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Dimethyl sulfoxide

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Dimethyl sulfoxide

1 Scope

This standard specifies the symbols and abbreviations, requirements, test methods, inspection rules and signs, labels, packaging, transportation, storage of dimethyl sulfoxide.

This standard applies to dimethyl sulfoxide, which is obtained by synthesizing dimethyl sulfide from methanol and carbon disulfide (or methanol and hydrogen sulfide), then oxidizing and refining with pure oxygen.

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.

GB/T 191 Packaging - Pictorial marking for handling of goods (eqv ISO 780:1997)

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

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

GB/T 1250 Rules for expression and judgement of limiting values

GB/T 3723 Sampling of chemical products for industrial use - Safety in sampling (idt ISO 3165:1976)

GB/T 6283 Chemical products - Determination of water Karl·Fischer method (general method) (eqv ISO 760:1978)

GB/T 6488 Liquid chemicals - Determination of refractive index at 20 °C

GB/T 6678 General principles for sampling chemical products

GB/T 6680 General rules for sampling liquid chemical products

GB/T 6682 Water for analytical laboratory use - Specification and test methods (eqv ISO 3696:1987)

5 Test method

5.1 Warning

Some of the test procedures, which are specified in the test method, may lead to hazardous situations. The operator shall take appropriate safety and health measures.

5.2 General provisions

Unless otherwise specified, only the reagents confirmed as analytical grade AND the grade-3 water, which is specified in GB/T 6682, are used in the analysis.

The standard titration solutions, preparations, products, which are used in the analysis, shall be prepared in accordance with the provisions of GB/T 601 and GB/T 603, unless other requirements are specified.

5.3 Determination of odor and appearance

5.3.1 Odor is detected by smell.

5.3.2 Add an appropriate volume of liquid sample into a 50 mL colorimetric tube with a stopper. Visually inspect it against a white background. When using distilled water as a reference, it shall not be deeper (if the sample is a crystal, place the sample, until it is melted for same-method comparison).

5.4 Determination of crystallization point

Determine according to the method specified in GB/T 7533.

Take the arithmetic mean of the results of the two parallel determinations, as the determination result. The absolute difference, between the two parallel determination results, is not greater than 0.1 °C.

5.5 Determination of acid value

Determine according to the method specified in GB/T 9736.

5.5.1 Principle

The free acid in the sample is neutralized with potassium hydroxide. The free acid value can be calculated, according to the consumption of potassium hydroxide standard titration solution.

$$RCOOH + KOH = RCOOK + H_2O$$

5.5.2 Instruments

5.5.2.1 Micro-burette: 2 mL, which has a subscale of 0.01 mL;

5.5.2.2 Conical flask: 250 mL.

5.5.3 Reagents

5.5.3.1 Potassium hydroxide standard titration solution: c (KOH) = 0.05 mol/L;

5.5.3.2 Phenolphthalein indicator solution: 0.1 g/L.

5.5.4 Analytical steps

5.5.4.1 Weigh about 50 g of sample, accurate to 0.01 g. Put it in a conical flask, which contains 100 mL of water in advance. Add 2 ~ 3 drops of phenolphthalein indicator solution. Use potassium hydroxide standard titration solution to titrate it, until pink color appears AND lasts for 1 min without fading, which is deemed as end point.

5.5.4.2 At the same time of the measurement, according to the same steps as the measurement, do a blank test, using the same volume of reagent solution, without adding the sample.

5.5.5 Calculation of results

The acid value, w_1, is calculated by the mass (milligrams) of potassium hydroxide, which is required to neutralize 1 g of the specimen. The value is expressed in milligrams per gram (mg/g), which is calculated in accordance with the formula (1):

$$w_1 = (V - V_1) \times c \times M/m \qquad \cdots\cdots (1)$$

Where:

V - The value of the volume of the potassium hydroxide standard titration solution (5.5.3.1), which is consumed by the sample, in milliliters (mL);

V_1 - The value of the volume of the standard potassium hydroxide titration solution, which is consumed by the blank test, in milliliters (mL);

c - The exact value of the concentration of potassium hydroxide standard titration solution, in mole per liter (mol/L);

M - The value of the molar mass of potassium hydroxide, in grams per mole (g/mol) (M = 56.1);

m - The value of the mass of the specimen, in grams (g).

Take the arithmetic mean of the results of the two parallel determinations as the determination result. The absolute difference, between the two parallel

determination results, is not greater than 0.001.

5.6 Determination of transmittance

Make determination, according to the method specified in GB/T 9721. Use the 5 cm optical path cuvette. Use the distilled water as a reference. The selected wavelength is 400 nm.

Take the arithmetic mean of the results of the two parallel determinations as the determination result. The absolute difference, between the two parallel determination results, is not more than 0.2%.

5.7 Determination of refractive index

Determine it according to the method specified in GB/T 6488.

Take the arithmetic mean of the results of two parallel determinations as the determination result. The absolute difference, between the results of the two parallel determinations, is not greater than 0.0003.

5.8 Determination of impurity content

5.8.1 Method summary

Using gas chromatography, under selected working conditions, the sample is vaporized through a chromatographic column, so that the components are separated. It is detected by a hydrogen flame ionization detector. The content of impurities is determined, according to the area normalization method.

5.8.2 Reagents

5.8.2.1 Polyethylene glycol 20M (fixative solution);

5.8.2.2 White silanized carrier: 0.18 mm ~ 0.15 mm (80 mesh ~ 100 mesh);

5.8.2.3 Hydrogen: The volume fraction is not less than 99.9%; it is dried and purified by silica gel and molecular sieve;

5.8.2.4 Nitrogen: The volume fraction is not less than 99.9%; it is dried and purified by silica gel and molecular sieve;

5.8.2.5 Air: It is dried and purified by silica gel and molecular sieve.

5.8.3 Instruments

5.8.3.1 Gas chromatograph: It is equipped with a flame ionization detector. The sensitivity and stability of the whole machine comply with the relevant requirements in GB/T 9722;

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