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GUN POWDER

FOR

155 MM AMMUNITION

(DS Cat No. 1376 – 000 093)

MASTER COPY

6/3/18

सत्यमेव जयते

गुणता से विश्वास

CONTROLLERATE OF QUALITY ASSURANCE (MILITARY EXPLOSIVES)

AUNDH ROAD, PUNE - 411 020

DEPARTMENT OF DEFENCE PRODUCTION

MINISTRY OF DEFENCE

MASTER COPY

GUN POWDER

FOR

155 MM AMMUNITION

AMENDMENT RECORD

Amendment		Authority letter	Clauses Affected	Remarks
D.C. No.	DATE			

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THIS SPECIFICATION OR ANY OTHER PATTERN, DRAWINGS OR ANY OTHER INFORMATION ISSUED IN CONNECTION THEREWITH MAY ONLY BE USED FOR A SPECIFIC ORDER PLACED BY THE COMPETENT AUTHORITY. IT IS NOT TO BE USED FOR ANY OTHER PURPOSE WHATSOEVER WITHOUT THE EXPRESS WRITTEN SANCTION OF THE DIRECTOR GENERAL OF QUALITY ASSURANCE, MINISTRY OF DEFENCE, NEW DELHI - 110 011.

0. FOREWORD

0.1 This specification has been prepared by the Controllerate of Quality Assurance (Military Explosives) Aundh Road, Pune - 411 020.

0.2 This specification is a revision of IND/ME/974(Prov) and supersedes the same.

0.3 For additional copies or any other enquiry regarding this specification, reference should be made to the Quality Assurance Authority i.e. CQA (ME) Aundh Road, Pune - 411 020.

1. SCOPE

1.1 This specification is meant to govern manufacture, supply and Quality Assurance of Gun Powder.

1.2 The material is suitable for use in 155 mm Ammunition.

2. RELATED SPECIFICATION AND DOCUMENTS

2.1 The related documents as mentioned in clause 2.2 are those applicable at the date of publication of this specification. It is manufacturer's/contractors responsibility to confirm their current applicability and to obtain from CQA (ME) Aundh Road, Pune - 411 020 information concerning any change that may be necessary due to cancellation, replacement or supersession of any of these documents.

2.2 The following related specifications have been referred to in preparation of this IND/ME specification.

i)	IS 301 : 1982, AMD-1 Reaffirmed 2011	---	Potassium Nitrate for explosive and pyrotechnic compositions
ii)	IS 138 : 1992, AMD-1 Reaffirmed 2009	---	Ready Mixed Paint, Marking for Packages and Petrol Containers
iii)	IS 460 (Part-1) : 1985, AMD-1, Reaffirmed 2008	---	Test Sieves : Part 1, wire cloth test sieves
iv)	JSG 0112 : 2015 (Rev No. 2)	---	General Methods of tests and assessment of impurities in chemicals / materials used in the manufacture of Explosives and Ammunition
v)	JSS 9110-144:2013 (Rev No. 1)	---	Charcoal Type I, II & III
vi)	JSS 6810-141:2010 (Rev No. 1)	---	Sulphur Grade II

2.3 The copies of this specification and of related specifications are obtainable on payment basis as follows :-

SPECIFICATION		SOURCE OF SUPPLY
(i) IND/ME/ Specification	:	C. Q. A. (ME), AUNDH ROAD, PUNE - 411 020
(ii) JSS / JSG	:	The Director Directorate of Standardization Standardization Documents Centre Ministry of Defence Room no 05, 'J' Block Nirman Bhawan PO New Delhi – 110 011
(iii) IS Specification	:	Bureau of Indian Standards, Manak Bhawan 9, Bahadur Shah Zafar Marg, NEW DELHI – 110 002 or Their regional / Branch offices

3. MATERIAL

3.1 The Gun Powder shall consist essentially of an intimate mixture of Potassium Nitrate, charcoal and sulphur in the proportion shown in clause 6.3.1 and shall be free from foreign matter, grit & visible impurities. The Gun Powder shall be in the form of brilliant homogenous black granules.

4. MANUFACTURE

4.1 The Gun Powder shall be manufactured by a process which has received authoritative approval. The Quality Assurance Officer shall be informed regarding the process used and shall be informed with prior notification any proposed deviation there form. All the deviations, however slight, shall be recorded immediately and all the material affected shall be set aside pending the decision of QA Officer/QA Authority.

4.2 All precautionary measures for handling of the store shall be strictly complied with.

5. TENDER SAMPLE

5.1 The Contractor/Manufacturer shall submit two tender samples of 250 g each essentially from the same batch/lot of manufacture, free of all charges & conforming to this specification to Quality Assurance Authority.

6. QUALITY ASSURANCE

6.1 INSPECTION

6.1.1. The material and the packages in which it is packed shall be subject to inspection by & to the approval of QA Officer/QA Authority.

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

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

6.1.4 The foregoing provisions shall equally apply to the prime contractor and sub contractors, if any.

6. 2. SAMPLING

6.2.1 A representative sample of 250 g each shall be drawn from batch/lot of manufacture. However, the number of samples to be drawn shall be at the discretion of QA Officer/QA Authority.

6.3 TEST REQUIREMENTS

6.3.1 The samples taken from any portion of the batch/lot of the material shall conform to clause 3.1 above and in addition shall conform to the following test requirements.

Sl. No.	Test	Passing Standard	Test Method
1.	Visual examination	Uniform brilliant homogenous black granules free from visible impurities & foreign matter	
2.	Moisture content Percent by mass Min Max	 0.90 1.40	Appendix 'A'
3.	Chlorides as KCl Percent by mass Max	 0.05	Appendix 'B'
4.	Sulphates as K_2SO_4 Percent by mass Max	 0. 10	Appendix 'C'
5.	Total Chlorine as $KClO_4$ Percent by mass Max	 0. 40	Appendix 'D'
6.	Sodium Compounds Calculated as Sodium Percent by mass Max	 0.08	Appendix 'E'
7.	Acidity as H_2SO_4 Percent by mass Max	 0.004	Appendix 'F'

Sl. No.	Test	Passing Standard	Test Method
8.	Mass of residue on Flashing Percent by mass Max	8.0	Appendix 'G'
9.	Lead fuze burning test rate in seconds/ metre Min Max	 93 99	Appendix 'H'
10.	Hygroscopicity Percent by mass Max	3.0	Appendix 'J'
11.	Absolute density g/CC Min	1.75	Appendix 'K'
12.	Composition (Percent by mass) a) KNO_3 Min Max	 74.0 77.0	Appendix 'L'
	b) Charcoal Min Max	 13. 5 15. 5	
	c) Sulphur Min Max	 9.0 11.0	
13.	Sieving (Percent by mass)		JSG 0112:1997 Method No. 18
	a) Passing through 2.0 mm IS Sieve	All shall pass	
	b) Passing through 2.0 mm & retained on 600 micrometer IS Sieve Min Max	 40.0 50.0	
	c) Passing 600 micrometre IS sieve & retained on 300 micrometre IS Sieve Min Max	 55.0 60.0	
	d) Passing 300 micrometre IS Sieve Min Max	 2.0 3.0	
	e) Passing 150 micrometre IS Sieve Max	0.50	

7. SUPPLIERS INSPECTION OF STORES/CONSIGNMENT

7.1 Before tendering the store for inspection, the supplier shall carry out a thorough inspection of each delivery to satisfy himself that the store fully conforms to this specification and shall render a certificate to that effect to the QA Officer/QA Authority.

8. WARRANTY

8.1 The store supplied against the contract shall deem to have been warranted against defective material and performance by the manufacturer/contractor for a period of 12 months from the date of receipt of the store at the consignee's end and if during this period any of the store supplied is found defective, the same shall be replaced by the manufacturer/contractor free of charge at the consignee's premises.

9. PACKAGING

9.1 The Gun Powder shall he packed in a suitable polythene bag of film thickness 0.13 mm minimum & bunch tied. It shall then be placed in a chemically neutral calico bag which shall also be bunch tied with tape cotton unproofed of suitable width. The calico bag shall then be placed in a suitable wooden case & the lid shall be fixed with brass screws. The quantity shall be 25 Kg or as agreed to between the supplier & purchaser.

9.2 The material for polythene bags shall conform to JSS 9330-03 & material for calico bag shall conform to IS 5088 part I (latest issue)

9.3 Any other form of the packages shall have the prior approval of QA Officer/QA Authority.

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

10. MARKING

10.1 All the packages containing the material shall be legibly and durably marked with the following details :-

i) Nomenclature and specification number of the material.

* ii) Name and address of the consignee.

* iii) A/T No. or S. O. No. & date.

iv) Consignment Number

v) Lot/Batch No. & Date of manufacture

vi) Gross & Net Mass.

vii) Consecutive no of packages and total no of packages in the consignment

viii) Date of Supply.

*ix) Contractor's initials or recognised trade mark.

* Not applicable when the store is manufactured, in Ordnance Factories.

10.2 In addition to above the OA Officer/OA Authority may suggest some more marking/Identification suitable at the time of inspection.

10.3 The paint used for marking shall conform to IS 138 (Latest Issue) and to the satisfaction of QA Officer/QA Authority.

10.4 Hazard division HD 1.1 marking of approved pattern shall be done on each container by means of a prescribed lable or applied by means stencil.

11. SAFETY OF OPERATIONS

11.1 Nothing in this specification shall relieve the manufacturer/Contractor/User of his responsibility for the safety of operations in manufacture, storage, transport or use of this store.

11.2 Safety certificate No. is applicable which is obtainable on application from CQA(ME) Aundh Road, Pune – 411 020.

12. DEFENCE STORES CATALOGUE NUMBER

12.1 The Defence stores catalogue number allotted to this store is 1376 – 000 093.

13. SUGGESTIONS FOR IMPROVEMENT

13.1 Any suggestion for improvement in this document shall be forwarded to The Controller, CQA(ME) Aundh Road, Pune – 411 020.

Date : 24·01·2017

(Mrs. MGP DHANRAJ)
CONTROLLER
CQA(ME), Aundh Road
Pune - 411 020

14. APPENDICES

APPENDIX 'A'

DETERMINATION OF MOISTURE CONTENT

Transfer about 10 g of the sample, rapidly to a tared, squat aluminium or glass dish (M_1) with a close fitting lid & weigh quickly (M_2). Remove the lid and expose the powder in an oven at 100 degree C plus minus 2 deg C for two hours. Close the dish, transfer it in to a dessicator to cool, & weigh again (M_3).

$$\text{Moisture Content Percent by mass} = \frac{M_2 - M_3}{M_2 - M_1} \times 100$$

II Destroy the gun powder used by treating with water.

APPENDIX 'B'

DETERMINATION OF CHLORIDES

Extract 5 g of the sample with distilled water & determine chlorides in the filtered extract by Volhard's Method using 0.01N silver nitrate and 0.01N ammonium thiocyanate. Carry out a blank.

$$\text{Chloride Content Percent by mass} = \frac{\text{Difference in titre x Factor of } NH_4 CNS \text{ x } 0.0746}{\text{Mass of Sample taken}}$$

ALTERNATIVELY

Chlorides can be estimated in the Chloride Titrator

Instrument :

Standard Analytic Chloride Titrator

Reagents:

Use chloride free distilled water for all solutions & rinsing the electrodes.

1) **Acid Reagent** : Add 100 ml of glacial acetic acid 7.1 ml of 60% nitric acid (Mass/Mass) to 500 ml water mix thoroughly and dilute to 1 litre.

2) **Gelatin reagent** : Dissolve 6.2 g of gelatin reagent (mixture of gelatin, thymol blue % thymol in proporation of 60:1:1 by mass) in little hot water dilute to 1 litre.

PROCEDURE

Weigh about 5 g of sample accurately dissolve in distilled water & dilute to 100 ml in a standard volumetric flask. Take 1 ml of this solution in a titration viol, add 4 ml of acid reagent and 0.2 ml of gelation reagent & titrate according to the instructions of the instrument. Record timer reading (R_2) Similarly titrate 0.1 ml of about 0.9% (C_1) sodium chloride (A.R. and record reading (R_1). Titrate blankwith 4 ml acid reagent and 0.2 ml gelatin reagent & record reading (b). Calculate % of Chlorides as follows :-

$$\text{Chlorides as KCl Percent by mass} = \frac{C_1 (R_2 - b) \times 10 \times 1.276}{(R_1 - b) \times \text{Mass of Sample}}$$

APPENDIX 'C'

DETERMINATION OF SULPHATES

Treat 5 g of the sample in a beaker with 100 ml distilled water at 30 to 40 degree centigrade & maintain at this temperature for 15 minutes with occasional strirring. Decant through a No.42 Whatman filter paper & wash the residue in the beaker with water twice, decanting each time as before. Make up the solution to 20 ml add 5 ml concentrated hydrochloric acid, heat to boiling, add 15 ml boiling 10% solution of Barium Chloride solution and digest for 15 minutes or keep aside overnight. Estimate the sulphates in the normal gravimetric manner & calculate as potassium sulphate percentage on the sample. Carry out a blank.

NOTE : A considerable amount of Barium Chloride is necessary to precipitate sulphates completely in the presence of much nitrate. The amounts quoted above are suitable only if the sulphates content does not exceed 0.1% by mass on the sample.

$$\text{Sulphates as } K_2SO_4 \text{ Percent by mass} = \frac{\text{Corrected mass of precipitate} \times 74.67}{\text{Mass of Sample taken}}$$

APPENDIX 'D'

DETERMINATION OF TOTAL CHLORINE

Extract 10 g of the sample with water. Dry the filtered extract, pulverise and transfer to a hard glass test tube. Insert the tube in a hole in a piece of asbestos sheet clamped in an inclined position. Close the tube with a hung carrying a delivery tube fitted to a Utube filled with water to form a seal. Heat the tube gradually until the active evaluties of was ceases, disconnect the tube & allow to cool. Extract the contents with water, into a 300 ml bottle, acidify with 10 ml Nitric acid, Add ether to form 10 ml layer on the surface. Add 10 ml of 0.01N silver nitrate dropwise with constant stirring. Shake well to coagulate the precipitate, add 1 ml of saturated ferric alum solution and titrate the excess silver nitrate with 0.01N ammonium thiocyanate, till a brown colouration appears in the aqueous layer. Carry out a blank.

The difference in the titres denotes the silver nitrate used by the chlorides and perchlorate together. Make allowance for the chlorides determined as in appendix 'C' & calculate the percentage of perchlorate in the sample as $KClO_4$.

1 ml of 0.01N NH_4 CNS = 0.001 39 g $KClO_4$

NOTE : If the total chlorine expressed as $KClO_4$ is less then 0.1% (i.e. if the titre difference is less than 8 ml there is no need to determine the chloride content separately.

APPENDIX 'E'

DETERMINATION OF SODIUM COMPOUNDS

Treat 5 g of the sample with 20 ml distilled water & allow to stand, with occasional stirring for 15 minutes Test the extract for sodium on a clean platinium wire over a bunsen flame. If no yellow colour is observed, record the sodium content as less than 0.03%. If a yellow colour is produced determine the sodium content by flame photometer.

APPENDIX 'F'

DETERMINATION OF ACIDITY

Treat 5 g of the sample with 20 ml of freshly boiled & cooled distilled water which is neutral to congo red paper. Decant the solution through a filter paper & examine for acidity in comparison with a solution of 40 parts per million of sulphuric acid. Test by spotting the liquids upon cango red paper of the grade sensitive to 40 ppm sulphuric acid. Estimate the acidity of the extract by comparison of the spots. Report the acidity of the sample to congo red paper as equal to, less than or greater than the standard acid, as the case may be.

APPENDIX 'G'

DETERMINATION OF RESIDUE ON FLASHING

Weigh 5 g of the sample, prepared as above, if necessary and distribute it evenly over the bottom of a tared, flat bottom porcelain dish, 100 mm diameter and 19 mm deep. Place the dish out in the open, but free from draught, or in a fume cupboard. Ignite the sample using a strand of thin cordite about 100 mm long. Observe the flash for excessive sparks. Cover the dish with a glass plate, allow to cool to room temperature and weigh. Examine the residue.

The sample shall burn with very few sparks, and the residue left shall be very finely divided and free from sparks of fused salt or carbon. It shall not weigh more than 0.4 g.

$$\text{Residue on Flashing Percent by mass} = \frac{\text{mass of residue}}{\text{mass of sample taken}} \times 100$$

NOTE : The cordite used for igniting the sample shall be free from mineral additives, and shall not contain picrite or anything that leaves excessive residue on burning

APPENDIX 'H'

LEAD FUZE BURNING TEST

The test consist of filling the sample into a lead tube and determining the rate of burning. Corrections are applied to the observed burning time for variation in temperature, barometric pressure and the moisture content of the sample.

The details of the test are obtainable from CQA(ME), Aundh Road, Pune - 411 020.

APPENDIX 'J'

DETERMINATION OF HYGROSCOPICITY

Weigh accurately about 65 g of the sample in a tared, flat dish with lid. Expose the above sample to the constant humidity condition obtained by saturated solution of potassium nitrate in contact with an excess of the solute. The temp shall be maintained at 13 to 18 degre c. After 24 hours exposure as described above, close the dish and weigh.

$$\text{Hygroscopicity, Percent by mass} = \frac{\text{Gain in mass} \times 100}{\text{Mass of the sample taken}} + \text{Moisture}$$

Note :- The period of conditioning shall be 24 hours for all grades of gun powders except pebble type in which case the period shall be 48 hours.

APPENDIX 'K'

DETERMINATION OF ABSOLUTE DENSITY

The absolute density of the gun powder is determined by means of the Bianchi densimeter, in which the volume of a known mass of the sample is determined by displacement of mercury. The air between the grains being removed by evacuating to low pressure. An efficient vacuum is required and it is necessary to insert a menometer between the pump and the apparatus to indicate the degree of evacuation.

PROCEDURE

Close and evacuate the apparatus, Ensure, by the memometer, that the apparatus is free from leaks.

Weigh accurately about 100 g of the sample and transfer, it through a funnel into the dry, empty globe of the densimeter Close the globe by screwing on the top cap and tighten by means of spanners.

Adjust the temperature of the mercury in the wooden bowl to either 15 degree C or 21 degree C, by stirring it with a test tube, containing cold water. Fit the saucer-shaped washer to the top cap of the globe and screw the globe loosely to the base of the capillary tube. Screw the nozzle, which projects into the mercury, to the bottom cap of the globe and tighten by spanners. Then screw the globe tightly to the base of the capillary tube.

Close the bottom tap of the globe and open the top tap. Evacuate until the pressure in the tube is 12.5 mm of mercury, i. e if an open manometer is used, its reading differs from the actual barometric height by not more than 12.5 mm. Open the tap the globe, almost to the top of the capillary. Renew the pumping, with the tap open until the manometer is steady at the height previously obtained.

Close the bottom tap of the globe and open the air inlet tap. The level of mercury in the capillary will fall slightly. Close the tap of the globe and partly unscrew the globe from the base of the capillary tube. Unscrew and remove the globe from the apparatus.

Using a steel probe and stiff Hair brush remove all mercury from the inlet and outlet passage at the bottom and top of the globe and from the screw threads of the caps. Place the globe on the stand of the pan of the special balance and weigh accurately (to the nearest 0.5 g) M_1.

Repeat the whole procedure, without the sample and obtain the mass of the globe when full of mercury (M_2).

Density of the sample =

$$\frac{\text{Mass of the sample taken x Relative density of mercury at temp. of test}}{(M_2 + \text{mass of sample taken}) - M_1}$$

NOTES

1) Wash leather gloves shall be used throughout the test.

2) After completion of the test, open the top tap of the globe hold the bottom tap over the mercury bowl & then open it so as to empty the mercury from the globe as completely as possible. Shake the globe to dislodge adherent mercury, close the taps and unscrew and remove the top tap of the globe. Empty the gun powder into the dish, remove the top tap of the globe. Empty the gun powder into the dish, remove all easily recoverable mercury, and the remainder after treating with water to destory the powder.

3) For convenience, tables are available in which the density of the sample is read directly from the difference between M_1 & M_2, for tests either at 15 degree C or 21 degree C.

4) The mercury used shall be clean, and its density shall be determined at the temperature of the test, i.e. either 15 degree C or 21 degree C.

ALTERNATIVE METHOD

The absolute Density may be determined by the modified Bianchi method using the glass apparatus as shown in Fig DR51/A.

Before starting the test, ensure that all ground joints of the apparatus have been well greased. Connect the upper end of the tube to the vacuum pump and draw pure mercury into the tube. Close the ground glass stoppers. Remove the mercury that may adhere to the outside of the apparatus and weigh (M_1).

Let out the mercury by opening the stopper, weigh out about 20 g of the sample accurately, and place it in the apparatus. Connect the apparatus to the vacuum pump and draw in mercury until no air bubbles remain. Close the stoppers, remove the mercury that may adhere to the outside of the apparatus and weigh again (M_2) Take the temperature of the mercury and calculate the absolute density as follows :-

Absolute Density of the sample at the observed temperature:-

$$\frac{M \times d}{M + (M_1 - M_2)}$$

Where,

M = Mass of sample

D = Relative density of mercury at the temperature of the test.

APPENDIX 'L'

DETERMINATION OF COMPOSITION

a) Extraction of Sulphur

Transfer about 5 g of the sample to a tared, G3 sintered glass crucible (M_1) & weigh accurately (M_2). Place the crucible firmly on Buchner flask. Add 25 ml of carbon disulphide. Allow to soak for a minute and draw the liquid out by applying gently suction. Repeat with three other 20 ml portions of carbon disulphide. Dry gently in an over at 100 degree C. Cool the crucible in a desiccator and weigh accurately (M_3).

Sulphur content in the sample, (Percent by mass) =

$$\left[\left(\frac{M_2 - M_3}{M_2 - M_1} \times 100\right) - \%\ \text{Moisture}\right] \times \frac{100}{100 - \text{Moisture}\ \%}$$

b) Charcoal Content

Replace the crucible in the Buchner flask & add about 25 ml hot water. Allow to soak for a minute and apply gently suction. Repeat the extraction with five more portions of hot water. Finally suck dry and transfer to an oven. Dry to constant mass. Cool in a desiccator and weigh (M_4).

$$\text{Charcoal content, Percent by mass} = \frac{M_4 - M_1}{M_2 - M_1} \times 100$$

c) Potassium Nitrate Content

Weigh accurately about 5 g of the sample into a 250 ml beaker (A), and 20 ml of hot water and stir well. Keep the beaker on a hot water bath until the insoluble matter has settled and decant through a Whatman No. 42 filter paper, into a second beaker (B) Perform this extraction four times using 20 ml of hot water on each occasion. Replace beaker B by another beaker (C) after the second extraction. This is necessary as later washing will be cloudy owing to the passage of charcoal through the filter. Finally transfer the whole of the contents of beaker A to the filter by washing and wash the filter with small amounts of hot water until the washings are free from potassium nitrate. Check freedom from nitrate by placing a drop of the filtrate on a white tile and spotting with concentrated sulphuric acid containing 1% diphenylamine. A blue colour indicates the presence of nitrate. Place beaker B on a Boiling water bath and evaporate to small bulk. Meanwhile evaporate the contents of beaker C in a tared silicon or porcelain dish and ignite gently over a small flame until all charcoal has been burned off. When cool moisten the residue with nitric acid and evaporate again. Finally add the contents of beaker B and evaporate the whole mass to dryness. Dry to constant mass by heating in an over at 100 degree C. Retain the residue for the estimation of perchlorates.

$$\text{Potassium Nitrate Percent by mass} = \frac{\text{Mass of residue} \times 100}{\text{Mass of sample taken}} \times \frac{100}{100 - \%\ \text{Moisture}}$$

NOTE : It total chlorine content has to be determined, the operations described above must be performed in an atmosphere free from chlorine or chlorine compounds, in order to avoid contamination of the residue.

Alternatively, the potassium nitrate content can be determined doducting the sum of the sulphur and carbon content from 100.

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