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DETERMINING OF THERMAL STABILITY OF EXPLOSIVES FOR CIVIL USES MODERN EQUIPMENT EQUIPPED WITH AUTOMATIC TEMPERATURE AND PRESSURE

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Abstract:
Thermal stability of explosives for civil use is a key security parameter. When the explosive is exposed to high temperatures in a given period of time can lead to undesirable phenomena such as decomposing or even very dangerous as uncontrolled detonation.

***Key words**: thermal stability, security parameter, continuous monitoring, temperature, pressure*

GENERAL CONSIDERATIONS ON THE DETERMINATION OF THE THERMAL STABILITY OF EXPLOSIVES FOR CIVIL USES

In Directive 93/15/EEC [2] are technical requirement on the security of explosives to be evaluated before placing on the market by testing the products according to harmonized standards, aimed finding of compliance/non-compliance with relevant safety requirements.

Test described in the standard SR EN 13631-2, Explosives for civil use, high explosives. Part 2: Determination of thermal stability of explosives, meets the essential requirements, required to be evaluated in section I.1; II.1 (b) and II.1 (d) of the European Directive, namely [4]:

- each explosive must be designed, manufactured and supplied in such a way as to present a minimal risk to the safety of human life and health, and to prevent damage to property and the environment under normal, foreseeable conditions, in particular as regards the safety rules and standard practices until such time as it is used,
- the physical and chemical stability of the explosive in all environ-mental conditions to which it may be exposed,
- compatibility of all components as regards their physical and chemical stability.

Thermal conditioning and verification of temperature resistance for explosives for civil use, is an operation lasting from several hours to several days with significant risk to staff involved in performing these tests, it reproduces the extreme conditions of exposure [1].

The equipment described in European Standard is one specially made for this test and allows a determination with high level of confidence of this feature.

Also, under NUCLEU Program 2013/Project PN 07 45 03 17 [3] was purchased an advanced equipment for the test for determining the thermal stability of explosives for civil uses, in accordance with EN 13621-2 (Fig. 1).

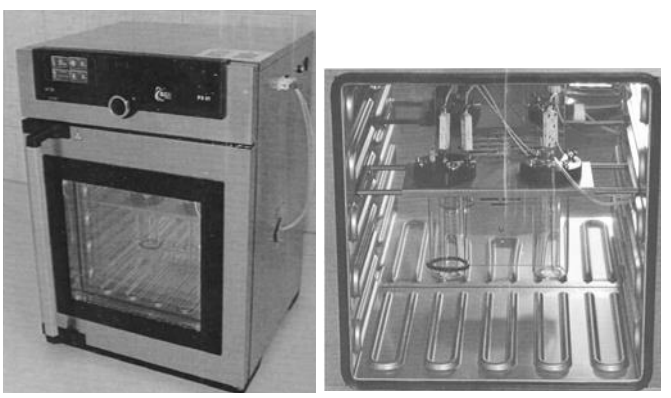

Fig. 1 Equipment to determine the thermal stability of explosives for civil uses type 75ST – Thermal Stability Tester (75 0C

For Measurement of thermal stability of explosives for civil uses the following equipment is needed for measurement and determination: oven capable of controlling temperature (75 ± 2)°C; balance, capable of weighing with accuracy of ± 0.1 g; Three thermocouples suitable inert substance tested; temperature recording system capable of measuring the temperature with accuracy of ± 1°C. Two glass tubes, with a flat bottom with an inside diameter of (50.5 ± 0.5) mm, length about 150 mm and a thickness of about 3 mm. In order to close the tube with the substance under test is provided with a device for continuous measurement of the pressure and a valve calibrated at a static pressure of 60 kPa standard; reference material with the same physical condition as that of the substance to be tested, stable at the test temperature (sand).

The test method consists in the evaluation stability of explosives by subjecting them to high temperature conditions, as compared to a reference material, stable at the test temperature.

If you do not have any information on the thermal behavior of the substance, first preliminary test should be

performed with a small amount of the substance (e.g., 5 g) to determine whether the substance exploding at 75°C.

Further, adjust the the oven temperature (75 ± 2)°C and after weighing an empty glass tube, the test sample is inserted into the tube. Granular solids are tested without compacted and the substance tested should fill the bottom of the tube up to a height of (50 ± 1) mm, resulting in a volume of test sample (100 ± 2) ml.

Shall be positioned the block into the lower part of tube and weigh the tube with the substance to be tested in order to determine the mass of the test sample, thereby achieving the charge density of the substance with the following formula:

$$\rho = \frac{M}{V} \tag{1}$$

where:
r - loading density, (g/ml);
M - weight of sample for testing, (g);
V - the sample volume for the test, (ml).

RUNNING A TEST

Every thermal stability test consists of the following steps [3, 4]: Ready to Start (1), Initializing Instrument (2); Warming Up Oven (3); Opening Pressure Valve (4); Depressurizing Sample (5); Closing Pressure Valve (6); Test in Progress (7); Shutting Down Instrument (8); Sample Examination (9); Test Completed (10).

During the test course, the program will execute all these steps sequentially from the first one to the last one.

1. Ready to Start:

This is the default step when a new test is created (Fig. 3).

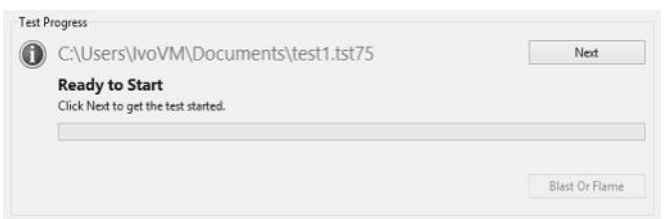

Fig. 3 Capture with the section "Ready to Start"

2. Initializing Instrument:

The purpose of this step is to establish a connection with the instrument and prepare it for the test. Among the other actions, the oven heating system is turned ON and the sample depressurization system is turned OFF (Fig. 4).

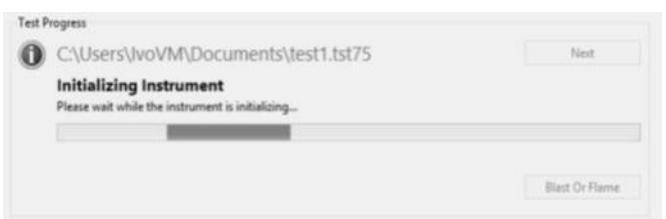

Fig. 4 Capture with the section "Initializing Instrument"

This step takes only a few moments and requires absolutely no user interaction.

After the instrument has been initialized, the test will switch to the *Warming Up Oven* step.

3. Warming Up Oven:

The purpose of this step is to establish a connection with the instrument and prepare it for the test (Fig. 5). Among the other actions, the oven heating system is turned ON and the sample depressurization system is turned OFF. In this step the oven is warming up and the program is waiting for both the sample temperature and the reference temperature to fall within the range of 75.0 ± 2.0°C. This step takes some time (depending on the difference between the current oven temperature and the test temperature) and requires absolutely no user interaction.

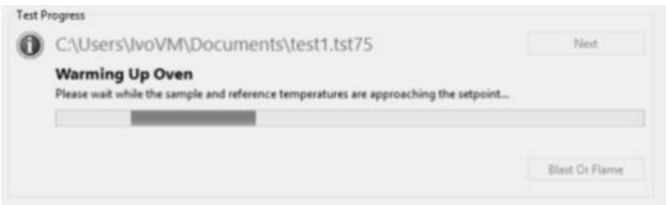

Fig. 5 Capture with the section "Warming Up Oven"

When the oven is warmed up, the test will switch to the *Opening Pressure Valve* step.

4. Opening Pressure Valve:

In this step the program is preparing the instrument for the sample depressurization process by remotely opening the pressure valve (Fig. 6). This step takes only a few moments and requires absolutely no user interaction.

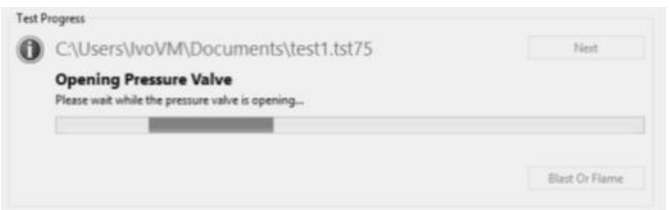

Fig. 6 Capture with the section "Warming Up Oven"

When the pressure valve is open, the test will switch to the *Depressurizing Sample* step.

5. Depressurizing Sample:

In this step the excess pressure is being released from the sample tube and the program is waiting for the gauge pressure to drop below 1.0 kPa (Fig. 7). This step takes some time and requires absolutely no user interaction.

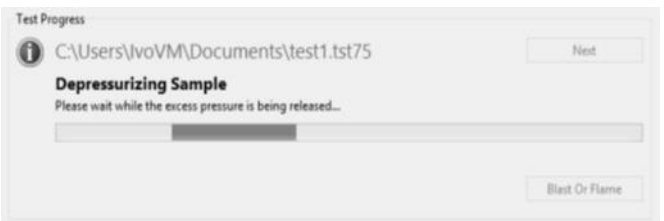

Fig. 7 Capture with the section "Depressurizing Sample"

When the sample is depressurized, the test will switch to the *Closing Pressure Valve* step.

6. Closing Pressure Valve:

In this step the excess pressure is being released from the sample tube and the program is waiting for the gauge pressure to drop below 1.0 kPa (Fig. 8). This step takes some time and requires absolutely no user interaction.

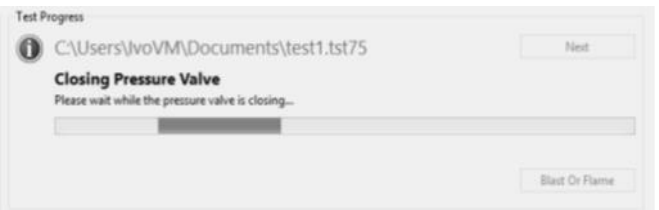

Fig. 8 Capture with the section "Closing Pressure Valve"

When the pressure valve is open, the test will switch to the *Test in Progress* step.

7. Test in Progress:

This is the main test step in which the temperature readings from all thermocouples and the gauge pressure readings are periodically recorded to a specified file and also visualized in the graph (Fig. 9).

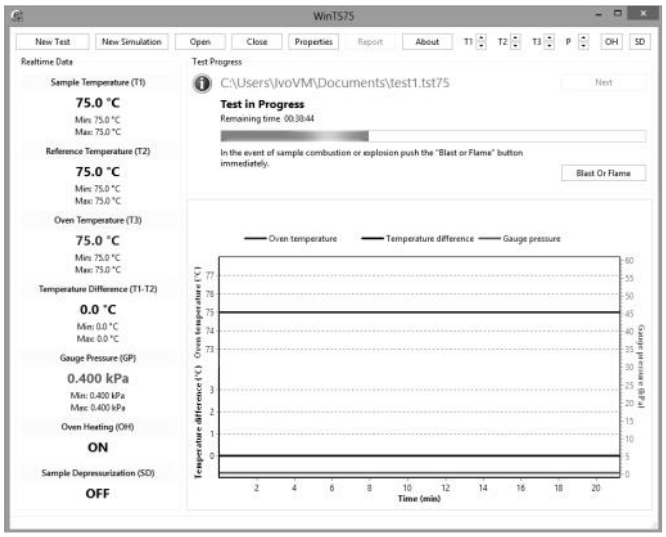

Fig. 9 Capture with the section "Test in Progress"

This step takes 48 hours to complete naturally, but there are some events (invoked procedure by the user or automatically by the program) which may cause the test to be interrupted at any time.

Below are the events that may lead to an immediate completion of this step:

a. blast or flame in the sample tube,
b. maximum self-heating of the sample,
c. sample pressure limit excess,
d. temperature control failure,
e. natural completion.

a. blast or flame in the sample tube:

This is the only event that requires user's attention. If a sample explosion or combustion occurs during the test, all you have to do is to push the "Blast" or "Flame" button and the program will stop the test immediately (according to the standard) (Fig. 9.1).

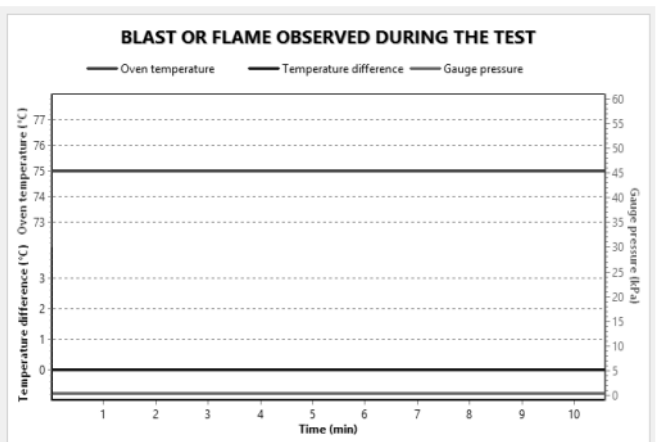

Fig. 9.1 Capture with the section "Blast or flame in the sample tube"

b. maximum self-heating of the sample:

This is an automatic event. If the temperature difference exceeds 3.0°C during the test, the program will stop the test immediately (according to the standard), (Fig. 9.2).

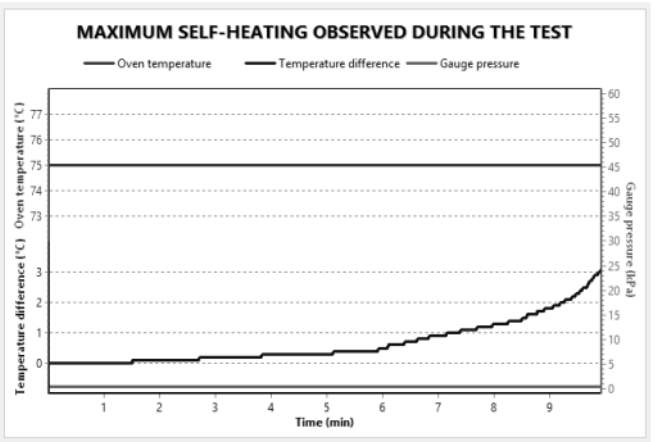

Fig. 9.2 Capture with the section "Maximum self-heating of the sample"

c. sample pressure limit exces:

This is an automatic event. If the gauge pressure in the sample tube exceeds 59 kPa during the test, the program will stop the test immediately (according to the standard), (Fig. 9.3).

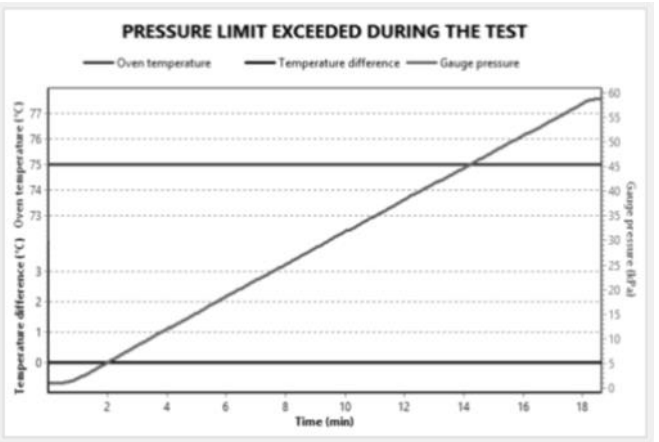

Fig. 9.3 Capture with the section "Sample pressure limit excess"

d. temperature control failure:

This is an automatic event. If the oven temperature goes outside the allowed range of 75.0 ± 2.0°C during the test, the program will stop the test immediately (according to the standard), (Fig. 9.4).

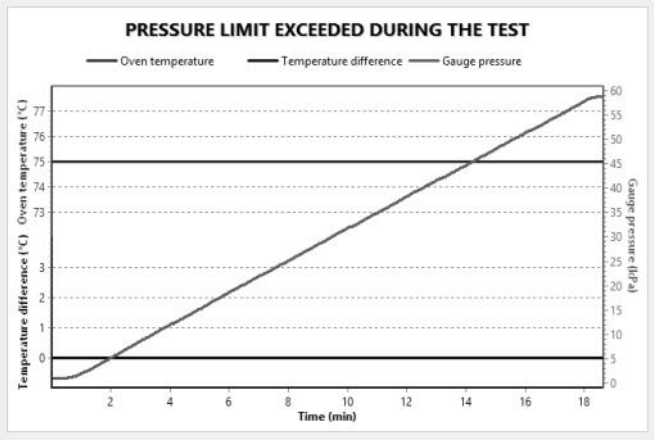

Fig. 9.4 Capture with the section "Temperature control failure"

e. natural completion:

If none of the above events occurs during the course of the test, it will complete naturally after 48 hours, (Fig. 9.5).

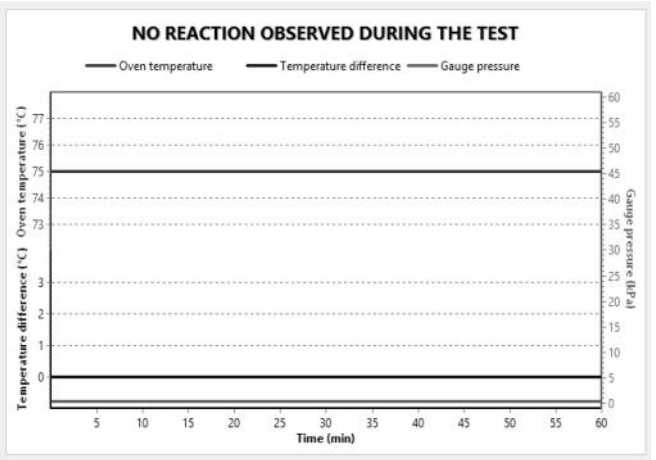

Fig. 9.5 Capture with the section "Natural completion"

8. Shutting Down Instrument:

The purpose of this step is to prepare the instrument for the end of the test and shut down the established connection with the instrument. Among the other actions, the oven heating system is turned OFF and the sample depressurization system is turned ON. This step takes only a few moments and requires absolutely no user interaction (Fig. 10).

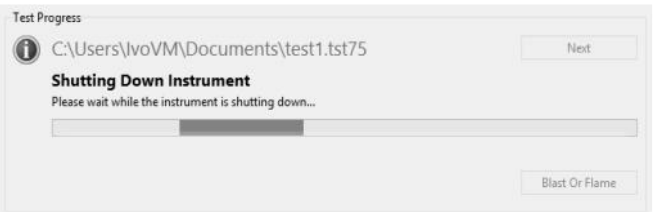

Fig. 10 Capture with the section "Shutting Down Instrument"

After the instrument has been shut down, the test will switch to the *Sample Examination* step.

9. Sample Examination:

This step requires the user interaction. Follow the on-screen instructions: Let the sample cool down, record the sample weight and also examine it for color changes, (Fig. 11).

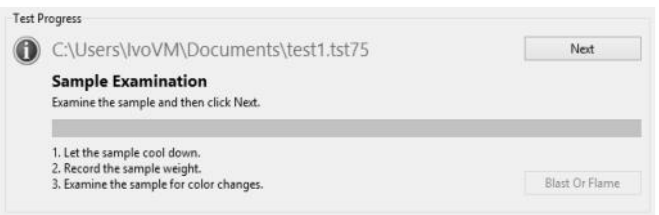

Fig. 11 Capture with the section "Sample Examination"

After push the ″Next″ button, the program will now display a dialog window where we can specify how the sample properties changed during the test.

The following properties must be specified at this time:

- weight of the tube filled with the sample (after test),
- sample color changes.

After, we will be push the ″OK″ button to apply the changes. The test will switch to the *Test Completed* step.

10. Test Completed:

This is the final test step. By this time the test has been completed, (Fig. 12).

After push the ”Next” button, the program will now display a dialog window where we can specify how the sample properties changed during the test.

Fig. 12 Capture with the section "Test Completed"

OPENING A TEST AND GENERATING A TEST REPORT

Following the test (Fig. 13, 14), in accordance with the applicable standard EN 13631-2, on a sample of 100 g nitrocellulose smokeless powder, in terms of standard samples of quartz sand, the following results were obtained.

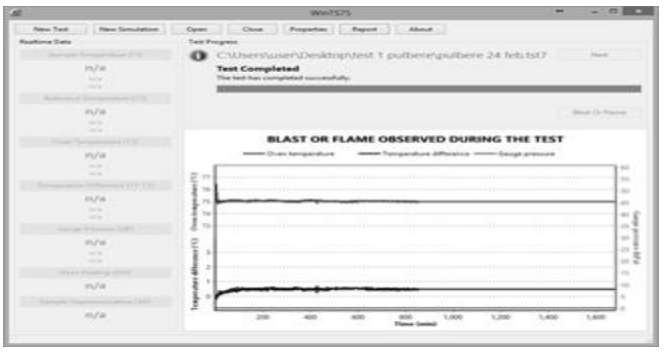

Fig. 13 Capture with the section "Opening a Test"

THERMAL STABILITY TEST (75°C) REPORT

Test Conditions	
Procedure	EN 13631-2:2002
Date Started	2/24/2014 1:43:49 AM
Sample Weight	5.0 g
Sample Volume	100.0 cm^3
Loading Density	0.1 $g.cm^{-3}$
Specimen Information	
Name	Pulbere fara fum
Specification	Pulbere fara fum cu nitroceluloza
Grain Size	
Typical Results	
Test Result	REACTION
Test Completion	Blast or flame observed during the test (28 hours and 2 minutes)
Sample Weight Change	Sample weight did not change
Sample Color Change	

Fig. 14 Capture with the section "Test Report"

CONCLUSIONS

1. Procedures determination of thermal stability of explosives, according to EN 13631-2, valid conditions for obtaining credible results, ensure the increasing technical quality premises for testing of explosives, while improving the confidence in evaluating the performance of these dangerous products, due to fully satisfy the safety requirements laid down in Directive 93/15/EEC.
2. Equipment purchased for verifying the security parameter allows the application requirements of the harmonized standard EN 13631-12 namely maintaining explosive at 75°C ± 2°C for 48 hours under continuous security monitoring of temperature and pressure and surveillance online.
3. The result after being tested on a sample of 100 g nitrocellulose smokeless powder, has highlighted the character thermally stable for this type of product, as confirmed by specific response highlighted in the document of the reporting, Test Report, namely,” No reaction’’.

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[3] Nucleu Program 2013/ Project PN 07 45 03 17. On the elaboration of technology for determining the thermal stability of explosives, Incd Insemex Petroşani, 2013.
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