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The danger of initiating explosives by electrostatic discharge. Checking the level of sensitivity of explosives to electrostatic discharges.

Dan Gabor[1]*, *Florin Adrian* Păun[1], and *Anca* Tăzlăuanu[1]

[1]National Research and Development Institute for Mining Safety and Explosion Protection – INSEMEX, G-ral Vasile Milea Street, no. 32-34, Postal code: 332047, Petrosani, Hunedoara County, Romania

Abstract. Explosives designed for civil uses can be, in some cases, be triggered by accident due to electrostatic discharge. Static electricity, as a source of electrostatic discharge, is a common phenomenon in the explosives manufacturing industry. Explosives designed for civil uses are substances, materials and accessories that present a high-risk factor during their production, packaging, storage, transport, use and disposal. In order to establish essential safety requirements for civilian uses explosives, national legislation has been harmonized with European legislation, respectively with Directive 2014/28/EU of the European Parliament and of the Council regarding the placing on the market and control of explosives for civil use, for handling with minimal risk to the safety of human life and health, to prevent damage to property and the environment, and to ensure the safety and health of persons coming into contact with civil uses explosives. In this context, it is necessary to apply high-performance test methods to determine the safety parameters for assessing the conformity of explosives for civil use with the safety requirements set out in the specified directive. This paper describes some aspects regarding the implementation of the testing method for checking the level of sensitivity to electrostatic energy of explosives within the Laboratory of Non-Electrical Ex Equipment, Electrostatics, Materials and PPE within INCD INSEMEX Petroșani [1, 2].

1 Introduction

Explosives for civilian use may, in some cases, be unexpected initiated due to electrostatic discharge. Propellants and rocket propellants also belong to the category of civilian explosives. The propellant is an explosive device used for propulsion or to reduce the friction of projectiles [3].

Static electricity, as a source of electrostatic discharge, is a common phenomenon in the explosives manufacturing industry. Explosives for civil use are substances, materials and

* Corresponding author: dan.gabor@insemex.ro

accessories with a high degree of risk in terms of their production, packaging, storage, transport, use and destruction. Determining the performance of the sensitivity of these devices to unexpected initiation by electrostatic discharge is very important as this depends on the safety and security of the person.

This paper presents some aspects regarding the implementation of the method of testing the resistance to electrostatic energy of propellants and rocket propellants in the laboratory of Non-Electrical Ex Equipment, Electrostatics, Materials and PPE from INCD INSEMEX Petrosani.

In order to establish essential safety requirements for explosives for civilian use, in order to handle with minimal risk to the safety of human life and health, to prevent damage to property and the environment, national legislation has been harmonized at European level. Thus, the placing on the market and control of explosives for civilian use is regulated by Directive 2014/28 / EU and the harmonized standards for the determination of resistance to electrostatic energy in rocket propellants and fuels are: SR EN 13938-1:2005/AC:2006 Explosives for Civil Uses. Propellants and Rocket Propellants: Part 1. Requirements and SR EN 13938-2:2005 Explosives for Civil Uses. Propellants and Rocket Propellants: Part 2. Determination of resistance to electrostatic discharge [4, 5].

In this context, the application of high-performance test methods for the determination of safety parameters is particularly important for the assessment of the conformity of explosives for civil use with the safety requirements of the specified directive.

The technical-scientific development in the field of both the production of rocket propellants and propellants and of laboratory equipment imposes the need to develop new methods of testing / evaluating the conformity of products with safety requirements in accordance with international principles and practices.

2 Determination of resistance to electrostatic discharges of propellants and rocket propellants. Operating procedure

In choosing the test method for determining the sensitivity to electrostatic discharge, the following shall be considered:

- The diversity of the types of products that must be tested for evaluation and for which specific test devices must be made,
- The diversity and complexity of the tests to which the products must be subjected in accordance with the requirements of European standards,
- The necessity to use precision apparatus calibrated to the prescribed parameters,
- The necessity to ensure special test conditions both in terms of the test environment / preconditioning and the use of hazardous materials,
- Ensuring the required level of reliability and traceability to standards.

The equipment and software used for testing, calibration and sampling must be capable of achieving the required accuracy and must comply with the relevant specifications for the respective tests and / or calibrations, state-of-the-art research and development equipment for the development of the above tests.

The method of testing propellants and rocket propellants to determine resistance to electrostatic energy is to apply a capacitive discharge pulse between two electrodes placed on a cell filled with the propellant fuel to be tested.

2.1 Identifying solutions for implementing a new method of testing propellants and rocket propellants for sensitivity to electrostatic discharge

For the testing of propellants and rocket propellants regarding the sensitivity to electrostatic discharge, the following equipment requirements have been developed:
• The equipment allows several test modes in accordance with the standards SR EN 13938-2:2005/AC:2006,
• The equipment must determine the exact value of the minimum energy sufficient for an accidental initiation of the sample,
• The test equipment must be safe and for this it must be able to perform the tests on small samples, so it must be of the small-scale electric spark sensitivity testing apparatus,
• The equipment must be designed and constructed for the accurate measurement of the initiation energy of crystalline energy materials in the field of discharge energies (from 25 pJ to 25 J) and at voltages up to 10 kV, with a typical probable mass of approximately 10mg,
• Maximum 40 tests to perform a complete test,
• Operation in two discharge regimes - oscillating and damped - for testing the sensitivity to electrostatic spark (electrostatic discharge),
• To allow the testing of all categories of civil explosives, from extremely sensitive primary explosives to insensitive large explosives,
• Analysis and evaluation software,
• Built-in high voltage capacitors and damping resistors,
• Working capacities: 5; 15; 30; 100; 150; 200 nF,
• Output voltage: 0.5 - 10 kV,
• Allow connection of external capacitors,
• Discharge energy range from 25 pJ to 25 J,
• Maximum sample volume/approx. weight: 2.2 mm^3 / 5 mg and 94 mm^3 / 188 mg,
• Requirements for generator:
  - The maximum error for capacity must be ± 10% of the required value,
  - The maximum voltage error must be ± 5% of the required value,
  - The total inductance of the discharge circuit must not exceed 5 μH,
  - The total dynamic resistance of the discharge circuit must be 5 Ω ± 0.1 Ω with a load of 10kV on the capacitor,
  - If an additional series resistance is used, this resistance must be R_0 ± 5%.

2.2 The chosen equipment for determination of resistance to electrostatic discharge

One of the equipment that meets the test requirements identified above is the **X Spark 10 small-scale electrostatic spark sensitivity tester**, figure 1. The equipment is produced by OZM-Research and it was developed in collaboration with the Institute of Energetic Materials, University of Pardubice, Czech Republic. This equipment was completed with the accessories requested by us (cells and cell caps, HV cables and so on). Only the generator in this equipment was used to determine the electrostatic discharge sensitivity of propellants and rocket propellants. The equipment is also used for testing electrical detonators and energetic materials [6].

We chose this device because it is one of the very few available instruments of this type, allows for the precise measurement of both the total spark energy discharged into the sample and the fraction of this energy that is absorbed by the sample to initiate its explosion. This feature allows exact determination of the minimum energy sufficient for an accidental initiation of the sample.

The X Spark 10 apparatus is also dedicated to measure the sensitivity of crystalline high explosives, pyrotechnics, as well as primary explosives in the granular or crystal-like form. The equipment is part of the latest generation of test instruments for the precise measurement of the initiation energy (sensitivity to electrostatic spark) of explosive materials. It is designed for the accurate measurement of the initiation energy of propellants and rocket propellants in the field of discharge energies (from 25 pJ to 25 J), at a voltage of up to 10 kV and with a typical probable mass of about 10 mg. With this equipment few tests are required to determine the sensitivity, usually about 30-40 tests with different spark energies are required to perform a complete test [6].

The compact design (single portable unit) of the equipment offers the best conditions for maximum electrostatic discharge efficiency. This increased efficiency is ensured by a low resistance and inductance of the discharge circuit, the shortest possible cable length and a high discharge energy at a low voltage value. The analysis and evaluation are performed with the WINSPARK software [6].

The small-scale equipment for determining the electrostatic resistance of propellants and rocket propellants consists of: a built-in high-voltage power supply, a capacitor bank, a damping resistor, a remotely controlled high-voltage switch, a set of additional external capacitors, a remote control, a set of accessories that include cells and the cell caps required by the standard SR EN 13938-2:2005/AC:2006 (figure 2) [5].

The working capacity is selectable and can be completed with external capacitors. The output voltage, of the order of kV, is variable and can be adjusted from a potentiometer or from the remote control. The voltage value is displayed on the equipment and on the remote-control screen [6].

Cells and cell caps (figure 2) consist of:
- a polyvinyl chloride disc, thickness (3.0 ± 0,1) mm, diameter (32 ± 1) mm with a central perforated hole, diameter (6.3 ± 0,1) mm,
- a copper disc about 1 mm thick, diameter (19 ± 1) mm, which forms the base of the cell.

The plastic disc is fixed to the copper disc with an adhesive border around the outer edge. The lid consists of a copper disc about 0.1 mm thick (16 ± 1) mm in diameter which is fixed with a double-sided tape to the top of the plastic disc.

Fig. 1. X Spark 10 small-scale electrostatic spark sensitivity tester.

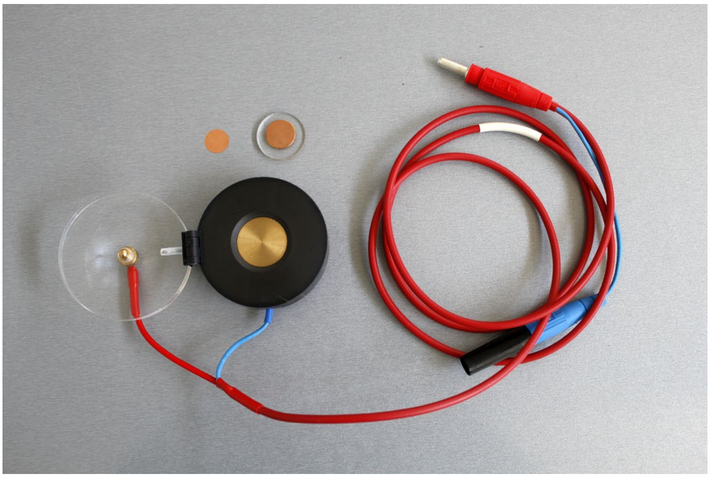

Fig. 2. Spark gap assembly according to SR EN 13938-2:2005/AC:2006. Cells and cell caps.

2.3 Preparation of the test sample

Take a 100 g sample and sieve according to the sieving method from SR ISO 565: 1997 - Test sieves - Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings [7]. The sieves used have the mesh size according to Table 1. For the test the fraction passing through the 1.0 mm mesh sieve shall be used when this fraction is greater than or equal to 5 g. Otherwise the test shall not be performed. The test sample should be conditioned at (20 ± 5) ^{0}C and (60 ± 10) % relative humidity for 24 h.

Table 1. Mesh size.

Number *n*	**1**	**2**	**3**	**4**	**5**	**6**	**7**	**8**
Mesh size (mm) *Xn*	0.10	0.16	0.25	0.40	0.63	1.0	1.6	2.5

2.4 Operating procedure

2.4.1 Calibration of the electrostatic energy generator

This calibration is necessary to ensure that the generator used by the equipment will provide satisfactory results from one test site to another.

Only the generator discharge circuit, including the cables used to connect the test cell, is calibrated.

The equipment required for calibration consists in a high voltage probe, a current probe (Rogowsky coil) and an oscilloscope.

The generator is calibrated according to the procedure given in SR EN 13938-2:2005, annex C [5].

Practically, this procedure calibrates the voltage and capacity of the generator and determines the inductance and dynamic resistance of the discharge circuit.

Two curves will be recorded, one voltage as a function of time and one current as a function of time (figure 3).

If a series resistor is used, an additional calibration must be made.

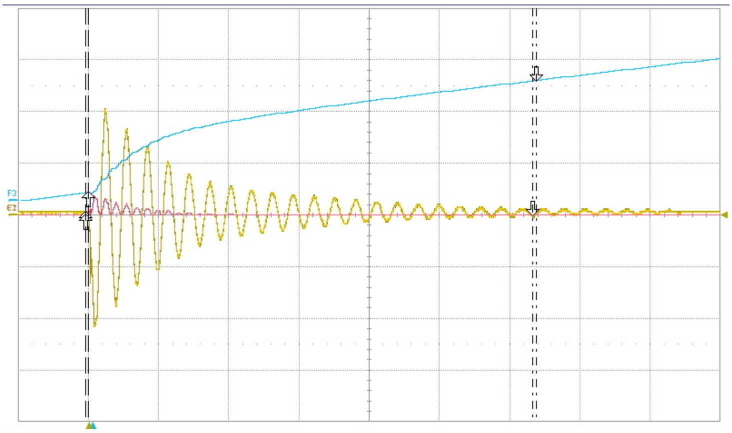

Fig. 3. Example of recording an electrostatic discharge (current as a function of time).

2.4.2 Description of the working method

After calibrating the equipment, tests can be performed [5]. To perform the tests, proceed as follows:

• Fill the cell, i.e., the hole in the plastic disc, with a part of the sample so that the lid is in contact with the propellant without pressing it. Close the cell with the lid using double-sided tape and leave at (20 ± 5) ^{0}C and (30 ± 10) % relative humidity,

• Place the cell on the lower electrode. Then bring the upper electrode into contact with the cell cover. Select a capacitor and charge it by applying a voltage of 10kV. Then discharge the capacitor through the electrodes,

• During the test and when the rest of the test sample is recovered, it is observed whether a reaction or a partial reaction occurs, i.e., a positive event. The reaction is the sound of an explosion, crackle, spark and / or flame and partial reaction is a change in color, the opening of a cell, or traces of heat on the cell surface,

• A new cell must be used for each series of tests. Starts with an energy level of 5 J (capacity 0.1μF). Test 20 parts of the sample at an energy level of 5 J. If a reaction or a partial reaction occurs, stop the test and proceed to an energy level of 0.5 J (capacity 0.01μF) for the next 20 tests. If a reaction or partial reaction is recorded, continue with an energy level of 0.05 J (capacity 0.001 μF) for another 20 tests if no reaction or partial reaction occurs,

• The test result is reported as the limit energy, i.e., the maximum energy level without reaction or partial reaction in a series of 20 tests. For example: if a reaction or partial reaction is obtained at an energy level of 0.05 J the limit energy will be expressed as < 0.05J.

3 Conclusions

The application of high-performance test methods for the determination of safety parameters is particularly important for the assessment of the conformity of products with the safety requirements set out in Directives 2014/28/ EU and 2013/29/ EU [1, 8].

The studies carried out in the research activity focused on the DEVELOPMENT OF TECHNICAL SOLUTIONS FOR THE REALIZATION OF TEST STANDS to verify the sensitivity of propellants and rocket propellants to electrostatic discharge, in order to prevent unexpected detonation by electrostatic discharges.

For the elaboration of the test procedure and for the implementation of the test method in the quality system of the laboratory, in order to extend the field of competence of the laboratory, the following were performed:

- Analysis of technical solutions for the construction of the test stand,
- Development of a test stand for testing propellants and rocket propellants on the performance of protection against uncontrolled initiation by electrostatic discharges,
- Experimentation and implementation of the procedure in the accredited laboratory for tests.

From the analysis of the standardized test requirements, the technical characteristics of the test equipment and the results obtained, it can be seen that the stand offers the possibility to determine the sensitivity of propellants and rocket propellants to electrostatic discharges, in accordance with standardized test requirements. By implementing the elaborated procedure, the field of competence of the laboratory was extended with a new accredited test.

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