MINISTRY OF EDUCATION AND RESEARCH
UNIVERSITY OF PETROŞANI

GABOR DAN SORIN, ENG.

DOCTORAL THESIS

- ABSTRACT -

RESEARCH INTO EXPLOSION RISKS CAUSED BY STATIC ELECTRICITY IN INDUSTRIAL ENVIRONMENTS

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PETROȘANI
-2020-

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IMPORTANCE AND NECESSITY OF THE TOPIC. THESIS OBJECTIVES AND STRUCTURE

Static electricity is a phenomenon frequently met in industrial activities. In the presence of combustible matter and/or matter capable of forming explosive atmospheres in air or in the presence of explosives, electrostatic discharges from equipment, materials or persons can generate fires and explosions.

In the thesis I approached the subject of explosion risk assessment of forming, accumulation and discharge of electrostatic charges in potentially explosive atmospheres, as well as the risks of inopportune initiation of electric detonators, pyrotechnic articles, rocket propellants and fuels. The aim of explosion risk assessment is to establish adequate protection measures for its prevention or limiting.

The importance of the paper lies in the necessity of creating application mechanisms of the European Directives, which were transposed in the national legislation:

- Directive 2014/34/EU of the European Parliament and of the Council of 26th February 2014 on the harmonisation of the laws of Member States relating to equipment and protective systems intended for use in potentially explosive atmospheres
- Directive 2014/28/EU of the European Parliament and of the Council of 26th February 2014 on the harmonisation of the laws of the Member States relating to the making available on the market and supervision of explosives for civil uses.
- Directive 2013/29/EU of the European Parliament ands of the Council of 12th June 2013 on the harmonization of the laws of the Member States relating to the making available on the market of pyrotechnic articles.

In the doctoral thesis I have presented the results of the theoretical studies and research conducted with the purpose of assessing the risk of ignition of explosive mediums, taking into account the requirements of the European norms and standards, focussing on laboratory trials and simulations of the identification of safety requirements with emphasis on determining the susceptibility of pyrotechnic articles, materials/devices of ignition by electrostatic discharges.

The paper has three chapters:

1. Static electricity, electric detonators, pyrotechnic devices, rocket propellants and fuels as initiation sources for explosive atmospheres.
2. Analysis of assessment/testing requirements of personal protective equipment (PPE) to reduce ignition risk of the explosive atmospheres by electrostatic discharges from persons;
3. Danger of electric detonators, pyrotechnic devices, rocket propellants and fuel ignition by static discharge.

The three chapters are followed by CONCLUSIONS, References and Annexes.

All along the paper I have highlighted the personal contributions, starting from the ***theoretical*** studies on charge/build up and discharge phenomena of electrostatic charges, as well as the factors of influence, ignition phenomena of explosive atmospheres or pyrotechnic devices/materials and identification of safety requirements, highlighting the present status of knowledge, and ending with the ***practical*** creation of laboratory testing stands.

The consistent and valuable part of the personal contributions lies in:

- *Innovative solutions in creating the laboratory testing stand for textile materials, stand to verify antistatic characteristics, followed by the trial/testing of the stand, validation of the method of* *testing and its implementation in the nationally accredited testing laboratory quality system GLI INSEMEX;*

- *technical solutions to design the testing stand/effective assembly of the stand for testing electric detonators regarding protection characteristics against uncontrolled initiation by static discharges, experimenting and implementing the procedure in the accredited testing laboratory;*
- *Innovative method to determine susceptibility to static discharge of rocket propellants and fuels, identification of technical solutions to conduct tests, development of solutions for the implementation of the new testing method of rocket propellants and fuels in order to assess conformity with the prevention requirements against inopportune detonation by static discharge.*

By the practical application of the results obtained, I have made an important contribution to the development of protective means and methods against static electricity, in view of providing a high level of labour safety in areas with potentially explosive atmospheres and in blasting works.

Theoretical and practical studies, analysis of technical and safety requirements, experimenting testing methods, laboratory research and testing methods to assess and test ignition danger for technical equipment, resulted in improvement of the effectiveness of the present system of testing and carrying out the assessment necessary for the certification of equipment and materials conformity with the requirements of the European Directives.

Development of the testing methods by developing testing and assessment methodologies, devising and designing testing stands, their experimentation, and validation of the testing methods, contribute to the development of the present testing/certification system's effectiveness of the Laboratory of Ex Nonelectric Equipment, Electrostatics, Materials and PPE within INCD-INSEMEX.

Development of new testing methods and procedures, following international principles and practices, provides accurate assessment of technical equipment's characteristics. This equipment is intended to be used in potentially explosive atmospheres. They align to the European practice in the field, as well as to the improvement of the testing laboratory according to principles and requirements of SR EN ISO/IEC 17025:2018 standard, to provide necessary data for the conformity assessment with the essential safety and health requirements stipulated in the applicable European Standards.

Development and creation of testing methods/procedures, by providing the necessary facilities/infrastructure, offers the possibility of knowing all the essential aspects related to the safety of the technical equipment, considering the generally ascending trend of increasing health and safety level of the workers in industries with explosion danger.

It is to be noted that I have an invention patent and 4 invention proposals in the field of mining engineering and electrical engineering registered at the State Office for Inventions and Trade Marks – OSIM.

CHAPTER 1
STATIC ELECTRICITY, ELECTRIC DETONATORS, PYROTECHNIC DEVICES, ROCKET PROPELLANTS AS INITIATION SOURCE IN EXPLOSIVE ATMOSPHERES

Formation, accumulation and discharge of electrostatic charges.

Electrostatic charges are formed and built up as a result of electrisation mechanisms, of which contact electrisation is the most frequently encountered. Two substances, of different nature, which are brought into contact and then separated, will carry electrostatic charges of equal size and of opposite signs.

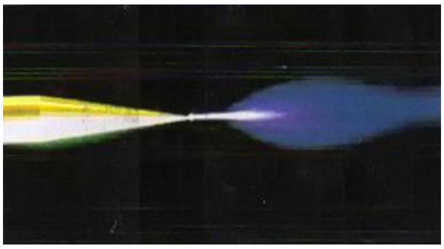
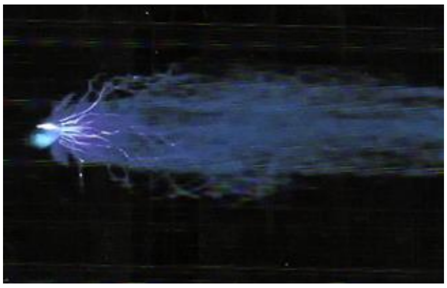
Similarly, another component of the electrisation mechanism is induction electrisation, when the conductive materials can be charged with static charges of electrostatic nature, from a nearby charged object. Products/materials can thus get transfer charges, either directly, from other objects, or by influence, from a stream of ions.

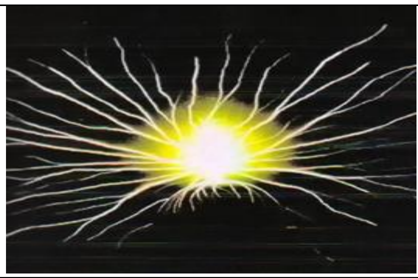
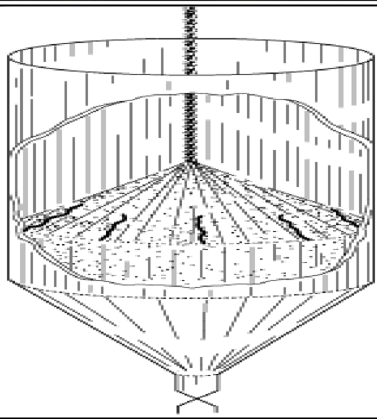
Electrostatic charges built up on a solid or on a liquid are dangerous only if they are transmitted (discharged) to another body or to the earth. These discharges vary a lot as type and degree of potential initiation.

After separation during the electrostatic charging process, the charges can rapidly recombine, either directly, by contact, or through the earth. The charges from a non-conductor are retained due to the material itself. But in order for a conductor to stay charged, it has to be isolated from other conductors and the earth.

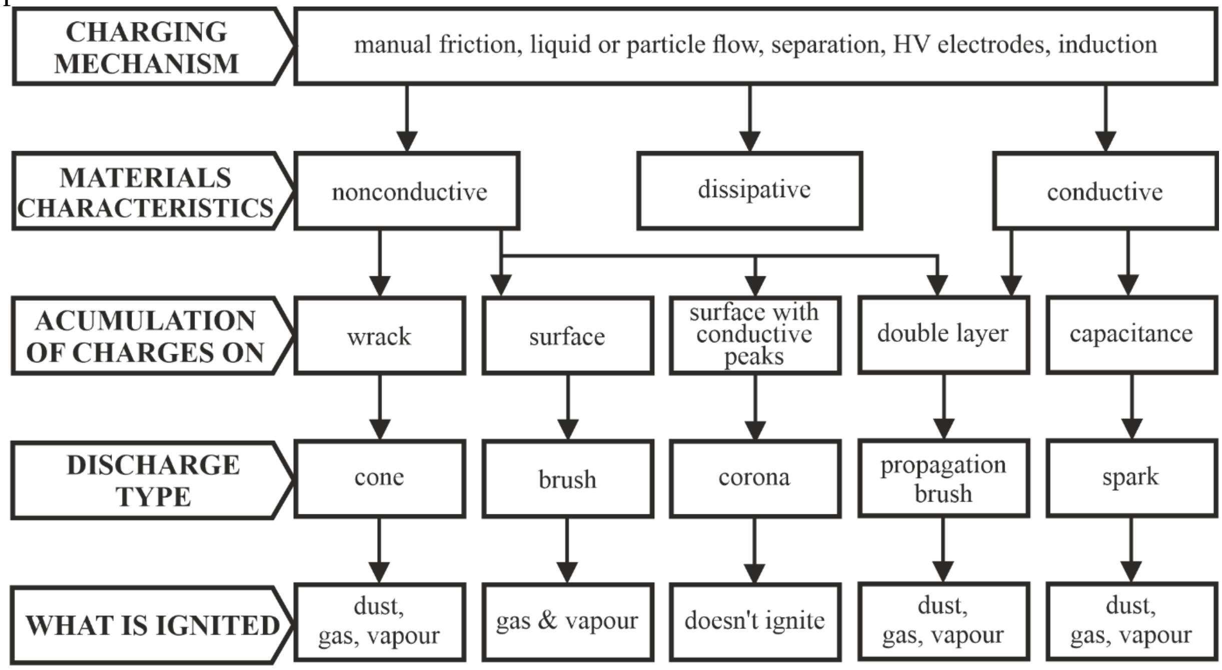
Static electricity discharges vary a lot as type and degree of potential initiation.

A synthesis of various types of electrostatic discharges is shown in the following Table:

Electrostatic discharges		
Sparks	Discharges between two conductors that can be solid or liquid	Spark energy $W = Q \cdot V = {}^{1}/_{2}\, C \cdot V^2$, where: W is dissipated energy, in Joule; Q is the charge quantity of the conductor in Coulomb; V is the electric potential in Volt; C is capacity, in Farads.
„Corona" discharges	Discharges take place in sharp areas or edges of the conductors. "Corona" discharge can take place when a grounded conductor, with sharp areas, is moved in the direction of a highly charged object, or when the latter's electric potential is significantly increased.	
Brush discharges	Can take place when round conductors (as opposed to sharp ones) connected to the grounding network, are moved towards poorly conductive, charged objects. This type of discharge can occur, for instance, between the finger of a person and a plastic surface.	

Propagation brush type discharges	Discharge between the two surfaces of a sheet of high resistivity material and a high dielectric resistance, highly charged with opposed polarity charges.	
Cone type discharge	Discharge occurring in silos or large containers when these are filled with dust (poorly conductive), highly charged (a high charge density area is generated inside the dust pile).	

The following Figure presents a synthesis of the capacity of being ignited of various discharge types:



Of the prevention measures of static electricity discharges, the following can be mentioned:

- grounding;
- use of adequate materials;
- rendering materials antistatic;
- selecting adequate constructive shape (surface, distance to grounded conductive elements, widths of nonconductive materials);
- avoiding dangerous frictions (limiting moving speed of belts or flow speed through ducts);
- environmental conditions (high humidity);
- using charge neutralizers.

Ignition risk assessment of explosive atmospheres by electrostatic discharges

Assessment and determination of ignition potential occurrence of electrostatic discharges in various real situations is the most important and difficult step in the analysis of the dangers generated by electrostatic discharges.

The data required for a reliable analysis are:

- exact knowledge of the characteristics of the potentially explosive mixture that might be present;
- resistances or conductivity of substances, apparatus, packages, materials and personal equipment used;
- volumes and geometrical arrangements of technical installations and devices, as well as
- accurate knowledge of the existent conditions of ground leakage and equilibrium conditions of the electric potential.

Explosion risk assessments should thus take into consideration the probability of forming electrostatic charges and their discharge both on equipment and on products or materials.

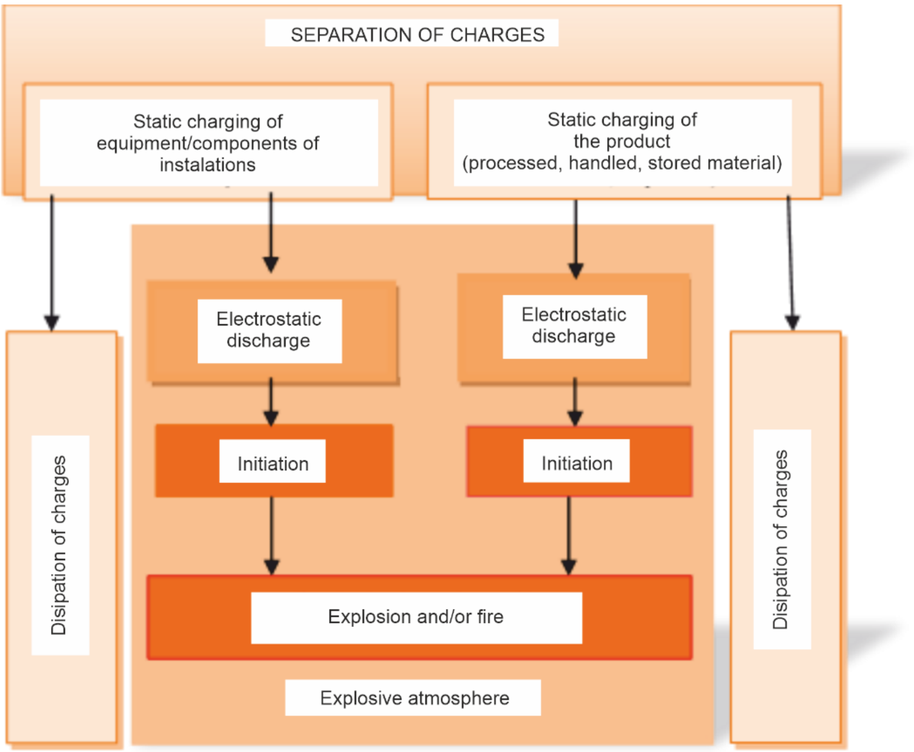


Diagram of stages leading to initiation of an explosive atmosphere by electrostatic discharges

Knowing the ignition susceptibility of the discharge (namely the released energy), and the sensitiveness of the potentially explosive atmosphere, as it was characterized by minimum ignition energy (MIE), it can be established whether the ignition occurs or not.

Unexpected initiation risk analysis of explosives and /or initiation devices by electrostatic discharges

In the case of electrical detonators, rocket propellants and fuels there is a risk of their being initiated unexpectedly, by means of electrostatic discharges. In the explosives manufacturing industries, the presence of static electricity and implicitly of electrostatic discharges cannot be totally prevented, although in this activity sector a risk assessment of uncontrolled (inopportune) initiation is necessary. For this assessment the susceptibility to electrostatic discharges ought to be determined.

Depending on the susceptibility to electrostatic discharges, adequate static electricity discharges prevention methods have to be applied, as in the case of explosive atmospheres.

For the initiation of explosive charges, several electric detonator types are used worldwide, as a consequence of the efforts of the manufacturing firms to satisfy the requirements expressed by the beneficiaries, corresponding to the different conditions in which the blasting operations are carried out, in order to obtain the required performance, as well as to provide a high safety level in order to prevent the risk of inopportune detonations from exterior initiation sources.

There are three ways of cap initiation by static discharges:

- discharge between wires, by the normal way of cap initiation – RR (Fig. 1a);
- discharge between the cap tube and a wire – T1R (Fig. 1b);
- discharge between the cap tube and the short-circuited wires – TRS (Fig 1c).

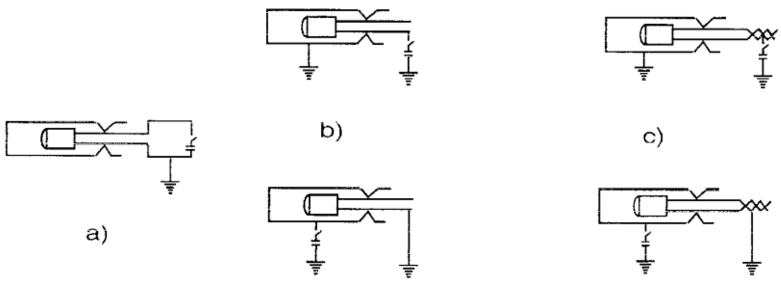
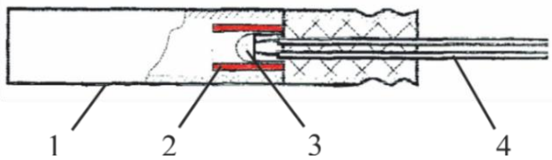


Fig. 1 – ***The three ways of electrostatic discharge***

I have conducted an analysis of the protection performance of the different types of detonators starting from the parameters guaranteed by the manufacturers, depending on the minimum energy of electrostatic discharge that might initiate the detonator, largely based on the research and studies performed at INCD INSEMEX and completed with information from the literature.

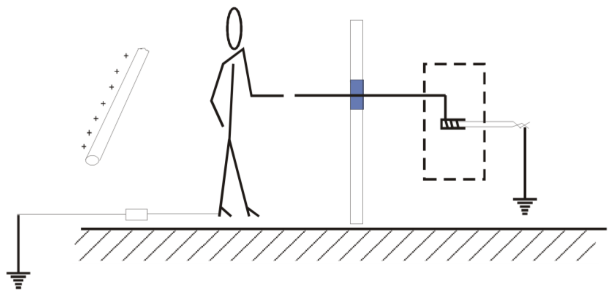
Similarly, I presented the research carried out at INCD INSEMEX to increase protection performance of detonators against electrostatic discharges, which were translated in the development of an adequate constructive solution of limiting the dangerous discharges between the tube and the electric ignition device, which lies in a protection sleeve put over the ignition device (Fig 2)



1 - tube, 2 – protection sleeve, 3 – ignition device, 4 - wires

Fig. 2 – ***Detonator with protection sleeve***

Similarly, the experiments made on the ignition risk of low intensity detonators by static discharge from people and the protective measures required depending on the type of detonator used are presented, including a case study on establishing the causes leading to a workplace accident due to inopportune initiation of an electric detonator, with one casualty.



Electrisation of people from charged equipments

CHAPTER 2

ANALYSIS OF ASSESSMENT/TESTING REQUIREMENTS OF PERSONAL PROTECTIVE EQUIPMENTS (PPE) TO REDUCE THE RISK OF EXPLOSIVE ATMOSPHERE IGNITION BY ELECTROSTATIC DISCHARGES FROM PEOPLE

In the first part of this chapter I have presented several general considerations on the charging, accumulating and discharge mechanisms of electrostatic charges on people and/or on personal protective equipment.

Persons insulated from the ground can easily acquire and retain an electrostatic charge. Insulation from the ground can be due to the fact that the cover of the floor or the soles of the shoes are made of non-conductive material. Electrostatic charging of a person can occur when people walk on the floor, stand up from the chair, take off their shoes, handle plastic materials, empty or collect charged materials in or from a container, stay close to seriously charged objects (for example next to a moving belt), or by induction.

When a statically charged person touches a conductive object, (for example the handle of a door, a rail, a metal container), a spark can occur in the contact point. Such sparks, that are less likely to be seen, heard or even felt by the person, can ignite gas, vapour or even sensitive dust. It is very important to prevent static charges on persons that might be exposed to flammable atmospheres, atmospheres that have low minimum ignition energy.

Requirements regarding the assessment of personal protective equipment (PPE) from the point of view of protection performance against dangerous static electricity

The requirements regarding prevention of dangerous electrostatic discharges from persons are given in the guide *IEC TS 60079-32-1:2013+AMD1:2017 „Explosive atmospheres - Part 32-1 Electrostatic hazards, guidance"* and in the Romanian Standard *SR EN 60079-32-2:2015 „Explosive atmospheres. Part 32-2: Electrostatic hazards. Tests".*

Requirements for the assessment of materials and PPE from the point of view of protection performance against hazardous static electricity are given in *Regulation (EU) 2016/425 of the European Parliament and of the Council of 9 March 2016 on personal protective equipment and repealing Council Directive 89/686/EEC* and in the standards specific to equipment types *(SR EN 1149-1,2,3,5)*, footwear *(SR EN ISO 20344, SR EN ISO 20345, SR EN ISO 20347)*, gloves (*SR EN 388:2004)* or protective helmets (*SR EN 812 + A1:2003, SR EN 443:2008, SR EN 397 + A1:2003).*

From the analysis of the above standards, one can notice that, with the exception of protective clothing, for all the other equipment, the protective requirements refer to the limiting of the electrical resistance, and the testing methods are applied in the laboratories of INCD INSEMEX.

Therefore, the studies carried out by me focussed on identifying technical solutions ***and designing and building the testing stand for textiles to measure dissipation capacity of charges*** in accordance with the requirements of SR EN 1149-3:2004 standard.

With regards to the ***selection of personal protective equipment (PPE) for use in areas with explosion hazard,*** I have carried out an ignition risk analysis associated with static discharges from clothing, depending on the presence of explosive atmospheres and their susceptibility to ignition. Various risk levels have been identified related to Ex hazardous areas, areas classified in zones according to EN 60079-10-1 and EN 60079-10-2 standards and depending on the susceptibility of the flammable material to ignition, and minimum ignition energy, respectively (MIE). In my analysis I took into consideration the probability or a charging mechanism to occur.

Table 1 provides guidance referring to statically dissipative protective clothing and other necessary PPE, recommended or which are not necessary based on global risk, which are a combination of the probability of occurrence of an explosive atmosphere, probability of occurrence of a charging mechanism, and ignition susceptibility of an explosive atmosphere by static discharges.

<table>
<tr><th rowspan="2">Hazardous zones</th><th rowspan="2">Probability of charging</th><th rowspan="2">0,016 mJ ≤ MIE ≤ 0,2 mJ
IIB and IIC explosion groups</th><th colspan="2">MIE > 0,2 mJ</th></tr>
<tr><th>IIA and IIB explosion groups</th><th>IIIA, IIIB and IIIC explosion groups</th></tr>
<tr><td rowspan="2">Zone 0</td><td>high</td><td rowspan="4">necessary</td><td rowspan="3">necessary</td><td rowspan="6"></td></tr>
<tr><td>low</td></tr>
<tr><td rowspan="2">Zone 1</td><td>high</td></tr>
<tr><td>low</td><td rowspan="2">recommended</td></tr>
<tr><td rowspan="2">Zone 2</td><td>high</td><td>Not required</td></tr>
<tr><td>low</td><td>Not required</td><td>Not required</td></tr>
<tr><td rowspan="2">Zones 20, 21 and 22</td><td>high</td><td colspan="2" rowspan="2"></td><td rowspan="2">Not required</td></tr>
<tr><td>low</td></tr>
</table>

Tab. 1 – Requirements for statically dissipative protective clothing and other PPE

Charging depends on a series of factors and should be evaluated from case to case. Although the nature of materials largely determines their tendency to be charged, environmental factors, namely temperature and humidity, also have a significant influence.

In subchapter 2.4 of the thesis, I have conducted *an analysis of the testing methods for textile clothing/materials to determine the protective effectiveness of ESD* with a view to establishing the most adequate methods of testing in order to assess the conformity and certification of PPE, according to the *Regulation (EU) 2016/425.*

First, I described the type of materials for clothing, highlighting the new types of composite materials, which have a network of conductive stripes inside the cotton, polyester or mixture of those, or which have conductive stripes manufactured from a mixture of conductive and non-conductive fibres (fibres that are conductive on the surface, conductive in the core, sandwich type fibres et.) , then analysed the testing methods applicable to those.

As new types of fabrics are developed, testing/characterisation of those become difficult indeed, quality control tests and effective functional tests being necessary. Certain international standardization bodies, including CEI, CENELEC and ISO, as well as various individual laboratories from several countries have responded to the challenge and approached projects or activities, which should develop standard testing methods in this field.

The static component of the homogeneous material, isotronic, is usually described by two features: surface or volume resistivity/resistance and charge decay. Static performances of the material of exterior clothing, of composite, heterogeneous materials, cannot be described in this manner, since by measuring the resistance between two electrodes placed on the material one can get a very low value if the electrodes are in contact with the conductive matrix, but one can also obtain a very high value if the contact is made only with the electrically non-conductive substrate. Similarly, the measurements of the electric discharge at macroscopic level can show extremely fast charge losses through the conductive matrix, but a quasi-infinite charge retain at the insulator. Since the contact between the electrodes and the material cannot be identical, the measurement cannot be repeated.

In order to avoid a failure as that of the initial approach, based on determining characteristics such as charge resistance and discharge, to determine the performances of the exterior protective footwear, the identification of new characteristics and/or adequate testing procedures became necessary, for a reliable characterization and/or assessment.

In chapter 2.4.2 I made a study of testing methods for textile clothing/materials to determine ESD protection performances.

The applicable tests for the determination of ESD protection parameters can be grouped in two categories:

- tests made on materials.
- tests made on clothing.

The following testing methods have been thus carried out for textile materials:

- Resistive methods of IEC 61340-5-1 (surface resistance, resistance point by point);
- Resistive methods of EN 1149-1 an EN 1149-2 (surface resistance, vertical-transversal resistance);
- Surface resistivity according to EN 100015-1 (substituted with IEC/EN 61340-5-1);
- Test of induction charging according to EN 1149-3;
- Test of tribo-electric charging according to EN 1149-3;
- Test of charge decay – contact charging (VTT method);
- Test of corona charging according to IEC / EN 61340-2-1;
- Test of “Capacitance test” of John Chubb (corona charge) with the help of JCI155 and JCI176 measuring devices.

Study of testing methods of finite clothing includes:

- Resistive methods described in standard IEC 61340-5-1;
- Resistive methods in standard ESD STM2.1;
- VTT measuring method of charge decay time for ESD protective clothing;
- SP 2175 method "Measuring charge decay time for ESD protective footwear”;
- STFI testing method nr. PS07 version 01/03 Rev. A "Testing method for the determination of the electric potential of the body and charge transfer by wearing statically dissipative protective footwear” (charge transfer);
- Shirley 202 method "Testing method for measuring static electricity generated during taking off clothing from the human body”;
- Method from standard JIS L 1094:1997 "Method of quantitative measurement of electricity generated by charging by friction”;

Analysis of applicability of testing methods to assess protection performances at static discharge of protective clothing used in potentially explosive atmosphere

The requirements for the prevention of discharges that can ignite explosive mixtures refer to the use of materials which are statically dissipative for exterior layers of protective clothing. These requirements might not be sufficient in the case of oxygen enriched atmospheres.

Since the requirements are for materials, the applicable testing methods, according to the standards in force, reefer to the testing of materials for their characterization from electrostatic point of view. Testing on complete clothing is still in study. As long as such tests are not available, it is not possible to make a complete assessment of the electrostatic features of protective equipment.

The present status of the knowledge in this field is reflected in the EN 1149 standards series „Protective clothing. Electrostatic features”, standard that includes five parts, namely:

- Part 1: Testing method for measuring surface resistivity;
- Part 2: Testing method for measuring electric resistance at traversing materials (certical resistance);
- Part 3: Testing method for measuring charge dissipation capacity;
- Part 4: Testing clothing (in development);
- Part 5: Performance requirements for materials and design requirements.

These standards have been developed following research carried out within a European project based on hydrogen atmosphere initiation testing. Consequently, the recommended acceptance limits are correlated with the minimum ignition energy or charge of flammable materials, gas or dust, with a covering safety coefficient dust to high sensitiveness of hydrogen mixture, compared to other flammable substances.

Performance requirements for materials from standard SR EN 1149-5 are for the materials that are dissipative from electrostatic point of view to correspond to at least one of the following requirements:

- $t_{50\%} < 4s$ or $S > 0,2$ when the material is tested with the second method (testing by induction) stipulated in SR EN 1149-3, where $t_{50\%}$ is the charge halving time and S is the protection coefficient;
- surface resistance to be $\leq 2,5 \times 10^9\ \Omega$ on at least one of the surfaces, when the material is tested according to standard 11491-1;
- for the materials containing grid shaped conductors, the distance between those should not be more than 10 mm in any direction.

PERSONAL CONTRIBUTIONS TO THE DEVELOPMENT OF LABORATORY TESTING FOR THE VERIFICATION OF ANTISTATIC FEATURES OF TEXTILE MATERIALS

Establishing the necessary tests for the assessment of conformity of clothing with the requirements applicable to safety, according to the new European standards

From the "analysis of the applicability of testing methods to assess protection against static discharges of protection clothing used in potentially explosive atmospheres", it results that the testing methods that should be carries out in the INCD INSEMEX laboratories for the verification of the dissipation characteristics of the static charges refer to the testing of textile materials for the determination of charge dissipation characteristics.

Consequently, the methods of characterization of materials as being statically dissipative are those described in SR EN 1149, namely:

- Part 1: Testing method for measuring surface resistivity;
- Part 2: Testing method for measuring electric resistance at traversing materials (vertical resistance);
- Part 3: testing method for measuring charge dissipation capacity – Method 2 with induction charging of the material.

Since the resistive methods have already been implemented in INCD INSEMEX laboratories, it was necessary to develop testing methodology for measuring charge dissipation capacity, and to determine the charge halving time of the screening factor, respectively, according to method 3 of SR EN 1149 – 3.

In this sense I developed the documentation for the design of the stand for measuring charge dissipation achieved by the testing method that uses induction charging, and constructed the testing stand.

The schematics of the principle governing the function of the stand for measuring charge dissipation capacity by the testing method using induction charging is presented in Fig. 3.

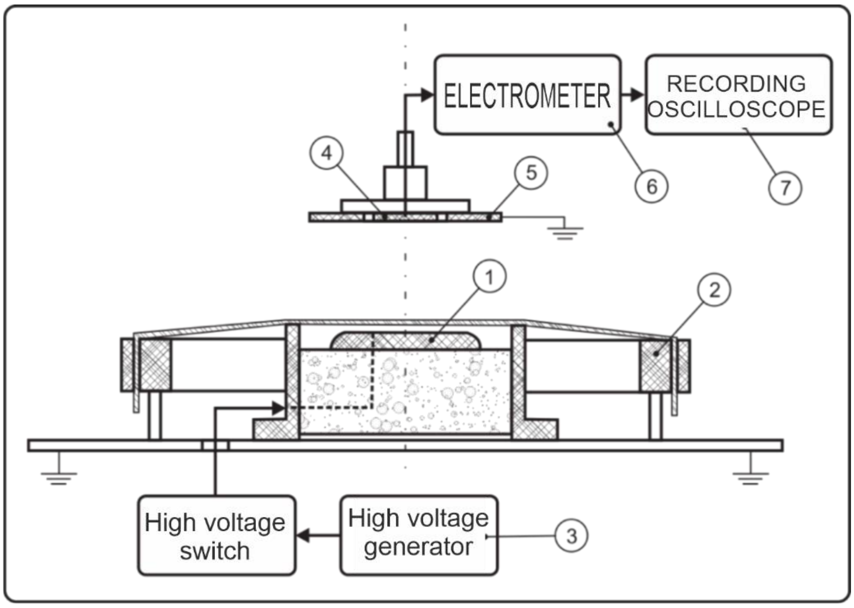
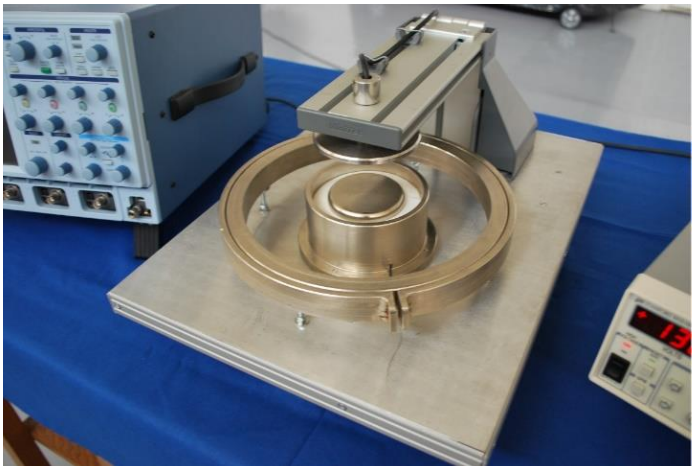


Fig. 3 - ***Schematics of the principle governing the function of the stand for testing textile materials with induction charging method***

The component parts of the testing stand are:

- 1- A disk shaped field generating electrode, of stainless-steel construction, 70 ± 1 mm diameter, fixed to a non-conductive support;
- 2 – A support ring (metal ring, 100 ± 1 mm inside diameter, earthed and placed around the field electrode) together with a ring for tightening the sample;
- 3 – A high voltage generator capable to provide the field electrode a stable direct current of 1200 ± 50 V. The tension is applied to the electrode with the help of a high-speed electronic switch, a switch providing a voltage rise front in 30 μs interval (an adjustable d. c. source of 5000V and a rapid HT switch);
- 4 – A field measuring probe ((30,0 ± 1 mm diameter metal disk);
- 5 – A guard(shielding) ring, earthed, screening the measuring probe;
- 6 – Electronic electrometer / coulomb meter connected to the field measuring probe;
- 7- Digital oscilloscope with memory function recording the voltage curve generated by the coulomb meter. The value of the static field intensity recorded comes from the outlet of the filed measuring probe. Time axe resolution and response time of the measuring device are less than 50 μs.

To design and construct the stand, I have used latest generation equipment. High performing equipment is required by the new standards that demand a high technical level of laboratory equipping.



Measuring electrodes system without the testing sample

Technical data of equipment's

Name of the equipment	Characteristics / Technical data	
Electrometer / Coulomb meter	Keithley ***6514*** *Programmable Electrometer, DC – Multimeter*. This programmable electrometer is capable of measuring voltages (V), currents(A), resistances (Ohm) and static charge (C). The stand is used for measuring electric charges, in nC. Constant incorporated current source; Up to 1200 reading /second; Analogous outlet 0 - 2V, module reversers Coulomb; Interface IEEE-488, RS-232C.	
High voltage source	Model PS325 – 2,50 kV High Voltage Power Supply, 25 Watts; Outlet voltage: 50VDC at ± 5000VDC Maximum current: 5mA	
Rapid high voltage switch with semiconductors	Static switch with MOSFET transistor; Nominal voltage: 6 kV; Nominal current: 50 A; Turn-On Rise Time: maxim 22ns.	
Accessories	Low noise cable, code 237-ALG-2, 3-conductors and at the end Triax contacts and boxes, to connect the probe to the electrometer; Code adaptor 7078-TRX-BNC, adaptor from Triax to BNC to connect to electrometer's oscilloscope	

For the high voltage relay control, I designed and built a *supply and control device.*

Figure 4 presents the electric schematics of the supply and control device for the high voltage relay, and Figures 5 and 6 present the printed circuit and the assembled circuit of the supply and control device of the high voltage relay.

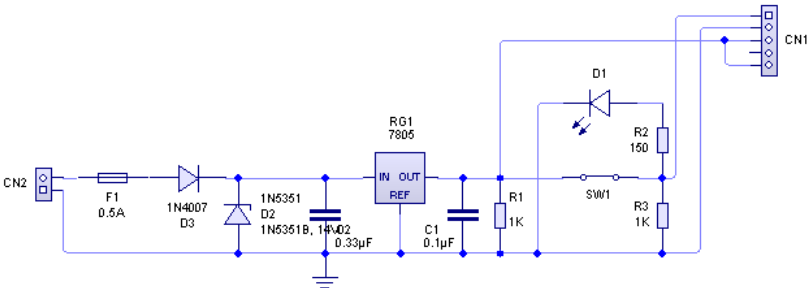


Fig. 4 – ***Electronic diagram.***

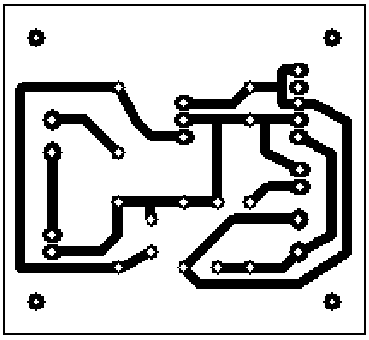


Fig. 5 – Printed circuit.

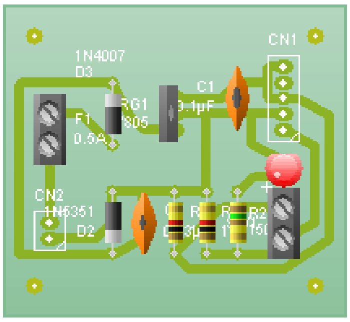
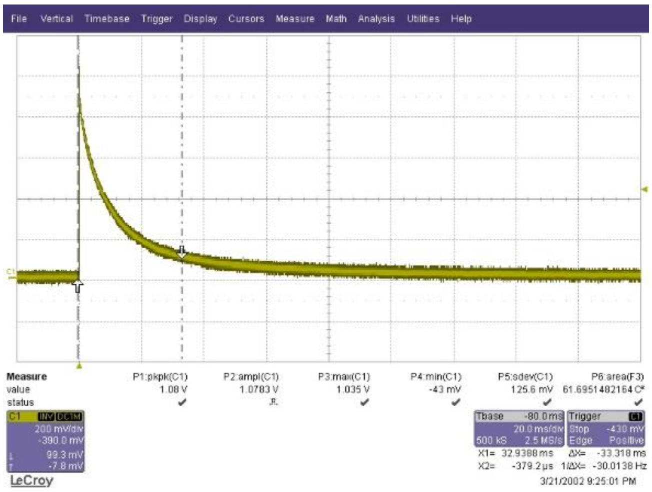


Fig. 6 – Assembled circuit.

Fig. 7 presents the assembled testing stand, equipped with the testing apparatus. The unit includes voltage generator (d.c. source), measuring electrometer and results recording oscilloscope.

Fig. 7 – ***Testing stand equipped with testing apparatus***

In the stage of ***Implementation of the testing method within the testing laboratory*** I conducted a series of trials to experiment the new method in view of identification of the factors of influence to ensure the repeatability and reproducibility of the tests. I ensured this would conform to requirements of SR EN 17025 standard for charge halving times determination tests. Similarly, the accuracy of the results obtained has been subsequently confirmed by inter-laboratory tests.



Oscilloscope result obtained for sample 1 (t_{50}<10ms).

Similarly, I set up the testing procedure to include the testing method in the quality assessment system of the laboratory, extending the area of competence of the laboratory. The new procedure is named as *PI-60 Tests for electrostatic characterization (resistance, conductibility, electric resistivity, electric capacity, electric charge, charge halving time).*

CHAPTER 3.
HAZARD OF INITIATION BY ELECTROSTATIC DISCHARGES OF ELECTRIC DETONATORS, PYROTECHNIC DEVICES, AND ROCKET PROPELLANTS AND FUELS

Explosives for civil uses, some of which are rocket propellants and fuels, and electric detonators, in certain situation can be inopportunely initiated, due to static discharges. Static electricity, as source of occurrence of static discharges, is a phenomenon frequently met in explosive manufacturing industry.

Determination of performances regarding sensitivity of these initiation devices to inopportune initiation by static discharges is very important, since the safety and security of people using these substances depend on them.

In the European Union Member States, regulations regarding testing standards, conformity assessment procedures and their transportation have been harmonized within the following regulations;

- UN recommendation on the transport of dangerous goods, prepared by the Committee of Experts of the Economic and Social Council at the session of 20.04.1957 with subsequent amendments;
- Directive 2014/28/EU of the European Parliament and of the Council of 26 February 2014 on the harmonization of the laws of the Member States referring to the making available on the market and control of *explosive for civil uses*;
- Directive 2013/29/EU of the European Parliament and of the Council of 12 June 2013 on the harmonization of laws of the Member States on the making available on the market of *pyrotechnic articles;*

The principal harmonized standards that support the requirements of *Directive 2014/28/EU* on protection against static electricity are:

- *SR EN 13938-1:2004* Explosives for civil uses. Rocket propellants and fuels. Part 1: Requirements;
- *SR EN 13938-2:2005* Explosives for civil uses. Rocket propellants and fuels. Part 2: Determination of resistance to static energy;
- *SR EN 13763-1* Explosives for civil uses. Detonators and delay relays. Part 1: Requirements;
- *SR EN 13763-13:2004* Explosives for civil uses. Detonators and delay relays. Part 13; Determination of resistance of electric detonators to electrostatic discharge.

The standards harmonized with the requirements of Directive 2013/29/EU on protection against static electricity is *SR EN 16265:2016 „Pyrotechnic articles. Other pyrotechnic articles. Ignition devices."*

Since the application of the new testing methods for the determination of safety parameters is especially important for the conformity assessment of explosives for civil uses and of pyrotechnic devices with the safety requirements foreseen in the specified directives, I have conducted a series of studies and research into the implementation of testing methods according to the new norms and standards in the laboratories of INCD INSEMEX.

In this sense, I have conducted a study resulting in the *DEVELOPMENT OF INNOVATIVE METHODS FOR THE TESTING OF ELECTRIC DETONATORS REGARDING SENSITIVENESS TO ELECTROSTATIC DISCHARGES, for THE DEVELOPMENT OF A TESTING STAND, I CONDUCTED LABORATORY TRIALS AND I IMPLEMENTED THE METHOD IN THE TESTING PROCEDURES IN ACCREDITED REGIME.*

During the study performed within a research project within the program NUCLEU, I have conducted an analysis of the assessment methods and testing of protection performances of electric detonators and I have identified the solutions of implementation of a new testing method of electric detonators for the assessment of conformity with the requirements of prevention of unwanted detonation by static discharges and I have established the necessary tests, testing conditions and application of static discharges. The study was finalized with the design, development and construction of the testing stand for the testing of detonators concerning protection performances against uncontrolled initiation by static discharges.

The testing stand developed conforms to the standardized scheme of principle, as it is shown in Fig. 8.

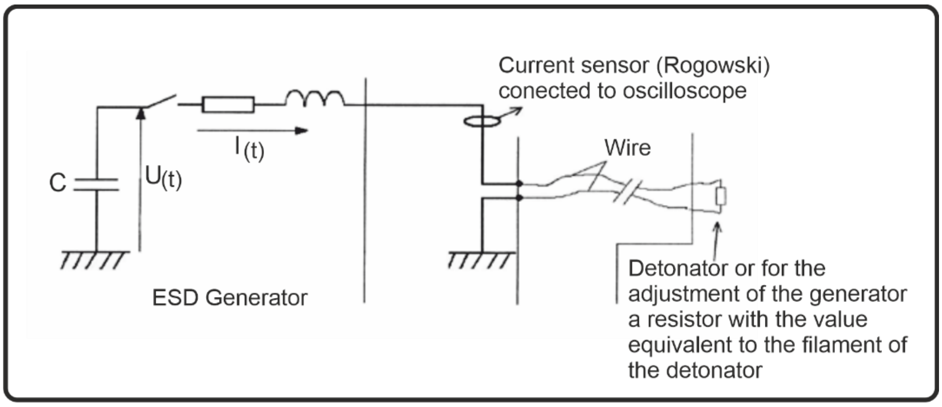


Fig. 8 – *Testing stand equipped.*

The testing stand for the detonator testing on the performances of protection against unexpected initiation by static discharges, developed by me (Fig.9), is made up of:

- *Static discharge generator* (ESD generator) made up of a battery of capacitors with capacities in the range of 500 pF up to 3500 pF, capacitors with more than 30 kV working voltage and a direct current of over 30 kV (HCP 260-120 000 type source with up to 120 kV voltage);
- *ESD current recording system and calculation of ESD impulse delivered to the detonator,* system made up of a Rogowski type CWT015B/1/80UM current sensor inductively connected, an oscilloscope with mathematical functions capable of recording and calculating square functions, with a band width of 500 MHz, LeCroy WaveRunner 6000A type, DSO series;
- Calibration resistance, high voltage connecting cables, vacuum electromagnetic relay, controlled from a supply source, air dryer to maintain relative humidity of maximum 60 %, HBC ADSORPTIONSENTFEUCHTER CR 750 type, conditioned air installation to maintain (20 ± 2) °C temperature.

The testing stand has been constructed using the apparatus purchased within research projects performed in INCD INSEMEX Petroșani.

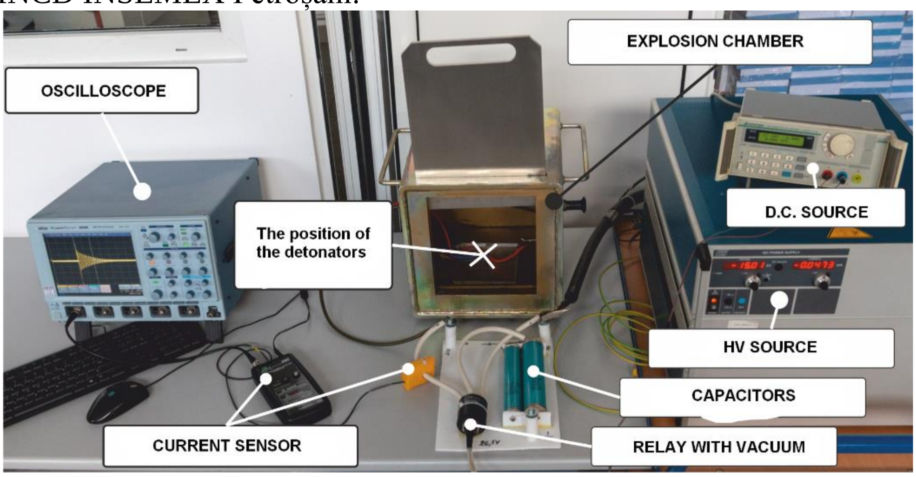


Fig. 9 – *Stand for testing detonators on protection performances against uncontrolled initiation by electrostatic discharges*

In the experimentation stage and implementation of the procedure in the accredited laboratory, I made several trials obtaining discharge curves with decreasing and oscillating shape (lightly harmonized) as in Fig. 10.

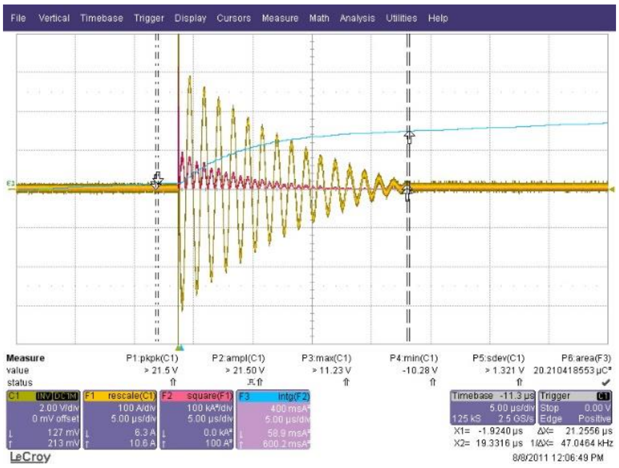


Fig. 10 – ***Impulse oscillograph and curves generated by mathematical functions***

From the trials made I found the following:

- The test results, namely the discharge impulse shape, are significantly influenced by the configuration of the discharge circuit. To obtain the discharge curve in decreasing and oscillating shape (lightly harmonized), as it is specified in the standard, it is necessary for the electric current inductance to be reduced as much as possible;
- Earthing, faulty earthing of the testing circuit leads to erroneous results. The discharge curve is significantly distorted due to stray(rogue) currents;
- Environmental temperature influences the measuring apparatus;
- The testing performances of the new testing stand are limited by the CWT1 inductively coupled current sensor, which supports a peak current of 300 A and by the vacuum relay, which for very high currents can stay with the armours stuck, causing defects.

From the analysis of the standardized testing requirements, of the technical characteristics of the testing apparatus, as well as from the testing results obtained, one can see that the applied testing method with the existing apparatus offers the possibility of correct determination of the sensitiveness of detonators to static charges, in conformity with the standardized testing requirements included in Standard SR EN 13763-13:2004.

Another research subject referred to the *DEVELOPMENT OF AN INNOVATIVE METHOD FOR THE TESTING OF ROCKET PROPELLANTS AND FUELS ON THE SENSITIVENESS TO ELECTROSTATIC DISCHARGES.*

Rocket propellants and fuels are part of the category of explosives for civil uses. The propellant is a deflagrating explosive used for the propulsion of projectiles or to reduce their friction. The propelling fuel can be used as component of gas generators or other devices.

Resistance to static energy is one of the requirements that should be fulfilled by solid rocket propellants and fuels, by powder cakes and by black powders for civil uses, it being a requirement included in Standard SR EN 13938-1.

In Standard SR EN 13938-1 it is mandated that for the testing of EN 13938-2, the sensitiveness to static energy should not be less than 0,5 J.

To determine sensitiveness to static discharges, I have identified the technical requirements for the testing stand that INCD INSEMEX purchased in 2019, a testing stand complying with the conditions stipulated in the national and European standards. As party responsible for the contract, assigned by INCD INSEMEX management, I drew up the justification note, work progress slip, with the requirements the equipment should meet, I have analysed the offers of the bidders, I have made the necessary completions and I have selected the technical offer that fulfilled all the necessary requirements.

The testing stand has as principal component the small-scale equipment X SPARK 10 (Fig. 11), equipment used to determine the resistance to static energy of energetic materials (materials with a high quantity of stored chemical energy).

Equipment X SPARK 10, produced by OZM Research Czech Republic, presents a series of advantages, such as:

- Allows testing of all explosives for civil uses, from primary primer explosives – extremely sensitive -, to powerful explosives – insensitive, and including rocket propellants and fuels;
- Exact measurement of initiation energy of crystalline energetic materials with typical probable mass of approximately 10 mg, in the field of discharge energies from 25 pJ to 25 J and voltages up to 10 kV;
- Performing a complete trial requiring maximum 40 tests, operates in two discharge regimes – oscillating and buffered.

The equipment is part of the newest generation of testing instruments intended to accurately measure the initiation energy (sensitiveness to electrostatic spark) of explosive materials. It is designed to accurately measure the initiation energy of crystalline explosive materials in the field of discharge energies (from 25 pJ to 25 J), at a voltage of up to 10 kV and with the probable typical mass of approximately 10 mg. This equipment is very productive since there are few tests required to determine sensitiveness, usually approximately 30-40 tests with various spark energies, in order to perform a complete test.

Small scale equipment to determine the resistance to static energy of rocket propellers and fuels are made of:

- A built-in high voltage supply source;
- A battery of capacitors;
- A buffer resistor;
- A pneumatically driven remote-controlled high voltage switch;
- A testing chamber including a suction fan as well;
- A set of additional exterior capacitors;
- A remote control;
- A set of accessories and an additional protective screen of Plexiglas, which protects the instrument against the influence of explosion effects during the nonconforming explosive samples testing (which initiate).

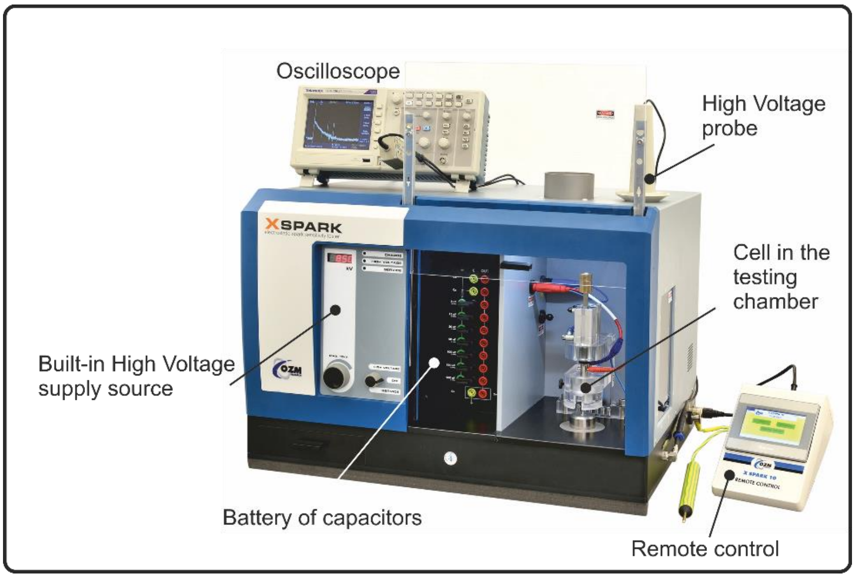


Fig. 11 – *Small scale equipment X Spark 10.*

The working capacity is selectable, it being supplemented with external capacitors. The outlet voltage, of the order of kV, is variable and is adjusted by a potentiometer. The Voltage value is shown on the equipment screen.

When the equipment is used to make tests from Standard SR EN 13938-2, it will be used in “oscillating mode”. The path of the discharging current is highlighted with dotted lines in Fig. 12.

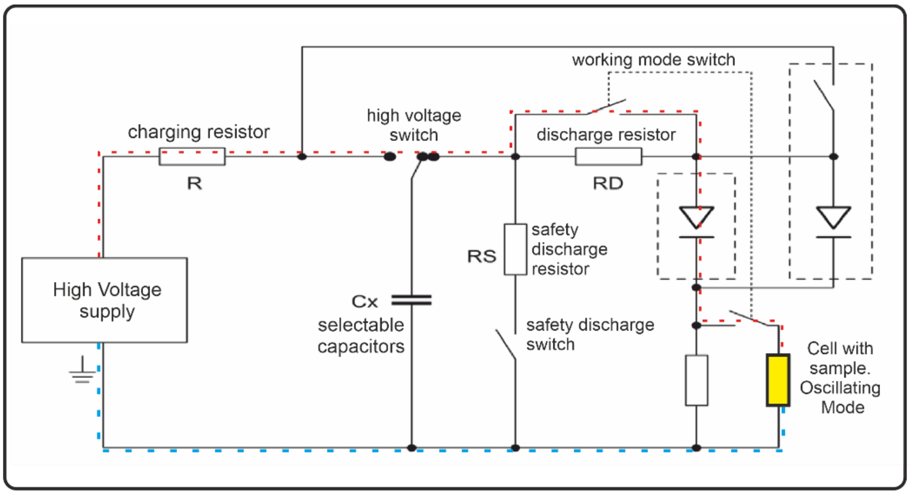


Fig. 12 – The path of the discharging current – Oscillating mode

In order to construct the stand according to Standard SR EN 13938-2 I have separately sourced the cells and copper covers (Fig.13), the special linking cable between X Spark and the cell, as well as a compressor without oil for the pneumatic drive of the high voltage switch.

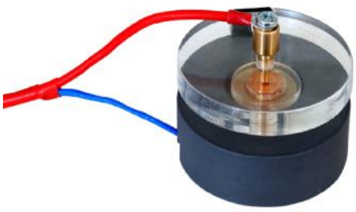


Fig. 13 – *Copper covered cell, assembled*

In view of extension of the competence field of GLI Group of Laboratories within INCD INSEMEX, accredited by RENAR according to the standard *SR EN ISO/IEC 17025 General requirements for the competence of testing and calibration laboratories* I developed the testing procedure for *Determination of resistance to electrostatic energy of explosives for civil uses - rocket propellants and fuels.* This testing procedure will be next validated by experimental laboratory tests.

1. FINAL CONCLUSIONS AND PERSONAL CONTRIBUTIONS

FINAL CONCLUSIONS

Conclusions on static electricity as source of initiation of explosive atmosphere or electric detonators, pyrotechnic articles, rocket propellants and fuels

Static electricity is one of the frequent phenomena met in industrial activities, and also in day to day lives. Many of the effects of static electricity are completely unnoticed, or do not lead to damage, but in some instances static electricity can create hazardous situations.

Static electricity can start fires and/or explosions, unwanted detonation of electric detonators, uncontrolled actuation of sensitive control and monitoring apparatus, and electric shocks to people, or electric shock combined with another hazard (fall, trip over).

Fire initiating static discharge is an initiation risk for gas, vapour of dust explosive atmospheres, unwanted initiation of detonators, pyrotechnic devices, rocket propellants and fuels.

Cone, brush, propagation brush, and spark type fire setting discharges set on fire explosive atmospheres and explosives for civil use. The only discharge that does not lead to ignition of explosive atmospheres of dust is corona type discharge, a discharge of lesser energetic density.

Safety in industry, in places with potentially explosive atmosphere, storage and transport of explosives for civil use, implies the analysis of each situation in part, taking into account potential sources of electrisation, the probability of presence and persistence of explosive atmospheres or explosives, and imposing adequate protection measures in order to reduce risk at an acceptable level.

Ignition risk assessment of explosive atmospheres by static discharges can be done by being aware of the fire setting capacity of the discharge (that is the released quantity of energy) and the sensitiveness of the exiting potentially explosive atmosphere, such as it was characterized by the minimum ignition energy.

Being aware of the fire setting capacity of discharge and sensitiveness of potential explosive atmospheres, one can establish whether the ignition takes place or not.

Assessment of the discharge occurrence of all types is practically the most important thing, and similarly, the most difficult step in the analysis of hazards created by static charges.

Assessment of the uncontrolled initiation risk of electric detonators, of rocket propellants and fuels depends on their sensitiveness to electrostatic discharges.

Conclusion on the analysis of assessment/testing requirements of personal protective equipment to reduce ignition risks of explosive atmospheres by static discharges from people.

If a statically charged person touches a conductive object (for instance, a door handle, a rail, a metal tank), a spark can occur at the point of contact. These sparks, although unlikely to be seen, heard or even felt by a person, can generate ignition of explosive atmospheres. The sparks coming from persons can ignite gas, vapour or even more sensitive dusts. It is very important to prevent static charging of persons that might be exposed to flammable atmospheres, atmospheres that have low minimum ignition energy.

During the study of testing methods for clothing/textile materials for the determination of ESD protective performances, I have highlighted the multitude of testing methods applied worldwide. Many of these methods cannot be used for the new composite materials, textile materials used for the new PPE. I have determined that the method that can be used for the assessment of all textile materials is the one that uses charge decay time, method in conformity with Standard EN 1149-3 method 2 (induction charging).

For the purpose of conducting this testing in the Laboratory of Nonelectric Equipment Ex, Electrostatics, Materials and Personal Protection Equipment within INCD INSEMEX, I have designed, constructed and trialled a new testing stand, which is described in the thesis.

Conclusions regarding the initiation hazard by static discharges of electric detonators, pyrotechnic articles, rocket propellants and fuels.

Explosives for civil uses, category of which rocket propellers, fuels and electric detonators are part, in certain situations, can be inopportunely initiated due to static charges. Static electricity, as source of occurrence of static charges, is a frequently met phenomenon in the explosive manufacturing industry.

Determination of performances related to the sensitiveness of these devices to inopportune initiation by static charges is very important, since of this depend the safety and security of the persons. From the study carried out related to the testing methods of the safety parameters of the electric detonators, pyrotechnic devices, rocket propellants and fuels concerning the initiation by static charges, I have found that they had not been implemented in INSEMEX laboratories. In this sense I have made a series of studies that have been finalized with the development of the testing stand for the testing of detonators regarding protection performances against uncontrolled initiation by static charges, and the purchase of a stand for the testing of rocket propellants and fuels regarding sensitiveness to static discharges. Both stands have been trialled and commissioned. Testing procedures have been developed that were implemented in the laboratory with a view of extending the field of competence of the Group of Testing Laboratories GLI within INCD INSEMEX, accredited by RENAR in conformity with the standard *SR EN ISO/IEC 17025.*

PERSONAL CONTRIBUTIONS

Theoretical contributions

- Ignition risk assessment of dust/air explosive atmospheres by static charges;
- Analysis of inopportune initiation of explosives and/or of initiation devices by electrostatic discharges;
- Analysis of protection performances against electrostatic discharges of various types of detonators;
- Analysis of initiation risk of low intensity electric detonators by electrostatic charges;
- Analysis of assessment/testing of personal protection equipment (PPE) to reduce ignition risk of explosive atmospheres by electrostatic discharges from persons;
- Analysis of clothing/textile materials testing to determine ESD protection performances;
- Analysis of applicability of testing methods to assess protection performances against electrostatic discharges of protective clothing used in potentially explosive atmospheres;
- Establishing testing required to assess conformity of clothing with the applicable safety requirements, in conformity with the new European Standards;
- Analysis of the methods of assessment and testing of protection performances (resistance to electrostatic discharges) of electric detonators.

Hardware contributions

- Design and execution of the stand for measuring charge dissipation capacity by the testing method using induction charging;
- Design and execution of the testing stand for the testing of detonators regarding protection performances against uncontrolled initiation by electrostatic discharges;
- Design and implementation of the stand for determination of sensitiveness to electrostatic discharges of rocket propellants and fuels.

Experimental and applicative contributions

- Development of the documentation of execution of the stand for measuring charge dissipation capacity by induction charging testing method, stand for the determination of PPR antistatic characteristics;
- Development of testing process for the testing method using induction charging and its implementation in the quality system of the laboratory, the aim being to extend the field of competence of the laboratory;
- Experimentation and implementation of the procedure in the accredited laboratory for testing;
- Observations made during the laboratory testing made on electric detonators in the experimentation stage of the stand;
- Development and implementation of the work procedure “Determination of the resistance to electrostatic energy of rocket propellants and fuels” in the laboratory accredited for testing.