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COMMITTEE OF EXPERTS ON THE TRANSPORT OF DANGEROUS GOODS (Twenty-first session, 4-12 December 2000 agenda item 2 (b))

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

Thermal Stability Tests on Ammonium Nitrate Emulsions

Transmitted by the Expert from the United Kingdom

1 At the eighteenth Session Sub-Committee of Experts on the Transport of Dangerous Goods, the Expert from the United Kingdom agreed to perform thermal stability tests on formulations of ammonium nitrate emulsions available in the UK using the procedure in Test Series 8 Test 8(a): Thermal stability test for ammonium nitrate emulsions.

2 This information paper gives some preliminary results of tests performed on two ammonium nitrate emulsion precursors for blasting explosives and, for comparative purposes, the results of tests on two blasting explosives. The blasting explosives are sensitised ammonium nitrate fuel oil (ANFO) emulsion explosives, one of which contains aluminium.

3 The procedure used is that given paper ST/SG/AC.10/2000/20. Para 18.4.1.1.2 of the procedure states:

18.4.1.1.2 Ammonium nitrate emulsions are usually loaded into the transport tank at high temperatures, typically about [80 to 90 °C]. This test is used to determine whether the emulsion is stable at such temperatures. In the way this type of test is normally carried out (see section 28.4.4), the 0.5 litre Dewar vessel is only representative for packagings, IBC's and small tanks. For the transport of ammonium nitrate emulsions, the test can be used to measure its stability during tank transport if the test is carried out at a temperature [20 °C] higher than the maximum temperature which may occur during transport, including the temperature at the time of loading.

Tests were performed in a 0.5 l Dewar vessel at either 20°C above the maximum loading or transport temperature or, in the case of the emulsion explosives which are transported at 'normal transport temperatures', at 75°C. The Dewar vessels were filled, as near as was possible, with 400 ml of substance. Suitable heat loss characteristics for the Dewar are given in the procedure as 80 to 100 mW.kg⁻¹.K⁻¹, the Dewar used for the majority of these tests had a somewhat lower value (49 mW.kg⁻¹.K⁻¹), therefore the tests may be considered to be slightly more severe.

The results are given in Table 1.

Sample	Test Temperature/Sample Mass/ Dewar Heat loss Characteristics ⁽¹⁾	Results/Observations/Other data
Emulsion explosive comprising primarily ammonium nitrate with sodium nitrate and mineral oil emulsion in water with microballoons. Tranported at ambient temperature	Test Temperature 75 °C Sample Mass 491.7 g Dewar heat loss 49 mW kg ⁻¹ K ⁻¹	PASS. No temperature excursion observed. Sample mass loss = 1.17 g Meets thermal stability requirement for Class 1 by Test Series 3 Test 3(c)
Emulsion explosive comprising primarily ammonium nitrate with sodium nitrate and mineral oil emulsion in water with aluminium and microballoons. Transported at ambient temperature.	Test Temperature 75 °C Sample Mass n/a Dewar heat loss 49 mW kg ⁻¹ K ⁻¹	PASS. No temperature excursion observed. Sample mass loss = n/a Meets thermal stability requirement for Class 1 by Test Series 3 Test 3(c)
Emulsion Matrix RC7192 ⁽²⁾ Exchem, EPC Group, Derbyshire, UK Transport temperature 60°C (loaded slightly hotter)	Test Temperature 85 °C Sample Mass 488.7 g Dewar heat loss 49 mW kg ⁻¹ K ⁻¹	Test data incomplete due to computer failure. Data for first 5 days only (3 days of data at test temperature). Mass loss 0.25 g
EP Gold emulsion ⁽²⁾ Orica, Greater Manchester, UK Loaded at 75°C (transported slightly cooler)	Test Temperature 95 °C Sample Mass 488.4 g Dewar heat loss 49 mW kg ⁻¹ K ⁻¹	No temperature excursion observed from available data. Temperatures after 7 days at test temperature showed no deviation. Mass loss 2.35 g
Emulsion Matrix RC7192 Exchem, EPC Group, Derbyshire, UK Transport temperature 60°C (loaded slightly hotter)	Test Temperature 85 °C Sample Mass 502.6 g Dewar heat loss 49 mW kg ⁻¹ K ⁻¹	In progress
EP Gold emulsion Orica, Greater Manchester, UK Loaded at 75°C (transported slightly cooler)	Test Temperature 95 °C Sample Mass 512.4 g Dewar heat loss 84 mW kg ⁻¹ K ⁻¹	In progress

The data for the emulsion explosive precursors is incomplete, as the data for the period from day 3 to day 5 (at the test temperature) is not available due a system failure. It may be considered that the likelihood of any significant exothermic decomposition having occurred during this period is low because:

- (i) the last temperature prior to system failure, and the first reading of temperature after monitoring was re-established were identical; and
- (ii) little mass loss occurred during the course of the test, consistent with little or no gas generation during an exothermic reaction.

No exotherm was observed for either of the blasting explosives.

The results, albeit on a limited number of samples, do not show any tendency of the emulsion precursors or blasting explosives examined to undergo any runaway exothermic decomposition in Test Series 8 Test 8(a). This should not be taken as evidence to support the use of Test Series 3 Test 3(c) as the means of assessing the thermal stability of AN emulsion precursors in bulk.

It is essential that any small-scale test used to assess behaviour in bulk reflects, as far as is possible, the parameters that are critical to modelling the bulk system. In a system where internal heat is (or may be) generated the critical parameters are the rate of heat generation and the heat flow out of the system time . The heat generated is a function of the quantity of material capable of reacting, heat loss (in a closed system) is through the walls of the container. Heat losses from the experimental arrangement in Test 3(c) are high (high surface to volume ratio), the heat losses from a bulk tank are low (low surface to volume ratio), and the test period is short. To model bulk behaviour on a small scale requires a system to compensate for this is essential, e.g. an insulated container, in the case of Test 8(a) this is the Dewar vessel at elevated temperature for a time period that reflects possible journey times. The heat loss characteristics of the vessel in Test 3(c) are suitable only for assessing the thermal stability of materials on a very small scale, little more than the volume used in the test.

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