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GB XXXX—20XX

Feed additives part 1: amino acids, amino Acid salt and its analogue L-valine

Feed additives — Part1: Amino acids, theirsaltsandanalogues — L-Valine

(Draft for comments)

20××-××-×× release 20××-××-×× implementation

State Administration for Market Regulation
release
National Standardization Management Committee

before Speak

GB 7300 "Feed Additives" is divided into several parts according to products.

This document is part 104 of GB 7300.

This document was drafted in accordance with the rules given in GB/T 1.1-2020.

This document was proposed and managed by the Ministry of Agriculture and Rural Affairs of the People's Republic of China.

Please note that certain contents of this document may involve patents. The issuing agency of this document is not responsible for identifying these patents.

This document was drafted by: Institute of Agricultural Quality Standards and Testing Technology, Chinese Academy of Agricultural Sciences [National Feed Quality Supervisic

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The main drafters of this document:

Feed additives Part 1: Amino acids, amino acid salts and their analogs L-valine

1 Scope

This part of GB 7300 specifies the technical requirements, test methods, inspection rules and labeling, packaging, and packaging of feed additive L-valine products. Transportation, storage and shelf life.

This document is applicable to corn, starch, sugar, etc. as the main raw materials, fermented with Corynebacterium glutamicum, after extraction, crystallization, drying, etc. L-valine, a feed additive made by art.

2 Normative references

The contents of the following documents constitute indispensable clauses of this document through normative references in the text. Among them, dated reference documents, Only the version corresponding to that date is applicable to this document; for undated references, the latest version (including all amendments) is applicable to this document file.

GB/T 601 Preparation of chemical reagent standard titration solution

GB/T 6435-2014 Determination of moisture in feed

GB/T 6438 Determination of crude ash in feed

GB/T 6682 Analytical laboratory water specifications and test methods

GB/T 8170 Numerical rounding rules and the expression and determination of limit values

GB/T 9725 General Rules for Potentiometric Titration of Chemical Reagents

GB 10648 Feed label

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

GB/T 14699.1 Feed sampling

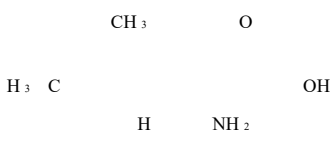
3 Chemical name, molecular formula, relative molecular mass and structural formula

Chemical name: L-2-amino-3-methylbutyric acid

Molecular formula: $C_5H_{11}NO_2$

Relative molecular mass: 117.15 (calculated as $C_5H_{11}NO_2$ and calculated according to the 2016 International Relative Atomic Mass Table)

Structural formula:



4 Technical requirements

4.1 Appearance and traits

Off-white or off-white crystalline powder, odorless, bitter, soluble in water, almost insoluble in organic solvents such as ethanol, ether, acetone, etc. Dissolve.

4.2 Identification:

4.2.1 Identification of Ninhydrin

Should meet the reaction characteristics of the amino acid ninhydrin test.

4.2.2 Infrared spectroscopy

The infrared absorption spectrum of the valine sample should be consistent with that of the reference substance.

4.2.3 Specific rotation method

The specific rotation meets the requirements of Table 1.

4.3 Technical indicators

The technical indicators of valine should meet the requirements in Table 1.

Table 1 Technical indicators

project		index
Valine ($C_5H_{11}NO_2$, on a dry basis)/%		98.0-101.5
Loss on drying/(%)	≤	1.0
Ignition residue/(%)	≤	0.5
Specific rotation $[\alpha]_D^{20}$/[(°)·m²/kg]		+26.0~+29.0
pH (5% aqueous solution)		4.0~7.0
Arsenic/(mg/kg)	≤	2.0
Heavy metals (calculated as Pb)/(mg/kg)	≤	15

5 sampling

According to the provisions of GB/T 14699.1.

6 Test method

The reagents and water used in this standard, unless otherwise specified, only use analytical reagents, and the water used for chromatography conforms to the first grade water in GI It is stipulated that the preparation of reagents and solutions should comply with the requirements of GB/T 601, GB/T 602 and GB/T 603.

6.1 Sensory inspection

Take an appropriate amount of sample and place it in a clean and dry white porcelain dish. Under natural light, it should be off-white or off-white crystalline powder. Smell its smell Odorless, bitter taste. Take a small amount of sample and dissolve it in water and ethanol. The sample is soluble in water and almost insoluble in ethanol.

6.2 Identification

6.2.1 Reagents or solutions

6.2.1.1 Water: meet the secondary water specified in GB/T 6682.

6.2.1.2 Potassium bromide: spectrally pure.

6.2.1.3 Ninhydrin solution: 1 g/L aqueous solution, weigh 0.1 g ninhydrin into a 100 mL volumetric flask, dissolve in water and dilute to the mark.

Shake well.

6.2.1.4 Hydrochloric acid solution: c(HCL)=6.0 mol/L, mix an equal volume of hydrochloric acid with water and shake well.

6.2.2 Instruments

6.2.2.1 Analytical balance: Sensitivity is 0.001g, 0.0001g.

6.2.2.2 Infrared spectrophotometer.

6.2.2.3 Polarimeter, a light source with a wavelength of 589.3 nm ± 0.3 nm.

6.2.2.4 Constant temperature water bath (temperature adjustable).

6.2.3 Identification method

6.2.3.1 Ninhydrin identification

Weigh 0.1 g of the sample, dissolve it in 100 mL of water, take 5 mL of the solution, add 1 mL of ninhydrin (6.2.1.3), and react in a boiling water bath for 15 minutes. The solution was blue-purple.

6.2.3.2 Infrared spectroscopy

Appropriate amount of sample was weighed out, added potassium bromide (6.2.1.2) polishing uniformity, tablet, cm & lt at 4000 ₁ ~400cm ₁ recording the reference and sample The infrared spectrum. The infrared absorption spectrum of the sample should be consistent with that of the reference substance (see Appendix A for the reference substance spectrum).

6.2.3.3 Specific rotation method

The sample is dried to constant weight at 105℃, weigh about 4g (accurate to 0.0001g) of the dried sample, dissolve it with 6.0 moL/L hydrochloric acid, and determine Fill to a 50 mL volumetric flask. Adjust the temperature of the solution to 20°C, filter with medium-speed qualitative filter paper, and measure the optical rotation of the filtrate with a pol

α is calculated according to formula (1):

The specific rotation of valine to the D line (569.2nm) of the sodium spectrum at 20°C $[\alpha]_D^{20}$

$$[\alpha]_D^{20} = \frac{100\,\alpha}{lc} \quad \square\square\square\square\square\ )1($$

Where:

α—The optical rotation of the sample solution, the unit is degree [(°)·m ₂ /kg];

l — The length of the polarimeter, in decimetres (dm);

c — The concentration of valine in the solution, in grams per 100 milliliters (g/100 mL).

The specific rotation should meet the requirements of Table 1.

6.3 Determination of Valine Content

6.3.1 Principle

The sample is dissolved in anhydrous formic acid and glacial acetic acid, titrated with perchloric acid standard titration solution, and replaced by the volume consumed by perchlori Enter the formula to calculate the valine content.

6.3.2 Reagents and solutions

6.3.2.1 Anhydrous formic acid.

6.3.2.2 Glacial acetic acid.

6.3.2.3 Perchloric acid standard titration solution: c (HCLO4) = 0.1 moL/L, prepared and calibrated according to GB/T 601.

6.3.2.4 α-naphthol phenylmethanol indicator solution: 2 g/L glacial acetic acid solution, weigh 0.2g of α-naphthol phenylmethanol in a 100mL volumetric flask In the medium, add glacial acetic acid to dissolve, dilute to volume, and mix well.

6.3.3 Apparatus and equipment

6.3.3.1 Analytical balance: the sensitivity is 0.0001 g.

6.3.3.2 Potentiometric titrator: the glass electrode is used as the indicator electrode, and the saturated calomel electrode is used as the reference electrode (or a composite electrode).

6.3.3.3 Acid burette.

6.3.4 Test procedure

Weigh about 0.1 g (accurate to 0.0001 g) dry to constant weight sample into a dry 100 mL beaker, add 1.0 mL anhydrous formic acid to make It is completely dissolved, then add 50 mL of glacial acetic acid, vortex to mix, insert the electrode into the solution, adjust the stirring speed until the solution is fully vortexed, Titrate perchloric acid standard titration solution (6.3.2.3) to the end point. Or use indicator, add α-naphthol phenyl methanol indicator solution (6.3.2.4) 3 Drop, titrate with perchloric acid standard titration solution (6.3.2.3), the solution turns from yellow to yellow-green as the end point. Do a blank test at the same time.

6.3.5 Result calculation

The content of valine ($C_5H_{11}NO_2$) ω_1 is expressed by mass fraction and calculated according to formula (2):

$$\omega_1 = \frac{c \times (v - v_0) \times 117.15}{m \times 1000} \times 100 \quad \square\square\square\square\square\square \quad (2)$$

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Where:

c ——Concentration of perchloric acid standard titration solution, the unit is mole per liter, mol/L;

v ——The volume of perchloric acid standard titration solution consumed by the titration sample, the unit is ml, ml;

v_0——Blank test consumes the volume of perchloric acid standard titration solution, the unit is ml, ml;

m ——sample mass, in grams, g;

117.15—The molar mass of valine, g/mol.

The test results are expressed as the arithmetic mean of parallel determination results, with three significant figures reserved.

6.3.6 Repeatability

The absolute difference between the two parallel determination results is not more than 0.5%.

6.4 Determination of loss on drying

According to the provisions of 8.1 in GB/T 6435-2014.

6.5 Determination of ignition residue

According to the provisions of GB/T 6438.

6.6 Determination of specific rotation

Same as 6.2.3.3.

6.7 Determination of pH

6.7.1 Instruments

6.7.1.1 Balance: Sensitivity is 0.001 g.

6.7.1.2 Acidity meter.

6.7.2 Test procedure

Accurately weigh 5.0 g of the sample (accurate to 0.001 g), place it in a beaker, add 100 mL of water to dissolve it, shake it well, and use it as a sample solution.

The pH meter measures the pH value of the sample solution, and when the reading is stable, record the result. The result is expressed as an arithmetic average, and two significant digits ar

6.8 Determination of Arsenic

Accurately weigh 1 g of the sample (accurate to 0.0001 g), and prepare the test solution in accordance with 5.4.1.3 dry ashing method in GB/T 13079-2006 5 silver salt method.

And carry out arsenic determination according to any method stipulated in this standard.

6.9 Determination of heavy metals (in pb) (modified in accordance with the 2015 edition of the Pharmacopoeia)

6.9.1 Reagents or solutions

6.9.1.1 Sulfuric acid.

Note: Sulfuric acid is a strong corrosive liquid. Operators need to wear protective glasses and gloves to prevent burns.

6.9.1.2 Nitric acid.

6.9.1.3 Hydrochloric acid.

6.9.1.4 10% ammonia solution: Take 40 mL of ammonia water, add an appropriate amount of water to make 100 mL, and shake well (prepared according to GB/T 603).

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

6.9.1.6 Hydrochloric acid solution II: Take 18 mL of hydrochloric acid, add an appropriate amount of water to make 100 mL, and shake well.

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

6.9.1.8 Acetate buffer (pH 3.5): Take 25 g of ammonium acetate, add 25 mL of water to dissolve, add 38 mL of hydrochloric acid solution I (6.9.1.5),

Use hydrochloric acid solution II (6.10.1.6) or ammonia solution (6.10.1.4) to accurately adjust the pH value to 3.5 (potentiometer indication), and dilute to 100 with water

mL, shake well.

6.9.1.9 Lead standard storage stock solution (1000 μg/mL): Weigh 0.1599 g of lead nitrate, place it in a 1000 mL measuring flask, add 5 mL of nitric acid and

After dissolving in 50 mL of water, dilute to the mark with water, shake well, and use as a stock solution. Or commercially available lead single element standard solution: 1000 μg/mL.

6.9.1.10 Lead standard working solution (10 μg/mL): accurately measure 2 mL of lead standard solution (6.10.1.9) and place it in a 200 mL measuring flask.

Dilute to the mark with water and shake well.

6.9.1.11 1% phenolphthalein indicator solution: take 1 g of phenolphthalein, add 100 mL of ethanol to dissolve it, and shake it evenly. The range of discoloration is pH 8.3 ~ 10.0 (colorles

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6.9.2 Test procedure

6.9.2.1 Sample solution preparation

Weigh 1 g of the sample (accurate to 10 mg), place it in a porcelain crucible, slowly burn it until it is completely carbonized, and let it cool. Add sulfuric acid (6.9.1.1) 0.5 mL ~ 1 mL to moisten, heat at low temperature until the sulfuric acid vapor is exhausted, burn it at 550℃ to completely ash, and let it cool. Add nitric acid (6.9.1.2) 0.5 mL, evaporate to dryness until the nitrogen oxide vapor is exhausted, then let cool. Add 2.0 mL of hydrochloric acid (6.9.1.3), evaporate to dryness on a water bath and add 15 mL of w Drop the ammonia solution (6.9.1.4) until the p-phenolphthalein indicator solution (6.10.1.11) is slightly red, and then add acetate buffer (6.10.1.8) 2.0 After dissolving by slight heat, transfer the Nessler's colorimetric tube, add water to dilute to 25 mL, and use it as the second tube.

6.9.2.2 Preparation of standard colorimetric solution

Take another reagent for preparing the sample solution, put it in a porcelain crucible and evaporate to dryness, add 2.0 mL of acetate buffer (6.10.1.8) and 15 mL of water, After dissolving by slight heat, transfer to Nessler's colorimetric tube, add 1.00 mL of lead standard working solution (6.9.1.11), and then dilute with water to 25 mL, as A tube.

6.9.3 Determination and result judgment

Add 5 drops of sodium sulfide solution (6.9.1.7) to the two tubes A and B respectively, shake well, place for 2 minutes, and place the same on white paper, from top to bottom See through and compare the colors of tube A and tube B with naked eyes. If tube B is not darker than tube A, it is judged to meet the requirements.

7 Inspection rules

7.1 Batch

The uniform products produced continuously with the same raw materials, the same production process, and the same shift constitute a production batch, but each batch of products Must exceed 150 tons.

7.2 Factory inspection

Among the items listed in Chapter 4, the appearance and properties, valine content, loss on drying, and pH are the factory inspection items.

7.3 Type inspection

Type inspection items are all items specified in Chapter 4 of this standard. Under normal production conditions, type inspection shall be carried out at least once every six months Test. Type inspection should also be carried out in one of the following situations:

a) When the product is finalized and put into production;

b) When there are major changes in the production process, formula or the source of the main raw materials, which may affect the product quality;

c) When production is stopped for more than 3 months and production is resumed;

d) When there is a big difference between the factory inspection result and the last type inspection result;

e) When the feed administrative department requests inspection.

7.4 Judgment rules

7.4.1 All inspected items are qualified, and the batch of products is judged to be qualified.

7.4.2 If any index in the inspection result does not meet the requirements of this standard, the samples from the same batch of products can be doubled for re-inspection. If the recheck is c If it still does not meet the requirements of this standard, the batch of products is determined to be unqualified.

7.4.3 Judgment of the limit value of each item index shall be implemented according to the comparison method of rounding value in 4.3.3 of GB/T 8170.

8 Labeling, packaging, transportation, storage and shelf life

8.1 Label

The label is implemented according to GB 10648.

8.2 Packaging

The packaging materials should be non-toxic, harmless and moisture-proof.

8.3 Transportation

During transportation, prevent packaging damage, sun exposure, rain, and transportation with toxic and hazardous substances.

8.4 Storage

Avoid sunlight and rain during storage, and prohibit mixed storage with toxic and hazardous substances.

8.4 Shelf life

For unopened products, under the specified transportation and storage conditions, the product shelf life is the same as the shelf life indicated on the label.

Appendix A

(Informative appendix)

Infrared spectrum of valine reference substance

The infrared spectrum of valine reference substance is shown in Figure A.1

through
Light
rate
/%

Figure A.1 Infrared spectrum of valine reference substance

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