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**Committee of Experts on the Transport of Dangerous Goods (Twenty-first session, Geneva, 4-13 December 2000, agenda item 2(b))**

**WORK OF THE SUB-COMMITTEE OF EXPERTS ON THE TRANSPORT OF DANGEROUS GOODS**

**Draft amendments to the Model Regulations**

**Comments on proposal resulting from the Working Group on Classification of Ammonium Nitrate Emulsions, Intermediate for Blasting Explosives, Document ST/SG/AC.10/2000/20**

**Transmitted by the expert from France**

France supports globally the proposal. Nevertheless some comments are presented in the view of updating the proposal.

**Thermal stability Test for ANE**

The proposed 8 (a) new test is originating from the Test Series H for Division 4.1 Self-Reactive Substances and for Division 5.2 Organic Peroxides - see Section 28 in the Manual of Tests and Criteria, Test H4 Heat accumulation storage test. The proposed new test appears to be unnecessary complicated [(1)] for the purpose of such an assessment. Moreover it is not easy to correlate to the Test 3 (c) thermal stability test at 75 °C (167 °F), to be performed in the first part of the procedure - see proposed new Figure 10.2. In addition, the Test 3 (c) is deemed to take into account the most severe hot ambient conditions which may be encountered during transport, including loading i.e. ca 50 °C (122 °F). So for dealing with the wide range of loading and subsequent transport temperatures for ANEs worldwide - from 40 °C to 80 °C (104 to 176 °F) approximately - France proposes to have a simple thermal stability test at 95 °C (203 °F) as Test 8 (a), based on Test 3 (c). The corresponding test description is given in Annex 1. The test should be applied, in addition to the test 3 (c) already performed in the first part of the procedure if the loading and transport temperatures exceed 50 °C only. France has performed this test at 95 °C with an ANE manufactured in France and loaded at ca. 80 °C giving a negative result.

[(1)] and potentially expansive if it is decided to perform it in a bunker due to the explosion hazard

**ANE Gap Test**

France performed testing for substances with various degrees of sensitiveness to a shock wave as it is the purpose of the proposed new Test 8 (b).

a) ANE 1 considered as the most sensitive amongst the ANEs manufactured in France

b) Sensitized emulsion 1 considered as one of the less sensitive emulsion type E explosive manufactured on site in France. The explosion is not manufactured with ANE 1 but with another less sensitive ANE

c) ANE 2 medium sensitive ANE

d) Sensitized emulsion 2 manufactured on site with ANE 2

The results are as follows

| 70 mm gap | 50 mm gap | Result |
|---|---|---|
| ANE 1 | | – |
| | ANE 1 | – |
| Sensitized emulsion 1 | | + |
| ANE 2 | | – |
| Sensitized emulsion 2 | | + |

The above results support the proposed test, which should retain the same gap thickness as in the base Test 7 (b) i.e. 70 mm. Those results could be introduced in the examples of results, para 18.4.2.5 in an appropriate manner.

**Koenen Test**

Five different ANEs manufactured in France have been tested giving all a negative result with a limiting diameter of less than 2.0 mm.

**USA Vented Pipe Test**

France was unable to perform testing in particular because of the large sample size, i.e. 42 litres. Such a size will lead to important difficulties for doing the test in countries like France where large scale detonation testing is to be performed in a very few military places. This test should be supported by testing results demonstrating its usefulness. France has some experience with an heating under confinement test at a 10 kg scale using a pressure cooker as an apparatus containing the ANE and a large gas burner underneath the apparatus as a heat source : five different ANEs manufactured in France have been tested in these conditions giving all no explosion. So a test at a similar scale seems of interest and could replace the USA Vented Pipe Test.

**ANNEX 1**

18.4.1 *Test 8 (a) : Thermal stability test for ammonium nitrate emulsions*

18.4.1.1 *Introduction*

This test is used to measure the stability of a candidate for "ammonium nitrate emulsion, intermediate for blasting explosives" subjected to elevated thermal conditions to determine if the emulsion is too dangerous to transport. The test should be performed only if the loading and transport temperatures exceed 50 °C.

18.4.1.2 *Apparatus and materials*

18.4.1.2.1 The following apparatus is required:

(a) An electric oven equipped with ventilation, explosion-proof electrical features, and thermostatic control adequate to maintain and record the temperature at 95 ± 2 °C. The oven should have dual thermostats or some kind of protection against thermal run-away if the thermostat malfunctions;

(b) A lipless beaker of 35 mm diameter and 50 mm high and a watch-glass of 40 mm diameter;

(c) A balance capable of determining the sample weight to ± 0.1 g;

(d) Three thermocouples and a recording system;

(e) Two flat-bottomed glass tubes of 50 ± 1 mm diameter and 150 mm length and two 0.6 bar (60 kPa) pressure resisting stoppers.

18.4.1.2.2 An inert substance, whose physical and thermal properties are similar to the emulsion, should be used as the reference substance.

18.4.1.3 *Procedure*

18.4.1.3.1 Uninstrumented test: A 50 g sample is weighed into a beaker, covered and placed in an oven. The oven is heated to 95 °C and the sample left at oven temperature for 48 hours or until ignition or explosion occurs, whichever is sooner. If ignition or explosion does not occur but there is evidence, e.g. fuming or decomposition, that some self-heating has occurred, the procedure given in 18.4.1.3.2 should be performed. However, if the emulsion shows no evidence of thermal instability, it may be regarded as thermally stable and no further testing of this property is necessary.

18.4.1.3.2 Instrumented test: A 100 g sample is placed in one tube and the same quantity of reference substance is placed in the other. Thermocouples $T_1$ and $T_2$ are inserted into the tubes at half-height of the emulsion and reference substance. If the thermocouples are not inert with respect to both the emulsion being tested and the reference substance, they should be enclosed in sheaths which are inert. Thermocouple $T_3$ and the covered tubes are placed in the oven as shown in figure 18.4.1.1. The temperature difference (if any) between test sample and reference is measured for 48 hours after the sample and the reference substance reach 95 °C. Evidence of decomposition of the sample is noted.

18.4.1.4 *Test criteria and method of assessing results*

18.4.1.4.1 The result from an uninstrumented test is considered "+ " if ignition or explosion occurs and "– " if no changes are observed. The result of a instrumented test is considered " + " if an ignition or explosion occurs or if a temperature difference (i.e. self-heating) of 3 °C or greater is recorded. If no ignition or explosion occurs but self-heating of less than 3 °C is noted, additional tests and/or evaluation may be required to determine if the sample is thermally unstable.

18.4.1.4.2 If the test result is "+ ", the emulsion should be considered too thermally unstable for transport.

18.4.1.5 *Examples of results*

| **Substances** | **Observations** | **Result** |
|---|---|---|
| ANE<br>loaded at ca. 80 °C | Mass loss < 1 % | – |

Figure 18.4.1.1: EXPERIMENTAL SET-UP
(Same figure as figure 13.6.1.1, pages 119 of the Manual of Tests and Criteria)

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