NATIONAL STANDARD OF THE

PEOPLE'S REPUBLIC OF CHINA

GB 1886.349-2021

National food safety standard - Food additives - Five carbon diacetal (also known as glutaraldehyde)

Issued on: August 4, 2021 **Implemented on: February 4, 2022**

Issued by: National Health Commission;
State Administration for Market Regulation

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1 Scope

This Standard applies to the food additive five-carbon diacetal (also known as glutaraldehyde) obtained by that vinyl methyl ether or vinyl ethyl ether reacts with acrolein to form 2-alkoxy-3,4-dihydropyran, which is then hydrolyzed.

2 Chemical name, molecular formula, structural

formula and relative molecular mass

2.1 Chemical name

Five-carbon diacetal (also known as glutaraldehyde)

2.2 Molecular formula

C5H8O2

2.4 Relative molecular mass

100.12 (according to 2018 international relative atomic mass)

3.1 Sensory requirements

Sensory requirements shall meet the requirements of Table 1.

Annex A Inspection methods

WARNING: Some reagents used in the inspection methods of this Standard are toxic or corrosive. Operate in accordance with relevant national regulations. Please be careful when using them. If it is splashed on the skin, rinse immediately with water. In severe cases, seek medical attention immediately. When using volatile acids, it shall be carried out in a fume hood.

A.1 General The reagents and water used in this Standard refer to analytically-pure reagents and grade 3 water specified in GB/T 6682 when other requirements are not indicated. The standard titration solution, standard solution for impurity determination, preparations and products used in the test are prepared in accordance with GB/T 601, GB/T 602 and GB/T 603 when other requirements are not indicated. The solution used in the test refers to an aqueous solution when it is not specified which solvent is used to prepare it.

A.2 Identification test A.2.1 Solubility Pipette about 1mL of specimen into a beaker. Add 1mLof water. Shake not less than 30s. Observe the dissolution of the sample within 5min. The specimen shall be easily dissolved in water.

A.2.2 Identification of the melting point of 2,4-dinitrophenylhydrazone A.2.2.1 Method principle Five carbon diacetal reacts with 2,4-dinitrophenylhydrazine to form 2,4-dinitrophenylhydrazone precipitation. 2,4-dinitrophenylhydrazone has a specific melting point.

A.2.2.2 Reagents and materials A.2.2.2.1 2,4-dinitrophenylhydrazine solution: Take 0.8g of 2,4-dinitrophenylhydrazine. Add 4mL of sulfuric acid. Stir until it is completely dissolved. Continue stirring. Add 6mL of water along with the wall in drops. Add 20mL of ethanol. Mix and shake well. Filter. Take the filtrate.

A.2.2.2.2 Absolute ethanol.

The carbonyl group of glutaraldehyde can react with hydroxylamine hydrochloride to produce glutaric oxime. Glutaraldehyde will release hydrochloric acid while generating glutaric oxime. Triethanolamine is weakly alkaline. It can neutralize and remove hydrochloric acid. Add excessive triethanolamine. Use hydrochloric acid standard solution to backdrop. The content of glutaraldehyde can be calculated by calibrating the blank determination at the same time.

A.3.2 Reagents and materials A.3.2.1 Triethanolamine solution: 74g/L. Dissolve 74g of triethanolamine with a certain amount of water and transfer to a 1L volumetric flask. Use water to set volume to the scale and mix well.

A.3.2.2 Hydroxylamine hydrochloride solution: 35g/L, pH is 3.60. Use a certain amount of water to dissolve 35.0g of hydroxylamine hydrochloride. Transfer to a 1L volumetric flask. Use water to set volume to the scale. Mix well. Use triethanolamine solution to adjust pH to 3.60.

A.3.2.3 Hydrochloric acid standard titration solution: c(HCl)=0.5mol/L.

A.3.3 Instruments and equipment A.3.3.1 Electronic balance: the resolution is 0.1g and 0.0001g.

A.3.3.2 Magnetic stirrer.

A.3.3.3 Acidity meter: the accuracy is +/-0.01.

A.3.4 Determination method Add 65.0mL of pH 3.60 hydroxylamine hydrochloride solution and 30.8mL of triethanolamine solution into a 200mL small beaker. Put a Teflon (or equivalent material) rotor in each small beaker. Place the small beaker on a magnetic stirrer and stir. Accurately add a certain amount of sample (approximately equivalent to 300mg of five carbon bisacetal). Put a lid and stir. Stir at room temperature for at least 60min, but not more than 90min. At the same time, do a reagent blank test without adding a specimen. After the stirring is over, use 0.5mol/L hydrochloric acid to titrate the specimen and the blank sample to pH

3.60 as the end point.

NOTE: In the process of neutralization analysis, stirring speed is a key factor. When stirring is required, make sure that no bubbles are mixed into the solution, and the specimen and the blank sample are always kept at the same stirring speed.

A.3.5 Result calculation The content of five-carbon diacetal is calculated as the mass fraction w1 of A.4.3 Instruments and equipment A.4.3.1 Gas chromatograph with hydrogen flame ionization detector (FID).

A.4.3.2 Electronic balance: Resolution is 0.1mg.

A.4.4 GC reference conditions A.4.4.1 Chromatographic column: Polyethylene glycol quartz capillary column;

column length is 18m, inner diameter is 0.53mm, film thickness is 1.0um, or equivalent column.

A.4.4.2 Chromatographic column temperature: Initial temperature is 40°C, keep for 1min; increase to 130°C at 4.0°C /min; increase to 200°C at 20°C /min, keep for 5min.

A.4.4.3 Detector temperature: 250°C.

A.4.4.4 Injection port temperature: 250°C.

A.4.4.5 Carrier gas flow rate: 1mL/min.

A.4.4.6 Hydrogen flow rate: 40mL/min.

A.4.4.7 Air flow rate: 400mL/min.

A.4.4.8 Injection volume: 1uL.

A.4.4.9 Split ratio: 20:1.

A.4.5 Analysis steps A.4.5.1 Preparation of standard solution A.4.5.1.1 Methanol standard stock solution: 5000mg/L. Accurately weigh 0.5g of methanol standard, to the nearest of 0.0001g. Place in a 100mL volumetric flask. Use isopropanol to set volume to the scale. Mix well. Store in a refrigerator at 0°C ~4°C.

A.4.5.1.2 Ethanol standard stock solution: 5000mg/L. Accurately weigh 0.5g of ethanol standard, to the nearest of 0.0001g. Place in a 100mL volumetric flask.

Use isopropanol to set volume to the scale. Mix well. Store in a refrigerator at 0°C ~4°C.

A.4.5.1.3 Mixed series standard working liquids: Pipette 0.05mL, 0.2mL, 0.4mL, 0.8mL, 1.6mL, 2mL of methanol standard stock solution and ethanol standard stock solution respectively into six 10mL volumetric flasks. Use isopropanol to set volume to the scale. Prepare into series standard solutions of which the methanol and ethanol content are 25mg/L, 100mg/L, 200mg/L, 400mg/L,