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-third session, 30 juin-4 July 2003
Agenda item 3(b))

EXPLOSIVES, SELF-REACTIVE SUBSTANCES AND ORGANIC PEROXIDES

Definition of ammonium nitrate emulsions, suspensions and gels
(related to document ST/SG/AC.10/C.3/2003/13)

Transmitted by the expert from Spain

SCOPE

Attached is a report that provides information on the behaviour of ammonium nitrate suspensions for their classification and safety in handling and transport.

To this end, different suspensions, containing percentages of perchlorates and/or water-soluble amine nitrate salts close to the limits indicated in the definition proposed in ST/SG/AC.10/C.3/2003/13, were submitted to the different Series 1, 2 and 8 tests of the Manual of Test and Criteria.

BACKGROUND

It is a well-known fact that the addition of perchlorates or water-soluble amine nitrates to emulsions implies a high increase in their sensitivity, increasing the risks involved in their handling and transport. However, this behaviour cannot be transposed to the case of ammonium nitrate suspensions. As will be explained later on, suspensions are less sensitive, per se, than emulsions and it is necessary to add perchlorates and/or water-soluble amine nitrates to their compositions to reach sensitivity levels similar to those of emulsions. As can be seen in the different tests carried out, despite containing perchlorates and/or amine nitrates, many suspensions show less drastic results than the standard emulsion, which fact implies a lower risk in their handling or transport.

In order to demonstrate that the range of compositions for suspensions and gels proposed to be included in the Special Provision 309 (Document ST/SG/AC.10/C.3/2003/13) does not raise the risk level of the substances included in UN 3375, Series 1, 2 and 8 tests were carried out on a group of suspensions that cover the proposed range of compositions. To obtain a better understanding of the risk level of these substances, the test results were measured and compared with results corresponding to different emulsions. Since emulsions are substances well-known by most experts and Competent Authorities, this comparison will permit the risk level of the suspensions in question to be determined.

From the results obtained it can be concluded that the suspensions tested present a lower risk than the usual emulsions, despite the fact that they contain perchlorates or water-soluble amine nitrate salts. It is also demonstrated that the increased risk involved when these substances are added to emulsions is not present in the case of suspensions. All the substances tested, except the emulsion with perchlorate, have passed the Series 8 tests.

The behaviour of the substances in these tests was measured and, in addition, the results were accompanied by those obtained by well-known standard emulsions in order to use them as a reference and be able to demonstrate that the composition range proposed for suspensions does not present a higher risk than that shown by the composition range already adopted for emulsions.

PERFORMANCE OF TEST SERIES 1, 2 & 8

ON

AMMONIUM NITRATE SUSPENSIONS

UNIÓN ESPAÑOLA DE EXPLOSIVOS, S.A. (UEE)

May 2003

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SUMMARY AND CONCLUSIONS

In order to demonstrate that the range of compositions for suspensions and gels that the Spanish Authority proposes to include in the Special Provision 309 (Document ST/SG/AC.10/C.3/2003/13) does not raise the risk level of the substances included in UN 3375, Series 1, 2 and 8 tests were carried out on a group of suspensions that cover the proposed range of compositions. To obtain a better understanding of the risk level of these substances, the test results were measured and compared with results corresponding to different emulsions. Since emulsions are substances well-known by most experts and Competent Authorities, this comparison will permit the risk level of the suspensions in question to be determined.

From the results obtained it can be concluded that the suspensions tested present a lower risk than the usual emulsions, despite the fact that they contain perchlorates or water-soluble amine nitrate salts. It is also demonstrated that the increased risk involved when these substances are added to emulsions is not present in the case of suspensions. Thus, according to the results, emulsion EM3 with 9.7% perchlorate does not pass the Series 2 and should be considered as a Class 1 substance. However, suspension SP1 with 11.0% perchlorate and suspension SP6 with 10% monomethylamine nitrate do pass Series 1 tests, and therefore should be considered as non-explosive substances. None of the emulsions tested passed the Series 1, although, with the exception of the emulsion with perchlorate, they did pass the Series 2 and 8 tests, so they cannot be classified as Class 1 substances. All the substances tested, except the emulsion with perchlorate, have passed the Series 8 tests.

1. INTRODUCTION

At its December 2002 meeting, the Committee of Experts on the Transport of Dangerous Goods approved the inclusion of Test Series 8 in the Manual of Tests and Criteria. Tests 8(a), 8(b) and 8(c) should be used to establish whether an ammonium nitrate emulsion or suspension or gel, intermediate for Type E blasting explosives (ANE) may be assigned to Class 5.1 (UN 3375).

At the request of the ANE Working Group in July 2002, Spain performed Tests 8(a), 8(b) and 8(c), for ammonium nitrate suspensions. The formulations tested were not current commercial formulations and were chosen to test the highest likely concentrations of different ingredients.

The results of these tests were presented in the report UN/SCETDG/22/INF.4. As it is shown in this report, the formulations tested have clearly passed the Tests 8(a), 8(b) and 8(c), showing lower sensitivity than standard emulsions. However the Spanish proposal for including these test results in "Examples of results" for Tests 8(a), 8(b) and 8(c) in the new edition of the Manual of Tests and Criteria, was not adopted, because several experts considered that the tests had been performed on suspensions the composition of which did not conform to the definition in Special Provision 309. This Special Provision describes a range of formulations for emulsions, suspensions and gels, which although appropriate for usual emulsion compositions, is not broad enough to include suspensions.

Consequently, and with the main object of making the content of Special Provision 309 totally coherent with the wording of UN 3375, the Spanish Authority has presented a proposal, Document ST/SG/AC.10/C.3/2003/13, consisting of a modification of Special Provision 309, introducing a paragraph referring to a specific range of compositions for suspensions and gels, in order to include various ingredients used specifically in suspensions and gels.

The purpose of this report is to provide technical support to the proposal, complementing Document UN/SCETDG/22/INF.4. It aims to give ample information on the behaviour of ammonium nitrate suspensions for their classification and safety in handling and transport. To this end, different suspensions, containing percentages of perchlorates and/or water-soluble amine nitrate salts close to the limits indicated in the definition proposed by the Spanish Authority, were submitted to the different Series 1, 2 and 8 tests.

The behaviour of the substances in these tests was measured and, in addition, the results were accompanied by those obtained by well-known standard emulsions in order to use them as a reference and be able to demonstrate that the composition range proposed for suspensions does not present a higher risk than that shown by the composition range already adopted for emulsions.

It is a well-known fact that the addition of perchlorates or water-soluble amine nitrates to emulsions implies a high increase in their sensitivity, increasing the risks involved in their handling and transport. However, this behaviour cannot be transposed to the case of ammonium nitrate suspensions. As will be explained later on, suspensions are less sensitive, per se, than emulsions and it is necessary to add perchlorates and/or water-soluble amine nitrates to their compositions to reach sensitivity levels similar to those of emulsions. As can be seen in the different tests carried out, despite containing perchlorates and/or amine nitrates, many suspensions show less drastic results than the standard emulsion, which fact implies a lower risk in their handling or transport.

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 that 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 acquires a semisolid rheology so it is more viscous and waterproof. These features can be advantageous in boreholes with cracks, by preventing leaks of explosive, interconnections between holes, etc.

The detonation phenomenon is originated by very quick redox reactions between fuels and oxidants that propagate at a rate faster than the speed of sound, resulting in a shock wave supported by chemical reaction. The larger the contact surface between the reactants, the higher the reaction rate will be. Because the suspension is a heterogeneous system, the degree of mixing between fuels and oxidants is limited, which leads in general to a lower reaction rate, lower sensitivity and higher minimum booster requirements than emulsions.

Suspensions have a more complex formulation than emulsions. In addition to inorganic nitrates, they may contain water-soluble amine nitrates salts and/or perchlorates in order to reach similar sensitivities to those of matrix emulsions. The examples of suspensions presented in this study have been chosen to cover the concentration range included in the Spanish proposal (Document ST/SG/AC.10/C.3/2003/13). The formulations of the suspensions tested together with those of reference emulsions are shown in Table 2.1.

Table 2.1. Substances tested

	EM1	EM2	EM3	SP1	SP2	SP3	SP4	SP5	SP6
Ammonium nitrate	76.0	82.1	74.9	62.3	55.0	67.4	71.4	66.4	68.4
Sodium nitrate	-	-	-	-	8.0	-	-	-	-
Sodium perchlorate	-	-	9.7	11.0	8.0	-	-	8.0	-
Methylamine nitrate	-	-	-	-	-	15.0	-	-	10.0
Hexamine nitrate	-	-	-	-	-	-	14.0	7.0	-
Water	17.0	12.3	9.0	13.0	14.0	12.0	14.0	12.0	13.0
Paraffinic oil	5.6	4.2	3.7	-	-	-	-	-	-
Glycol	-	-	-	13.0	14.0	5.0	-	6.0	8.0
Emulsifier	1.4	1.4	2.7	-	-	-	-	-	-
Thickener	-	-	-	0.7	1.0	0.6	0.6	0.6	0.6

3. TESTS 1(a) & 2(a): UN GAP TESTS

3.1. Procedure

The tests were carried out according to the procedures described in Sections 11.4.1 and 12.4.1 of the Manual of Tests and Criteria, ST/SG/AC.10/11/Rev.3. Photographs 3.1 and 3.2 show the layout of the different elements in Tests 1(a) and 2(a), respectively.

3.1. Test 1(a)

3.2. Test 2(a)

Donor charge consisted of cast cylinders of Pentolite 50/50 weighing 160 g, with a 50 mm diameter and 50 mm in height. The density of the charges used was between 1631 and 1638 kg/m^3. Cold-drawn, seamless, carbon steel tubes were used. The tubes had a 48.5 mm outside diameter and measured 400 mm in length, with a thickness of 4.0 mm. The witness plates were of mild steel measuring 150×150×3.2 mm. For Test 2(a) polymethyl methacrylate cylinders were used with a diameter of 50 mm and a height of 50 mm.

In all cases, according to the French cavitation method, microspheres K15 from 3M were added to the substances to be tested in the proportion of 500 mg per litre of substance. The mixtures were mixed to the point of total dispersion of the microspheres.

The test was repeated twice for each substance. Table 3.1 shows the test in which the most drastic result was obtained.

3.2. Results

The data and results of the different tests are shown in Table 3.1. As can be seen, the standard EM1 emulsion passed Test 1(a). However, the EM2 emulsion, with a lower water content, did not pass the test and had to be tested under the Series 2.

All the suspensions tested passed Test 1(a). In Table 3.1 it can be seen that the majority of the suspensions show fractured tube length values below those of the standard EM1 emulsion.

Table 3.1. Tests 1(a) & 2(a): UN gap test

Substances	Density (kg/m^3)	Gap (mm)	Temp (°C)	Fragmentation length (cm)	Witness plate	Result	
						Series 1	Series 2
EM1 Ammonium nitrate 76.0%, Water 17.0%, Paraffinic oil 5.6%, Emulsifier 1.4%	1340	0	19	23	No damage	-	
EM2 Ammonium nitrate 82.1%, Water 12.3%, Paraffinic oil 4.2%, Emulsifier 1.4%	1370	0	20	40	Slightly domed	+	
		50	20	0	No damage		-
SP1 Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Glycol 13.0%, Thickener 0.7%	1440	0	20	19	Slightly domed	-	
SP2 Ammonium nitrate 55.0%, Sodium nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Glycol 14.0%, Thickener 1.0%	1430	0	20	16	Slightly domed	-	
SP3 Ammonium nitrate 67.4%, Methylamine nitrate 15.0%, Water 12.0%, Glycol 5.0%, Thickener 0.6%	1460	0	20	32	Slightly domed	-	
SP4 Ammonium nitrate 71.4%, Hexamine nitrate 14.0%, Water 14.0%, Thickener 0.6%	1450	0	20	18	No damage	-	
SP5 Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine nitrate 7.0%, Water 12.0%, Glycol 6.0%, Thickener 0.6%	1460	0	20	23	Slightly domed	-	
SP6 Ammonium nitrate 68.4%, Methylamine nitrate 10.0%, Water 13.0%, Glycol 8.0%, Thickener 0.6%	1430	0	20	17	No damage	-	

4. TESTS 1(b), 2(b) & 8(c): KOENEN TEST

4.1. Procedure

The tests have been carried out following the procedure described in sections 11.5.1 and 12.5.1 of Manual of Tests and Criteria, ST/SG/AC.10/11/Rev.3, and in section 18.6.1 of Document ST/SG/AC.10/29/Add.2, 17 February 2003. Photographs 4.1 and 4.2 show the equipment used to carry out this test. Reichelt & Partner GmbH, official distributors of this equipment, supplied the apparatus and materials used.

4.1. Device

4.2. Tube

According to the test procedure, three trials have to be performed without getting any ‘F’, ‘G’ or ‘H’ type results which would indicate an ‘explosion’. The moment such a result is achieved, the next larger diameter hole has to be used. The largest diameter at which at least one ‘explosion’ is obtained is called the 'limiting diameter'. For a substance to have a ‘-‘ result in this test, the limiting diameter has to be less than 1.0 mm for Series 1, or less than 2.0 for Series 2 and 8.

4.2. Results

The results obtained for this test are presented in Table 4.1. The mass range used for the different substances is shown. The 'hole diameter' columns show the effect on the tube after the tests according to the procedure code. Letter 'O' means that the tube was intact and letter 'F' means that the tube was fragmented into three or more large pieces, which in some case were connected to each other by a narrow metal strip.

All substances tested, except the emulsion with sodium perchlorate (EM3), passed the Series 2 and 8 tests, since the limiting diameters found were below 2.0 mm. If we compare the results obtained by the suspensions with those obtained by a standard emulsion (EM1), it can be seen that in one case they are similar, SP4, but the rest of the suspensions show smaller limiting diameters. In the case of suspensions SP1 and SP6 the limiting diameter is smaller than the smallest contained in the procedure (1.0 mm), so they passed the Series 1 tests.

It should be noted that the EM3 emulsion which contains 9.7 % sodium perchlorate shows a limiting diameter of 2.0 mm, whilst the SP1 suspension that contains 11.0 % sodium perchlorate shows a limiting diameter of less than 1.0 mm.

Table 4.1. Tests 1(b), 2(b) & 8(c): Koenen test

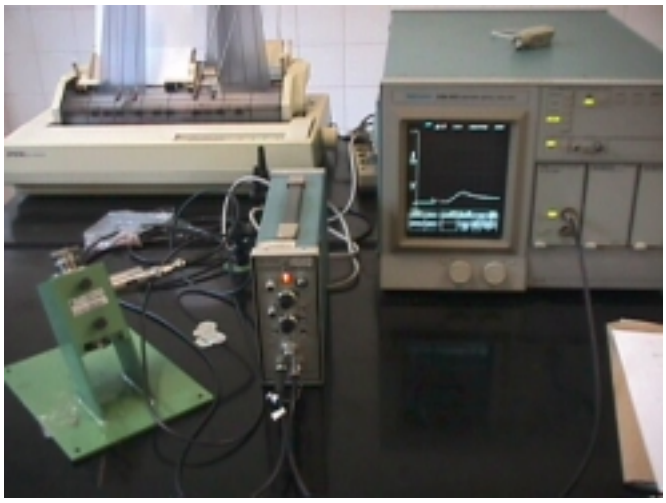
Substances	Mass (g)	Hole diameter (mm)				Limiting diameter (mm)	Result		
		1.0	1.5	2.0	3.0		Series 1	Series 2	Series 8
EM1 Ammonium nitrate 76.0%, Water 17.0%, Paraffinic oil 5.6%, Emulsifier 1.4%	36.2-39.7	F	F	O,O,O		1.5	+	-	-
EM2 Ammonium nitrate 82.1%, Water 12.3%, Paraffinic oil 4.2%, Emulsifier 1.4%	37.9-38.1		F	O,O,O		1.5	+	-	-
EM3 Ammonium nitrate 74.9%, Sodium perchlorate 9.7%, Water 9.0%, Paraffinic oil 3.7%, Emulsifier 2.7%	37.7-38.8			O,O,F	O,O,O	2.0	+	+	+
SP1 Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Glycol 13.0%, Thickener 0.7%	39.2-39.7	O,O,O	O			<1.0	-	-	-
SP2 Ammonium nitrate 55.0%, Sodium nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Glycol 14.0%, Thickener 1.0%	39.5-41.1	F	O,O,O			1.0	+	-	-
SP3 Ammonium nitrate 67.4%, Methylamine nitrate 15.0%, Water 12.0%, Glycol 5.0%, Thickener 0.6%	39.5-41.1	O,F	O,O,O			1.0	+	-	-
SP4 Ammonium nitrate 71.4%, Hexamine nitrate 14.0%, Water 14.0%, Thickener 0.6%	39.0-41.0		F	O,O,O		1.5	+	-	-
SP5 Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine nitrate 7.0%, Water 12.0%, Glycol 6.0%, Thickener 0.6%	39.6-39.9	F	O,O,O			1.0	+	-	-
SP6 Ammonium nitrate 68.4%, Methylamine nitrate 10.0%, Water 13.0%, Glycol 8.0%, Thickener 0.6%	39.9-40.1	O,O,O	O			<1.0	-	-	-

5. TESTS 1(c)(i) & 2(c)(i): TIME/PRESSURE TEST

5.1. Procedure

The tests have been carried out following the procedure described in sections 11.6.1 and 12.6.1 of Manual of Tests and Criteria, ST/SG/AC.10/11/Rev.3. Photographs 5.1 and 5.2 show the equipment used to carry out this test.

5.1. Apparatus and ignition system



5.2. Test setting

A Kistler piezoelectric gauge model 7005 connected to a charge amplifier Vibro-meter model TA-3/0 was used in these tests. The resulting signal was registered in a Tektronix digital osciloscope model DSA 601 of 1 GHz. The calibration factor for this system in the trial conditions was 1013 kPa/V. In order to ensure hermetic conditions, soft copper washers were used in all plugs of the test chamber.

The ignition system used consisted of an electric fusehead of the type commonly used in low tension detonators, covered with a 13 mm square piece of primed cambric. The primed cambric consisted of a linen fabric weighing approx. 79 g/m^2, coated on both sides with a pyrotechnic mixture with the following composition: potassium nitrate 40%, sulphur 40% and gunpowder without sulphur 20% (Defence Standard nº 07-5, issue 1). Once primed, the cloth had an approx. weight of 700 g/m^2.

In order to protect it from damp, the initiation system was covered with a thin sheet of PVC before the sample was added. In each test 5.0 g samples were used.

The test was repeated three times for each substance, and the less favourable result, highest maximum pressure and/or shortest time for a pressure rise from 670 kPa to 2070 kPa, is shown in Table 5.1.

The criteria to pass the test depend on the series. Thus, for Series 1 the result is positive if the maximum pressure is equal to or above 2070 kPa. In the case of Series 2, the result is positive if the maximum pressure is equal or above 2070 kPa and the time for a pressure rise from 670 to 2070 kPa is below 30 ms.

5.2. Results

The data and results of the tests are shown in Table 5.1. Figures 5.1 and 5.2 show as an example two pressure-time curves for two different suspensions.

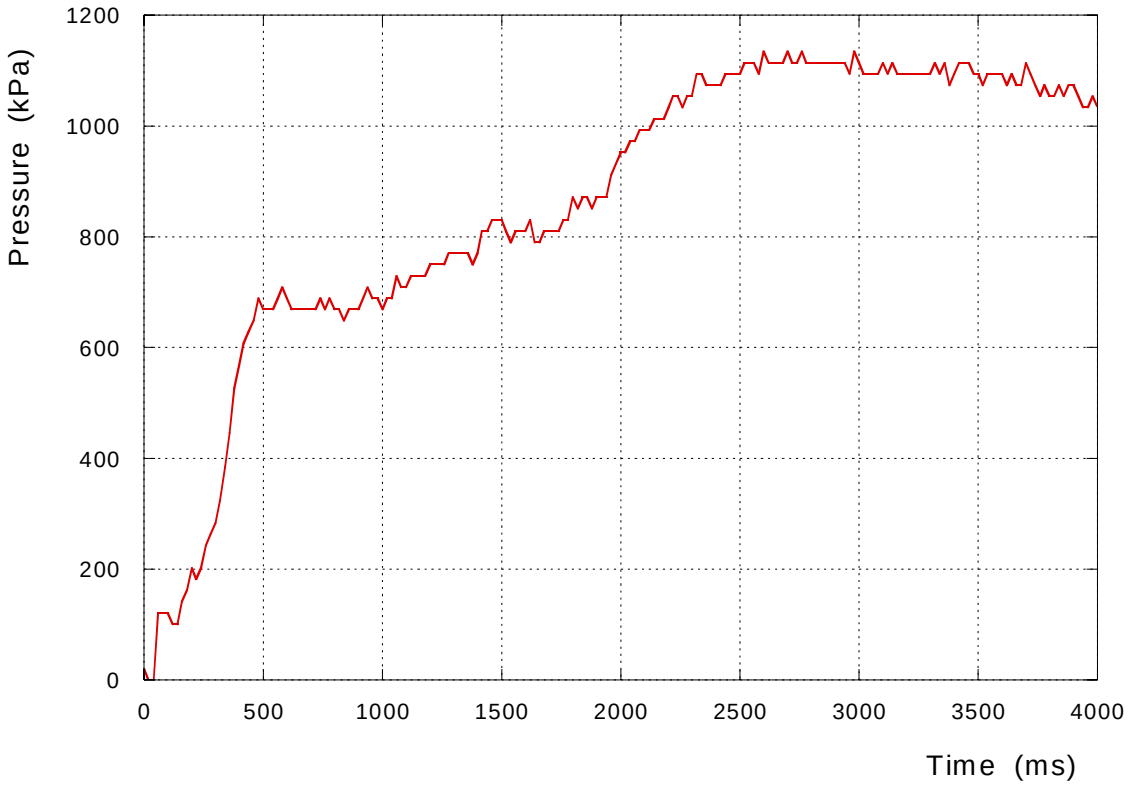


Figure 5.1. Pressure-time curve for suspension SP1

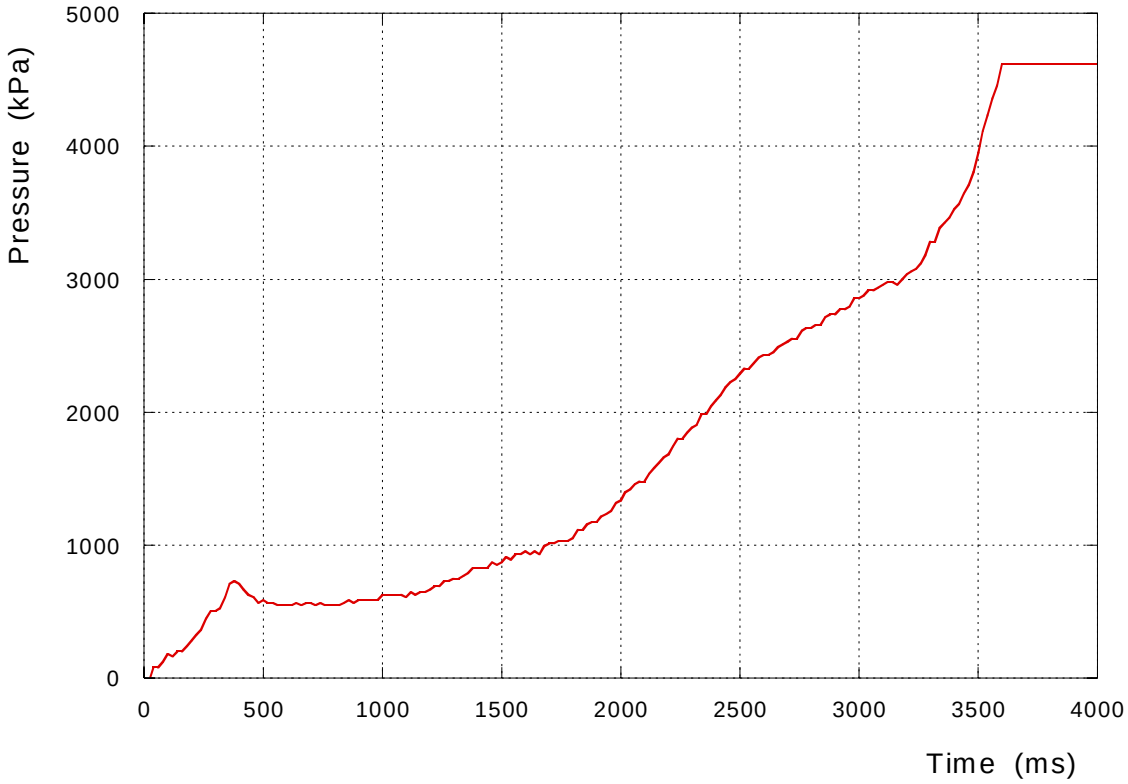


Figure 5.2. Pressure-time curve for suspension SP4

Contrary to what occurred with the EM1 and EM2 emulsions tested, three suspensions did not pass the Test 1(c)(i), although they passed with ease the Series 2 tests. The time required for the pressure to rise was in the order of seconds, whilst the test limit is set at 30 ms, indicating, therefore, a very slow decomposition.

Table 5.1. Tests 1(c)(i) & 2(c)(i): Time/pressure test

Substances	Time to reach the maximum pressure (ms)	Maximum pressure (kPa)	Time for a pressure rise from 690 to 2070 kPa (ms)	Result	
				Series 1	Series 2
EM1 Ammonium nitrate 76.0%, Water 17.0%, Paraffinic oil 5.6%, Emulsifier 1.4%	1190	567	-	-	-
EM2 Ammonium nitrate 82.1%, Water 12.3%, Paraffinic oil 4.2%, Emulsifier 1.4%	400	445	-	-	-
SP1 Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Glycol 13.0%, Thickener 0.7%	2760	1135	-	-	-
SP2 Ammonium nitrate 55.0%, Sodium nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Glycol 14.0%, Thickener 1.0%	>4000	>2500	3490	+	-
SP3 Ammonium nitrate 67.4%, Methylamine nitrate 15.0%, Water 12.0%, Glycol 5.0%, Thickener 0.6%	3370	1378	-	-	-
SP4 Ammonium nitrate 71.4%, Hexamine nitrate 14.0%, Water 14.0%, Thickener 0.6%	>3000	>4619	2060	+	-
SP5 Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine nitrate 7.0%, Water 12.0%, Glycol 6.0%, Thickener 0.6%	>2000	>4616	1280	+	-
SP6 Ammonium nitrate 68.4%, Methylamine nitrate 10.0%, Water 13.0%, Glycol 8.0%, Thickener 0.6%	8700	669	-	-	-

6. TEST 8(a): THERMAL STABILITY TEST

6.1. Procedure

The test has been carried out following the procedure described in section 18.4.1 of Document ST/SG/AC.10/29/Add.2, 17 February 2003.

6.1. Dewar vessel

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 obtain this volume, the lids were modified by sticking a piece of plastic to them so that the final volume was 0.5 l. A small hole was made in the lid through which a type 'T' thermocouple was inserted. Three identical Dewar vessels were prepared. Photograph 6.1 shows the described vessel.

The three vessels, each of them with its thermocouple, were introduced into an oven which had previously been checked as being capable of maintaining a 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 an electronic data logger, which allowed the temperature data to be transferred to the computer for later analysis.

In order to determine beforehand the heat loss characteristics of the system, the vessels were filled with hot water and, once closed and left at room temperature, the drop in water temperature was recorded as a function of time. As can be seen in Figure 6.1, the data could be fitted to three exponential curves from which the constants were determined.

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

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

where L (mW/kg·K) is the heat loss per unit of mass, Cp (J/kg·K) is the specific heat and $t_{1/2}$ (s) is the cooling half time, the heat loss for the three Dewar vessels was determined as the following values: 9.6 (1), 7.7 (2) and 8.1 mW/kg·K (3). These values are significantly lower than those required by the procedure (80-100 mW/kg·K), so they more than meet the requirements.

According to the procedure, the test has to be carried out at a temperature 20 °C above the maximum temperature which may occur during transport. Considering that, due to their 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. In this way the suspensions were also tested at the temperature normally used for testing emulsions.

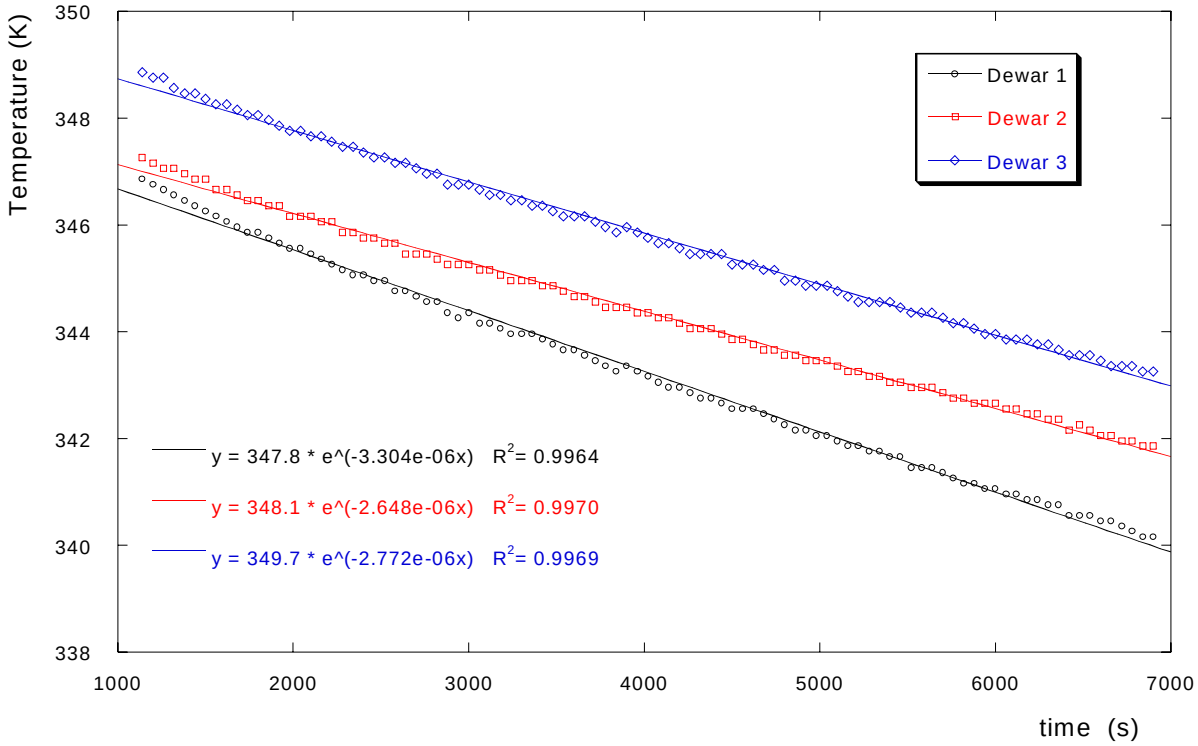


Figure 6.1. Cooling rates of the water contained in the three Dewar vessels

6.2. Results

Figures 6.2 and 6.3 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 6.1. 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 results of the suspensions tested were negative, and therefore all suspensions passed the test.

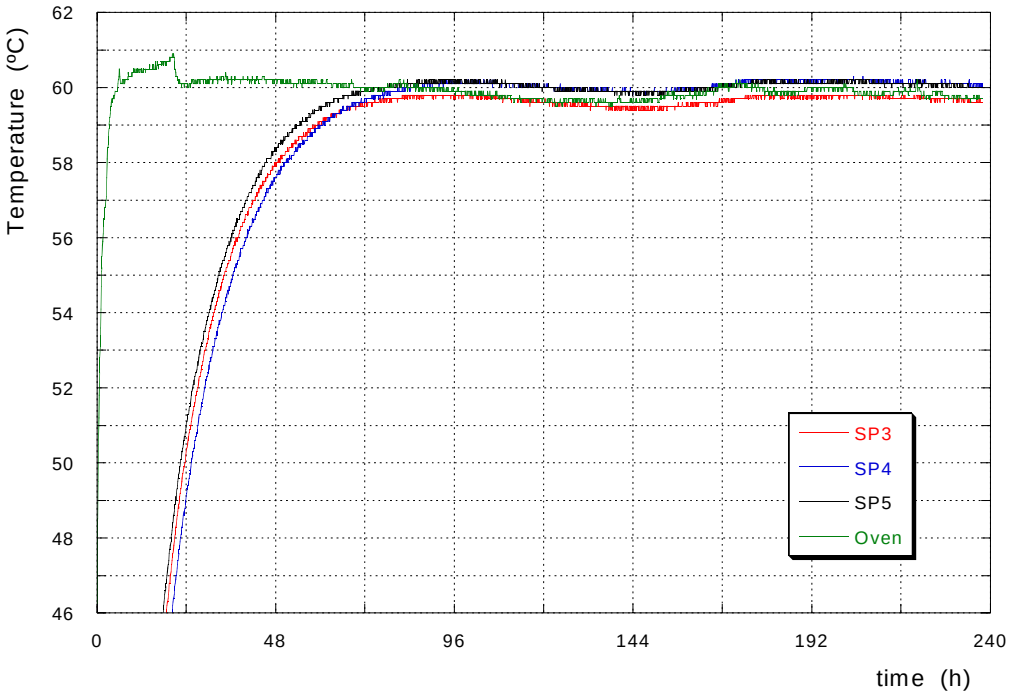


Figure 6.2. Record of temperatures for the test carried out at 60 °C

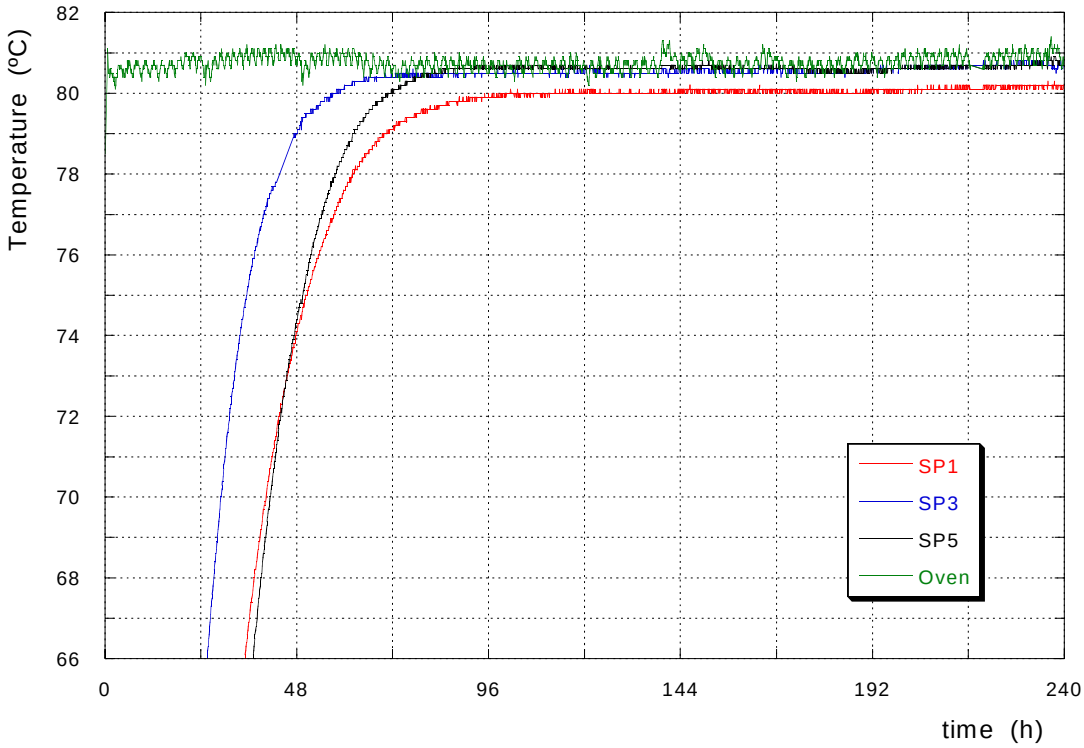


Figure 6.3. Record of temperatures for the test carried out at 80 °C

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Table 6.1. Test 8(a): Thermal stability test

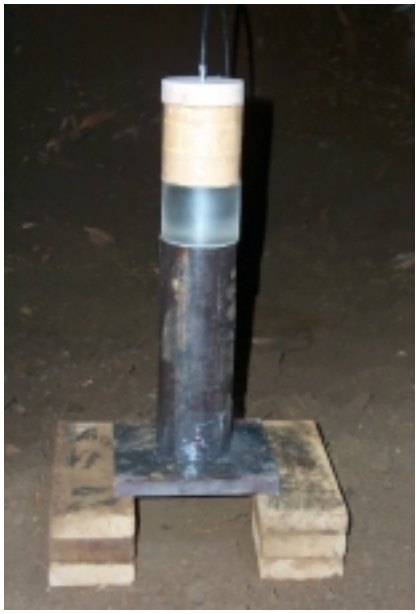
Substances	Sample mass (g)	$T_{AVE,OVE}$ (°C)	$T_{MAX,OVE}$ (°C)	Test time (h)	$T_{INI,SAM}$ (°C)	$T_{AVE,SAM}$ (°C)	$T_{MAX,SAM}$ (°C)	Δt ($T_{INI,SAM} \rightarrow T_{AVE,OVE}$-2) (h)	Δt ($T_{AVE-OVE}$-2 $\rightarrow T_{MAX,SAM}$) (h)	Mass loss (%)	Comments	Result
SP1 Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Glycol 13.0%, Thickener 0.7%	580.8	80.7	81.1	241.67	18.5	80.6	80.8	67.24	168.49	0.62	brown coloration crystal growth thickener break down	-
SP3 Ammonium nitrate 67.4%, Methylamine nitrate 15.0%, Water 12.0%, Glycol 5.0%, Thickener 0.6%	567.2	59.9	60.3	237.83	19.7	59.7	59.8	49.71	32.34	0.21	slight brown coloration crystal growth	-
	567.2	80.7	81.1	241.67	17.6	80.0	80.2	46.28	189.45	0.29	brown coloration crystal growth, thickener break down	-
SP4 Ammonium nitrate 71.4%, Hexamine nitrate 14.0%, Water 14.0%, Thickener 0.6%	582.4	59.9	60.3	237.83	19.5	60.1	60.2	52.05	40.35	0.19	yellow coloration crystal growth	-
SP5 Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine nitrate 7.0%, Water 12.0%, Glycol 6.0%, Thickener 0.6%	592.8	59.9	60.3	237.83	18.9	60.1	60.2	46.76	36.72	0.19	yellow coloration crystal growth	-
	595.2	80.7	81.1	241.67	18.3	80.6	80.8	60.62	176.21	0.50	yellow coloration crystal growth	-

$T_{AVE,OVE}$: average oven temperature. $T_{MAX,OVE}$: maximum oven temperature. $T_{INI,SAM}$: initial sample temperature, $T_{AVE,SAM}$: average sample temperature once stabilized.
$T_{MAX,SAM}$: maximum sample temperature. **Δt ($T_{INI,SAM} \rightarrow T_{AVE,OVE}$-2)** : time taken for the sample to rise from initial temperature to 2 °C below the oven temperature.
Δt ($T_{AVE,OVET}$-2 $\rightarrow T_{MAX,SAM}$) : time taken for the sample to rise from 2 °C below the oven temperature to its maximum temperature.

7. TEST 8(b): ANE GAP TEST

7.1. Procedure

The test has been carried out following the procedure described in section 18.5.1 of Document ST/SG/AC.10/29/Add.2, 17 February 2003. Photographs 7.1 and 7.2 show the setting of the different elements used to carry out this test.



7.1. Test setting

7.2. Test setting

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^3 and the masses ranged from 1064 to 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 according to the procedures.

7.2. Results

The data and the results of the different tests are shown in Table 7.1. Photographs 7.1 and 7.2 show two examples of the results obtained.

7.3. Emulsion EM1

7.4. Suspension SP1

In all cases the non-sensitised emulsions and suspensions pass the test. Nevertheless, the effect on the tube and the plate show different sensitivities among the tested substances.

When comparing the results for the different substances tested, it can be observed that all suspensions show less effects than the standard emulsion (EM1). The effect closest to that of the standard emulsion was that of the suspension SP3. In both cases the tube was fragmented and the plate was considerably domed; however, in the case of suspension SP3 product remains were found after the test, while this was not the case with the emulsion. In the case of the rest of the suspensions, the difference with regard to the standard emulsion is even greater: none of the tubes were fragmented, the plates remained virtually intact, and the residual substance remaining after the test was considerable.

To analyse these substances in depth in a shock wave, they were also studied once they had been sensitised by adding K1 microspheres from 3M. The minimum/maximum densities at which a negative/positive result is obtained were determined. The values obtained are shown in Table 7.2 together with the reduction in density necessary for the substance to give a positive result.

As in other tests carried out, it can be seen how the presence of sodium perchlorate or water-soluble amine nitrates in the suspensions do not imply a higher sensitivity than in a standard emulsion. Thus, while the standard EM1 emulsion presents a positive result at a density of 1280 kg/m^3, the SP1 suspension with 11,0% perchlorate needs to reduce its density to 1220 kg/m^3 to obtain a positive result. In the last column of Table 7.2, it can be seen that the reductions in density necessary to obtain positive results are always greater for all the suspensions tested when compared to the emulsions tested.

Table 7.1. Test 8(b): ANE gap test

Substances	Density (kg/m^3)	Gap (mm)	T (°C)	Tube	Plate	Product remains	Result
EM1 Ammonium nitrate 76.0%, Water 17.0%, Paraffinic oil 5.6%, Emulsifier 1.4%	1340	70	17	Fragmented in large pieces	Domed	No	-
EM2 Ammonium nitrate 82.1%, Water 12.3%, Paraffinic oil 4.2%, Emulsifier 1.4%	1370	70	20	Fragmented in large and medium pieces	Very domed	No	-
SP1 Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Glycol 13.0%, Thickener 0.7%	1450	70	19	Not fragmented	Slightly domed	Much	-
SP2 Ammonium nitrate 55.0%, Sodium nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Glycol 14.0%, Thickener 1.0%	1440	70	18-23	Not fragmented	Slightly domed	Much	-
SP3 Ammonium nitrate 67.4%, Methylamine nitrate 15.0%, Water 12.0%, Glycol 5.0%, Thickener 0.6%	1410	70	18-25	Fragmented in large pieces	Domed	Very little	-
SP4 Ammonium nitrate 71.4%, Hexamine nitrate 14.0%, Water 14.0%, Thickener 0.6%	1460	70	18	Not fragmented	Slightly domed	Much	-
SP5 Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine nitrate 7.0%, Water 12.0%, Glycol 6.0%, Thickener 0.6%	1480	70	17-19	Not fragmented	Domed	Little	-
SP6 Ammonium nitrate 68.4%, Methylamine nitrate 10.0%, Water 13.0%, Glycol 8.0%, Thickener 0.6%	1430	70	21	Not fragmented	No damage	Much	-

Table 7.2 Test 8(b) on sensitized substances

Substances	Matrix density (kg/m^3)	Minimum density of sensitized matrix for a negative result (kg/m^3)	Maximum density of sensitized matrix for a positive result (kg/m^3)	Density decrease to achieve a positive result (kg/m^3)
EM1 Ammonium nitrate 76.0%, Water 17.0%, Paraffinic oil 5.6%, Emulsifier 1.4%	1340	1300	1280	60
EM2 Ammonium nitrate 82.1%, Water 12.3%, Paraffinic oil 4.2%, Emulsifier 1.4%	1370	1340	1320	50
SP1 Ammonium nitrate 62.3%, Sodium perchlorate 11.0%, Water 13.0%, Glycol 13.0%, Thickener 0.7%	1450	1250	1220	230
SP2 Ammonium nitrate 55.0%, Sodium nitrate 8.0%, Sodium perchlorate 8.0%, Water 14.0%, Glycol 14.0%, Thickener 1.0%	1440	1190	1170	270
SP3 Ammonium nitrate 67.4%, Methylamine nitrate 15.0%, Water 12.0%, Glycol 5.0%, Thickener 0.6%	1410	1360	1340	70
SP4 Ammonium nitrate 71.4%, Hexamine nitrate 14.0%, Water 14.0%, Thickener 0.6%	1460	1220	1190	270
SP5 Ammonium nitrate 66.4%, Sodium perchlorate 8.0%, Hexamine nitrate 7.0%, Water 12.0%, Glycol 6.0%, Thickener 0.6%	1480	1320	1300	180
SP6 Ammonium nitrate 68.4%, Methylamine nitrate 10.0%, Water 13.0%, Glycol 8.0%, Thickener 0.6%	1430	1280	1250	180

8. TEST 8(c): KOENEN TEST

Since this test coincides with Test 2 (b) of Series 2, the corresponding trials have been included in Section 4.