



FFI-RAPPORT

17/01019

Electrically heated tube test of MCX-6100 EMTAP 42 Test

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EMTAP 42 Test

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Summary

The EMTAP 42 tube test electrically heated has been performed with three different heating rates during the STANAG 4170 qualification of MCX-6100. With a heating rate of 300°C per hour the tube opens up after 39 minutes at a temperature of 162.7°C in the centre of the filling. The reaction is a burn or degree 1 response.

With a heating rate of 60°C per hour the tube reacts after 168 minutes with a temperature of 194.9°C in the filling. The reaction gives six fragments from the tube body. The response is a deflagration or degree 2 reaction.

With a heating rate of 10°C per hour the tube reacts after 906 minutes with a temperature of 200.3°C in the filling. Eight 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 is 30°C higher than on the outside tube surface. The response is a deflagration or degree 2 reaction.

The achieved results all show mild reactions for all heating rates. One can therefore expect that live munitions containing MCX-6100 has the potential to fulfill the IM requirements when facing the slow heating threat.

Sammendrag

EMTAP-test 42 elektrisk oppvarming har blitt gjennomført med tre forskjellige oppvarmingshastigheter for kvalifisering av komposisjon MCX-6100. Med en oppvarmingshastighet på 300 °C per time åpnes røret etter 39 minutter med en temperatur på 162,7 °C i sprengstoffet. Reaksjonen er en brann eller grad 1.

Med oppvarmingshastigheten 60 °C per time åpnes røret etter 168 minutter og med en temperatur på 194,9 °C i sprengstoffet. Reaksjonen medfører fragmentering av røret. Det ble gjenfunnet seks fragmenter fra røret. Reaksjonen er en deflagrasjon eller grad 2.

Med 10 °C per time åpnes røret etter 903 minutter ved en temperatur på 200,3 °C i sprengstoffet. Det ble gjenfunnet åtte fragmenter fra røret. Temperaturmålingen i sprengstoffet viser en sterk eksoterm reaksjon. Når reaksjonen inntreffer, er temperaturen i sprengstoffet 30 °C høyere enn på utsiden av røret. Reaksjonen er en deflagrasjon eller grad 2.

Oppnådde resultater viser en moderat reaksjon for alle oppvarmingshastigheter. Det er derfor å forvente at ammunisjon fylt med MCX-6100-sprengstoff vil ha potensial til å tilfredsstillere IM-kravet hvis stilt overfor denne trusselen.

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Abbreviations

DNAN	2,4-dinitroanisole
DOSG	<u>D</u> efence <u>O</u> rdnance <u>S</u> afety <u>G</u> roup
EMTAP	<u>E</u> nergetic <u>M</u> aterials <u>T</u> esting and <u>A</u> ssessment <u>P</u> olicy
HMX	octogen/1,3,5,7-tetranitro-1,3,5,7-tetraazacyclooctane
IM	<u>I</u> nsensitive <u>M</u> unitions
MCX	<u>M</u> elt <u>C</u> ast <u>eX</u> plosive
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	<u>T</u> heoretical <u>M</u> aximum <u>D</u> ensity
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). To characterize these compositions both experimental and theoretical studies have been performed. MCX-6100 containing DNAN in addition to NTO and RDX is one of these compositions that have shown good properties as an IM-filler for large calibre munitions (4-6). Properties like shock sensitivity by intermediate scale gap test (7-8) and performance like detonation pressure and velocity were determined (9). However, if MCX-6100 shall be applied as main filler the composition needs to be qualified according to STANAG 4170 (10).

One required property to be characterized for qualification is explosive response when ignited (confined and unconfined). STANAG 4491 (11) 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 tube tests we decided to perform were EMTAP 41 and EMTAP 42 (12).

In this report we will report on the EMTAP 42 tube test – electrically heated. The qualification of MCX-6100 has been a collaboration between the manufacture 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 the filler and filling it into the test vehicles have been performed by Chemring Nobel AS.

Norway has earlier used tube tests for qualification of DPX-6 (13). 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 have now 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 when ignited has been characterized by EMTAP 41 – fast heating by use of fuel fire in (14). In this report we have performed EMTAP 42 tube test – electrically heating with three different heating rates, 10, 60 and 300°C/hour. In reference 15 and 16 EMTAP 41 and 42 tube tests were performed for MCX-8100. There is no specific requirement to fulfil to pass these tests. However, for munitions like 155 mm shell, a mild reaction in the tube tests is preferable and will increase the possibility to fulfil the IM requirements

The filled tubes were, before testing, X-rayed to investigate the quality and homogeneity of the fillings. In addition the densities of the fillings were determined by weighing of the tubes before and after being filled with MCX-6100 composition.

2 Experimentally

2.1 Casting the test objects

All test objects were casted by Chemring Nobel AS.

For these test objects the MCX-6100 charge 6027/14 was used as filler. The empty tubes were stored in a heat cabinet at 100-102°C over the night before filling took place with the filler holding the same temperature (100-102°C). The filled tubes were then placed in the heat cabinet for 2 hours before being cooled at ambient temperature, 25°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 Tubes No 1 to No 3 as received from Chemring Nobel.

The tubes were cleaned and the end surface corrected. Figure 2.2 shows the tubes after this operation. Figure 2.3 shows that all the tubes have minor cracks in the end surface. Before

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



Figure 2.2 Tube No 1 to tube No 3 after cleaning and modification of the top surface.



Figure 2.3 Picture of the end surface for tube No 1 to tube No 3.

2.2 Quality of the fillings

2.2.1 Density

The density of the MCX-6100 fillings was measured by weighing empty and filled tubes.

Tube No	Weight Filled Tube (g)	Weight empty tube ¹ (g)	Weight of Filling (g)	Filled with MCX-6100 Charge	Filling density (g/cm ³) ²	To be tested in
1	2972.18	2636.1	336.08	6027/14	1.698	SCO
2	2969.22	2632.0	337.22	6027/14	1.704	SCO
3	2978.65	2641.5	337.15	6027/14	1.703	SCO

¹Body+1 end cap. ²Volume 197.9451 cm³ calculated from drawing specification of diameter 31.5 mm and height 254 mm).

Table 2.1 Properties of the tubes to be tested by electrically heated 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 1.702 ±0.002 g/cm³. TMD (Theoretical Maximum Density) for MCX-6100 is 1.7629 g/cm³. This gives an average filling percentage of 96.6.

2.2.2 X-ray

To investigate the MCX-6100 filling for unexpected defects all tubes were X-rayed. Figure 2.4 shows a picture of the X-rayed tubes. All tubes have some pores in addition to areas with lower densities 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.

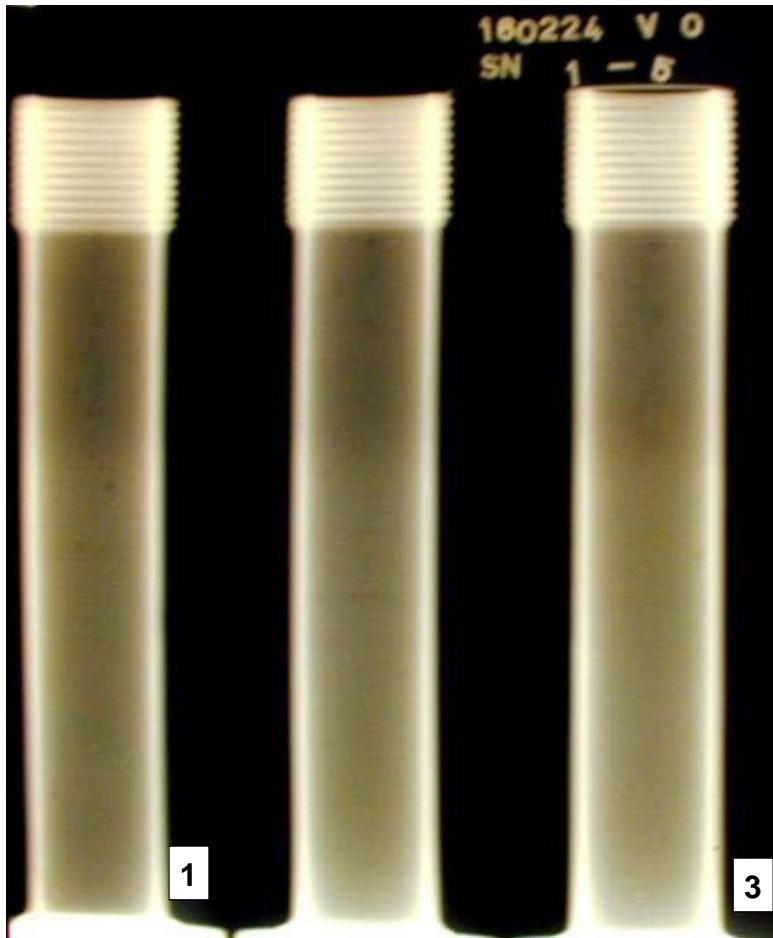


Figure 2.4 X-ray of tubes No 1 to tube No 3 filled with MCX-6100 CH 6027/14.

2.3 Tube design

We received the drawings of the tube parts from UK DOSG. Copies of these drawings are given 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 (12) and STANAG 4491 (11).

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 2.6 and Figure 2.7.

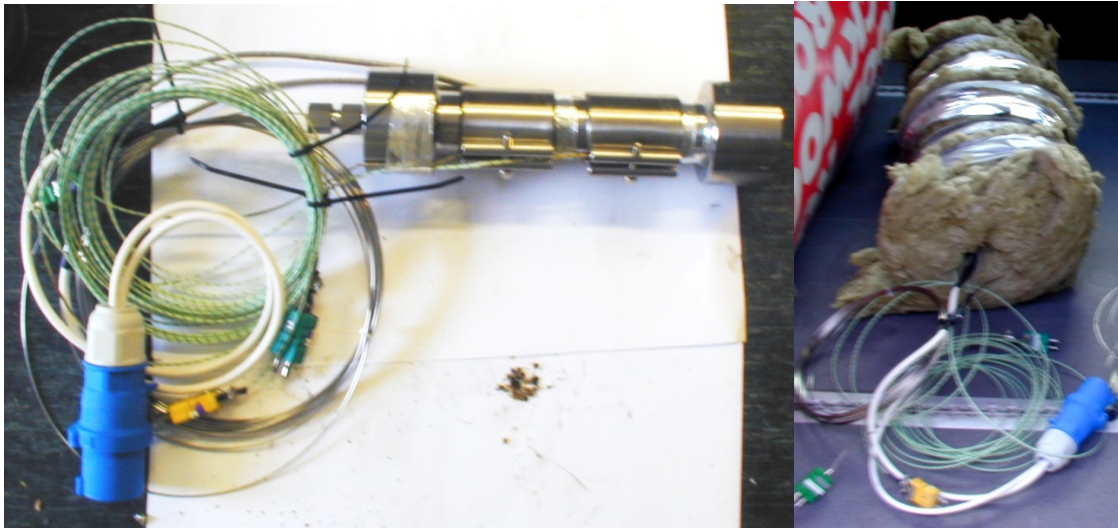


Figure 2.5 Picture of the tube before testing to the left. To the right the test item after isolation with rock wool.



Figure 2.6 Picture of the test area.

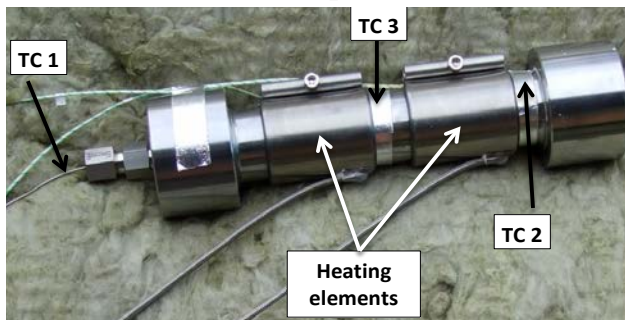


Figure 2.7 Picture of the test item with two heating elements and three thermocouples.

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 600) 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°C/hour

0.5 Hz for 60°C/hour

0.1 Hz for 10°C/hour

3 Results

3.1 Test No 15 – Tube No 1 Heating rate 300°C/hour

The first tube tested in the electrically heated tube test was tube No 1. Properties of the tube are given in Table 2.1. We wanted to have a heating rate of 300°C/hour or 5°C/min for this test. The test was started with a high degree of isolation around the tube, Figure 2.5. After the heating was turned on the temperature increase was too high and out of control. The trial was stopped two times and isolation removed to obtain better temperature control. During these starts and stops the temperature of the filling increased. In the final run applied isolation is shown in Figure 3.1 and the start temperature in the middle of the filling was 68°C.



Figure 3.1 Pictures of the test item in the test place.

The temperature registrations on the three thermocouples during the test are shown in Figure 3.2. The programmed heating started by holding the temperature of the TC 3 (tube outer surface between the heating elements) for 5 minutes or 300 seconds before activation of the heating elements. The reaction terminated after 2677 seconds or 44 minutes and 37 seconds including the 5 minutes at ambient temperature. Manual measured time to reaction was 39 minutes.

Before the temperature reached the event temperature some irregularities is observed in the filler temperature. At 89.55 °C an endothermic reaction took place and lasted until 90.8°C. This reaction is interpreted as melting of DNAN. The temperature is slightly lower than expected but can probably be explained with the bad heating control and start conditions.

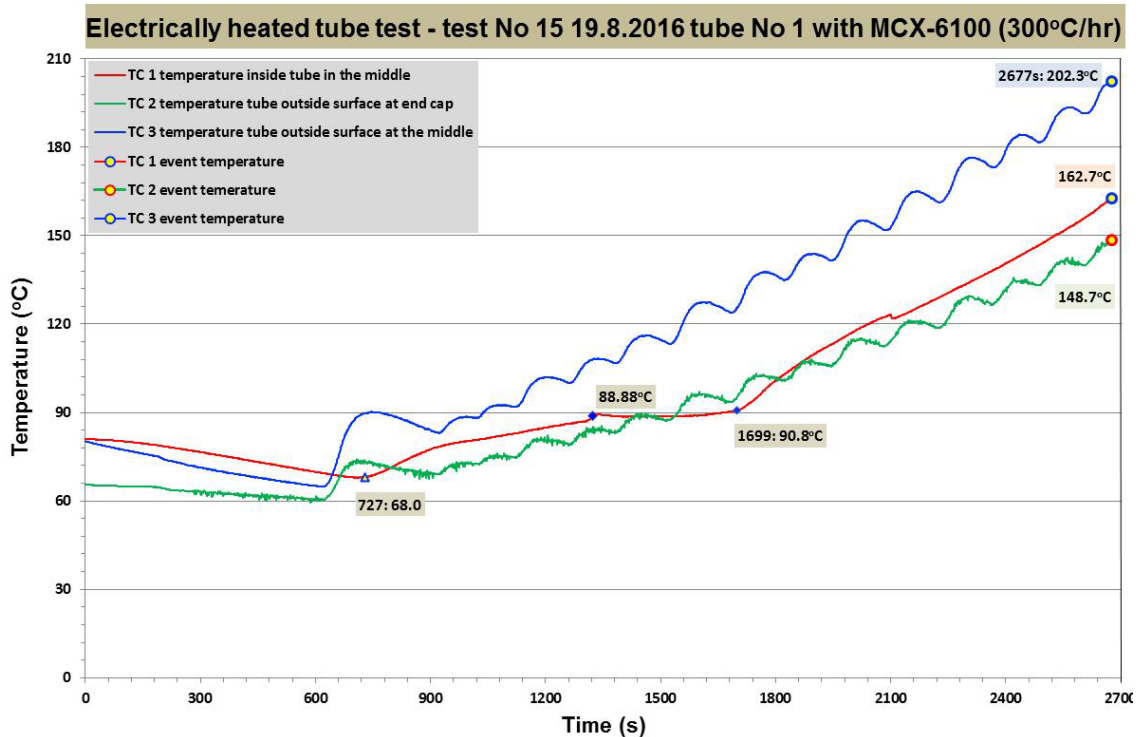


Figure 3.2 Temperature registrations on the three thermocouples during the electrically heated test with heating rate 300°C/hr. for tube No 1 filled with MCX-6100.

The reaction of the tube took place at a temperature of 162.7°C in the explosive. Figure 3.3 shows the test tube in the test chamber after the reaction. Figure 3.4 shows that the tube has only been split open without producing any fragments. The recovered tube had a weight slightly higher than the empty tube, Table 3.3. The added weight comes from soot and combustion deposits. All the MCX-6100 filler was consumed.



Figure 3.3 Pictures of tube No 1 after reaction.

TUBE TEST TYPE: SCO
DATE: 18.08.2016
COMPOSITION: MCX-6100
TEST No: 15
Tube No: 1
HEATING RATE: 300°C/hour



Figure 3.4 Recovered test item rest from test of tube No 1 filled with MCX-6100 CH 6027/14.

3.1 Test No 17 – Tube No 2 heating rate 60°C/hour

The second tube with MCX-6100 tested was tube No 2. This tube was tested with a heating rate of 60°C/hour or 1°C/min. Figure 3.5 shows the temperature readings on the three thermocouples used for registration of the temperatures inside and outside the tube during the heating. In addition the plot contains a line (dash black) for the required temperature for heating rate 1°C/min. The blue line, tube outside surface temperature follows this line. The green line, outside surface temperature at the end cap (TC 2), shows an increasing deviation from the required temperature (dash black line) as the temperature increase. At the event temperature the deviation is 26.5°C. The red curve, the temperature in the center of the filling, is most of the time below the outside temperature (blue line). However, at 144.6°C the temperature on the tube outside surface and in the explosive filling are equal. At 160°C the temperature in the filling is higher than on the tube surface. At the event temperature 194.9°C the temperature in the explosive filling is 11.7°C higher than the temperature at the steel tube surface. This shows that a relative strong exothermic reaction took place in the filling before the tube collapsed.

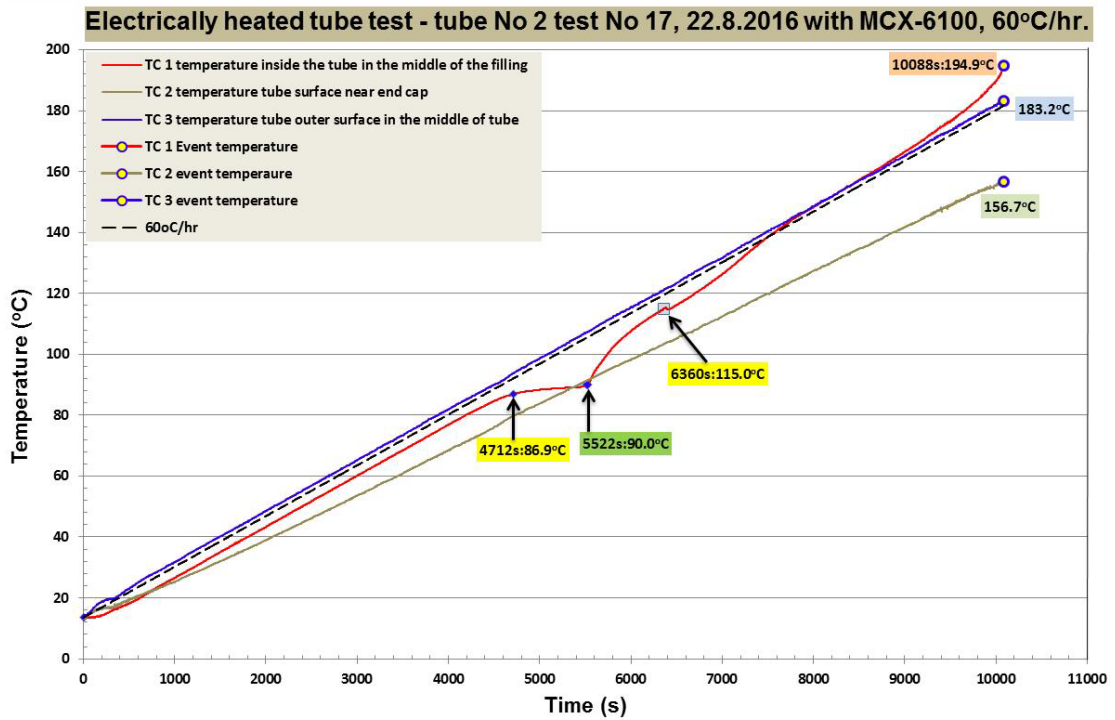


Figure 3.5 Temperature registrations on the three applied thermocouples during testing of tube No 2.

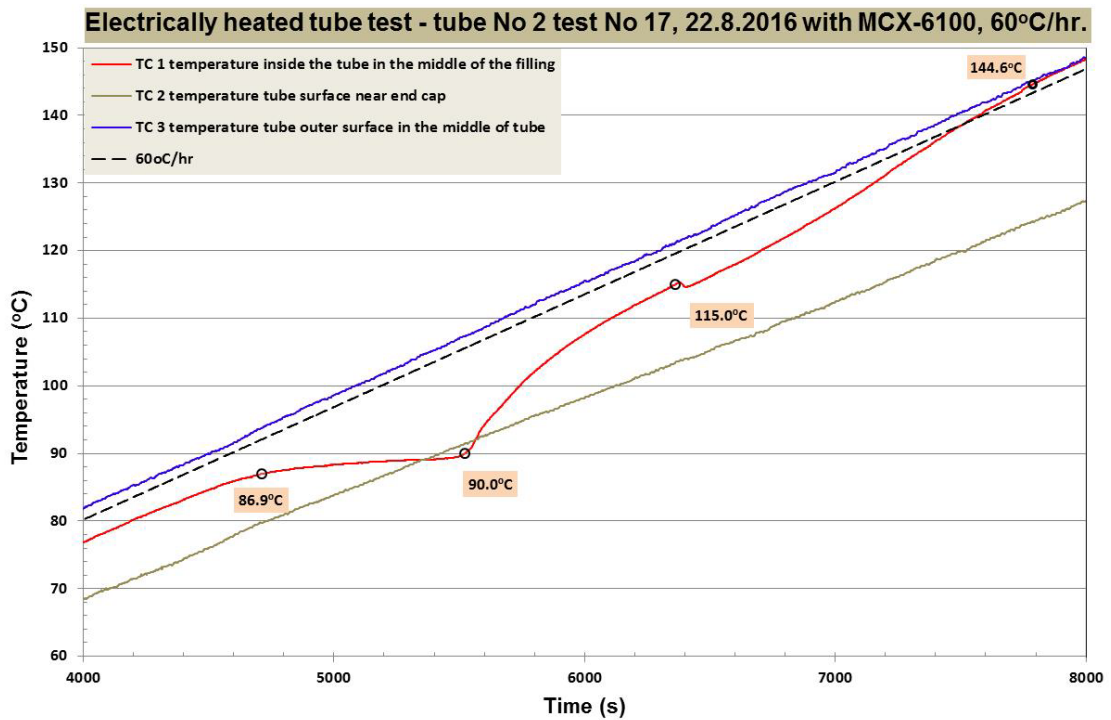


Figure 3.6 Temperature registrations on the three applied thermocouples during testing of tube No 2.

Figure 3.6 shows an enlarged part of Figure 3.5. The first deviation of the temperature in the explosive filling from the outside tube temperature occur at 86.9°C and last for 810 seconds until the filling has a temperature of 90.0°C. During this time the temperature should have increased by 13.5°C and not the measured 3.1°C. This endothermic reaction is interpreted as melting of DNAN. The next endothermic reaction in the filling is observed at 115°C. This is a reaction with little energy absorption.



Figure 3.7 Picture of the recovered items from tube No2 filled with MCX-6100 CH 6027/14.

Tube No 2 had an empty weight of 3754 g. Figure 3.7 shows that 8 items were recovered including the two end caps. The weight of these items in Table 3.1 shows that recovered mass is slightly higher than the mass of the empty tube. However, Figure 3.7 shows that one of the recovered items contains unreacted MCX-6100 filler. We did not melt the unreacted filler out of

the end cap, but visually consideration indicates that one small fragment is missing since the weight of the recovered MCX-6100 is higher than 1 g.

Fragment No – Weight (g)								Total weight (g)
1	2	3	4	5	6	7	8	
1721.56*	1415.13	164.13	159.28	99.43	76.19	65.94	53.68	3755.34

*Contain rest of the MCX-6100 filling, see Figure 3.7.

Table 3.1 Weight of recovered fragments from test No 17 of tube No 2.

3.2 Test No 19 – Tube No 3 Heating rate 10°C/hour

The last of the tubes filled with MCX-6100 CH 6027/14 we tested was tube No 3. For this tube a heating rate of 10°C/hour was used. Figure 3.8 shows the temperature registrations on the three applied thermocouples. The test was performed in the same location as shown in Figure 3.1. From the start at room temperature (20°C) to reaction at 200.3°C it took 54340 seconds or 15 hours 5 minutes and 40 seconds.

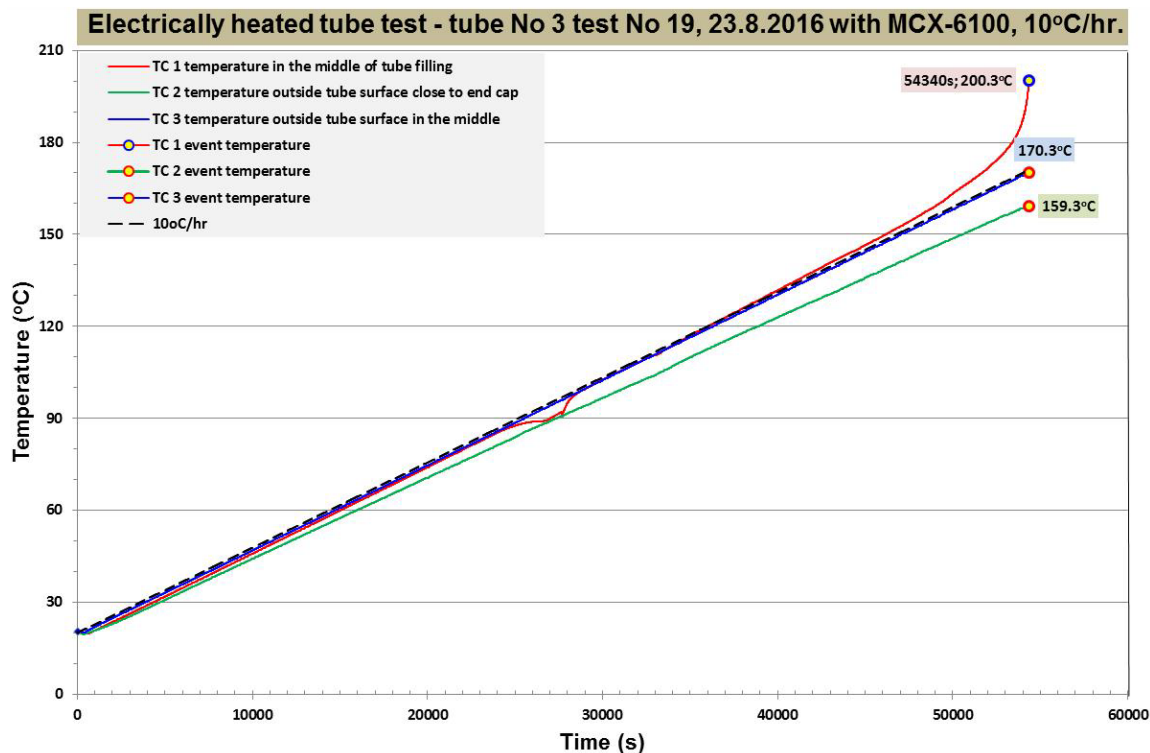


Figure 3.8 Temperature registrations on the three applied thermocouples during testing of tube No 3 filled with MCX-6100 CH 6027/14 composition.

In Figure 3.8 the 10°C/hour heating rate line is plotted as dashed black. This temperature and the outside tube surface temperature between the two heating elements follow each other. For the thermocouple TC 2 a deviation of 11°C at the event temperature is observed.

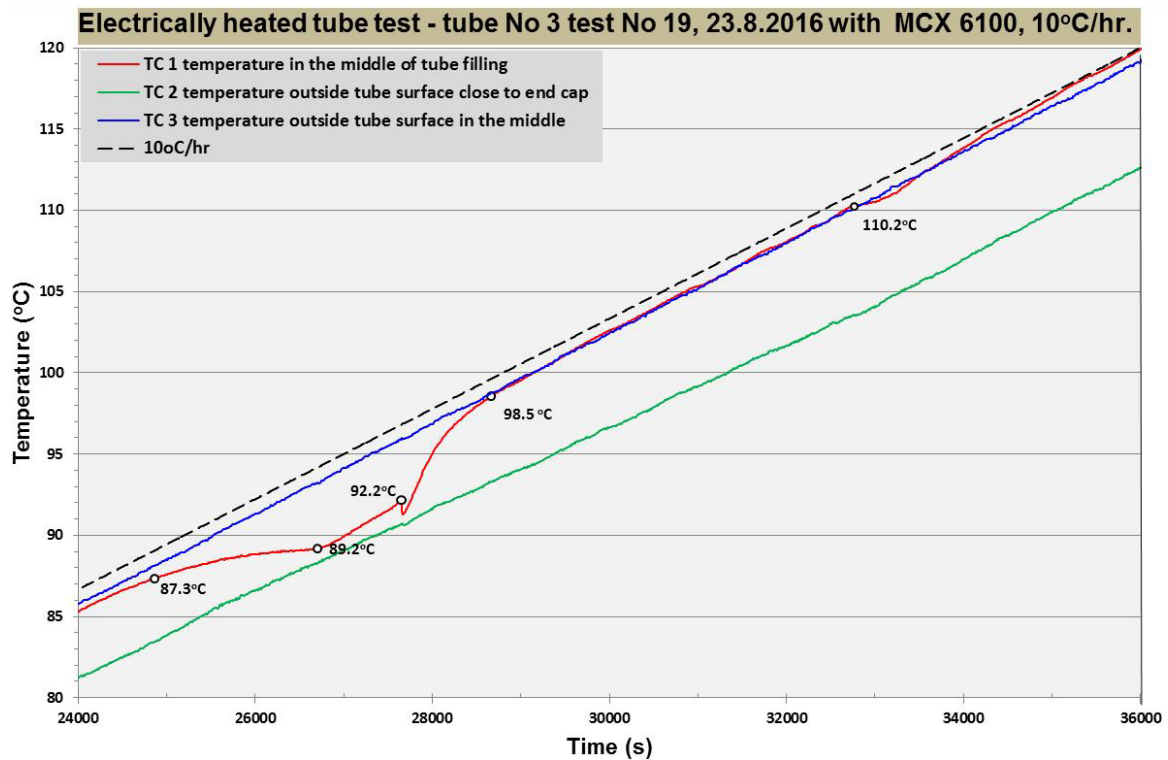


Figure 3.9 Temperature registrations on the three applied thermocouples during testing of tube No 3 filled with MCX-6100 CH 6027/14 composition.

The temperature inside the explosive filling shows some deviations from the steel tube surface. The first difference is observed from 87.3°C (after 24850 seconds) to 89.2°C (after 26720 seconds) where the change in temperature is only 1.9°C and should have been 5.2°C. From 89.2°C to 92.2°C (after 27760 seconds) the temperature increase is 3°C and should have been 2.9°C. At 92.2°C an endothermic reaction is observed before TC 1 and TC 3 gradually approach each other and at 98.5°C shows the same temperature. At 110.2°C a new endothermic reaction is observed. From approximately 130°C TC 1 is higher than TC 3. The difference gradually increases and at the event temperature 200.3°C the difference is 30°C. This shows that a strong exothermic reaction takes place in the explosive filling before the tube collapses.

In Table 3.2 the weights of recovered fragments are given. Figure 3.10 shows a picture of the recovered rests of the tube. Among the recovered parts is not the grand one with a weight of 81.52 g. Adding this weight to 3517.67g, the weight of recovered fragments, we obtain 3599.19 g. Therefore we lack 164.2 g or 4.36 % of the tube mass. This can be one or more fragments. However, looking on the fragments the response seems to be a degree 2 or deflagration.

Table 3.2 Weight of recovered fragment of tube No 3.

Fragment	
No	Weight (g)
1	1423.53
2	1355.44
3	232.49
4	127.81
5	86.22
6	82.11
7	71.60
8	63.17
9	44.76
10	30.54
All	3517.67



Figure 3.10 Picture of recovered fragments of tube No 3.

3.3 Summary of the test results

In the test description (12) the guidance below to interpret 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 3.3 and 3.4 for MCX-6100 CH 6027/14. The event temperature increases as the heating rate goes down. The event temperatures for heating rates 300, 60 and 10°C/hour are 162.7°C, 194.9°C and 200.3°C. The temperature on the tube surface goes in the opposite direction being 202.9°C with heating rate 300°C/hour to 170.3°C for heating rate 10°C/hour. TC 3 with heating rate 300°C/hour is 30.2°C higher than TC 1, and the TC 3 temperature with heating rate 10°C/hour is 30°C lower than the TC 1 temperature. The difference 60.2°C shows that with a heating rate of 10°C/hour a very strong exothermic reaction takes place inside the explosive filling.

Test No	Tube No	Tube weight empty (g)	Heating Rate (°C/hour)	Event temperature (°C)			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				
15	1	3758.7	300	162.7	148.7	202.9	2677	1	3767.52	1
17	2	3754.6	60	194.9	156.7	189.2	10088	8	3755.34*	2
19	3	3764.1	10	200.3	159.3	170.3	54340	10	3517.67	2

*Contain no reacted MCX-6100 filling.

Table 3.3 Summary of the results for the EMTAP test No 42 tube test electrically heated of MCX-6100 CH 6027/14.

Round No	Vehicle No	Heating Rate (°C/hour)	Time Date	Filling Density (g/cm ³)	Recovered fragment			% Filling Recovered	Degree of Reaction	Time to Event (minutes)	Comments
					Total No	Body No	%Wt				
15	1	300	180816	1.698	1	1	100	0	1	39	
17	2	60	220816	1.704	8	6	?	?	2	168	Filling not removed
19	3	10	230816	1.703	10	8	93.6	0	2	906	

Table 3.4 Summary of the results for the EMTAP test No 42 tube test electrically heated of MCX-6100 CH 6027/14.

The degree of reaction goes from degree 1, burning, to degree 2, deflagration, when the heating rate decreases from 300°C/hour to 10°C/hour. The reaction degree for the two most violent reactions is a deflagration. Compared with the test results for MCX-8100, MCX-6100 is slightly more violent in the EMTAP 42 test. For both heating rate 60°C/hour and heating rate 10°C/hour more fragments is produced with MCX-6100 than with MCX-8100. For most of the tests the event temperatures in MCX-8100 take place 10°C above the event temperatures for MCX-6100. The difference in decomposition temperature between RDX and HMX may probably explain

this difference. If that is the explanation, these two ingredients determine the cook-off temperature for MCX-6100 and MCX-8100.

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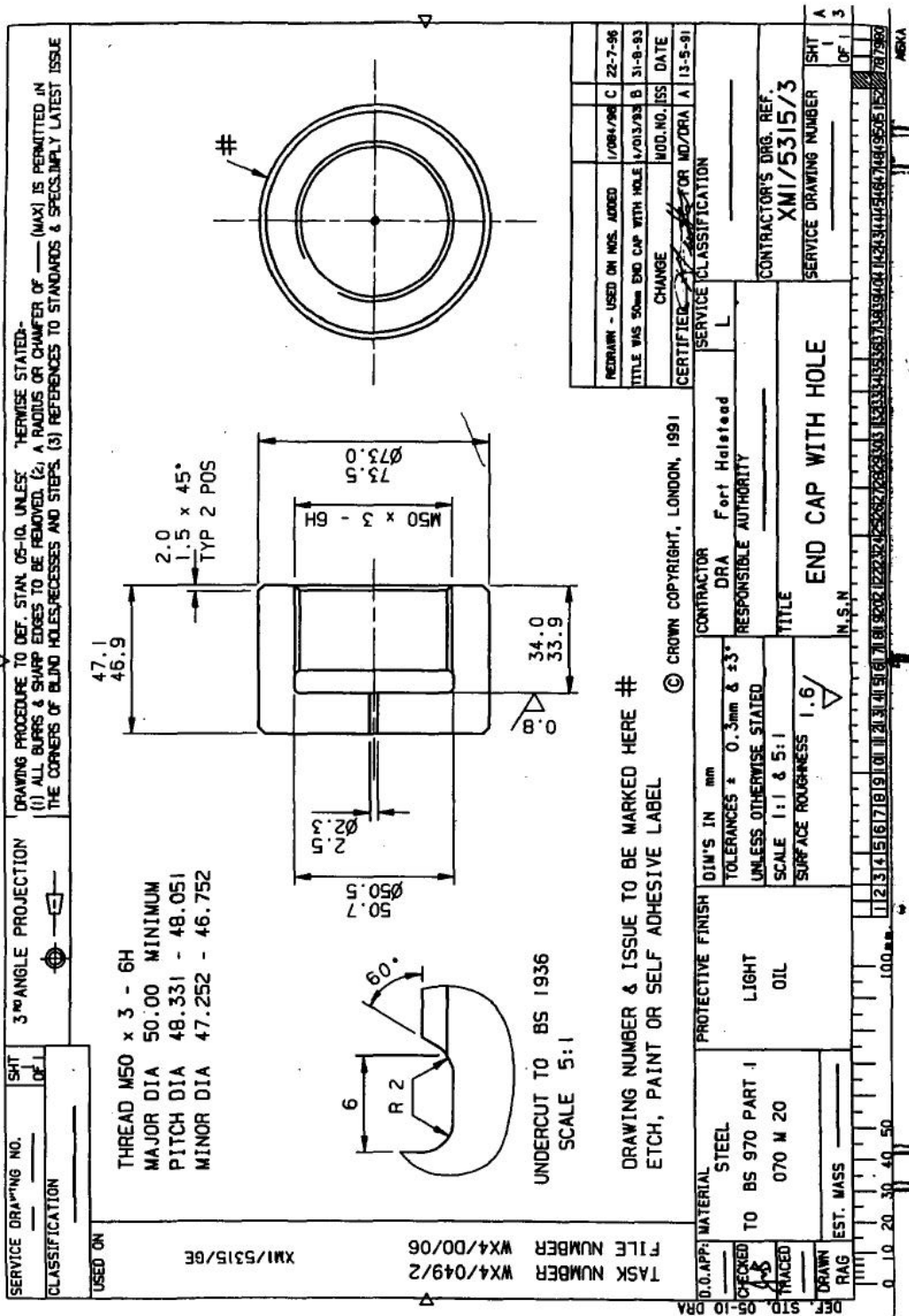


Figure A.2 Drawing of the end cap with hole.

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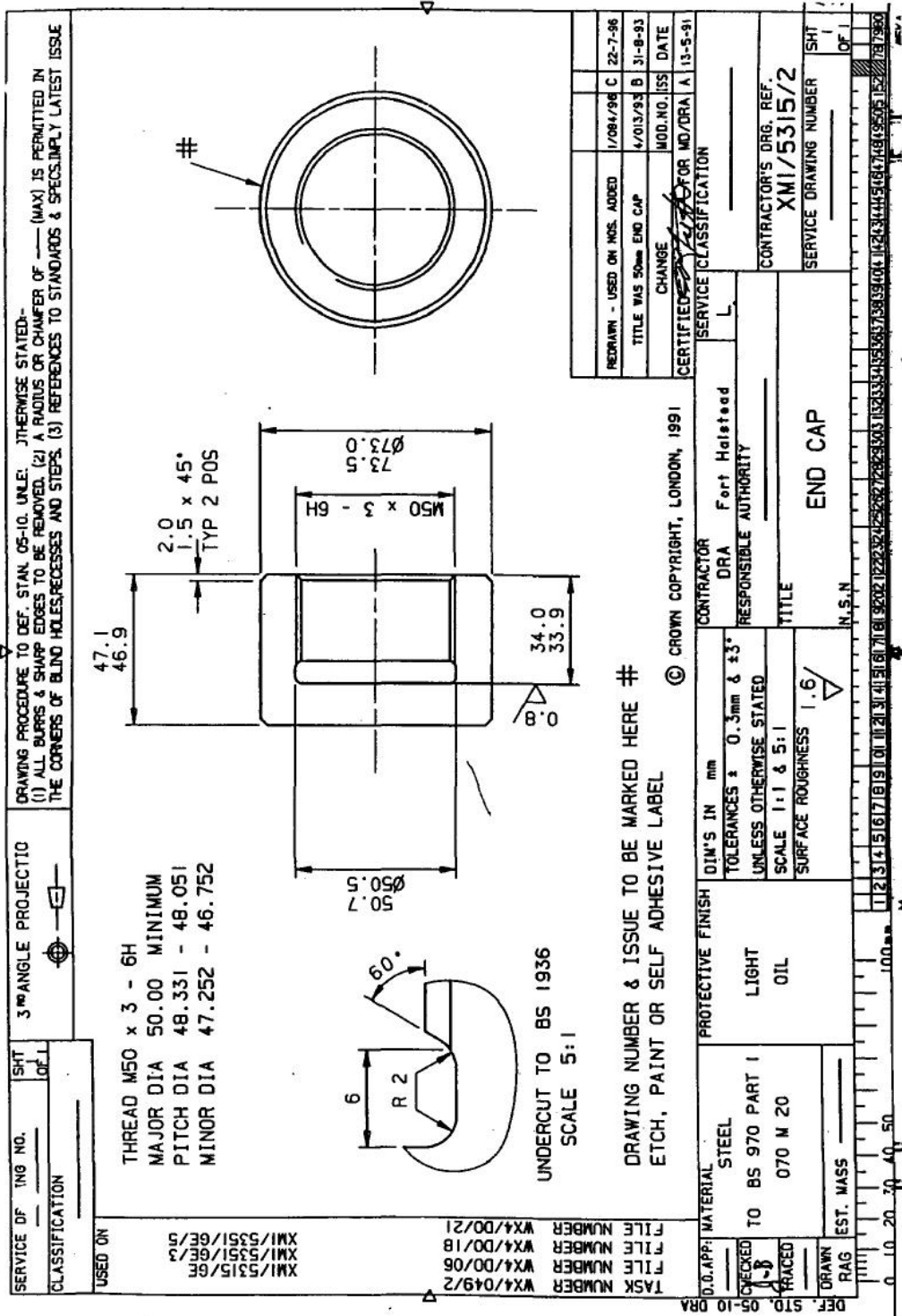



Figure A.3 Drawing end cap with thread dimensions.

SECURITY CLASSIFICATION UNCLASSIFIED		CONTROLLED ON 60001		USED ON	
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 3.93 ohm/m RESISTANCE.		AS REQD	KANTHAL LTD, CANAL ARM FESTIVAL PARK,STOKE-ON-TRENT OR EAST ANGLIAN FINE WELD FOR SMALL QUANTITIES.
9	ND	THERMOCOUPLE FINE WIRE TYPE K FIBREGLOSS RS No 409-4920		1	
10	ND	GLASS SCRIM TAPE, 50mm WIDE, PRODUCT CODE GS1053/050/50.		AS REQD	CHESHIRE RIBBON, KINGSTON MILLS, MANCHESTER Rd, SK14 2BZ.
11	ND	HEAT RESISTANT POLYAMIDE TAPE, COLOUR GOLD 36 YARDS, 1 INCH WIDE RS 216-2302		AS REQD	
DRAWN PBM		CHECKED NEP		CERTIFIED DC/09/080	
TITLE TEST No. 42 TUBE TEST - ELECTRICALLY HEATED, ASSEMBLY (WITH INTERNAL THERMOCOUPLE)					
QinetiQ		SECURITY CLASSIFICATION: UNCLASSIFIED		08/07/09	A
MATERIAL No. 10011838		ITEM LIST FOR FHD-6701-AF		CHANGE No	DATE
				No. OF SHEETS	ISSUE
				SHT.No. 1	
FH-PROE-REL3-1L (REVISED 03/2009)		Copyright © 2009 QinetiQ			

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

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.

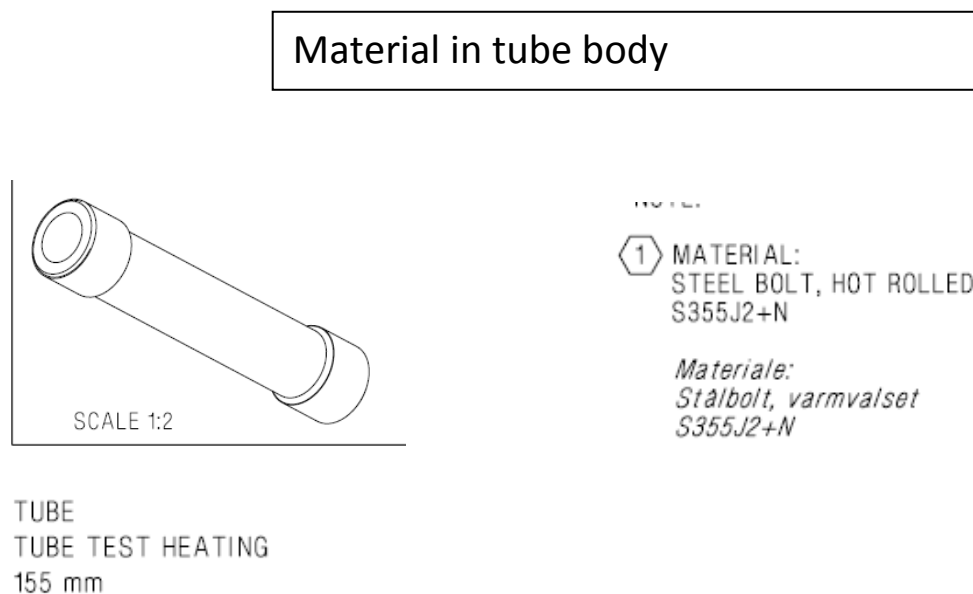
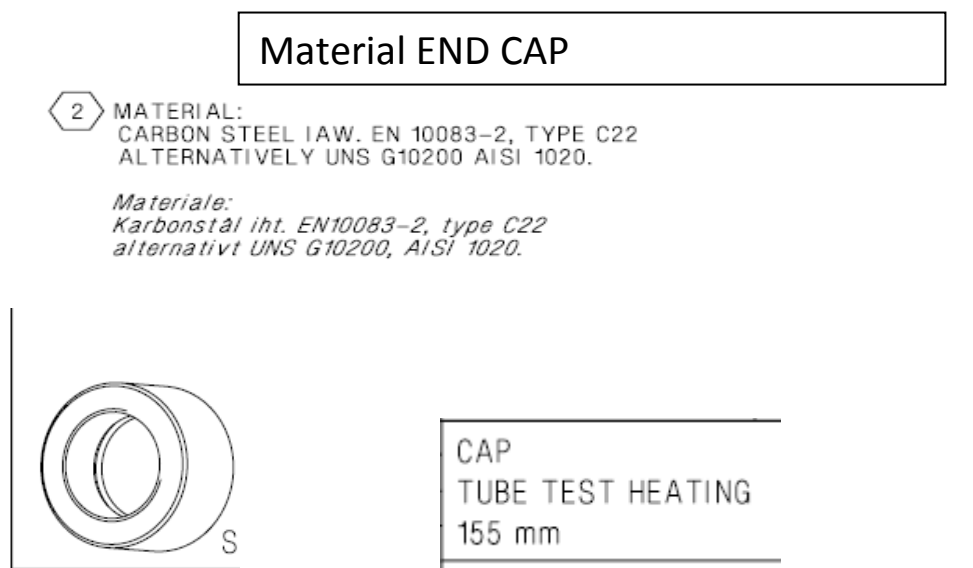
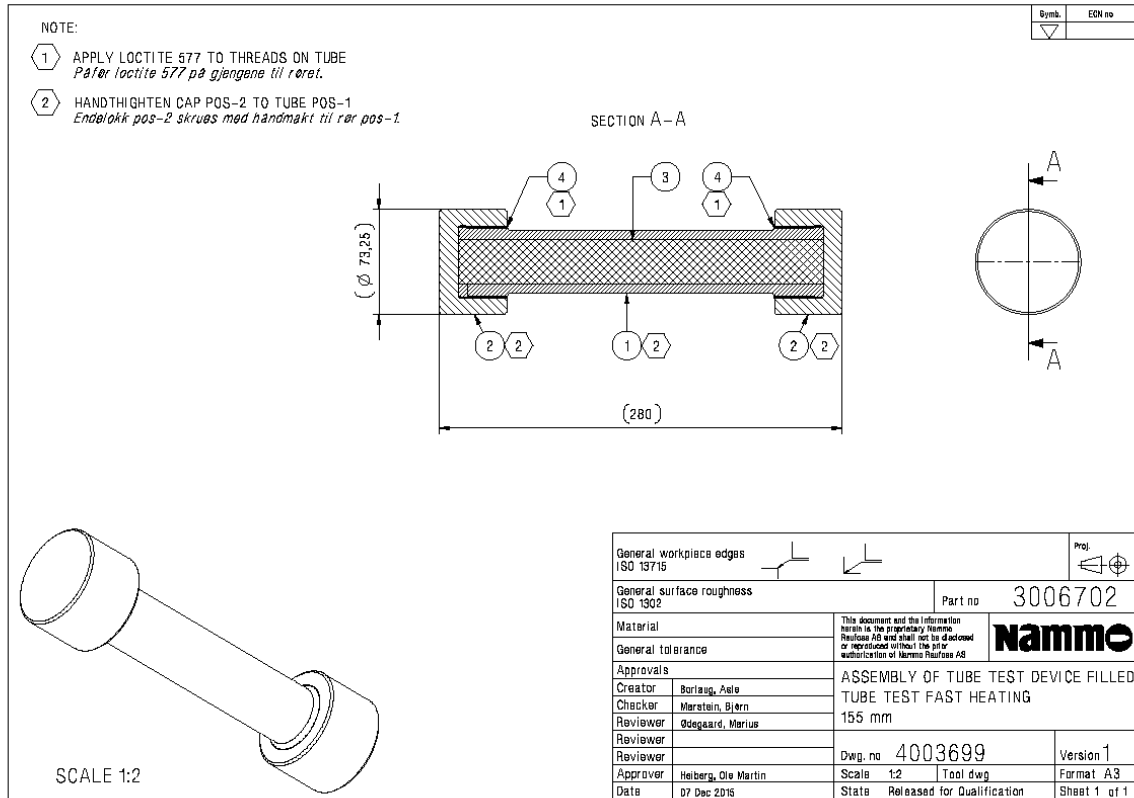


Figure B.1 Information about selected materials used in production of tube body and end cap.



PARTSLIST REPORT

Part no.:		Ver.:		State:					
3006702		1.11		Released for Qualification					
Part name:				Level:					
ASSEMBLY OF TUBE TEST DEVICE				0					
Page:				Date:					
1 of 1				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 tube tests test vehicle production.

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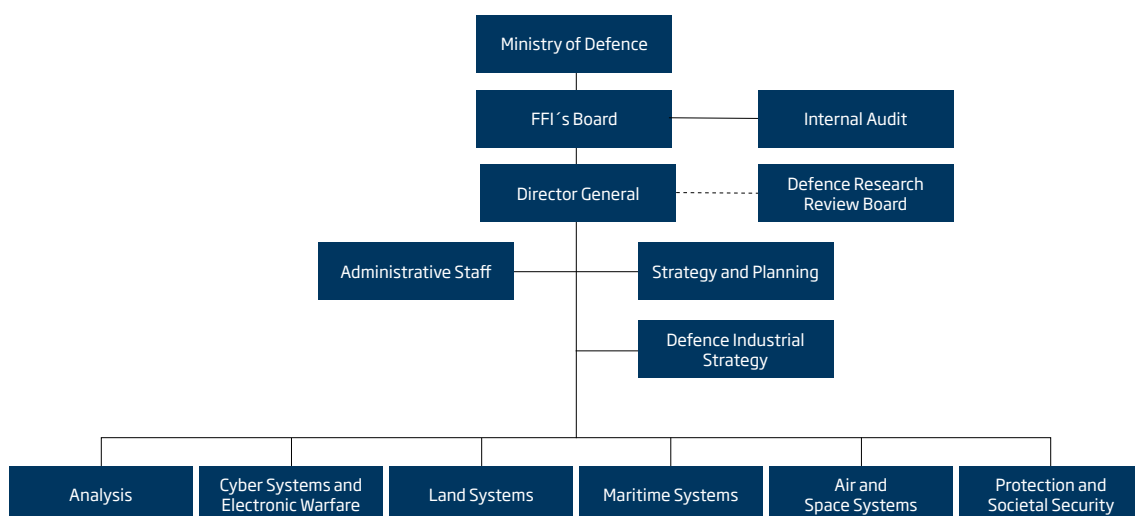
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