



भारत सरकार
GOVERNMENT OF INDIA

रक्षा मंत्रालय
MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश
JOINT SERVICES SPECIFICATION

ON

GRAPHITE DRY AND GRAPHITE DRY SULPHUR FREE
DS Cat. No. 9620-000 004 to 9620-000 013

Issued by

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RECORD OF AMENDMENTS

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

	CONTENTS	<i>Page No.</i>
0	FOREWORD	1
1	SCOPE	3
2	RELATED SPECIFICATIONS/DOCUMENTS	3
3	MATERIAL/FINISH	3
4	MANUFACTURE	4
5	TENDER SAMEPLE	4
6	PRE-INSPECTION OF STORES/CONSIGNMENT	4
7	QUALITY ASSURANCE	4
8	WARRANTY	6
9	PACKAGING	6
10	MARKING	7
11	DEFENCE STORES CATALOGUE NUMBER	7
12	SUGGESTIONS FOR IMPROVEMENT	8
13	APPENDIX 'A' TO 'F'	9-14

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 9620-01 : 2016, (Revision No. 3).

a) was revised in the year 2001.

b) is a revision of JSS 9620-01 : 2010, (Revision No. 2) and supersedes the same.

0.4 This specification would be used for Manufacture, Supply and Quality Assurance of Graphite, Dry and Graphite, Dry, Sulphur free.

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 9620-01 : 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

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**. 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 Manufacture, Supply and Quality Assurance of Graphite, Dry and Graphite, Dry, Sulphur free, of sizes 45, 63, 90, 125 and 150 micrometre.

1.2 Graphite, Dry shall be suitable for use in Pyrotechnic Compositions.

1.3 Graphite, Dry, Sulphur free shall be suitable for use in propellant manufacture, pelleting of Composition Exploding and initiatory compositions and for such other purposes where the presence of Sulphur in Graphite, Dry is not desirable.

2 RELATED SPECIFICATIONS/DOCUMENTS

2.1 Reference is made in this specification to:

<i>S No.</i>	<i>Specification No. & 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)	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 The material shall be of a good uniform, commercial quality (synthetic or natural), ground fine flaky powder. It shall be free from gritty particles, foreign matter, non-graphite carbon and visible impurities. It shall exhibit the characteristic soapy feeling when rubbed between fingers.

3.2 The size of the material may be one of these indicated below:

Size 150 Micrometre
Size 125 Micrometre
Size 90 Micrometre
Size 63 Micrometre
Size 45 Micrometre

3.3 The purchaser shall state in the contract the size of the material and the type i.e. Graphite, Dry or Graphite, Dry, Sulphur Free required by him.

4 MANUFACTURE

4.1 The material shall be manufactured by a process which will produce the product conforming to this specification.

5 TENDER SAMPLE

5.1 The manufacturer/contractor shall submit a tender sample of 250 g free of all charges and conforming to this specification.

5.2 If practical trial for end use is **CONSIDERED NECESSARY**, the manufacturer/contractor shall supply the quantity as required by the Quality Assurance Officer or Indentor (See Note below).

NOTE- A quantity of 500 g of sample shall be supplied free of cost to the consignee viz. ordnance factory to assess the suitability of material by a practical trial after the sample has passed all other clauses in this specification. Graphiting of a quantity of not less than 50 kg of propellant shall be done by ordnance Factory and the Graphite used shall not show any adverse effect on Heat Test of the propellant.

6 PRE-INSPECTION OF STORES/CONSIGNMENT

6.1 Manufacturers/contractors must satisfy themselves that the stores are in accordance with the terms of the 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 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 Graphite, Dry and Graphite, Dry, Sulphur free and packages in which it is contained shall be subject to inspection by and to the approval of the Quality Assurance Officer/Quality Assurance Authority.

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

7.2 Sampling

7.2.1 Normally two representative samples each of 250 g shall be drawn from each batch/lot/consignment of supply/manufacture. However the number of samples to be drawn shall be at the discretion of the Quality Assurance Officer/Quality Assurance Authority.

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 taken from any portion of the batch/lot/consignment of the material shall conform to clause 3.1 and in addition shall conform to the following test requirements:

Test Requirements of Graphite Dry and Graphite, Dry, Sulphur Free

<i>S No.</i>	<i>Characteristics</i>	<i>Passing Standard</i>		<i>Test Method</i>
		<i>Graphite Dry</i>	<i>Graphite Dry Sulphur Free</i>	
a)	Volatile Matter after drying for 2 h at 105°C ±2°C, % by mass	0.25 Max	0.25 Max	JSG 0112 Method 1 (a)
b)	PH of Aqueous Extract	8.0 Max 5.0 Min	8.0 Max 5.0 Min	JSG 0112 Method 5 (b)
c)	Matter soluble in cold water, % by mass	0.1 Max	0.1 Max	Appendix 'A'
d)	Matter soluble in Ethyl Ether, % by mass	0.25 Max	0.25 Max	Appendix 'B'
e)	Sulphur and compounds of Sulphur calculated as Sulphuric Acid, % by mass	Nil	0.25 Max	Appendix 'C'
f)	Ash, % by mass	4.0 Max	1.0 Max	Appendix 'D'
g)	Gritty particles in the ash (Ash after extraction with Aqua Regia) retained on 63 micrometre IS Sieve, % by mass	0.03 Max	0.03 Max	Appendix 'E'

JSS 9620-01 : 2016
(Revision No. 3)

S No.	Characteristics	Passing Standard		Test Method
		Graphite Dry	Graphite Dry Sulphur Free	
h)	Sieving requirements: % by mass			Appendix 'F'
	i) For size 150 micrometre Retained on 150 micrometre IS Sieve	Nil	Nil	& IS 460 (Part 1)
	ii) For size 125 micrometre Retained on 125 micrometre IS Sieve	Nil	Nil	
	iii) For size 90 micrometre Retained on 90 micrometre IS Sieve	Nil	Nil	
	iv) For size 63 micrometre Retained on 63 micrometre IS Sieve	Nil	Nil	
	v) For size 45 micrometre Retained on 45 micrometre IS Sieve	Nil	Nil	

8 WARRANTY

8.1 The stores supplied against the contract shall be deemed to bear a warranty against the defective material and performance by the contractor for a period of 12 months from the date of receipt of the stores at the consignee's end and shall retain the properties described above. If during this period any of the stores supplied is found defective, the same shall be replaced by the manufacturer/supplier/contractor free of charges at the consignee's premises.

9 PACKAGING

9.1 The graphite Dry, and graphite Dry Sulphur free shall be packed in polythene bag, having film thickness of 0.13 mm approximate and the bag placed inside any of the following:

- a) A double gunny bag and stitched without damage to the polythene bag and contents.
- or
- b) A wooden or plywood box lined with neutral packing paper all round the polythene bag.
- or
- c) A new clean dry rust free MS drum.
- or
- d) A fiber board drum or plywood drum.

9.2 The quantity per package shall be 25 kg or as per contract.

9.3 The packages shall be painted externally or unpainted as required by the terms of contract. Any paint used on the packages shall be of approved quality to the satisfaction of the Quality Assurance Officer/Quality Assurance Authority.

10 MARKING

10.1 All packages containing the material shall be indelible and legibly 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 the Consignment.
- h) Date of Supply.
- j) Manufacturer's Initials or Recognized 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 DEFFENCE STORES CATALOGUE NUMBER

11.1 The Deffence Stores Catalogue Number as applicable to this specification are as under.

<i>S No.</i>	<i>Nomenclature</i>	<i>DS Cat No.</i>
a)	Graphite Dry Size 45 micrometre	9620-000 004
b)	Graphite Dry Size 63 micrometre	9620-000 005
c)	Graphite Dry Size 90 micrometre	9620-000 006
d)	Graphite Dry Size 125 micrometre	9620-000-007
e)	Graphite Dry Size 150 micrometre	9620-000-008
f)	Graphite Dry Sulphur free Size 45 micrometre	9620-000 009
g)	Graphite Dry Sulphur free Size 63 micrometre	9620-000 010
h)	Graphite Dry Sulphur free Size 90 micrometre	9620-000 011
j)	Graphite Dry Sulphur free Size 125 micrometre	9620-000-012
k)	Graphite Dry Sulphur free Size 150 micrometre	9620-000-013

12 SUGGESTIONS FOR IMPROVEMENT

12.1 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

Appendix 'A'

A DETERMINATION OF MATTER SOLUBLE IN COLD WATER

A.1 Place 10 g of the sample accurately weighed in a stoppered glass cylinder, add 100 ml of boiled and cooled distilled water and shake for 1 h. Allow the solid matter to settle and decant off the supernatant liquid through a filter paper rejecting the first 10 ml. Transfer an aliquot portion of the filtrate (V ml) not less than 50 ml to a tared evaporator dish (W_1). Evaporate to dryness and heat to constant mass at 105°C to 110°C and weigh (W_2).

$$\begin{array}{l} \text{Matter soluble in cold water,} \\ \text{\% by mass} \end{array} = \frac{(W_2 - W_1) \times 100}{\text{mass of sample}} \times \frac{100}{V}$$

B DETERMINATION OF MATTER SOLUBLE IN ETHYL ETHER

B.1 Take about 5 g of the sample accurately weighed in a paper thimble and extract in soxhlet apparatus for 3 h with Ethyl Ether. Ten changes of solvent should occur each hour, otherwise extend the extraction time. Evaporate the extract in a tared (W_1) flask on a hot water bath and then dry to constant mass in a boiling water oven and weigh as (W_2). From the mass of the residue ($W_2 - W_1$), calculate the % of matter soluble in Ethyle Ether.

$$\begin{array}{l} \text{Matter soluble in Ethyl Ether,} \\ \text{\% by mass} \end{array} = \frac{(W_2 - W_1)}{\text{mass of sample}} \times 100$$

Appendix 'C'

C DETERMINATION OF SULPHUR AND COMPOUNDS OF SULPHUR

C.1 Weigh accurately 0.5 g of the sample and transfer into a 400 ml tall Pyrex beaker. Add 15 ml of concentrated Nitric Acid and Wet the material with the same. Add in small quantities at a time about 2 g of Potassium Chlorate. Warm very gently until all the Potassium Chlorate is dissolved. Place the beaker on a sand bath. Start heating gradually on a Bunsen burner. Add 1 g of Potassium chlorate at an interval of every 10 minutes till all the material is oxidized, i.e. clear solution is obtained. Ensure that in the beaker the quantity of Nitric Acid is replenished in order that the contents of the beaker do not become completely dry during the evaporation. After complete oxidation, evaporate contents to near dryness. Add 20 ml of Concentrated Hydrochloric Acid. Evaporate to near dryness. Repeat the addition of Hydrochloric acid and evaporate to dryness to remove the last traces of Nitric acid completely.

C.2 Add 50 ml of dilute Hydrochloric acid (1:2). Warm a little and then filter the solution in a beaker and make up the volume of the filtrate to about 500 ml then heat it to boiling. To the boiling solution add slowly 10 ml of 10% Barium chloride solution, with stirring and digest on sand bath for 30 minutes. Allow to stand over night. Filter the precipitate through No. 42 Whatman filter paper. Wash the precipitate with hot water till all chlorides are removed. Test the filtrate for freedom from chlorides when the filtrate is free from chlorides, dry the precipitate in a water-oven and then ignite it in a tared silica crucible (W_1) in a muffle furnace and weigh after cooling (W_2). Carry out a blank using same quantity of the reagents under identical conditions. Deduct the quantity of the precipitate in the blank. The excess obtained is the quantity representing sulphur content of the sample to be calculated as sulphuric acid.

Calculation

$$\begin{array}{l} \text{Percentage of Sulphur and} \\ \text{Compounds of Sulphur} \\ \text{as H}_2\text{SO}_4 \end{array} = \frac{(\text{W}_2 - \text{W}_1) \times 0.4202}{\text{mass of sample taken}} \times 100$$

D DETERMINATION OF ASH CONTENT

D.1 Heat Platinum dish/Porcelain crucible in the muffle furnace at 950°C for 15 minutes. Cool in a desiccator and weigh (W₁). Place the dish and its sample spread in a thin layer and weigh (W₂). Place the dish and its contents in the muffle furnace maintained at 500°C to 550°C. Heat for about one hour. Pass a gentle stream of air through the furnace sufficient to supply ample oxygen for combustion, but not at a rate to disturb the sample or the ash. Raise the temperature of the furnace to 750°C to 800°C and heat for one hour. When the carbon is completely removed, as indicated by the absence of black particles, raise the temperature to 950°C and heat it for one hour. Treatment of heating at 950°C to 1000 °C is repeated till constant mass is obtained (W₃).

$$\text{Ash content, \% by mass} = \frac{W_3 - W_1}{W_2 - W_1} \times 100$$

Deduct percentage volatile matter and moisture to obtain correct % of Ash content.

NOTE- If the muffle furnace is not ventilated, the ash/sample may be mixed with finely ground Ammonium nitrate and heated to the temperature. In this case a blank test using only Ammonium nitrate is to be carried out and necessary correction is to be applied.

Appendix 'E'

E DETERMINATION OF GRITTY PARTICLES IN THE ASH

E.1 Treat the ash obtained in Appendix 'D' with hot Agua Regia Decant carefully, wash with distilled water and then treat with 40% Sodium hydroxide solution. Allow the gritty matter to settle, decant and wash with successive portions of distilled water. Filter on a No. 40 Whatman paper. Wash with distilled water, dry in a oven at 100°C to 105°C, ignite gently in a silica/porcelain crucible and cool in a desiccator to room temperature. Brush the ignited gritty matter successively on 63 micrometre IS Sieve using a small camel hair brush and weigh the portion retained. Express as percent of the original sample, gritty matter retained on 63 micrometre IS Sieve.

F DETERMINATION OF SIEVING REQUIREMENTS

F.1 Place 10 g of sample weighed accurately to the second place of decimal on the test sieve. Gently immerse the sieve to a trough of pure kerosene taking care to avoid overflowing and spillage of sample. Raise the sieve and dip gently as above. Repeat the same two or three times. Take out the sieve gently and introduce into another trough of pure kerosene and repeat the sieving process as before. Take out this and ensure not more than one or two drops of kerosene drips before introducing in to a trough of pure Acetone, free from moisture. Gently dip and raise. Take out the sieve and keep it in a oven maintained at $60^{\circ}\text{C} \pm 2^{\circ}\text{C}$ for half an hour. After all vapours of Acetone are removed, cool it to ambient temperature and gently remove the retained matter using a camel hairbrush. Express the mass of the material retained on the sieve as a percentage on the original sample.