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Organization standard

T/SDBFIA 001—2022

D-Allulose

D-Allulose, D-Psicose

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Foreword

This document is drafted in accordance with the provisions of GB/T 1.1-2020 "Guidelines for Standardization Work Part 1: Structure and Drafting Rules for Standardization Documents".

This document was proposed and managed by Shandong Biological Fermentation Association.

D-Allulose

1 Scope

This document specifies the technical requirements, hygienic requirements during the production, inspection methods, inspection rules, marking, packaging, transportation and storage of D-Allulose .

This document applies to D-Allulose.

2 Normative references

The following documents are indispensable for the application of this document. For dated references, only the dated version applies to this document. For undated reference documents, the latest version (including all amendments) applies to this document.

GB 14881 National Food Safety Standard -- General Hygienic Practice for Food Production

GB 15203 Starch sugar

GB/T 191 Packaging--Pictorial markings for handling of goods

GB 2760 National Food Safety Standard -- Standard for Uses of Food Additives

GB 2762 National Food Safety Standard -- Maximum Levels of Contaminants in Foods

GB 31637 Edible starch

GB 4789.2 National Food Safety Standard -- Microbiological Examination of Total Bacterial Count for Food Hygiene

GB 4789.3 National Food Safety Standard -- Microbiological Examination of Coliform Bacteria Count Food Hygiene

GB 5009.11 National Food Safety Standard -- Determination of Total Arsenic and Inorganic Arsenic in Foods

GB 5009.12 National Food Safety Standard -- Determination of Lead in Foods

GB 5009.3 National Food Safety Standard -- Determination of Moisture in Foods

GB 5009.3 4 National Food Safety Standard -- Determination of Sulfur Dioxide in Foods

GB 5009.4 National Food Safety Standard -- Determination of Ash Content in Food

GB 5749 Standards for Drinking Water Quality

GB/T 601 Chemical Reagent - Preparation of Reference Titration Solutions

GB/T 602 Preparation of Standard Solution for Determination of Impurities

GB/T 603 Preparation of Reagent Solutions for Use in Test Methods

GB /T 6682 Water for Analytical Laboratory Use—Specification and Test Methods

3 Terms and Definitions, Chemical Name, Molecular Formula, Structural Formula, Relative Molecular Mass

3.1 Terms and Definitions

D-Allulose

It is a liquid or crystalline solid product with the epimer corresponding to the third carbon of D-fructose as the main component, which is made from edible starch or starch sugar through enzymatic conversion, purification and separation, and refining.

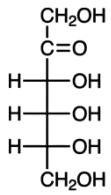
3.2 Chemical name

D-Allulose (D-Allulose, D-Psicose)

3.3 Molecular formula

$C_6H_{12}O_6$

3.4 Structural formula



3.5 Relative molecular mass

180.16 (according to the international relative atomic mass in 2018)

4 Technical requirements

4.1 Raw and auxiliary materials

4.1.1 Edible starch

Should comply with the provisions of GB 31637.

4.1.2 Starch sugar

Should comply with the provisions of GB 15203.

4.1.3 Water for production

Should comply with the provisions of GB 5749 .

4.1.4 Other raw and auxiliary materials

It should meet the requirements of relevant food safety standards.

4.2 Sensory indicators

It should meet the requirements in Table 1.

Table 1 Sensory Indicators

Item	Indicators	
	Solid	Liquid
State	Powder or granule	Transparent, viscous liquid
Color	white to near white	Light yellow or yellow
Smell, taste	Odorless, with a soft, refreshing sweet taste	Odorless, with a soft, refreshing sweet taste
Impurities	No visible impurities with normal vision	No visible impurities with normal vision

4.3 Physical and chemical indicators

It should meet the requirements in Table 2.

Table 2 Physical and chemical indicators

Item		Indicators	
		Solid	Liquid
Moisture, %	≤	1.0	——
Solid substance, %	≥	——	70
Ash content, %	≤	0.5	
D-Allulose content (dry basis), %	≥	98.5	95.0
pH		4.0-7.0	3.0－7.0
Residual sulfur dioxide, mg/kg	≤	10	

4.4 Microbial indicators

It should meet the requirements in Table 3.

Table 3 Microbial indicators

Item		Collection plan [a] and limit amount		
		n	c	m
Total Aerobic Count, CFU/g	≤	3	0	10^3

Coliforms, CFU/g	≤	3	0	10
Note: [a] The collection and processing of samples shall be carried out according to GB 4789.1.				

4.5 Pollutant Limit Requirements

It should meet the limit requirements of "total arsenic" and "lead" in "sugar and starch sugar" in GB 2762, see Table 4.

Table 4 Pollutant limit indicators

Item		Indicators	
		Solid	Liquid
Total arsenic (calculated as As), mg/kg	≤	0.5	
Lead (as Pb), mg/kg	≤	0.5	

5 Food Additive Requirements

Should comply with the provisions of GB 2760.

6 Hygienic requirements for production and processing

Should comply with the provisions of GB 14881.

7 Inspection method

7.1 General requirements

The water used in this document shall comply with the water specifications in GB/T 6682 unless other requirements are specified; the reagents used shall refer to analytical reagent unless other specifications are specified. The standard titration solution, standard solutions for impurity determination, preparations and products used in the analysis shall be prepared according to the provisions of GB/T 601, GB/T 602 and GB/T 603 unless other requirements are specified.

7.2 Sensory test

7.2.1 Liquid

Take an appropriate amount of sample and put in a colorless, clean, dry beaker , put it in a bright place, observe its color and state, and check whether there are impurities visible in normal vision; take an appropriate amount of sample to smell its taste, put it in the mouth and taste it (Before the second sample taste, you should rinse your mouth with water), and make records.

7.2.2 Solid

Take an appropriate amount of sample and put it in a clean and dry container. Under suitable natural light, observe the color and state of the sample to check whether there are any impurities visible to the naked eye ; Before the second sample taste, you should rinse your mouth with water), and make records.

7.3 Physical and chemical tests

7.3.1 Moisture

Should apply Karl Fischer method in GB 5009.3 .

7.3.2 Solid

7.3.2.1 Instruments and equipment

7.3.2.1.1 Abbe refractometer: Accuracy is 0.0001 units.

7.3.2.1.2 Glass Rod: The end is bent and flattened.

7.3.2.2 Instrument Calibration

At 20°C, the refractive index of the refractometer with testing water is 1.3330, which is equivalent to zero content of dry matter (solid matter). The instrument is calibrated at least once a day.

Analysis steps: place the refractometer in a place with sufficient light, adjust the temperature of the refractometer prism to 20°C, separate the two prisms, add a small amount of sample (1-2 drops) to the fixed prism surface with a glass rod (the glass rod must not touch prism surface, and the sample coating time should be less than 2 seconds) , immediately close the prism and stay for a few minutes to allow the sample to reach the temperature of the prism. Adjust the spiral of the prism until the field of view is divided into light and dark parts, turn the compensator knob to eliminate iridescence and make the dividing clear line between light and dark, continue to adjust the spiral and align the dividing line between light and dark on the cross line, and read the solid content from the scale, and then re-read immediately, take the average as a measurement value. Clean and dry the two prisms, and perform the second measurement on the same sample as above. Take the arithmetic mean of the two times and report the result.

7.3.2.3 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions should not exceed 1% of the arithmetic mean.

7.3.3 pH

7.3.3.1 Instruments and Equipment

pH meter: the accuracy is 0.01pH, equipped with glass electrode and calomel electrode (or combination electrode).

7.3.3.2 Analysis steps

Debug and calibrate the pH meter according to the instrument instruction manual.

Take an appropriate amount of sample, and prepare a test solution with about 30% dry matter in boiled and cooled water with a pH of 5.0-7.0. Then, rinse the electrode probe with water, dry it gently with filter paper, then insert the electrode into the sample solution, adjust the temperature regulator so that the temperature indicated by the instrument is the same as the temperature of the solution, and do the reading when stable. The result is expressed to one decimal place .

7.3.3.3 Precision

The absolute difference between two independent measurement results obtained under repeatability conditions should not exceed 3% of the arithmetic mean.

7.3.4 Ash content

Measured according to the method specified in GB 5009.4 .

7.3.5 D-Allulose content

Measured according to the method specified in Appendix A.

7.3.6 Residual amount of sulfur dioxide

Measured according to the method specified in GB 5009.34 .

7.4 Microbiological test

7.4.1 Total Aerobic Count

Measured according to the method specified in GB 4789.2 .

7.4.2 Coliforms

Measured according to the method specified in GB 4789.3 .

7.5 Inspection of pollutant limit

7.5.1 Lead

Measured according to the method specified in GB 5009.12 .

7.5.2 Total Arsenic

Measured according to the method specified in GB 5009.11 .

8 Inspection rules

8.1 Batch formation and sampling

8.1.1 Batch formation

The products with uniform quality, which are continuously produced with the same raw materials, the same formula, the same process and on the same production line, are a batch.

8.1.2 Sampling

8.1.2.1 The inspection of each batch of products shall draw samples according to Table 5.

Table 5 Product Sampling Form

Batch range (minimum packaging unit)	Number of samples (minimum packaging unit)	Number of sample taken from per packaging unitt [a] (bottles, bags, barrels)
<100	2	1
100 ~ 500	4	1
>500	6	1
[a] The number of unit packaging refers to the small packaging unit in the large packaging.		

8.1.2.2 Every tank truck loaded with products must be inspected.

8.1.2.3 Samples from tank trucks and barreled products should be taken from 10cm below the liquid level, and the sampler should comply with relevant regulations.

8.1.2.4 According to the principle of equal sampling from each sample, each sample volume of products in tank trucks shall not be less than 1kg; each sample volume of product in drums shall not be less than 1kg; each sample volume of product in bottles and bags shall not be less than 600g.

8.1.2.5 Use a clean and dry sampling tool to place the samples in three clean and dry containers, seal them, and indicate the product name, batch number, sampling time, and the name of the sample-taking person, etc. One for physical and chemical testing, one for microbiological testing, and another is sealed for future reference.

8.2 Inspection

8.2.1 Factory inspection

8.2.1.1 Before leaving the factory, the products should be carried out batch by batch according to the regulations in this document, and the products can only leave the factory after the inspection meets the requirements of this document.

8.2.1.2 Factory inspection items

——Liquid products: sensory, solid substance content, D-Allulose content, pH, total number of colonies, coliform bacteria;

——Solid product: sensory, moisture, D-Allulose content, pH, total number of colonies, coliform bacteria.

8.2.2 Type inspection

The type inspection items are all the items in the requirements of this document. Generally, the type inspection is once every six months. In one of the following situations, the type inspection should also be carried out:

a) When there are major changes in raw and auxiliary materials;

b) When changing key processes or equipment;

c) When the production of the trial-produced product or the normal product is stopped for more than three months, and the production is resumed;

d) When there is a large difference between the factory inspection result and the previous type inspection;

e) When required by relevant laws and regulations.

8.3 Judgment rules

8.3.1 After sample inspection, all inspection items meet the requirements, and it is determined that the product complies with this document;

8.3.2 If one or two of the inspection items do not meet the requirements, twice the amount of samples should be taken from the same batch of products for re-inspection, and the re-inspection results shall prevail. If there is still one item that does not meet the requirements, it is determined that the batch of products does not meet the requirements of this document. If there are three or more items in the inspection results that do not meet the requirements, it is determined that the batch of products does not meet the requirements of this document. If one of the microbiological indicators does not meet the requirements, it is determined that the batch of products does not meet this document.

9 Marking, packaging, transportation, storage

9.1 Marking

The pictogram of product packaging, storage and transportation should meet the requirements of GB/T 191 .

9.2 Packaging

9.2.1 The packaging container should be clean and undamaged.

9.2.2 For tank trucks transporting liquid products, the tank truck should be special for the use.

9.3 Transportation

9.3.1 The means of transport should be clean;

9.3.2 It should not be mixed with toxic, harmful, corrosive and odorous items, and should be protected from moisture, pressure, and exposure to the sun. It should be handled with care when loading and unloading, and the packaging should not be directly hooked.

9.4 Storage

9.4.1 The product should be stored in a ventilated, dry and clean warehouse, and exposure to the sun and rain is strictly prohibited. Fire is strictly prohibited.

9.4.2 It should not be stored together with toxic, harmful, corrosive and odorous items.

Appendix A
Measurement of D-Allulose content

A.1 Reagents

a) Water: Ultra-pure water;

b) D-Allulose standard product, purity ≥ 95%, prepare 6 standard solution series with different concentrations within the range of 0.1 mg/mL to 10 mg/mL.

A.2 Instruments

a) High-performance liquid chromatography (equipped with refractive index detector and constant column-temperature system);

b) Mobile phase vacuum filtration degassing device;

c) Analytical balance (accuracy 0.0001g) ;

d) Quantitative loop;

e) Microsampler 50μL or 100μL.

A.3 Reference chromatographic conditions

a) Chromatographic column: 6.5mm×300mm calcium-type cation exchange resin chromatographic column or other equivalent chromatographic column.

b) Mobile phase: Ultra-pure water;

c) Detector temperature: 40°C;

d) Column temperature: 85°C;

e) Flow rate: 0.5mL/min;

f) Injection volume: 10μL;

g) The above chromatographic conditions can be adjusted according to the actual situation.

A.4 Draw standard curve

Under the above-mentioned chromatographic conditions, the standard solution of D-Allulose was used to inject samples respectively, and a standard curve was made based on the concentration of the standard sample versus the peak area. The linear correlation coefficient should be above 0.9990.

A.5 Preparation of sample solution

Weigh an appropriate amount of liquid or solid sample (the content of various sugars should be within the linear range of the standard curve in A.4), dissolve in water to a volume of 50mL and mix well, filter with a 0.22μm or 0.45μm microporous membrane, and the filtrate is used as the The test solution is ready for use.

Note: The sample can be diluted according to the requirements of the standard curve range, and the dilution factor is recorded as D.

A.6 Sample determination

Inject the prepared sample solution, and characterize the chromatographic peak of D-Allulose in the sample according to the retention time of the standard. Calculate the content of D-Allulose by external standard method or area normalization method (external standard method is the arbitration method).

A.7 Calculation of results

A.7.1 External standard method

of D-Allulose is calculated according to formula (1):

$$X_{\mathrm{i}} = \frac{\rho_i \times V_i \times D}{1000 \times m} \times 100 \quad \text{.....(1)}$$

In the formula:

X_{i} -- content of D-Allulose in the sample (on a dry basis), g/100g;

ρ_i -- check the curve to get the mass concentration of D-Allulose in the solution to be tested, mg/mL;

V_i -- the dilution volume of the sample, mL;

D -- the dilution factor of the sample;

m -- the mass of the sample. In the liquid, it refers to the mass of the dry matter in the weighed sample, and in the solid, it refers to the mass of the weighed sample after removing moisture, g.

The calculation result retains one decimal place.

A.7.2 Peak area normalization method

of D-Allulose is calculated according to formula (2):

$$P_{\mathrm{i}} = \frac{A_i}{\sum A_{\mathrm{i}}} \times 100 \quad \text{.....(2)}$$

In the formula:

P_{i} -- content of D-Allulose in the sample (on a dry basis), g/100g;

A_{i} -- the peak area of D-Allulose in the sample;

$\sum A_{\mathrm{i}}$ -- the sum of the peak areas of all components in the sample.

The calculation result retains one decimal place.

A.8 Precision

The absolute difference between the two independent measurement results obtained under repeatability measurement conditions shall not exceed 5% of the arithmetic mean value.