FFI-RAPPORT

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Electrically heated tube test of MCX-8100 EMTAP 42 Test

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EMTAP 42 Test

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Summary

The EMTAP 42 electrically heated tube test has been performed with three different heating rates during the STANAG 4170 qualification of MCX-8100. With the fastest heating rate of 300^{o}C per hour, the tube opens after 41 minutes, at a temperature of 177.2^{o}C in the centre of the filling. Not all MCX-8100 was consumed. The reaction is a burn or degree 1 response.

With a heating rate of 60^{o}C per hour the tube reacts after 186 minutes with a temperature of 206.5^{o}C in the center of the filling. All the MCX-8100 was consumed. The reaction is a burn or degree 1 response.

With a heating rate of 10^{o}C per hour the tube reacts after 997 minutes, with a temperature of 201.5^{o}C in the center of the filling. Four fragments from the tube body were recovered. The temperature measurement in the filling shows a strong exothermic reaction taking place before it reacts. At the event temperature the temperature inside the explosive filling was 16.2^{o}C higher than at the outside tube surface. The reaction is a deflagration or degree 2 response.

All achieved results showed a mild reaction for all heating rates. One can therefore expect that live munitions containing MCX-8100 has the potential to fulfill the IM requirement for the tested threat.

Sammendrag

EMTAP test 42 elektrisk oppvarming har blitt gjennomført med tre forskjellige oppvarmingshastigheter for kvalifisering av komposisjonen MCX-8100. Med en oppvarmingshastighet på 300 ^{o}C per time åpnes røret etter 41 minutter ved en temperatur i sprengstoffet på 177,2 ^{o}C. Ikke alt sprengstoff reagerte. Reaksjonen er en brann eller grad 1.

Med en oppvarmingshastighet på 60 ^{o}C per time åpnes røret etter 186 minutter ved en temperatur på 206,5 ^{o}C i sprengstoffet. Alt MCX-8100-sprengstoff reagerte. Reaksjonen er en brann eller grad 1.

Med 10 ^{o}C per time åpnes røret etter 997 minutter ved en temperatur på 201,5 ^{o}C i sprengstoffet. Reaksjonen medfører fragmentering av røret i fire fragmenter. Temperaturmålingen i sprengstoffet viser en sterk eksoterm reaksjon. Når reaksjonen inntreffer, er temperaturen 16,2 ^{o}C høyere i sprengstoffet enn på utsiden av røret. Reaksjonen er en deflagrasjon eller grad 2.

Oppnådde resultater viser moderate reaksjoner ved alle oppvarmingshastigheter. Det er derfor å forvente at ammunisjon fylt med MCX-8100-sprengstoff vil ha potensial til å tilfredsstille IM-kravet for denne trusselen.

Content

Abbreviations

DNAN	2,4-dinitroanisole
DOSG	Defence Ordnance Safety Group
EMTAP	Energetic Materials Testing and Assessment Policy
HMX	octogen/1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane
IM	Insensitive Munitions
MCX	Melt Cast eXplosive
MCX-6002	TNT/NTO/RDX 34/51/15
MCX-6100	DNAN/NTO/RDX 32/53/15
MCX 8001	TNT/NTO/HMX 36/52/12
MCX-8100	DNAN/NTO/HMX 35/53/12
NTO	3-nitro-1.2.4 triazole-5-one
RDX	hexogen/1,3,5-trinitro-1,3,5-triazacyclohexane
STANAG	Standard Agreement
TMD	Theoretical Maximum Density
TNT	2,4,6-trinitrotoluene

1 Introduction

Norway has for some years studied melt-cast compositions for use as main charge fillers in different calibres (1-3). Both experimental and theoretical studies to characterize these compositions have been performed. MCX-8100 containing DNAN in addition to NTO and HMX is one of the compositions that has shown good properties as an IM-filler for large calibre munitions (4). Properties as detonation pressure and velocity have been determined (5). However, if MCX-8100 shall be applied as main filler, the composition needs to be qualified according to STANAG 4170 (6).

One required property to be characterized for qualification is explosive response when ignited (confined and unconfined). STANAG 4491 (7) describes approved or recommended tests or test methods for performing this characterization. From STANAG 4491 Norway has selected to use the UK tube tests as test vehicle for this characterization. The two tests we decided to perform were EMTAP 41 and EMTAP 42 (8).

In this report we will report on the EMTAP 42 electrically heated tube test. The qualification of MCX-8100 has been a collaboration between the manufacturer of the composition, Chemring Nobel AS, and the user of the composition, Nammo Raufoss AS. The production of the tube bodies, the end caps and testing has been the responsibility of Nammo Raufoss AS. Manufacturing of filler and filling it into the test vehicles has been performed by Chemring Nobel AS.

Norway has earlier used tube tests for qualification of DPX-6 (9). However, DPX-6 is a press filled composition. The tubes used for that qualification had a thinner end wall (9 mm) in the end caps and fewer threads. Some tests were performed with that tube design also for MCX-6100, without success. The high pressure inside the tube when DNAN melts resulted in a hole in the end caps. A response not accepted for a valid test. In the testing carried out in this report we have used a newer design of the tube vehicle from DOSG UK. The end caps now have a wall thickness of 13 mm and in addition the number of threads has been increased. With the new design either failure of the end caps or leakage of explosive filler has been observed.

The explosive response of MCX-8100 when ignited has been characterized by EMTAP 41 tube test – fast heating in (10). In this report we have performed EMTAP 42 tube test – electrically heated. In reference 11 and 12 similar tests were performed on MCX-6100. There is no specific requirement to fulfil to pass these tests. However, for munitions as 120 mm and 155 mm shells filled with these compositions, a mild reaction in the tube tests is preferable for these compositions and will increase the possibility to fulfil the IM requirements.

Before testing the filled tubes were X-rayed to investigate the quality and homogeneity of the fillings. In addition the densities of the fillings were determined by weighing the tube before and after being filled with MCX-8100 composition.

2 Experimentally

2.1 Casting test objects

The test objects were casted by Chemring Nobel AS.

For these test objects the MCX-8100 charge 168001 was used as filler. The empty tubes were stored in a heat cabinet at 100-102^{o}C over the night before filling took place with the filler holding the same temperature (100-102^{o}C). The filled tubes were then placed in the heat cabinet for 2 hours before cooling at ambient temperature 25^{o}C. During the cooling the top of the tubes were protected by an isolating hat. A picture of the tubes as we received them at FFI, is shown in Figure 2.1.

Figure 2.1 Tube No 11 to tube No 13 as received from Chemring Nobel.

The tubes were cleaned and the end surfaces corrected. Figure 2.2 shows tube No 11 to tube No 13 after this operation.

Figure 2.2 Tube No 11 to tube No 13 after cleaning and modification of the top surface.

2.2 Quality of the fillings

2.2.1 Density

Before finalizing the tubes for testing the weights were measured and the densities of the fillings determined. The results with regard to densities of the fillings are given in table 2.1.

Tube No	Weight Filled Tube (g)	Weight Empty Tube (g)[1]	Weight of Filling (g)	Filled with MCX-8100 Charge	Filling density (g/cm^3)[2]	To be tested in
11	2976.81	2636.0	340.81	168001	1.722	SCO
12	2975.17	2635.0	340.17	168001	1.719	SCO
13	2975.90	2635.0	340.90	168001	1.722	SCO

[1]Body+1 end cap. [2]Volume 197.9451 cm^3 calculated from drawing (diameter 31.5 mm - height 254 mm).

Table 2.1 Properties of the tubes to be tested by electrically heating tube test – EMTAP 42.

In the top the tubes have visual cracks. However, the densities of the fillings have low variation with an average of 1.721 $\pm$0.001 g/cm^3. TMD for MCX-8100 is 1.7650 g/cm^3. This gives an average filling percentage of 97.5.

2.2.2 X-ray

All tubes were x-rayed to control the fillings for unexpected defects.

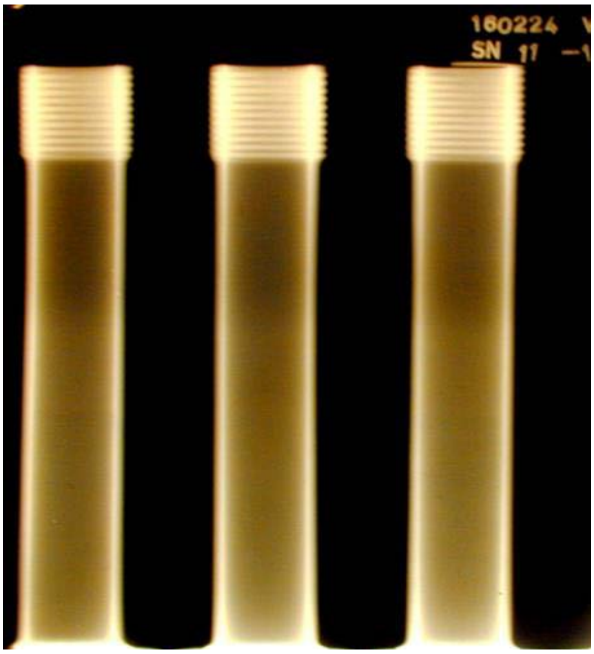


Figure 2.3 X-ray of tube No 11 to No 13 filled with MCX 8100.

Figure 2.3 shows a picture of the X-ray results. All tubes have some pores in addition to areas with lower density in the upper third of the filling. No large empty space is observed. These defects explain the moderate density of the fillings in Table 2.1.

2.3 Tube design

We received the drawings of the tube parts from UK DOSG. Copies of these drawings are shown in Appendix A. Our tube has the same dimensions. The only difference is the steel quality. The type of steel Nammo used in the production of the tube bodies and the end caps is given in Appendix B.

2.4 Test performance

The test "Tube Test – Electrically Heated (EMTAP test No 42)" was performed according to the description in the DOSG Manual of Tests (8) and STANAG 4591 (7).

2.4.1 Test item

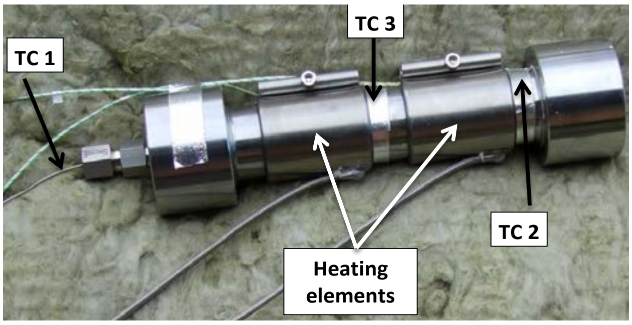
In all tubes space for installing the thermocouple were drilled in the center before the end cap was screwed in. The end cap contained a gland for the central thermocouple so the tube was tight even at elevated temperature. The test was performed inside a larger steel tube in a bunker as shown in Figure 2.4 and Figure 2.5.

Figure 2.4 Picture of the test setup.

Figure 2.5 Picture of test item with the two heating elements and three thermocouples.

2.4.2 Instrumentation

All tubes were equipped with three thermocouples calibrated in an oven before use.
Position of the thermocouples:



TC1: In the center of the explosive filling.

TC2: On the tube surface between the end cap and the heating element.

TC3: On the tube surface between the heating elements (used to regulate the heating).

Test equipment

Thermocouples:	TC1: Type K 1.5 mm (Inconel) length 10 m
	TC 2 and TC 3: Type K (Type C20KX)
Heating element:	Watlow MB01M2JX-1 230 V 300 W (2 units on each tube, 600 W)
Compensation cable:	Type K
Tape:	3M363
Data logger:	Agilent 34972 “TEMP-4” MY49004556
Laptop:	Lenovo ThinkPad T520 ID: NO88848
Software:	BenchLink Data Logger 3
Sampling rate:	1 Hz for 300^{o}C/hour
	0.5 Hz for 60^{o}C/hour
	0.1 Hz for 10^{o}C/hour

3 Results

3.1 Test No 16 – Tube No 11 - Heating rate 300^{o}C/hour

The first tube tested in the electrically heated tube test was tube No 11. Properties of the tube are given in Table 2.1. We wanted to have a heating rate of 300^{o}C/hour or 5^{o}C/min for this test. As described in reference (12) this was difficult to achieve. All isolation was removed except in the bottom of the steel protection tube, Figure 3.1. The effect of the heating was too strong to obtain a straight line. Transport of heat from the tube surface to the explosive filling took some time.

Figure 3.1 Pictures of the test item in the test environment seen from both sides of the test setup.

The temperature registrations on the three thermocouples during the test are shown in Figure 3.2. The programmed heating was at hold for 5 minutes or 300 seconds before activation. The reaction terminated after 2478 seconds or 41 min 18 seconds including the 5 minutes at ambient temperature

Before the temperature reached the event temperature some irregularities are observed in the temperature in the filler. At 89.55^{o}C an endothermic reaction took place and lasted until 90.8^{o}C. This reaction is interpreted as melting of DNAN. The temperature is slightly lower than expected.

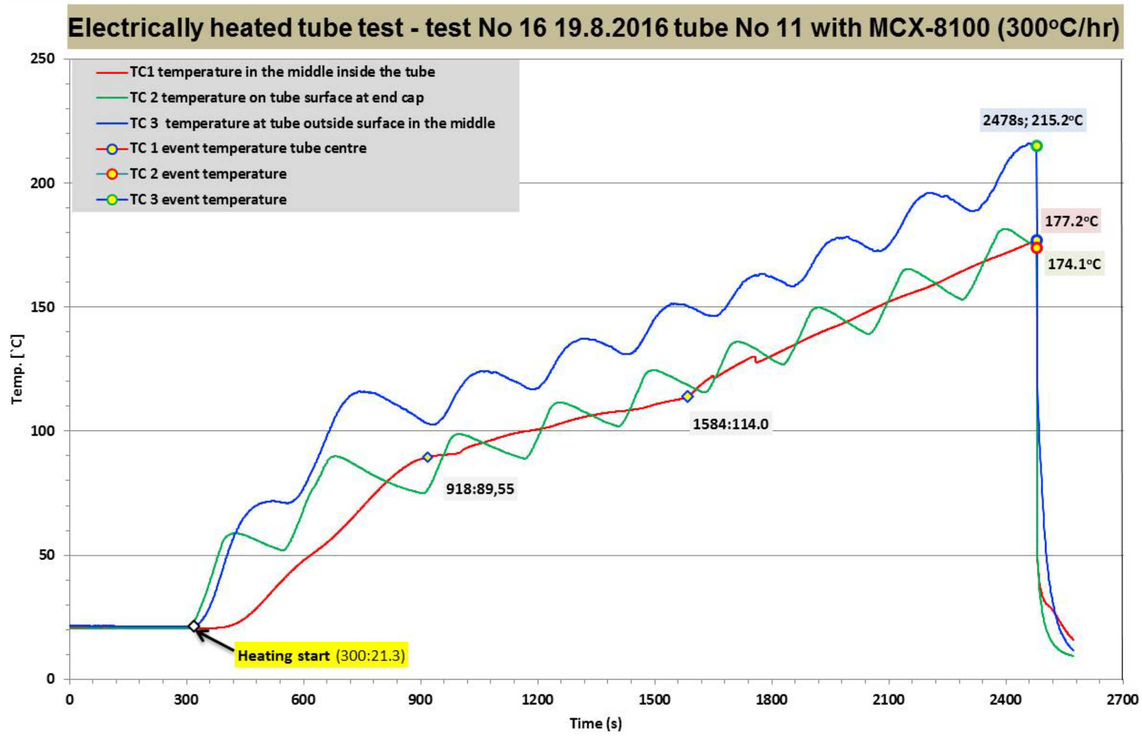


Figure 3.2 Temperature registrations for the three thermocouples in the electrically heated tube test with heating rate 300°C/hr. for tube No 11 filled with MCX-8100.

The reaction of the tube took place at 177.2°C. The left picture in Figure 3.3 shows the test tube in the test chamber after the reaction. The right picture in Figure 3.3 shows that the tube only has been split open without producing any fragments. The weight of the recovered tube was 3771.08 g. The weight of the empty tube was 3758.6 g. The extra weight comes from soot and rest of the explosive filling.

Figure 3.3 The left picture shows the tube inside the test tube after reaction. The right picture shows the tube after reaction.

3.2 Test No 18 – Tube No 12 - Heating rate 60°C/hour

The second tube with MCX-8100 filling tested in the electrically heated tube test was tube No 18. For this tube a heating rate of 60°C/hour was used. Figure 3.4 shows the observed temperatures on the three thermocouples placed inside the tube and on the tube outside surface during the test. From Figure 3.4 we see that only the thermocouple on the tube surface close to the end cap (TC 2) deviate significantly from the 60°C/hour heating rate. TC 1, the thermocouple in the center of the filling, shows that different reactions take place in different temperature ranges, Figure 3.5, during the heating of the tube. At 91°C there is an endothermic reaction due to melting of DNAN. At 114.3°C a new endothermic peak is observed. We have at the moment no explanation of what this reaction might be. From 180°C the temperature inside the tube is equal or higher than on the outside of the tube due to an exothermic decomposition reaction. The reaction started slowly but with increasing strength, and from approximately 190°C the temperature in the filling deviates more and more from the temperature on the outside tube surface. At 206.5°C in the explosive filling and 201.3 °C on the tube surface the tube collapsed. The explosive ignited and the filler was consumed.

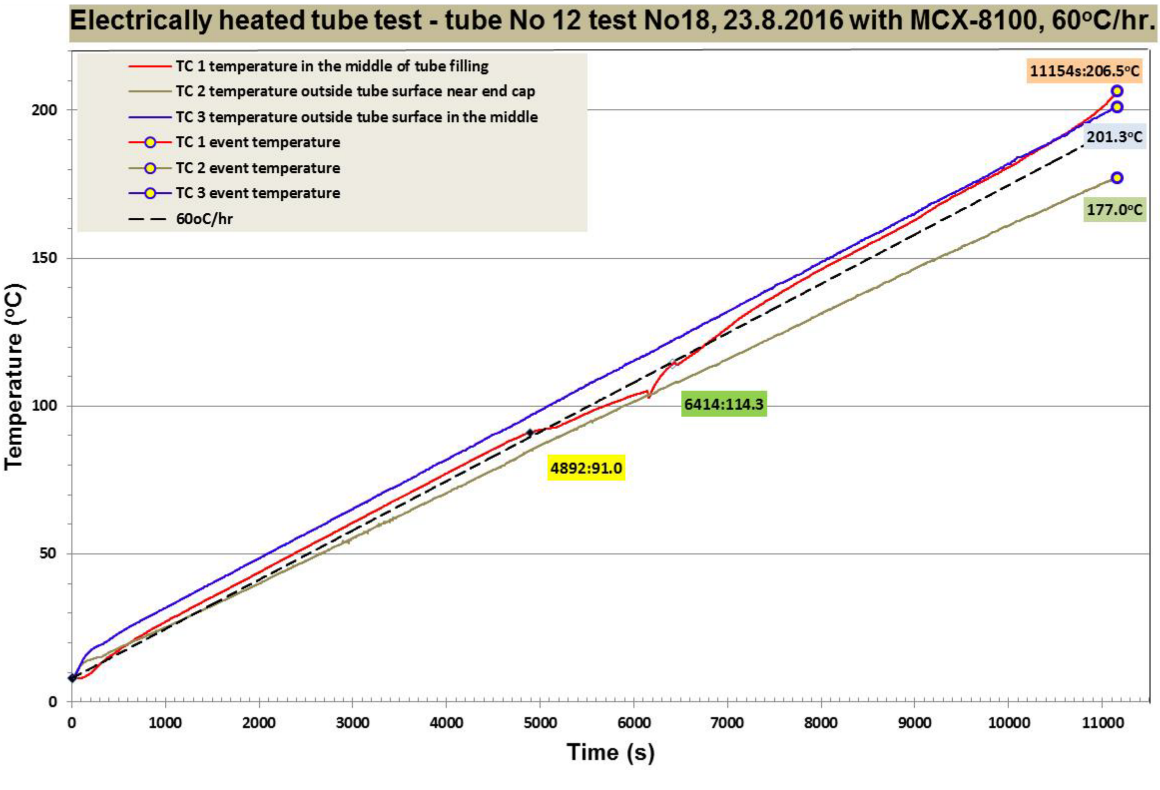


Figure 3.4 Temperature registrations for the three thermocouples in the electrically heated tube test for tube No 12 filled with MCX-8100.

Figure 3.6 shows the tube after reaction of the explosive filler. The weight of the recovered tube was 3629.37 g. The empty tube had a weight of 3757.63 g. This indicates that at least one fragment is missing. The recovered tube did not contain unreacted explosive.

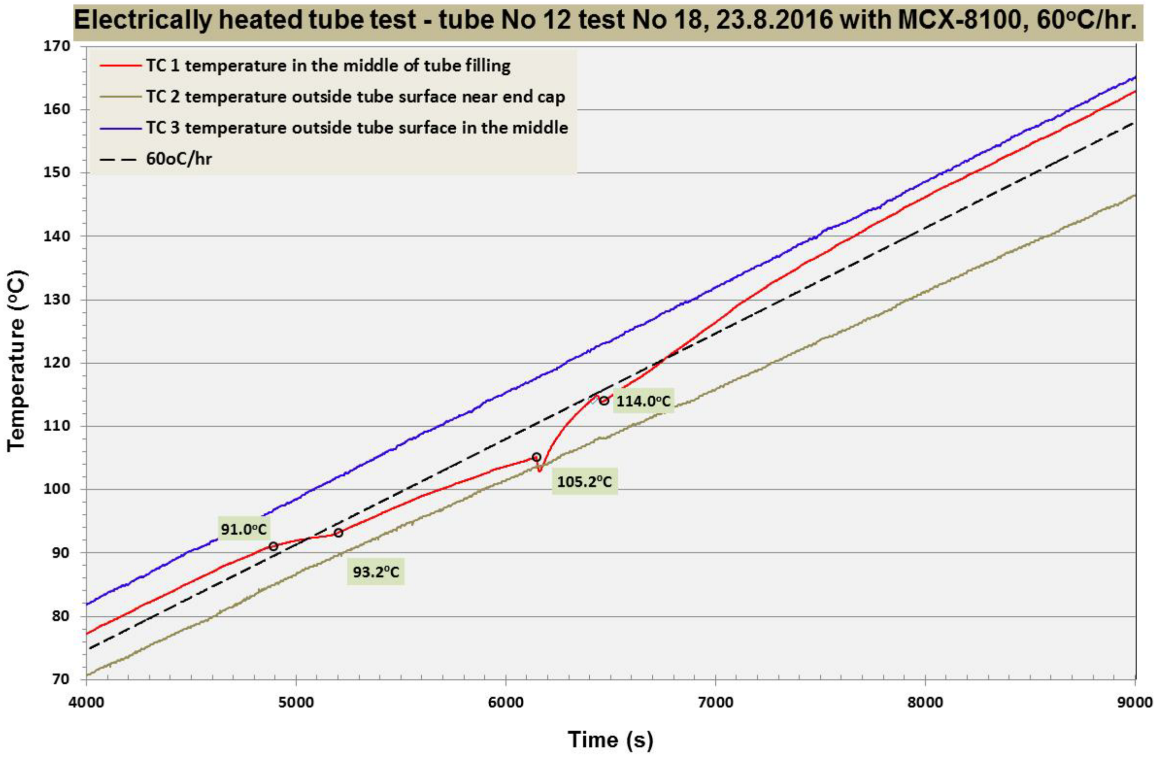


Figure 3.5   Temperature registrations for the three thermocouples in the electrically heated tube test for tube No 12 filled with MCX-8100.

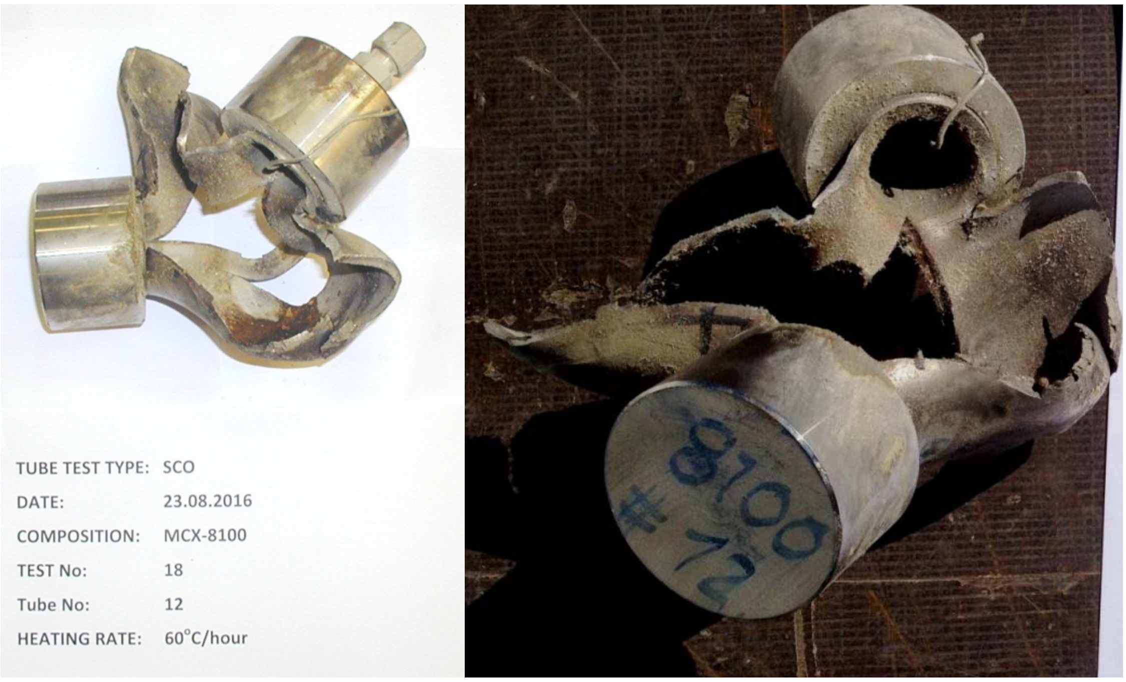


Figure 3.6   Recovered test vehicle No 12 after reaction in tube test No 18.

3.3 Test No 20 – Tube No 13 - Heating rate 10°C/hour

The last tube filled with MCX-8100, No 13, was heated with 10°C/hour. Figure 3.7 shows the registered temperatures on the three thermocouples during the heating process. The temperature on the tube surface, blue curve, follows the black curve (10°C/hour), the required temperature to fulfill the wanted test condition. For the green curve, the temperature at the tube outer surface close to the end cap – TC 2, a deviation from the black line is observed from start to the end of the test. At the event temperature this difference is 10.4°C.

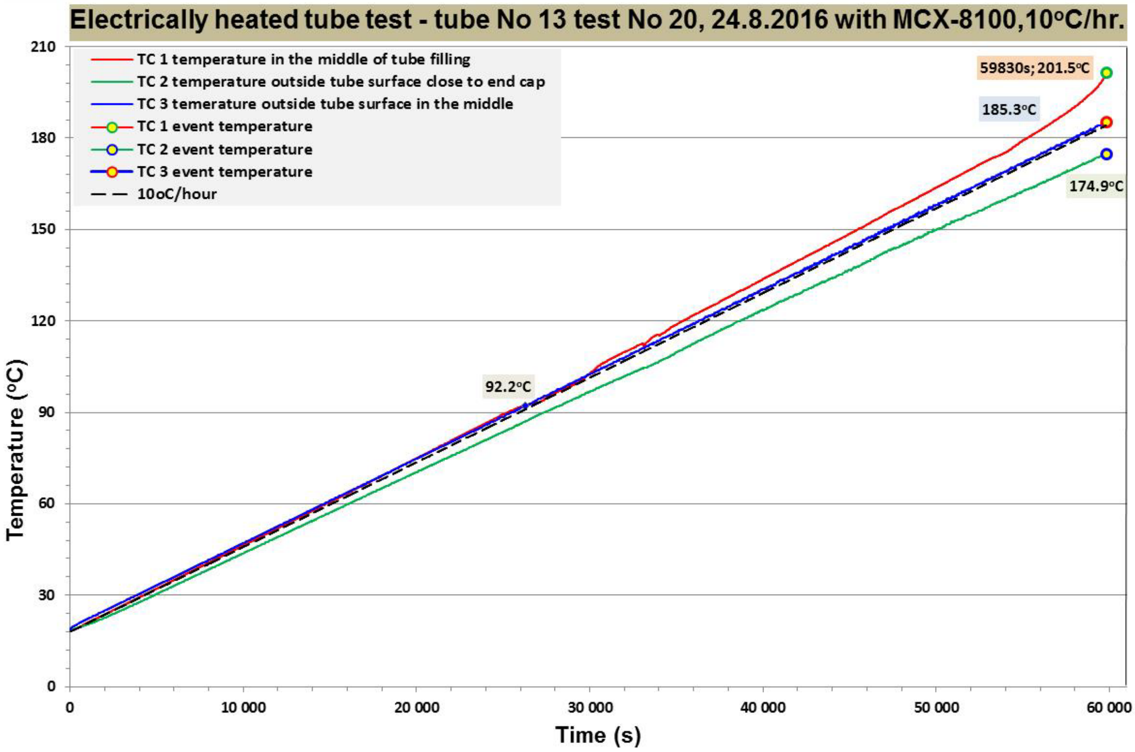


Figure 3.7 Temperature registration for the three thermocouples in the electrically heated tube test for tube No 13 filled with MCX-8100.

For the red curve, the temperature in the center of the filling, there are several deviations from the straight line showing that different processes are occurring in the filling. At the event temperature 201.5°C an exothermic reaction starting slowly at 120°C has taken place. At approximately 180°C the intensity of this exothermic reaction increases, and at the event temperature the temperature in the explosive filling is 16.2°C higher than on the outer tube surface.

Figure 3.8 shows the measured temperatures on the three thermocouples from 80°C to 120°C. In this temperature range several endothermic reactions in the MCX-8100 filling are observed. The first one starting at 92.3°C and ending at 94.4°C interpreted as melting of DNAN. The last two endothermic reactions start at 112.7°C and 115.5°C. We have no explanations for these reactions. From 100.1°C to 105°C an exothermic reaction takes place.

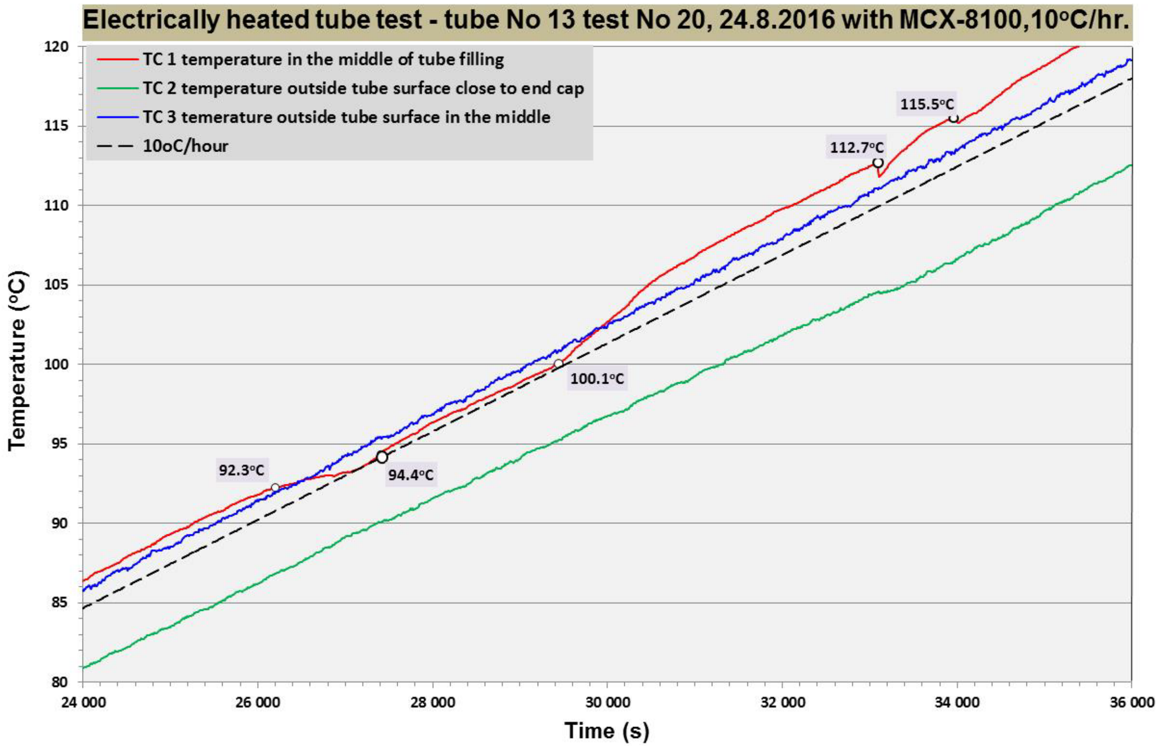


Figure 3.8 Temperature registrations for the three thermocouples in the electrically heated tube test for tube No 13, filled with MCX-8100, in the temperature range 80-120°C.

In Figure 3.9 one can observe the recovered fragments after reaction had taken place. 6 fragments including the end caps were recovered. The end cap with hole shows that the gland for the thermocouple is missing. The gland has a weight of 81.52 g. Adding this to the recovered weight, we totally get 3751.62 g. Comparing this number with the weight of the empty tube of 3757.6 g indicate that we should not expect to find more fragments than those recovered.

Table 3.1 The table gives the weight of recovered fragments for tube test No 20.

Fragment	
No	**Weight (g)**
1	1373.75
2	1350.45
3	479.68
4	247.54
5	194.32
6	24.56
All	**3670.30**

Figure 3.9 Picture of recovered fragments for tube test No 20 of tube No 13 with MCX-8100 filler.

3.4 Summary of the test results

In the test description (8) the guidance below for interpreting the results is given.

For all tests, the relative *explosiveness* of the composition under the test conditions is assessed from the degree of fragmentation of the tube body, not end caps.

Degree 0 No reaction
Degree 1 Burning
Degree 2 Deflagration, 2-9 fragments of tube body
Degree 3 Explosion 10 to < 100 fragments
Degree 4 Detonation >100 fragments
NB: End cap fragments not counted

By using this guidance we obtain the results given in Table 3.2 and 3.3 for MCX-8100 CH 1680001. For heating rate 300^{o}C/hour and 60^{o}C/hour the degree of reaction is a degree 1. For the slowest heating rate 10^{o}C/hour we get a deflagration reaction or degree 2. For the second test the weight of the recovered fragment is less than the weight of the empty test vehicle. We however have chosen to interpret the test results from the recovered items and not what is missing.

Test No	Tube No	Tube weight empty (g)	Heating Rate (^oC/hour)	Event temperature (^oC)			Time to event (s)	Number of fragments	Weight of recovered fragments (g)	Degree of reaction
				TC 1 Inside tube	TC 2 Outside end cap	TC 3 Outside tube middle				
16	11	3758.6	300	177.2	174.1	215.2	2478	1	3771.08*	1
18	12	3757.6	60	206.5	177.0	201.9	11154	1	3629.37	1
20	13	3757.6	10	201.5	174.9	185.3	59830	6	3670.30	2

*Contains rests of the filling.

Table 3.2 Summary of the results for the EMTAP test No 42 tube test electrically heated of MCX- 8100 CH 168001.

Round No	Vehicle No	Time Date	Filling Density (g/cm^3)	Recovered fragment			% Filling Recovered	Degree of Reaction	Time to Event (minutes)	Comments
				Total No	Body No	%Wt				
16	11	180816	1.722	1	1	100	3.7	1	41	
18	12	230816	1.719	1	1	96.59	0	1	186	
20	13	240816	1.722	6	4	100	0	2	997	

Table 3.3 Summary of the results for the EMTAP test No 42 tube test electrically heated of MCX 8100 CH 168001.

The reaction temperature of the MCX-8100 fillings increase as the heating rate is reduced. The degree of reaction goes from degree 1, burn, to degree 2, deflagration, when the heating rate decreases from 300^oC/hour to 10^oC/hour. In (12) MCX-6100 was tested in EMTAP 42 with event temperatures of 162.7, 194.9 and 200.3^oC with heating rates 300, 60 and 10^oC/hour. Compared with these results the observed event temperature with the 10^oC/hour for MCX-8100 may be slightly lower than expected. But it's difficult to conclude from only 6 tests. However, the event temperatures for MCX-8100 for all heating rates are higher than for MCX-6100. With regard to explosiveness MCX-8100 gives equal or lower reaction degrees. The number of fragments is lower for the two slowest heating rates.

Appendix

A Tube drawings

A.1 Of the body and end caps

We received the following drawings of the tube test vehicle from UK DOSG.

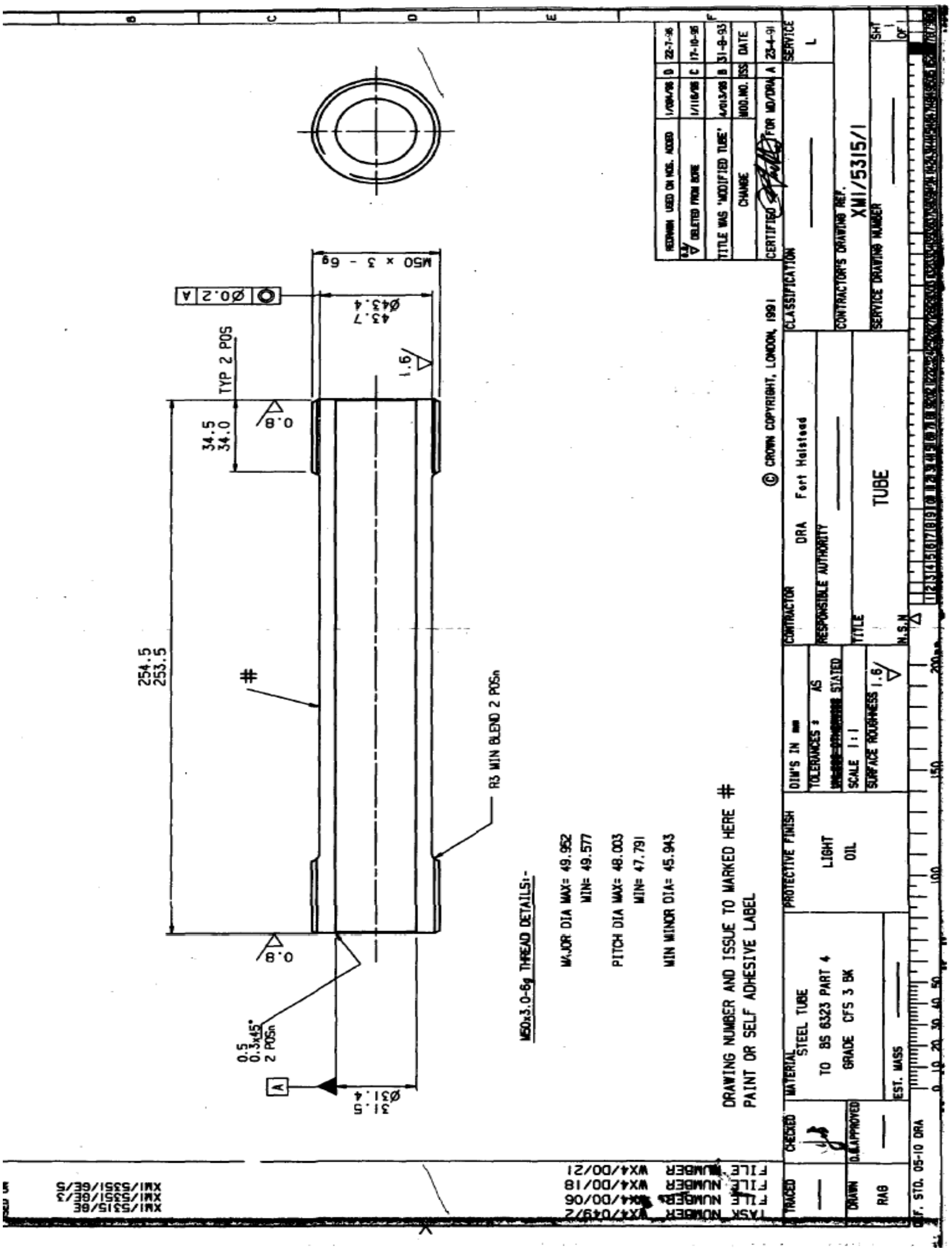


Figure A.1 Drawing of the tube body.

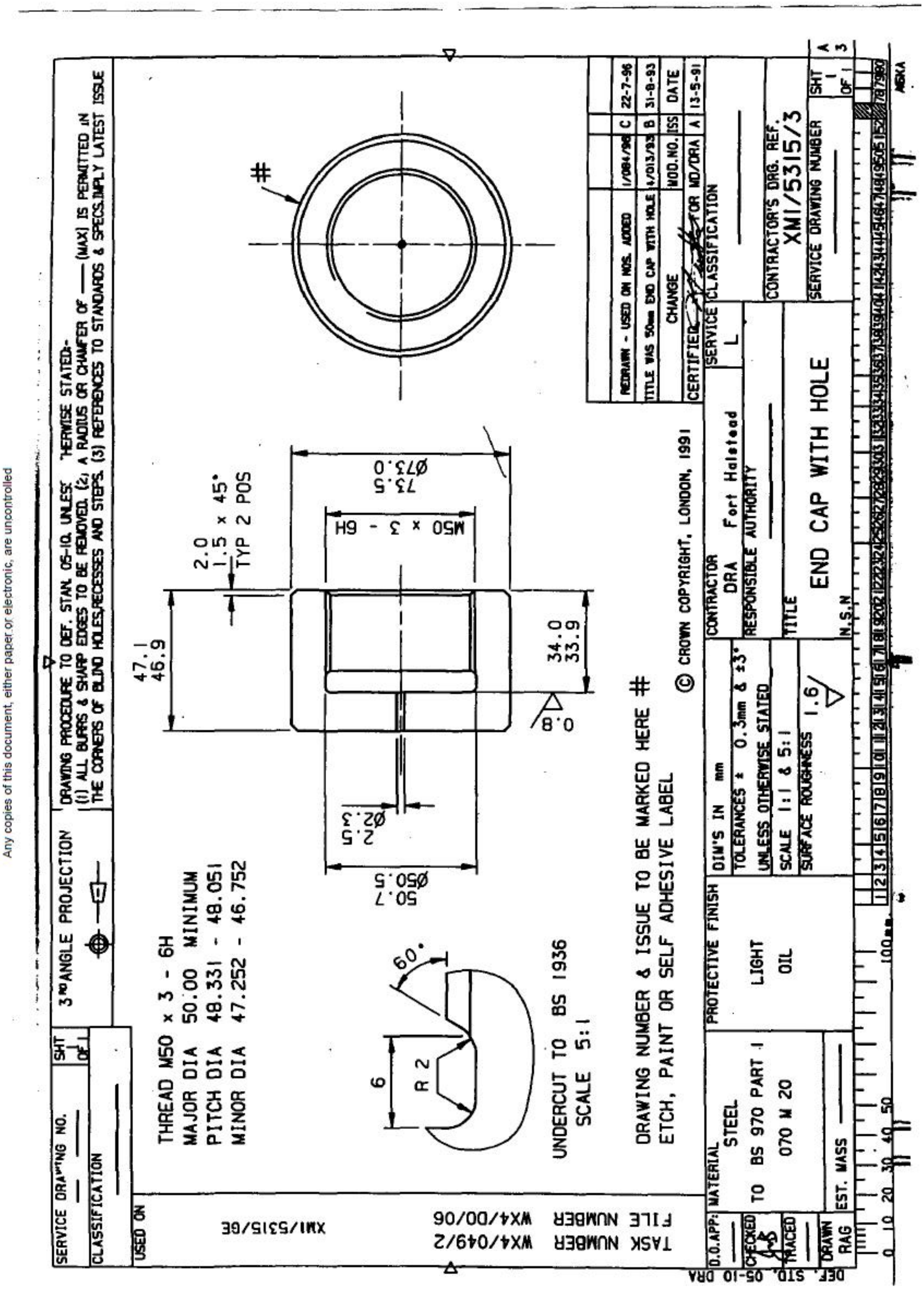


Figure A.2 Drawing of the end cap with hole.

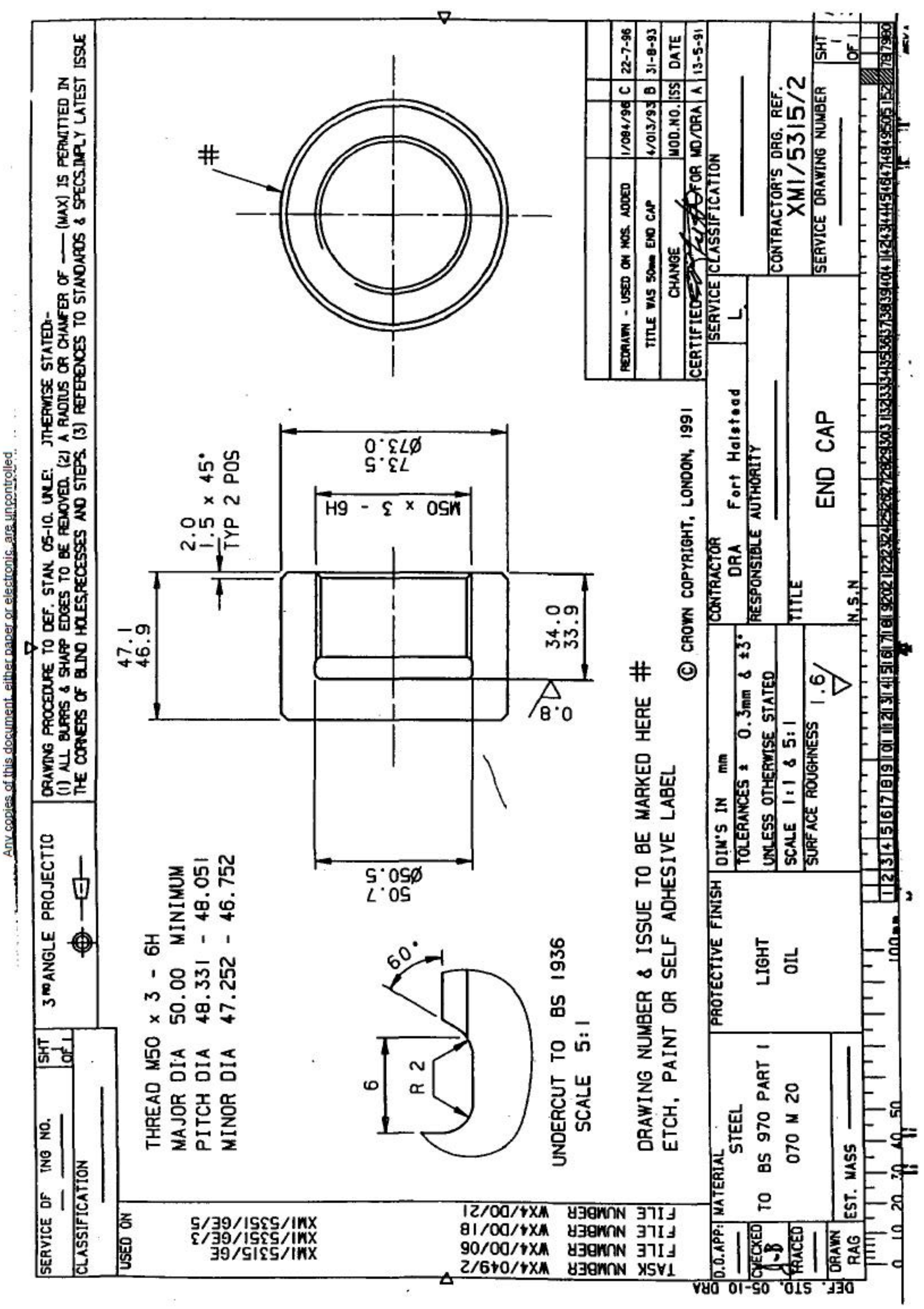


Figure A.3 Drawing the end cap and thread dimension.

A.2 Electrically heated design

The original tube design is shown in Figure A.4 with accompanying parts list in Figure- A.5.

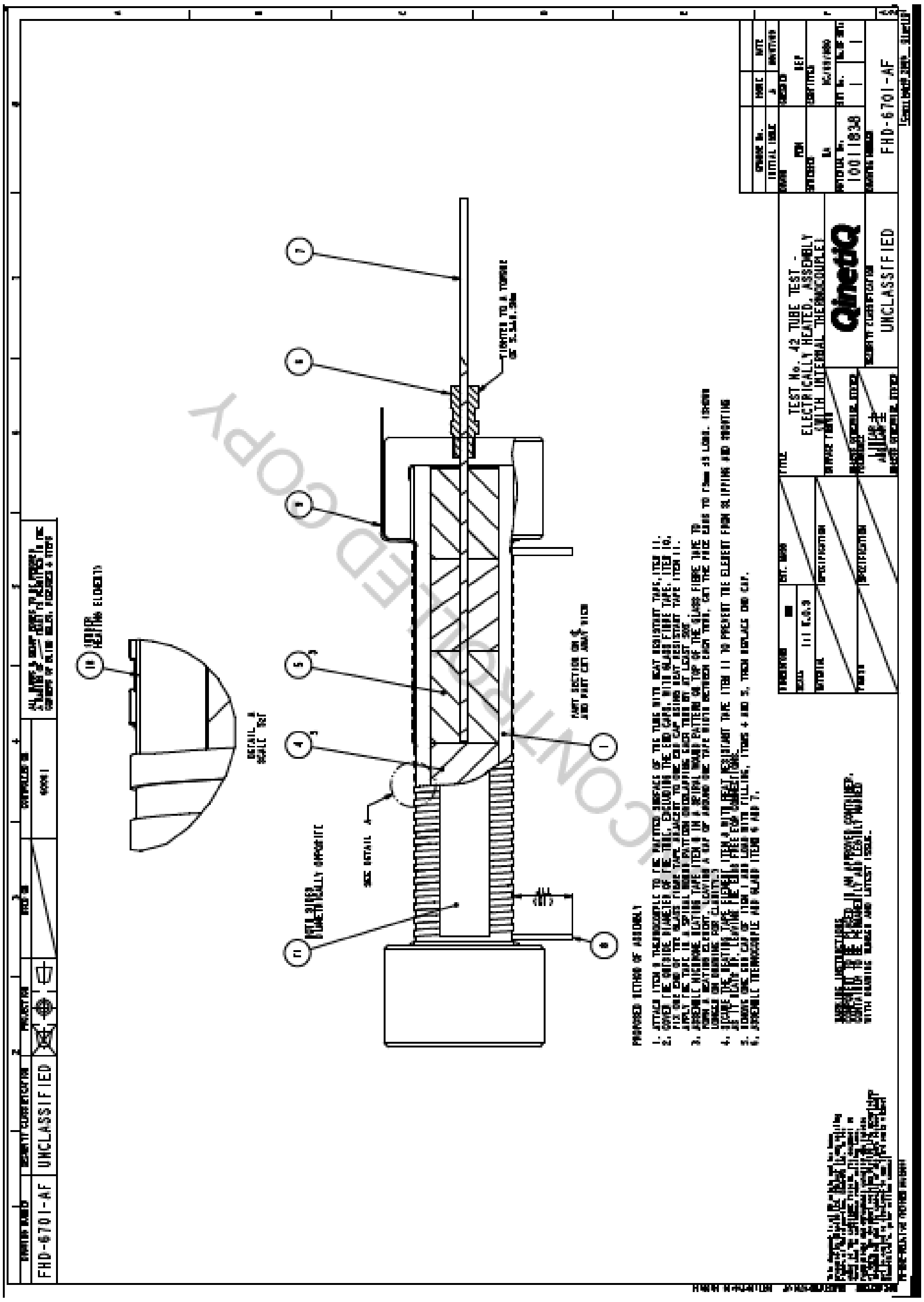


Figure A.4 Electrically heated tube design.

SECURITY CLASSIFICATION	CONTROLLED ON	USED ON
UNCLASSIFIED	60001	

ITEM	DRAWING No.	TITLE	No OFF	REMARKS
1	FHD-7206-SE	TUBE ASSEMBLY EMPTY		
		(WITH THERMOCOUPLE)	1	MATL No 10012179
4	XMI-5351-1	PELLET	3	MATL No 10007888
5	FHD-7188	PELLET	3	MATL No 10012080
6	ND	BRASS COMP GLAND, 1/8"BSPT 3mm ID		
		RS No 286-721	1	
7	ND	THERMOCOUPLE K TYPE S/STEEL		
		3mm DIA RS No 219-4387	1	
8	ND	HEATING TAPE Nikrothal60 3mm x 0.10mm		KANTHAL LTD, CANAL ARM FESTIVAL PARK, STOKE-ON-TRENT
		3.93 ohm/m RESISTANCE.	AS REQD	OR EAST ANGLIAN FINE WELD FOR SMALL QUANTITIES.
9	ND	THERMOCOUPLE FINE WIRE TYPE K		
		FIBREGLASS RS No 409-4920	1	
10	ND	GLASS SCRIM TAPE, 50mm WIDE,		CHESHIRE RIBBON, KINGSTON
		PRODUCT CODE GS1053/050/50.	AS REQD	MILLS, MANCHESTER Rd, SK14 2BZ.
11	ND	HEAT RESISTANT POLYAMIDE TAPE, COLOUR		
		GOLD 36 YARDS, 1 INCH WIDE RS 216-2302	AS REQD	

DRAWN	CHECKED	CERTIFIED
PBM	NEP	DC/09/080

TITLE
TEST No. 42 TUBE TEST -
ELECTRICALLY HEATED, ASSEMBLY
(WITH INTERNAL THERMOCOUPLE)

CHANGE No	DATE	ISSUE
	08/07/09	A

QinetiQ

SECURITY CLASSIFICATION:
UNCLASSIFIED

No. OF SHEETS 1

MATERIAL No.	ITEM LIST FOR	SHT. No.
10011838	FHD-6701-AF	1

A4

NAME:NEPENNY OBJECT:FHD-6701-AF-1L DATE:25-Feb-10 14:33:46

FH-PROE-REL3-1L (REVISED 03/2009)

Figure A.5 Specification of the different components of the electrically heated tube.

B Nammo tube design

B.1 Material applied

Not all materials specified in the drawing from UK were available in Norway. To replace these, similar materials were selected, and the specifications of these are given in Figure B-1.

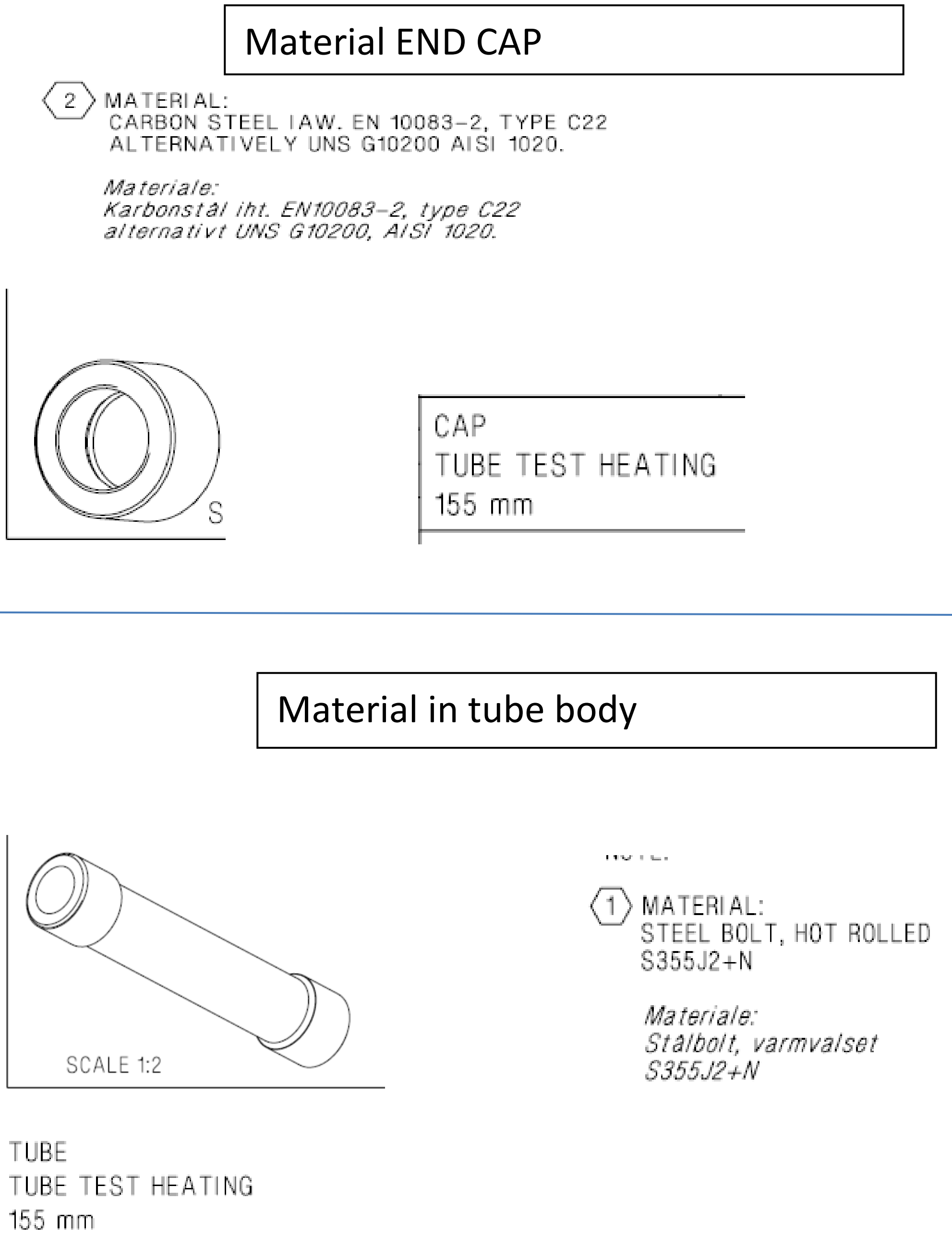
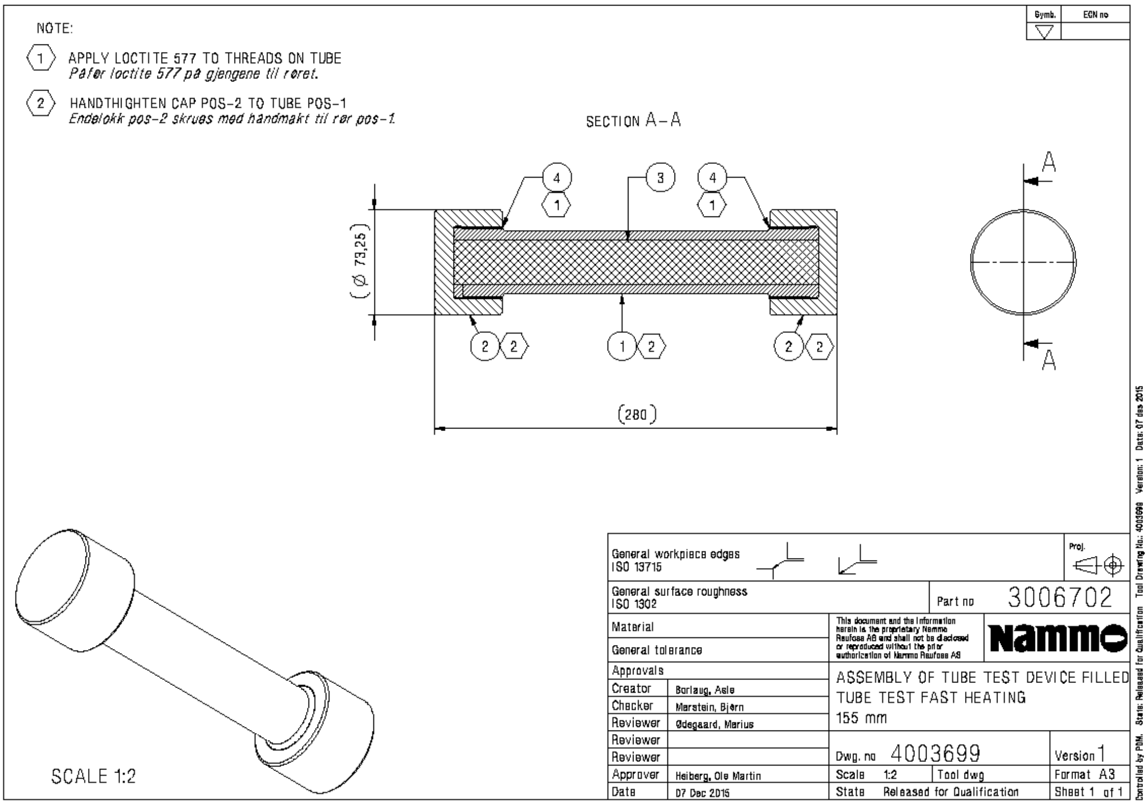


Figure B.1 Information about selected materials used in the production of the tube body and the end cap.



PARTSLIST REPORT

Nammo				Part no.:	3006702	Ver.:	1.11	State:	Released for Qualification	
				Part name:	ASSEMBLY OF TUBE TEST DEVICE			Level:	0	
				Page:	1 of 1			Date:	01 Sep 2016	
Pos.no.	**Part no.**	**Ver.**	**State**	**Part name**		**Qty.**	**Unit**	**Ref.dwg.no.**	**Ver.**	**State**
	3006702	1.11	Released for Qualification	ASSEMBLY OF TUBE TEST DEVICE				4003699	1.10	Released for Qualification
1	3006701	1.3	Released for Qualification	TUBE		1	each	4003698	1.11	Released for Qualification
2	3006693	1.3	Released for Qualification	CAP		2	each	4003690	1.8	Released for Qualification
3	769119	1.1	Released	EXPLOSIVE MCX-6100		1	as needed			
4	142244	1.3	Released	Glue, Loctite 577, 250 ml		1	as needed			

Figure B.2 Nammo drawings for the tube tests test vehicle production.

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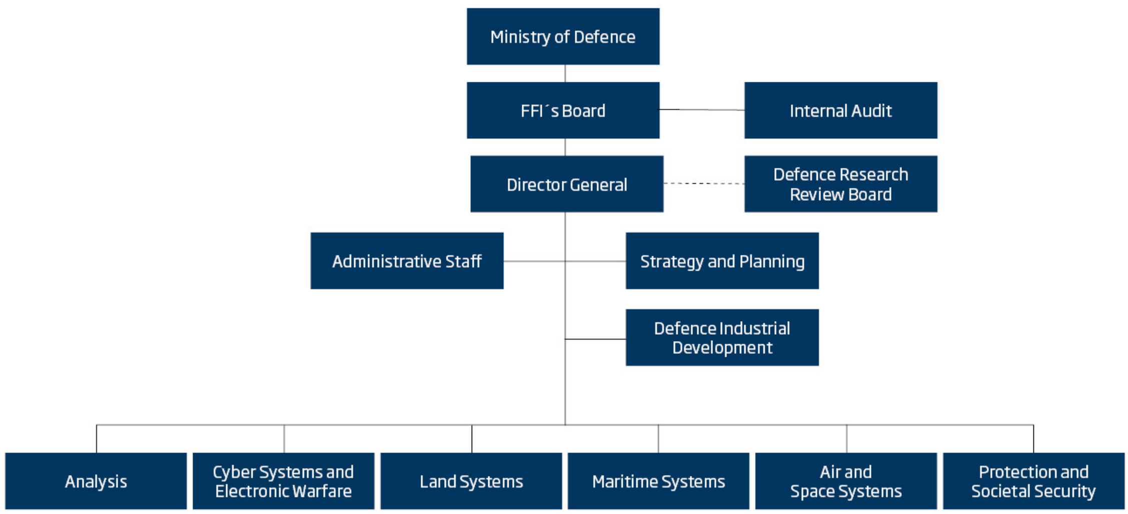

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