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Feed additives - Part 1: Amino acids, their salts and analogues - Methionine hydroxy analogue

饲料添加剂 第 1 部分:氨基酸、氨基酸盐及其类似物 蛋氨酸羟基类似物

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Feed additives - Part 1: Amino acids, their salts and analogues - Methionine hydroxy analogue

1 Scope

This part of GB 7300 specifies the requirements, test methods, inspection rules and labeling, packaging, transportation, storage, and shelf life requirements for feed additives methionine hydroxy analogues.

This part applies to feed additives methionine hydroxy analogues produced by chemical synthesis with acrolein, methyl mercaptan, and hydrogen cyanide used as the main raw materials.

Chemical name: 2-Hydroxy-4-(methylthio)butyric acid

Molecular formula: $C_5H_{10}O_3S$

Relative molecular mass: 150.2 (in $C_5H_{10}O_3S$, calculated according to the 2016 International Standard Relative Atomic Mass)

2 Normative references

The following documents are essential to the application of this document. For the dated referenced documents, only the versions with the indicated dates are applicable to this document; for the undated referenced documents, only the latest version (including all the amendments) is applicable to this document.

GB/T 601 Chemical reagent - Preparations of reference titration solutions

GB/T 602 Chemical reagent - Preparations of standard solutions for impurity

GB/T 603 Chemical reagent - Preparations of reagent solutions for use in test methods

GB/T 6680 General rules for sampling liquid chemical products

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

GB 10648 Feed label

GB/T 13079-2006 Determination of total arsenic in feeds

4.2.1.3 2,7-Dihydroxynaphthalene.

4.2.1.4 Anhydrous copper sulfate saturated sulfuric acid solution: Take anhydrous copper sulfate (4.2.1.1), add sulfuric acid (4.2.1.2) and stir until it is insoluble.

4.2.1.5 2,7-dihydroxynaphthalene sulfuric acid solution: 0.01%; weigh 0.01 g of 2,7-dihydroxynaphthalene (4.2.1.3), dissolve it with sulfuric acid (4.2.1.2) and make up the volume to 100 mL, and newly prepare it when it will be used.

4.2.2 Identification test

4.2.2.1 Take 25 mg of this product and put it into a dry test tube, add 1 mL of anhydrous copper sulfate saturated sulfuric acid solution (4.2.1.4), the solution immediately turns yellow, and then turns yellow-green.

4.2.2.2 Take 1 drop of this product and put it into a dry test tube, add the newly prepared 2,7-dihydroxynaphthalene sulfuric acid solution (4.2.1.5), put the tube in a boiling water bath and boil for 10 min~15 min, and then the color turns from yellow to reddish brown.

4.3 Determination of content of methionine hydroxy analogues

4.3.1 Principle

In an acidic medium, methionine hydroxy analogues undergo the following redox reactions:

$$3C_4H_6O_3SCH_3+BrO_3^-\rightarrow 3C_4H_6O_3SOCH_3+Br^-$$

The color change of bromine can be used to judge the end point of the reaction.

4.3.2 Reagents

4.3.2.1 Potassium bromate.

4.3.2.2 Potassium bromide.

4.3.2.3 Potassium iodide.

4.3.2.4 Hydrochloric acid.

4.3.2.5 Hydrochloric acid solution: hydrochloric acid + water = 1 + 1.

4.3.2.6 Acid solution: glacial acetic acid + water + concentrated hydrochloric acid = 50 + 10 + 3.

4.3.2.7 Sodium thiosulfate standard titration solution: $c(Na_2S_2O_3)$ = 0.1 mol/L (according to the provision of GB/T 601).

4.5.1.2 Nitric acid.

4.5.1.3 Hydrochloric acid.

4.5.1.4 Lead standard solution: 1000 μg/mL; weigh 0.1599 g of lead nitrate, put it in a 1000 mL measuring bottle, add 5 mL of nitric acid and 50 mL of water to dissolve; dilute with water to the mark, shake well, and use it as a stock solution (according to the provisions of GB/T 602); or use a commercially available lead single-element standard solution of 1000 μg/mL.

4.5.1.5 Ammonia solution (10%): Take 40 mL of ammonia water, add an appropriate amount of water to make up to 100 mL, and shake well (prepared according to GB/T 603).

4.5.1.6 Hydrochloric acid solution I: Take 63 mL of hydrochloric acid, add an appropriate amount of water to make up to 100 mL, and shake well.

4.5.1.7 Hydrochloric acid solution II: Take 18 mL of hydrochloric acid, add water to make up to 100 mL, and shake well.

4.5.1.8 Sodium sulfide solution: Take 100 g of sodium sulfide and add water to dissolve it into 1000 mL.

4.5.1.9 Acetate buffer solution (pH 3.5): Take 25 g of ammonium acetate, add 25 mL of water to dissolve, add 38 mL of hydrochloric acid solution I (4.5.1.6), and use hydrochloric acid solution II (4.5.1.7) or ammonia solution (4.5.1.5) to accurately adjust the pH value to 3.5 (indicated by the potentiometer); dilute to 100 mL with water, and shake well.

4.5.1.10 Phenolphthalein indicator solution: Take 1 g of phenolphthalein, add 100 mL of ethanol to dissolve it, and then the solution is obtained (prepared according to GB/T 603). The range of discoloration is pH 8.3~10.0 (colorless→red).

4.5.1.11 Preparation of lead standard working solution: Accurately measure 2 mL of lead standard solution (4.5.1.4), put it in a 200 mL measuring bottle, dilute with water to the mark, and shake well (each mL solution is equivalent to 10 μg of Pb).

4.5.2 Analysis steps

4.5.2.1 Sample solution preparation

Weigh 1 g of the sample (accurate to 10 mg), place it in a porcelain crucible, blaze slowly until it is completely carbonized, and let it cool. Add 0.5 mL~1 mL of sulfuric acid (4.5.1.1) to make it moist, heat at low temperature until the sulfuric acid vapor is completely removed, burn it at 550 ℃ to make it completely ash, and let it cool. Add 0.5 mL of nitric acid (4.5.1.2), evaporate to dryness until the nitrogen oxide vapor is

completely removed, and let it cool. Add 2.0 mL of hydrochloric acid (4.5.1.3), evaporate to dryness on a water bath, and add 15 mL of water; add dropwise ammonia solution (4.5.1.5) until it reacts to phenolphthalein indicator solution (4.5.1.10) and the solution turns slightly red, and then add 2.0 mL of acetate buffer (4.5.1.9); after slightly heating to dissolve, transfer the solution to a Nessler colorimetric tube, add water to dilute it to 25 mL, which is used as tube B.

4.5.2.2 Preparation of standard colorimetric solution

Take another reagent for preparing the sample solution, evaporate it to dryness in a porcelain crucible, add 2.0 mL of acetate buffer solution (4.5.1.9) and 15 mL of water; after slightly heating to dissolve, transfer the solution to a Nessler colorimetric tube, add 1.00 mL of lead standard working solution (4.5.1.11), and then dilute it with water to 25 mL, which is used as tube A.

4.5.3 Measurement and result judgment

Add 5 drops of sodium sulfide solution (4.5.1.8) to tubes A and B respectively, shake well, and let them stand for 2 minutes; place them on white paper, see through from top to bottom, and compare the colors of tubes A and B with naked eyes. If the color in tube B is not darker than that in tube A, it is judged that the requirements are met.

4.6 Determination of ammonium salt

4.6.1 Reagents

4.6.1.1 Magnesium oxide.

4.6.1.2 Hydrochloric acid solution: hydrochloric acid + water = 1 + 3 (volume ratio).

4.6.1.3 Sodium hydroxide solution: 10%.

4.6.1.4 Saturated aqueous solution of mercuric chloride: Add mercuric chloride into water and stir until it is insoluble.

4.6.1.5 Nessler reagent: Dissolve 10 g of potassium iodide in 10 mL of water, and add mercuric chloride saturated aqueous solution (4.6.1.4) while stirring, until the formed red precipitate is no longer dissolved; add potassium hydroxide and dissolve it, then add 1 mL of mercuric chloride saturated aqueous solution, and add water to 200 mL; let it stand, and then take the supernatant out and store it in a brown bottle.

4.6.1.6 Ammonium standard solution: 0.01 mg/mL (according to the provisions of GB/T 602).

4.6.2 Analysis steps

Accurately weigh 0.20 g of the sample, put it in a distillation bottle, add 70 mL of water,

then add 1 g of magnesium oxide (4.6.1.1), and carry out distillation; use 5 mL of hydrochloric acid solution (4.6.1.2) as the absorption liquid, put the lower end of the condenser tube to be immersed in the absorption liquid, collect about 70 mL of the distillate, and stop the distillation; dilute the distillate to 100 mL with water, and accurately measure 1 mL of the distillate into a Nessler colorimetric tube; add 2 mL of sodium hydroxide solution (4.6.1.3), 20 mL of water, 1 mL of Nessler reagent (4.6.1.5), diluted with water to 50 mL, and shake well.

Accurately pipette 3 mL of ammonium standard solution (4.6.1.6) into another Nessler colorimetric tube, and at the same time, carry out the colour reaction with the same steps as the sample test; the color of the sample solution shall not be darker than the standard solution.

4.7 Determination of cyanide

4.7.1 Reagents or materials

4.7.1.1 Tartaric acid solution: Dissolve 10 g of tartaric acid in water, and dilute to 100 mL.

4.7.1.2 Ferrous sulfate solution: Take 8 g of ferrous sulfate ($FeSO_4 \cdot 7H_2O$), and add 100 mL of freshly boiled cold water.

4.7.1.3 Sodium hydroxide solution: Take 4.3 g of sodium hydroxide, dissolve it in water, and dilute the solution to 100 mL.

4.7.1.4 Ferric chloride solution: Take 9 g of ferric chloride, dissolve it in water, and dilute the solution to 100 mL.

4.7.1.5 Alkaline ferrous sulfate test paper: Before use, take a piece of filter paper and add 1 drop of ferrous sulfate test solution (4.7.1.2) and 1 drop of sodium hydroxide test solution (4.7.1.3).

4.7.1.6 Hydrochloric acid.

4.7.2 Instruments and apparatus

The instruments and apparatus are shown in Figure 1.

4.7.3 Analysis steps

Weigh 1 g of the sample (accurate to 0.0001 g), add 10 mL of water and 3 mL of tartaric acid solution (4.7.1.1), quickly and closely plug a gas-guide tube containing alkaline ferrous sulfate test paper (4.7.1.5) into the conical flask; shake well, heat on a low heat, and boil slightly for 1 min. Take off the alkaline ferrous sulfate test paper, and add 1 drop of ferric chloride solution (4.7.1.4) and 1 drop of hydrochloric acid (4.7.1.6); the

When one of the following situations occurs, type inspection shall be carried out, and the inspection items include all technical indexes specified in Chapter 3:

a) When the product is finalized, and when there are major changes in raw materials, production processes, and equipment;

b) During normal production, once every six months;

c) When the production is resumed after the suspension of production for more than 3 months;

d) When the industry administrative department requests a type inspection;

e) When the factory inspection results are significantly different from the last type inspection results.

5.5 Judgment rules

If one of the indexes of the inspection results does not meet the requirements of this part, samples shall be taken from a doubled number of packaging units for re-inspection. If one of the indexes of the re-inspection results still does not meet the requirements of this part, the batch of products is judged as unqualified.

6 Labeling, packaging, transportation, and storage

6.1 Labeling

Labeling shall be implemented according to GB 10648. The corrosion warning labels shall be indicated in a prominent position.

6.2 Packaging

This product shall be packaged in acid-resistant plastic barrels or other acid-resistant containers and sealed.

6.3 Transportation

Collision shall be strictly prohibited during the transportation of this product to prevent damage to the packaging, and transportation mixed with toxic and harmful substances is strictly prohibited.

6.4 Storage

This product shall be stored in a clean place, protected from sunlight, rain, and moisture, and it is strictly forbidden to store it with toxic and harmful items.

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