



PRODUCT MANUAL FOR CARAMEL ACCORDING TO IS 4467 : 1996

This Product Manual shall be used as reference material by all Regional/Branch Offices & licensees to ensure coherence of practice and transparency in operation of certification under Scheme-I of Bureau of Indian Standards (Conformity Assessment) Regulations, 2018 for various products. The document may also be used by prospective applicants desirous of obtaining BIS certification licence/certificate.

1.	Product	:	CARAMEL
	Title	:	IS 4467 : 1996
	No. of Amendments	:	02
2	Sampling Guidelines	:	
a	Raw Material	:	Raw materials shall be as per Clause 5 of IS 4467. Declaration regarding compliance to clause 5 from the manufacturer may be accepted.
b	Grouping Guidelines	:	Sample may be tested for each Type to cover respective type.
c	Sample Size	:	500 gm
3	List of Test Equipments	:	Please refer-ANNEX-A
4	Scheme of Inspection and Testing	:	Please refer-ANNEX-B
5	Possible Test in a Day :		
	i. Solid content ii. Colour intensity iii. Ammoniacal nitrogen iv. 4-Methylimidazole v. Lead vi. Arsenic		
6	Scope of the Licence:		
	“Licence is granted to use Standard mark as per IS 4467 : 1996 with the following scope:		
	Name of the product	:	CARAMEL

ANNEX A

TO PRODUCT MANUAL FOR CARAMEL ACCORDING TO IS 4467 : 1996

LIST OF TEST EQUIPMENTS

Major test equipment required to test as per the Indian Standard

Sl No.	Tests used in with Clause Reference	Test Equipment
1.	Solid Content 6.1.2 & Table 1 (Annex-A of IS 4467)	<u>METHOD -1(Specific gravity method)</u>
		Pycnometer-100ml volumetric flask, neck of 8mm internal dia
		Chromic acid
		Distilled water
		Constant Temperature bath 20° C
		Alcohol
		Weighing balance
		Caramel
		Glass rod
		Ether
		Clean cloth
		<u>METHOD 2</u>
		Pure quartz sand
		Whatman no.40 sieve
		Oven
		Beaker
		Hydrochloric acid
		Measuring jar
		Caramel
		Weighing balance
		Temperature 60C & Pressure 50mm/Hg (Chamber)
2.	Colour Intensity 6.1.2 & Table 1 Annex B of IS 4467	<u>EBC METHOD B-1</u>
		Color comparator-Lovibond-Tintometer
		Color slides-EBC glass slides
		Glass cell-25mm cell
		Artificial North Daylight (commission international de l'Eclairage standard B (equivalent black body radiation 4800K) intensity 100-1000 lux)
		Distilled water
		Centrifuge
		Caramel solution

		Sodium Carbonate/Sodium Hydroxide
		Cotton wool
		Measuring jar
		Weighing balance
		<u>METHOD-2 B-2</u>
		Absorbance method
		Spectrophotometer (monochromator band width 12nm)
		Beckman DU, Beckman BD-G, Gausch and lamb spec
		Caramel
		Centrifuge
		Distilled water
		Measuring jar
		Weighing balance
3.	Ammoniacal Nitrogen 6.1.2 & Table 1 (Annex C of IS 4467)	Distillation Apparatus-500ml receiving flask
		Kjeldahl connecting bulb and a condenser
		Sulphuric acid
		Magnesium oxide-carbonate free
		Methyl red indicator-Methyl red, Ethanol
		Sodium hydroxide
		Caramel
		Weighing balance
		Boiling chips
		Measuring jar
4.	Methylimidazole 6.1.2 & Table 1 (Annex D of IS 4467)	Beaker
		<u>Method-1 D-1</u>
		Chromatography Column with stop cock 250mm× 250mm
		Chromatography Chamber-250mm ×250mm× 250 mm
		Whatman No.1 Chromatography paper 225mm× 270mm
		Vacuum flask-100ml
		Const. Temperature water bath
		Glass wool
		Micropipette-10µl
		Spraying bottles
		Air drier
		Separating funnel-250ml
		Spectrophotometer/Spectrocolorimeter
		pH meter
		Celite 545
		Sodium hydroxide

	Elution Solvent-Chloroform, Ethanol/Methanol(80:20)
	Sulphuric acid solution
	Developing solvent
	System1-Diethyl ether, Chloroform, Methy Alcohol and NH ₄ OH (80:20:20:4)
	System 2-n-Propyl alcohol, Ammonia Solution
	Spraying reagent No.1
	Reagent (a)-NaNO ₂ cool to 0°C
	Reagent (b)-Sulfanilic acid, Conc.HCl, 100ml flask
	Spraying Reagent No.2-Sodium Carbonate Solution
	Stock solution of 4-Mel
	4-Methylimidazole, H ₂ SO ₄
	Stand solution of 4-Mel
	Pipette, 1ml of 4-Mel stock solution, Solid Sodium Carbonate, 10ml-distilled flask
	Diazo Sulfanilic acid
	Sodium carbonate
	pH test paper
	Distilled water
	Refrigerator
	Vaccum flask
	Volumetric flask-1000ml
	Temperature-5°C
	Method-2 D-2
	Polypropylene Beaker-150ml(Nalge 1201)
	Pyrex glass wool(22mm 300mm) chromatography column with PTFE stopcock (Kimax 17800)
	Round bottom flask-250ml(Pyrex 4320)
	Powder funnel-75mm
	Spatula
	Rotary Vaccum Evaporator (45-50kPa)
	Hot plate
	Pan for water bath
	Disposable Pasteur pipette
	Volumetric flask-50ml
	Reagents should be ACS grade
	Acetone
	Celite545
	Methylene chloride

		Sodium hydroxide
		Caramel
		Weighing balance
		Tetrahydrofuran
		Aliquot
		Gas chromatography equipped with Hydrogen flame detector
		Carbowax 20M+2% KOH
		Measuring jar
		Round bottom flask-250ml
		Water bath (35 C)
		2-Methylimidazole
		90/100 MESH Anakrom ABS
		GLC Carrier,Nitrogen,50ml/min Hydrogen,50ml/min Oxygen 80ml/min Injection part 200° C Column isothermal 180° C Detector 250° C, Sample size 5µl
5.	Lead 6.1.2 &Table 1 (Cl. 15 of IS 1699)	<u>Chemical method 15.2</u>
		Digestory funnel
		Separatory funnel
		Nitric acid
		Sulphuric acid –sp.gr 1.84
		Measuring jar
		Weighing balance
		Ammonium acetate –citrate solution
		Ammonium acetate
		Ammonium citrate
		Distilled water
		Concentrated Ammonia
		Thymol blue paper
		Dithizone
		Chloroform
		Ammonia solution
		Mineral acid
		Aqueous Ammoniacal Solution
		Carbon tetrachloride

		Potassium cyanide
		Hydroxyl amine hydrochloride
		Buffer pH-2(HCl, KCl & water)
		Volumetric flask -200ml,1000ml
		Dish
		Calcium sulphate
		Ammonium hydroxide
		Burner
		<u>Atomic Absorption method 15.1.6</u>
		Metal free distilled water(Column of cation exchange resin, Amberlite IR120CH)
		Nitric acid –sp.gr 1.42
		Perchloric acid
		Sulphuric acid
		Hydrochloric acid-sp.gr 1.16 to 1.18
		Sodium sulphate
		Standard lead solution(Lead nitrate, Nitric acid, distilled water, burner, volumetric flask-1000ml)
		Air/acetylene gases
		Flame atomic absorption spectrophotometer (180 to 600nm)
		Volumetric flask-100ml
		Pipette
		Kjeldahl flask-100-150ml
6.	Arsenic 6.1.2 & Table 1 (Cl. 15.2.2 of IS 1699)	Chemical method 15.2.2.1
		Mayercon-Bereret method
		Weighing balance
		Measuring jar
		Distillation apparatus
		Conical flask
		Sulphuric acidsp.gr 1.84
		Potassium permanganate
		Ferrous sulphate
		HCl acid
		Potassium bromide
		Aluminium strips-8mm× 8mm ×1mm

		Tin chloride solution(Tin &Hydrochloric acid)
		Test paper-Saturated ethanolic solution of mercuric bromide
		Nitric acid
		Dish
		Lime
		Micro burner
		Conical flask-25ml
		Rubber stopper
		Cotton gauze socked in 5%lead acetate solution
		Modified Gutzeit method
		5.1
		2088:1983
		Distilled water
		Lead acetate solution(Acetic acid IS 695:1975)
		Mercuric bromide solution(Mercuric bromide &Rectified spirit IS 323:1959)
		Lead acetate paper strips(Filter paper strips of 70× 50mm size, lead acetate solution, glass bottle)
		Mercuric bromide strips(filter paper 70× 50 mm, Mercuric bromide solution, amber-coloured glass bottle)
		Absorbent Cotton wool impregnated with lead acetate
		Rubber stopper& tubes
		Stannous chloride solution (stannous chloride ,volumetric flask-100ml,Hydrochloric acid)
		Dil. Sulphuric acid
		Potassium iodide solution
		Zinc granules(0.5 to1.0mm)
		Tweezers
		<u>Atomic Absorption hydride technique</u>
		15.1.7
		1699:1995
		Metal free distilled water(Amberlite IR 120CH)
		Sulphuric acid
		Con .Hydrochloric acid
		Standard Arsenic Solution(Arsenious oxide, Sodium hydroxide,beaker-100ml,Phenolphthalein indicator, sulphuric acid, sodium carbonate, volumetric flask-1000ml)
		Weighing balance

		Measuring jar
		Volumetric flask-100ml,1000ml
		Sodium hydroxide pellets
		Atomizer
		Argon
		Generation vessel
		Rubber balloons
		Hollow cathode/electrodeless discharge lamp
		Burette
		Atomic absorption photometer(180to 300nm)
		Potentiometric recorder (1-20m V)
		Stopper
		Flame
7.	Total Sulphur 6.2.1 & Table 2 (Annex E of IS 4467)	Electric furnace
		Magnesium oxide/Magnesium nitrate
		Sucrose
		Nitric acid
		Caramel
		Casserole
		Steam bath
		Temperature(525C)
		HCl acid
		Filter paper
		Funnel
		Barium chloride
		Leco-Combustion/Titration
		Cold electric muffle
		Measuring jar
		Weighing balance
8.	Sulphur Dioxide 6.2.1 & Table 2 (Annex F of IS 4467)	Hydrogen peroxide
		Barium Chloride
		Hydrochloric acid
		Conc. Hydrochloric acid sp gr 1.18
		Carbon dioxide gas cylinders

		Bromophenol Blue indicator (Bromophenol & Sodium hydroxide)
		U –tubes(Wohler tube)
		Funnel
		Joint with clamps
		Sodium hydroxide
		Weighing balance
		Rubber tube
		Ball joint
		Conical flask-250ml
		Capillary tube
		Reflux condenser
		Round bottom flask
		Separating Funnel
		Pressure gauge 30KPa
		Burner
		Saturated solution of Na ₂ CO ₃
		Weighing balance
		Measuring jar
		Distilled water
		Volumetric flask-500ml
9.	Total Nitrogen 6.2.1 & Table 2 (Annex-G of IS 4467)	Semi micro Kjeldhal apparatus
		Potassium sulphate
		Cupric sulphate
		Distilled water
		Sulphuric acid
		Hydrogen peroxide
		Burner/electric heater
		Sodium hydroxide
		Steam distillation apparatus
		Boric acid
		Methyl red/methylene blue
		Condensing tube
		Absorption flask
10.	2-Acetyl-4-Tetrahydroxy Butylimidazole (Thi) 6.2.1 & Table 2 (Annex H of IS 4467)	2,4-dinitrophenyl hydrazine hydrochloride reagent

		Hydrochloric acid
		Erlenmeyer flask-100ml
		Ethanol
		Steam bath
		Desiccator
		Dimethoxymethane
		2,4-dinitrophenyl hydrazine Hydrochloride
		Funnel
		Methanol
		Lichrosorb RP-8 HPLC-385nm
		Cation -exchange resin (strong) Dowex-50 AG×8 H ⁺ ,100-200 mesh
		Cation-exchange resin(weak) Amberlite CG AGI,H ⁺ (100-200)
		Methanol Carbonyl- free Girard P reagent & Methanol
		Dimethoxyethane-2,4-dinitrophenyl hydrazine
		Sodium hydroxide
		Neutral Aluminium
		Filter paper
		Weighing balance
		Measuring jar
		Septum-capped vial
		Refrigerator
		Dropping funnel
		Combination column (Upper column -150mm× 12.5mm ² Lower column 175mm length internal dia 10mm with capillary outlet &Teflon stopcock)
11	Heavy Metals 6.2.1 & Table 2 (Cl. 16 of IS 1699)	Ammonia Solution
		Ammonium hydroxide
		Distilled water
		Weighing balance
		Measuring jar
		Volumetric flask
		Nitric acid
		Hydrogen sulphide
		Hydrochloric acid
		Sulphuric acid

		Lead nitrate stock solution(Lead nitrate, distilled water, nitric acid)
		Volumetric flask-1000ml
		Lead free glass containers
		Standard Lead solution
		Nessler tube-50ml
		Acetic acid
		Crucible
		Muffle furnace(500 to 600° C)
		Steam bath
		Litmus paper
		Cold water
12	Copper 6.2.1 & Table 2 (Cl. 15 of IS 1699)	A CHEMICAL METHOD 15.2.3
		Citric acid
		Ammonium hydroxide-sp-gr 0.92
		Conc. Hydrochloric acid
		Dithizone (diphenyl thiocarbazon)
		Chloroform
		Con. Nitric acid
		Con. Sulphuric acid
		Citric acid solution
		Gum Arabic solution
		Sodium diethyl dithiocarbamate
		Standard Strong Solution of Copper
		Pure crystallized Copper sulphate
		Distilled water
		Beaker-100ml
		Standard dilute solution of Copper
		Graduated flask-100ml
		Litmus paper
		Separating funnel
		Tube of heat resistant glass
		Water bath
		Nessler cylinder
		Flame Atomic Absorption Method 15.1.6
		Metal free-distilled water
		Nitric acid-sp.gr 1.42
		Perchloric acid

		Sulphuric acid
		Hydrochloric acid-sp.gr 1.16 to 1.18
		Standard Copper solution(Copper sulphate, Volumetric flask-1000ml,distilled water, standard flask)
		Weighing balance
		Measuring jar
		Volumetric flask-100ml,1000ml
		Pipette
		Air/Acetylene gases
		Atomic absorption spectrophotometer (180 to 600nm)
13	Mercury 6.2.1 & Table 2 (Cl. 15.1.8 of IS 1699)	Atomic Absorption Cold Vapour Technique
		Weighing balance
		Measuring jar
		Nitric acid- sp.gr 1.40
		Sulphuric acid sp.gr 1.84
		Hydrochloric acid sp gr 1.18
		Distilled water
		Potassium permanganate
		Hydroxylamine hydrochloride solution
		Stannous chloride solution(stannous chloride, Hydrochloric acid,beaker-250ml,Nitrogen)
		Beaker-500ml
		Metallic tin
		Nitrogen
		Chromic acid mixture
		Potassium chromate
		Volumetric flask-1000ml
		Magnesium perchlorate granules
		Mercuric chloride
		Mineralization apparatus
		Reflux condenser
		Bubblers(ground glass stopper fitted)
		Stopcock
		Receiver flask
		Condenser
		Mercury
		Glass beads
		Atomic Absorption Spectrophotometer
		Beaker -300ml

		Water vapour absorption apparatus
		Condensate reservoir
		Burner

List above is indicative only and may not be considered as exhaustive.

ANNEX B

SCHEME OF TESTING AND INSPECTION

FOR CARAMEL IS 4467 : 1996

1. **LABORATORY-** A laboratory shall be maintained which shall be suitably equipped (as per the requirement given in column 2 of Table 1) and staffed, where different tests given in the specification shall be carried out in accordance with the methods given in the specification.
 - 1.1 The manufacturer shall prepare a calibration plan for the test equipment.
2. **TEST RECORDS-** The manufacturer shall maintain test records for the tests carried out to establish conformity.
3. **PACKING AND MARKING-** The Standard Mark as given in Schedule of the licence shall be stenciled/printed on each container of **Caramel** or printed on the labels applied to the container, as the case may be, provided always that the material in each container to which this mark is thus applied conforms to every requirement of the specification.
 - 3.1 **Packing** – The material shall be filled in amber-coloured glass or high density polyethylene containers or any other well-closed suitable containers with as little air space as possible. The containers shall be such as to preclude contamination of the contents with metals or other impurities.
 - 3.2 **Marking** - Each container shall legibly and indelibly marked with the details mentioned under clause 7.2 of IS 4467. In addition, the following details shall be mentioned on each container legibly and indelibly:
 - a) BIS Licence No. CM/L_____.
 - b) BIS website details i.e – “For details of BIS Certification please visit www.bis.gov.in”
4. **Control Unit-** For the purpose of this Scheme, the total quantity of **Caramel** produced in a pan at one time shall constitute a control unit.
5. **LEVELS OF CONTROL –**
 - 5.1 **Routine Tests** : The routine tests on caramel, as indicated in Table 1 and at the levels of control specified therein, shall be carried out on the whole production of the factory which is covered by this scheme and appropriate records and charts maintained in accordance with paragraph 2 above. All the production which conforms to the Indian Standards and covered by this license shall be marked with the Standard Mark.

- 5.2 **Type Tests:** In addition to the routine tests, type tests as given in Table 1 shall be carried out to approve the formulation and quality of the product in the beginning of marketing and or certification. Thereafter, a sample from a control unit be subjected to these tests once in a month or whenever fresh consignment of sugar is received.

Note: These tests are also conducted periodically to supplement to the routine tests or whenever the basic formula or method is changed.

6. **RAW MATERIAL** – It is recommended that routine analysis of various raw materials used in the manufacture of Caramel shall be made on each lot received in the factory and appropriate records to be maintained.
7. **HYGIENIC CONDITIONS:** The whole factory should be maintained in a clean and hygienic condition in accordance with code of Hygienic conditions for food processing units as laid down in IS 2491.
8. **REJECTION-** Disposal of non-conforming product shall be done in such a way so as to ensure that there is no violation of provisions of BIS Act, 2016.

TABLE 1
LEVELS OF CONTROL

(1)				(2)	(3)		
Test Details				Test equipment requirement R: required (or) S: Sub-contracting permitted	Levels Of Control		
Cl.	Requirements	Test Method Cl. Ref.	Test Method IS		No. of Samples	Frequency	Remarks
6.1.1	Description	3.1.1	IS 4467	R	One	Each Control Unit	
6.1.2 &Table 1	ROUTINE TEST REQUIREMENT				One	Each Control Unit/Each mix	If control units are separately produced and not mixed then from a control unit and if c.u. produced in a day are mixed then from each mix. Please See Notes 3 of Table 1.
	Solid Content	Annex-A	IS 4467	R			
	Colour intensity	Annex-B		R			
	Ammoniacal Nitrogen	Annex-C		R			
	4-Methylimidazole *	Annex D		R			
	Lead	15	IS 1699	R			
	Arsenic	15		R			
6.2.1 & Table 2	TYPE TEST REQUIREMENT				One	Once in a month or whenever fresh consignment of sugar is received.	
	Total Sulphur	Annex-E	IS 4467	S			
	Sulphur dioxide **	Annex-F		S			
	Total Nitrogen	Annex-G		S			
	2-Acetyl-4-tetrahydroxy butylimidazole ***	Annex-H		S			
	Heavy Metals	16	IS 1699	S			
	Mercury	15		S			
	Copper	15		S			

Note-1: Whether test equipment is required or sub-contracting is permitted in column 2 shall be decided by the Bureau and shall be mandatory. Sub-contracting is permitted to a laboratory recognized by the Bureau or Government laboratories empaneled by the Bureau.

Note-2: Levels of control given in column 3 are only recommendatory in nature. The manufacturer may define the control unit/batch/lot and submit his own levels of control in column 3 with proper justification for approval by BO Head.

Note-3: All control units produced in a day, mixed together shall constitute a “Mix”.

Note-4: * Applicable for Type III and Type IV caramel only. ** Applicable for Type II and Type IV caramel only. ***Applicable for Type III Caramel only.