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Sebacic Acid for Industrial Use

工业用癸二酸

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Sebacic Acid for Industrial Use

1 Scope

This document specifies the technical requirements, test methods, inspection rules and marking, packaging, transportation and storage of sebacic acid for industrial use.

This document applies to sebacic acid for industrial use, which is produced through hydrolysis and splitting decomposition of castor oil.

NOTE: the molecular formula of sebacic acid is $C_{10}H_{18}O_4$, and the structural formula is $HOOC-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-CH_2-COOH$, the relative molecular mass is 202.25 (in accordance with the international relative atomic mass of 2022).

2 Normative References

The contents of the following documents constitute indispensable clauses of this document through the normative references in the text. In terms of references with a specified date, only versions with a specified date are applicable to this document. In terms of references without a specified date, the latest version (including all the modifications) is applicable to this document.

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 617 Chemical Reagent - General Method for the Determination of Melting Range

GB/T 3143 Color Determination Method of Liquid Chemicals (Hazen unit - platinum - cobalt scale)

GB/T 6283-2008 Chemical Products - Determination of Water - Karl • Fischer Method (general method)

GB/T 6284 Chemical Products for Industrial Use - General Method for Determination of Water Content - The Loss of Mass on Drying Method

GB/T 6678 General Principles for Sampling Chemical Products

GB/T 6679 General Rules for Sampling Solid Chemical Products

GB/T 6682 Water for Analytical Laboratory Use - Specification and Test Methods

GB/T 7531 Determination of Ignition Residue for Organic Chemical Products

GB/T 8170 Rules of Rounding off for Numerical Values & Expression and Judgement of Limiting Values

solution to directly titrate it, and use phenolphthalein as the indicator.

5.3.2 Reagents and solutions

5.3.2.1 Neutral ethanol solution: add 2 ~ 3 drops of phenolphthalein indicator solution to every 50 mL of 95% ethanol and use sodium hydroxide standard titration solution to neutralize it, until it turns slightly red (prepare right before use).

5.3.2.2 Sodium hydroxide standard titration solution: $c(\text{NaOH}) = 0.5 \text{ mol/L}$.

5.3.2.3 Phenolphthalein indicator solution: 10 g/L.

5.3.3 Instruments and equipment

5.3.3.1 50 mL alkali burette.

5.3.3.2 Electronic balance: with a division value of 0.0001 g.

5.3.4 Analytical steps

Weigh-take 1.5 g of sample (accurate to 0.0001 g) and place it in a 250 mL conical flask. Add 50 mL of neutral ethanol solution. After the specimen is completely dissolved, add 2 drops of phenolphthalein indicator solution and use sodium hydroxide standard solution to titrate it, until it turns slightly red. If it does not fade for 30 s, the end point is reached.

5.3.5 Result calculation

The mass fraction of total acid (calculated as sebacic acid) is calculated in accordance with Formula (1):

$$w = \frac{c \times V \times M}{2 \times 1\,000 \times m} \times 100 \quad \dots\dots\dots (1)$$

Where:

w ---the mass fraction of total acid (calculated as sebacic acid) in the sample, expressed in (%);

c ---the actual concentration of sodium hydroxide standard titration solution, expressed in (mol/L);

V ---the volume of sodium hydroxide standard titration solution consumed by titrating the sample, expressed in (mL);

M ---the molar mass of sebacic acid, expressed in (g/mol);

2---conversion factor;

1,000---conversion factor;

m ---the mass of the sample, expressed in (g).

5.3.6 Allowable difference

The absolute difference between the results of two parallel determinations is not greater than 0.2%. The arithmetic mean of the results of the two parallel determinations is taken as the determination result. The calculation result is expressed to one decimal place.

5.4 Determination of Sebacic Acid

5.4.1 Method summary

Adopt gas chromatography. Sebacic acid and methanol undergo esterification reaction under the action of concentrated sulfuric acid to generate fatty acid methyl esters, which are extracted with n-hexane and, under selected working conditions, are vaporized and passed through a capillary chromatographic column to separate the components. Use a hydrogen flame ionization detector to detect the components and quantify them by the area normalization method.

5.4.2 Reagents and materials

5.4.2.1 Anhydrous methanol.

5.4.2.2 Concentrated sulfuric acid.

5.4.2.3 n-hexane: under the chromatographic conditions in this document, there shall be no impurity peak, otherwise it shall be deducted during calculation.

5.4.2.4 Nitrogen: volume fraction is not lower than 99.99%. Before use, it shall be purified with a dehydration device, silica gel, molecular sieve or activated carbon.

5.4.2.5 Hydrogen: volume fraction is not lower than 99.99%. Before use, it shall be purified with a dehydration device, silica gel, molecular sieve or activated carbon.

5.4.2.6 Air: free of corrosive impurities. Before use, carry out degreasing and dehydration.

5.4.3 Instruments and equipment

5.4.3.1 Constant temperature water bath.

5.4.3.2 Gas chromatograph: equipped with hydrogen flame ionization detector (FID) and injection splitter device. The sensitivity and stability of the whole instrument shall comply with the relevant provisions of GB/T 9722. The linear range of the instrument shall satisfy the requirements of quantitative analysis.

5.4.3.3 Recorder: computer chromatographic workstation.

5.4.3.4 Micro-injector: 2 μL .

5.4.4 Chromatographic columns and chromatographic operating conditions

in the sample;

ΣA_i ---the sum of the chromatographic peak areas of the various components in the sample, excluding the peak areas of n-hexane and other impurities in n-hexane.

The arithmetic mean of the results of two parallel determinations is taken as the determination result, and the absolute difference between the results of two parallel determinations is not greater than 0.2%. The calculation result is expressed to one decimal place.

5.5 Determination of Ash Content

In accordance with the provisions of GB/T 7531, carry out the determination. The sample weight of ordinary samples is 15 g ~ 20 g, the sample weight of refined samples is 30 g ~ 50 g, and the ignition temperature is 750 °C.

5.6 Determination of Moisture Content

5.6.1 Loss of mass on drying method (arbitration method)

In accordance with the provisions of GB/T 6284, carry out the determination, and the sample weight is about 5 g.

The arithmetic mean of the results of two parallel determinations is taken as the determination result, and the absolute difference between the results of two parallel determinations is not greater than 0.05%. The calculation result is expressed to two decimal places.

5.6.2 Karl • Fischer method

In accordance with the direct coulometric titration method in Chapter 8 of GB/T 6283-2008, carry out the determination, and the sample weight is about 0.5 g.

The arithmetic mean of the results of two parallel determinations is taken as the determination result, and the absolute difference between the results of two parallel determinations is not greater than 0.05%. The calculation result is expressed to two decimal places.

5.7 Determination of Alkali Solubility

In accordance with the provisions of GB/T 3143, carry out the determination. Weigh-take 3 g of specimen (accurate to 0.1 g), place it in a 50 mL colorimetric tube, add 100 g/L potassium hydroxide solution to the scale and shake it well. After the specimen is completely dissolved, compare the color with the standard platinum-cobalt color scale.

5.8 Determination of Melting Point Range

In accordance with the provisions of GB/T 617, carry out the determination.

6 Inspection Rules

6.1 The items specified in Chapter 4 are all type inspection items, among which, all items except the mass fraction of sebacic acid are exit-factory inspection items. Under normal circumstances, type inspection shall be carried out at least once a year. When one of the following situations occurs, type inspection shall be carried out:

- a) When key production processes are updated;
- b) When there are changes in the main raw materials;
- c) When production is resumed after suspension;
- d) When there is a significant difference between the exit-factory inspection results and the previous type inspection results.

6.2 Batches shall be made in accordance with production shifts or dates. Each production shift or every 24 hours shall constitute one batch.

6.3 Sampling shall be carried out in accordance with the provisions of GB/T 6678 and GB/T 6679. The sampling volume shall not be less than 500 g. Use a clean and dry sampler to take samples from the top, middle and bottom of the packaging bag. After evenly mixing, put them respectively into two clean and dry ground-mouth bottles or Ziplock bags, seal them for storage, and attach labels, indicating the product name, production batch No., sampling date and name of the person performing the sampling. One copy is for inspection and the other is kept as a reserved sample for future reference.

6.4 The inspection results shall be determined in accordance with the rounded value comparison method specified in GB/T 8170. If any of the indicators in the inspection results do not meet the requirements of this document, re-sampling shall be carried out from twice the amount of packaging for re-inspection. If the re-inspection results still show that the indicators do not meet the requirements of this document, then, the entire batch of products will be determined as disqualified.

7 Marking, Packaging, Transportation and Storage

7.1 Marking

7.1.1 The packaging bags of sebacic acid for industrial use shall have clear, obvious and firm markings, which shall at least include the following contents:

- a) Product name, trademark and model;
- b) Name and address of manufacturer;
- c) Batch No. or production date;

- d) Net content (kg);
- e) The serial No. of this document.

7.1.2 Each batch of sebacic acid for industrial use shipped out of the factory shall be accompanied by a quality certificate, which shall at least include the following contents:

- a) Name and address of manufacturer;
- b) Product name;
- c) Batch No. or production date;
- d) Product quality inspection results or conclusions;
- e) The serial No. of this document.

7.2 Packaging

Sebacic acid for industrial use shall be packaged in polyethylene bags, plastic woven bags or woven bags lined with plastic bags, with a net weight of 25 kg, 500 kg or 1,000 kg per bag, or in packaging agreed upon by the supply-side and demand-side and in compliance with safety regulations.

7.3 Transportation

During transportation, sebacic acid for industrial use shall be avoided from being mixed with other acids or alkalis, and from contact with oxidizing and corrosive substances, so to prevent packaging damage and product deterioration.

7.4 Storage

Sebacic acid for industrial use shall be stored in a cool and ventilated warehouse, and shall be protected from fire and moisture.

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