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JSS 1376-02 : 2017
(Fifth Revision)



भारत सरकार
GOVERNMENT OF INDIA
रक्षा मंत्रालय
MINISTRY OF DEFENCE

संयुक्त सेवा विनिर्देश
JOINT SERVICES SPECIFICATION

ON

TRI NITRO TOLUENE (TNT)
GRADES SPECIAL, 1E, 1A, 1B AND GRADE 2

मानकीकरण निदेशालय
रक्षा उत्पादन विभाग
रक्षा मंत्रालय
'एच'-ब्लॉक, निर्माण भवन डाकघर
नई दिल्ली-११००११

DIRECTORATE OF STANDARDISATION
DEPARTMENT OF DEFENCE PRODUCTION
MINISTRY OF DEFENCE
'H' BLOCK, NIRMAN BHAVAN POST OFFICE
NEW DELHI-110011

LIST OF MEMBERS ASSOCIATED WITH FORMULATION OF THIS STANDARD

1. This Joint Services Specification has been approved by Shri RS Gauba, Sc 'G', Member Secretary, Armament Research Board, DRDO, Chairman, Armament Standardisation Sub-committee by circulation.
2. The representatives of following organisations have been present/consulted in approving the document:

S. No.	Organisations
1.	Director PM, O/o DG (ACE), Pune
2.	ADGWE/GS (WE-2/3), New Delhi
3.	Dte of Arty (GS/Artillery - 5), New Delhi
4.	Dte Gen of Naval Armt, Naval HQ, New Delhi
5.	Dte of Armt & Safety Eqpt, Air HQ, New Delhi
6.	DGEME, Army HQ, New Delhi
7.	DGNAI, Naval HQ, New Delhi
8.	DGAQA, JD (Armt) Gp, New Delhi
9.	CQA (ME), Pune
10.	CQA (Amn), Pune
11.	CQA (SA), Ichapur, West Bengal
12.	CQA (W), Jabalpur
13.	HEMRL, DRDO, Pune
14.	ARDE/DRDO Orgn, Pune
15.	Ammunition Factory, Pune
16.	Secretary ASSC

RECORD OF AMENDMENTS

Amendment		Amendment pertains to S. No./Para No./ Column No.	Authority	Amended by	Signature & Date
No.	Date			Name & Appointment (In Block Letters)	

TABLE OF CONTENTS

S. No.	Topic	Page No.
1.	FOREWORD	1
2.	SCOPE	3
3.	RELATED SPECIFICATIONS/DOCUMENTS	3
4.	MATERIAL	4
5.	MANUFACTURE	5
6.	PRE-INSPECTION OF STORES/CONSIGNMENT	5
7.	QUALITY ASSURANCE	6
8.	PACKAGING	9
9.	MARKING	9
10.	SAFETY OF OPERATIONS	10
11.	DEFENCE CATALOGUE & NSN NUMBERS	10
12.	SUGGESTIONS FOR IMPROVEMENT	11
APPENDICES		
APPX 'A'	DETERMINATION OF SETTING POINT	12
APPX 'B'	DETERMINATION OF ACIDITY	13
APPX 'C'	DETERMINATION OF SULPHATED ASH	14
APPX 'D'	DETERMINATION OF ORGANIC MATTER INSOLUBLE IN TOLUENE	15
APPX 'E'	MELTING TEST-COLOUR STANDARD	16
APPX 'F'	DETERMINATION OF SODIUM	17
APPX 'G'	DETERMINATION OF PARTICLE SIZE	18

Table of Contents (Concluded)

S. No.	Topic	Page No.
APPX 'H'	VACUUM STABILITY TEST	19
	DRAWING	20

0. FOREWORD

0.1 This Joint Services Specification has been prepared by 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 1376-02 : 2017 (Fifth Revision):

- a) was prepared in the year 1982.
- b) was revised in the year 1990, 1996, 2004 & 2012 and supersedes the same.
- c) is revision of JSS 1376-02 : 2012 (Fourth Revision) and supersedes the same.

0.4 This specification would be used for Manufacture, Supply and Quality Assurance of Trinitiro Toluene (TNT), Grades Special, 1E, 1A, 1B and Grade 2.

0.5 Quality Assurance Authority for the items covered by this specification is the Controller, Controllerate of Quality Assurance (Military Explosives), Aundh Road, Pune-411020. 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-110011.

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-110011.

JSS 1376-02 : 2017
(Fifth Revision)

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 Trinitro Toluene, Grades Special, 1E, 1A, 1B and Grade 2.

1.2 Exclusively for all Military Explosives, TNT Special Grade, 1E Grade and Grade 1A shall only be used.

a) Grade Special - (TNT with setting point 80.6°C and above) : TNT of high purity suitable for filling of ammunition of 155 mm Gun, HE filling and Anti-Tank Mines.

b) Grade 1E - (TNT with setting point minimum 80.4°C but below 80.6°C): Suitable for PC Sheet, and for the following purposes according to its physical form:

i) As crystals or crushed flakes, for use in exploders, gains and detonators (formerly known as TNT, grade 1 for exploders).

ii) As flakes for use in HE compositions where high purity TNT is essential.

iii) As flakes for use in the preparation of crushed flakes as referred to at (i) above.

c) Grade 1A - (TNT with setting point minimum 80.2°C but below 80.4°C) : As crystals, flakes, biscuits or lumps, suitable for use as an HE filling and as an ingredient of HE compositions, suitable for slab demolition, charge demolition No. 1, 2, 11 and 14.

1.3 For Ammunition shell 130 mm HE and shell 105 mm HE - TNT Grade 1E for pour filling and 1A/1E for screw filling to be used.

1.4 For Mortar Bomb 81 mm and 120 mm, Mine APM and Cartridge 40 mm L70 - TNT Grade 1A or 1E to be used.

2. RELATED SPECIFICATIONS/DOCUMENTS

2.1 References are made in this specification to:

Table 1 Related Specification

S. No.	Specification/Document No. & Year	Nomenclature
a)	IS 138 : 1992 (Third Revision) AMD 1 Reaffirmed 2014	Ready mixed paint, Marking for Packages and Petrol Containers-Specification
b)	IS 460 (Part 1) : 1985 (Third Revision) AMD 1 Reaffirmed 2013	Specification for Test Sieves Part 1 : Wire Cloth Test Sieves
c)	IS 537 : 2011 (Second Revision)	Toluene-Specification
d)	IS 2362 : 1993 (Second Revision) Reaffirmed 2015	Determination of Water by Karl Fischer-Test Method

Table 1 Related Specification (Concluded)

S. No.	Specification/Document No. & Year	Nomenclature
e)	IS 2500 (Part 1) : 2000 (Third Revision) AMD 2 Reaffirmed 2016	Sampling Procedure for Inspection by Attributes Part 1 : Sampling Schemes Indexed by Acceptable Quality Limit (AQL) for Lot-By-Lot Inspection
f)	IS 8397 : 1976 Reaffirmed 2016	Specification for 2-Nitrotoluene
g)	JSS 8140-02 : 2013 (Third Revision)	Drum, Plywood, TNT
h)	JSS 8140-03 : 2013 (Third Revision)	Drum, Fibre, TNT (25 kg)
j)	Drawing No. IND/ME/29	Case HE Mk 1 (Modified) A/L
k)	Drawing No. IND/ME/34	Bag, Cotton for Packing TNT/DNT, 23 kg
m)	Drawing No. IND/ME/71 (B) or CQA (ME)/Drawing/246	Bag, Cotton for Packing TNT 25 kg
n)	Drawing No. IND/ME/72 Sheet No. 1 and 2	Drum, Plywood, TNT (For packing 25 kg TNT)
p)	Drawing No. CI/M/142	Setting Point Thermometer
q)	Drawing No. CIME/ DRG/213	Case, HE, Mk-1 A/L, Wood for Packing 23 kg TNT, CE or 25 kg RDX/TNT, 60/40
r)	Drawing No. IND/ME/Stencil/28	Marking on Plywood Drums

2.2 Copies of drawings can be obtain on payment from:

The Controller,
 Controllerate of Quality Assurance (Military Explosives),
 Aundh Road,
 Pune- 411020.

3. MATERIAL

3.1 TNT, Grades Special, 1E, 1A or 1B shall consist of 2 : 4 : 6 Trinitro Toluene (alpha Trinitro Toluene) $C_6H_2CH_3(NO_2)_3$ produced by the direct nitration of Toluene or 2-Nitro Toluene (or the Nitrotoluene) and shall not contain material derived from recovery and repurification of TNT previously used in ammunition.

3.1.1 TNT Grade 2 shall consist essentially of alpha Trinitro Toluene produced by direct nitration of Toluene or derived by purification by an approved process from TNT recovered from munitions filled either with TNT alone or with amatol or it shall consist of material derived from both of these sources.

3.2 TNT shall be supplied in the form of crystals, flakes, lumps or biscuits.

3.3 TNT in the form of biscuits shall be free from piping and porosity and shall comply with the requirements as to density, thickness and size of fragments as specified in the contract.

3.4 TNT in the form in which it is supplied, shall comply with the requirements of Clause **6.4 (f)** with regard to the properties of the molten material. It shall be free from grit, visible impurities and foreign matter.

4. MANUFACTURE

4.1 TNT, Grades Special, 1E, 1A, 1B or Grade 2 shall be manufactured by a process which has received authoritative approval. The Quality Assurance Authority shall be informed regarding the process used and shall be given prior notification of any proposed deviation therefrom. All deviations from the approved process, however slight, shall be recorded immediately and all material affected shall be set aside pending the decision of the Quality Assurance Authority.

4.2 Toluene used in the manufacture shall be of nitration grade and comply with the requirements of IS 537. The following limit in respect of Benzene, Paraffin, and purity of Toluene shall be strictly maintained.

- a) Benzene, *Max* 0.3%
- b) Paraffin, *Max* 0.5%
- c) Purity of Toluene, *Min* 99.5%

4.3 2-Nitrotoluene used in the manufacture shall comply with the requirements of IS 8397.

5. PRE-INSPECTION OF STORES/CONSIGNMENT

5.1 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 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.

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

6. QUALITY ASSURANCE

6.1 Inspection

JSS 1376-02 : 2017
(Fifth Revision)

6.1.1 TNT Grades special, 1E, 1A, 1B or Grade 2 and the packages in which it is packed shall be subject to inspection by and to the approval of the Quality Assurance Officer/Quality Assurance Authority.

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

6.2 Sampling

6.2.1 One lot of TNT shall contain 1000 kg packed in 40 approved containers of 25 kg each.

6.2.2 The sampling shall be in accordance with Table 2 Inspection Level IV of IS 2500 (Part 1). 8 samples each of 125 g shall be drawn at random from every 5th container. These samples are thoroughly mixed to make a composite sample of 1000 g. From this composite sample 500 g shall be subjected to the tests viz. visual examination, setting point, melting test, acidity and matter insoluble in Toluene as per the test requirements given in Para **6.4**.

6.2.3 Out of the remaining 500 g composite sample, 400 g shall be retained for reference purposes. Remaining 100 g each from consecutive five lots shall be thoroughly mixed to make a composite sample of 500 g, which shall be subjected to the tests viz. moisture content, sulphated ash, sodium content and particle size as per the test requirements given in Para **6.4.1**.

6.2.4 Once the individual lot conforms to the test requirements for the five tests viz. visual examination, setting point, melting test, acidity and matter insoluble in Toluene and the composite sample conforms to the requirements for the remaining four tests viz. moisture content, sulphated ash, sodium content and particle size as given in table of Para **6.3.1**, the five lots shall be accepted.

6.3 Criteria of Conformity

6.3.1 If, on examination, any sample is found not to conform to this specification, the whole lot may be rejected.

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

6.4 Test Requirements

Samples taken from any portion of the batch/lot/consignment of the material shall conform to Clause **3** and in addition shall conform to the following test requirements:

Table 2 Test Requirements

S. No.	Characteristics	Passing Standard				Test Method
		* Spl	* Gr 1E	Gr 1A	Gr 1B & Gr 2	
		(For All Military Explosives)				
a)	Setting point, °C	80.6 <i>Min</i>	80.4 <i>Min</i>	80.2 <i>Min</i>	80.0 <i>Min</i>	Appx 'A'
b)	Acidity as H ₂ SO ₄ percent by mass	0.003 <i>Max</i>	0.003 <i>Max</i>	0.005 <i>Max</i>	0.005 <i>Max</i>	Appx 'B'
c)	Moisture, percent by mass	0.10 <i>Max</i>	0.10 <i>Max</i>	0.10 <i>Max</i>	0.10 <i>Max</i>	IS 2362
d)	Sulphated ash, percent by mass	0.05 <i>Max</i>	0.05 <i>Max</i>	0.05 <i>Max</i>	0.05 <i>Max</i>	Appx 'C'
e)	Organic matter insoluble in Toluene, percent by mass:					Appx 'D'
	1) Total matter insoluble minus Sulphated ash, percent by mass	0.05 <i>Max</i>	0.05 <i>Max</i>	0.05 <i>Max</i>	0.05 <i>Max</i>	
	2) Gritty particle retained on 250 micrometre IS Sieve	Nil	Nil	Nil	Nil	
f)	Melting Test:					Appx 'E'
	1) Scum, suspended matter and deposit	Nil	Nil	Nil	Nil	
	2) Colour of freshly melted material	Not darker than the standard				
	3) Darkening on heating for 2 h	No appreciable change				
g)	Total Sodium calculated as Sodium, percent by mass	0.001 <i>Max</i>	0.001 <i>Max</i>	0.001 <i>Max</i>	0.001 <i>Max</i>	Appx 'F'
h)	Particle size (dimensional requirements):					Appx 'G'
	1) Crystal form	The whole of the material should be capable of passing 600 micrometre IS Sieve				
	2) Flake form					
	i) Average thickness of flakes, mm	0.6 <i>Max</i>	0.6 <i>Max</i>	0.6 <i>Max</i>	0.6 <i>Max</i>	

Table 1 Test Requirements (Concluded)

S. No.	Characteristics	Passing Standard				Test Method
		* Spl	* Gr 1E	Gr 1A	Gr 1B & Gr 2	
		For All Military Explosives				
	ii) Thickness of individual flake, mm,	1.0 <i>Max</i>	1.0 <i>Max</i>	1.0 <i>Max</i>	1.0 <i>Max</i>	
	iii) Aggregates	Nil	Nil	Nil	Nil	
	3) Crushed flake form					
	i) Passing through 850 micrometre IS Sieve, percent by mass	95 <i>Min</i>	95 <i>Min</i>	95 <i>Min</i>	95 <i>Min</i>	
	ii) Passing through 300 micrometre IS Sieve, percent by mass	20 <i>Min</i> 40 <i>Max</i>	20 <i>Min</i> 40 <i>Max</i>	20 <i>Min</i> 40 <i>Max</i>	20 <i>Min</i> 40 <i>Max</i>	
j)	Vacuum stability Test (Volume of gas evolved per 10 g of 50/50 Amatol test sample/16 h/ 120°C calculated to N.T.P.) ml,	-	-	-	2.0 <i>Max</i>	Appx 'H'

* For all Military Explosives, TNT special Grade, Grade 1E or Grade 1A shall only be used:

NOTES

1. Particulars of IS Sieves referred to in this specification shall be found in IS 460 (Part 1).
2. TNT has a slight toxic action on the skin and mucous membrane. As such care must be taken to avoid unnecessary handling with bare hands and inhalation of TNT dust through nose and mouth.
3. Carry out all the operations likely to give rise to TNT dust, either in a fume cupboard or on a hooded bench. Wear a light cotton pad respirator wherever there is risk of the dust being inhaled.
4. After working with TNT, scrub the hands well with sulphite soap and cold water.
5. Setting point grading of TNT shall be done as under:
 - a) Lots giving setting point less than 80.2°C but minimum 80.0°C shall be graded as Grade 1B.
 - b) Lots giving setting point less than 80.4°C but minimum 80.2°C shall be graded as Grade 1A.
 - c) Lots giving setting point less than 80.6°C but minimum 80.4°C shall be graded as Grade 1E.

- d) Lots giving setting point minimum 80.6°C and above shall be graded as special grade.
- e) Where TNT with lower setting point has been specified for use, TNT with higher setting point shall be automatically permitted, that is:
 - i) Grade Special and Grade 1E will be permitted in lieu of Grade 1A.
 - ii) Grade Special will be permitted in lieu of Grade 1E and 1A.
 - iii) Lower setting point obtained from testing two samples from the same lot should be recorded as setting point of the lot.

7. PACKAGING

7.1 TNT, Grades Special, 1E, 1A, 1B or Grade 2 shall be packed in sound, clean, dry cotton bag, conforming to drawing No. IND/ME/34 and IND/ME/71 (B) or CQA (ME)/drg/246. The bag shall be securely closed and placed inside a suitable wooden cases/plywood drums conforming to:

- a) JSS 8140-02 Drum, Plywood, TNT
- b) JSS 8140-03 Drum, Fibre, TNT (25 kg)
- c) Drawing No. IND/ME/29 - Case, HE, Mk 1 (modified) A/L
- d) Drawing No. IND/ME/72 - Drum, Plywood, TNT (Sheet No. 1 and 2)
- e) The quantity per package shall not exceed 25 kg.
or
Drawing No. CIME/DRG/213-Case, HE, Mk-1 A/L and the quantity per package shall not exceed 23 kg.

7.2 Material packed in any other containers/packages shall have the prior approval of the Quality Assurance Authority.

7.3 The inclusion of any foreign matter or impurities in any of the containers/packages, detected on visual examination shall render the whole consignment liable to rejection.

8. MARKING

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

- a) Nomenclature and specification number of the material.
- b) Name and address of the consignee.
- c) A/T or SO number and date.
- d) *Consignment number.

JSS 1376-02 : 2017
(Fifth Revision)

- e) Lot/Batch number 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) Contractor's initials or recognised trademark.
- k) Station unit e.g. HEFK to be stenciled.

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

NOTES

1. Marking on the plywood drums shall be in accordance with drawing number IND/ME/Stencil/28.
2. Grade of the material i.e. Grade special, 1A/1B/ 1E/Grade 2 shall be shown by affixing labels of cloth-seal.
3. The following colour code shall be used:

a)	Grade 1B & Grade 2	Blue seals
b)	Grade 1A	White seals
c)	Grade 1E	Red seals
d)	Grade Special	Green colour cloth seals and letters 'Special grade' to be written in black colour

8.2 A Government Explosive Group 'D' and UN Hazard Division 1.1 D marking of approved pattern shall be affixed to each container by means of a prescribed label. Alternatively, the marking may be affixed by means of a stencil.

8.3 The paint used for marking should conform to IS 138 (latest issue) and to the satisfaction of the Quality Assurance Officer/Quality Assurance Authority.

9. SAFETY OF OPERATIONS

9.1 The safety of all the operations during manufacture shall be the responsibility of the manufacturer.

9.2 Safety certificate SC/H/65/55 is applicable (available on application from CQA (ME), Aundh Road, Pune-411 020).

10. DEFENCE CATALOGUE & NATO STOCK NUMBERS

10.1 The Defence Catalogue numbers/NATO STOCK Numbers allotted to this store are as under:

<i>S. No.</i>	<i>Grades</i>	<i>DCAN</i>	<i>NSN</i>
a)	TNT Grade Special	1376-000 088	1376720302018
b)	TNT Grade 1E	1376-000 004	1376720301990
c)	TNT Grade 1A	1376-000 005	1376720301991
d)	TNT Grade 1B	1376-000 006	1376720301992
e)	TNT Grade 2	1376-000 107	1376720301993

11. SUGGESTIONS FOR IMPROVEMENT

11.1 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-110011.

DETERMINATION OF SETTING POINT

A-1. Heat about 80 g of the TNT in a shallow porcelain dish in boiling water bath together with a test tube (150 mm x 25 mm dia) and an Aluminium wire stirrer. Continue the heating for 2 hours after the TNT has fully melted. In the meantime, place the setting point thermometer, range 76°C to 85°C graduated in 1/10th degree (Drg No. CI/M/142 for reference), in the oven at 80°C to 90°C. Fill the tube with the molten TNT, leaving about 25 mm from the top and insert it into another larger tube (175 mm x 40 mm dia), supported by a cork, the larger tube being supported in a glass jar by a wooden cover. Without delay, insert the stirrer and adjust the setting point thermometer centrally in the melt, so that the extremity of the bulb is 25 mm from the bottom of the tube. Place a screen in position round the glass jar. The thermometer is held by a clamp and no corks are to be used. The temperature of the TNT at this stage should not be lower than 85°C.

A-2. Commence stirring vigorously at once and continue as the temperature falls and until the first rise of temperature occurs. The thermometer reading is determined by means of a lens during the rise in temperature and the maximum temperature indicated is recorded to the nearest 0.1°C. It is necessary to check by gentle tapping of the thermometer that no sticking of the mercury thread has occurred at the time of reading.

A-3. Without delay, ascertain the temperature of the air adjacent to the exposed stem of the setting point thermometer, the number of degree divisions of the setting point thermometer exposed and make the necessary correction for the emergent stem as follows:

$$t = 0.000143 (t_0 - t_1) \cdot n$$

where,

t = Number of degree Celsius to be added;

t₀ = Observed setting point in degree Celsius;

t₁ = Temperature of the air adjacent to the middle of the exposed column of mercury; and

n = Number of degree divisions covered by the exposed column of the mercury.

A-4. Access of strong light to the TNT during the setting point determinations must be avoided and it is, therefore, essential that a screen, wholly covering the sides of the glass jar, should be in place throughout the determination.

A-5. Lower setting point obtained from testing two samples from a lot should be reported as setting point of the lot.

APPX 'B'
(Clause 6.4)

DETERMINATION OF ACIDITY

B-1. Transfer 10 g \pm 0.1 g of the sample, accurately weighed, to a 500 ml flask. Add 25 ml of dry Toluene and dissolve TNT by warming on a boiling water bath. Add 100 ml of freshly boiled and cooled distilled water and shake vigorously for one minute. Transfer to a separating funnel, allow to separate and run off the aqueous layer into a 250 ml conical flask and titrate with 0.02 N Sodium hydroxide solution using Phenolphthalein as indicator. Carry out a blank test at the same time under identical conditions.

B-2. Calculate the acidity as percentage of Sulphuric acid using the factor:

$$1 \text{ ml } 0.02 \text{ N NaOH} = 0.00098 \text{ g H}_2\text{SO}_4$$

B-3. Acidity as H₂ SO₄, Percent by mass =
$$\frac{(t_1 - t_2) \times f \times 0.00098 \times 100}{\text{Mass of sample}}$$

where,

t₁ = ml of 0.02 N NaOH required for sample;

t₂ = ml of 0.02 N NaOH required for blank; and

f = Factor of 0.02 N NaOH.

DETERMINATION OF SULPHATED ASH

C-1. Transfer about 5 g of composite sample of TNT, to a clean dry tared porcelain crucible (M_1) and weigh accurately (M_2). Moisten with 1 ml of concentrated Sulphuric acid and heat gently until TNT ignites. Discontinue heating while TNT burns strongly but apply the burner as necessary towards the end of combustion in order to carry it to completion. Heat gently to drive off the Sulphuric acid and then more strongly to burn off the residual carbon. (Use goggles during the ignition of TNT).

C-2. Cool in a desiccator to room temperature and reweigh (M_3).

C-3. Calculate the percentage of Sulphated ash on the original sample.

C-4. Sulphated ash, percent by mass =
$$\frac{(M_3 - M_1) \times 100}{(M_2 - M_1)} \dots (A)$$

APPX 'D'
(Clause 6.4)

DETERMINATION OF ORGANIC MATTER INSOLUBLE IN TOLUENE

D-1. Transfer 10 g ± 0.1 g of the sample, accurately weighed to a 300 ml beaker. Add 150 ml of dry Toluene, cover with a clock glass and place it on a boiling water bath so adjusted that the solution is just below its boiling point. Stir occasionally to assist solution and then allow to stand on the water bath for about 3 hours to coagulate the insoluble matter. Decant the solution through a clean dry and tared sintered glass crucible (M₁) and wash the residue from the beaker into the crucible with warm dry Toluene, continue washing until free from TNT. Heat the crucible and the contents in boiling water oven for 2 hours, cool in a desiccator and weigh (M₂).

D-2. Organic Matter insoluble in Toluene, Percent by mass = $\frac{(M_2 - M_1) \times 100}{\text{mass of sample}}$ A

where,

A = percentage of Sulphated ash (from Appx 'C').

D-3. Brush the insoluble matter on to a 250 micrometre IS Sieve. Count and note the nature of the particles, if any, retained on the sieve.

NOTE - For the purpose of the test, the term gritty particles are defined as the number of organic insoluble particles retained on 250 micrometre IS Sieve.

MELTING TEST-COLOUR STANDARD

E-1. Prepare the colour standard by mixing the following aqueous solutions in the proportions shown below:

- a) Potassium dichromate ($K_2 Cr_2 O_7$) 80 g per litre - 35 ml
- b) Potassium permanganate ($KMnO_4$) 3.16 g per litre - 1.25 ml
- c) Copper sulphate crystals ($CuSO_4 \cdot 5H_2 O$) 200 g per litre - 13.7 ml

E-2. The constituent solution should be mixed freshly for each occasion of use and filtered before mixing if cloudy.

E-3. It is normally possible to observe the colour of the melt and the presence of scum, suspended matter and deposit during the determination of setting point.

E-4. Nearly fill a 150 mm x 25 mm dia test tube with molten TNT and compare it with similar tube filled with freshly prepared liquid colour standard as given above, employing either a back ground of diffused light or sheet of white paper.

APPX 'F'
(Clause 6.4)

DETERMINATION OF SODIUM

F-1. Before quantitative determination, the presence/absence of Sodium sulphite (used in the manufacture of TNT) is confirmed qualitatively by the following method:

F-2. Transfer about 10 g of composite sample of TNT to a 150 ml beaker, add 50 ml of distilled water and heat on a boiling water bath till all TNT is molten. Cool the contents with stirring to room temperature. Filter the liquid through a No. 1 Whatman filter paper. Wash the residual TNT in the beaker with further 25 ml of distilled water and filter. Collect the filtrate and washings.

F-3. To the combined filtrate, add 2 ml to 3 ml of starch solution (approx 0.5%, freshly prepared) and a drop of 0.1 N Iodine solution.

F-4. Appearance of blue colour which does not disappear on shaking for about 2 to 3 minutes shows the absence of Sodium sulphite/Sodium.

F-5. Quantitative Determination

a) Transfer about $10 \text{ g} \pm 0.1 \text{ g}$ of the sample to a clean, dry silica dish and heat slowly on a Bunsen burner so as to retain all Sodium in the ash (The heating is continued till all material is converted into ash).

b) Extract the ash with 25 ml of hot distilled water and filter through a No. 1 Whatman filter paper. Wash the residual ash again with 25 ml of hot distilled water and filter. Collect the filtrate and washings. The combined filtrate is concentrated to reduce the volume to about 50 ml. Transfer the filtrate into a 50 ml standard volumetric flask and make up to 50 ml volume. Estimate Sodium content by flame photometer and express the results as Sodium (Na).

DETERMINATION OF PARTICLE SIZE

G-1. Brush $20 \text{ g} \pm 0.1 \text{ g}$ of the sample on a 600 micrometre IS Sieve for 15 minutes and weigh the amount retained on the sieve.

G-2. Flake Form

a) Select 20 flakes at random and measure the thickness of each to the nearest 0.025 mm by means of a micrometer screw gauge.

G-3. Crushed Flake Form

a) Prepare a nest of 850 micrometre and 300 micrometre IS Sieve over a receiver. Place $20 \pm 0.1 \text{ g}$ of the sample on the top sieve and brush the sample for 15 minutes. Weigh the amount retained on the sieve. Remove the top sieve and brush the sample on the bottom sieve for 15 minutes. Weigh the amount retained on the sieve.

b) From the above data, calculate the amount passing 850 micrometre IS Sieve and 300 micrometre IS Sieve and express as percentage on the original sample.

APPX 'H'
(Clause 6.4)

VACUUM STABILITY TEST

H-1. Thoroughly dry recovered TNT and Ammonium nitrate Grade 1 separately and take care that during weighing, mixing etc Ammonium nitrate is exposed to the air as little as possible. Mix 5 g of TNT (dried) with 5 g of Ammonium nitrate (dried) in the vacuum stability test tube by corking and then shaking. Affix the manometer using lubricant for ground glass joint, a 50/50 mixture of DNT and DNB in place of usual non-reactive grease and evacuate the tube.

H-2. Insert the prepared apparatus (vacuum stability test tube with manometer) in the heating bath at 120°C and carry out the test for 16 hours, taking 'zero' time at half an hour after the insertion of the tube into the bath.

H-3. The volume of gas evolution at the end of the test is calculated at NTP and expressed as the vacuum stability.

H-4. Details of the method for vacuum stability can be obtained on application to the Controller, CQA (ME), Aundh Road, Pune-411020.

