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

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22 November 1999

MAS/1436-PPS/4556

See CNAD AC/310 STANAG distribution

STANAG 4556 PPS (EDITION 1) - EXPLOSIVES: VACUUM STABILITY TEST

Reference:

AC/310-D/156 dated 25 February 1998 (Edition 1) (Ratification draft)

1. The enclosed NATO Standardization Agreement which has been ratified by nations as reflected in page iii is promulgated herewith.
2. The reference listed above is to be destroyed in accordance with local document destruction procedures.
3. AAP-4 should be amended to reflect the latest status of the STANAG.

ACTION BY NATIONAL STAFFS

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



A. GRØNHEIM
Major General, NOAF
Chairman MAS

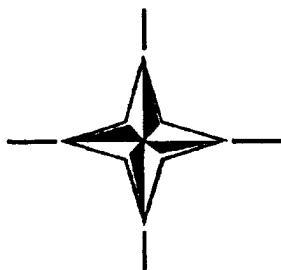
Enclosure:

STANAG 4556 (Edition 1)

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STANAG No. 4556
(Edition 1)

**NORTH ATLANTIC TREATY ORGANIZATION
(NATO)**

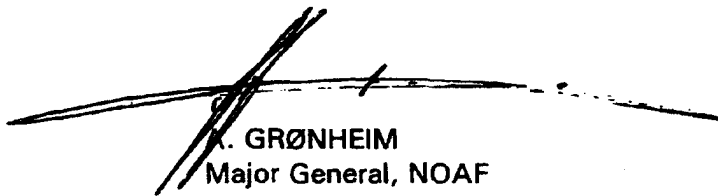


**MILITARY AGENCY FOR STANDARDIZATION
(MAS)**

**STANDARDIZATION AGREEMENT
(STANAG)**

SUBJECT: EXPLOSIVES: VACUUM STABILITY TEST

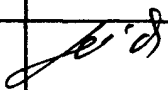
Promulgated on 22 November 1999


K. GRØNHEIM
Major General, NOAF
Chairman, MAS

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RECORD OF AMENDMENTS

No.	Reference/date of amendment	Date entered	Signature
1	MPS 10267-PPS 14556 v. 20.02.01	28.02.02	

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 "In NATO Standardization, the fulfilment by which a member nation formally accepts, with or without reservation, the content of a Standardization Agreement" (AAP-6).
5. Implementation is "In NATO Standardization, the fulfilment by a member nation of its obligations as specified in a Standardization Agreement" (AAP-6).
6. Reservation is "In NATO Standardization, the stated qualification by a member nation that describes the part of a Standardization Agreement that it will not implement or will implement only with limitations" (AAP-6).

RATIFICATION, IMPLEMENTATION AND RESERVATIONS

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

FEEDBACK

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

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NAVY/ARMY/AIR

NATO STANDARDIZATION AGREEMENT
(STANAG)

EXPLOSIVES: VACUUM STABILITY TEST

Annexes:

- A: Test procedures
- B: Data sheet for the vacuum stability test
- C: Figures

Related Documents: None

AIM

1. The aim of this agreement is to standardize test procedures for the determination of the thermal stability of explosive materials by using the vacuum stability test.

AGREEMENT

2. The NATO Participating Nations of the ratifying countries agree to adopt the test procedures described in Annex A for the determination of the thermal stability of explosive materials by means of the vacuum stability test and to use the data sheet at Annex B for reporting test results.

GENERAL

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

IMPLEMENTATION OF THE AGREEMENT

4. The STANAG will be considered implemented when ratifying countries comply with the test procedure described in this STANAG for the vacuum stability test.

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TEST PROCEDURES**1. PRINCIPLE**

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

A sample of the explosive compound is heated for a specified time in an evacuated tube in a heating bath maintained at a constant specified temperature. The volume of gas evolved is determined by either a mercury manometric method or by using a pressure transducer. A minimum of two tests shall be conducted on each material to be tested.

The mass of explosive, the temperature and duration of the test together with the allowable upper limit of the volume (at STP) of gas evolved is prescribed in the specification for the particular explosive under test.

The most commonly used test conditions are given below:

(a)	test duration :	40 hours
(b)	test temperature - explosives :	100°C, 120°C
	single base propellants :	100°C
	double base propellants :	90°C
	nitrate ester plasticized polyether (NEPE) propellants :	90°C
	composite propellants :	100°C
	pyrotechnics :	100°C

The test conditions used shall be recorded on the data sheet for the particular explosive being tested.

2. APPLICABILITY

The test is applicable to solid high explosives, propellants and pyrotechnics used in conventional armaments. However, some authorities require supporting data from other tests when this test is applied to propellants and pyrotechnics.

3. SPECIMEN

The explosive specimen shall consist of a 5.0 ± 0.01 g representative portion of the explosive or such mass as shall be prescribed in the relevant specification. For the testing of primary explosives or explosives of similar sensitivity, the specimen weight shall be in the range 0.20 to 0.50 g.

The specimen shall be dried or prepared in accordance with the requirements for the explosive under test.

NOTE: Moisture and humidity may play a significant role in gas evolution when pyrotechnic compositions are tested and it may, therefore, be inappropriate to dry these specimens to the extent applicable to high explosives or propellants.

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With necessary precautions, grind, rasp or subdivide the explosive to pass through the 2 mm sieve. Discard material which passes the 0.2 mm sieve. When the explosive contains a major ingredient (such as trinitrotoluene) which melts during the course of the test, a larger grit size is permissible. Explosives which do not require grinding or subdivision to pass a 2 mm sieve shall not be sieved before use to remove fine particles.

4. PROCEDURE 1, VACUUM STABILITY TEST (MANOMETER METHOD)**4.1 Apparatus**

- a. Constant temperature bath able to maintain the tube containing the sample at the desired temperature to within $\pm 0.5^{\circ}\text{C}$.

The following alternatives may be used:

- Metal block type: an electrically heated block controllable to within $\pm 0.2^{\circ}\text{C}$ and insulated on all sides. The block shall contain holes provided with appropriate reducing sleeves to accommodate the total length of the sample heating tubes. The inner diameter of the sleeves shall be approximately 1 to 2 mm greater than the diameter of the heating tube. The centre of the holes shall be more than 30 mm from the edge of the block.

or

- Thermostatically controlled bath filled with a liquid appropriate for the specified temperature, controllable to within $\pm 0.2^{\circ}\text{C}$.

For each alternative an electrical cut-out shall be fitted which operates at 5°C above the nominal working temperature. A calibrated 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. A continuous recording temperature device is recommended.

- b. Vacuum stability measuring apparatus in glass comprising a heating tube (numbered) and a capillary tube as illustrated in Annex C, Fig. 1, with a free volume of $25 \pm 2 \text{ cm}^3$.
- The tube and manometer, particularly the latter, are rather delicate and great care must be taken at all times in handling and storage.
- c. Vacuum system:
- Vacuum pump able to attain a pressure of less than 5 mm mercury absolute (6.7 mbar).
 - Vacuum line, with rubber adapter for connection to the capillary reservoir; a T-bore stopcock in the vacuum line may be useful.
 - Pressure gauge, indicating the range 0 to 20 mm Hg absolute.
- d. Barometer, calibrated.
- e. Mercury thermometer or thermometric gauge, calibrated, readable to 0.2°C .
- f. Analytical balance, readable to 1 mg.
- g. Sieves with 2 mm and 0.2 mm aperture sizes.

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- h. Petroleum jelly or a high vacuum silicone grease as a lubricant, not evolving gas at the specified test temperature and compatible with the explosive to be tested.

Compatibility test:

Add 0.015 g of the lubricant under test to 5.0 ± 0.01 g of the explosive and mix well. Carry out the compatibility test on the mixture at the specified 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^3 .

- i. Mercury, clean and dry.

4.2 Calibration of the vacuum stability apparatus

Calibrate the vacuum stability measuring apparatus as follows:

a. Heating tube

- Determine the volume of the heating tube to 0.1 cm^3 by filling it with a suitable liquid until this reaches the level at which it will contact the ground glass joint of the capillary tube. Determine the mass of the liquid to 10 mg and the temperature to the nearest 0.5°C .
- The volume of the heating tube is:

$$V_t = \frac{m}{d}$$

where: V_t = volume of the heating tube (in cm^3).
 m = mass of the liquid (in g).
 d = density of the liquid at the calibration temperature $t^\circ\text{C}$ (in g/cm^3).

b. Capillary tube

- Determine the unit capacity of the capillary to $0.0001 \text{ cm}^3/\text{mm}$ by placing approximately 10 g, weighed to 0.01 g, of mercury in the reservoir and manipulate the tube so that all the mercury passes into the long (850 mm) section of the capillary. Ensure that the mercury remains as a continuous column. Measure the length of the mercury column at three positions in the long section of the capillary and calculate the average of the three measurements.
- Measure the temperature of the mercury to the nearest 0.5°C .
- Calculate the unit capacity of the capillary, using the following formula:

$$C = \frac{m}{d \cdot L}$$

where: C = unit capacity of the capillary (in cm^3/mm).
 m = mass of the mercury (in g).
 d = density of the mercury at the calibration temperature $t^\circ\text{C}$ (in g/cm^3).
 L = average length of the mercury column (in mm).

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4.3 Stability Testing

- a. Transfer a sample of the prepared explosive or propellant 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). A minimum of two tests shall be conducted.
- b. Coat the ground glass joint of the capillary tube with a light film of the lubricant (use the minimum amount of lubricant to ensure a good joint). Make an airtight connection between the heating tube and the capillary by pressing the tube up against the capillary with a gentle twisting motion, until the interface of the joint is clear.
Take care to prevent the tube falling from the manometer before it is held by the vacuum.
- c. Mount the assembled apparatus in a suitable rack so that the long section of the capillary is nearly vertical and the bottom reservoir rests on a solid support.
- d. Add to the capillary reservoir a sufficient amount of clean mercury to fill the capillary and the reservoir after evacuation (about 7 cm³ or one-third filled).
- e. Connect the capillary reservoir to the vacuum pump using a vacuum line and rubber adapter. Tilt the assembly to bring the reservoir towards the horizontal until the capillary opening is free of mercury. Evacuate the heating tube and manometer until the pressure is reduced to a maximum of 5 mm of mercury (6.7 mbar). Tap the heating tube lightly to facilitate release of any occluded air from the sample.
- f. When the evacuation is completed, return the capillary to the vertical. Allow mercury to enter the capillary by slowly admitting air in the vacuum line through the stopcock. This operation must be done very carefully to prevent the mercury rising too quickly in the manometer tube. Disconnect the vacuum line. Pour a little mercury into the cup of the heating tube to serve as a secondary seal and cover the mercury with a thin layer of glycerol or water to prevent evaporation of the mercury.
- g. 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) to 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) to 1 mm.
 - The room temperature at the beginning of the test (t_1) to 0.5°C.
 - The barometric pressure at the beginning of the test (P_1) to 0.5 mm Hg.
- h. Ascertain that the bath temperature is constant at the specified temperature. Carefully place the heating tube of the prepared test assembly in the constant temperature bath, being careful not to loosen the connection between the heating tube and the capillary. Support the capillary reservoir.
- i. Heat the tube for 40 hours (unless otherwise specified).
- j. Remove the tube from the constant temperature bath and allow to cool to room temperature.
- k. 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) to 1 mm.

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- The height of the mercury column above the surface of the mercury in the reservoir at the end of the test (H_2) to 1 mm.
- The room temperature at the end of the test (t_2) to 0.5°C.
- The barometric pressure at the end of the test (P_2) to 0.5 mm Hg.
- Any changes in colour or physical appearance of the explosive or propellant and any appearance of condensation on the walls of the tube.

4.4 Calculation

Calculate the volume of gas, at standard temperature and pressure (273 K and 760 mm Hg), liberated from the sample during the test as follows:

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

with: $A = V_t - \frac{m}{d}$

where:	V	=	volume of gas liberated from the sample (in cm ³ , at STP).
	A	=	available gas space in the heating tube (in cm ³).
	B ₁	=	total length of the capillary tube minus the vertical height of the column of mercury in the reservoir at the beginning of the test (in mm).
	B ₂	=	total length of the capillary tube minus the vertical height of the column of mercury in the reservoir at the end of the test (in mm).
	C	=	unit capacity of the capillary tube (in cm ³ /mm).
	H ₁	=	height of the mercury column above the surface of the mercury in the reservoir at the beginning of the test (in mm).
	H ₂	=	height of the mercury column above the surface of the mercury in the reservoir at the end of the test (in mm).
	P ₁	=	barometric pressure at the beginning of the test (in mm Hg).
	P ₂	=	barometric pressure at the end of the test (in mm Hg).
	t ₁	=	room temperature at the beginning of the test (in °C).
	t ₂	=	room temperature at the end of the test (in °C).
	V _t	=	volume of the heating tube (in cm ³).
	m	=	mass of the explosive (in g).
	d	=	density of the explosive (in g/cm ³).

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4.5 Dismantling

Remove the mercury from the cup 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 it 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.

4.6 Cleaning

Empty the heating tube and remove residual traces of the sample 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.

5. PROCEDURE 2, VACUUM STABILITY TEST (TRANSDUCER METHOD)

NOTE: For Procedure 2, different assemblies for the connection between heating tubes and pressure transducers are possible. Examples for the assemblies are shown in Annex C, Fig. 2 and 3.

Also for the calibration and the stability testing, different methods are possible depending on the assembly used, as presented in Procedures 2A and 2B.

Minor modifications are permissible but they should be mentioned in the Data Sheet.

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5.1 Apparatus

a. Constant temperature bath, metal block type:

- An electrically heated metal block controllable to within $\pm 0.2^\circ\text{C}$. The block shall contain holes provided with appropriate reducing sleeves and able to accommodate the total length of the sample heating tubes and transducers (if possible). The dimensions of the sleeves shall be such that the diameter is approximately 1 to 2 mm greater than the diameter of the heating tube.
- An electrical cut-out shall be fitted which operates at 5°C above the nominal working temperature. A calibrated 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. A continuous recording temperature device is recommended. An automatic overpressure cut-out device is permitted.

b. Transducers:

- The transducers must be capable of operation in the pressure range 0 to 1 bar absolute and be able to detect a variation in volume of $\pm 0.02 \text{ cm}^3$. The transducers should be allowed to stabilize for approximately 30 minutes after power up.
- The transducers convert a fluid's variation to pressure exerted on a membrane into an electric signal by the imbalance of a Wheatstone bridge formed by four active strain gauges.

See also the Note in Section 5.2.

The transducers can be attached to the heating tubes as follows:

- with the steel male part of a ground metal joint. This part is welded to the transducer and includes a hole for gas extraction (see Annex C, Fig. 2).
- with an adapter which is fitted to the transducer with a hexagon nut. A gastight fit is made to the heating tube using two O-rings. The adapter includes an evacuation port (see Annex C, Fig. 3).

c. Heating tubes in glass:

The tubes should be of uniform bore with a wall thickness of 2 mm. All the tubes must be fitted to the transducers so that each transducer-tube assembly has a free volume of $25 \pm 2 \text{ cm}^3$.

Examples of suitable test apparatus are shown in Annex C, Fig. 2 and 3.

- d. Digital voltmeters or data-logging equipment, capable of measuring the output voltage of the pressure transducer used.
A continuous recording pressure device is recommended.
- e. Vacuum pump, able to attain a pressure of less than 5 mm mercury absolute (6.7 mbar).
- f. Mercury thermometer or thermometric gauge, calibrated, readable to 0.2°C .
- g. Barometer, calibrated.
- h. Pressure gauge indicating the range 0 to 20 mm mercury absolute (0 to 27 mbar).
- i. Analytical balance, readable to 1 mg.

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- j. Sieves with 2 mm and 0.2 mm aperture sizes.
- k. Calibrated gastight syringe of 5 cm³ capacity (for Procedure 2A).
- l. Petroleum jelly or a high vacuum silicone grease as a lubricant, not evolving gas at the specified test temperature and compatible with the explosive to be tested.

Compatibility test:

Add 0.015 g of the lubricant under test to 5.0 ± 0.01 g of the explosive and mix well. Carry out the compatibility test on the mixture at the specified 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 cm³.

5.2 Stability Testing (Procedure 2A)

NOTE: See also Annex C, Fig. 2. This procedure uses pressure transducers accompanied by calibration certificates supplying the information necessary for the conversion of the voltage measured into pressure. This formula can be of a polynomial type. Corrections must be applied to transducers not temperature-compensated when they are used at a temperature different to that at which they were calibrated.

5.2.1 Calibration of the apparatus

a. Heating tube:

Determine the volume of the heating tube to 0.1 cm³ by filling the tube with a suitable liquid until this reaches the level at which it will contact the conical part of the transducer adapter. Determine the mass of the liquid to 10 mg and the temperature to 0.5°C.

The volume of the heating tube (V_t) is given by:

$$V_t = \frac{m}{d}$$

where: V_t = volume of the heating tube (in cm³).

m = mass of the added liquid (in g).

d = density of the liquid at the calibration temperature t°C (in g/cm³).

b. Transducer/adapter:

Determine the volume of the transducer with adapter to 0.1 cm³ by filling these with a suitable liquid until this reaches the end of the conical part. The opening in the conical part has to be sealed (by means of adhesive tape, for example). Determine the mass of the liquid to 10 mg and the temperature to 0.5°C.

The volume of the transducer and adapter (V_c) is given by:

$$V_c = \frac{m}{d}$$

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where: V_c = volume of the transducer and adapter (in cm^3).
 m = mass of the added liquid (in g).
 d = density of the liquid at the temperature $t^\circ\text{C}$ (in g/cm^3).

5.2.2 Method

- Transfer a sample of the prepared explosive 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). A minimum of two tests shall be conducted on each material to be tested.
- Coat the conical part of the transducer-adapter with a light film of the lubricant (use the minimum amount of lubricant to ensure a good joint). Make an airtight connection between the heating tube and the adapter by pressing the tube up against it with a gentle twisting motion, until the interface of the joint is clear.
Take care to prevent the tube falling from the adapter before it is held by the vacuum.
- Twist the heating tube so that the holes in the joints correspond. Verify that these are free of lubricant.
Connect the assembly to the vacuum pump using a vacuum line and rubber adapter. Evacuate the assembly until the pressure is reduced to a maximum of 5 mm of mercury (6.7 mbar). Tap the heating tube lightly to facilitate release of any occluded air from the sample. Twist the heating tube carefully over 180° .
- Record the voltmeter reading of the transducer at room temperature and check for any drift on the voltmeter. The transducers need a warming up period to allow stabilization.
- Record the voltmeter reading of the transducer (R_1) and the room temperature at the beginning of the test ($t_1^\circ\text{C}$).
- Ascertain that the bath temperature is constant at the specified temperature. Carefully place the heating tube of the prepared test assembly in the constant temperature bath.
- Heat the tube for 40 hours (unless otherwise specified). Check the bath temperature every day.
- Remove the tube from the constant temperature bath and allow to cool to room temperature.
- Record the voltmeter reading (R_2) and the room temperature at the end of the test ($t_2^\circ\text{C}$).
- Any changes in colour or physical appearance of the explosive or propellant and any appearance of condensation on the walls of the tube.

5.2.3 Calculation

Calculate the gas pressures P_1 and P_2 (in bar) corresponding to the voltmeter readings R_1 and R_2 , using the calibration graphs of the transducer.

Calculate the volume of gas V (at 273 K and 1.013 bar) liberated during the test, as follows:

$$V = \left[V_c + V_t - \frac{m}{d} \right] \times \left[\frac{P_2 \times 273}{273 + t_2} - \frac{P_1 \times 273}{273 + t_1} \right] \times \frac{1}{1.013}$$

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where:	V	=	volume of gas liberated from the sample (in cm ³ , at STP).
	V _c	=	volume of the transducer and adapter (in cm ³).
	V ₁	=	volume of the heating tube (in cm ³).
	m	=	mass of the sample (in g).
	d	=	density of the sample (in g/cm ³).
	P ₁	=	calculated pressure at the beginning of the test (in bar).
	P ₂	=	calculated pressure at the end of the test (in bar).
	t ₁	=	room temperature at the beginning of the test (in °C).
	t ₂	=	room temperature at the end of the test (in °C).

5.3 Stability Testing (Procedure 2B)

5.3.1 Method

NOTE: This method includes the calibration. See also Annex C, Fig. 3.

- Transfer a sample of the prepared explosive to the heating tube, taking care to avoid contamination of the neck of the tube (the introduction of the explosive can be facilitated by the use of a wide-bore short-stemmed glass funnel). A minimum of two tests shall be conducted on each material to be tested.
- Lightly smear the O-rings of the adapter with the lubricant. Assemble the apparatus as in Annex C, Fig. 3. Connect the adapter evacuation port to the vacuum line, slide the inner tube outwards and evacuate the tube to less than 5 mm of mercury (6.7 mbar).
- 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 (R₀).
- Set the plunger of the calibrated syringe to the required calibrating volume (3.0 cm³) and connect to the adapter port. Inject 3 cm³ of air into the heating tube. Record the voltmeter reading R₃.
- Inject a further 5.0 cm³ of air with the calibrated syringe and record the digital voltmeter reading (R₈), the barometric pressure (P₁ mm Hg), and the room temperature (t₁ °C). The difference in readings is the digital response for 2.0 cm³ of air at P₁ and t₁ °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.
- Detach the syringe, connect the port to the vacuum line and evacuate the tube to a maximum of 5 mm of Hg (6.7 mbar). Check that the apparatus is sealed effectively by monitoring the transducer voltage for at least a minute.
- Record the initial digital voltmeter reading of the transducer (E₁).

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- h. Ascertain that the bath temperature is constant at the specified temperature. Carefully place the heating tube of the prepared test assembly in the constant-temperature bath.
- i. Heat the tube for 40 hours (unless otherwise specified). Check the bath temperature every day.
- j. Remove the test assembly from the constant-temperature bath and allow to cool to room temperature.
- k. Record the final digital voltmeter reading of the transducer (E_2) and the room temperature at the end of the test (t_2 °C).
- l. Any changes in colour or physical appearance of the explosive or propellant and any appearance of condensation on the walls of the tube.

5.3.2 Calculation

Calculate the response factor (f) for each assembly of heating tube and transducer, for 1 cm³ of air at standard temperature and pressure:

$$f = \frac{(R_5 - R_3) - (R_3 - R_0)}{2} \times \frac{760}{P_1} \times \frac{(273 + t_1)}{273}$$

Calculate the volume of gas V (at 273 K and 760 mm Hg) liberated during the test from the sample:

$$V = \frac{E_2 (273 + t_1)}{f (273 + t_2)} - \frac{E_1}{f}$$

where:	f	=	response factor for each assembly of heating tube and transducer (in mV/cm ³).
	V	=	Volume of gas liberated from the sample (in cm ³ , at STP)
	R ₀	=	empty tube voltmeter reading (in mV).
	R ₃	=	voltmeter reading after adding 3.0 cm ³ air (in mV).
	R ₅	=	voltmeter reading after adding an additional 5.0 cm ³ air (in mV).
	E ₁	=	voltmeter reading before heating (in mV).
	E ₂	=	voltmeter reading at the end of the test (in mV).
	t ₁	=	room temperature at the time of the calibration (in °C).
	t ₂	=	room temperature at the end of the test (in °C).
	P ₁	=	atmospheric pressure at the time of the calibration (in mm Hg)

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5.4 Dismantling and cleaning

Release the vacuum in the apparatus (by sliding up the inner adapter tube or by slowly twisting the ground joints). Dismantle the apparatus, empty the heating tube and remove residual traces of the 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.

Clean the pressure transducers according to the manufacturer's recommendations.

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ANNEX B to
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NATO STANAG 4556 DATA SHEET	
Report Reference Number: (Unique Reference Number)	Vacuum Stability Test (Instruction Sheet)
Page of Page(s)	
TEST SITE INFORMATION Laboratory: (Name of Laboratory) Date: (Date That Form Was Completed) Test Procedure: (Name of Test Procedure Used) Date Tested: (Date of Test Period) POC: (Point of Contact)	TEST CONDITIONS Test Material (g): (Amount of Test Material) Time (hours): (Test Time) Temperature (°C): (Test Temperature) Procedure: (Manometer or Transducer Method) Sample Preparation: (Grinding; If Applicable, Include Details of Drying or Solvent Removal)
SPECIMEN INFORMATION Explosive: (Type of Explosive) Identification: (Trade Name and/or Identity Code) Manufacturer: (Name of Manufacturer) Lot, Batch or Consignment Number: Date of Manufacture or Receipt: Special Storage Conditions: (If Applicable)	TYPICAL RESULTS Volume of Gas (cm ³): (Mean Volume of Gas from Test Material) Physical Changes: (Change of Colour or Physical Appearance, Condensation on Walls of Tube) Divergence's from Standard Procedure: (If Applicable)
Composition: (Components and Percentages) 	
Data Sent To: (Name and Address of Person Receiving This Information)	Comments:

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ANNEX C to
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(Edition 1)

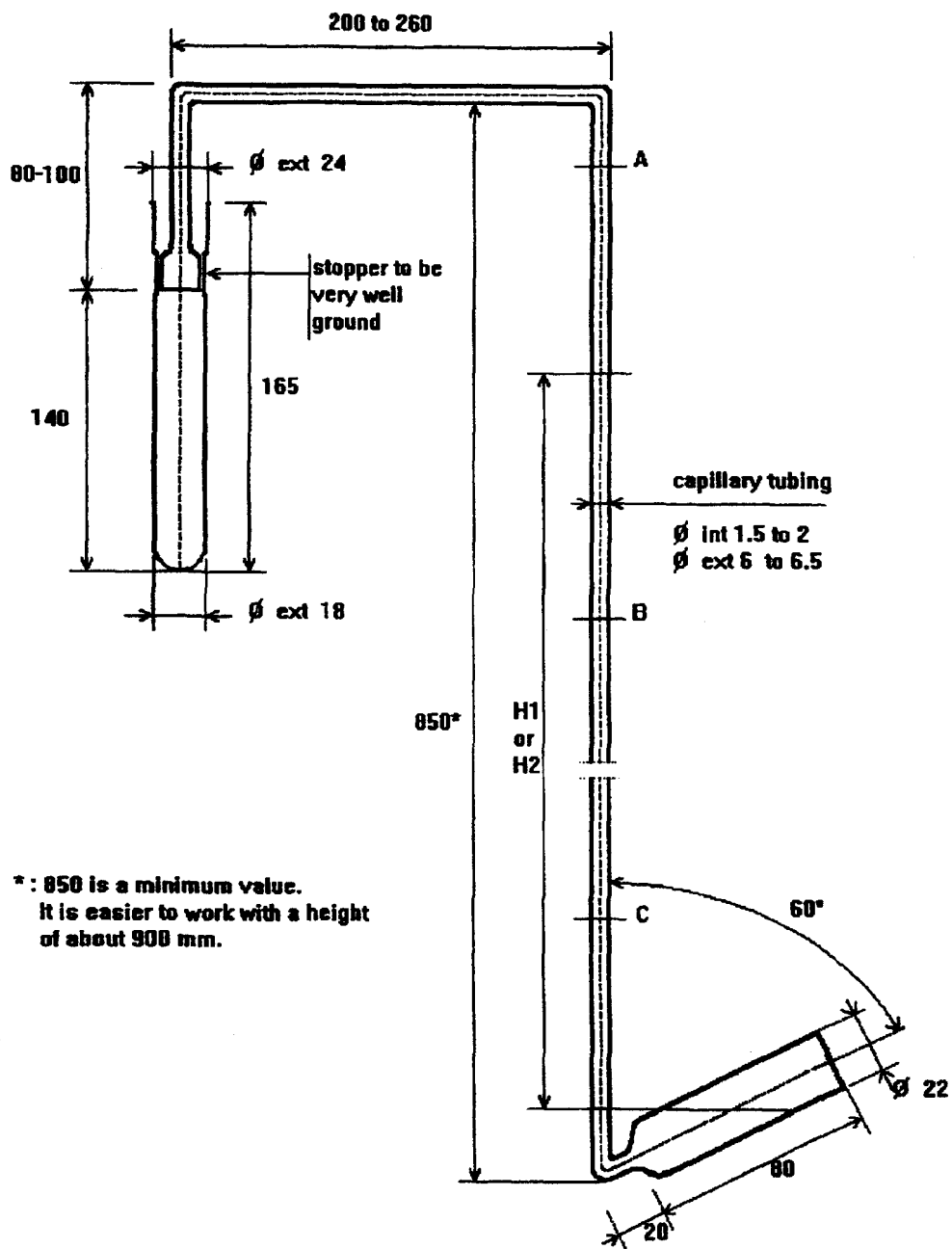


Fig. 1. Vacuum Stability Apparatus (Manometer Method, Procedure 1); all values in mm.

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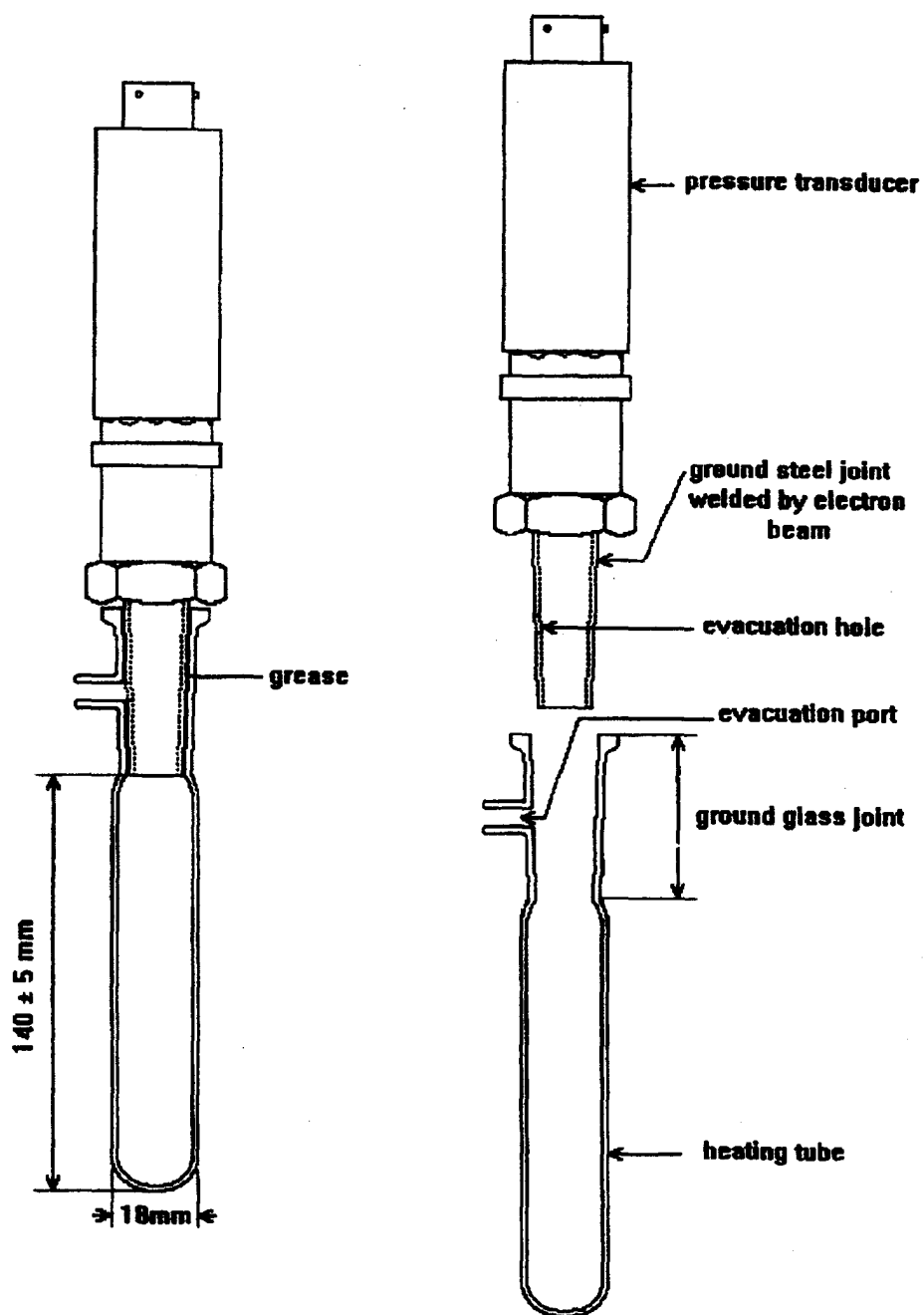


Fig. 2. Vacuum Stability Apparatus (Transducer Method, Procedure 2A).

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ANNEX C to
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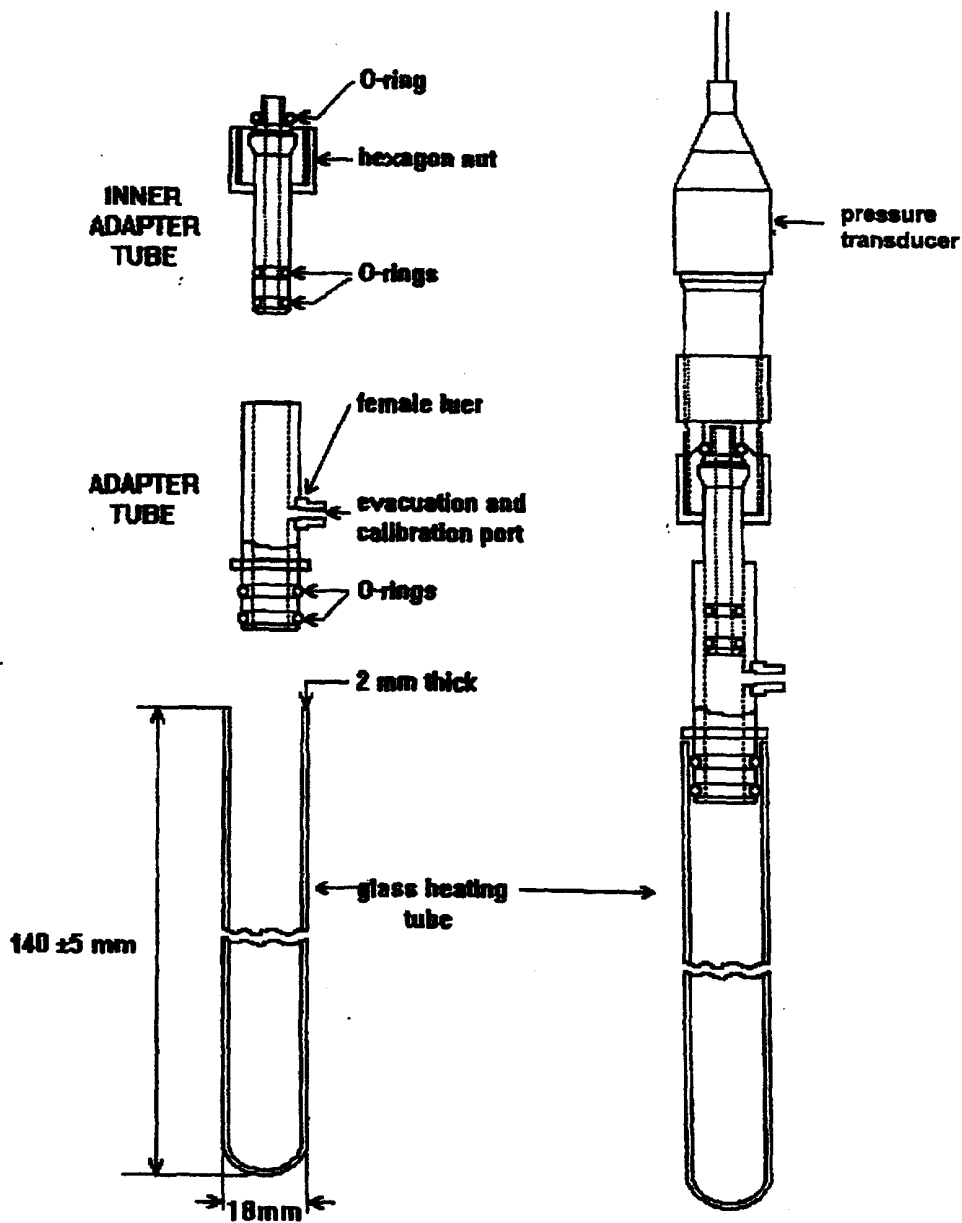


Fig. 3. Vacuum Stability Apparatus (Transducer Method, Procedure 2B).

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