

# UN/SCETDG/22/INF.4

## 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  
(Twenty-second session, 2-6 December 2002,  
agenda item 3(b))

### EXPLOSIVES, SELF-REACTIVE SUBSTANCES AND ORGANIC PEROXIDES

#### Classification of ammonium nitrate emulsions, suspensions and gels

##### Test Results of ANE

##### Transmitted by the expert from Spain

#### 1 Background

During the 19th session of the Sub-Committee, July 2001, the test methods for assigning substances to the new entry for Ammonium Nitrate Emulsions, Suspensions and Gels (ANEs) (UN 3375) were considered. Then draft test series 8(a), 8(b) and 8(c) were adopted.

As requested by the Working Group, Spain has performed draft test series 8(a), 8(b) and 8(c), for some Ammonium Nitrate Suspensions, and the results of the tests are attached as an Annex 1. The tests were validated by ATISAE, Notified Body for Spain.

#### 2 Results and comments

##### *Test 8(a): Thermal Stability Test*

The results are shown in Table 1 of Annex 1. Suspensions are produced at temperatures close to room temperature. Although the transportation temperature is generally lower than 40 °C, tests were carried out at 60 and 80 °C.

All the suspensions have proved to be thermally stable at the test temperatures.

##### *Test 8(b): ANE Gap Test*

The results are shown in Table 2 of Annex 1. All tests were carried out at room temperature (17-24 °C).

All the unsensitised suspensions failed to detonate, split the tubing or perforate the witness plates. Therefore they are not excluded from classification as Ammonium Nitrate Suspension.

***Test 8(c): Koenen Test***

The results are shown in Table 3 of Annex 3.

The limiting diameters in all cases were less than 2 mm and hence the results for all suspensions are negative ‘-’.

**3 Proposal**

It is proposed that the test results shown in the Annex be added to "Examples of results" for test series 8(a), 8(b) and 8(c) in the next edition of the Manual of Tests and Criteria (ST/SG/AC.10/C.3/38/Add.1, Annex 2).

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## ANNEX 1

Table 1 Test 8(a): Thermal Stability test

Substances	Sample mass g	Test T °C	Result	Comments
<b>Suspension - SP1</b> Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Thickener 0.7%, Glycol 13.0%	581	80	-	brown colouration crystal growth thickener break down
<b>Suspension - SP3</b> Ammonium nitrate 67.4%, MAN 15.0%, Water 12.0%, Thickener 0.6%, Glycol 5.0%	567	80	-	brown colouration crystal growth, thickener break down
<b>Suspension - SP4</b> Ammonium nitrate 71.4%, Hexamine 10.0%, Nitric acid 4.0%, Water 14.0%, Thickener 0.6%	582	60	-	yellow colouration crystal growth
<b>Suspension - SP5</b> Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine 5.0%, Nitric acid 2%, Water 12.0%, Thickener 0.6%, Glycol 6.0%	595	80	-	yellow colouration crystal growth

Table 2 Test 8(b): ANE gap Test

Substances	Density g/cm <sup>3</sup>	Gap mm	Result	Comments
<b>Suspension - SP1</b> Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Thickener 0.7%, Glycol 13.0%	1.45	70	-	Tube not fragmented Plate slightly indented
<b>Suspension - SP2</b> Ammonium nitrate 55.0%, Sodium Nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Thickener 1.0%, Glycol 14.0%	1.44	70	-	Tube not fragmented Plate slightly indented
<b>Suspension - SP3</b> Ammonium nitrate 67.4%, MAN 15.0%, Water 12.0%, Thickener 0.6%, Glycol 5.0%	1.41	70	-	Tube fragmented in large fragments Plate indented
<b>Suspension - SP4</b> Ammonium nitrate 71.4%, Hexamine 10.0%, Nitric acid 4.0%, Water 14.0%, Thickener 0.6%	1.46	70	-	Tube not fragmented Plate slightly indented
<b>Suspension - SP5</b> Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine 5.0%, Nitric acid 2%, Water 12.0%, Thickener 0.6%, Glycol 6.0%	1.48	70	-	Tube not fragmented Plate indented
<b>Suspension sensitised - SP3s</b> Ammonium nitrate 66.6%, MAN 14.9%, Water 11.9%, Thickener 0.6%, Glycol 5%, microballons 1.0%	1.24	70	+	Tube fragmented in small pieces Plate perforated and split
<b>Suspension sensitised - SP4s</b> Ammonium nitrate 69.4%, Hexamine 9.9%, Nitric acid 3.9%, Water 13.8%, Thickener 0.6%, microballons 1.4%	1.15	70	+	Tube fragmented in large and small pieces Plate perforated and split
<b>Suspension sensitised - SP5s</b> Ammonium nitrate 65.7%, Sodium perchlorate 7.9%, Hexamine 5.0%, Nitric acid 2.0%, Water 11.9%, Thickener 0.6%, Glycol 5.9%, microballons 1.0%	1.20	70	+	Tube fragmented in large and small pieces Plate perforated and split

**Table 3 Test 8(c): Koenen Test**

<b>Substances</b>	<b>Result</b>	<b>Comments</b>
<b>Suspension - SP1</b> Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Thickener 0.7%, Glycol 13.0%	-	Limiting diameter: <1.0 mm
<b>Suspension - SP2</b> Ammonium nitrate 55.0%, Sodium Nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Thickener 1.0%, Glycol 14.0%	-	Limiting diameter: 1.0 mm
<b>Suspension - SP3</b> Ammonium nitrate 67.4%, MAN 15.0%, Water 12.0%, Thickener 0.6%, Glycol 5.0%	-	Limiting diameter: 1.0 mm
<b>Suspension - SP4</b> Ammonium nitrate 71.4%, Hexamine 10.0%, Nitric acid 4.0%, Water 14.0%, Thickener 0.6%	-	Limiting diameter: 1.5 mm
<b>Suspension - SP5</b> Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine 5.0%, Nitric acid 2.0%, Water 12.0%, Thickener 0.6%, Glycol 6.0%	-	Limiting diameter: 1.0 mm

## **Annex 2**

# **PERFORMANCE OF TESTS 8(a), 8(b), 8(c) ON AMMONIUM NITRATE SUSPENSIONS**

UNIÓN ESPAÑOLA DE EXPLOSIVOS

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Dr José R. Quintana and Dr Fernando Beitia,

Senior Research Chemists at the R&D Department, UEE's Technology Direction

## SUMMARY AND CONCLUSIONS

Tests 8(a), 8(b) and 8(c) were carried out on five ammonium nitrate matrix suspensions (ANE) with the aim of Spain presenting the results as examples to be included in the Manual of Tests and Criteria (MTC) at the next meeting of the Committee of Experts on the Transport of Dangerous Goods, December 2002. A standard matrix emulsion was also tested in order to verify the procedure by comparing with the results of other laboratories.

The tested formulations (see Table 2.1) have ammonium nitrate as the main component combined with other inorganic nitrates, perchlorates and amine nitrates. All tested formulations have passed the tests 8(a), 8(b) and 8(c), which allows for their inclusion in the new entry UN 3375 established for 'non sensitised emulsions, suspensions and gels consisting primarily of a mixture of ammonium nitrate and a fuel phase, intended to produce a Type E blasting explosive only after processing prior to use'. From the analysis of the results obtained it can be concluded that the sensitivity of the tested suspensions is lower than that of the standard emulsion. This conclusion is supported by the following experimental data:

- lower limit diameters obtained in the Koenen test,
- lesser effects on the witness plate and tube in the gap test,
- a larger amount of microballoons needed to achieve the sensitisation of the suspensions.

## 1. INTRODUCTION

During the 19th session of the Sub-Committee of Experts on the Transport of Dangerous Goods, in July 2001, the draft for tests 8(a) Thermal Stability Test, 8(b) ANE Gap Test and 8(c) Koenen Test was adopted. At this and further sessions, several countries submitted results of these tests performed on ANE emulsions. At the 21<sup>st</sup> session, held in July 2002, it was decided that these three tests were to be used to assign substances to the new entry for non sensitised emulsions, suspensions and gels consisting primarily of a mixture of ammonium nitrate and a fuel phase, intended to produce a Type E blasting explosive only after processing prior to use (UN 3375), whereas test 8(d): Vented Pipe Test would provide information for those who wanted to evaluate their suitability for bulk transport. A modified form of the latter test will be developed over the next biennium.

Because *Unión Española de Explosivos* (UEE) is the only company that produces ANE matrix suspensions, Spain committed itself to present the results of tests 8(a), 8(b) and 8(c) performed on suspensions at the next Sub-Committee session (December 2002) for inclusion into the MTC (reference, UN/SCETG/21/INF.69, agenda item 2).

UEE has undertaken this task.

This report describes the procedure and results obtained when these tests are performed on a set of suspensions which cover a wide range of possible formulations. The tests were validated by ATISAE, the Spanish Notified body.

## 2. AMMONIUM NITRATE SUSPENSIONS

The technology for the manufacturing and application of suspensions, slurries or watergels was developed in the fifties but it was not until the sixties it was used in mining. Later on, explosive emulsions proliferated as alternative products and spread widely around the world,. The expansion of emulsion explosives has been so dominant that the great majority of manufacturers have abandoned the development of suspensions, slurries or watergels.

An ammonium nitrate suspension is a system constituted of a continuous liquid phase that consists of a saturated aqueous solution of oxidant salts and hydrosoluble fuels and a solid phase that consists of small particles of oxidants and fuels suspended in the liquid phase. To keep the solid particles in suspension, polymeric thickeners of natural or synthetic origin are used to increase the viscosity of the medium.

Although the suspensions were developed as an alternative to 'ANFO' for wet blastholes, a suspension is not waterproof until it has been made to react with crosslinking agents, which join the polymeric chains creating a gel network. Once the suspension is crosslinked, the gel gets a semisolid reology so it is more viscous and waterproof. These features can be advantageous in boreholes with cracks, by hindering leaks of explosive, interconnections between holes, etc.

The detonation phenomenon is originated by very quick redox reaction between fuels and oxidants which propagates at a rate faster than the speed of sound, resulting in a shock wave. The larger the contact surface between the reactants, the higher the reaction rate will be. Because the suspension is a heterogeneous system, the mixing degree between fuels and oxidants is limited, which leads to a lower reaction rate and, in consequence, the detonation initiation energy has to be higher.

Suspensions have a more complex formulation than emulsions. In addition to inorganic nitrates, they may contain, amine nitrates and / or perchlorates in order to compensate for their lower sensitivity to initiation. The examples of suspensions presented in this study have been chosen to cover a wide range of possible formulations. The formulations of the tested suspensions together with that of a standard emulsion are shown in Table 2.1. The sensitised suspensions have been adjusted to densities that range between 1150 and 1250 kg/m<sup>3</sup>, by adding a microballoon percentage range between 1.0 % and 1.4 %

**Table 2.1. Substances tested**

Type	SP0	SP0s	SP1	SP2	SP3	SP4	SP5	SP3s	SP4s	SP5s
	Emulsion	Sensitised emulsion	Suspension					Sensitised suspension		
Viscosity (p)	450	480	280	360	220	260	320	210	280	330
Density (kg/m <sup>3</sup> )	1340	1280	1450	1440	1410	1460	1480	1240	1150	1200
Ammonium nitrate	76.0	75.7	62.3	55.0	67.4	71.4	66.4	66.6	69.4	65.7
Sodium nitrate	-	-	-	8.0	-	-	-	-	-	-
Sodium perchlorate	-	-	11.0	8.0	-	-	8.0	-	-	7.9
Nitric acid	-	-	-	-	-	4.0	2.0	-	3.9	2.0
Water	17.0	17.0	13.0	14.0	12.0	14.0	12.0	11.9	13.8	11.9
Methyl amine nitrate	-	-	-	-	15.0	-	-	14.9	-	-
Hexamine	-	-	-	-	-	10.0	5.0	-	9.9	5.0
Paraffinic oil	5.6	5.6	-	-	-	-	-	-	-	-
Glycol	-	-	13.0	14.0	5.0	-	6.0	5.0	-	5.9
Emulsifier	1.4	1.4	-	-	-	-	-	-	-	-
Thickener	-	-	0.7	1.0	0.6	0.6	0.6	0.6	0.6	0.6
Microballons	-	0.3	-	-	-	-	-	1.0	1.4	1.0

### 3. TEST 8(a): THERMAL STABILITY TEST

#### 3.1. Procedure

The test has been carried out following the procedure described in section 18.4.1 of Annex 2 in Document ST/SG/AC.10/C.3/38/Add.1, July 31, 2001.



**Photograph 3.1**

The only commercial Dewar vessels that could be found had a higher capacity than the 0.5 L prescribed by the procedure. In order to get this volume, the lids were modified by adjusting a plastic piece to them. A small hole was made in the lid through which a thermocouple type 'T' was inserted. Three identical Dewar vessels were prepared. Photograph 3.1 shows the described vessel.

The three vessels, each of them with its thermocouple, were introduced into an oven which had previously been checked for being able to keep the temperature deviation of not more than 1 °C for up to 10 days. A fourth thermocouple was placed together with the three vessels to record the oven temperature. The four thermocouples were connected to a data logger, which allowed the temperature data to be transferred to the computer for later analysis. This test setting is showed in Photographs 3.2 and 3.3.

In order to determine the heat loss characteristics of the system, the vessels were filled with hot water and, once covered and left at room temperature, the water temperature decrease was recorded as a function of time. As can be seen in Figure 3.1, the data could be fitted to three exponential curves from which the constants were determined.



**Photograph 3.2**



**Photograph 3.3**

Considering that the exponential constants are the reciprocal of the half time of cooling,  $t_{1/2}$ , and using the following equation,

$$L = \ln 2 \cdot \frac{C_p}{t_{1/2}}$$

where  $L$  (mW/kg·K) is the heat loss per unit of mass,  $C_p$  (J/K) is the specific heat and  $t_{1/2}$  (s) is the half time of cooling, the heat loss for the three Dewar vessels were determined as the following values: 9.6 (1), 7.7 (2) and 8.1 mW/kg·K (3). These values are lower than that prescribed (80-100 mW/kg·K), which means that the experimental system sufficiently fulfils the requirements demanded.

The test has to be carried out at a temperature 20 °C higher than the maximum temperature which may occur during transport. Considering that, by their own nature, the suspensions are manufactured, transported and used at temperatures close to room temperature, in general the maximum transport temperature is no higher than 40 °C. Thus, the first tests were carried out at 60 °C. However, in order to

increase the safety margin, tests were also carried at 80 °C, 40 °C over transport temperature. This way the suspensions were also tested at the temperature that emulsions are usually tested.

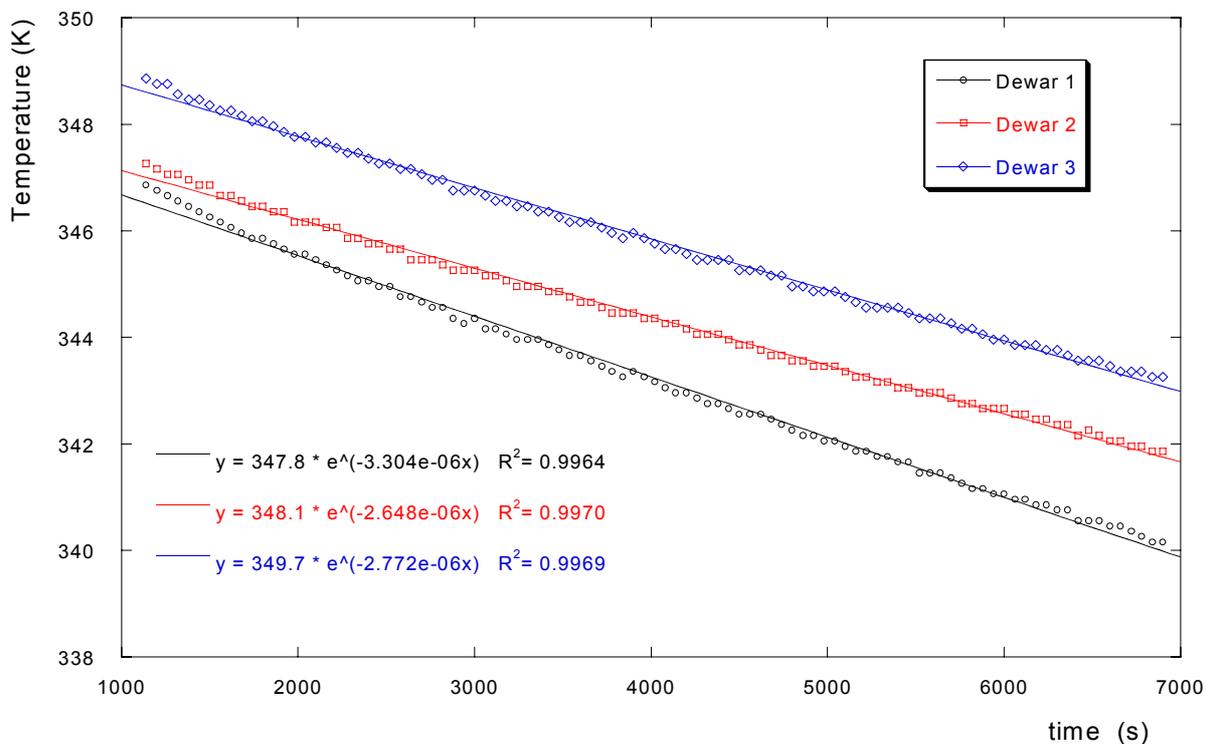


Figure 3.1. Cooling rates of the water contained in the three Dewar vessels.

## 3.2. Results

Figures 3.2 and 3.3 show the temperatures of the samples and the oven as a function of time in the tests performed at 60 and 80 °C, respectively. Detailed information is given in Table 3.1. The sample temperatures were always lower than those of the oven, which indicates that no exothermic reactions occur within the samples. In all cases, the tested substances were thermally stable and could be further tested as candidates for 'ammonium nitrate emulsion, suspension or gel, intermediate for blasting explosives'.

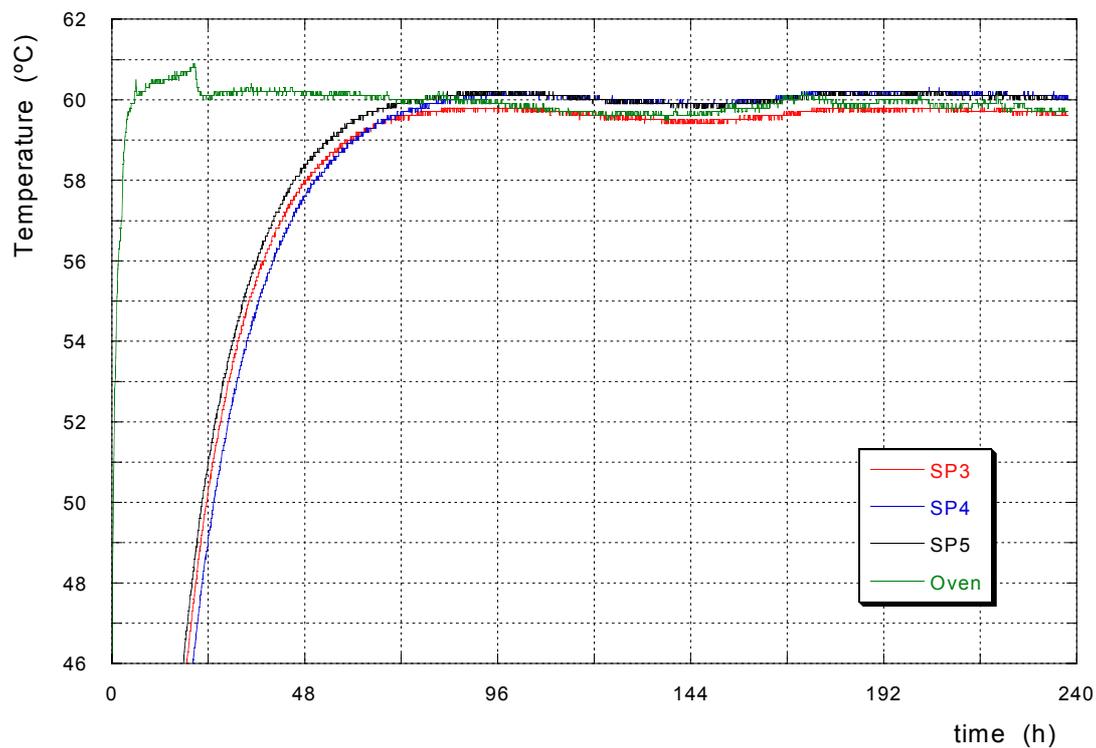


Figure 3.2. Record of temperatures for the test carried out at 60 °C.

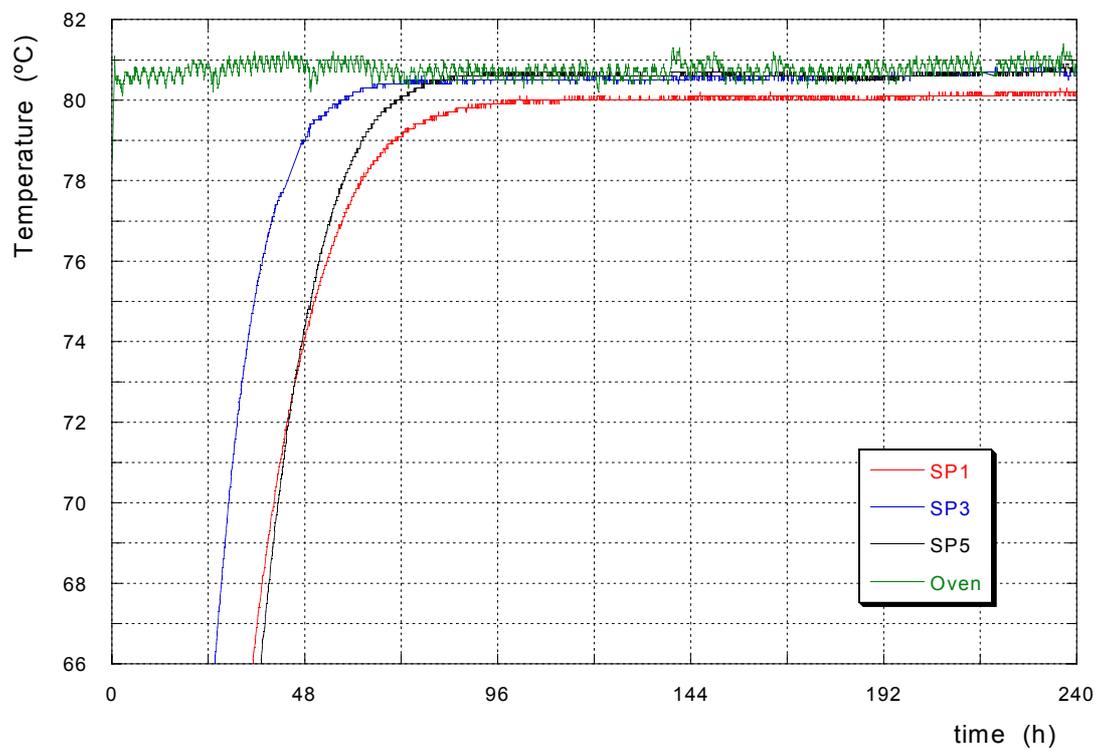


Figure 3.3. Record of temperatures for the test carried out at 80 °C.

**Table 3.1. Test 8(a): Thermal Stability test**

Substances	Sample mass (g)	Dewar	T <sub>AVE,OVE</sub> (°C)	T <sub>MAX,OVE</sub> (°C)	Test time (h)	T <sub>INI,SAM</sub> (°C)	T <sub>AVE,SAM</sub> (°C)	T <sub>MAX,SAM</sub> (°C)	$\Delta t$ (T <sub>INI,SAM</sub> → T <sub>AVE,OVE-2</sub> ) (h)	$\Delta t$ (T <sub>AVE,OVE-2</sub> → T <sub>MAX,SAM</sub> ) (h)	Mass loss (%)	Comments	Result
<b>Suspension - SP3</b> Ammonium nitrate 67.4%, MAN 15.0%, Water 12.0%, Thickener 0.6%, Glycol 5.0%	567.2	1	59.9	60.3	237.83	19.7	59.7	59.8	49.71	82.05	0.21	slight brown colouration crystal growth	-
<b>Suspension - SP4</b> Ammonium nitrate 71.4%, Hexamine 10.0%, Nitric acid 4.0%, Water 14.0%, Thickener 0.6%	582.4	2	59.9	60.3	237.83	19.5	60.1	60.2	52.05	92.40	0.19	yellow colouration crystal growth	-
<b>Suspension - SP5</b> Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine 5.0%, Nitric acid 2%, Water 12.0%, Thickener 0.6%, Glycol 6.0%	592.8	3	59.9	60.3	237.83	18.9	60.1	60.2	46.76	83.48	0.19	yellow colouration crystal growth	-
<b>Suspension - SP1</b> Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Thickener 0.7%, Glycol 13.0%	580.8	2	80.7	81.1	241.67	18.5	80.6	80.8	67.24	218.05	0.62	brown colouration crystal growth thickener break down	-
<b>Suspension - SP3</b> Ammonium nitrate 67.4%, MAN 15.0%, Water 12.0%, Thickener 0.6%, Glycol 5.0%	567.2	1	80.7	81.1	241.67	17.6	80.0	80.2	46.28	206.21	0.29	brown colouration crystal growth, thickener break down	-
<b>Suspension - SP5</b> Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine 5.0%, Nitric acid 2%, Water 12.0%, Thickener 0.6%, Glycol 6.0%	595.2	3	80.7	81.1	241.67	18.3	80.6	80.8	60.62	231.85	0.50	yellow colouration crystal growth	-

T<sub>AVE,OVE</sub> : average oven temperature. T<sub>MAX,OVE</sub> : maximum oven temperature. T<sub>INI,SAM</sub> : initial sample temperature, T<sub>AVE,SAM</sub> : average sample temperature once stabilised. T<sub>MAX,SAM</sub> : maximum sample temperature.  $\Delta t$  (T<sub>INI,SAM</sub> → T<sub>AVE,OVE-2</sub>) : time taken for the sample to rise from initial temperature to 2 °C below the oven temperature.  $\Delta t$  (T<sub>AVE,OVE-2</sub> → T<sub>MAX,SAM</sub>) : time taken for the sample to rise from 2 °C below the oven temperature to its maximum temperature.

## 4. TEST 8(b): ANE GAP TEST

### 4.1. Procedure

The test has been carried out following the procedure described in section 18.5.1 of Annex 2 in Document ST/SG/AC.10/C.3/38/Add.1, July 31, 2001. Photographs 4.1 and 4.2 show the setting of the different elements used to carry out this test.

Donor charge cylinders of pentolite 50/50 with a diameter of 95mm and a height of 95 mm were used. The charge densities used ranged between 1631 and 1638 kg/m<sup>3</sup> and the masses ranged between 1064 and 1107 g.

The tube and witness plate steel was ST-52. This steel has a tensile strength between 500 and 650 MPa and an elongation between 19 and 21 %. The tubes had an outer diameter of 95.0 mm, a thickness of 11.3 mm and a height of 280 mm. The plate dimensions were 200×200×20 mm.

The polymethyl methacrylate cylinders had a diameter of 95 mm and a height of 70 mm.

### 4.2. Results

The data and the results of the different tests are shown in Table 4.1 and in photographs 4.1-4.16. In all cases the non-sensitised standard emulsion and suspensions pass the test (negative result). Nevertheless, the effect on the tube and the plate show different sensitivities among the tested substances. In all cases, the sensitised ANEs perforated the witness plate.

When comparing the results for the different tested substances, it can be observed that all suspensions show lesser effects than the standard emulsion. The closest effect to the standard emulsion was that of the suspension SP3. In both cases the tube was fragmented and the plate was considerably indented; however, in the case of the suspension SP3 product remains were found after the test, while this did not happen in the case of the emulsion. In the case of the rest of the suspensions, the difference from the standard emulsion is even greater: in all cases the tubes were not fragmented, the plates remained virtually intact, and the residual substance which remained after the test was considerable.

**Table 4.1 Test 8(b): ANE Gap Test**

<b>Substances</b>	<b>Density (kg/m<sup>3</sup>)</b>	<b>Gap (mm)</b>	<b>T (°C)</b>	<b>Tube</b>	<b>Plate</b>	<b>Product remains</b>	<b>Result</b>
<b>Emulsion - SP0</b> Ammonium nitrate 76.0%, Water 17.0%, Emulsifier 1.4%, Paraffinic oil 5.6%	1340	70	17	Fragmented in large pieces	Indented	No	-
<b>Emulsion sensitised - SP0s</b> Ammonium nitrate 75.7%, Water 17.0%, Emulsifier 1.4%, Paraffinic oil 5.6%, Microballons 0.3%	1280	70	19	Fragmented in large and small pieces	Perforated and split	-	+
<b>Suspension - SP1</b> Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Thickener 0.7%, Glycol 13.0%	1450	70	19	Not fragmented	Slightly indented	Much	-
<b>Suspension - SP2</b> Ammonium nitrate 55.0%, Sodium Nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Thickener 1.0%, Glycol 14.0%	1440	70	18-23	Not fragmented	Slightly indented	Much	-
<b>Suspension - SP3</b> Ammonium nitrate 67.4%, MAN 15.0%, Water 12.0%, Thickener 0.6%, Glycol 5.0%	1410	70	18-24	Fragmented in large pieces	Indented	Very little	-
<b>Suspension - SP4</b> Ammonium nitrate 71.4%, Hexamine 10.0%, Nitric acid 4.0%, Water 14.0%, Thickener 0.6%	1460	70	18	Not fragmented	Slightly indented	Much	-
<b>Suspension - SP5</b> Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine 5.0%, Nitric acid 2%, Water 12.0%, Thickener 0.6, Glycol 6.0%	1480	70	17-19	Not fragmented	Indented	Little	-
<b>Suspension sensitised - SP3s</b> Ammonium nitrate 66.6%, MAN 14.9%, Water 11.9%, Thickener 0.6%, Glycol 5%, microballons 1.0%	1240	70	19	Fragmented in small pieces	Perforated and split	-	+
<b>Suspension sensitised - SP4s</b> Ammonium nitrate 69.4%, Hexamine 9.9%, Nitric acid 3.9%, Water 13.8%, Thickener 0.6%, microballons 1.4%	1150	70	19	Fragmented in large and small pieces	Perforated and split	-	+
<b>Suspension sensitised - SP5s</b> Ammonium nitrate 65.7%, Sodium perchlorate 7.9%, Hexamine 5.0%, Nitric acid 2.0%, Water 11.9%, Thickener 0.6%, Glycol 5.9%, microballons 1.0%	1200	70	19	Fragmented in large and small pieces	Perforated and split	-	+

**Photographs 4.1 - 4.10. ANE Gap Test**



**4.1. Test setting**



**4.2. Test setting**



**4.3. Emulsion - SP0**



**4.4. Emulsion sensitised - SP0s**



**4.5. Suspension - SP1**



**4.6. Suspension - SP1**



**4.7. Suspension - SP2**



**4.8. Suspension - SP2**



**4.9. Suspension - SP3**



**4.10. Suspension - SP4**

**Photographs 4.11 - 4.16. ANE Gap Test**



4.11. Suspension - SP4



4.12. Suspension - SP5



4.13. Suspension - SP5



4.14. Suspension sensitised -  
SP3s



4.15. Suspension sensitised -  
SP4s



4.16. Suspension sensitised -  
SP5s

## 5. TEST 8(c): KOENEN TEST

### 5.1. Procedure

The test has been carried out following the procedure described in section 18.6.1 of Annex 2 in Document ST/SG/AC.10/C.3/38/Add.1, July 31, 2001. Photographs 5.1 and 5.2 show the equipment used to carry out this test.

The tools and materials were supplied by Reichelt and Partern GmbH, official supplier for this equipment.

According to the test procedure, three trials have to be performed without getting any effect types 'F', 'G' or 'H' which would indicate an 'explosion'. The moment when such a result is achieved, the next larger diameter hole has to be used. The larger diameter at which at least one 'explosion' is obtained is called 'limiting diameter'. For a substance to have a '-' result at this test, the limiting diameter cannot be higher than 1.5 mm.

### 5.2. Results

The results obtained for this test are presented at Table 5.1. The mass range used for the different substances is shown. The 'hole diameter' columns show the effect of the tube after the tests by the procedure code. Letter 'O' means that the tube was unchanged and letter 'F' means that the tube was fragmented into three or more mainly large pieces, which in some case may be connected with each other by a narrow strip.

Photographs 5.3-5.8 show the state of the tube after some tests, as an example.

All substances tested passed, since the limiting diameters found were never higher than 1.5 mm. When comparing the results obtained for the suspensions with the result obtained for the standard emulsion, in the case of SP4, these are similar, but the rest of the suspensions show lower limiting diameters. Even in the case of the suspension SP4, containing mainly ammonium nitrate and sodium perchlorate, the limiting diameter is lower than the minimum for this procedure, 1 mm.

**Table 5.1. Test 8(c): Koenen Test**

Substances	Mass (g)	Hole diameter (mm)			Limiting diameter (mm)	Result
		1.0	1.5	2.0		
<b>Emulsion - SP0</b> Ammonium nitrate 76.0%, Water 17.0%, Emulsifier 1.4%, Paraffinic oil 5.6%	36.2-39.7	F	F	O,O,O	1.5	-
<b>Suspension - SP1</b> Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Thickener 0.7%, Glycol 13.0%	39.5-39.7	O,O,O	O	-	<1.0	-
<b>Suspension - SP2</b> Ammonium nitrate 55.0%, Sodium Nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Thickener 1.0%, Glycol 14.0%	39.5-41.1	F	O,O,O	-	1.0	-
<b>Suspension - SP3</b> Ammonium nitrate 67.4%, MAN 15.0%, Water 12.0%, Thickener 0.6%, Glycol 5.0%	39,5-41.1	O,F	O,O,O	-	1.0	-
<b>Suspension - SP4</b> Ammonium nitrate 71.4%, Hexamine 10.0%, Nitric acid 4.0%, Water 14.0%, Thickener 0.6%	39.0-41.0	-	F	O,O,O	1.5	-
<b>Suspension - SP5</b> Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine 5.0%, Nitric acid 2%, Water 12.0%, Thickener 0.6%, Glycol 6.0%	39.6-39.9	F	O,O,O	-	1.0	-

**Photographs 5.1- 5.8. Test 8(c): Koenen Test**



**5.1. Test setting**



**5.2. Tube**



**5.3. Emulsion - SP0  
1.5 mm hole**



**5.4. Suspension - SP1  
1.0 mm hole**



**5.5. Suspension - SP2  
1.0 mm hole**



**5.6. Suspension - SP3  
1.0 mm hole**



**5.7. Suspension - SP4  
1.5 mm hole**



**5.8. Suspension - SP5  
1.0 mm hole**