

Methodology For Testing Physical Samples (Models) Of A Chemical Power Source Intended For Single Use In Defence Industry Products

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Abstract. *The proposed and developed methodology is designed to determine the indicators of reliability and operability of disposable chemical power sources. It is recommended for conducting tests of physical samples (real models) under various environmental impacts, under mechanical-dynamic loads, as well as in various operating conditions. The methodology includes subjecting the samples to testing the impact of destabilizing factors of the environment in order to determine their workability and reliability. Conducting this independent test is required due to the importance of the power source and in order to obtain sufficient statistical information about the reliability of its electrochemical elements. To ensure the completeness and comprehensiveness of the study, an additional methodology has been compiled – Methodology for Identification of Physical Models of a Power Source.*

Key words: *methodology, physical samples, destabilizing factors.*

I. INTRODUCTION

The purpose of the Methodology is to subject to testing chemical power sources applicable in defence industry.

This methodology is implemented in accordance with the following specifications and safety aspects:

- the electro-chemical elements used for building the power sources (PS) and the power sources themselves should be designed so that they can operate without creating conditions for temperature higher than the critical temperature specified by the manufacturer;
- the electro-chemical elements used as well as the power sources should be designed so that a brief short circuit is avoided in normal conditions of exploitation and during transportation;
- each individual power source should be designed so that the same electro-chemical elements are used from the same manufacturer, and the usage of constructive

elements from different manufacturers in one and the same sample is forbidden;

- lithium power sources containing lithium elements or sequence of elements connected in parallel should be equipped efficiently so that they can prevent reverse current, jacket swelling or casing rupture.

II. MATERIALS AND METHODS IN TESTING PHYSICAL SAMPLES OF POWER SOURCES

The tests of the electro-chemical systems containing the chemical element lithium (Li) are conducted subject to additional conditions. They are specified depending on the amount of lithium (Li) in the tested elements. The testing of these physical models (experimental samples) is considered as an individual type, and they are subject to additional tests depending on the mass of lithium in them. When the lithium (Li) is more than 0.1 g or when it is more than 20 % of the total mass, regardless of where it is contained – in the electrodes or the electrolyte, additional tests of the samples are conducted. The sets of electrochemical elements in the power source (battery) containing more than 500 g of lithium are not subject to testing provided that they have been assembled using electrical bonding. The power sources must have gone through all necessary tests or be equipped with a system capable of controlling the pressure in the assembly and preventing short circuits.

The selection of samples for testing includes each individual type of electrochemical elements and power sources which are to be tested by means of developed physical models. To conduct the tests, physical samples (models) of power sources have been developed. They were used to perform tests of reliability indicators of the electrochemical systems that make up the power source. The number of physical samples of power sources needed to perform the tests are indicated in Table 1 [1].

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TABLE 1 NUMBER OF SAMPLES SUBJECT TO TESTING [1]

Primary elements and batteries				
Number of samples T-1 – T-5	Elements		Batteries	
	10 elements	10 elements	5 batteries	5 batteries
Number of samples T-6	Elements		Batteries	
	5 elements	5 elements	5 compound	5 compound
Number of samples T-6	Elements		Batteries	
		10 elements	Tests are not mandatory.	

The tests are repeated if they prove that the primary lithium elements or the power sources assembled with them do not correspond to the requirements set by the tactical-technical assignment (TTA). Before the tests are repeated, the necessary measures must be taken to eliminate the fault or faults leading to failure in the conducted tests.

III. RESULTS AND DISCUSSION

Safety instructions – in conducting the indicated tests, procedures are used which may result in injuries if the necessary safety measures are not taken.

Temperature of the environment – if nothing else has been indicated, all tests are to be conducted in room temperature of 20 ± 5 °C.

Permissible error when measuring parameters – the total error of the controlled and measured values should be within the following interval:

- a/ ± 1 % – for the voltage;
- b/ ± 1 % – for the current;
- c/ ± 2 °C – for the temperature;
- d/ $\pm 0,1$ % – for the time;
- e/ $\pm 0,1$ % – for the dimensions;
- f/ ± 1 % – for the capacity.

The given limit values also include the combined values of the error of the measuring instruments, the error of the used technology of the conducted measurement and other sources of error arising in the process of conducting the tests.

The electrochemical elements used are subjected to an external inspection and should meet the optimal characteristics and safety determined by the manufacturer [2].

A. Evaluation of the test results of power sources

- *Displacement* in the test process occurs if one or more electrochemical elements of the power source, during the test, have fallen out of their package, changed their position inside the package or there is a displacement of the elements relative to each other.

- *Deformation* of the electrochemical elements and power sources (batteries), in the process of their testing, happens if there is a change in their physical dimensions by more than 10%.

- A *short circuit* in the test process occurs if the values of the voltage of the open circuit of electrochemical elements or the power source, after the

test is completed, is less than 90 % of the value measured immediately before the test. This requirement does not apply when the complete discharge of the electrochemical elements or the power source (battery) built with them is subjected to the test.

- *Overheating* of electrochemical elements and power sources in the test process occurs if the housing external temperature exceeds the temperature of 170°C.

- The *mass loss* of electrochemical elements and power sources $\Delta m/m\%$ is calculated by formula 1:

$$\Delta m/m = \frac{m_1 - m_2}{m_1} 100 \%, \quad (1)$$

m_1 – mass before the test, g;

m_2 – mass at the end of the test, g.

- *Airtightness violation* is considered if, during the test, there has been gas leakage from the tested electrochemical elements through a device designed to reduce the internal pressure. The exhaust gas may contain entrained particles.

- *Ignition* of the electrochemical elements and power sources, in the process of testing, occurs if a fire is observed coming out of them.

- *Destruction* of electrochemical elements or power sources, in the test process, occurs when mechanical destruction of the housing is observed, which is accompanied by the release of gas or the leakage of electrolyte, but there is no release of internal solid materials from their composition.

- An *explosion* in the test process takes place if penetration of solid particles is observed from the electrochemical element or the power source through a mesh screen made of tempered aluminium wire with a diameter of 0.25 mm with a mesh density of 6 - 7 wires per 1 cm, placed at a distance of 25 cm from them [3].

B Transportation testing of physical samples of power sources designed as a defence product

The presented Table 2 provides an overview of the conducted tests and the set requirements for power sources and their constituent elements. To ensure safety during transportation and in cases of improper use of the electrochemical elements and power sources, it is necessary to conduct a test of their packaging. According to the proposed methodology, transport tests include:

TABLE 2 TESTS AND REQUIREMENTS TO TESTING SAMPLES [3]

Indication of	Name	Requirements
Transportation requirements	T-1	Altitude HM(NM); HЭ(NL); HГ(NV); HK3(NC); HP(NR); HB(NE); HO(NF);
	T-2	Temperature cycle HM(NM); HЭ(NL); HГ(NV); HK3(NC); HP(NR); HB(NE); HO(NF);
	T-3	Vibrations HM(NM); HЭ(NL); HГ(NV); HK3(NC); HP(NR); HB(NE); HO(NF);
	T-4	Shock HM(NM); HЭ(NL); HГ(NV); HK3(NC);

			HP(NR); HB(NE); HO(NF);
T-5	External short circuit		HT (NT); HP(NR); HB(NE); HO(NF);
T-6	Dynamic load		HT (NT); HB(NE); HO(NF);
Abbreviation code: HM(NM) – absence of mass loss; HK3(NC) – absence of external short circuit; HИ(ND) – absence of deformations; HB(NE) – absence of explosion; HO(NF) – absence of burning; HО(NL) – absence of leaks; HP(NR) – absence of destruction; HИ(NS) – absence of displacement; HT(NT) – absence of overheating; HГ(NV) – absence of airtightness violation;			

1. Testing T-1: Altitude

a) *Purpose:* Testing T-1: Altitude models transport by air in low ambient pressure conditions.

b) *Conducting the test:* The power sources are placed in a chamber with a temperature of 0 ± 2 °C reached. The pressure is reduced to a value of 19.20 ± 0.31 kPa (145 Torr). The duration of the stay is 40 min. The voltage values of the tested products are measured, and the duration of the test is 10 min. Then, the temperature and air pressure in the chamber with the experimental samples are increased to values under normal climatic conditions: $T = 20 \pm 5$ °C and atmospheric pressure 101.32 ± 0.31 kPa (760 Torr) again with a duration of 10 min. The temperature of the tested products is stabilized at 20 ± 5 °C for 30 min. The indicators of the tested products are measured.

c) *Requirements:* In the process of testing electrochemical elements and power sources, there should be no loss of mass, electrolyte leakage, airtightness violation, brief short circuit, destruction, explosion or burning.

2. Testing T-2: Temperature cycle

a) *Purpose:* The test is designed to evaluate the integrity of assemblies, airtightness, and internal connections of electrochemical elements and power sources. The test is carried out in a temperature cycle.

b) *Conducting the test:* The experiment is conducted after the temperature of the tested power sources has stabilized at a temperature of 17.8 ± 0.1 °C for 2 (two) hours. The voltage of the tested power sources is measured at idle.

Conditions for conducting a test for one cycle:

The indicators of the power sources are measured after placing them in a chamber and reaching a temperature of minus 40 ± 2 °C for 30 min. The voltage of the tested products is measured at idle speed for 2 min. The power sources are placed in a chamber at a temperature of 50 ± 2 °C. The duration of exposure to the specified temperature is 30 min. The technological time of moving the tested products from one chamber to the other is 2.3 ± 0.1 min. The measurement of the studied indicator (electrical voltage) of the tested products at idle speed is about 2 min. The total time for testing one of the products in one cycle is 1 h and 6 min [4, 5].

c) *Requirements:* In the process of testing electrochemical elements and power sources, there should be no loss of mass, electrolyte leakage, airtightness

violation, brief short circuit, destruction, explosion or burning.

3. Testing T-3: Vibrations

a) *Purpose:* The specified test models the impact of vibrations during transportation of electrochemical elements and power sources. The test conditions are based on the range of vibrations (vibration stress) during service handling and transportation of the elements and power sources.

b) *Conducting the test:* The electrochemical elements and power sources under test must be firmly fixed to the platform of the vibration test device without being deformed, but also so that the vibrations are transmitted as accurately as possible. The test items are subjected to sinusoidal vibration in accordance with Table 3. This cycle is repeated up to 6 times in each of the three mutually-perpendicular directions. One of the directions must necessarily be perpendicular to the surface of the test sample with electrical terminals [6].

TABLE 3 PARAMETERS OF SINUSOIDAL VIBRATIONS [6]

Range of frequency		Amplitude	Duration of the logarithm	Axes	Number of cycles
From	to				
$f_1 = 7\text{Hz}$	f_2	$a_1 = 1\text{gn}$	15 min	X	12
f_2	f_3	$s = 0.8\text{mm}$ m		Y	12
f_3	$f_4 = 200\text{Hz}$	$a_2 = 8\text{gn}$		Z	12
and return frequency $f_1 = 7\text{Hz}$				Total time	36
<i>Note:</i> Vibration amplitude – this is the maximum absolute value of displacement or acceleration. For example, a displacement amplitude of 0.8mm corresponds to a displacement scale of 1.6mm. Abbreviation code: f_1 and f_4 – lower and upper frequency; f_2 and f_3 – transition frequencies; a_1 and a_2 – acceleration amplitude; s – displacement amplitude.					

c) *Requirements:* In the process of testing the elements and batteries, there should be no loss of mass, electrolyte leakage, airtightness violation, brief short circuit, destruction, explosion or burning.

4. Testing T-4: Shock impact

a) *Purpose:* This test simulates a sharp mechanical impact on the elements or batteries during transportation.

b) *Test conducting procedure:* The test items shall be securely fastened to the test rig by means of fasteners securing all mounting surfaces of each test item and battery. They must be subjected to three shocks in each of the three mutually-perpendicular mounting positions. The parameters of each stroke must correspond to the data in table 3.

TABLE 4 PARAMETERS OF THE SHOCK INTENDED FOR TESTING [7]

Product type	Wave type	Maximum acceleration	Impulse length, ms	Number of shocks
Small	semi-sinusoidal	150 gn	6	3
Big	semi-sinusoidal	50 gn	11	3

c) *Requirements:* In the process of testing the elements and batteries, there should be no loss of mass, electrolyte leakage, airtightness violation, brief short circuit, destruction, explosion or burning.

5. Testing T-5: External short circuit

a) *Purpose:* The test simulates the state of an external short circuit.

b) *Conducting the test:* Tested cells and batteries are stabilized at external temperature and then subjected to a momentary external short circuit. The samples are monitored for 6 hours after the end of the exposure [8].

c) *Requirements:* In the process of testing and monitoring the samples during all the 6 hours, there should be no overheating, destruction, explosion or burning.

6. Dynamic load (impulse shock)

a) *Purpose:* The test simulates an external short circuit.

b) *Conducting the test:* Test cells and batteries are placed on a flat metal plate. Each component of the battery must be subjected to one dynamic shock. The test samples were observed for 6 hours after the final impact. Products that have not passed other transport tests are subjected to this test [8].

c) *Requirements:* In the process of testing and monitoring the samples during all the 6 hours, there should be no overheating, destruction, explosion or burning.

C Safety measure when conducting the tests

When conducting the tests, the necessary safety measures must be observed. The normal operation of power sources is associated with the release of a certain amount of heat into the environment. Depending on the operating mode of the product and the possibility of heat transfer to surrounding objects, overheating, burning or explosion may occur. For this reason, it is recommended to use safety glasses, clothes and gloves, and if there is a risk of exploding the test object, the experiment should be carried out behind safety glass.

The defective condition of the electrical equipment in the test laboratory or its improper handling can lead to accidents or be the cause of fire or explosion.

Current up to 36 V has been found to be safe, and therefore, all portable electrical equipment in the laboratory must operate at 12 V (the tested power sources have a nominal voltage of 12 V).

The methodology is designed to prove the performance of batteries built from different electrochemical systems. The proposed tests are aimed at batteries intended for single use.

D Methodology for identification of physical models of the power source

According to the limitations set in the methodology, the tested physical samples (batteries) of a product for the defence industry are intended for single use. The components from which the experimental samples are built are identical, except for the chemical composition of the power source. The electrochemical elements that make

up the power sources of the physical models are made up of three electrochemical systems – manganese-zinc, silver-zinc and lithium. The choice of the type of electrochemical cells used is dictated by their availability and variety of sizes.

The main task of the proposed methodology is related to the possibility of correct identification of each physical model during laboratory tests. Therefore, each of them should be labelled starting from the type of electrochemical system used. The characteristics of the elements that make up the electrochemical system are also described – electrodes, electrolyte, separators, and, if possible, the chemical reactions taking place.

a) Electrochemical systems used for building the power source of the physical models of the product

The following electrochemical systems designed for a disposable power source have been developed and prepared for testing:

- Chemical current sources intended for single use based on manganese and zinc, which can be with salt or alkaline electrolyte. In manganese-zinc current sources with salt electrolyte $\text{Zn}|\text{NH}_4\text{Cl}|\text{MnO}_2$, the battery case is also an **anode** (made of Zn), the active substance of the **cathode** is electrolytic manganese dioxide (MnO_2) or chemical manganese dioxide, NH_4Cl , ZnCl_2 or a mixture of the two substances is used as an **electrolyte**. The electrolyte is located in the thickened areas or in a microporous separator. To reduce the corrosion rate of zinc, corrosion inhibitors are added to the electrolyte. The advantages of these sources of electric current (batteries) are related to the low price and the large number of sizes, the relatively simple production technology and the readiness to be used immediately. The disadvantages are related to the downward discharge curve during operation, the relatively low specific energy released, and a significant drop in performance at high load and low temperatures.

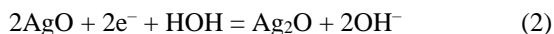
Manganese-zinc power sources are also produced with alkaline electrolyte $\text{Zn}|\text{KOH}|\text{MnO}_2$. They use powdered Zn as **anode** and MnO_2 as **cathode**. The **electrolyte** is a gel solution of KOH or KOH in a matrix. Corrosion inhibitors are included in the composition of the anode and electrolyte. Compared to salt electrolyte power sources, alkaline ones have a higher capacity and energy density in operation, especially at high loads and low temperature, but have a higher price value [9].

- In the silver-zinc primary cells $\text{Zn}/\text{Ag}_2\text{O}$ or $\text{Zn}|\text{KOH}|\text{Ag}_2\text{O}, \text{Ag}^+$, powdered zinc is used for the **anode**, silver oxide for the **cathode**, and KOH or NaOH solution for the **electrolyte**. Silver is reduced on the cathode from Ag(I) to Ag(II) during operation of the chemical current source (the battery), i.e. in the discharge mode, the alkaline electrolyte is used as a donor of hydroxyl groups, and the following electrochemical processes take place:

- an oxidation reaction of zinc metal takes place at the anode:



- the following reaction takes place at the cathode:



i.e. a reduction reaction of the divalent silver ion to a monovalent ion and subsequently to pure silver occurs according to the scheme:



the total equation is written in the form:



Silver power sources (batteries) of this type have a horizontal discharge curve, high energy density and low self-discharge. They can work at high loads, but the disadvantage is the high cost of their components.

- Lithium primary current sources with a **solid cathode and aprotic electrolyte**, the reducing agent is lithium, and the oxidizing agent is metal oxides, sulphides, or fluorocarbons. The **electrolytes** are solutions of lithium salts (LiClO_4 , LiBF_4 or LiBr) in aprotic solvents. In lithium current sources with **liquid or dissolved oxidant** $\text{Li}|\text{LiBr}|\text{SO}_2$ and $\text{Li}|\text{LiAlCl}_4, \text{SOCl}_2|\text{SOCl}_2$, the cathodes in the electrochemical current source are insoluble and made of carbon materials deposited on aluminium (for SO_2), based of nickel steel or stainless steel. The **electrolyte** in lithium sulphur dioxide cells is LiBr dissolved in acetonitrile; in the elements with thionyl chloride and sulphuryl chloride – LiAlCl_4 SOCl_2 or SO_2Cl_2 with additives.

Lithium primary electrochemical current sources have higher capacity and energy density, a wider operating temperature range, better performance at lower temperatures, and a lower self-discharge rate compared to the same parameters of manganese-zinc sources of power supply. Their main drawback is their high price. Lithium primary sources of electrochemical current are used in medical, industrial and military electronics.

b) Specifying the reliability and operability requirements for the power sources

When designing the power sources (batteries), the requirements for their reliability and operability are of great importance. These two indicators are affected by the magnitude of the electric current and the electric voltage, as well as the duration of the operation of the final product. It is also essential to determine correctly the storage period of the power source before it is put into use. The requirements for the constructed and tested power sources can be summarized as follows:

- electrical voltage from 6 to 15 V and more;
- nominal load current from 10 μA to 1 A and more;
- working time from 1 ms to 60 min and more;
- operability in a temperature range from minus 40 °C to plus 50 °C;
- storage period from 5 to 20 years.

One of the most important requirements for the power sources is their high reliability when working in conditions of high accelerations that occur at the time of their operation.

c) Building and numbering the power sources of the

physical model

Depending on the physical parameters of the power source, the elements of the appropriate size are selected. This is done during their construction, depending on the offered sizes, operating characteristics and indicators of the building electrochemical elements.

The first power supplies are developed for research purposes and are made of manganese-zinc electrochemical elements (alkaline elements) and silver-zinc electrochemical cells, which are denoted LR and SR, respectively. According to the different standards, the used building electrochemical elements are marked as model: LR44; SR44; A76; SR44SW; AG13; SG13; LR154; S76; EPX76; 157; 303 or 357. They have a weight of 1.8 g, a diameter of 11.6 mm, a height of 5.4 mm, a nominal voltage of 1.5 V and a capacity of 110 – 125 mAh. Lithium power sources are also presented, and they are denoted by CR, and CR-1/3N model elements were also used for their construction. The latter have a diameter of 11.6 mm, a height of 10.8 mm, a nominal voltage of 3 V and a capacity of 170 mAh.

Eight 1.5 V electrochemical cells or four 3 V lithium electrochemical cells were used to construct the power source. The resulting final rated voltage was 12 V.

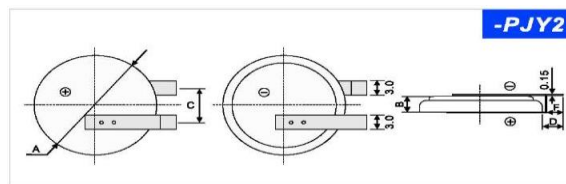


Fig. 1. Connection type used when constructing the physical power source models

The P-JY2 series connection between the electrochemical elements in the power source is made with nickel-plated connection plates of the AA-4-20-13R type with a thickness of 0.13 mm and a width of 4 mm. An ARM-10KAS spot welding machine was used.

In parallel with the construction of the physical samples, a visual model was also built. With its help, the position of the electrochemical elements, the location of the connecting elements, the size of the channel for the connecting wires and, last but not least, the dimensions of the final model (sample) were determined in 3D space.

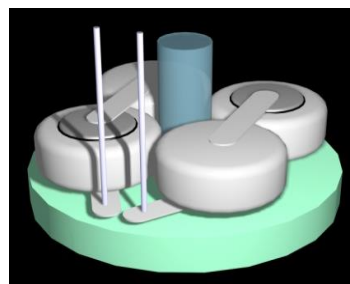


Fig. 2 Visual 3D model of a power source built with 4 electrochemical elements on one level [10]

The conducted experiments and the positive results shown by the tested physical models of the power source necessitate the transition to the stage of development of a sample made up of two levels of electrochemical elements

and, accordingly, a virtual sample of the proposed power source.

The numbering of the power sources intended for physical samples of the product is carried out according to a developed numbering system.

It is based on the type of electrochemical elements used, their size and operating voltage.

To indicate the type of the electrochemical system of the elements, the designation LR is used for manganese-zinc elements, SR – for silver-zinc elements, and CR – for lithium elements.

The following system is proposed for numbering the power sources (batteries) related to their identification during the tests:

1. The batch number is indicated in brackets, and it consecutively includes: the year and month of creation of the power source (PS); the type of electrochemical system (respectively for LR it is indicated by the number 1, for SR – by the number 2, and for CR – by the number 3), and the serial number of the power source in the batch.

2. The model of the electrochemical elements, when it is different from the one specified, is written after the batch number in brackets.

3. The manufacturer of the electrochemical elements is specified at the end of the identification number, after the brackets.

The serial number obtained from the power source is printed on the housing. It is entered during the tests and serves to compare the achieved results.

IV. CONCLUSIONS RELATED TO THE PROPOSED METHODOLOGY

The methodology is recommended when testing physical samples (real models) as part of a product intended for the defence industry. The tested samples are subjected to various environmental influences and mechanical-dynamic loads. The tests are selected depending on the expected conditions of transportation, storage and operation of the final product and the specified requirements in the tactical-technical task (TTT) for it.

As required, the power source samples used and developed are exact models of the final product. The methodology envisages that they should be tested for the impact of destabilizing environmental factors in order to determine their operability and reliability. Conducting this independent test is required due to the importance of the power source to guarantee the operability of the entire

product and in order to obtain sufficient statistical information on the reliability of the electrochemical elements that make it up. In connection with this, it is appropriate to test three electrochemical types of power sources (alkaline, silver and lithium). They differ in the electrochemical elements used for their construction – their electrochemical composition, sizes, and performance characteristics. The proposed Methodology for Identification of the Physical Models of a Power Source makes it easier to work with them.

With the developed Methodology, a series of physical samples were tested, and the results shown satisfy the requirements of the specifications.

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