NORTH ATLANTIC TREATY ORGANIZATION
ORGANISATION DU TRAITE DE L'ATLANTIQUE NORD

MILITARY AGENCY FOR STANDARDIZATION (MAS)
BUREAU MILITAIRE DE STANDARDISATION (BMS)
1110 BRUSSELS

MAS/302-MMS/4025
20 November 1991

To : See MAS Distribution List No. 2

Subject : STANAG 4025 MMS (EDITION 3) - SPECIFICATION FOR TNT (TOLITE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

References : a. MAS(ARMY)(62)644 dated 25 September 1962 (Edition 2)
b. AC/310-D/43 dated 25 April 1985

Enclosure : STANAG 4025 (Edition 3)

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page iii is promulgated herewith.

2. The references listed above are to be destroyed in accordance with local document destruction procedures.

3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

4. National staffs are requested to examine page iii of the STANAG and if they have not already done so, to advise the Defence Support Division, IS, through their national delegation as appropriate of their intention regarding its ratification and implementation.

DISTRIBUTION STATEMENT C
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E. Stai
E. STAI
Major-General, NOAF
Chairman, MAS

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MAS302A

STANAG 4025
(Edition 3)

NORTH ATLANTIC TREATY ORGANIZATION
(NATO)

MILITARY AGENCY FOR STANDARDIZATION
(MAS)

STANDARDIZATION AGREEMENT

SUBJECT: SPECIFICATION FOR TNT (TOLITE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

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Promulgated on 20 November 1991

E. STAI
Major-General, NOAF
Chairman, MAS

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Chairman MAS under the authority vested in him by the NATO Military Committee.

2. No departure may be made from the agreement without consultation with the tasking authority. Nations may propose changes at any time to the tasking authority where they will be processed in the same manner as the original agreement.

3. Ratifying nations have agreed that national orders, manuals and instructions implementing this STANAG will include a reference to the STANAG number for purposes of identification.

DEFINITIONS

4. Ratification is "The declaration by which a nation formally accepts the content of this Standardization Agreement".

5. Implementation is "The fulfilment by a nation of its obligations under this Standardization Agreement".

6. Reservation is "The stated qualification by a nation which describes that part of this Standardization Agreement which it cannot implement or can implement only with limitations".

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

7. Page iii gives the details of ratification and implementation of this agreement. If no details are shown it signifies that the nation has not yet notified the tasking authority of its intentions. Page iv (and subsequent) gives details of reservations and proprietary rights that have been stated.

Agreed English/French Texts

STANAG 4025
(Edition 3)

NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT (STANAG)

SPECIFICATION FOR TNT (TOLITE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Annex: A. TNT Solidification Point Apparatus

Related documents: None

AIM

1. The aim of the Agreement is to establish a common minimum specification for deliveries of TNT (Tolite) from one NATO nation to another.

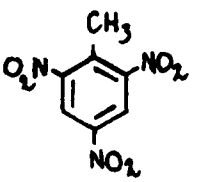
AGREEMENT

2. Participating nations agree that all TNT manufactured by NATO nations to be delivered to another NATO nation, except when ordered for particular uses, must comply with the following minimum requirements:

PART I - PHYSICAL AND CHEMICAL CHARACTERISTICS

3. a. Composition

The TNT must consist essentially of 2, 4, 6 - trinitrotoluene corresponding to the formula:



The purchaser may require of the supplier evidence of the purity of the material and more particularly of the mononitrotoluene content and/or of its propensity towards exudation, as determined by tests to be specified by the purchaser.

b. Process

This STANAG is applicable only to TNT that has been prepared by the direct nitration of toluene and has been refined with Na_2SO_3.

4. Appearance and Granulation

a. General use TNT must be in the form of yellow flakes and is intended for use in the loading of shells and bombs and in the manufacture of demolition blocks and explosive mixtures. The average thickness of flakes shall not be more than 0.025 inch (0.63 mm). The thickness of any individual flake shall not be more than 0.04 inch (1.0 mm). If supplied as crystals, biscuits or in lump form, the dimensional requirement shall be specified by the purchaser.

b. Special use TNT is suitable for the following purposes according to its physical form. a) as crystal or crushed flake for use in exploders, gaines and detonators; b) as flake for use in high explosive compositions where a high quality TNT is essential.

c. Utility use TNT is of a similar description as general use TNT and used for similar purposes. It is of slightly lower quality, however, as determined by its solidification point.

d. Ultra-pure TNT must be in the form of flakes, crushed flakes or yellow crystals and is used in circumstances where the most exacting purity standards are demanded.

e. The colour of the sample will be specified in the order, making use of the purchaser's standards.

f. TNT samples will be examined for foreign matter and for any abnormalities as specified in the order. Any scum, suspended matter and sediment, if present, will be detected and reported using the method defined in paragraph 8.1.

5. Chemical Data

a. Solidification point: The four qualities of TNT are differentiated by their solidification point determined using the method of paragraph 8.a. These qualities are defined as:

Quality	Solidification point
Utility use	min 79.5°C
General use	min 80.2°C
Special use	min 80.4°C
Ultra-pure	min 80.6°C

All four qualities of TNT must conform to the specifications in the following list:

b.	Moisture	max. 0.1%
c.	Acidity	max. 0.005% as H_2SO_4 or 1.0 meq/kg
d.	Alkalinity	none
e.	Insoluble in Toluene	max. 0.05%
f.	Sodium	max. 0.001%
g.	Grit-retained on a 0.25 mm seive	none
	-retained on a 0.063 mm seive	max. 0.05%
h.	Scum, suspended matter and sediment	none
i.	Sulphated Ash	max. 0.05%.

PART II - TESTING METHODS

6. General

Normal laboratory procedure applies to the determination of the characteristics of the material. The following descriptions are limited to the definitions of particulars to be complied with when making the tests.

7. Physical Tests

a. Granulation: Place 100 ± 0.1 gm of the sample on the specified sieve (or sieves) fitted with a lid and bottom pan. Shake the sieve (or sieves) by hand or by a suitable mechanical means for 3 minutes and weigh the amount retained on each sieve(s) and if required the amount passing the bottom sieve. Report as percentage retained on or passing the sieve(s).

b. Thickness of Flake: Measure 20 flakes with a micrometer accurate to at least 0.001 inch (0.025 mm).

8. Chemical Tests

a. Crystallizing Point (Solidification Point):

Apparatus: Suitable apparatus for the determination of the crystallizing point is shown in Annex A. If the outer bottle is not of amber glass an aluminium screen should be placed round the apparatus to protect the sample from direct sunlight during the test.

The standard thermometer must be accurately calibrated (to 0.01°) over the range 79-81°C at least.

Procedure: Melt about 50 g of sample in a covered beaker by immersing the beaker in a water bath at 95-100°C or placing it in a suitable oven. Pour the melt into the inner tube of the apparatus to a level between

25 and 35 mm from the top. At this stage the melt should be at a temperature not less than 85°C; if necessary, reheat the tube in a hot water bath and then return it to the apparatus. Insert the thermometer and stirrer, preferably pre-heated to avoid encrustation with solid TNT, so that the bulb is located centrally in the tube and about 25 mm from the bottom, and stir the melt continuously by vertical movement of the stirrer. Observe the temperature, which will fall steadily and then begin to rise as crystallization sets in: discontinue stirring at this stage and note the minimum temperature reached.

The temperature will rise to a maximum and remain steady for some minutes. Tap the thermometer gently to ensure that the mercury is not sticking and record this maximum temperature.

NOTE: If the amount of super-cooling exceeds 1.5°C, the observed setting point may be lower than the true figure and the operation should then be repeated on a fresh sample. In this context, "super-cooling" refers to the difference in temperature between the maximum temperature recorded during solidification of the melt and the minimum temperature recorded before solidification begins.

Calculation: Report the maximum temperature recorded as described under "Procedure" as the Crystallizing Point unless a total immersion thermometer has been used and there is a significant length of mercury thread exposed above the sample. In this case it is necessary to apply a correction for emergent stem. The correction is added to the observed temperature and is calculated from the formula:

$$C = 0.00016\ (T_0 - T_1)\,n$$

where:

- C = the correction in degrees Celsius
- T_0 = the observed reading in °C
- T_1 = the mean temperature of the exposed column of mercury as indicated by the thermometer adjacent to the middle of the emergent stem
- n = the number of degree divisions in the length of the exposed column of mercury

b. Moisture

Determine the moisture content of the sample by the Karl Fischer method, using the back-titration technique to an electrometric end-point. Details of suitable equipment and procedures are given in MIL-STD-650 Method 101.4 (USA) and British Standard 2511 (UK). Use 8-10 g of sample, accurately weighed, dissolved in approximately 100 ml of a solvent consisting of equal proportions of dry toluene and dry methanol.

c. Acidity or Alkalinity:

Procedure (1): Transfer an accurately weighed portion of about 10 g of sample to a 250 ml glass-stoppered iodine flask or equivalent. Add 40 ml toluene from a graduated cylinder to the sample and also to an empty flask which will serve as a blank. Stopper the flasks. Swirl the sample flask until solution is complete. Put 0.75 ml (approx. 20 drops) of 0.05% aqueous bromothymol blue indicator in a 100 ml graduated cylinder and dilute to the mark with CO_2-free distilled water. Transfer the 100 ml of water containing the indicator to the blank flask and replace stopper. Repeat this procedure with the sample flask. Swirl both sample and blank flasks vigorously for 10-20 seconds to ensure interaction of toluene and water layers.

NOTE: Too vigorous swirling or shaking must be avoided or an emulsion will be produced which may be slow to disperse.

Titrate the blank solution first. If the lower (aqueous) layer is blue, add a measured amount 0.01 N H_2SO_4 dropwise until it turns green or yellow and add an equal amount to the sample. If the solution is green or yellow at the start, begin to titrate with 0.01N NaOH. Add the NaOH dropwise, stoppering the flask after each addition and swirling vigorously for 5-10 seconds (see note above). The end point is taken as a blue colour which persists for 2 minutes after the toluene and water have separated into distinct layers and which persists after one additional 5-10 seconds swirling.

NOTE: The blue colour may fade somewhat or acquire a trace of green coloring after the final swirl but this is acceptable.

The sample is now titrated in a manner similar to that for the blank titration. The end point is a persistent blue as described in the blank determination but care must be taken to look through the aqueous layer horizontally against a white or colourless background since transmitted or reflected light from the yellow toluene solution will cause a green colouration. In addition incomplete separation of toluene and water may result in a cloudiness which may also impart a slight green cast to the aqueous layer.

Calculation

Acidity: Milliequivalents per kilogram =

$$\frac{1000\ (V_1-V_2)N}{W}$$

or

$$wt\%H_2SO_4 = \frac{4.9(V_1-V_2)N}{W}$$

where:

V_1 = ml NaOH for sample
V_2 = ml NaOH for blank

N = Normality of NaOH
W = Sample wt., grams

Procedure (2): Weigh 10 g of the TNT sample with an accuracy of ± 0.01 g in a 500 ml flask and dissolve with 50 ml acetone. Add 150 ml distilled water, shake thoroughly and wait (1 to 2 min.) until the precipitated TNT has settled. Then add 10 drops of indicator (0.1 g methyl red and 0.05 g methylene blue in 100 ml of 96% ethyl alcohol) and titrate with 0.01 N sodium carbonate solution or 0.01 N sulphuric or hydrochloric acid according to whether an acid condition is indicated by violet or an alkaline condition is indicated by a green coloration. The volume V_1 (in ml) of the consumed titre is recorded. Simultaneously conduct a blank test and determine the volume V_2 consumed. The acidity is

$$\frac{1000\ (V_1 - V_2)N}{W}\ \text{(meq/kg)}$$

or $$\frac{4.9\ (V_1 - V_2)N}{W}\ (\text{wt\%}H_2SO_4)$$

where N is the normality of the titre and W is the weight in grams of the TNT sample. Volumes are taken as positive when titrating with base and negative when titrating with acid.

d. Alkalinity: The specimen will be considered unsatisfactory with respect to alkalinity if:

- in Procedure (1), the sample solution is still alkaline after correction for any alkalinity in the blank, or if V_2 is greater than V_1, or
- in Procedure (2), V_2 is greater than V_1 when calculating acidity and when negative volume values are used when titrating with acid and positive volume values are used when titrating with base.

e. Insoluble Matter

Procedure: Take 10 ± 0.1 g of the sample, and place it in a 400 ml beaker. Add 150 ml of dry toluene, cover with a clock glass, and place the beaker on a hot water bath. Stir occasionally to assist solution, and then leave the beaker on the bath to coagulate the insoluble matter. At the end of this time (at least 30 minutes) pour the solution through a previously prepared and weighed (W_1) filtering crucible of porosity grade G_4 as

used in the UK, or the equivalent. Wash the residue with warm dry toluene, and continue washing until the residue is free from TNT. Dry the crucible and contents at 100°C, cool in a dessicator, and weigh (W_2).

Calculation: Percentage insoluble matter = $10(W_2 - W_1)$

f. Grit

NOTE. Grit is defined as the inorganic matter greater than 63 micrometres in size which remains after treatment with aqua regia.

Apparatus: Sieves having the following dimensions and aperture sizes:

4 cm diameter, 9 cm high, 63 µm

4 cm diameter, 1 cm high, 250 µm

Procedure: Transfer 50 g of sample to the tall 63 µm sieve. Stand the sieve in a 400 ml beaker, add 200 ml of toluene, and place on a boiling water hot plate. Stir the material carefully to assist solution. Stand the sieve in a fresh beaker and repeat as necessary until all the TNT has dissolved. Wash the sieve with toluene and dry on a boiling water hot plate or in a suitable oven.

Transfer the contents of the sieve to a 50 ml beaker, add 5 ml aqua regia, and heat on a hot plate until the bulk is reduced to 2-3 ml. Allow to cool, dilute with water, add a few drops of phenolphthalein indicator, and make slightly alkaline by adding sodium hydroxide solution, 100 g/litre. Alternatively wash the residue with successive portions of water by decantation until free from acid.

Pour the liquid through the 63 µm sieve; wash thoroughly with water, and finally with acetone. Dry the sieve, cool it, and weigh (W) any material retained. Transfer the residue to the 250 µm sieve, and count any gritty particles retained.

Calculation: Calculate the percentage grit retained on the 63 µm sieve as follows:

Percentage grit retained on 63 µm BS sieve = 2 W.

Report the number of particles per 50 g sample retained on the 250 µm BS sieve.

g. Sulphated Ash:

WARNING - Goggles must be worn during the ignition of the TNT

Procedure: Prepare a 6-7 cm diameter porcelain crucible by igniting it in a Bunsen flame. Cool in a dessicator for 20 minutes and weigh

accurately (W_1). Place 5 ± 0.1 g of the sample in the crucible and add 1 ml of concentrated sulphuric acid. Warm the crucible and contents for 30 minutes on a boiling water bath and then transfer it to a pipeclay or silica triangle supported upon a tripod in a fume cupboard. Heat with a Bunsen flame adjusted to 4 to 5 cm in length and, as soon as the contents of the crucible begin to fume, apply the flame to the surface; repeat this treatment until the TNT catches fire. Discontinue heating while the TNT burns strongly, but apply the burner as necessary towards the end of the 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.

Cool in a dessicator for 20 minutes and weigh accurately (W_2).

Calculation: Percentage sulphated ash = 20 (W_2 - W_1).

h. Sodium Content: The sodium content of the TNT is determined using a suitable atomic absorption spectrophotometer or flame photometric technique. Two procedures are described; the first is based on the use of a solution of TNT in a solvent whereas in the second procedure the sodium is determined in an aqueous extract of the TNT. Alternative procedures may be used if they can be shown to be of similar accuracy and precision.

Apparatus: A suitable spectrophotometer maintained and operated according to the manufacturer's instructions.

Reagents: Use sodium free water, or alternatively carry out the determinations using a homogeneous supply of water so that proper corrections will be made for the sodium content.

Adjustment of the Spectrophotometer: Set up the apparatus for the determination of sodium. If a flame photometer is being used, set the wavelength at 589 nanometers and then adjust the wavelength setting to give maximum response on the detector when a solution containing approximately 10 ppm of sodium is aspirated.

Determination of Sodium - Direct Solution Procedure

Calibration of Instrument

Concentrated Sodium Salicylate Solution: Weigh accurately 0.6963 g of reagent grade sodium salicylate, transfer it to a clean, dry 1000 ml volumetric flask and dissolve it in 700 ml of acetone and sufficient water to make the total volume up to the mark. This solution contains 100 ppm of sodium.

Calibration Solutions: Transfer to separate 100 ml volumetric flasks 2, 4, 6, 8, 10 and 12 ml of the concentrated sodium salicylate solution, add 70 ml of acetone to each flask, make up to mark with water and mix thoroughly. These solutions contain respectively 2, 4, 6, 8, 10 and 12 ppm sodium.

Calibration Curve: Aspirate into the flame of the spectrophotometer a "blank" solution consisting of a mixture of 70 ml of acetone and 30 ml of water and record the detector response.

Repeat with the calibration solutions described under "Calibration Solutions" and plot the graph of sodium concentration against detector response.

Test Procedure: Weigh out 5 ± 0.05 g of the sample and transfer it to a 100 ml volumetric flask. Add 70 ml of acetone and swirl the flask until the TNT has dissolved. Make up to the mark with water and mix thoroughly. Spray the solution into the flame under the same conditions as were used in the calibration process and record the detector response. From the calibration curve obtain the corresponding concentration of sodium in the test solution.

Calculation: Percentage sodium in sample = C x 0.002

where:

C = concentration of sodium in sample solution (under "Test Procedure").

Determination of Sodium - Aqueous Extract Procedure

Calibration of Instrument

Standard Sodium Solution: Weigh accurately 0.2545 g of reagent grade sodium chloride (dried at 105°C for 1 hour) and dissolve it in water in a 1 litre volumetric flask; make up to the mark with water when solution is complete. This standard solution contains 100 ppm of sodium.

Calibration Solutions: Pipette into separate 100 ml volumetric flasks 2, 4, 6, 8, 10 and 12 ml of the standard sodium solution (100 ppm Na), make up to the mark with water and mix thoroughly. The resultant calibration solutions contain respectively 2, 4, 6, 8, 10 and 12 ppm of sodium.

Calibration Curve: Aspirate into the flame of the spectrophotometer a "blank" consisting of the water used for the preparation of solutions and record the detector response.

Repeat with the calibration solutions described under "Calibration Solutions" and plot the graph of sodium concentration against detector response.

Test Procedure: Weigh out 5 ± 0.05 g of the sample into a clean platinum dish and add 1 ml of concentrated sulphuric acid. Warm the platinum dish and contents for 30 minutes on a steam bath and then transfer it to a pipe clay or silica triangle supported upon a tripod in a fume cupboard. Heat with a Bunsen flame adjusted to 4 to 5 cm in length and, as soon as the contents of the crucible begin to fume, apply the flame to the surface; repeat this treatment until the TNT catches fire. Discontinue heating while TNT burns strongly, but apply the burner as necessary towards the end of the combustion in order to carry it to completion. Heat gently to drive off the sulphuric acid and then heat the dish in a muffle furnace at 600°C to remove carbonaceous material. Cool the dish and dissolve the residue in water, transfer the solution quantitatively to a 25 ml volumetric flask, make up to the mark with water and mix thoroughly.

Spray the solution into the flame of the spectrophotometer under the same conditions as were used in the calibration process and record the detector response. From the calibration curve obtain the corresponding concentration of sodium in the test solution.

Calculation: Percentage sodium in sample = $C \times 5 \times 10^{-4}$

where:

C = concentration of sodium in sample solution (under "Test Procedure").

i. Scum, Suspended Matter and Sediment

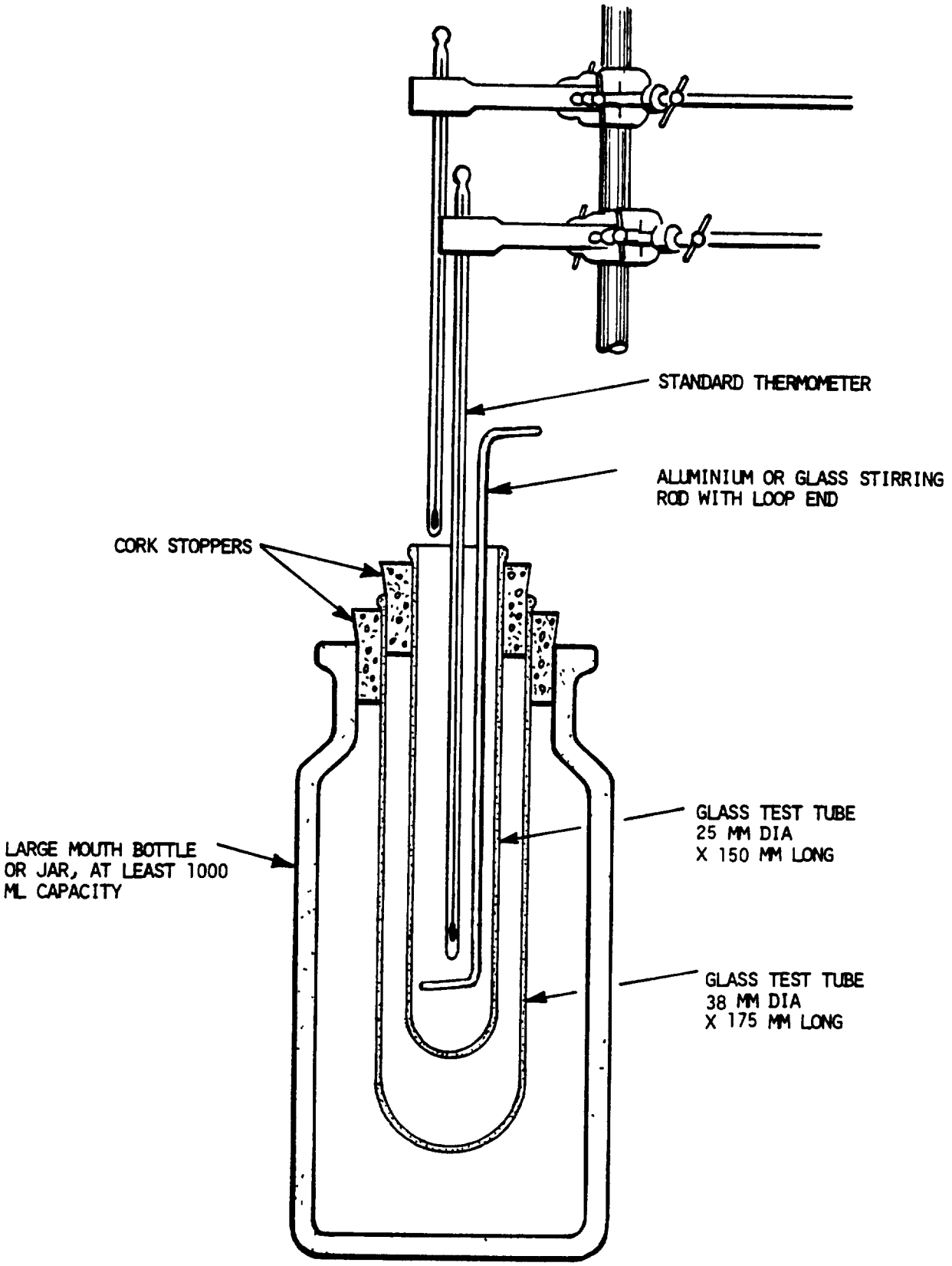
Melt at least 50 g of the sample in a beaker or porcelain dish and examine the liquid for the presence of scum, suspended matter or sediment.

IMPLEMENTATION OF THE AGREEMENT

9. This STANAG is implemented when a nation has issued the necessary orders/instructions putting the contents of this agreement into effect.

FIGURE 1

TNT SOLIDIFICATION POINT APPARATUS



RATIFICATION AND IMPLEMENTATION DETAILS
STADE DE RATIFICATION ET DE MISE EN APPLICATION

NATION	NATIONAL RATIFICATION REFERENCE DE LA RATIFICATION NATIONALE	NATIONAL IMPLEMENTING DOCUMENT NATIONAL DE MISE EN APPLICATION	IMPLEMENTATION/MISE EN APPLICATION					
			FORECAST DATE DATE PREVUE			ACTUAL DATE DATE REELLE		
			NAVY MER	ARMY TERRE	AIR	NAVY MER	ARMY TERRE	AIR
BE	GS 5673 of/du 20.11.85					11.91	11.91	11.91
CA								
DA	M.204.69-S.4025-MAS-23257 of/du 7.10.85					10.85	10.85	10.85
FR								
GE	BMVg Fü S IV 1 Az 03-51-60 of/du 30.10.85		3.92	3.92	3.92			
GR								
IT								
LU	GS 5673 of/du 20.11.85						11.91	
NL	M85/0310/12281 of/du 14.10.85					11.91	11.91	
NO	MAS-705/86/B/HST ORG 2/BO/EN/STANAG 4025 of/du 2.5.86					1.87	1.87	1.87
PO								
SP								
TU								
UK	ARM 1/2/1 of/du 20.9.85		5.92	5.92	5.92			
US*	AMSAC-MC/S of/du 4.9.87	MIL-T-248C				11.91	11.91	11.91

*See reservation overleaf/
Voir réserve au verso

RESERVATIONS/RESERVES

UNITED STATES: The U.S. maximum acidity will be .02 percent.

ETATS-UNIS : L'acidité maximum des Etats-Unis sera de .02%