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Carbon disulfide for industrial use

工业二硫化碳

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Carbon disulfide for industrial use

Warning: According to the provisions of Chapter 6 of GB 12268-2012, this product belongs to Class 3 flammable liquids and Class 6, Item 6.1 toxic substances, which require careful handling. Personnel using this document shall have practical experience in formal laboratory work. This document does not address all possible security issues. It is the responsibility of the user to take appropriate safety and health measures and to ensure compliance with the conditions which are set by the relevant national regulations. Some of the reagents used in the test method are corrosive. If they are splashed on the skin or eyes, use water to wash them immediately; in severe cases, seek medical attention immediately.

1 Scope

This document specifies the requirements, test methods, inspection rules and marking, labeling, packaging, transportation and storage of carbon disulfide for industrial use.

This document applies to carbon disulfide for industrial use.

Note: This product is mainly used in the production of viscose fiber, cellophane, pesticides, rubber additives, flotation agents, chemical raw materials and solvents, etc.

2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. For dated references, only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document.

GB/T 191-2008, Packaging - Pictorial marking for handling of goods

GB/T 615-2006, Chemical reagent - General method for the determination of distillation range

GB/T 6678, General principles for sampling chemical products

GB/T 6680, General rules for sampling liquid chemical products

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

GB/T 8170, Rules of rounding off for numerical values & expression and judgment of limiting values

6 Test methods

6.1 General provisions

The reagents and water used in this document refer to analytical reagents and grade-3 water specified in GB/T 6682-2008, unless other requirements are specified. The standard titration solutions, preparations and products used in the test shall be prepared according to the provisions of HG/T 3696.1 and HG/T 3696.3 unless other requirements are specified.

6.2 Appearance inspection

In the fume hood, pipette an appropriate amount of sample into the colorimetric tube and seal it with water; judge it visually under natural light.

6.3 Determination of distillation rate

6.3.1 Principle

Use the method of distillation to measure the distilled volume of the sample at boiling range 45.6 °C ~ 46.6 °C.

6.3.2 Instruments and apparatuses

Same as Appendix A of GB/T 615-2006.

6.3.3 Test procedure

6.3.3.1 Calculation of observed boiling range temperature

Calculate according to 6.1 in GB/T 615-2006.

6.3.3.2 Distillation

In a well-ventilated fume hood, install the distillation device as shown in Figure 1 in GB/T 615-2006; remove the outer cover of the distillation flask; replace it with a water bath for heating and distillation; use an ice-water bath to cool the outside of the receiver.

Use a clean and dry receiver to measure 100 mL ± 0.5 mL of the sample at a temperature of 5°C ~ 10°C; pour it into the distillation flask; do not let it flow into the branch pipe; add a few clean and dry zeolites; use a stopper inserted with a thermometer to close the distillation flask containing the sample. Make the axes of the thermometer and the distillation flask coincide, and make the upper end of the mercury bulb and the lower edge of the inner wall of the branch pipe of the distillation flask on the same horizontal line. Attach the auxiliary thermometer to the main thermometer; locate the mercury bulb at 1/2 of the height of the mercury column exposed outside the plug of the main thermometer. Place the receiver (without drying) at the lower end of the condenser tube, so that the mouth of the condenser tube enters the receiver part by not less than 25 mm, and is not lower than the 100 mL scale line; cover the mouth with wetted filter paper; place the receiver in a water batch at 5 °C ~10 °C.

Connect the cooling water; immerse the spherical part of the distillation flask in a water bath preheated to about 50 °C; control the time from the beginning of heating to the first drop of distillate dripping into the receiver to be 5 min ~ 10 min. Record the temperature of the main thermometer (initial boiling point) and the reading of the auxiliary thermometer when the first drop of distillate flows out. Control the temperature of the water bath at about 75 °C, and the distillation rate at 3 mL/min ~ 5 mL/min. The liquid level of the water bath shall not exceed the carbon disulfide liquid level at all times. When the thermometer reading just exceeds the final boiling point temperature (calibrated and calculated according to 6.3.3.1), immediately remove the water bath, leave it for 5 min, and record the distillate volume. When the initial boiling point temperature corrected by 6.3.3.1 is not lower than 45.6 °C, the distillate volume value is the distillation rate.

Take the arithmetic mean of the parallel determination results as the determination result; the absolute difference between the two parallel determination results shall not be larger than 0.5%.

6.4 Determination of density

6.4.1 Principle

Use a densimeter to measure the density of the sample; then, convert it to the density at 20 °C according to the formula.

6.4.2 Instruments and apparatuses

6.4.2.1 Densimeter: density range 1.240 g/mL ~ 1.300 g/mL, division value 0.001 g/mL.

6.4.2.2 Thermometer: temperature range 0 °C ~ 50 °C, division value 0.5 °C.

6.4.3 Test procedure

Inject the sample into a 200 mL graduated cylinder; put the densimeter in; read the density (reading on the lower edge of the meniscus) after the densimeter is suspended and stabilized; measure the temperature of the sample.

6.4.4 Test data processing

Calculate the density (ρ_{20}) of carbon disulfide according to Formula (1):

$$\rho_{20} = \rho_t + 0.0015(t - 20) \quad \cdots\cdots\cdots\cdots (1)$$

Where:

Take the arithmetic mean of the parallel determination results as the determination result; the absolute difference between the two parallel determination results shall not be larger than 0.001%.

6.6 Determination of iodine reduction content

6.6.1 Principle

Use iodine standard titration solution to titrate the sample, where the end point is indicated by the pink color of a slight excess of iodine in carbon disulfide solution.

6.6.2 Reagents or materials

Iodine standard titration solution: $c(1/2I_2)$ about 0.01 mol/L; use a pipette to pipette 10 mL of iodine standard titration solution prepared and calibrated according to HG/T 3696.1; put it in a 100 mL volumetric flask; use freshly boiled and cooled water to dilute to the mark; shake well; prepare just before use.

6.6.3 Instruments and apparatuses

6.6.3.1 Micro burette: graduation value 0.02 mL.

6.6.3.2 Iodine measuring bottle: 150 mL.

6.6.4 Test procedure

Use a clean and dry iodine measuring bottle to weigh about 50 g of sample, accurate to 0.1 g; use iodine standard titration solution for titration; add 1 ~ 2 drops each time; cover the bottle cap and shake for 15 s until a stable pink color appears in the carbon disulfide layer.

6.6.5 Test data processing

Calculate the iodine reduction content, by the mass fraction w_2 of hydrogen sulfide (H_2S), according to Formula (3):

$$w_2 = \frac{VcM}{1\ 000\ m} \times 100\% \qquad \cdots\cdots (3)$$

Where:

V – the value of the volume of the iodine standard titration solution consumed by the titration sample, in milliliters (mL);

c – the exact value of the concentration of the iodine standard titration solution, in moles per liter (mol/L);

M – the value of the molar mass of hydrogen sulfide ($1/2H_2S$), in grams per mole (g/mol) (M = 17.04);

m – the value of the sample mass, in grams (g).

Take the arithmetic mean of the parallel determination results as the determination result; the absolute difference between the two parallel determination results shall not be larger than 0.000 02%.

6.7 Determination of sulfate content

6.7.1 Principle

Use water to extract sulfate ions in the sample; barium ions and sulfate ions form white barium sulfate suspended particles in acidic medium; observe the degree of turbidity of the solution.

6.7.2 Reagents or materials

6.7.2.1 Hydrochloric acid solution: 1+1.

6.7.2.2 Barium chloride solution: 100 g/L.

6.7.3 Instruments and apparatuses

6.7.3.1 Pear-shaped separating funnel: 100 mL.

6.7.3.2 Colorimetric tube: 25 mL.

6.7.4 Test procedure

Measure 25 mL ± 1 mL of the sample and place it in the separating funnel; use a pipette to add 25 mL of water accurately; shake for 5 min; let stand for delamination; separate; retain the aqueous phase as the test solution A.

Take 10 mL of test solution A and place it in a colorimetric tube; add 1 drop of hydrochloric acid solution and 1 mL of barium chloride solution; mix well. If the solution does not show turbid within 15 min, consider it passing the test.

6.8 Determination of free acid content

6.8.1 Reagents or materials

Blue litmus test paper.

6.8.2 Test procedure

Take about 2 mL of test solution A (see 6.7.4); place it in a colorimetric tube; use blue litmus test paper to test it. If the test paper does not turn red, the test is passed.

6.9 Tests of sulfur and other sulfides

6.9.1 Principle

Sulfur and other sulfides in carbon disulfide react with mercury to form mercury sulfide. Observe the surface color of the mercury ball.

6.9.2 Reagents or materials

Mercury.

6.9.3 Test procedure

Take 2 mL of the sample and inject it into the test tube; add 1 drop of mercury (3 mm ~ 4 mm in diameter); shake for 5 min. Observe the surface of the mercury ball: the mercury ball has passed the inspection if the mercury ball maintains a metallic luster, while its surface can turn light yellow, and it does not deform. (In order to prevent the pollution of mercury-containing waste liquid, the waste mercury droplets obtained after mercury determination shall be collected and handed over to the environmental protection department for unified disposal).

7 Inspection rules

7.1 The index items listed in the requirements of this document are ex-factory inspection items, which shall be inspected batch by batch.

7.2 Products of the same grade, which are continuously produced by the enterprise or produced in the same group, of the same material, under basically the same production conditions, are a batch. Each batch of products shall not exceed 150 t.

7.3 Determine the number of sampling units as specified in GB/T 6678. Sampling shall be carried out according to the provisions of GB/T 6680: the sample volume shall not be less than 1 000 mL. Put into two clean brown glass bottles with ground stoppers; seal after sealing with water. Stick a label on the bottle, indicating: the name of the manufacturer, product name, grade, batch number and sampling date, and the name of the sampler. Use one bottle for inspection, and store the other bottle for future reference. The storage time is determined by the manufacturer according to the actual situation.

7.4 Where there are indexes in the test results that do not meet the requirements of this document, products in barrels shall be re-inspected for twice the volume of samples from the packaging barrels, and products in tank cars shall be re-inspected for twice the volume of samples from the tank car again. Even if only one index of the re-inspection results fails to meet the requirements of this document, the entire batch of products shall be considered unqualified.

7.5 Use the rounded value comparison method that is specified in GB/T 8170 to determine whether the inspection results meet the standard.

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