

#### PRODUCT MANUAL FOR HEXANE, FOOD GRADE ACCORDING TO IS 3470:2017

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	:	IS 3470:2017				
	Title	:	HEXANE, FOOD GRADE				
	No. of Amendments	:	01				
2.	Sampling Guidelines:						
a)	Raw material	:	No specific requirement				
b)	Grouping guidelines	:	NA (No varieties of the product mentioned in IS)				
c)	c) Sample Size		1 litre of sample for complete testing.				
3.	List of Test Equipment	: Please see ANNEX - A					
4.	Scheme of Inspection and Testing	:	Please see ANNEX - B				
5.			sity at 20 °C, Colour (Saybolt), Determination of non n volatile residue, sulphur, lead, benzene content, polycyclic				
6.	Scope of the Licence :						
	"Licence is granted to use Standard Mark as per IS 3470:2017 with the following scope:						
	Name of the product	Hexane, Food Grade					

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## ANNEX-A TO PRODUCT MANUAL FOR HEXANE, FOOD GRADE ACCORDING TO IS 3470:2017

### LIST OF TEST EQUIPMENT

Major test equipment required to test as per the Indian Standard

S.No.	Test Equipment	Tests used in with clause Reference		
1.	Westphal or equivalent balance, Balance case, Sample Container with support, Water circulating pump, Constant Temperature bath, thermometer (or)	Density at 20 °C Table 1 Clause 3.2 (i)		
	Apparatus for Determination of Density or Relative Density by Capillary Stoppered Pyknometer and Graduated Bicapillary Pyknometer Methods as per IS 1448 (Part 32): Graduated Bicapillary Pyknometer, constant temperature bath or Capillary stoppered Pyknometer, bath thermometer, weighing balance capable of measuring 0.1mg, pyknometer holder (or)			
	Apparatus for Determination of Density through Hydrometer Method as per IS 1448 (Part 16): Hydrometer cylinder, glass hydrometers, constant temperature bath, thermometer, glass or plastics stirring rod			
2.	As per ASTM D 1218, Method A: high resolution refractometer with range 1.3300 to 1.500 (or) Method B: Automatic digital refractometer with range 1.3300 to 1.500	Refractive index Table 1 Clause 3.2 (ii)		
3.	Distillation Flasks, Condenser and cooling bath, metal shield or enclosure for flask, Gas burner/ electric Heater, graduated cylinders, thermometers	Distillation Table 1 Clause 3.2 (iii)		
4.	Oven (Thermostatically Controlled at 105+-5 deg C), 150 ml glass evaporating dish, dessicator, analytical weighing balance, Methyl Orange Indicator, distilled	Non-volatile residue Table 1 Clause 3.2 (iv)  Reaction of Non-volatile residue		
5.	water, laboratory glassware and reagents  Trace sulphur determination apparatus, Balance — 'Torsion' or equivalent, 500 g Capacity, heating mantle, nitrogen cylinder, transformer and other laboratory glassware/chemicals as per Annex-C of IS 3470:2017	Table 1 Clause 3.2 (v) Sulphur content Table 1 Clause 3.2 (vi)		
6.	Spectrophotometer — Fitted with covered absorption cells having a 1 cm light path, Shaking Machine (Optional) — Capable of	Lead as Pb Table 1 Clause 3.2 (vii)		

7.	approximately 250 rpm, Separatory Funnel — Squibb-type 125 ml volume and other reagents/glassware as per Annex – D of IS 3470:2017  Gas Chromatograph with flame ionization detector, aback flush system & suitable electronic recorder, columns and reagents as per Annex - E	Benzene Content Table 1 Clause 3.2 (viii)
8.	As per ASTM D 4367 – (for benzene only) Gas chromatograph with flame ionization detector, a back flush system & suitable electronic recorder /ASTM D 6730 –(all hydrocarbons) with hydrogen flame ionization detector, aback flush system & suitable electronic recorder	Aromatics Table 1 Clause 3.2 (ix)
9.	Separatory Funnel with PTFE stopcocks, Fused qurtz Spectrophotometric cells of 5.0 ± 0.005 cm, UV Spectrophotometer, iso-octane, DMSO, n-hexane (UV spectroscopic Grade), Naphthalene (Analytical grade) and reagents/glassware as per Annex F	Polycyclic aromatic hydrocarbons Table 1 Clause 3.2 (x)
10.	Adsorption columns, Zone measuring device, zero measuring device, UV light source, electric vibrator, hypodermic syringe, silica gel, fluorescent indicator – dyed gel and other apparatus/reagents as per IS 1448 (Part 23)	Saturates Table 1 Clause 3.2 (xi)
11.	Saybolt Chronometer and other apparatus/reagents as per IS 1448 (Part 14)	Colour, Saybolt – Table 1 Clause 3.2 (xii)

The above list is indicative in nature and may not be taken as exhaustive

# ANNEX – B SCHEME OF INSPECTION AND TESTING FOR HEXANE, FOOD GRADE ACCORDING TO IS 3470:2017

- 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 equipments.
- 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 the Schedule of the licence, shall be stenciled/printed on each container of Hexane, Food Grade or printed on the label applied to it, as the case may be, provided always that the material in each container to which this mark is thus applied, conform to every requirement of the specification.
- 3.1 Packing and marking shall be done as per the provision of the Indian Standard. 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.im".
- **4. CONTROL UNIT –** For the purpose of this scheme, the total quantity of Hexane, Food Grade from each storage tank of Hexane, Food Grade, at the refinery when ready for delivery and whenever fresh material is received in the tank for delivery shall constitute one control unit.
- 5. LEVELS OF CONTROL The tests as indicated in column 1 of Table 1 and the levels of control in column 3 of Table 1, shall be carried out on the whole production of the factory which is covered by this plan and appropriate records maintained in accordance with paragraph 2 above.
- 5.1 All the production which conforms to the Indian Standards and covered by the licence should be marked with Standard Mark.
- 5.2 On the basis of the test results, decision regarding conformity or otherwise of the material to the requirements of the specification shall be made as follows:
- 5.2.1 Two representative samples per day shall be drawn from the receiving tanks at the refinery and tested for the requirements of:
  - i) Distillation;
  - ii) Initial boiling point; and
  - iii) Distillation between 64 70°C
- 5.2.2 In case the sample is not meeting the above requirements of the specification or as indicated by the Refinery Process Control Equipment, suitable corrective measures shall be taken. A record of the test results obtained shall be maintained in Proforma I (attached).

- 5.2.3 One representative sample shall be drawn from each hexane storage tank at the refinery when ready for delivery and shall be tested for all the requirements of IS 3470:2017. When every fresh material is received into this tank, marking will be recommenced only after the tank is resampled and tested for all the requirements of this specification.
  - 5.2.4 If the sample drawn and tested as at 5.2.3 above conforms to all the requirements of the specification, the material may be marked. A record of the test results obtained shall be maintained in Proforma II (attached).
  - 5.2.5 Once in three months, a representative sample shall be drawn from each storage tank at the regional depots and tested for the following requirements:
    - i. Distillation;
    - ii. Density at 20°C;
  - iii. Non-volatile residue; and
  - iv. Reaction of non-volatile residue
  - 5.2.6 All records of test results obtained for sample drawn from Regional Depots shall be maintained at the Refinery/Central Office of the licensee in Proforma III attached to this Scheme.
  - 5.2.7A record of dispatch of consignments made from the main installation to the regional depots shall be maintained in Performa IV attached.
  - 5.2.8 A record of supplies of consignments received shall be maintained in Proforma V by the regional depots.
  - 5.2.9 A record of supplies delivered to the consignees shall be maintained in Proforma VI by the supply points.
  - 6.0 **TEST CERTIFICATE**: Each supply of Hexane, Food Grade made from the main installation or regional depots shall be accompanied by a certificate bearing standard mark as per proforma VII provided always that the material so supplied fully conforms to all the requirements of the specification.
  - 7. **REJECTIONS**—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	L	_evels of Con	itrol
CI.	Requirement	Test Method Cl. Ref.	Test Method IS	equipment requireme nt R: required (or)S: Sub- contractin g permitted	No. of Sampl e	Frequenc y	Remarks
3.1	General	3.1	IS 3470 : 2017	R	-	-	Visual
3.2 and Tabl e 1	i)Density at 20°C	-	(P: 32) <sup>1)</sup> /P:16 of IS 1448*/AST M D 4502/ISO 3675/ISO 12185	R	One	Twice a day from the receiving tank	
-do-	ii)Refracti ve index,	-	ASTM D 1218	R	One	-do-	
-do-	iii) Distillation	-	(P:18) <sup>2)</sup> of IS 1448/AST M D 86/ ISO 3405	R	One	-do-	
	a)Initial boiling point, °C	-					
	b)Distilled between 64- 700 °C	-					
-do-	iv)Non – volatile residue, g/100 ml	Annex A	IS 3470 : 2017	R	One	Once in 3 months each storage tank at regional depots	
-do-	v)Reaction of non- volatile residue	Annex B	IS 3470 : 2017	R	One	-do-	
-do-	vi) Sulphur content, mg/kg	Annex C <sup>3)</sup>	IS 3470 : 2017/ ASTM D 5453	R	One	Each control unit	

-do-	vii) Lead, as pb, mg/kg	Annex D <sup>4)</sup>	IS 3470 : 2017/AST M D 3605	R	One	-do-
-do-	viii)Benzene content, percent (v/v)	Annex E <sup>5)</sup>	IS 3470: 2017/ ASTM D 4367/AST M D 6730/ASTM D 6229	R	One	-do-
-do-	ix)Aromatic s, percent (v/v)	-	ASTM D 4367/AST M D 6730	R	One	-do-
-do-	x) Polycyclic aromatic hydrocarbons	Annex F	IS 3470 : 2017	R	One	-do-
-do-	xi) Saturates, percent by mass	-	P : 23 of IS 1448	R	One	-do-
-do-	xii) Colour, Saybolt		P: 14 of IS 1448	R	One	-do-

<sup>\*</sup> Method of test for petroleum and its products

- $^{1)}$  In case of dispute (P : 32) shall be referee method.
- <sup>2)</sup> In case of dispute (P: 18) shall be referee method.
- 3) In case of dispute Annex C shall be referee method.
- $^{\rm 4)}$  In case of  $\,$  dispute Annex D shall be referee method.
- 5) In case of dispute Annex E shall be referee method.

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: The control unit and levels of control as decided by the Bureau are obligatory to which the licensee shall comply with.

### PROFORMA I (Clause 5.2.2 of the SIT) (Refinery Receiving Tank)

Date	Sample	Receiving	С	Remarks	
			Initial boiling point °C Distillation between 64-70° C		
(1)	(2)	(3)	(4)	(5)	(6)

#### PROFORMA II

### (Clause 5.2.4 of the SIT) (Refinery Delivery Tank)

Date	Refinery Tank No.	General	Density at 20°C	Referactive Index	Dis	tillation	Non-Volatile residue g/100ml	Reaction of non- volatile residue
					Initial boiling point °C	Distilled between 64-70°C		
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)

Sulphur content, mg/kg	Lead as pb mg/kg	Benzene content % (v/v)	Polycyclic aromatic hydrocarbons	Saturates % by mass	Remarks
(10)	(11)	(12)	(13)	(14)	(15)

### **PROFORMA III**

### (Clause 5.2.6 of the SIT)

e of receipt ne sample	From	Storage tank No. (to be informed by the depot sending the sample)	Date of testing	General	Density at 20°C	Refractive Index	Distillation	
							Initial boiling point <sup>o</sup> C	Distilled between 64-70°C
(1)	(2)	(3)	(4)	(5)	(6)	(7)	(8)	(9)

Non-volatile residue g/100 ml	Reaction of non- volatile residue	Sulphur, mg/kg	Lead mg/kg	Benzene % (v/v)	Polycyclic aromatic hydro carbons	Saturates % by mass	Colour, Saybolt	Remarks
(10)	(11)	(12)	(13)	(14)	(15)	(16)	(17)	(18)

### TO BE MAINTAINED AT THE MAIN INSTALLATION PROFORMA IV

#### (Reference Clause 5.2.7 of the SIT)

#### (Record of Dispatches to the Regional Depots)

Date	Storage Tank No.	Tank wagon/Tank Lorry/Barrel Ref No. and capacity	Quantity Filled	Destination	Remarks
(1)	(2)	(3)	(4)	(5)	(6)

### TO BE MAINTAINED AT THE REGIONAL DEPOTS PROFORMA V

### (Reference Clause 5.2.8 of the SIT) (Records of Receipts)

Place-----

Date of Receipt	Tank Wagon/Tank Lorry/Barrel Ref No. and capacity	Quantity Received	Storage Tank No.	Remarks
(1)	(2)	(3)	(4)	(5)

### **PROFORMA VI**

### (Reference Clause 5.2.9 of the SIT) (Record of Dispatches to consignees)

Date	Storage Tank No.	Tank Wagon/Tank Lorry/Barrel Ref No. and capacity	Consignee	Quantity Delivered	BIS Mark Certificate No.	Remarks
(1)	(2)	(3)	(4)	(5)	(6)	(7)

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(Name of the Oil Company)

### PROFORMA VII

(Reference Clause 6.0 of the SIT)

### TEST CERTIFICATE FOR HEXANE, FOOD GRADE

Licence No	Serial No						
Place	Date						
Consignee's Name and Address							
Quantity Supplied :	Litres						
Ex our storage Tank No.							
Ex our Tank Lorry No.							
Ex In our Barrels							
Certified that the quantity supplied as above fully confo 3470:2017 Specification for Hexane, Food Grade.	Certified that the quantity supplied as above fully conforms to the requirements of IS 3470:2017 Specification for Hexane, Food Grade.						
	( Signature )						