

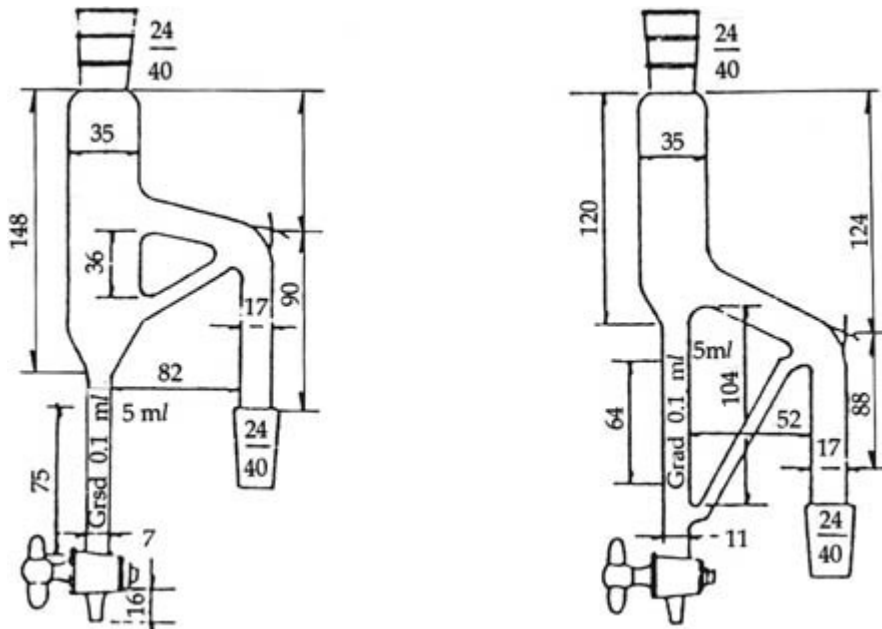
Standard and Specification > Natural Additives > Oleoresin Paprika

Oleoresin Paprika			
Definition	Paprika Extract colorant is a carotenoid colorant that is obtained by extracting fruits of paprika (<i>Capsicum annum L.</i>) of solanaceae with organic solvents (extracting solvent for spices such as oleoresin). Its major colorant component is capsanthin (C ₄₀ H ₅₆ O ₃ = 587). Dilutant, antioxidant, or other food additives (emulsifier, thickening agent, etc).		
[Compositional Specifications of Oleoresin Paprika]			
Content	Color Value (ASTA) of Paprika Extract colorant should be higher than the indicated value.		
Description	Paprika Extract colorant is orange ~ dark brown liquid, paste, or powder with a slight characteristic odor.		
Identification	<p>(1) Test Solution obtained in Color Value section shows a maximum absorption band near 453 nm or 470 nm.</p> <p>(2) When 2 ml of sulfuric acid is added to 0.5 g of Paprika Extract colorant, the color of the liquid changes from orange to blue.</p> <p>(3) When antimony trichloride solution is added to Paprika Extract colorant, it shows a blue.</p>		
	<p>(1) Arsenic : 0.25 g of Paprika Extract colorant is placed in a platinum, quartz, or porcelain crucible. 10 ml of magnesium nitrate in ethyl alcohol (1→50) is added to the crucible and then alcohol is ignited. It is then reduced to ash by heating at 450~550°. If carbonaceous substance persists, it is wetted with minute amount of nitric acid, which is further heat treated at 450~550°. After cooling, 3 ml of hydrochloric acid is added to the residue, which is then dissolved by heating in a water bath. When test for arsenic is carried out with this test solution, it should not be more than 4ppm.</p> <p>(2) Heavy Metals : 0.5 g of Paprika Extract colorant are carbonized by heating mildly in a quartz or porcelain crucible. After cooling, add 2 ml of nitric acid and 5 drops of sulfuric acid, it is heated until white smoke disappears, which is then reduced to ash by further heating at 450~550°. After cooling, 2 ml of hydrochloric acid is added, which is then evaporated to dryness in a water bath. 3 drops of hydrochloric acid and 10 ml of hot water are added to the resulting residue, which is then heated for 2 minutes. After cooling, 1 drop of phenolphthalein indicator solution is added, then ammonia solution is added until the color of the solution becomes pale red. The resulting solution is transferred into a Nestler cylinder by rinsing with water. 50 ml of test solution is prepared by adding 2 ml of diluted acetic acid (1→20) and water. When this solution tested for heavy metals, the content should not be more than 40ppm. Color standard solution is prepared by the following procedure. 2 ml of nitric acid, 5 drops of sulfuric acid, and 2 ml of hydrochloric acid are added and evaporated to dryness in a crucible that is made of the same material used for test solution preparation. 3 drops of hydrochloric acid are added to the residue, which is then transferred into another Nestler cylinder as described above. Finally, 2 ml of lead standard solution, 2 ml of diluted acetic acid (1→20), and water are added to bring the total volume to 50 ml.</p> <p>(3) Lead : 0.8 g of Paprika Extract colorant (if it is liquid, it is concentrated by evaporation in a water bath) is slowly carbonized by heating, which is reduced ash by further heat treatment at a temperature below 500°. Carefully 20 ml of dilute nitric acid is added to the ash, which is then gently boiled for 5 minutes. It is then filtered (if necessary), the residue is washed with water, which is then added to the filtrate. Water is added so that total volume of this solution becomes 50 ml. This test solution is tested for lead. The detected amount of lead should not be more than 10ppm.</p> <p>(4) Residual Solvents : When Paprika Extract colorant is tested by the following test method, it should contain,</p> <table border="0" style="width: 100%; margin-top: 10px;"> <tr> <td style="width: 50%;">Methylene chloride, trichloro ethylene</td> <td style="width: 50%;">Not more than 30ppm</td> </tr> </table>	Methylene chloride, trichloro ethylene	Not more than 30ppm
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	(individual or sum if used together)
Acetone	Not more than 30ppm
Isopropyl alcohol	Not more than 50ppm
Methyl alcohol	Not more than 50ppm
Hexane	Not more than 25ppm

[Test Method]

① Distilling head : A clevenger trap, that is designed to used for oils (heavier than water), is



(a) For oils heavier than water (b) For oils lighter than water

Distilling head : Clevenger Traps (Units : mm)

② Agents

□ Toluene : The purity of toluene for this test should be such that it does not contain any solvents that are measured in this test as determined by gas chromatography using the following column or its equivalent.

- A) 35~80 mesh Chromosorb W Ucon 75 H 90,000
- B) 35~80 mesh Chromosorb W Ucon LB 135 20%
- C) 60~80 mesh Chromosorb W Ucon LB 1715 15%
- D) Porapak Q 50~60 mesh

Test proceed as directed under Test Procedure. The same amount of toluene is injected as the amount used in solvent analysis. If any peaks for interfering impurities appear before the toluene peak, they should be removed by separation.

- Benzene : It should not contain any interfering impurities. Its purity is measured by the same method as toluene.
- Detergent & antifoam : It should not contain flammable matter. If it does, an aqueous solution of the material should be heated until the flammables are removed.
- Reference Solution A : A solution containing 2,500ppm of benzene in toluene is prepared. If the toluene contains benzene as the only impurity, the concentration should be adjusted so that it contains 2,500ppm of benzene as determined by gas chromatography.
- Reference Solution B : A solution containing 0.63% v/w of acetone in water is prepared.

③ Preparation of Test Solution

□ Test Solution A (for solvents except methyl alcohol) : Small amount of detergent & antifoam, 50 ml of water, 10 g of anhydrous sodium sulfate, 1 ml of Reference Solution A, and 50 g of sample are added in a 250 ml single neck round bottom flask with a 24/40 ground joint. 400 mm reflux condenser, distilling head, and a collector is connected to the flask and 15 ml of distillate is collected. 15 g of anhydrous potassium carbonate is added to the distillate. It is then cooled while shaking. It is set aside until phases are separated. The toluene phase contains all the solvents except methyl alcohol and is used in the following Test Procedure. The aqueous phase is used as Test Solution B.

□ Test Solution B (for methyl alcohol only) : 50 ml of Test Solution B (aqueous phase obtained in Test Solution A) is transferred into a round bottom flask, where 2~3 glass balls and 1 ml of Reference Solution B are added. Approximately 1 ml of distillate is collected. This distillate contains acetone and methyl alcohol in the sample. It is used in the following Test Procedure.

④ Test Procedure : Gas chromatography equipped with a thermally conductive detector and sample injection port is used. Under typical operation conditions, a 0.3 mm × 2 m column (isothermalized at 70~80°) is used. Flow rate of a carrier gas is 50~80 ml/minute and injected amount of sample is 15~20 µl. The choice of a column depends on the components to be analyzed and is up to the tester to a certain degree. The columns described in A), B), C), and D) in Toluene section are used as follows.

A) This column separates acetone and methyl alcohol from the aqueous phase. This also can be used to analyze or separate hexane, acetone, and trichloro ethylene in the toluene phase obtained in Test Solution A. The order of effluence is acetone, methyl alcohol and water or hexane, acetone, isopropyl alcohol+methylene chloride, benzene, trichloro ethylene, and dichloro ethylene+toluene.

B) This column separates methylene chloride, isopropyl alcohol, and dichloro ethylene. The order of effluence is hexane+acetone, methylene chloride, isopropyl alcohol, benzene, dichloro ethylene, trichloro ethylene, toluene.

C) This is the most generally used column except for methyl alcohol. The order of effluence is hexane, acetone, methylene chloride, isopropyl alcohol, benzene, and dichloro ethylene+trichloro ethylene, and toluene.

D) This column is used to measure methyl alcohol, which appears right after a huge peak of water.

⑤ Preparation of Calibration Curve : A mixture of a solvent and toluene with previously known benzene content is injected into gas chromatography. The response of the detector for the known ratios of solvents is measured. The peak sizes of benzene in toluene and the solvents should be the same as those of the sample. Peaks areas for the solvents are calculated according to benzene and the weighing factor F is calculated as follows.

$$F(\text{solvent}) = \frac{\text{Wt\% solvent}}{\text{wt\% benzene}} \times \frac{\text{peak area of benzene}}{\text{peak area of solvent}}$$

When it is compared in terms of benzene recovery rate, the recovery rates of various solvents from oleoresin sample are as follows.

hexane 52%, acetone 85%, isopropyl alcohol 100%, methylene chloride 87.5%, trichloro ethylene 113%, dichloro ethylene 102%, methyl alcohol 87%

⑥ Calculation : The concentration of residual solvents (except for methyl alcohol) is calculated by the following equation.

$$\text{Residual Solvent} = \frac{43.4 \times F(\text{solvent}) \times 100}{\text{Solvent recovery rate(\%)}} \times \frac{\text{peak area of solvent}}{\text{peak area of benzene}}$$

43.4 is the concentration of internal standard benzene (inppm) related to 50 g of oleoresin sample used in the test.

$$100 \times F(\text{methylalcohol}) \times \frac{\text{peak area of methylalcohol}}{\text{peak area of benzene}}$$

	<p>Methyl alcohol = $\frac{\text{peak area of acetone}}{0.87} \times \text{100}$</p> <p>100 is the concentration of internal standard acetone (inppm) related to 50 g of oleoresin sample used in the test.</p> <p>(5) Spicy Taste : 400 mg of oleoresin paprika is weighed into a 100 ml volumetric flask, which is filled with alcohol. It is then mixed by shaking and settled. 60 ml of 10% sugar solution in water is added to 0.15 ml of the supernatant. When 5 people consume 5 ml each of the resulting solution, should not be more than 3 people who feel the spicy taste.</p>
<p>Assay(Color Value)</p>	<p>Appropriate amount of oleoresin paprika is precisely weighed into a 100 ml volumetric flask the absorbance to be measured will be within a range of 0.2 to 0.7. and dissolved in acetone to make total volume 100 ml. (If it is water soluble, water is used instead of acetone). After settling for 2 minutes, 1 ml of this solution is diluted to 100 ml with acetone (For water soluble sample, diluting with acetone may cause severe turbidity. In this case, 1 ml of alkaline lead acetate solution (1→50) is added to the solution and then diluted to 100 ml with acetone. It is then centrifuged and the supernatant is used.). Absorption of the resulting solution (As) is measured at 460 nm with 1 cm path length using acetone as a reference. Using the same method, absorption of NBS (National Bureau of Standard) color standard glass plate 2030 (AF) is measured.</p> $\text{Color Value (ASTA)} = \frac{\text{As} \times 164 \times \text{F}}{\text{W}} \times 10$ <p>W : Weight of sample(g) ASTA : American Spice Trade Association F : AN/AF, where AN is absorption of glass filter defined by NBS. Therefore, F is an activity for correcting the instrument. * If colorimetric glass plate is not available, a color standard solution is used.</p> <p>□ Color standard Solution : $\text{CoSO}_4(\text{NH}_4)_2\text{SO}_4 \cdot 6\text{H}_2\text{O}$ is dried for 1 week in a desiccator with dried silica gel. 0.3005 g of $\text{K}_2\text{Cr}_2\text{O}_7$ and 34.960 g of dried $\text{CoSO}_4(\text{NH}_4)_2\text{SO}_4$ are dissolved in 1.8 M sulfuric acid to make 1,000 ml. The absorption of this solution at 460 nm with 1 cm path length is 0.600.</p>
<p>Permitted Use Level of Paprika Extract Pigments and Ingredients Containing it</p>	<p>Paprika Extract Pigments should not be used for the food items below.</p> <ol style="list-style-type: none"> 1. Natural food [Natural food(meat, seafood (whale meat included), vegetables, fruits, marine algae, bean, and their simply processed food (peeled or cut)] 2. Tea 3. Red pepper powder or shredded red pepper 4. Kimchi 5. Fermented hot pepper soybean paste 6. Vinegar