NATO STANDARD

AOP-4147

ENERGETIC MATERIALS, CHEMICAL COMPATIBILITY WITH MUNITION COMPONENTS

Edition A, Version 1

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NORTH ATLANTIC TREATY ORGANIZATION

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NATO LETTER OF PROMULGATION

13 June 2023

1. The enclosed Allied Ordnance Publication AOP-4147, Edition A, Version 1, ENERGETIC MATERIALS, CHEMICAL COMPATIBILITY WITH MUNITION COMPONENTS, which has been approved by the nations in the CNAD Ammunition Safety Group (AC/326), is promulgated herewith. The agreement of nations to use this publication is recorded in STANAG 4147.

2. AOP-4147, Edition A, Version 1, is effective upon receipt.

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4. This publication shall be handled in accordance with C-M(2002)60.

Dimitrios SIGOULAKIS
Lieutenant General, GRC (A)
Director, NATO Standardization Office

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RECORD OF RESERVATIONS

CHAPTER	RECORD OF RESERVATION BY NATIONS

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RECORD OF SPECIFIC RESERVATIONS

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CHAPTER 1 INTRODUCTION

1.1. AIM

The aim of this agreement is to standardize the means of assessment, including test procedures, by which the chemical compatibility of high explosives, pyrotechnics, primary explosives and propellants (referred to throughout this document as energetic materials) with other substances or other energetic materials used in armament manufacture are judged. The test procedures and criteria have been studied and agreed in order to facilitate cross-procurement and to provide a means whereby nations can be assured that munitions and other energetic material stores have been designed and manufactured with proper regard to the need for chemical compatibility between energetic materials, or energetic materials and non-energetic components. Most of the test methods have been described in separate STANAGs (see list in ‘Related Documents’). Therefore, this AOP will not contain test descriptions which can be found in related standards. Only test methods which are not contained in other standards and test procedures directly connected with compatibility testing will be listed.

1.2. AGREEMENT

Participating nations agree that the scope of the agreement as described in Chapter 2, the Requirements and Assessment Criteria in Chapter 3, the Principles in Chapter 4 and the Test Procedures and Acceptances Limits in Annex A shall form the basis upon which chemical compatibility of energetic materials with other munition components shall be judged.

1.3. WARNING

This AOP 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 statutory obligations relating to health and safety at any stage during use.

1.4. IMPLEMENTATION

The AOP is considered implemented by a nation when that nation has issued instructions that the test procedures for judging the chemical compatibility of munition components with energetic materials will be in accordance with the procedures detailed in this agreement.

1.5. RELATED DOCUMENTS

STANAG 4515 (Explosives, Thermal Analysis using Differential Thermal Analysis (DTA), Differential Scanning Calorimetry (DSC), Heat Flow Calorimetry (HFC) and Thermogravimetric Analysis (TGA)).

STANAG 4556 (Explosives, Vacuum Stability Test).

STANAG 4582 (Explosives, Nitrocellulose Based Propellants - Stability Test Procedure and Requirements Using Heat Flow Calorimetry).

STANAG 4620/AOP-48 (Explosives, Nitrocellulose Based Propellants - Stability Test Procedures and Requirements Using Stabilizer Depletion – Implementation of AOP-48).

Stephan Wilker, Gabriele Pantel, Uldis Ticmanis, Pierre Guillaume, “Detailed investigation of sensitivity and reproducibility of heat flow calorimetry“, *Proc. Symp. Chem. Probl. Connected Stabil. Explos*. 11, 383 (**1998**); Stephan Wilker, Pierre Guillaume, “International Round Robin Test on Compatibility”, *Proc. Int. Annu. Conf. ICT* 33, 110 (**2002**).

CHAPTER 2 SCOPE OF THE AGREEMENT

The AOP is limited to matters relating to chemical compatibility of munition components with energetic materials and does not extend to procedures and requirements, which concern the avoidance of incompatibility from other interactions.

Attention is necessarily drawn to other interactions, which, for energetic materials, include:

(i) Migration of soluble or volatile ingredients at contact surfaces or through the vapour phase or other physico-chemical interactions, leading to unacceptable changes in physical and mechanical properties (e.g. migration of nitroglycerin from gun and rocket propellants to polymeric coatings, post-curing of polymeric binders, antioxidant depletion, [increased nitrocellulose] molecular mass reduction).

(ii) Changes in the sensitiveness of the energetic material to initiation by impact, shock, friction, electrostatic discharge or other external stimuli (e.g. lead azide in contact with copper alloys).

(iii) Changes in performance properties due to decrease of active ingredient content or increase of decomposition products (e.g. degradation of magnesium metal forming inactive magnesium hydroxide in pyrotechnics).

These other forms of incompatibility might be of the same importance for safety and suitability for service of energetic materials and munitions as chemical incompatibility. The necessity to carry out tests investigating these aspects for a specific munition system is left to the discretion of the responsible National Authority. The appropriate tests, their methodologies and criteria shall be selected on a case-by-case basis by the National Authority.

The AOP is not concerned with compatibility between ingredients in energetic material compositions and the consequent stability of such compositions.

The requirements of Chapter 3 and the tests in Annex A ensure that the chemical compatibility of energetic materials with other munition components is at the necessary standard for safety during manufacture, storage and use, and for reliability after storage under approved conditions. The procedures outlined in this AOP only apply to issues surrounding compatibility, and do not address overall stability. The issue of stability of nitrocellulose based propellants is addressed in STANAG 4582 and STANAG 4620/AOP-48.

Energetic materials vary widely in their nature and properties and standardization of all compatibility test procedures is unlikely to be achieved. To assist in the harmonization of procedures to the maximum extent, Chapter 4 presents principles to which acceptable compatibility tests should conform and which apply in cases where an agreed procedure from Annex A is not used.

The test Procedures in Annex A provide data which relate to the extent to which energetic materials react or decompose as a consequence of contact or proximity with the materials under test. The properties of energetic materials are diverse and no single test method is capable of overall applicability. A statement of applicability in each procedure provides guidance on the suitability of the procedure for the different classes of energetic materials.

CHAPTER 3 REQUIREMENTS AND ASSESSMENT CRITERIA

Conformity to Test Procedures and Acceptance Limits in Annex A is proof of chemical compatibility within the limits of applicability stated in the Test Procedures.

Since different test methods sometimes yield contradictory results, at least two tests based on different principles shall be performed to examine compatibility. If the result of one of the tests ‘fails’, the energetic material is not compatible with the contact material.
However, in case of a technical impossibility to perform several tests, the use of a single test that is reliable and confirmative enough for the particular energetic material could be sufficient, providing approval by the National Authority.

Many energetic material compositions differ to only a minor extent from other related compositions. Compatibility data applicable to one such energetic material may give an indication of the compatibility of related compositions. It is not possible to define limits within which such judgements may be admissible but their acceptability when agreed between the parties concerned is to be recognized as consistent with the requirements of this AOP.

Test Procedures which are not included in Annex A must conform to the principles stated in Chapter 4. In such cases a special (or ad hoc) agreement on the procedure proposed by a manufacturer will be reached.

Confirmation of compatibility by testing may be waived by special (or ad hoc) agreement when the material complies with the requirements of paragraph 3 of this Chapter and is shown to have had previous use in conjunction with the same or very similar energetic materials, which demonstrates its compatibility and suitability for a proposed application. Information from accelerated ageing trials may also be presented in support of statements of satisfactory compatibility.

To be accepted as compatible a material must conform to the requirements of the relevant specification. It must be subject to quality control procedures which ensure that its chemical composition corresponds to that of the specimen or supply whose behaviour in a compatibility test or in previous usage provides the basis of the acceptance. Alternatively, if the above requirements cannot be met, the testing of each lot or consignment of the material for compatibility is required.

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CHAPTER 4 PRINCIPLES GOVERNING COMPATIBILITY TESTS

4.1. GENERAL

The purpose of a compatibility test in the context of this AOP is to provide evidence that a material may be used in an item of munitions without detriment to the safety or reliability of an energetic material with which it is in contact or proximity (see Chapter 1, paragraphs 1, 2 and 3).

The tests and acceptance criteria must provide for judgments of satisfactory compatibility with adequate margins of confidence but should, as far as is practicable, avoid the rejection of otherwise satisfactory material because of observed but unimportant levels of reactivity.

The methods of monitoring reactivity during heating tests or of examination subsequent to accelerated aging should preferably provide quantitative data. They must be appropriate to the energetic material being tested and its required application.

This AOP describes the following tests and procedures:

- vacuum stability test (VST) (Test 1) (see also STANAG 4556)
- heat flow calorimetry (HFC) (Test 2) (see also STANAG 4582)
- thermogravimetric analysis (TGA) (Test 3) (see also STANAG 4515)
- differential scanning calorimetry (DSC) (Test 4) (see also STANAG 4515)
- chemical analysis after ageing (HPLC) (Test 5) (see also STANAG 4620/AOP-48)

Whenever possible at least two of the above tests based on different principles should be used to determine the compatibility of the test material with energetic materials. Two procedures based on the same physical principle (as in procedures 3A and 3B, both based on TGA) do not constitute independent methods of assessing compatibility.

Valid test procedures require that a combination of energetic material and test material be heated together at a temperature and for a time, which can reasonably be considered to be equivalent to the anticipated storage and service life of the energetic material. The stringency of the procedure increases with temperature and time of heating but the quantitative effects of temperature upon the rates of reactions between particular energetic materials and test materials will not as a rule be known or investigated unless the compatibility problem is of special importance.

The minimum duration versus temperature is given with each test.

The proportions of test material to energetic material and the degree of contact which is achieved by grinding and mixing them, have a marked effect upon the extent of reaction which is observed in compatibility tests. The normal proportion for mixing is 1:1, with the exception of test 5.

If the 1:1 combination fails the test criteria it shall be stated as being incompatible. There are applications where a contact material cannot be replaced. Where an energetic material and contact material is used in a weight ratio far above 1:1 (large excess of energetic material), the test may be repeated at a different ratio resembling more the application in the weapon. If this mixture passes the test, it shall be reported as incompatible however the incompatibility is sufficiently low in this mixing ratio and the material can be used at the discretion of the National Authority. Additional testing may be required to assure that degradation caused by the contact substance is acceptable.

The influence of moisture on incompatibility reactions is frequently important, as is the degree of confinement of the test mixture, the availability of atmospheric oxygen and the presence of solvent during the accelerated ageing heating period. Testing authorities should define the conditions controlling the above variables during their tests and be prepared to present an explanation for them.

At the end of or during the heating period the reactivity of the test material with the energetic material is measured with the object of determining the extent to which the energetic material has reacted or decomposed. The procedure must include parallel testing of bare energetic material and separate testing of the bare test material in order to distinguish between the incompatibility of contact materials with the energetic material and the intrinsic instability of energetic material and/or test material under the test condition.

The choice of acceptance criteria for procedures which provide quantitative data may be made in two alternative ways:

a. relative criteria: by reference to the extent of the decomposition suffered by the energetic material in the test in the absence of incompatibility (A proportionate increase in decomposition will be allowed depending upon the severity of the test and the stability properties of the energetic material. In general, the criteria for compatibility with less stable energetic materials will be more stringent).

b. absolute criteria: arbitrarily being set at a level which will restrict the observed reactivity to a safe acceptable level.

Alternative (b) is more relevant than (a) not only for the case of rather stable high explosives but even more for propellants with a relatively low level of stability. The reason is that only absolute criteria are proportional to the reaction degree or the rate of the reaction of the energetic material with the contact material. They can therefore be used for the extrapolation to lower temperatures.

Some materials to be tested for compatibility may contain solvents or impurities which could vary from one lot to another and affect the results obtained. Consequently it is important to uniquely identify the material by lot number and commercial reference.

Sample preparation has a big influence on the results obtained and should be noted on the data sheet.

4.2. ENERGETIC MATERIALS - PROPERTIES AND TESTS

In this section the compatibility properties of the various groups of energetic materials are considered and the principles of suitable tests are indicated. The grouping of the energetic materials is based upon their chemical properties and in particular upon their more frequent modes of decomposition or incompatibility.

4.2.1 High Explosives including plastic bonded explosives (PBX)

Energetic materials containing substances like TNT, RDX, HMX, PETN, HNS, NTO, CL20, or Ammonium Nitrate as principal ingredients are included in this group.

Energetic materials containing nitroglycerin or other liquid nitrate esters, as one of the principal ingredient, are not in this category for compatibility consideration. They may fall more appropriately into Section 4.2.2.

Incompatibility with high explosives can be determined using tests measuring gas evolution, TGA, and also measuring heat change. Tests 1, 2, 3 and 4, in Annex A are applicable in determining incompatibility with high explosives. Test 3 may not be applicable for several PBX compositions, because of the high volatility of some plasticizers.

To be suitable, materials in contact with this type of energetic materials are required to be free from strong acidity or alkalinity. Prescreening tests should highlight strongly acidic or alkaline materials before testing begins.

4.2.2 Propellants containing Nitrocellulose as a Principal Ingredient

These comprise single, double and triple base propellants usually manufactured by extrusion or casting. An increased rate of decomposition caused by incompatibilities with these propellants can be determined using tests measuring gas evolution, heat generation, stabilizer depletion, and mass loss (this only for single base propellants). These tests found in Annex A are applicable in determining incompatibility with these propellants. Test 1 is considered applicable by several nations but may not be accepted by all nations unless supported by data from other tests. Test 2 is regarded as being most reliable for propellants. Test 5 may show contradictory results compared with Test 1 and Test 2 depending on the nature of contact material.

Dynamic tests may in some cases not be very useful for stability/compatibility testing because the early nitrocellulose/nitroglycerin decomposition at around 160°C may hide incompatibility reactions.

Although properly formulated propellants of this class, prepared from stable ingredients, can be expected to remain safe in storage at normal temperatures over many years, they contain ingredients of comparatively low stability and standards for compatibility acceptance are required to be comparatively severe.

Test methods should provide quantitative data for the rates of decomposition of the propellant in the presence and absence of the test material and so that the comparative effect of any reactivity on the stability of the propellant and its safe life can be judged.

To be suitable, materials in contact with this type of propellant are required to be free from strong acidity or alkalinity.

4.2.3 Pyrotechnics

Numerous energetic materials with widely differing compositions fall into this category and exceptions to the following generalized comments may arise. Most pyrotechnics are intimate mixtures of substances which are themselves individually stable but capable of reacting together to produce the desired effect.

Many pyrotechnics are formulated to decompose or react without generating significant volumes of gas. Gas evolution tests are not generally applicable and methods based on thermal, chemical or physico-chemical analysis after periods of heating (in accordance with the principles described in Section 1 of this Chapter) are preferred. Test 2, 3 and 4 in Annex A are considered suitable for determining incompatibility with pyrotechnics.

Problems of instability or incompatibility with such mixtures are frequently increased significantly by the presence of moisture. In practice, most munitions are sealed to some degree, so that there is either no reaction or only a very slow interaction with humidity from the outside atmosphere. In cases of ineffective sealing or where there is a considerable built-in reservoir of moisture (e.g. gas phase contact with moisture containing propellant in a sealed cartridge), the procedures should introduce an appropriate level of humidity so that the energetic material alone is affected to a small but measurable extent. Most easily, this can be realized in Test 2 by additionally introducing a glass tube containing a saturated salt solution, a water/sulfuric acid mixture (CAUTION!) or a water/glycerol mixture into the sealed HFC container. Thus constant relative humidity is maintained in the internal gas phase throughout the whole experiment.

4.2.4 Primary Explosives

Several of the above statements pertaining to pyrotechnics are applicable to primary explosives. The numerous compositions may be either single substances or mixtures and many of the individual compounds which are used are thermally stable to fairly high temperatures. Some of them (azides and styphnates) produce only small volumes of gas when heated at desirable temperatures for compatibility tests, unless undesirably large quantities are used. In these cases such tests, based on gas evolution measurement, are of doubtful value and methods based on thermal, chemical or physico-chemical analysis after periods of heating (Section 1) are preferred. Tests 2, 3, and 4 in Annex A are considered suitable for determining incompatibility with primary explosives. For reasons of safety, Test 2 should only be performed with very small sample amounts (not exceeding 100 mg).

Characteristically the use of primary explosives in munitions requires only small charges to be present. These will exist in an assembly with much greater quantities of surrounding materials. If the compatibility of the energetic material with these materials is in question, the tests must incorporate this disparity in amounts.

The stability and reactivity of several important primary explosives (e.g. lead azide) is much influenced by the presence of moisture. If it is relevant for the application, a modified HFC setup as mentioned in 4.2 in Test 2 should be used.

4.2.5 Composite Propellants

These propellants are generally composed of energetic materials bound together using a binder. These propellants tend to be thermally stable, but can suffer deterioration by exposure to moisture. Another cause of deterioration results, for example, from changes in the cross linking of the inert binder, leading to unacceptable changes in the mechanical properties of the propellant. Changes of this nature are not covered by this AOP but tests used for monitoring such changes in mechanical properties can be found in STANAGs 4443, 4506, 4507, 4525, 4540, and 4581. Tests 1, 2, 3 and 4 of this AOP are considered suitable for determining any incompatibility with composite propellants, resulting in deterioration of thermal or chemical stability of the propellant.

4.2.6. Liquid High Explosives and Energetic Liquid Monopropellants

Some methods described in this AOP may also be used for liquid high explosives and energetic liquid monopropellants. However, modifications will be necessary to account for the properties of liquid samples, in particular high vapour pressures and incompatibilities with container materials. HFC, preferably in hermetically sealed glass containers, should be applicable in most cases. For liquid high explosives and energetic liquid monopropellants containing stabilizers, Test 5 may be applied. In addition, special safety precautions have to be taken.

4.3. TESTS FOR OTHER FORMS OF INCOMPATIBILITY

Selection of appropriate tests for investigation of other forms of incompatibility as specified in Chapter 1 paragraph 1. Their methodologies and criteria strongly depend on compositions and properties of an energetic material and a contact material and shall thus be left to the discretion of the National Authority. Generally, possible interactions between an energetic material and a contact material can be induced by an artificial ageing at increased temperatures or a combination of temperature/humidity. Revealing the changes in properties caused by incompatibility reactions above changes in the samples aged separately under the same conditions can be done using test methods already specified in other STANAGs or by case-specific testing procedures.

Examples of appropriate tests for different types of energetic materials are specified in Table 4-1.

As none of the test methods listed in Table 4-1 was specifically designed for compatibility testing, appropriate adjustments in the testing procedures might be necessary. These tests also do not contain specific criteria of compatibility or incompatibility and such conclusion shall be made by a qualified assessment of the results by the National Authority.

Table 4-1

Test/property	Types of energetic materials	Test methods
Artificial ageing	All	STANAG 4170, AOP-7, STANAG 4581, AOP-48
Plasticizer migration	Plastic bonded explosives, gun propellants, rocket propellants	STANAG 4581
Antioxidant depletion	Plastic bonded explosives, composite rocket propellants	STANAG 4581
Stabilizer depletion	Nitrocellulose based energetic materials	STANAG 4620, AOP-48
Decrease in active ingredient content or increase of decomposition products	Primary explosives, pyrotechnics	STANAG 4300, FTIR, Titrimetric methods
Tensile strength	plastic bonded explosives, rocket propellants	STANAG 4506
Compressive strength	plastic bonded explosives, rocket propellants	STANAG 4443
Glass transition temperature	plastic bonded explosives, rocket propellants	STANAG 4540
Impact sensitivity	All	STANAG 4489
Friction sensitivity	All	STANAG 4487
Electrostatic discharge sensitivity	All	STANAG 4490

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ANNEX A TEST PROCEDURES AND ACCEPTANCE LIMITS

TEST 1 - VACUUM STABILITY TEST

1. SUMMARY DESCRIPTION

The volume of gas evolved, when a mixture of equal parts of an energetic material and the material under test is heated at a constant temperature of 100°C for 40 hours in an initial vacuum, is compared with the volumes evolved from the energetic material and the test material when heated separately under otherwise identical conditions. Compatibility is judged by means of the volume of additional gas produced because of the contact between the two components of the mixture.

Most tests by this method are conducted at 100°C for 40 hours and this time and temperature can therefore be regarded as adequate. Tests at 80°C for 250 hours are considered adequate for double base propellants and energetic materials which decompose too rapidly when heated at 100°C.

Temperatures much higher than 100°C are undesirable because the test conditions become increasingly unrealistic. Conversely, lower test temperatures are desirable and are essential for some types of test materials and energetic materials. Longer periods of test then become necessary.

For lower test temperatures test times must be longer to reach a comparable thermal load. According to van´t Hoff´s rule the test times are multiplied by 2.5 for every temperature decrease of 10°C (e.g. 40h@100°C → 26d@70°C → 65d@60°C).

For the vacuum stability test, any apparatus and method described in STANAG 4556 (manometric method, two transducer methods) can be used.

2. APPLICABILITY

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

3. INTERPRETATION OF RESULTS

Materials which fail to satisfy the requirements of this test will be judged as unsuitable for use in applications where permanent direct contact or close proximity with the test energetic material or propellant is required or where adequate precautions to guard against such contact are not taken.

4. PREPARATION OF TEST MATERIALS

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the energetic material. Where the intended use requires the test material to be applied in an intermediate (uncured or partly cured) condition to the energetic material, the compatibility test shall be made on mixtures so prepared and then treated in the manner prescribed to bring the test material to its final (cured) condition.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the energetic material during the test.

Subdivision of solid materials in block form must reduce them to particles with a maximum size of 2 mm and the preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the energetic material which is at least as effective. Discard material that has been produced by the process of subdivision and which passes a 0.2 mm sieve.

Test materials should not be artificially dried before testing but the presence of excessive quantities of volatile ingredients may make the test impossible to perform. To remove excessive quantities of water, preliminary drying over silica gel for 24 hours at ambient temperature is permitted. To remove volatile solvents other methods may be used. These treatments must be recorded in the test report.

5. APPARATUS, CALIBRATION, PREPARATION OF ENERGETIC MATERIALS, PROCEDURES

The requirements on apparatus, calibration procedures, preparation of energetic materials, test procedures, dismantling and cleaning procedures and calculation of volume of gases evolved during the test are described in the appropriate sections of STANAG 4556 of the appropriate method (manometric, transducer). The following sections only describe changes in these procedures applied for compatibility testing.

6. PRELIMINARY TEST

When testing materials of unknown chemical reactivity it is desirable to confirm by means of simple tests, in which small quantities are mixed with energetic materials and warmed, that reactions between the substances being tested for compatibility are not liable to cause explosion or fire during the test. Test 4 of this AOP is recommended for this purpose because only very small amounts of materials are used.

7. COMPATIBILITY TEST

Generally the compatibility test is performed with

2.5 ± 0.01 g of the energetic material
2.5 ± 0.01 g of the contact material
5.0 ± 0.01 g of the mixture, containing of 2.5 g of energetic material and 2.5 g of the contact material.

All three experiments have to be conducted under identical conditions.

If smaller amounts of material or mixture ratios deviating from 1:1 are used, note this in the Data Sheet.

For the description of the procedure see STANAG 4556.

8. CALCULATION

8.1 Calculate with the appropriate equation of STANAG 4556, according to the applied method, the volumes of gas at STP evolved by the mixture, and by the energetic material and the test material when heated separately. Reduce the values to cm³/g.

8.2 Calculate:

$$V_R = 5 \cdot \left[V_M - V_{M,calc}\right] = 5 \cdot \left[V_M - \left(m_E V_E + m_C V_C\right) / \left(m_E + m_C\right)\right] \qquad \text{(eq. 1.1)}$$

with

V_R = normalized volume of gas from interreaction of 5 g mixture [cm³]

V_M = normalized volume of gas evolved by the mixture [cm³/g]

$V_{M,calc}$ = calculated normalized volume of gas evolved by the mixture assuming no reaction between the two compounds [cm³/g]

V_E = normalized volume of gas evolved by the test energetic material [cm³/g]

V_C = normalized volume of gas evolved by the contact material [cm³/g]

m_E = mass of test energetic material in the mixture [g]

m_C = mass of contact material in the mixture [g]

8.3 Report the results of the test on the Data Sheet.

9. ACCEPTANCE LIMIT

9.1. For materials to which paragraph 2 on page A-1 does not apply, the volume of gas as calculated from eq. 1.1 for 5 g of the mixture must not exceed 5 cm^3 at STP.

9.2. Materials supplied to specifications which include requirements for testing for compatibility with high explosives or propellants by the Vacuum Stability Test, and for which the permitted volume of evolved gas V_R as defined in paragraph 1 on page A-1, is less than 5 cm^3 at STP must be tested and sentenced in accordance with the requirements of their specifications.

9.3. When V_R approaches 5 cm^3, a retest is recommended to confirm the result. Confirmation, using a different test method, should also be sought. If this second test fails, then the test material is not compatible with the energetic material.

DATA SHEET for Test … Procedure …

(Side A identical for all tests)

NATO AOP 4147 DATA SHEET (Side A)

Report Reference Number:
(Unique Reference Number)

Page __ of __ Page(s)

TEST SITE INFORMATION

Laboratory: (Name of Laboratory)	Date Tested: (Date of test period)
Date: (Date that form was completed)	POC: (Point of contact)
Test Procedure: (Name of test procedure used)	

EM INFORMATION	TEST MATERIAL INFORMATION
Identification of Energetic Material (Trade name and/or Identity code)	Identification of Test Material: (Trade name and/or Identity code)
Manufacturer: (Name of Manufacturer)	Manufacturer: (Name of Manufacturer)
Specification: (State if specification controls chemical composition of product)	Test Material, Specification: (State if specification controls chemical composition of product)
Lot, Batch or Consignment Number:	Lot, Batch or Consignment Number:
Date of Manufacture or Receipt:	Date of Manufacture or Receipt:
Special Storage Conditions: (If applicable)	Special Storage Conditions: (If applicable)
Chemical Composition:	Chemical Composition:

TEST 1 - DATA SHEET

NATO AOP 4147 DATA SHEET (Side B) Report Reference Number: (Unique Reference Number) Page __ of __ Page(s)	
EM PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (**including any divergence from standard tests procedure**) Test temperature (°C): Test time (h): Mass of samples (g) EM: Test material: Admixture: EM in admixture (m_E): Test material in admixture (m_C):	**TEST RESULTS** V_M (cm³/g): V_E (cm³/g): V_C (cm³/g):
CALCULATIONS V_R (cm³/5 g admixture): **(see eq. 1.1)**	**ASSESSMENT** COMPATIBLE ☐ YES ☐ NO
DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

TEST 2 - HEAT FLOW CALORIMETRY TEST

1. GENERAL

Heat flow calorimetry (also known as microcalorimetry) is a sensitive heat generation rate test at constant temperature.

The test method described is based on experience with HFC stability testing of nitrocellulose based propellants. Sample sizes, test conditions, and test criteria are adapted for these types of propellants. For other types of energetic materials different procedures and criteria may be necessary.

Due to the quantity of energetic material used in the HFC method the representativeness of the sample with respect to its chemical composition is better than, for instance, a screening test like DSC.

For safety reasons, given the potential quantity of energetic materials that may be used in the HFC method (gram quantities) the preliminary screening tests listed in Section 7 of this Test should be completed before any full scale HFC tests commence.

In general 1:1 (w/w) mixtures of energetic material and contact material are tested.

2. SUMMARY DESCRIPTION

The method determines the heat generation rate as a function of time produced by decomposing the samples at constant temperature. The heat release rate during isothermal storage simulates thermal ageing for a certain period of time at a storage temperature. For propellants, storage for 6 days at 85°C is a convenient test condition. Other test times and temperatures suitable for propellants are presented in STANAG 4582, Annex C. These test conditions may also be used for other types of energetic materials if approved by the involved parties. Test time and test temperature shall be specified in the data sheet.

The heat generation rate, as a function of time, of the mixture of energetic material and test material, over a certain period of time is compared to a reference curve, which is the sum of the heat generation rates of the energetic material and test material when heated separately. Acceptance criteria for propellants are given in Section 9.

3. APPLICABILITY

The heat flow calorimetry test is applicable to energetic materials, in particular for all types of nitrate ester based propellants. Due to insufficient experience, pass/fail criteria are not provided for energetic materials other than nitrate ester based propellants.

The test should be performed under atmospheric conditions. In special cases helium or nitrogen atmospheres may be used.

As the results will depend on the test conditions, these conditions must resemble as closely as possible the practical situation and must be recorded in the data sheet.

4. APPARATUS

4.1 Calorimeter with a measuring range corresponding to at least 10 to 3000 µW/g. The reproducibility of heat release determination must be less than 2%. If necessary to reach this level the drift of the baseline must be estimated and used for correction.

4.2 Sample vessels with a minimum volume of 2 cm³ that can be hermetically sealed. It must be proved that the vessel material does not react with energetic materials, the contact material and their decomposition products.

Vials should be filled with as little excess space as possible. In case vials can´t be filled completely with test materials they should be topped up with appropriate inert materials, as otherwise the air in the excess space may influence the results.

4.3 Appropriate data collecting and calculating system including integration.

5. PREPARATION OF ENERGETIC MATERIALS

With necessary precautions, grind, rasp or subdivide the energetic material to pass through a sieve with a 2 mm opening. Discard material which passes a 0.2 mm sieve. Samples that melt at ‘standard’ test temperatures (e.g. TNT) should be measured at lower temperatures; of course then longer measuring times are required. Energetic materials which do not require grinding or sub-division to pass a 2 mm sieve shall not be sieved before use, to remove fine particles.

In order to keep the energetic material in a state as close to application as possible, artificial drying shall be avoided or be done at very gentle conditions. Otherwise volatile contents, that might influence the compatibility in the application, could be lost for the test.

6. PREPARATION OF TEST MATERIALS

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the energetic material. Where the intended use requires the test material to be applied in an intermediate (uncured or partly cured) condition to the energetic material, shifting of curing peaks might appear during the test giving a false assessment of compatibility.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the energetic material during the test.

Subdivision of solid materials in block form must reduce them to particles with a maximum size of 2 mm and the preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the energetic material which is at least as effective. Discard material which has been produced by the process of subdivision and which passes a 0.2 mm sieve.

7. PRELIMINARY TEST

When testing materials of unknown chemical reactivity, it is vitally important to test small quantities of the energetic material and test material to confirm that when mixed and heated reactions between the test substances do not cause an explosion or fire during the test period.

A preliminary TGA, DTA or DSC (see Test 4, procedure A-2) measurement of the mixture should be carried out before larger quantities of energetic materials are tested.

Before vials are placed in an HFC instrument, it is recommended to test them in a safe and sturdy environment (like a suitable blast screened cabinet containing a thermal block bath) under the same or somewhat harsher conditions intended for the HFC experiment.

8. COMPATIBILITY TEST

The apparatus, including the reference sample vessel, shall be brought to the test temperature prior to inserting the sample vessel into the apparatus. Before a measurement can be performed, the blank signal (baseline) and the sensitivity of the heat generation rate meter shall be determined (calibration). This value can vary depending on the temperature used.

Mix the test energetic material with the contact material and transfer the mixture to the sample vessel, then insert it into the calorimeter. In most cases it is recommended to measure under munition-like conditions (normally sample vessels completely filled and tightly closed).

After insertion of the sample vessel, the measurement should be started immediately. Evaluation should start after temperature equilibration of the mixture has taken place. The time until this is reached is the starting time of the evaluation of the energetic material and the contact material.

Measure the heat generation rate of the mixture for the period of 6 days at 85°C or at times equivalent to 10 years at 25°C (see table 2-1; which is valid for nitrocellulose based propellants). Repeat the experiment under identical conditions, on the energetic material and the test material separately. Discard the part of the experiment before temperature equilibration has occurred.

Determine the time integrated heat generation per unit of mass for the Mixture (M), the energetic material (E) and the contact material (C).

All tests shall be conducted in duplicate.

Table 2-1. test times t_m for different test temperatures T_m (taken from STANAG 4582). These values are recommended for nitrocellulose based propellants

T_m [°C]	t_m [days]
60	123
61	108
62	95.0
63	83.6
64	73.6
65	64.9
66	57.2
67	50.5
68	44.6
69	39.4
70	34.8
71	30.8
72	27.3
73	24.2
74	21.5
75	19.0
76	16.9
77	15.0
78	13.4
79	11.9
80	10.6
81	9.43
82	8.41
83	7.50
84	6.70
85	5.98
86	5.35
87	4.78
88	4.28
89	3.83
90	3.43

9. CRITERIA

9.1 Nitrate ester based propellants

Calculate the absolute increase in the heat generation of the mixture of energetic material and test material, compared to the heat generation of the components separately:

$$Q_R = Q_M - Q_{M,calc} = Q_M - (m_E \cdot Q_E + m_C \cdot Q_C)/(m_E + m_C) \qquad \text{(eq. 2.1)}$$

with
Q_C = energy release of contact material until t [J/g]
Q_E = energy release of energetic material until t [J/g]

Q_M = energy release of mixture until t [J/g]
$Q_{M,calc}$ = calculated energy release of mixture until t [J/g]
Q_R = energy release of inter-reaction until t [J/g]
m_C = mass of contact material in the mixture [g]
m_E = mass of energetic material in the mixture [g]

Criterion

If Q_R exceeds 30 J/g (approximately 1% of a propellant´s heat of decomposition) within t then the test material must be considered incompatible with the propellant. If it is below 30 J/g it can be considered as being compatible.

NOTE 1: Whereas the final compatibility assessment is based on the Q_R-value ("absolute incompatibility"), there are several other indicators which give further information about the compatibility of the mixture under investigation. These indicators are namely (i) D-value = $Q_M/Q_{M,calc}$ ("relative incompatibility" the relation of released energy of the mixture and the theoretical value without reaction between propellant and contact material, as used in earlier versions of this AOP as the only criterion), (ii) M-value ("stability of the mixture"), and (iii) course of the heat flow curve for the incompatibility reaction. D-values of ≤ 2, M-values not exceeding the limiting heat flow for the chosen temperature as described in STANAG 4582, and constant or decreasing courses of heat flow curve for the incompatibility reaction towards the end of the sequence indicate compatibility. Similarly, D-values of > 3, M-values which exceed the limiting heat flow for the chosen temperature as described in STANAG 4582, and courses of heat flow curve for the incompatibility reaction which increase strongly towards the end of the sequence indicate incompatibility. If two or even all of these three indicators differ from the assessment basing on the Q_R-value, further investigations are recommended (e.g. re-testing the compatibility with an alternative method).

9.2 Other energetic materials

Insufficient data is available to set any pass/ fail criteria. Therefore, test criteria should be either established by the National Authority or agreed between the involved parties.

The following procedure may be used as a guideline: The limit of Q_R should depend on the heat released by the first exotherm of the decomposition of the energetic material as e.g. determined by DSC in a hermetically sealed crucible (Test 4, Procedure A-2). So for high explosives including PBX, which have about the same decomposition heat as propellants, the same criterion ($Q_R \leq 30$ J/g) can be applied. This value would however correspond to a complete decomposition of the tetracene content of a friction sensitive mixture (e.g. SINOXID).

Therefore at least heterogeneous energetic materials like pyrotechnics and primary explosives mixtures need individual criteria e.g. Q_R = 2 % of the first DSC decomposition peak area.

If this area is small and the heat generation of the test material is high at the test temperature, it may be difficult to recognize incompatibility. In such cases an alternative would be to take 1% of the total heat of explosion as criterion.

TEST 2 - DATA SHEET

(side A see on page A-5)

<table>
<tr><td colspan="2">NATO AOP 4147 DATA SHEET (Side B)
Report Reference Number:
(Unique Reference Number)
Page __ of __ Page(s)</td></tr>
<tr><td>EM PREPARATION</td><td>TEST MATERIAL PREPARATION</td></tr>
<tr><td>TEST CONDITIONS
(including divergences from standard tests)
Test temperature (°C):
Test time (d):
Mass of samples (g):
EM:
Test material:
Admixture:
EM in admixture (m_E):
Test material in admixture (m_C):</td><td>TEST RESULTS
Q_M (J/g):
Q_E (J/g):
Q_C (J/g):</td></tr>
<tr><td>CALCULATIONS
Q_R (J/g):
(see eq. 2.1)</td><td>ASSESSMENT
COMPATIBLE
☐ YES
☐ NO</td></tr>
<tr><td>DATA SENT TO: (Name and address of person receiving this information)</td><td>COMMENTS</td></tr>
</table>

TEST 3 - THERMOGRAVIMETRY (TGA)

TEST 3, PROCEDURE A - DYNAMIC TGA

GENERAL

Dynamic TGA is carried out at 2°C/min in a nitrogen or air atmosphere on the energetic material and test material and on a mixture of the energetic material and test material in a 1:1 ratio by weight. The sum of the weight losses of the energetic material and test material is compared to that of a mixture of the energetic material and test material at a selected temperature.

Two alternative ways of evaluation are described, differing mainly in the temperature selected. The first one refers to the lowest derivative TGA (DTGA) peak temperature (procedure A-1), the second one to the temperature corresponding to 10 % mass loss of the first TGA step (procedure A-2).

NOTE 2: It is recommended to perform additionally a DSC measurement of the mixture. If an endothermic reaction is detected before the first TGA step and if it is caused by melting, then an incompatibility detected by TGA may not necessarily be relevant for lower temperatures. In this case an isothermal TGA (Test 3B) at about 10°C below the melting point or another suitable test should be performed.

Test 3 A-1: Dynamic TGA, Mass Loss at DTGA Peak Temperature

1. SUMMARY

The temperature selected is the lowest derivative TGA peak of the admixture. If the observed weight loss of the mixture is greater than the sum of the individual energetic materials and test materials then this is an indication of incompatibility. The greater the difference in percent weight loss the greater the degree of incompatibility at that temperature. The limits for the various degrees of incompatibility for TGA data in percent weight loss were chosen to correspond to the peak temperature shifts in DSC data.

2. APPLICABILITY

The test is applicable to energetic materials likely to come into contact with plasticizers, fuels, additives, and polymeric materials or other energetic materials.

If the energetic material/contact material combination is known to be used in a closed system (e.g. a detonator) and at the same time contains volatile compounds the test may give irrelevant results. A closed test set-up (like in test 2) should be preferred in these cases.

The material used for the test shall be, as far as is practical, in its proposed condition for contact with the energetic material. When the intended use requires the test material to be applied in an intermediate (uncured or partially cured) condition to the energetic material, the compatibility test shall be made on mixtures so prepared and then treated in the manner prescribed to bring the test material to its final (cured) condition.

Most solid materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the energetic material during the test. Subdivision of solid materials in block form must reduce them to the smallest size particles as practical. The preparation of materials as shavings or by cutting up of thin sheets or other means must allow a degree of mixing and contact with the energetic material that is effective.

Energetic materials can be subdivided for testing by grinding or rasping the energetic material using the proper safety precautions. In some instances, applying pressure to the energetic material once placed in the sample pan will cause it to break apart or spread so that sufficient surface area for contact with the test material has been achieved.

NOTE 3: Actual samples used in formulations are tested with the same particle size that would be used in the formulation. Reducing particle size may influence test results in this particular case. It is essential to ensure that the energetic material is homogeneous so that the sample taken is representative of the composition.

Samples are prepared by placing a ratio as close as possible to 1:1 (w/w) of the test energetic material and material in the sample pan. The sample weight of each component tested is at least about 1 mg. If runaway reactions can be excluded by a previous DSC measurement, larger amounts (up to 5 mg) are recommended to achieve more representative samples.

When run individually, the energetic material and the contact material tested should be approximately the same weight as used in the admixture. Within the admixture, samples should be in physical contact with each other and not placed side-by-side. Specific placements for different matrices are as follows:

3. SAMPLE PREPARATION

3.1 Non-volatile liquid and powdered sample: Solid is added first to the sample pan and the liquid placed on top.

3.2 Film/solid piece and powdered sample: Place the film in the pan first and add the powder to the top.

3.3 Two powdered/film samples: Place the sample which decomposes first on the bottom of the pan.

4. EXPERIMENTAL CONDITIONS

The initial temperature selected is normally room temperature. However, if the thermal characteristics of the material are known, the test may be started at a higher temperature, but this should be at least 50˚C lower than the onset of first mass loss. TGA sample pans must be inert to the test materials, preferably of platinum or aluminum construction. An aluminum liner made out of aluminum foil may be placed on the inside of the platinum sample pan to prolong the life of the platinum pan. The aluminum liner cannot be used above 600°C. Aluminum pans may also be used but, as with the liner, may cause problems with reactive materials. Practical experience shows that these problems will rarely occur if aluminium of high purity (> 99.99 %) is used. Open pans may be used but a loose closing by a lid is recommended in order not to suppress autocatalytic effects. Test conditions including pan type should be specified on the data sheet. For propellants a lower heating rate than 2°C/min may be necessary to avoid an explosion.

5. PROCEDURE

5.1. Each energetic material and test material is run individually to determine the number of weight loss steps in the selected temperature range. Plot the weight loss and the calculated derivative versus temperature.

NOTE 4: The term energetic material refers to the finished energetic material formulation and may contain several different energetic materials as ingredients. The finished formulation is treated as one test material unless there is a requirement to need to know which specific energetic material or ingredient is causing the incompatibility.

5.2 If the test material is another energetic material, the two energetic materials are run individually and in contact with each other to determine the number of weight loss steps in the selected range. A derivative maximum peak temperature is selected for each energetic material.

5.3 Admixtures are then prepared in a 1:1 ratio for energetic material to non-energetic test materials. Weight losses are measured at the selected temperature corresponding to a weight loss step. The admixtures are run in duplicate.

When the admixture is a combination of two energetic materials, weight losses are measured at the two selected temperatures from step 5.2.

6. ANALYSIS

For the determination of a weight loss step see procedure A-2, para 6.1. Record the data using the data sheet given.

6.1 For dynamic TGA, the difference between the observed weight loss and the total calculated weight loss of the energetic material and test material in the admixture is noted. The temperature selected is that of the derivative TGA peak.

6.2. Calculation of mass loss due to inter-reaction

$$W_R = W_M - W_{M,calc} = W_M - (m_E \cdot W_E + m_C \cdot W_C)/(m_E + m_C) \qquad \text{(eq. 3.1)}$$

with

W_R = mass loss due to inter-reaction [%]

W_M = mass loss of the mixture [%]

$W_{M,calc}$ = mass loss of the mixture, calculated without inter-reaction [%]

W_E = mass loss of the energetic material in the single measurement [%]

W_C = mass loss of the contact material in the single measurement [%]

m_E = mass of the energetic material in the mixture [mg]

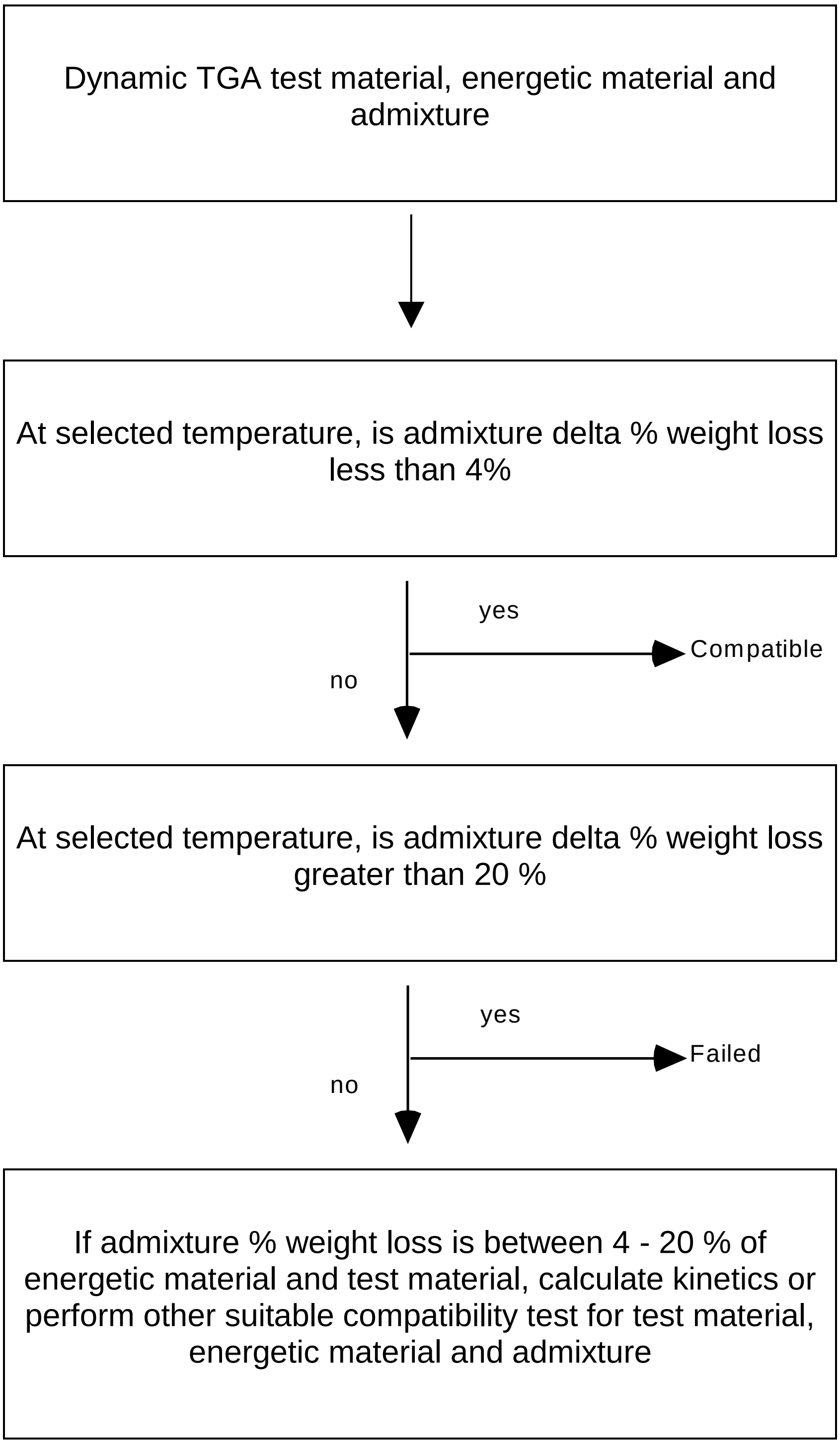
m_C = mass of the contact material in the mixture [mg]

7. CRITERIA

7.1 A change of less than 4% by weight difference (% observed weight loss - % calculated weight loss), calculated by eq. 3.1 indicates the materials are compatible. A change between 4% and 20% indicates a degree of incompatibility may exist and further testing is required. A change greater than 20% by weight difference indicates incompatibility of the material and energetic material under test.

7.2 A flowchart in Figure 3A-1 outlines the decision making process used to determine compatibility. When a material falls within the grey area of 4-20 %, other suitable test methods should be conducted. Test 3 Procedure B-1 outlines a more stringent application of TGA in the isothermal mode, and can be used as a confirmation test for the dynamic TGA procedure. Kinetics studies can also be used as a confirmation test method. The kinetic test procedure is Test 3 Procedure C. It should be noted that kinetic studies are extremely time consuming and should be only performed when other test methods are not suitable.

TEST 3, Procedure A-1



Dynamic TGA test material, energetic material and admixture

At selected temperature, is admixture delta % weight loss less than 4%

At selected temperature, is admixture delta % weight loss greater than 20 %

If admixture % weight loss is between 4 - 20 % of energetic material and test material, calculate kinetics or perform other suitable compatibility test for test material, energetic material and admixture

Fig. 3A.1: Thermogravimetry compatibility scheme using one test material and energetic material

TEST 3 A-1 DATA SHEET

(Side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B) Report Reference Number: (Unique Reference Number) Page __ of __ Page(s)	
EM PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (including divergences from standard tests) Sample atmosphere: Heating rate (°C/min): Temperature of evaluation (°C): Type of pan: (material, pierced, open) Mass of samples (mg): EM: Test material: Admixture: EM in admixture (m_E): Test material in admixture (m_C):	**TEST RESULTS** W_M (%): W_E (%): W_C (%):
CALCULATIONS W_R (%): (see eq. 3.1)	**ASSESSMENT** COMPATIBLE ☐ YES ☐ NO or ☐ Confirmation test required
DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

Test 3 A-2: Dynamic TGA, Mass Loss at 10% of First Step

1. SUMMARY

The temperature selected corresponds to 10% mass loss of the first TGA step of the mixture. The mass loss due to a reaction between energetic material and test material (interreaction) is calculated. The time to reach this mass loss at ambient temperature (25°C) is extrapolated using a conservative value of activation energy (100 kJ/mole). Assuming a zero order reaction up to 1% mass loss caused by interreaction, the temperature to reach this value after an isothermal storage of 20 years is calculated. If the calculated temperature is higher than 25°C then the energetic material is compatible with the contact material.

NOTE 5: It is recommended to perform additionally a DSC measurement of the mixture. If an endothermic reaction is detected before the first TGA step and if it is caused by melting, then an incompatibility detected by TGA may not necessarily be relevant for lower temperatures. In this case an isothermal TGA (Test 3B) at about 10°C below the melting point or another suitable test should be performed.

2. APPLICABILITY

Analogous to Test 3, Procedure A-1

3. SAMPLE PREPARATION

The sample weight of each component tested is about 10 mg (5 mg for primary explosives). For some energetic materials smaller sample weights or preferably lower heating rates may be used to avoid explosion. Otherwise sample preparation is analogous to Test 3, Procedure A-1.

4. EXPERIMENTAL CONDITIONS

Analogous to Test 3, Procedure A-1. The temperature range of decomposition shifts towards lower temperatures at lower heating rates which makes extrapolations to ambient conditions more reliable. Therefore heating rates below 2°C/min may also be used.

5. PROCEDURE

Run the mixture at first at least until the first step is performed at a certain temperature. The single measurements of the energetic material and the material may then be stopped at this temperature as well. However, for better interpretation of the admixture curves, it is recommended to continue the measurements up to higher temperature.

6. ANALYSIS

Record the data using the data sheet.

Calculate the weight loss due to inter-reaction W_R as described in Test 3 Procedure A-1 Chapter 6.2 and the weight loss relative to the reaction step under investigation W_{rel} (see Test 3 Procedure A-2 Chapter 6.2).

6.1 The starting temperature of the decomposition is the temperature at which a mass loss becomes detectable. It can be detected as the first visible deviation of the extrapolated baseline of an onset construction from the TGA curve or as the first deviation from zero slope of the derivative TGA curve (DTGA), see fig. 3A2.1. If small amounts of a volatile component like water or solvent are present, the starting temperature is the first (flat) TGA inflection point or the corresponding DTGA peak (see fig. 3A2.2).

The end temperature of a step is the temperature at which the TG curve reaches the extrapolated end baseline. If a second step follows, the end temperature corresponds to the flat inflection point between the steps or the DTGA peak (see fig. 3A2.1).

The temperature selected for 10% mass loss of the mixture (T_{10}) is found between the starting and the end temperature of the first step (fig. 3A2.1).

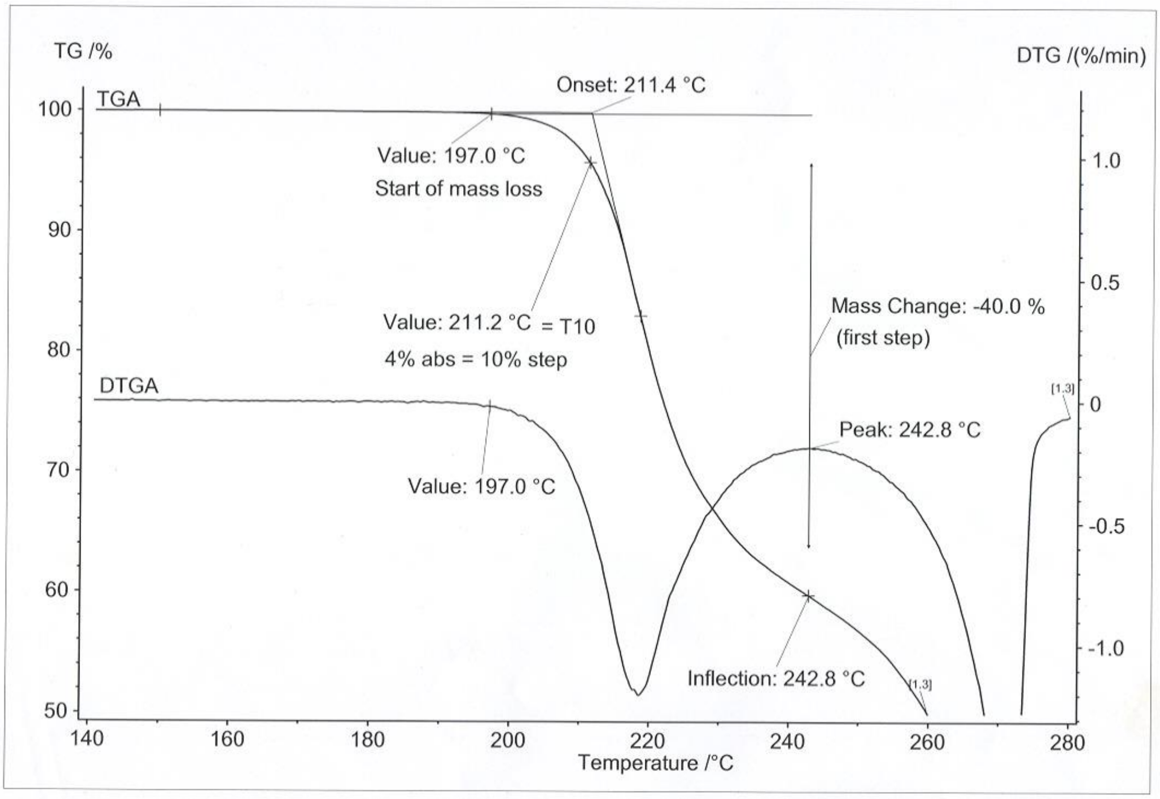


Fig. 3A2.1. Estimation of the reference temperature T_{10} for non-separated TGA steps

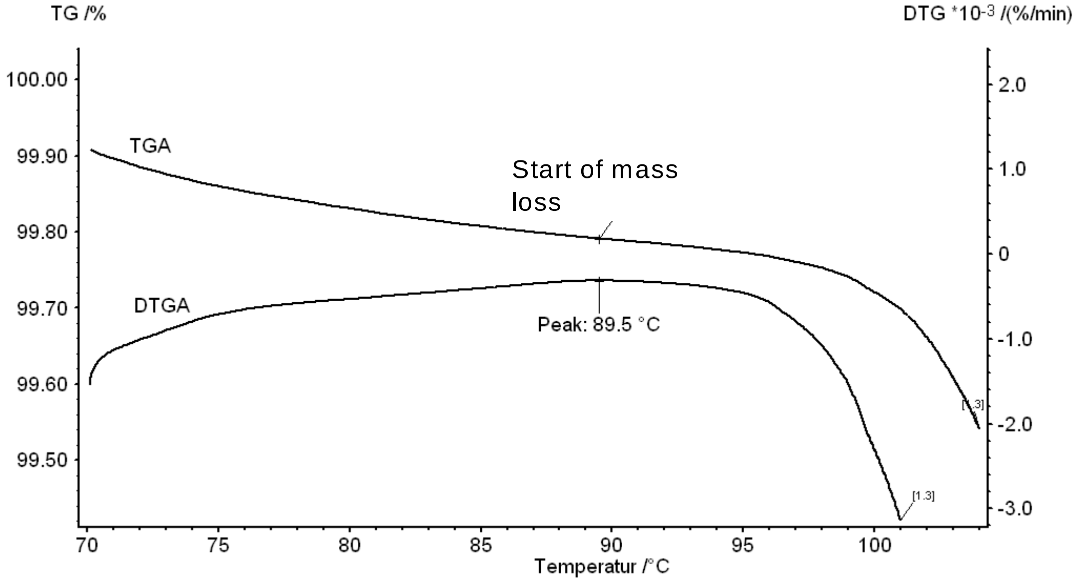


Fig. 3A2.2. Starting point of TGA step of samples containing volatile fractions.

6.2 Calculation of mass loss due to interreaction

W_R is calculated analogous to Test 3, Procedure A-1.

The weight loss as percent of the reaction step under investigation is

$$W_{rel} = 100 \cdot W_R / W_{tot,1st\ step} \qquad \text{(eq. 3.2)}$$

$W_{tot,1st\ step}$ = total weight loss of the first exothermal reaction step

6.3 Extrapolation of the interreaction to an isothermal storage of 20 years

$$T_{20y} = \frac{E}{R \cdot \ln \left[\dfrac{100 \cdot t_{20y} \cdot H \cdot W_R}{W_{tot} \cdot \int_{T_0}^{T_{10}} e^{-E/RT}\, dT} \right]} \qquad \text{(eq. 3.3)}$$

with

T_{20y} = isothermal storage temperature, at which 1% interreaction would be achieved after a storage period of 20 years [K]

T_{10} = temperature at which 10 % decomposition of the first step of the mixture is performed [K]

T_0 = starting temperature of the TGA measurement [K]

R = gas constant

E = activation energy of the interreaction (conservative value of 100 kJ/mole is used)

t_{20y} = 10,519,200 min (= 20 years)

H = heating rate (2°C/min)

W_{tot} = total mass loss of first TGA step of the mixture [%]

The equation 3.3 takes into account at which temperature range the interreaction is observed. The interreaction is assumed to be of zero order.

The integral must be calculated by iteration (e.g. using the Simpson rule) but it can be replaced by very exact approximations e.g.

$$\int_{T_0}^{T_{10}} e^{\frac{-E}{RT}}\, dT \approx \Big\{ [A \cdot (B + RT/E)^n - C] \cdot (RT^2/E) \cdot e^{-E/RT} \Big\}_{T_0}^{T_{10}} \qquad \text{(eq. 3.4)}$$

A = 2.19648063	B = 0.1923358	C = 1.7627246	n = -0.13913

(see Uldis Ticmanis, Manfred Kaiser, Gabriele Pantel, “Combined Dynamic/Isothermal TGA for Stability and Compatibility Testing”, *Proc. Int. Annu. Conf. ICT* 34, 76 (**2003**)).

7. CRITERIA

7.1 Criterion 1: The materials are compatible if the mass loss due to interreaction (W_{rel}) as calculated by eq. 3.2 is less than 1 % of the first TGA step (smaller values may be caused by experimental errors).

7.2 Criterion 2: If $W_{rel} > 1$ % the materials are compatible if the temperature for 1% interreaction after a 20-years isothermal storage is higher than 25°C (calculation by eq. 3.3).

TEST 3 A-2- DATA SHEET

(side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B) Report Reference Number: (Unique Reference Number) Page(s)	Page __ of __
EM PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (including divergences from standard tests) Sample atmosphere: Heating rate (°C/min): Temp. range, total (°C): Temp. range, 1st TGA step (°C): Type of pan: (material, pierced, open) Mass of samples (mg): EM: Test material: Admixture: EM in admixture (m_E): Test material in admixture (m_C):	**TEST RESULTS** W_{tot} (%): T_{10} (°C): W_M (%): W_E (%): W_C (%):
CALCULATIONS 1. W_R (%) (see eq. 3.1 in A-1) 2. W_{rel} (%) (see eq. 3.2) 3. T_{20y} (°C) if 2. > 1% (see eq. 3.3)	**ASSESSMENT** COMPATIBLE ☐ YES ☐ NO
DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

TEST 3, PROCEDURE B - ISOTHERMAL TGA

GENERAL

A second stringent TGA test for compatibility consists of heating the admixture, energetic material and test material, at a specific temperature for 1000 minutes. The weight loss of the admixture, energetic material and test material are compared after a heating period of 1000 minutes at the temperature of interest, as estimated from the results of Test 3A. The same guidelines for compatibility as in dynamic TGA are applied.

Test 3 B-1: Isothermal TGA at 20°C below first DTGA Peak Temperature

1. SUMMARY

The weight losses of the energetic material, the test material and the admixture after a heating period of 1000 minutes at the specified temperature are judged in the same way as in Test 3A, Procedure A-1.

2. APPLICABILITY

Analogous to Test 3, Procedure A-1.

3. SAMPLE PREPARATION

Analogous to Test 3, Procedure A-1.

4. EXPERIMENTAL CONDITIONS

The test temperature is determined by Test 3, Procedure A-1. Use the temperature of the first extrapolated onset of exotherm in the admixture corresponding to a weight loss or 20°C below the weight loss derivative peak. A different isothermal temperature can be used provided prior agreement by all involved parties. This temperature is held for 1000 minutes under an anhydrous nitrogen atmosphere with a flow rate of 50 cm^3/min.

5. PROCEDURE

Run the energetic material, the test material and a 1:1 mixture for 1000 minutes at the specified temperature. Use high heating rates (e.g. 5 or 10°C/min) to reach the constant temperature.

6. ANALYSIS

The weight loss of the energetic material, the test material and the admixture at the end of the heating period are evaluated in the same way as described in Test 3, Procedure A-1.

7. CRITERIA

Analogous to Test 3, Procedure A-1.

TEST 3, Procedure B-1

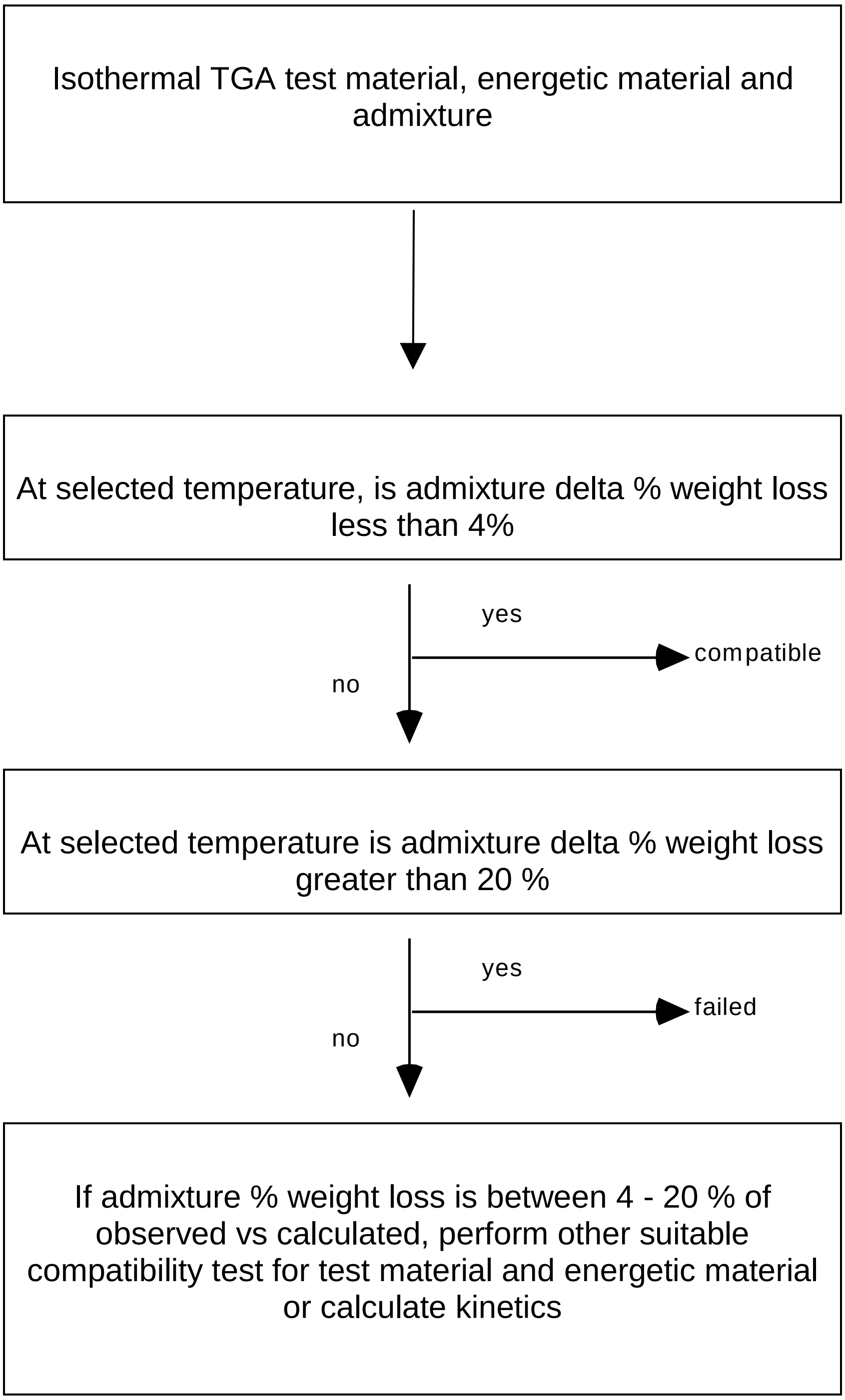


Fig. 3B.1: Isothermogravimetry compatibility scheme using one test material and energetic material.

TEST 3, Procedure B-1 - DATA SHEET

(side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B)

Report Reference Number:
(Unique Reference Number)

Page __ of __ Page(s)

EM PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (including divergences from standard tests) Sample atmosphere: Test temperature (°C): Type of pan: (material, pierced, open) Mass of samples (mg): EM: Test material: Admixture: EM in admixture (m_E): Test material in admixture (m_C):	**TEST RESULTS** W_M (%): W_E (%): W_C (%):
CALCULATIONS W_R (%): (see eq. 3.1 in A-1)	**ASSESSMENT** COMPATIBLE ☐ YES ☐ NO or ☐ Confirmation test required
DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

Test 3 B-2: Isothermal TGA at Calculated Test Temperature

1. SUMMARY

The weight losses of the energetic material, the test material and the admixture after a heating period of 1000 minutes at the specified temperature are judged in the same way as in Test 3A, Procedure A-2.

2. APPLICABILITY

Analogous to Test 3, Procedure A-1.

3. SAMPLE PREPARATION

Analogous to Test 3, Procedure A-1.

4. EXPERIMENTAL CONDITIONS

The test temperature T_{iso} [K] is calculated from the results of Test 3, Procedure A-2.

$$T_{iso} = \frac{E}{R \cdot \ln \dfrac{t_{iso} \cdot H}{[A \cdot (B + RT_{10} / E)^n - C] \cdot (R \cdot T_{10}^2 / E) \cdot e^{-E/RT_{10}}}} \qquad \text{(eq. 3.5)}$$

Calculate the temperature for the isothermal runs using 100 kJ/mole for E and 500 min for t_{iso}. If the true activation energy varies in the range from 80 to 120 kJ/mole, 10% decomposition of the first TGA step of the mixture will be achieved at 250 to 1000 min.

5. PROCEDURE

Analogous to Test 3, Procedure B-1.

6. ANALYSIS

6.1 For isothermal TGA the difference between the observed weight loss of the mixture and the total calculated weight loss of the energetic material and the test material in the admixture at a given time is calculated. The time selected corresponds to 10% mass loss of the first TGA step of the mixture as derived from test 3, Procedure A-2.

6.2 The mass loss during the ‘warming up period’ is neglected. Estimate the time to reach 10% mass loss of the first TGA step of the mixture and the mass loss of the energetic material and the test material within the same time.

6.3 Calculation of mass loss due to interreaction is analogous to that in Test 3, Procedure A-1 (eq. 3.1).

6.4 For extrapolation to the temperature corresponding to 1% interreaction after an isothermal storage of 20 years ($T_{20\,y}$ [K]), use eq. 3.5.

$$T_{20\,y} = \frac{1}{\left(R/E\right)\cdot \ln[(100\cdot t_{20\,y}\cdot W_R)/(t_{10\%}\cdot W_{tot})] + 1/T_{iso}} \qquad \text{(eq. 3.6)}$$

with

$t_{10\%}$ = time after which 10 % decomposition of first TGA step of the mixture is performed [min]

T_{iso} = temperature of isothermal experiment [K]

E = 100 kJ/mole

7. <u>CRITERIA</u>

Analogous to Test 3, Procedure A-2.

TEST 3, Procedure B-2 - DATA SHEET

(side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B) Report Reference Number: (Unique Reference Number)	Page __ of __ Page(s)
EM PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (including divergences from standard tests) Sample atmosphere: Test temperature (°C): Type of pan: (material, pierced, open) Mass of samples (mg): EM: Test material: Admixture: EM in admixture (m_E): Test material in admixture (m_C):	**TEST RESULTS** W_{tot} (%) (from Proc A-2) $t_{10\%}$ (min) W_M (%): W_E (%): W_C (%):
CALCULATIONS 1. W_R (%): (see eq. 3.1 in A-1) 2. W_{rel} (%) (see eq. 3.2 in A-2) 3. T_{20y} (°C) if 2. is > 1% (see eq. 3.6)	**ASSESSMENT** COMPATIBLE ☐ YES ☐ NO
DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

TEST 3, PROCEDURE C - DETERMINATION OF THE KINETICS OF DECOMPOSITION BY TGA

GENERAL

Two alternative procedures are described below. The first is based on dynamic TGA at different heating rates (Procedure C-1), the second on a combination of dynamic and isothermal TGA (Procedure C-2).

Test 3 C-1: Determination of Kinetics Using Multiple Heating Rates

1. SUMMARY

Thermal compatibility determinations are not ideal because they take place at elevated temperatures i.e., > 100°C, in order to detect changes in physical properties that can be measured by DSC and TGA. Incompatibility should be determined at room temperature. Kinetic analysis requires determining the activation energy (E), Arrhenius frequency factor (A), and calculating the decomposition rate constant (k) for the admixture, energetic material and test material. This can be conducted using a variable heating rate method.

DSC and derivative TGA (DTGA) curves are determined for samples using three linear heating rates and recording any exothermic reaction peaks. The kinetic values of E, A and k are calculated from the peak temperature and heating rates following the Kissinger method (Ref. 8-1 and 8-2)

If the test material lowers the activation energy, increases the frequency factor or rate of reaction for decomposition of the admixture, then the test material is incompatible. The reaction rate constant at room temperature may be calculated from the equation:

$$\ln k = \ln A - \frac{E}{RT} \qquad \text{(eq. 3.7)}$$

However, it must be assumed that the mechanism of reaction does not change with change in temperature i.e., that E and A are invariant. If the rate constant for the admixture is significantly higher (a thousand fold) then the test material is likely to be incompatible at room temperature.

The kinetic constants obtained by the variable heating rate method must be verified by performing a 60-minute half-life determination (see paragraphs 6.7 to 6.10 for details). The mass loss is determined and must agree to within ± 5 % of the calculated value.

The determination of the kinetics of decomposition is very labour intensive. It is suggested that whenever possible other compatibility tests, as described in this AOP, are performed to confirm the compatibility results of Tests 3, Procedure A-1 and B-1. Test 3C-1 should only be undertaken when either kinetic data is required or compatibility of a material cannot be confirmed by any other means.

2. APPLICABILITY

Analogous to Test 3, Procedure A-1.

3. SAMPLE PREPARATION

Analogous to Test 3, Procedure A-1.

4. EXPERIMENTAL CONDITIONS

Analogous to Test 3, Procedure A-1.

5. PROCEDURE

5.1 Run each individual energetic material, test material and admixture at a heating rate of 0.2°C/min. The starting temperature should be ambient or at least 50°C below the first observed exothermic peak. Continue heating until the corresponding TGA step is finished. Generally the first decomposition peak or weight loss step is the peak of interest. This may or may not be due to the energetic ingredient.

5.2 Repeat step 5.1 using at least two other heating rates between 0.2 and 2°C/min. A minimum of three heating rates is required. Suggested heating rates are 0.5, 1, and 2°C/min. In the case of energetic materials with high activation energies, it may be necessary to expand the heating rate range.

6. ANALYSIS

6.1 Temperatures of DTGA reaction peak maxima are recorded for each energetic material, test material and admixture. Temperatures should be corrected for temperature scale nonlinearity, heating rate changes, and thermal lag according to instrument manuals.

6.2 Plot $\ln(H/T^2)$ versus 1/T. H is the heating rate in °C/min and T is the corrected peak maximum temperature in K.

6.3 The activation energy can be determined from the slope, S, of the line according to the following equation:

$$E = -S \cdot R \qquad \text{(eq. 3.8)}$$

with

E = Activation energy (kJ mole^{-1})

R = Gas constant (0.0083143 kJ K^{-1} mole^{-1})

S = Slope

6.4 The Arrhenius frequency factor, A (min^{-1}), can be calculated from the following equation. H used in this equation is the heating rate from one of the middle heating rates.

$$A = \frac{H \cdot E \cdot e^{(E / RT)}}{R \cdot T^2} \qquad \text{(eq. 3.9)}$$

6.5 Other methods of calculation may be used (Ref 8-2).

6.6 The specific reaction rate constant, k, can then be determined using the Arrhenius equation,

$$k = A \cdot e^{\frac{-E}{RT}} \qquad \text{(eq. 3.10)}$$

The value of k should be calculated for all three temperatures for each energetic material, test material and admixture.

6.7 Calculate the half-life times of reaction for k values using the following equation which assumes a first order reaction:

$$t_{½} = \frac{0.693}{k} \qquad \text{(eq. 3.11)}$$

An example is as follows:

Set 60 minutes = $t_{1/2}$

Then k = 0.693/60 = 0.1155 min^{-1}

The rate for 60 minutes is then 0.11555 min^{-1}

Substitute into the equation log k = log A - E/2.3·R·T and solve for T.

6.8 Age a sample of the admixture isothermally at the calculated temperature for 60 minutes. Repeat this test once again.

6.9 Run both aged samples and an unaged sample under the same temperature and heating rate conditions.

6.10 The weight loss of the aged sample should be ½ of that of the unaged sample. Determination of mass loss must be performed at exactly the same temperature range used to determine the original k value. Reaction kinetics are confirmed if observed results are within ± 5 % of the calculated values.

7. CRITERIA

7.1 The rate constant, frequency factor and activation energy of the admixture and energetic material are compared. The following criteria apply when interpreting the data.

7.2 If the activation energy is lower and the frequency factor is larger for the admixture than for the energetic material, then the test material is considered incompatible.

7.3 If only small differences in activation energy and frequency factors are observed but the rate constant of the admixture is greater than that of the energetic material at room temperature, then the test material is considered incompatible.

7.4 However small the difference in rate constants, if the value for the admixture at the temperature of interest is equal to or less than 10, then there is a strong indication that the test material is compatible.

7.5 If the temperature of interest is elevated (i.e. > 200°C) then the rate constant would be much lower at room temperature and would be compatible.

8. REFERENCES

H.E. Kissinger, *Analytical Chem*. 29, 1702 (**1957**)

H.E. Kissinger, *J. Res. NBS* 57, 217 (**1956**).

TEST 3, Procedure C-1 - DATA SHEET

(side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B)

Report Reference Number:
(Unique Reference Number)
Page __ of __ Page(s)

EM PREPARATION

TEST MATERIAL PREPARATION

TEST CONDITIONS
(including divergences from standard tests)

Sample atmosphere:
Heating rates H (°C/min):
Total temperature range (°C):
Type of pan:
(material, pierced, open)
Mass of samples (mg):
EM:
Test material:
Admixture:
EM in admixture (m_E):
Test material in admixture (m_C):

TEST RESULTS

Derivative peak maxima (°C):

	H1	H2	H3
EM			
Test mat.			
Admixt.			

CALCULATIONS

Activation energy E (kJ/mole)
(see eq. 3.8)

Frequency factor A (min^{-1})
(see eq. 3.9)

Rate constant k (min^{-1})
(see eq. 3.10)

ASSESSMENT

Dynamic procedure
COMPATIBLE
☐ YES
☐ NO

Isothermal half-life test confirmation
☐ YES
☐ NO

DATA SENT TO: (Name and address of person receiving this information)

COMMENTS

NATO AOP 4147 DATA SHEET (Side C)
HALF LIFE TEST

Report Reference Number:
(Unique Reference Number)

Page __ of __ Page(s)

TEST CONDITIONS
(including divergences from standard tests)

Sample atmosphere:
Ageing temperature for 1 h (°C):
Heating rate (°C/min):
Type of pan:
(material, pierced, open)
Mass of samples (mg):
EM:
Test material:
Admixture:
EM in admixture (m_E):
Test material in admixture (m_C):

TEST RESULTS

Mass loss (%):	Aged	Unaged
Average		

CALCULATIONS

1. av. mass loss, aged W_A (%)
2. av. mass loss, aged W_U (%)
3. 100·[1-(W_A/W_U)] (%)
(Deviation from prediction)

ASSESSMENT

Deviation from prediction within ± 5 %

☐ YES
☐ NO

DATA SENT TO: (Name and address of person receiving this information)

COMMENTS

Test 3 C-2 Determination of Kinetics Using Dynamic and Isothermal TGA

1. SUMMARY

If an energetic material is found to be incompatible by tests 3, Procedure A-2 and Procedure B-2, the data obtained from these tests can be used for the determination of the activation energy of the interreaction without additional experiments. This determined value is then applied for interpolation to ambient temperature instead of using 100 kJ/mole. The reference point (1% mass loss of the first TGA step of the mixture) remains unchanged.

2. APPLICABILITY

Analogous to test 3, Procedure A-1, but it is recommended to perform test 3C only if the criteria for compatibility are failed in test 3, Procedure A-2 and Procedure B-2.

3. SAMPLE PREPARATION

Analogous to test 3, Procedure A-1.

4. EXPERIMENTAL CONDITIONS

Analogous to test 3, Procedure A-2 and B2

5. PROCEDURE

If the data are not available from test 3, Procedure A-2 and B-2, perform measurements as defined there.

6. ANALYSIS

A suitable programme for mathematical treatment of TGA curves must be available. For determination of the first weight loss step see procedure A-2, para 6.1.

6.1 Calculate the mass loss curve due to interreaction in the dynamic series taking into account deviations from 1:1 ratio in the mixture (see eq. 3.1 in test 3, Procedure A-1).

6.2 Calculate the mass loss curve of the interreaction in the isothermal series in the same way.

6.3 Select about 5 isoconversional points within the total range of the interreaction from the dynamic and the isothermal curves. (e.g. 0.5; 1; 1.5; 2; and 2.5 %).

6.4 These corresponding dynamic temperatures T_{dyn} and isothermal times t_{iso} are used to calculate the best value for the activation energy from

$$\sum \left[\frac{t_{iso} - f(T_{dyn})}{t_{iso}} \right]^2 = min\ imum \qquad \text{(eq. 3.12)}$$

where

$$f\left(T_{dyn}\right) = \frac{1}{H} \cdot e^{\frac{E}{RT_{iso}}} \cdot \int_{T_0}^{T_{dyn}} e^{\frac{-E}{RT}} dT \qquad \text{(eq. 3.13)}$$

where T_{dyn} = dynamic temperature to reach a certain degree of W_R [K]

t_{iso} = isothermal time to reach the same degree of W_R [min]

The integral can be replaced by the approximation given in eq. 3.4 test 3, Procedure A-2 (replace T_{10} by T_{dyn})

6.5 Use the time estimated for 1% of interreaction from the isothermal series ($t_{1,\ iso}$ in years) to calculate the temperature T_{20y} from:

$$T_{20y} = \frac{1}{\frac{R \cdot \ln(20 / t_{1,iso})}{E} + \frac{1}{T_{iso}}} \qquad \text{(eq. 3.14)}$$

7. <u>CRITERION</u>

If the temperature T_{20y} calculated for 1% interreaction after an isothermal storage for 20 years is more than 25°C, the materials are compatible.

TEST 3, Procedure C-2 - DATA SHEET

(side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B)

Report Reference Number:
(Unique Reference Number)

Page __ of __ Page(s)

EM PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (including divergences from standard tests) dynamic TGA see Data Sheet Test 3-A2 isothermal TGA see Data Sheet Test 3-B2	**TEST RESULTS** Results from Test 3-A2 and Test 3-B2 should be noted here.
CALCULATIONS Isoconversional points selected:	**ASSESSMENT** COMPATIBLE ☐ YES ☐ NO

W_R (%)	T_{dyn} (°C)	t_{iso} (min)

Activation energy E (kJ/mole)
(see eq. 3.12)

T_{20y} (°C)
(see eq. 3.14)

DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

TEST 4 - DIFFERENTIAL SCANNING CALORIMETRY (DSC)

For comparison of compatibility results performed by DSC it is absolutely necessary to document the degree of sealing of a sample container (hermetically closed, closed with a pierced lid or open), because for many energetic materials (e.g. propellants and high explosives) both the total heat released by the decomposition and the heat generation rate at a given temperature are much higher in a hermetically closed container than in an open one.

TEST 4, PROCEDURE A - SINGLE HEATING RATE DSC

GENERAL

Two alternative ways of evaluation are described. The first refers to peak temperature shifting due to interreaction (Procedure A-1), the second to additional heat release (Procedure A-2).

Test 4 A-1: Temperature Shifting of DSC Peak Maxima

1. SUMMARY DESCRIPTION

The DSC sample is contained either in a crimped, sealed pan with a pin hole in the lid or in a coated hermetically sealed pan in order to determine if the evolved gases affect the decomposition reaction.

Dynamic DSC is carried out at 2°C/min in a nitrogen or air atmosphere on the energetic material, the test material and a 1:1 mixture by weight. To ensure that all relevant thermal effects fall in the stable dynamic part of the measurement, the test should start at ambient temperature or a minimum of 50°C below the onset of the peak of interest of the admixture.

The peak temperatures of decomposition of the admixture, energetic material and test materials are noted. If the peak due to the admixture is shifted to a lower temperature than the first observed peak due to the energetic material or test material, then this is an indication of a degree of incompatibility. As the temperature difference between the admixture, energetic material and test material peaks increases so does the degree of incompatibility.

2. APPLICABILITY

Analogous to Test 3, Procedure A-1.

3. SAMPLE PREPARATION

Analogous to Test 3, Procedure A-1.

4. EXPERIMENTAL CONDITIONS

Analogous to Test 3, Procedure A-1 (Exception: Open pans are not recommended for DSC).

5. PROCEDURE

5.1 Decomposition profiles are run on each individual material following the experimental conditions.

5.2 Maximum peak temperatures are determined for the primary reaction peak.

5.3 Energetic materials and test materials are combined in a 1:1 ratio and decomposition profiles determined. A matrix can be used if an investigation requires more than two materials to be tested.

5.4 Maximum peak temperatures of the resulting exotherms of the combinations are determined.

NOTE 6: A number of exotherms may be obtained.

5.5 A DSC for each individual energetic material, test material and admixture shall be run in duplicate as a minimum. The average of duplicate runs shall be used in the calculations.

6. ANALYSIS

6.1 Shifts in the peak temperature of the lowest exotherm in the mixture are examined. If the shift of this peak is towards lower temperature than that of the less stable of both individual components then this indicates incompatibility.

6.2 Calculation of the shift in peak temperatures can be determined by subtracting the lowest of both the peak temperature of the energetic material or the test material from the lowest mixture peak temperature and is referred to as the delta.

NOTE 7: For those materials that undertake a phase change and also demonstrate a marked shift in the reaction peak of interest, further investigation is necessary.

6.3 Peak shapes must also be examined qualitatively for any changes that may occur due to an interaction between energetic material and test materials. Specific changes to look for include:

6.3.1 Evidence of a peak becoming narrower (peak height to half-width)

6.3.2 Peak forms a loop or leans towards a higher temperature. These effects correspond to a peak in the time-temperature curve due to a very high heat flow. The measurement should be repeated with smaller sample amounts or lower heating rates.

6.3.3 Appearance or disappearance of new or old peaks.

7. CRITERIA

7.1 Any delta of 4°C or less indicates the test material is compatible. If the shift in peak temperature is towards a lower temperature, and greater than 4°C then the test material shows a degree of incompatibility. If the shift in peak temperature is towards a lower temperature, and greater than 20°C then the test material is incompatible.

7.2 The appearance of additional peaks or disappearance of old peaks may indicate incompatibility and further investigations must be undertaken.

7.3 Changes in peak shape may indicate incompatibility and further investigations must be undertaken.

7.4 A flow chart has been developed to follow the process of decisions required when determining whether a sample is compatible or not. Figure 4.1 lists a flow diagram which describes the decision process when one test material and one energetic material are tested for compatibility using DSC.

NOTE 8: A shift in peak temperature of the admixture of between 4°C and 20°C below the original peak temperatures of the energetic material and test materials indicates a degree of incompatibility. If test data falls into this “grey area” it may be necessary to obtain supporting data from alternative compatibility tests. Kinetic information can be used to provide specific information and procedures are outlined in Test 3C. However, it should be noted that reaction kinetic tests are extremely time consuming and other tests described in this AOP may be more suitable for confirming compatibility.

TEST 4, Procedure A-1

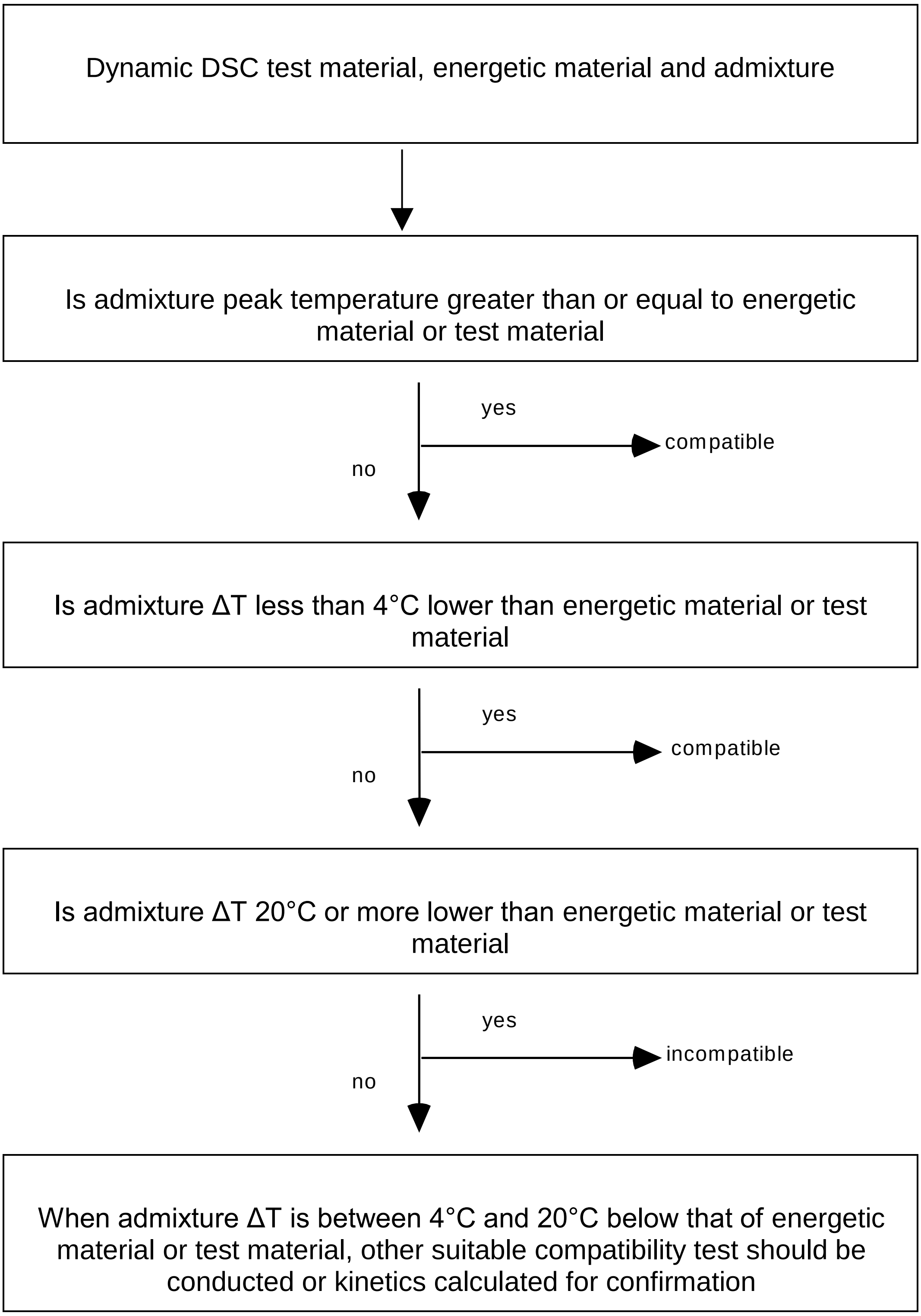


Fig. 4A.1: DSC compatibility scheme using one ingredient and energetic material

TEST 4, Procedure A-1 - DATA SHEET

(side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B) Report Reference Number: (Unique Reference Number) Page __ of __ Page(s)	
EM PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (including divergences from standard tests) Sample atmosphere: Heating rate (°C/min): Temperature range (°C): Type of pan: (material, sealed, pierced, open) Mass of samples (mg): EM: Test material: Admixture: EM in admixture (m_E): Test material in admixture (m_C):	**TEST RESULTS** First DSC peak temperatures (°C): Admixture T_M (°C) EM T_E (°C) Test material T_C (°C)
CALCULATIONS T_M - T_E or T_C (the lower of both) (°C):	**ASSESSMENT** COMPATIBLE ☐ YES ☐ NO or ☐ Confirmation test required
DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

Test 4 A-2: Heat Release at 10% of DSC Peak Area

1. SUMMARY DESCRIPTION

Dynamic DSC is carried out at 2°C/min in nitrogen or air atmosphere on the energetic material, the test material and a 1:1 mixture by weight. The sum of the heat release of the energetic material and test material is compared to that of the mixture of both at a selected temperature. The temperature selected corresponds to 10% heat release of the first exothermic or endothermic peak of the mixture. The heat release due to interreaction is calculated.

2. APPLICABILITY

Analogous to Test 3, Procedure A-1.

3. SAMPLE PREPARATION

Analogous to Test 3, Procedure A-1.

4. EXPERIMENTAL CONDITIONS

Analogous to Test 3, Procedure A-1.

5. PROCEDURE

Run the mixture initially at least until the first peak is exhibited at a certain temperature. The single measurements of the energetic material and the material may then be stopped at this temperature as well. However, for better interpretation of the admixture curves, it is recommended to continue the measurements up to higher temperature.

6. ANALYSIS

6.1 In dynamic DSC the difference between the observed heat release by the mixture and the sum of calculated heat release by the energetic material and the test material in the mixture at a given temperature is noted. The temperature selected corresponds to a 10% area of the first DSC peak of the mixture (T_{10}).

6.2 For integration use preferably a tangential baseline type, which connects the extrapolated first and second (end) baseline with a curve corresponding to the momentary inversion (see fig. 4A2.1).

If the software cannot construct this baseline, the best approximation is a two-step integration by extrapolating the first and second baseline up to the peak temperature (see fig. 4A2.2).

A linear baseline of the peak connecting the first deviation of the curve from the first and second baseline can cause larger errors in the estimation of T_{10} see fig. 4A2.3. In the example T_{10} is found as 272.60°C, which corresponds to 12.3 % of the peak area instead of 10% if the (true) tangential baseline is used.

In many cases there will be a first peak not completely separated from the next, sometimes overlapping with a preceding phase transition. The evaluation of these types of DSC curves is demonstrated in fig. 4A2.4.

The temperatures of starting and ending integration should be kept the same for evaluation of the mixture and the individual components.

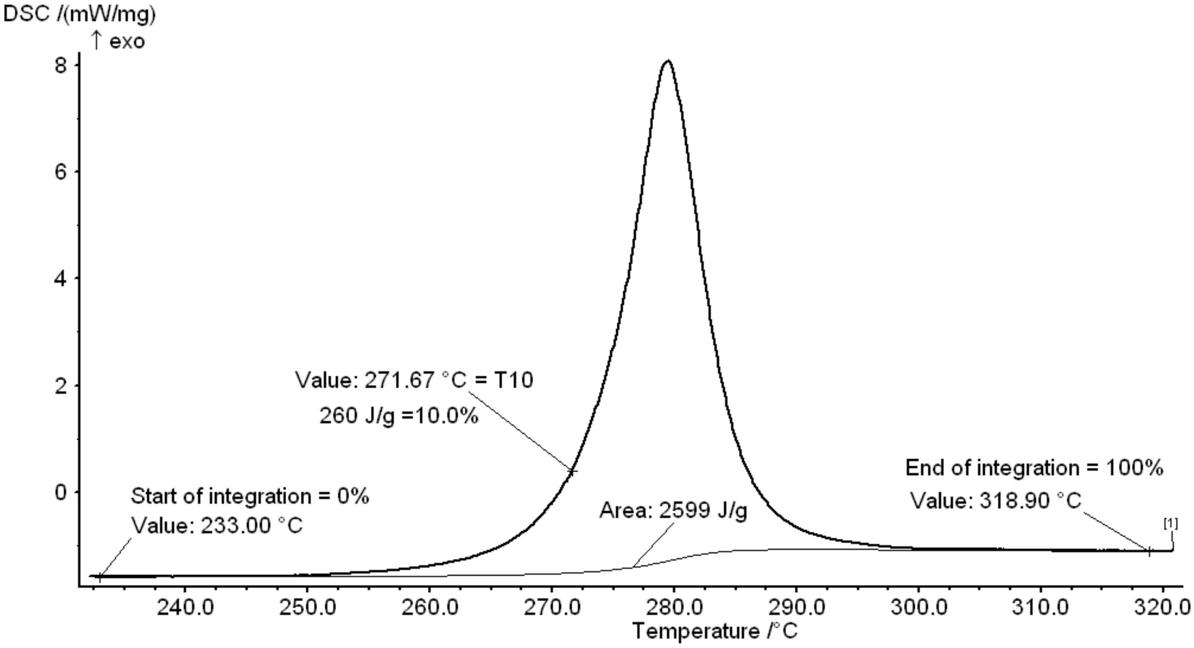


Fig. 4A2.1. Evaluation of T_{10} using a tangential baseline

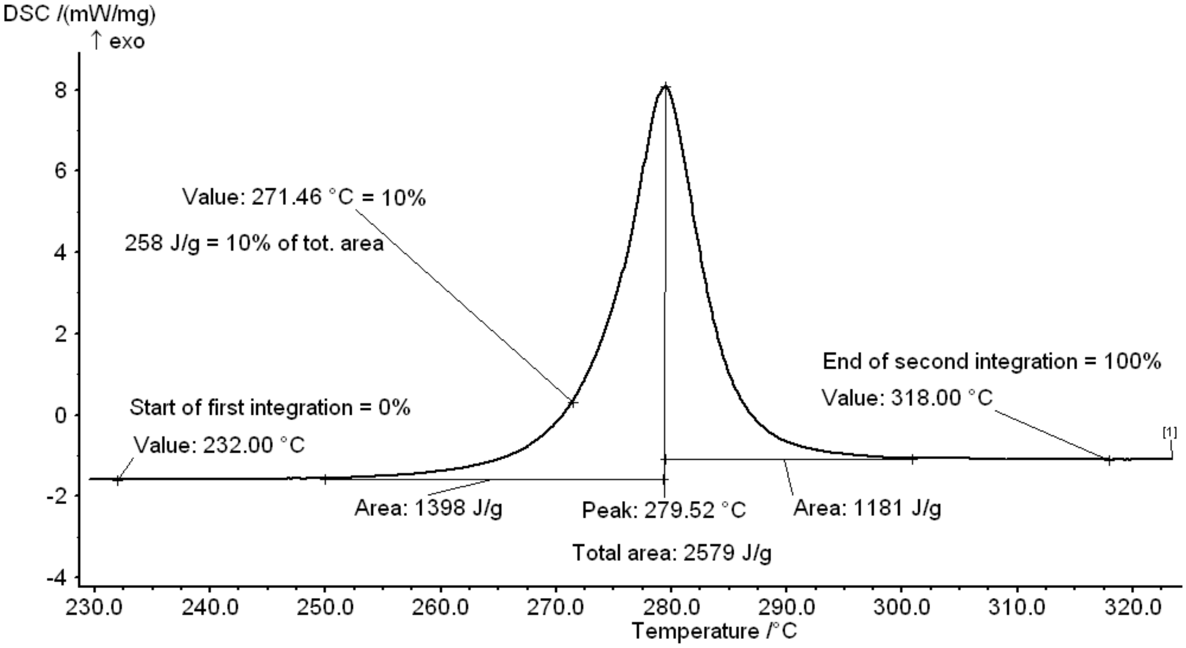


Fig. 4A2.2. Evaluation of T_{10} using a two-step integration

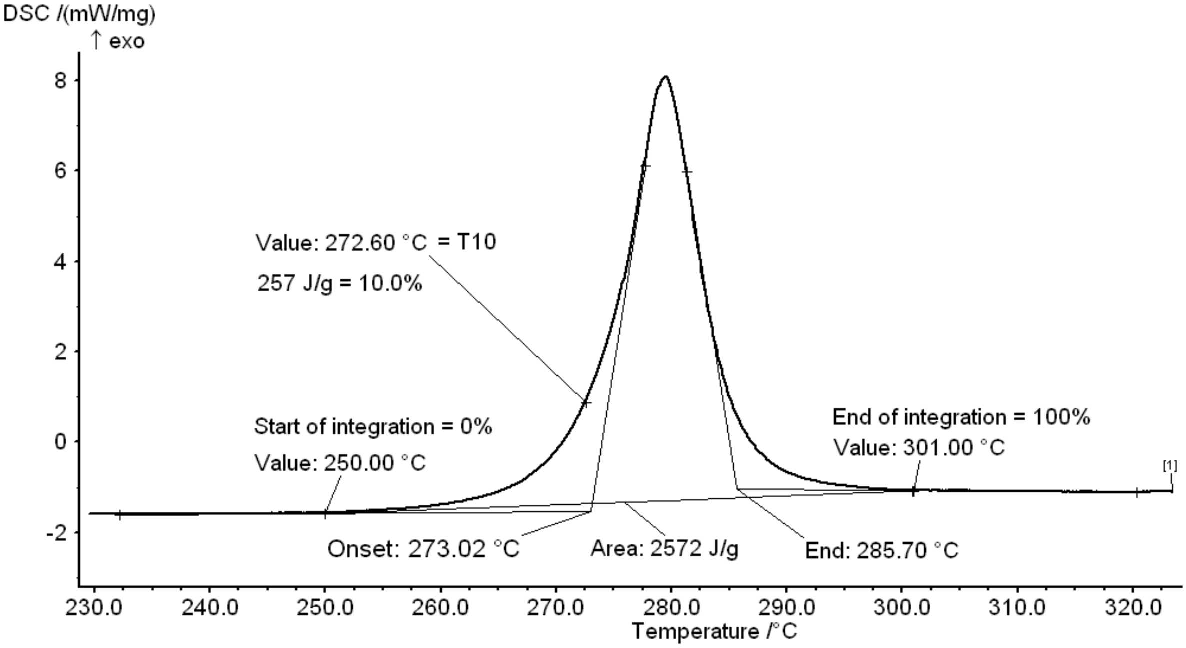


Fig. 4A2.3. Evaluation of T_{10} using a linear baseline

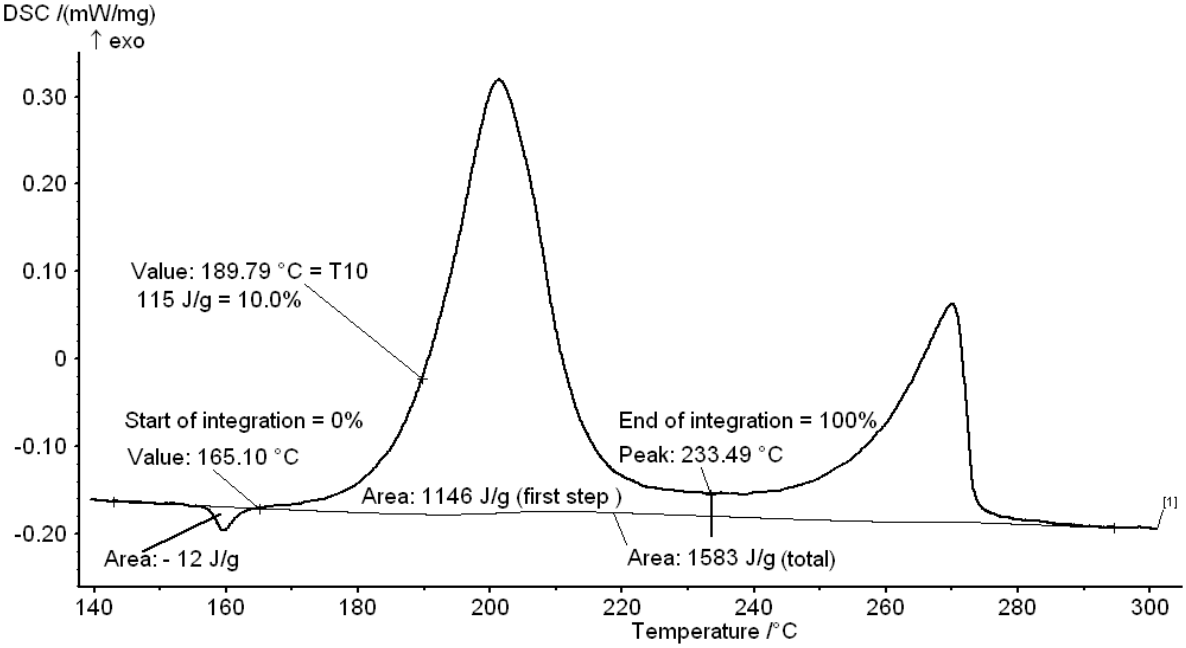


Fig. 4A2.4. Evaluation of T_{10} for a non-separated two-step reaction

6.3 Calculation of the heat release due to interreaction

$$Q_R = Q_M - Q_{M,calc} = Q_M - (m_E \cdot Q_E + m_C \cdot Q_C)/(m_E + m_C) \qquad \text{(eq. 4.1)}$$

with

Q_R = energy release due to interreaction [J/g]

Q_M = energy release of the mixture [J/g]

$Q_{M,calc}$ = energy release of the mixture, calculated without interreaction [J/g]

Q_E = energy release of the energetic material in the single measurement [J/g]

Q_C = energy release of the contact material in the single measurement [J/g]

m_E = mass of the energetic material in the mixture [mg]

m_C = mass of the contact material in the mixture [mg]

The heat release due to interreaction as percent of the 1st peak´s area is

$$Q_{rel} = 100 \cdot Q_R / Q_{tot} \qquad \text{(eq. 4.2)}$$

with

Q_{tot} = total energy release of first DSC peak of the mixture [J/g]

6.4 Calculation of temperature for 2 % interreaction after an isothermal storage period of 20 years (T_{20y} [K])

$$T_{20y} = \frac{E}{R \cdot \ln\left[\dfrac{100 \cdot t_{20y} \cdot H \cdot Q_R}{Q_{tot} \cdot P_{cr} \cdot \int_{T_0}^{T_{10}} e^{-E/RT}\, dT}\right]} \qquad \text{(eq. 4.3)}$$

with

t_{20y} = 10,519,200 min (= 20 years)

T_{10} = temperature at which 10 % decomposition of the first step of the mixture is performed [K]

T_0 = starting temperature of the DSC measurement [K]

R = gas constant

E = activation energy of the interreaction (conservative value of 100 kJ/mole is used)

H = heating rate (2°C/min)

P_{cr} = tolerable fraction of Q_{tot} due to interreaction (= 2 %)

7. CRITERIA

Criterion 1:

The materials are compatible if the interreaction heat Q_{rel} is between -2 and + 2 %.

Criterion 2:

If Q_{rel} > +2 % the materials are compatible if the temperature corresponding to 2 % interreaction after a storage period of 20 years is more than 25°C.

Criterion 3:

If Q_{rel} < -2 % the test is unable to distinguish a true stabilizing effect from an endothermic interreaction. In this case tests based on gas evolution or mass loss (Tests 1 or 3A) should be performed additionally).

TEST 4, Procedure A-2 - DATA SHEET

(side A see page A-5)

<table>
<tr><td colspan="2">NATO AOP 4147 DATA SHEET (Side B)
Report Reference Number:
(Unique Reference Number)
Page __ of __ Page(s)</td></tr>
<tr><td>EM PREPARATION</td><td>TEST MATERIAL PREPARATION</td></tr>
<tr><td>TEST CONDITIONS
(including divergences from standard tests)
Sample atmosphere:
Heating rate (°C/min):
Temp. range, total (°C):
Temp. range, 1st DSC peak (°C):
Type of pan:
(material, sealed, pierced, open)
Mass of samples (mg):
EM:
Test material:
Admixture:
EM in admixture (m_E):
Test material in admixture (m_C):</td><td>TEST RESULTS
Q_{tot} (J/g):
T_{10} (°C):
Q_M (J/g):
Q_E (J/g):
Q_C (J/g):</td></tr>
<tr><td>CALCULATIONS
1. Q_R (J/g):
(see eq. 4.1)
2. Q_{rel} (%)
(see eq. 4.2)
3. T_{20y} (°C) if 2. > 2 %
(see eq. 4.3)</td><td>ASSESSMENT
COMPATIBLE
☐ YES
☐ NO</td></tr>
<tr><td>DATA SENT TO: (Name and address of person receiving this information)</td><td>COMMENTS</td></tr>
</table>

TEST 4, PROCEDURE B -
DETERMINATION OF KINETICS BY MULTIPLE HEATING RATE

1. SUMMARY

Analogous to Test 3, Procedure C-1. (Exception: Additional TGA measurements are not necessary)

2. APPLICABILITY

Analogous to Test 3, Procedure A-1.

3. SAMPLE PREPARATION

Analogous to Test 3, Procedure A-1.

4. EXPERIMENTAL CONDITIONS

Analogous to Test 3, Procedure A-1.

5. PROCEDURE

5.1. Run each individual energetic material, test material and admixture at a heating rate of 0.2°C/min. The starting temperature should be ambient or at least 50°C below the first observed exothermic peak. Continue heating through the decomposition peak until the curve returns to or near the baseline that was observed before the decomposition peak. Generally the first decomposition peak is the peak of interest. This may or may not be due to the energetic ingredient.

5.2. Repeat step 5.1 using at least two other heating rates between 0.2 and 2°C/min. A minimum of three heating rates is required. Suggested heating rates are 0.5, 1, and 2°C/min. In the case of energetic materials with high activation energies, it may be necessary to expand the heating rate range.

6. ANALYSIS

Analogous to Test 3, Procedure C-1, but with following modifications:

- Replace DTGA by DSC
- (mod. in 6.8) Age a sample isothermally at the calculated temperature for 60 minutes. Remove the sample and cool to ambient temperature. Allow the DSC to cool to room temperature. Repeat this test a minimum of 5 times because the integration is performed on a peak that has a mass which is decreasing due to decomposition.

- (mod. in 6.9) Run each of the five aged samples and an unaged sample under the same temperature and heating rate conditions.
- (mod. in 6.10) The peak area or displacement from the baseline of the aged sample should be ½ of that of the unaged sample. Integration of the peak areas must be performed at exactly the same temperature range used to determine the original k value. The five integrations are averaged if DSC is used. Reaction kinetics are confirmed if observed results are within ± 5 % of the calculated values.

7. CRITERIA

Analogous to Test 3, Procedure C-1.

TEST 4, Procedure B - DATA SHEET

(side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B)

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EM PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (including divergences from standard tests) Sample atmosphere: Heating rates H (°C/min): Total temperature range (°C): Type of pan: (material, sealed, pierced, open) Mass of samples (mg): EM: Test material: Admixture: EM in admixture (m_E): Test material in admixture (m_C):	**TEST RESULTS** DSC peak maxima (°C):
CALCULATIONS Activation energy E (kJ/mole) (see eq. 3.8) Frequency factor A (min^{-1}) (see eq. 3.9) Rate constant k (min^{-1}) (see eq. 3.10)	**ASSESSMENT** Dynamic procedure COMPATIBLE ☐ YES ☐ NO Isothermal half-life test confirmation ☐ YES ☐ NO
DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

DSC peak maxima (°C):

	H1	H2	H3
EM			
Test mat.			
Admixture			

NATO AOP 4147 DATA SHEET (Side C)
HALF LIFE TEST

Report Reference Number:
(Unique Reference Number)

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TEST CONDITIONS

(including divergences from standard tests)

Sample atmosphere:
Ageing temperature for 1 h (°C):
Heating rate (°C/min):
Type of pan:
(material, sealed, pierced, open)
Mass of samples (mg):
EM:
Test material:
Admixture:
EM in admixture (m_E):
Test material in admixture (m_C):

TEST RESULTS

Peak area (J/g):	Aged	Unaged
Average		

CALCULATIONS

1. av. peak area, aged Q_A (J/g)
2. av. peak area, aged Q_U (J/g)
3. $100 \cdot [1-(Q_A/Q_U)]$ (%)
   (Deviation from prediction)

ASSESSMENT

Deviation from prediction within ± 5 %
☐ YES
☐ NO

DATA SENT TO: (Name and address of person receiving this information)

COMMENTS

TEST 5 - CHEMICAL ANALYSIS OF PROPELLANTS AFTER AGEING

1. SUMMARY DESCRIPTION

Artificial aging and chemical analysis can be used for the assessment of compatibility with nitrate ester based propellants. The procedure describes the method for assessing the compatibility of nitrate ester based propellants.

The percentage stabilizer remaining, when a propellant sample in contact with a 10 % addition of the material under test is heated at a constant elevated temperature for 336 hours (14 days), is compared with the percentage stabilizer in an unheated sample and a sample heated under the same test conditions but in the absence of test material. Compatibility is judged by means of the additional loss of stabilizer because of contact with the material under test.

NOTE 9: Reactions between propellants and contact material may occur which produce gas and generate heat but do not lead to stabilizer depletion. Therefore the results of Test 5 may differ from those of Test 1 and Test 2.

2. APPLICABILITY

The compatibility test is used for determining the reactivity of materials of munition components with nitrate ester based propellants.

3. INTERPRETATION OF RESULTS

Materials which fail to satisfy the requirements of this test will be judged as unsuitable for use in applications where permanent direct contact or close proximity with the test propellant is required or where adequate precautions to guard against such contact are not taken.

4. APPARATUS

One of the following, or a suitable equivalent, may be used to heat the propellant samples.

A constant temperature oven capable of maintaining a specified temperature to ± 0.2°C over a period of 336 hours (14 days).

An electrically heated metal block bath capable of maintaining a specified temperature to ± 0.2°C over a period of 336 hours (14 days). The block shall contain drilled holes able to accommodate the total length of the sample heating tubes. The diameter of the holes shall not be more than 2 mm greater than the diameter of the heating tube. An electrical cut-out shall be fitted that operates at 5°C above the nominal working temperature.

Glass heating tube of uniform bore, 25 mm x 152 mm with a ground glass joint capable of accepting a ground glass stopper, as illustrated in Figure 5.1.

5. PREPARATION OF PROPELLANT TEST SAMPLE

Where appropriate and with necessary precautions, grind, rasp or subdivide the propellant to pass through a sieve with a 2.0 mm opening. Discard material which passes a 0.2 mm sieve. Propellants which do not require grinding or sub-division to pass a 2 mm sieve are not to be sieved before use to remove fine particles.

Propellant shall not be artificially dried before testing.

6. PREPARATION OF CONTACT MATERIAL SAMPLE

The contact material used for the test shall be, as far as is practicable, in the condition proposed for contact with the propellant. Where the intended use requires the contact material to be applied in an intermediate (uncured or partly cured) condition to the propellant, the compatibility test shall be made on mixtures so prepared.

Most solid contact materials require to be subdivided for testing to obtain effective mixing and a high degree of contact with the propellant during the test.

Wherever possible, grind, rasp or cut solid contact material to particles with a maximum size of 2 mm. Discard material which has been produced by the process of subdivision and which passes a 0.2 mm sieve.

Contact materials should not be artificially dried before testing but the presence of excessive quantities of volatile ingredients may make the test difficult to perform. To remove excessive quantities of water, preliminary drying over silica gel for 24 hours at ambient temperature is permitted. Volatile solvents should be removed according to the manufacturers recommended drying procedures. These treatments must be recorded on the data sheet.

7. COMPATIBILITY TEST

7.1 Preliminary test

A preliminary test is applied before testing materials such as accelerators, catalysts, amines, hardeners, alkaline materials or other reactive compounds thought to be capable of causing a rapid and hazardous reaction with the propellant under test.

Conduct the test in a fume cupboard or safety cabinet. On a watch-glass, mix approximately 25 mg each of the propellant and test material. Allow to stand at room temperature for one hour. If no reaction occurs, place on a boiling water bath and observe.

If the mixture darkens and fumes when cold, the test material is incompatible with the propellant and no further testing should be carried out.

If the reaction occurs when heated, the test material is likely to be incompatible with the propellant and further testing should be at the discretion of the Testing Authority.

7.2 Procedure

7.2.1 Preparation of the admixture

Mix 5 ± 0.05 g of the prepared propellant with 0.5 ± 0.01 g of the test material and transfer the mixture to a glass heating tube as illustrated in Figure 5.1, taking care to avoid contamination of the ground glass joint of the tube (the introduction of the propellant and the test material can be facilitated by the use of a wide-bore short stemmed glass funnel). Lightly close using a ground glass stopper and place the tube containing propellant and the test material mixture into the heating apparatus set at the specified test temperature.

Smaller sample sizes may be used, but the ratio of propellant to test material shall be maintained. The amount of sample used shall be recorded on the data sheet.

Perform each test in duplicate.

7.2.2 Preparation of the control samples

Transfer accurately weighed samples of about 5 g, weighed to an accuracy of ± 0.05 g, of the prepared propellant to two glass heating tubes.

Lightly stopper the tubes using ground glass stoppers as illustrated in Figure 5.1, and place one tube into the heating apparatus set at the specified test temperature. Store the second tube of propellant at ambient temperature. Perform each test in duplicate.

7.2.3 Sample aging

Determine the temperature at which, after aging for 336 hours (14 days), a minimum of 20% of the propellant stabilizer is consumed but 50% depletion is not exceeded. 80°C has been found to be a suitable test temperature for most propellants stabilized with 1% or more of ethyl centralite or with diphenylamine. A lower temperature is recommended for propellants stabilized with 2-nitrodiphenylamine and for diphenylamine stabilized propellants containing nitroglycerin.

The test temperature should not exceed 80°C. If necessary a longer test time should be used to obtain the minimum of 20% stabilizer consumption.

Heat the admixture and the control propellant sample in the heating apparatus at the required temperature for 336 hours (14 days). At the end of this time period remove the samples from the heating apparatus and record any visible signs of reaction.

NOTE 10: The test is designed to assess the compatibility of solid materials or liquids of low volatility with solid propellants. The test can be used to assess liquid and gaseous materials and to assess the compatibility of materials with liquid nitrate ester propellants. However in both cases, appropriate modifications to the test apparatus are necessary to ensure it remains leak tight for the duration of the test.

8. ANALYSIS

Determine the residual stabilizer content of the propellant samples by an appropriate analytical method such as HPLC according to procedures specified in AOP-48. They have been found suitable for the analysis of all types of propellants.

If chromatographic techniques are used, an extract of the test material shall be analyzed. Peaks due to extractable components of the test material, that may interfere with the signal from the propellant stabilizer, can then be identified.

Record the results on the data sheet.

NOTE 11: Wherever possible the complete sample shall be analyzed for residual stabilizer content. No attempt should be made to separate the propellant from the test material. The propellant and test material are intimately mixed and obtaining a smaller, but representative sample of propellant, by subdivision of the test sample may be difficult.

9. ACCEPTANCE LIMIT

The decrease in stabilizer content of the propellant in contact with the test material shall not exceed one and a half times the decrease in the stabilizer content of the propellant when heated in the absence of the test material, i.e.

$$D = \frac{A - B}{A - C} \leq 1.5 \qquad \text{(eq. 5.1)}$$

with

A = stabilizer content of the unheated propellant;

B = stabilizer content of the propellant after heating in contact with the test material;

C = stabilizer content of the propellant after heating on its own.

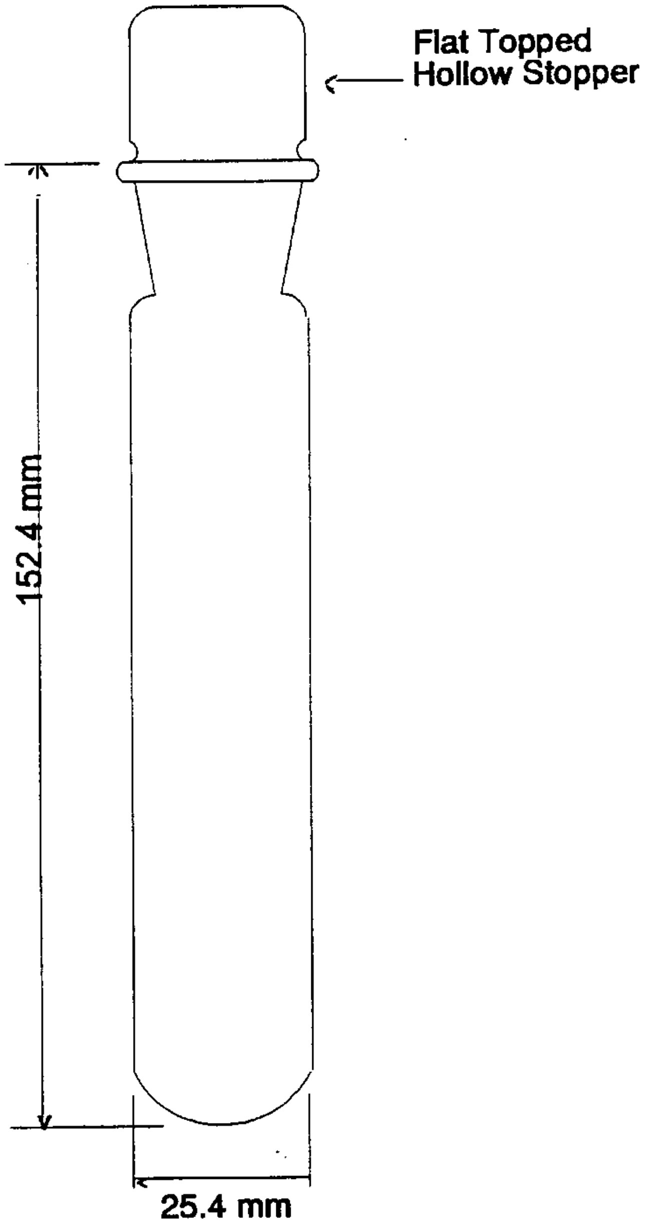

Figure 5.1. Proposed test tube for Test 5 (Ageing of propellant with test material)

TEST 5 - DATA SHEET

(side A see page A-5)

NATO AOP 4147 DATA SHEET (Side B) Report Reference Number: (Unique Reference Number) Page __ of __ Page(s)	
PROPELLANT PREPARATION	**TEST MATERIAL PREPARATION**
TEST CONDITIONS (including divergences from standard tests) Test temperature (°C): Test time (h): Mass of propellant (g): Unheated: Heated: Mass of admixture (g): Mass of propellant: Mass of test material:	**TEST RESULTS** Percentage stabilizer in the unheated propellant sample (A%): Percentage stabilizer in the propellant sample heated with the 10 % addition of test material (B%): Percentage stabilizer in the heated propellant sample (C%):
CALCULATIONS $\frac{A-B}{A-C}=D$ (eq. 5.1)	**ASSESSMENT** COMPATIBLE ☐ YES ☐ NO
DATA SENT TO: (Name and address of person receiving this information)	**COMMENTS**

ANNEX B *DOCUMENT HISTORY*

Technical Note - Development of AOP-4147 (A) (1)

1. Development History

STANAG 4147 Ed.2 [6] was promulgated in 2001. The development of Edition 3 already started in 2003, as a significant need for improvement in sections about heat flow calorimetry (HFC), thermogravimetry (TGA), and differential scanning calorimetry (DSC) had been identified.
Due to the extent of changes from Edition 2 to Edition 3 the main amount of technical discussions has been done by a Custodian Nation Group (CNG) supporting AC/326 SG/1. A compilation of the CNG´s Notes concerning STANAG 4147 [1] is available on the DI portal.
Edition 3 was considered technically complete by AC/326 SG/1 in November 2007. The ratification process started in May 2013 after the French translation was available.
Edition 3 RD 1 [2] has been withdrawn due to inconsistencies in the evaluation of two TGA and DSC tests which were reported by the custodian [4].
Due to changed format requirements of AAP-3 (J) STANAG 4147 Edition 3 has been split into STANAG 4147 Edition 1 and AOP-4147 Edition 1 Version 1.

2. Principal changes STANAG Edition 2 → AOP Edition 1

A principle need for improvement was identified for the following reasons:

- remove detailed test descriptions already covered by stability STANAGs
- availability of additional test equipment (HFC: TAM)
- availability of new test results (like e.g. from the Round Robin Test ‚Compatibility‘ [5])
- change in philosophy for compatibility testing
- assessment of the significance for usual storage conditions for results from Tests 3 and 4

Extensive changes were applied to the general sections about compatibility testing, aiming to give better guidance for users of the STANAG methods.

2.1. Test 2 – HFC

STANAG 4582 deals with HFC for stability testing of nitrocellulose based propellants. For those materials there is vast experience with the test method while other explosives are less often investigated by HFC.

- Matrix of test temperatures and corresponding times has been extended, introducing table 2-1 from STANAG 4582 with times/temperatures equivalent to 10y@25°C (with kinetic parameters for gun propellants)

- Compatibility criterion was changed

  from relative criterion $D = Q_M/Q_{M,calc}$
  to absolute criterion $Q_R=Q_M-Q_{M,calc}$

  Reason: with relative criterion D incompatibility could be overestimated for explosives with high heat flow or underestimated for explosives with low heat flow

- Introduction of additional, informative criteria (heat flow of mixture, additional exothermal effects, anomalous curve shapes) that might help to identify irregularities that don´t show in the integral heat generation for the complete test time

- Introduction of guidelines for criteria based on DSC for other explosives

- Equation generalized for calculation of Q also for mixtures diverging from ratio 1:1 to simplify sample preparation by allowing deviation from exact 1:1 mixture

2.2. Tests 3 & 4 – TGA and DSC

Standard TGA and DSC tests are done using temperature programs with constant heating rates. This gives an advantage for the investigation of very stable explosives as their reactions can be actually observed even if their decomposition temperatures are above 150°C.
Available methods assessing changes in reaction temperatures don´t consider the absolute temperature range in which reactions take place. A shift of 10 degrees to lower temperature certainly has more relevance for storage if it´s from 180 to 170°C compared to 250 to 240°C.
In addition these methods focus on the temperature of DSC or DTGA peaks. Highest decomposition rates are usually reached around 50% of the total decomposition. For negative impacts due to incompatibility reactions, it´s more informative to look at few percent of decomposition.

Therefore two new tests (TGA: Test 3 A-2; DSC: Test 4A-2) have been introduced that

- evaluate the same DSC or TGA tests
- assess interreaction at a degree of decomposition of 10% instead of the peak temperature
- introduce an additional criterion “Storage Temperature for 1% interreaction after 20 years” T_{20y} which allows extrapolation towards relevant temperatures

2.3 Test 5 - Chemical Analysis

Test 5b (Chemical Analysis - assessment of the compatibility of ammunition component materials with lead and silver azide) from edition 2 was deleted. The CNG agreed to delete it, as it was rarely used and had notable differences in methodology compared to all other compatibility tests ([1] Fall Meeting 2005). UK plans to transfer the procedure to the national section in AOP-7.

3 Changes Edition 3 RD1 → Draft Feb. 17

According to the description of inconsistencies in Tests 3A-2 and 4A-2 [4] and as agreed by SG/A EMT [3] the following changes have been applied:

- explicit introduction of parameters W_{rel} and Q_{rel} for interreaction as percent of the reaction step under investigation with equations for their calculation; the parameters where already in RD1, but only implicit by description in the text and equations for their calculation in the data sheets
- correction to use of the exceeding of W_{rel}/Q_{rel} criteria (instead of W_R/Q_R) as precondition for the calculation of the temperature criterion T_{20y}
- subsequent renumbering of the equations in ANNEX D Tests 3 and 4 (eq. 3.2 ff. and eq. 4.2)

4 Changes Ed. 3 Draft Feb. 17 → STANAG 4147 Ed.1/AOP-4147(A) (1) RD1

In order to fulfil the format requirements of AAP-3 (J) the draft document as of February 2017 has been split into a covering STANAG and an AOP using the respective templates. While content and general structure of sections have not been changed, in the course of reformatting the numbering of chapters and annexes was modified:

- Annexes A to C → Chapters 2 to 4
- Annex D → Annex A
- Introduction of new Annex B “Document History”

5 References

[1] AC/326(SG/A)D(2015)0001(PFP)/The Historical Record of the Development of STANAG 4147 Ed.3 (2003-2012)
[2] STANAG 4147 Ed. 3 Ratification Draft 1, 30-05-2013
[3] AC/326(SG/A)DS(2013)0002-REV1 (PFP) Item 6.3.1.2 (SG decision on [4])
[4] Technical Note concerning “Inconsistencies in STANAG 4147 Ed.3 Test 3A-2 and 4A-2 compatibility criteria” (4147_TN_4147_Ed3 .pdf)
[5] Wilker, S., Guillaume, P., “International Round Robin Test on Compatibility”, ICT-Jahrestagung 33, 110 (2002).
[6] STANAG 4147 Ed. 2, 05-06-2001

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