

ICS 67.220.20
CCS X 40



**NATIONAL STANDARD OF THE
PEOPLE'S REPUBLIC OF CHINA**

GB 29223-2012

**National food safety standards -- Food additives --
Dehydrogenation acid**

食品添加剂 脱氢乙酸

(English Translation)

Issued on: December 25, 2012

Implemented on: January 25, 2013

Issued by: Ministry of Health of the People's Republic of China

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1 Scope

This standard applies to the diacyl ketone polymerization reaction of the food additives dehydrogenated acetic acid.

2.1 Chemical name

3-acetyl-6-methyl-de (2), 4-de (3) hydrodione

2.2 Molecular formula

C₈H₈O₄

2.4 Relative molecular mass

168.15 (according to the 2007 International Relative Atomic Quality)

3.1 Sensory requirements

Shall comply with the provisions of Table 1.

Table 1 sensory requirements

The project requires a test method Color white to light yellow State powder Take appropriate sample, placed in a clean, dry white porcelain dish, in the natural light, observe the color and status

3.2 Physical and chemical indicators

Shall comply with the provisions of Table 2.

Table 2 Physical and chemical indicators

Item Index Test Method Dehydrogenated acetic acid ($C_8H_8O_4$) content (on a dry basis), w /% 98.0 ~ 100.5 Appendix A A.4 Melting point/ $^{\circ}C$ 109.0 ~ 111.0 Appendix A A.5 Dry reduction, w /% ≤ 1.0 Appendix A A.6 Residue on ignition, w /% ≤ 0.1 Appendix A A.7 Lead (Pb)/(mg/kg) ≤ 0.5 Appendix A A.8 Appendix A Testing method A.1 Warning the test procedure specifies that some of the test procedures may lead to dangerous conditions and that the operator should take appropriate safety and protective measures.

A.2 General provisions Unless otherwise stated, only the reagents identified as analytical reagents and the tertiary water specified in GB/T 6682-2008 are used in the analysis. test Method used in the standard titration solution, impurity determination standard solution, preparation and products, without any other requirements, according to GB/T 601, GB/T 602 and GB/T 603; the solution used in the absence of the solvent, refers to the aqueous solution.

A.3 Identification test A.3.1 Reagents and solutions A.3.1.1 Ethanol.

A.3.1.2 Sodium hydroxide solution. 250 g/L.

A.3.1.3 Salicylic acid ethanol solution..200 g/L.

A.3.1.4 Potassium tartrate solution. 140 g/L.

A.3.1.5 copper acetate solution. take copper acetate 13.3g, add 300g/L acetic acid solution 5mL, add water 195mL, shake.

A.3.2 Identification method A.3.2.1 Acetyl test. take 0.5g sample, add sodium hydroxide solution 2mL, salicylic acid ethanol solution 3 drops, after heating in the water bath Red reaction.

A.3.2.2 copper salt test. take 0.02g sample, add 2mL of ethanol dissolved, add potassium tartrate solution 3 drops, 3 drops of copper acetate solution, After the oscillation of purple white precipitate formed.

A.4 Dehydrogenated acetic acid ($C_8H_8O_4$) content (measured on dry basis) A.4.1 Methodological Summary Specimen with neutral alcohol as solvent, phenolphthalein as indicator, with sodium hydroxide standard titration solution titration, according to the consumption of sodium hydroxide standard the volume of the titration solution is calculated for the amount of dehydrogenated acetic acid.

A.4.2 Reagents and materials A.4.2.1 Sodium hydroxide standard titration solution. c (NaOH) = 0.1 mol/L.

A.4.2.2 Neutral Ethanol. 95% ethanol was neutralized with sodium hydroxide solution using the phenolphthalein indicator as an indicator.

A.4.2.3 phenolphthalein indicator solution. 10g/L.

A.4.3 Analysis steps Weigh about 0.5g sample, accurate to 0.0002g, placed in a conical flask, add 30mL neutral ethanol dissolved, add 3 drops of phenolphthalein instructions Liquid, titration with sodium hydroxide titration solution to reddish, and at least 30s to keep fading is the end point.

A.4.4 Calculation of results the mass fraction w_1 of dehydroacetic acid (calculated as $C_8H_8O_4$) is calculated according to formula (A.1) $w_1 = (V/1000) cM m (1-w_2) \times 100\%$ (A.1) where.

V --- sample volume of sodium hydroxide standard titration solution, in milliliters (mL);

c - the actual concentration of sodium hydroxide standard titration solution in moles per liter (mol/L);

1000 --- conversion factor;

m --- the quality of the sample, in grams (g);

M - dehydroacetic acid ($C_8H_8O_4$), in grams per mole (g/mol) ($M = 168.2$);

w_2 --- A.6 Mass fraction of dry reduction measured, %.

The arithmetic mean of the results of two parallel measurements is the result of the report. The absolute difference between the two parallel determinations is not more than 0.2%.

A.5 Determination of melting point Heating device can choose high-type beaker. in the beaker to install a fixed a serpentine glass tube, snake-shaped glass tube fixed a power of about 300W of the electric wire, the electric wire is connected with the output end of an adjustable voltage device. Place the beaker on a magnetic stirrer and start the magnetic stir during heating.

The rest according to the provisions of GB/T 617-2006 4.1.

A.6 Determination of dry reduction A.6.1 Analysis steps Weigh 2g ~ 3g sample, accurate to 0.0002g, placed in the pre-105 °C $\pm$ 2 °C dry to the constant quality of the weighing bottle, paved 5mm below the layer. In 80 °C $\pm$ 2 °C constant temperature drying oven for 1h, placed in a dryer for 30min cooling.

A.6.2 Calculation of results the mass fraction w_2 of the drying reduction is calculated according to the formula (A.2) $w_2 = m - m_1 m \times 100\%$ (A.2) where.

m - the mass of the sample before drying, in grams (g);

m_1 --- the quality of the sample after drying, in grams (g).

The arithmetic mean of the results of two parallel measurements is the result of the measurement. The absolute difference between the two parallel determinations is not more than 0.1%.

A.7 Determination of burning residue A.7.1 Method of measurement in the already constant quality porcelain crucible weighed about 2g sample, accurate to 0.0002g, first with a small fire slowly heating carbonization, careful not to make Fire, smoke to be removed after the lid, set the high temperature furnace in the 510 °C ~ 600 °C burning ashing, called the constant quality.

A.7.2 Result calculation the mass fraction w_3 of the burning residue is calculated according to equation (A.3) $w_3 = \frac{m_2}{m_1} \times 100\%$ (A.3) where.

m_1 - the mass of the sample before burning, in grams (g);

m_2 - the mass of the sample after ignition, in grams (g).

The arithmetic mean of the results of two parallel measurements is the result of the measurement. The absolute difference between the two parallel determinations is not more than 0.02%.

A.8 Determination of lead (Pb) Weigh 10.0g sample, accurate to 0.001g, placed in the porcelain crucible, add the appropriate amount of sulfuric acid wet sample, carefully carbonized, add 4mL Acid and 10 drops of sulfuric acid, carefully heated until the white smoke waved, into the high temperature furnace, at 550 °C ± 25 °C ashing completely. After cold out.

Add 2mL nitric acid (1 1) solution, heat to dissolve the ash, the sample solution transferred to 50mL volumetric flask (if necessary, filter), and with a small amount Water washing crucible, lotion and into the volumetric flask, add water to the mark, mix spare. Each 5 mL solution corresponds to 1.0 g of sample. Other press GB 5009.12.