrogen sulfide solution: Add dilute hydrochloric acid into a generator containing iron sulfide, to obtain hydrogen sulfide gas. Lead this gas into distilled water, to make a saturated solution. This solution shall be prepared before use.

15.3 Instruments

Commonly used laboratory instruments as well as the following:

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