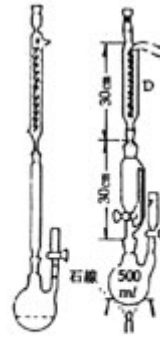


Standard and Specification > Natural Additives > Caramel Color

Caramel Color	
Definition	<p>There are caramel I, II, III, and IV. Caramel I (Plain Caramel, Caustic Caramel) is obtained by heat treating edible carbohydrates of sugars, hydrolyzed starch, and molasses. Or it can be obtained by treating with acids or alkalis (free of ammonium compounds and sulfites) followed by heat treatment. Caramel II (Caustic Sulfite Caramel) is obtained by treating edible carbohydrates of sugars, hydrolyzed starch, and molasses with sulfites (ammonium compound free) followed by heat treatment. Or it can be obtained by treating with sulfites, acids, or alkalis (free of ammonium compounds) followed by heat treatment. Caramel III (Ammonia Caramel) is obtained by treating edible carbohydrates of sugars, hydrolyzed starch, and molasses with small amount of ammonium compounds, followed by heat treatment. It can be treated with acids or alkalis (free of sulfites) prior to heat treatment. Caramel IV (Sulfite Ammonia Caramel) is obtained by treating edible carbohydrates of sugars, hydrolyzed starch, and molasses with small amount of ammonium compounds and sulfites, followed by heat treatment. It can be treated with acids or alkalis prior to heat treatment.</p>
[Compositional Specifications of Caramel Color]	
Description	Caramel Color is black ~ bluish brown liquid, solid or powder with characteristic carbonizing sugar scent and refreshing bitter taste.
Identification	<p>(1) Arsenic : 0.77 g of Caramel Color is tested according to Purity (1) for [Guar Gum] (Not more than 1.3ppm).</p> <p>(2) Heavy Metals : 1 g of Caramel Color is tested according to Purity (2) for [Guar Gum] (Not more than 25ppm). However, 2.5 ml of lead standard solution is to be used.</p> <p>(3) Lead : 5 g of Caramel Color is tested according to Purity (3) for [Locust Bean Gum] (Not more than 2ppm).</p> <p>(4) Nitrogen as Ammonia : 25 ml of 0.1 N sulfuric acid is added to a 500 ml Erlenmeyer flask. Kjeldahl distillation apparatus is set up so that the end of the condenser is immersed right beneath the surface of the sulfuric acid solution. Separately, approximately 2 g of Caramel Color is precisely weighed into a 800 ml Kjeldahl flask for decomposition, where 2 g of magnesium oxide, 200 ml of water, and boiling stones. The content is dissolved by shaking and the apparatus is immediately connected. It is then boiled and 100 ml of distillate is collected. The end of the condenser is washed with water, which is added to the distillate. 4 ~ 5 drops of methyl red solution are added to the distillate, which is then titrated with (consumed amount is S). Separately, a blank test is carried out and consumed amount of 0.1 N sodium hydroxide solution is B. Nitrogen content as ammonia is calculated by the following equation and it should not be more than 0.6%. (for a product with color value of 0.1).</p> $\text{Content of Nitrogen as ammonia (\%)} = \frac{(B - S) \times 0.0014 \times 100}{\text{Weight of sample(g)}}$ <p>(5) Mercury : 5 g of Caramel Color is taken into a flask for mercury decomposition, where 10 ml of water and 20 ml of nitric acid are added and then set aside. 10 ml of sulfuric acid is slowly added. A reflux condenser is attached to the flask, which is then heated carefully until generation of NO₂ gas stops. If the decomposed liquid does not turn pale orange in color, it is cooled and 5 ml of nitric acid is added. It is then heated again. If necessary, this procedure is repeated until the decomposed liquid becomes transparent. After cooling, 50 ml of water and 10 ml of 10% urea solution are added to the decomposed liquid, which is boiled for 10 minutes. After cooling, 1 g of potassium permanganate is added and mixed for 10 minutes while shaking occasionally. If purple red color disappears, small amount of potassium permanganate is added to the solution, which is heated for 20 minutes. This is repeated until the purple red color persists. After cooling, 10% of hydrogen peroxide solution is drop-wise added until</p>

the solution becomes colorless and transparent (Test Solution). The Test Solution is tested by reductive evaporation of atomic absorption spectrophotometry. The absorption of the Test Solution should not be higher than that of 5 ml of mercury standard solution (Not more than 0.1ppm).



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(6) 4-Methyl Imidazole : Preparation of Test Solution : A 3:2 mixture (W/V) of diatomaceous earth (Celite 545 or its equivalent) for chromatography and 2 N sodium hydroxide solution is prepared. 5 g of this mixture is filled and tapped into a 2525 × 250 mm chromatography column. 10 g of Caramel Color is added and mixed into a beaker with 6 g of 20% sodium carbonate solution. 15 g of diatomaceous earth for chromatography is well mixed the solution. This is transferred into the column and tapped. The top of the column is plugged with glass wool. A mixture of chloroform : alcohol (80 : 20) is passed through the column at a rate of 5 ml per minute. 125 ml of filtrate is collected into a 250 ml separatory funnel with a stopcock. The filtrate is extracted with 25 ml of 0.05 N sulfuric acid. It is again extracted twice with 10 ml each of 0.05 N sulfuric acid (if ammonia content is high, more extracting solvent is needed). This acidic solution is concentrated to 5 ~ 6 ml using a vacuum rotary evaporator. The temperature of water bath should not exceed 55°. Care must be taken so that the volume of the concentrated does not become more than 3 ml. This concentrate is transferred into a 10 ml volumetric flask. The evaporation flask is washed with small amount of water, which is added to the volumetric flask so that the total volume becomes 10 ml. It is well shaken with a stopper. It is then transferred into a vial with a Teflon cap. Sodium carbonate is added until carbon dioxide gas doesn't evolve and the pH is adjusted to 9.0 or higher (Test Solution). The Test Solution is tested by gas chromatography in General Test Methods under the operation conditions below. The content of 4-Methyl Imidazole should not be more than 0.025% (for a product with color value of 0.1).

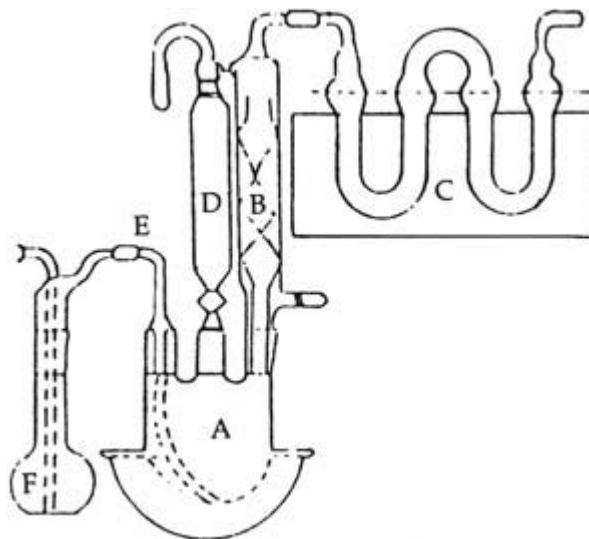
[Operation Conditions]

- Column : A stainless steel tube 1.23 m × 3 mm(inner diameter). Prior to use, 5% of alcoholic solution of potassium hydroxide is passed through the column which is then dried.
- Column Filler : Anakrom AB, or its equivalent (80 ~ 90 mesh) coated with Carbowax 20 M (5%). Column filler is alkalized by treating with 2% alcoholic solution of sodium hydroxide.
- Detector : (Hydrogen) Flame Ionization Detector (FID)
- Temperature at injection hole : 180°
- Column Temperature : 200°
- Detector Temperature : 250°
- Carrier gas and flow rate : Helium, 75ml/min

□ Preparation of Standard Solution : Reagent grade 4-methyl imidazole (boiling point 92~93°, 0.05 mmHg) is redistilled, 50 mg of which is placed in a 50 ml volumetric flask and diluted to 50 ml with 0.1 N sulfuric acid (Undiluted Standard Solution). It is stored in a refrigerator. 1.0, 1.5, 2.0, 2.5, 3.0, 3.5, 4.0, and 5.0 ml each of undiluted standard solution is placed in 10 ml volumetric flask. Sodium carbonate is added to each flask until carbon dioxide does not evolve. Each flask is filled to 10 ml with 1% sodium

m carbonate (Standard Solution). Each standard solution contains 100, 150, 200, 250, 300, 350, 400, and 500 µg of 4-methyl imidazole per 1 ml. It is stored in a refrigerator.

(7) Sulfur Dioxide : Apparatus : The following apparatus is used.



- A : 1,000 ml three neck round bottom flask
- B : 30 cm Allihn reflux condenser
- C : absorption tube
- D : 125 ml separatory funnel with a stopcock
- E : introduction tube
- F : 250 ml gas wash bottle connected to a nitrogen cylinder

Purity

4.5 g of pyrogallol and 5 ml of water are ground in a small mortar. The supernatant is transferred into a wash bottle. The remaining solid is ground again and washed with water, which is then transferred into the wash bottle. It is then flushed with nitrogen to replace air. Using a long stem funnel, a cooled solution of 65 g potassium hydroxide in 85 ml of water. The wash bottle is again flushed with nitrogen. It is connected to the entrance of a distillation flask. Gas washing solution is prepared prior to use. Two glass rods (8 mm diameter 25 mm length) are placed in the absorption tube. 10 ml of glass balls, 10 ml of 3% hydrogen peroxide solution, and 1 drop of methyl red solution are added to the exit side. After the apparatus is connected, the stopcock of the separatory funnel is closed. A small amount of gas is passed through to the apparatus, which is then set aside and checked for gas leak by observing the liquid level.

Test Procedure : 25 g of Caramel color is dissolved in 300 ml of water (freshly boiled and cooled) in a distillation flask. Water is added to bring the total volume to approximately 400 ml. 90 ml of 4 N hydrochloric acid is slowly added to the distillation flask using a separatory funnel with a stopcock. In a flowing nitrogen atmosphere, the solution is brought to a stable reflux and maintained for 2 hours. Then cooling water for the condenser is stopped. When vapor gets condensed in the first absorption tube and the tube gets warm, heating is stopped. When the top connection part of the condenser is cooled, it is disconnected. The first absorption tube is separated from the second one. The solution in the first absorption tube is titrated with 0.1 N sodium hydroxide solution. The connection tube is connected to the exit part of the second absorption tube. The solution is titrated again. The consumed amount of is S. Separately, a blank test is carried out and the amount of consumed 0.1 N sodium hydroxide solution is B. When the content of sulfur dioxide is calculated by the following equation, it should not be more than 0.2% (Based on product with color value of 0.1).

$$\text{Content of Sulfur Dioxide(\%)} = \frac{(S - B) \times 0.0032 \times 100}{\text{---}}$$

Weight of sample(g)

(8) Color Value : 100 mg of Caramel color is precisely weighed and dissolved in water to make 100 ml. If necessary, the solution is centrifuged and the supernatant is used. Absorption A of this solution is measured with 1 cm of liquid layer path length at 610 nm, using water as a reference. Color value is obtained from the following equation (converted into a solid matter). The contents should be 0.1 ~ 0.6.

$$\text{Color Value} = \frac{A_{610} \times 100}{\text{Content of solid matter(\%)}}$$

Provided, the content of solid matter is obtained as follows. 30 g of quartzsand and glass rod are placed in a weighing bottle, which is dried at 60° and under reduced pressure of 50 mm/Hg until the weight becomes constant. 1.5 ~ 2.0 g of the sample is precisely weighed into the weighing bottle. Solid matter content is calculated by the following equation. Quartz sand (purified quartzsand, particle size : No.40 ~ No.60 mesh) is decomposed by hydrochloric acid and washed with water until it doesn't show acidity. It is then dried and heat treated before use.

$$\frac{(\text{weight of quartzsand and sample after drying(g)} - \text{weight of quartzsand}) \times 100}{\text{Weight of sample(g)}}$$

(9) Total Nitrogen : When Caramel color is tested by Kjeldahl Method in Nitrogen Determination, the amount should not be more than 3.3% (as based on product with color value of 0.1).

※ Calculation of impurities based on color value of 0.1

Using the content of each impurity (nitrogen as ammonia, 4-methyl imidazole, sulfur dioxide, total nitrogen), Cs (conversion to solid matter) is obtained. On the basis of color value 0.1, the content of each impurity is calculated by the following equation.

$$\text{Cs} = \frac{\text{Cs} \times 0.1}{\text{Color Value}} \times 100$$
$$\text{Cs} : \frac{\text{Content Impurities} \times 100}{\text{Content of solid matter(\%)}}$$

Permitted Use Level of Caramel Color

Should not be used for the food items listed below.

1. Natural food [Natural food[meat, seafood (whale meat included), vegetables, fruits, marine algae, bean, and their simply processed food (peeled or cut)]
2. Hot pepper powder, red pepper powder or shredded red pepper
3. Kimchi
4. Fermented hot pepper soybean paste
5. Concentrated ginseng and ginseng tea
6. Concentrated red ginseng and red ginseng tea