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

NATO STANDARDIZATION AGENCY (NSA)
AGENCE OTAN DE NORMALISATION (AON)
1110 BRUSSELS

29 April 2002 NSA/0466-PPS/4230

See CNAD AC/310 STANAG distribution

STANAG 4230 PPS (EDITION 1) – EXPLOSIVES, SPECIFICATION FOR HNS (HEXANITROSTILBENE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Reference: AC/310-D/182, dated 18 August 2000

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

2. The reference listed above is 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, advise the Defence Support Division through their national delegation as appropriate of their intention regarding its ratification and implementation.

Jan H ERIKSEN
Rear Admiral, NONA
Director, NSA

Enclosure:
STANAG 4230 (Edition 1)

STANAG 4230
(Edition 1)

NORTH ATLANTIC TREATY ORGANIZATION
(NATO)

NATO STANDARDIZATION AGENCY
(NSA)

STANDARDIZATION AGREEMENT
(STANAG)

SUBJECT: EXPLOSIVES, SPECIFICATION FOR HNS (HEXANITROSTILBENE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Promulgated on 29 April 2002

Jan H ERIKSEN
Rear Admiral, NONA
Director, NSA

RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature

EXPLANATORY NOTES

AGREEMENT

1. This NATO Standardization Agreement (STANAG) is promulgated by the Director, NSA 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 "In NATO Standardization, the fulfilment by which a member nation formally accepts, with or without reservation, the content of a Standardization Agreement" (AAP-6).

5. Implementation is "In NATO Standardization, the fulfilment by a member nation of its obligations as specified in a Standardization Agreement" (AAP-6).

6. Reservation is "In NATO Standardization, the stated qualification by a member nation that describes the part of a Standardization Agreement that it will not implement or will implement only with limitations" (AAP-6).

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.

FEEDBACK

8. Any comments concerning this publication should be directed to NATO/NSA - Bvd Leopold III, 1110 Brussels - BE.

STANAG 4230
(Edition 1)

NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT
(STANAG)

EXPLOSIVES, SPECIFICATION FOR HNS (HEXANITROSTILBENE) FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

Related Document: None

AIM

1. The aim of this agreement is to ensure that hexanitrostilbene (HNS) shall possess properties that make it suitable for military use and to provide, within NATO, an acceptable basis for the procurement and certification of HNS.

AGREEMENT

2. Participating nations agree that HNS, proposed for military use, shall meet all the physical and chemical requirements of Table I of this document for HNS-Type I, II, or III. The test procedures used to verify the requirements of Table I are described in Part III (paragraphs 7 through 19, inclusive) and the rejection criteria shall be in accordance with Part II, paragraph 5 of this document.

a. Use. HNS-I and HNS-II are intended primarily for use in man-rated systems, including space applications and escape systems for high-speed aircraft. Typically, HNS-I is used in the booster tips of shielded mild detonating cords (SMDC) and flexible linear shaped charges (FLSC), while HNS-II is used in SMDC and FLSC transfer lines. HNS-III is intended for use in strictly non man-rated applications as, for example, use as a TNT crystal growth modifier.

b. **WARNING**. This STANAG calls for the use of substances and test procedures that may be injurious to health if adequate precautions are not taken. It refers only to technical suitability, and in no way absolves the user from the statuary obligations relating to health and safety at any stage during use.

DETAILS OF THE AGREEMENT

PART I - PHYSICAL AND CHEMICAL PROPERTIES

3. Physical and Chemical Properties. Test procedures for the determination of the physical and chemical properties, for each type of HNS Explosive, shall be as specified in Table I. The values given for HNS-Type I and Type II reflect standards derived from data collected on samples of HNS loaded and tested in explosive ordnance systems. Limits placed on these values are above those found to be acceptable from material exposed to the elevated temperature at or below 190°C for 50 hours. Use of the material above 190°C should be considered only after an evaluation of data acquired in tests described herein and in accordance with the limits prescribed in this specification.

Table 1. Physical and Chemical Requirements

REQUIREMENT	VALUE FOR HNS TYPES			TEST PROCEDURE	
	I	II	III	HNS-I/II	HNS-III
A VACUUM STABILITY (NOTE 1):	3.o (max)	0.6 (max)	NR**	7	
(a) Milliliter per gram for first 20 minutes	1.1 (max	0.6 (max	NR	7	
(b) Milliliter per gram per hour for additional 2 hours					
B SURFACE MOISTURE/VOLATILES: (percent by weight)	0.05 (max)	0.05 (max)	NR	8	
C. WATER-SOLUBLE MATERIAL: (percent by weight)	0.2 (max)	0.03 (max)	0.2 (max)	9	15
D. INSOLUBLE MATERIAL IN DMF (NOTE 2): (Percent by weight)	0.1 (max)	0.03 (max)	NR	10	
E. CHEMICAL ANALYSIS (NOTE 3): (HNBiB) (percent by weight)	5.0 (max)	2.0 (max)	NR	11	
Other Impurities (percent by weight)	0.6 (max)	0.05 (max)	NR	11	
F. SURFACE AREA ANALYSIS (NOTE 4): (cm^2/cm^3)	30,000 (min)	10,000 (max)	NR	12	
G. MELTING POINT (°C):	310 (min)	310 (Min)	310 (min)	13	13
H. ALKALINITY (Na_2CO_3): (percent by weight)	NR	NR	nil		14
I. ACIDITY (H_2SO_4): (percent by weight)	NR	NR	0.05 (max)		14
J. MATTER INSOLUBLE IN DMF/γ-BUTYROLACTONE: (PERCENT BY WEIGHT)	NR	NR	0.1 (max)		16
K. GRITTY MATTER INSOLUBLE IN AQUA REGIA:					
(a) Retained on a 63 micron BS sieve*: (percent by weight)	NR	NR	0.01(max)		17
(b) Retained on a 250 micron BS sieve*: (percent by weight)	NR	NR	nil		17
L. SULPHATED ASH: (percent by weight)	NR	NR	0.02 (max)		18
M. SIEVING: Material retained on a 125 micron BS sieve*: (percent by weight)	NR	NR	Nil		19

EXPLANATION OF TABLE I.

* Particulars of the sieves shall be found in British Standard 410.
** Not required.

Notes:

1. Specified values are volumes of gas calculated at 0 °°C and 760 mm pressure after evolution at 260 ± 0.5 °C.
2. DMF is dimethyl formamide.
3. HNBiB is hexanitrobibenzyl.
4. cm^2/cm^3 is square centimeter per cubic centimeter.

PART II - SAMPLING AND REJECTION CRITERIA

4. Sampling. Fifty-gram samples for HNS-I and HNS-II and 500-gram samples for HNS-III should be selected at random from each inspection lot as follows:

Inspection Lot Size in Kilograms		Number of Samples
From	To and Including	
0.0-	7.3	2
7.4-	24.5	3
24.6-	45.5	4

5. Rejection Criteria. Failure of any specimen or sample of HNS Explosive to meet the requirements specified herein shall cause rejection of the lot of HNS Explosive material from which it was taken.

PART III - TESTING METHODS

6. Physical and Chemical Tests. Adequate safety precautions shall be taken during the processing, testing, and handling of the HNS Explosive to protect personnel from accidents, fires, or explosions, and to limit damage to equipment and processing areas.

7. Vacuum Thermal Stability

a. Definition. The vacuum thermal stability (VTS) of an explosive is the volume of gas, at standard conditions, produced by the constant temperature, constant volume thermal decomposition of the explosive using a specified apparatus, heating period, explosive weight, and an initial pressure of 2 mm Hg (266 Pa) or less. For HNS, the weight of explosive used is 0.2 g; the temperature is 260 ± 0.5^{o}C; the heating period is as specified in Table I; the volume of the sample tube is 10 cm^{3}; and the standard conditions are 1 atmosphere and 0^{o}C (0.101 MPa and 273.2^{o}K).

b. Specimen Preparation. Prior to sampling, the batch or lot of the candidate explosive chosen to provide the material for the specimen shall be dried for 16 hours at a pressure of 2 mm Hg (266 Pa) or less at ambient temperature. A representative portion of the dried explosive (0.2 grams) shall be transferred to the bottom of the test chamber using a powder funnel.

c. Test Apparatus. Figure 1 depicts the type of apparatus used to measure the vacuum thermal stability of HNS. It should be fabricated from a heat resistant glass, such as Pyrex^{R}. A heating block, controlled at 260 ± 0.5^{o}C, is required to heat the test chamber in which the tube containing the sample is placed.

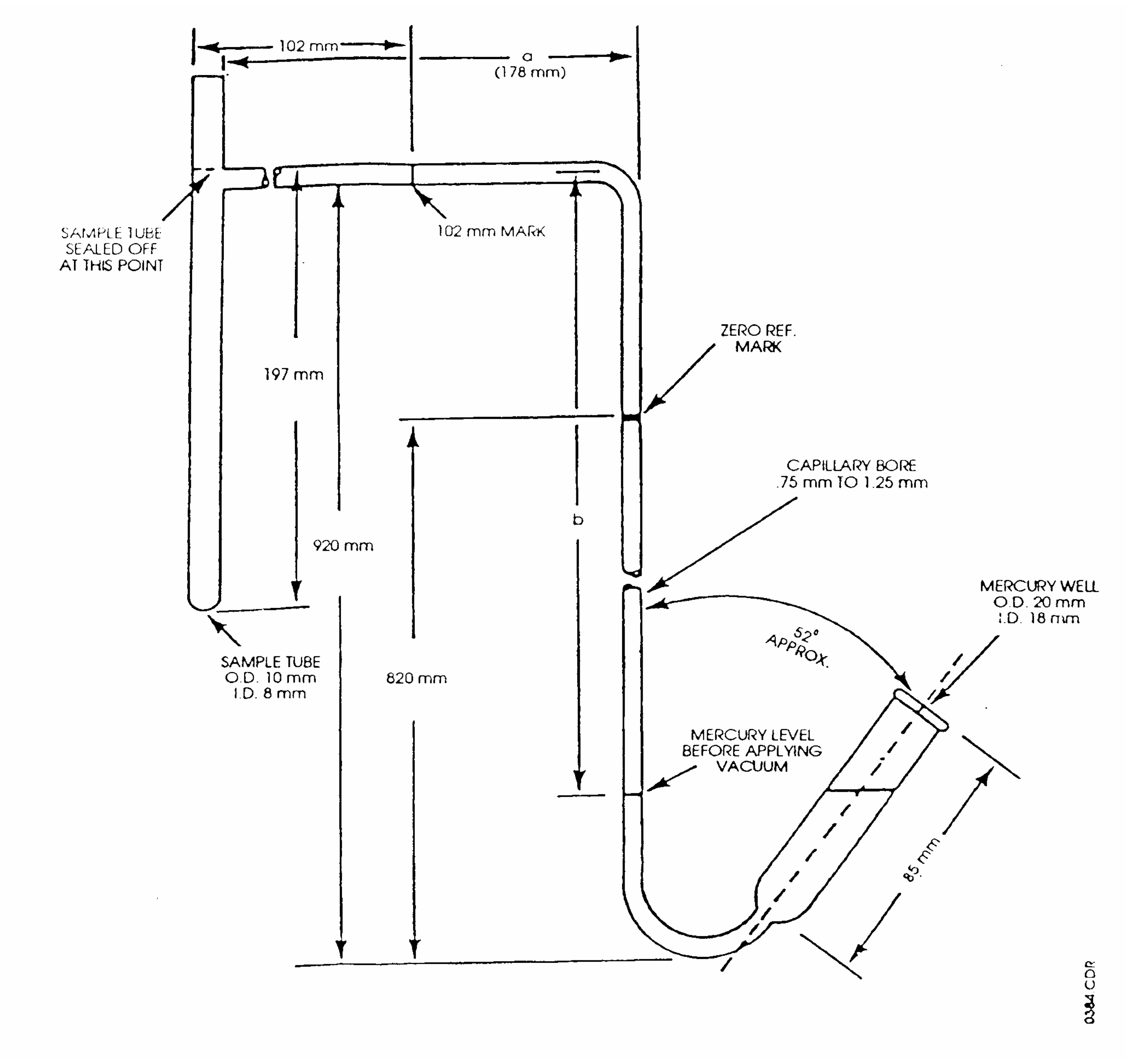

FIGURE 1. DIMENSIONS OF VACUUM THERMAL STABILITY SAMPLE TUBE

d. Procedure for Measuring the Volume of the Test Chamber. Mark the position on the riser where the leak-proof seal will be made. Determine the weight of water required to fill the test chamber to this mark. Convert this weight to a volume using the accepted value for the density of water at the temperature of the determination. Record this volume as "A" (in cm^3). Determine the volumetric capacity of the capillary tubing, in cm^3 per cm length by weighing the amount of mercury required to fill a measured length of the tubing. Convert this weight to a volume using the accepted value for the density of mercury at the temperature of the determination. Record this value as "B" (in cm^3) per cm length.

e. Test Procedure. Place the specimen prepared according to paragraph 7.b. in the test chamber. Make a leak-proof seal on the riser at the calibration mark (see paragraph 7.d.). Place triply distilled mercury in the mercury reservoir. Tilt the apparatus at an angle that allows the mercury to flow away from and to clear completely the point at which the capillary tubing is sealed to the mercury reservoir. The reservoir must be firmly supported to avoid stress on the glass seals. The test apparatus is then evacuated through the mercury reservoir to a pressure of 2 mm Hg (266 Pa) or less. During the evacuation, the reservoir should be tapped gently to dislodge any air bubbles that may be trapped in the mercury. While the apparatus is being evacuated, it is returned to the normal position (test chamber vertical). The evacuation line is then by-passed and air is slowly bled in until the height of the mercury column remains constant. If this height, h_o , remains constant for at least 12 hours, the test assembly is leak-proof and can be used to generate reliable VTS data. If the height of the mercury column drops, a leak is present; the mercury must be removed from the reservoir, the defective seal made leak-proof, and the mercury manometer reconstructed.

The test apparatus is then positioned so that the test chamber, riser, and the horizontal portion of the manometer are maintained at 260 ± 0.5^{o}C by a heating block. Immediately after the positioning of the test assembly, the time and barometric pressure, P_o , shall be recorded. After 20 minutes, the barometric pressure, P_{20} , and the height of the mercury column above the reference level, h_{20} , shall be recorded. The final reading shall be taken 2 hours and 20 minutes after the start of the test. The following data shall be recorded: The barometric pressure P_f ; the ambient temperature in ^{o}C, t_f ; the height of the mercury column above the reference level h_f . Report the volume of gas evolved per gram of specimen during the first 20 minutes, S_{20} , and the average volume of gas evolved per hour per gram during the following two-hour period, VTS.

f. Calculation of the 20-Minute Surge, S_{20} , for the Candidate Explosive. The 20-minute surge for the candidate explosive, S_{20} , shall be calculated from the following equation:

$$S_{20} = \frac{(h_o - h_{20} + P_{20} - P_o)\,(A - g/\rho + aB + bB)\ x\ 273.2}{(t_f + 273.2)\ x\ 760\ x\ g}$$

where: h_0 is the initial height of the mercury column in mm Hg;
P_0 is the initial barometric pressure in mm Hg;
and the other symbols have the definitions given in paragraph 7.g.

When S_{20} is calculated by the above equation, S_{20} will have the units of cm^3/g/20 minutes. Each specimen of HNS Explosive tested shall meet the requirements of Table I.

g. Calculation of the Vacuum Thermal Stability of the Candidate Explosive. The vacuum thermal stability (VTS) of the candidate explosive at 260^{o}C shall be calculated from the following equations:

$$VTS = \frac{(h_{20} - h_f + P_f - P_{20})(A - g/\rho + aB + bB)\ x\ 273.2}{2\ x\ (t_f + 273.2)\ x\ 760\ x\ g}$$

Where: h_{20} is the height of the mercury column above the reference level after 20 minutes, in mm Hg.

h_f is the height of the mercury column above the reference level after 2 hours and 20 minutes in mm Hg.

P_{20} is the barometric pressure after 20 minutes, in mm Hg.

P_f is the barometric pressure after 2 hours and 20 minutes in mm Hg.

A is the volume of the test chamber as determined by the procedure given in paragraph 7.d., in cm^3.

g is the weight of the specimen, in grams.

t_f is the temperature of test in ^oC.

ρ is the density of candidate explosive (determined following the drying procedure given in paragraph 7.b.) in g/cm^3.

B is the volumetric capacity of the capillary tubing (determined by the procedure given in paragraph 7.d.) in cm^3 per cm length.

a is the length designated by "a" in Figure 1, in cm.

b is the length designated by "b" in Figure 1, in cm.

When VTS is calculated by this equation, it will have the units of $cm^3/g/hr$.

8. Surface Moisture and Volatiles Content Test. One 5-gram specimen of HNS Explosive from each sample required by paragraph 4 shall be placed in a vitreous 100 milliliter (ml) tall form beaker and weighed. The beaker and contents shall be transferred to a vacuum oven, evacuated, and heated at 100 ± 5^oC for 2 hours. The beaker shall then be removed from the vacuum oven, placed in a desiccator until cool, and weighed. After weighing, the HNS sample shall be stored in a desiccator until further testing (see paragraph 9 below) is resumed. The surface moisture and volatiles content obtained by the difference in weight before and after heating shall be reported on a percentage basis. The surface moisture and volatiles content of each specimen of HNS Explosive tested shall meet the requirement of Table I.

9. Water-Soluble Material Content Test. The water soluble material content is determined on the same sample used in the surface moisture and volatiles content test of paragraph 8. The sample is washed three times by decantation. Use 40 ml of water containing 5 volume percent of methanol for each wash. Allow the sample to remain in contact with the water-methanol for 20 minutes. Stir the sample with a flat tipped stirring rod to aid in wetting the particles and break up lumps. All of the wash solution is filtered through a previously weighed 50 ml medium porosity, high form, sintered-glass crucible. After the sample has been quantitatively transferred to the crucible with water-methanol, it is dried to constant weight at $100^oC \pm 3^oC$, cooled, and weighed. After weighing, the HNS sample shall be stored in a desiccator until further testing (see paragraph 10 below) is resumed. The loss in weight (based on the original 5 g sample weight) is reported as water-soluble material. The water-soluble material content of each specimen of HNS Explosive tested shall meet the requirement of Table I.

10. Insoluble Material Content Test. The insoluble material content shall be determined by an extraction of the HNS Explosive (from the crucible used in the water-soluble determination) using hot dimethyl formamide (DMF). About 25 ml of near boiling DMF shall be added to the crucible and the sample stirred for about one minute. The extract is then removed by suction. The extraction is repeated seven additional times or until the filtrate from the crucible is clear. Dry the crucible to a constant weight at 100 ± 3^{o}C. The crucible is then cooled to room temperature and weighed. The increase in weight of the crucible shall be reported as percent insoluble matter. The insoluble material content of the HNS Explosive tested shall meet the requirement of Table I.

11. Chemical Analysis. The chemical analysis test shall be carried out on one specimen from each of the samples required by paragraph 4. The purity of HNS-Type I and HNS-Type II shall be determined by the following method:

a. Apparatus. A high performance liquid chromatograph (HPLC) equipped with a 254 nm ultraviolet (UV) absorbance detector and a signal recorder.

b. Materials. Analytical reverse-phase column (e.g., Whatman Partisil PXS10/25 ODS-2), methanol (HPLC grade), distilled water, dimethylsulfoxide (DMSO)-reagent grade, phenolphthalein (Merck U.S.P) or equivalent materials. HNS used as reference material shall pass the following requirements:

Melting Point - equal to or greater than 315^{o}C.

Vacuum Thermal Stability Test as described in paragraph 7.

HPLC: no other peaks visible in the chromatogram with the unit set at highest sensitivity setting, as described in paragraph 11.d.

NOTE: The following description of instrument parameters and sample size is given for information only, since each instrument and column will require conditions specific to the instrument used.

CAUTION: When using the following conditions, HNS samples of greater than 1.2 mg in 10 ml of DMSO tend to overload the analytical reverse-phase column. Therefore, final HNS concentrations should be kept below 1.2 mg per 10 ml.

Depending on the size of the HNS crystals, heating the HNS-DMSO mixtures on a steam bath may be necessary to obtain complete solution.

Since HNS and some of the HNS impurities are subject to photolysis, solid and/or solutions of HNS should be stored in the dark and in actinic glassware.

HNBiB used as reference material shall meet the following requirements:

Melting Point - equal to or greater than 218^{o}C.

HPLC: no other peaks visible in the chromatogram with unit set at highest sensitivity setting, as described in paragraph 11.d.

Since HNBiB is subject to photolysis, solid and/or solution of HNBiB should be stored in the dark and in actinic glassware.

c. Conditions. Range setting at appropriate sensitivity, mobile solvent phase 40 percent methanol and 60 percent distilled water by volume, isocratic solvent flow rate 2.0 ml per minute or best separation conditions for HPLC unit used.

d. Procedure. The sample solution and reference solution (made with HNS meeting the requirements of 11.b) shall be made by dissolving 10.0 mg of HNS, weighed to 0.1 mg, and 20 mg of phenolphthalein as the internal standard, in 10 ml of dimethysulfoxide (see Figure 2 - HPLC trace of HNS, internal standard and possible impurities). A 1.0 ml aliquot of the HNS sample and reference (the solvent phase of 40 percent methanol and 60 percent distilled water by volume) solutions shall be diluted to 10 ml with DMSO. A 25 micro-liter sample of diluted HNS solution is then injected into the HPLC with the flow rate set at 2.0 ml per minute, a 40% methanol and 60% distilled water solvent phase, and the recorder set at 0.5 cm per minute. To determine the percent of HNS impurities (e.g. HNBiB, etc.) in the sample, the HPLC UV detector shall be placed at the greatest sensitivity setting 0.005 absorbance units full scale (AUFS). The percentage of HNS, HNBiB, and all other impurities will be calculated from the integration of their respective peak areas relative to the internal standard. A 25 micro-liter sample of the reference solutions and DMSO solvent shall also be chromatographed, with the HPLC unit set at the greatest sensitivity, to determine the position and area of any peaks which are due to the HNBiB and other impurities present in the reference solution and the solvent alone. The percentages of HNBiB and all the other impurities will be calculated from the integration of their peak areas relative to the internal standard as described in paragraph 11.e. The impurity of each specimen of HNS-Type I and HNS-Type II Explosive tested shall meet the requirements of Table I.

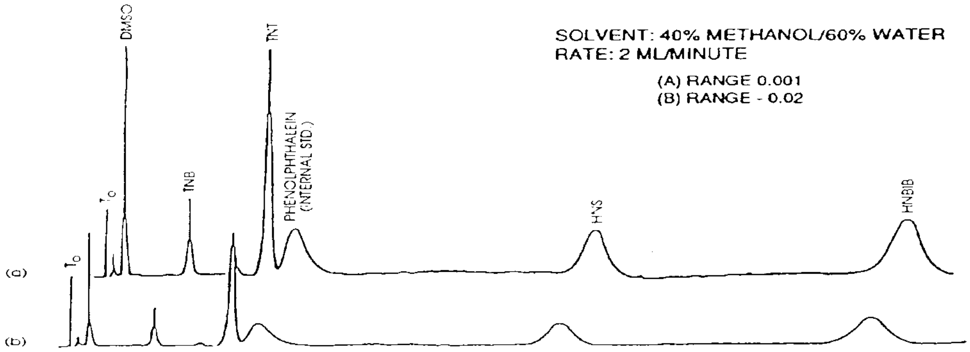

FIGURE 2. HPLC TRACE OF HNS AND POSSIBLE IMPURITIES

e. Calculations. The concentrations of the major components and any impurities shall be determined as follows:

$$A = \frac{\text{Area of X Component in Sample Solution}}{\text{Area of Internal Standard in Sample Solution}} = \underline{\qquad}$$

$$B = \frac{\text{Area of Internal Standard in Reference Solution}}{\text{Area of Known X in Reference Solution}} = \underline{\qquad}$$

$$C = \frac{\text{Weight of Known X in Reference Solution}}{\text{Weight of Internal Standard in Reference Solution}} = \underline{\qquad}$$

$$D = \frac{\text{100 X (Weight of Internal Standard in Sample Solution)}}{\text{Weight of Sample}} = \underline{\qquad}$$

$$A \times B \times C \times D = \underline{\qquad} \text{ \% of X (Component or impurity to be determined)}$$

12. Surface Area Analysis. The surface area analysis shall be carried out on one specimen from each of the samples required by paragraph 4. The surface area of HNS-Type I and HNS-Type II shall be determined by the following method:

a. Apparatus. The surface area of crystalline HNS-Type I and HNS-Type II is measured with a Micromeritics High Speed Surface Area Analyzer, model 2205 or equivalent, by determining the quantity of gas necessary to form a single layer (monomolecular layer) of gas on a representative sample. Using argon gas at the temperature of liquid nitrogen, the gas molecules are adsorbed (strongly attracted and held) on the surface of the sample. The volume of gas actually adsorbed between the ice/water temperature point and the temperature of liquid nitrogen is measured in the apparatus. The magnitude of this shift is a measure of the gas adsorbed as a monomolecular layer, hence the surface area of the sample.

b. Materials. Water pumped argon, liquid nitrogen, ice/water mixture, heat gun, vacuum oven.

NOTE: The following information on the determination of Surface Area is given only for the Micromeritics apparatus, since each instrument will require conditions specific to the instrument used.

c. Sample Size. - HNS-Type I - 0.5-1.5 gram sample
HNS-Type II - 3.0 gram sample
Duplicate samples shall be run.

d. Sample Preparation. Weighed samples must be dried in a vacuum oven at around 70^{o}C for 24 hours prior to analysis. The HNS sample will then be outgassed on the 2205 Unit for approximately 30 minutes at 125-150^{o}C.

e. Standards. Standard samples are prepared from B.E.T. surface area reference materials.

Material:	Fluid Catalyst Carrier
Surface Area:	0.49 ± 0.03 m^2g
Part No.:	004/16104/01 (Micromeritics)

Two standard samples weighed to measure between 20 to 40 counts (for Micromeritics apparatus) and 90 to 110 cts shall be prepared in 5 mm bore, 15 ml volume sample tubes and run on the Surface Area Analyzer. The measured surface areas shall agree to within 10 percent of the calculated values.

f. Procedure. The operational and analysis procedures contained in the Instruction Manual of the High Speed Surface Area Analyzer Model 2205 shall be followed.

g. Calculations. The surface area is calculated in square centimeters per cubic centimeter by the following formula:

$$cm^2 / cm^3 = \left(m^2 / g\right) x\ 10{,}000\ x\ 1.73$$

$$\text{where: } 10{,}000 = \text{conversion factor}$$

$$1.73 = \text{density of HNS}$$

$$m^2 / g = \text{meter reading (square meters)} / g \text{ (sample weight in grams)}$$

The surface area of each specimen of HNS Explosive tested shall meet the requirement of Table I.

13. Determination of Melting Point

a. Apparatus

(1) A melting point apparatus, which can be controlled to heat the sample at a rate of 1°C per minute.

(2) A suitably calibrated thermometer covering not less than the range 280°C to 330°C and graduated in 0.5°C divisions.

(3) Melting point tubes, thin walled and closed at one end.

b. Procedure. Introduce sufficient of the dried sample into a melting point tube to fill the tube to a depth of 10 millimeters (nominal size of melting point tube: 1.8 mm (OD)/1.6 mm (ID) x 90 mm). Adjust the control of the melting point apparatus to bring the temperature quickly to 280°C. Reduce the rate of heating to 1 to 2°C per minute and insert the melting point tube into the apparatus when the temperature reaches 300°C. Position the sample 2 to 3 mm adjacently to and vertically even with the mercuric bulb of the thermometer. Note the temperature when half the material is melted. Apply any correction necessary to the calibrated thermometer. Report the observed temperature to the nearest 0.5°C as the melting point. The temperature at which melting commences should not be more than 5°C lower than the reported melting point.

14. Determination of Acidity and Alkalinity. The dry material is ground in water and total acidity or alkalinity determined by titration.

a. Procedure. Transfer 5 ± 0.1g (W) of the dried sample to a 100 mm diameter, 50 mm deep ceramic mortar. Add 20 ml of distilled water and grind with a pestle until a smooth paste is obtained. Add a further 20 ml of distilled water and 1 ml of methyl red indicator. Titrate in the mortar using 0.02M sodium hydroxide solution or 0.02M hydrochloric acid solution as appropriate (T_1). Carry out a blank determination in a similar manner using 40 ml of distilled water and 1 ml of indicator (T_2).

b. Calculation and Reporting. Calculate the acidity (as H_2SO_4) or alkalinity (as Na_2CO_3) as a percentage by weight of the sample.

$$Percentage\ acidity = \frac{(T_1 - T_2) \times 49.04 \times M \times 100}{1000 \times W}$$

$$Percentage\ alkalinity = \frac{(T_1 - T_2) \times 52.99 \times M \times 100}{1000 \times W}$$

Note: M = Molarity of solution hydroxide or hydrochloric acid solution used.

15. Determination of Matter Soluble in Water. A thoroughly dried portion of the sample is extracted with water containing a little methanol to aid the wetting of the material. The water soluble matter content is calculated from the loss in weight.

a. Reagent. Water/methanol mixture 95/5 V/V.

b. Procedure. Wash a grade P40 (as defined in ISO 4793) sintered glass crucible with water followed by acetone and dry at 103 ± 2°C for 30 minutes. Cool the crucible for not less than 30 minutes and weigh accurately (W_1). Transfer 5 ± 0.1g of dried HNS to the crucible and redry the crucible and contents at 103 ± 2°C for 2 hours. Cool for not less than 30 minutes and weigh (W_2). Add 30 ml of the water/methanol mixture to the crucible and stir carefully with a small stirring rod to break up any lumps. Stand the crucible, with the stirring rod, in a dish to which water has been added to come up to the same level as the liquid in the crucible. Allow to stand for 20 minutes, and then remove the liquid from the crucible by suction. Add 20 ml of water/methanol to the crucible, stir well and allow to stand in the water bath with the liquid adjusted to the level of the liquid in the crucible for a further 20 minutes. Remove the liquid from the crucible by suction. Repeat this process once more with 20 ml of water/methanol, washing all the HNS from the stirring rod into the crucible, and removing as much liquid as possible by suction. Dry at 103 ± 2°C for at least 4 hours, cool for at least 30 minutes, and weigh (W_3).

c. Calculations and Reporting. Calculate the water soluble matter and report as a percentage by weight of the sample.

$$Percentage\ water\ soluble\ matter\ content = \frac{(W_2 - W_3) \times 100}{(W_2 - W_1)}$$

16. Determination of Matter Insoluble in DMF/γ-Butyrolactone. The HNS is dissolved in DMF, the solution is filtered and the residual insoluble matter is washed with γ-butyrolactone and acetone then dried and weighed. To aid filtration, a diatomaceous filter such as "Hyflo" supercel is used.

a. Preparation of Filter Aid. Place approximately 100g of the filter aid into a 400 ml glass beaker and add 200 ml of DMF which has been filtered through a Whatman No. 41 filter paper. Heat on a steam-bath for 20 minutes stirring occasionally then filter through a Whatman No. 41 filter paper held in a Buchner type funnel using suction. Wash with a further 100 ml of hot DMF and suck as dry as possible. Extract the material with two separate 100 ml portions of hot γ-butyrolactone which has been filtered through a Whatman No 41 filter paper. Wash the now extracted material with similarly filtered acetone and suck as dry as possible. Dry in a 103 ± 2°C oven for 2 hours.

b. Procedure. Place approximately 2g of prepared filter aid in a grade P16 (as defined in ISO 4793) sintered glass crucible. Add cold, filtered acetone to the crucible, stir, allow to settle, then remove the acetone using suction. Dry the crucible for one hour at 103°C, cool for not less than 30 minutes and weigh accurately (W_1). Replace the tared crucible into the oven. Weigh 5 ± 0.lg of the dried sample and put into a 600 ml glass beaker. Add 300 ml of DMF, cover with a watch-glass and heat on a steam-tray stirring occasionally, until the HNS has dissolved. Filter the hot solution through the hot tared crucible using suction, taking care not to disturb the filter aid.

Wash out the beaker with two 20 ml portions of hot DMF followed by three 20 ml portions of hot acetone sucking dry after each portion and ensuring that any insoluble residue is transferred from the beaker to the crucible. Wash the contents of the crucible with three 20 ml portions of hot γ-butyrolactone followed by three 20 ml portions of hot acetone, using suction. Dry the crucible for 1 hour at 103 ± 2°C, cool for not less than 30 minutes and weigh (W_2).

c. Calculation and Reporting. Calculate the insoluble matter content and report as a percentage by weight of the sample.

$$\textit{Percentage material insoluble in DMF} / \gamma\textit{-butyrolactone} = \left(W_2 - W_1\right) \textit{x } 20.$$

17. Determination of Gritty Matter. Gritty matter is defined as any hard material larger than 63 microns in size, that is insoluble in aqua regia.

a. Apparatus

(1) 200 mm diameter, 63 micron B.S. sieve.

(2) 50 mm diameter, 63 micron B.S. sieve.

(3) 50 mm diameter, 250 micron B.S. sieve.

(4) small platinum crucible.

(5) stainless steel 500 ml beaker.

(6) 100 ml glass beaker.

b. Reagents

(1) Methanol filtered through a 63 micron B.S. sieve.

(2) Aqua regia. Mix 1 volume of concentrated nitric acid (16M) with three volumes of concentrated hydrochloric acid (11M) immediately before it is to be used.

(3) γ-butyrolactone.

(4) Distilled water filtered through a 63 micron B.S. sieve.

(5) 5M sodium hydroxide solution.

(6) Phenolphthalein indicator solution.

c. Procedure. Using well-mixed wet material, weigh the calculated equivalent of 50g of dry material into a 500 ml stainless steel beaker. Add 200 ml of filtered methanol and stir, using a metal rod, until a thin uniform slurry is obtained. Transfer a portion of the slurry to a 200 mm diameter 63 micron B.S. sieve. Using a jet of tap water which has been filtered through a 63 micron sieve and with the aid of a clean 2.5 cm wide camel hair brush wash the HNS through the sieve. Repeat with successive portions until all the slurry has been sieved. Allow the sieve to drain. Wash any residue on the sieve back into the stainless steel beaker using a jet of acetone. Add 100 ml of γ-butyrolactone to the beaker, stir well and heat on a steam-bath until any residual sample has dissolved. Cool and pour through a 50 mm diameter 63 micron B.S. sieve. Rinse the beaker with acetone, pouring the rinses through the sieve then wash the sieve with acetone and allow to dry. Transfer the residue from the sieve quantitatively, to a small platinum crucible. Ignite for 5 minutes at 800°C. Transfer any residue to a 100 ml glass beaker, add 5 ml of aqua regia and boil for 5 minutes on a hot plate. Allow the beaker to cool, dilute to 25 ml with filtered, distilled water and neutralize to phenolphthalein using 5M sodium hydroxide solution. Pour the neutralized liquid through a 50 mm diameter 63 micron B.S sieve and wash the sieve and any residue first with water and then acetone. Dry the sieve and residue and quantitatively transfer the residue to an accurately weighted watch-glass (W_1) and reweigh (W_2). Transfer the residue to a 50 mm diameter 250 micron B.S. sieve and brush lightly with a small camel hair brush until no more material passes through the sieve. Count the number of particles which are retained on the 250 micron sieve (N).

d. Reporting. Grit retained on a 250 micron B.S. sieve = N particles/50g.

$$\text{Percentage grit retained on a 63 micron B.S. sieve} = 2(W_2 - W_1).$$

18. Determination of Sulphated Ash. The dry material is warmed gently with sulphuric acid and then heated more strongly to burn off the organic matter. The reaction is very vigorous and great care should be exercised. The reaction must be carried out behind a suitable safety screen and protective goggles and gloves must be worn.

a. Procedure. Heat a clean 100 mm diameter silica dish in a muffle furnace at 700°C for 30 minutes. Cool in a desiccator for 45 minutes and weigh accurately (W_1). Transfer 5 ± 0.1g of the dry material into the dish and carefully add 10 ml of concentrated sulphuric acid (18M) so that it is evenly distributed over the surface of the sample. Warm the dish and contents on a steam bath for 30 minutes to ensure that the sample is completely wetted by the acid. With a Bunsen flame gently apply heat to the dish held on a clay triangle until the sample begins to char. Remove the flame and allow the reaction to proceed unaided. As the reaction subsides, apply sufficient heat to sustain the reaction until all the sample has charred. Continue heating gently until no more sulphuric acid fumes are evolved. Heat the dish to dull red until all residual carbon has been burned off. Place in the muffle furnace at 700°C for 30 minutes. Cool the dish in a desiccator for 45 minutes and reweigh (W_2).

b. Calculation and Reporting. Calculate the sulphated ash content and report as a percentage by weight of the sample.

$$\text{Percentage sulphated ash} = (W_2 - W_1) \times 20.$$

19. Determination of Material Retained on a 125 Micron Sieve. This test may be performed in conjunction with the examination for grit (see 17.) by sieving through a 125 micron B.S. sieve prior to sieving through the 63 micron sieve. The test is carried out on 50g of material using a wet sieving procedure.

a. Procedure. Dry a 200 mm 125 micron B.S. sieve at 103 ± 2°C for 30 minutes. Cool and weigh (W_1). Using the wet material, transfer the equivalent of 50g of dry material to a stainless steel beaker of 500 ml capacity. Add 200 ml of methanol which has been filtered through a 63 micron B.S. sieve. Stir with a metal rod until a smooth slurry is obtained and transfer a portion of the slurry to the 125 micron sieve. With the aid of a jet of filtered tap water and a clean 2.5 cm wide camel hair brush, wash the HNS through the sieve. Repeat with successive portions of slurry, finally washing the remains of the slurry onto the sieve. Dry the sieve at 103 ± 2°C for 2 hours, cool and weigh (W_2).

b. Calculation and Reporting. Calculate the weight retained on the 125 micron sieve and report this as a percentage of the dry sample.

$$\textit{Percentage material retained on a 125 micron B.S. sieve} = (W_2 - W_1) \times 2.$$

PART IV - GENERAL INFORMATION

20. HNS-Type I/III. HNS is usually prepared from the reaction of TNT with aqueous sodium hypochlorite in tetrahydrofuran/methanol at 0°C to 5°C. HNS-Types I and III are materials which have not been subjected to recrystallization.

21. HNS-Type II. HNS-Type II is recrystallized HNS-Type I.

22. Hexanitrobibenzyl (HNBiB). HNBiB is the major impurity in HNS-Type I.

23. High Temperature Degradation. The presence of small quantities of impurities in the HNS Explosive can seriously degrade its high temperature stability characteristics.

IMPLEMENTATION OF THE AGREEMENT

24. This STANAG is considered implemented by a nation when that nation has issued the necessary orders and instructions putting the contents of this agreement into effect.