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भारतीय मानक मसौदा

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(आइ एस 6798 का दूसरा पुनरीक्षण)

*Draft Indian Standard*

**OCTYL GALLATE, FOOD GRADE - SPECIFICATION**

*(Second Revision of IS 6798)*

**ICS No. 67.220.20**

Food Additives Committee, FAD 08

**Last Date of Comments:** 15 February 2025

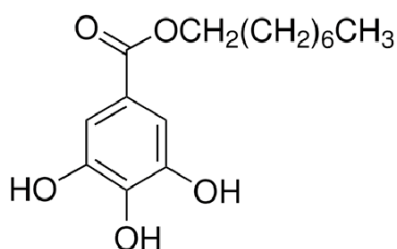
**FOREWORD**

*(Formal clause would be added later)*

Food additives are added to improve the appearance, flavour, texture or storage properties, etc of the processed foods. As certain impurities in these substances have been found to be harmful, it is necessary to have a strict quality control of these food additives. A series of standards have, therefore, been prepared to cover purity and identification of these substances. These standards would help in checking purity, which is required to be checked at the stage of manufacture, for it is extremely difficult to detect the impurity once these substances have been added to the processed foods. Besides, these standards are intended to guide the indigenous manufacturers in making their product conform to specifications that are accepted by scientists, health authorities and national/ international bodies.

Use of octyl gallate, food grade is permitted under the *Food Safety and Standards (Food Products Standards and Food Additives) Regulations, 2011* as an anti-oxidant in edible oils and fats except *GHEE* and butter.

Its chemical names are octyl gallate and n-octyl ester of 3, 4, 5-trihydroxybenzoic acid. Its empirical formula is  $C_{15}H_{22}O_5$ . Its molecular mass is 282.34. Structural formula of octyl gallate is as follows:



**January 2025**

This standard was first published in 1972. In the preparation of this standard, considerable assistance was derived from Compendium of Food Additive Specifications, Volume 2, Joint FAO/WHO Expert Committee on Food Additives (JECFA), 1992 and this standard is harmonized with the standard of FAO/WHO.

It was first revised in 1997 to the requirement for solubility property to keep it in line with Food Chemical Codex NRC; to provide limits for heavy metals, chlorinated organic compounds, free acid and absorption; and to provide for 'directions for storage' and 'expiry/best before date' under marking clause.

In this revision, two amendments issued to the previous version of the standard has been incorporated and the following major changes have been made:

- a) The requirement for heavy metals has been removed as the limit of lead (contaminant in food colours) is already covered through the standard; and
- b) The marking requirements have been updated.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 2002 'Rules for rounding off numerical values (revised)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

## **1 SCOPE**

This standard prescribes the requirements and methods of sampling and test for octyl gallate, food grade.

## **2 REFERENCES**

The following standards contain provisions, which through reference in this text, constitute provision of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard is encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No.</i>	<i>Title</i>
IS 1070 : 2023	Reagent grade water – Specification ( <i>fourth revision</i> )
IS 1699 : 1995	Methods of sampling and test for food colours ( <i>second revision</i> )
IS 2362 : 1993	Determination of water by Karl Fischer method - Test method ( <i>second revision</i> )

## **3 DESCRIPTION**

Octyl gallate shall be white to creamy-white odourless solid which may have a slightly bitter taste. The material shall be insoluble in water and freely soluble in ethanol, ether, propylene glycol and fat.

NOTE - The solubility is intended only as information regarding approximate solubility and is not to be considered as a quality requirement and is of minor significance as a means of identification or determination of purity.

## **4 REQUIREMENTS**

### **4.1 Identification Tests**

**4.1.1** Melting range shall be 99 °C to 102 °C after drying at 90 °C for 6 h.

**4.1.2** Add 1 ml of ammonium hydroxide to 5 ml of one percent ethanolic solution of octyl gallate. A pink to red colour shall appear.

**4.1.3** *TLC separation of Gallate Esters* — Use a thin-layer plate prepared with silica gel G. Prepare a sample solution by dissolving 10 mg of sample in 10 ml ethanol. Prepare control solution A by dissolving 10 mg of octyl gallate in 10 ml ethanol and control solution B by dissolving 10 mg of propyl gallate and 10 mg of octyl gallate in 10 ml ethanol.

**4.1.3.1** Place 5 µl of each solution on the plate. Develop the chromatogram to about 15 cm from the starting point using a developing solvent containing 20 volumes glacial acetic acid, 40 volumes petroleum ether and 40 volumes toluene.

**4.1.3.2** Dry the plate in air. Spray the plate with an indicator solution, containing 20 percent (*m/v*) phosphomolybdic acid in ethanol until a yellow colouration persists.

**4.1.3.3** Examine in daylight. After a few min there is a progressive change to blue colouration. After 5 to 10 min expose the plate to ammonia vapours until the background is white.

4.1.3.4 Examine in daylight. The principal spot of the sample solution corresponds with that for octyl gallate in the control solutions. Suitable resolution of propyl and octyl gallate spots is determined from control solution B.'

4.2 The materials shall also conform to the requirements given in Table 1.

**Table 1 Requirements for Octyl Gallate, Food Grade**  
(Clause 4.2)

Sl. No.	Characteristic	Requirements	Method of Test, Ref to
(1)	(2)	(3)	(4)
i)	Purity as C <sub>15</sub> H <sub>22</sub> O <sub>5</sub> , percent by mass, <i>Min</i>	98.5	Annex A
ii)	Moisture, percent by mass, <i>Max</i>	0.5	Annex B
iii)	Sulphated Ash, percent by mass, <i>Max</i>	0.05	Annex C
iv)	Chlorinated organic compounds (as Chlorine), mg/kg, <i>Max</i>	100	Annex D
v)	Free acid (as gallic acid), percent by mass, <i>Max</i>	0.5	Annex E
vi)	Specific Absorption at 275 nm, <i>Min 1%</i> <i>Max 1 cm</i>	375 390	Annex F
vii)	Arsenic (as As), percent by mass, <i>Max</i>	3.0	IS 1699
ix)	Lead (as Pb), percent by mass, <i>Max</i>	2.0	IS 1699

## 5 PACKING, STORAGE AND MARKING

### 5.1 Packing

The material shall be securely packed in well-filled containers with minimum access to light and air. The containers shall be such as to preclude contamination of the contents with metals or other impurities.

### 5.2 Storage

The material shall be stored in a cool and dry place so as to avoid exposure to heat.

### 5.3 Marking

5.3.1 Each container shall be legibly and indelibly marked with the following information:

- Name of the material including the words 'Food Grade';
- Source of manufacture;
- Net content when packed;
- Batch or code number;
- Date of manufacture; and
- Expiry/ Best before date;
- Any other requirements as specified under the *Legal Metrology (Packaged Commodities) Rules, 2011* and *Food Safety and Standards (Labelling and Display) Regulations, 2020*.

### 5.3.2 BIS Certification Marking

**January 2025**

The product(s) conforming to the requirements of this standard may be certified as per the conformity assessment schemes under the provisions of the *Bureau of Indian Standards Act, 2016* and the Rules and Regulations framed thereunder, and the products may be marked with the Standard Mark.

## **6 SAMPLING**

The representative samples of the material shall be drawn according to the method prescribed in IS 1699.

## **7 TESTS**

**7.1** Tests shall be carried out by the methods specified in col (4) of Table 1.

### **7.2 Quality of Reagents**

Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in tests.

NOTE - 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

**ANNEX A**  
 [Table 1, S. No. (i)]  
**PURITY ANALYSIS OF OCTYL GALLATE**

**A-1 GENERAL**

Two methods, namely, spectrophotometric and bismuth nitrate, have been specified. Either could be used depending upon the facilities available.

**A-2 SPECTROPHOTOMETRIC METHOD**

**A-2.1** Dry a suitable quantity of sample in an oven at 90 °C for 6 h. Cool it in a desiccator. Prepare a solution of a dried sample in 80 percent ethanol containing 5 g/ml and determine the extinction at 218 and 276 nm.

$$\Sigma 1\%_{1\text{cm}}(218 \text{ nm}) 935-960;$$

$$\Sigma 1\%_{1\text{cm}}(276 \text{ nm}) 373$$

**A-2.1 Calculation**

$$\text{Octyl Gallate, percent by mass} = \frac{\Sigma 1\%_{1\text{cm}} \text{ of sample}}{\Sigma 1\%_{1\text{cm}} \text{ of pure standard solution}} \times 100$$

**A-2 BISMUTH NITRATE PRECIPITATION****A-2.1 Reagents****A-2.1.1 Acetone**

**A-2.1.2 Bismuth nitrate** – Dissolve 5 g of bismuth nitrate in 25 ml of distilled water and 25 ml of glacial acetic acid. Dilute the solution to 250 ml.

**A-2.1.3 Acetic acid** – 0.1 N.

**A-2.1.4 Nitric acid** – 0.05 N.

**A-2.2 Procedure**

Dry a suitable quantity of sample in an oven at 90 °C for 6 h. Cool it in a desiccator. Weigh 100 mg of octyl gallate into a 250 ml beaker. Add 15 ml of acetone and 10 ml of water and bring to the boiling point. Add 10 ml of bismuth nitrate and bring again to the boiling point. Cool to room temperature. Filter through a tared sintered glass crucible. Wash twice with acetic acid and twice with water. Wash six times with nitric acid at 80 °C pressing the precipitate down well between each wash. Wash twice with water. Dry at 110 °C to constant mass.

**A-3.3 Calculation**

$$\text{Octyl gallate, percent by mass} = \frac{\text{Mass of Precipitate} \times 0.5574}{\text{Mass of the sample}} \times 100$$

**ANNEX B**

[Table 1, S. No. (ii)]

**DETERMINATION OF MOISTURE**

**B-1 PROCEDURE**

Oven-drying and Karl Fischer methods have been specified. In case of dispute, Karl Fischer method shall be used.

**B-1.1 Oven Drying**

Weigh about 2 g of well-ground material in the tared dish. Place the dish containing the material in a ventilated oven at  $(90 \pm 1)$  °C for 6 h. Cool the dish in a desiccator and weigh. Calculate loss on drying percent by mass.

**B-1.2** Karl Fischer method as described in IS 2362 shall be used.

**ANNEX C**

[Table 1, S. No. (iii)]

**DETERMINATION OF SULPHATED ASH**

**C-1 REAGENT**

**C-1.1 Concentrated Sulphuric Acid**

**C-2 PROCEDURE**

Weigh about 2 g of the material in a tared crucible. Ignite gently until the material is thoroughly charred, cool, moisten the residue with 1 ml of sulphuric acid and ignite gently again till the carbon is completely consumed. Cool the crucible in a desiccator and weigh.

Note – Carry out the ignition in a place protected from air currents and use as low a temperature as possible to effect the combustion of carbon.

**C-3 CALCULATION**

$$\text{Sulphated ash, percent by mass} = \frac{M_1}{M_2} \times 100$$

where,

$M_1$  = mass, in g, of the residue; and

$M_2$  = mass, in g, of the material for the test.

**ANNEX D**

[Table 1, S. No. (iv)]

**DETERMINATION OF CHLORINATED ORGANIC COMPOUNDS**

**D-1 REAGENTS**

**D-1.1 Sodium Hydroxide** – 0.1 N.

**D-1.2 Nitric Acid**

**D-1.3 Calcium Carbonate**

**D-1.4 Silver Nitrate** – 0.1 N.

**D-1.5 Hydrochloric Acid** – 0.01 N.

**D-2 PROCEDURE**

Dissolve 1 g of the sample in 10 ml of 0.1 N sodium hydroxide. Acidify with nitric acid solution and filter off the precipitate. Mix the precipitate with 2 g of calcium carbonate, dry the mixture and then ignite. Take up the ignition residue in 20 ml of dilute nitric acid and filter. Mix the solution with 0.5 ml of 0.1 N silver nitrate. The turbidity should not be more than that obtained in 20 ml of dilute nitric acid by addition of 0.5 ml of 0.1 N silver nitrate and 0.3 ml of 0.01 N hydrochloric acid.

**ANNEX E**

[Table 1, S. No. (v)]

**DETERMINATION OF FREE ACIDS (AS GALLIC ACID)**

**E-1 REAGENTS**

**E-1.1 Acetone**

**E-1.2 Hydrochloric Acid** – 0.005 N.

**E-1.3 Buffer** – pH 5.

**E-1.4 Bromocresol Green** – Dissolve 50 mg of bromocresol green in 100 ml of alcohol and filter if necessary.

**E-1.5 Sodium Hydroxide** – 0.05 N.

**E-2 PROCEDURE**

To a mixture of 50 ml of carbon dioxide-free water and 50 ml of acetone, add 5 drops of bromocresol green and titrate with 0.005 N hydrochloric acid to match a buffer containing the same amount of indicator. Dissolve 0.4 g of the sample in 50 ml of acetone and add 50 ml of carbon dioxide-free water, 5 drops of bromocresol green and the amount of 0.005 N hydrochloric acid found in the preliminary test to bring the solvent to pH 5. Titrate the solution back to pH 5 with 0.05 N sodium hydroxide, matching against the buffer. Each ml of 0.05 N sodium hydroxide is equivalent to 8.506 mg of gallic acid.



**ANNEX F**

[*Table 1, S. No. (vi)*]

**DETERMINATION OF SPECIFIC ABSORPTION**

Prepare one percent solution of the sample in ethanol and find out its specific absorption in a suitable spectrophotometer using 1 cm cell at 275 nm.