Tentative Translation

# JAS 0801

# JAPANESE AGRICULTURAL STANDARD

# **Brewed vinegar**

Date of Establishment: 1979-6-8 Date of Revision: 2019-12-13

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Food and Agricultural Materials Inspection Center, Incorporated Administrative Agency

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# **Foreword**

This Japanese Agricultural Standard has been revised by the Minister of Agriculture, Forestry and Fisheries through deliberations at the Council for the Japanese Agricultural Standards the Act on Japanese Agricultural Standards. This edition replaces the previous edition of JAS for Brewed vinegar (JAS 0801:2019), which has been technically revised.

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# JAPANESE AGRICULTURAL STANDARD (Tentative Translation)

(Tentative Translation) 0801 : 2019

JAS

# **Brewed vinegar**

# 1 Scope

This document specifies the quality of brewed vinegar.

#### 2 Normative references

The following documents are referred to in the text in such a way that some or all of their content constitutes requirements of this document. The latest edition of the referenced documents (including any amendments) apply.

CODEX STAN 192, General Standard for Food Additives

JIS K 0557, Water used for industrial water and wastewater analysis

JIS R 3505, Volumetric glassware

# 3 Terms and definitions

For the purposes of this document, the following terms and definitions apply.

#### 3 1

# brewed vinegar

any of the following:

- a) liquid seasoning obtained by acetous fermentation of *moromi* made from grains (including processed products such as *sake kasu*; the same applies hereinafter), fruits (including processed products such as extracted fruit juice, fruit wine; the same applies hereinafter), vegetables (including processed products such as extracted vegetable juice; the same applies hereinafter.), other agricultural products (including sugar canes, etc. and the extracted juice thereof; the same applies hereinafter) or honey, or these *moromi* with additional alcohol or sorts of sugar; and processed without using glacial acetic acid or acetic acid;
- b) liquid seasoning obtained by acetous fermentation of alcohol or alcohol with additional saccharified grains, fruits, vegetables, other agricultural products or honey; and processed without using glacial acetic acid or acetic acid;
- c) a mixture of a) and b);
- d) liquid seasoning prepared by adding sorts of sugar, acidifier (excluding glacial acetic acid and acetic acid), seasoning (amino acid, etc.), salt, etc. (excluding spice) to a), b) or c); and its content percentages of non-volatile acid, total sugar or total nitrogen are less than 1,0 %, 10,0 % or 0,2 %, respectively (each of content percentage shall be converted as if their acidity being 4,0 %)

#### 3.2

# grain vinegar

brewed vinegar (3.1) prepared by using one or more types of grains as the ingredient (limited to the products prepared without using agricultural products other than grains and fruits or honey); and, of which the used amount of grains in total is 40 g or more per 1 L of brewed vinegar (3.1)

#### 3.3

#### fruit vinegar

brewed vinegar (3.1) prepared by using one or more types of fruits as the ingredient (limited to the products prepared without using agricultural products other than grains and fruits or honey); and, of which the used amount of fruits in total is 300 g or more as the extracted fruit juice per 1 L of brewed vinegar (3.1)

#### 3.4

# rice black vinegar

grain vinegar (3.2) prepared by using rice (excluding milled rice with all of the bran layer removed from husked rice; hereinafter the same applies in 3.4) or a mixture of rice and wheat or barley as the only ingredient; and, of which the used amount of rice is 180 g or more per 1 L of grain vinegar (3.2); and colored brown or blackish brown by fermentation and aging

#### 3.5

#### rice vinegar

grain vinegar (3.2) of which the used amount of rice is 40 g or more per 1 L of grain vinegar (3.2) [excluding rice black vinegar (3.4)]

#### 3.6

# apple vinegar

*fruit vinegar* (3.3) of which the used amount of extracted juice of apple is 300 g or more per 1 L of *fruit vinegar* (3.3)

# 3.7

# grape vinegar

*fruit vinegar* (3.3) of which the used amount of extracted juice of grape is 300 g or more per 1 L of *fruit vinegar* (3.3)

# 4 Quality

# 4.1 Properties

The properties shall be of having a particular color and luster, of having a good flavor, and of not having an objectionable taste nor odor.

#### 4.2 Acidity

The acidity shall be 4,0 % (4,2 % for grain vinegar and 4,5 % for fruit vinegar) or more, when tested by the method specified in 6.2; provided, however, that, for business use products, the acidity shall conform to each of the aforementioned values, and also to the labeled acidities.

4.3 Soluble solids excluding salt content (excluding the products made from one type of grain, fruits, vegetables, other agricultural products or honey as the only ingredient; rice

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# black vinegar; and the business use products made without sorts of sugar, hydrolyzed protein and additives)

The soluble solids excluding salt content shall be as follows, when tested by the methods specified in 6.3:

#### a) Grain vinegar

1,3 % to 8,0 % (for rice vinegar, 1,5 % to 8,0 %; provided, however, that, for those made without sorts of sugar, hydrolyzed protein and additives, 1,5 % to 9,8 %);

# b) Fruit vinegar

1,2 % to 5,0 % (for apple vinegar, 1,5 % to 5,0 %);

# c) Brewed vinegar other than grain vinegar or fruit vinegar

1,2 % to 4,0 %;

### d) Vinegar to be diluted for use

for grain vinegar, the acidity of which is prepared at 4,2 %, the value specified in a); for fruit vinegar, the acidity of which is prepared at 4,5 %, the value specified in b); and, for brewed vinegar other than grain vinegar or fruit vinegar, the acidity of which is prepared at 4,0 %, the value specified in c).

# 4.4 Total nitrogen content (limited to rice black vinegar)

The total nitrogen content shall be 0,12 % or more, when tested by the method specified in 6.4.

### 4.5 Degree of coloring (limited to rice black vinegar)

The degree of coloring shall be 0,30 or more, when tested by the method specified in 6.5.

# 4.6 Ingredients

Only the following ingredients may be used:

- a) grains, fruits, vegetables, other agricultural products and honey;
- **b)** alcohol (limited to those manufactured by distilling the liquid obtained from the alcohol fermentation of carbohydrates such as starch, sorts of sugar);
- c) sorts of sugar, salt and hydrolyzed protein.

#### 4.7 Additives

The additives shall be as follows:

- **a)** They shall conform to the provisions of 3.2 of CODEX STAN 192, and the conditions of use conform to the provisions of 3.3 of the document; provided, however, that, for rice black vinegar, they shall not use additives at all;
- b) The amounts of use shall be accurately recorded and the record shall be kept;
- c) Information that the additives conform to the provision of a) shall be provided to general consumers by one of the following methods; provided, however, that this does not apply to the cases where additives

are added to business use products:

- 1) methods of making it available for public inspection via the internet;;
- 2) methods of displaying it on brochures, leaflets and any other publications where it is easily seen by general consumers;
- 3) methods of displaying it at a place where it is easily seen by general consumers in stores;
- **4)** methods of providing it to general consumers at their request, while clearly indicating the contact address on the products.

### 4.8 Net contents

The net contents shall conform to the declared weight.

# 5 Information on labeling, method of labeling and style of labeling, etc. (limited to products for business use)

In addition to complying with the provisions of the Food Labeling Standards (Cabinet Office Order No. 10 of 2015), the acidity of the content shall be shown in percentage to the first decimal place with the unit being clearly indicated, in an easily read area of the container or package or invoice.

# 6 Test methods

#### 6.1 General

Reagents and apparatus used for the testing shall be as follows:

- a) Water, grade A2 specified in JIS K 0557, or of equivalent or higher quality.
- b) Reagents, conforming to the standards such as the special grade of Japanese Industrial standards.
- **c) Kjeldahl catalysts,** mixture of potassium sulfate, 9 g, and copper (II) sulfate pentahydrate, 1 g; homogenized with mortar.
- **d) Bromocresol green and methyl red mixture indicator,** prepared by dissolving bromocresol green and methyl red in 95 % ethanol to contain 0,15 g of bromocresol green and 0,10 g of methyl red in 200 mL.
- **e) Glycine,** of 99 % or higher purity, with a description of the nitrogen proportion.
- f) Volumetric glassware, class A specified in JIS R 3505, or of equivalent or higher quality.
- **g) Potentiometric titrator,** used with a silver electrode for an indicator electrode, and a silver-silver chloride electrode as a reference electrode, or with a composite silver electrode (limited to 6.3).
- **h) Variable power digestion apparatus,** capable of boiling a beaker which contains 2 to 3 boiling stones and 100 mL of water within 5 min on the heat source which has been maintained at the maximum output for 10 min.

- i) **Block digestor,** capable of boiling a digestion tube which contains 2 to 3 boiling stones and 50 mL of water, on the heating block preset at 400 °C, within 2 min 30 s.
- j) Automatic distillation apparatus, capable of rapidly and automatically conducting steam distillation of the Kjeldahl method (including a combined apparatus of automatic distillation apparatus and automatic titrator).
- k) Apparatus for measuring total nitrogen by combustion method, with the following characteristics:
  - 1) equipped with a furnace capable of keeping the operating temperature at least at 870 °C or above to pyrolyze a sample in oxygen (of 99,9 % or higher purity);
  - 2) having a structure capable of separating free nitrogen (N<sub>2</sub>) from other combustion products to measure nitrogen (N<sub>2</sub>) with a thermal conductivity detector;
  - 3) having a mechanism for converting nitrogen oxide (NOx) to nitrogen  $(N_2)$ ;
  - 4) having an average value of the nitrogen content within  $\pm 0.15$  % of the theoretical value and the relative standard deviation at 1,3 % or below in 10 consecutive measurements with nicotinic acid (of 99 % or higher purity).

# 6.2 Acidity

# 6.2.1 Preparation of sample

Pour, with accuracy, 3 mL to 10 mL of the sample (the amount of sample shall be determined according to the sodium hydroxide standard solution in 6.2.3, the amount of which will be 10 mL to 20 mL) into a container of capacity about 200 mL, using a volumetric pipette, and add 100 mL of water which does not contain carbon dioxide. Use this as the sample solution.

### 6.2.2 Calibration of hydrogen ion exponent (pH) meter

Carry out the calibration of pH meter at least at two points between which pH 8,2 lies, using pH standard solution.

#### 6.2.3 Titration

The titration shall be either of the following. It shall be carried out within 30 min after the sampling, in order to prevent volatile acid substance to be volatilized:

### a) Manual titration with pH meter

Immerse a glass electrode of the pH meter into the sample solution, and, while stirring well, carry out the titration with 0,5 mol/L sodium hydroxide standard solution. The end point shall be at pH 8,2  $\pm$ 0,3, and make sure that the pH within that range last for more than 30 s. In addition, carry out the titration on the same procedure, without adding the sample, as the blank test.

NOTE A phenolphthalein indicator may be added to the test solution as a guide to determine the endpoint.

#### b) Automatic titration (method using potentiometric titrator)

Following the operation procedure of the potentiometric titrator, set the end point at pH 8,2. Immerse an electrode into the sample solution, and, while stirring, carry out the titration with 0,5 mol/L sodium hydroxide standard solution. In addition, carry out the titration on the same procedure, without adding the sample, as a blank test.

#### 6.2.4 Calculation

Calculate the acidity, in terms of acetic acid, by the following formula:

Acidity (%) = 
$$\frac{0.03 \times (T - B) \times F}{V} \times 100$$

where

- *T* is the volume of 0,5 mol/L sodium hydroxide standard solution required for the titration of the sample (mL);
- *B* is the volume of 0,5 mol/L sodium hydroxide standard solution required for the titration of the blank test (mL);
- *F* is the factor of 0,5 mol/L sodium hydroxide standard solution;
- *V* is the volume of the sample (mL);
- 0,03 is the mass of acetic acid equivalent to 1 ml of 0,5 mol/L sodium hydroxide solution (g).

# 6.3 Soluble solids excluding salt content

#### 6.3.1 Measurement of the soluble solids content

Pour 10 mL of the sample into a glass weighing tube or a platinum flat-bottomed dish, 50 mm in diameter, weighed in advance, evaporate to dryness on the water bath, and carry out, three times, the procedure of adding water and evaporating to dryness, dry until the constant mass is obtained at  $105\,^{\circ}$ C and weigh; and the soluble solids content is calculated as a percentage of the mass obtained relative to the volume of the sample.

# 6.3.2 Measurement of the salt content

# 6.3.2.1 Measurement procedure

The measurement procedure of the salt content shall be either of the followings; provided, however, that, for 6.3.2.1 b), the procedure shall not be applied to the colored sample on which the end point is difficult to judge:

#### a) Automatic titration (method using potentiometric titrator)

Put 3 mL to 10 mL of the sample into a 100 mL or 200 mL beaker with a volumetric pipette, add water to immerse the electrode in the solution, attach to the potentiometric titrator, and, while stirring, carry out the titration with 0,1 mol/L silver nitrate solution. Follow the operation procedure of the titrator and detect the end point. When the end point is not detected, the titration value shall be 0 mL.

# b) Colorimetric titration by Mohr method

Put 3 mL to 10 mL of the sample into a porcelain evaporating dish or a 200 mL conical flask with a volumetric pipette, adjust the hydrogen ion exponent at pH 6.5 to pH 10 by adding 0,25 mol/L sodium carbonate solution, add, as an indicator, 1 mL of 2 % potassium chromate, and carry out the titration with 0,1 mol/L silver nitrate solution. The end point shall be when the color of the solution turns either pale orange or, slightly, red brown. When the color which clearly exceeds the end point is shown with one drop, the titration value shall be 0 mL.

#### 6.3.2.2 Calculation

Calculate the salt content by the following formula:

Salt content (%) = 
$$\frac{0,005 844 \times T \times F}{V} \times 100$$

where

- *T* is the volume of 0,1 mol/L silver nitrate solution required for the titration (mL);
- *F* is the factor of 0,1 mol/L silver nitrate solution;
- *V* is the volume of the sample (mL);

0,005 844 is the mass of sodium chloride, equivalent to 1 mL of 0,1 mol/L silver nitrate solution (g).

# 6.3.3 Calculation of soluble solids excluding salt content

The soluble solids excluding salt content shall be the value given by subtracting salt content (see 6.3.2) from soluble solids content (see 6.3.1).

#### 6.4 Total nitrogen content

#### 6.4.1 General

The total nitrogen content shall be measured either by the Kjeldahl method or the combustion method.

#### 6.4.2 The Kjeldahl method

# 6.4.2.1 Measurement procedure

The measurement procedure shall be as follows:

### a) Digestion of sample

The digestion of the sample shall be either of the following:

#### 1) In the case of using a variable power digestion apparatus

1.1) Pour, with accuracy, 5 mL to 15 mL of the sample [the amount of the sample shall be determined according to the sulfuric acid standard solution in 6.4.2.1 c), the amount of which will be 10 mL to 25 mL; the same applies hereinafter] into a 300 mL Kjeldahl flask, with a volumetric pipette, and add approximately 10 g of Kjeldahl catalysts and approximately 15 mL of sulfuric acid. While stirring well, gently add approximately 10 mL of 30 % hydrogen peroxide solution; and put it on the heat source of the digestion apparatus which has been kept

warm;

- **1.2)** Start heating at 200 °C, and, as the bubbling comes to an end, gradually turn up to 400 °C. After the digestion solution becomes clear, keep it heating for 90 min to 120 min;
- **1.3)** After the digestion is over, allow it to cool down to room temperature, add approximately 70 mL to 100 mL of water, and dissolve the decomposed sample;
- **1.4)** Carry out the same procedure from 1.1) to 1.3) without putting in the sample (blank test).

### 2) In the case of using a block digestor

- 2.1) Weigh, with accuracy, 5 mL to 15 mL of the sample into a Kjeldahl digestion tube with a capacity between 250 mL to 300 mL, using a volumetric pipette, and add 10 g of Kjeldahl catalysts and approximately 15 mL of sulfuric acid. While stirring well, gently add approximately 10 mL of 30 % hydrogen peroxide solution, and put it on the heat source of the block digestor which has been kept warm;
- **2.2)** Start heating at 200 °C, and, as the bubbling comes to an end, gradually turn up to 400 °C. After the digestion solution becomes clear, keep it heating for 90 min to 120 min;
- **2.3)** Carry out the same procedure from 2.1) and 2.2) without putting in the sample (blank test).

# b) Distillation

The distillation shall be as follows:

# 1) In the case of using a steam distillation apparatus [in the case of decomposing the sample by a) 1)]

Put 30 mL of 1 % to 4 % boric acid solution into a receiver for distillate of 300 mL or more capacity (hereinafter referred to as "the receiver"), add a few drops of bromocresol green and methyl red mixture indicator, and place it so that the distillate outflow port is immersed in the solution of the receiver. Connect the Kjeldahl flask which contains the digestion solution to the distillation apparatus, add 25 % to 45 % sodium hydroxide solution for neutralization (which contains 28 g or more of sodium hydroxide), and distil until approximately 100 mL or more of the distillate is obtained. Take the outflow port out of the solution and wash the port end with a small amount of water.

# 2) In the case of using an automatic distillation apparatus [in the case of decomposing the sample by a) 2)]

Carry out the distillation following the operation procedure of the apparatus. Put 25 mL to 50 mL of the solution, to which 20 mL to 50 mL of 1 % to 4 % boric acid solution and a few drops of bromocresol green and methyl red mixture indicator are added, into the receiver; and attach it so that the distillate outflow port is immersed in the solution of the receiver. Add 40 mL to 60 mL of water and 25 % to 45 % sodium hydroxide solution for neutralization (which contains 28 g or more of sodium hydroxide) and distill until approximately 100 mL or more of the distillate is

obtained. Take the outflow port out of the solution and wash the port end with a small amount of water. With a combined apparatus of automatic distillation apparatus and automatic titrator, or the like, carry out a distillation and a titration in a manner suitable for the apparatus.

# c) Titration

The titration shall be either of the following:

# 1) Manual titration (a method in which the end point of titration is visually judged by the discoloration of the indicator)

Titrate the distillate with 0,05 mol/L standard solution of sulfuric acid. Judge the end point of titration as the color of solution changes from green through impurity-containing colorless to pale greyish red. For the distillate obtained from the sample for blank test, titrate it in the same procedure.

# 2) Automatic titration (a method with an apparatus which automatically judges the end point of titration)

Titrate the distillate with 0,05 mol/L standard solution of sulfuric acid. Detect the end point following the operation procedure of the apparatus. For the distillate obtained from the sample for blank test, titrate it in the same procedure.

#### 6.4.2.2 Calculation

Calculate the total nitrogen content by the following formula. When the color which clearly exceeds the end point with one drop, on the titration of the blank test, the titration value shall be 0 mL.

Total nitrogen content (%) = 
$$\frac{1,401 \times 10^{-3} \times (T - B) \times F}{V} \times 100$$

where

- *T* is the titration value of the sample (mL);
- *B* is the titration value on the blank test (mL);
- *F* is the factor of 0,05 mol/L sulfuric acid;
- *V* is the volume of the sample (mL);

 $1,401\times10^{-3}$  is the mass of nitrogen, equivalent to 1 mL of 0,05 mol/L sulfuric acid (g).

### 6.4.3 The Combustion method

### 6.4.3.1 Measurement procedure

The measurement procedure shall be as follows:

a) Weigh, with accuracy, the required amount of the reference standard for preparing calibration curves [use glycine or other reference standards with the same purity (excluding nicotinic acid)], to the nearest 0.1 mg or less, following the operating procedure of the apparatus for measuring total nitrogen by combustion method, and dissolve with water (standard solution). Weigh, with accuracy, the standard

solution to the nearest 0.1 mg or less, carry out the measurement by a procedure suitable for the apparatus, and draw the calibration curve. The amount of the standard solution for drawing the calibration curve shall match the weight of the sample;

**b)** Weigh, with accuracy, approximately 200 mg to 1 000 mg of the sample, to the nearest 0,1 mg or less, and carry out the measurement by a procedure suitable for the apparatus.

#### 6.4.3.2 Calculation

Calculate the total nitrogen content as follows:

- a) Calculate the nitrogen content (%) from a calibration curve;
- **b)** Measure the relative density of the sample and convert the nitrogen content calculated in mass into volume.

# 6.5 Degree of coloring

The degree of coloring shall be the value determined by putting the sample into a cell, 10 mm in width, and measuring absorbance at a wavelength of 420 nm with a spectrometer.