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भारतीय मानक मसौदा

विस्फोटक उद्योग के लिए पेंटाएरीथ्रिटोल के लिए विशिष्टि
(पहला पुनरीक्षण)

Draft Indian Standard

SPECIFICATION FOR PENTAERYTHRITOL FOR
EXPLOSIVE INDUSTRY

(First Revision)

ICS 71.100.30

Explosives and Pyrotechnics Sectional Committee, CHD 26

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FOREWORD

(Formal Clause to be added later)

Indian Standard specification for pentaerythritol (IS 7619) covers the requirements of the material for use in the manufacture of paint coatings and printing inks. It does not cover the nitration grade of the material required by the explosive industry. This standard is intended to cover the material used for the production of pentaerythritol tetranitrate for use in explosive and pyrotechnic compositions.

It is advisable for the users to carry out a trial nitration test with the material after ensuring conformity to this specification to assess the desired quality of the end product.

This Indian Standard was originally published in 1984. This first revision has been taken up to update the ICS No and several other editorial changes in order to bring out the standard in the latest style and format of the Indian Standards.

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 : 2022 'Rules for rounding off numerical values (*second revision*)'. The number of significant places retained in the rounded off value should be the same as that of the specified value in this standard.

Draft Indian Standard

**SPECIFICATION FOR PENTAERYTHRITOL FOR EXPLOSIVE
 INDUSTRY**

(First Revision)

1 SCOPE

1.1 This standard prescribes requirements and the methods of sampling and test for pentaerythritol, nitration grade, for explosive industry.

2 REFERENCE

The standards listed below contain provisions which through reference in this text, constitute provisions of this standard. At the time of publication, the editions indicated were valid. All standards are subject to revision and parties to agreements based on this standard are encouraged to investigate the possibility of applying the most recent editions of the standards indicated below:

<i>IS No</i>	<i>Title</i>
IS 1070 : 2023	Reagent Grade Water Specification (<i>fourth revision</i>)
IS 4905 : 2015/ISO 24153 : 2009	Random sampling and randomization procedures (<i>first revision</i>)
IS 5762 : 1970	Methods for determination of melting point and melting range
IS 7619 : 1987	Specification for Pentaerythritol (<i>first revision</i>)

3 GRADES

3.1 The material shall be of two grades, namely, Grade 1 and Grade 2.

4 REQUIREMENTS

4.1 The material shall be in the form of white crystalline powder, free from impurities visible to the naked eye.

4.2 Flow Properties — The material shall flow without interruption from a glass funnel with a 5-mm bore and 60° angle.

4.3 Particle Size —The particle size of the material shall be as follows:

- a) Passing through 1 mm IS Sieve, percent by mass, *Max*: 100
- b) Passing through 212 micron IS Sieve, percent by mass, *Max*: 1

4.4 The material shall also comply with the requirements laid down in Table 1 when tested according to the methods prescribed in **Annex A** is given in column 5 of the table.

TABLE 1 REQUIREMENTS FOR PENTAERYTHRITOL FOR EXPLOSIVE INDUSTRY
(Clause 4.4)

SL NO.	CHARACTERISTIC	REQUIREMENT		METHOD OF TEST (REF TO CL NO. IN ANNEX A)
		Grade 1	Grade 2	
(1)	(2)	(3)	(4)	(5)
i)	Moisture, percent by mass, <i>Max</i>	0.1	0.1	A-3

ii)	Melting point, °C	252 to 260	249 to 255	A-4
iii)	Pentaerythritol (mono PE content), percent by mass, <i>Min</i>	99.2	97.5 to 98.5	A-5
iv)	Hydroxyl value, percent by mass, <i>Min</i>	49.5	49.0	A-6
v)	Di Penta content, percent by mass, <i>Max</i>	0.4	0.9 } 0.5 }	A-7
vi)	PE formals, percent by mass, <i>Max</i>	0.4		
vii)	Sulphated ash, percent by mass, <i>Max</i>	0.05	0.1	A-8
viii)	Water insoluble, percent by mass, <i>Max</i>	0.1	0.1	A-9
ix)	Chlorides (as Cl), percent by mass, <i>Max</i>	0.01	0.01	A-10
x)	Formic acid, percent by mass, <i>Max</i>	0.005	0.005	A-11
xi)	Iron (as Fe), percent by mass, <i>Max</i>	0.002	0.002	A-12

5 PACKING AND MARKING

5.1 Packing — The material shall be packed in polyethylene-lined jute or paper bags or drums as agreed to between the purchaser and the supplier.

5.2 Marking — Each container shall be securely closed and shall bear legibly and indelibly the following information:

- a) Name and grade of the material;
- b) Net mass;
- c) Month and year of manufacture;
- d) Name of the manufacturer and/or his trade – mark, if any; and
- e) Lot number in code or otherwise to enable the batch of manufacture to be traced from records.

5.2.1 BIS Certification Marking

The containers may also be marked with the Standard Mark. The use of the Standard Mark is governed by the provisions of The Bureau of Indian Standards Act, 2016 and the Rules and Regulations made thereunder. The details of the conditions under which the licence for use of the Standard Mark may be granted to manufacturers or producers, may be obtained from the Bureau of Indian Standards.

6 SAMPLING

6.1 Representative samples of the material shall be drawn and their conformity to the standard determined as prescribed in **Annex B**.

ANNEX A

METHODS OF TEST FOR PENTAERYTHRITOL FOR EXPLOSIVE INDUSTRY

(Clause 4.4)

A-1 QUALITY OF REAGENTS

A-1.1 Unless specified otherwise, pure chemicals and distilled water (*see* IS 1070) shall be employed in text.

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

A-2 PREPARATION OF SAMPLE

A-2.1 Weigh about 100 g of the material and dry for 30 minutes in an oven at a temperature of (110 ± 2) °C. Cool in a desiccator and weigh. Dry again and repeat until constant mass is attained.

A-3 DETERMINATION OF MOISTURE

A-3.1 Procedure — Weigh accurately about 1.0 g of the sample into a weighing bottle which has been dried, cooled in the desiccator and then weighed. Place the weighing bottle in the air oven for about 1 hour at (100 ± 1) °C. Remove the weighing bottle from the oven, cool in a desiccator to room temperature and weigh. Repeat this procedure. Keep the weighing bottle in the oven only for half an hour each time until the difference between two successive weighing is less than 1 mg.

A-3.2 Calculation

$$\text{Moisture content, percent by mass} = 100 \times \frac{M_1}{M}$$

where

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

M = mass in g of the sample taken for the test.

A-4 DETERMINATION OF MELTING POINT

A-4.1 Procedure — Carry out the determination of melting point according to the method prescribed in IS 5762.

A-5 DETERMINATION OF PENTAERYTHRITOL (MONO PE CONTENT)

A-5.1 Outline of the Method — Pentaerythritol reacts with benzaldehyde in the presence of strong acid to form dibenzylacetal.



The dibenzylacetal so formed is filtered off, washed and dried to constant mass. A correction is made for the slight solubility of dibenzylacetal under the conditions of the test. Dipentaerythritol and polypentaerythritols which may be present in the sample do not react.

A-5.2 Reagents**A-5.2.1 Concentrated Hydrochloric Acid**

A-5.2.2 Benzaldehyde Reagent — Mix 15 ml of benzaldehyde with 100 ml of anhydrous methanol. The benzaldehyde bottle is sealed immediately to avoid oxidation to benzoic acid.

A-5.2.3 Wash Solution — Mix methanol and water in 1 : 1 ratio and cool in ice.

A-5.3 Procedure — Weigh 0.5 g of the prepared sample into a dry 250 ml council flask. Add 5 ml of water. Heat it slightly to dissolve the sample. While hot, add 20 ml of the benzaldehyde followed by the addition of 12 ml of concentrated hydrochloric acid. Stopper the flask with a rubber cork and allow to stand for 30 minutes. Swirl the contents of the flask occasionally. Then keep the flask in ice for 1 hour with occasional swirling. Filter the solution through a dried and weighed G4 sintered crucible. Wash precipitate with 6 ml to 10 ml portions of ice cold wash

solution. Totally 40 ml of ice cold wash solution is used for complete transfer and washing of the precipitate. Dry the crucible with the precipitate at 100 °C and weigh to a constant mass.

A-5.4 Calculation

$$\text{Pentaerythritol (mono PE content), percent by mass} = \frac{(M_1 + 0.03) \times 43.59}{M_2}$$

where

M_1 = mass in g of the precipitate obtained, and

M_2 = mass in g of the material taken for the test.

NOTES

1— 0.03 is the correction taken for calculation as the precipitate is soluble in methanol/ water mixture at the cold temperature.

2— The benzaldehyde method of determination of purity of PE gives a slightly higher value as the PE formals also get precipitated along with the mono PE in the benzaldehyde complex. Hence, a correction has to be made for the PE formals by subtracting the value for PE formals obtained by the GLC method.

A-6 DETERMINATION OF HYDROXYL VALUE

A-6.1 Outline of a Method — The material is acetylated with acetic anhydride. The excess of anhydride is titrated back after hydration with caustic soda.

A-6.2 Reagents

A-6.2.1 Acetic Anhydride

A-6.2.2 Pyridine

A-6.2.3 Potassium Hydroxide

A-6.2.4 Sodium Hydroxide Solution — 1 N.

A-6.2.5 Acetic Anhydride — Pyridine Solution — 2.5 N in pyridine prepared as follows:

29.6 ml of acetic anhydride made up to 250 ml with pure pyridine is dried over potassium hydroxide for 16 hours. The mixture is agitated and kept in the dark in a glass-stoppered bottle. This reagent has a yellow colour, which tends to decrease with time. (If the colour turns darker, or if the reagent is older than one week, it should not be used).

A-6.2.6 Phenolphthalein Indicator Solution — 0.5 percent (*m/v*). Dissolve 0.5 g of phenolphthalein in 100 ml of rectified spirit and make it faintly pink by the addition of dilute ammonium hydroxide.

A-6.3 Procedure — Place 0.5 to 0.6 g of the prepared sample in the acetylation flasks and add 25 ml of acetic anhydride – pyridine solution with a burette. Place air-cooled condenser on top and heat the flask in the glycerine bath at 150°C for 30 minutes. (They should not be heated any longer, otherwise the results will be too low).

Remove the flasks from the bath, rinse the condenser with 30 ml to 50 ml of water and cool the flasks for 20 minutes under a jet of water.

When the temperature has reached 20°C, titrate the contents of the flasks with 1 N sodium hydroxide solution using phenolphthalein as indicator. The titrating solution shall be added at a rate of 15 to 20 drops per minute. The last 10 ml shall be added very slowly under good shaking. Carry out blank test on 25 ml of acetic anhydride- pyridine solution.

A-6.4 Calculation

$$\text{Hydroxyl value, percent by mass} = \frac{1.70 \times (V_1 - V_2) \times N}{M}$$

where

V_1 = volume in ml of standard sodium hydroxide solution used in the blank,

V_2 = volume in ml of standard sodium hydroxide solution used in the titration,

N = normality of sodium hydroxide solution, and

M = mass in g of the sample taken for the test.

A-7 DETERMINATION OF DIPENTA AND PE FORMALS

A-7.1 Principle — Finely ground pentaerythritol (PE) is treated with hexamethyl disilazane (HMDS) and trimethyl chlorosilane (TMCS). The volatile TMCS ethers of PE's are produced which are injected into gas-liquid chromatograph for separation. The chromatograms are used for finding the percentage of each component from the peak areas.

A-7.2 Preparation of Silyl Ethers

A-7.2.1 Method I — 20 mg of pentaerythritol is taken and 0.5 ml of pyridine is added and heated to 70°C in a water-bath with stirring. To this stirred solution, 0.5 ml of hexamethyl disilazane is added. The mixture is then heated for 15 minutes at 70°C with through stirring. The contents are then cooled to 30°C and 0.8 ml trimethyl chlorosilane is added. The contents are again heated with brisk stirring. The reaction is complete at the end of 3 hours at 70°C. The solution is allowed to cool and then centrifuged. The supernatant liquid is the volatile silyl ethers of PE's and other compounds. These ethers are quantitatively produced under the conditions mentioned above and they are ready for injecting into the gas-liquid chromatograph.

A-7.2.2 Method II — 20 ml of pentaerythritol is mixed with 30 mg of anhydrous sodium sulphate. To this mixture, 1.5 ml to HMDS is added and heated for 30 minutes at 70°C in a water-bath. There shall be continuous stirring. Hydrochloric acid gas is passed into the mixture for a minute and stirred briskly. The gas is generated either by heating concentrated sulphuric acid-hydrochloric acid mixture or by the addition 50 ml conical flask with a two holes rubber stopper, one carrying a small thistle funnel and the other a bent tube for hydrochloric acid gas delivery. The mixture is heated for 3 hours at 70 °C so that silylation is complete. The mixture is cooled and centrifuged. The TMS ethers are ready for gas chromatographic analysis.

A-7.3 Reagents

A-7.3.1 Pyridine — Dried over potassium hydroxide flakes.

A-7.3.2 Hexamethyl Disilazane

A-7.3.3 Trimethyl Chlore Silane

A-7.3.4 Sodium Sulphate — anhydrous.

A-7.3.5 Hydrochloric Acid — gas.

A-7.4 Column Details

Column	15 cm long, 3 mm OD stainless steel column
Coating agent	2 percent silicone
Detector	Flame ionization detector
Carrier gas	Nitrogen
Sample size	2 µl
Mode of operation	Programmed heating
Initial temperature, °C	100
Final temperature, °C	250
Initial time, minutes	3
Rate of heating, °C/min	8
Final time, minutes	8
Injection Point temperature, °C	300

Manifold temperature, °C	300
Attenuation for PE	X 32
For others	X 2
Range	X 100
Order of elusion	Solvent, mono-PE, di-PE, bisformal and tri-PE
Solvent used	Pyridine
Standard sample Solid support	Gas chrom – p 100/120 mesh

A-7.5 Determination of Calibration Factors

A-7.5.1 The standard samples are silylated as per procedure and the silyl ethers are injected into the GLC column as described in **A-7.4**. The peak areas are calculated. The area are divided by the known percent-ages and calibration factor determined as follows:

Area of the peak PE	<i>A</i>
Area of the peak di-PE	<i>B</i>
Area of the peak bisformal	<i>C</i>
Area of the peak tri-PE	<i>D</i>

A-7.5.2 Percentage in the Sample

PE	98/ <i>A</i>	(<i>A</i>)
Di-PE	1.0	
Bisformal	0.5	
Tri-PE	0.5	

A-7.5.3 Calibration factors are:

PE	98	(<i>A</i>)
Di-PE	$\frac{1.0}{B}$	(<i>B</i>)
Bisformal	$\frac{0.5}{C}$	(<i>C</i>)
Tri-PE	$\frac{0.5}{D}$	(<i>D</i>)

NOTE— Run the standard samples whenever a new set of sample is to be analyzed.

A-7.6 Determination of Percentage in the Sample of PE

A-7.6.1 The sample is silylated as per procedure described. The silyl ether is injected into the column as described in **A-7.4**. The peak areas are calculated and the percentages determined as follows:

Peak area of PE	<i>E</i>
Peak area of di-PE	<i>F</i>

Peak area of bisformal	G
Peak are of tri-PE	H
Mass unit of PE	$E \times A' = E'$
Mass unite of di-PE	$F \times B' = F'$
Mass unit of bisformal	$G \times C' = G'$
Mass unit of tri-PE	$H \times D' = H'$
Let $E' + F' + G' + H'$	K
PE, percent by mass	$\frac{(E')}{(K)} \times 100$
Di-PE, percent by mass	$\frac{F'}{K} \times 100$
Bisformal, percent by mass	$\frac{G'}{K} \times 100$
Tri-PE, percent by mass	$\frac{H'}{K} \times 100$

A-8 DETERMINATION OF SULPHATED ASH

A-8.1 Procedure — Weigh accurately about 3 g of the material and cautiously evaporate in a tared porcelain crucible of 45 mm diameter and 40 mm high, over a gentle flame. Calcine the residue by adding a drop of concentrated sulphuric acid for 15 minutes at 800 to 1 000 °C and weigh the crucible till constant mass in obtained.

A-8.2 Calculation

$$\text{Sulphated ash, present by mass} = \frac{M_1}{M} \times 100$$

where

M_1 = mass in g of the residue obtained after ignition, and

M = mass in g of the material taken for the test.

A-9 DETERMINATION OF WATER INSOLUBLES

A-9.1 Procedure — Weigh 10 g of dry pentaerythritol and dissolve in 100 ml of warm water. Filter through a tared G 4 sintered glass filter funnel. Wash with water, dry for 2 hours at 70 °C and weigh to a constant mass.

A-9.2 Calculation

$$\text{Water insoluble residue, percent by mass} = \frac{M_1 - M_2}{M} \times 100$$

where

M_1 = mass in g of filter funnel after the filtration,

M_2 = tare in g of filter funnel, and

M = mass in g of the material taken for the test.

A-10 DERERMINATION OF CHLORIDE

A-10.1 Principle — Chloride is completely precipitated as silver chloride by the addition of an excess of standard silver nitrate solution and the excess is back titrated with ammonium sulphocyanide solution

A-1.2 Reagents**A-10.2.1** *Silver Nitrate Solution* — 0.1 N.**A-10.2.2** *Ammonium Sulphocyanide* — 0.1 N.**A-10.2.3** *Concentrated Nitric Acid***A-10.2.4** *Ferric Alum Indicator* — 40 percent.

A-10.3 Procedure — Weigh about 10 g of dry pentaerythritol accurately in a 250 ml conical flask. Dissolve with heating in 100 ml of water. Prepare a blank by taking 100 ml water in another 250 ml conical flask. Add to each flask 2 ml concentrated nitric acid followed by 250 ml 0.1 N silver nitrate solution through a pipette. The solutions are titrated against 0.1 N ammonium sulphocyanide using 2 ml of ferric alum indicator till there is a persistent colour change.

A-10.4 Calculation

$$\text{Chloride, as percent by mass} = (A - B) \times \frac{35.45}{1\ 000} \times \frac{100}{M}$$

where

A = volume of ammonium sulphocyanide consumed for blank titration,*B* = volume of ammonium sulphocyanide consumed for sample titration,*N* = normality of ammonium sulphocyanide solution, and*M* = mass in g of sample taken for the test.**A-11 DETERMINATION OF FORMIC ACID****A-11.1 Reagent****A-11.1.1** *Sodium Hydroxide Solution* — 0.02 N.

A-11.2 Procedure — Weigh about 10 g of dry pentaerythritol accurately in a 200 ml beaker and add 100 ml of boiled water. After cooling titrate with sodium hydroxide solution using phenolphthalein as indicator. The acidity is expressed as formic acid

A-11.3 Calculation

$$\text{Formic acid, percent by mass} = \frac{A \times N \times 46}{100} \times \frac{1\ 000}{M}$$

where

A = volume of sodium hydroxide solution consumed,*N* = normality of sodium hydroxide solution, and*M* = mass in g of the material taken for the test.**A-12 DETERMINATION OF IRON****A-12.1 Principle** — Pentaerythritol is incinerated and the iron content estimated in the ash colorimetrically.**A-12.2 Apparatus****A-12.2.1** *Nessler Cylinder* — 50 ml capacity.**A-12.3 Reagents**

A-12.3.1 *Standard Iron Solution* — 0.863 5 g of ferrous ammonium sulphate $[\text{NH}_4\text{Fe}(\text{SO}_4)_2 \cdot 12\text{H}_2\text{O}]$ is dissolve in 100 ml of water and made up to 1 litre (1ml — 0.1 mg of Fe).

A-12.3.2 *Ammonium Thiocyanate Solution* — 4 N.

A-12.3.3 Dilute Hydrochloric Acid — 1 : 1.

A-12.4 Procedure — Weigh accurately about 5 g of the material in a previously weighed porcelain crucible. Incinerate slowly at a low temperature and then for 1 hour at 800 °C to 900 °C. Cool the crucible and weigh to obtain the ash content. Dissolve the ash in 5 ml of 1:1 hydrochloric acid and filter, if necessary, through Whatman No. 42 filter paper and wash the residue with water. Collect the filtrate and washings in a Nessler cylinder. A reference solution is made in another Nessler cylinder comprising 5 ml of 1:1 hydrochloric acid, 2 ml of 4 N ammonium sulphocyanide solution and the volume made up to 50 ml with water. Similarly to the sample solution, 2 ml of 4 N ammonium sulphocyanide is added and the volume made up to 50 ml with water. Standard iron solution is added to the reference cylinder dropwise with shaking till the colour matches with that developed in the sample cylinder. Note the volume of standard iron solution consumed.

A-12.5 Calculation

$$\text{Iron, percent by mass} = \frac{100}{M} \times \frac{0.1}{1\,000} \times V$$

where

V = volume of standard iron solution, and

M = mass in g of the material taken for the test.

ANNEX B**SAMPLING OF PENTAERYTHRITOL**

(Clause 6.1)

B-1 GENERAL REQUIREMENTS OF SAMPLING

B-1.1 In drawing, preparing, storing and handling test samples, the following precautions and directions shall be observed.

B-1.2 Samples shall not be taken in an exposed place.

B-1.3 The sampling instrument shall be clean and dry.

B-1.4 Precautions shall be taken to protect the samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination.

B-1.5 To draw a representative sample, the contents of each container selected for sampling shall be mixed as thoroughly as possible by suitable means.

B-1.6 The samples shall be placed in suitable clean, dry and air-tight containers on which the material has no action.

B-1.7 The sample containers shall be of such a size that they are almost but not completely filled by the sample.

B-1.8 Each sample container shall be sealed air-tight after filling and marked with full identification particulars, such as sample number, the date of sampling, batch of manufacture of the material and other important particulars of the consignment.

B-1.9 Samples shall be stored in such a manner that the temperature of the material does not vary unduly from the normal temperature.

B-2 SCALE OF SAMPLING

B-2.1 Lot — In a single consignment all packages of the same size, containing material from the same batch of manufacture, shall constitute a lot. If a consignment is known to consist of packages belonging to different batches of manufacture, packages belonging to the same batch of manufacture shall be grouped together and each such group shall constitute a lot.

B-2.2 For ascertaining the conformity of the material in a lot to the requirements of this specification, samples shall be tested on each lot separately.

B-2.3 The number of packages to be sampled for this purpose shall depend on the size of the lot and shall be in accordance with column 1 and 2 of Table 2.

TABLE 2 SCALE OF SAMPLING

LOT SIZE	NUMBER OF CONTAINERS TO BE SELECTED
<i>N</i> (1)	<i>n</i> (2)
Up to 50	3
51 to 100	4
101 to 150	5
151 to 300	6
301 to 500	7
501 to 800	8
801 to 1 300	9
1 301 and above	10

B-2.3.1 These packages shall be selected at random. In order to ensure the randomness of selection, procedure given in IS 4905 may be followed.

B-3 PREPATION OF SAMPLES

B-3.1 From each of the packages selected, draw with an appropriate sampling instrument small quantity of material from different portions of the package and mix thoroughly. This shall constitute the representative sample for the package.

B-3.2 From the samples representing different packages selected in **B-2.3**, small but approximately equal quantity of material shall be taken and mixed thoroughly to form a composite sample which shall be sufficient for carrying out triplicate tests for the characteristics given in Table 1. The composite sample shall be divided into three equal parts, one for the purchaser, another for the supplier and the third for the referee.

B-3.4 The composite samples shall be transferred to separate containers. These sample containers shall then be sealed with stoppers and labelled with full identification particulars given in **B-1.8**.

B-4 NUMBER OF TESTS

B-4.1 Tests for all characteristics given in Table 1 shall be conducted on the composite sample.

B-5 CRITERIA FOR CONFORMITY

B-5.1 The lot shall be declared as conforming to this specification, if the test results on the composite sample for each of the characteristics meet relevant requirements given in Table 1.