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मानक

IS 13522 (1992): Charcoal for Explosives and Pyrotechnic Compositions [CHD 26: Explosives and Pyrotechnics]



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Indian Standard

CHARCOAL FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

UDC 662.71 : 662.1/.4

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BUREAU OF INDIAN STANDARDS MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG NEW DELHI 110002

Price Group 3

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Explosives and Pyrotechnics Sectional Committee had been approved by the Chemical Division Council.

Charcoal is an important raw material used extensively in explosives and pyrotechnic industry. As there is no specification available on this subject, this Indian Standard has been prepared to meet the requirements of this industry and help them procure standard material.

In the preparation of this standard, assistance has been derived from JSS 1058 : 1968, issued by the Department of Standardization, Ministry of Defence, Government of India, New Delhi.

For the purpose of deciding whether a particular requirement of this standard is complied with, the final value, observed or calculated, expressing the result of a test or analysis, shall be rounded off in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

AMENDMENT NO. 2 JUNE 2005 TO IS 13522 : 1992 CHARCOAL FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

(*Page* 1, *clause* **4.3**) — Insert the following subclause after **4.3**:

'4.4 For defence purpose there shall be three classes A, B, C under material of Grade II with the following requirements:

a) Total carbon content, percent by mass:

*Class A — 66 - 80

Class B — 76 - 86

Class C — 63.5 - 70

*Class A shall be of such quality that suit the end use.

b) Ash content, percent by mass (Max)

Class A and B — 3.0

Class C — 2.0

c) Sieve requirement may be included for Grade II material in place of particle size with the following requirements:

For all classes:

- a) Size 600 micrometre Retained on 600 micrometre IS Sieve — Nil
- b) Size 250 micrometre Retained on 250 micrometre IS Sieve — Nil
- c) Size 125 micrometre Retained on 125 micrometre IS Sieve — Nil'

(CHD 26)

Reprography Unit, BIS, New Delhi, India

AMENDMENT NO. 1 MARCH 2000 TO IS 13522 : 1992 CHARCOAL FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS — SPECIFICATION

[*Page 2, Table 1, Sl No.* (vii), *col 2*] — Substitute the following for the existing:

'Total carbon content, percent by mass'.

(CHD 26)

29 FEB 2000

Reprography Unit, BIS, New Delhi, India

Indian Standard

CHARCOAL FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS -SPECIFICATION

1 SCOPE

This standard prescribes requirements and methods of sampling and test for charcoal for use in explosives and pyrotechnic compositions.

2 REFERENCES

The Indian Standards listed below are necessary adjuncts to this standard:

IS	No.	Title

- 323 : 1959 Rectified spirit (*revised*)
- 1070 : 1992 Reagent grade water Specification (*third revision*)
- 1350 Methods of test for coal and coke
- (Part 1): 1984 Proximate analysis (second revision)
- (Part 4/Sec 1): Ultimate analysis, Section 1 1974 Determination of carbon and hydrogen (*first revision*)

3 GRADES

There shall be two grades of the material as follows:

- Grade I For use in explosives and pyrotechnic compositions for general purposes, and
- Grade II For use in explosives and pyrotechnic compositions for defence purposes.

4 REQUIREMENTS

4.1 Description

The material shall be prepared from suitable species of wood, for example, Sal, Salai (*Bosewellia Thurifera*), etc. It shall be uniformly charred throughout (a brown tint usually indicates incomplete carbonization) and except as permitted in Sl No. (ii), (iii) and (iv) under col 1 of Table 1, shall be tree from foreign matter.

4.2 Fineness

The material shall be supplied in the form of 'lump' or 'stick' and shall be such that the sticks are not greater than 7.62 cm in diameter. For

use in fuse head manufacture, the material shall be in the form of finely ground black powder of particle size as agreed to between the purchaser and the supplier, and free from gritty matter.

4.3 The material shall also comply with the requirements given in Table 1 when tested in accordance with the methods prescribed in Annex A. Reference to the relevant clauses of Annex A is given in col 4 of the Table 1.

5 PACKING AND MARKING

5.1 Packing

The material shall be packed in clean bags or multiwall paper sacks tied at the mouth with tape, string or rope. Closure of the bags shall not be effected by means of wire or metal clips. The material may also be packed in heavy duty polyethylene sack, properly sealed and then inserted in closely knit gunny bag/woven high density polyethylene bag, sewn closely at the open ends. The bags shall be suitably stencilled.

5.2 Marking

Each package shall be legibly and indelibly marked with the following information:

- a) Name of the material,
- b) Net mass of the material,
- c) Indication of the source of manufacture,
- d) Month and year of manufacture, and
- e) Lot number or batch number, in code or otherwise.

5.2.1 The packages may also be marked with the Standard Mark of the Bureau.

5.3 Fire Hazard

The following cautionary note shall appear on each package in yellow colour in bold letters:

DANGEROUS, IGNITES SPONTANEOUS-LY WHEN FRESHLY CLAIMED AND EXPOSED TO HUMID ATMOSPHERE, HAZARDOUS WHEN FRESHLY GROUND AND TIGHTLY PACKED, FLAMMABLE SOLID. SHALL BE STORED INDOOR, IN A COOL AND DRY PLACE.

SI	Characteristic	Requireme	Requirement for		Method of Test, Ref to	
No.		Grade 1	Grade 2		IS No.	Annex
(1)	(2)	(3)	(4)		(5)	(6)
i)	Moisture, percent by mass, Max	6.0	—			A-3
ii)	Ash content, percent by mass, Max	8.0	3.0		—	A-4
iii)	Iron content, percent by mass, Max	0.025			—	A-5
iv)	Grit content, retained on 63 micrometre IS Sieve, percent by mass, <i>Max</i>	0.10	0.10			A-6
V)	Volatile matter, percent by mats, Max	10.0 to 18.0	5.0	7 of 13 1984	50 (Part 1):	
vi)	Fixed carbon content, percent by mass, Min	68	—			A-7
vii)	Total carbon content, percent by mass, <i>Min</i>	82-88	66-86	5 of 13 Sec 1	50 (Part 4/): 1974	—
viii)	Bulk density (packing), g/cc	0.35 to 0.45			_	A-8
ix)	Particle size*	To pass the test				A-9
x)	Acidity (as H ₂ SO ₂), percent by mass, <i>Max</i> :	Ĩ				
	a) to methyl orangeb) to phenolphthalein	—	Nil 0.1	}		A-10
xi)	Alkalinity (as Na ₂ CO ₂), percent by mass, <i>Max</i> :					
	a) to methyl orange		0.1)		. 10
	b) to phenolphthalein	—	0.01	}	_	A-10
xii)	Water soluble chlorides (as NaCl), percent by mass, <i>Max</i>	_	0.05	-	—	A-11

 Table 1 Requirements for Charcoal for Explosives and Pyrotechnic Compositions

 (Clauses 4.1 and 4.3)

The agreed to between the parenaser and the su

6 SAMPLING

6.1 The representative samples of the material

shall be drawn and their conformity to the standard determined as prescribed in Annex B.

ANNEX A

(Clause 4.3)

METHODS OF TEST FOR CHARCOAL FOR EXPLOSIVES AND PYROTECHNIC COMPOSITIONS

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (see IS 1070: 1992) shall be used in tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

A-2 INSPECTION OF CONSIGNMENT

A-2.1 In order to reduce risk of fire, charcoal, as taken into stock, shall be practically in equili-

brium with atmosphere. The following inspection test shall be carried out:

With a thermometer having a range of 0-100°C and a stem length of at least 30 cm, note the outside temperature in the neighbourhood of the stock or railway van or other transport carrying the stock. Select a bag from the middle of one end, preferably one which is not in contact with the floor or sides, open it at the neck and gently push the thermometer into the charcoal until the

whole stem is immersed. Give the bag an occasional shake to ensure that the charcoal is in contact with the bulb of the thermometer. Allow the thermometer to remain in the charcoal until it acquires the temperature of the charcoal. Note this temperature. Repeat this determination with a bag from the other end and one from the middle of the stack. If any undue difference between the temperature of the charcoal and the outside temperature is found, repeat the determination on the bags in the neighbourhood of the offending bag and isolate all bags showing unduly high temperature. An unduly high temperature is taken to be the one which is more than 5°C higher than the outside temperature.

A-3 DETERMINATION OF MOISTURE CONTENT

A-3.1 Procedure

Weigh about 10 g of the prepared sample in a shallow, stoppered, weighed, and dried weighing bottle and heat for 90 minutes in a boiling water oven. Cool the bottle and its contents in a desiccator and weigh again. Repeat this procedure until constant weight, that is, until the difference between two successive weighings is less than 1 mg.

A-3.1.1 Calculation

Moisture contentma, percent

$$\frac{100 \times M}{M}$$

where

 M_1 = loss in mass, in g, of the sample upon drying; and

M = mass of sample taken for the test.

A-3.2 Other methods as described in IS 1350 (Part 1): 1984 as applicable to coal and coke, may also be used for the determination of the moisture content.

A-4 DETERMINATION OF ASH

by

A-4.1 Outline of the Method

The sample is heated until the carbon is completely burnt and the remaining residue attains a constant mass.

A-4.2 Apparatus

A-4.2.1 Muffle Furnace

Capable of giving red heat (about 800-900°C) and provided with proper ventilation system.

A-4.2.2 Dish

Of silica, porcelain or platinum, 10 to 15 mm deep and provided with a lid.

A-4.3 Procedure

A-4.3.1 Thoroughly mix for one minute the airdried, ground charcoal. Weigh accurately about 2 g of the sample in a clean, dry, empty and previously weighed dish. Distribute the material so that the thickness of the layer does not exceed 0.15 g/cm². Insert the partly uncovered dish into the muffle furnace at room temperature, raise the temperature to red hot and continue heating until the carbon is completely burnt.

A-4.3.2 Cover the dish with its lid if the ash is light and fluffy. Transfer the dish to a desiccator and allow it to cool. Weigh after the dish has been in the desiccator for 15 minutes. From the difference in mass, calculate the percentage of ash on the mass of the sample taken.

A-4.4 Calculation

on dry basis

Ash, percent by mass,

100
$$\frac{M_{\rm B}-M_{\rm I}}{M_{\rm I}}$$

where

 $M_3 =$ mass of dish with lid and ash,

=

 M_3 = mass of dish with lid, and

 M_1 = mass of dry sample.

A-5 DETERMINATION OF IRON

A-5.1 Outline of the Method

Ferric iron reacts with thiocyanate to give red coloured compound which remains in true solution. The intensity of the colour is compared with the standard concentration.

A-5.2 Reagents

A-5.2.1 Standard Solution of Ferric Iron

Dissolve 0.854 g of ferric ammonium sulphate (A. R. grade) in water, add 10 ml of concentrated hydrochloric acid and dilute to one litre. One millimetre of this solution will correspond to one milligram of ferric iron.

A-5.2.2 Thiocyanate Solution

Dissolve 20 g of ammonium or potassium thiocyanate in 100 ml of water.

A-5.3 Procedure

A-5.3.1 Transfer the ash obtained in A-4.2 to a small hard glass beaker, add 5 ml of concentra-ted hydrochloric acid, and heat gently for 5 minutes. Dilute the solution with 100 ml water, allow the residue, if any, to settle and decant the supernatant liquor through a What-man filter paper No. 41 into 1 litre standard flask. Repeat the washing and decanting three times. Dilute the solution to 1 litre with water, mix it well and determine the iron by taking a 50 ml aliquot.

IS 13522 : 1992

A-5.3.2 Place 50 ml of the above solution in a Nessler cylinder, add 5 ml of the thiocyanate solution and 2.4 ml of 4 N nitric or hydrochloric acid. Measure the intensity of the colour using a photoelectric colorimeter by determining the transmittance of the solution at or near 480 nm. Compare this with previously made calibration chart of standard iron solution and calculate the concentration.

A-6 DETERMINATION OF GRIT

A-6.1 Grit

It is the material insoluble in *aqua regia* and retained on 125 micron IS Sieve.

A-6.2 Apparatus

A-6.2.1 Container

Silica basin having a diameter of approximately 16 cm.

A-6.2.2 Sieve — 125 micron IS Sieve.

A-6.3 Procedure

A-6.3.1 Weigh 100 g of the sample into the silica basin and heat the sample over Bunsen burner or in Muffle furnace till all carbon has burnt away. Allow the basin and its contents to cool. Transfer the ash to the sieve and brush it with a soft brush. Transfer the material obtained on the sieve to a 150-ml beaker. Add 20 ml of *aqua regia* in the beaker and cover the liquid with a watch glass. Digest the content of the beaker over a small Bunsen flame until only siliceous matter remains. Dilute the liquid with water, and filter it through Whatman filter paper No. 41. Wash the residue free from acid, ignite it in a porcelain/silica crucible.

A-6.3.2 Cool the crucible and its contents, and transfer the residue in the crucible to a 125 micron IS Sieve. Brush it with a soft brush, and transfer the material retained on the sieve to a tared watch glass. Calculate the increase in weight of the watch glass to percent on the dry weight of sample taken and report the result as percent grit.

A-7 DETERMINATION OF FIXED CARBON

A-7.1 The fixed carbon content is determined by subtracting from 100 the sum of the percentages by mass of moisture, volatile matter and ash.

A-8 DETERMINATION OF BULK DENSITY

A-8.1 Apparatus

A-8.1.1 Assemble the apparatus as shown in Fig. 1. The base of the measuring cylinder A shall be ground flat and the empty measuring cylinder A together with the rubber bung shall weigh 250 ± 5 g. It shall be accurately calibrat-

ed to 250 ml with an error, if any, of less than one millilitre. The distance between zero and 220 ml graduation on the measuring cylinder Ashall be not less than 200 mm and not more than 240 mm. The distance between the flat-ground part of the base of measuring cylinder A and the rubber base pad B, when the measuring cylinder A raised to full height shall be 25 ± 2 mm.



All dimensions in millimetres.

FIG. 1 APPARATUS FOR DETERMINATION OF BULK DENSITY

A-8.1.2 Rubber Base-Pad

The rubber base-pad B shall have a shore hard-ness of 42 to 50.

A-8.1.3 *Balance*

Pans of the balance shall be at least 10 cm in diameter and the balance shall be sensitive to a mass less than 0.1 g.

A-8.2 Procedure

Sieve about 40 g of the material through 250micron IS Sieve on to a tared glaze paper and weigh it accurately. Slip the powder gently and smoothly into the measuring cylinder which should be held at 45° to the vertical, without knocking or squeezing. Assemble the apparatus as shown in Fig. 1. With the thumb and four fingers of one hand, gently grasp the upper part of the cylinder and within one second, lift it about 25 mm (taking care not to jerk the cylinder by knocking it against the upper stop) and let it drop. Note the volume after dropping it once. Continue lifting and dropping until 50 complete drops have been given to the cylinder. During this operation, give a gentle turn of about 10° in the clockwise direction to the cylinder after every two drops. As soon as 50 drops are completed, raise the cylinder to eye level and read the volume of the material.

A-8.3 Calculation

Bulk density, g/ml :

a) After one tap = $\frac{m}{V_1}$

b) After 50 taps =
$$\frac{m}{V_{MO}}$$

where

- m = mass, in g, of the material taken for the test;
- V_1 = volume, in ml, of the material after one tap; and
- V_{50} = volume, in ml, of the material after 50 taps.

A-9 DETERMINATION OF PARTICLE SIZE

A-9.1 The sieve size shall be according to the particle size as agreed to between the purchaser and the supplier.

A-9.2 Take 100 g of the sample and sieve through the required size sieves with gentle tapping. Determine the percentage of the material retained on the top sieve and the percentage of the material passing through the bottom sieve as oversize and undersize, respectively.

A-10 TEST FOR ACIDITY OR ALKALINITY

A 10.1 Reagents

A-10.1.1 Rectified Spirit — see IS 323 : 1959.

A-10.1.2 Phenolphthalein Indicator Solution

Dissolve 0.10 g of phenolphthalein in 100 ml of 60 percent rectified spirit.

A-10.1.3 Methyl Orange

Dissolve 0.01 g of methyl orange in 100 ml of water.

A-10.1.4 *Standard Hydrochloric Acid* — 0.10 N.

A-10.2 Procedure

Suspend 10 g of the material in 50 ml of rectified spirit and shake for half an hour. Filter the solution and make up the volume to 100 ml with rectified spirit. Take 25 ml of this solution and add 5 drops of phenolphthalein indicator, if alkalinity (red colouration) is observed, titrate with N/10 hydrochloric acid. If no alkalinity is observed, add 5 drops of meth' l orange indicator to another aliquot of the solution. If orange red colouration is observed, titrate against N/10 sodium hydroxide.

A-10.3 Calculations

Percent alkalinity as sodium carbonate $= \frac{TV \times 0.0053 \times 100 \times 4}{10} = TV \times 0.2120$ Percent acidity as sulphuric acid $= \frac{TV \times 0.0049 \times 100 \times 4}{10} = TV \times 0.1960$

where

TV = titre value.

A-11 DETERMINATION OF SOLUBLE CHLORIDES

A-11.1 Reagents

A-11.1.1 Standard Silver Nitrate Solution — 0.1 N approximately.

A-11.1.2 Concentrated Nitric Acid

A-11.1.3 Ferric Ammonium Sulphate Indicator — 10 percent (m/v).

A-11.1.4 *Standard Ammonium Thiocyanate Solution* — 0.1 N approximately.

A-11.1.5 Nitrobenzene

A-11.2 Procedure

Weigh accurately 2.0 g of the material and add about 50 ml of water. Shake for 15 to 20 minutes and filter. To the filtrate, add 10 ml of standard silver nitrate solution, 2 ml of concentrated nitric acid and 5 ml of nitrobenzene. Shake vigorously for one minute. Add 2 ml of ferric ammonium sulphate indicator solution and titrate excess silver nitrate with standard ammonium thiocyanate solution shaking well between successive additions to appearance of first persistent colour change.

A-11.3 Calculation

Chlorides (as NaCl),
percentby mass =
$$-\frac{5\cdot846 (10 N_1 - VN_1)}{M}$$

where

- N_1 = normality of standard silver nitrate solution;
- V = volume, in ml, of standard ammonium thiocyanate solution used in titration;
- N_2 = normality of standard ammonium thiocyanate solution; and
- M =mass, in g, of the material taken for the test.

ANNEX B

(Clause 6.1)

SAMPLING OF CHARCOAL FOR EXPLOSIVES AND PYROTECHNIC INDUSTRY

B-1 METHOD OF SAMPLING

According to the size of the lot, select the number of bags to be sampled, as given in the followine table.

roportion to	Minimum No.
e Sampled	of Bags to be
(Percent)	Sampled
20	2
10	4
7.5	6
5	15
4	25
3	40
	roportion to be Sampled (Percent) 20 10 7.5 5 4 3

Open each bag selected for sampling and remove several pieces of varying sizes to collect a weight of approximately 100 g. When all the bags have been sampled, mix the sample well and retain half portion as reference sample.

B-2 PREPARATION OF SAMPLES FOR TEST

The sample pieces are to be crushed with a brass hammer and sieved using sieve size as per the requirements of the tests.

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