

Explosives for civil uses — Propellants and rocket propellants —

Part 2: Determination of resistance to electrostatic energy

The European Standard EN 13938-2:2004 has the status of a
British Standard

ICS 71.100.30

National foreword

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The UK participation in its preparation was entrusted to Technical Committee CII/61, Explosives for civil uses, which has the responsibility to:

- aid enquirers to understand the text;
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Explosives for civil uses - Propellants and rocket propellants - Part 2: Determination of resistance to electrostatic energy

Explosif à usage civil - Poudre propulsive et propergol pour
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électrostatique

Explosivstoffe für zivile Zwecke - Treibladungspulver und
Raketentreibstoffe - Teil 2: Bestimmung der
Widerstandsfähigkeit gegen elektrostatische Energie

This European Standard was approved by CEN on 23 August 2004.

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Foreword

This document (EN 13938-2) has been prepared by Technical Committee CEN/TC 321 "Explosives for Civil Uses", the secretariat of which is held by AENOR.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by April 2005, and conflicting national standards shall be withdrawn at the latest by April 2005.

This document has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association, and supports essential requirements of EU Directive(s).

For relationship with EU Directive(s), see informative annex ZA, which is an integral part of this document.

This European Standard is one of a series of standards with the generic title *Explosives for civil uses - Propellants and Rocket Propellants*. The other parts of this series are listed below:

- EN13938-1 *Part 1: Requirements.*
- EN13938-3 *Part 3: Determination of deflagration to detonation transition.*
- EN13938-4 *Part 4: Determination of burning rate under ambient conditions.*
- EN13938-5 *Part 5: Solid rocket propellants. Determination of voids and fissures*
- EN13938-6 *Part 6: Solid rocket propellants. Guide for the determination of integrity of inhibitor coatings.*
- EN13938-7 *Part 7: Determination of properties of black powder.*

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1 Scope

This document specifies a method for the determination of resistance to electrostatic energy for propellants containing a mass fraction of at least 5 % of particles which pass through a 1 mm sieve. This method does not apply to black powder.

NOTE: If the mass fraction of particles smaller than 1 mm size is less than 5 % the propellant is considered to be insensitive to electrostatic energy and this test is not performed.

2 Normative references

The following referenced documents are indispensable for the application of this document. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies

EN 13857-1:2003, *Explosives for civil uses — Part 1: Terminology*.

EN ISO/IEC 17025, *General requirements for the competence of testing and calibration laboratories (ISO/IEC 17025:1999)*.

ISO 565, *Test sieves; Metal wire cloth, perforated metal plate and electroformed sheet - Nominal sizes of openings*

3 Terms and definitions

For the purposes of this document, the terms and definitions given in EN 13857-1:2003 and the following apply.

3.1 reaction

occurrence of report, crackling, sparkling and/or flame

3.2 partial reaction

change of colour, opening of the cell or heat traces at the surface of the cell

4 Apparatus

4.1 Cells and covers (see Figure 1).

The cell consists of:

- a plastics disc, e.g. polyvinylchloride, thickness $(3,0 \pm 0,1)$ mm, diameter (32 ± 1) mm, with a centred drilled hole, diameter $(6,3 \pm 0,1)$ mm;
- a copper disc, thickness approximately 1 mm, diameter (19 ± 1) mm, which forms the cell base.

The plastics disc is fixed to the copper disc by means of a bead of adhesive around the outer edge.

The cover consists of a copper disc, thickness approximately 0,1 mm, diameter (16 ± 1) mm which is fixed to the upper part of the plastics disc by means of a double-sided adhesive tape.

4.2 Electrostatic energy supply (see Figure 2)

The electrostatic energy supply consists of:

- generator capable of applying a 10 kV continuous voltage;
- three capacitors: capacitance 0,001 μF , 0,01 μF and 0,1 μF , each with a relative tolerance of $\pm 10\%$;
- coaxial cable, length 1,85 m, characteristic impedance 50 Ω , capacitance 100 pF/m, attenuation 95×10^{-3} dB/m at 200 MHz;
- two brass electrodes.

and, if necessary:

- selector switch;
- change-over relay (in vacuum).

5 Preparation of test sample

Take a 100 g sample and sieve it according to the sieving method given in annex B. The fraction which passes through the 1,0 mm sieve shall be used for the test, when this fraction is greater than or equal to 5 g. If not, the test shall not be performed. The test sample shall be conditioned at $(20 \pm 5)^\circ\text{C}$ and $(60 \pm 10)\%$ relative humidity for 24 h.

6 Procedure

Calibrate the generator according to the procedure given in annex C. Alternatively, the screening procedure described in annex D can be used.

Fill the cell, i.e. the hole in the plastics disc, with a portion of the test sample, ensuring that the cover will be in contact with the propellant and without tamping. Close the cell with the cover, using double-sided adhesive tape, and maintain it at $(20 \pm 5)^\circ\text{C}$ and $(30 \pm 10)\%$ relative humidity.

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

During testing and when recovering the remainder of the test portion, observe whether reaction or partial reaction occurs, i.e. a positive event.

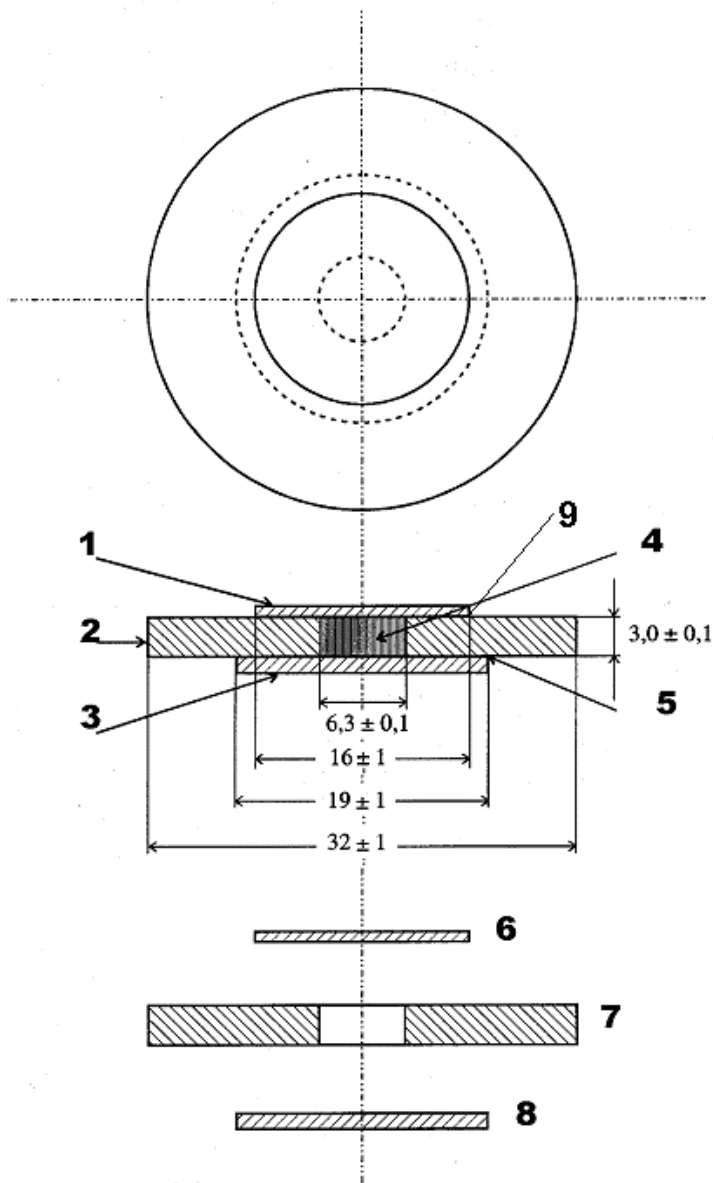
Carry out a series of test runs by using a new cell for each run. Start with a 5 J energy level (capacitance 0,1 μF). Test 20 test portions with the 5 J energy level. If a reaction or partial reaction occurs stop the test and proceed with a 0,5 J energy level (capacitance 0,01 μF) for the next 20 runs. If there is a reaction or partial reaction continue with a 0,05 J energy level (capacitance 0,001 μF) for another 20 runs unless a reaction or partial reaction occurs.

Report the test result as the limiting energy, i.e. the maximum energy level without reaction or partial reaction in a series of 20 runs. For example: if a reaction or partial reaction is obtained at the 0,05 J energy level, express the limiting energy as < 0.05 J.

7 Test report

The test report shall conform to EN ISO/IEC 17025. In addition the following information shall be given:

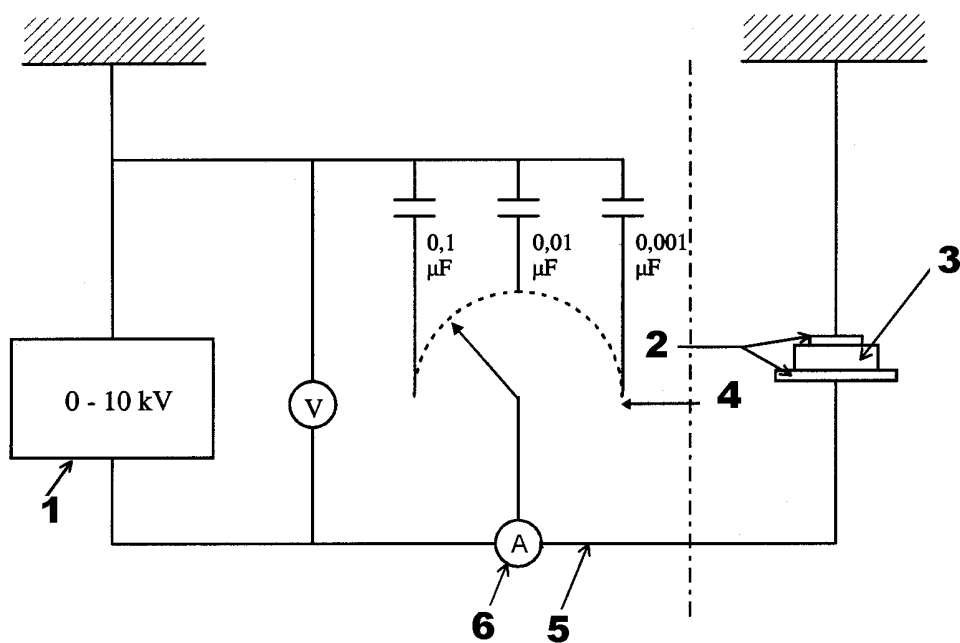
- a) reference to this standard;
- b) complete identification of the substance under test, including grain size distribution;
- c) individual test results, as: 'no reaction', 'partial reaction' or 'reaction';
- d) limiting energy.



Key

- 1 Upper copper disc
- 2 Plastics disc
- 3 Lower copper disc
- 4 Substance under test
- 5 Adhesive bead
- 6 Upper copper disc (cover)
- 7 Plastics disc
- 8 Lower copper disc (base)
- 9 Double-sided adhesive tape

Figure 1 - Cell and cover



Key

- 1 Generator
- 2 Electrodes
- 3 Cell and cover
- 4 Selector switch
- 5 Coaxial cable
- 6 Discharge switch

Figure 2 - Electrostatic energy supply

Annex A
(informative)

Range of applicability of the test method

Range of applicability of the test method: - 30 °C to + 80 °C.

Annex B (normative)

Sieving method

The method uses sieves according to ISO 565 and a sieving machine, e.g. "Fritsch analysette 3". The aperture sizes of the sieves are given in Table B.1.

Table B.1 – Aperture size of the sieves

Number	<i>n</i>	1	2	3	4	5	6	7	8
Aperture size (mm)	X_n	0,10	0,16	0,25	0,40	0,63	1,0	1,6	2,5

The sieves, cover lid and lower collection bin are made of stainless steel.

Take 1 000 g (*M*) of the sample. Place the sieves on top of the collection bin in the order of the indicated numbers. Put the sample in the top sieve and close the column of sieves with the cover lid. Operate the sieving machine for 15 min. Weigh the different fractions, m_1 is the amount of sample passing through sieve number 1, m_2 is the amount of sample passing through sieve number 2 and retained on sieve number 1, etc.

The different percentages are calculated according to:

$$P_1 = 100 \times m_1/M$$

$$P_2 = 100 \times (m_1+m_2)/M$$

.....

$$P_8 = 100 \times (m_1+m_2+\dots+m_8)/M$$

and the balance:

$$B = 100 \times (m_1+m_2+\dots+m_8+m_9)/M$$

The amplitude of the sieving machine is adjusted, if the machine has this option, to a medium level, e.g. "5" for the Fritsch apparatus.

Annex C (normative)

Calibrating procedure for electrostatic discharge generator

C.1 General

This annex describes the procedure to be used to calibrate the generator ; this calibration is necessary to ensure that the generator used will provide consistent results from one testing place to another.

C.2 Device to be tested

Only the discharge circuit of the generator is calibrated, including cables which are used to connect the testing cell.

If the generator includes an optional serial resistance, R_o (for example: 330 Ω additional resistance to simulate the "human body discharge"), the calibration shall be performed without the optional resistance (C.4.1 and C.4.2); then a supplementary calibration procedure as described in C.4.3 shall be carried out.

C.3 Apparatus

- **High Voltage probe** with a calibrated high input impedance (usually approximately 1 G Ω)

Calibration of the input impedance may be obtained by simple measurement with a Ohmmeter; voltage at which the measurement is made shall be chosen as high as possible (approximately 1 kV for example)

- **Current probe** (using inductive effect)

With a transfer function of 0,1 V/A

- **Oscilloscope**

Band width \geq 20 MHz

C.4 Procedure

C.4.1 Voltage and capacitor calibration

- Connect the output of the generator on the High Voltage probe.
- Set the generator at the highest voltage intended to be used for the testing.
- Discharge the generator in the same manner as used for testing a sample.
- Record the voltage versus time.

The curve should be highly damped (exponential decrease).

- Determine U_{\max} .

- Calculate the time constant $\tau = RC$ using the following equation:

$$\tau = (t_2 - t_1) \text{Ln} \frac{U_1}{U_2}$$

where, U_i is voltage and t_i is time at the point i of the exponential discharge curve.

Preferably choose points 1 and 2 respectively as 10 % and 90 % of U_{\max} for calculation.

- Calculate C (capacitance of the discharge circuit) from τ and R (resistance of the discharge circuit \equiv impedance of the high voltage probe).

C.4.2 Discharge circuit calibration

- Place the current probe on the discharge circuit.
- Shunt the output of the generator, including connecting cable.
- Discharge the generator in the same manner as used for testing a sample.
- Record the current versus time.

The curve should be weakly damped (pseudo-periodic).

- Signal analysis

- Determine the voltage (or current) at two extremes of the pseudo-periodic signal. Usually the best accuracy is obtained when these two extremes are chosen in such a way that $0,5 \leq n \leq 3$, where n is the number of pseudo-periods between the two extremes

- Determine the pseudo-period T ; for the best accuracy, calculate T from values of t for which $U(t) = 0$ (or $I(t) = 0$)

- Calculate L (inductance) and R (dynamic resistance) of the discharge circuit using the following equations:

$$L = \frac{T^2}{C(4\Pi^2 + a^2T^2)} \text{ and } R = 2aL; \text{ with } a = \frac{2}{nT} \text{Ln} \frac{|M_1|}{|M_2|}$$

where:

C is the capacitance determined in C.4.1

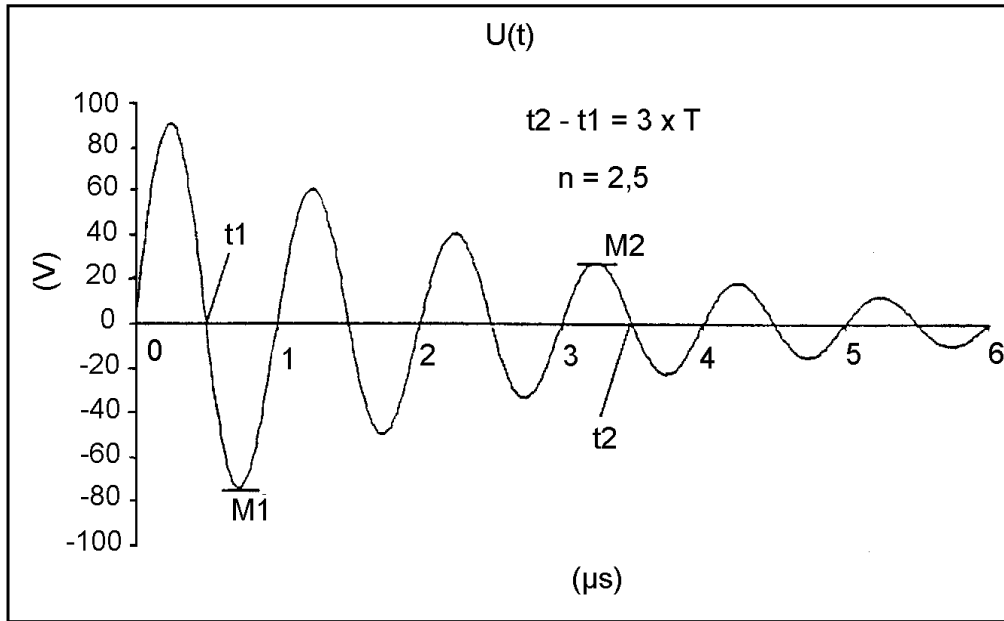
T is the pseudo-period

Π is 3,14...

a is the damping factor

n is the number of pseudo-periods between M_1 and M_2

M_1, M_2 are the voltage (or current) at extremes 1 and 2



Key

V voltage

μs time

Figure C.1 – Example for a discharge record (voltage vs. time)

C.4.3 Additional calibration in case of an additional series resistance

- a) Shunt the output of the generator (including the additional resistance).
- b) Connect the High Voltage probe to the "hot point" of the additional resistor (opposite side from ground).
- c) Set the generator at the highest voltage intended to be used for the testing.
- d) Discharge the generator in the same manner as used for testing a sample.
- e) Record the voltage versus time.

The curve shall be highly damped (exponential decrease).

- f) Determinate U_{max} .
- g) Calculate the time constant $\tau = RC$ using the following equation:

$$\tau = (t_2 - t_1) \text{Ln} \frac{U_1}{U_2}$$

where, U_i is voltage and t_i is time at the point i of the exponential discharge curve .

Preferably choose points 1 and 2 respectively as 10 % and 90 % of U_{max} for calculation.

- h) Calculate R (total resistance of the discharge circuit) from τ and C (capacitance of the discharge circuit) determined in C.4.1.

C.5 Requirements for the generator

Maximum error on capacitance shall be $\pm 10\%$ from required value.

Maximum error on voltage shall be $\pm 5\%$ from required value.

Total inductance of the discharge circuit shall not be higher than $5\ \mu\text{H}$.

Total dynamic resistance of the discharge circuit shall be $5\ \Omega \pm 0,1\ \Omega$ with a 10kV load of the capacitor.

If an additional series resistance is used, the resistance shall be $R_o \pm 5\%$.

Annex D (normative)

Screening method for electrostatic discharge generator

D.1 Material

Pentaerythritol tetranitrate (PETN), technical grade
Two particle sizes: < 20 µm and 0,125 mm to 0,5 mm.

D.2 Procedure

To check that the calculated amount of energy is dissipated into the sample, a screening method using PETN can be used as an alternative to the calibration procedure described in annex C. The calibration procedure shall be used at regular intervals, especially if the apparatus has not been used for some time or when repair or changes are made to the apparatus. The screening method described here can be used at irregular time intervals (e.g. during a long test series) or to get a quick check.

The two grades of PETN described in clause D.1 shall be used. A comparative study performed by four different laboratories, using their own apparatus and source of PETN, has confirmed that the smallest particle grain size (i.e. < 20 µm) has a limiting energy of < 0,05 J and the largest particle size (i.e. 0,125 mm to 0,5 mm) has a limiting energy of 0,5 J.

When the same limiting energies are obtained, it can be assumed that the apparatus is functioning properly.

Annex ZA (informative)

Clauses of this European Standard addressing essential requirements or other provisions of EU Directives.

This European standard has been prepared under a mandate given to CEN by the European Commission and the European Free Trade Association and supports essential requirements of EU Directive 93/15/EEC.

WARNING : Other requirements and other EU Directives may be applicable to the product(s) falling within the scope of this standard.

The clauses of this standard are likely to support requirements of Directive 93/15/EEC.

Annex 1, Essential safety requirements General requirement I.1

Special requirement II.1(i).

Compliance with this standard provides one means of conforming with the specific essential requirements of the Directive concerned and associated EFTA regulations.

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