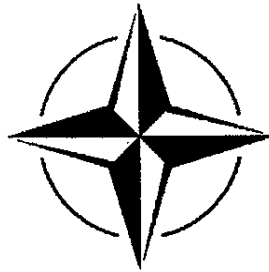


NORTH ATLANTIC TREATY ORGANIZATION  
(NATO)

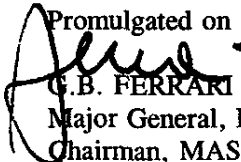


MILITARY AGENCY FOR STANDARDIZATION

(M A S)

# STANDARDIZATION AGREEMENT

SUBJECT: HMX (OCTOGENE), PHYSICAL AND CHEMICAL REQUIREMENTS,  
SPECIFICATION FOR DELIVERIES FROM ONE NATO NATION TO  
ANOTHER

Promulgated on 24 October 1996  
  
G.B. FERRARI  
Major General, ITAF  
Chairman, MAS

STANAG 4284  
(Edition 1)

RECORD OF AMENDMENTS

N°	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.

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

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24 October 1996

MAS/324-PCS/4284

See Distribution List MAS No. 2

**STANAG 4284 PCS (EDITION 1) - HMX (OCTOGENE), PHYSICAL AND CHEMICAL REQUIREMENTS, SPECIFICATION FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER**

Reference:

AC/310-D/111 dated 1 march 1993

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 (and AP if applicable).

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, IS, through their national delegation as appropriate of their intention regarding its ratification and implementation.

  
G.B. FERRARI  
Major-General, ITAF  
Chairman, MAS

Enclosure:

STANAG 4284 (Edition 1)

NATO STANDARDISATION AGREEMENT  
(STANAG)

HMX (OCTOGENE), PHYSICAL AND CHEMICAL REQUIREMENTS,  
SPECIFICATION FOR DELIVERIES FROM ONE NATO NATION TO ANOTHER

- Annexes : A. : Table 1 : Physical and Chemical Requirements for  
                  type I HMX  
          B. : Table 2 : Physical and Chemical Requirements for  
                  type II HMX  
          C. : Table 3 : Physical and Chemical Requirements for  
                  type III HMX  
          D. : Test Procedures.

AIM

1. The aim of this agreement is to establish a minimum common specification for deliveries of HMX (octogene) from one NATO nation to another.

AGREEMENT

2. The participating countries agree that octogene manufactured by NATO countries for delivery to other NATO countries - except when the order is intended for special purposes - must fulfil the minimum conditions of Tables 1, 2 or 3 below.

3. The octogene shall consist essentially of cyclotetramethylene-tetranitramine (HMX). It must be in the form of beta crystals but may contain traces of alpha form crystals. It may contain cyclotrimethylene-trinitramine (hexogene or RDX).

4. The three grades of HMX are essentially distinguished by their RDX content :

type I	RDX level 7 %	maximum
type II	RDX level 2 %	"
type III	RDX level 0,2 %	"

GENERAL

Manufacturing process

5. All information on the proposed manufacturing process must be provided in confidence at the request of the responsible bodies in the purchasing country. Any deviation from this accepted process must be noted and the product thus manufactured must be put aside until the responsible bodies of the purchaser have decided whether to accept or reject it.

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Appearance and granulation

6. The product shall be in the form of white crystals of various shapes. In his order the purchaser must clearly specify the type of product he requires.

7. As the particle size distribution is a major characteristic of the product, this data may be specified by the purchaser.

Definition of Lot

8. A lot shall consist of the total quantity of cross-blended material. For noncross-blended material a lot shall consist of the quantity produced in a single batch, or when manufactured by a continuous process a lot shall consist of the total quantity offered for acceptance at one time.

Sampling

9. A representative sample (s) of at least 200 g shall be taken from each lot by a sampling procedure which has been agreed by the purchasing authority.

IMPLEMENTATION OF THE AGREEMENT

10. This STANAG will be implemented when a country has given the necessary orders to enforce it.

TABLE 1  
Physical and chemical requirements of type I HMX

Characteristics	Value	Test Method See Annex D para
RDX Content (%)	7 max	2 or 3
Alpha HMX content (%)	0,5 max	3 or 6
Melting point (°C)	277 min	8
(see note)	275 min	9
Insoluble Matter content (%)	0,05 max	11 or 12
Number of gritty particles (particles per 50 g)		15 or 16
>0,42 or 0,50 mm	nil	
> 0,25 mm	5 max	
Inorganic matter (ash) (%)	0,03 max	18 or 19
Acidity (meq/kg)	3,3 max	20 or 21

note : when measuring the melting point only one of these specifications - which are deemed to be equivalent - should be followed.

Remark : at the purchaser's request the manufacturer must be able to ensure by means of an appropriate test that the explosive has a suitably low sensitiveness guaranteeing the absence of sensitizing products.

TABLE 2  
Physical and chemical requirements of type II HMX

Characteristics	Value	Test Method See Annex D para
RDX Content (%)	2 max	2 or 3
Alpha HMX content (%)	0,5 max	3 or 6
Melting point (°C) (see note)	282 min 277 min 275 min	7 8 9
Insoluble Matter content (%)	0,05 max	11 or 12
Number of gritty particles (particles per 50 g)		15 or 16
> 0,42 or 0,50 mm	nil	
> 0,25 mm	5 max	
Inorganic matter (ash) (%)	0,03 max	18 or 19
Acidity (meq/kg)	3,3 max	20 or 21

note : when measuring the melting point only one of these specifications - which are deemed to be equivalent - should be followed.

Remark : at the purchaser's request the manufacturer must be able to ensure by means of an appropriate test that the explosive has a suitably low sensitiveness guaranteeing the absence of sensitizing products.

TABLE 3  
Physical and chemical requirements of type III HMX

Characteristics	Value	Test Method See Annex D para
RDX Content (%)	0,2 max	2 or 4
Alpha HMX content		
a. sequential count of alpha crystals with maximum dimensions in the range 30 to 120 $\mu\text{m}$		5
Alpha crystals per 600 crystals....	2 max*	
Alpha crystals per 1000 crystals...	8 max*	
Alpha crystals per 2000 crystals...	20 max	
b. Alpha crystals exceeding 10 $\mu\text{m}$ minimum dimension or 120 $\mu\text{m}$ in maximum dimension.....	nil	5
c. Additional infra-red absorption over that produced by pure beta HMX in the spectral ranges 848 to 850 $\text{cm}^{-1}$ and 1030 to 1035 $\text{cm}^{-1}$	nil	6
Melting point ( $^{\circ}\text{C}$ )	270 min	10
Insoluble matter content (total (%))	0,05 max	13 or 14
(inorganic (%))	0,02 max	
Gritty particles. Grit retained on a : 63 $\mu\text{m}$ sieve (%)	0,01 max	17
250 $\mu\text{m}$ sieve (particles per 50 g)	nil	
Acidity (meq/kg)	1,7 max	20 or 21
Alkalinity (meq/kg)	4,0 max	20 or 21
Vacuum stability at 120 $^{\circ}\text{C}$ for 40 hours (cm <sup>3</sup> of gas/gramme)	1,0 max	22



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C-2

Characteristics	Value	Test Method See Annex D para
Cyclohexanone content (%)	0,2 max	23
Figure of Insensitiveness	55 min	24

\* If more than two or more than eight alpha crystals are counted at the appropriate sequential step, counting shall be continued within the limits of a maximum 20 alpha crystals per 2000 crystals.

TEST PROCEDURES

PARAGRAPH 1

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Acidity and Alkalinity

Determination of acidity or alkalinity

20

Determination of acidity

21

Vacuum stability

22

Determination of Cyclohexanone

23

Figure of Insensitiveness

24

TEST PROCEDURES

PARAGRAPH 2

RDX CONTENT - LIQUID PHASE CHROMATOGRAPHY METHOD

2.1 Principle

The HMX is dissolved in acetone, with an appropriate internal standard. Assay is performed by liquid phase chromatography (HPLC).

2.2 Reagent

- pure acetone, Analytical Reagent quality
- pure methanol, Analytical Reagent quality
- pure acetonitrile, for HPLC quality
- pure RDX
- pure HMX
- pure dimethylphthalate

2.3 Equipment

- high performance liquid phase chromatograph, with a flow rate self-regulated pump
- variable wave length U.V. detector
- potentiometric recorder
- data integrator, and data processing system
- 0,5  $\mu$ m porosity filtering assembly
- solvent degassing system
- common laboratory glass equipment

2.4 Procedure

2.4.1 Instrumental conditions

- eluant : methanol, water and acetonitrile in a 40/55/5 volume in degassed mixture
- column : silica gel support, with grafted C18 groups
- detector wave length : 220 nm

2.4.2 Standard solution preparation

Dissolve about :

- 10 mg of RDX
- 10 mg of HMX
- 10 mg of dimethylphthalate

weighed to within 0.01 mg, in 100 cm<sup>3</sup> of acetone.

#### 2.4.3. Preparation of the solution to be analyzed

Dissolve about 1 g of the HMX (or the RDX) to be analysed, weighed to within 1 mg, in 100 cm<sup>3</sup> of acetone, with about 10 mg of dimethylphthalate weighed to within 0,1 mg.

Filter through 0,5 µm porosity filters.

#### 2.4.4. Dosage

Inject 3 mm<sup>3</sup>.

Standard and tested solution injections must be performed under the same operating conditions.

#### 2.5. Expression of the results

Let :

$A_i$  be the height of the compound i peak  
 $A_E$  be the height of the dimethylphthalate  
 $m_i$  and  $m_E$  the respective masses.

Then, the response coefficient is

$$KF_i = \frac{m_i \times A_E}{m_E \times A_i}$$

Results : let M be the high explosive mass, used for dissolution :

$$\text{percentage of compound i} = \frac{m_E}{M} \times \frac{KF_i \times A_i \times 100}{A_E}$$

#### 2.6. Note : typical chromatogram

##### 2.6.1. Column

- Stainless steel
- length : 25 cm
- interior diameter : 4 mm
- filling : lichrosorb RP 18 \* 5 µm (Merck)

2.6.2. Experimental conditions

Eluant : methanol - water - acetonitrile 40-55-5  
Eluant flow rate : 0,8 ml/min  
Pressure at the column's top : about 200 bars  
Temperature : 35°C  
Wave length of detector : 220 nm  
Injected volume : 3 µl

2.6.3. Typical chromatogram

See page D-5

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TEST PROCEDURES  
PARAGRAPH 3RDY CONTENT/ALPHA HMX CONTENT - X RAY DIFFRACTION METHOD3.1. Principle

The alpha HMX, beta HMX, and RDX content of HMX are determined by X-ray diffraction. The patterns for alpha HMX, RDX, and beta HMX indicate characteristic diffraction at 17.710 degrees and 25.10 degrees two theta, respectively, for RDX and alpha HMX in the presence of beta HMX. Background intensities for RDX and alpha HMX can be measured at 16.90 degrees and 24.10 degrees two theta, respectively. Beta HMX, the major component, is determined by difference.

3.2. Reagents

- Acetone (50% aqueous solution)
- 1,2 Dichloroethane
- Diethylether
- Dimethylsulfoxide
- Distilled Water
- Glacial acetic acid
- HMX (from a high purity production batch)
- Nitric acid (70 % by weight)
- RDX (from a high purity production batch)
- Sodium acetate

3.3. Apparatus

3.3.1. X-Ray diffractometer. Philips Electronic Instrument X-Ray diffractometer (or equivalent) equipped with voltage and current stabilizer, Scintillation detector, and copper target tube. The energy source of the instrument should be capable of an excitation of 40 kV and a filament current of 20 ma. A pulse height analyzer capable of passing copper K radiation should be used. A nickel filter may be used to remove copper K radiation. Tube voltage and filament current can be selected by analyzing five of the calibration standards at two different settings.

3.3.2. Laboratory glassware and equipment

- Beaker (11)
- Medium Porosity crucible filter
- Vacuum oven
- Balance
- Wrist action shaker
- Fritted glass crucible (fine porosity)
- Filtering Flask
- Conical Flask
- Vacuum source
- 250 ml Erlenmeyer Flask
- Graph paper
- Mortar



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### 3.4. Procedure

3.4.1. Equipment Operation. The X-Ray equipment is operated in accordance with the manufacturer's instructions.

3.4.1.1. Beta HMX. Obtain a sample (approximately 1/2 pound) of a production batch of high purity HMX. Place the sample in a beaker and add four parts (by weight) of a buffer solution and heat at 90°C for two hours. (Buffer solution 6.0 cm<sup>3</sup> glacial acetic acid and 13.6 g sodium acetate diluted to one liter (pH = 4.6). Filter the slurry using medium porosity crucible. Dry the HMX at 100°C for two hours.

3.4.1.2. Alpha HMX. Add 4g of the purified beta HMX to 80 cm<sup>3</sup> of (70% by weight) nitric acid. Heat until the HMX dissolves. Filter the solution with filter paper and cool slowly to 30°C. After one hour, filter the slurry using a medium porosity crucible. Wash the precipitate thoroughly with distilled water. Dry in a vacuum oven at 60°C for two hours.

3.4.1.3. RDX. Obtain a 100 g sample from a production batch of high purity RDX. Heat the sample with four parts, (by weight) of buffer solution at 90° for two hours. The buffer solution is prepared as described in 4.1.1. Filter the slurry using a medium porosity crucible. Dry the RDX at 100°C for two hours. Heat 1 part by weight, RDX and 1-1/2 parts by volume dimethylsulfoxide (DMSO) to 92-96°C. If necessary add up to 1 additional part DMSO in order to dissolve completely all the RDX. Digest at 92-96°C for 30 minutes. Add distilled water until solution becomes cloudy. Reheat until the solution clear, then cool rapidly to room temperature and filter. Wash and dry a small sample of the precipitate for purity analysis by the EDC procedure as described in paragraph 4.2. Repeat the above procedure until a very pure product is indicated. Wash with 50% aqueous acetone. Dry at 100°C for 2 hours.

3.4.2. Purity analysis of RDX standard. Prepare RDX solvent by stirring 1,2 dichloroethane at room temperature for four hours in contact with HMX crystals. Solubility of HMX in 1,2 dichloroethane at room temperature (24°C) is 0.02 g per 100 cm<sup>3</sup>. Accurately weigh approximately 0.2 g of the RDX calibration standard (as per 4.3) to the nearest 0.1 mg into a tared 125 cm<sup>3</sup> glass stoppered conical flask. Add 100 cm<sup>3</sup> of 1,2 dichloroethane that has been saturated with HMX. Secure the glass stopper and shake the flask on a wrist action shaker for one hour. Accurately weigh a fine porosity 30 cm<sup>3</sup> fritted glass crucible and place it on a filtering flask. Apply vacuum to the filtering flask and transfer the contents of conical flask into the crucible with saturated 1,2 dichloroethane. Wash residue in the crucible 2 times with 100 cm<sup>3</sup> portions of diethylether.

Continue the application of vacuum for 15 minutes in order to dry the RDX. Place the crucible in a desiccator; allow it to come to room temperature and weigh crucible and insoluble residue. The insoluble residue is HMX. Therefore, the percentage of RDX in the sample is :

$$\frac{(W_g - W_r) \times 100}{W_g}$$

where : Wg - is original sample weight, g.  
Wr - is insoluble residue weight, g.

3.4.3. Calibration Standards. Preparation of calibration standard samples. Prepare 5 g samples of the calibration standard mixtures listed in Table I as follows. Accurately weigh to the nearest 0.1 mg. The ingredients listed in Table I (use purified materials which pass through a US STD Sieve No. 260) in the proper proportions to yield a 5.0 g sample. Place the ingredients in 250 cm<sup>3</sup> Erlenmeyer flasks and mix thoroughly for a minimum of three hours with a wrist action shaker.

Table I - Composition of calibration standard mixtures

Beta HMX, %	Alpha HMX, %	RDX, %
99.70	0.30	0.00
99.40	0.60	0.00
99.00	1.00	0.00
98.00	2.00	0.00
97.00	3.00	0.00
96.00	4.00	0.00
95.00	5.00	0.00
99.00	0.00	1.00
98.00	0.00	2.00
97.00	0.00	3.00
96.00	0.00	4.00
95.00	0.00	5.00
94.00	0.00	6.00
93.00	0.00	7.00
92.00	0.00	8.00
91.00	0.00	9.00
90.00	0.00	10.00

3.4.4 Calibration Procedure. Use the X-ray diffraction apparatus to measure the angular intensities of the calibration standard mixtures at 16.90, 17.81, 24.10 and 25.10 degrees two theta as outlined in 4.5. Use the X-ray diffraction data obtained for the calibration standard curves as indicated below.

RDX - Plot the corrected intensity (cps) at 17.81 degrees two theta vs RDX concentration, weight percent. (Curve I, page D-11).

Alpha HMX - Plot the corrected intensity (cps) at 25.10 degrees two theta vs. alpha HMX concentration, weight percent. (Curve II, page D-12).

Correction curve for determining alpha HMX in the presence of RDX. Plot the corrected intensity (cps) AT 25.10 degrees vs RDX concentration, weight percent, for those samples free of alpha HMX. (Curve III, page D-13).

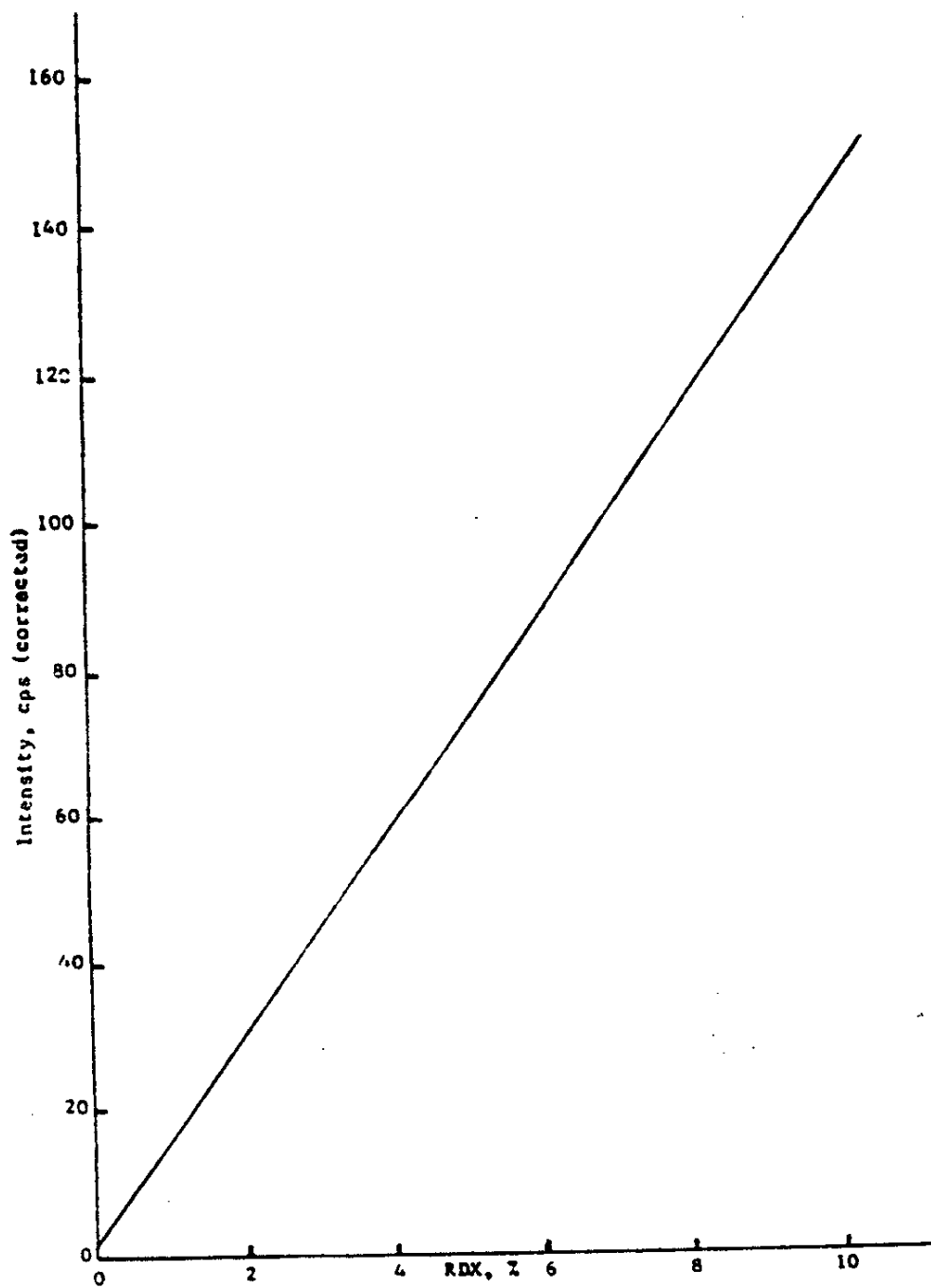
3.4.5. Preparation and Analysis of HMX sample. Reduce the HMX sample particle size to less than 62 micron. HMX particle size is usually small enough to permit analysis as received. It may, however, occasionally be necessary to crush lightly the material in a mortar. Recrystallized HMX requires grinding in a mortar to reduce the particle size. This is accomplished safely by grinding 0.1 g portions in small mortar. A sample size for grinding 0.4 g is required to properly fill the cavity type sample holder. Press sample into cavity of sample holder. Place aluminium sample holder (grooved side down) on a very smooth surface such as polished stainless steel. Place sample into cavity of holder and press with spatula. Add additional powdered sample and hand press by placing a stainless steel block over the cavity area and exerting pressure with hand 25 to 50 kg is sufficient). Remove sample and holder and examine surface of sample on grooved side of sample holder. Sample surface must not have any voids, cracks, etc. Remove loose explosive from sample holder. The grooved side of the sample holder must be completely free of particles prior to insertion into the diffractometer.

Intensity Measurement. Remove shield from diffractometer (shutters must be closed at all times when intensity measurements are not being made). Insert sample. The groove on a sample holder must be coincident with the groove on the goniometer axis of rotation. The rear edge of the sample holder must be flush against the sample stage. Replace the shield being careful not to move the sample. If the electronic panel is maintained ready, move toggle switch to the up position. Set diffractometer to 16.900 degrees two theta. Open shutters on X-ray tube tower by pulling out as far as possible.

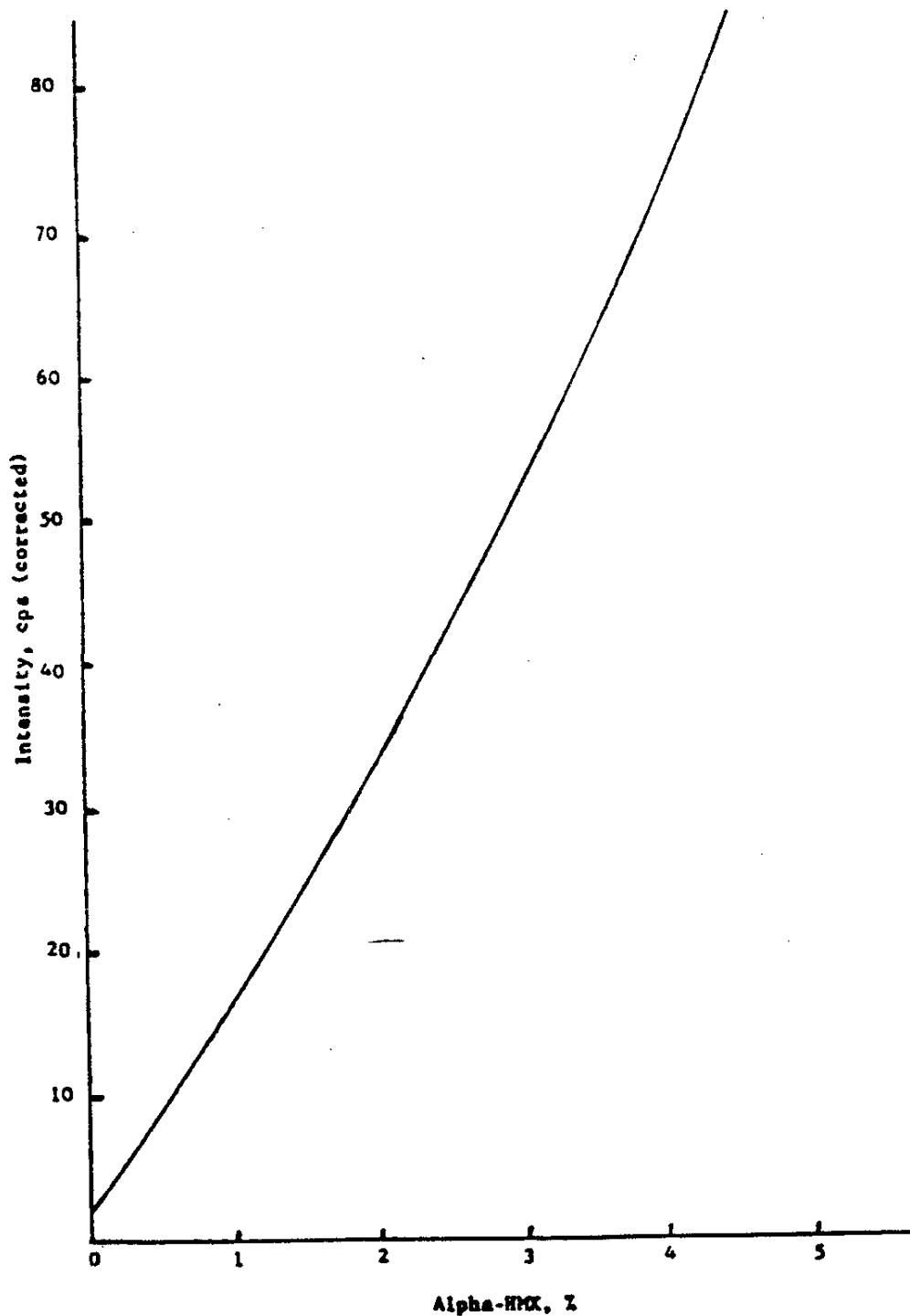
After the 30 seconds, push toggle switch toward scan to print out the time registered on rate meter dekatron tubes. Reset goniometer to 17.810 degrees two theta. Repeat above. Reset goniometer to 24.100 degrees two theta. Repeat above. Reset goniometer to 25.100 degrees two theta. Repeat. Close shutters on X-ray tower. When no additional samples are ready for analysis, turn off electronic panel by moving toggle switch downward.

### 3.5. Calculation and Reporting

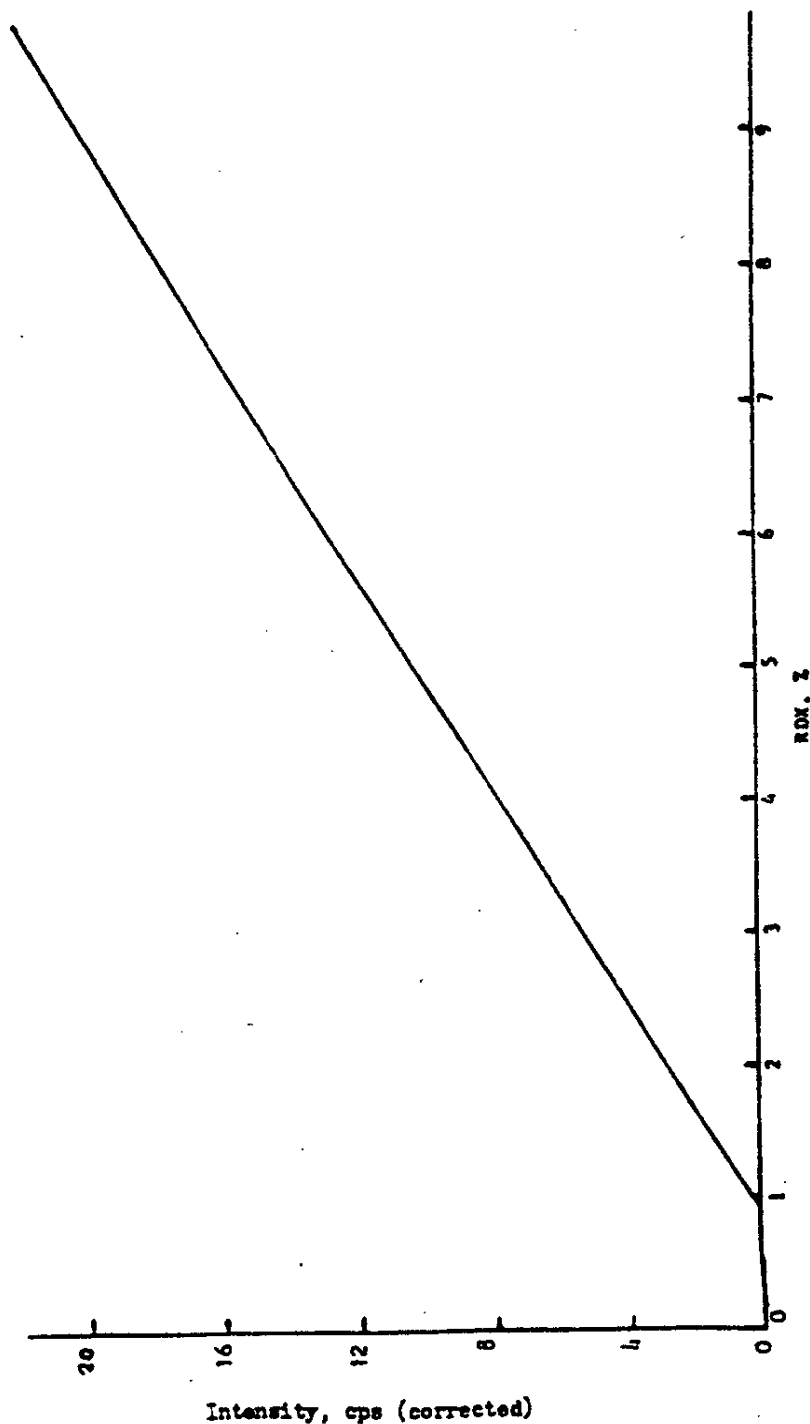
Interpretation of Data. Record the counts (times 10<sup>-1</sup>) that are accumulated in 100 seconds, that are printed out under the respective two theta angle. Place the decimal point one place to the left in the printout to obtain the representative counts per second (cps). Subtract cps at 16.900 degrees two theta from cps at 17.810 degrees two theta. This is the intensity (cps) due to the RDX percent. Opposite the cps obtained, read the RDX concentration from curve I (RDX calibration curve). Subtract cps at 24.10 degrees two theta from the cps at 25.10 degrees two theta. This intensity is due to alpha HMX. If the RDX concentration is greater than 1%, determine from curve III the correction required by reading the cps opposite the RDX percentage. Subtract this correction from the cps. From Curve II, read percentage alpha HMX composite the intensity (corrected or uncorrected).



CURVE I  
CALIBRATION CURVE FOR THE DETERMINATION OF RDX IN PRESENCE OF  
BETA HMX



CURVE II  
CALIBRATION CURVE FOR THE DETERMINATION OF ALPHA HMX IN PRESENCE OF  
BETA HMX



CURVE III  
ALPHA HMX ANGULAR INTENSITY CORRECTION AS A FUNCTION OF THE  
RDX CONCENTRATION

TEST PROCEDURES  
PARAGRAPH 4

RDX CONTENT - COLORIMETRIC METHOD

4.1. Principle

Interaction of RDX and sodium nitroprusside in alkaline aqueous acetone gives a green solution with an absorption band at 625 to 635 nm. Measurement of the optical density affords the method of determination. Pure HMX develops no colour under the conditions chosen. A calibration graph is prepared with known quantities of RDX in HMX. It is necessary to adhere rigidly to the conditions for the development of the colour.

4.2. Apparatus

A suitable UV/visible spectrophotometer.

Ice/water-bath

4.3. Reagents

Aqueous acetone, 50 per cent. Mix equal volumes of water and acetone, both of which should be  $20 \pm 2^\circ\text{C}$ .

Sodium nitroprusside solution. Prepare fresh each day. Dissolve  $0.16 \pm 0.001$  g of sodium nitroprusside in water to make 300 ml of solution.

Sodium Hydroxide solution.  $0.250 \pm 0.002$  M.

RDX-free HMX. Weigh  $25 \pm 1$  g of Type III HMX into a paper extraction thimble, and extract it with 500 ml aqueous acetone. Cool, and filter off the extracted HMX, washing it first with aqueous acetone, then with water. Ensure that no sensitive polymorphs of HMX are present, and dry for 2 hours in an oven at  $103 \pm 2^\circ\text{C}$ .

4.4. Procedure

Place  $10 \pm 0.1$  g of the sample, previously dried in an oven for 2 hours at  $103 \pm 2^\circ\text{C}$ , in a paper extraction thimble. Extract on a water-bath with 50 ml of aqueous acetone, using a vapour extraction tube a 100 ml flat-bottomed flask and an efficient condenser.

Continue the extraction until complete, then allow the contents of the flask to cool, agitating it from time to time. Make the volume up to 55 ml with acetone; this addition should not be more than 5 ml.

Attach a small piece of glass tubing filled with glass wool to the tip of a 5 ml pipette using a short piece of rubber tubing. Using this as a

filter, pipette 5 cm<sup>3</sup> of the sample extract into a 25 cm<sup>3</sup> stoppered flask, removing the filter before emptying the pipette. Add to the flask the following reagents by burettes, shaking the flask during each addition.

7.5 cm <sup>3</sup>	acetone	} in this order
7.5 cm <sup>3</sup>	sodium nitroprusside solution	
1.0 cm <sup>3</sup>	0.250 M sodium hydroxide solution	

Immerse the flask in an ice-cold water-bath for 4 hours, and then remove the flask from the bath and measure the optical density of the solution at 625 to 635 nm. Use a 1 or 4 cm cell according to the intensity of the colour, and water in a similar cell for reference.

Determine the amount of RDX present ( $W_1$ ) by reference to the calibration graph (see clause 5).

NOTE : To avoid interference from condensation on the outside of the cells it has been found to be useful to swab both the sample and water cell faces with the dilute surfactant solution.

#### 4.5. Preparation of calibration graph

Prepare a number of standards by weighing accurately amounts of dry RDX over the range 0.001 to 0.026 g into paper extraction thimbles and adding  $10 \pm 0.1$  g of RDX-free HMX as prepared in clause 3. Proceed by the method previously described in clause 4.1 to obtain the optical densities. Prepare a calibration graph by plotting optical density against mass of RDX.

#### 5.6. Reporting

$$\% \text{ RDX content} = W_1 \times 10$$

where  $W_1$  = Mass of RDX in sample.



TEST PROCEDURES  
PARAGRAPH 5

ALPHA HMX CONTENT - MICROSCOPY METHOD

5.1. Principle

A small amount of the explosive is dispersed in a liquid of refractive index  $1.563 \pm 0.001$  (at  $23^{\circ}\text{C}$ ) and the  $\alpha$  HMX crystals present detected microscopically using a polarizing microscope. The procedure is concerned with crystals with a maximum dimension greater than  $30\text{ }\mu\text{m}$  or a minimum dimension greater than  $10\text{ }\mu\text{m}$ .

5.2. Definitions

For the purpose of this method, the minimum dimensions of a crystal, as viewed under the microscope, is taken to be the dimension shown by the crystal at right-angles to the direction of the major dimensions. (This avoids the possibility of a large elongated triangular crystal, for example, being considered to have zero minimum dimensions).

A large alpha HMX crystal is considered to be :

one with a maximum (major) dimension exceeding  $120\text{ }\mu\text{m}$  or  
one with a minimum dimension exceeding  $10\text{ }\mu\text{m}$ .

5.3. Apparatus

A polarizing microscope having a magnification of 60 to 90 times, fitted with a rotating object stage, an attachable mechanical stage, and an eye-piece containing a calibrated squared grid. The grid is used to define the field of view and to determine the size of the crystals.

5.4. Reagent

Immersion liquid. Add 1-bromonaphthalene to bromobenzene until a refractive index of  $1.563 \pm 0.001$  at  $23^{\circ}\text{C}$  is obtained.

5.5. Procedure

Transfer successive small portions from various parts of the sample to a  $10\text{ cm}^3$  beaker until there is approximately 2 g of the sample in the beaker. Dry the beaker and contents for 2 hours in an oven at  $103 \pm 2^{\circ}\text{C}$ .

Wet a small amount of the dried sample with a minimum amount of the immersion liquid. Take a small portion of the wetted sample, and prepare a

slide using the immersion liquid. Secure the slide on the mechanical stage, and bring one corner of the main mass of product crystals into the field of view. Carry out the examination with the polarizer in position. Count the total number of crystals of any type that are seen within the grid and that have a maximum dimension of over 30  $\mu\text{m}$  or a minimum dimension greater than 10  $\mu\text{m}$ . Identify the alpha HMX crystals among these by rotating the stage to two extinction positions 90° apart, at one (at least) of which the alpha HMX crystals will disappear (i.e. match the background). Ensure that the extinction occurs in these positions by inserting the analyser. Count these alpha HMX crystals, and record their size if the maximum dimension exceeds 120  $\mu\text{m}$  or if the minimum dimension exceeds 10  $\mu\text{m}$ . Return the rotating stage to its original setting, and move the mechanical stage to the next field of view. Continue the examination until 200 crystals have been examined. Prepare new slides and repeat the examination until the requisite number of crystals have been examined.

#### 5.6. Reporting

- Report (a) the number of alpha HMX crystals with maximum dimensions in the range 30  $\mu\text{m}$  to 120  $\mu\text{m}$  per 600, 1000 and 2000 crystals, as appropriate ;
- (b) the number of alpha HMX crystals exceeding 10  $\mu\text{m}$  in minimum dimension or 120  $\mu\text{m}$  in maximum dimension.

TEST PROCEDURES  
PARAGRAPH 6

ALPHA HMX CONTENT - INFRA-RED SPECTROPHOTOMETRY METHOD

6.1. Principle

A sample of the explosive is ground in the presence of pure liquid paraffin and the  $\alpha$  HMX content of the resultant paste is determined by infra-red spectrophotometry.

6.2. Apparatus

A double-beam, recording infra-red spectrophotometer

Polished rock-salt plates, 40 x 40 x 5 mm.

Aluminium, lead or polytetrafluoroethylene foil nominal thickness of 0.05 mm.

A ball mill of approximately 130 mm internal diameter (approximately 1 litre capacity) with a tight fitting lid containing twenty 25 mm diameter, and five 20 mm diameter ceramic balls.

6.3. Reagent

Liquid paraffin, pure, to Def Stan 65-14/1 or equivalent.

6.4. Procedure

Place the ceramic balls in a clean, dry ball mill jar and add 2.0 cm<sup>3</sup> of pure liquid paraffin from a hypodermic syringe. Shake the jar until all the balls and the inside of the jar are coated with liquid paraffin. Add  $4 \pm 0.01$  g of the sample, previously dried in an oven for 2 hours at  $103 \pm 2^\circ\text{C}$ , to the jar and mill for  $30 \pm 1$  minutes at approximately 80 revolutions per minute.

(CAUTION : The milling operation must be done under remote control.) Remove the paste.

Place a thin wedge of the paste down the centre of a rock-salt plate. Place a thin strip, not more than 5 mm wide, of aluminium, lead or polytetrafluoroethylene foil, 0.05 mm thick along each of two opposite margins of one face of the plate. Fit another rock-salt plate on top of the first one and press down gently until the paste spreads out, and the top plate bears down firmly on both spacers.

Place this cell in the sample beam of a double-beam IR spectrophotometer and place a similarly prepared cell of pure  $\beta$  HMX in the reference

beam. Adjust the spectrophotometer by means of the optical balance, or using a wedge-cell, or by the use of a suitable dilution with rock-salt in the reference cell or by any other suitable combination of these methods so that it records 90 per cent transmission at a wave number of  $995\text{ cm}^{-1}$ . Run the spectrum from  $1100$  to  $950\text{ cm}^{-1}$ .

In the absence of RDX the spectrum should reveal no evidence of adsorption at  $1035$  to  $1030\text{ cm}^{-1}$  additional to the normal absorption due to the pure  $\beta$  HMX. If RDX is present, the band at  $850$  to  $848\text{ cm}^{-1}$  may be used instead but, being a weaker band, the sensitivity is somewhat less.

#### 6.5. Reporting

Report the percentage of alpha HMX in the sample.

TEST PROCEDURES

PARAGRAPH 7

MELTING POINT - BLOCK MAQUENNE METHOD

7.1 Principle

The instantaneous melting point of the high explosive is the minimum temperature at which melting occurs in less than 30 seconds after the H.E. is placed on the block Maquenne.

The melting point is determined by comparison with that of a standard explosive.

7.2 Reagents

Standard lot of HMX.

7.3 Equipment

A block Maquenne with electrical heating and shielded from air currents.

A thermometer with 0,5°C graduations.

7.4 Procedure

A few grams of homogenized material are dried at 100°C for one hour and then pulverized to a fine powder.

Select a thermometer (7-3) such that the reservoir can be located in the central portion of the block and the column is as short as possible. Mark the location of the reservoir on the block. The surface of the block must be perfectly clean.

Regulate the block heating rate such that the temperature rise is about 1°C in 2 to 3 minutes in the zone corresponding to the melting point area.

Place a very small quantity of material on the block such that the crystals are spread over the temperature reservoir. The melting occurs in less than 30 seconds after the material is placed on the block.

Repeat the test on the standard explosive with the same thermometer positioned exactly in the same place.

7.5. Expression of the results

Let:

$t_0$  = the accepted melting point of the standard lot  
 $t_1$  = the temperature observed when the test material  
melted  
 $t_2$  = the temperature observed when the standard  
material melted  
 $t$  = the melting point of the test material

Then:

$$t = t_1 + t_0 - t_2$$

TEST PROCEDURES  
PARAGRAPH 8

MELTING POINT - FISHER-JOHNS METHOD

8.1. Principle

Determine the temperature at which a sample of HMX transitions from solid to liquid state.

8.2. Reagents

HMX (representatives sample from batch).

8.3. Apparatus

- Fisher-Johns hot stage melting point apparatus.
- Small agate mortar.
- 18 mm diameter cover glasses.

8.4. Procedure

A Fisher-Johns or equivalent hot stage melting point apparatus is used to determine the melting point of HMX. The thermometer of the apparatus is calibrated using appropriate melting point standards. A portion for the sample is ground in a small agate mortar and a very small quantity (approximately 0.05 g) of the finely pulverized sample placed between two clean 18 mm diameter cover glasses. These are gently but firmly pressed together and placed in the circular depression on the stage. The powerstat is turned up and the unit allowed to heat. The heating rate may be very rapid to within 15°C of the melting point. Thereafter a heating rate of approximately 1°C per minute is used.

8.5. Calculation and Reporting

When the sample begins to melt, the thermometer is read and the temperature reading, adjusted to reflect the thermometer calibration correction, is recorded as the melting point.

TEST PROCEDURES  
PARAGRAPH 9MELTING POINT HERAEUS FUS-O-MAT METHOD9.1. Apparatus

Heraeus Fus-O-Mat

9.2. Procedure

A portion of the explosive sample shall be finely ground in an agate mortar. A very small quantity (5-10 mg) of the pulverized sample is filled to a height of 3 to 5 mm into the 1 mm diameter melting point tube.

The stainless steel encased thermocouple is positioned in the sample material so that it reaches the bottom of the melting point tube.

The prepared tube is then placed in the heating oven and fastened. The plotter is adjusted to the proper range and the "Start" button of the Fus-O-Mat is pressed.

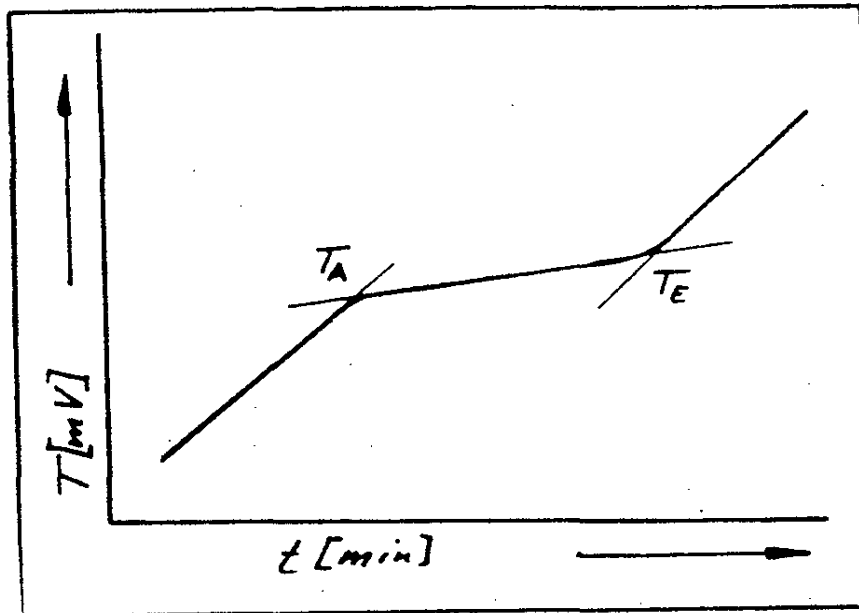
A heating rate of 100°C/min may be chosen to within 30 to 40°C of the melting point. Thereafter the heating rate is reduced to 10°C/min and the plotter is engaged to record the temperature curve.

After completion of the melting process, for HMX the beginning of decomposition, both oven and plotter are shut off. The thermocouple is removed from the melt, cleaned with acetone, and returned to the storage tube. The used melting point tube is discarded.

9.3. Determination of the melting point

The melting process is marked in the recorded curve by a momentary pause in the rate of temperature increase (point of coalescence). It is immediately followed by the exothermic decomposition reaction of the HMX. The melting Point (temperature at the beginning of the melting process) is taken from the temperature curve as shown in the following figure page D-23.





$T_A$  = Beginning of melting (melting point)

$T_E$  = End of melting

**TEST PROCEDURES**  
**PARAGRAPH 10**

MELTING POINT - TOWNSON AND MERCER METHOD

10.1. Principle

The melting point is determined on a finely ground sample of the explosive contained in a capillary tube using an electrically heated melting point apparatus. The melting point is recorded as the temperature at which the first signs of liquid appear.

10.2. Apparatus

Electrically heated melting point apparatus.

Melting point tubes supplied by the manufacturer of the apparatus.

Glass thermometers covering the range 140 to 300°C graduated in 0.5°C divisions.

10.3. Procedure

Take a representative sample of approximately 1 g and finely grind by any approved method. Dry the finely ground HMX for 4 hours in an oven at  $103 \pm 2^\circ\text{C}$ . Introduce sufficient of the ground sample into a melting point tube to give a height of about 5 mm after tapping down. Insert the tube in the apparatus when the thermometer registers  $250^\circ\text{C}$ . Increase the temperature at a rate of 2 to  $3^\circ\text{C}$  per minute and record the temperature at which the first sign of melting is observed. Apply any temperature corrections for the thermometer as required.

10.4. Reporting

Report the temperature (corrected) at which the first signs of liquid appear as the melting point of the explosive.

TEST PROCEDURES  
 PARAGRAPH 11

INSOLUBLE MATTER CONTENT - DIMETHYLSULFOXIDE METHOD

11.1. Principle

Dissolving the explosive in DMSO, filtering the solution through a crucible, and weighing the residue.

11.2. Reagents

Dimethylsulfoxide

11.3. Equipment

- 250 ml beaker
- filtering quartz crucible with a porosity of 20 to 40 micrometers
- water bath

11.4. Procedure

In a 250 ml beaker, weigh  $10 \text{ g} \pm 2 \text{ g}$  of dry explosive to within 0,1 g ( $m$ ). Dissolve it in 50 ml of DMSO by heating the beaker and solution in a water bath. Filter through a crucible that has been calcinated at  $700 \pm 50^\circ\text{C}$ . Wash the crucible with 50 ml of acetone.

Dry for one hour at  $100^\circ\text{C}$  and weigh to within 0,1 mg ( $m_2$ ).

11.5. Expression of the results

$$\text{Percentage of insoluble matter} = \frac{(m_2 - m_1) \times 100}{m}$$

Where:

- $m$  = the mass of the dry explosive in grams
- $m_1$  = the mass of the empty crucible in grams
- $m_2$  = the mass of the crucible containing the insoluble mater in grams

**11.6 Note**

This test can be performed with damp explosive (up to about 20%) by accounting for the moisture in reporting the percent insoluble matter. The resultant expression is:

$$\text{Percent insoluble matter} = \frac{(m_2 - m_1) \times 100}{m (100-H)}$$

Where:

m = the damp explosive mass in grams  
H = the percentage moisture

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TEST PROCEDURES  
PARAGRAPH 12

INSOLUBLE MATTER CONTENT - ACETONE METHOD 1

12.1. Principle

The explosive is dissolved in hot acetone and then filtered through a sintered glass crucible. Any residue is dried and weighed.

12.2. Apparatus

600 ml beaker  
Medium porosity pyrex filtering crucible  
Muffle furnace  
Analytical balance  
Air driven motor or stirring rod  
Watch glass cover  
Steam heated hot plate

12.3. Reagents

Acetone, technical grade.

12.4. Procedure

Weigh an approximately 10 g portion of the sample accurately to the nearest 0.001 g, place the sample in a 600 ml beaker and add 400 ml of filtered acetone. Cover the beaker with a watch glass and place the beaker and contents on a steam bath. Stir occasionally until all of the HMX has dissolved. Additional acetone may be added if required. Filter the HMX solution through a medium porosity pyrex filtering crucible, which has been ignited in a muffle furnace at  $700 \pm 20^\circ\text{C}$ , and tared to the nearest 0.0001 g. Transfer any remaining insoluble material from the beaker to the crucible with aid of a stream of acetone from a wash bottle. Wash the material remaining in the crucible three times with 20 ml portions of acetone and aspirate until the odor of acetone is no longer noticeable. Dry the crucible in an oven at  $100 \pm 5^\circ\text{C}$  for thirty minutes, cool in a desiccator and weigh to the nearest 0.0001 g. Reserve the crucible and contents for determination of inorganic insoluble materials.

12.5. Reporting

$$\text{Percent Acetone Insoluble Material} = \frac{(A-B) \times 100}{W}$$

Where A : the final weight of the acetone insoluble material and the crucible in grammes.

B : the tare weight of the crucible in grammes.

W : the HMX sample weight in grammes.

TEST PROCEDURES  
PARAGRAPH 13INSOLUBLE MATTER CONTENT - ACETONE METHOD 213.1. Principle

The explosive is dissolved in hot acetone and then filtered through a sintered glass crucible. Any residue retained is weighed.

13.2. Apparatus

Sintered glass crucible Grade P16(G4) complying with BS 1752.

13.3. Reagents

Acetone analytical reagent grade.

13.4. Procedure

Place a portion of the sample as received equivalent to  $10.0 \pm 0.1$  g dry mass in a 1 dm<sup>3</sup> beaker, add 600 to 700 cm<sup>3</sup> of analytical reagent grade acetone. Cover with a clock-glass and heat on a boiling water-bath until no more material will dissolve. Filter the hot solution through a tared P16 (G4) sintered-glass crucible ( $W_1$ ) taking care to transfer all the insoluble matter to the crucible. Wash thoroughly with 100 cm<sup>3</sup> of hot acetone to ensure that no crystals of HMX have been left on the bottom of the crucible. Dry the crucible for 30 to 35 minutes at  $103 \pm 2^\circ\text{C}$ . Allow to cool in a desiccator and reweigh ( $W_2$ ).

13.5. Reporting

Total insoluble matter present per cent =  $(W_2 - W_1) \times 10$ .

NOTE : If the quantitative result exceeds the specification limit the material must be retained for examination.

TEST PROCEDURES  
PARAGRAPH 14

INSOLUBLE MATTER CONTENT - ACETONE METHOD 3

14.1. Principle

The explosive is dissolved in acetone, filtered through paper-pulp pad, which is subsequently ignited at 600°C and the amount of residue determined.

14.2. Apparatus

Shake a mixture of gravimetric quality filter-paper and water in a tall bottle until the paper disintegrates. Prepare a pad of this paper-pulp on a perforated porcelain disc in an ordinary glass filter-funnel, as in the preparation of a Gooch crucible. Finally wash the pad with acetone.

14.3. Reagent

Acetone analytical reagent grade.

14.4. Procedure

Prepare a hot solution of a portion of the sample as received equivalent to  $10.0 \pm 0.1$  g dry mass by dissolving it in 600 to 700 cm<sup>3</sup> of analytical reagent grade acetone and filter it with suction through the prepared pad. Wash thoroughly with hot acetone and continue suction for a minute or so to remove most of the residual acetone.

Transfer the pad with insoluble matter (using a mounting needle) to a tared crucible ( $W_3$ ). Clean the inside of the funnel with a piece of filter-paper and add this to the crucible. Ignite the pad in the crucible until all the carbonaceous matter has disappeared, and finally heat at  $600 \pm 10^\circ\text{C}$  for 10 to 15 minutes. (CAUTION : this operation must be performed in a fume cupboard with full ventilation.) Allow the crucible to cool in a desiccator and reweigh ( $W_4$ ).

14.5. Reporting

Inorganic insoluble matter per cent =  $(W_4 - W_3) \times 10$ .

NOTE : If the quantitative result exceeds the specification limit the material must be retained for examination.

TEST PROCEDURES

PARAGRAPH 15

GRITTY PARTICLES - METHOD 1

15.1 Principle

Extraction of the explosive through a sieve by means of an appropriate solvent and verification of the nature of the remaining gritty particles.

15.2 Reagents

Dimethylsulfoxide (DMSO)

15.3 Equipment

Metalic sieve with 0,5 mm and 0,25 mm mesh wire screens. Finding the gritty particles is easier with 5 cm diameter sieves.

15.4 Procedure

Place the 0,5 mm mesh screen over the 0,25 mm mesh screen. Place  $50 \pm 2$  grams on the 0,5 mm mesh screen. Extract with DMSO. Once the extraction is completed, collect separately the particles remaining on the two screens. Check the nature of the gritty particles by placing them one by one between two glass plates which are slid on against the other. Gritty particles will grind and scratch the glass. Count the number of gritty particles retained on each screen ( $n_1$  and  $n_2$ ).

15.5 Expression of the results

Number of gritty particles:

> 0,5 mm :  $n_1$  (held on 0,5 mm mesh screen)  
0,5 - 0,25 :  $n_2$  (between 0,5 and 0,25 mm)



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**TEST PROCEDURES**  
**PARAGRAPH 16**

GRITTY PARTICLES - METHOD 2

16.1. Principle

The explosive is extracted with acetone using a Soxhlet extractor. The number of particles retained on 420 and 250  $\mu\text{m}$  sieves are counted.

16.2. Apparatus

Soxhlet extractor  
Steam bath  
U.S. Standard No. 60 sieve  
U.S. Standard No. 40 sieve

16.3. Reagent

Acetone analytical.

16.4. Procedure

A  $50 \pm 0.5$  g sample of HMX is transferred to a thimble of Soxhlet extractor and placed in the Soxhlet apparatus or other suitable extractor. Sufficient acetone is added to the flask and the specimen is extracted on a steam bath until all HMX is dissolved. Any insoluble material in the thimble is transferred to a U.S. Standard No. 60 sieve complying with specification RR-S-366. The particles are counted. The particles are brushed on a U.S. Standard No. 40 sieve, and any that are retained are counted.

16.5. Reporting

Report the number of particles on the No. 60 sieve (250  $\mu\text{m}$ )  
Report the number of particles on the No. 40 sieve (420  $\mu\text{m}$ )

TEST PROCEDURES  
PARAGRAPH 17DETERMINATION OF GRIT/GRITTY PARTICLES RETAINED ON A 250 AND/OR 63  $\mu$ m SIEVE17.1. Principle

The explosive is extracted with acetone using a modified soxhlet apparatus. Any residue is weighed and examined for gritty particles.

17.2. Apparatus

An extraction apparatus comprising an extractor body fitted with a suitable condenser and containing a tall cylindrical 63  $\mu$ m sieve supported on a triangle in a glass dish. (See Diagram page D-34).

Sieves 63  $\mu$ m and 250  $\mu$ m complying with BS 410.

17.3. Reagent

Acetone analytical Reagent Grade.

17.4. Procedure

Place a portion of the sample as received equivalent to  $50 \pm 1$  g dry mass into a tall cylindrical 63  $\mu$ m sieve (90 x 38 mm). Assemble the apparatus as shown in Materials Centre Diagram 378 and place on a steam-heated water-bath. Heat the acetone so that continuous refluxing of the solvent dissolves all the HMX. Remove and dry the sieve.

Transfer the residue to an accurately weighed ( $\pm 0.0001$  g) watch-glass ( $W_7$ ) and reweigh ( $W_8$ ).

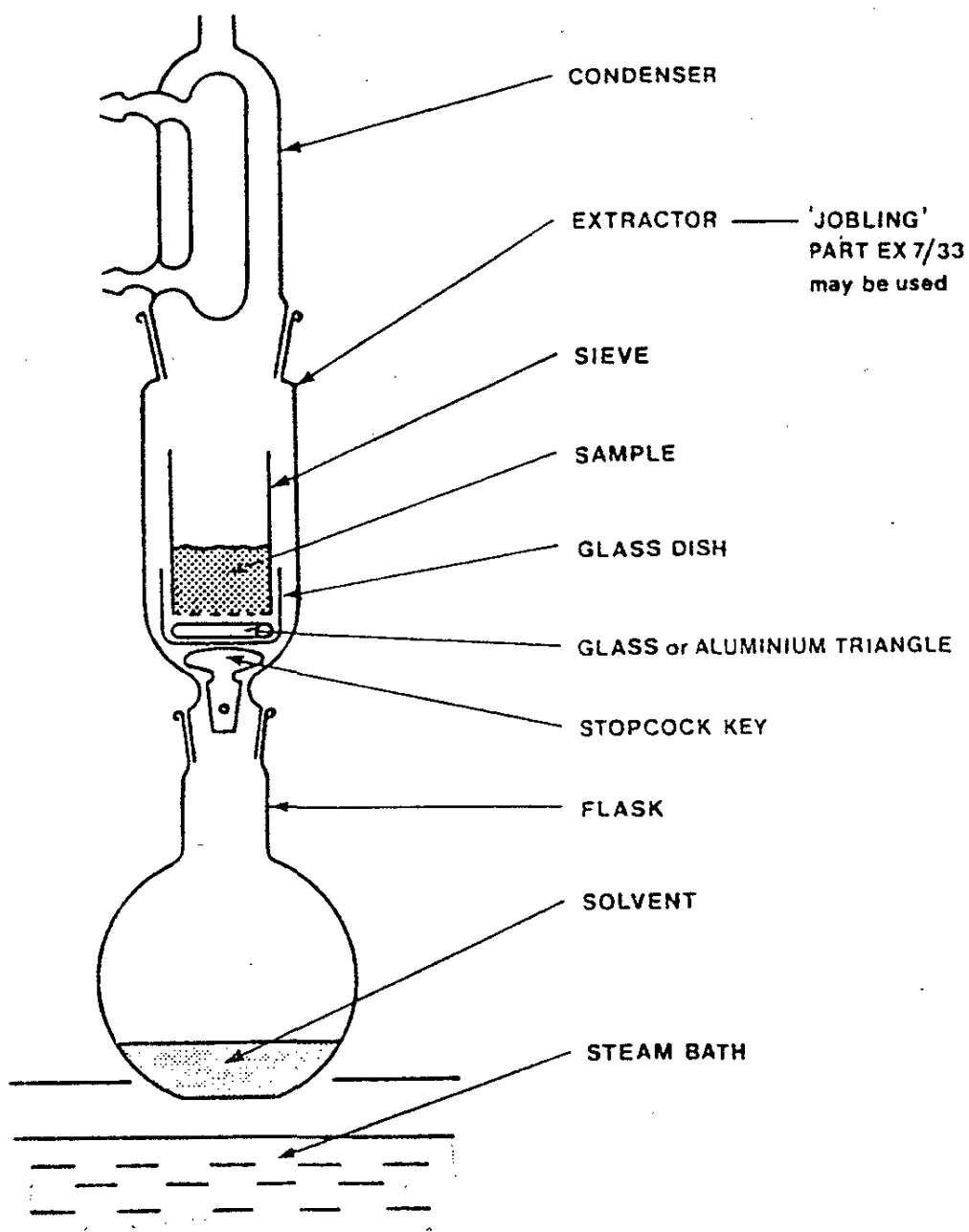
Transfer the residue on the watch-glass to a 250  $\mu$ m sieve and examine the residue for the presence of gritty particles.

17.5. Reporting

Grit retained on a 63  $\mu$ m sieve, per cent =  $(W_8 - W_7) \times 2$ .

Report the number of particles of grit retained on the 250  $\mu$ m sieve.

VAPOUR/SOLVENT EXTRACTOR SYSTEM  
(MODIFIED SOXHLET)



MATERIALS CENTRE DIAGRAM 378

**TEST PROCEDURES**  
**PARAGRAPH 18**

ASH CONTENT - METHOD 1

18.1. Principle

Red calcination of the insoluble matter obtained, by the mean of procedure paragraph 11.

18.2. Equipment

Muffle furnace, set at  $700 \pm 50^{\circ}\text{C}$ .

18.3. Procedure

Calcinate the quartz crucible, containing the insoluble matter. Be sure not overheating ( $700 \pm 50^{\circ}\text{C max}$ ). Cool in a dessicator, and weight to within 1 mg ( $m_2$ ).

18.4. Expression of results

$$\text{Ash \%} = \frac{(m_2 - m_1) \times 100}{m}$$

where :

$m_1$  : empty crucible mass (in grammes)

$m_2$  : mass of crucible, after calcination (in grammes)

$m$  : mass of tested explosive, before extraction (in grammes).

TEST PROCEDURES  
PARAGRAPH 19

ASH CONTENT - METHOD 2

19.1. Principle

Residue from determination of matter insoluble in acetone (paragraph 12) is ignited at 700°C and the amount of residue is determined.

19.2. Equipment

Muffle furnace  
Analytical balance

19.3. Reagents

19.4. Procedure

Ignite the sample reserved from the acetone insoluble material determination, in a muffle furnace at  $700 \pm 10^\circ\text{C}$  for  $30 \pm 5$  minutes, cool in a desiccator and weigh to the nearest 0.0001 g.

19.5. Reporting

$$\text{Percent inorganic insoluble material} = \frac{(C-B) \times 100}{W}$$

where C : the weight of the crucible and contents after ignition in grammes.

B : the tare weight of the crucible in grammes.

W : the HMX sample weight in grammes.

TEST PROCEDURES  
PARAGRAPH 20DETERMINATION OF ACIDITY OR ALKALINITY20.1. Principle

The explosive is dissolved in acidified acetone and the acidity or alkalinity present is determined by back titration with barium hydroxide solution.

20.2. Apparatus

Iodine flasks complying with BS 2755 or equivalent.

Condensers complying with BS 5922/ISO 4799.

Burettes complying with BS 846/ISO 385.

20.3. Reagents

Potassium chloride solution. Add 5 cm<sup>3</sup> of saturated potassium chloride solution to 1 litre of boiled water and store the product under carbon dioxide-free conditions.

Acidified acetone. Add 1 cm<sup>3</sup> of 0.1 M acetic acid to each litre of boiled and cooled acetone and store the product under carbon dioxide-free conditions.

Standard barium hydroxide solution (Baryta solution). Barium hydroxide standard solution of 0.01 M strength stored under carbon dioxide-free conditions.

Cresol-red indicator. A 0.3 per cent solution in methylated spirit (complying with BS 3591).

Air or nitrogen. A stream of air or nitrogen which has been scrubbed free of carbon dioxide by a series of alkaline scrubbers.

20.4. Procedure

Place a quantity of water in each of three 500 cm<sup>3</sup> iodine flasks, and boil for 5 minutes. Discard the water, and cool the flasks. Into each of flasks 1 and 3 weigh out a portion of sample as received equivalent to  $3.0 \pm 0.1$  g (dry mass) of HMX and add 100 cm<sup>3</sup> of the acidified acetone to each of the three flasks. Fit the condenser(s) to the flask(s) and reflux on a boiling water bath until the HMX has dissolved. Remove the condenser

and add 100 cm<sup>3</sup> of the potassium chloride solution to each flask, shaking the flasks during the addition. Stopper the flask(s) and cool rapidly in a stream of cold water.

Meanwhile, allow a stream of the gas to pass through the scrubber system. Unstopper flask 1 and using a simple type of delivery tube allow the carbon dioxide-free gas to bubble through the contents. Add 10 drops of the indicator solution and titrate with the baryta solution to the cresol-red end-point. Decant the bulk of the liquor to waste, add the contents of flask 2 to the residual precipitate of HMX in flask 1, and with the gas bubbling through, again titrate with baryta ( $V_1$  cm<sup>3</sup>) using another 10 drops of the cresol-red indicator.

With the gas bubbling through as before, titrate ( $V_2$  cm<sup>3</sup>) the contents of flask 3 to the same permanent pink end-point and using the same quantity of indicator.

#### 20.5. Reporting

- When  $(V_2 - V_1)$  cm<sup>3</sup> is + ve, the sample is acidic
- When  $(V_2 - V_1)$  cm<sup>3</sup> is - ve, the sample is alkaline.

Acidity or alkalinity in meq/kg is  $(V_2 - V_1) \times 6.67$ .

TEST PROCEDURES  
PARAGRAPH 21DETERMINATION OF ACIDITY21.1. Principle

The explosive is dissolved in acetone and the acidity is determined by titration.

21.2. Apparatus

800 ml beaker  
Steam bath  
Burette

21.3. Reagent

Acetone reagent grade  
Phenolphthalein (1% in ethyl alcohol) or  
Methyl red (0.1 g/60 ml of ethyl alcohol)  
0.05 N sodium hydroxide solution  
Distilled Water

21.4. Procedure

Weigh an approximately 10 g portion of the dried sample accurately to the nearest 0.001 g, place in a clean 800 ml beaker and add 500 ml of acetone. Place the beaker on a steam bath and heat with occasional stirring until the sample has completely dissolved. Add 100 ml of distilled water and 3 or 4 drops of phenolphthalein (1 % in ethyl alcohol) or methyl red (0.1 g/60 ml of ethyl alcohol) indicator and titrate to the end point with 0.05 N sodium hydroxide solution. Run blank determination using all of the reagents except the HMX sample in the same amounts as were used in the sample acidity determination.

21.5. Reporting

$$\text{Acidity, milliequivalent/kg} = \frac{6.0 (S-B) N}{W}$$

where S : the volume of sodium hydroxide used in the sample titration in ml.

B : the volume of sodium hydroxide used in the blank titration in ml.

N : the normality of sodium hydroxide used in the titrations.

W : the weight of sample used in g.



TEST PROCEDURES  
PARAGRAPH 22

THE APPLICATION OF THE VACUUM STABILITY TEST TO EXPLOSIVES

22.1. Principle

The vacuum stability test is used to assess the thermal stability of an explosive by measuring the volume of gas evolved on heating the explosive under specified conditions. It is undesirable to attempt the test on explosives which exhibit an appreciable vapour pressure.

A sample of the explosive is heated for the specified time in an evacuated tube in a heating bath maintained at a constant specified temperature. The volume of gas evolved over the specified period of time is determined by either a mercury manometric method or by using a pressure transducer.

22.2. Apparatus

Constant - temperature bath - metal block type. An electrically heated block bath with temperature control over the range 50 to 150°C controllable to within  $\pm 0.2^\circ\text{C}$  and insulated on all sides. An electrical cut-out shall be fitted which operates at 5°C above the nominal working temperature. The block contains holes drilled out to accommodate the sample heating tubes. The dimensions of the holes shall be such that the diameter is approximately 1 to 2 mm greater than the diameter of the heating tube, and the depth is approximately 165 mm. The centre of the holes shall not be less than 30 mm from the edge of the block. A thermometer is positioned in a sand-filled glass heating tube in one of the holes to afford the measurement of the bath temperature. Checks shall be carried out daily to ensure that the temperature of the bath remains constant.

Vacuum Pump. The pump shall be a rotary type of robust construction capable of rapidly attaining a pressure of less than 5 mm mercury absolute.

Pressure gauge, indicating the range 0 to 20 mm absolute.

Barometer. A Fortin or similar type barometer for determining atmospheric pressure.

Burettes complying with BS 846 (class A)

Sieves complying with BS 410.

22.3. Reagent

Water complying with BS 3978.

22.4. Procedure 1, vacuum stability test (manometer method)22.4.1. Apparatus

Heating tube and manometer as illustrated in Diagram page D/44. The tubes and manometers, particularly the latter, are rather delicate and great care must be taken at all times in handling and storage.

22.4.2. Reagents

Mercury, clean and dry.

Petroleum jelly or a high vacuum silicone grease compatible with the appropriate explosive as a lubricant.

22.4.3. Calibration of the vacuum stability apparatus

## - The heating tube

Determine the volume (X) of the heating tube by filling the tube with mercury from a burette until the mercury reaches the level at which it will contact the ground glass joint of the capillary tube.

The available gas space (A) is given by :

$$A = X - \frac{\text{mass of explosive (g)}}{\text{explosive density (g/cm}^3\text{)}}$$

## - The capillary (manometer)

Place  $10.0 \pm 0.1$  g ( $W_1$ ) of mercury in the cup and manipulate the tube so that the mercury passes into the long (850 mm) section of the capillary. Ensure that the mercury remains in a continuous column. Measure the length in mm of the mercury column at three positions in the long section of the capillary and calculate the average of the three measurements (L).

## - Calculation

Calculate the mean internal volume per mm length of capillary (C) :

$$C = \frac{W_1}{d \times L}$$

where d = the density of the mercury at the calibration temperature.

#### 22.4.4. Procedure

Transfer a 5.0 g portion of HMX, which has been previously dried for 4 hours at  $103 \pm 2^\circ\text{C}$ , to the heating tube taking care to avoid contamination of the ground glass joint of the tube. (The introduction of the explosive can be facilitated by the use of a wide-bore short-stemmed glass funnel). Coat the male ground glass joint of the capillary with the lubricant. Attach the capillary to the heating tube ensuring a gas-tight joint by pressing the two components together with a gentle twisting motion until the interface of the joint is clear. Use the minimum amount of lubricant to ensure a good joint. Take care to prevent the tube falling from the manometer before it is held by the vacuum. Mount the assembled apparatus in a suitable rack so that the longest section of the capillary is nearly vertical and the bottom reservoir is supported.

Add to the capillary reservoir sufficient clean mercury (about 7 cm<sup>3</sup> or one-third filled) to fill the capillary and the reservoir after evacuation.

Connect the capillary reservoir to the vacuum pump using a vacuum line and rubber bung adapter. Tilt the assembly to bring the reservoir towards the horizontal so that there is a clear passage over the mercury for the air being evacuated from the heating tube and manometer. Start the pump and slowly allow the air to be evacuated. Evacuate until the pressure is below 5 mm of mercury, lightly tapping the heating tube to facilitate release of any occluded air from the sample.

When evacuation is complete, return the capillary to the vertical, carefully remove the vacuum line and allow the mercury to enter the capillary. This operation must be done very carefully to prevent the mercury rising too quickly up the manometer tube and overshooting into the heating tube. Pour a little mercury into the cup of the heating tube to serve as a secondary seal. Disconnect the pump.

Record the following data :

The total length of the capillary tube minus the vertical height of the column of mercury in the reservoir before heating ( $B_1$  mm).

The height of the mercury column above the surface of the mercury in the reservoir at the beginning of the test ( $H_1$  mm).

The room temperature at the beginning of the test ( $t_1$  °C).

The barometric pressure at the beginning of the test ( $P_1$  mm).

Ascertain that the bath temperature is constant at  $120^\circ\text{C}$ . Carefully place the heating tube of the prepared test assembly in the constant-temperature bath, ensuring that the connection between the heating tube and the capillary is not loosened. Support the capillary reservoir.

Heat the tube for 40 hours.

Remove the tube from the constant-temperature bath and allow the tube to cool to room temperature.

Record the following data :

The total length of the capillary tube minus the vertical height of the column of mercury in the reservoir after heating ( $B_2$  mm).

The height of the mercury column above the surface of the mercury in the reservoir at the end of the test ( $H_2$  mm).

The room temperature at the end of the test ( $t_2$  °C).

The barometric pressure at the end of the test ( $P_2$  mm).

#### 22.4.5. Calculation

Calculate the volume of gas  $V$  (at STP) liberated during the test as follows :

$$V \text{ cm}^3 = [A+C(B_2-H_2)] \times \frac{273 (P_2-H_2)}{760 (273+t_2)} - [A+C(B_1-H_1)] \times \frac{273 (P_1-H_1)}{760 (273+t_1)}$$

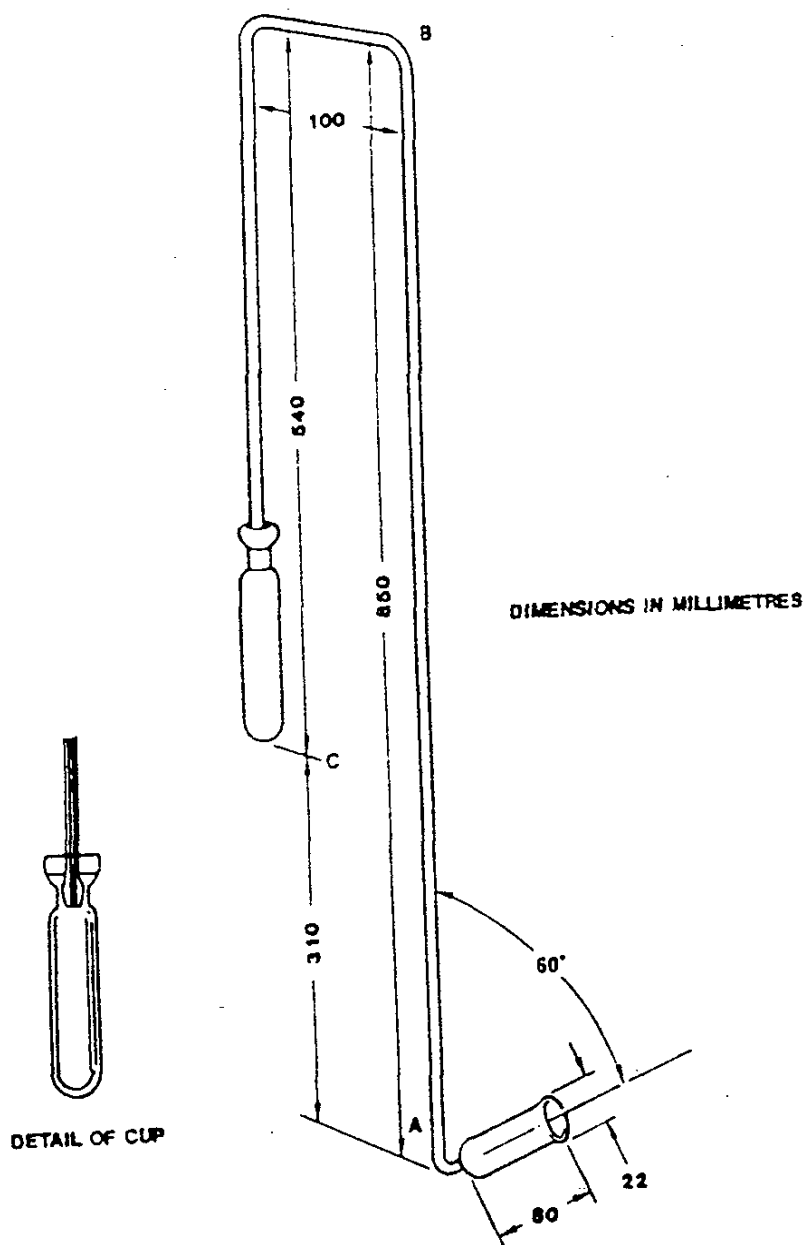
where  $A$ ,  $B_1$ ,  $B_2$ ,  $C$ ,  $H_1$ ,  $H_2$ ,  $P_1$ ,  $P_2$ ,  $t_1$  and  $t_2$  are as defined above.

#### 22.4.6. Dismantling

Remove the apparatus from the heating bath and suspend it from a hook. Remove the mercury from the well of the heating tube by suction and connect the capillary reservoir to the vacuum pump. Tilt the assembly and evacuate the capillary and heating tube. Admit air to the apparatus taking care that the heating tube does not fall from the capillary. Return the capillary to the vertical position and disconnect from the vacuum pump. Pour out the mercury into the bottle kept specially for 'dirty' mercury and separate the capillary and heating tube for cleaning.

#### 22.4.7. Cleaning

Empty the heating tube and remove residual traces of explosive and any lubricant round the joint with suitable solvents. Rinse with acetone and then with water. Fill the heating tube and capillary with a suitable solution for glassware cleaning and allow to stand for 24 hours. Finally rinse with water followed by acetone and blow-dry with clean, dry air. Store the cleaned heating tubes in a desiccator until required for use.



MERCURY MANOMETER APPARATUS  
DQA/TS Materials Centre Diagram 464A

## 22.5. Procedure 2, Vacuum stability test (transducer method)

### 22.5.1. Apparatus

Glass heating tube and assembly as illustrated in Diagram page D/48 and D/49. The tube is uniform bore, 265 mm long by 14 mm internal diameter with a wall thickness of 2 mm. The adapter is fitted with a hexagon nut for connection to the transducer. The side port is for connection as required to the vacuum line or to a gas syringe used in calibration of the assembly.

A strain gauge pressure transducer is used which is temperature-compensated to better than 5 % for a 100°C range of temperature, and which is capable to operation in the pressure range 0 to 1 bar absolute.

Devices found to be suitable are :

Type N° BHL-4050-00-01M0 range 0 to 1 bar absolute, or  
Type N° 4-366-01M0 range 0 to 1 bar absolute.  
Manufactured by Transamerica Ltd, Lennox Road, Basingstoke, Hants.

The transducer should be allowed to stabilize for at least 30 minutes after power up. The measuring system must be capable of detecting gas volumes to not less than  $\pm 0.02 \text{ cm}^3$  (STP).

Data-logging equipment comprising a regulated DC power supply with an output voltage suitable for the transducer ( $10.00 \pm 0.02 \text{ V DC}$  for the transducers previously quoted). The equipment shall be capable of measuring the transducer output voltages in sequence and recording the values obtained. In addition, the equipment shall have inputs for platinum resistance thermometers and be capable of recording temperature in sequence with the measured pressures. Simplified measuring systems using digital voltmeters may be used as alternatives to the data-logging equipment.

Calibrated gas-tight syringe. A Luer syringe of  $5 \text{ cm}^3$  capacity that can be attached to the adapter is required for calibration purposes.

### 22.5.2. Reagent

Petroleum jelly or a high vacuum silicone grease compatible with the appropriate explosive as a lubricant.

### 22.5.3. Procedure

Transfer a 5.0 g sample of the HMX, which has been previously dried for 4 hours at  $103 \pm 2^\circ\text{C}$  to the heating tube. Care shall be taken to ensure that the neck of the tube is free from adhering particles. (The introduction of the explosive can be facilitated by the use of a wide-bore short-stemmed glass funnel).

Lightly smear the O-rings of the adapter with the lubricant. Assemble the apparatus as in Diagram page D48. Connect the adapter evacuation port to the vacuum line, slide the inner tube outwards and evacuate the tube to less than 5 mm. When evacuation is complete, indicated by a steady reading of the digital voltmeter, seal the evacuation port by sliding the inner adapter inwards. Check that the seal is effective by monitoring for any drift on the voltmeter. Disconnect the vacuum line and record the voltmeter reading  $R_0$ . Set the plunger of the calibrated syringe to the required calibrating volume (3 cm<sup>3</sup>) and connect to the adapter port. Inject 3.0 cm<sup>3</sup> of air into the heating tube. Record the voltmeter reading  $R_3$ .

Inject a further 5.0 cm<sup>3</sup> of air and record the digital voltmeter reading  $R_5$ , the barometric pressure,  $P_3$  mm, and the room temperature  $t_3$  °C.

The difference in readings is the digital response for 2 cm<sup>3</sup> of air at  $P_3$  and  $t_3$  °C. The response is determined from the two volumes of air to compensate for the 'dead volume' between the syringe and adapter core. This calibrates the free space available in the apparatus after adding the test specimen to the heating tube. Repeat the calibration for every test.

Ascertain that the bath temperature is constant at 120°C. Detach the syringe, connect the port to the vacuum line and evacuate the tube. Check that the apparatus is sealed effectively by monitoring the transducer voltage for about a minute.

Record the initial digital voltmeter reading  $E_1$ .

Place the heating tube of the prepared test assembly in the constant-temperature bath and heat the tube for the 40 hours heating period. Remove the test assembly from the constant-temperature bath and allow the tube to cool to room temperature.

Record the final digital voltmeter reading  $E_2$  and the room temperature  $t_4$  °C.

#### 22.5.4. Calculation

Calculate the response factor for each heating tube and transducer, "f", for 1 cm<sup>3</sup> air at STP :

$$f = \frac{(R_5 - R_3) - (R_3 - R_0)}{2} \times \frac{760}{P_3} \times \frac{(273 + t_3)}{273}$$

Calculate the volume of gas,  $V$ , evolved from the specimen at STP :

$$V = \frac{E_2 \times (273 + t_3)}{f \times (273 + t_4)} - \frac{E_1}{f}$$

where :  $R_0$  = empty tube voltmeter reading

$R_3$  = voltmeter reading for 3.0 cm<sup>3</sup> air

$R_5$  = voltmeter reading for 5.0 cm<sup>3</sup> air

$E_1$  = voltmeter reading before heating

$E_2$  = voltmeter reading after the specified heating period

$t_3$  = room temperature °C at the time of calibration of the apparatus

$t_4$  = room temperature °C at the end of the test

$P_3$  = atmospheric pressure (mm of mercury)

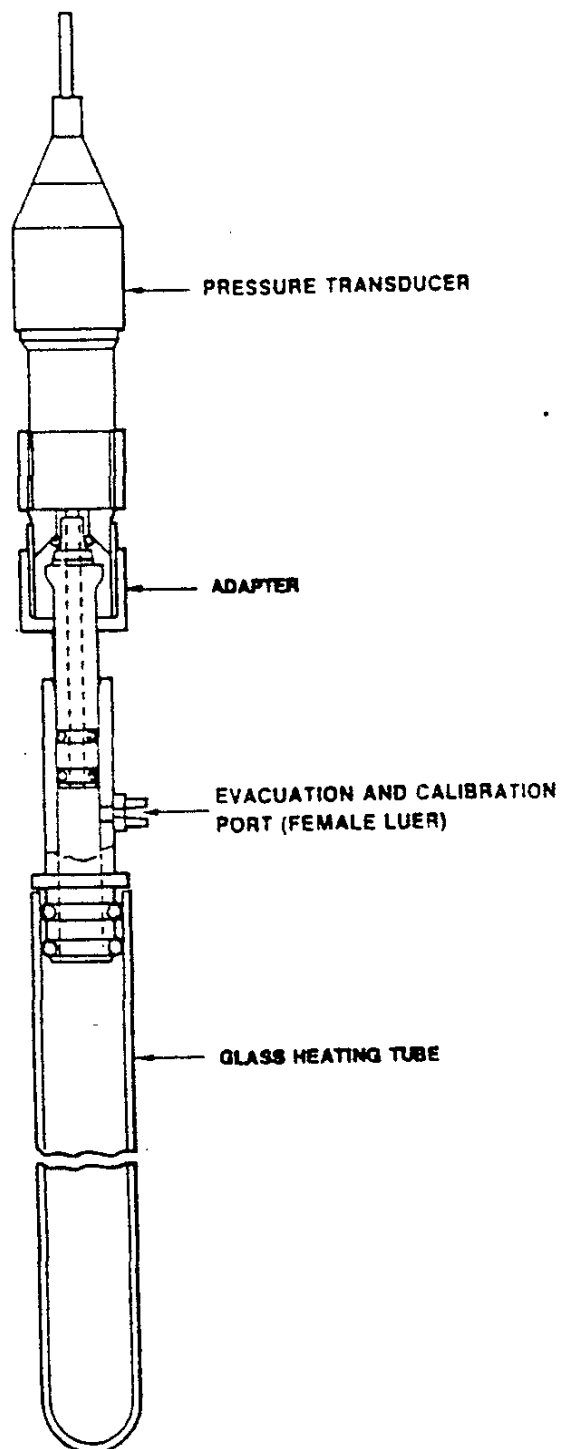
#### 22.5.5. Dismantling and cleaning

Remove the apparatus from the heating bath and release the vacuum by sliding up the inner adapter tube. Dismantle the apparatus, empty the heating tube and remove residual traces of explosive from the apparatus with suitable solvents. Rinse the heating tube with acetone and then with water. Fill the tube with a suitable solution for glassware cleaning and allow to stand for 24 hours. Finally rinse with water followed by acetone and blow-dry with clean, dry air. Store the cleaned heating tubes in a desiccator until required for use.

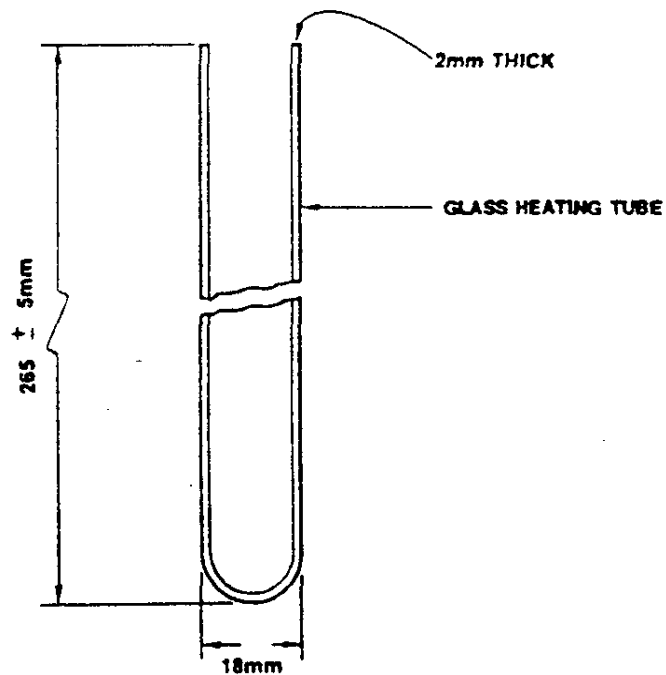
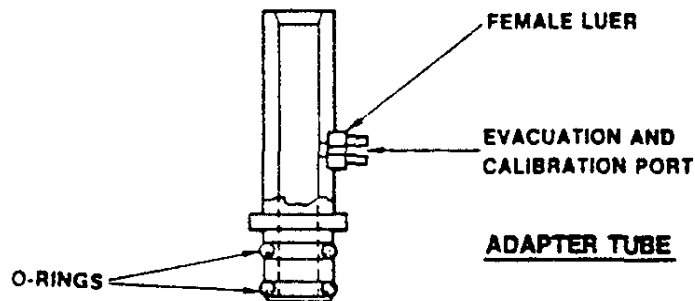
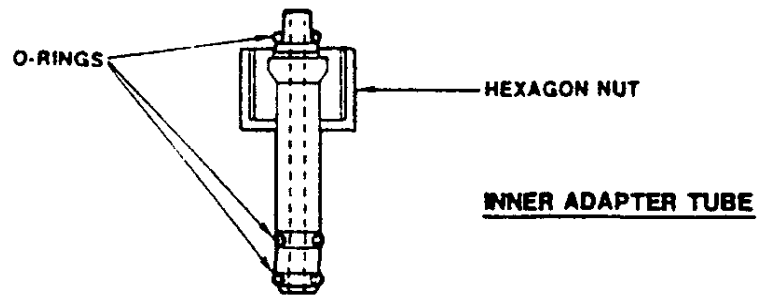
#### 22.6. Compatibility test on joint lubricant

Add 0.015 g of the lubricant under test to 5.0 g of the explosive and mix well. Carry out the compatibility test on the mixture at the appropriate temperature. Carry out blank determinations on the explosive alone and the lubricant alone, at the same time. The volume of gas evolved from the mixture of explosive and lubricant, minus the volume of gas from the lubricant alone, shall not exceed the volume of gas evolved by the explosive alone by more than 1.0 cm<sup>3</sup>.





ASSEMBLED TRANSDUCER APPARATUS  
DQA/TS Materials Centre Diagram 463



TRANSDUCER APPARATUS  
DQA/TS Materials Centre Diagram 462

TEST PROCEDURES  
PARAGRAPH 23

DETERMINATION OF CYCLOHEXANONE BY GAS CHROMATOGRAPHY

23.1. Principle

The explosive is dissolved in gamma-butyrolactone (or acetone) and the cyclohexanone content determined by gas chromatography.

23.2. Apparatus

A gas chromatograph with the following requirements :

The chromatographic column shall be contained in an oven at about 160°C, the temperature shall be controlled to  $\pm 0.1^\circ\text{C}$ .

The detector/amplifier system shall be such that 5.0 mm<sup>3</sup> of 0.001 per cent solution of cyclohexanone in gamma-butyrolactone or acetone, will generate a signal of 75 per cent full scale deflection on a 254 mm potentiometric strip chart recorder, with a noise level from the detector/amplifier or recorder not greater than  $\pm 0.2$  per cent full scale deflection and with a drift of less than 1 per cent per hour. Flame ionization is the recommended method of detection.

The column shall be capable of complete resolution of cyclohexanone from gamma-butyrolactone or acetone within 5 minutes.

The carrier gas flow rate shall be controlled to  $\pm 1$  per cent.

Precision syringes capable of delivery liquid volumes of 5.0 mm<sup>3</sup>.

23.3. Reagent

Gamma-butyrolactone (preferred solvent). The gamma-butyrolactone must be free from any impurity which coincides chromatographically with the cyclohexanone peak. Before use, the gamma-butyrolactone should be fractionally distilled under reduced pressure at about 20 mm Hg rejecting the first 15 per cent and last 10 per cent of the distillate, and retaining the remainder for use in the test.

Acetone (Alternative solvent). Analytical reagent grade.

23.4. Procedure

23.4.1. Preparation of the Calibration Graph. Weigh into 100 cm<sup>3</sup> one-mark volumetric flasks separate portions of cyclohexanone in increments to cover the range 0.001 to 0.020 g. Dilute to 100 cm<sup>3</sup> with gamma-butyrolactone or acetone.

Inject 5.0 mm<sup>3</sup> portion of each solution onto the gas chromatograph column and measure the peak height (or area) of the eluted cyclohexanone. Plot a graph of peak height (or area) against the mass of cyclohexanone.

23.4.2. Analysis of sample. Transfer a representative sample of the explosive (Approximately 40 g) to a clean glass dish. Dry the dish and contents for 2 hours in an oven at 103 ± 2°C. Dissolve 10 ± 0.1 g of the dried sample in 90 cm<sup>3</sup> of the gamma-butyrolactone (or acetone) warming gently if required. Cool the solution and transfer to a 100 cm<sup>3</sup> one-mark volumetric flask and make up to the mark with the appropriate solvent. Inject 5.0 mm<sup>3</sup> of the sample solution into the gas chromatograph column. Measure the peak-height (or area) of the eluted cyclohexanone and determine the amount present (W<sub>1</sub>) by reference to the calibration graph (see 4.1).

23.5. Reporting

$$\% \text{ cyclohexanone} = W_1 \times 10$$

where W<sub>1</sub> = mass of cyclohexanone in sample.

TEST PROCEDURES  
PARAGRAPH 24

DETERMINATION OF THE FIGURE OF INSENSITIVENESS (F of I) OF HMX

24.1. Principle

This method describes the impact sensitiveness tests to be applied to HMX and the assessment of the results.

For approval of the material the tests are required to demonstrate that the estimate of sensitiveness of each approved batch or blend is statistically consistent with the material having an F of I of 55 or above for HMX type III and ensures with 98% confidence that all approved material shall have a true F of I not lower than a certain minimum value (less than the above value) which takes into account the standard deviation of the test.

24.2. Related Documents

Document relevant to this Annex :

Drawing No. D/2242/GA 'TEM Instruments Ltd, Crawley'  
OSRD Report No. 4040 : AMP 'Statistical analysis for a new procedure  
Report No. 101.IR.SRG-PNo.40 in sensitivity experiments'  
July 1944.

Sensitiveness Collaboration Manual of tests, October 1988  
Committee (SCC)

Reference in this method to any related document means the current edition, unless a specific edition is indicated.

24.3. Apparatus and Basic Methods of Test

The apparatus to be used is the Rotter-Type Impact Sensitiveness Machine manufactured by Test Equipment Ltd. (now known as TEM Instruments Ltd), Gatwick Road, Crawley, Sussex (Drawing No. D/2242/GA) using a five kilogram weight. The anvils, brass caps, explosive dispenser, tamping device and gas-measuring burette shall be in accordance with test N° 1A of the SCC Manual of tests.

The procedure to be used is the Bruceton Staircase Technique where the five kilogram weight is dropped onto a succession of brass caps each containing a measured volume of explosive. The selection of an initial drop-height of testing ( $h_0$ ) is usually dictated by experience. If an ignition occurs the next cap is tested at a lower drop-height ( $h_1$ ). If a non-ignition occurs the next cap is tested at a higher drop-height ( $h_1$ ). The difference in drop-heights, ( $h_0 - h_1$ ) or ( $h_1 - h_0$ ) is known as the 'interval' (d) and is normally 10 cm. A change from an ignition to a

non-ignition, or vice versa is termed an 'inversion', and the test starts on the cap preceding the first inversion and consists of 50 caps in total. The statistical implications in the methods are detailed in "Statistical Analysis for a New Procedure in Sensitivity Experiments" (OSRD Report No 4040).

#### Preparation of Apparatus

In addition to normal routine maintenance of the apparatus carry out the following checks before each test run :

Check that the apparatus is level and that the drop-height calibration is correct.

Check that the striker is polished and perfectly flat. When repolishing the striker ensure that the surface remains square to the axis.

Place the assembled impact chamber in position under the drift. Rotate the drift in its guide while looking at the adjoining surfaces of drift and striker. No light should be seen between these surfaces.

Check that :

- The drift moves freely vertically in its guide, and that there is no lateral movement.
- The drift and striker are vertically aligned.

Ensure that the O-ring chamber seal is suitable and that there is adequate grease packing between the striker and screwed bush in the lid of the chamber. Failure to ensure that these gas seals are satisfactory can result in incorrect measurement of gas volumes.

NOTE : A convenient way to test for gas leaks is to assemble the impact chamber with the striker raised into the chamber lid. Close the gas-measuring burette tap and then press down the striker into contact with the anvil pip ; this displaces air from the impact chamber into the burette, if the apparatus is leak-free, the meniscus will stay depressed.

Ensure that the flexible tubing connecting the chamber to the gas-measuring burette is not perished and that the bore is not blocked with debris from previous tests.

Check that the chamber is clean and fit a new anvil ensuring that the clamp ring is fully tightened.

Clean the caps by degreasing in a suitable solvent, dry and store them in a dust-free container. Before use, polish the inside of the caps with a rotating felt bluff.

Ensure that the equipment and samples do not become contaminated by adventitious material eg, airborne dust, as this can affect the results obtained by significantly altering the median height.

#### 24.4. Standard Symbols and Functions required in the Assessment

##### 24.4.1. MH

This refers to the estimate of the median height of an explosive, appropriate to the number of caps tested for 50 % ignition in the test.

An ignition is defined as the evolution of 1 cm<sup>3</sup> or more of products, recorded on the gas-measuring burette, or indicated by a non-standard transient movement of the burette fluid, confirmed by the present of smoke when opening the anvil housing.

##### 24.4.2. MH<sub>std</sub> HMX

The estimate of the median height of the standard HMX (see para 24.8.2).

##### 24.4.3. F of I<sub>std</sub> HMX

The estimate of the Figure of Insensitiveness of the standard HMX (see para 24.8.1).

$$= \frac{\text{MH}_{\text{std HMX}} \times 80}{\text{median height for PERME standard RDX}}$$

##### 24.4.4. S<sub>tr</sub>

This refers to the trend-free standard deviation of median height determinations using 50-cap tests on standard HMX. It is calculated from the mean difference between successive values of median height, neglecting sign ( $\bar{w}_2$ ) in a series of the previous  $N_s$  50-cap determinations of median height on the standard HMX as follows :

$$S_{tr} = \frac{\bar{w}_2}{1.128} \quad \text{where} \quad \bar{w}_2 = \frac{(\sum \text{differences})}{N_s - 1}$$

NOTE : Results from void tests on the standard HMX (see para 24.8.2) are excluded.

24.4.5.  $N_x$ 

This refers to the number of 50-cap runs on the material being tested. The number varies from 1 to 6.

24.4.6.  $N_s$ 

This refers to the number of 50-cap runs on the standard HMX. The number is constant at 6 in this method.

24.4.7.  $SE_p$  or  $I$ 

This refers to the standard error for estimates of the F of I values of HMX when determined against standard HMX. It has been shown to have the value :

$$SE_p \text{ or } I = \frac{F \text{ of } I_{std} \text{ HMX} \times S_{tr}}{MH_{std} \text{ HMX}} \times \sqrt{\frac{N_x + N_s}{N_x \times N_s}}$$

The expression is virtually a constant and is unaffected by Rotter machine response provided the operator's technique is of a high standard.

24.4.8.  $S_i$ 

The estimate of the internal standard deviation of the median height calculated as described in clause 5.

24.5. Calculation of the Median Height and Internal Standard Deviation

A record is kept of the results which is a series of ignitions or non-ignitions at different test drop-heights which are denoted by 'X's and 'O's respectively. The results are tabulated for each drop-height used in the test.

Sum the X's =  $N_x$   
Sum the O's =  $N_o$   
Check  $N_x + N_o = 50$

Assign a value of  $i$  to each drop given by :

$$i = \frac{\text{Drop-Height} - c}{d}$$

where  $c$  = lowest drop-height used  
 $d$  = drop-height interval

Take the lower value of  $N_x$  or  $N_o$ , or if both = 25 take either.



Evaluate  $in_i$  and  $i^2n_i$

where  $i = 0, 2, 3, \dots$  etc, and  $n$  is the number of X's or O's at the relevant drop-height.

Evaluate  $\sum in_i = A$

and  $\sum i^2n_i = B$

Evaluate the Median Height (MH) from :

$$MH = c + d \left( \frac{A}{N_x} - \frac{1}{2} \right) \text{ for X's or}$$

$$MH = c + d \left( \frac{A}{N_o} + \frac{1}{2} \right) \text{ for O's}$$

Evaluate  $M = \frac{NB - A^2}{N^2}$  and determine the Internal Standard Deviation  $S_i$

where  $N = N_x$  (or  $N_o$ ) as appropriate from the Table in the Annex corresponding to  $M$ .

Example of the calculation of the median height, MH, and internal standard deviation  $S_i$

Drop-Height	$i$	$x = n_i$	$O = n_i$	$in_i$	$i^2n_i$
90	0	0	1	0	0
100	1	1	8	8	8
110	2	8	9	18	36
120	3	9	7	21	63
130	4	7	0	0	0
		$N_x = 25$	$N_o = 25$	$A = 47$	$B = 107$

$$MH = 90 + 10 \left( \frac{47}{25} + \frac{1}{2} \right)$$

$$MH = 113.8$$

$$M = \frac{25 \times 107 - 47^2}{25^2}$$
$$= 0.746$$

- $S_1 = 12.6$  (from Table in Appendix)

#### 24.6. Outline of Method

24.6.1 The method uses a Rotter-Impact Test machine to determine the Figure of Insensitiveness of the samples of HMX. The standard HMX shall be tested first (with the provisos of clauses 24.6.3 and 24.8.2), followed by the HMX sample(s). A new test on the standard HMX shall be carried out on each test day.

24.6.2 The F of I values quoted in clause 24.6.1 shall be referred ultimately to RARDE standard RDX for which the F of I has been assigned the value of 80. The first 'Standard HMX' batch was standardized against PERME 'Standard RDX'. Subsequent batches are to be standardized against the preceding sample of 'Standard HMX'. By this means parameters external to the material, such as machine and operator, can be expected to affect both the batch and standard equally.

24.6.3 The estimate of the median height of the HMX standard is to be assessed from the running mean of the last six 50-cap determinations with a proviso that for a satisfactory running mean it shall always be demonstrated that the median height from the most recent 50-cap run is from the same population as the previous six runs. If such agreement is not found for any run, testing shall be continued until two satisfactory consecutive runs are obtained. In this way a sensitive control on any external changes to the explosives, such as machine and operator variables, is obtained and at the same time 300 caps provide a sufficiently accurate value of the median height of the standard in the absence of other variables. The above technique, to control the validity of the running mean, is additional to an organized schedule to control the mechanical condition of the machine.

24.6.4 The median height and Figure of Insensitiveness of test material is first estimated from a single 50-cap test. If the estimate of F of I is not less than two standard errors above a value referred to as the 'REJECTION LIMIT' (see below) the material may be accepted. If the F of I lies between this acceptance limit and the rejection limit, retesting up to a total of 6 x 50-cap runs is permissible. In such cases the cumulative value of the F of I is used together with a new and lower value of standard error for the

test appropriate to the number of caps in the cumulative run. The 'REJECTION LIMIT' referred to above represents the lowest value for the true F of I of any material which has been found acceptable, calculated for a 98 % degree of confidence. For HMX Type III the 'REJECTION LIMIT' is the F of I which is equivalent to a median height of two trend-free, standard deviations, below the mean median height of HMX Type III, having an F of I of 55 (during 1966).

24.6.5 The assessment provides for cases where a minority of the tests produce F of I values below the rejection limit.

24.6.6 A maximum is set for the internal standard deviation of a test, for it to be considered satisfactory.

#### 24.7. Sampling and Sample Preparation

A set of up to six separate representative samples shall be taken from the material to be tested. Transfer a representative portion of approximately 10 g of each of the samples required for the determination of the Figure of Insensitiveness to a clean glass dish. Dry the dish and its contents in an oven at  $103 \pm 2^\circ\text{C}$  for 2 hours.

Remove from the oven, cool and place in a desiccator. These samples must be prevented from becoming contaminated by airborne or other adventitious matter.

Using the explosive dispenser and tamping device fill 55 caps with the standard and 55 caps with the sample. For finely divided samples spread the explosive evenly over the base of the cap using a polished brass drift.

Store the filled caps in covered dust-free containers in the test bay for at least 12 hours immediately preceding testing.

#### 24.8. Maintenance of Standards

##### 24.8.1. F of I Standard HMX (F of $I_{std}$ HMX)

A supply of HMX shall be set aside as a standard. Its F of I shall be determined against the laboratory stock of PERME Standard RDX, using the results from at least 6 x 50-cap runs on each material. The range of median height values in each of the groups of six or more shall not exceed  $3S_{tr}$ , and the mean  $S_{tr}$  and the mean  $S_i$  for the 6 x 50-caps in each case shall be within the limits  $17 \pm 4$  cm. (A mean  $S_i$  value of 17.3 has been found over a large number of determinations of 50-cap runs on HMX when the testing was on a linear scale of drop-height with an interval of 10 cm).

Before the supply of standard HMX is exhausted a further batch shall be selected to succeed it and its F of I determined as above against the existing standard batch. A self-consistent series of standard batches of HMX will thus be maintained in the laboratory which will be independent of the RDX standard after calibration of the first batch.

#### 24.8.2. Median Height of Standard HMX (MHStd HMX)

A running mean of 6 x 50-cap determinations of the median height of the current HMX standard shall be maintained. The running mean shall be updated continuously while testing is in progress and at least one 50-cap run shall be made on the standard on the same day as each test run. If the value of  $S_i$  exceeds 30 cm (ca  $2S_i$ ) the run is declared void and shall be replaced by an additional run. When the latest 50-cap run on the standard gives a median height value which differs from the current value of the running mean by more than  $2S_i$ , it is still used to calculate the next value of the running mean but further 50-cap runs shall be made and new values of the running mean calculated until two successive 50-cap median-height values have been obtained within  $2S_i$ , or 5 cm (whichever is the greater) of the latest running mean.

This requirement minimizes error caused by shift in response of the Rotter machine. While a new stable value for the median height of the standard is being established in this way, no testing of any material shall be made.

#### 24.9. Procedure for Testing Sample Material

##### 24.9.1. F of I Requirements

A sequence of up to six 50-cap runs is made on the test material until a decision is reached. The F of I is calculated from the median height value for each 50-cap run by the expression :

$$F \text{ of } I = \frac{F \text{ of } I_{std} \text{ HMX} \times MH \text{ test material}}{MH_{std} \text{ HMX}}$$

If the F of I of the test material is not less than the 1 x 50-cap 'ACCEPTANCE LIMIT' (see clause 24.10.2) the test material shall be accepted. If it is less, a further 50-caps shall be tested and a F of I calculated from the 2 x 50-cap runs. If this is not less than the 2 x 50-cap 'ACCEPTANCE LIMIT' the material shall be accepted. This procedure is continued if necessary through three, four, five or six 50-cap runs using the appropriate acceptance limit in each case. If two 50-cap median-height values fall below a fixed value known as the 'REJECTION LIMIT' (see clause

10.1), the material shall be rejected.

NOTE 1 : Only one 50-cap run may be carried out on any single batch or blend during any one day unless the test is void.

NOTE 2 : If the cumulative 6 x 50-cap run has a median height between the acceptance and rejection limits of the material the Quality Assurance Representative and the Design Authority shall be consulted.

#### 24.9.2. S<sub>i</sub> Requirements

The internal standard deviation of the median height (S<sub>i</sub>) for each 50-cap run shall be calculated. If the value exceeds 30 cm, the run is void and shall be replaced by an additional run on another sample from the same test material. If any two samples from the same test material gives values of S<sub>i</sub> exceeding 30 cm, the material is rejected.

#### 24.10. 'Rejection' and 'Acceptance' Limits

##### 24.10.1. Rejection limit (F of IREJ)

This is the F of I corresponding to the established median height of the standard HMX (MH<sub>std HMX</sub>) less two trend-free standard deviations of the median height (S<sub>tr</sub>) :

$$F \text{ of } I_{REJ} = \frac{F \text{ of } I_{std HMX} (MH_{std HMX} - 2S_{tr})}{MH_{std HMX}}$$

##### 24.10.2. Acceptance Limit (F of IAcc)

The F of I acceptance limit (F of IAcc) for each stage of the sequential test (1 x 50-caps, 2 x 50-caps etc) are obtained by adding twice the standard error of the F of I, obtained from the equation for SE<sub>F of I</sub> in para 4.7 to the rejection limit (F of I<sub>REJ</sub>). Thus the acceptance limit after N<sub>x</sub> x 50-caps runs is:

$$F \text{ of } IAcc = (F \text{ of } I_{REJ}) + \left[ \frac{(F \text{ of } I_{std HMX} \times 2S_{tr})}{MH_{std HMX}} \times \sqrt{\frac{N_x + 6}{6N_x}} \right]$$

Where 6 is the fixed number of 50-caps runs (N<sub>6</sub>) on the standard HMX.

##### 24.10.3. Example of the Calculation and Use of the Limits

Assuming that the required parameters for the standard HMX have been obtained, (paras 24.6.3, 24.6.4, 24.8) and are as follows:

$$MH_{STD} \text{ HMX} = 89.3 \text{ cm}$$

$$F \text{ of } I_{std} \text{ HMX} = 55$$

$$S_{tr} = 5 \text{ cm}$$

Then by clause 24.10.1

$$F \text{ of } I_{REJ} = \frac{55 (89.3 - 10)}{89.3} = 48.8$$

and by clause 24.10.2

$$F \text{ of } I_{Acc} = 48.8 + \frac{55 \times 10}{89.3} \times \sqrt{\frac{N_x + 6}{6N_x}}$$

where  $N_x$  can be from 1 ... 6 (50-cap runs)

$$= 48.8 + 6.16 \times \sqrt{\frac{N_x + 6}{6N_x}}$$

$$(= FI_{REJ} + 2SE_{F \text{ of } I}) \text{ (see clause 24.4.7)}$$

The following table can be constructed for  $N_x = 1 \dots 6$

Number of 50-cap runs ( $N_x$ )	F of $I_{REJ}$	$2SE_{F \text{ of } I}$ Twice the Standard Error of the F of I	F of $I_{Acc}$
1		6.6	55.4
2	↑	5.0	53.8
3		4.4	53.2
4	48.8	4.0	52.8
5		3.7	53.5
6	↓	3.6	52.4

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If, using the above illustrative parameters, a sequence of tests on test material gave the following results :

1st 50-cap run F of I = 52 (not acceptable)

2nd 50-cap run F of I = 56 (not sentenced on this run alone).

Cumulative answer for 2 x 50-cap runs F of I = 54 (acceptable).

The material would be accepted after the 2nd run and there would be 98 % confidence that the true F of I of the material were above 48.8.

## APPENDIX TO PARA 24

INTERNAL STANDARD DEVIATIONS

M	Si	M	Si	M	Si	M	Si
	5.0	0.530-0.535	9.1	0.783-0.788	13.2	1.036-1.043	17.3
0.298-0.302	.1	0.536-0.542	.2	0.789-0.795	.3	1.044-1.049	.4
0.303-0.306	.2	0.543-0.548	.3	0.796-0.801	.4	1.050-1.055	17.5
0.307-0.311	.3	0.549-0.554	.4	0.802-0.807	13.5	1.056-1.061	.6
0.312-0.316	.4	0.555-0.560	9.5	0.808-0.813	.6	1.062-1.068	.7
0.317-0.321	5.5	0.561-0.566	.6	0.814-0.819	.7	1.069-1.074	.8
0.322-0.326	.6	0.567-0.572	.7	0.820-0.826	.8	1.095-1.080	.9
0.327-0.332	.7	0.573-0.579	.8	0.827-0.832	.9	1.081-1.086	18.0
0.333-0.337	.8	0.580-0.585	.9	0.833-0.838	14.0	1.087-1.093	.1
0.338-0.342	.9	0.586-0.590	10.0	0.839-0.844	.1	1.094-1.099	.2
0.343-0.348	6.0	0.590-0.597	.1	0.845-0.850	.2	1.100-1.105	.3
0.349-0.354	.1	0.598-0.603	.2	0.851-0.857	.3	1.106-1.111	.4
0.355-0.359	.2	0.604-0.609	.3	0.858-0.863	.4	1.112-1.117	18.5
0.360-0.365	.3	0.610-0.615	.4	0.864-0.869	14.5	1.118-1.124	.6
0.366-0.371	.4	0.616-0.622	10.5	0.870-0.875	.6	1.125-1.130	.7
0.372-0.377	6.5	0.623-0.628	.6	0.876-0.881	.7	1.131-1.136	.8
0.378-0.383	.6	0.629-0.634	.7	0.882-0.888	.8	1.137-1.142	.9
0.384-0.389	.7	0.635-0.640	.8	0.890-0.894	.9	1.143-1.149	19.0
0.390-0.395	.8	0.641-0.646	.9	0.895-0.900	15.0	1.150-1.155	.1
0.396-0.401	.9	0.647-0.653	11.0	0.901-0.906	.1	1.156-1.161	.2
0.402-0.407	7.0	0.654-0.659	.1	0.907-0.912	.2	1.162-1.167	.3
0.408-0.413	.1	0.660-0.665	.2	0.913-0.919	.3	1.168-1.173	.4
0.414-0.419	.2	0.666-0.671	.3	0.920-0.925	.4	1.174-1.180	19.5
0.420-0.425	.3	0.672-0.677	.4	0.926-0.931	15.5	1.181-1.186	.6
0.426-0.431	.4	0.678-0.683	11.5	0.932-0.937	.6	1.187-1.192	.7
0.432-0.437	7.5	0.684-0.689	.6	0.938-0.944	.7	1.193-1.198	.8
0.438-0.443	.6	0.690-0.696	.7	0.945-0.950	.8	1.199-1.205	.9
0.444-0.449	.7	0.697-0.702	.8	0.951-0.956	.9	1.206-1.211	20.0
0.450-0.455	.8	0.703-0.708	.9	0.957-0.962	16.0	1.212-1.217	.1
0.456-0.462	.9	0.709-0.714	12.0	0.963-0.968	.1	1.218-1.223	.2
0.463-0.468	8.0	0.715-0.720	.1	0.969-0.974	.2	1.224-1.230	.3
0.469-0.474	.1	0.721-0.727	.2	0.975-0.981	.3	1.231-1.236	.4
0.475-0.480	.2	0.728-0.733	.3	0.982-0.987	.4	1.237-1.242	20.5
0.481-0.486	.3	0.734-0.739	.4	0.988-0.993	16.5	1.243-1.248	.6
0.487-0.492	.4	0.740-0.745	12.5	0.994-0.999	.6	1.249-1.254	.7
0.493-0.498	8.5	0.746-0.751	.6	1.000-1.006	.7	1.255-1.261	.8
0.499-0.505	.6	0.752-0.757	.7	1.007-1.012	.8	1.262-1.267	.9
0.506-0.511	.7	0.758-0.764	.8	1.013-1.018	.9	1.268-1.273	21.0
0.512-0.517	.8	0.765-0.770	.9	1.019-1.024	17.0	1.274-1.279	.1
0.518-0.523	.9	0.771-0.776	13.0	1.025-1.030	.1	1.280-1.286	.2
0.524-0.529	9.0	0.777-0.782	.1	1.031-1.037	.2	1.287-1.292	.3



## APPENDIX (CONT.....)

M	Si	M	Si	M	Si	M	Si
1.293-1.298	21.4	1.549-1.554	25.5	1.805-1.810	29.6	2.061-2.066	33.7
1.299-1.304	.5	1.555-1.560	.6	1.811-1.816	.7	2.067-2.072	.8
1.305-1.311	.6	1.561-1.566	.7	1.817-1.822	.8	2.073-2.079	.9
1.312-1.317	.7	1.567-1.573	.8	1.823-1.829	.9	2.080-2.085	34.0
1.318-1.323	.8	1.574-1.579	.9	1.830-1.835	30.0	2.086-2.091	.1
1.324-1.329	.9	1.580-1.585	26.0	1.836-1.841	.1	2.092-2.097	.2
1.330-1.336	22.0	1.586-1.591	.1	1.842-1.847	.2	2.098-2.104	.3
1.337-1.342	.1	1.592-1.598	.2	1.848-1.854	.3	2.105-2.110	.4
1.343-1.348	.2	1.599-1.604	.3	1.855-1.860	.4	2.111-2.116	34.5
1.349-1.354	.3	1.605-1.610	.4	1.861-1.866	30.5	2.117-2.122	.6
1.355-1.360	.4	1.611-1.616	26.5	1.867-1.872	.6	2.123-2.129	.7
1.361-1.367	22.5	1.617-1.623	.6	1.873-1.879	.7	2.130-2.135	.8
1.368-1.373	.6	1.624-1.629	.7	1.880-1.885	.8	2.136-2.141	.9
1.374-1.379	.7	1.630-1.636	.8	1.886-1.891	.9	2.142-2.147	35.0
1.380-1.385	.8	1.637-1.641	.9	1.892-1.897	31.0	2.148-2.154	.1
1.386-1.392	.9	1.642-1.648	27.0	1.898-1.904	.1	2.155-2.160	.2
1.393-1.398	23.0	1.649-1.654	.1	1.905-1.910	.2	2.161-2.166	.3
1.399-1.404	.1	1.655-1.660	.2	1.911-1.916	.3	2.167-2.172	.4
1.405-1.410	.2	1.661-1.666	.3	1.917-1.922	.4	2.173-2.179	35.5
1.411-1.417	.3	1.667-1.672	.4	1.923-1.929	31.5	2.180-2.185	.6
1.418-1.423	.4	1.673-1.679	27.5	1.930-1.935	.6	2.186-2.191	.7
1.424-1.429	23.5	1.680-1.685	.6	1.936-1.941	.7	2.192-2.197	.8
1.430-1.435	.6	1.686-1.691	.7	1.942-1.947	.8	2.198-2.204	.9
1.436-1.442	.7	1.692-1.697	.8	1.948-1.954	.9	2.205-2.210	36.0
1.443-1.448	.8	1.689-1.704	.9	1.955-1.960	32.0	2.211-2.216	.1
1.449-1.454	.9	1.705-1.710	28.0	1.961-1.966	.1	2.217-2.222	.2
1.455-1.460	24.0	1.711-1.716	.1	1.967-1.972	.2	2.223-2.229	.3
1.461-1.466	.1	1.717-1.722	.2	1.973-1.979	.3	2.230-2.235	.4
1.467-1.473	.2	1.723-1.729	.3	1.980-1.985	.4	2.236-2.241	36.5
1.474-1.479	.3	1.730-1.735	.4	1.986-1.991	32.5	2.242-2.247	.6
1.480-1.485	.4	1.736-1.741	28.5	1.992-1.997	.6	2.248-2.254	.7
1.486-1.491	24.5	1.742-1.747	.6	1.998-2.004	.7	2.255-2.260	.8
1.492-1.490	.6	1.748-1.754	.7	2.005-2.010	.8	2.261-2.266	.9
1.499-1.504	.7	1.755-1.760	.8	2.011-2.016	.9	2.267-2.273	37.0
1.505-1.510	.8	1.761-1.766	.9	2.017-2.022	33.0	2.274-2.279	.1
1.511-1.516	.9	1.767-1.772	29.0	2.023-2.029	.1	2.280-2.285	.2
1.517-1.523	25.0	1.773-1.779	.1	2.030-2.035	.2	2.286-2.291	.3
1.524-1.529	.1	1.780-1.785	.2	2.036-2.041	.3	2.292-2.298	.4
1.530-1.535	.2	1.786-1.791	.3	2.042-2.047	.4	2.299-2.304	37.5
1.536-1.541	.3	1.792-1.797	.4	2.048-2.054	33.5	2.305-2.310	.6
1.542-1.548	.4	1.798-1.804	29.5	2.055-2.060	.6	2.311-2.316	.7
						2.317-2.322	.8
						2.323-2.328	.9