



भारत सरकार  
GOVERNMENT OF INDIA

रक्षा मंत्रालय  
MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश  
JOINT SERVICES SPECIFICATION

ON

**CARBON BLACK TYPE 1, 2, 3 & 4**  
(DS Cat. No.)

(Carbon Black Type 1 6810-000 964)  
(Carbon Black Type 2 6810-000 965)  
(Carbon Black Type 3 6810-001 129)  
(Carbon Black Type 4 6810-001 130)

Issued by

मानकीकरण निदेशालय  
रक्षा उत्पादन विभाग  
रक्षा मंत्रालय  
'एच' ब्लॉक, निर्माण भवन डाकघर  
नई दिल्ली-110 011

**DIRECTORATE OF STANDARDISATION  
DEPARTMENT OF DEFENCE PRODUCTION  
MINISTRY OF DEFENCE  
'H' BLOCK, NIRMAN BHAVAN POST OFFICE  
NEW DELHI-110 011**

**LIST OF MEMBERS ASSOCIATED WITH FORMULATION OF THIS STANDARD**

**1** This Joint Services Specification has been approved by Shri RS Gauba, Sc 'G', Associate Director, PO-II, DRDO, Chairman, Armament Standardisation Sub-committee.

**2** The following members have been present/consulted in preparing the document:

<i>S No.</i>	<i>Name &amp; Designation</i>	<i>Organisation</i>
1	Shri Benzamin Lionel, Sc 'G' HPO-II	Programme Office-II, DRDO Orgn, New Delhi
2	Col Sushil Chander	ADGWE / GS (WE - 2 / 3), New Delhi
3	Col Himanshu Tiwari	Dte of Arty (GS / Artillery - 5), New Delhi
4	Capt. Roopak Barua, DNAI	Dte Gen of Naval Armt, Naval HQ, New Delhi
5	Air Cmde Sunil Padegaonkar, PDA (ASE)	Dte of Armt & Safety Eqpt, Air HQ, New Delhi
6	Lt. Col A.V Kulkarni, JD, EME, Armt	DGEME, Army HQ, New Delhi
7	Shri. P Upadhyay, PDONA/DGONA	DGNAI, Naval HQ, New Delhi
8	Shri A.K. Parashar, Director	DGAQA, JD (Armt) Gp, New Delhi
9	Shri L Srinivasan, SSO-I	CQA (ME), Pune
10	Shri Ashok Kumar, PScO (NFSG)	CQA (Amn), Pune
11	Lt Col Tapan Sharma	CQA (SA), Ichapur, West Bengal
12	Shri K. Yadav, PScO	CQA (W), Jabalpur
13	Dr.[Mrs.] N. Sikder, Sc 'G'	HEMRL, DRDO, Pune
14	Shri P. W. Sonawane, Jt. Director	ARDE / DRDO Orgn, Pune
15	Shri V.K. Tiwari, Addl GM	Ammunition Factory, Pune
16	Shri Satvinder Singh, SSO - II	Secretary ASSC

**RECORD OF AMENDMENTS**

<i>Amendment</i>		<i>Amendment pertains to : S No./Para No./ Column No.</i>	<i>Authority</i>	<i>Amended by</i>	<i>Signature &amp; Date</i>
<i>No.</i>	<i>Date</i>			<i>Name &amp; Appointment (IN BLOCK LETTERS)</i>	

	<b>CONTENTS</b>	<i>Page No.</i>
0	FOREWORD	1
1	SCOPE	3
2	RELATED SPECIFICATIONS/DOCUMENTS	3
3	MATERIAL/FINISH	3
4	MANUFACTURE	3
5	TENDER SAMPLE	4
6	PRE-INSPECTION OF STORES/CONSIGNMENT	4
7	QUALITY ASSURANCE	4
8	WARRANTY	7
9	PACKAGING	7
10	MARKING	8
11	DEFENCE STORES CATALOGUE NUMBER	8
12	SAFETY OF OPERATIONS	8
13	SUGGESTIONS FOR IMPROVEMENT	9
	APPENDICES 'A' TO 'H'	10-18

## **0 FOREWORD**

**0.1** This Joint Services Specification has been prepared by the Armament Standardisation Sub Committee on the authority of the Standardisation Committee, Ministry of Defence.

**0.2** This specification has been approved by the Ministry of Defence and is mandatory for use by the Defence Services.

**0.3** This JSS 6810-81 : 2016, (Revision No. 3).

a) was revised in the year 2004.

b) is a revision of JSS 6810-81 : 2010, (Revision No. 2) and supersedes the same.

**0.4** This specification would be used for Supply and Quality Assurance of Carbon Black Type 1, 2, 3 & 4.

**0.5** Quality Assurance Authority for the item covered by this specification is the Controller, Controllerate of Quality Assurance (Military Explosives), Aundh Road, Pune-411 020. Enquiries regarding technical parameters shall be addressed to the Quality Assurance Authority, while other enquiries shall be referred to:

The Director,  
Directorate of Standardisation,  
Ministry of Defence,  
'H'-Block, Nirman Bhawan PO,  
New Delhi-110 011

**0.6** Non registered users can obtain the following on payment:

**(a) Copies of IS from:**

Bureau of Indian standards,  
Manak Bhawan,  
9, Bahadur Shah Zafar Marg,  
New Delhi-110 002

or

Their regional/Branch offices.

**(b) Copies of JSSs/JSGs from:**

The Director,  
Directorate of Standardisation  
Standardisation Documents Centre,  
Ministry of Defence, Room No. 05,  
'J'-Block, Nirman Bhawan PO,  
New Delhi-110 011

**JSS 6810-81 : 2016**  
**(Revision No. 3)**

**0.7** Indian Standard (IS) specifications are available free of cost for registered users on:

Directorate of Standardisation Website

**[www.ddpdos.gov.in](http://www.ddpdos.gov.in)**

For registration visit our website.

**0.8** This specification holds good only for the supply order for which it is issued.

**0.9 Directorate of Standardisation Website:** All the approved JSSs/JSGs are available on the Directorate of Standardisation Website **[www.ddpdos.gov.in](http://www.ddpdos.gov.in)**. Defence Organisations desirous of accessing a copy of this document are requested to approach the Directorate of Standardisation for obtaining user id/password to access the website.

## **1 SCOPE**

**1.1** This specification is meant to govern supply and quality assurance of Carbon Black Type 1, 2, 3 & 4.

- a) Type 1 and 2 For use in explosives and propellants having Iodine adsorption value as given in clause 7.4.1.
- b) Type 3 For making master batch by incorporation in the high density polythene used for bar mines. Also suitable for use in the repair of mines.
- c) Type 4 For use in the manufacture of charge 8 and 9 of 155 mm Ammunition having adsorption value as given in clause 7.4.1.

## **2 RELATED SPECIFICATIONS/DOCUMENTS**

**2.1** Reference is made in this specification to:

<i>S No.</i>	<i>Specification No. &amp; Year</i>	<i>Nomenclature</i>
a)	IS 138 : 1992 (Third Revision) Reaffirmed 2014 AMD 1	Ready Mixed Paint, Marking for Packages and Petrol Containers-Specification.
b)	IS 460 (Part 1) : 1985 (Third Revision) Reaffirmed 2008 AMD 1	Specification for Test Sieves : Part I Wire Cloth Test Sieves.
c)	IS 7498 : 1985 (First Revision) Reaffirmed 2012	Methods of Sampling and Test for Carbon Black.
d)	JSG 0112 : 2015 (Revision No. 2)	General Methods of Tests and Assessment of Impurities in Chemicals/Materials used in the Manufacture of Explosives and Ammunition.

## **3 MATERIAL/FINISH**

**3.1** Carbon Black Type 1, 2, 3 & 4 shall be uniform, soft, dry and black fine powder free from foreign matter, grit and visible impurities.

## **4 MANUFACTURE**

**4.1** Carbon Black Type 1, 2, 3 & 4 shall be manufactured by the incomplete combustion of carbonaceous fuels and shall be of such quality as to meet the requirements of this specification.

## **5 TENDER SAMPLE**

**5.1** The manufacturer/supplier/contractor shall submit three tender samples of 250 g, essentially from the same batch/lot of manufacture, free of all charges and conforming to this specification, when called for in the tender to the Quality Assurance Officer/Quality Assurance Authority stated in the tender.

**5.2** If practical trials are required at the tender stage the same shall be specified in the tender and the quantity required shall be indicated in the tender enquiry.

**5.3** Carbon Black Type 1, 2, 3 & 4 with their respective Iodine adsorption value as given in clause 7.4.1 shall be specified in the tender enquiry.

## **6 PRE-INSPECTION OF STORES/CONSIGNMENT**

**6.1** Manufacturers/contractors must satisfy themselves that the stores are in accordance with the terms of contract and fully conform to the required specification, by carrying out a thorough pre-inspection of each lot before actually tendering the same for inspection to the Quality Assurance Officer nominated under the terms of the contract. A declaration by the contractor that a necessary pre-inspection has been carried out on the stores tendered will be submitted along with the challan. The declaration will also indicate the method followed in carrying out pre-inspection showing the features checked/tested and will have the test certificate attached to the challan/declaration.

**6.2** If the Quality Assurance Officer finds that the pre-inspection of the consignment as required above has not been carried out, the consignment is liable for rejection.

## **7 QUALITY ASSURANCE**

### **7.1 Inspection**

**7.1.1** The Carbon black type 1, 2, 3 & 4 and the packages in which it is contained shall be subject to inspection by and to the final approval of the Quality Assurance Officer/Quality Assurance Authority.

**7.1.2** Sample of the material and of the packages may be taken from any portion of the batch/lot/consignment.

### **7.2 Sampling**

**7.2.1** A representative sample of 250g shall be drawn from each container. Normally the number of containers to be selected at random from a batch/lot shall depend on the size of the batch/lot and shall be in accordance with the following table:

<i>No. of Containers in a Batch/Lot</i>	<i>No. of Containers to be Sampled</i>
Up to 25	3
26 to 50	4



<i>No. of Containers in a Batch/Lot</i>	<i>No. of Containers to be Sampled</i>
51 to 100	5
101 to 150	6
151 to 300	7
301 to 500	8
501 and above	10

### 7.3 Criteria for Conformity

**7.3.1** If on examination, any sample is found not to conform to this specification, the whole batch/lot/consignment shall be rejected.

**7.3.2** The foregoing provisions shall apply equally to prime contractors and to any sub-contractor.

### 7.4 Test Requirements

**7.4.1** Samples from any portion of batch/lot shall be in accordance with the clause 3 above and shall comply with the following test requirements:

#### **TEST REQUIREMENTS OF CARBON BLACK TYPE 1, 2, 3 & 4**

<i>S No.</i>	<i>Characteristics</i>	<i>Passing Standard</i>				<i>Test Method</i>
		<i>Type 1</i>	<i>Type 2</i>	<i>Type 3</i>	<i>Type 4</i>	
a)	Volatile Matter at 100°C -105°C for 3 h, % by mass	3.0 Max	3.0 Max	9.0 Max	2.5 Max	JSG 0112 Method 1(a)
b)	Moisture content, % by mass	-	-	1.0 Max	-	IS 7498 Clause 9
c)	@ Bulk density g/ml	-	-	-	0.33 Min 0.43 Max	Appendix 'A'
d)	Matter soluble in water, % by mass	0.30 Max	0.30 Max	-	-	Appendix 'B'
e)	*pH of water extract	6.0 Min 8.0 Max	6.0 Min 8.0 Max	6.0 Min 8.0 Max	7.5 Min 8.5 Max	JSG 0112 Method 5(b)
f)	Matter soluble in ether, % by mass	0.50 Max	0.50 Max	- Max	- Max	Appendix 'C'
g)	Ash at 950°C - 1000°C, % by mass	0.50 Max	0.50 Max	1.0 Max	0.75 Max	JSG 0112 Method 2(a)
h)	Grit on ash, at S No. 7 passing through 63 micrometre IS Sieve	All to Pass	All to Pass	Free from grit	Free from grit	JSG 0112 Method 6

**JSS 6810-81 : 2016**  
**(Revision No. 3)**

S No.	Characteristics	Passing Standard				Test Method
		Type 1	Type 2	Type 3	Type 4	
j)	Fixed Carbon, % by mass	91.0 <i>Min</i>	91.0 <i>Min</i>	-	-	Appendix 'D'
k)	Sulphur Compounds, Calculated as Sulphuric acid (H <sub>2</sub> SO <sub>4</sub> ) per cent by mass	0.6 <i>Max</i>	0.6 <i>Max</i>	1.0 <i>Max</i>	1.0 <i>Max</i>	Appendix 'E'
m)	*** Iodine adsorption, mg/g by mass	35 <i>Min</i> -	75 <i>Min</i> -	- -	79 <i>Min</i> 85 <i>Max</i>	Appendix 'F'
n)	Toluene extractable matter, % by mass	-	-	0.1 <i>Max</i>	-	Appendix 'G'
p)	Relative density in g/ml	- -	-	1.5 <i>Min</i> 2.0 <i>Max</i>	-	Appendix 'H'
q)	Particle size, micrometer	**0.05 <i>Min</i> 0.095 <i>Max</i>	**0.03 <i>Min</i> 0.05 <i>Max</i>	0.02 <i>Min</i> -	- -	** See remark below
r)	Water soluble chlorides, calculated as Sodium chloride (NaCl), % by mass	-	-	0.1 <i>Max</i>	-	JSG 0112 Method 7 (b)
s)	Water soluble sulphates, calculated as Sodium sulphate (Na <sub>2</sub> SO <sub>4</sub> ), % by mass	-	-	0.25 <i>Max</i>	-	JSG 0112 Method 8
t)	Sieving	All to pass Through 63 micrometer IS Sieve	All to pass 63 micrometer IS Sieve	Retention on 45 micrometer IS Sieve, 0.5% <i>Max</i>	****(a) Retention on 125 micrometer IS Sieve, 1.0% <i>Max</i>	JSG 0112 Method 18

<i>S No.</i>	<i>Characteristics</i>	<i>Passing Standard</i>				<i>Test Method</i>
		<i>Type 1</i>	<i>Type 2</i>	<i>Type 3</i>	<i>Type 4</i>	
					****(b) Retention on 2 mm Sieve 1.0% Max	

NOTES-

**1** Particulars of IS Sieve references shall be found in IS 460 (Part 1).

**2** Particle size has to be certified by the manufacturer.

\* pH can be relaxed by 0.5 on either side for indigenous supplies base on individual merit of cases.

\*\* Limits suggested are based on Yugo requirements of specific surface. Test method by surface area analyzer based on low temperature absorption technic or any other suitable method.

\*\*\* Type 1 is similar to ROF 51 of Yugo.

Type 2 is similar to ROF 58 of Yugo.

\*\*\*\* a) and b) as per Bofor's requirements.

@ At present this test will be carried out for generating data.

## **8 WARRANTY**

**8.1** The stores supplied against this specification shall be deemed to bear warranty for 12 months from the date of receipt of store at consignee's end and against defective design/material/workmanship/performance. If during this period any of the stores supplied is found defective, the same shall be rectified/replaced by the contractor, free of charge, at the user's premises within a period of three months from date of intimation of defect.

## **9 PACKAGING**

**9.1** The store shall be packed in a polythene bag 0.13 mm thick suitably closed and then in sound clean dry airtight tin containers or mild steel drums painted externally.

**9.2** The quantity in each package shall be 25 kg or as stated in the contract.

**JSS 6810-81 : 2016**  
**(Revision No. 3)**

**9.3** Any other form of package shall have the prior approval of the Quality Assurance Officer/Quality Assurance Authority.

**9.4** The inclusion of any foreign matter or impurities in any of the packages shall render the whole batch/lot/consignment liable to rejection.

## **10 MARKING**

**10.1** All packages containing the material shall be indelibly and legibly marked with the following details:

- a) Nomenclature and Specification Number of the Material.
- b) Name and Address of the Consignee.
- c) A/T or S.O. Number and Date.
- d) Consignment Number.
- e) Batch No. and Date of Manufacture.
- f) Gross and Net Mass.
- g) Consecutive Number of Package and Total Number of Packages in Consignment.
- h) Date of Supply.
- j) Manufacturer's Initials or Recognised Trademark.

**10.2** In addition to the above, the Quality Assurance Officer may suggest some more markings/identifications suitable at the time of inspection.

**10.3** The paint used for marking should conform to IS 138 and to the satisfaction of the Quality Assurance Officer/Quality Assurance Authority.

## **11 DEFENCE STORES CATALOGUE NUMBERS**

**11.1** The Defence Stores Catalogue Numbers allotted to Carbon Black Type 1, 2, 3 & 4 are:

<i>S No.</i>	<i>Nomenclature</i>	<i>DS Cat. No.</i>
a)	Carbon Black Type 1	6810-000 964
b)	Carbon Black Type 2	6810-000 965
c)	Carbon Black Type 3	6810-001 129
d)	Carbon Black Type 4	6810-001 130

## **12 SAFETY OF OPERATIONS**

**12.1** Nothing in this specification shall relieve the supplier/contractor of his responsibility for the safety of operations in the manufacture, storage, transit or use of this store.

**13 SUGGESTIONS FOR IMPROVEMENT**

**13.1** Any suggestion for improvement in this document may be forwarded to:

The Director,  
Directorate of Standardisation,  
Ministry of Defence,  
'H' Block, Nirman Bhawan PO,  
New Delhi-110 011

**A DETERMINATION OF BULK DENSITY**

**A.1** Place about 5 g of the material accurately weighed (M) in a glass cylinder measuring about 150 mm height and 18 to 20 mm in internal diameter and graduated in 0.5 ml. Close the cylinder with a rubber stopper. Drop it 100 times vertically from a height of 65 mm on a piece of hard leather. Level off the surface of the column of sample, by minimum tapping on the side of the cylinder. Read the volume occupied by the sample.

$$\text{Bulk Density} = \frac{\text{Mass of sample}}{\text{Volume}}$$

NOTES-

- 1 The reading of a duplicate test on the sample should be within 2 ml.
- 2 The above procedure is conveniently arranged by sliding the glass cylinder through 2 wooden filter stand rings, clamped one above the other on the same support, lower ring being so arranged as to limit the level of the cylinder to 65 mm.

Appendix 'B'

**B DETERMINATION OF MATTER SOLUBLE IN WATER**

**B.1** Shake about 5 g of the accurately weighed sample ( $M_1$ ) with 100 ml of distilled water for about 2 hours. Centrifuge the mixture and filter by decantation. Collect the filtrate and make upto mark with distilled water in a 250 ml volumetric flask. Pippetee out 50 ml of this solution after mixing well into a tared dish ( $M_2$ ) and evaporate nearly to dryness, carefully. Dry in an oven at 100°C to 105°C to constant mass. Cool and weigh ( $M_3$ ).

$$\text{Matter soluble in water, \% by mass} = \frac{(M_3 - M_2)}{M_1} \times 500$$

**C DETERMINATION OF ETHER SOLUBLE MATTER**

**C.1** Weigh accurately 2 g of the sample (M) into an extraction thimble. Plug well with a wad of glass wool and with a filter paper. Place the thimble in a soxhlet and extract with Sulphuric ether for two hours. The solvent shall siphon at least 8 times per hour, otherwise the period shall be suitably prolonged.

NOTE- If extraction thimble is not available the sample may be made into a packet with No. 42 Whatman filter paper.

Take the extract and evaporate to dryness on a water bath. Place in an oven at 100°C to 105°C and dry to constant mass.

$$\text{Matter soluble in ether, \% by mass} = \frac{(M_2 - M_1) \times 2.841}{M} \times 100$$

Where:

M = Mass of sample taken.

M<sub>1</sub> = Mass of empty flask.

M<sub>2</sub> = Mass of flask containing residue.



**Appendix 'D'**

**D DETERMINATION OF FIXED CARBON**

**D.1** Weigh a clean dry platinum or silica crucible with lid ( $M_1$ ). Transfer about 2 g of the sample into the crucible, close and reweigh ( $M_2$ ). Place the crucible with lid in a muffle furnace at a temperature of 930°C to 970°C for seven minutes. Remove, cool and reweigh ( $M_3$ ).

$$\text{Fixed carbon, \% by mass} = 100 - \left( \text{Ash \%} + \frac{M_2 - M_3}{M_2 - M_1} \times 100 \right)$$

NOTE- It is essential to follow the correct temperature and duration of heating.

**E DETERMINATION OF SULPHUR BY ESCHKA METHOD**

**E.1** Weigh accurately about 1 g of the sample and mix thoroughly on a glazed paper with 3 g of Eschka mixture (Thoroughly mix two parts (by mass) of light calcined Magnesium oxide and 1 part (by mass) of anhydrous Sodium carbonate). Transfer to a porcelain or platinum crucible of 30 ml capacity and cover with 1 g of Eschka mixture.

**E.2** Place the crucible in a cold muffle furnace and gradually raise the temperature to 800°C to 850°C in about an hour. Maintain at this temperature for about 90 minutes. Remove the crucible and cool before transferring to a 250 ml beaker.

**E.3** Digest with 100 ml hot distilled water for 30 to 40 minutes with occasional stirring. Remove the crucible and wash the contents in the crucible thoroughly into the beaker with distilled water. Filter the supernatant liquid through a No. 41 Whatman filter paper. Wash the insoluble matter successively with at least 5 portions of about 25 ml distilled water and filter. Collect the filtrate and washing and boil down to 150 ml. Add about 20 ml of saturated Bromine water and boil and make slightly acidic with Hydrochloric acid. Boil again to expel excess of Bromine. Make just neutral to Methyl orange with 10% solution of Sodium hydroxide. Add 1 ml of 1 N Hydrochloric acid and boil again and add slowly with constant stirring 10 ml of 10% Barium chloride solution. Boil for about 15 minutes and digest the precipitate for atleast two hours at a temperature just bellow boiling or preferably leave overnight at room temperature.

**E.4** Filter through ashless filter paper (e.g. No. 42 Whatman) or through asbestos padded gooch, crucible and wash with hot distilled water until free from chlorides. Dry the funnel and contents in a hot air oven at about 100°C. Place the filter paper and precipitate in a tared porcelain, silica or platinum crucible, burn off the filter paper at as low a temperature as possible and finally ignite at 900°C to 950°C to constant mass. In case gooch crucible is used, dry at 100°C and ignite at 900°C to 950°C to constant mass.

**E.5** Carry out a blank exactly as described above using the same amount of all reagents that were employed in the regular determination.

$$\begin{array}{l} \text{Sulphur as Sulphuric acid,} \\ \text{\% by mass} \end{array} = \frac{(\text{Mass of precipitate-blank}) \times 42.08}{\text{Mass of sample}}$$

Appendix 'F'

**F DETERMINATION OF IODINE ADSORPTION**

**F.1** Dry an adequate sample of carbon black for 1 hour at 105°C. Weigh accurately about 0.5 g of the dried sample into a weighing bottle.

**F.2** Pipette 25.0 ml of 0.05 N Iodine solution into the weighing bottle and put stopper immediately. Shake for 30 s and allow to stand for 5 minutes. If a centrifuge is used, allow to shake for 30 s, then centrifuge immediately.

**F.3** Filter with slight suction through a clean, dry, gooch crucible into a clean, dry 50 ml test tube placed under the end of the crucible holder inside a 1 litre filtering flask (Witt's). The end of the crucible holder should be above the surface of the solution in the test tube after filtering. If a centrifuge is used filtration is not necessary and the solution may be decanted into a 50 ml beaker.

**F.4** Immediately after filtering the iodine solution into the test tube or decanting the centrifuged solution, pipette 20.0 ml into an Iodine flask. Titrate the Iodine solution with 0.05 N Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution until a pale yellow colour remains. Add approximately 5 ml of 0.02% starch solution as indicator and continue titrating until the blue colour just disappears (V).

**F.5** Carry out a blank (V1) exactly as above but without the sample.

**Calculation**

Calculate the Iodine adsorption number, in milligrams of iodine per gram of Carbon Black, as follows:

$$I = \frac{(V1-V) \times 6.346 \times f \times 1.25}{M}$$

Where:

I = Iodine adsorption number, in milligrams of iodine per gram of Carbon Black.

V = Millilitres of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution required for titration of the sample (Para 4 above).

V1 = Millilitres of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> solution required for titration of the blank (para 5 above).

f = factor of 0.05 N Sodium thiosulphate solution.

M = Mass of sample taken for the test.

**G DETERMINATION OF TOLUENE EXTRACTABLE MATTER**

**G.1 Apparatus**

- a) Extraction thimbles-Double thickness.
- b) Soxhlet apparatus.
- c) Shallow weighing dish.
- d) Oven.
- e) Balance.
- f) Desiccator.

**G.2 Reagent :** Analytical reagent type toluene sulphur free.

**G.3 Procedure**

- (a) Place 5 to 8 g of carbon black in a paper extraction thimble. Insert the thimble into the soxhlet extractor. Measure 50 to 60 ml of toluene into the soxhlet flask.
- (b) Assemble the soxhlet apparatus and extract for 22 h.
- (c) Evaporate successive small portions of the extract solution (filtered of necessary) nearly to dryness in the previously cleaned, dried and tared 50 ml shallow glass weighing dish. Rinse the extraction flask with toluene and add the washings to the dish. Evaporate the combined extracts on a hot plate to a volume of approximately 5 ml to 10 ml and finally dry the dish and the contents in an oven at 115°C until dry, cool in a desiccator to room temperature and weigh.

$$\text{Toluene extractable Matter, \% by mass} = \frac{\text{Mass of the extract}}{\text{Mass of sample}} \times 100$$

NOTE- If extraction thimble is not available the sample may be made into a packet with a No. 42 Whatman filter paper.

Appendix 'H'

**H DETERMINATION OF RELATIVE DENSITY**

**H.1 Apparatus**

- i) Relative density bottle of 50 ml capacity with a capillary stopper provided with a cap.
- ii) Water bath.
- iii) Desiccator.
- iv) Weighing bottle-fitted with cork, the neck being small enough to fit inside the neck of the relative density bottle
- v) Vacuum pumps :
  - aa) Water pump-to expel greater portion of the air in the desiccator.
  - ab) oil pumps-provided with a motor to give a vacuum of not more than 3 mm.
- vi) Monometer : An open tube manometer made of glass tubing 6 mm dia., filled with mercury to approximately 86 cm fitted with rubber pressure tubing attached to a T-joining leading to the desiccator and the pump.
- vii) Thermometer : having a range of 0°C to 60°C graduated in 0.1°C.

**H.2 Procedure**

*Relative density of kerosene* : Before proceeding with the determination of relative density of the carbon black, it is necessary to determine the relative density of the Kerosene to be used in the test.

Fill the relative density bottle with freshly boiled cooled distilled water, place it in a thermostat maintained at 30°C till the temperature equilibrium is reached and then wipe, dry and weigh. Next, fill the relative density bottle with Kerosene to be used in the determination, bring to 30°C and wipe, dry and weigh.

$$\text{Relative density of Kerosene} = \frac{\text{Mass in g of Kerosene}}{\text{Mass in g of water}}$$

**JSS 6810-81 : 2016**  
**(Revision No. 3)**

Dry the carbon black at 105°C for two hours. Pour sufficient Kerosene into the bottle to form a 6 mm layer at the bottom and add about 4 g of sample from the previously weighed weighing bottle. Weigh the weighing bottle again. Stir the sample with polished round bottomed glass rod until completely covered by the Kerosene, adding more Kerosene if necessary, wash the rod with Kerosene into the relative density bottle. Place the relative density bottle in the desiccator, close and connect the desiccator to the water pump and evacuate greater part of air from the system. This may take 5 to 10 minutes. Close the system with a pinch cock and connect the desiccator to the oil pump for the removal of the small amounts of air given off at the low pressure obtainable with oil pump. Read the manometer to see the proper vacuum is maintained and when it indicates the vacuum, which should not be greater than 3 mm is constant, cut off the oil pump for short period taking precautions to prevent leakage. It will be noticed that bubbles of air come from the carbon black very rapidly at first and that this action gradually decreases and finally stops altogether. Which may require from 30 minutes to 2 h. When no more bubbles can be seen, assume that all the occluded air has been given off and the carbon black is thoroughly wet with Kerosene. Admit air into the desiccator slowly by means of a pinch cock.

Remove the relative density bottle and fill it with Kerosene taking care to prevent the formation of air bubbles. Place the thermometer in the water bath which is maintained at 25°C to 28°C. Carefully place the relative density bottle in the bath and allow it to come to the constant temperature. Then insert the capillary stopper, add warm water to the water bath to raise the temp. quickly to 29°C in order to expand the kerosene and prevent it from creeping down the capillary and admitting a small quantity of air. Add more warm water and gradually raise the temp. of water bath at 30°C. Wipe the capillary stopper with filter paper and put on the cap. Remove the relative density bottle from the bath and wipe dry. Allow it to stand for 30 minutes to enable it to come to room temperature and weigh.

Allow the relative density bottle to stand approximately for the same length of time before each weighing so as to compensate for slight errors due to evaporation at the joints.

$$\text{Relative density, \% by mass} = \frac{M1 \times S}{(M1 + M2) - M3}$$

Where:

M1 = Mass of Carbon Black.

S = Relative density of Kerosene.

M2 = Mass in g of bottle filled with Kerosene only.

M3 = Final mass in g of the bottle with Carbon black and Kerosene.