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Committee of Experts on the Transport of Dangerous Goods and on the Globally Harmonized System of Classification and Labelling of Chemicals

Report of the Committee of Experts on the Transport of Dangerous Goods and on the Globally Harmonized System of Classification and Labelling of Chemicals on its fifth session

Held in Geneva on 10 December 2010

Addendum

Annex II

Amendments to the fifth revised edition of the Recommendations on the Transport of Dangerous Goods, Manual of Tests and Criteria (ST/SG/AC.10/11/Rev.5)

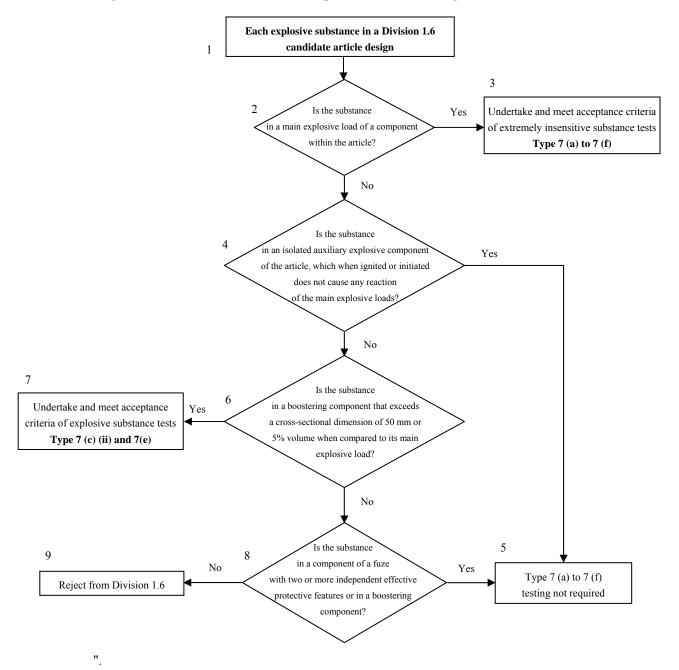


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Section 10

After Figure 10.4, insert the following new Figure 10.5:

"Figure 10.5: Procedure to determine required substance testing for Division 1.6



Consequential amendments:

Renumber Figures 10.5 to 10.9 as Figures 10.6 to 10.10. In 10.5.1, replace "Figures 10.5 to 10.8" with "Figures 10.6 to 10.9". In 10.5.2, replace "Figure 10.9" with "Figure 10.10".

10.4.2.4 Amend the first paragraph to read as follows:

"The question "Is it an extremely insensitive explosive article?" (box 40, Figure 10.3) is answered by series 7 tests and any candidate for Division 1.6 should be assessed against each of the eleven types of test comprising the series. The protocol for determining the test requirements is given in Figure 10.5. The first six types of test (7(a) to 7(f)) are used to establish if a substance is an Extremely Insensitive Substance (EIS). The purpose of these tests is to develop an understanding of the sensitivity of substance(s) contained within the article, which informs and provides confidence in the article tests. The remaining five types of test (7(g), 7(h), 7(j), 7(k) and 7 (l)) are used to determine if an article predominantly containing EIS may be assigned to Division 1.6. The eleven test types are:".

After test "Type 7 (k)", add a new test to read as follows:

"Type 7 (l): a test to determine the sensitivity of an article to shock directed at vulnerable components.".

10.4.3.6 Amend to read as follows:

"10.4.3.6 Tests types 7 (a) to 7 (f) should be used to establish that a substance is an extremely insensitive substance and then test types 7 (g), 7 (h), 7 (j), 7 (k) and 7 (l) used to establish that the articles predominantly containing EIS(s) may be assigned to Division 1.6.".

Add a new paragraph 10.4.3.7 to read as follows:

"10.4.3.7 Tests of types 7 (g), 7 (h), 7 (j), 7 (k) and 7(l) should be performed to determine if an article with EIS main explosive load(s) and appropriately insensitive boostering components may be assigned to Division 1.6. These tests are applied to articles in the condition and form in which they are offered for transport, except that non-explosive components may be omitted or simulated if the competent authority is satisfied that this does not invalidate the results of the tests. The procedure detailing testing requirements is given in Figure 10.5 and some points of explanation are given below.

(a) Complex articles may contain multiple substances and this procedure should be completed for all substances within the article to be classified.

(b) The question "Is the substance in a main explosive load of a component within the article?" (Box 2 of Figure 10.5) is answered by examining the design of the article. Main explosive load substances are those loaded into components within the article that are not fuze, boostering, or isolated auxiliary explosive components. All substances in main explosive loads must "Undertake and meet acceptance criteria of extremely insensitive substance tests, Type 7 (a) to 7 (f)" (Box 3 of Figure 10.5). If a '+' result is obtained for any main explosive load substance to any Type 7 (a) to 7 (f) test, the substance is not an EIS and the answer to the question in Box 24 of Figure 10.3 is "No". The article is not a candidate for Division 1.6.

(c) Answering the question "Is the substance in an isolated auxiliary explosive component of the article, which when ignited or initiated does not cause any reaction of the main explosive loads?" (Box 4 of Figure 10.5) requires knowledge of the design of the article plus the explosive effects that occur when such components are initiated or ignited, either in their design mode or accidentally. Typically these will be small explosive actuators or pyromechanical devices that produce movement, cutting or opening functions. If the answer is 'yes' to this question, Type 7 (a) to 7 (f) testing is not required for substances in isolated auxiliary explosive components and the article remains a candidate for Division 1.6.

(d) The question "Is the substance in a boostering component that exceeds a cross-sectional dimension of 50 mm or 5% volume when compared to its main explosive load?" (Box 6 of Figure 10.5) is answered by examining the design of the article. All substances in such larger boostering components, including those contained in explosive components of dual-protected fuzes in an article, must "Undertake and meet acceptance criteria of explosive substance tests, Type 7 (c) (ii) and 7 (e)" (box 7 of Figure 10.5). If a '+' result is obtained for any such larger boostering component substance to either Type 7 (c) (ii) and 7 (e) tests, the answer to the question in Box 24 of Figure 10.3 is "No". The article is not a candidate for Division 1.6.

(e) The question "Is the substance in a component of a fuze with two or more independent effective protective features or in a boostering component" (Box 8 of Figure 10.5) is answered by an understanding of the design and development of the article. If the answer is 'no', the article is not considered to have suitable intrinsic safety characteristics and the answer to the question in Box 24 of Figure 10.3 is 'No' the article is not a candidate for Division 1.6.

NOTE: Knowledge of the design and explosive effects can be obtained by modelling or indicative tests etc.".

Section 17

17.1 Amend the first paragraph to read as follows:

"The question "Is it an extremely insensitive explosive article?" (box 40 of Figure 10.3) is answered by series 7 tests and any candidate for Division 1.6 should be assessed against each of the eleven types of test comprising the series. The first six types of test (7(a) to 7(f)) are used to establish if a substance is an Extremely Insensitive Substance (EIS) and the remaining five types of test (7 (g), 7 (h), 7 (j), 7 (k) and 7 (l)) are used to determine if an article predominantly containing EIS(s) may be assigned to Division 1.6. The eleven test types are:".

After test "Type 7 (k)", add a new test to read as follows:

- "Type 7 (l): a test to determine the sensitivity of the article to shock directed at vulnerable components.".
- Table 17.1 Replace "EIDS" with "EIS" wherever it appears.

At the end of the table, add a new row to read as follows:

"7 (l) 1.6 article fragment impact test 17.14.1".

17.3 Add a new paragraph 17.3.1 to read as follows:

"17.3.1 All explosive components must always be present in articles during Series 7 testing of types 7 (g) to 7 (l). Smaller explosive components containing substances not subjected to tests of type 7 (a) to 7 (f) shall be specifically targeted in tests 7 (j) and 7 (l) when it is assessed that they will cause the most severe reaction from the test article, to ensure the probability of accidental initiation or propagation of a Division 1.6 article remains negligible.".

Renumber existing 17.3.1 to 17.3.3 as 17.3.2 to 17.3.4.

17.3.1 (renumbered 17.3.2) In the first sentence, replace "use as the explosive load" with "use as a main explosive load". Insert a new second sentence to read as follows: "A substance intended for use as a larger (dimensionally) boostering component in an article of Division 1.6, where the volumetric size limit relative to the main explosive load it is

boostering is met, should be tested in accordance with Test Series 3 and tests of type 7 (c) (ii) and 7 (e).".

17.3.2 (renumbered 17.3.3) Amend to read as follows:

"17.3.3 An article being considered for inclusion in Division 1.6 should not undergo Series 7 testing until after main explosive load and certain boostering component substances have undergone appropriate tests of type 7 (a) to 7 (f) to determine whether they meet the substance requirements for Division 1.6. Guidance on the substance testing determination process is given under section 10.4.3.6.".

17.3.3 (renumbered 17.3.4) Amend the first sentence to read as follows: "Tests of types 7 (g), 7 (h), 7 (j), 7 (k) and 7 (l) should be performed to determine if an article with EIS main load(s) and appropriately insensitive boostering components may be assigned to Division 1.6.".

Insert a new paragraph 17.3.5 to read as follows:

"17.3.5 Response levels referred to within the following individual Test Series 7 test prescriptions are provided at Appendix 8 (Response descriptors), to aid in the assessment of the results of tests of types 7 (g), 7 (h), 7 (j), 7 (k) and 7 (l) and should be reported to the competent authority to support assignment to Division 1.6.".

17.10.1 Amend to read as follows:

"17.10.1 Test 7 (g): 1.6 article (or component level) external fire test".

17.10.1.3 Renumber existing paragraph as 17.10.1.3.1 and add the following new paragraphs:

"17.10.1.3.2 Colour still photographs are taken to document the condition of the test item and the test equipment before and after the test. Explosive substance remains, fragmentation, blast, projections, cratering, witness screen damage, and thrust are documented as an indication of the article's response level.

17.10.1.3.3 Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information.

17.10.1.3.4 To classify complex articles containing multiple EIS main explosive loads, external fire testing at the individual main load component level should be conducted to fully characterise the article's response level.".

17.10.1.4 Replace "If there is a reaction more severe than burning" with "If there is a response level more severe than burning as outlined in Appendix 8".

17.11.1 Amend to read as follows:

"17.11.1 Test 7 (h): 1.6 article or component level slow cook-off test".

17.11.1.3.2 Amend to read as follows:

"17.11.1.3.2 Colour still photographs are taken to document the condition of the test item and the test equipment before and after test. Explosive substance remains, fragmentation, blast, projections, cratering, witness plate damage, and thrust are documented as an indication of the article's response level. Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information.".

17.11.1.3.3 Add a new second sentence to read as follows: "To classify complex articles containing multiple EIS main explosive loads, slow cook-off testing at the individual main load component level should be conducted to fully characterise the article's response level.".

17.11.1.4 Amend the sentence to read as follows: "If there is a response level more severe than burning as outlined in Appendix 8, the result is noted as "+" and the items are not classified as Division 1.6 articles.".

17.12.1 Amend to read as follows:

"17.12.1 Test 7 (j): 1.6 article or component level bullet impact test".

17.12.1.2 to 17.12.1.4 Amend to read as follows:

"17.12.1.2 Apparatus and materials

Three 12.7 mm guns are used to fire service 12.7 mm armour-piercing ammunition with a projectile mass of 0.046 kg. Standard propellant loads may require adjustment to achieve projectile velocities within tolerance. The guns are fired by remote control and protected from fragment damage by firing through a hole in a heavy steel plate. The firing gun muzzles should be at a minimum range of at least 10 m from the test item to assure bullet stabilization prior to impact, and at a maximum range of 30 m from the test item depending upon the explosive weight of the test item. The test item should be secured in a holding device capable of restraining the test item against dislodgement by the projectiles.

17.12.1.3 *Procedure*

17.12.1.3.1 The candidate Division 1.6 article is subjected to a three-round burst fired at 840 ± 40 m/s velocity and 600 rounds/minute rate of fire. The test is repeated in three different orientations, striking the test item in the most vulnerable areas as assessed by the competent authority. These are areas for which an assessment of the explosive sensitivity (explosiveness and sensitiveness) combined with knowledge of the article design indicate the potential producing the most violent response level.

17.12.1.3.2 Colour still photographs are taken to document the condition of the test item and the test equipment before and after the test. Explosive substance remains, fragmentation, blast, projections, cratering, witness plate damage, and thrust are documented as an indication of the article's response level.

17.12.1.3.3 Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information.

17.12.1.3.4 To classify complex articles containing multiple EIS main explosive loads, bullet impact testing at the individual main load component level should be conducted to fully characterise the article's response level.

17.12.1.4 Test criteria and method of assessing results

If there is a response level more severe than burning as outlined in Appendix 8, the result is noted as "+" and the items are not classified as Division 1.6 articles.".

17.13.1.2 Amend to read as follows:

"17.13.1.2 Apparatus and materials

The experimental set-up is the same as for test 6 (b) (see 16.5.1.2), with one trial conducted confined, and another unconfined. The test should only be conducted on detonable candidate Division 1.6 articles; the test 7 (k) article stack test is waived for non-detonable candidates for Division 1.6 (evidence is available to demonstrate that the article cannot support a detonation). Where the article is designed to provide a detonation output, the article's own means of initiation or a stimulus of similar power shall be used to initiate the donor. If the article is not designed to detonate but is capable of supporting a detonation, the donor shall be detonated using an initiation system selected to minimise the influence of its explosive effects on the acceptor $\operatorname{article}(s)$."

17.13.1.3 Amend to read as follows:

"17.13.1.3 *Procedure*

The experimental set-up is the same as for test 6 (b) (see 16.5.1.3). The test is performed twice unless detonation of an acceptor is observed earlier. Colour still photographs are taken to document the condition of the test item and the test equipment before and after the test. Explosive substance remains, fragmentation, blast, projections, cratering, witness plate damage, and thrust are documented and used to assess whether or not any acceptor has detonated (including partially). Blast data may be used to supplement this decision. Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information. Comparing data from the two stack test trials to data from a single donor calibration shot, or to a calculated donor detonation pressure, can be useful in assessing the response level of acceptors."

17.13.1.4 Amend the second sentence to read as follows: "Acceptor article response levels assessed as no reaction, burning, deflagration, or explosion as outlined in Appendix 8 are considered as negative results and noted as "—".".

Add the following new sub-section 17.14:

"17.14 Series 7 type (l) test prescription

17.14.1 Test 7 (l): 1.6 article (or component level) fragment impact test

17.14.1.1 Introduction

This test is used to determine the response of an article in its transport configuration to a localised shock input representative of a fragment strike typical of that produced from a nearby detonating article.

17.14.1.2 Apparatus and materials

To reduce variability due to yaw, a gun system is recommended for firing a standard 18.6 gram steel fragment in the shape of a right-circular cylinder with a conical nose, as detailed in Figure 17.14.1, at a candidate Division 1.6 article. The distance between the firing device and the test item should ensure that the fragment is ballistically stable at impact. Barricades should protect the remote control gun system from the potential damaging effects of the test item's reaction.

17.14.1.3 *Procedure*

17.14.1.3.1 The test is repeated in two different orientations, striking the test item in the most vulnerable areas as assessed by the competent authority. These are areas for which an assessment of the explosive sensitivity (explosiveness and sensitiveness) combined with

knowledge of the article design indicate the potential for producing the most violent response level. Typically, one test would be conducted targeting a non-EIS boostering component and the second test would target the centre of the main explosive load. The orientation of impact should generally be normal to the outer surface of the article. The fragment impact velocity should be 2530 ± 90 m/s.

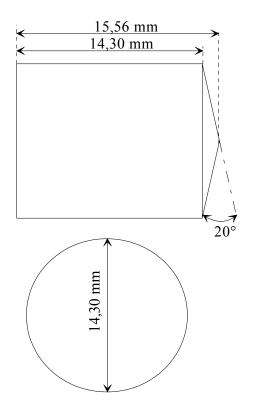
17.14.1.3.2 Colour still photographs are taken to document the condition of the test item and the test equipment before and after the test. Explosive substance remains, fragmentation, blast, projections, cratering, witness plate damage, and thrust are documented as an indication of the article's response level.

17.14.1.3.3 Colour video for the duration of each trial can be vital to assessment of response. In sitting the camera(s), it is important to ensure that the field of view will not be obstructed by any of the test facilities or instrumentation and that the field of view will include all necessary information.

17.14.1.3.4 To classify complex articles containing multiple EIS main explosive loads, fragment impact testing at the individual main load component level should be conducted to fully characterise the article's response level.

17.14.1.4 Test criteria and method of assessing results

If there is a response level more severe than burning as outlined in Appendix 8, the result is noted as "+" and the items are not classified as Division 1.6 articles.



Notes:

Shape:	a conical ended cylinder with the ratio $\frac{L \text{ (length)}}{D \text{ (diameter)}} > 1$ for stability;	
Tolerances:	$\pm 0.05 \text{ mm and } \pm 0^{\circ}30';$	
Fragment mass:	18.6 grams;	
Fragment material:	a mild carbon steel with a Brinell Hardness (HB) less than 270.	

Figure 17.14.1: Standard fragment for 1.6 article fragment impact test

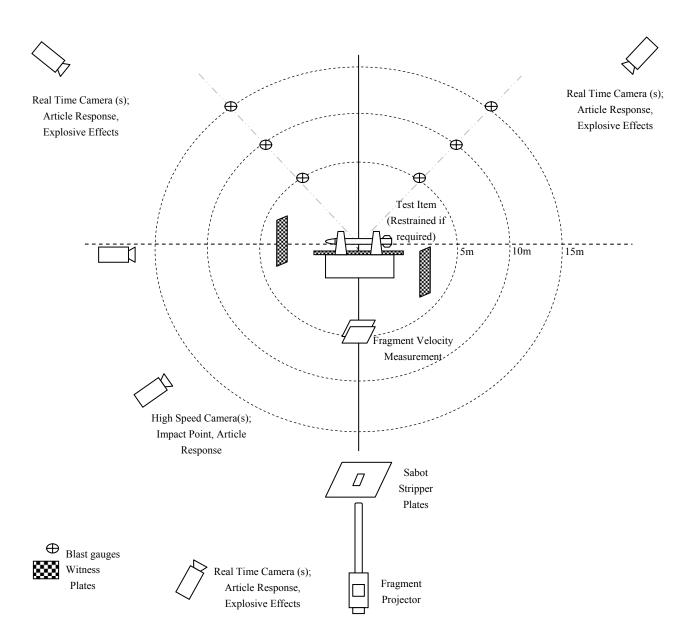


Figure 17.14.2: Typical setup for 1.6 article fragment impact test".

Consequential amendments:

In Table 1.2 and in sub-sections 17.4, 17.5, 17.6, 17.7, 17.8 and 17.9, replace "EIDS" with "EIS" wherever it appears.

Part III

Insert the following new Section 35:

"Section 35 Determination of chemical instability of gases and gas mixtures

35.0 Introduction

This section presents the United Nations scheme for the classification of gases and gas mixtures as chemically unstable. The text should be used in conjunction with the classification principles given in Chapter 2.2 of the Globally Harmonized System of Classification and Labelling of Chemicals (GHS) and the test methods given in this section.

35.1 Purpose

35.1.1 This test method is used to determine the chemical instability of a gas or gas mixture by ignition tests in a closed vessel at ambient and elevated temperature and pressure.

35.1.2 For the purposes of this test method the following definitions apply:

Chemical instability means the propensity of a gas or gas mixture to react dangerously even in the absence of any reaction partner (e.g. air or oxygen) by decomposing and thereby causing a temperature and/or pressure increase;

Test gas means the gas or gas mixture to be evaluated by this test method;

Corresponding initial pressure means the pressure at which the test at 65 °C is carried out. For test gases that are completely gaseous the corresponding initial pressure is the pressure that a gas develops at 65 °C based on the maximum (filling) pressure at ambient temperature. For liquefied test gases the corresponding initial pressure is the vapour pressure at 65 °C.

35.2 Scope

35.2.1 The test method does not cover gas decomposition under process conditions in chemical plants and possible dangerous reactions between different gases in gas mixtures.

35.2.2 Mixtures of gases, where the components can react dangerously with each other, e.g. flammable and oxidizing gases, are not regarded as chemically unstable in the sense of this test method.

35.2.3 If the calculations in accordance with ISO 10156:2010 show that a gas mixture is not flammable it is not necessary to carry out the tests for determining chemical instability for classification purposes.

35.2.4 Expert judgement should be applied to decide whether a flammable gas or gas mixture is a candidate for classification as chemically unstable in order to avoid unnecessary testing of gases where there is no doubt that they are stable. Functional groups indicating chemical instability in gases are triple-bonds, adjacent or conjugated double-bonds, halogenated double-bonds and strained rings.

35.3 Concentration limits

35.3.1 *Generic concentration limits*

35.3.1.1 Gas mixtures containing only one chemically unstable gas are not considered as chemically unstable and therefore do not have to be tested for classification purposes if

the concentration of the chemically unstable gas is below the higher of the following generic concentration limits:

- (a) The lower explosion limit (LEL) of the chemically unstable gas; or
- (b) 3 mole%.

35.3.2 Specific concentration limits

35.3.2.1 The following tables contain information about some gases with regard to their classification as chemically unstable. Specific concentration limits for their mixtures are given. Gas mixtures containing only one chemically unstable gas in concentrations below the specific concentration limit are not considered as chemically unstable and therefore do not have to be tested for classification purposes.

Table 35.1: Information about gases with regard to their chemical instability and concentration limits for their mixtures below which the mixtures are not classified as chemically unstable

	Information	Information about its mixtures			
Chemical Name	Molecular formula	CAS No.	UN No.	Classification	Specific concentration limit (see Notes 1 and 2)
Acetylene	C ₂ H ₂	74-86-2	1001 3374	Chem. Unst. Cat. A	See Table 35.2 For other mixtures: Partial pressure of 1 bar abs
Bromotrifluoro- ethylene	C ₂ BrF ₃	598-73-2	2419	Chem. Unst. Cat. B	8.4 mole% (LEL)
Butadiene-1,2	C ₄ H ₆	590-19-2	1010	Not classified as chemically unstable	
Butadiene-1,3	C ₄ H ₆	106-99-0	1010	Not classified as chemically unstable	
Butyne–1, Ethylacetylene	C ₄ H ₆	107-00-6	2452	Chem. Unst. Cat. B	The specific concentration limits for acetylene may be applied, see Table 35.2. For other mixtures: Partial pressure of 1 bar abs
Chlorotrifluoro- ethylene	C ₂ ClF ₃	79-38-9	1082	Chem. Unst. Cat. B	4.6 mole% (LEL)
Ethylene oxide	C ₂ H ₄ O	75-21-8	1040	Chem. Unst. Cat. A	15 mole% for mixtures containing rare gases. 30 mole% for other mixtures
Vinyl methyl ether	C ₃ H ₆ O	107-25-5	1087	Chem. Unst. Cat. B	3 mole%
Propadiene	C ₃ H ₄	463-49-0	2200	Chem. Unst. Cat. B	The specific concentration limits for acetylene may be applied, see Table 35.2. For other mixtures: Partial pressure of 1 bar abs
Propyne	C ₃ H ₄	74-99-7	3161	Chem. Unst. Cat. B	The specific concentration limits for acetylene may be applied, see Table 35.2. For other mixtures: Partial pressure of 1 bar abs
Tetrafluoroethylene	C_2F_4	116-14-3	1081	Chem. Unst. Cat. B	10.5 mole% (LEL)
Trifluoroethylene	C ₂ HF ₃	359-11-5	1954	Chem. Unst. Cat. B	10.5 mole% (LEL)

	Information	Information about its mixtures			
Chemical NameMolecular formulaCAS No.UN No.Classificati		Classification	Specific concentration limit (see Notes 1 and 2)		
Vinyl bromide	C ₂ H ₃ Br	593-60-2	1085	Chem. Unst. Cat. B	5.6 mole% (LEL)
Vinyl chloride	C ₂ H ₃ Cl	75-01-4	1086	Chem. Unst. Cat. B	3.8 mole% (LEL)
Vinyl fluoride	C ₂ H ₃ F	75-02-5	1860	Chem. Unst. Cat. B	3 mole%

NOTE 1: The maximum pressure should be limited in order to avoid condensation.

NOTE 2: The test method is not applicable to liquefied gas mixtures. In case the gaseous phase above a liquefied gas mixture may become chemically unstable after withdrawal, this shall be communicated via the safety data sheet.

Table 35.2:Specific concentration limits for binary mixtures with acetylene. These
concentration limits may also be applied to butyne-1 (ethylacetylene),
propadiene and propyne

Concentration limit	Maximum (filling) pressure in bar for a mixture with						
for acetylene in mol %	N_2	CO ₂	NH ₃	H_2	CH ₄	C ₃ H ₈	C_2H_4
3.0	200.0				200.0		
4.0	100.0						
5.0				40.0			40.0
6.0	80.0						
8.0	60.0						
10.0	50.0	38.0	5.6	20.0	100.0	6.0	20.0
15.0	30.0	30.0		10.0			10.0
20.0	25.0	20.0	6.2	5.0	50.0	6.6	7.5
25.0	20.0	15.0					5.0
30.0	10.0	10.0	6.9		25.0	7.3	
35.0			7.3				
40.0					15.0	8.2	
45.0							
50.0					5.0	9.3	
60.0						10.8	

35.4 Test method

35.4.1 Introduction

35.4.1.1 The propensity of a gas to decompose depends strongly on pressure, temperature and in case of gas mixtures on the concentration of the chemically unstable component. The possibility of decomposition reactions shall be evaluated at conditions which are relevant for handling and use and transport. Therefore two types of tests shall be performed:

- (a) At ambient temperature and pressure,
- (b) At 65 °C and the corresponding initial pressure.

35.4.2 Apparatus and material

35.4.2.1 The test apparatus (see Figure 35.1) consist of a pressure resistant test vessel (heatable) made of stainless steel; an ignition source; a measuring and recording system to record the pressure inside the ignition vessel; a gas supply; a venting system with bursting disc and additional piping, fitted with remote-controlled valves and cocks.

(a) Pressure resistant test vessel

The test vessel is a cylindrical stainless steel vessel with an inner volume of about 1 dm³ and an inner diameter of 80 mm. An exploding wire ignition source is screwed into the bottom of the vessel. The vessel is equipped with a heating jacket which is connected to a temperature control unit that heats the outer vessel wall with an accuracy of ± 2 K. The test vessel is insulated with temperature resistant insulation material to avoid heat loss and temperature gradients. The test vessel shall be pressure resistant up to 500 bar (50 MPa).

(b) Exploding wire igniter

The ignition source is an exploding (fusing) wire igniter similar to that described in ASTM E 918 and EN 1839. The igniter consists of two insulated electrodes at 3 mm to 6 mm distance, holding a nickeline wire of 0.12 mm diameter at its ends. The ignition energy is provided by a 1.5 kVA/230 (115) V insulating transformer, which is switched for a short time period to this igniter. The wire melts and then an electrical arc burns between the electrodes for a period extending in maximum to half a period of the supply voltage (10 (8.3) ms). An electronic control unit allows switching different time periods of the mains voltage half wave to the igniter. The corresponding energy delivered shall be in the range of 15 J \pm 3 J. The energy can be measured by recording the current and voltage during ignition.

(c) Pressure and temperature recording equipment

The pressure inside the ignition vessel shall be measured with a calibrated piezoresistive pressure transducer. The measuring range shall be 20 times higher than the initial pressure. The sensitivity shall be at least 0.1% of the full scale and the accuracy shall be better than 0.5% of the full scale.

The temperature of the ignition vessel shall be measured and controlled with a 3 mm thermocouple type "K" (NiCr/NiAl) mounted 50 mm below the top inside the autoclave.

After ignition the digitized pressure signal shall be recorded with a computer. The initial pressure (p0) and the highest pressure (pex) are derived from the raw data.

(d) Gas supply

Two different types of gas supply are necessary, one for test gases that are completely in the gaseous phase and one for liquefied test gases. Test gases in the gaseous phase are metered volumetric or by flow measurement and liquefied test gases are metered gravimetric.

(e) Bursting disc

The bursting disc is supposed to protect the test vessel. It is connected to a vent pipe for the exhaust gas. The free diameter of the bursting disc should be at least 10 mm, the inner diameter of the pipe at least 15 mm. The opening pressure of the bursting disc shall amount to 250 bar (25 MPa).

(f) Additional piping and valves

The piping and valves which are fitted directly to the test vessel, shall be pressure resistant up to 500 bar (50 MPa). The test apparatus shall be operated by remotely operated valves.

35.4.3 *Test procedure*

35.4.3.1 The test gas is charged into a pressure resistant stainless steel vessel at controlled temperature and pressure. The vessel is equipped with a bursting disc. Ignition of the test gas is achieved with an exploding wire igniter. Whether a decomposition reaction has occurred is deduced from the pressure rise produced.

35.4.3.2 The tests shall be executed in the following sequence:

(a) Test at ambient temperature and pressure

For the tests at 20 °C and 1.01 bar (101.3 kPa) the exploding wire igniter shall be arranged in the middle of the test vessel. The test vessel and the piping are evacuated. The test gas is filled into the test vessel by using the remotely operated valves until ambient pressure (initial pressure) is reached. After closing the valves the igniter is fired. The ignition energy shall be about 15 J to avoid over-initiation in the test vessel at this relatively low pressure. Criterion for a reaction is a pressure rise of more than 20% after ignition (f = pex/p0 > 1.20). If no such pressure rise has occurred two further re-tests shall be carried out.

If the test gas shows a pressure rise of more than 20% in either of the tests it is to be classified as "chemically unstable at 20 °C and a standard pressure of 101.3 kPa". No further tests are required.

(b) Test at elevated temperature and pressure

If in the tests according to 35.4.3.2 (a) no pressure rise of more than 20% has occurred, further tests at 65 °C and the corresponding initial pressure shall be performed. The procedure is the same as in 35.3.3.2 (a) but care should be taken with potentially unstable gases under pressure. The ignition energy shall be about 15 J. If no pressure rise of more than 20% has occurred two further re-tests shall be carried out.

If the test gas shows a pressure rise of more than 20% in either of the tests it is to be classified as "chemically unstable at a temperature greater than 20 °C and/or a pressure greater than 101.3 kPa".

35.4.4 Safety precautions

35.4.4.1 Adequate shielding of the test apparatus must be provided to prevent injury in the event of equipment rupture. The apparatus is to be set up in such a way that the operator does not have to be in the same room as long as the vessel contains test gas. Alternatively, the test apparatus is separated by a blast proof barrier from the operator. Activation of the ignition source should be possible only from a position shielded from the test vessel.

35.4.4.2 The test vessel shall be fitted with a bursting disc connected to a vent pipe that allows venting the exhaust gas safely. Therefore it has to be taken into account that the exhaust gas can be hazardous itself (e.g. flammable or toxic).

35.4.4.3 The gas cylinder containing the test gas shall be equipped with a non-return valve and shall be separated from the test apparatus before the igniter is fired to avoid backfiring into the cylinder. The cylinder valve has to be closed immediately after finishing the filling.

35.4.4.4 Some chemically unstable gases can explode very violently especially at higher pressures. Therefore it is strongly recommended to start with the experiments at atmospheric pressure.

35.4.5 Test criteria and method of assessing results

35.4.5.1 Chemically unstable gases or gas mixtures shall be classified as "chemically unstable at 20 °C and a standard pressure of 101.3 kPa" or "chemically unstable at a temperature greater than 20 °C and/or a pressure greater than 101.3 kPa" according to the test results as follows:

(a) The gas is classified as "chemically unstable at 20 °C and a standard pressure of 101.3 kPa" if the test at 20 °C and 1.01 bar (101.3 kPa) shows a pressure rise of more than 20% of the initial absolute pressure.

(b) The gas is classified as "chemically unstable at a temperature greater than 20 °C and/or a pressure greater than 101.3 kPa" if the test at 65 °C and the corresponding initial pressure shows a pressure rise of more than 20% of the initial absolute pressure but no such pressure rise at 20 °C and 1.01 bar (101.3 kPa).

35.4.5.2 The gas is not classified according to this test method (i.e. it is chemically stable) if it does not show a pressure rise of more than 20% of the initial absolute pressure in either of the tests.

NOTE: Chemically unstable gases not submitted to the classification procedure in this section should be classified as chemically unstable, Category A (see Chapter 2.2 of the GHS).

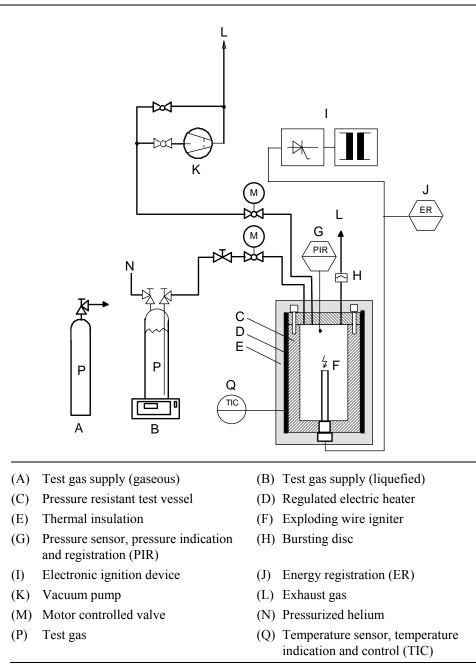


Figure 35.1: Test apparatus".

Section 38

38.3.2 Insert a new paragraph 38.3.2.1 to read as follows:

"38.3.2.1 All cell types shall be subjected to tests T.1 to T.6 and T.8. All nonrechargeable battery types, including those composed of previously tested cells, shall be subjected to tests T.1 to T.5. All rechargeable battery types, including those composed of previously tested cells, shall be subjected to tests T.1 to T.5 and T.7. In addition, rechargeable single cell batteries with overcharge protection shall be subjected to test T.7. A component cell that is not transported separately from the battery it is part of needs only to be tested according to tests T.6 and T.8. A component cell that is transported separately from the battery shall be tested as a cell.".

Renumber existing paragraph 38.3.2.1 as 38.3.2.2.

38.3.2.1 (renumbered 38.3.2.2) In sub-paragraph (b), insert "in nominal energy" before "in Watt-hours" and "nominal" before "voltage". Amend sub-paragraph (c) to read as follows:

"(c) A change that would lead to failure of any of the tests,".

Before the last paragraph reading "In the event that...", insert the following new Note:

"**NOTE**: The type of change that might be considered to differ from a tested type, such that it might lead to failure of any of the test results, may include, but is not limited to:

(a) A change in the material of the anode, the cathode, the separator or the electrolyte;

- (b) A change of protective devices, including hardware and software;
- (c) A change of safety design in cells or batteries, such as a venting valve;
- (d) A change in the number of component cells; and
- (e) A change in connecting mode of component cells.".

After the last paragraph reading "In the event that...", insert the following new Note:

"**NOTE**: Batteries are subject to the tests required by special provisions 188 and 230 of Chapter 3.3 of the Model Regulations irrespective of whether the cells of which they are composed have been so tested.".

Renumber existing paragraph 38.3.2.2 as 38.3.2.3.

38.3.2.2 (renumbered 38.3.2.3) In the definition of "*Battery*", amend the first paragraph to read as follows:

"*Battery* means two or more cells which are electrically connected together and fitted with devices necessary for use, for example, case, terminals, marking and protective devices. A single cell battery is considered a "cell" and shall be tested according to the testing requirements for "cells" for the purposes of the Model Regulations and this Manual (see also the definition for "cell")."

The Note remains unchanged.

Amend the definition of "Large cell" to read as follows:

"Large cell means a cell with a gross mass of more than 500 g.".

Amend the definition of "Leakage" to read as follows:

"*Leakage* means the visible escape of electrolyte or other material from a cell or battery or the loss of material (except battery casing, handling devices or labels) from a cell or battery such that the loss of mass exceeds the values in Table 1.".

Amend the definition of "Mass loss" to read as follows:

"Mass loss means a loss of mass that exceeds the values in Table 1 below.

Mass <i>M</i> of cell or battery	Mass loss limit
M < 1 g	0.5%
$l g \le M \le 75 g$	0.2%
M > 75 g	0.1%

Table 1: Mass loss limit

NOTE: In order to quantify the mass loss, the following procedure is provided:

Mass loss (%) =
$$\frac{(M_1 - M_2)}{M_1} \times 100$$

where M_1 is the mass before the test and M_2 is the mass after the test. When mass loss does not exceed the values in Table 1, it shall be considered as "no mass loss".".

In the definition of "Primary", insert "cell or battery" after "Primary".

Amend the definition of "Rated capacity" to read as follows:

"*Rated capacity* means the capacity, in ampere-hours or milliampere-hours, of a cell or battery as measured by subjecting it to a load, temperature and voltage cut-off point specified by the manufacturer.

NOTE: The following IEC standards provide guidance and methodology for determining the rated capacity:

(1) IEC 61960 (First Edition 2003-12): Secondary cells and batteries containing alkaline or other non-acid electrolytes – Secondary lithium cells and batteries for portable applications;

(2) IEC 62133 (First Edition 2002-10): Secondary cells and batteries containing alkaline or other non-acid electrolytes – Safety requirements for portable sealed secondary cells, and for batteries made from them, for use in portable applications;

(3) IEC 62660-1 (First Edition 2011-01): Secondary lithium-ion cells for the propulsion of electric road vehicles – Part 1: Performance testing.".

In the definition of "Rechargeable", insert "cell or battery" after "Rechargeable".

Amend the definition of "Small cell" to read as follows:

"Small cell means a cell with a gross mass of not more than 500 g.".

Insert the following new definitions:

"Fire means that flames are emitted from the test cell or battery.".

"*Nominal energy in watt-hours* means the energy value of a cell or battery determined under specified conditions and declared by the manufacturer. The nominal energy is calculated by multiplying nominal voltage by rated capacity.".

"*Nominal voltage* means the approximate value of the voltage used to designate or identify a cell or battery.".

"*Open circuit voltage* means the voltage across the terminals of a cell or battery when no external current is flowing.".

"*Single cell battery* means a single electrochemical unit fitted with devices necessary for use, for example, case, terminals, marking and protective devices.".

38.3.3 In sub-paragraphs (a) and (b), first sentence, replace "tests 1 to 5" with "tests T.1 to T.5".

In sub-paragraph (c), first sentence, replace "test 6" with "test T.6". Delete the last paragraph after sub-paragraph (iv).

In sub-paragraph (d), first sentence, replace "rechargeable batteries under test 7" with "rechargeable batteries or rechargeable single cell batteries under test T.7".

Amend sub-paragraph (e) to read as follows:

"(e) When testing primary and rechargeable cells and component cells under test T.8, the following shall be tested in the quantity indicated:

- (i) ten primary cells in fully discharged states;
- (ii) ten primary component cells in fully discharged states;
- (iii) ten rechargeable cells, at first cycle in fully discharged states;
- (iv) ten rechargeable component cells, at first cycle in fully discharged states;
- (v) ten rechargeable cells after 50 cycles ending in fully discharged states; and
- (vi) ten rechargeable component cells after 50 cycles ending in fully discharged states.".

In sub-paragraph (f), first sentence, delete "cells or", replace "tests 3, 4 and 5" with "tests T.3, T.4 and T.5" and replace "test 7" with "test T.7".

38.3.4 Delete the first sentence. Replace "Tests 1 to 5" with "Tests T.1 to T.5" (twice), "Tests 6 and 8" with "Tests T.6 and T.8" and "Test 7" with "Test T.7".

38.3.4.1.3, 38.3.4.2.3 and 38.3.4.4.3 Delete "no mass loss,".

38.3.4.2.2 In the first sentence, replace " 75 ± 2 °C" with " 72 ± 2 °C". In the third sentence, replace "10 times" with "until 10 total cycles are complete".

38.3.4.3.2 Amend the last paragraph to read as follows:

"The logarithmic frequency sweep shall differ for cells and batteries up to 12 kg (cells and small batteries), and for batteries 12 kg and greater (large batteries).

For cells and small batteries: from 7 Hz a peak acceleration of $1 g_n$ is maintained until 18 Hz is reached. The amplitude is then maintained at 0.8 mm (1 6 mm total excursion) and the frequency increased until a peak acceleration of 8 g_n occurs (approximately 50 Hz). A peak acceleration of 8 g_n is then maintained until the frequency is increased to 200 Hz.

For large batteries: from 7 Hz to a peak acceleration of 1 g_n is maintained until 18 Hz is reached. The amplitude is then maintained at 0.8 mm (1.6 mm total excursion) and the frequency increased until a peak acceleration of 2 g_n occurs (approximately 25 Hz). A peak acceleration of 2 g_n is then maintained until the frequency is increased to 200 Hz.".

38.3.4.3.3 Amend the first sentence to read as follows: "Cells and batteries meet this requirement if there is no leakage, no venting, no disassembly, no rupture and no fire during the test and after the test and if the open circuit voltage of each test cell or battery directly after testing in its third perpendicular mounting position is not less than 90% of its voltage immediately prior to this procedure.".

38.3.4.5.2 Delete the last sentence.

38.3.4.5.3 Replace "within six hours of this test" with "during the test and within six hours after the test".

38.3.4.6 Amend to read as follows:

"38.3.4.6 Test T.6: Impact / Crush

38.3.4.6.1 Purpose

These tests simulate mechanical abuse from an impact or crush that may result in an internal short circuit.

38.3.4.6.2 Test procedure – Impact (applicable to cylindrical cells greater than 20 mm in diameter)

The sample cell or component cell is to be placed on a flat smooth surface. A 15.8 mm \pm 0.1mm diameter, at least 6 cm long, or the longest dimension of the cell, whichever is greater, Type 316 stainless steel bar is to be placed across the centre of the sample. A 9.1 kg \pm 0.1 kg mass is to be dropped from a height of 61 \pm 2.5 cm at the intersection of the bar and sample in a controlled manner using a near frictionless, vertical sliding track or channel with minimal drag on the falling mass. The vertical track or channel used to guide the falling mass shall be oriented 90 degrees from the horizontal supporting surface.

The test sample is to be impacted with its longitudinal axis parallel to the flat surface and perpendicular to the longitudinal axis of the 15.8 mm \pm 0.1mm diameter curved surface lying across the centre of the test sample. Each sample is to be subjected to only a single impact.

38.3.4.6.3 Test Procedure – Crush (applicable to prismatic, pouch, coin/button cells and cylindrical cells not more than 20 mm in diameter)

A cell or component cell is to be crushed between two flat surfaces. The crushing is to be gradual with a speed of approximately 1.5 cm/s at the first point of contact. The crushing is to be continued until the first of the three options below is reached.

1. The applied force reaches 13 kN \pm 0.78 kN;

Example: The force shall be applied by a hydraulic ram with a 32 mm diameter piston until a pressure of 17 MPa is reached on the hydraulic ram.

- 2. The voltage of the cell drops by at least 100 mV; or
- 3. The cell is deformed by 50% or more of its original thickness.

Once the maximum pressure has been obtained, the voltage drops by 100 mV or more, or the cell is deformed by at least 50% of its original thickness, the pressure shall be released.

A prismatic or pouch cell shall be crushed by applying the force to the widest side. A button/coin cell shall be crushed by applying the force on its flat surfaces. For cylindrical cells, the crush force shall be applied perpendicular to the longitudinal axis.

Each test cell or component cell is to be subjected to one crush only. The test sample shall be observed for a further 6 h. The test shall be conducted using test cells or component cells that have not previously been subjected to other tests.

38.3.4 6.4 Requirement

Cells and component cells meet this requirement if their external temperature does not exceed 170 °C and there is no disassembly and no fire during the test and within six hours after this test.".

38.3.4.7.3 and 38.3.4.8.3 Replace "within seven days of the test" with "during the test and within seven days after the test".

Section 41

41.2.2 Amend to read as follows:

"41.2.2 *MEGCs*

(a) A decrease in the maximum design temperature, not affecting thickness;

(b) An increase in the minimum design temperature, not affecting thickness;

(c) A decrease in the maximum permissible gross mass;

(d) A decrease in the mass of each individual element and its lading or a decrease in the total mass of the elements and their lading;

(e) An increase of no more than 10% or a decrease of no more than 40% in the diameter of the elements;

(f) A change of no more than 10% in the length of the elements;

(g) A decrease of no more than 3.1 metres (10 feet) in the length of the MEGC framework;

(h) A decrease of no more than 50% in the height of the MEGC;

(i) A change of no more than 50% in the number of elements;

(j) An increase in the thickness of the materials of the framework provided the thickness stays within the range permitted by the welding procedure specifications;

(k) A change to the service equipment and manifold such that the total mass of the service equipment and manifold changes no more than 10% of the maximum permissible gross mass (but not resulting in an increase in the maximum permissible gross mass as compared to that of the already-tested prototype);

(l) The use of a different grade of the same type of material for the construction of the framework, provided that:

- (i) The results of the design calculations for the different grade, using the most unfavourable specified values of mechanical properties for that grade, meet or exceed the results of the design calculation for the existing grade; and
- (ii) The alternate grade is permitted by the welding procedure specifications.".

NOTE: For permitted MEGC design variations not requiring additional impact testing, the mounting apparatus attaching the elements to the framework must remain the same as that for the already-tested prototype MEGC design.

Appendices

Add a new Appendix 8 to read as follows:

"Appendix 8

Response descriptors

These response descriptors are to be used for the purposes of Test Series 7 criteria and designed to be used by the competent authority to determine the response type of articles. For example, articles vary greatly in size, type, packaging and explosive substances; these differences need to be taken into account. For a reaction to be judged a particular type, the primary evidence (denoted P in the table below) for that type would need to be present. The entire (both primary and secondary) body of evidence must be weighed carefully and used in its entirety by the competent authority to assess the reaction. The secondary evidence provides other indicators that may be present.

	Observed or measured effects								
Response level	Explosive Substances (ES)	Case	Blast	Fragment or ES projection	Other				
Detonation	Prompt consumption of all ES once the reaction starts	(P) Rapid plastic deformation of the metal casing contacting the ES with extensive high shear rate fragmentation	(P) Shock wave with magnitude & timescale = to a calculated value or measured value from a calibration test	Perforation, fragmentation and/or plastic deformation of witness plates	Ground craters of a size corresponding to the amount of ES in the article				
Partial detonation		(P) Rapid plastic deformation of some, but not all, of the metal casing contacting the ES with extensive high shear rate fragmentation	(P) Shock wave with magnitude & timescale < that of a calculated value or measured value from a calibration test Damage to neighboring structures	Perforation, plastic deformation and/or fragmentation of adjacent witness plates. Scattered burned or unburned ES.	Ground craters of a size corresponding to the amount of ES that detonated.				
Explosion	(P) Rapid combustion of some or all of the ES once the article reaction starts	(P) Extensive fracture of metal casings with no evidence of high shear rate fragmentation resulting in larger and fewer fragments than observed from purposely detonated calibration tests ¤	Observation or measurement of a pressure wave throughout the test arena with peak magnitude << and significantly longer duration that of a measured value from a calibration test	Witness plate damage. Significant long distance scattering of burning or unburned ES.	Ground craters.				
Deflagration	(P) Combustion of some or all of the ES	(P) Rupture of casings resulting in a few large pieces that might include enclosures or attachments. *	Some evidence of pressure in the test arena which may vary in time or space.	(P) At least one piece (casing, enclosure or attachment) travels beyond 15m with an energy level > 20J based on the distance/mass relationship of Figure 16.6.1.1. Significant scattered burning or unburned ES, generally beyond 15 m.	 (P) There is no primary evidence of a more severe reaction and there is evidence of thrust capable of propelling the article beyond 15m. Longer reaction time than would be expected in an explosion reaction. 				

D			Observed or measur	red effects	
Response level	Explosive Substances(ES)	Case	Blast	Fragment or ES projection	Other
Burn	(P) Low pressure burn of some or all of the ES	(P) The casing may rupture resulting in a few large pieces that might include enclosures or attachments. *	Some evidence of insignificant pressure in the test arena.	 (P) No item (casing, enclosure, attachment or ES) travels beyond 15m with an energy level > 20J based on the distance/mass relationship detailed at Figure 16.6.1.1. (P) A small amount of burning or unburned ES relative to the total amount in the article may be scattered, generally within 15m but no farther than 30m. 	(P) No evidence of thrust capable of propelling the article beyond 15m. For a rocket motor a significantly longer reaction time than if initiated in its design mode.
No Reaction	 (P) No reaction of the ES without a continued external stimulus. (P) Recovery of all or most of the unreacted ES with no indication of a sustained combustion. 	(P) No fragmentation of the casing or packaging greater than that from a comparable inert test item. *	None	None	None

* Note: Mechanical threats will directly induce damage causing disruption of the article or even a pneumatic response resulting in parts, particularly closures, being projected. This evidence can be misinterpreted as being driven by the reaction of the explosive substance contained in the article, which may result in a more severe response descriptor being assigned. Comparison of observed evidence with that of a corresponding inert article can be useful in helping to determine the article's response.".