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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

Sub-Committee of Experts on the Transport of Dangerous Goods

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Item 3 (b) of the provisional agenda

EXPLOSIVES, SELF-REACTIVE SUBSTANCES AND ORGANIC PEROXIDES

Ammonium nitrate emulsions

Test Series 8(d)

Proposal for the adoption of a "Modified" Vented Pipe Test as the optional Test Series 8(d)

Transmitted by the expert from Sweden

Introduction

Various options for a Series 8(d) Vented Pipe Test were discussed by the ANE Working Group at the UN Sub-Committee of Experts on the Transport of Dangerous Goods (UNSCETDG), in Geneva, July 2002 [1]. This test is intended to examine the response of ammonium nitrate based emulsions and suspensions to prolonged heating in a large scale test. Both the Scandinavians [2, 3] and the South Africans [4] presented their results from wood-fired tests, recommending that such wood-fired tests not be adopted as part of a classification method. The Australians [5] presented test results from a modified procedure using a gas burner as the heat source, calibrated to standardise the effects. The Working Group decided that this Modified Vented Pipe Test (MVPT) [6] should be developed further over the next biennium for consideration at a later date. In the mean time, the existing wood-fired version of the test was to be kept as the discretionary Series 8(d) test in the current Manual of Tests and Criteria [7].

Since then, an extensive series of tests has been performed in Spain using the MVPT procedure on a variety of suspensions and emulsion compositions [8, 9 and 10]. Further testing in Australia has yielded MVPT results for a variety of proprietary emulsion compositions, and for variety of ammonium nitrate based suspensions [11].

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A review [12] was performed of all 87 MVPT trials conducted prior to September 2003 in Australia and in Spain on a variety of compositions including pure ammonium nitrate (AN) and AN-based emulsions and suspensions, and the results presented to the ANE Working Group in Geneva in December 2003.
There are four key phases in the establishment of a classification test — firstly the development of a robust test procedure, secondly the identification of the physical mechanisms controlling the test outcomes, thirdly an assessment of how those mechanisms might lead to the development of potential hazards during transport, and fourthly, the assignment of test criteria to discriminate between acceptable and unacceptable levels of hazard during transport. Each of these phases will be discussed in turn below.

(1) The recommended test procedure for the MVPT is given in the Annex. The procedure appears to be robust, with the test apparatus having been assembled in two different countries and giving reproducible results as exemplified by similar heating conditions, similar thermocouple traces and similar observed behaviours over a range of compositions. Corrections can be made for differences in heating rate and sample initial temperature. The heating conditions were found to be realistic, with the constant heat flux into the MVPT vessel [5] exceeding the peak heat flux measured during experimental tests of pool fire engulfment of LPG tanks [13], and the heated surface area to volume ratio matching that of the Kuosanen full-scale burning test [14].

(2) The review of the MVPT trials [12] described how the thermocouple traces and video records from the trials could be analysed jointly to give considerable insight into the physical mechanisms occurring during the breakdown, boiling, concentration and decomposition of AN-based emulsions and suspensions.

(3) The review [12] also discussed the relevance of these mechanisms to the development of potential hazards during transport. For example, the probable emission of toxic fume could be inferred from the measured temperature histories together with published decomposition schemes for AN [15], while the shock sensitivity of the heated ANE at the time of vessel overflow could be inferred knowing the average density at that time and the measured shock sensitivity of molten AN [16]. Several of these hazards have been identified only by inference at this stage, and have not been verified by direct experimental evidence. This reflects the current state of incomplete knowledge, and suggests directions for future research.

(4) In many ways, the criteria are the most difficult part of a classification test to define, especially when not all of the potential hazards have been demonstrated experimentally. The review [12] discussed how a time-based criterion could accommodate the uncertainty in the development of potential hazards during the degradation of AN-based emulsions and suspensions following prolonged heating.

Proposal

It is proposed that the Modified Vented Pipe Test as detailed in the Annex be adopted as a replacement for Test Series 8d. It is further recommended that it should be a discretionary test.

The recommended criteria to be used in interpreting the results of the MVPT are also given in the Annex. Separate criteria are defined to support the existing UN No. 3375 for ammonium nitrate emulsions, suspensions and gels and the proposed new UN No. 3XXX for chemically sensitised emulsions, suspensions and gels. In summary, the criteria are as follows:

For UN No. 3XXX the criteria for acceptance for transport in bulk are as follows:

- If the vessel is vented within a time less than the “run time for venting” (as defined in the Annex, Clause 4.6) or the vessel is ruptured within a time less than the “run time for rupture” (as defined in the Annex, Clause 4.6) the outcome is “positive” and the test substance is not suitable for transport in bulk as Class 5.1. The substance should be considered for transport in bulk only as Class 1.5.

- If the vessel is vented within a time greater than the “run time for venting”, and the vessel is not ruptured within a time less than the “run time for rupture” the outcome is “negative” and the test substance is suitable for transport in bulk as Class 5.1.

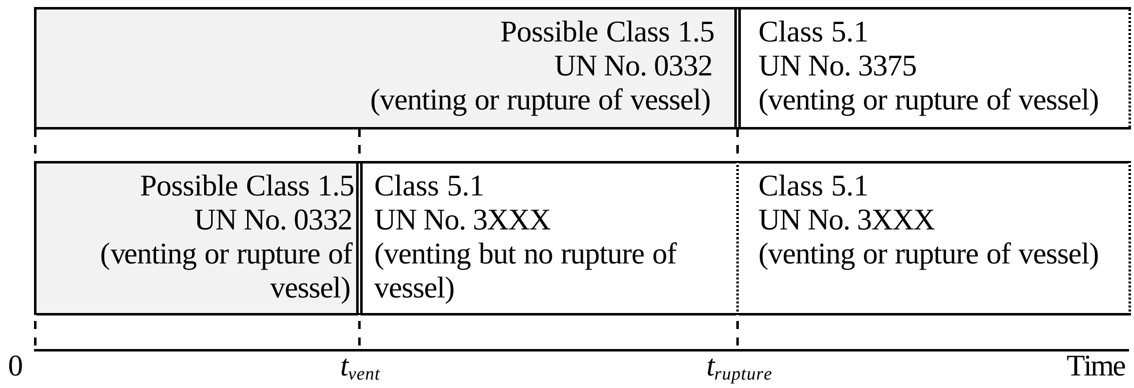
For UN No. 3375 the criteria for acceptance for transport in bulk are as follows:

- If the vessel is vented or ruptured within a time less than the “run time for rupture” the outcome is “positive” and the test substance is not suitable for transport in bulk as Class 5.1. The substance should be considered for transport in bulk only as Class 1.5.
- If the vessel is not vented or ruptured within a time greater than the “run time for rupture” the outcome is “negative” and the test substance is suitable for transport in bulk as Class 5.1.

A summary of the criteria is given below:

	UN No. 3XXX	UN No. 3375
Definition	Must meet SP 3ZZ	Must meet SP 309
Test 8a	Negative	Negative
Test 8b	Negative	Negative
Test 8c	Negative	Negative
Test 8d (*discretionary*)	$t_{conclusion} > t_{venting}$ (without rupture prior to $t_{rupture}$)	$t_{conclusion} > t_{rupture}$

A schematic for the time criteria is given below illustrating the possible outcomes of the MVPT. While the actual times depend on corrections for the heating rate and for any difference between the maximum shipping temperature and the actual temperature at which the sample was tested, the “run time for venting” will typically be about 30 minutes, and the “run time for rupture” typically about 60 minutes.



The proposed procedure for the MVPT given in the Annex includes three parameters enclosed in square brackets, namely the vent diameter in Clause 2(a), and the multipliers for the two run-times defined in Clause 4.6. The final values of these parameters should be set by consensus after suitable discussion by the UNSCETDG.

Safety implications
Enhanced safety

Feasibility
No problems are foreseen

Enforceability
No problems are foreseen
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Annex

Recommended procedure

EXPLOSIVES, SELF-REACTIVE SUBSTANCES AND ORGANIC PEROXIDES.

Classification of Ammonium Nitrate Emulsions, Suspensions and Gels for UN No. 3375 and UN No. 3XXX.

Manual of Tests and Criteria. Test 8(d) – Modified Vented Pipe Test

1 Introduction

The modified vented pipe test is used to assess the effect of exposure of a candidate for "ammonium nitrate emulsion or suspension or gel, intermediate for blasting explosives" under either UN No. 3375 or UN No. 3XXX to a large fire under confined, vented conditions. It will determine whether or not the candidate can be transported in tanks as a dangerous good of Class 5.1.

The candidate is subjected to this test only after it has given negative outcomes during Test 8(a), Test 8(b) and Test 8(c) and has been deemed by those tests to be a dangerous good of Class 5.1.

The same test procedure is applied to candidates for both UN No. 3375 and UN No. 3XXX, but different acceptance criteria are applied to each to differentiate between the two.

2 Apparatus and materials

The following items are needed:

(a) A vented vessel consisting of mild drawn steel pipe with an inner diameter of 265 ± 10 mm, a length of 580 ± 10 mm and a wall thickness of 5.0 ± 0.5 mm. Both the top and the base plates are made from 300 mm square, 6.0 ± 0.5 mm thick mild steel plates. The top and base plates are fixed to the pipe with a single 5 mm fillet weld. The top plate has a vent diameter of [87] ± 1 mm. A minimum of three, and preferably up to five, small holes are drilled in the top plate to accommodate neatly thermocouple probes, these holes being equally spaced around a circle of radius 90 ± 5 mm concentric with the vent;

(b) A concrete block about 400 mm square and 50 to 75 mm thick;

(c) A metal stand for supporting the vessel at a height of 150 mm above the concrete block;

(d) A gas burner capable of accommodating an LPG flow rate of up to 35 ± 2 litres per minute. This rests on the concrete block under the stand. A typical example of a suitable burner is a 32-jet Mongolian wok burner;

(e) A sheet metal shield to protect the LPG flame from side winds. This can be fabricated from approximately 0.5 mm thick galvanised sheet metal. The diameter of the wind shield is 600 mm and the height is 250 mm. Four adjustable vents 150 mm wide and 100 mm high are spaced equally around the shield to ensure adequate air reaches the gas flame;

(f) LPG bottle(s) connected via a manifold and fed into a pressure regulator. The pressure regulator should reduce the LPG bottle pressure from 650 kPa down to about 250 kPa. The gas then flows through a gas rotameter capable of measuring up to 40 litres per minute of LPG gas. An electrical solenoid valve is used to switch the LPG flow on and off remotely. Typically four 9kg LPG bottles will achieve the desired gas flow rate for the duration of up to five tests;

(g) One thermocouple with 600 mm long stainless steel probe and a minimum of three, though preferably five, thermocouples with 500 mm long stainless steel probes and fibre-glass coated lead wires;

(h) A data-logger capable of recording the output from the thermocouples;

(i) Cine or video cameras, preferably high speed and normal speed, to record events in colour;

(j) Pure water for calibration;

(k) The candidate to be tested;

(l) Blast gauges, radiometers and associated recording equipment may also be used.

3 Calibration

3.1 The vessel is filled to the 75% level (i.e. to a depth of 435 ± 10 mm) with the pure water, and heated using the procedure specified in Section 4. The time taken to reach the boiling point of the water is recorded, and is used to define the "calibration-time" for the given combination of vessel and heat source. The boiling point has been reached when all thermocouple traces converge at approximately 100 °C (or lower if at altitude).

3.2 If the initial temperature of the water was not 25 °C, corrections to the calibration-time must be made based on the measured mean heating rate, $\dot{T}_{cal}$, over the temperature interval between 40 °C and 80 °C as recorded by the thermocouple T3 in the water. An example of the required correction is given in Section 6.1.

3.3 The calibration-time, t_{cal}, is defined as the corrected time taken to heat water from 25 °C to boiling point within the test equipment, and must be 24 minutes ± 2 minutes 30 seconds. If t_{cal} lies outside this time window, the calibration must be repeated from Step 3.1 with a fresh sample of water making appropriate adjustments to the gas flow. Appropriate values for the pressure and the flow rate at which to conduct the first water calibration procedure are 250 kPa and 35 litres per minute respectively. If sufficient adjustment is not available from the gas flow, it may be necessary to alter the height of the metal stand supporting the vessel above the gas burner.

3.4 This calibration must be performed prior to the testing of any candidate substance. The same calibration-time, t_{cal}, and mean heating rate, $\dot{T}_{cal}$, can be applied to any test conducted within a week of the calibration provided no change is made to the vessel construction, LPG burner type or gas supply.

4 Test Procedure

4.1 The concrete block is placed on a sandy base and levelled using a spirit level. The LPG burner is positioned in the centre of the concrete block and connected to the gas supply line. The metal stand is placed over the burner.

4.2 The vessel is placed vertically on the stand and secured from tipping over. The vessel is filled to 75% of its volume (to a height of 435 ± 10 mm) with the candidate under test without tamping during loading. The initial temperature of the candidate must be recorded. The substance is carefully packed to prevent adding voids. The wind shield is positioned around the base of the assembly to protect the LPG flame from heat dissipation due to side winds.

4.3 The thermocouple positions are:

- the first 500 mm long probe (T1) in the LPG flame;
- the second 500 mm long probe (T2) in the headspace 20 ± 5 mm into the vessel;
- the third 500 mm long probe (T3) in the sample 175 ± 5 mm into the vessel;

- the 600 mm long probe (T4) in the sample 570 ± 5 mm into the vessel;

If used, the extra two thermocouples are placed

- the fourth 500 mm long probe (T5) in the sample 360 ± 5 mm into the vessel;
- the fifth 500 mm long probe (T6) in the headspace 100 ± 5 mm into the vessel.

The thermocouples are connected to the data-logger and the thermocouple leads and data-logger are adequately protected from the test apparatus in case of explosion.

4.4 LPG pressure and flow rate are checked and adjusted to the values used during the water calibration described in Section 3. Video cameras and any other recording equipment are checked and started. Thermocouple functioning is checked and data logging is started, with a time step between thermocouple readings not exceeding 20 seconds, and preferably shorter. The test should not be performed under conditions where the wind speed exceeds 6 m/s.

4.5 The LPG burner may be started locally or remotely and all workers immediately retreat to a safe location. Progress of the test is followed by monitoring thermocouple readings and closed circuit television images. The start time of the trial is defined by the time at which the flame thermocouple trace T1 first begins to rise.

4.6 The "run-time for rupture" $t_{rupture}$ for both UN3375 and UN3XXX candidate is calculated as [2.8] times the calibration-time t_{cal} for water, adjusted by a suitable correction based on the measured heating rate $\dot{T}_{cal}$ for water if the initial temperature of the candidate is below the normal shipping temperature. The correction procedure is illustrated in Section 6. The candidate should not be tested at an initial temperature above the normal range of shipping temperatures.

For a UN3XXX candidate only, the "run-time for venting" t_{vent} is calculated as [1.4] times the calibration-time t_{cal} for water, adjusted by a suitable correction based on the measured heating rate $\dot{T}_{cal}$ for water if the initial temperature of the candidate is below the normal shipping temperature.

4.7 The candidate is heated for its "run-time for rupture" $t_{rupture}$ or longer, unless it reaches an earlier conclusion according to Section 4.9. At the end of this run-time, or earlier if the test is deemed to have reached its conclusion, the LPG supply may at the discretion of the workers be switched off remotely using the solenoid valve. Alternatively, the candidate may continue to be heated until the test is deemed to have reached its conclusion according to Section 4.9. The choice as to how long to prolong heating past the run-time should be based on a detailed risk assessment of the relative hazards and environmental impacts of handling and disposing of hot degraded candidate versus the generation of toxic fume and the possible projection of hot metal shrapnel.

4.8 Once the vessel and any remaining candidate have cooled to a safe handling temperature, the vessel and any remaining candidate should be disposed of in an environmentally responsible manner and according to local statutory requirements.

4.9 The test outcome is determined by whether or not the test reaches a conclusion prior to the run-time, together with the manner of conclusion in the case of a candidate for UN No. 3XXX. Evidence of test conclusion is based on:

- The visual and aural observation of vessel rupture accompanied by possible loss of thermocouple traces, or
- The visual and aural observation of vigorous venting accompanied by peaking of two or more vessel thermocouple traces, or
- The visual observation of decreased levels of fuming following the peaking of two or more thermocouple traces at temperatures in excess of 300 ºC.

In all cases, the conclusion time $t_{conclusion}$ is taken as the time at which the maximum temperature was recorded inside the vessel. For the purposes of assessing results, the term "rupture" includes any failure of welds and any fracture of metal in the test vessel, while the term “venting” denotes the absence of rupture.

5 *Test criteria and method of assessing results*

The candidate is heated under the set test conditions for its run-time for rupture $t_{rupture}$ or longer, unless it reaches an earlier conclusion according to Section 4.9.

5.1 For UN No. 3XXX the criteria for acceptance for transport in bulk are as follows:

- If the vessel is vented within a time less than the “run time for venting” tvent or the vessel is ruptured within a time less than the “run time for rupture” trupture, the outcome is “positive” (+) and the test candidate is not suitable for transport in bulk as Class 5.1. The candidate should be considered for transport in bulk only as Class 1.5.
- If the vessel is vented within a time greater than the “run time for venting” t_{vent}, and the vessel is not ruptured within a time less than the “run time for rupture” $t_{rupture}$, the outcome is “negative” (–) and the test candidate is suitable for transport in bulk as Class 5.1.

5.2 For UN No. 3375 the criteria for acceptance for transport in bulk are as follows:

- If the vessel is vented or ruptured within a time less than the “run time for rupture” trupture, the outcome is “positive” (+) and the test candidate is not suitable for transport in bulk as Class 5.1. The candidate should be considered for transport in bulk only as Class 1.5.
- If the vessel is not vented or ruptured within a time less than the “run time for rupture” trupture, the outcome is “negative” (–) and the test candidate is suitable for transport in bulk as Class 5.1.

6 *Examples of results*

6.1 *Example of calibration calculation based on heating rate of water:*

Calibration for water:
Initial temperature of water T_0 = 32°C.
Time to heat water from 32°C to 100°C T_{boil} = 21 minutes 30 seconds.
Measured mean heating rate between 40°C and 80°C $\dot{T}_{cal}$ = 3.50°C/minute.
Calibration-time t_{cal} = $T_{boil} + (T_0 - 25°C)/\dot{T}_{cal}$
= 21 minutes 30 seconds + (32°C -25°C)/3.50°C/minute
= 23 minutes 30 seconds.

6.2 *Example of run-time corrections and outcomes for UN 3375 candidate:*

Example run-time correction for Test Substance 1:
Initial temperature of substance T_0 = 21°C.
Maximum shipping temperature T_{ship} = 60°C.
Run-time for rupture $t_{rupture}$ = $[2.8]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [2.8]×(23 minutes 30 seconds) + (60-21)/3.50 minutes
= 76 minutes 57 seconds.
(Observed conclusion mode: Venting)
Observed conclusion time $t_{conclusion}$ = 109 minutes 48 seconds.
Test outcome: Negative, since $t_{conclusion} > t_{rupture}$

Example run-time correction for Test Substance 2:
Initial temperature of substance T_0 = 20°C.
Maximum shipping temperature T_{ship} = 60°C.
Run-time for rupture $t_{rupture}$ $= [2.8]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [2.8]×(24 minutes 7 seconds) + (60-20)/3.41 minutes
= 79 minutes 16 seconds.
(Observed conclusion mode: Venting)
Observed conclusion time $t_{conclusion}$ = 67 minutes 37 seconds.
Test outcome: Positive, since $t_{conclusion} < t_{rupture}$

Example run-time correction for Test Substance 3:
Initial temperature of substance T_0 = 10°C.
Maximum shipping temperature T_{ship} = 60°C.
Run-time for rupture $t_{rupture}$ $= [2.8]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [2.8]×(23 minutes 30 seconds) + (60-10)/3.50 minutes
= 80 minutes 5 seconds.
(Observed conclusion mode: Rupture)
Observed conclusion time $t_{conclusion}$ = 91 minutes 19 seconds.
Test outcome: Negative, since $t_{conclusion} > t_{rupture}$

6.3 *Example of run-time corrections and outcomes for UN 3XXX candidate:*

Example run-time correction for Test Substance 4:
Initial temperature of substance T_0 = 6°C.
Maximum shipping temperature T_{ship} = 60°C.
Run-time for venting t_{vent} $= [1.4]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [1.4]×(23 minutes 30 seconds) + (60-6)/3.50 minutes
= 48 minutes 20 seconds.
Run-time for rupture $t_{rupture}$ $= [2.8]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [2.8]×(23 minutes 30 seconds) + (60-6)/3.50 minutes
= 76 minutes 57 seconds.
Observed conclusion mode: Rupture
Observed conclusion time $t_{conclusion}$ = 58 minutes 35 seconds.
Test outcome: Positive, since rupture occurred with $t_{conclusion} < t_{rupture}$

Example run-time correction for Test Substance 5:
Initial temperature of substance T_0 = 37°C.
Maximum shipping temperature T_{ship} = 40°C.
Run-time for venting t_{vent} $= [1.4]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [1.4]×(23 minutes 30 seconds) + (40-37)/3.50 minutes
= 33 minutes 45 seconds.
Run-time for rupture $t_{rupture}$ $= [2.8]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [2.8]×(23 minutes 30 seconds) + (40-37)/3.50 minutes
= 66 minutes 39 seconds.
Observed conclusion mode: Venting
Observed conclusion time $t_{conclusion}$ = 23 minutes 59 seconds.
Test outcome: Positive, since venting occurred with $t_{conclusion} < t_{vent}$

Example run-time correction for Test Substance 6:

Initial temperature of substance T_0 = 31°C.
Maximum shipping temperature T_{ship} = 40°C.
Run-time for venting t_{vent} $= [1.4]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [1.4]×(23 minutes 30 seconds) + (40-31)/3.50 minutes
= 35 minutes 28 seconds.
Run-time for rupture $t_{rupture}$ $= [2.8]\times t_{cal} + (T_{ship} - T_0)/\dot{T}_{cal}$
= [2.8]×(23 minutes 30 seconds) + (40-31)/3.50 minutes
= 68 minutes 22 seconds.
Observed conclusion mode: Venting
Observed conclusion time $t_{conclusion}$ = 40 minutes 25 seconds.
Test outcome: Negative, since venting but no rupture occurred with $t_{vent} < t_{conclusion} < t_{rupture}$

Example of typical results:

	<u>**Substance**</u>	<u>**Result**</u>	<u>**Class / UN No..**</u>
1.	82.1 AN / 12.3 Water / 4.2 DO / 1.6 emulsifier	-	5.1 / 3375
2.	82.1 AN / 12.3 Water / 4.2 PO / 1.6 emulsifier	+	1.5 / 0332
3.	68.3 AN / 17.6 SN / 6.5 Water / 5.7 DO / 1.9 emulsifier	-	5.1 / 3375
4.	74.8 AN / 9.7 SP / 9.0 Water / 3.7 PO / 2.7 emulsifier	+	1.5 / 0332
5.	60.5 AN / 17.0 HMN / 12.0 Water / 10.0 EG / 0.5 guar gum	+	1.5 / 0332
6.	60.5 AN / 17.0 MAN / 12.0 Water / 10.0 EG / 0.5 guar gum	-	5.1 / 3XXX

Abbreviations: AN ammonium nitrate; DO diesel oil; PO paraffin oil; SN sodium nitrate; SP sodium perchlorate; HMN hexamine nitrate; EG ethylene glycol; MAN methylamine nitrate.