TOCK (Type A)

UN Test Series 2 Explosive Properties Testing

Client	AMITY A.Ş.
Client location	Ankara, Turkey
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1. PROJECT DETAILS AND TEST WORK APPROVAL STATEMENT

Quotation Number	3016012466
Job Number	4028009530
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Reviewer	Dr Charles Baker, Laboratory Scientist
Peer Reviewer	Mr Paul Carter, Safety Testing Specialist
Study Initiation date*	08/11/2022
Start Date of Experimental Work	17/11/2022
Completion Date of Experimental Work	30/11/2022

* Sample, purchase order or last information receipt date, whichever is the latter.

This report has been issued in digital format. In order to ensure that the integrity of the data is maintained, the digital version at DEKRA UK will be considered the source document. All original test records are kept in a secure archive for a minimum of 10 years after the date of this report. Any remaining material(s) will be stored for a minimum of 1 month after the issue date of this report. This report was prepared by Ingrid Gledhill.

2. INTRODUCTION

This report contains test data for AMITY A.Ş. relating to the explosive properties of TOCK (Type A) which is a chemical mixture designed to be used inside a totally sealed TOCK Module Set. The set comprises of the chemical mixture and an electronic ignition component and is intended to be used for rock and concrete breaking. In this assessment testing has been conducted to determine the explosive properties of TOCK (Type A). The following tests were conducted in accordance with the methods described in the UN Manual of Tests and Criteria[1]:

- Gap test : UN Test 2 (a). Conducted by TNO.
- Koenen tube test : UN Test 2 (b)
- Time / pressure test : UN Test 2 (c)(i)

This work is in response to quotation number 3016012466. A formal hazard assessment of the process / plant has not been conducted by DEKRA Organisational and Process Safety and the consequences of specific process deviations have not been examined[2].

Detailed characterisation of the material tested in this study is provided in Section 3 of this report (with results summarised in Section 7)[3].

The material used in this assessment was supplied by AMITY A.Ş.

Mr Yusuf V Ozalp was present at DEKRA on 30th November 2022 to witness the Koenen and Time / Pressure testing.

3. SAMPLE INFORMATION

Product name	TOCK (Type A)
Composition	*This data is available within the scope of the original version of this document, ready to be shared under necessary conditions.* *It is removed with the aim to share where and when official presentation is required.*
Batch number	N/A
DEKRA sample reference	400031590
Appearance	Fine light brown powder

[1] Manual of Tests and Criteria, 7th revised edition, 2019, ISBN 978-92-1-130394-0 and Amendment 01 2021
[2] Process safety requires that all possible explosion, thermal stability and chemical reaction hazards are evaluated and that a suitable basis for safe operation is determined and implemented. Should the materials or processing conditions change then consideration should be given to re-assessment.
[3] A description of the test procedures together with full test results and information on their interpretation is given in the test sections of this report. The appendix provides background information on the subject matter. DEKRA Organisational and Process Safety's Laboratories are GLP (Good Laboratory Practice) compliant and this study was carried out to the principles of GLP.

4. UN GAP TEST

Test Objective and Information

The test is designed to measure the shock sensitivity and detonation propagation of a solid or liquid substance. The test is conducted according to the method described in the UN Manual of Tests and Criteria, Test 2 (a).

The sample is charged to a carbon steel tube with an external diameter of 48 ± 2 mm, a wall thickness of 4.0 ± 0.1 mm and a length of 400 ± 5 mm. If the test substance may react with steel, then the inside of the tube can be coated with fluorocarbon resin. The bottom of the tube is closed with two layers of 0.08 mm thick polythene sheet pulled tightly over the bottom of the tube and held in place with rubber bands. The booster charge consists of 160 g RDX / wax (95/5) or PETN / TNT (50/50), 50 ± 1 mm in diameter with a density of 1600 ± 50 kg/m^3 giving a length of 50 mm. A mild steel witness plate 150 ± 10 mm square and 3.2 ± 0.2 mm thick may be mounted at the upper end of the steel tube and separated from it by spacers 1.6 ± 0.2 mm thick. For UN Test 2 (a), a polymethylmethacrylate spacer is employed between the explosive charge and the test substance.

Solid samples are loaded into the tube to the density attained by tapping the tube until further settling becomes imperceptible. The sample mass is determined and the apparent density calculated. The density should be as close as possible to the shipping density. The tube is placed in a vertical position and the booster charge is placed in direct contact with the sheet which seals the bottom of the tube. The detonator is fixed in place against the booster charge and initiated. Two tests should be performed unless detonation of the sample is observed.

Generic equipment and test information is contained in Table 4.1.

Table 4.1: Equipment Configuration / Settings for UN Gap Test

Parameter	Setting / Configuration
Tube type	Carbon steel
Detonator type	No8 AI
Booster type	Debrix 13
Booster mass	160 g
Test temperature	Ambient

Interpretation of Results

The test results are assessed on the basis of the type of fragmentation of the tube and whether or not a hole is punched through the witness plate. The test giving the most severe result should be used for classification purposes.

For UN Test 2 (a), the test result is considered positive (+) and the substance to propagate a detonation if the tube is fragmented completely or a hole is punched through the witness plate. Otherwise the result is considered negative (-).

4.1 Test Results for TOCK (Type A)

Date	17/11/2022
Operator	W. Mak (TNO)
Test System	Gap System
Test Method	UN Test 2 (a)
Sample Preparation	Tested as received

Table 4.2: Full Test Results

Test	Apparent density ($kg.m^{-3}$)	Fragmentation length (mm)
1	1000	0
2	1000	0

The tube remained intact during both tests. The test result is therefore considered negative (-) (according to the UN Manual of Tests and Criteria).

Test Result: Negative

The full report from TNO is provided in Appendix 1.

5. KOENEN TUBE TEST

Test Objective and Information

The test is designed to measure the sensitiveness of solid and liquid substances to the effect of intense heat when under partial and defined confinement. The test is conducted according to the method described in the UN Manual of Tests and Criteria, Test 2 (b).

The apparatus consists of a steel tube (deep drawn from sheet steel) with a re-usable closing device which is installed in a heating and protective device. The closing device consists of a threaded collar and nut and contains an orifice through which gases from decomposition of the sample can pass. Orifice plates are available with diameters of 1.0, 1.5, 2.0, 2.5, 3.0, 5.0, 8.0, 12,0 and 20.0 mm. The heating device consists of four Bunsen burners positioned around the steel tube. The burners are lit simultaneously using an electrical ignition device. Propane is used as the fuel and the gas pressure is regulated to give a heating rate of 3.3 ± 0.3 $K.s^{-1}$. Since the tube is likely to rupture during the test heating is conducted in a protective welded box.

Normally substances are tested as received. Solid substances are filled into the tube in three increments of similar volumes. Each increment is then pressed with a force of 80 N applied to the total cross section of the tube. The tube is filled to a level 15 mm from the top of the tube. Liquids and gels are filled into the tube to a level 15 mm from the top of the tube. The mass of sample in the tube is recorded. The threaded collar is slipped onto the tube from below, the appropriate orifice plate is inserted and the nut tightened. The tube is suspended between two rods in the protective box and when all four burners are burning correctly, a stopwatch is started to measure the time elapsing between ignition of the burners and the occurrence of the first noticeable reaction of the substance. After each trial the fragments of the tube are collected and weighed. The types of fragmentation are shown in Table 5.1.

Table 5.1: Fragmentation Types

Type	Fragmentation	Test Result
O	Tube unchanged	No explosion
A	Bottom of tube bulged out	No explosion
B	Bottom and wall of tube bulged out	No explosion
C	Bottom of tube split	No explosion
D	Wall of tube split	No explosion
E	Tube split into two fragments	No explosion
F	Tube fragmented into three or more mainly large pieces	Explosion
G	Tube fragmented into many mainly small pieces, closing device undamaged	Explosion
H	Tube fragmented into many very small pieces, closing device bulged out or fragmented	Explosion

The series of trials is started with a single trial using an orifice plate of diameter 20 mm. If at 20 mm "no explosion" occurs, the series is continued with further trials using smaller orifice diameters until at one of these diameters the result "explosion" is obtained. Subsequently, further trials are conducted using increasing orifice diameters until only negative results have been recorded for three tests at the same orifice diameter.

The apparatus is shown below and generic equipment and test information is contained in Table 5.2.

Typical Koenen Tube Apparatus

Table 5.2: Equipment Configuration / Settings for Koenen Tube Test

Parameter	Setting / Configuration
Tube type	Steel
Orifice diameters	1 - 20 mm
Heating type	Bunsen burners (propane fuelled)
Heating rate	3.3 ± 0.3 $K.s^{-1}$

Interpretation of Results

The limiting diameter of a substance is defined as the largest diameter at which an explosion occurs. If a substance exhibits “no explosion” at 1 mm the limiting diameter is recorded as being less than 1 mm.

For UN Test 2 (b), the test result is considered positive (+) and the substance to show a violent effect on heating under confinement if the limiting diameter is 2 mm or more. Otherwise the result is considered negative (-).

5.1 Test Results for TOCK (Type A)

Date	30/11/2022
Operator	K. Arif
SOP Reference	SOP 310
Test System	Koenen System
Test Method	UN Test 2 (b)
Sample Preparation	Tested as received

Table 5.3: Full Test Results

Sample mass (g)	Orifice diameter (mm)	Time to first ignition (s)	Total test time (min)	Fragmentation type	Result
26.1	2	14	5	O	No explosion
26.1	1	16	5	A	No explosion
26.1	1	15	15	E	No explosion
26.1	1	17	17	E	No explosion

The sample has been shown to have a limiting diameter of < 1 mm. The test result is therefore considered negative (-) (according to the UN Manual of Tests and Criteria).

Test Result: Negative

6. TIME / PRESSURE TEST

Test Objective and Information

This test is designed to determine the effects of igniting a substance under confinement in order to determine if the ignition leads to a deflagration with explosive violence at pressures which can be attained with substances in normal commercial packages. The test is conducted according to the method described in the UN Manual of Tests and Criteria, Test 2 (c) (i).

The apparatus consists of a cylindrical steel pressure vessel sealed at one end with a firing plug and at the other end with a vent plug containing an aluminium burst disc (burst pressure approximately 2200 kPa). A soft lead washer is used with both plugs to ensure a good seal. A side arm is fitted to the vessel in order to connect a pressure transducer and a support stand holds the assembly in the correct attitude during use. The ignition system consists of an electric fusehead together with a 13 mm^2 piece of primed cambric which is a linen fabric coated on both sides with a pyrotechnic composition.

The apparatus is assembled complete with the pressure transducer and firing plug but without the aluminium bursting disk in position. A 5.0 g sample of the substance is then introduced into the apparatus so as to be in contact with the ignition system. Normally no tamping is carried out when filling the apparatus, unless it is necessary in order to get the 5.0 g charge into the vessel. For liquid samples the ignition system is covered with a thin piece of PVC sheathing. The lead washer and aluminium bursting disk are placed in position and the vent plug screwed in tightly. The charged vessel is then transferred to the firing support stand.

The data logging is commenced, a power supply is connected to the external terminals of the firing plug and the charge is fired. The signal produced by the pressure transducer is recorded by the data logging system (capable of recording data at 1 ms intervals).

The test is carried out three times. The time taken for the pressure to rise from 690 kPa to 2070 kPa above atmospheric is noted. The shortest time interval should be used for classification.

The apparatus is shown below and generic equipment and test information is contained in Table 6.1

Typical Time / Pressure Apparatus

Table 6.1: Equipment Configuration / Settings for Time / Pressure Test

Parameter	Setting / Configuration
Sample mass	5 g
Burst disc type	Aluminium, 30 mm diameter
Pressure range	0 – 2500 kPa
Data acquisition system	DASYLab®

Interpretation of Results

The test results are interpreted in terms of whether a gauge pressure of 2070 kPa is reached and, if so, the time taken for the pressure to rise from 690 kPa to 2070 kPa gauge.

For UN Test 2 (c) (i), the result is considered positive (+) and the substance to show the ability to deflagrate rapidly if the time for a pressure rise from 690 kPa to 2070 kPa is less than 30 ms. Otherwise the result is considered negative (-).

6.1 Test Results for TOCK (Type A)

Date	30/11/2022
Operator	K. Arif
SOP Reference	SOP 313
Test System	Time / Pressure System
Test Method	UN Test 2 (c) (i)
Sample Preparation	Tested as received

Table 6.2: Full Test Results

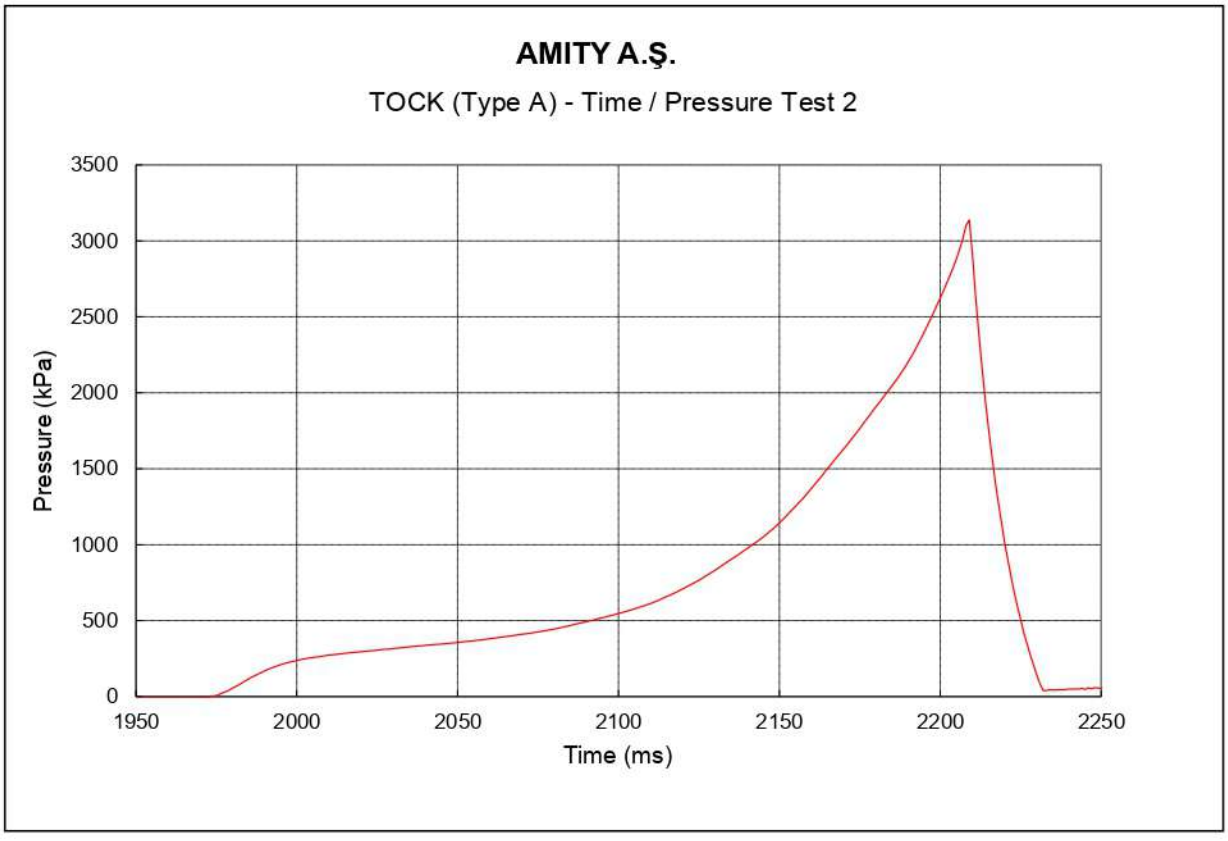
Test	Sample mass (g)	Maximum pressure (kPa)	Time from 690 to 2070 kPa (ms)
1	4.97	2717.3	72.7
2	5.10	3135.9	67.5
3	5.01	3137.2	69.6

Graph 6.1.a illustrates the time / pressure data from the most significant of the three tests.

The shortest time for a pressure rise from 690 to 2070 kPa was greater than 30 ms. The test result is therefore considered negative (-) and the substance to show slow deflagration (according to the UN Manual of Tests and Criteria).

Test Result: Negative

Graph 6.1.a: Pressure versus Time



7. SUMMARY OF TEST DATA OBTAINED

1. UN test series 2 testing was conducted on a sample of TOCK (Type A). The test results are summarised in Table 7.1.

Table 7.1: Summary of Test Results

Test	Result
UN Gap Test	Negative
Koenen Test	Negative
Time / Pressure Test	Negative

2. The sample is therefore exempt from classification as an explosive substance of UN Class 1.

8. APPENDIX 1

8.1 Copy of TNO Gap Test Report

TNO innovation for life

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TNO report

TNO 2022 R12428

Detonability of a sample of TOCK

Date	November 2022
Author(s)	Ing. W.A. Mak
Number of pages	8
Sponsor	DEKRA Process Safety, Southamtpon, United Kingdom
Project number	060.52062/01.37

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Summary

DEKRA Process Safety, Southampton, United Kingdom, requested TNO to assess the detonability of a sample of TOCK in accordance with the test method and criteria described in the United Nations Manual of Tests and Criteria, seventh revised edition, test series 2.

Based on the results of the study, it is concluded that the investigated sample of TOCK is not sensitive to detonative shock in the sense of the UN Manual of Tests and Criteria, test series 2.

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1 Introduction

DEKRA Process Safety, Southampton, United Kingdom, requested TNO to assess the detonability of a sample of TOCK in accordance with the test method and criteria described in the United Nations Manual of Tests and Criteria, seventh revised edition, test series 2.

The UN gap test (UN test 2(a)) was carried out.

2 Sample description[1]

Sample name	:	TOCK
Composition (SDS)	:	*This data is available within the scope of the original version of this document, ready to be shared under necessary conditions.* *It is removed with the aim to share where and when official presentation is required.*
Appearance	:	fine light brown powder
Supplier	:	AMITY A.Ş., Ankara, Turkey
TNO sample id.	:	22EM0323

The sample was received at the The Hague-Ypenburg location of TNO on 3 November 2022. Upon receipt, the sample was registered and stored at ambient conditions.
The sample was tested in the form as received.

[1] According to the information provided by the assignor.

3 Results

The detonative properties of the sample were determined using the UN gap test, as described in the United Nations Manual of Tests and Criteria, seventh revised edition, test 2(a).
A steel tube with a length of 400 mm, an outer diameter of 48 mm and a wall thickness of 4 mm was used. The booster charge consists of RDX/wax (95/5) and is placed under the bottom the tube. The booster charge is initiated using a detonator. Between the booster charge and the tube a gap is provided in the form of a PMMA spacer with a length of 50 mm.
The test results are assessed on basis of the fragmentation of the tube.
The results are shown in Table 1 and the tubes in Figure 1.

Table 1 Results of the detonation test

test	apparent density [kg/m^3]	fragmentation length [mm]	result
1	1,000	0	no detonation
2	1,000	0	no detonation

Figure 1 The tubes after the test.

4 Conclusions

Based on the test results and in accordance with the criteria specified in the UN Manual of Tests and Criteria, it is concluded that the investigated sample of TOCK is not sensitive to a detonative shock under the conditions of UN test series 2.

5 Signature

We, the undersigned, herewith declare that this report contains an accurate description of the results of the study.

The Hague

Dr. A. Burik
Research manager

TNO

ing. W.A. Mak
Project leader, Author

9. APPENDIX 2

9.1 Background Information – Classification of Explosives

The identification of potentially explosive or high rate decomposing properties in a substance is a pre-requisite for safe handling and transportation. Any substance which contains functional groups with known explosive properties (e.g. nitro, peroxy, azide etc.) should be tested to identify its reaction to various forms of explosion initiation. Initially, small scale tests are undertaken to assess the sensitivity of the substance to mechanical and thermal initiation. Additional tests are then necessary to assess the explosive properties of the substance when heated under confinement and exposed to detonable shock.

The most complete assessment methods for determining the explosive properties of a substance are provided by the United Nations[4]. These test methods are recognised by authorities within almost all countries of the world. The following rationale is based on the standard methods prescribed.

Initial Screening

An initial screening of the substance under study can be performed employing Benson's method of group contributions to estimate the potential for highly energetic decompositions or detonable properties. This is normally undertaken using the CHETAH[5] computer program which ranks substances as low, medium or high risk in terms of decomposition energy, based on the maximum enthalpy of decomposition and the oxygen balance. If this desk based screening exercise shows potential for an energetic decomposition then it is appropriate to conduct further experimental trials to determine the sensitivity of the substance.

Energy of Decomposition

Experimental testing will normally take the form of preliminary screening tests using small quantities of substance. Primarily, the substance will be tested for its ignitability, followed by Differential Scanning Calorimetry (DSC) to determine the quantity of heat evolved during a thermally initiated decomposition. Should the measured energy of decomposition be greater than 500 $J.g^{-1}$, the substance may have explosive properties. Consequently, it should only be handled in small quantities whilst further investigations are made.

UN Test Procedures

The following test series (as defined by the UN) should be followed in order to classify the substance as a candidate for inclusion as a Class 1 (Explosive) Substance (or to exclude the substance from Class 1). The above initial investigations should be conducted before proceeding with larger scale trials to gauge the likely hazards of the substance. Furthermore, the test series need not be completed in full or performed in the order in which they appear. For classification purposes it would be normal to conduct test series 3 first, since this uses the smallest quantities of substance and provides essential stability and sensitivity information required for larger scale handling and testing.
The standard test methods also provide excellent data for non-classification purposes (i.e. assessing the hazards of handling, storing or drying the substance). For example, sensitivity to friction should always be confirmed for a potentially energetic substance which requires milling.

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[4] Manual of Tests and Criteria, 7th revised edition, 2019, ISBN 978-92-1-130394-0

[5] The ASTM Computer Program for Chemical Thermodynamic and Energy Release Evaluation, version 9.0, 2009, ISBN 978-0-8031-7007-0

Table 9.1 and Table 9.2 give details of each test series, the tests involved and the approximate consequences of results. The UN Manual of Tests and Criteria should be consulted for a much more detailed description of the test methods and classification procedure. It should be borne in mind that explosive properties are highly scale dependent. For example, while a substance may not detonate in a test tube, it may detonate under similar conditions in a larger receptacle. For this reason, some of the latter classification tests are conducted on packages of the substance as it will be supplied for transportation (often implying significant sample test sizes).

Table 9.1: UN Test Series 1-3 Information

Test Series	Test Number	Test Name	Test Result
1	1(a) 1(b) 1(c)(i)	Gap test Koenen tube test Time / pressure test	Substance has explosive properties if any of the three tests gives a positive result
2	2(a) 2(b) 2(c)(i)	Gap test Koenen tube test Time / pressure test	Substance is a candidate for inclusion in Class 1 if any of the three tests gives a positive result
3	3(a)(ii) 3(b)(i) 3(c) 3(d)	BAM fallhammer BAM friction Thermal stability test at 75°C Small scale burning test	Substance is deemed too sensitive for transport in the form in which it was tested if any of the four tests gives a positive result

If the sample is still a candidate for inclusion in Class 1 after completion of the tests above, further tests are conducted as detailed in Table 9.2. UN Test Series 4 is conducted on substances which have failed any test of UN Test Series 3 to determine if they can be accepted into Class 1 after encapsulation or desensitisation.

Table 9.2: UN Test Series 4-7 Information

Test Series	Test Number	Test Name	Test Result
4	4(a) 4(b)(i) 4(b)(ii)	Thermal stability test for unpackaged articles and packaged articles Steel Tube Drop Test for Liquids Twelve Metre Drop Test for Unpackaged Articles, Packaged Articles and Packaged Substances	If the substance passes these tests, it can be considered for inclusion in Class 1 using the classification tests prescribed in Test Series 5, 6 and 7
5	5(a) 5(b)(ii) 5(c)	Cap Sensitivity Test USA DDT Test External Fire Test for Division 1.5	If the substance passes these tests, it is a candidate for classification as a Class 1.5 substance. If the substance fails any of the tests, Test Series 6 is required for further classification.
6	6(a) 6(b) 6(c)	Single Package Test Stack Test External Fire (Bonfire) Test	Classification from this test series can be complex due to the large number of outcomes. However, if the substance passes all three tests, it is excluded from Class 1.
7	7(a)-(k)	Ten tests in all (not individually specified here).	If the substance is found to be an extremely insensitive article, it can be excluded from Class 1.

A wide range of other UN classification tests can be conducted by DEKRA Organisational and Process Safety. These include, but are not limited to, the following:

UN Class 2+3 : Flammable gases / liquids classification tests
UN Class 4.1 : Flammable solids, self-reactive substances and solid desensitised explosives classification tests
UN Class 4.2 : Substances liable to spontaneous combustion classification tests
UN Class 5.1 : Oxidising substances classification tests
UN Class 5.2 : Organic peroxides classification tests
UN Class 8 : Corrosive substances classification tests

In addition, DEKRA Organisational and Process Safety are also capable of providing regulatory physico-chemical property tests to recognised international methods for the purposes of Notification of New Substances, etc.

For further details on any of the tests outlined above, or for further information on the range of services available from DEKRA Organisational and Process Safety, please do not hesitate to contact us.